Total Synthesis of the *Strychnos* Alkaloid (+)-Minfiensine: Tandem Enantioselective Intramolecular Heck–Iminium Ion Cyclization

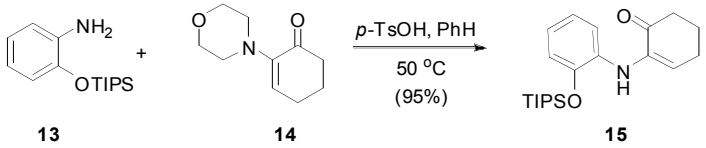
Amy B. Dounay, Philip G. Humphreys, Larry E. Overman,* and Aaron D. Wrobleski

Department of Chemistry, 1102 Natural Sciences II, University of California, Irvine, California 92697–2025, USA

Supporting Information – Table of Contents

For materials and methods, see: Becker, M. H.; Chua, P.; Downham, R.; Douglas, C. J.; Garg, N. K.; Hiebert, S.; Jaroch, S.; Matsuoka, R. T.; Middleton, J. A.; Ng, F. W.; Overman, L. E. *J. Am. Chem. Soc.* **2007**, *129*, 11987–12002.

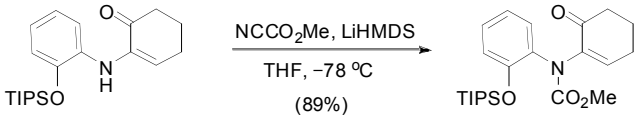
Experimental Procedures.



13 **14** **15**

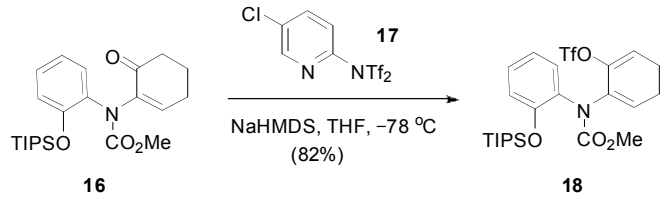
Enone 15. A mixture of **13**[1] (19.1 g, 71.4 mmol), enone **14**[2] (15.6 g, 86.1 mmol), benzene (180 mL), and *p*-toluenesulfonic acid mono hydrate (14.9 g, 78.3 mmol) was heated at 50 °C under Ar for 2 h, during which time the solution became homogeneous. The mixture was cooled to rt, diluted with EtOAc (100 mL), and washed with saturated aqueous $NaHCO_3$ (2 × 100 mL). The separated organic layer was washed with brine (100 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% EtOAc/hexanes) gave **15** (24.4 g, 67.8 mmol, 95%) as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 (dd, J = 8.0, 1.5

Hz, 1H), 6.99 (s, 1H), 6.89 (ddd, J = 15.0, 8.0, 1.5 Hz, 2H), 6.74 (ddd, J = 8.0, 8.0, 1.5 Hz, 1H), 6.46 (t, J = 5.0 Hz, 1H), 2.58 (t, J = 6.0 Hz, 2H), 2.49 (q, J = 6.0 Hz, 2H), 2.03 (dddd, J = 6.0, 6.0, 6.0, 6.0 Hz, 2H), 1.35 (m, 3H), 1.14 (d, J = 7.5 Hz, 18 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.6, 145.5, 136.1, 133.6, 121.2, 120.1, 118.1, 116.6, 116.1, 38.0, 25.0, 23.1, 18.2, 13.2; IR (neat) 3377, 2944, 2867, 1679, 1596, 1522, 1476 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{33}NO_2SiH$ [M + H]: 359.2281. Found: 359.2278.



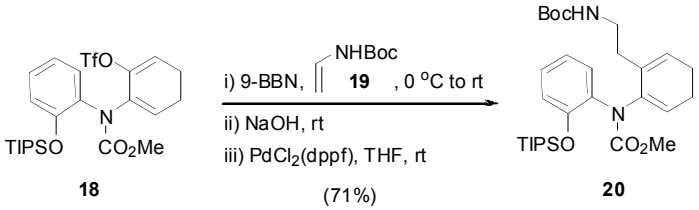
15 **16**

Carbamate 16. *n*-BuLi (2.48 M in hexanes, 26.9 mL, 66.7 mmol) was added dropwise over 10 min to a stirring solution of HMDS (13.9 mL, 66.7 mmol) in THF (90 mL) at –78 °C under Ar. Following warming to rt over 30 min, the solution was added dropwise *via* cannula over 1 h to a stirring solution of **15** (8.0 g, 22.3 mmol), methyl cyanoformate (6.90 mL, 89.0 mmol) and THF (70 mL) at –78 °C under Ar. After 15 min, saturated aqueous NH_4Cl (20 mL) was added. The resultant mixture was warmed to rt and diluted with EtOAc (100 mL). The separated aqueous phase was washed with EtOAc (3 × 100 mL) and the combined organic phase was washed with brine (100 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 15% EtOAc/hexanes) gave **16** (8.3 g, 19.9 mmol, 89%) as a cream solid: mp 110–112 °C; ^{1}H NMR (500 MHz, C_6D_6, 60 °C)[3] δ 7.54 (d, J = 7.5 Hz, 1H), 6.94 (ddd, J = 9.0, 9.0, 1.5 Hz, 1H), 6.85 (m, 2H), 6.74 (ddd, J = 8.5, 8.5, 1.0 Hz, 1H), 3.48 (s, 3H), 2.24 (br s, 2H), 1.75 (br s, 2H), 1.41 (br s, 2H), 1.30 (m, 3H), 1.15 (d, J = 7.5 Hz, 18 H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 193.6, 155.4, 153.1, 143.4, 142.4, 134.8, 130.8, 128.7, 122.0, 119.3, 52.9, 39.0, 25.9, 23.1, 18.6, 13.8; IR (neat) 2946, 2867, 1721, 1692, 1596, 1499, 1439, 1333 cm^{-1}; HRMS (CI) calcd for $C_{23}H_{35}NO_4SiH$ [M + H]: 418.2414. Found: 418.2400.



16 18

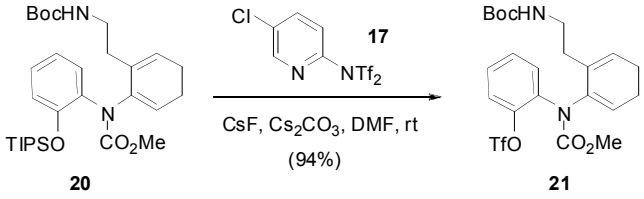
Triflate 18. NaHMDS (1.0 M in THF, 48.3 mL, 48.3 mmol) was added dropwise to a solution of **16** (13.5 g, 32.3 mmol), 2-[*N*,*N*-bis(trifluoromethylsulfonyl)amino]-5-chloropyridine (**17**) (19.0 g, 48.3 mmol), and THF (300 mL) at –78 °C under Ar. After 15 min, saturated aqueous $NaHCO_3$ (75 mL) was added, the mixture was warmed to rt, and extracted with EtOAc (200 mL). The separated aqueous phase was washed with additional EtOAc (100 mL) and the combined organic phases were washed with brine (150 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **18** (14.5 g, 26.4 mmol, 82%) as a pale yellow solid: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.50 (d, *J* = 7.5 Hz, 1H), 6.94 (ddd, *J* = 8.0, 8.0, 1.5 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.74 (t, *J* = 7.5 Hz, 1H), 5.62 (m, 2H), 3.50 (s, 3H), 1.70 (m, 4H), 1.24 (m, 3H), 1.10 (d, *J* = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 152.9, 143.6, 130.0, 128.7, 128.3, 128.0, 127.7, 121.9, 119.7 (q, $J_{C,F}$ = 319 Hz), 119.0, 116.4, 53.3, 22.1, 22.0, 18.7, 13.9; IR (neat) 2948, 2869, 1729, 1598, 1499, 1420, 1320 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{34}F_3NO_6SSiH$ [M + H]: 550.1907. Found: 550.1890.



18 20

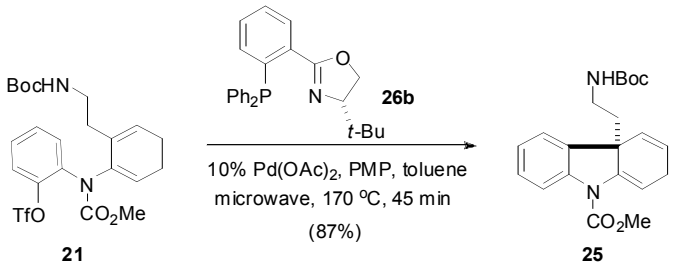
Biscarbamate 20. 9-BBN (0.5 M in THF, 32 mL, 16 mmol) was added dropwise over 15 min to a solution of *N*-vinyl-*tert*-butyl carbamate (**19**)[4] (2.1 g, 14.6 mmol) in THF (45 mL) at 0 °C under Ar. The reaction was allowed to warm to rt over a period of 1 h, then maintained at rt for an additional 15 h. An aqueous solution of NaOH (15 mL of a 3.0 M solution, 45 mmol) was added, and the resulting mixture was degassed by sparging with Ar through a submerged needle for 45 minutes. The mixture was transferred via cannula to a degassed solution of **18** (4.0 g, 7.3 mmol), $PdCl_2$(dppf) (715

mg, 0.87 mmol), and THF (45 mL) at rt. After 40 min, the reaction mixture was cooled to 0 °C, and pH 7 buffer solution (50 mL) was added followed by slow addition of 30% aqueous H_2O_2 (50 mL). When gas evolution slowed, the mixture was warmed to room temperature. After an additional 30 min at rt, saturated aqueous NH_4Cl (50 mL) was added, the mixture was extracted with EtOAc (2 × 75 mL), and the combined organic extracts were washed with brine (75 mL), dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% EtOAc/hexanes) gave **20** (2.8 g, 5.2 mmol, 71%) as a colorless foam: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.21(d, *J* = 7.5 Hz, 1H), 6.94 (t, *J* = 8.0 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 6.76 (t, *J* = 7.5 Hz, 1H), 5.78 (s, 1H), 5.56 (s, 1H), 3.51 (s, 3H), 3.36 (s, 2H), 2.41 (s, 2H), 1.85 (s, 4H), 1.45 (s, 9H), 1.28 (m, 3H), 1.14 (d, *J* = 7.0 Hz, 18H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 156.3, 152.8, 134.4, 129.2, 128.9, 128.7, 128.5, 128.3, 125.0, 122.8, 121.7, 119.2, 78.8, 52.9, 40.8, 31.7, 29.0, 23.1, 22.7, 18.7, 13.9; IR (neat) 3363, 2946, 2869, 1715, 1499, 1441, 1335 cm^{-1}; HRMS (CI) calcd for $C_{30}H_{48}N_2O_5Si$: 544.3333. Found: 544.3326.

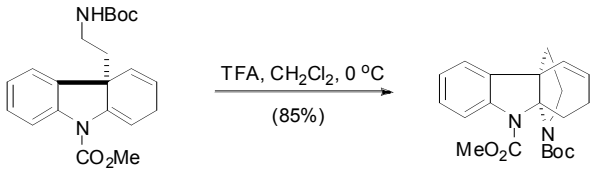


Triflate 21. CsF (13.8 g, 90.8 mmol) was added to a stirring solution of **20** (9.9 g, 18.2 mmol) and 2-[*N*,*N*-bis(trifluoromethylsulfonyl)amino]-5-chloropyridine (**17**) (14.3 g, 36.2 mmol) in anhydrous DMF (150 mL) under Ar.[5] Following stirring for 3 min, crushed cesium carbonate (24.0 g, 73.6 mmol) was added and the resultant suspension was stirred vigorously for 15 min. Saturated aqueous NH_4Cl (100 mL) was added, the mixture was diluted with Et_2O (100 mL), and the separated aqueous phase was extracted with Et_2O (3 × 100 mL). The combined organic extracts were washed with brine (3 × 100 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (15% to 25% EtOAc/hexanes) gave **18** (8.90 g, 17.1 mmol, 94%) as a colorless foam: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.18–7.13 (m, 2H), 6.84 (ddd, *J* = 7.5, 7.5, 1.5 Hz, 1H), 6.70 (ddd, *J* = 8.5, 8.5, 1.5 Hz, 1H),

5.76 (t, J = 4.5 Hz, 1H), 5.54 (m, 1H), 4.42 (s, 1H), 3.54 (s, 3H), 3.28 (q, J = 6.5 Hz, 2H), 2.27 (t, J = 7.0 Hz, 2H), 1.83 (m, 4H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 156.3, 155.1, 145.7, 140.3, 136.3, 133.6, 129.0, 128.9, 128.0, 126.0, 125.9, 122.2, 119.6 (q, $J_{C,F}$ = 318 Hz), 78.9, 53.6, 40.6, 31.8, 28.9, 23.1, 22.5; IR (neat) 3436, 3363, 3037, 2977, 1713, 1675, 1493, 1422, 1329 cm^{-1}; HRMS (CI) calcd for $C_{22}H_{27}F_3N_2O_7Si$: 520.1491. Found: 520.1483.



Dihydrocarbazole 25. Toluene was sparged with Ar for 30 min prior to use in this reaction. A microwave reaction tube containing $Pd(OAc)_2$ (5 mg, 23 μmol) and (*S*)-4-*tert*-butyl-2-[2-(diphenylphosphinyl)phenyl]-4,5-dihydrooxazole (**26b**)[6] (27 mg, 69 μmol) under Ar was charged with a solution of **21** (120 mg, 0.23 mmol), toluene (1.5 mL), and 1,2,2,6,6-pentamethylpiperidine (170 μL, 0.92 mmol). After stirring at rt for 10 min, the reaction mixture was microwave-heated (CEM Discover System, 60 Hz and 300 W instrument) at 170 °C for 45 min. Following cooling, the reaction mixture was concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **25** (74 mg, 0.20 mmol, 87%) as a colorless foam: $[\alpha]_{589}$ +94.1, $[\alpha]_{577}$ +98.9, $[\alpha]_{546}$ +114, $[\alpha]_{435}$ +229 (*c* 3.7, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.00 (d, J = 8.0 Hz, 1H), 7.08 (ddd, J = 8.0, 8.0, 1.5 Hz, 1H), 6.98 (d, J = 7.5 Hz, 1H), 6.89 (t, J = 7.5 Hz, 1H), 6.15 (d, J = 5.5 Hz, 1H), 6.00 (dd, J = 9.5, 3.0 Hz, 1H), 5.71 (dd, J = 9.0, 4.5 Hz, 1H), 3.90 (br s, 1H), 3.52 (s, 3H), 2.95 (m, 2H), 2.61 (dddd, J = 22.0, 2.0, 2.0, 2.0 Hz, 1H), 2.48 (ddd, J = 22.0, 5.5, 5.5 Hz, 1H), 1.50 (t, J = 7.5 Hz, 2H), 1.39 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 155.9, 153.5, 143.9, 141.8, 135.9, 129.8, 128.9, 127.3, 124.0, 122.4, 116.5, 108.1, 78.8, 52.7, 47.4, 44.2, 37.4, 28.9, 27.6; IR (neat) 3375, 2974, 1715, 1514, 1475 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{26}N_2O_4$: 370.1893. Found: 370.1886.

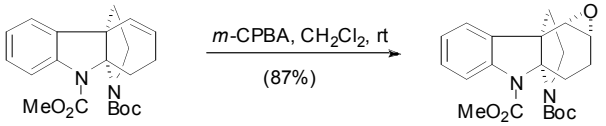


25 **27**

Iminoethanocarbazole 27. Trifluoroacetic acid (0.16 mL, 2.1 mmol) was added to a stirring solution of **25** (260 mg, 0.70 mmol) and CH_2Cl_2 (8 mL) at 0 ˚C under Ar. After 30 min, saturated aqueous $NaHCO_3$ (10 mL) was added. The separated aqueous phase was washed with CH_2Cl_2 (15 mL), and the combined organic fractions were washed with brine (15 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **27** (220 mg, 0.59 mmol, 85%) as a colorless foam: $[\alpha]_{589}$ –41.8, $[\alpha]_{577}$ –44.1, $[\alpha]_{546}$ –49.8, $[\alpha]_{435}$ –86.6, $[\alpha]_{405}$ –110 (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.13 (d, *J* = 8.0 Hz, 1H), 7.09 (ddd, *J* = 7.5, 7.5, 1.5 Hz, 1H), 6.90 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.86 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 5.54 (ddd, *J* = 10.0, 4.0, 4.0 Hz, 1H), 5.47 (ddd, *J* = 10.0, 2.0, 2.0 Hz, 1H), 3.65 (s, 3H), 3.47 (m, 1H), 3.11 (ddd, *J* = 10.5, 9.0, 7.5 Hz, 1H), 3.03 (ddd, *J* = 13.5, 5.5, 5.5 Hz, 1H), 2.40 (ddd, *J* = 13.5, 8.0, 5.0 Hz, 1H), 2.11 (m, 1H), 2.02 (m, 1H), 1.85 (ddd, *J* = 12.5, 7.5, 3.5 Hz, 1H), 1.67 (ddd, *J* = 12.5, 8.5, 8.5 Hz, 1H), 1.43 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 155.1, 153.8, 142.6, 135.3, 128.9, 128.7, 127.1, 123.6, 122.6, 117.8, 99.4, 79.7, 57.0, 52.3, 47.1, 35.5, 29.0, 28.8, 23.5; IR (neat) 2975, 1706, 1480, 1439, 1378 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{26}N_2O_4$: 370.1893. Found: 370.1895. HPLC (Daicel Chiracel OD-H column, column temperature 23 °C, *n*-hexane/*i*-propanol = 98:2, flow rate 1.0 mL·min^{-1}): 6.7 min (minor enantiomer), 14.1 min (major enantiomer), 99% ee. See page S47 for chiral HPLC traces

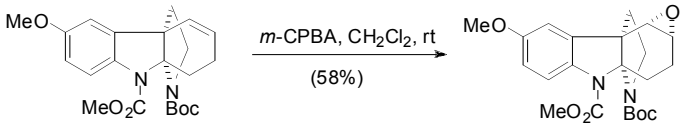
Generation of 27 by a Cascade Sequence: A solution of triflate **21** (96 mg, 0.19 mmol) and toluene (1.2 ml) in a 10 mL microwave reaction vessel was sparged with Ar for 10 min. To this solution was added $Pd(OAc)_2$ (6.4 mg, 0.028 mmol), (*S*)-4-*tert*-butyl-2-[2-(diphenylphosphinyl)phenyl]-4,5-dihydrooxazole (**26b**)[6] (33 mg, 0.084 mmol), and 1,2,2,6,6-pentamethylpiperidine (105 μl, 0.57 mmol). The reaction was then degassed for an additional 20 min. This solution was subsequently heated with stirring in a

microwave reactor (CEM Discover System, 60 Hz and 300 W instrument) at 170 °C for 30 min. The reaction was then cooled to 0 °C, and to it was added a 0 °C solution of trifluoroacetic acid (0.17 ml, 2.28 mmol) in CH_2Cl_2 (2 ml). After stirring for 15 min at this temperature, the reaction was poured into saturated aqueous $NaHCO_3$. The isolated organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (15% EtOAc/hexanes) gave tetracycle **27** (51 mg, 0.14 mmol, 75%) as a colorless oil: $[\alpha]_{589}$ –41.8.



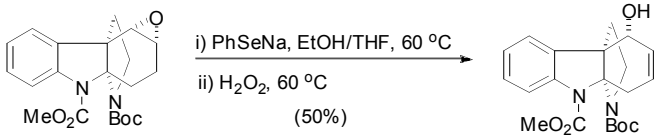
27 **34**

Epoxide 34. *m*-chloroperoxybenzoic acid (400 mg of 70% purity reagent, 1.6 mmol) was added to a stirring solution of **27** (240 mg, 0.65 mmol) in CH_2Cl_2 (7 mL) at 0 °C and the reaction mixture was slowly warmed to rt over 30 min. After 2 h, the reaction was cooled to 0 °C, and another portion of *m*-chloroperoxybenzoic acid (400 mg of 70% purity reagent, 1.6 mmol) was added. The reaction mixture was allowed to warm to rt over 30 min. After 2.5 h, the reaction was diluted with H_2O (10 mL) and Et_2O (20 mL). The separated organic phase was washed with 1 M NaOH (3 × 10 mL) and brine (10 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **34** (217 mg, 0.56 mmol, 87%) as a colorless crystalline solid: mp 178–180 °C; $[\alpha]_{589}$ –79.3, $[\alpha]_{577}$ –82.8, $[\alpha]_{546}$ –94.6, $[\alpha]_{435}$ –166, $[\alpha]_{405}$ –209 (*c* 2.1, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.14 (d, *J* = 8.0 Hz, 1H), 7.08 (m, 1H), 6.86 (m, 2H), 3.70 (s, 3H), 3.54 (ddd, *J* = 14.0, 3.5, 3.5 Hz, 1H), 3.36 (t, *J* = 9.0 Hz, 1H), 2.85 (ddd, *J* = 12.0, 9.5, 6.5 Hz, 1H), 2.80 (d, *J* = 3.5 Hz, 1H), 2.78 (m, 1H), 2.33 (ddd, *J* = 12.0, 12.0, 8.5 Hz, 1H), 1.90−1.83 (m, 3H), 1.73 (ddd, *J* = 13.5, 11.0, 5.5 Hz, 1H), 1.34 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 155.6, 153.6, 145.1, 132.2, 129.8, 128.9, 123.8, 123.3, 117.8, 86.9, 79.9, 56.5, 55.2, 53.5, 52.5, 45.8, 30.2, 28.9, 24.6, 22.5; IR (neat) 2977, 1702, 1602, 1478, 1437, 1382, 1358 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{26}N_2O_5$: 386.1842. Found: 386.1842.



33 **35**

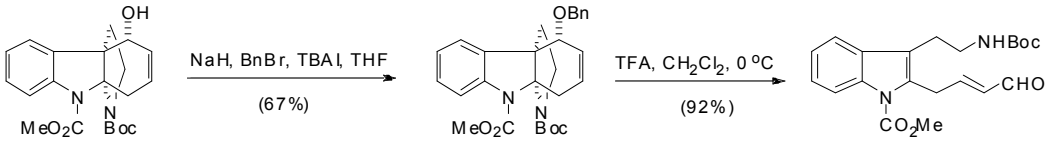
Epoxide 35. *m*-chloroperoxybenzoic acid (215 mg of 70% purity reagent, 0.88 mmol) was added to a stirred solution of racemic **33** (140 mg, 0.35 mmol) in CH_2Cl_2 (4 mL) at 0 °C, and the reaction mixture was allowed to warm to rt. After 1 h at rt, the reaction was re-cooled to 0 °C, and another portion of *m*-chloroperoxybenzoic acid (140 mg of 70% purity reagent, 0.88 mmol) was added. The reaction mixture was allowed to warm to rt and the reaction was stirred for 1 h at rt. The reaction was then diluted with H_2O (10 mL) and Et_2O (20 mL). The separated organic phase was washed with 1 M NaOH (3 × 10 mL) and brine (10 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **35** (85 mg, 0.14 mmol, 58%) as a colorless crystalline solid[7]: mp 185–187 °C; ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.08 (d, J = 8.8 Hz, 1H), 6.65−6.63 (m, 2H), 3.71 (s, 3H), 3.55 (dt, J = 13.9, 3.2 Hz, 1H), 3.36 (br s, 4H), 2.91−2.85 (m, 1H), 2.80−2.78 (m, 2H), 2.33−2.26 (m, 1H), 1.92−1.82 (m, 2H), 1.72 (ddd, J = 16.8, 11.9, 4.9 Hz, 1H), 1.36 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 157.5, 155.1, 153.8, 138.8, 133.9, 118.9, 114.5, 110.3, 87.2, 80.1, 56.6, 55.9, 55.5, 53.7, 52.6, 45.9, 30.3, 29.1, 24.8, 22.8; IR (neat) 2949, 1698, 1486, 1436, 1382, 1359 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}N_2O_6Na$: 439.1845. Found: 439.1849.



34 **36**

Allylic Alcohol 36. Following a general procedure,[8] a bulk solution of NaSePh was prepared by addition of two portions of $NaBH_4$ (12 mg, 0.32 mmol each) to a stirring yellow suspension of Ph_2Se_2 (50 mg, 0.16 mmol) in absolute ethanol (1.5 mL, degassed by sparging with Ar for 30 min prior to use) at 0 °C. The reaction mixture was warmed to rt over 20 min and maintained at rt for 20 min until the solution became colorless, indicating complete reduction of Ph_2Se_2. A solution of **34** (22 mg, 57 μmol) and THF

(0.5 mL, degassed by sparging with Ar for 30 min prior to use) under Ar at rt was charged with a portion of the bulk NaSePh solution (0.5 mL, 50 mmol). The reaction mixture was heated to 60 °C and maintained at this temperature for 2 h. An additional portion of NaSePh solution (0.25 mL, 25 mmol) was added. Two additional portions of $NaBH_4$ (5 mg each) were added at 2 h intervals over the next 4 h to maintain a colorless reaction mixture and reduce any Ph_2Se_2 that formed during the course of the reaction. The reaction mixture was cooled to 0 °C, and 30% H_2O_2 (0.5 mL) was added dropwise. After 20 min, the reaction mixture was warmed to rt and maintained at this temperature for ca. 30 min, until gas evolution ceased. TLC analysis showed complete consumption of the alkylselenide intermediate. The reaction mixture was heated at 60 °C for 1.5 h. The reaction mixture was cooled to rt and diluted with H_2O (1 mL) and Et_2O (3 mL). The separated aqueous layer was extracted with Et_2O (3 × 3 mL). The combined organic fractions were washed with saturated aqueous $NaHCO_3$ (3 × 3 mL), brine (5 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **36** (11 mg, 28 mmol, 50%) as a colorless foam. **Note**: See preparation of **44** for an improved procedure. $[\alpha]_{589}$ –98.6, $[\alpha]_{577}$ –104, $[\alpha]_{546}$ –118, $[\alpha]_{435}$ –206, $[\alpha]_{405}$ –259 (*c* 1.1, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.22 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 7.0 Hz, 1H), 7.14 (dd, *J* = 8.0, 7.5 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 5.82 (m, 1H), 5.59 (ddd, *J* = 9.5, 2.5, 2.5 Hz, 1H), 4.28 (dd, *J* = 15.0, 6.5 Hz, 1H), 3.99 (s, 1H), 3.75 (dd, *J* = 11.5, 7.5 Hz, 1H), 3.63 (s, 3H), 2.83 (ddd, *J* = 11.5, 11.5, 4.5 Hz, 1H), 2.37 (m, 1H), 1.95 (ddd, *J* = 12.0, 12.0, 7.5 Hz, 1H), 1.70 (dd, *J* = 12.0, 5.0 Hz, 1H), 1.42 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 153.8, 152.4, 143.6, 134.6, 133.7, 128.8, 126.6, 124.1, 123.4, 115.7, 91.1, 79.3, 73.2, 64.5, 51.9, 46.2, 34.3, 31.3, 28.5; IR (neat) 3464, 2977, 2854, 1711, 1690, 1484, 1389 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{26}N_2O_5Na$ [M + Na]: 409.1740. Found: 409.1736.

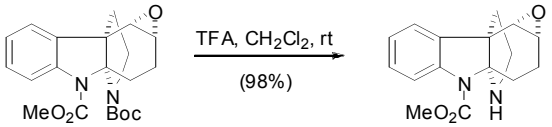


36 **37** **38**

Indole 38: NaH (60% dispersion in mineral oil, 8 mg, 0.22 mmol) was added to a stirring solution of alcohol **36** (17 mg, 0.044 mmol) and THF (0.5 mL) at 0 °C under Ar. The reaction was allowed to warm to rt over 10 min and then re-cooled to 0 °C. To this was added benzyl bromide (13 μl, 0.11 mmol) and tetrabutylammonium iodide (6 mg, 0.016 mmol), and the reaction was warmed to rt and stirred for 3 h. The reaction was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The combined organic extract was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (15% hexanes/Et_2O) gave **37** (14 mg, 0.030 mmol, 67%) as a clear, colorless oil: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.25 (d, *J* = 8.1 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.21−7.09 (m, 6H), 6.89 (t, *J* = 7.6 Hz, 1H), 5.97−5.92 (m, 1H), 5.84 (dt, *J* = 9.5, 2.4 Hz, 1H), 4.44 (d, *J* = 11.7 Hz, 1H), 4.38 (dd, *J* = 14.9, 6.6 Hz, 1H), 4.29 (d, *J* = 11.7 Hz, 1H), 3.96 (br s, 1H), 3.79 (dd, *J* = 11.2, 7.6, 1H), 3.65 (s, 3H), 2.87−2.82 (m, 1H), 2.49 (d, *J* = 15.2 Hz, 1H), 2.22−2.15 (m, 1H), 1.82 (dd, *J* = 12.5, 5.1 Hz, 1H), 1.43 (s, 9H).

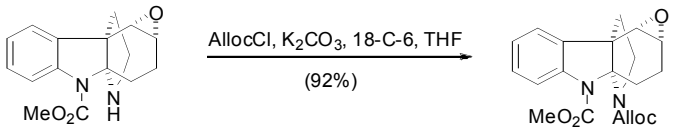
Trifluoroacetic acid (25 μL, 20 eq) was added to a stirred solution of benzyl ether **38** (8 mg, 0.017 mmol) in CH_2Cl_2 (0.5 mL) at 0 °C. Following stirring for 10 min at 0 °C, saturated aqueous $NaHCO_3$ was added. The aqueous phase was then extracted with EtOAc, and the combined organic extract was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (20% to 35% EtOAc/hexanes) gave **38** (6 mg, 0.016 mmol, 92%) as a clear, colorless oil. ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 9.33 (d, *J* = 7.5 Hz, 1H), 8.08 (d, *J* = 8.3 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.18 (dt, *J* = 1.3, 7.2 Hz, 1H), 7.10–7.14 (m, 1H), 6.42 (dt, *J* = 6.0, 15.7 Hz, 1H), 5.96 (ddt, *J* = 1.6, 7.5, 15.7 Hz, 1H), 3.97 (br s, 1H), 3.65 (dd, *J* = 1.5, 6.0 Hz, 2H), 3.33 (s, 3H), 3.01 (q, *J* = 6.7 Hz, 2H), 2.54 (t, *J* = 7.0 Hz, 2H), 1.38 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 191.8, 153.4, 151.7, 136.0, 133.1, 132.6, 129.8, 128.2, 124.4, 123.0, 118.6, 118.2, 115.9, 78.5, 52.5, 40.5, 29.4, 28.1, 24.6;

IR (neat) 3382, 1735, 1688 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{26}N_2O_5Na$ [M + Na]: 409.1740. Found: 409.1743.



34 **42**

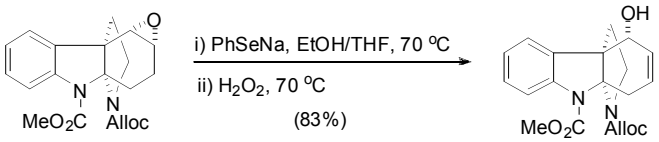
Epoxide 42. Trifluoroacetic acid (3.2 mL) was added to a stirring solution of **34** (500 mg, 1.29 mmol) in CH_2Cl_2 (16 mL) at 0 °C under Ar. The cooling bath was removed, and the reaction mixture was allowed to warm to rt for 2.5 h. The reaction mixture then was cooled to 0 ˚C and saturated aqueous $NaHCO_3$ (40 mL) was added dropwise. The mixture was diluted with EtOAc (100 mL), and the separated organic phase was washed with brine (50 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (50% EtOAc/hexanes) gave **42** (360 mg, 1.26 mmol, 98%) as a colorless oil: $[\alpha]_{589}$ –146, $[\alpha]_{577}$ –153, $[\alpha]_{546}$ –176, $[\alpha]_{435}$ –330, $[\alpha]_{405}$ –431 (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.94 (d, *J* = 7.0 Hz, 1H), 7.10 (ddd, *J* = 6.5, 6.5, 2.0 Hz, 1H), 6.88 (m, 2H), 3.48 (s, 3H), 3.08 (d, *J* = 4.0 Hz, 1H), 2.77 (t, *J* = 8.5 Hz, 1H), 2.71 (m, 1H), 2.68 (ddd, *J* = 7.0, 7.0, 2.0 Hz, 1H), 2.61 (br s, 1H), 2.50 (ddd, *J* = 10.0, 9.0, 5.0 Hz, 1H), 2.39 (ddd, *J* = 12.5, 11.5, 7.0 Hz, 1H), 2.02 (ddd, *J* = 13.5, 13.5, 4.0 Hz, 1H), 1.84 (ddd, *J* = 11.5, 5.0, 2.0 Hz, 1H), 1.64 (dddd, *J* = 15.0, 4.0, 4.0, 4.0 Hz, 1H), 1.37 (dddd, *J* = 13.5, 13.5, 3.5, 1.0 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.2, 144.3, 133.7, 129.2, 124.2, 123.6, 115.8, 88.8, 57.6, 54.4, 53.2, 52.2, 43.3, 38.0, 27.8, 21.4; IR (neat) 3377, 2958, 2850, 1698, 1598, 1484, 1439, 1374 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{19}N_2O_3$ [M + H]: 287.1396. Found: 287.1396.



42 **43**

Epoxide 43. Allyl chloroformate (3.40 mL, 31.1 mmol) was added to a stirring solution of aminal **42** (2.96 g, 10.3 mmol) in THF (125 mL) at 0 °C. K_2CO_3 (4.30 g, 31.1

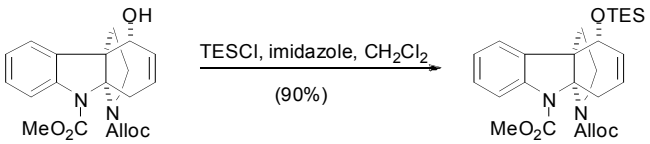
mmol) was added, followed by 18-crown-6 (300 mg, 1.04 mmol). The reaction was warmed to rt over 2 h, stirred for an additional 18 h, and then quenched with saturated aqueous NH_4Cl (50 mL). The aqueous layer was extracted with Et_2O, and the combined organic fractions were washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (20% EtOAc/hexanes) gave epoxide **43** (3.52 g, 9.5 mmol, 92%) as a thick, clear oil: $[\alpha]_{589}$ –81.1, $[\alpha]_{577}$ –84.4, $[\alpha]_{546}$ –96.4, $[\alpha]_{435}$ –167, $[\alpha]_{405}$ –208 (*c* 2.10, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.11 (d, *J* = 8.2 Hz, 1H), 7.07−7.10 (m, 1H), 6.86−6.92 (m, 2H), 5.65−5.71 (m, 1H), 5.00 (dd, *J* = 17.2, 1.6 Hz, 1H), 4.90 (dd, *J* = 10.5, 1.4 Hz, 1H), 4.41−4.45 (m, 1H), 4.30 (dd, *J* = 13.4, 5.4 Hz, 1H), 3.69 (s, 3H), 3.42 (ddd, *J* = 14.1, 3.3, 3.3 Hz, 1H), 3.37 (d, *J* = 9.3 Hz, 1H), 2.85 (ddd, *J* = 12.0, 10.5, 6.5 Hz, 1H), 2.79 (s, 2H), 2.31 (ddd, *J* = 12.6, 12.6, 8.5 Hz, 1H), 1.93 (dd, *J* = 12.7, 6.4 Hz, 1H), 1.81−1.83 (m, 2H), 1.63−1.68 (m, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.6, 153.4, 144.5, 133.6, 131.6, 129.5, 123.6, 123.0, 117.5, 116.9, 86.6, 65.6, 56.0, 54.8, 53.1, 52.2, 45.1, 29.7, 24.1, 22.1; IR (neat) 1698 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{22}O_5N_2Na$ [M + Na]: 393.1426. Found: 393.1415.



43 **44**

Allyl alcohol 44. Following a general procedure,[8] a mixture of PhSeSePh (1.71 g, 5.48 mmol) and absolute EtOH (20 mL) was stirred at 0 °C. Solid $NaBH_4$ (420 mg, 11.0 mmol) was added in portions to this mixture over a period of 3 min (rapid gas evolution resulted). The originally yellow mixture turned to a clear, colorless solution upon complete addition of $NaBH_4$. The resulting sodium phenyl selenide solution was warmed to rt and stirred for 5 min. To this solution was added epoxide **43** (1.84 g, 4.97 mmol) in 1:1 THF/EtOH (20 mL), and the reaction was heated to 70 °C for 2 h. To reduce any PhSeSePh formed during the course of the reaction, which resulted in yellowing of the reaction, ca. 10 mg portions of $NaBH_4$ were added twice. The reaction was then cooled to 0 °C, diluted with THF (15 mL), and 30% H_2O_2 (11 mL) was added slowly. The

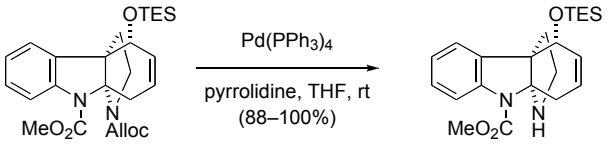
reaction was again heated to 70 °C for 20 min during which time rapid gas evolution was noticed. The reaction then was poured into saturated $NaHCO_3$, extracted with CH_2Cl_2, and the combined organic layers were dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (20% EtOAc/hexanes) gave allyl alcohol **44** (1.54 g, 4.15 mmol, 83%) as clear, colorless oil: $[\alpha]_{589}$ –126, $[\alpha]_{577}$ –132, $[\alpha]_{546}$ –150, $[\alpha]_{435}$ –258, $[\alpha]_{405}$ –320 (*c* 1.30, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.15 (d, *J* = 8.2 Hz, 1H), 7.44 (dd, *J* = 1.1, 7.5 Hz, 1H), 7.11−7.15 (m, 1H), 6.91 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 5.80−5.83 (m, 1H), 5.72−5.80 (m, 2H), 5.09 (ddd, *J* = 17.2, 1.6, 1.6 Hz, 1H), 4.95 (dd, *J* = 10.5, 1.4 Hz, 1H), 4.41−4.51 (m, 2H), 4.23 (dd, *J* = 15.2, 6.5 Hz, 1H), 4.13 (d, *J* = 1.7 Hz, 1H), 3.77 (dd, *J* = 11.1, 7.3 Hz, 1H), 3.61 (s, 3H), 2.85 (ddd, *J* = 11.3, 11.3, 5.5 Hz, 1H), 2.39−2.42 (m, 2H), 2.05 (ddd, *J* = 12.4, 12.4, 7.9 Hz, 1H), 1.79 (dd, *J* = 12.4, 5.3 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 153.8, 153.1, 143.4, 134.6, 134.2, 133.7, 128.8, 126.2, 124.3, 123.6, 117.0, 115.7, 91.3, 73.1, 65.7, 64.4, 52.0, 46.1, 34.4, 31.2; IR (neat) 3462, 1690, 1728 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{22}O_5N_2Na$ [M + Na]: 393.1426. Found: 393.1415.



44 **45**

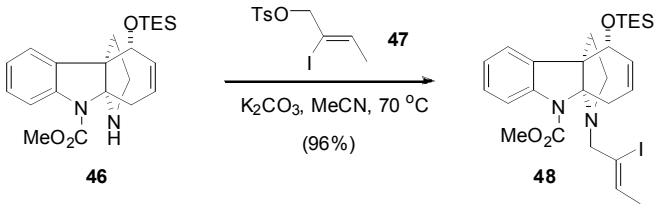
Silyl Ether 45. TESCl (1.64 mL, 9.73 mmol) was added to a stirred solution of allyl alcohol **44** (2.40 g, 6.48 mmol) and imidazole (660 mg, 9.73 mmol) in CH_2Cl_2 (60 mL) at rt. TLC analysis after 20 min indicated complete consumption of the starting alcohol, and the reaction was then quenched with H_2O (50 mL). The aqueous layer was extracted with CH_2Cl_2, and the combined organic layers were washed with saturated aqueous NH_4Cl, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% EtOAc/hexanes) gave silylated alcohol **45** (2.82 g, 5.83 mmol, 90%) as a colorless amorphous solid: $[\alpha]_{589}$ −132, $[\alpha]_{577}$ –138, $[\alpha]_{546}$ –157, $[\alpha]_{435}$ –272, $[\alpha]_{405}$ –339 (*c* 1.13, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.23 (d, *J* = 8.2 Hz, 1H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.13−7.15 (m, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 5.83−5.86 (m, 1H), 5.74−5.82 (m, 2H), 5.10 (dd, *J* = 17.2,

1.6 Hz, 1H), 4.95 (dd, *J* = 10.5, 1.4 Hz, 1H), 4.44−4.53 (m, 2H), 4.38 (s, 1H), 4.25 (dd, *J* = 15.3, 6.4 Hz, 1H), 3.80 (dd, *J* = 11.1, 7.8 Hz, 1H), 3.63 (s, 3H), 2.87 (ddd, *J* = 12.3, 5.4, 4.6 Hz, 1H), 2.55−2.59 (m, 1H), 2.15 (ddd, *J* = 12.4, 7.9, 7.9 Hz, 1H), 1.79 (dd, *J* = 12.4, 5.3 Hz, 1H), 0.94 (t, *J* = 8.0 Hz, 9H), 0.57 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 153.8, 152.9, 143.9, 134.2, 134.0, 133.9, 129.0, 126.7, 124.4, 123.1, 116.8, 115.7, 91.3, 74.2, 65.5, 65.1, 51.9, 46.1, 34.6, 31.3, 7.0, 5.6; IR (neat) 1692, 1727 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{36}O_5N_2SiNa$ [M + Na]: 507.2291. Found: 507.2298.



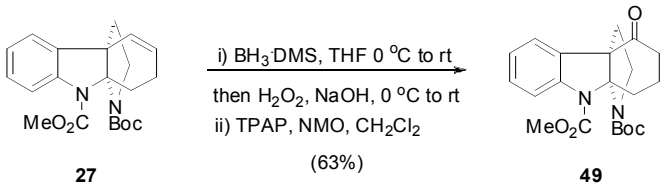
45 **46**

Tetracyclic Amine 46. A solution of **45** (2.50 g, 5.16 mmol), pyrrolidine (8.7 mL, 103 mmol) and THF (50 mL) was degassed with Ar for 30 min and stirred at rt. To this mixture was added $Pd(PPh_3)_4$ (605 mg, 0.52 mmol) in one portion, and stirring was continued for an additional 30 min. The reaction was then concentrated under reduced pressure. Purification of the crude residue by column chromatography (20% to 50% EtOAc/hexanes) gave aminal **46** (2.07 g, 5.16 mmol, 100%) as a thick, light yellow oil: $[\alpha]_{589}$ –78.1, $[\alpha]_{577}$ –82.1, $[\alpha]_{546}$ –94.2, $[\alpha]_{435}$ –171, $[\alpha]_{405}$ –216 (*c* 1.60, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.95 (br s, 1H), 7.57 (d, *J* = 7.4 Hz, 1H), 7.13−7.15 (m, 1H), 6.96 (t, *J* = 7.5 Hz, 1H), 5.85 (ddd, *J* = 9.4, 9.4, 2.7 Hz, 1H), 5.70−5.73 (m, 1H), 4.41 (d, *J* = 2.1 Hz, 1H), 3.51 (s, 3H), 3.28 (br s, 1H), 3.05 (br s, 1H), 2.72 (t, *J* = 8.0 Hz, 1H), 2.53−2.58 (m, 1H), 2.29 (ddd, *J* = 11.8, 11.8, 6.9 Hz, 1H), 2.13 (d, *J* = 14.4 Hz, 1H), 1.93 (dd, *J* = 11.9, 4.7 Hz, 1H), 0.98 (t, *J* = 8.0 Hz, 9H), 0.63 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 153.6, 143.7, 136.1, 134.0, 128.4, 126.8, 125.1, 123.0, 114.7, 92.1, 75.1, 63.0, 51.7, 43.6, 37.9, 34.5, 7.0, 5.7; IR (neat) 3378, 3047, 1691 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{32}O_3N_2SiH$ [M + H]: 401.2260. Found: 401.2253.



Vinyl iodide 48. A mixture of **46** (372 mg, 0.93 mmol), (*Z*)-2-iodo-2-butenyl tosylate (**47**)[9] (660 mg, 1.87 mmol), K_2CO_3 (640 mg, 4.65 mmol) and MeCN (10 mL) was stirred at 70 °C for 18 h. After cooling to rt, the reaction was partitioned between Et_2O and water. The aqueous layer was extracted with additional Et_2O, and the combined organic layers were washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (5% EtOAc/hexanes) gave vinyl iodide **48** (520 mg, 0.89 mmol, 96%) as a clear, colorless oil: $[\alpha]_{589}$ +22.7, $[\alpha]_{577}$ +23.8, $[\alpha]_{546}$ +27.4, $[\alpha]_{435}$ +52.1, $[\alpha]_{405}$ +67.1 (*c* 1.55, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.98 (d, *J* = 8.1 Hz, 1H), 7.57 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.13−7.15 (m, 1H), 6.98 (dt, *J* = 7.5, 1.0 Hz, 1H), 5.84−5.92 (m, 2H), 5.45 (q, *J* = 6.4 Hz, 1H), 4.49 (d, *J* = 14.7 Hz, 1H), 4.41 (d, *J* = 2.1 Hz, 1H), 3.59 (dd, *J* = 15.8, 7.1 Hz, 1H), 3.50 (s, 3H), 3.33 (d, *J* = 14.7 Hz, 1H), 2.69 (t, *J* = 6.9 Hz, 1H), 2.20−2.28 (m, 3H), 1.93−1.96 (m, 1H), 1.59 (dd, *J* = 6.4, 1.8 Hz, 3H), 0.97 (t, *J* = 8.0 Hz, 9H), 0.61 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.5, 144.0, 135.3, 134.1, 130.1, 128.5, 126.8, 125.5, 122.9, 115.9, 112.2, 93.5, 74.4, 65.6, 59.9, 51.8, 48.2, 34.5, 33.1, 21.6, 7.0, 5.6; IR (neat) 1704 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{37}O_3IN_2SiH$ [M + H]: 581.1697. Found: 581.1698.

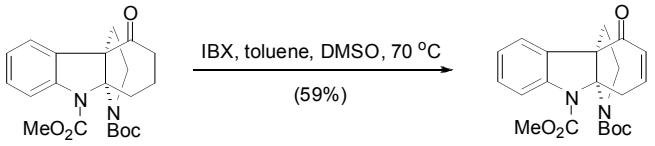
It was observed that extended exposure of **48** to silica gel during chromatography caused partial isomerization to olefin **63** (see below).



Ketone 49. A solution of **27** (575 mg, 1.55 mmol) and THF (25 mL) under Ar was cooled to 0 °C, and BH_3·DMS (0.51 mL, 5.5 mmol) was added dropwise. The reaction mixture was gradually warmed to rt over 0.5 h and maintained at rt for 1 h. TLC

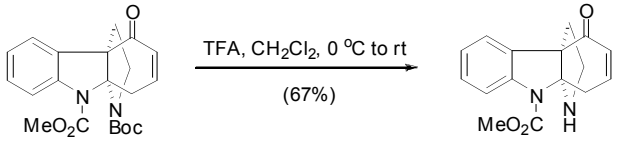
analysis showed incomplete conversion, so the solution was cooled to 0 ˚C and additional BH_3·DMS (0.51 mL, 5.5 mmol) was added. The reaction mixture was gradually warmed to rt over 0.5 h and maintained at rt for 2 h. The reaction mixture was cooled to 0 ˚C, and 3 N NaOH and 30% H_2O_2 (1 mL each) were added sequentially, and the mixture was stirred at 0 ˚C for 30 min and at rt for an additional 30 min. The mixture was diluted with EtOAc (30 mL), and the separated organic phase was washed sequentially with saturated aqueous $NaHCO_3$ (15 mL), H_2O (15 mL), and brine (15 mL). The organic phase was dried (Na_2SO_4), filtered and concentrated under reduced pressure to afford a clear, colorless oil which was used immediately without purification.

A stirring rt solution of this crude residue (1.55 mmol theoretical) in CH_2Cl_2 (25 mL) was charged sequentially with crushed 4 Å molecular sieves (~1 g), TPAP (55 mg, 0.16 mmol), and NMO (360 mg, 3.1 mmol). The reaction mixture was maintained at rt for 20 min, then concentrated to ~5 mL under a stream of N_2. The mixture was filtered through a small SiO_2 plug (20% EtOAc/hexanes) and the filtrate was concentrated under reduced pressure. Purification of the crude residue by column chromatography (10% to 20% EtOAc/hexanes) gave **49** (380 mg, 0.98 mmol, 63%) as a colorless foam (**50** was isolated in 21% yield; see below for optimized procedure for **50**): **49** ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.97 (d, *J* = 8.0 Hz, 1H), 6.92 (ddd, *J* = 7.5, 7.5, 1.5 Hz, 1H), 6.85 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.62 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 3.45 (s, 3H), 3.41 (ddd, *J* = 8.5, 8.5, 3.5 Hz, 1H), 2.82 (ddd, *J* = 11.0, 9.0, 7.5 Hz, 1H), 2.56 (m, 2H), 2.31 (ddd, *J* = 13.0, 9.0, 9.0 Hz, 1H), 1.86 (ddddd, *J* = 17.0, 17.0, 17.0, 7.0, 7.0 Hz, 2H), 1.50 (ddd, *J* = 13.0, 7.5, 3.5 Hz, 1H), 1.30 (m, 2H), 1.26 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 206.7, 153.9, 153.1, 143.7, 130.6, 129.9, 123.9, 123.8, 116.7, 93.3, 80.0, 69.0, 52.4, 46.8, 38.1, 34.1, 32.1, 28.9, 19.4; IR (neat) 2958, 1707, 1483, 1444, 1383 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{26}N_2O_5$: 386.1842. Found: 386.1846.



