Eantioselective Mannich reaction of a highly reactive Horner-Wadsworth-Emmons reagent with imines catalyzed by a bifunctional thiourea

Depeng Zhao,[a] Dongxu Yang,[a] Yijie Wang,[a] Yuan Wang,[a] Linqing Wang,[a] Lijuan Mao,[a] and Rui Wang*[a,b]

[a] *Key Laboratory of Preclinical Study for New Drugs of Gansu Province, School of Medicine, State Key Laboratory of Applied Organic Chemistry, and Institute of Biochemistry and Molecular Biology, Lanzhou University, Lanzhou, 730000, P. R. China,*

[b] *State Key Laboratory of Chiroscience and Department of Applied Biology and Chemical Technology, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong*

wangrui@lzu.edu.cn

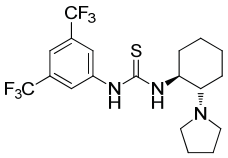
Table of Contents:

General remarks

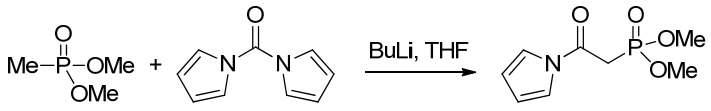
^{1}H, ^{13}C, ^{31}P NMR spectra were recorded on Bruker Avance 300. The chemical shifts are reported in ppm relative to internal standard TMS (^{1}H NMR), to residual signals of the solvents ($CHCl_3$, 7.26 ppm for ^{1}H NMR and 77.0 ppm for ^{13}C NMR) and to external standard 85% H_3PO_4 (^{31}P NMR). IR spectra were recorded on Nicolet NEXUS 670 FT-IR and only major peaks were reported. Optical rotations were measured on a Perkin-Elmer 341 polarimeter at rt. HRMS was measured with an APEX II 47e mass spectrometer. The enantiomeric excess was determined by HPLC analysis.

Materials

The catalyst **3e** was synthesized according to the procedures reported by Yoshiji Takemoto and coworkers by using (1S,2S)-2-Pyrrolidin-1-yl-cyclohexylamine and 3,5-bis(trifluoromethyl)phenyl isothiocyanate.[1] The substrate **1e** and **1c** were synthesized according to the procedures reported by Shibasaki and co-workers.[2]

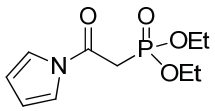


3e yellow solid; ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.09 (brs, 1H), 8.24 (brs, 1H), 8.21 (s, 2H), 7.68 (s, 1H), 4.24 (s, 1H), 3.44 (brs, 1H), 2.81 – 2.51 (m, 4H), 2.12 (d, J = 9.3 Hz, 1H), 1.83 (d, J = 12.1 Hz, 1H), 1.68 (m, 6H), 1.47 – 1.06 (m, 4H) ppm; ^{13}C NMR (75 MHz, DMSO-d_6) δ 178.8, 141.9, 130.4 (q, J = 32.6 Hz), 123.3 (q, J = 271.1 Hz), 121.2 (m), 115.5 (m), 60.8, 55.5, 47.4, 30.4, 23.9, 23.7, 23.4, 22.5 ppm.



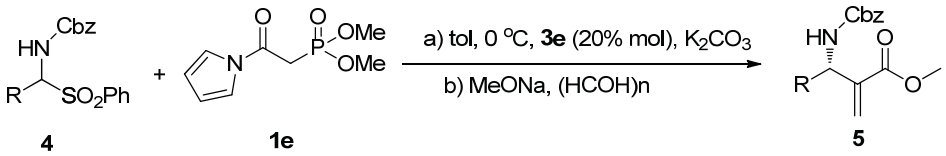
To a stirred solution of dimethyl methylphosphonate (1.06 g, 8.49 mmol) in THF (30 mL) at -78 °C was added BuLi (8.5 mmol, 5.3 mL, 1.6 M in hexane) slowly over 30 min. The mixture was stirred at -65 °C for 90 min, and then carbonyl dipyrrole (1.25 g, 7.76 mmol) in THF (5 mL) was added slowly over 20 min. The mixture was stirred at the same temperature for 1 h, and then was gradually warmed to room temperature over 2 h. The reaction mixture was quenched with sat. aq. NH_4Cl and the aqueous phase was extracted with ethyl acetate. The organic layer was washed with brine,

and dried over Na_2SO_4. After removing the solvent, the residue was purified by silica gel flash column chromatography to give the HWE reagent **3e** in 70% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 (brs, 2H), 6.32-6.30 (m, 2H), 3.82-3.74 (m, 6H), 3.09 (d, J = 22.2 Hz, 2H) ppm; C^{13} NMR (75 MHz, $CDCl_3$) δ 162.3, 119.3, 113.4, 52.9, 33.6 (d, $J_{(C-P)}$ = 133 Hz) ppm; **^{31}P NMR** (121 MHz, $CDCl_3$) δ 21 ppm.

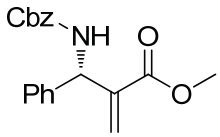


3d ^{1}H NMR (300 MHz, $CDCl_3$) δ, 7.35 (brs, 2H), 6.32 (t, J = 2.4 Hz, 2H), 4.23-4.13 (m, 4H), 3.46 (d, J = 22.2 Hz, 2H) 1.32 (t, J = 6.9 Hz, 6H) ppm; **^{31}P NMR** (121 MHz, $CDCl_3$) δ 18 ppm.

General procedure for the reaction of the HWE reagent with α-amido sulfones

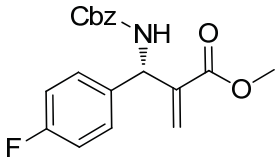


Catalyst **3e** (21.9 mg, 0.05 mmol, 20 mol %), HWE reagent **1e** (81.3 mg, 0.375 mmol) were dissolved in toluene (4 mL) at 0 ^{o}C. Then α-amidosulfones **4** (0.25 mmol) was added followed by addition of an aqueous solution of K_2CO_3 (1.5 M, 0.2 mL). After the stated reaction time, the intermediate product was quickly isolated by column chromatography. Then it was dissolved in THF (2 mL)**,** and a precooled solution of MeONa (2.2 equiv) in MeOH (0.5 mL) was added at **-**10 ^{o}C. After the reaction was stirred 30 min 0 ^{o}C, paraformaldehyde (5 equiv) was added, and the mixture was stirred for another 4 hours. The reaction process was monitored by TLC. Upon completion, the reaction was quenched with sat. aq. NaCl and extracted with ethyl acetate and dried over Na_2SO_4. After concentration of the solvents, the residue was purified on a silica gel column to give the corresponding product

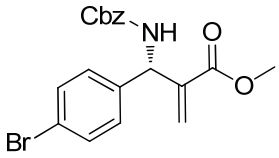


5a Colorless oil; 87% yield; 91% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 85/15, flow rate = 1.0 mL/min, t_{major} = 16.0 min, t_{minor} = 20.3 min); $[\alpha]^{20}_D$ = +16.0 (c = 1.25, $CHCl_3$); **^{1}H NMR** (300 MHz, CDCl3) δ 7.40-7.31 (m, 6H), 7.30 – 7.24 (m, 4H), 6.38 (s, 1H), 5.93 (s, 1H), 5.87 – 5.65 (m, 2H), 5.13 (s, 2H), 3.66 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.9, 155.5, 139.6, 139.5, 136.3, 128.6, 128.5, 128.1, 127.6, 127.0, 126.4, 67.0,

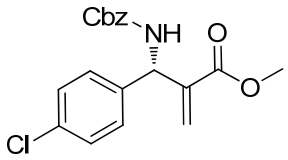
56.7, 51.9 ppm; **IR** (neat): 3334, 2952, 1723, 1500, 1233, 1042, 700 cm^{-1}; **HRMS** (ESI): $C_{19}H_{19}NO_4$ $[M+H]^+$ calcd: 326.1387, found: 326.1376.



5b Colorless oil; 87% yield; 90% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 11.6 min, t_{minor} = 15.2 min); $[\alpha]^{20}_D$ = +1.8 (*c* = 1.14, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.44 – 7.23 (m, 5H), 7.17 (dd, *J* = 8.4, 5.5 Hz, 2H), 6.91 (t, *J* = 8.7 Hz 2H), 6.29 (s, 1H), 5.84 (s, 1H), 5.75 (d, *J* = 8.2 Hz, 1H), 5.65 (d, *J* = 8.9 Hz, 1H), 5.05 (s, 2H), 3.59 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.9, 162.1 (d, J_{C-F} = 244.5 Hz), 155.5, 139.4, 136.2, 135.3 (d, J_{C-F} = 3.1 Hz), 128.5, 128.2, 128.1, 128.0, 127.2, 115.5 (d, J_{C-F} = 21 Hz), 67.1, 56.2, 52.0 ppm; **IR** (neat): 3333, 2953, 1722, 1507, 1225, 1043, 837, 699 cm^{-1}; **HRMS** (ESI): $C_{19}H_{18}FNO_4$ $[M+H]^+$ calcd: 344.1293, found: 344.1282.

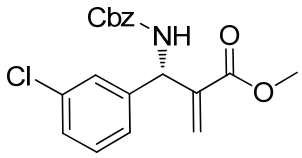


5c Colorless oil; 87% yield; 91% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 14.9 min, t_{minor} = 19.3 min); $[\alpha]^{20}_D$ = +22 (*c* = 1.0, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.53 – 7.42 (d, *J* = 8.5 Hz, 2H), 7.42 – 7.27 (m, 5H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.38 (s, 1H), 5.94 (s, 1H), 5.84 (d, *J* = 8.4 Hz, 1H), 5.69 (d, *J* = 9.2 Hz, 1H), 5.13 (s, 2H), 3.68 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.84, 155.57, 139.1, 138.7, 136.2, 131.7, 128.6, 128.3, 128.2 (overlapped), 129.1, 127.7, 121.6, 67.2, 56.4, 52.1 ppm; **IR** (neat): 3331, 2920, 2851, 1720, 1511, 1261, 1041, 812, 698 cm^{-1}; **HRMS** (ESI): $C_{19}H_{18}BrNO_4$ $[M+H]^+$ calcd: 404.0492, found: 404.0495.

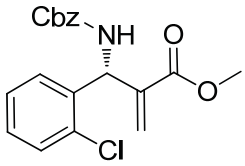


5d Colorless oil; 86% yield; 86% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 15.3 min, t_{minor} = 20.1 min); $[\alpha]^{20}_D$ = -6.14 (*c* = 1.14, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.42 – 7.31 (m, 5H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.21 (d, *J* = 8.6 Hz, 2H), 6.38 (s, 1H), 5.92 (s, 1H), 5.84 (d, *J* =

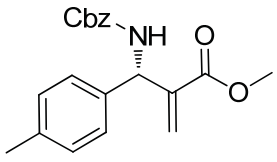
9.2 Hz, 1H), 5.71 (d, J = 9.1 Hz, 1H), 5.13 (s, 2H), 3.67 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.8, 155.5, 139.2, 138.2, 136.2, 133.4, 128.8, 128.6, 128.3, 128.2 (overlapped), 127.8, 127.7, 67.1, 56.3, 52.1 ppm; **IR** (neat): 3333, 2952, 1721, 1494, 1233, 1043, 819, 698 cm^{-1}; **HRMS** (ESI): $C_{19}H_{18}ClNO_4$ $[M+H]^+$ calcd: 360.0997, found: 360.0990.



5e Colorless oil; 80% yield; 92% *ee* determined by HPLC on a Chiracel OD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 8.0 min, t_{minor} = 9.1 min); $[\alpha]^{20}_D$ = -14.6 (c = 0.96, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.38-7.22 (m, 5H), 7.21 – 7.00 (m, 4H), 6.32 (s, 1H), 5.86 (s, 1H), 5.81 (d, J = 9.0 Hz, 1H), 5.64 (d, J = 9.3 Hz, 1H), 5.06 (s, 2H), 3.60 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.8, 155.6, 141.7, 138.9, 136.2, 134.6, 129.9, 128.6, 128.3, 128.2 (overlapped), 128.0, 127.8, 126.5, 124.5, 67.2, 56.4, 52.1 ppm; **IR** (neat): 3331, 2952, 1719, 1506, 1230, 1042, 697 cm^{-1}; **HRMS** (ESI): $C_{19}H_{18}ClNO_{44}$ $[M+Na]^+$ calcd: 382.0817, found: 382.0830.

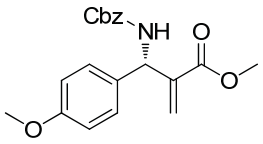


5f Colorless oil; 84% yield; 91% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 11.7 min, t_{minor} = 17.1 min); $[\alpha]^{20}_D$ = +12.5 (c = 1.20, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.42 – 7.29 (m, 6H), 7.29 – 7.14 (m, 3H), 6.40 (s, 1H), 6.15 (d, J = 8.6 Hz, 1H), 5.92 (s, 1H), 5.69 (d, J = 8.3 Hz, 1H), 5.11 (s, 2H), 3.67 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.2, 138.6, 136.9, 136.3, 133.6, 130.1, 129.1, 128.5, 128.2 (multi-shifts overlapped), 127.7, 127.0, 67.1, 53.5, 52.1 ppm; **IR** (neat): 3332, 2952, 1724, 1522, 1237, 1040, 816, 757, 700 cm^{-1}; **HRMS** (ESI): $C_{19}H_{18}ClNO_4$ $[M+Na]^+$ calcd: 382.0817, found: 382.0828.

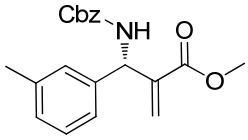


5g Colorless oil; 79% yield; 88% *ee* determined by HPLC on a Chiralpak AD-H column, (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 14.4 min, t_{minor} = 19.0 min); $[\alpha]^{20}_D$ = +13.8 (c = 1.45, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.45 – 7.27 (m, 5H), 7.24 – 7.04 (q, J = 8.2 Hz, 4H), 6.36 (s, 1H), 5.91 (s, 1H), 5.80 – 5.56 (m, 2H), 5.12 (s,

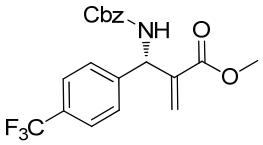
2H), 3.66 (s, 3H), 2.31 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.5, 139.7, 137.3, 136.5, 136.3, 129.3, 128.5, 128.2(overlapped), 126.7, 126.3, 67.0, 56.4, 51.9, 21.0 ppm; **IR** (neat): 3336, 2951, 1721, 1508, 1230, 1042, 815, 699 cm^{-1}; **HRMS** (ESI): $C_{20}H_{21}NO_4$ $[M+H]^+$ calcd: 340.1543, found: 340.1537.



5h Colorless oil; 76% yield; 83% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{rmajor} = 22.7 min, t_{minor} = 28.5 min); $[\alpha]^{20}_D$ = +15.1 (*c* = 1.26, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.46 – 7.27 (m, 5H), 7.19 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.35 (s, 1H), 5.90 (s, 1H), 5.83 – 5.52 (m, 2H), 5.12 (s, 2H), 3.77 (s, 3H), 3.66 (s, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.1, 159.0, 155.5, 139.9, 136.3, 131.6, 128.5, 128.2, 127.7, 126.5, 114.0, 67.0, 56.2, 55.2, 52.0 ppm; **IR** (neat): 3340, 2953, 1722, 1511, 1248, 1037, 826, 699 cm^{-1}; **HRMS** (ESI): $C_{20}H_{21}NO_5$ $[M+Na]^+$ calcd: 378.1312, found: 378.1310.

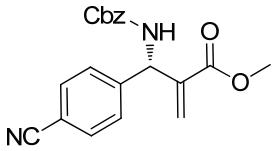


5i Colorless oil; 87% yield; 89% *ee* determined by HPLC on a Chiralpak OJ-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 12.8 min, t_{minor} = 16.6 min); $[\alpha]^{20}_D$ = +11.9 (*c* = 1.18, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.46 – 7.27 (m, 5H), 7.20 (dd, *J* = 14.3, 6.6 Hz, 1H), 7.14 – 6.98 (m, 3H), 6.36 (s, 1H), 5.91 (s, 1H), 5.79 (d, *J* = 8.6 Hz, 1H), 5.71 (d, *J* = 8.9 Hz, 1H), 5.12 (s, 2H), 3.65 (s, 3H), 2.31 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.5, 139.6, 139.3, 138.2, 136.3, 128.5, 128.4 (overlapped), 128.3, 128.1, 127.1, 126.8, 123.4, 66.9, 56.6, 51.9, 21.4 ppm; **IR** (neat): 3335, 2952, 1724, 1502, 1237, 1043, 700 cm^{-1}; **HRMS** (ESI): $C_{20}H_{21}NO_4$ $[M+Na]^+$ calcd: 362.1363, found: 362.1355.

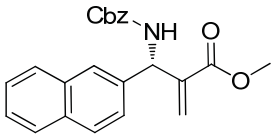


5j Colorless oil; 81% yield; 92% *ee* determined by HPLC on a Chiracel OD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{rmajor} = 10.3 min, t_{mino} = 13.7 min); $[\alpha]^{20}_D$ = -24.7 (*c* = 1.30, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.2 Hz, 2H), 7.49 – 7.27 (m, 7H), 6.42 (s, 1H), 6.20 – 5.86 (m, 2H), 5.80 (d, *J* = 9.2 Hz, 1H), 5.15 (s, 2H), 3.68 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.8, 155.7, 143.7, 138.9, 136.1, 130.0 (q, J_{C-F} = 32.3 Hz), 128.6,

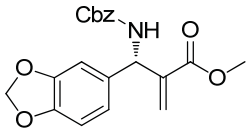
128.3, 128.2, 126.7, 124.0 (q, $J_{C\text{-}F}$ = 270.0 Hz), 125.6 (q, $J_{C\text{-}F}$ =4.1 Hz), 67.2, 56.7, 52.3 ppm; **IR** (neat): 3331, 2954, 1729, 1503, 1327, 1067, 843, 737, 699, 616 cm^{-1}; **HRMS** (ESI): $C_{20}H_{18}F_3NO_4$ $[M+Na]^+$ calcd: 416.1080, found: 416.1072.



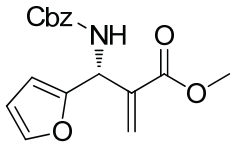
5k Colorless oil; 88% yield; 93% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 26.4 min, t_{minor} = 35.7 min); $[\alpha]^{20}_D$ = -37.0 (*c* = 1.0, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.3 Hz, 2H), 7.53 – 7.27 (m, 7H), 6.42 (s, 1H), 6.01 – 5.95 (m, 2H), 5.77 (d, *J* = 9.4 Hz, 1H), 5.14 (s, 2H), 3.68 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.6, 155.7, 145.1, 138.5, 136.0, 132.4, 128.8, 128.6, 128.3, 128.3, 127.0, 118.6, 111.5, 67.3, 56.8, 52.2 ppm; **IR** (neat): 3341, 2920, 2229, 1721, 1506, 1261, 1043, 818, 699 cm^{-1}; **HRMS** (ESI): $C_{20}H_{18}N_2O_4$ $[M+H]^+$ calcd: 351.1339, found: 351.1334.



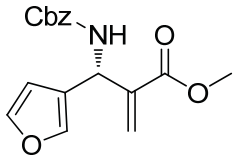
5l Colorless oil; 80% yield; 87% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 14.6 min, t_{minor} = 18.2 min); $[\alpha]^{20}_D$ = +11.8 (*c* = 1.36, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.89 – 7.76 (m, 3H), 7.73 (s, 1H), 7.53 – 7.45 (m, 2H), 7.45 – 7.27 (m, 6H), 6.44 (s, 1H), 6.00 (s, 1H), 5.97 – 5.80 (m, 2H), 5.16 (s, 2H), 3.66 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.6, 139.6, 136.9, 136.3, 133.2, 132.8, 128.6, 128.5, 128.2, 128.0, 127.6, 127.3, 126.3, 126.1, 125.1, 124.7, 67.1, 56.8, 52.0 ppm; **IR** (neat): 3333, 2953, 1721, 1504, 1235, 1043, 818, 747, 699, 478 cm^{-1}; **HRMS** (ESI): $C_{23}H_{21}NO_4$ $[M+Na]^+$ calcd: 398.1363, found: 398.1355.



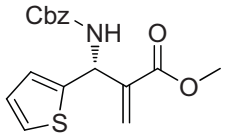
5m Colorless oil; 78% yield; 96% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 22.0 min, t_{minor} = 24.1 min); $[\alpha]^{20}_D$ = +10.8 (*c* = 1.20, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41 – 7.29 (m, 5H), 6.77 (s, 1H), 6.76 – 6.70 (m, 2H), 6.36 (s, 1H), 5.93 (s, 2H), 5.90 (s, 1H), 5.73 (d, *J* = 8.7 Hz, 1H), 5.65 (d, *J* = 8.8 Hz, 1H), 5.12 (s, 2H), 3.68 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.4, 147.9, 147.0, 139.6, 136.2, 133.4, 128.5, 128.2, 126.8, 119.7, 108.3, 107.1, 101.1, 67.0, 56.4, 52.0 ppm; **IR** (neat): 3358, 2955, 1716, 1491, 1227, 1039, 816, 699, 531 cm^{-1}; **HRMS** (ESI): $C_{20}H_{19}NO_6$ $[M+Na]^+$ calcd: 392.1115, found: 392.1115.



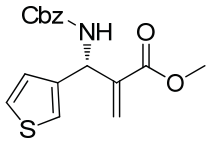
5n Colorless oil; 72% yield; 84% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 9.7 min, t_{minor} = 10.6 min); $[\alpha]^{20}_{D}$ = +3.6 (*c* = 1.38, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 6H), 6.40 (s, 1H), 6.30 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.18 (d, *J* = 3.2 Hz, 1H), 5.92 (s, 1H), 5.82 (s, 2H), 5.13 (s, 2H), 3.73 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.8, 155.4, 152.2, 142.2, 137.7, 136.2, 128.6 (overlapped), 128.5, 128.2, 127.7, 110.5, 110.3, 106.8, 67.1, 52.1, 51.3 ppm; **IR** (neat): 3340, 2953, 1723, 1505, 1234, 1043, 820, 742, 699, 599 cm^{-1}; **HRMS** (ESI): $C_{17}H_{17}NO_5$ $[M+Na]^+$ calcd: 338.0999, found: 338.1002.



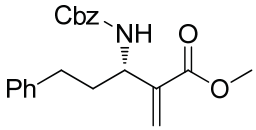
5o Colorless oil; 71% yield; 81% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 11.1 min, t_{minor} = 12.5 min); $[\alpha]^{20}_{D}$ = +4.7 (*c* = 0.85, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.34 – 7.50 (m, 6H), 7.27 (s, 1H), 6.30 (s, 1H), 6.29 (s, 1H), 5.88 (s, 1H), 5.82 (d, *J* = 8.6 Hz, 1H), 5.65 (d, *J* = 9.1 Hz, 1H), 5.12 (s, 2H), 3.71 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.4, 143.4, 139.5, 139.2, 136.2, 128.5, 128.1, 126.7, 125.2, 109.3, 66.9, 52.0, 49.8 ppm. **IR** (neat): 3340, 2953, 1724, 1504, 1235, 1028, 734 cm^{-1}; **HRMS** (ESI): $C_{17}H_{17}NO_5$ $[M+H]^+$ calcd: 316.1179, found: 316.1185.



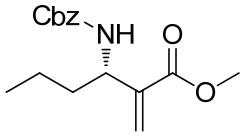
5p Colorless oil; 80% yield; 94% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 11.4 min, t_{minor} = 14.2 min); $[\alpha]^{20}_{D}$ = +1.8 (*c* = 1.14, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.42 – 7.25 (m, 5H), 7.19 (d, *J* = 4.2 Hz, 1H), 6.93 (dd, *J* = 5.0, 3.6 Hz, 1H), 6.88 (d, *J* = 3.2 Hz, 1H), 6.36 (s, 1H), 6.14 – 5.84 (m, 3H), 5.14 (s, 2H), 3.72 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.8, 155.3, 143.8, 139.1, 136.1, 128.5, 128.2, 128.1, 127.3, 127.0, 125.0, 124.6, 67.1, 53.2, 52.1 ppm; **IR** (neat): 3336, 2952, 1721, 1503, 1223, 1040, 755 cm^{-1} **HRMS** (ESI): $C_{17}H_{17}NO_4S$ $[M+NH_4]^+$ calcd: 349.1217, found: 349.1212.



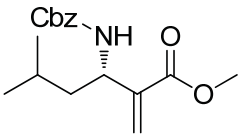
5q Colorless oil; 85% yield; 86% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 12.7 min, t_{minor} = 17.5 min); $[\alpha]^{20}_{D}$ = +6.3 (*c* = 1.05, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.43 – 7.29 (m, 5H), 7.25 (dd, *J* = 3.3, 0.9 Hz, 1H), 7.07 (d, *J* = 3.0 Hz, 1H), 6.97 (dd, *J* = 5.1, 0.9 Hz, 1H), 6.34 (s, 1H), 5.95 – 5.85 (m, 2H), 5.78 (d, *J* = 9.0 Hz, 1H), 5.13 (s, 2H), 3.69 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 155.4, 141.0, 139.5, 136.2, 128.5, 128.2, 128.1(overlapped), 126.9, 126.3, 125.5, 121.4, 67.0, 53.3, 52.0 ppm; **IR** (neat): 3344, 2953, 1721, 1503, 1225, 1043, 736 cm^{-1}; **HRMS** (ESI): $C_{17}H_{17}NO_4S$ $[M+H]^+$ calcd: 332.0951, found: 332.0947.



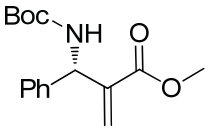
5r Colorless oil; 65% yield; 63% *ee* determined by HPLC on a Chiracel OJ-H column (hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, t_{major} = 19.7 min, t_{minor} = 23.0 min); $[\alpha]^{20}_{D}$ = -2 (*c* = 1.0, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 4H), 7.24 – 7.13 (m, 3H), 7.13 – 7.01 (m, 3H), 6.14 (s, 1H), 5.67 (s, 1H), 5.50 (d, *J* = 9.5 Hz, 1H), 5.08 – 4.92 (m, 2H), 4.43 (q, *J* = 7.5 Hz, 1H), 3.66 (s, 3H), 2.65 – 2.44 (m, 2H), 2.00 – 1.82 (m, 2H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.2, 155.6, 141.0, 139.5, 136.3, 128.5, 128.3, 128.3, 128.1, 127.1, 125.9, 66.7, 54.1, 51.9, 36.0, 32.6 ppm; **IR** (neat): 3336, 3029, 2951, 1719, 1522, 1451, 1240, 1045, 699 cm^{-1}; **HRMS** (ESI): $C_{21}H_{23}NO_4$ $[M+Na]^+$ calcd: 376.1519, found: 376.1526.



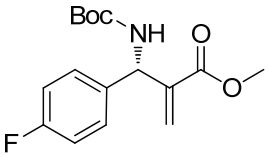
5s Colorless oil; 69% yield; 70% *ee* determined by HPLC on a Chiralpak OD-H column (hexane/2-propanol = 95/5, flow rate = 0.75 mL/min, t_{major} = 11.8 min, t_{minor} = 12.6 min); $[\alpha]^{20}_{D}$ = +2.1 (*c* = 0.96, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.45 – 7.28 (m, 5H), 6.20 (s, 1H), 5.74 (s, 1H), 5.50 (d, *J* = 9.2 Hz, 1H), 5.19 – 4.98 (m, 2H), 4.46 (q, *J* = 7.5 Hz, 1H), 3.75 (s, 3H), 1.64 (q, *J* = 7.5 Hz, 2H), 1.44 – 1.19 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.3, 155.6, 139.8, 136.4, 128.4, 128.0, 128.04 (overlapped), 126.6, 66.6, 54.0, 51.8, 36.5, 19.5 13.6 ppm; **IR** (neat): 3339, 2958, 1729, 1506, 1250, 740, 689 cm^{-1}; **HRMS** (ESI): $C_{16}H_{21}NO_4$ $[M+Na]^+$ calcd: 314.1363, found: 314.1354.



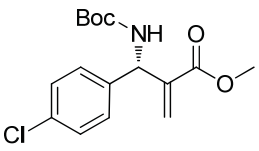
5t Colorless oil; 57% yield; 57% *ee* determined by HPLC on a Chiralpak AS column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{minor} = 5.3 min, t_{major} = 9.3 min); $[\alpha]^{20}_{D}$ = +2.1(*c* = 0.96, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.44 – 7.25 (m, 5H), 6.19 (s, 1H), 5.76 (s, 1H), 5.44 (d, *J* = 9.3 Hz, 1H), 5.20 – 4.96 (m, 2H), 4.54 (q, *J* = 8.1 Hz, 1H), 3.75 (s, 3H), 1.69 – 1.38 (m, 3H), 0.93 (d, *J* = 6.3 Hz, 3H), 0.91 (d, *J* = 6.3 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.3, 155.6, 140.2, 136.5, 128.4, 128.1, 126.4, 66.6, 52.5, 51.8, 43.6, 25.0, 22.5, 22.2 ppm; **IR** (neat): 3339, 2956, 1720, 1524, 1229, 1046, 698 cm^{-1}; **MS** (ESI): $C_{17}H_{23}NO_4$ $[M+H]^+$ calcd: 306.2, found: 306.3.



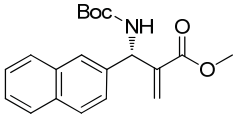
5aa White solid, mp: 74 - 77 °C; 86% yield; 86% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 95/5, flow rate = 0.75 mL/min, t_{major} = 17.0 min, t_{minor} = 20.8 min). $[\alpha]^{20}_{D}$ = +17.5 (*c* = 0.8, $CHCl_3$) for 78% *ee*. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 6.38 (s, 1H), 5.92 (s, 1H), 5.69 (d, *J* = 8.7 Hz, 1H), 5.50 (d, *J* = 8.0 Hz, 1H), 3.67 (s, 3H), 1.45 (s, 9H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.1, 154.9, 140.0, 139.8, 128.5, 127.5, 126.5, 79.8, 56.1, 51.9, 28.3 ppm. **HRMS** (ESI): $C_{16}H_{21}NO_4$ $[M+Na]^+$ calcd: 314.1363, found: 314.1354.



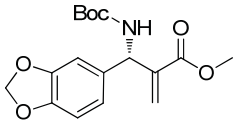
5ab Colorless oil; 89% yield; 86% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 95/5, flow rate = 0.75 mL/min, t_{major} = 13.7 min, t_{minor} = 16.3 min); $[\alpha]^{20}_{D}$ = +3.5 (*c* = 0.85, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.31 – 7.18 (m, 2H), 7.07 – 6.92 (t, *J* = 7.5 Hz, 2H), 6.37 (s, 1H), 5.92 (s, 1H), 5.66 (d, *J* = 8.4 Hz, 1H), 5.52 (brs, 1H), 3.68 (s, 3H), 1.45 (s, 9H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.0, 162.1 (d, $J_{C\text{-}F}$ = 244.5 Hz), 154.9, 139.8, 135.7 (d, $J_{C\text{-}F}$ = 3.2 Hz), 128.2 (d, $J_{C\text{-}F}$ = 8.2 Hz), 126.8, 115.4 (d, $J_{C\text{-}F}$ = 21.0 Hz), 80.0, 55.6, 51.9, 28.3 ppm; **IR** (neat): 3360, 2978, 1721, 1506, 1228, 1164, 838 cm^{-1}; **HRMS** (ESI): $C_{16}H_{20}FNO_4$ $[M+Na]^+$ calcd: 332.1269, found: 332.1260.



5ad Colorless oil; 84% yield; 89% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, t_{major} = 16.3 min, t_{minor} = 20.2 min); $[\alpha]^{20}_{D}$ = +2.1 (*c* = 0.96, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.7 Hz, 2H), 7.22 (d, *J* = 8.7 Hz, 2H), 6.37 (s, 1H), 5.92 (s, 1H), 5.65 (d, *J* = 8.5 Hz, 1H), 5.55 (brs, 1H), 3.68 (s, 3H), 1.45 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.9, 154.9, 139.5, 138.5, 133.2, 128.7,127.8, 127.1, 80.0, 55.7, 52.0, 28.3 ppm; **IR** (neat): 3363, 2977, 1717, 1493, 1166, 757 cm^{-1}; **HRMS** (ESI): $C_{16}H_{20}ClNO_4$ $[M+Na]^+$ calcd: 348.0973, found: 348.0983.

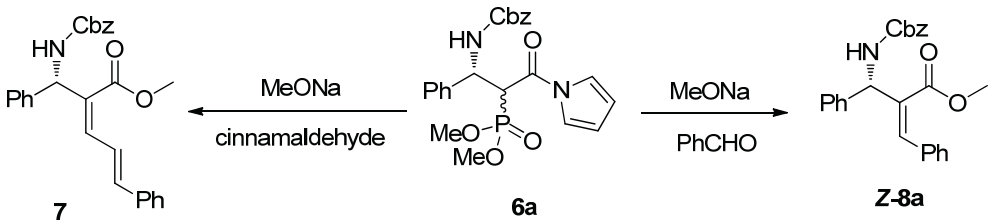


5al Colorless oil; 72% yield; 83% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, t_{major} = 18.1 min, t_{minor} = 23.3 min); $[\alpha]^{20}_{D}$ = +5.7 (*c* = 1.05, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.97 – 7.74 (m, 3H), 7.72 (s, 1H), 7.55 – 7.30 (m, 3H), 6.43 (s, 1H), 5.98 (s, 1H), 5.85 (d, *J* = 8.8 Hz, 1H), 5.58 (d, *J* = 6.7 Hz, 1H), 3.67 (s, 3H), 1.47 (s, 9H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.1, 154.9, 139.9, 137.3, 133.2, 132.7, 128.4, 128.0, 127.6, 126.7, 126.2, 126.0, 125.2, 124.9, 79.9, 56.20, 51.9, 28.4 ppm; **IR** (neat): 3362, 2977, 1717, 1494, 1165, 815 cm^{-1}; **HRMS** (ESI): $C_{20}H_{23}NO_4$ $[M+Na]^+$ calcd: 364.1519, found: 364.1530.



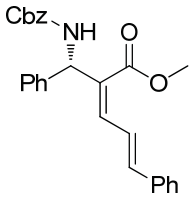
5am Colorless oil; 79% yield; 87% *ee* determined by HPLC on a Chiralpak AD-H column (hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, t_{major} = 12.4min, t_{minor} = 17.5min); $[\alpha]^{20}_{D}$ = +11.1(*c* = 1.08, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 6.78 (s, 1H), 6.76 - 6.65 (m, 2H), 6.35 (s, 1H), 5.93 (s, 2H), 5.89 (s, 1H), 5.59 (d, *J* = 7.9 Hz, 1H), 5.44 (brs, 1H), 3.69 (s, 3H), 1.45 (s, 9H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.1, 154.8, 147.8, 146.9, 140.0, 133.8, 126.3, 119.8, 108.2, 107.3, 101.1, 79.8, 55.8, 51.9, 28.3 ppm; **IR** (neat): 3381, 2978, 1719, 1491, 1243, 1166, 1041, 735 cm^{-1}; **HRMS** (ESI): $C_{17}H_{21}NO_6$ $[M+Na]^+$ calcd: 358.1261, found: 358.1264.

Transformations of the Mannich products

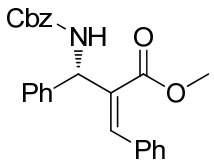


The intermediate product **6** was prepared according the above mentioned general procedure. Then phosphonate **6** (104.5 mg, 0.23 mmol) was dissolved in THF (2 mL), and a precooled solution of MeONa (2.2 equiv, 78 mg, 0.506 mmol) in

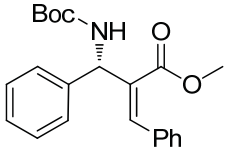
MeOH (2 mL) was added at -10 °C. After the reaction was stirred 30 min 0 °C, the corresponding aldehyde (1.5 equiv) was added. And the reaction was stirred at the same temperature for another 20 hours. The reaction process was monitored by TLC. Upon completion, the reaction was quenched with sat. aq. NaCl and extracted with EA and dried over Na_2SO_4. After concentration of the solvents, the residue was purified on a silica gel column to give the corresponding product.



7 Following the above procedure, the product (72.1 mg, 73% yield), and the corresponding value of *Z*: *E* was 4.5 : 1. White solid, mp: 102-104 °C; $[\alpha]^{20}_D$ = -12.8 (*c* = 1.09, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 15.6, 11.1 Hz, 1H), 7.49 (d, *J* = 6.9 Hz, 2H), 7.50 – 7.26 (m, 13H), 6.91 (d, *J* = 11.4, 1H), 6.85 (d, *J* = 15.7, 1H), 5.89 (d, *J* = 9.0 Hz, 1H), 5.77 (d, *J* = 9.3 Hz, 1H), 5.14 (s, 2H), 3.66 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.5, 155.6, 142.3, 141.6, 140.2, 136.3, 128.9, 128.7, 128.5, 128.2, 128.15 (overlapped), 127.4, 127.3 (overlapped), 126.1, 124.9, 67.0, 58.5, 51.5 ppm; **IR** (neat): 3335, 3004, 2952, 1710, 1497, 1228, 1152, 1032, 752, 697 cm^{-1}; **HRMS** (ESI): $C_{27}H_{25}NO_4$ $[M+Na]^+$ calcd: 450.1676, found: 450.1675.

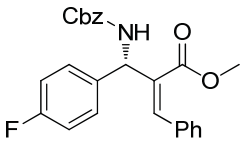


***Z*-8a** Colorless oil; 81% yield, *Z* : *E* = 3.5 : 1; $[\alpha]^{20}_D$ = 2.8 (*c* = 1.44, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.35 – 7.21 (m, 15H), 6.97 (s, 1H), 5.97 (d, *J* = 9.5 Hz, 1H), 5.80 (d, *J* = 9.0 Hz, 1H), 5.13 (s, 2H), 3.45 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.3, 155.5, 139.0, 136.8, 136.2, 134.9, 132.8, 128.6, 128.4, 128.1, 127.7, 126.5, 67.0 58.8, 51.6 ppm; **IR** (neat): 3327, 3031, 2951, 1722, 1499, 1229, 1036, 750, 699 cm^{-1}; **MS** (ESI): $C_{25}H_{23}NO_4$ $[M+NH_4]^+$ calcd: 419.2, found: 419.1.

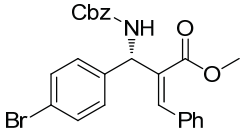


***Z*-8aa** Colorless oil; 76% yield, *Z* : *E* = 6.6 : 1; $[\alpha]^{20}_D$ = –53.6 (*c* = 0.97, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.45 – 7.25 (m, 10H), 6.95 (s, 1H), 5.74 (d, *J* = 7.8 Hz, 1H), 5.65 (brs, 1H), 3.50 (s, 3H), 1.46 (s, 9H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ

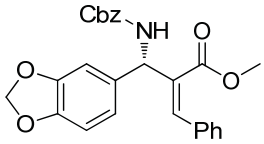
168.5, 154.9, 139.4, 136.2, 135.1, 133.4, 128.6, 128.4, 128.3, 128.1, 127.6, 126.6, 79.8, 58.2, 51.6, 28.3 ppm; **IR** (neat): 3432, 2976, 1714, 1492, 1166, 752, 699 cm^{-1}; **HRMS** (ESI): $C_{22}H_{25}NO_4$ $[M+Na]^+$ calcd: 390.1676, found: 390.1686.



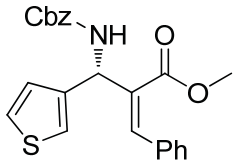
***Z*-8b** Colorless oil; 75% yield，Z : E = 3.8 : 1; $[\alpha]^{20}_D$ = –57.8 (c = 0.97, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.48 – 7.29 (m, 12H), 7.09 – 6.92 (m, 3H), 5.97 (d, J = 8.6 Hz, 1H), 5.77 (d, J = 9.0 Hz, 1H), 5.14 (s, 2H), 3.48 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 162.2 (d, $J_{C\text{-}F}$ = 244.5 Hz), 155.5, 137.1, 136.2, 134.9 (d, $J_{C\text{-}F}$ = 3.0 Hz), 134.8, 132.5, 128.6 (overlapped), 128.5, 128.2 (m), 115.5 (d, $J_{C\text{-}F}$ = 21.8 Hz), 67.1, 58.4, 51.7 ppm; **IR** (neat): 3333, 2952, 1719, 1506, 1225, 1038, 752, 698 cm^{-1}; **HRMS** (ESI): $C_{25}H_{22}FNO_4$ $[M+Na]^+$ calcd: 442.1425, found: 442.1422.



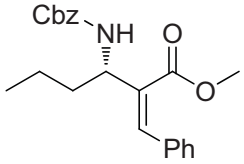
***Z*-8c** Colorless oil; 90% yield，Z : E = 2.9 : 1; $[\alpha]^{20}_D$ = –52.0 (c = 0.98, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.43 (d, J = 8.5 Hz, 2H), 7.39 – 7.27 (m, 8H), 7.28 – 7.17 (m, 4H), 6.99 (s, 1H), 6.04 (d, J = 8.9 Hz, 1H), 5.74 (d, J = 9.1 Hz, 1H), 5.13 (s, 2H), 3.47 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.2, 155.5, 138.2, 137.6, 136.1, 134.7, 132.1, 131.7, 128.6, 128.5, 128.2 (overlapped), 128.1, , 121.6, 67.1, 58.5, 51.7 ppm; **IR** (neat): 3327, 2955, 1709, 1494, 1226, 1036, 697 cm^{-1}; **HRMS** (ESI): $C_{25}H_{22}BrNO_4$ $[M+Na]^+$ calcd: 502.0624, found: 502.0611.



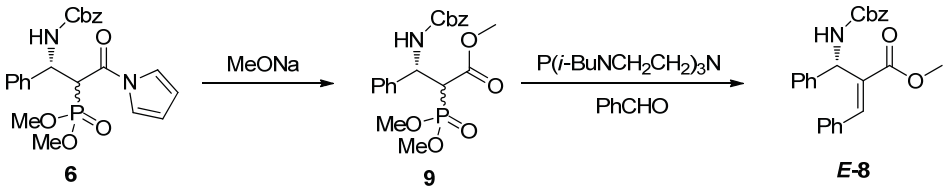
***Z*-8m** Colorless oil; 72% yield，Z : E = 3.2 : 1; $[\alpha]^{20}_D$ = –23.4 (c = 0.93, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.55 – 7.13 (m, 10H), 6.96 (s, 1H), 6.68 – 7.13 (m, 3H), 5.94 (s, 2H), 5.87 (d, J = 7.2 Hz, 1H), 5.70 (d, J = 7.9 Hz, 1H), 5.14 (s, 2H), 3.51 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.4, 155.5, 147.9, 147.1, 136.6, 136.2, 134.9, 133.0, 132.8, 128.5[1], 128.5 (overlapped), 128.2, 119.90, 108.3, 107.2, 101.1, 67.1, 58.6, 51.7 ppm; **IR** (neat): 3333, 2951, 1719, 1495, 1235, 1037, 697 cm^{-1}; **HRMS** (ESI): $C_{26}H_{23}NO_6$ $[M+Na]^+$ calcd: 468.1418, found: 468.1408.



***Z*-8q** Colorless oil; 93% yield，$Z:E = 4.8:1$; $[\alpha]^{20}_D = -34.8$ ($c = 0.89$, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.48 – 7.20 (m, 11H), 7.18 (s, 1H), 7.02 (d, $J = 4.7$ Hz, 2H), 5.94 (d, $J = 8.7$ Hz, 1H), 5.81 (d, $J = 9.0$ Hz, 1H), 5.15 (s, 2H), 3.52 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.4, 155.5, 140.6, 136.9, 136.2, 134.9, 132.4, 128.5, 128.2, 126.44 (overlapped), 126.4, 121.7, 67.0, 55.7, 51.7 ppm; **IR** (neat): 3332, 2951, 1717, 1501, 1226, 1037, 697 cm^{-1}; **HRMS** (ESI): $C_{23}H_{21}NO_4S$ $[M+Na]^+$ calcd: 430.1083, found: 430.1072.



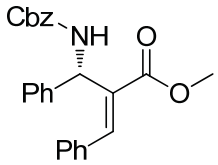
***Z*-8s** Colorless oil; 91% yield，$Z:E = 2.0:1$; $[\alpha]^{20}_D = -13.7$ ($c = 1.02$, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41 – 7.15 (m, 10H), 6.88 (s, 1H), 5.50 (d, $J = 9.1$ Hz, 1H), 5.24 – 4.90 (m, 2H), 4.49 (q, $J = 7.8$ Hz, 1H), 3.60 (s, 3H), 1.65 (q, $J = 7.5$ Hz, 2H), 1.39 – 1.60 (m, 2H), 0.93 (t, $J = 7.3$ Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.9, 155.6, 136.4, 135.5, 135.2, 133.4, 128.4, 128.2, 128.1, 128.0, 127.9, 66.6, 55.9, 51.5, 36.2, 19.3, 13.6 ppm; **IR** (neat): 3334, 2957, 1723, 1504, 1219, 750, 697 cm^{-1}; **MS** (ESI): $C_{22}H_{25}NO_4$ $[M+H]^+$ calcd: 368.2, found: 368.0.



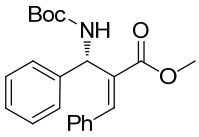
To the intermediate product **6a** (104.5 mg, 0.23 mmol) in THF (2 mL) was added a precooled solution of MeONa (1.1 equiv, 39 mg, 0.25 mmol) in MeOH (2 mL) at -10 °C. And the reaction was allowed to gradually warm to 0 °C. After 12 hours at this temperature, the reaction was then quenched with sat. aq. NaCl and extracted with EtOAc. The combined extract was dried over Na_2SO_4 and concentrated. The residue was purified on a silica gel column to give the methyl ester **9a** (96.8 mg, quantitative). The diastereomeric ratio was determined to be 51:49 by ^{31}P NMR spectroscopy analyses of the crude mixture. ^{1}H NMR [signals of both diastereoisomers] (300 MHz, $CDCl_3$) δ 7.50 – 7.05 (m, 10H), 6.73 (d, $J = 9.2$ Hz, 0.47H), 6.45 (brs, 0.42H), 5.57 – 5.25 (m, 1H), 5.23 – 4.92 (m, 2H), 3.84 – 3.34 (m, 10H). ^{13}C NMR [signals of both diastereoisomers] (75 MHz, $CDCl_3$) δ 168.30, 167.11 (d, $J = 4.3$ Hz), 155.4, 155.3, 139.84 (d, $J = 12.3$ Hz), 136.4 (d, $J = 5.0$ Hz), 128.6, 128.5 128.4, 128.3, 128.1, 128.0, 127.9[5], 127.9, 127.7, 126.6, 126.0, 66.8, 66.7, 53.7 (d, $J = 7.0$ Hz), 53.6 (d, $J = 6.0$ Hz), 53.4 (d, $J = 6.8$ Hz), 53.1 (d, $J = 6.3$ Hz), 52.7 (d, $J = 8.8$ Hz), 52.4 (d, $J = 4.5$ Hz), 51.1(d, $J = 130.3$ Hz), 50.3 (d, $J = 130.3$ Hz) ^{31}P NMR (121 MHz, $CDCl_3$) δ 22.0 (s, 0.49P), 20.1 (s, 0.51P). **HRMS** (ESI): $C_{20}H_{24}NO_7P$ $[M+NH_4]^+$ calcd:

444.1183, found: 444.1193.

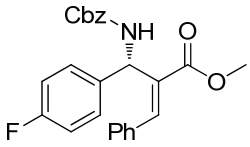
Then methyl ester **9a** was dissolved in THF and P[N(*i*-Bu)CH_2CH_2]$_3$N was added (1.5 equiv, 118 mg, 0.345 mmol) under an argon atmosphere at rt. Then benzaldehyde (1.5 equiv, 33 *u*L, 0.34 mmol) was added, and the reaction was stirred for 24 hours at the same temperature. Then the mixture was purified by silica gel column to give the product.



***E*-8a** Colorless oil; 64% yield，*E* : *Z* = 4.5 : 1; $[\alpha]^{20}_D$ = -39 (*c* = 1.05, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.49 (d, *J* = 6.9 Hz, 2H), 7.38 – 7.21 (m, 13H), 6.40 (m, 2H), 5.21 – 5.09 (m, 2H), 3.71 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.2, 155.9, 142.2, 140.3, 136.4, 134.1, 130.6, 129.3, 129.1, 128.8, 128.5, 128.4, 128.1, 128.1 (overlapped), 128.07, 125.6, 66.9, 52.0, 51.4 ppm; **IR** (neat): 3327, 3061, 2951, 1723, 1498, 1230, 699 cm^{-1}; **HRMS** (ESI): $C_{25}H_{23}NO_4$ $[M+NH_4]^+$ calcd: 419.1965, found: 419.1967.

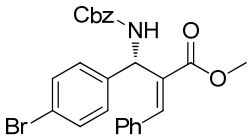


***E*-8aa** Colorless oil; 72% yield, *E* : *Z* = 3.2 : 1; $[\alpha]^{20}_D$ = –67.1 (*c* = 0.97, $CHCl_3$); **^{1}H NMR** [some signals show multiple resonances for the presence of two rotamers] (300 MHz, $CDCl_3$) δ7.95 (s, 1H), 7.55 – 7.14 (m, 10H), 6.32 (d, *J* = 10.2 Hz, 0.8H), 6.23 – 6.09 (m, 1H), 5.75 (d, *J* = 10.2 Hz, 0.2H), 3.73 (s, 2.3H), 3.69 (s, 0.7H), 1.48 (s, 7.1H), 1.22 (s, 2.2H) ppm; ^{13}C NMR [some signals show multiple resonances for the presence of two rotamers] (75 MHz, $CDCl_3$) δ 167.4, 155.3, 141.8, 140.9, 134.1, 130.8, 129.3, 129.2 (overlapped), 128.8, 128.4, 126.9, 125.7/125.5, 79.5, 52.0, 50.7, 28.4/28.0 ppm; **IR** (neat): 3440, 2975, 1711, 1491, 1166, 698 cm^{-1}; **HRMS** (ESI): $C_{22}H_{25}NO_4$ $[M+Na]^+$ calcd: 390.1676, found: 390.1679.

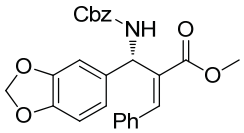


***E*-8b** Colorless oil; 73% yield，*E* : *Z* = 5.7 : 1; $[\alpha]^{20}_D$ = –28.7 (*c* = 1.08, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.51 – 7.29 (m, 9H), 7.26 – 7.15 (m, 3H), 6.96 (t, *J* = 8.6 Hz, 2H), 6.42 (d, *J* = 9.6 Hz, 1H), 6.34 (d, *J* = 9.6 Hz, 1H), 5.26 – 4.96 (m, 2H), 3.72 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.2, 161.9 ($J_{C\text{-}F}$ = 243.8 Hz), 155.8, 142.3, 136.3, 136.1 ($J_{C\text{-}F}$ = 3.0 Hz), 133.9, 130.4, 129.4, 129.1, 129.0, 128.5, 128.2, 128.1, 127.34 (d, $J_{C\text{-}F}$ = 8.1 Hz), 115.3 (d, $J_{C\text{-}F}$ = 28.1 Hz), 67.0, 52.1,

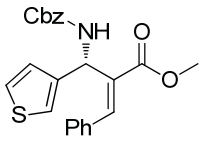
50.9 ppm; **IR** (neat): 3430, 2953, 1715, 1502, 1245, 1038, 761, 698 cm^{-1}; **HRMS** (ESI): $C_{25}H_{22}FNO_4$ $[M+Na]^+$ calcd: 442.1425, found: 442.1420.



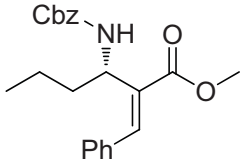
***E*-8c** Colorless oil; 74% yield，E : Z = 10.4 : 1; $[\alpha]^{20}_D$ = –61.1 (c = 0.95, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.53 – 7.26 (m, 12H), 7.12 (d, J = 9.4 Hz, 2H), 6.39 (d, J = 10.1 Hz, 1H), 6.31 (d, J = 10.0 Hz, 1H), 5.22 – 4.97 (m, 2H), 3.73 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 155.9, 142.6, 139.5, 136.3, 133.9, 131.5, 130.1, 129.5, 129.1, 128.9, 128.5, 128.2, 128.1, 127.5, 121.1, 67.0, 52.2, 51.0 ppm; **IR** (neat): 3326, 2951, 1717, 1492, 1245, 1037, 697 cm^{-1}; **HRMS** (ESI): $C_{25}H_{22}BrNO_4$ $[M+Na]^+$ calcd: 502.0624, found: 502.0614.



***E*-8m** Colorless oil; 83% yield，E : Z = 6.0 : 1; $[\alpha]^{20}_D$ = –40.8 (c = 0.98, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.94 (S, 1H), 7.53 – 7.42 (m, 2H), 7.42 – 7.19 (m, 8H), 6.82 – 6.64 (m, 3H), 6.39 (d, J = 10.0 Hz, 1H), 6.27 (d, J = 10.0 Hz, 1H), 5.91 (s, 2H), 5.23 – 4.94 (m, 2H), 3.74 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.3, 155.8 147.9, 146.6, 142.1, 136.3, 134.3, 133.9, 130.5, 129.3, 129.1, 128.8, 128.5, 128.12, 128.07, 118.8, 108.1, 106.5, 101.0, 66.9, 52.1, 51.2 ppm; **IR** (neat): 3426, 2955, 1715, 1494, 1244, 1037, 758, 698 cm^{-1}; **HRMS** (ESI): $C_{26}H_{23}NO_6$ $[M+Na]^+$ calcd: 468.1418, found: 468.1422.

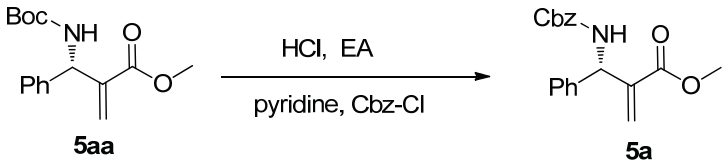


***E*-8q** Colorless oil; 74% yield，E : Z = 8.8 : 1; $[\alpha]^{20}_D$ = –44.1 (c = 1.02, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.50 – 7.27 (m, 9H), 7.24 (dd, J = 5.2, 2.8 Hz, 2H), 7.11 – 6.99 (m, 1H), 6.93 (d, J = 6.0 Hz, 1H), 6.46 (d, J = 10.0 Hz, 1H), 6.32 (d, J = 9.9 Hz, 1H), 5.23 – 4.96 (m, 2H), 3.75 (s, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.2, 155.7, 142.0, 141.8, 136.4, 133.9, 130.5, 129.3, 129.1, 128.8, 128.5, 128.1, 128.0, 126.2, 126.1, 120.7, 66.8, 52.1, 48.8 ppm; **IR** (neat): 3425, 2951, 1717, 1498, 1221, 1037, 696 cm^{-1}; **HRMS** (ESI): $C_{23}H_{21}NO_4S$ $[M+Na]^+$ calcd: 430.1083, found: 430.1071.



