Supporting Information – Table of Contents

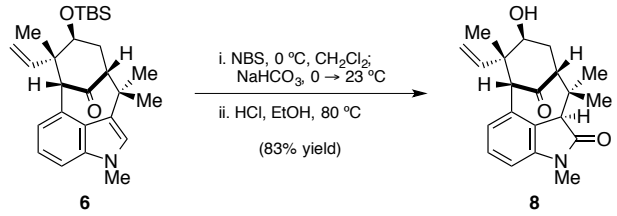
Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled or passed through activated alumina columns). All commercially available reagents were used as received unless otherwise specified. $Mn(OAc)_3$ (dried *in vacuo* over P_2O_5) and $LiAlD_4$ were obtained from Acros. AgOTf and $CuBr_2$ were obtained from Strem. Bathophenanthroline was obtained from Alfa Aesar. 2-Iodoxybenzoic acid (IBX)[1] and Dess–Martin periodinane[2] were prepared from known literature procedures. 1,4-dioxane was distilled from Na/benzophenone and stored in a Schlenk tube prior to use. Unless stated otherwise, reactions were performed at room temperature (RT, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, iodine, and potassium permanganate staining. Silicycle silica gel 60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (500 MHz). Data for ^{1}H spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), integration and are referenced to the residual solvent peak 7.26 ppm for $CDCl_3$, 5.32 ppm for CD_2Cl_2, 4.79 ppm for D_2O, and 1.94 ppm for CD_3CN. Data for ^{2}H NMR spectra are reported as follows: chemical shift (δ ppm, at 77 MHz), multiplicity, coupling constant, integration and are referenced to the residual solvent peak 7.26 ppm for $CDCl_3$. ^{13}C NMR spectra are reported in terms of chemical shift (at 125 MHz) and are referenced to the residual solvent peak 118.26 ppm for CD_3CN, 77.16 ppm for $CDCl_3$, and 53.84 for CD_2Cl_2. IR spectra were recorded on a Perkin-Elmer 100 spectrometer and are reported in terms of frequency absorption (cm^{-1}). Optical rotations were measured with a Rudolph Autopol III Automatic Polarimeter. Uncorrected melting points were measured using a Mel-Temp II melting point apparatus with a Fluke 50S thermocouple and a Digimelt MPA160 melting point apparatus. High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility.

Complete Reference 4r from manuscript:

D. B. Freeman, A. A. Holubec, M. W. Weiss, J. A. Dixon, A. Kafefuda, M. Ohtsuka, M. Inoue, R. G. Vaswani, H. Ohki, B. D. Doan, S. E. Reisman, B. M. Stoltz, J. J. Day, R. N. Tao, N. A. Dieterich, J. L. Wood, *Tetrahedron* **2010**, *66*, 6647–6655.

[1] M. Frigerio, M. Santagostino, S. Sputore, *J. Org. Chem.* **1999**, *64*, 4537–4538.
[2] C. Niu, T. Pettersson, M. J. Miller, *J. Org. Chem.* **1996**, *61*, 1014–1022.

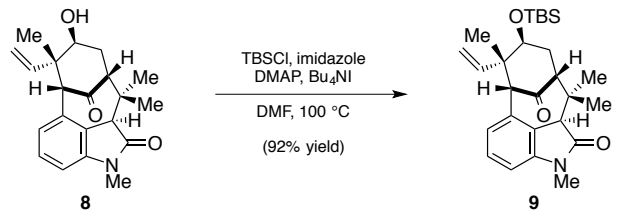
Experimental Procedures.



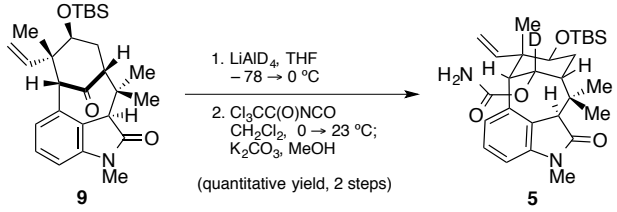
6 **8**

Oxindole 8. To a solution of indole **6**[3] (200 mg, 0.457 mmol, 1.0 equiv) in CH_2Cl_2 (9.2 mL) at 0 ºC was added NBS (82.0 mg, 0.462 mmol, 1.01 equiv) in one portion. The reaction vial was flushed with N_2, and allowed to stir at 0 ºC. After 15 min, solid $NaHCO_3$ (200 mg, 100 wt %) was added in one portion. The reaction was removed from the 0 ºC bath and allowed to stir at room temperature for 5 min. The resulting suspension was evaporated under reduced pressure. Absolute ethanol (9.2 mL) and concentrated aqueous HCl (9.2 mL) were added. After heating to 80 ºC for 17 h, the reaction was cooled to room temperature and transferred to a separatory funnel with H_2O (15 mL) and EtOAc (15 mL). To the funnel was added solid $NaHCO_3$ until gas evolution was no longer observed. The resulting biphasic mixture was extracted with EtOAc (3 x 15 mL) and the organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (1:1:1 hexanes:CH_2Cl_2:Et_2O) to afford oxindole **8** (130 mg, 83% yield) as a brown solid. Oxindole **8**: mp: 203.2 ºC; R_f 0.23 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.22 (dd, *J* = 7.8, 7.7, 1H), 6.73 (d, *J* = 7.7, 1H), 6.67 (d, *J* = 7.8, 1H), 5.56 (dd, *J* = 17.5, 10.9, 1H), 5.22 (d, *J* = 10.9, 1H), 5.19 (d, *J* = 17.5, 1H), 4.22 (ddd, *J* = 12.2, 5.6, 2.8, 1H), 3.73 (s, 1H), 3.32 (d, *J* = 1.4, 1H), 3.19 (s, 3H), 2.59 (dd, *J* = 14.9, 5.6, 1H) 2.53 (d, *J* = 8.2, 1H), 2.07 (ddd, *J* = 14.9, 12.2, 8.2, 1H), 1.65 (s, 3H), 1.58 (d, *J* = 2.8, 1H), 1.17 (s, 3H), 0.68 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$, 20 of 21 observed): δ 208.3, 174.9, 144.8, 142.0, 130.7, 128.5, 127.2, 125.2, 116.6, 107.2, 68.4, 68.2, 62.2, 53.5, 50.1, 40.2, 30.0, 26.4, 26.3, 22.6; IR (film): 1703, 1687, 1611, 1588, 1461 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{21}H_{25}NO_3Na$, 362.1732; found 362.1737; $[\alpha]^{25.2}_D$ +6.60° (*c* = 1.000, $CHCl_3$).

[3] A. D. Huters, K. W. Quasdorf, E. D. Styduhar, N. K. Garg, *J. Am. Chem. Soc.* **2011**, *133*, 15797–15799.



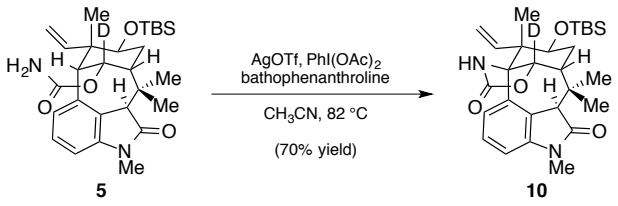
Silyl ether 9. To a solution of oxindole **8** (609 mg, 1.80 mmol, 1.0 equiv) in DMF (18.0 mL) was added imidazole (611 mg, 8.97 mmol, 5.0 equiv), DMAP (219 mg, 1.79 mmol, 1.0 equiv), and tetrabutylammonium iodide (663 mg, 1.79 mmol, 1.0 equiv). The resulting solution was stirred at room temperature for 5 min, and then TBSCl (809 mg, 5.37 mmol, 3.0 equiv) was added in one portion. The flask was fitted with a reflux condenser, flushed with N_2, and then heated to 100 °C. After 25 h, the reaction mixture was cooled to room temperature and transferred to a separatory funnel with EtOAc (50 mL), H_2O (20 mL), and a solution of saturated aqueous NH_4Cl (20 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 50 mL). The organic layers were combined, washed with H_2O (3 x 50 mL), dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (9:1 hexanes:EtOAc) to afford silyl ether **9** (746 mg, 92% yield) as a white solid. Silyl ether **9**: mp: 184.5 ºC; R_f 0.64 (2:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.21 (dd, *J* = 7.9, 7.7, 1H), 6.73 (d, *J* = 7.7, 1H), 6.66 (d, *J* = 7.9, 1H), 5.40 (dd, *J* = 17.5, 10.8, 1H), 5.15–5.00 (m, 2H), 4.17 (dd, *J* = 11.6, 5.5, 1H), 3.62 (s, 1H), 3.26 (s, 1H), 3.21 (s, 3H), 2.47 (d, *J* = 8.2, 1H), 2.40 (dd, *J* = 15.1, 5.5, 1H), 2.10 (ddd, *J* = 15.1, 11.6, 8.2, 1H), 1.62 (s, 3H), 1.17 (s, 3H), 0.82 (s, 9H), 0.67 (s, 3H), 0.04 (s, 3H), -0.09 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$, 24 of 25 observed): δ 208.6, 175.0, 144.6, 143.6, 131.2, 128.4, 127.4, 124.9, 114.6, 107.1, 70.0, 69.2, 62.5, 53.7, 50.3, 40.1, 32.0, 26.4, 25.8, 22.4, 18.1, 16.1, -3.8, -4.4; IR (film): 1707, 1689, 1609, 1590, 1465 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{27}H_{39}NO_3SiNa$, 476.2597; found 476.2600; $[\alpha]^{26.1}_D$ +12.60° (*c* = 1.000, $CHCl_3$).



Carbamate 5. To a solution of silyl ether **9** (178 mg, 0.393 mmol, 1.0 equiv) in THF (13.0 mL) at –78 °C was added a solution of $LiAlD_4$ (1.0 M in THF, 1.18 mL, 1.18 mmol, 3.0 equiv) in a dropwise manner. After stirring at –78 °C for 20 min, the solution was then allowed to warm to 0 ºC. After 20 min, the reaction was quenched at 0 °C with slow addition of a saturated aqueous solution of Rochelle's salt (10 mL), and then allowed to warm to 23 ºC. The resulting biphasic mixture was stirred at room temperature for 1 h, transferred to a separatory funnel with EtOAc (25 mL) and H_2O (20 mL), and extracted with EtOAc (3 x 25 mL). The organic layers were combined, dried

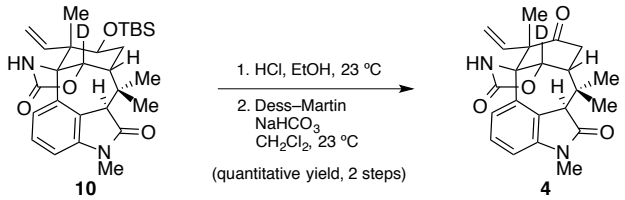
over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was used in the subsequent step without further purification.

To a flask containing the crude residue from the previous step was added CH_2Cl_2 (7.85 mL), cooled to 0 ºC, followed by addition of trichloroacetyl isocyanate (58 μL, 0.490 mmol, 1.25 equiv) in a dropwise manner. The resulting mixture was allowed to stir at 0 °C for 5 min, and then at room temperature for 20 min. The solvent was evaporated under reduced pressure. To the resulting residue was added MeOH (7.85 mL) followed by solid K_2CO_3 (298 mg, 2.16 mmol, 5.5 equiv) in one portion. The reaction was flushed with N_2 and left to stir at room temperature for 80 min. The reaction was quenched with saturated aqueous NH_4Cl (7 mL), and the resulting biphasic mixture was transferred to a separatory funnel with EtOAc (25 mL) and H_2O (20 mL). After extracting with EtOAc (3 x 25 mL), the organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (2:1 hexanes:EtOAc) to afford carbamate **5** (203 mg, quant. yield, over two steps) as a white solid. Carbamate **5**: mp: 106.1 ºC; R_f 0.47 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.15 (ddd, *J* = 7.8, 7.7, 0.7, 1H), 6.70 (dd, *J* = 7.7, 0.7, 1H), 6.63 (d, *J* = 7.8, 1H), 5.23 (dd, *J* = 17.4, 10.7, 1H), 5.03 (dd, *J* = 17.4, 1.3, 1H), 4.93 (dd, *J* = 10.7, 1.3, 1H), 4.34 (s, 2H), 4.03 (dd, *J* = 12.5, 5.1, 1H), 3.60 (s, 1H), 3.20 (s, 3H), 2.83 (s, 1H) 2.30 (ddd, *J* = 15.0, 5.1, 1.9, 1H), 2.25 (d, *J* = 6.1, 1H), 1.98 (ddd, *J* = 15.0, 12.5, 6.1, 1H), 1.58 (s, 3H), 1.36 (s, 3H), 0.88 (s, 3H), 0.82 (s, 9H), 0.03 (s, 3H), -0.12 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.51 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.8, 156.1, 146.3, 144.6, 138.1, 128.6, 127.4, 125.3, 113.7, 106.3, 74.5 (t, $J_{C–D}$ = 22.1), 70.9, 56.8, 55.9, 49.0, 48.9, 37.3, 32.4, 29.2, 26.3, 25.9, 24.3, 18.1, 17.1, -3.8, -4.3; IR (film): 2956, 2923, 2857, 1728, 1701, 1607, 1596, 1463 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{28}H_{41}N_2O_4SiDNa$, 522.2874; found 522.2872; $[\alpha]^{26.5}_D$ +11.40° (*c* = 1.000, $CHCl_3$).



Oxazolidinone 10. A 1-dram vial containing CH_3CN, a second 1-dram vial charged with bathophenanthroline (10.6 mg, 0.0319 mmol, 0.5 equiv), and a third 1-dram vial containing carbamate **5** (33.0 mg, 0.0660 mmol, 1.0 equiv) and $PhI(OAc)_2$ (42.5 mg, 0.132 mmol, 2.0 equiv) were transferred into the glovebox. AgOTf (8.5 mg, 0.033 mmol, 0.5 equiv) and CH_3CN (950 μL) were added to the vial containing the bathophenanthroline, and the resulting suspension was allowed to stir at room temperature for 20 min. Next, CH_3CN (950 μL) was added to the vial containing the carbamate, and the AgOTf/bathophenanthroline suspension was also added to this vial. The vial was then sealed, removed from the glovebox, and the resulting mixture was heated to 82 °C. After 24 h, the reaction was cooled to room temperature and filtered by passage over a plug of silica gel (EtOAc eluent, 10 mL). The filtrate was evaporated

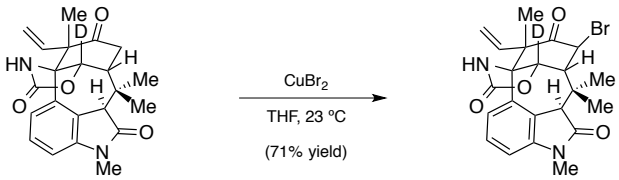
under reduced pressure, and the resulting residue was purified by preparative thin layer chromatography (2:1 hexanes:EtOAc) to afford oxazolidinone **10** (23 mg, 70% yield) as a yellow solid and recovered silyl ether **9** (2 mg, 7% yield) as a white solid. Oxazolidinone **10**: mp: 288.0 ºC (decomp.); R_f 0.50 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.21 (app t, *J* = 8.0, 1H), 6.77–6.73 (m, 2H), 6.54 (s, 1H), 5.19–5.09 (m, 3H), 3.87 (dd, *J* = 11.7, 5.2, 1H), 3.47 (s, 1H), 3.22 (s, 3H), 2.44 (d, *J* = 6.7, 1H), 2.31 (dd, *J* = 15.2, 5.2, 1H), 2.01 (ddd, *J* = 15.2, 11.7, 6.7, 1H), 1.60 (s, 3H), 1.33 (s, 3H), 0.96 (s, 3H), 0.80 (s, 9H), 0.00 (s, 3H), -0.16 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 4.97 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$, 25 of 26 observed): δ 174.8, 159.1, 144.1, 144.0, 137.6, 128.0, 125.9, 124.7, 116.3, 107.4, 72.2, 69.9, 55.2, 49.9, 46.8, 37.7, 32.3, 28.0, 26.4, 25.8, 22.8, 18.1, 12.9, -3.8, -4.4; IR (film): 1758, 1708, 1691, 1607, 1590, 1461 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{28}H_{39}N_2O_4SiDNa$, 520.2718; found 520.2719; $[\alpha]^{27.0}_D$ +1.40° (*c* = 1.000, $CHCl_3$).



Ketone 4. A flask was charged with oxazolidinone **10** (576 mg, 1.16 mmol, 1.0 equiv), absolute ethanol (23.0 mL), and concentrated aqueous HCl (23.0 mL). After stirring at 23 ºC for 1 h, the reaction mixture was transferred to a separatory funnel with H_2O (50 mL) and EtOAc (50 mL). To the funnel was added solid $NaHCO_3$ until no more gas evolution was observed. The resulting biphasic mixture was extracted with EtOAc (3 x 50 mL) and the organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was used in the subsequent step without further purification.

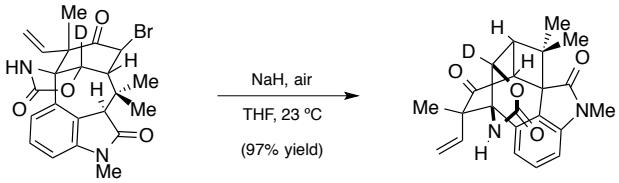
To a flask containing the crude product from the previous step was added solid $NaHCO_3$ (487 mg, 5.80 mmol, 5.0 equiv) in one portion. The reaction vessel was flushed with N_2, and then CH_2Cl_2 (11.6 mL) was added. To the resulting suspension was added the Dess–Martin periodinane reagent (645 mg, 1.52 mmol, 1.3 equiv) in one portion. The flask was flushed with N_2, and the reaction mixture was allowed to stir at room temperature. After 1 h, the reaction mixture was diluted with a 1:1 mixture of saturated aqueous $NaHCO_3$ and saturated aqueous $Na_2S_2O_3$ (10.0 mL). The resulting biphasic mixture was vigorously stirred until both layers appeared clear. The mixture was then transferred to a separatory funnel with EtOAc (30 mL) and H_2O (30 mL), and then extracted with EtOAc (3 x 30 mL). The organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (1:1 hexanes:EtOAc) to afford ketone **4** (454 mg, quant. yield, over two steps) as a yellow solid. Ketone **4**: mp: 293.5 ºC; R_f 0.42 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.20 (app t, *J* = 8.0, 1H), 7.10 (s, 1H), 6.77–6.73 (m, 2H), 5.34 (dd, *J* = 17.6, 10.9, 1H), 5.22–5.16 (m, 2H), 3.34 (s, 1H), 3.18 (s, 3H), 3.17–3.06 (m, 2H), 2.86–2.82 (m, 1H), 1.62 (s, 3H), 1.56 (s, 3H), 0.92 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.45 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$, 21 of 22 observed): δ 209.8,

174.3, 159.3, 144.5, 137.9, 136.0, 128.5, 124.3, 123.4, 116.8, 108.0, 70.8, 57.4, 51.9, 48.2, 40.2, 38.4, 27.3, 26.5, 22.5, 19.8; IR (film): 1760, 1752, 1689, 1611, 1590 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{23}N_2O_4DNa$, 404.1696; found 404.1700; $[\alpha]^{27.3}_D$ –26.67° (*c* = 0.870, $CHCl_3$).



4 11

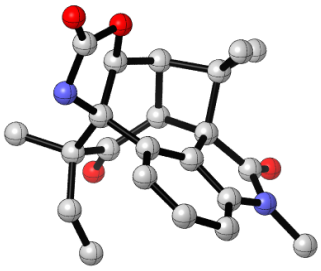
Bromoketone 11. A 1-dram vial was charged with ketone **4** (50.0 mg, 0.131 mmol, 1.0 equiv) and transferred into the glovebox. $CuBr_2$ (59.0 mg, 0.264 mmol, 2.0 equiv) was then added and the vial was removed from the glovebox. THF (2.1 mL) was added and the reaction vial was sealed and left to stir at room temperature. After 19 h, the reaction mixture was filtered by passage over a plug of celite (THF eluent, 10 mL). The filtrate was collected in a test tube, diluted with H_2O (5 mL), and then extracted with $CHCl_3$ (3 x 5 mL). The organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was adsorbed onto silica gel (2 mL) and purified by flash chromatography (4:1 hexanes:acetone) to afford bromoketone **11** (42.5 mg, 71% yield) as a white solid. Bromoketone **11**: mp: 273.0 ºC (decomp.); R_f 0.62 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_3CN): δ 7.32 (ddd, *J* = 8.2, 7.6, 0.8, 1H), 7.21 (s, 1H), 6.89–6.84 (m, 2H), 5.27–5.19 (m, 1H), 5.12–5.03 (m, 2H), 3.10 (s, 3H), 3.05 (s, 2H), 1.87 (s, 3H), 1.59 (s, 3H), 0.81 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.87 (br. s, 1D); ^{13}C NMR (21 of 22 observed, 125 MHz, CD_3CN): δ 204.9, 173.3, 157.4, 144.7, 138.3, 136.2, 128.4, 123.7, 122.8, 115.8, 108.0, 70.5, 58.2, 56.0, 50.4, 46.8, 38.7, 25.84, 25.75, 23.3, 21.5; IR (film): 1762, 1702, 1607, 1464 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{22}O_4DN_2BrNa$, 484.0784; found 484.0809; $[\alpha]^{25.8}_D$ +179.20° (*c* = 1.000, $CHCl_3$).