49 **52**

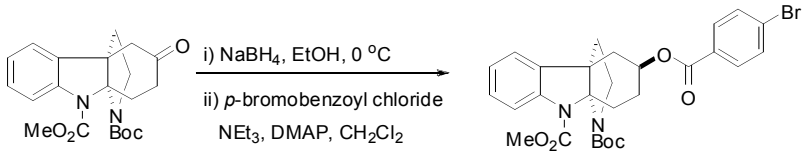
Enone 52. A solution of **49** (200 mg, 0.52 mmol) in toluene (3.5 mL) and DMSO (1.7 mL) under Ar was charged with IBX (580 mg, 2.07 mmol), and the reaction mixture was heated to 70 ˚C.[10] Over the course of a 24 h period, the reaction was charged with two additional portions of IBX (580 mg, 2.07 mmol each). The reaction was cooled to rt and diluted with Et_2O (20 mL). Saturated aqueous $NaHCO_3$ (35 mL) and saturated aqueous $Na_2S_2O_3$ (35 mL) were added, and the mixture was stirred vigorously for 1 h. The separated aqueous phase was washed with ether (30 mL), and the combined organic fractions were washed with brine (25 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% EtOAc/hexanes) gave **52** (119 mg, 0.31 mmol, 59%) as a colorless foam: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.47 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.07 (ddd, *J* = 8.0, 8.0, 1.0 Hz, 1H), 6.85 (t, *J* = 7.5 Hz, 1H), 6.43 (ddd, *J* = 9.5, 4.5, 4.5 Hz, 1H), 5.89 (ddd, *J* = 10.0, 1.5, 1.5 Hz, 1H), 3.55 (s, 3H), 3.37 (m, 3H), 3.05 (ddd, *J* = 11.0, 8.0, 8.0 Hz, 1H), 2.18 (ddd, *J* = 13.0, 8.5, 8.5 Hz, 1H), 1.98 (ddd, *J* = 12.5, 8.0, 4.5 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 195.4, 153.9, 153.6, 147.0, 142.9, 130.2, 129.9, 129.7, 124.6, 124.5, 117.4, 90.5, 80.2, 65.9, 52.4, 47.6, 33.2, 30.4, 28.9; IR (neat) 2979, 1694, 1675, 1478, 1387 cm^{-1}; HRMS (CI) calcd for $C_{21}H_{24}N_2O_5$: 384.1685. Found: 384.1685.



52 **53**

Enone 53. Trifluoroacetic acid (0.75 mL) was added to a stirred solution of **52** (115 mg, 0.30 mmol) in CH_2Cl_2 (4 mL) at 0 ˚C under Ar. The cooling bath was removed, and the reaction mixture was allowed to warm to rt for 2.5 h. The reaction mixture then was cooled to 0 ˚C and saturated aqueous $NaHCO_3$ (10 mL) was added dropwise. The

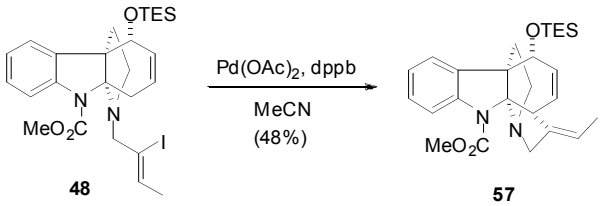
mixture was diluted with EtOAc (30 mL), and the separated organic phase was washed with brine (15 mL), dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% EtOAc/hexanes) gave **53** (58 mg, 0.20 mmol, 67%) as a pale orange foam: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.79 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.77 (br s, 1H), 7.08 (ddd, *J* = 8.5, 8.5, 1.5 Hz, 1H), 6.86 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 6.06 (ddd, *J* = 10.0, 5.5, 3.0 Hz, 1H), 5.93 (ddd, *J* = 10.0, 3.0, 1.5 Hz, 1H), 3.47 (s, 3H), 3.46 (br s, 1H), 2.98 (m, 1H), 2.61 (ddd, *J* = 8.5, 8.5, 2.0 Hz, 1H), 2.53 (ddd, *J* = 10.0, 10.0, 6.5 Hz, 1H), 2.42 (ddd, *J* = 12.5, 10.0, 8.0 Hz, 1H), 2.34 (m, 1H), 2.00 (ddd, *J* = 12.5, 6.5, 2.0 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 196.5, 153.2, 145.4, 131.4, 128.8, 128.3, 127.7, 125.1, 123.8, 114.9, 91.1, 62.0, 51.9, 42.9, 41.8, 35.7; IR (neat) 3365, 2954, 2846, 1692, 1671, 1482 cm^{-1}; HRMS (CI) calcd for $C_{16}H_{16}N_2O_3$: 284.1161. Found: 284.1162.



Proof of Absolute Configuration by Heavy Atom X-Ray Analysis of Benzyl Ester 51. Solid $NaBH_4$ (20 mg, 0.52 mmol) was added in one portion to ketone **50** (100 mg, 0.26 mmol) and EtOH (3 mL) at 0 °C. After 15 min at 0 °C, the reaction was quenched with saturated aqueous NH_4Cl and warmed to rt. The reaction was extracted with CH_2Cl_2, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% to 50% EtOAc/hexanes) gave a 1:1 ratio of cleanly separated diastereomeric alcohols (91 mg combined, 0.24 mmol, 91%). The stereochemistry of each alcohol was not determined at this stage. The alcohols were distinguished based on their R_f values (35% EtOAc/hexanes): lower isomer R_f 0.1; upper isomer R_f 0.2.

DMAP (~1 mg) was added to a stirring solution of the lower R_f diastereomeric alcohol (10 mg, 0.026 mmol) and Et_3N (8 μL, 0.052 mmol) in CH_2Cl_2 (1 mL) at rt. The mixture was then treated with *p*-bromobenzoyl chloride (8.5 mg, 0.039 mmol) and stirred at rt for 4 h. After this time, 1 N NaOH was added to the reaction and stirred for 20 min.

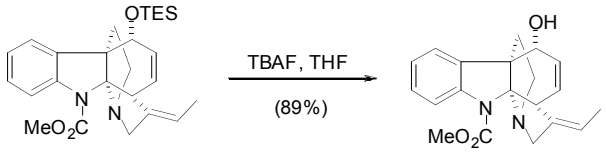
The reaction was extracted with CH_2Cl_2, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by preparative TLC (50% EtOAc/hexanes) gave **51** as a clear, colorless oil (13 mg, 0.023 mmol, 88%) that slowly solidified. Re-crystallization from CH_2Cl_2/hexanes gave X-ray quality crystals[7]: $[\alpha]_{589}$ −90.2, $[\alpha]_{577}$ –93.6, $[\alpha]_{546}$ –106, $[\alpha]_{435}$ –183, $[\alpha]_{405}$ –222 (*c* 0.84, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.07 (d, *J* = 7.8 Hz, 1H), 7.42–7.44 (m, 2H), 7.14–7.16 (m, 2H), 7.00 (dt, *J* = 1.5, 7.7 Hz, 1H), 6.70 (dd, *J* = 1.1, 7.4 Hz, 1H), 6.66 (dt, *J* = 0.9, 7.4 Hz, 1H), 5.02–5.07 (m, 1H), 3.59 (s, 3H), 3.31–3.35 (m, 1H), 3.07 (ddd, *J* = 8.4, 8.4, 9.9 Hz, 1H), 2.80–2.84 (m, 1H), 2.54 (br s, 1H), 1.82 (dd, *J* = 6.8, 14.6 Hz, 1H), 1.65–1.74 (m, 2H), 1.56 (dd, *J* = 3.4, 14.6 Hz, 1H), 1.50 (ddd, *J* = 5.0, 8.4, 13.0 Hz, 1H), 1.42–1.46 (m, 1H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 164.7, 153.9, 153.2, 141.3, 136.1, 131.5, 131.3, 129.8, 128.0, 128.1, 122.9, 121.6, 117.2, 89.3, 79.3, 69.4, 54.3, 51.7, 46.2, 35.1, 34.0, 28.3, 26.9, 26.4; IR (neat) 1708 cm^{-1}; HRMS (ESI) calcd for $C_{28}H_{31}BrN_2O_6Na$ [M + Na]: 593.1263. Found: 593.1262.



48 **57**

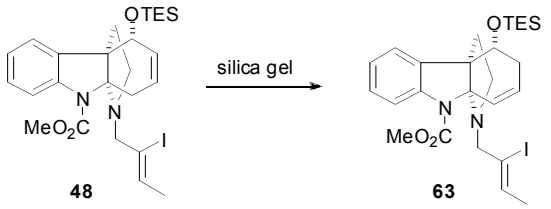
Pentacycle 57. A solution of **48** (40 mg, 0.069 mmol) and MeCN (1.2 mL) was degassed with argon for 15 min. To this solution was added $Pd(OAc)_2$ (8.0 mg, 0.034 mmol) and bis(diphenylphosphoryl)butane (22 mg, 0.053 mmol), and degassing was continued for an additional 10 min. The sealed reaction vial was then heated to 80 °C for 3 h. Following cooling, the mixture was concentrated under pressure. Purification of the crude residue by column chromatography (10% EtOAc/hexanes) gave **57** (15 mg, 0.033 mmol, 48%) as a clear, colorless oil: $[\alpha]_{589}$ +21.6, $[\alpha]_{577}$ +22.6, $[\alpha]_{546}$ +26.4, $[\alpha]_{435}$ +53.8, $[\alpha]_{405}$ +71.6 (*c* 1.80, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.30 (d, *J* = 8.1 Hz, 1H), 7.45 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.17−7.20 (m, 1H), 6.96 (ddd, *J* = 7.4, 7.4, 1.0 Hz, 1H), 5.78 (ddd, *J* = 10.0, 3.4, 1.8 Hz, 1H), 5.39 (ddd, *J* = 10.0, 10.0, 2.4 Hz, 1H), 5.10−5.13 (m, 1H), 4.37 (q, *J* = 1.8 Hz, 1H), 3.73 (d, *J* = 12.5 Hz, 1H), 3.46 (br.s, 4H), 3.15−3.19 (m, 1H), 2.72 (ddd, *J* = 11.9, 11.9, 5.1 Hz, 1H), 2.39 (dd, *J* = 11.7, 6.9 Hz,

1H), 2.17 (ddd, *J* = 12.2, 12.2, 7.2 Hz, 1H), 2.02 (dd, *J* = 12.4, 5.0 Hz, 1H), 1.54−1.56 (m, 3H), 0.94 (t, *J* = 8.0 Hz, 9H), 0.59 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.4, 145.8, 143.6, 134.0, 132.2, 128.8, 128.7, 124.7, 122.3, 115.6, 111.0, 102.1, 73.8, 63.2, 60.8, 53.1, 51.1, 46.4, 33.0, 13.8, 7.0, 5.6; IR (neat) 1694 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{37}O_3N_2Si$ [M + H]: 453.2574. Found: 453.2567.

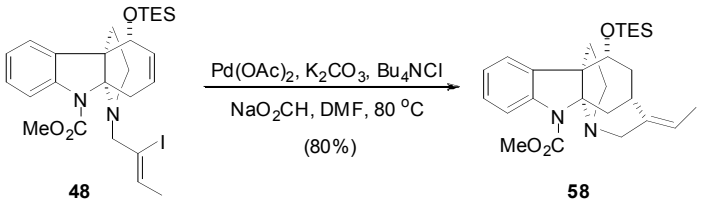


57 **59**

Alcohol 59. Tetrabutylammonium fluoride (1.0 M in THF, 60 μl, 0.060 mmol) was added to a stirring solution of aminal **57** (13 mg, 0.029 mmol) in THF (1 ml) at rt. Following stirring for 30 min, the reaction was partitioned between water and CH_2Cl_2. The organic layer was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (50% EtOAc/hexanes) gave **59** (8.6 mg, 0.026 mmol, 89%) as a colorless, amorphous solid. Crystallization from CH_2Cl_2/hexanes afforded X-ray quality crystals[7]: $[\alpha]_{589}$ +56.1, $[\alpha]_{577}$ +55.5, $[\alpha]_{546}$ +65.7, $[\alpha]_{435}$ +132, $[\alpha]_{405}$ +171 (*c* 0.47, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.26 (d, *J* = 8.2 Hz, 1H), 7.24 (dd, *J* = 1.0, 7.4 Hz, 1H), 7.14 (dt, *J* = 1.4, 7.5 Hz, 1H), 6.87 (dt, *J* = 1.0, 7.4 Hz, 1H), 5.57 (ddd, *J* = 1.9, 3.3, 10.0 Hz, 1H), 5.29 (ddd, *J* = 2.6, 2.6, 10.0 Hz, 1H), 5.04−5.06 (m, 1H), 3.94 (br s, 1H), 3.64 (d, *J* = 12.4 Hz, 1H), 3.51 (br s, 1H), 3.40 (s, 3H), 3.05−3.09 (m, 1H), 2.62 (ddd, *J* = 5.0, 11.9, 11.9 Hz, 1H), 2.31 (dd, *J* = 6.9, 11.8 Hz, 1H), 1.98 (ddd, *J* = 7.1, 12.1 12.1 Hz, 1H), 1.85 (dd, *J* = 4.0, 12.3 Hz, 1H), 1.45−1.47 (m, 3H), 1.31 (br s, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.0, 145.0, 143.2, 133.9, 131.0, 128.4, 128.2, 123.3, 122.5, 115.3, 110.8, 101.3, 72.2, 62.3, 60.2, 52.4, 50.8, 46.0, 32.2, 13.4; IR (neat) 3473, 1692 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{23}N_2O_3$ [M + H]: 339.1709. Found: 339.1700.

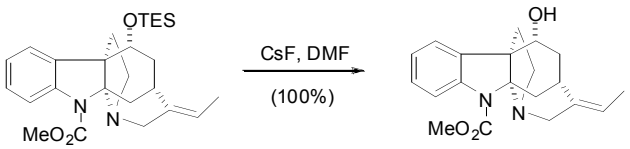


Alkene 63. Exposure of **48** to silica gel during chromatography for extended periods of time (>10 min) resulted in the formation of the chromatographically inseparable olefin **63**.[11] ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.91 (d, *J* = 8.1 Hz, 1H), 7.37 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.13−7.15 (m, 1H), 6.94−6.98 (m, 1H), 6.85 (br d, *J* = 10.5 Hz, 1H), 5.51 (ddd, *J* = 4.1, 4.1, 10.5 Hz, 1H), 5.46 (q, *J* = 6.4 Hz, 1H), 4.15 (br d, *J* = 15.0 Hz, 1H), 3.82 (t, *J* = 8.1 Hz, 1H), 3.63 (d, *J* = 15.0 Hz, 1H), 3.52 (s, 3H), 2.69 −2.72 (m, 1H), 2.55 (ddd, *J* = 3.4, 3.4, 7.0 Hz, 1H), 2.15−2.19 (m, 1H), 2.07−2.10 (m, 2H), 2.00−2.05 (m, 1H), 1.55 (d, *J* = 6.4 Hz, 1H), 0.84 (t, *J* = 8.0 Hz, 9H), 0.43 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.4, 142.0, 136.6, 129.4, 128.3, 125.5, 125.4, 124.0, 122.8, 116.8, 111.7, 90.5, 72.4, 61.3, 60.2, 52.0, 49.7, 31.5, 27.7, 21.5, 6.94, 5.56.



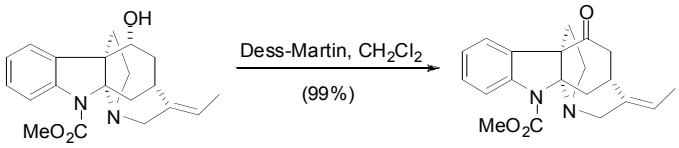
Pentacycle 58. A suspension of vinyl iodide **48** (1.20 g, 2.07 mmol), K_2CO_3 (1.43 g, 10.4 mmol), $Bu_4NCl•H_2O$ (1.43 g, 5.18 mmol), NaO_2CH (170 mg, 2.48 mmol) and DMF (30 mL) was degassed with Ar for 15 min. To this suspension was added $Pd(OAc)_2$ (5 mg, 0.02 mmol), and degassing was continued for an additional 10 min. The sealed reaction vial was then heated at 80 °C for 90 min by which time TLC analysis indicated complete consumption of starting material. After cooling to rt, the reaction was diluted with Et_2O and washed with brine. The organic layer was then dried (Na_2SO_4) and concentrated under reduced pressure. Purification of the crude residue by column chromatography (5% to 10% EtOAc/hexanes) gave **58** (750 mg, 1.65 mmol, 80%) as a clear, colorless oil: $[\alpha]_{589}$ –55.2, $[\alpha]_{577}$ –57.9, $[\alpha]_{546}$ –64.9, $[\alpha]_{435}$ –106, $[\alpha]_{405}$ –127 (*c* 1.25, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.15 (br s, 1H), 7.60 (dd, *J* = 7.5, 1.1 Hz, 1H), 7.17−7.20 (m, 1H), 6.96 (ddd, *J* = 7.5, 7.5, 1.1 Hz, 1H), 5.03 (q, *J* = 6.8 Hz,

1H), 4.29 (br s, 1H), 4.15 (dd, *J* = 11.4, 7.0 Hz, 1H), 3.57 (s, 3H), 3.04 (ddd, *J* = 14.6, 7.3, 3.5 Hz, 1H), 2.93 (d, *J* = 16.0 Hz, 1H), 2.79−2.74 (m, 1H), 2.66 (d, *J* = 11.1 Hz, 2H), 2.63−2.57 (m, 1H), 2.23 (ddd, *J* = 13.4, 11.5, 7.0 Hz, 1H), 1.72 (ddd, *J* = 13.4, 7.3, 6.1 Hz, 1H), 1.59 (dd, *J* = 13.9, 3.2 Hz, 1H), 1.44 (dd, *J* = 6.8, 2.0 Hz, 3H), 1.35−1.40 (m, 1H), 0.99 (t, *J* = 8.0 Hz, 9H), 0.63 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.1, 144.5, 141.9, 136.8, 128.4, 124.9, 122.5, 117.1, 115.3, 93.0, 73.4, 60.9, 55.7, 54.4, 51.6, 36.6, 34.0, 27.3, 25.8, 13.0, 7.1, 6.1; IR (neat) 1698 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{38}O_3N_2SiH$ [M + H]: 455.2730. Found: 455.2739.



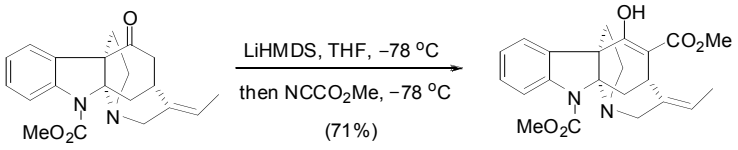
58 **64**

Alcohol 64. CsF (1.5 g, 9.90 mmol) was added to a stirred solution of aminal **58** (750 mg, 1.65 mmol) in DMF (25 mL) at rt. TLC analysis after 4 h indicated complete consumption of the starting material, and the reaction was subsequently partitioned between EtOAc and water. The aqueous layer was extracted with EtOAc, and the combined organic layers were washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (50% EtOAc/hexanes) gave alcohol **64** (562 mg, 1.65 mmol, 100%) as a colorless, amorphous solid. Crystallization from benzene/chloroform afforded X-ray quality crystals[7]: $[\alpha]_{589}$ –66.8, $[\alpha]_{577}$ –69.5, $[\alpha]_{546}$ –78.1, $[\alpha]_{435}$ –127, $[\alpha]_{405}$ –150 (*c* 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (br s, 1H), 7.51 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.23 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 7.00 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 5.32 (q, *J* = 6.7 Hz, 1H), 4.26 (br d, *J* = 14.6 Hz, 1H), 4.13−4.20 (m, 1H), 3.92 (s, 3H), 3.18 (ddd, *J* = 14.7, 7.3, 3.8 Hz, 1H), 3.12 (d, *J* = 15.9 Hz, 1H), 2.99−3.06 (m, 2H), 2.79 (br d, *J* = 10.9 Hz, 1H), 2.55 (ddd, *J* = 14.7, 7.5, 5.5 Hz, 1H), 2.50 (ddd, *J* = 13.8, 7.2, 2.2 Hz, 1H), 1.78−1.87 (m, 3H), 1.64 (dd, *J* = 6.7, 1.2 Hz, 3H), 1.41−1.47 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.4, 143.7, 141.2, 136.5, 128.5, 124.6, 123.2, 118.3, 115.1, 93.3, 72.3, 60.5, 55.8, 54.6, 52.6, 36.4, 33.4, 27.2, 26.3, 13.6; IR (neat) 3449, 1686 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{24}O_3N_2H$ [M + H]: 341.1865. Found: 341.1854.



64 **65**

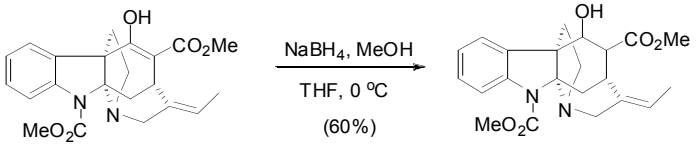
Ketone 46. Dess-Martin periodinane (2.1 g, 4.96 mmol) was added in one portion to a stirred solution of alcohol **64** (560 mg, 1.65 mmol) in CH_2Cl_2 (100 mL) at rt. The reaction was stirred for 16 h, and subsequently poured into saturated aqueous $NaHCO_3$. The aqueous layer was extracted with CH_2Cl_2, and the organic layer was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% to 50% EtOAc/hexanes) gave ketone **65** (553 mg, 1.63 mmol, 99%) as a colorless foam: $[\alpha]_{589}$ –165, $[\alpha]_{577}$ –175, $[\alpha]_{546}$ –200, $[\alpha]_{435}$ –404, $[\alpha]_{405}$ –545 (*c* 1.36, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.06 (br s, 1H), 7.91 (d, *J* = 7.5 Hz, 1H), 7.14 (m, 1H), 6.87 (t, *J* = 7.5, 1H), 5.08 (q, *J* = 6.8 Hz, 1H), 4.06 (br d, *J* = 15.0 Hz, 1H), 3.56 (s, 3H), 2.85−2.89 (m, 1H), 2.76 (d, *J* = 15.6 Hz, 1H), 2.68 (br d, *J* = 12.6 Hz, 1H), 2.50−2.58 (m, 2H), 2.42 (ddd, *J* = 12.4, 6.3, 1.9 Hz, 1H), 2.38 (d, *J* = 18.7 Hz, 1H), 2.20 (dd, *J* = 18.7, 7.7 Hz, 1H), 1.84 (ddd, *J* = 12.0, 7.4, 7.4 Hz, 1H), 1.41 (dd, *J* = 13.8, 2.1 Hz, 1H), 1.27 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 207.9, 153.8, 140.9, 137.8, 129.8, 129.1, 124.4, 122.8, 122.1, 115.6, 93.1, 67.7, 54.2, 53.2, 51.8, 45.1, 39.2, 28.4, 28.2, 13.0; IR (neat) 1701 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{22}O_3N_2Na$ [M + Na]: 361.1528. Found: 361.1528.



65 **66**

β-Ketoester 66. LiHMDS (1.0 M in THF, 3.1 ml, 3.10 mmol) was added dropwise to a stirred solution of ketone **65** (530 mg, 1.57 mmol) in THF (40 mL) at –78 °C. The reaction was then warmed to 0 °C for 30 min and subsequently re-cooled to –78 °C. $NCCO_2Me$ (0.5 ml, 6.28 mmol) was added and following stirring for 30 min at –78 °C, saturated aqueous NH_4Cl was added and the mixture was allowed to warm to rt.

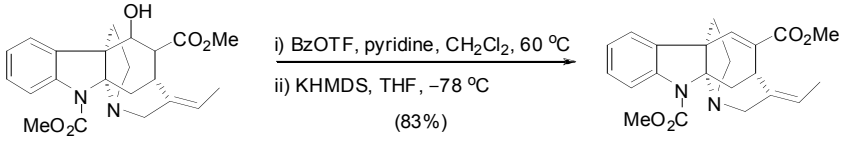
The reaction was extracted with EtOAc, and the organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the crude residue by column chromatography (20% to 35% EtOAc/hexanes) gave β-ketoester **66** (443 mg, 1.12 mmol, 71%) as a clear, colorless oil: $[\alpha]_{589}$ –196, $[\alpha]_{577}$ –207, $[\alpha]_{546}$ –237, $[\alpha]_{435}$ –438, $[\alpha]_{405}$ –566 (*c* 1.40, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 13.1 (s, 1H), 8.06 (br d, *J* = 8.1 Hz, 1H), 7.90 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.15 (m, 1H), 6.89 (dt, *J* = 7.5, 1.0 Hz, 1H), 5.20 (q, *J* = 6.5 Hz, 1H), 4.11 (br d, *J* = 15.9 Hz, 1H), 3.64 (br s, 1H), 3.54 (s, 3H), 3.34 (s, 3H), 2.89−2.96 (m, 2H), 2.58−2.62 (m, 1H), 2.54−2.57 (m, 1H), 2.44 (dd, *J* = 12.6, 6.1 Hz, 1H), 1.96 (ddd, *J* = 11.8, 7.4, 7.4 Hz, 1H), 1.52−1.56 (m, 4H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 172.4, 171.4, 153.7, 140.7, 137.1, 130.9, 129.0, 125.1, 122.8, 120.7, 115.7, 103.7, 92.0, 59.8, 53.06, 53.05, 51.8, 50.9, 37.7, 28.70, 28.67, 13.5; IR (neat) 1702, 1645 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{24}O_5N_2Na$ [M + Na]: 419.1583. Found: 419.1584.



66 **67**

β-Hydroxyester 66. $NaBH_4$ (243 mg, 6.36 mmol) was added in three portions over a 2 h to a stirring solution of β-ketoester **66** (420 mg, 1.06 mmol) in 10:1 THF/MeOH (10 mL) at –20 °C. After the final $NaBH_4$ addition, the reaction was warmed to 0 °C and stirred at this temperature for 2 h. The mixture was quenched with saturated aqueous NH_4Cl and allowed to warm to rt. The reaction was extracted with CH_2Cl_2, and the organic layer was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% to 50% EtOAc/hexanes) gave β-hydroxyester **67** (254 mg, 0.64 mmol, 60%) as a clear, colorless oil and ~10-15% recovered starting material. **66**: $[\alpha]_{589}$ –89.7, $[\alpha]_{577}$ –93.0, $[\alpha]_{546}$ –106, $[\alpha]_{435}$ –176, $[\alpha]_{405}$ –215 (*c* 0.90, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.14 (br s, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.79 (t, *J* = 7.4 Hz, 1H), 6.68 (d, *J* = 7.3 Hz, 1H), 5.05 (q, *J* = 6.8 Hz, 1H), 4.56 (d, *J* = 4.1 Hz, 1H), 4.25 (br s, 1H), 3.81 (br s, 1H), 3.52 (s, 3H), 3.51 (s, 3H), 2.85−2.93 (m, 3H), 2.72−2.76 (m, 2H),

2.51−2.56 (m, 1H), 1.75 (ddd, J = 12.7, 8.2, 8.2 Hz, 1H), 1.53−1.57 (m, 2H), 1.51 (d, J = 6.7 Hz, 3H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 173.2, 154.1, 143.6, 131.9, 129.1, 128.5, 128.3, 122.8, 118.1, 116.0, 91.4, 72.8, 61.9, 56.9, 54.0, 51.7, 51.6, 49.8, 38.8, 28.7, 26.0, 13.2; IR (neat) 3461, 1741, 1702 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{26}O_5N_2H$ [M + H]: 399.1920. Found: 399.1914.

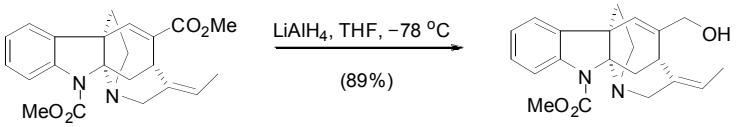


67 **68**

Enoate 68. Benzoyl triflate[12] (1.65 ml, 9.8 mmol) was added to a stirring solution of β-hydroxyester **67** (195 mg, 0.49 mmol) and pyridine (1.6 ml, 19.6 mmol) in CH_2Cl_2 (6 mL) at 0 °C. The reaction vial was sealed and heated to 60 °C for 24 h. The bright red solution was then cooled to rt and quenched with saturated aqueous $NaHCO_3$. The reaction was extracted with CH_2Cl_2, and the organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the crude residue by column chromatography (35% to 50% EtOAc/hexanes) gave the benzoate (246 mg, 0.49 mmol, 100%) as a clear, colorless oil which was used in the subsequent elimination step: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.00 (d, J = 8.6 Hz, 1H), 7.41–7.29 (m, 4H), 7.17–7.14 (m, 2H), 6.99 (d, J = 7.3 Hz, 1H), 6.92 (t, J = 7.6 Hz, 1H), 6.69 (t, J = 7.3 Hz, 1H), 6.22 (d, J = 4.9 Hz, 1H), 5.29–5.25 (m, 1H), 4.20 (br s, 1H), 3.81 (s, 3H), 3.59 (s, 1H), 3.46 (s, 3H), 3.07–2.85 (m, 4H), 2.46 (d, J = 13.0 Hz, 1H), 2.23–2.17 (m, 1H), 1.96–1.91 (m, 1H), 1.43 (d, J = 6.8 Hz, 3H).

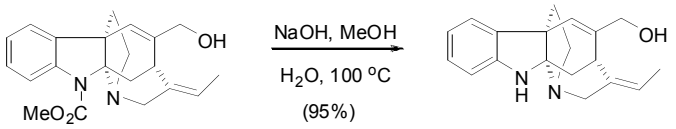
KHMDS (0.5 M in toluene, 1.7 ml, 0.85 mmol) was added dropwise to a stirring solution of this benzoate intermediate (215 mg, 0.43 mmol) in THF (8 mL) at –78 °C. After 10 min the reaction was quenched with saturated aqueous $NaHCO_3$ and warmed to rt. The reaction was extracted with Et_2O, and the organic layer was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by column chromatography (50% EtOAc/hexanes) gave enoate **68** (135 mg, 0.36 mmol, 83%) as a clear, colorless oil: $[\alpha]_{589}$ –124, $[\alpha]_{577}$ –131, $[\alpha]_{546}$ –149, $[\alpha]_{435}$ –268, $[\alpha]_{405}$ –340 (*c* 1.05, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.04 (br s, 1H),

7.12 (dt, *J* = 7.5, 1.4 Hz, 1H), 6.94 (s, 1H), 6.90 (dd, *J* = 7.3, 1.0 Hz, 1H), 6.82 (dt, *J* = 7.4, 1.0 Hz, 1H), 5.23 (q, *J* = 7.0 Hz, 1H), 4.17 (br d, *J* = 15.5 Hz, 1H), 3.92 (br s, 1H), 3.54 (s, 3H), 3.47 (s, 3H), 2.88−2.90 (m, 2H), 2.54−2.59 (m, 2H), 1.85 (ddd, *J* = 12.5, 7.3, 7.3 Hz, 1H), 1.70 (dd, *J* = 7.0, 2.3 Hz, 3H), 1.63 (dd, *J* = 12.4, 5.9 Hz, 1H), 1.45 (dd, *J* = 12.8, 2.7 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 167.2, 153.9, 140.6, 136.5, 136.3, 136.0, 132.3, 128.7, 122.50, 122.48, 121.9, 116.0, 91.7, 56.7, 53.3, 52.6, 51.8, 51.5, 37.9, 30.7, 28.5, 13.7; IR (neat) 1705 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{24}O_4N_2H$ [M + H]: 381.1814. Found: 381.1820.



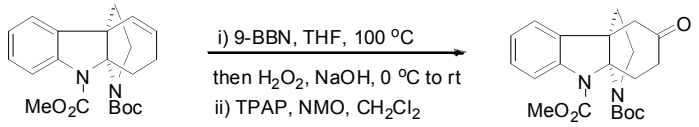
68 **69**

Alcohol 69. A solution of enoate **68** (100 mg, 0.26 mmol) in THF (2 mL) was added dropwise to a stirring suspension of $LiAlH_4$ (100 mg, 2.60 mmol) in THF (10 mL) at –20 °C. After 10 min the reaction was quenched with water (0.1 ml), followed by 1 N NaOH (0.1 ml), and finally H_2O (0.3 ml). The resulting mixture was stirred for 10 min and then $MgSO_4$ was added. The solid materials were removed by filtration and the filtrate was concentrated under reduced pressure. Purification of the crude residue by column chromatography (50% to 75% EtOAc/hexanes) gave alcohol **69** (82 mg, 0.23 mmol, 89%) as a clear, colorless oil: $[\alpha]_{589}$ +15.9, $[\alpha]_{577}$ +16.5, $[\alpha]_{546}$ +19.8, $[\alpha]_{435}$ +46.1, $[\alpha]_{405}$ +63.6 (*c* 0.90, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 8.08 (br s, 1H), 7.14 (m, 1H), 6.94 (s, 1H), 7.06 (d, *J* = 7.4 Hz, 1H), 6.89 (m, 1H), 5.67 (s, 1H), 5.16 (q, *J* = 6.8 Hz, 1H), 4.22 (br s, 1H), 3.92 (q, *J* = 13.5 Hz, 2 H), 3.57 (s, 3H), 3.23 (br s, 1H), 2.94−2.99 (m, 2H), 2.51−2.56 (m, 2H), 1.87 (ddd, *J* = 12.4, 7.1, 7.1 Hz, 1H), 1.59−1.61 (m, 4H), 1.52 (dd, *J* = 12.1, 5.6 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 154.2, 144.6, 140.7, 137.7, 134.0, 128.4, 122.6, 122.4, 121.2, 119.5, 116.0, 92.1, 65.5, 56.2, 53.0, 52.9, 51.8, 38.5, 31.1, 28.1, 14.3; IR (neat) 3394, 1697 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{24}O_3N_2H$ [M + H]: 353.1865. Found: 353.1854.



69 (+)-minfiensine (**1**)

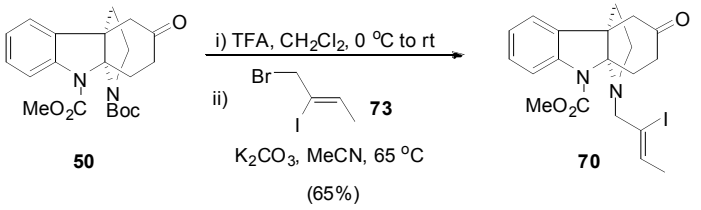
Minfiensine (1). 3 N NaOH (3.2 ml, 9.7 mmol) was added to a stirring solution of carbamate **69** (68 mg, 0.19 mmol) in 1:1 MeOH/H_2O (12 ml). The resultant solution was heated to 100 °C for 2.5 h and subsequently cooled to rt. The reaction was diluted with saturated aqueous $NaHCO_3$, extracted with EtOAc, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the crude residue by column chromatography (100% EtOAc then 99% MeCN/1% NH_4OH) gave (+)-minfiensine (**1**) (54 mg, 0.18 mmol, 95%) as a colorless foam. The ^1H and ^{13}C NMR spectra of the synthetic material were identical to reported literature values.[13] Synthetic minfiensine displayed $[\alpha]_D^{23}$ +125 (*c* 0.82, $CHCl_3$), whereas $[\alpha]_D^{23}$ +134 (*c* 0.82, $CHCl_3$) is reported for natural minfiensine.[13]



27 **50**

Ketone 50. Olefin **27** (305 mg, 0.82 mmol) and 9-BBN (0.5 M in THF, 5.0 mL, 2.50 mmol) were added to an 8 mL microwave reaction vial and capped. The reaction mixture was microwave-heated (CEM Discover System, 60 Hz and 300 W instrument) at 100 °C for 1 h. After cooling to rt, TLC analysis showed incomplete conversion. Additional 9-BBN (0.5 M in THF, 0.83 mL, 0.41 mmol) was added, and heating was continued at 100 °C for 20 min. The reaction mixture was cooled to 0 °C, and 3 N NaOH and 30% H_2O_2 (2 mL each) were added sequentially, and the mixture was stirred at 0 °C for 10 min and at rt for an additional 30 min. The mixture was partitioned between Et_2O and brine, and the separated organic phase was dried (Na_2SO_4), filtered, and concentrated under reduced pressure to afford a clear, colorless oil that was filtered through a plug of silica gel (50% EtOAc/hexanes). After concentration, this residue (0.82 mmol theoretical) in CH_2Cl_2 (10 mL) was charged sequentially with crushed 4 Å molecular sieves (~1 g), TPAP (29 mg, 0.082 mmol), and NMO (985 mg, 8.2 mmol). The reaction

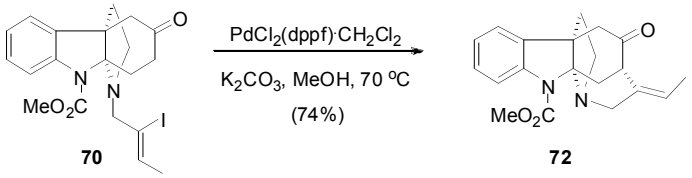
mixture was maintained at rt for 20 min, then concentrated to ~1 mL under reduced pressure. Purification of the crude residue by column chromatography (10% to 25% EtOAc/hexanes) gave **50** (200 mg, 0.52 mmol, 63%) as a colorless foam (**49** was isolated in 25% yield; see above for optimized procedure): **50** $[\alpha]_{589}$ –157, $[\alpha]_{577}$ –159, $[\alpha]_{546}$ –183, (*c* 0.52, $CHCl_3$); ^{1}H NMR (600 MHz, C_6D_6, 60 °C) δ 8.05 (d, *J* = 8.2 Hz, 1H), 7.05 (dt, *J* = 1.1, 8.2 Hz, 1H), 6.79 (t, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.2 Hz, 1H), 3.58–3.61 (m, 4H), 3.12 (ddd, *J* = 4.4, 8.0, 14.5 Hz, 1H), 2.78 (ddd, *J* = 6.1, 11.4, 17.5 Hz, 1H), 2.57 (ddd, *J* = 4.5, 9.0, 13.9 Hz, 1H), 2.35 (q, *J* = 15.2 Hz, 2H), 2.16 (ddd, *J* = 4.4, 9.0, 18.6 Hz, 1H), 1.98 (ddd, *J* = 4.5, 8.1, 18.6 Hz, 1H), 1.48 (dd, *J* = 4.9, 11.3 Hz, 1H), 1.43 (dd, *J* = 7.9, 11.3 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (150 MHz, C_6D_6, 60 °C) δ 206.3, 153.2, 152.3, 142.3, 133.5, 128.6, 123.2, 122.3, 115.3, 88.7, 79.3, 57.1, 51.6, 47.9, 45.3, 38.6, 34.7, 29.0, 28.0; IR (neat) 1684, 1719 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{26}N_2O_5Na$ [M + Na]: 409.1740. Found: 409.1732.



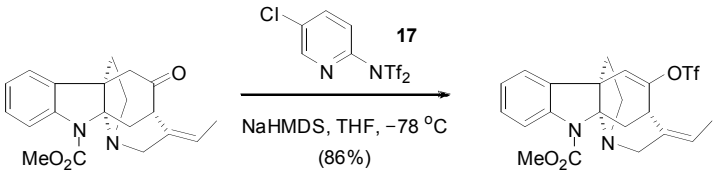
Ketone 70. Using procedures described above (**52** → **53** and **46** → **48**), **50** (104 mg, 0.27 mmol) was *N*-Boc deprotected (trifluoroacetic acid) to give the NH aminal: ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.15 (br s, 1H), 7.05 (t, *J* = 7.3 Hz, 1H), 6.81 (dt, *J* = 7.6, 0.9 Hz, 1H), 6.73 (dd, *J* = 7.3, 1.3, Hz, 1H), 4.32 (s, 1H), 3.44 (br s, 3H), 2.55−2.51 (m, 2H), 2.43−2.29 (m, 3H), 2.02−1.85 (m, 3H), 1.66−1.60 (m, 1H), 1.57 (dd, *J* = 12.2, 5.4 Hz, 1H).

Subsequent alkylation with (*Z*)-1-bromo-2-iodo-2-butene (**73**)[14] and purification of the crude residue by column chromatography (25% EtOAc/hexanes) gave ketone **70** (81 mg, 0.17 mmol, 65% over two steps) as a clear, colorless oil: $[\alpha]_{589}$ +79.4, $[\alpha]_{577}$ +82.8, $[\alpha]_{546}$ +95.0, $[\alpha]_{435}$ +187, $[\alpha]_{405}$ +246 (*c* 0.66, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.83 (br d, *J* = 8.0 Hz, 1H), 7.03 (dt, *J* = 1.4, 7.4 Hz, 1H), 6.79 (dt, *J* = 0.9, 7.5 Hz, 1H), 6.66 (dd, *J* = 1.1, 7.5 Hz, 1H), 5.31 (q, *J* = 6.4 Hz, 1H), 4.26 (br d, *J* = 14.1 H, 1H), 3.43 (s, 3H), 3.13 (d, *J* = 14.3 Hz, 1H), 2.88 (dt, *J* = 4.6, 12.1 Hz, 1H), 2.65 (ddd, *J*

= 4.5, 12.1, 18.9 Hz, 1H), 2.49–2.53 (m, 2H), 2.25 (d, *J* = 15.6 Hz, 1H), 2.04 (dt, *J* = 4.1, 19.0 Hz, 1H), 1.95–2.00 (m, 1H), 1.82–1.88 (m, 1H), 1.51–1.58 (m, 2H), 1.49 (dd, *J* = 1.8, 6.4 Hz, 3H); ^{13}C NMR (150 MHz, C_6D_6, 60 °C) δ 207.0, 154.0, 142.7, 135.0, 130.6, 128.0, 123.2, 122.5, 115.7, 111.1, 90.8, 59.7, 56.5, 51.4, 48.2, 46.7, 38.4, 35.1, 29.0, 21.1; IR (neat) 1702, 1717 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{23}IN_2O_3Na$ [M + Na]: 489.0651. Found: 489.0654.

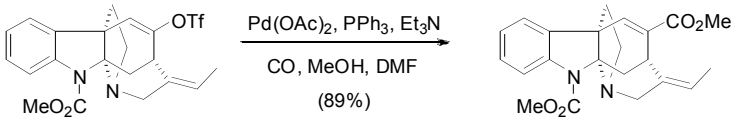


Palladium-Catalyzed Intramolecular Enolate/Vinyl Iodide Coupling. Preparation of Pentacycle 72: To a 1 dram vial was added ketone **70** (20 mg, 0.043 mmol), $PdCl_2(dppf)•CH_2Cl_2$ (3.5 mg, 0.0043 mmol), K_2CO_3 (24 mg, 0.17 mmol), and MeOH (1.5 ml). The vial was sealed with a Teflon septum cap, and the solution was degassed with argon for 10 min. The sealed vial was heated to 70 °C, and at this temperature, the solid K_2CO_3 completely dissolved. After 1 h the reaction was cooled to rt and partitioned between Et_2O and brine. The organic phase was dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by preparative TLC (75% EtOAc/hexanes) gave pentacycle **72** (10.7 mg, 0.032 mmol, 74%) as a clear, colorless oil. $[\alpha]_{589}$ +98.7, $[\alpha]_{577}$ +105, $[\alpha]_{546}$ +125, $[\alpha]_{435}$ +301, $[\alpha]_{405}$ +431 (*c* 1.13, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.83 (br s, 1H), 7.07 (dt, *J* = 1.4, 7.4 Hz, 1H), 6.79 (dt, *J* = 1.0, 7.4 Hz, 1H), 6.68 (dd, *J* = 1.3, 7.4 Hz, 1H), 5.27 (q, *J* = 6.8 Hz, 1H), 4.27 (br s, 1H), 3.49 (s, 3H), 3.05 (br s, 1H), 2.93 (d, *J* = 16.2 Hz, 1H), 2.77 (ddd, *J* = 2.4, 7.5, 9.8 Hz, 1H), 2.54–2.60 (m, 2H), 2.48 (s, 2H), 1.79 (dd, *J* = 1.8, 6.4 Hz, 3H), 1.66–1.73 (m, 2H), 1.50 (ddd, *J* = 2.4, 6.1, 12.6 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 207.7, 153.6, 135.6, 134.7, 128.5, 127.4, 122.8, 122.7, 122.1, 115.5, 91.4, 56.1, 53.3, 52.8, 51.5, 47.7, 44.3, 40.6, 26.9, 14.3; IR (neat) 1694 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{22}N_2O_3Na$ [M + Na]: 361.1528. Found: 361.1531.



72 **75**

Triflate 62. A THF solution of NaHMDS (90 μL of a 2.0 M solution, 0.18 mmol) was added dropwise to a solution of **72** (30 mg, 0.089 mmol), 2-[*N*,*N*-bis(trifluoromethylsulfonyl)amino]-5-chloropyridine (70 mg, 0.18 mmol), and THF (2 mL) at –78 °C under Ar. After 15 min, TLC analysis indicated incomplete conversion. Additional 2-[*N*,*N*-bis(trifluoromethylsulfonyl)amino]-5-chloropyridine (18 mg, 0.045 mmol) and NaHMDS (25 μL of a 2.0 M solution, 0.045 mmol) were added, and the reaction was stirred at –78 °C for 20 min. At this time, TLC indicated complete conversion, and the reaction was then quenched with saturated aqueous NH_4Cl and warmed to rt. The separated aqueous phase was washed with Et_2O, and the combined organic phases were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the crude residue by preparative TLC (50% EtOAc/hexanes) gave **75** (36 mg, 0.077 mmol, 86%) as a clear, colorless oil: $[\alpha]_{589}$ –14.0, $[\alpha]_{577}$ –15.2, $[\alpha]_{546}$ –17.2, $[\alpha]_{435}$ –23.6, $[\alpha]_{405}$ –26.3 (*c* 0.92, $CHCl_3$); ^{1}H NMR (500 MHz, C_6D_6, 60 °C) δ 7.92 (br s, 1H), 7.05 (dt, *J* = 1.4, 7.4 Hz, 1H), 6.81 (dd, *J* = 1.1, 7.2 Hz, 1H), 6.76 (t, *J* = 7.4 Hz, 1H), 5.68 (s, 1H), 5.15 (q, *J* = 6.9 Hz, 1H), 4.00 (br s, 1H), 3.47 (s, 3H), 3.13 (br s, 1H), 2.77–2.82 (m, 2H), 2.45 (ddd, *J* = 5.8, 8.7, 14.3 Hz, 1H), 2.34 (br s, 1H), 1.71 (ddd, *J* = 7.3, 12.6, 12.6 Hz, 1H), 1.58 (dd, *J* = 2.1, 6.9 Hz, 3H), 1.55 (dd, *J* = 2.9, 13.2 Hz, 1H), 1.38 (dd, *J* = 5.7, 12.5 Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6, 60 °C) δ 153.5, 153.0, 140.1, 133.4, 131.4, 128.8, 122.8, 122.6, 122.1, 119.0 (q, $J_{C,F}$ = 319 Hz), 115.7, 115.3, 90.6, 56.3, 52.2, 51.64, 51.60, 37.9, 34.9, 27.7, 13.7; IR (neat) 1698 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{22}F_3N_2O_5S$ [M + H]: 471.1201. Found: 471.1193.

