SUPPORTING INFORMATION

Multicomponent Approach to the Synthesis of Oxidized Amides through Nitrile Hydrozirconation

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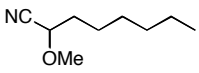
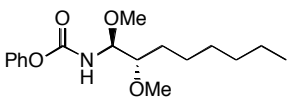
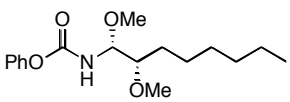
General Experimental Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were recorded on Bruker Avance 300 spectrometer at 300 MHz and 75 MHz or Bruker Avance 500 spectrometer at 500 MHz and 125 MHz if specified. The chemical shifts are given in parts per million (ppm) on the delta (δ) scale. The solvent peak was used as a reference value, for ^{1}H NMR: $CDCl_3$ = 7.27 ppm, CD_3OD = 3.31, for ^{13}C NMR: $CDCl_3$ = 77.23, CD_3OD = 49.00. Data are reported as follows: (s = singlet; d = doublet; t = triplet; q = quartet; sept = septet; sext = sextet; dd = doublet of doublets; ddd = doublet of doublet of doublets; dt = doublet of triplets; td = triplet of doublets; dtd = doublet of triplet of doublets; br = broad). High resolution and low resolution mass spectra were recorded on a VG 7070 spectrometer. Infrared (IR) spectra were collected on a Mattson Cygnus 100 spectrometer. Samples for IR were prepared as a thin film on a NaCl plate by dissolving the compound in CH_2Cl_2 and then evaporating the CH_2Cl_2. Optical rotations were measured on a Perkin-Elmer 241 polarimeter at ambient temperature. Tetrahydrofuran and diethyl ether were distilled from sodium and benzophenone. Methylene chloride and benzene was distilled under N_2 from CaH_2. All acid chlorides were freshly distilled prior to use. Analytical TLC was performed on E. Merck pre-coated (25 mm) silica gel 60F-254 plates. Visualization was done under UV (254 nm). Flash chromatography was done using ICN SiliTech 32-63 60 Å silica gel. Reagent grade ethyl acetate, diethyl ether and hexanes (commercial mixture) were purchased from EM Science and used as is for chromatography. All reactions were performed in oven or flame-dried glassware under argon with magnetic stirring unless otherwise noted. All the reactions related to Schwartz reagent were performed under argon unless otherwise specified.



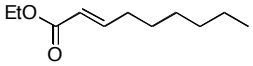
2-Methoxyoctanenitrile (3)
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.04 (t, *J* = 6.6 Hz, 1H), 3.49 (s, 3H), 1.86-1.82 (m, 2H), 1.52-1.46 (m, 2H), 1.37-1.27 (m, 6H), 0.90 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 118.4, 70.9, 58.2, 33.6, 31.7, 28.9, 24.9, 22.7, 14.2. The ^{1}H NMR spectral data are consistent with those reported in the literature.[1]





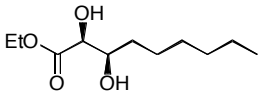
(±)-Phenyl (1 *R*,2*R*)-1,2-dimethoxyoctylcarbamate (5) and **(±)-phenyl (1*S*,2*R*)-1,2-dimethoxyoctylcarbamate (4)**
To a solution of methoxynitrile **4** (70 mg, 0.45 mmol) in CH_2Cl_2 (43.5 mL) was added $Cp_2Zr(H)Cl$ (140 mg, 0.541 mmol). The reaction was stirred for 15 min, then cooled to 0 °C and phenyl chloroformate (79 μL, 0.63 mmol) was added dropwise. The cold bath was removed and the mixture was stirred for 10 min. After that time, the flask was cooled to 0 °C and phenyl chloroformate (56 μL, 0.45 mmol) was added. The mixture was stirred at room temperature for 15 min and then cooled to 0 °C. A solution of MeOH (0.36 ml, 9.0 mmol) in CH_2Cl_2 (0.6 mL) was added dropwise. The reaction was stirred for 15 min at 0 °C and then quenched with saturated $NaHCO_3$ (25 mL). The mixture was extracted with CH_2Cl_2 (4 x 20 mL) and the combined organic extracts were dried (Na_2SO_4) and

concentrated. The residue was purified by column chromatography (6% - 20% EtOAc in hexanes containing 0.5% Et_3N) to give the desired products (77.2 mg, 55.3%) as a colorless oil in a 2.4:1.0 diastereomeric ratio. Further purification by column chromatography (8% - 14% EtOAc in hexanes containing 0.5% Et_3N) yielded pure samples. For the faster eluting *anti*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (app t, *J* = 7.7 Hz, 2H), 7.24-7.12 (m, 3H), 5.90 (d, *J* = 9.8 Hz, 1H), 4.88 (d, *J* = 10.0 Hz, 1H), 3.59-3.49 (m, 1H), 3.52 (s, 3H), 3.44 (s, 3H), 1.55-1.27 (m, 10H), 0.90 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 155.2, 151.0, 129.5, 125.6, 121.7, 85.6, 82.4, 59.7, 56.0, 31.9, 31.4, 29.5, 25.6, 22.8, 14.3; IR (neat) 3322, 2930, 2857, 1747, 1515, 1487, 1334, 1206, 1103, 1025, 952, 738; HRMS (ESI): *m*/*z* calcd for $C_{17}H_{27}NO_4Na$ [M^++Na] 332.1838, found 332.1830. For the slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.34 (m, 2H), 7.29-7.22 (m, 3H), 5.82 (d, *J* = 9.7 Hz, 1H), 5.00 (dd, *J* = 10.0, 2.9 Hz, 1H), 3.41 (s, 3H), 3.40 (s, 3H), 3.18 (dt, *J* = 6.8, 2.9 Hz, 1H), 1.62-1.55 (m, 2H), 1.40-1.24 (m, 8H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.9, 151.0, 129.5, 125.7, 121.7, 82.9, 82.6, 58.4, 56.5, 31.9, 29.7, 29.0, 25.6, 22.8, 14.3; IR (neat) 3324, 2928, 2857, 1747, 1523, 1488, 1356, 1209, 1086, 954 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{17}H_{27}NO_4Na$ (M^++Na) 332.1838, found 332.1841.



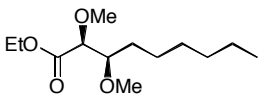
(*E*)-Ethyl non-2-enoate

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.97 (td, *J* = 15.6, 7.0 Hz, 1H), 5.81 (td, *J* = 15.7, 1.5 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.20 (qd, *J* = 7.0, 1.5 Hz, 2H), 1.50-1.41 (m, 2H), 1.36-1.27 (m, 9H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0, 149.7, 121.4, 60.3, 32.4, 31.8, 29.0, 28.2, 22.8, 14.5, 14.3.



(2*S*,3*R*)-Ethyl 2,3-dihydroxynonanoate

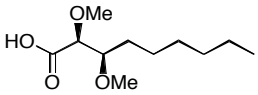
To a mixture of AD-mix-β in ^{t}BuOH/H_2O (20 mL, 1:1, v/v) at 0 °C was added $CH_3SO_2NH_2$ (0.190 g, 2.00 mmol) followed by a solution of enoate (0.368 g, 2.00 mmol) in ^{t}BuOH (0.5 mL). The mixture was stirred at 0 °C for 6 h and then at room temperature for 10 h. The mixture was cooled to 0 °C and quenched with aqueous Na_2SO_3 (10%, 30 mL). After stirring at 0 °C for 1 h, the mixture was extracted with CH_2Cl_2 (3 x 30 mL). The organic extracts were dried ($MgSO_4$) and concentrated. The residue was purified by column chromatography (30% - 40% EtOAc in hexanes) to give the diol (0.402 g, 92.0%) as a white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.29 (q, *J* = 7.1 Hz, 2H), 4.08 (dd, *J* = 5.3, 2.0 Hz, 1H), 3.88 (dtd, *J* = 8.9, 6.9, 2.1 Hz, 1H), 3.12 (d, *J* = 5.3 Hz, 1H), 1.98 (d, *J* = 9.2 Hz, 1H), 1.64-1.58 (m, 2H), 1.52-1.44 (m, 1H), 1.39-1.25 (m, 10H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.9, 73.2, 72.7, 62.3, 34.0, 32.0, 29.4, 25.9, 22.8, 14.4, 14.3; IR (neat) 3377, 2925, 2854, 1737, 1462, 1294, 1136, 1099, 1072 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{11}H_{22}O_4Na$ (M^++Na) 241.1416, found 241.1420; $[\alpha]_D^{25}$ = +12.6 ($CHCl_3$, *c* 0.98).



(2*S*,3*R*)-Ethyl 2,3-dimethoxynonanoate

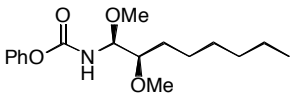
To a solution of the diol (170 mg, 0.779 mmol) in CH_2Cl_2 (4.0 mL) were added Ag_2O (271 mg, 1.17 mmol) and MeI (0.22 mL, 3.5 mmol). The reaction was stirred at reflux for 10 h, and Ag_2O (271 mg, 1.17 mmol) and MeI (0.22 mL, 3.5 mmol) were added sequentially. After 12 h, Ag_2O (271 mg, 1.17 mmol) and MeI (0.22 mL, 3.50 mmol) were added. The mixture was stirred at reflux for another 6 h, then filtered through Celite. The residue was washed with CH_2Cl_2 (30 mL), the combined filtrate was concentrated, and the resulting residue was purified by column chromatography (5% - 15% EtOAc in hexanes) to give the desired product (59.4 mg, 31.0%) as a colorless oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.32-4.21 (m, 2H), 3.78 (d, *J* = 4.1 Hz, 1H), 3.51 (dt, *J* = 6.5, 4.1

Hz, 1H), 3.44 (s, 3H), 3.39 (s, 3H), 1.61-1.54 (m, 2H), 1.34-1.26 (m, 11H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 82.7, 82.0, 61.1, 59.2, 58.6, 32.0, 30.1, 29.6, 25.8, 22.8, 14.5, 14.3; IR (neat) 2927, 1747, 1464, 1261, 1190, 1143, 1105, 1031 cm^{-1}; HRMS (ESI): m/z calcd for $C_{13}H_{26}O_4Na$ (M^++Na) 269.1729, found 269.1713; $[\alpha]_D^{25}$ = –29.7 ($CHCl_3$, c 0.63).



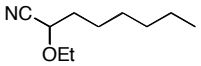
(2*S*,3*R*)-2,3-Dimethoxynonanoic acid (9)

To a solution of the ethyl ester (40 mg, 0.16 mmol) in 1,2-dimethoxyethane/H_2O (2.8 mL, 4:1, v/v) was added LiOH·H_2O (14 mg, 0.33 mmol). After 3 and 4 h, LiOH·H_2O (7 mg, 0.16 mmol) was added, respectively. The reaction was stirred for another 3 h, then quenched with aqueous HCl (0.5 N, ~1.0 mL) to pH~1.5 and extracted with Et_2O (5 x 10 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (30% EtOAc in hexanes followed by 50% MeOH in EtOAc) to give the unreacted ester (7.6 mg, 19.0%) and carboxylic acid (28.1 mg, 79.4%) as a white sticky solid: ^{1}H NMR (300 MHz, CD_3OD) δ 3.66 (d, J = 3.0 Hz, 1H), 3.54 (dt, J = 6.7, 3.1 Hz, 1H), 3.42 (s, 3H), 3.41 (s, 3H), 1.69-1.57 (m, 2H), 1.46-1.29 (m, 8H), 0.91 (t, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, CD_3OD) δ 178.2, 84.9, 83.9, 59.4, 58.9, 32.9, 31.2, 30.5, 26.8, 23.7, 14.4 cm^{-1}; IR (neat) 3401, 2926, 2856, 1618, 1418, 1194, 1091; HRMS (ESI): m/z calcd for $C_{11}H_{22}O_4Na$ (M^++Na) 241.1416, found 241.1407; $[\alpha]_D$ = –26.0 (CH_3OH, c 0.77).



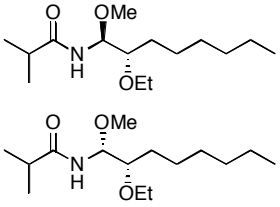
Phenyl (1*S*,2*R*)-1,2-dimethoxyoctylcarbamate (4)

To a stirred solution of the carboxylic acid (15 mg, 70 μmol) in benzene (2.0 mL) was added Et_3N (0.12 ml, 0.85 mmol) followed by diphenyl phosphoryl azide (61 μL, 0.28 mmol). After 2 h, diphenylphosphoryl azide (30 μL, 0.14 mmol) was added. The reaction was stirred for 2 h, then quenched with water (10 mL) and extracted with Et_2O (3 x 20 mL). The organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (5% - 15% EtOAc in hexanes) to give carbamate (11.4 mg, 52.3%) as a colorless oil: $[\alpha]_D^{25}$ = –3.8 ($CHCl_3$, c 0.52). No other diastereomer was observed.



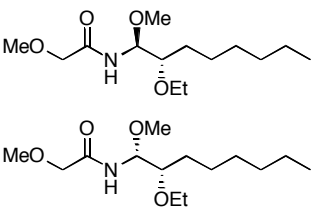
2-Ethoxyoctanenitrile (10)

To a mixture of heptanal (4.00 g, 35.0 mmol), absolute EtOH (80 ml), $(EtO)_3CH$ (5.8 mL, 35.0 mmol) and activated 4Å molecular sieves (4.00 g) at 0 °C was added concentrated H_2SO_4 (2.0 ml) dropwise and the mixture was stirred at room temperature overnight. After that time, the reaction mixture was concentrated to ~30 mL and slowly poured onto a cold saturated $NaHCO_3$ solution (80 mL) at 0 °C. The resulting mixture was filtered through Celite. The filtrate was extracted with CH_2Cl_2 (3 x 80mL) and the extracts were dried (Na_2SO_4) and concentrated. The resulting residue was dissolved in CH_2Cl_2 (70 mL), and $BiBr_3$ (1.57 g, 3.50 mmol) and TMSCN (5.60 ml, 42.0 mmol) were added sequentially. The reaction was stirred overnight, then quenched with saturated $NaHCO_3$ solution (50 mL)/water (20 mL) and extracted with CH_2Cl_2 (3 x 100mL). The combined extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (2% - 5% EtOAc in hexanes) to give the ethoxy cyanide (4.51 g, 76.0%) as a colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.11 (t, J = 6.6 Hz, 1H), 3.82 (qd, J = 8.8, 6.9 Hz, 1H), 3.51 (qd, J = 8.9, 7.0 Hz, 1H), 1.87-1.80 (m, 2H), 1.52-1.44 (m, 2H), 1.38-1.30 (m, 6H), 1.26 (t, J = 7.0 Hz, 3H), 0.89 (t, J = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 118.9, 69.0, 66.4, 33.8, 31.7, 28.9, 24.9, 22.7, 15.0, 14.2; IR (neat) 2957, 2930, 2860, 1468, 1335, 1126, 1108, 735 cm^{-1}; HRMS (EI): m/z calcd for $C_{10}H_{19}NO$ (M^+) 169.1467, found 169.1474.



Representative procedure for the preparation of acyl aminals: **(±)-*N*-((1*R*,2*R*)-2-Ethoxy-1-methoxyoctyl)isobutyramide** (**11**) and **(±)-*N*-((1*S*,2*R*)-2-ethoxy-1-methoxyoctyl)isobutyramide** (**12**)

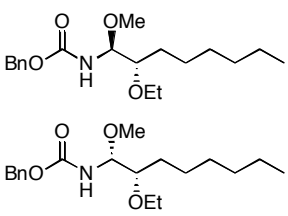
To a solution of ethoxynitrile **11** (100.0 mg, 0.591 mmol) in CH_2Cl_2 (4.5 mL) was added $Cp_2Zr(H)Cl$ (229 mg, 0.886 mmol). The reaction was stirred for 15 min, then cooled to 0 °C and isobutyroyl chloride (94 μL, 0.89 mmol) was added dropwise. The mixture was stirred for 15 min at 0 °C and MeOH (1.0 mL, 24 mmol) was added dropwise. The reaction was stirred for 15 min at 0 °C and quenched with AcOH (2.0 mL)/water (20 mL). The mixture was extracted with CH_2Cl_2 (3 x 25 mL) and the combined organic extracts were washed with saturated $NaHCO_3$ (15 mL), dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (15% - 30% EtOAc in hexanes) to give the desired product (121 mg, 75.1%) as a white solid in a 2.3:1.0 diastereomeric ratio. Further purification (15% - 30% EtOAc in hexanes) yielded pure samples. For faster eluting *anti*-product **12**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.20 (d, *J* = 9.5 Hz, 1H), 5.01 (dd, *J* = 9.7, 1.4 Hz, 1H), 3.74 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.54 (qd, 9.4, 7.1 Hz, 1H), 3.47-3.43 (m, 1H), 3.29 (s, 3H), 2.40 (sept, *J* = 6.9 Hz, 1H), 1.37-1.23 (m, 10H), 1.18-1.14 (m, 9H), 0.84 (app t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.9, 82.6, 80.6, 67.3, 55.8, 36.1, 31.9, 31.8, 29.4, 25.6, 22.7, 19.8, 19.7, 15.8, 14.2; IR (neat) 3271, 2965, 2920, 1653, 1540, 1467, 1233, 1113, 1101 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{14}H_{28}NO_2$ (M^+-CH_3O) 242.2120, found 242.2123. For slower eluting *syn*-product **13**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.20 (d, *J* = 9.7 Hz, 1H), 5.17 (dd, *J* = 9.8, 2.9 Hz, 1H), 3.66 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.46 (qd, *J* = 9.3, 7.0 Hz, 1H), 3.36 (s, 3H), 3.24 (dt, *J* = 6.8, 2.9 Hz, 1H), 2.42 (sept, *J* = 6.9 Hz, 1H), 1.63-1.55 (m, 2H), 1.40-1.25 (m, 10H), 1.23-1.17 (m, 9H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.5, 81.0, 80.0, 66.1, 56.4, 36.2, 31.9, 29.8, 29.7, 25.6, 22.8, 19.9, 19.7, 15.8, 14.2; IR (neat) 3273, 2971, 2921, 1651, 1538, 1467, 1154, 1103, 1072 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{14}H_{28}NO_2$ (M^+-CH_3O) 242.2120, found 242.2119.



(±)-*N*-((1*R*,2*R*)-2-Ethoxy-1-methoxyoctyl)-2-methoxyacetamide (**13**) and **(±)-*N*-((1*S*,2*R*)-2-ethoxy-1-methoxyoctyl)-2-methoxyacetamide**

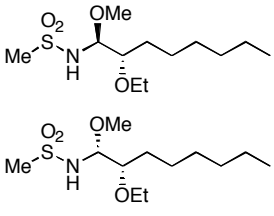
The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (100.0 mg, 0.591 mmol), CH_2Cl_2 (4.5 mL), $Cp_2Zr(H)Cl$ (168 mg, 0.650 mmol), methoxyacetyl chloride (65 μL, 0.71 mmol) MeOH (1.0 ml, 24 mmol). The reaction was quenched with 1 N HCl (2.0 mL)/water (15 mL) and extracted with CH_2Cl_2 (3 x 25 mL). The combined organic extracts were washed with saturated $NaHCO_3$ (15 mL), dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (20% - 40% EtOAc in hexanes) to give the desired product (112 mg, 68.7%) as a colorless oil in a 1.7:1.0 diastereomeric ratio. Further purification (20% - 40% EtOAc in hexanes) yielded pure samples. For faster eluting *anti*-product **14**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18 (d, *J* = 9.8 Hz, 1H), 5.06 (dd, *J* = 10.0, 1.5 Hz, 1H), 3.98 (d, *J* = 15.2 Hz, 1H), 3.90 (d, *J* = 15.2 Hz, 1H), 3.76 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.56 (qd, *J* = 9.3, 7.0 Hz, 1H), 3.49-3.45 (m, 1H), 3.43 (s, 3H), 3.33 (s, 3H), 1.45-1.25 (m, 10H), 1.18 (t, *J* = 7.0 Hz, 3H), 0.86 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 82.4, 80.5, 72.0, 67.3, 59.4, 56.1, 31.9, 31.8, 29.4, 25.7, 22.8, 15.8, 14.2; IR (neat) 3413, 2930, 2858, 1695, 1506, 1113 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{13}H_{26}NO_3$ (M^+-CH_3O) 244.1913, found 244.1925. For slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 10.0 Hz, 1H), 5.18

(dd, J = 10.1, 3.0 Hz, 1H), 3.98 (d, J = 15.3 Hz, 1H), 3.92 (d, J = 15.3 Hz, 1H), 3.66 (qd, J = 9.2, 7.0 Hz, 1H), 3.50 (qd, J = 9.2, 7.0 Hz, 1H), 3.43 (s, 3H), 3.36 (s, 3H), 3.26 (dt, J = 6.8, 3.0 Hz, 1H), 1.63-1.55 (m, 2H), 1.41-1.29 (m, 8H), 1.21 (t, J = 7.0 Hz, 3H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 81.0, 79.8, 72.0, 66.5, 59.4, 56.5, 31.9, 29.9, 29.6, 25.7, 22.8, 15.7, 14.3; IR (neat) 3417, 2928, 2858, 1686, 1510, 1112, 1078 cm^{-1}; HRMS (EI): m/z calcd for $C_{13}H_{26}NO_3$ (M^+-CH_3O) 244.1913, found 244.1917.



(±)-Benzyl (1 *R*,2*R*)-2-ethoxy-1-methoxyoctylcarbamate (**14**) and **(±)-benzyl (1*S*,2*R*)-2-ethoxy-1-methoxyoctylcarbamate**

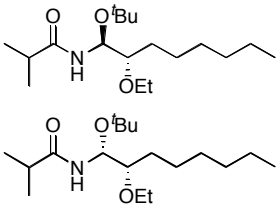
The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (60.0 mg, 0.354 mmol), CH_2Cl_2 (3.5 mL), $Cp_2Zr(H)Cl$ (110.0 mg, 0.425 mmol). After completion of the hydrozirconation, the reaction mixture was cooled to 0 °C and benzyl chloroformate (71 μL, 0.500 mmol) was added dropwise. The cold bath was removed and the mixture was stirred for 10 min. After that time, the flask was cooled to 0 °C and benzyl chloroformate (50 μL, 0.35 mmol) was added. The mixture was stirred at room temperature for 30 min and then cooled to 0 °C. A solution of MeOH (0.28 ml, 7.1 mmol) in CH_2Cl_2 (0.5 mL) was added dropwise. The reaction was stirred for 10 min at 0 °C and then quenched with saturated $NaHCO_3$ (20 mL). The mixture was extracted with CH_2Cl_2 (3 x 20 mL) and the combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (5% - 20% EtOAc in hexanes containing 0.5% Et_3N) to give the desired product (76.3 mg, 63.8%) as a colorless oil in a 1.5:1.0 diastereomeric ratio. Further purification (10% - 13% EtOAc in hexanes containing 0.5% Et_3N) yielded pure materials. For faster eluting *anti*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.30 (m, 5H), 5.66 (d, J = 9.8 Hz, 1H), 5.14 (s, 2H), 4.82 (dd, J = 9.9, 1.0 Hz, 1H), 3.74 (qd, J = 9.3, 7.0 Hz, 1H), 3.56 (qd, J = 9.2, 7.0 Hz, 1H), 3.48-3.44 (m, 1H), 3.37 (s, 3H), 1.46-1.28 (m, 10H), 1.17 (t, J = 7.0 Hz, 3H), 0.89 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.8, 136.6, 128.7, 128.4, 128.2, 85.6, 80.6, 67.4, 67.1, 55.7, 31.9, 29.5, 25.7, 22.8, 15.9, 14.3; IR (neat) 3337, 2929, 2858, 1731, 1497, 1456, 1326, 1216, 1107, 966, 735 cm^{-1}; HRMS (ESI): m/z calcd for $C_{19}H_{31}NO_4Na$ (M^++Na) 360.2151, found 360.2148. For slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.31 (m, 5H), 5.54 (d, J = 10.0 Hz, 1H), 5.15/5.14 (two s, 2H), 4.94 (dd, J = 10.1, 2.9 Hz, 1H), 3.64 (qd, J = 9.2, 7.0 Hz, 1H), 3.48 (qd, J = 9.2, 7.0 Hz, 1H), 3.38 (s, 3H), 3.28 (dt, J = 6.8, 2.9 Hz, 1H), 1.63-1.52 (m, 2H), 1.41-1.26 (m, 8H), 1.20 (t, J = 7.0 Hz, 3H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.6, 136.5, 128.8, 128.5, 128.4, 82.9, 81.1, 77.4, 67.2, 66.2, 56.3, 31.9, 29.7, 29.6, 25.7, 22.8, 15.8, 14.3; IR (neat) 3334, 2928, 2858, 1729, 1501, 1455, 1232, 1097, 737 cm^{-1}; HRMS (ESI): m/z calcd for $C_{19}H_{31}NO_4Na$ (M^++Na) 360.2151, found 360.2149.



(±)-*N*-(2-Ethoxy-1-methoxyoctyl)methanesulfonamide (15)

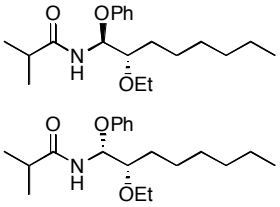
The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (100.0 mg, 0.591 mmol), CH_2Cl_2 (4.5 mL), $Cp_2Zr(H)Cl$ (228 mg, 0.886 mmol). After addition of methanesulfonic anhydride (144 mg, 0.827 mmol), The mixture was stirred for 2 min at 0 °C and MeOH (1.0 mL, 23.6 mmol) was added dropwise. The reaction was stirred for 10 min at 0 °C and quenched with saturated $NaHCO_3$ (15 mL). The mixture was extracted with CH_2Cl_2 (3 x 20 mL) and the combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (20% - 30% EtOAc in hexanes) to give the desired product (40.8 mg, 24.5%) as a colorless oil in a 2.4:1.0

diastereomeric ratio: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.42 (d, *J* = 9.4 Hz, 71% of 1H), 5.22 (d, *J* = 9.5 Hz, 29% of 1H), 4.64 (dd, *J* = 9.5, 3.1 Hz, 29% of 1H), 4.48 (dd, *J* = 9.4, 2.4 Hz, 71% of 1H), 3.72-3.52 (m, 2H), 3.45 (s, 29% of 3H), 3.46-3.42 (m, 71% of 1H), 3.40 (s, 71% of 3H), 3.32 (ddd, *J* = 7.2, 5.6, 3.1 Hz, 29% of 1H), 3.06 (s, 29% of 3H), 3.05 (s, 71% of 3H), 1.56-1.25 (m, 10H), 1.21 (t, *J* = 6.9 Hz, 29% of 3H), 1.18 (t, *J* = 7.0 Hz, 71% of 3H), 0.88 (app t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 88.1 (major), 86.0 (minor), 81.2 (minor), 79.5 (major), 67.2 (major), 66.4 (minor), 56.5, (minor), 55.7 (major), 43.3 (major), 43.2 (minor), 31.9, 31.6 (major), 29.6 (minor), 29.5 (major), 29.3 (minor), 25.8 (minor), 25.4 (major), 22.8, 15.8 (major), 15.7 (minor), 14.2; IR (neat) 3286, 2926, 2858, 1458, 1328, 1161, 1110, 978, 766 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{11}H_{24}NO_3S$ (M^+-CH_3O) 250.1477, found 250.1466.



(±)-*N*-((1*R*,2*R*)-2-Ethoxy-1-*tert*-butoxyoctyl)isobutyramide and **(±)-*N*-((1*S*,2*R*)-2-ethoxy-1-*tert*-butoxyoctyl)isobutyramide** (**16**)

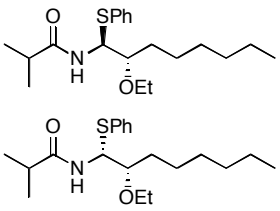
The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (60.0 mg, 0.354 mmol), CH_2Cl_2 (3.5 mL), $Cp_2Zr(H)Cl$ (110 mg, 0.425 mmol). After addition of isobutyryl chloride (52 μL, 0.50 mmol), the cold bath was removed and the mixture was stirred for 10 min. After that time, the flask was cooled to 0 °C and a solution of ^{t}BuOH (0.67 ml, 7.08 mmol) in CH_2Cl_2 (0.5 mL) was added dropwise to the reaction mixture over 3 min. The reaction was stirred for 10 min at 0 °C, then diluted with CH_2Cl_2 (10 mL) and quenched with saturated $NaHCO_3$ (20 mL). The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 15 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (10% - 20% EtOAc in hexanes containing 0.5% Et_3N) to give the desired product (79.2 mg, 70.8%) as a white solid in a 1.0:2.0 diastereomeric ratio. Further purification (12% - 18% EtOAc in hexanes containing 0.5% Et_3N) yielded pure samples. For faster eluting *anti*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.10 (d, *J* = 9.2 Hz, 1H), 5.32 (dd, *J* = 9.4, 2.0 Hz, 1H), 3.84 (qd, *J* = 9.6, 7.1 Hz, 1H), 3.58 (qd, *J* = 9.6, 7.0 Hz, 1H), 3.30-3.26 (m, 1H), 2.33 (sept, *J* = 6.9 Hz, 1H), 1.38-1.27 (m, 10H), 1.22 (s, 9H), 1.19-1.12 (m, 9H), 0.87 (t, *J* = 6.8 Hz, 3H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.8, 83.0, 75.5, 74.6, 67.6, 36.1, 32.0, 31.6, 29.5, 28.6, 25.9, 22.8, 19.7, 19.4, 15.9, 14.3; IR (neat) 3246, 2969, 2922, 2858, 1648, 1552, 1466, 1109, 1069 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{18}H_{37}NO_3Na$ (M^++Na) 338.2671, found 338.2663. For slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.00 (d, *J* = 9.2 Hz, 1H), 5.39 (dd, *J* = 9.4, 4.0 Hz, 1H), 3.64-3.54 (m, 2H), 3.14-3.09 (m, 1H), 2.30 (sept, *J* = 6.9 Hz, 1H), 1.51-1.38 (m, 2H), 1.35-1.24 (m, 8H), 1.20 (s, 9H), 1.19-1.11 (m, 9H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.3, 82.1, 74.9, 74.3, 66.8, 36.1, 32.0, 30.2, 29.6, 28.5, 26.0, 22.8, 19.5, 19.4, 15.8, 14.3; IR (neat) 3254, 2960, 2920, 2856, 1646, 1544, 1459, 1365, 1193, 1109, 1072, 731 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{18}H_{37}NO_3Na$ (M^++Na) 338.2671, found 338.2666.



(±)-*N*-((1*R*,2*R*)-2-Ethoxy-1-phenoxyoctyl)isobutyramide (**17**) and **(±)-*N*-((1*S*,2*R*)-2-ethoxy-1-phenoxyoctyl)isobutyramide**

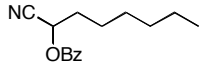
The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (60.0 mg, 0.354 mmol), CH_2Cl_2 (3.5 mL), $Cp_2Zr(H)Cl$ (110 mg, 0.425 mmol). After addition of isobutyryl chloride (52 μL, 0.50 mmol), the cold bath was removed and the mixture was stirred for 10 min. The mixture was cooled to °C and a solution of PhOH (333 mg, 3.54 mmol) in CH_2Cl_2 (0.5 mL) was added dropwise. The

reaction was stirred at °C for 40 min, then quenched with saturated $NaHCO_3$ solution (20 mL) and extracted with CH_2Cl_2 (4 x 15 mL). The organic extracts were washed with saturated Na_2CO_3 solution (20 mL), dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (7% - 10% EtOAc in hexanes containing 0.5% Et_3N) to give the desired product (81.7 mg, 68.7%) as a white solid in a 5.6:1.0 diastereomeric ratio. Further purification (7% - 10% EtOAc in hexanes containing 0.5% Et_3N) yielded pure samples. For faster eluting *anti*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.23 (m, 2H), 7.03 (app td, *J* = 7.8, 1.0 Hz, 2H), 6.96 (app tt, *J* = 7.3, 0.9 Hz, 1H), 6.36 (d, *J* = 9.9 Hz, 1H), 5.92 (dd, *J* = 9.9, 1.4 Hz, 1H), 3.96 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.72 (qd, *J* = 9.5, 7.0 Hz, 1H), 3.68-3.64 (m, 1H), 2.37 (sept, *J* = 6.9 Hz, 1H), 1.47-1.37 (m, 4H), 1.33-1.24 (m, 9H), 1.15 (d, *J* = 6.9 Hz, 3H), 1.08 (d, *J* = 6.9 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.1, 156.4, 129.7, 121.8, 116.5, 80.7, 80.0, 68.1, 36.0, 31.9, 29.4, 25.7, 22.8, 19.6, 19.5, 16.0, 14.2; IR (neat) 3290, 2964, 2929, 2859, 1657, 1595, 1534, 1495, 1222, 1107, 753 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{33}NO_3Na$ (M^++Na) 358.2358, found 358.2359. For slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 7.07-7.04 (m, 2H), 6.97 (app tt, *J* = 7.3, 0.9 Hz, 1H), 6.24 (d, *J* = 9.8 Hz, 1H), 6.02 (dd, *J* = 9.8, 3.4 Hz, 1H), 3.72 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.62 (qd, *J* = 9.4, 7.0 Hz, 1H), 3.44 (dt, *J* = 7.0, 3.3 Hz, 1H), 2.36 (sept, *J* = 6.9 Hz, 1H), 1.71-1.62 (m, 2H), 1.47-1.21 (m, 11H), 1.15 (d, *J* = 6.9 Hz, 3H), 1.10 (d, *J* = 6.9 Hz, 3H), 0.87 (app t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 156.8, 129.7, 121.9, 116.2, 81.2, 77.8, 66.8, 36.0, 31.9, 30.1, 29.6, 25.7, 22.8, 19.6, 19.5, 15.9, 14.3; IR (neat) 3288, 2963, 2926, 2857, 1653, 1535, 1495, 1220, 1109, 1042, 752 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{33}NO_3Na$ (M^++Na) 358.2358, found 358.2328.



