Total Synthesis of Oxidized Welwitindolinones and (–)-*N*-Methylwelwitindolinone C Isonitrile

Kyle W. Quasdorf, Alexander D. Huters, Michael W. Lodewyk[†], Dean J. Tantillo[†], and Neil K. Garg*

Department of Chemistry and Biochemistry, University of California, Los Angeles, CA 90095

and

[†]*Department of Chemistry, University of California, Davis, One Shields Avenue, Davis, CA 95616*

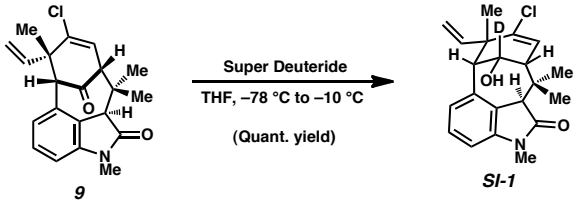
Supporting Information – Table of Contents

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. (*S*)-carvone was obtained from Aldrich. 5-bromoindole was obtained from Biosynth. $NaNH_2$ was obtained from Alfa Aesar. Comins' reagent was obtained from Aldrich. Hexamethylditin was obtained from Aldrich. Tetrakis(triphenylphosphine)palladium(0) was obtained from Strem. Anhydrous $CuCl_2$ was obtained from Aldrich. Trichloroacetyl isocyanate was obtained from Aldrich. $LiEt_3BD$ ("super deuteride") was obtained from Aldrich. AgOTf was obtained from Strem. Bathophenanthroline was obtained from Alfa Aesar. *O*,*O*-di(2-pyridinyl) thiocarbonate was obtained from Aldrich. 2-Iodoxybenzoic acid (IBX) and Dess–Martin periodinane were prepared from known literature procedures.[1,2] *t*-BuOH was distilled from CaH_2 and stored in a Schlenk tube prior to use. 1,4-dioxane was distilled from Na/benzophenone prior to use. 1,2-dichloroethane was distilled from P_2O_5 and stored in a Schlenk tube over 4Å molecular sieves 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 $CDCl_3$ and 5.32 ppm for CD_2Cl_2. Data for ^{2}H NMR spectra are reported as follow: 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 77.16 ppm for $CDCl_3$, 53.84 for CD_2Cl_2, and 128.06 for C_6D_6. 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 Rudolf Autopol IV Automatic Polarimeter. Uncorrected melting points were measured with a Mel-Temp II melting point apparatus and a Fluke 50S thermocouple. High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility.

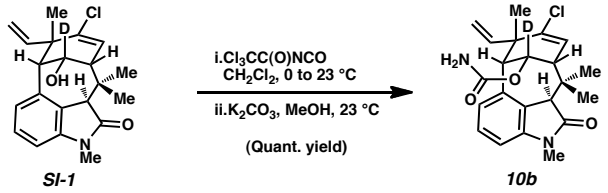
Complete Reference 4s from manuscript:

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

Experimental Procedures.

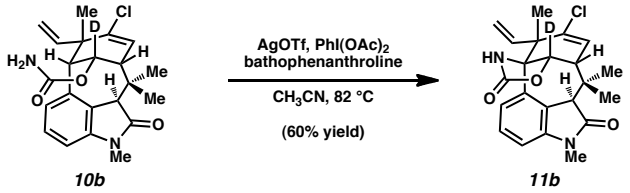


SI-1. To a solution of ketone **9**[3] (367 mg, 1.03 mmol, 1.0 equiv) in THF (34.0 mL) at –78 °C was added a solution of $LiEt_3BD$ ("super deuteride", 1.0 M in THF, 1.13 mL, 1.13 mmol, 1.1 equiv) in a dropwise manner. After stirring at –78 °C for 10 min the reaction was warmed to –10 °C and stirred for an additional 1 h. The reaction was then quenched with the addition of MeOH (5 mL) and warmed to room temperature. The resulting mixture was transferred to a separatory funnel with EtOAc (50 mL), H_2O (15 mL), and brine (25 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 50 mL), the organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (1:1:1 hexanes:CH_2Cl_2:Et_2O) to afford **SI-1** (370 mg, quant. yield) as a white solid. **SI-1**: R_f 0.12 (2:1:1 hexanes:CH_2Cl_2:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.17 (ddd, J = 7.8, 7.7, 0.9, 1H), 6.70 (d, J = 7.8, 0.9 1H), 6.68 (d, J = 7.7, 1H), 6.19 (d, J = 6.7, 1H), 5.23 (dd, J = 17.4, 10.7, 1H), 5.03 (dd, J = 17.4, 0.7, 1H), 4.89 (dd, J = 10.7, 0.7, 1H), 3.62 (s, 1H), 3.18 (s, 3H), 3.13 (d, J = 0.9, 1H), 2.57 (dd, J = 6.7, 0.9, 1H), 1.57 (s, 3H), 1.53 (s, 3H), 0.95 (s, 3H).

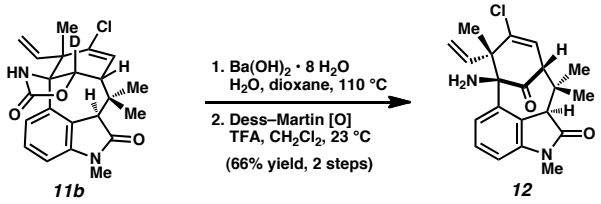


Carbamate 10b. To a solution of **SI-1** (370 mg, 1.03 mmol, 1.0 equiv) in CH_2Cl_2 (21.0 mL) at 0 °C was added trichloroacetyl isocyanate (129 μL, 1.08 mmol, 1.05 equiv) in a dropwise manner. The resulting mixture was stirred at 0 °C for 5 min, and then at room temperature for an additional 20 min. The solvent was evaporated under reduced pressure. To the resulting residue was added MeOH (21.0 mL) and solid K_2CO_3 (784 mg, 5.67 mmol, 5.5 equiv) in one portion. The reaction was flushed with N_2 and left to stir at room temperature for 3.5 h. The reaction was quenched with a saturated aqueous NH_4Cl solution (50 mL) and the resulting biphasic mixture was transferred to a separatory funnel with EtOAc (50 mL) and H_2O (25 mL). After extracting with EtOAc (3 x 50 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 carbamate **10b** (416 mg, quant. yield) as a white solid. Carbamate **10b**: mp: 135 ºC; R_f 0.41 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.13 (dd, J = 7.8, 7.7, 1H), 6.68 (d, J = 7.7, 1H), 6.61 (d, J = 7.8, 1H), 6.18 (d, J = 6.7, 1H), 5.19 (dd, J = 17.3,

10.6, 1H), 5.04 (dd, *J* = 17.3, 0.8, 1H), 4.91 (dd, *J* = 10.6, 0.8, 1H), 4.46 (br. s, 2H), 3.62 (s, 3H), 3.17 (s, 3H), 3.14 (s,1H), 2.77 (dd, *J* = 6.7, 0.9, 1H), 1.60 (s, 3H), 1.52 (s, 3H), 0.87 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.48 (br. s, 1D); ^{13}C NMR (21 of 22 observed, 125 MHz, $CDCl_3$): δ 176.3, 155.9, 144.3, 141.2, 141.0, 136.9, 127.7, 127.3, 126.4, 125.7, 114.7, 106.6, 55.8, 52.6, 50.6, 49.0, 38.7, 28.0, 26.3, 26.2, 22.7; IR (film): 3493, 3351, 2929, 2875, 1723, 1698, 1609, 1469, 1375, 1084 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^{+}$ calcd for $C_{22}H_{24}DN_2O_3ClNa$, 424.1514; found 424.1504; $[\alpha]^{24.2}_{D}$ –151.0° (*c* = 1.000, CH_2Cl_2).

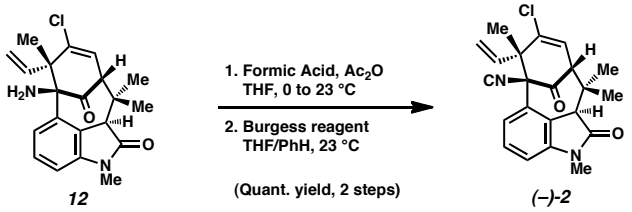


Oxazolidinone 11b. A 20 mL scintillation vial containing CH_3CN and two separate 20 mL scintillation vials each charged with bathophenanthroline (40.1 mg, 0.124 mmol, 0.5 equiv) were transferred into the glovebox. AgOTf (32.0 mg, 0.124 mmol, 0.5 equiv) and CH_3CN (7.00 mL) were added to each vial containing the bathophenanthroline, and the resulting suspensions were stirred at room temperature for 20 min. Next, two additional 20 mL scintillation vials each containing carbamate **10b** (100 mg, 0.249 mmol, 1.0 equiv) and $PhI(OAc)_2$ (160 mg, 0.498 mmol, 2.0 equiv) were transferred into the glovebox and a AgOTf/bathophenanthroline suspension was added to each of these vials. The vials were then sealed, removed from the glovebox, and the resulting mixtures were allowed to stir at 82 °C. After 24 h, the reactions were cooled to room temperature and combined then filtered through a plug of silica gel (EtOAc eluent, 50 mL). The filtrate was evaporated under reduced pressure, and the resulting residue was purified by flash chromatography (4:1 benzene:EtOAc) to afford oxazolidinone **11b** (59.3 mg, 60% yield) as a white solid and recovered ketone **9** (7.0 mg, 8% yield) as a white solid. Oxazolidinone **11b**: mp: 329 ºC; R_f 0.35 (2:1 benzene:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53 (br. s, 1H), 7.15 (ddd, *J* = 8.3, 7.6, 0.7 1H), 6.72 (d, *J* = 8.3, 1H), 6.71 (d, *J* = 7.6, 1H), 6.29 (d, *J* = 5.9, 1H), 5.19–5.05 (m, 3H), 3.62 (s, 1H), 3.19 (s, 3H), 2.97 (d, *J* = 5.9, 1H), 1.65 (s, 3H), 1.56 (s, 3H), 1.04 (s, 3H); ^{2}H NMR (77 MHz, $CDCl_3$) δ 5.02 (br. s, 1D); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.9, 159.2, 144.2, 141.1, 140.6, 136.8, 128.2, 125.9, 125.5, 123.9, 116.4, 107.4, 80.9 (t, $J_{C–D}$ = 21.9), 69.8, 54.2, 52.1, 49.4, 38.6, 27.3, 26.4, 22.0, 20.2; IR (film): 3280, 2997, 1757, 1707, 1610, 1460, 1346 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^{+}$ calcd for $C_{22}H_{22}DN_2O_3ClNa$, 422.1358; found 422.1357; $[\alpha]^{25.2}_{D}$ –147.6° (*c* = 1.000, CH_2Cl_2).



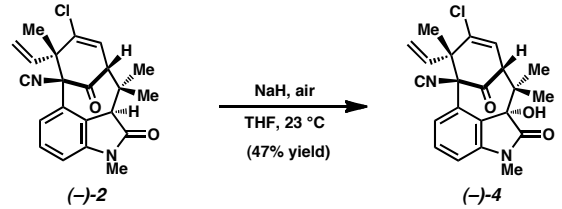
Aminoketone 12. A Schlenk tube was charged with oxazolidinone **11b** (20 mg, 0.050 mmol, 1.0 equiv) and $Ba(OH)_2 \cdot 8\ H_2O$ (79 mg, 0.250 mmol, 5.0 equiv). The reaction vessel was then evacuated and backfilled with N_2 five times. A 2:1 mixture of 1,4-dioxane:H_2O (1.9 mL) that had been taken through seven freeze-pump-thaw cycles prior to use was then added to the Schlenk tube. The vessel was sealed, and the reaction vessel was heated to 110 °C. After 14 h, the reaction was cooled to room temperature, and the contents were transferred to a test tube with EtOAc (6 mL), H_2O (3 mL), and brine (3 mL). The resulting biphasic mixture was extracted with EtOAc (5 x 5 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 the crude residue was added CH_2Cl_2 (1.1 mL) and TFA (4.2 μL, 0.0413 mmol, 1.1 equiv). The resulting solution was stirred at room temperature for 2 min. Dess–Martin periodinane (28 mg, 0.065 mmol, 1.3 equiv) was then added in one portion, and the vial was flushed with N_2. After stirring at room temperature for 17 h, the reaction was diluted with a 1:1 mixture of saturated aqueous solutions of $NaHCO_3$ and $Na_2S_2O_3$ (2 mL). The resulting biphasic mixture was vigorously stirred until both layers were no longer cloudy. The resulting mixture was transferred to a test tube with EtOAc (2 mL). After extracting with EtOAc (4 x 2 mL), the organic layers were combined, dried over $MgSO_4$, and evaporated under reduced pressure. The resulting residue was purified by flash chromatography (4:1 hexanes:EtOAc) to afford aminoketone **12** (12.3 mg, 66% yield, over two steps) as an amorphous solid. Aminoketone **12**: R_f 0.42 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34 (d, J = 8.4, 1H), 7.27 (dd, J = 8.4, 7.6, 1H), 6.75 (d, J = 7.6, 1H), 6.18 (d, J = 4.2, 1H), 5.44 (dd, J = 17.3, 10.9, 1H), 5.22 (d, J = 10.9, 1H), 5.17 (d, J = 17.3, 1H), 3.82 (s, 1H), 3.18 (s, 3H), 3.15 (d, J = 4.2, 1H), 1.71 (br. s, 2H), 1.69 (s, 3H), 1.31 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4, 174.7, 144.1, 140.6, 138.9, 135.2, 128.2, 124.2, 123.8, 123.7, 116.2, 107.7, 71.8, 62.8, 56.7, 53.8, 40.0, 26.4, 25.9, 21.6, 20.6; IR (film): 2973, 1709, 1698, 1609, 1583, 1457 cm^{-1}; HRMS-ESI (m/z) $[M + H]^+$ calcd for $C_{21}H_{24}N_2O_2Cl$, 371.1526; found 371.1516; $[\alpha]^{23.8}_D$ –70.2° (c = 1.000, $CHCl_3$).



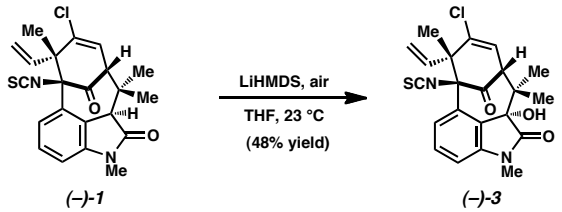
(–)-*N*-Methylwelwitindolinone Isonitrile (2). A 1-dram vial was charged with 96% formic acid (0.100 mL) and acetic anhydride (0.100 mL), and then stirred at 60 °C for 1 h. The reaction vessel was cooled to room temperature and 68 μL of the 96% formic acid/acetic anhydride mixture was added to a solution of aminoketone **12** (7.5 mg, 0.0203 mmol, 1 equiv) in THF (450 μL) at 0 °C. The reaction was stirred at 0 °C for 5 minutes, and then warmed to room temperature. After stirring for an additional 30 minutes, the reaction mixture was then transferred to a test tube containing EtOAc (1 mL) and a saturated solution of aqueous $NaHCO_3$ (1 mL). The resulting biphasic mixture was extracted with EtOAc (3 x 3 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 the crude residue was added THF (1 mL) and benzene (1 mL), followed by the addition of Burgess reagent (12 mg, 0.0406 mmol, 2 equiv). The vial was flushed with N_2 and allowed to stir at room temperature for 1 h. The reaction was then filtered through a plug of silica gel (EtOAc eluent, 20 mL). The filtrate was evaporated under reduced pressure, and the resulting residue was purified by prep TLC (1:1 hexanes:EtOAc) to afford (–)-**2** (7.8 mg, quant. yield) as an amorphous solid. (–)-*N*-Methylwelwitindolinone C isonitrile (**2**). Spectral data for synthetic **2** was consistent with literature reports[4]: R_f 0.60 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 (ddd, *J* = 8.5, 7.7, 0.9, 1H), 7.27 (dd, *J* = 8.5, 0.9, 1H), 6.81 (dd, *J* = 7.7, 0.9, 1H), 6.18 (d, *J* = 4.4, 1H), 5.37–5.30 (m, 3H), 3.73 (s, 1H), 3.23 (d, *J* = 4.4, 1H), 3.18 (s, 3H), 1.68 (s, 3H), 1.53 (s, 3H), 0.79 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 173.9, 163.5, 144.5, 138.3, 136.2, 128.7, 127.7, 124.6, 123.3, 122.8, 118.3, 108.8, 81.9, 61.6, 55.6, 53.2, 40.7, 26.4, 25.6, 22.6, 21.3; IR (film): 2969, 2141, 1735, 1711, 1609, 1587, 1460, 1341 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{22}H_{21}N_2O_2ClNa$, 403.1189; found 403.1178; $[\alpha]^{24.2}_D$ –90.4° (*c* = 0.25, CH_2Cl_2).[5]



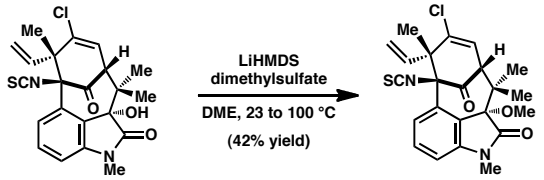
(–)-3-Hydroxy-*N*-Methylwelwitindolinone C Isonitrile (4). To a solution of (–)-**2** (7.8 mg, 0.0205 mmol, 1.0 equiv) in THF (1.1 mL) was added NaH (60% dispersion in mineral oil, 4.0 mg, 0.103 mmol, 5 equiv) in one portion. The vial was sealed under ambient atomospheric

conditions and allowed to star at room temperature. After 2.5 h, the reaction was filtered through a plug of silica gel (EtOAc eluent, 20 mL). The filtrate was evaporated under reduced pressure and the resulting residue was purified by prep TLC (1:1 hexanes:EtOAc) to afford (–)-**2** (3.8 mg, 47% yield) as an amorphous solid. (–)-3-Hydroxy-*N*-methylwelwitindolinone C isonitrile (**4**). Spectral data for synthetic **4** was consistent with literature reports[4]: R_f 0.46 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.44 (dd, *J* = 8.2, 7.6, 1H), 7.33 (dd, *J* = 8.2, 0.9, 1H), 6.89 (dd, *J* = 7.6, 0.9, 1H), 6.40 (d, *J* = 4.6, 1H), 5.50 (dd, *J* = 17.2, 10.4, 1H), 5.40 (dd, *J* = 17.2, 0.8 1H), 5.37 (dd, *J* = 10.4, 0.8 1H), 3.18 (d, *J* = 4.6, 1H), 3.15 (s, 3H), 1.71 (s, 3H), 1.56 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (125 MHz, C_6D_6): δ 192.6, 173.2, 166.5, 145.1, 137.4, 132.8, 130.3, 128.8, 126.4, 126.00, 125.98, 117.8, 109.2, 82.3, 80.2, 60.6, 55.4, 42.3, 25.6, 22.6, 22.0, 20.8; IR (film): 3395, 2973, 2922, 2142, 1723, 1610, 1587, 1459 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{22}H_{21}N_2O_3ClNa$, 419.1138; found 419.1137; $[\alpha]^{23.1}_D$ –90.0° (*c* = 0.4, CH_2Cl_2).[5]



(–)-1 *(–)-3*

(–)-3-Hydroxy-*N*-Methylwelwitindolinone C Isothiocyanate (3). A 1-dram vial was charged with (–)-**1** (2.4 mg, 0.0058 mmol, 1.0 equiv) and then sealed under ambient atmospheric conditions. THF (300 μL) was then added, followed by the dropwise addition of 100 μL of an 11 mg/mL solution of LiHMDS in THF. The reaction was stirred at room temperature for 6 h, and then another 50 μL of the LiHMDS solution was added. After an additional 90 minutes, another 50 μL of the LiHMDS solution was added and the reaction was stirred for an additional 14 h. The reaction was then filtered through a plug of silica gel (EtOAc eluent, 20 mL). The filtrate was evaporated under reduced pressure and the resulting residue was purified by prep TLC (1:1 hexanes:EtOAc) to afford (–)-**3** (1.2 mg, 48% yield) as an amorphous solid. (–)-3-hydroxy-*N*-methylwelwitindolinone C isothiocyanate (**3**)[6]: R_f 0.46 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.41 (dd, *J* = 8.4, 7.6, 1H), 7.25 (dd, *J* = 8.4, 0.9, 1H), 6.87 (dd, *J* = 7.6, 0.9, 1H), 6.40 (d, *J* = 4.5, 1H), 5.48 (dd, *J* = 17.5, 10.2, 1H), 5.33–5.29 (m, 2H), 3.21 (d, *J* = 4.5, 1H), 3.14 (s, 3H), 1.71 (s, 3H), 1.50 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (125 MHz, CD_2Cl_2): δ 196.3, 173.7, 145.5, 140.7, 138.0, 133.8, 130.7, 130.6, 126.2, 126.1, 125.9, 117.9, 109.7, 84.5, 80.6, 61.1, 57.1, 42.9, 26.6, 22.9, 21.7, 21.2; IR (film): 3399, 2966, 2929, 2044, 1721, 1610, 1585, 1457 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{22}H_{21}N_2O_3SClNa$, 451.0859; found 451.0860; $[\alpha]^{25.2}_D$ –206.0° (*c* = 1.00, CH_2Cl_2).[5]



(–)-3 *14*

Methyl Ether 14. To a stirred solution of (–)-**3** (2.3 mg, 0.0054 mmol, 1.0 equiv) in DME (200 μL) was added 100 μL of a 10 mg/mL solution of LiHMDS in DME. The reaction was stirred at room temperature for 1 h. Dimethylsulfate (10.2 μL, 0.107 mmol, 20 equiv) was added and the reaction was heated to 100 °C. After 24 h, the reaction was cooled to room temperature and filtered through a plug of silica gel (EtOAc eluent, 20 mL). The filtrate was evaporated under reduced pressure and the resulting residue was purified by prep TLC (2:1:1 hexanes:CH_2Cl_2:Et_2O) to afford **14** (1.0 mg, 42% yield) as an amorphous solid. Methyl Ether **14**: R_f 0.58 (1:1 hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (dd, *J* = 8.5, 7.9, 1H), 7.32 (dd, *J* = 8.5, 0.9, 1H), 6.82 (dd, *J* = 7.9, 0.9, 1H), 6.31 (d, *J* = 4.4, 1H), 5.46 (dd, *J* = 17.5, 10.1, 1H), 5.34 (d, *J* = 17.5, 1H), 5.34 (d, *J* = 10.1, 1H), 3.19 (s, 3H), 3.16 (d, *J* = 4.4, 1H), 1.70 (s, 3H), 1.49 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 196.3, 172.0, 145.7, 140.7, 137.9, 132.3, 131.1, 130.7, 126.7, 126.1, 122.1, 117.7, 108.6, 85.2, 84.4, 61.1, 56.6, 51.3, 43.7, 26.1, 22.9, 21.62, 21.55; IR (film): 2916, 2050, 1725, 1607, 1583, 1455 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{23}H_{23}N_2O_3SClNa$, 465.1016; found 465.1028; $[\alpha]^{25.2}_D$ –118.7° (*c* = 0.15, CH_2Cl_2).

Computational Data.

Computed NMR chemical shift data

Table SI–1. Comparison of Experimental and Computed NMR Chemical Shifts for Structure **4**.

^{13}C NMR Chemical Shifts (ppm)				^{1}H NMR Chemical Shifts (ppm)			
Nucleus #[a]	Expt.[b]	Computed[c] Original	Computed[c] C3 epimer	Nucleus #[a]	Expt.[b]	Computed[c] Original	Computed[c] C3 epimer
N CH_3	26.60	24.75	24.68	*N* CH_3	3.15	3.02	3.00
2	173.60	171.48	172.62	OH	2.65	2.20	1.96
3	80.60	80.47	80.08	5	7.33	7.20	7.37
4	128.40	129.46	132.98	6	7.44	7.39	7.36
5	126.20	124.05	123.51	7	6.89	6.82	6.78
6	130.80	128.65	128.49	14	6.40	6.40	5.83
7	110.00	108.52	108.75	15	3.18	3.22	3.09
8	145.50	144.64	143.77	17	1.71	1.66	0.94
9	126.40	126.15	127.18	18	0.81	0.77	1.60
10	193.60	196.02	198.73	19	1.55	1.55	1.36
11	82.00	82.13	77.56	20	5.49	5.48	6.25
12	55.50	60.92	60.84	21 *E*	5.34	5.36	5.65
13	133.30	140.06	142.36	21 *Z*	5.40	5.40	5.58
14	126.00	128.32	127.63				
15	61.00	61.60	59.56				
16	42.80	48.39	47.55				
17	22.80	19.42	21.41				
18	21.20	21.49	21.51				
19	22.10	20.56	20.48				
20	137.10	139.50	140.61				
21	118.40	118.25	120.00				
23	164.30	168.17	166.42				
	CMAD[d]	**2.13**	**2.69**		**CMAD[d]**	**0.08**	**0.36**

[a]See page S11). [b]Data taken from isolation report; see reference 4. [c]Conformationally averaged values – see page S11). Largest outliers are indicated in red. Note that higher than average errors are expected for the carbon atom bearing a chlorine atom (C13) – due to heavy-atom effects, and for the hydroxyl proton – due to concentration-dependent hydrogen bonding.[7] [d]CMAD = corrected mean absolute deviation and is computed as $\frac{1}{n}\sum_{i}^{n}|\delta_{comp}-\delta_{exp}|$ where δ_{comp} refers to the scaled computed chemical shifts.