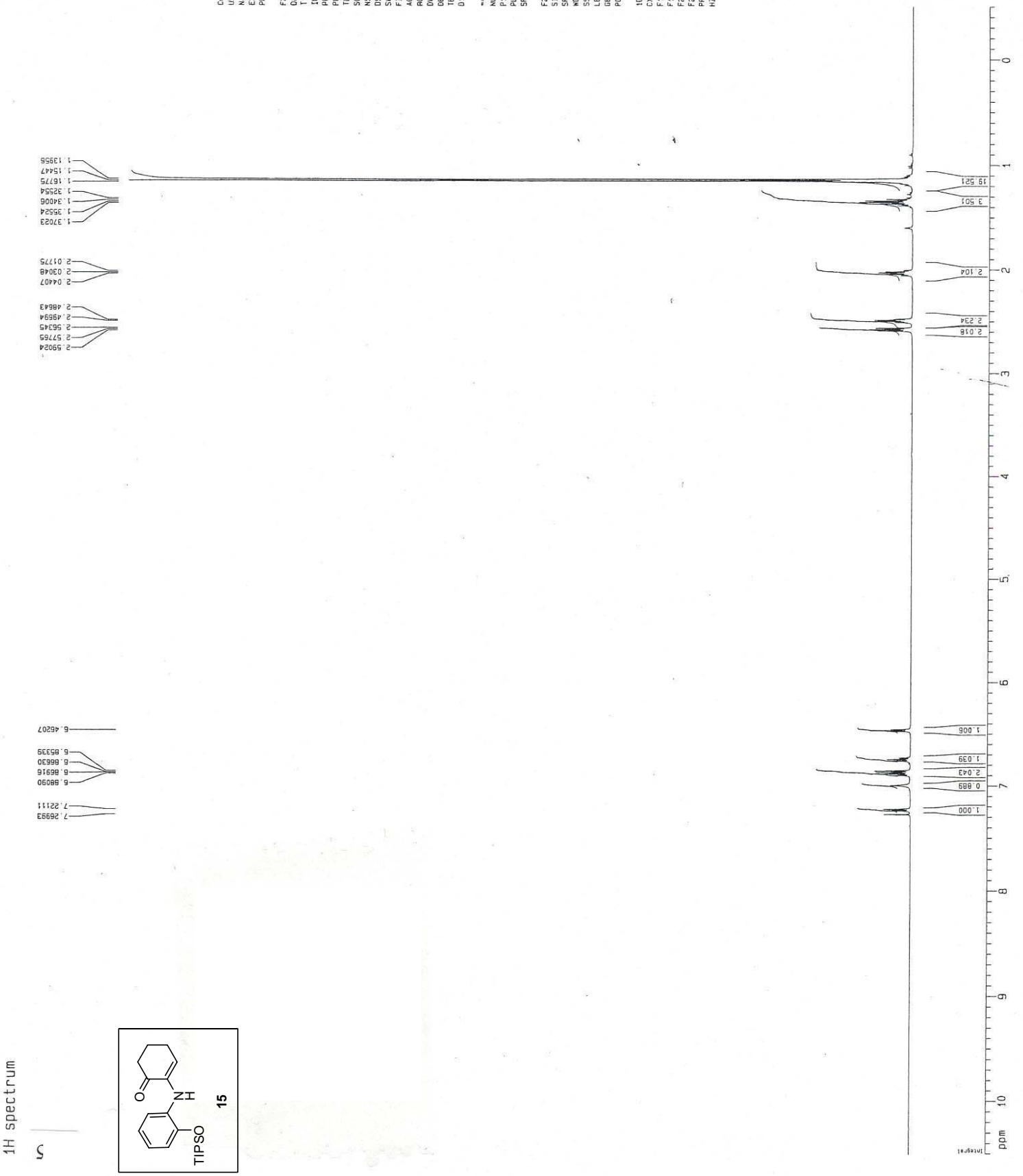


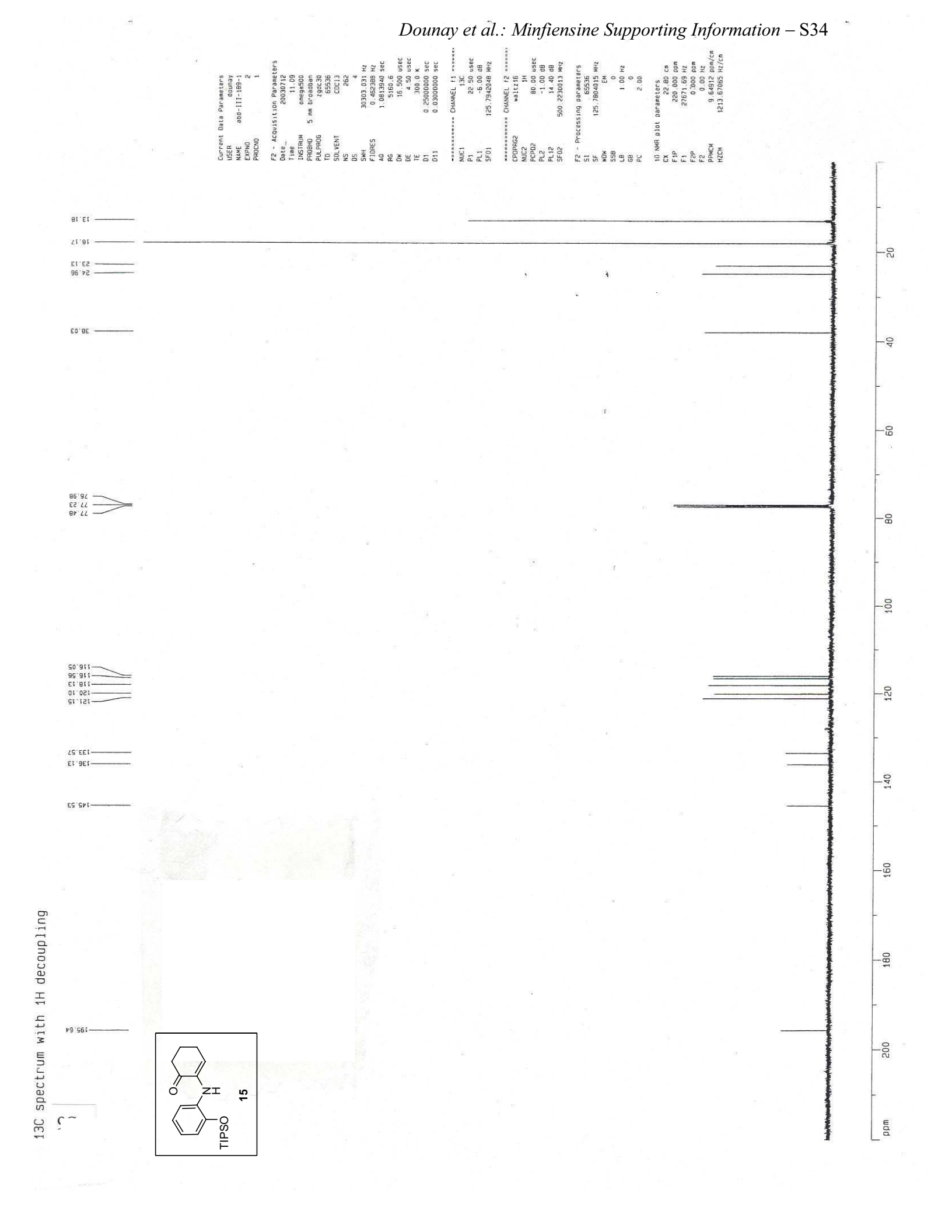
75 **68**

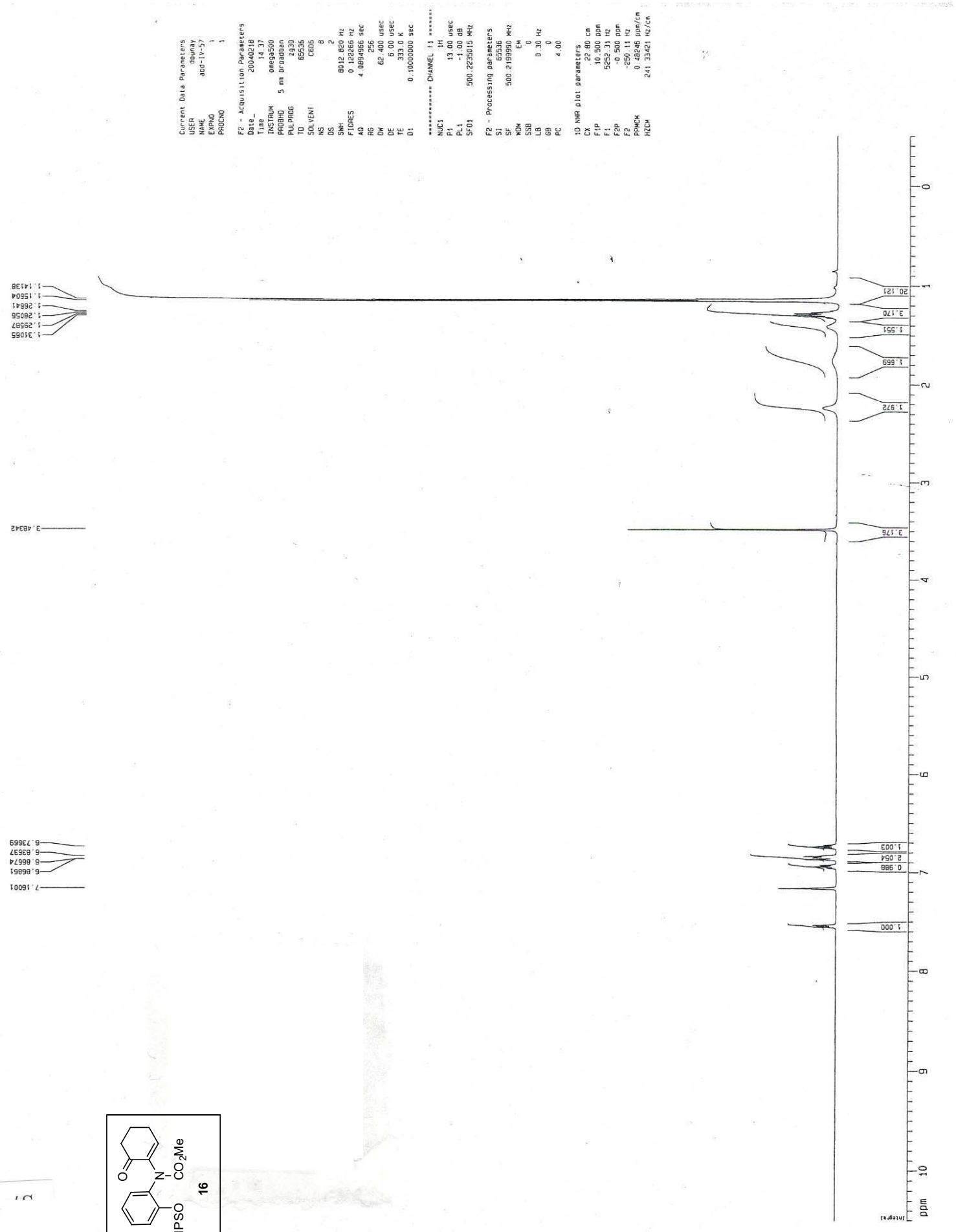
Enoate 56. To a 1 dram vial was added triflate **75** (34 mg, 0.072 mmol), $Pd(OAc)_2$ (5 mg, 0.022 mmol), PPh_3 (11 mg, 0.43 mmol), Et_3N (30 μl, 0.22 mmol), MeOH (1 ml), and DMF (1 ml). The vial was sealed with a Teflon septum cap, and the solution was degassed with a balloon of CO for 5 min. The sealed vial was then heated to 50 °C under a balloon of CO. After 3.5 h the reaction was cooled to rt and partitioned between Et_2O and brine. The organic phase was washed an additional time with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. Purification of the crude residue by preparative TLC (50% EtOAc/hexanes) gave methyl ester **68** (24 mg, 0.064 mmol, 89%) as a colorless oil. The ^{1}H and ^{13}C NMR of **68** were identical to the spectral values reported above.

References.

1. Kondo, Y.; Kojima, S.; Sakamoto, T. *J. Org. Chem.* **1997**, *62*, 6507–6511.
2. Ohashi, M.; Takahashi, T.; Inoue, S.; Sato, K. *Bull. Chem. Soc. Jpn.* **1975**, *48*, 1892–1896.
3. Because of carbamate rotomers, most ^{1}H and ^{13}C NMR spectra in Supporting Information were conducted in d_6-benzene at 60 °C.
4. Hart, R. *Bull. Soc. Chim. Belg.* **1956**, *65*, 291–296.
5. Cesium carbonate and cesium fluoride were dried in a vacuum oven set to 120 °C for 24 h.
6. Loiseleur, O.; Meier, P.; Pfaltz, A. *Angew. Chem. Int. Ed.* **1996**, *35*, 200-202.
7. For X-ray crystallographic analysis data, see accompanying cif file.
8. Sharpless, K.B.; Lauer, R.F. *J. Am. Chem. Soc.* **1973**, *95*, 2697-2699.
9. Rawal, V.H.; Michoud, C. *Tetrahedron Lett.* **1991**, *32*, 1695-1698.
10. Nicolaou, K.C.; Zhong, Y.-L.; Baran, P.S. *J. Am. Chem. Soc.* **2000**, *122*, 7596–7597.
11. Copies of ^{1}H and ^{13}C NMR data for **63** are presented as a mixture with **48** in Supporting Information, see page S81.
12. Brown, L.; Koreeda, M. *J. Org. Chem.* **1984**, *49*, 3875-3880.
13. Massiot, G.; Thépenier, P.; Jacquier, M.; Le Men-Olivier, L.; Delaude, C. *Heterocycles* **1989**, *29*, 1435-1438.
14. Rawal, V.H.; Michoud, C.; Monestel, R.F. *J. Am. Chem. Soc.* **1993**, *115*, 3030-3031.

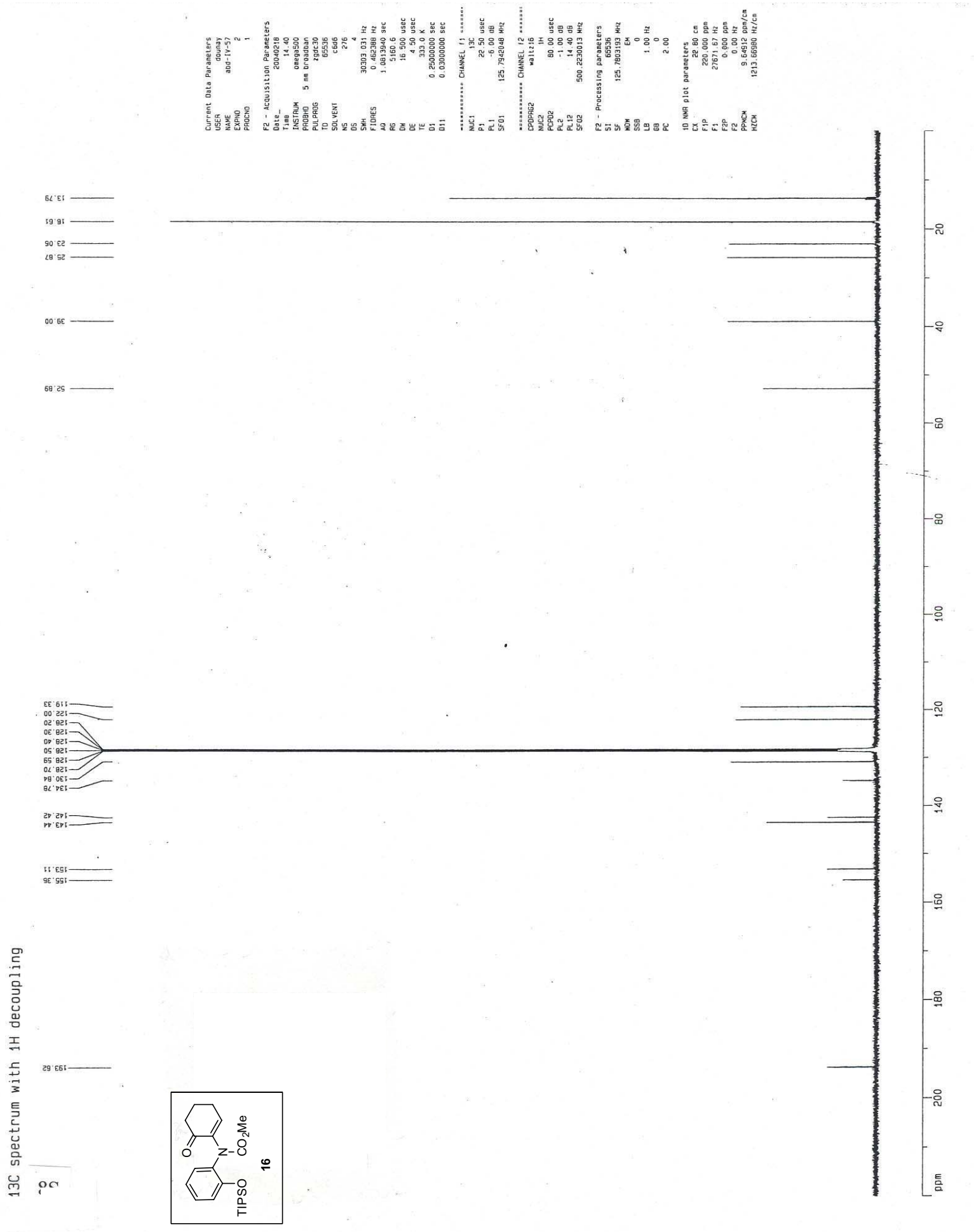

Current Data Parameters
USER dounay
NAME add-III-189-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030712
Time_ 11.05
INSTRUM DMEGA500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200262 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm
1.13956
1.15447
1.16775
1.32554
1.34006
1.35524
1.37023
2.01775
2.03048
2.04407
2.48643
2.49894
2.56345
2.57765
2.59024
6.46207
6.85339
6.86630
6.86916
6.88090
7.26993
7.27111
19.521
3.501
2.104
2.234
2.018
1.006
1.039
2.043
0.889
1.000
integral
ppm
1H spectrum
TIPSO
15

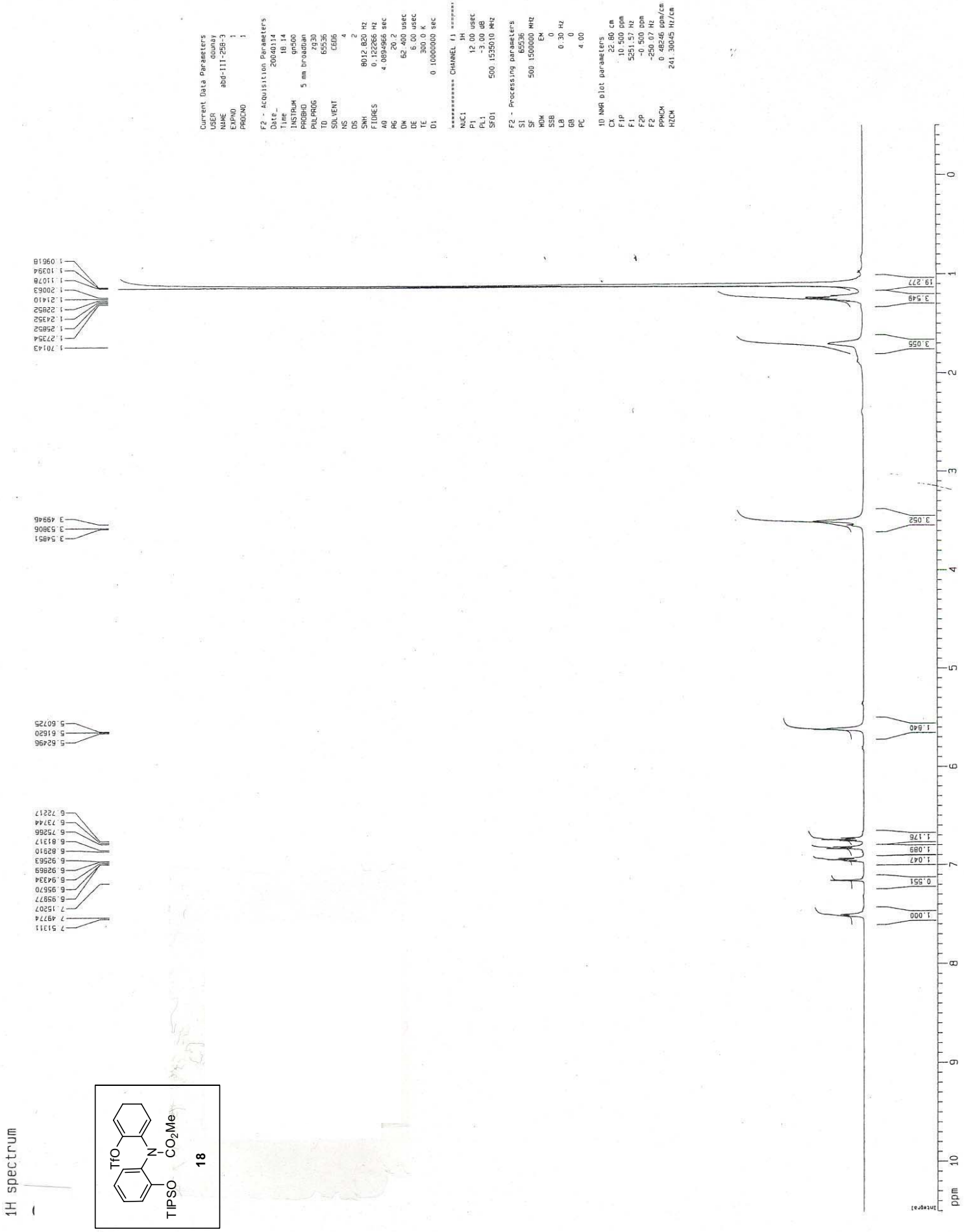

13C spectrum with 1H decoupling
TIPSO
15
Current Data Parameters
USER dounay
NAME abd-III-189-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030712
Time 11.09
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 262
DS 4
SWH 30303.031 Hz
FIDRES 0.462308 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7804015 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P 0.300 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm
13.18
18.17
23.13
24.96
38.03
76.98
77.23
77.48
116.05
116.56
118.13
120.10
121.15
133.57
136.13
145.53
195.64
ppm

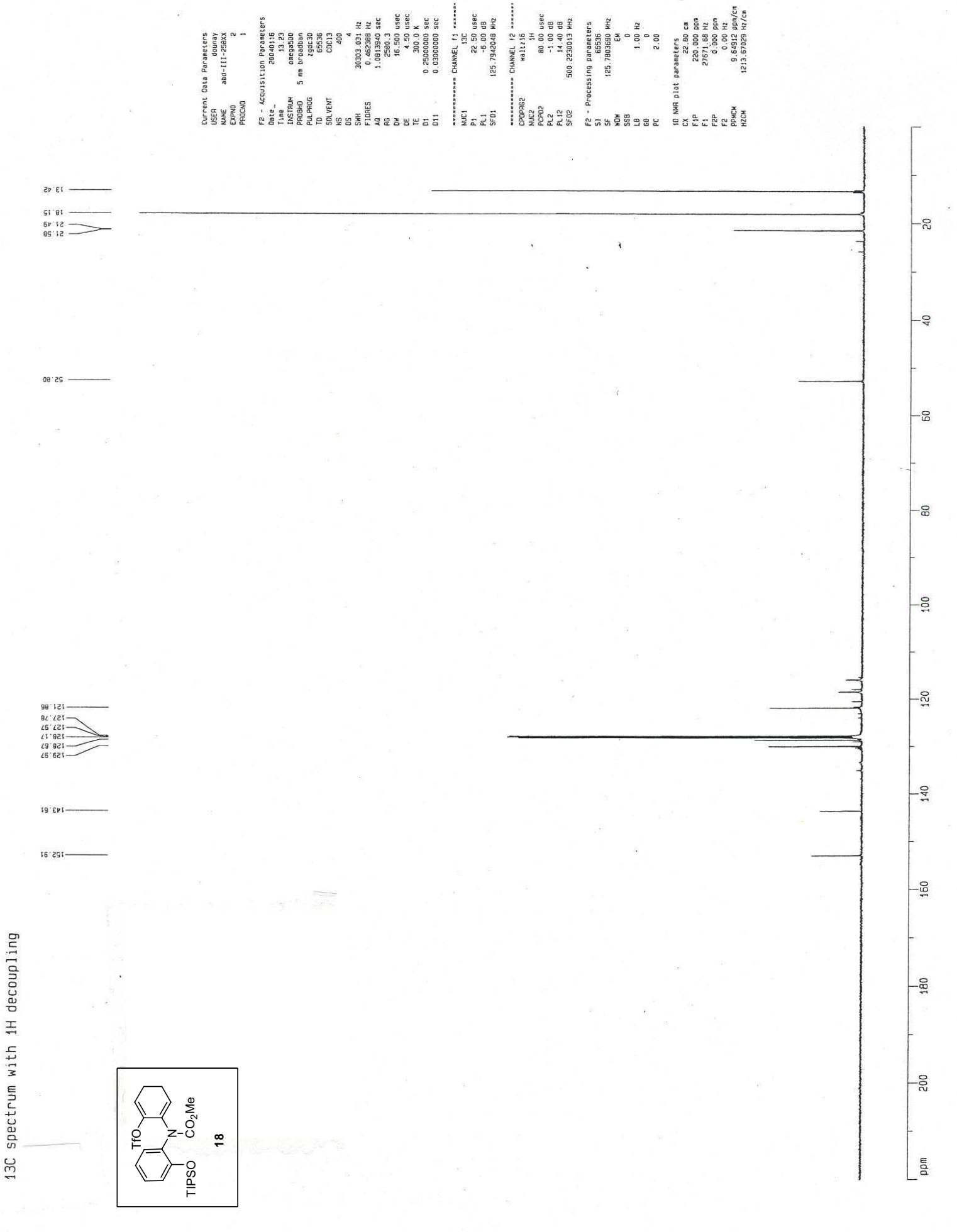

Current Data Parameters
USER dounay
NAME dod-IV-57
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040218
Time 14.37
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG 2g30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894956 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2199990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
3.13055
3.29587
3.28056
3.26641
3.15604
3.14138
3.48342
7.16001
6.86861
6.86574
6.86337
6.73669
20.121
3.170
1.551
1.669
1.972
3.176
1.003
2.054
0.988
1.000
ppm

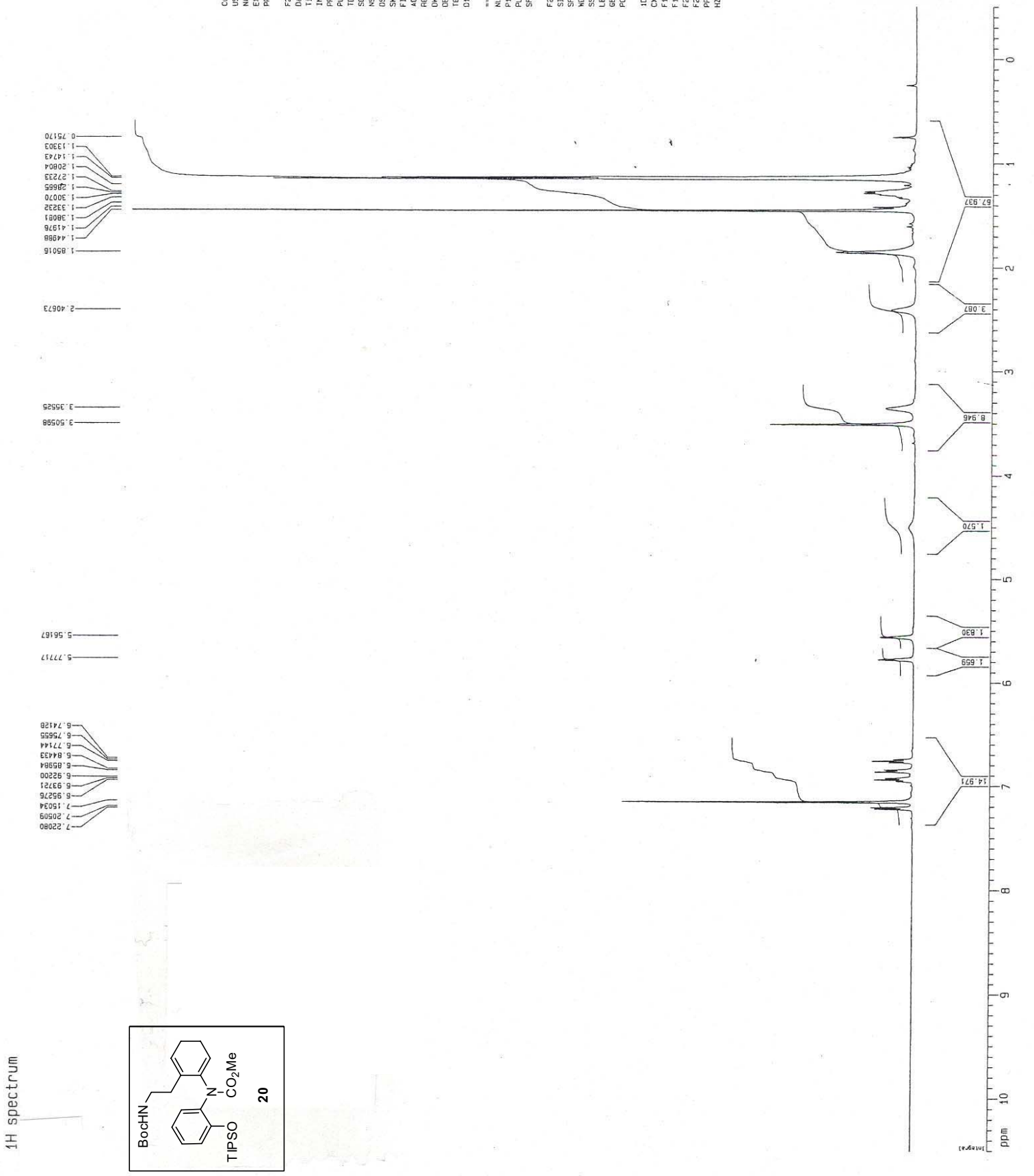
O
N
CO2Me
TIPSO

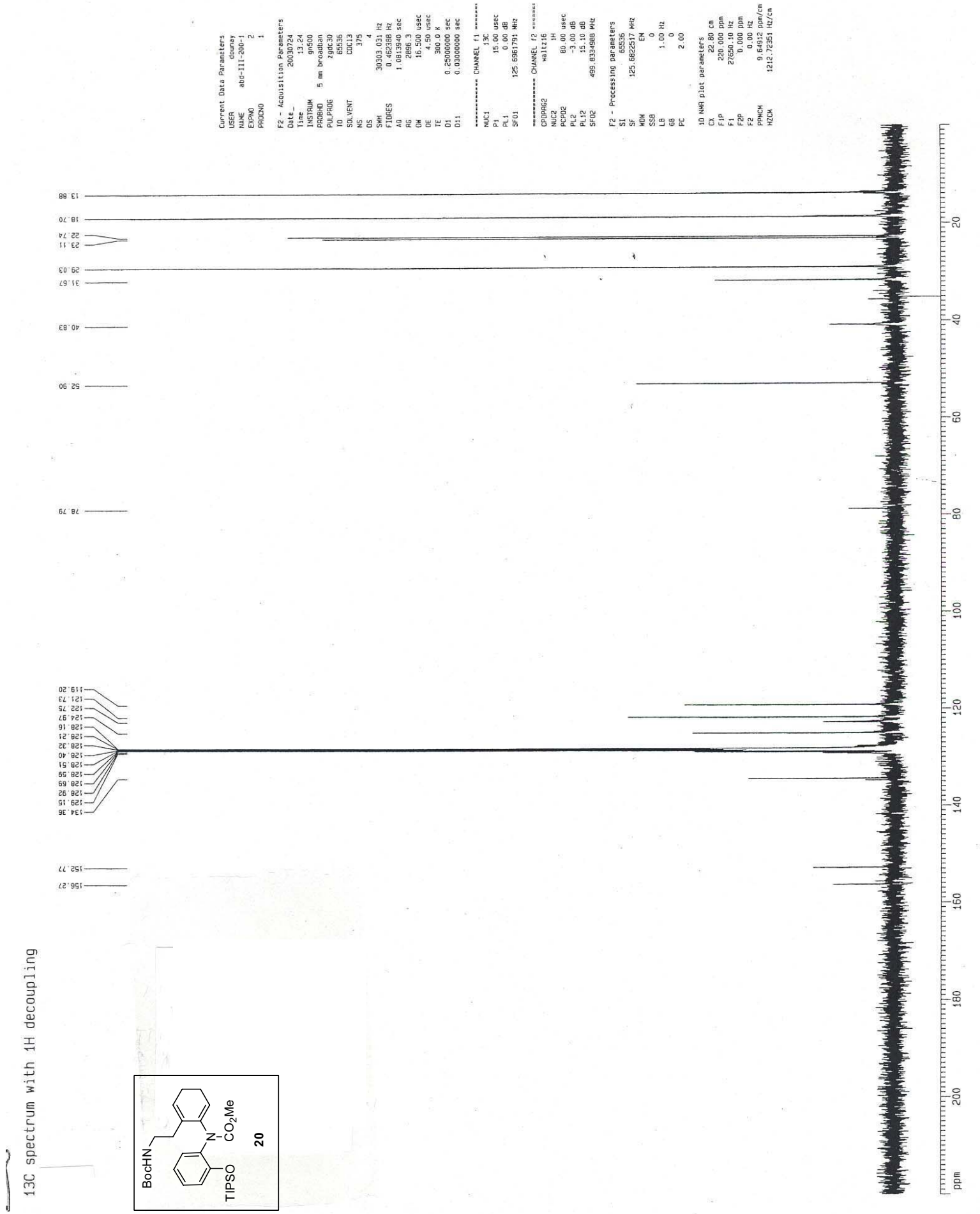
16

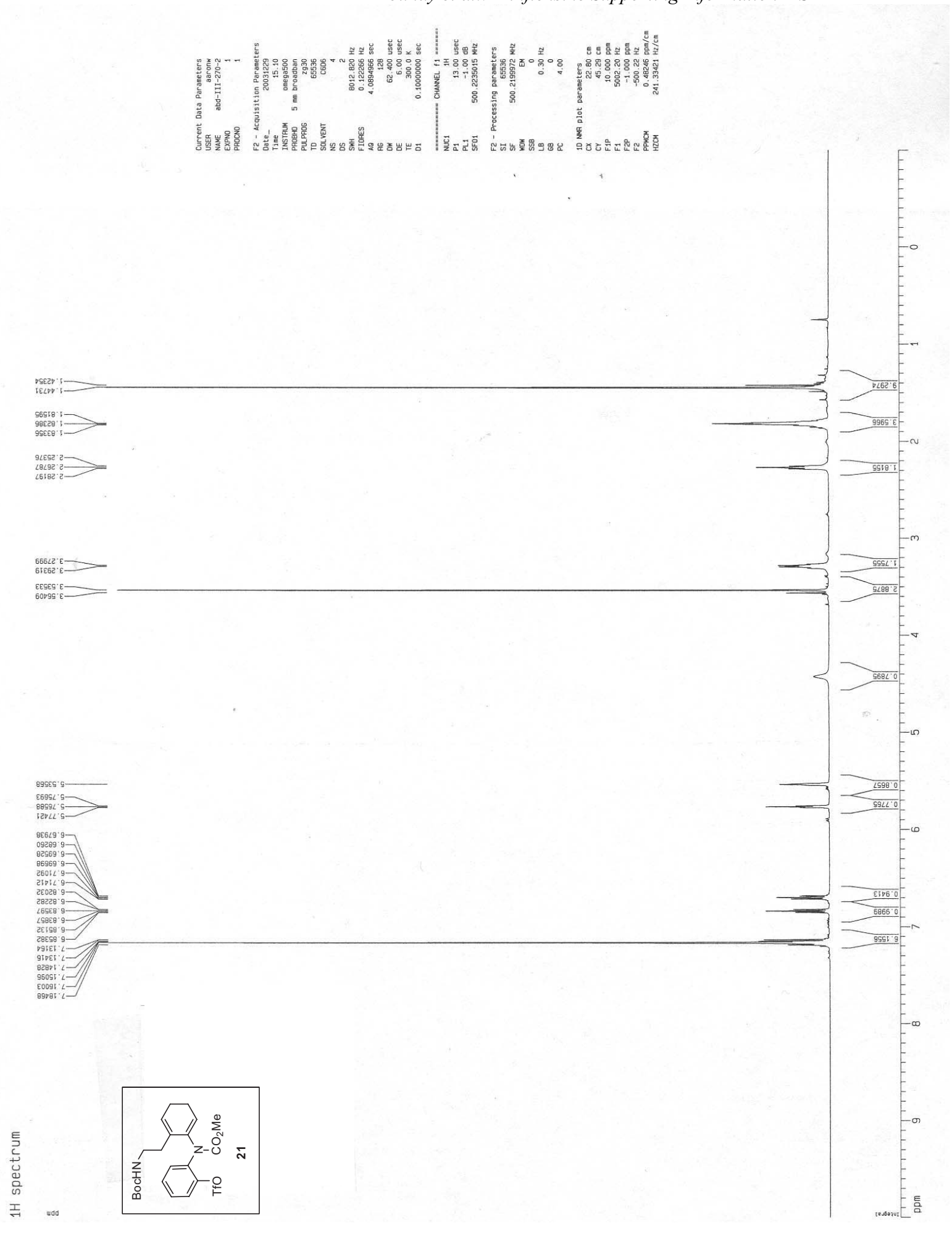

13C spectrum with 1H decoupling
TIPSO
CO2Me
16
Current Data Parameters
USER dounay
NAME ajd-IV-57
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040218
Time 14.40
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT c6d6
NS 276
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ========
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.66980 Hz/cm
193.62
155.36
153.11
143.44
142.42
134.78
130.84
128.70
128.59
128.50
128.40
128.30
128.20
122.00
119.33
52.89
39.00
25.87
23.06
18.61
13.79
ppm

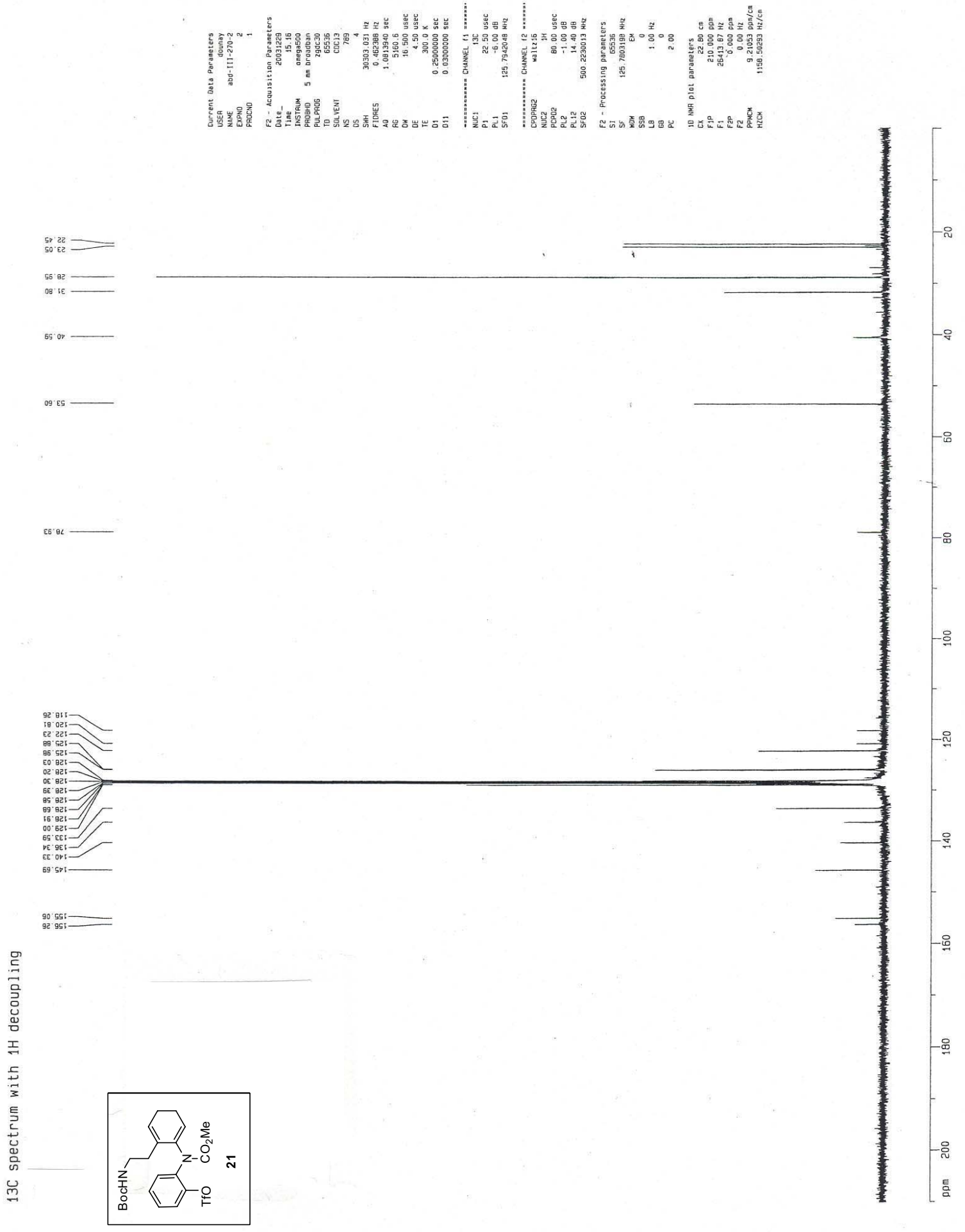

1H spectrum
TfO
N
CO2Me
TIPSO
18
ppm

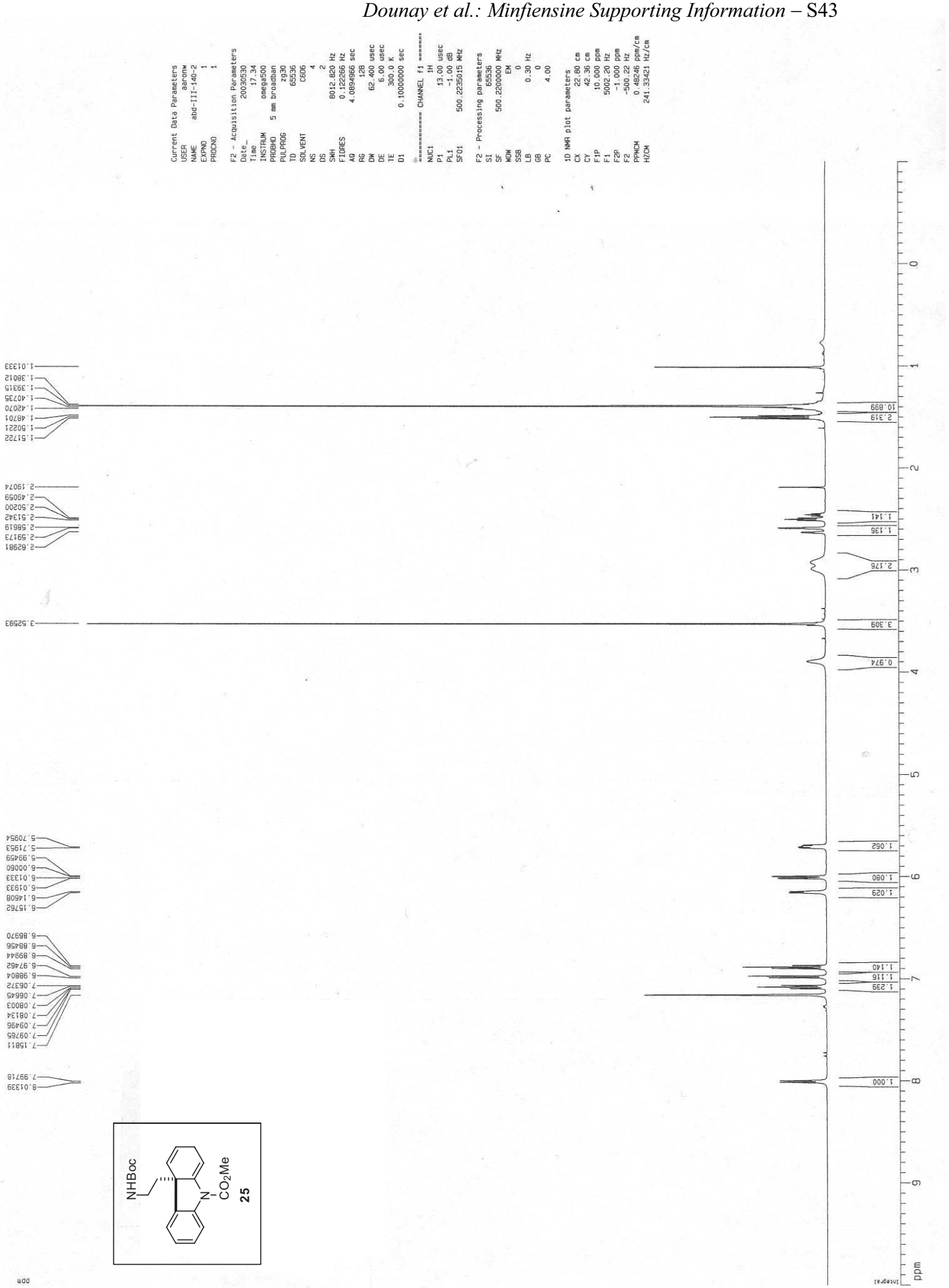

13C spectrum with 1H decoupling
Current Data Parameters
USER dounay
NAME abd-III-258XX
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040116
Time 13.23
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 30303.031 Hz
FIDRES 0.462398 Hz
AQ 1.0813940 sec
RG 2580.3
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ========
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SF01 125.7942048 MHz
========== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SF02 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.68 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67029 Hz/cm
13.42
18.15
21.49
21.58
52.80
121.86
127.78
127.97
128.17
128.67
129.97
143.61
152.91
ppm
TfO
N
CO2Me
TIPSO
18

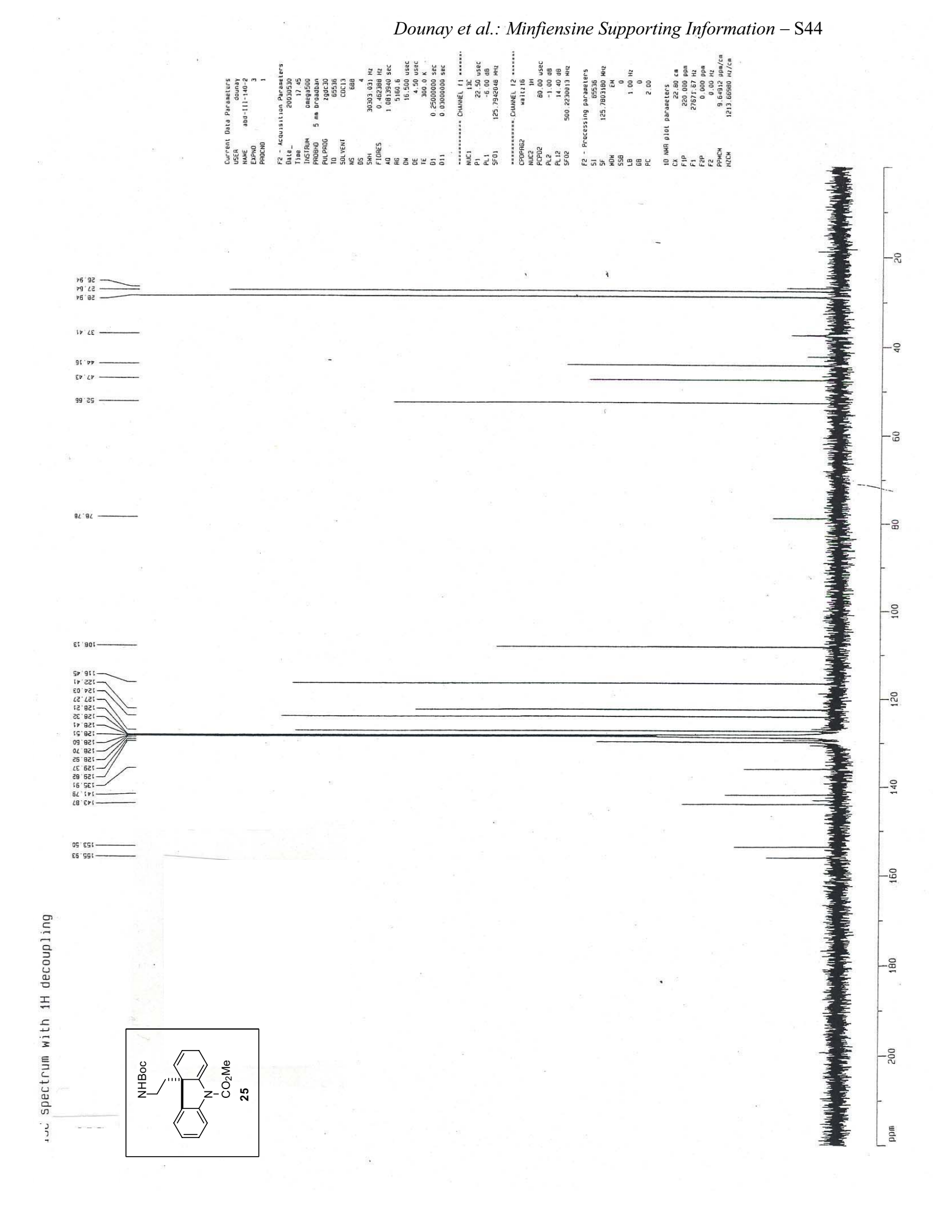

Current Data Parameters
USER dounay
NAME abd-III-200-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030724
Time 13.22
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 181
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8334988 MHz
F2 - Processing parameters
SI 65536
SF 499.8300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.500 ppm
F1 5248.21 Hz
F2P -0.500 ppm
F2 -249.91 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.14606 Hz/cm
0.75170
1.13303
1.14743
1.20804
1.27233
1.28665
1.30070
1.33232
1.38081
1.41976
1.44986
1.85016
2.40673
3.35525
3.50598
5.56167
5.77717
6.74128
6.75655
6.77144
6.84433
6.85984
6.92200
6.93721
6.95276
7.15034
7.20509
7.22080
67.937
3.087
8.946
1.570
1.830
1.659
14.973
ppm
1H spectrum
BocHN
TIPSO
N
CO2Me
20

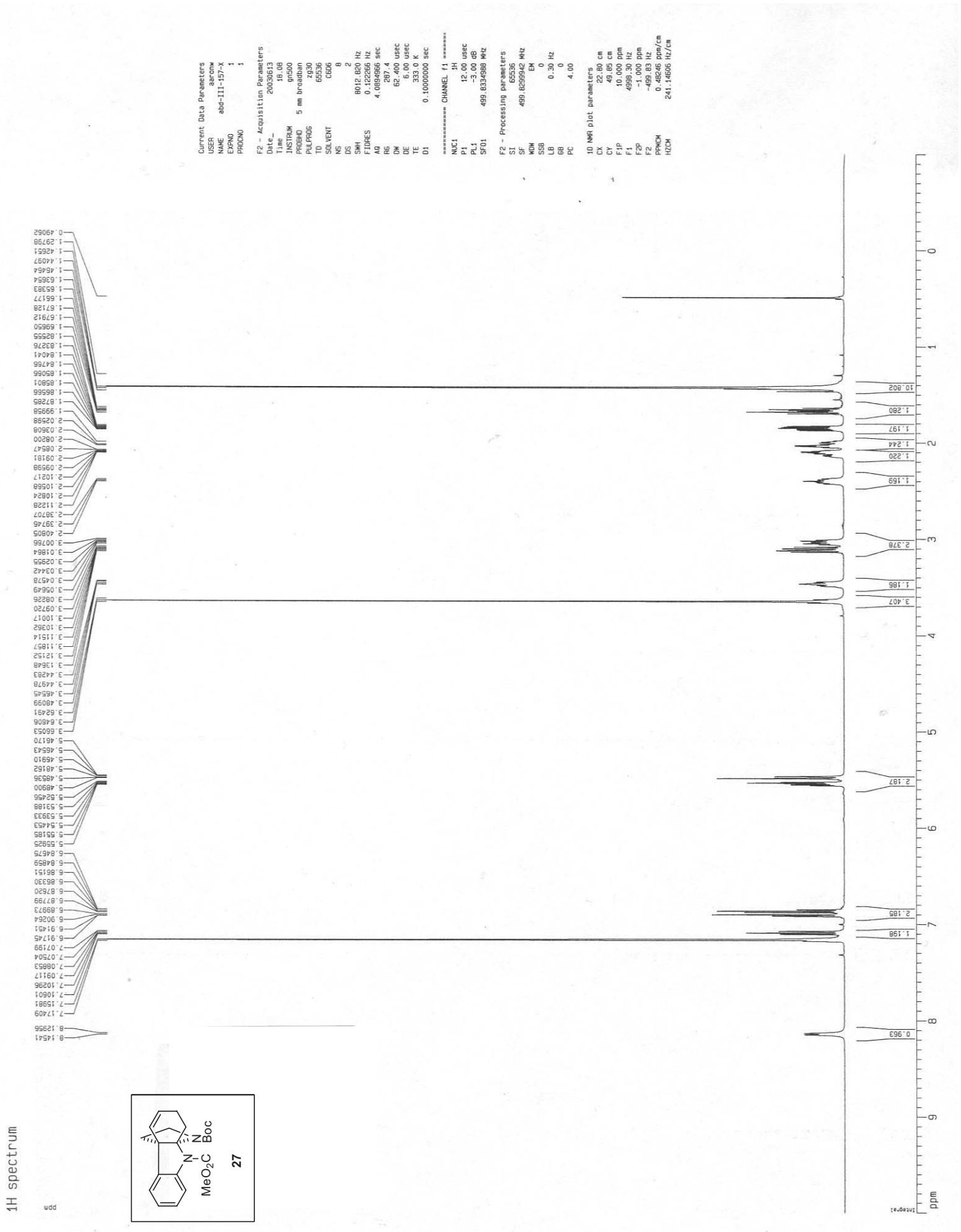

13C spectrum with 1H decoupling
BocHN
TIPSO
N
CO2Me
20
ppm

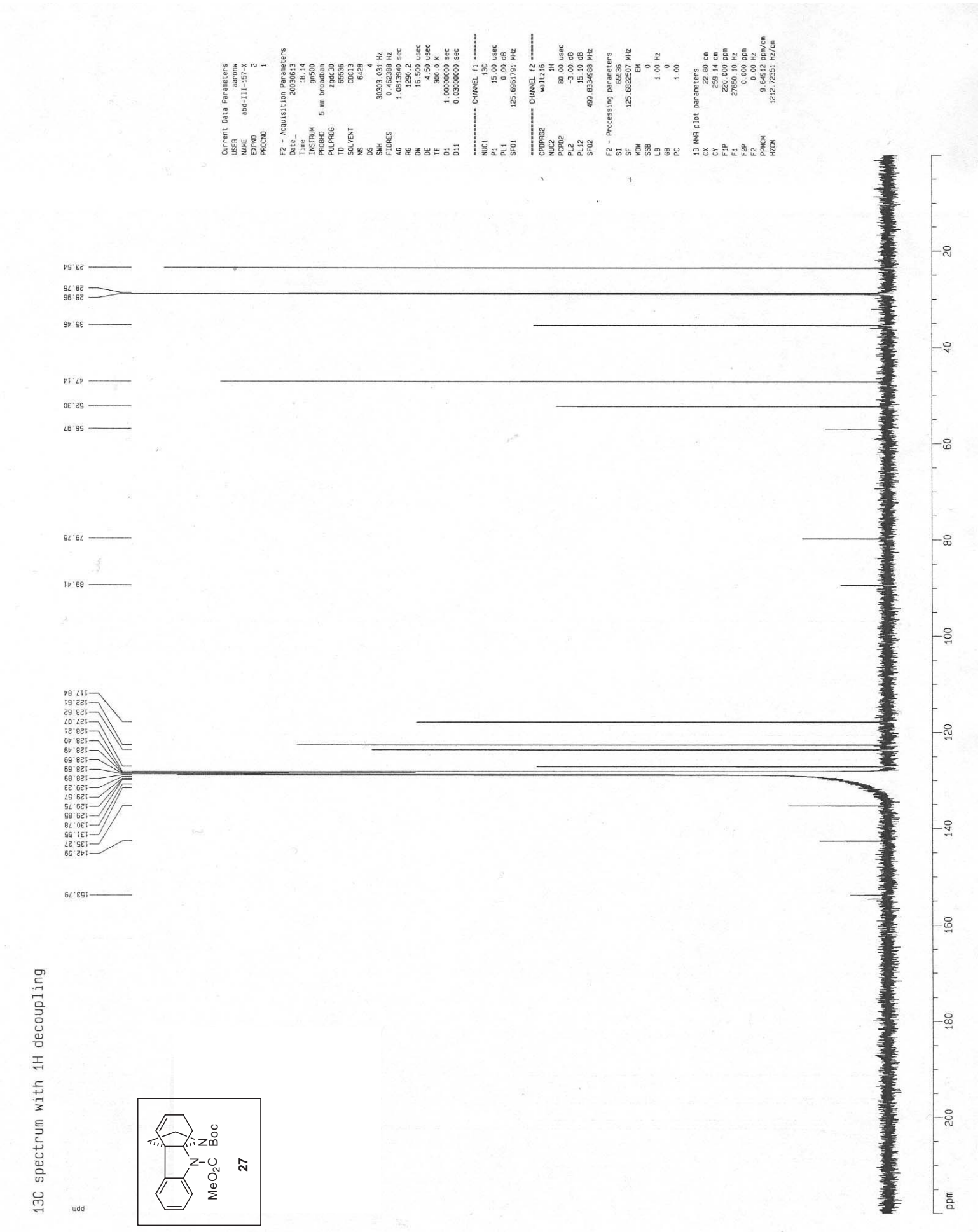

1H spectrum
BocHN
TfO
N
CO2Me
21
ppm


13C spectrum with 1H decoupling
Current Data Parameters
USER dounay
NAME abd-III-270-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031220
Time 15.16
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803198 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 210.000 ppm
F1 26413.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.50293 Hz/cm
22.45
23.05
28.95
31.80
40.59
53.60
78.93
118.26
120.81
122.23
125.88
125.98
128.03
128.20
128.30
128.39
128.58
128.68
128.91
129.00
133.59
136.34
140.33
145.69
155.06
156.26
20
40
60
80
100
120
140
160
180
200
ppm
BocHN
TfO
N
CO2Me
21