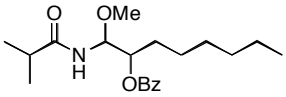
(±)-*N*-((1*R*,2*R*)-2-Ethoxy-1-(phenylthio)octyl)isobutyramide and **(±)-*N*-((1*S*,2*R*)-2-ethoxy-1-(phenylthio)octyl)isobutyramide** (**18**) The title compounds were prepared by following the representative procedure with the following amounts of reagents: ethoxynitrile (60.0 mg, 0.354 mmol), CH_2Cl_2 (3.5 mL), $Cp_2Zr(H)Cl$ (110 mg, 0.425 mmol). After addition of isobutyryl chloride (52 μL, 0.500 mmol), the cold bath was removed and the mixture was stirred for 10 min. The mixture was cooled to °C and a solution of PhSH (117 mg, 1.06 mmol) in CH_2Cl_2 (0.3 mL) was added dropwise. The reaction was stirred at °C for 10 min, then quenched with saturated $NaHCO_3$ solution (20 mL) and extracted with CH_2Cl_2 (4 x 15 mL). The organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (7% - 13% EtOAc in hexanes containing 0.5% Et_3N) to give the desired product (89.4 mg, 71.7%) as a white solid in a 1.0:7.1 diastereomeric ratio. Further purification (10% - 16% EtOAc in hexanes containing 0.5% Et_3N) yielded pure samples. For faster eluting *anti*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49-7.45 (m, 2H), 7.31-7.20 (m, 3H), 6.00 (d, *J* = 9.9 Hz, 1H), 5.58 (dd, *J* = 10.0, 1.8 Hz, 1H), 3.76-3.63 (m, 2H), 3.56 (dt, *J* = 6.5, 1.7 Hz, 1H), 2.29 (sept, *J* = 6.9 Hz, 1H), 1.60-1.48 (m, 1H), 1.40-1.14 (m, 12H), 1.08 (d, *J* = 6.9 Hz, 3H), 1.00 (d, *J* = 6.9 Hz, 3H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.3, 133.9, 132.3, 129.1, 127.5, 81.7, 67.3, 60.0, 35.9, 32.5, 31.9, 29.4, 25.7, 22.8, 19.7, 19.6, 15.9, 14.3; IR (neat) 3302, 2962, 2928, 2859, 1652, 1497, 1440, 1379, 1223, 1098, 739 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{33}NO_2SNa$ (M^++Na) 374.2130, found 374.2130. For slower eluting *syn*-product: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.44 (m, 2H), 7.30-7.18 (m, 3H), 6.00 (d, *J* = 9.5 Hz, 1H), 5.63 (dd, *J* = 9.7, 3.2 Hz, 1H), 3.69-3.49 (m, 3H), 2.25 (sept, *J* = 6.9 Hz, 1H), 1.73-1.66 (m, 2H), 1.42-1.28 (m, 8H), 1.21 (t, *J* = 7.0 Hz, 3H), 1.06 (d, *J* = 6.9 Hz, 3H), 0.98 (d, *J* = 6.9 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.0, 133.6, 132.0, 129.1, 127.3, 82.0, 66.0, 59.8, 35.8, 31.9, 31.6, 29.5, 25.8, 22.7, 19.6, 19.5, 15.7, 14.2; IR (neat) 3293,

2962, 2927, 2858, 1650, 1526, 1223,, 1100, 736 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{33}NO_2SNa$ (M^++Na) 374.2130, found 374.2115.



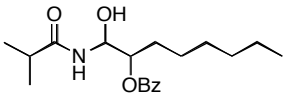
1-Cyanoheptyl benzoate (**19**)

To s solution of 2-hydroxyoctanenitrile[2] (0.600 g, 4.25 mmol) in CH_2Cl_2 (14 mL) were added Et_3N (1.2 mL, 8.5 mmol), DMAP (5.2 mg, 42 μmol) and benzoyl chloride (0.60 mL, 5.1 mmol). The reaction was stirred for 1 h, then quenched with water (30 mL) and extracted with CH_2Cl_2 (3 x 30 mL). The organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (5%-10% Et_2O in hexanes) to give the benzoate **20** (1.04 g, 94.1%) as a colorless oil. The spectral data were consistent with those reported in the literature.[2]



(±)-1-(Isobutyramido)-1-methoxyoctan-2-yl benzoate (**20**)

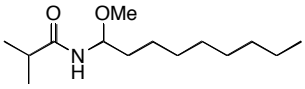
The title compound was prepared by following the representative procedure with the following amounts of reagents: benzoate **19** (100 mg, 0.408 mmol), CH_2Cl_2 (4.0 mL), $Cp_2Zr(H)Cl$ (158 mg, 0.612 mmol), isobutyryl chloride (52 μL, 0.49 mmol), MeOH (0.7 mL, 17 mmol). After the reaction was complete, it was quenched with 1 N HCl (1.5 mL) and water (15 mL) and extracted with CH_2Cl_2 (3 x 25 mL). The organic extracts were washed with saturated $NaHCO_3$ (15 mL), dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (15% - 30% EtOAc in hexanes) to give the product (90.4 mg, 63.5%) as a white solid in a 1.4:1.0 diastereomeric ratio. Further purification (15% - 30% EtOAc in hexanes) yielded pure materials. For the faster eluting product: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03-8.01 (m, 2H), 7.60-7.56 (m, 1H), 7.48-7.44 (m, 2H), 5.97 (d, *J* = 9.5 Hz, 1H), 5.24 (dd, *J* = 9.6, 6.6 Hz, 1H), 5.19 (ddd, *J* = 8.6, 6.6, 3.8 Hz, 1H), 3.38 (s, 3H), 2.32 (sept, *J* = 7.0 Hz, 1H), 1.86-1.80 (m, 1H), 1.78-1.70 (m, 1H), 1.44-1.23 (m, 7H), 1.21-1.15 (m, 1H), 1.10 (d, *J* = 7.0 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H), 0.86 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.0, 167.0, 133.5, 130.0, 129.9, 128.7, 81.9, 74.5, 56.3, 36.1, 31.8, 31.3, 29.3, 25.3, 22.8, 19.6, 19.5, 14.2; IR (neat) 3295, 2959, 2929, 2858, 1721, 1663, 1529, 1452, 1273, 1113, 712 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{31}NO_4Na$ (M^++Na) 372.2151, found 372.2123. For the slower eluting product: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (app d, *J* = 7.3 Hz, 2H), 7.59 (app t, *J* = 7.4 Hz, 1H), 7.47 (app t, *J* = 7.8 Hz, 2H), 6.01 (d, *J* = 9.6 Hz, 1H), 5.33 (dd, *J* = 9.8, 4.0 Hz, 1H), 5.12 (td, *J* = 8.6, 4.4 Hz, 1H), 3.38 (s, 3H), 2.40 (sept, *J* = 6.9 Hz, 1H), 1.82-1.73 (m, 2H), 1.44-1.26 (m, 8H), 1.18 (d, *J* = 7.0 Hz, 3H), 1.17 (d, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.6, 166.7, 133.4, 130.1, 130.0, 128.7, 81.0, 75.8, 56.7, 36.1, 31.8, 30.6, 29.3, 25.4, 22.7, 19.8, 19.7, 14.2; IR (neat) 3299, 2929, 2858, 1722, 1661, 1527, 1453, 1273, 1113, 712 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{31}NO_4Na$ (M^++Na) 372.2151, found 372.2133.



1-(Isobutyramido)-1-hydroxyoctan-2-yl benzoate (**21**)

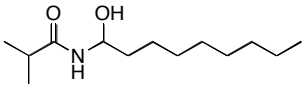
The title compound was prepared by following the representative procedure with the following amounts of reagents: benzoate **19** (100 mg, 0.408 mmol), CH_2Cl_2 (4.0 mL), $Cp_2Zr(H)Cl$ (158 mg, 0.612 mmol), isobutyryl chloride (52 μL, 0.49 mmol). The reaction was quenched with water (15 mL) and extraction of the mixture with EtOAc (3 x 25 mL). After evaporation of the solvent, the crude product was purified by column chromatography (20% - 60% EtOAc in hexanes containing 0.5% Et_3N) gave the product **22** (71.6 mg, 52.4%) as a colorless oil in a 3.0:1 diastereomeric ratio. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10-8.07 (m, 1.5H), 8.03-7.99 (m, 0.5H), 7.61-7.56 (m, 1H), 7.48-7.42 (m, 2H), 7.11 (d, *J* = 7.5 Hz, 0.75H), 6.74 (d, *J* = 8.3 Hz, 0.25H), 5.53-5.36 (m, 1H), 5.22-5.17 (m, 0.25H), 5.15-5.10 (m, 0.75H), 4.74 (br s, 0.25H), 4.52 (br s, 0.75H), 2.47-2.22 (m, 1H), 1.92-1.78 (m, 2H), 1.44-1.20 (m, 8H), 1.13 (d, *J* = 6.8 Hz, 2.25H), 1.11 (d, *J* =

6.8 Hz, 2.25H), 1.04 (d, *J* = 6.9 Hz, 0.75H), 0.99 (d, *J* = 6.9 Hz, 0.75H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (for major diastereomer) δ 178.4, 167.7, 133.6, 130.1, 128.6, 76.2, 75.0, 35.6, 31.8, 30.7, 29.2, 25.5, 22.7, 19.5, 19.3, 14.2; IR (neat) 3338, 2959, 2928, 2858, 1720, 1657, 1530, 1451, 1274, 1119, 1070, 711 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{19}H_{28}NO_3$ (M^+-OH) 318.2069, found 318.2064.



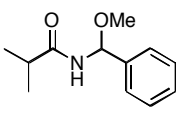
***N*-(1-Methoxynonyl)isobutyramide (23)**

The title compound was prepared by following the representative procedure with the following amounts of reagents: octyl cyanide (84 mg, 0.60 mmol), THF (6.0 mL), $Cp_2Zr(H)Cl$ (194 mg, 0.754 mmol). The hydrozirconation reaction was stirred for 30 min, then cooled to 0 °C and isobutyryl chloride (95 μL, 0.90 mmol) was added dropwise. The reaction was stirred for 10 min at 0 °C and MeOH (0.73 mL, 18 mmol) was added dropwise. The reaction was stirred at 0 °C for 15 min, then quenched with a solution of Et_3N (0.25 mL) in water (15 mL) and extracted with CH_2Cl_2 (4 x 20 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by column chromatography (15% - 25% EtOAc in hexanes containing 0.5% Et_3N) to gave the title product (91.7 mg, 62.3%) as a white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.66 (d, *J* = 9.5 Hz, 1H), 5.10 (td, *J* = 9.8, 6.1 Hz, 1H), 3.31 (s, 3H), 2.37 (sept, *J* = 6.9 Hz, 1H), 1.66-1.59 (m, 1H), 1.52-1.43 (m, 1H), 1.38-1.24 (m, 12H), 1.18 (app d, *J* = 6.4 Hz, 3H), 1.16 (app d, *J* = 6.4 Hz, 3H), 0.86 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.4, 81.1, 55.9, 36.1, 35.8, 32.0, 29.6, 29.5, 29.4, 25.0, 22.8, 19.9, 19.7, 14.2; IR (neat) 3281, 2920, 2853, 1651, 1538, 1466, 1377, 1236, 1081, 929, 720 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{13}H_{26}NO$ (M^+- CH_3O) 212.2014 found 212.2010.



***N*-(1-Hydroxynonyl)isobutyramide (24)**

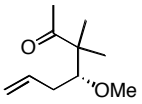
The title compound was prepared by following the representative procedure with the following amounts of reagents: octyl cyanide (84.0 mg, 0.603 mmol), CH_2Cl_2 (4.5 mL), $Cp_2Zr(H)Cl$ (171 mg, 0.663 mmol). After hydrozirconation was complete, a solution of isobutyryl chloride (76 μL, 0.72 mmol) and Et_3N (0.25 mL, 1.8 mmol) in CH_2Cl_2 (0.5 mL) was added dropwise at 0 °C. The reaction was stirred for 15 min at 0 °C and quenched with water (20 mL). The mixture was acidified by adding 1 N HCl to pH~1.0 and extracted with CH_2Cl_2 (3 x 25 mL). The combined organic extracts were washed with saturated $NaHCO_3$ (20 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by column chromatography (10% - 70% EtOAc in hexanes) to give acyl hemiaminal **25** (74.7 mg, 54.0%) as a white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.17 (br s, 1H), 5.30 (q, *J* = 6.6 Hz, 1H), 4.27 (br s, 1H), 2.35 (sept, *J* = 6.9 Hz, 1H), 1.73-1.61 (m, 1H), 1.59-1.48 (m, 1H), 1.40-1.26 (m, 12H), 1.15 (d, *J* = 6.9 Hz, 3H), 1.14 (d, *J* = 6.9 Hz, 3H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.4, 74.5, 35.7, 35.3, 32.0, 29.6, 29.5, 29.4, 25.1, 22.8, 19.6, 19.4, 14.3; IR (neat) 3298, 2934, 2854, 1653, 1540, 1462, 1231, 1095 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{13}H_{26}NO$ (M^+-OH) 212.2014 found 212.2015.



(±)-*N*-(Methoxy(phenyl)methyl)isobutyramide (26)

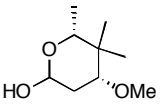
By following the representative procedure, reaction of benzonitrile (60.0 mg, 0.582 mmol) with $Cp_2Zr(H)Cl$ (240 mg, 0.931 mmol) in THF (5.8 mL) for 2.5 h followed by acylation with isobutyryl chloride (92 μL, 0.87 mmol) and addition of MeOH (0.71 mL, 17 mmol) in CH_2Cl_2 (0.5 mL) gave the title product (87.9 mg, 72.9%) as a white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.30 (m, 5H), 6.14 (d, *J* = 9.4 Hz, 1H), 6.02 (d, *J* = 8.8 Hz, 1H), 3.45 (s, 3H), 2.40 (sept, *J* = 6.9 Hz, 1H), 1.21 (d, *J* = 6.9

Hz, 3H), 1.18 (d, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.4, 139.6, 128.8, 128.6, 126.0, 81.3, 56.1, 36.0, 19.8, 19.6; IR (neat) 3286, 2967, 1653, 1535, 1451, 1230, 1099, 1046, 951, 746 cm^{-1}; HRMS (EI): m/z calcd for $C_{11}H_{14}NO_2$ (M^+-CH_3) 192.1024, found 192.1031.



4-Methoxy-3,3-dimethylhept-6-2-ene

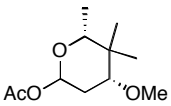
To a solution of **27** (2.40 g, 15.4 mmol) in CH_2Cl_2 (15.4 mL) cooled to –10 °C was added 2,6-di-*tert*-butyl pyridine (5.12 mL, 23.0 mmol), followed by methyl trifluoromethanesulfonate (2.26 mL, 19.9 mmol). The reaction mixture was stirred at -10 °C for 10 minutes then subsequently warmed to room temperature. After 48 hours H_2O (10 mL) was added and the reaction mixture was diluted with CH_2Cl_2 (15 mL). The reaction mixture was subsequently extracted with CH_2Cl_2 (3 x 10 mL). The organic layers were combined, washed with satd. $NaHCO_3$ (aq) (15 mL) and brine (15 mL), then dried (Na_2SO_4) and filtered. After *careful* concentration under reduced pressure, the crude residue was purified via flash column chromatography (CH_2Cl_2) to afford the desired product as a colorless oil (2.12 g, 81% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.96-5.82 (m, 1H), 5.15-5.08 (m, 1H), 5.06-5.03 (m, 1H), 3.43 (dd, J = 6.5, 5.5 Hz, 1H), 3.39 (s, 3H), 2.21-2.25 (m, 2H), 2.17 (s, 3H), 1.16 (s, 3H), 1.09 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 212.9, 136.0, 116.4, 86.0, 59.9, 52.4, 36.7, 26.5, 10.9, 20.2; IR (neat) 2977, 2827, 1704, 1469, 1100 cm^{-1}.



4-Methoxy-5,5,6-trimethyl-2*H*-tetrahydropyran-2-ol

To a solution of the ketone (4.46 g, 26.2 mmol) in CH_2Cl_2 (262 mL) at -78 °C was slowly added dimethylaluminum chloride (65.5 mL, 1M in hexanes). After 5 minutes, tributyltin hydride (7.6 g, 28.8 mmol) was slowly added at -78 °C. After one hour, satd. $NaHCO_3$ (aq) (60 mL) was added and the reaction was warmed to room temperature. The reaction mixture was extracted with CH_2Cl_2 (3 x 50 mL). The organic layers were combined, washed with brine (150 mL), dried (Na_2SO_4), and filtered. After concentration under reduced pressure, the crude residue was purified via flash column chromatography (20% Et_2O in pentane) to afford the desired product containing tin impurities. The material was used without further purification.

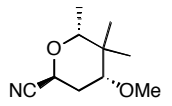
To a solution of crude **32** in CH_2Cl_2 (50.0 mL) at –78 °C was bubbled O_3. After 20 minutes, N_2 was bubbled through the deep blue solution for 10 minutes, followed by addition of PPh_3 (6.43 g, 24.5 mmol) at –78 °C. The reaction was subsequently warmed to room temperature. After 40 minutes, the reaction mixture was concentrated under reduced pressure and purified via column chromatography (40% Et_2O in pentane) to afford the desired product as colorless solid (2.2 g, 48% yield over 2 steps): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.36 (d, J=3.3 Hz, 0.70H), 4.7 (d, J=9 Hz, 0.30H), 4.26 (br s, 0.40 H), 3.87 (q, J=6.3 Hz, 1H), 3.49 (br s, 1H), 3.34 (s, 3H), 3.26 (m, 0.80H), 2.85 (m, 0.20), 2.18 (m, 0.31H), 2.02 (dd, J=4.5, 12.9 Hz, 0.69H), 1.57 (m, 1H), 1.41 (m, 0.80H), 1.12 (d, J=6.3Hz, 0.68H), 1.05 (d, J=6.3 Hz, 2.32H), 0.92 (s, 2.32H), 0.89 (s, 0.68H), 0.84 (s, 0.68H), 0.82 (s, 2.32 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 94.6, 92.3, 83.6, 80.0, 76.5, 71.3, 57.4, 57.2, 39.0, 38.2, 34.1, 31.0, 22.6, 22.5, 14.3, 12.4, 11.55; IR (neat): 3404, 2976, 2942, 1470, 1384, 1266, 1100 cm^{-1}.



Acetic acid 4-Methoxy-5,5,6-trimethyl-2*H*-tetrahydropyran-2-yl ester

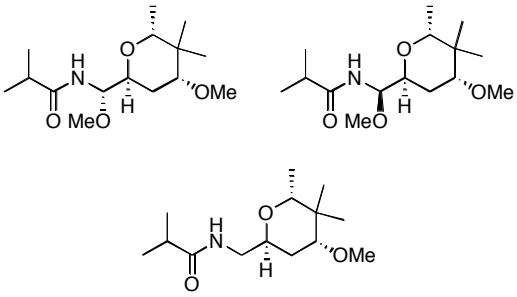
To a solution of the tetrahydropyranol (1.00 g, 5.74 mmol) in CH_2Cl_2 (12 mL) and pyridine (24 mL) at 0 °C was added acetic anhydride (1.1 mL, 12 mmol) followed by DMAP (0.15 g, 1.2 mmol), and the reaction was warmed to room temperature. After 30 minutes, H_2O (20 ml) was added, and the reaction mixture was extracted with CH_2Cl_2 (3 x 30 mL). The organic layers were combined and washed with satd. $CuSO_4$ (aq) (5 x 30 mL) followed by brine (30 mL). The organic extracts were dried (Na_2SO4), filtered and

concentrated under reduced pressure. The crude residue was purified via flash chromatography (30% Et_2O in pentane) to afford the desired product (0.94 g, 75% yield): ^1H NMR (300 MHz, $CDCl_3$) δ 6.19 (d, *J* = 3.3 Hz, 0.77H), 5.60 (d, *J* = 9.6 Hz, 0.41 H), 3.66 (q, *J*=6.3 Hz, 1H), 3.31 (s, 3H), 3.25-3.12 (m, 0.77H), 2.91-2.86 (m, 0.23H), 2.13 (s, 3H), 2.13-1.96 (m, 1H), 1.70-1.44 (m, 1H), 1.11 (d, *J* = 6.3 Hz, 1.1H), 1.04 (d, *J* = 6.3 Hz, 1.9H), 0.91 (s, 1.9H), 0.87 (s, 1.1H), 0.83 (s, 1.1H), 0.81 (s, 1.9H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.6, 169.2, 92.9, 92.7, 84.2, 83.2, 80.0, 77.3, 74.0, 57.2, 38.5, 38.2, 31.3, 29.4, 22.5, 22.4, 21.1, 21.0, 14.3, 14.2, 12.3, 11.6; IR (neat): 2980, 2253, 1745, 1469 cm^{-1}.



(±)- (2*S*,4*R*,6*R*)-Tetrahydro-4-methoxy-5,5,6-trimethyl-2*H*-pyran-2-carbonitrile (29)

To a solution of the tetrahydropyranyl acetate (0.300 g, 1.39 mmol) in CH_2Cl_2 (9.30 mL) cooled to -78 °C was added TMSCN (0.55 mL, 4.16 mmol) followed by BF3·OEt_2 (0.21 mL, 1.67 mmol). The reaction was stirred at -78 °C for 10 minutes, and then warmed to -42 °C. After 45 minutes, the reaction mixture was poured into a 0 °C solution of pH 7 buffer and CH_2Cl_2 (20 mL, 1:1, pH 7 buffer to CH_2Cl_2) and subsequently warmed to room temperature. The reaction mixture was extracted with CH_2Cl_2 (3 x 10 mL), and the combined organic layers were washed with brine (20 mL), dried (Na_2SO_4), and filtered. The organic extracts were concentrated under reduced pressure to afford to afford the desired product as a colorless solid (0.22 g, 85% yield).^1H NMR (300 MHz, $CDCl_3$) δ 4.90 (dd, *J* = 6.0, 1.2 Hz, 1H), 3.63 (q, *J* = 6.3 Hz, 1H), 3.37 (s, 3H), 3.17 (dd, *J* = 11.7, 4.5 Hz, 1H), 2.06 (ddd, *J* = 13.5, 4.5, 1.4 Hz, 1H), 1.84 (ddd, *J* = 13.4, 11.8, 6.1 Hz, 1H), 1.12 (d, *J* = 6.3 Hz, 3H), 0.96 (s, 3H), 0.83 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 118.0, 81.4, 78.0, 64.2, 57.8, 39.4, 29.3, 22.6, 14.6, 12.2; IR (neat) 2980, 2941, 2874, 1470, 1450, 1391, 1164, 1104, 954, 867, 718 cm^{-1}; HRMS (EI): *m*/*z* calcd for $C_{10}H_{17}NO_2$ (M^+) 183.1259, found 183.1255.

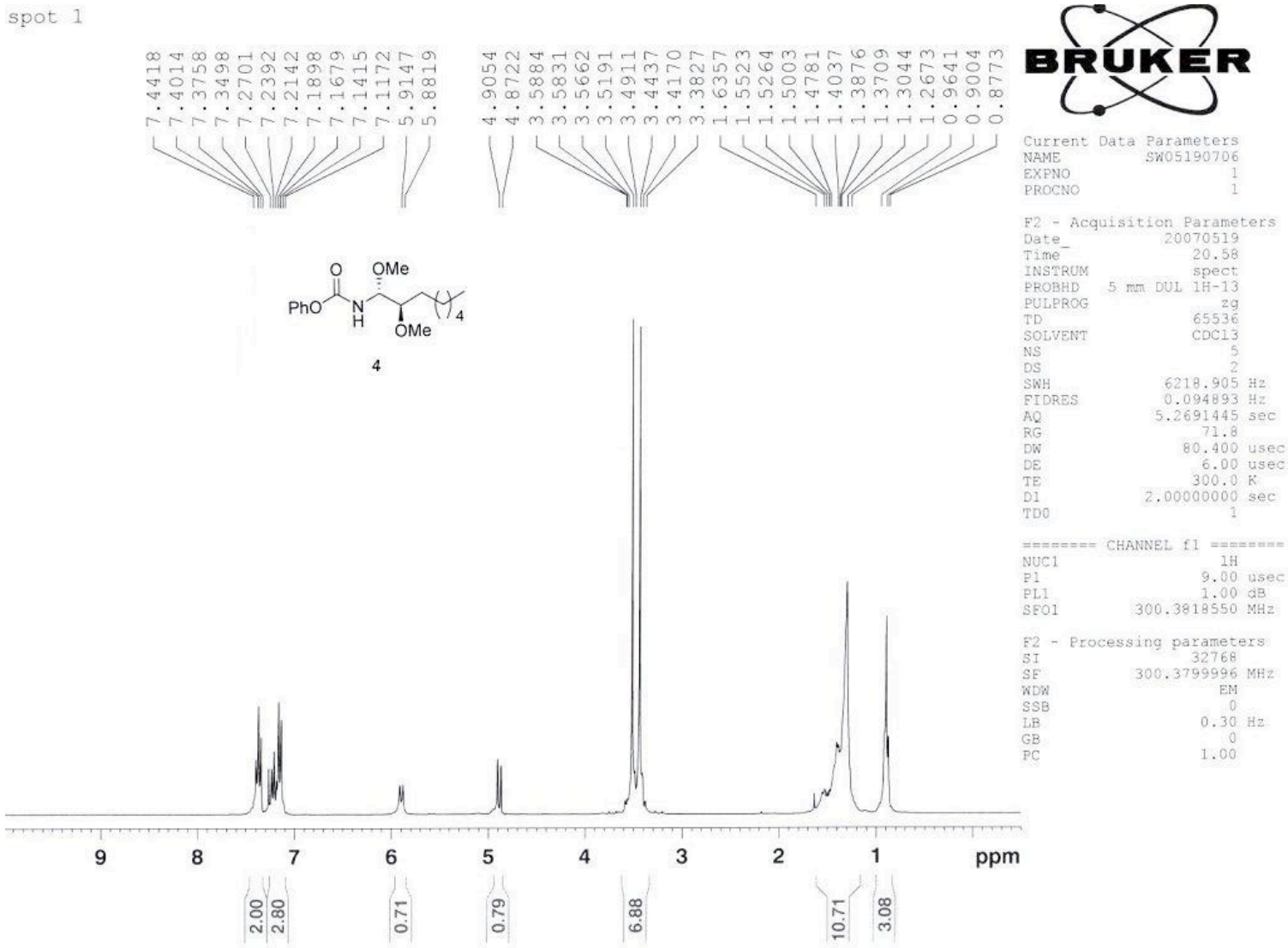


(±)-*N*-((*S*)-((2*S*,4*R*,6*R*)-Tetrahydro-4-methoxy-5,5,6-trimethyl-2*H*-pyran-2-yl)(methoxy)methyl)isobutyramide (30), (±)-*N*-((*R*)-((2*S*,4*R*,6*R*)-Tetrahydro-4-methoxy-5,5,6-trimethyl-2*H*-pyran-2-yl)(methoxy)methyl)isobutyramide (31) and **(±)-*N*-(((2*S*,4*R*,6*R*)-Tetrahydro-4-methoxy-5,5,6-trimethyl-2*H*-pyran-2-yl)methyl)isobutyramide**

To a solution of tetrahydropyranyl cyanide (50.0 mg, 0.273 mmol) in CH_2Cl_2 (2.7 mL) was added Schwartz reagent (84.5 mg, 0.328 mmol). The mixture was stirred for 15 min, then cooled to 0 °C and isobutyryl chloride (40 μL, 0.38 mmol) was added dropwise. The cold bath was removed and the mixture was stirred for 10 min. After that time, the flask was cooled to –78 °C and $Mg(ClO_4)_2$ (61 mg, 0.27 mmol) was added in one portion. After 30 min, a pre-cooled solution (–78 °C) of MeOH (0.22 ml, 5.5 mmol) in CH_2Cl_2 (0.5 mL) was cannulated dropwise to the reaction mixture over 5 min. After completion of addition, the reaction was stirred at –78 °C for 15 min, then quenched with saturated $NaHCO_3$ solution (15 mL) and warmed to room temperature. The biphasic mixture was extracted with CH_2Cl_2 (3 x 20 mL) and the combined organic extracts were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (20% - 70% EtOAc in hexanes containing 0.5% Et_3N) to give the desired products **34** and **35** (60.2 mg, 76.8%) in a 2.3:1.0 diastereomeric ratio as a colorless oil and the over-reduction product **36** (6.8 mg, 9.7%) as a colorless oil. For **33**: ^1H NMR (300 MHz, $CDCl_3$) δ 5.77 (br s, 1H), 4.05-3.97 (m, 1H), 3.48-3.39 (m, 3H), 3.32 (s, 3H), 3.03 (t, *J* = 6.4 Hz, 1H), 2.38 (sept, *J* = 6.9 Hz,

1H), 1.74-1.69 (m, 2H), 1.18-1.14 (m, 9H), 0.96 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.3, 82.0, 74.5, 69.0, 57.7, 41.0, 38.6, 35.9, 27.6, 24.5, 19.9, 19.8, 15.6, 15.5; IR (neat) 3305, 2970, 2933, 2874, 1651, 1548, 1468, 1386, 1243, 1103 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{14}H_{27}NO_3Na$ (M^++Na) 280.1889, found 280.1899. Further purification (20% - 40% EtOAc in hexanes containing 0.5% Et_3N) of a mixture of **31** and **32** yielded pure diastereomers. For faster eluting product **31** (major, white solid): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.01 (d, *J* = 9.0 Hz, 1H), 5.26 (dd, *J* = 9.5, 6.5 Hz, 1H), 3.84-3.80 (m, 1H), 3.39 (s, 3H), 3.36 (q, *J* = 6.5 Hz, 1H), 3.33 (s, 3H), 3.04 (dd, *J* = 8.6, 4.1 Hz, 1H), 2.44 (sept, *J* = 6.9 Hz, 1H), 1.92 (td, *J* = 13.7, 4.5 Hz, 1H), 1.66 (ddd, *J* = 13.8, 8.6, 5.2 Hz,1H), 1.20 (d, *J* = 6.9 Hz, 3H), 1.19 (d, *J* = 6.9 Hz, 3H), 1.10 (d, *J* = 6.6 Hz, 3H), 0.94 (s, 3H), 0.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.2, 82.0, 80.0, 76.2, 70.9, 57.7, 56.4, 38.2, 36.2, 26.0, 24.5, 19.9, 19.8, 16.1, 15.3; IR (neat) 3300, 2972, 2938, 1659, 1536, 1468, 1387, 1103 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{15}H_{29}NO_4Na$ (M^++Na) 310.1994, found 310.1985. For slower eluting product **35** (minor, colorless oil): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.26 (d, *J* = 9.3 Hz, 1H), 5.16 (dd, *J* = 9.6, 3.9 Hz, 1H), 3.83-3.78 (m, 1H), 3.67 (q, *J* = 6.6 Hz, 1H), 3.38 (s, 3H), 3.31 (s, 3H), 3.18 (dd, *J* = 7.0, 3.8 Hz, 1H), 2.42 (sept, *J* = 6.9 Hz, 1H), 1.96 (ddd, *J* = 13.7, 6.6, 3.9 Hz, 1H), 1.71-1.63 (m, 1H), 1.22-1.17 (m, 9H), 1.00 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.5, 82.4, 81.7, 70.3, 57.8, 56.7, 38.0, 36.1, 26.3, 25.4, 19.9, 19.7, 17.6, 15.6; IR (neat) 3293, 2970, 2934, 1658, 1531, 1468, 1387, 1170, 1102 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{15}H_{29}NO_4Na$ (M^++Na) 310.1994, found 310.2002.