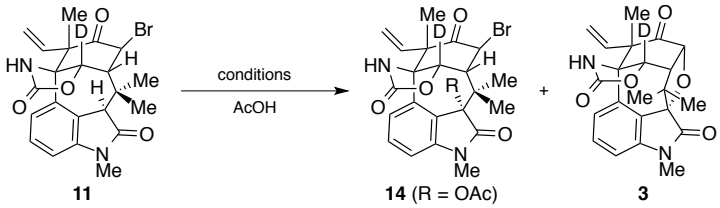


11 13

Cyclobutane 13. To a solution of bromoketone **11** (4.0 mg, 0.0087 mmol, 1.0 equiv) in THF (870 μL) was added NaH (60% dispersion in mineral oil, 1.7 mg, 0.043 mmol, 5.0 equiv) in one portion. The reaction vial was then opened to air for 10 seconds, sealed and left to stir at room temperature. After 2 h, the reaction mixture was filtered by passage over a plug of silica gel (EtOAc eluent, 6 mL). The filtrate was evaporated under reduced pressure, and the resulting residue was purified by flash chromatography (2:1

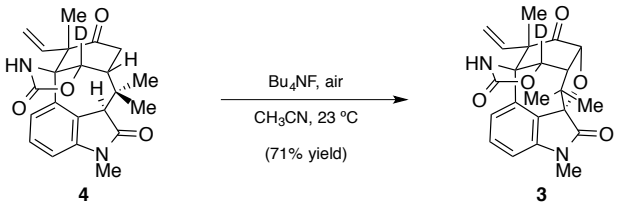
hexanes:EtOAc) to afford cyclobutane **13** (3.3 mg, 97% yield) as a white solid. Crystals suitable for X-ray diffraction studies (CCDC 960226) were obtained by concentration of **13** from a mixture of $CHCl_3$ and pentane. Cyclobutane **13**: mp: 284.0 ºC; R_f 0.51 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.27 (app t, J = 8.0, 7.8, 1H), 6.88 (d, J = 8.0, 1H), 6.79 (d, J = 7.8, 1H), 6.42 (s, 1H), 5.09–5.02 (m, 2H), 4.90 (dd, J = 17.7, 10.8, 1H), 4.00 (d, J = 7.4, 1H), 3.17 (s, 3H), 3.09 (d, J = 7.4, 1H), 1.49 (s, 3H), 1.41 (s, 3H), 0.68 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.33 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.7, 172.3, 159.2, 142.9, 137.2, 135.5, 128.9, 124.6, 120.3, 117.4, 108.5, 80.4 (t, $J_{C–D}$ = 21.6), 69.3, 61.1, 57.4, 46.0, 45.3, 41.6, 26.7, 26.1, 21.1, 18.7; IR (film): 1758, 1710, 1607, 1469 cm^{-1}; HRMS-ESI (m/z) [M + Na]$^+$ calcd for $C_{22}H_{21}O_4DN_2Na$, 402.1540; found 402.1540; $[\alpha]^{27.3}_D$ –94.00° (c = 0.500, $CHCl_3$).



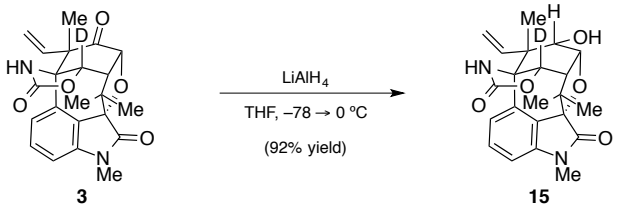


General Procedure for Acetoxyoxindole 14 + Ether 3 From Table 1. A 1-dram vial charged with bromoketone **11** (5.0 mg, 0.011 mmol, 1.0 equiv) was transferred into the glovebox. $Mn(OAc)_3$ was added and the vial was removed from the glovebox. AcOH (1.1 mL) was then added and the reaction vial was sealed and left to stir at the indicated temperature. For entries 1–2, after stirring for 24 h the reaction mixture was cooled to room temperature, transferred to a test tube with $CHCl_3$ (2 mL) and aqueous 2M HCl (1 mL), and then extracted with $CHCl_3$ (3 x 2 mL). The organic layers were combined, washed with saturated aqueous $NaHCO_3$ (2 x 2 mL), H_2O (2 x 2 mL), and then dried over $MgSO_4$ and evaporated under reduced pressure. The resulting residue was purified by preparative thin layer chromatography (1:1 $CHCl_3$:EtOAc) to afford acetoxyoxindole **14** and/or ether **3** as white solids. For entry 3, after stirring for 24 h the reaction mixture was cooled to room temperature and concentrated under reduced pressure. To the resulting residue was added a 1:1 mixture of MeOH:H_2O (1.1 mL) and solid K_2CO_3 (3.8 mg, 0.272 mmol, 25.0 equiv) in one portion. The reaction vial was flushed with N_2, sealed and allowed to stir at 70 ºC. After 19 h, the reaction mixture was cooled to room temperature, diluted with H_2O (2 mL) and transferred to a test tube with $CHCl_3$ (2 mL). The resulting biphasic mixture was extracted with $CHCl_3$ (3 x 2 mL). The organic layers were

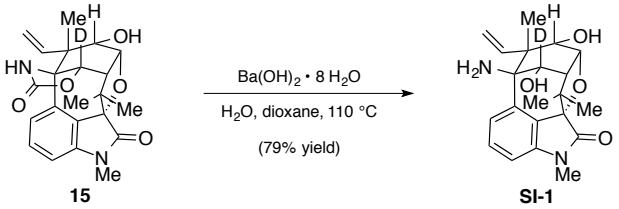
combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by preparative thin layer chromatography (1:1 $CHCl_3$:EtOAc) to afford ether **3** (2.4 mg, 56% yield) as a white solid. Acetoxyoxindole **14**: mp: 283.0 ºC (decomp.); R_f 0.65 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 (app t, *J* = 8.0, 1H), 6.94 (s, 1H), 6.80–6.76 (m, 2H), 5.37 (s, 1H), 5.31 (d, *J* = 17.0, 1H), 5.21 (d, *J* = 10.7, 1H), 5.01 (dd, *J* = 17.0, 10.7, 1 H), 3.20 (s, 3H), 3.04 (s, 1H), 1.91 (s, 3H), 1.88 (s, 3H), 1.64 (s, 3H), 1.02 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.87 (br. s, 1D); ^{13}C NMR (23 of 24 observed, 125 MHz, $CDCl_3$): δ 203.1, 171.4, 168.9, 158.4, 145.9, 137.3, 136.5, 131.0, 124.7, 122.6, 118.0, 108.2, 84.3, 70.4, 58.2, 56.3, 48.9, 41.2, 27.1, 25.3, 22.7, 22.6, 21.4; IR (film): 1762, 1732, 1712, 1611, 1597, 1463 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{24}H_{24}O_6DN_2BrNa$, 540.0856; found 540.0858; $[\alpha]^{26.9}_D$ –18.40° (*c* = 1.000, $CHCl_3$). Ether **3**: mp: 317.0 ºC (decomp.); R_f 0.53 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.67 (s, 1H), 7.25 (app t, *J* = 7.9, 1H), 6.93 (dd, *J* = 7.9, 0.64, 1H), 6.81 (dd, *J* = 7.9, 0.64, 1H), 5.22 (dd, *J* = 17.2, 10.7, 1H), 4.96–4.89 (m, 2H), 4.78 (d, *J* = 10.7, 1H), 3.30 (d, *J* = 7.5, 1H), 3.23 (s, 3H), 1.45 (s, 3H), 1.42 (s, 3H), 1.02 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 4.69 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.2, 171.9, 158.7, 142.8, 137.6, 136.3, 130.4, 126.6, 122.3, 116.4, 108.6, 88.8, 83.3, 81.4 (t, $J_{C–D}$ = 22.6), 70.2, 59.4, 51.4, 48.9, 26.9, 25.4, 21.2, 19.6; IR (film): 1774, 1724, 1710, 1605 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{21}O_5DN_2Na$, 418.1489; found 418.1476; $[\alpha]^{21.3}_D$ –157.20° (*c* = 1.000, $CHCl_3$).



Ether 3. To a solution of ketone **4** (30 mg, 0.079 mmol, 1.0 equiv) in CH_3CN (7.9 mL) was added Bu_4NF (1.0 M in THF, 236 μL, 0.236 mmol, 3.0 equiv) in a dropwise manner. The reaction mixture was stirred for 1 min, then opened to air for 30 seconds. The reaction vessel was sealed and left to stir at room temperature. After 2 h, the reaction was quenched with a solution of saturated aqueous NH_4Cl (5 mL). The resulting mixture was transferred to a separatory funnel with EtOAc (10 mL) and H_2O (10 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 10 mL). The organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The crude residue was purified by flash chromatography (1:1 hexanes:EtOAc) to afford ether **3** (22 mg, 71% yield) as a white solid.

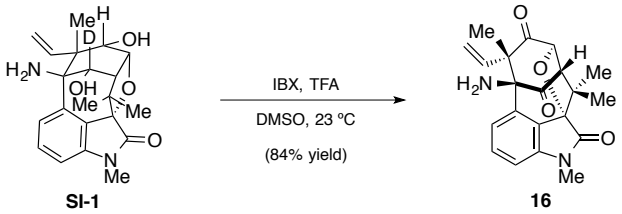


Alcohol 15. To a solution of ether **3** (9.4 mg, 0.024 mmol, 1.0 equiv) in THF (2.3 mL) at –78 ºC was added $LiAlH_4$ (1.0 M in THF, 24 μL, 0.024 mmol, 1.0 equiv) in a dropwise manner. After 5 min, the reaction mixture was warmed to 0 ºC. After 20 min, the reaction was quenched at 0 °C with slow addition of a saturated aqueous solution of Rochelle's salt (3.0 mL) and then allowed to warm to 23 ºC. The mixture was stirred at room temperature for 30 min, and then transferred to a separatory funnel with EtOAc (10 mL) and H_2O (10 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 10 mL). The organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by preparative thin layer chromatography (1:2 $CHCl_3$:EtOAc) to afford alcohol **15** (8.7 mg, 92% yield) as a white solid. Alcohol **15**: mp: 263.0 ºC; R_f 0.15 (1:3 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (s, 1H), 7.19 (app t, *J* = 7.9, 1H), 6.82 (d, *J* = 7.9, 1H), 6.77 (d, *J* = 7.9, 1H), 5.21 (dd, *J* = 17.4, 10.8, 1 H), 5.09–5.03 (m, 2H), 4.94 (d, *J* = 10.8, 1H), 3.72 (dd, *J* = 11.1, 2.0, 1H), 3.22 (s, 3H), 3.02 (d, J = 7.5, 1H), 2.02 (d, J = 11.1, 1 H), 1.43 (s, 3H), 1.41 (s, 3H), 0.94 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 4.79 (br. s, 1D); ^{13}C NMR (21 of 22 observed, 125 MHz, $CDCl_3$): δ 173.0, 159.4, 142.4, 139.0, 136.4, 130.2, 126.9, 122.4, 117.9, 108.2, 87.3, 82.0, 76.1, 70.9, 49.2, 48.7, 48.3, 26.8, 25.6, 21.3, 19.6; IR (film): 1752, 1722, 1603, 1462 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{23}O_5DN_2Na$, 420.1646; found 420.1631; $[\alpha]^{24.1}_D$ –49.20° (*c* = 1.000, $CHCl_3$).



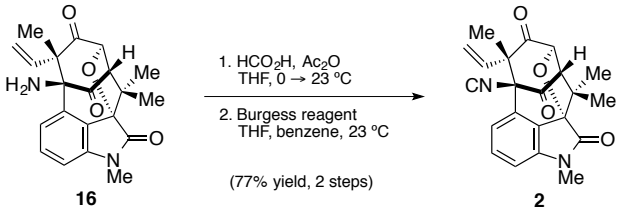
SI-1. A Schlenk tube was charged with alcohol **15** (7.30 mg, 0.0184 mmol, 1.0 equiv) and $Ba(OH)_2{\cdot}8H_2O$ (46.3 mg, 0.147 mmol, 8.0 equiv). The reaction vessel was then evacuated and backfilled with N_2 three times. A 2:1 mixture of 1,4-dioxane:H_2O (634 μL) that had been taken through six freeze-pump-thaw cycles prior to use was then added to the Schlenk tube. The vessel was then sealed and allowed to stir at 110 °C. After 21 h, the contents of the Schlenk tube were transferred to a separatory funnel with EtOAc (5 mL) and H_2O (5 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 5 mL). The organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting residue was purified by preparative thin layer chromatography (3:1 $CHCl_3$:MeOH) to afford **SI-1** (5.4 mg, 79% yield) as a white solid. **SI-1**: R_f 0.12 (9:1 $CHCl_3$:MeOH); ^{1}H NMR (500 MHz, D_2O, 22 of 25 observed): δ 7.37 (app t, *J* = 8.3, 1H),

7.18 (d, *J* = 8.3, 1H), 7.02 (d, *J* = 8.2, 1H), 5.50 (dd, *J* = 17.6, 11.0, 1H), 4.96 (dd, *J* = 7.3, 1.8, 1 H), 4.92 (d, *J* = 17.6, 1H), 3.88 (d, *J* = 1.8, 1H), 3.23 (s, 3H), 2.83 (d, *J* = 7.3, 1H), 1.91 (s, 2H), 1.37 (s, 3H), 1.28 (s, 3H), 0.85 (s, 3H).



SI-1 16

Aminodiketone 16. To a solution of **SI-1** (5.4 mg, 0.014 mmol, 1.0 equiv) in DMSO (500 μL) was added TFA (1.2 μL, 0.016 mmol, 1.10 equiv). The mixture was stirred at room temperature. After 5 min, IBX (20.4 mg, 0.0728 mmol, 5.0 equiv) was added in one portion and the vial was flushed with N_2. After stirring at room temperature for 20 h, the reaction mixture transferred to a separatory funnel with a solution of aqueous K_2CO_3 (5 mL, concentration of 50 mg/mL) and EtOAc (5 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 5 mL) and the organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting residue was purified by preparative thin layer chromatography (9:1 $CHCl_3$:MeOH) to afford aminodiketone **16** (4.5 mg, 84% yield) as a white solid. Aminodiketone **16**: mp: 190–191 ºC; R_f 0.73 (9:1 CH_3Cl:MeOH); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (dd, *J* = 8.2, 7.6, 1H), 7.29 (dd, *J* = 8.2, 1.0, 1H), 6.80 (dd, *J* = 7.6, 1.0, 1H), 5.64 (dd, *J* = 17.4, 10.7, 1H), 5.40 (d, *J* = 10.7, 1 H), 5.21 (d, *J* = 17.4, 1H), 4.93 (d, *J* = 7.6, 1H), 3.49 (d, *J* = 7.6, 1H), 3.20 (s, 3H), 2.04 (br. s, 2H), 1.59 (s, 3H), 1.19 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (21 of 22 observed, 125 MHz, $CDCl_3$): δ 204.9, 204.4, 170.2, 143.6, 134.8, 134.8, 130.3, 125.8, 124.2, 118.9, 108.9, 86.5, 79.4, 69.4, 62.5, 62.1, 52.4, 26.9, 25.3, 20.0, 17.6; IR (film): 1718, 1609, 1582, 1459, 1366 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{21}H_{22}O_4DN_2Na$, 389.1477; found 389.1469; $[\alpha]^{22.9}_D$ +29.20° (*c* = 1.000, $CHCl_3$).



16 2

(+)-*N*-Methylwelwitindolinone D Isonitrile (2). A 1-dram vial was charged with 96% formic acid (0.100 mL) and acetic anhydride (0.100 mL). The resulting mixture was stirred at 60 °C for 1 h. The reaction vessel was cooled to room temperature and 39 μL of the 96% formic acid/acetic anhydride mixture was added to a solution of aminodiketone **16** (4.2 mg, 0.010 mmol, 1.0 equiv) in THF (765 μL) at 0 °C. The reaction was then warmed to room temperature. After 2 h, the reaction mixture was transferred to a test tube with EtOAc (2 mL) and a solution of saturated aqueous $NaHCO_3$ (1 mL). The

resulting biphasic mixture was extracted with EtOAc (4 x 2 mL). The organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure to afford the crude product, which was used directly in the subsequent reaction.

To a vial containing the crude product from the previous step was added THF (600 μL) and benzene (600 μL), followed by Burgess reagent (3.5 mg, 0.011 mmol, 1.0 equiv). The vial was flushed with N_2 and allowed to stir at room temperature for 40 min. An additional amount of Burgess reagent (3.5 mg, 0.015 mol, 1.0 equiv) was then added, and the reaction was allowed to stir at room temperature for 10 min. The reaction was then filtered by passage over a plug of silica gel (EtOAc eluent, 10 mL). The filtrate was evaporated under reduced pressure, and the resulting residue was purified by preparative thin layer chromatography (1:1 hexanes:EtOAc) to afford (+)-**2** (3.3 mg, 77% yield, 2 steps) as a white solid. Spectral data for ^{1}H NMR, ^{13}C NMR, and IR for synthetic **2** was consistent with literature reports.[4] (+)-*N*-Methylwelwitindolinone D isonitrile (**2**): mp: 156 ºC; R_f 0.50 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.45 (dd, *J* = 8.3, 7.9, 1H), 7.29 (dd, *J* = 8.3, 0.7, 1H), 6.93 (dd, *J* = 7.9, 0.7, 1H), 5.48 (dd, *J* = 16.3, 10.6, 1H), 5.43 (dd, *J* = 10.6, 1.5, 1H), 5.35 (dd, *J* = 16.3, 1.5, 1H), 4.92 (d, *J* = 7.5, 1H), 3.57 (d, *J* = 7.5, 1H), 3.19 (s, 3H), 1.55 (s, 3H), 1.39 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (125 MHz, CD_2Cl_2): δ 201.3, 192.8, 169.9, 165.5, 144.4, 133.0, 131.4, 126.8, 125.8, 124.0, 120.6, 110.5, 86.7, 81.1, 79.7, 62.0, 61.7, 53.6, 27.1, 25.0, 20.1, 19.8; IR (film): 2980, 2940, 2139, 1730, 1609, 1592, 1463, 1366, 1344 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{20}N_2O_4Na$, 399.1321; found 399.1322; $[\alpha]^{22.3}_D$ +4.30° (*c* = 0.37, CH_2Cl_2). Note: This specific rotation differs from that reported for the natural product ($[\alpha]_D$ –30°, *c* = 0.37, CH_2Cl_2). This is most likely due to a tabulation error in the isolation report as: (a) the synthesis begins with (S)-carvone >96%+ ee, (b) the specific rotation of bicycle **6** in this synthesis is consistent with that of material used in previous syntheses of welwitindolinones,[3,5c] and (c) our specific rotation data has given consistent results across a range of samples and concentrations. Although the sign of rotation differs, the compound we have prepared is assumed to be the natural occurring enantiomer due to its biosynthetic relationship to the *N*-methylweltindolinone C series of natural products, whose absolute configuration have previously been established.[3,5]

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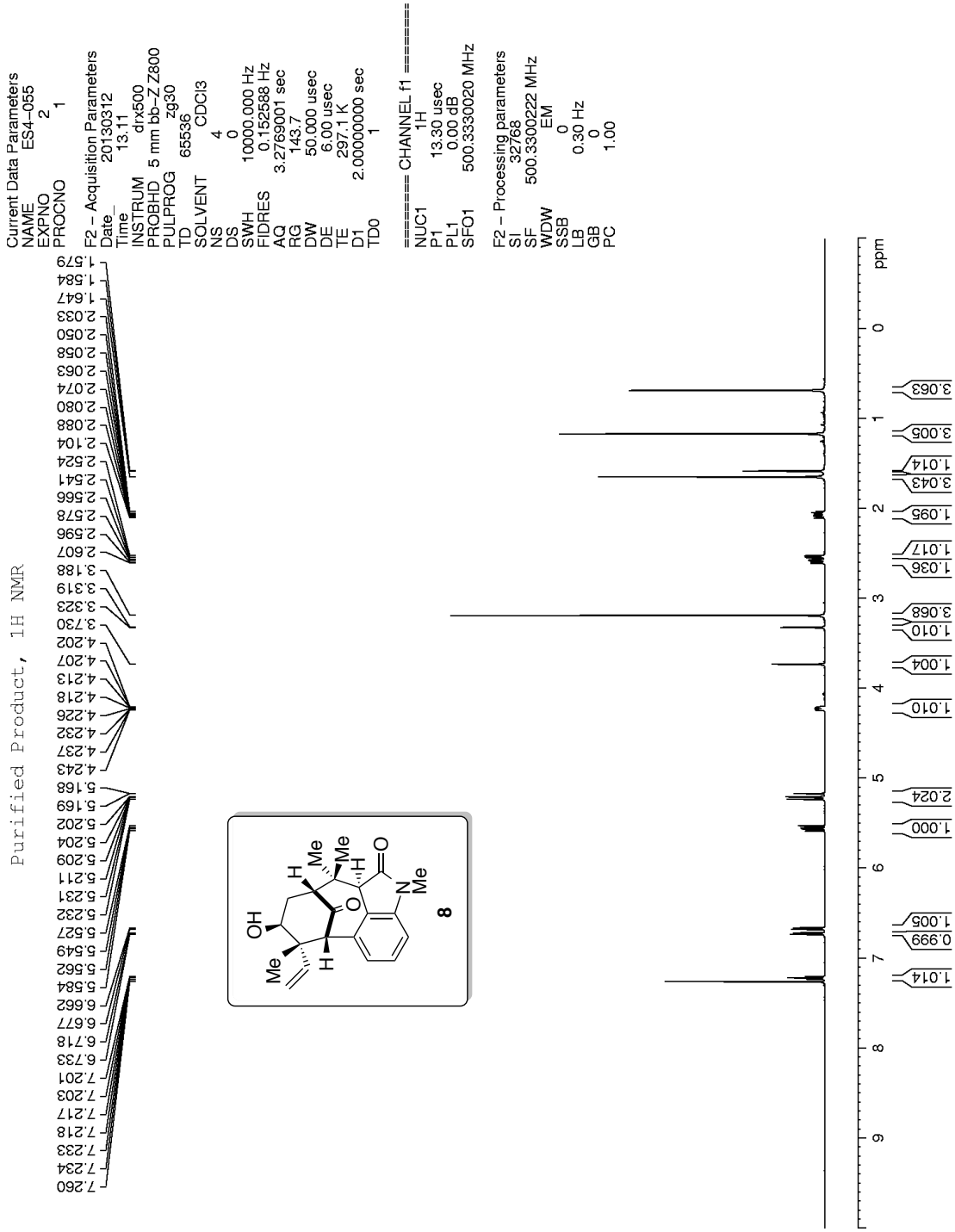
[4] J. L. Jimenez, U. Huber, R. E. Moore, G. M. L. Patterson, *J. Nat. Prod.* **1999**, *62,* 569–572.