Current Data Parameters
USER aaronw
NAME abd-III-140-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030530
Time 17.34
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT C606
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SF01 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 42.36 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
1.01333
1.38012
1.39315
1.40735
1.42070
1.48701
1.50221
1.51722
2.19074
2.49059
2.50200
2.51342
2.58619
2.59173
2.62981
3.52593
5.70954
5.71953
5.99459
6.00060
6.01333
6.01933
6.14608
6.15762
6.86970
6.88456
6.89944
6.97462
6.98804
7.06372
7.06645
7.08003
7.08134
7.09496
7.09765
7.15811
7.99718
8.01339
10.899
2.319
1.141
1.136
2.175
3.309
0.974
1.062
1.080
1.029
1.140
1.116
1.239
1.000
NHBoc
CO2Me
25
ppm
integral


Current Data Parameters
USER dounay
NAME abd-III-140-2
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030530
Time 17.45
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 688
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SF01 125.7942048 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SF02 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803180 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.66980 Hz/cm
26.94
27.64
28.94
37.41
44.16
47.43
52.66
78.78
108.13
116.45
122.41
124.03
127.27
128.21
128.32
128.41
128.51
128.60
128.70
128.92
129.37
129.82
135.91
143.87
153.50
155.93
20
40
60
80
100
120
140
160
180
200
ppm
spectrum with 1H decoupling
NHBoc
N
CO2Me
25

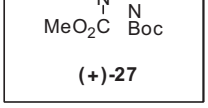

1H spectrum
ppm
integral
MeO2C
Boc
N
27


13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME abd-III-157-X
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030613
Time 18.14
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 6428
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 1290.2
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961791 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz
F2 - Processing parameters
SI 65536
SF 125.6822507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 259.14 cm
F1P 220.000 ppm
F1 27650.10 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1212.72351 Hz/cm
23.54
28.75
28.96
35.46
47.14
52.30
56.97
79.75
89.41
117.84
122.61
123.62
127.07
128.21
128.40
128.49
128.59
128.69
128.89
129.23
129.57
129.75
129.85
130.78
131.55
135.27
142.59
153.79
ppm
20
40
60
80
100
120
140
160
180
200
MeO_2C
N
N
Boc
27

Data File C:\HPCHEM\1\DATA\ABD1\10020301.D Sample Name: abd-iii-157

~~rac~~aminal

```
==========================================================================
Injection Date  : 10/2/03 6:47:16 PM            Seq. Line :   1
Sample Name     : abd-iii-157                    Location : Vial 1
Acq. Operator   : abd                               Inj :   1
                                                 Inj Volume : 5 µl
Sequenc                          l\SEQUENCE\DEF_LC.S
Method                           \ABD1.M
Last ch                          )7:35 PM by abd
                                 after loading)
```



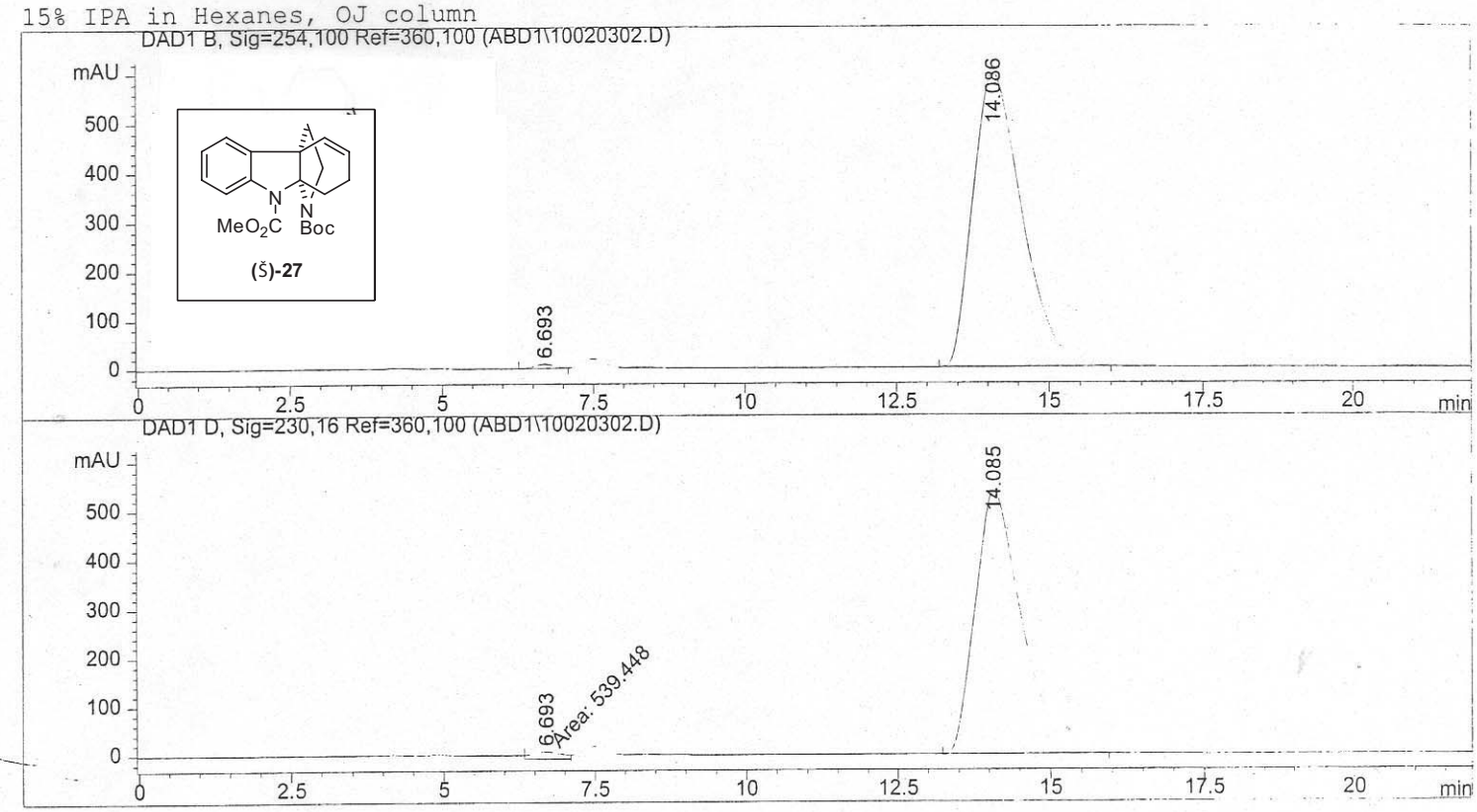
(+)-27


```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,100 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.708 BB    0.3724  193.30757    8.28051  50.1980
   2  14.437 PP    0.7832  191.78249    3.66048  49.8020

Totals :                   385.09006   11.94099

 Results obtained with enhanced integrator!

Signal 2: DAD1 D, Sig=230,16 Ref=360,100
```

abd-III-270
t-bu Pfaltz ligand
98:2 Hex/IPA, OD-H Column, 1.0 ml/min

Injection Date	: 10/2/03 7:10:29 PM	Seq. Line	: 2
Sample Name	: abd-III-270	Location	: Vial 2
Acq. Operator	: abd	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\METHODS\ABD1.M		
Last changed	: 10/2/03 7:09:28 PM by abd (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\SK1.M		
Last changed	: 2/26/04 4:35:08 PM by AMY (modified after loading)		

15% IPA in Hexanes, OJ column



Area Percent Report

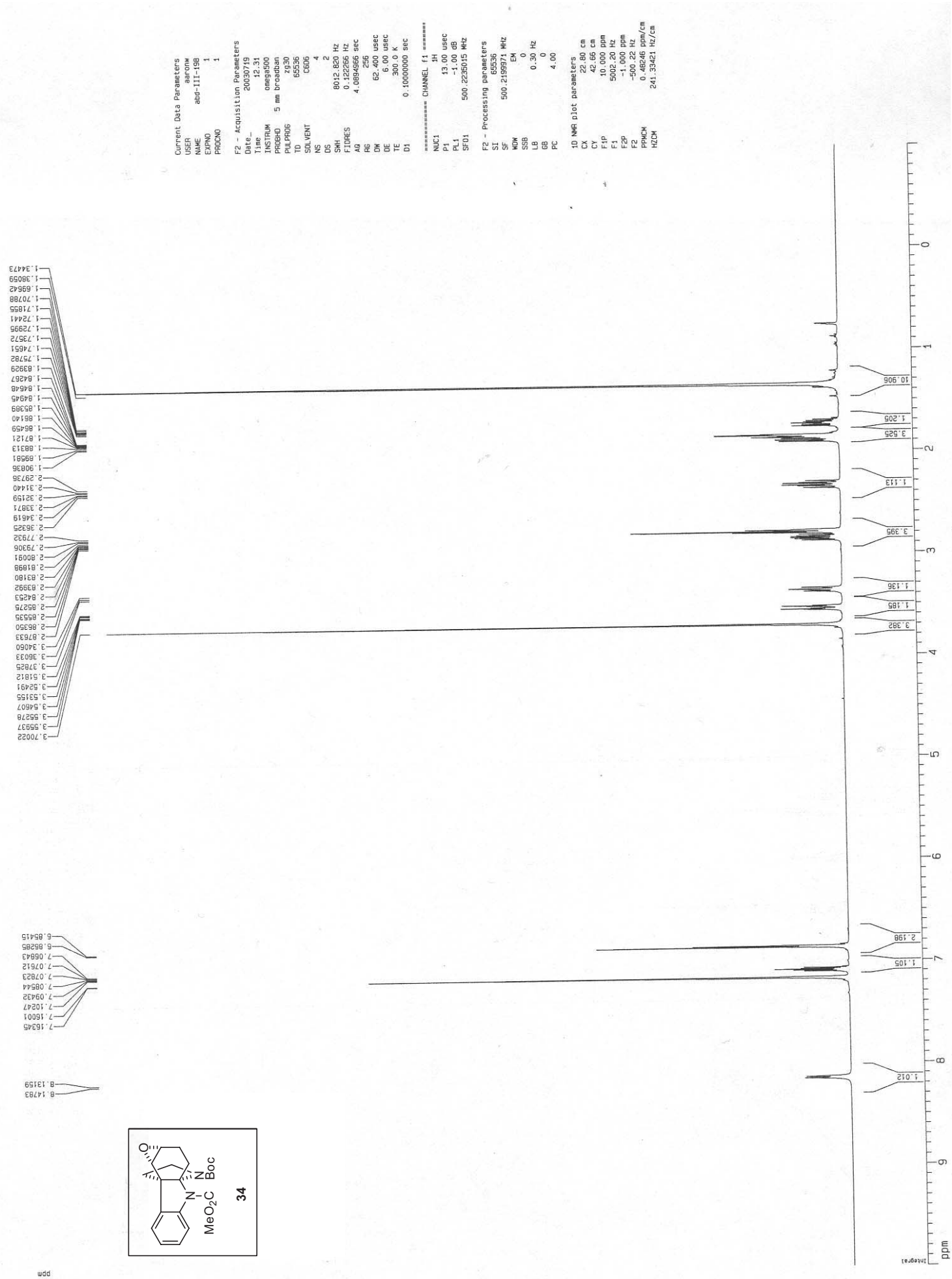
Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

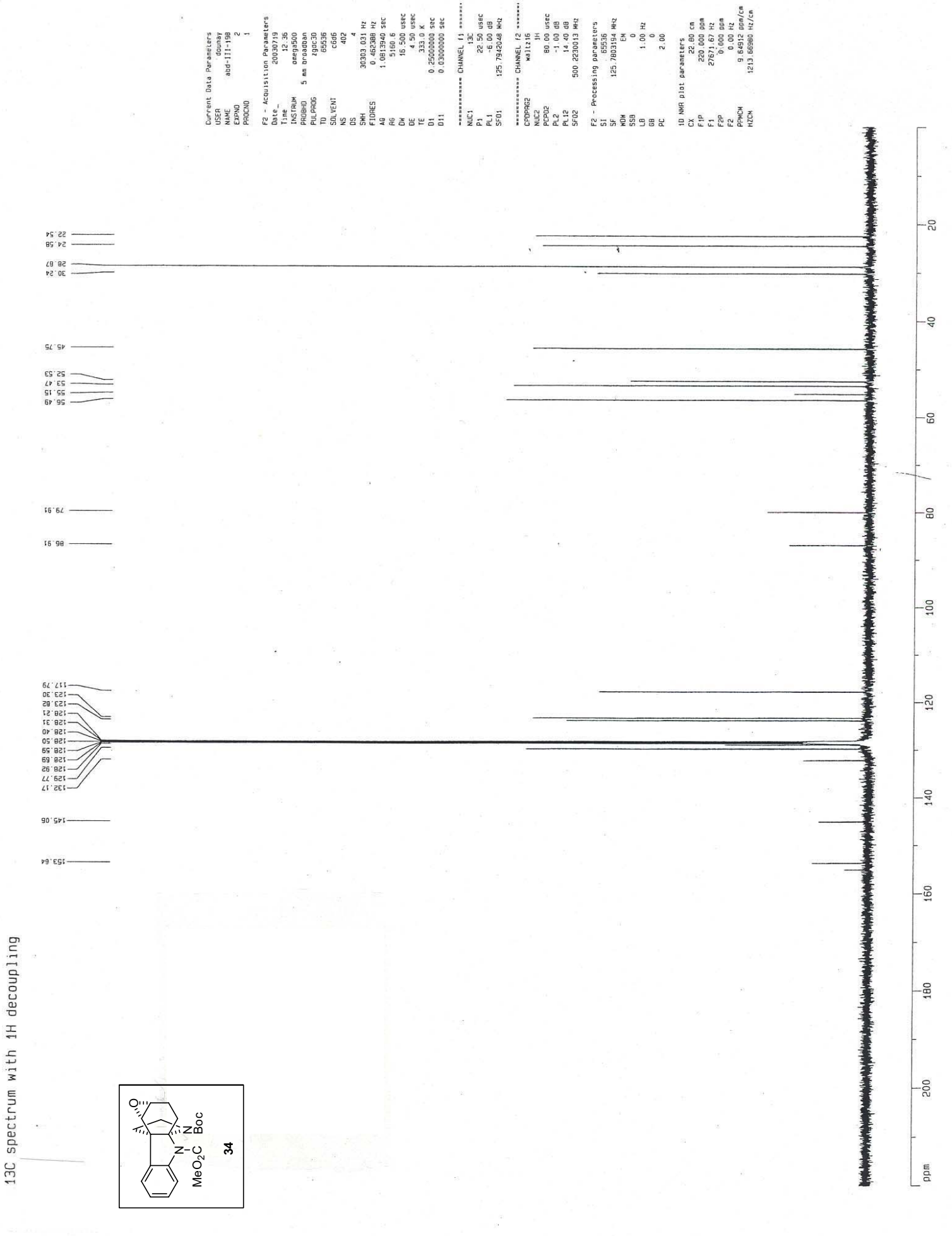
Use Multiplier & Dilution Factor with ISTDs

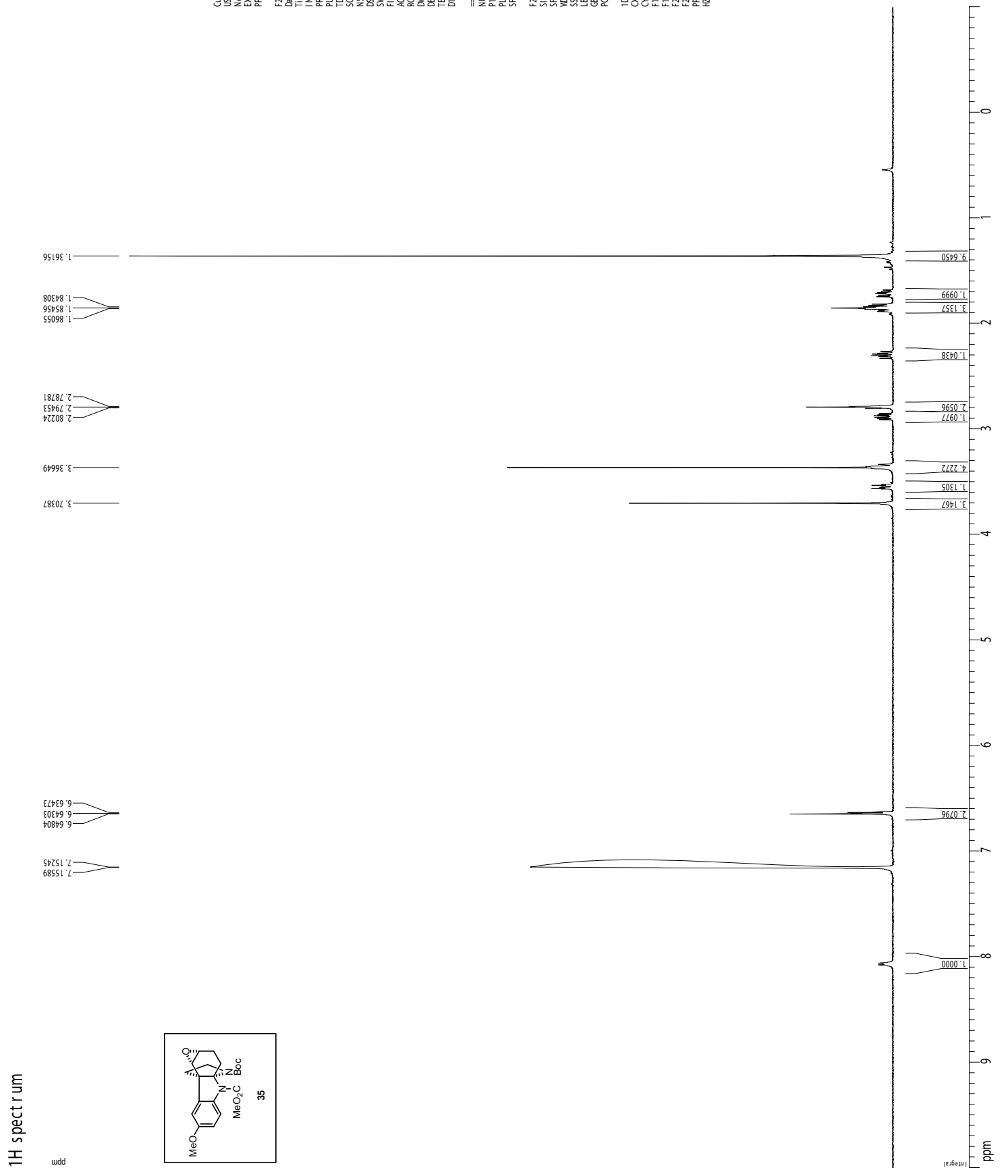
Signal 1: DAD1 B, Sig=254,100 Ref=360,100

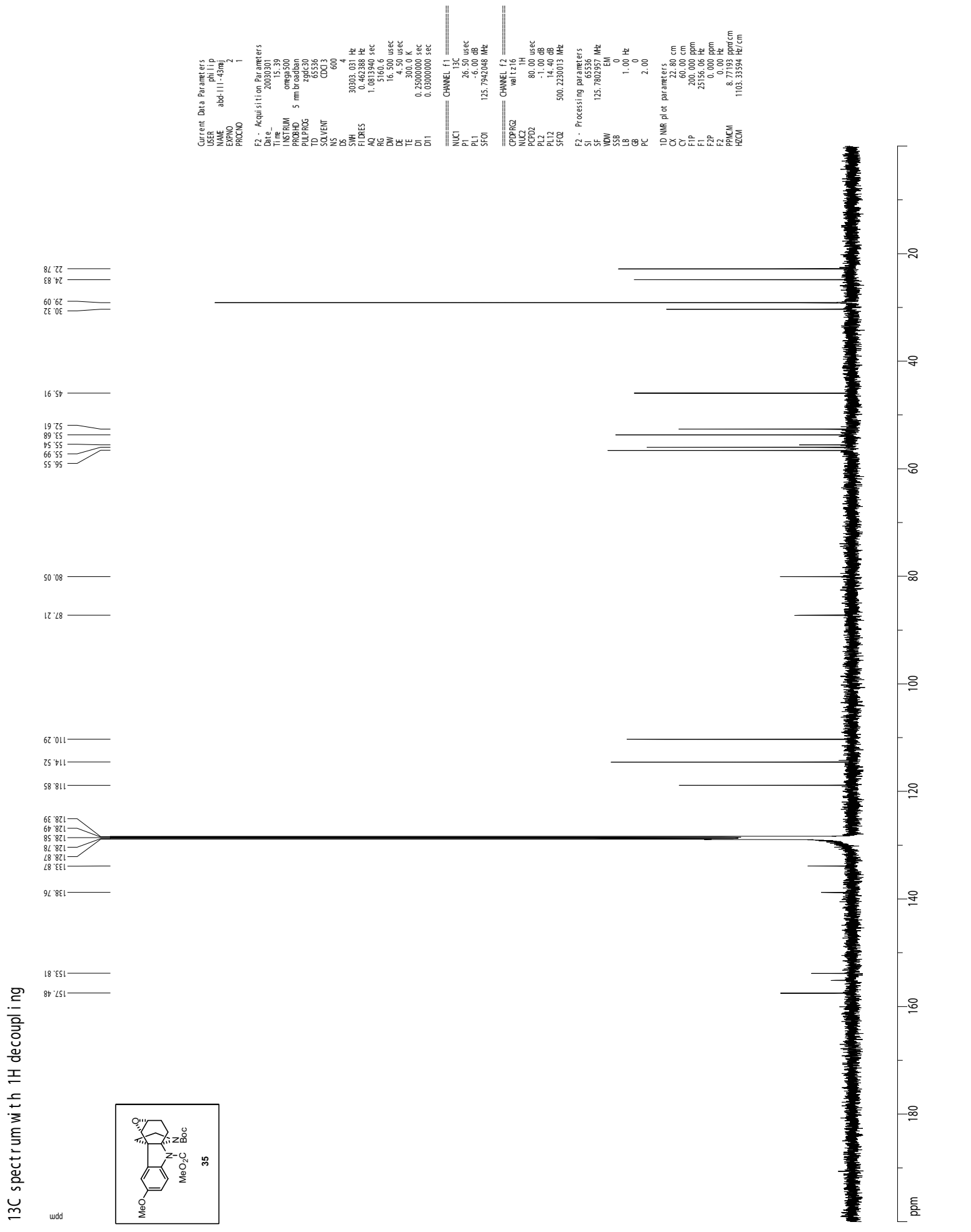
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.693	PV	0.3618	180.33255	8.09739	0.5394
2	14.086	BB	0.8746	3.32488e4	598.03790	99.4606
Totals :				3.34291e4	606.13529	

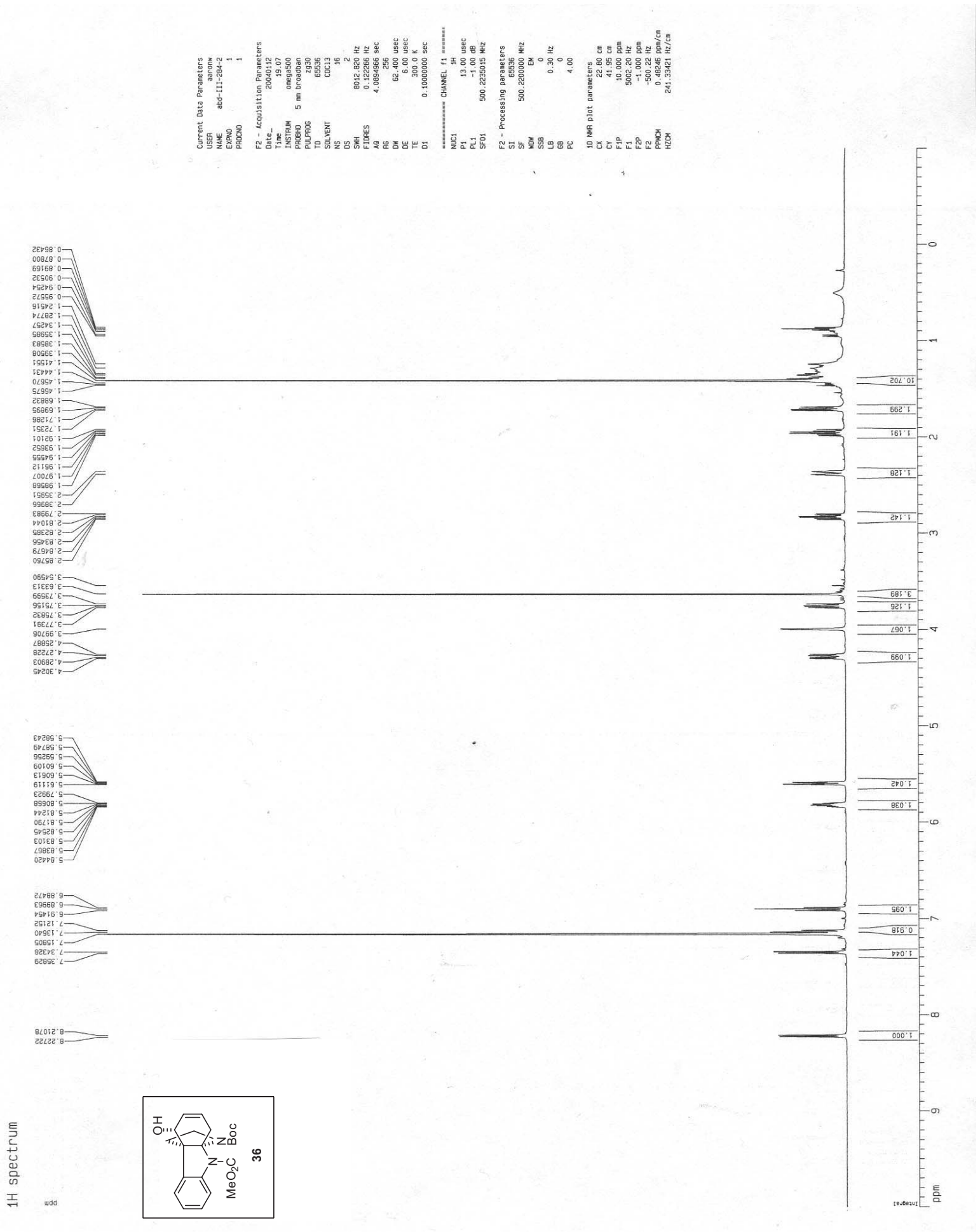
Results obtained with enhanced integrator!

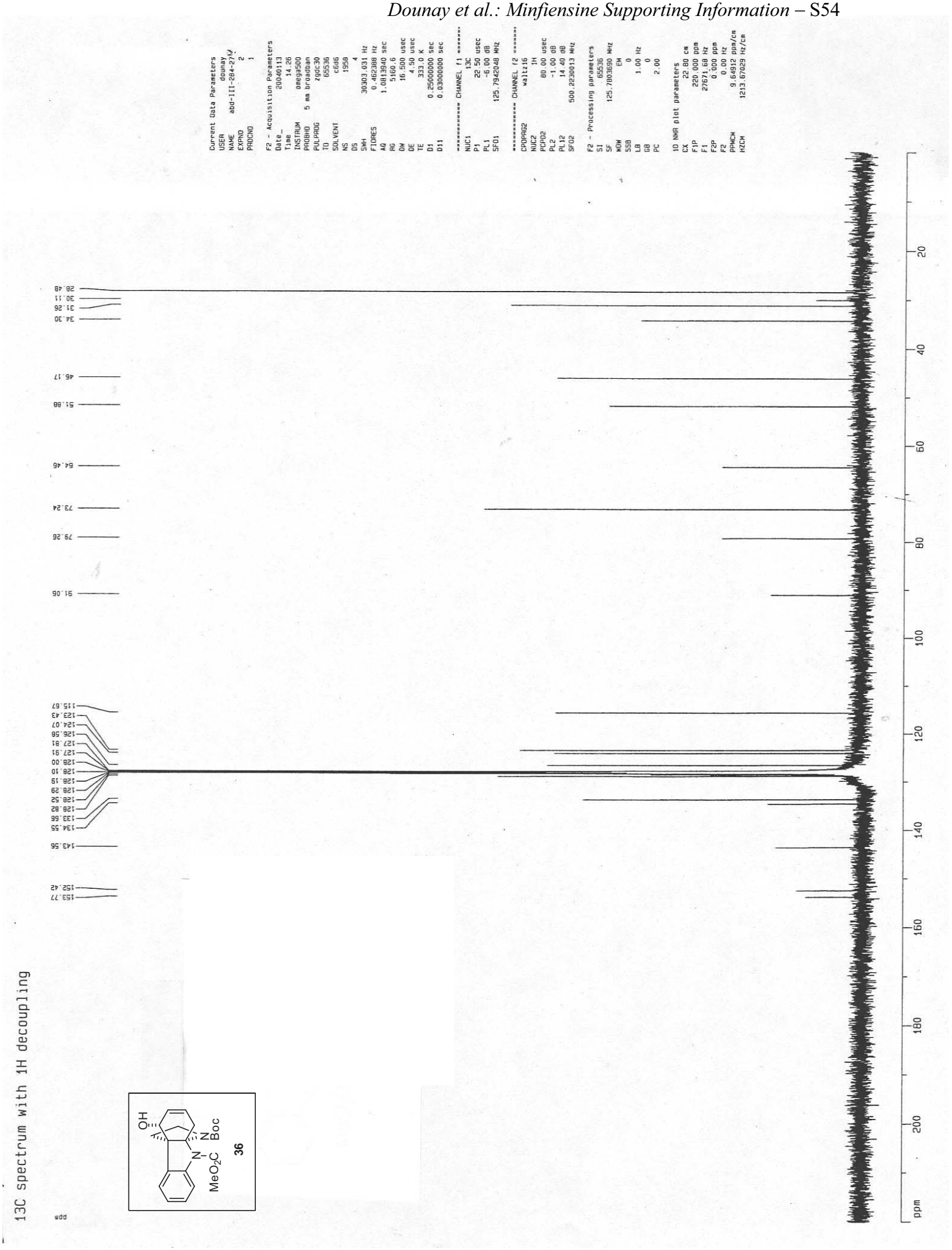

Current Data Parameters
USER aaronw
NAME abd-III-198
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030719
Time 12.31
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2199971 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 42.66 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
1.34473
1.38059
1.69642
1.70788
1.71855
1.72441
1.72995
1.73572
1.74651
1.75782
1.83929
1.84267
1.84648
1.84945
1.85389
1.86140
1.86459
1.87121
1.88313
1.89581
1.90836
2.29736
2.31440
2.32159
2.33871
2.34619
2.36325
2.77932
2.79306
2.80091
2.81898
2.83180
2.83992
2.84253
2.85275
2.85535
2.86350
2.87633
3.34060
3.36033
3.37825
3.51812
3.52491
3.53155
3.54607
3.55278
3.55937
3.70022
6.85415
6.86285
7.06843
7.07612
7.07823
7.08544
7.09432
7.10247
7.16001
7.16345
8.13159
8.14783
10.905
1.205
3.525
1.113
3.395
1.136
1.185
3.382
2.198
1.105
1.012
ppm
integral
MeO2C
N
Boc
34

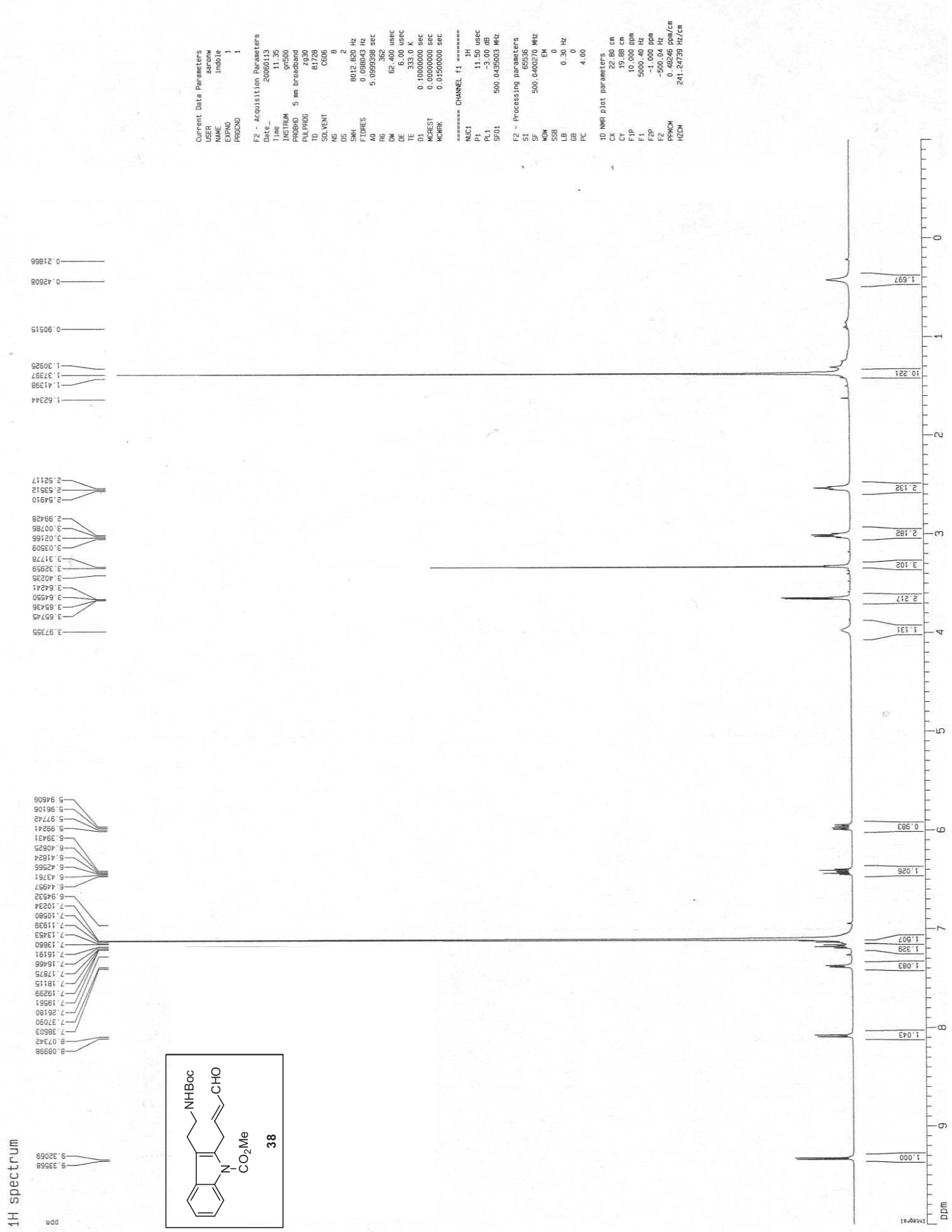

Current Data Parameters
USER dounay
NAME abd-III-198
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030719
Time 12.36
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT c606
NS 402
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 =======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803194 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.66980 Hz/cm
22.54
24.58
28.87
30.24
45.75
52.53
53.47
55.15
56.49
79.91
86.91
117.79
123.30
123.82
128.21
128.31
128.40
128.50
128.59
128.69
128.92
129.77
132.17
145.06
153.64
20
40
60
80
100
120
140
160
180
200
ppm
13C spectrum with 1H decoupling
MeO2C
Boc
34

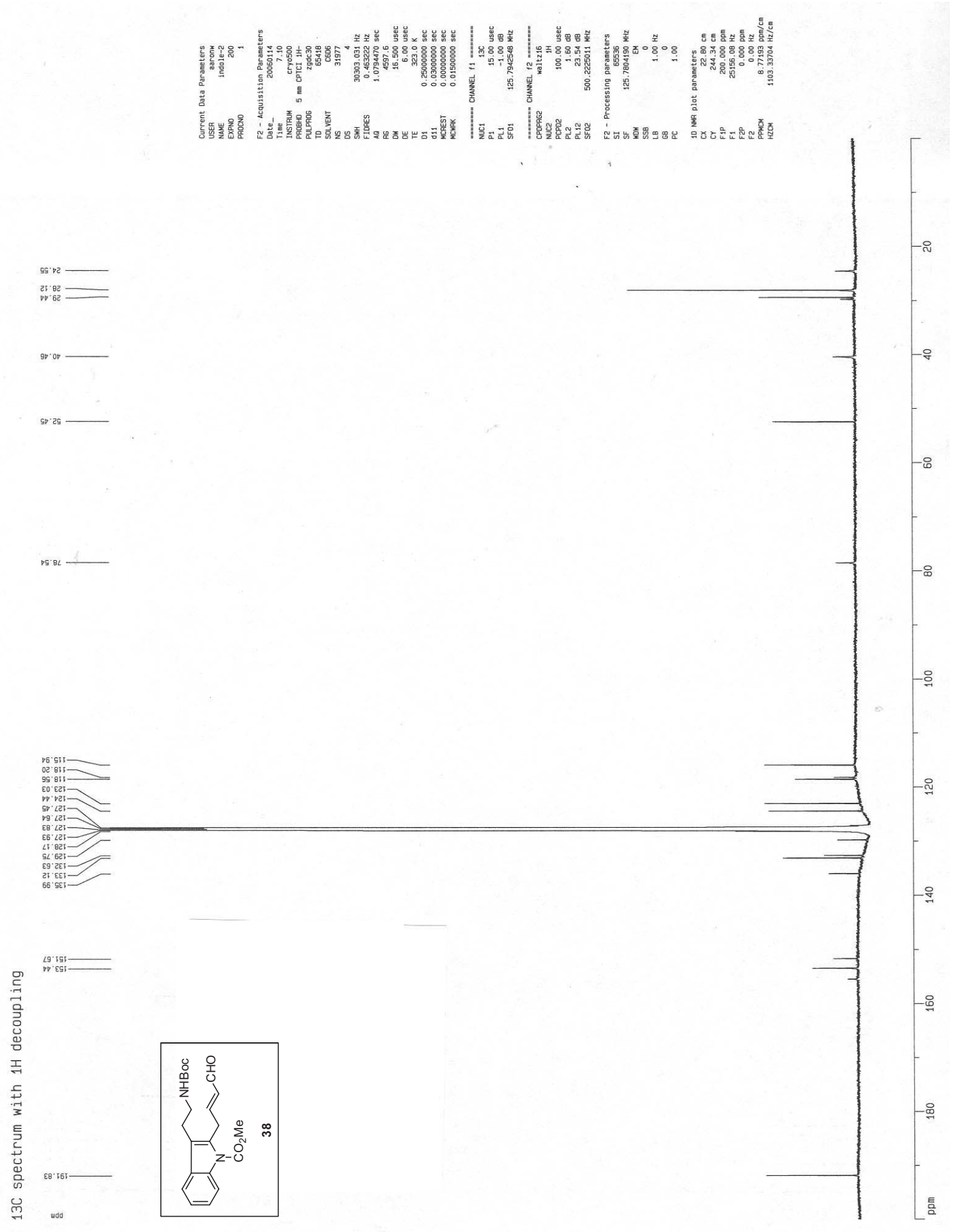

1H spectrum
Current Data Parameters
USER philip
NAME abd-III-43mej2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030303
Time 11.36
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
CHANNEL f1 1H
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200010 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
1.36156
1.84308
1.85456
1.86055
2.78781
2.79453
2.80224
3.36649
3.70387
6.63473
6.64303
6.64804
7.15245
7.15589
9.6450
1.0999
3.1357
1.0438
2.0596
1.0977
4.2272
1.1305
3.1467
2.0796
1.0000
MeO
MeO2C
Boc
N
O
35
ppm
Integral

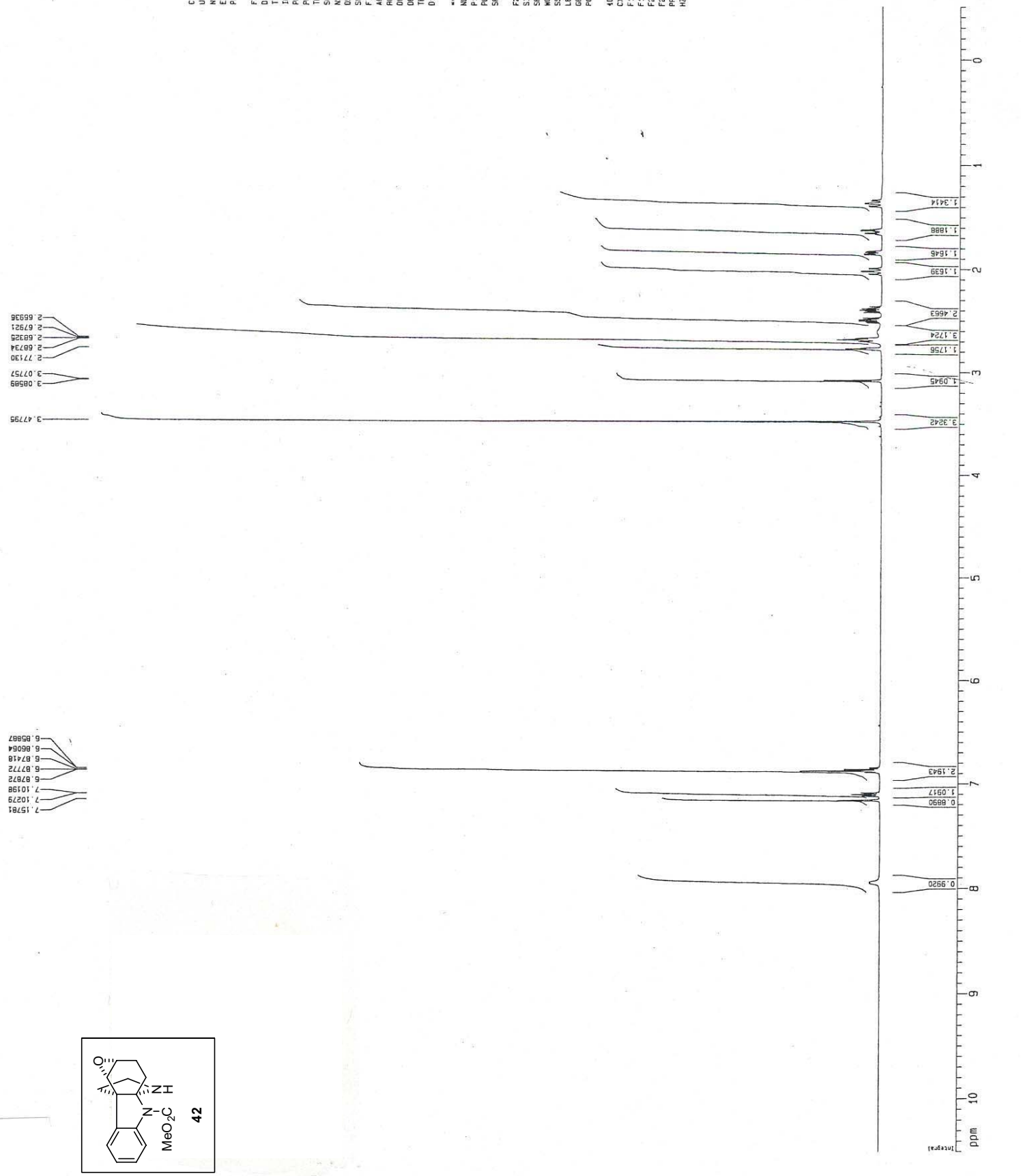

13C spectrum with 1H decoupling
Current Data Parameters
USER philip
NAME abd-III-43mej
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030301
Time 15.39
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 26.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7802957 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 60.00 cm
F1P 200.000 ppm
F1 25156.06 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 1103.33594 Hz/cm
22.78
24.83
29.09
30.32
45.91
52.61
53.68
55.54
55.99
56.55
80.05
87.21
110.29
114.52
118.85
128.39
128.49
128.58
128.78
128.87
133.87
138.76
153.81
157.48
ppm
20
40
60
80
100
120
140
160
180
ppm
MeO
MeO2C
N
Boc
O
35


1H spectrum
ppm
integral
OH
N
N
Boc
MeO2C
36


13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER dounay
NAME abd-III-284-27
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040113
Time 14.26
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT cdd6
NS 1958
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942040 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.68 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67029 Hz/cm
28.48
30.11
31.26
34.30
46.17
51.88
54.46
73.24
79.26
91.06
115.67
123.43
124.07
126.58
127.81
127.91
128.00
128.10
128.19
128.29
128.52
128.82
133.66
134.55
143.56
152.42
153.77
OH
N
N
Boc
MeO2C
36

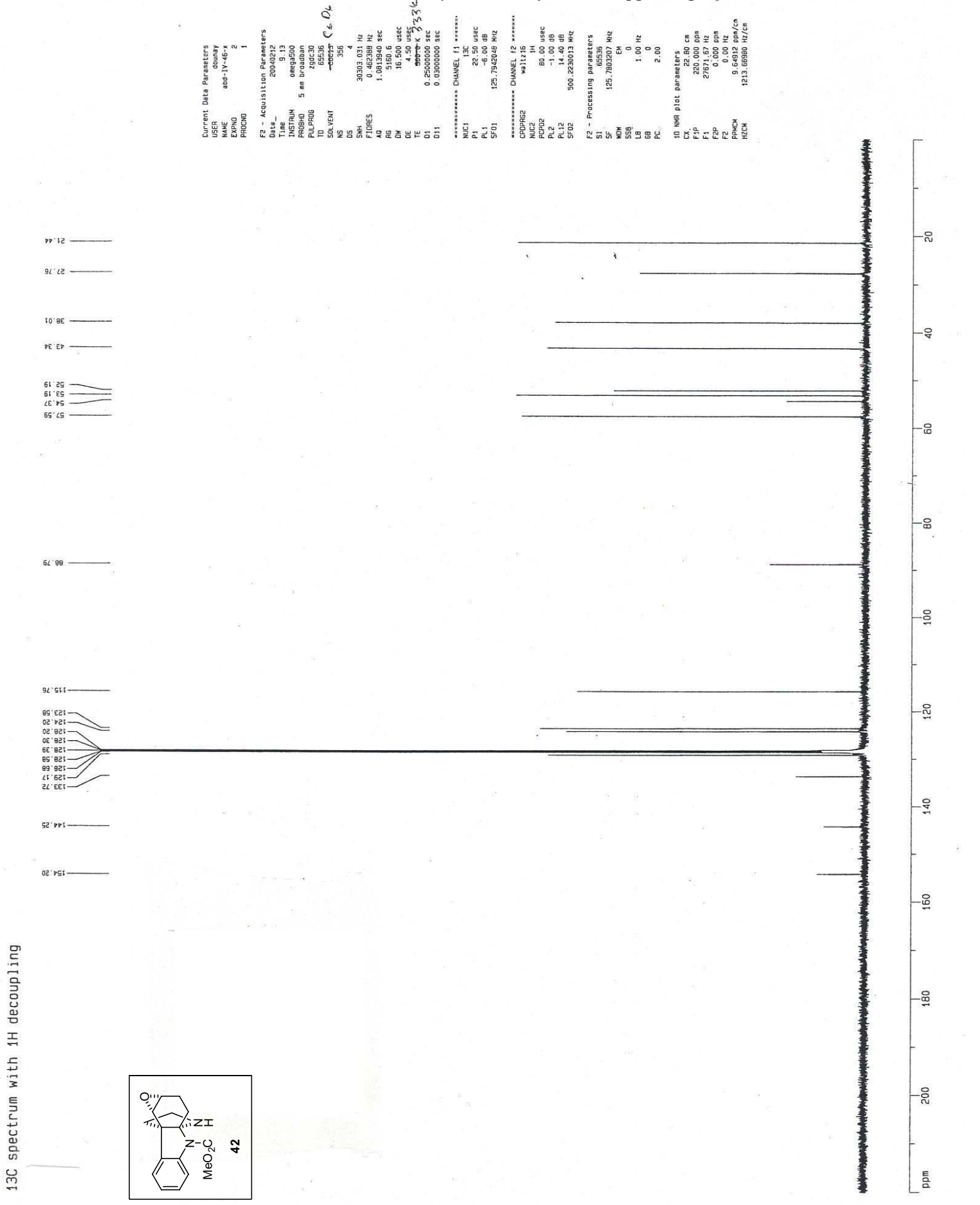

1H spectrum
Current Data Parameters
USER aaronw
NAME indole
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060113
Time 11.35
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 362
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SF01 500.0435003 MHz
F2 - Processing parameters
SI 65536
SF 500.0400270 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 19.88 cm
F1P 10.000 ppm
F2P -1.000 ppm
F1 5000.40 Hz
F2 -500.04 Hz
PPMCM 0.40246 ppm/cm
HZCM 241.24739 Hz/cm
NHBoc
CHO
N
CO2Me
38
ppm
integral