(1) Torii, S.; Inokuchi, T.; Takagishi, S.; Horike, H.; Kuroda, H.; Uneyama, K. *Bull. Chem. Soc. Jpn*, **1987**, *60*, 2173.
(2) Watahiki, T.; Ohba, S.; Oriyama, T. *Org. Lett.* **2003**, *5*, 2679.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7.1679
7.1415
7.1172
5.9147
5.8819
4.9054
4.8722
3.5884
3.5831
3.5662
3.5191
3.4911
3.4437
3.4170
3.3827
1.6357
1.5523
1.5264
1.5003
1.4781
1.4037
1.3876
1.3709
1.3044
1.2673
0.9641
0.9004
0.8773
PhO
O
OMe
N
H
OMe
4
4
9
8
7
6
5
4
3
2
1
ppm
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2.80
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0.79
6.88
10.71
3.08
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PROCNO 1
F2 - Acquisition Parameters
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Time 20.58
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PULPROG zg
TD 65536
SOLVENT CDCl3
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DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
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RG 71.8
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
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P1 9.00 usec
PL1 1.00 dB
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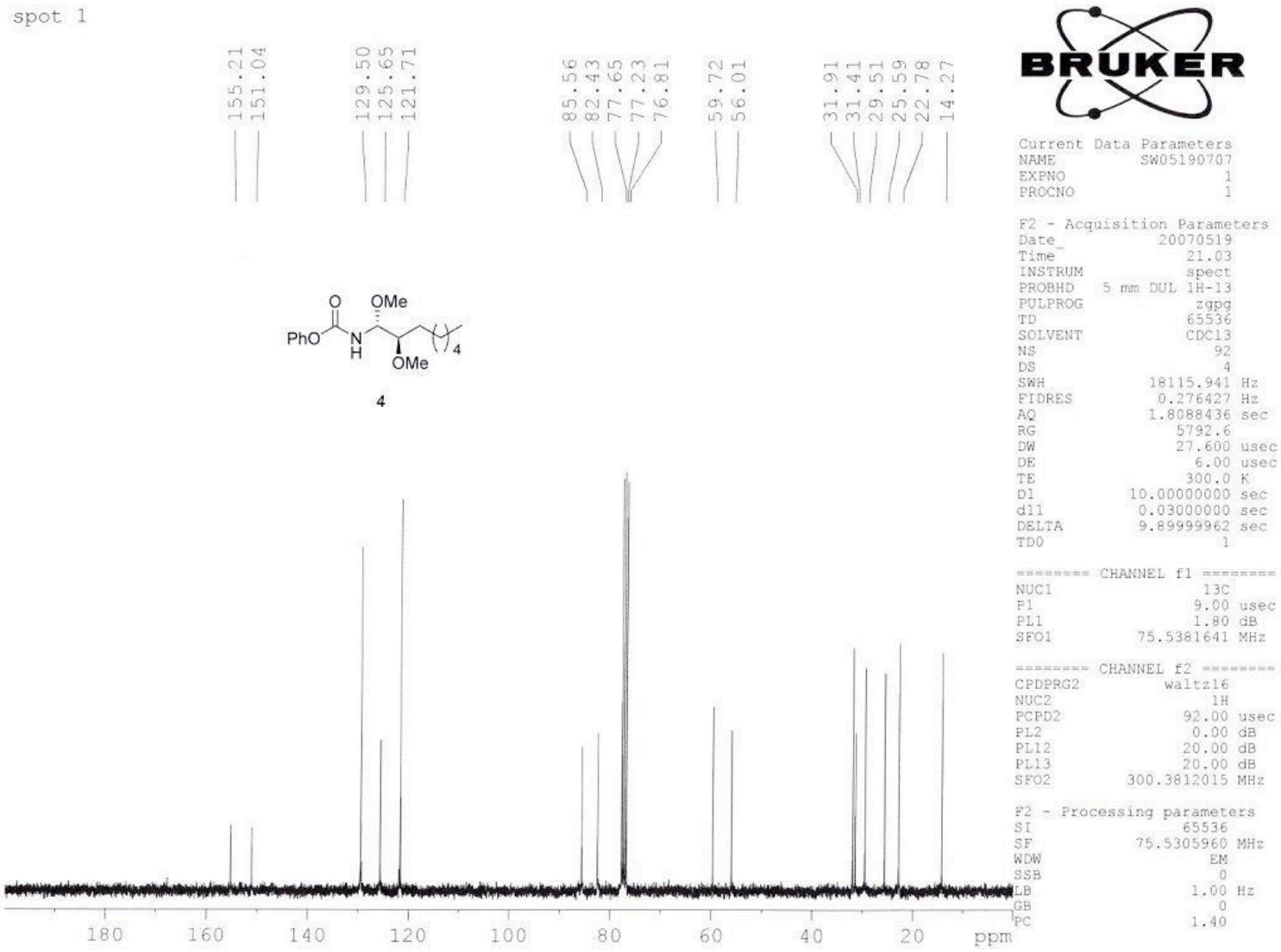
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29.51
25.59
22.78
14.27
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Current Data Parameters
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PROCNO 1
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SOLVENT CDCl3
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SWH 18115.941 Hz
FIDRES 0.276427 Hz
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RG 5792.6
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DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
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P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
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NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
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SF 75.5305960 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
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H
OMe
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140
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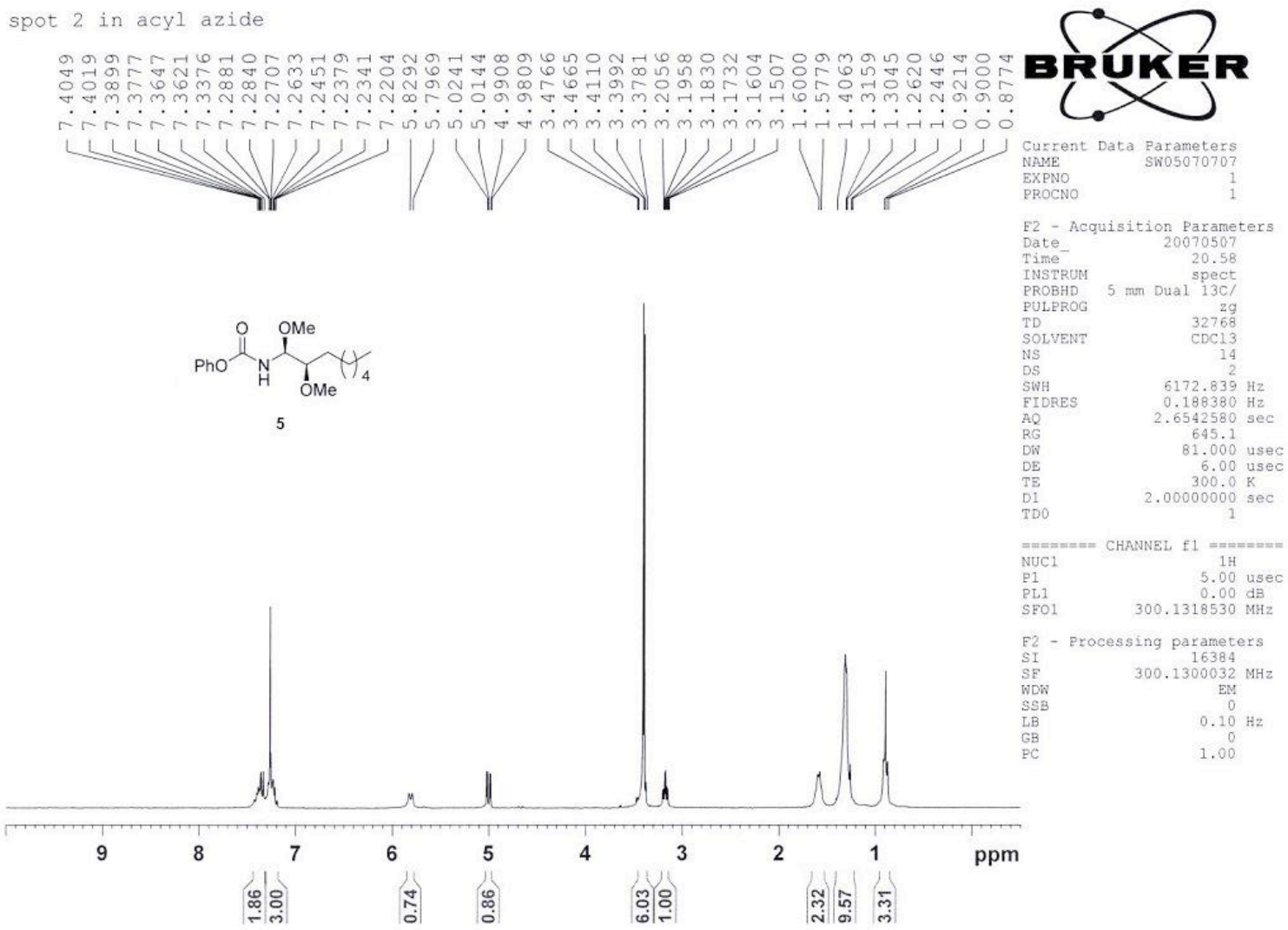
80
60
40
20
ppm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7.2707
7.2633
7.2451
7.2379
7.2341
7.2204
5.8292
5.7969
5.0241
5.0144
4.9908
4.9809
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3.4665
3.4110
3.3992
3.3781
3.2056
3.1958
3.1830
3.1732
3.1604
3.1507
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EXPNO 1
PROCNO 1
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PULPROG zg
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SOLVENT CDCl3
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SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 645.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
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P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300032 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
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O
N
H
OMe
OMe
4
5
9
8
7
6
5
4
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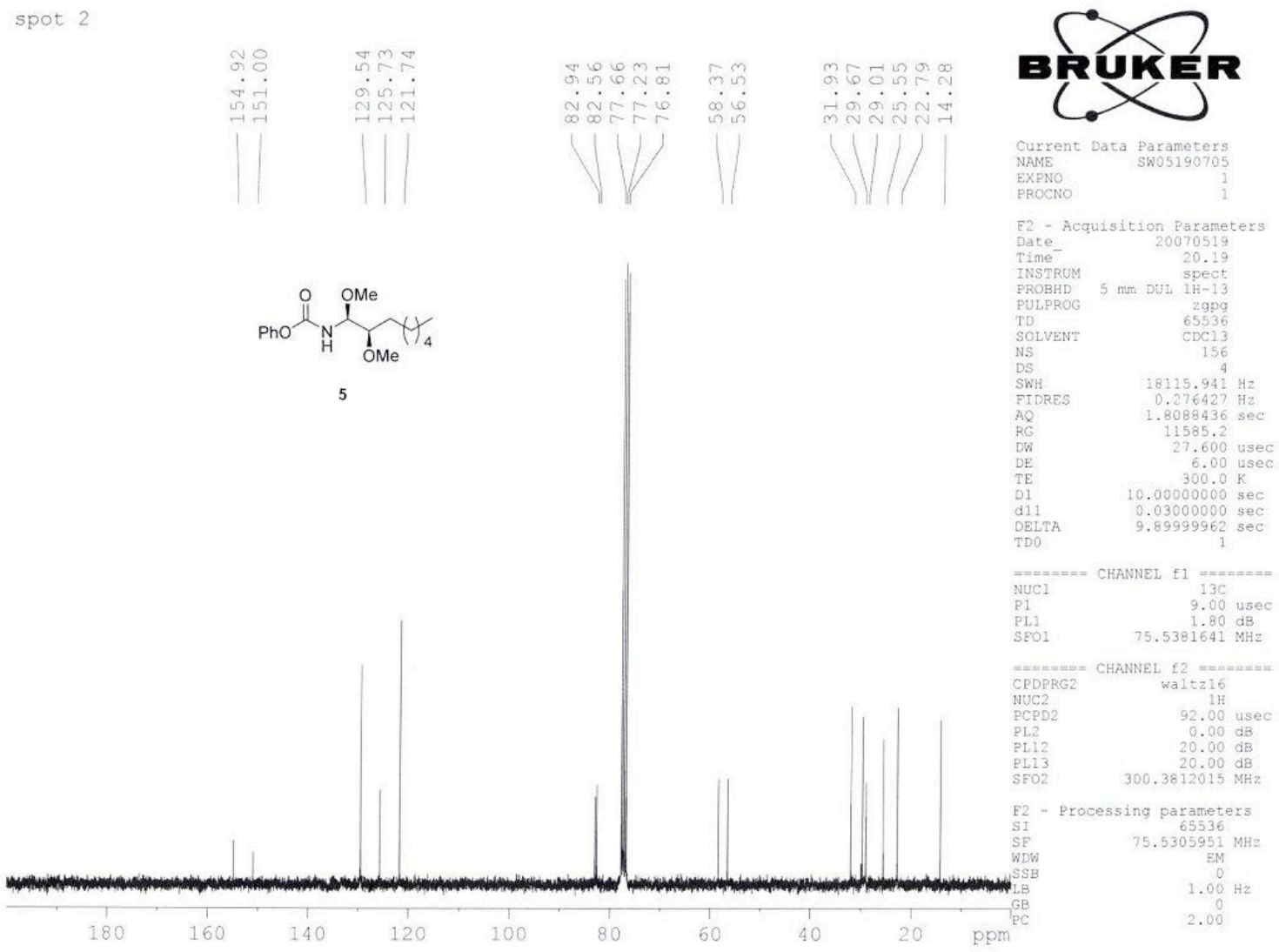
9.57
3.31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77.23
76.81
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29.67
29.01
25.55
22.79
14.28
O
OMe
PhO
N
H
OMe
4
5
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SW05190705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070519
Time 20.19
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PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 156
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 11585.2
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
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F2 - Processing parameters
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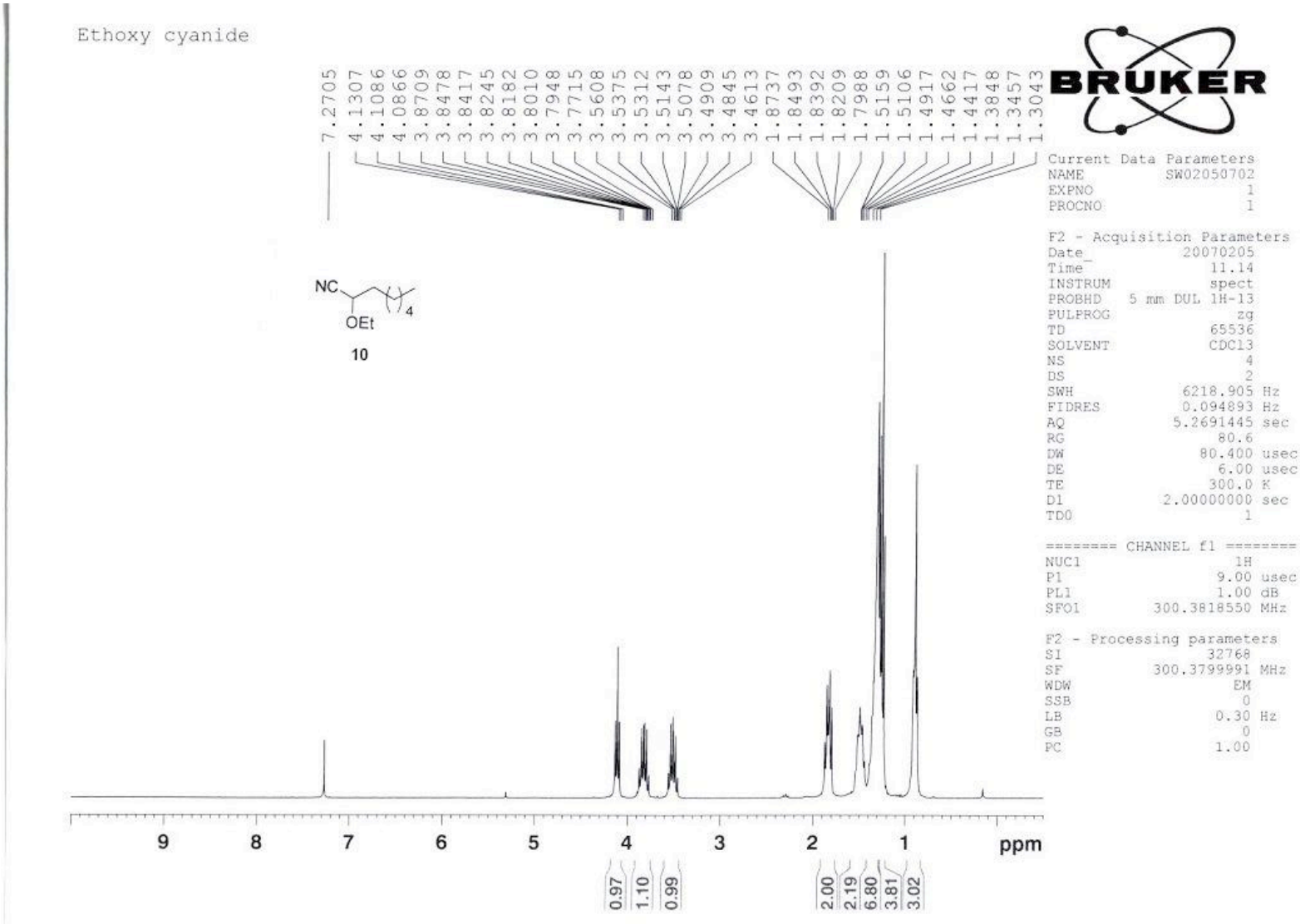
SSB 0
LB 1.00 Hz
GB 0
PC 2.00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3.7948
3.7715
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3.5375
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3.4909
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3.4613
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1.8493
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1.8209
1.7988
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BRUKER
NC
OEt
4
10
Current Data Parameters
NAME SW02050702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 11.14
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PULPROG zg
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SOLVENT CDCl3
NS 4
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 80.6
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799991 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
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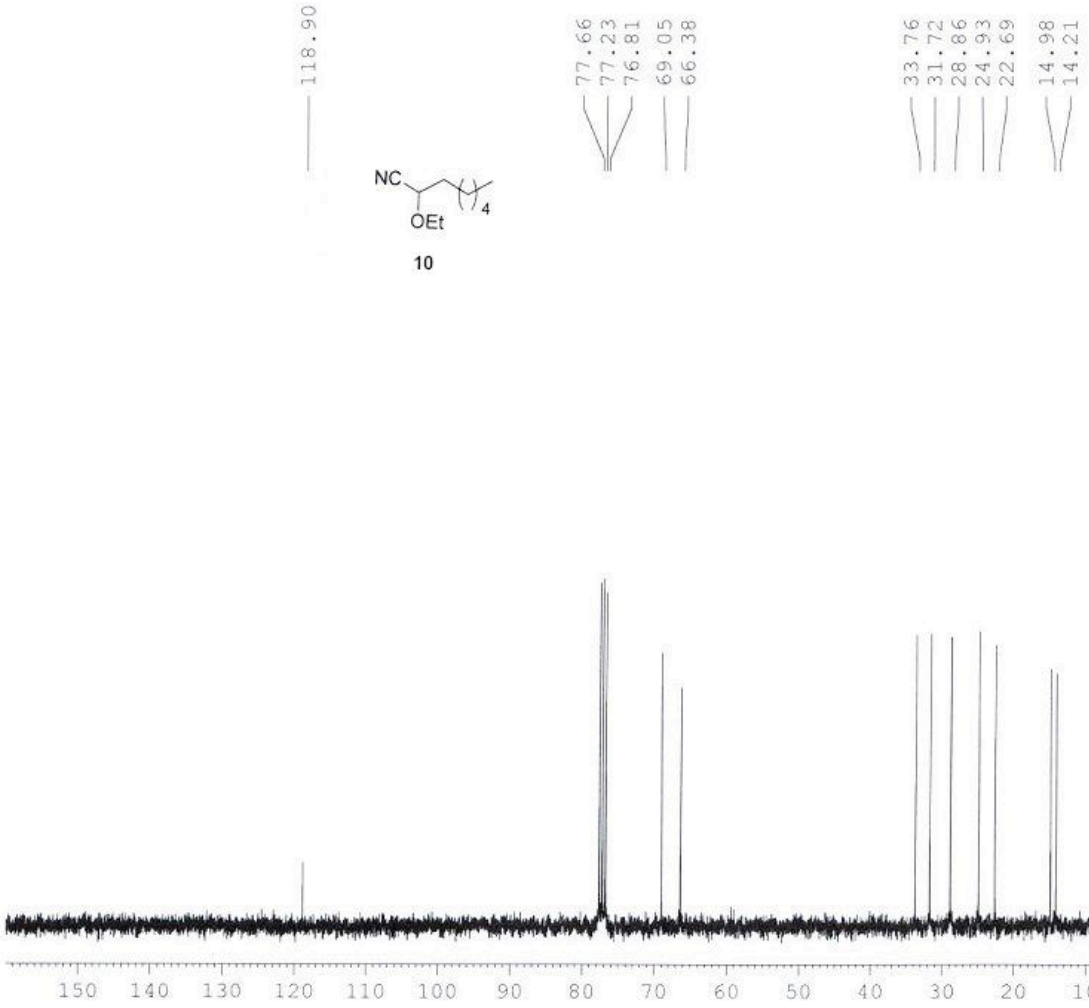
6.80
3.81
3.02

Ethoxy cyanide



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Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 11.05
INSTRUM spect
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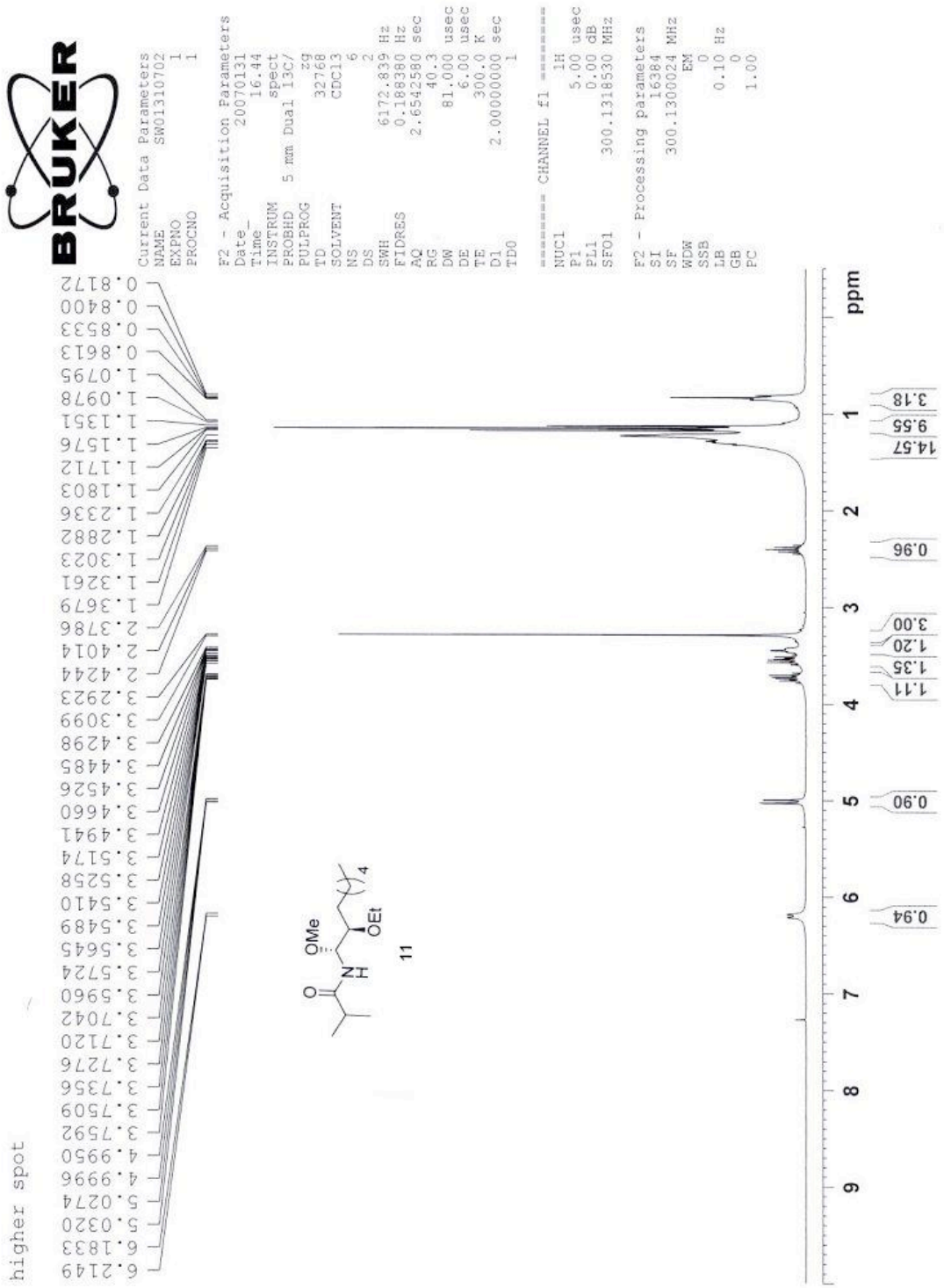
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 29
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 8192
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

F2 - Processing parameters
SI 65536
SF 75.5305950 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 6
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 40.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300024 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
OMe
OEt
11
ppm
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1.35
1.11
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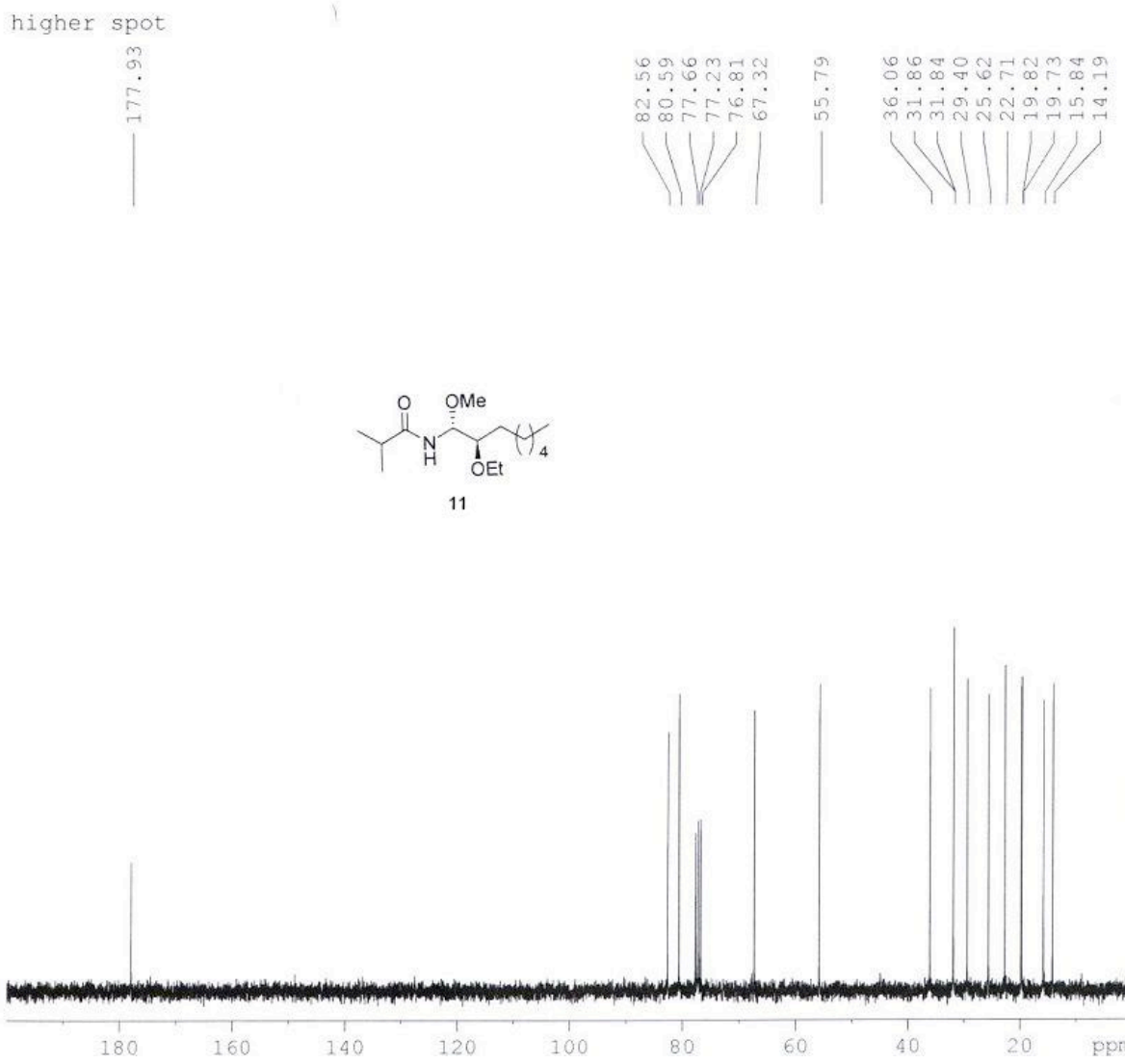
higher spot



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Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 19.39
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PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 9
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 6502
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

F2 - Processing parameters
SI 65536
SF 75.5305970 MHz
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SSB 0
LB 1.00 Hz
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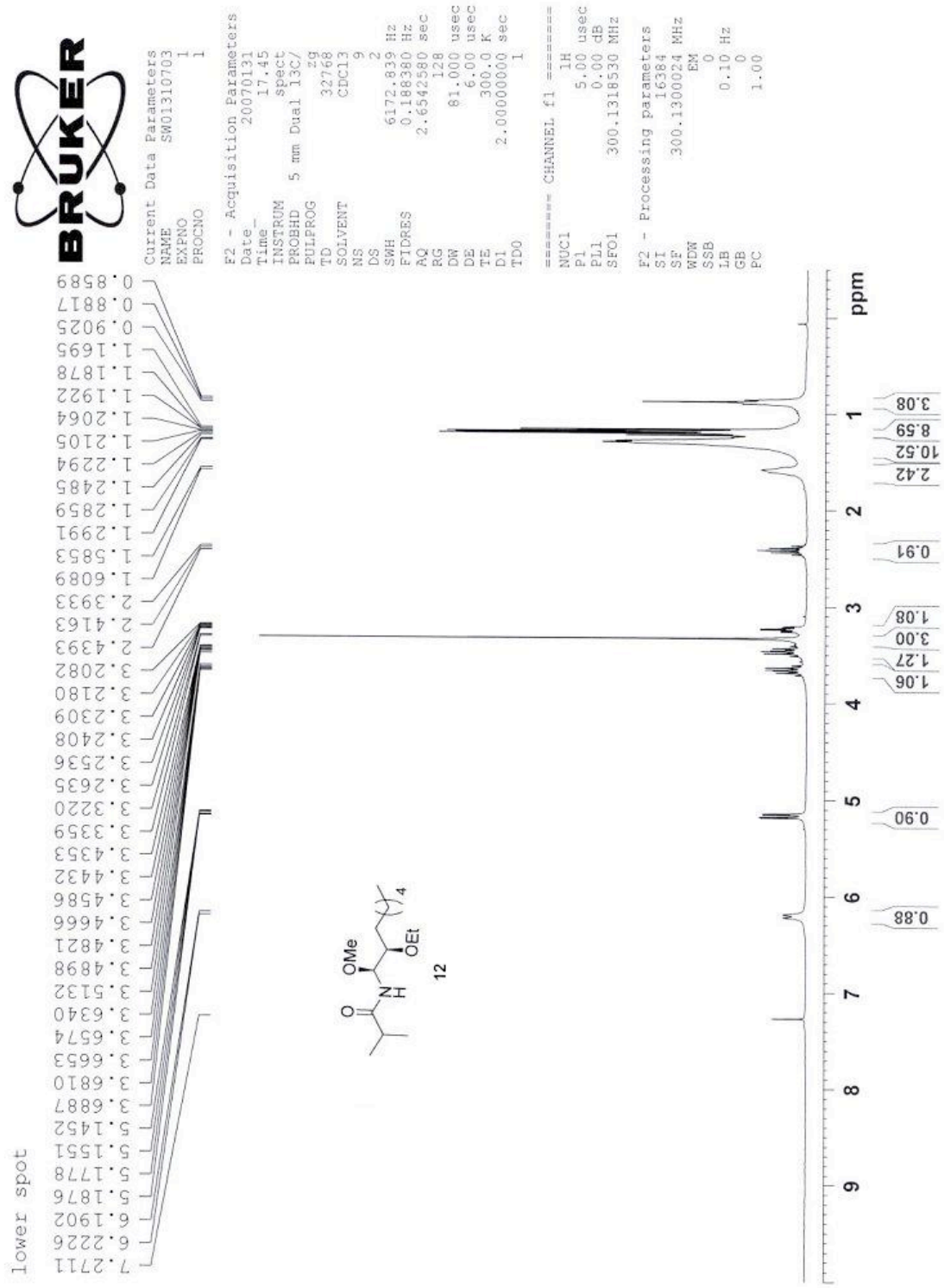
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EXPNO 1
PROCNO 1
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Time_ 17.45
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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 9
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300024 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
lower spot
7.2711
6.2226
6.1902
5.1778
5.1551
5.1452
3.6887
3.6810
3.6653
3.6574
3.6340
3.5132
3.4898
3.4821
3.4666
3.4586
3.4432
3.4353
3.3359
3.3220
3.2635
3.2536
3.2408
3.2309
3.2180
3.2082
2.4393
2.4163
2.3933
1.6089
1.5853
1.2991
1.2859
1.2485
1.2294
1.2105
1.2064
1.1922
1.1878
1.1695
0.9025
0.8817
0.8589
OMe
OEt
12
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3.00
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1.06
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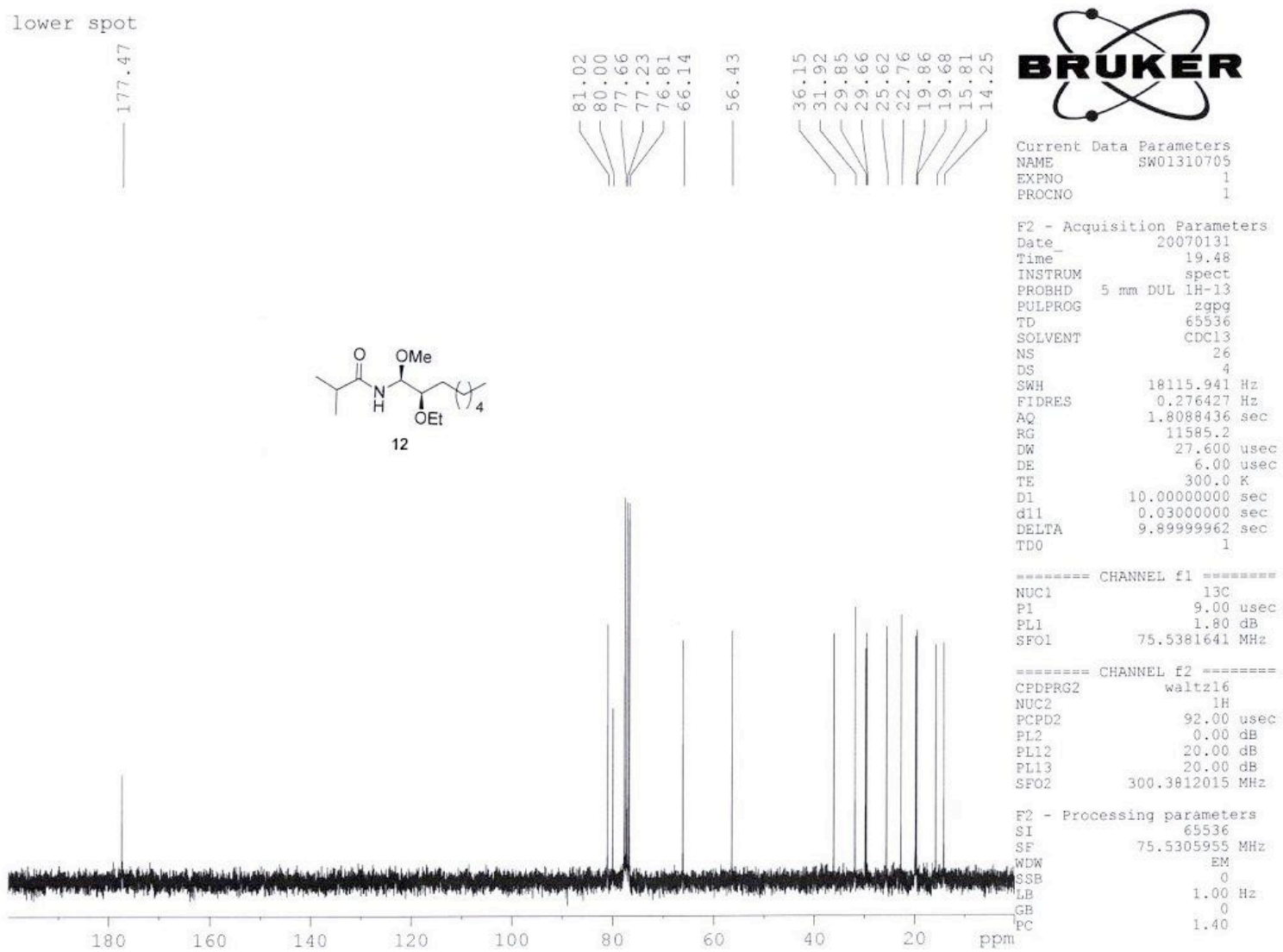
0.88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22.76
19.86
19.68
15.81
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O
OMe
N
H
OEt
4
12
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SW01310705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070131
Time 19.48
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PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 26
DS 4
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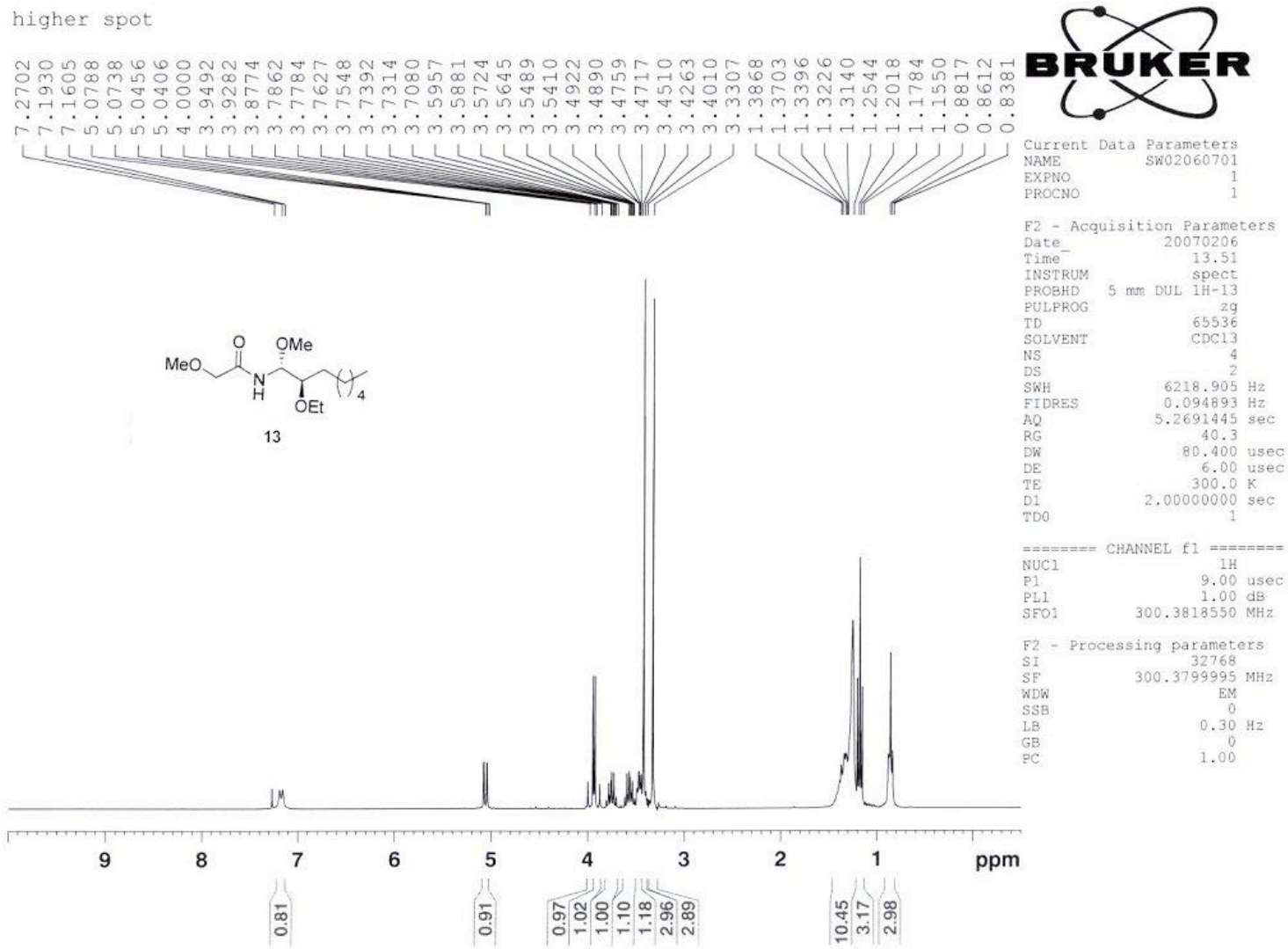
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 11585.2
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305955 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


