Supporting Information

Diastereocontrolled Construction of Pactamycin's Complex Ureido Triol Functional Array

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Table of Contents

Methods: General. Infrared (IR) spectra were obtained using a Jasco 460 Plus Fourier transform infrared spectrometer. Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on a Bruker model Avance 400 (^{1}H NMR at 400 MHz and ^{13}C at 100 MHz), Bruker model Avance 500 (^{1}H NMR at 500 MHz and ^{13}C NMR at 125 MHz), or a Bruker Avance III 600 (^{1}H NMR at 600 MHz and ^{13}C NMR at 150 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, br d = broad doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Mass spectra were obtained using a Bruker BioTOF II spectrometer with electrospray ionization calibrated with CsOAc or an Agilent Technologies 6520, Accurate – Mass QTOF LCMS, 1200 series LC with dual spray ESI source. All samples were prepared in methanol. Analytical thin layer chromatography (TLC) was performed on Sorbent Technologies 0.20 mm Silica G TLC plates. Visualization was accomplished with UV light, $KMnO_4$, and/or aqueous ceric ammonium nitrate solution followed by heating. Purification of the reaction products was carried out by flash chromatography using Siliaflash-P60 silica gel (40-63μm) purchased from Silicycle. Supercritical fluid chromatography was performed on a Berger SFC system equipped with a Chiralcel OD column. Samples were eluted with SFC grade CO_2 at the indicated percentage of MeOH. Unless otherwise noted, all reactions were carried out under an atmosphere of dry nitrogen in oven-dried glassware with magnetic stirring. Yield refers to isolated yield of analytically pure material unless otherwise noted. Yields are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments.

Materials: General. Tetrahydrofuran, diethyl ether, dichloromethane, and toluene were dried by passage through a column of neutral alumina under nitrogen prior to use. Cerium trichloride was dried under high-vacuum at 60 °C for 2 h, 80 °C for 2 h, and 140 °C for 12 h prior to storage in a nitrogen-filled glove box.[1] Triethylamine was freshly distilled from calcium hydride prior to use. Pentane was freshly distilled from sodium hydride prior to use. Methyl diazoacetoacetate (**7**) was prepared by a known procedure.[2] 2-bromopropenol (**15**) was prepared via the procedure of Corey.[3]

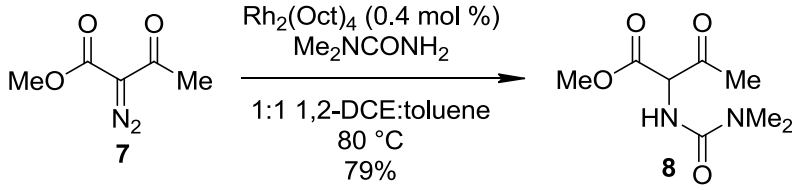
All other reagents were purchased from commercial sources and were used as received unless otherwise noted.

[1] For $CeCl_3$ purification methods see: (a) Dimitrov, V.; Kostova, K.; Genov, M. *Tetrahedron Lett.* **1996**, *37*, 6787. (b) Imamoto, T.; Kusumoto, T.; Tawarayama, Y.; Sugiura, Y.; Mita, T.; Hatanaka, Y.; Yokoyama, M. *J. Org. Chem.* **1984**, *49*, 3904.

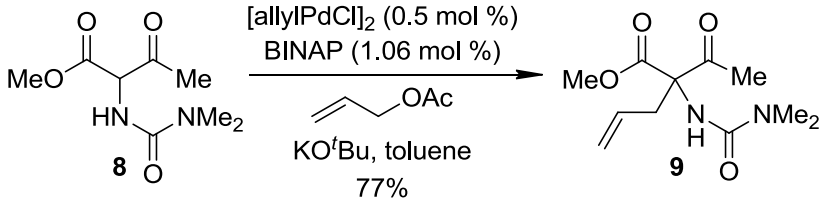
[2] Greszler, S. N.; Johnson, J. S. *Angew. Chem. Int. Ed*. **2009**, *48*, 3689.

[3] Snyder, S. A.; Corey, E. J. *J. Am. Chem. Soc.* **2006**, *128*, 740–742.

Experimental Procedures:

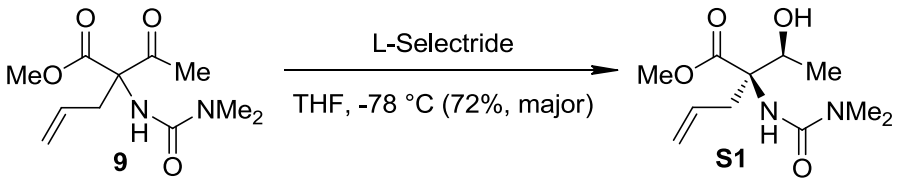


Methyl 2-(3,3-dimethylureido)-3-oxobutanoate (8): A 1-L round-bottomed flask was charged with methyl diazoacetoacetate **7** (13.17 g, 92.0 mmol, 1.00 equiv) and finely ground 1,1-dimethylurea (12.16 g, 138 mmol, 1.50 equiv). Toluene (275 mL) and 1,2-dichlorethane (275 mL) were added and the suspension was heated to 80 °C in a sand bath with magnetic stirring. The solution gradually became homogenous upon heating. Four portions of $Rh_2(Oct)_4$ (0.071 g, 0.001 mmol, 0.001 equiv, each portion) suspended in toluene were added over 30 min. The reaction was allowed to stir until consumption of starting material was indicated by TLC analysis, typically 1 h. The reaction was allowed to cool to rt; excess 1,1-dimethylurea precipitated upon cooling. The urea was removed via filtration (cotton) and the filtrate was concentrated *in vacuo*. The product was purified via flash chromatography (70:30 to 60:40 petroleum ether/acetone) to give the desired product as a yellow solid (14.78 g, 79%). Analytical data: **mp** 61-64 °C; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 5.59 (s, 1H), 5.17 (d, J = 6.0 Hz, 1H), 3.78 (s, 3H), 2.92 (s, 6H), 2.35 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 199.8, 167.6, 156.8, 64.3, 53.1, 36.1, 28.0; **HRMS (ESI$^+$)** Calcd. for $C_8H_{14}N_2O_4$+Na, 225.0852; Found, 225.0844; **IR** (thin film, cm^{-1}) 3431, 2955, 1751, 1647, 1522, 1382, 1270, 1206; **TLC** (70:30 petroleum ether/acetone): R_f = 0.20.



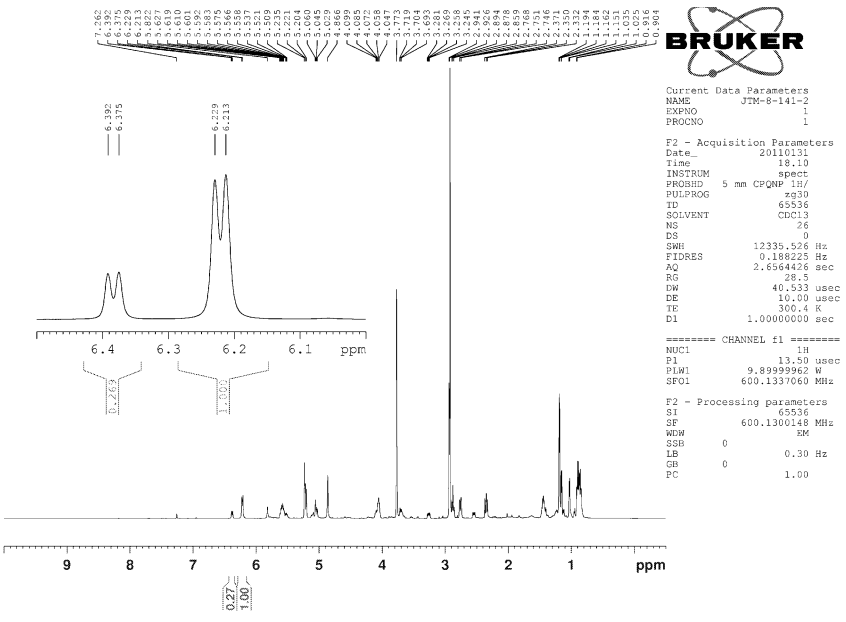
Methyl 2-acetyl-2-(3,3-dimethylureido)pent-4-enoate (9): In a nitrogen-filled glove box a flame-dried 100-mL round-bottomed flask was charged with allylpalladium chloride dimer (0.120 g, 0.328 mmol, 0.005 equiv) and *rac*-BINAP (0.432 g, 0.695 mmol, 0.0106 equiv). Toluene (20 mL) was added and the suspension was stirred for 10 min, capped with a rubber septum, and removed from the glove box. Allyl acetate (7.85 mL, 72.13 mmol, 1.10 equiv) was added and the catalyst solution was stirred for an additional 10 min. A separate flame-dried 1000-mL round-bottomed flask was charged with β-keto ester **8** (13.26 g, 65.57 mmol, 1.00 equiv) and KO^{t}Bu (7.72 g, 68.84 mmol, 1.05 equiv). Toluene (360 mL) was added and the suspension was stirred under a nitrogen atmosphere. The catalyst solution was introduced via cannula transfer, and the reaction was stirred for 12 h. The reaction was quenched with 1 M HCl (200 mL) and extracted with EtOAc (2 x 100 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (70:30 petroleum ether/acetone) to give the desired product as a pale

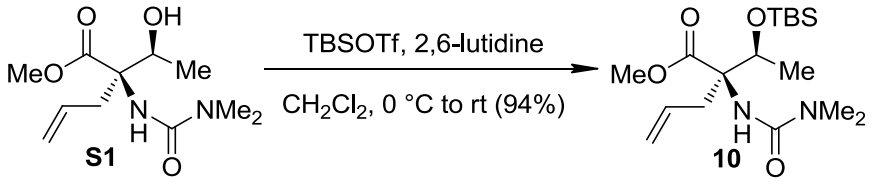
yellow oil (12.27 g, 77%). Analytical data: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 5.95 (s, 1H), 5.54-5.48 (m, 1H), 5.10-5.06 (m, 2H), 3.75 (s, 3H), 3.15 (dd, *J* = 14.4, 6.6 Hz, 1H), 2.99 (dd, *J* = 14.4, 7.8 Hz, 1H), 2.90 (s, 6H), 2.16 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 200.1, 169.5, 156.0, 131.6, 119.3, 72.0, 53.3, 36.9, 36.0, 24.7; **HRMS (ESI$^+$)** Calcd. for $C_{11}H_{18}N_2O_4$+Cs, 375.0321; Found, 375.0319; **IR** (thin film, cm^{-1}) 3429, 2953, 1726, 1653, 1517, 1368, 1280, 1226; **TLC** (70:30 petroleum ether/acetone): R_f = 0.30.



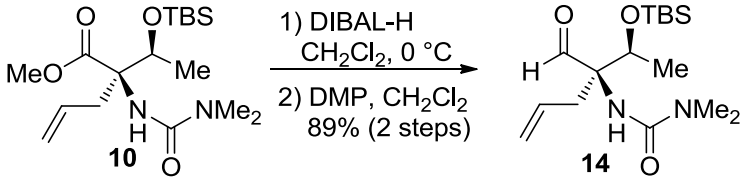
Methyl 2-(3,3-dimethylureido)-2-(1-hydroxyethyl)pent-4-enoate (S1): A flame-dried 250-mL round-bottomed flask was charged with ketone **9** (3.56 g, 14.71 mmol, 1.00 equiv) and THF (60 mL). The solution was cooled to -78 °C, and L-Selectride® (1 M in THF, 22.07 mL, 22.07 mmol, 1.50 equiv) was added dropwise. The reaction was stirred under a nitrogen atmosphere until consumption of starting material was indicated by TLC analysis, typically 4 h. The reaction was quenched by the sequential addition (5 mL) of H_2O, EtOH, 1 M NaOH, and 30% H_2O_2 and allowed to warm to rt. Saturated aqueous $Na_2S_2O_4$ (30 mL) was added and the solution was then extracted with EtOAc (3 x 20 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (80:20 to 60:40 petroleum ether/acetone) to give the product diastereomers as pale yellow solids (major, 2.59 g, 72%, minor, 0.737 g, 21%). Analytical data: **mp** 71-74 °C; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.18 (d, *J* = 10.2 Hz, 1H), 5.60-5.57 (m, 1H), 5.23-5.20 (m, 2H), 4.86 (s, 1H), 4.06-4.03 (m, 1H), 3.78 (s, 3H), 2.92 (s, 6H), 2.75 (d, *J* = 13.2, 1H), 2.34 (t, *J* = 12.6, 1H), 1.18 (d, *J* = 6.0, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 172.8, 158.7, 132.5, 120.8, 71.4, 68.1, 52.6, 40.4, 36.6, 18.1; **HRMS (ESI$^+$)** Calcd. for $C_{11}H_{20}N_2O_4$+Cs, 377.0477; Found, 377.0464; **IR** (thin film, cm^{-1}) 3421, 2980, 1753, 1636, 1524, 1457, 1377, 1220, 1120; **TLC** (70:30 petroleum ether/acetone): R_f = 0.20.

Crude ^{1}H NMR Spectrum of S1:





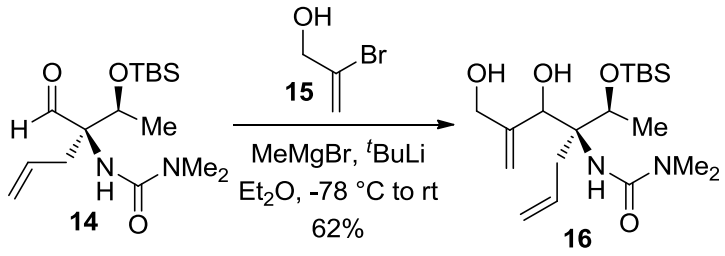
Methyl 2-(1-((*tert*-butyldimethylsilyl)oxy)ethyl)-2-(3,3-dimethylureido)pent-4-enoate (10): A flame-dried 500-mL round-bottomed flask was charged with alcohol **S1** (8.50 g, 35.12 mmol, 1.00 equiv) and CH_2Cl_2 (175 mL). 2,6-lutidine (12.10 mL, 105 mmol, 3.00 equiv) was added and the solution was cooled 0 °C. TBSOTf (16.11 mL, 70.24 mmol, 2.00 equiv) was added dropwise and the reaction was allowed to slowly warm to rt over 12 h by allowing the ice-water bath to expire. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (100 mL). The solution was extracted with Et_2O, washed with 1 M HCl and brine. The organic extracts were dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (95:5 to 90:10 petroleum ether/acetone) to give the desired product as a pale yellow oil (11.06 g, 88%). Analytical data: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 5.84-5.76 (m, 1H), 5.31 (s, 1H), 4.98-4.95 (m, 2H), 4.28 (q, *J* = 6.0, 1H), 3.70 (s, 3H), 3.01 (dd, *J* = 13.8, 6.0 Hz, 1H), 2.86 (s, 6H), 2.73 (dd, *J* = 13.8, 7.8 Hz, 1H), 1.10 (d, *J* = 6.0, 3H), 0.85 (s, 9H), 0.04 (s, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 173.4, 157.1, 134.5, 177.2, 72.0, 67.2, 52.3, 36.1, 35.3, 25.58, 18.75, 17.72, -4.1, -5.1; **HRMS (ESI$^+$)** Calcd. for $C_{17}H_{34}N_2O_4Si$+Cs, 491.1342; Found, 491.1341; **IR** (thin film, cm^{-1}) 3437, 2954, 2857, 1739, 1655, 1509, 1380, 1264, 1128, 834, 739; **TLC** (70:30 petroleum ether/acetone): R_f = 0.60.



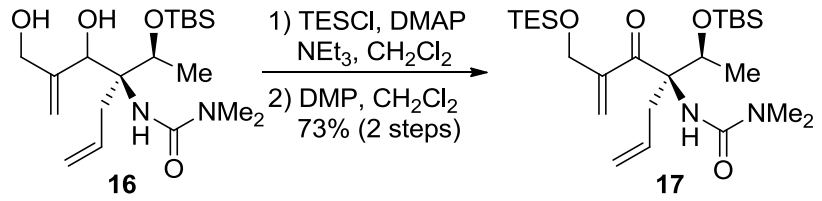
3-(2-((*tert*-butyldimethylsilyl)oxy)-3-formylhex-5-en-3-yl)-1,1-dimethylurea (14): A flame-dried 500-mL round-bottomed flask was charged with ester **10** (9.87 g, 27.7 mmol, 1.00 equiv) and CH_2Cl_2 (150 mL). The solution was cooled -78 °C and a solution of DIBAL-H was added (10.87 mL in 50 mL CH_2Cl_2, 61.0 mmol, 2.20 equiv). The reaction was allowed to warm to 0 °C was stirred at that temperature under a nitrogen atmosphere under a nitrogen atmosphere until consumption of starting material was indicated by TLC analysis, typically 2 h. The reaction was then cooled to -78 °C and acetone (300 mL) was added. Stirring continued for 10 min and 10% aqueous Rochelle's salt (200 mL) was added. The reaction was allowed to warm to rt and stirred for 1 h. The organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The crude alcohol product was taken on directly to the next step.

A 500-mL round-bottomed flask was charged with the crude alcohol and CH_2Cl_2 (150 mL). Dess Martin's periodinane (12.91 g, 30.50 mmol, 1.10 equiv) was added. The reaction was stirred at rt until consumption of starting material was indicated by TLC analysis, typically 30 min. Saturated $NaHCO_3$, $Na_2S_2O_4$, and Et_2O (50 mL, each) were added and stirring was continued

until two clear layers formed. The organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (90:10 to 85:15 petroleum ether/acetone) to give the desired product as a waxy solid (8.04 g, 89%). Analytical data: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.61 (s, 1H), 5.64-5.57 (m, 1H), 5.42 (s, 1H), 5.04 (d, *J* = 6.6, 1H), 5.02 (s, 1H), 4.57 (q, *J* = 6.0, 1H), 3.07 (dd, *J* = 14.4, 7.2 Hz, 1H), 2.91 (s, 6H), 2.84 (dd, *J* = 14.4, 7.8 Hz, 1H), 1.09 (d, *J* = 6.6, 3H), 0.91 (s, 9H), 0.14 (s, 3H), 0.09 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 202.0, 156.9, 133.0, 118.4, 70.9, 68.7, 36.2, 33.8, 25.8, 19.0, 17.9, -4.2, -5.0; **HRMS (ESI$^+$)** Calcd. for $C_{16}H_{32}N_2O_3Si$+Na, 351.2080; Found, 351.2076; **IR** (thin film, cm^{-1}) 2930, 2858, 1731, 1652, 1515, 1377, 1256, 1103, 836; **TLC** (90:10 petroleum ether/acetone): R_f = 0.20.

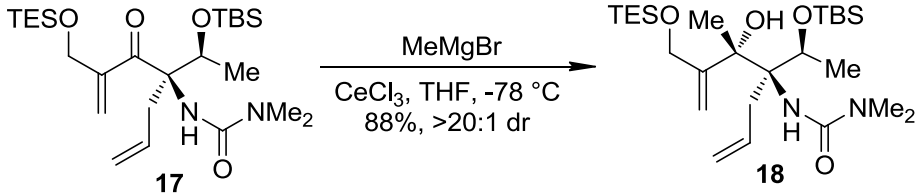


3-(1-((*tert*-butyldimethylsilyl)oxy)ethyl)-3-hydroxy-2-(hydroxymethyl)hepta-1,6-dien-4-yl)-1,1-dimethylurea (16): A flame-dried 250-mL round-bottomed flask was charged with 2-bromopropenol **15** (2.49 g, 18.17 mmol, 2.20 equiv) and Et_2O (100 mL). The solution was cooled to 0 °C under a nitrogen atmosphere. Methylmagnesium bromide (3.0 M, 6.05 mL, 18.17 mmol, 2.20 equiv) was added dropwise and the reaction was cooled to -78 °C. *tert*-Butyllithium (1.5 M, 24.0 mL, 36.34 mmol, 4.40 equiv) was added over 1 h via syringe pump. After the addition was complete the solution was warmed to 0 °C and stirred for 3 h. The reaction was cooled to -78 °C, and a solution of aldehyde **14** in Et_2O (15 mL) was added (2.71 g, 8.26 mmol, 1.00 equiv). The reaction was allowed to warm to rt over 12 h. Saturated NH_4Cl was added (50 mL) and the organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product diastereomers (~1:1, inconsequential, not separated) were purified via flash chromatography (90:10 to 70:30 petroleum ether/diethyl ether) to give the desired product as a colorless oil (1.99 g, 60%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.72 (d, *J* = 8.8 Hz), 7.67 (d, *J* = 9.6 Hz), 6.18-6.12 (m), 6.00-5.95 (m), 5.12 (s), 5.35 (s), 5.31 (s), 5.17-4.96 (m), 4.86 (q, *J* = 6.4 Hz), 4.78 (s), 4.33-4.27 (m), 4.18-4.07 (m), 3.12 (dd, *J* = 14.8, 9.6 Hz), 2.90 (s), 2.86 (s), 2.55 (dd, *J*=13.6, 9.6 Hz), 2.41 (dd, *J*=15.2, 6.0 Hz), 2.17 (dd, *J* = 14.4, 9.2 Hz), 1.32 (d, *J* = 6.0 Hz), 1.29 (d, *J* = 6.0 Hz), 0.89 (s), 0.87 (s), 0.10 (s), 0.07 (s), 0.07 (s), -0.07(s); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.6, 159.0, 149.6, 149.4, 136.2, 135.2, 118.8, 116.8, 115.3, 115.0, 79.8, 77.9, 71.9, 67.8, 64.6, 64.2, 62.7, 37.4, 36.9, 36.6, 34.3, 25.8, 19.0, 18.9, 17.9, 17.9, -4.1, -4.2, -4.2, -5.5; **HRMS (ESI$^+$)** Calcd. for $C_{19}H_{38}N_2O_4Si$+H, 387.2679; Found, 387.2685; **IR** (thin film, cm^{-1}) 3410, 2930, 1630, 1527, 1265, 838, 739; **TLC** (70:30 petroleum ether/acetone): R_f = 0.30.



