Total Synthesis of (+)-Condylocarpine, (+)-Isocondylocarpine, and (+)-Tubotaiwine

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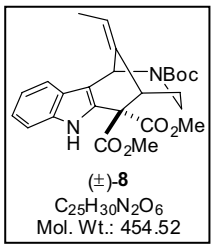
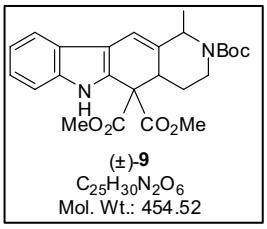
Materials and Methods. Reactions were performed in oven-dried glassware under a positive pressure of nitrogen or argon. Dichloromethane, methanol, toluene, and DMSO were dried by passage through columns packed with activated alumina or molecular sieves. Tetrahydrofuran was dried and deoxygenated by distillation from sodium/benzophenone ketyl. Thin layer chromatography was performed on Silicycle 60 Å F-254 pre-coated silica gel plates. TLC plates were visualized by exposure to UV light (254 nm) or a combination of *p*-anisaldehyde or ceric ammonium sulfate (CAS) staining. Flash column chromatography was performed using normal-phase silica gel (60 Å, 230–400 mesh, Silicycle SiliaFlash P60) or reverse-phase C18 silica gel (125 Å, 55–105 mesh, Waters). Neutralized silica gel was prepared by adding 5 wt % of pH 7 phosphate buffer solution to normal-phase silica gel and mixing by rotation in a round-bottom flask overnight. Normal-phase HPLC was performed using a 250 mm x 22 mm Alltima Silica (5μm) column. NMR spectra were recorded with Brucker Advanced spectrometers at 500 MHz or 600 MHz as indicated. ^{13}C spectra were recorded at 125 MHz and hydrogen multiplicities were determined by HMQC correlation. Chemical shifts are reported in ppm relative to peaks corresponding to residual protons of the deuterated solvents unless otherwise indicated. Optical rotations were recorded with a Jasco P-1010 polarimeter. Infrared spectra were recorded using ASI ReactIR 1000 or Varian 800-IR spectrometers. High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility. Abbreviations used can be found on the Internet at http://pubs.acs.org/paragonplus/submission/joceah/joceah_abbreviations.pdf.



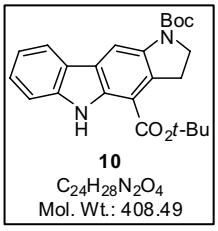
(±)-**8**
$C_{25}H_{30}N_2O_6$
Mol. Wt.: 454.52

Tetracyclic Ethylidene (±)-8. A solution of potassium *tert*-butoxide (1.04 g, 9.23 mmol) in 10 mL of THF was added by cannula transfer to a 0 °C suspension of ethyltriphenylphosphonium bromide (3.64 g, 9.81 mmol, azeotropically dried with toluene) and 40 mL of toluene. The resulting red suspension was allowed to warm to room temperature and stirred for 2 h. A solution of tetracyclic ketone (±)-**7a** (0.506 g, 1.14 mmol) in 5 mL of THF was then added by cannula transfer, and the residue in the transfer vessel was washed into the reaction mixture with an additional 2 mL of THF. The resulting mixture was stirred for 45 min at room temperature, before it was diluted with 20 mL of a saturated aqueous solution of NH_4Cl and 20 mL of EtOAc. The phases were separated and the aqueous phase was extracted with EtOAc (2 x 20 mL). The combined organic layers were washed with brine (1 x 10 mL), dried with $MgSO_4$, and concentrated in vacuo to afford 2.8 g of an orange oil. This residue was purified by flash silica gel chromatography (15%→25% EtOAc in hexanes) to afford 0.38 g (73%) of (±)-**8** as a tan foam: ^{1}H NMR (600 MHz, $CDCl_3$, mixture of rotamers) δ 8.58 (s, 1H of major rotamer), 8.53 (s, 1H of minor rotamer), 7.84 (d, *J* = 7.9 Hz, 1H of minor rotamer), 7.65 (d, *J* = 8.0 Hz, 1H of major rotamer), 7.41 (d, *J* = 8.2 Hz, 1H of major rotamer), 7.37 (d, *J* = 8.0 Hz, 1H of minor rotamer), 7.25–7.21 (m, 1H of both rotamers), 7.17–7.12 (m, 1H of both rotamers), 6.42 (s, 1H of minor rotamer), 6.28 (s, 1H of major rotamer), 5.62–5.56 (m, 1H of both rotamers), 3.85 (s, 3H of both rotamers), 3.81 (dd, *J* = 13.9, 5.7 Hz, 1H of major rotamer), 3.72 (s, 3H of both rotamers), 3.61–3.56 (m, 1H of minor rotamer), 3.57 (d, *J* = 3.6 Hz, 1H of both rotamers), 2.84 (app t, *J* =13.8 Hz, 1H of minor rotamer), 2.78 (app t, *J* = 13.5, 1H of major rotamer), 2.04–1.94 (m, 1H of both rotamers), 1.83–1.80 (m, 3H of both rotamers), 1.63 (s, 9H of major rotamer), 1.63–1.56 (m, 1H of both rotamers), 1.42 (s, 9H of minor rotamer); ^{13}C NMR (125 MHz, $CDCl_3$, mixture of rotamers) δ 170.0 (C), 168.8 (C), 168.1 (C), 168.0 (C), 154.7 (C), 153.9 (C), 136.9 (C), 134.8 (C), 134.3 (C), 130.0 (C), 129.7 (C), 124.8 (C), 124.7 (C), 123.1 (CH), 123.1 (CH), 120.4 (CH), 120.1 (CH), 120.0 (CH), 119.8 (CH), 119.2 (CH), 119.1 (CH), 113.5 (C), 112.7 (C), 111.6 (CH), 111.2 (CH), 79.9 (C), 79.5 (C), 60.5 (C), 60.3 (C), 53.33 (CH_3), 53.26 (CH_3), 44.6 (CH), 43.5 (CH), 42.7 (CH), 42.6 (CH), 37.5 (CH_2), 36.1 (CH_2), 30.7 (CH_2), 30.6 (CH_2), 28.9 (CH_3), 28.6 (CH_3), 13.6 (CH_3), 13.4 (CH_3); IR (thin film) 3204, 1737, 1671, 1457, 1414, 1250, 1227, 1150, 909 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{25}H_{30}N_2O_6Na$ $(M+Na)^+$ 477.2002, found 477.2001.



(±)-**9**
$C_{25}H_{30}N_2O_6$
Mol. Wt.: 454.52

2-*tert*-Butyl 5,5-dimethyl 1-methyl-4,4a-dihydro-1*H*-pyrido[4,3-*b*]carbazole-2,5,5(3*H*,6*H*)-tricarboxylate ((±)-9). A solution of ethylidene (±)-**8** (40 mg, 0.088 mmol), AcCl (0.6 μL, 8 μmol), and CD_3OD (0.5 μL, 10 μmol) in 2.3 mL of $CDCl_3$ was maintained at room temperature and monitored by ^{1}H NMR.[1] After 1.5 h, the solution was concentrated in vacuo to afford 42 mg of an orange oil. This residue was purified by flash silica gel chromatography (20→25% EtOAc in hexanes) to afford 32 mg (80%) of (±)-**9** as a colorless foam: ^{1}H NMR (500 MHz, DMSO-d_6, 70 °C) δ 11.1 (s, 1H), 7.59 (d, J = 7.9 Hz, 1H), 7.43 (d, J = 8.2 Hz, 1H), 7.11 (t, J = 7.6 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 6.60 (app s, 1H), 4.74 (q, J = 6.3 Hz, 1H), 3.74 (s, 3H), 3.71 (s, 3H), 3.47 (dt, J = 13.3, 7.0 Hz, 1H), 3.29–3.20 (m, 2H), 1.98–1.93 (m, 1H), 1.85–1.79 (m, 1H), 1.46 (s, 9H), 1.37 (d, J = 6.6 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, 70 °C) δ 168.4 (C), 167.5 (C), 154.0 (C), 136.6 (C), 131.5 (C), 126.9 (C), 123.3 (C), 121.3 (CH), 119.0 (CH), 117.6 (CH), 113.5 (CH), 111.7 (CH), 110.8 (C), 78.3 (C), 58.7 (C), 52.8 (CH_3), 52.7 (CH), 52.1 (CH_3), 40.6 (CH), 38.9 (CH_2), 27.8 (CH_3), 23.2 (CH_2), 19.7 (CH_3); IR (thin film) 3396, 3321, 1737, 1675, 1412, 1258, 1169 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{25}H_{30}N_2O_6Na$ $(M+Na)^+$ 477.2002, found 477.1982.

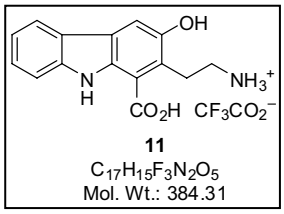


10
$C_{24}H_{28}N_2O_4$
Mol. Wt.: 408.49

Di-*tert*-butyl 2,3-dihydropyrrolo[3,2-b]carbazole-1,4(5H)-dicarboxylate (10). A solution of (±)-**7b** (40 mg, 0.076 mmol), AcCl (6 μL, 0.08 mmol), and CD_3OD (7 μL, 0.2 mmol) in 0.50 mL of $CDCl_3$ was maintained at room temperature and monitored by ^{1}H NMR. After 2 h,[2] the solution was concentrated in vacuo to afford 44 mg of a brown oil. This residue was purified by flash silica gel chromatography (5%→25% EtOAc in hexanes) to afford 17 mg (55%) of **10** as a pale yellow amorphous solid: ^{1}H NMR (500 MHz, $CDCl_3$, mixture of rotamers) δ 9.92 (s, 1H of both rotamers), 8.84 (br s, 1H of major rotamer), 8.40 (br s, 1H of minor rotamer), 8.08 (br s, 1H of major rotamer), 7.99 (br s, 1H of minor rotamer), 7.45–7.39 (m, 2H of both rotamers), 7.22 (t, J = 7.2 Hz, 1H of both rotamers), 4.09 (br s, 2H of both rotamers), 3.56 (br s, 2H of both rotamers), 1.70 (s, 9H of both rotamers), 1.62 (br s, 9H of both rotamers); ^{13}C NMR (125 MHz, $CDCl_3$, only the major rotamer is reported) δ 167.1 (C), 153.0 (C), 140.0 (C),

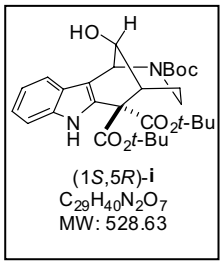
[1] After 1 h, the ratio of **9**:**8** was 11:1, and only ~10 mol % of the AcCl had undergone methanolysis.
[2] This reaction was not run to full conversion of **7b**, and 10 mg (25%) of this starting material was recovered after column chromatography.

137.0 (C), 132.9 (C), 126.0 (CH), 123.9 (C), 122.9 (C), 120.7 (CH), 119.6 (CH), 111.2 (CH), 111.1 (CH), 82.3 (C), 80.5 (C), 48.3 (CH_2), 30.1 (CH_2), 28.8 (CH_3), 28.7 (CH_3); IR (thin film) 3444, 2975, 1684, 1453, 1392, 1330, 1142 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{24}H_{28}N_2O_4Na$ $(M+Na)^+$ 431.1947, found 431.1932.



11
$C_{17}H_{15}F_3N_2O_5$
Mol. Wt.: 384.31

2-(1-Carboxy-3-hydroxy-9*H*-carbazol-2-yl)ethanaminium trifluoroacetate (11). A solution of ketone (±)-**7b** (0.140 g, 0.265 mmol) in 5 mL of TFA was maintained for 4 h at room temperature. This solution was then concentrated in vacuo and the residue was purified by reverse phase C18 chromatography (0%→20%→40% MeOH in H_2O with 1% TFA) to afford 0.064 g (63%) of **11** as a yellow foam: ^{1}H NMR (500 MHz, CD_3OD) δ 7.97 (d, *J* = 8.0 Hz, 1H), 7.76 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.37 (app t, *J* = 8.0 Hz, 1H), 7.15 (app t, *J* = 8.0 Hz, 1H), 3.53 (d, *J* = 7.2 Hz, 1H), 3.31 (d, *J* = 7.2 Hz, 1H coincident with CD_2HOD peak); ^{13}C NMR (125 MHz, CD_3OD) δ 170.5 (C), 150.4 (C), 142.1 (C), 136.1 (C), 127.4 (CH), 125.4 (C), 125.3 (C), 123.3 (C), 120.9 (CH), 120.1 (CH), 114.8 (C), 112.5 (CH), 111.0 (CH), 41.4 (CH_2), 27.3 (CH_2); IR (thin film) 3455, 3036, 1668, 1489, 1437, 1185, 1140 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{15}H_{15}N_2O_3$ $(M+H–CF_3CO_2)^+$ 271.1083, found 271.1092.

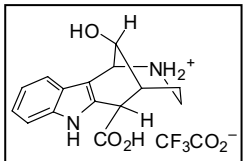


(1*S*,5*R*)-**i**
$C_{29}H_{40}N_2O_7$
MW: 528.63

Tetracyclic Alcohol (1*S*,5*R*)-i. Sodium borohydride (143 mg, 3.76 mmol) was added to a 0 °C solution of ketone (1*S*,5*R*)-**7b** (1.98 g, 3.76 mmol, 91% ee) in 40 mL of methanol and THF (1:1 v/v). This mixture was stirred for 10 min at 0 °C, before TFA (0.87 mL, 11.3 mmol) was added and the mixture allowed to warm to room temperature. The resulting solution was concentrated in vacuo to afford 3.3 g of crude (1*S*,5*R*)-**i** as a colorless oil, which was of sufficient purity to be used in the next step without further purification.

An analytical sample of (1*S*,5*R*)-**i** was prepared following the procedure described above with a modified quench and purification. Sodium borohydride (2 mg, 0.05 mmol) was added to a 0 °C solution of ketone (1*S*,5*R*)-**7b** (29 mg, 0.055 mmol, 91% ee) in 0.6 mL of methanol and THF (1:1 v/v). The mixture was stirred for 10 min at 0 °C, before it was quenched with acetic acid (3 drops) and allowed to warm to room temperature. The resulting solution was diluted with 2 mL of EtOAc and 1 mL of a saturated aqueous solution of $NaHCO_3$. The layers were separated

and the aqueous layer was extracted with EtOAc (2 x 1 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 31 mg of a colorless solid. This residue was purified by flash silica gel chromatography (10→20% EtOAc in hexanes) to afford 27 mg (93%) of (1*S*,5*R*)-**i** as a colorless foam: $[\alpha]^{24}_{589}$ + 69.6, $[\alpha]^{24}_{577}$ + 70.3, $[\alpha]^{24}_{546}$ + 79.7, $[\alpha]^{24}_{435}$ + 124.2, $[\alpha]^{24}_{405}$ + 140.1 (*c* 0.31, CH_2Cl_2, 91% ee); ^{1}H NMR (500 MHz, $CDCl_3$, mixture of rotamers) δ 8.39 (s, 1H of both rotamers), 7.79 (d, *J* = 7.9 Hz, 1H of minor rotamer), 7.68 (d, *J* = 8.0 Hz, 1H of major rotamer), 7.39 (d, *J* = 8.2 Hz, 1H of major rotamer), 7.37 (d, *J* = 8.4 Hz, 1H of minor rotamer), 7.23–7.19 (m, 1H of both rotamers), 7.14–7.09 (m, 1H of both rotamers), 5.71 (d, *J* = 2.9 Hz, 1H of minor rotamer), 5.58 (d, *J* = 3.1 Hz, 1H of major rotamer), 4.28 (d, *J* = 2.8 Hz, 1H of both rotamers), 3.84 (d, *J* = 2.1 Hz, 1H of major rotamer), 3.70 (dd, *J* = 13.3, 5.2 Hz, 1H of major rotamer), 3.53–3.48 (m, 1H of minor rotamer), 3.48 (s, 1H of minor rotamer), 3.23 (app s, 1H of both rotamers), 2.54–2.46 (m, 1H of both rotamers), 1.97–1.91 (m, 1H of both rotamers), 1.72 (app d, *J* = 14.3 Hz, 1H of both rotamers), 1.60 (s, 9H of major rotamer); 1.55 (s, 9H of both rotamers), 1.51 (s, 9H of both rotamers), 1.41 (s, 9H of minor rotamer); ^{13}C NMR (125 MHz, $CDCl_3$, mixture of rotamers) δ 170.3 (C), 169.9 (C), 168.3 (C), 168.1 (C), 154.8 (C), 154.4 (C), 136.9 (C), 128.3 (C), 128.1 (C), 126.2 (C), 122.9 (CH), 122.8 (CH), 120.6 (CH), 119.9 (CH), 119.8 (CH), 119.7 (CH), 111.2 (CH), 110.9 (CH), 110.3 (C), 110.0 (C), 84.9 (C), 84.5 (C), 83.6 (C), 83.4 (C), 80.2 (C), 79.7 (C), 71.1 (CH), 70.9 (CH), 59.0 (C), 58.9 (C), 48.8 (CH), 47.7 (CH), 38.0 (CH), 37.9 (CH), 36.2 (CH_2), 34.8 (CH_2), 28.8 (CH_3), 28.5 (CH_3), 28.09 (CH_3), 28.07 (CH_3), 27.7 (CH_2), 27.5 (CH_2); IR (thin film) 3390, 1706, 1675, 1368, 1287, 1254, 1159, 1141 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{29}H_{40}N_2O_7Na$ $(M+Na)^+$ 551.2733, found 551.2750.

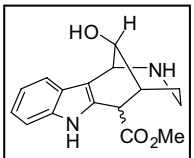


S1
$C_{17}H_{17}F_3N_2O_5$
Mol. Wt.: 386.32

Amino Acid S1. The crude tetracyclic alcohol (1*S*,5*R*)-**i** described in the preceding section was dissolved in 125 mL of TFA and water (4:1 v/v), and the resulting solution was maintained at room temperature for 13 h, before it was concentrated in vacuo to afford 3.0 g of **S1**[3] as a brown oil. This crude material was of sufficient purity to be use in the next step without further purification. Diagnostic data: ^{1}H NMR δ (600 MHz, CD_3OD) δ 7.59 (d, *J* = 7.9 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H), 7.15 (t, *J* = 7.3 Hz, 1H), 7.09 (t, *J* = 7.2 Hz, 1H), 4.94 (d, *J* = 3.1 Hz, 1H), 4.41

[3] Amino acid **S1** was formed as a single diastereomer whose relative configuration was not determined.

(t, *J* = 3.4 Hz, 1H), 4.36 (d, *J* = 5.9 Hz, 1H), 2.94 (dd, *J* = 12.9, 4.6 Hz, 1H), 2.86 (app s, 1H), 2.81 (td, *J* = 13.4, 4.1 Hz, 1H), 2.09–2.03 (m, 1H), 1.97 (app d, *J* = 15.4 Hz, 1H).

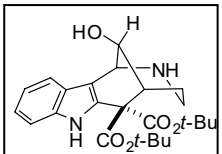


17
$C_{16}H_{18}N_2O_3$
Mol. Wt.: 286.33

Amino Ester 17. A 0.50 M solution of methanolic HCl was prepared by adding thionyl chloride (2.7 mL, 37 mmol) to 150 mL of methanol at 0 °C. This solution was added to the crude amino acid **S1** described in the preceding paragraph, and the resulting solution was maintained at 50 °C for 2.5 h. This solution was then concentrated in vacuo to afford a brown solid, to which a mixture of 20 mL of EtOAc and 15 mL of a saturated aqueous solution of $NaHCO_3$ was added. The mixture was stirred vigorously for 30 min and then filtered. The layers of the filtrate were separated and the aqueous layer was extracted with EtOAc (9 x 15 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 0.92 g (86% from (1*S*,5*R*)-**7b**) of **17** as a light brown foam, which was of sufficient purity to be used in the next step without further purification. Diagnostic data: NMR (500 MHz, CD_3OD, 2:1 mixture of ester epimers) δ 7.55 (d, *J* = 7.7 Hz, 1H of minor epimer), 7.54 (d, *J* = 7.8 Hz, 1H of major epimer), 7.35 (app t, *J* = 7.6 Hz, 1H of both epimers), 7.11–7.06 (m, 1H of both epimers), 7.03–6.99 (m, 1H of both epimers), 4.40 (d, *J* = 2.6 Hz, 1H of major epimer), 4.38 (d, *J* = 2.9 Hz, 1H of minor epimer), 4.23 (t, *J* = 3.6 Hz, 1H of major epimer), 4.17 (t, *J* = 3.6 Hz, 1H of minor epimer), 3.83 (s, 1H of minor epimer), 3.82 (s, 3H of major epimer), 3.76 (s, 1H of major epimer), 3.74 (s, 3H of minor epimer), 3.03–3.01 (m, 1H of minor epimer), 2.74–2.71 (m, 1H of major epimer), 2.54 (td, *J* = 13.1, 3.4 Hz, 1H of major epimer), 2.49–2.44 (m, 1H of both epimers), 2.30 (td, *J* = 13.3, 3.5 Hz, 1H of minor epimer), 2.00–1.92 (m, 1H of minor epimer), 1.86–1.78 (m, 1H of both epimers), 1.60 (app d, *J* = 14.4 Hz, 1H of major epimer).

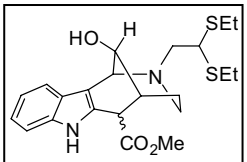
Reaction of (±)-i with a 0.15 M solution of DCl. A solution of (±)-**i** (40 mg, 0.076 mmol), AcCl (6 μL, 0.08 mmol), and CD_3OD (7 μL, 0.2 mmol) in 0.50 mL of $CDCl_3$ was maintained at room temperature for 2 h. Triethylamine (13 μL, 0.093 mmol) and 1,3,5-trimethoxybenzene (NMR internal standard, 93 μL of a 0.26 M solution in $CDCl_3$, 0.024 mmol) were then added, and the solution was analyzed by ^{1}H NMR. This analysis revealed that the Boc deprotection product, (±)-(±)-**ii,** was formed in 36% yield.[4]

[4] This analysis indicated that 48% of (±)-**i** remained unreacted.