higher spot
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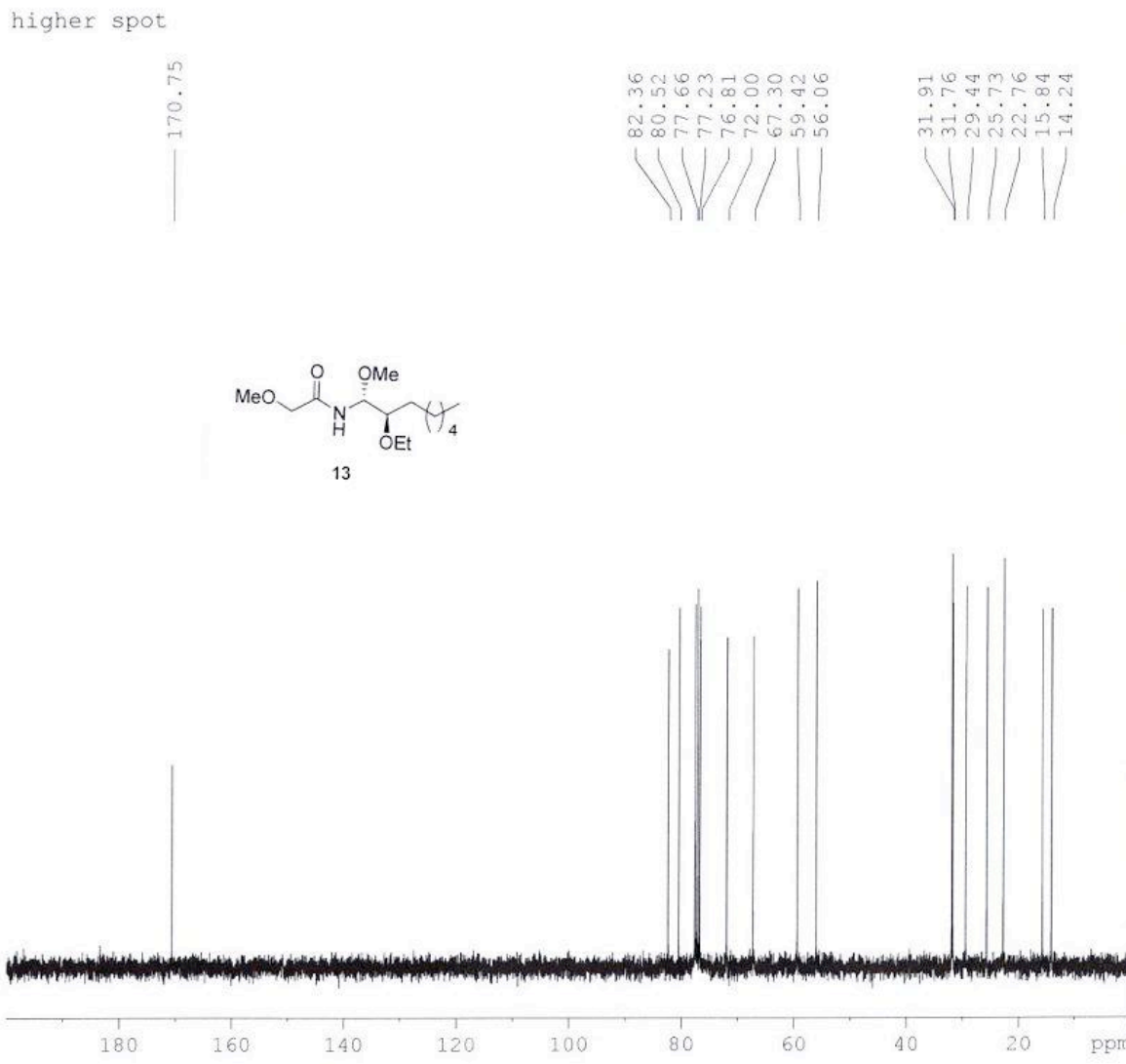
5.0456
5.0406
4.0000
3.9492
3.9282
3.8774
3.7862
3.7784
3.7627
3.7548
3.7392
3.7314
3.7080
3.5957
3.5881
3.5724
3.5645
3.5489
3.5410
3.4922
3.4890
3.4759
3.4717
3.4510
3.4263
3.4010
3.3307
1.3868
1.3703
1.3396
1.3226
1.3140
1.2544
1.2018
1.1784
1.1550
0.8817
0.8612
0.8381
MeO
O
OMe
N
H
OEt
4
13
9
8
7
6
5
4
3
2
1
ppm
0.81
0.91
0.97
1.02
1.00
1.10
1.18
2.96
2.89
10.45
3.17
2.98
BRUKER
Current Data Parameters
NAME SW02060701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070206
Time 13.51
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 40.3
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799995 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

higher spot



BRUKER

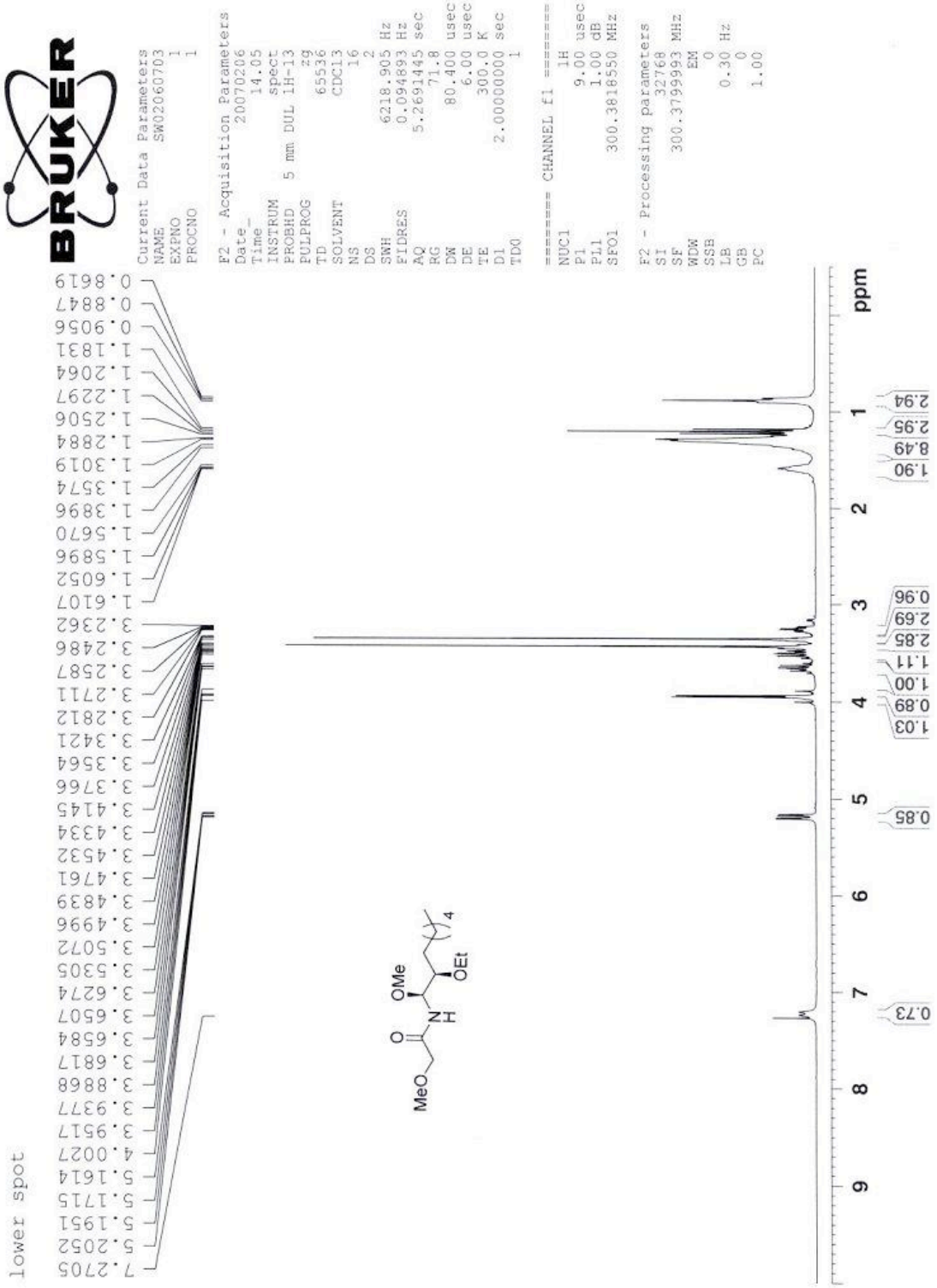
Current Data Parameters
NAME SW02060702
EXPNO 1
PROCNO 1

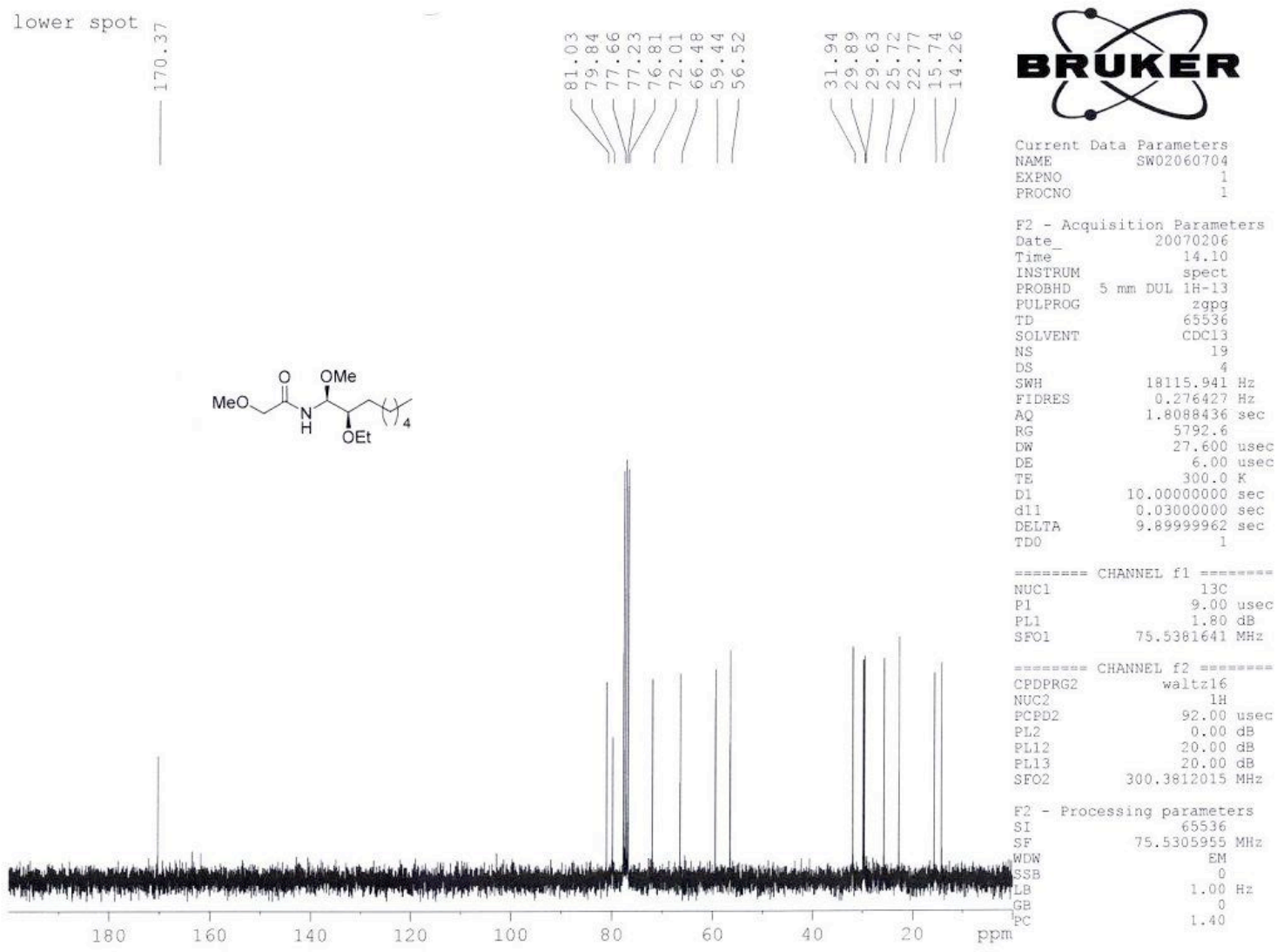
F2 - Acquisition Parameters
Date_ 20070206
Time 13.55
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 20
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 4597.6
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

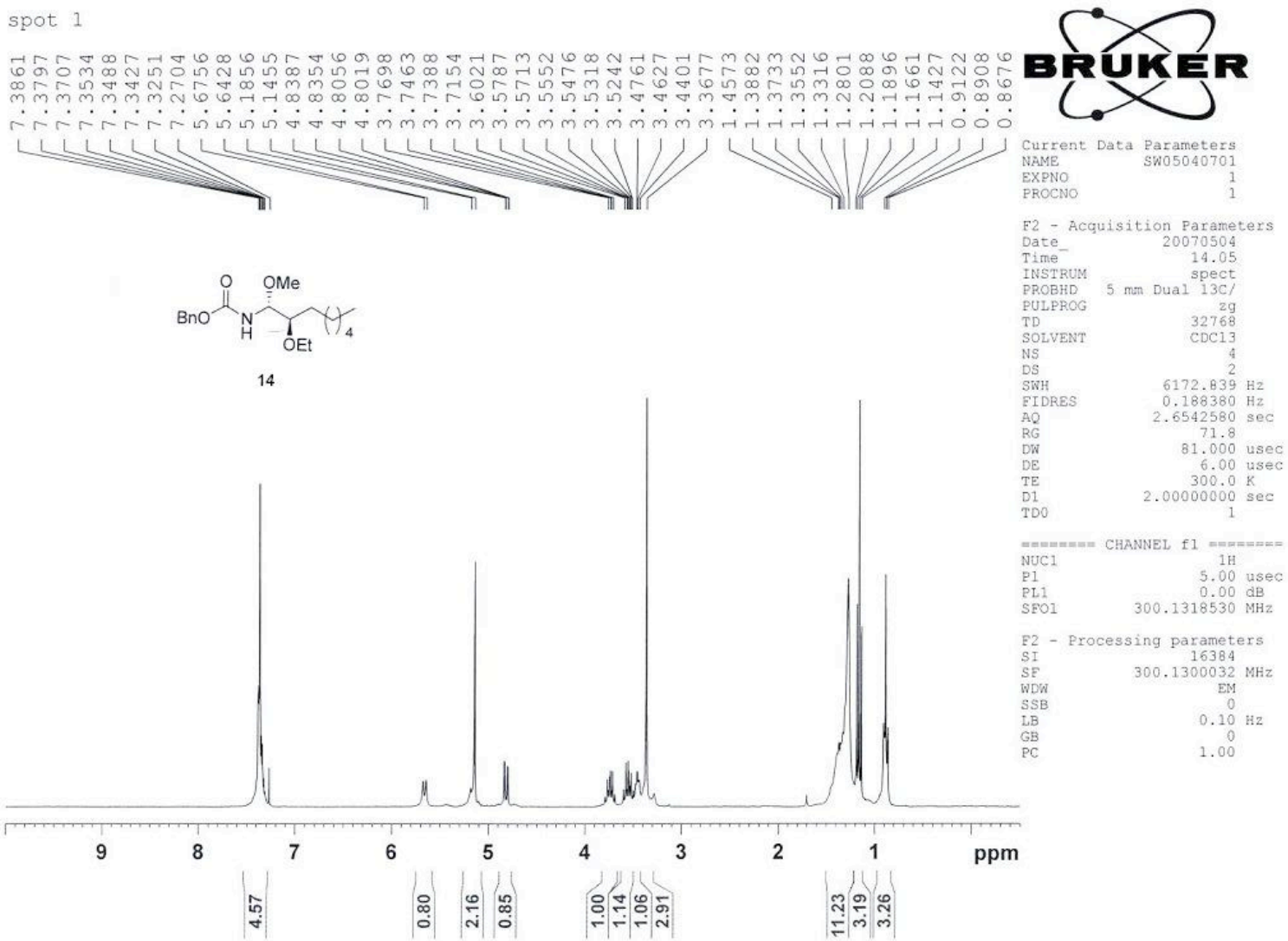
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

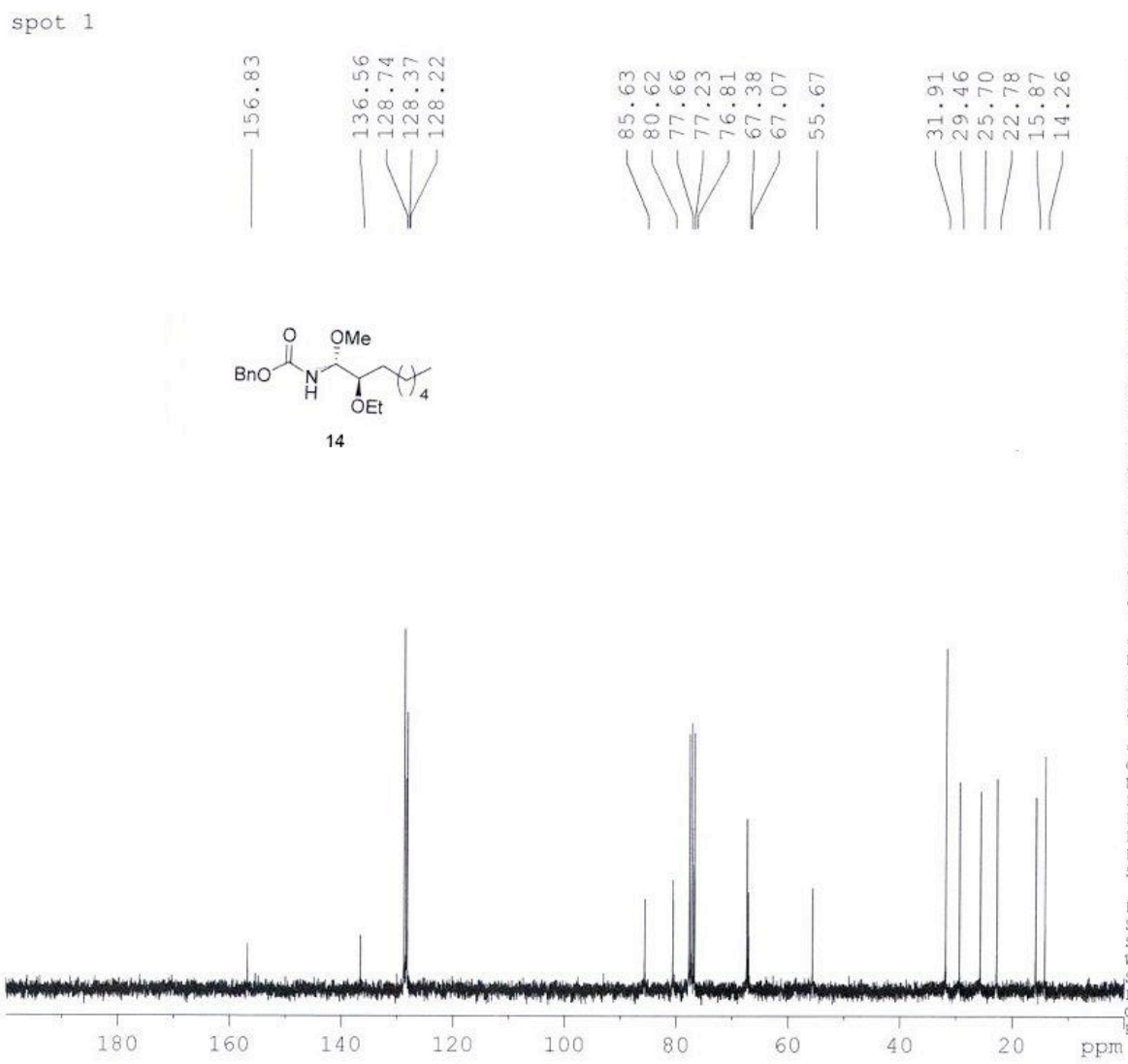
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

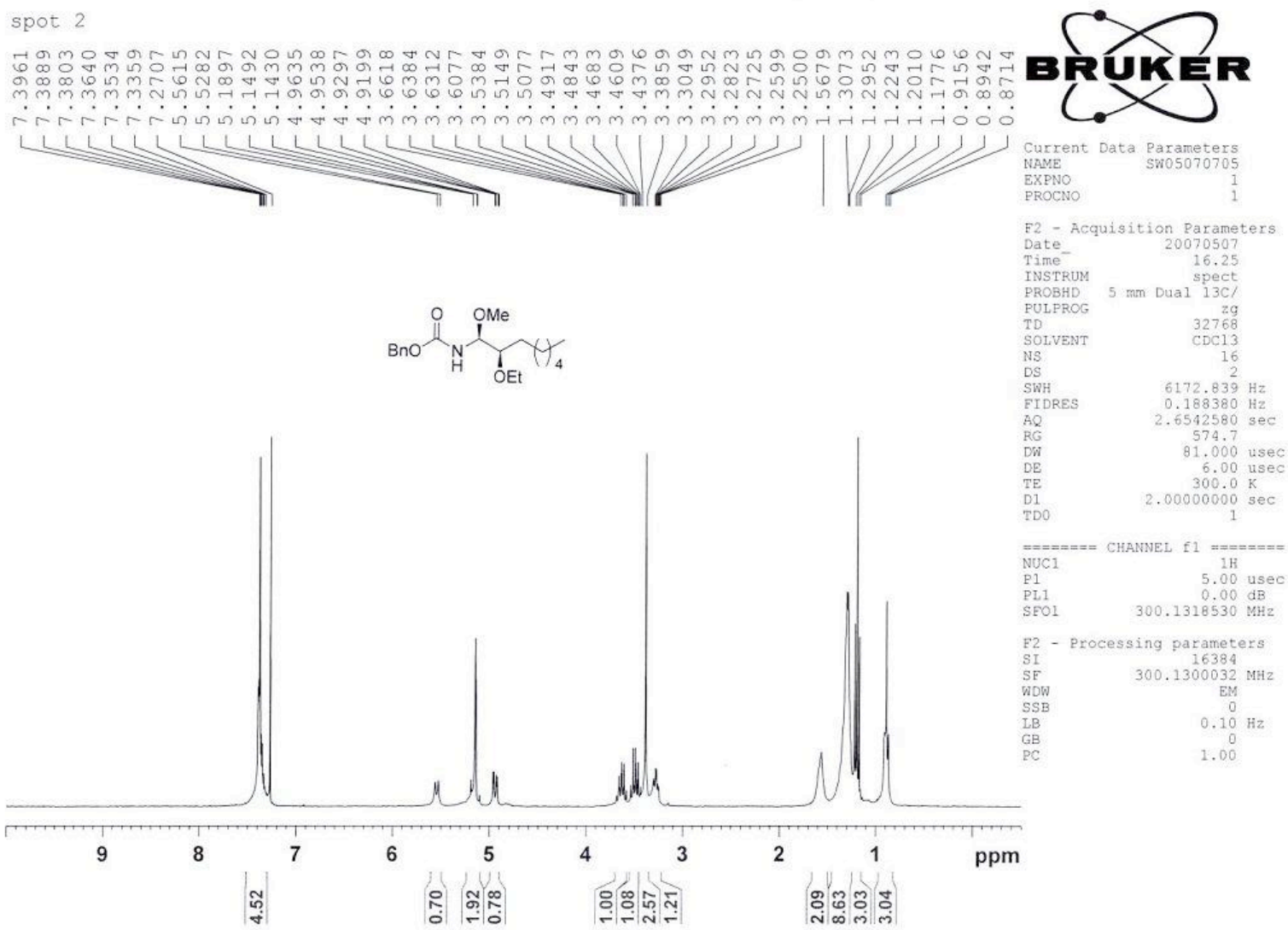
F2 - Processing parameters
SI 65536
SF 75.5305959 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

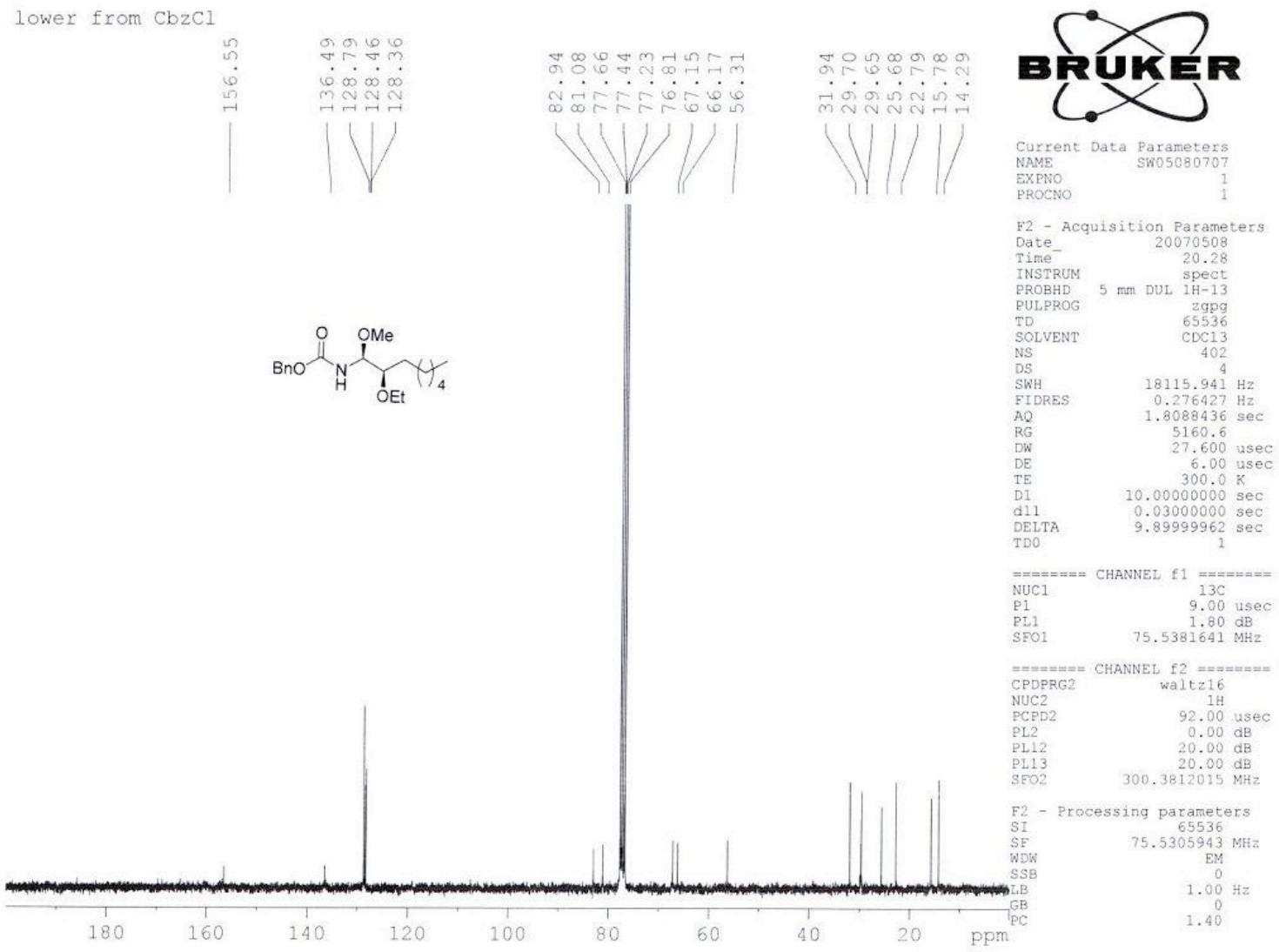

BRUKER
Current Data Parameters
NAME SW02060703
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070206
Time 14.05
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 71.8
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
lower spot
0.8619
0.8847
0.9056
1.1831
1.2064
1.2297
1.2506
1.2884
1.3019
1.3574
1.3896
1.5670
1.5896
1.6052
1.6107
3.2362
3.2486
3.2587
3.2711
3.2812
3.3421
3.3564
3.3766
3.4145
3.4334
3.4532
3.4761
3.4839
3.4996
3.5072
3.5305
3.6274
3.6507
3.6584
3.6817
3.8868
3.9377
3.9517
4.0027
5.1614
5.1715
5.1951
5.2052
7.2705
OMe
OEt
MeO
N
H
O
4
ppm
1 2 3 4 5 6 7 8 9
0.73
0.85
1.03
0.89
1.00
1.11
2.85
2.69
0.96
1.90
8.49
2.95
2.94

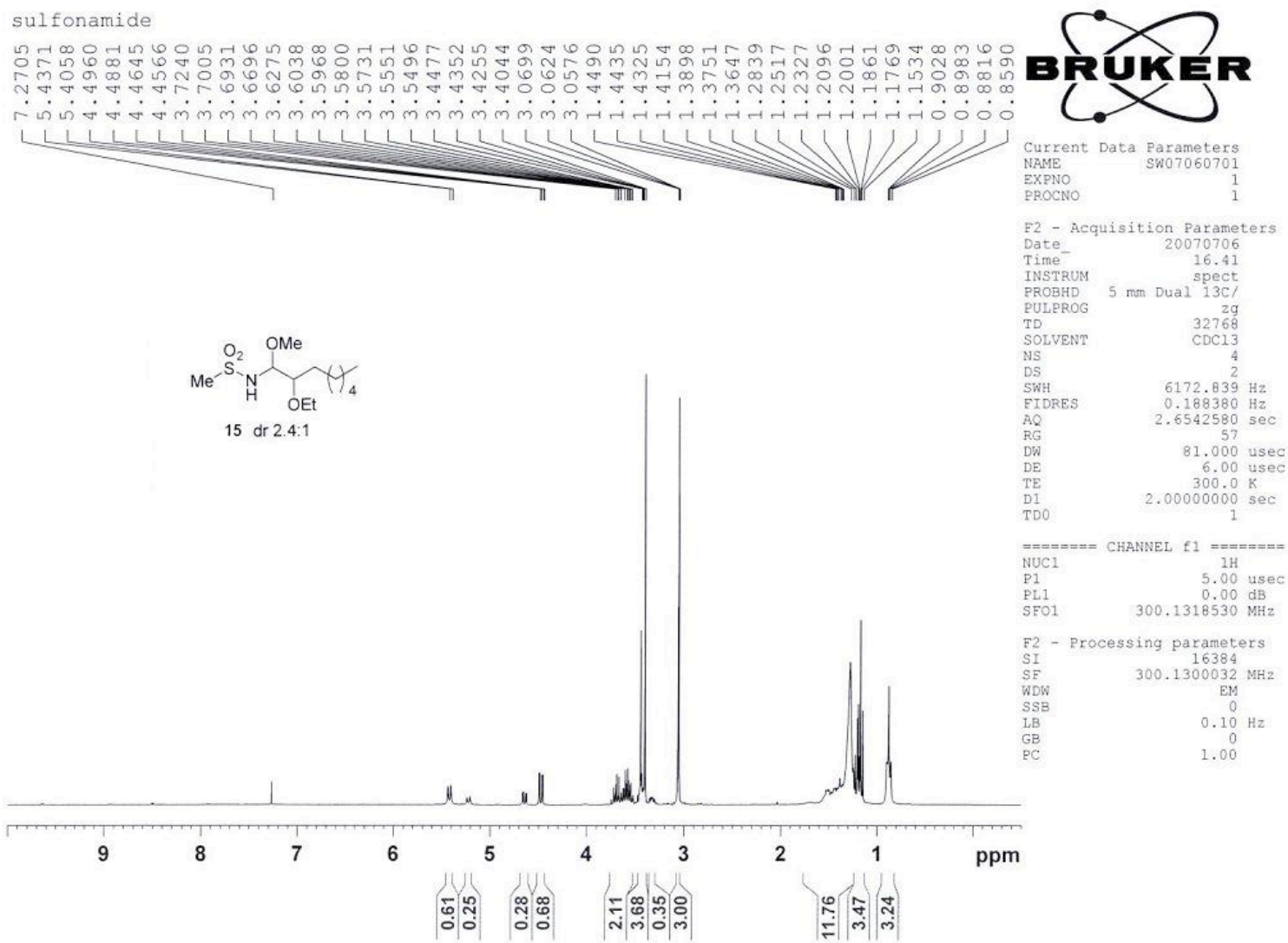

lower spot
170.37
81.03
79.84
77.66
77.23
76.81
72.01
66.48
59.44
56.52
31.94
29.89
29.63
25.72
22.77
15.74
14.26
BRUKER
MeO
O
OMe
N
H
OEt
4
Current Data Parameters
NAME SW02060704
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070206
Time 14.10
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 19
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 5792.6
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305955 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
160
140
120
100
80
60
40
20
ppm


spot 1
7.3861
7.3797
7.3707
7.3534
7.3488
7.3427
7.3251
7.2704
5.6756
5.6428
5.1856
5.1455
4.8387
4.8354
4.8056
4.8019
3.7698
3.7463
3.7388
3.7154
3.6021
3.5787
3.5713
3.5552
3.5476
3.5318
3.5242
3.4761
3.4627
3.4401
3.3677
1.4573
1.3882
1.3733
1.3552
1.3316
1.2801
1.2088
1.1896
1.1661
1.1427
0.9122
0.8908
0.8676
BnO
O
N
H
OMe
OEt
4
14
9
8
7
6
5
4
3
2
1
ppm
4.57
0.80
2.16
0.85
1.00
1.14
1.06
2.91
11.23
3.19
3.26
BRUKER
Current Data Parameters
NAME SW05040701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070504
Time 14.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 71.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300032 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