Note: For the C3 epimer structure, a modest improvement in the match to experimental data is found if the C17 and C18 methyl protons are switched in their experimental assignments

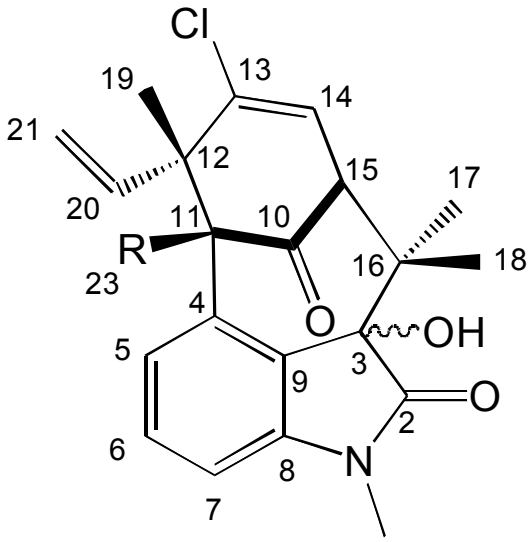
(CMAD = 0.26 ppm). This amount of improvement is not sufficient to change our overall conclusion.

Table SI-2. Comparison of Experimental and Computed NMR Chemical Shifts for Structure **3**.

^{13}C NMR Chemical Shifts (ppm)				^{1}H NMR Chemical Shifts (ppm)			
Nucleus #[a]	Expt.[b]	Computed[c] Original	Computed[c] C3 epimer	Nucleus #[a]	Expt.[b]	Computed[c] Original	Computed[c] C3 epimer
N CH_3	26.60	24.89	24.57	*N* CH_3	3.14	3.02	3.01
2	173.70	172.44	173.12	OH	*not obsv.*		
3	80.60	80.37	79.50	5	7.25	7.14	7.38
4	130.60	132.41	132.88	6	7.41	7.30	7.26
5	126.20	124.24	123.93	7	6.87	6.83	6.73
6	130.70	128.78	127.83	14	6.40	6.34	5.87
7	109.70	108.20	108.22	15	3.21	3.20	3.29
8	145.50	144.63	144.49	17	1.71	1.62	0.96
9	125.90	125.36	126.44	18	0.81	0.79	1.61
10	196.30	198.52	201.21	19	1.50	1.49	1.38
11	84.50	86.70	82.20	20	5.48	5.42	6.30
12	57.10	61.87	60.36	21 *E*	5.33–5.29	5.25	5.56
13	133.80	140.56	143.45	21 *Z*	5.33–5.29	5.30	5.42
14	126.10	126.98	127.83				
15	61.10	60.63	59.72				
16	42.90	48.15	47.78				
17	22.90	19.33	21.88				
18	21.70	21.38	21.69				
19	21.70	19.95	20.29				
20	138.00	141.57	142.17				
21	117.90	116.31	118.28				
23	140.60	144.85	146.31				
	CMAD[d]	**2.25**	**2.50**		**CMAD[d]**	**0.06**	**0.33**

[a] See page S11). [b] Data obtained from Prof Philip Williams (University of Hawaii); see ref 6. [c]Lowest energy conformation – see page S11). Largest outliers are indicated in red. Note that higher than average errors are expected for the carbon atom bearing a chlorine atom (C13) – due to heavy-atom effects, and for the hydroxyl proton – due to concentration-dependent hydrogen bonding.[7] [d] CMAD = corrected mean absolute deviation and is computed as $\frac{1}{n}\sum_{i}^{n}|\delta_{comp} - \delta_{exp}|$ where δ_{comp} refers to the scaled computed chemical shifts. Where the experimental value is a range, the mean value is used.

Atom #'s used in Tables SI-1 & SI-2, taken from reference 4.



Methods

General

Calculations (geometry optimization, frequency, and NMR chemical shift) were performed on C3-hydroxyl-N-methylwelwitindolinone C isonitrile (structure **4**) and its C3 epimer, as well as C3-hydroxyl-N-methylwelwitindolinone C isothiocyanate (structure **3**) and its C3 epimer.

Calculations were performed with GAUSSIAN09.[8] Geometries were optimized in the gas-phase using the B3LYP/6-31+G(d,p)[9] level of theory. Frequency calculations (at 298.15 K) at the same level of theory were used to confirm the nature of all stationary points as minima and also provided values for computed free energies. NMR single point calculations (GIAO)[10] were performed on these geometries at the mPW1PW91/6-311+G(d,p)[11] level of theory in an implicit chloroform solvent continuum (SMD[12] method).

Conformational Analysis

For structure **4** and its C3 epimer, nine candidate conformers (three conformations of the vinyl group and three conformations of the hydroxyl group) for each epimer were subjected to geometry optimization. This resulted in four unique conformers for structure **4** and six unique conformers of its C3 epimer. For both epimers, Boltzmann-weighted averaging of the computed chemical shifts based on the relative computed free energies at 298.15 K of each conformer was performed, using the equation below to determine relative populations.

$$\frac{P_i}{P_j} = e^{\frac{-(E_i - E_j)}{RT}}$$

P_i = population of conformer *i* relative to lowest energy conformer *j*
E_i, E_j = computed free energies (in J/mol)
R = molar gas constant (8.314510 J mol^{-1} K^{-1})
T = 298.15 K

The relative populations were then converted to Boltzmann-weighting factors by means of a set of linear equations.

Although only one conformer of the ring system seemed to be likely, both epimers of structure **4** were subjected to a conformational search (in Spartan'10).[13] As expected, only a single conformation of the ring system was found in each case.

For the isothiocyanate structure **3**, the major contributing (lowest energy) conformer of isonitrile structure **4** was converted into the corresponding isothiocyanate, and subjected to geometry optimization, followed by frequency and NMR chemical shift calculations (for both epimers).

Empirical scaling of computed NMR chemical shifts

Computed chemical shifts are commonly scaled empirically in order to remove systematic error that results from a variety of sources. The scaling factors themselves are generally determined by comparison of computed NMR data with known experimental chemical shifts for large databases of molecules. These factors (slope and intercept from a best fit line) are specific for each level of theory used computationally. We have generated numerous such scaling factors for ^{1}H and ^{13}C chemical shifts utilizing a database originally compiled by Rablen and co-workers and have made them available on our web site at http://cheshirenmr.info.

One of our preferred methods for obtaining high quality computed chemical shifts at reasonable costs is to use mPW1PW91/6-311+G(2d,p) NMR calculations (with the SMD chloroform continuum model) on B3LYP/6-31+G(d,p) geometries. After scaling, this method produces average errors (CMAD's) of 0.11-0.15 ppm for ^{1}H and 1.8-2.5 ppm for ^{13}C on diverse sets of small organic molecules. Details and numerous references on linear regression methods applied to computed chemical shifts can be found in our review paper.[7]

The specific scaling factors used in this study are given below and are applied to the computed NMR isotropic shielding constants by way of the equation shown.

	^{1}H	^{13}C
Slope	-1.0936	-1.0533
Intercept	31.8018	186.5242

$$\delta = \frac{b - \sigma}{-m}$$

δ = computed chemical shift relative to TMS
σ = computed isotropic shielding constant
m = slope, b = intercept

DP4 Probability Analysis

For further support of our assignment to the C3(*S*) diastereomer for isonitrile structure **4**, we utilized the DP4 probability analysis of Smith and Goodman.[14] When both possible epimers were compared to the experimental data, the analysis suggested a 67.5% probability of C3(*S*) being correct based on the ^{13}C data, a 100% probability based on the ^{1}H data, and a 100% probability based on both sets of data.

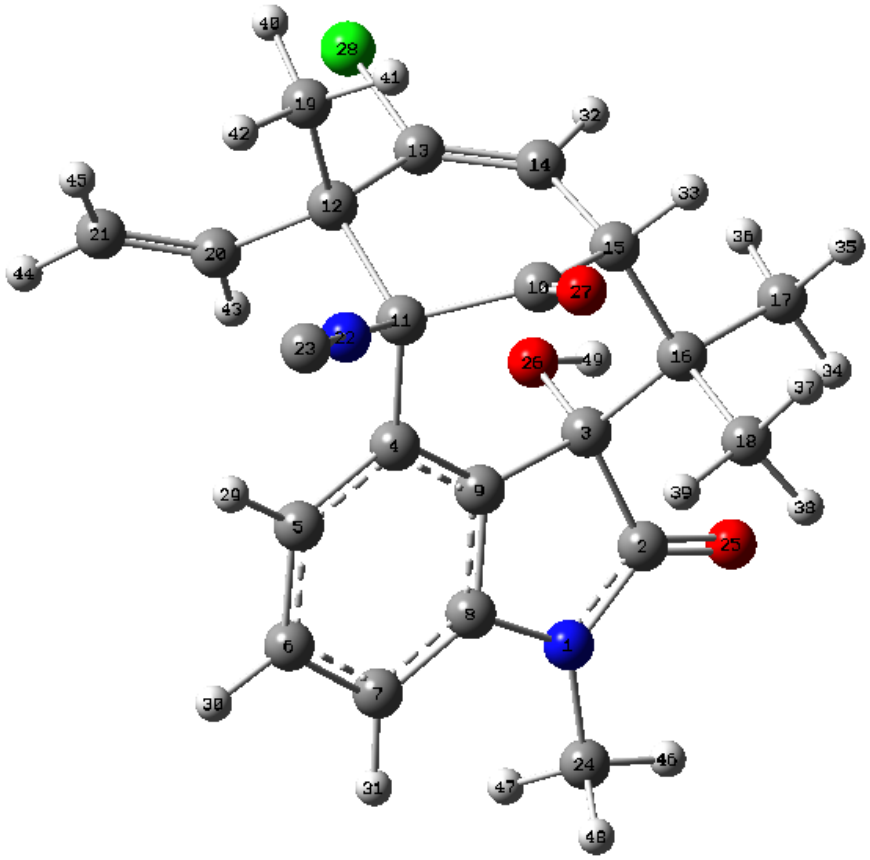
Energies, coordinates, and NMR isotropic shielding constants

Structure 4, conformer 1

Sum of electronic and thermal free energies = -1645.99519 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.380511	0.751109	-0.617354
2	6	-3.108729	-0.579788	-0.477610
3	6	-1.576958	-0.739243	-0.229973
4	6	0.100593	1.342374	0.224316
5	6	0.166355	2.747156	0.083118
6	6	-0.926998	3.509133	-0.308650
7	6	-2.164939	2.908981	-0.547955
8	6	-2.224961	1.528726	-0.433403
9	6	-1.115549	0.717867	-0.099250
10	6	0.911574	-0.488942	1.794213
11	6	1.361546	0.651230	0.832091
12	6	2.441035	0.080430	-0.232322
13	6	1.991257	-1.302366	-0.688458
14	6	1.114541	-2.103441	-0.085843
15	6	0.340831	-1.738622	1.154370
16	6	-1.226129	-1.694004	0.949197
17	6	-1.694972	-3.141817	0.675179
18	6	-1.918249	-1.203489	2.239070
19	6	3.813867	-0.103077	0.460532
20	6	2.502368	1.034876	-1.415368
21	6	3.497088	1.873522	-1.710270
22	7	2.033204	1.616907	1.637432
23	6	2.586836	2.357422	2.363512
24	6	-4.681175	1.269742	-0.999762
25	8	-3.902935	-1.495419	-0.652588
26	8	-1.090993	-1.225047	-1.496027
27	8	1.040237	-0.387499	2.994192
28	17	2.810643	-1.908346	-2.135113
29	1	1.094865	3.254364	0.304017
30	1	-0.819288	4.585337	-0.400957
31	1	-3.037307	3.497044	-0.810138
32	1	0.936332	-3.087914	-0.503238
33	1	0.500607	-2.520825	1.907413
34	1	-2.776929	-3.181623	0.543748
35	1	-1.426114	-3.773261	1.528812
36	1	-1.231478	-3.576643	-0.214547
37	1	-1.578758	-1.786674	3.099890
38	1	-3.002030	-1.330487	2.154846
39	1	-1.711653	-0.151953	2.456298
40	1	4.498705	-0.599373	-0.230537
41	1	3.714525	-0.727653	1.352535
42	1	4.252712	0.847527	0.762572
43	1	1.626966	1.010046	-2.060573
44	1	3.429437	2.518753	-2.581112
45	1	4.403556	1.951460	-1.119072
46	1	-5.361472	0.421841	-1.084425
47	1	-4.622074	1.785227	-1.964582
48	1	-5.056951	1.965669	-0.242570
49	1	-1.689279	-1.931402	-1.787016

2	C	Isotropic =	5.3824	29	H	Isotropic =	23.9179
3	C	Isotropic =	101.9705	30	H	Isotropic =	23.7070
4	C	Isotropic =	49.7929	31	H	Isotropic =	24.3199
5	C	Isotropic =	55.7165	32	H	Isotropic =	24.8577
6	C	Isotropic =	51.0288	33	H	Isotropic =	28.2880
7	C	Isotropic =	72.1794	34	H	Isotropic =	29.1701
8	C	Isotropic =	34.1558	35	H	Isotropic =	30.8685
9	C	Isotropic =	53.9450	36	H	Isotropic =	30.0279
10	C	Isotropic =	-20.0573	37	H	Isotropic =	30.5775
11	C	Isotropic =	100.0258	38	H	Isotropic =	30.8627
12	C	Isotropic =	122.5598	39	H	Isotropic =	31.4457
13	C	Isotropic =	39.6653	40	H	Isotropic =	30.0039
14	C	Isotropic =	51.8523	41	H	Isotropic =	30.6930
15	C	Isotropic =	121.7132	42	H	Isotropic =	29.6167
16	C	Isotropic =	135.6107	43	H	Isotropic =	25.8003
17	C	Isotropic =	166.2994	44	H	Isotropic =	25.9664
18	C	Isotropic =	164.0007	45	H	Isotropic =	25.8850
19	C	Isotropic =	165.0069	46	H	Isotropic =	27.5588
20	C	Isotropic =	39.3145	47	H	Isotropic =	28.9537
21	C	Isotropic =	62.5941	48	H	Isotropic =	28.9597
23	C	Isotropic =	9.4942	49	H	Isotropic =	29.4283
24	C	Isotropic =	160.3779				

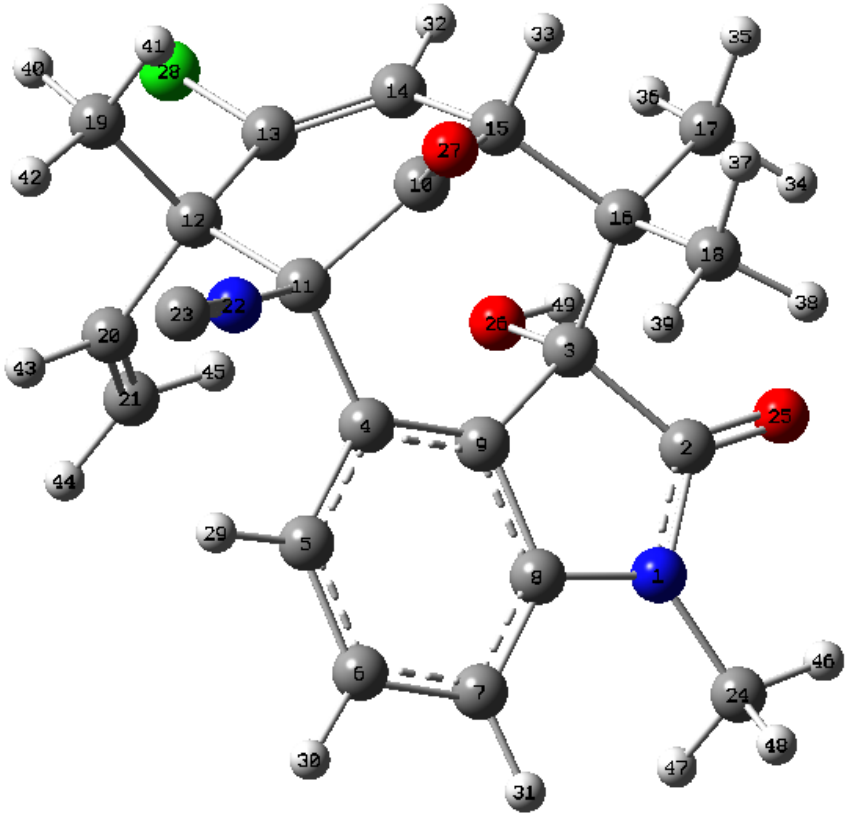


Structure 4, conformer 2

Sum of electronic and thermal free energies = -1645.992378 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.389910	0.411641	-0.584188
2	6	-2.965521	-0.872239	-0.398840
3	6	-1.427194	-0.846062	-0.141642
4	6	0.012876	1.426251	0.172806
5	6	-0.088399	2.823398	-0.011922
6	6	-1.268385	3.441804	-0.404375
7	6	-2.431329	2.695800	-0.605917
8	6	-2.329062	1.322773	-0.445527
9	6	-1.129801	0.657371	-0.101630
10	6	1.030557	-0.187507	1.832779
11	6	1.356655	0.894958	0.758969
12	6	2.436090	0.330558	-0.322514
13	6	2.169786	-1.147259	-0.573941
14	6	1.396904	-1.962253	0.142545
15	6	0.567402	-1.538793	1.325806
16	6	-0.991904	-1.665159	1.111119
17	6	-1.311230	-3.169442	0.949863
18	6	-1.738553	-1.147243	2.358981
19	6	3.869594	0.421973	0.274214
20	6	2.371154	1.204721	-1.566937
21	6	1.672684	0.966313	-2.677686
22	7	1.980856	1.972917	1.454311
23	6	2.499432	2.806413	2.101395
24	6	-4.742986	0.763434	-0.974618
25	8	-3.645808	-1.880969	-0.539796
26	8	-0.879667	-1.372703	-1.364425
27	8	1.169300	0.037695	3.014619
28	17	3.113201	-1.882915	-1.883336
29	1	0.776650	3.444181	0.173272
30	1	-1.286911	4.519781	-0.530763
31	1	-3.369095	3.170462	-0.871934
32	1	1.366618	-3.013474	-0.122288
33	1	0.786676	-2.220689	2.157072
34	1	-2.383033	-3.328798	0.826617
35	1	-0.982069	-3.705397	1.846601
36	1	-0.803240	-3.618029	0.092046
37	1	-1.359121	-1.638557	3.259701
38	1	-2.806525	-1.374337	2.280869
39	1	-1.625833	-0.069313	2.502672
40	1	4.565784	-0.076892	-0.403155
41	1	3.926828	-0.068257	1.250262
42	1	4.188723	1.458815	0.387579
43	1	2.951041	2.123283	-1.491520
44	1	1.689639	1.674541	-3.500899
45	1	1.066322	0.074939	-2.805571
46	1	-5.322154	-0.159042	-1.024872
47	1	-4.746801	1.248298	-1.956963
48	1	-5.193416	1.438110	-0.239220
49	1	-1.404343	-2.153019	-1.604425

2	C	Isotropic =	4.9313	29	H	Isotropic =	23.8898
3	C	Isotropic =	101.8846	30	H	Isotropic =	23.7819
4	C	Isotropic =	47.7504	31	H	Isotropic =	24.3539
5	C	Isotropic =	56.1850	32	H	Isotropic =	24.8360
6	C	Isotropic =	50.5833	33	H	Isotropic =	28.3687
7	C	Isotropic =	72.3931	34	H	Isotropic =	29.2774
8	C	Isotropic =	34.0780	35	H	Isotropic =	30.8967
9	C	Isotropic =	54.5485	36	H	Isotropic =	29.9107
10	C	Isotropic =	-19.4205	37	H	Isotropic =	30.6386
11	C	Isotropic =	98.4760	38	H	Isotropic =	30.9444
12	C	Isotropic =	120.3660	39	H	Isotropic =	31.4424
13	C	Isotropic =	39.7763	40	H	Isotropic =	29.7032
14	C	Isotropic =	48.9210	41	H	Isotropic =	30.6575
15	C	Isotropic =	121.5678	42	H	Isotropic =	29.9034
16	C	Isotropic =	135.6714	43	H	Isotropic =	25.7159
17	C	Isotropic =	166.5375	44	H	Isotropic =	26.1295
18	C	Isotropic =	164.1755	45	H	Isotropic =	26.4239
19	C	Isotropic =	158.0736	46	H	Isotropic =	27.5589
20	C	Isotropic =	40.9847	47	H	Isotropic =	28.9945
21	C	Isotropic =	54.8881	48	H	Isotropic =	28.9520
23	C	Isotropic =	7.6178	49	H	Isotropic =	29.4036
24	C	Isotropic =	160.4337				

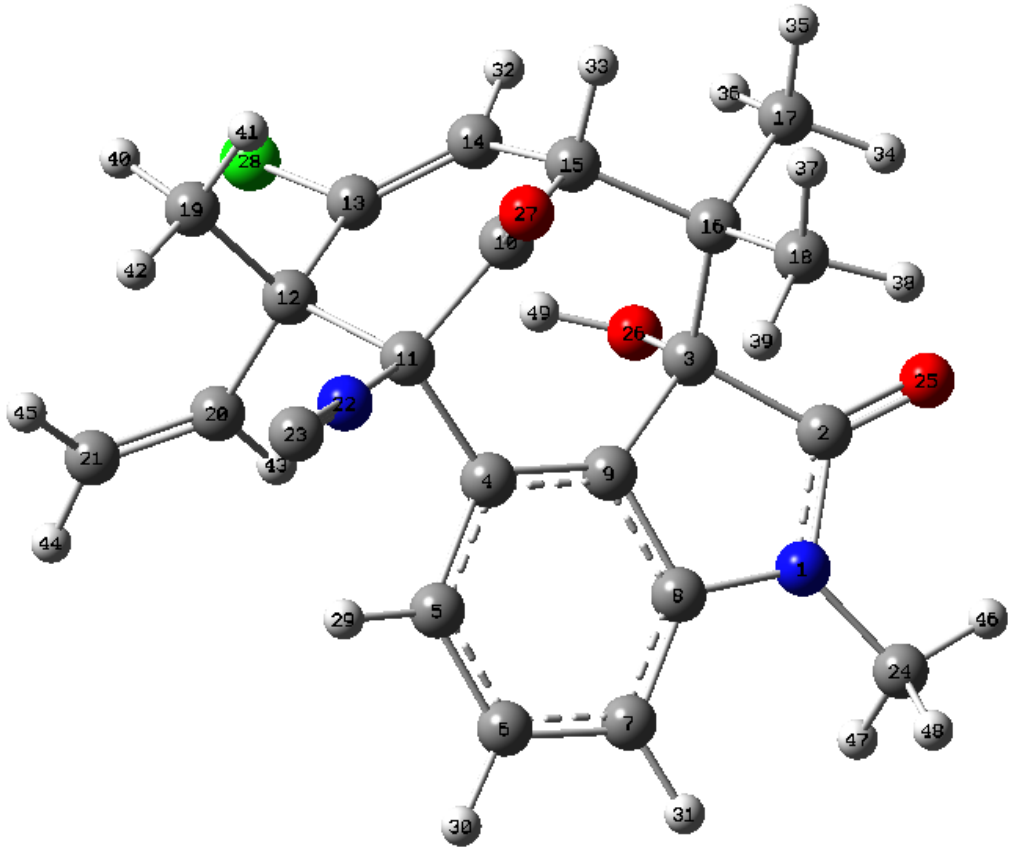


Structure 4, conformer 3

Sum of electronic and thermal free energies = -1645.993819 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.368932	0.825648	-0.629677
2	6	-3.157544	-0.516396	-0.430269
3	6	-1.615809	-0.722439	-0.255060
4	6	0.110041	1.315147	0.269538
5	6	0.216937	2.720030	0.145044
6	6	-0.846054	3.511664	-0.269219
7	6	-2.092278	2.944669	-0.546407
8	6	-2.196843	1.566140	-0.436883
9	6	-1.116419	0.723442	-0.077750
10	6	0.893998	-0.610775	1.760484
11	6	1.349943	0.587841	0.876648
12	6	2.449760	0.069134	-0.194197
13	6	1.964466	-1.253081	-0.780820
14	6	1.076899	-2.090895	-0.235152
15	6	0.318438	-1.813115	1.038195
16	6	-1.251673	-1.752414	0.852265
17	6	-1.730562	-3.171703	0.473467
18	6	-1.918724	-1.347152	2.184506
19	6	3.784465	-0.234372	0.531732
20	6	2.603352	1.114144	-1.288207
21	6	3.654511	1.911392	-1.484484
22	7	2.010776	1.505779	1.742841
23	6	2.556233	2.206743	2.513309
24	6	-4.666021	1.390904	-0.949739
25	8	-4.017924	-1.377950	-0.464930
26	8	-1.278846	-1.185915	-1.578005
27	8	1.025223	-0.591597	2.963826
28	17	2.785009	-1.759528	-2.265224
29	1	1.151272	3.201081	0.398496
30	1	-0.709502	4.585596	-0.349512
31	1	-2.941161	3.557803	-0.827679
32	1	0.888501	-3.043511	-0.719605
33	1	0.487428	-2.651677	1.725197
34	1	-2.816004	-3.189593	0.373942
35	1	-1.437135	-3.875270	1.260605
36	1	-1.313172	-3.510827	-0.476299
37	1	-1.562267	-1.983655	2.999523
38	1	-3.002208	-1.472190	2.108315
39	1	-1.710453	-0.310705	2.465781
40	1	4.481365	-0.696440	-0.171052
41	1	3.625627	-0.926805	1.362777
42	1	4.242145	0.670104	0.931887
43	1	1.750170	1.203559	-1.957080
44	1	3.652028	2.630787	-2.297749
45	1	4.544204	1.883991	-0.864411
46	1	-5.377827	0.566846	-1.006027
47	1	-4.632832	1.908513	-1.914460
48	1	-4.985630	2.095613	-0.174126
49	1	-0.322909	-1.100994	-1.701002