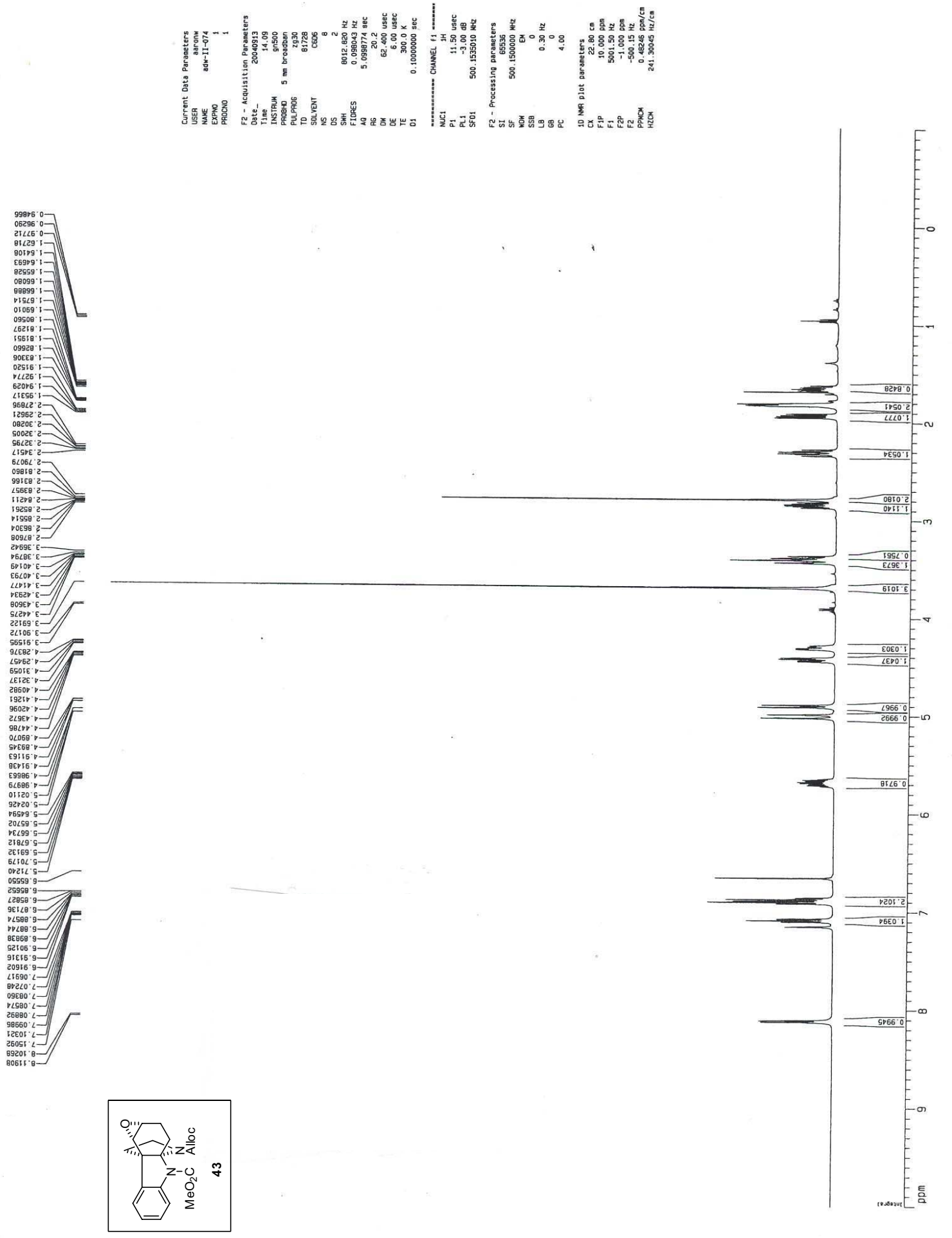

13C spectrum with 1H decoupling
ppm
NHBoc
CHO
N
CO2Me
38
Current Data Parameters
USER aaronw
NAME indole-2
EXPNO 200
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060114
Time 7.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 31977
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 4597.6
DW 16.500 usec
DE 6.00 usec
TE 323.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz
F2 - Processing parameters
SI 65536
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 244.34 cm
F1P 200.000 ppm
F1 25156.08 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 1103.33704 Hz/cm
24.55
28.12
29.44
40.46
52.45
78.54
115.94
118.20
118.56
123.03
124.44
127.45
127.64
127.83
127.93
128.17
129.75
132.63
133.12
135.99
151.67
153.44
191.83

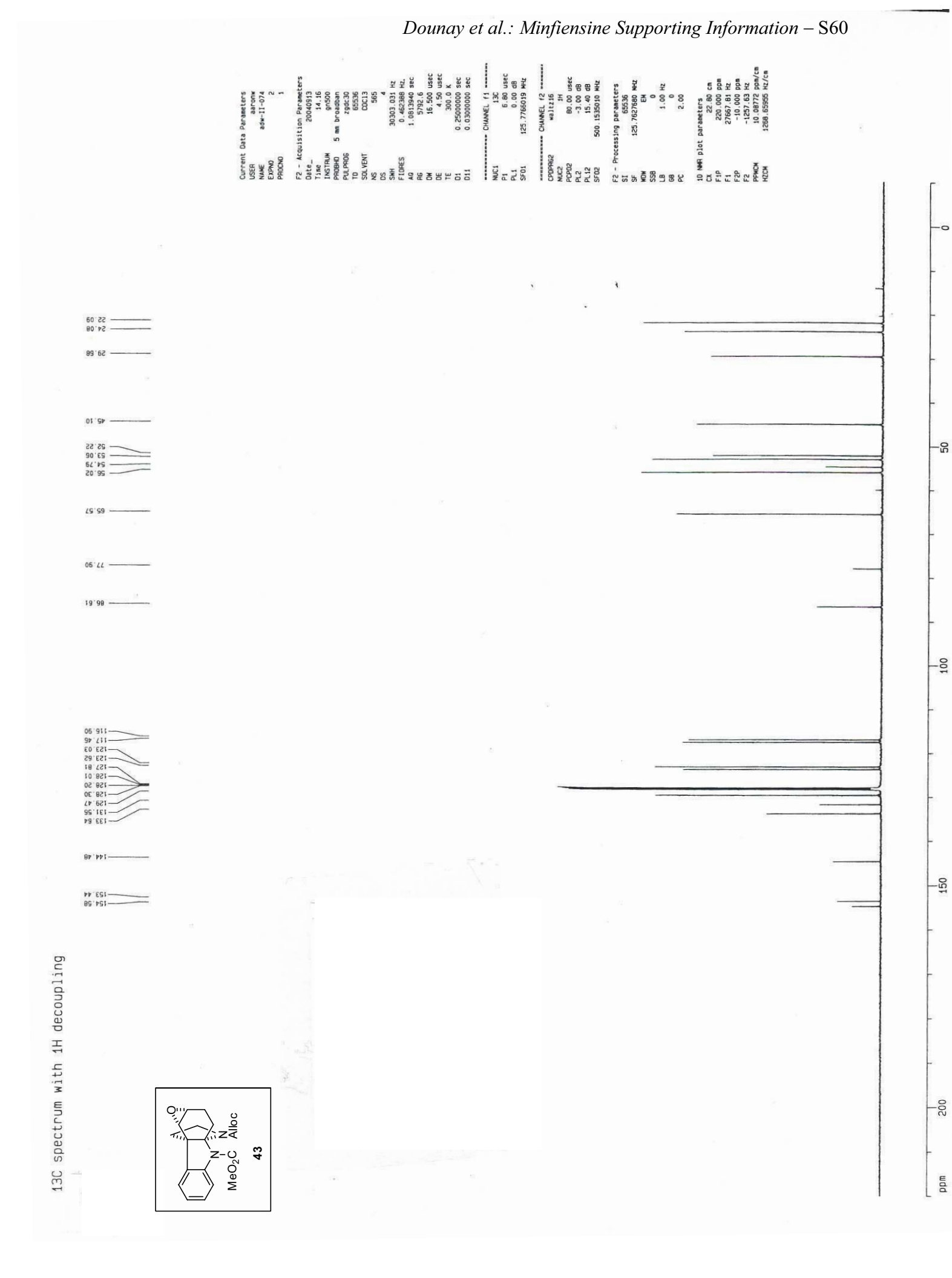

Current Data Parameters
USER dounay
NAME abd-IV-46-x
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040212
Time 9.08
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 4
DS 2
SWH 6012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0398774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
========== CHANNEL f1 =======
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
2.66936
2.67921
2.68325
2.68734
2.77130
3.07757
3.08589
3.47795
6.85887
6.86064
6.87418
6.87772
6.87872
7.10198
7.10279
7.15781
1.3414
1.1888
1.1646
1.1539
2.4663
3.1724
1.1756
1.0945
3.3242
2.1943
1.0917
0.8890
0.9920
ppm
Integral

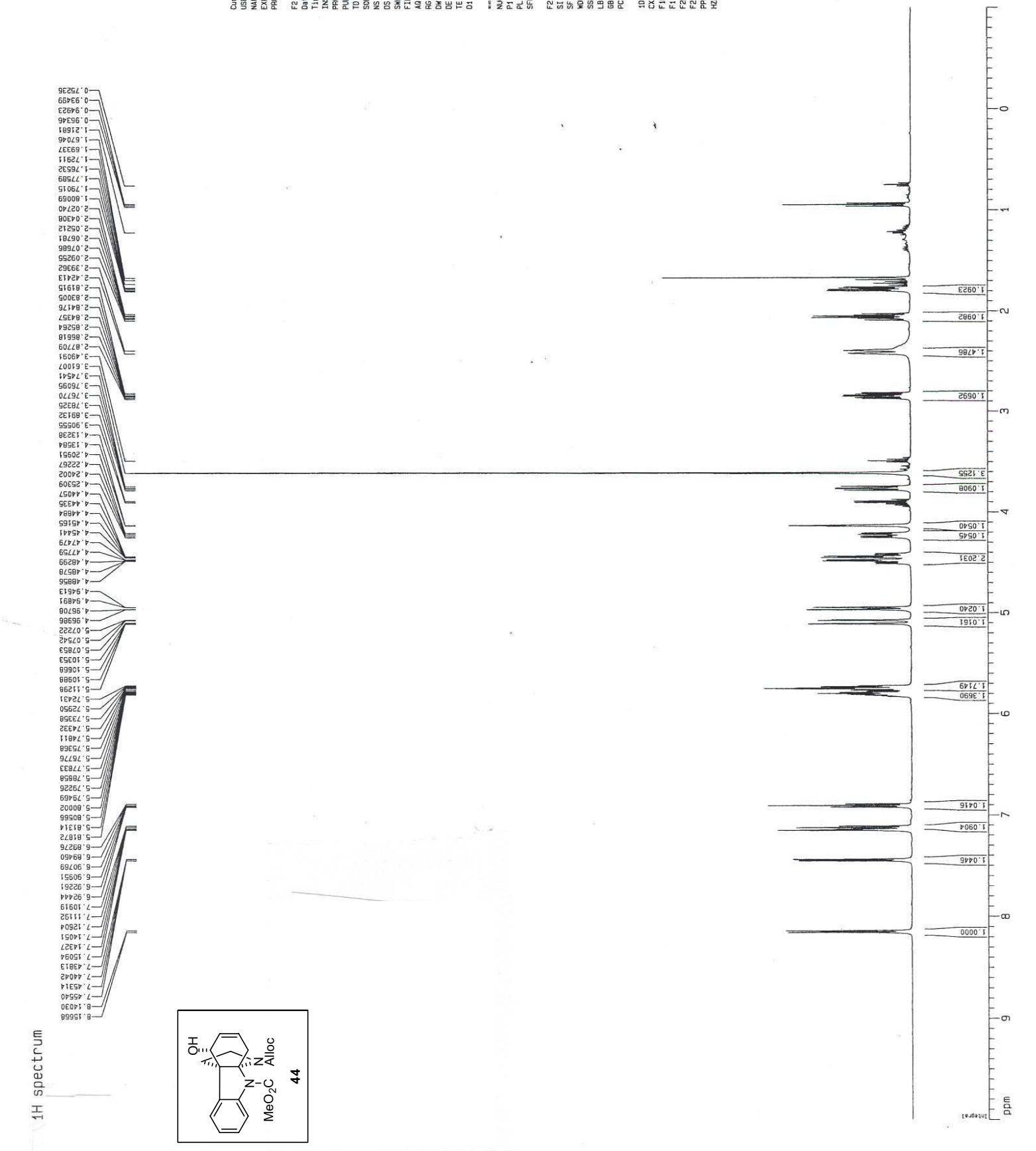
^{1}H spectrum

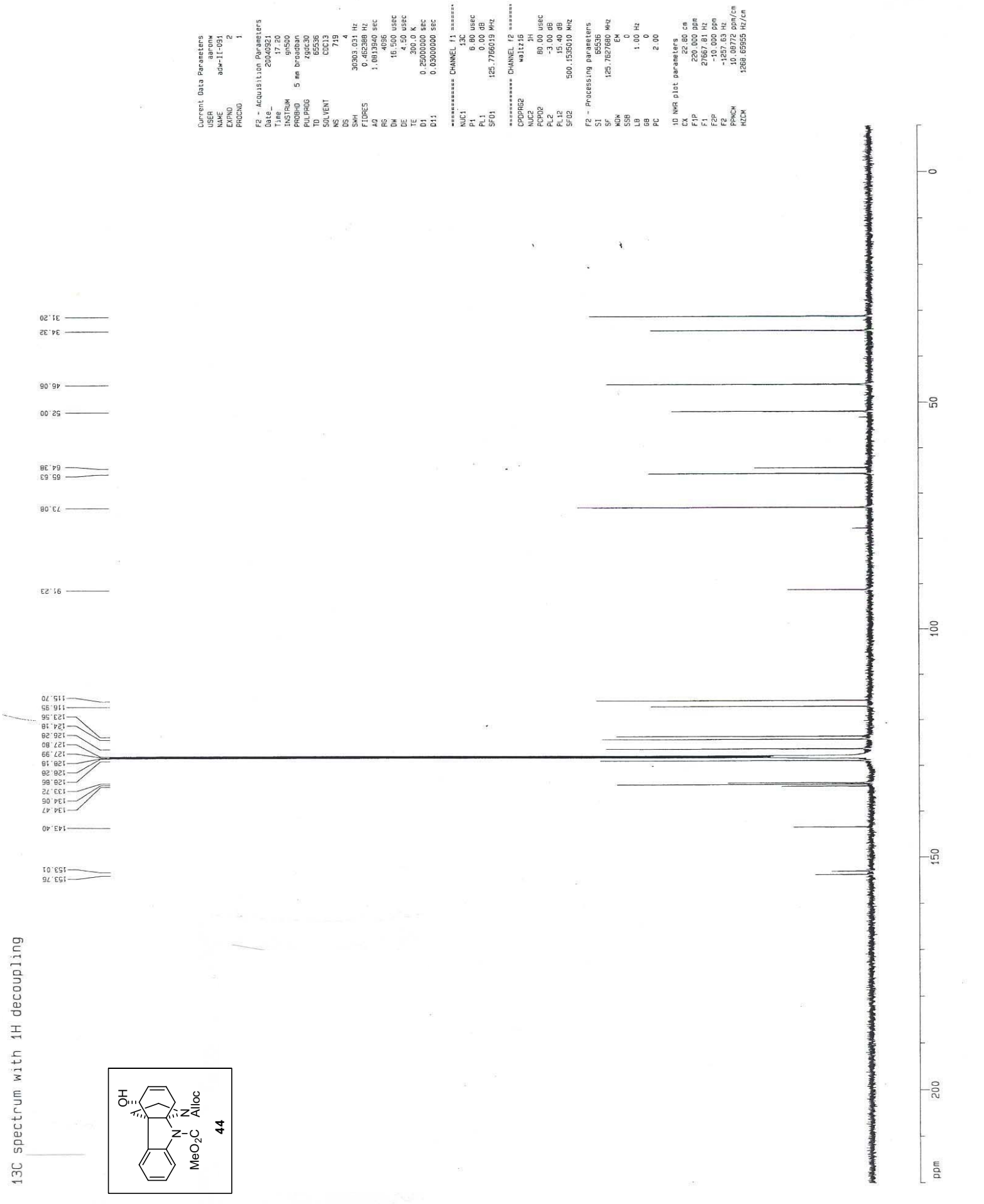
O
N
H
N
MeO2C
42

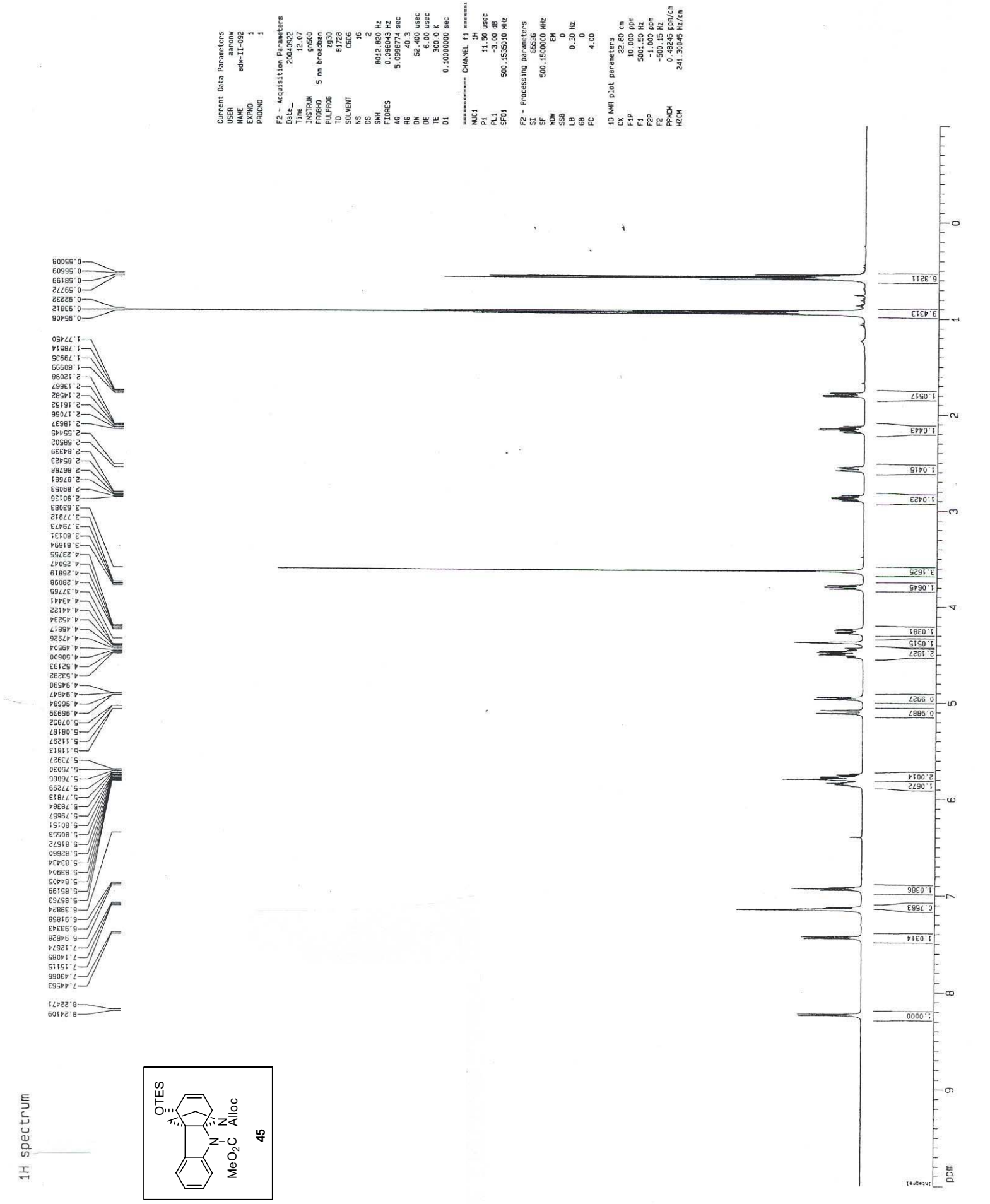

Current Data Parameters
USER dounay
NAME abd-1V-46-x
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Data_ 20040212
Time 9.13
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 356
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 333.4 K
D1 0.25000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SF01 125.7942048 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SF02 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803207 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.66980 Hz/cm
21.44
27.76
38.01
43.34
52.19
53.19
54.37
57.59
80.79
115.76
123.58
124.20
128.20
128.30
128.39
128.58
128.68
129.17
133.72
144.25
154.20
ppm
13C spectrum with 1H decoupling
MeO2C
42

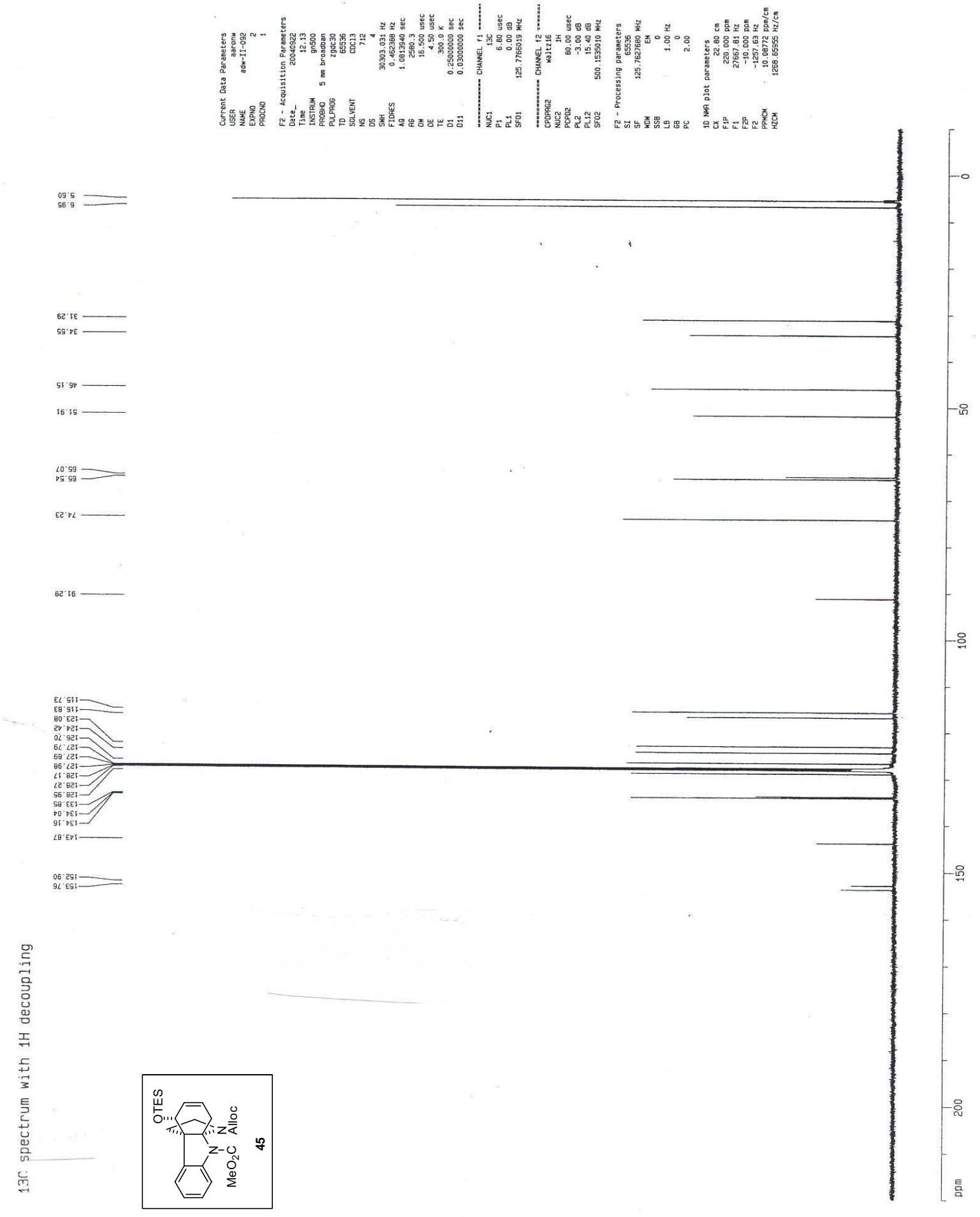

Current Data Parameters
USER aaronw
NAME adw-II-074
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040913
Time 14.09
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 20.2
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
0.8428
2.0541
1.0777
1.0534
2.0180
1.1140
0.7561
1.3673
3.1019
1.0303
1.0437
0.9967
0.9992
0.9718
2.1024
1.0394
0.9945
Integral
ppm
0
1
2
3
4
5
6
7
8
9
1H spectrum
MeO2C
Alloc
43

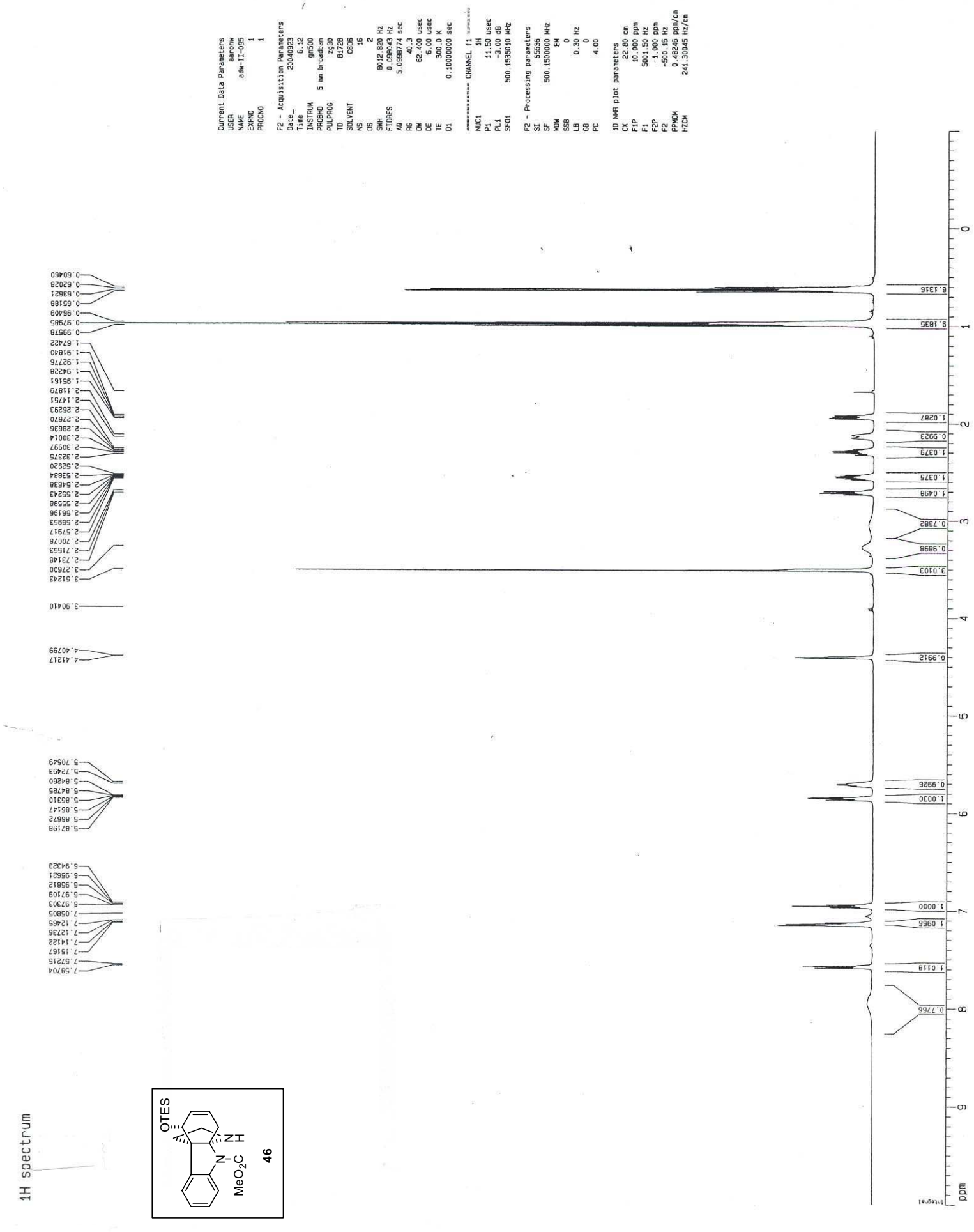

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-074
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040913
Time 14.16
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 565
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
---------- CHANNEL f1 -------
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
---------- CHANNEL f2 -------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.65995 Hz/cm
22.09
24.08
29.68
45.10
52.22
53.06
54.79
56.02
65.57
77.90
86.61
116.50
117.46
123.03
123.62
127.81
128.01
128.20
128.30
129.47
131.56
133.64
144.48
153.44
154.58
ppm
0
50
100
150
200
MeO2C
Alloc
43

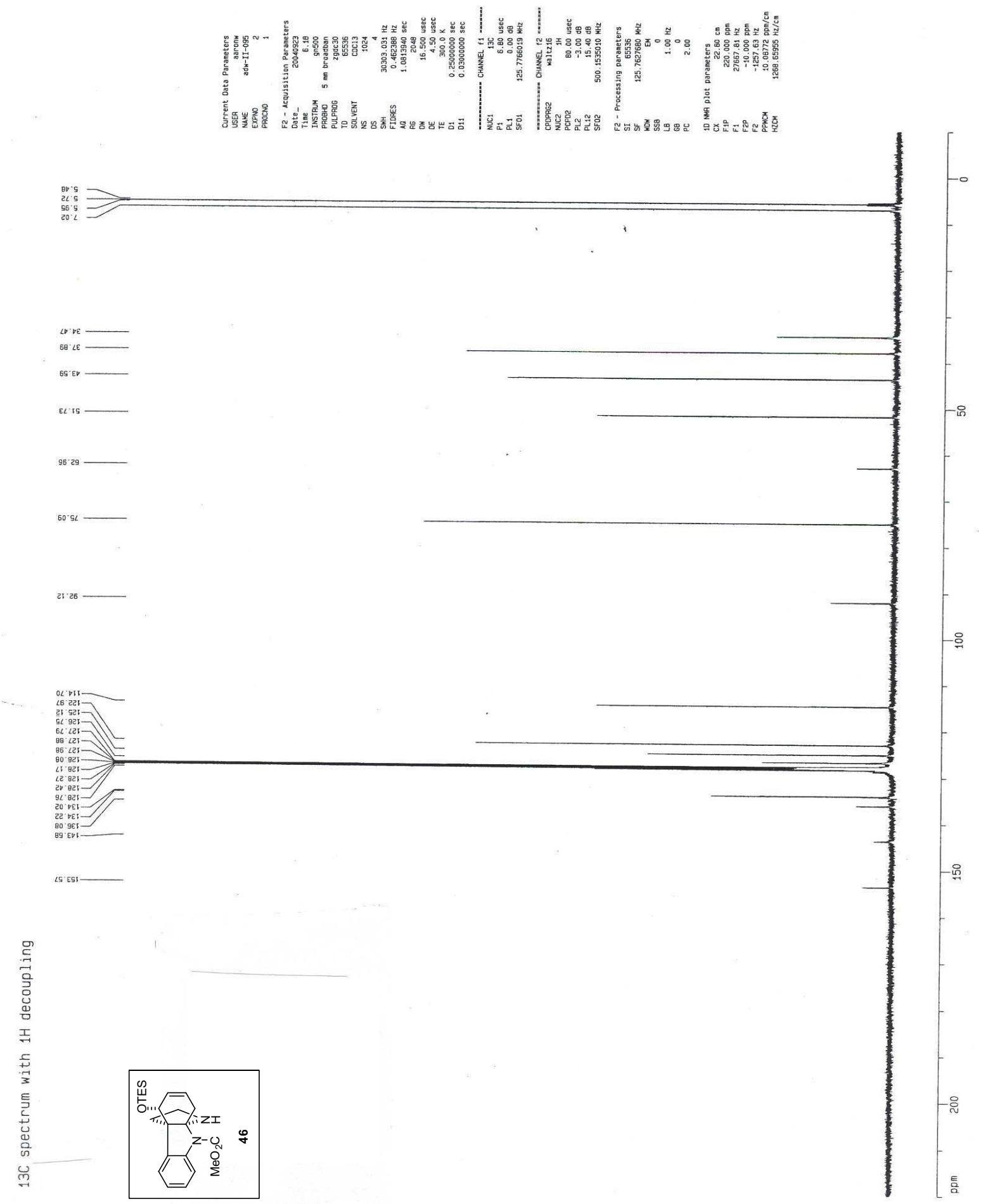

Current Data Parameters
USER aaronw
NAME adw-II-087
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040916
Time 13.16
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 40.3
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
1.0923
1.0982
1.4786
1.0692
3.1255
1.0908
1.0540
1.0545
2.2031
1.0240
1.0161
1.7149
1.3690
1.0416
1.0904
1.0446
1.0000
ppm
integral
1H spectrum
OH
N
N
Alloc
MeO2C
44

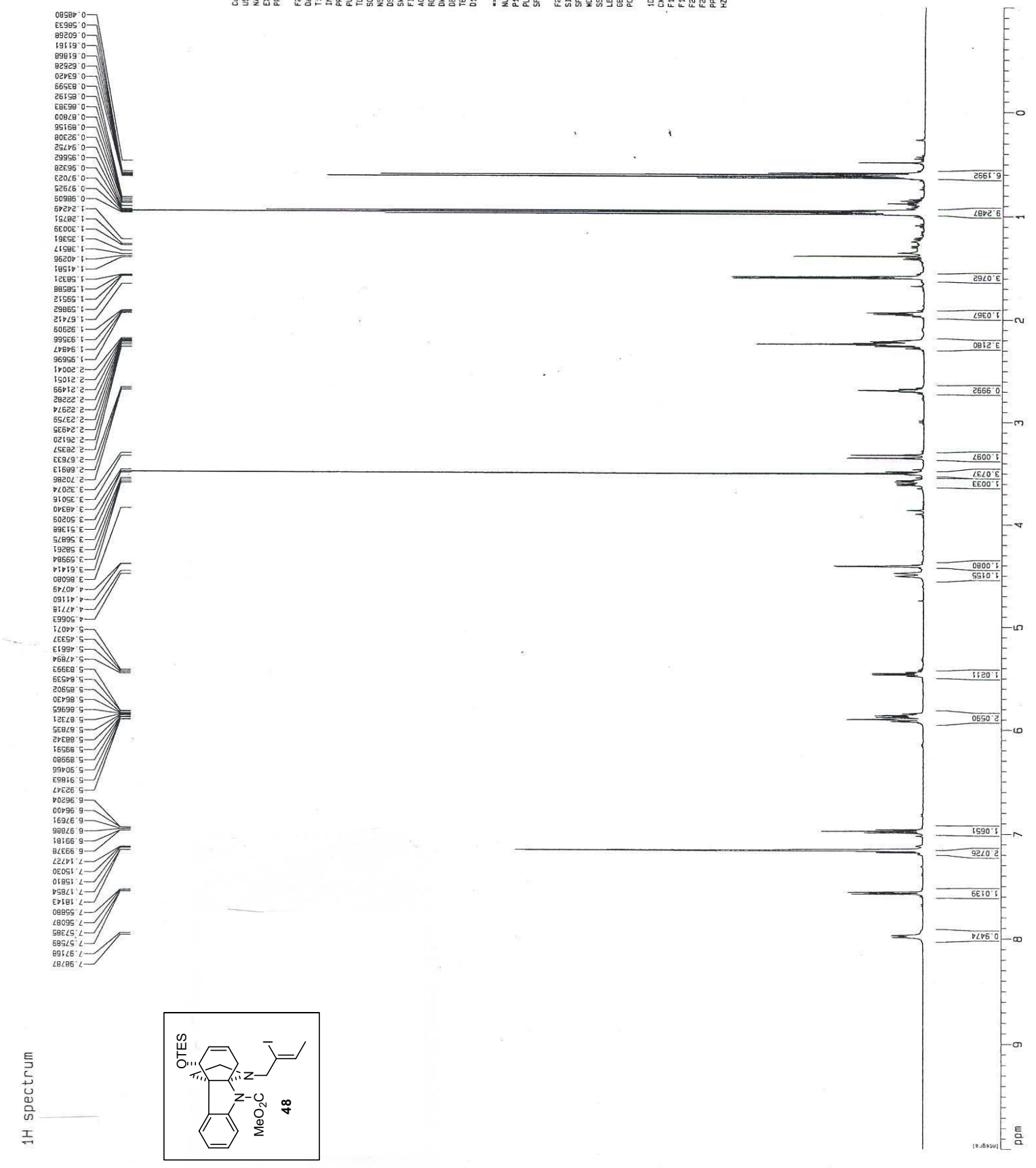

Current Data Parameters
USER aaronw
NAME adw-II-091
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040921
Time 17.20
INSTRUM spn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 719
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.65955 Hz/cm
31.20
34.32
46.06
52.00
64.38
65.63
73.08
91.23
115.70
116.95
123.56
124.18
126.28
127.80
127.99
128.18
128.28
128.86
133.72
134.06
134.47
143.40
153.01
153.76
0
50
100
150
200
ppm
13C spectrum with 1H decoupling
OH
N
N
Alloc
MeO2C
44

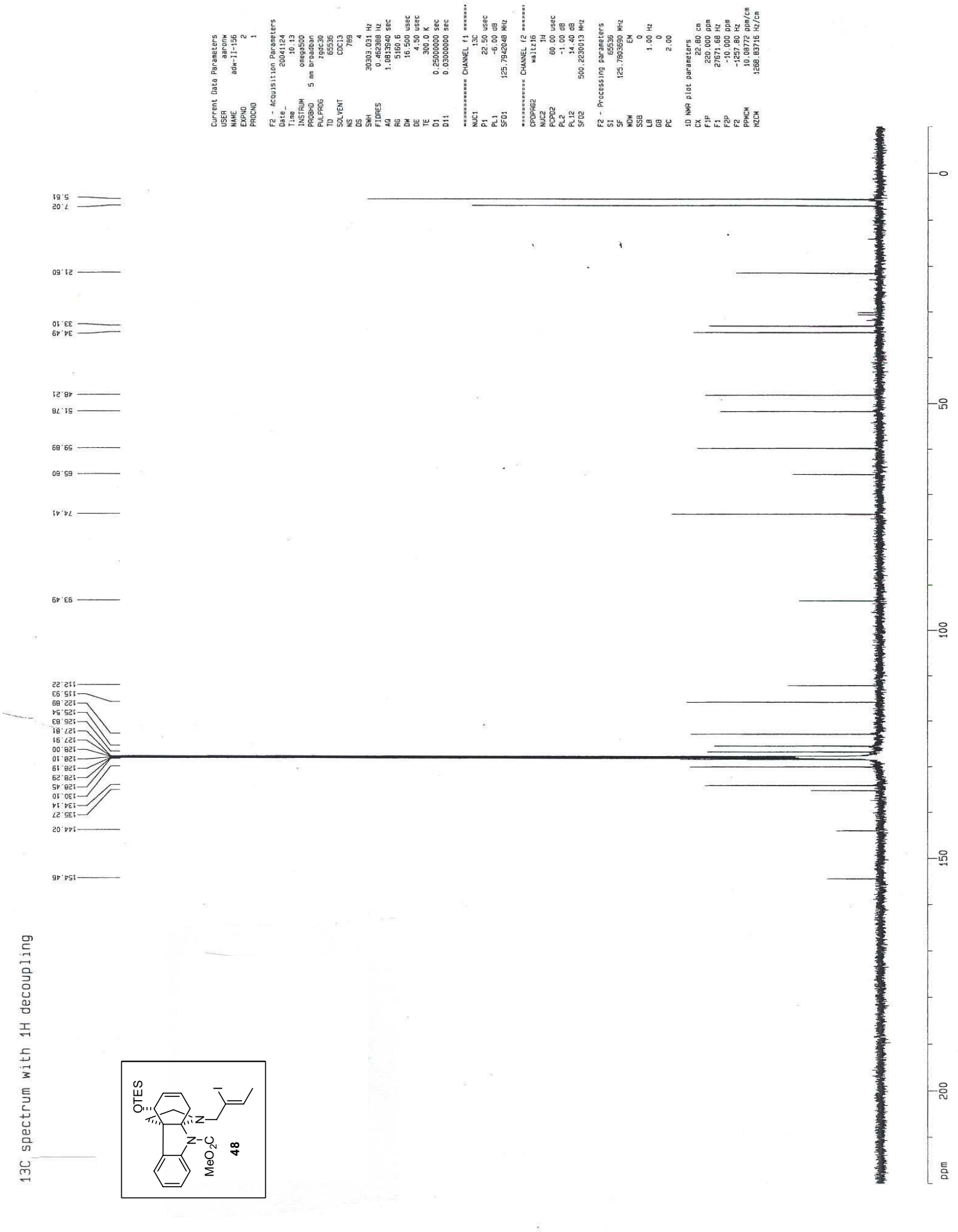

Current Data Parameters
USER aaronw
NAME adw-II-092
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040922
Time 12.07
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C606
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 40.3
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
6.3211
9.4313
1.0517
1.0443
1.0415
1.0423
3.1625
1.0645
1.0381
1.0515
2.1827
0.9927
0.9887
2.0014
1.0672
1.0386
0.7563
1.0314
1.0000
ppm
1H spectrum
OTES
MeO2C
Alloc
45

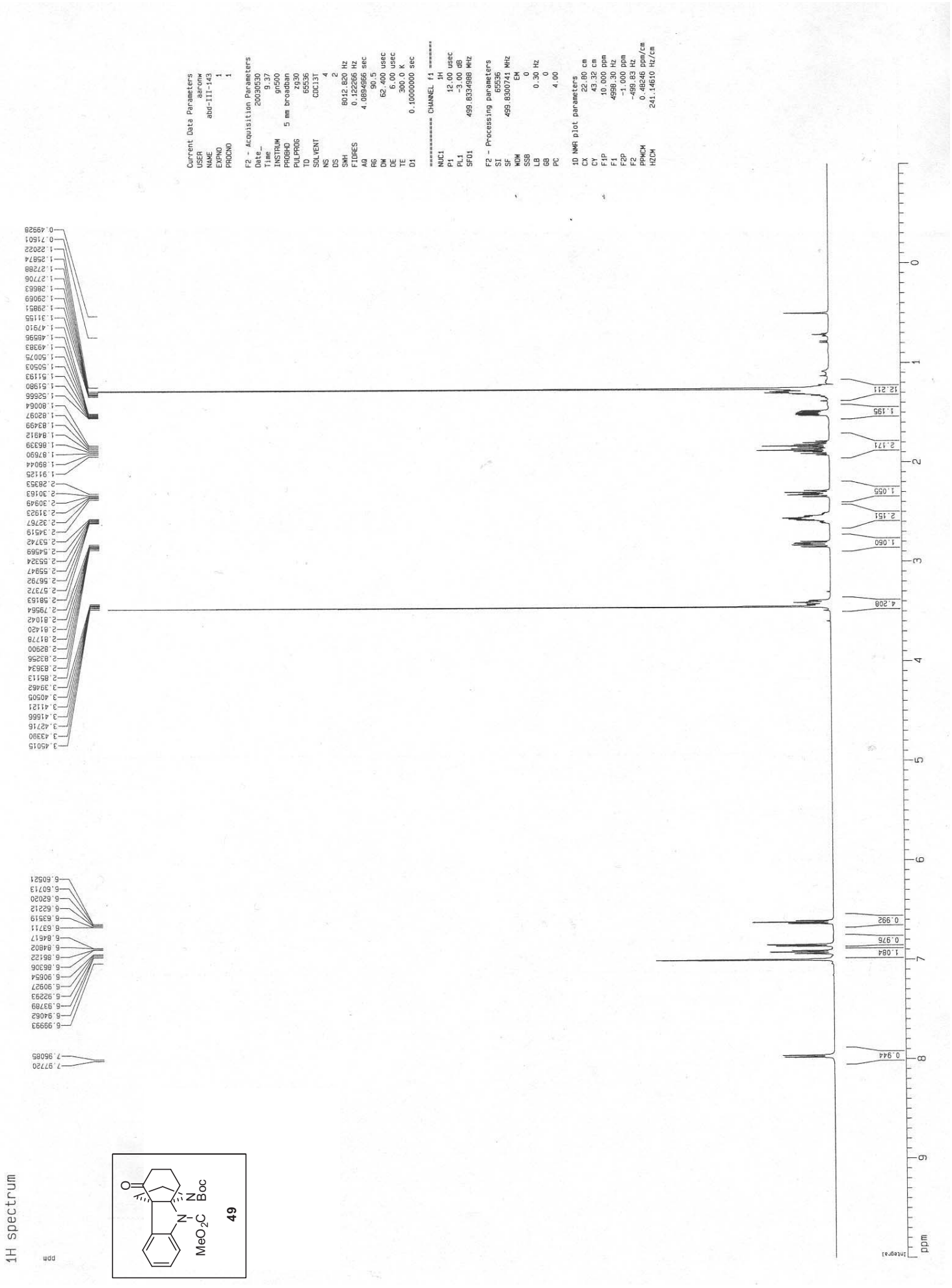

Current Data Parameters
USER aaronw
NAME adw-11-092
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040922
Time 12.13
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 712
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2580.3
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.60 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.65955 Hz/cm
5.60
6.95
31.29
34.55
46.15
51.91
65.07
65.54
74.23
91.29
115.73
116.83
123.08
124.42
126.70
127.79
127.89
127.98
128.17
128.27
128.95
133.85
134.04
134.16
143.87
152.90
153.76
0
50
100
150
200
ppm
13C spectrum with 1H decoupling
OTES
N
N
Alloc
MeO2C
45

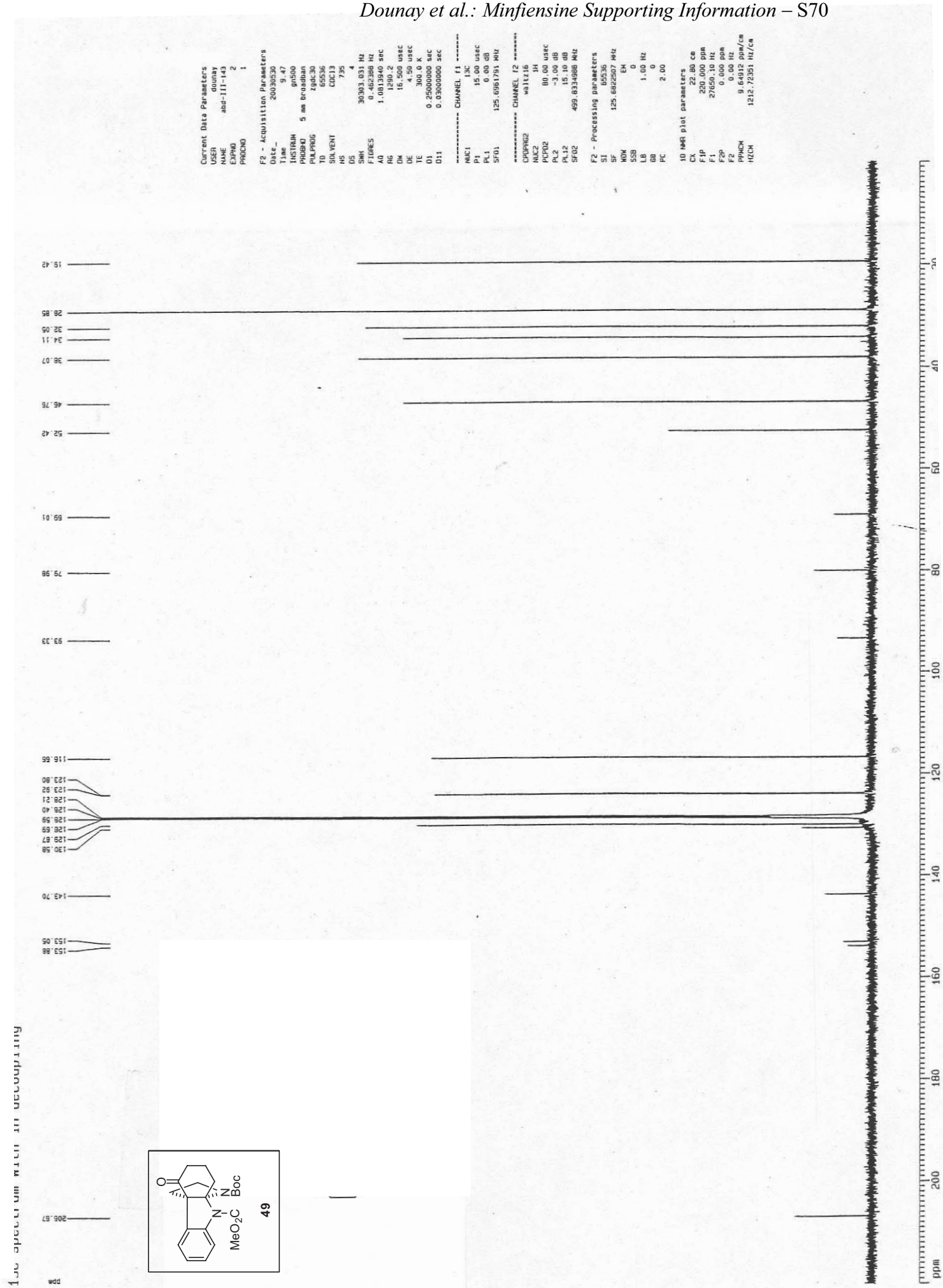

Current Data Parameters
USER aaronw
NAME adw-II-095
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040923
Time 6.12
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 40.3
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
0.60460
0.62028
0.63621
0.65188
0.96409
0.97985
0.99578
1.67422
1.91840
1.92776
1.94228
1.95161
2.11879
2.14751
2.26293
2.27670
2.28636
2.30014
2.30997
2.32375
2.52920
2.53884
2.54638
2.55243
2.55598
2.56196
2.56953
2.57917
2.70078
2.71553
2.73148
3.27600
3.51243
3.90410
4.40799
4.42127
5.70549
5.72493
5.84260
5.84785
5.85310
5.86147
5.86672
5.87198
6.94323
6.95621
6.95812
6.97109
6.97303
7.05805
7.12465
7.12736
7.14122
7.15167
7.57215
7.59704
6.1316
9.1835
1.0287
0.9923
1.0379
1.0375
1.0498
0.7382
0.9698
3.0103
0.9912
0.9926
1.0330
1.0000
1.0966
1.0118
0.7766
ppm
1H spectrum
OTES
N
H
N
MeO2C
46