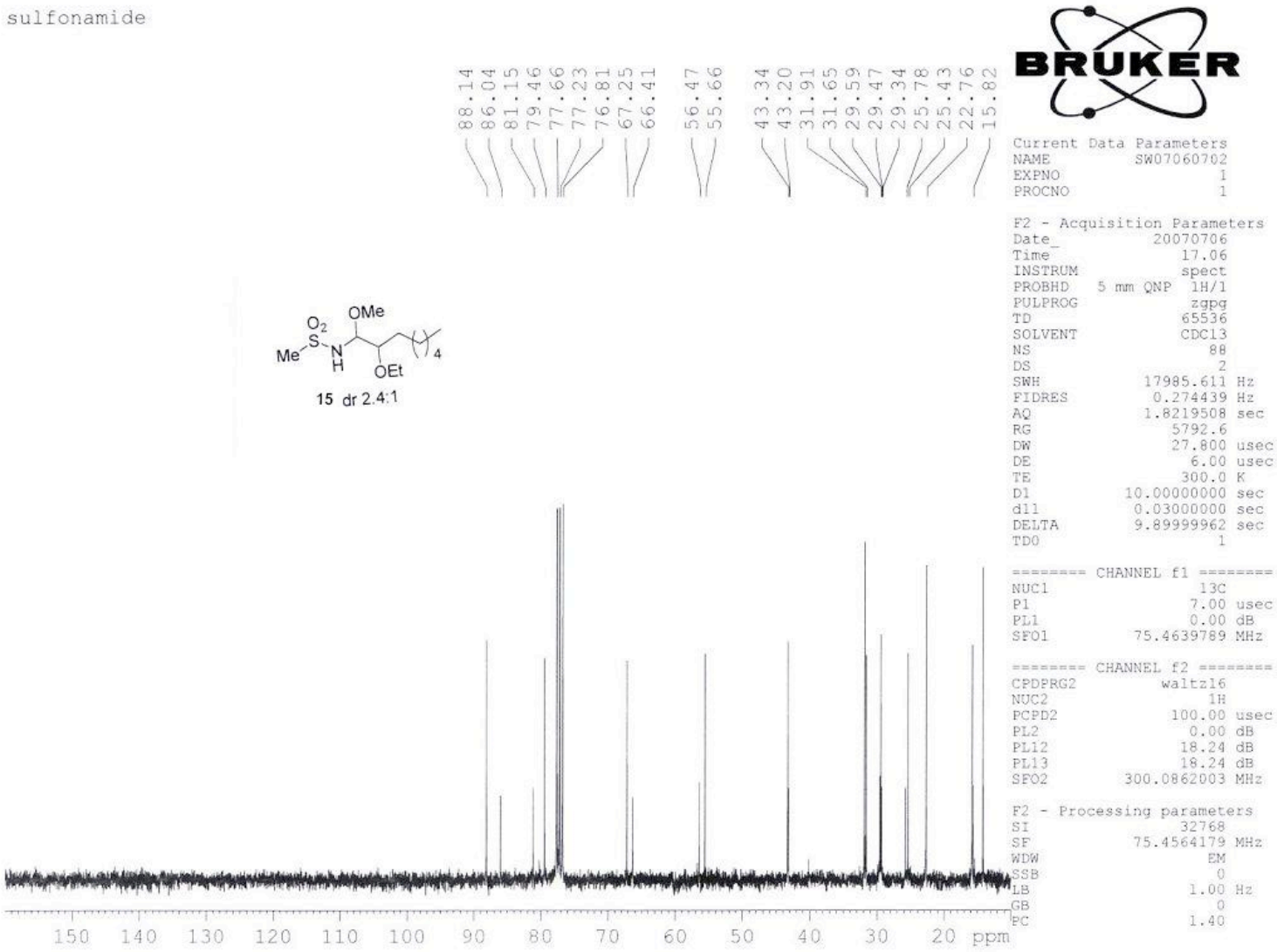

spot 1
156.83
136.56
128.74
128.37
128.22
85.63
80.62
77.66
77.23
76.81
67.38
67.07
55.67
31.91
29.46
25.70
22.78
15.87
14.26
BnO
O
OMe
N
H
OEt
4
14
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME SW05040702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070504
Time 13.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 34
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz
F2 - Processing parameters
SI 32768
SF 75.4564183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


spot 2
7.3961
7.3889
7.3803
7.3640
7.3534
7.3359
7.2707
5.5615
5.5282
5.1897
5.1492
5.1430
4.9635
4.9538
4.9297
4.9199
3.6618
3.6384
3.6312
3.6077
3.5384
3.5149
3.5077
3.4917
3.4843
3.4683
3.4609
3.4376
3.3859
3.3049
3.2952
3.2823
3.2725
3.2599
3.2500
1.5679
1.3073
1.2952
1.2243
1.2010
1.1776
0.9156
0.8942
0.8714
BRUKER
Current Data Parameters
NAME SW05070705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070507
Time 16.25
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 574.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300032 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
O
OMe
BnO
N
H
OEt
4
9
8
7
6
5
4
3
2
1
ppm
4.52
0.70
1.92
0.78
1.00
1.08
2.57
1.21
2.09
8.63
3.03
3.04


lower from CbzCl
156.55
136.49
128.79
128.46
128.36
82.94
81.08
77.66
77.44
77.23
76.81
67.15
66.17
56.31
31.94
29.70
29.65
25.68
22.79
15.78
14.29
BnO
O
N
H
OMe
OEt
4
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SW05080707
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time 20.28
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 402
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 5160.6
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


sulfonamide
7.2705
5.4371
5.4058
4.4960
4.4881
4.4645
4.4566
3.7240
3.7005
3.6931
3.6696
3.6275
3.6038
3.5968
3.5800
3.5731
3.5551
3.5496
3.4477
3.4352
3.4255
3.4044
3.0699
3.0624
3.0576
1.4490
1.4435
1.4325
1.4154
1.3898
1.3751
1.3647
1.2839
1.2517
1.2327
1.2096
1.2001
1.1861
1.1769
1.1534
0.9028
0.8983
0.8816
0.8590
BRUKER
Current Data Parameters
NAME SW07060701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070706
Time 16.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 57
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300032 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
OMe
O2
S
Me
N
H
OEt
4
15 dr 2.4:1
9
8
7
6
5
4
3
2
1
ppm
0.61
0.25
0.28
0.68
2.11
3.68
0.35
3.00
11.76
3.47
3.24

sulfonamide



BRUKER

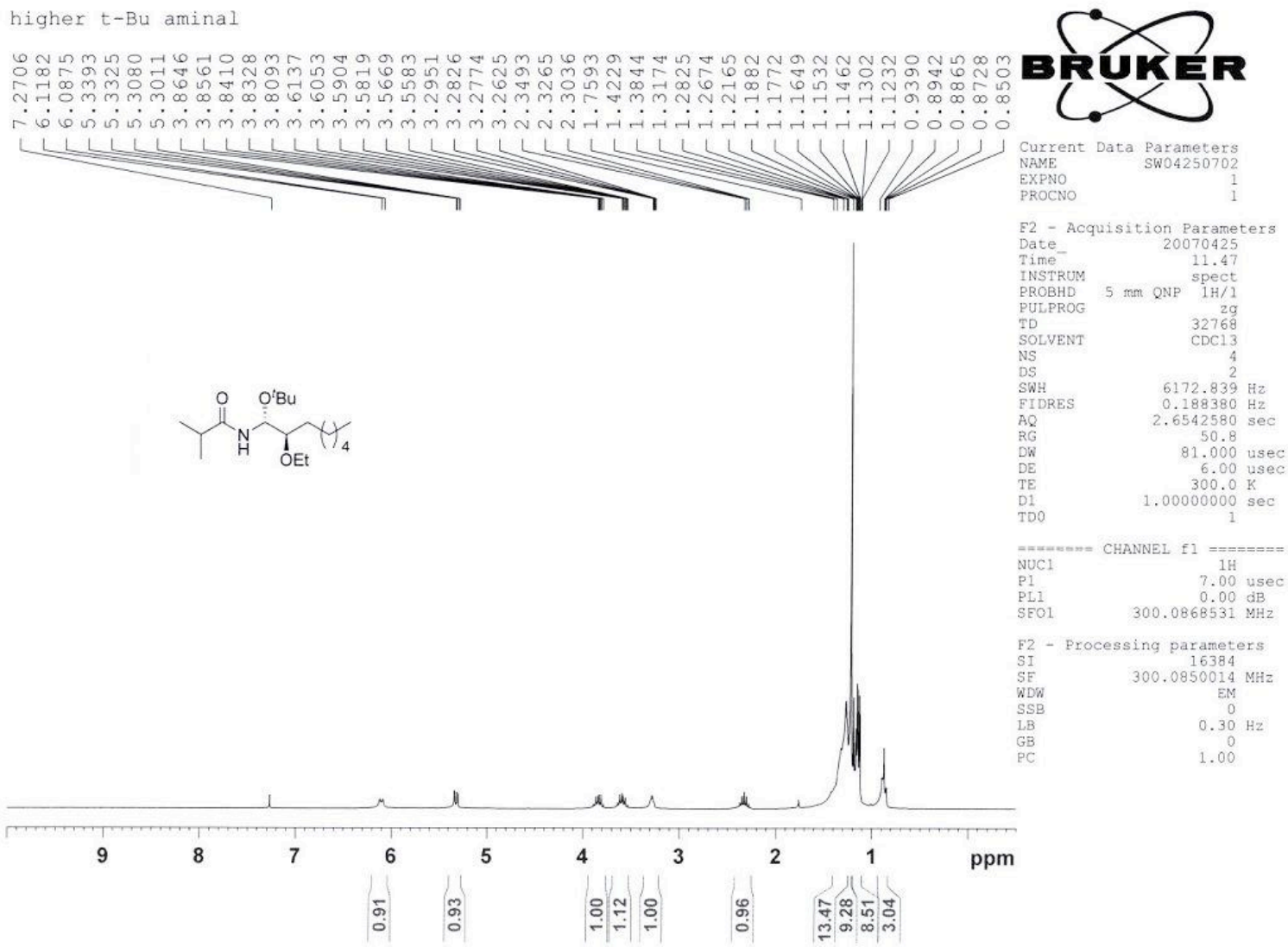
Current Data Parameters
NAME SW07060702
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070706
Time 17.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 88
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 5792.6
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

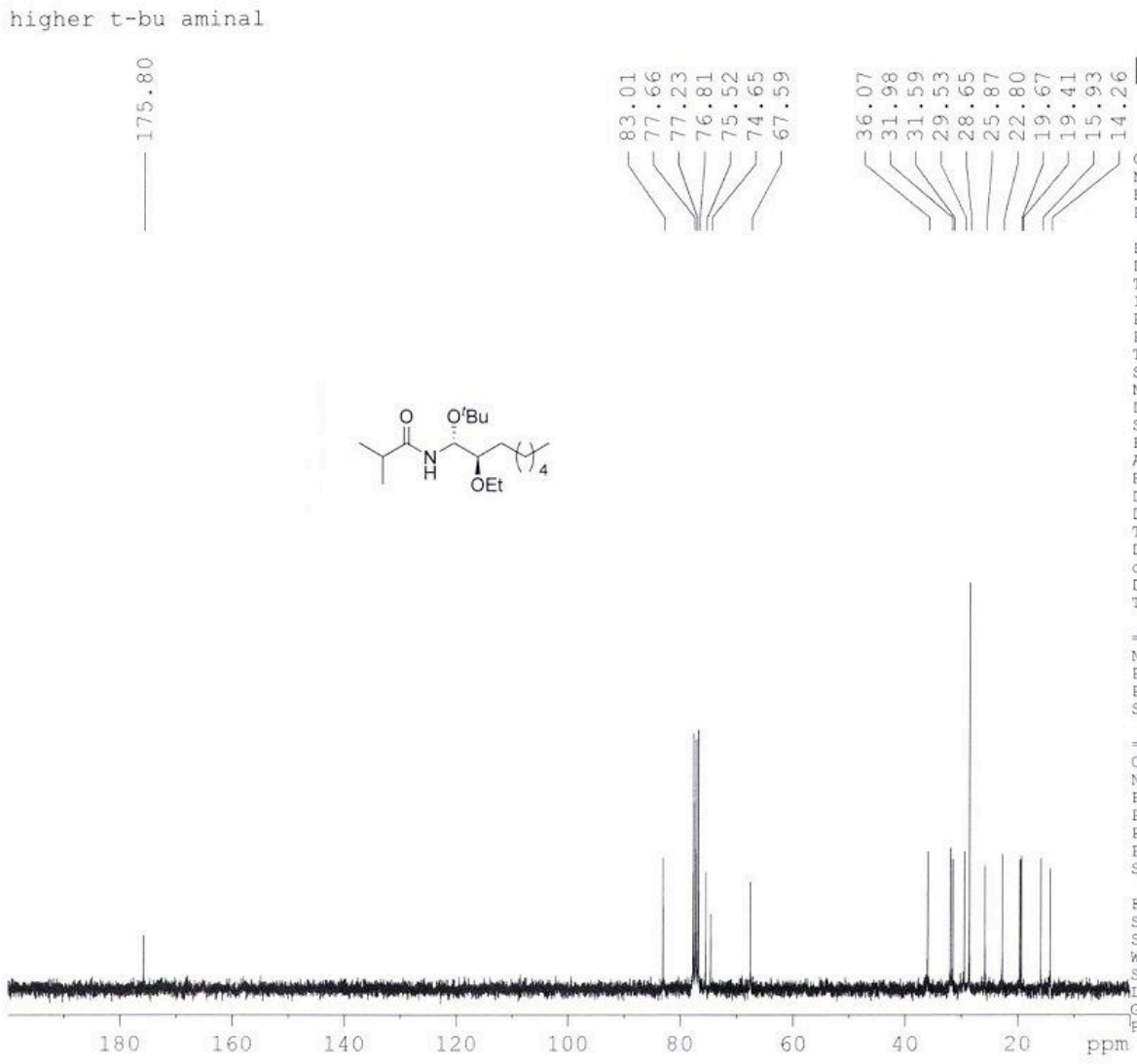
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz

F2 - Processing parameters
SI 32768
SF 75.4564179 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


higher t-Bu aminal
7.2706
6.1182
6.0875
5.3393
5.3325
5.3080
5.3011
3.8646
3.8561
3.8410
3.8328
3.8093
3.6137
3.6053
3.5904
3.5819
3.5669
3.5583
3.2951
3.2826
3.2774
3.2625
2.3493
2.3265
2.3036
1.7593
1.4229
1.3844
1.3174
1.2825
1.2674
1.2165
1.1882
1.1772
1.1649
1.1532
1.1462
1.1302
1.1232
0.9390
0.8942
0.8865
0.8728
0.8503
BRUKER
Current Data Parameters
NAME SW04250702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070425
Time 11.47
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 50.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 0.00 dB
SFO1 300.0868531 MHz
F2 - Processing parameters
SI 16384
SF 300.0850014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O'Bu
N
H
OEt
4
9
8
7
6
5
4
3
2
1
ppm
0.91
0.93
1.00
1.12
1.00
0.96
13.47
9.28
8.51
3.04

higher t-bu aminal



BRUKER

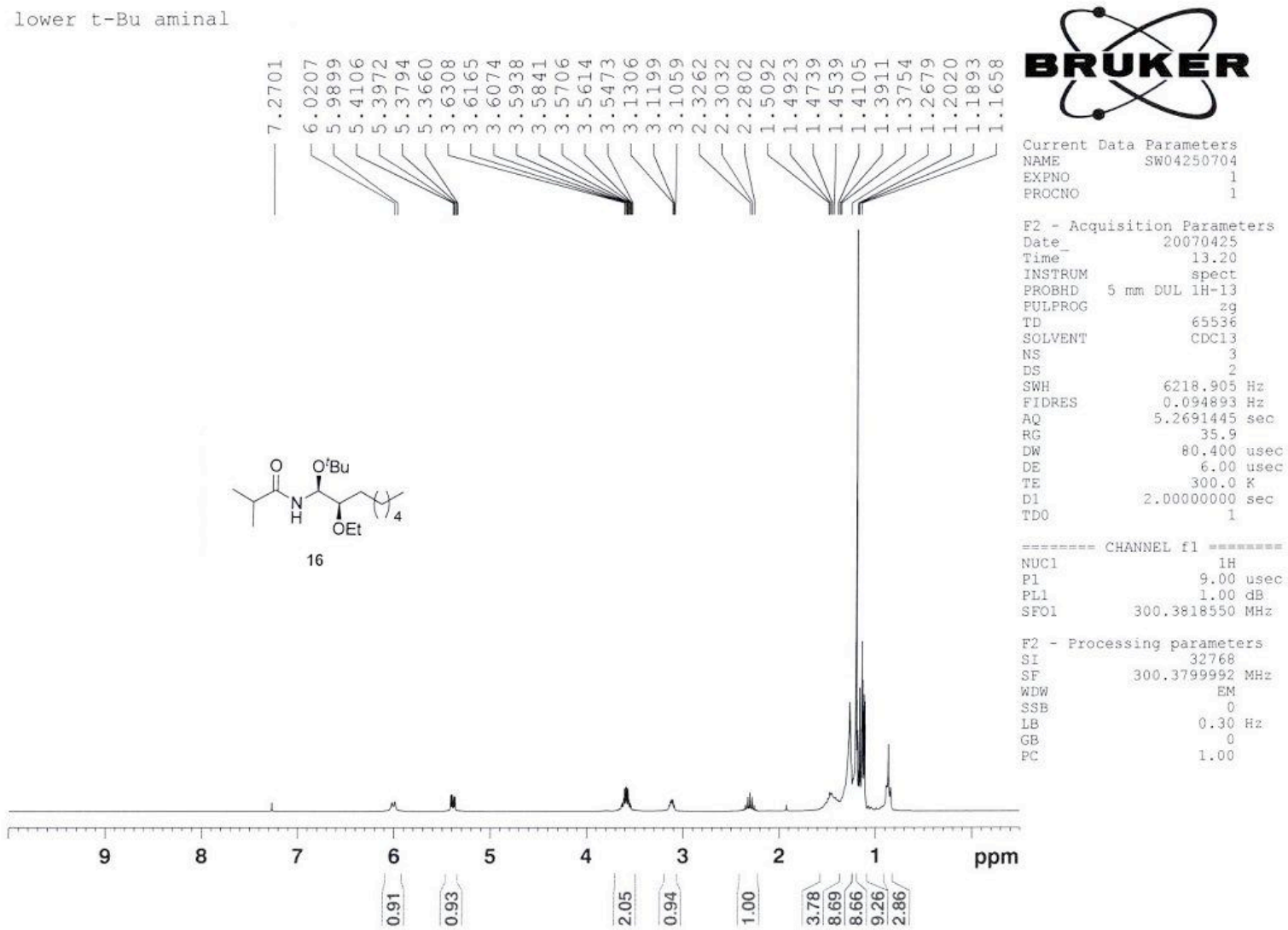
Current Data Parameters
NAME SW04250701
EXPNO 1
PROCNO 1

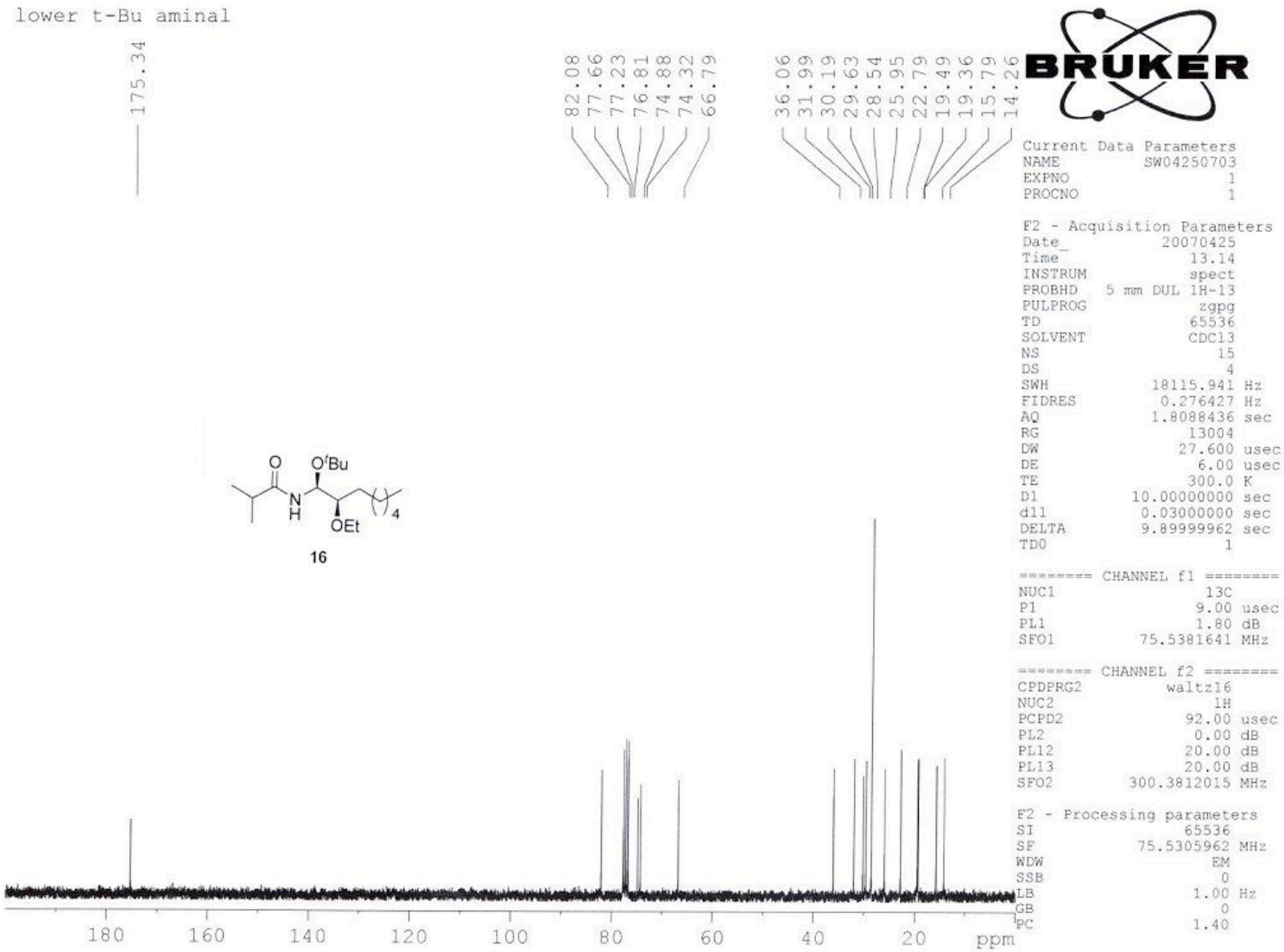
F2 - Acquisition Parameters
Date_ 20070425
Time 11.37
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDC13
NS 56
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

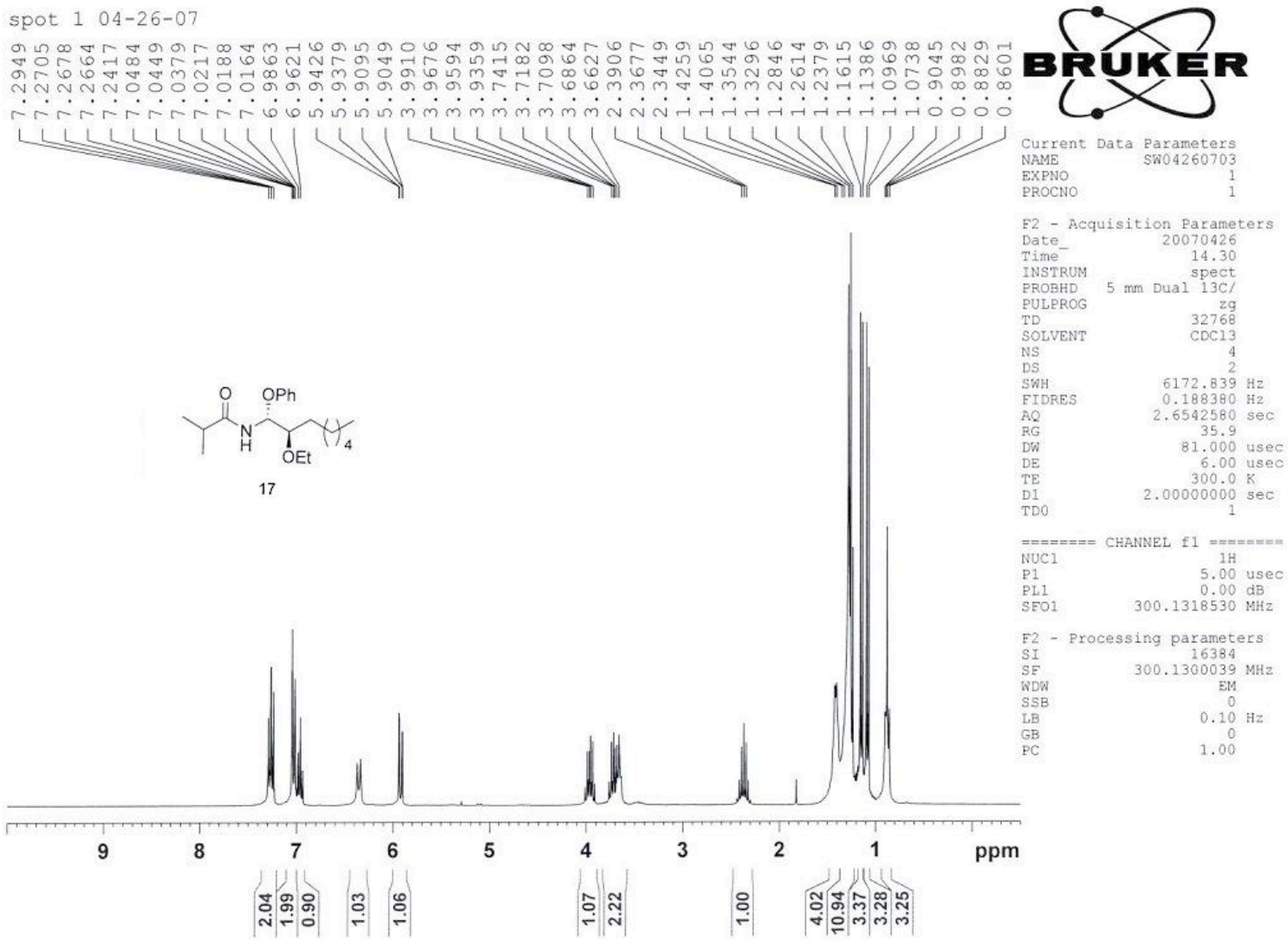
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz

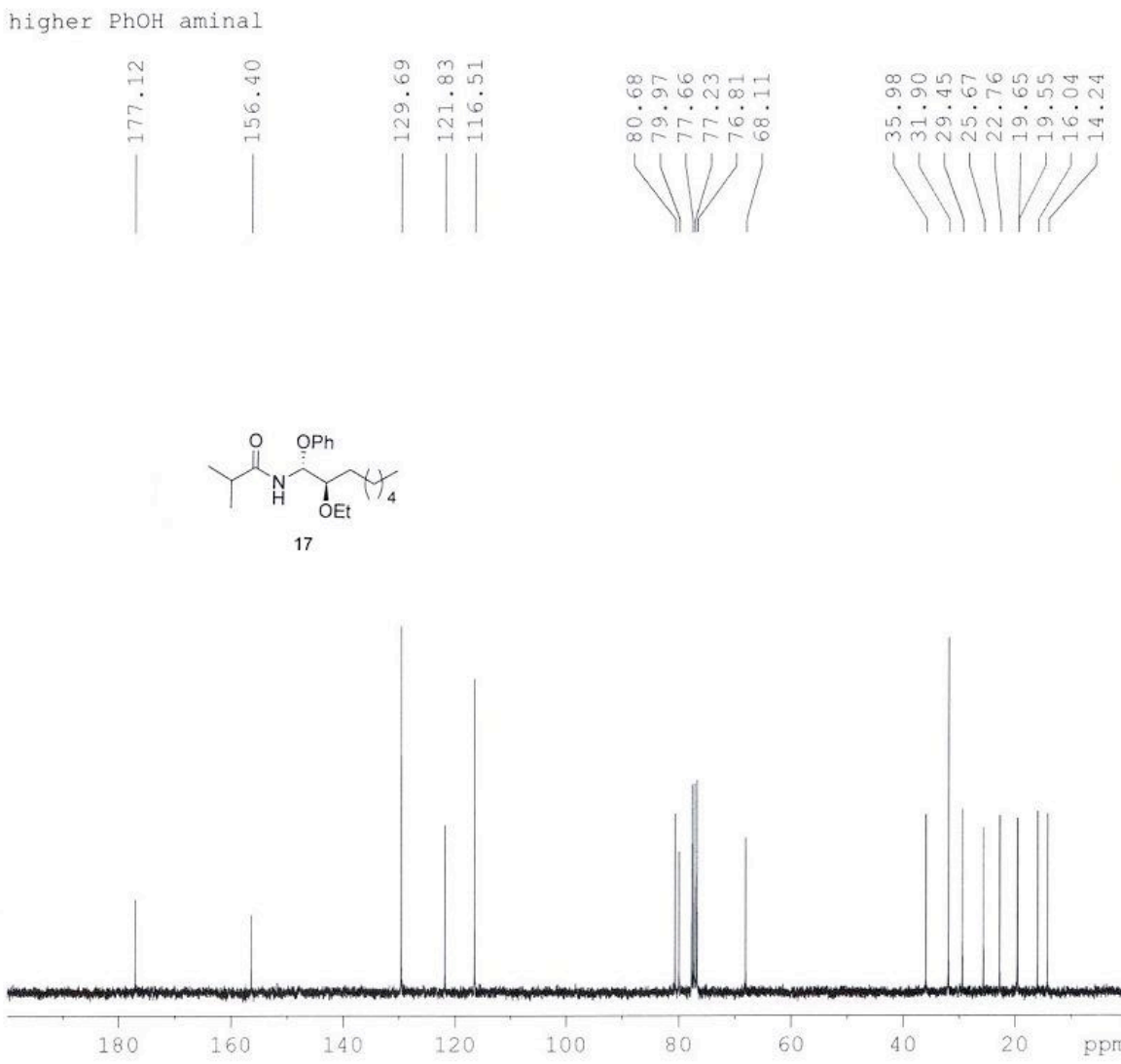
F2 - Processing parameters
SI 32768
SF 75.4564164 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


lower t-Bu aminal
7.2701
6.0207
5.9899
5.4106
5.3972
5.3794
5.3660
3.6308
3.6165
3.6074
3.5938
3.5841
3.5706
3.5614
3.5473
3.1306
3.1199
3.1059
2.3262
2.3032
2.2802
1.5092
1.4923
1.4739
1.4539
1.4105
1.3911
1.3754
1.2679
1.2020
1.1893
1.1658
O
O'Bu
N
H
OEt
4
16
9
8
7
6
5
4
3
2
1
ppm
0.91
0.93
2.05
0.94
1.00
3.78
8.69
8.66
9.26
2.86
BRUKER
Current Data Parameters
NAME SW04250704
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070425
Time 13.20
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 35.9
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


lower t-Bu aminal
175.34
82.08
77.66
77.23
76.81
74.88
74.32
66.79
36.06
31.99
30.19
29.63
28.54
25.95
22.79
19.49
19.36
15.79
14.26
BRUKER
Current Data Parameters
NAME SW04250703
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070425
Time 13.14
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 15
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 13004
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305962 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O'Bu
N
H
OEt
4
16
180
160
140
120
100
80
60
40
20
ppm


spot 1 04-26-07
7.2949
7.2705
7.2678
7.2664
7.2417
7.0484
7.0449
7.0379
7.0217
7.0188
7.0164
6.9863
6.9621
5.9426
5.9379
5.9095
5.9049
3.9910
3.9676
3.9594
3.9359
3.7415
3.7182
3.7098
3.6864
3.6627
2.3906
2.3677
2.3449
1.4259
1.4065
1.3544
1.3296
1.2846
1.2614
1.2379
1.1615
1.1386
1.0969
1.0738
0.9045
0.8982
0.8829
0.8601
BRUKER
Current Data Parameters
NAME SW04260703
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070426
Time 14.30
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 32768
SOLVENT CDC13
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
O
OPh
N
H
OEt
4
17
9
8
7
6
5
4
3
2
1
ppm
2.04
1.99
0.90
1.03
1.06
1.07
2.22
1.00
4.02
10.94
3.37
3.28
3.25

higher PhOH aminal



BRUKER

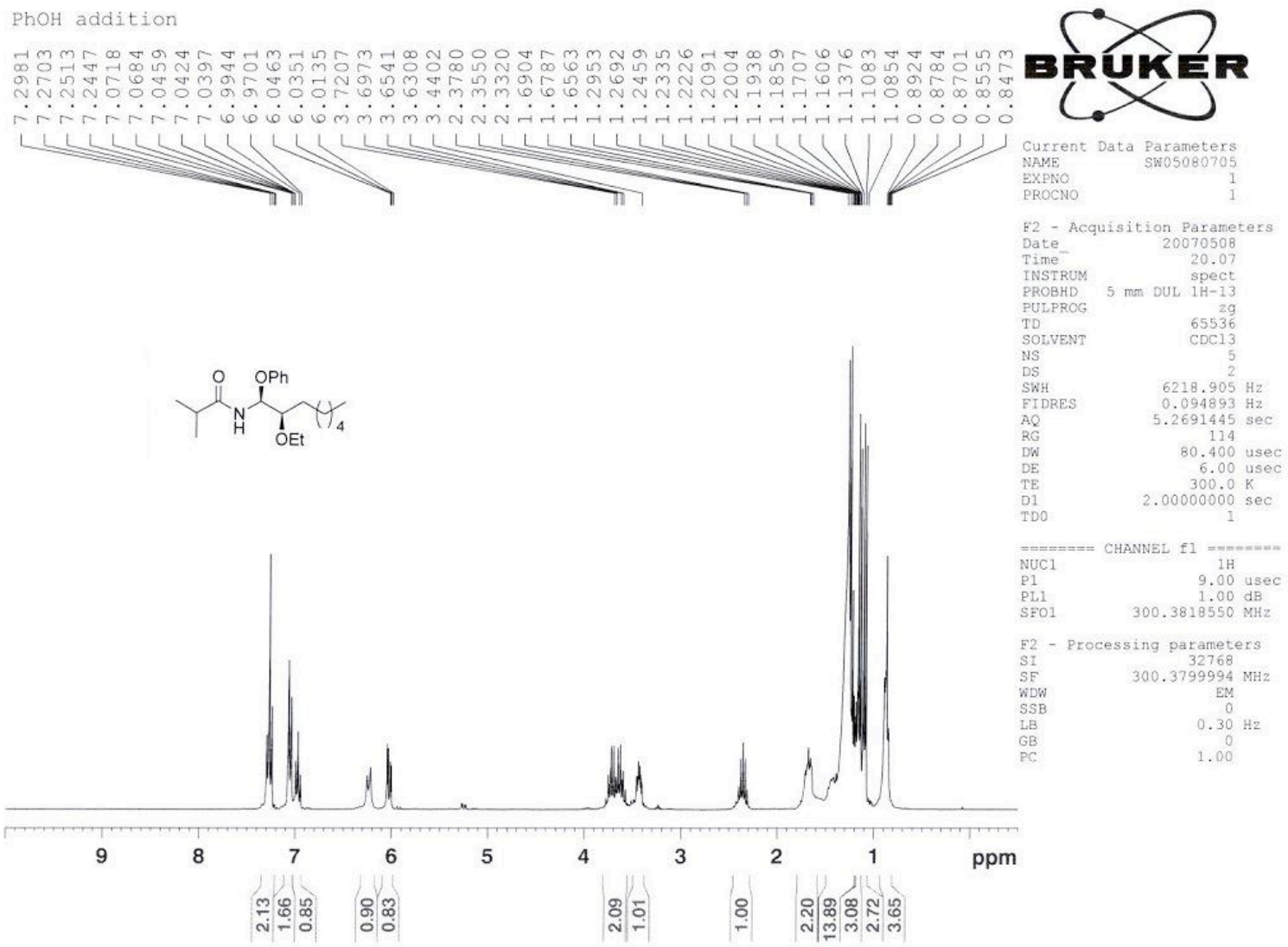
Current Data Parameters
NAME SW04280701
EXPNO 1
PROCNO 1