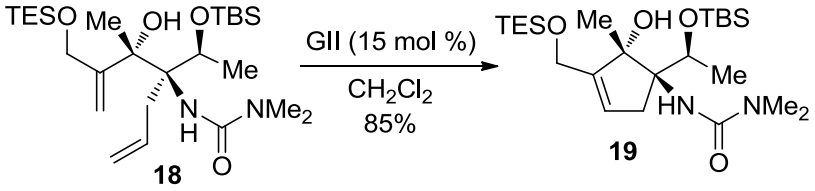
3-(6-allyl-11,11-diethyl-2,2,3,3,5-pentamethyl-8-methylene-7-oxo-4,10-dioxa-3,11-disilatridecan-6-yl)-1,1-dimethylurea (17): A flame-dried 250-mL round-bottomed flask was charged with diol **16** (0.90 g, 2.33 mmol, 1.00 equiv), DMAP (0.085 g, 0.699 mmol, 0.30 equiv), and CH_2Cl_2 (60 mL). The solution was cooled to 0 °C under a nitrogen atmosphere and triethylamine (0.65 mL, 4.66 mmol, 2.00 equiv) was added. Triethylsilyl chloride (0.47 mL, 2.79 mmol, 1.20 equiv) was added dropwise and the reaction was allowed to slowly warm to rt over 12 h. Saturated $NaHCO_3$ was added (50 mL) and the organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The crude monoalcohol was taken on directly to the next step.

A 100-mL round-bottomed flask was charged with the crude monoalcohol and CH_2Cl_2 (40 mL). Dess Martin's periodinane (1.48 g, 3.50 mmol, 1.50 equiv) was added. The reaction was stirred at rt until consumption of starting material was indicated by TLC analysis, typically 1 h. Saturated $NaHCO_3$, $Na_2S_2O_4$, and Et_2O (15 mL, each) were added and stirring was continued until two clear layers formed. The organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (90:10 petroleum ether/acetone) to give the desired product as a colorless oil (0.793 g, 68%). Analytical data: **^{1}H NMR** (500 MHz, $CDCl_3$): δ 6.50 (s, 1H), 6.01 (s, 1H), 5.88-5.79 (m, 1H), 5.58 (s, 1H), 4.97-4.89 (m, 2H), 4.53 (q, *J* = 6.5 Hz, 1H), 4.36 (s, 2H), 2.93 (dd, *J* = 14.0, 6.5 Hz, 1H), 2.86 (s, 6H), 2.78 (dd, *J* = 14.5, 14.0 Hz, 1H), 1.08 (d, *J* = 6.0 Hz, 3H), 0.95 (t, *J* = 8.0 Hz, 9H), 0.93 (s, 9H), 0.61 (q, *J* = 8.0 Hz), 0.15 (s, 3H), 0.11 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 199.4, 157.2, 145.1, 135.4, 120.8, 117.0, 71.4, 70.7, 61.9, 36.2, 35.4, 25.7, 18.8, 17.9, 6.7, 4.4, -4.1, -4.8; **HRMS (ESI$^+$)** Calcd. for $C_{25}H_{50}N_2O_4Si_2$+H, 499.3387; Found, 499.3391; **IR** (thin film, cm^{-1}) 3427, 2955, 1655, 1502, 1097, 950, 834, 738; **TLC** (90:10 Petroleum Ether/Acetone): R_f = 0.30.



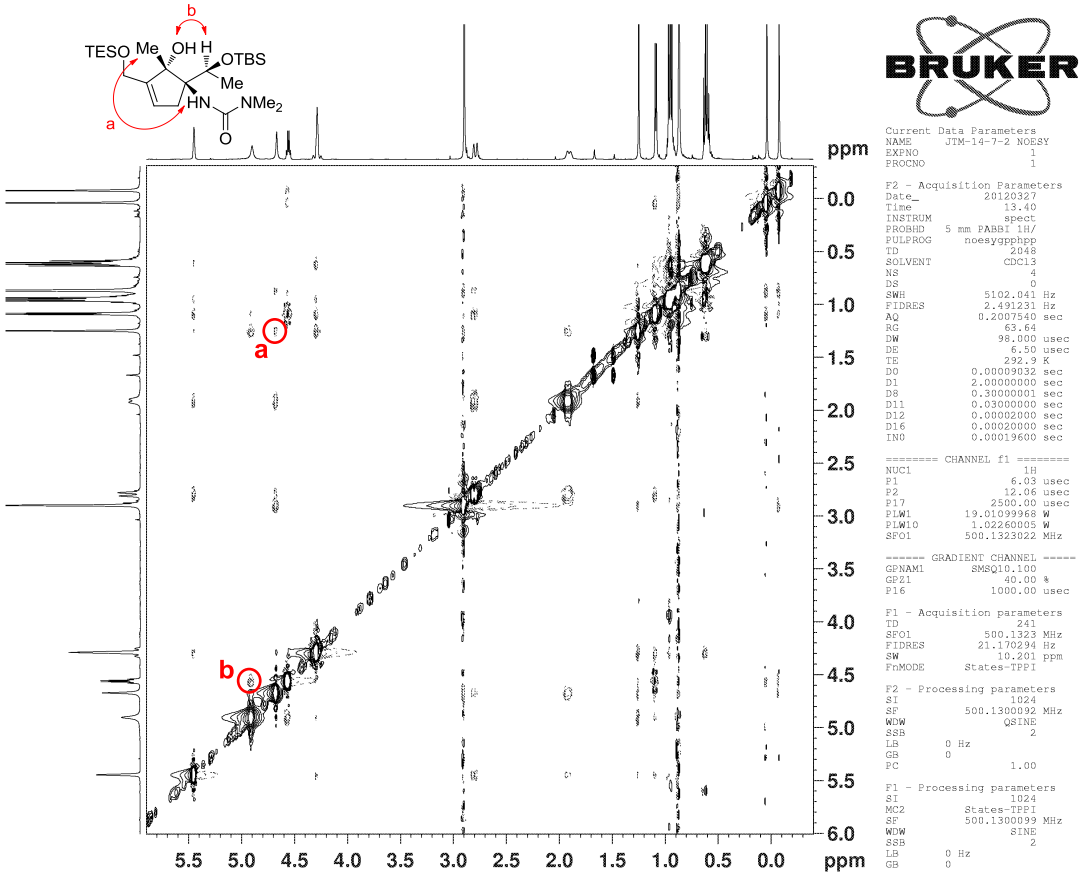
3-(6-allyl-11,11-diethyl-7-hydroxy-2,2,3,3,5,7-hexamethyl-8-methylene-4,10-dioxa-3,11-disilatridecan-6-yl)-1,1-dimethylurea (18): In a nitrogen-filled glove box a flame-dried 100-mL round-bottomed flask was charged with cerium trichloride (1.29 g, 5.25 mmol, 5.00 equiv).[1] The flask was capped with a rubber septum and removed from the glove box. THF (20 mL) was added at 0 °C and stirred at that temperature for 3 h. The solution was cooled to -78 °C and methylmagnesium bromide (3.0 M, 1.75 mL, 5.25 mmol, 5.00 equiv) was added. The reaction

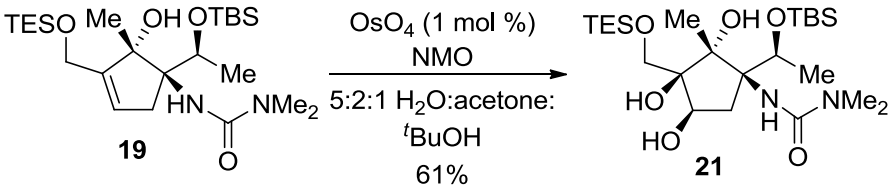
was stirred for 1 h followed by the addition of a THF solution (5 mL) of enone **17** (0.524 g, 1.05 mmol, 1.00 equiv). The reaction was then stirred at -78 °C for 5 h. Aqueous acetic acid (0.5 M, 20 mL) was added and the solution was warmed to rt. The organic layer was extracted with Et_2O, washed with saturated $NaHCO_3$, brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (95:5 petroleum ether/acetone) to give the desired product with >20:1 diasteromeric ratio as a colorless oil (0.476 g, 88%). Analytical data: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.81 (s, 1H), 6.02-5.94 (m, 1H), 5.53 (s, 1H), 5.42 (s, 1H), 5.08 (d, *J* = 17.4 Hz, 1H), 5.05 (d, *J* = 10.2 Hz, 1H), 4.98 (s, 1H), 4.58 (q, *J* = 6.0 Hz, 1H), 4.39 (d, *J* = 15.6 Hz, 1H), 4.21 (d, *J* = 15.6 Hz, 1H), 2.88 (dd, *J* = 12.0, 6.0 Hz, 1H), 2.85 (s, 6H), 2.58 (dd, *J* = 15.0, 5.4 Hz, 1H), 1.33 (d, *J* = 6.0 Hz, 3H), 1.32 (s, 3H), 0.94 (t, *J* = 8.4 Hz, 9H), 0.87 (s, 9H), 0.59 (q, *J* = 7.8 Hz, 6H), 0.11 (s, 3H), 0.02 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 159.4, 154.4, 135.9, 117.7, 110.6, 79.3, 69.0, 67.2, 62.9, 36.7, 34.4, 27.8, 21.5, 17.9, 6.8, 4.4, -4.1, -4.6; **HRMS (ESI$^+$)** Calcd. for $C_{26}H_{54}N_2O_4Si_2$+Na, 537.3520; Found, 537.3525; **IR** (thin film, cm^{-1}) 3390, 2955, 1631, 1265, 1080, 837, 740; **TLC** (90:10 petroleum ether/acetone): R_f = 0.30.



3-(1-(1-((tert-butyldimethylsilyl)oxy)ethyl)-2-hydroxy-2-methyl-3-(((triethylsilyl)oxy)methyl)cyclopent-3-en-1-yl)-1,1-dimethylurea (19): In a nitrogen-filled glove box a flame-dried 250-mL round-bottomed flask was charged with Grubbs's second generation catalyst (0.152 g, 0.18 mmol, 0.15 equiv). The flask was capped with a rubber septum and removed from the glove box. CH_2Cl_2 (70 mL) was added followed by a CH_2Cl_2 solution (5 mL) of diene **18** (0.618 g, 1.20 mmol, 1.00 equiv). The reaction stirred for 12 h under a nitrogen atmosphere. The reaction was then concentrated *in vacuo*. The product was purified via flash chromatography (90:10 to 80:20 hexanes/ethyl acetate) to give the desired product as white solid (0.495 g, 85%). Analytical data: **mp** 67-70 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 5.45 (s, 1H) 4.90 (s, 1H), 4.67 (s, 1H), 4.56 (q, *J* = 6.0 Hz, 1H), 4.31 (d, *J* = 16.0, 1H), 4.27 (d, *J* = 16.5, 1H), 2.90 (s, 6H), 2.80 (d, *J* = 15.5 Hz, 1H), 1.91 (d, *J* = 15.0 Hz, 1H), 1.25 (s, 3H), 1.09 (d, *J* = 6.5 Hz, 3H), 0.95 (t, *J* = 8.0 Hz, 9H), 0.87 (s, 9H), 0.61 (q, *J* = 8.0 Hz, 6H), 0.04 (s, 3H), -0.08 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.0, 149.5, 118.9, 83.7, 73.1, 68.0, 59.3, 36.6, 36.4, 25.9, 24.8, 21.8, 17.9, 6.7, 4.3, -4.1, -5.4; **HRMS (ESI$^+$)** Calcd. for $C_{24}H_{50}N_2O_4Si_2$+H, 487.3387; Found, 487.3386; **IR** (thin film, cm^{-1}) 3417, 2955, 1632, 1517, 1265, 1084, 834, 739; **TLC** (90:10 hexanes/EtOAc): R_f = 0.40.

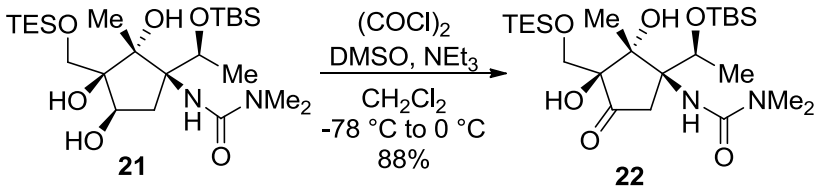
^{1}H nOesy analysis of 19:



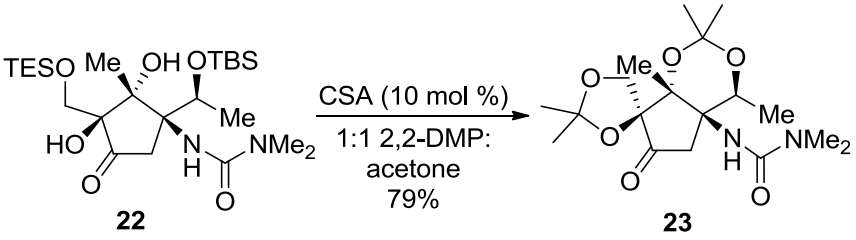


3-(1-(1-((tert-butyldimethylsilyl)oxy)ethyl)-2,3,4-trihydroxy-2-methyl-3-(((triethylsilyl)oxy)methyl)cyclopentyl)-1,1-dimethylurea (21): A 20-mL scintillation vial was charged with *N*-methylmorpholine *N*-oxide (0.40 g, 3.45 mmol, 5.00 equiv) and H_2O (7 mL). ^{t}BuOH (2 mL) was then added followed by OsO_4 (0.002 g, 0.007 mmol, 0.01 equiv). The mixture was stirred and an acetone solution (3 mL) of cyclopentene **19** (0.336 g, 0.69 mmol, 1.00 equiv) was added. The suspension was stirred vigorously until consumption of starting material was indicated by TLC analysis, typically 24-48 h. Talc (0.5 g) was added followed by $Na_2S_2O_4$ (5 mL). The solids were filtered off and the organics were removed *in vacuo*. The aqueous solution was extracted with CH_2Cl_2 (3 x 5 mL), washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (70:30 hexanes/ethyl acetate) to give the desired product with >20:1 diasteromeric ratio as a pale brown oil (0.218 g, 61%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.66 (s, 1H), 5.55 (s, 1H), 5.09 (s, 1H), 4.68 (q, *J* = 6.4 Hz, 1H), 4.37-4.31 (m, 1H), 3.75 (d, *J* = 10.0 Hz, 1H), 3.68 (d, *J* =

10.4 Hz, 1H), 3.48 (d, *J* = 9.6 Hz, 1H), 2.89 (s, 6H), 2.40 (dd, *J* = 14.8, 6.4 Hz, 1H), 2.17 (dd, *J* = 14.8, 9.2 Hz, 1H), 1.28 (s, 3H), 1.14 (d, *J* = 6.0 Hz, 3H), 0.95 (t, *J* = 8.0 Hz, 9H), 0.87 (s, 9H), 0.62 (q, *J* = 8.0 Hz, 6H), 0.13 (s, 3H), 0.09 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.7, 85.7, 79.6, 71.0, 69.0, 64.1, 37.6, 36.2, 25.9, 19.6, 19.4, 17.8, 6.5, 4.0, -4.2, -4.4; **HRMS (ESI$^+$)** Calcd. for $C_{24}H_{52}N_2O_6Si_2$+Na, 543.3262; Found, 543.3264; **IR** (thin film, cm^{-1}) 3420, 2955, 1628, 1535, 1375, 1253, 1051, 829, 738; **TLC** (60:40 hexanes/EtOAc): R_f = 0.40.

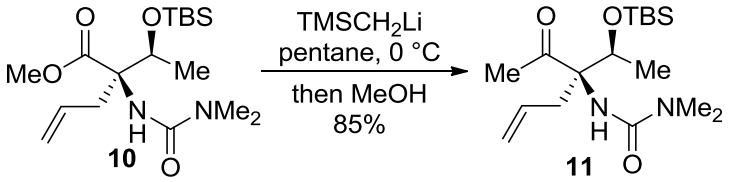


3-(1-(1-((tert-butyldimethylsilyl)oxy)ethyl)-2,3-dihydroxy-2-methyl-4-oxo-3-(((triethylsilyl)oxy)methyl)cyclopentyl)-1,1-dimethylurea (22): A flame-dried 20-mL scintillation vial was charged with oxalyl chloride (0.023 mL, 0.265 mmol, 1.20 equiv) and CH_2Cl_2 (4 mL). The solution was cooled to -78 °C and DMSO (0.038 mL, 0.528 mmol, 2.40 equiv) was added dropwise. The reaction was stirred for 10 min and a CH_2Cl_2 solution (2 mL) of triol **21** (0.115 g, 0.22 mmol, 1.00 equiv) was added. Stirring was continued for 30 min followed by the dropwise addition of triethylamine (0.153 mL, 1.10 mmol, 5.00 equiv). The reaction was allowed to warm slowly in the dry ice/acetone bath until consumption of starting material was indicated by TLC analysis, typically 1-2 h. H_2O was added (5 mL) and the organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (60:40 hexanes/ethyl acetate) to give the desired product as a pale brown oil (0.100 g, 88%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.95 (s, 1H), 5.80 (s, 1H), 5.15 (s, 1H), 4.80 (q, *J* = 6.4 Hz, 1H), 3.97 (d, *J* = 10.4 Hz, 1H), 3.79 (d, *J* = 10.4 Hz, 1H), 2.87 (s, 6H), 2.72 (d, *J* = 18.4 Hz, 1H), 2.24 (d, *J* = 18.4 Hz, 1H), 1.36 (s, 3H), 1.11 (d, *J* = 6.0 Hz, 3H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.90 (s, 9H), 0.60 (q, *J* = 8.0 Hz, 6H), 0.17 (s, 3H), 0.13 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 213.6, 159.0, 84.0, 78.9, 69.9, 66.2, 64.8, 44.6, 36.2, 25.9, 19.7, 19.7, 17.7, 6.5, 4.0, -4.2; **HRMS (ESI$^+$)** Calcd. for $C_{24}H_{50}N_2O_6Si_2$+Na, 541.3105; Found, 541.3107; **IR** (thin film, cm^{-1}) 3419, 2956, 1752, 1623, 1532, 1265, 1077, 740; **TLC** (60:40 hexanes/EtOAc): R_f = 0.50.

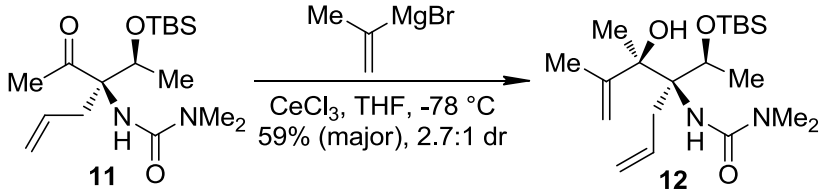


3-(2,2,2',2',4,7a-hexamethyl-6-oxotetrahydro-4H-spiro[cyclopenta[d][1,3]dioxine-7,4'-[1,3]dioxolan]-4a-yl)-1,1-dimethylurea (23): A flame-dried 20-mL scintillation vial was charged with diol **22** (0.165 g, 0.318 mmol, 1.00 equiv) and 1:1 acetone/2,2-dimethoxypropane

(6 mL). CSA (0.007 g, 0.032 mmol, 0.10) was added and the vial was capped. The reaction was allowed to stir at rt for 48 h. The solvent was then removed *in vacuo*. The product was purified via flash chromatography (60:40 hexanes/ethyl acetate) to give the desired product as a white solid (0.093 g, 79%). Analytical data: **mp** 137-139 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.20 (s, 1H), 4.35 (q, *J* = 6.4 Hz, 1H), 4.18 (d, *J* = 9.6 Hz, 1H), 4.11 (d, *J* = 9.6 Hz, 1H), 3.48 (d, *J* = 20.0 Hz, 1H), 2.85 (s, 6H), 2.72 (d, *J* = 20.0 Hz, 1H), 1.59 (s, 3H), 1.44 (s, 3H), 1.43 (s, 3H), 1.39 (d, *J* = 6.4 Hz, 3H), 1.35 (s, 3H), 1.24 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 212.6, 157.7, 112.1, 99.7, 86.7, 79.9, 73.2, 65.3, 61.0, 47.9, 36.0, 30.6, 26.3, 25.3, 25.1, 18.7, 15.4; **HRMS (ESI$^+$)** Calcd. for $C_{18}H_{30}N_2O_6$+H, 371.2182; Found, 371.2186; **IR** (thin film, cm^{-1}) 3430, 3055, 2988, 1754, 1657, 1523, 1382, 1265, 740; **TLC** (60:40 hexanes/EtOAc): R_f = 0.30.