(±)-**ii**
$C_{24}H_{32}N_2O_5$
Mol. Wt.: 428.52

Tetracyclic amino di-*tert*-butyl malonate (±)-ii. An analytical sample was obtained by repeating the above procedure without quenching with triethylamine, but instead washing the reaction solution with a saturated aqueous solution of $NaHCO_3$, concentrating the organic layer in vacuo, and purifying the residue by flash silica gel chromatography (6%→8% MeOH in CH_2Cl_2 with 1% aq NH_4OH): ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 4.47 (d, *J* = 2.3 Hz, 1H), 4.38 (t, *J* = 3.3 Hz, 1H), 3.57 (br s, 1H), 3.22 (app d, *J* = 3.1 Hz, 1H), 2.49–2.41 (m, 2H), 1.96–1.88 (m, 1H), 1.72–1.63 (m, 2H, coincident with signal arising from residual H_2O), 1.54 (s, 9H), 1.51 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.2 (C), 168.5 (C), 137.2 (C), 128.3 (C), 126.1 (C), 122.7 (CH), 119.7 (CH), 118.7 (CH), 111.3 (CH), 111.2 (C), 84.5 (C), 83.3 (C), 72.0 (CH), 59.5 (C), 50.3 (CH), 38.8 (CH), 36.5 (CH_2), 28.7 (CH_2), 28.1 (CH_3), 28.1 (CH_3); IR (thin film) 3487, 1730, 1710, 1457, 1369, 1278, 1255, 1157, 1142 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{24}H_{33}N_2O_5$ $(M+H)^+$ 429.2390, found 429.2378.



18
$C_{22}H_{30}N_2O_3S_2$
Mol. Wt.: 434.62

Tertiary Amine 18. A sample of the crude amino ester **17** (0.824 g, 2.88 mmol) described in the preceding section was combined with 2,2-bis(ethylthio)acetaldehyde[5] (0.95 mL, 5.8 mmol) in 21 mL of methanol.[6] The resulting solution was maintained for 20 h at room temperature, before $NaCNBH_3$ (0.236 g, 3.75 mmol) and bromocresol green indicator (~1 mg) were added. The pH of the resulting solution was adjusted with a 0.5 M solution of HCl in methanol until the solution's color changed from green to orange. Drops of methanolic HCl were periodically added to maintain this orange color over 30 min, after which time the solution was diluted with 60 mL of EtOAc and 15 mL of a saturated aqueous solution of $NaHCO_3$. The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 1.76 g of a yellow oil. This residue was purified by flash silica gel chromatography (25% acetone in hexanes) to afford

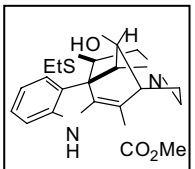
[5] For preparation of this aldehyde, see: Bates, G. S.; Ramaswamy, S. *Can. J. Chem.* **1983**, *61*, 2466–2475. The aldehyde was purified by flash silica gel chromatography (0→10% Et_2O in hexanes) immediately prior to use in order to remove 2-(ethylthio)-2-hydroxyacetaldehyde, which is a contaminant that leads to the formation of a major byproduct during the reductive alkylation step.

[6] This step was performed under an argon atmosphere with methanol that was degassed prior to use.

0.87 g (70%) of **18** as a colorless foam. Analysis by ^{1}H NMR spectroscopy indicated that **18** was formed as a 1:0.8 mixture of ester epimers.

Analytical samples of each diastereomer were obtained by normal phase HPLC (5% EtOAc in hexanes). The major diastereomer, **18a**, was obtained as a colorless foam: $[\alpha]^{23}_{589}$ – 1.76, $[\alpha]^{23}_{577}$ – 2.58, $[\alpha]^{23}_{546}$ – 4.98, $[\alpha]^{23}_{435}$ – 23.9, $[\alpha]^{23}_{405}$ – 33.5 (*c* 0.37, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (s, 1H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.3 Hz, 1H), 4.37 (app t, *J* = 3.6 Hz, 1H), 4.30 (d, *J* = 3.4 Hz, 1H), 4.26 (d, *J* = 5.8 Hz, 1H), 3.85 (s, 3H), 3.10 (dd, *J* = 13.3, 7.8 Hz, 1H), 2.81 (app s, 1H), 2.79–2.60 (m, 4H), 2.54 (dd, *J* = 13.3, 7.8 Hz, 1H), 2.44 (dd, *J* = 10.9, 3.7 Hz, 1H), 1.99 (td, *J* = 12.4 Hz, 3.1 Hz, 1H), 1.96–1.88 (m, 1H), 1.70–1.59 (m, 2H, overlaps with signal arising from residual H_2O), 1.30 (t, *J* = 7.4 Hz, 3H), 1.25 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.3 (C), 137.0 (C), 132.1 (C), 128.3 (C), 122.2 (CH), 120.2 (CH), 119.1 (CH), 111.3 (CH), 104.4 (C), 71.5 (CH), 62.0 (CH_2), 57.4 (CH), 52.5 (CH_3), 50.2 (CH), 43.4 (CH_2), 40.9 (CH), 36.0 (CH), 27.3 (CH_2), 24.7 (CH_2), 24.6 (CH_2), 14.9 (CH_3), 14.8 (CH_3); IR (thin film) 3391, 1728, 1460, 1448, 1300, 1265, 1204, 1160 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{22}H_{31}N_2O_3S_2$ $(M+H)^+$ 435.1776, found 435.1764.

The minor diastereomer, **18b**, was obtained as a colorless foam: $[\alpha]^{23}_{589}$ – 53.3, $[\alpha]^{23}_{577}$ – 56.0, $[\alpha]^{23}_{546}$ – 61.8, $[\alpha]^{23}_{435}$ – 99.0, $[\alpha]^{23}_{405}$ – 108.4 (*c* 0.51, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (s, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.20 (t, *J* = 6.7 Hz, 1H), 7.14 (t, *J* = 7.4 Hz, 1H), 4.32 (app s, 2H), 4.05 (t, *J* = 7.0 Hz, 1H), 3.82 (s, 3H), 3.74 (s, 1H), 3.14 (dd, *J* = 13.3, 7.5 Hz, 1H), 2.97 (app s, 1H), 2.81–2.63 (m, 5H), 2.55 (dd, *J* = 13.3, 6.6 Hz, 1H), 2.54–2.48 (m, 1H), 2.13–1.98 (m, 2H), 1.70 (app d, *J* = 11.5 Hz, 1H), 1.31 (t, *J* = 7.4 Hz, 3H), 1.26 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.8 (C), 136.8 (C), 130.7 (C), 128.5 (C), 122.4 (CH), 120.3 (CH), 119.5 (CH), 111.1 (CH), 106.3 (C), 70.4 (CH), 62.1 (CH_2), 57.0 (CH), 53.3 (CH_3), 50.2 (CH), 43.59 (CH), 43.57 (CH_2) 35.7 (CH), 31.6 (CH_2), 24.7 (CH_2), 24.6 (CH_2), 14.84 (CH_3), 14.82 (CH_3); IR (thin film) 3387, 1718, 1458, 1300, 1266, 1200, 1175, 1163 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{22}H_{31}N_2O_3S_2$ $(M+H)^+$ 435.1776, found 435.1775.



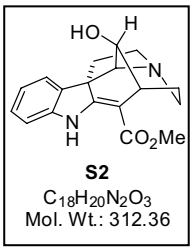
19
$C_{20}H_{24}N_2O_3S$
Mol. Wt.: 372.48

Pentacyclic Thioether 19. A suspension of dimethyl(methylthio)sulfonium tetrafluoroborate (DMTSF, 0.447 g, 2.28 mmol) and activated 4Å molecular sieves (3.8 g) in 250 mL of CH_2Cl_2 was stirred for 1 h. Separately, a mixture of tertiary amine **18** (0.825 g, 1.90 mmol) and activated 4Å molecular sieves (1.8 g) in 70 mL of CH_2Cl_2 was stirred for 1 h. The mixture containing **18** was then transferred by cannula into the suspension containing DMTSF, and the resulting mixture was stirred at room temperature for 20 min. This mixture was then diluted with 30 mL of a saturated aqueous solution of $NaHCO_3$ and filtered through a pad of Celite. The layers of the filtrate were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 30 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 0.78 g of a brown oil. This residue was purified by flash silica gel chromatography (5% MeOH in CH_2Cl_2) to afford 0.53 g (75%) of **19** as a colorless foam:[7] $[\alpha]^{24}_{589}$ + 685.8, $[\alpha]^{24}_{577}$ + 733.6, $[\alpha]^{24}_{546}$ + 887.9, $[\alpha]^{24}_{435}$ + 2333, $[\alpha]^{24}_{405}$ + 2944 (*c* 1.04, $CHCl_3$, >99% ee); ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.82 (s, 1H) 7.18–7.14 (m, 2H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 4.16 (app t, *J* = 3.0 Hz, 1H), 4.00–3.97 (m, 2H), 3.77 (s, 3H), 3.29–3.25 (m, 2H), 3.02 (dt, *J* = 12.0, 4.5 Hz, 1H), 2.87 (t, *J* = 11.7 Hz, 1H), 2.47–2.42 (m, 1H), 2.28 (dq, *J* = 12.4, 7.4 Hz, 1H), 2.22 (dq, *J* = 12.4, 7.4 Hz, 1H), 2.07 (br s, 1H, O*H*), 1.87–1.83 (m, 2H), 1.03 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.7 (C), 168.8 (C), 145.5 (C), 132.3 (C), 128.5 (CH), 124.0 (CH), 120.3 (CH), 109.7 (CH), 94.0 (C), 68.4 (CH), 67.1 (CH), 63.3 (CH_2), 58.7 (C), 55.5 (CH), 51.5 (CH_3), 45.6 (CH_2), 34.9 (CH), 27.2 (CH_2), 26.9 (CH_2), 14.9 (CH_3); IR (thin film) 3362, 1668, 1602, 1463, 1237, 1112 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{20}H_{25}N_2O_3S$ $(M+H)^+$ 373.1586, found 373.1585.

Enhancing the Enantiomeric Purity of Pentacyclic Thioether 19 from 91% ee to >99% ee. Pentacyclic alcohol **19** (0.453 g, 91% ee) was added to 2.3 mL of toluene, and the resulting suspension was heated to 100 °C until the mixture became homogeneous. This solution was allowed to cool to room temperature for 18 h, during which time crystals formed. The crystals were removed by filtration, washed with 1 mL of toluene, and the combined filtrates were concentrated in vacuo to afford 0.41 g (91%) of **19** as a colorless foam. A sample of this material was desulfurized by the procedure described in the following paragraph and analyzed by

[7] The methyl thioether analog **iv** was isolated as an inseparable contaminant (5–15 mol %, variable from run to run) as determined by ^{1}H NMR (diagnostic singlet corresponding to methyl thioether group at 1.81 ppm) and LRMS analysis.

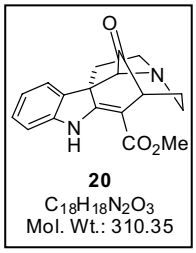
enantioselective HPLC, which indicated an enantiomeric excess of >99% [Chiralpak AD column; flow: 1.0 mL/min; 70:30 *n*-hexanes:*i*-PrOH; 254 nm; major enantiomer (+)-**S2**, t_R = 12.7 min; minor enantiomer (–)-**S2**, t_R = 11.1 min].



S2
$C_{18}H_{20}N_2O_3$
Mol. Wt.: 312.36

Pentacyclic Alcohol S2.[8] Activated Raney nickel (10.7 g, 50% slurry in water from Acros) was suspended in 10 mL of THF, stirred, and allowed to settle, before the supernatant was decanted. This wash was repeated nine more times before a solution of pentacyclic thioether **19** (0.264 g, 0.710 mmol) in 16 mL of THF was added. This suspension was stirred at 50 °C for 1 h, before it was filtered through a pad of Celite.[9] The filter cake was suspended in 50 mL of MeOH and EtOAc (1:1 v/v) and stirred for 3 h at room temperature. This suspension was then filtered, and the filtrate was combined with the filtrate from the previous filtration. The filter cake was extracted in this way two more times. The combined filtrates were concentrated in vacuo then diluted with 5 mL of a saturated aqueous solution of NaCl and 15 mL of EtOAc. The layers were separated and the aqueous layer was extracted with EtOAc (3 x 15 mL).[10] The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 0.195 g of a pale yellow solid. This residue was purified by flash silica gel chromatography (10% MeOH in $CHCl_3$, neutralized silica gel) to afford 0.17 g (75%) of **S2** as a colorless foam: $[\alpha]^{23}_{589}$ + 564.4, $[\alpha]^{23}_{577}$ + 604.9, $[\alpha]^{23}_{546}$ + 732.0, $[\alpha]^{23}_{435}$ + 1913, $[\alpha]^{23}_{405}$ + 1303 (*c* 1.39, $CHCl_3$, >99% ee); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.90 (s, 1H), 7.16 (d, *J* = 7.4 Hz, 1H), 7.10 (t, *J* = 7.7 Hz, 1H), 6.89 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.8 Hz, 1H), 4.13 (t, *J* = 3.4 Hz, 1H), 3.91–3.88 (m, 1H), 3.75 (s, 3H), 3.23 (app s, 1H), 3.05–2.97 (m, 1H), 2.92–2.81 (m, 3H), 2.34 (td, *J* = 12.1, 5.1 Hz, 1H), 2.06 (br s, 1H), 1.84–1.73 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.3 (C), 169.0 (C), 143.8 (C), 137.1 (C), 127.5 (CH), 121.5 (CH), 119.8 (CH), 110.0 (CH), 93.1 (C), 68.4 (CH), 67.3 (CH), 55.4 (C), 54.4 (CH_2), 51.3 (CH_3), 44.4 (CH_2), 43.7 (CH_2), 35.3 (CH), 27.0 (CH_2); IR (thin film) 3362, 1673, 1601, 1463, 1238, 1147, 1100 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{18}H_{21}N_2O_3$ $(M+H)^+$ 313.1552, found 313.1541.

[8] This compound is alkylated by CH_2Cl_2 at an appreciable rate.
[9] This filter cake was not suctioned to dryness.
[10] This aqueous wash removed Celite that had dissolved with the product during extractions of the Raney nickel filter cake. This wash was especially useful when crude **S2** was elaborated without chromatographic purification.



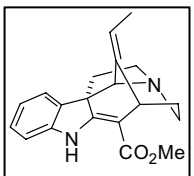
20
$C_{18}H_{18}N_2O_3$
Mol. Wt.: 310.35

Pentacyclic Ketone 20. A solution of pentacyclic alcohol **S2** (0.164 g, 0.56 mmol) and Ac_2O (1.1 mL, 11 mmol) in 8.4 mL of dry DMSO was maintained for 3 h at room temperature. This solution was then diluted with 40 mL of EtOAc and 28 mL of a saturated aqueous solution of $NaHCO_3$, and the mixture was stirred for 1 h at room temperature to hydrolyze excess Ac_2O. The layers were separated and the aqueous layer was extracted with EtOAc (4 x 30 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo[11] to afford 0.156 g (96%) of **20** as an orange oil, which was of sufficient purity to be used in the next step without further purification:[12] $[\alpha]^{23}_{589}$ + 641.2, $[\alpha]^{23}_{577}$ + 688.0, $[\alpha]^{23}_{546}$ + 833.1, $[\alpha]^{23}_{435}$ + 2158 (*c* 0.69, $CHCl_3$, >99% ee); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.85 (s, 1H), 7.15 (d, *J* = 7.5 Hz, 1H), 7.14 (t, *J* = 7.3, Hz), 6.91 (t, *J* = 7.5 Hz, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 3.91 (app s, 1H), 3.77 (s, 3H), 3.49–3.46 (m, 1H), 3.27–3.21 (m, 1H), 3.20–3.10 (m, 2H), 2.94–2.87 (m, 2H), 2.45–2.37 (m, 1H), 2.26–2.19 (m, 1H), 2.17–2.10 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.4 (C), 167.6 (C), 166.8 (C), 144.6 (C), 133.9 (C), 128.6 (CH), 121.9 (CH), 120.9 (CH), 110.3 (CH), 97.8 (C), 67.6 (CH), 64.1 (C), 53.8 (CH_2), 51.7 (CH_3), 47.1 (CH_2), 46.3 (CH_2), 42.0 (CH), 30.3 (CH_2); IR (thin film) 3351, 1733, 1677, 1602, 1464, 1234, 1198, 1101 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{18}H_{19}N_2O_3$ $(M+H)^+$ 311.1396, found 311.1404.

(+)-Condylocarpine (2a) and (+)-Isocondylocarpine (2b). A 2.5 M solution of *n*-BuLi (0.59 mL, 1.5 mmol) was added to a suspension of ethyltriphenylphosphonium bromide (0.579 g, 1.56 mmol) in 5.7 mL of toluene, and the resulting orange suspension was stirred for 1 h at room temperature. A solution of pentacyclic ketone **20** (55 mg, 0.18 mmol) in 1.0 mL of THF was then added by cannula, and the residue in the transfer vessel was added to the reaction mixture with an additional 0.4 mL of THF. The resulting mixture was stirred for 3.5 h at room temperature, before it was diluted with 5 mL of EtOAc and 5 mL of a saturated aqueous solution of $NaHCO_3$. The layers were separated and the aqueous layer was extracted with EtOAc (3 x 5 mL). The combined organic layers were dried with $MgSO_4$ and concentrated in vacuo to afford 0.190 g of an orange residue. This residue was purified by flash silica gel chromatography (4%→5%

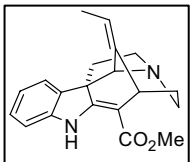
[11] DMSO was removed under vacuum (10^{-4} mm) for 3 days.
[12] An analytical sample of the racemate, which was obtained by crystallization from toluene (mp 225 °C dec), was used to obtain analytical data other than the specific rotation.

MeOH in $CHCl_3$, neutralized silica gel) to afford 44 mg (77%) of a 1:1 mixture of **2a** and **2b** as a colorless foam.



2a
$C_{20}H_{22}N_2O_2$
Mol. Wt.: 322.40

Analytical samples of each diastereomer were obtained by normal phase HPLC (15% reagent alcohol in $CHCl_3$). (+)-Condylocarpine (**2a**) was isolated as a colorless amorphous solid:[13] $[\alpha]^{23}_{589}$ + 854, $[\alpha]^{23}_{577}$ + 916, $[\alpha]^{23}_{546}$ + 1100, $[\alpha]^{23}_{435}$ + 2752, $[\alpha]^{23}_{405}$ + 3433 (*c* 0.39, $CHCl_3$, >99% ee); $[\alpha]^{23}_{589}$ + 631, $[\alpha]^{23}_{577}$ + 674, $[\alpha]^{23}_{546}$ + 807, $[\alpha]^{23}_{435}$ + 2026, $[\alpha]^{23}_{405}$ + 2414 (*c* 0.59, EtOH, >99% ee); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (s, 1H), 7.18 (d, *J* = 7.3 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 6.89 (t, *J* = 7.4 Hz, 1H), 6.78 (d, *J* = 7.7 Hz, 1H), 5.32 (q, *J* = 6.7 Hz, 1H), 4.12 (s, 1H), 3.93–3.89 (m, 1H), 3.80 (s, 3H), 3.08 (ddd, *J* = 11.3, 10.0, 6.8 Hz, 1H), 3.03 (ddd, *J* = 13.0, 7.0, 6.1 Hz, 1H), 2.98 (ddd, *J* = 11.3, 6.9, 3.3 Hz, 1H), 2.78 (ddd, *J* = 13.0, 9.6, 7.1 Hz, 1H), 2.66 (ddd, *J* = 12.8, 7.6, 5.2 Hz, 1H), 1.96 (ddd, *J* = 12.9, 6.7, 3.3 Hz, 1H), 1.94–1.89 (m, 1H), 1.89–1.83 (m, 1H), 1.59 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.7 (C), 168.3 (C), 144.7 (C), 137.8 (C), 135.7 (C), 127.8 (CH), 121.2 (CH), 120.4 (CH), 117.4 (CH), 109.9 (CH), 101.6 (C), 69.1 (CH), 60.2 (C), 53.4 (CH_2), 51.4 (CH_3), 46.3 (CH_2), 45.5 (CH_2), 29.2 (CH), 28.6 (CH_2), 13.3 (CH_3); IR (thin film) 3367, 2948, 2919, 2860, 1674, 1603, 1474, 1463, 1435, 1381, 1357, 1269, 1232, 1196, 1160, 1117, 1099, 1062, 910, 732 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{20}H_{23}N_2O_2$ $(M+H)^+$ 323.1760, found 323.1767.

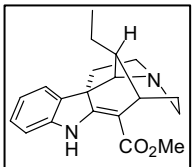


2b
$C_{20}H_{22}N_2O_2$
Mol. Wt.: 322.40

(+)-Isocondylocarpine (**2b**) was isolated as a colorless amorphous solid:[13] $[\alpha]^{23}_{589}$ + 798, $[\alpha]^{23}_{577}$ + 858, $[\alpha]^{23}_{546}$ + 1035, $[\alpha]^{23}_{435}$ + 2638, $[\alpha]^{23}_{405}$ + 3785 (*c* 0.145, $CHCl_3$, >99% ee); $[\alpha]^{23}_{589}$ + 560, $[\alpha]^{23}_{577}$ + 600, $[\alpha]^{23}_{546}$ + 727, $[\alpha]^{23}_{435}$ + 1870, $[\alpha]^{23}_{405}$ + 2633 (*c* 0.22, EtOH, >99% ee); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (s, 1H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 6.89 (t, *J* = 7.4 Hz, 1H), 6.78 (d, *J* = 7.8 Hz, 1H), 5.41 (q, *J* = 6.7 Hz, 1H), 4.65 (s, 1H), 3.80 (s 3H), 3.43 (app t, *J* = 4.2 Hz, 1H), 3.11 (ddd, *J* = 11.2, 9.7, 6.9 Hz, 1H), 3.03 (ddd, *J* = 12.4, 6.4, 6.1 Hz, 1H), 2.99 (ddd, *J* = 11.3, 7.0, 3.4 Hz, 1H), 2.80 (ddd, *J* = 12.9, 9.6, 7.1 Hz, 1H), 2.65 (ddd, *J* = 13.2, 6.6, 6.3 Hz, 1H), 1.98 (ddd, *J* = 12.9, 6.8, 3.2 Hz, 1H), 1.91 (app q, *J* = 5.8 Hz, 2H), 1.60 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.2 (C), 168.2 (C), 144.8 (C), 137.6 (C), 135.6 (C), 127.8 (CH), 121.2 (CH), 120.2 (CH), 117.9 (CH), 109.9 (CH), 102.3 (C), 60.5 (CH), 60.1 (C),

[13] Analytical samples were washed with aqueous $NaHCO_3$ before analysis. NMR spectra were sensitive to traces of DCl; hence, $CDCl_3$ was filtered through activated basic alumina prior to use.

