Supporting Information

Investigation of the Stereochemistry of Intermolecular Conjugate Additions of Nucleophiles to Acyclic Nitrosoalkenes

Jason A. Witek and Steven M. Weinreb*

Department of Chemistry, The Pennsylvania State University

University Park, Pennsylvania 16802, United States

E-Mail: smw@chem.psu.edu

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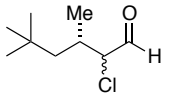
General. All non-aqueous reactions were carried out under a positive atmosphere of argon in flame-dried glassware unless otherwise noted. Anhydrous THF and CH_2Cl_2 were obtained from a solvent dispensing system. All other solvents and reagents were used as obtained from commercial sources without further purification. ^1H and ^{13}C NMR spectra were recorded on 300, 360 or 400 MHz spectrometers. Flash column chromatography was performed using silica gel 60 (230-400 mesh).

General Procedure for the Synthesis of α-Chloro-*O*-silylaldoximes. To a stirred solution of the aldehyde (1 mmol) in $CHCl_3$ at 0 °C was added a catalytic amount of proline (0.05 mmol) and NCS (1.2 mmol). The resulting solution was warmed to rt and stirred for 12 h. The reaction mixture was diluted with pentane, filtered and washed with water. The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel eluting with a mixture of ethyl acetate and hexanes to provide the α-chloroaldehyde.

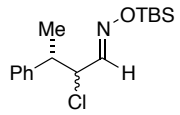
To a stirred solution of the α-chloroaldehyde (1 mmol) in CH_2Cl_2 (2 mL) and a spatula of 4 Å molecular sieves was added H_2NOTBS (147 mg, 1 mmol) and PPTS (13 mg, 0.05 mmol). The reaction mixture was stirred at rt for 12 h and then filtered through a pad of Celite. The solution was concentrated *in vacuo* and the residue was purified by flash column chromatography on silica gel eluting with a mixture of ethyl acetate and hexanes to provide the α-chloro-*O*-silylaldoxime.

2-Chloro-3,5,5-trimethylhexanal. The product was obtained as a clear oil (5.36 g) in 86% yield as an inseparable mixture of diastereomers in ~1:1 ratio: Isomer A: ^1H NMR (300 MHz, $CDCl_3$) δ 9.52 (d, J = 2.4 Hz, 1H), 4.15 (dd, *J* = 4.4, 2.5 Hz, 1H), 2.35-2.26 (m, 1H), 1.54-1.51 (m, 2H), 1.18 (d, *J* =

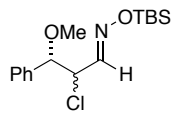


11.9 Hz, 3H), 0.94 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.0, 71.8, 47.6, 33.3, 31.4, 30.2, 19.9; Isomer B: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.52 (d, *J* = 2.9 Hz, 1H), 4.06 (dd, *J* = 4.9, 2.9 Hz, 1H), 2.26-2.23 (m, 1H), 1.50-1.46 (m, 2H), 1.03 (d, *J* = 12.7 Hz, 3H), 0.94 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.6, 71.3, 46.5, 32.6, 31.3, 30.1, 18.3

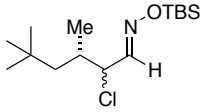
2-Chloro-3-phenylbutanal *O*-TBS-oxime (3). The product was obtained as a clear oil (485 mg, 64% yield) as an inseparable mixture of isomers: Data for major isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.8 Hz, 1H), 7.38-7.20 (m, 5H), 4.64 (dd, *J* = 16.2 , 8.1 Hz, 1H), 3.22 (m, 1H), 1.51, (d, *J* = 7.0 Hz, 3H), 0.95 (s, 9H), 0.84 (s, 6H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 153.1, 141.6, 128.7, 128.0, 127.3, 63.0, 55.8, 45.6, 44.5, 26.0, 18.2; HRMS-ES+ ($C_{16}H_{27}NOSiCl$) calcd 312.1550 (MH^+), found 312.1550.



2-Chloro-3-methoxy-3-phenylpropanal *O*-TBS-oxime (12). The product was obtained as a clear oil (667 mg, 19% yield) as an inseparable ~1: 1 mixture of isomers: Isomer A: ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.6 Hz, 1H), 7.41-7.34 (m, 5H), 4.68 (dd, *J* = 8.6, 6.6 Hz, 1H), 4.45 (d, *J* = 6.6 Hz, 1H), 3.38 (d, *J* = 6.5 Hz, 3H), 0.84 (s, 9H), 0.13 (s, 6H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 151.4, 137.1, 128.5, 127.6, 127.4, 85.1, 60.8, 57.4, 54.0, 25.7, 18.2. Isomer B: ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.6 Hz, 1H), 7.41-7.34 (m, 5H), 4.64 (dd, *J* = 8.6, 5.2 Hz, 1H), 4.51 (d, *J* = 5.2 Hz, 1H), 3.35 (d, *J* = 3.9 Hz, 3H), 0.84 (s, 9H), 0.13 (s, 6H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 151.4, 136.9, 128.6, 127.6, 127.4, 85.7, 60.0, 57.6, 52.4, 26.0, 18.2; HRMS-ES+ ($C_{16}H_{27}NO_2ClSi$) calcd 328.1500 (MH^+), found 328.1509.



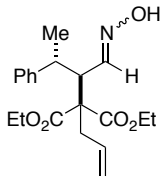
2-Chloro-3,5,5-trimethylhexanal *O*-TBS-oxime (16). The product was obtained as a clear oil (2.72 g, 86% yield) as an



inseparable mixture of isomers: Data for major isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (d, *J* = 6.1 Hz, 1H), 4.40 (m, 1H), 2.03 (m, 1H), 1.56 (m, 2H), 1.52 (d, *J* = 2.8 Hz, 2H), 0.95 (s, 9H), 0.97 (s, 9H) , 0.20 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.8, 64.8, 59.1, 47.7, 47.3, 31.3, 30.3, 26.5, 19.5, 18.6; HRMS-ES+ ($C_{15}H_{33}NOClSi$) calcd 306.2020 (MH^+), found 306.2020.

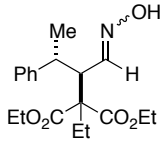
General Procedure for the Conjugate Additions to Nitrosoalkenes. To a stirred solution of the malonate or sulfonamide (2 mmol) in THF (2.2 mL) was added KHMDS (4 mL, 0.5 M in PhMe, 2 mmol) at -78 °C. The resulting solution was then stirred for 45 min at that temperature. The *O*-TBS oxime (1 mmol) dissolved in THF (600 uL) was added slowly over 1 min, followed by dropwise addition of TBAF (2 mL, 1.0 M in THF, 2 mmol) over 3 min. The resulting solution was immediately transferred to an ice bath and stirred for an additional 2 h. The reaction mixture was diluted with concentrated aqueous NH_4Cl and EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to give a residue which was purified by flash column chromatography on silica gel eluting with a mixture of ethyl acetate and hexanes.

Diethyl 2-Allyl-2-(1-(hydroxyimino)-3-phenylbutan-2-yl)malonate (5). The product was obtained as a clear oil (43 mg, 74% yield) as a ~9:1 mixture of *E*/*Z* oxime isomers: *(E)*-Oxime isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (br s, 1H), 7.66 (d, *J* = 9.6 Hz, 1H), 7.34-20 (m, 5H), 5.78-5.64 (m, 1H), 5.05 (br s, 1H), 5.00 (br d, *J* = 5.6 Hz, 1H), 4.28-4.01 (m, 4H), 3.39-3.32 (m, 1H), 3.25 (dd, J = 9.6, 4.6 Hz, 1H), 2.57 (d, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 3.6 Hz, 3H), 1.26 (t, *J* = 3.6 Hz, 3H), 1.18 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ

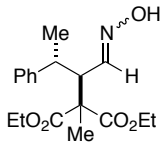


170.4, 170.3, 151.0, 146.8, 132.8, 128.7, 128.0, 126.9, 119.7, 62.0, 61.8, 60.9, 49.7, 40.0, 39.4, 19.5, 14.5, 14.4; HRMS-ES+ ($C_{20}H_{28}NO_5$) calcd 362.1967 (MH+), found 362.1971.

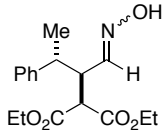
Diethyl 2-Ethyl-2-(1-(hydroxyimino)-3-phenylbutan-2-yl)malonate (6). The product was obtained as a clear oil (93 mg, 72% yield) as a ~ 10:1 mixture of *E*/*Z* oxime isomers: *(E)*-oxime isomer: ^{1}H NMR (360 MHz, $CDCl_3$) δ 8.46 (br s, 1H), 7.65 (d, *J* = 9.7 Hz, 1H), 7.33-7.19 (m, 5H), 4.31-4.04 (m, 4H), 3.35-3.32 (m, 1H), 3.24 (dd, *J* = 9.7, 4.3 Hz, 1H), 1.91-1.84 (m, 2H), 1.34 (t, *J* = 1.8 Hz, 3H), 1.25 (t, *J* = 1.8 Hz, 3H), 1.17 (d, *J* = 7.7 Hz, 3H), 0.84 (t, *J* = 3.7 Hz, 3H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 170.5, 170.4, 150.5, 146.7, 128.3, 127.6, 126.4, 61.4, 61.3, 60.7, 48.9, 39.1, 28.4, 18.9, 14.1, 14.0, 8.8; HRMS-ES+ ($C_{19}H_{28}NO_5$) calcd 350.1967 (MH+), found 350.1963.



Diethyl 2-(1-(Hydroxyimino)-3-phenylbutan-2-yl)-2-methylmalonate (7). The product was obtained as a clear oil (37 mg, 63% yield): ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.65 (d, *J* = 9.1 Hz, 1H), 7.49 (br s, 1H), 7.33-7.19 (m, 5H), 4.13-4.00 (m, 4H), 3.30 (m, 2H), 1.43 (s, 3H), 1.36-1.15 (m, 9H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 170.8, 151.4, 146.2, 128.3, 127.8, 126.5, 61.5, 56.9, 50.3, 39.4, 20.9, 20.0, 13.9; HRMS-ES+ ($C_{18}H_{26}NO_5$) calcd 336.1811 (MH+), found 336.1810.

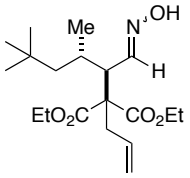


Diethyl 2-(1-(Hydroxyimino)-3-phenylbutan-2-yl)malonate (8). The product was obtained as a clear oil (35 mg, 68% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (br s, 1H), 7.67 (d, *J* = 7.5 Hz, 1H), 7.38-7.19 (m, 5H) 4.19 (m, 4H), 3.41 (d, *J* = 5.7 Hz, 1H), 3.22-3.08 (m, 2H), 1.30-1.23 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.8, 168.3, 151.4, 144.0, 129.2, 127.9, 127.4, 62.2, 62.0,

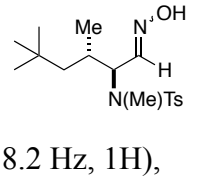


53.7, 47.1, 40.3, 19.6, 14.5; HRMS-ES+ ($C_{17}H_{23}NO_5Na$) calcd 344.1474 (M+Na), found 344.1478.

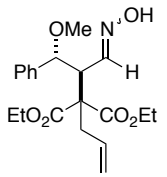
Diethyl 2-Allyl-2-(1-(hydroxyimino)-3,5,5-trimethylhexan-2-yl)malonate (17). The product was obtained as a clear oil (89 mg, 76% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.51 (br s, 1H), 7.58 (d, *J* = 9.4 Hz, 1H), 5.78-5.69 (m, 1H), 5.10 (d, *J* = 8.3 Hz, 1H), 5.05 (s, 1H), 4.25-4.11 (m, 4H), 2.88 (dd, *J* = 9.3, 2.0 Hz, 1H), 2.80 (q, *J* = 2.1 Hz, 1H), 2.60 (q, *J* = 2.1 Hz, 1H), 2.1 (m, 1H), 1.36 (dd, *J* = 11.8, 2.3 Hz, 1H), 1.33 (t, *J* = 3.6 Hz, 3H), 1.24 (t, *J* = 3.6 Hz, 3H), 1.10 (dd, *J* = 14.1, 7.7 Hz, 1H), 0.90 (br s, 9H), 0.83, (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.6, 150.8, 132.7, 119.6, 61.9, 61.8, 60.5, 51.9, 49.7, 39.5, 31.7, 30.9, 29.3, 17.9, 14.6, 14.4; HRMS-ES+ ($C_{19}H_{33}NO_5$) calcd 356.2437 (MH+), found 356.2428.



***N*-(1-(Hydroxyimino)-3,5,5-trimethylhexan-2-yl)-*N*,4-dimethylbenzenesulfonamide (20)**. The product was obtained as a clear oil (41 mg, 74% yield): ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.2 Hz, 1H), 7.31 (d, *J* = 9.1 Hz, 2H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.91 (s, 1H), 4.23 (dd, *J* = 10.4, 7.8 Hz, 1H), 2.75 (s, 3H), 2.45 (s, 3H), 1.86 (m, 1H), 1.07 (dd, *J* = 11.5, 7.8 Hz, 2H), 0.98 (d, *J* = 6.6 Hz, 3H) 0.95 (s, 9H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 147.8, 143.4, 135.9, 129.5, 127.6, 76.7, 61.9, 46.3, 30.8, 30.0, 29.8, 21.5, 19.7; HRMS-ES+ ($C_{17}H_{29}N_2O_3S$) calcd 341.1899 (MH+), found 341.1894.



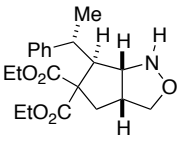
Synthesis of Diethyl 2-Allyl-2-(3-(Hydroxyimino)-1-methoxy-1-phenylpropan-2-yl)malonate (13). To a stirred solution of diethyl allyl malonate (1.2 mmol) in THF (2.2 mL) was added KHMDS (2.4 mL,



0.5 M in PhMe, 1.2 mmol) at -78 °C. The resulting solution was then stirred for 45 min at that temperature. TBAF was added (1.2 mL, 1.0 M in THF, 1.2 mmol) followed by dropwise addition of a solution of the *O*-TBS oxime **12** (1 mmol) dissolved in THF (600 uL). The resulting solution was immediately transferred to a 0 °C bath and stirred for an additional 1 h. The reaction mixture was diluted with concentrated aqueous NH_4Cl and EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to give a gradient of 25-50% EtOAc/hexanes. The product was obtained as a clear oil (39 mg) in 68% yield: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (br s, 1H), 7.62 (d, *J* = 9.6 Hz, 1H), 7.37-7.24 (m, 5H), 5.68-5.54 (m, 1H), 5.11-5.05 (m, 2H), 4.81 (s, 1H), 4.38-4.16 (m, 4 H), 3.12 (s, 3H), 3.06 (d, *J* = 9.6 Hz, 1H), 2.72-2.70 (m, 2H) 1.36 (t, *J* = 3.6 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 170.6, 148.6, 140.4, 131.5, 128.6, 127.9, 127.1, 120.2, 82.3, 61.9, 59.2, 57.2, 50.3, 38.9, 14.6, 14.5; HRMS-ES+ ($C_{20}H_{28}NO_6$) calcd 378.1917 (MH+), found 1378.1920.

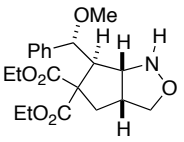
General Procedure for the Synthesis of Isoxazolidines. A solution of α-alkyl aldoxime (0.1 mmol) in toluene (4 mL) was heated and stirred in a sealed tube at 190 °C for 5 h. The solution was concentrated *in vacuo* and the residue was purified by flash column chromatography on silica gel eluting with a mixture of ethyl acetate and hexanes.

Diethyl 6-(1-Phenylethyl)tetrahydro-1*H*-cyclopenta[*c*]isoxazole-5,5(3*H*)-dicarboxylate (10). The product was obtained as a clear oil (49 mg, 57% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.22 (m, 4H), 7.16-7.11 (m, 1H), 5.20 (br s, 1H), 4.26 (q, *J* = 2.4 Hz, 2H), 4.04 (t, *J* = 4.4 Hz, 1H), 3.91 (br d, *J* = 8.5 Hz, 1H), 3.67-3.56 (m, 2H), 3.54-3.45 (m, 1H), 3.40-



3.28 (m, 1H), 3.06-2.95 (m, 1H), 2.67 (t, J = 4.5 Hz, 1H), 2.57 (dd, J = 12.7, 8.0 Hz, 1H), 2.07-1.98 (m, 1H), 1.34 (d, J = 5.2 Hz, 3H), 1.31 (t, J = 2.6 Hz, 3H), 1.00 (t, J = 3.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 170.8, 146.7, 128.5, 128.3, 126.4, 77.5, 69.6, 63.8, 61.5, 61.4, 58.2, 46.6, 41.8, 39.7, 22.5, 14.5, 14.0; HRMS-ES+ ($C_{20}H_{28}NO_5$) calcd 362.1967 (MH+), found 362.1970.

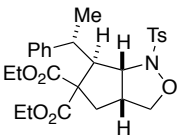
Diethyl 6-(Methoxy(phenyl)methyl)tetrahydro-1*H*-cyclopenta[*c*]isoxazole-5,5(3*H*)-dicarboxylate (14). The product was



obtained as a clear oil (26 mg, 66% yield): ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.37-7.27 (m, 5H), 4.86 (d, J = 1.8 Hz, 1H), 4.42-4.35 (m, 2H), 4.33-4.09 (m, 4H), 3.73 (d, J = 8.4 Hz, 1H), 3.49-3.35 (m, 2H), 3.20 (s, 3H), 2.76 (dd, J = 8.0, 1.85 Hz, 1H), 2.66 (dd, J = 13.0, 8.2 Hz, 1H), 1.63 (dd, J = 13.0, 9.3 Hz, 1 H), 1.36 (t, J = 3.6 Hz, 3H), 1.30 (t, J = 3.6 Hz, 3H); ^{13}C NMR (90 MHZ, $CDCl_3$) δ 171.6, 171.3, 140.4, 128.3, 127.3, 126.4, 79.9, 76.7, 63.9, 62.7, 61.3, 61.2, 59.1, 56.8, 47.7, 39.9, 14.1, 14.0; HRMS-ES+ ($C_{20}H_{28}NO_6$) calcd 378.1917 (MH+), found 378.1927.

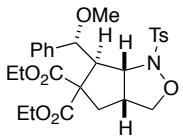
General Procedure for Synthesis of *N*-Tosyl Isoxazolidines. To a stirred solution of isoxazolidine (0.1 mmol) was added TsCl (19 mg, 0.1 mmol) and K_2CO_3 (28 mg, 0.2 mmol). The reaction mixture was heated at reflux for 60 h and then diluted with H_2O and CH_2Cl_2. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4 and concentrated in vacuo to give a residue, which was purified by flash column chromatography on silica gel eluting with a mixture of ethyl acetate and hexanes.

Diethyl 6-(1-Phenylethyl)-1-(4-methylbenzenesulfonyl)tetrahydro-1*H*-cyclopenta[*c*]isoxazole-



5,5(3*H*)-dicarboxylate (11). The product was obtained as a white solid (38 mg, 75% yield); X-ray quality crystals were prepared via slow evaporation from isopropanol/dichloromethane; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.1 Hz, 2H), 7.33-7.26 (m, 4H), 7.19-7.18 (m, 1H), 5.05 (t, *J* = 4.4 Hz, 1H), 4.51 (t, *J* = 3.6 Hz, 1H), 4.29 (q, *J* = 2.3 Hz, 2H), 3.84-3.78 (m, 1H), 3.72-3.63 (m, 2H), 3.41 (t, *J* = 3.9, 1H), 2.84 (t, *J* = 3.3 Hz, 1H), 2.68 (dd, *J* = 12.3, 8.7 Hz, 1H), 2.45 (s, 3H), 1.88 (t, *J* = 5.2 Hz, 1H), 1.48 (d, *J* = 6.7 Hz, 3H), 1.34 (t, *J* = 3.4 Hz, 3H), 1.11 (t, *J* = 3.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 170.6, 146.8, 145.3, 134.0, 130.0, 129.3, 128.6, 128.3, 126.3, 65.9, 64.1, 62.0, 61.8, 57.4, 45.9, 41.8, 38.5, 22.1, 19.1,14.5, 14.2; HRMS-ES+ ($C_{27}H_{34}NO_7S$) calcd 516.2056 (MH+), found 516.2053.

Diethyl 6-(Methoxy(phenyl)methyl)-1-(4-methylbenzenesulfonyl)tetrahydro-1*H*-cyclopenta[*c*]isoxazole-5,5(3*H*)-dicarboxylate (15).



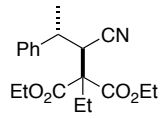
The product was obtained as a white solid (22 mg, 68% yield); X-ray quality crystals were prepared via slow evaporation from isopropanol/dichloromethane; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.1 Hz, 2H), 7.43 (t, *J* = 3.5 Hz, 2H), 7.34-7.25 (m, 3H), 7.16 (d, *J* = 8.1 Hz, 2H), 5.45 (t, *J* = 4.5 Hz, 1H), 4.90 (s, 1H), 4.47 (t, *J* = 3.7 Hz, 1H), 4.41-4.32 (m, 1H), 4.31-4.17 (m, 4H), 3.64 (t, *J* = 4.3 Hz, 1H), 3.54 (d, *J* = 8.2 Hz, 1H), 3.30 (s, 3H), 2.93 (d, *J* = 8.9 Hz, 1H), 2.78 (dd, *J* = 13.3, 8.7 Hz, 1H), 2.39 (s, 1H), 1.69-1.61 (m, 2H), 1.41 (t, *J* = 3.6 Hz, 3H), 1.33 (t, *J* = 3.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 171.3, 144.7, 140.5, 134.1, 129.5, 129.3, 128.5, 127.4, 127.0, 79.1, 77.5, 63.3, 62.1, 62.0, 58.7, 57.7, 46.8, 40.6, 30.1, 22.0, 14.5; HRMS-ES+ ($C_{27}H_{34}NO_8S$) calcd 532.2005 (MH+), found 532.1996.