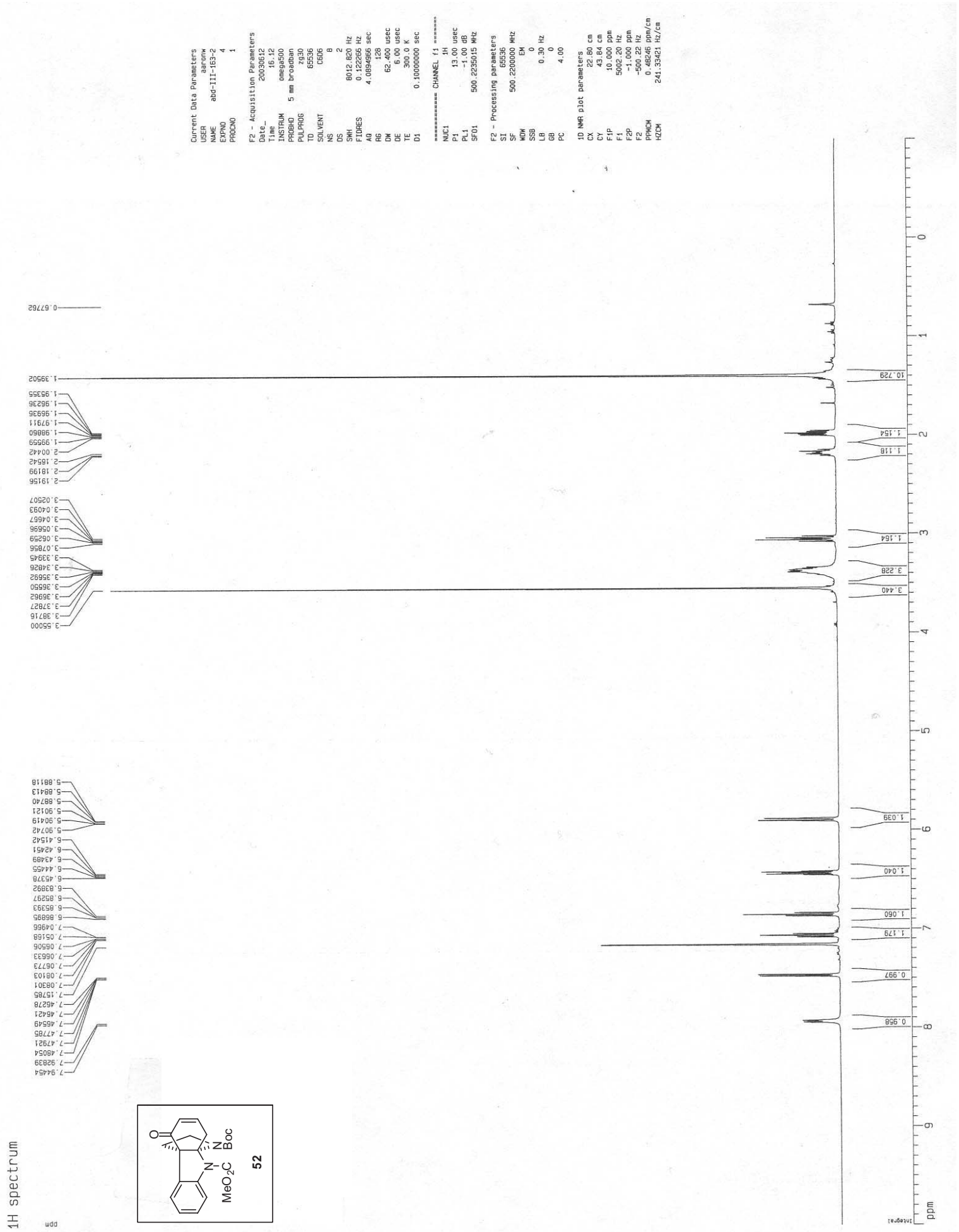

13C spectrum with 1H decoupling
OTES
N
H
N
MeO2C
46
Current Data Parameters
USER aaronw
NAME adw-II-095
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040923
Time 6.18
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2048
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.61 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.65955 Hz/cm
5.48
5.72
5.95
7.02
34.47
37.89
43.59
51.73
62.96
75.09
92.12
114.70
122.97
125.12
126.75
127.79
127.88
127.98
128.08
128.17
128.27
128.42
128.76
134.02
134.22
136.08
143.68
153.57
0
50
100
150
200
ppm

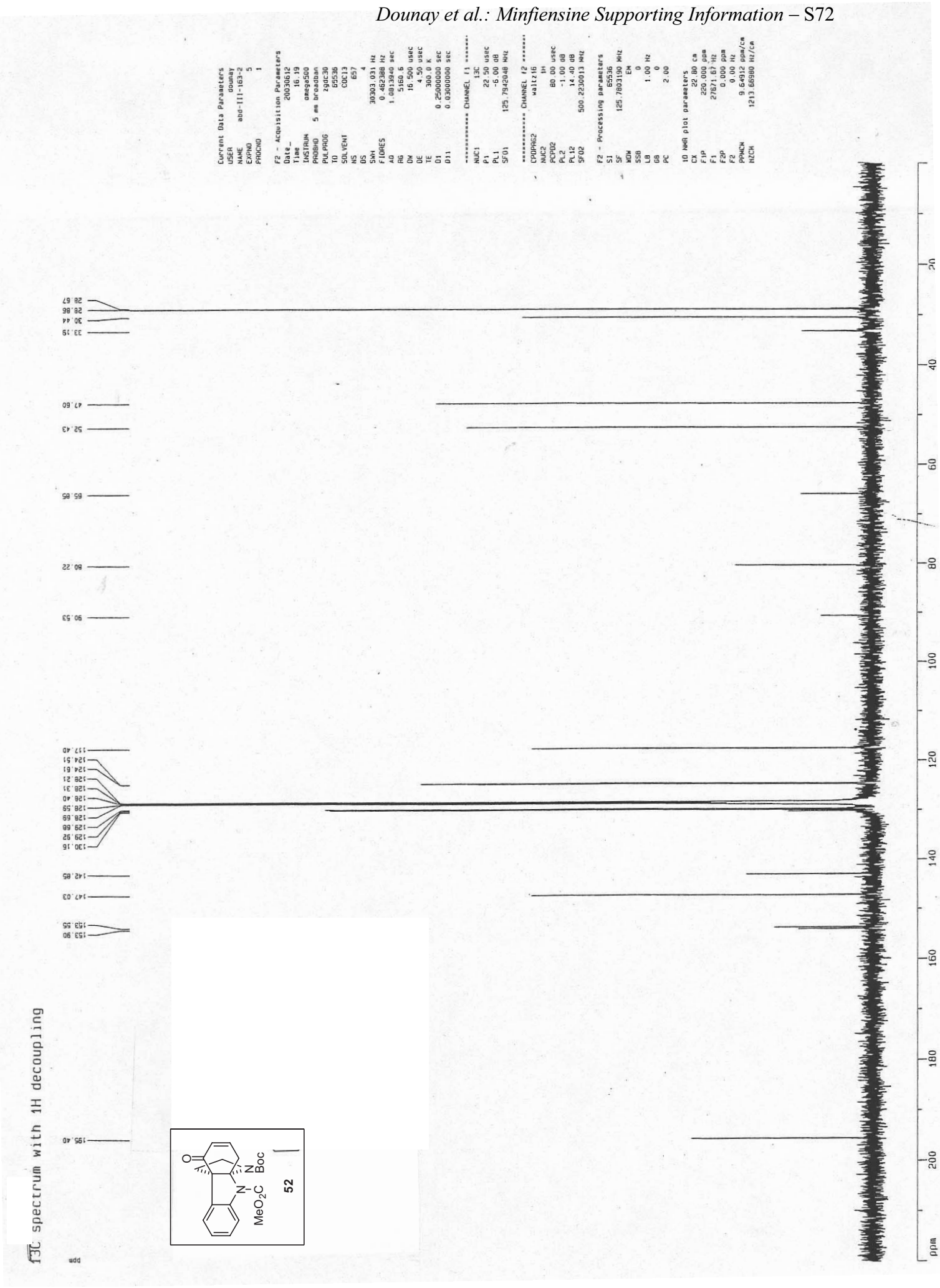

Current Data Parameters
USER aaronw
NAME adw-II-156
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041124
Time 10.09
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
6.1992
9.2487
3.0762
1.0367
3.2180
0.9992
1.0097
3.0737
1.0033
1.0080
1.0155
1.0211
2.0590
1.0651
2.0726
1.0139
0.9474
ppm
1H spectrum
OTES
N
N
MeO2C
48

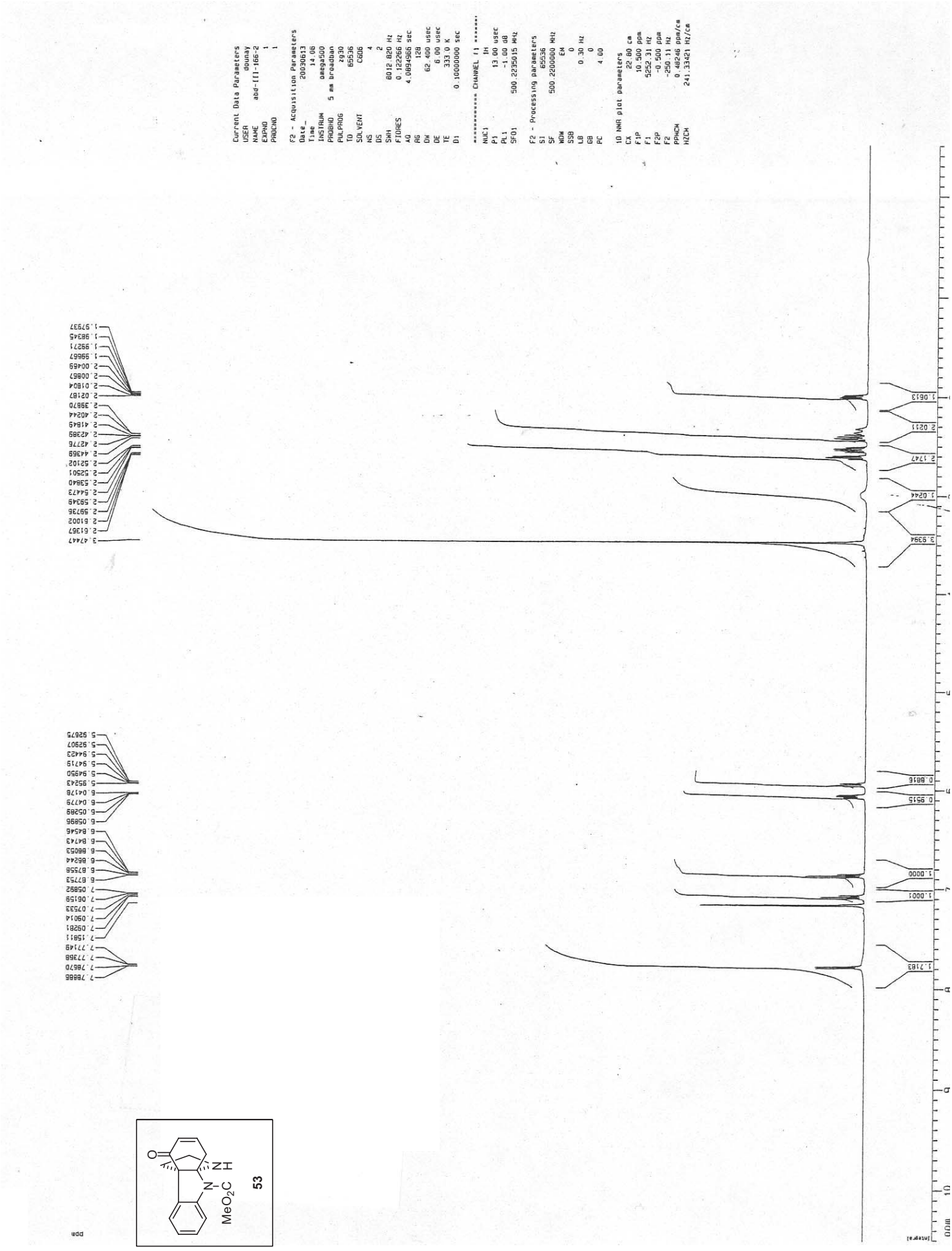

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-156
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041124
Time_ 10.13
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 789
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813840 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
FIP 220.000 ppm
F1 27671.68 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83716 Hz/cm
5.61
7.02
21.60
33.10
34.49
48.21
51.78
59.89
65.80
74.41
93.49
112.22
115.93
122.89
125.54
126.83
127.81
127.91
128.00
128.10
128.19
128.29
128.45
130.10
134.14
135.27
144.02
154.46
0
50
100
150
200
ppm
OTES
N
N
MeO2C
48

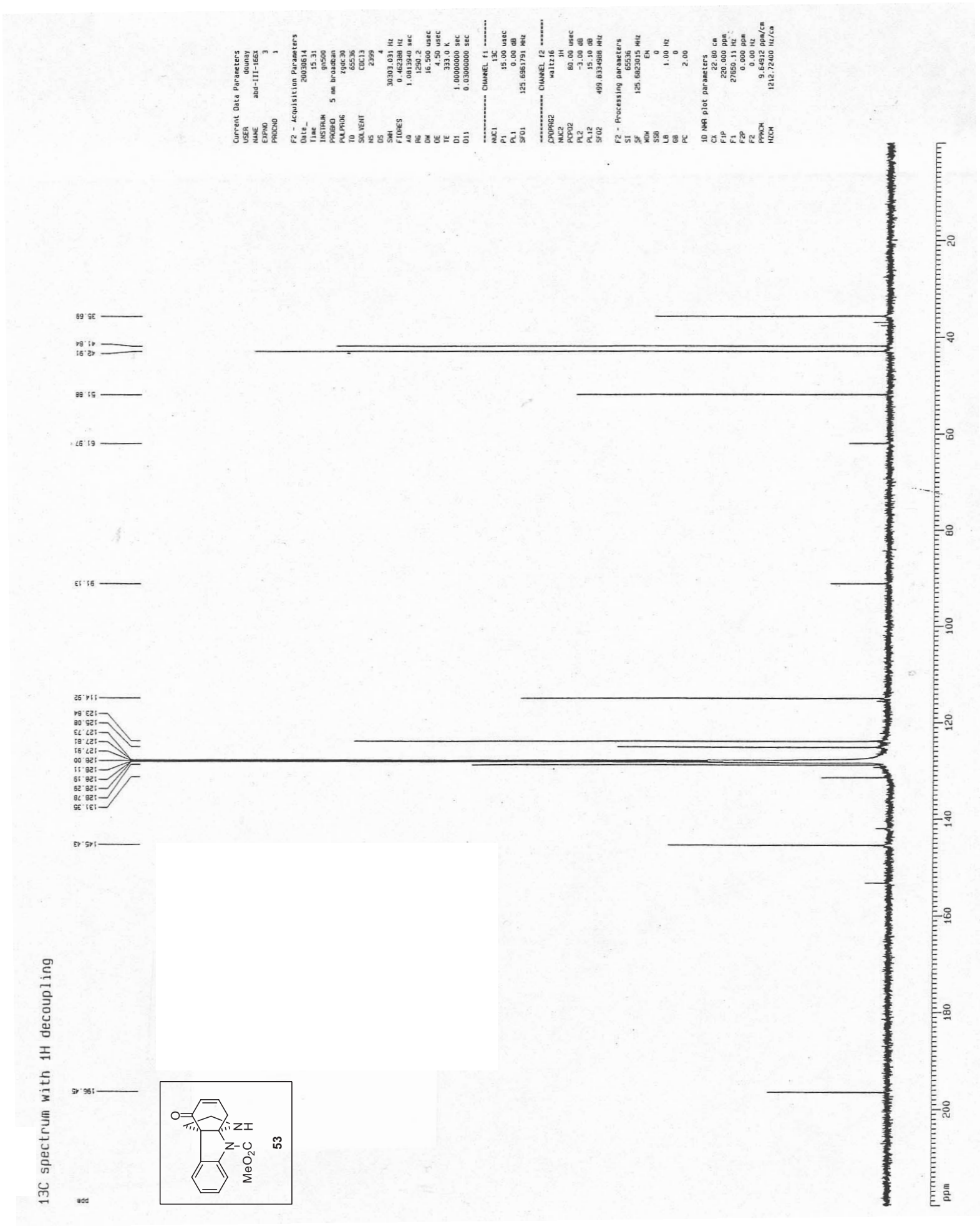

1H spectrum
ppm
MeO2C
Boc
N
N
O
49
Current Data Parameters
USER aaronw
NAME abd-III-143
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030530
Time 9.37
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8334988 MHz
F2 - Processing parameters
SI 65536
SF 499.8300741 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 43.32 cm
F1P 10.000 ppm
F1 4998.30 Hz
F2P -1.000 ppm
F2 -499.83 Hz
PPMCM 0.49246 ppm/cm
HZCM 241.14610 Hz/cm
integral
12.211
1.195
2.171
1.055
2.151
1.060
4.208
0.992
0.976
1.084
0.944

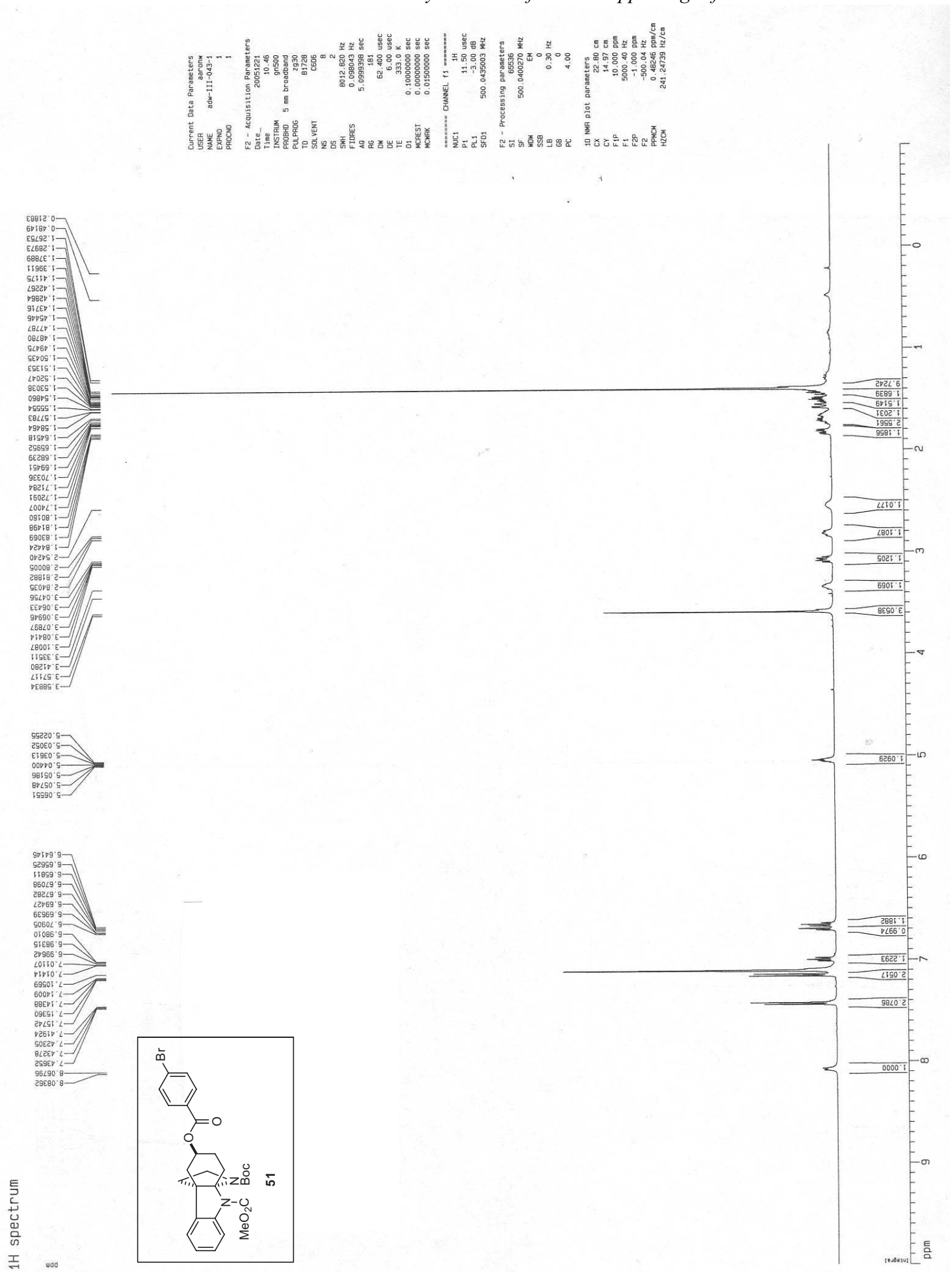

Current Data Parameters
USER dounay
NAME abd-III-143
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030530
Time 9.47
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 735
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 1290.2
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
---------- CHANNEL f1 ----------
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961791 MHz
---------- CHANNEL f2 ----------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz
F2 - Processing parameters
SI 65536
SF 125.6822507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27650.10 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1212.72351 Hz/cm
19.42
28.86
32.05
34.11
36.07
46.76
52.42
59.01
79.98
93.33
116.66
123.80
123.92
126.21
126.40
126.59
126.69
129.87
130.58
143.70
153.88
153.05
206.67
ppm
MeO2C
Boc
N
N
O
49

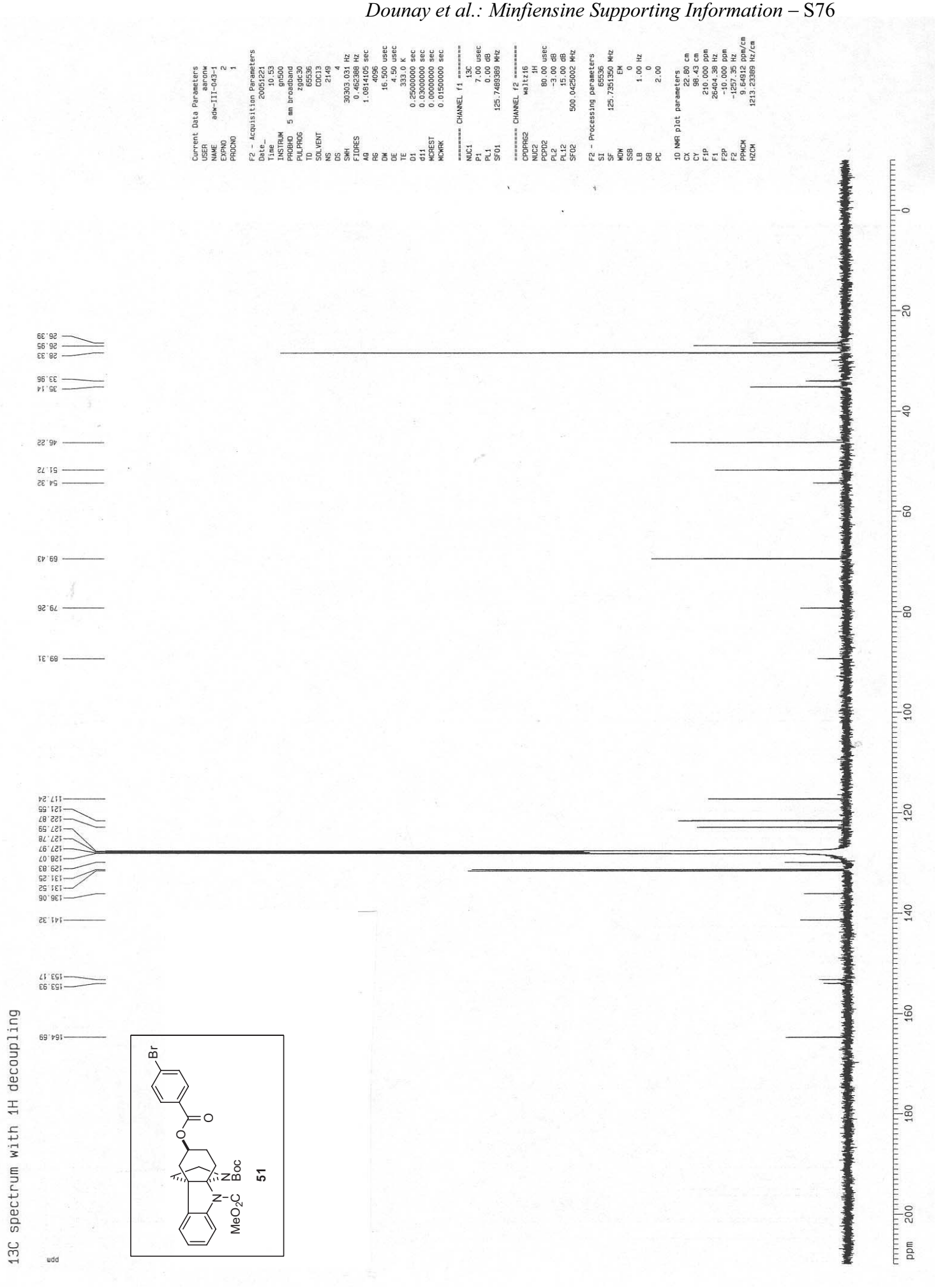

1H spectrum
Current Data Parameters
USER aaronw
NAME abd-III-163-2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030612
Time 16.12
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SF01 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 43.84 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
MeO2C
N
N
Boc
O
52
ppm
Integral

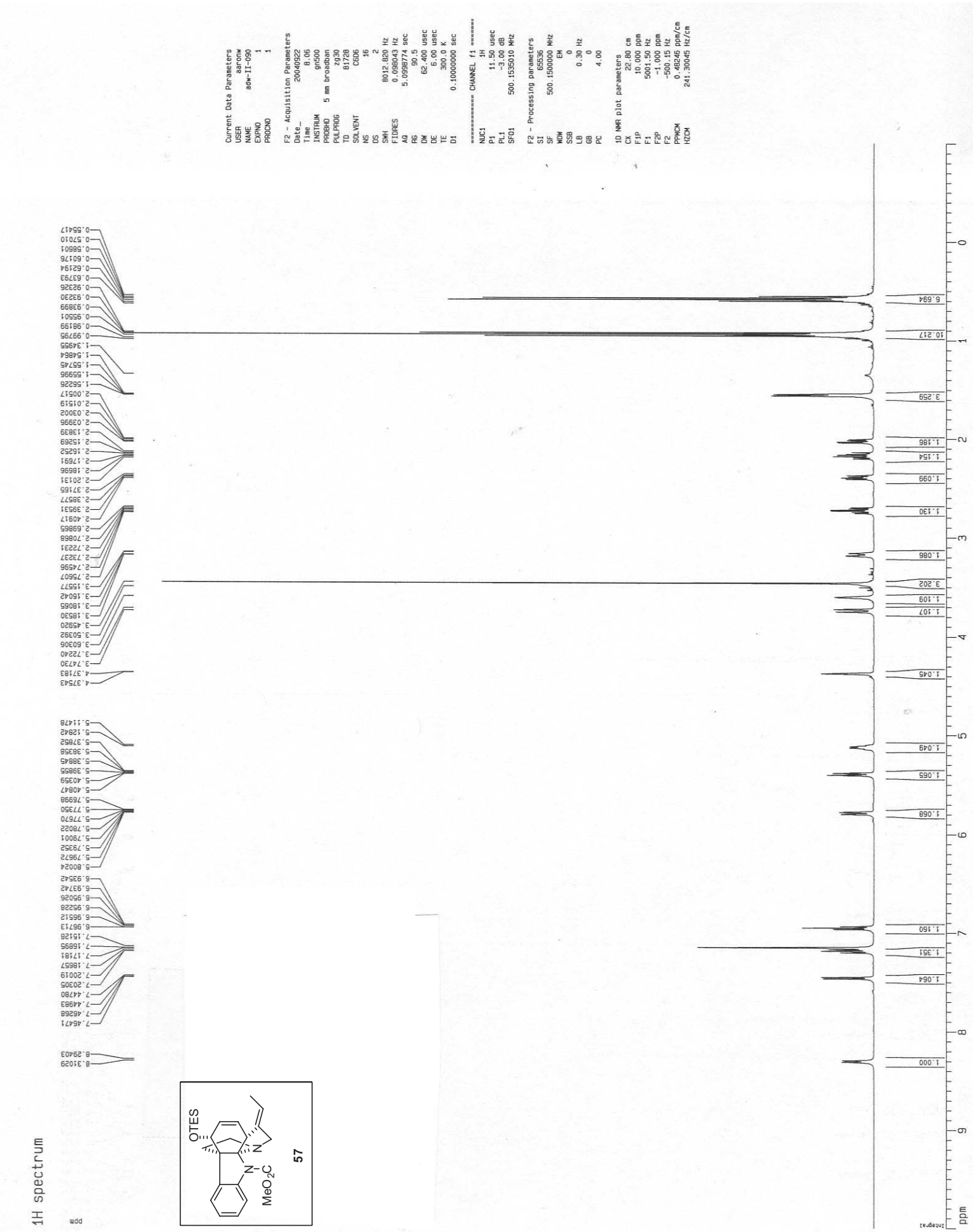

13C spectrum with 1H decoupling
Current Data Parameters
USER dounay
NAME abd-III-163-2
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030612
Time 16.19
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 657
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
---------- CHANNEL f1 ----------
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
---------- CHANNEL f2 ----------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.66980 Hz/cm
28.67
28.86
30.44
33.19
47.60
52.43
65.85
80.22
90.53
117.40
124.51
124.61
128.21
128.31
128.40
128.55
128.69
129.66
129.92
130.16
142.85
147.03
153.90
153.55
195.40
ppm
MeO2C
Boc
N
N
O
52

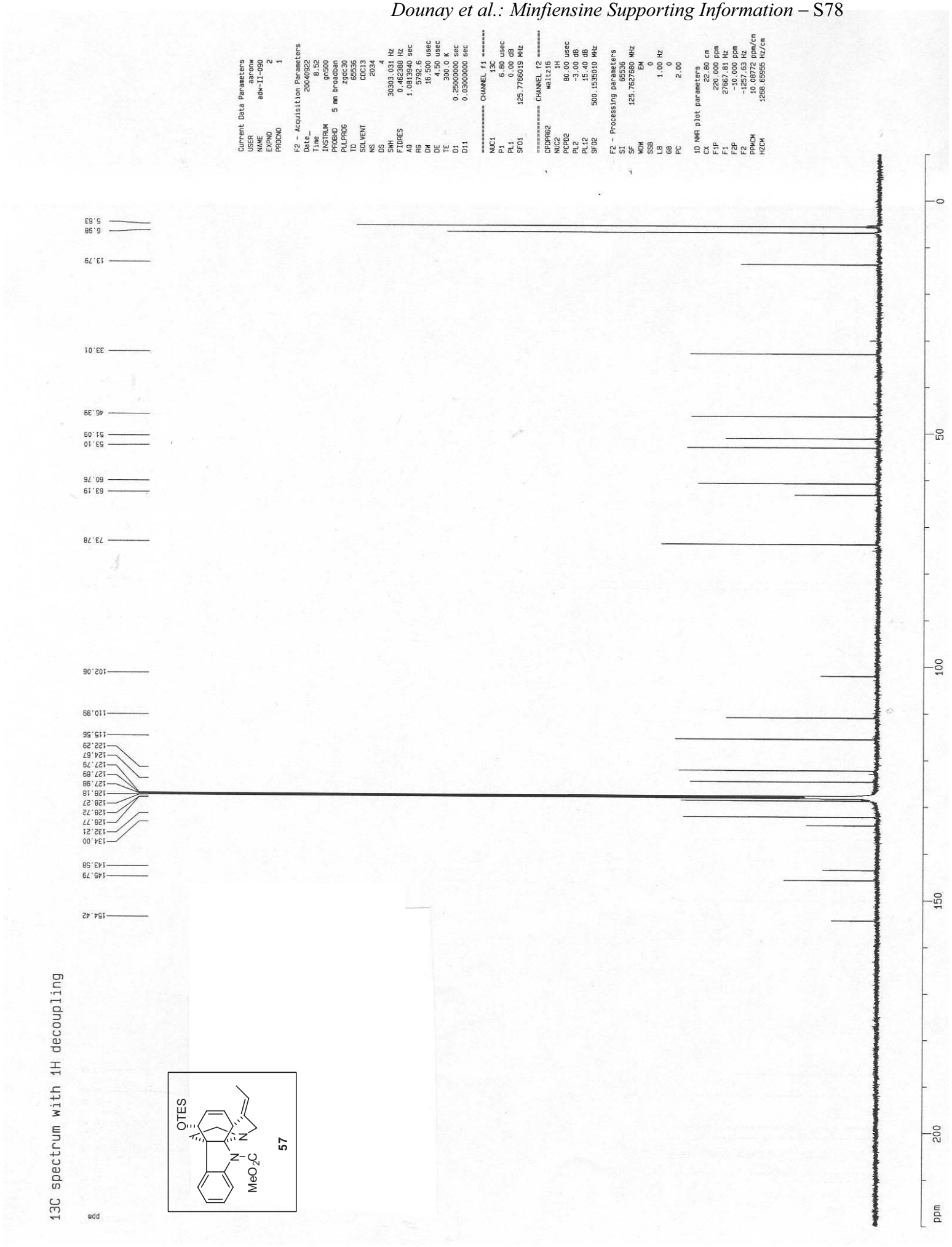

Current Data Parameters
USER dounay
NAME abd-[1]-166-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030613
Time 14.06
INSTRUM amega500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
1.0613
2.0211
2.1747
1.0244
3.9394
0.8816
0.9515
1.0000
1.0001
1.7183
MeO2C
53
ppm
Integral

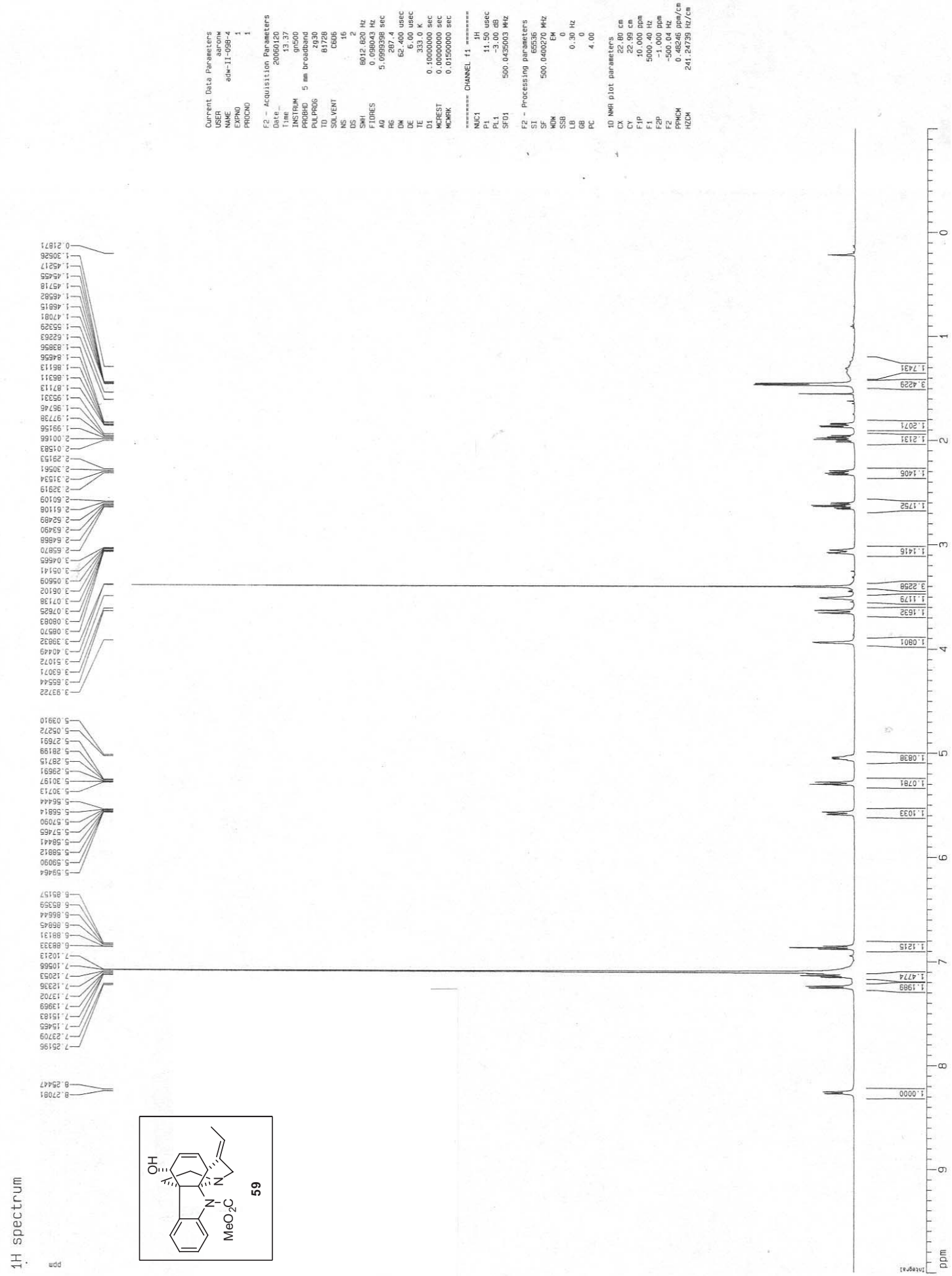

13C spectrum with 1H decoupling
Current Data Parameters
USER dounay
NAME abd-III-166X
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030614
Time 15.31
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2399
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 1290.2
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 1.00000000 sec
D11 0.03000000 sec
---------- CHANNEL f1 ------
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961791 MHz
---------- CHANNEL f2 ------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz
F2 - Processing parameters
SI 65536
SF 125.6823015 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27650.11 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1212.72400 Hz/cm
35.69
41.84
42.91
51.88
61.97
91.13
114.92
123.84
125.08
127.73
127.81
127.91
128.00
128.11
128.19
128.29
128.78
131.35
145.43
196.45
ppm
20
40
60
80
100
120
140
160
180
200
MeO2C
53

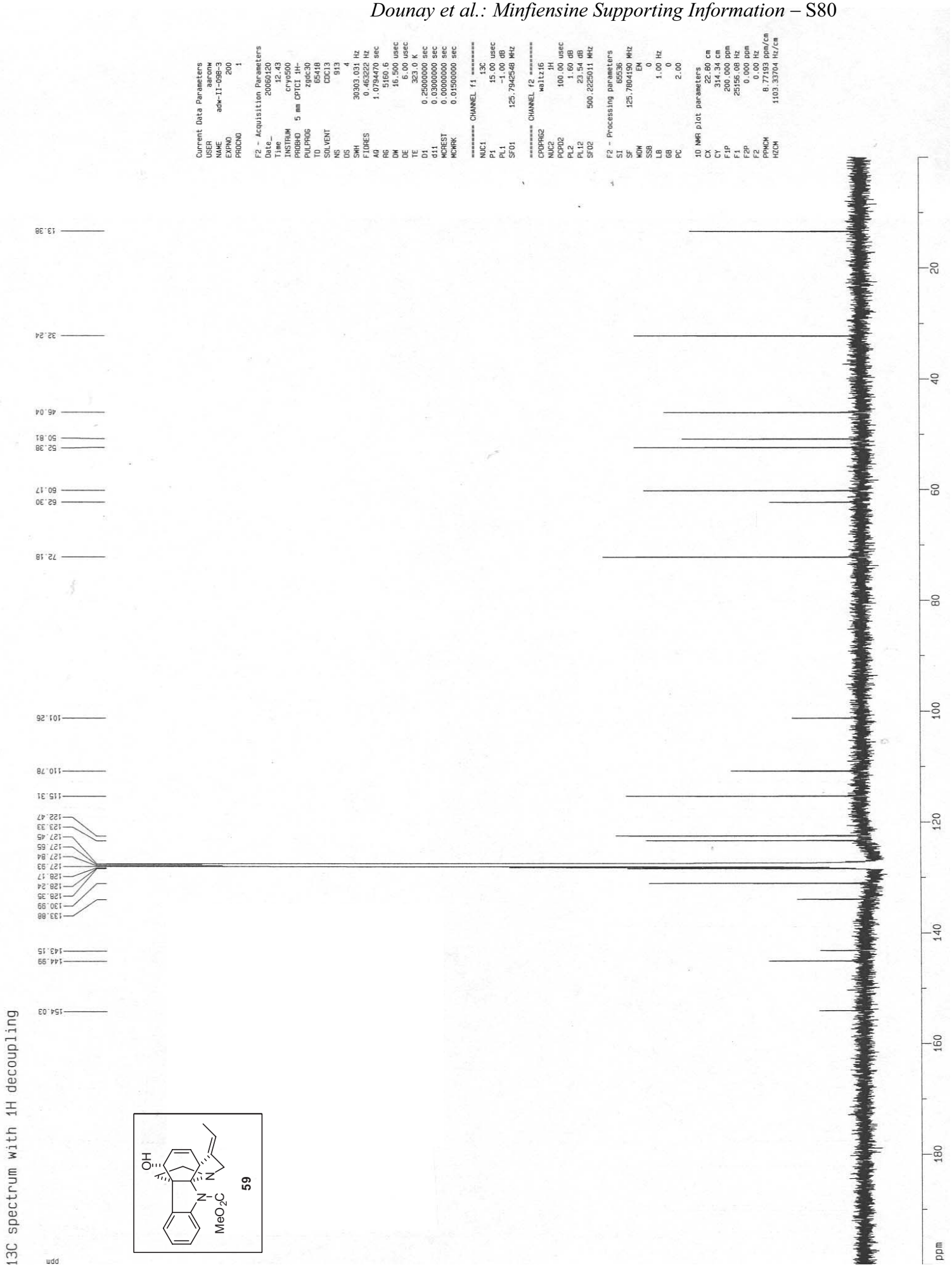

1H spectrum
ppm
Br
O
O
N
Boc
N
MeO2C
51


13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-III-043-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051221
Time 10.53
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2149
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 125.7489389 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
SFO2 500.0425002 MHz
F2 - Processing parameters
SI 65536
SF 125.7351350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 98.43 cm
F1P 210.000 ppm
F1 26404.38 Hz
F2P -10.000 ppm
F2 -1257.35 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.23389 Hz/cm
26.39
26.95
28.33
33.96
35.14
46.22
51.72
54.32
69.43
79.26
89.31
117.24
121.55
122.87
127.59
127.78
127.97
128.07
129.83
131.25
131.52
136.06
141.32
153.17
153.93
164.69
ppm
Br
O
O
N
N
Boc
MeO2C
51

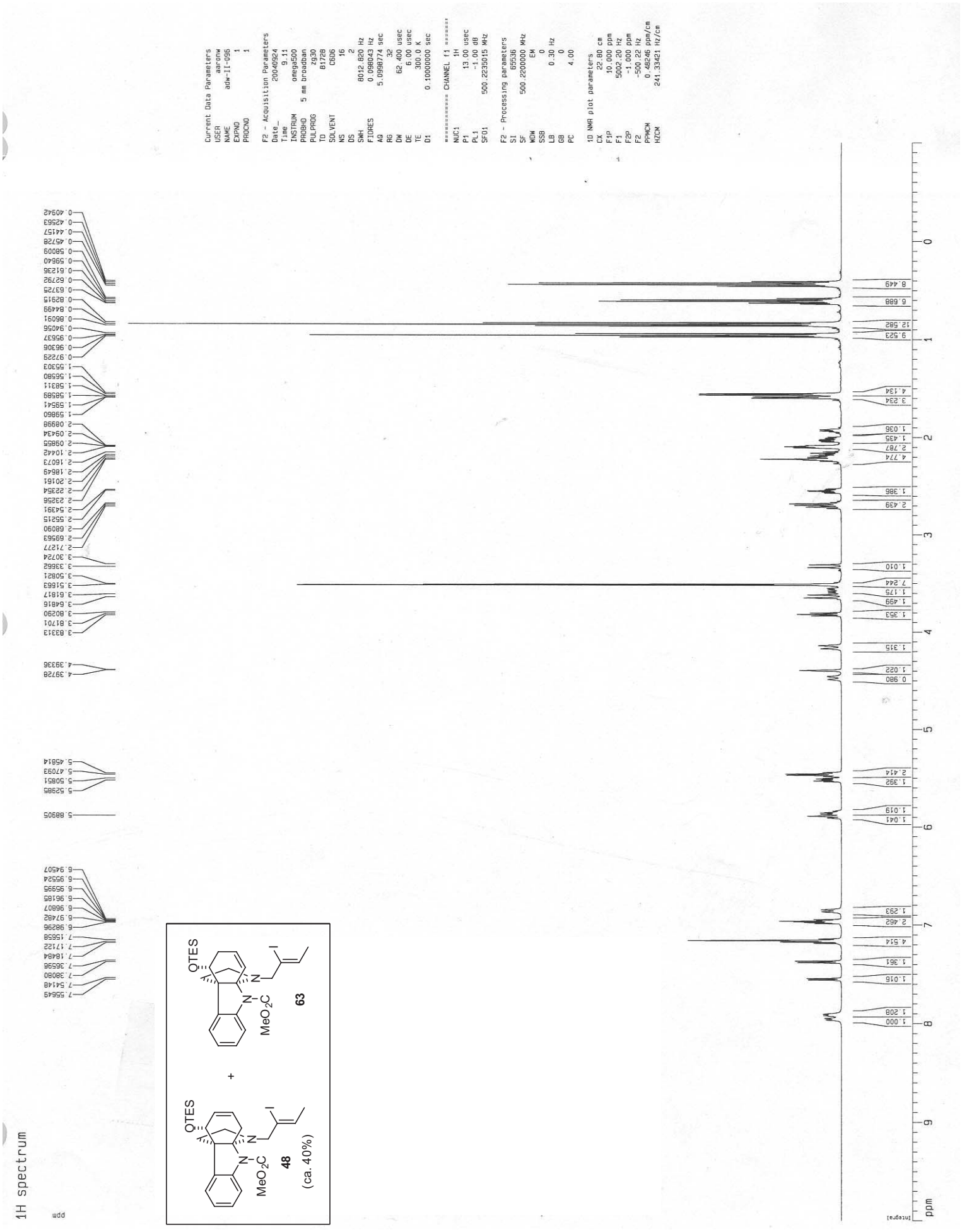

1H spectrum
OTES
N
N
MeO2C
57
ppm
Integral

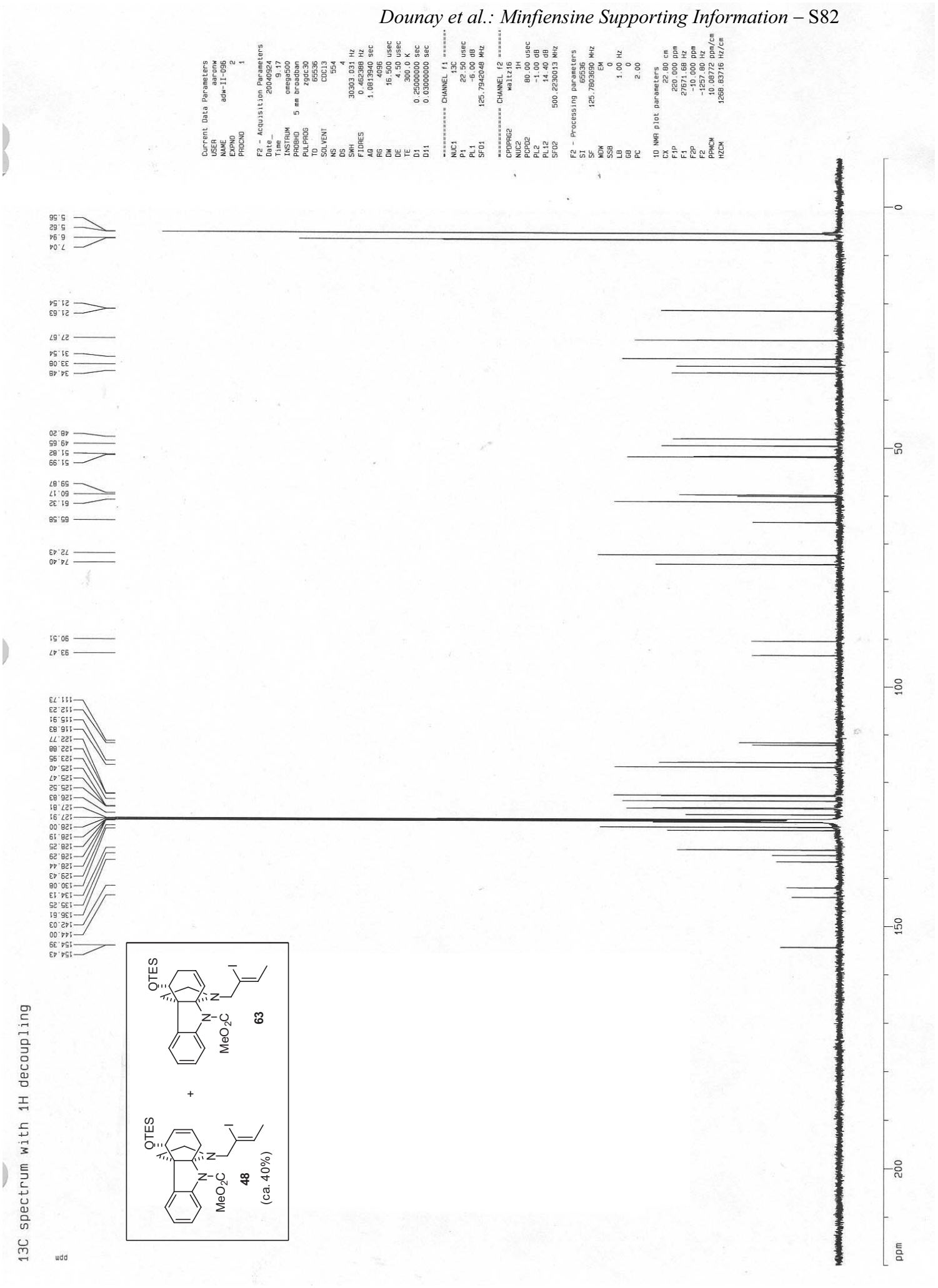

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-090
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040922
Time 8.52
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2034
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5792.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.65955 Hz/cm
5.63
5.98
13.79
33.01
46.39
51.09
53.10
60.76
63.19
73.78
102.06
110.99
115.56
122.29
124.67
127.79
127.89
127.98
128.18
128.27
128.72
128.77
132.21
134.00
143.58
145.79
154.42
OTES
N
N
MeO2C
57
ppm
0
50
100
150
200