***E*-8s** Colorless oil; 77% yield，*E* : *Z* = 4.7 : 1; $[\alpha]^{20}_D$ = +59.8 (*c* = 1.02, $CHCl_3$); **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.56 – 7.04 (m, 10H), 5.91 (d, *J* = 10.0 Hz, 1H), 5.20 – 4.99 (m, 3H), 3.80 (s, 3H), 1.88 – 1.70 (m, 1H), 1.68 – 1.48 (m, 1H), 1.35 – 1.08 (m, 2H), 0.78 (t, *J* = 7.3 Hz, 3H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.4, 155.7, 141.0, 136.5, 134.4, 131.8, 129.0, 128.7, 128.6, 128.4, 127.9, 127.8, 66.5, 51.9, 48.6, 37.2, 19.4, 13.6 ppm; **IR** (neat): 3435, 2957, 1719, 1501, 1250, 1082, 776, 699 cm^{-1}; **MS** (ESI): $C_{22}H_{25}NO_4$ $[M+H]^+$ calcd: 368.2, found: 368.1.

Determination of the absolute configuration of compound 5aa and 5a.

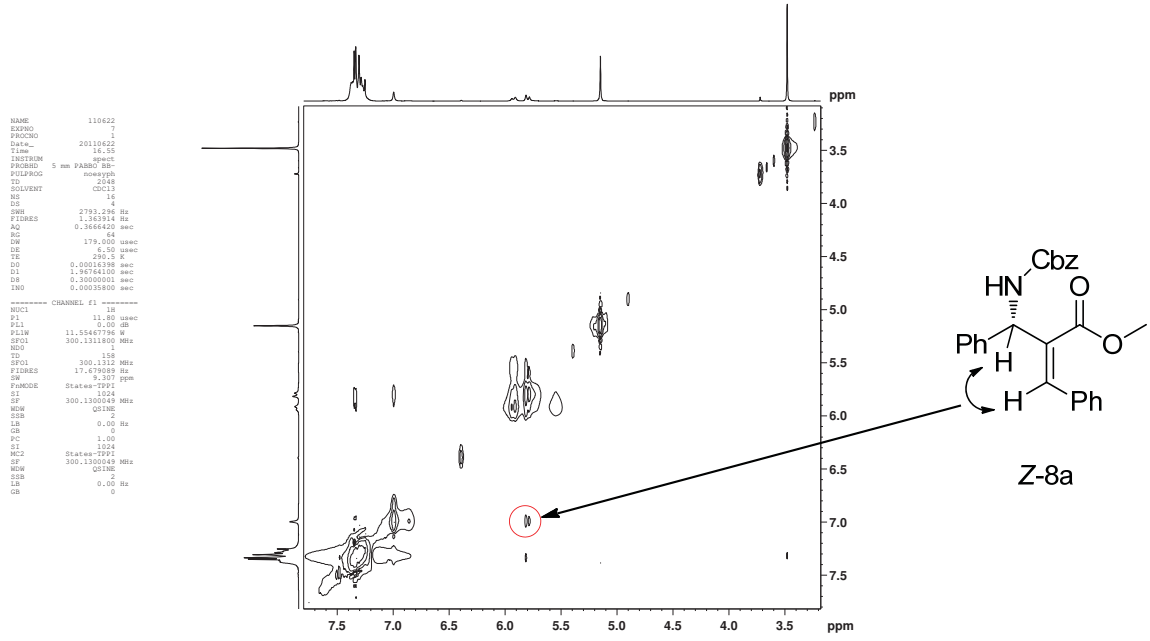


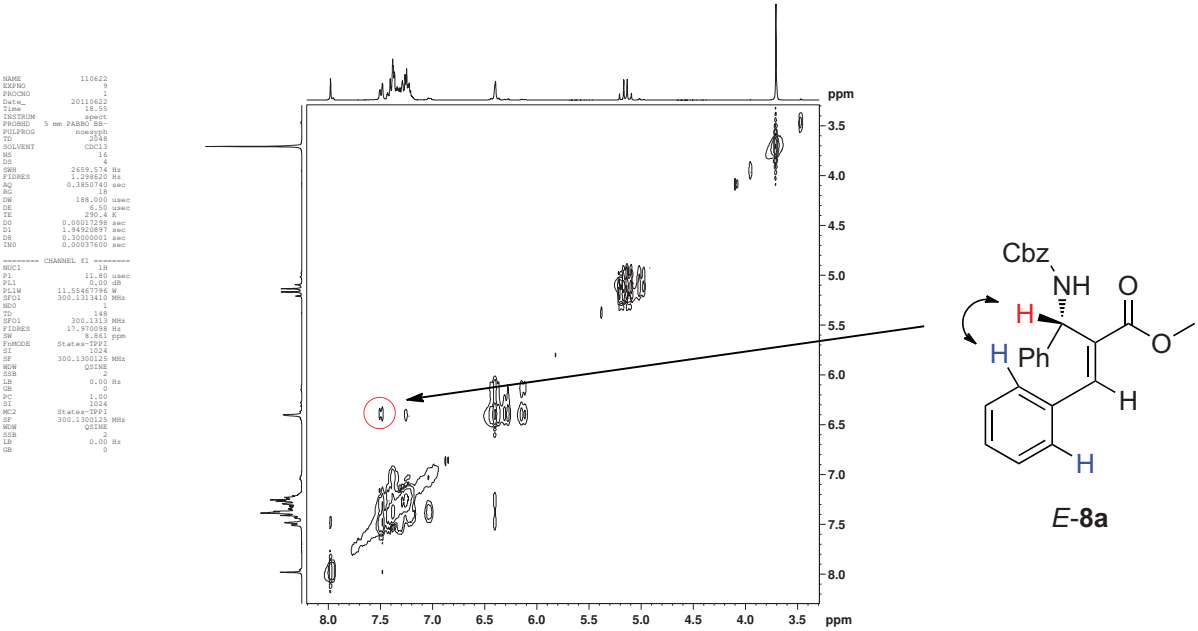
5aa **5a**

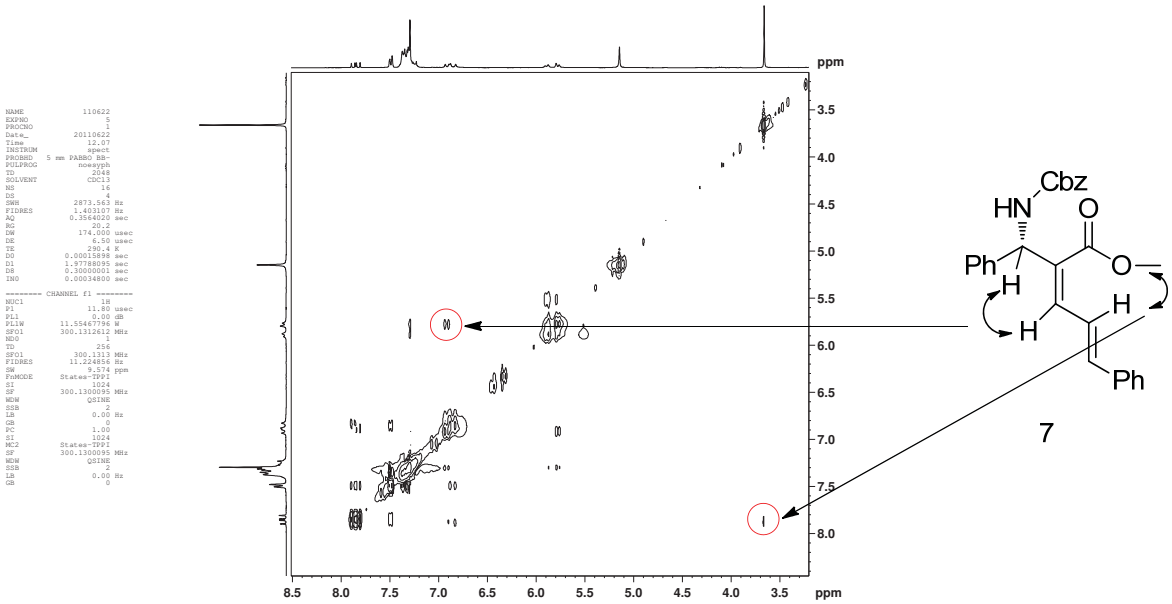
The absolute configuration of compound **5aa** was determined to be *S* by comparison of the optical rotation [$[\alpha]^{20}_D$ = +17.5 (*c* = 0.8, $CHCl_3$, 78% ee)] with a literature value [$[\alpha]^{20}_D$ = +21 (*c* = 0.68, $CHCl_3$) for the *S*-isomer (91% *ee*)].[3] In order to determine the absolute configuration of compound **5a**, **5aa** was converted to **5a**. The resulting product **5a** in this transformation has a accordant value [$[\alpha]^{20}_D$ = +10.6 (*c* = 1.03, $CHCl_3$), 78% ee] to our previous result [$[\alpha]^{20}_D$ = +16.0 (*c* = 1.25, $CHCl_3$) , 91% ee]. So the absolute configuration of compound **5a** in our experiments was determined to be S.

To a solution of **5aa** (97 mg, 0.33 mmol) in EA (1.5 mL) was added concentrated HCl (0.28 mL) at 0 °C. And the mixture was stirred at rt for 2 h. Then the reaction was diluted with water and the aqueous phase was washed with ether. The aqueous phase was then neutralized with NH_3•H_2O and extracted with EA. The organic phase was dried over $NaSO_4$ and evaporated under reduced pressure. The residue was then dissolved in CH_2Cl_2, and pyridine (27 μL, 0.33 mmol) and Cbz-Cl (97 μL, 0.33 mmol) were added at 0 °C. And the reaction was stirred overnight at rt. Then solvent was then evaporated under reduced pressure. The residue was purified by column chromatography and **5a** was obtained in 63% yield (61.5 mg).

Determination of the relative configuration of E-8, Z-8 and 7 by ^{1}H-^{1}H NOESY







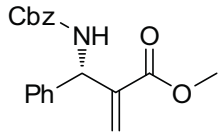
References

[1] T. Okino, Y. Hoashi, Y. Takemoto, *J. Am. Chem. Soc*., 2003, **125**, 12672–12673.

[2] N. Yamagiwa, H. Qin, S. Matsunaga, M. Shibasaki, *J. Am. Chem. Soc*., 2005, **127**, 13419–13427.

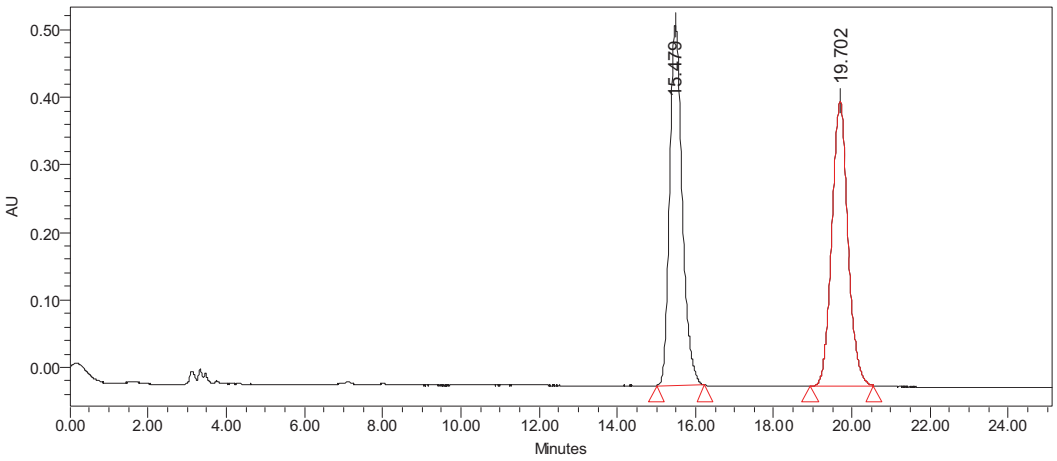
[3] C. Cassani, L. Bernardi, F. Fini, A. Ricci, *Angew. Chem. Int. Ed.,* 2009, **48**, 5694 –5697.

Copies of HPLC results

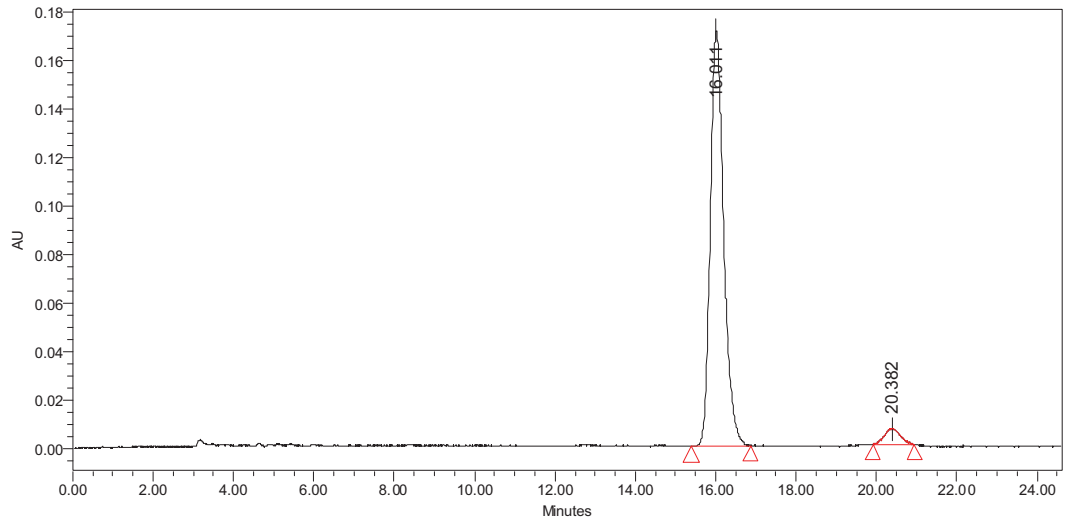


5a

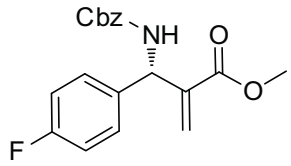
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 85/15, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	15.479	11942706	49.79	533201
2	19.702	12044967	50.21	421524

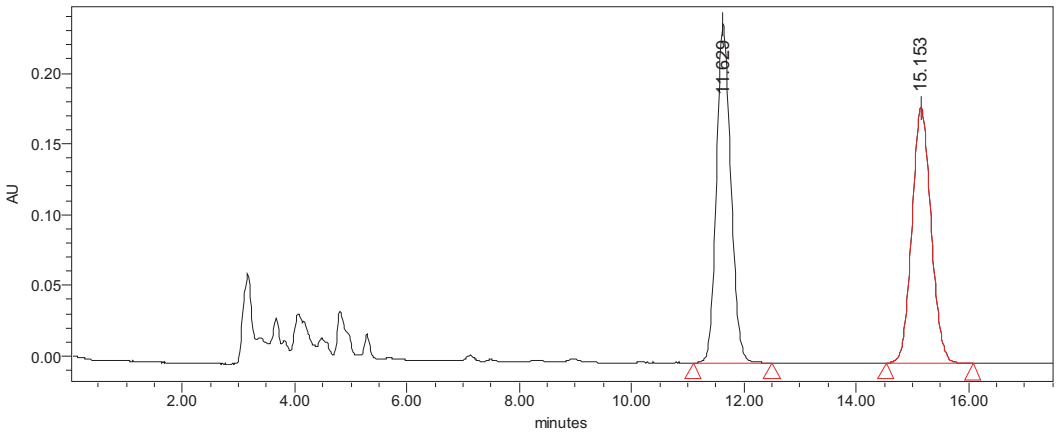


Name	Retention Time	Area	% Area	Height
1	16.011	3969118	95.87	171573
2	20.382	171006	4.13	6391

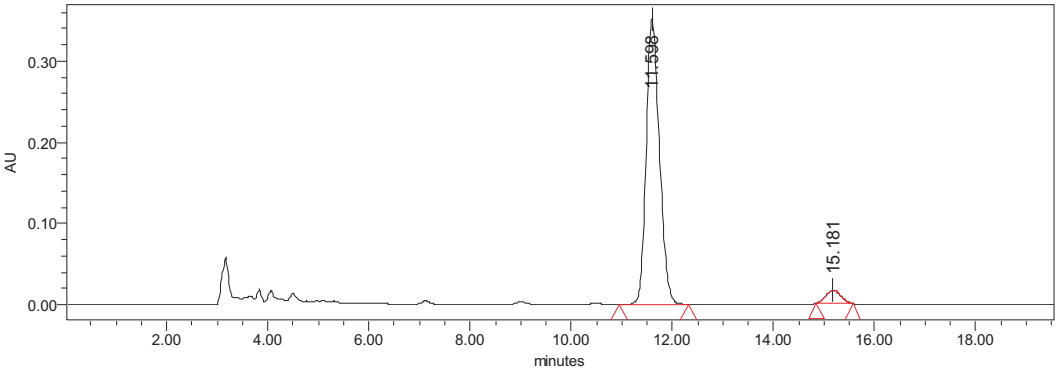


5b

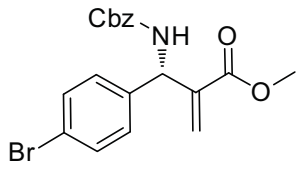
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	11.629	4285773	50.04	239695
2	15.153	4278429	49.96	180704

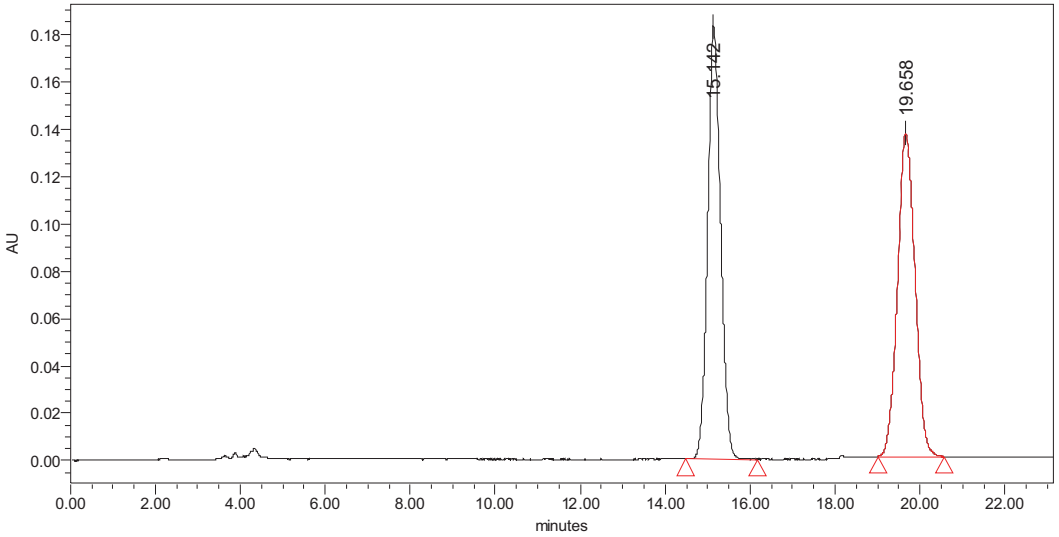


Name	Retention Time	Area	% Area	Height
1	11.598	6383457	94.99	351612
2	15.181	336737	5.01	15982

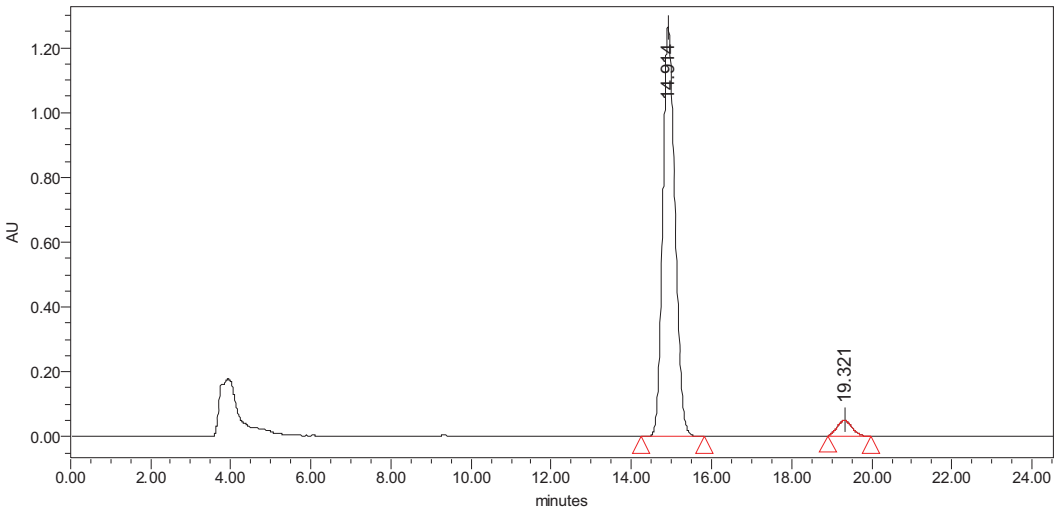


5c

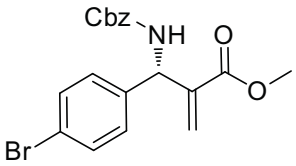
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		15.142	3895406	49.85	182734
2		19.658	3918491	50.15	136472

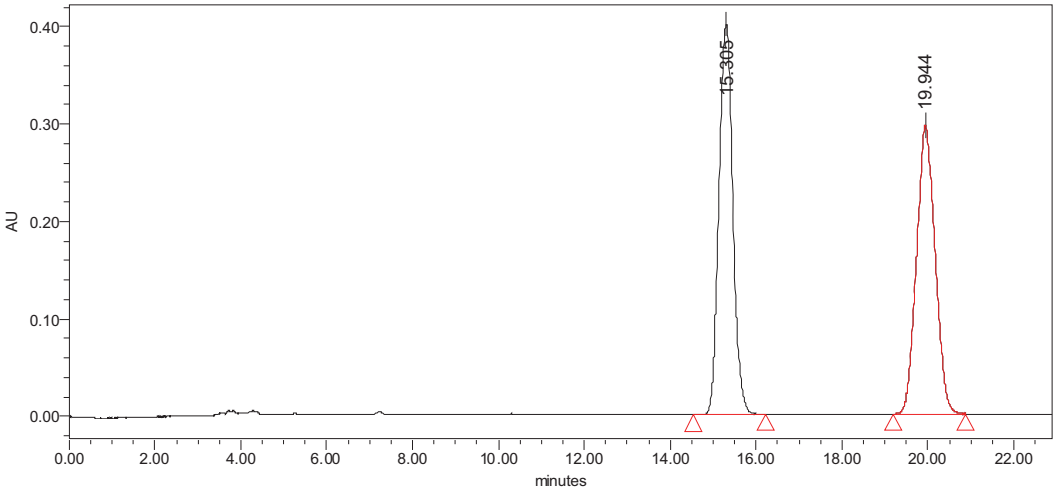


	Name	Retention Time	Area	% Area	Height
1		14.914	26848287	95.50	1262336
2		19.321	1264908	4.50	48251

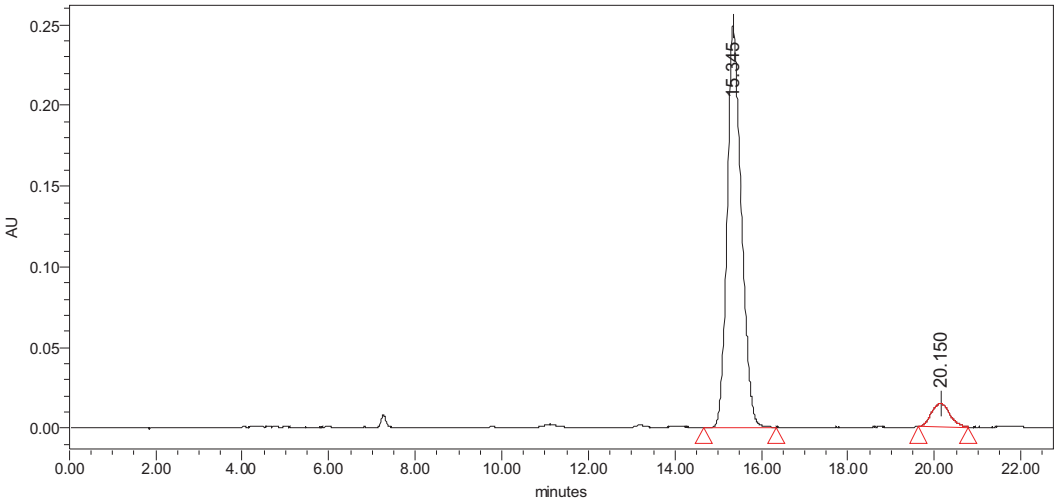


5d

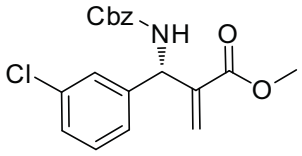
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	15.305	9041766	50.46	400427
2	19.944	8875362	49.54	296249

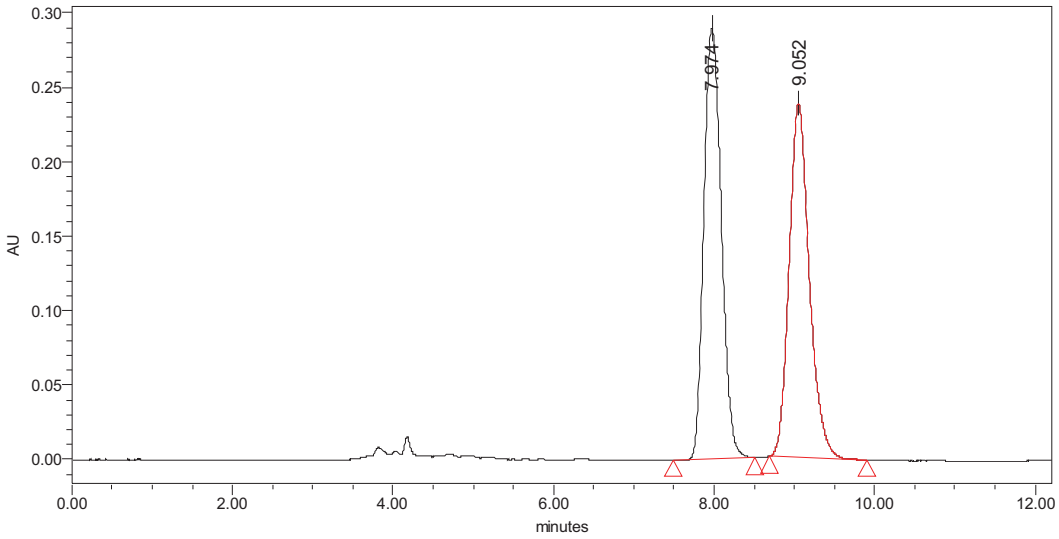


Name	Retention Time	Area	% Area	Height
1	15.345	5672922	93.04	248897
2	20.150	424479	6.96	14435

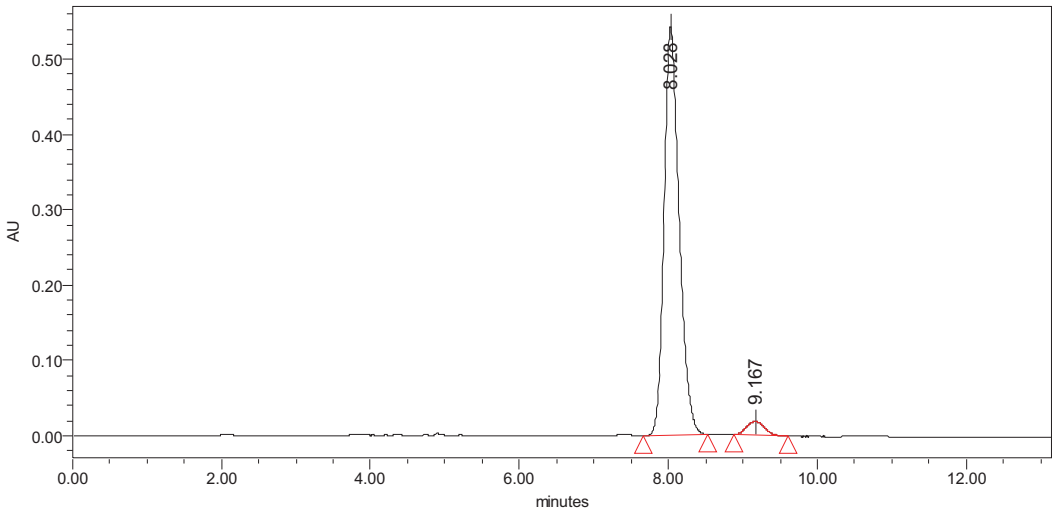


5e

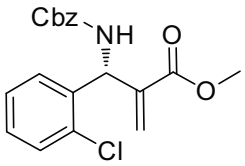
HPLC using an OD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	7.974	4395841	51.30	289348
2	9.052	4173660	48.70	237741

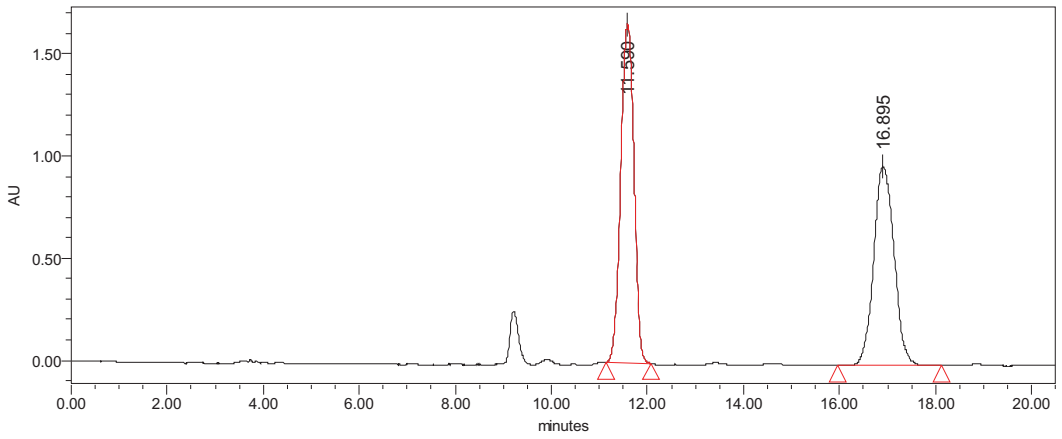


Name	Retention Time	Area	% Area	Height
1	8.028	7688916	96.04	542736
2	9.167	316819	3.96	18285

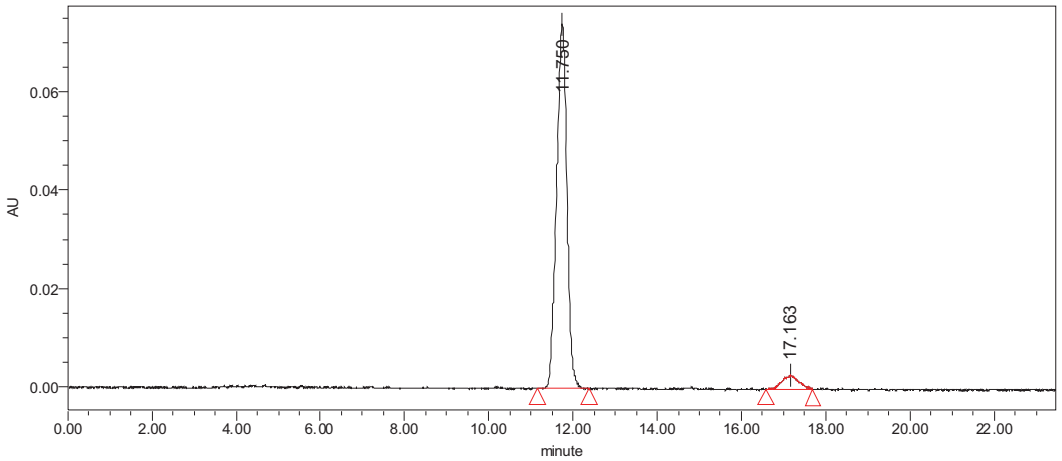


5f

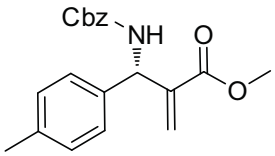
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		11.590	31800541	52.52	1656888
2		16.895	28748866	47.48	975259

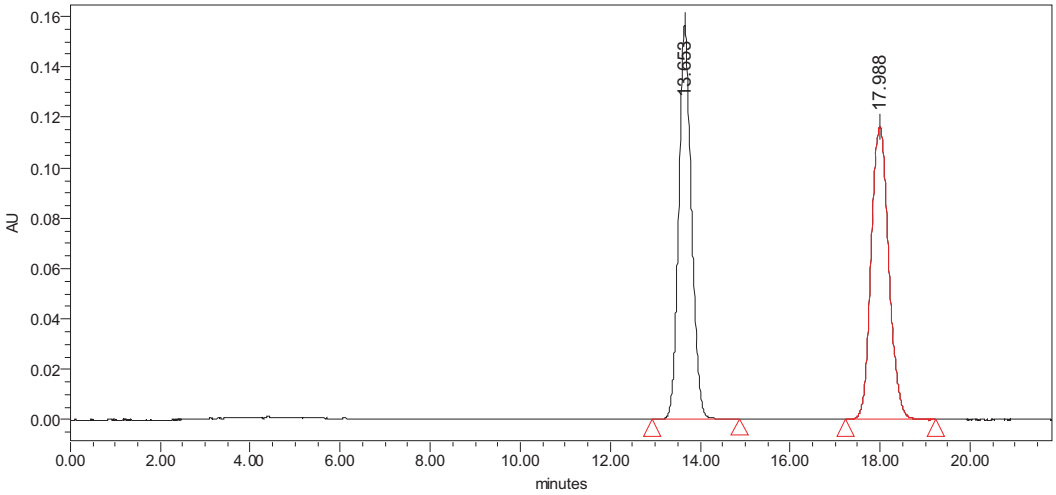


	Name	Retention Time	Area	% Area	Height
1	11.750	7249977	95.22	424011	11.749
2	17.163	364191	4.78	13850	17.173

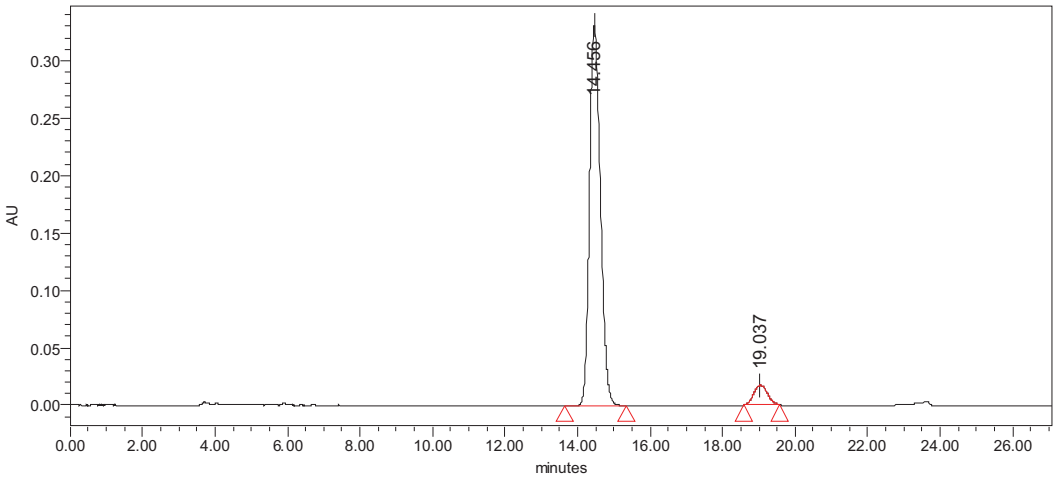


5g

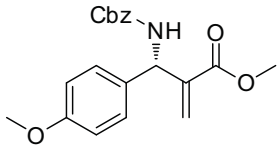
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	13.653	3090878	49.38	156513
2	17.988	3168804	50.62	116256

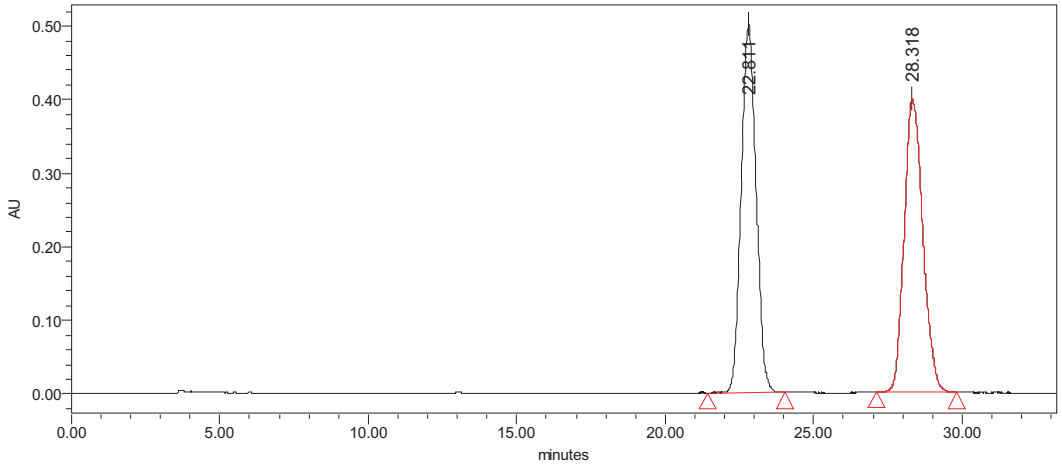


Name	Retention Time	Area	% Area	Height
1	14.456	7019815	93.89	331516
2	19.037	456470	6.11	17225

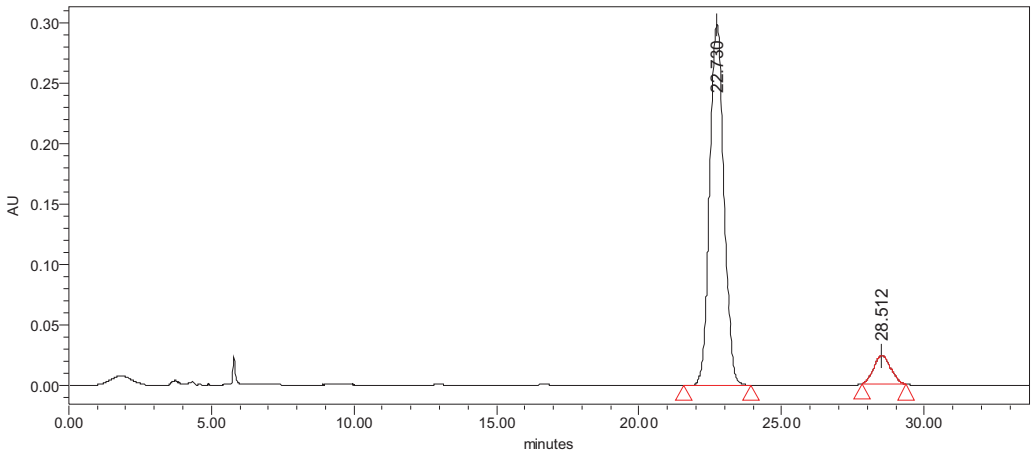


5h

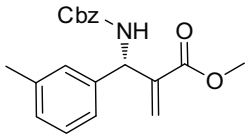
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



	Name	Retention Time	Area	% Area	Height
1		22.811	17547557	50.10	501873
2		28.318	17474980	49.90	398233

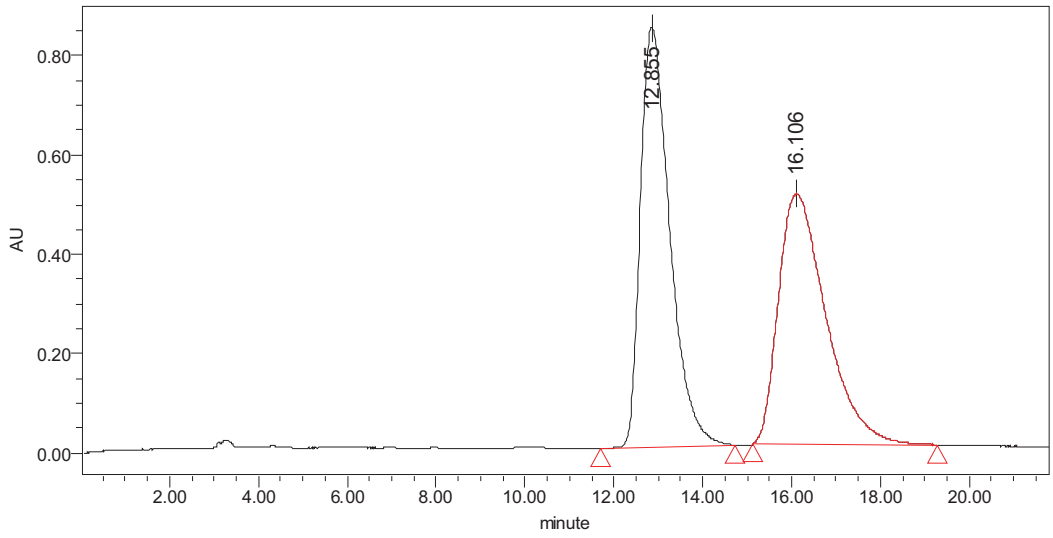


	Name	Retention Time	Area	% Area	Height
1		22.730	10417780	91.41	297937
2		28.512	979352	8.59	23263

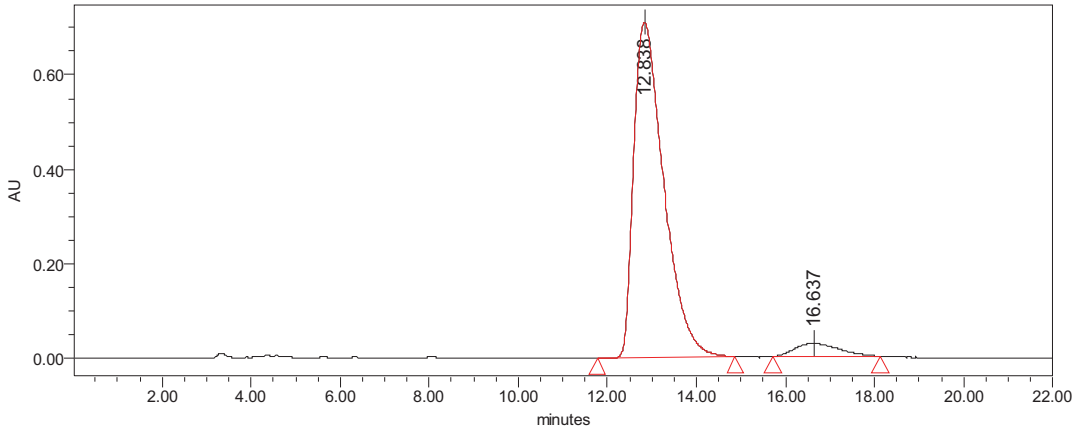


5i

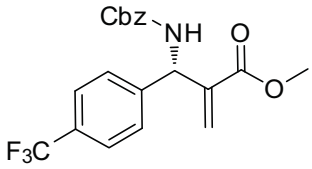
HPLC using an OJ-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	12.855	38697782	50.78	845119
2	16.106	37508851	49.22	504886

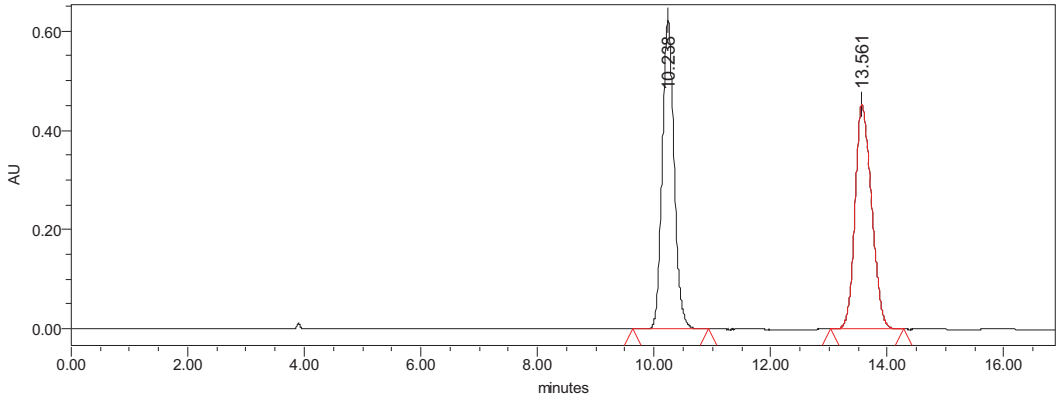


Name	Retention Time	Area	% Area	Height
1	12.838	33249911	94.57	710374
2	16.637	1908606	5.43	27714

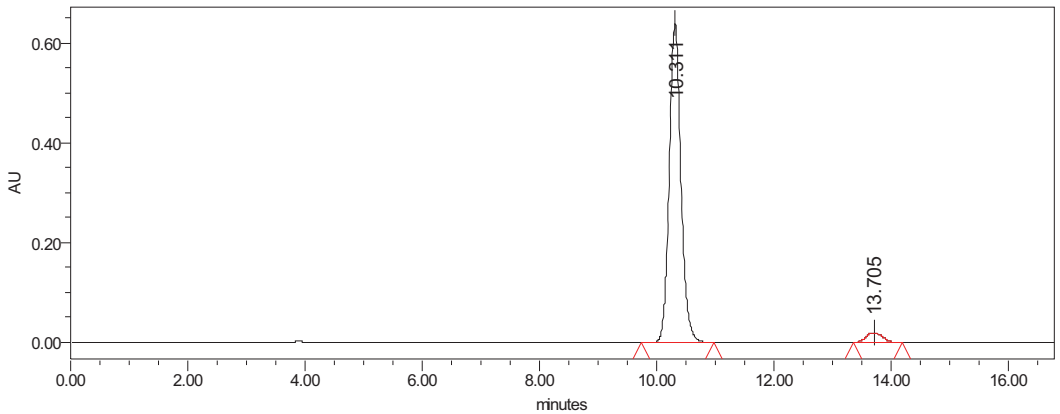


5j

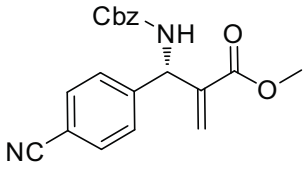
HPLC using an OD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	10.238	8827014	49.63	622102
2	13.561	8958924	50.37	453134

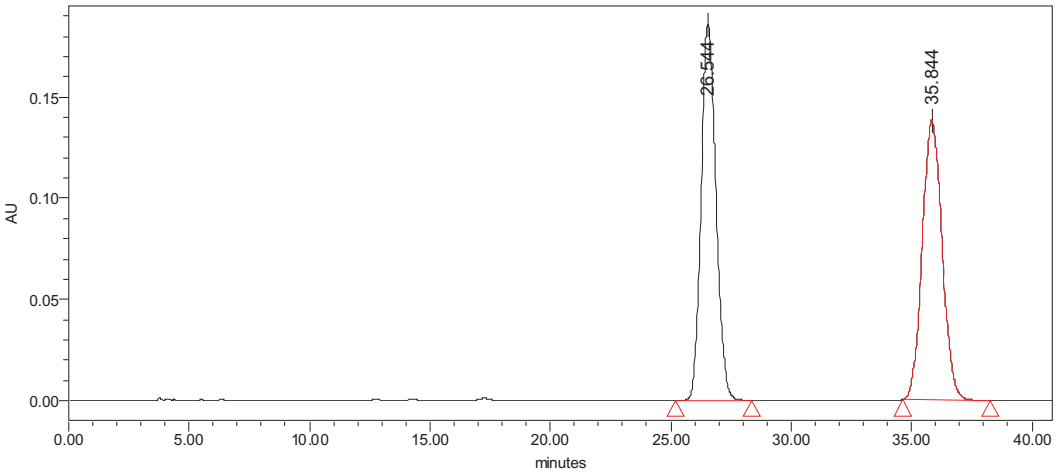


Name	Retention Time	Area	% Area	Height
1	10.311	8846796	95.89	639964
2	13.705	379119	4.11	19040

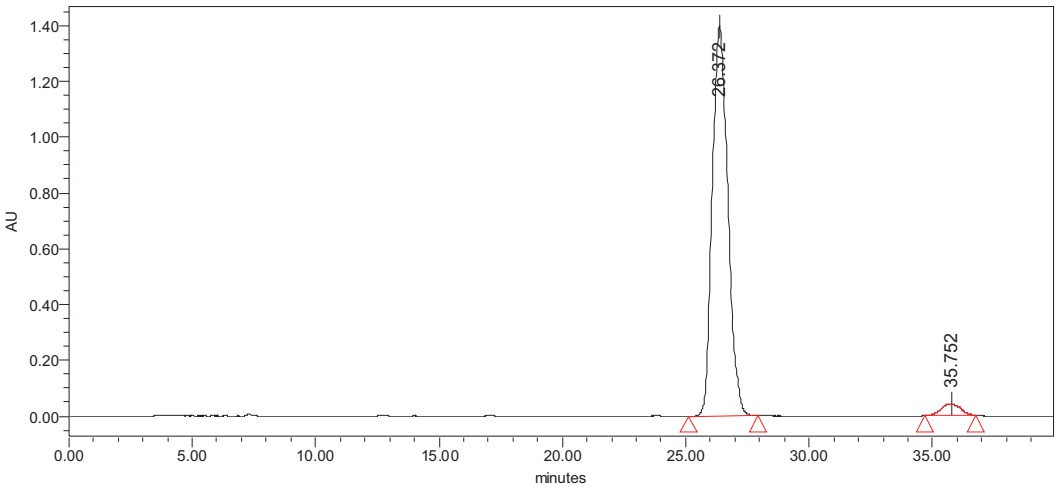


5k

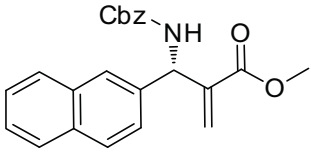
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	26.544	7929876	50.15	185475
2	35.844	7883654	49.85	137948

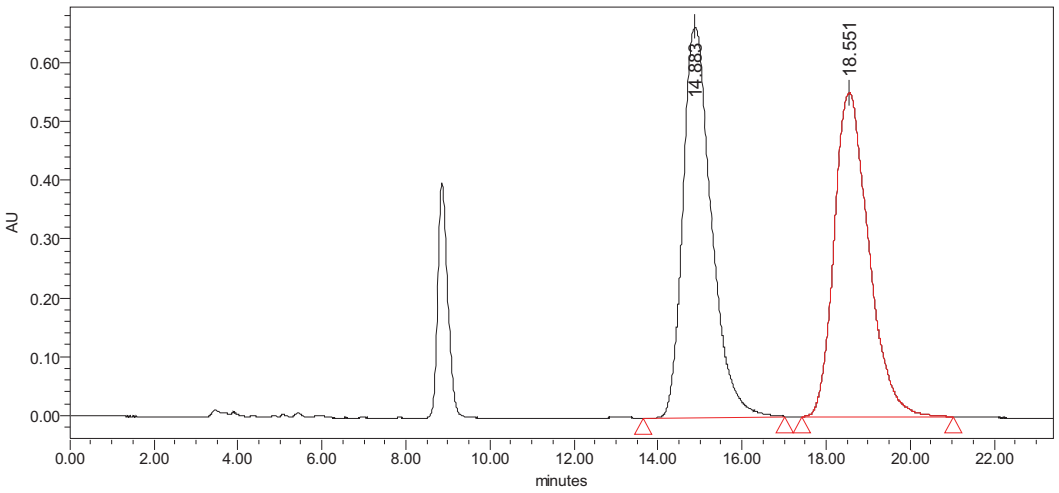


Name	Retention Time	Area	% Area	Height
1	26.372	61682719	96.28	1398023
2	35.752	2385530	3.72	43433

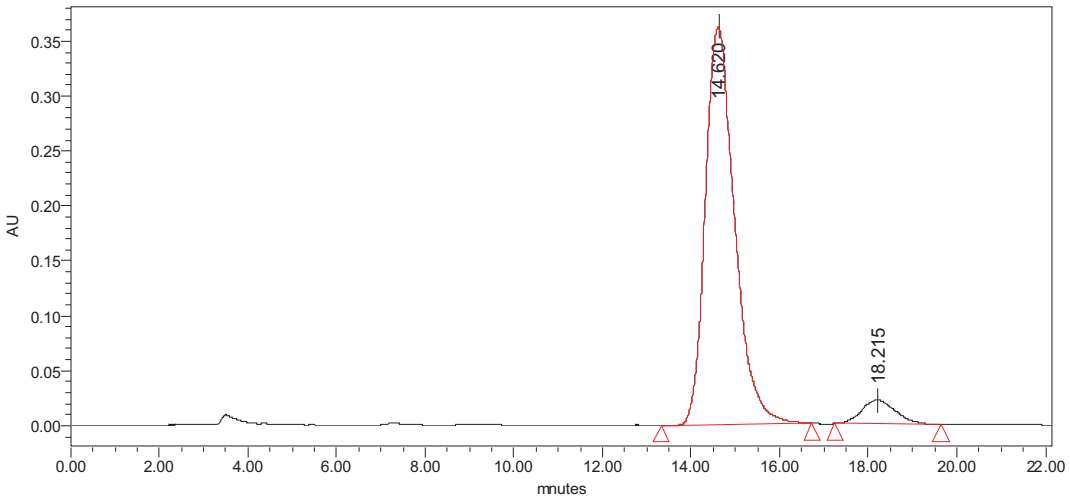


5l

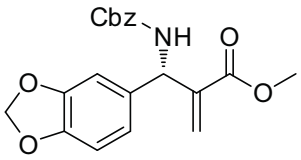
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	14.883	31424703	50.00	664890
2	18.551	31427098	50.00	551302

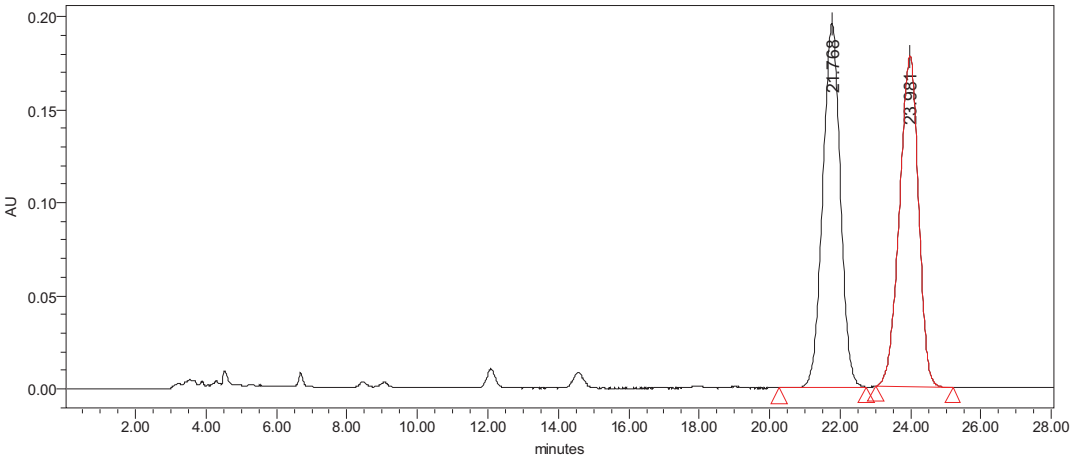


Name	Retention Time	Area	% Area	Height
1	14.620	16681524	93.41	362295
2	18.215	1177426	6.59	21674