[5] a) V. Bhat, K. M. Allan, V. H. Rawal, *J. Am. Chem. Soc.* **2011**, *133*, 5798–5801; b) K. W. Allan, K. Kobayashi, V. H. Rawal, *J. Am. Chem. Soc.* **2012**, *134*, 1392–1395; c) K. W. Quasdorf, A. D. Huters, M. W. Lodewyk, D. J. Tantillo, N. K. Garg, *J. Am. Chem. Soc.* **2012**, *134*, 1396–1399.

^{1}H and ^{2}H NMR Spectra: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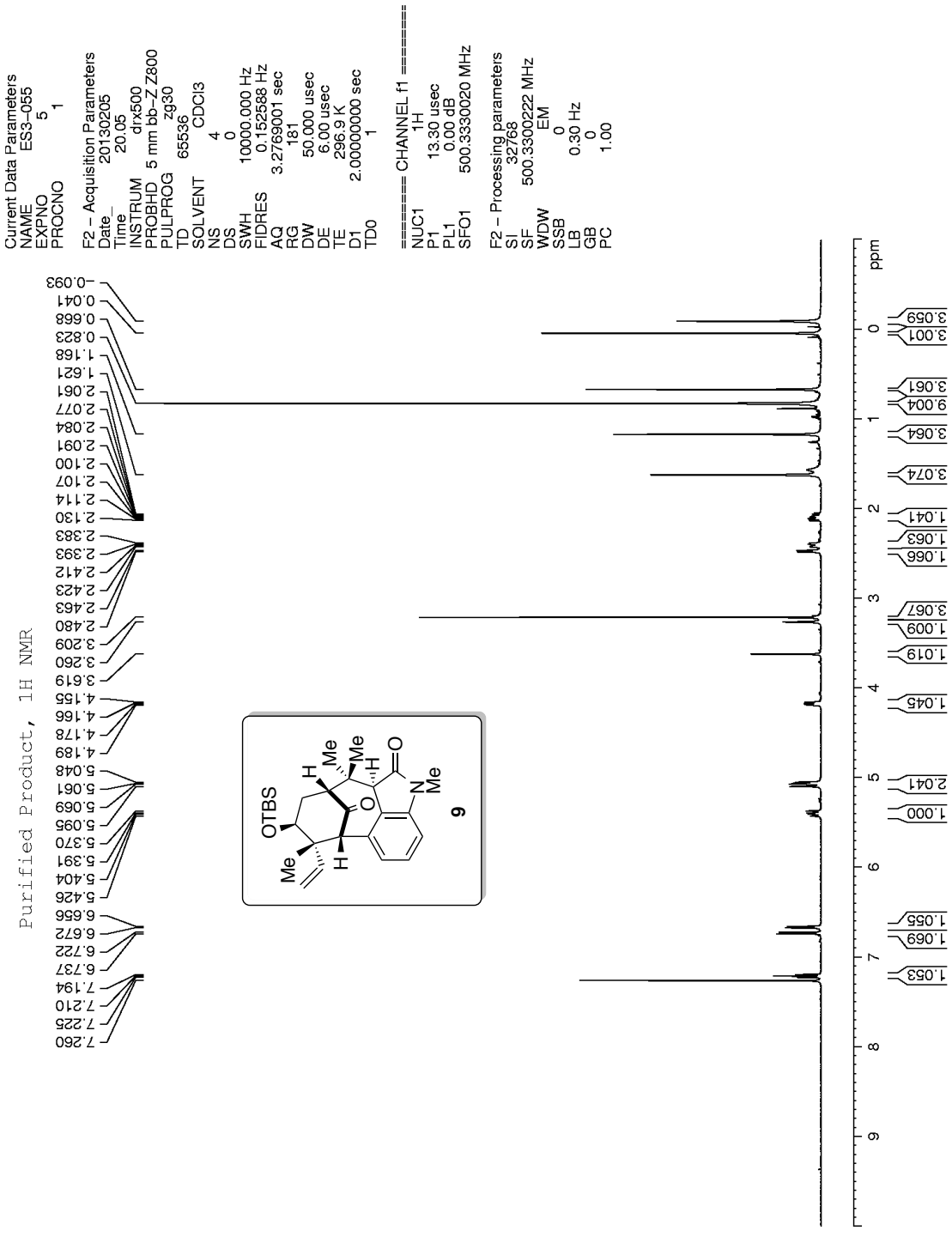
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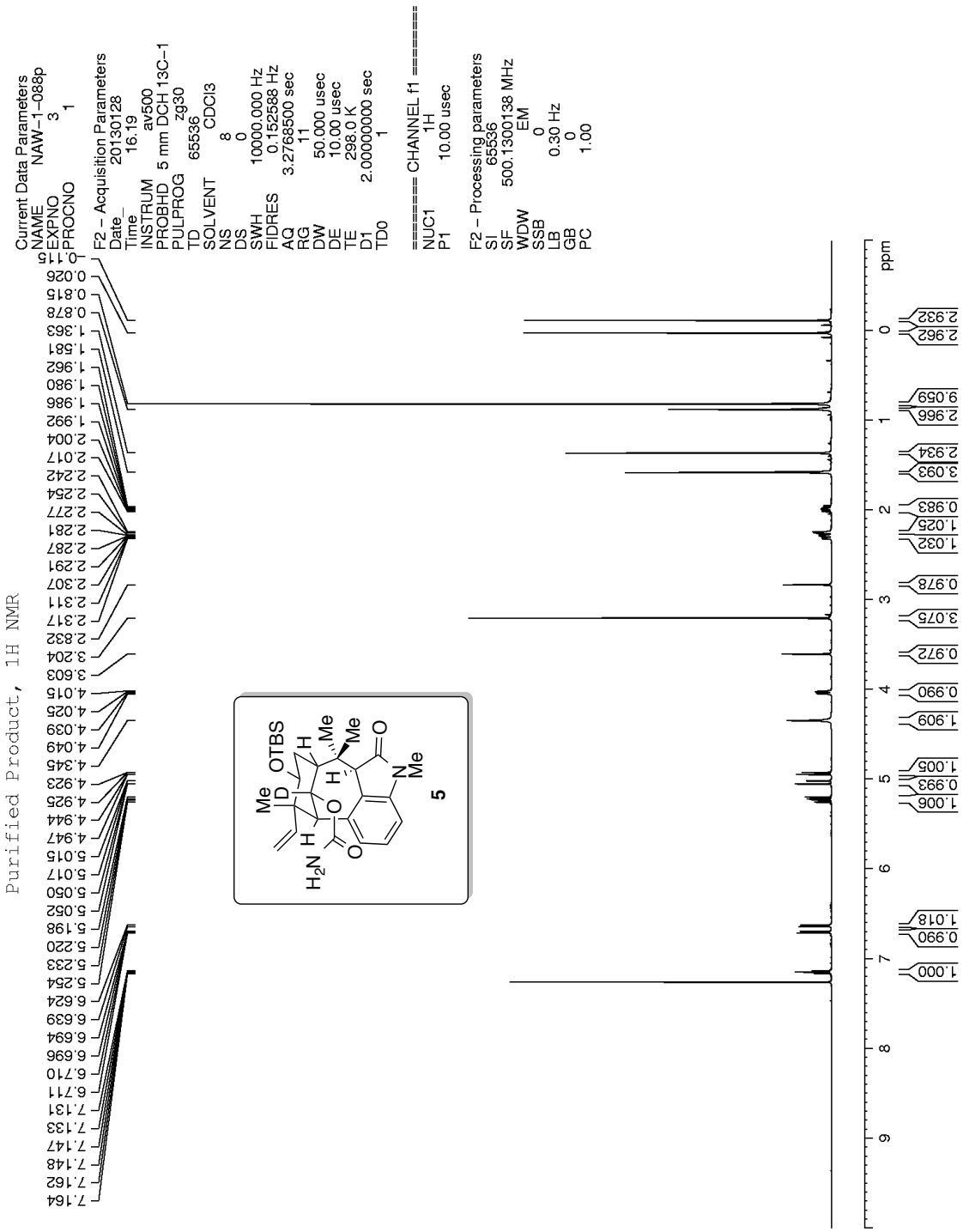
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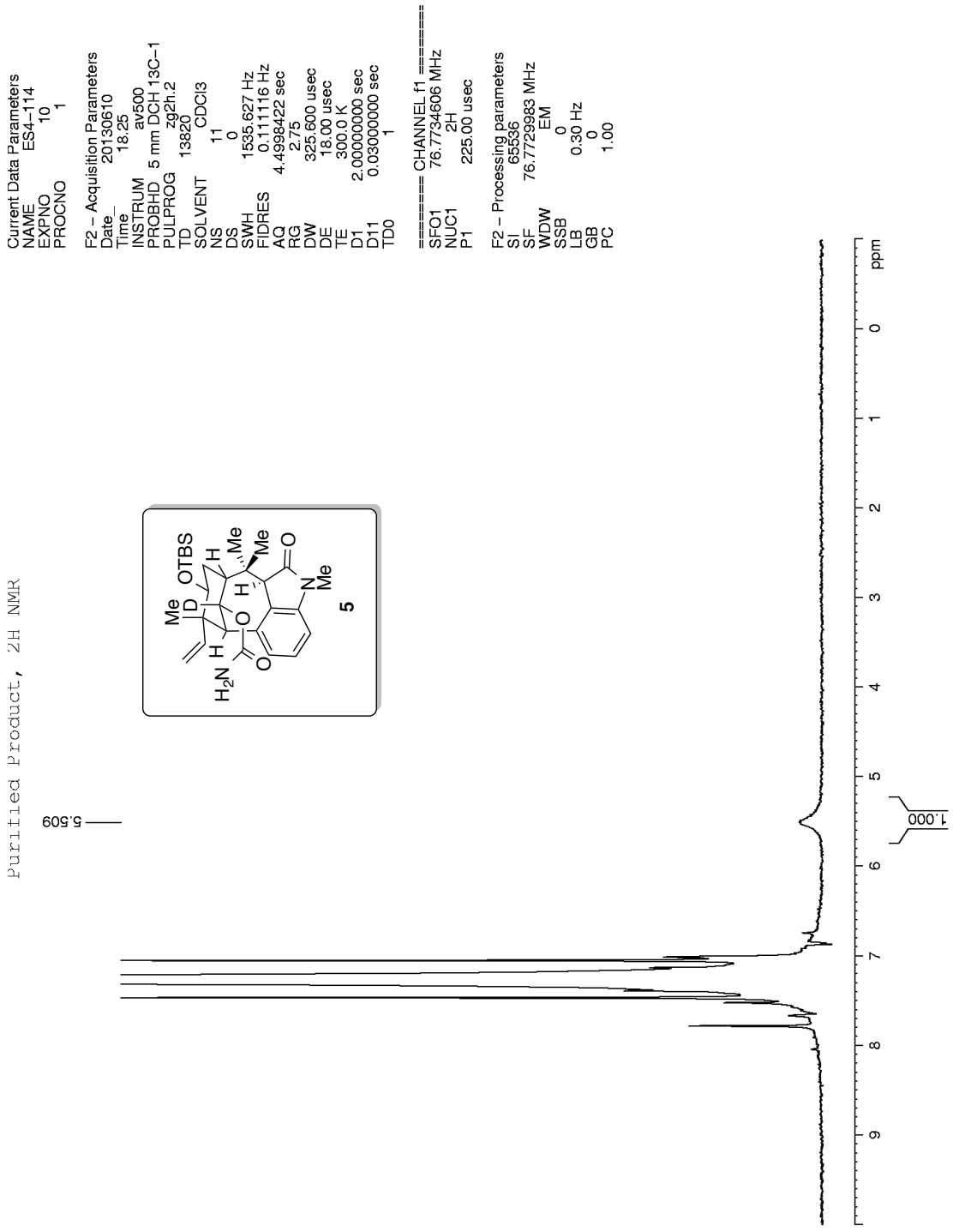
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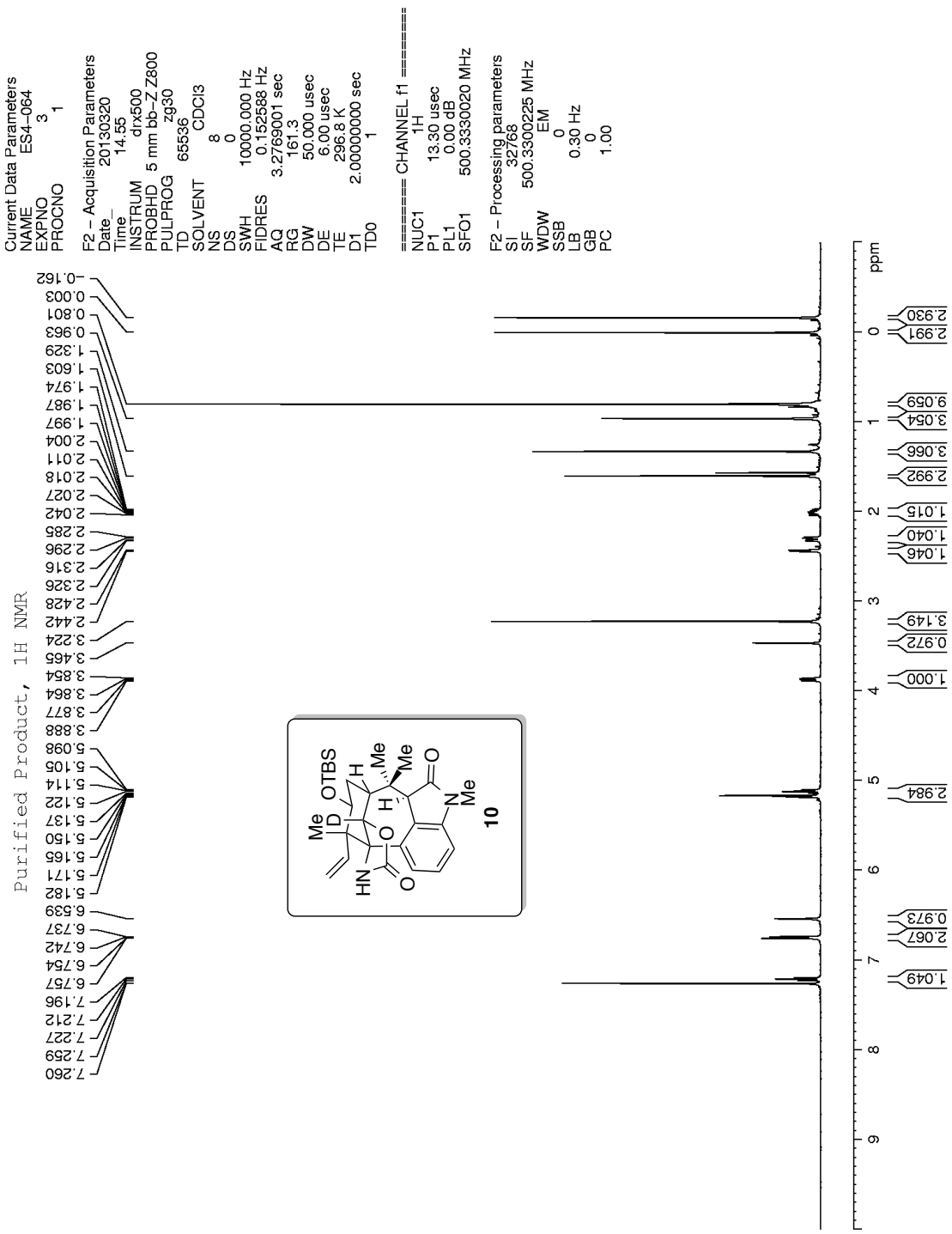
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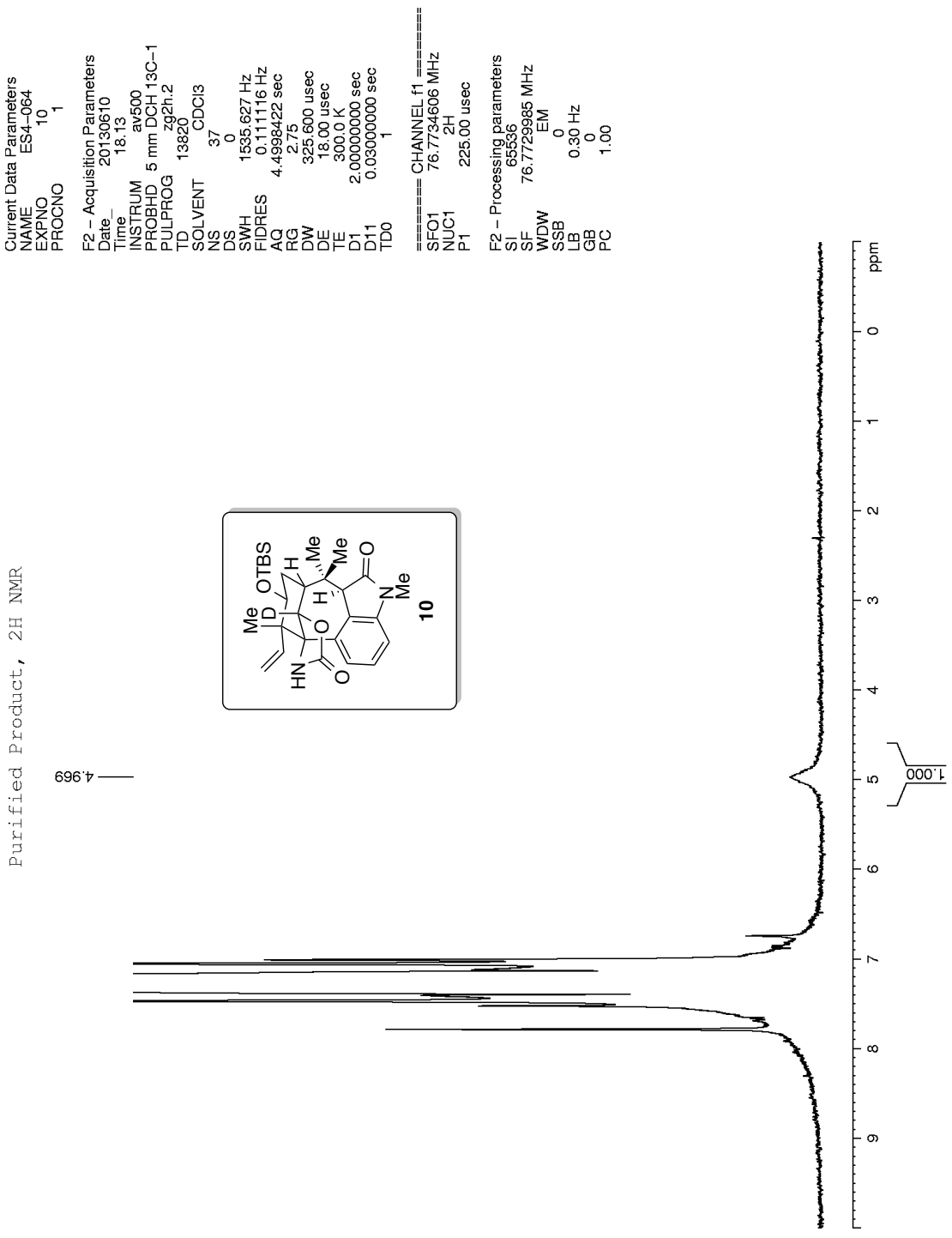
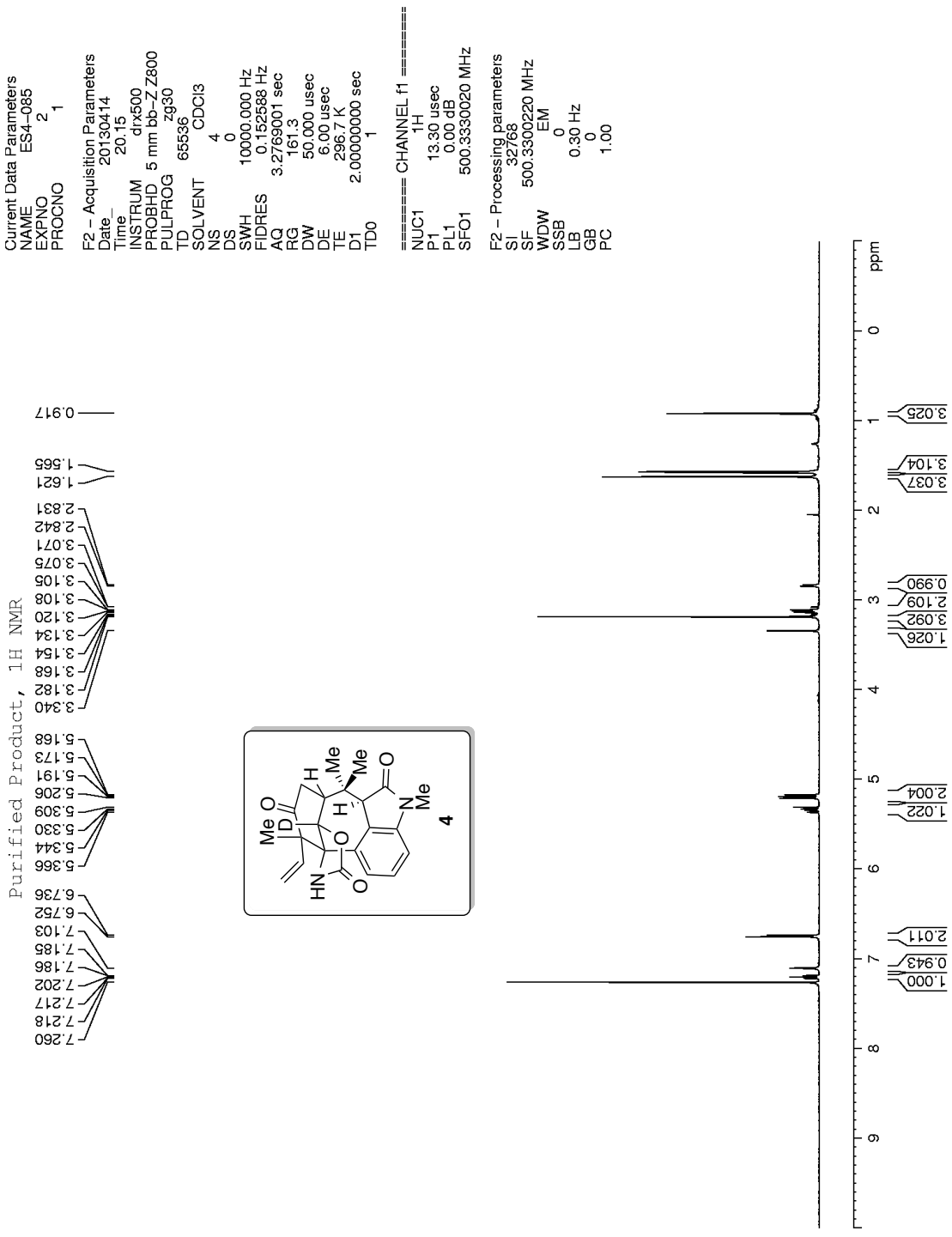
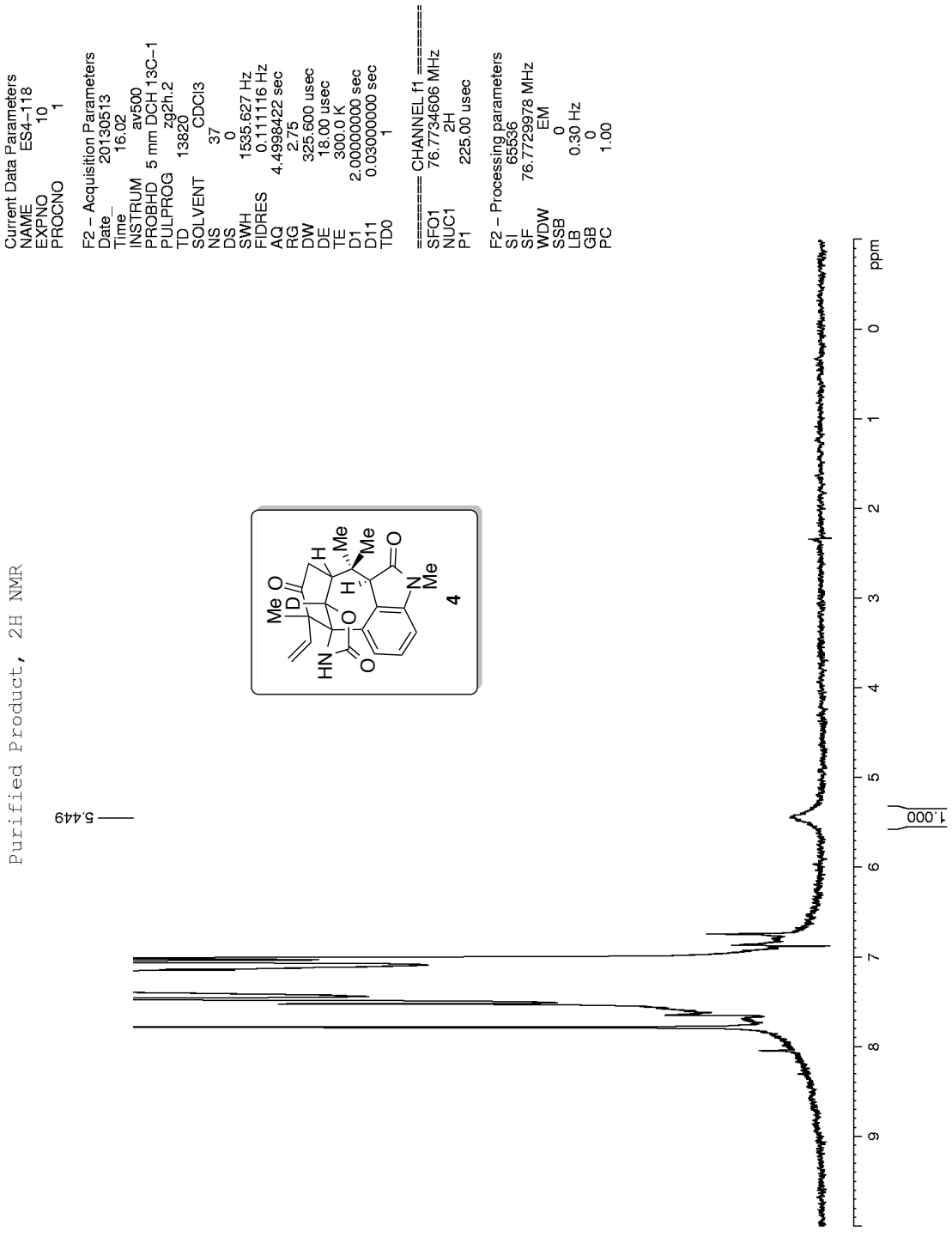
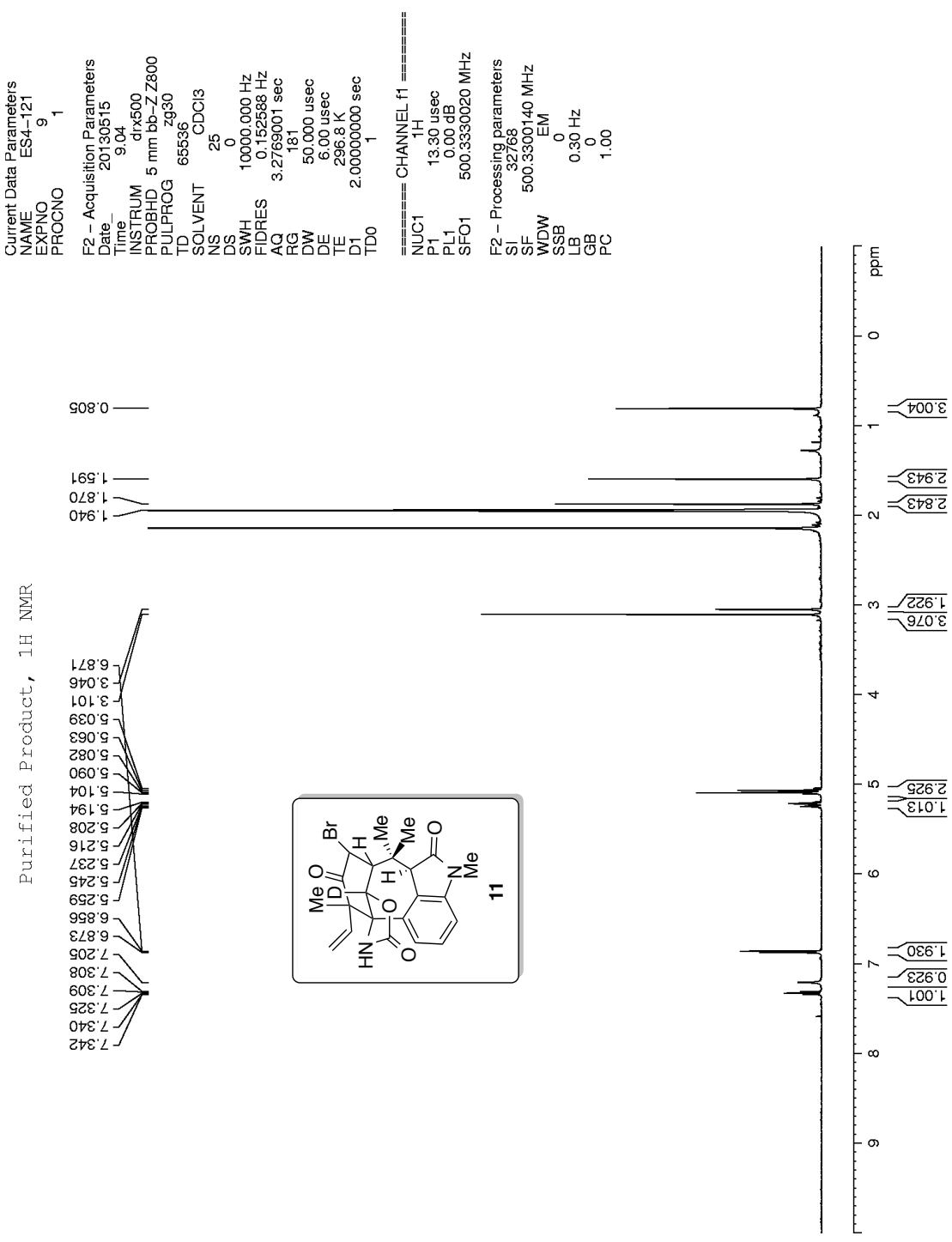
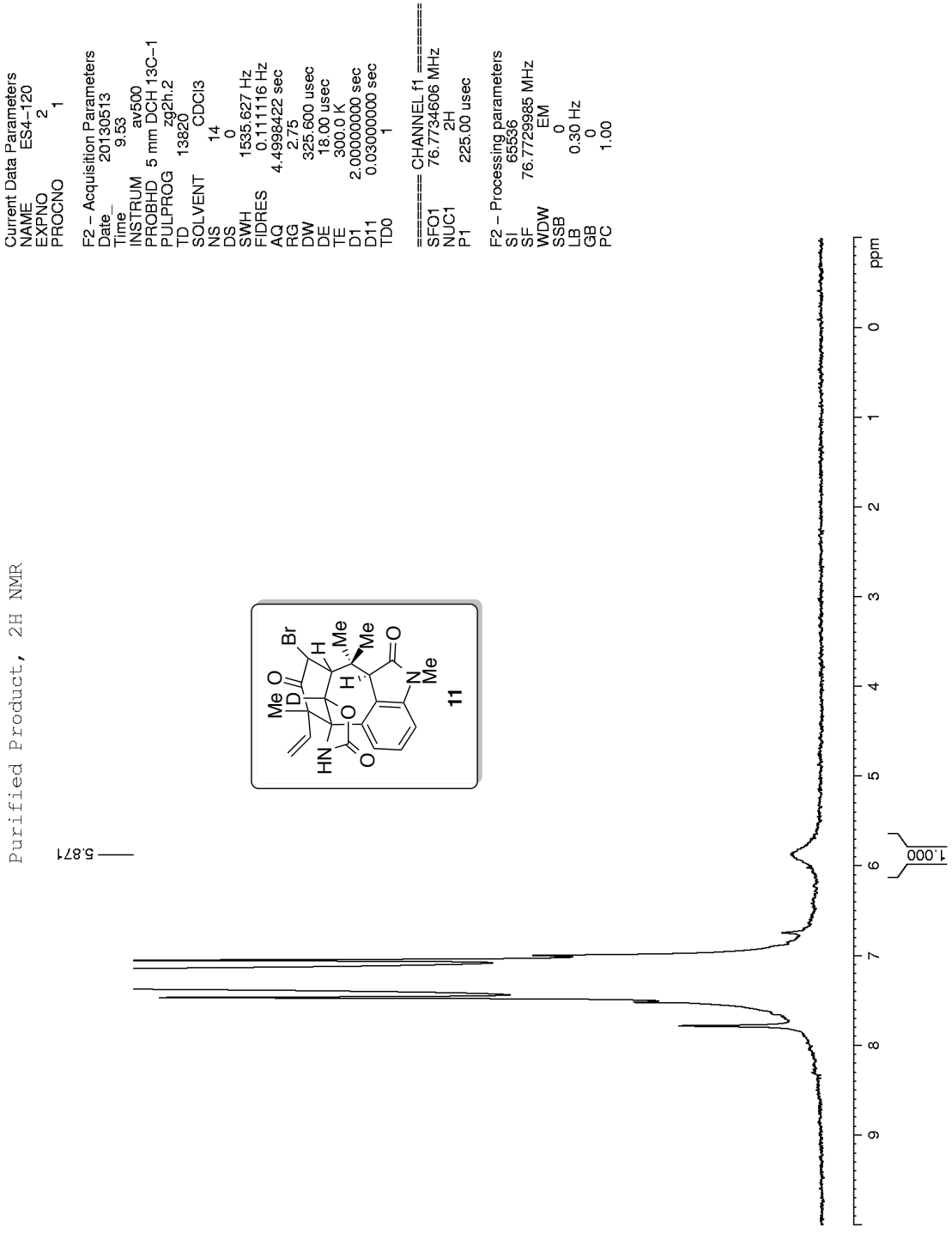
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300225 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
ppm