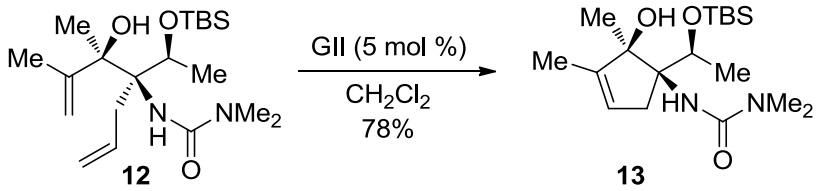


3-(1-((*tert*-butyldimethylsilyl)oxy)ethyl)-3-((1-(dimethylamino)vinyl)amino)hex-5-en-2-one (11): A flame-dried 50-mL round-bottomed flask was charged with ester **10** (0.665 g, 1.86 mmol, 1.00 equiv) and pentane (10 mL). The solution was cooled to 0 °C and $TMSCH_2Li$ (1 M, 5.57 mL, 5.57 mmol, 3.00 equiv) was added dropwise. The reaction was stirred until consumption of starting material was indicated by TLC analysis, typically 3 h. MeOH (3 mL) was then added and the reaction was warmed to rt and stirred for 1 h. H_2O (10 mL) was added and the organic layer was extracted with Et_2O, washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (90:10 to 70:30 petroleum ether/acetone) to give the desired product as a white solid (0.541 g, 85%). Analytical data: **mp** 41-42 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.89 (s, 1H), 5.53-5.42 (m, 1H), 5.02-4.95 (m, 2H), 4.67 (q, *J* = 6.4 Hz, 1H), 3.33 (dd, *J* = 14.4, 6.8 Hz, 1H), 2.87 (s, 6H), 2.77 (dd, *J* = 14.4, 8.0 Hz, 1H), 2.29 (s, 3H), 0.96 (d, *J* = 6.4 Hz, 3H), 0.89 (s, 9H), 0.15 (s, 3H), 0.07 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 208.5, 156.6, 133.1, 117.9, 71.9, 69.3, 36.1, 34.7, 27.2, 25.8, 18.7, 17.8, -4.4, -4.9; **HRMS (ESI$^+$)** Calcd. for $C_{17}H_{34}N_2O_3Si$+Na, 365.2237; Found, 365.2245; **IR** (thin film, cm^{-1}) 3412, 2931, 2858, 1656, 1510, 1374, 1257, 1101, 981, 832; **TLC** (70:30 petroleum ether/acetone): R_f = 0.60.



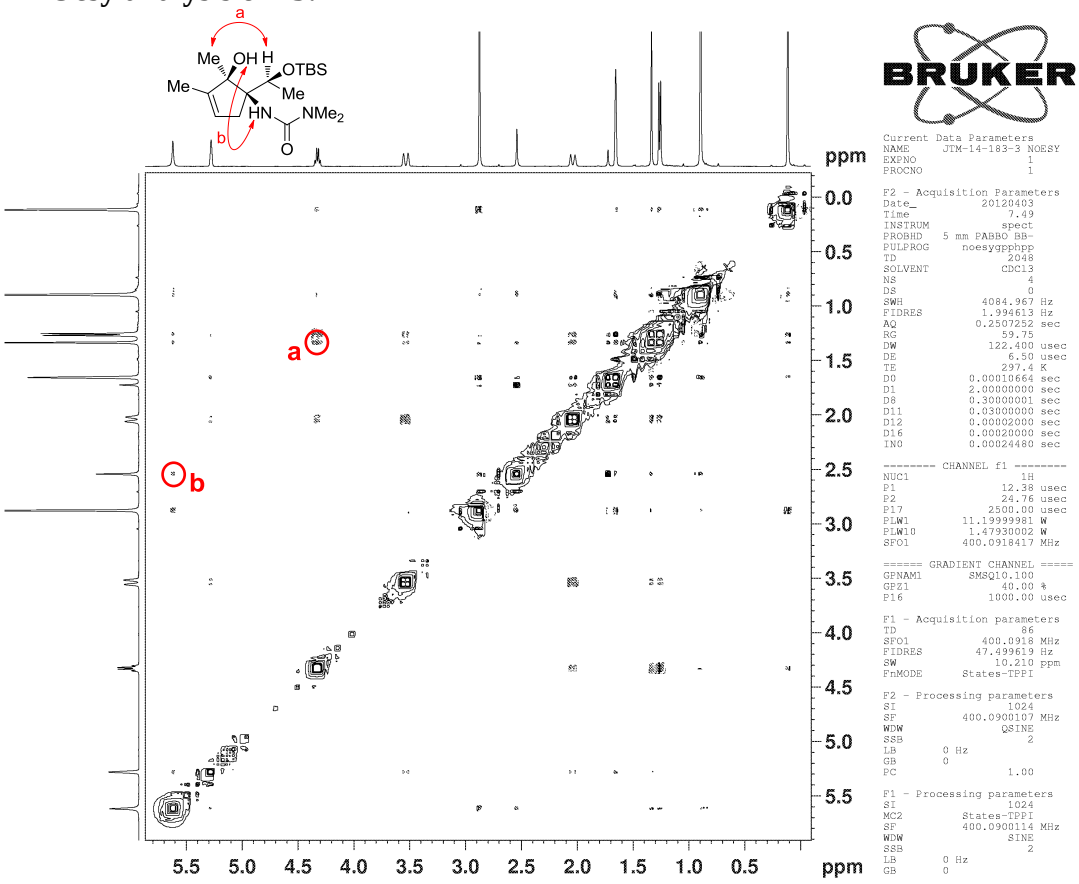
4-(1-((*tert*-butyldimethylsilyl)oxy)ethyl)-4-((1-(dimethylamino)vinyl)amino)-2,3-dimethylhepta-1,6-dien-3-ol (12): In a nitrogen-filled glove box a flame-dried 20-mL scintillation vial was charged with cerium trichloride (0.718 g, 2.92 mmol, 5.00 equiv).[1] The vial was capped with a rubber septum and removed from the glove box. THF (8 mL) was added at 0

°C and stirred at that temperature for 3 h. The solution was cooled to -78 °C and isopropenylmagnesium bromide (0.5 M, 5.85 mL, 2.92 mmol, 5.00 equiv) was added. The reaction was stirred for 1 h followed by the addition of a THF solution (2 mL) of ketone **11** (0.200 g, 0.585 mmol, 1.00 equiv). The reaction was then stirred at -78 °C for 5 h. Aqueous acetic acid (0.5 M, 5 mL) was added and the solution was warmed to rt. The organic layer was extracted with Et_2O, washed with saturated $NaHCO_3$, brine, dried with magnesium sulfate, and concentrated *in vacuo*. Crude NMR indicated a 2.7:1 diasteromeric ratio. The product was purified via flash chromatography (90:10 to 80:20 hexanes/ethyl acetate) to give the major diastereomer as a colorless oil (0.132 g, 59%). Analytical data: **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.15 (s, 1H), 6.07-5.98 (m, 1H), 5.52 (s, 1H), 5.10 (s, 1H), 5.04 (d, *J* = 6.0 Hz, 1H), 5.02 (s, 1H), 4.87 (s, 1H), 4.40 (q, *J* = 6.0 Hz, 1H), 2.94 (dd, *J* = 14.5, 10.0 Hz, 1H), 2.84 (s, 6H), 2.51 (dd, *J* = 15.0, 5.0 Hz, 1H), 1.87 (s, 3H), 1.34 (s, 3H), 1.32 (d, *J* = 6.0 Hz, 3H), 0.88 (s, 9H), 0.09 (s, 3H), 0.02 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 159.1, 150.1, 136.7, 117.4, 113.8, 79.1, 70.8, 66.7, 36.6, 34.2, 25.9, 25.6, 21.9, 21.0, 18.0, -4.4, -4.4; **HRMS (ESI$^+$)** Calcd. for $C_{20}H_{40}N_2O_3Si$+Na, 407.2706; Found, 407.2717; **IR** (thin film, cm^{-1}) 3403, 3055, 1631, 1422, 1265, 1070, 739; **TLC** (70:30 petroleum ether/acetone): R_f = 0.70.



2-(1-((*tert*-butyldimethylsilyl)oxy)ethyl)-2-((1-(dimethylamino)vinyl)amino)-1,5-dimethylcyclopentanol (13): In a nitrogen-filled glove box a flame-dried 20-mL scintillation vial was charged with Grubbs's second generation catalyst (0.003 g, 0.003 mmol, 0.05 equiv). The vial was capped with a rubber septum and removed from the glove box. CH_2Cl_2 (3 mL) was added followed by a CH_2Cl_2 solution (2 mL) of diene **12** (0.02 g, 0.052 mmol, 1.00 equiv). The reaction stirred for 6 h under a nitrogen atmosphere. The reaction was then concentrated *in vacuo*. The product was purified via flash chromatography (75:25 hexanes/ethyl acetate to 70:30 petroleum ether/acetone) to give the desired product as a white solid (0.014 g, 78%). Analytical data: **mp** 143-146 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.62 (s, 1H), 5.28 (s, 1H), 4.32 (q, *J* = 6.4 Hz, 1H), 3.53 (d, *J* = 16.0 Hz, 1H), 2.88 (s, 6H), 2.54 (s, 1H), 2.03 (d, *J* = 16.0 Hz, 1H), 1.66 (s, 3H), 1.33 (s, 3H), 1.26 (d, *J* = 6.4 Hz, 3H), 0.89 (s, 9H), 0.11 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.5, 143.5, 122.4, 86.4, 72.6, 71.0, 36.3, 33.8, 26.0, 21.7, 20.1, 18.0, 11.7, -3.1, -4.5; **HRMS (ESI$^+$)** Calcd. for $C_{18}H_{36}N_2O_3Si$+H, 357.2573; Found, 357.2575; **IR** (thin film, cm^{-1}) 3315, 3054, 2931, 2857, 2305, 1613, 1520, 1265, 1172, 739; **TLC** (70:30 petroleum ether/acetone): R_f = 0.60.

^{1}H nOesy analysis of 13:



BRUKER

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F1 - Processing parameters
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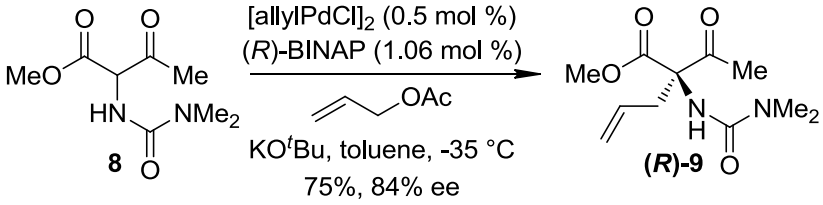
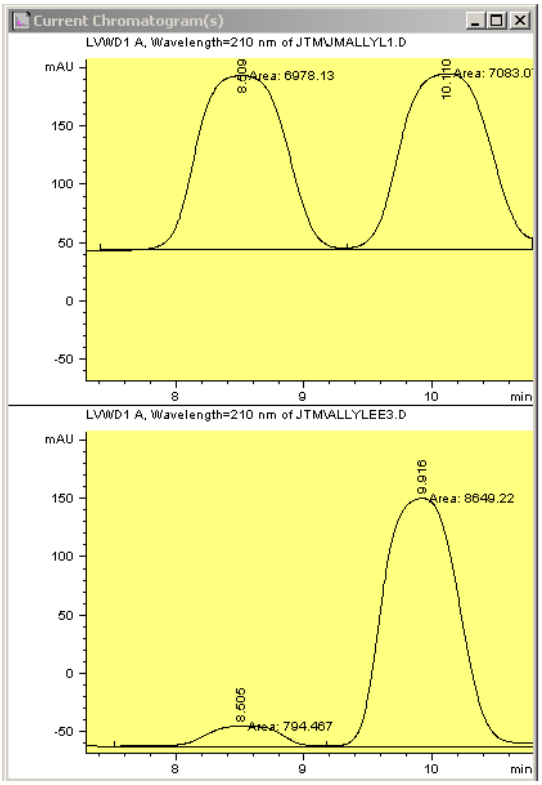
Preparation of Enantioenriched (*R*)-9:

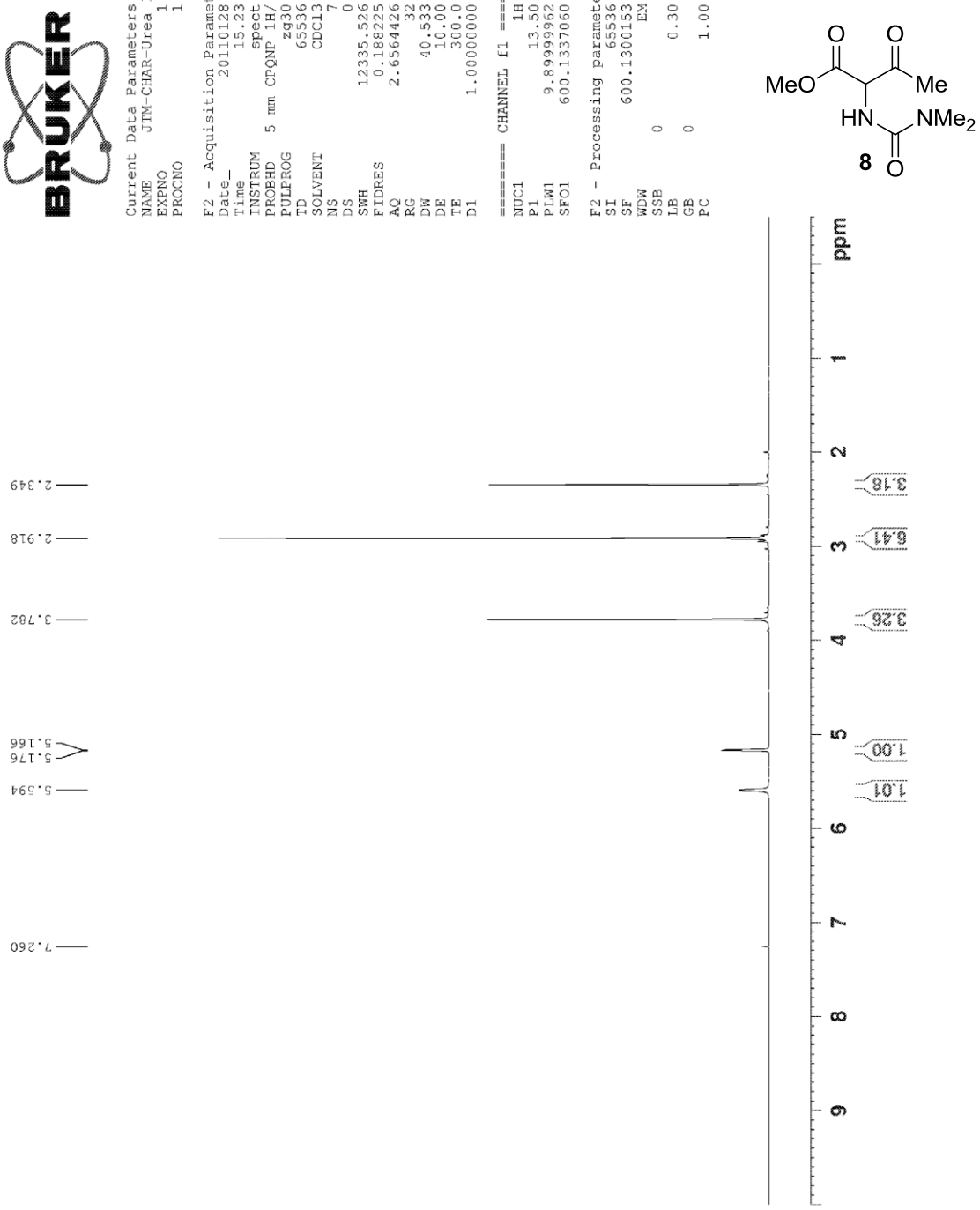


(*R*)-methyl 2-acetyl-2-(3,3-dimethylureido)pent-4-enoate ((*R*)-9): In a nitrogen-filled glove box a flame-dried 4-mL vial was charged with allylpalladium chloride dimer (0.001 g, 0.002 mmol, 0.005 equiv) and (*R*)-BINAP (0.003 g, 0.005 mmol, 0.0106 equiv). Toluene (0.5 mL) was added and the suspension was stirred for 10 min, capped with a rubber septum, and removed from the glove box. Allyl acetate (0.056 mL, 0.52 mmol, 1.10 equiv) was added and the catalyst solution was stirred for an additional 10 min. A separate flame-dried 20-mL scintillation vial was charged with **8** (0.095 g, 0.47 mmol, 1.00 equiv) and KO^{t}Bu (0.055 g, 0.49 mmol, 1.05 equiv). Toluene (1.5 mL) was added and the suspension was stirred under a nitrogen atmosphere at -35 °C. The catalyst solution was introduced via cannula transfer, and the reaction was stirred for 24 h at -35 °C. The reaction was quenched with 1 M HCl (1 mL), and extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The product was purified via flash chromatography (80:20 to 70:30 petroleum ether/acetone) to give the desired product as a colorless oil (0.085 g, 75%, e.r. 92:8). The enantiomer ratio was determined by SFC analysis (Chiralcel, OD, 2.0% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_R-minor 8.5 min, t_R-major 9.9 min).

SFC Trace:



^{1}H, ^{13}C NMR Spectra:



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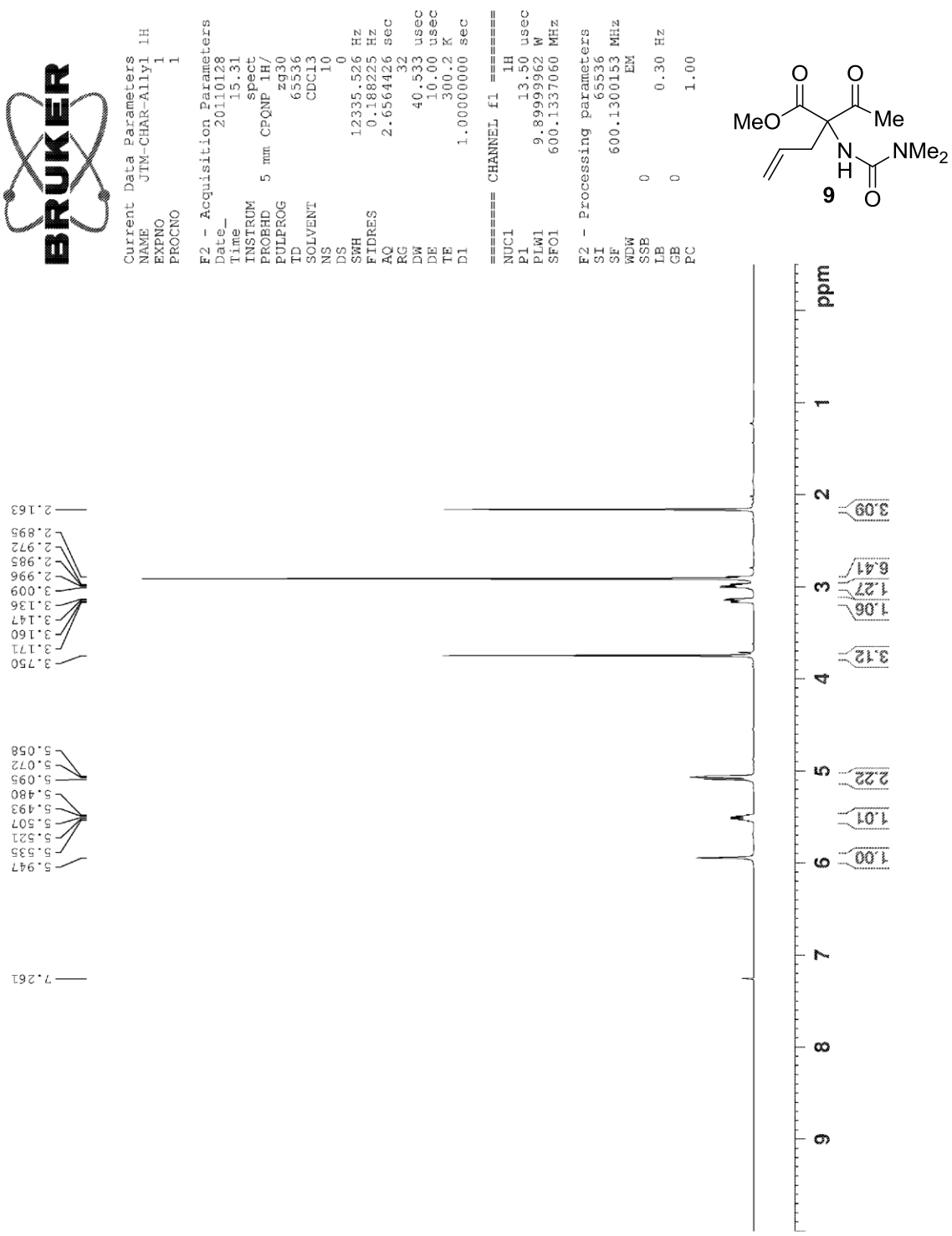
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 10.12 usec
PLW1 33.00000000 W
SFO1 150.9178981 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 9.89999962 W
PLW12 2.50000000 W
PLW13 0.18043000 W
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028228 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
Me
HN
NMe2
8
27.95
36.07
53.08
64.26
76.79
77.00
77.21
156.81
167.63
199.79
ppm