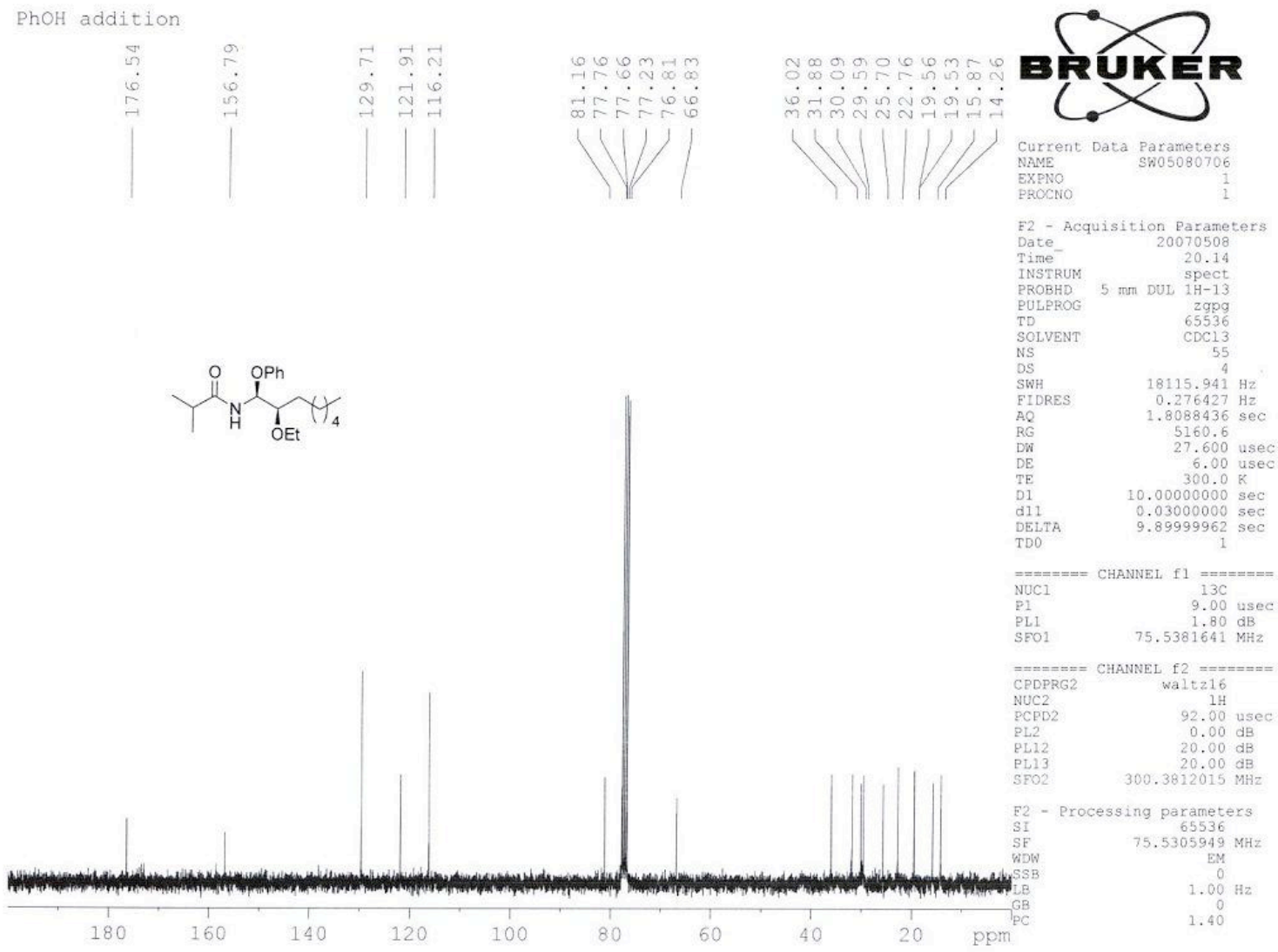
F2 - Acquisition Parameters
Date_ 20070428
Time 12.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 57
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

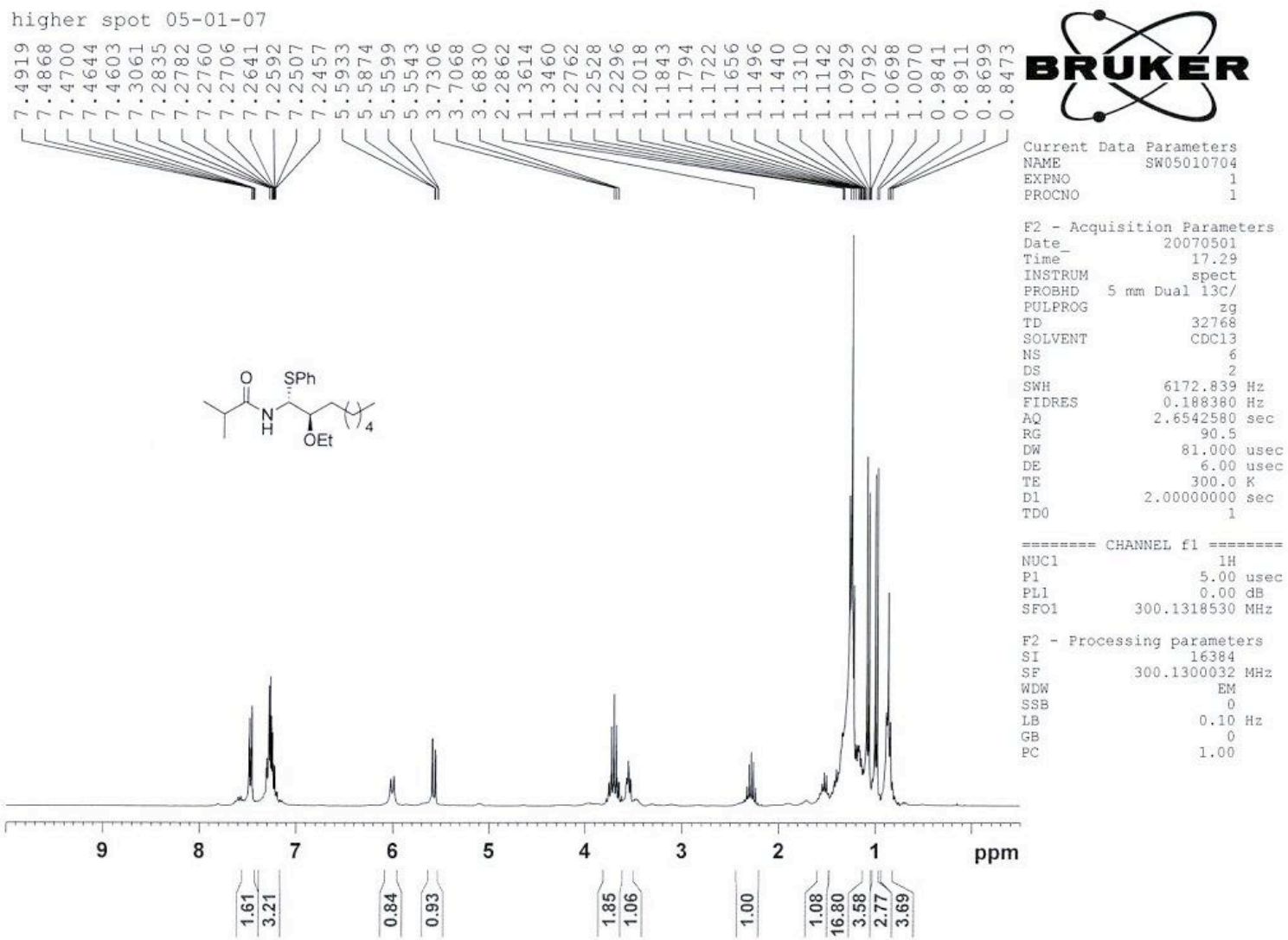
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz

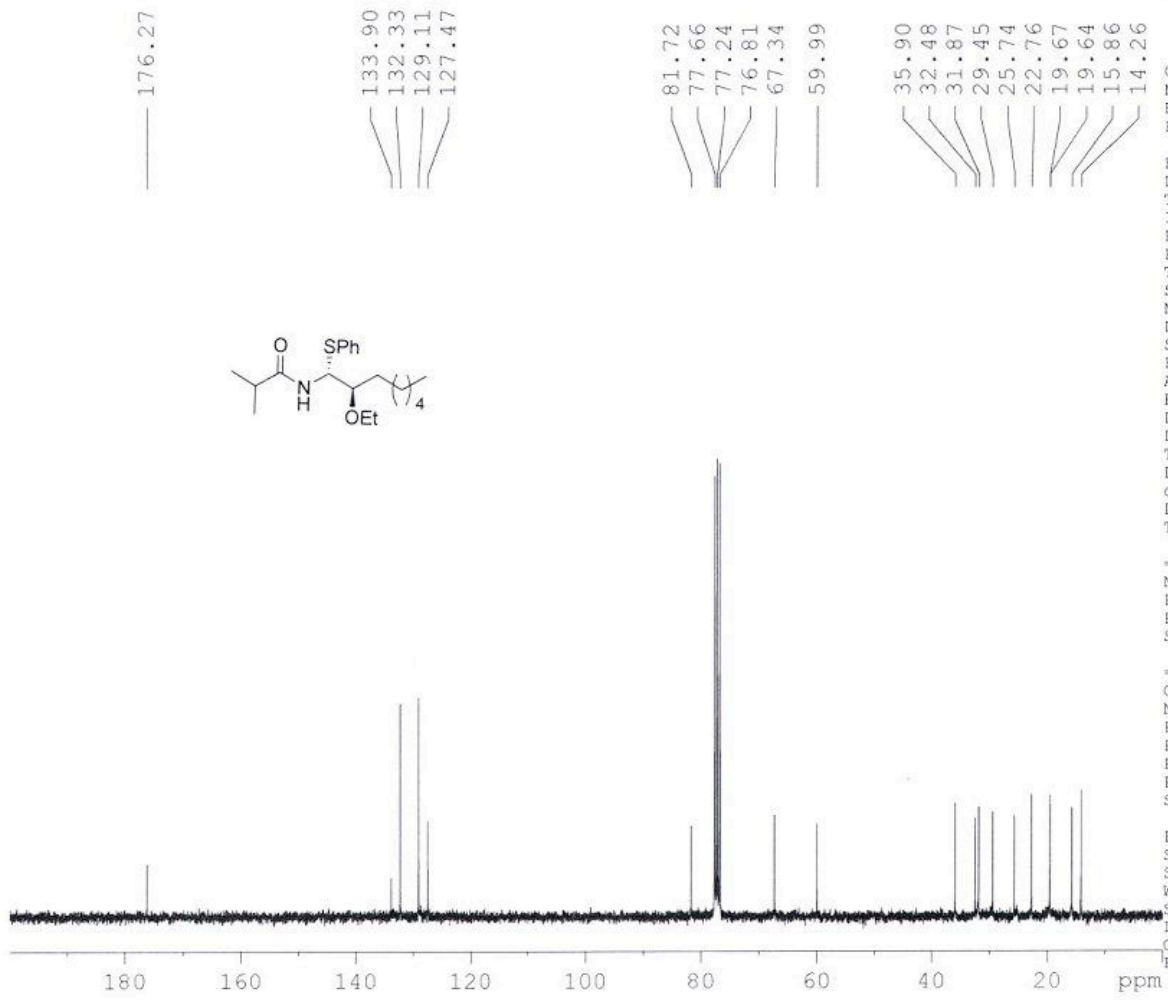
F2 - Processing parameters
SI 32768
SF 75.4564179 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


PhOH addition
7.2981
7.2703
7.2513
7.2447
7.0718
7.0684
7.0459
7.0424
7.0397
6.9944
6.9701
6.0463
6.0351
6.0135
3.7207
3.6973
3.6541
3.6308
3.4402
3.3780
2.3550
2.3320
1.6904
1.6787
1.6563
1.2953
1.2692
1.2459
1.2335
1.2226
1.2091
1.2004
1.1938
1.1859
1.1707
1.1606
1.1376
1.1083
1.0854
0.8924
0.8784
0.8701
0.8555
0.8473
BRUKER
Current Data Parameters
NAME SW05080705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time 20.07
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 114
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799994 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OPh
N
H
OEt
4
9
8
7
6
5
4
3
2
1
ppm
2.13
1.66
0.85
0.90
0.83
2.09
1.01
1.00
2.20
13.89
3.08
2.72
3.65


PhOH addition
176.54
156.79
129.71
121.91
116.21
81.16
77.76
77.66
77.23
76.81
66.83
36.02
31.88
30.09
29.59
25.70
22.76
19.56
19.53
15.87
14.26
BRUKER
Current Data Parameters
NAME SW05080706
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time 20.14
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 55
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 5160.6
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305949 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OPh
N
H
OEt
4
180
160
140
120
100
80
60
40
20
ppm


higher spot 05-01-07
7.4919
7.4868
7.4700
7.4644
7.4603
7.3061
7.2835
7.2782
7.2760
7.2706
7.2641
7.2592
7.2507
7.2457
5.5933
5.5874
5.5599
5.5543
3.7306
3.7068
3.6830
2.2862
1.3614
1.3460
1.2762
1.2528
1.2296
1.2018
1.1843
1.1794
1.1722
1.1656
1.1496
1.1440
1.1310
1.1142
1.0929
1.0792
1.0698
1.0070
0.9841
0.8911
0.8699
0.8473
O
SPh
N
H
OEt
4
9
8
7
6
5
4
3
2
1
ppm
1.61
3.21
0.84
0.93
1.85
1.06
1.00
1.08
16.80
3.58
2.77
3.69
BRUKER
Current Data Parameters
NAME SW05010704
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070501
Time 17.29
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 6
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 16384
SF 300.1300032 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

higher spot



BRUKER

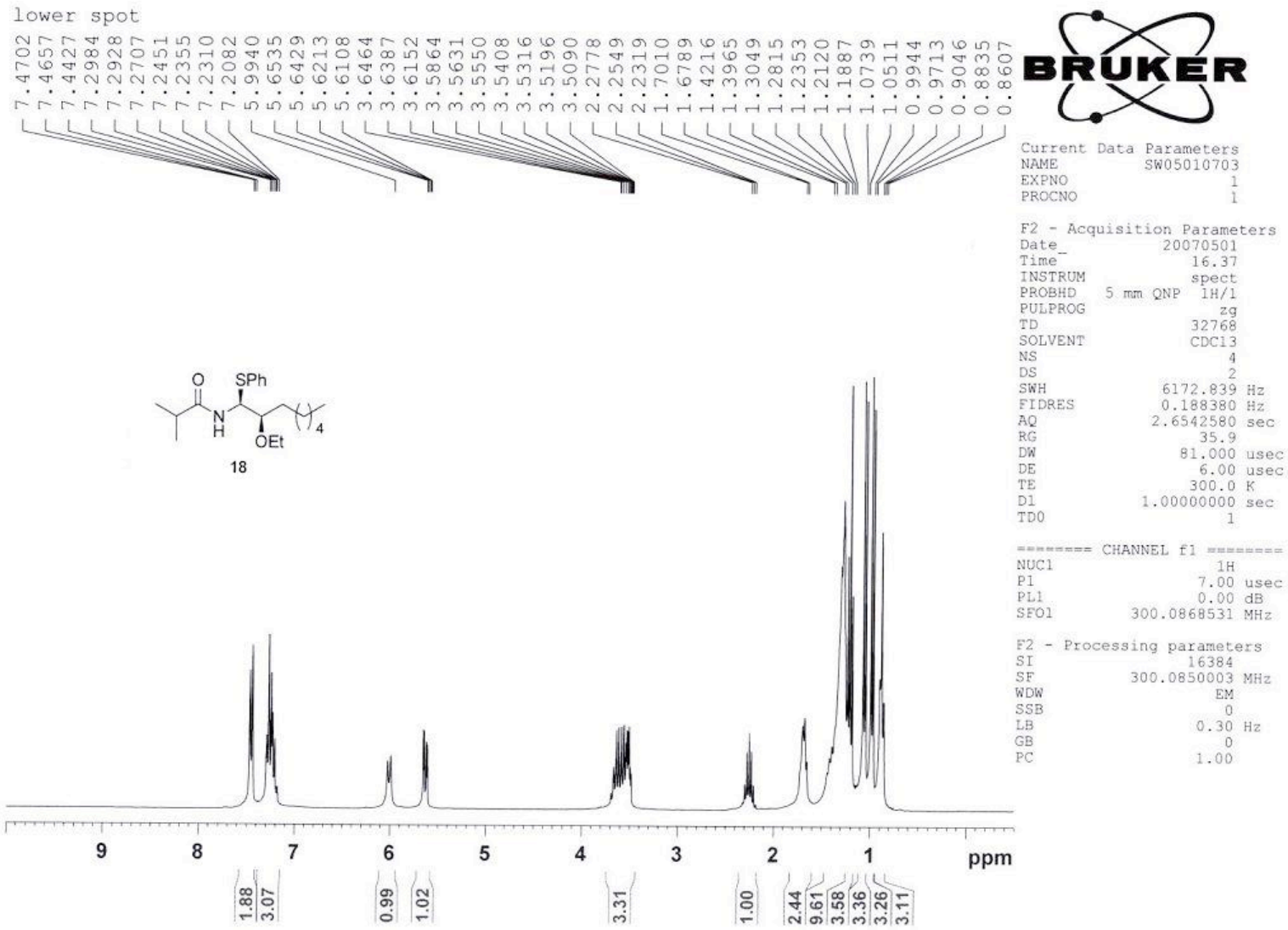
Current Data Parameters
NAME SW05020703
EXPNO 1
PROCNO 1

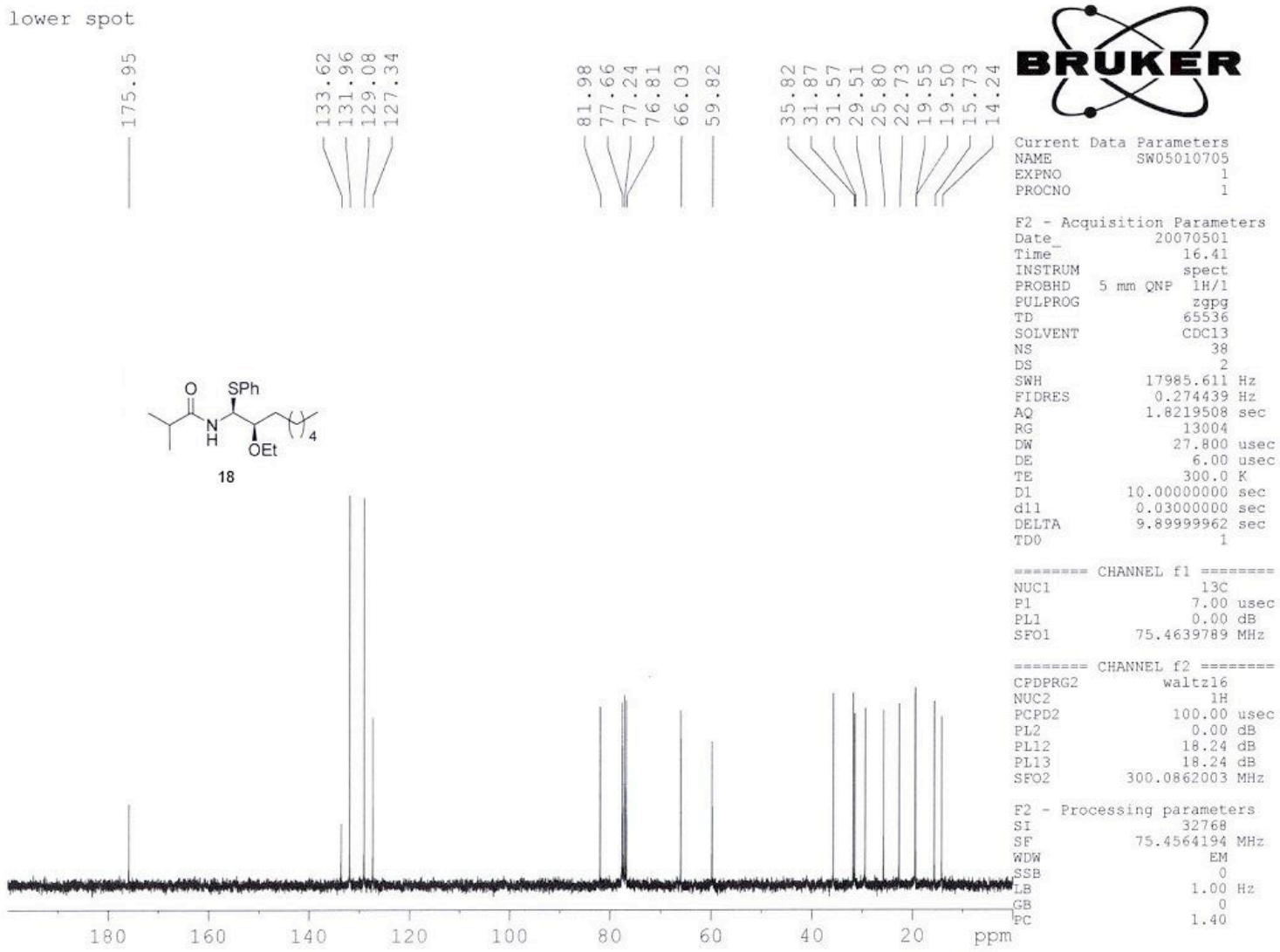
F2 - Acquisition Parameters
Date_ 20070502
Time 20.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDC13
NS 275
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

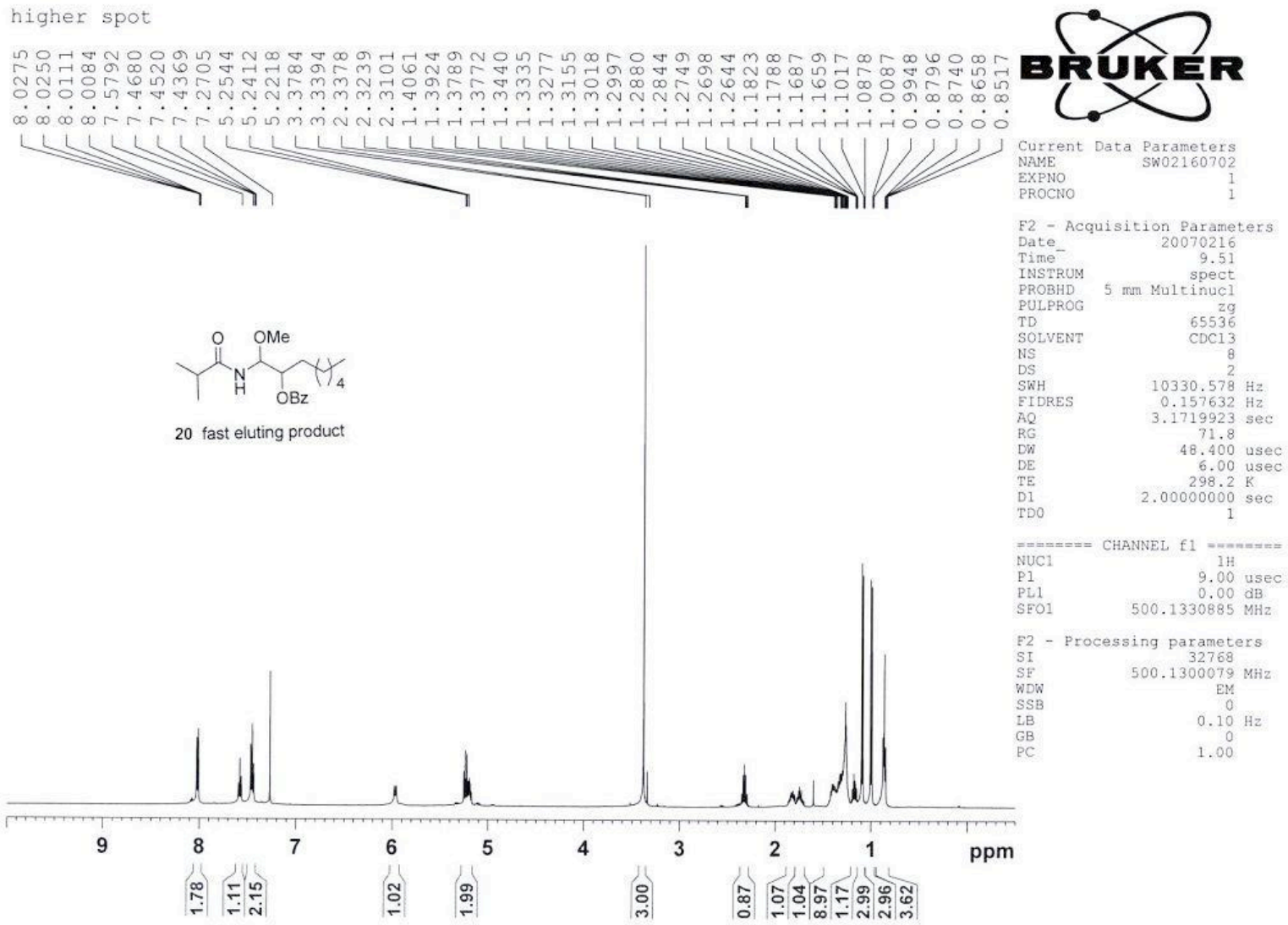
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz

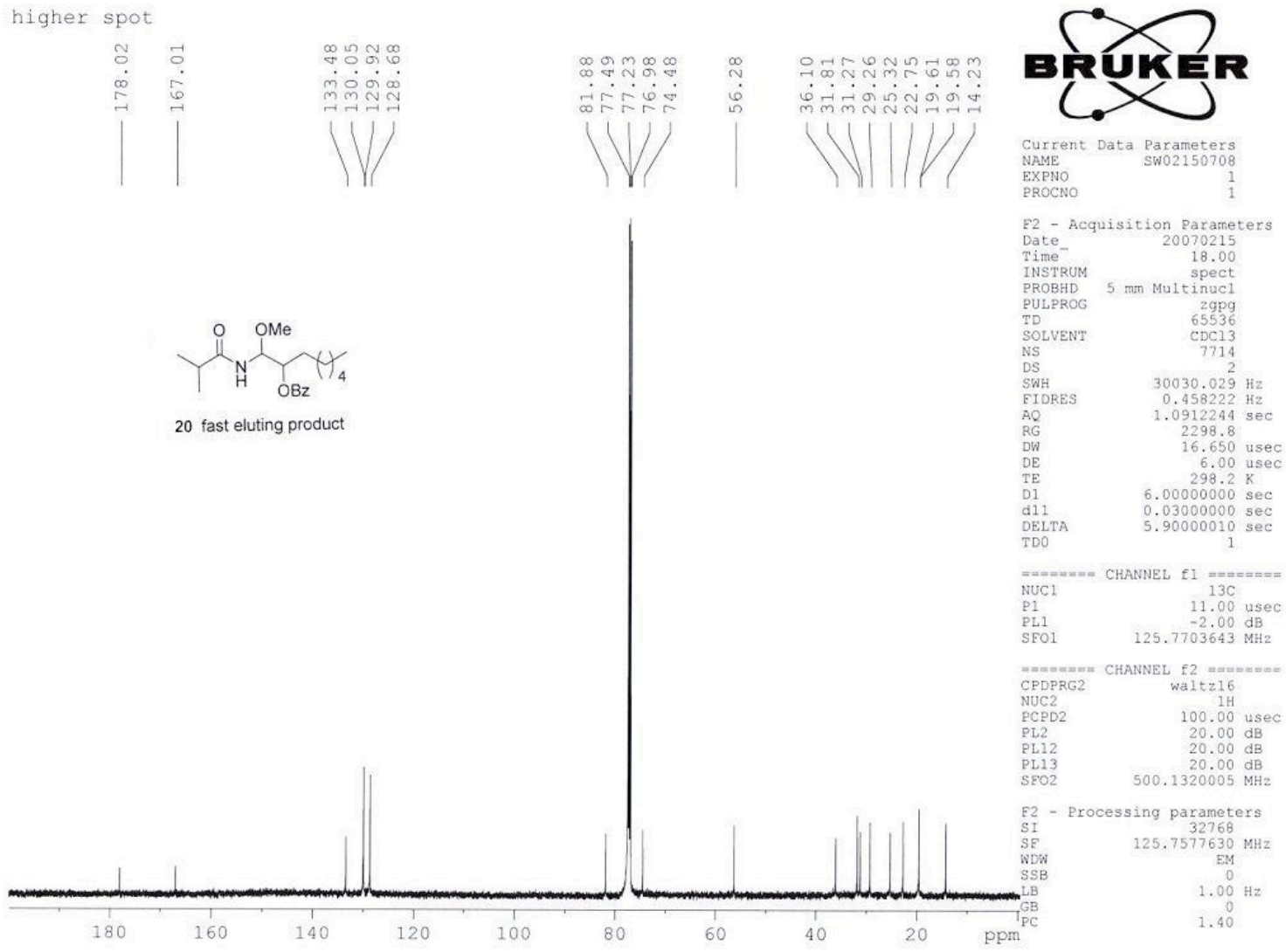
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz

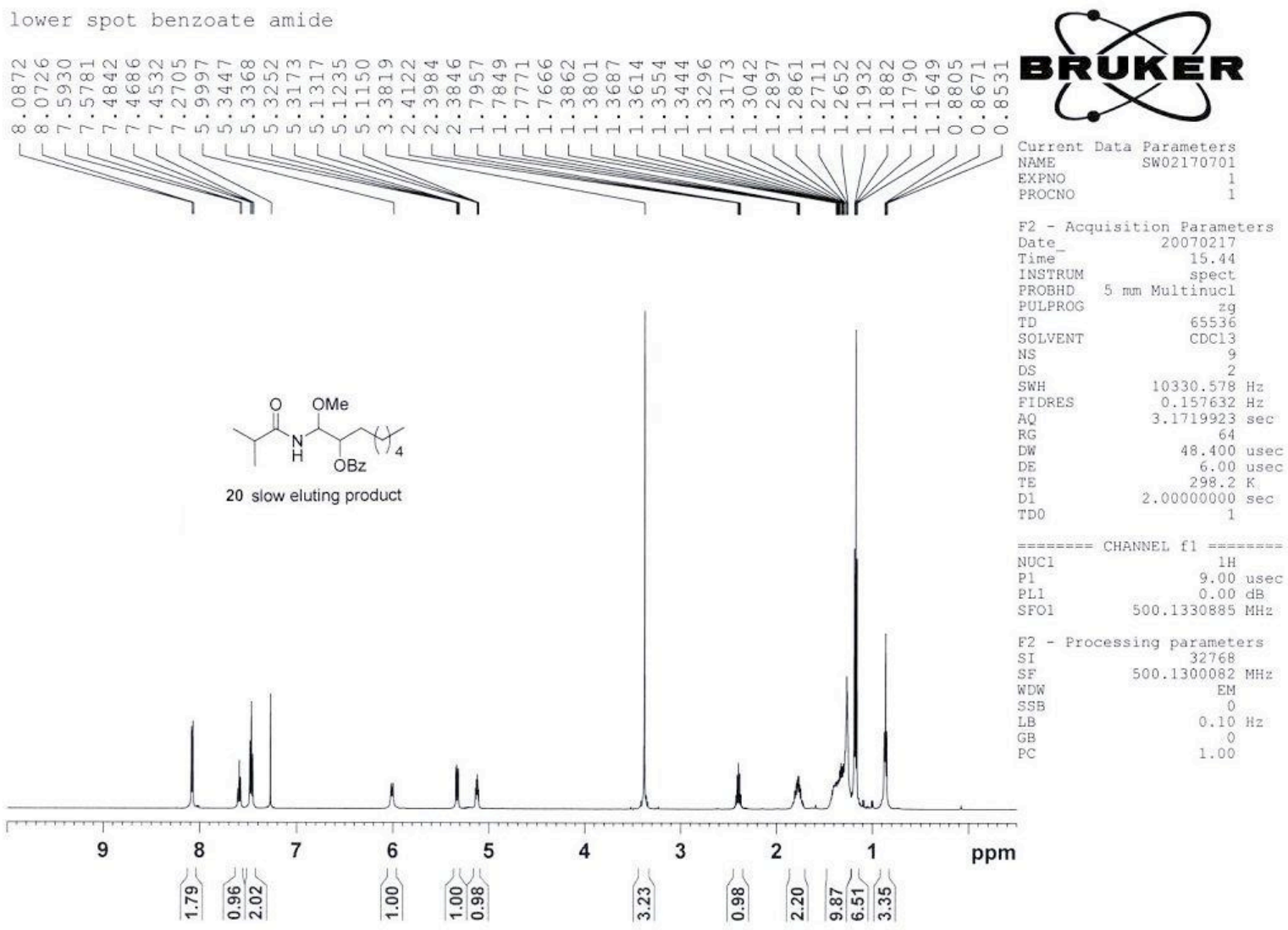
F2 - Processing parameters
SI 32768
SF 75.4564167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


lower spot
7.4702
7.4657
7.4427
7.2984
7.2928
7.2707
7.2451
7.2355
7.2310
7.2082
5.9940
5.6535
5.6429
5.6213
5.6108
3.6464
3.6387
3.6152
3.5864
3.5631
3.5550
3.5408
3.5316
3.5196
3.5090
2.2778
2.2549
2.2319
1.7010
1.6789
1.4216
1.3965
1.3049
1.2815
1.2353
1.2120
1.1887
1.0739
1.0511
0.9944
0.9713
0.9046
0.8835
0.8607
BRUKER
Current Data Parameters
NAME SW05010703
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070501
Time 16.37
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 0.00 dB
SFO1 300.0868531 MHz
F2 - Processing parameters
SI 16384
SF 300.0850003 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
SPh
N
H
OEt
4
18
9
8
7
6
5
4
3
2
1
ppm
1.88
3.07
0.99
1.02
3.31
1.00
2.44
9.61
3.58
3.36
3.26
3.11