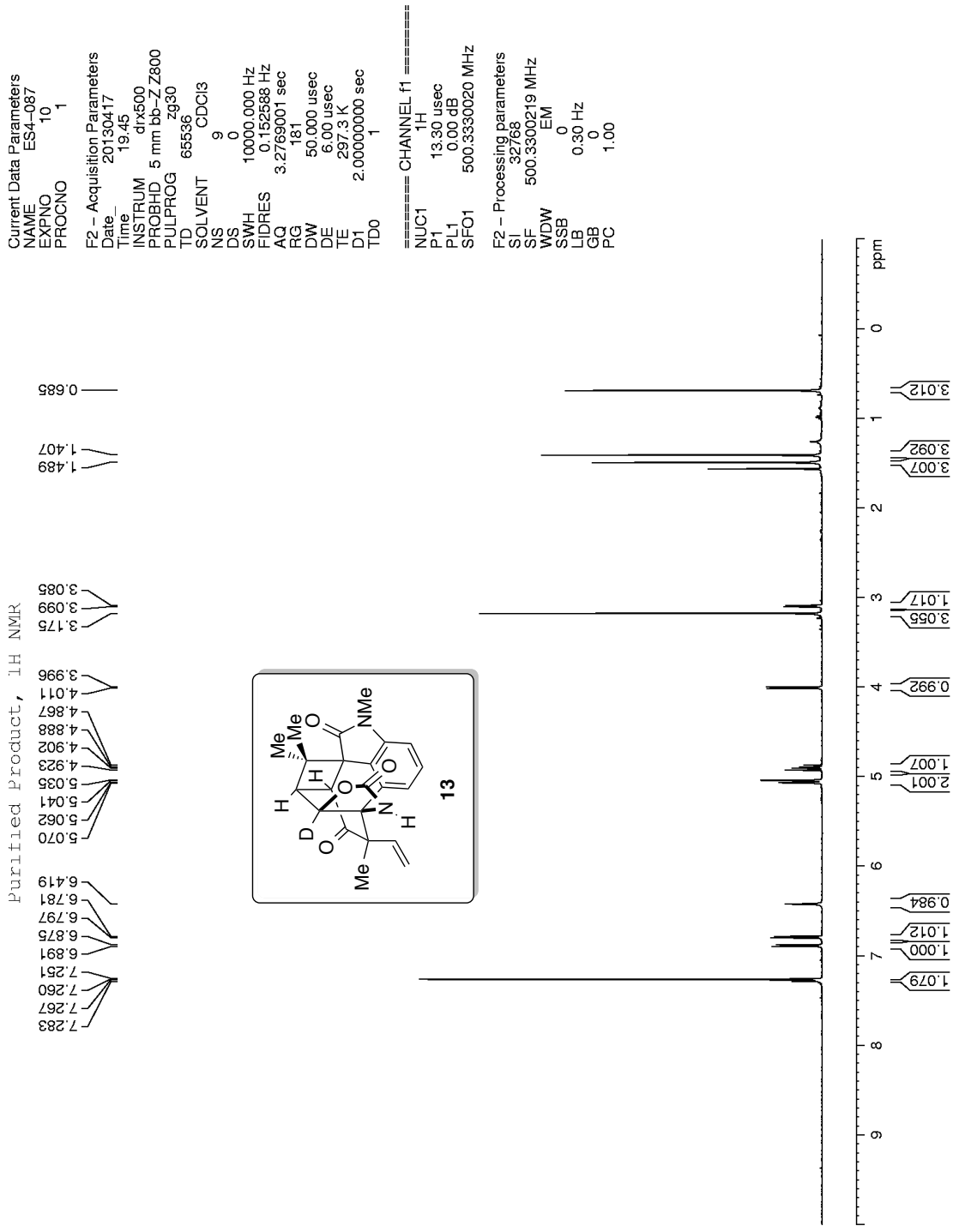

Purified Product, 2H NMR
4.969
Current Data Parameters
NAME ES4-064
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130610
Time 18.13
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 37
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 - Processing parameters
SI 65536
SF 76.7729985 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
D
OTBS
H
Me
Me
O
H
N
Me
HN
O
O
10
1.000
ppm

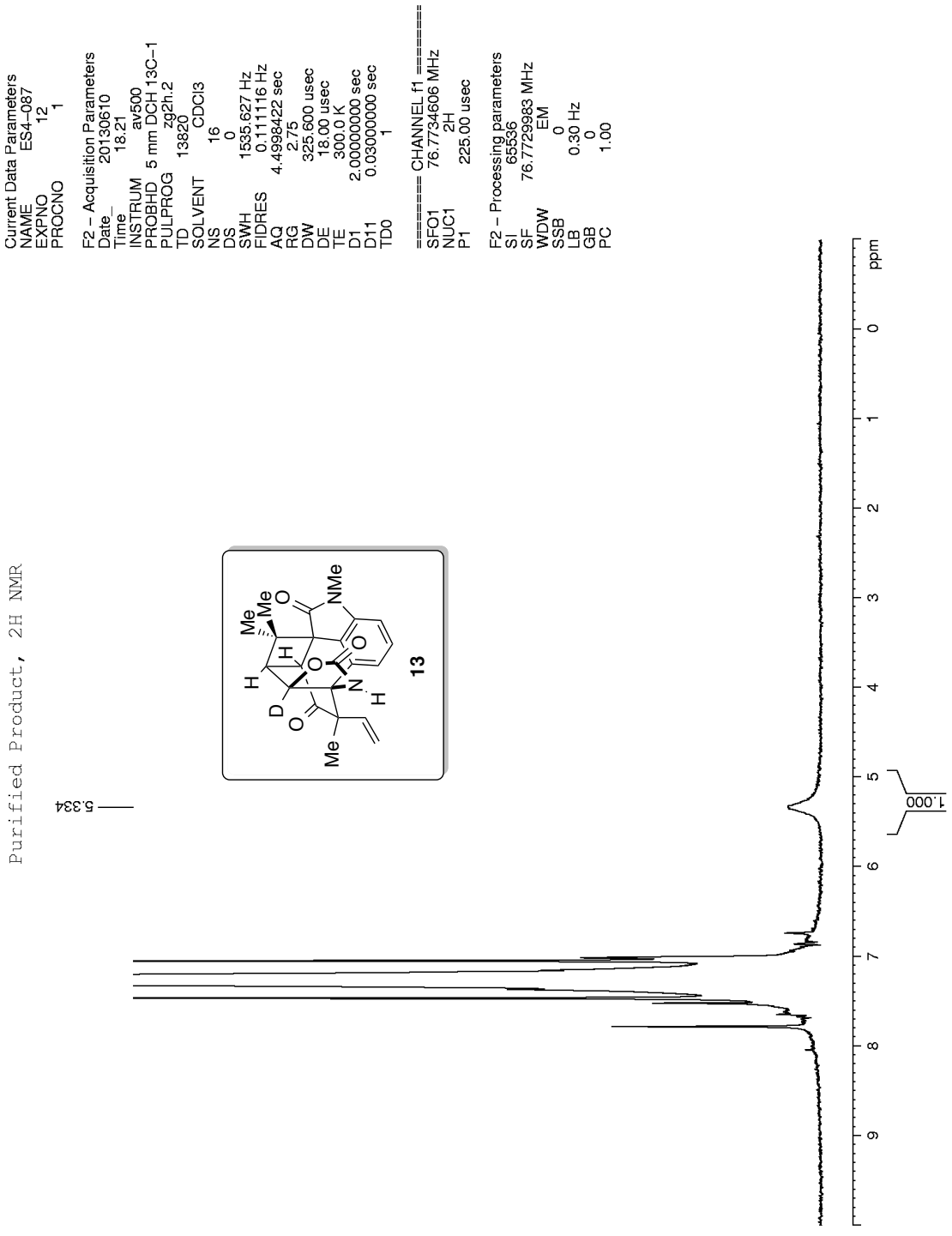

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-085
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130414
Time 20.15
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 161.3
DW 50.000 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.917
1.565
1.621
2.831
2.842
3.071
3.075
3.105
3.108
3.120
3.134
3.154
3.168
3.182
3.340
5.168
5.173
5.191
5.206
5.309
5.330
5.344
5.366
6.736
6.752
7.103
7.185
7.186
7.202
7.217
7.218
7.260
Me O
D
H
Me
Me
O
H
N
Me
O
HN
O
4
ppm
0
1
2
3
4
5
6
7
8
9
3.025
3.104
3.037
0.990
2.109
3.092
1.026
1.022
2.004
2.011
1.000
0.943