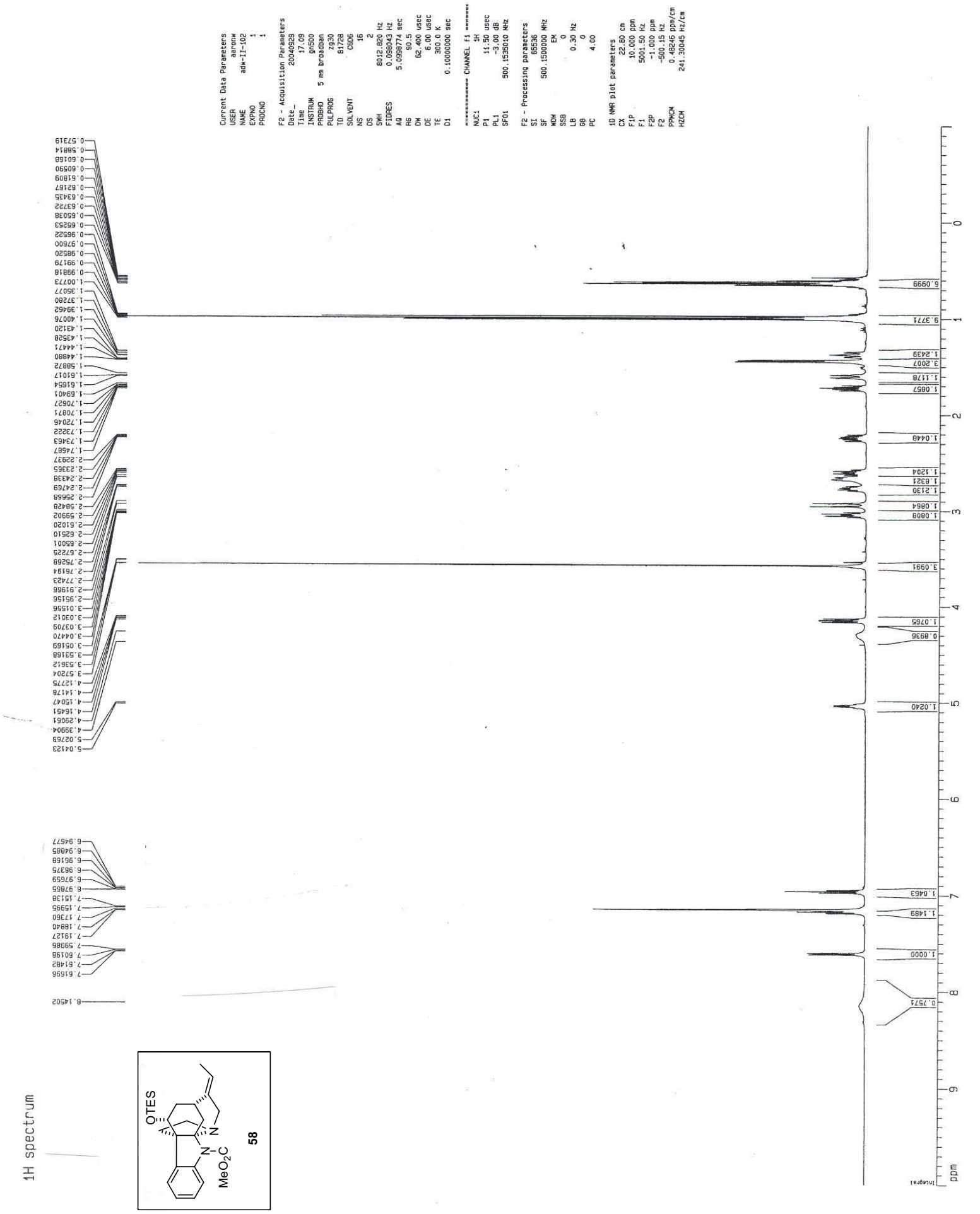

1H spectrum
wdd
ppm
integral
OH
N
N
MeO2C
59
Current Data Parameters
USER aaronw
NAME adw-II-098-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060120
Time_ 13.37
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 287.4
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.0435003 MHz
F2 - Processing parameters
SI 65536
SF 500.0400270 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 22.99 cm
F1P 10.000 ppm
F1 5000.40 Hz
F2P -1.000 ppm
F2 -500.04 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.24739 Hz/cm

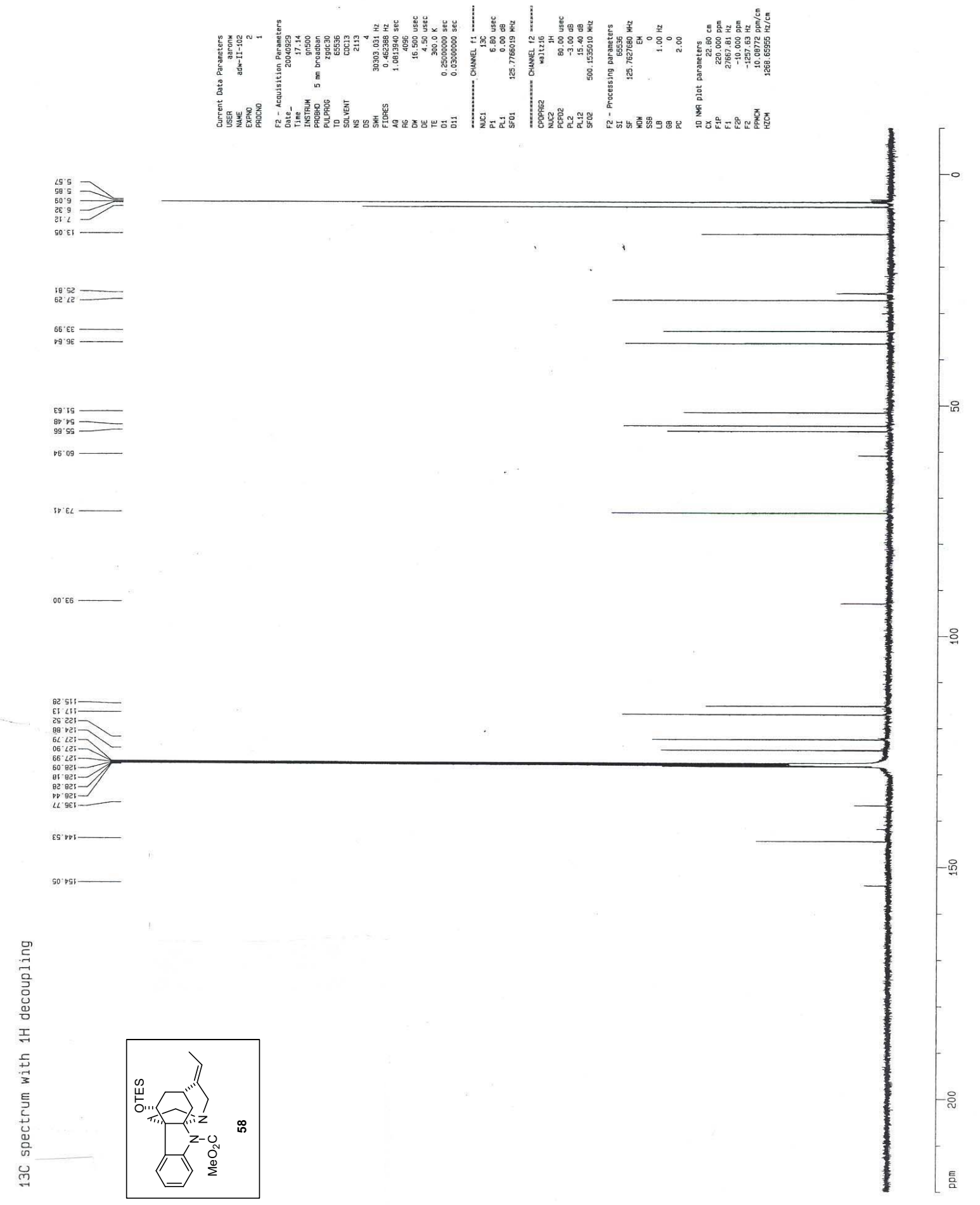



```
Current Data Parameters
USER      aaronw
NAME      adw-II-098-3
EXPNO         200
PROCNO          1

F2 - Acquisition Parameters
Date_      20060120
Time          12.43
INSTRUM       cryo500
PROBHD  5 mm CPTCI 1H-
PULPROG       zgdc30
TD            65418
SOLVENT       CDCl3
NS              512
DS                4
SWH           30303.031 Hz
FIDRES        0.463222 Hz
AQ            1.0794470 sec
RG            5160.6
DW              16.500 usec
DE               6.00 usec
TE             323.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1             13C
P1             15.00 usec
PL1            -1.00 dB
SFO1      125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2             1H
PCPD2          100.00 usec
PL2              1.60 dB
PL12            23.54 dB
SFO2      500.2225011 MHz

F2 - Processing parameters
SI            65536
SF        125.7804190 MHz
WDW              EM
SSB               0
LB             1.00 Hz
GB                0
PC             2.00

1D NMR plot parameters
CX            22.80 cm
CY            314.34 cm
F1P           200.000 ppm
F1            25156.08 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM          8.77193 ppm/cm
HZCM        1103.33704 Hz/cm
```

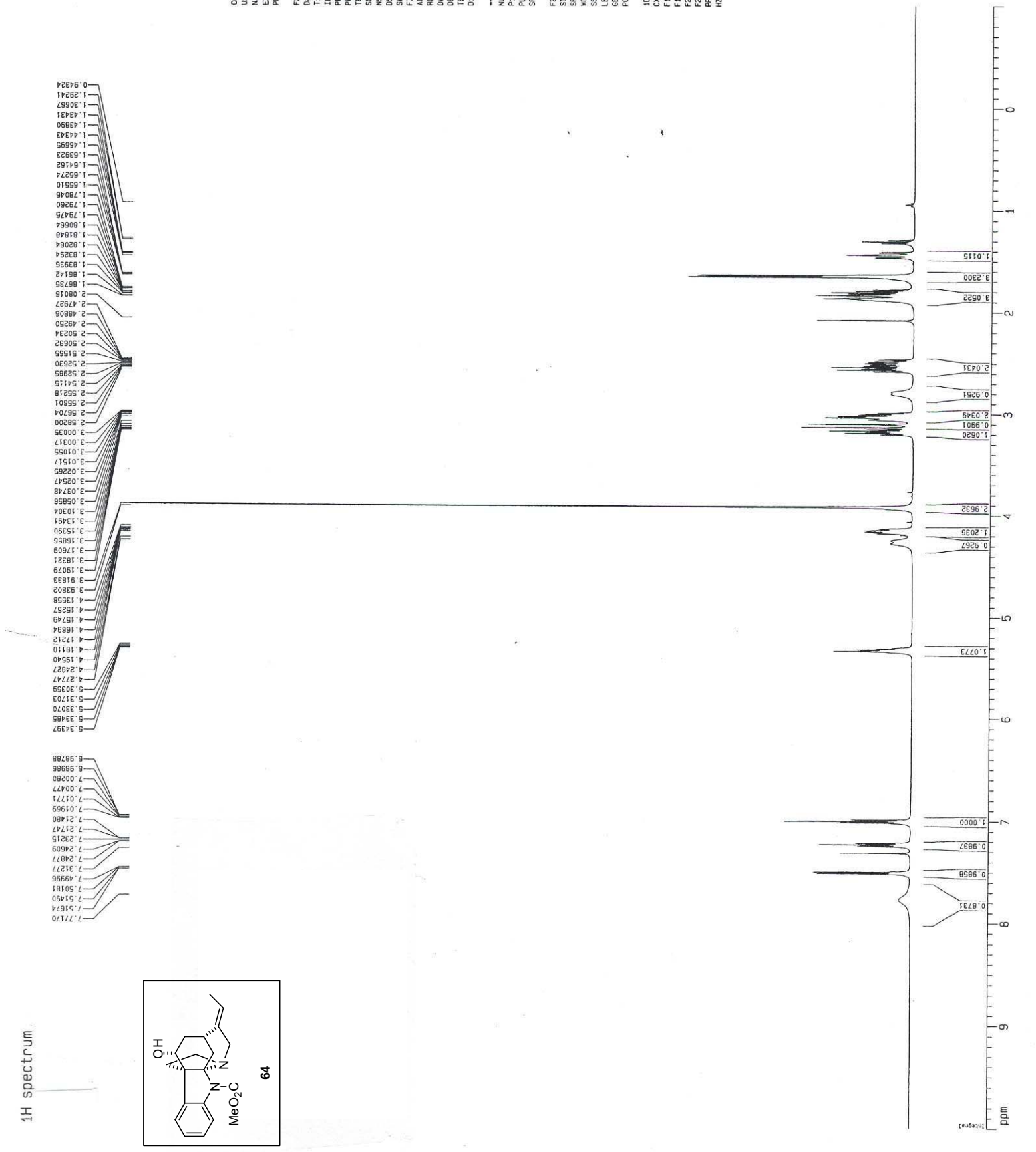

1H spectrum
ppm
Integral
OTES
MeO2C
63
+
OTES
MeO2C
48
(ca. 40%)

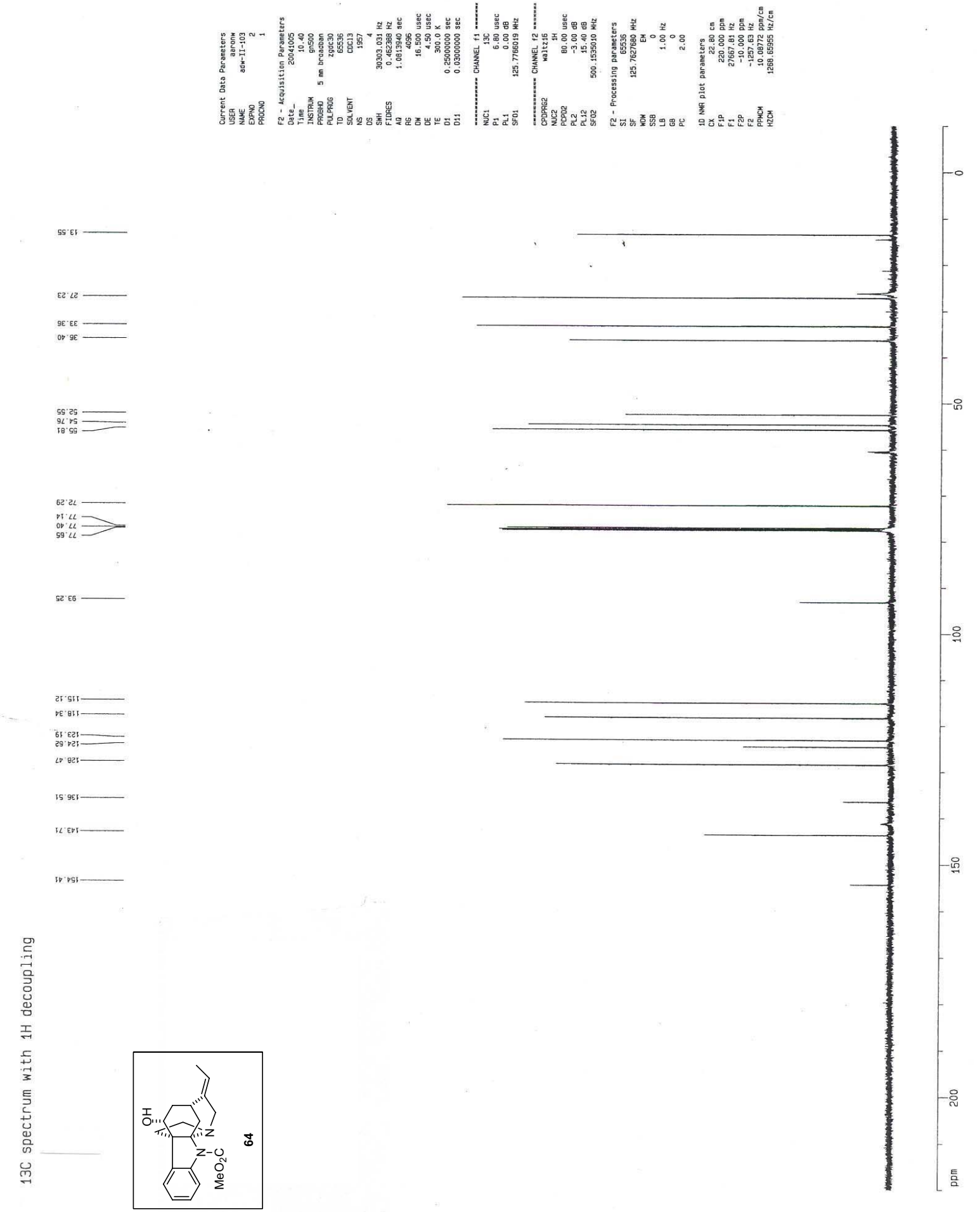

13C spectrum with 1H decoupling
ppm
OTES
MeO2C
63
+
OTES
MeO2C
48
(ca. 40%)

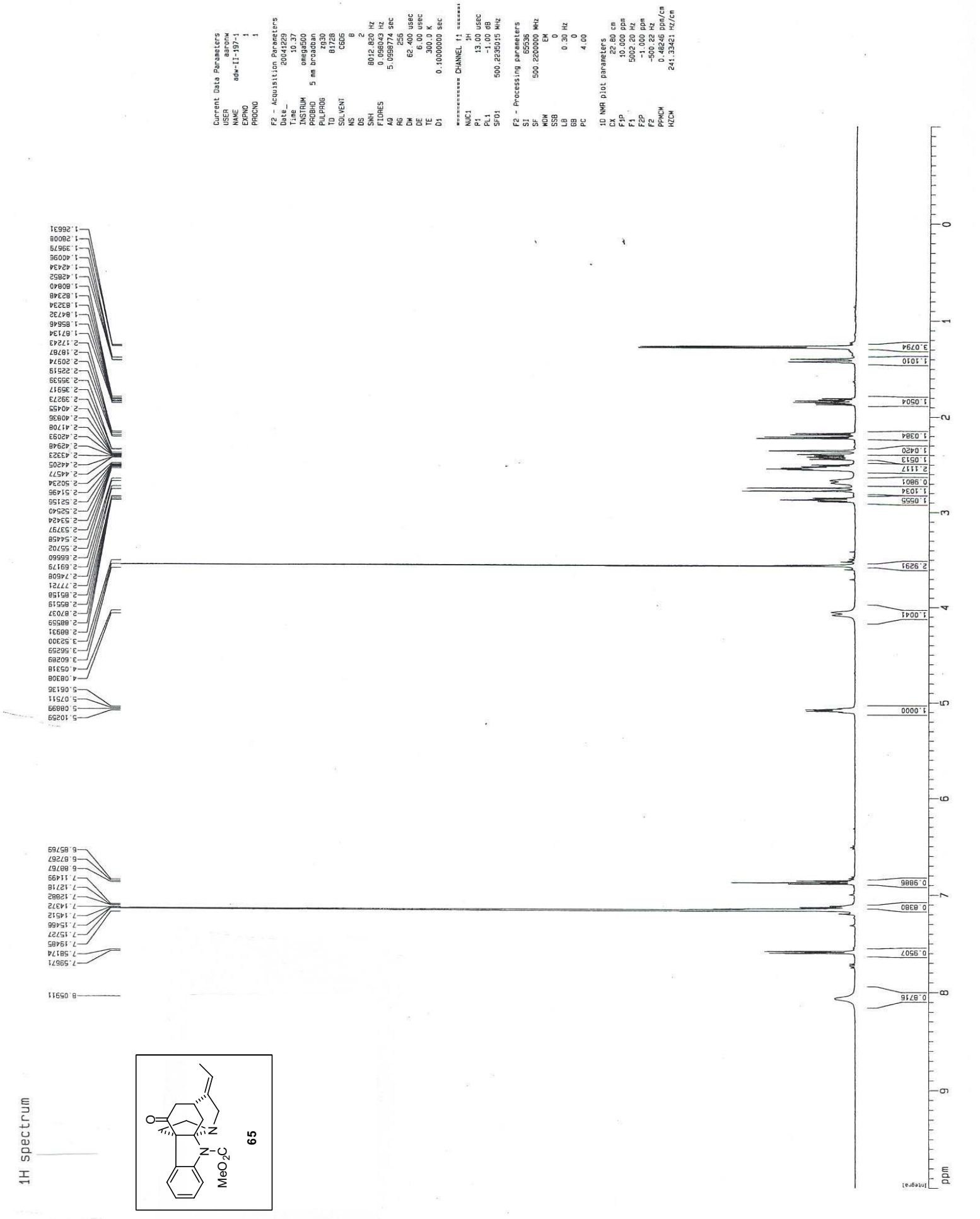

Current Data Parameters
USER aaronw
NAME adw-II-102
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040929
Time 17.09
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
5.0999
9.3771
1.2439
3.2007
1.1178
1.0857
1.0448
1.1204
1.0321
1.2130
1.0864
1.0808
3.0991
1.0765
0.8936
1.0240
1.0463
1.1489
1.0000
0.7571
ppm
OTES
N
N
MeO2C
58
1H spectrum

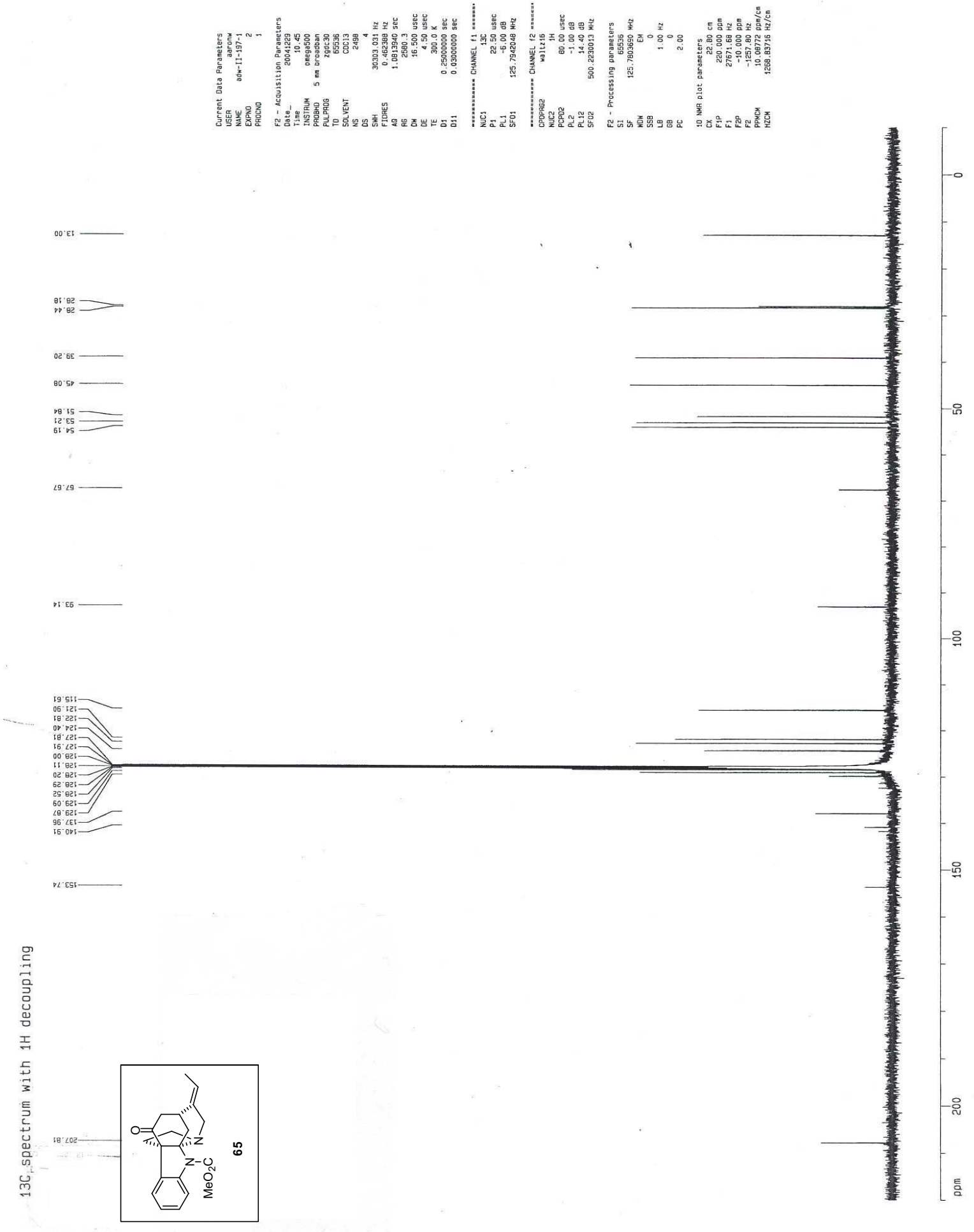


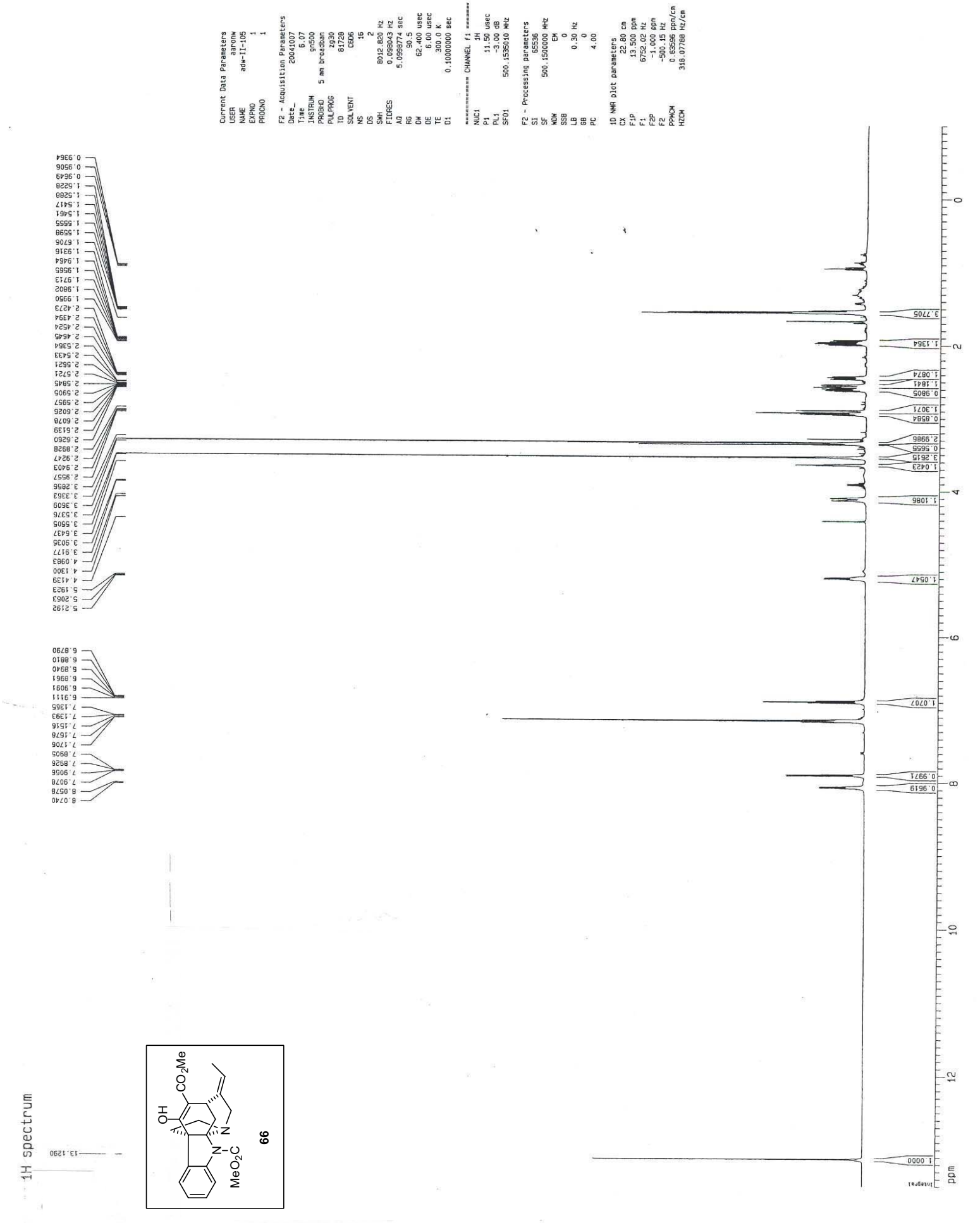
13C spectrum with 1H decoupling

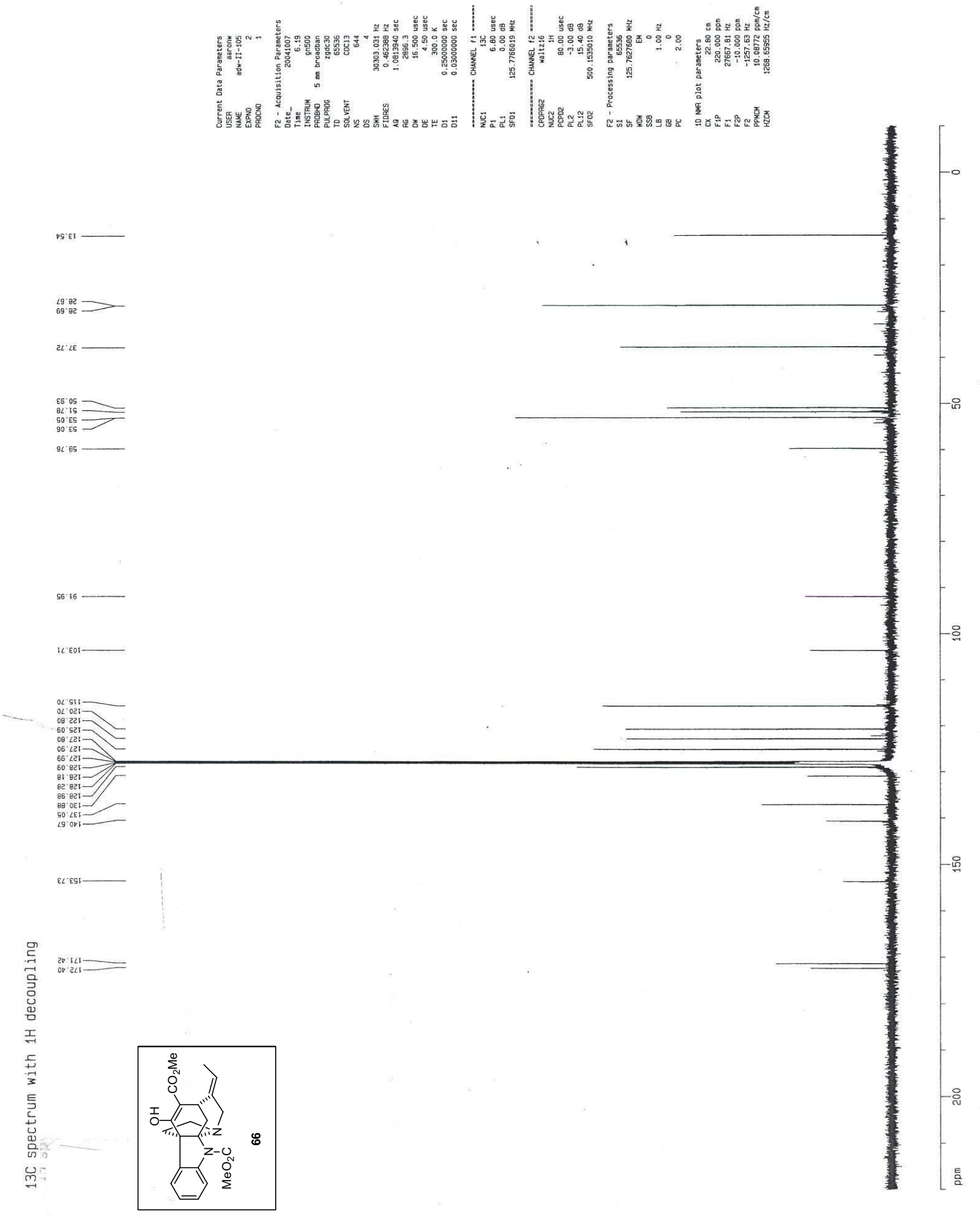

Current Data Parameters
USER aaronw
NAME adw-II-103
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041005
Time 10.35
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 6012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0988774 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
1.0115
3.2300
3.0522
2.0431
0.9261
2.0349
0.9901
1.0620
2.9632
1.2036
0.9267
1.0773
1.0000
0.9837
0.9858
0.8731
integral
ppm
1H spectrum
OH
N
N
MeO2C
64

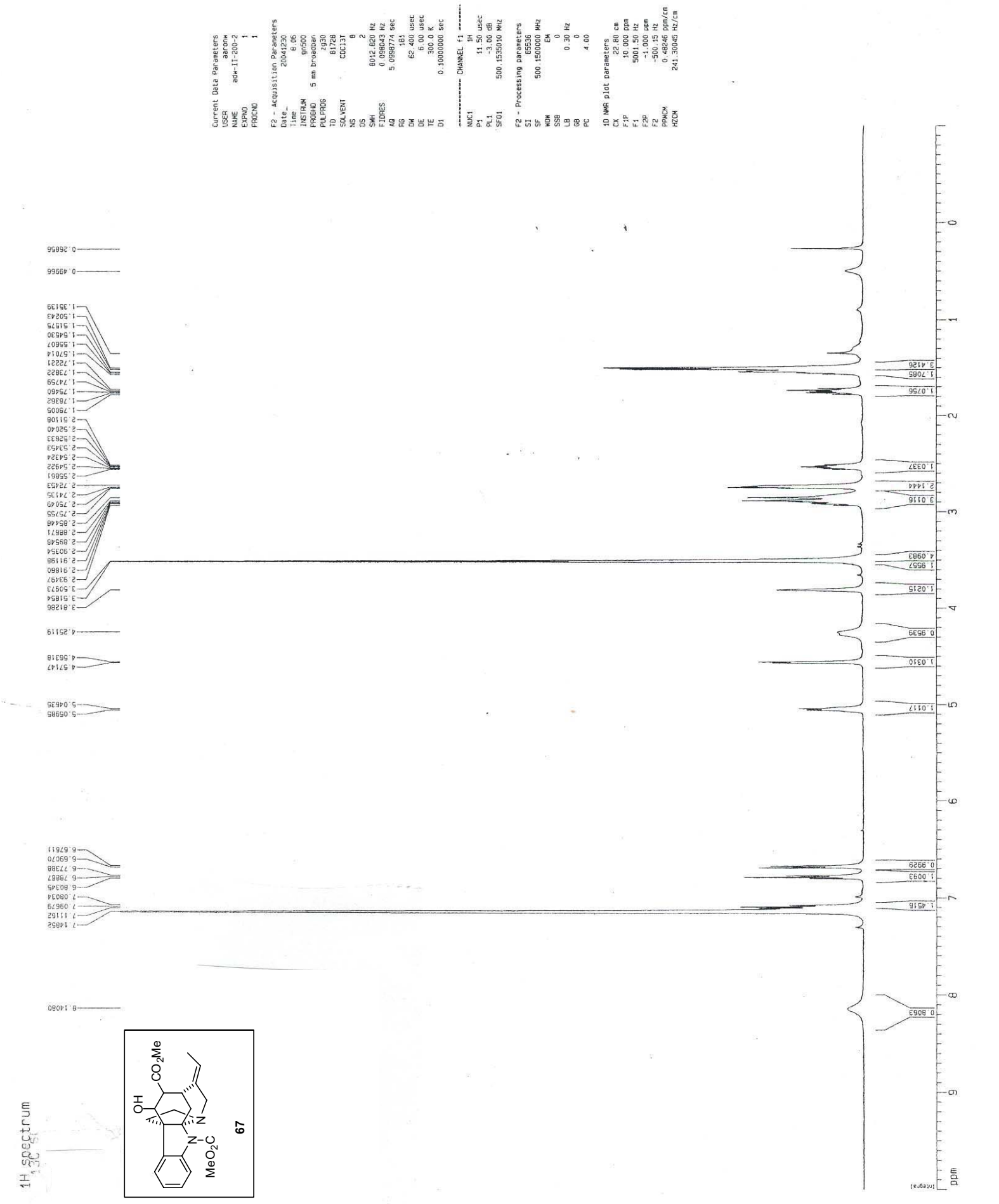

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-103
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041005
Time 10.40
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgpc30
TD 65536
SOLVENT CDCl3
NS 1957
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7760019 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1266.65955 Hz/cm
13.55
27.23
33.36
36.40
52.55
54.76
55.81
72.29
77.14
77.40
77.65
93.25
115.12
118.34
123.19
124.62
128.47
136.51
143.71
154.41
0
50
100
150
200
ppm
OH
N
N
MeO2C
64

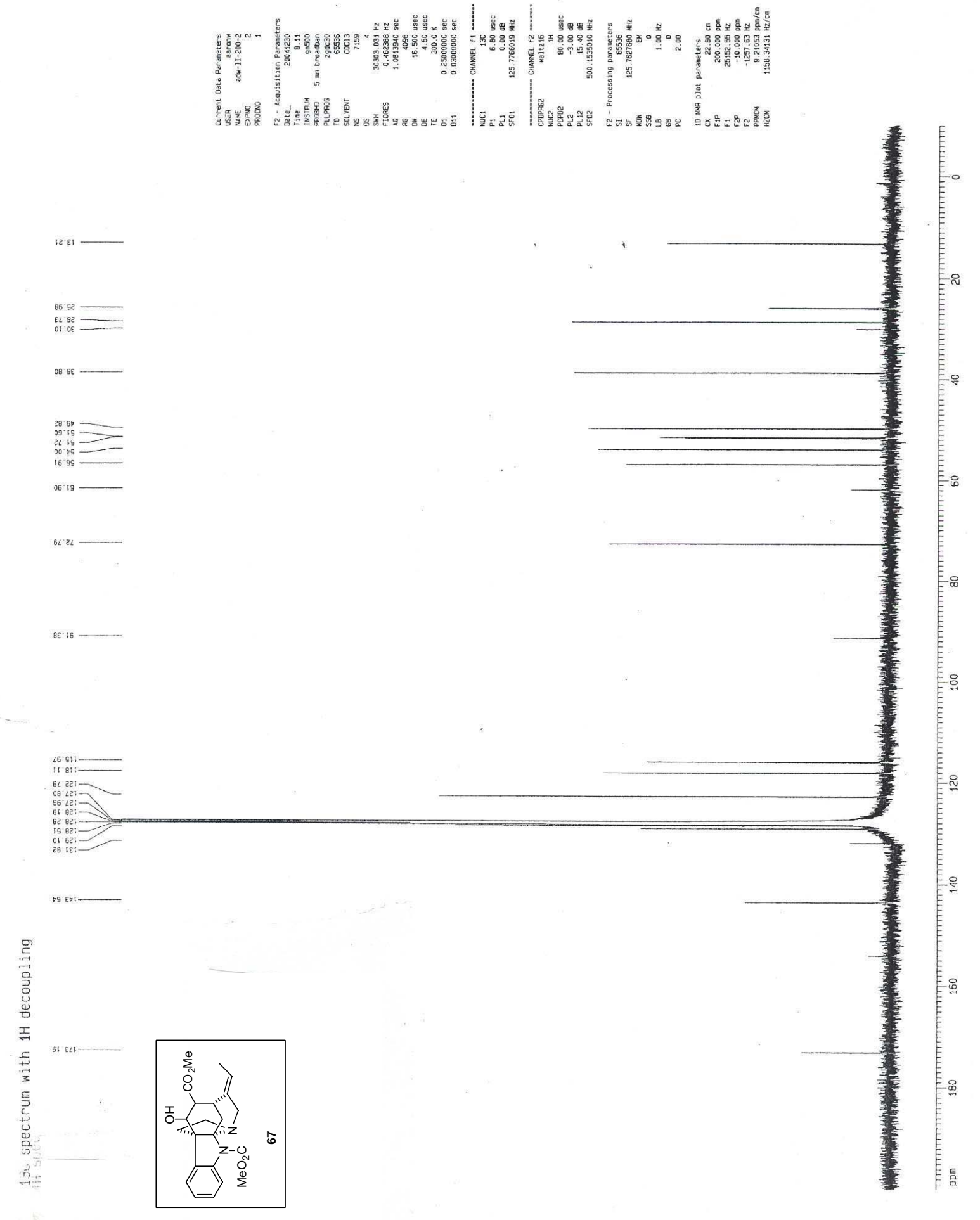

1H spectrum
65
MeO2C
ppm

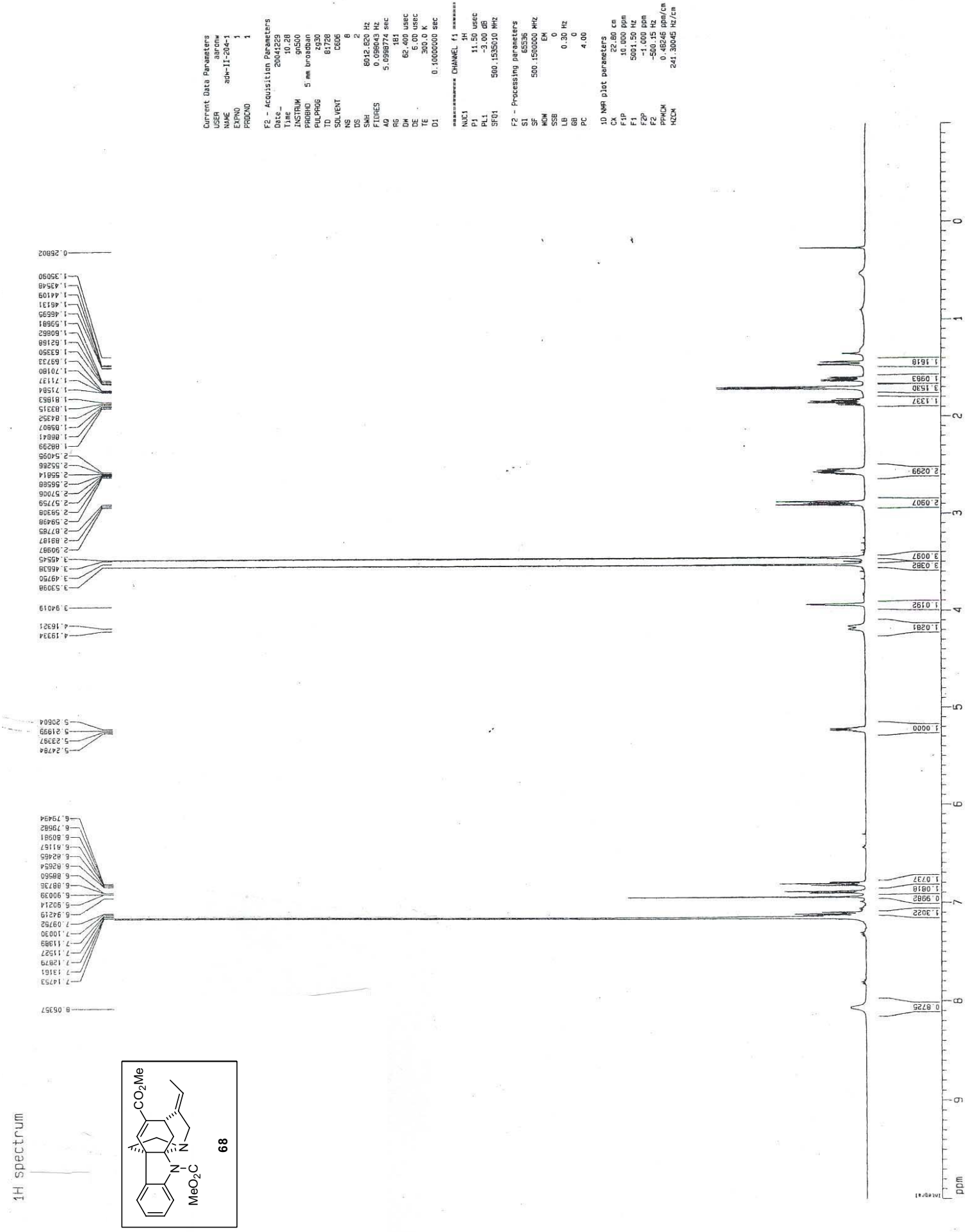

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-197-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041229
Time 10.45
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2498
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2580.3
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27671.68 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83716 Hz/cm
13.00
28.18
28.44
39.20
45.08
51.84
53.21
54.19
57.67
93.14
115.61
121.90
122.81
124.40
127.81
127.91
128.00
128.11
128.20
128.29
128.52
128.59
129.09
129.87
137.96
140.91
153.74
207.81
MeO2C
65
0
50
100
150
200
ppm

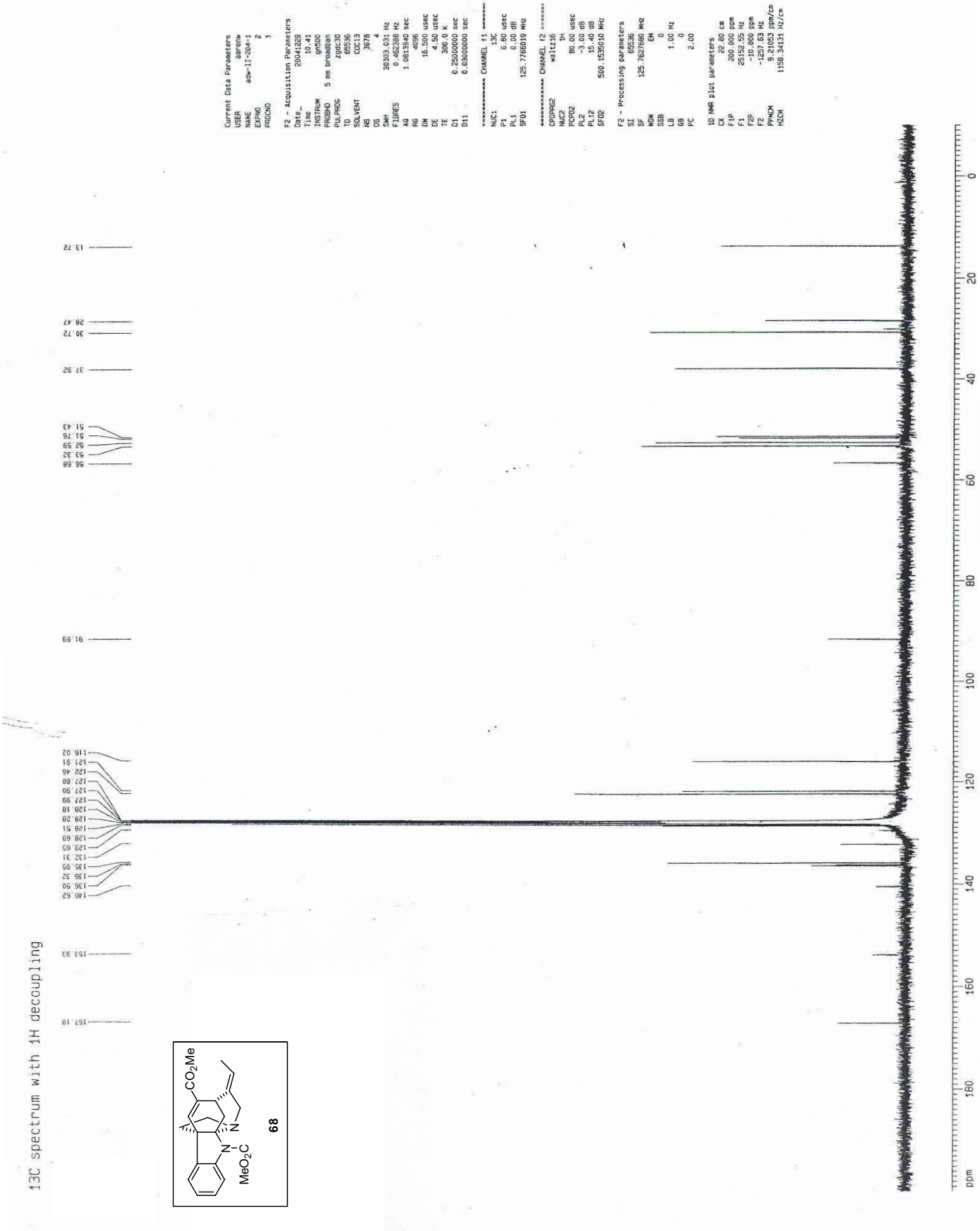

Current Data Parameters
USER aaronw
NAME adw-II-105
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041007
Time 6.07
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.099774 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SF01 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 13.500 ppm
F1 6752.02 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.63596 ppm/cm
HZCM 318.07788 Hz/cm
1H spectrum
ppm
integral
66