Current Data Parameters
NAME JTM-CHAR-Allyl 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110128
Time 15.31
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 32
DW 40.533 usec
DE 10.00 usec
TE 300.2 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.89999962 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 65536
SF 600.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
Me
NMe2
9
ppm
3.09
6.41
1.27
1.06
3.12
2.22
1.01
1.00
2.163
2.895
2.972
2.985
2.996
3.009
3.136
3.147
3.160
3.171
3.750
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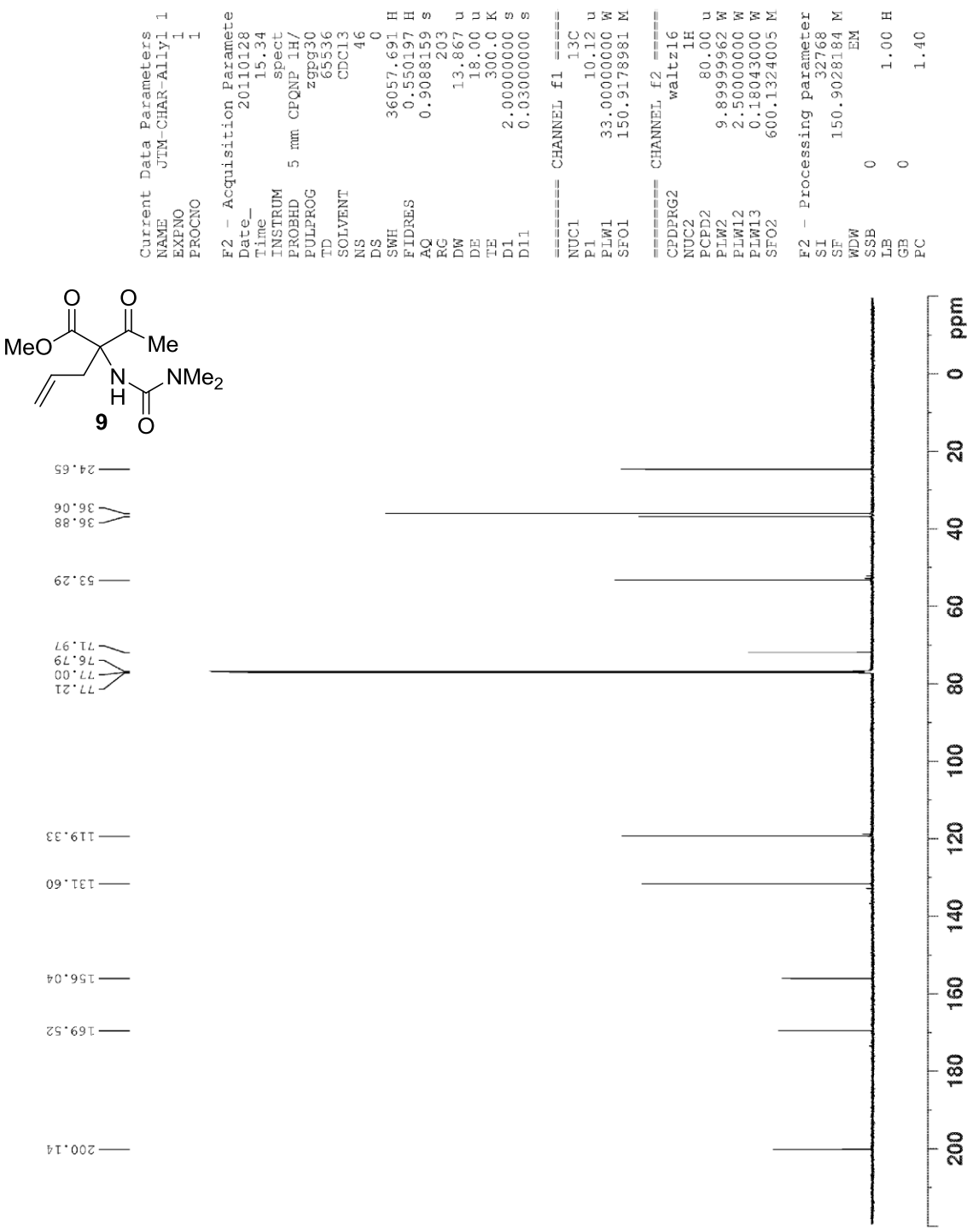
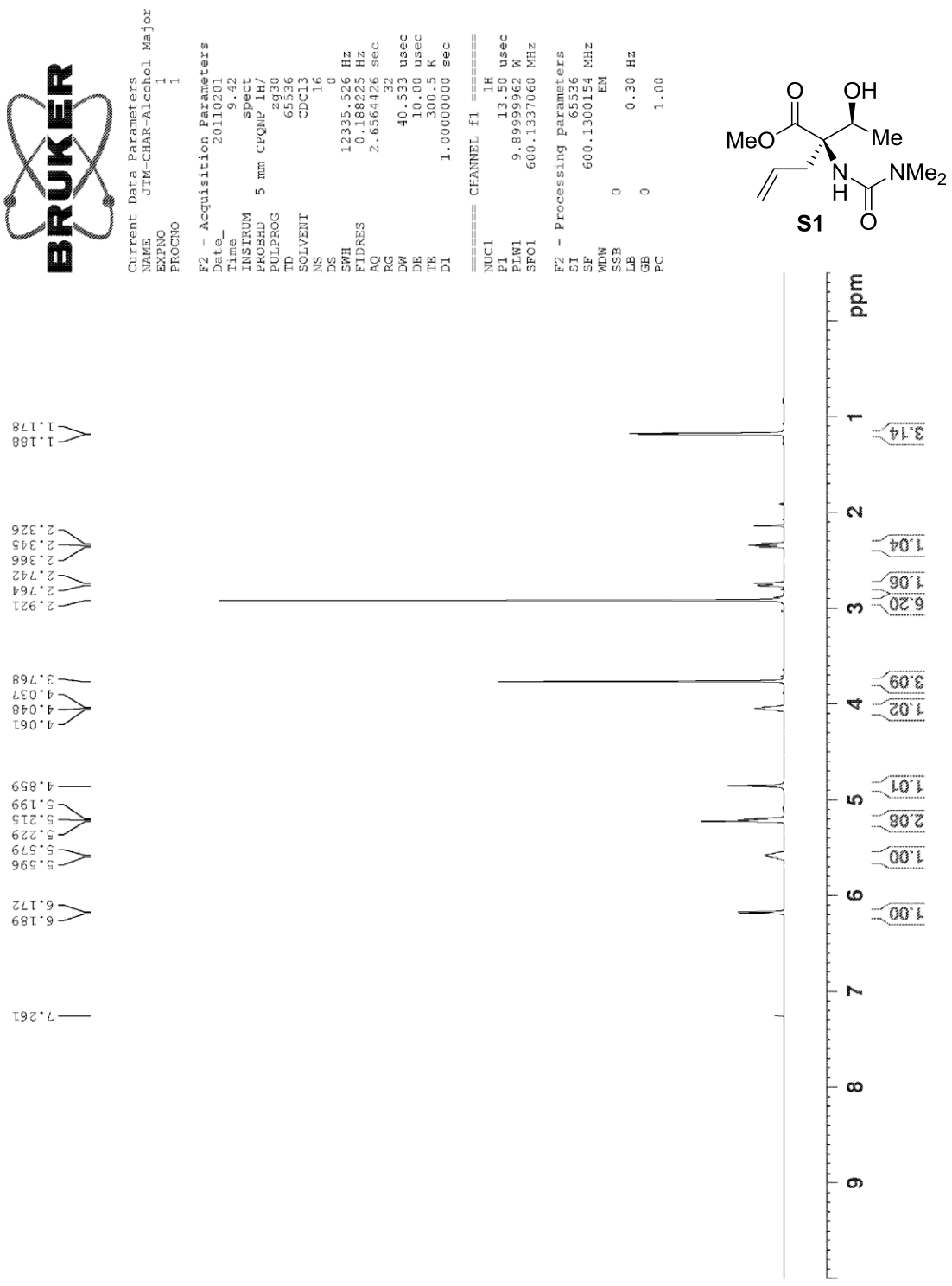
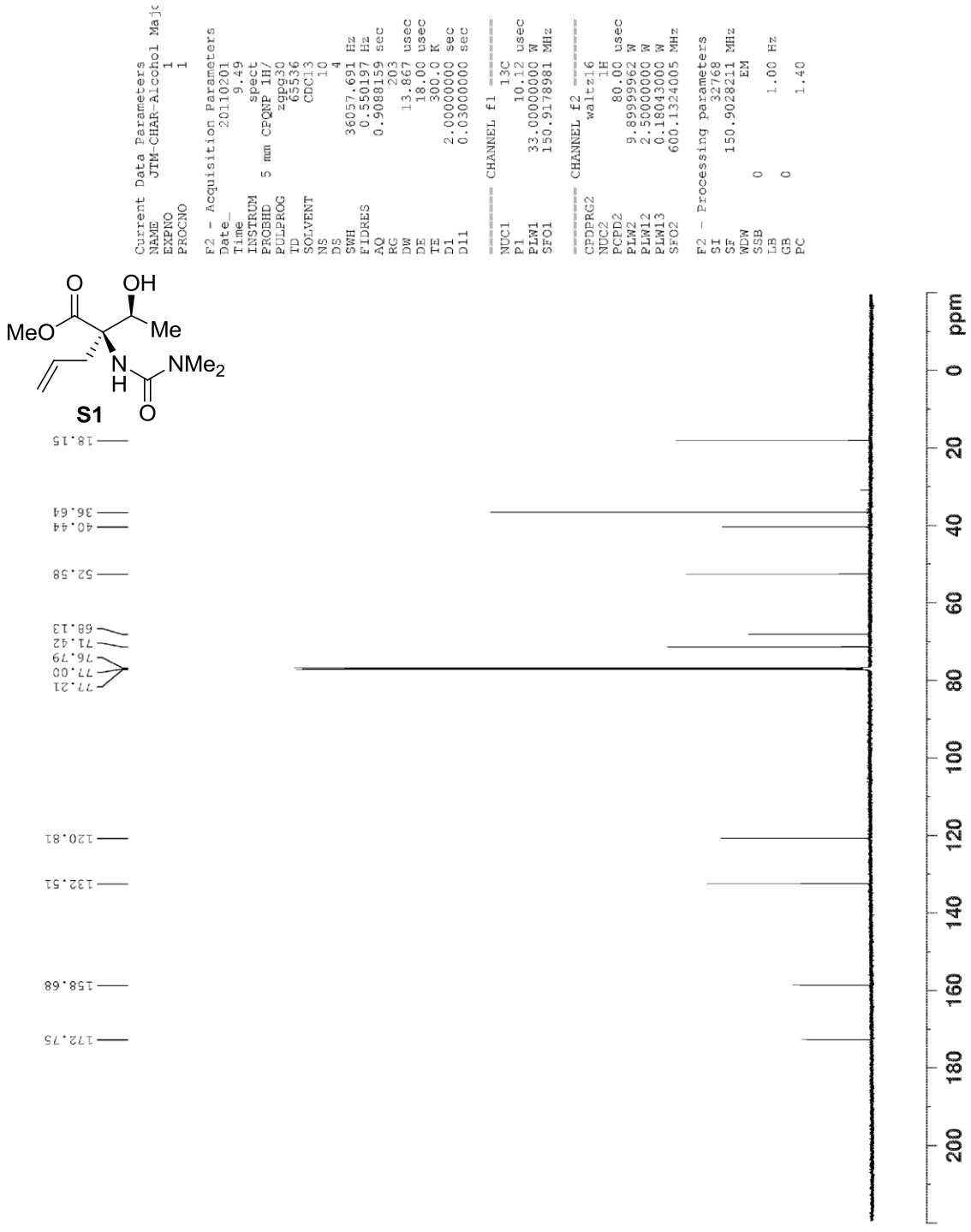
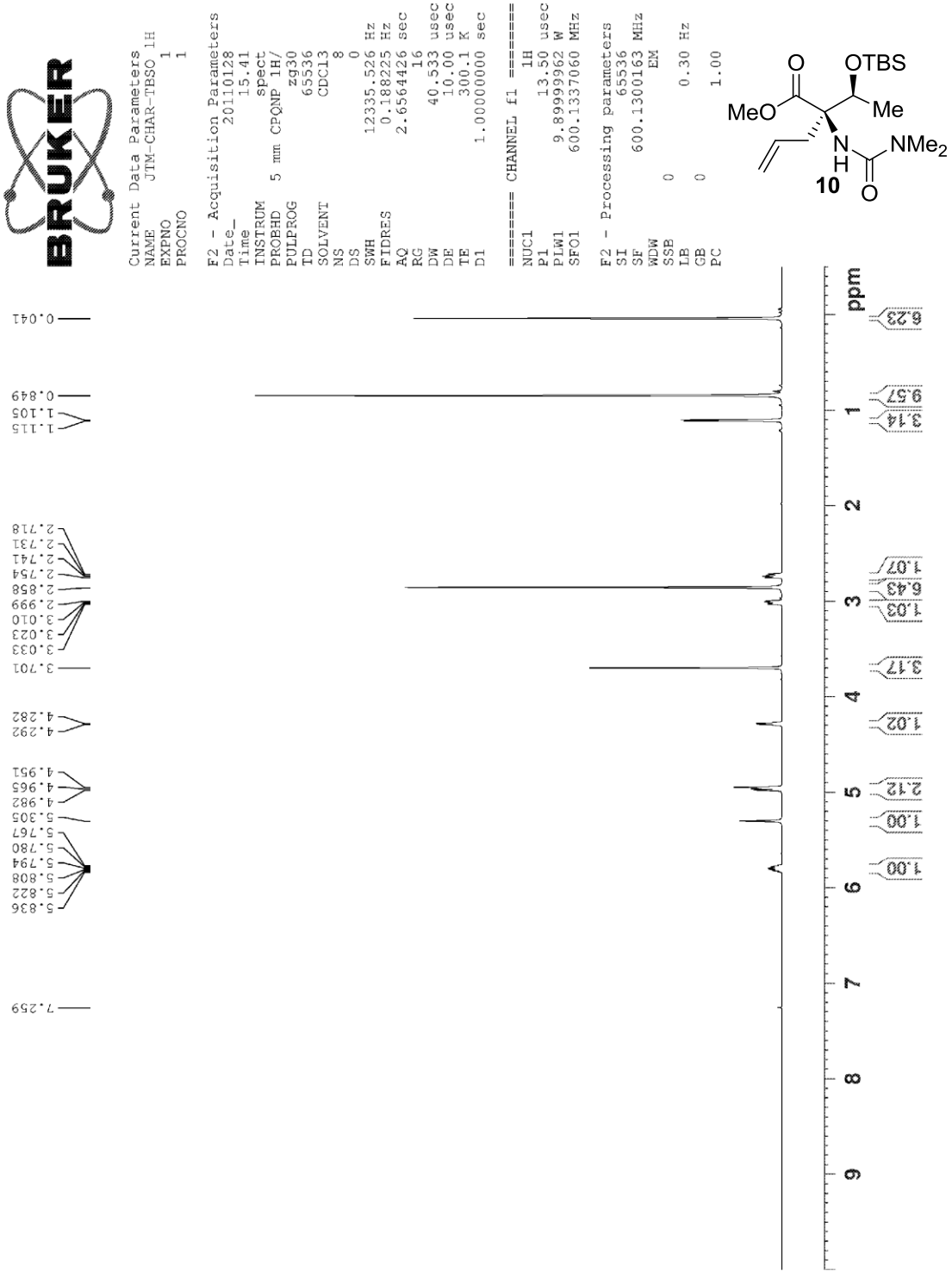
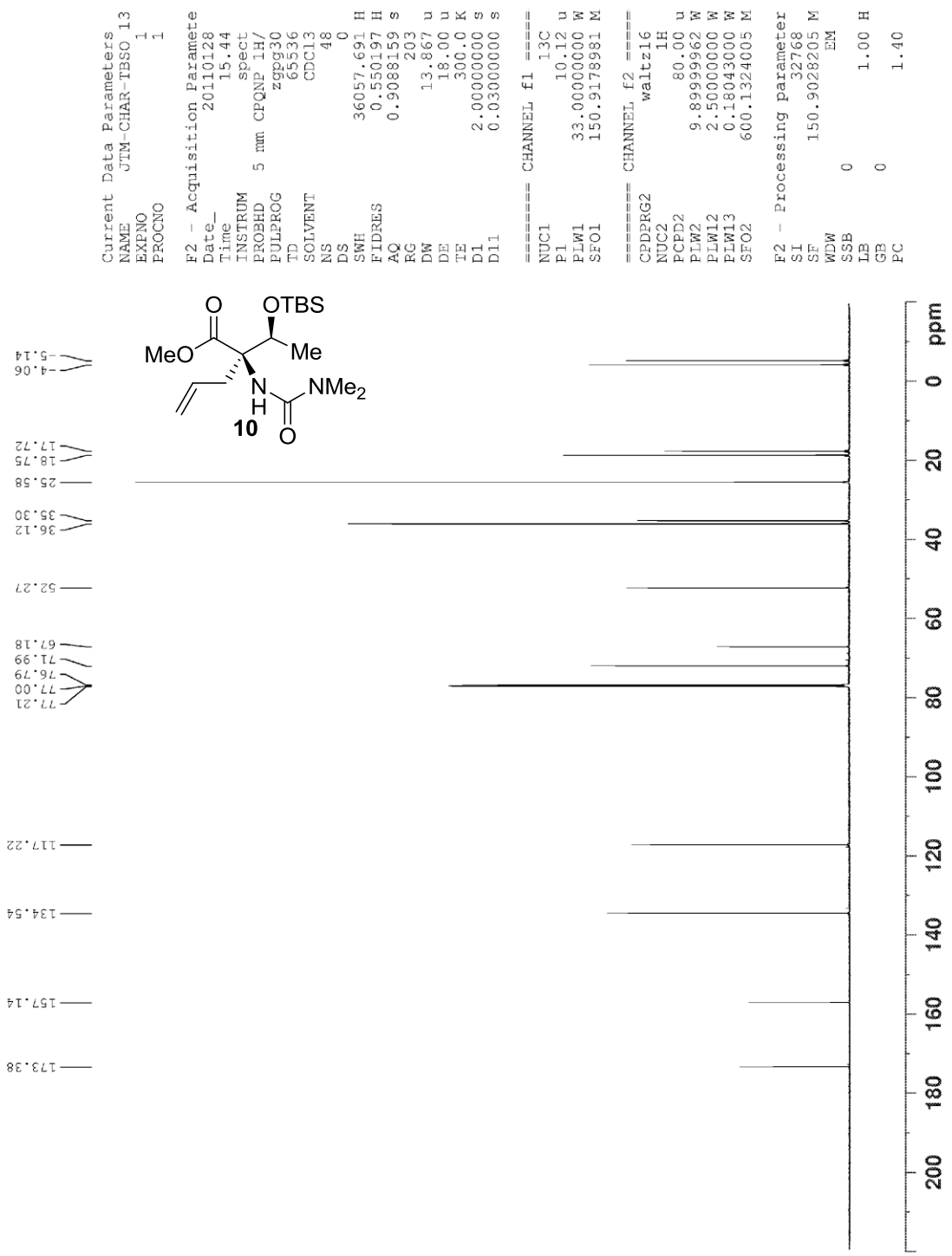
5.072
5.095
5.480
5.493
5.507
5.521
5.535
5.947
7.261

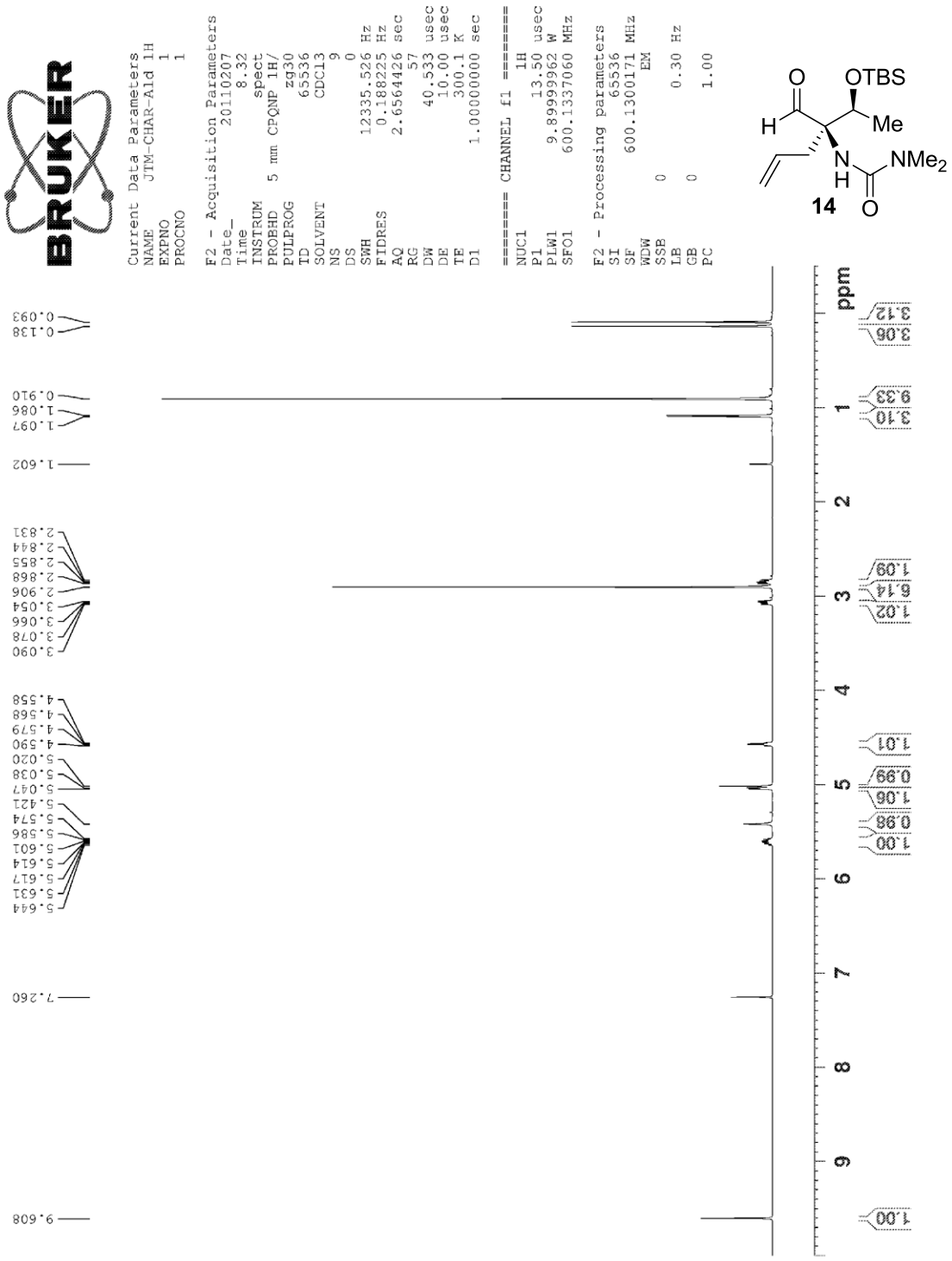

Current Data Parameters
NAME JTM-CHAR-Allyl 1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110128
Time 15.34
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 46
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.12 usec
PLW1 33.00000000 W
SFO1 150.9178981 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 9.89999962 W
PLW12 2.50000000 W
PLW13 0.18043000 W
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028184 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
Me
NMe2
N
H
9
24.65
36.06
36.88
53.29
71.97
76.79
77.00
77.21
119.33
131.60
156.04
169.52
200.14
ppm
0
20
40
60
80
100
120
140
160
180
200