2	C	Isotropic =	8.2332	29	H	Isotropic =	23.9698
3	C	Isotropic =	100.9151	30	H	Isotropic =	23.7605
4	C	Isotropic =	52.2624	31	H	Isotropic =	24.4222
5	C	Isotropic =	56.4224	32	H	Isotropic =	24.6004
6	C	Isotropic =	51.0441	33	H	Isotropic =	28.2420
7	C	Isotropic =	72.3600	34	H	Isotropic =	28.9353
8	C	Isotropic =	34.2295	35	H	Isotropic =	30.8612
9	C	Isotropic =	52.2202	36	H	Isotropic =	29.6794
10	C	Isotropic =	-19.6241	37	H	Isotropic =	30.4989
11	C	Isotropic =	100.3225	38	H	Isotropic =	30.7159
12	C	Isotropic =	122.0071	39	H	Isotropic =	31.5247
13	C	Isotropic =	36.0314	40	H	Isotropic =	30.1151
14	C	Isotropic =	50.0042	41	H	Isotropic =	30.6694
15	C	Isotropic =	121.3573	42	H	Isotropic =	29.6456
16	C	Isotropic =	135.2910	43	H	Isotropic =	25.8986
17	C	Isotropic =	165.0367	44	H	Isotropic =	25.7683
18	C	Isotropic =	163.3592	45	H	Isotropic =	25.7723
19	C	Isotropic =	166.0095	46	H	Isotropic =	27.5553
20	C	Isotropic =	40.3261	47	H	Isotropic =	28.9550
21	C	Isotropic =	61.0648	48	H	Isotropic =	29.0701
23	C	Isotropic =	9.3674	49	H	Isotropic =	29.2466
24	C	Isotropic =	160.7578				

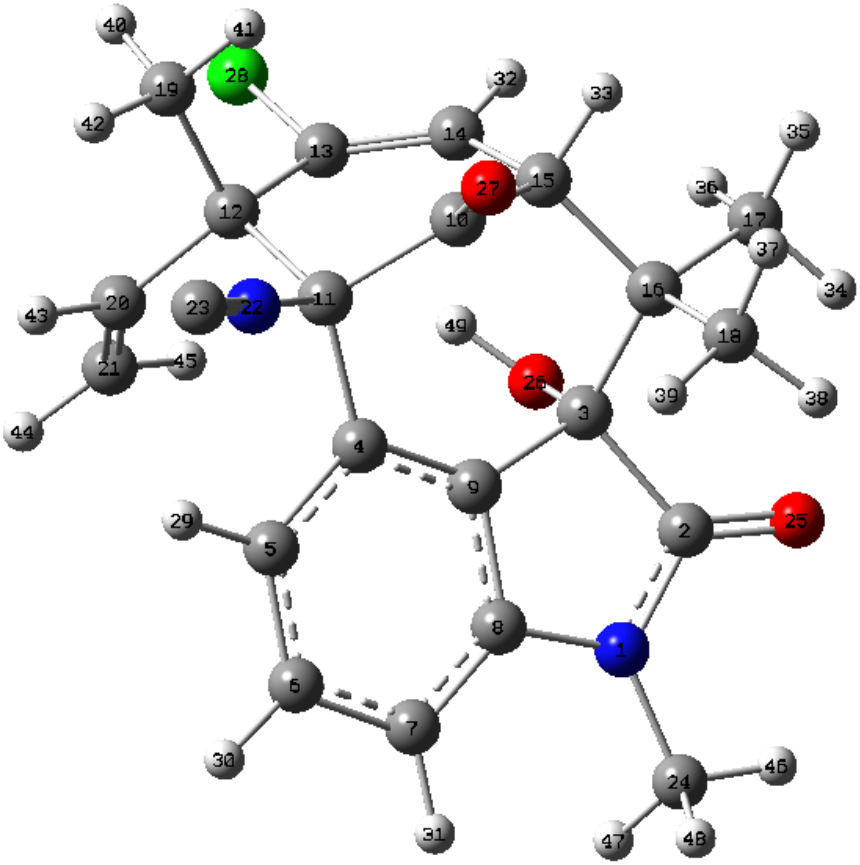


Structure 4, conformer 4

Sum of electronic and thermal free energies = -1645.990807 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.383740	0.566778	-0.610315
2	6	-3.053866	-0.749632	-0.404647
3	6	-1.502026	-0.816780	-0.214802
4	6	0.041698	1.369611	0.272369
5	6	0.019676	2.777398	0.141515
6	6	-1.111888	3.469606	-0.267287
7	6	-2.302822	2.791942	-0.538165
8	6	-2.281883	1.410361	-0.422782
9	6	-1.130076	0.669650	-0.061469
10	6	0.985067	-0.440706	1.799880
11	6	1.344407	0.756735	0.872524
12	6	2.471665	0.293020	-0.205474
13	6	2.119896	-1.097394	-0.718871
14	6	1.302069	-1.977261	-0.128904
15	6	0.502608	-1.706429	1.120501
16	6	-1.064392	-1.781722	0.924539
17	6	-1.419350	-3.245934	0.582480
18	6	-1.770669	-1.396033	2.241871
19	6	3.848229	0.151181	0.507979
20	6	2.589793	1.375056	-1.266159
21	6	2.063455	1.389910	-2.491065
22	7	1.949458	1.746286	1.702698
23	6	2.450688	2.501879	2.451465
24	6	-4.725876	1.012629	-0.932908
25	8	-3.833050	-1.685569	-0.439157
26	8	-1.108252	-1.282675	-1.518983
27	8	1.118726	-0.370258	3.001027
28	17	3.064853	-1.681170	-2.100933
29	1	0.906429	3.344477	0.387287
30	1	-1.072432	4.551192	-0.351229
31	1	-3.204506	3.324865	-0.818126
32	1	1.228235	-2.977031	-0.544828
33	1	0.731191	-2.503770	1.838738
34	1	-2.499142	-3.360044	0.484947
35	1	-1.066253	-3.902404	1.385728
36	1	-0.973398	-3.569377	-0.359684
37	1	-1.371713	-1.981726	3.075163
38	1	-2.840486	-1.607855	2.163737
39	1	-1.645868	-0.339626	2.496583
40	1	4.568505	-0.279372	-0.191013
41	1	3.779438	-0.504005	1.381016
42	1	4.225967	1.121416	0.832729
43	1	3.160923	2.240385	-0.934546
44	1	2.211380	2.246374	-3.141755
45	1	1.480996	0.570431	-2.899529
46	1	-5.360630	0.127838	-0.989069
47	1	-4.738278	1.530177	-1.898183
48	1	-5.108798	1.686745	-0.158694
49	1	-0.151893	-1.164749	-1.608721

2	C	Isotropic =	8.2386	29	H	Isotropic =	23.9665
3	C	Isotropic =	100.8098	30	H	Isotropic =	23.7824
4	C	Isotropic =	50.6591	31	H	Isotropic =	24.4111
5	C	Isotropic =	55.8937	32	H	Isotropic =	24.4481
6	C	Isotropic =	51.4584	33	H	Isotropic =	28.2770
7	C	Isotropic =	72.3683	34	H	Isotropic =	28.9619
8	C	Isotropic =	34.4162	35	H	Isotropic =	30.8492
9	C	Isotropic =	52.3402	36	H	Isotropic =	29.6213
10	C	Isotropic =	-19.4601	37	H	Isotropic =	30.5030
11	C	Isotropic =	99.3093	38	H	Isotropic =	30.7587
12	C	Isotropic =	119.5882	39	H	Isotropic =	31.5047
13	C	Isotropic =	36.6404	40	H	Isotropic =	29.7433
14	C	Isotropic =	46.8267	41	H	Isotropic =	30.5658
15	C	Isotropic =	121.3607	42	H	Isotropic =	29.8611
16	C	Isotropic =	135.3283	43	H	Isotropic =	25.4891
17	C	Isotropic =	165.1747	44	H	Isotropic =	26.0009
18	C	Isotropic =	163.5269	45	H	Isotropic =	26.6548
19	C	Isotropic =	159.0525	46	H	Isotropic =	27.5470
20	C	Isotropic =	42.1685	47	H	Isotropic =	28.9975
21	C	Isotropic =	56.4925	48	H	Isotropic =	29.0701
23	C	Isotropic =	8.0406	49	H	Isotropic =	29.0085
24	C	Isotropic =	160.7613				

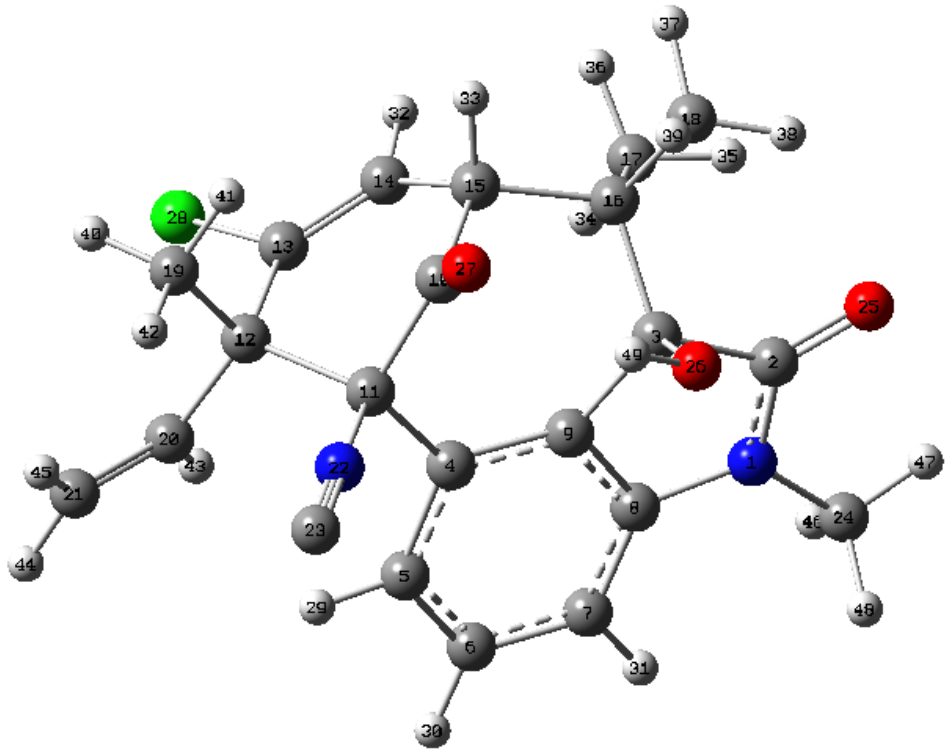


Structure 4, C3 epimer, conformer 1

Sum of electronic and thermal free energies = -1645.990153 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.288099	1.175668	-0.492135
2	6	-3.342285	-0.126122	-0.051393
3	6	-1.919172	-0.517146	0.467896
4	6	0.216887	1.116132	0.417828
5	6	0.574596	2.469902	0.244971
6	6	-0.328608	3.404260	-0.250180
7	6	-1.640543	3.038434	-0.570087
8	6	-2.007208	1.721176	-0.330716
9	6	-1.109312	0.751089	0.163076
10	6	0.677127	-1.267257	1.184698
11	6	1.289584	0.140867	0.966776
12	6	2.575046	-0.092689	0.000328
13	6	2.049458	-0.793428	-1.248061
14	6	0.990422	-1.602758	-1.280469
15	6	0.181495	-1.977392	-0.064002
16	6	-1.406789	-1.847788	-0.199949
17	6	-1.804230	-1.873909	-1.691333
18	6	-2.052391	-3.058982	0.505371
19	6	3.567136	-1.054323	0.705381
20	6	3.251073	1.220955	-0.359613
21	6	4.341876	1.730577	0.216459
22	7	1.754250	0.642592	2.213633
23	6	2.123883	1.053143	3.252223
24	6	-4.450401	1.908166	-0.958184
25	8	-4.337098	-0.828475	-0.041046
26	8	-2.162345	-0.633914	1.873909
27	8	0.582003	-1.758320	2.292595
28	17	3.012929	-0.604441	-2.717114
29	1	1.565825	2.796251	0.528119
30	1	-0.012579	4.435246	-0.376291
31	1	-2.349097	3.766197	-0.949937
32	1	0.745583	-2.103865	-2.209250
33	1	0.362853	-3.043980	0.123176
34	1	-1.454774	-0.989199	-2.233915
35	1	-2.890367	-1.935564	-1.783427
36	1	-1.388636	-2.758513	-2.183704
37	1	-1.801792	-3.972569	-0.046034
38	1	-3.138164	-2.948786	0.529867
39	1	-1.696158	-3.173178	1.531216
40	1	4.421744	-1.229297	0.047779
41	1	3.103579	-2.019656	0.922715
42	1	3.929574	-0.637874	1.646851
43	1	2.788680	1.770696	-1.173443
44	1	4.746011	2.680106	-0.121828
45	1	4.868284	1.245979	1.031658
46	1	-4.286205	2.289523	-1.971632
47	1	-5.294088	1.217278	-0.961382
48	1	-4.673133	2.747444	-0.289889
49	1	-1.356329	-0.880342	2.350000

2	C	Isotropic =	8.1779	29	H	Isotropic =	23.7702
3	C	Isotropic =	101.0505	30	H	Isotropic =	23.7763
4	C	Isotropic =	47.8541	31	H	Isotropic =	24.4329
5	C	Isotropic =	57.3462	32	H	Isotropic =	25.3772
6	C	Isotropic =	51.2848	33	H	Isotropic =	28.2214
7	C	Isotropic =	71.9486	34	H	Isotropic =	31.0741
8	C	Isotropic =	35.1191	35	H	Isotropic =	30.3962
9	C	Isotropic =	50.6689	36	H	Isotropic =	30.6682
10	C	Isotropic =	-31.7935	37	H	Isotropic =	30.6696
11	C	Isotropic =	103.2573	38	H	Isotropic =	29.3534
12	C	Isotropic =	123.3461	39	H	Isotropic =	29.7256
13	C	Isotropic =	36.3752	40	H	Isotropic =	30.2138
14	C	Isotropic =	53.0628	41	H	Isotropic =	30.9754
15	C	Isotropic =	124.3429	42	H	Isotropic =	29.6424
16	C	Isotropic =	136.0421	43	H	Isotropic =	24.9394
17	C	Isotropic =	162.8294	44	H	Isotropic =	25.6098
18	C	Isotropic =	161.6207	45	H	Isotropic =	25.7081
19	C	Isotropic =	164.8084	46	H	Isotropic =	29.0151
20	C	Isotropic =	39.1842	47	H	Isotropic =	27.6030
21	C	Isotropic =	59.8987	48	H	Isotropic =	29.0021
23	C	Isotropic =	10.1194	49	H	Isotropic =	29.7586
24	C	Isotropic =	160.7944				

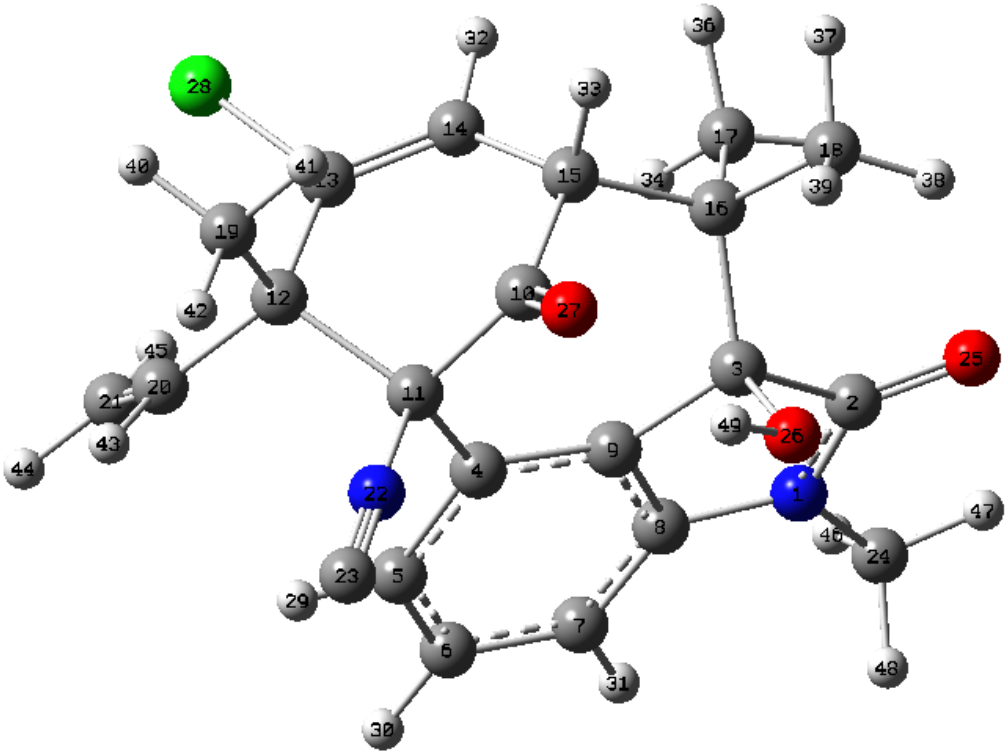


Structure 4, C3 epimer, conformer 2

Sum of electronic and thermal free energies = -1645.984941 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.278393	1.117972	-0.533461
2	6	-3.310383	-0.209469	-0.178852
3	6	-1.902906	-0.587507	0.389296
4	6	0.185832	1.105916	0.535168
5	6	0.498441	2.480029	0.471956
6	6	-0.414085	3.416622	-0.001700
7	6	-1.694216	3.031209	-0.411083
8	6	-2.023780	1.689516	-0.276956
9	6	-1.117140	0.717749	0.196082
10	6	0.678948	-1.295351	1.201769
11	6	1.266480	0.132206	1.075058
12	6	2.607114	-0.025916	0.162271
13	6	2.176542	-0.689437	-1.139566
14	6	1.130719	-1.511176	-1.253244
15	6	0.261511	-1.951592	-0.102602
16	6	-1.317902	-1.862164	-0.319460
17	6	-1.631250	-1.813554	-1.830465
18	6	-1.963050	-3.129295	0.280033
19	6	3.588562	-1.007686	0.872083
20	6	3.341014	1.300907	0.055371
21	6	3.389177	2.161408	-0.962656
22	7	1.668367	0.584181	2.365535
23	6	1.977950	0.945099	3.441586
24	6	-4.440003	1.843416	-1.011636
25	8	-4.279727	-0.942319	-0.263731
26	8	-2.211929	-0.787911	1.773533
27	8	0.548325	-1.842008	2.279464
28	17	3.272230	-0.583978	-2.525662
29	1	1.463443	2.821562	0.813100
30	1	-0.129282	4.463551	-0.041017
31	1	-2.410581	3.759099	-0.775830
32	1	0.969213	-2.010491	-2.201256
33	1	0.466678	-3.020853	0.041535
34	1	-1.277141	-0.890238	-2.301119
35	1	-2.708330	-1.897056	-1.988036
36	1	-1.163913	-2.657356	-2.347271
37	1	-1.653516	-4.002908	-0.305300
38	1	-3.051393	-3.051544	0.248445
39	1	-1.661113	-3.290452	1.316941
40	1	4.477093	-1.126810	0.246868
41	1	3.145988	-1.995540	1.023409
42	1	3.899830	-0.615425	1.842892
43	1	3.899484	1.542858	0.957834
44	1	3.981398	3.068289	-0.877126
45	1	2.857633	2.015263	-1.895536
46	1	-4.242538	2.286829	-1.993526
47	1	-5.261236	1.130562	-1.092827
48	1	-4.718029	2.636531	-0.308601
49	1	-1.420214	-1.020993	2.279312

2	C	Isotropic =	8.5407	29	H	Isotropic =	23.7140
3	C	Isotropic =	100.7337	30	H	Isotropic =	23.8762
4	C	Isotropic =	48.2975	31	H	Isotropic =	24.4155
5	C	Isotropic =	56.1226	32	H	Isotropic =	25.3406
6	C	Isotropic =	52.0942	33	H	Isotropic =	28.1967
7	C	Isotropic =	72.0936	34	H	Isotropic =	31.0889
8	C	Isotropic =	35.5088	35	H	Isotropic =	30.4055
9	C	Isotropic =	50.9200	36	H	Isotropic =	30.6679
10	C	Isotropic =	-31.9216	37	H	Isotropic =	30.7191
11	C	Isotropic =	103.7739	38	H	Isotropic =	29.3263
12	C	Isotropic =	120.9632	39	H	Isotropic =	29.6950
13	C	Isotropic =	38.4030	40	H	Isotropic =	29.8207
14	C	Isotropic =	50.6647	41	H	Isotropic =	30.8758
15	C	Isotropic =	123.5190	42	H	Isotropic =	29.8531
16	C	Isotropic =	137.1848	43	H	Isotropic =	25.2441
17	C	Isotropic =	162.7858	44	H	Isotropic =	25.4090
18	C	Isotropic =	161.7333	45	H	Isotropic =	26.1240
19	C	Isotropic =	159.8194	46	H	Isotropic =	29.0313
20	C	Isotropic =	43.5803	47	H	Isotropic =	27.5970
21	C	Isotropic =	52.4288	48	H	Isotropic =	29.0234
23	C	Isotropic =	9.4675	49	H	Isotropic =	29.7756
24	C	Isotropic =	160.9283				