Synthesis of Diethyl 6-(4,4-Dimethylpentan-2-yl)-1-(4-nitrobenzenesulfonyl)tetrahydro-1*H*-cyclopenta[*c*]isoxazole-5,5(3*H*)-dicarboxylate (19). A solution of diethyl 2-allyl-2-(1-(hydroxyimino)-3,5,5-trimethylhexan-2- yl)malonate (**17**, 0.04 mmol) in toluene (4 mL) was heated and stirred in a sealed tube at 190 °C for 5 h. The solution was concentrated *in vacuo*. The crude residue was dissolved in CH_2Cl_2 (430 uL) and to this solution was added NsCl (11 mg, 0.05 mmol) and TEA (7 uL, 0.05 mmol). The reaction mixture was stirred for 7 h at rt and then diluted with CH_2Cl_2. The combined organic layers were washed with water, dried over Na_2SO_4 and concentrated *in vacuo* to give a residue which was purified by flash column chromatography on silica gel eluting with 10-50% EtOAc/hexanes. The product was obtained as a white solid (16 mg, 68% yield); X-ray quality crystals were prepared via slow evaporation from isopropanol/dichloromethane; ^{1}H NMR (360 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.8 Hz, 2H), 8.18 (d, *J* = 8.8 Hz, 2H), 5.02 (t, *J* = 3.6 Hz, 1H), 4.46 (t, *J* = 3.9 Hz, 1H), 4.32-4.12 (m, 4H), 3.73 (d, *J* = 8.1 Hz, 1H), 3.66-3.58 (m, 1H), 2.73 (t, *J* = 6.5 Hz, 2H), 2.33 (br s, 1H), 1.73 (dd, *J* = 13.4, 9.1 Hz, 1H), 1.65 (d, *J* = 3.5 Hz, 1H), 1.61 (s, 1H), 1.45 (*J* = 13.9, 7.4 Hz, 1H), 1.32 (t, *J* = 3.6 Hz, 3H), 1.27 (t, *J* = 3.6 Hz, 3H), 1.00 (m, 11H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 171.0, 170.4, 150.7, 142.9, 130.1, 124.1, 76.7, 63.8, 63.5, 61.7, 61.4, 57.4, 52.4, 46.3, 40.3, 31.3, 30.0, 27.9, 17.6, 14.1, 13.9; HRMS-ES+ ($C_{25}H_{37}N_2O_9S$) calcd 541.2220 (MH+), found 541.2206.

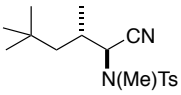
General Procedure for the Conversion of Aldoximes to Nitriles. A solution of α-alkyl aldoxime in pyridine (710 uL) was added MsCl (55 uL, 0.7 mmol) at 0 °C and the mixture was stirred for 12 h. The solution was diluted with H_2O and extracted with

CH_2Cl_2. The combined organic layers were washed with water, dried over Na_2SO_4 and concentrated *in vacuo* to give a residue which was purified by flash column chromatography on silica gel eluting with 5- 25% EtOAc/hexanes.

Diethyl 2-(1-Cyano-2-phenylpropyl)-2-ethylmalonate. The product was obtained as a clear oil (17 mg, 73% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.27 (m, 5H), 4.32-4.19 (m, 4H), 3.59 (d, *J* = 3.1 Hz, 1H), 3.25-3.16 (m, 1H), 2.25-2.14 (m, 2H), 1.43 (d, *J* = 7.1 Hz, 3H), 1.36-1.29 (m, 6H), 0.94 (t, *J* = 3.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 169.3, 145.0, 129.3, 127.7, 127.4, 118.5, 62.5, 59.8, 42.9, 37.3, 27.2, 18.8, 14.4, 9.1; HRMS-ES+ ($C_{19}H_{26}NO_4$) calcd 332.1862 (MH+), found 332.1860.



***N*-(1-Cyano-2,4,4-trimethylpentyl)-*N*,4-dimethylbenzenesulfonamide (21)**. The product was obtained as a clear oil (17 mg, 60% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 4.43 (d, *J* = 10.3 Hz, 1H), 2.83 (s, 3H), 2.48 (s, 3H), 1.92-1.86 (m, 1H), 1.75 (d, *J* = 13.9 Hz, 2H), 1.22 (d, *J* = 6.6 Hz, 3H), 0.97 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 133.8, 130.5, 128.0, 115.7, 56.5, 46.1, 32.2, 31.7, 30.6, 30.1, 22.1, 20.1; HRMS-ES+ ($C_{17}H_{27}N_2O_2S$) calcd 323.1793 (MH+), found 323.1797.



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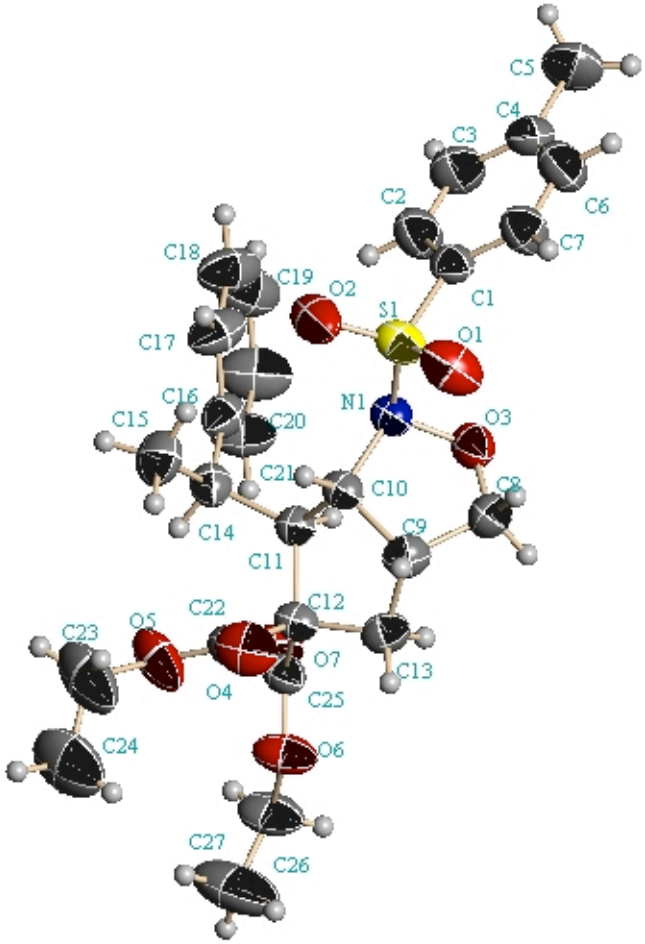
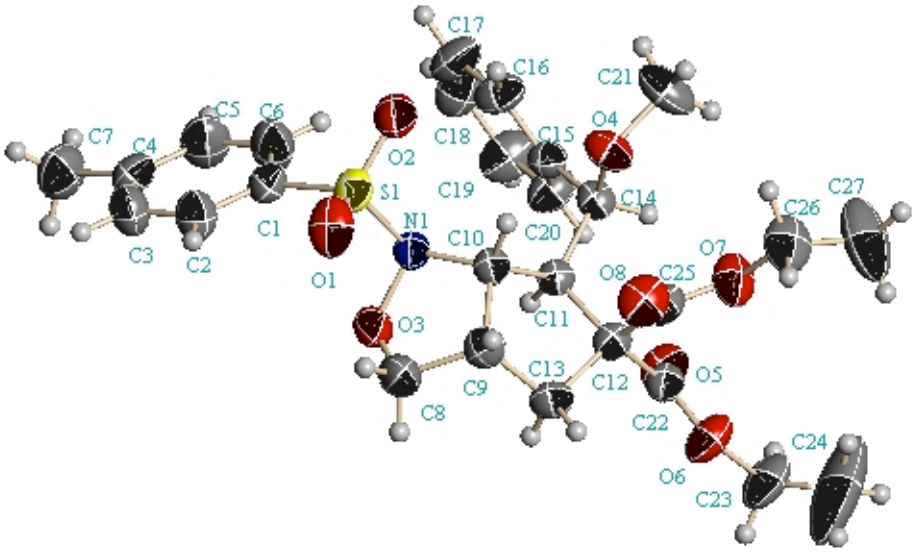
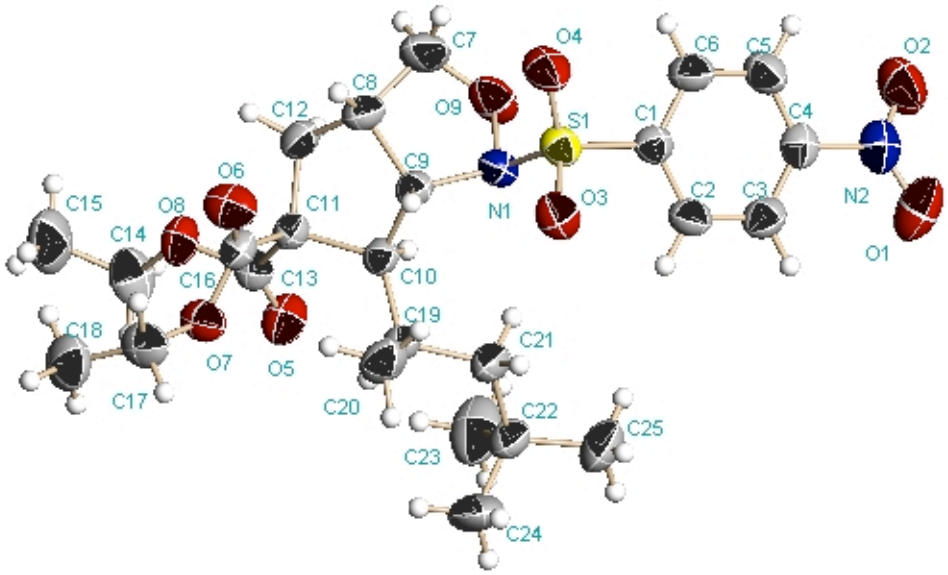
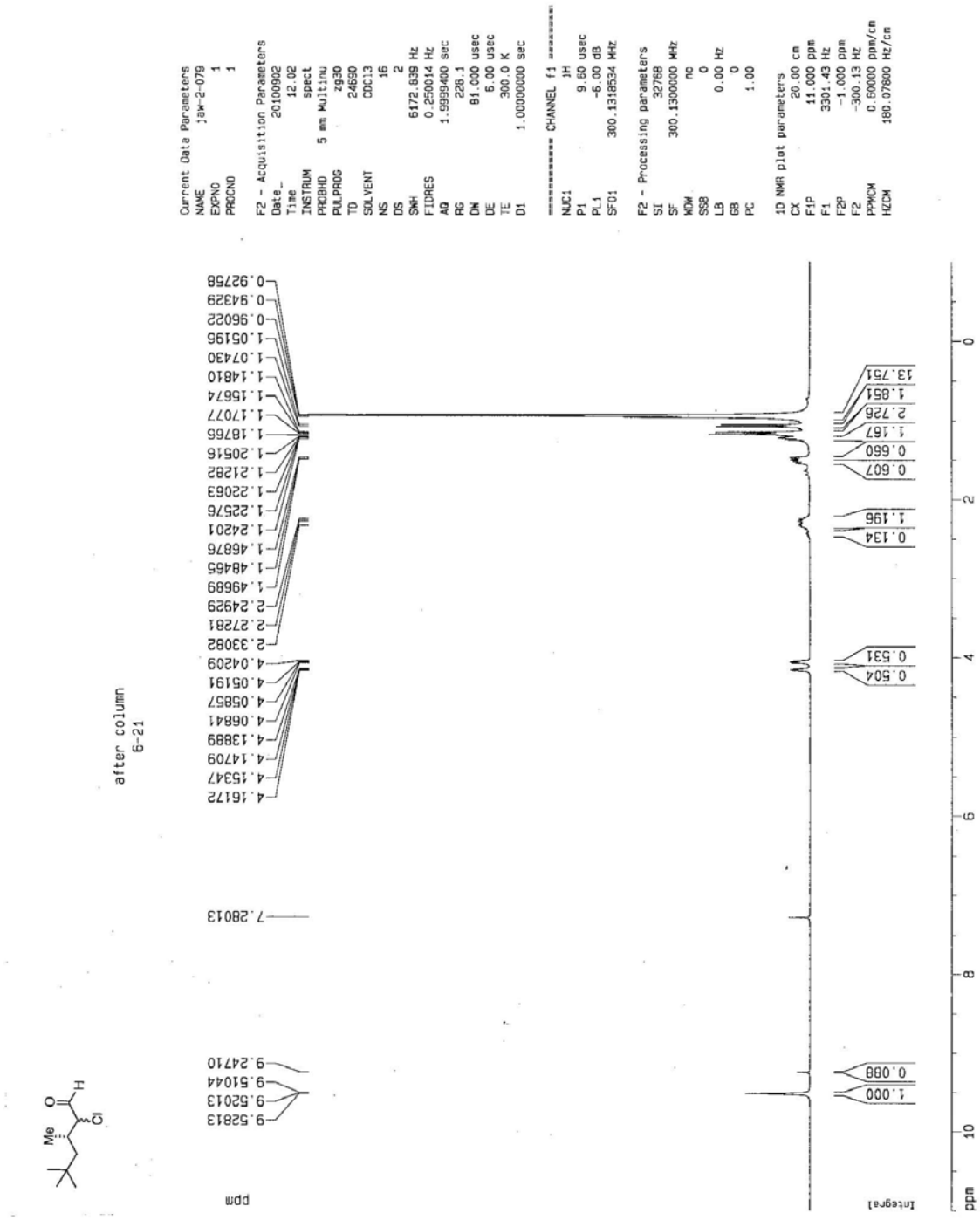


X-Ray Structure of Compound **15**.



X-Ray Structure of Compound **19**.