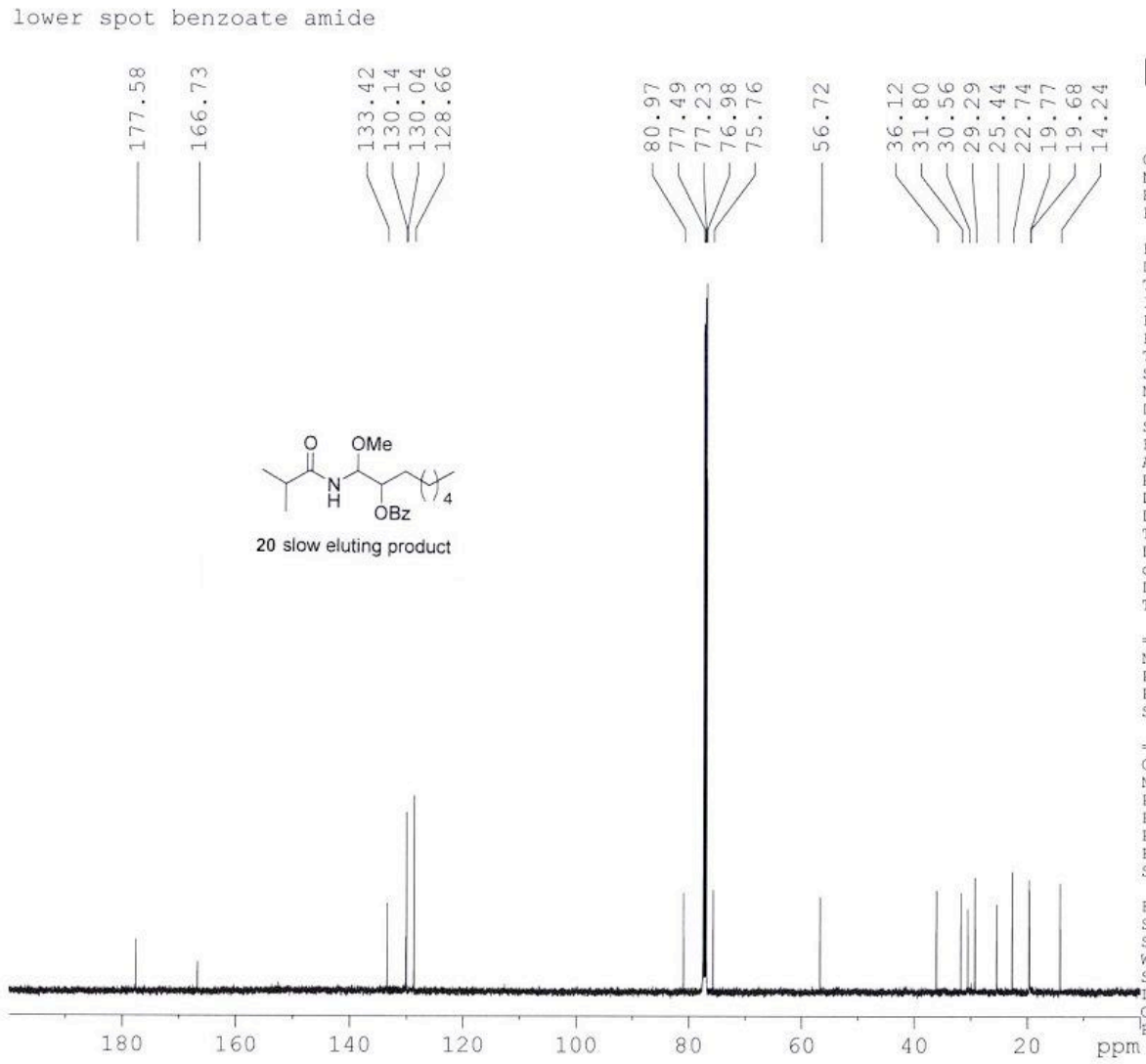

lower spot
175.95
133.62
131.96
129.08
127.34
81.98
77.66
77.24
76.81
66.03
59.82
35.82
31.87
31.57
29.51
25.80
22.73
19.55
19.50
15.73
14.24
BRUKER
Current Data Parameters
NAME SW05010705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070501
Time_ 16.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 38
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz
F2 - Processing parameters
SI 32768
SF 75.4564194 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
SPh
N
H
OEt
4
18
180
160
140
120
100
80
60
40
20
ppm


higher spot
8.0275
8.0250
8.0111
8.0084
7.5792
7.4680
7.4520
7.4369
7.2705
5.2544
5.2412
5.2218
3.3784
3.3394
3.3378
2.3239
2.3101
1.4061
1.3924
1.3789
1.3772
1.3440
1.3335
1.3277
1.3155
1.3018
1.2997
1.2880
1.2844
1.2749
1.2698
1.2644
1.1823
1.1788
1.1687
1.1659
1.1017
1.0878
1.0087
0.9948
0.8796
0.8740
0.8658
0.8517
BRUKER
Current Data Parameters
NAME SW02160702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070216
Time_ 9.51
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 71.8
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300079 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
O
OMe
N
H
OBz
4
20 fast eluting product
9
8
7
6
5
4
3
2
1
ppm
1.78
1.11
2.15
1.02
1.99
3.00
0.87
1.07
1.04
8.97
1.17
2.99
2.96
3.62


higher spot
178.02
167.01
133.48
130.05
129.92
128.68
81.88
77.49
77.23
76.98
74.48
56.28
36.10
31.81
31.27
29.26
25.32
22.75
19.61
19.58
14.23
O
OMe
N
H
OBz
4
20 fast eluting product
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SW02150708
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070215
Time 18.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 7714
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 2298.8
DW 16.650 usec
DE 6.00 usec
TE 298.2 K
D1 6.00000000 sec
d11 0.03000000 sec
DELTA 5.90000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 20.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577630 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


lower spot benzoate amide
8.0872
8.0726
7.5930
7.5781
7.4842
7.4686
7.4532
7.2705
5.9997
5.3447
5.3368
5.3252
5.3173
5.1317
5.1235
5.1150
3.3819
2.4122
2.3984
2.3846
1.7957
1.7849
1.7771
1.7666
1.3862
1.3801
1.3687
1.3614
1.3554
1.3444
1.3296
1.3173
1.3042
1.2897
1.2861
1.2711
1.2652
1.1932
1.1882
1.1790
1.1649
0.8805
0.8671
0.8531
BRUKER
Current Data Parameters
NAME SW02170701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070217
Time 15.44
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300082 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
O
OMe
N
H
OBz
4
20 slow eluting product
9
8
7
6
5
4
3
2
1
ppm
1.79
0.96
2.02
1.00
1.00
0.98
3.23
0.98
2.20
9.87
6.51
3.35

lower spot benzoate amide



BRUKER

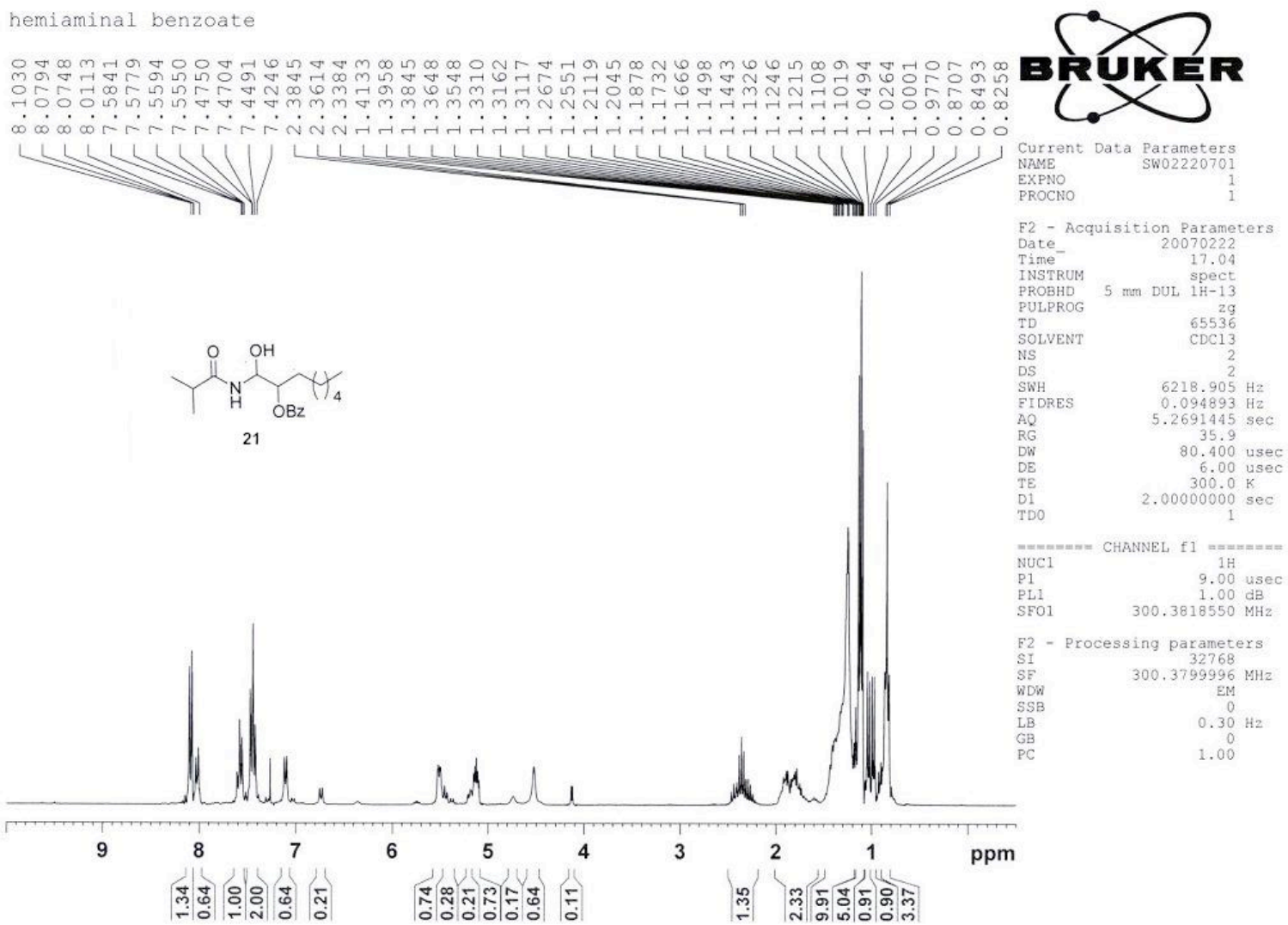
Current Data Parameters
NAME SW02170703
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070217
Time 15.50
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2523
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 2580.3
DW 16.650 usec
DE 6.00 usec
TE 298.2 K
D1 6.00000000 sec
d11 0.03000000 sec
DELTA 5.90000010 sec
TD0 1

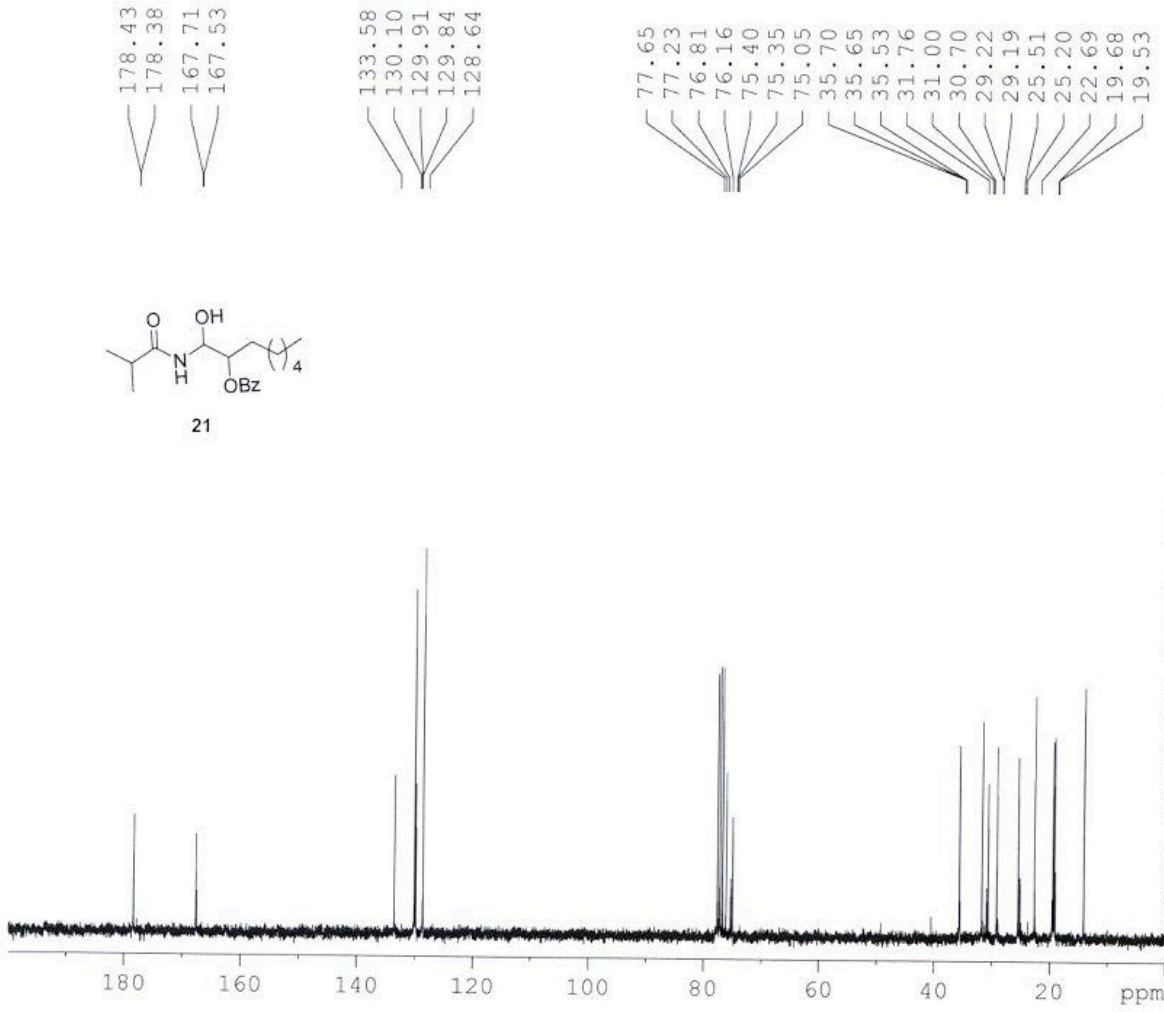
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 20.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577628 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


hemiaminal benzoate
8.1030
8.0794
8.0748
8.0113
7.5841
7.5779
7.5594
7.5550
7.4750
7.4704
7.4491
7.4246
2.3845
2.3614
2.3384
1.4133
1.3958
1.3845
1.3648
1.3548
1.3310
1.3162
1.3117
1.2674
1.2551
1.2119
1.2045
1.1878
1.1732
1.1666
1.1498
1.1443
1.1326
1.1246
1.1215
1.1108
1.1019
1.0494
1.0264
1.0001
0.9770
0.8707
0.8493
0.8258
O
OH
N
H
OBz
4
21
9
8
7
6
5
4
3
2
1
ppm
1.34
0.64
1.00
2.00
0.64
0.21
0.74
0.28
0.21
0.73
0.17
0.64
0.11
1.35
2.33
9.91
5.04
0.91
0.90
3.37
BRUKER
Current Data Parameters
NAME SW02220701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070222
Time 17.04
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 35.9
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

hemiaminal benzoate



BRUKER

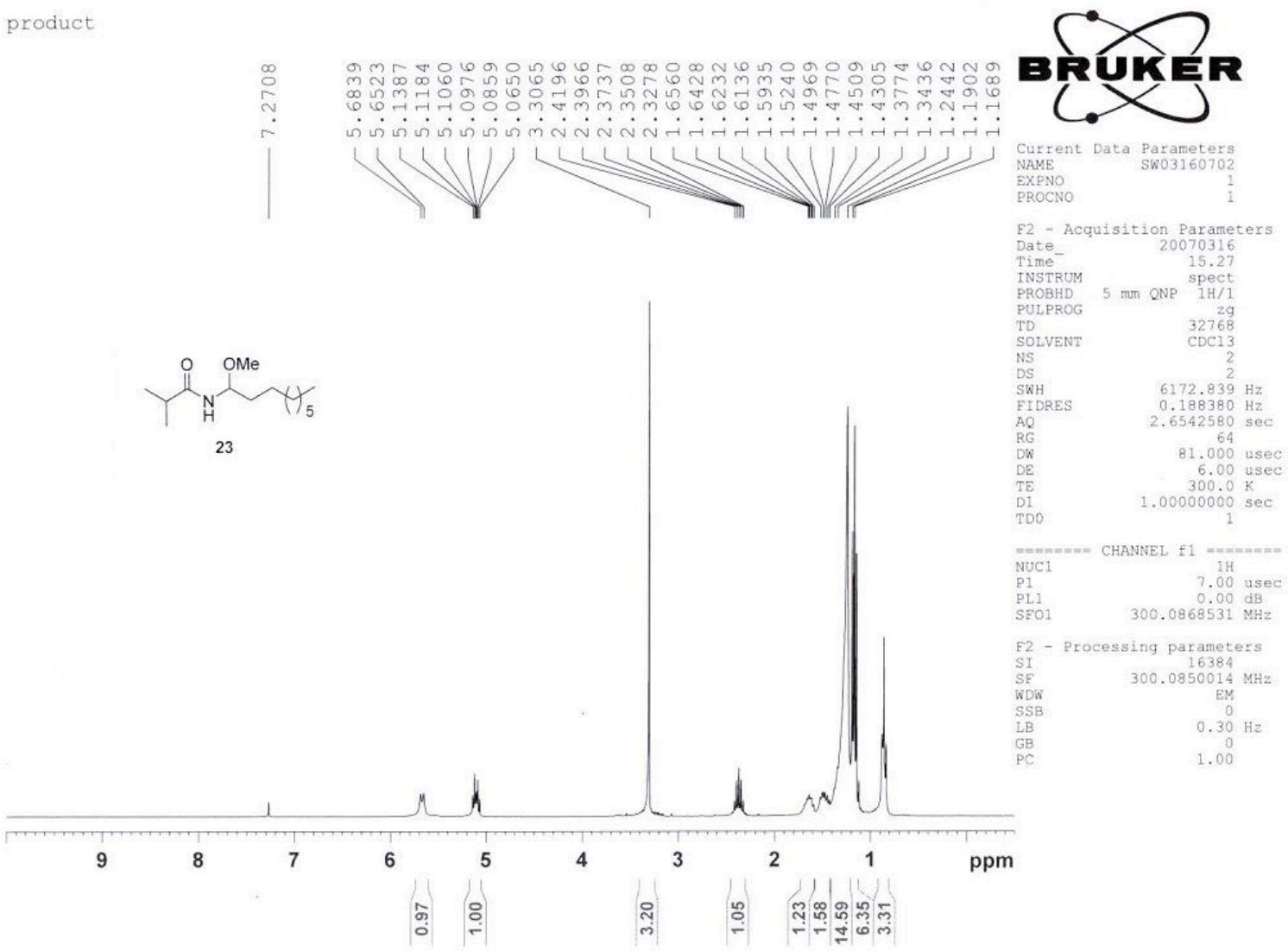
Current Data Parameters
NAME SW02220702
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070222
Time 17.09
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 37
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 10321.3
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

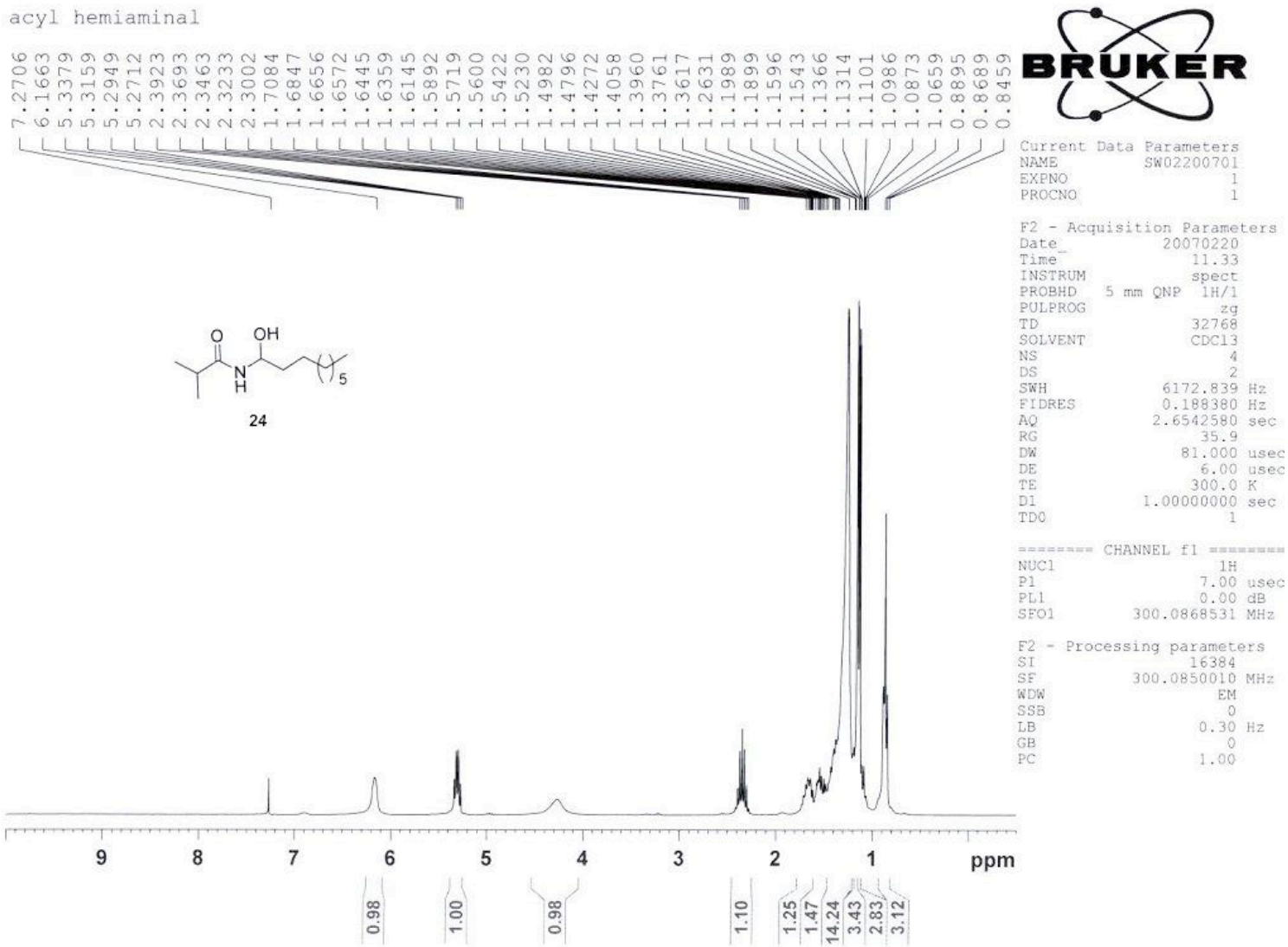
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

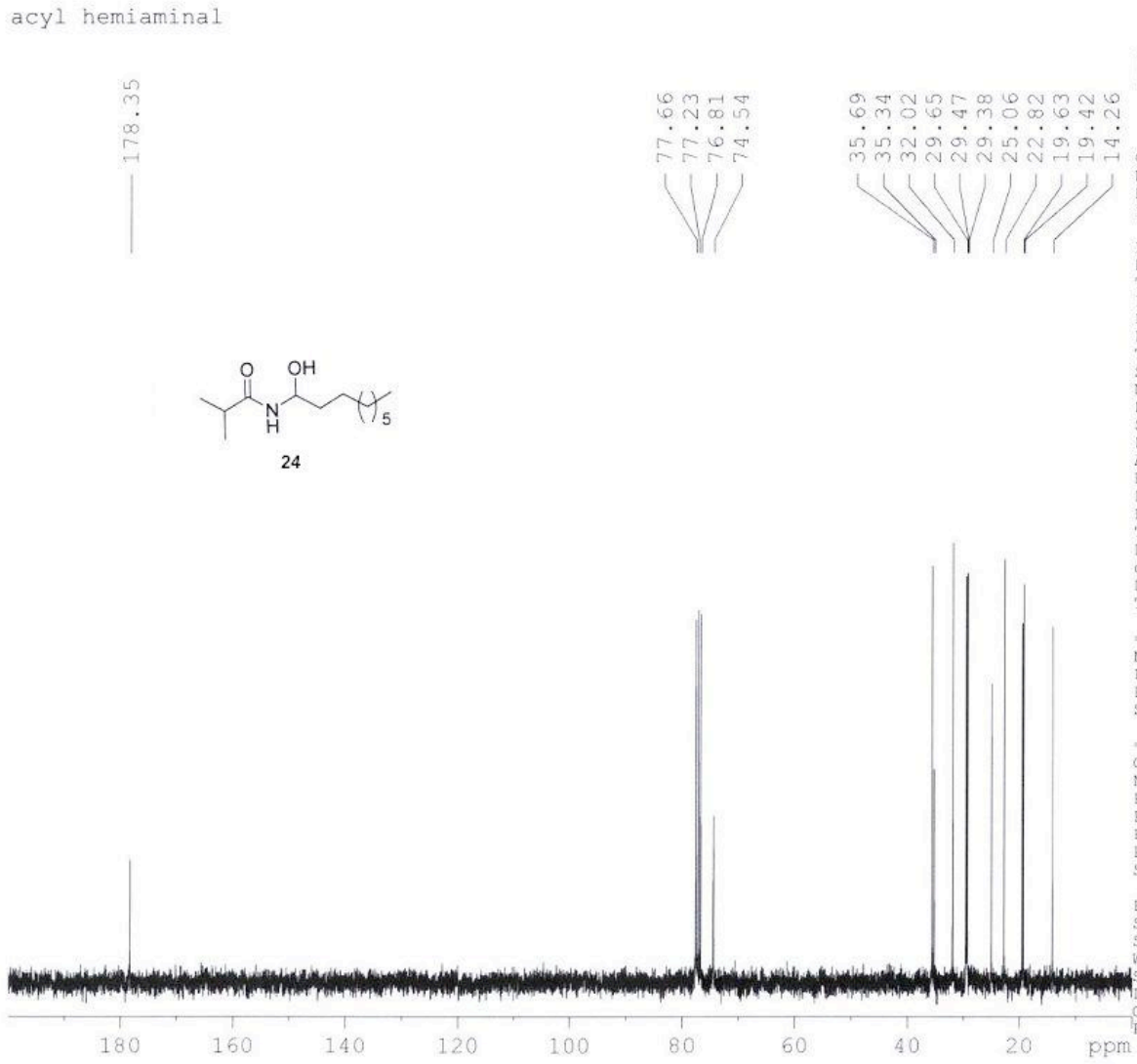
F2 - Processing parameters
SI 65536
SF 75.5305975 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


product
7.2708
5.6839
5.6523
5.1387
5.1184
5.1060
5.0976
5.0859
5.0650
3.3065
2.4196
2.3966
2.3737
2.3508
2.3278
1.6560
1.6428
1.6232
1.6136
1.5935
1.5240
1.4969
1.4770
1.4509
1.4305
1.3774
1.3436
1.2442
1.1902
1.1689
O
OMe
N
H
5
23
9
8
7
6
5
4
3
2
1
ppm
0.97
1.00
3.20
1.05
1.23
1.58
14.59
6.35
3.31
BRUKER
Current Data Parameters
NAME SW03160702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070316
Time 15.27
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 2
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 64
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 0.00 dB
SFO1 300.0868531 MHz
F2 - Processing parameters
SI 16384
SF 300.0850014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

BRUKER
Current Data Parameters
NAME SW03170701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070317
Time 12.39
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 49
DS 2
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 1448.2
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
d11 0.03000000 sec
DELTA 5.90000010 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 5.00 usec
PL1 0.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 24.44 dB
PL13 24.44 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677335 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
14.24
19.72
19.89
22.82
25.04
29.35
29.48
29.63
32.00
35.82
36.09
55.89
76.81
77.23
77.66
81.10
177.45
OMe
N
H
O
5
23


acyl hemiaminal
7.2706
6.1663
5.3379
5.3159
5.2949
5.2712
2.3923
2.3693
2.3463
2.3233
2.3002
1.7084
1.6847
1.6656
1.6572
1.6445
1.6359
1.6145
1.5892
1.5719
1.5600
1.5422
1.5230
1.4982
1.4796
1.4272
1.4058
1.3960
1.3761
1.3617
1.2631
1.1989
1.1899
1.1596
1.1543
1.1366
1.1314
1.1101
1.0986
1.0873
1.0659
0.8895
0.8689
0.8459
BRUKER
Current Data Parameters
NAME SW02200701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070220
Time 11.33
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 0.00 dB
SFO1 300.0868531 MHz
F2 - Processing parameters
SI 16384
SF 300.0850010 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OH
N
H
5
24
9
8
7
6
5
4
3
2
1
ppm
0.98
1.00
0.98
1.10
1.25
1.47
14.24
3.43
2.83
3.12

acyl hemiaminal



BRUKER

Current Data Parameters
NAME SW02200702
EXPNO 1
PROCNO 1

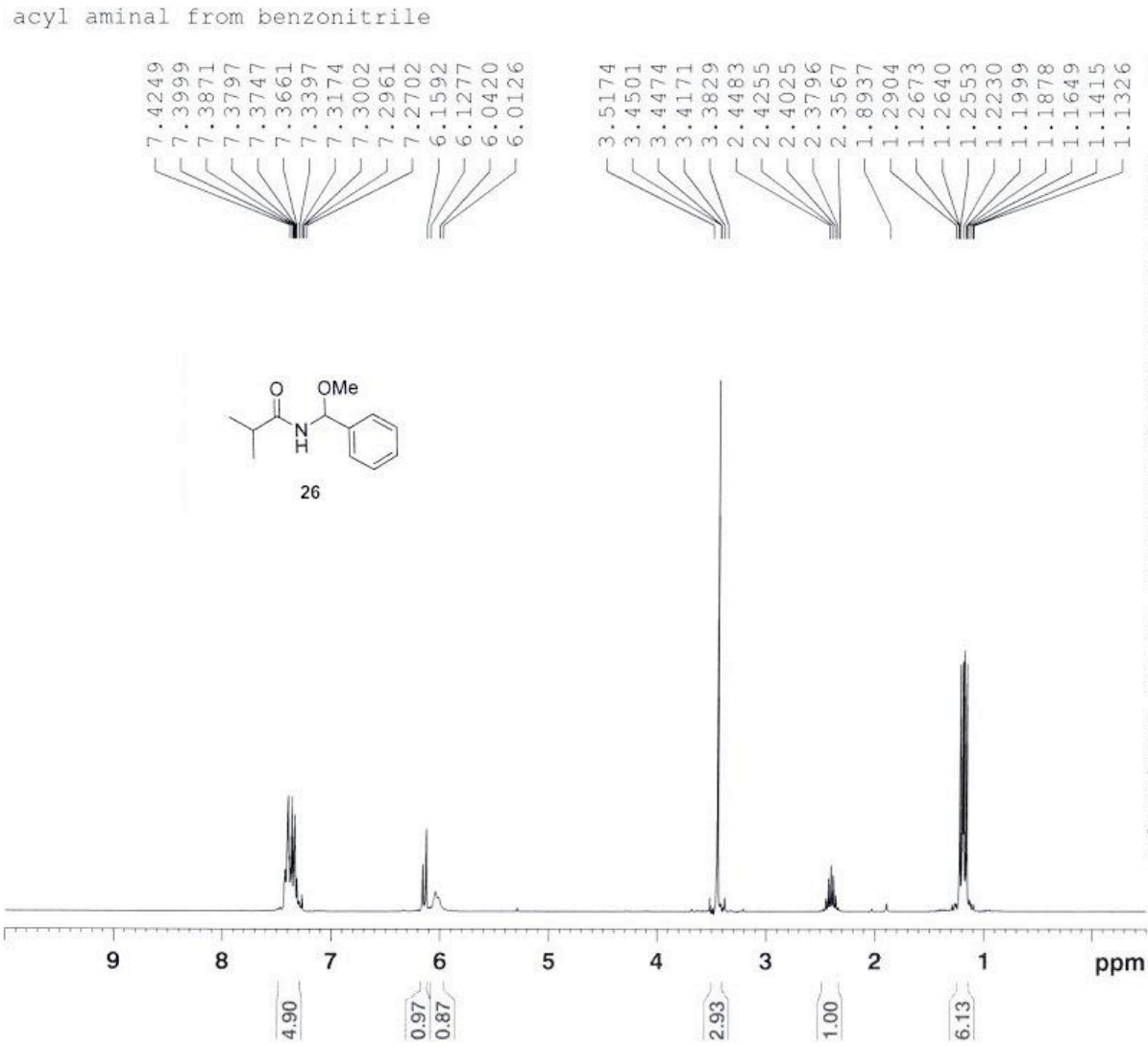
F2 - Acquisition Parameters
Date_ 20070220
Time 11.39
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 28
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz

F2 - Processing parameters
SI 32768
SF 75.4564178 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

acyl aminal from benzonitrile



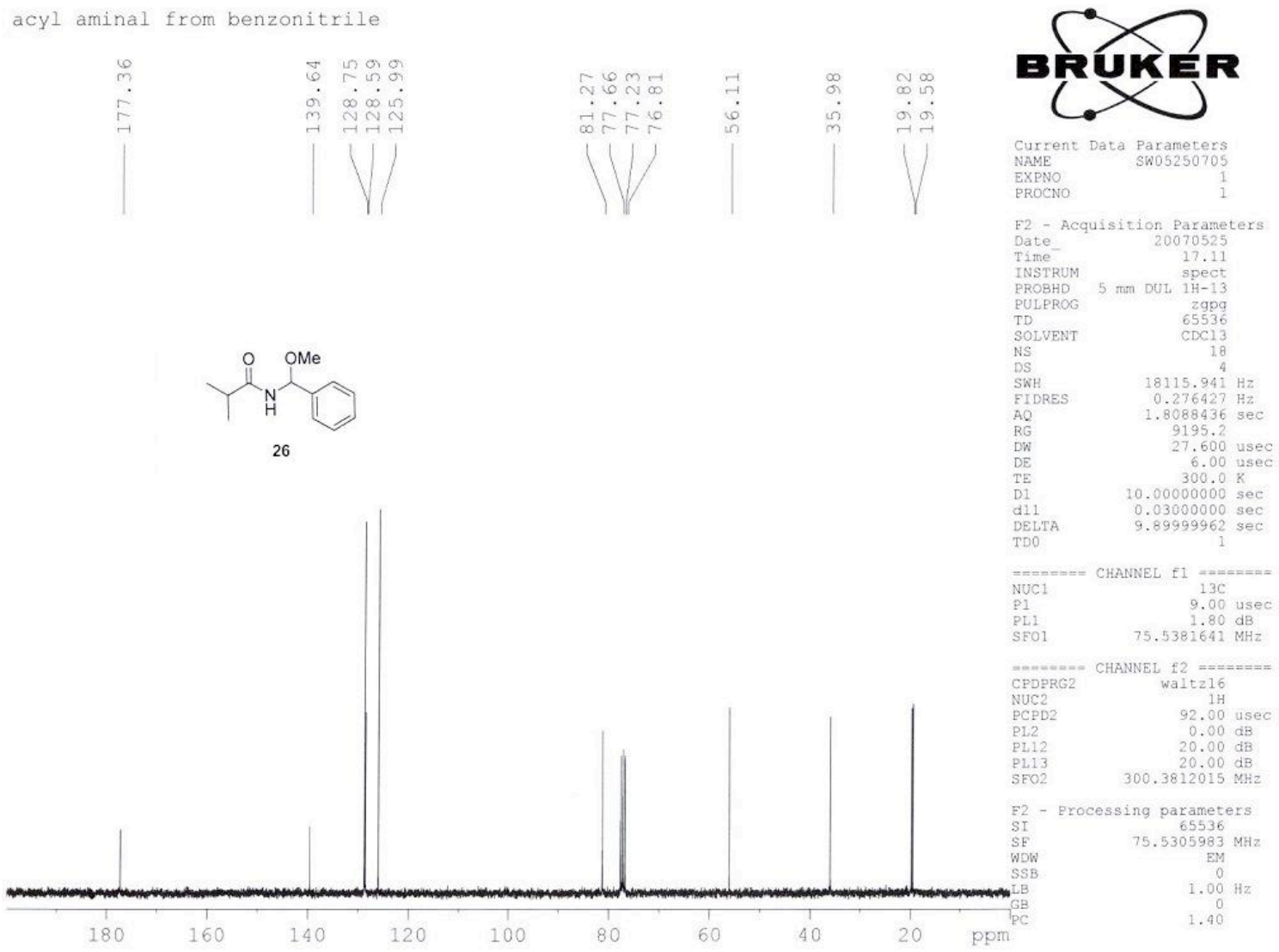
BRUKER

Current Data Parameters
NAME SW05250704
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070525
Time 17.05
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 57
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz

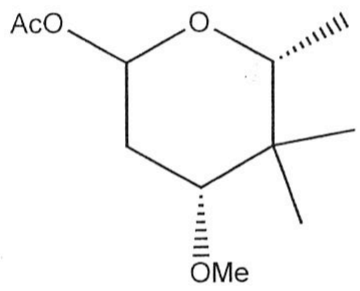
F2 - Processing parameters
SI 32768
SF 300.3799996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


acyl aminal from benzonitrile
177.36
139.64
128.75
128.59
125.99
81.27
77.66
77.23
76.81
56.11
35.98
19.82
19.58
O
OMe
N
H
26
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SW05250705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070525
Time 17.11
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 18
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 9195.2
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305983 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5.961
5.938
5.928
5.914
5.904
5.880
5.871
5.857
5.847
5.824
5.151
5.146
5.140
5.135
5.094
5.089
5.083
5.078
5.067
5.063
5.060
5.057
5.053
5.034
5.030
5.027
5.023
5.019
3.453
3.435
3.431
3.413
3.383
3.367
2.220
2.216
2.212
2.197
2.193
2.189
2.178
2.174
2.170
2.166
1.160
1.084
MeO
O

20.20
20.86
26.46
35.60
52.28
59.86
85.83
116.47
136.00
212.98

O
MeO


AcO
O
OMe

6.160
5.620
5.588
3.687
3.666
3.645
3.624
3.312
3.253
3.233
3.214
3.174
3.160
3.135
3.121
2.916
2.903
2.877
2.864
2.136
2.070
2.056
2.017
2.002
1.972
1.957
1.704
1.694
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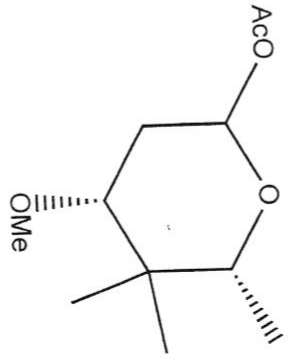
1.620
1.610
1.553
1.516
1.478
1.439

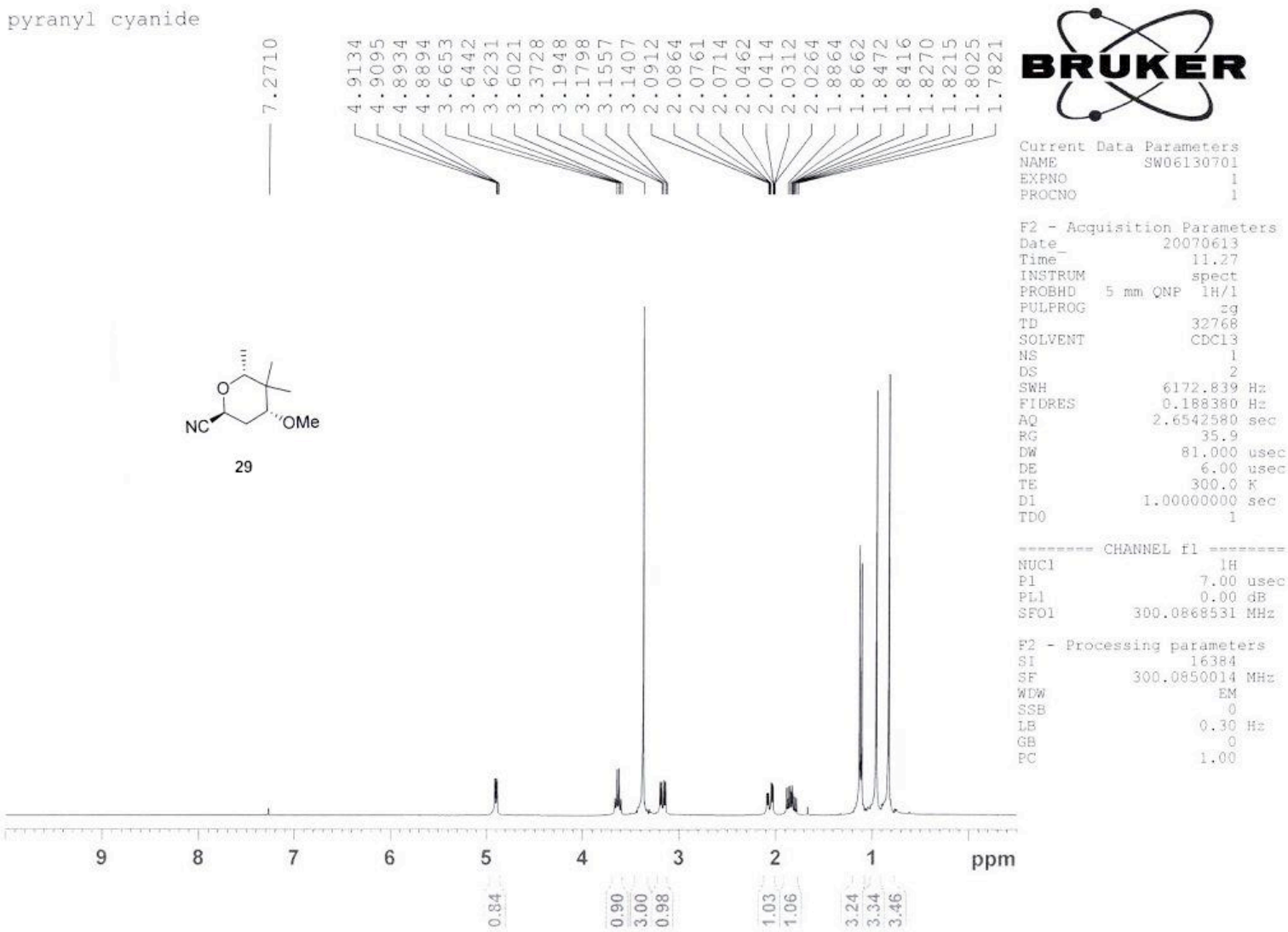


169.56
169.16

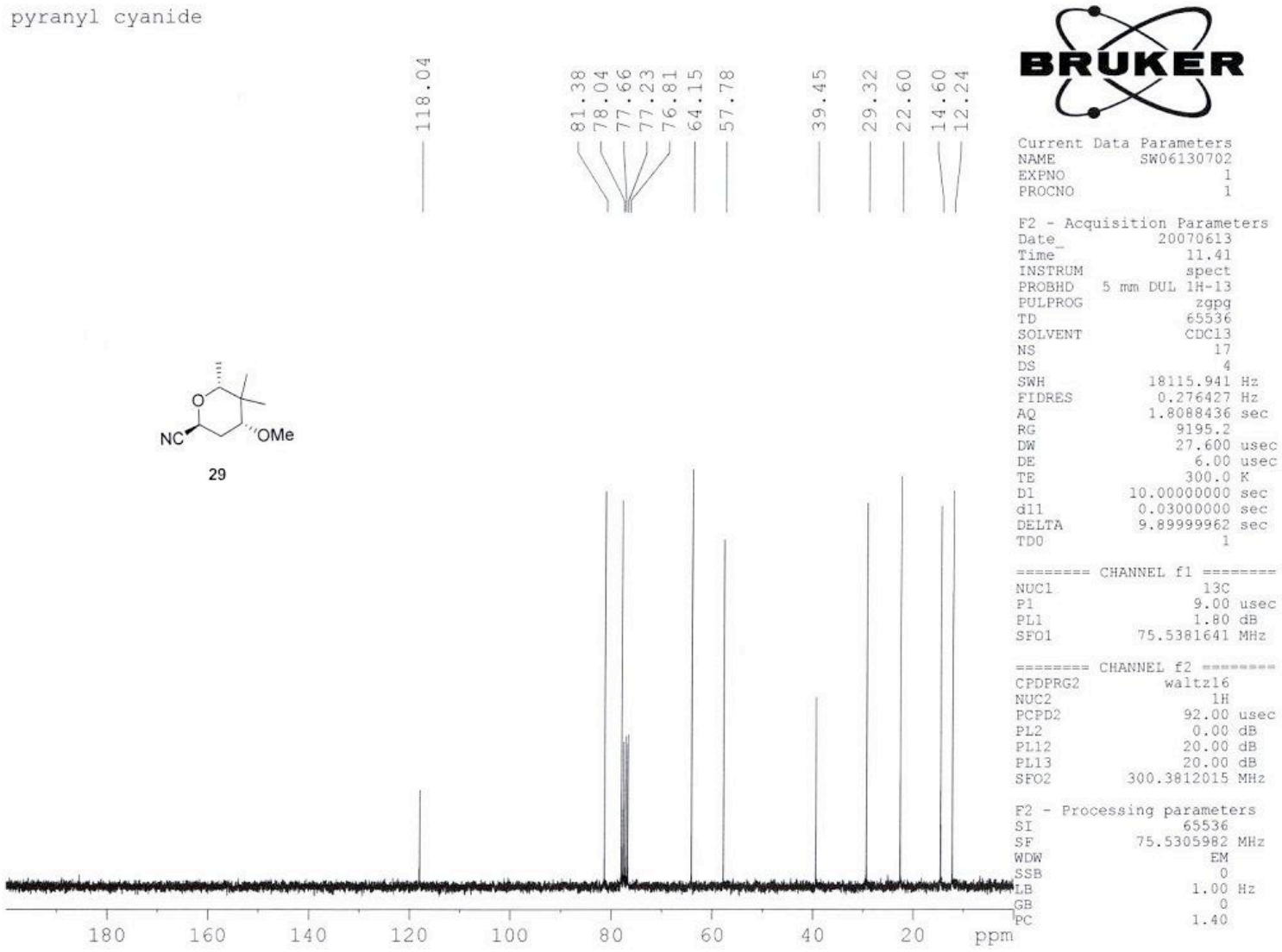
92.94
92.72
92.34
84.18
83.21
80.04
77.43
77.31
77.00
76.58
74.33
73.96

57.36
57.24
57.22

38.70
38.45
38.21
31.27
29.86
29.44
22.48
22.38
22.17
21.12
21.04
19.23
14.77
14.27
14.21
12.30
11.55


pyranyl cyanide
7.2710
4.9134
4.9095
4.8934
4.8894
3.6653
3.6442
3.6231
3.6021
3.3728
3.1948
3.1798
3.1557
3.1407
2.0912
2.0864
2.0761
2.0714
2.0462
2.0414
2.0312
2.0264
1.8864
1.8662
1.8472
1.8416
1.8270
1.8215
1.8025
1.7821
BRUKER
Current Data Parameters
NAME SW06130701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20070613
Time 11.27
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 1
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 0.00 dB
SFO1 300.0868531 MHz
F2 - Processing parameters
SI 16384
SF 300.0850014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NC
OMe
O
29
9
8
7
6
5
4
3
2
1
ppm
0.84
0.90
3.00
0.98
1.03
1.06
3.24
3.34
3.46

pyranyl cyanide



BRUKER

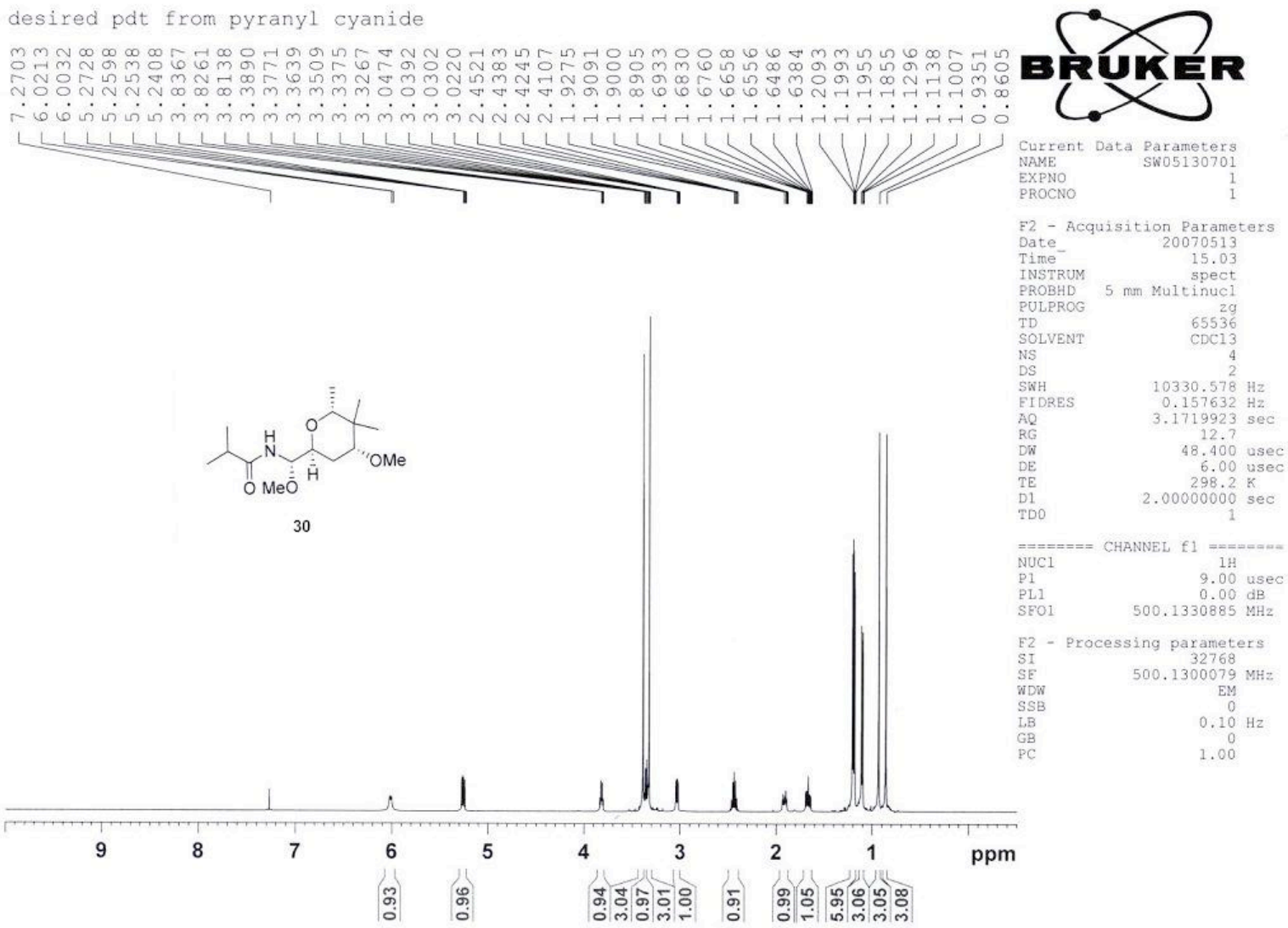
Current Data Parameters
NAME SW06130702
EXPNO 1
PROCNO 1

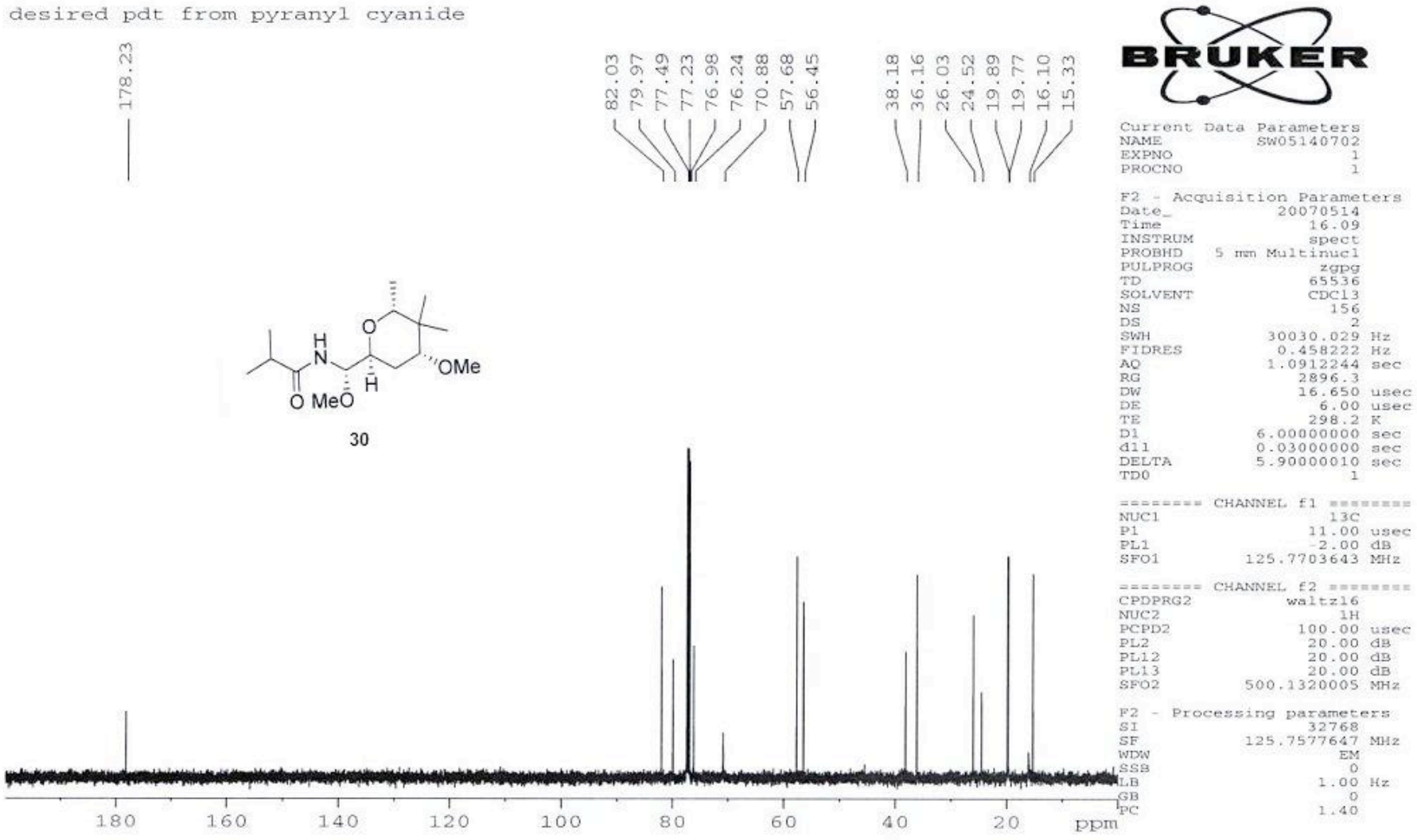
F2 - Acquisition Parameters
Date_ 20070613
Time 11.41
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 17
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 9195.2
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1

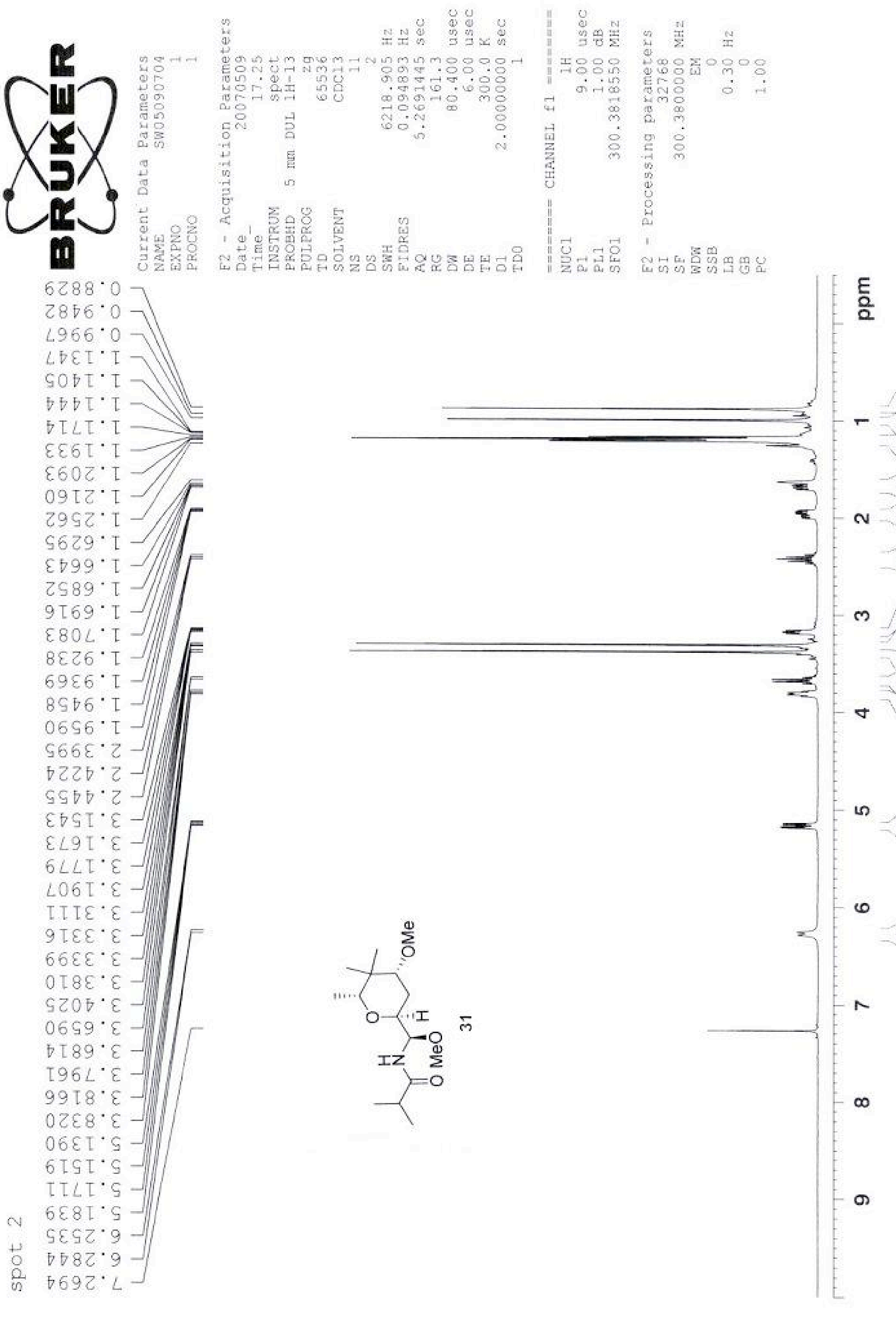
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz

F2 - Processing parameters
SI 65536
SF 75.5305982 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

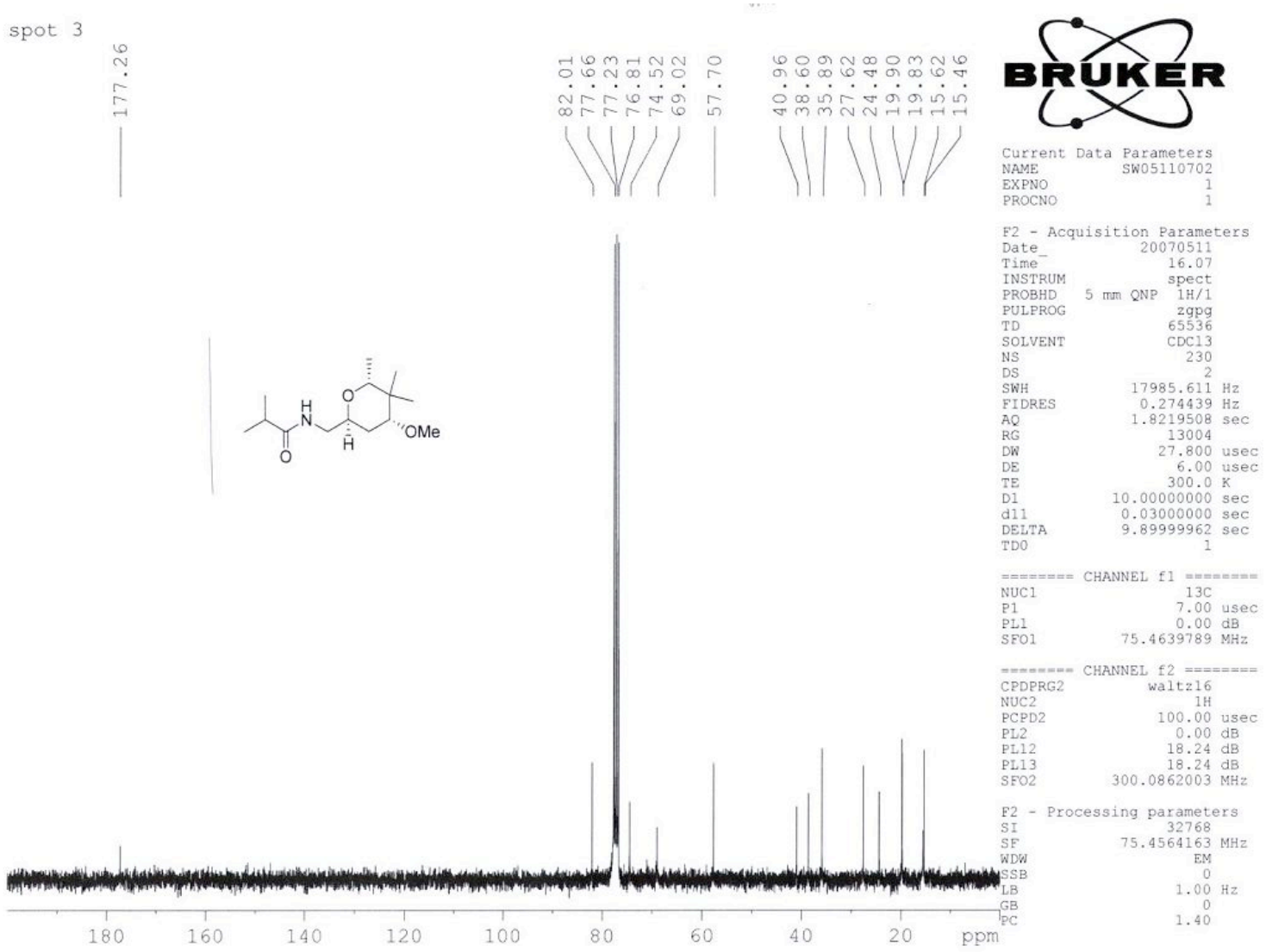

desired pdt from pyranyl cyanide
7.2703
6.0213
6.0032
5.2728
5.2598
5.2538
5.2408
3.8367
3.8261
3.8138
3.3890
3.3771
3.3639
3.3509
3.3375
3.3267
3.0474
3.0392
3.0302
3.0220
2.4521
2.4383
2.4245
2.4107
1.9275
1.9091
1.9000
1.8905
1.6933
1.6830
1.6760
1.6658
1.6556
1.6486
1.6384
1.2093
1.1993
1.1955
1.1855
1.1296
1.1138
1.1007
0.9351
0.8605
BRUKER
Current Data Parameters
NAME SW05130701
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070513
Time_ 15.03
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 12.7
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300079 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
OMe
O MeO
30
9
8
7
6
5
4
3
2
1
ppm
0.93
0.96
0.94
3.04
0.97
3.01
1.00
0.91
0.99
1.05
5.95
3.06
3.05
3.08


desired pdt from pyranyl cyanide
178.23
82.03
79.97
77.49
77.23
76.98
76.24
70.88
57.68
56.45
38.18
36.16
26.03
24.52
19.89
19.77
16.10
15.33
BRUKER
Current Data Parameters
NAME SW05140702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070514
Time 16.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 156
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 2896.3
DW 16.650 usec
DE 6.00 usec
TE 298.2 K
D1 6.00000000 sec
d11 0.03000000 sec
DELTA 5.90000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -2.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 20.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577647 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
H
N
OMe
O MeO
H
30
180
160
140
120
100
80
60
40
20
ppm


spot 2
7.2694
6.2844
6.2535
5.1839
5.1711
5.1519
5.1390
3.8320
3.8166
3.7961
3.6814
3.6590
3.4025
3.3810
3.3399
3.3316
3.3111
3.1907
3.1779
3.1673
3.1543
2.4455
2.4224
2.3995
1.9590
1.9458
1.9369
1.9238
1.7083
1.6916
1.6852
1.6643
1.6295
1.2562
1.2160
1.2093
1.1933
1.1714
1.1444
1.1405
1.1347
0.9967
0.9482
0.8829
BRUKER
Current Data Parameters
NAME SW0509070
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070509
Time 17.25
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 161.3
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3800000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
O
H
N
H
O MeO
31
0.89
0.91
0.94
0.96
3.09
3.03
1.03
1.00
1.00
1.64
9.19
3.02
3.18
ppm
9
8
7
6
5
4
3
2
1

BRUKER
Current Data Parameters
NAME SM05090705
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070509
Time_ 17.33
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 137
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.80884436 sec
RG 13004
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 1.80 dB
SFO1 75.5381641 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.3812015 MHz
F2 - Processing parameters
SI 65536
SF 75.5305942 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
spot 2
177.54
82.39
81.68
77.66
77.44
77.23
76.81
70.26
57.75
56.66
38.01
36.13
26.32
25.37
19.86
19.73
17.55
15.60
OMe
H
N
O
MeO
O
31

BRUKER
spot 3
Current Data Parameters
NAME SW05090706
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070509
Time_ 21.27
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 6218.905 Hz
FIDRES 0.094893 Hz
AQ 5.2691445 sec
RG 161.3
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz
F2 - Processing parameters
SI 32768
SF 300.3799993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.2699
5.7665
4.0323
4.0140
4.0050
3.9866
3.4782
3.4564
3.4349
3.4280
3.4205
3.4079
3.3913
3.3606
3.3556
3.3155
3.2987
3.2521
3.0531
3.0316
3.0088
2.4272
2.4042
2.3812
2.3583
2.3353
1.7459
1.7265
1.7226
1.7058
1.6866
1.2553
1.2249
1.1799
1.1575
1.1365
1.1152
1.0923
1.0731
1.0253
0.9566
0.9243
0.8806
0.8165
OMe
H
N
O
O
ppm
9
8
7
6
5
4
3
2
1
0.80
0.90
3.03
2.81
0.89
1.00
2.07
8.13
2.91
2.66


spot 3
177.26
82.01
77.66
77.23
76.81
74.52
69.02
57.70
40.96
38.60
35.89
27.62
24.48
19.90
19.83
15.62
15.46
OMe
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME SW05110702
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070511
Time 16.07
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 230
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 13004
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 10.00000000 sec
d11 0.03000000 sec
DELTA 9.89999962 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 75.4639789 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.24 dB
PL13 18.24 dB
SFO2 300.0862003 MHz
F2 - Processing parameters
SI 32768
SF 75.4564163 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40