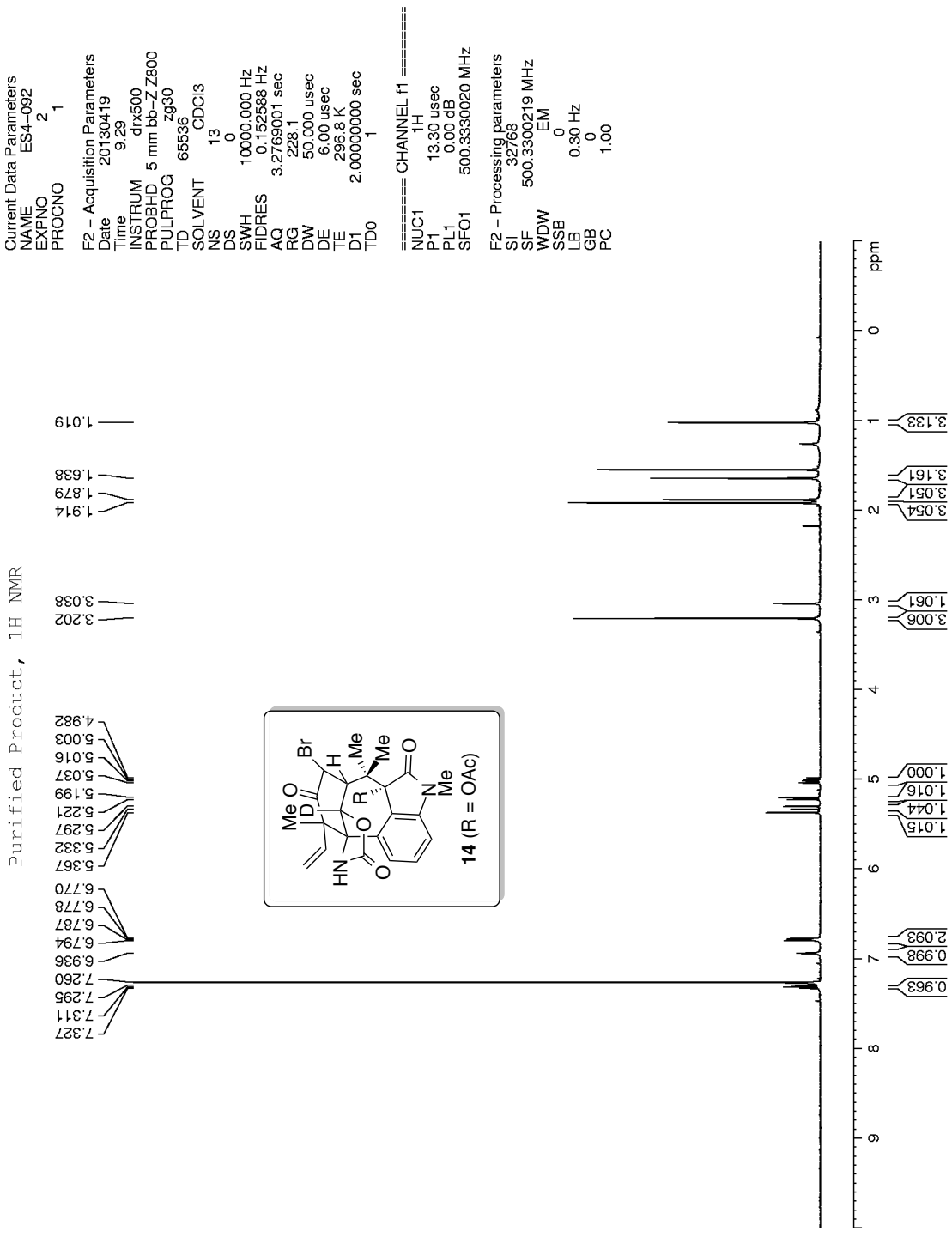

Purified Product, 2H NMR
5.449
Me O
D
HN
O
H
Me
Me
O
H
O
N
Me
O
4
1.000
ppm
Current Data Parameters
NAME ES4-118
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130513
Time 16.02
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 37
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 - Processing parameters
SI 65536
SF 76.7729978 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

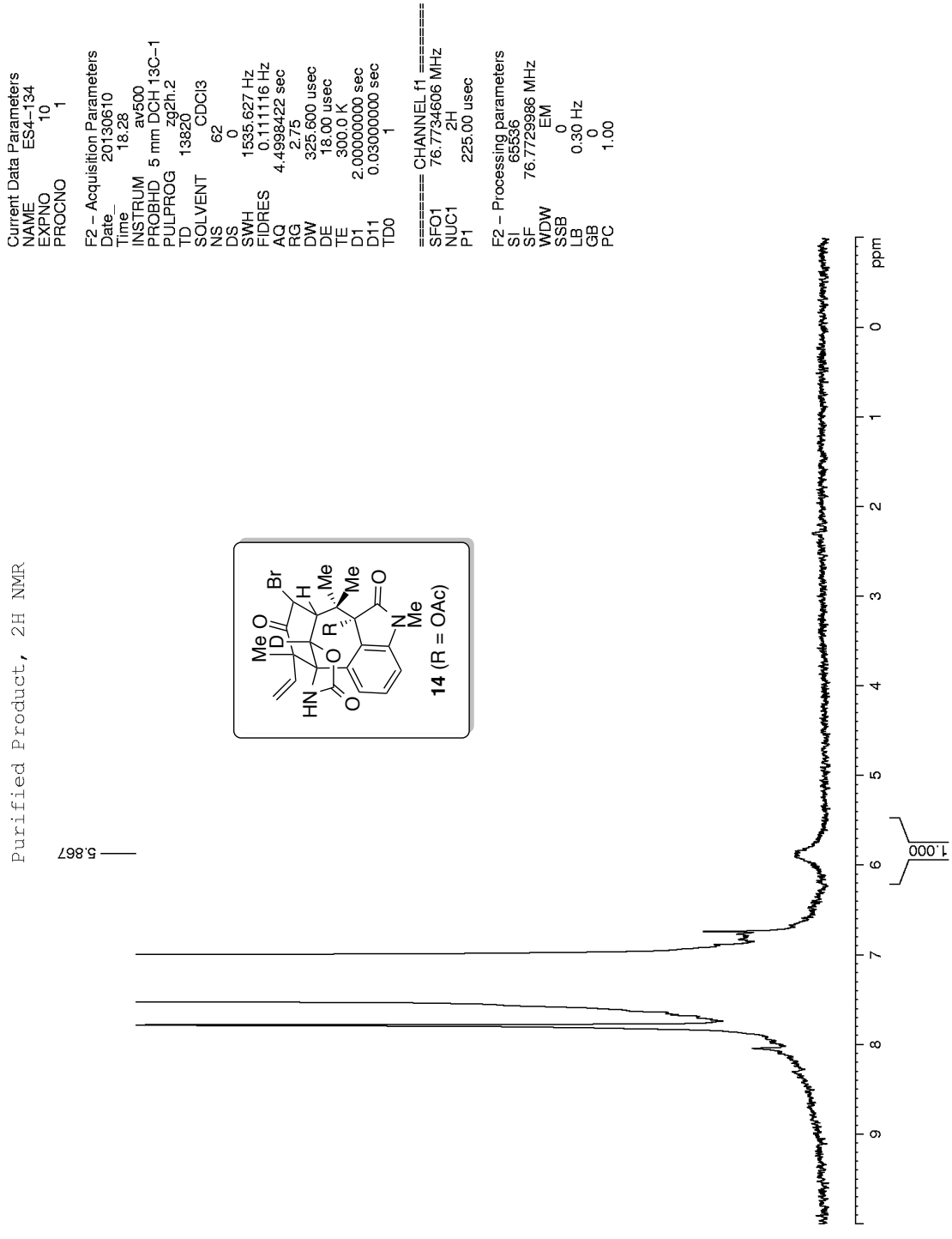

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-121
EXPNO 9
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130515
Time 9.04
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
ppm

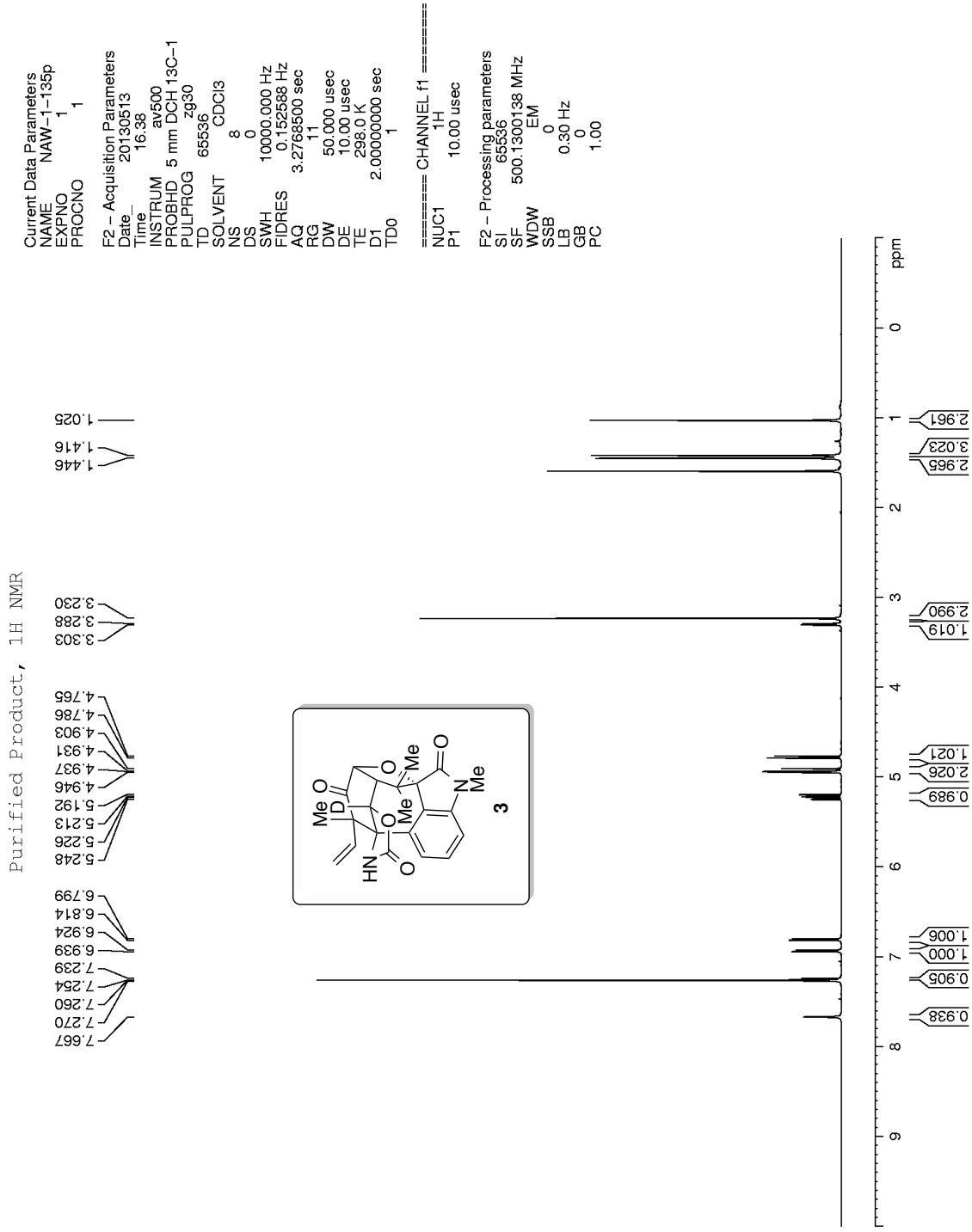

Purified Product, 2H NMR
5.871
Current Data Parameters
NAME ES4-120
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130513
Time 9.53
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 14
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 - Processing parameters
SI 65536
SF 76.7729985 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me O
D
Br
H
Me
Me
O
N
Me
H
O
HN
O
11
1.000
ppm

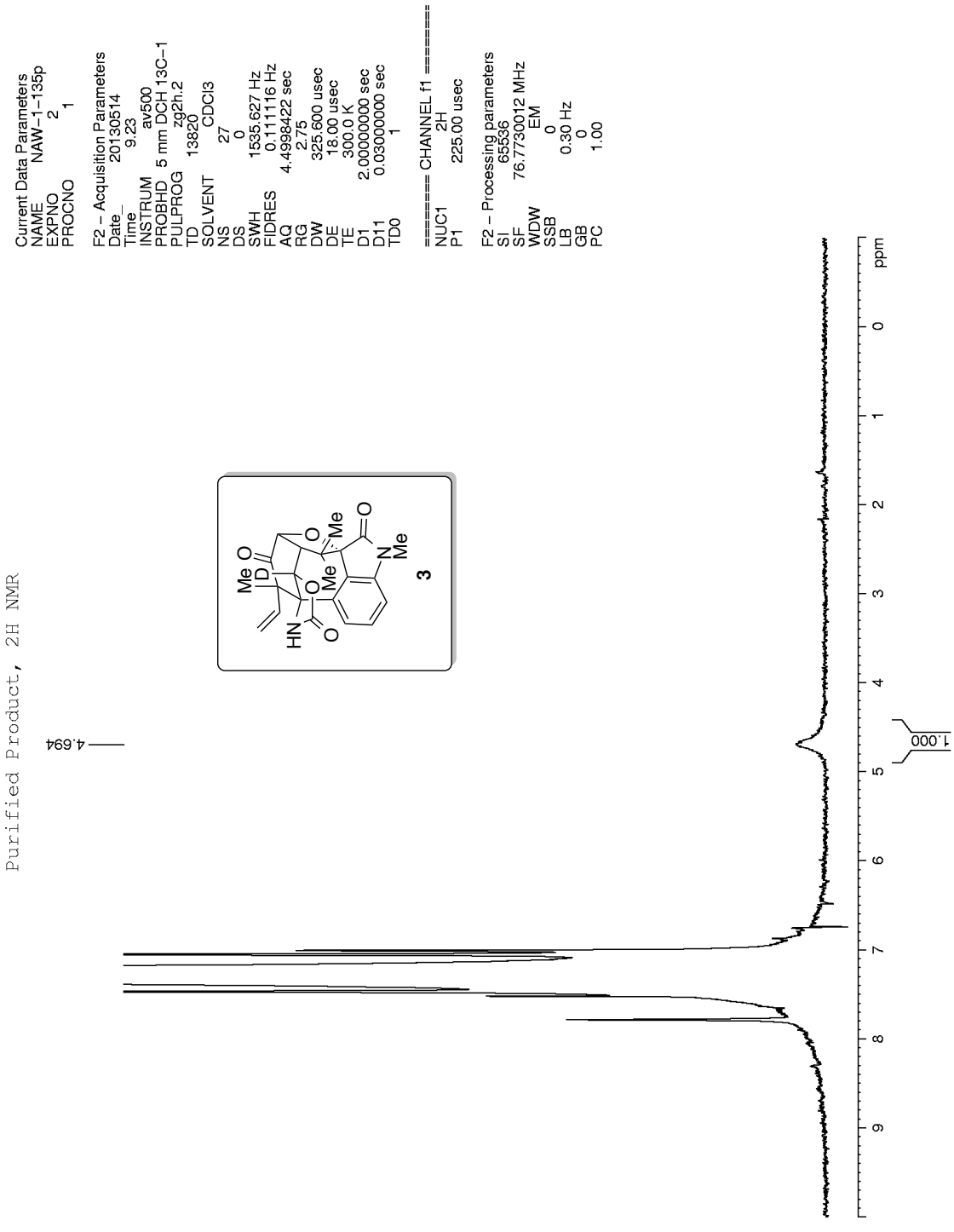

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-087
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date 20130417
Time 19.45
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.283
7.267
7.260
7.251
6.891
6.875
6.797
6.781
6.419
5.070
5.062
5.041
5.035
4.923
4.902
4.888
4.867
4.011
3.996
3.175
3.099
3.085
1.489
1.407
0.685
Me
Me
O
NMe
H
O
D
O
N
H
Me
13
ppm
0
1
2
3
4
5
6
7
8
9
3.012
3.092
3.007
1.017
3.055
0.992
1.007
2.001
0.984
1.012
1.000
1.079

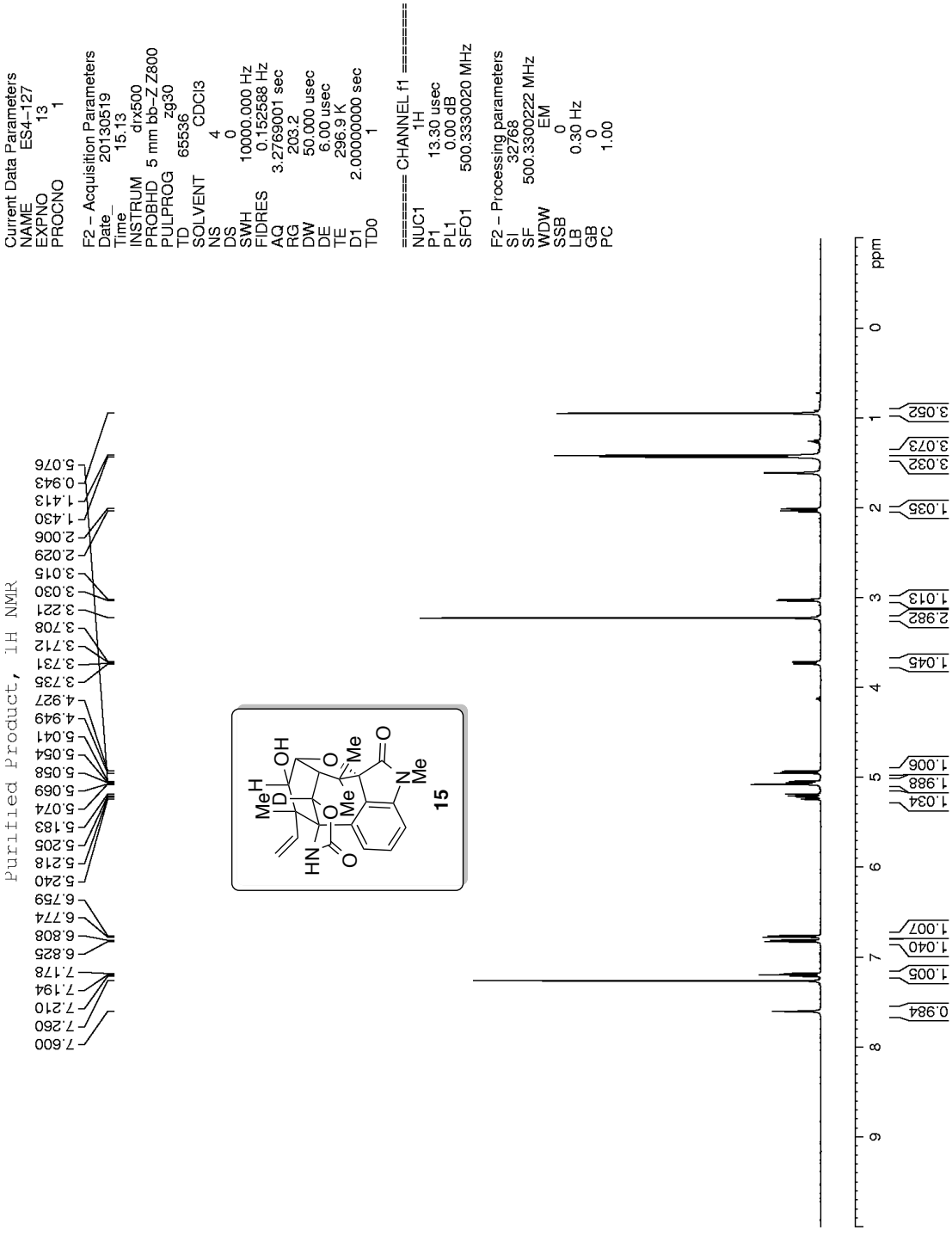

Purified Product, 2H NMR
5.334
Me
Me
O
NMe
H
O
H
D
O
O
N
H
Me
13
Current Data Parameters
NAME ES4-087
EXPNO 12
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130610
Time 18.21
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 16
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 – Processing parameters
SI 65536
SF 76.7729983 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
ppm

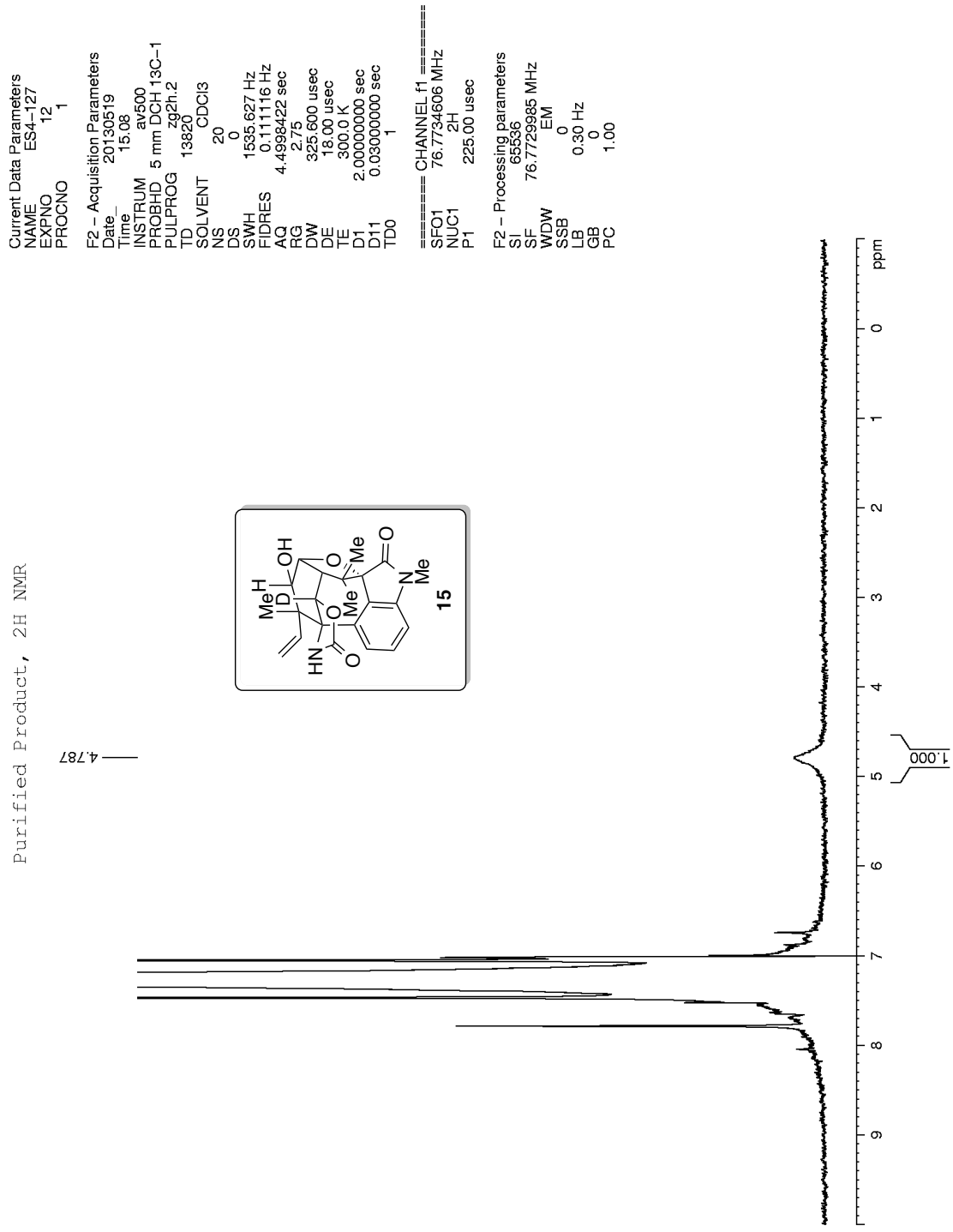

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-092
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130419
Time 9.29
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 228.1
DW 50.000 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.019
1.638
1.879
1.914
3.038
3.202
4.982
5.003
5.016
5.037
5.199
5.221
5.297
5.332
5.367
6.770
6.778
6.787
6.794
6.936
7.260
7.295
7.311
7.327
0.963
0.998
2.093
1.000
1.016
1.044
1.075
1.061
3.006
3.054
3.051
3.161
3.133
ppm
14 (R = OAc)