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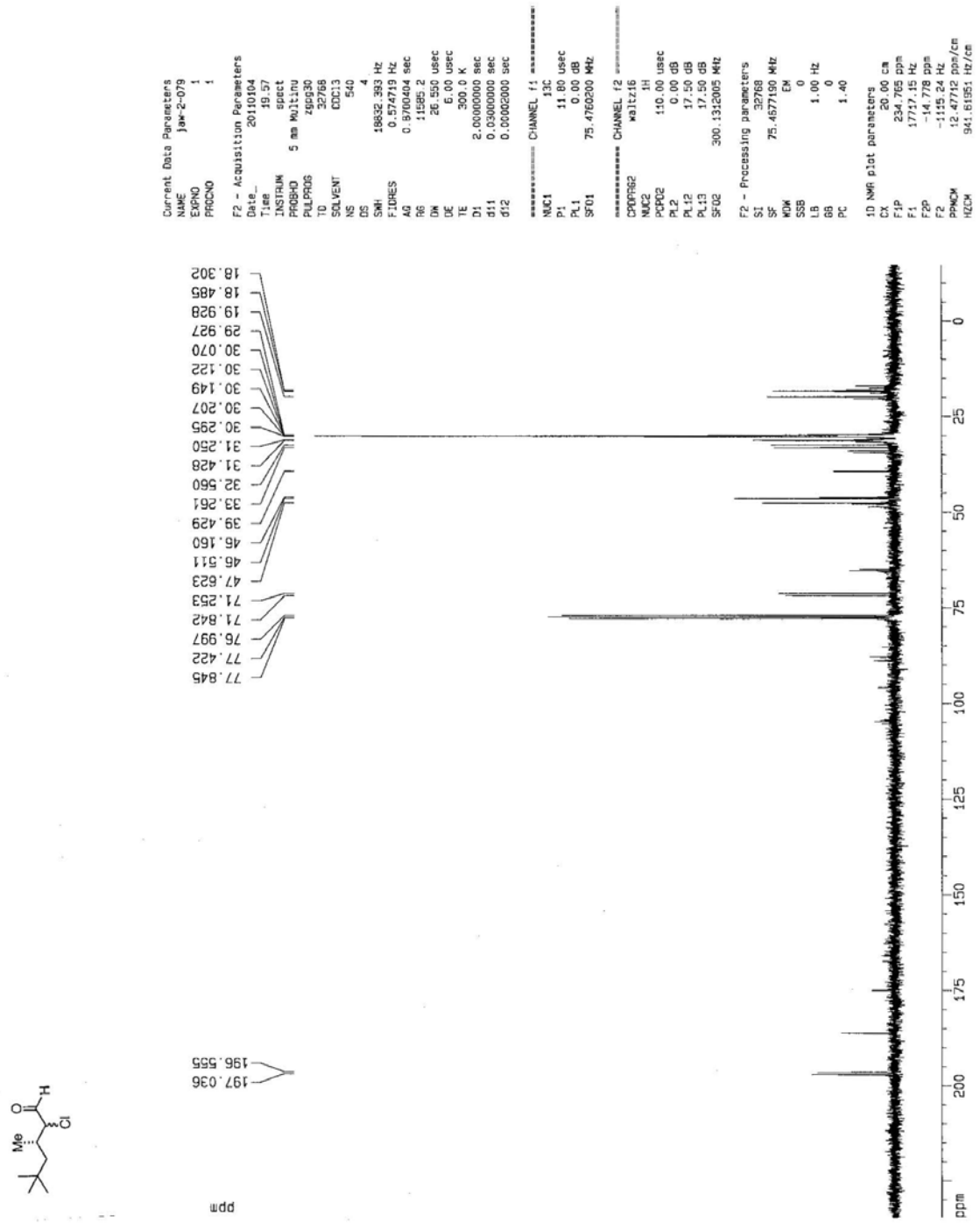
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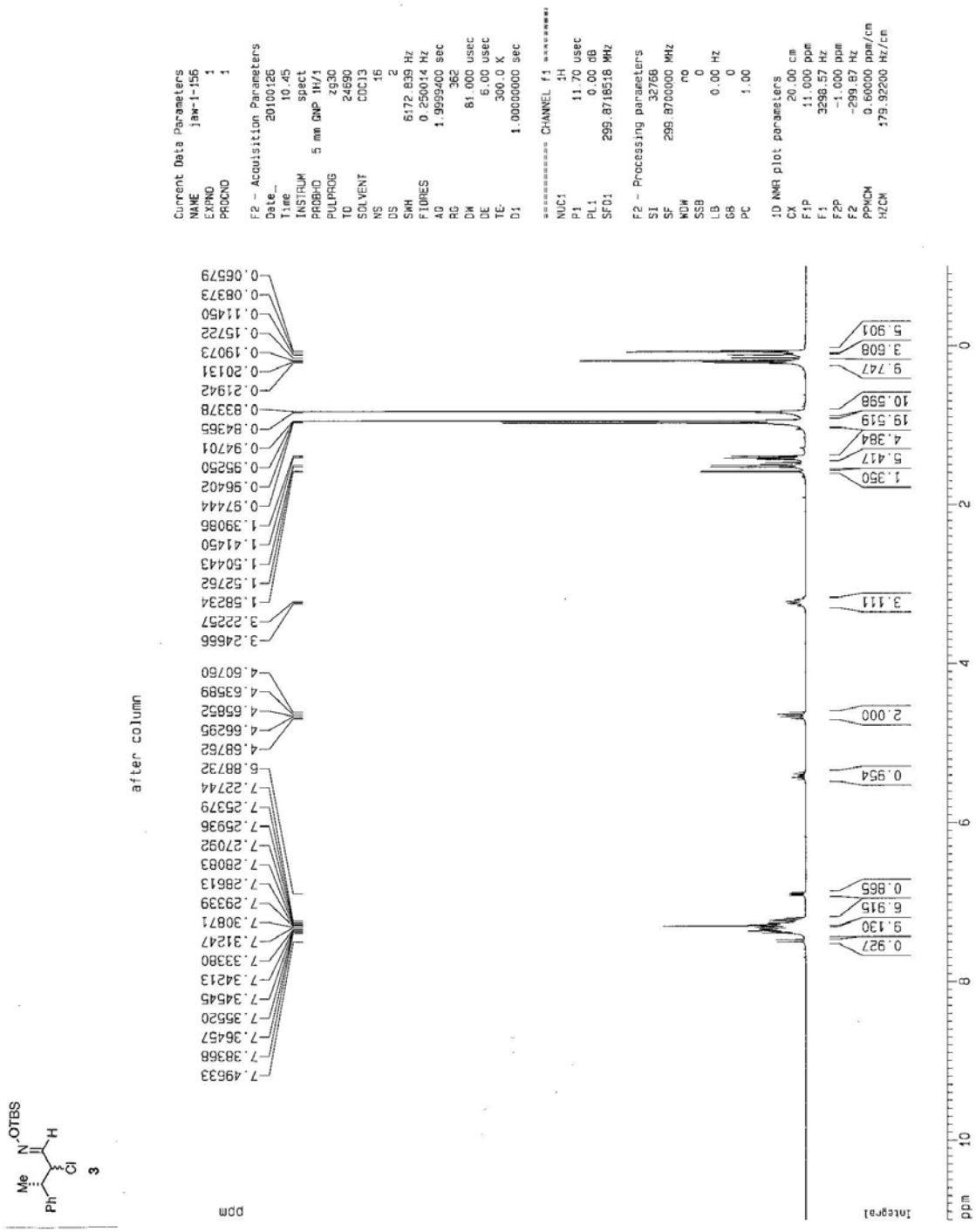
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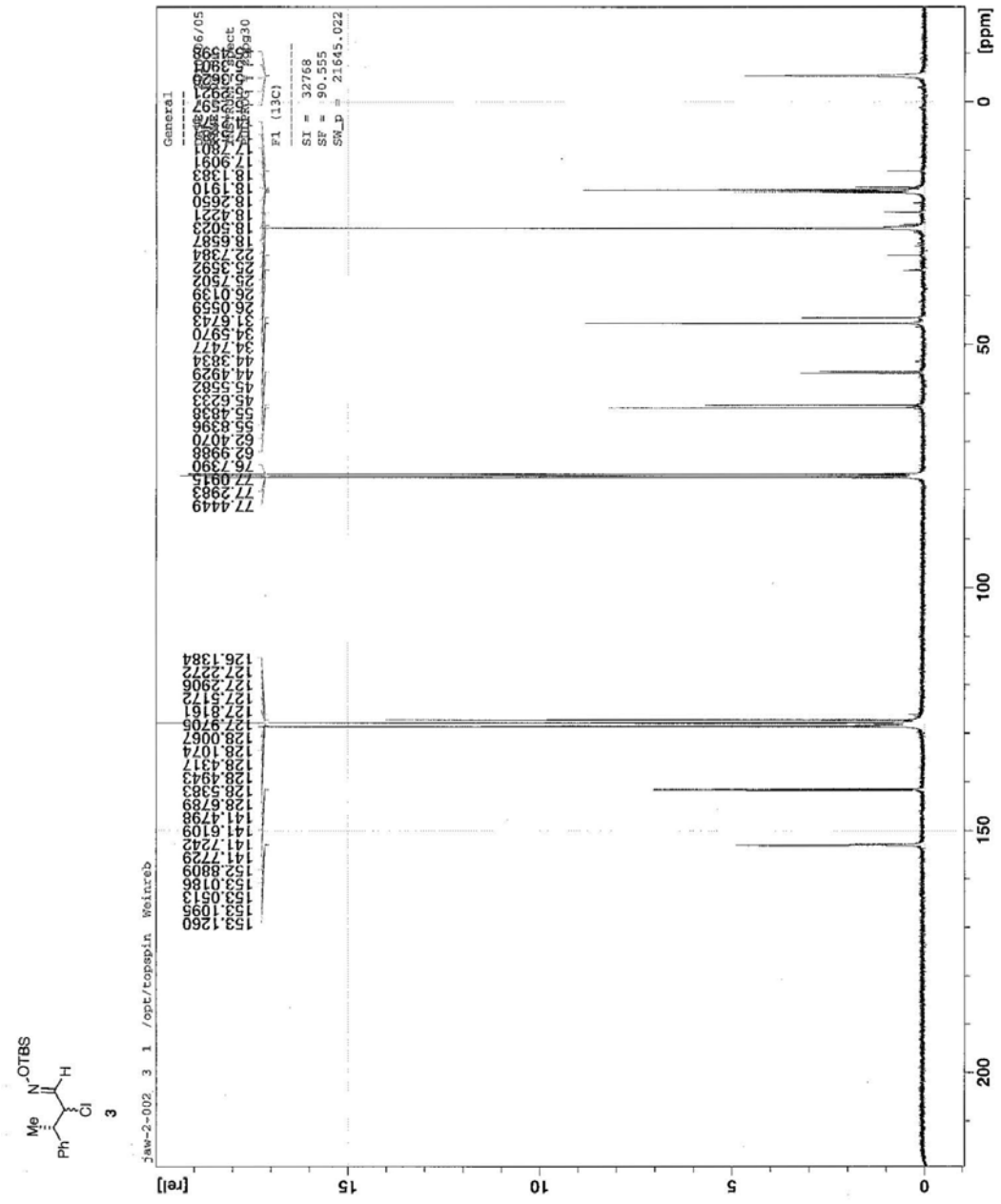
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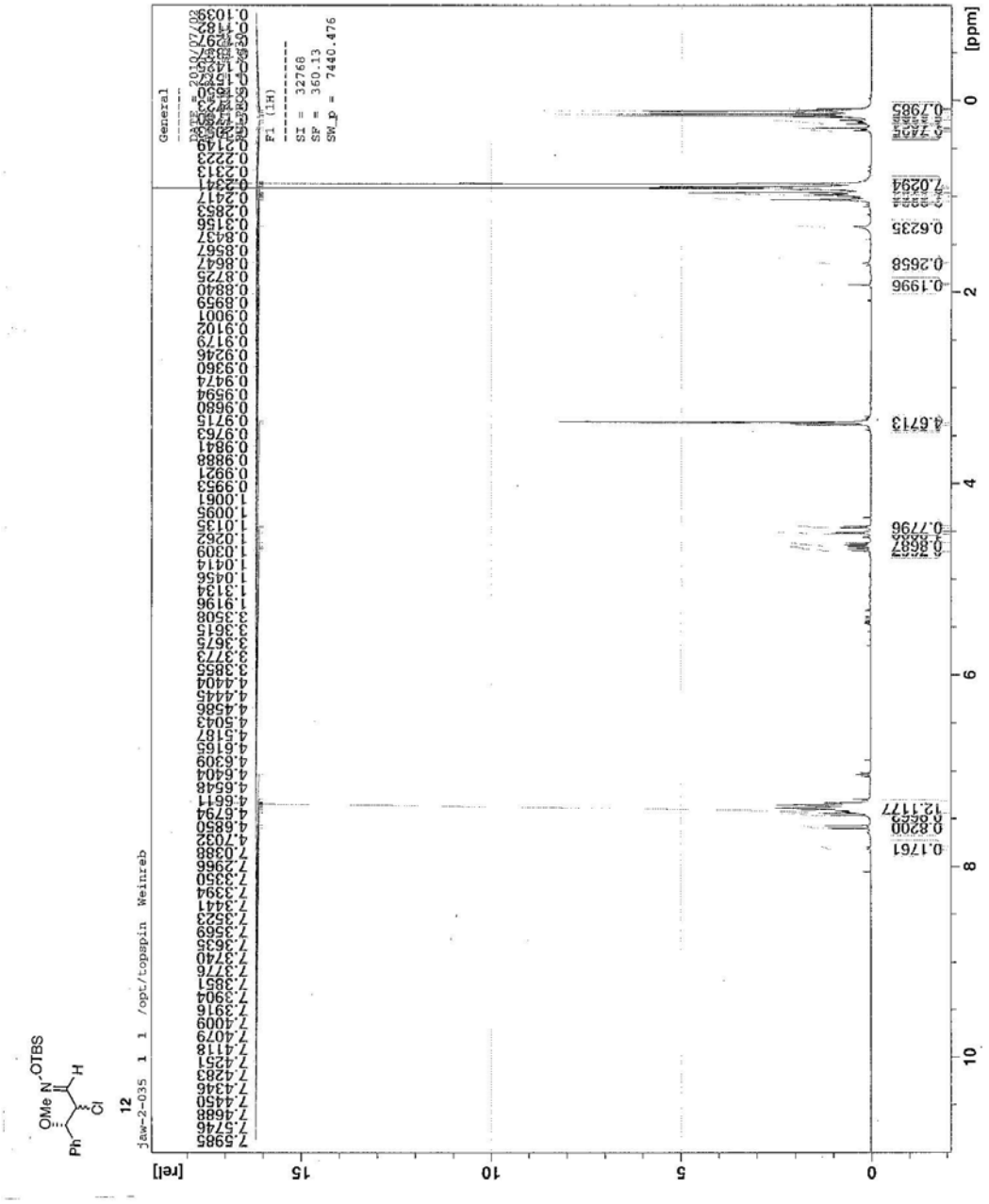
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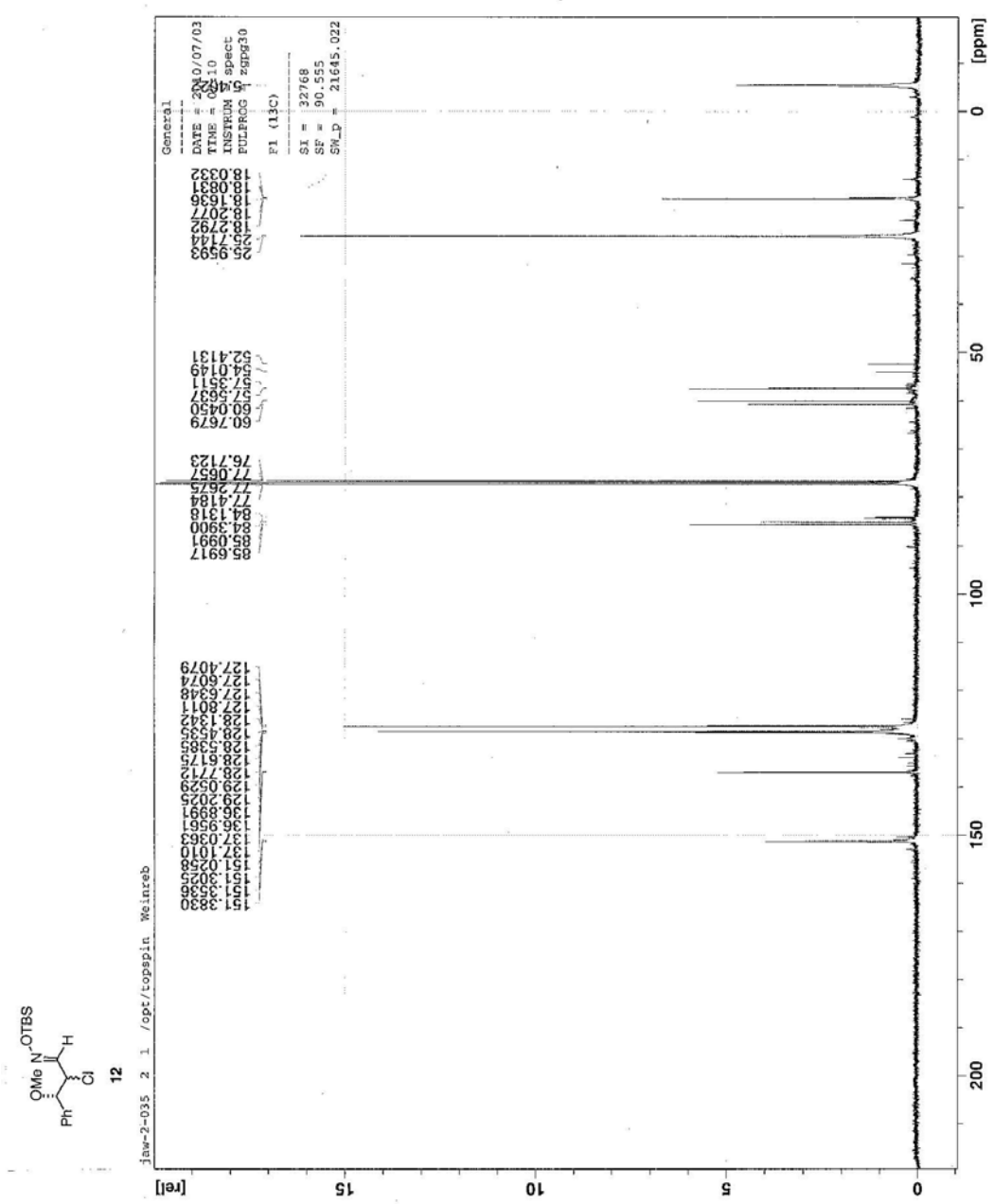
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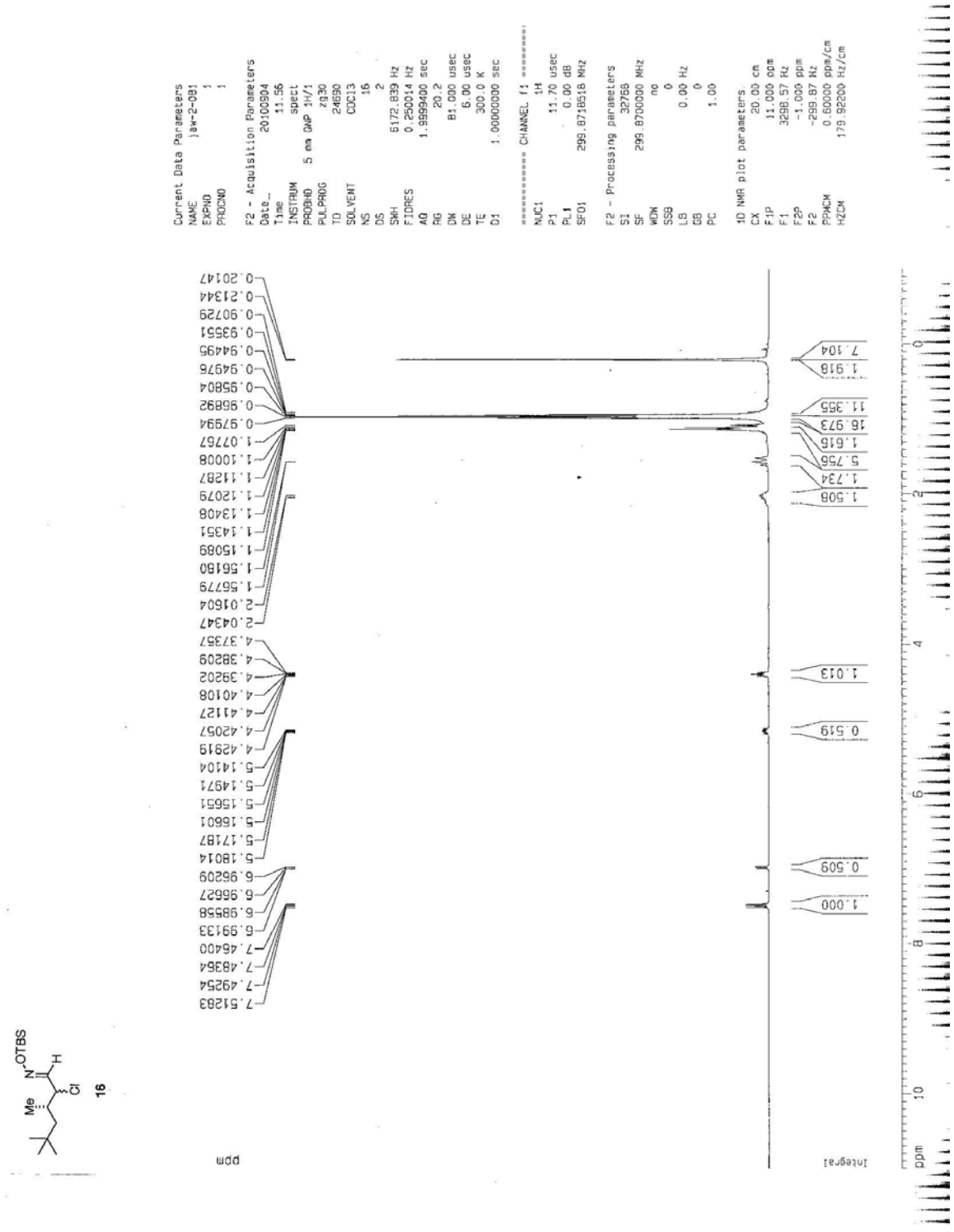
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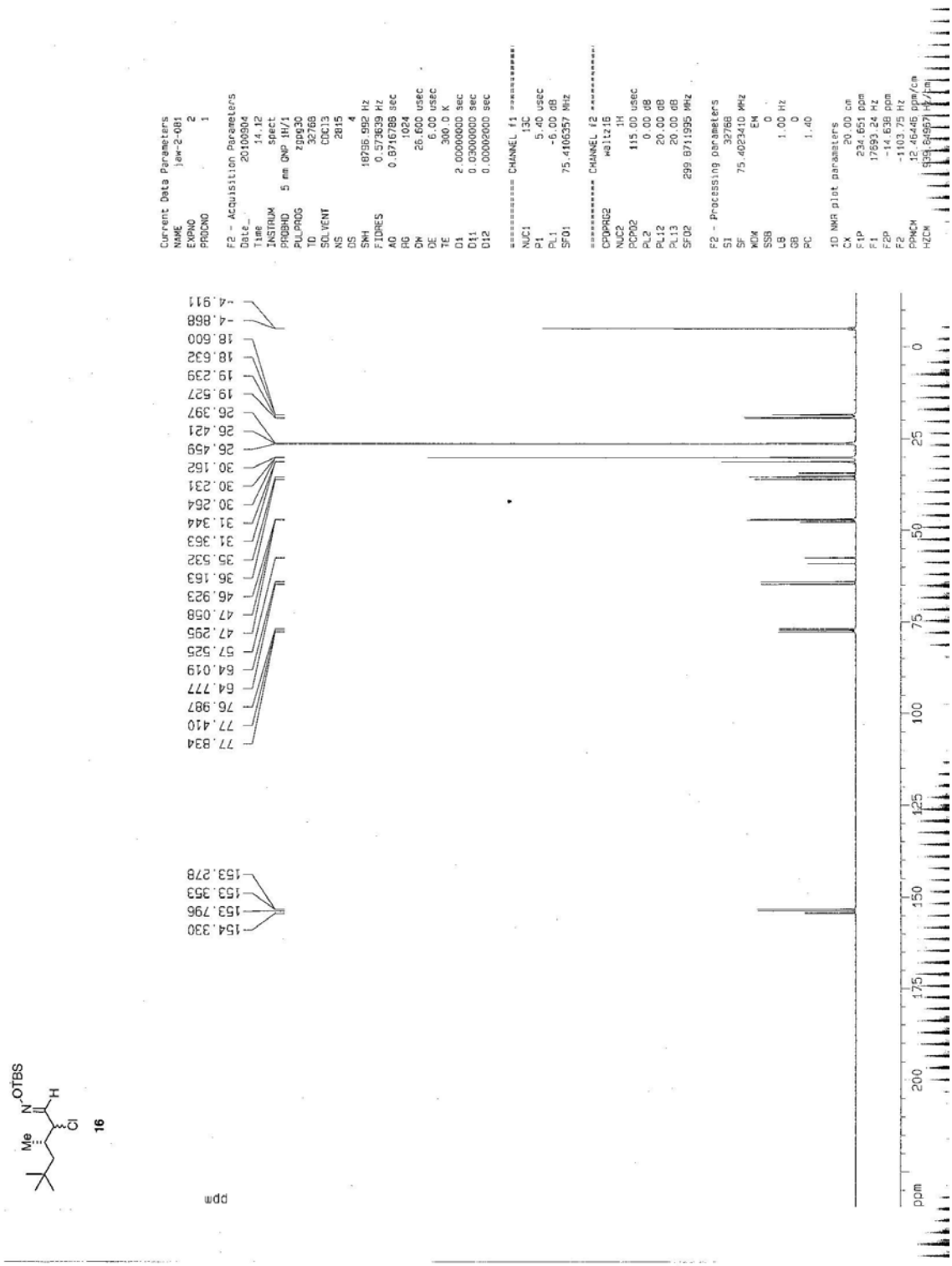
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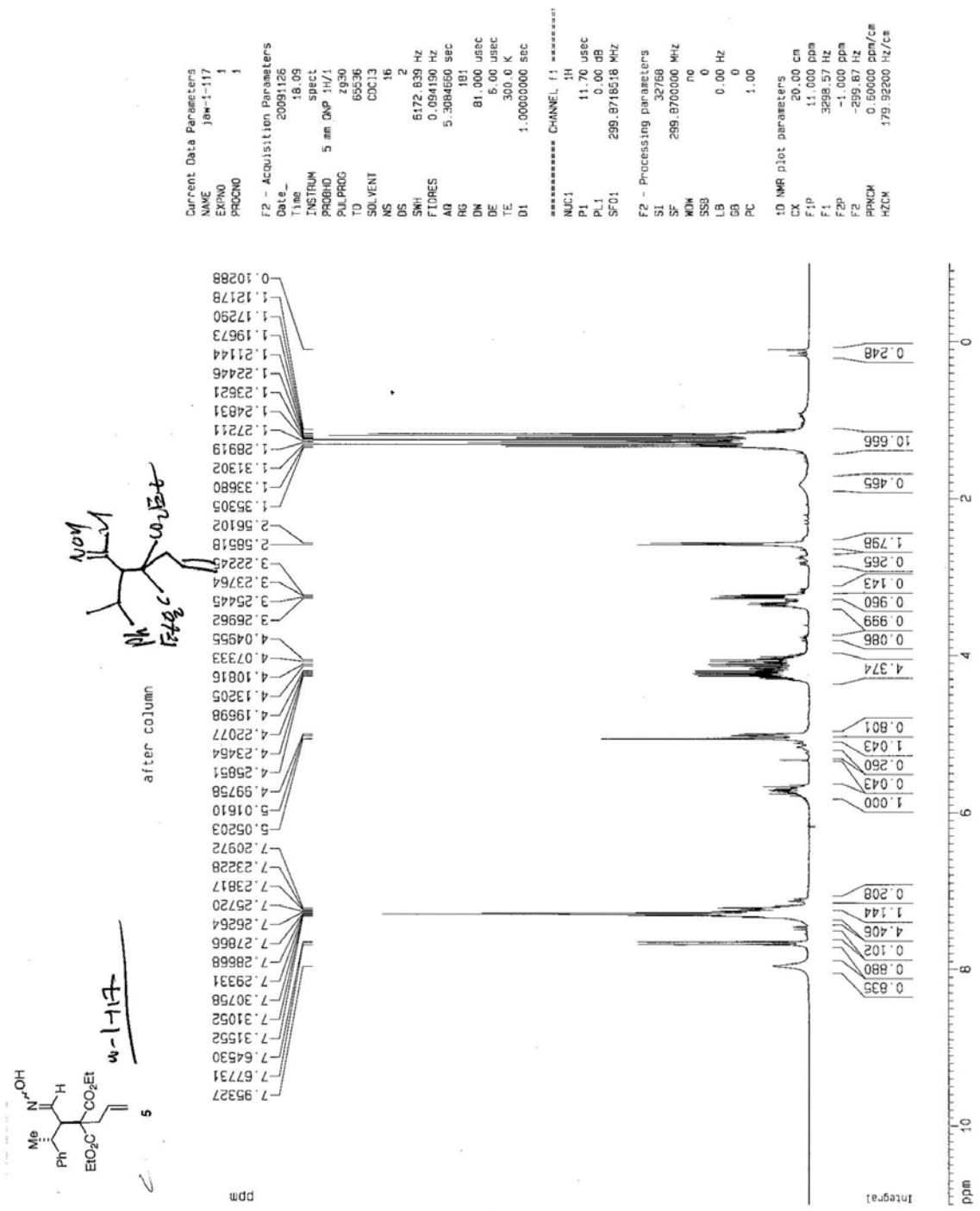
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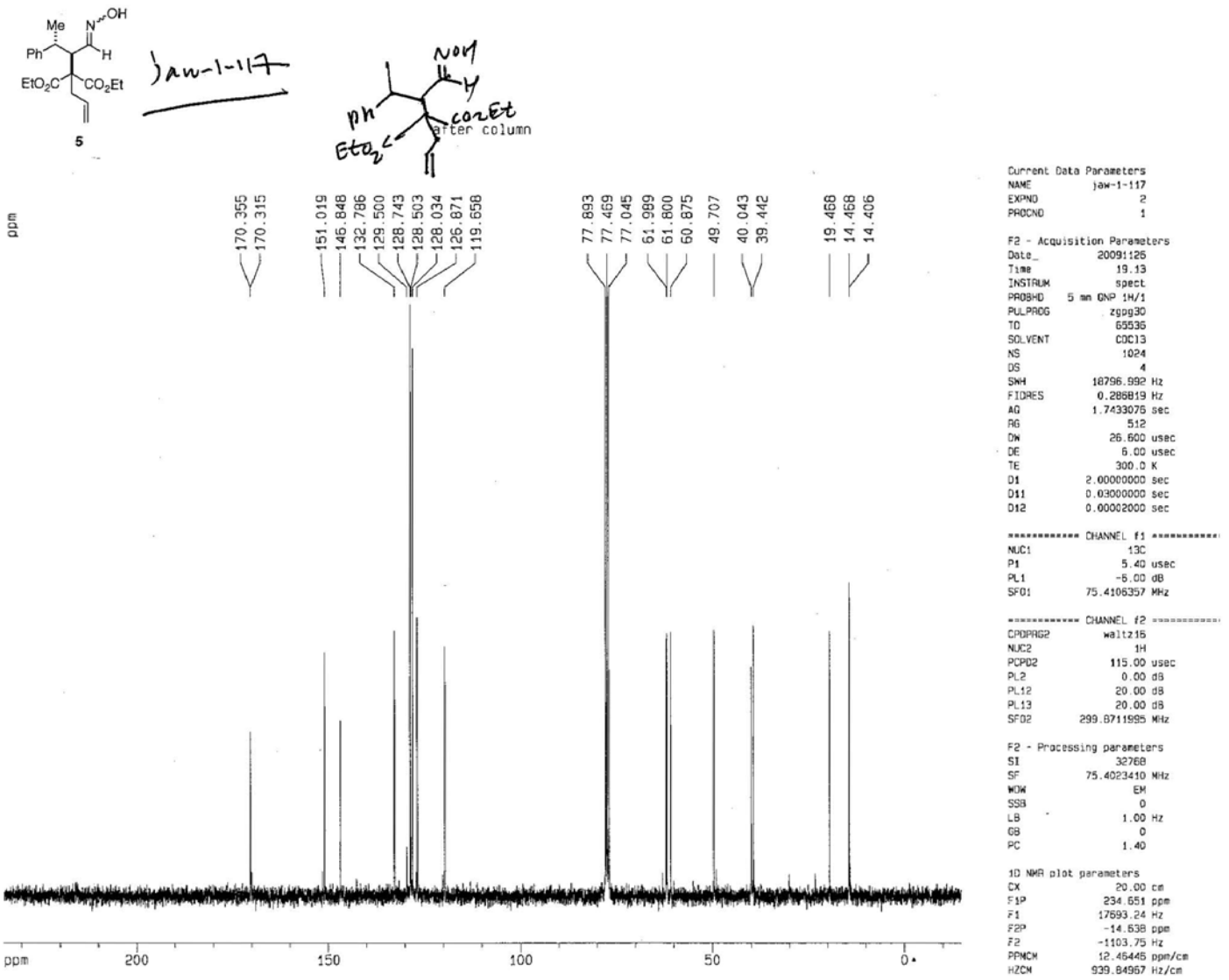
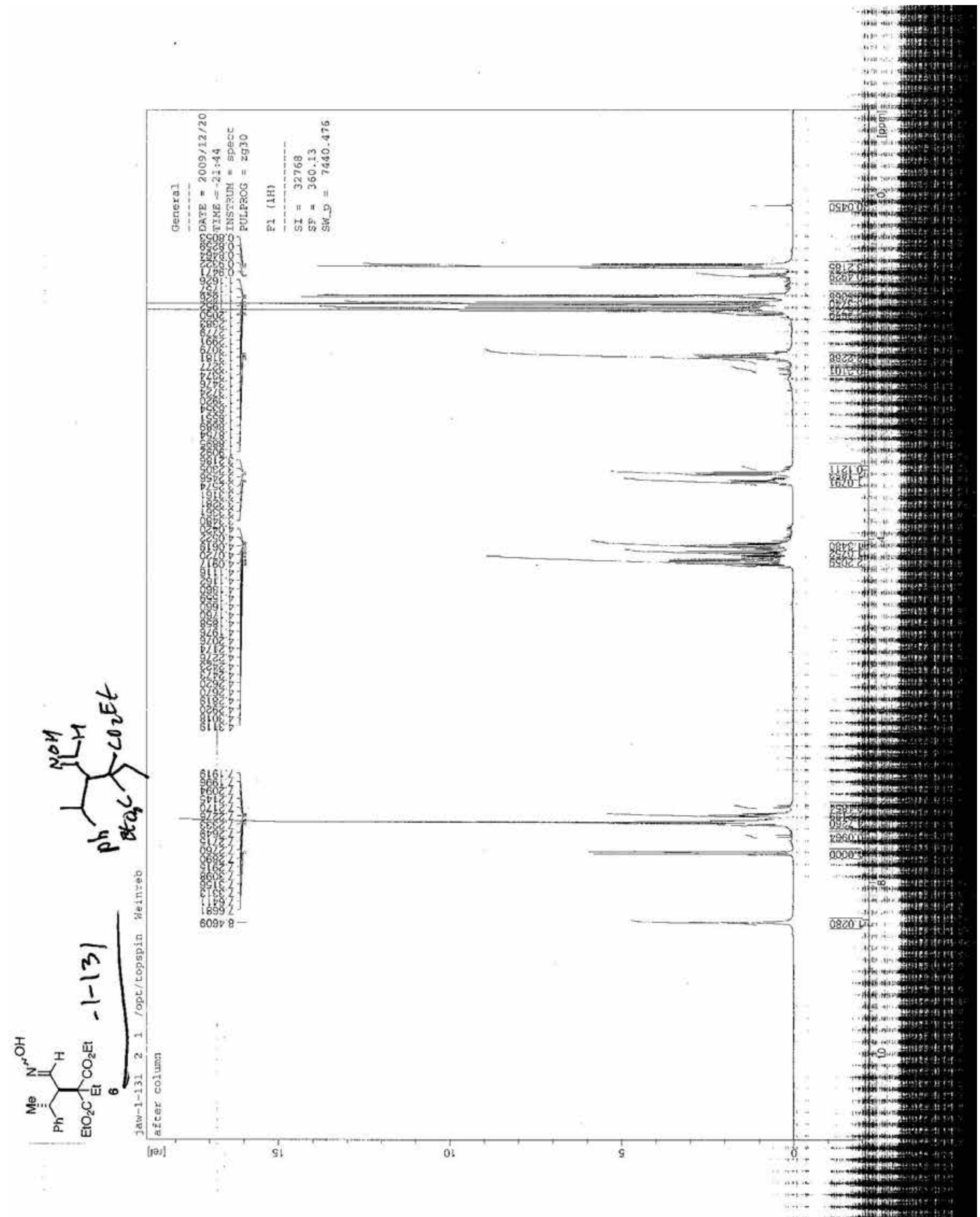
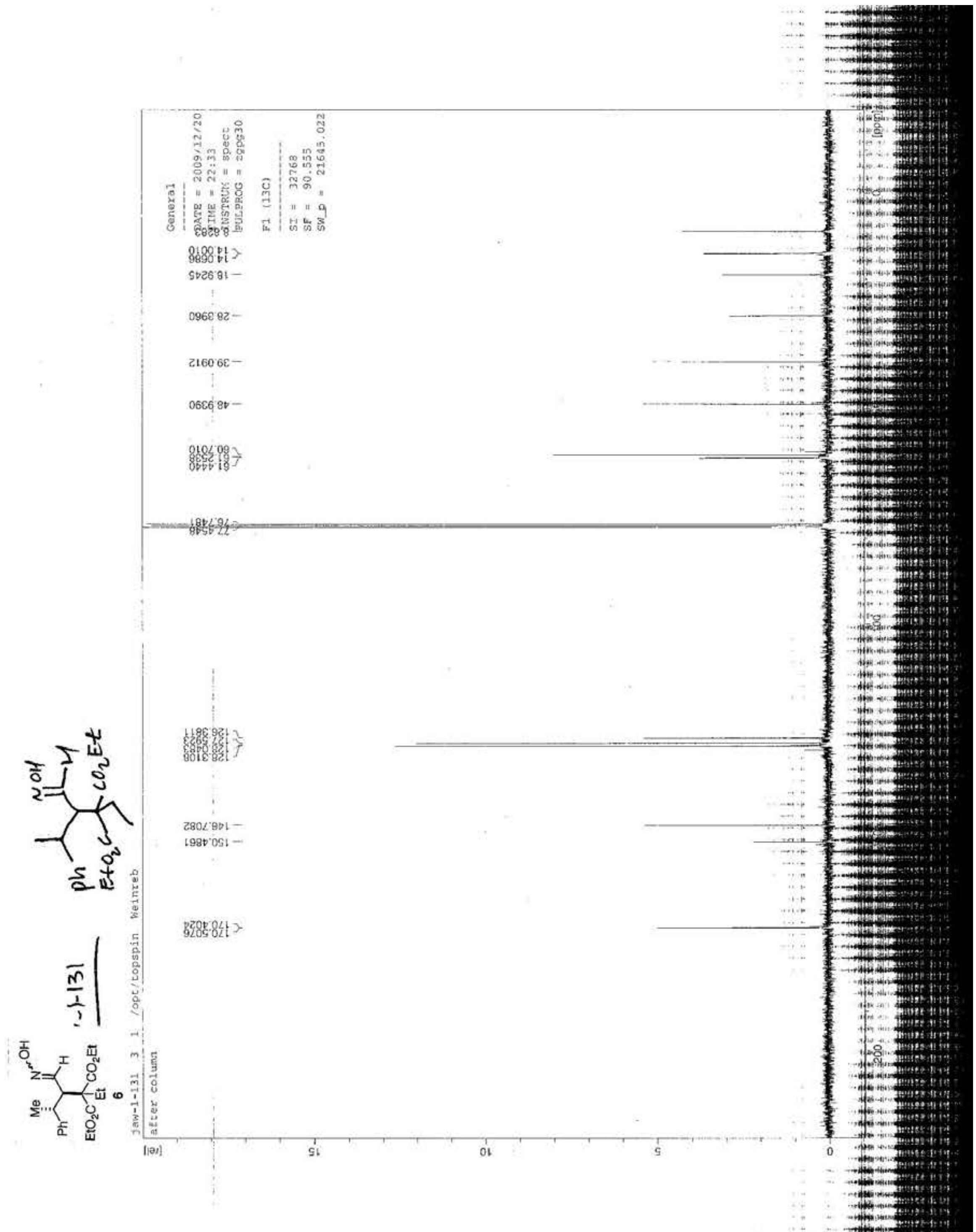
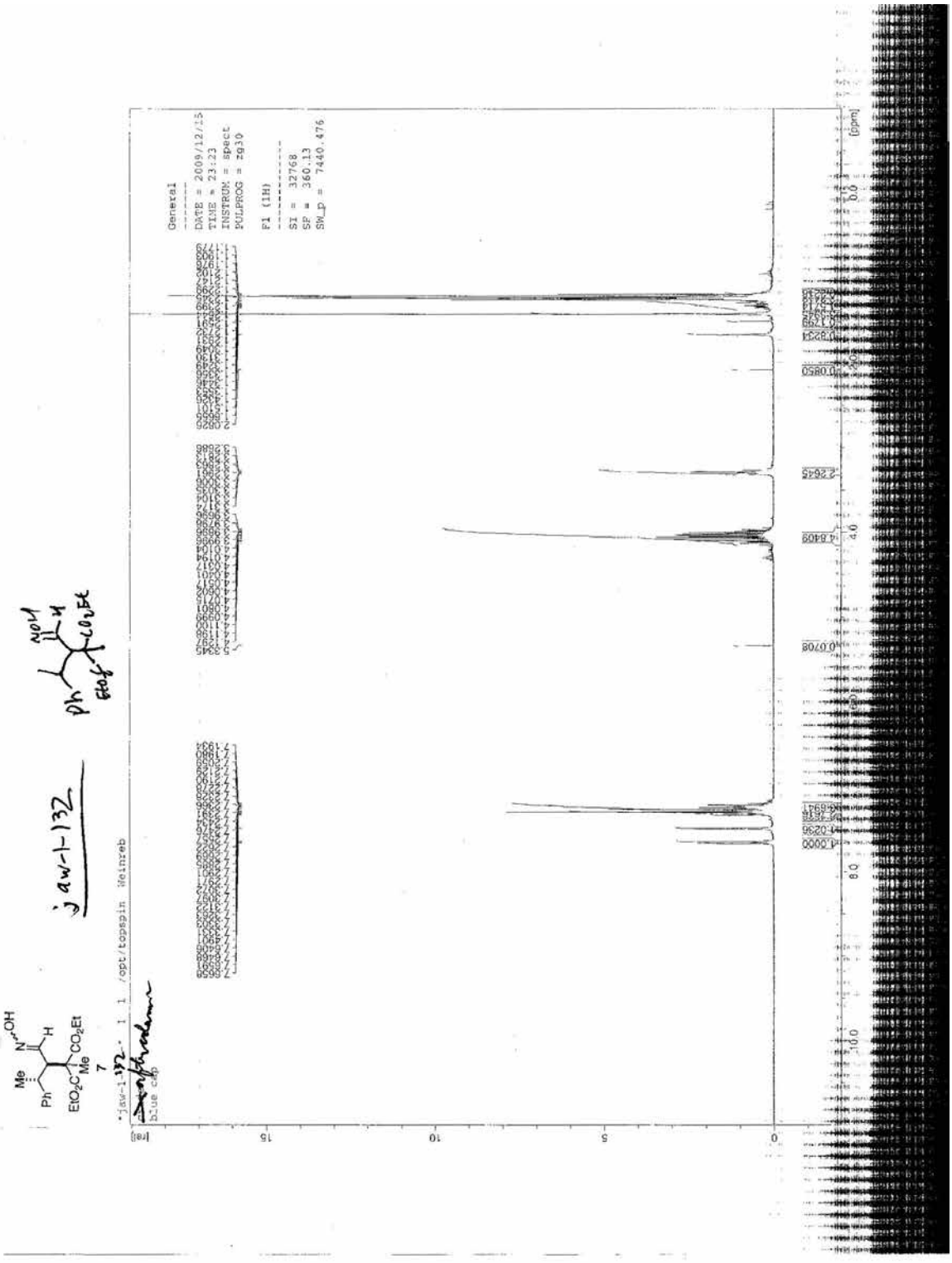
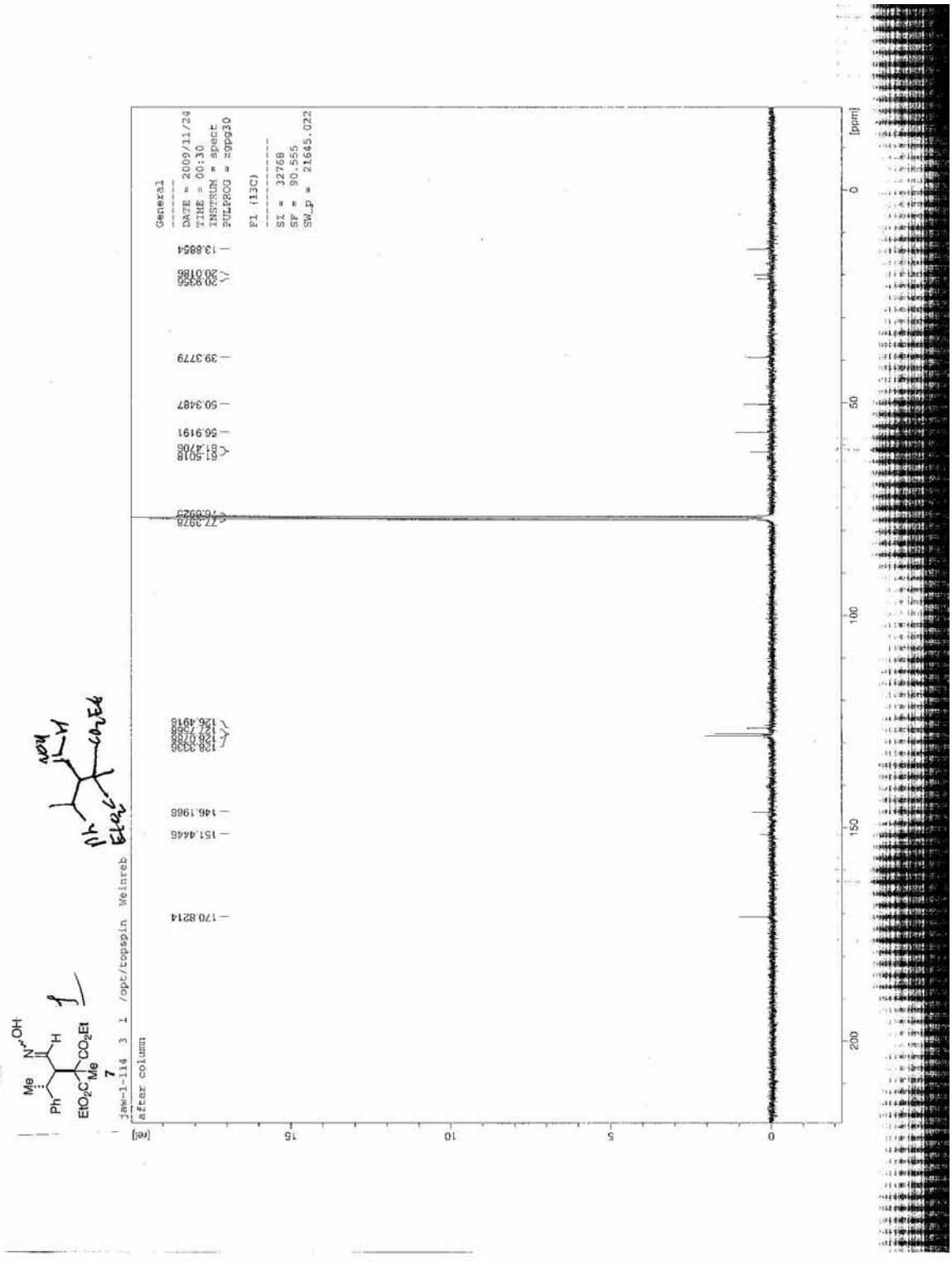
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.67 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
w-1-117
after column
5
ppm
Integral