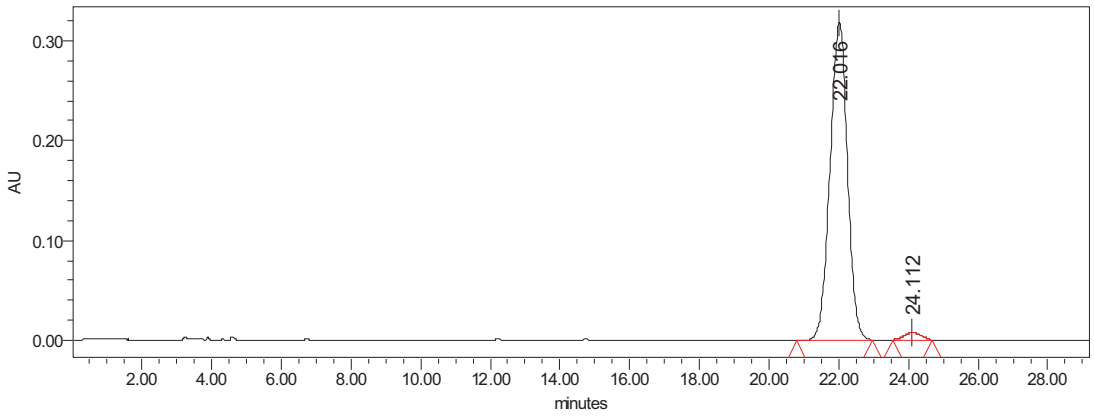


5m

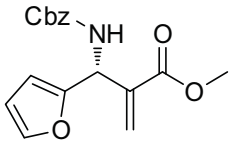
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	21.768	6809774	50.42	195387
2	23.981	6696452	49.58	177599

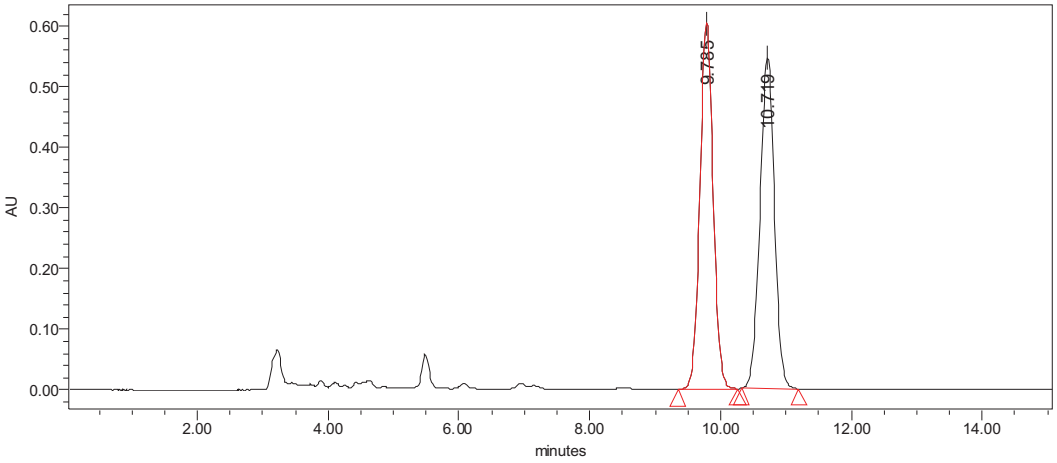


Name	Retention Time	Area	% Area	Height
1	22.016	11106099	97.88	318175
2	24.112	240957	2.12	7206

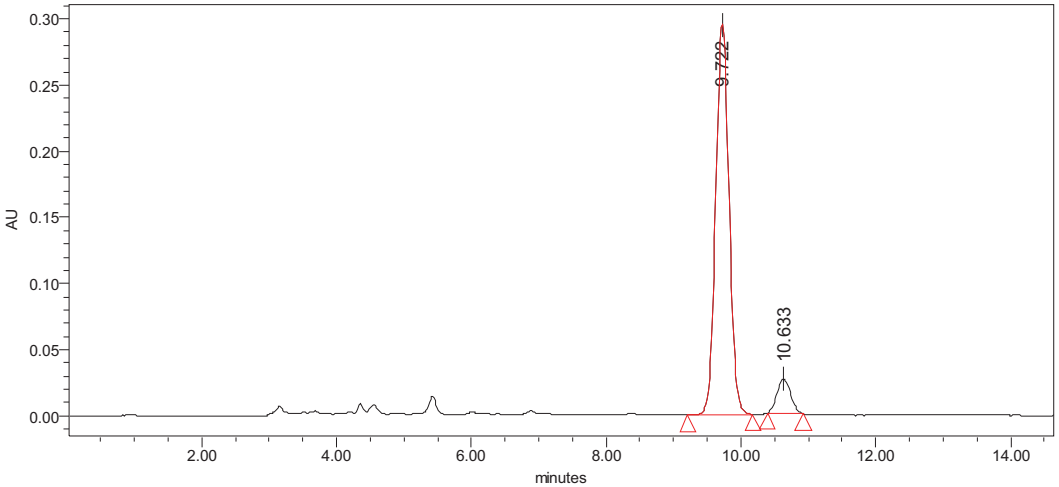


5n

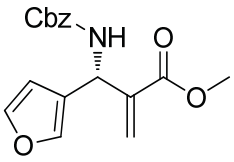
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	9.785	8526490	50.34	604288
2	10.719	8412600	49.66	546545

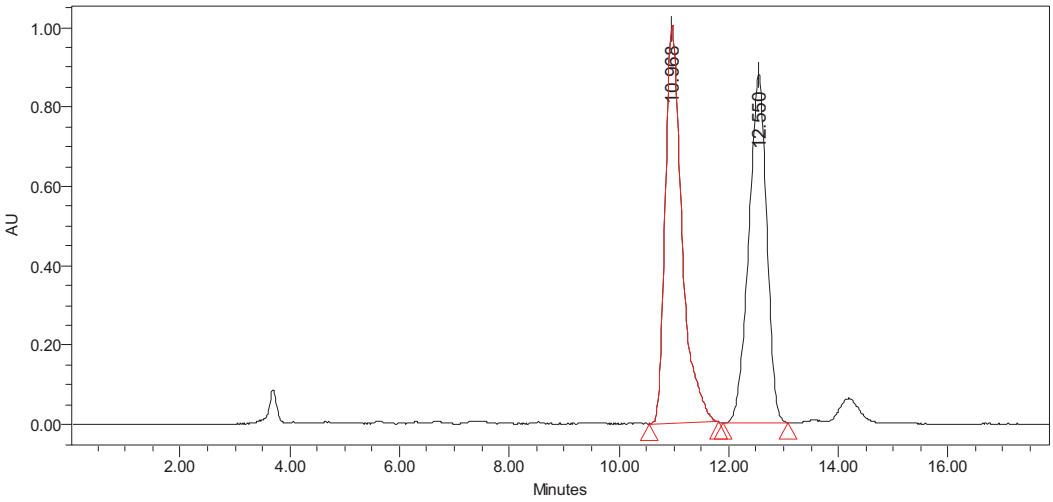


Name	Retention Time	Area	% Area	Height
1	9.722	4067597	91.76	295378
2	10.633	365365	8.24	25791

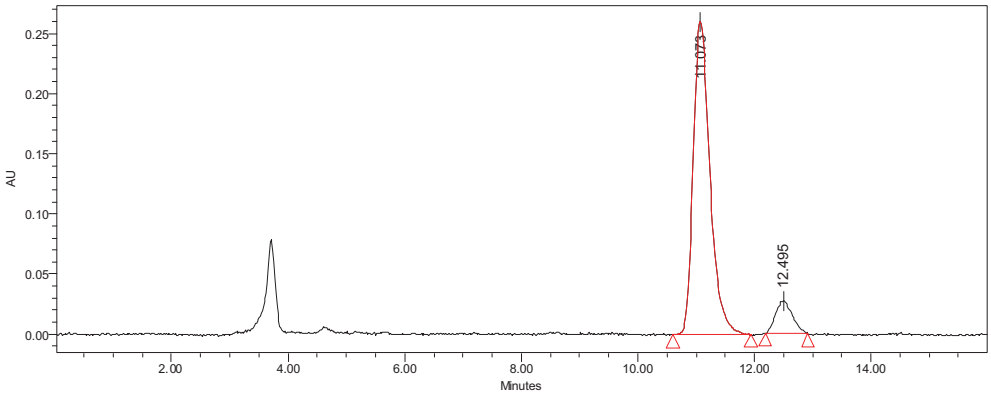


5o

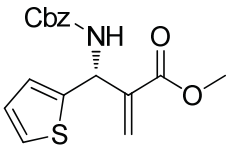
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	10.968	21242179	51.63	995315
2	12.550	19897864	48.37	877061

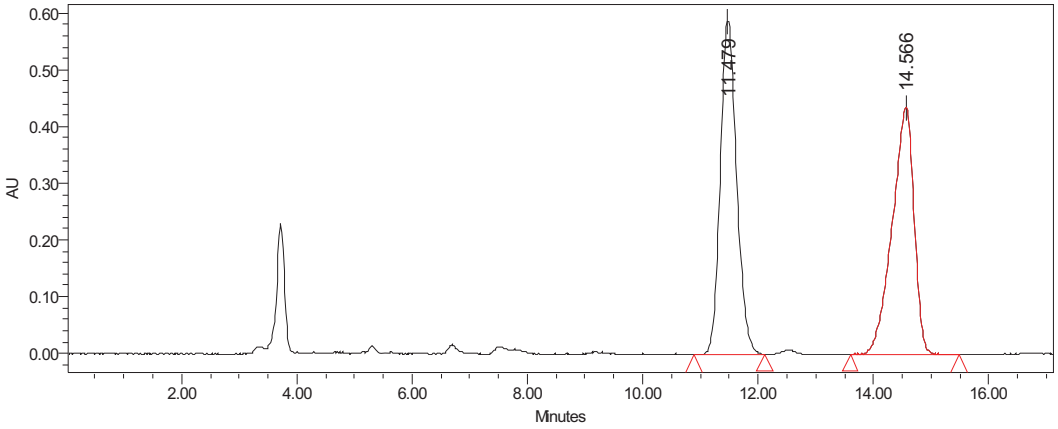


Name	Retention Time	Area	% Area	Height
1	11.073	5200100	90.55	260417
2	12.495	542865	9.45	26987

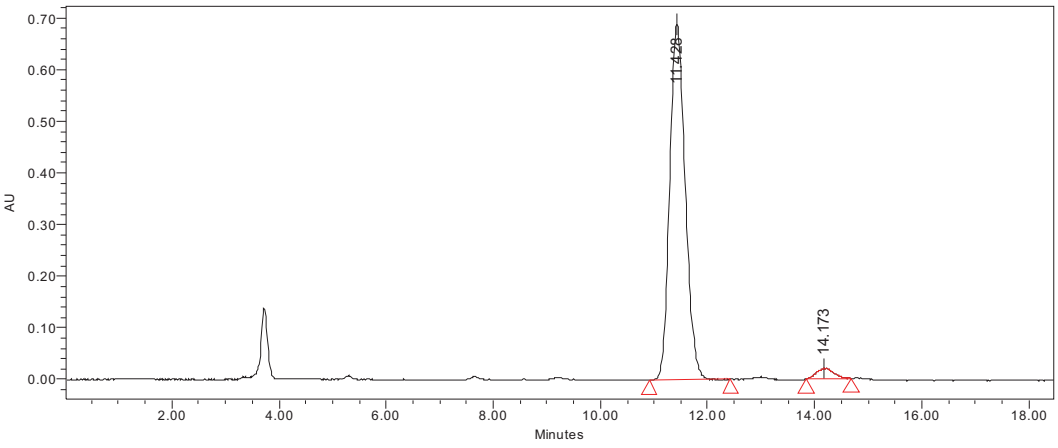


5p

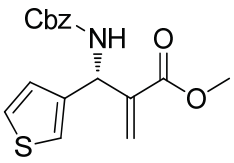
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	11.479	11630577	50.38	589183
2	14.566	11454482	49.62	435798

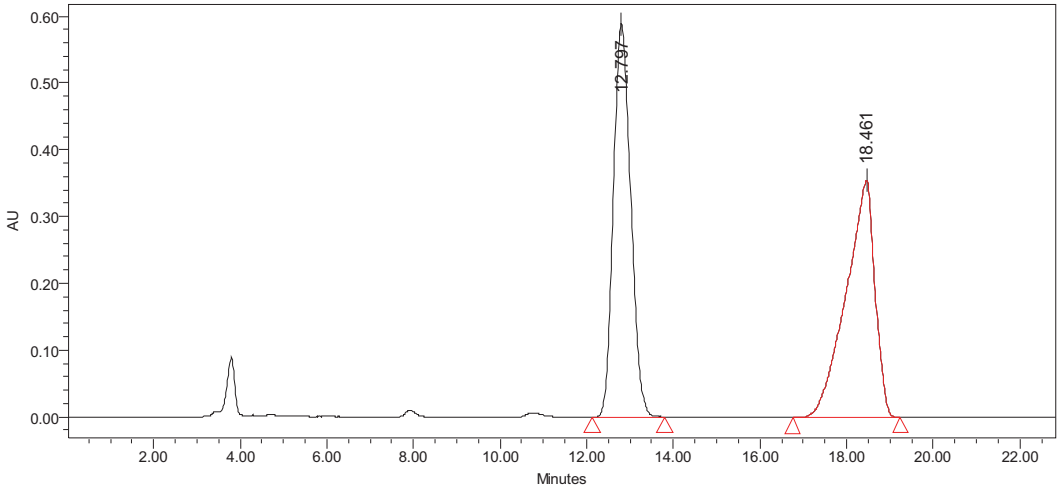


Name	Retention Time	Area	% Area	Height
1	11.428	13820894	96.85	689960
2	14.173	450198	3.15	19726

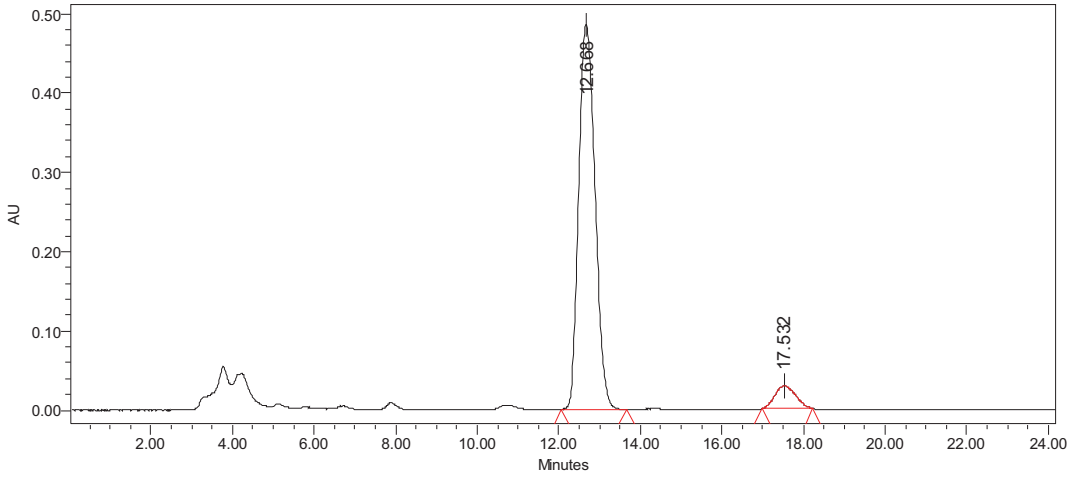


5q

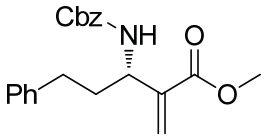
HPLC using an AD-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	12.797	16458367	50.01	588887
2	18.461	16448977	49.99	355226

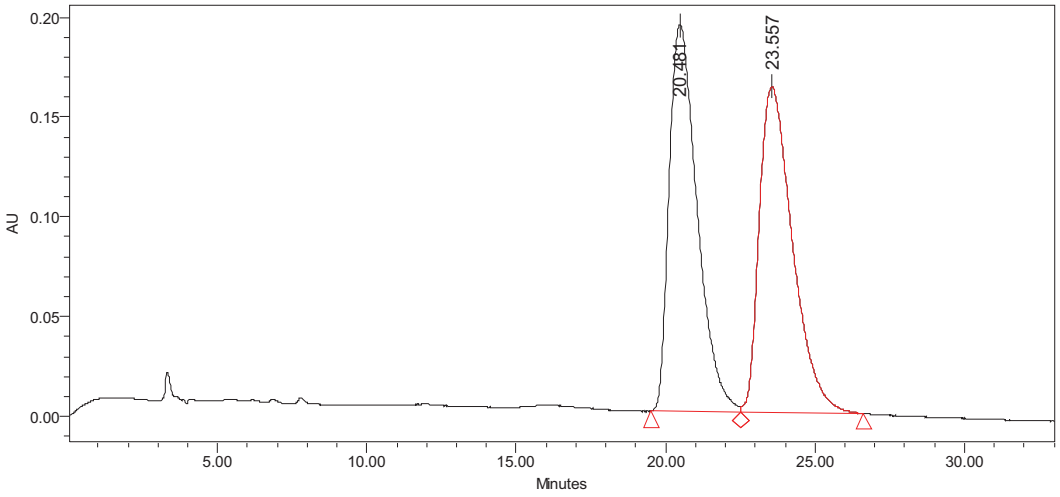


Name	Retention Time	Area	% Area	Height
1	12.668	13365625	92.88	485954
2	17.532	1024105	7.12	28531

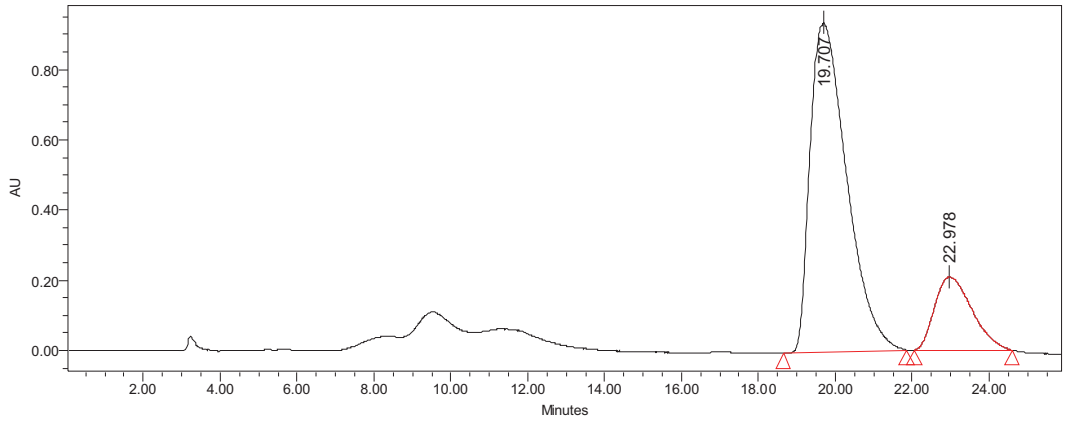


5r

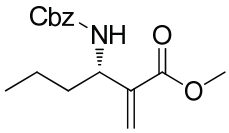
HPLC using an OJ-H (*n*-Hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	20.481	12645693	49.94	193531
2	23.557	12673913	50.06	163285

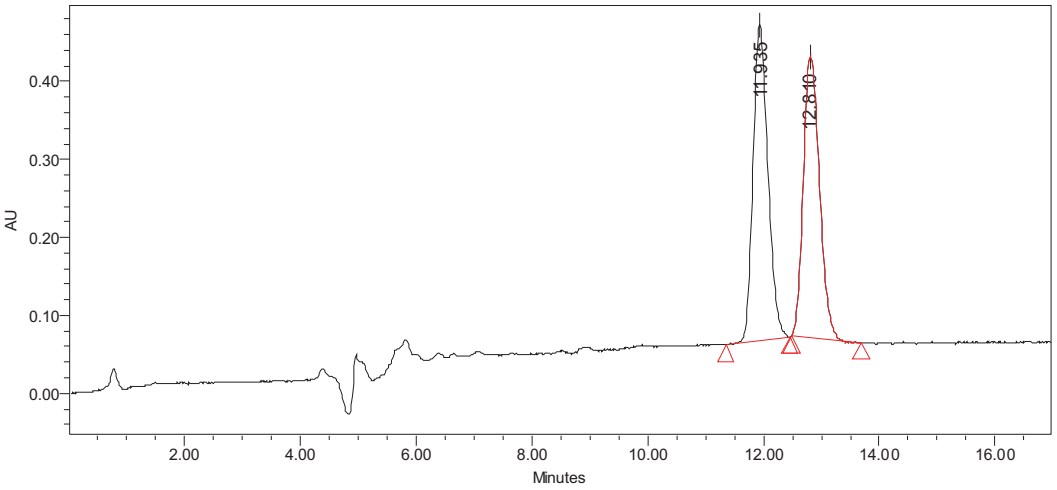


Name	Retention Time	Area	% Area	Height
1	19.707	62532916	81.70	940014
2	22.978	14011251	18.30	208346

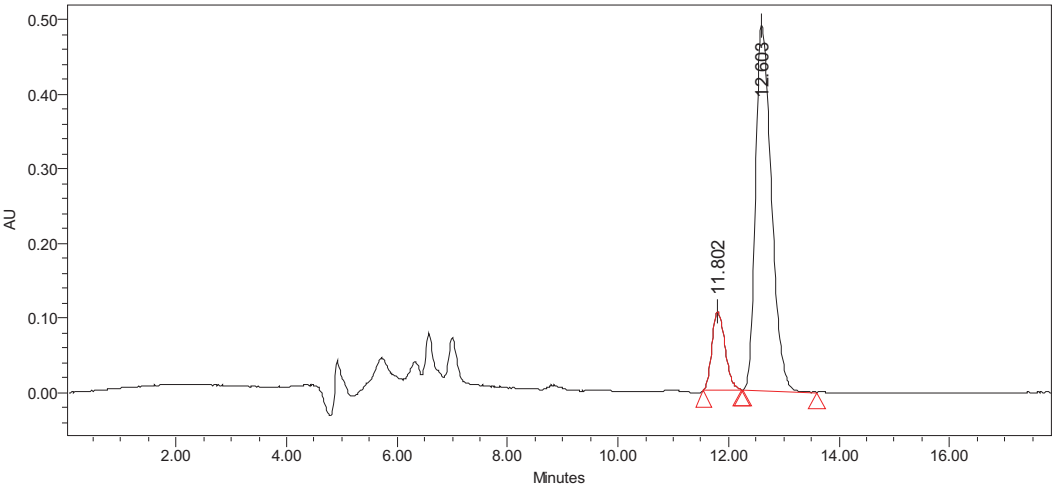


5s

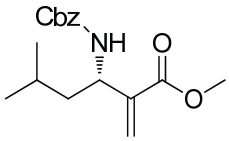
HPLC using an OD-H (*n*-Hexane/*i*PrOH = 95/5, flow rate 0.75 mL/min)



Name	Retention Time	Area	% Area	Height
1	11.935	7194102	50.54	405207
2	12.810	7039630	49.46	362363

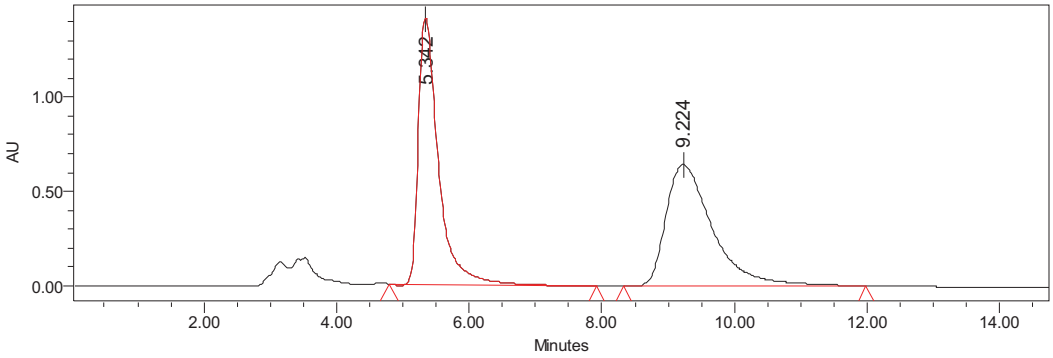


Name	Retention Time	Area	% Area	Height
1	11.802	1740720	15.05	105165
2	12.603	9822470	84.95	489487

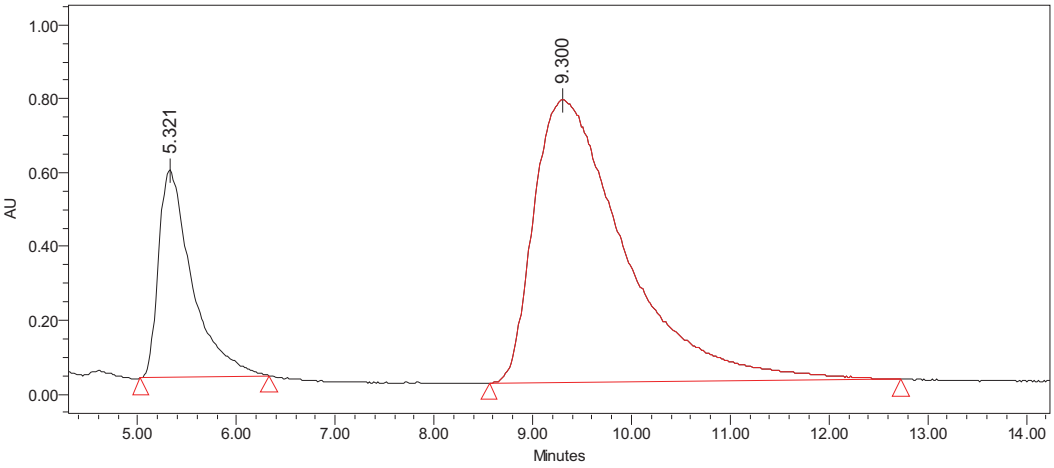


5t

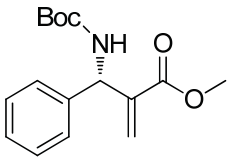
HPLC using an AS (*n*-Hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	5.342	29958703	48.81	1413767
2	9.224	31414115	51.19	643457

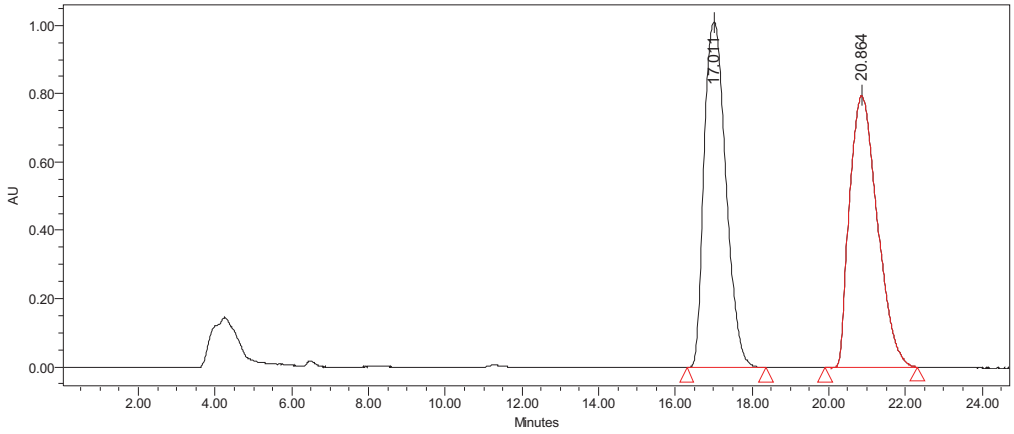


Name	Retention Time	Area	% Area	Height
1	5.321	13313258	21.37	562599
2	9.300	48983471	78.63	763405

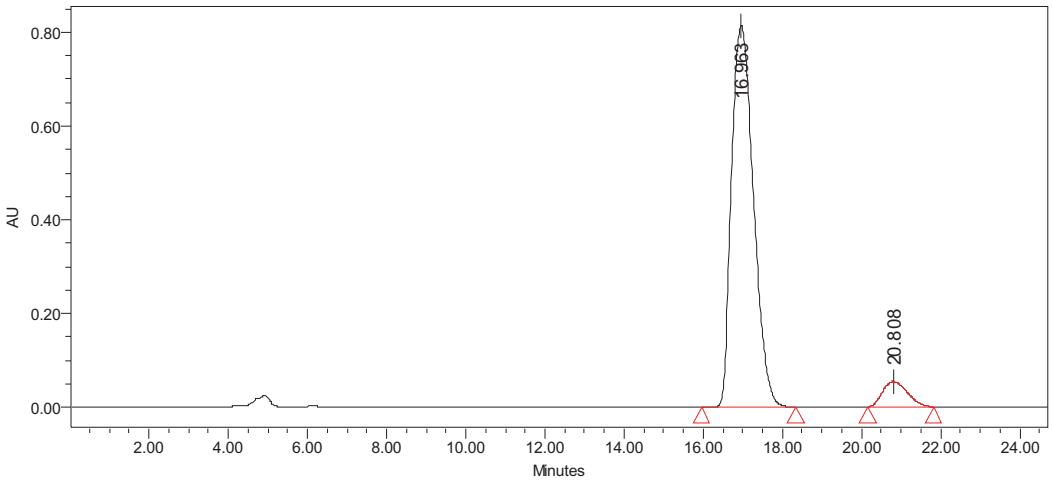


5aa

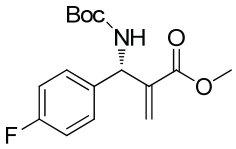
HPLC using an AD (*n*-Hexane/*i*PrOH = 95/5, flow rate 0.75 mL/min)



Name	Retention Time	Area	% Area	Height
1	17.011	40050653	50.05	1011877
2	20.864	39969933	49.95	796547

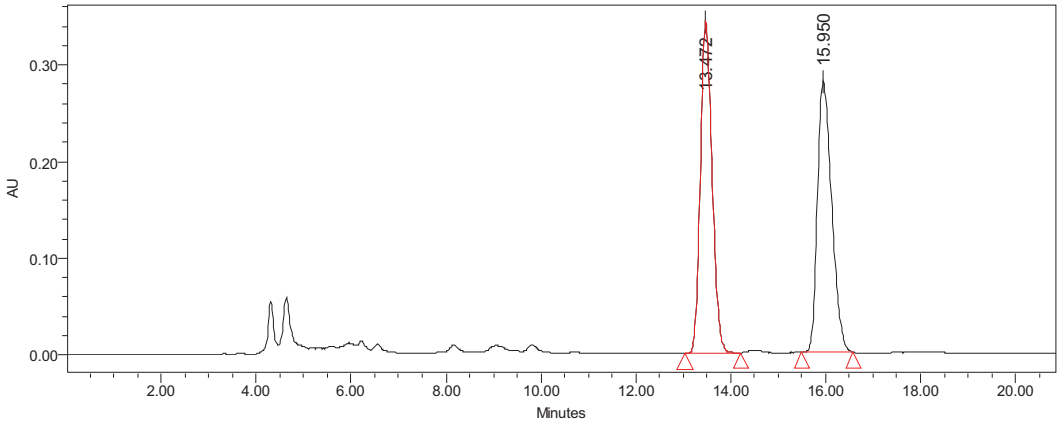


Name	Retention Time	Area	% Area	Height
1	16.963	31431189	92.99	813506
2	20.808	2368506	7.01	53579

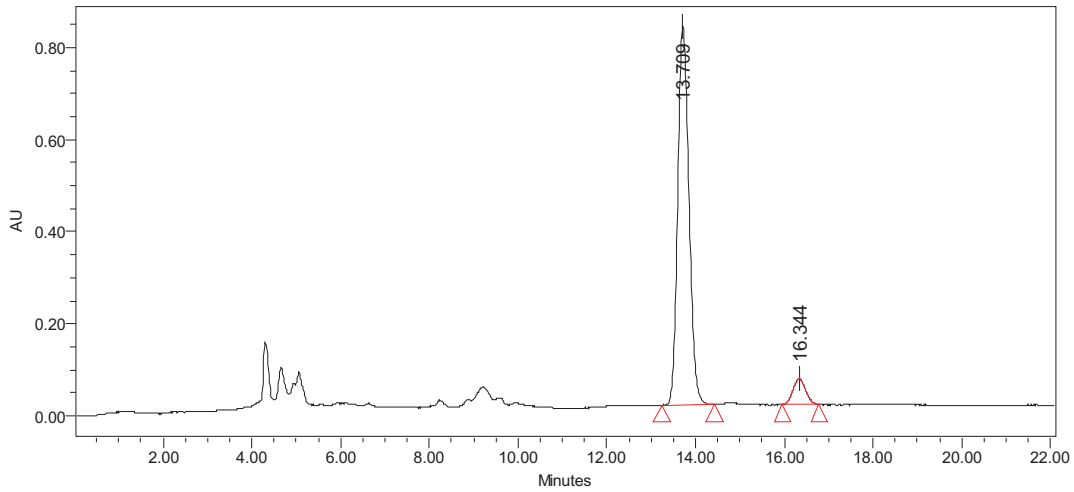


5ab

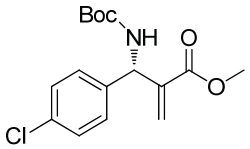
HPLC using an AD (*n*-Hexane/*i*PrOH = 95/5, flow rate 0.8 mL/min)



	Name	Retention Time	Area	% Area	Height
1		13.472	5874253	50.15	343613
2		15.950	5838007	49.85	280491

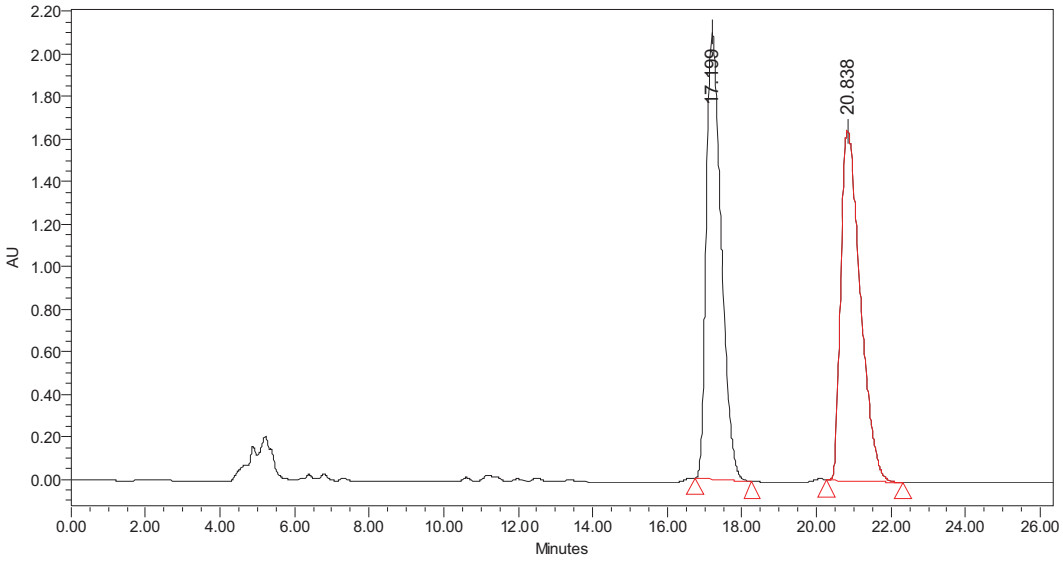


	Name	Retention Time	Area	% Area	Height
1		13.709	14595671	92.96	823303
2		16.344	1105863	7.04	56612

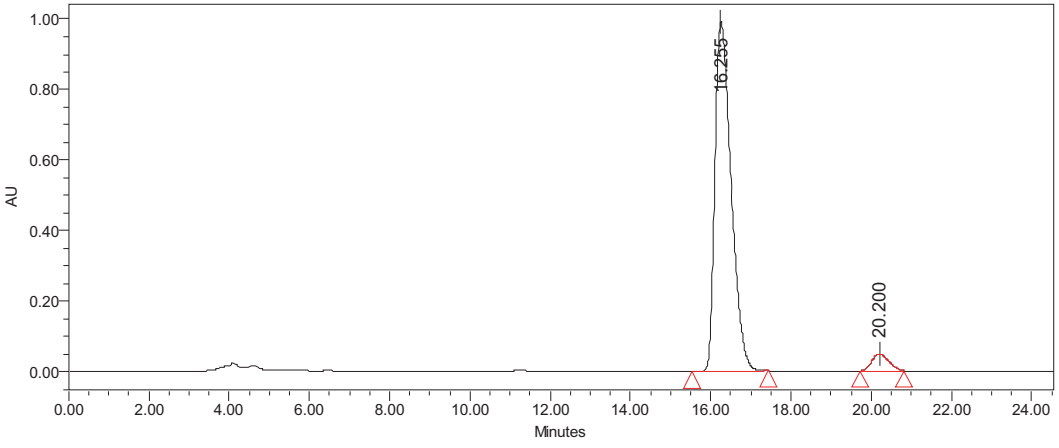


5ad

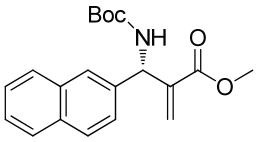
HPLC using an AD (*n*-Hexane/*i*PrOH = 95/5, flow rate 0.8 mL/min)



Name	Retention Time	Area	% Area	Height
1	17.199	60155248	52.80	2102641
2	20.838	53779523	47.20	1650916

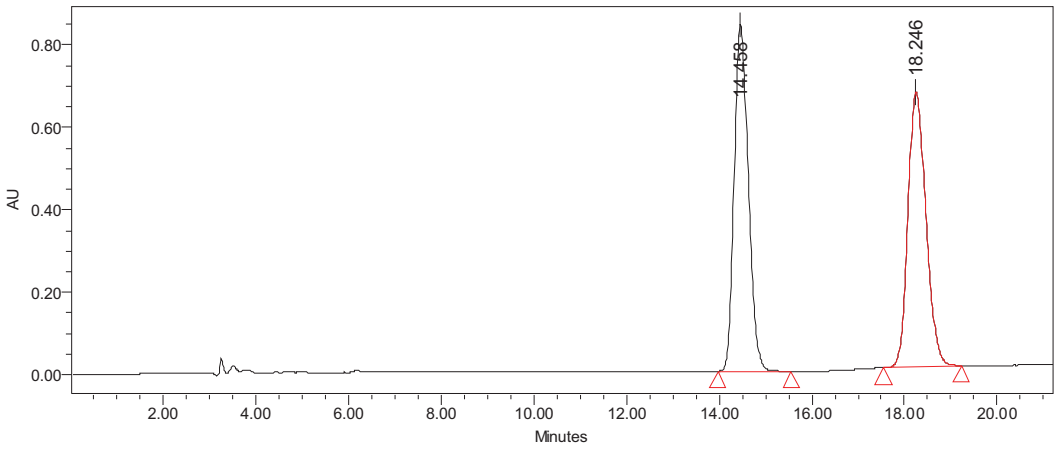


Name	Retention Time	Area	% Area	Height
1	16.255	25328933	94.41	991054
2	20.200	1498319	5.59	48366

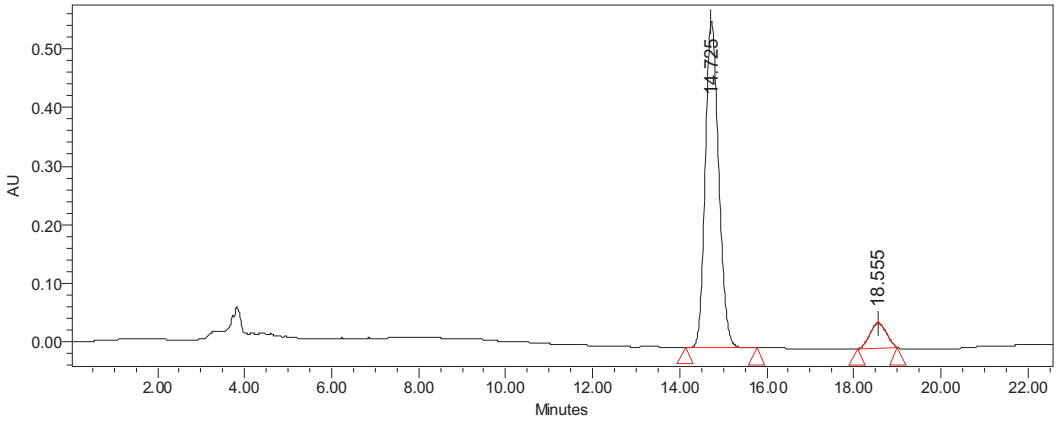


5al

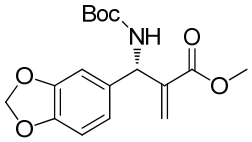
HPLC using an AD (*n*-Hexane/*i*PrOH = 95/5, flow rate 1.0 mL/min)



Name	Retention Time	Area	% Area	Height
1	14.458	17866060	49.95	841907
2	18.246	17901610	50.05	667220

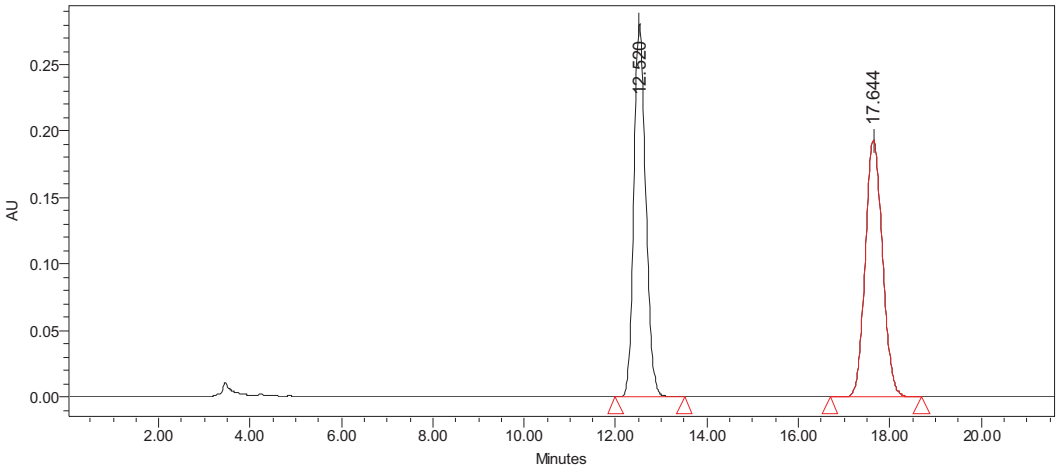


Name	Retention Time	Area	% Area	Height
1	14.725	11910436	91.25	556903
2	18.555	1142800	8.75	42806

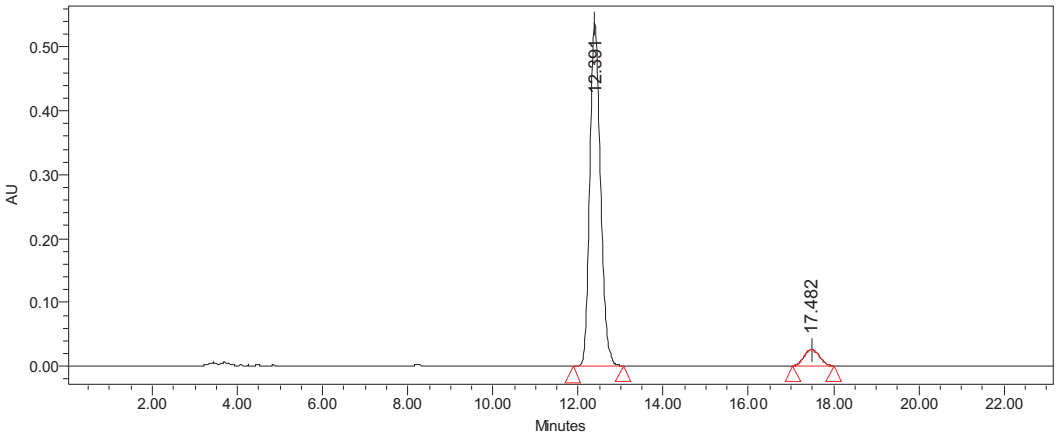


5am

HPLC using an AD (*n*-Hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min)

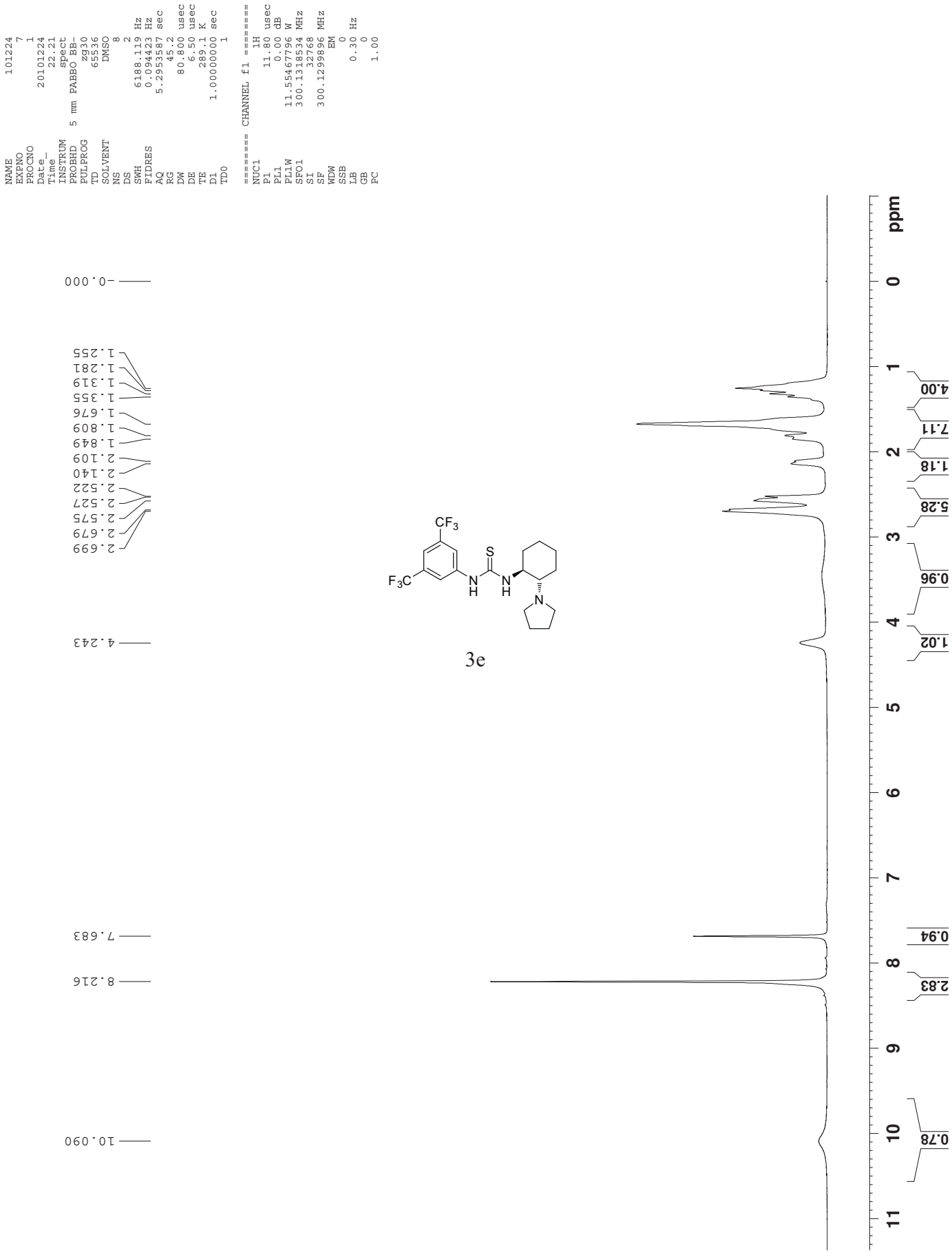


	Name	Retention Time	Area	% Area	Height
1		12.520	5079794	50.05	280641
2		17.644	5070638	49.95	193282

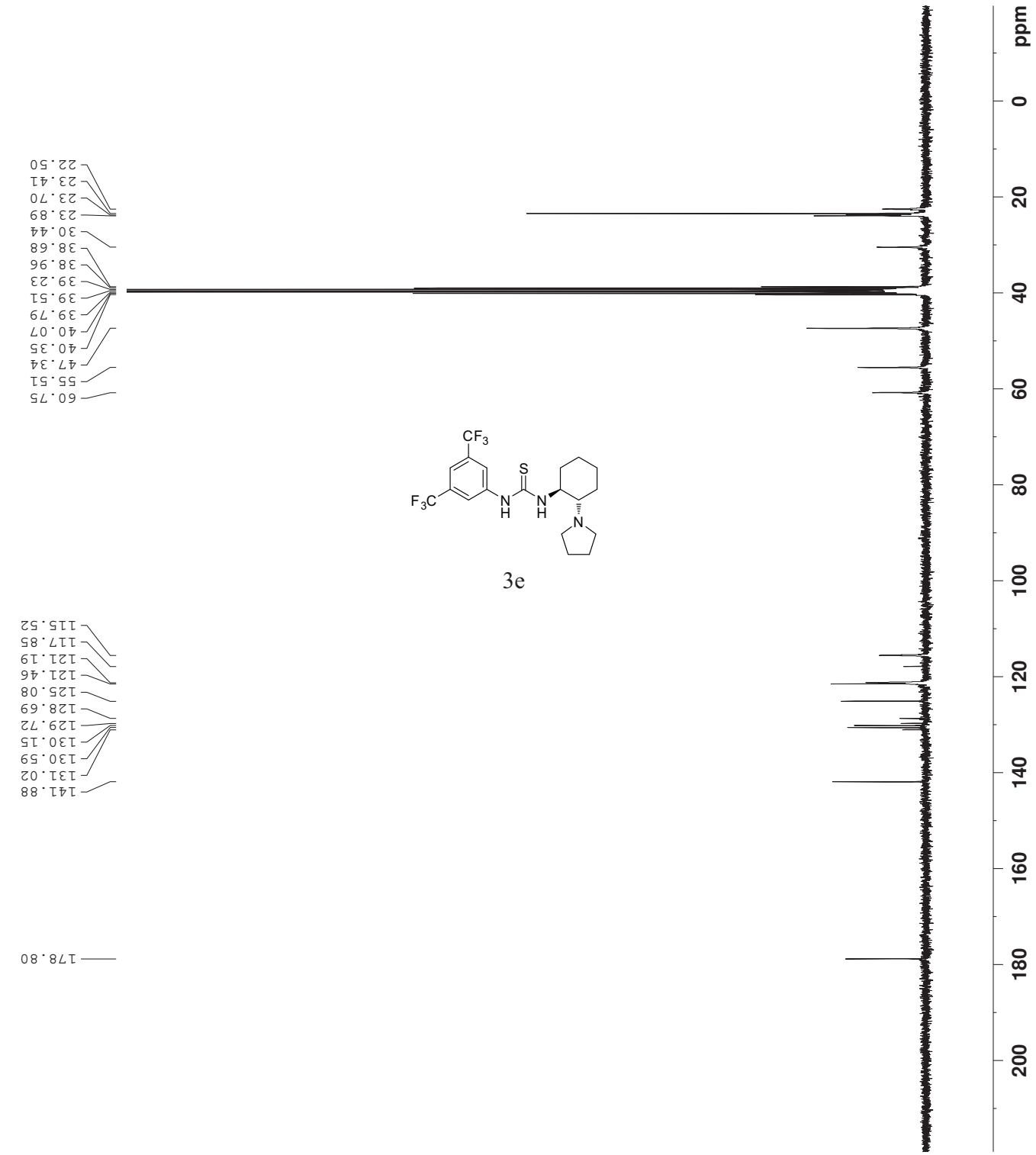


	Name	Retention Time	Area	% Area	Height
1		12.391	9562876	93.63	536781
2		17.482	650734	6.37	26001