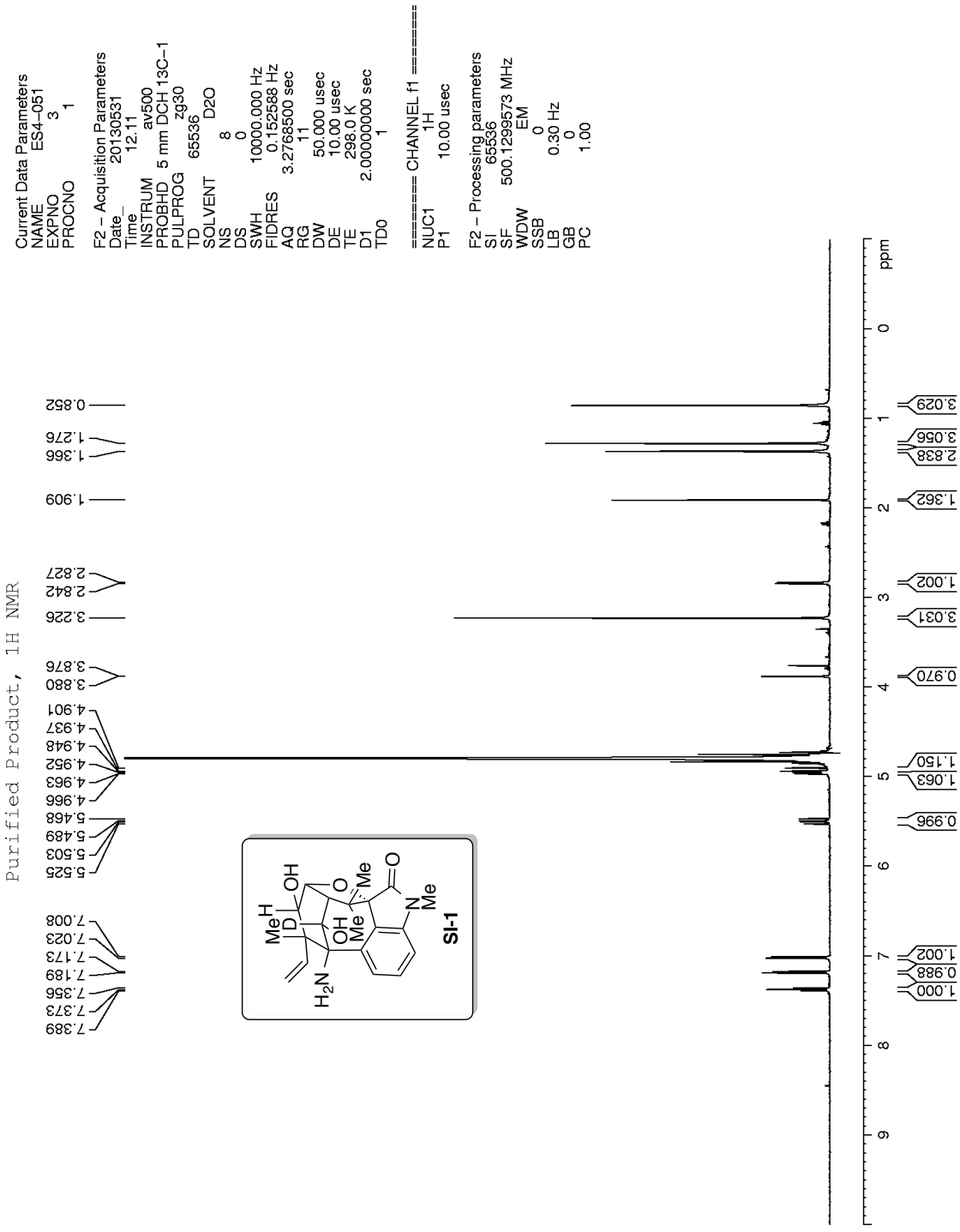

Purified Product, 2H NMR
5.867
Current Data Parameters
NAME ES4-134
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130610
Time 18.28
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 62
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 – Processing parameters
SI 65536
SF 76.7729986 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14 (R = OAc)
1.000
ppm

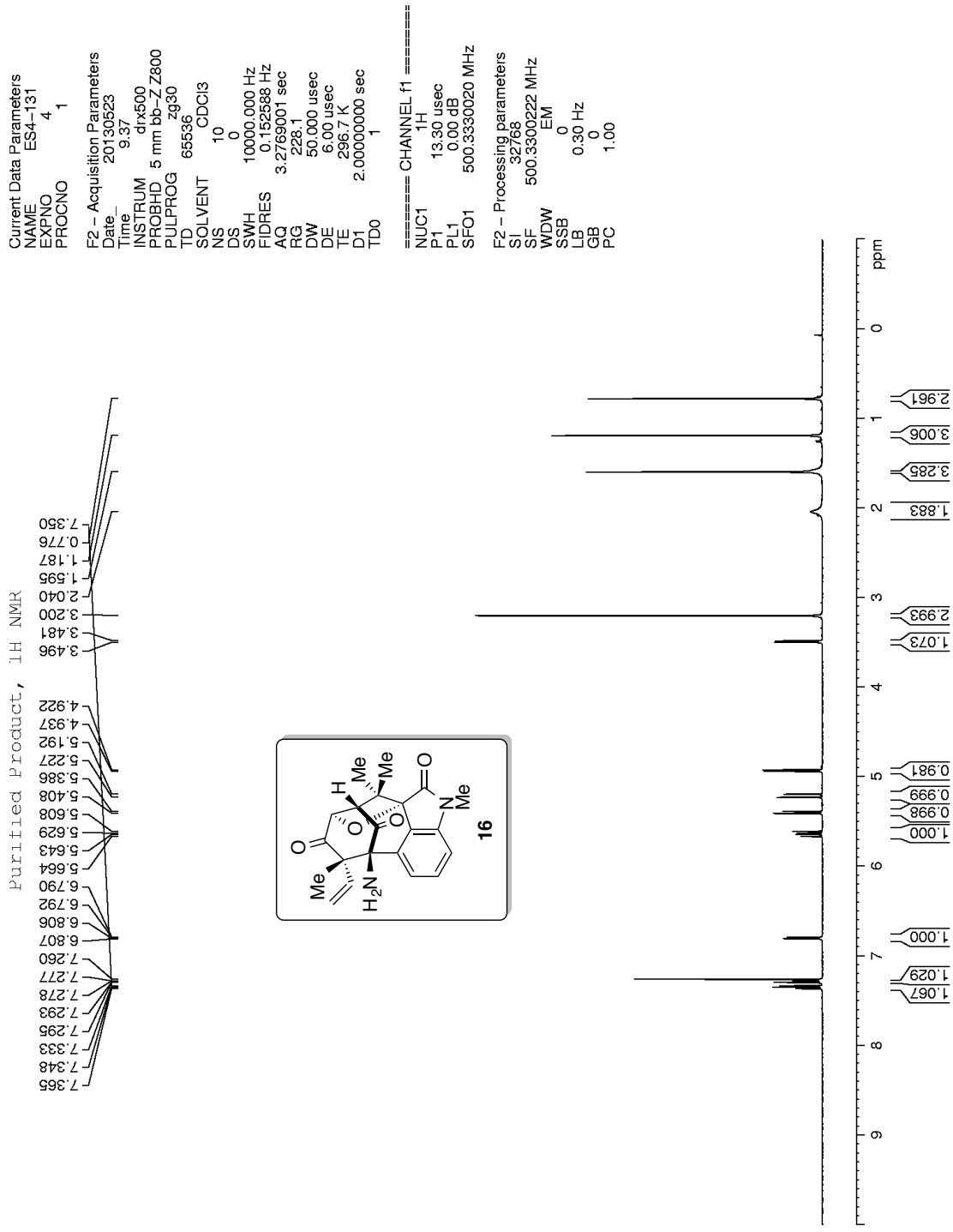

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-1-135p
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130513
Time 16.38
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 11
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
F2 – Processing parameters
SI 65536
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.667
7.270
7.260
7.254
7.239
6.939
6.924
6.814
6.799
5.248
5.226
5.213
5.192
4.946
4.937
4.931
4.903
4.786
4.765
3.303
3.288
3.230
1.446
1.416
1.025
0.938
0.905
1.000
1.006
0.989
2.026
1.021
1.019
2.990
2.965
3.023
2.961
ppm
3

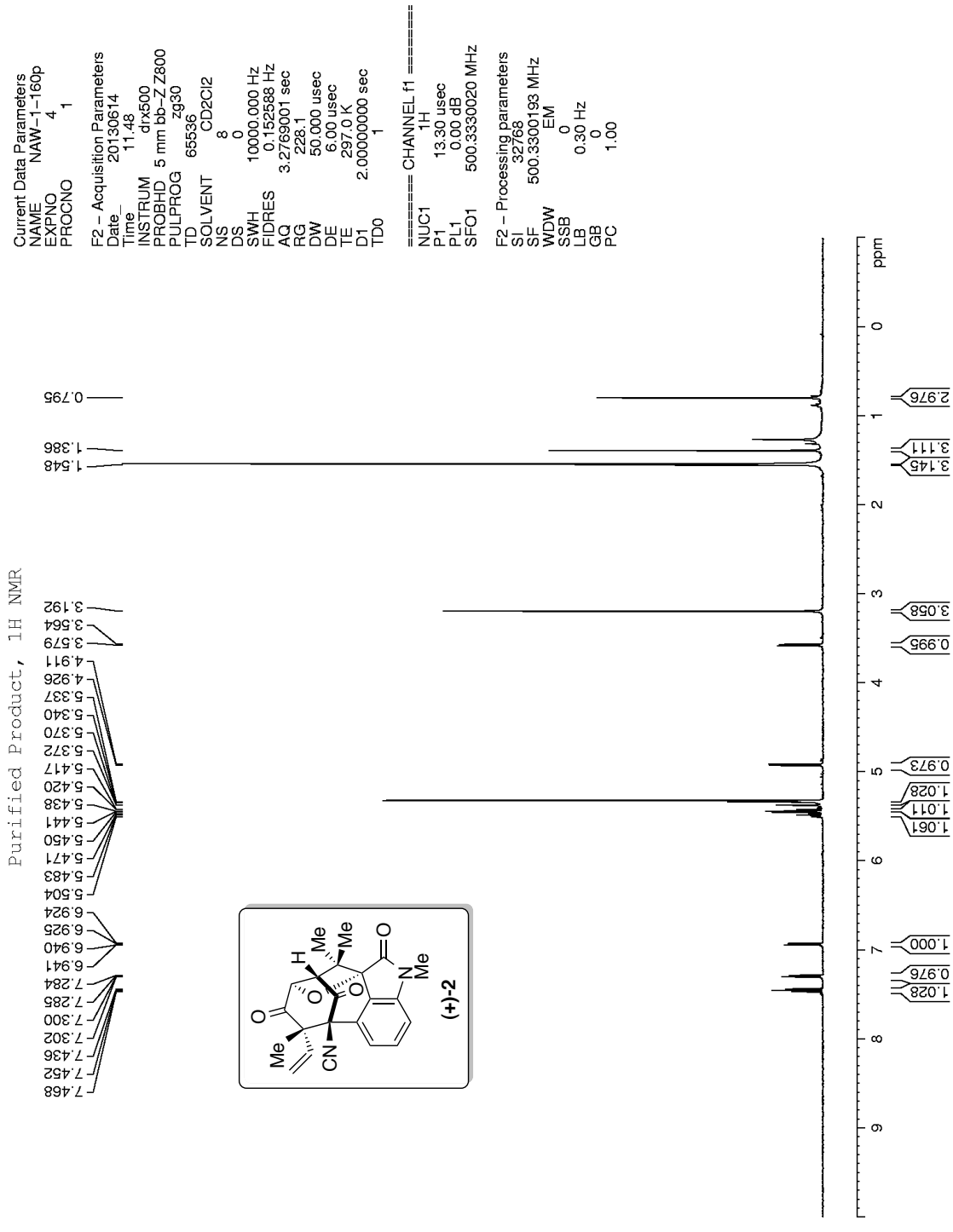

Purified Product, 2H NMR
4.694
Current Data Parameters
NAME NAW-1-135p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130514
Time 9.23
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 27
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 2H
P1 225.00 usec
F2 - Processing parameters
SI 65536
SF 76.7730012 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me O
D
HN
O
O
Me
O
Me
O
N
Me
3
1.000
ppm


Purified Product, 1H NMR
Current Data Parameters
NAME ES4-127
EXPNO 13
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130519
Time 15.13
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 203.2
DW 50.000 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15
ppm

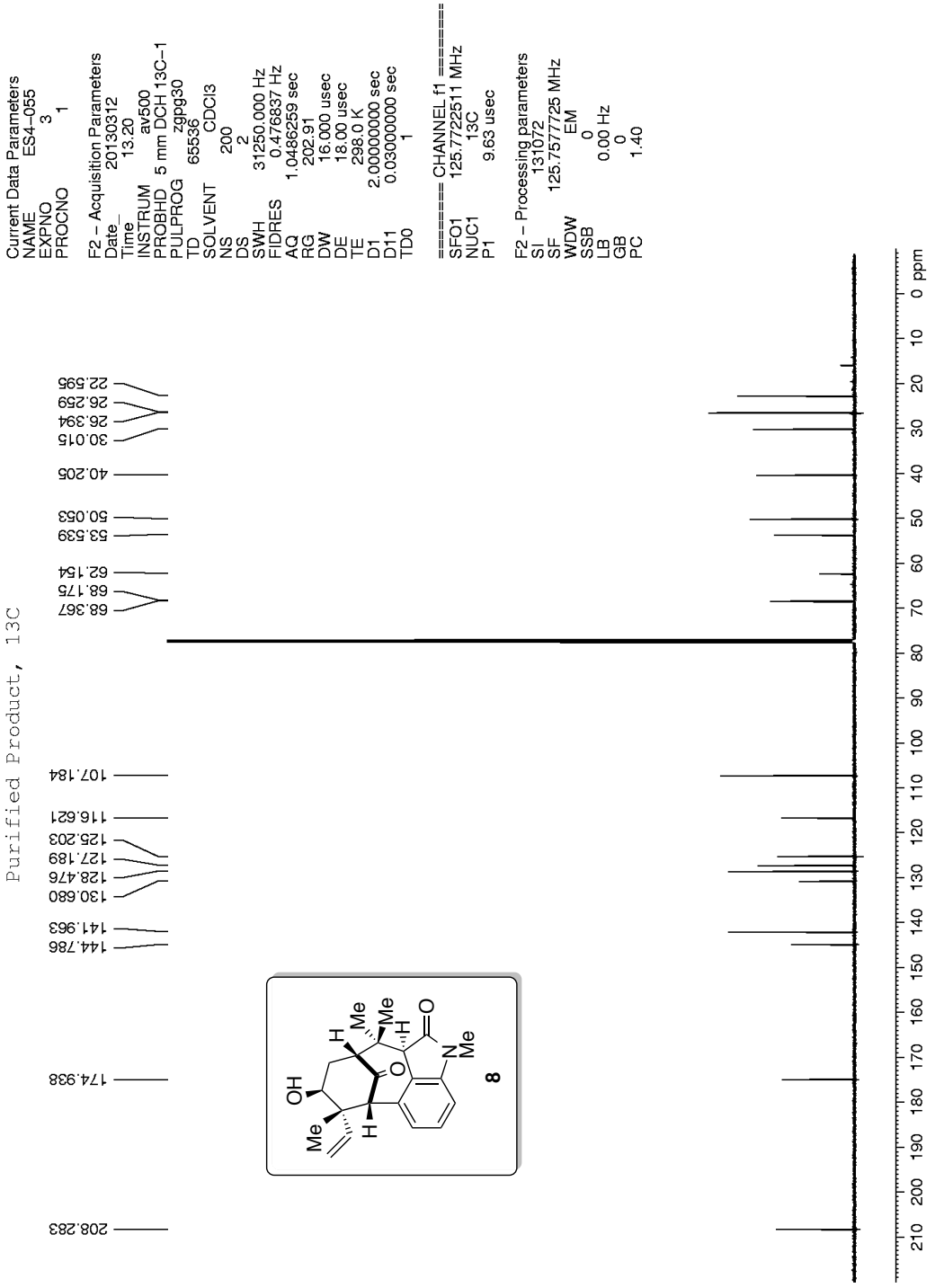

Purified Product, 2H NMR
4.787
Current Data Parameters
NAME ES4-127
EXPNO 12
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130519
Time_ 15.08
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zg2h.2
TD 13820
SOLVENT CDCl3
NS 20
DS 0
SWH 1535.627 Hz
FIDRES 0.111116 Hz
AQ 4.4998422 sec
RG 2.75
DW 325.600 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 76.7734606 MHz
NUC1 2H
P1 225.00 usec
F2 – Processing parameters
SI 65536
SF 76.7729985 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15
1.000
ppm

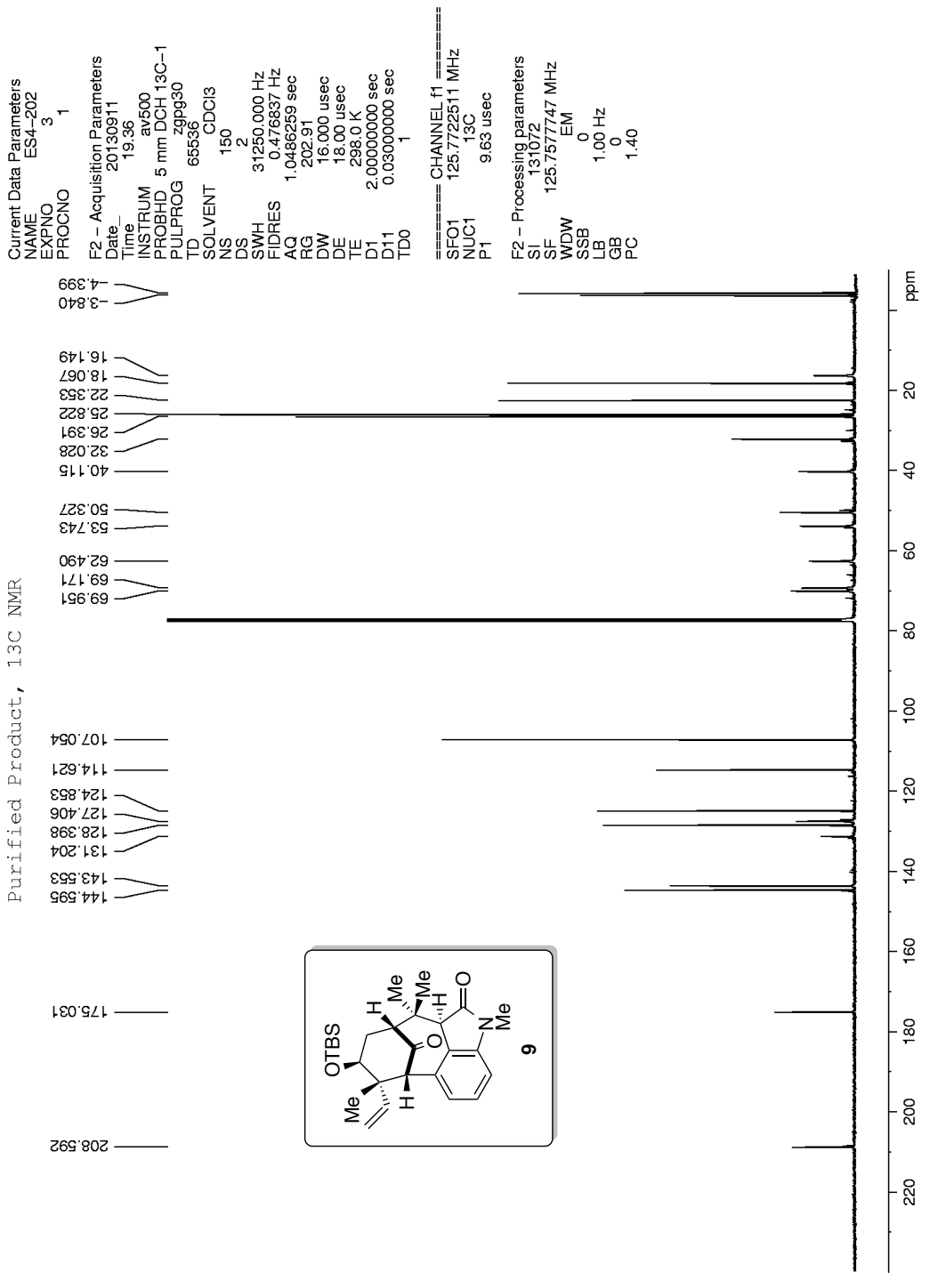

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-051
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130531
Time 12.11
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zg30
TD 65536
SOLVENT D2O
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 11
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
F2 – Processing parameters
SI 65536
SF 500.1299573 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
SI-1
ppm