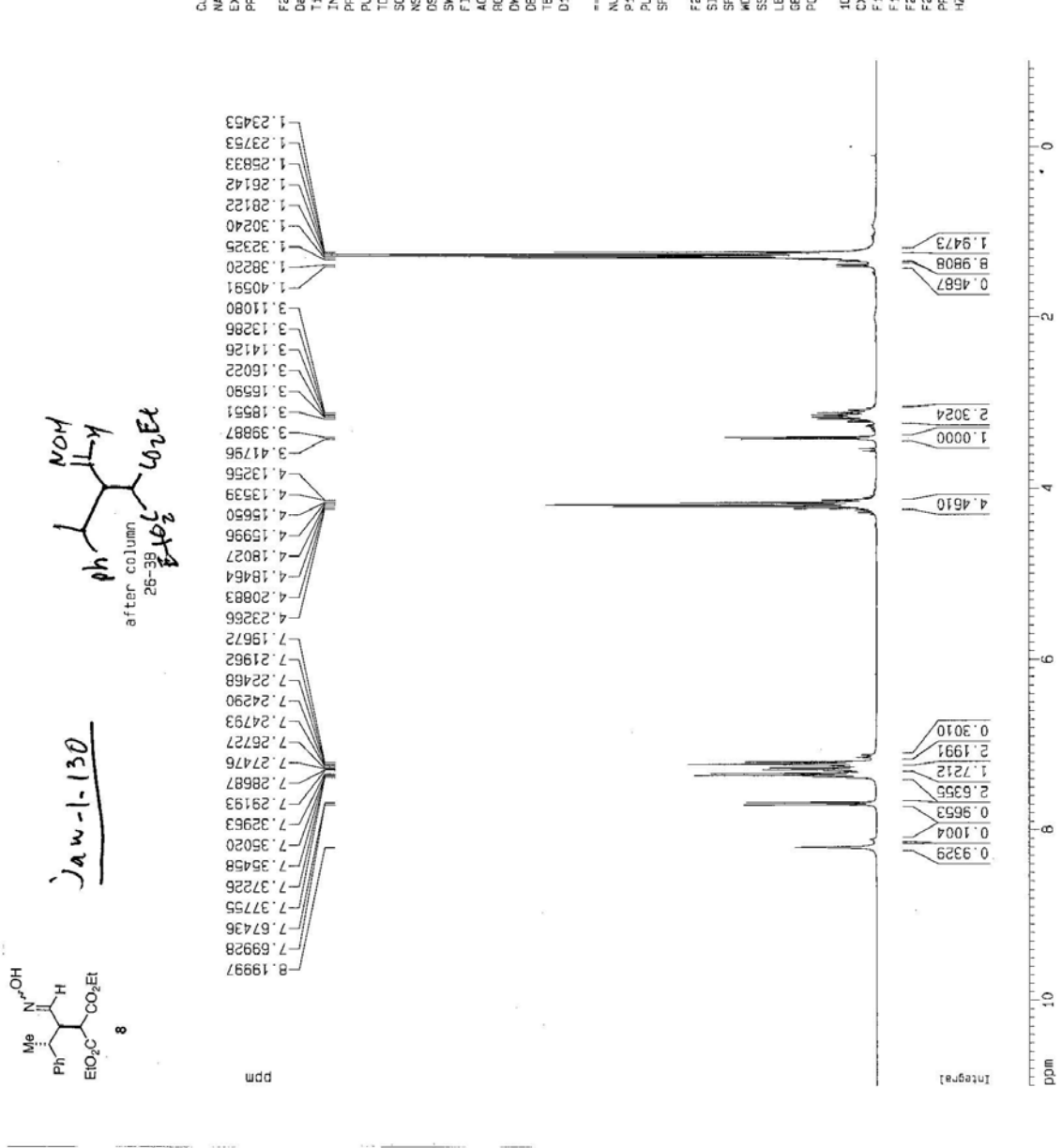

Me
N
OH
Ph
H
EtO2C
CO2Et
5
jaw-1-117
NOH
Ph
CO2Et
EtO2C
after column
ppm
170.355
170.315
151.019
145.848
132.786
129.500
128.743
128.503
128.034
126.871
119.658
77.893
77.469
77.045
61.989
61.800
60.875
49.707
40.043
39.442
19.468
14.468
14.406
ppm
200
150
100
50
0
Current Data Parameters
NAME jaw-1-117
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091126
Time 19.13
INSTRUM spect
PROBHD 5 mm CNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433075 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46445 ppm/cm
HZCM 939.84967 Hz/cm