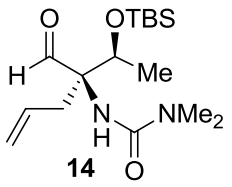

Current Data Parameters
NAME JTM-CHAR-Alcohol Major
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110201
Time 9.42
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 32
DW 40.533 usec
DE 10.00 usec
TE 300.5 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.89999962 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 65536
SF 600.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
O
OH
Me
N
H
NMe2
O
S1
ppm
1.178
1.188
2.326
2.345
2.366
2.742
2.764
2.921
3.768
4.037
4.048
4.061
4.859
5.199
5.215
5.229
5.579
5.596
6.172
6.189
7.261
3.14
1.04
1.06
6.20
3.09
1.02
1.01
2.08
1.00
1.00


Current Data Parameters
NAME JTM-CHAR-Alcohol Majc
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110201
Time 9.49
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.12 usec
PLW1 33.00000000 W
SFO1 150.9178981 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 9.89999962 W
PLW12 2.50000000 W
PLW13 0.18043000 W
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028211 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
OH
Me
N
H
NMe2
O
O
S1
18.15
36.64
40.44
52.58
68.13
71.42
76.79
77.00
77.21
120.81
132.51
158.68
172.75
ppm
0
20
40
60
80
100
120
140
160
180
200

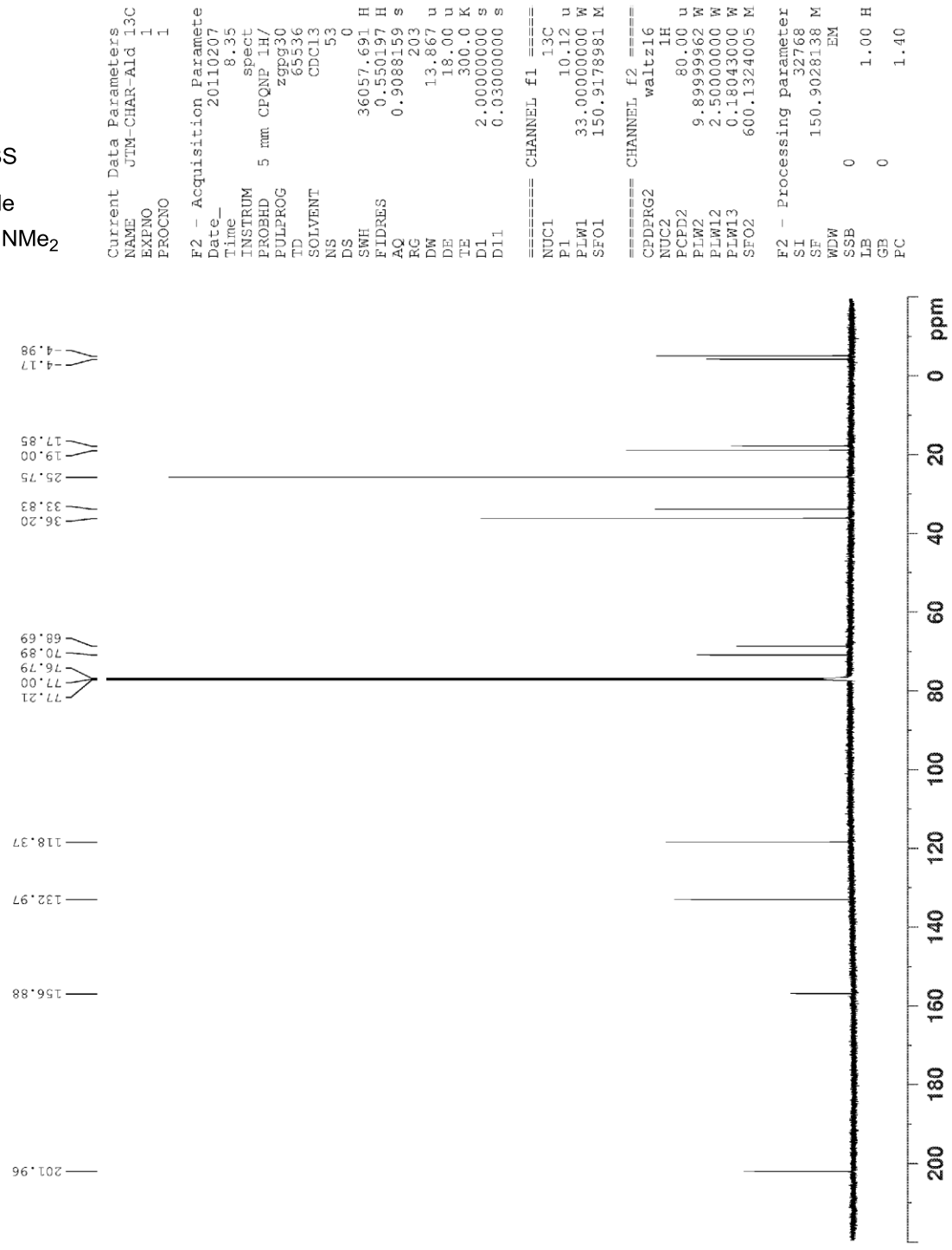

Current Data Parameters
NAME JTM-CHAR-TBSO 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110128
Time 15.41
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 16
DW 40.533 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.89999962 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 65536
SF 600.1300163 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
O
OTBS
Me
N
H
NMe2
O
10
ppm

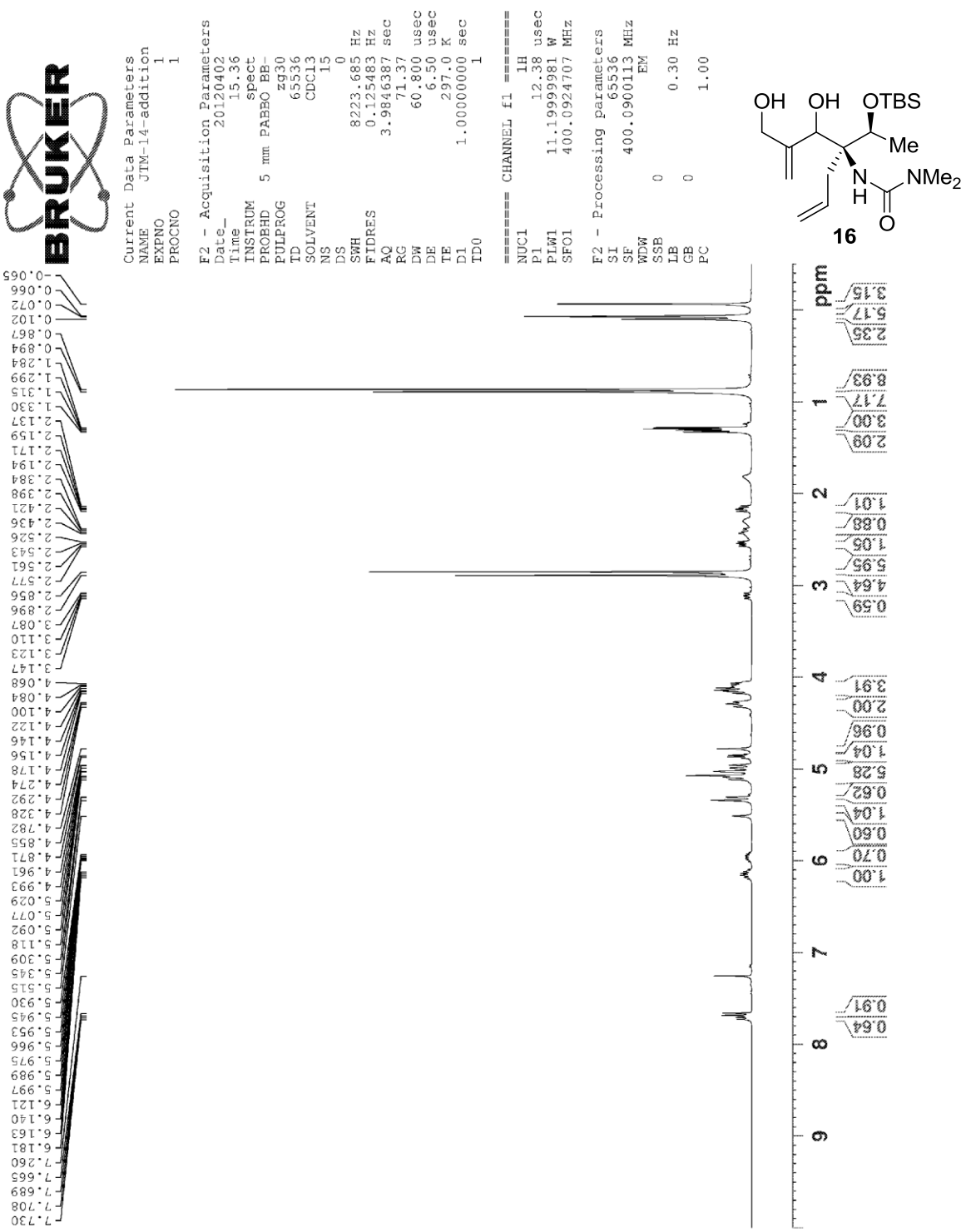

Current Data Parameters
NAME JTM-CHAR-TBSO 13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110128
Time 15.44
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.12 usec
PLW1 33.00000000 W
SFO1 150.9178981 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 9.89999962 W
PLW12 2.50000000 W
PLW13 0.18043000 W
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028205 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OTBS
MeO
Me
N
H
NMe2
O
10
-5.14
-4.06
17.72
18.75
25.58
35.30
36.12
52.27
67.18
71.99
76.79
77.00
77.21
117.22
134.54
157.14
173.38
ppm
200
180
160
140
120
100
80
60
40
20
0

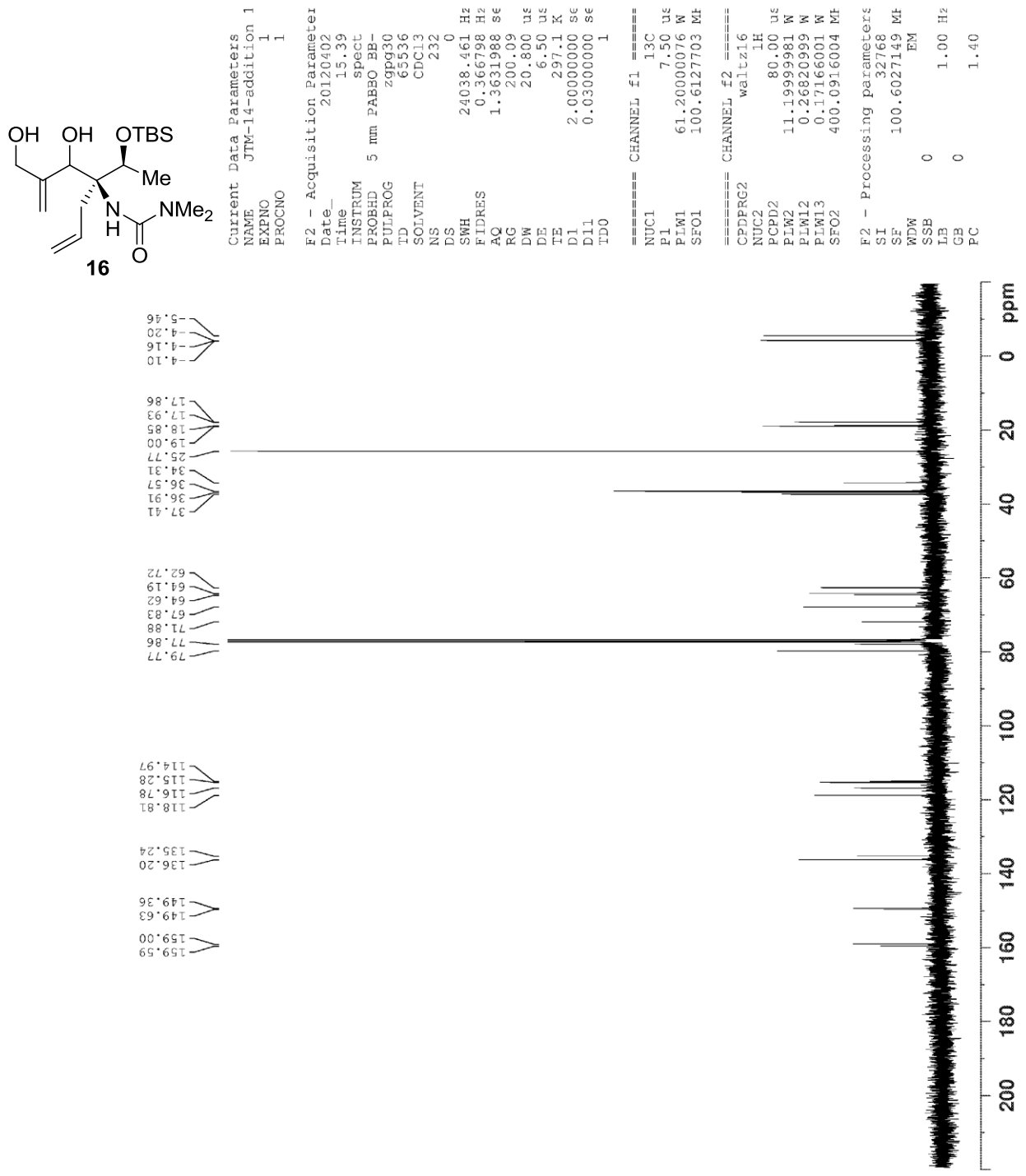

Current Data Parameters
NAME JTM-CHAR-Ald 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110207
Time 8.32
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 57
DW 40.533 usec
DE 10.00 usec
TE 300.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.89999962 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 65536
SF 600.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OTBS
Me
NMe2
14
ppm