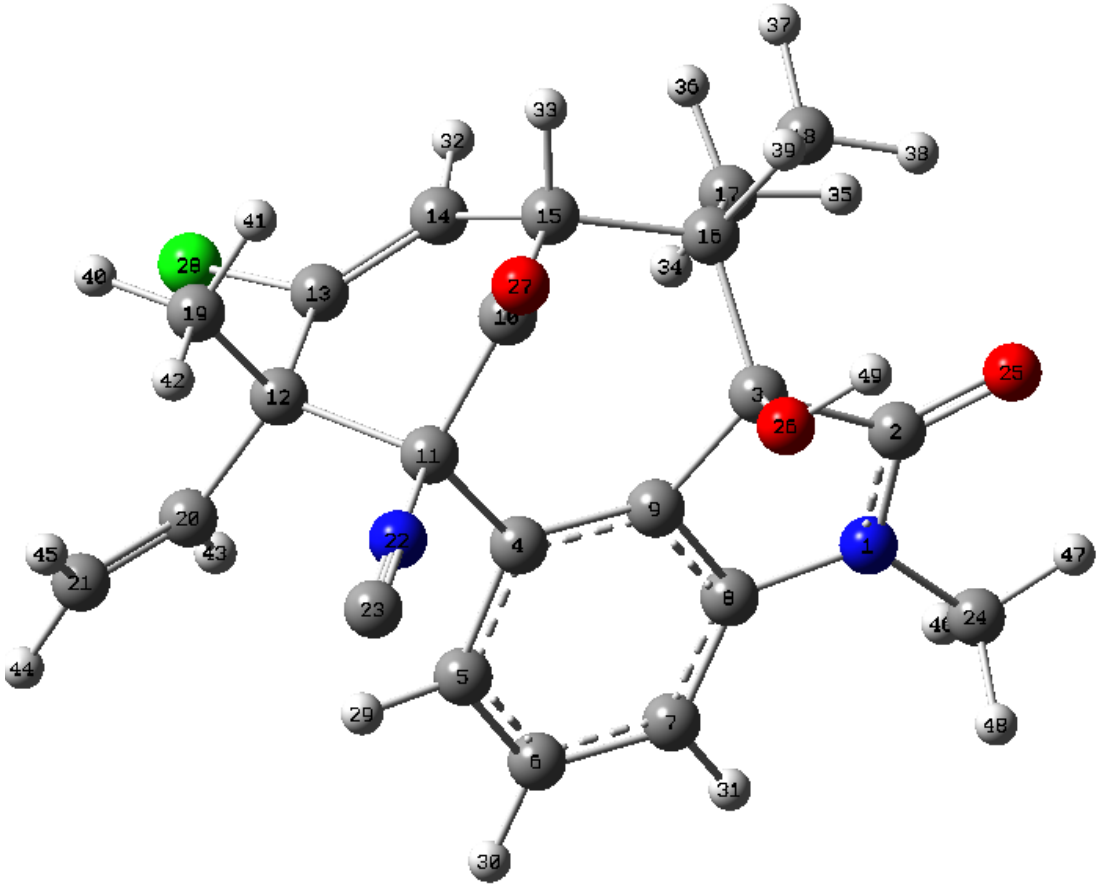


Structure 4, C3 epimer, conformer 3

Sum of electronic and thermal free energies = -1645.993218 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.304913	1.158080	-0.473247
2	6	-3.343767	-0.124220	0.012860
3	6	-1.900808	-0.526113	0.438143
4	6	0.202583	1.098414	0.441505
5	6	0.549857	2.458531	0.300853
6	6	-0.349169	3.394542	-0.201661
7	6	-1.654658	3.030875	-0.552499
8	6	-2.016782	1.709150	-0.335573
9	6	-1.114512	0.741754	0.146499
10	6	0.712752	-1.342329	1.145440
11	6	1.262646	0.108362	0.982553
12	6	2.560517	-0.074405	0.005008
13	6	2.038092	-0.733226	-1.267192
14	6	0.977493	-1.539015	-1.335178
15	6	0.165346	-1.970739	-0.137086
16	6	-1.410666	-1.832481	-0.278582
17	6	-1.827944	-1.804664	-1.763284
18	6	-2.052035	-3.069296	0.389982
19	6	3.578225	-1.040036	0.666839
20	6	3.217110	1.261431	-0.309193
21	6	4.289669	1.774829	0.297376
22	7	1.719682	0.579075	2.241375
23	6	2.085755	0.958664	3.292619
24	6	-4.488163	1.905444	-0.858646
25	8	-4.346060	-0.811932	0.151027
26	8	-1.913560	-0.658144	1.862321
27	8	0.831333	-1.960728	2.176980
28	17	3.004922	-0.490953	-2.728440
29	1	1.529805	2.790146	0.614743
30	1	-0.036243	4.429139	-0.304160
31	1	-2.360881	3.764169	-0.926083
32	1	0.737318	-2.002744	-2.285010
33	1	0.350153	-3.045699	-0.012173
34	1	-1.474030	-0.906708	-2.279741
35	1	-2.916945	-1.848538	-1.846404
36	1	-1.430496	-2.676692	-2.291910
37	1	-1.805258	-3.964359	-0.191192
38	1	-3.141320	-2.978058	0.419119
39	1	-1.671232	-3.222910	1.403675
40	1	4.445008	-1.142476	0.009787
41	1	3.150534	-2.030982	0.826388
42	1	3.914830	-0.662151	1.634015
43	1	2.757918	1.825543	-1.114875
44	1	4.680780	2.740848	-0.008234
45	1	4.811897	1.277404	1.107423
46	1	-4.380090	2.298264	-1.874694
47	1	-5.337383	1.222389	-0.820208
48	1	-4.662884	2.738110	-0.167863
49	1	-2.611489	-1.284980	2.106659

2	C	Isotropic =	4.5082	29	H	Isotropic =	23.7408
3	C	Isotropic =	102.2206	30	H	Isotropic =	23.7433
4	C	Isotropic =	46.4347	31	H	Isotropic =	24.3823
5	C	Isotropic =	56.3911	32	H	Isotropic =	25.4292
6	C	Isotropic =	51.1940	33	H	Isotropic =	28.4365
7	C	Isotropic =	71.9627	34	H	Isotropic =	31.0058
8	C	Isotropic =	35.0494	35	H	Isotropic =	30.6109
9	C	Isotropic =	52.6692	36	H	Isotropic =	30.6921
10	C	Isotropic =	-22.5454	37	H	Isotropic =	30.6963
11	C	Isotropic =	104.8751	38	H	Isotropic =	29.5775
12	C	Isotropic =	122.4370	39	H	Isotropic =	29.9092
13	C	Isotropic =	36.5446	40	H	Isotropic =	30.3197
14	C	Isotropic =	52.0920	41	H	Isotropic =	30.9179
15	C	Isotropic =	123.7915	42	H	Isotropic =	29.7019
16	C	Isotropic =	136.4725	43	H	Isotropic =	24.9580
17	C	Isotropic =	164.0214	44	H	Isotropic =	25.6285
18	C	Isotropic =	164.0273	45	H	Isotropic =	25.6935
19	C	Isotropic =	164.9704	46	H	Isotropic =	28.9284
20	C	Isotropic =	38.3761	47	H	Isotropic =	27.6248
21	C	Isotropic =	60.1109	48	H	Isotropic =	29.0147
23	C	Isotropic =	11.2619	49	H	Isotropic =	29.5616
24	C	Isotropic =	160.4922				

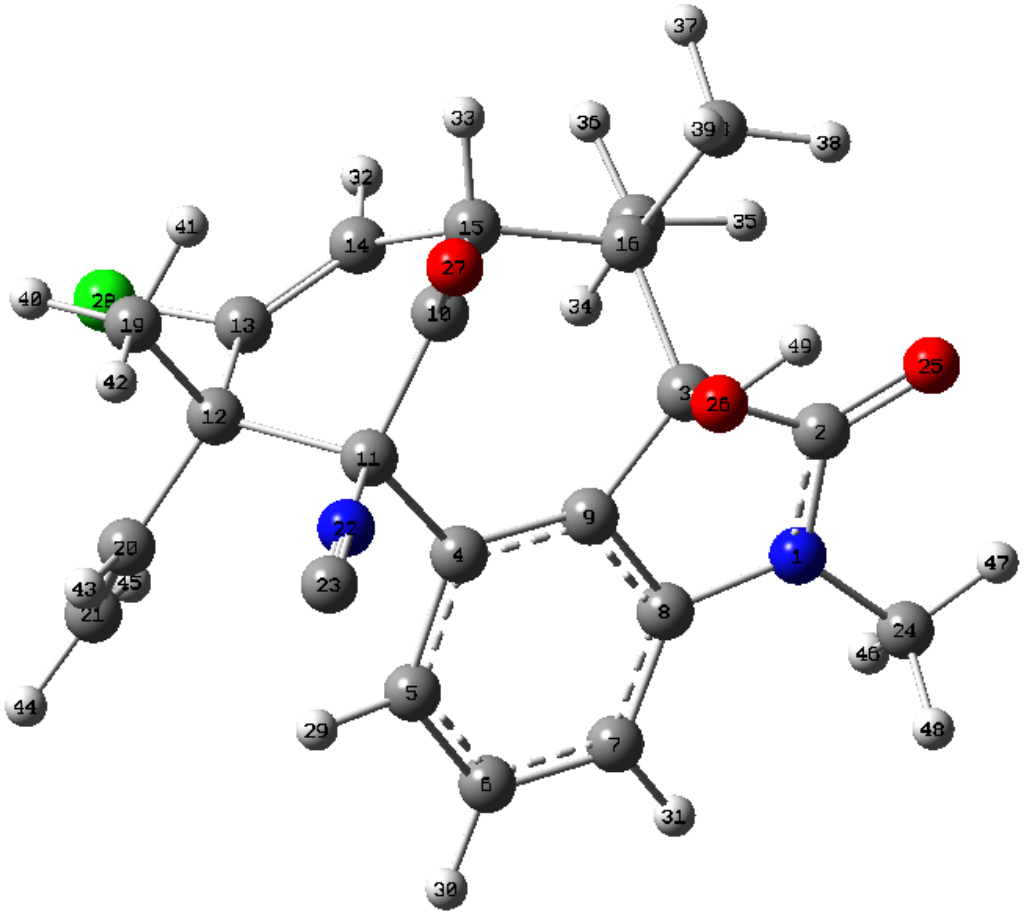


Structure 4, C3 epimer, conformer 4

Sum of electronic and thermal free energies = -1645.987825 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.296992	1.098062	-0.519742
2	6	-3.317437	-0.210481	-0.111600
3	6	-1.884018	-0.593435	0.359311
4	6	0.167951	1.087122	0.557247
5	6	0.468669	2.464755	0.525766
6	6	-0.438472	3.403086	0.042077
7	6	-1.709896	3.021859	-0.399926
8	6	-2.034115	1.677458	-0.287498
9	6	-1.123219	0.709965	0.176459
10	6	0.714456	-1.368430	1.162040
11	6	1.234122	0.098595	1.090693
12	6	2.587794	-0.006527	0.172366
13	6	2.166070	-0.632158	-1.150719
14	6	1.121171	-1.449679	-1.302106
15	6	0.245808	-1.942975	-0.174963
16	6	-1.320668	-1.840181	-0.402156
17	6	-1.650628	-1.730027	-1.905135
18	6	-1.963149	-3.132190	0.150963
19	6	3.600765	-0.983010	0.844450
20	6	3.295345	1.337588	0.109250
21	6	3.360863	2.218882	-0.890052
22	7	1.625594	0.519045	2.392415
23	6	1.929422	0.848788	3.479734
24	6	-4.483964	1.831598	-0.919345
25	8	-4.301992	-0.935598	-0.063345
26	8	-1.957268	-0.806376	1.772259
27	8	0.808081	-2.034886	2.165788
28	17	3.272247	-0.483221	-2.526924
29	1	1.419198	2.810462	0.901120
30	1	-0.157996	4.451844	0.025838
31	1	-2.423665	3.754808	-0.759696
32	1	0.969832	-1.914653	-2.269756
33	1	0.455063	-3.017670	-0.091631
34	1	-1.288388	-0.794356	-2.343013
35	1	-2.731464	-1.790928	-2.056824
36	1	-1.202105	-2.559204	-2.460985
37	1	-1.657828	-3.982723	-0.468039
38	1	-3.054361	-3.070632	0.122706
39	1	-1.636281	-3.339575	1.173934
40	1	4.498186	-1.032525	0.222334
41	1	3.198684	-1.992566	0.946629
42	1	3.887046	-0.623190	1.835406
43	1	3.824146	1.570334	1.031876
44	1	3.936363	3.132432	-0.768057
45	1	2.859783	2.084733	-1.841219
46	1	-4.339782	2.288596	-1.903624
47	1	-5.310886	1.122067	-0.964380
48	1	-4.720173	2.615082	-0.190315
49	1	-2.645567	-1.466065	1.947961

2	C	Isotropic =	4.8033	29	H	Isotropic =	23.7027
3	C	Isotropic =	101.9481	30	H	Isotropic =	23.8476
4	C	Isotropic =	47.3036	31	H	Isotropic =	24.3863
5	C	Isotropic =	54.9469	32	H	Isotropic =	25.4145
6	C	Isotropic =	52.1010	33	H	Isotropic =	28.4066
7	C	Isotropic =	72.2651	34	H	Isotropic =	31.0133
8	C	Isotropic =	35.5006	35	H	Isotropic =	30.6263
9	C	Isotropic =	52.9277	36	H	Isotropic =	30.6721
10	C	Isotropic =	-22.4746	37	H	Isotropic =	30.6941
11	C	Isotropic =	105.2378	38	H	Isotropic =	29.5224
12	C	Isotropic =	119.2896	39	H	Isotropic =	29.8903
13	C	Isotropic =	38.7276	40	H	Isotropic =	29.9140
14	C	Isotropic =	49.9270	41	H	Isotropic =	30.7893
15	C	Isotropic =	122.8680	42	H	Isotropic =	29.8884
16	C	Isotropic =	137.0406	43	H	Isotropic =	25.2216
17	C	Isotropic =	163.9270	44	H	Isotropic =	25.4410
18	C	Isotropic =	164.2834	45	H	Isotropic =	26.1526
19	C	Isotropic =	160.3357	46	H	Isotropic =	28.9489
20	C	Isotropic =	42.8063	47	H	Isotropic =	27.6230
21	C	Isotropic =	53.4203	48	H	Isotropic =	29.0286
23	C	Isotropic =	10.6569	49	H	Isotropic =	29.5085
24	C	Isotropic =	160.6551				

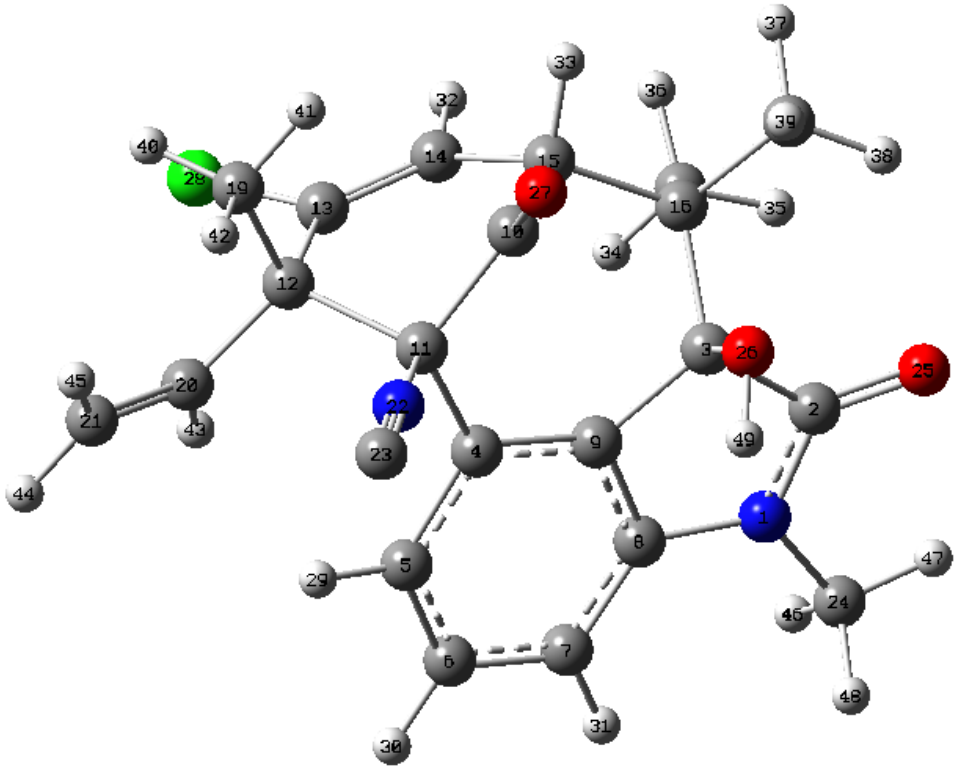


Structure 4, C3 epimer, conformer 5

Sum of electronic and thermal free energies = -1645.991068 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.316210	1.144844	-0.470852
2	6	-3.353000	-0.145626	0.016414
3	6	-1.900274	-0.530040	0.456442
4	6	0.195691	1.110009	0.426253
5	6	0.535916	2.470473	0.269947
6	6	-0.371177	3.397256	-0.235857
7	6	-1.677562	3.024701	-0.573098
8	6	-2.033824	1.702602	-0.342798
9	6	-1.122774	0.741996	0.142090
10	6	0.725498	-1.322317	1.175416
11	6	1.263098	0.131843	0.975234
12	6	2.556738	-0.062327	-0.003870
13	6	2.036001	-0.767570	-1.251864
14	6	0.981869	-1.583705	-1.293192
15	6	0.165831	-1.980456	-0.085158
16	6	-1.411863	-1.842012	-0.237032
17	6	-1.816382	-1.832304	-1.725165
18	6	-2.065012	-3.064028	0.447132
19	6	3.592983	-0.991378	0.680623
20	6	3.190944	1.272471	-0.365900
21	6	4.266338	1.816945	0.207755
22	7	1.721041	0.632824	2.223792
23	6	2.081051	1.038479	3.267421
24	6	-4.496318	1.870426	-0.902808
25	8	-4.354671	-0.833775	0.108351
26	8	-1.897460	-0.778516	1.865497
27	8	0.880065	-1.916681	2.215989
28	17	3.001830	-0.566105	-2.720418
29	1	1.517588	2.808976	0.570713
30	1	-0.063227	4.431928	-0.351967
31	1	-2.388990	3.750671	-0.951099
32	1	0.747195	-2.079790	-2.227923
33	1	0.342733	-3.052577	0.068531
34	1	-1.453253	-0.945486	-2.255049
35	1	-2.904431	-1.874170	-1.816303
36	1	-1.419353	-2.715718	-2.234178
37	1	-1.807241	-3.967991	-0.116408
38	1	-3.151829	-2.961174	0.463288
39	1	-1.713413	-3.182768	1.473244
40	1	4.452775	-1.107859	0.016800
41	1	3.178446	-1.980092	0.881730
42	1	3.937337	-0.574397	1.628771
43	1	2.712322	1.806697	-1.180794
44	1	4.640389	2.777977	-0.133001
45	1	4.808607	1.349899	1.022736
46	1	-4.369044	2.238269	-1.926233
47	1	-5.338560	1.178550	-0.866659
48	1	-4.697542	2.719456	-0.239400
49	1	-1.981566	0.058863	2.343130

2	C	Isotropic =	5.1952	29	H	Isotropic =	23.6966
3	C	Isotropic =	102.1608	30	H	Isotropic =	23.7777
4	C	Isotropic =	46.0600	31	H	Isotropic =	24.4347
5	C	Isotropic =	56.4614	32	H	Isotropic =	25.3922
6	C	Isotropic =	50.9654	33	H	Isotropic =	28.3243
7	C	Isotropic =	72.0633	34	H	Isotropic =	31.0289
8	C	Isotropic =	35.4876	35	H	Isotropic =	30.5817
9	C	Isotropic =	52.2471	36	H	Isotropic =	30.7290
10	C	Isotropic =	-21.8691	37	H	Isotropic =	30.7986
11	C	Isotropic =	105.0030	38	H	Isotropic =	29.4384
12	C	Isotropic =	122.2357	39	H	Isotropic =	29.6777
13	C	Isotropic =	36.8419	40	H	Isotropic =	30.2849
14	C	Isotropic =	51.7461	41	H	Isotropic =	30.8969
15	C	Isotropic =	123.6310	42	H	Isotropic =	29.7062
16	C	Isotropic =	136.1821	43	H	Isotropic =	24.9935
17	C	Isotropic =	163.9169	44	H	Isotropic =	25.6372
18	C	Isotropic =	163.1060	45	H	Isotropic =	25.7188
19	C	Isotropic =	165.0023	46	H	Isotropic =	28.9835
20	C	Isotropic =	38.3576	47	H	Isotropic =	27.5879
21	C	Isotropic =	60.6279	48	H	Isotropic =	29.0337
23	C	Isotropic =	11.3981	49	H	Isotropic =	30.5600
24	C	Isotropic =	160.7619				

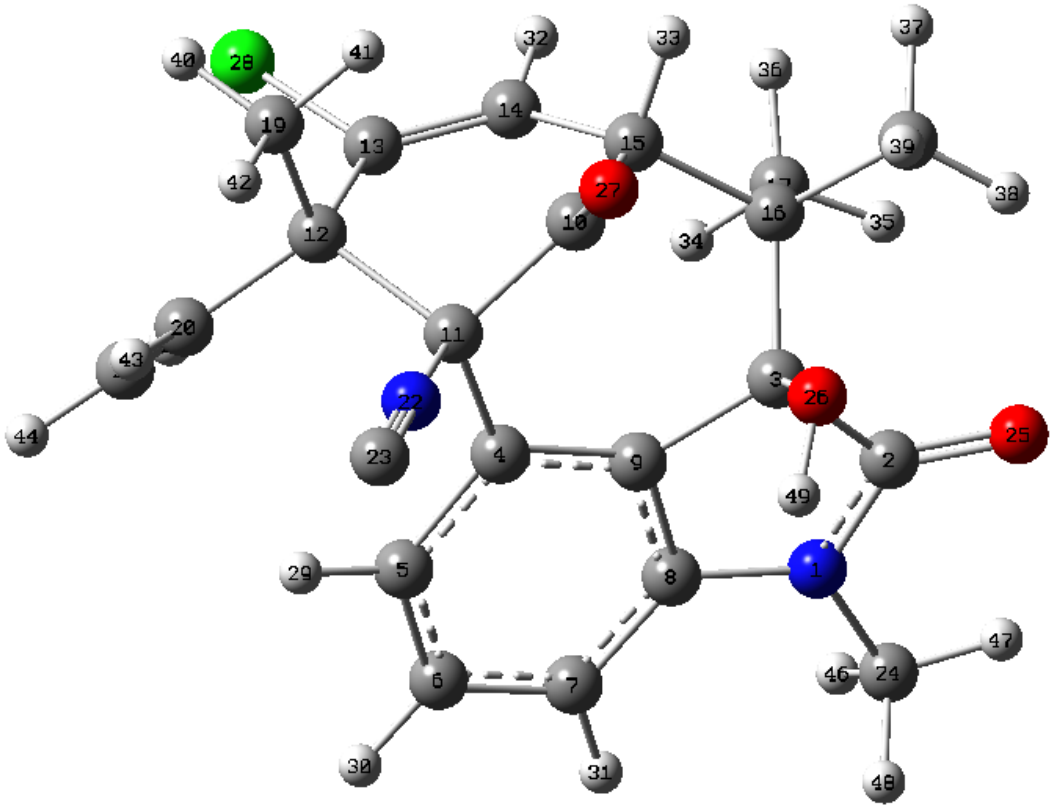


Structure 4, C3 epimer, conformer 6

Sum of electronic and thermal free energies = -1645.986160 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	-3.312624	1.070087	-0.517719
2	6	-3.323492	-0.246966	-0.110163
3	6	-1.880062	-0.604648	0.379318
4	6	0.156962	1.103937	0.542283
5	6	0.443307	2.484536	0.495961
6	6	-0.476925	3.408969	0.009569
7	6	-1.747298	3.010784	-0.419833
8	6	-2.058333	1.663388	-0.294924
9	6	-1.133600	0.707541	0.171626
10	6	0.735897	-1.340861	1.196557
11	6	1.236535	0.133516	1.082426
12	6	2.584691	0.023322	0.157121
13	6	2.167810	-0.661675	-1.137978
14	6	1.133465	-1.497661	-1.257865
15	6	0.255063	-1.954172	-0.117195
16	6	-1.312905	-1.858185	-0.354434
17	6	-1.630140	-1.772956	-1.861439
18	6	-1.962419	-3.137148	0.220416
19	6	3.624972	-0.902505	0.857785
20	6	3.258600	1.380226	0.032286
21	6	3.287376	2.220559	-1.003236
22	7	1.631285	0.589556	2.372781
23	6	1.930996	0.948564	3.451996
24	6	-4.497128	1.778340	-0.965471
25	8	-4.301112	-0.975325	-0.109797
26	8	-1.933180	-0.927604	1.772579
27	8	0.869596	-1.978865	2.213930
28	17	3.274935	-0.554405	-2.518122
29	1	1.393378	2.843624	0.859876
30	1	-0.206985	4.460228	-0.019166
31	1	-2.470221	3.732707	-0.783612
32	1	0.991383	-2.002287	-2.206844
33	1	0.458766	-3.026367	-0.000361
34	1	-1.261198	-0.847576	-2.316215
35	1	-2.709337	-1.836217	-2.020688
36	1	-1.179506	-2.614846	-2.395392
37	1	-1.640594	-3.999269	-0.374910
38	1	-3.051135	-3.069846	0.176867
39	1	-1.667942	-3.302582	1.258096
40	1	4.515211	-0.960810	0.226305
41	1	3.244144	-1.914013	1.008749
42	1	3.916936	-0.494254	1.828047
43	1	3.794607	1.662499	0.936979
44	1	3.840698	3.152754	-0.929232
45	1	2.777150	2.032186	-1.940362
46	1	-4.335374	2.210442	-1.958528
47	1	-5.313299	1.056398	-1.011084
48	1	-4.762161	2.579062	-0.265378
49	1	-2.081707	-0.121526	2.286962