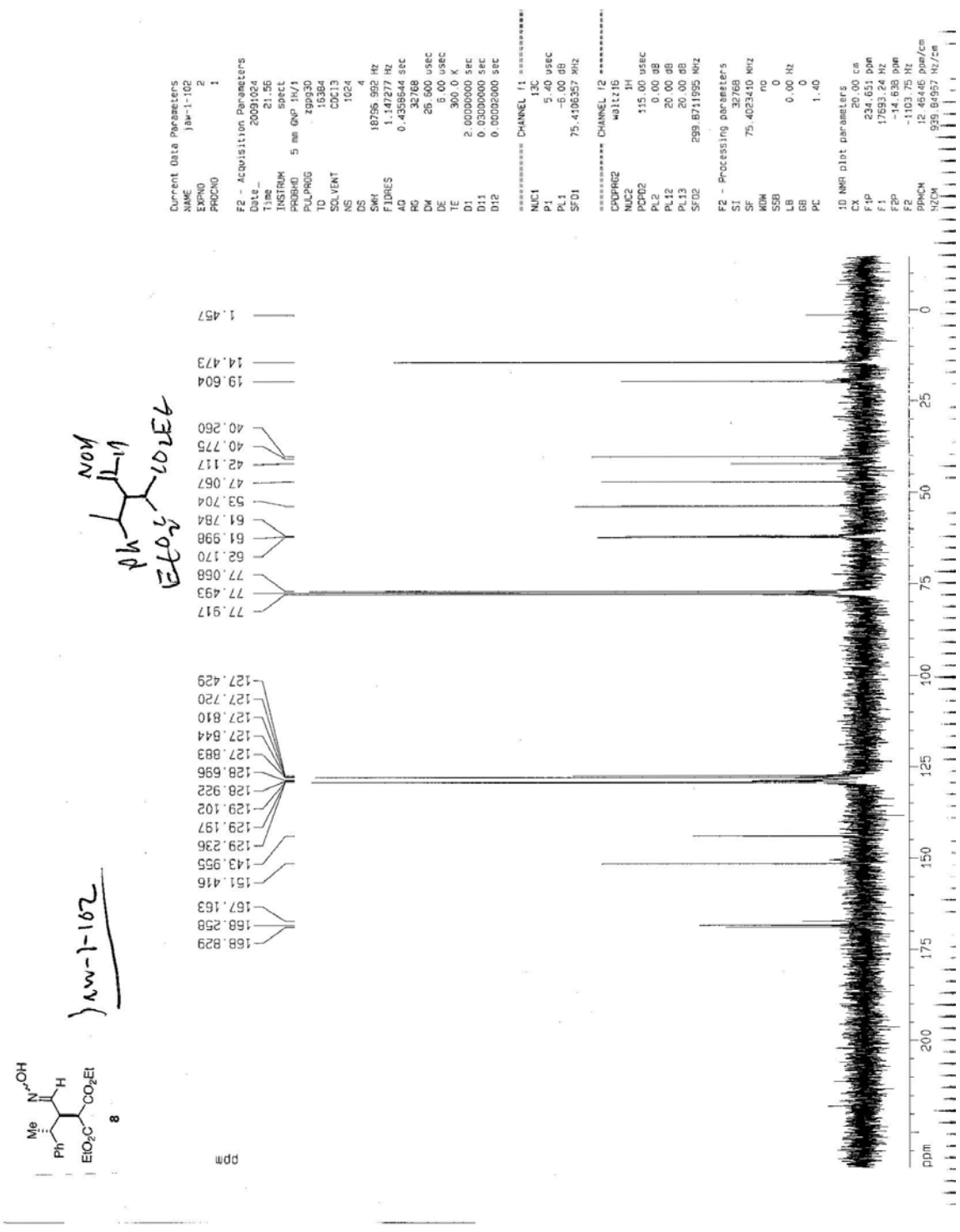

jaw-1-131 2 1 /opt/topspin Weinreb
after column
General
DATE = 2009/12/20
TIME = 21:44
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
[ppm]

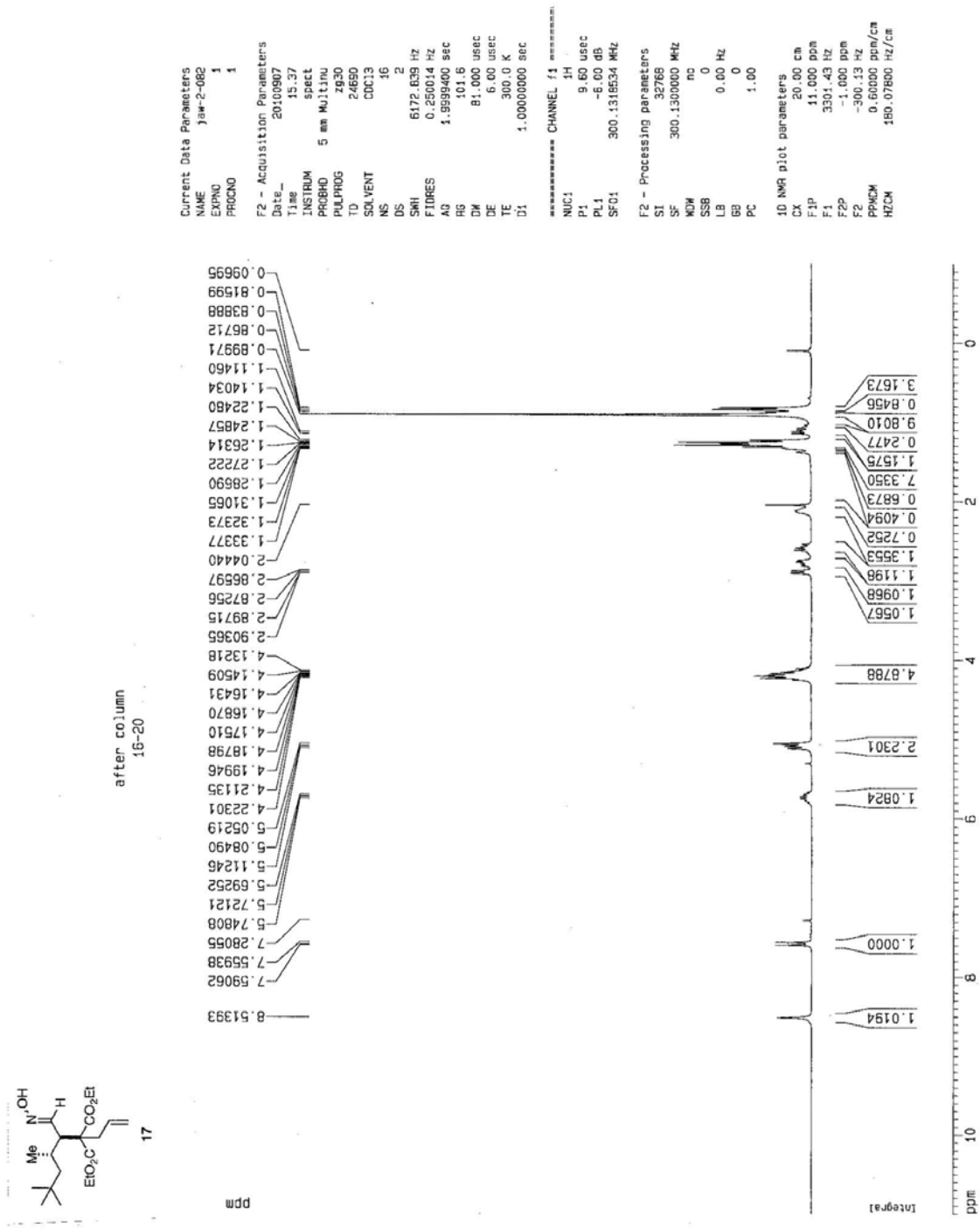

General
DATE = 2009/12/20
TIME = 22:33
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
jaw-1-131 3 1 /opt/topspin Weinreb
after column
170.5076
170.4024
150.4861
148.7082
77.4548
76.7481
61.4440
60.7010
48.9990
39.0912
28.8960
18.9245
14.0686
14.0010
6

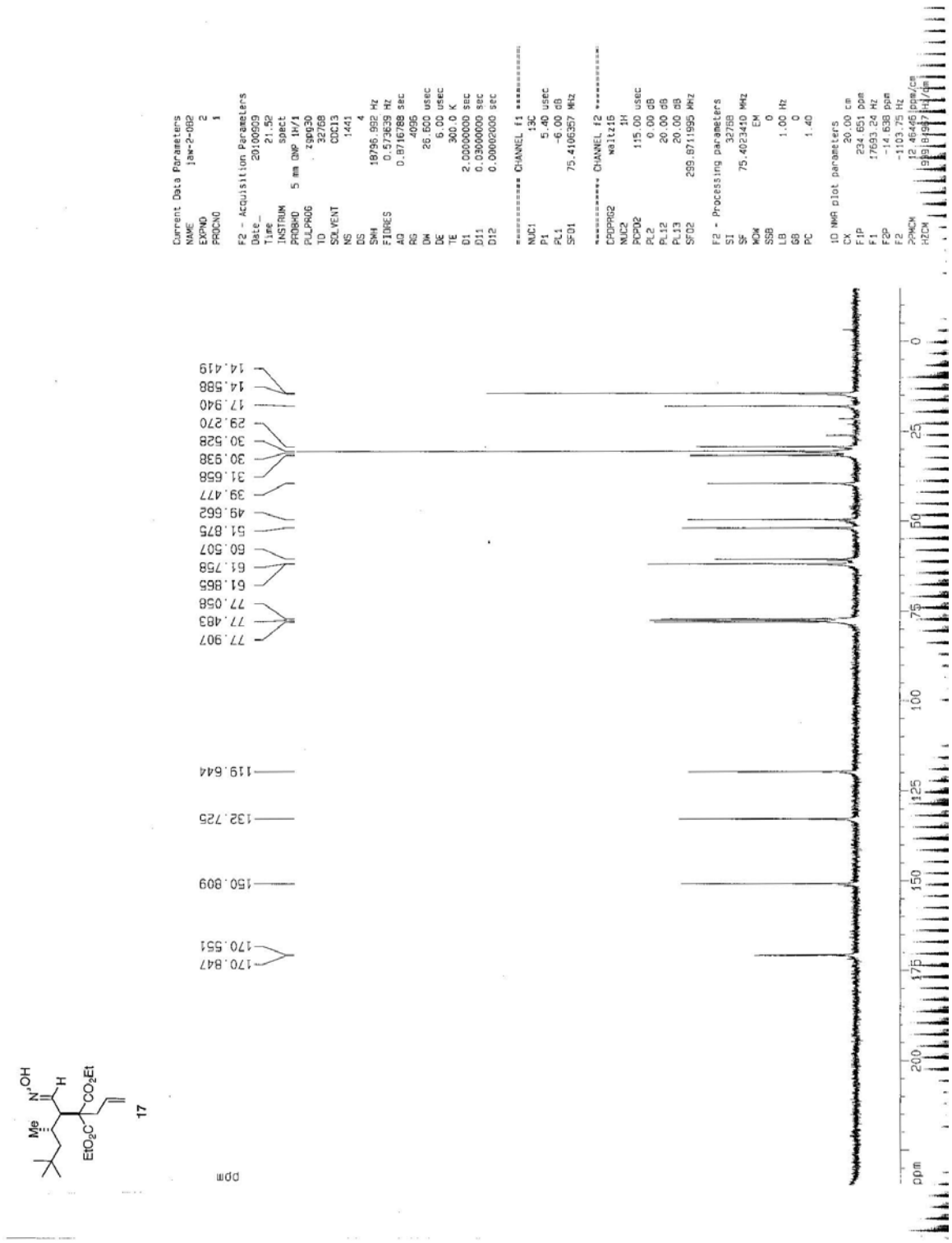


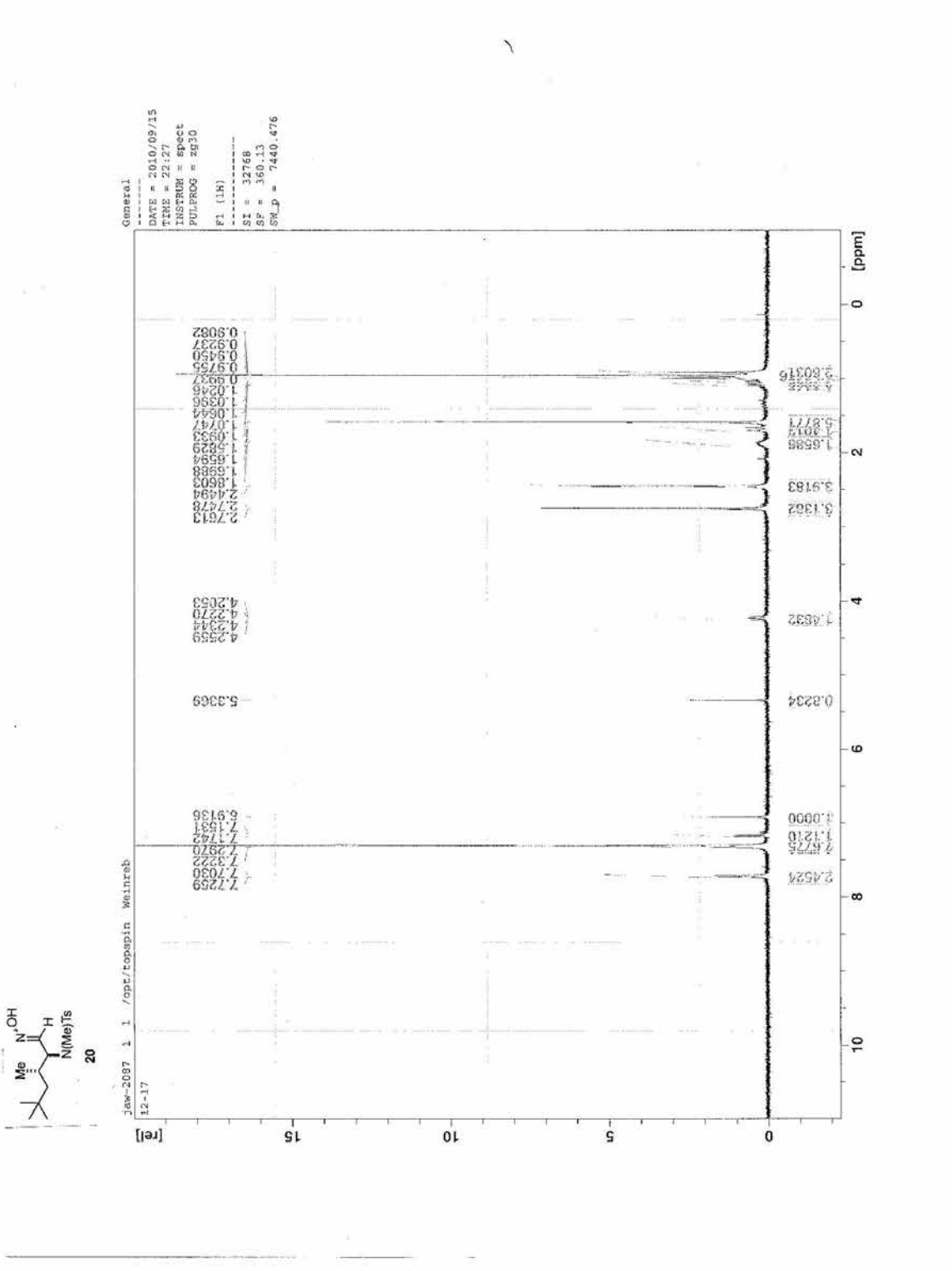

Me
N
OH
H
Ph
EtO2C
CO2Et
Me
7
jaw-1-114 3 1 /opt/topspin Weinreb
after column
General
DATE = 2009/11/24
TIME = 00:30
INSTRUN = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
170.8214
151.4446
146.1988
128.3336
126.4916
77.3978
61.5018
61.4706
56.9191
50.3487
39.3779
20.9950
20.0186
13.8854
[rel]
15
10
5
0
[ppm]
200
150
100
50
0

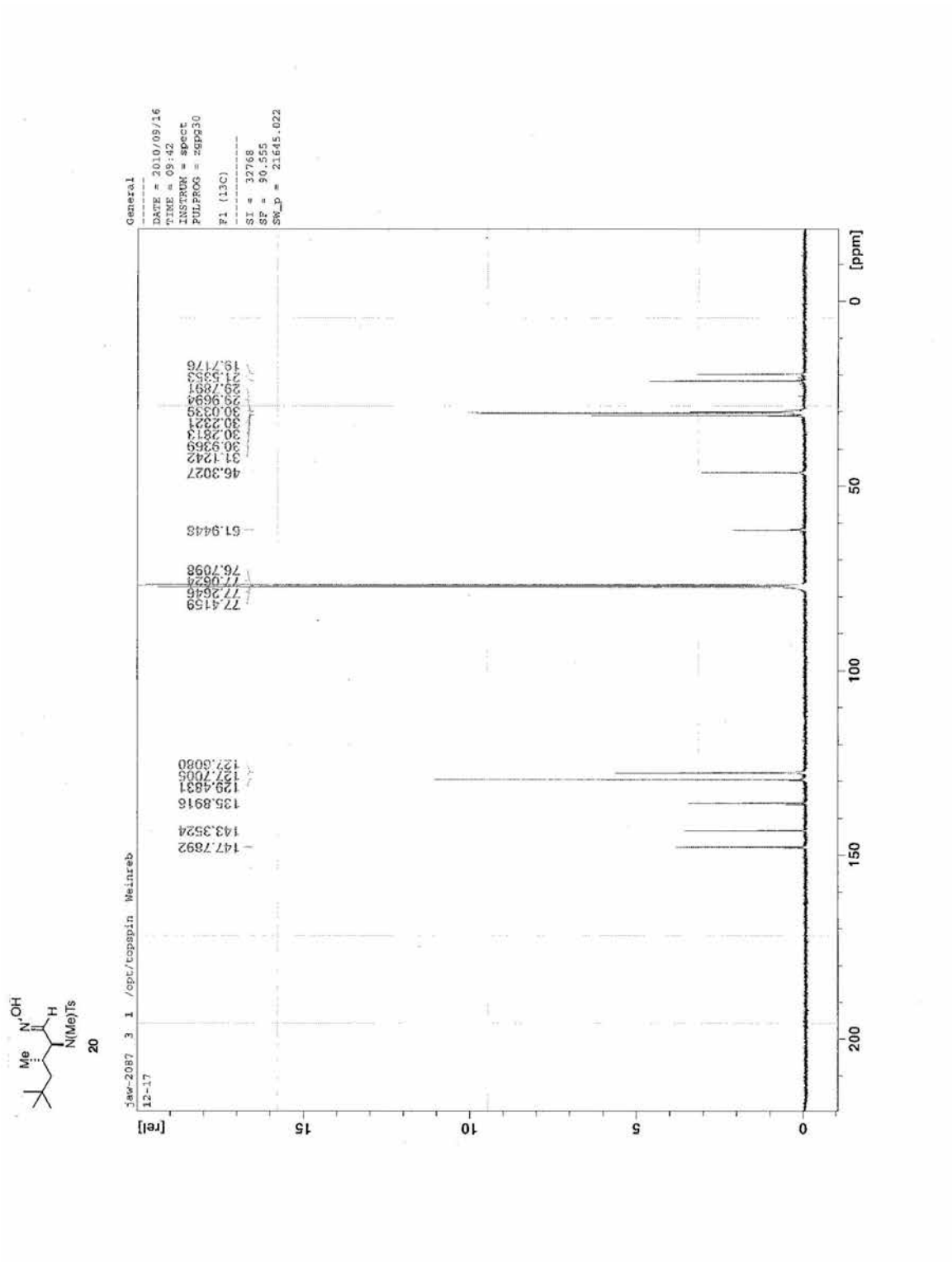

Me
N
OH
H
Ph
EtO2C
CO2Et
8
jaw-1-130
after column
26-38
Current Data Parameters
NAME jaw-1-130
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 22.10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8716518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
ppm
Integral
8.19897
7.69928
7.67436
7.37755
7.37226
7.35458
7.35020
7.32963
7.29193
7.28687
7.27476
7.26727
7.24793
7.24290
7.22468
7.21962
7.19672
4.23256
4.20883
4.18464
4.18027
4.15996
4.15650
4.13539
4.13256
3.41796
3.39887
3.18551
3.16590
3.16022
3.14126
3.13286
3.11080
1.40591
1.38220
1.33325
1.30240
1.28122
1.26142
1.25833
1.23753
1.23453
0.9329
0.1004
0.9653
2.6355
1.7212
2.1991
0.3010
4.4610
1.0000
2.3024
0.4687
8.9808
1.9473

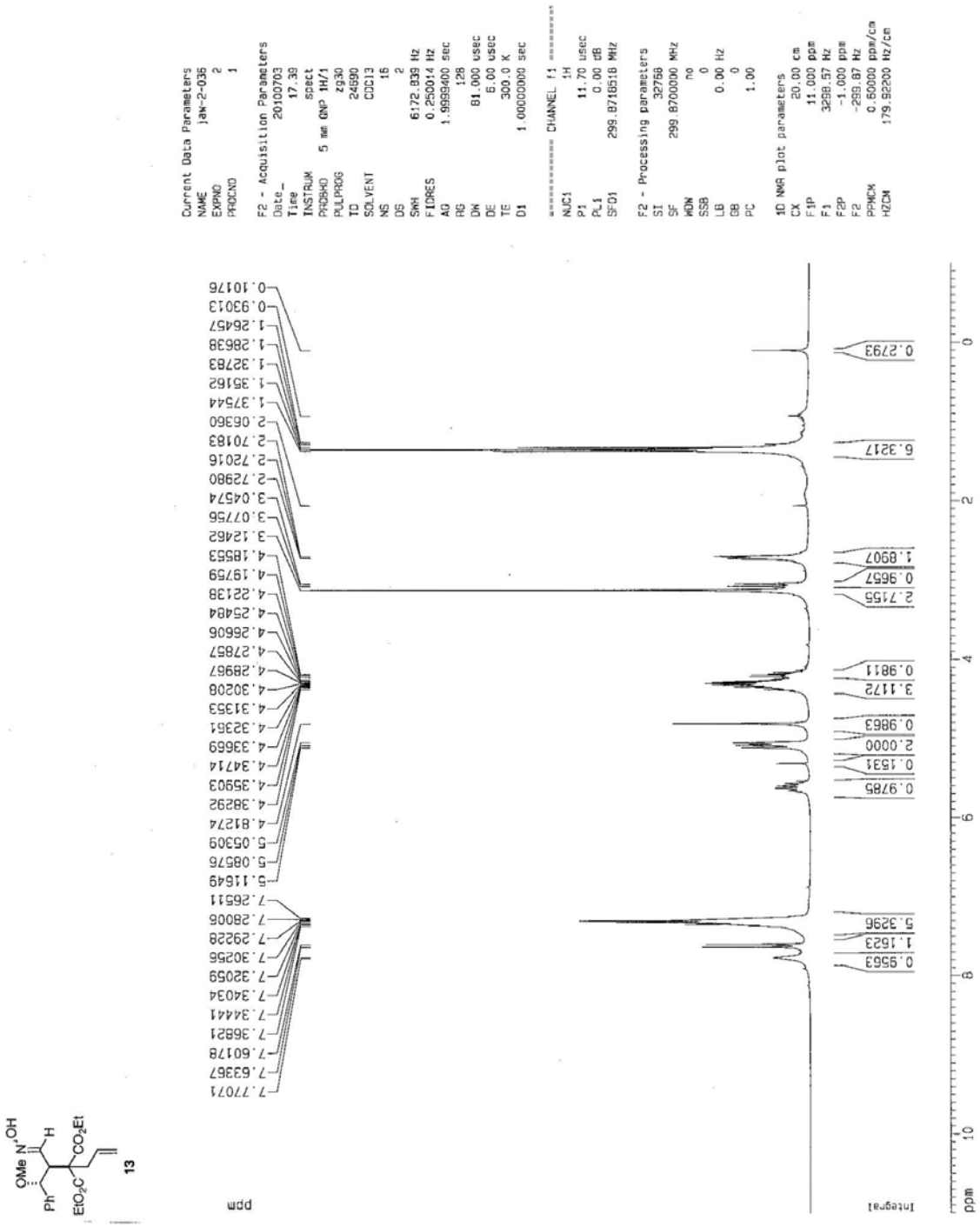

Me
N
OH
H
Ph
EtO2C
CO2Et
8
jaw-1-102
ppm
168.829
168.258
167.163
151.416
143.955
129.236
129.197
129.102
128.922
128.696
127.883
127.844
127.810
127.720
127.429
77.917
77.493
77.068
62.170
61.998
61.784
53.704
47.067
42.117
40.775
40.260
19.604
14.473
1.457
Current Data Parameters
NAME jaw-1-102
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091024
Time 21.56
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 1024
DS 4
SWH 18796.992 Hz
FIDRES 1.147277 Hz
AQ 0.4356644 sec
RG 32768
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46446 ppm/cm
HZCM 939.84957 Hz/cm
ppm
200
175
150
125
100
75
50
25
0