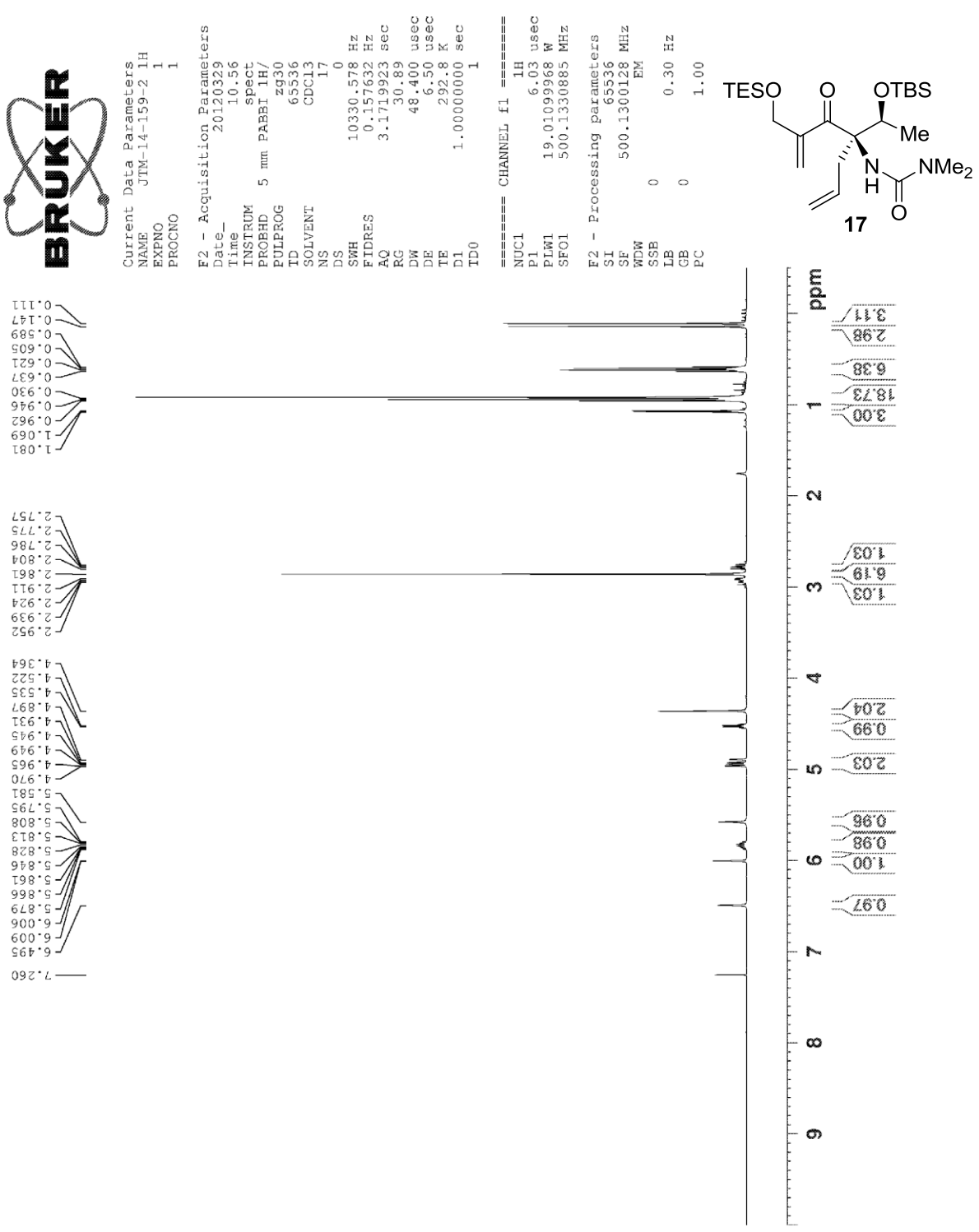

O
OTBS
H
Me
N
H
NMe2
O

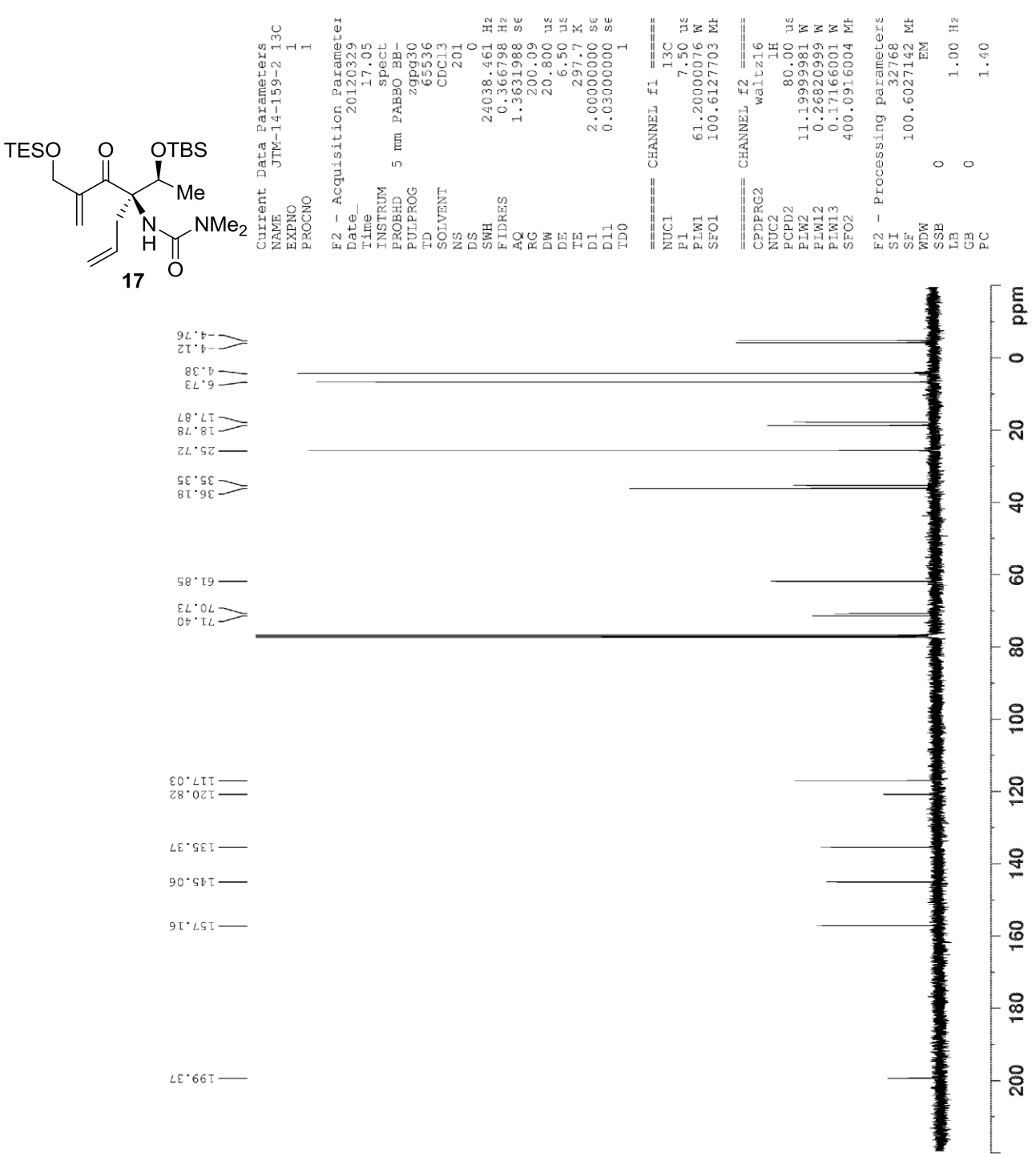
14

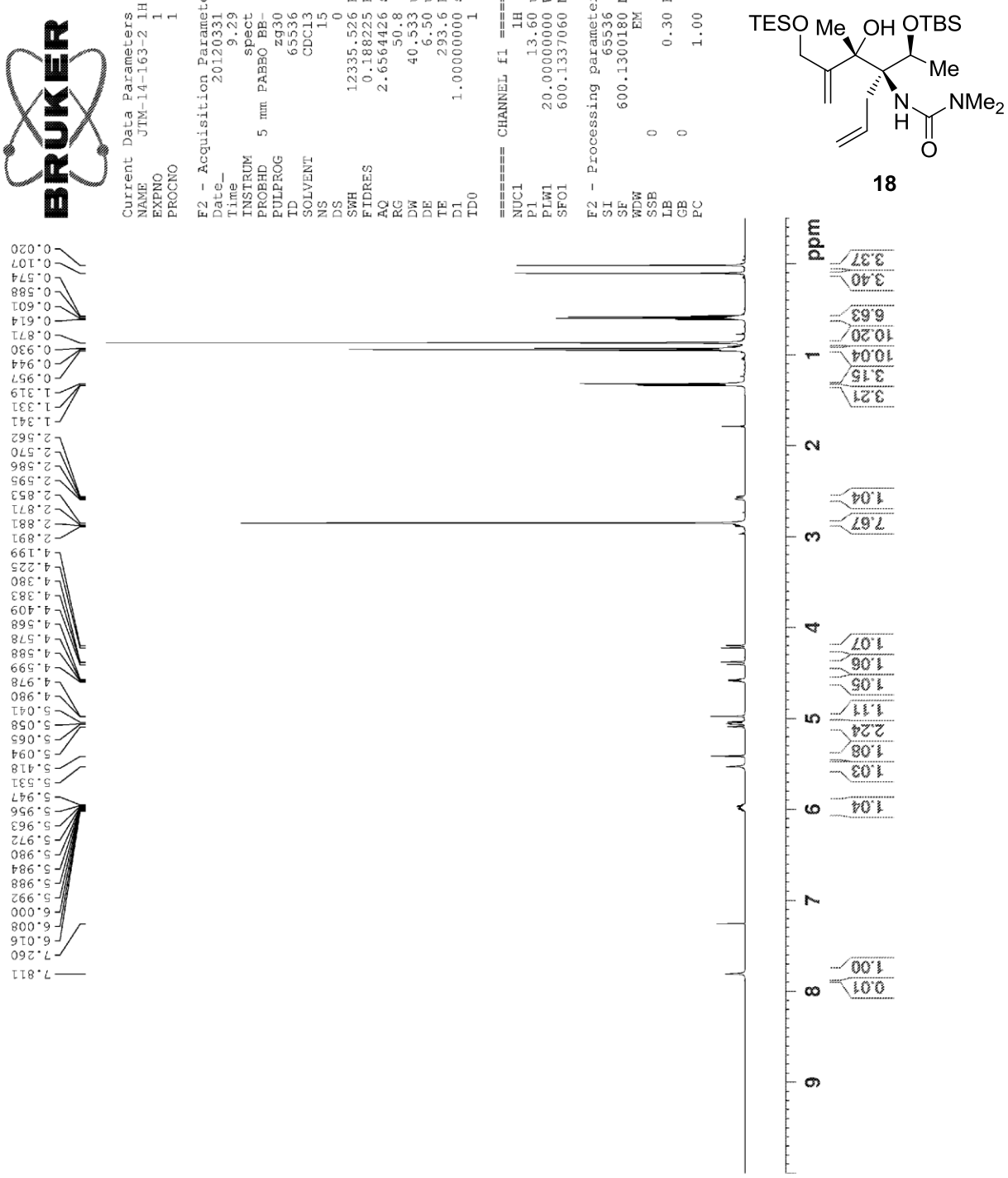

Current Data Parameters
NAME JTM-CHAR-Ald 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110207
Time 8.35
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 53
DS 0
SWH 36057.691 H
FIDRES 0.550197 H
AQ 0.9088159 s
RG 203
DW 13.867 u
DE 18.00 u
TE 300.0 K
D1 2.00000000 s
D11 0.03000000 s
======== CHANNEL f1 ========
NUC1 13C
P1 10.12 u
PLW1 33.00000000 W
SFO1 150.9178981 M
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PLW2 9.89999962 W
PLW12 2.50000000 W
PLW13 0.18043000 W
SFO2 600.1324005 M
F2 - Processing parameters
SI 32768
SF 150.9028138 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40
-4.98
-4.17
17.85
19.00
25.75
33.83
36.20
68.69
70.89
76.79
77.00
77.21
118.37
132.97
156.88
201.96
ppm
0
20
40
60
80
100
120
140
160
180
200

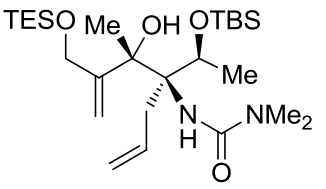

BRUKER
Current Data Parameters
NAME JTM-14-addition
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120402
Time 15.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.37
DW 60.800 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OH
OTBS
Me
N
H
NMe2
O
16
ppm


OH
OH
OTBS
Me
N
H
NMe2
O
16
Current Data Parameters
NAME JTM-14-addition 1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120402
Time 15.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 232
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.1 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027149 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-5.46
-4.20
-4.16
-4.10
17.86
17.93
18.85
19.00
25.77
34.31
36.57
36.91
37.41
62.72
64.19
64.62
67.83
71.88
77.86
79.77
114.97
115.28
116.78
118.81
135.24
136.20
149.36
149.63
159.00
159.59
ppm
0
20
40
60
80
100
120
140
160
180
200

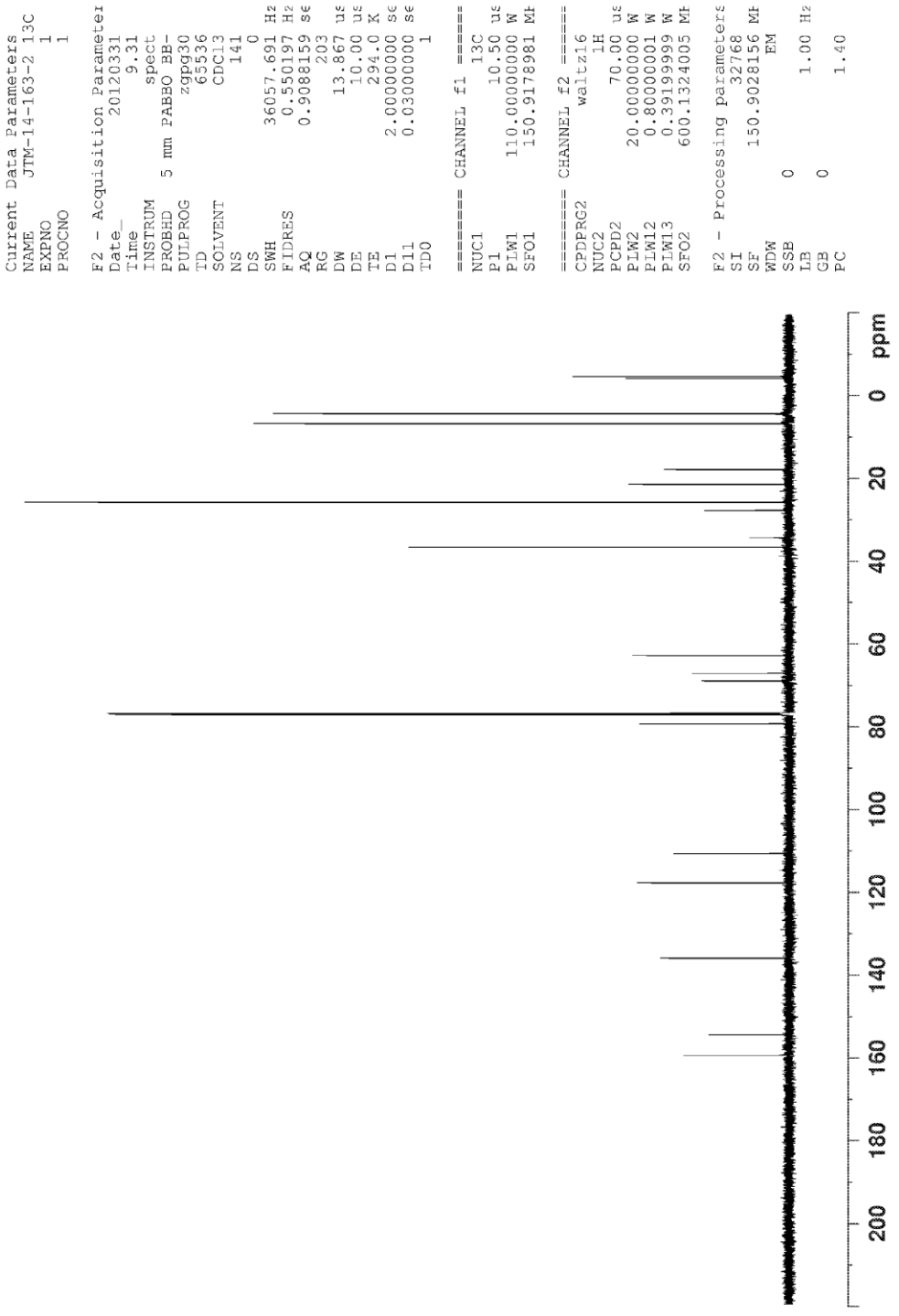

Current Data Parameters
NAME JTM-14-159-2 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120329
Time 10.56
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 30.89
DW 48.400 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.03 usec
PLW1 19.01099968 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 65536
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TESO
O
OTBS
Me
N
H
NMe2
O
17
ppm
1.081
1.069
0.962
0.946
0.930
0.637
0.621
0.605
0.589
0.147
0.111
2.757
2.775
2.786
2.804
2.861
2.911
2.924
2.939
2.952
4.364
4.522
4.535
4.897
4.931
4.945
4.949
4.965
4.970
5.581
5.795
5.808
5.813
5.828
5.846
5.861
5.866
5.879
6.006
6.009
6.495
7.260
3.11
2.98
6.38
18.73
3.00
1.03
6.19
1.03
2.04
0.99
2.03
0.96
0.98
1.00
0.97

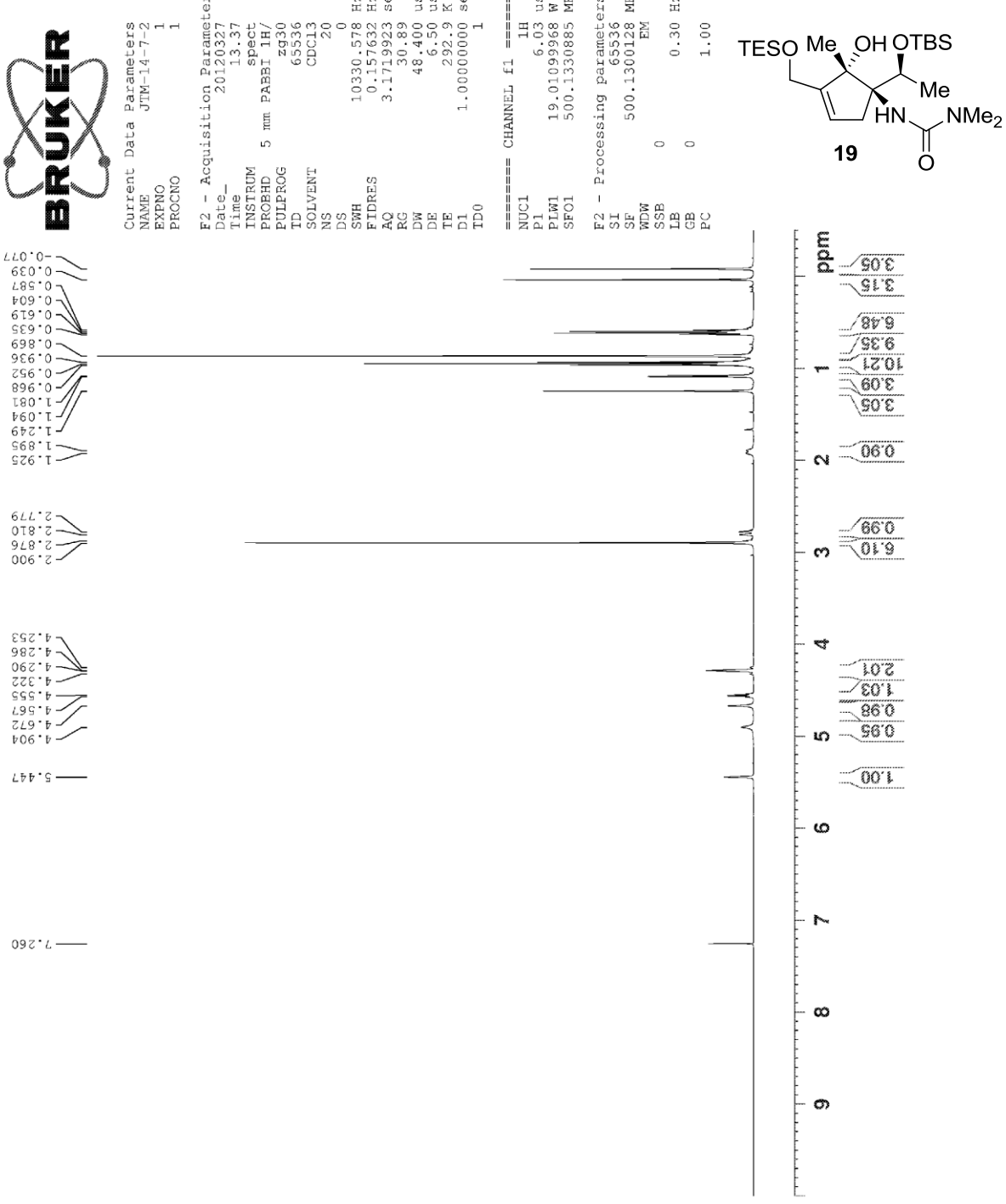

TESO
O
OTBS
Me
N
H
NMe2
O
17
Current Data Parameters
NAME JTM-14-159-2 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120329
Time 17.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.7 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027142 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.76
-4.12
4.38
6.73
17.87
18.78
25.72
35.35
36.18
61.85
70.73
71.40
117.03
120.82
135.37
145.06
157.16
199.37
ppm
0
20
40
60
80
100
120
140
160
180
200

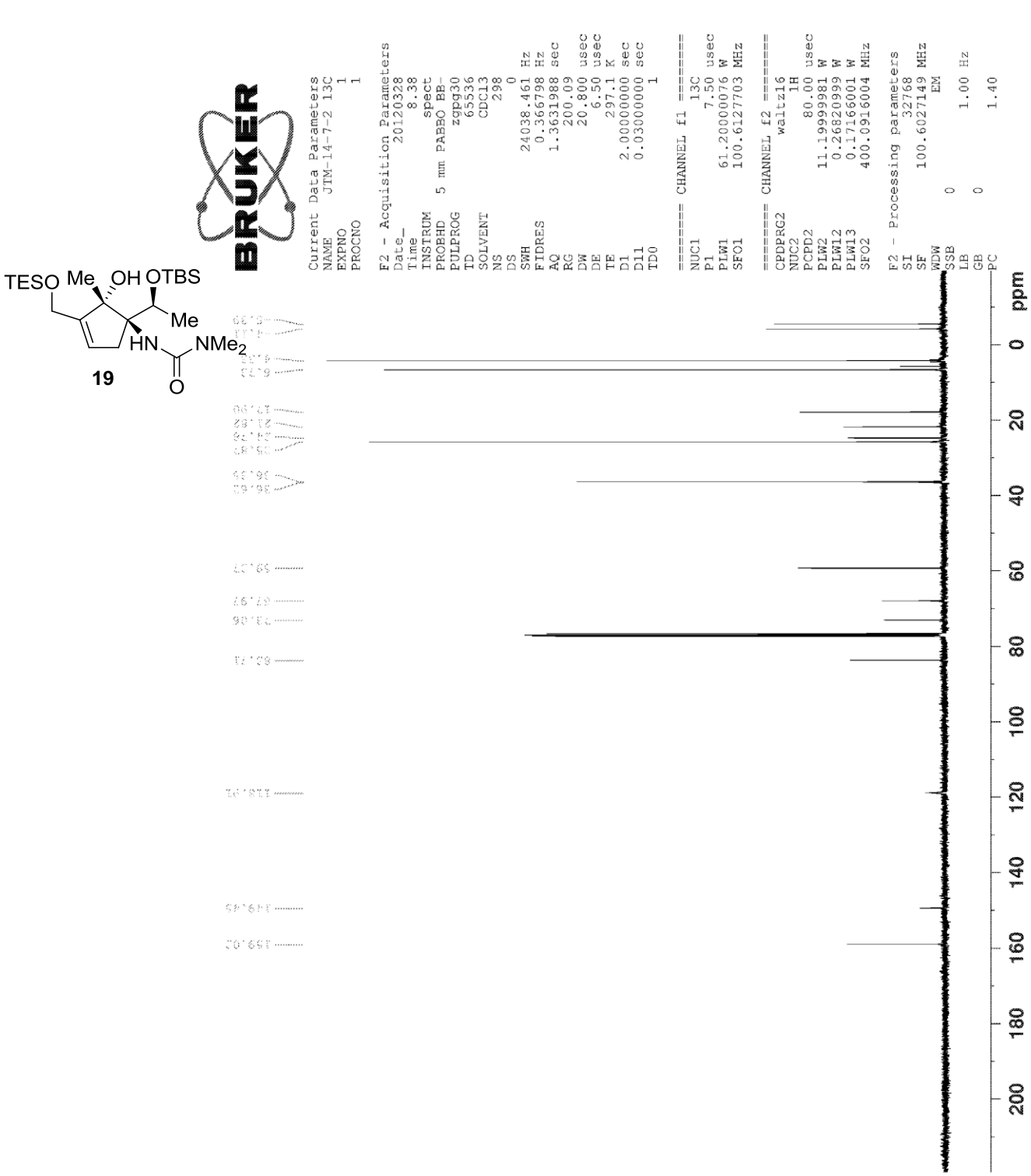

Current Data Parameters
NAME JTM-14-163-2 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120331
Time 9.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 50.8
DW 40.533 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 65536
SF 600.1300180 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TESO Me OH OTBS
Me
N
H
NMe2
O
18
ppm