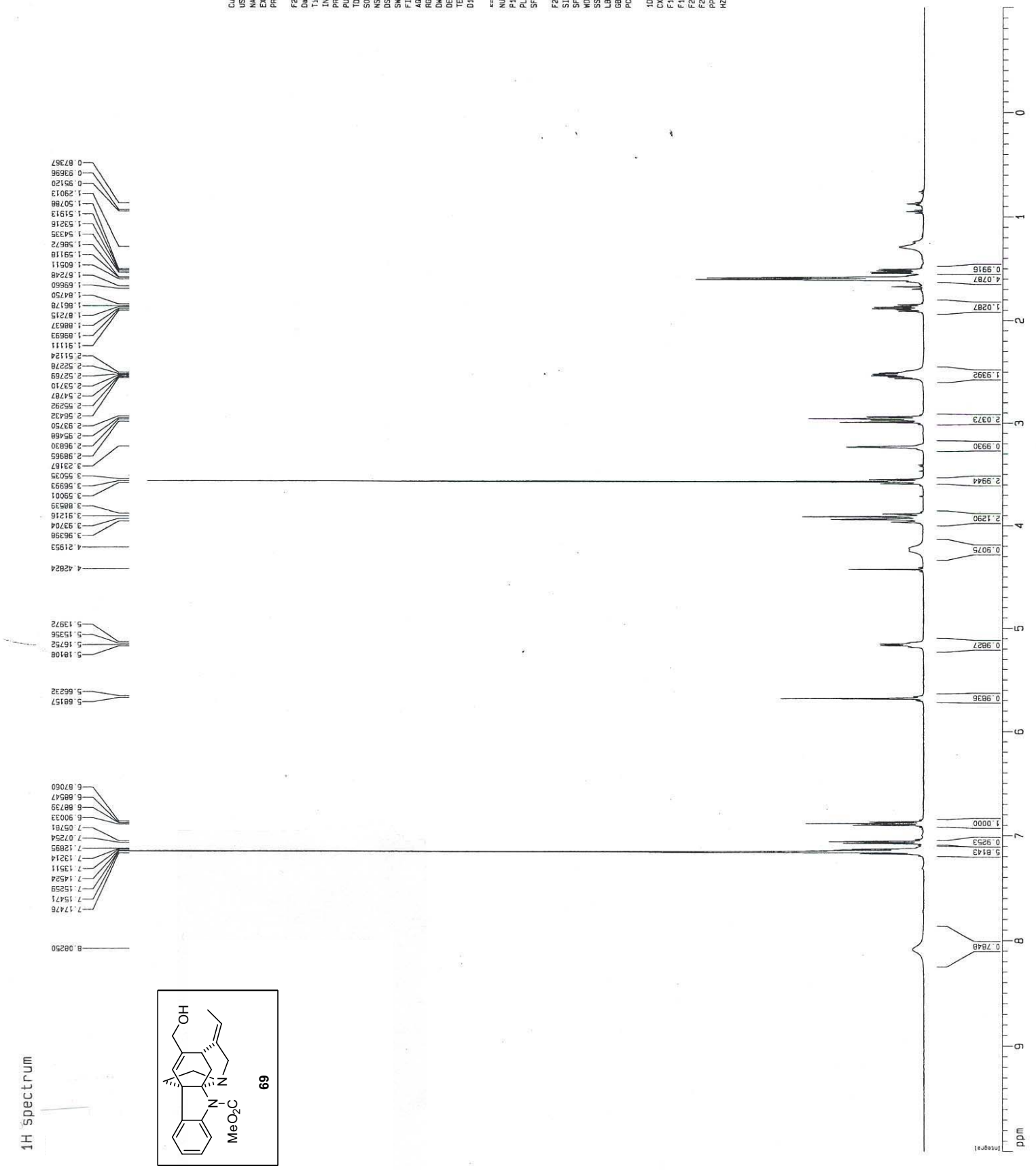

13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-II-105
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041007
Time 6.19
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 644
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2896.3
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 =======
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
========== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627600 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27667.81 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.69955 Hz/cm
13.54
28.67
28.69
37.72
50.93
51.78
53.05
53.06
59.76
91.95
103.71
115.70
120.70
122.80
125.09
127.80
127.90
127.99
128.09
128.18
128.28
128.98
130.88
137.05
140.67
153.73
171.42
172.40
0
50
100
150
200
ppm
OH
CO2Me
N
N
N
MeO2C
66

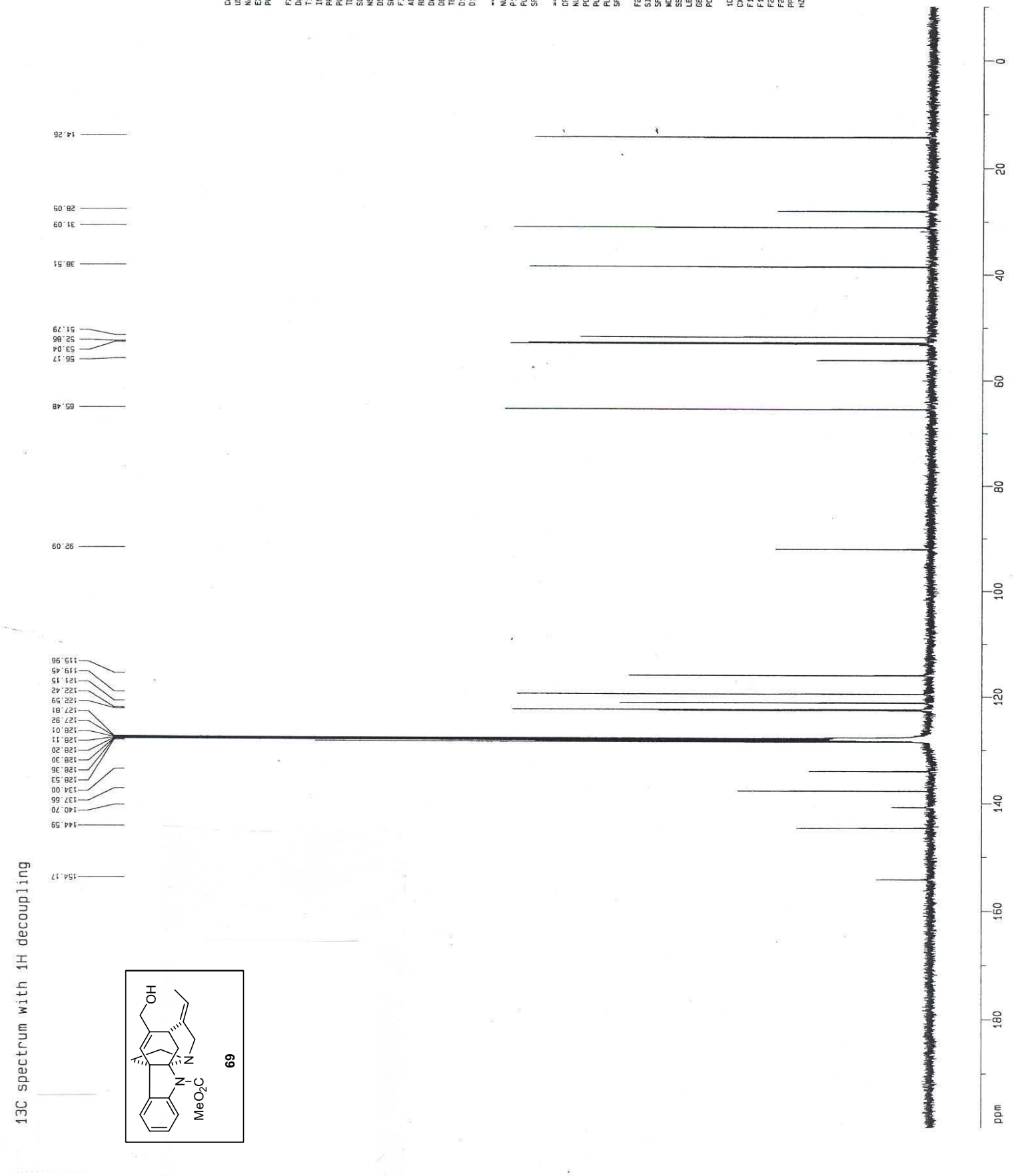

Current Data Parameters
USER aaronw
NAME adw-II-200-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041230
Time 6.06
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 181
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5001.50 Hz
F2P -1.000 ppm
F2 -500.15 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.30045 Hz/cm
8.14080
7.14852
7.11162
7.09679
7.08034
6.80345
6.78867
6.77388
6.69070
6.67611
5.05985
5.04635
4.57147
4.56318
4.25119
3.81286
3.51854
3.50973
2.93497
2.91860
2.91198
2.90354
2.89549
2.88671
2.85448
2.79755
2.75049
2.74135
2.72453
2.55861
2.54922
2.54324
2.53453
2.52633
2.52040
2.51108
1.97005
1.96362
1.95460
1.94759
1.93822
1.92221
1.90607
1.89530
1.88575
1.87243
1.85139
0.49866
0.26856
3.4126
1.7085
1.0756
1.0337
2.1444
3.0116
4.0983
1.8557
1.0215
0.9539
1.0310
1.0117
0.9929
1.0093
1.4516
0.8063
integral
ppm
1H spectrum
OH
CO2Me
N
N
MeO2C
67

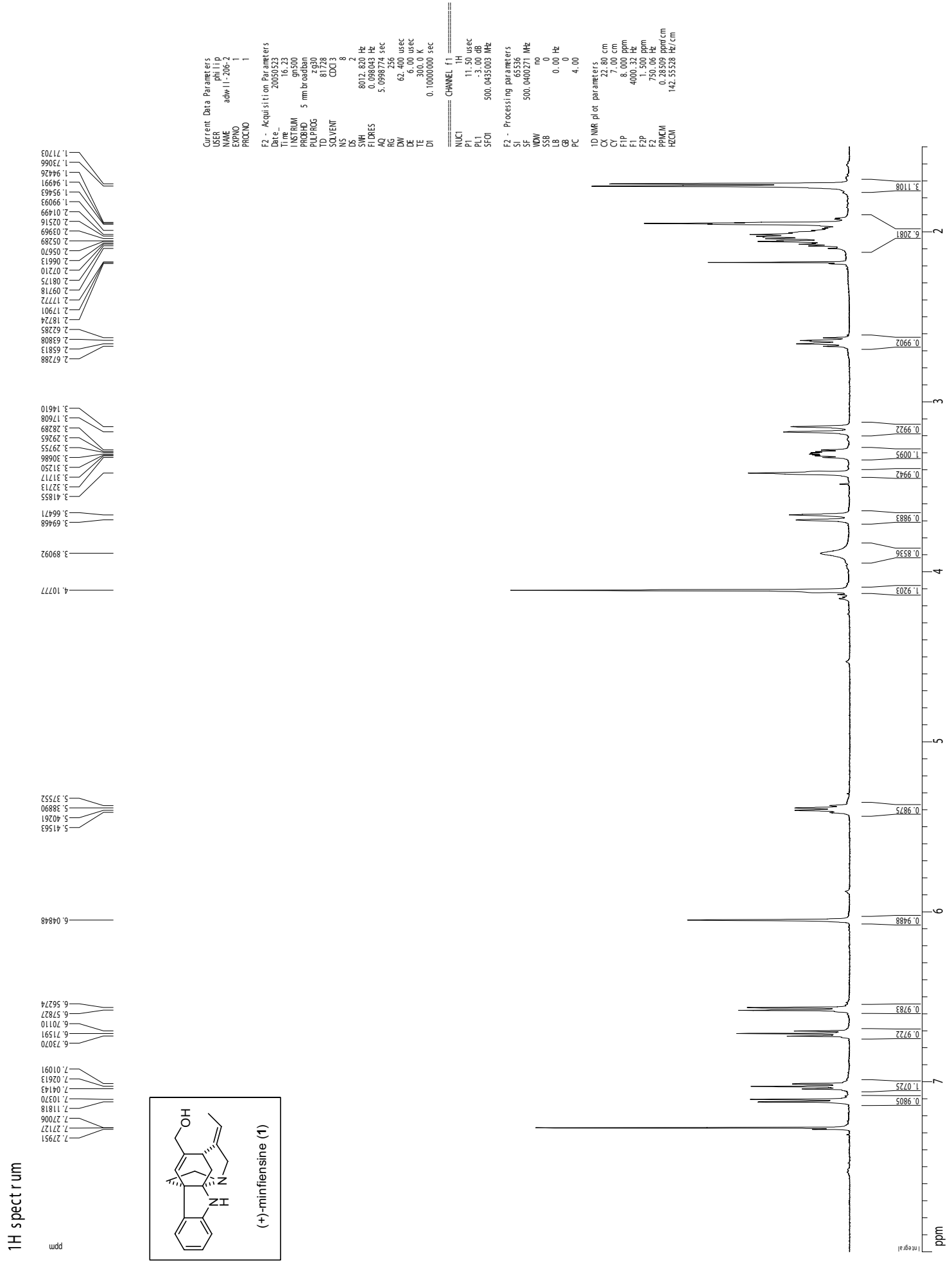

Current Data Parameters
USER aaronw
NAME adw-II-200-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041230
Time 8.11
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 7159
DS 4
SWH 30303.031 Hz
FIDRES 0.462368 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7768019 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25152.55 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.34131 Hz/cm
13C spectrum with 1H decoupling
13.21
25.98
28.73
30.10
38.80
49.82
51.60
51.72
54.00
56.91
61.90
72.79
91.38
115.97
118.11
122.78
127.80
127.95
128.10
128.28
128.51
129.10
131.92
143.64
173.19
ppm
0
20
40
60
80
100
120
140
160
180
OH
CO2Me
N
N
MeO2C
67


1H spectrum
68
CO_2Me
MeO_2C
ppm

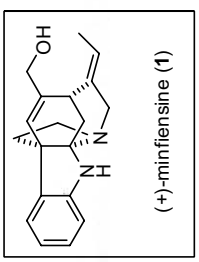

13C spectrum with 1H decoupling
Current Data Parameters
USER aarow
NAME adw-11-204-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041229
Time 10.41
INSTRUM spect
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 3678
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 6.60 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.40 dB
SFO2 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 125.7627680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25152.55 Hz
F2P -10.000 ppm
F2 -1257.63 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.34131 Hz/cm
13.72
20.47
30.72
37.92
51.43
51.76
52.59
53.32
56.68
91.69
116.02
121.91
122.46
127.08
127.90
127.99
128.01
128.28
128.51
128.69
129.65
132.31
135.95
136.32
136.50
140.62
153.93
157.19
ppm
0
20
40
60
80
100
120
140
160
180
CO2Me
N
N
MeO2C
68


Current Data Parameters
USER aaronw
NAME adw-11-205
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041221
Time 9.26
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT C606
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 64
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2335015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33421 Hz/cm
integral
ppm
1H spectrum
OH
N
N
MeO2C
69


Current Data Parameters
USER aaronw
NAME adw-II-205
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041221
Time 9.31
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 809
DS 4
SWH 30303.031 Hz
FIDRES 0.462398 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ======
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz
========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz
F2 - Processing parameters
SI 65536
SF 125.7803690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25156.07 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.50342 Hz/cm
14.26
28.05
31.09
38.51
51.79
52.86
53.04
55.17
65.48
92.09
115.96
119.45
121.15
122.42
122.59
127.81
127.92
128.01
128.11
128.20
128.30
128.36
128.53
134.00
137.66
140.70
144.59
154.17
0
20
40
60
80
100
120
140
160
180
ppm
13C spectrum with 1H decoupling
OH
N
N
MeO2C
69


1H spectrum
(+)-minfiensine (1)
OH
N
H
N
Current Data Parameters
USER philip
NAME adwll-206-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050523
Time 16.23
INSTRUM gn500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1 1H
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.0435003 MHz
F2 - Processing parameters
SI 65536
SF 500.0400271 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 7.00 cm
F1P 8.000 ppm
F1 4000.32 Hz
F2P 1.500 ppm
F2 750.06 Hz
PPMCM 0.28509 ppm/cm
HZCM 142.55528 Hz/cm
integral
ppm

13C spectrum with 1H decoupling



(+)-minfiensine (1)

Current Data Parameters
USER aaronw
NAME adw-II-206-1
EXPNO 2
PROCNO 1

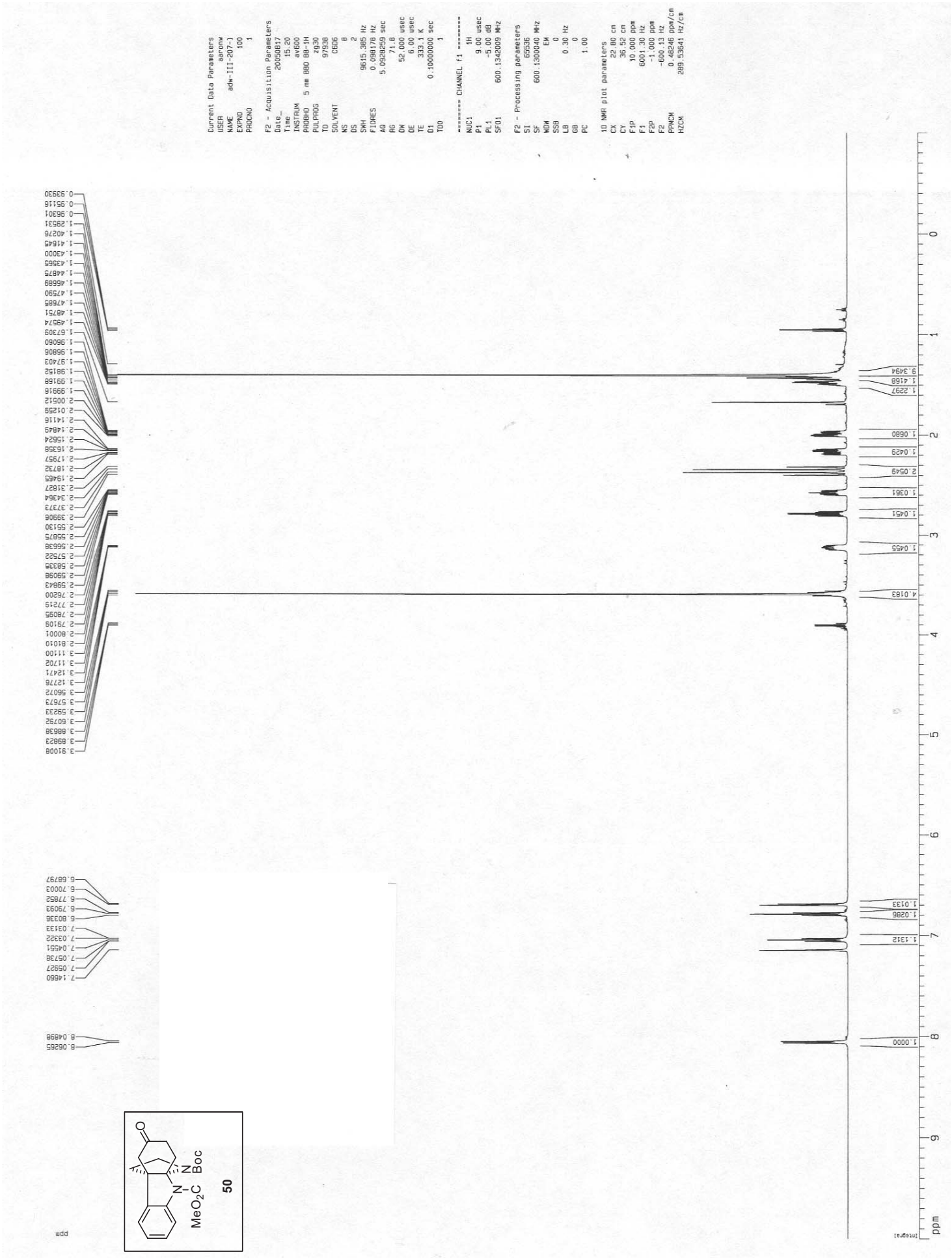
F2 - Acquisition Parameters
Date_ 20041222
Time 15.05
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 725
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3649.1
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec

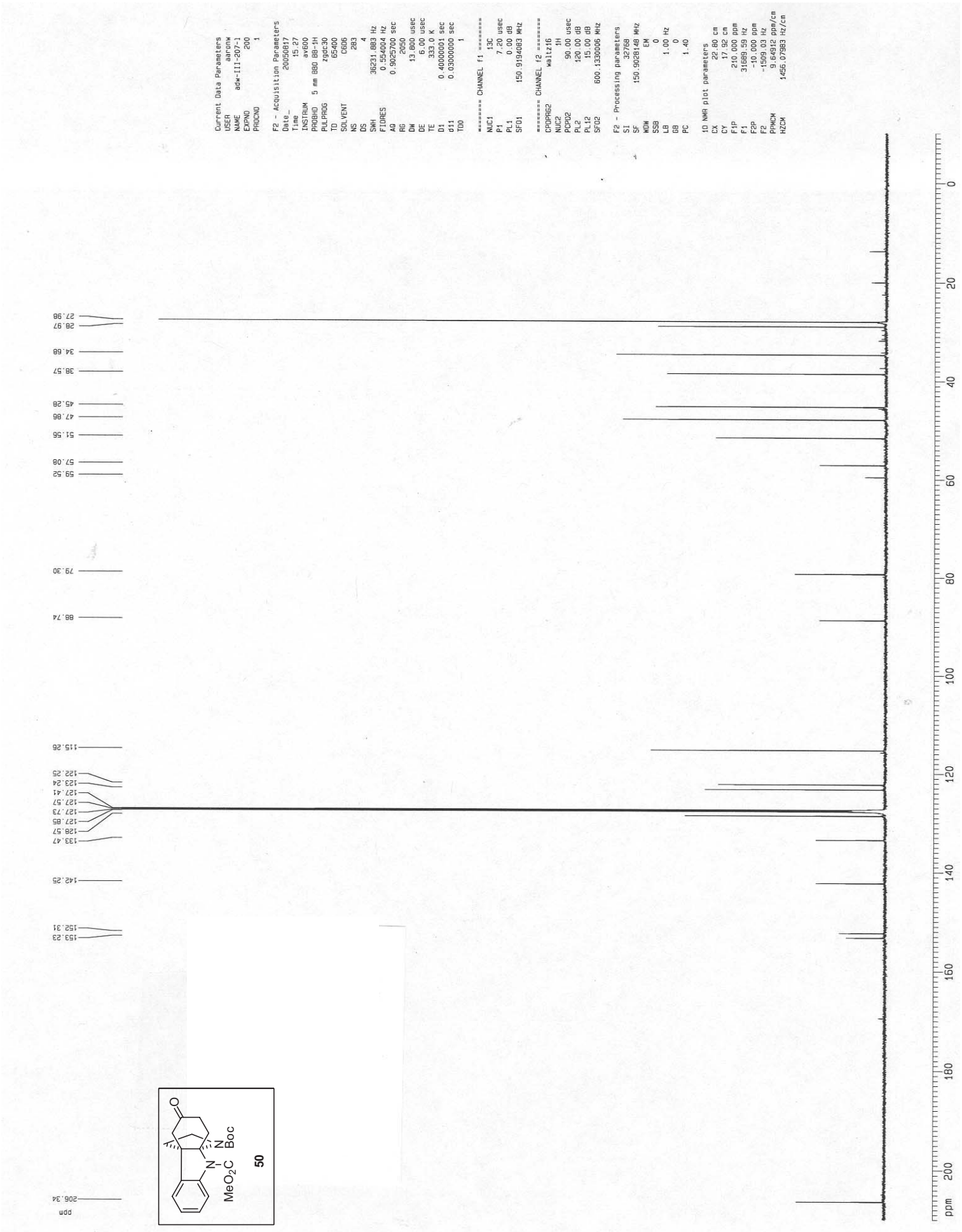
========== CHANNEL f1 ========
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz

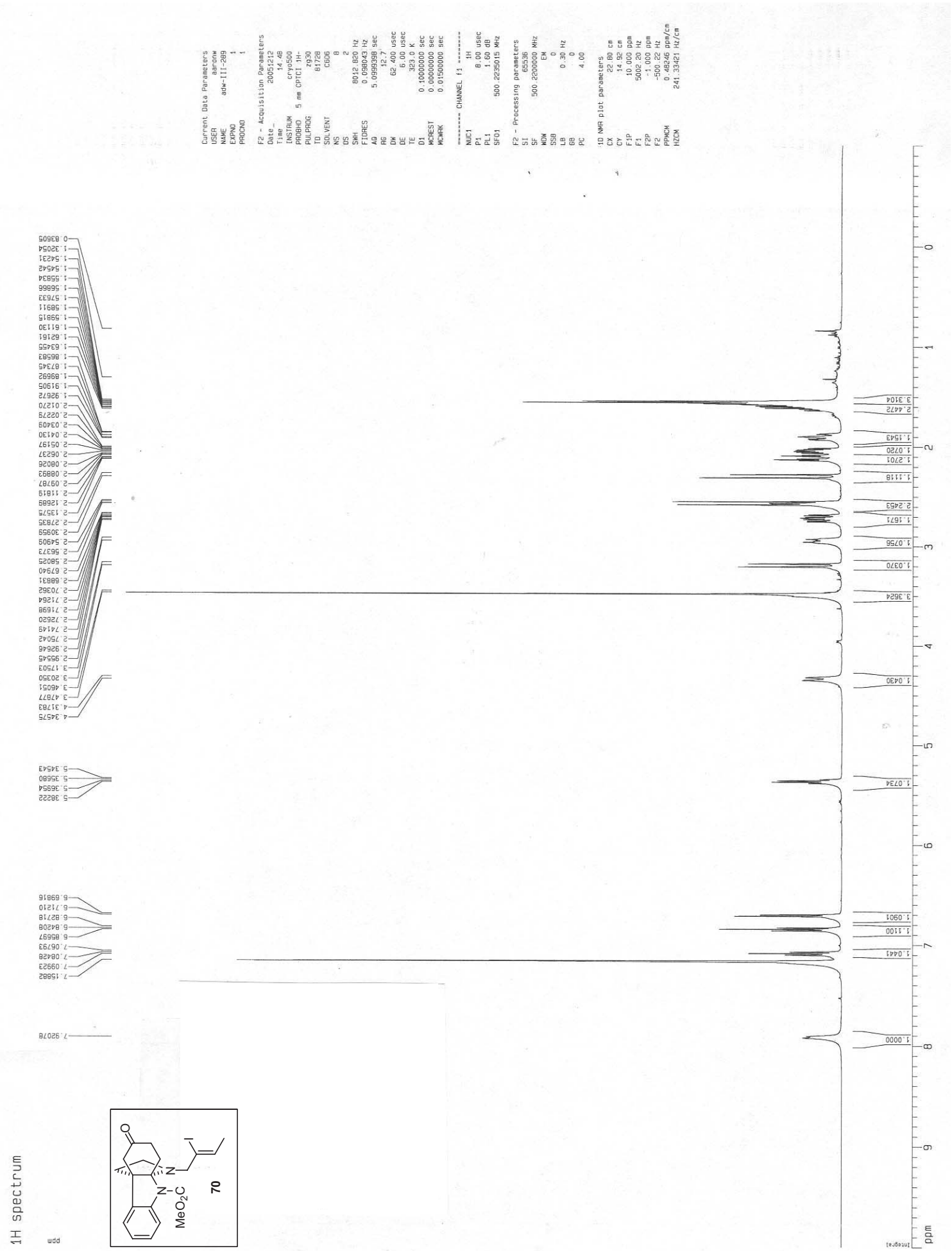
========== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz

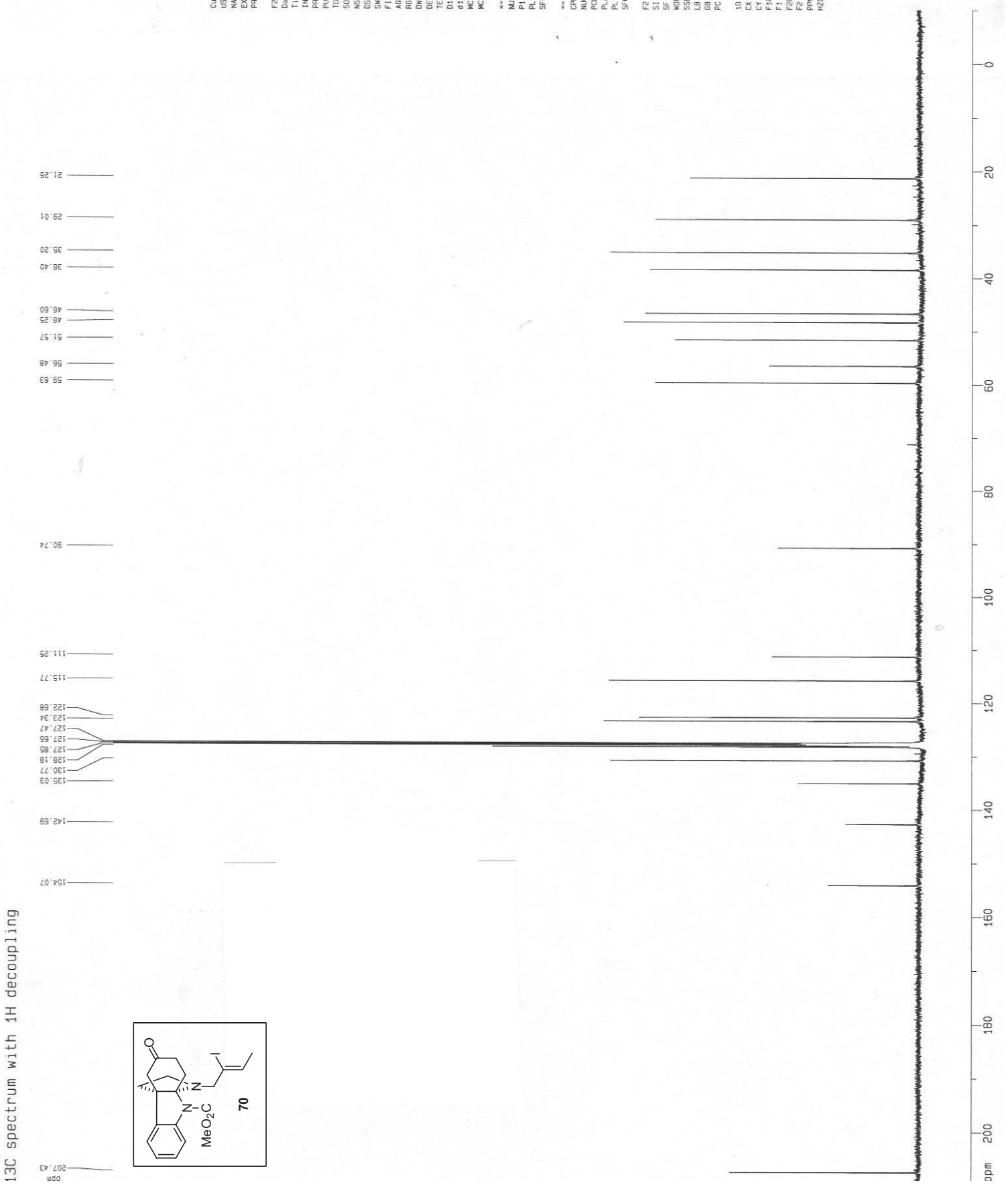
F2 - Processing parameters
SI 65536
SF 125.7804373 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

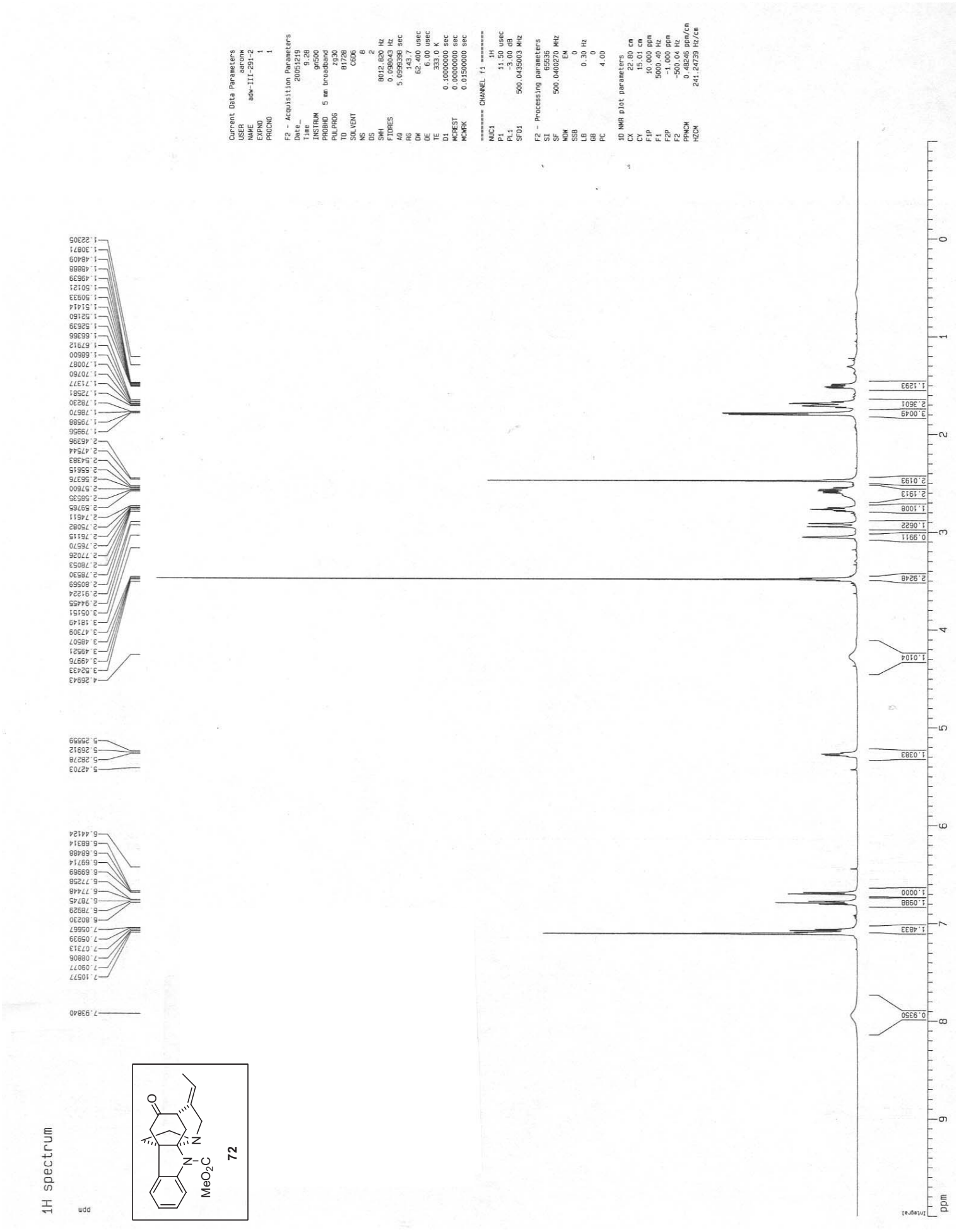
1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25156.08 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.21053 ppm/cm
HZCM 1158.50391 Hz/cm

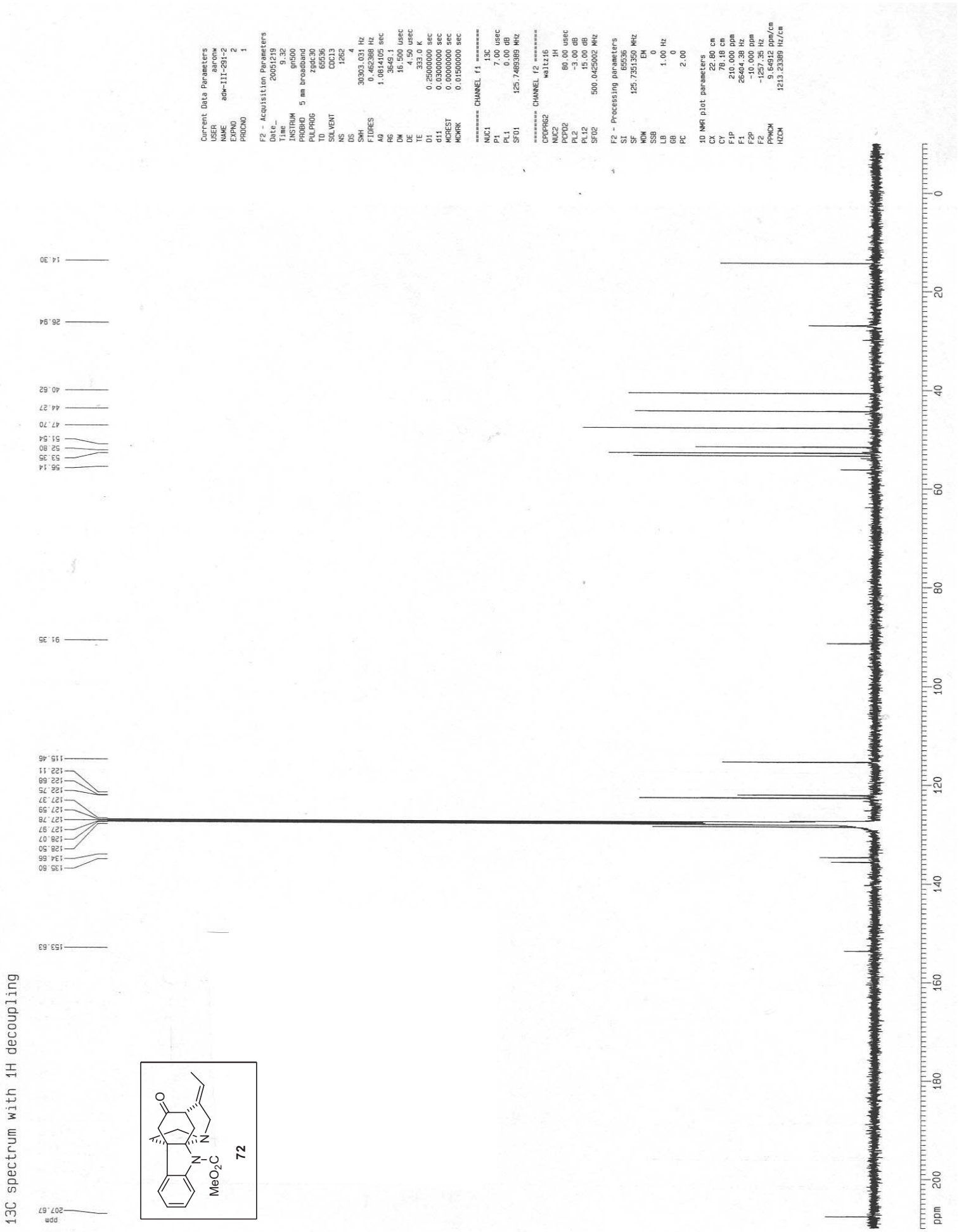

Current Data Parameters
USER aaronw
NAME adw-III-207-1
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050817
Time 15.20
INSTRUM av600
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 97938
SOLVENT CDCl3
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928259 sec
RG 71.8
DW 52.000 usec
DE 6.00 usec
TE 333.1 K
D1 0.10000000 sec
TD0 1
======= CHANNEL f1 =======
NUC1 1H
P1 9.00 usec
PL1 -5.00 dB
SFO1 600.1342009 MHz
F2 - Processing parameters
SI 65536
SF 600.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 36.52 cm
F1P 10.000 ppm
F1 6001.30 Hz
F2P -1.000 ppm
F2 -600.13 Hz
PPMCM 0.48246 ppm/cm
HZCM 289.53641 Hz/cm
ppm
Integral
MeO2C
Boc
N
50

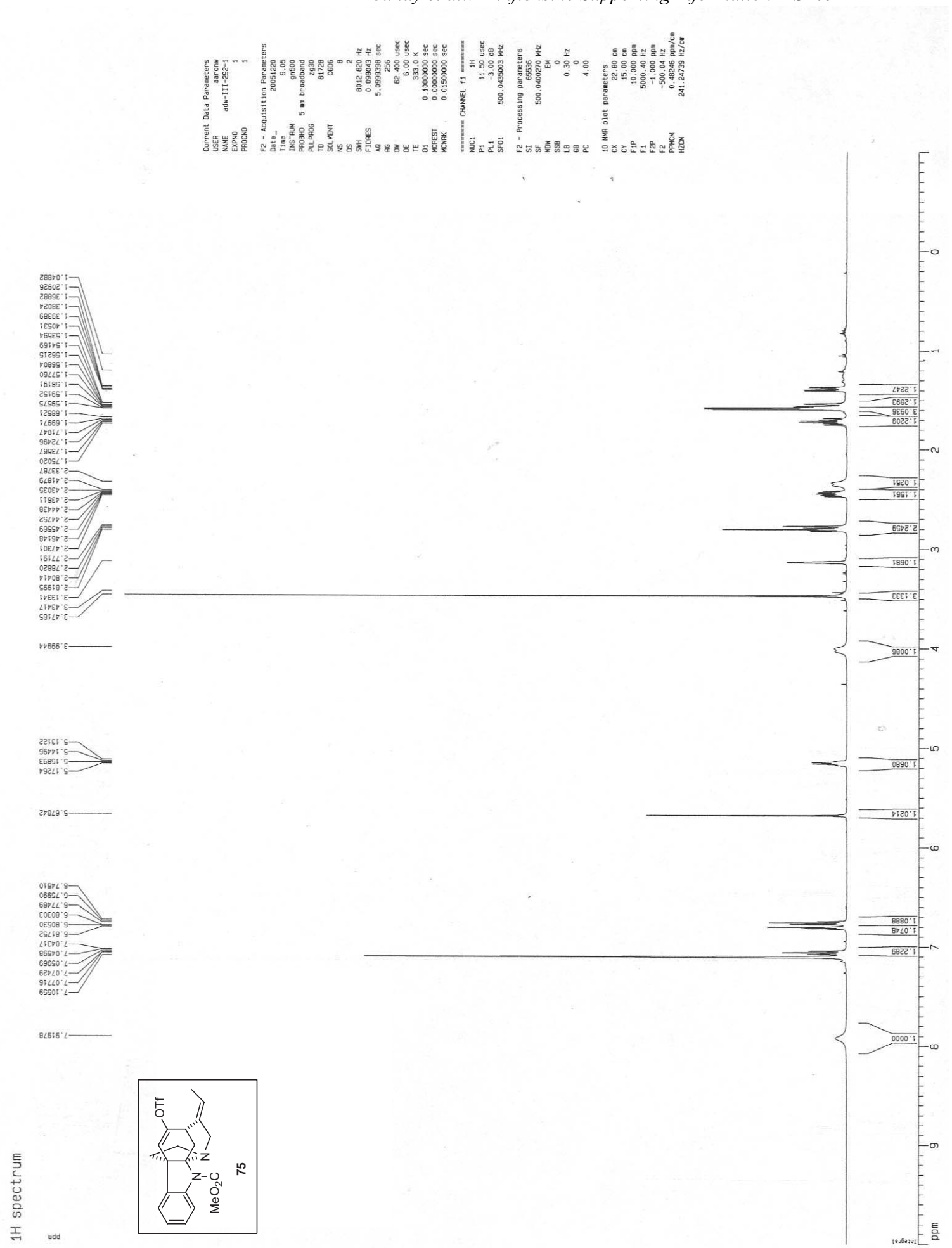

Current Data Parameters
USER aaronw
NAME adw-III-207-1
EXPNO 200
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050817
Time 15.27
INSTRUM av600
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65400
SOLVENT CDCl3
NS 283
DS 4
SWH 36231.883 Hz
FIDRES 0.554004 Hz
AQ 0.9025700 sec
RG 2050
DW 13.800 usec
DE 6.00 usec
TE 333.0 K
D1 0.40000001 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 0.00 dB
SFO1 150.9194083 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 15.00 dB
SFO2 600.1330006 MHz
F2 - Processing parameters
SI 32768
SF 150.9028148 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.80 cm
CY 17.92 cm
F1P 210.000 ppm
F1 31689.59 Hz
F2P -10.000 ppm
F2 -1509.03 Hz
PPMCM 9.64912 ppm/cm
HZCM 1456.07983 Hz/cm
27.98
28.97
34.68
38.57
45.28
47.86
51.56
57.08
59.52
79.30
88.74
115.26
122.25
123.24
127.41
127.57
127.73
127.85
128.57
133.47
142.25
152.31
153.23
206.34
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm
O
N
N
Boc
MeO2C
50

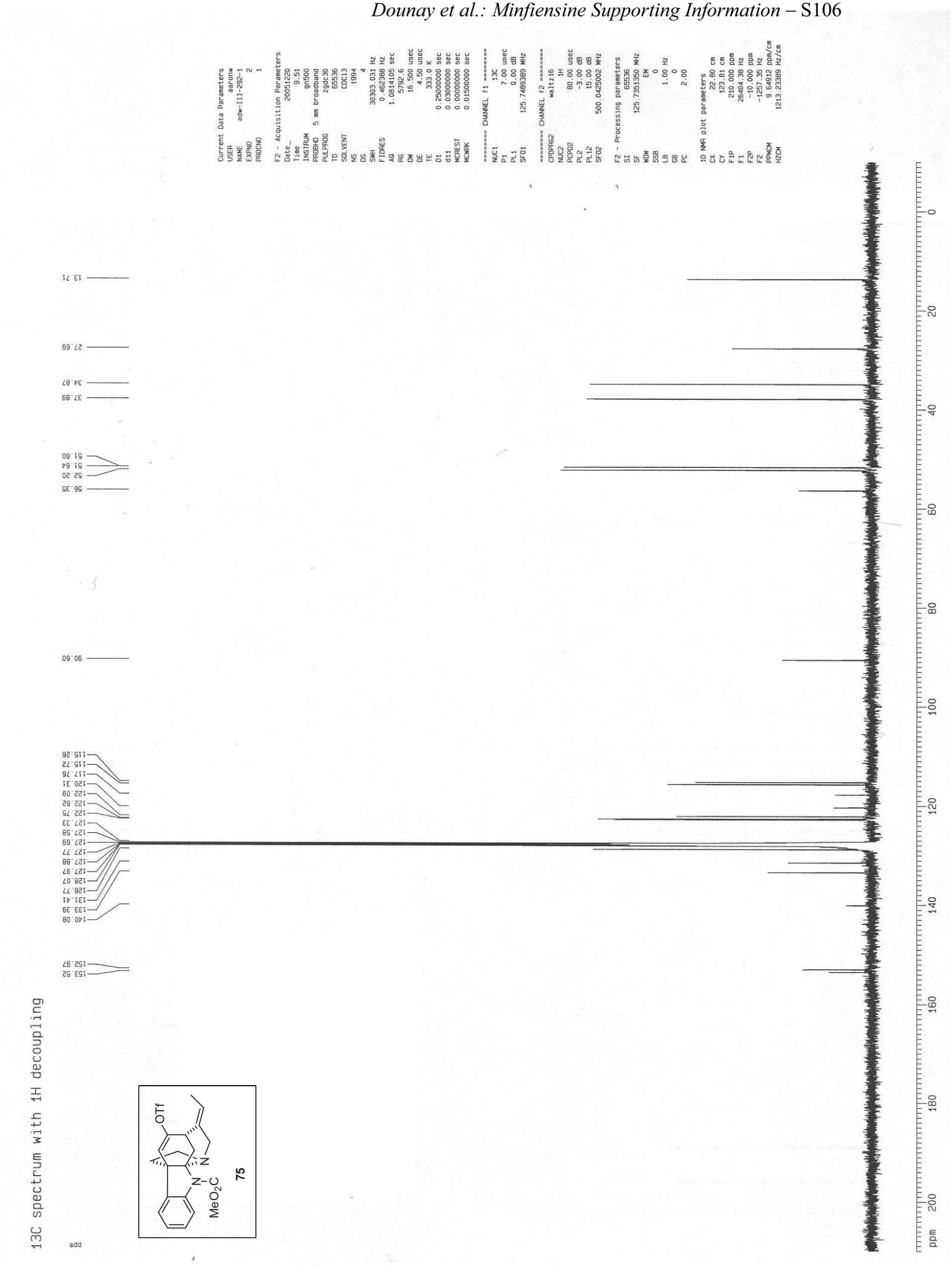

1H spectrum
Current Data Parameters
USER aaronw
NAME adw-III-289
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051212
Time 14.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 12.7
DW 62.400 usec
DE 6.00 usec
TE 323.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 14.92 cm
F1P 10.000 ppm
F1 5002.28 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.40246 ppm/cm
HZCM 241.33421 Hz/cm
MeO2C
70
ppm
Integral


13C spectrum with 1H decoupling
Current Data Parameters
USER aaronw
NAME adw-III-289
EXPNO 300
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051212
Time 15.00
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65410
SOLVENT CDCl3
NS 230
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 5792.6
DW 16.500 usec
DE 6.00 usec
TE 323.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SF01 125.7942540 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SF02 500.2225011 MHz
F2 - Processing parameters
SI 65536
SF 125.7604190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 37.86 cm
F1P 210.000 ppm
F1 26413.89 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67078 Hz/cm
21.26
29.01
35.20
38.40
46.60
48.25
51.57
56.48
59.63
90.74
111.25
115.77
122.68
123.34
127.47
127.66
127.85
128.18
130.77
135.03
142.69
154.07
207.43
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm
MeO2C
70


1H spectrum
Current Data Parameters
USER aaronw
NAME adw-III-291-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051219
Time 9.28
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 143.7
DW 62.400 usec
DE 6.00 usec
TE 333.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SF01 500.0435003 MHz
F2 - Processing parameters
SI 65536
SF 500.0400270 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.01 cm
F1P 10.000 ppm
F1 5000.40 Hz
F2P -1.000 ppm
F2 -500.04 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.24739 Hz/cm
MeO2C
72
ppm
Integral


13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER aaronw
NAME adw-III-291-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051219
Time 9.32
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgdc30
TD 65536
SOLVENT CDC13
NS 1262
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 3649.1
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 125.7489389 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
SFO2 500.0425002 MHz
F2 - Processing parameters
SI 65536
SF 125.7351350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 78.18 cm
F1P 210.000 ppm
F1 26404.38 Hz
F2P -10.000 ppm
F2 -1257.35 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.23389 Hz/cm
207.67
153.63
135.60
134.66
128.50
128.07
127.97
127.78
127.59
127.37
122.75
122.69
122.11
115.46
91.35
56.14
53.35
52.80
51.54
47.70
44.27
40.62
26.94
14.30
MeO2C
72


1H spectrum
ppm
integral
OTf
N
N
MeO2C
75


13C spectrum with 1H decoupling
ppm
OTf
N
N
MeO_2C
75
Current Data Parameters
USER aaronw
NAME adw-III-292-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051220
Time 9.51
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1994
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 5792.6
DW 16.500 usec
DE 4.50 usec
TE 333.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 125.7489389 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
SFO2 500.0425002 MHz
F2 - Processing parameters
SI 65536
SF 125.7351350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 123.81 cm
F1P 210.000 ppm
F1 26404.38 Hz
F2P -10.000 ppm
F2 -1257.35 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.23389 Hz/cm
13.71
27.69
34.87
37.89
51.60
51.64
52.20
56.35
90.60
115.26
115.72
117.76
120.31
122.09
122.62
122.75
127.33
127.58
127.69
127.77
127.88
127.97
128.07
128.77
131.41
133.39
140.08
153.52
152.97
0
20
40
60
80
100
120
140
160
180
200
ppm