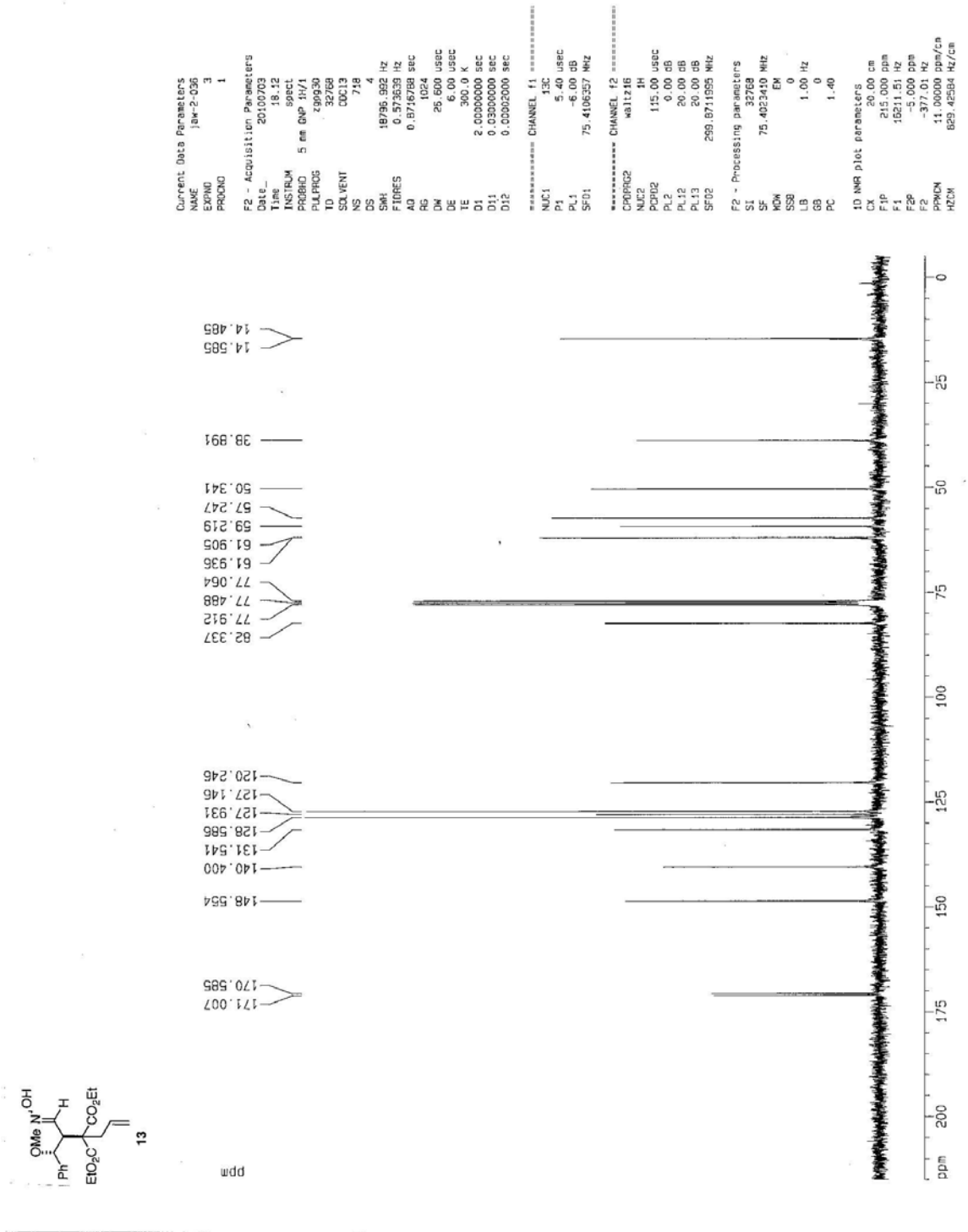

after column
16-20
17
Me
N
OH
H
CO2Et
EtO2C
Current Data Parameters
NAME jaw-2-082
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100907
Time 15.37
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 24680
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.66000 ppm/cm
HZCM 180.07800 Hz/cm
8.51393
7.59062
7.55938
7.28055
5.74808
5.72121
5.69252
5.11246
5.08490
5.05219
4.22301
4.21135
4.19946
4.18798
4.17510
4.16431
4.14509
4.13218
2.90365
2.89715
2.87256
2.86597
2.04440
1.33377
1.32373
1.31065
1.28690
1.27222
1.26314
1.24857
1.22480
1.14034
1.11460
0.89971
0.86712
0.83888
0.81599
0.09695
1.0194
1.0000
1.0824
2.2301
4.8788
1.0567
1.0968
1.1198
1.3553
0.7252
0.4094
0.6873
7.3350
1.1575
0.2477
9.8010
0.8456
3.1673
ppm
Integral

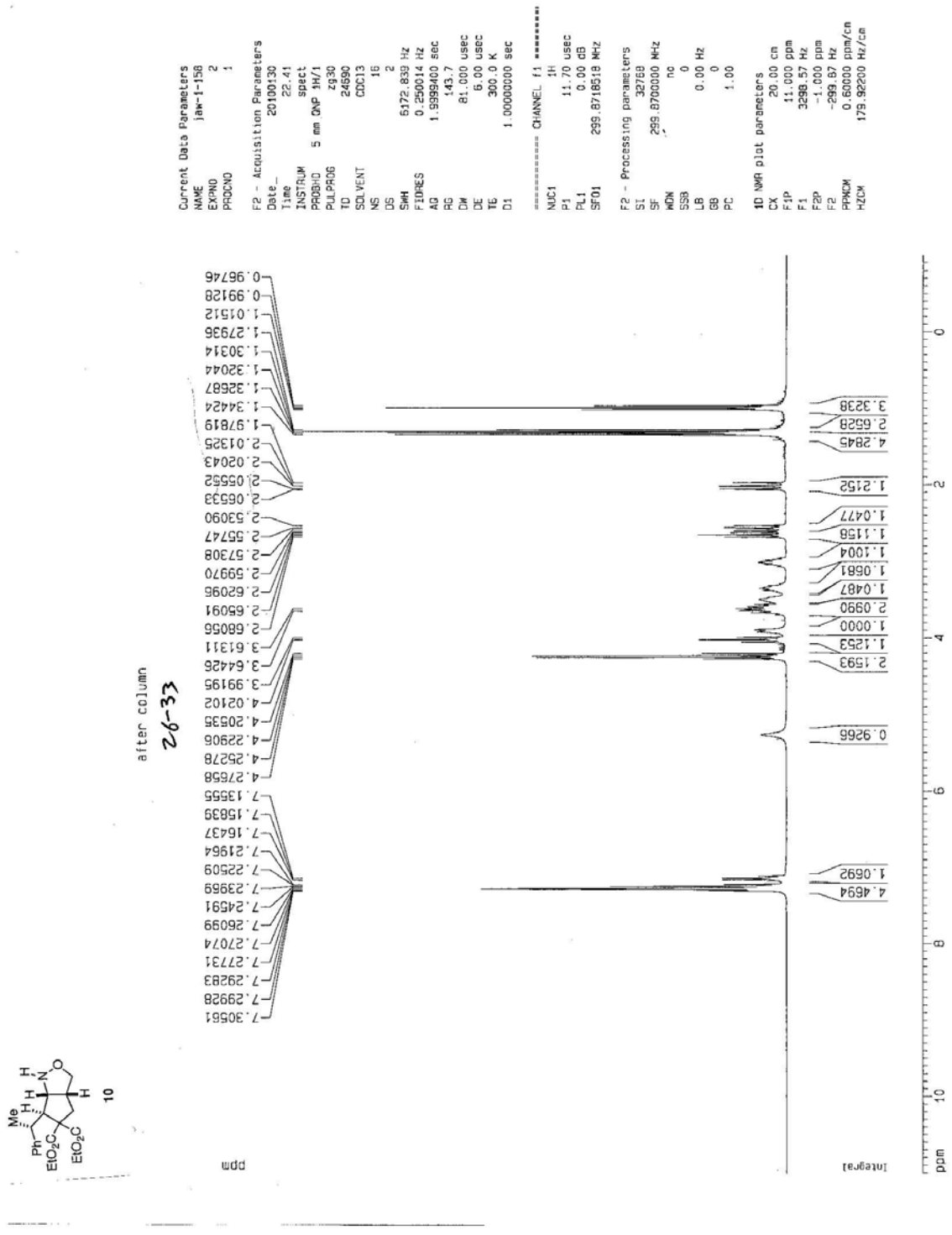

Current Data Parameters
NAME jaw-2-082
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100909
Time 21.52
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1441
DS 4
SWH 18796.992 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 4095
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46446 ppm/cm
HZCM 939.81967 Hz/cm
14.419
14.588
17.940
29.270
30.528
30.938
31.658
39.477
49.662
51.875
60.507
61.758
61.865
77.058
77.483
77.907
119.644
132.725
150.809
170.551
170.847
ppm
Me
N
OH
H
EtO2C
CO2Et
17

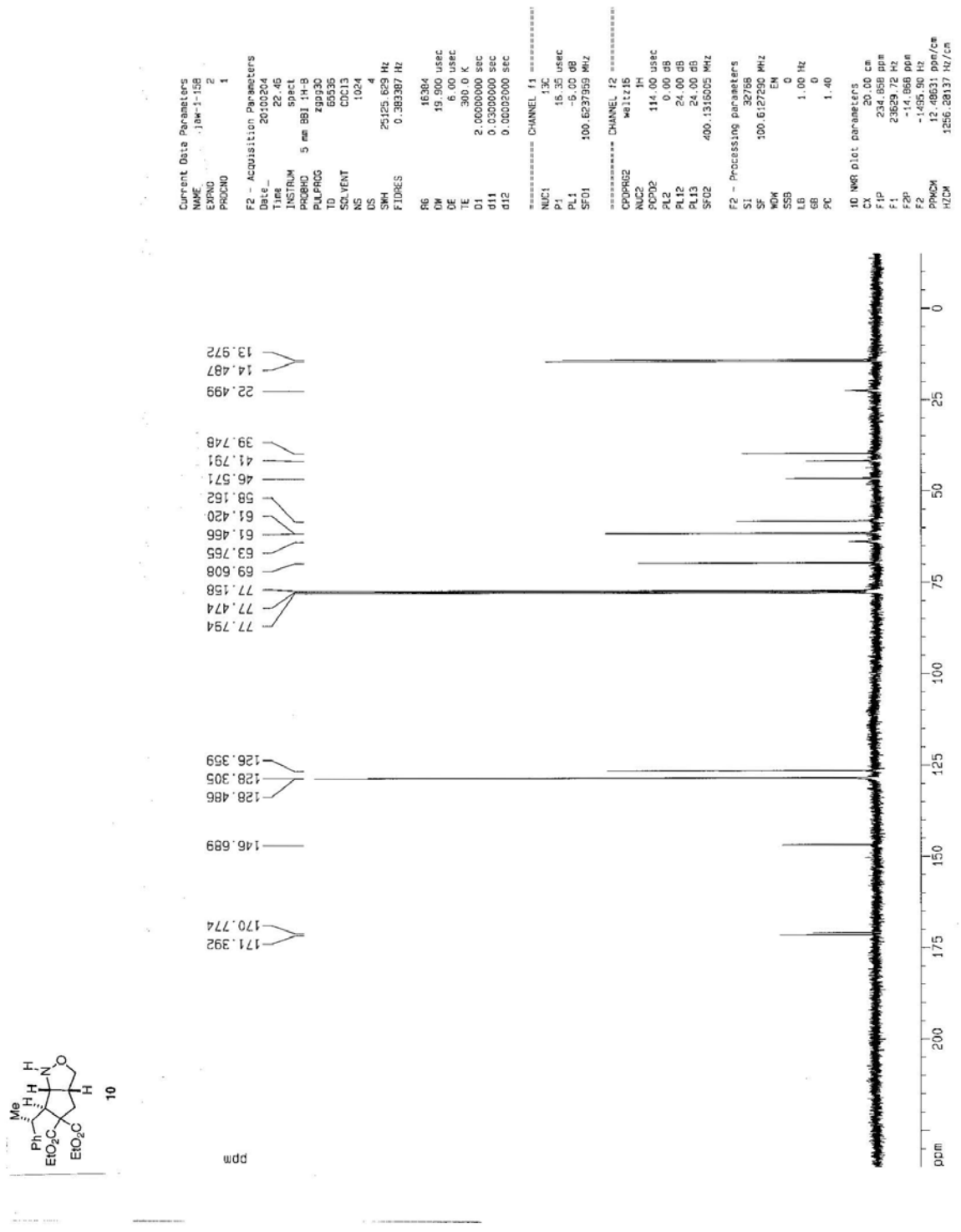

Me
N
OH
H
N(Me)Ts
20
jaw-2097 1 1 /opt/topspin Weinreb
12-17
General
DATE = 2010/09/15
TIME = 22:27
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
[rel]
[ppm]

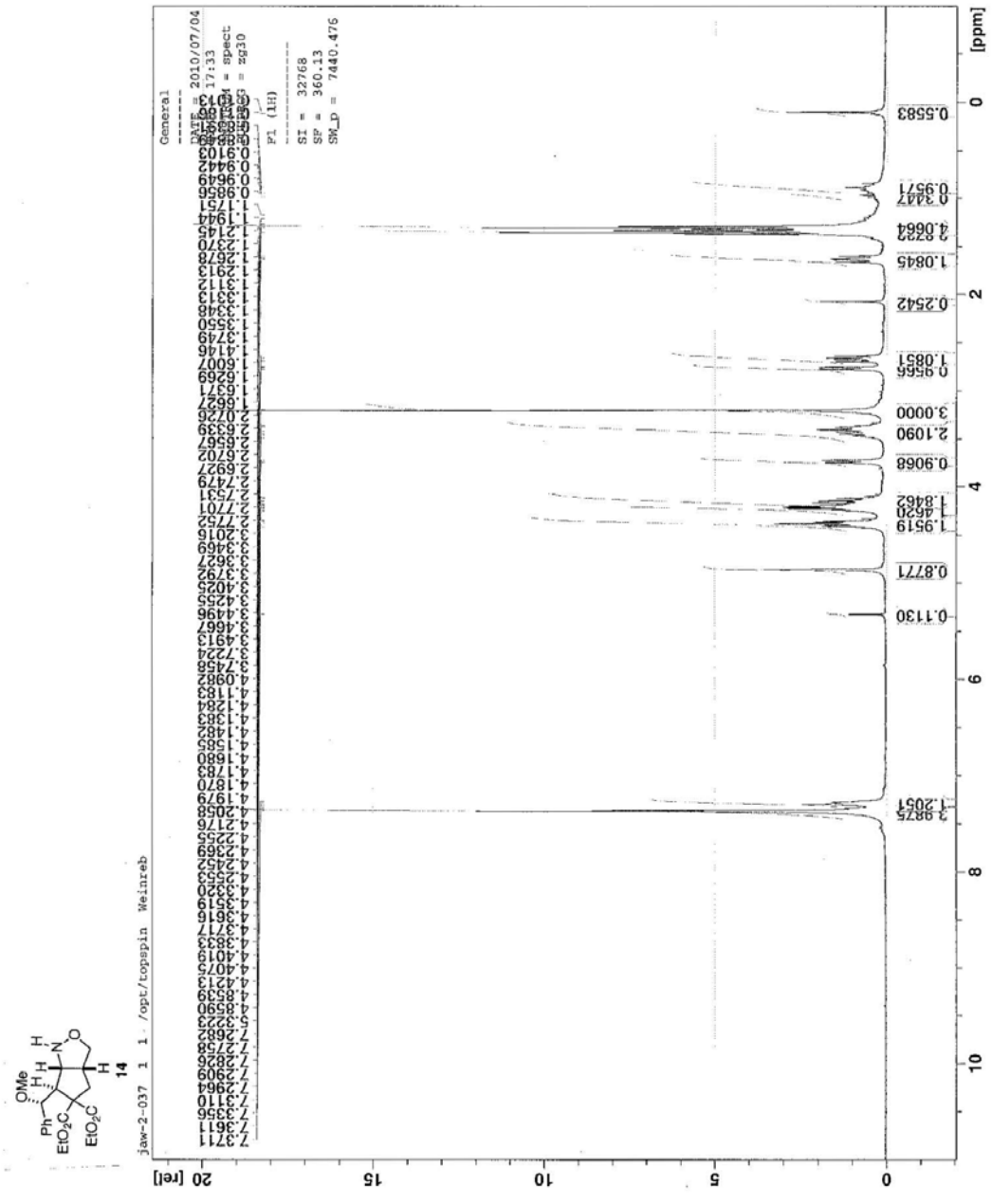

20
Me
OH
N
H
N(Me)Ts
jaw-2087 3 1 /opt/topspin Weinreb
12-17
General
DATE = 2010/09/16
TIME = 09:42
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
147.7892
143.3524
135.8916
129.4831
127.7005
127.6080
77.4159
77.2646
77.0624
76.7098
61.9448
46.3027
31.1242
30.9369
30.2813
30.2321
30.0339
29.9694
29.7891
21.3353
19.7176
[rel]
15
10
5
0
[ppm]
0
50
100
150
200

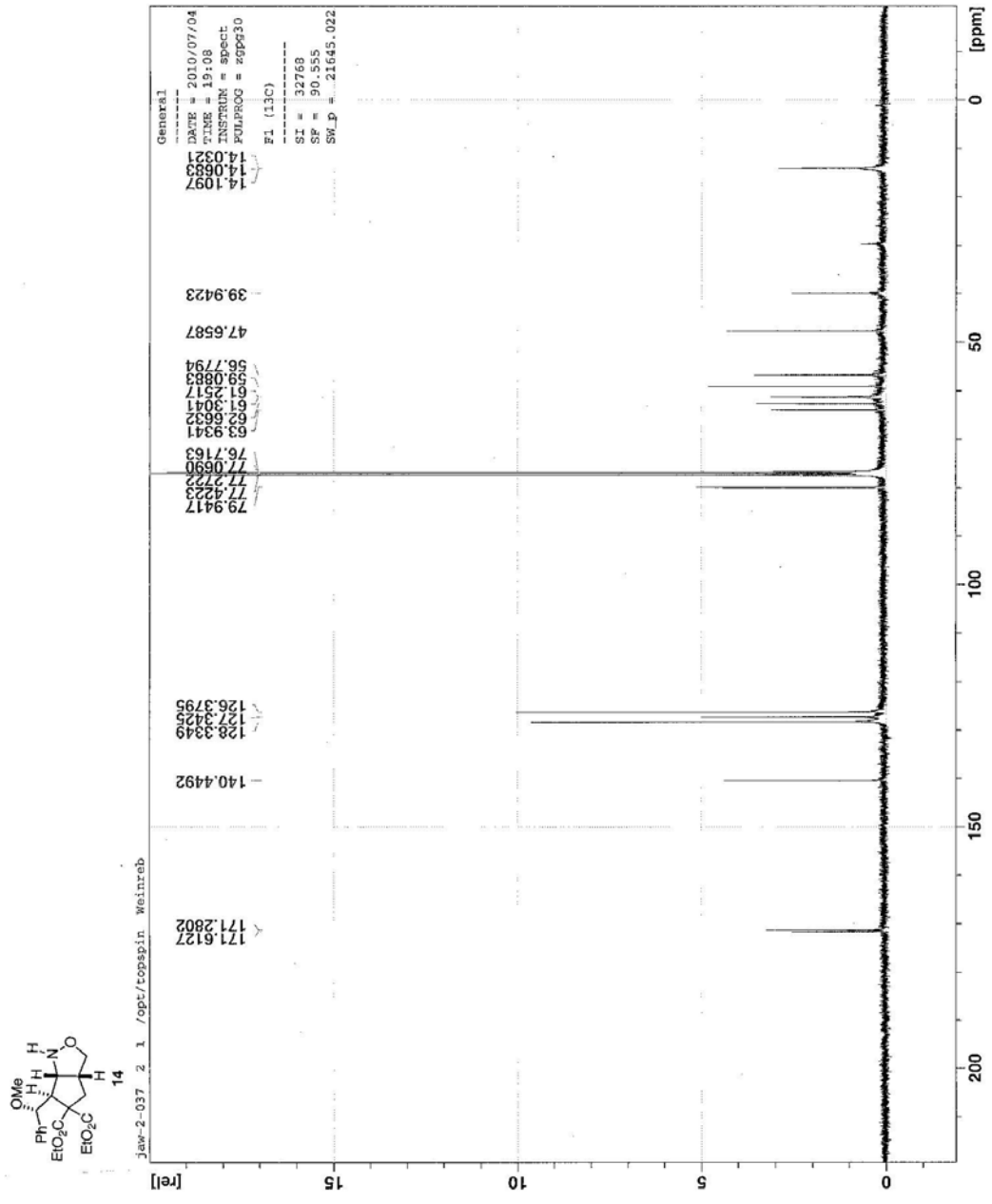

Current Data Parameters
NAME jan-2-036
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100703
Time 17.39
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8716516 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
7.77071
7.63367
7.60178
7.36821
7.34441
7.34034
7.32059
7.30256
7.29228
7.28006
7.26511
5.11649
5.08576
5.05309
4.81274
4.38292
4.35903
4.34714
4.33669
4.32361
4.31353
4.30208
4.28967
4.27857
4.26606
4.25484
4.22138
4.19759
4.18553
3.12462
3.07756
3.04574
2.72980
2.72016
2.70183
2.06360
1.37544
1.35162
1.32783
1.28638
1.26457
0.93013
0.10176
0.2793
6.3217
1.8907
0.9657
2.7155
0.9811
3.1172
0.9863
2.0000
0.1531
0.9785
5.3296
1.1623
0.9563
ppm
Integral
OMe
N
OH
H
Ph
EtO2C
CO2Et
13