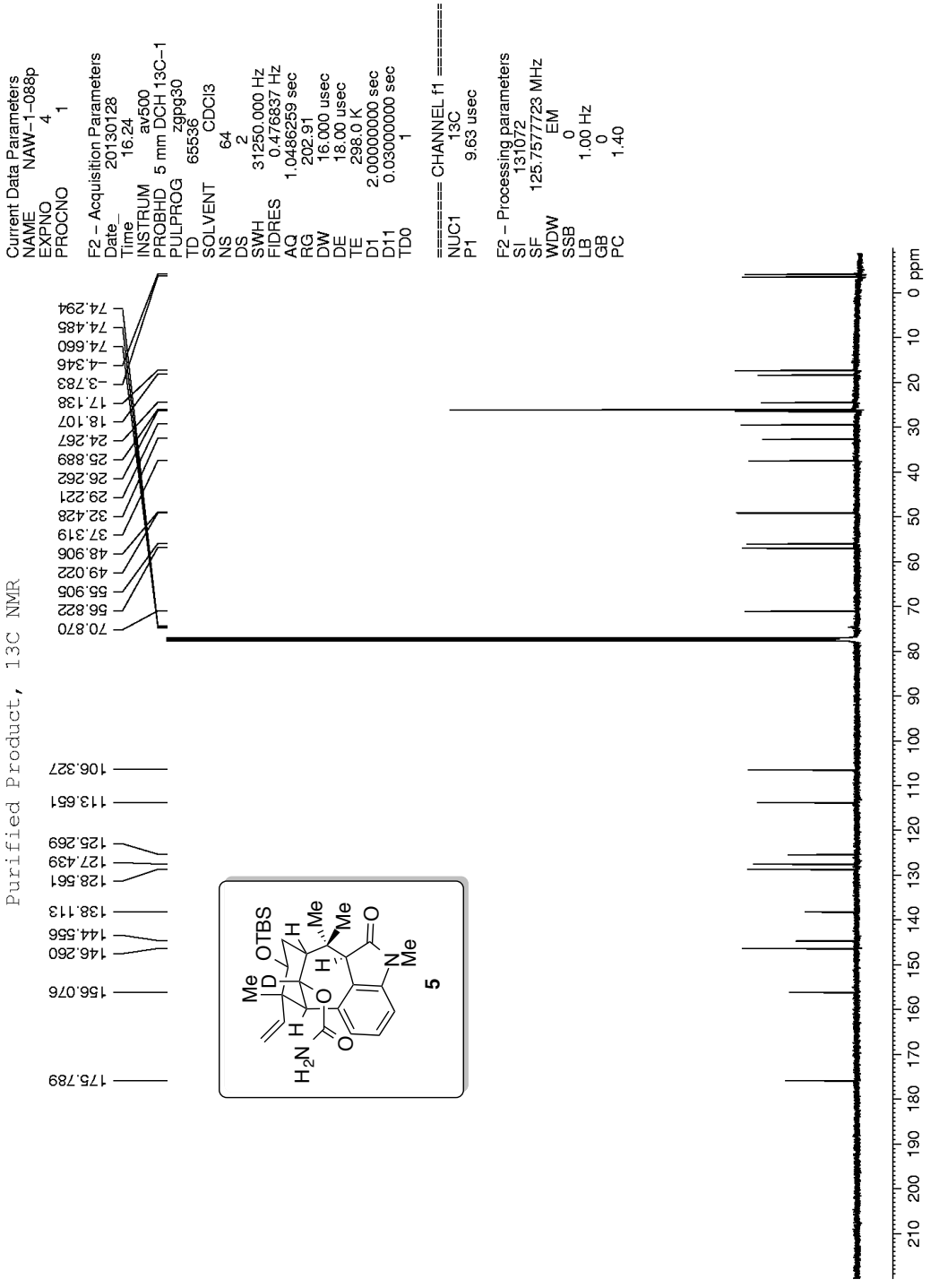

Purified Product, 1H NMR
Current Data Parameters
NAME ES4-131
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130523
Time 9.37
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 228.1
DW 50.000 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.365
7.348
7.333
7.295
7.293
7.278
7.277
7.260
6.807
6.806
6.792
6.790
5.664
5.643
5.629
5.608
5.408
5.386
5.227
5.192
4.937
4.922
3.496
3.481
3.200
2.040
1.595
1.187
0.776
7.350
Me
O
H
Me
Me
O
O
N
Me
H2N
16
ppm
1.067
1.029
1.000
0.981
0.999
0.998
1.000
1.073
2.993
1.883
3.285
3.006
2.961

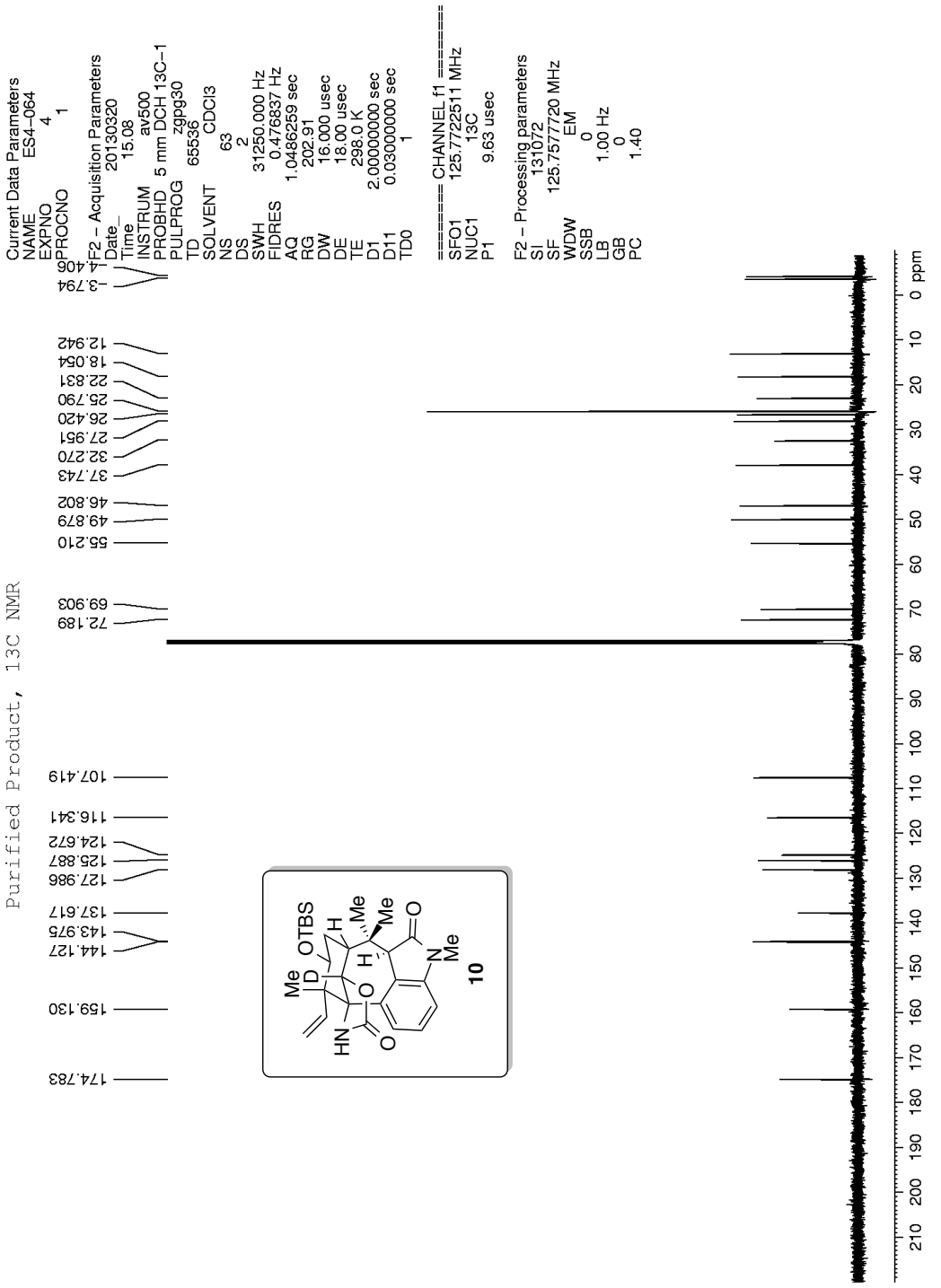

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-1-160p
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130614
Time 11.48
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 228.1
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.795
1.386
1.548
3.192
3.564
3.579
4.911
4.926
5.337
5.340
5.370
5.372
5.417
5.420
5.438
5.441
5.450
5.471
5.483
5.504
6.924
6.925
6.940
6.941
7.284
7.285
7.300
7.302
7.436
7.452
7.468
2.976
3.111
3.145
3.058
0.995
0.973
1.028
1.011
1.061
1.000
0.976
1.028
ppm
(+)-2

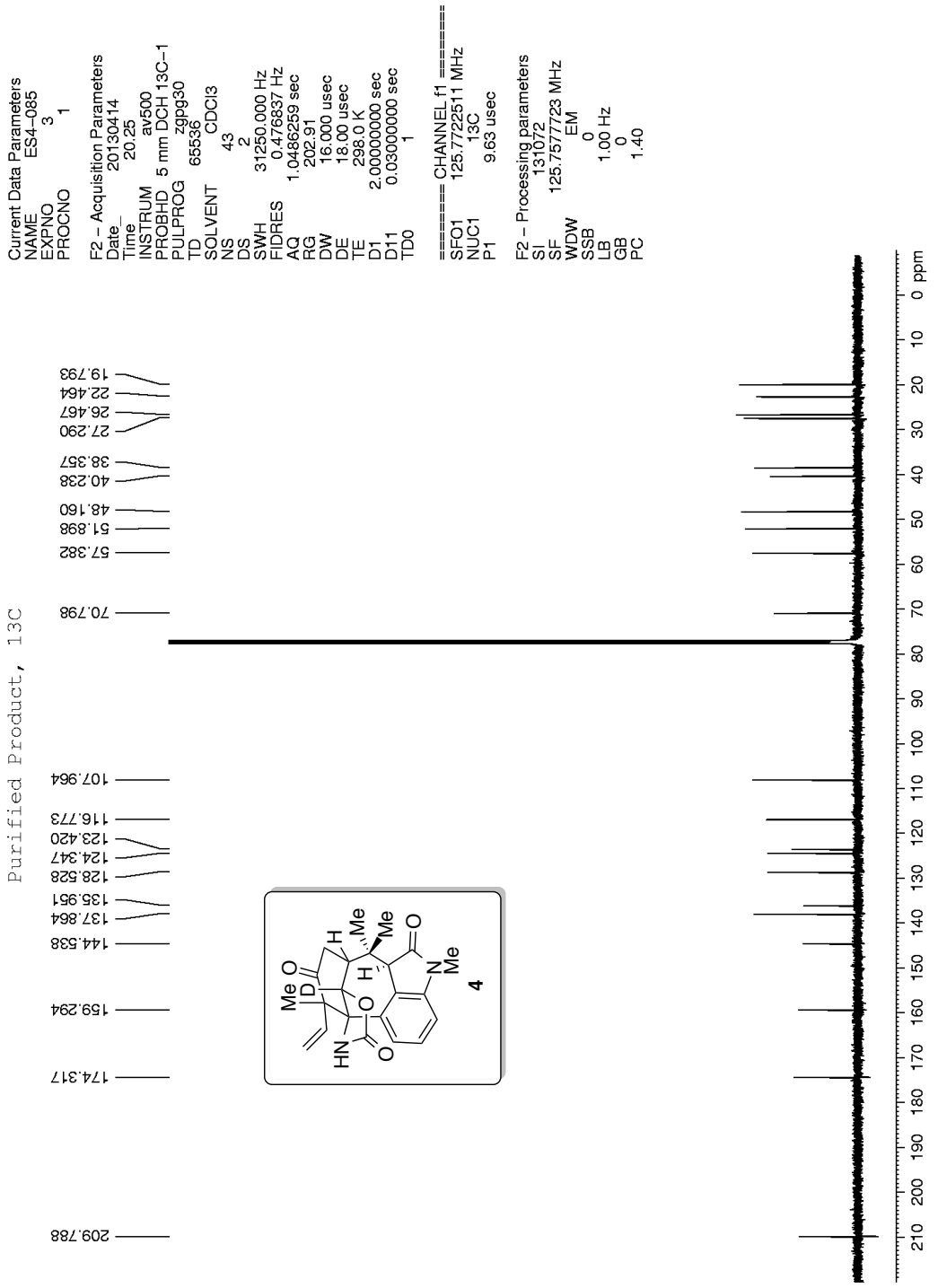
^{13}C NMR Spectra:

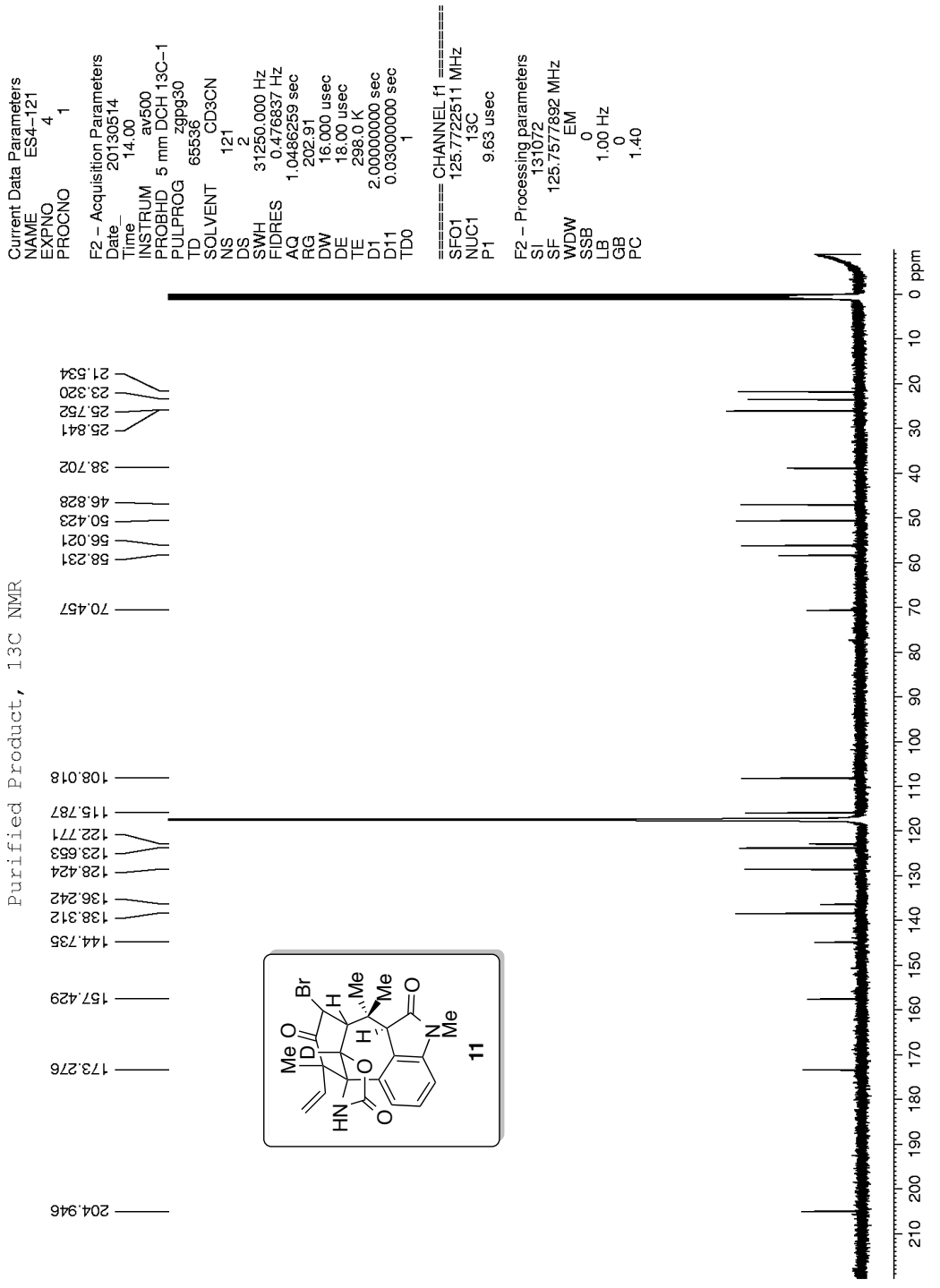

Purified Product, 13C
Current Data Parameters
NAME ES4-055
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130312
Time 13.20
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577725 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
208.283
174.938
144.786
141.963
130.680
128.476
127.189
125.203
116.621
107.184
68.367
68.175
62.154
53.539
50.053
40.205
30.015
26.394
26.259
22.595
OH
Me
H
Me
Me
H
O
O
N
Me
8
0 ppm

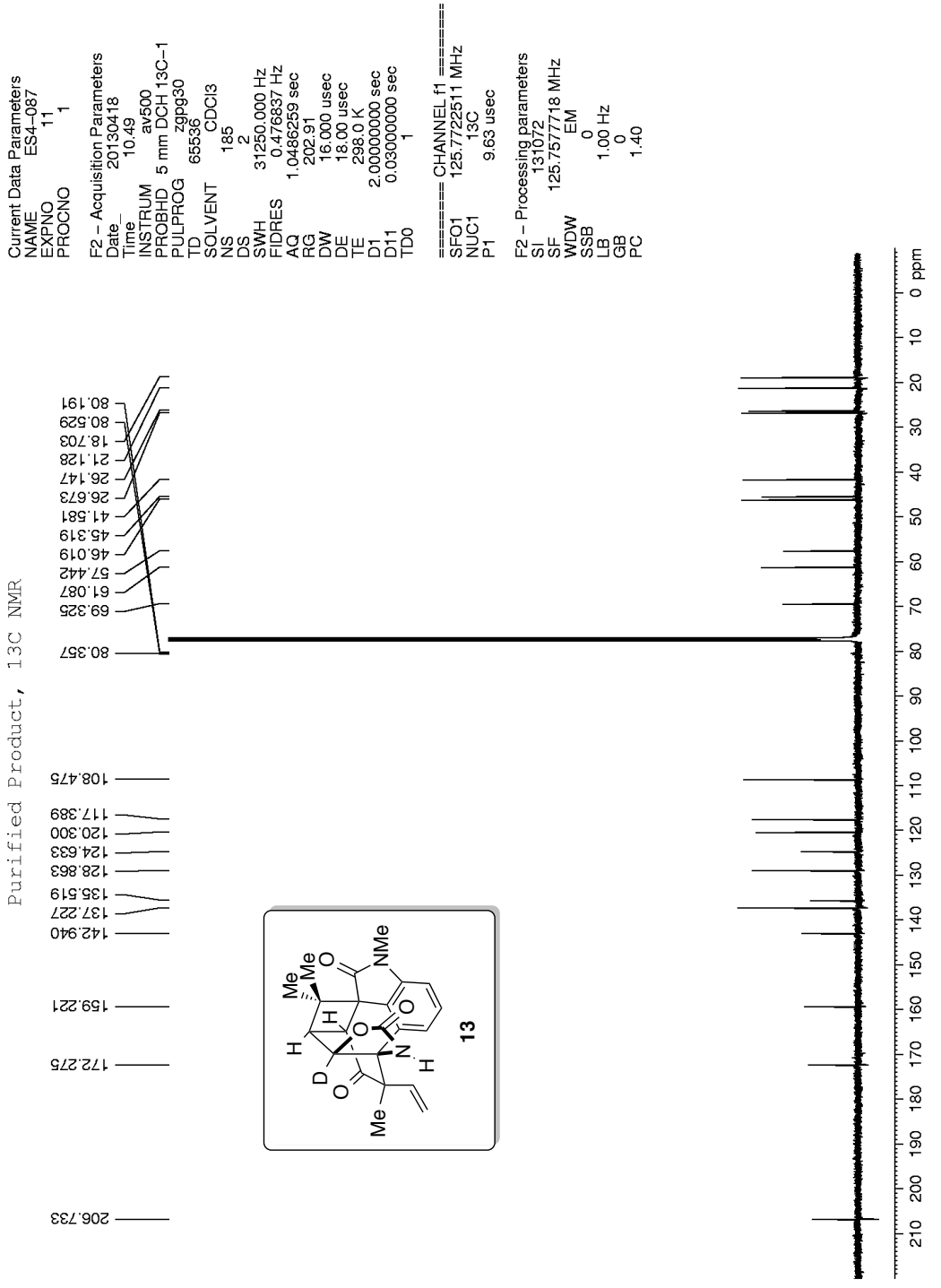

Purified Product, 13C NMR
Current Data Parameters
NAME ES4-202
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130911
Time 19.36
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 150
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577747 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-4.399
-3.840
16.149
18.067
22.353
25.822
26.391
32.028
40.115
50.327
53.743
62.490
69.171
69.951
107.054
114.621
124.853
127.406
128.398
131.204
143.553
144.595
175.031
208.592
OTBS
Me
H
Me
Me
H
O
O
N
Me
H
9
ppm