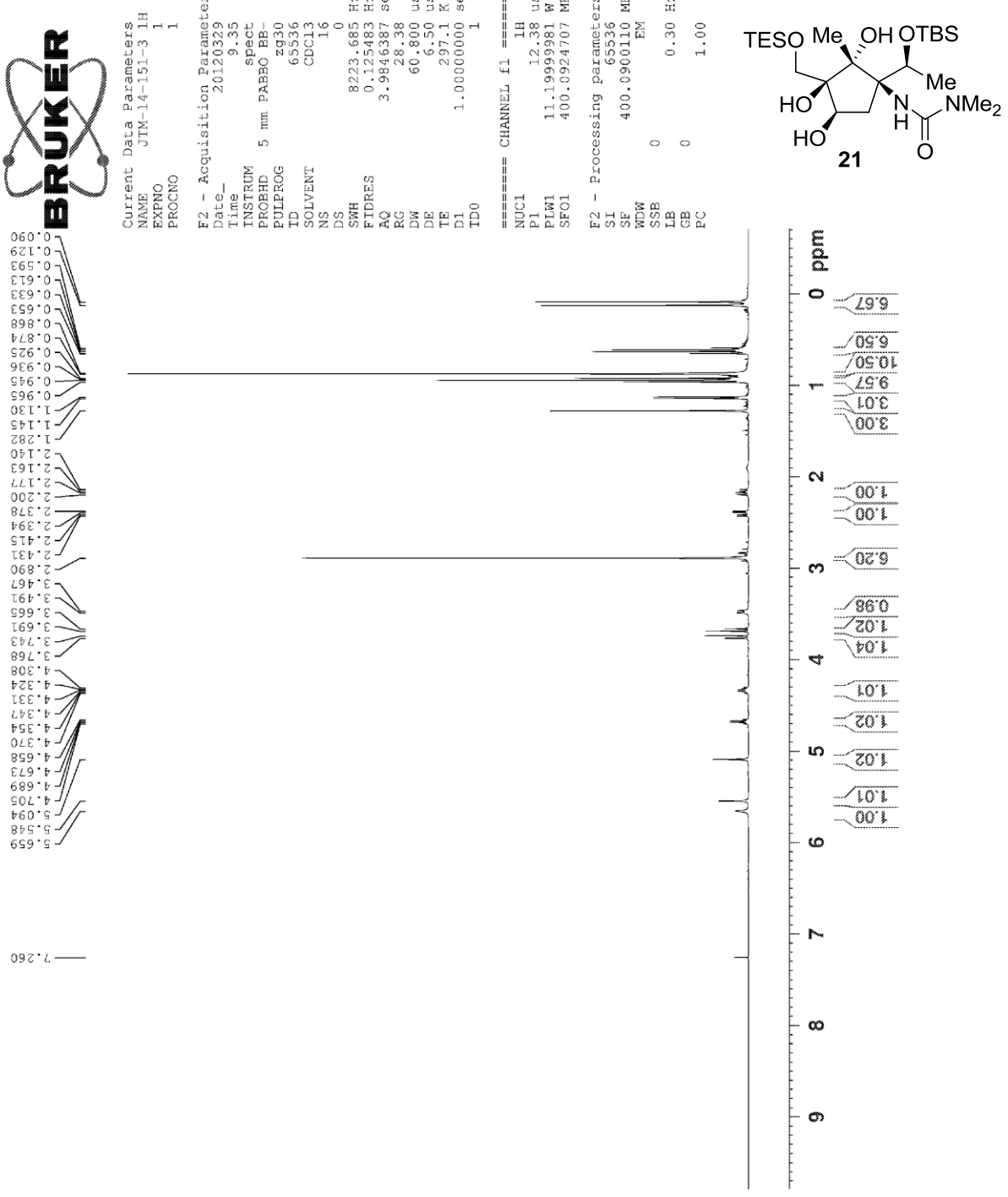

TESO
Me
OH
OTBS
Me
N
H
NMe2
O

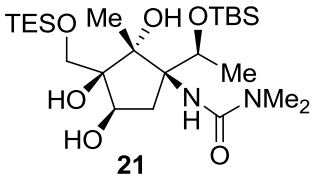
18


Current Data Parameters
NAME JTM-14-163-2 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120331
Time 9.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 141
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 se
RG 203
DW 13.867 us
DE 10.00 us
TE 294.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 10.50 us
PLW1 110.00000000 W
SFO1 150.9178981 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 us
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MH
F2 - Processing parameters
SI 32768
SF 150.9028156 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.55
-4.11
6.81
4.42
17.93
21.49
27.83
34.42
36.69
62.88
67.19
69.02
79.27
110.63
117.68
135.91
154.41
159.40
ppm
0
20
40
60
80
100
120
140
160
180
200


Current Data Parameters
NAME JTM-14-7-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120327
Time 13.37
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 30.89
DW 48.400 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.03 usec
PLW1 19.01099968 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 65536
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TESO Me OH OTBS
Me
HN NMe2
O
19
ppm
7.260
5.447
4.904
4.672
4.567
4.555
4.322
4.290
4.286
4.253
2.900
2.876
2.810
2.779
1.925
1.895
1.249
1.094
1.081
0.968
0.952
0.936
0.869
0.635
0.619
0.604
0.587
0.039
-0.077
3.05
3.15
6.48
9.35
10.21
3.09
3.05
0.90
0.99
6.10
2.01
1.03
0.98
0.95
1.00

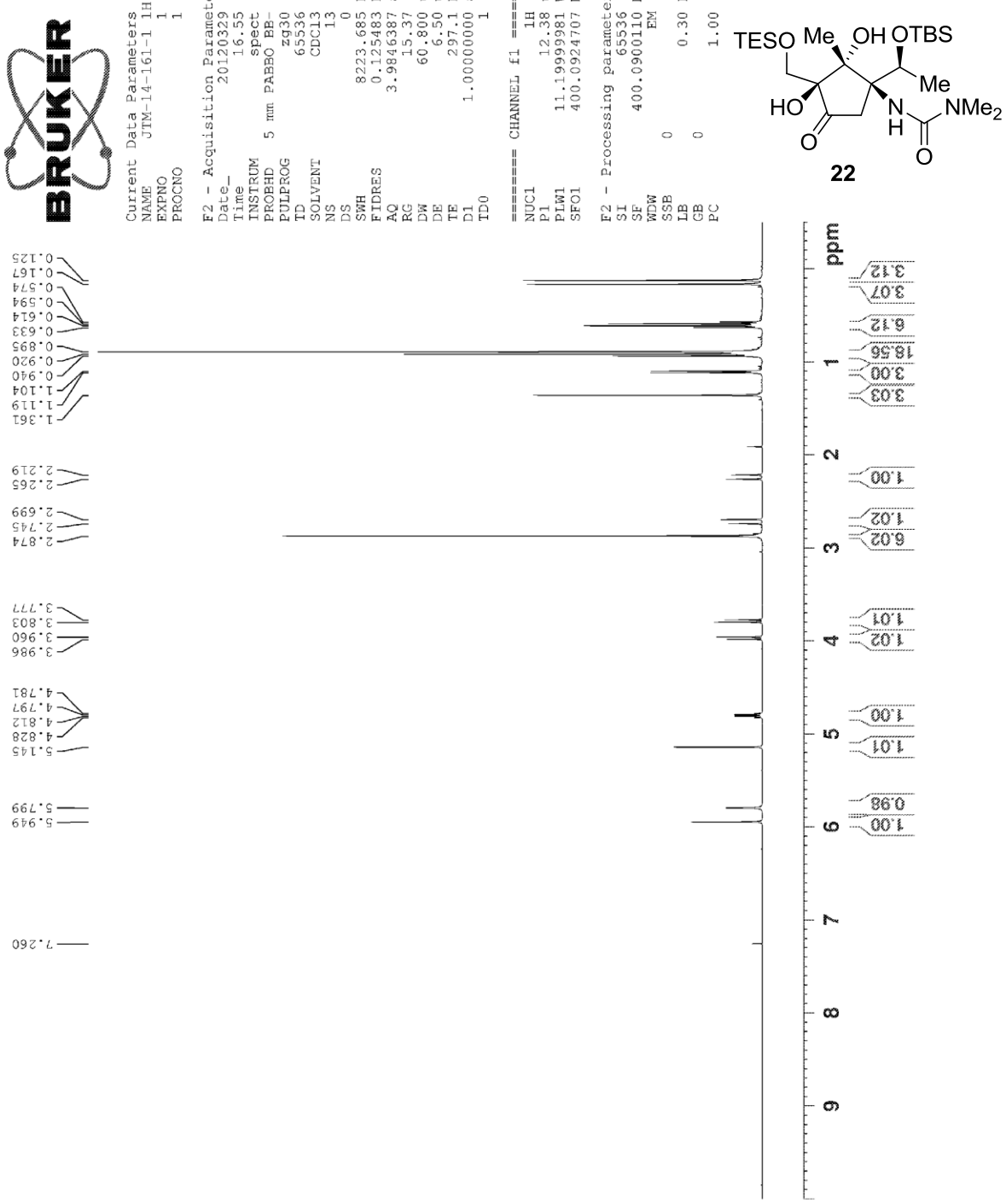

TESO
Me
OH
OTBS
Me
HN
NMe2
O
19
BRUKER
Current Data Parameters
NAME JTM-14-7-2 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120328
Time 8.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 298
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PLW1 61.20000076 W
SFO1 100.6127703 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz
F2 - Processing parameters
SI 32768
SF 100.6027149 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
200
180
160
140
120
100
80
60
40
20
0

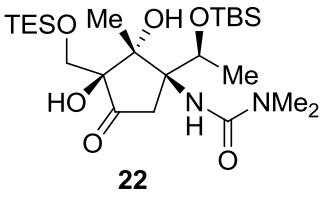

Current Data Parameters
NAME JTM-14-151-3 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120329
Time 9.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 28.38
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TESO
Me
OH
OTBS
Me
HO
N
H
NMe2
HO
O
21
ppm


TESO
Me
OH
OTBS
Me
HO
N
H
NMe2
HO
O

21

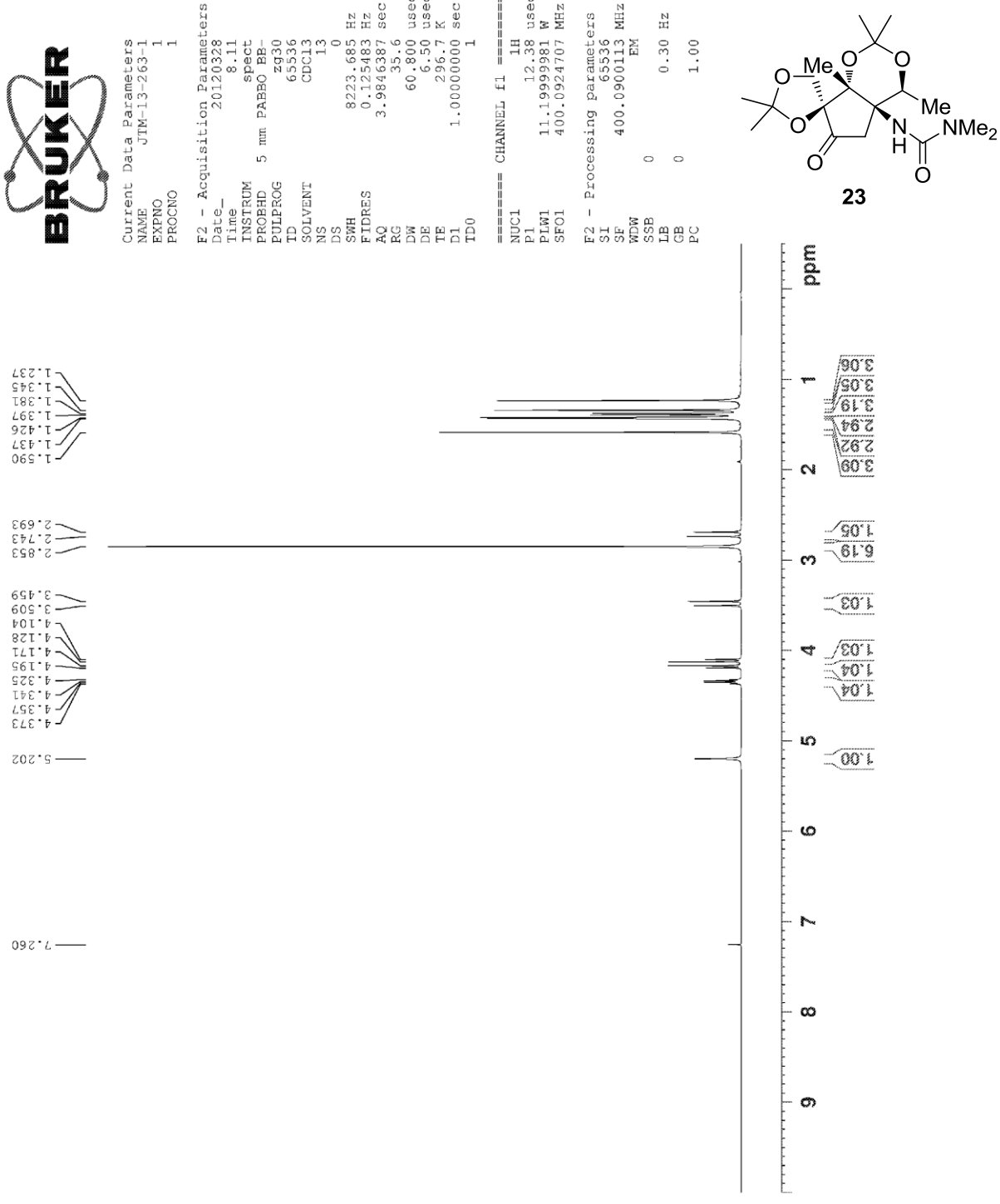
Current Data Parameters
NAME JTM-14-151-3 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120329
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 65
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027164 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.36
-4.24
4.02
6.53
17.81
19.44
19.56
25.90
36.22
37.64
64.05
68.95
70.97
79.57
85.67
158.69
ppm
0
20
40
60
80
100
120
140
160
180
200

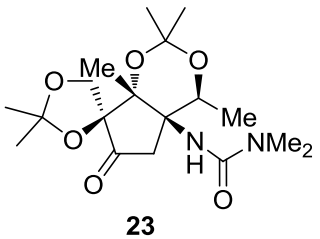

Current Data Parameters
NAME JTM-14-161-1 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120329
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 15.37
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TESO
Me
OH
OTBS
Me
HO
N
H
NMe2
O
O
22
ppm


TESO
Me
OH
OTBS
Me
HO
N
H
NMe2
O
O

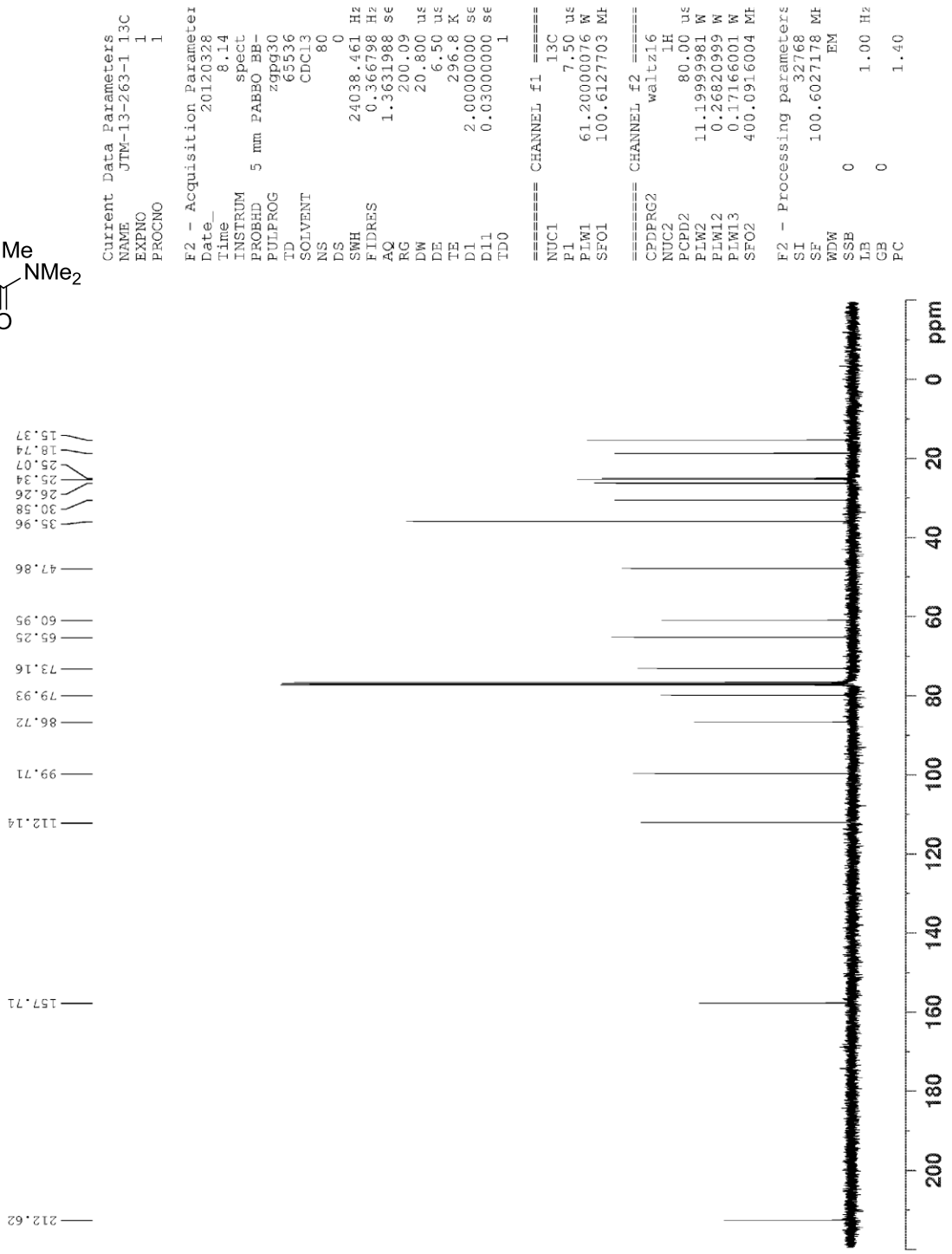
22

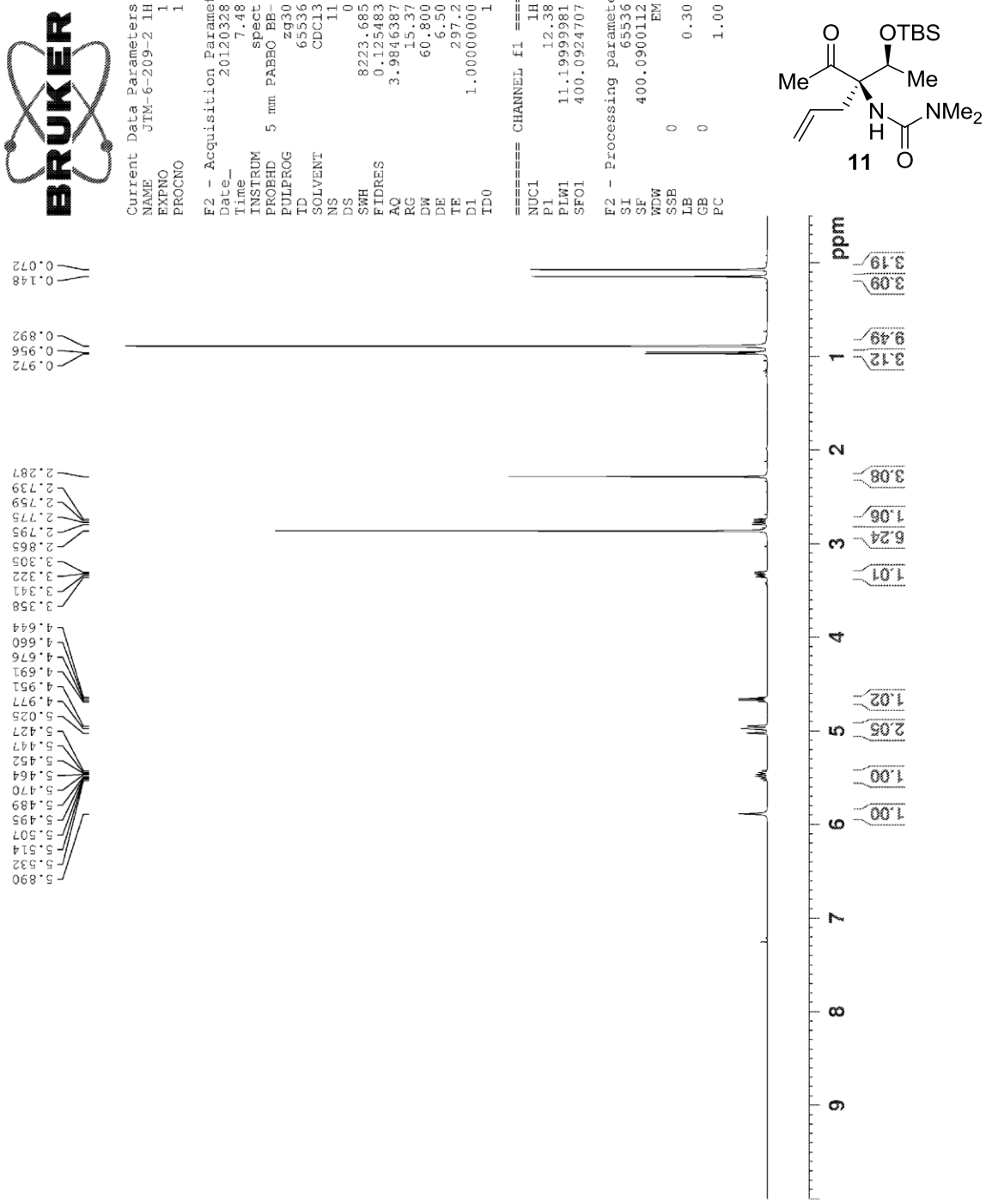
Current Data Parameters
NAME JTM-14-161-1 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120329
Time_ 17.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027193 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.18
3.95
6.46
17.72
19.65
19.67
25.85
36.20
44.57
64.82
66.19
69.87
78.93
83.95
159.02
213.62
ppm
0
20
40
60
80
100
120
140
160
180
200