Copies of NMR spectra

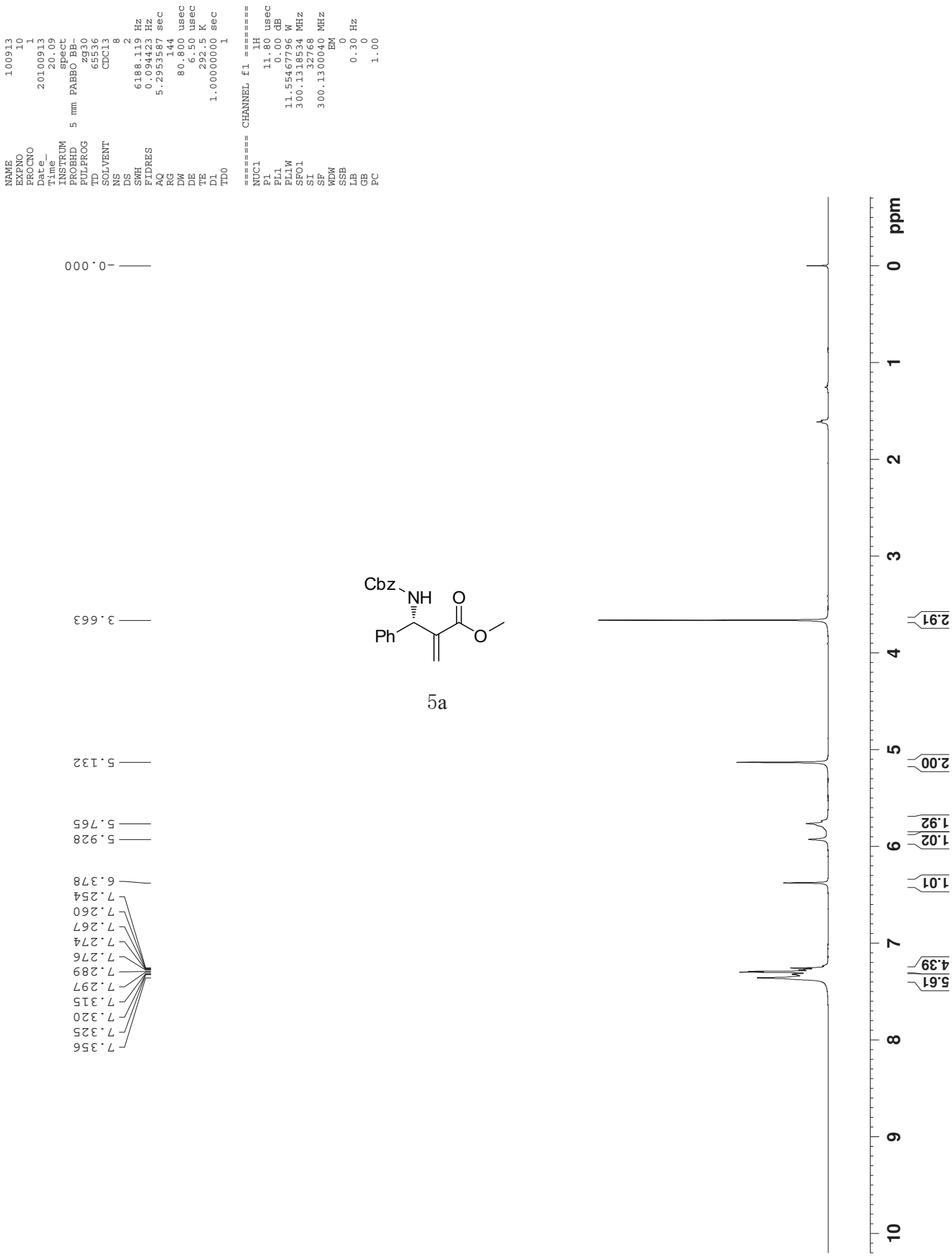


3e

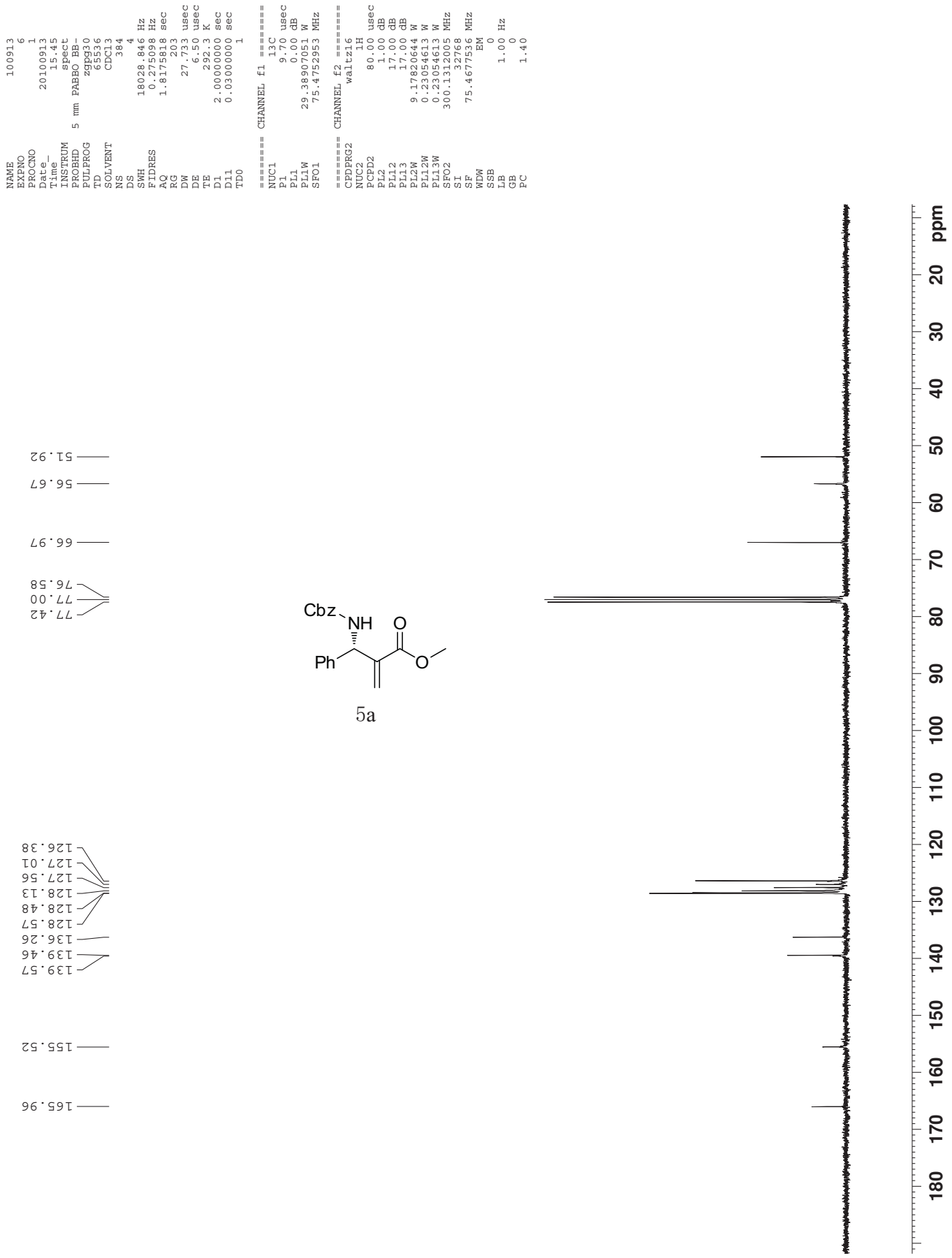

NAME 101224
EXPNO 8
PROCNO 1
Date_ 20101224
Time 23.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
22.50
23.41
23.70
23.89
30.44
38.68
38.96
39.23
39.51
39.79
40.07
40.35
47.34
55.51
60.75
115.52
117.85
121.19
121.46
125.08
128.69
129.72
130.15
130.59
131.02
141.88
178.80
ppm
0
20
40
60
80
100
120
140
160
180
200

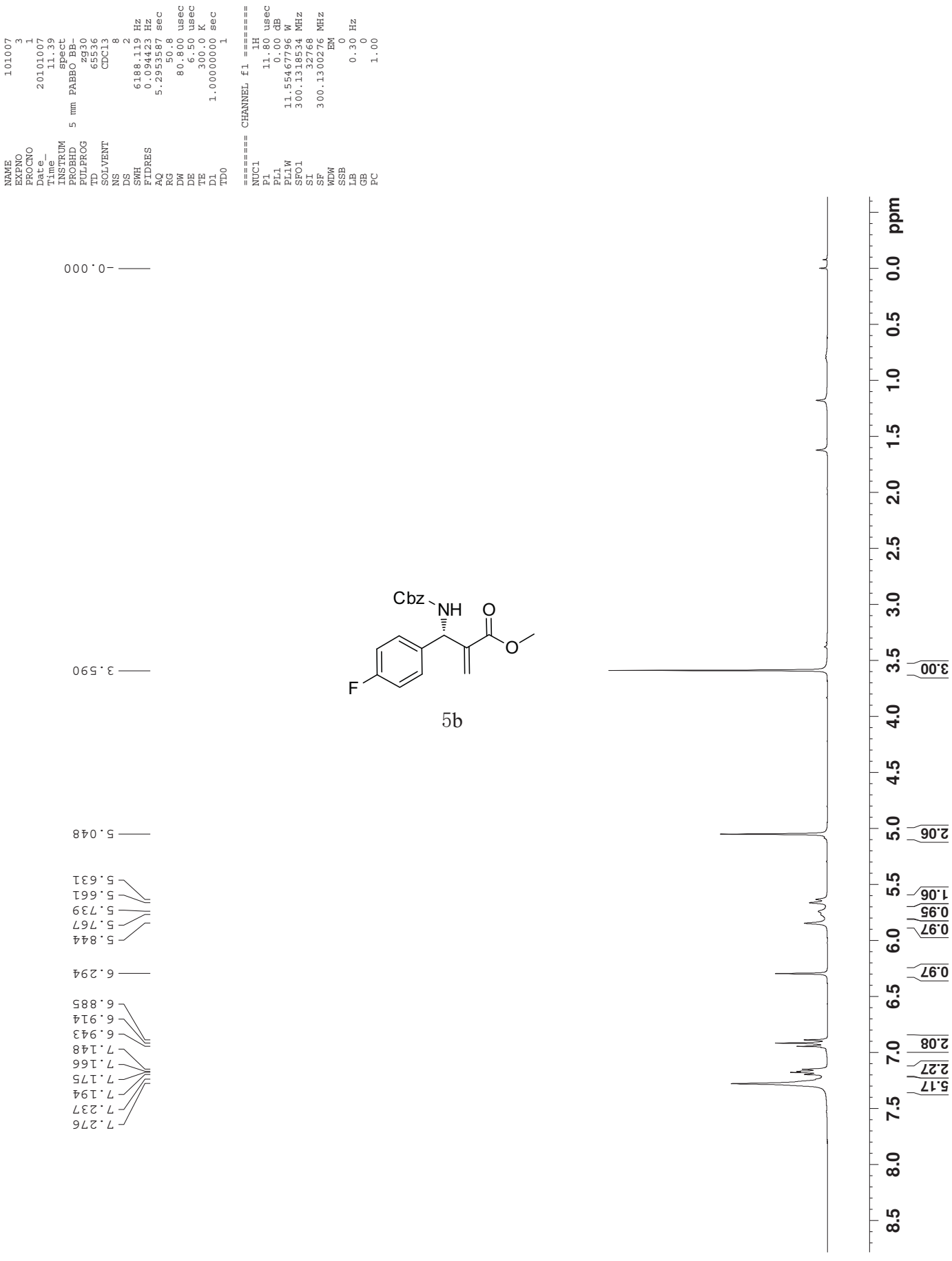
CF3
F3C
S
N
H
N
H
N

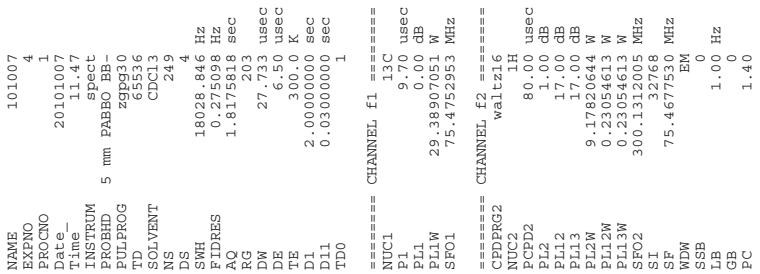
3e

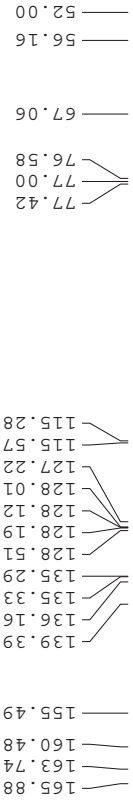

NAME 100913
EXPNO 10
PROCNO 1
Date_ 20100913
Time_ 20.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.663
5.132
5.765
5.928
6.378
7.254
7.260
7.267
7.274
7.276
7.289
7.297
7.315
7.320
7.325
7.356
Cbz
NH
O
Ph
O
2.91
2.00
1.92
1.02
1.01
4.39
5.61
ppm
0
1
2
3
4
5
6
7
8
9
10

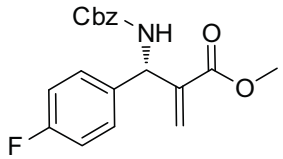
5a


NAME 100913
EXPNO 6
PROCNO 1
Date_ 20100913
Time 15.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.92
56.67
66.97
76.58
77.00
77.42
126.38
127.01
127.56
128.13
128.48
128.57
136.26
139.46
139.57
155.52
165.96
Cbz
NH
O
Ph
O
5a
ppm

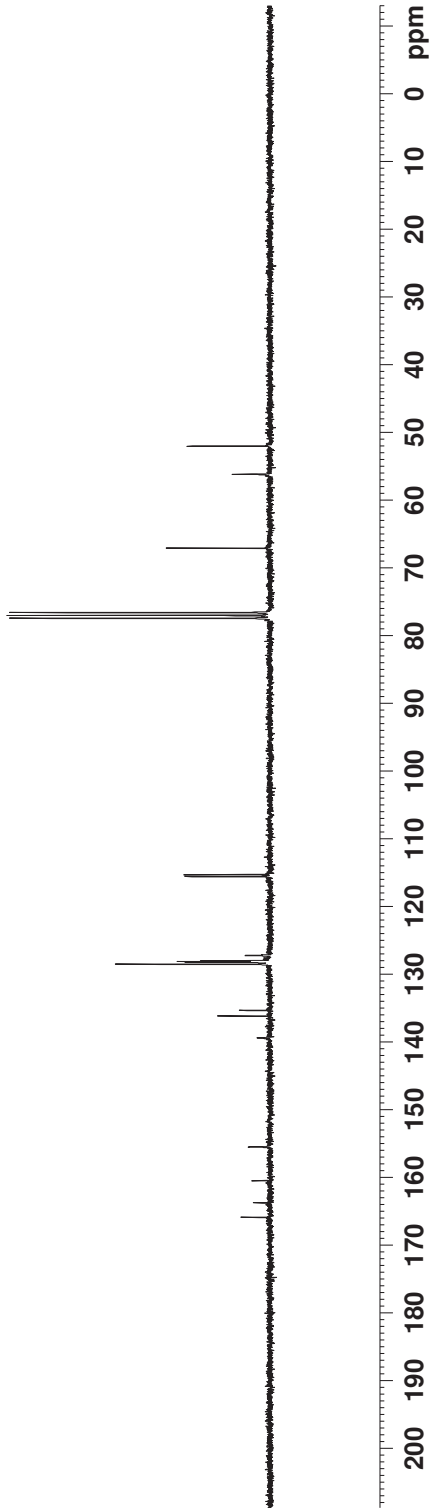

NAME 101007
EXPNO 3
PROCNO 1
Date_ 20101007
Time_ 11.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300276 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.590
5.048
5.631
5.661
5.739
5.767
5.844
6.294
6.885
6.914
6.943
7.148
7.166
7.175
7.194
7.237
7.276
Cbz
NH
O
O
F
5b
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.00
2.06
1.06
0.95
0.97
0.97
2.08
2.27
5.17

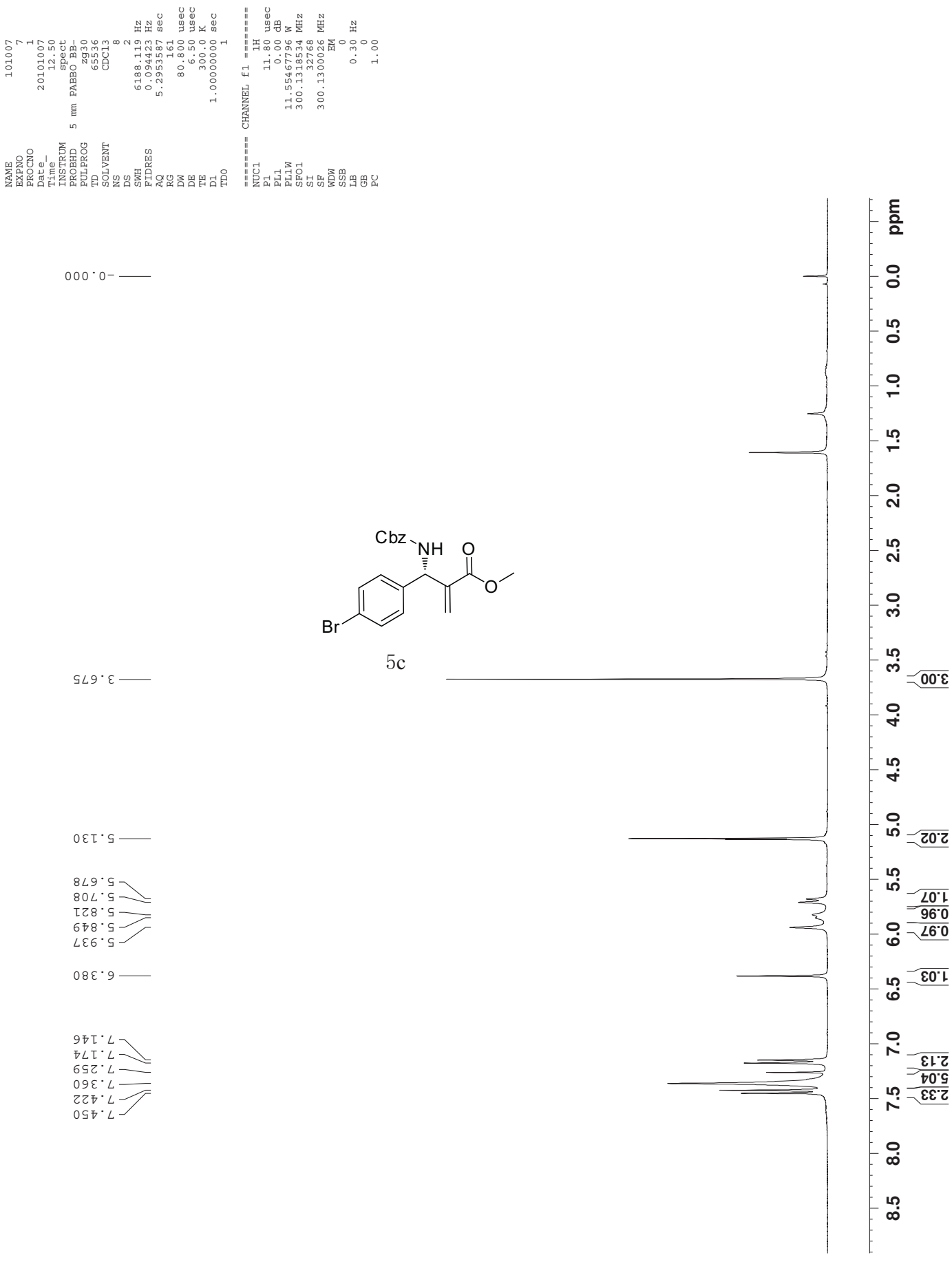

NAME 101007
EXPNO 4
PROCNO 1
Date_ 20101007
Time 11.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 249
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677530 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


52.00
56.16
67.06
76.58
77.00
77.42
115.28
115.57
127.22
128.01
128.12
128.19
128.51
135.29
135.33
136.16
139.39
155.49
160.48
163.74
165.88

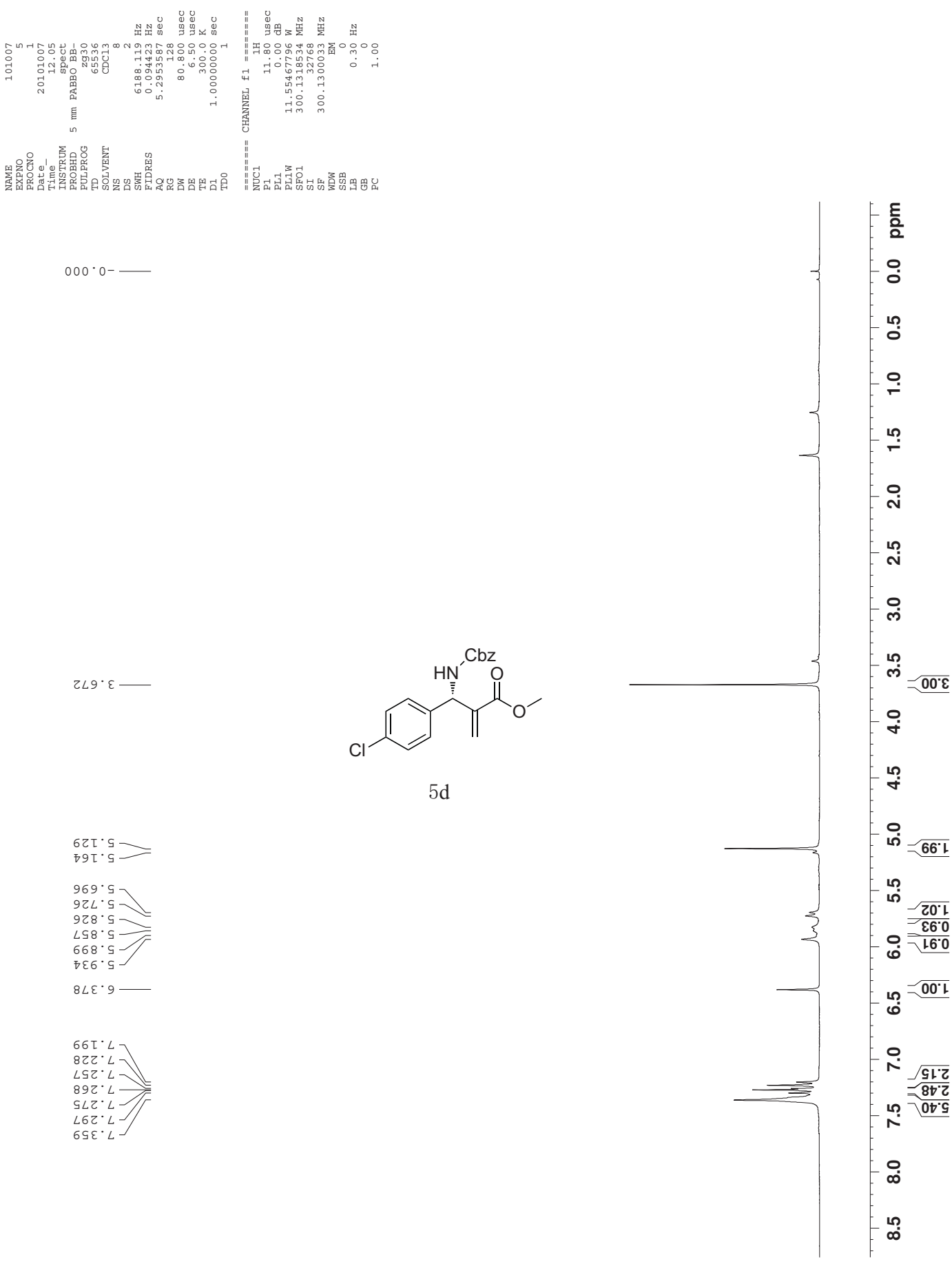

Cbz
NH
O
O
F

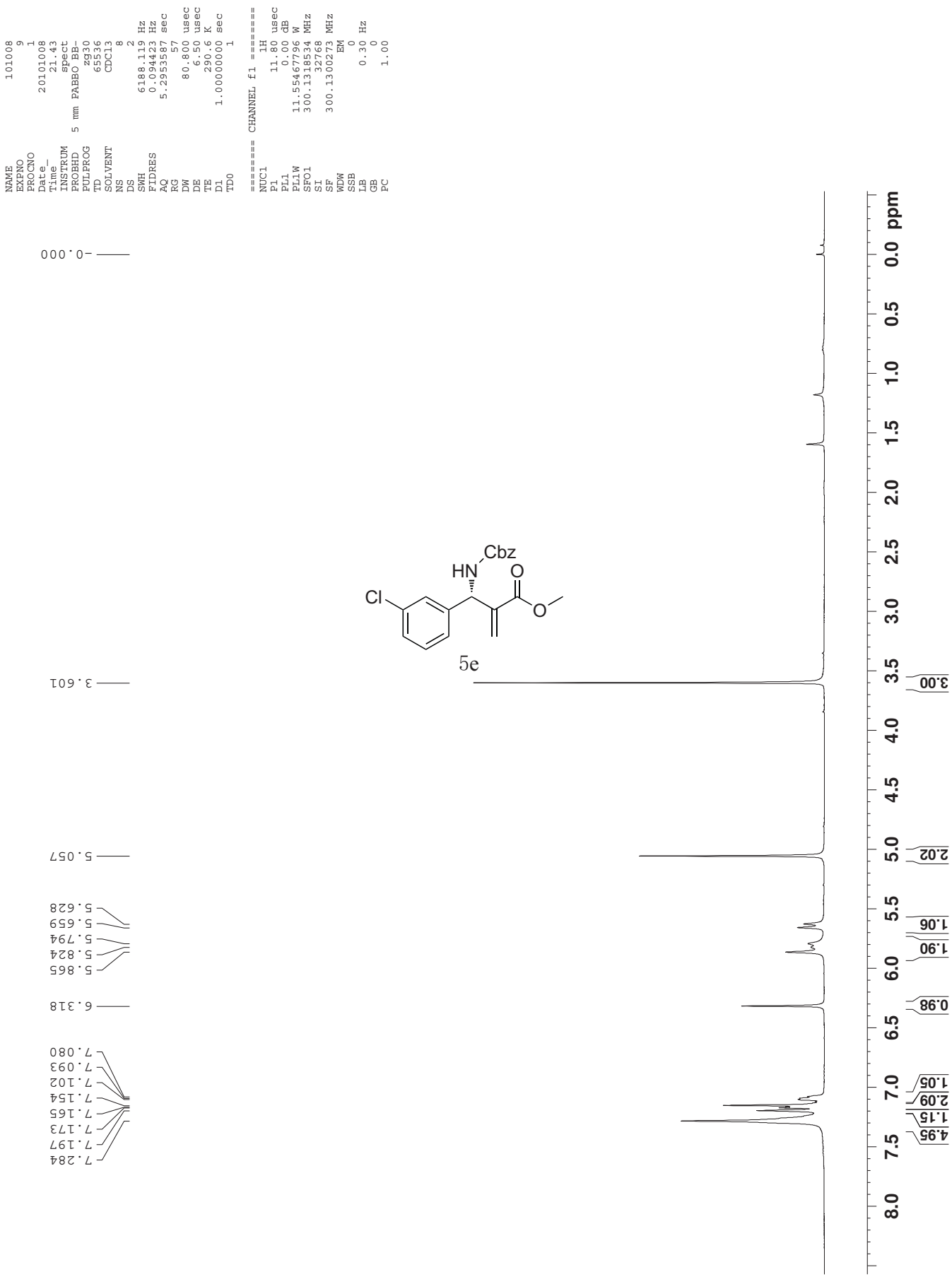
5b


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

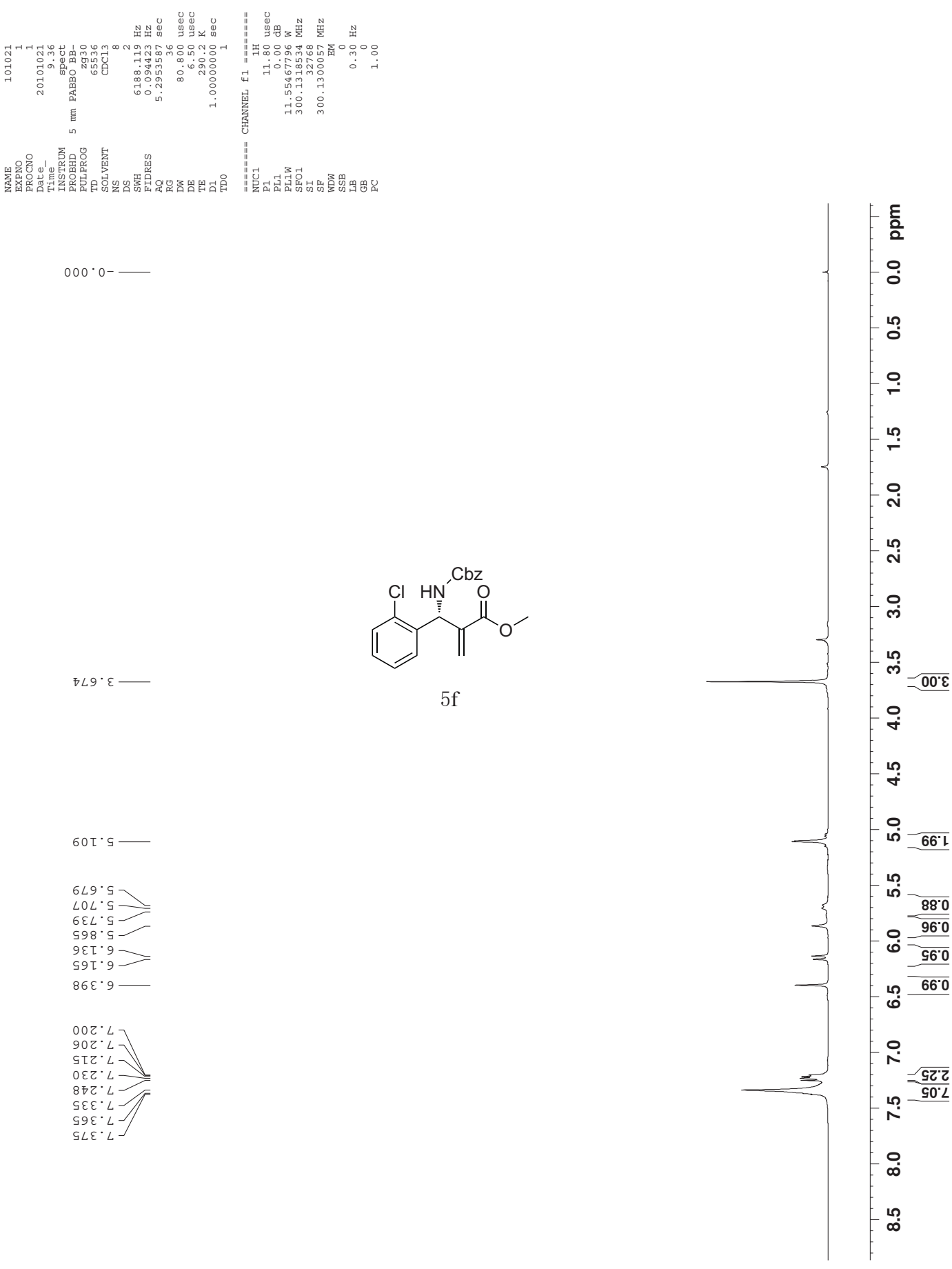


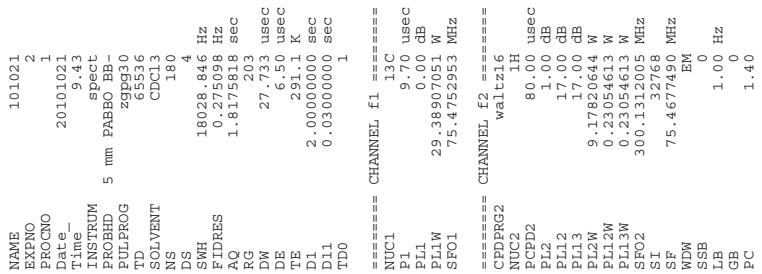

NAME 101007
EXPNO 8
PROCNO 1
Date_ 20101007
Time 12.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 627
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.10
56.41
67.16
76.59
77.02
77.44
121.58
127.72
128.09
128.27
128.57
131.70
136.16
138.71
139.11
155.56
165.84
Cbz
NH
O
O
Br
5c
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

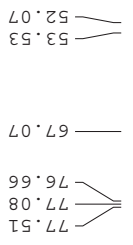

NAME 101007
EXPNO 5
PROCNO 1
Date_ 20101007
Time_ 12.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 128
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.672
5.129
5.164
5.696
5.726
5.826
5.857
5.899
5.934
6.378
7.199
7.228
7.257
7.268
7.275
7.297
7.359
Cbz
HN
O
O
Cl
5d
3.00
1.99
1.02
0.93
0.91
1.00
2.15
2.48
5.40
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

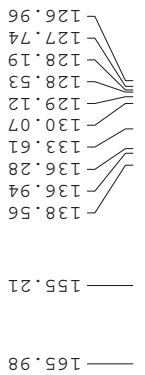


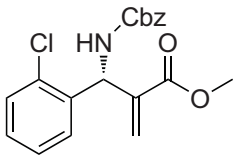

NAME 101008
EXPNO 10
PROCNO 1
Date_ 20101008
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 203
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.12
56.43
67.18
76.62
77.05
77.47
124.55
126.53
127.78
128.00
128.26
128.57
129.87
134.56
136.17
138.94
141.74
155.58
165.79
Cbz
HN
O
Cl
O
5e
0 ppm



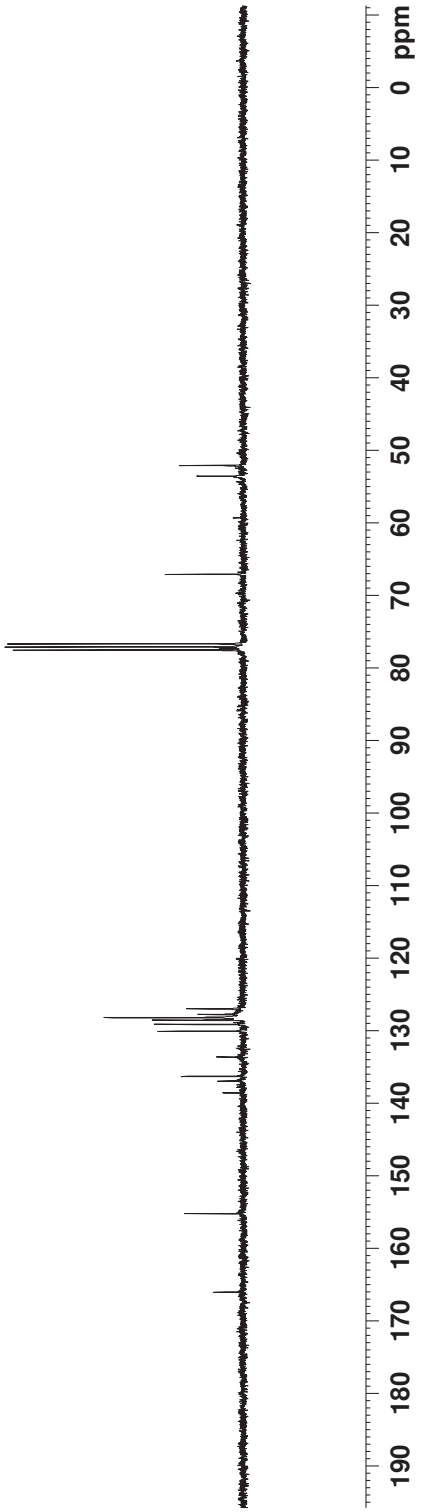


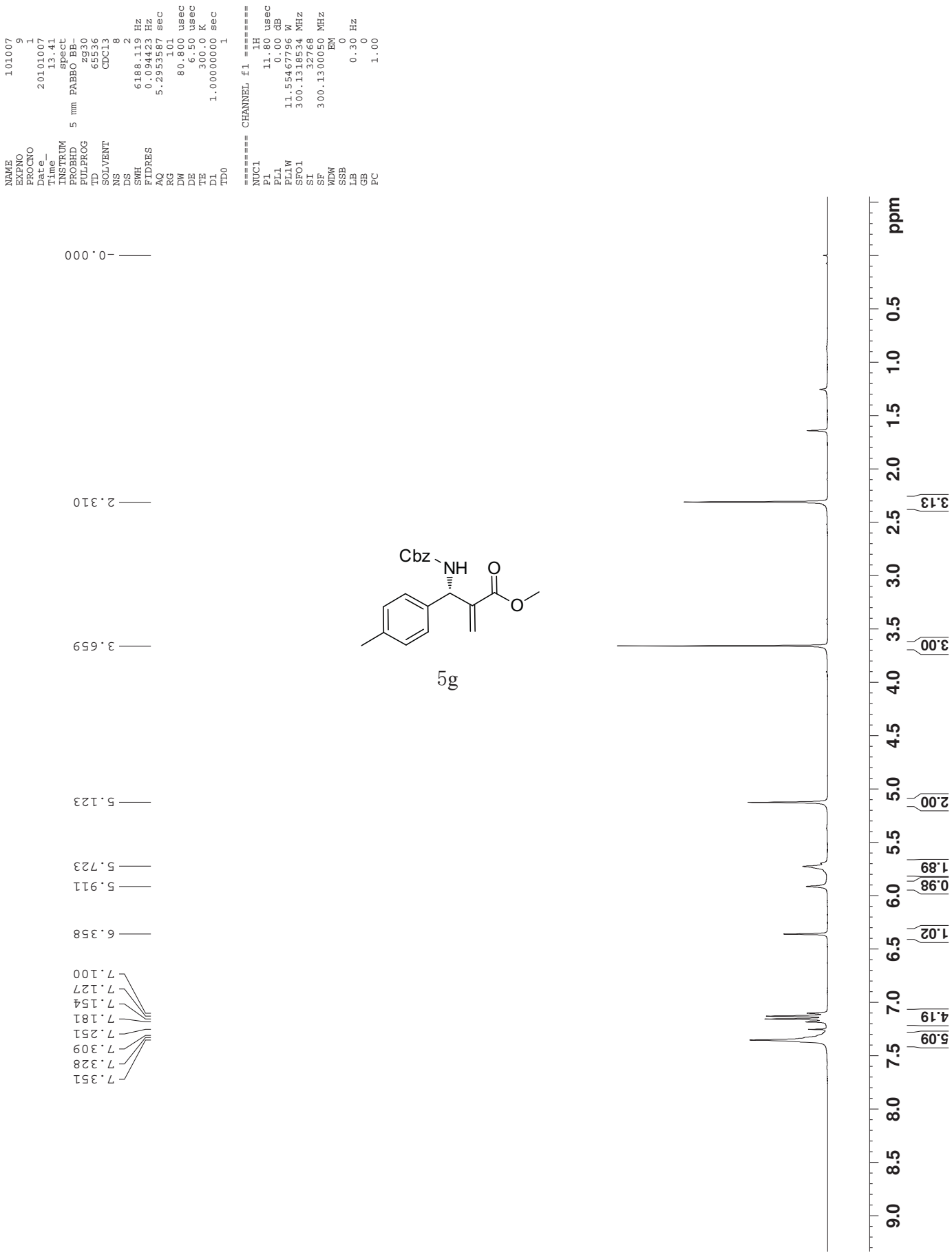




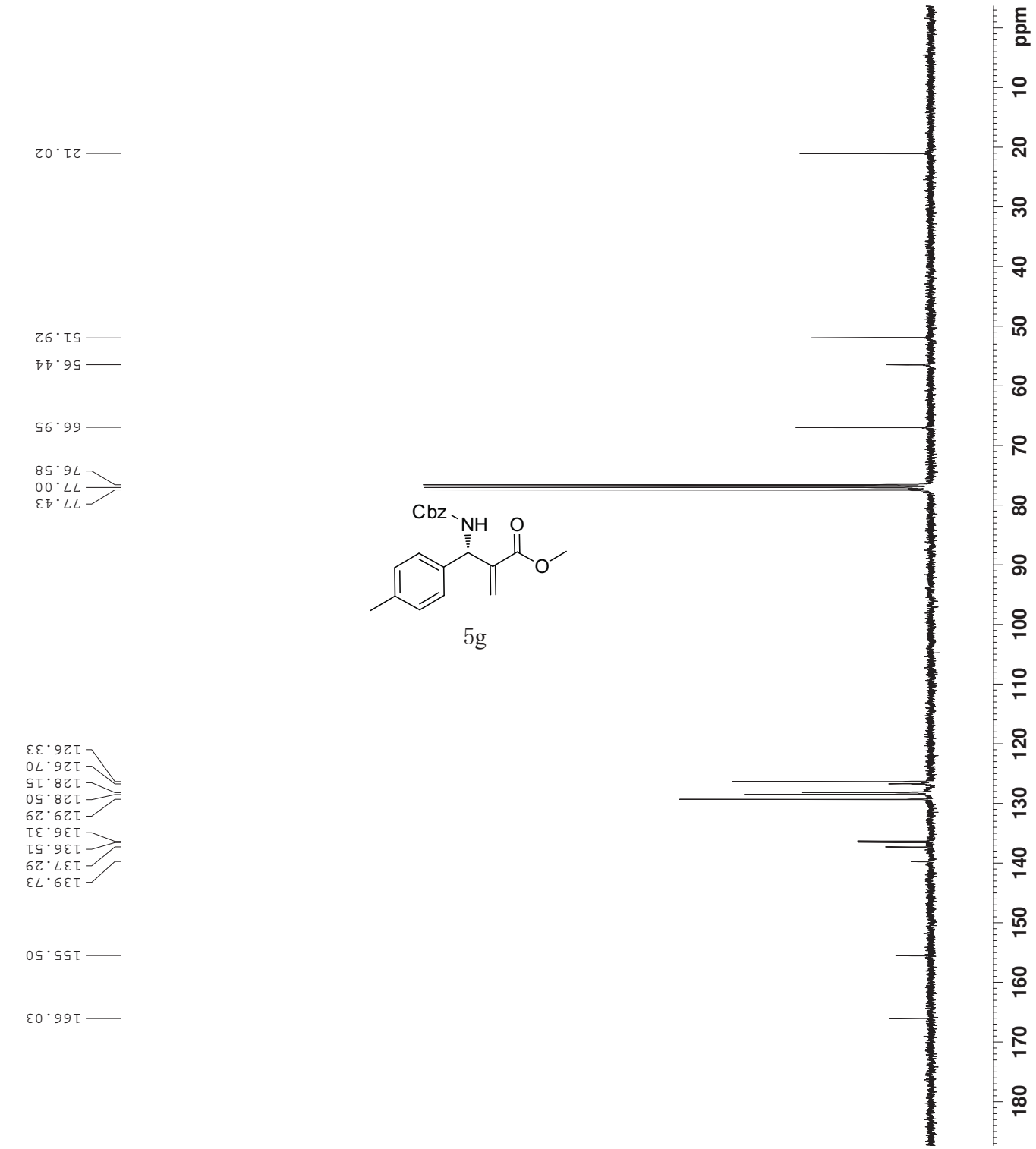


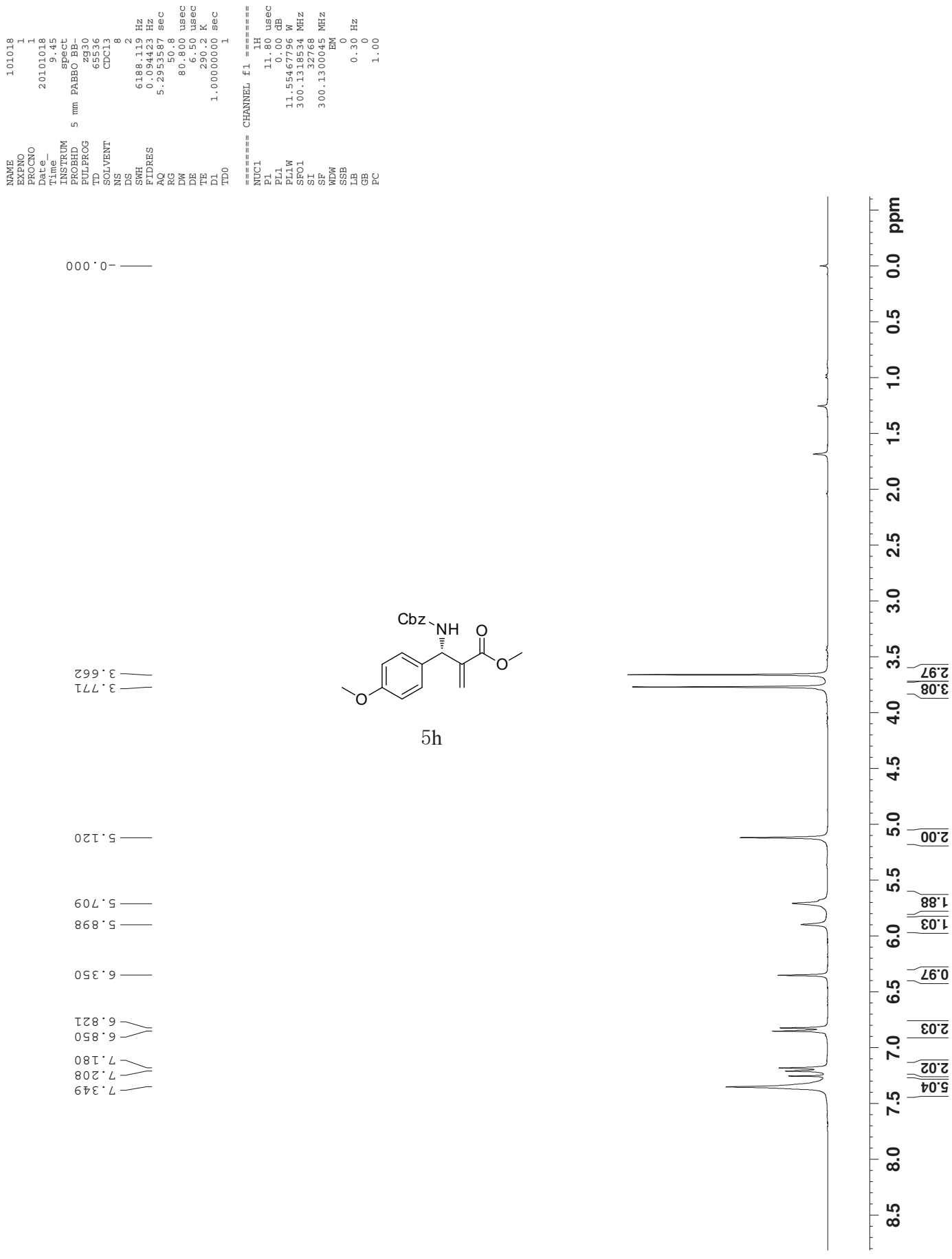
5f

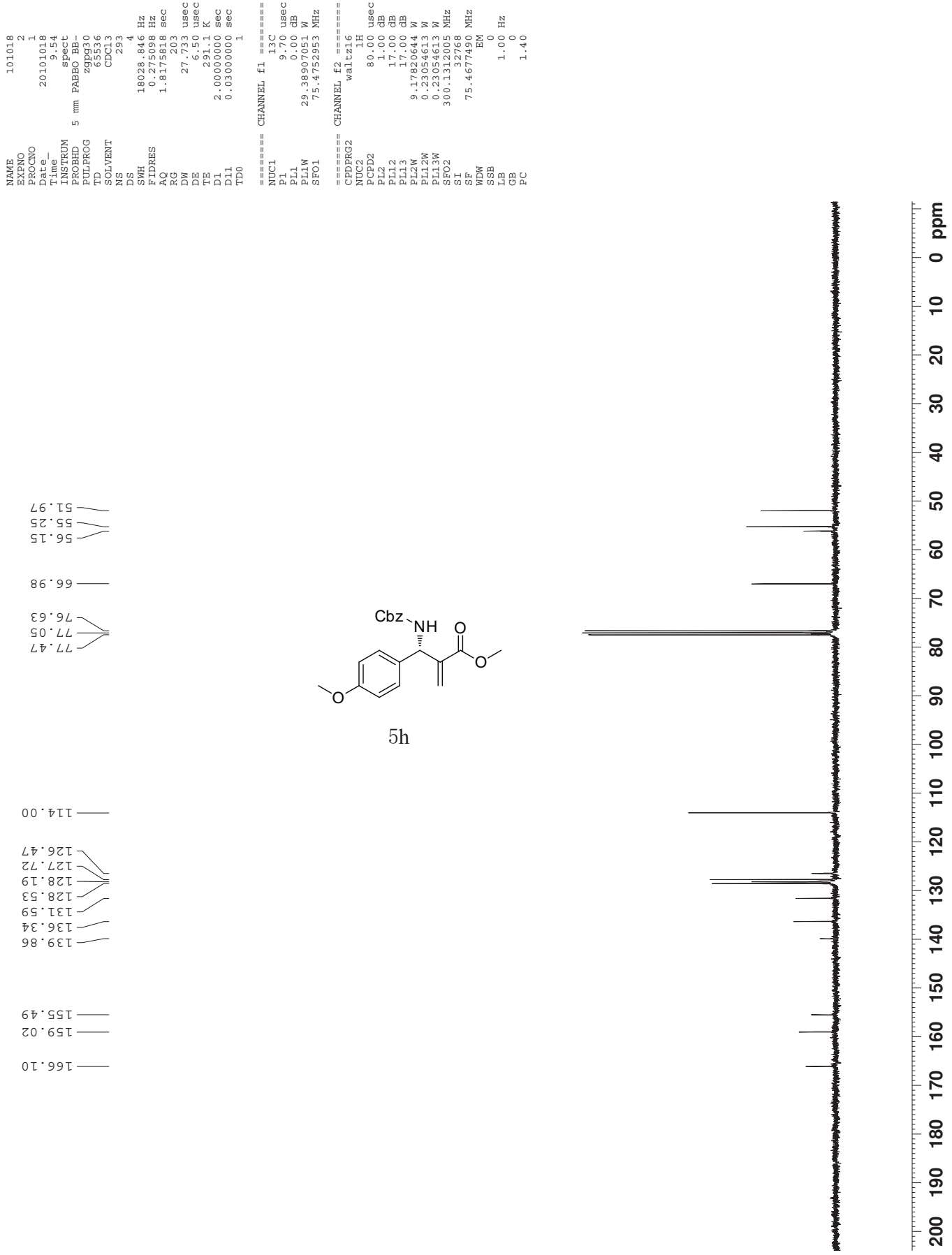


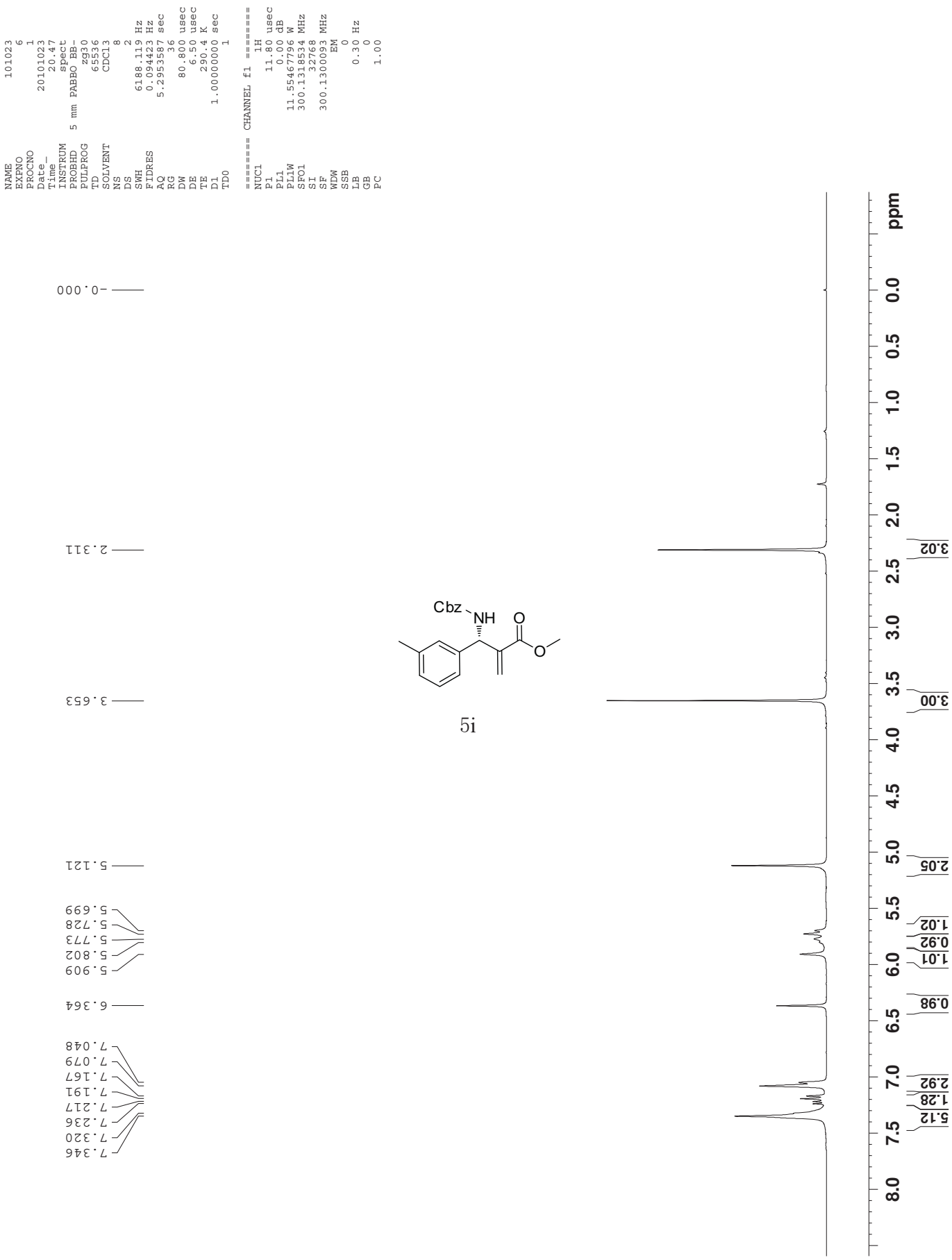

NAME 101007
EXPNO 9
PROCNO 1
Date_ 20101007
Time_ 13.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 101
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
2.310
3.659
5.123
5.723
5.911
6.358
7.100
7.127
7.154
7.181
7.251
7.309
7.328
7.351
Cbz
NH
O
O
3.13
3.00
2.00
1.89
0.98
1.02
4.19
5.09
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

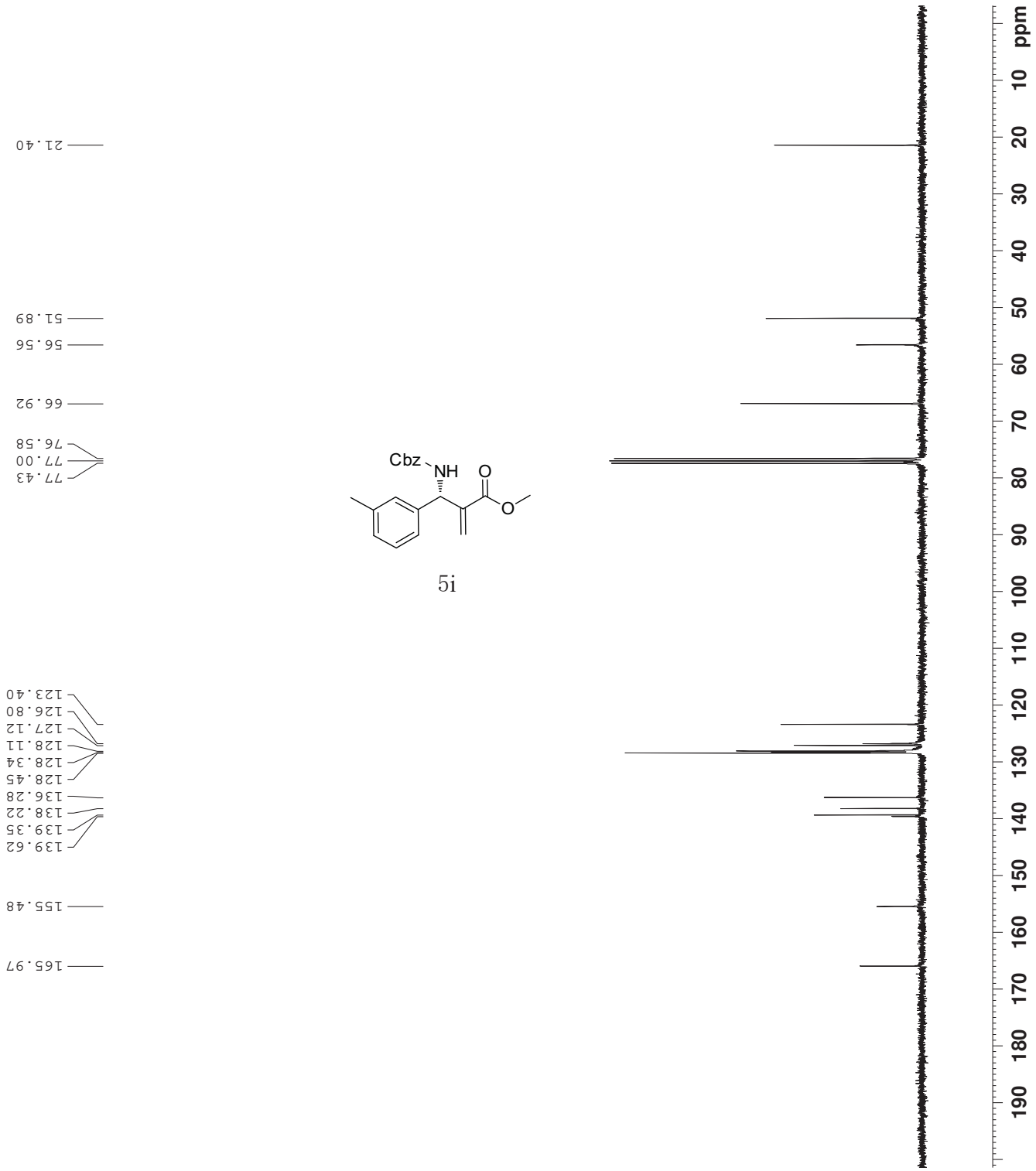
5g

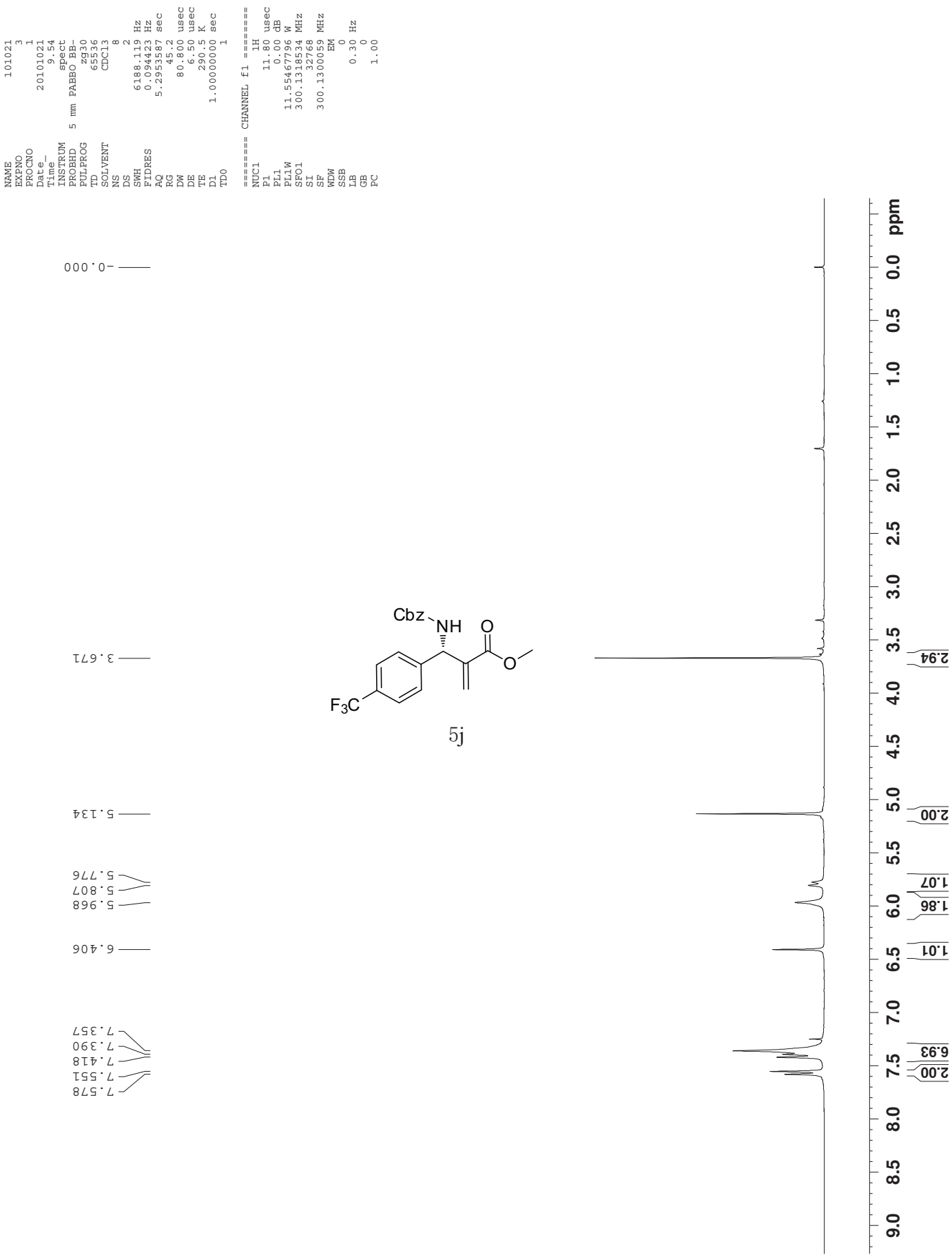

NAME 101007
EXPNO 10
PROCNO 1
Date_ 20101007
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 702
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.02
51.92
56.44
66.95
76.58
77.00
77.43
126.33
126.70
128.15
128.50
129.29
136.31
136.51
137.29
139.73
155.50
166.03
Cbz
NH
O
O
5g
ppm