53.6 (CH_2), 51.4 (CH_3), 46.2 (CH_2), 45.3 (CH_2), 37.7, (CH), 29.1 (CH_2), 13.1 (CH_3); IR (thin film) 3366, 2948, 2918, 2860, 2849, 1674, 1603, 1475, 1463, 1436, 1382, 1358, 1271, 1235, 1200, 1160, 1116, 1103, 1066, 910, 733 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{20}H_{23}N_2O_2$ $(M+H)^+$ 323.1760, found 323.1762.



21
$C_{20}H_{24}N_2O_2$
Mol. Wt.: 324.42

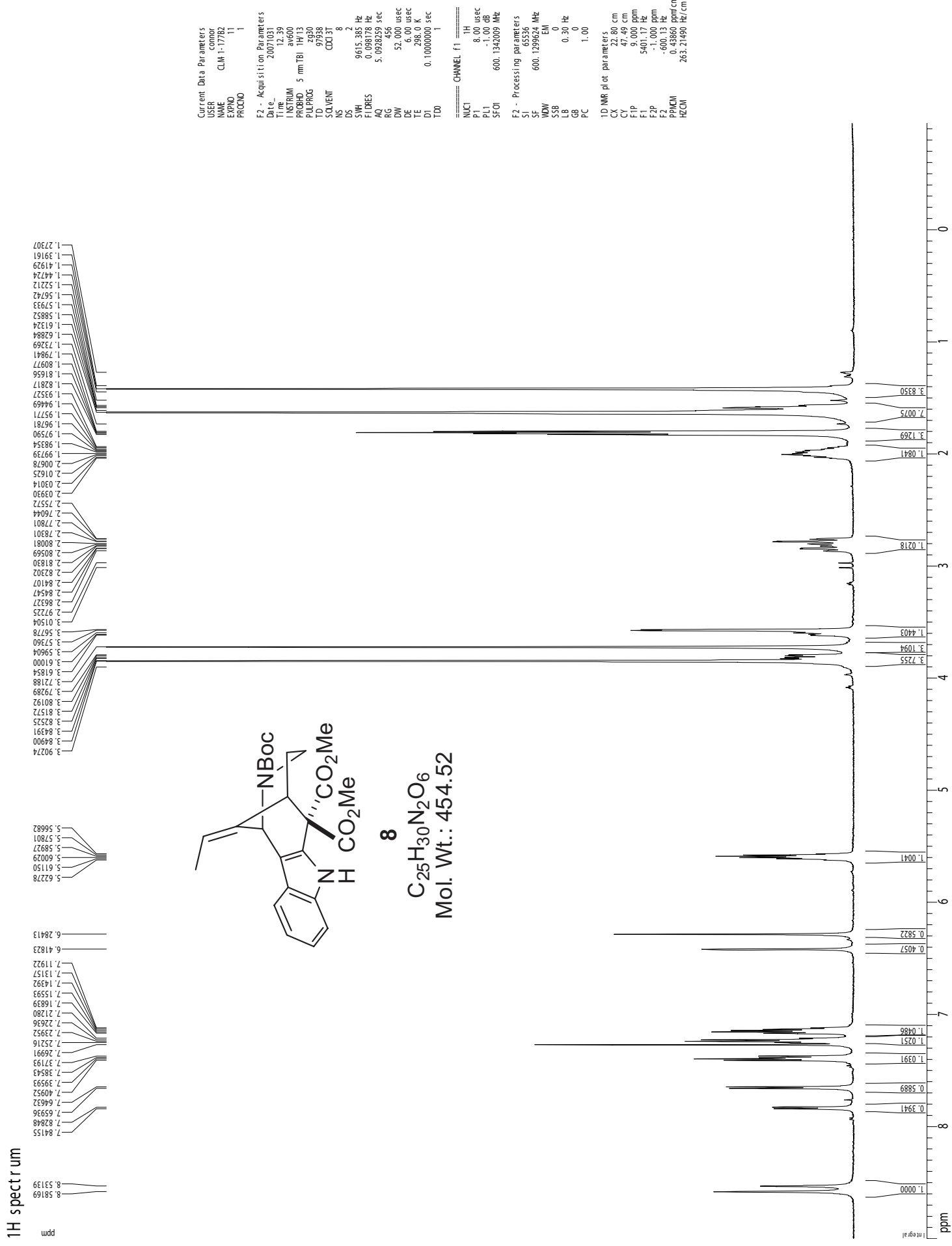
(+)-Tubotaiwine (21). Platinum oxide (10 mg, 0.044 mmol) was added to a solution of (+)-condylocarpine (**2a**) and (+)-isocondylocarpine (**2b**) (19 mg, 0.059 mmol, **2a**:**2b** = 1:1) in 2 mL of ethanol, and the mixture was stirred under an atmosphere of hydrogen[14] for 1 h at room temperature.[15] This suspension was then filtered and concentrated in vacuo to afford 17 mg (91%) of pure (+)-tubotaiwine (**21**) as a colorless amorphous solid:[13] $[\alpha]^{22}_{589}$ + 584, $[\alpha]^{23}_{577}$ + 625, $[\alpha]^{23}_{546}$ + 756, $[\alpha]^{23}_{435}$ + 1972 (*c* 0.91, $CHCl_3$, >99% ee); $[\alpha]^{23}_{589}$ + 504, $[\alpha]^{23}_{577}$ + 539, $[\alpha]^{23}_{546}$ + 653, $[\alpha]^{23}_{435}$ + 1721, $[\alpha]^{23}_{405}$ + 2358 (*c* 0.67, EtOH, >99% ee); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.86 (s, 1H), 7.14 (d, *J* = 7.3 Hz, 1H), 7.11 (t, *J* = 7.7 Hz, 1H), 6.88 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 3.81 (s, 1H), 3.77 (s, 3H), 3.06–2.99 (m, 2H), 2.96 (dt, *J* = 11.8, 4.0 Hz, 1H), 2.94–2.87 (m, 1H), 2.84 (dd, *J* = 10.8, 7.1 Hz, 1H), 2.46 (ddd, *J* = 11.7, 9.6, 8.6 Hz, 1H), 2.00–1.95 (m, 1H), 1.82–1.76 (m, 3H), 0.86–0.78 (m, 2H), 0.70 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9 (C), 169.1 (C), 143.9 (C), 137.5 (C), 127.3 (CH), 121.2 (CH), 119.9 (CH), 109.8 (CH), 95.8 (C), 65.8 (CH), 55.4 (C), 54.2 (CH_2), 51.3 (CH_3), 45.5 (CH_2), 44.3 (CH_2), 41.5 (CH), 31.2 (CH), 28.7 (CH_2), 24.1 (CH_2), 11.8 (CH_3); IR (thin film) 3363, 2957, 2931, 2874, 1674, 1602, 1475, 1463, 1435, 1279, 1235, 1204, 1151, 1123, 1099, 1027, 748 cm^{-1}; HRMS (ESI-TOF) m/z calcd for $C_{20}H_{25}N_2O_2$ $(M+H)^+$ 325.1916 found 325.1913.

[14] A balloon of hydrogen was used.
[15] Schumann, D.; Schmid, H. *Helv. Chim. Acta* **1963**, *46*, 1996–2003.

Table S1. Optical Rotations Reported for Condylocarpine, Isocondylocarpine, and Tubotaiwine

Condylocarpine	Isocondylocarpine	Tubotaiwine
+ 900 (*c* 0.556, $CHCl_3$) [16] + 870 (*c* 0.40, EtOH)	+ 537 (*c* 0.1, $CHCl_3$)[17]	+ 628 (*c* 0.80, $CHCl_3$)[18]
+ 899 (*c* 0.426, $CHCl_3$)[19]		+ 611 (*c* 1.02, $CHCl_3$)[20]
+ 870 (*c* 0.1)[21]		+ 598 (*c* 0.13, $CHCl_3$)[22]
+ 501 (*c* 0.7, $CHCl_3$)[23]		+ 584 (*c* 0.676, $CHCl_3$)[24]
		+ 538 ($CHCl_3$)[25]
		+ 417 ($CHCl_3$)[26]
		+ 400 (*c* 3, $CHCl_3$)[27]
		+ 336 (*c* 1.06, MeOH)[28]