2	C	Isotropic =	5.6260	29	H	Isotropic =	23.7169
3	C	Isotropic =	101.7692	30	H	Isotropic =	23.8617
4	C	Isotropic =	46.8953	31	H	Isotropic =	24.4447
5	C	Isotropic =	54.4966	32	H	Isotropic =	25.3600
6	C	Isotropic =	51.9572	33	H	Isotropic =	28.2891
7	C	Isotropic =	72.3771	34	H	Isotropic =	31.0062
8	C	Isotropic =	36.0630	35	H	Isotropic =	30.6048
9	C	Isotropic =	52.3668	36	H	Isotropic =	30.7046
10	C	Isotropic =	-21.6655	37	H	Isotropic =	30.8278
11	C	Isotropic =	105.1827	38	H	Isotropic =	29.3924
12	C	Isotropic =	119.2166	39	H	Isotropic =	29.6543
13	C	Isotropic =	39.0046	40	H	Isotropic =	29.9060
14	C	Isotropic =	49.4286	41	H	Isotropic =	30.7720
15	C	Isotropic =	122.6971	42	H	Isotropic =	29.9302
16	C	Isotropic =	136.9722	43	H	Isotropic =	25.2462
17	C	Isotropic =	163.8095	44	H	Isotropic =	25.4644
18	C	Isotropic =	163.3482	45	H	Isotropic =	26.2006
19	C	Isotropic =	160.5311	46	H	Isotropic =	28.9944
20	C	Isotropic =	43.0063	47	H	Isotropic =	27.5786
21	C	Isotropic =	53.1354	48	H	Isotropic =	29.0435
23	C	Isotropic =	10.8252	49	H	Isotropic =	30.5209
24	C	Isotropic =	160.9110				



Structure 3

Sum of electronic and thermal free energies = -2044.228627 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	3.491312	-1.137705	-0.667141
2	6	3.468337	-0.007255	0.098748
3	6	1.986878	0.464263	0.228362
4	6	-0.133580	-0.961406	-0.663930
5	6	-0.448307	-2.047557	-1.512488
6	6	0.528610	-2.836392	-2.106567
7	6	1.883265	-2.602645	-1.856977
8	6	2.193617	-1.523155	-1.044414
9	6	1.225434	-0.665468	-0.473704
10	6	-0.926198	0.041919	1.521845
11	6	-1.341403	-0.285430	0.058195
12	6	-1.939853	1.036289	-0.661648
13	6	-1.153515	2.259394	-0.206882
14	6	-0.324087	2.348248	0.831109
15	6	0.033732	1.196340	1.733996
16	6	1.559826	0.785272	1.692658
17	6	2.368264	1.966387	2.277266
18	6	1.797448	-0.449467	2.587994
19	6	-3.404688	1.245858	-0.205723
20	6	-1.813324	0.867272	-2.169007
21	6	-2.805218	0.640241	-3.031626
22	7	-2.400780	-1.249285	0.099800
23	6	-3.026178	-1.987924	0.812450
24	6	4.713110	-1.784422	-1.109825
25	8	4.449019	0.604419	0.503796
26	8	1.928088	1.619649	-0.628398
27	8	-1.377734	-0.588383	2.453942
28	17	-1.455073	3.726927	-1.150965
29	1	-1.488757	-2.280390	-1.692551
30	1	0.230614	-3.658003	-2.750493
31	1	2.653856	-3.237184	-2.280203
32	1	0.129295	3.307096	1.055940
33	1	-0.141596	1.501619	2.773437
34	1	3.432782	1.731081	2.308767
35	1	2.026890	2.166259	3.298657
36	1	2.244315	2.887411	1.701496
37	1	1.399713	-0.274079	3.591868
38	1	2.870180	-0.644828	2.683311
39	1	1.317164	-1.351718	2.199932
40	1	-3.775657	2.193237	-0.602938
41	1	-3.467205	1.285730	0.885515
42	1	-4.056144	0.443475	-0.552501
43	1	-0.795340	0.940058	-2.545759
44	1	-2.595628	0.529276	-4.091361
45	1	-3.845373	0.563052	-2.732652
46	1	5.550706	-1.247887	-0.663214
47	1	4.797453	-1.744513	-2.201407
48	1	4.734444	-2.830131	-0.786015
49	1	2.712081	2.160658	-0.443242
50	16	-3.967455	-3.023543	1.559218

2	C	Isotropic =	4.8855	29	H	Isotropic =	23.9984
3	C	Isotropic =	101.8740	30	H	Isotropic =	23.8156
4	C	Isotropic =	47.0518	31	H	Isotropic =	24.3353
5	C	Isotropic =	55.6586	32	H	Isotropic =	24.8723
6	C	Isotropic =	50.8762	33	H	Isotropic =	28.3058
7	C	Isotropic =	72.5542	34	H	Isotropic =	29.2384
8	C	Isotropic =	34.1863	35	H	Isotropic =	30.8878
9	C	Isotropic =	54.4800	36	H	Isotropic =	29.9737
10	C	Isotropic =	-22.5835	37	H	Isotropic =	30.5709
11	C	Isotropic =	95.1988	38	H	Isotropic =	30.8818
12	C	Isotropic =	121.3605	39	H	Isotropic =	31.3575
13	C	Isotropic =	38.4718	40	H	Isotropic =	30.0976
14	C	Isotropic =	52.7774	41	H	Isotropic =	30.6923
15	C	Isotropic =	122.6604	42	H	Isotropic =	29.7414
16	C	Isotropic =	135.8042	43	H	Isotropic =	25.8781
17	C	Isotropic =	166.1601	44	H	Isotropic =	26.0590
18	C	Isotropic =	164.0081	45	H	Isotropic =	26.0025
19	C	Isotropic =	165.5144	46	H	Isotropic =	27.5723
20	C	Isotropic =	37.4105	47	H	Isotropic =	28.9626
21	C	Isotropic =	64.0130	48	H	Isotropic =	28.9522
23	C	Isotropic =	33.9484	49	H	Isotropic =	29.4462
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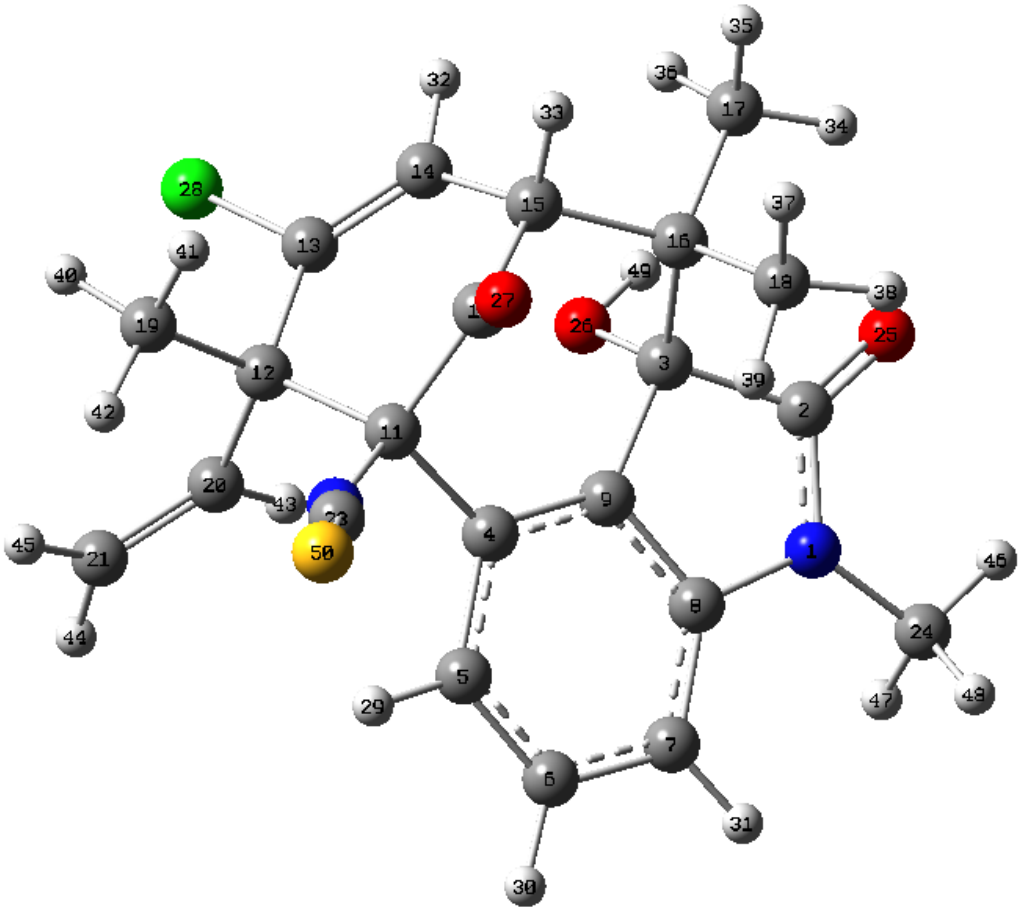
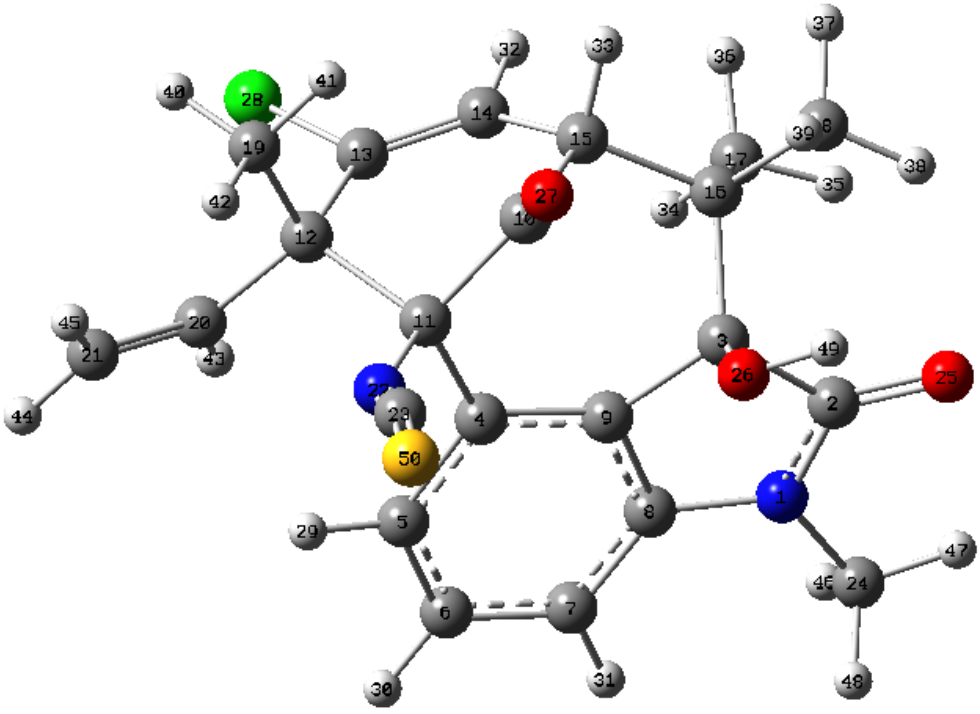


Structure 3, C3 epimer

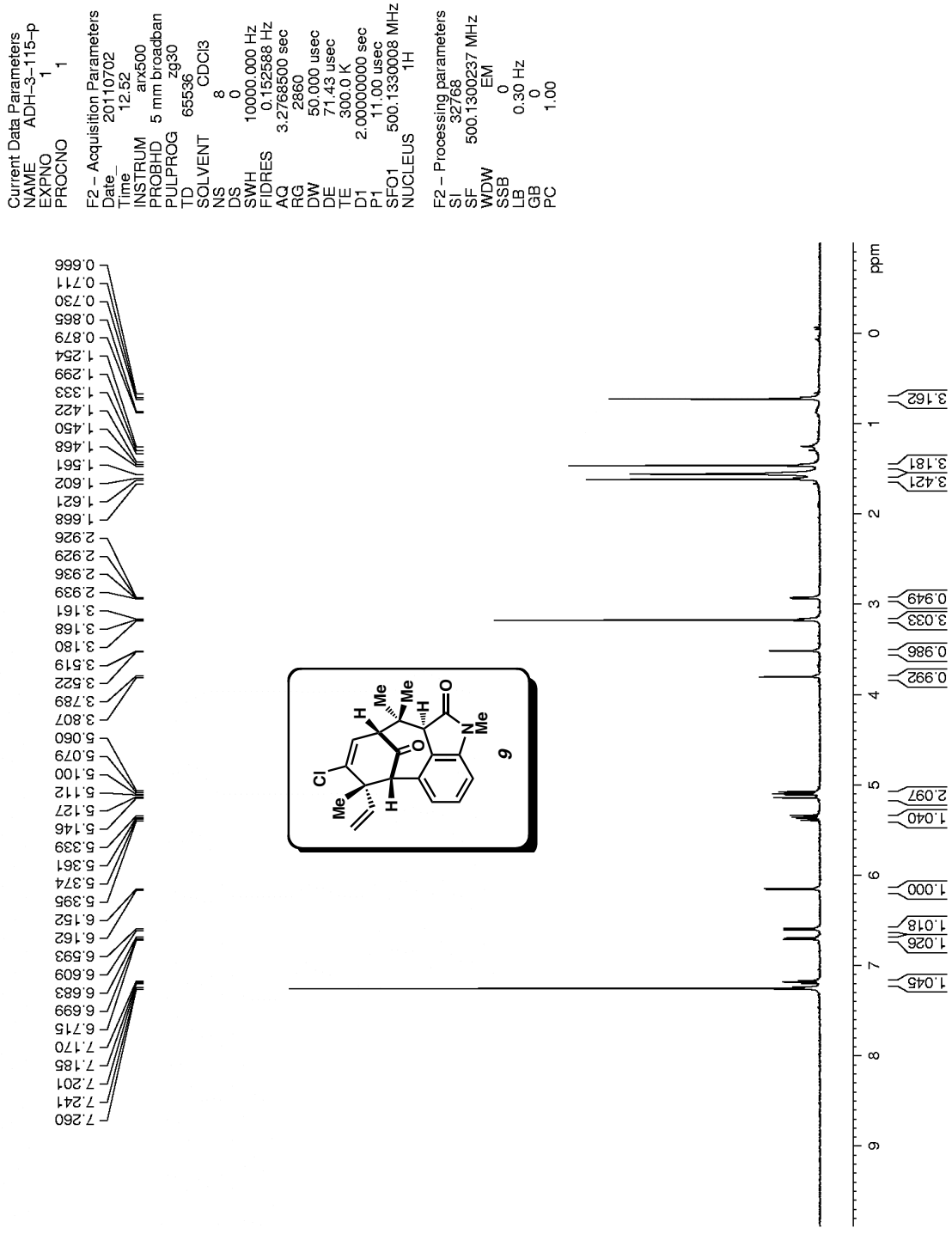
Sum of electronic and thermal free energies = -2044.226582 H

Center Number	Atomic Number	Coordinates (Angstroms) X	Y	Z
1	7	3.508897	0.034700	1.185185
2	6	3.461453	0.331733	-0.153051
3	6	1.966000	0.401803	-0.582116
4	6	-0.111304	0.205516	1.063527
5	6	-0.435091	0.191248	2.437306
6	6	0.537608	0.016818	3.417095
7	6	1.890944	-0.110910	3.081768
8	6	2.214182	-0.013667	1.736157
9	6	1.242227	0.135284	0.728037
10	6	-0.725084	0.459204	-1.433944
11	6	-1.250533	0.387511	0.031499
12	6	-2.314605	-0.848701	0.005622
13	6	-1.550717	-2.067344	-0.500202
14	6	-0.494633	-2.028799	-1.313731
15	6	0.069515	-0.759401	-1.906174
16	6	1.641233	-0.566342	-1.772568
17	6	2.342801	-1.921963	-1.550555
18	6	2.151556	0.042890	-3.098071
19	6	-3.443991	-0.532057	-1.008790
20	6	-2.897771	-1.117065	1.385510
21	6	-4.077035	-0.690080	1.842611
22	7	-1.968062	1.577656	0.363280
23	6	-2.174575	2.717022	0.037974
24	6	4.738606	0.001779	1.955649
25	8	4.422504	0.567687	-0.872661
26	8	1.697331	1.770336	-0.903737
27	8	-1.030153	1.365191	-2.175190
28	17	-2.213061	-3.648178	-0.059791
29	1	-1.461894	0.344490	2.738410
30	1	0.241807	-0.000457	4.461714
31	1	2.651105	-0.228044	3.846218
32	1	-0.072017	-2.964162	-1.662136
33	1	-0.123922	-0.810457	-2.985629
34	1	2.085865	-2.370219	-0.585513
35	1	3.427262	-1.794094	-1.598822
36	1	2.068332	-2.632093	-2.336814
37	1	2.038672	-0.692049	-3.902375
38	1	3.211259	0.303132	-3.027751
39	1	1.576691	0.928942	-3.382477
40	1	-4.158028	-1.358859	-1.016138
41	1	-3.055202	-0.406748	-2.020645
42	1	-3.973162	0.383702	-0.737975
43	1	-2.282292	-1.729326	2.037431
44	1	-4.399704	-0.946336	2.847548
45	1	-4.757634	-0.080157	1.258455
46	1	4.831705	-0.949218	2.489822
47	1	5.568994	0.110459	1.257192
48	1	4.765071	0.824457	2.679261
49	1	2.335551	2.059062	-1.573813
50	16	-2.567777	4.231745	-0.213587

2	C	Isotropic =	4.1786	29	H	Isotropic =	23.7346
3	C	Isotropic =	102.7844	30	H	Isotropic =	23.8586
4	C	Isotropic =	46.5561	31	H	Isotropic =	24.4441
5	C	Isotropic =	55.9890	32	H	Isotropic =	25.3803
6	C	Isotropic =	51.8771	33	H	Isotropic =	28.1997
7	C	Isotropic =	72.5350	34	H	Isotropic =	30.9148
8	C	Isotropic =	34.3350	35	H	Isotropic =	30.6643
9	C	Isotropic =	53.3429	36	H	Isotropic =	30.6711
10	C	Isotropic =	-25.4087	37	H	Isotropic =	30.7396
11	C	Isotropic =	99.9395	38	H	Isotropic =	29.5377
12	C	Isotropic =	122.9421	39	H	Isotropic =	29.8480
13	C	Isotropic =	35.4253	40	H	Isotropic =	30.2733
14	C	Isotropic =	51.8841	41	H	Isotropic =	30.9084
15	C	Isotropic =	123.6216	42	H	Isotropic =	29.6995
16	C	Isotropic =	136.1963	43	H	Isotropic =	24.9076
17	C	Isotropic =	163.4768	44	H	Isotropic =	25.7231
18	C	Isotropic =	163.6791	45	H	Isotropic =	25.8722
19	C	Isotropic =	165.1487	46	H	Isotropic =	28.9125
20	C	Isotropic =	36.7707	47	H	Isotropic =	27.5861
21	C	Isotropic =	61.9335	48	H	Isotropic =	29.0421
23	C	Isotropic =	32.4181	49	H	Isotropic =	29.5902
24	C	Isotropic =	160.6404				



^{1}H and ^{2}H NMR Spectra: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EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20110702
Time 12.52
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 2860
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300237 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
ppm
3.162
3.421
3.181
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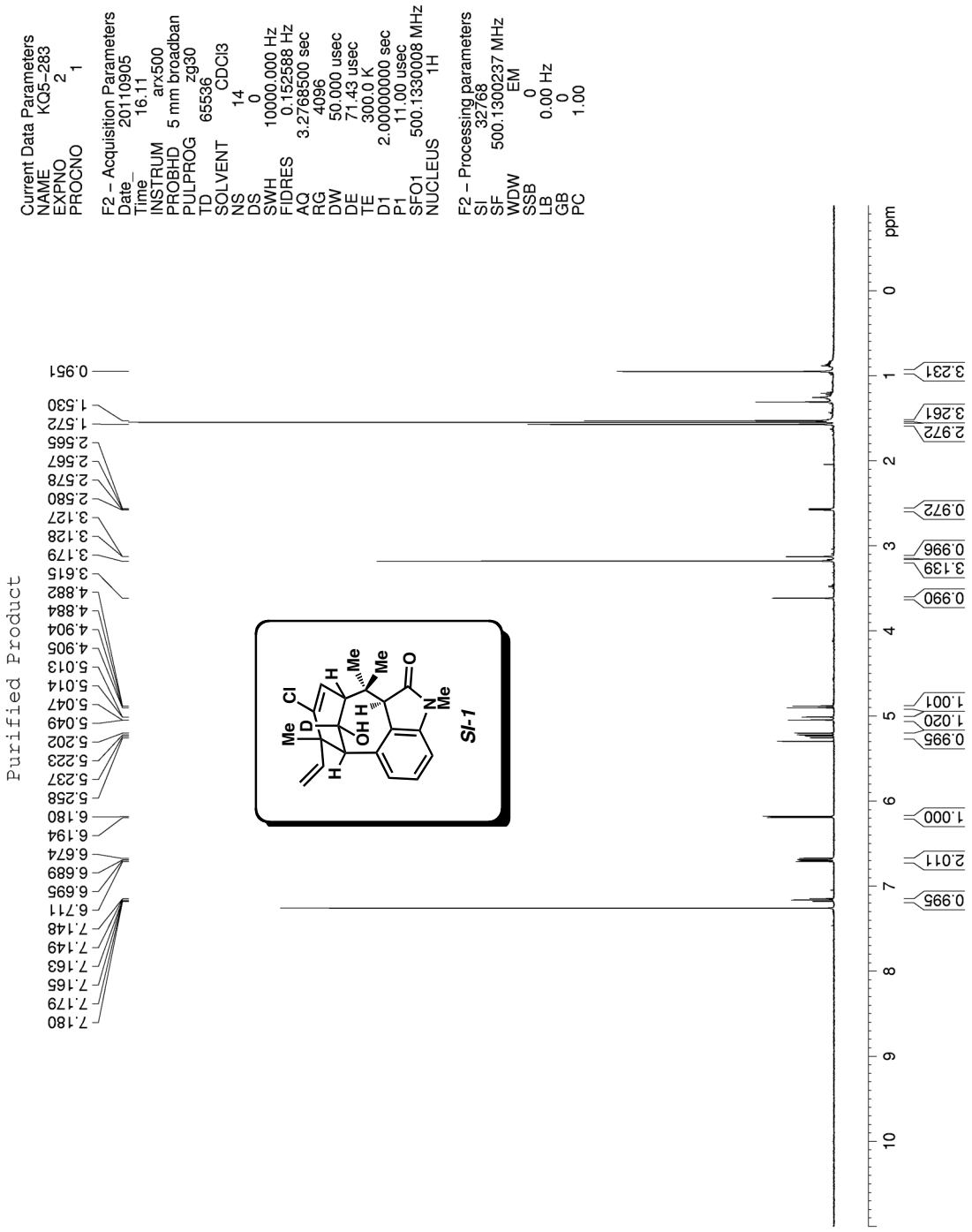
3.033
0.986
0.992
1.040
2.097
1.000
1.026
1.018
1.045