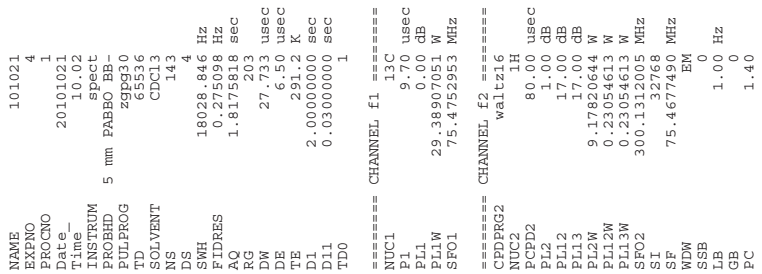

NAME 101018
EXPNO 1
PROCNO 1
Date_ 20101018
Time 9.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.771
3.662
5.120
5.709
5.898
6.350
6.821
6.850
7.180
7.208
7.349
Cbz
NH
O
O
O
5h
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
2.97
3.08
2.00
1.88
1.03
0.97
2.03
2.02
5.04

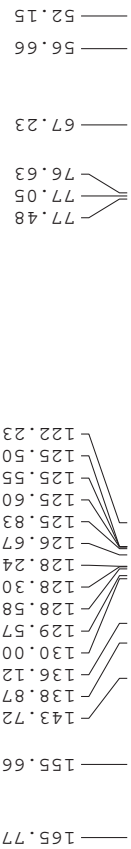

NAME 101018
EXPNO 2
PROCNO 1
Date_ 20101018
Time 9.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 293
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
NH
O
O
O
5h
51.97
55.25
56.15
66.98
76.63
77.05
77.47
114.00
126.47
127.72
128.19
128.53
131.59
136.34
139.86
155.49
159.02
166.10
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

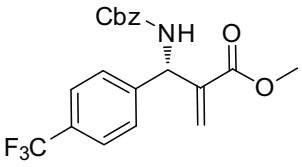

NAME 101023
EXPNO 6
PROCNO 1
Date_ 20101023
Time 20.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 290.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
O
5i
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.02
3.00
2.05
1.02
0.92
1.01
0.98
2.92
1.28
5.12
-0.000
2.311
3.653
5.121
5.699
5.728
5.773
5.802
5.909
6.364
7.048
7.079
7.167
7.191
7.217
7.236
7.320
7.346


NAME 101023
EXPNO 7
PROCNO 1
Date_ 20101023
Time 20.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 321
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81758185 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.40
51.89
56.56
66.92
76.58
77.00
77.43
123.40
126.80
127.12
128.11
128.34
128.45
136.28
138.22
139.35
139.62
155.48
165.97
Cbz
NH
O
O
5i
ppm

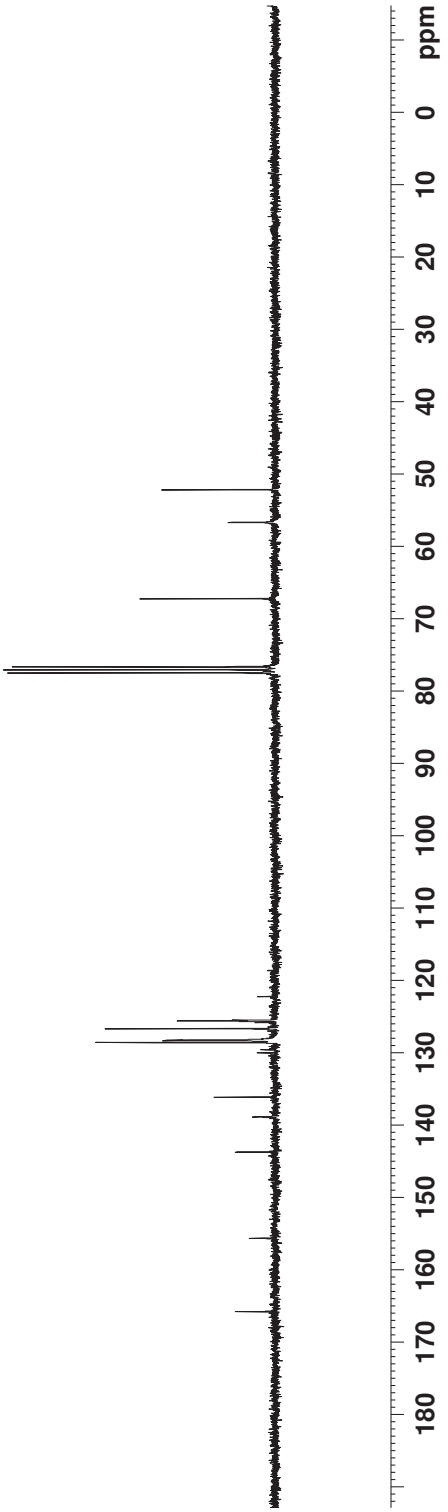


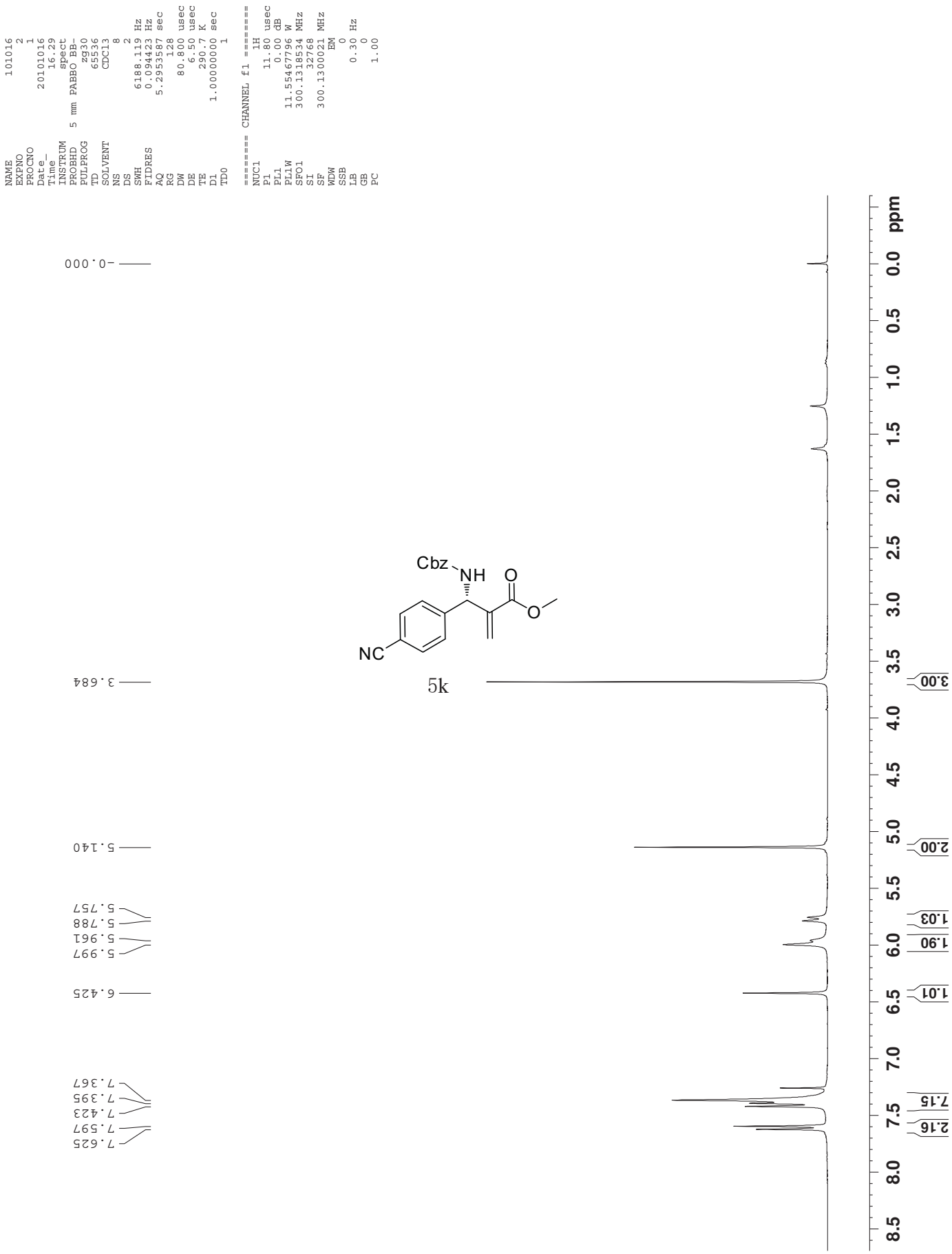

NAME 101021
EXPNO 4
PROCNO 1
Date_ 20101021
Time 10.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81755818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

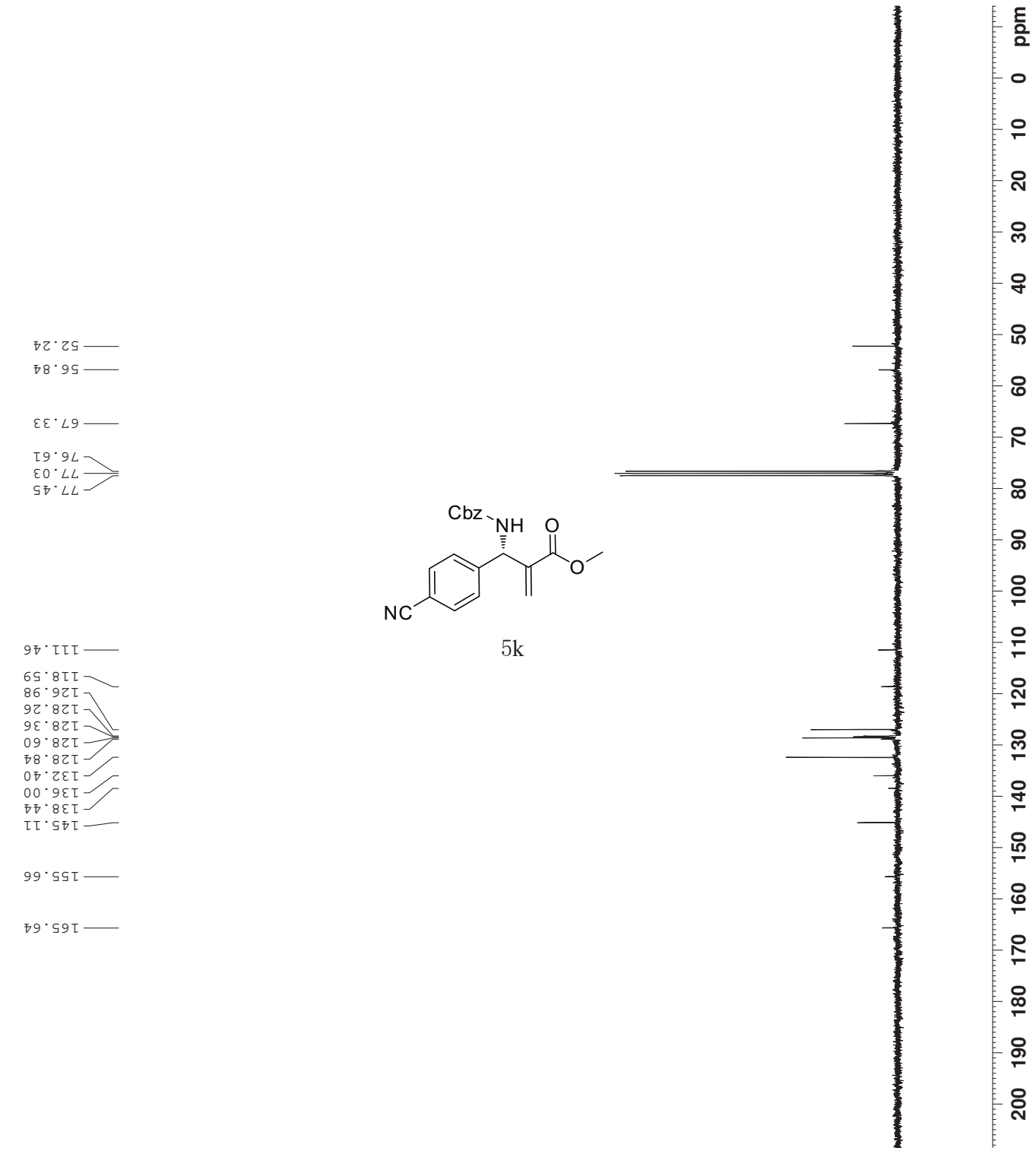

52.15
56.66
67.23
76.63
77.05
77.48
122.23
125.50
125.55
125.60
125.83
126.67
128.24
128.30
128.58
129.57
130.00
136.12
138.87
143.72
155.66
165.77

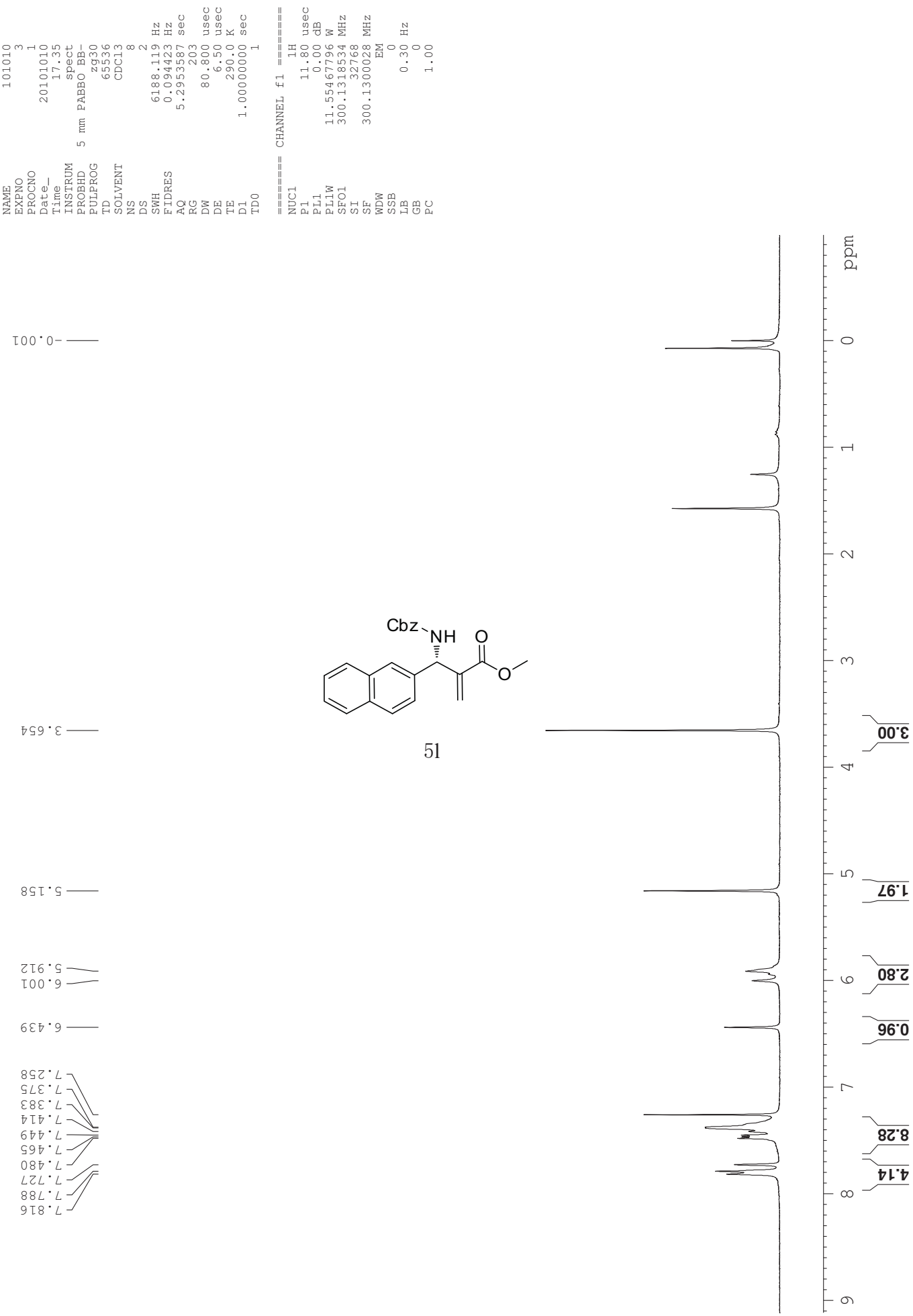

Cbz
NH
O
O
F3C

5j

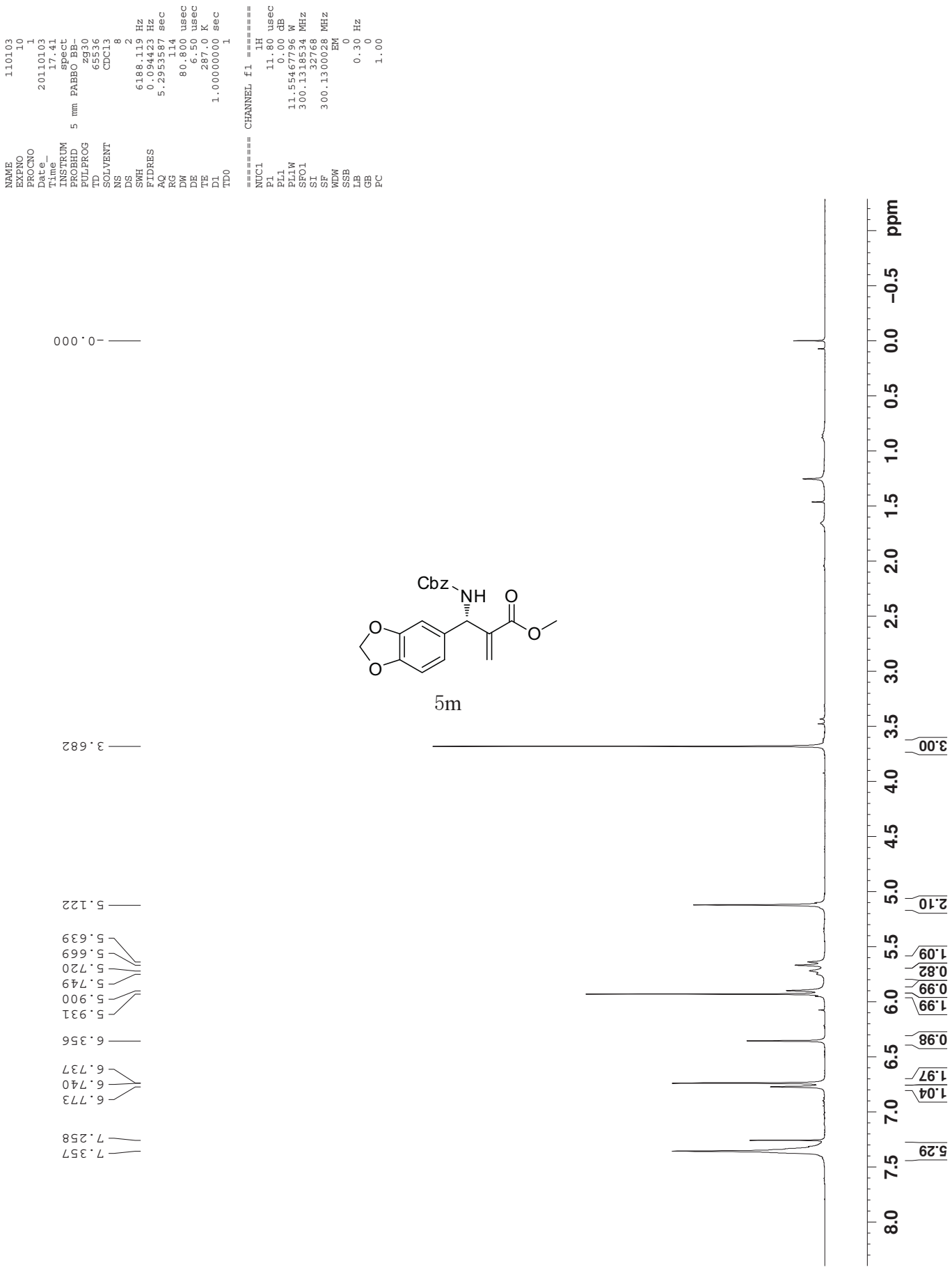

ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

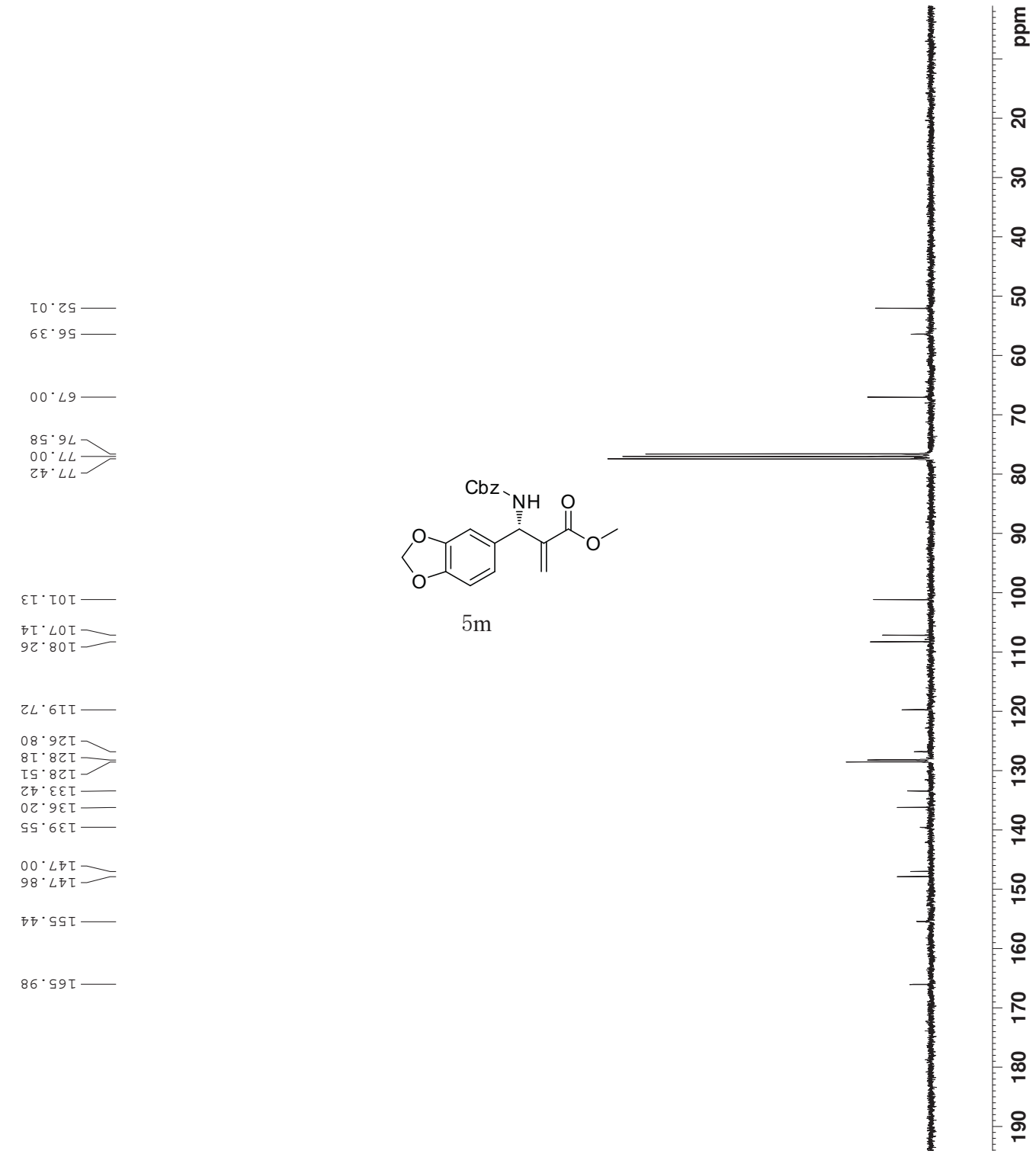

NAME 101016
EXPNO 2
PROCNO 1
Date_ 20101016
Time_ 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.29533587 sec
RG 128
DW 80.800 usec
DE 6.50 usec
TE 290.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
O
NC
5k
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.00
2.00
1.03
1.90
1.01
7.15
2.16
3.684
5.140
5.757
5.788
5.961
5.997
6.425
7.367
7.395
7.423
7.597
7.625

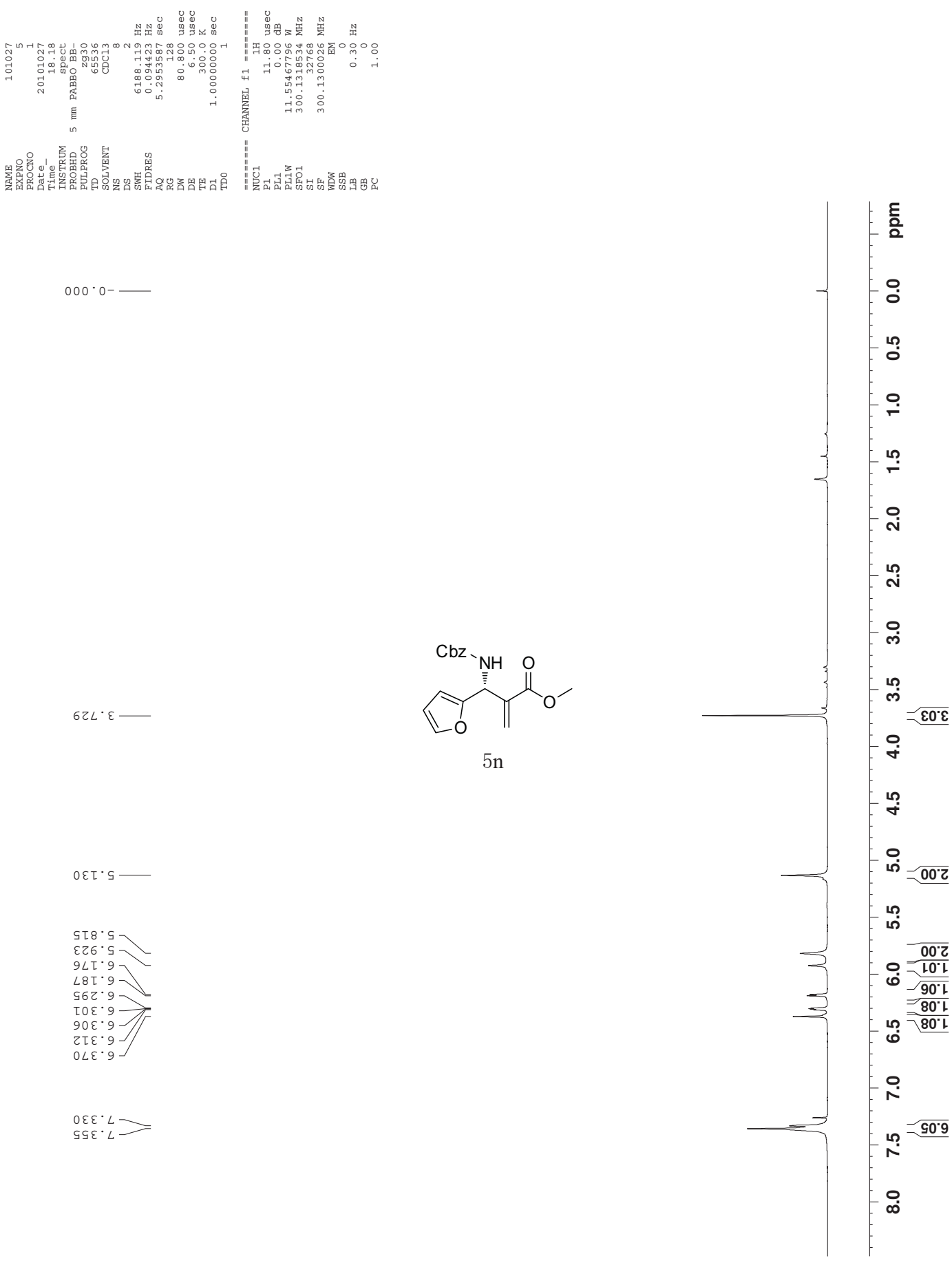

NAME 101016
EXPNO 3
PROCNO 1
Date_ 20101016
Time 16.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 323
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.24
56.84
67.33
76.61
77.03
77.45
111.46
118.59
126.98
128.26
128.36
128.60
128.84
132.40
136.00
138.44
145.11
155.66
165.64
Cbz
NH
O
O
NC
5k
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

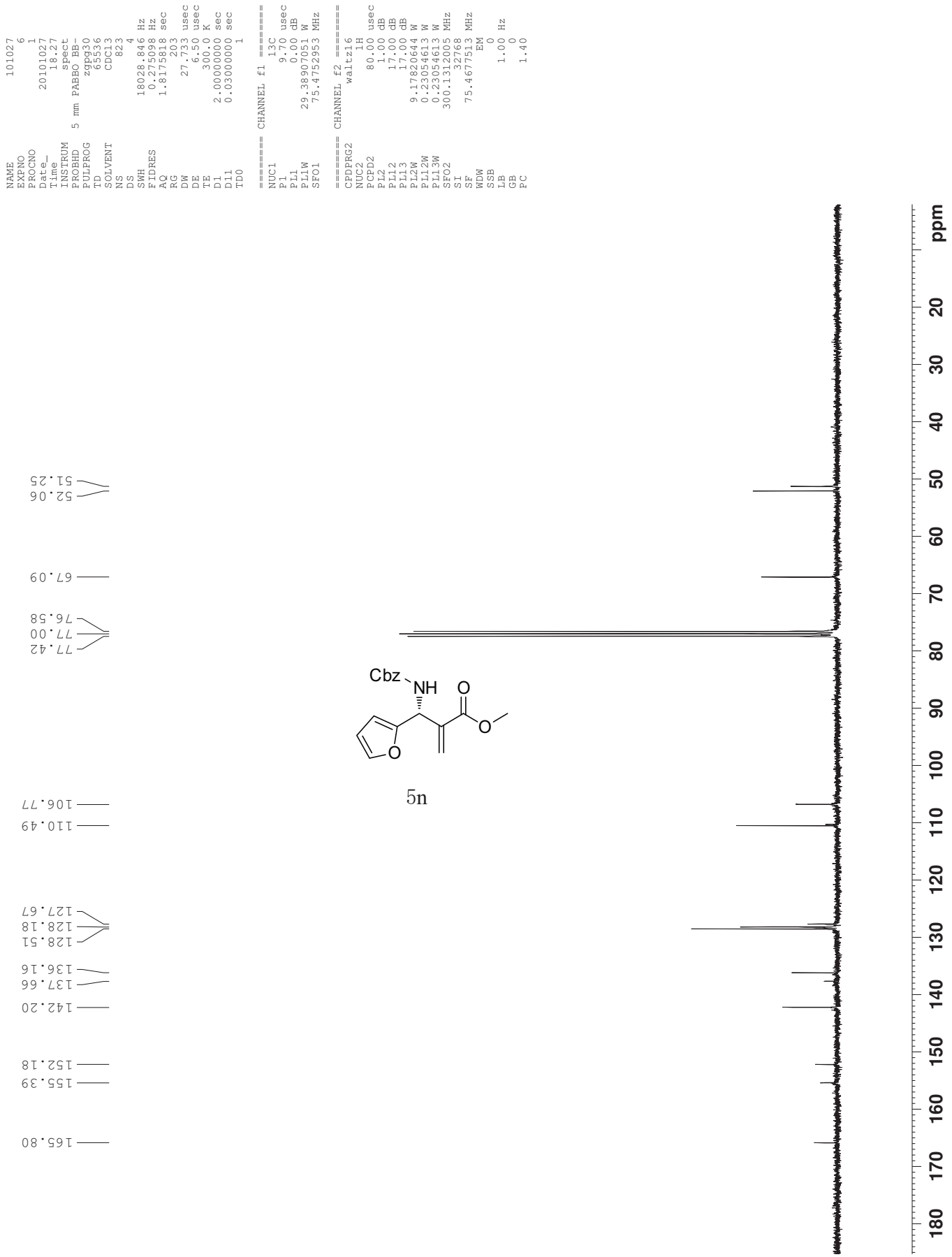

NAME 101010
EXPNO 3
PROCNO 1
Date_ 20101010
Time 17.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 290.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
O
51
ppm
3.00
1.97
2.80
0.96
8.28
4.14
-0.001
3.654
5.158
6.001
5.912
6.439
7.258
7.375
7.383
7.414
7.449
7.465
7.480
7.727
7.788
7.816

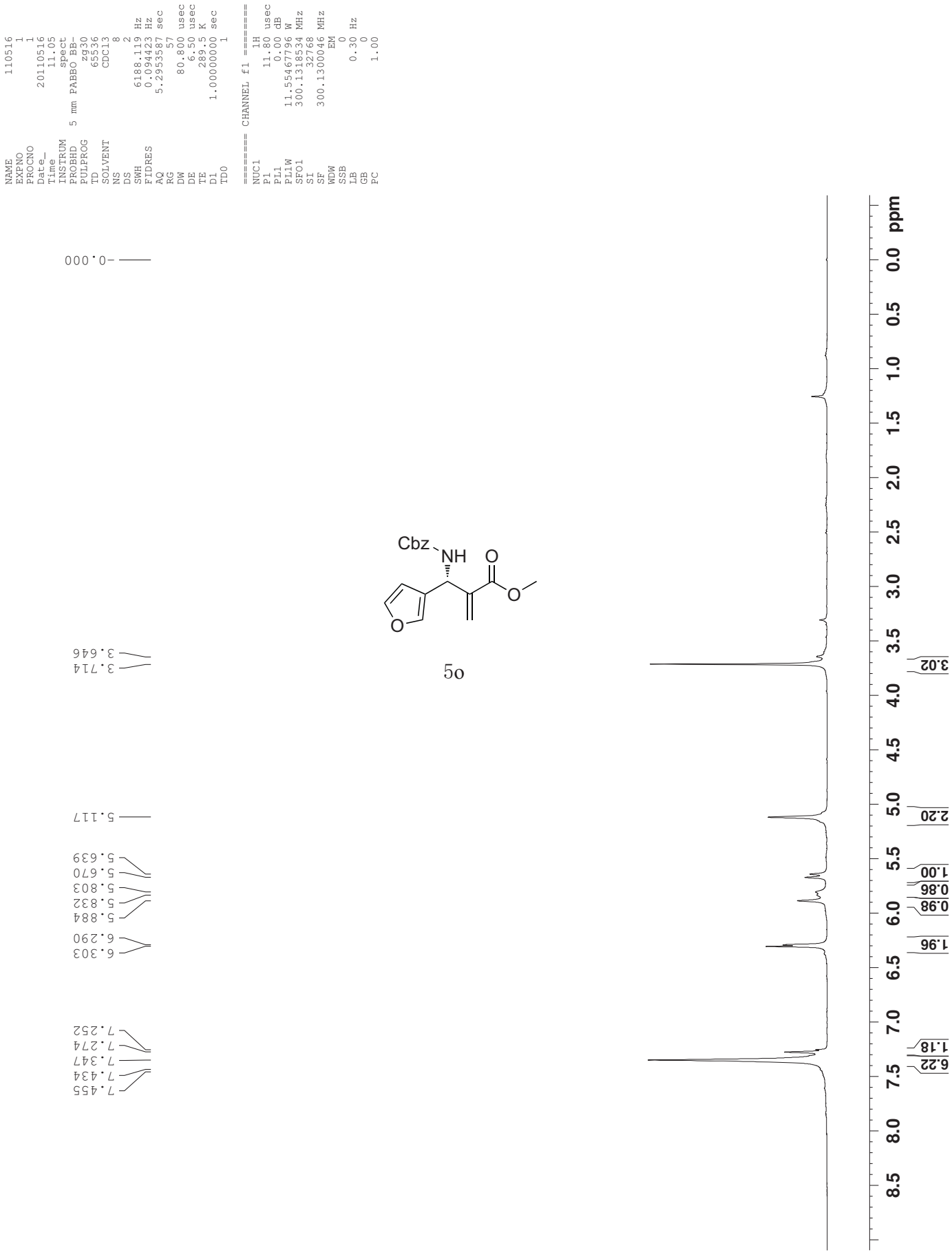

NAME 101011
EXPNO 2
PROCNO 1
Date_ 20101011
Time 10.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 797
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.03
56.82
67.11
76.60
77.02
77.45
124.68
125.11
126.09
126.27
127.31
127.61
128.03
128.24
128.48
128.56
132.78
133.23
136.28
136.93
139.57
155.62
166.04
Cbz
NH
O
O
51
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

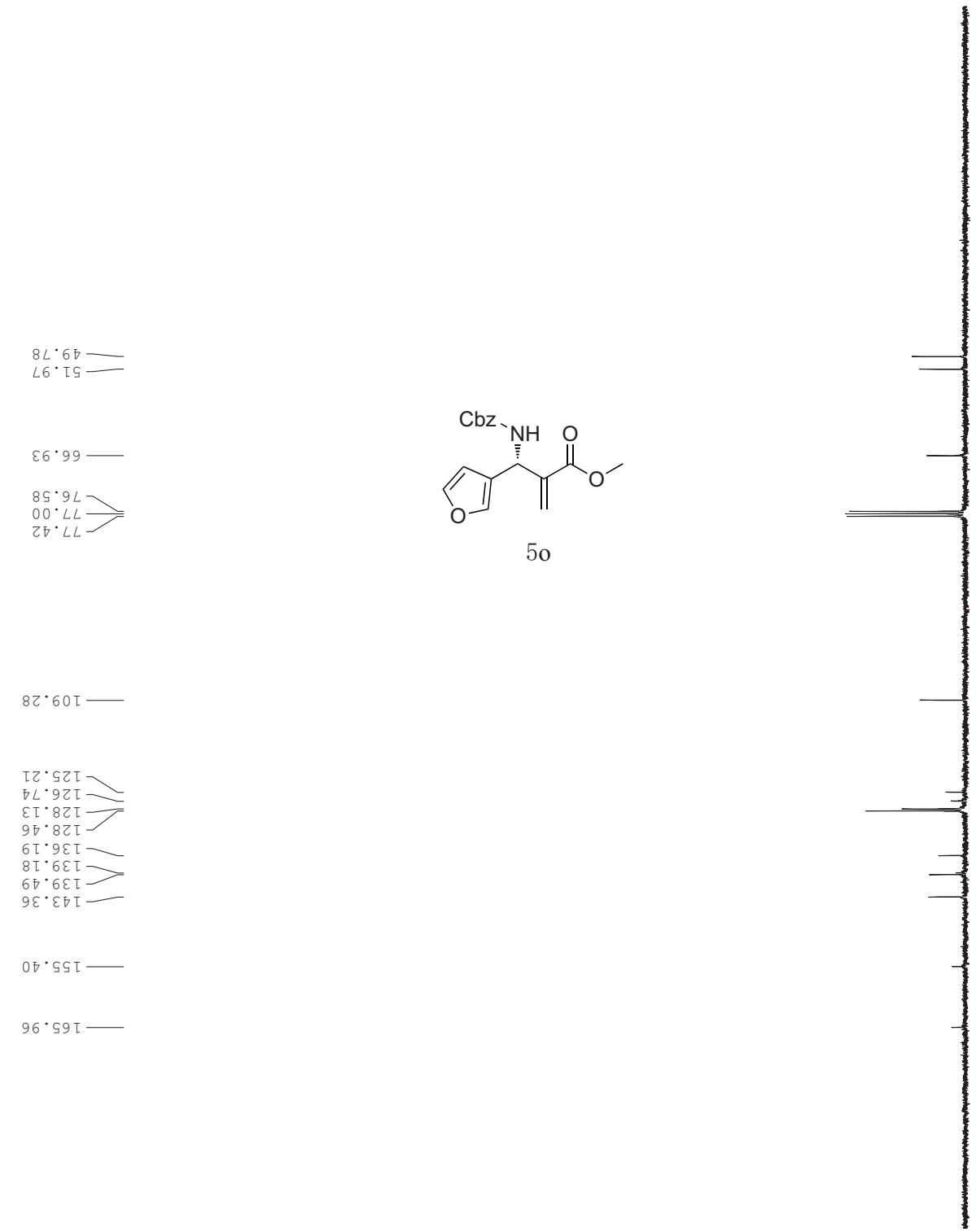

NAME 110103
EXPNO 10
PROCNO 1
Date_ 20110103
Time_ 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 114
DW 80.800 usec
DE 6.50 usec
TE 287.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
O
O
O
5m
ppm
3.00
2.10
1.09
0.82
0.99
1.99
0.98
1.97
1.04
5.29

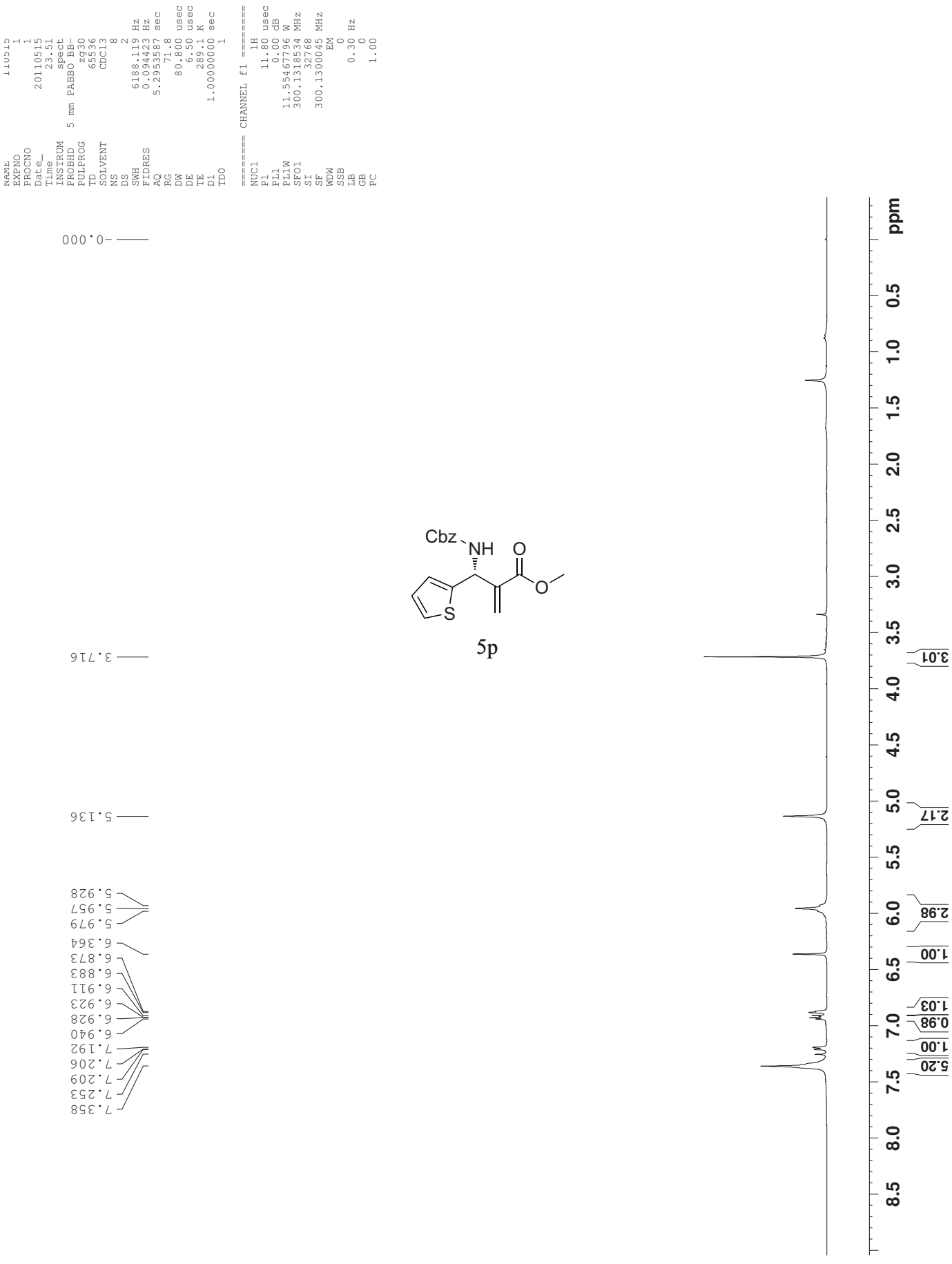

NAME 110103
EXPNO 12
PROCNO 1
Date_ 20110103
Time 17.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 251
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677811 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.01
56.39
67.00
76.58
77.00
77.42
101.13
107.14
108.26
119.72
126.80
128.18
128.51
133.42
136.20
139.55
147.00
147.86
155.44
165.98
Cbz
NH
O
O
O
O
5m
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20


NAME 101027
EXPNO 5
PROCNO 1
Date_ 20101027
Time 18.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 128
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.729
5.130
5.815
5.923
6.176
6.187
6.295
6.301
6.306
6.312
6.370
7.330
7.355
Cbz
NH
O
O
O
5n
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.03
2.00
2.00
1.01
1.06
1.08
1.08
6.05

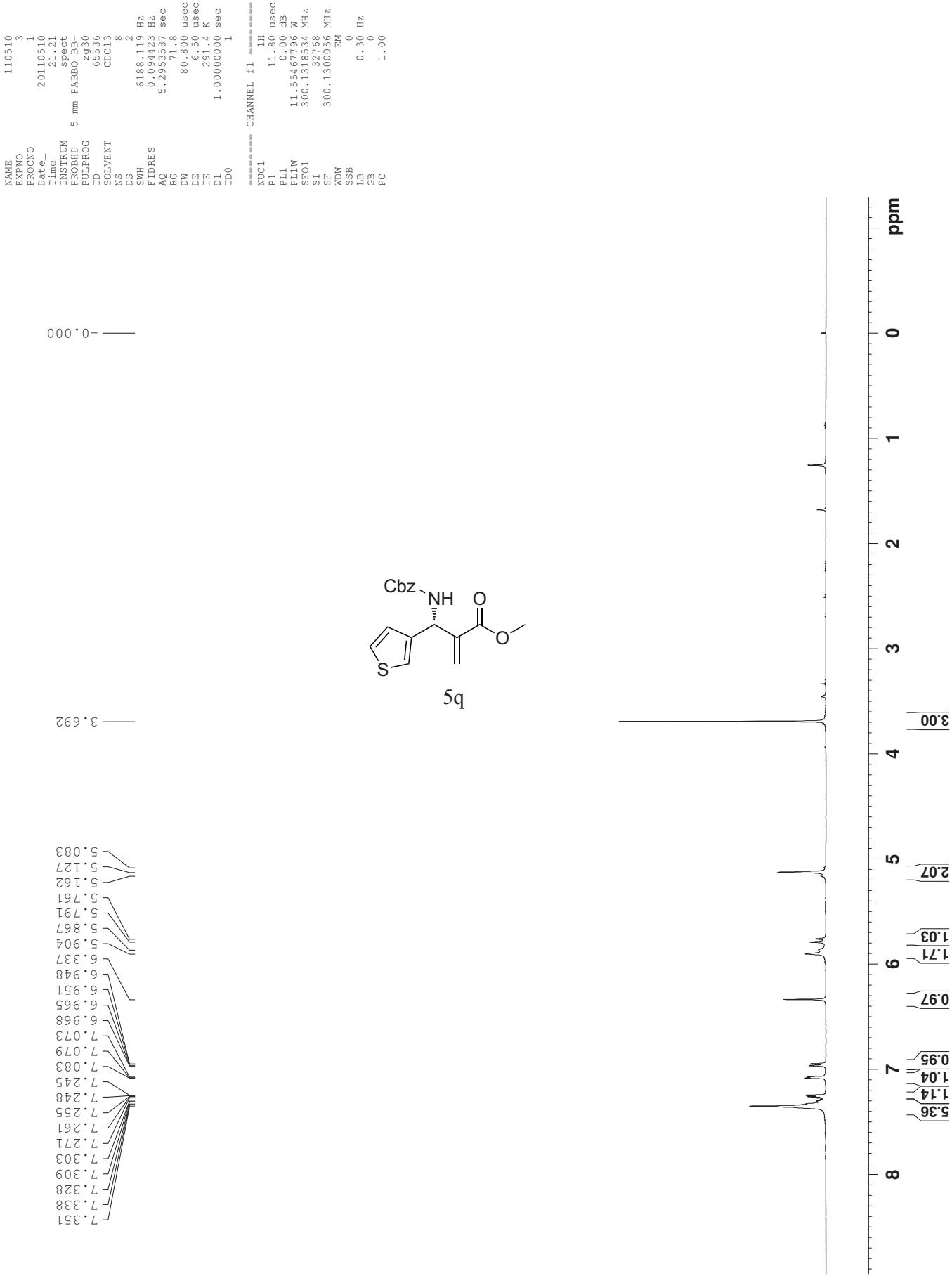


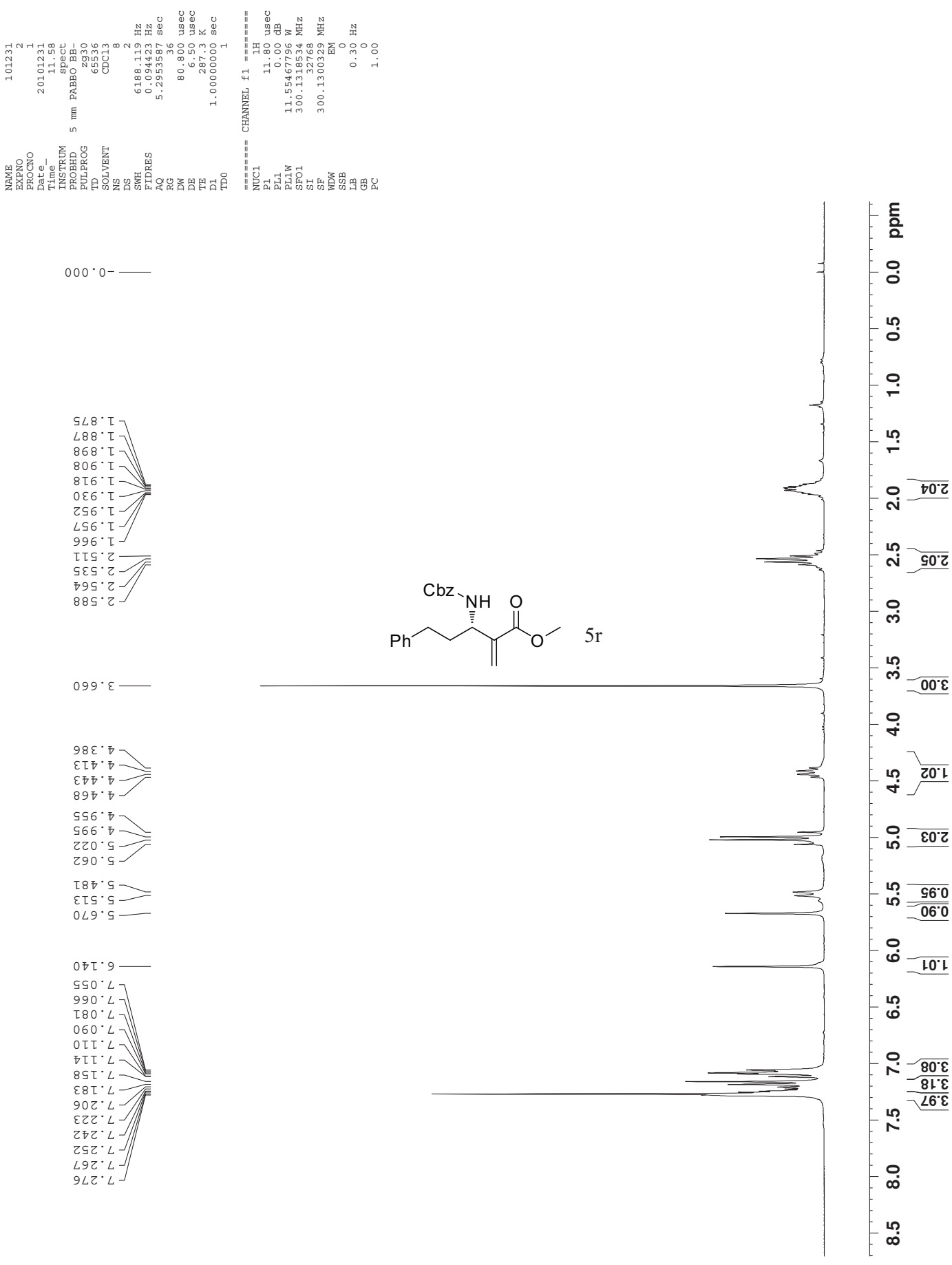


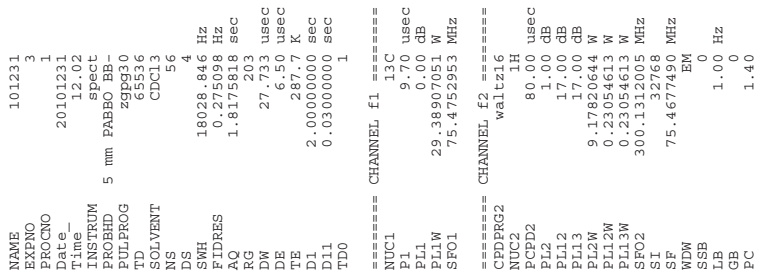

NAME 110516
EXPNO 2
PROCNO 1
Date_ 20110516
Time 11.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 170
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
NH
O
O
O
5o
49.78
51.97
66.93
76.58
77.00
77.42
109.28
125.21
126.74
128.13
128.46
136.19
139.18
139.49
143.36
155.40
165.96
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190