[16] Stauffacher, D. *Helv Chim Acta* **1961**, *44*, 2006–2015.
[17] Wu, Y.; Kitajima, M.; Kogure, N.; Wang, Y.; Zhang, R.; Takayama, H. *J. Nat. Med.* **2009**, *63*, 283–289.
[18] Pinar, M.; Renner, U.; Hesse, M.; Schmid, H. *Helv. Chim. Acta* **1972**, *55*, 2972–2974.
[19] Linde, H. H. A. *Helv Chim Acta* **1965**, *48*, 1822–1842.
[20] Kump, W. G.; Patel, M. B.; Rowson, J. M.; Schmid, H. *Helv. Chim. Acta* **1964**, *47*, 1497–1503.
[21] Achenbach, H.; Raffelsberger, B. *Z. Naturforsch. B: Anorg. Chem., Org. Chem*. **1980**, *35*, 885–891. Specific rotation data was accessed from MDL Crossfire Commander, version 7.1; Elsevier Information Systems GmbH: 2007; BRN 6238018.
[22] Sheludko, Y.; Gerasimenko, I.; Platonova, O. *Planta Med.* **2000**, *66*, 656–659.
[23] Achenbach, H.; Benirschke, M.; Torrenegra, R. *Phytochemistry* **1997**, *45*, 325–335.
[24] Pinar, M.; Schmid, H. *Liebigs Ann. Chem*. **1963**, *668*, 97–104.
[25] Gunasekera, S. P.; Cordell, G.; Farnsworth, N. R. *Phytochemistry* **1980**, *19*, 1213–1218.
[26] Panas, J. M.; Morfaux, A. M.; Lemen, J.; Olivier, L. *Ann. Pharm. Fr.* **1972**, *30*, 273. Specific rotation data was accessed from MDL Crossfire Commander, version 7.1; Elsevier Information Systems GmbH: 2007; BRN 626683.
[27] Morfaux, A. M.; Mulamba, T.; Richard, B.; Delaude, C.; Massiot, G.; Le Men-Olivier, L. *Phytochemistry* **1982**, *21*, 1767–1769.
[28] Yamauchi, T.; Abe, F.; Padolina, W. G.; Dayrit, F. M. *Phytochemistry* **1990**, *29*, 3321–3325.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PULPROG zg30
TD 97938
SOLVENT CDCl3T
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928259 sec
RG 456
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DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
TD0 1
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PL1 -1.00 dB
SFO1 600.1342009 MHz
F2 - Processing parameters
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SF 600.1299624 MHz
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
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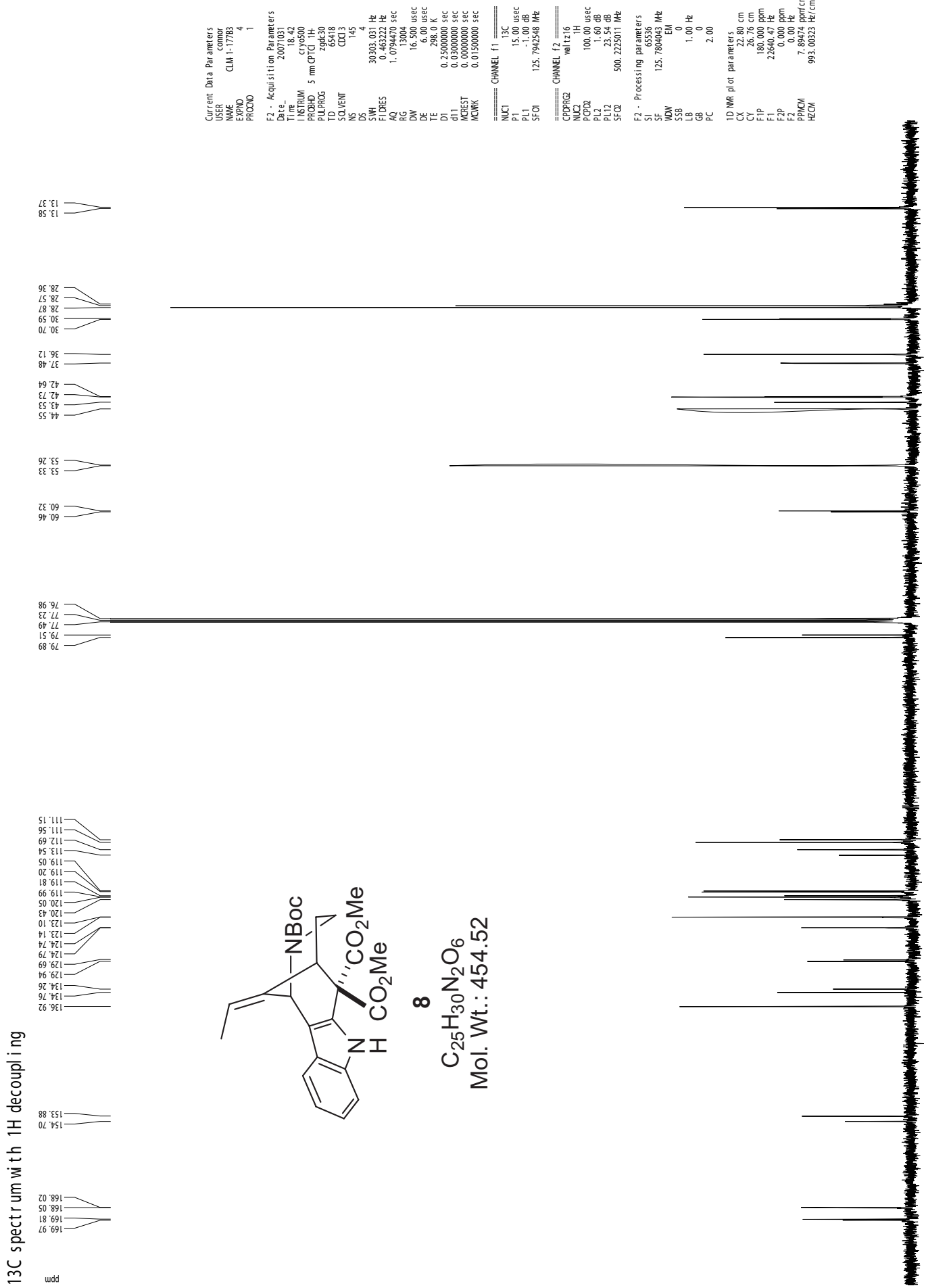
CY 47.49 cm
F1P 9.000 ppm
F1 5401.17 Hz
F2P -1.000 ppm
F2 -600.13 Hz
PPMCM 0.43860 ppm/cm
HZCM 263.21490 Hz/cm
NBoc
CO2Me
CO2Me
N
H
8
C25H30N2O6
Mol. Wt.: 454.52
ppm
integral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USER connor
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PROCNO 1
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Time_ 18.42
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PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 145
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz
F2 - Processing parameters
SI 65536
SF 125.7804043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 26.76 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
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13.58
28.36
28.57
28.87
30.59
30.70
36.12
37.48
42.64
42.73
43.53
44.55
53.26
53.33
60.32
60.46
76.98
77.23
77.49
79.51
79.89
111.15
111.56
112.69
113.54
119.05
119.20
119.81
119.99
120.05
120.43
123.10
123.14
124.74
124.79
129.69
129.94
134.26
134.76
136.92
153.88
154.70
168.02
168.05
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CO2Me
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H
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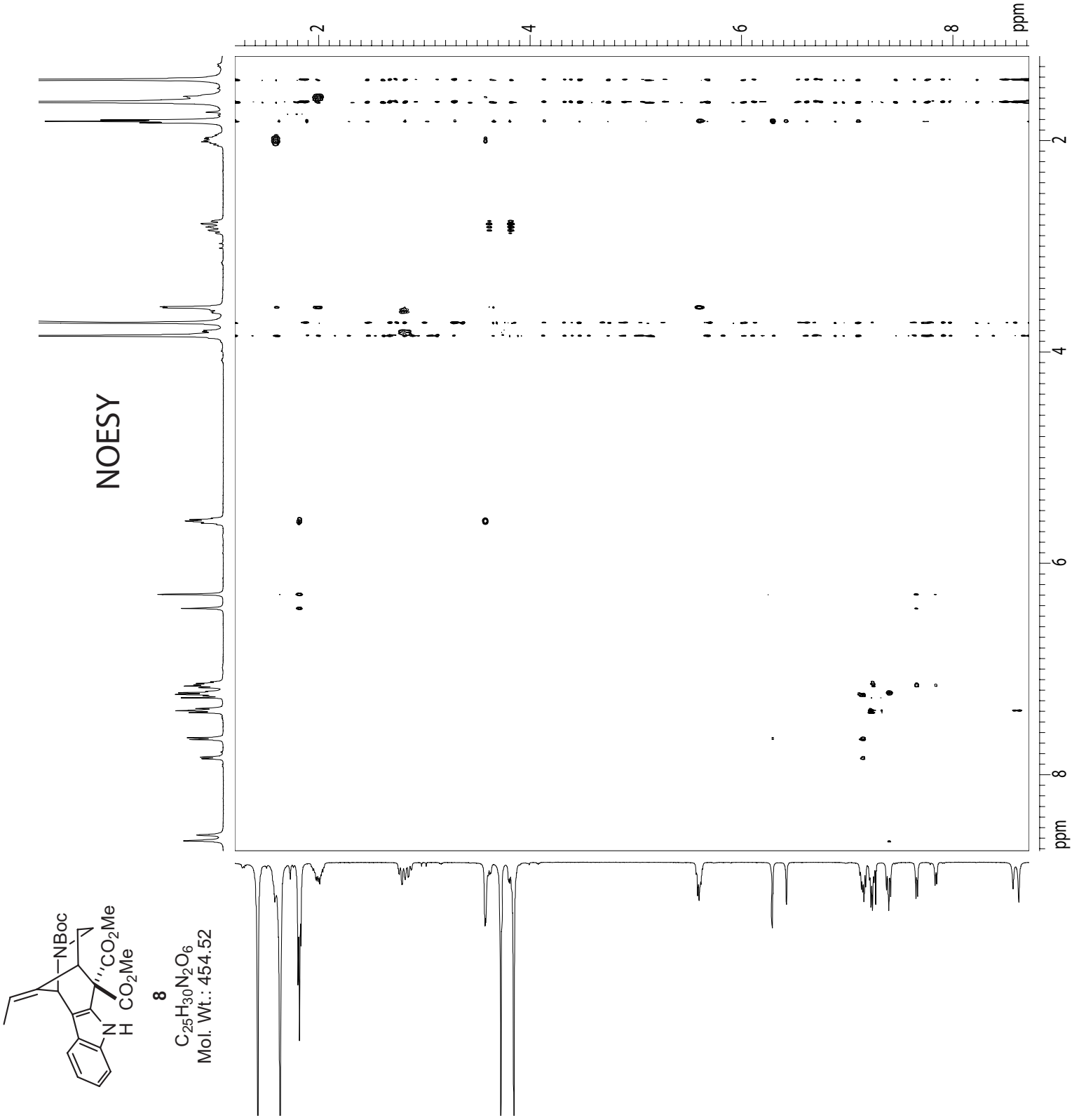
C25H30N2O6
Mol. Wt.: 454.52
ppm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N
H
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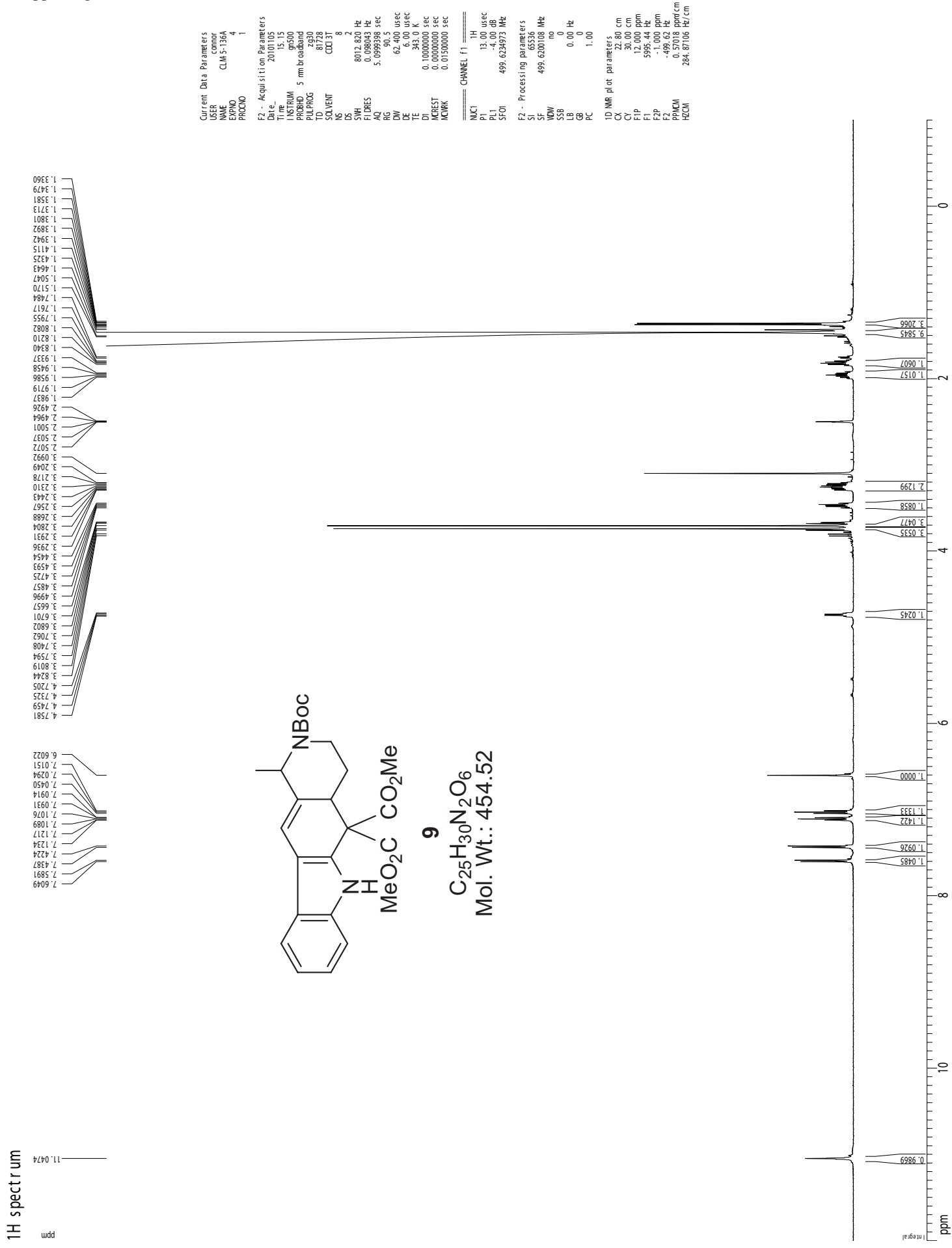
C25H30N2O6
Mol. Wt.: 454.52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TD 81728
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SWH 8012.820 Hz
FIDRES 0.098043 Hz
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RG 90.5
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DE 6.00 usec
TE 343.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
F2 - Processing parameters
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WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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CY 30.00 cm
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F1 5995.44 Hz
F2P -1.000 ppm
F2 -499.62 Hz
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HZCM 284.87106 Hz/cm
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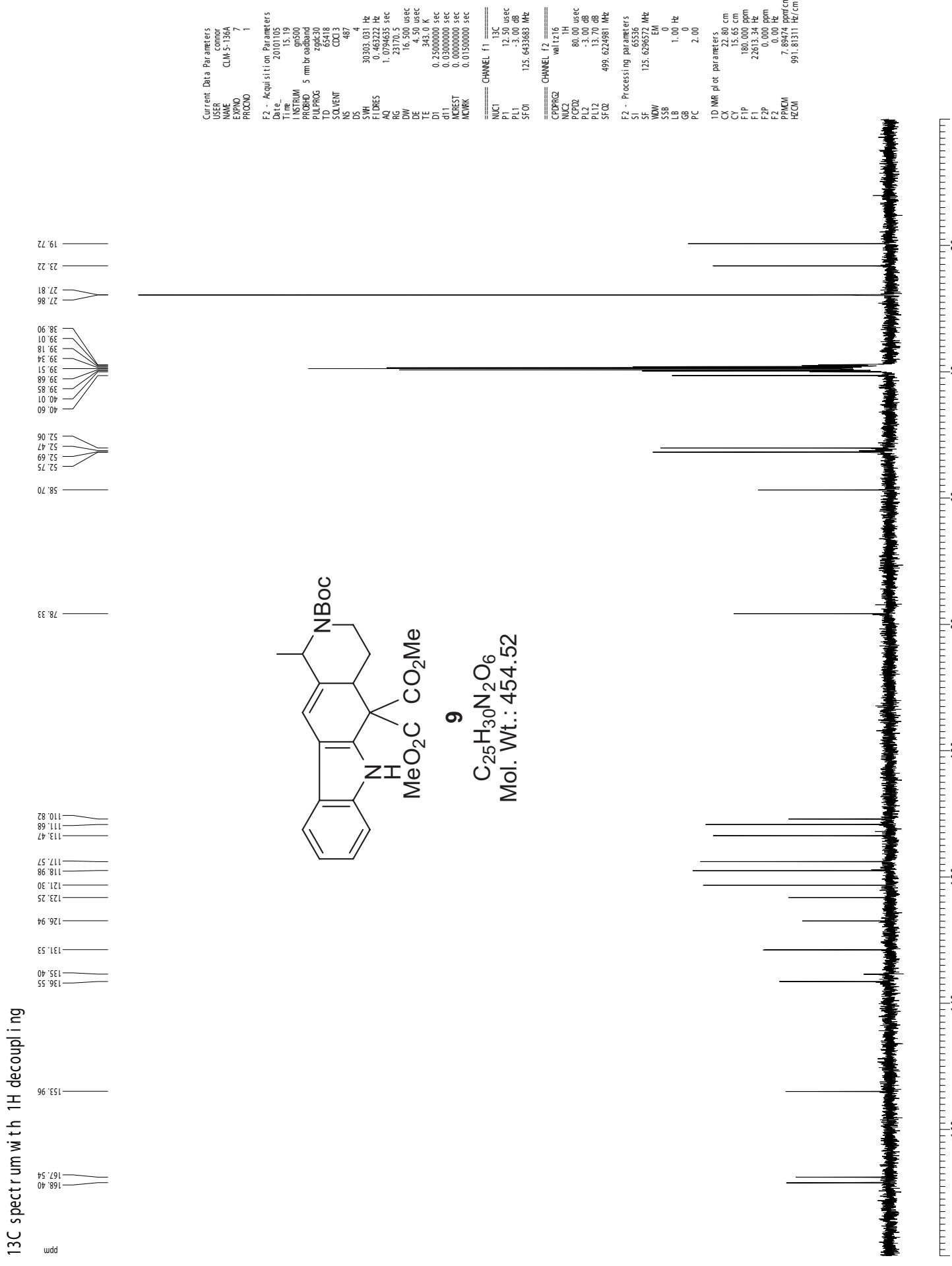
CO2Me
MeO2C
N
H
9
C25H30N2O6
Mol. Wt.: 454.52
ppm
integral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Current Data Parameters
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NAME CLM-5-136A
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
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Time 15.19
INSTRUM qnl500
PROBHD 5 mm broadband
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 487
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 23170.5
DW 16.500 usec
DE 4.50 usec
TE 343.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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NUC1 13C
P1 12.50 usec
PL1 -3.00 dB
SFO1 125.6433683 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 13.70 dB
SFO2 499.6224981 MHz
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
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CY 15.65 cm
F1P 180.000 ppm
F1 22613.34 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 991.81311 Hz/cm
NBoc
N
H
MeO2C CO2Me
9
C25H30N2O6
Mol. Wt.: 454.52
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153.96
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135.40
131.53
126.94
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118.98
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113.47
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110.82
78.33
58.70
52.75
52.69
52.47
52.06
40.60
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39.68
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38.90
27.86
23.22
19.72
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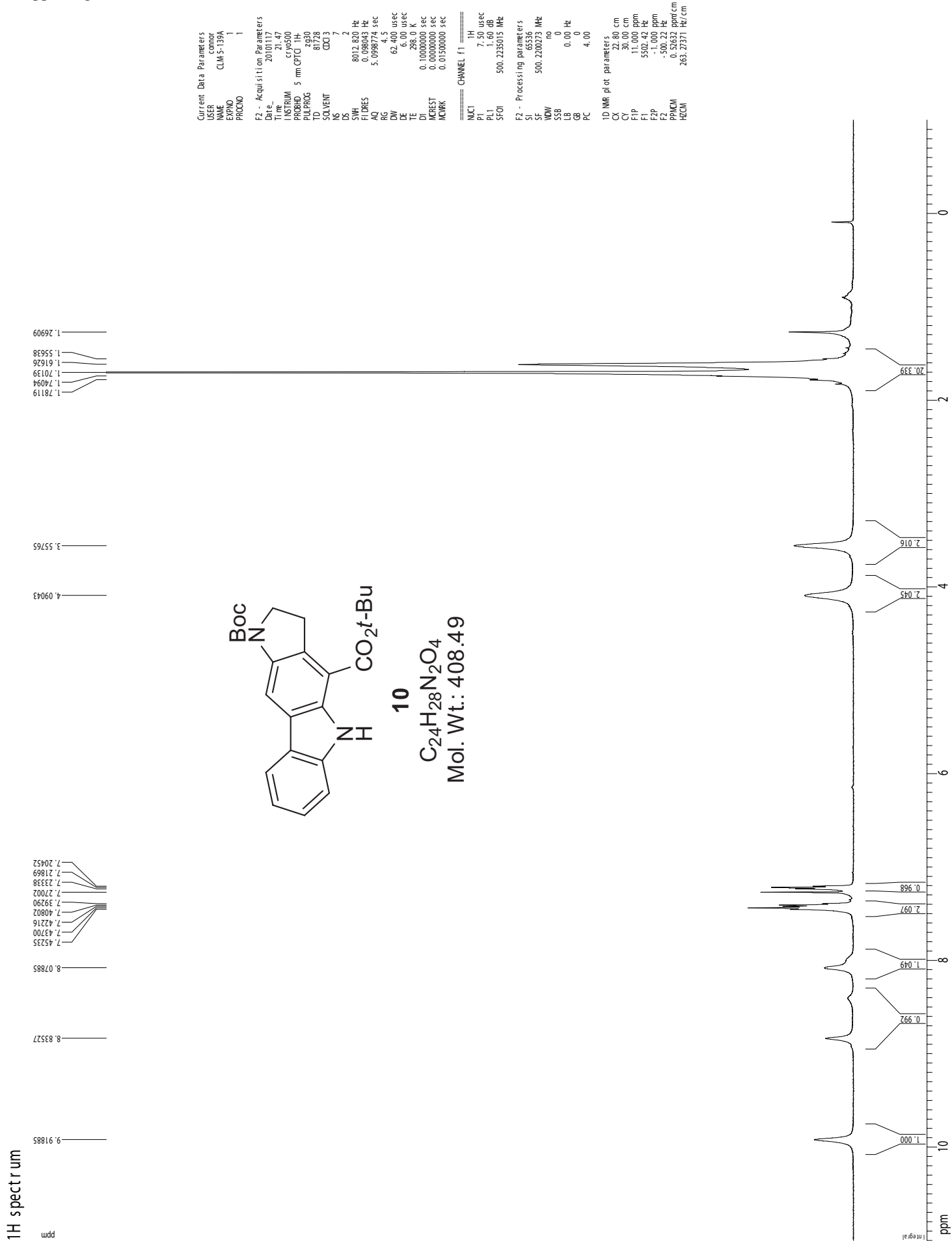
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PULPROG zg30