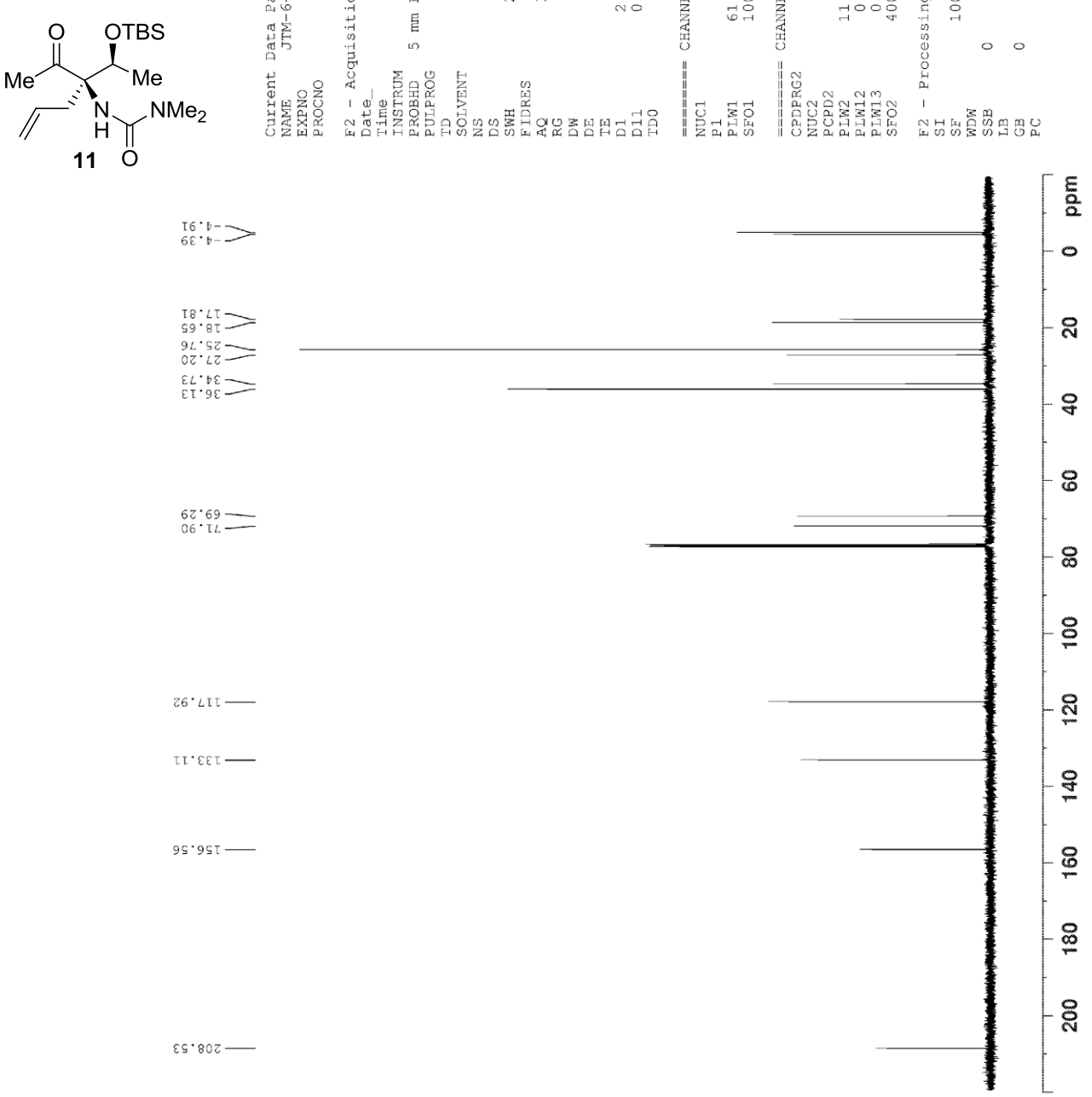

Current Data Parameters
NAME JTM-13-263-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120328
Time_ 8.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 35.6
DW 60.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
NMe2
N
H
O
23
ppm

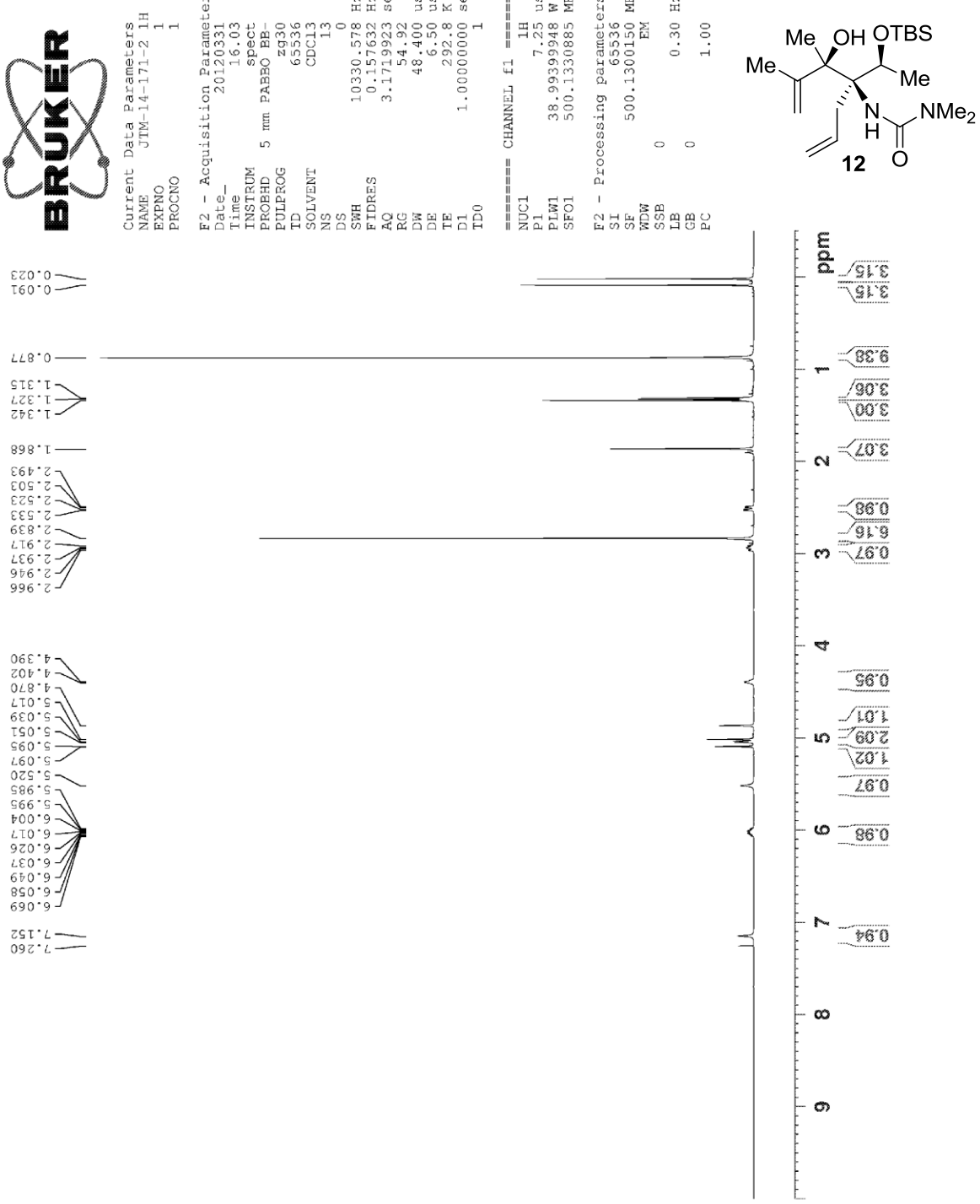

Me
O
O
O
Me
N
H
NMe2
O
O

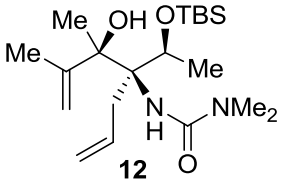
23


Current Data Parameters
NAME JTM-13-263-1 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120328
Time 8.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 296.8 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027178 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
212.62
157.71
112.14
99.71
86.72
79.93
73.16
65.25
60.95
47.86
35.96
30.58
26.26
25.34
25.07
18.74
15.37
200
180
160
140
120
100
80
60
40
20
0
ppm

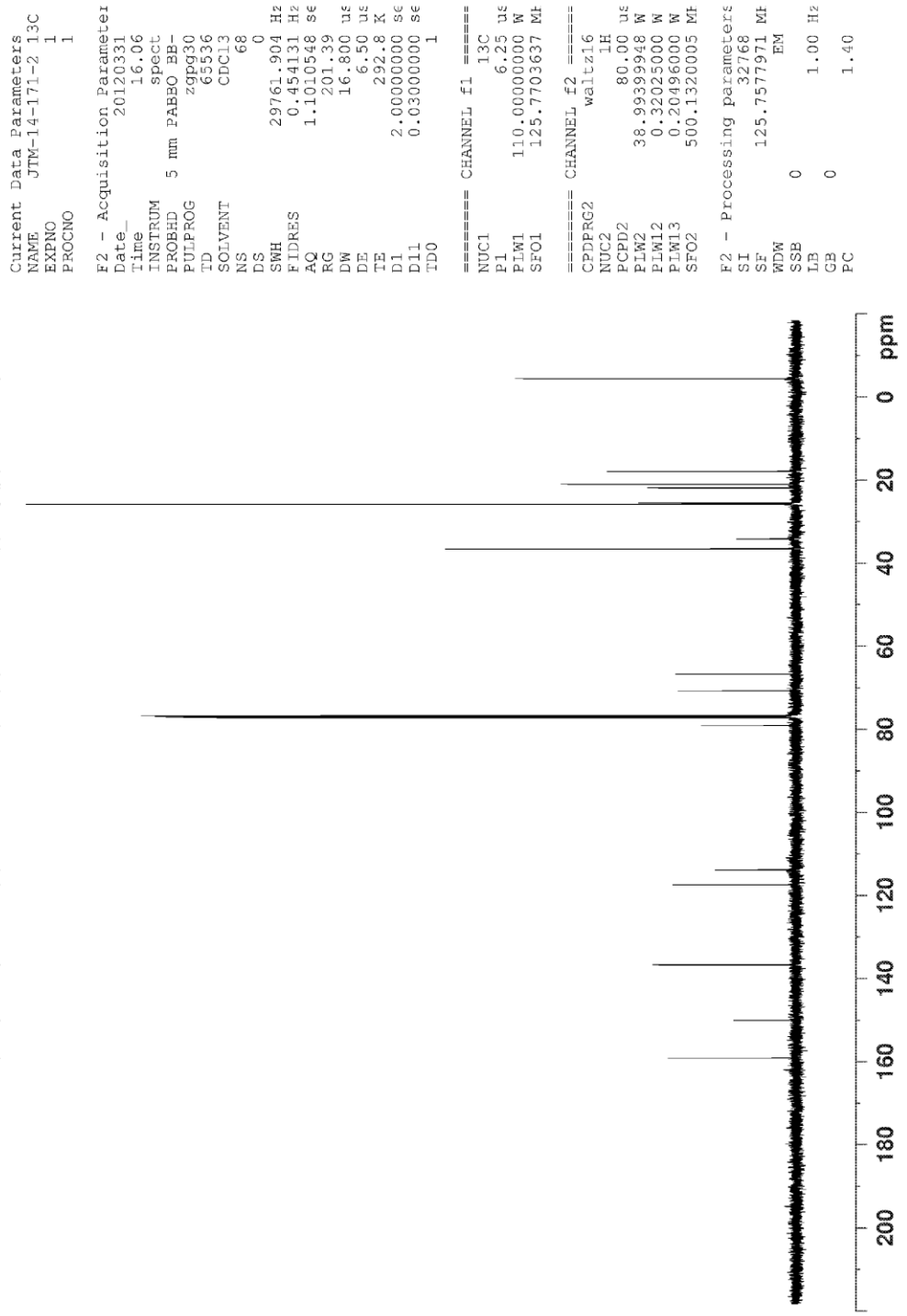

Current Data Parameters
NAME JTM-6-209-2 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120328
Time 7.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 15.37
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
Me
OTBS
Me
N
H
NMe2
O
O
11

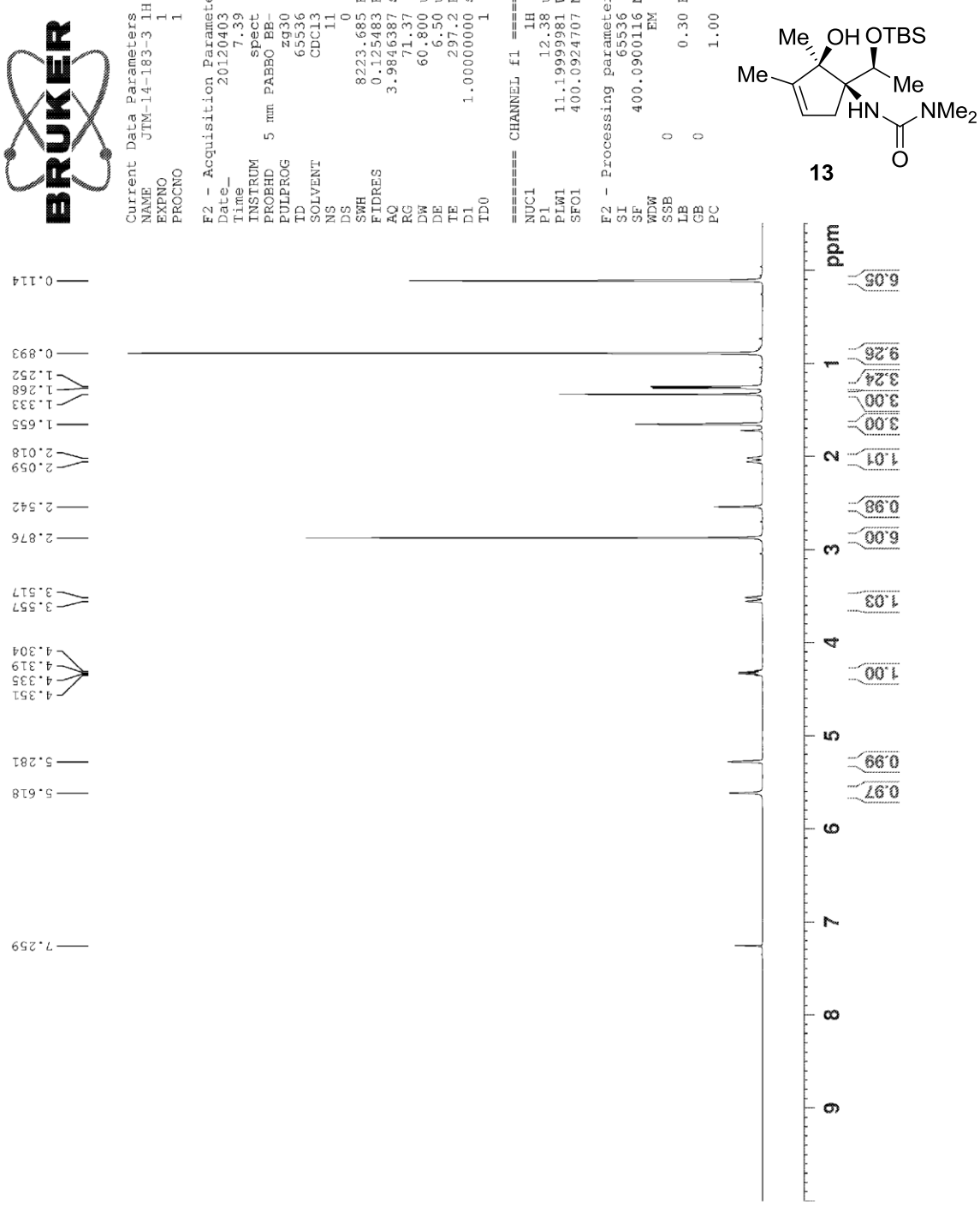

O
OTBS
Me
Me
N
H
NMe2
O
11
Current Data Parameters
NAME JTM-6-209-2 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120328
Time 7.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.7 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027178 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.91
-4.39
17.81
18.65
25.76
27.20
34.73
36.13
69.29
71.90
117.92
133.11
156.56
208.53
ppm
0
20
40
60
80
100
120
140
160
180
200

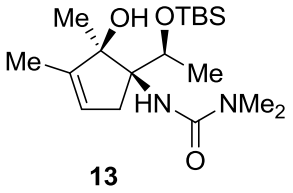

Current Data Parameters
NAME JTM-14-171-2 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120331
Time 16.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 54.92
DW 48.400 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.25 usec
PLW1 38.99399948 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 65536
SF 500.1300150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
Me
OH
OTBS
Me
Me
N
H
NMe2
O
12


Me
OH
OTBS
Me
Me
N
H
NMe2
O

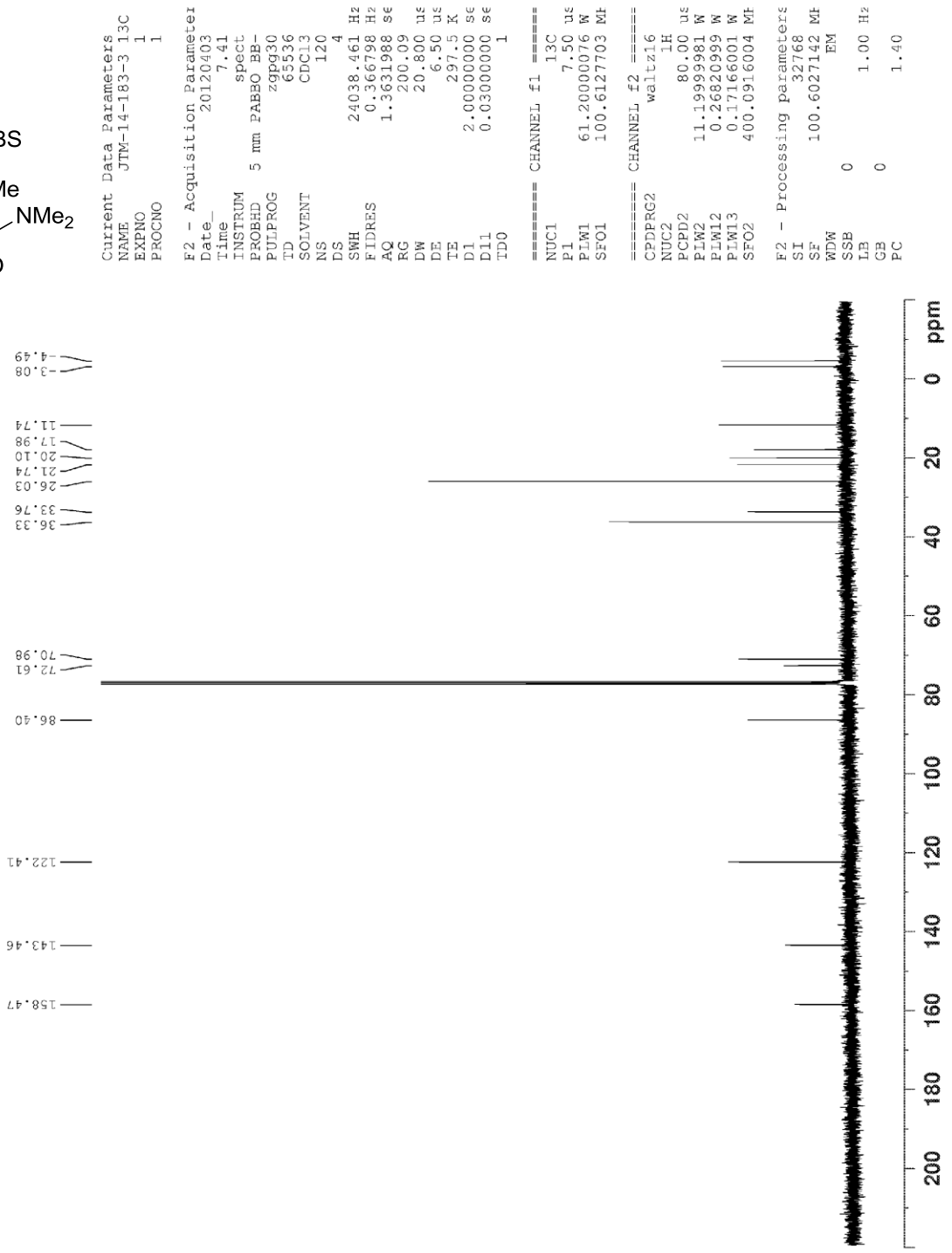
12


Current Data Parameters
NAME JTM-14-171-2 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120331
Time_ 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 se
RG 201.39
DW 16.800 us
DE 6.50 us
TE 292.8 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 6.25 us
PLW1 110.00000000 W
SFO1 125.7703637 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 38.99399948 W
PLW12 0.32025000 W
PLW13 0.20496000 W
SFO2 500.1320005 MH
F2 - Processing parameters
SI 32768
SF 125.7577971 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.39
-4.37
17.96
21.04
21.92
25.56
25.86
34.21
36.60
66.75
70.79
79.15
113.81
117.40
136.70
150.06
159.12
ppm
0
20
40
60
80
100
120
140
160
180
200


Current Data Parameters
NAME JTM-14-183-3 1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120403
Time 7.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.37
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
OH
OTBS
Me
Me
HN
NMe2
O
13
ppm


Me
OH
OTBS
Me
Me
HN
NMe2
O

13


Current Data Parameters
NAME JTM-14-183-3 13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20120403
Time 7.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 297.5 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MH
F2 - Processing parameters
SI 32768
SF 100.6027142 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.49
-3.08
11.74
17.98
20.10
21.74
26.03
33.76
36.33
70.98
72.61
86.40
122.41
143.46
158.47
ppm
0
20
40
60
80
100
120
140
160
180
200