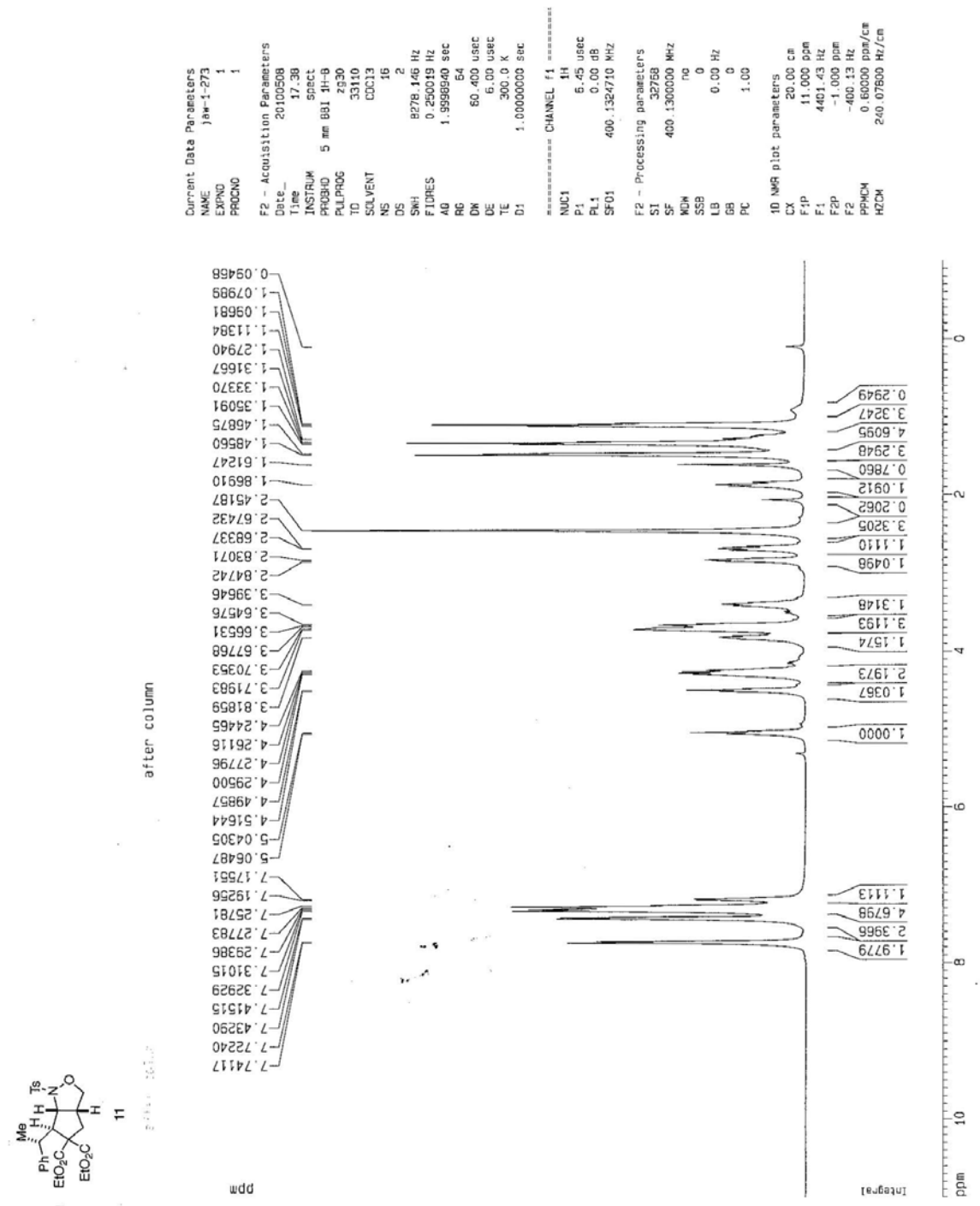

Current Data Parameters
NAME jaw-2-036
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100703
Time 18.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 718
DS 4
SWH 18796.992 Hz
FIDRES 0.573639 Hz
AQ 0.87167808 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16211.51 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42584 Hz/cm
14.485
14.585
38.891
50.341
57.247
59.219
61.905
61.936
77.064
77.488
77.912
82.337
120.246
127.146
127.931
128.586
131.541
140.400
148.554
170.585
171.007
ppm
200
175
150
125
100
75
50
25
0
OMe N OH
Ph
H
EtO2C
CO2Et
13

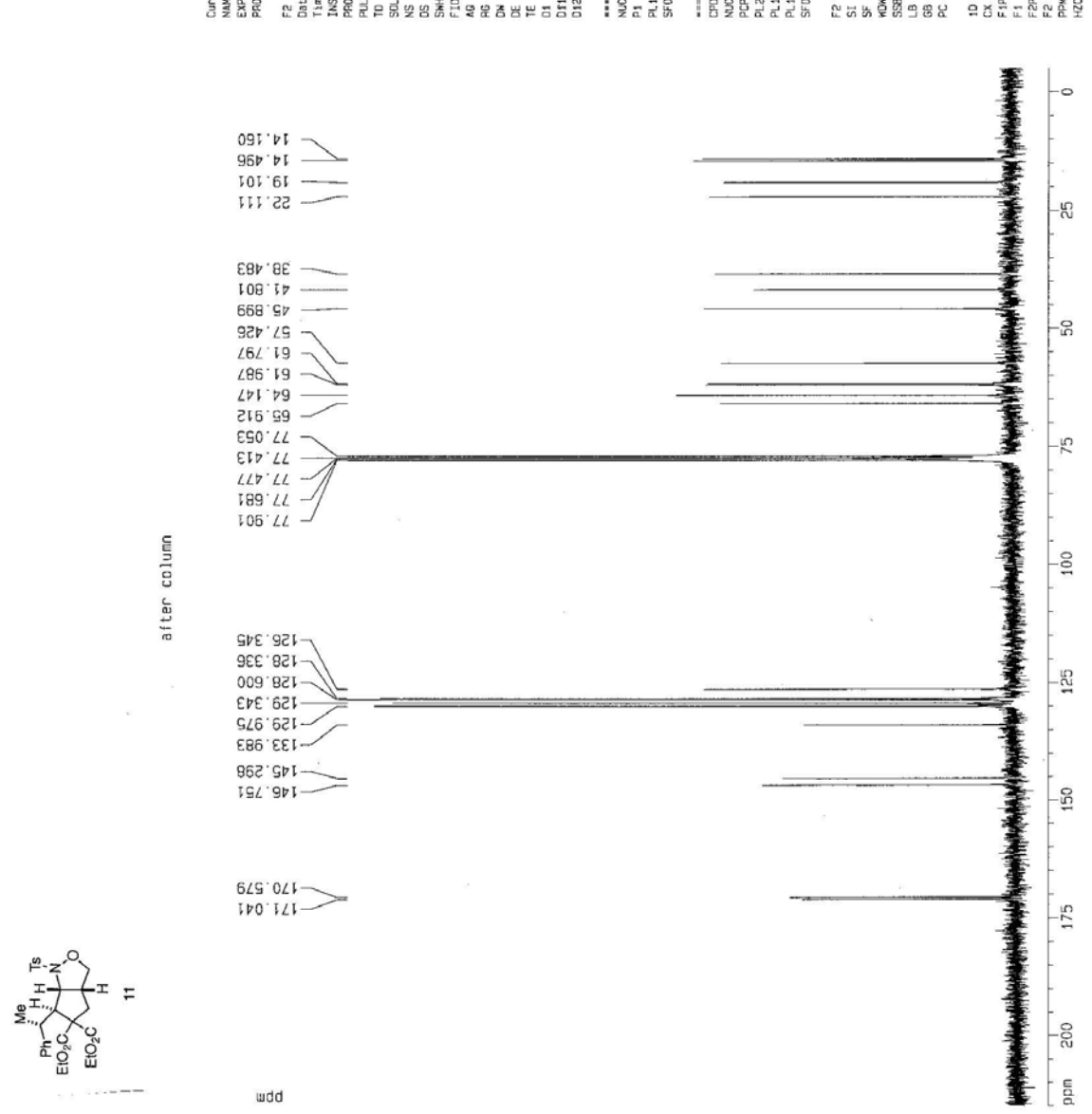

Current Data Parameters
NAME jaw-1-158
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100130
Time 22.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24590
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
after column
26-33
10
ppm
Integral
ppm

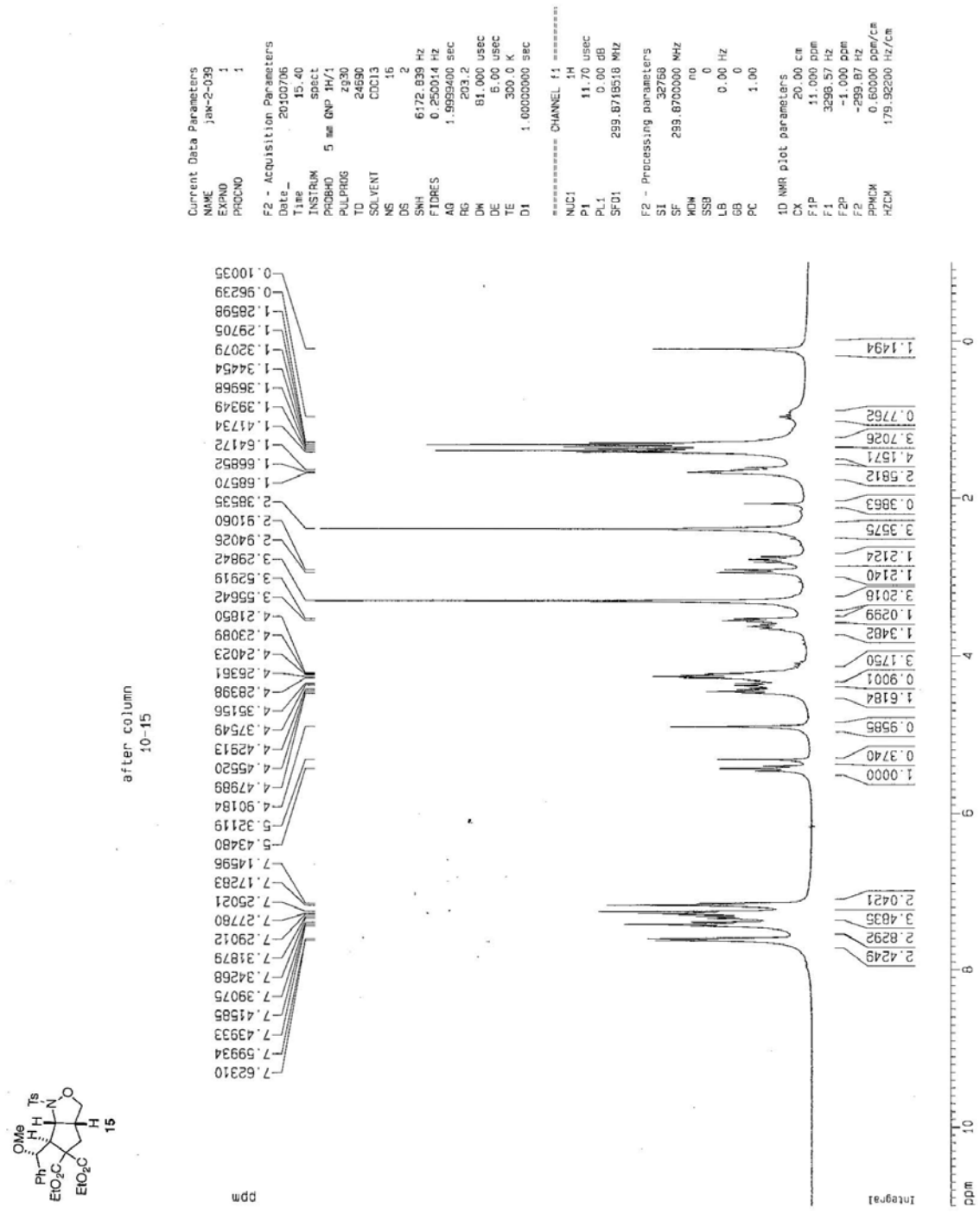

Current Data Parameters
NAME jaw-1-158
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100204
Time 22.46
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
RG 16384
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 16.35 usec
PL1 -5.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 114.00 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.858 ppm
F1 23629.72 Hz
F2P -14.866 ppm
F2 -1495.90 Hz
PPMCM 12.49631 ppm/cm
HZCM 1256.28137 Hz/cm
13.972
14.487
22.499
39.748
41.791
46.571
58.162
61.420
61.466
63.765
69.608
77.158
77.474
77.794
126.359
128.305
128.486
146.689
170.774
171.392
ppm
Me
Ph
EtO2C
EtO2C
10

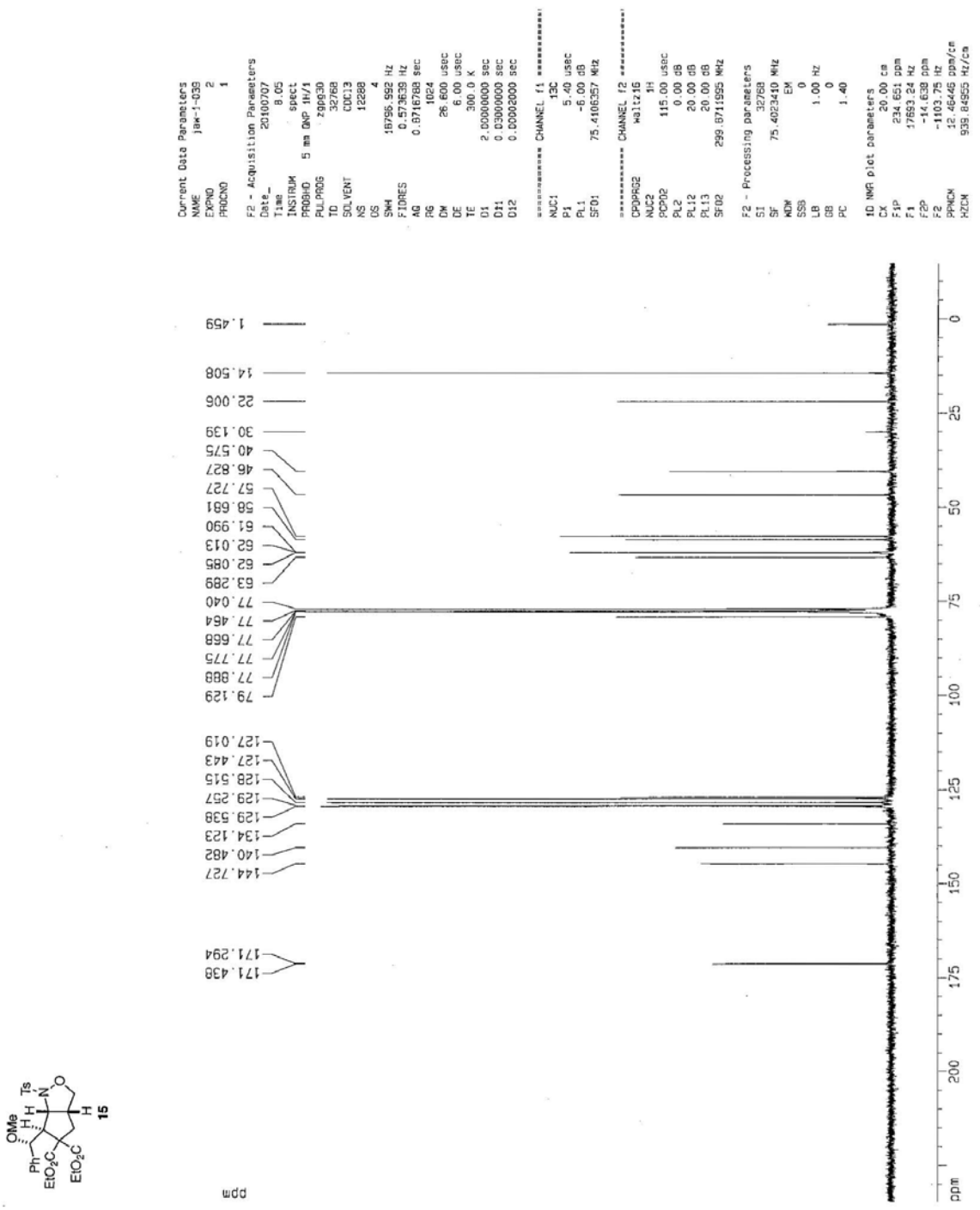

jaw-2-037 1 1 /opt/topspin Weinreb
14
OMe
Ph
EtO2C
EtO2C
General
SI = 32768
SP = 360.13
SW_p = 7440.476
[rel]
[ppm]
0.5583
0.9571
0.3447
4.0664
2.8229
1.0845
0.2542
1.0851
0.9556
3.0000
2.1090
0.9068
1.8462
1.4620
1.9519
0.8771
0.1130
3.9825
1.2051

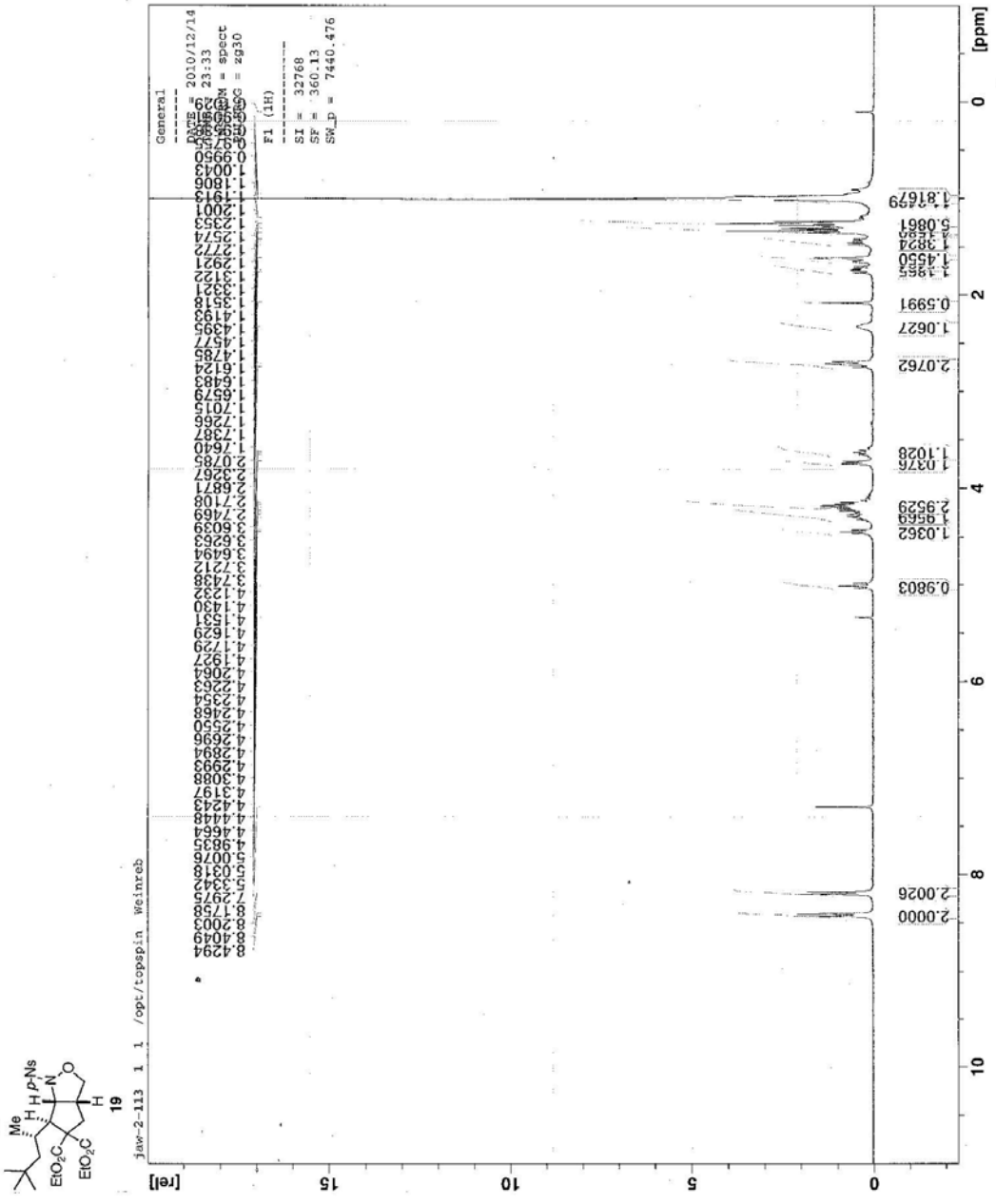

jaw-2-037 2 1 /opt/topspin Weinreb
14
OMe
Ph
EtO2C
EtO2C
[rel]
[ppm]
General
DATE = 2010/07/04
TIME = 19:08
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
171.6127
171.2802
140.4492
128.3349
127.3425
126.3795
79.9417
77.4223
77.2722
77.0690
76.7163
63.9341
62.6632
61.3041
61.2517
59.0883
56.7794
47.6587
39.9423
14.1097
14.0683
14.0321

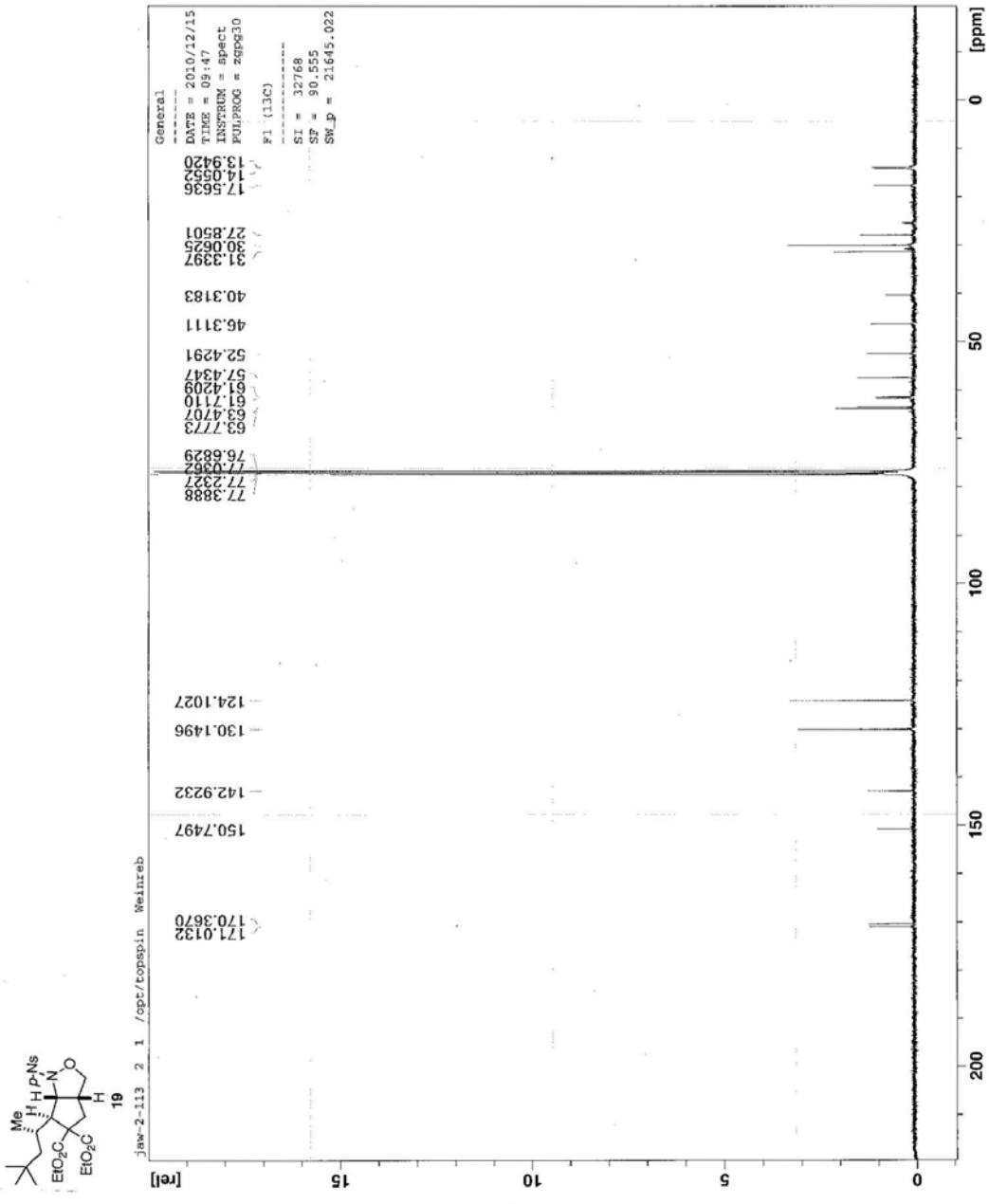

11
after column
Current Data Parameters
NAME jaw-1-273
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100508
Time 17.38
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 33110
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.250019 Hz
AQ 1.9998940 sec
RG 64
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SF01 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm
ppm
Integral