TD 81728
SOLVENT CDCl3
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FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200273 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
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CY 30.00 cm
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F1 5502.42 Hz
F2P -1.000 ppm
F2 -500.22 Hz
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HZCM 263.27371 Hz/cm
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CO2t-Bu
N
H
10
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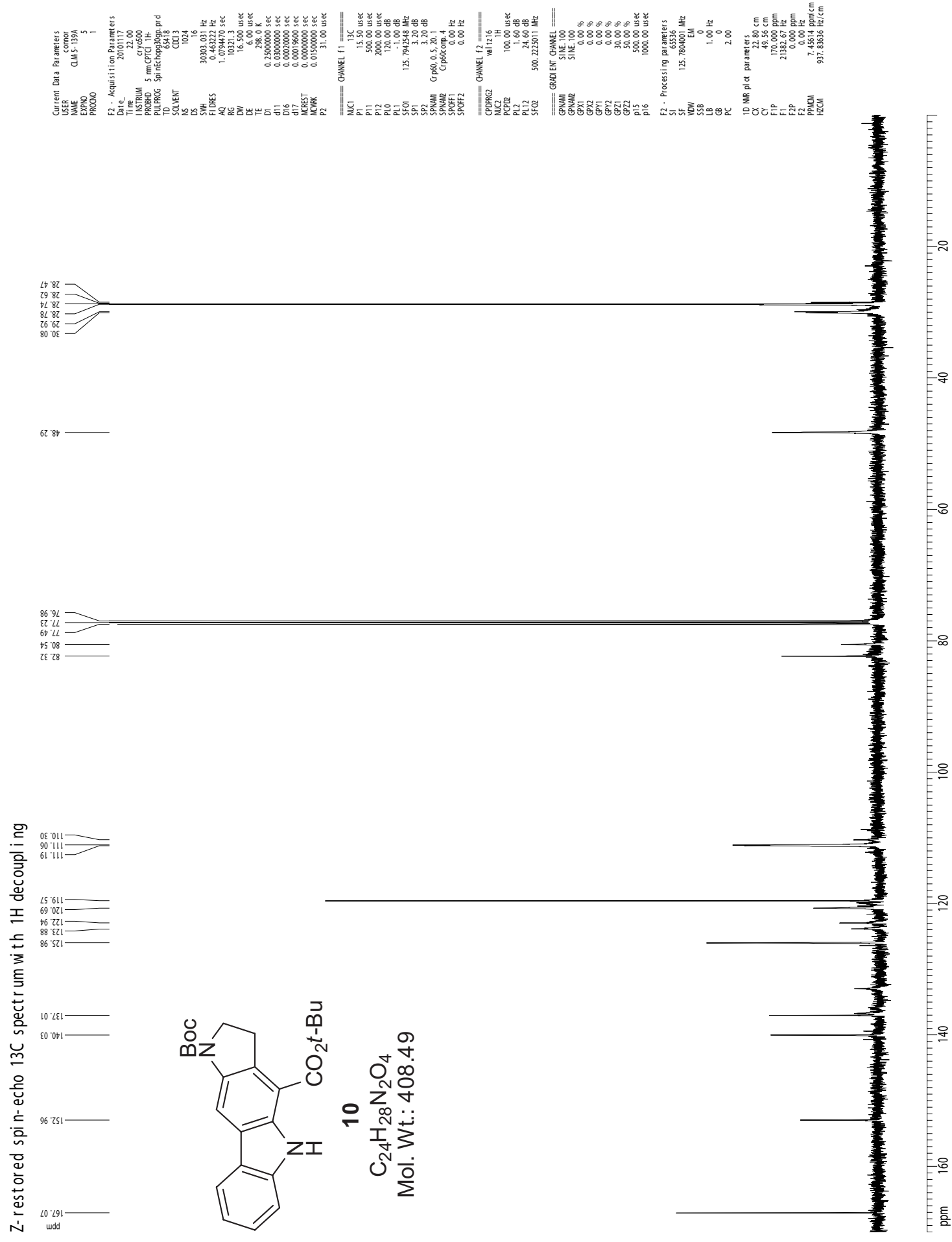
Mol. Wt.: 408.49
integral
ppm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CO2t-Bu
N
H
10
C24H28N2O4
Mol. Wt.: 408.49
ppm
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152.96
140.03
137.01
125.98
123.88
122.94
120.69
119.57
111.19
111.06
110.30
82.32
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77.23
76.98
48.29
30.08
29.92
28.78
28.74
28.62
28.47
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EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
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PULPROG spiecho93gp.prd
TD 65418
SOLVENT CDCl3
NS 1024
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SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00220000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
CHANNEL f1
NUCl 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
CHANNEL f2
waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
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GPNAM2 SINE.100
GPX1 0.00 %
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GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804001 MHz
WDW EM
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LB 1.00 Hz
GB 0
PC 2.00
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CY 49.56 cm
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F1 21382.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
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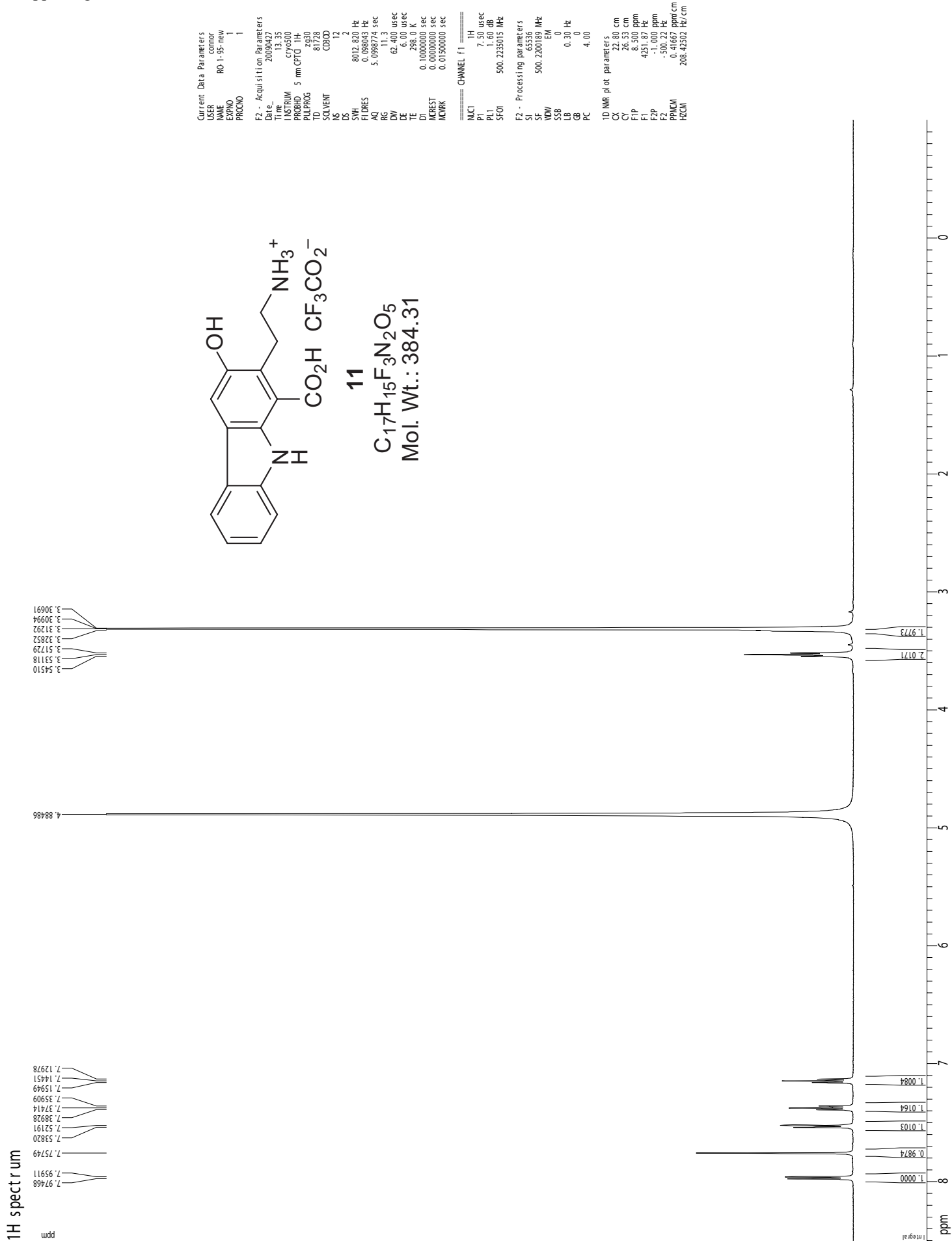
HZCM 937.83836 Hz/cm
ppm
20
40
60
80
100
120
140
160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PULPROG zg30
TD 81728
SOLVENT CD3OD
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 11.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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P1 7.50 usec
PL1 1.60 dB
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F2 - Processing parameters
SI 65536
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SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
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CY 26.53 cm
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F1 4251.87 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm
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NH3+
CO2H CF3CO2−
N
H
11
C17H15F3N2O5
Mol. Wt.: 384.31
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7.95911
7.75749
7.53820
7.52191
7.38928
7.37414
7.35909
7.15949
7.14451
7.12978
4.88486
3.54510
3.53118
3.51729
3.32852
3.31292
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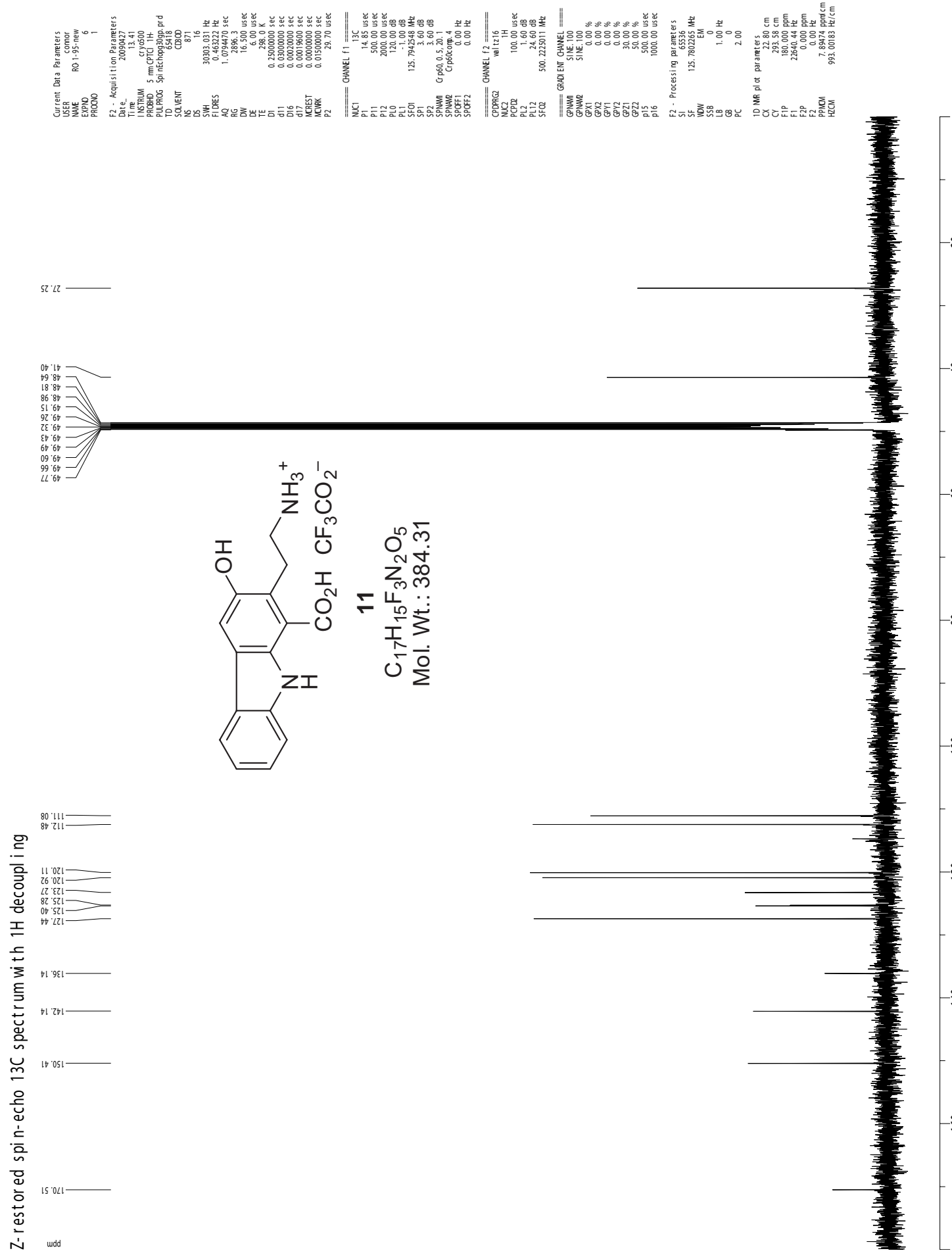
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
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125.28
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120.92
120.11
112.48
111.08
49.77
49.66
49.60
49.49
49.43
49.32
49.26
49.15
48.98
48.81
48.64
41.40
27.25
OH
NH3+
CO2H CF3CO2−
N
H
11
C17H15F3N2O5
Mol. Wt.: 384.31
Current Data Parameters
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NAME RO1-95-new
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PROCNO 1
F2 - Acquisition Parameters
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PULPROG Spiechopg30pp.prd
TD 65418
SOLVENT CD3OD
NS 871
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 2896.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec
======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM0 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
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GPNAM2 SINE.100
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GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
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WDW EM
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LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 20.58 cm
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F1 22640.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
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HZCM 993.00183 Hz/cm
ppm
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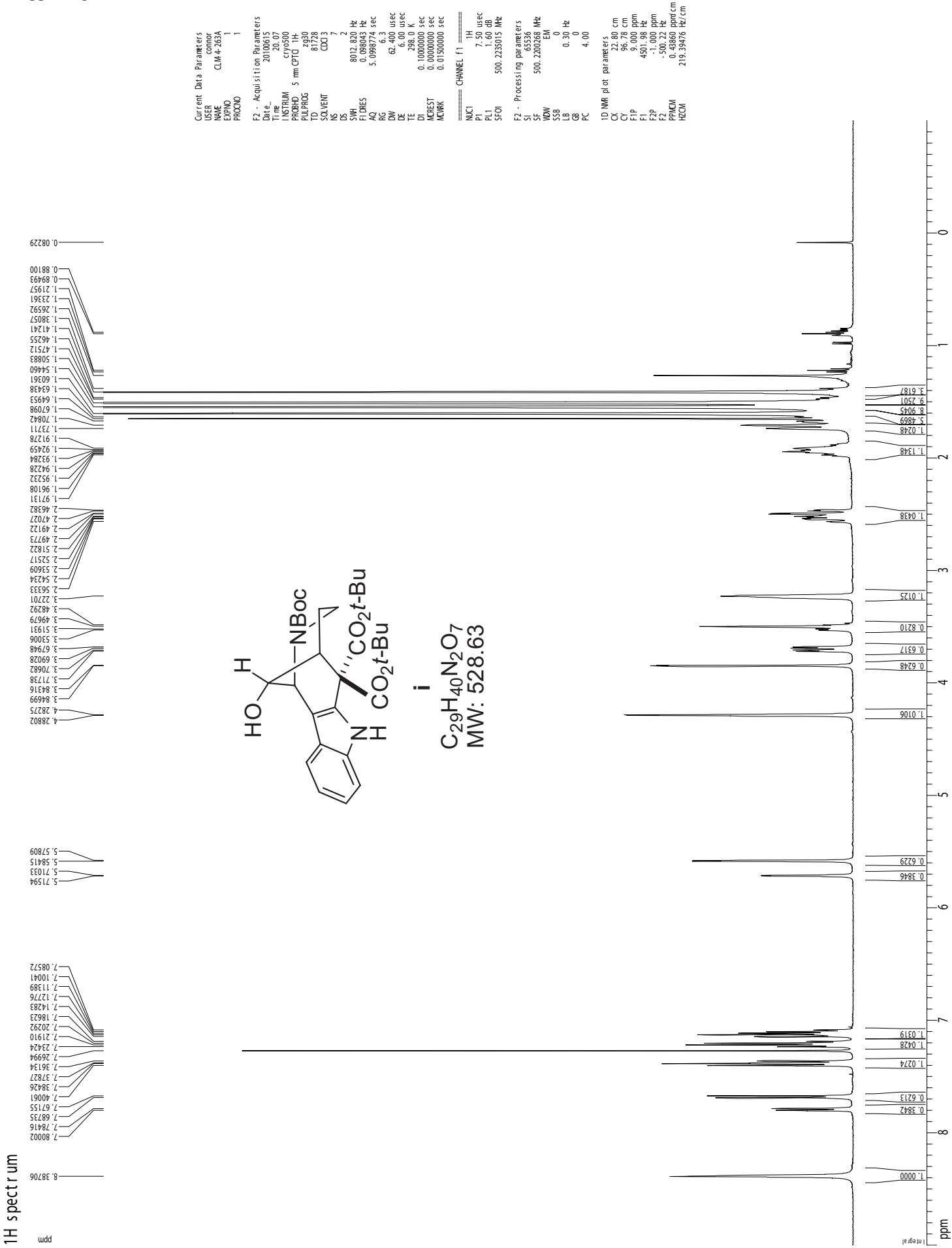
40
60
80
100
120
140
160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PROCNO 1
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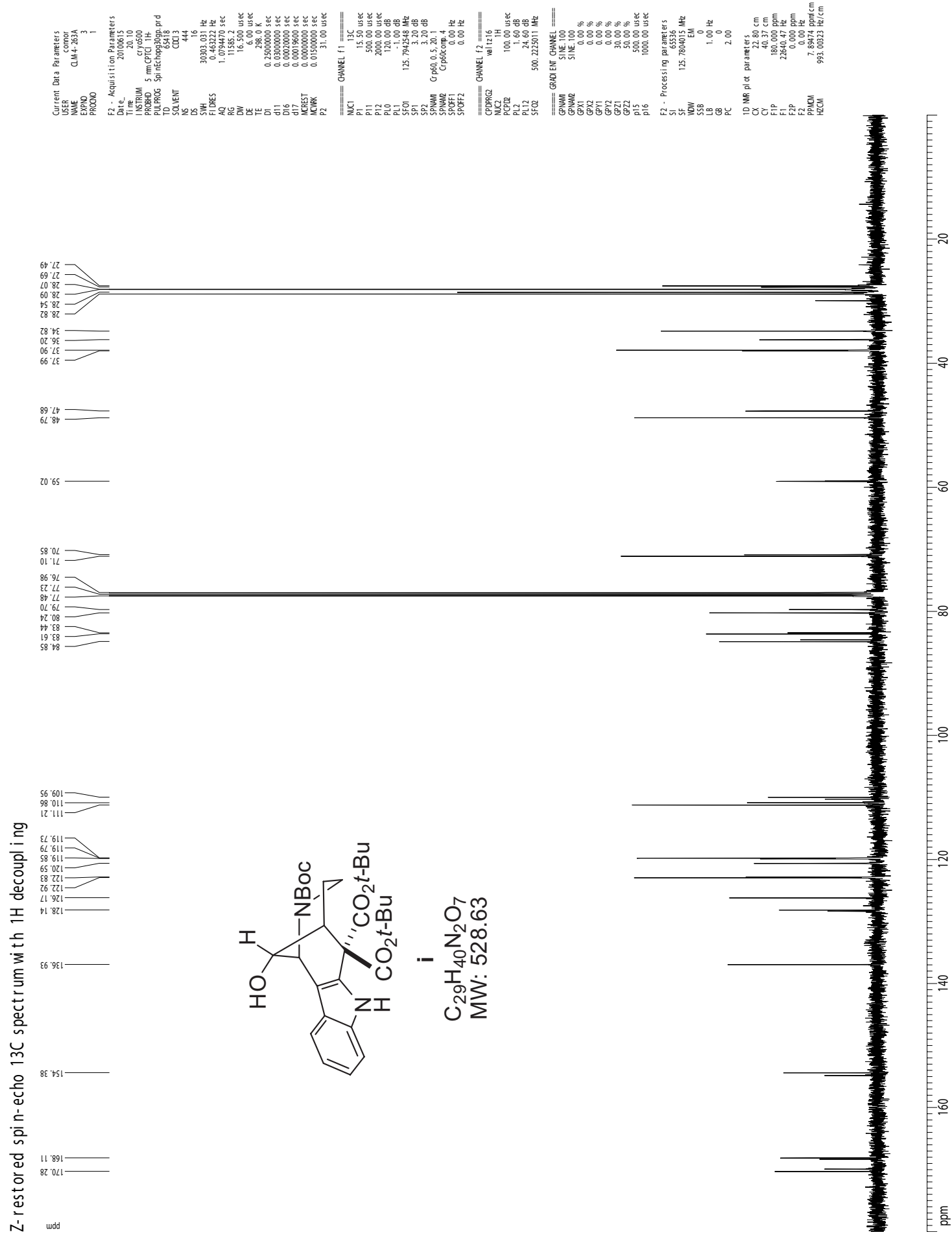
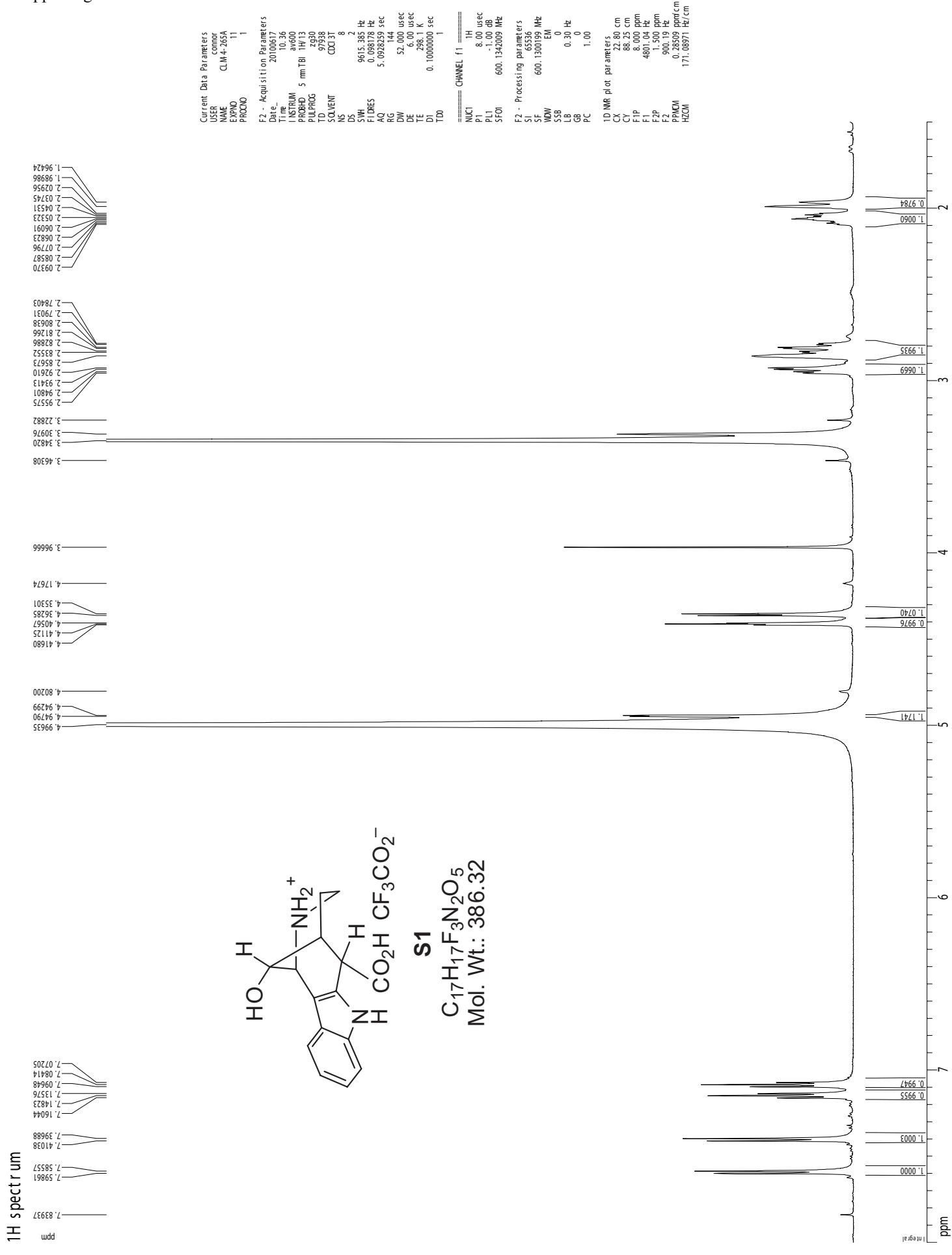
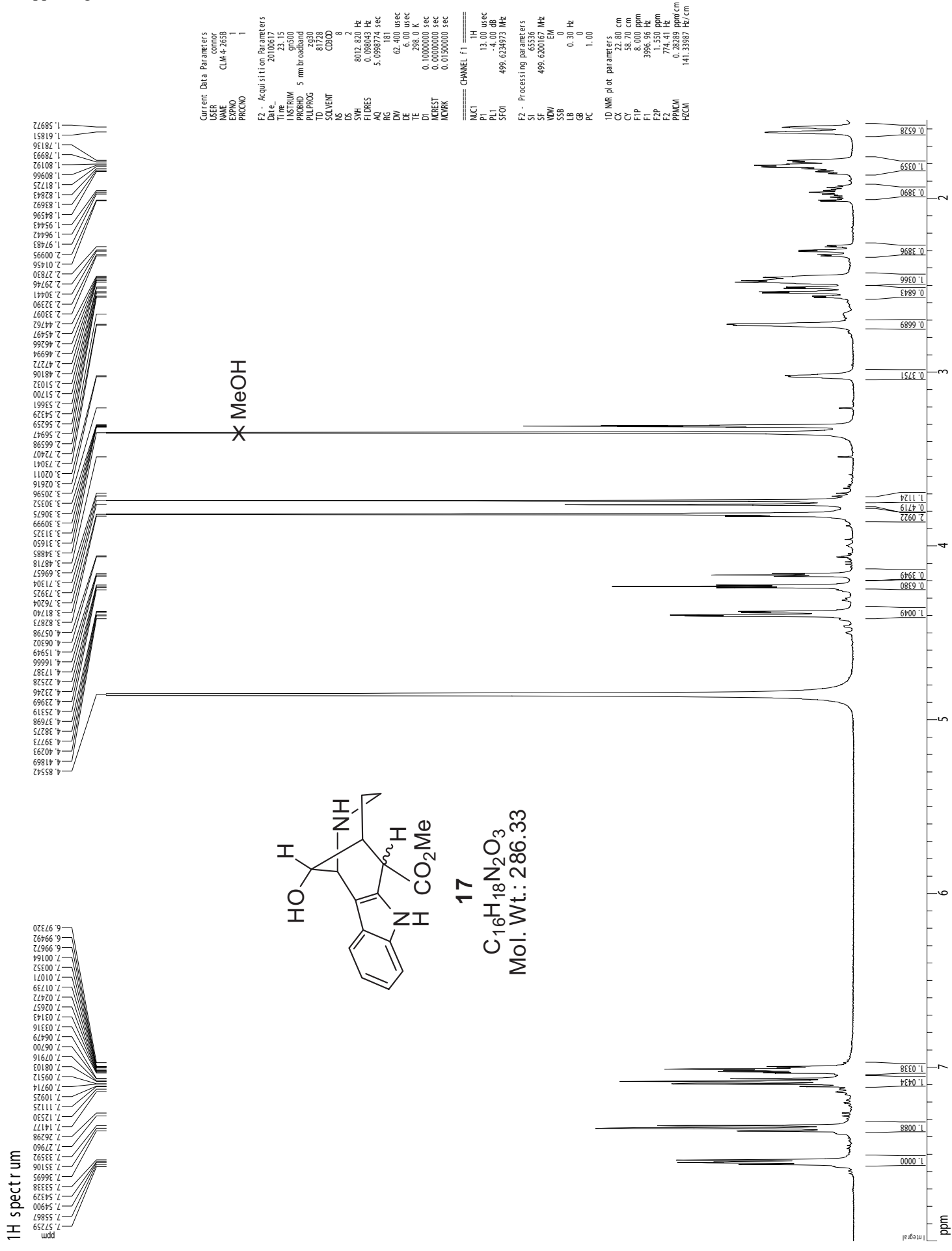
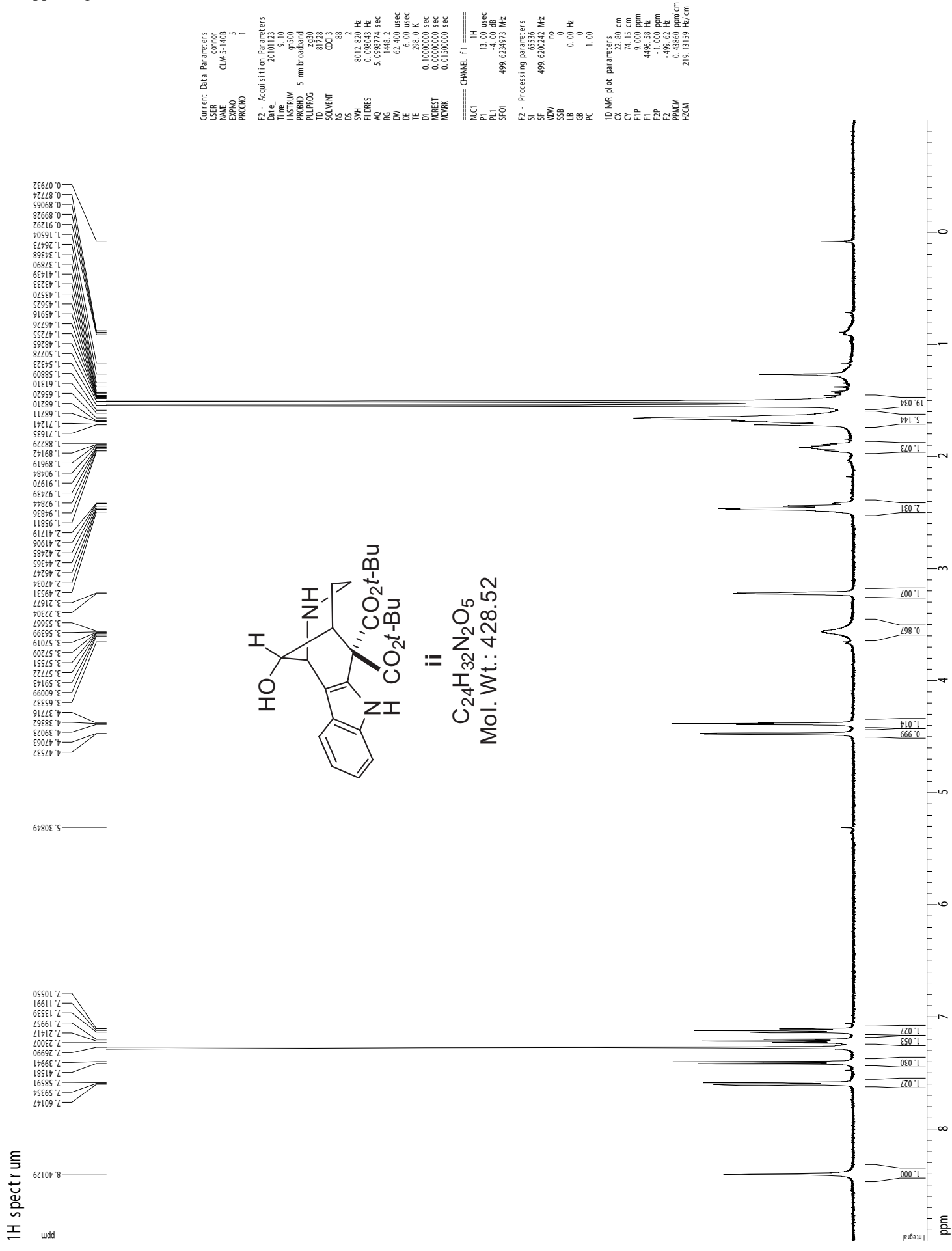
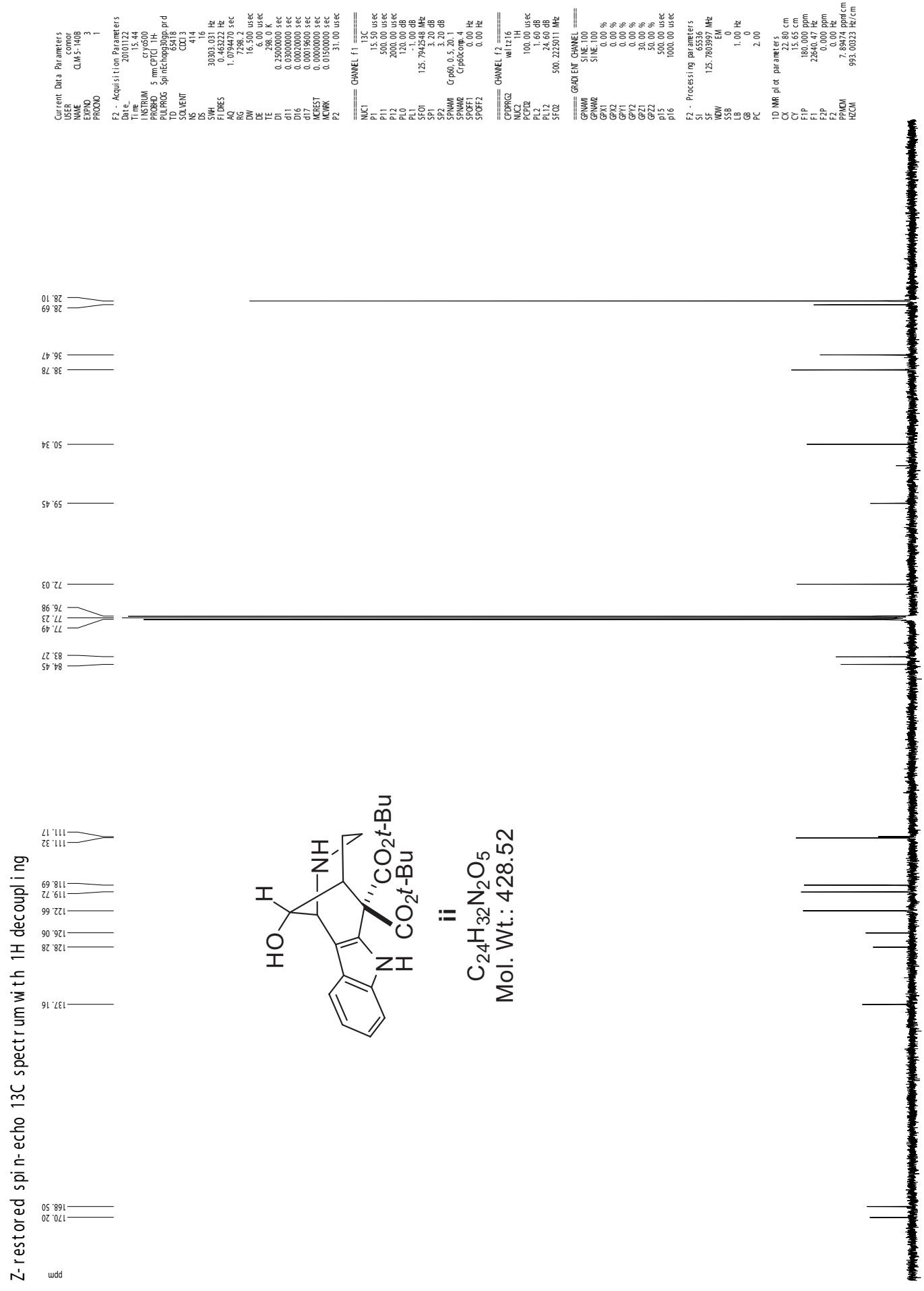
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUCl 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200268 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 96.78 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
0.08229
0.88100
0.89493
1.21957
1.23361
1.26592
1.38057
1.41241
1.46255
1.47512
1.50883
1.54460
1.60361
1.63438
1.64953
1.67098
1.70842
1.73711
1.91278
1.92459
1.93284
1.94228
1.95232
1.96108
1.97131
2.46382
2.47027
2.49122
2.49773
2.51822
2.52517
2.53609
2.54234
2.56333
3.22701
3.48292
3.49679
3.51931
3.53006
3.67948
3.69028
3.70682
3.71738
3.84316
3.84699
4.28275
4.28802
5.57809
5.58415
5.71033
5.71594
7.08572
7.10041
7.11389
7.12776
7.14283
7.18623
7.20292
7.21910
7.23424
7.26994
7.36134
7.37827
7.38426
7.40061
7.67155
7.68735
7.78416
7.80002
8.38706
1.0000
1.0428
1.0319
1.0274
0.6213
0.3842
0.6229
0.3846
1.0106
0.6248
0.6317
0.8210
1.0125
1.0438
1.1348
1.0248
5.4869
8.9045
9.2501
3.6187
HO
H
NBoc
CO2t-Bu
CO2t-Bu
H
N
i
C29H40N2O7
MW: 528.63
1H spectrum
ppm
integral
ppm