Purified Product
Current Data Parameters
NAME KQ5-283
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110905
Time 16.11
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 4096
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300237 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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1.572
2.565
2.567
2.578
2.580
3.127
3.128
3.179
3.615
4.882
4.884
4.904
4.905
5.013
5.014
5.047
5.049
5.202
5.223
5.237
5.258
6.180
6.194
6.674
6.689
6.695
6.711
7.148
7.149
7.163
7.165
7.179
7.180
Me
Cl
D
H
Me
Me
O
OH
H
N
Me
H
Sl-1
ppm
0
1
2
3
4
5
6
7
8
9
10
3.231
3.261
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1.020
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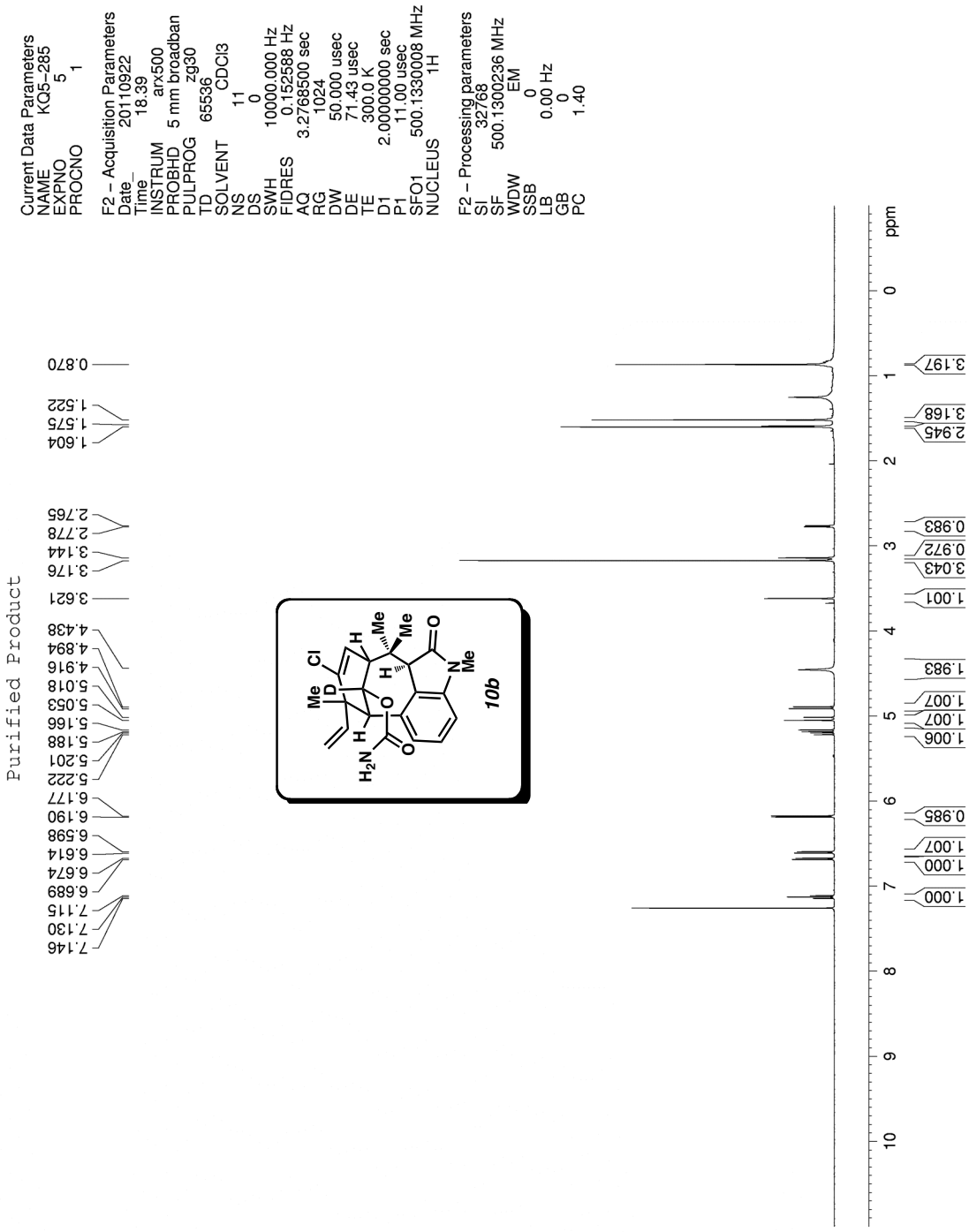
1.000
2.011
0.995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Current Data Parameters
NAME KQ5-285
EXPNO 5
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110922
Time 18.39
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
0.870
1.522
1.575
1.604
2.765
2.778
3.144
3.176
3.621
4.438
4.894
4.916
5.018
5.053
5.166
5.188
5.201
5.222
6.177
6.190
6.598
6.614
6.674
6.689
7.115
7.130
7.146
10b
3.197
3.168
2.945
0.983
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1.007
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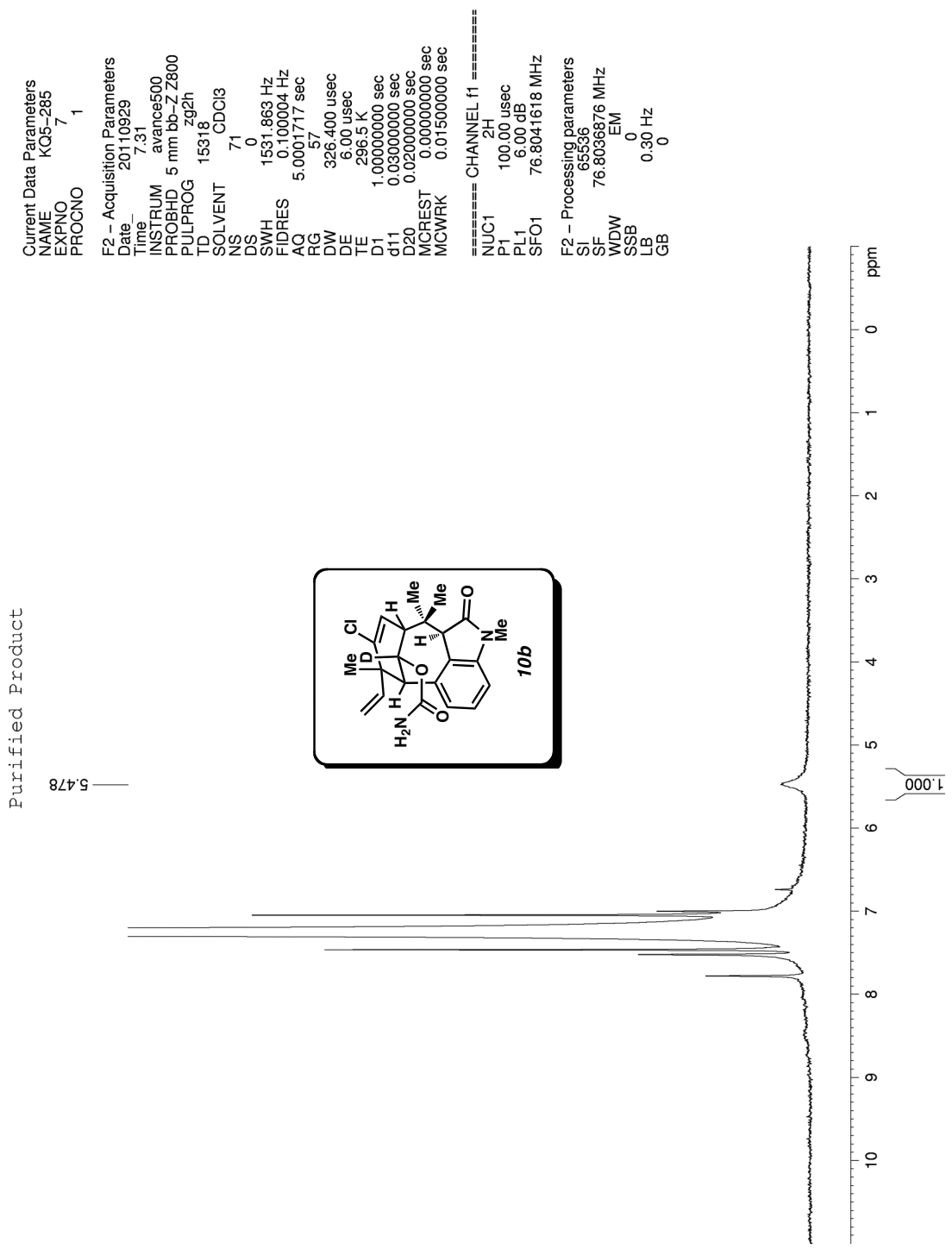
1.000
ppm


Purified Product
5.478
Current Data Parameters
NAME KQ5-285
EXPNO 7
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110929
Time 7.31
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg2h
TD 15318
SOLVENT CDCl3
NS 71
DS 0
SWH 1531.863 Hz
FIDRES 0.100004 Hz
AQ 5.0001717 sec
RG 57
DW 326.400 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
d11 0.03000000 sec
D20 0.02000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 2H
P1 100.00 usec
PL1 6.00 dB
SFO1 76.8041618 MHz
F2 – Processing parameters
SI 65536
SF 76.8036876 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
Me
Cl
D
H
Me
Me
O
H
N
Me
H
O
H2N
O
10b
1.000
ppm
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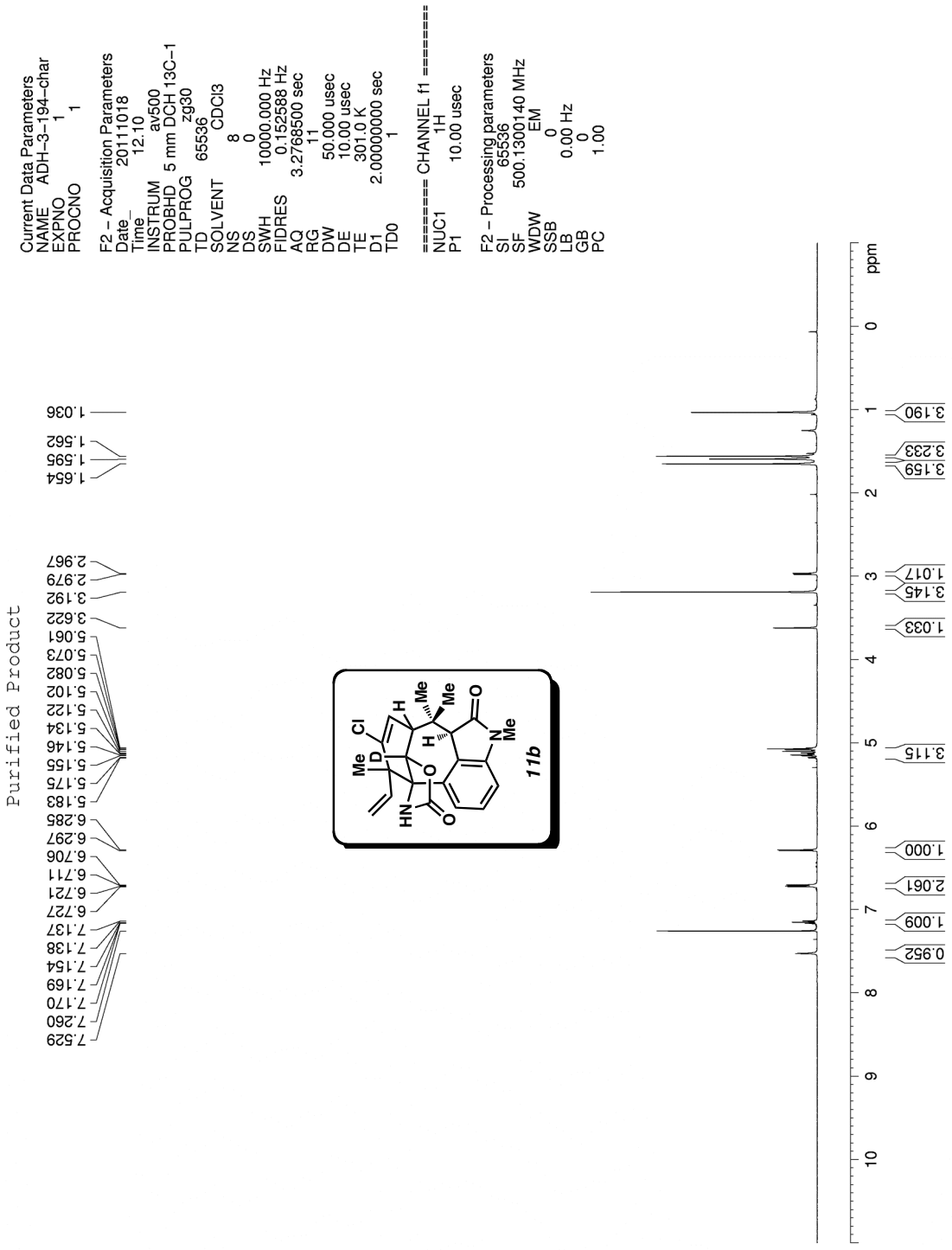
2
3
4
5
6
7
8
9
10


Purified Product
Current Data Parameters
NAME ADH–3–194–char
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111018
Time 12.10
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 301.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
F2 – Processing parameters
SI 65536
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.036
1.562
1.595
1.654
2.967
2.979
3.192
3.622
5.061
5.073
5.082
5.102
5.122
5.134
5.146
5.155
5.175
5.183
6.285
6.297
6.706
6.711
6.721
6.727
7.137
7.138
7.154
7.169
7.170
7.260
7.529
Me
Cl
D
H
Me
Me
O
H
N
Me
HN
O
O
11b
ppm
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3.115
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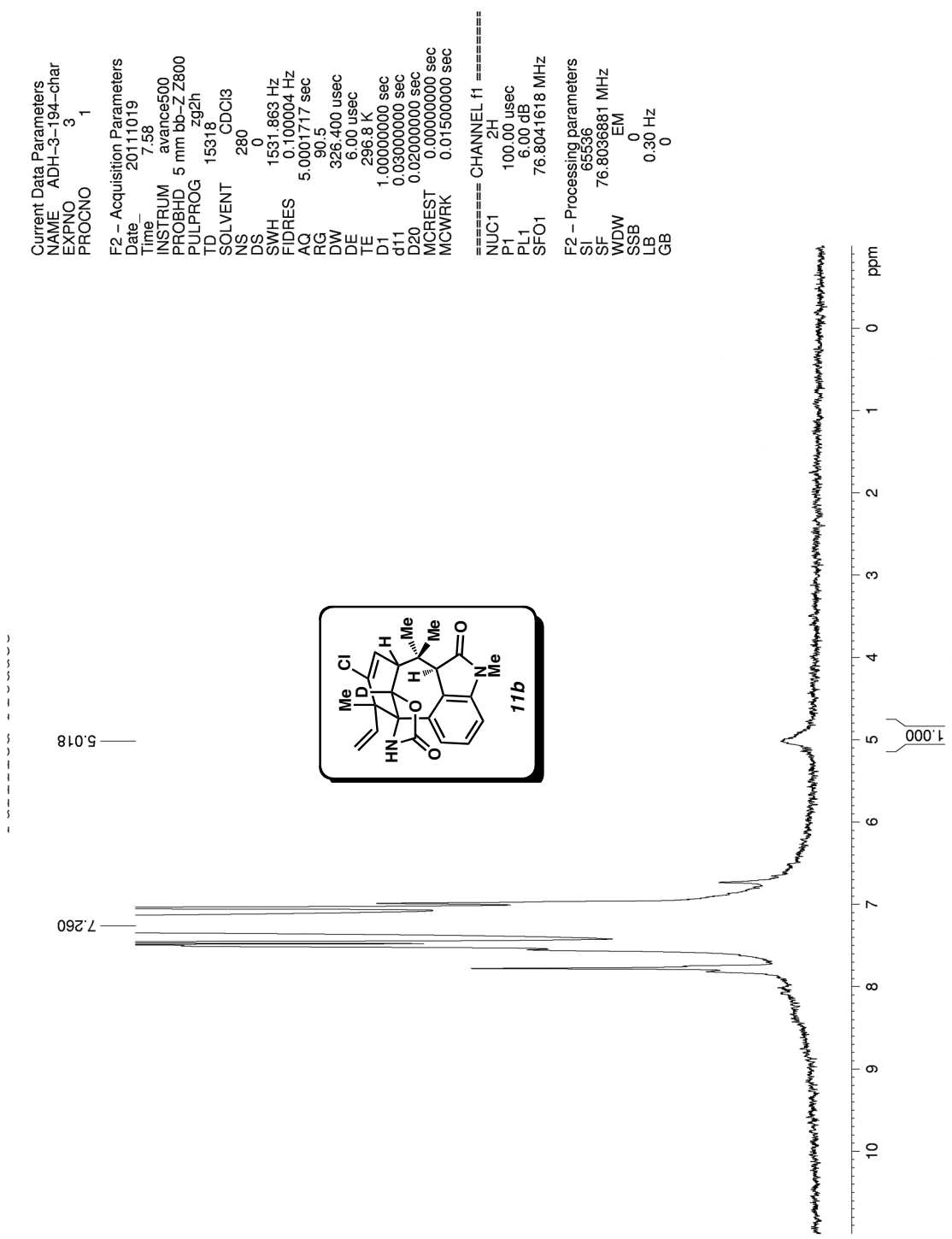
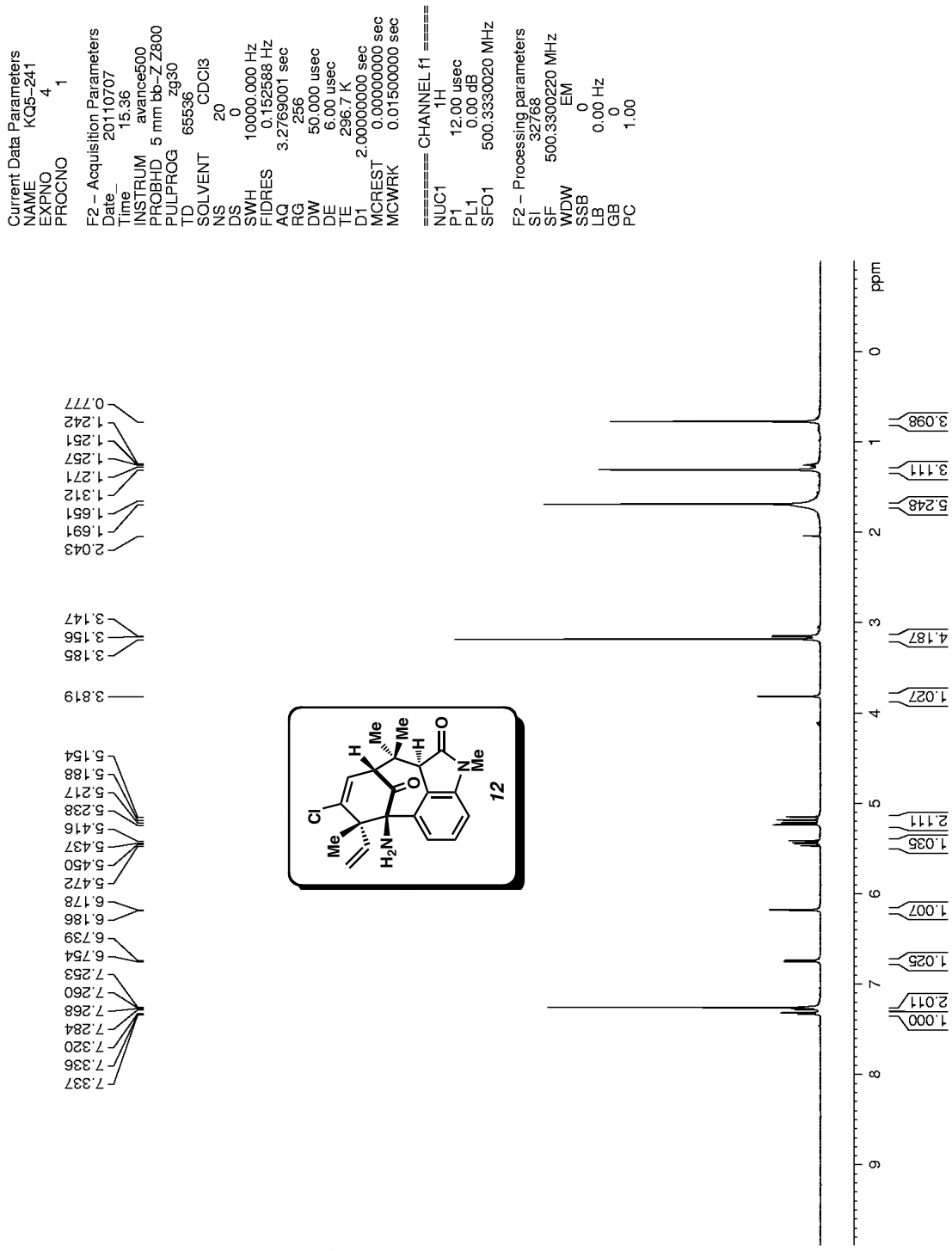
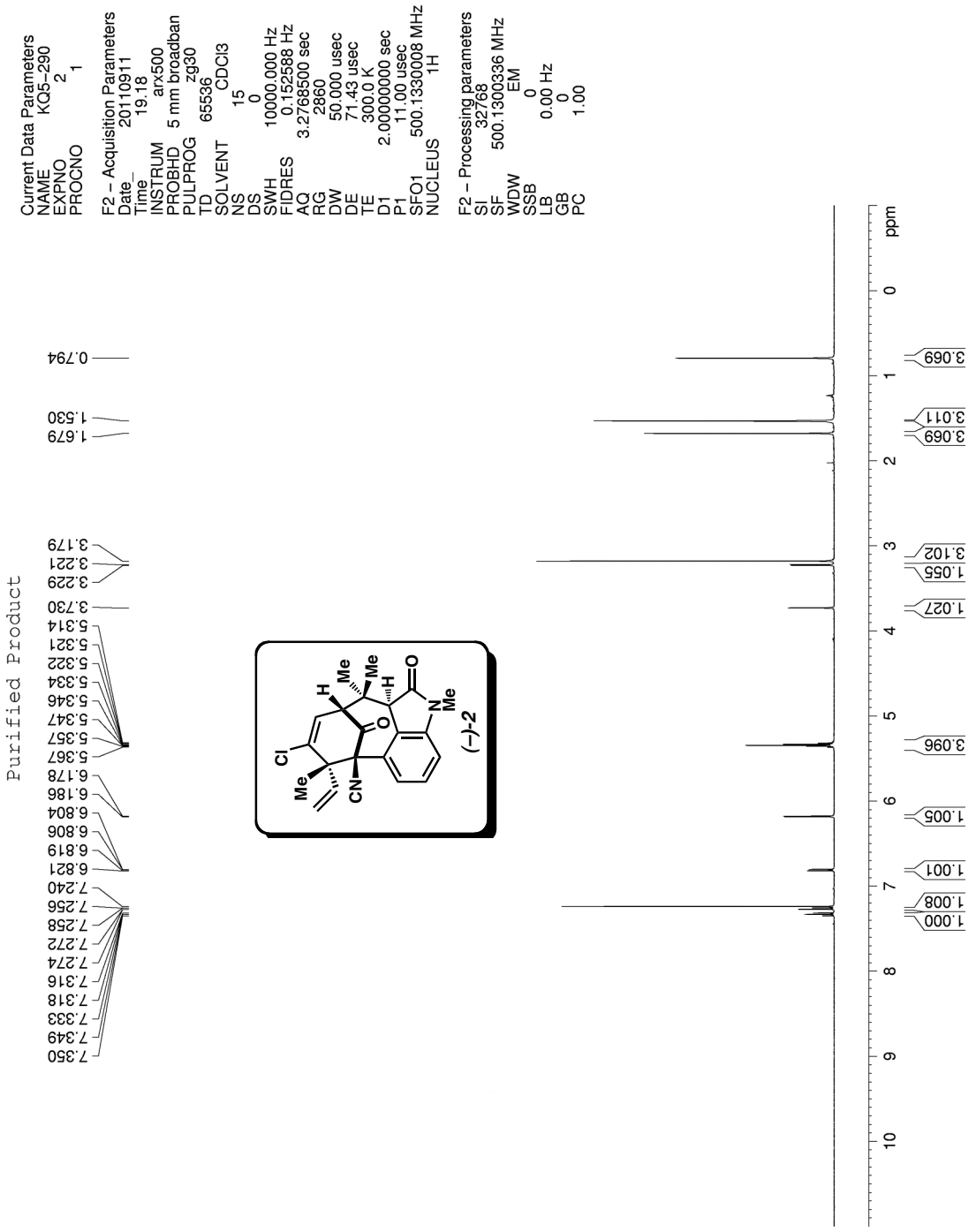
0.952