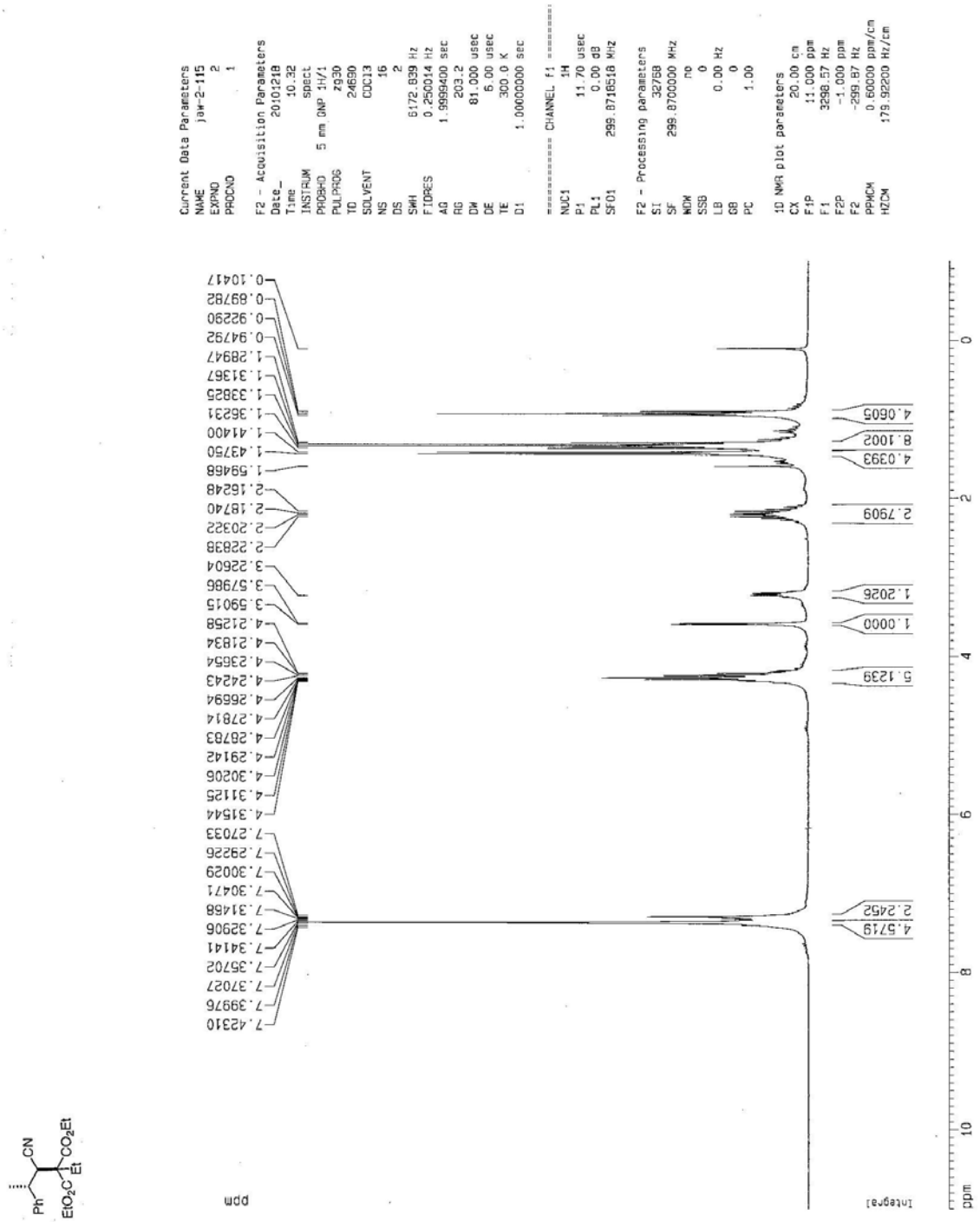

11
after column
ppm
Current Data Parameters
NAME jaw-1-273
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100509
Time 6.57
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 9216
DS 4
SWH 18796.992 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 16384
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 16211.50 Hz
F2P -5.000 ppm
F2 -377.01 Hz
PPMCM 11.00000 ppm/cm
HZCM 829.42578 Hz/cm
14.160
14.496
19.101
22.111
38.483
41.801
45.899
57.426
61.797
61.987
64.147
65.912
77.053
77.413
77.477
77.681
77.901
125.345
128.336
128.600
129.343
129.975
133.983
145.298
146.751
170.579
171.041
0
25
50
75
100
125
150
175
200

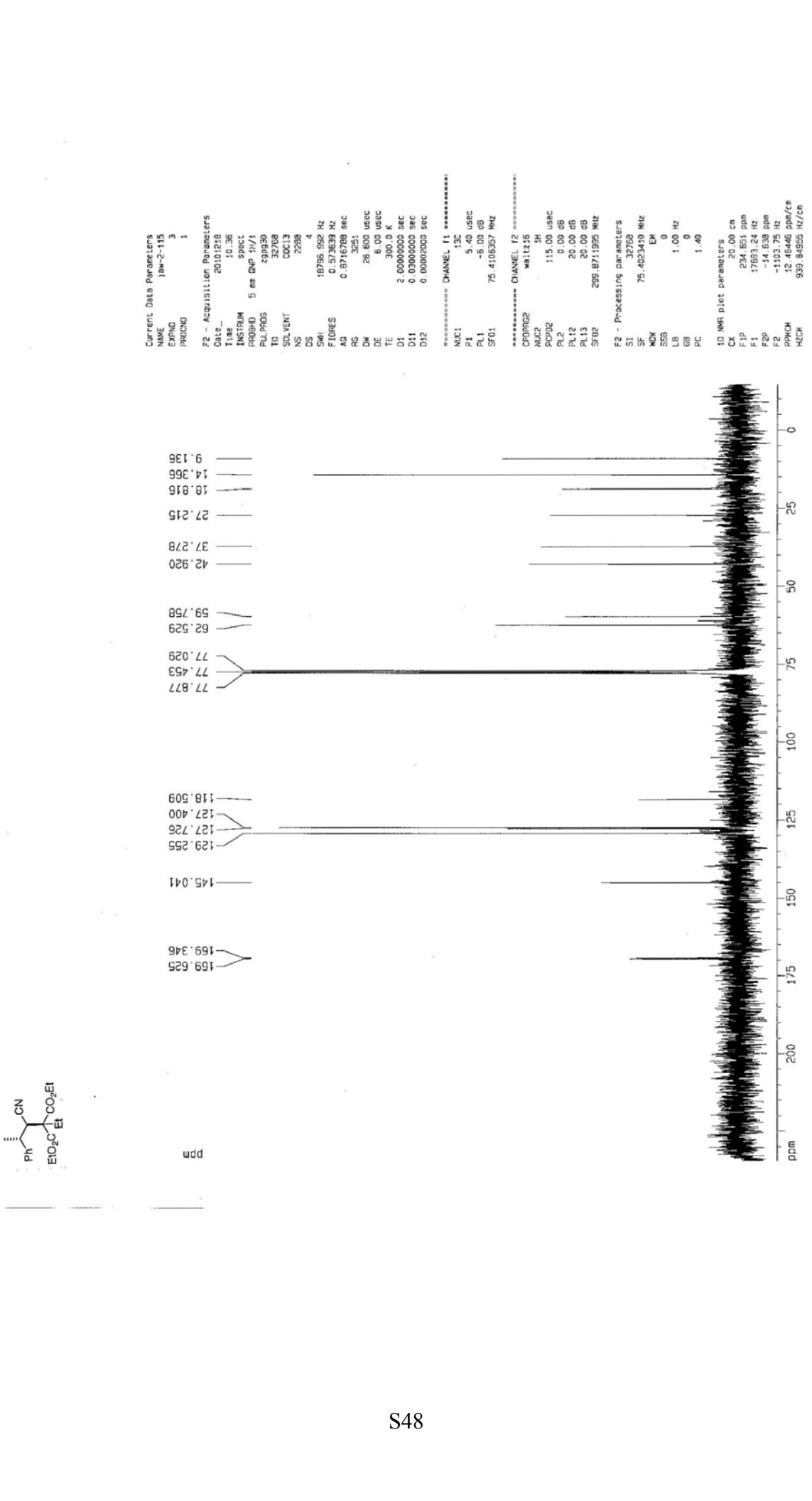

after column
10-15
15
Ts
OMe
Ph
EtO2C
EtO2C
Current Data Parameters
NAME jak-2-039
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100706
Time 15.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8716518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3296.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
7.62310
7.59934
7.43933
7.41585
7.39075
7.34268
7.31879
7.29012
7.27780
7.25021
7.17283
7.14596
5.43480
5.32119
4.90184
4.79889
4.45520
4.42913
4.37549
4.35156
4.28398
4.26361
4.24023
4.23089
4.21850
3.55642
3.52919
3.29842
2.94026
2.91060
2.38535
1.68570
1.66852
1.64172
1.41734
1.39349
1.36968
1.34454
1.32079
1.29705
1.28598
0.96239
0.10035
1.1494
0.7762
3.7026
4.1571
2.5812
0.3863
3.3575
1.2124
1.2140
3.2018
1.0299
1.3482
3.1750
0.9001
1.6184
0.9585
0.3740
1.0000
2.0421
3.4835
2.8292
2.4249
Integral
ppm

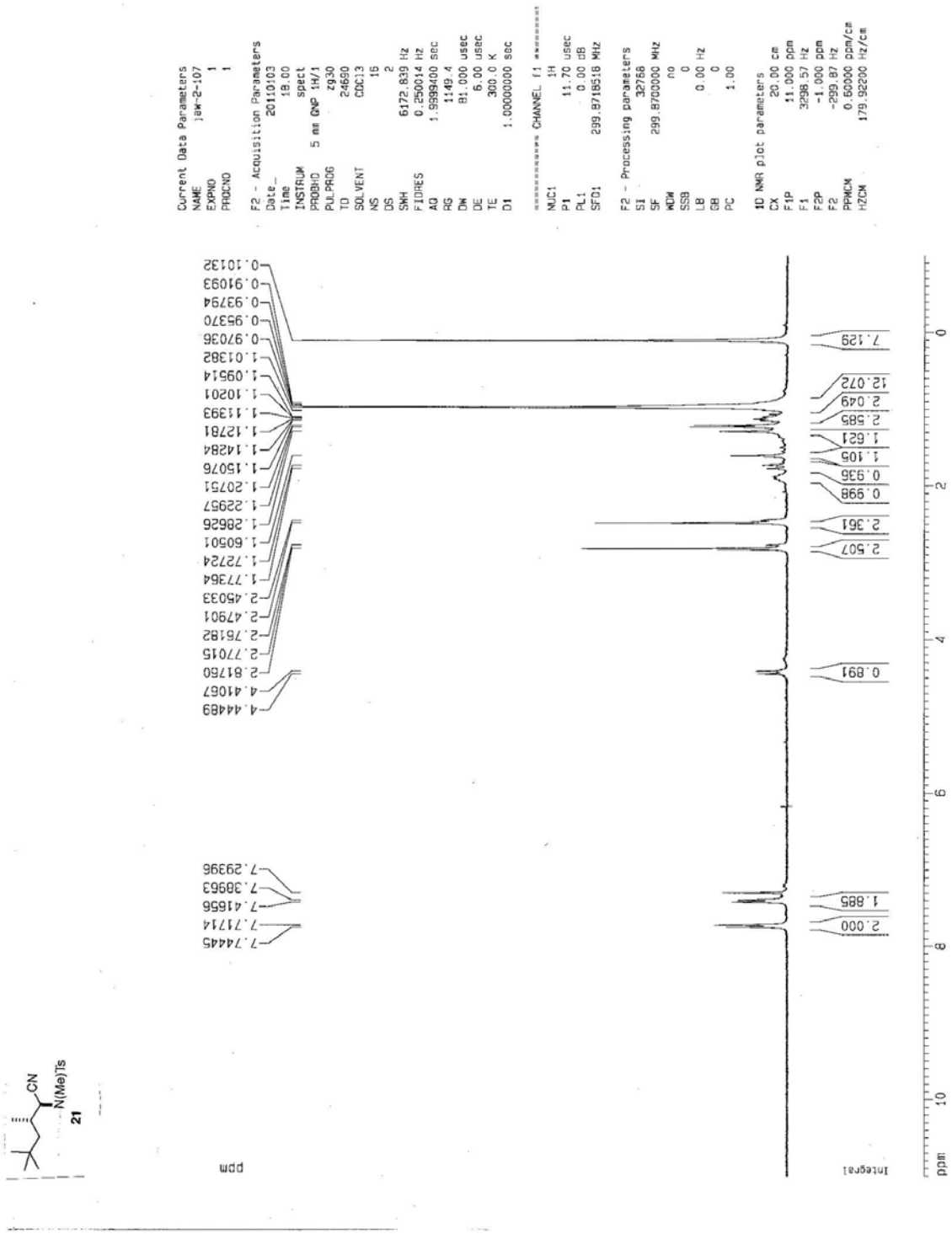

Current Data Parameters
NAME jaw-1-039
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100707
Time 8.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1228
DS 4
SWH 18796.992 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46446 ppm/cm
HZCM 939.84955 Hz/cm
1.459
14.508
22.006
30.139
40.575
46.827
57.727
58.681
61.990
62.013
62.085
63.289
77.040
77.464
77.668
77.775
77.888
79.129
127.019
127.443
128.515
129.257
129.538
134.123
140.482
144.727
171.294
171.438
ppm
OMe
H
Ts
N
O
H
Ph
EtO2C
EtO2C
15

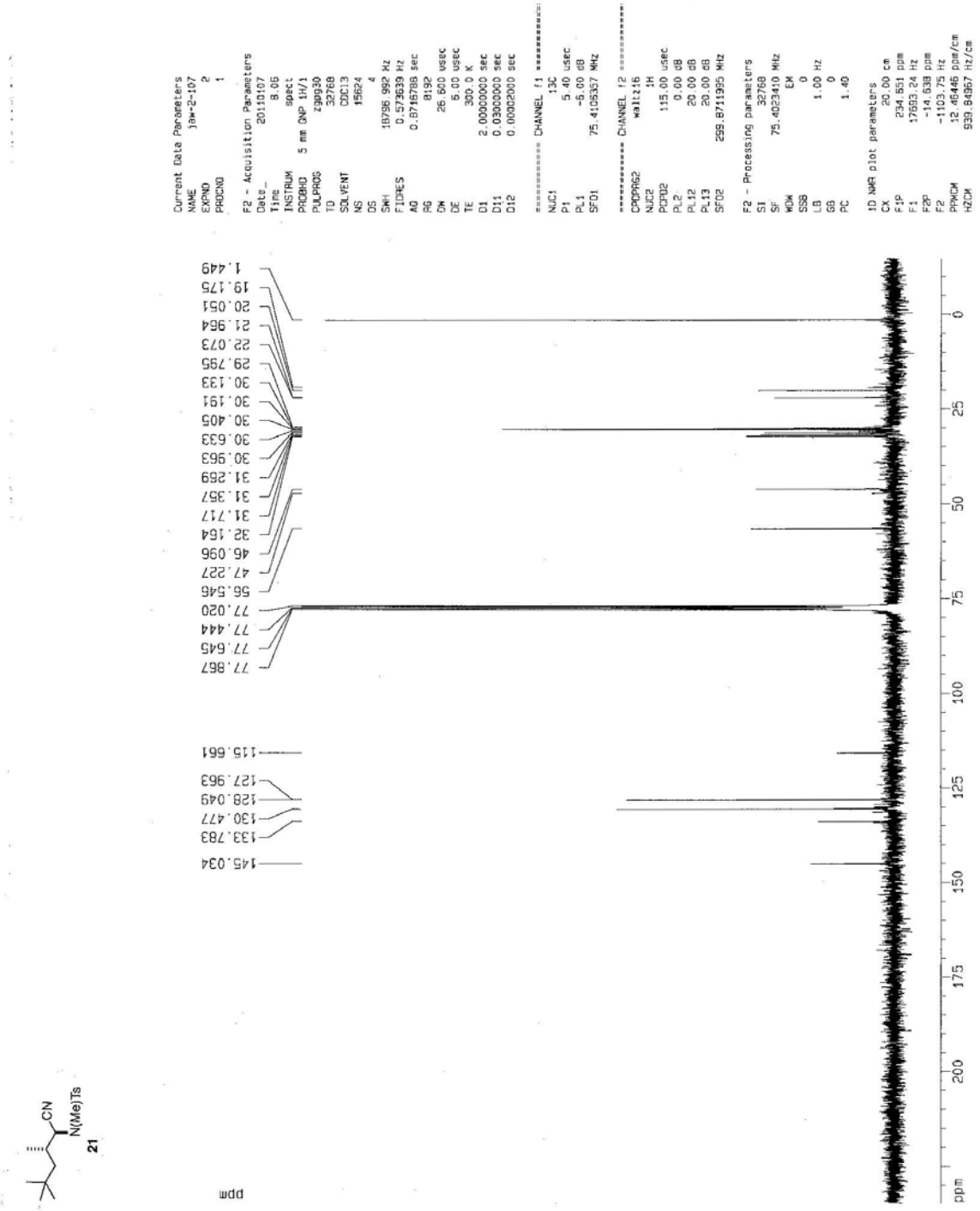

19
Me
H
p-Ns
N
O
H
EtO2C
EtO2C
jaw-2-113 1 1 /opt/topspin Weinreb
General
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
[rel]
[ppm]
1.81679
5.08611
1.45501
0.59911
1.06271
2.07621
1.10281
2.95291
0.98031
2.00261
2.00000


jaw-2-113 2 1 /opt/topspin Weinreb
General
DATE = 2010/12/15
TIME = 09:47
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
171.0132
170.3670
150.7497
142.9232
130.1496
124.1027
77.3888
77.2327
77.0362
76.6829
63.7773
63.4707
61.7110
61.4209
57.4347
52.4291
46.3111
40.3183
31.3397
30.0625
27.8501
17.5636
14.0552
13.9420
[rel]
15
10
5
0
[ppm]
200
150
100
50
0
Me
H
H
PNs
N
O
H
19
EtO2C
EtO2C


Current Data Parameters
NAME jaw-2-115
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101218
Time 10.32
INSTRUM spect
PROBHD 5 mm DNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SF01 299.8716518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
7.42310
7.39976
7.37027
7.35702
7.34141
7.32906
7.31468
7.30471
7.30029
7.29226
7.27033
4.31544
4.31125
4.30206
4.29142
4.28783
4.27814
4.26694
4.24243
4.23654
4.21834
4.21258
3.59015
3.57986
3.22604
2.22838
2.20322
2.18740
2.16248
1.59468
1.43750
1.41400
1.36231
1.33825
1.31367
1.28947
0.94792
0.92290
0.89782
0.10417
4.0605
8.1002
4.0393
2.7909
1.2026
1.0000
5.1239
2.2452
4.5719
ppm
Integral
Ph
CN
Et
CO2Et
EtO2C


Ph
CN
EtO2C
CO2Et
Et
ppm
9.136
14.366
18.816
27.215
37.278
42.920
59.758
62.529
77.029
77.453
77.877
118.509
127.400
127.726
129.255
145.041
159.625
159.346
Current Data Parameters
NAME jaw-2-115
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101218
Time 10.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2288
DS 4
SWH 18796.992 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 3251
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.851 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46446 ppm/cm
HZCM 939.84855 Hz/cm


Current Data Parameters
NAME jaw-2-107
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110103
Time 19.00
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 24690
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.250014 Hz
AQ 1.9999400 sec
RG 1149.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718516 MHz
F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3298.57 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.60000 ppm/cm
HZCM 179.92200 Hz/cm
0.10132
0.91093
0.93794
0.95370
0.97036
1.01382
1.09514
1.10201
1.11393
1.12781
1.14284
1.15076
1.20751
1.22957
1.28626
1.60501
1.72724
1.77364
2.45033
2.47901
2.76182
2.77015
2.81760
4.41067
4.44489
7.29396
7.38963
7.41656
7.71714
7.74445
7.129
12.072
2.049
2.585
1.621
1.105
0.936
0.998
2.361
2.507
0.891
1.885
2.000
Integral
ppm
CN
N(Me)Ts
21


CN
N(Me)Ts
21
ppm
1.449
19.175
20.051
21.964
22.073
29.795
30.133
30.191
30.405
30.633
30.963
31.259
31.357
31.717
32.164
46.096
47.227
56.546
77.020
77.444
77.645
77.867
115.661
127.963
128.049
130.477
133.783
145.034
Current Data Parameters
NAME jax-2-107
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110107
Time 8.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 15624
DS 4
SWH 16796.992 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 8192
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -5.00 dB
SFO1 75.4109357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.638 ppm
F2 -1103.75 Hz
PPMCM 12.46446 ppm/cm
HZCM 939.84967 Hz/cm
0
25
50
75
100
125
150
175
200
ppm