Cbz
NH
O
O
S
5p
ppm
3.01
2.17
2.98
1.00
1.03
0.98
1.00
5.20

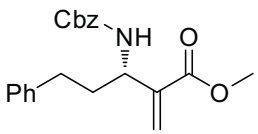

NAME 110515
EXPNO 2
PROCNO 1
Date_ 20110515
Time 23.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 167
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 289.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677526 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
52.09
53.25
67.08
76.58
77.00
77.42
124.64
124.96
127.04
127.35
128.18
128.51
136.15
139.11
143.85
155.33
165.80
Cbz
NH
O
O
S
5p
ppm


NAME 110510
EXPNO 3
PROCNO 1
Date_ 20110510
Time 21.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 71.8
DW 80.800 usec
DE 6.50 usec
TE 291.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
O
S
5q
ppm
0
1
2
3
4
5
6
7
8
3.00
2.07
1.03
1.71
0.97
0.95
1.04
1.14
5.36

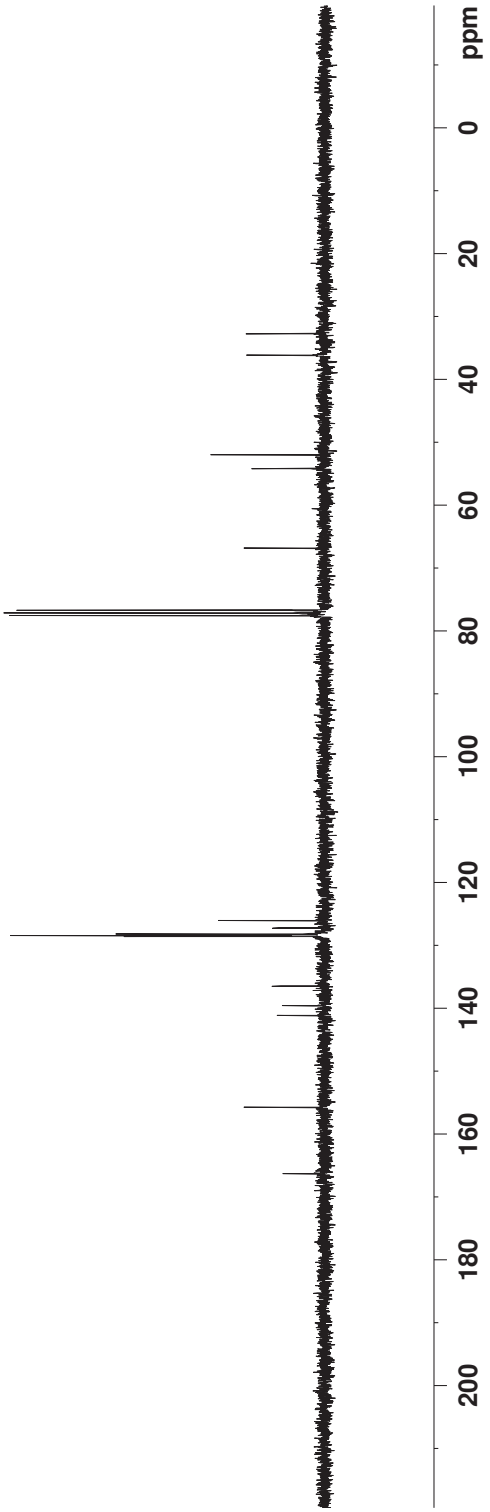

NAME 101231
EXPNO 2
PROCNO 1
Date_ 20101231
Time_ 11.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 287.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300329 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
NH
O
Ph
O
5r
ppm
2.04
2.05
3.00
1.02
2.03
0.95
0.90
1.01
3.08
3.18
3.97

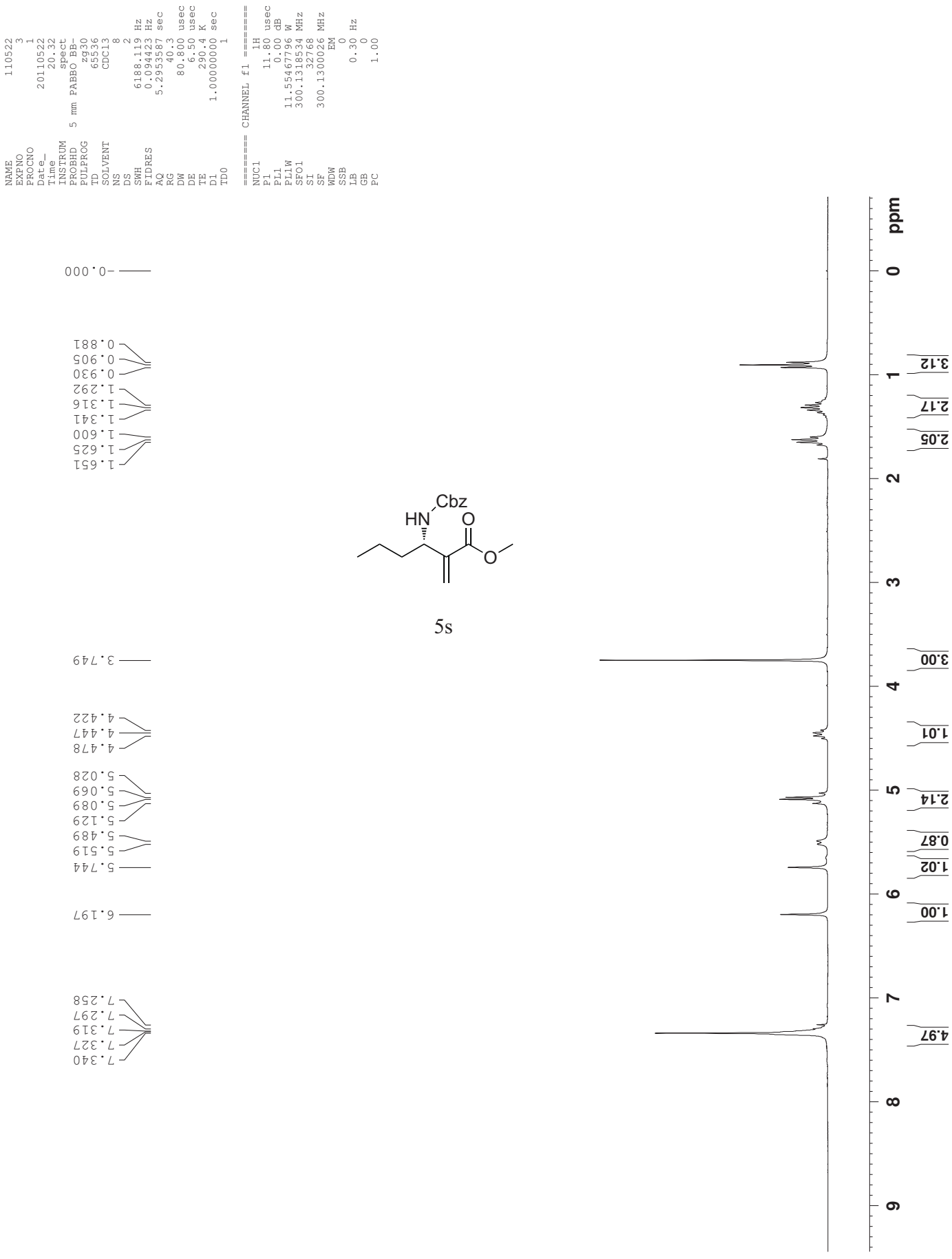

NAME 101231
EXPNO 3
PROCNO 1
Date_ 20101231
Time_ 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 287.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

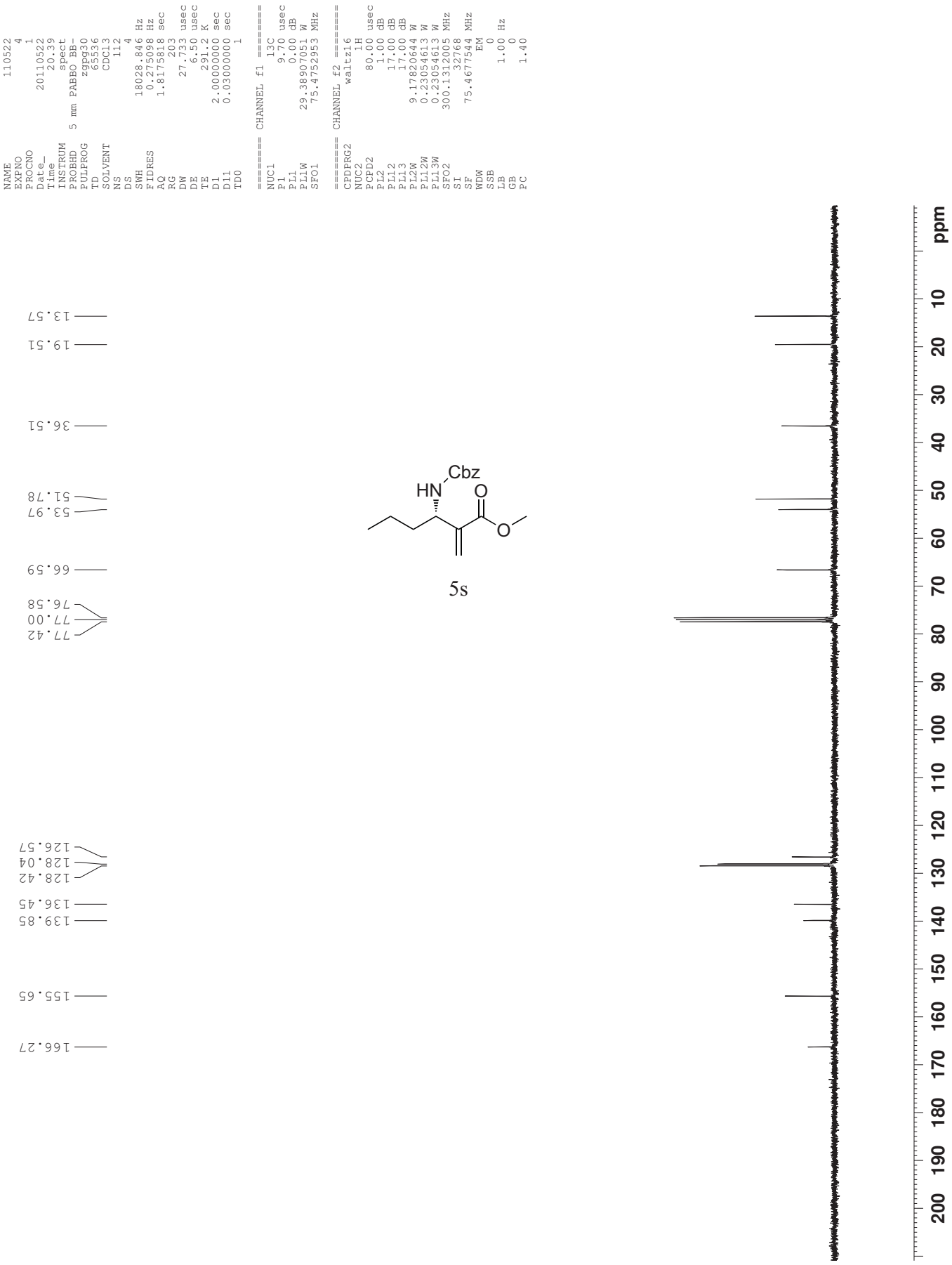

32.72
36.10
51.95
54.17
66.78
76.68
77.10
77.53
126.02
127.22
128.18
128.39
128.44
128.55
136.46
139.57
141.12
155.74
166.29

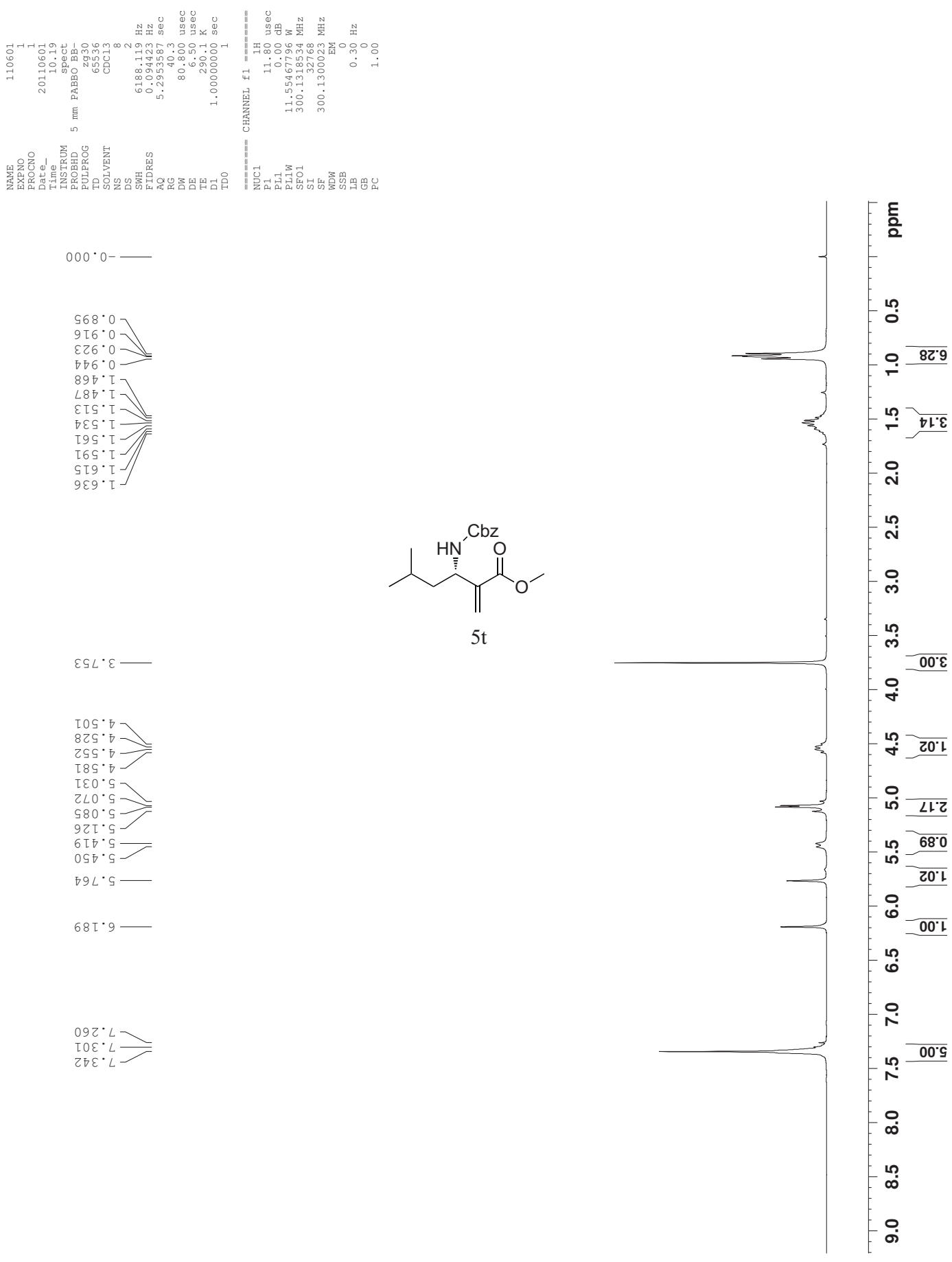

Cbz
NH
O
Ph
O

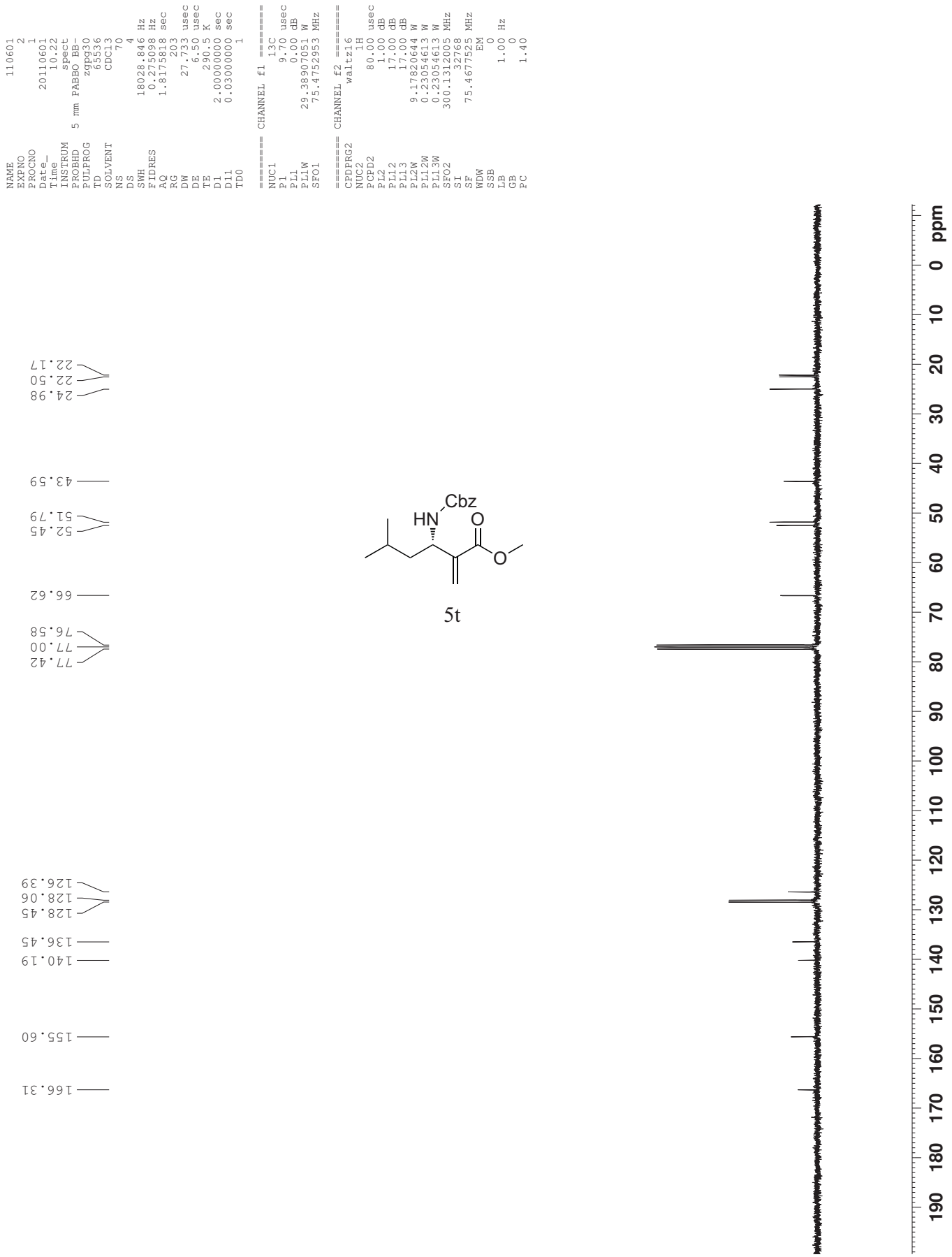
5r

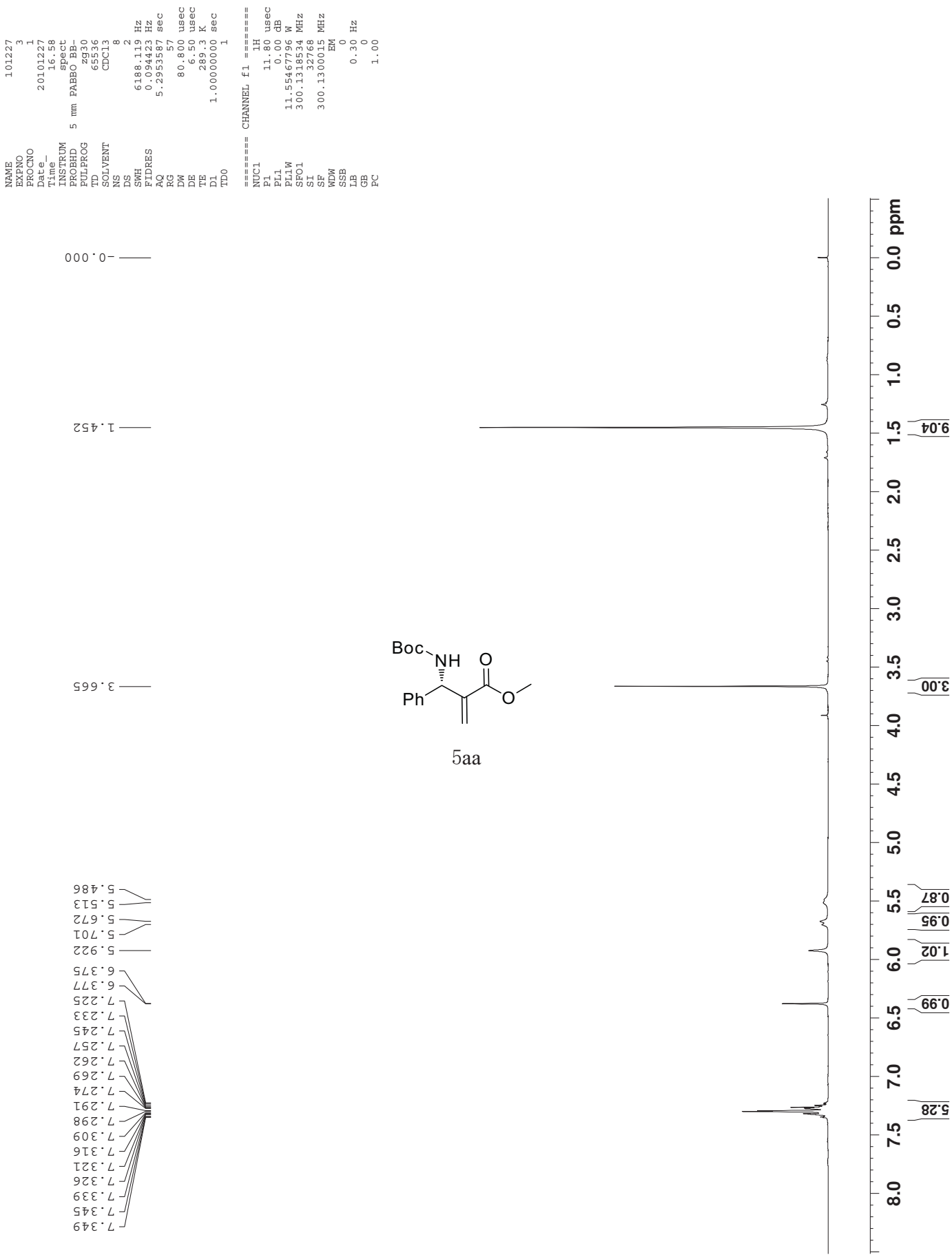

ppm
0
20
40
60
80
100
120
140
160
180
200

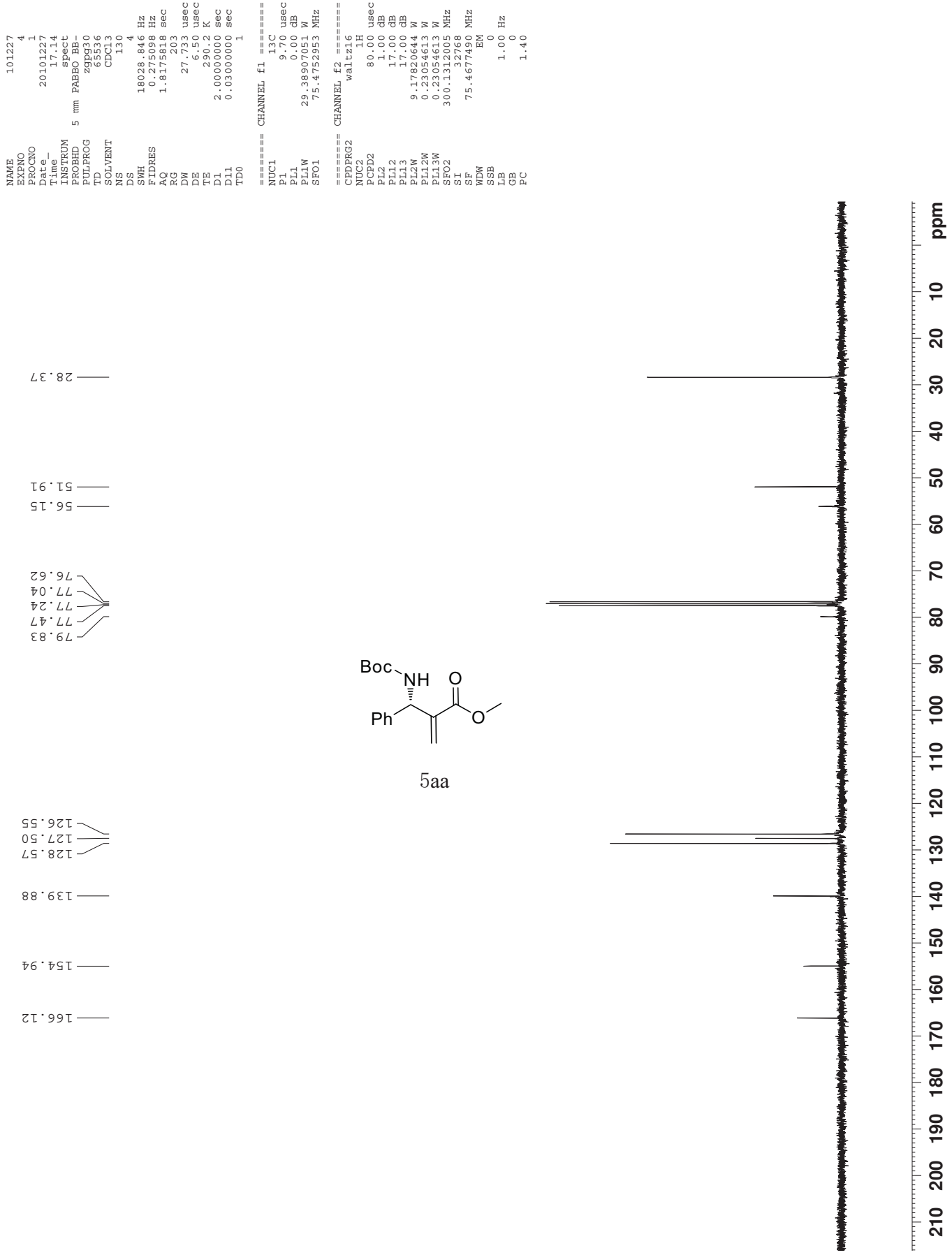

NAME 110522
EXPNO 3
PROCNO 1
Date_ 20110522
Time 20.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.29535387 sec
RG 40.3
DW 80.800 usec
DE 6.50 usec
TE 290.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
0.881
0.905
0.930
1.292
1.316
1.341
1.600
1.625
1.651
3.749
4.422
4.447
4.478
5.028
5.069
5.089
5.129
5.489
5.519
5.744
6.197
7.258
7.297
7.319
7.327
7.340
Cbz
HN
O
O
5s
ppm
0
1
2
3
4
5
6
7
8
9
3.12
2.17
2.05
3.00
1.01
2.14
0.87
1.02
1.00
4.97

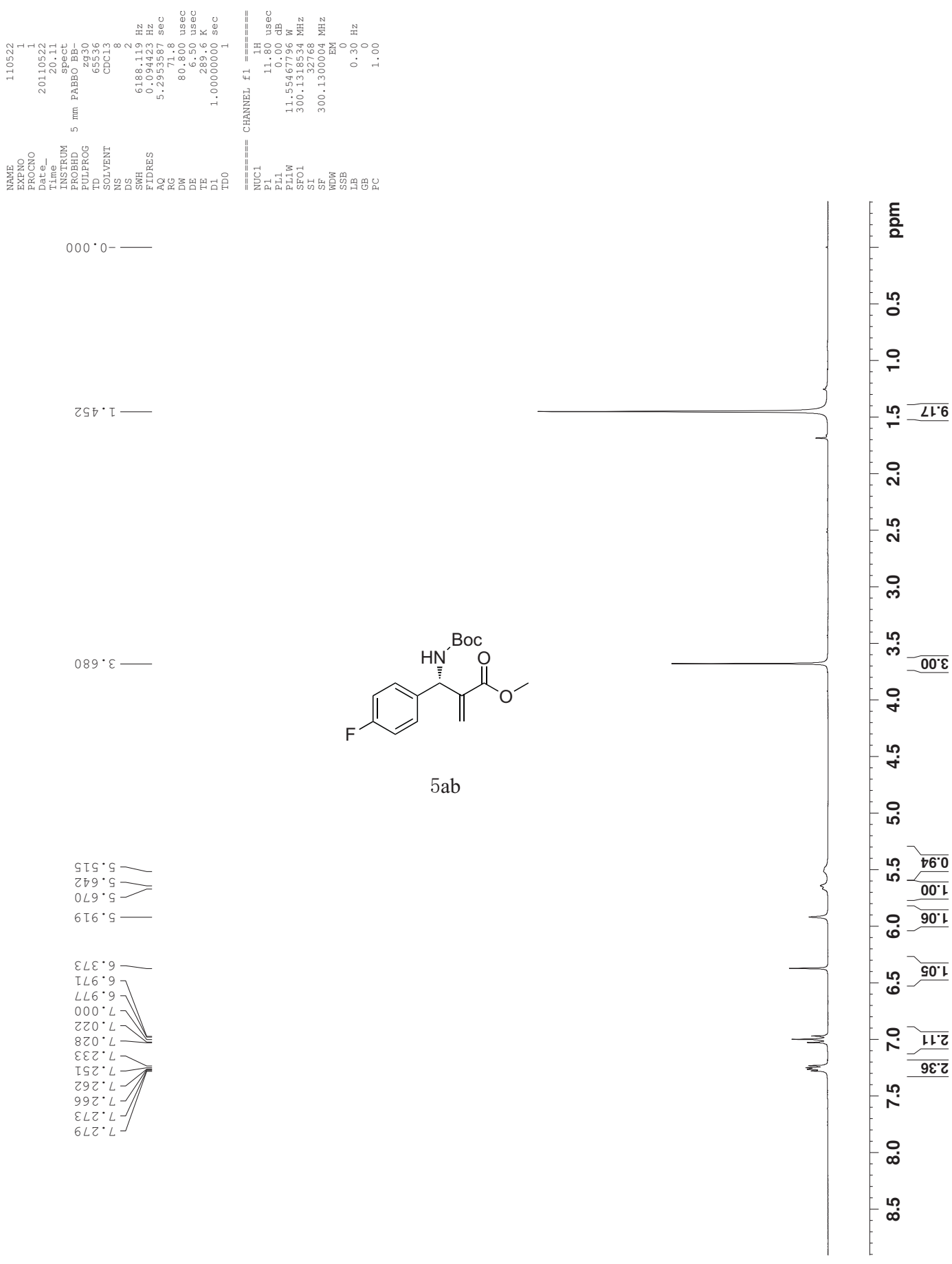

NAME 110522
EXPNO 4
PROCNO 1
Date_ 20110522
Time 20.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 112
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81755818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.57
19.51
36.51
51.78
53.97
66.59
76.58
77.00
77.42
126.57
128.04
128.42
136.45
139.85
155.65
166.27
Cbz
HN
O
O
5s
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200


NAME 110601
EXPNO 1
PROCNO 1
Date_ 20110601
Time 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 40.3
DW 80.800 usec
DE 6.50 usec
TE 290.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300023 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
HN
O
O
5t
ppm
6.28
3.14
3.00
1.02
2.17
0.89
1.02
1.00
5.00

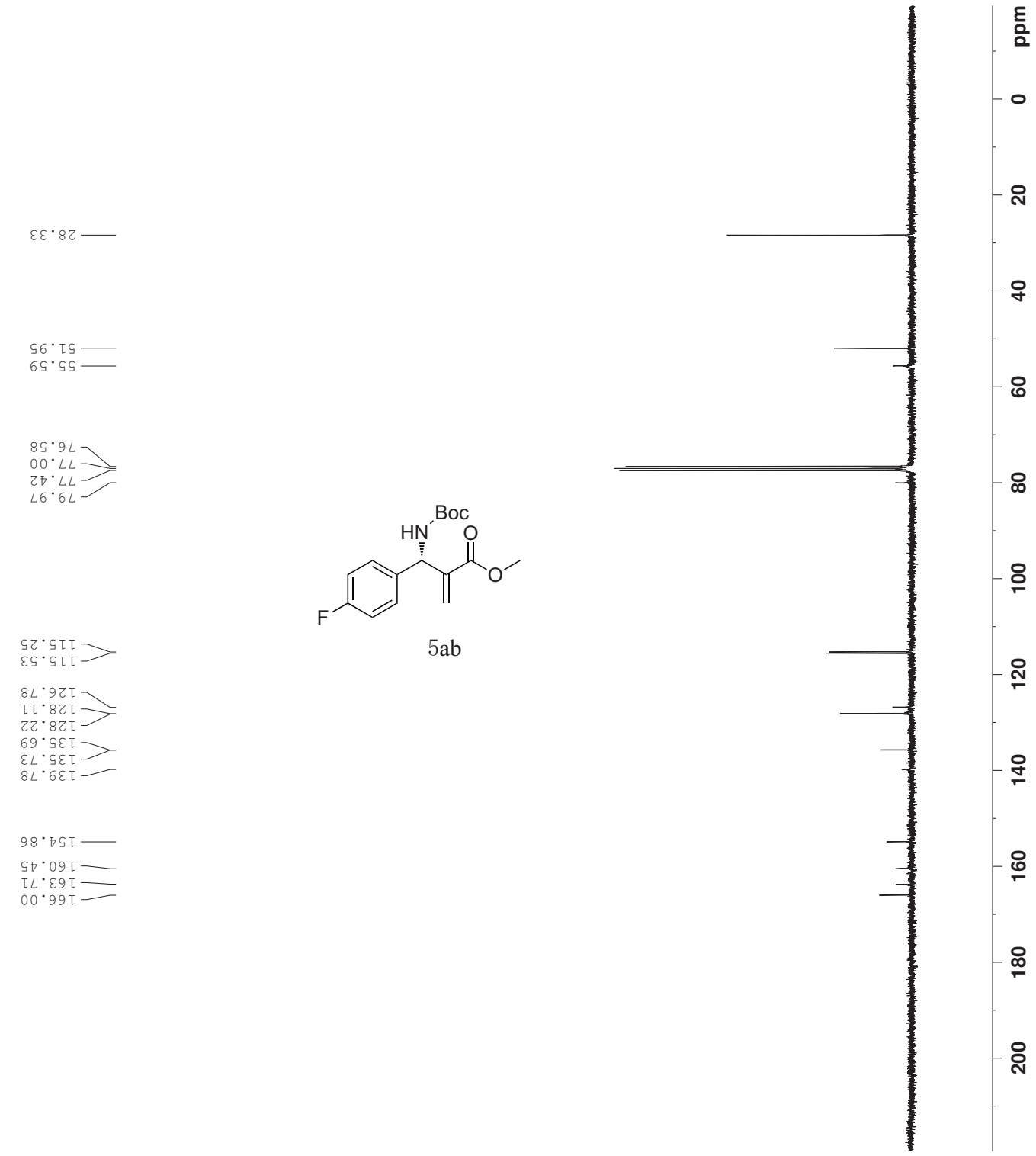



NAME 101227
EXPNO 3
PROCNO 1
Date_ 20101227
Time_ 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 289.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300015 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.452
3.665
5.486
5.513
5.672
5.701
5.922
6.375
6.377
7.225
7.233
7.245
7.257
7.262
7.269
7.274
7.291
7.298
7.309
7.316
7.321
7.326
7.339
7.345
7.349
Boc
NH
O
Ph
O
5aa
ppm
9.04
3.00
0.87
0.95
1.02
0.99
5.28


NAME 101227
EXPNO 4
PROCNO 1
Date_ 20101227
Time 17.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.37
51.91
56.15
76.62
77.04
77.24
77.47
79.83
126.55
127.50
128.57
139.88
154.94
166.12
Boc
NH
O
Ph
O
5aa
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

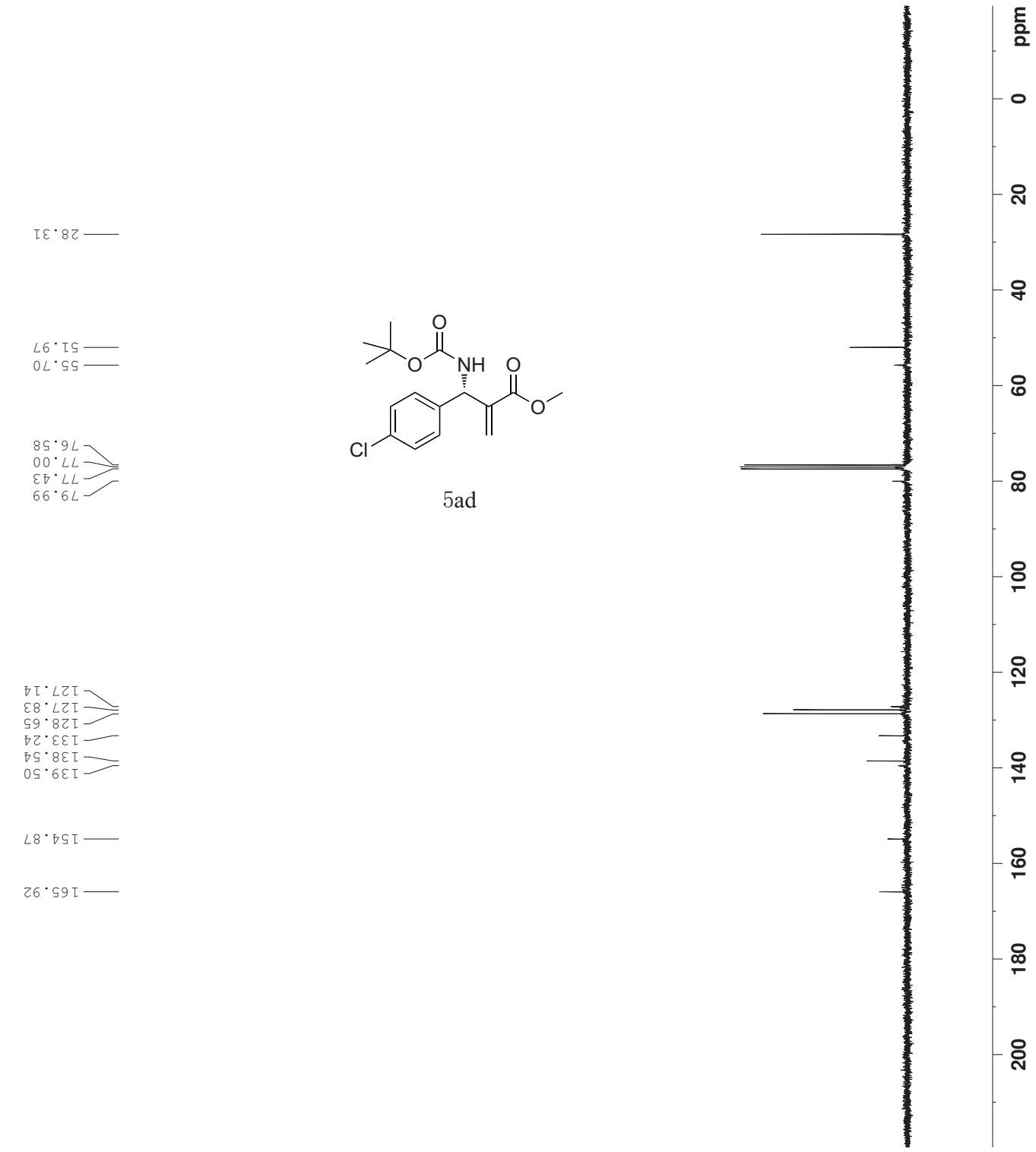

NAME 110522
EXPNO 1
PROCNO 1
Date_ 20110522
Time 20.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 71.8
DW 80.800 usec
DE 6.50 usec
TE 289.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300004 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.452
3.680
5.515
5.642
5.670
5.919
6.373
6.971
6.977
7.000
7.022
7.028
7.233
7.251
7.262
7.266
7.273
7.279
Boc
HN
O
O
F
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.17
3.00
0.94
1.00
1.06
1.05
2.11
2.36

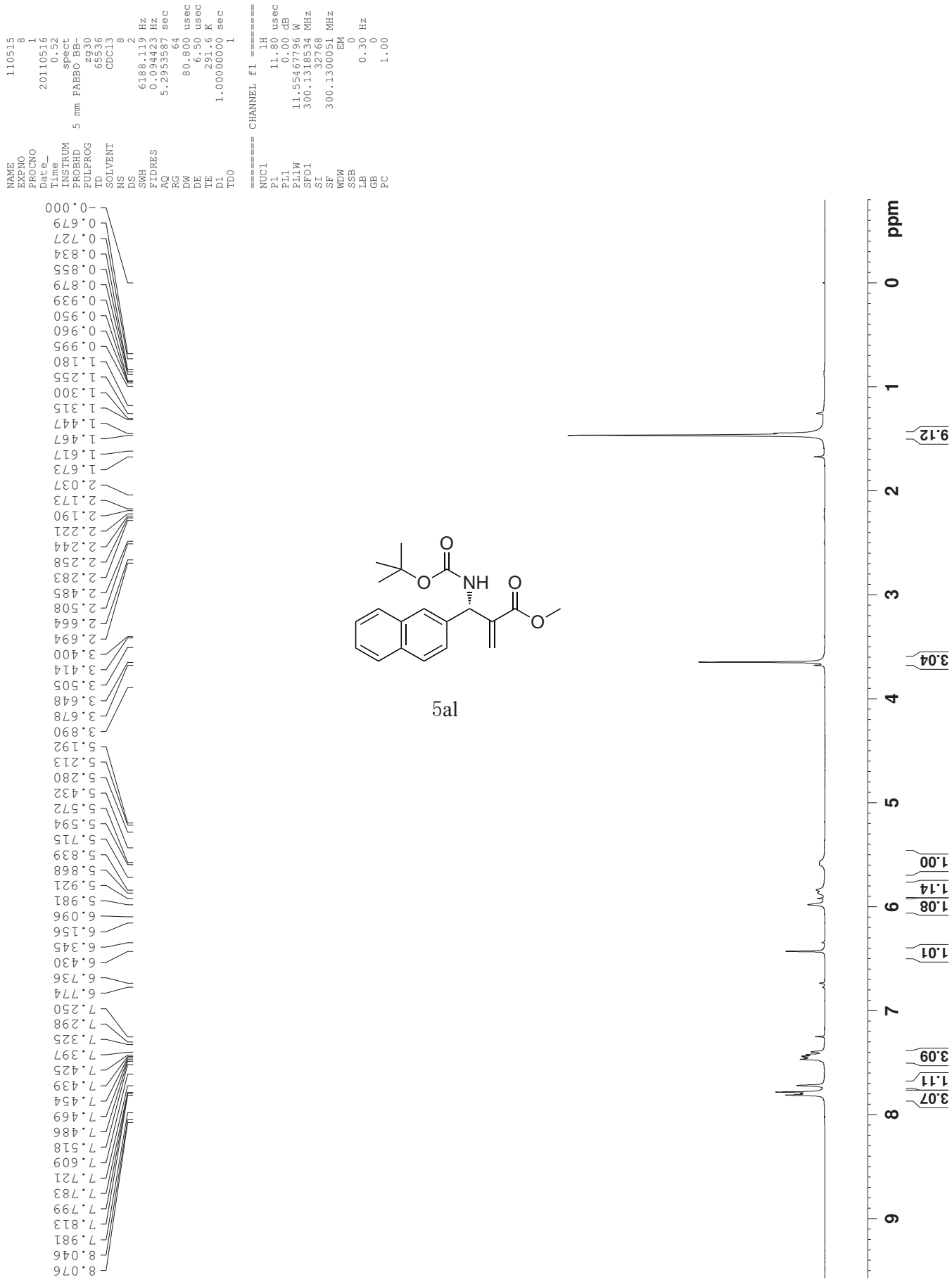
5ab


NAME 110522
EXPNO 5
PROCNO 1
Date_ 20110522
Time 21.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 438
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.33
51.95
55.59
76.58
77.00
77.42
79.97
115.25
115.53
126.78
128.11
128.22
135.69
135.73
139.78
154.86
160.45
163.71
166.00
Boc
HN
O
O
F
5ab
ppm
0
20
40
60
80
100
120
140
160
180
200


NAME 110515
EXPNO 4
PROCNO 1
Date_ 20110516
Time_ 0.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.29535387 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 290.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.450
3.679
5.554
5.632
5.661
5.923
6.374
7.206
7.235
7.271
7.300
ppm
0
1
2
3
4
5
6
7
8
9
8.96
3.00
0.87
0.98
1.05
0.99
3.98
O
O
NH
O
O
Cl

5ad

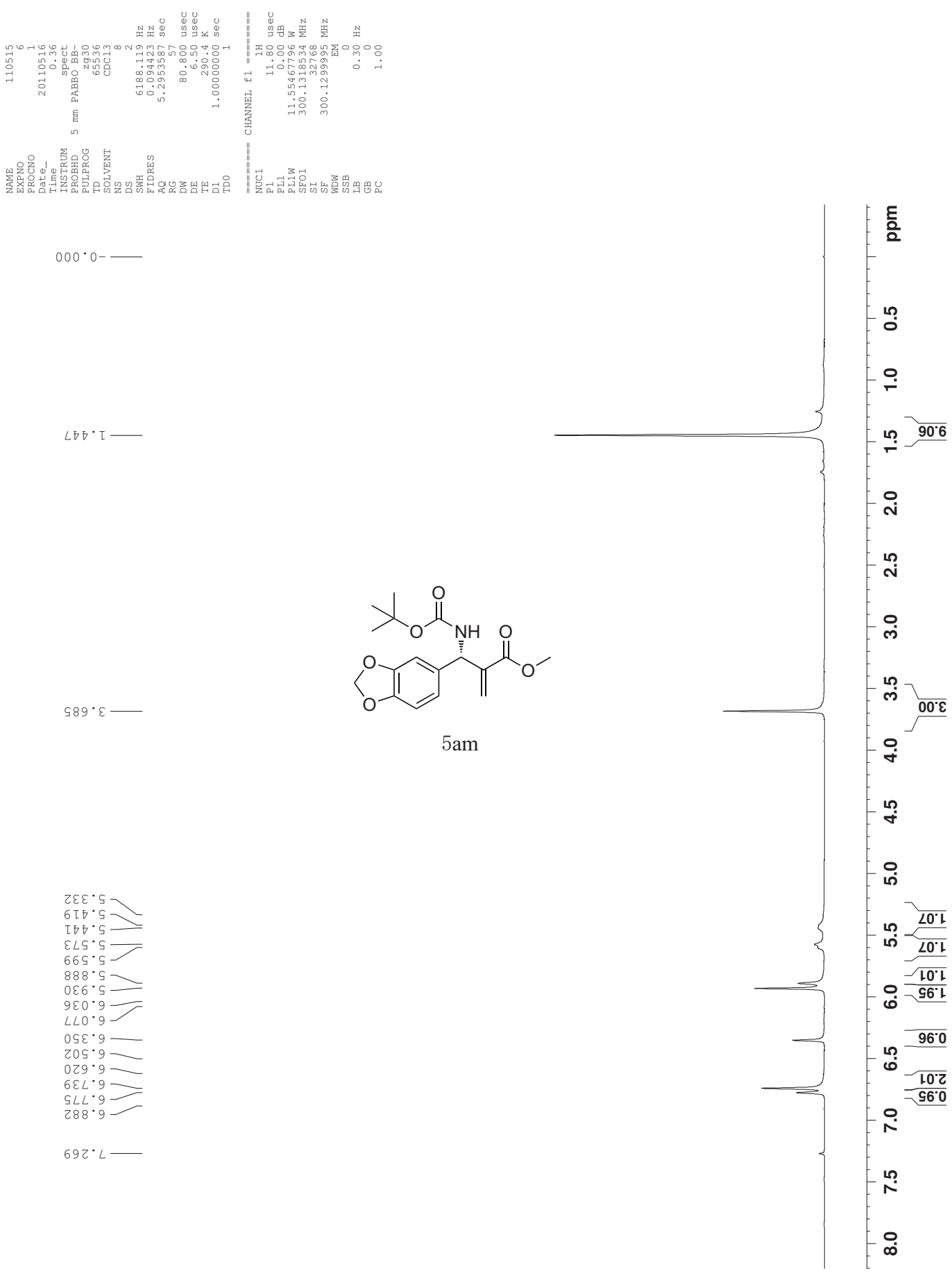

NAME 110515
EXPNO 5
PROCNO 1
Date_ 20110516
Time 0.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 101
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81755818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677518 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.31
51.97
55.70
76.58
77.00
77.43
79.99
127.14
127.83
128.65
133.24
138.54
139.50
154.87
165.92
O
O
NH
O
O
Cl
5ad
ppm
0
20
40
60
80
100
120
140
160
180
200


NAME 110515
EXPNO 8
PROCNO 1
Date_ 20110516
Time_ 0.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 64
DW 80.800 usec
DE 6.50 usec
TE 291.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
9.12
3.04
1.00
1.14
1.08
1.01
3.09
1.11
3.07

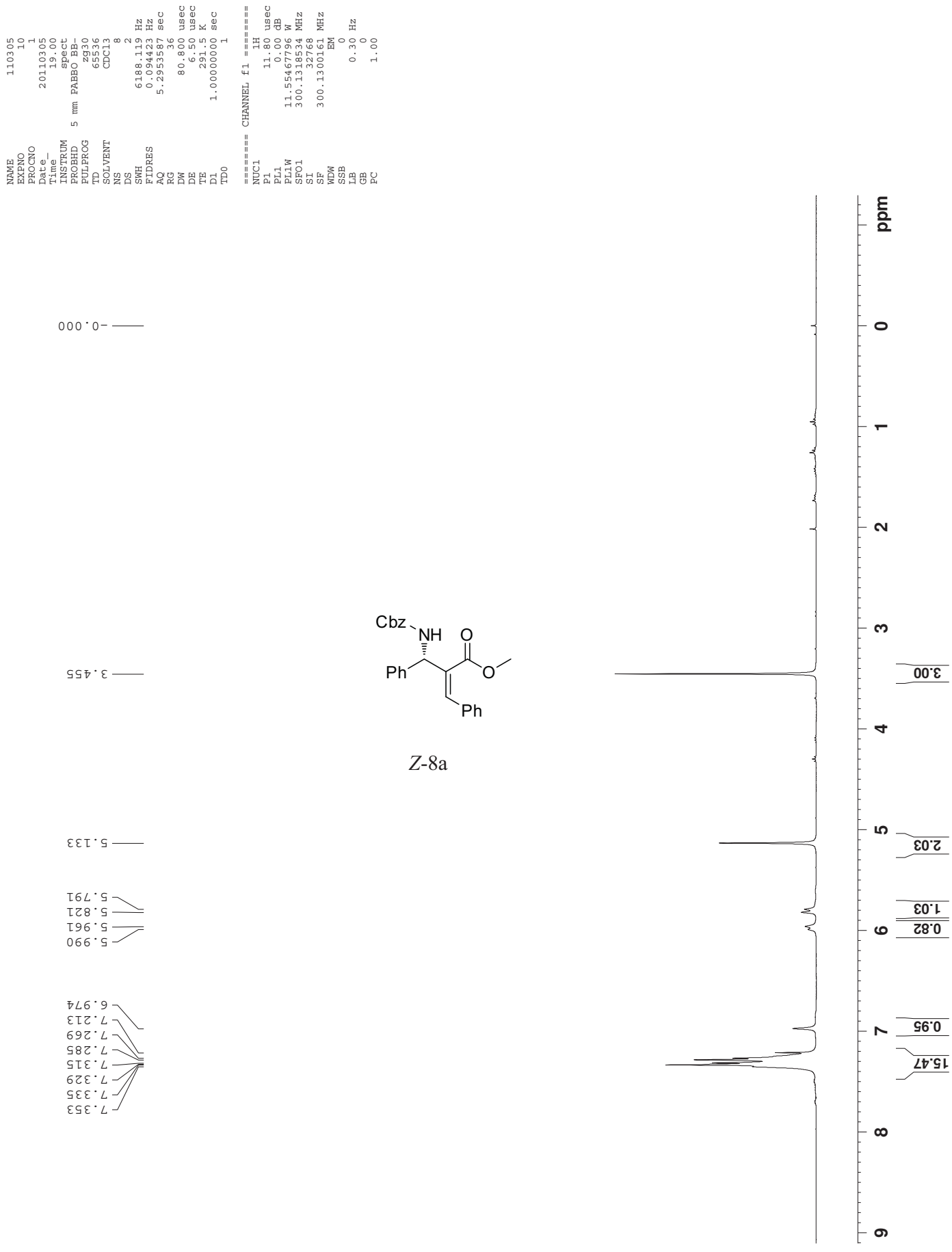
O
O
NH
O
O

5al

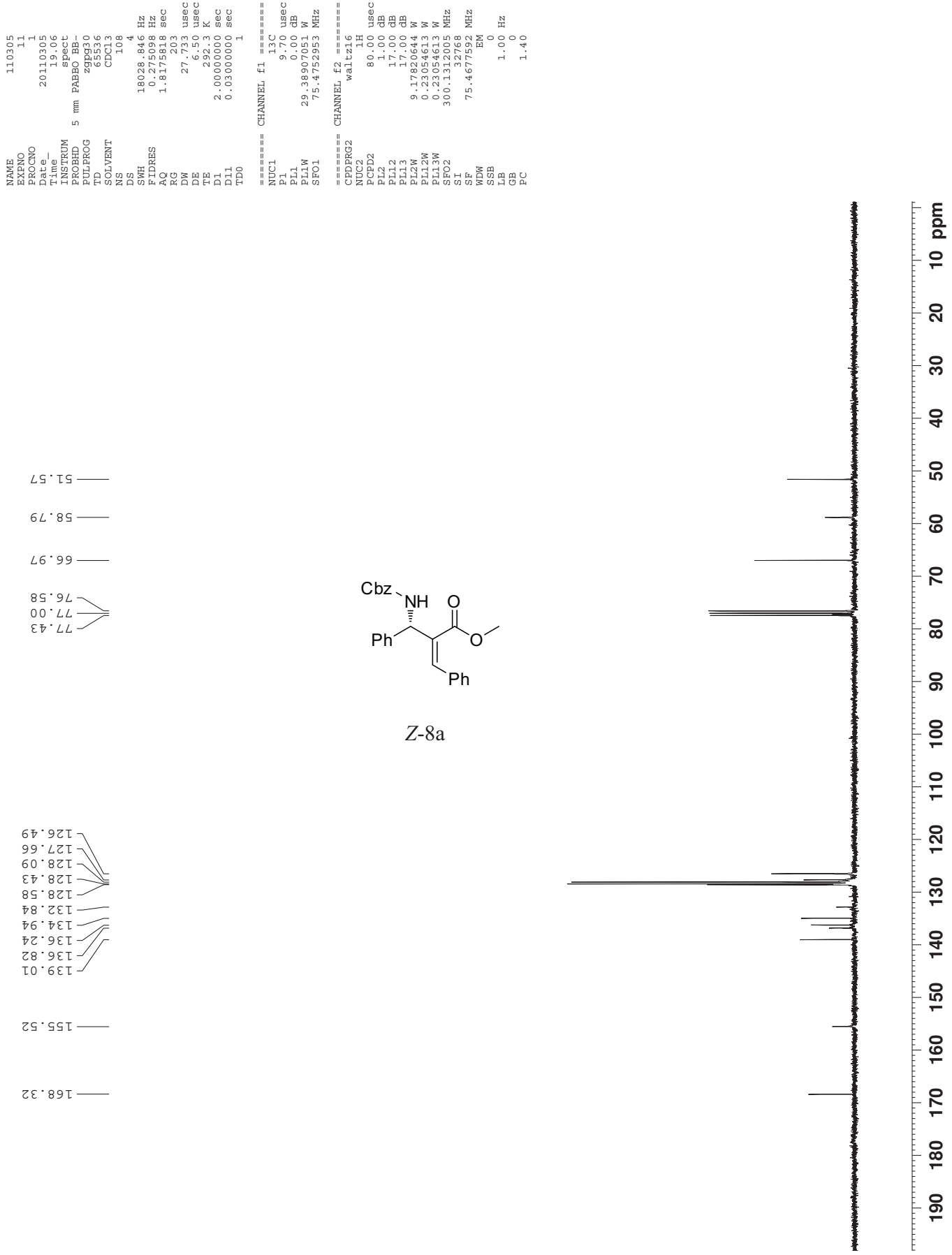

NAME 110515
EXPNO 9
PROCNO 1
Date_ 20110516
Time 1.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 264
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677517 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.36
51.91
56.20
76.58
77.00
77.42
79.89
124.89
125.19
125.97
126.16
126.69
127.56
127.97
128.37
132.73
133.23
137.32
139.95
154.94
166.12
O
O
NH
O
O
5al
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190