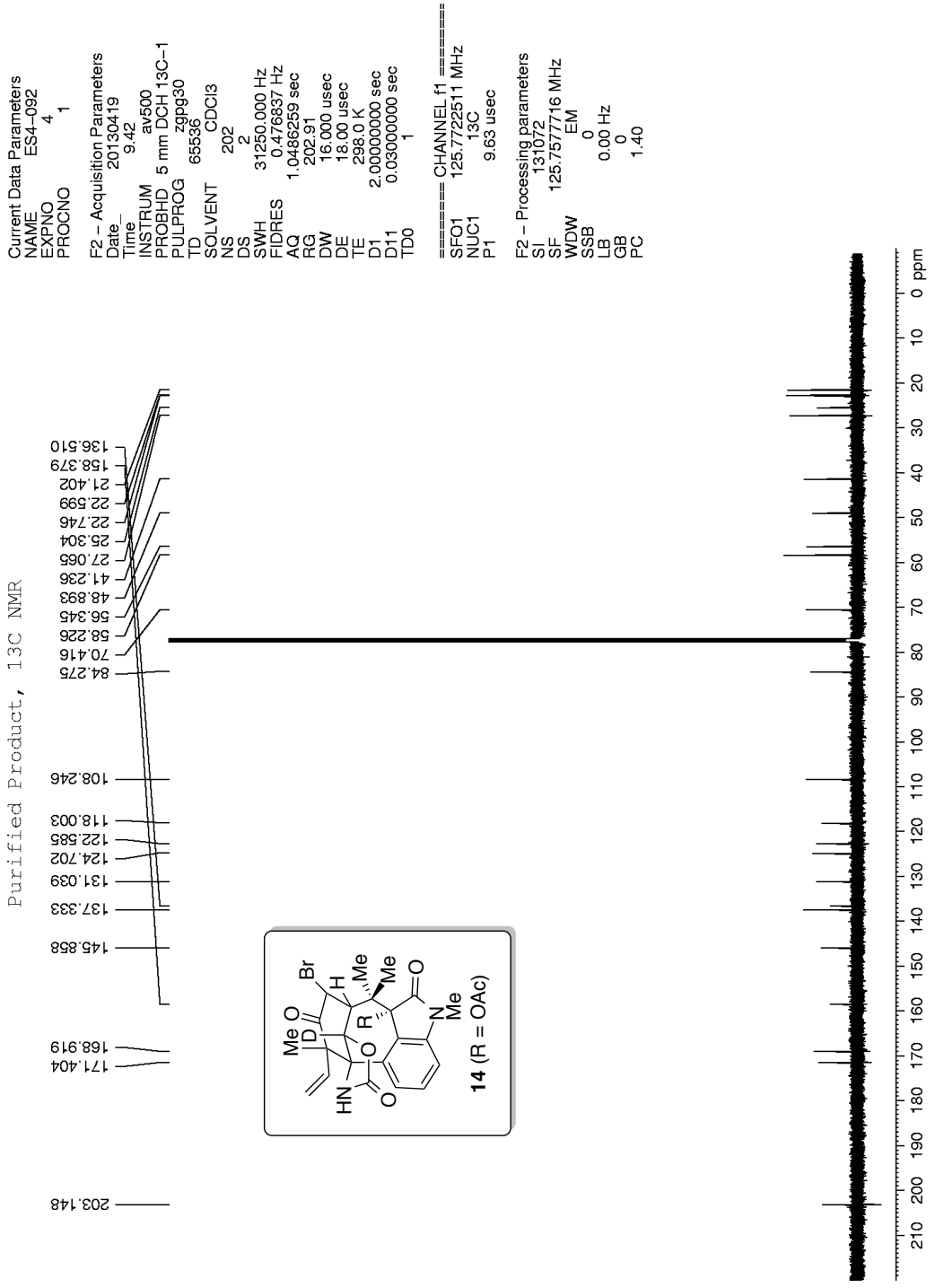

Purified Product, 13C NMR
Current Data Parameters
NAME NAW-1-088p
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130128
Time 16.24
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
70.870
56.822
55.905
49.022
48.906
37.319
32.428
29.221
26.262
25.889
24.267
18.107
17.138
-3.783
-4.346
74.660
74.485
74.294
106.327
113.651
125.269
127.439
128.561
138.113
144.556
146.260
156.076
175.789
OTBS
Me
D
H
Me
Me
H
O
O
N
Me
H2N
O
5
0 ppm

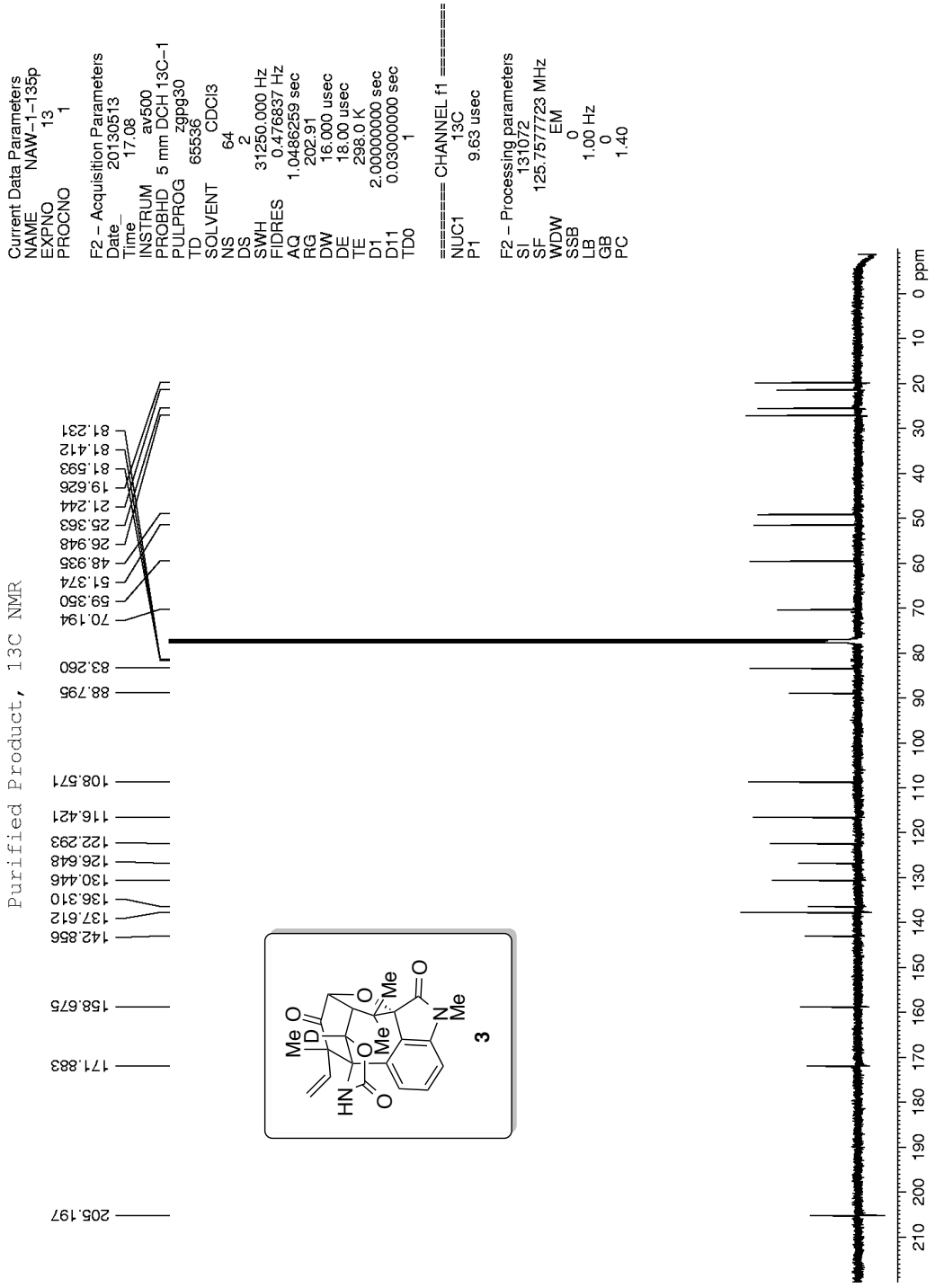

Purified Product, 13C NMR
Current Data Parameters
NAME ES4-064
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date 20130320
Time 15.08
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 63
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-3.794
4.406
12.942
18.054
22.831
25.790
26.420
27.951
32.270
37.743
46.802
49.879
55.210
69.903
72.189
107.419
116.341
124.672
125.887
127.986
137.617
143.975
144.127
159.130
174.783
10
0 ppm

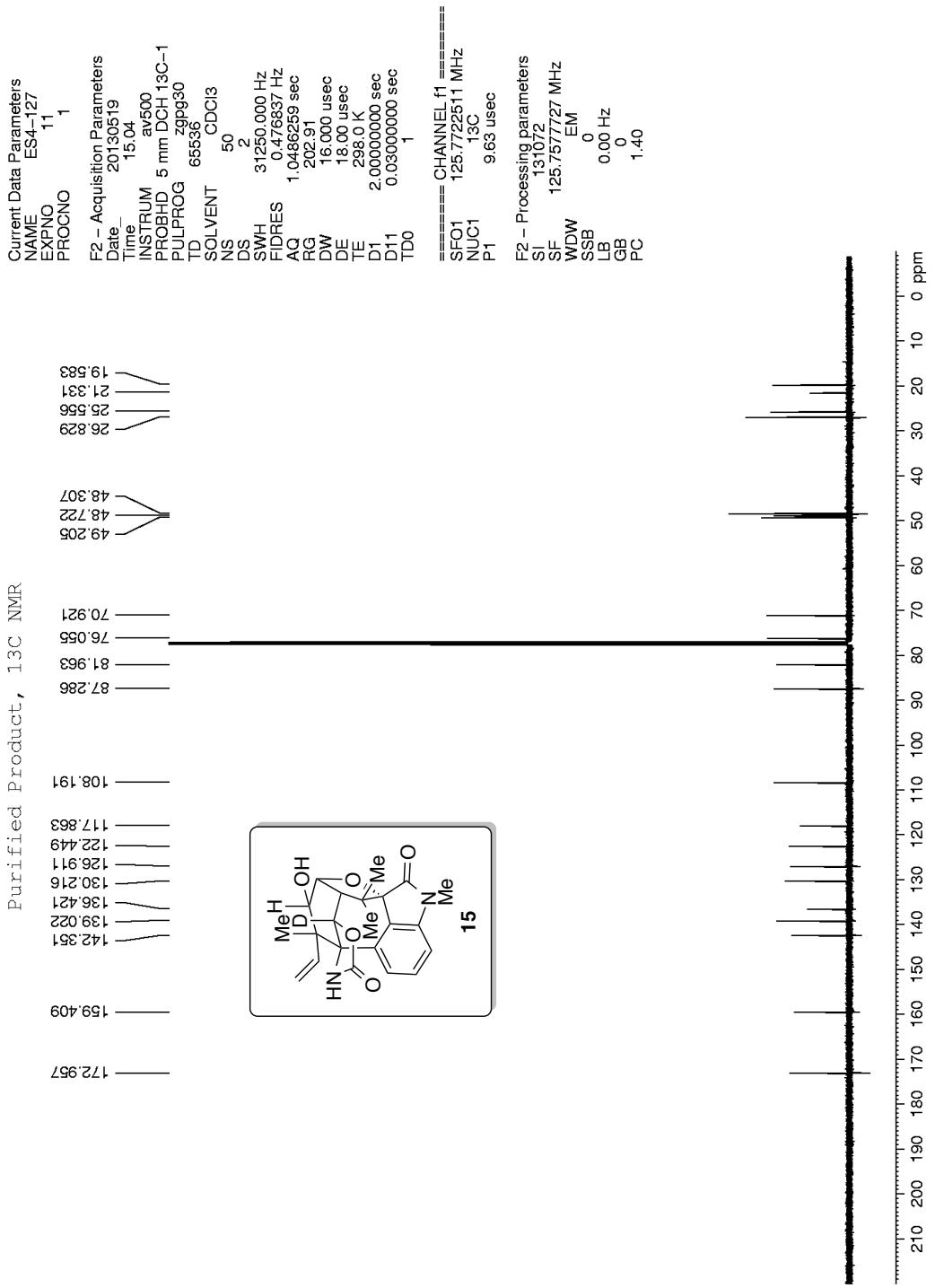

Purified Product, 13C
Current Data Parameters
NAME ES4-085
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130414
Time 20.25
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 43
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 - Processing parameters
SI 131072
SF 125.7577723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
209.788
174.317
159.294
144.538
137.864
135.951
128.528
124.347
123.420
116.773
107.964
70.798
57.382
51.898
48.160
40.238
38.357
27.290
26.467
22.464
19.793
Me O
D
H
Me
Me
O
H
N
Me
HN
O
O
4
0 ppm

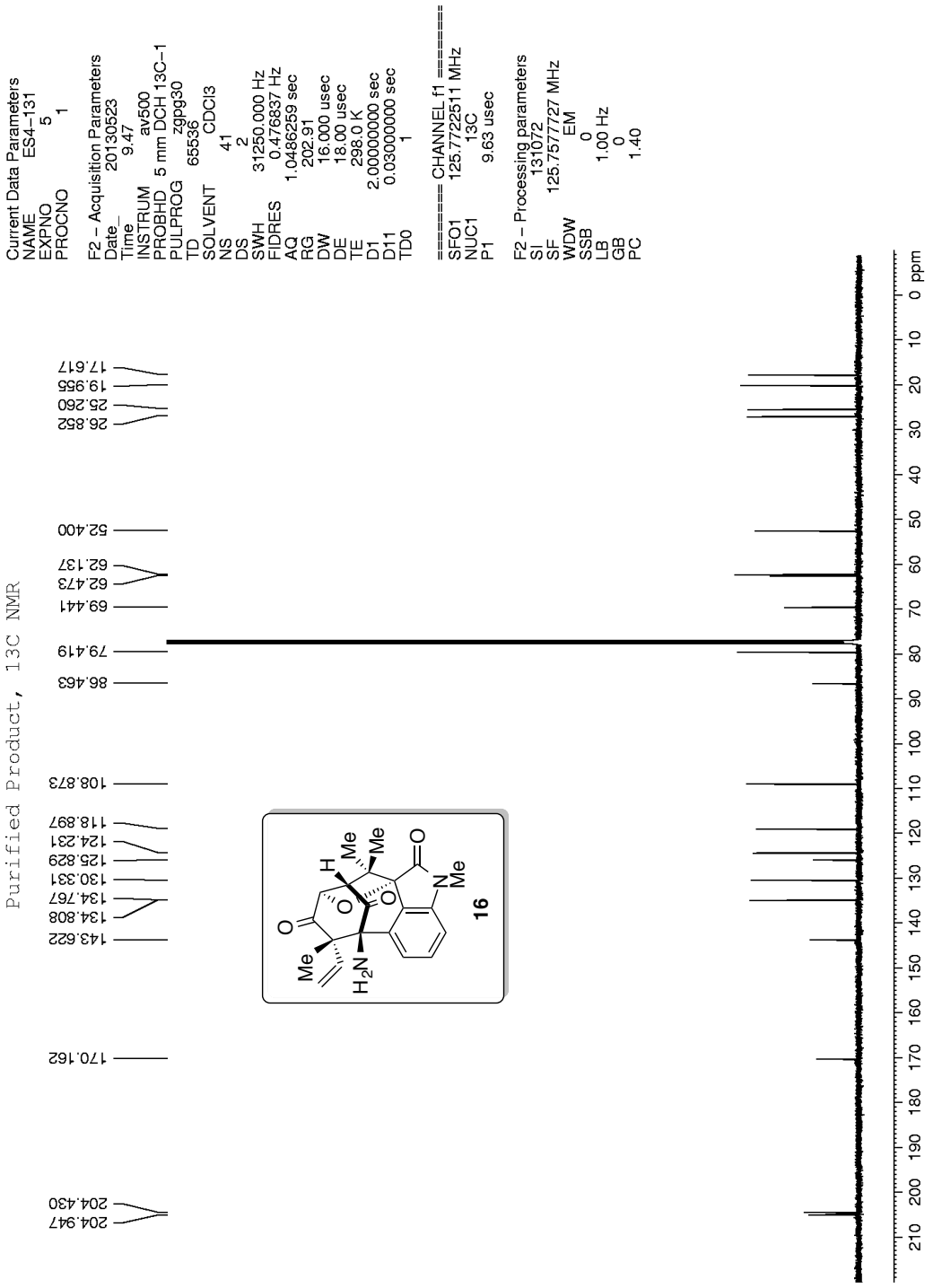

Purified Product, 13C NMR
Current Data Parameters
NAME ES4-121
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130514
Time 14.00
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 121
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 - Processing parameters
SI 131072
SF 125.7577892 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
204.946
173.276
157.429
144.735
138.312
136.242
128.424
123.653
122.771
115.787
108.018
70.457
58.231
56.021
50.423
46.828
38.702
25.841
25.752
23.320
21.534
Me O
D
Br
H
Me
Me
O
N
Me
H
O
HN
O
11
0 ppm

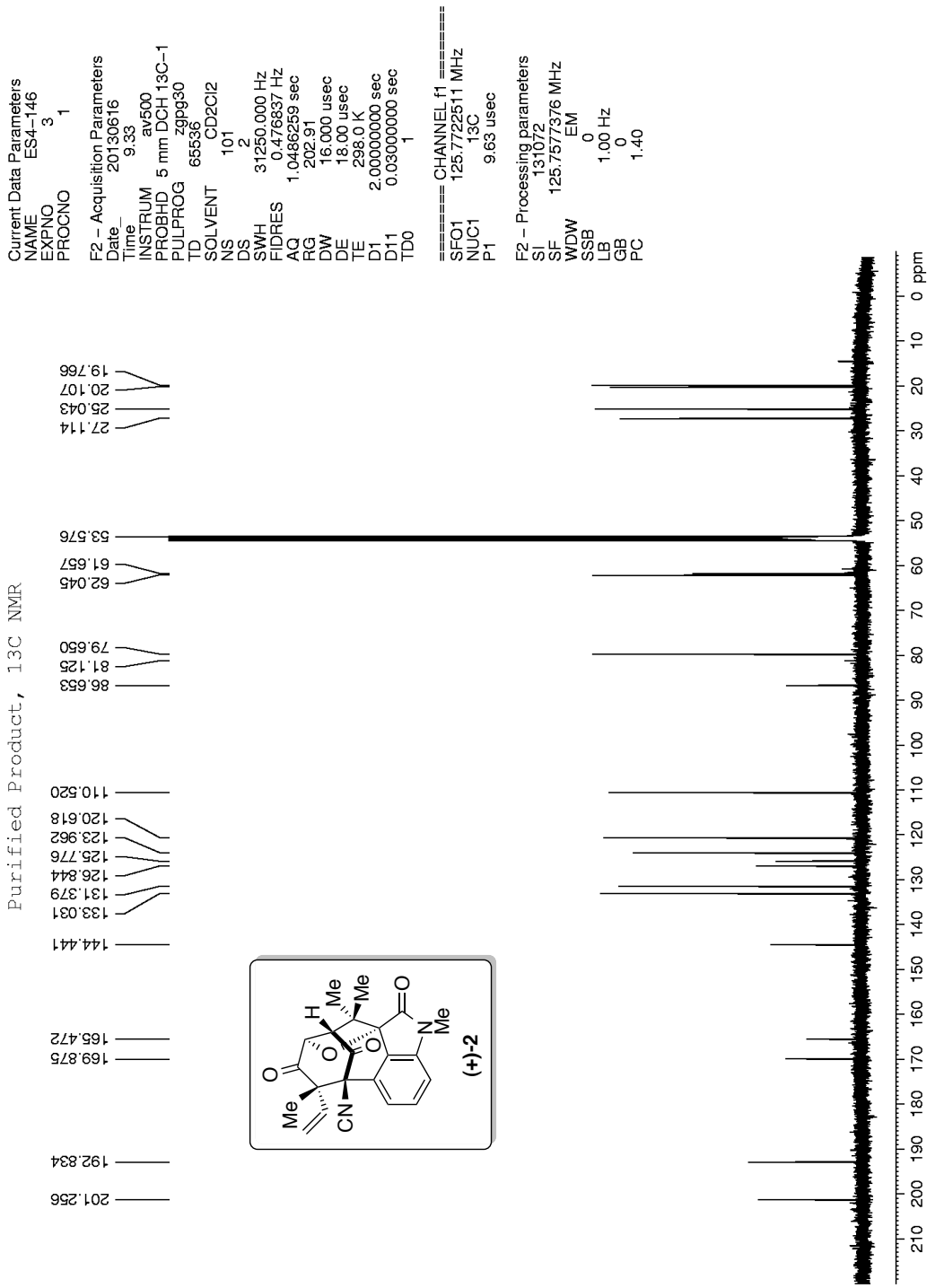

Purified Product, 13C NMR
Current Data Parameters
NAME ES4-087
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130418
Time 10.49
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 185
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
206.733
172.275
159.221
142.940
137.227
135.519
128.863
124.633
120.300
117.389
108.475
80.357
69.325
61.087
57.442
46.019
45.319
41.581
26.673
26.147
21.128
18.703
80.529
80.191
13
0 ppm


Purified Product, 13C NMR
Current Data Parameters
NAME ES4-092
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20130419
Time 9.42
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 202
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577716 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
203.148
171.404
168.919
145.858
137.333
131.039
124.702
122.585
118.003
108.246
84.275
70.416
58.226
56.345
48.893
41.236
27.065
25.304
22.746
22.599
21.402
158.379
136.510
14 (R = OAc)
0 ppm


Purified Product, 13C NMR
Current Data Parameters
NAME NAW-1-135p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130513
Time 17.08
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.63 usec
F2 - Processing parameters
SI 131072
SF 125.7577723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
205.197
171.883
158.675
142.856
137.612
136.310
130.446
126.648
122.293
116.421
108.571
88.795
83.260
70.194
59.350
51.374
48.935
26.948
25.363
21.244
19.626
81.593
81.412
81.231
Me O
D
HN
O
O
Me
Me
O
N
Me
3
0 ppm


Purified Product, 13C NMR
Current Data Parameters
NAME ES4-127
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date 20130519
Time 15.04
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577727 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
19.583
21.331
25.556
26.829
48.307
48.722
49.205
70.921
76.055
81.963
87.286
108.191
117.863
122.449
126.911
130.216
136.421
139.022
142.351
159.409
172.957
15
0 ppm


Purified Product, 13C NMR
Current Data Parameters
NAME ES4-131
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130523
Time 9.47
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 41
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 - Processing parameters
SI 131072
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
204.947
204.430
170.162
143.622
134.808
134.767
130.331
125.829
124.231
118.897
108.873
86.463
79.419
69.441
62.473
62.137
52.400
26.852
26.260
19.955
17.617
16
0 ppm


Purified Product, 13C NMR
Current Data Parameters
NAME ES4-146
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date 20130616
Time 9.33
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 101
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 202.91
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19.766
20.107
26.043
27.114
53.576
61.657
62.045
79.650
81.125
86.653
110.520
120.618
123.962
125.776
126.844
131.379
133.031
144.441
165.472
169.875
192.834
201.256
(+)-2
0 ppm