Current Data Parameters
NAME ADH–3–194–char
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111019
Time 7.58
INSTRUM avance500
PROBHD 5 mm bb–Z 2800
PULPROG zg2h
TD 15318
SOLVENT CDCl3
NS 280
DS 0
SWH 1531.863 Hz
FIDRES 0.100004 Hz
AQ 5.0001717 sec
RG 90.5
DW 326.400 usec
DE 6.00 usec
TE 296.8 K
D1 1.00000000 sec
d11 0.03000000 sec
D20 0.02000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 2H
P1 100.00 usec
PL1 6.00 dB
SFO1 76.8041618 MHz
F2 – Processing parameters
SI 65536
SF 76.8036881 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
5.018
7.260
1.000
11b
ppm


Current Data Parameters
NAME KQ5-241
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date 20110707
Time 15.36
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 256
DW 50.000 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
12
ppm


Purified Product
Current Data Parameters
NAME KQ5-290
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110911
Time 19.18
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 2860
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300336 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0.794
1.530
1.679
3.179
3.221
3.229
3.730
5.314
5.321
5.322
5.334
5.346
5.347
5.357
5.367
6.178
6.186
6.804
6.806
6.819
6.821
7.240
7.256
7.258
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7.318
7.333
7.349
7.350
Me
H
Me
H
O
O
N
Me
Cl
Me
CN
(–)-2
ppm
0
1
2
3
4
5
6
7
8
9
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3.069
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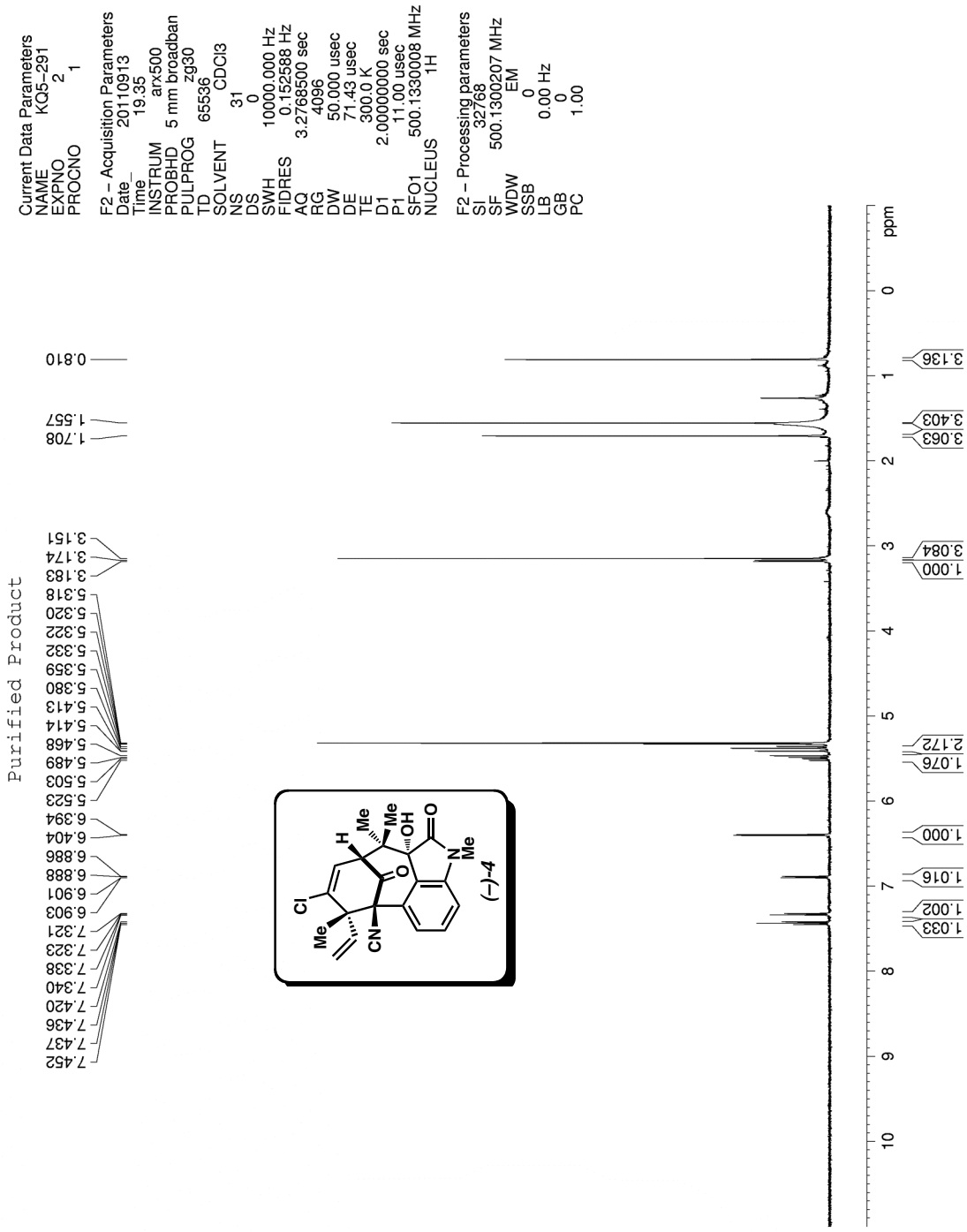
1.055
1.027
3.096
1.005
1.001
1.008
1.000


Purified Product
Current Data Parameters
NAME KQ5-291
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110913
Time 19.35
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 31
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 4096
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300207 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0.810
1.557
1.708
3.151
3.174
3.183
5.318
5.320
5.322
5.332
5.359
5.380
5.413
5.414
5.468
5.489
5.503
5.523
6.394
6.404
6.886
6.888
6.901
6.903
7.321
7.323
7.338
7.340
7.420
7.436
7.437
7.452
Me
Me
OH
H
O
N
Me
O
Cl
Me
CN
(–)-4
ppm
0
1
2
3
4
5
6
7
8
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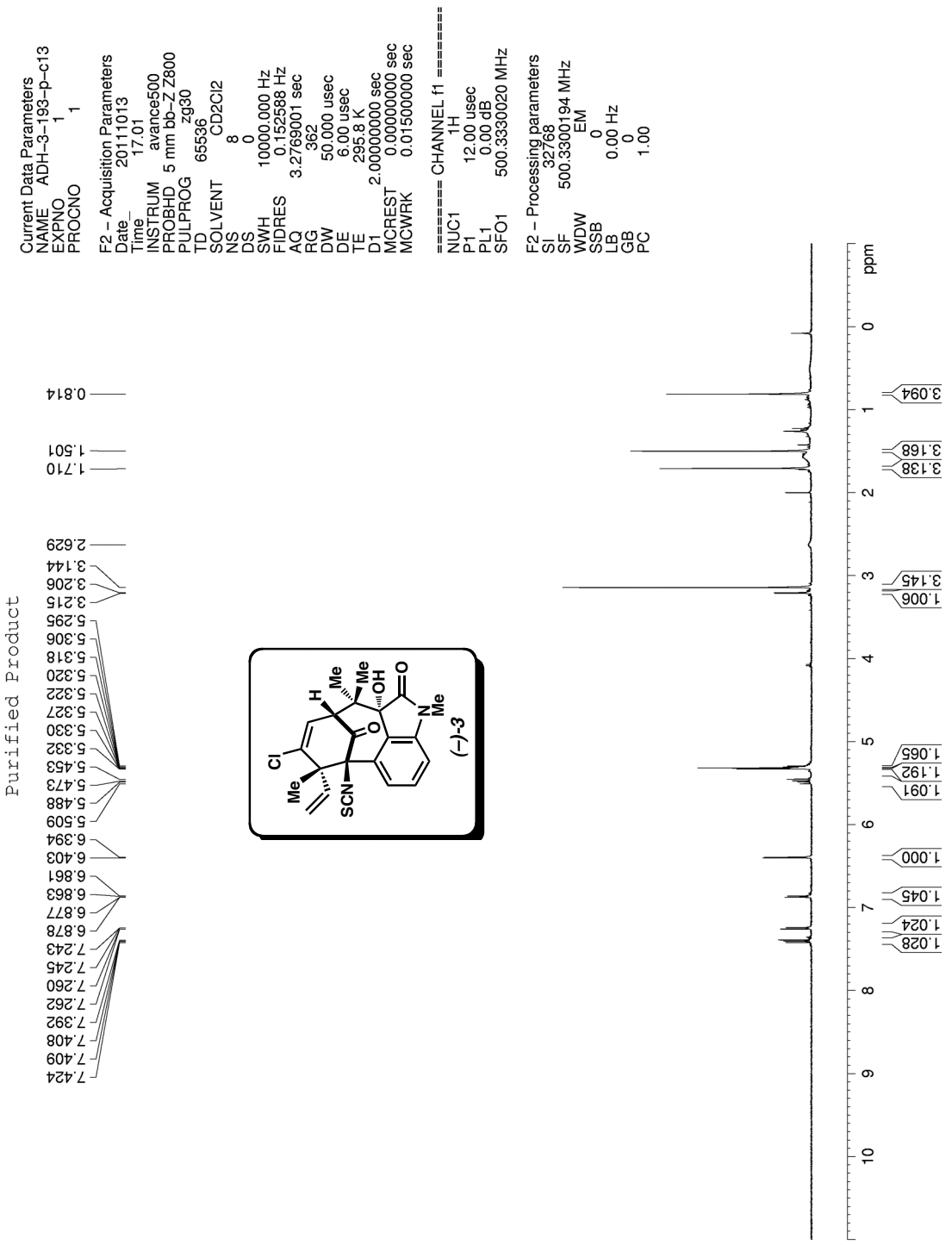
1.033


Purified Product
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5.306
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5.320
5.322
5.327
5.330
5.332
5.453
5.473
5.488
5.509
6.394
6.403
6.861
6.863
6.877
6.878
7.243
7.245
7.260
7.262
7.392
7.408
7.409
7.424
Cl
Me
SCN
H
Me
Me
OH
O
O
N
Me
(–)-3
Current Data Parameters
NAME ADH–3–193–p–c13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111013
Time 17.01
INSTRUM avance500
PROBHD 5 mm bb–Z 2800
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 362
DW 50.000 usec
DE 6.00 usec
TE 295.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300194 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
3.094
3.138
3.168
1.006
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1.065
1.000
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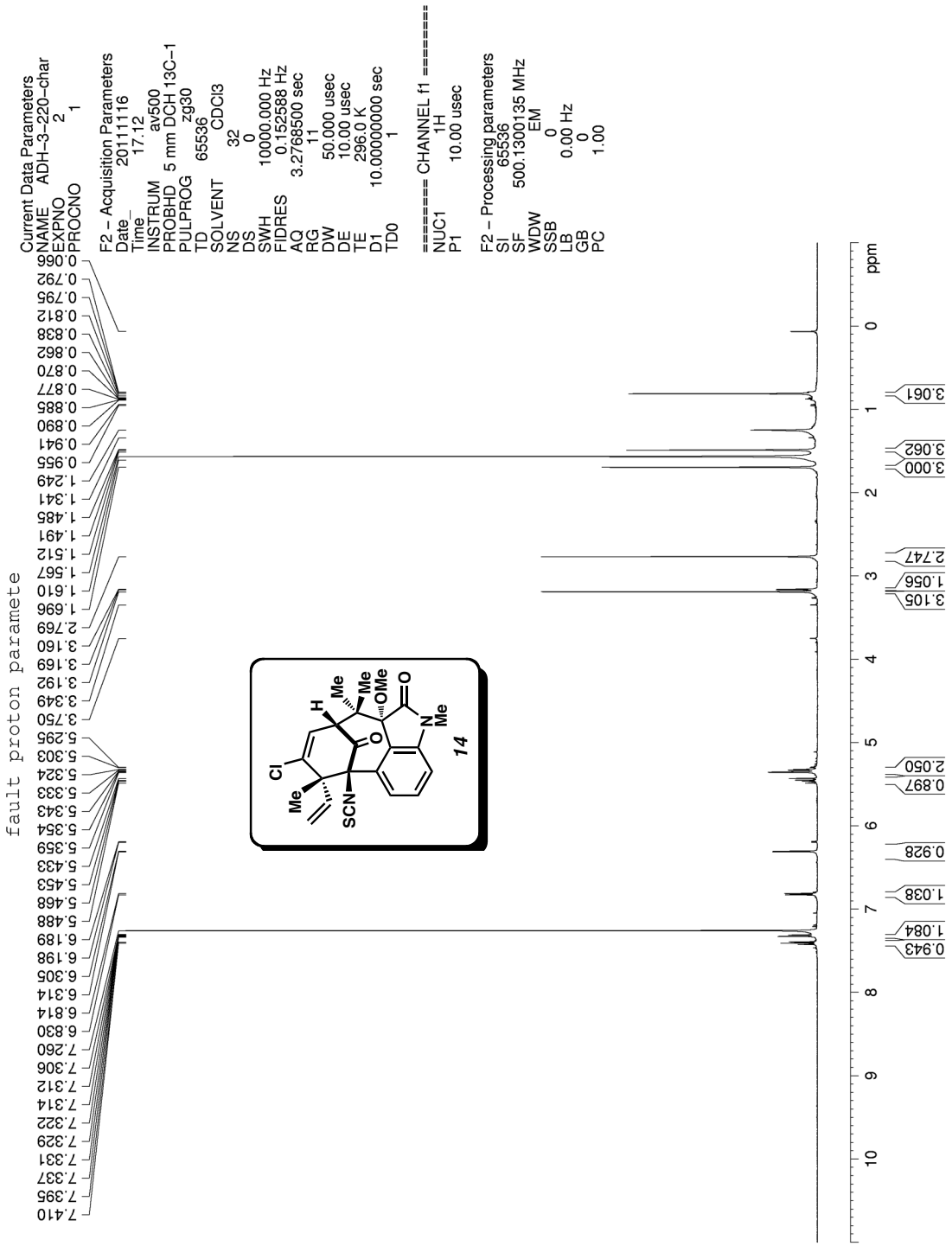
1.028
ppm


fault proton paramete
Current Data Parameters
NAME ADH–3–220–char
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111116
Time 17.12
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
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 296.0 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
F2 – Processing parameters
SI 65536
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.410
7.395
7.337
7.331
7.329
7.322
7.314
7.312
7.306
7.260
6.830
6.814
6.314
6.305
6.198
6.189
5.488
5.468
5.453
5.433
5.359
5.354
5.343
5.333
5.324
5.303
5.295
3.750
3.349
3.192
3.169
3.160
2.769
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1.567
1.512
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1.485
1.341
1.249
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0.890
0.885
0.877
0.870
0.862
0.838
0.812
0.795
0.792
0.990
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Me
Me
OMe
O
N
Me
O
Cl
Me
SCN
14
ppm
0
1
2
3
4
5
6
7
8
9
10
3.061
3.062
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3.105
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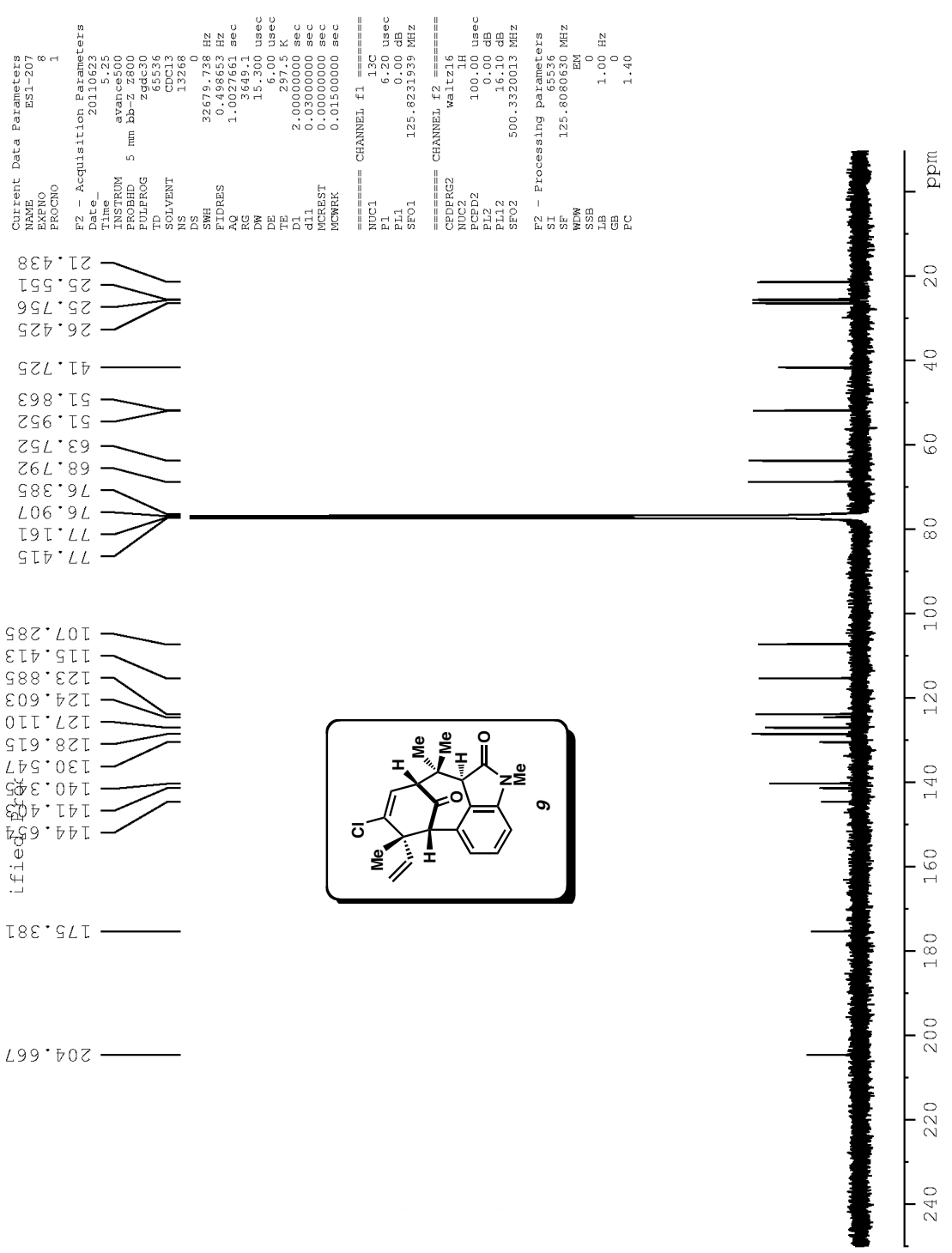
1.084
0.943