NAME 110515
EXPNO 6
PROCNO 1
Date_ 20110516
Time 0.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 290.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299995 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.447
3.685
5.332
5.419
5.441
5.573
5.599
5.888
5.930
6.036
6.077
6.350
6.502
6.620
6.739
6.775
6.882
7.269
O
O
NH
O
O
O
O
5am
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
9.06
3.00
1.07
1.07
1.01
1.95
0.96
2.01
0.95

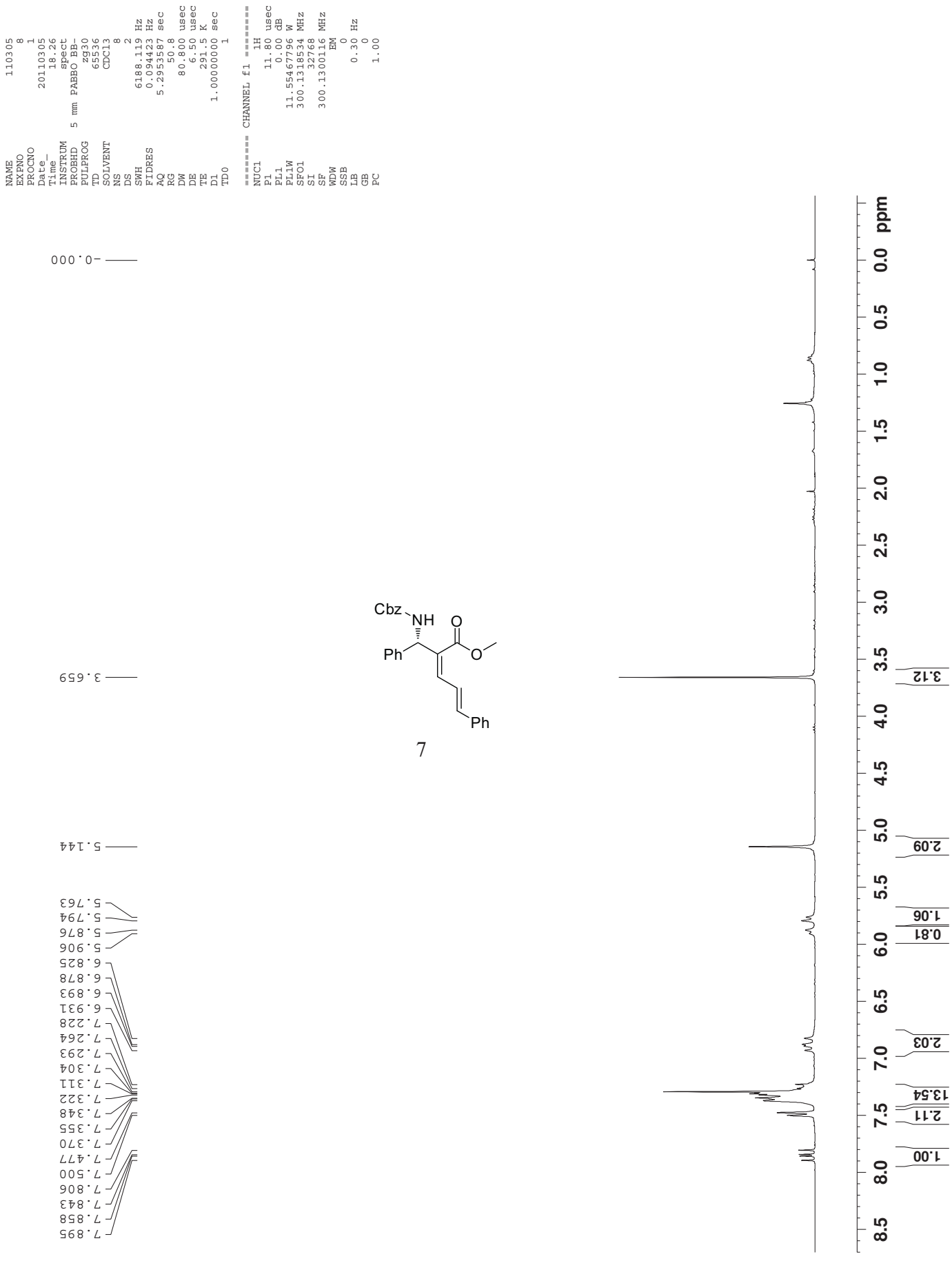

NAME 110515
EXPNO 10
PROCNO 1
Date_ 20110516
Time_ 1.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 293.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677517 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.32
51.89
55.81
76.58
77.00
77.42
79.81
101.06
107.27
108.21
119.83
126.21
133.83
140.01
146.91
147.81
154.81
166.08
O
O
NH
O
O
O
O
5am
ppm
0
20
40
60
80
100
120
140
160
180
200

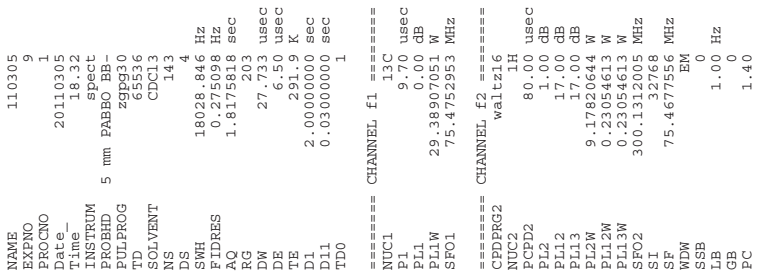

NAME 110305
EXPNO 10
PROCNO 1
Date_ 20110305
Time 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 291.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300161 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.455
5.133
5.791
5.821
5.961
5.990
6.974
7.213
7.269
7.285
7.315
7.329
7.335
7.353
Cbz
NH
O
Ph
O
Ph
3.00
2.03
1.03
0.82
0.95
15.47
ppm
9
8
7
6
5
4
3
2
1
0

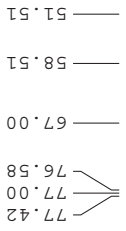
Z-8a

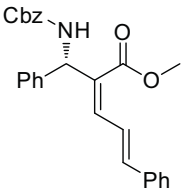

NAME 110305
EXPNO 11
PROCNO 1
Date_ 20110305
Time 19.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 108
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677592 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.57
58.79
66.97
76.58
77.00
77.43
126.49
127.66
128.09
128.43
128.58
132.84
134.94
136.24
136.82
139.01
155.52
168.32
Cbz
NH
O
Ph
O
Ph
10 ppm
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

Z-8a

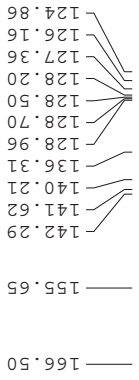

NAME 110305
EXPNO 8
PROCNO 1
Date_ 20110305
Time_ 18.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 291.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.659
5.144
5.763
5.794
5.876
5.906
6.825
6.878
6.893
6.931
7.228
7.264
7.293
7.304
7.311
7.322
7.348
7.355
7.370
7.477
7.500
7.806
7.843
7.858
7.895
Cbz
NH
O
Ph
O
Ph
7
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.12
2.09
1.06
0.81
2.03
13.54
2.11
1.00

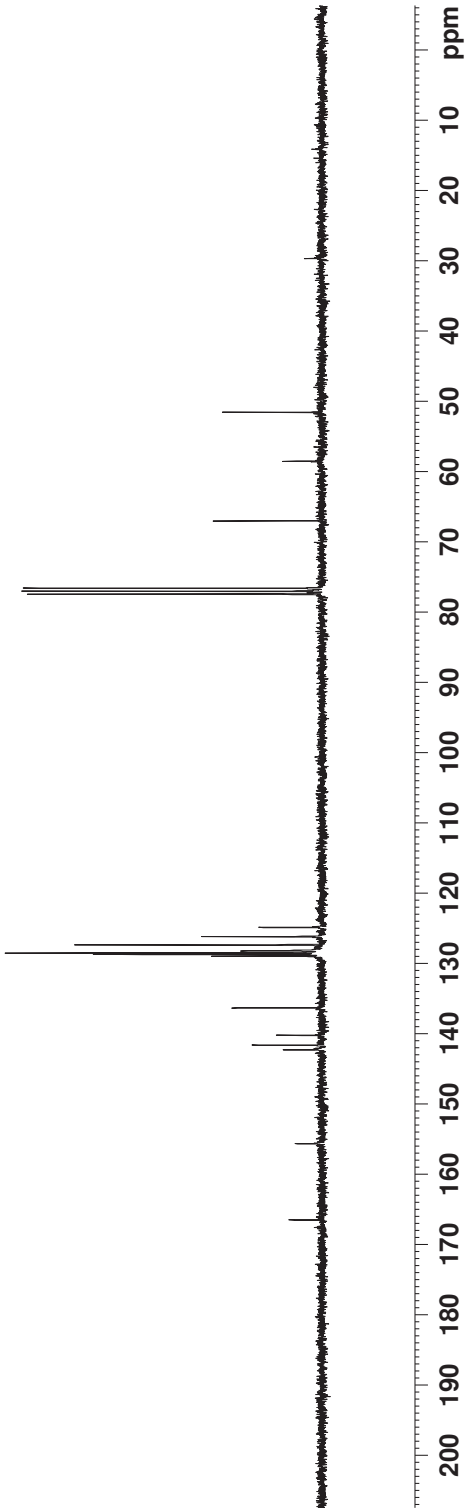

NAME 110305
EXPNO 9
PROCNO 1
Date_ 20110305
Time 18.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

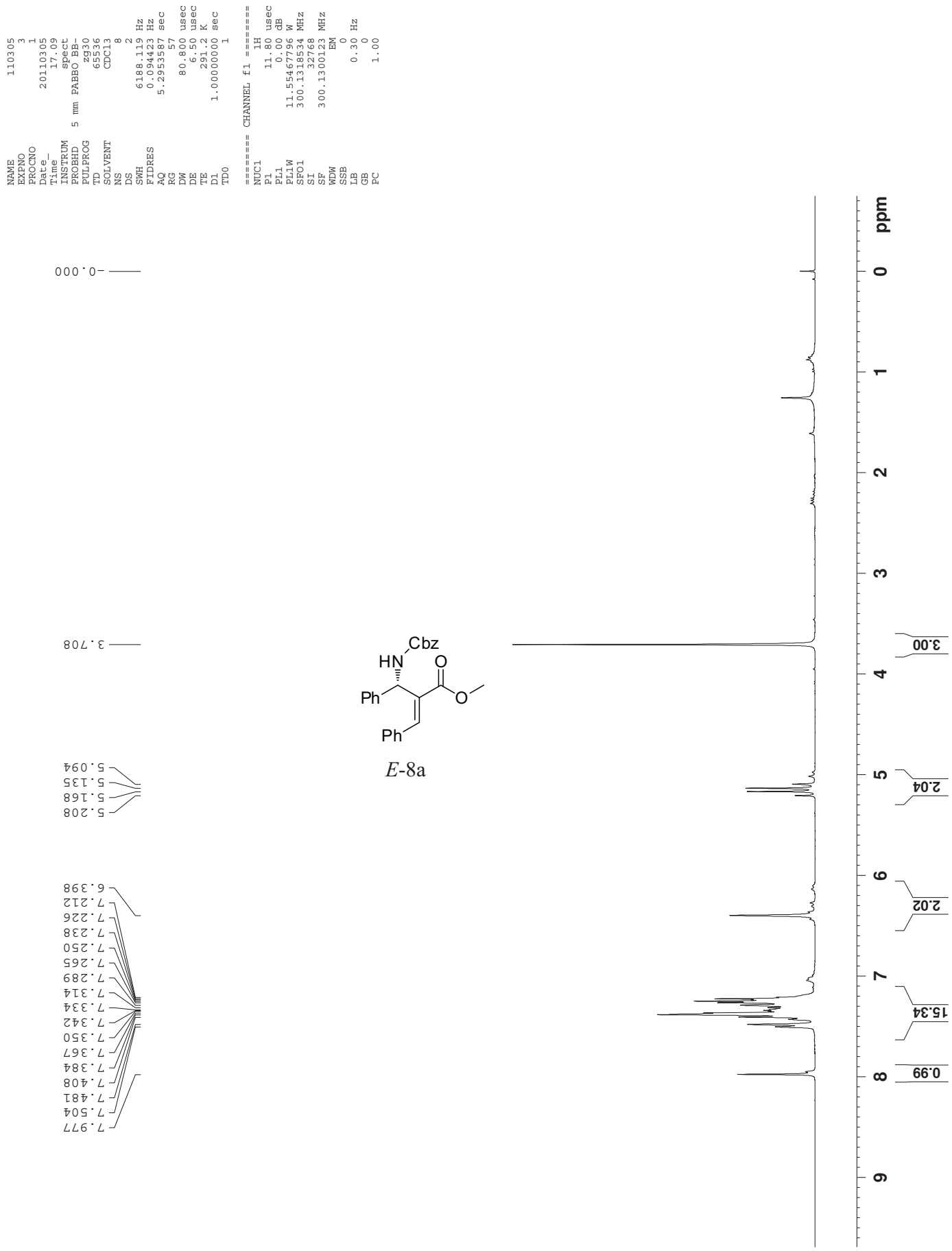

51.51
58.51
67.00
76.58
77.00
77.42


Cbz
NH
O
Ph
O
Ph

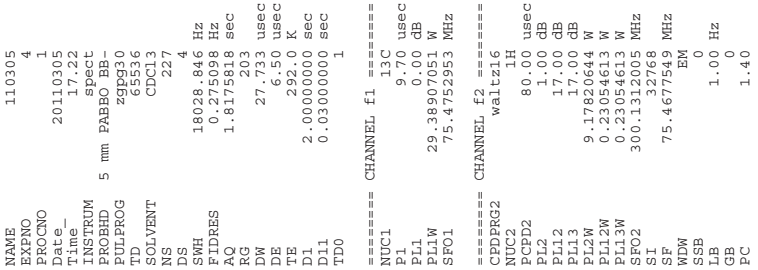
7

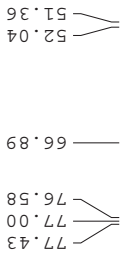

124.86
126.16
127.36
128.20
128.50
128.70
128.96
136.31
140.21
141.62
142.29
155.65
166.50

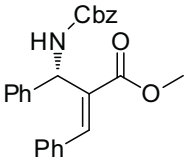

ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200



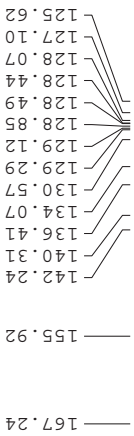
E-8a

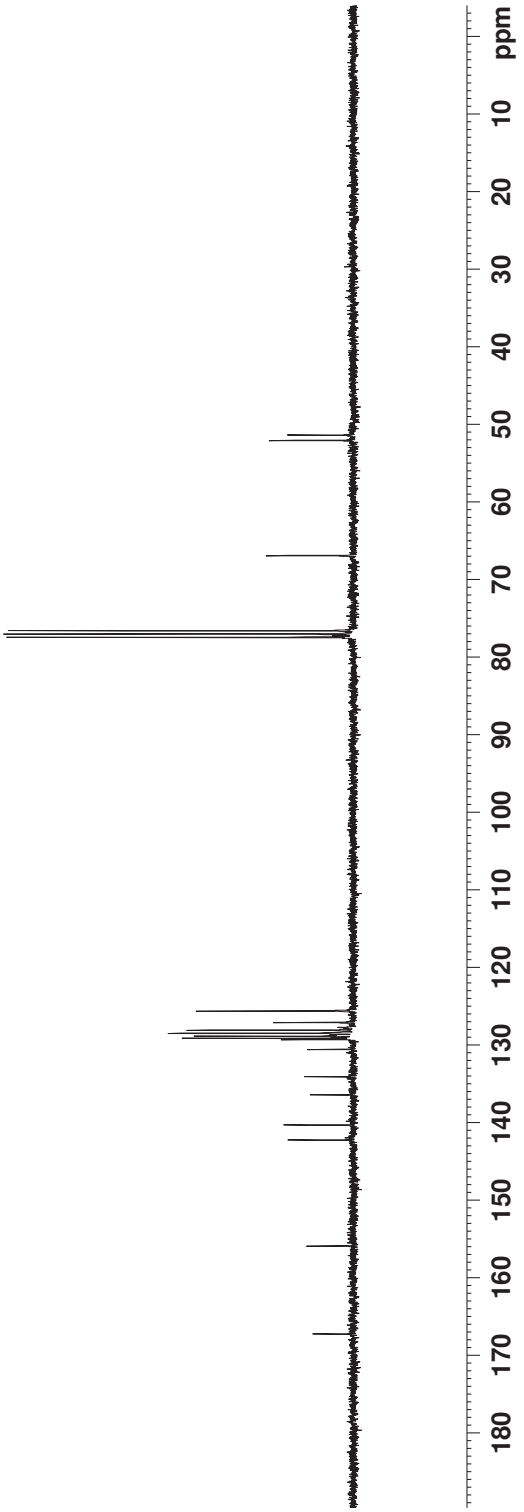

NAME 110305
EXPNO 4
PROCNO 1
Date_ 20110305
Time 17.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 227
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 292.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677549 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

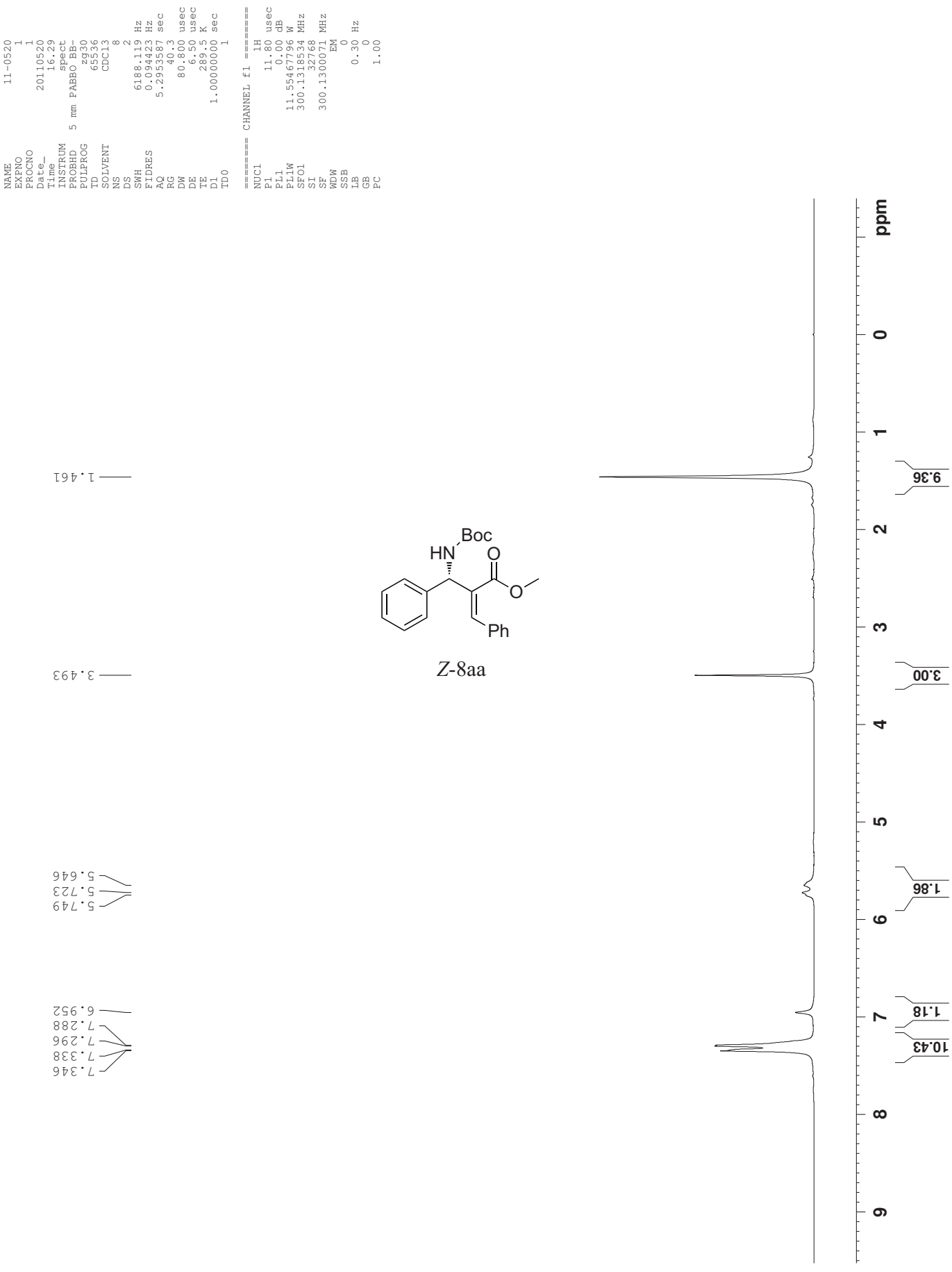

51.36
52.04
66.89
76.58
77.00
77.43


Cbz
HN
O
Ph
O
Ph

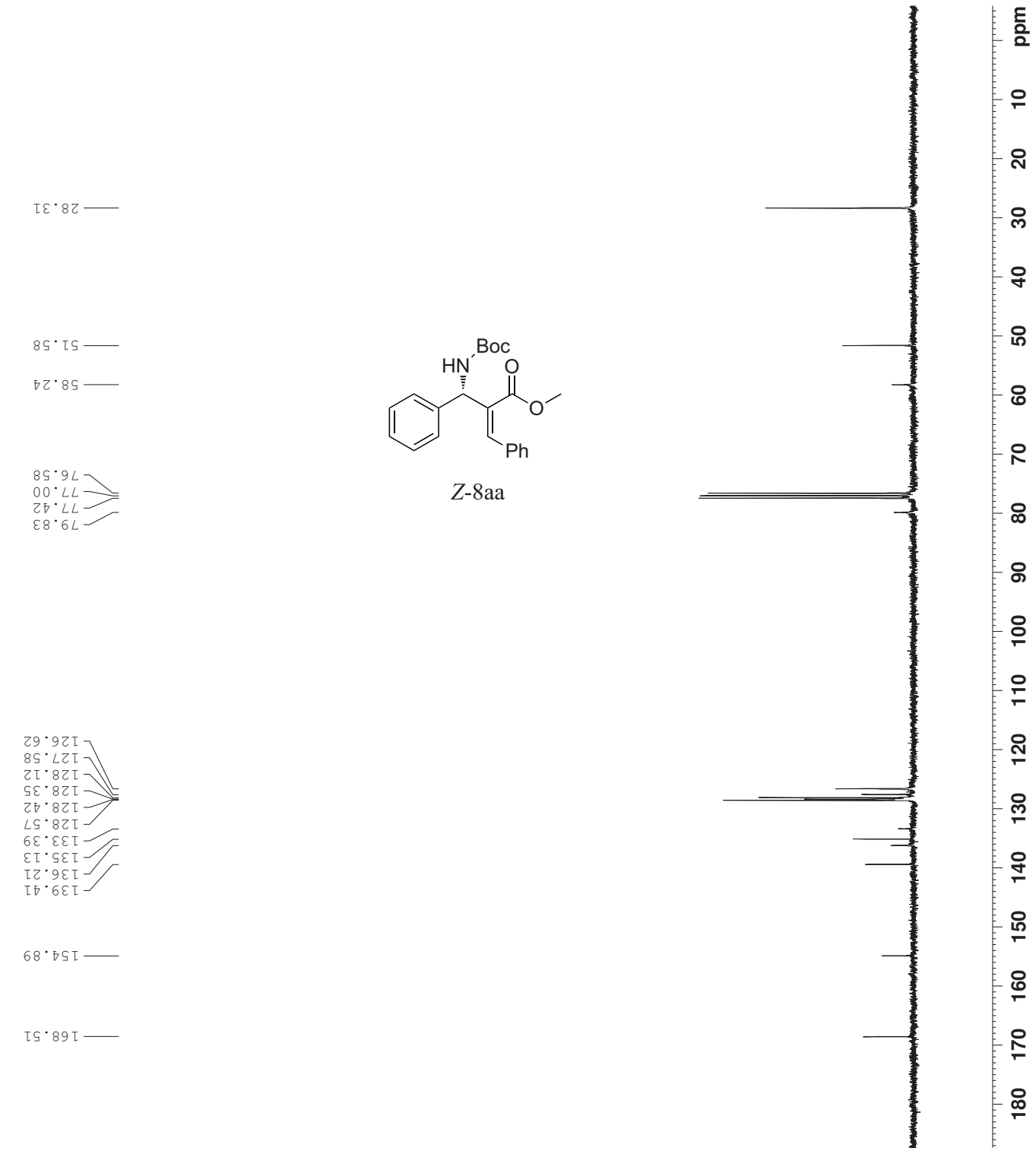
E-8a


125.62
127.10
128.07
128.44
128.49
128.85
129.12
129.29
130.57
134.07
136.41
140.31
142.24
155.92
167.24

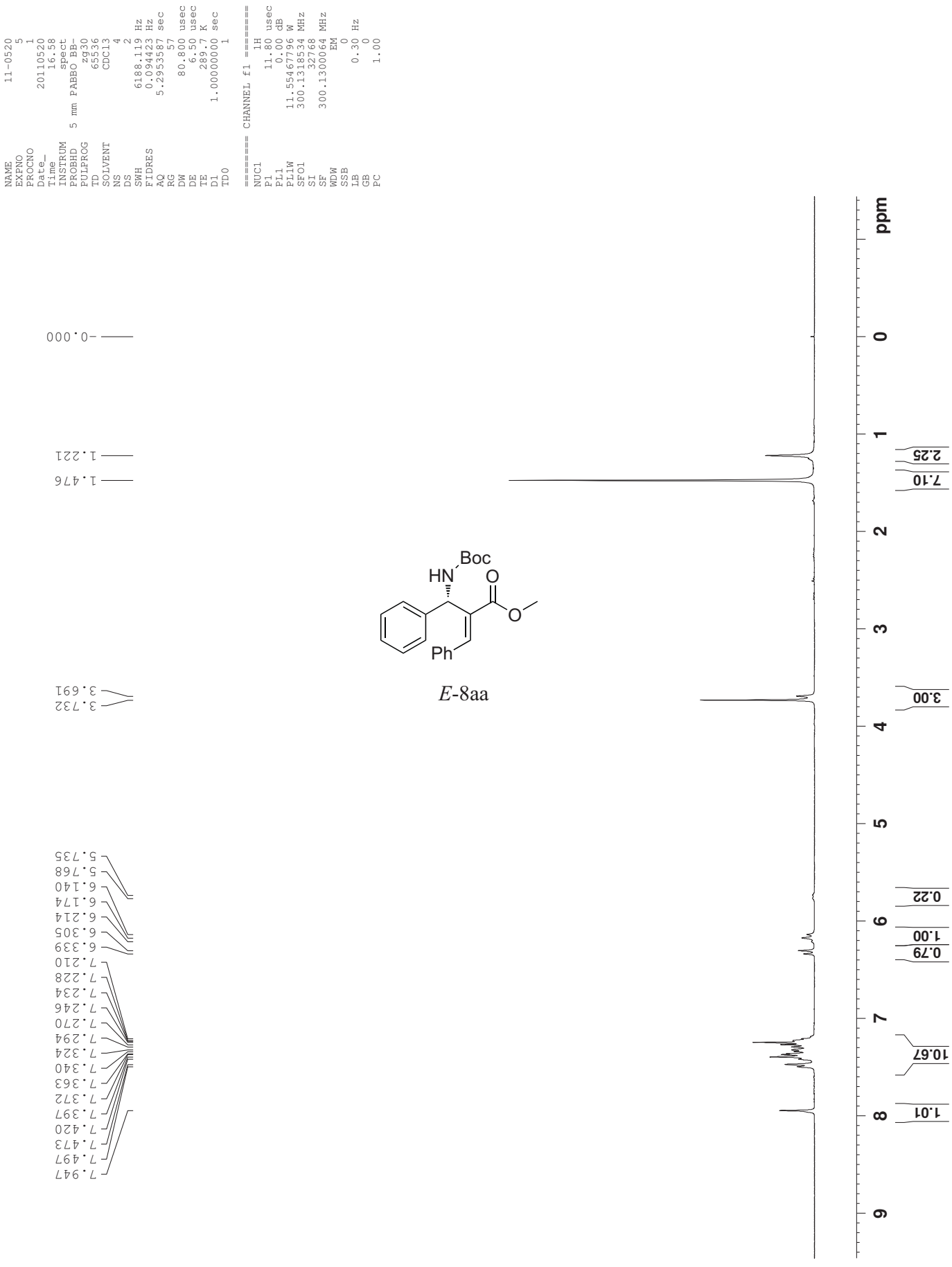

ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180



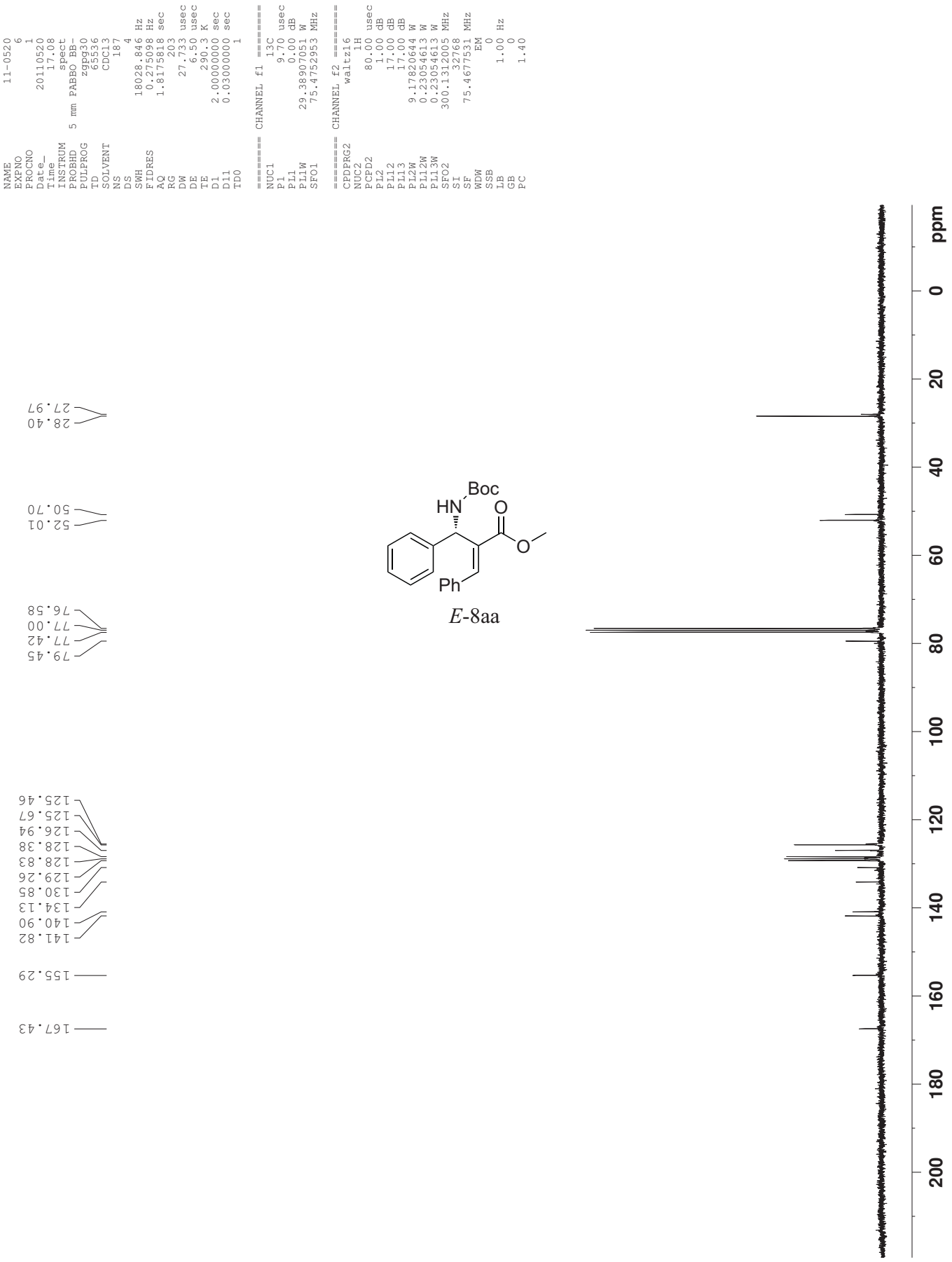
Z-8aa

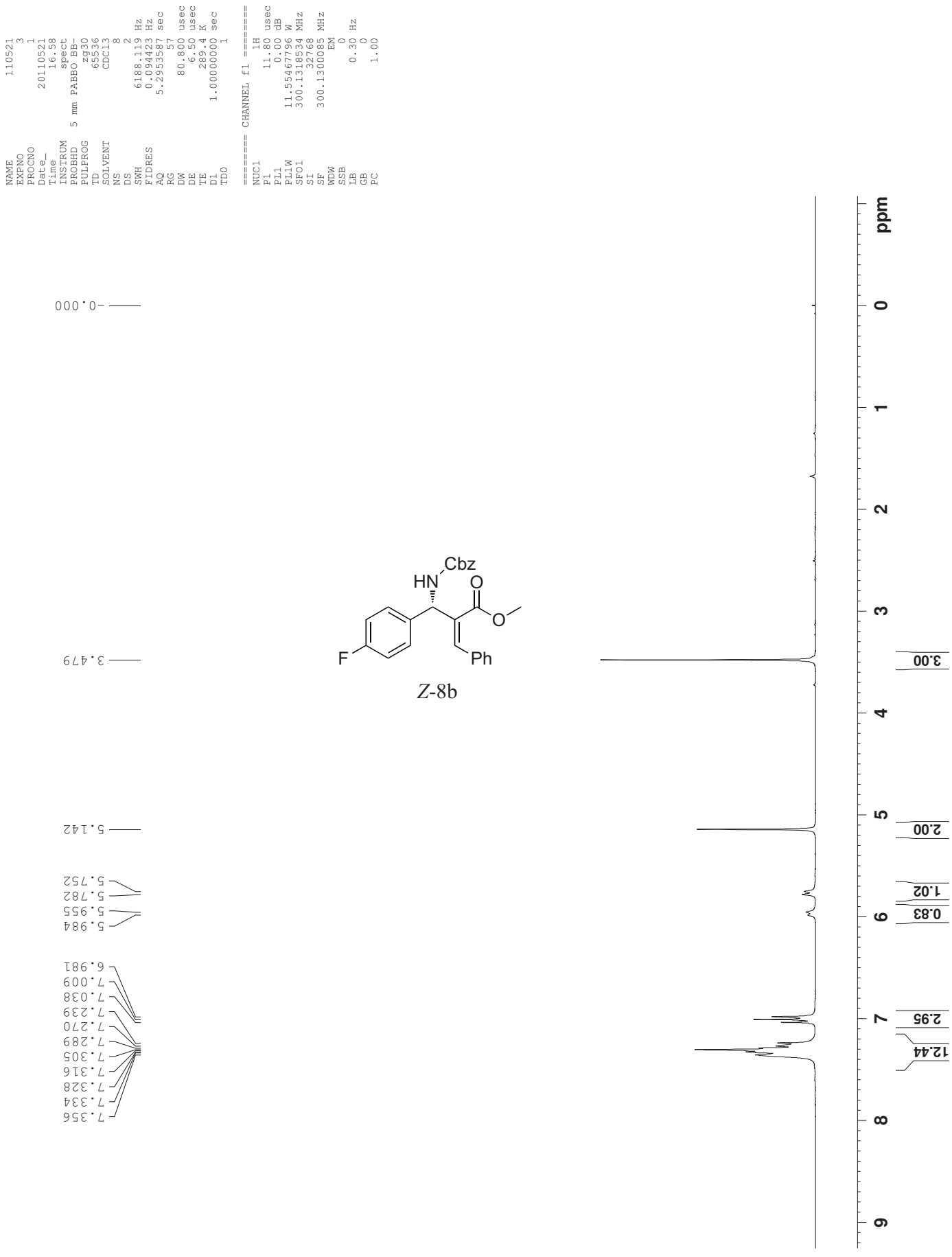

NAME 11-0520
EXPNO 3
PROCNO 1
Date_ 20110520
Time 16.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 151
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81755818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.31
51.58
58.24
76.58
77.00
77.42
79.83
126.62
127.58
128.12
128.35
128.42
128.57
133.39
135.13
136.21
139.41
154.89
168.51
Boc
HN
O
O
Ph
ppm

Z-8aa


NAME 11-0520
EXPNO 5
PROCNO 1
Date_ 20110520
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 4
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 289.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.221
1.476
3.691
3.732
5.735
5.768
6.140
6.174
6.214
6.305
6.339
7.210
7.228
7.234
7.246
7.270
7.294
7.324
7.340
7.363
7.372
7.397
7.420
7.473
7.497
7.947
Boc
HN
O
O
Ph
ppm
0
1
2
3
4
5
6
7
8
9
2.25
7.10
3.00
0.22
1.00
0.79
10.67
1.01

E-8aa

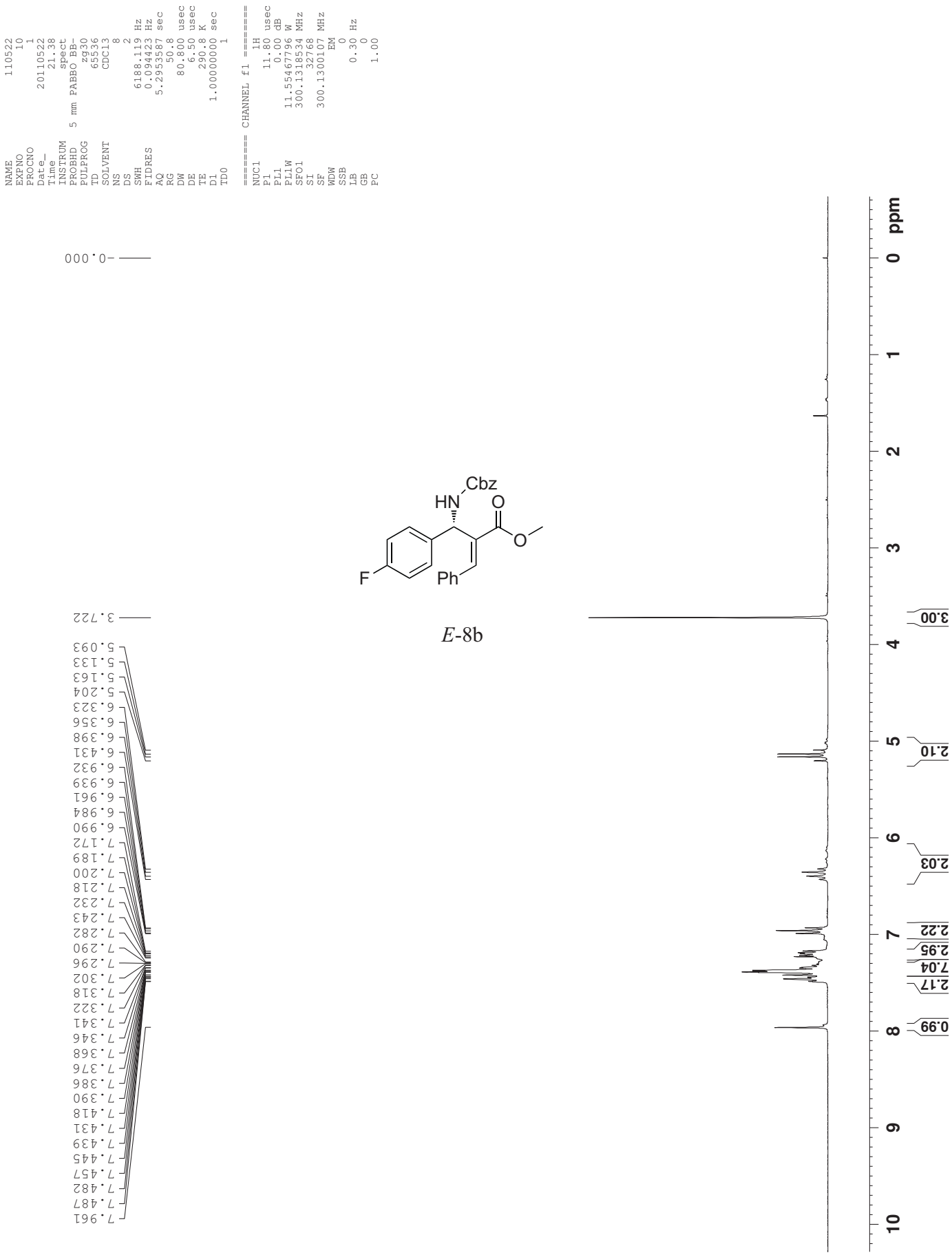



NAME 110521
EXPNO 3
PROCNO 1
Date_ 20110521
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 289.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.000
3.479
5.142
5.752
5.782
5.955
5.984
6.981
7.009
7.038
7.239
7.270
7.289
7.305
7.316
7.328
7.334
7.356
Cbz
HN
O
O
F
Ph
ppm
0
1
2
3
4
5
6
7
8
9
3.00
2.00
1.02
0.83
2.95
12.44

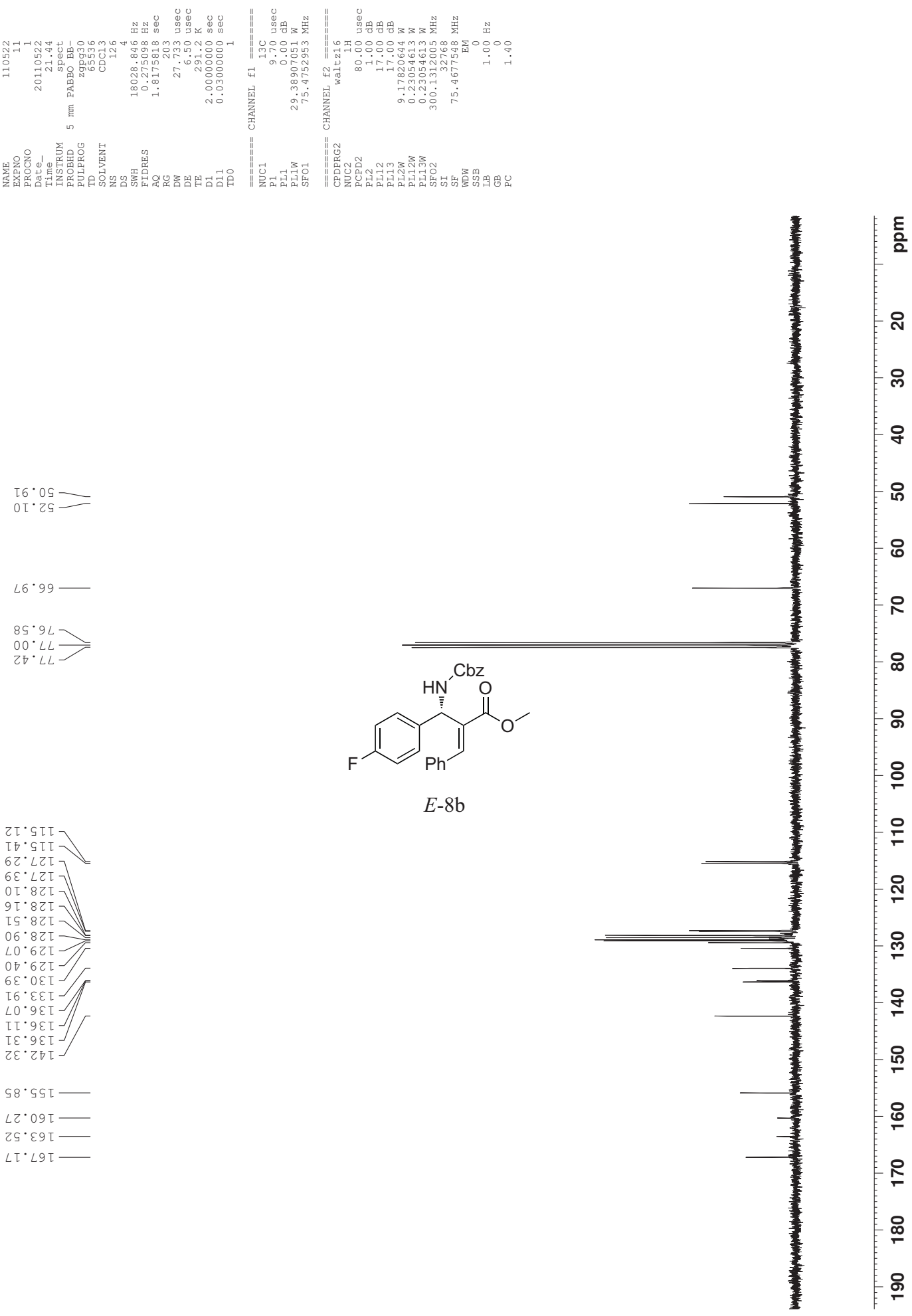
Z-8b

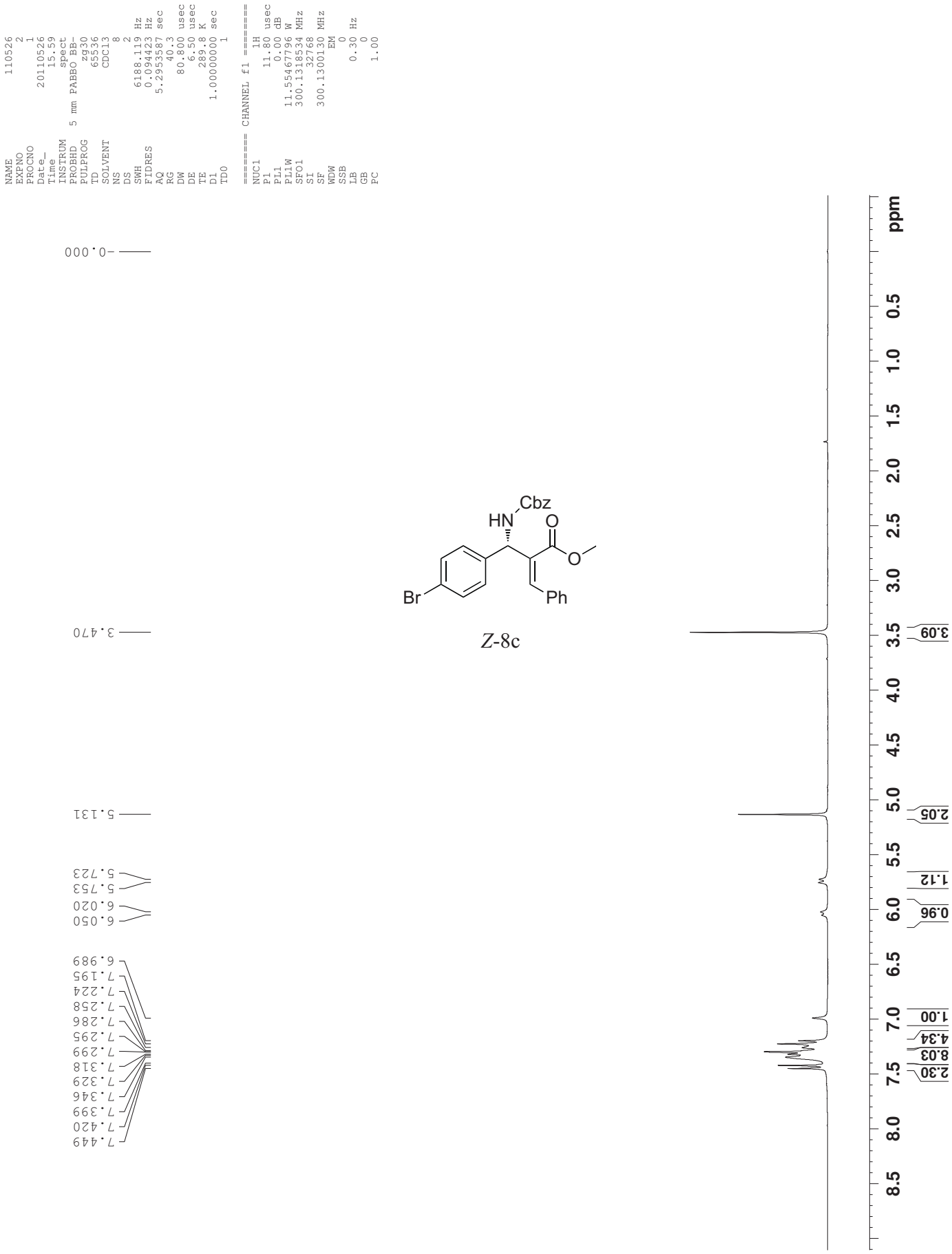

NAME 110521
EXPNO 4
PROCNO 1
Date_ 20110521
Time 17.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 207
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.70
58.36
67.11
76.58
77.00
77.42
115.36
115.65
128.18
128.48
128.62
132.53
134.79
134.86
134.90
136.16
137.13
155.51
160.56
163.83
168.33
Cbz
HN
O
O
F
Ph
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

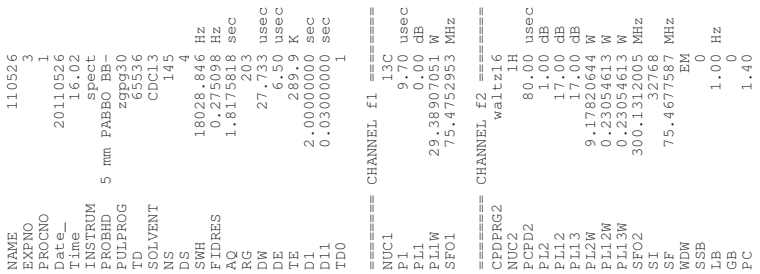
Z-8b



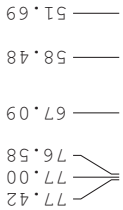
E-8b

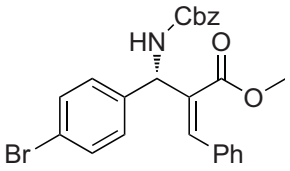

NAME 110522
EXPNO 11
PROCNO 1
Date_ 20110522
Time 21.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 126
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
50.91
52.10
66.97
76.58
77.00
77.42
115.12
115.41
127.29
127.39
128.10
128.16
128.51
128.90
129.07
129.40
130.39
133.91
136.07
136.11
136.31
142.32
155.85
160.27
163.52
167.17
HN
Cbz
O
O
F
Ph
E-8b
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20



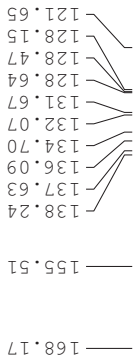


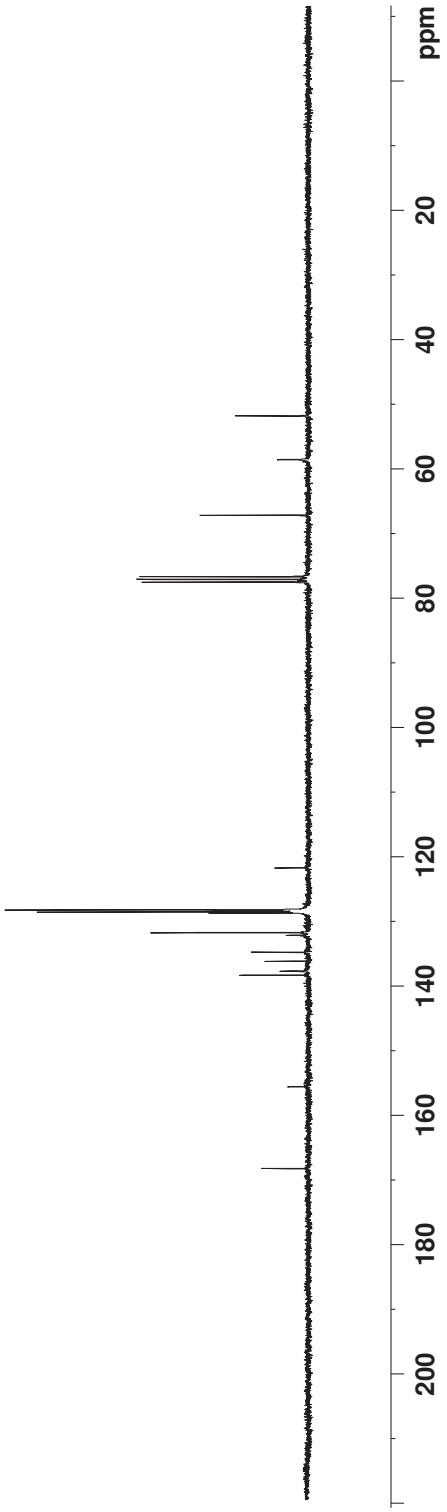
```
NAME                110526
EXPNO                    3
PROCNO                   1
Date_             20110526
Time                16.02
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     145
DS                       4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.81755818 sec
RG                       203
DW                  27.733 usec
DE                   6.50 usec
TE                   289.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                   9.70 usec
PL1                   0.00 dB
PL1W         29.38907051 W
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2         300.1312005 MHz
SI                   32768
SF           75.4677587 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                    1.40
```