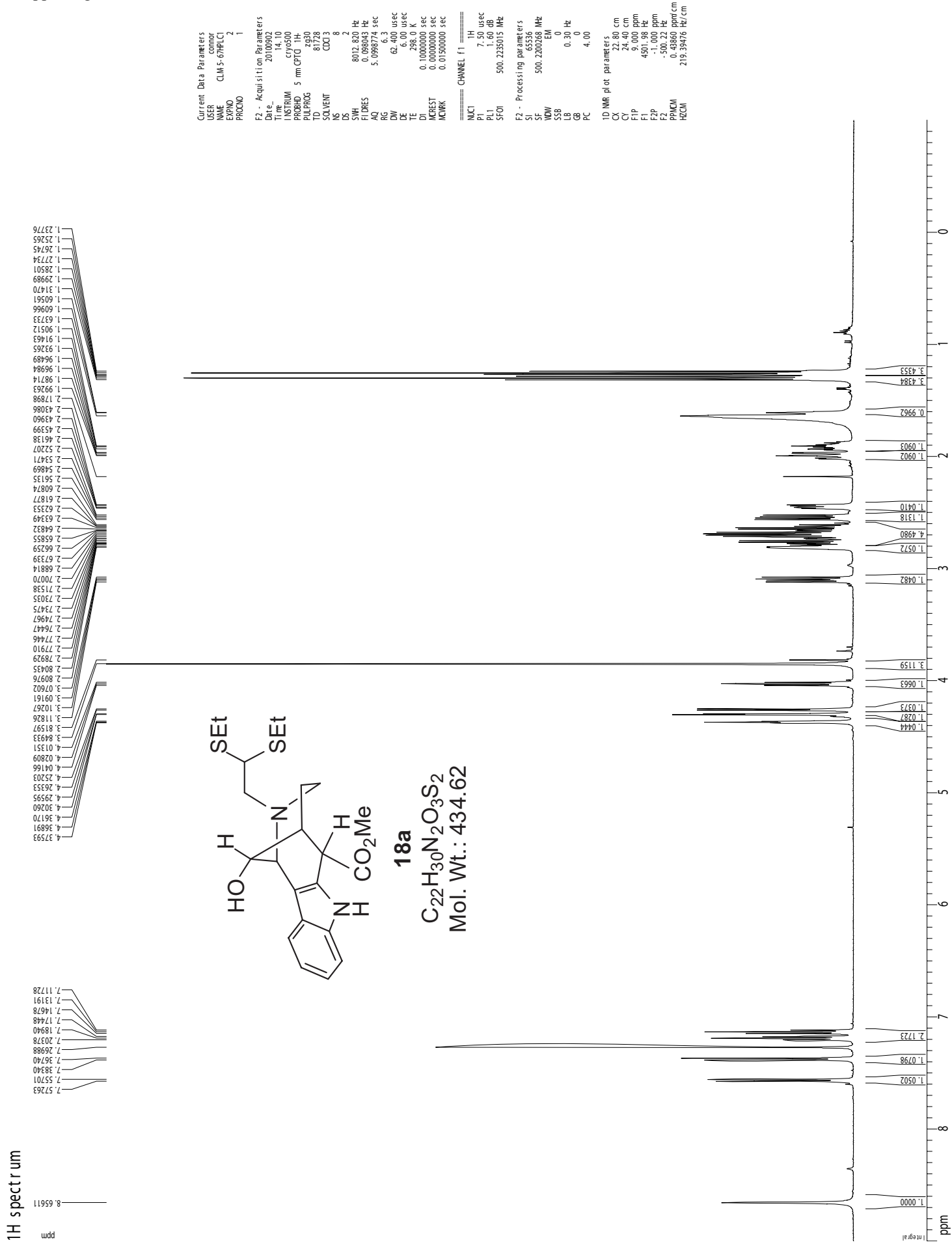

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
HO
H
NBoc
CO2t-Bu
H
N
CO2t-Bu
i
C29H40N2O7
MW: 528.63

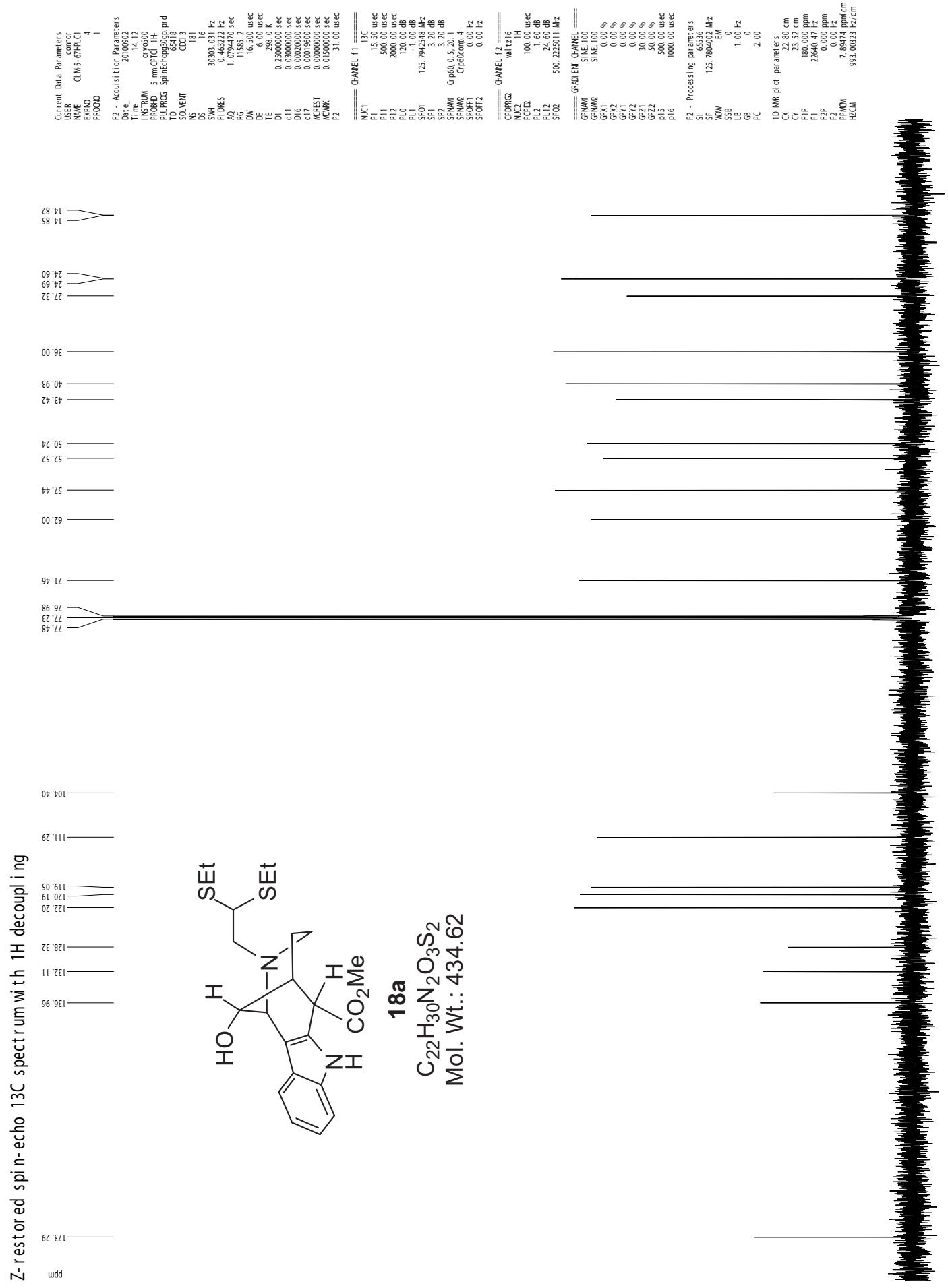

1H spectrum
Current Data Parameters
USER connor
NAME CLM4-265A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100617
Time 10.36
INSTRUM av600
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 97938
SOLVENT CDCl3T
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928359 sec
RG 144
DW 52.000 usec
DE 6.00 usec
TE 298.1 K
D1 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 -1.00 dB
SFO1 600.1342009 MHz
F2 - Processing parameters
SI 65536
SF 600.1300199 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 88.25 cm
F1P 8.000 ppm
F1 4801.04 Hz
F2P 1.500 ppm
F2 900.19 Hz
PPMCM 0.28509 ppm/cm
HZCM 171.08971 Hz/cm
HO
H
NH2+
H
N
H
CO2H
CF3CO2−
S1
C17H17F3N2O5
Mol. Wt.: 386.32
ppm
integral

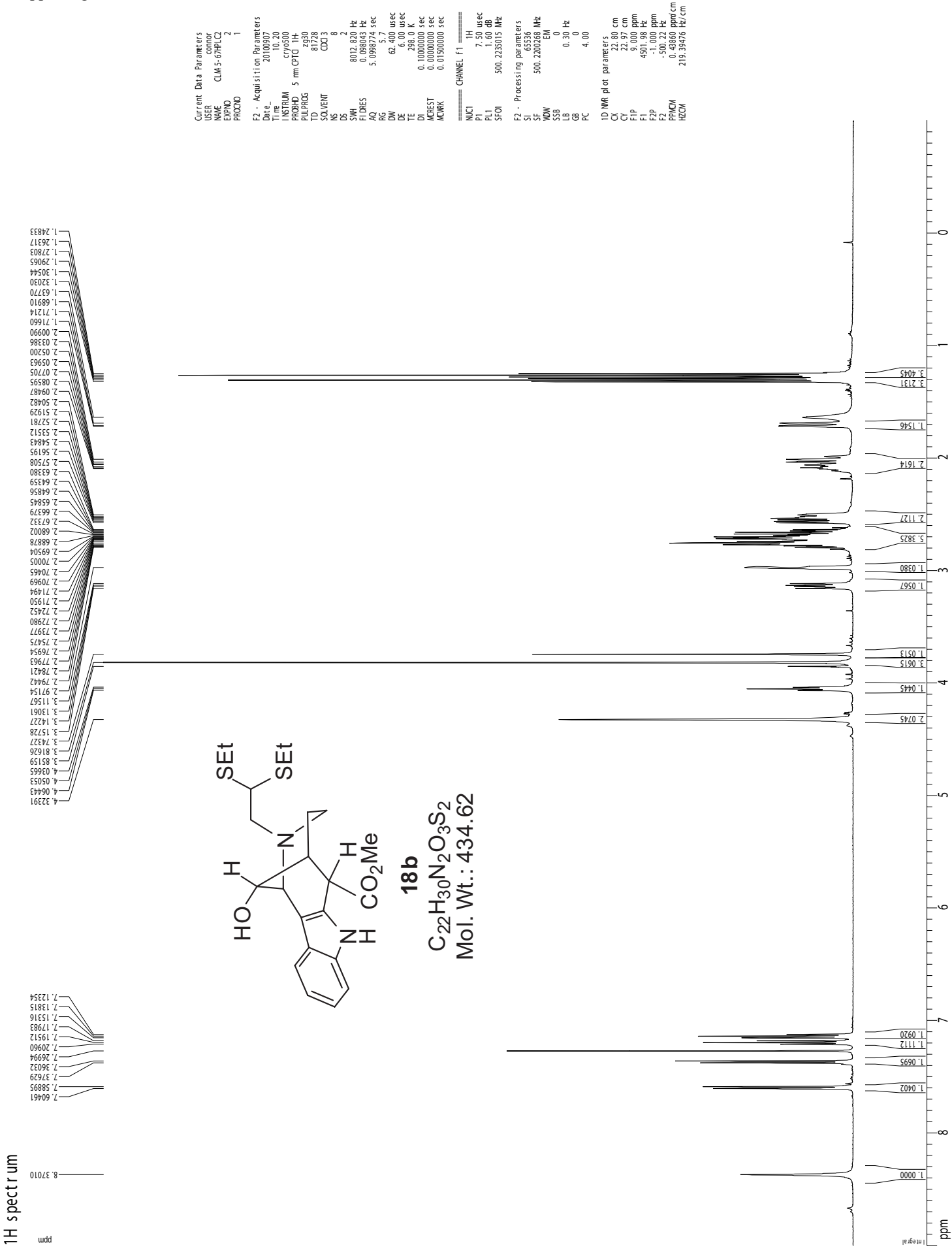

MeOH
17
$C_{16}H_{18}N_2O_3$
Mol. Wt.: 286.33
1H spectrum
ppm

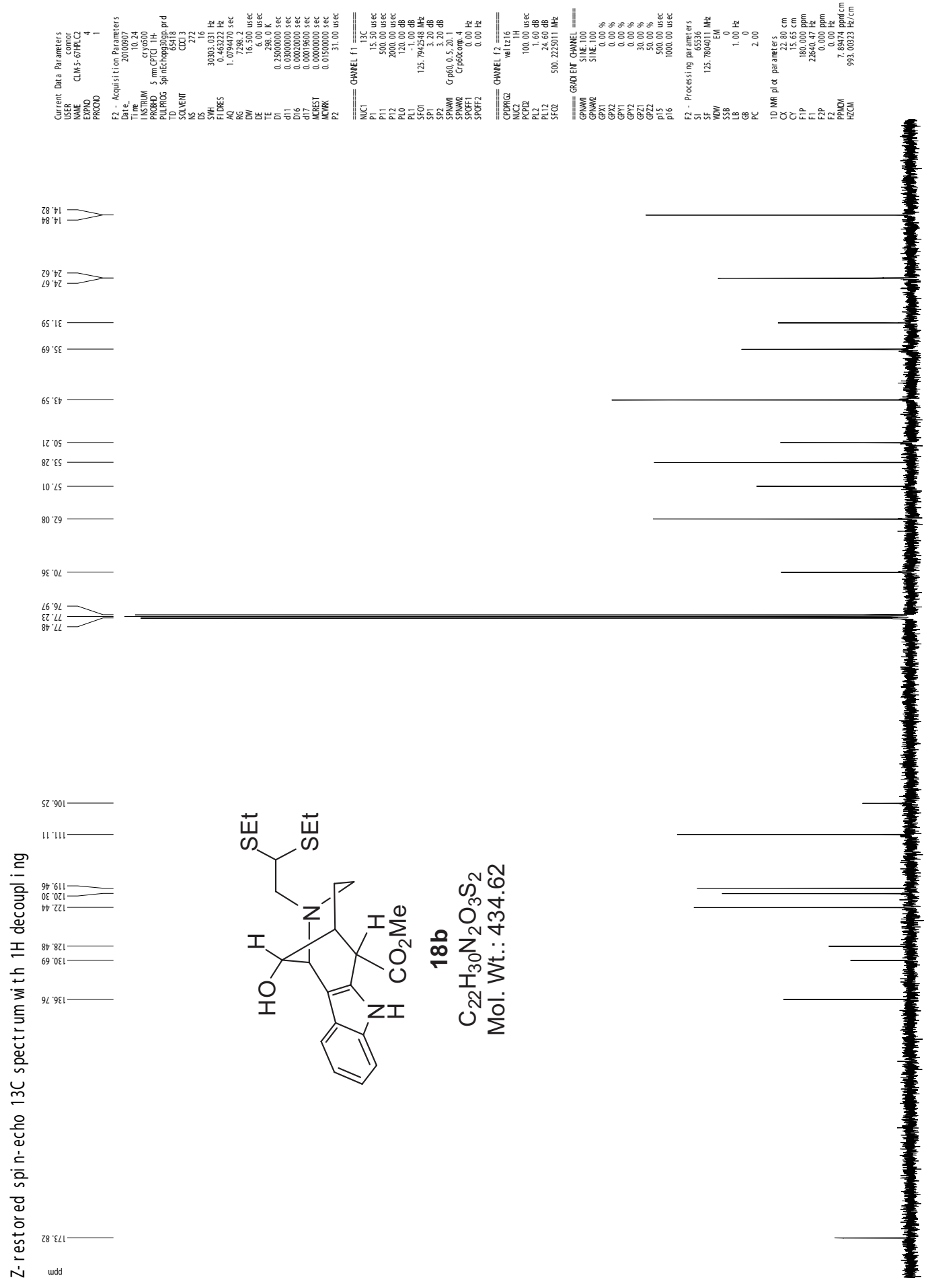

1H spectrum
Current Data Parameters
USER connor
NAME CLM-5-140B
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101123
Time 9.10
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 88
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 1448.2
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 13.00 usec
PL1 -4.00 dB
SFO1 499.6234973 MHz
F2 - Processing parameters
SI 65536
SF 499.6200242 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 24.15 cm
F1P 9.000 ppm
F1 4496.58 Hz
F2P -1.000 ppm
F2 -499.62 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.13159 Hz/cm
ii
C24H32N2O5
Mol. Wt.: 428.52
integral
ppm

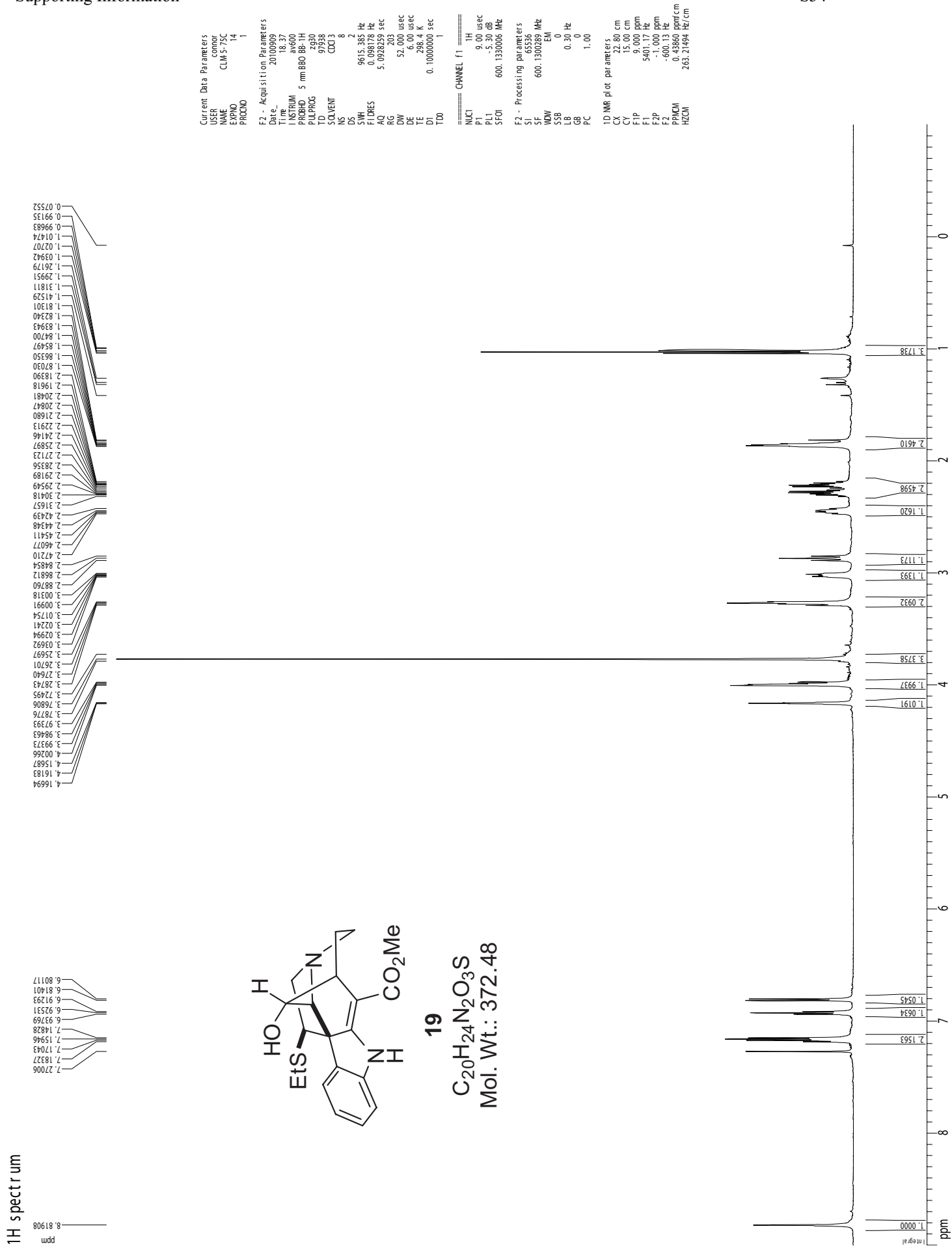

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER connor
NAME CLM5-1408
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101122
Time 15.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65418
SOLVENT CDCl3
NS 414
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 7296.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM0 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
170.20
168.50
137.16
128.28
126.06
122.66
119.72
118.69
111.32
111.17
84.45
83.27
77.49
77.23
76.98
72.03
59.45
50.34
38.78
36.47
28.69
28.10
HO
H
NH
CO2t-Bu
CO2t-Bu
N
H
ii
C24H32N2O5
Mol. Wt.: 428.52
ppm