^{13}C NMR Spectra: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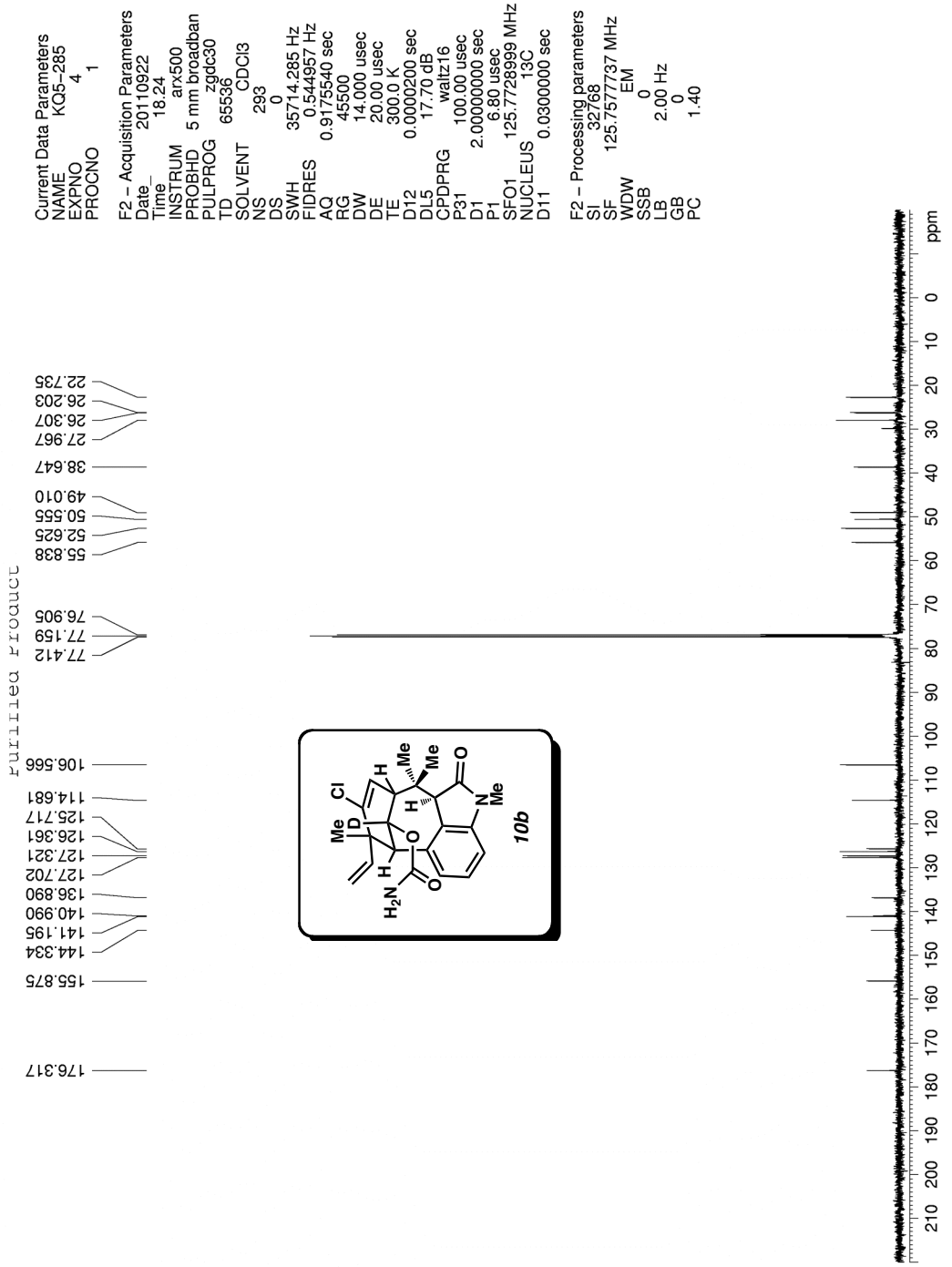
NAME ES1-207
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110623
Time 5.25
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 13268
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3649.1
DW 15.300 usec
DE 6.00 usec
TE 297.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080630 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.438
25.551
25.756
26.425
41.725
51.863
51.952
63.752
68.792
76.385
76.907
77.161
77.415
107.285
115.413
123.885
124.603
127.110
128.615
130.547
140.363
141.463
144.663
175.381
204.667
Me
Me
H
O
O
N
Me
Cl
Me
H
g
ppm
20
40
60
80
100
120
140
160
180
200
220
240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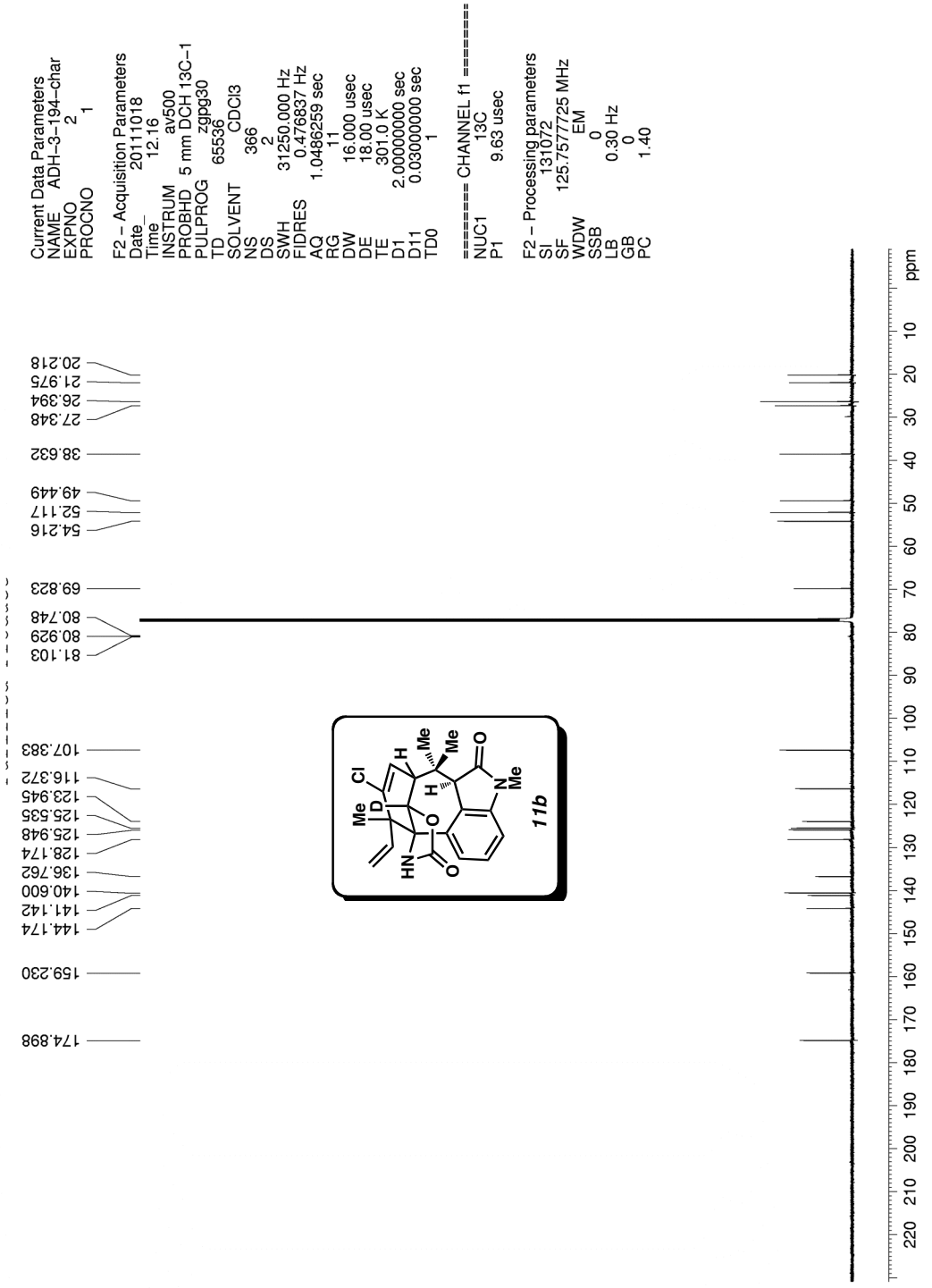
NAME KQ5–285
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110922
Time 18.24
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 293
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577737 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
Purified Product
176.317
155.875
144.334
141.195
140.990
136.890
127.702
127.321
126.361
125.717
114.681
106.566
77.412
77.159
76.905
55.838
52.625
50.555
49.010
38.647
27.967
26.307
26.203
22.735
10b
ppm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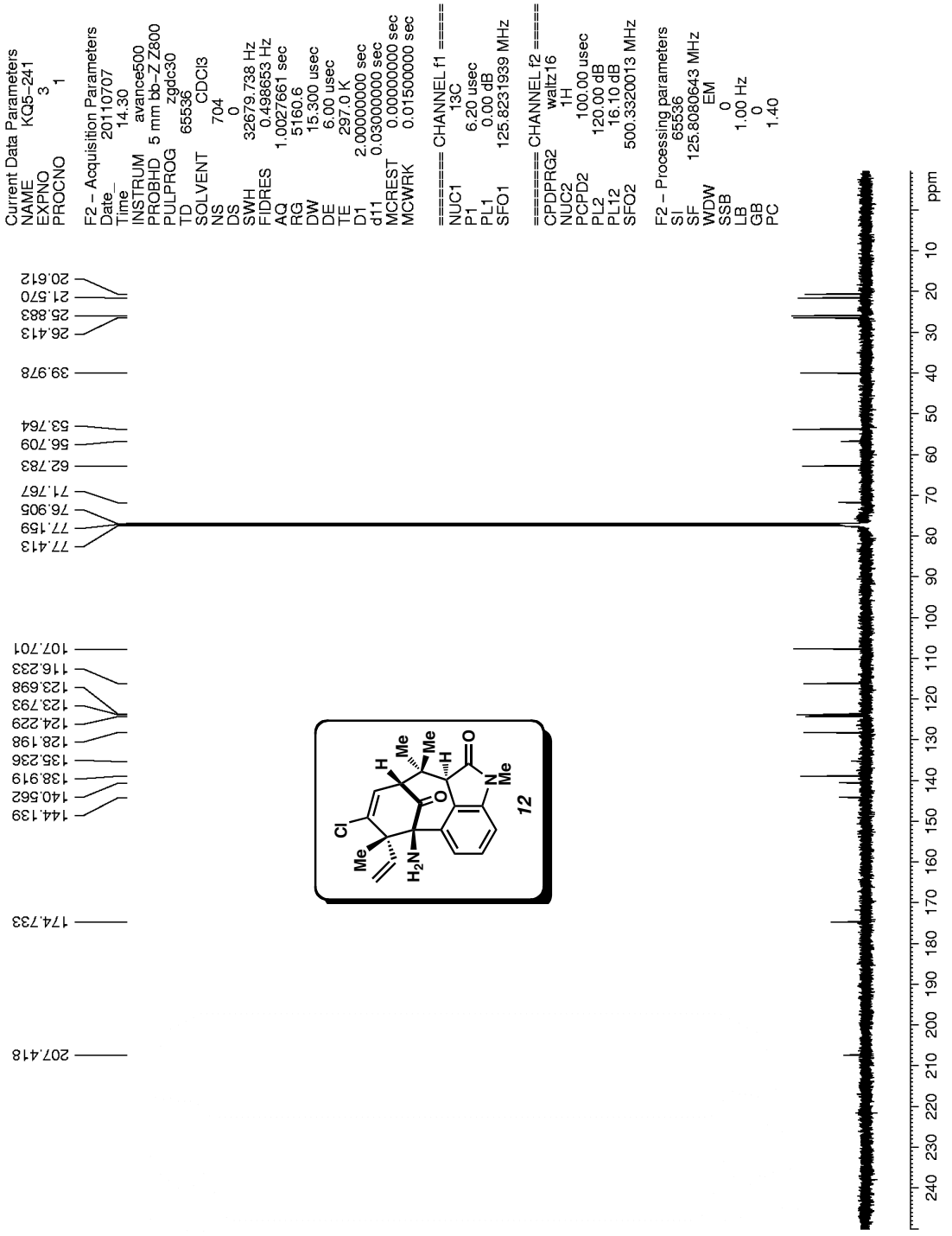
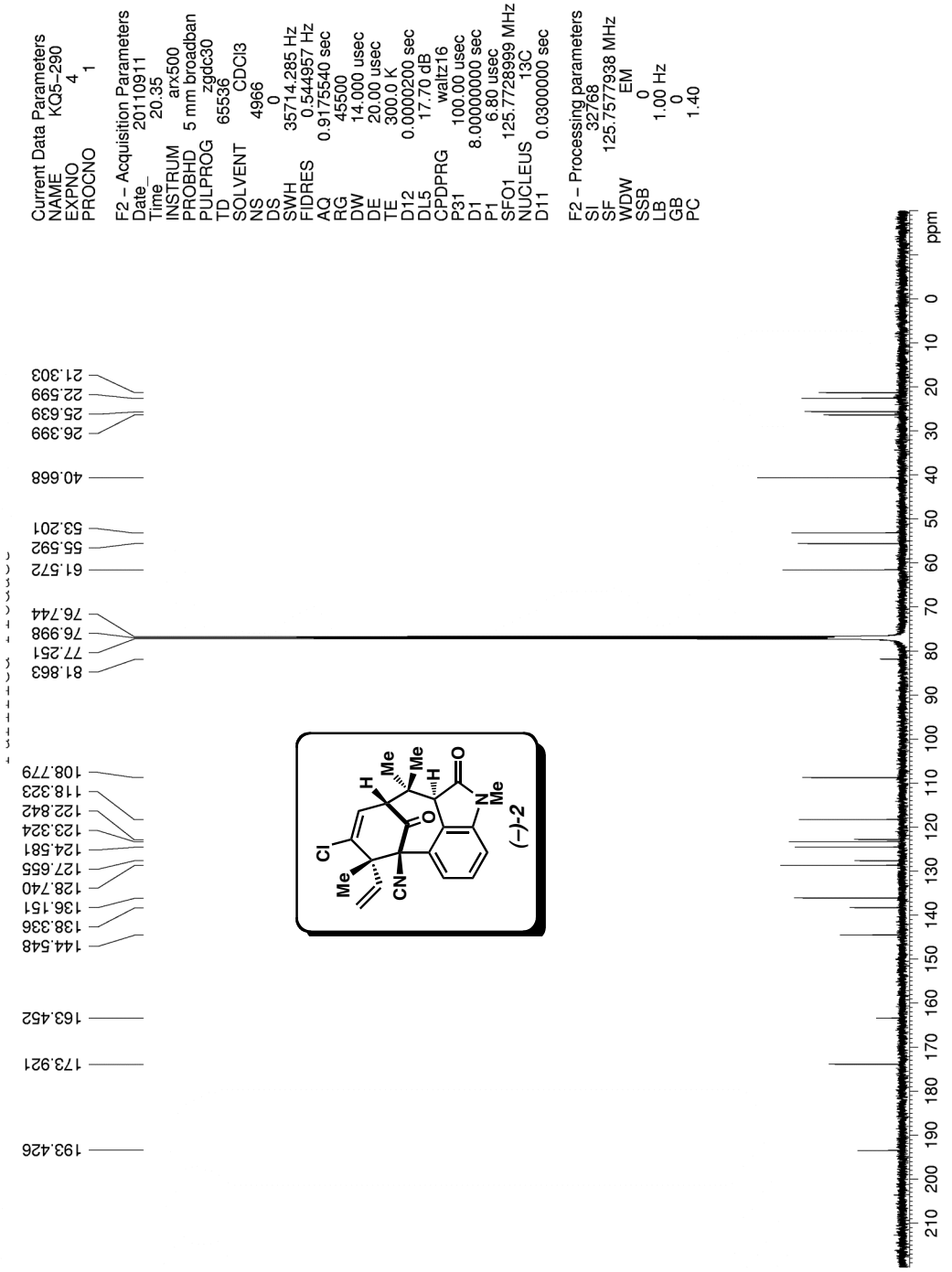
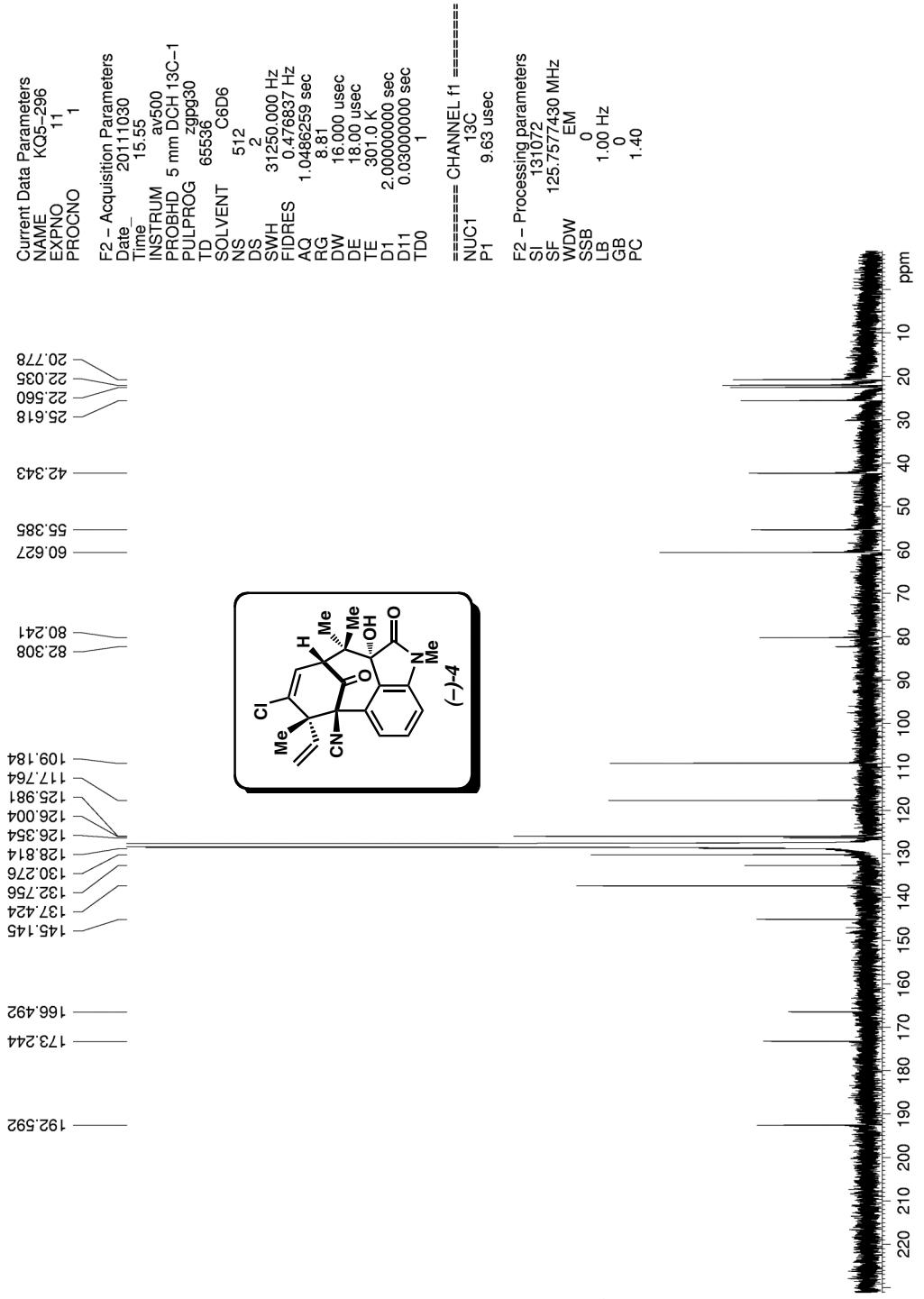
NAME ADH–3–194–char
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111018
Time 12.16
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 366
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 11
DW 16.000 usec
DE 18.00 usec
TE 301.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.63 usec
F2 – Processing parameters
SI 131072
SF 125.7577725 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
20.218
21.975
26.394
27.348
38.632
49.449
52.117
54.216
69.823
80.748
80.929
81.103
107.383
116.372
123.945
125.535
125.948
128.174
136.762
140.600
141.142
144.174
159.230
174.898
Me
Cl
D
H
Me
Me
O
H
N
Me
HN
O
O
11b
ppm


Current Data Parameters
NAME KQ5-241
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110707
Time 14.30
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 704
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 5160.6
DW 15.300 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080643 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.612
21.570
25.883
26.413
39.978
53.764
56.709
62.783
71.767
76.905
77.159
77.413
107.701
116.233
123.698
123.793
124.229
128.198
135.236
138.919
140.562
144.139
174.733
207.418
ppm
12


Current Data Parameters
NAME KQ5–290
EXPNO 4
PROCNO 1
F2 – Acquisition Parameters
Date_ 20110911
Time 20.35
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 4966
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 8.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577938 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.303
22.599
25.639
26.399
40.668
53.201
55.592
61.572
76.744
76.998
77.251
81.863
108.779
118.323
122.842
123.324
124.581
127.655
128.740
136.151
138.336
144.548
163.452
173.921
193.426
ppm
(–)-2


Current Data Parameters
NAME KQ5–296
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111030
Time 15.55
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 512
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 8.81
DW 16.000 usec
DE 18.00 usec
TE 301.0 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 125.7577430 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.778
22.035
22.560
25.618
42.343
55.385
60.627
80.241
82.308
109.184
117.764
125.981
126.004
126.354
128.814
130.276
132.756
137.424
145.145
166.492
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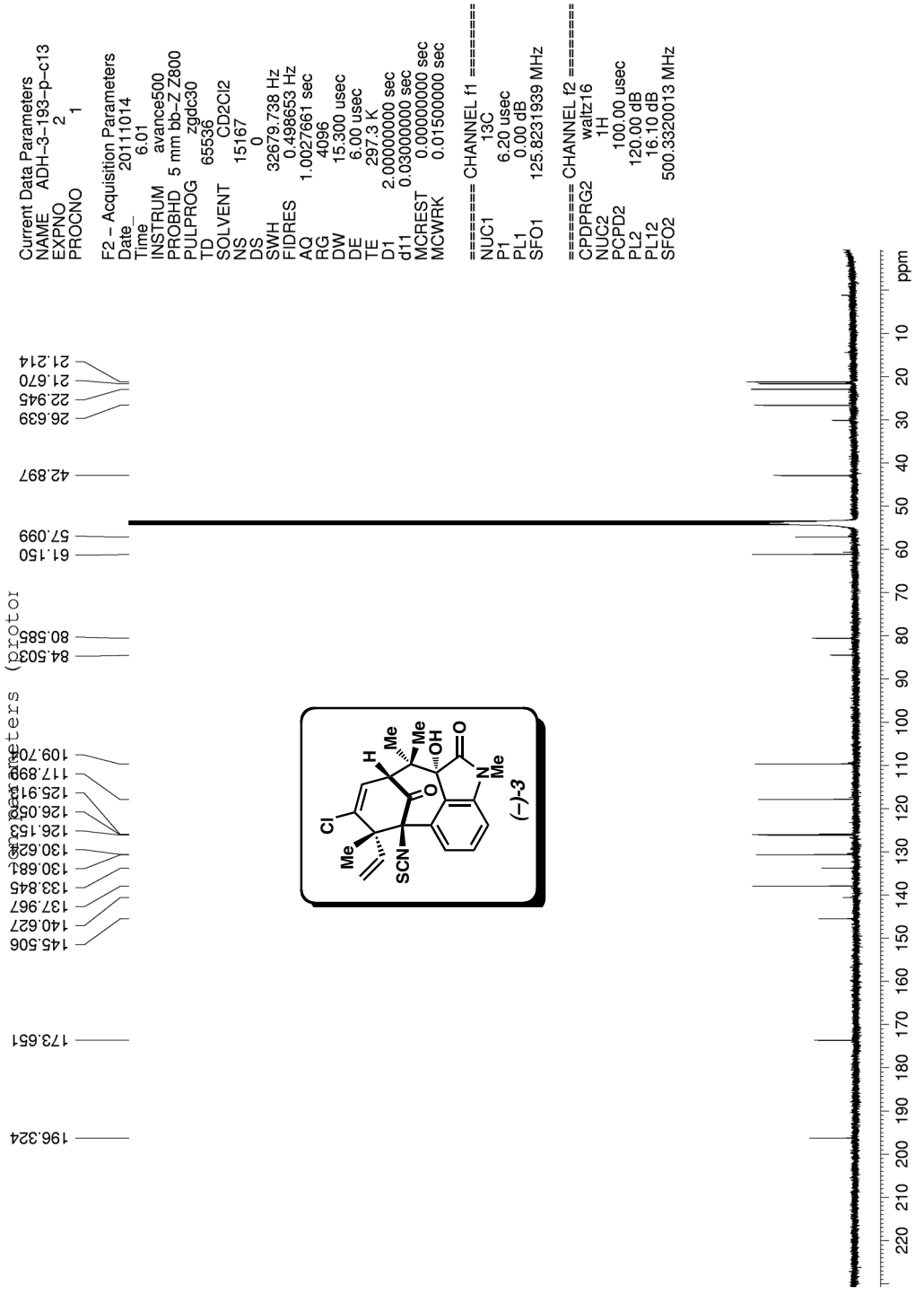
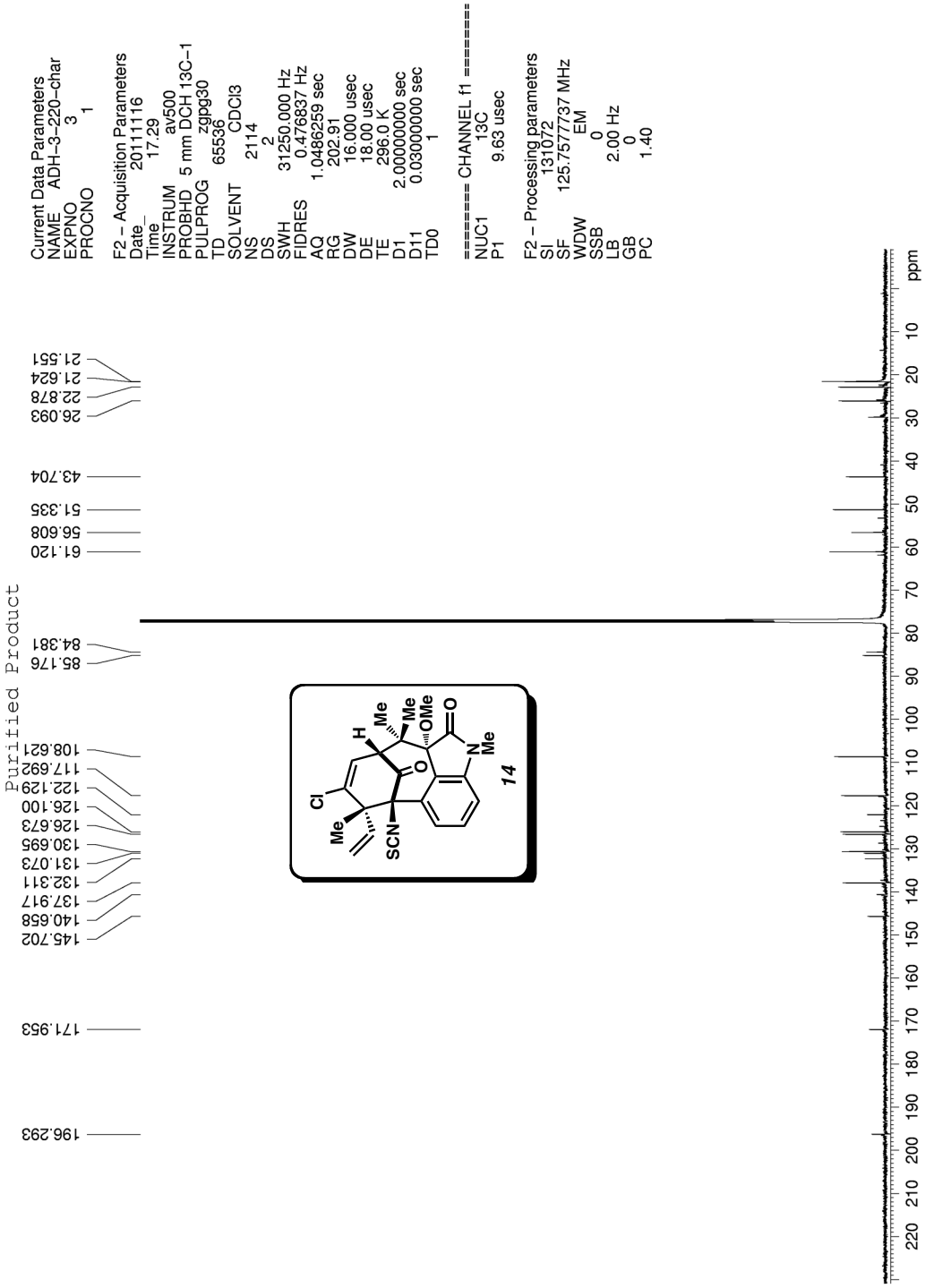
192.592
Me
H
Me
OH
O
N
Me
Cl
O
Me
CN
(–)-4
ppm


Current Data Parameters
NAME ADH-3-193-p-c13
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111014
Time 6.01
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CD2Cl2
NS 15167
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
196.324
173.651
145.506
140.627
137.967
133.845
130.681
130.628
126.152
126.050
125.914
117.899
109.708
84.503
80.585
61.150
57.099
42.897
26.639
22.945
21.670
21.214
ppm
(–)-3


Purified Product
Current Data Parameters
NAME ADH–3–220–char
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20111116
Time 17.29
INSTRUM av500
PROBHD 5 mm DCH 13C–1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2114
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 296.0 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 125.7577737 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
14
ppm

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[5] Reported values for specific rotations can be highly variable; for a pertinent discussion, see: Gawley, R. E. *J. Org. Chem.* **2006**, *71*, 2411–2416.

[6] NMR data for synthetic **3** did not match the tabulated data provided in the original isolation report (see reference 4). However, an authentic sample of **3** was recently located at the University of Hawaii, and subsequent NMR analysis revealed that the NMR data for **3** reported upon isolation was mis-tabulated. Indeed, synthetic **3** matched natural **3** by all spectroscopic means. We thank Philip Williams and Wesley Yoshida (University of Hawaii) for resolving this discrepancy and providing the correct NMR data for natural **3**.

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