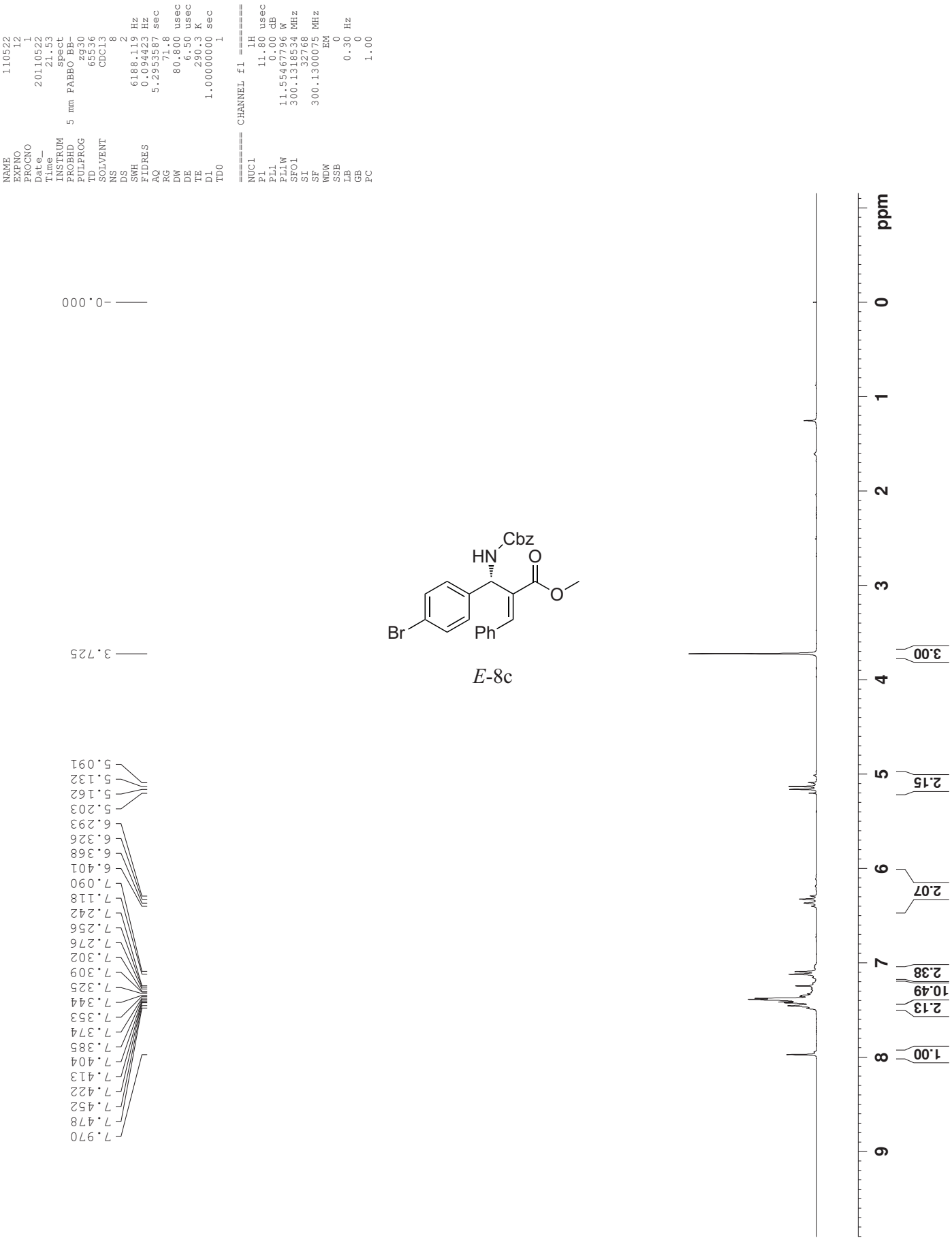




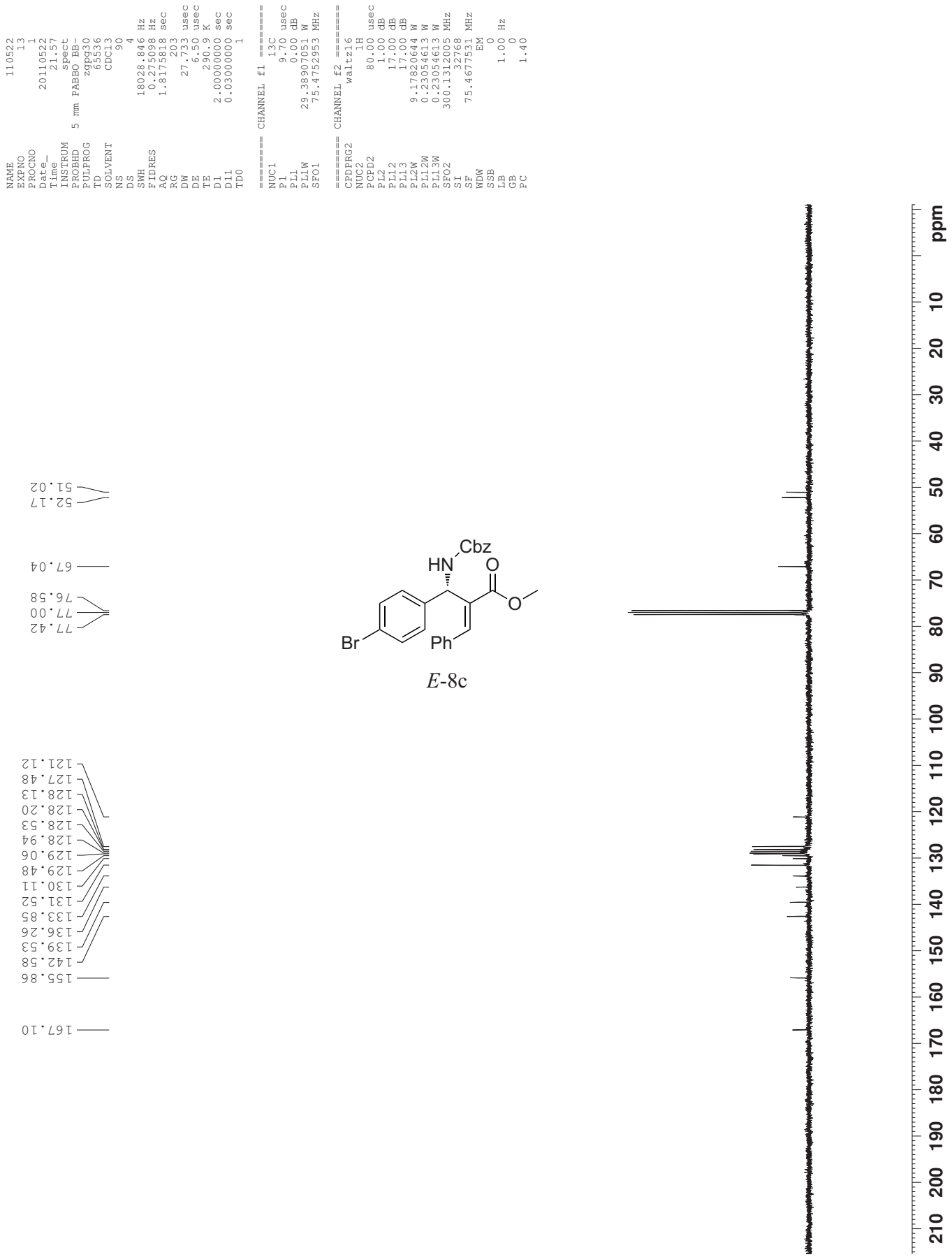
Z-8c



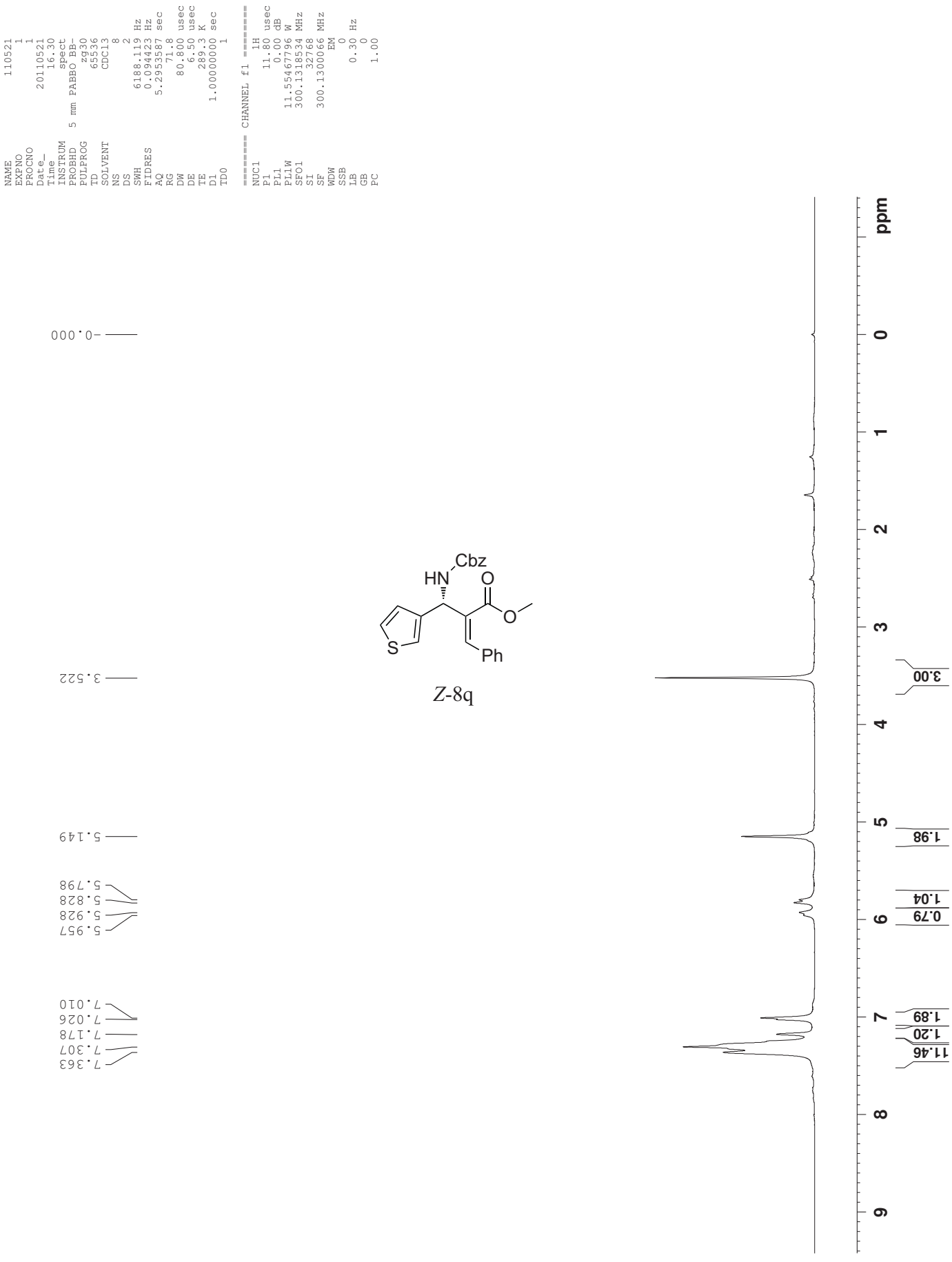



NAME 110522
EXPNO 12
PROCNO 1
Date_ 20110522
Time 21.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.29535387 sec
RG 71.8
DW 80.800 usec
DE 6.50 usec
TE 290.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.725
5.091
5.132
5.162
5.203
6.293
6.326
6.368
6.401
7.090
7.118
7.242
7.256
7.276
7.302
7.309
7.325
7.344
7.353
7.374
7.385
7.404
7.413
7.422
7.452
7.478
7.970
Cbz
HN
O
O
Br
Ph
ppm
0
1
2
3
4
5
6
7
8
9
3.00
2.15
2.07
2.38
10.49
2.13
1.00

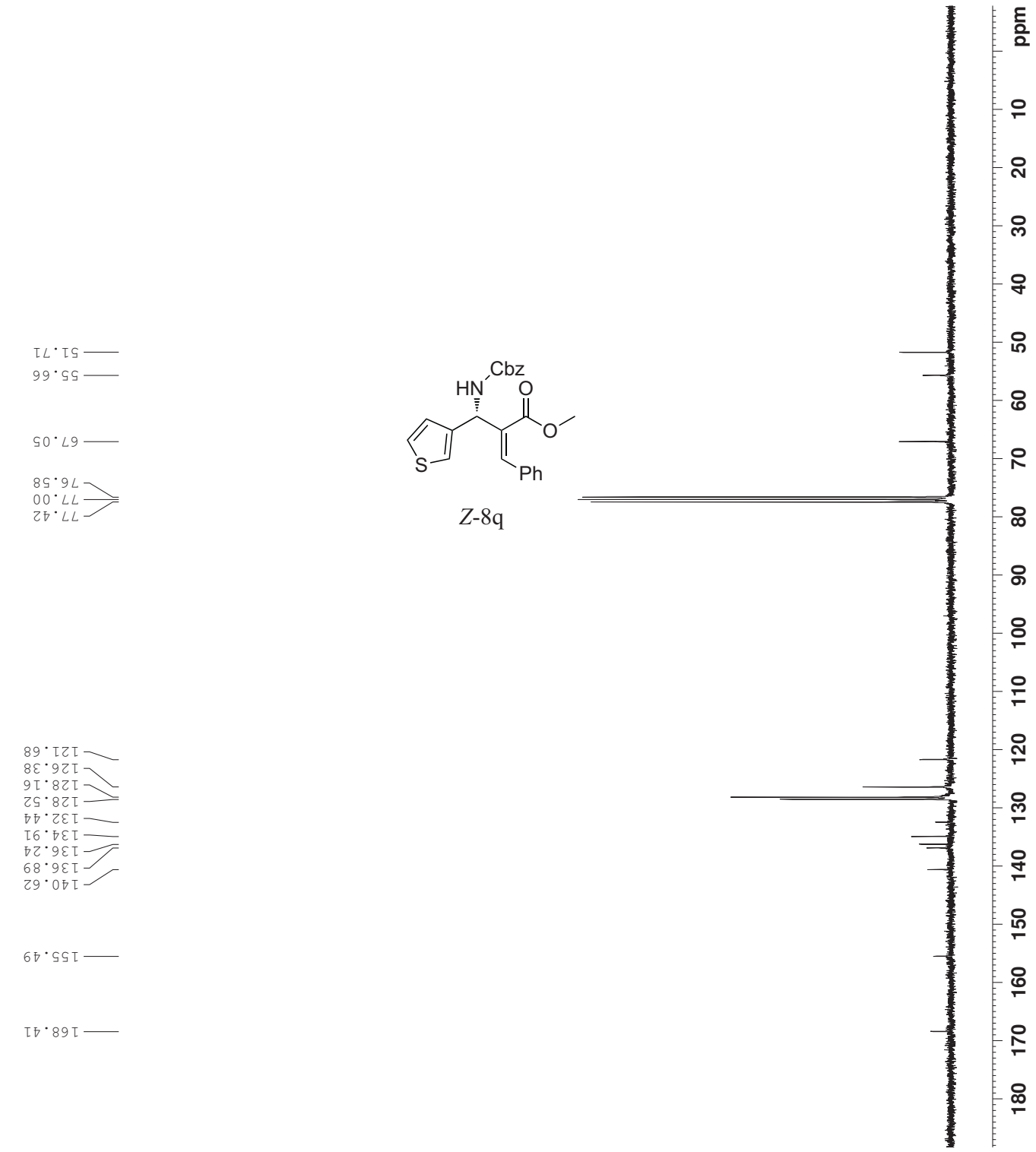
E-8c

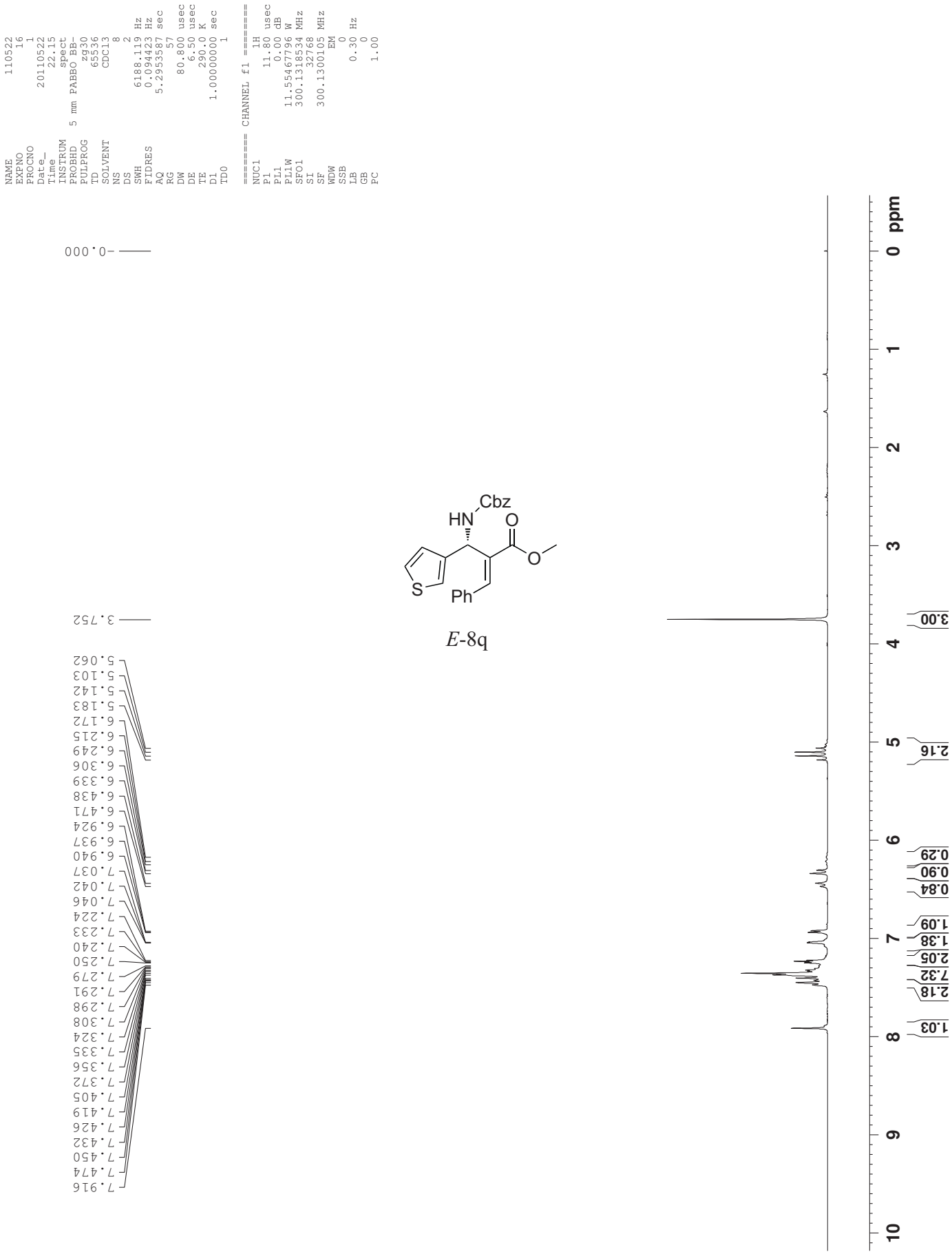

NAME 110522
EXPNO 13
PROCNO 1
Date_ 20110522
Time 21.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 90
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.02
52.17
67.04
76.58
77.00
77.42
121.12
127.48
128.13
128.20
128.53
128.94
129.06
129.48
130.11
131.52
133.85
136.26
139.53
142.58
155.86
167.10
Cbz
HN
O
O
Br
Ph
ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

E-8c

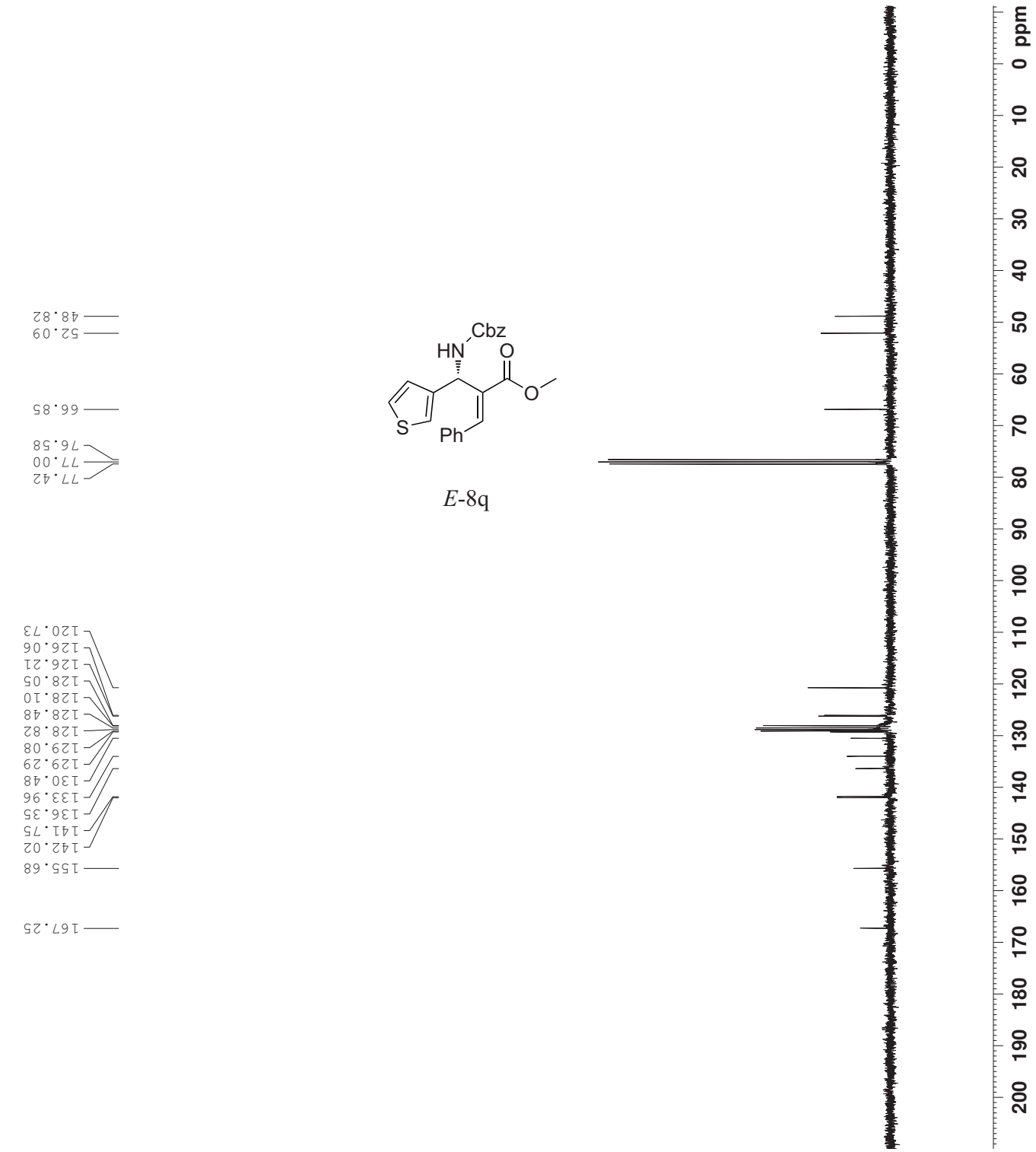

NAME 110521
EXPNO 1
PROCNO 1
Date_ 20110521
Time_ 16.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.295358 sec
RG 71.8
DW 80.800 usec
DE 6.50 usec
TE 289.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
3.522
5.149
5.798
5.828
5.928
5.957
7.010
7.026
7.178
7.307
7.363
Cbz
HN
O
O
S
Ph
ppm
0
1
2
3
4
5
6
7
8
9
3.00
1.98
1.04
0.79
1.89
1.20
11.46

Z-8q

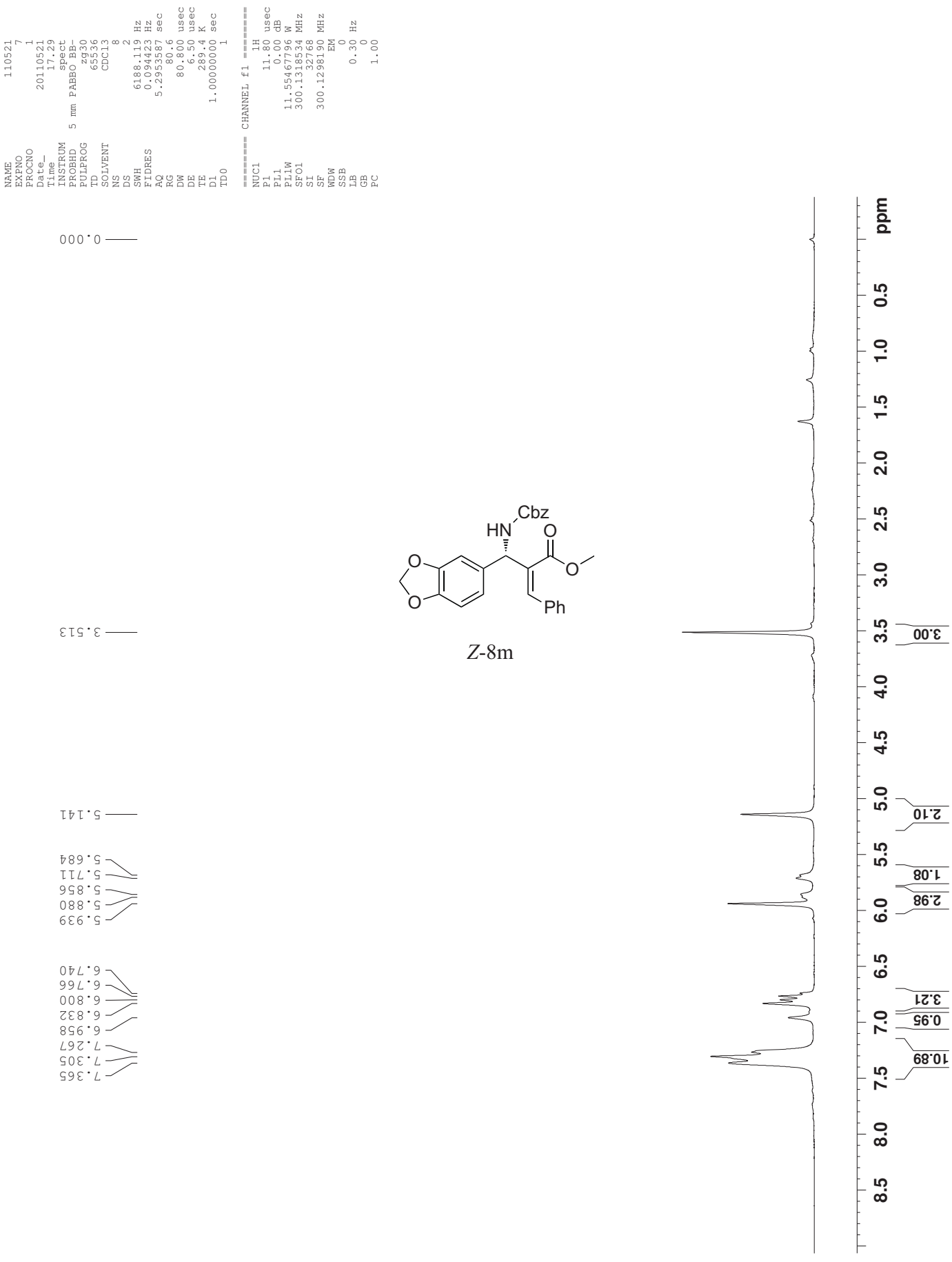

NAME 110521
EXPNO 2
PROCNO 1
Date_ 20110521
Time 16.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 308
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677532 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.71
55.66
67.05
76.58
77.00
77.42
121.68
126.38
128.16
128.52
132.44
134.91
136.24
136.89
140.62
155.49
168.41
HN
Cbz
O
O
S
Ph
Z-8q
ppm
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


NAME 110522
EXPNO 16
PROCNO 1
Date_ 20110522
Time 22.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 290.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
HN
O
O
S
Ph
ppm
3.00
2.16
0.84
0.90
0.29
1.09
1.38
2.05
7.32
2.18
1.03

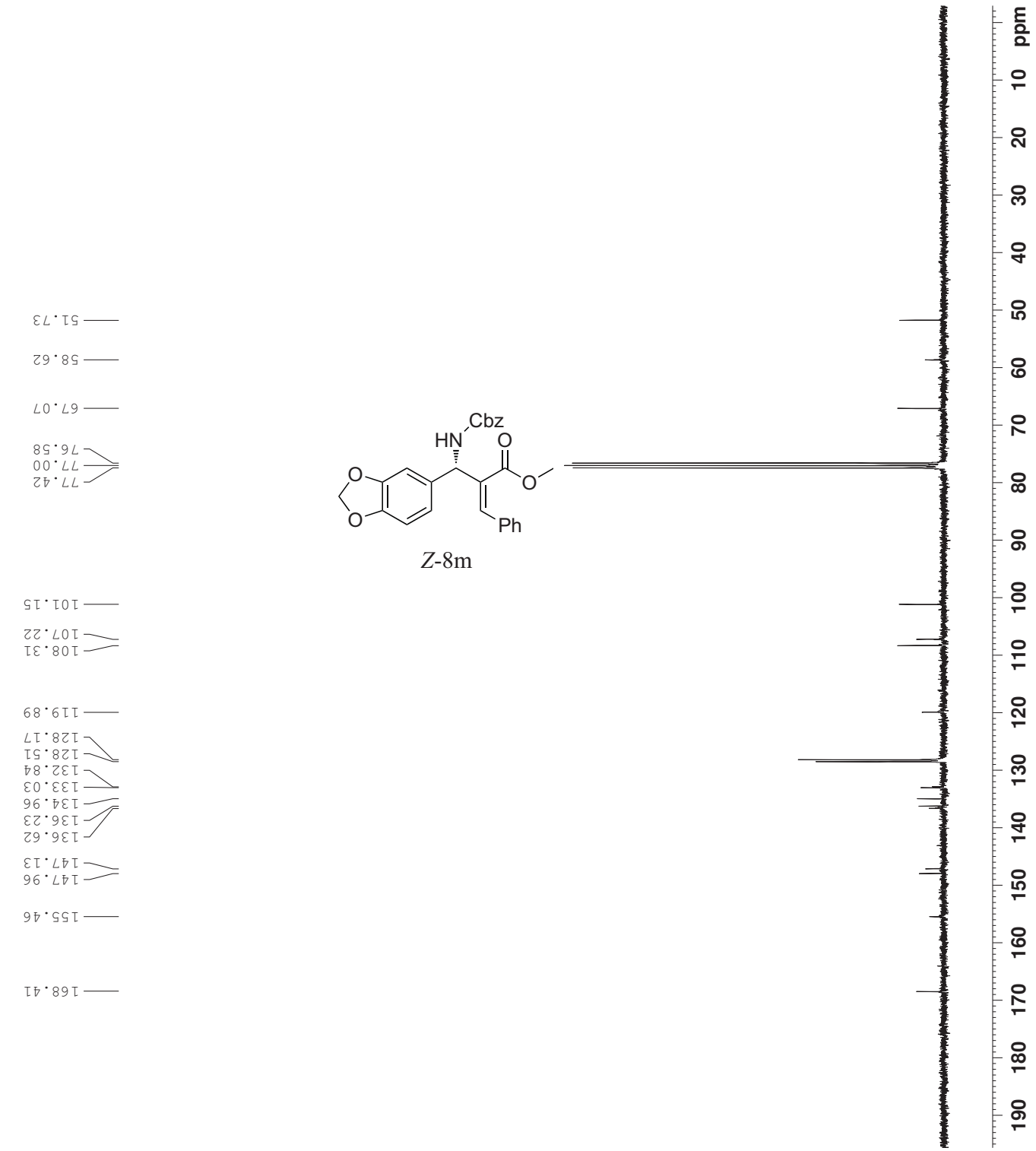
E-8q


NAME 110522
EXPNO 17
PROCNO 1
Date_ 20110522
Time 22.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
48.82
52.09
66.85
76.58
77.00
77.42
120.73
126.06
126.21
128.05
128.10
128.48
128.82
129.08
129.29
130.48
133.96
136.35
141.75
142.02
155.68
167.25
Cbz
HN
O
O
S
Ph
0 ppm

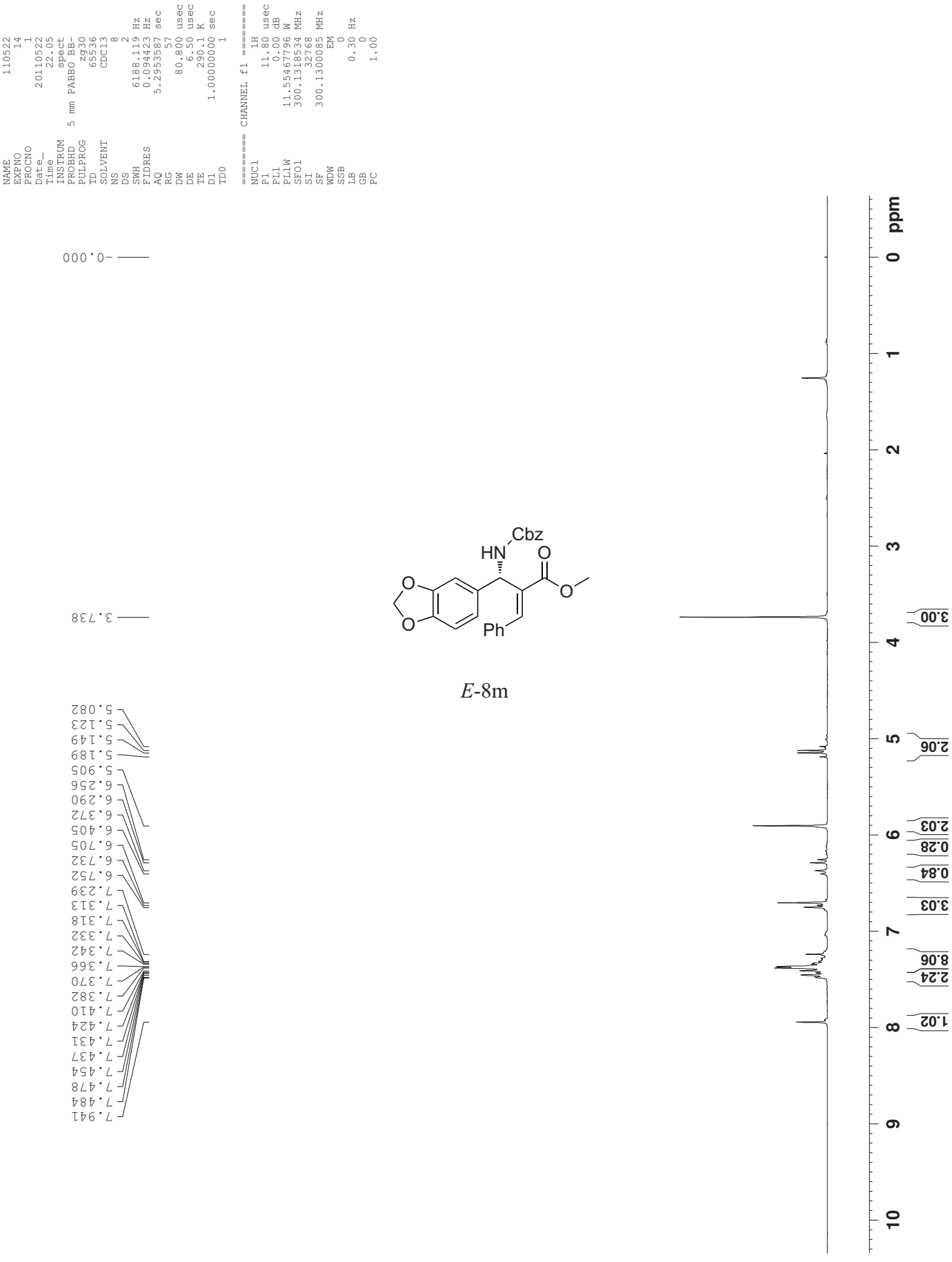
E-8q


NAME 110521
EXPNO 7
PROCNO 1
Date_ 20110521
Time_ 17.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 80.6
DW 80.800 usec
DE 6.50 usec
TE 289.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1298190 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.000
3.513
5.141
5.684
5.711
5.856
5.880
5.939
6.740
6.766
6.800
6.832
6.958
7.267
7.305
7.365
HN
Cbz
O
O
O
O
Ph
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.00
2.10
1.08
2.98
3.21
0.95
10.89

Z-8m

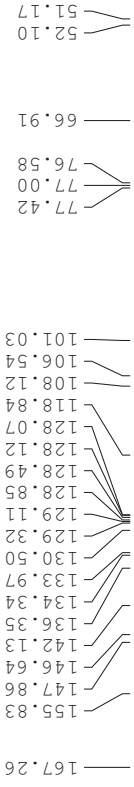

NAME 110521
EXPNO 8
PROCNO 1
Date_ 20110521
Time 17.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 403
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677059 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
51.73
58.62
67.07
76.58
77.00
77.42
101.15
107.22
108.31
119.89
128.17
128.51
132.84
133.03
134.96
136.23
136.62
147.13
147.96
155.46
168.41
HN
Cbz
O
O
O
O
Ph
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

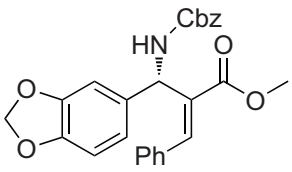
Z-8m



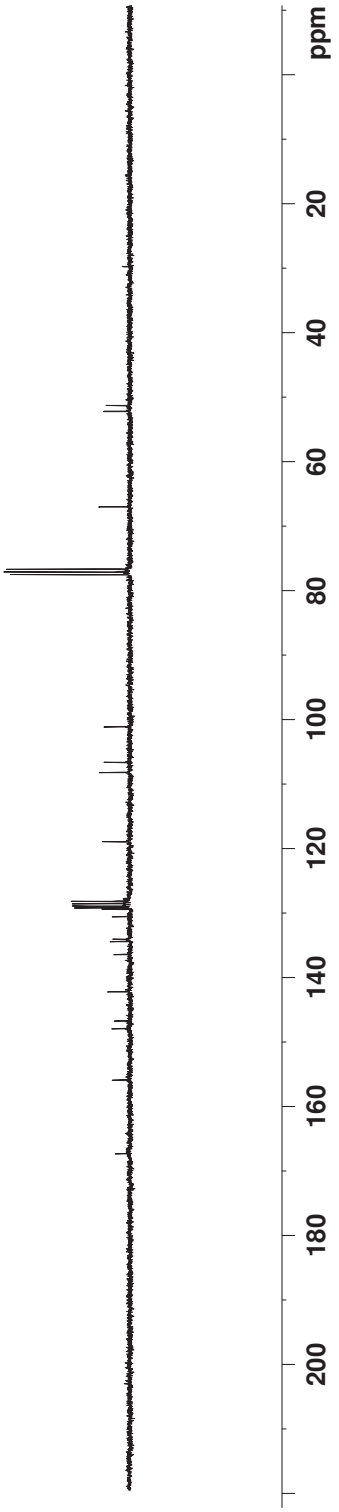
E-8m

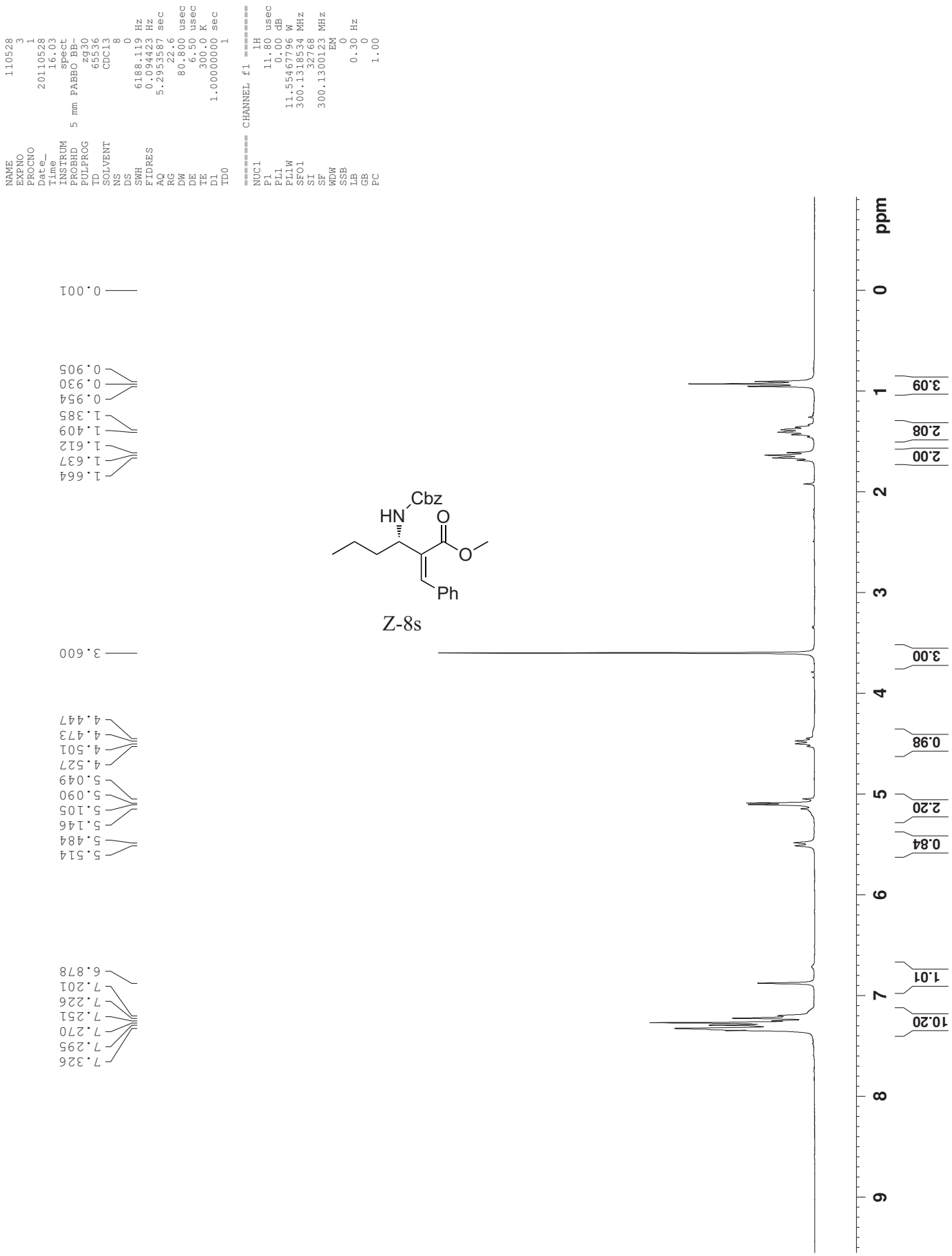
```
NAME                110522
EXPNO                   15
PROCNO                   1
Date_             20110522
Time                 22.09
INSTRUM               spect
PROBHD  5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                       4
DS                       4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175818 sec
RG                       203
DW                  27.733 usec
DE                   6.50 usec
TE                  290.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                   9.70 usec
PL1                  0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                   32768
SF              75.4677544 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

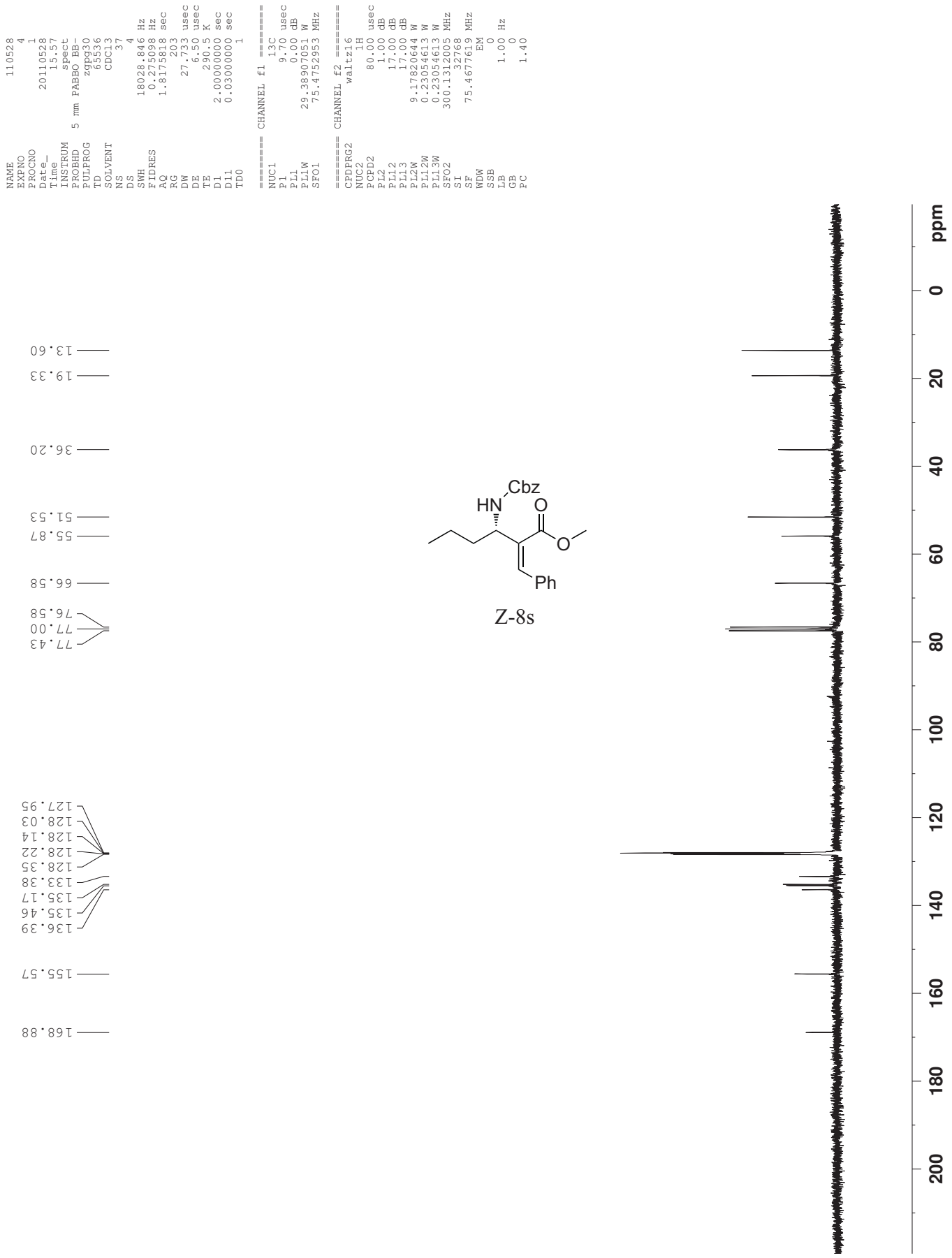


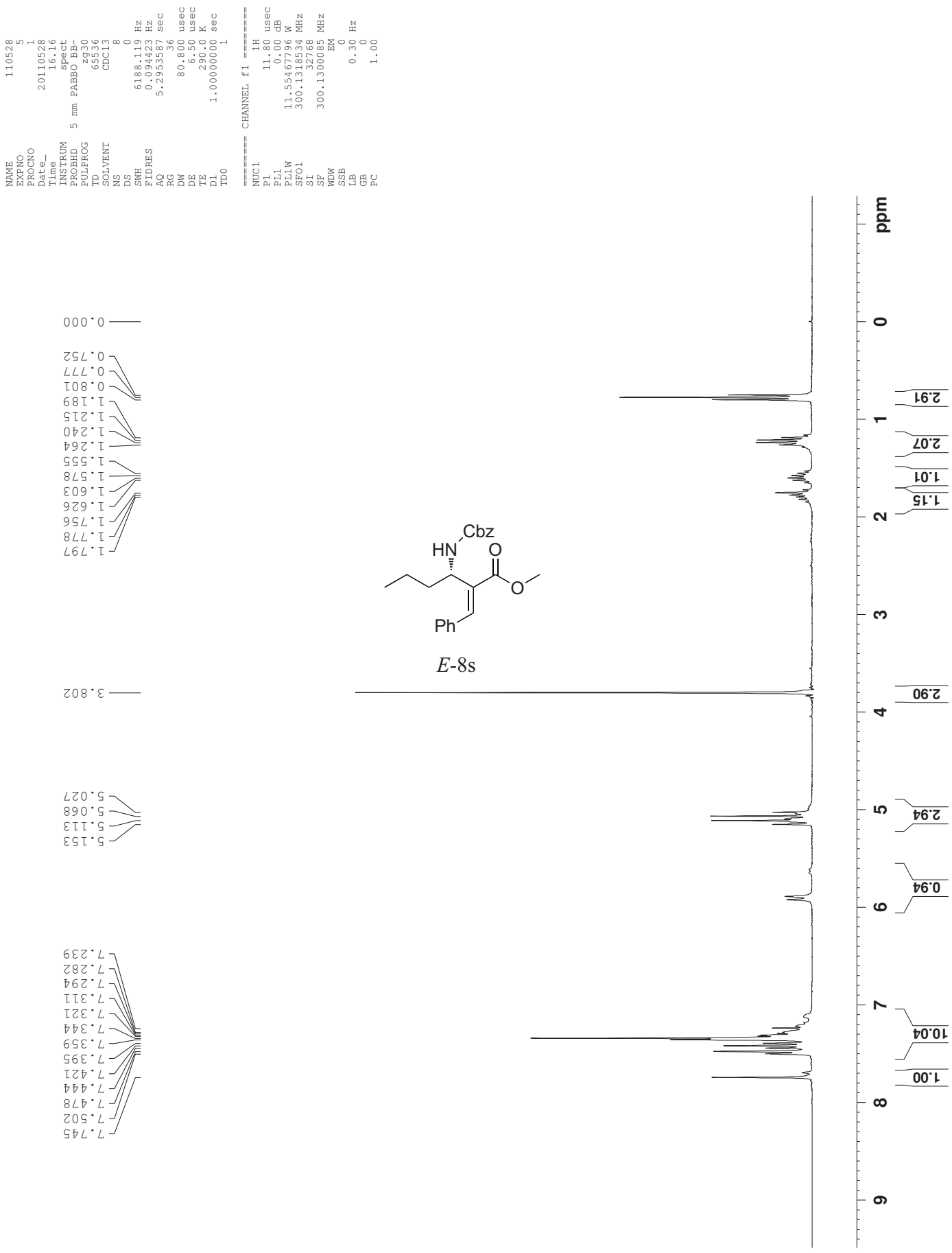


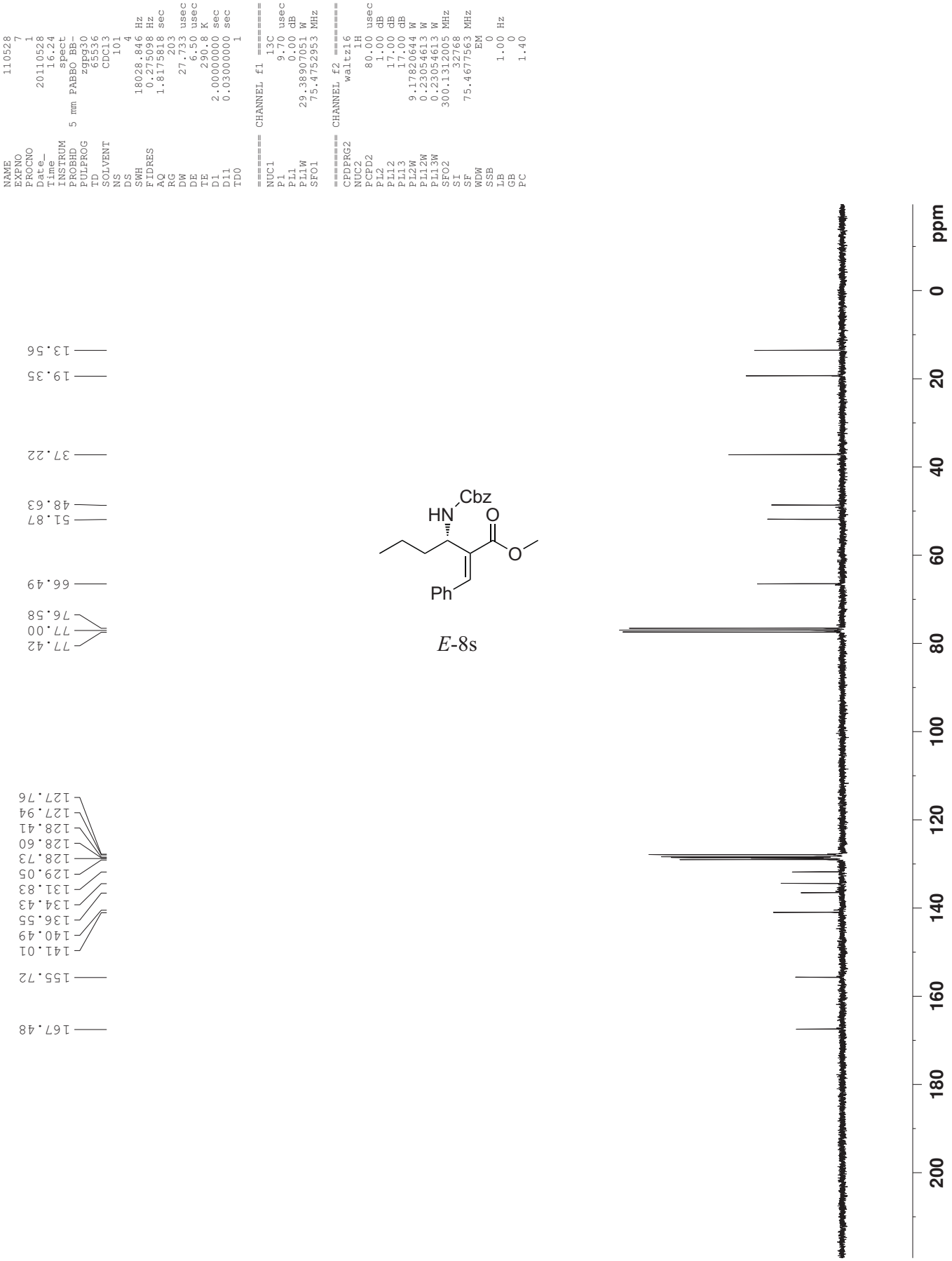
E-8m










NAME 110528
EXPNO 7
PROCNO 1
Date_ 20110528
Time 16.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 101
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 290.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677563 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.56
19.35
37.22
48.63
51.87
66.49
76.58
77.00
77.42
127.76
127.94
128.41
128.60
128.73
129.05
131.83
134.43
136.55
140.49
141.01
155.72
167.48
Cbz
HN
O
O
Ph
ppm
0
20
40
60
80
100
120
140
160
180
200

E-8s