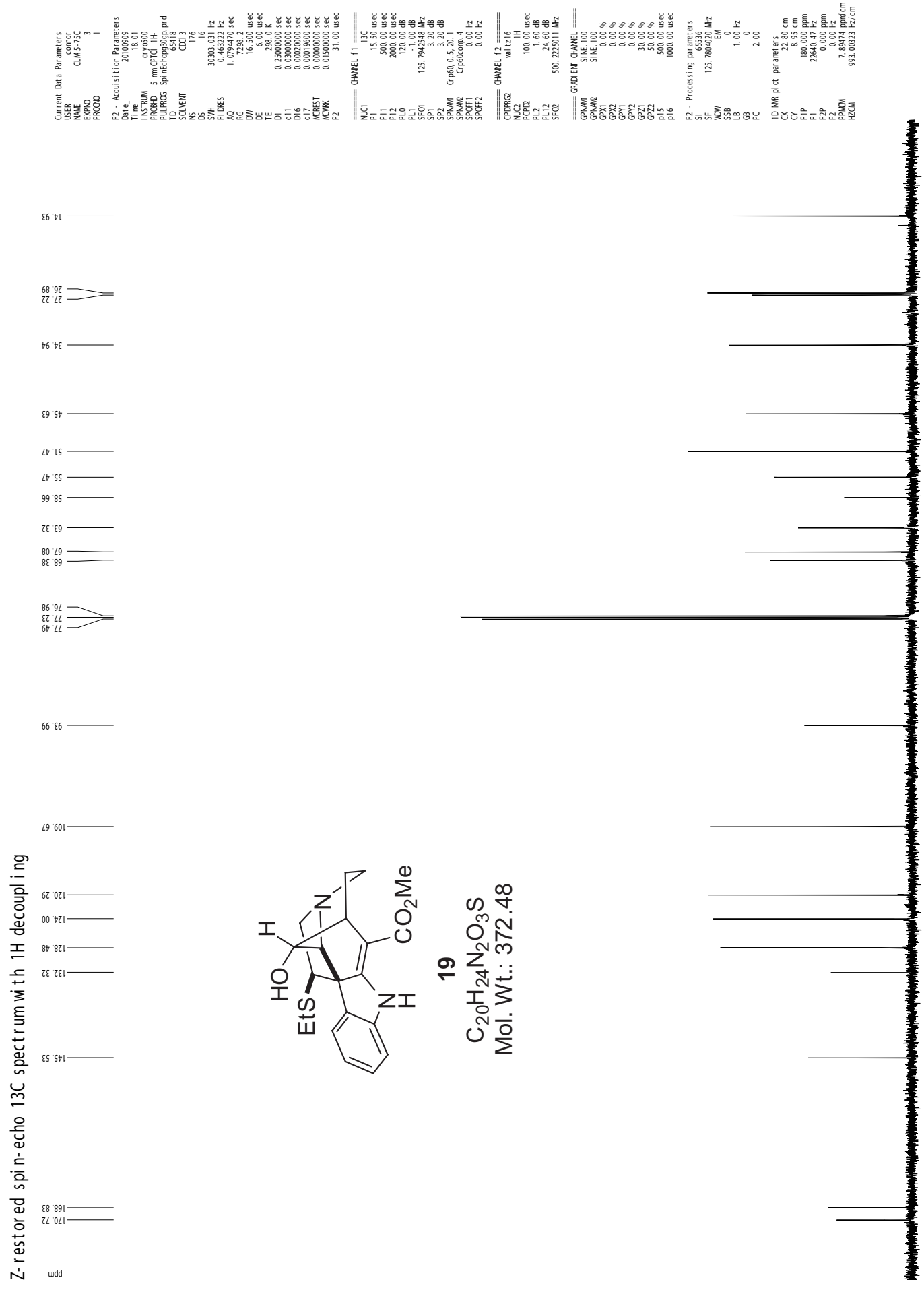

1H spectrum
Current Data Parameters
USER connor
NAME CLM-5-67HPLC1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100902
Time 14.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200268 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 24.40 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
SEt
SEt
H
N
HO
H
N
H
CO2Me
18a
C22H30N2O3S2
Mol. Wt.: 434.62
integral
ppm

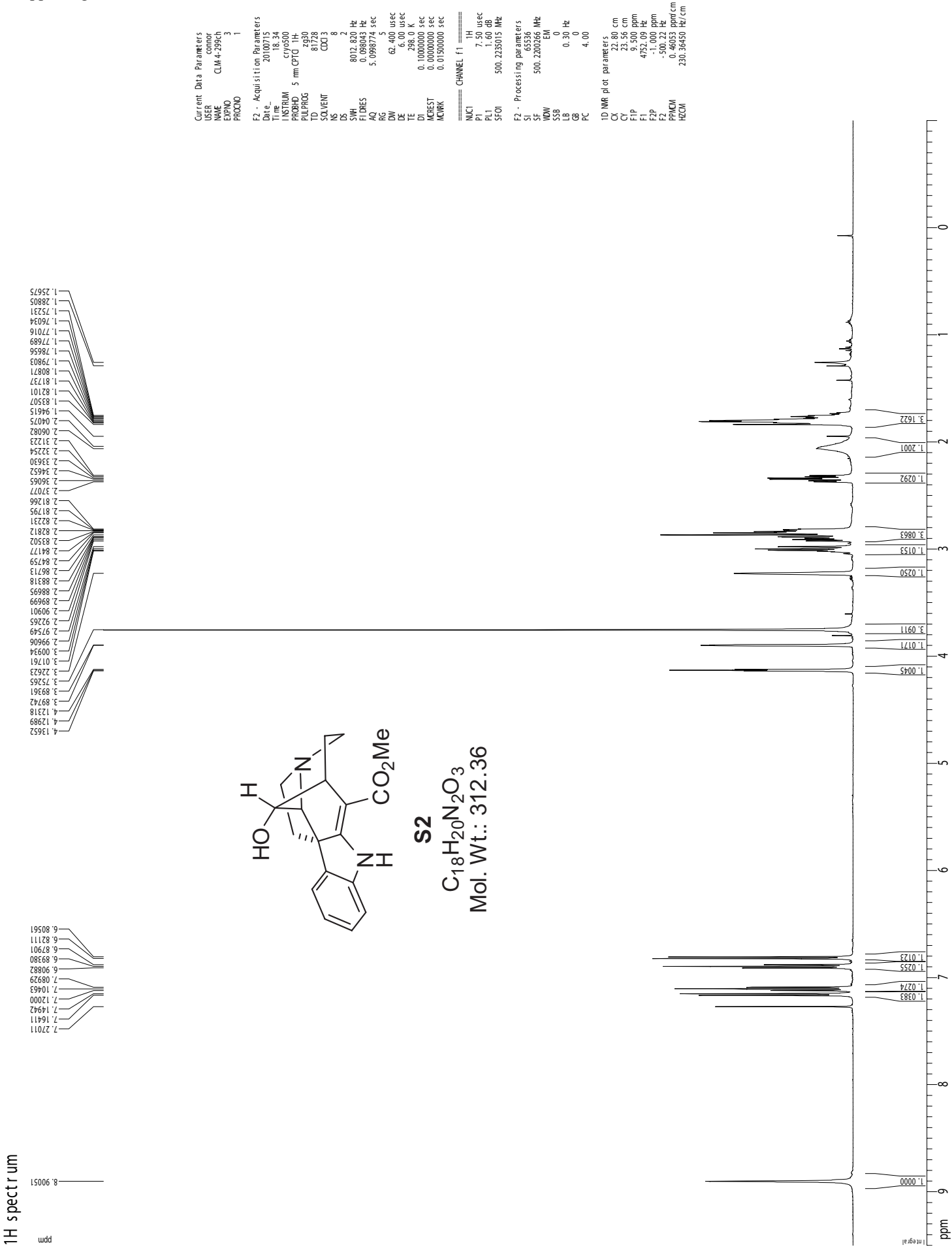

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER connor
NAME CLM5-67HPLC1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100502
Time 14.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG spiechopg30gp.prd
TD 65418
SOLVENT CDCl3
NS 181
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM0 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 23.52 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
14.85
14.82
24.60
24.69
27.32
36.00
40.93
43.42
50.24
52.52
57.44
62.00
71.46
76.98
77.23
77.48
104.40
111.29
119.05
120.19
122.20
128.32
132.11
136.96
173.29
SEt
SEt
N
H
HO
H
CO2Me
N
H
18a
C22H30N2O3S2
Mol. Wt.: 434.62
ppm

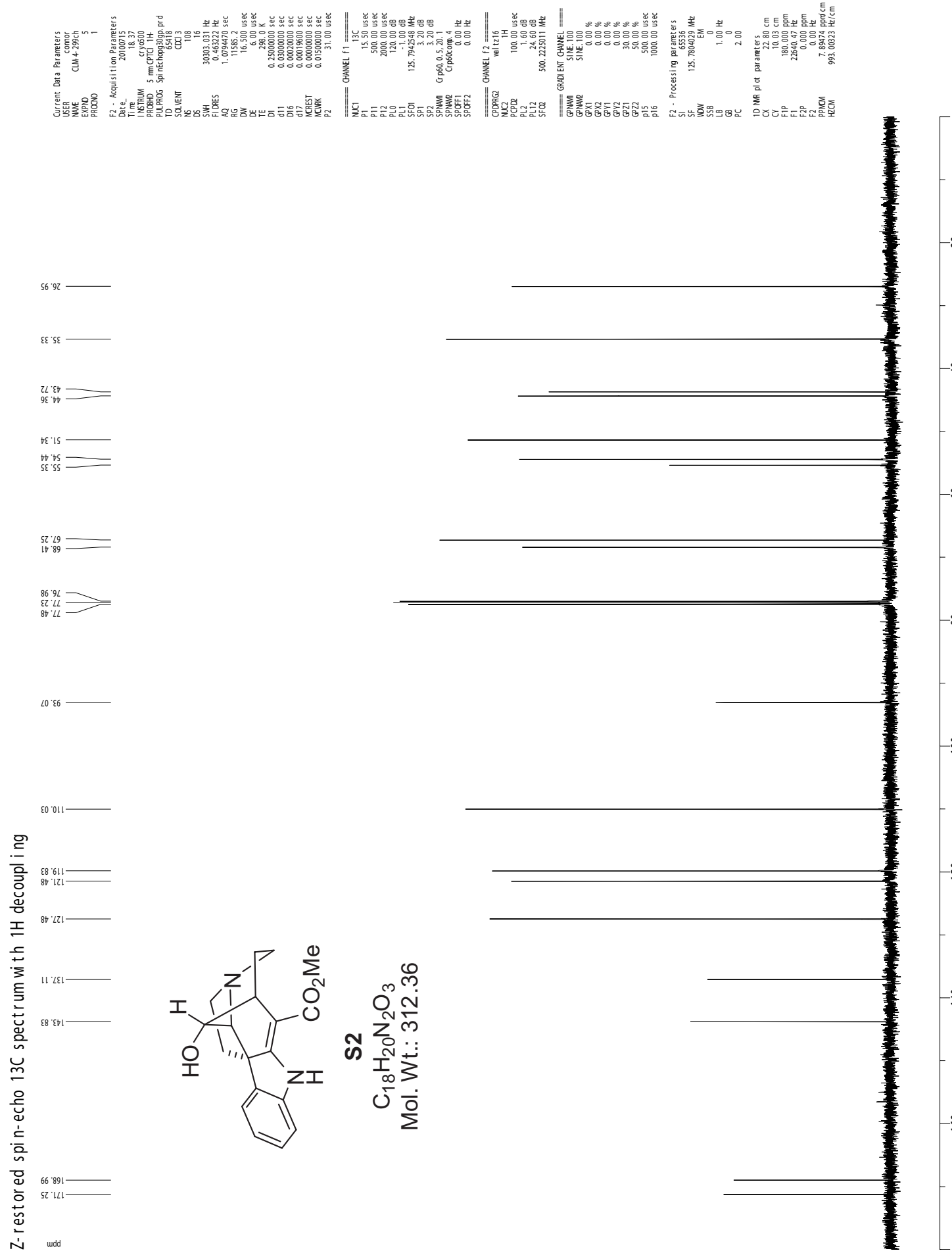

1H spectrum
Current Data Parameters
USER connor
NAME CLM-5-67HPLC2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100907
Time 10.20
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUCl 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200268 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 22.97 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
SEt
SEt
N
H
HO
H
CO2Me
N
H
18b
C22H30N2O3S2
Mol. Wt.: 434.62
integral
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER connor
NAME CLM5-67HPLC2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100507
Time 10.24
INSTRUM cryds500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchop3Dgp.prd
TD 65418
SOLVENT CDCl3
NS 272
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 7296.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804011 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
173.82
136.76
130.69
128.48
122.44
120.30
119.46
111.11
106.25
77.48
77.23
76.97
70.36
62.08
57.01
53.28
50.21
43.59
35.69
31.59
24.67
14.84
14.82
SEt
SEt
H
HO
N
H
N
H
CO2Me
18b
C22H30N2O3S2
Mol. Wt.: 434.62
ppm

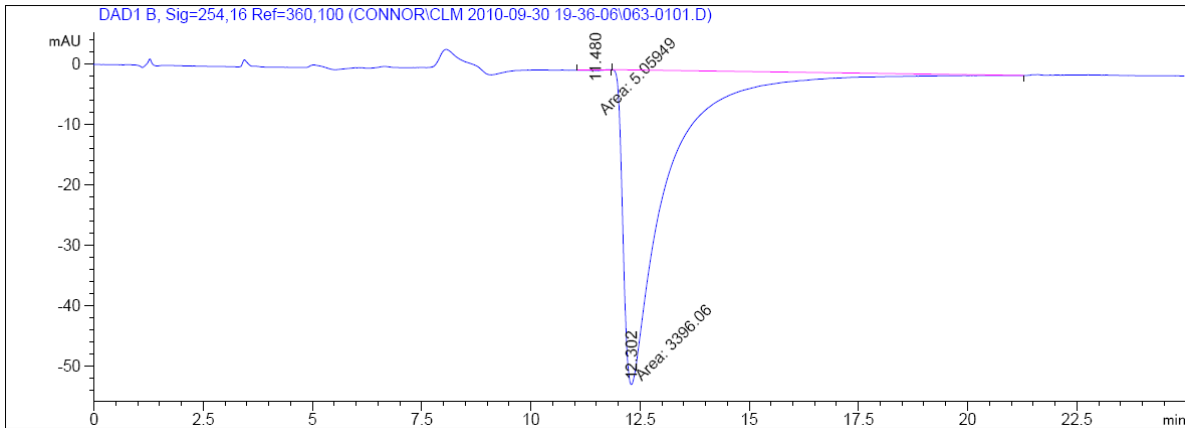

1H spectrum
Current Data Parameters
USER connor
NAME CLM5-75C
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100909
Time 16.37
INSTRUM av600
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 97938
SOLVENT CDCl3
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928359 sec
RG 203
DW 52.000 usec
DE 6.00 usec
TE 298.4 K
D1 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -5.30 dB
SFO1 600.1330006 MHz
F2 - Processing parameters
SI 65536
SF 600.1300289 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 5401.17 Hz
F2P -1.000 ppm
F2 -600.13 Hz
PPMCM 0.43860 ppm/cm
HZCM 263.21494 Hz/cm
HO
EtS
H
N
N
H
CO2Me
19
C20H24N2O3S
Mol. Wt.: 372.48
ppm
Integral


Z-restored spin-echo 13C spectrum with 1H decoupling
Current Data Parameters
USER connor
NAME CLM-5-75C
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100909
Time 18.01
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchop3Dgp.prd
TD 65418
SOLVENT CDCl3
NS 176
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 7296.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM0 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 8.95 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
14.93
26.89
27.22
34.94
45.63
51.47
55.47
58.66
63.32
67.08
68.38
76.98
77.23
77.49
93.99
109.67
120.29
124.00
128.48
132.32
145.53
168.83
170.72
ppm
20
40
60
80
100
120
140
160
ppm
H
HO
EtS
N
N
H
CO2Me
19
C20H24N2O3S
Mol. Wt.: 372.48


1H spectrum
Current Data Parameters
USER connor
NAME CLM4-299ch
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100715
Time 18.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200266 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 23.56 cm
F1P 9.500 ppm
F1 4752.09 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.46053 ppm/cm
HZCM 230.36450 Hz/cm
S2
C18H20N2O3
Mol. Wt.: 312.36
HO
H
N
CO2Me
N
H
integral
ppm

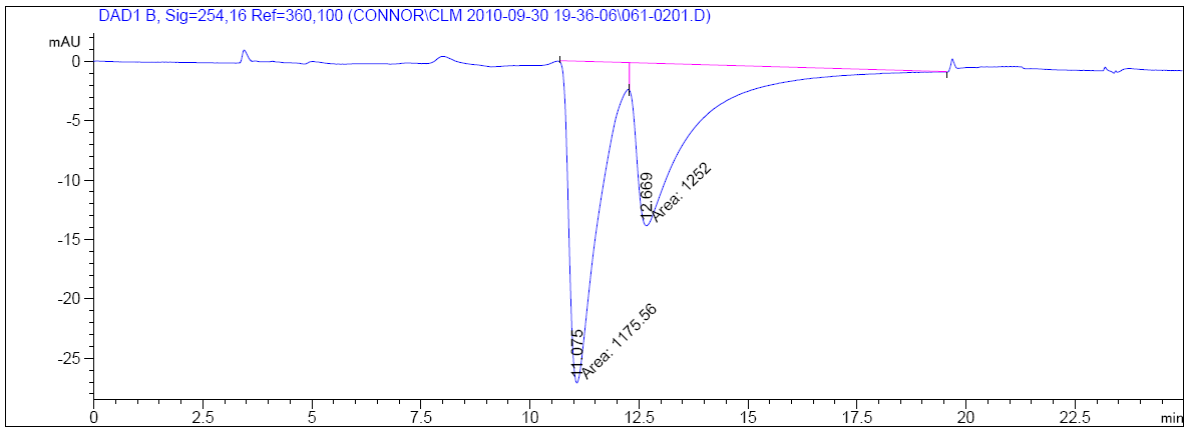

Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
168.99
171.25
143.83
137.11
127.48
121.48
119.83
110.03
93.07
77.48
77.23
76.98
68.41
67.25
55.35
54.44
51.34
44.36
43.72
35.33
26.95
HO
H
N
N
H
CO2Me
S2
C18H20N2O3
Mol. Wt.: 312.36
Current Data Parameters
USER connor
NAME CLM-4-299ch
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100715
Time 18.37
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gpo.prd
TD 65418
SOLVENT CDCl3
NS 108
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM0 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804029 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 12.80 cm
CY 10.03 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
ppm
20
40
60
80
100
120
140
160

HPLC chromatogram of (+)-**S2**



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.480	MM N	0.4782	5.05949	1.76351e-1	0.1488
2	12.302	PM N	1.0859	3396.06201	52.12215	99.8512

HPLC chromatogram of (±)-**S2**



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.075	MF N	0.7232	1175.56458	27.09213	48.4256
2	12.669	FM N	1.5239	1252.00403	13.69289	51.5744

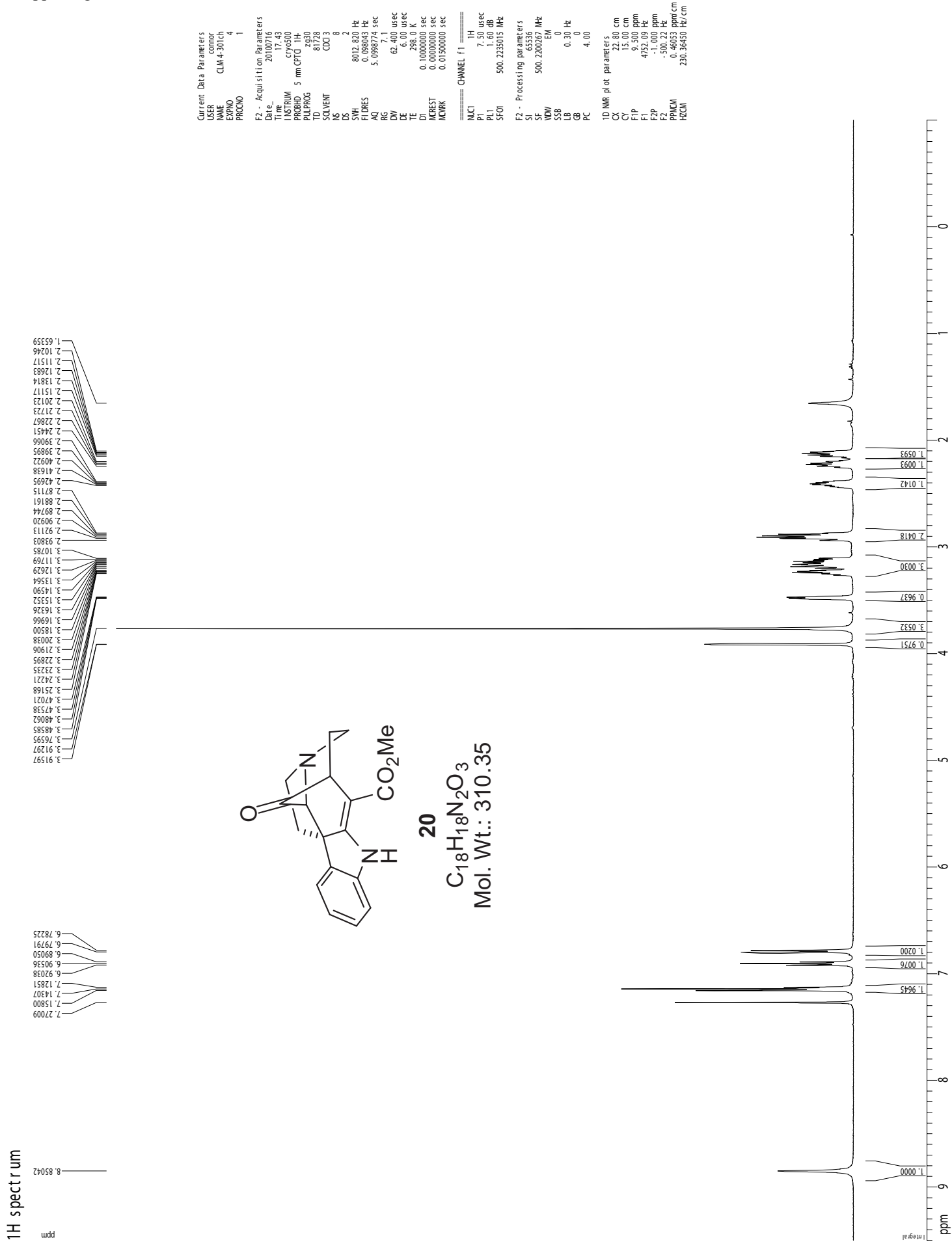
Conditions:

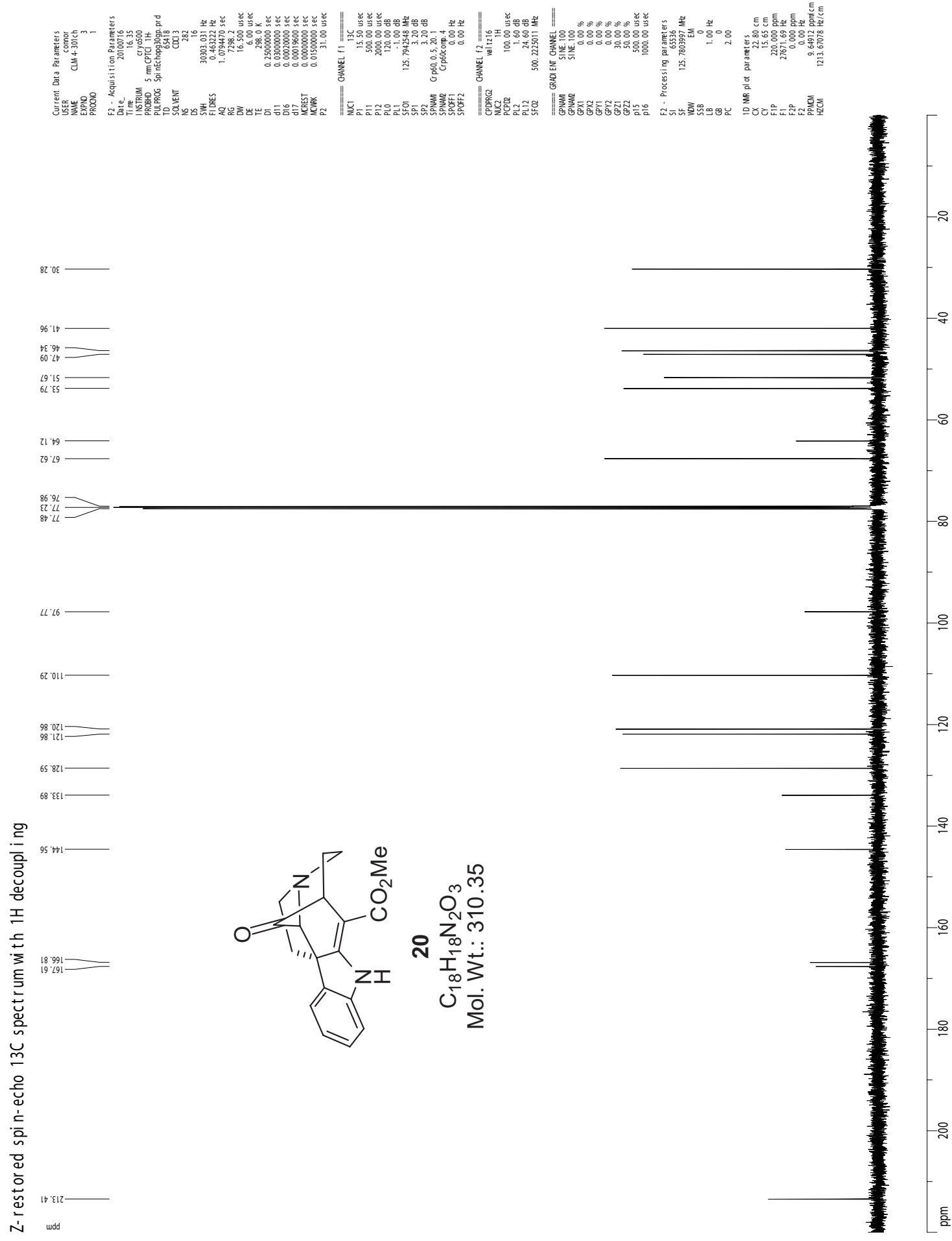
Column: Chiralpak AD

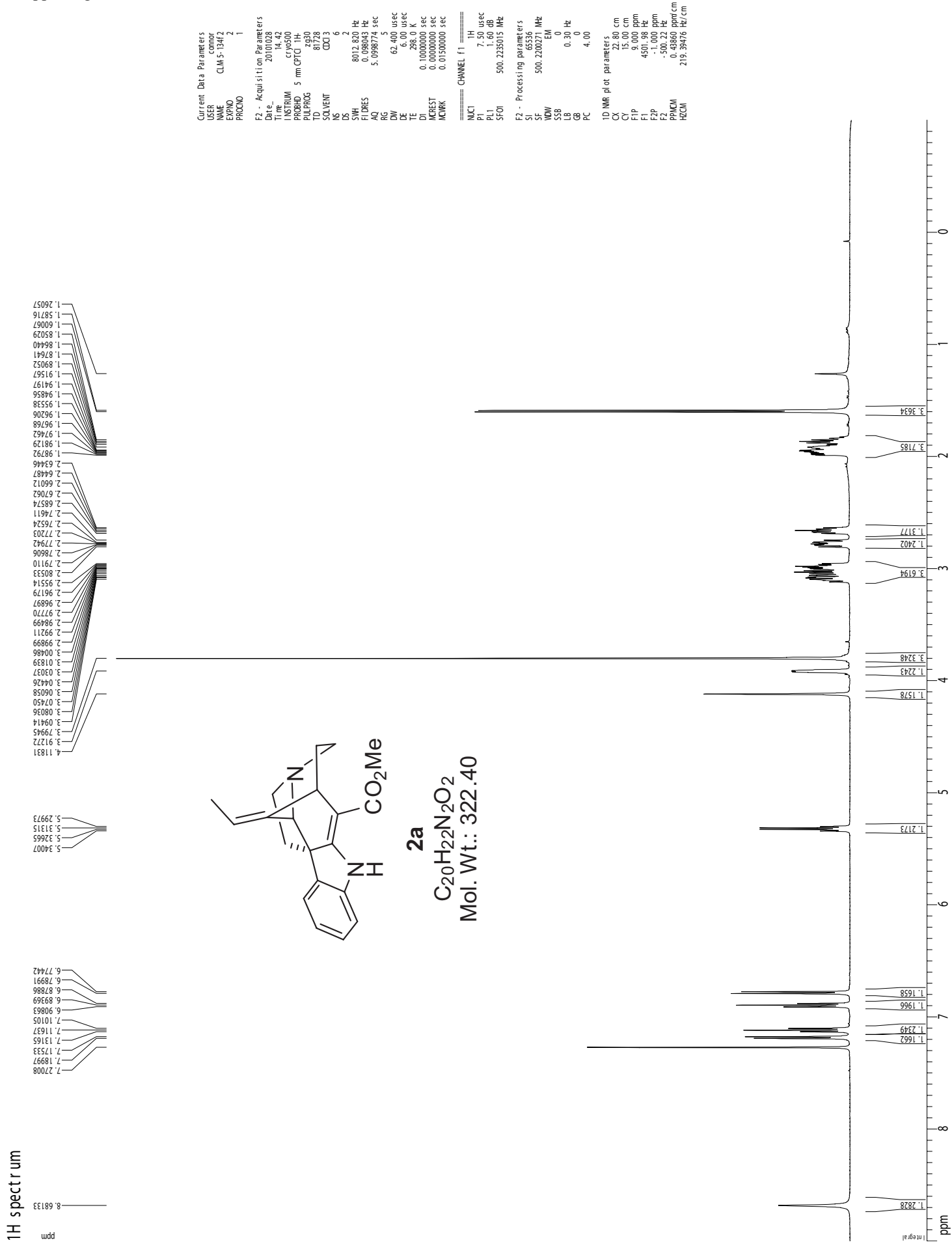
Flow: 1.0 mL/min

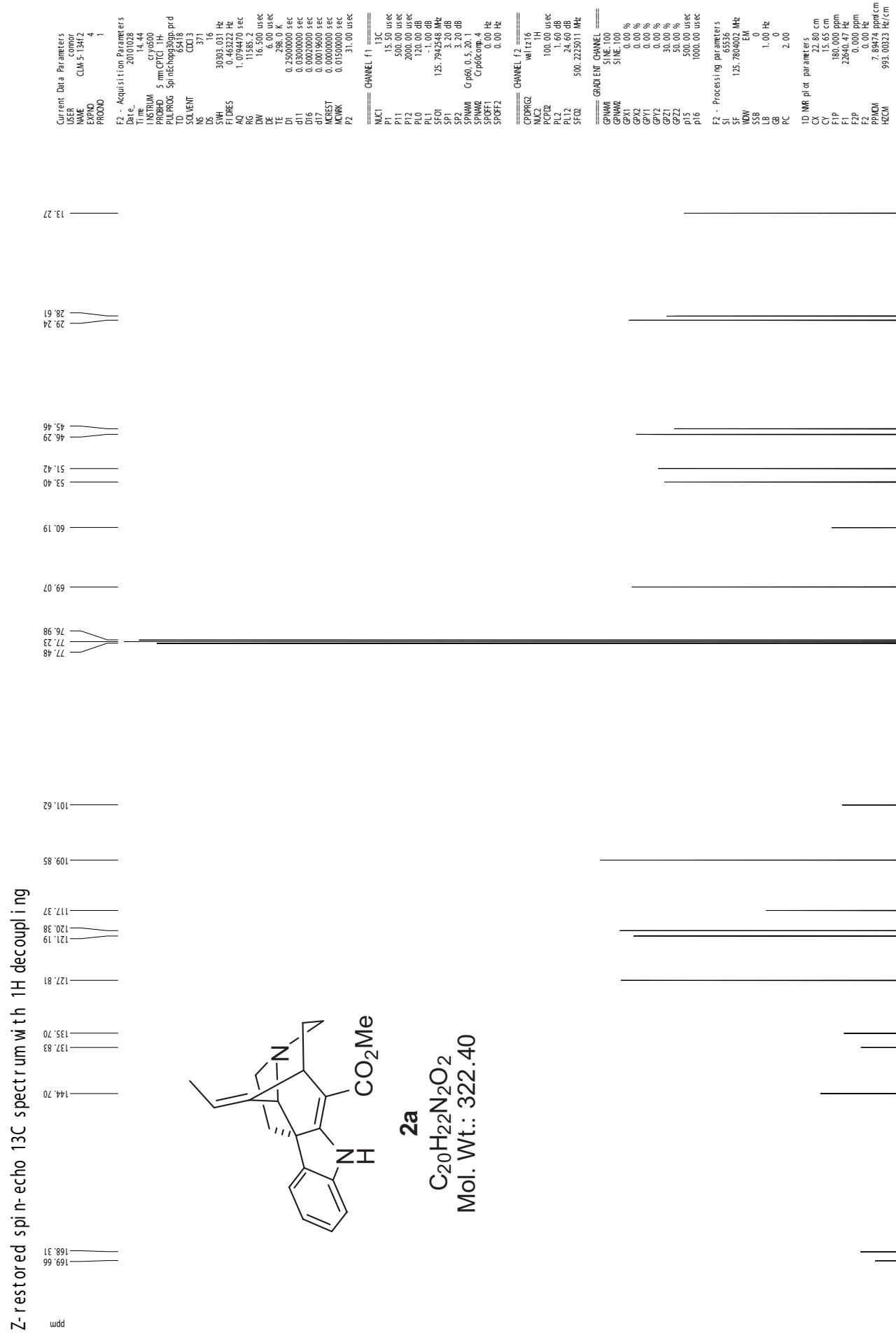
Mobile Phase: 70:30 *n*-hexanes:i-PrOH

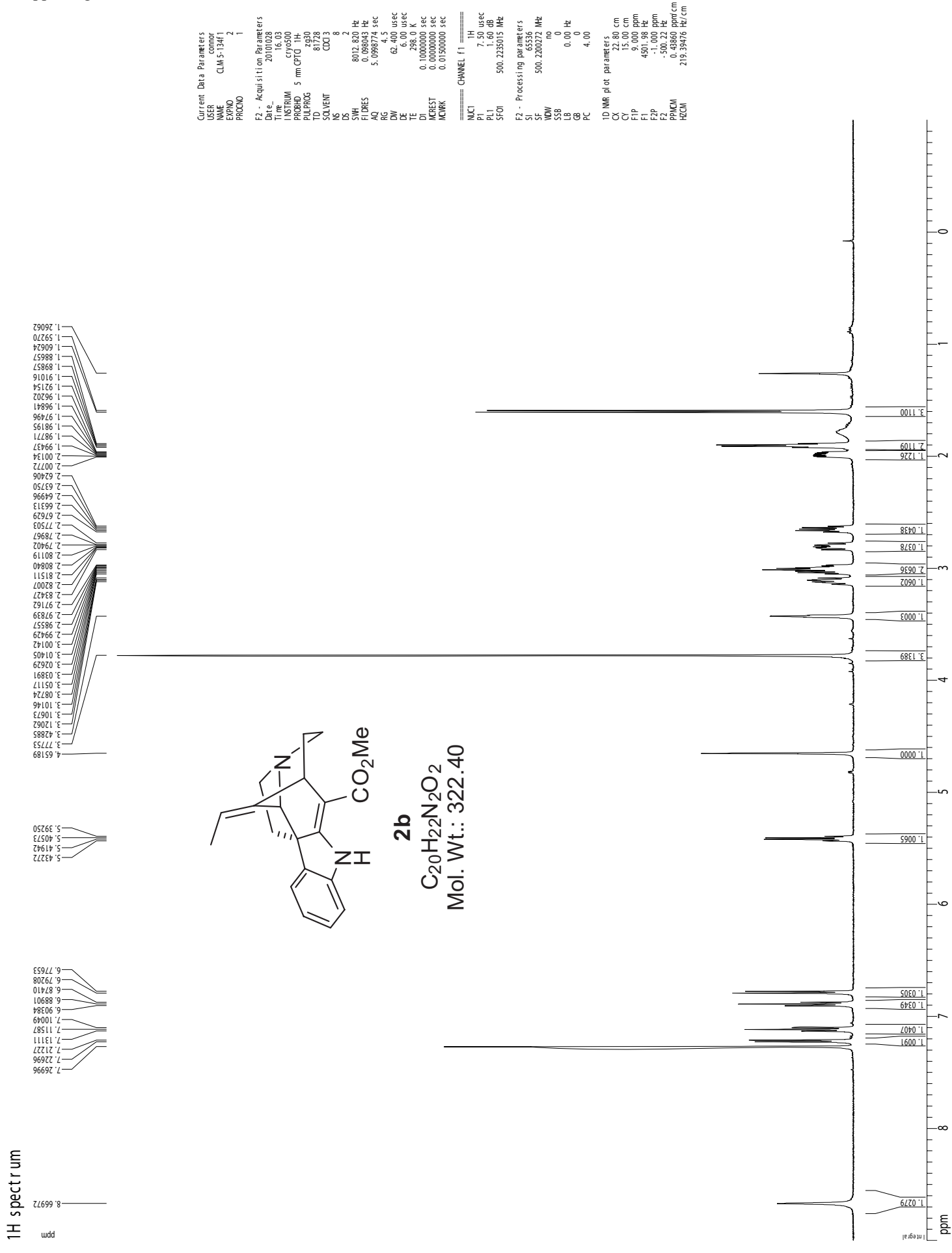
Wavelength: 254 nm

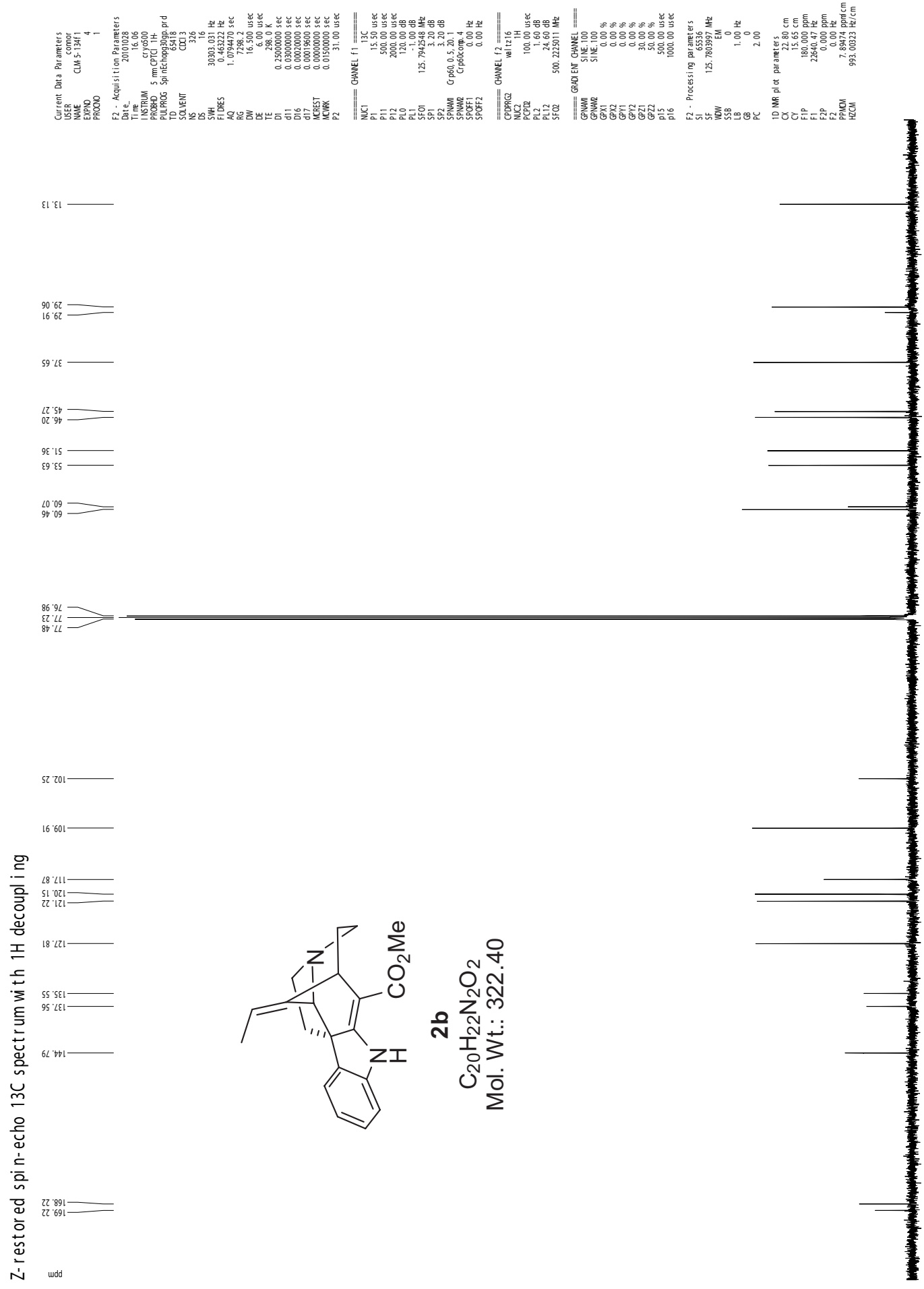

1H spectrum
Current Data Parameters
USER connor
NAME CLM-4-301ch
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100716
Time 17.43
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200267 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.500 ppm
F1 4752.09 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.46055 ppm/cm
HZCM 230.36450 Hz/cm
O
N
CO2Me
N
H
20
C18H18N2O3
Mol. Wt.: 310.35
integral
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
213.41
166.81
167.61
144.56
133.89
128.59
121.86
120.86
110.29
97.77
77.48
77.23
76.98
67.62
64.12
53.79
51.67
47.09
46.34
41.96
30.28
Current Data Parameters
USER connor
NAME CLM-4-301ch
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100716
Time 16.35
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchop3gpp rd
TD 65418
SOLVENT CDCl3
NS 282
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 7296.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 12.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.69912 ppm/cm
HZCM 1213.67078 Hz/cm
O
N
N
H
CO2Me
20
C18H18N2O3
Mol. Wt.: 310.35
ppm
20
40
60
80
100
120
140
160
180
200


1H spectrum
Current Data Parameters
USER connor
NAME CLM-5-134f2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101028
Time 14.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 6
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200271 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
CO2Me
N
H
2a
C20H22N2O2
Mol. Wt.: 322.40
ppm
integral


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER connor
NAME CLM-13Hf2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101028
Time 14.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchop3Dgp.prd
TD 65418
SOLVENT CDCl3
NS 371
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL ====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 12.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
13.27
28.61
29.24
45.46
46.29
51.42
53.40
60.19
69.07
76.98
77.23
77.48
101.62
109.85
117.37
120.38
121.19
127.81
135.70
137.83
144.70
168.31
169.66
20
40
60
80
100
120
140
160
ppm
2a
$C_{20}H_{22}N_2O_2$
Mol. Wt.: 322.40
N
H
CO_2Me

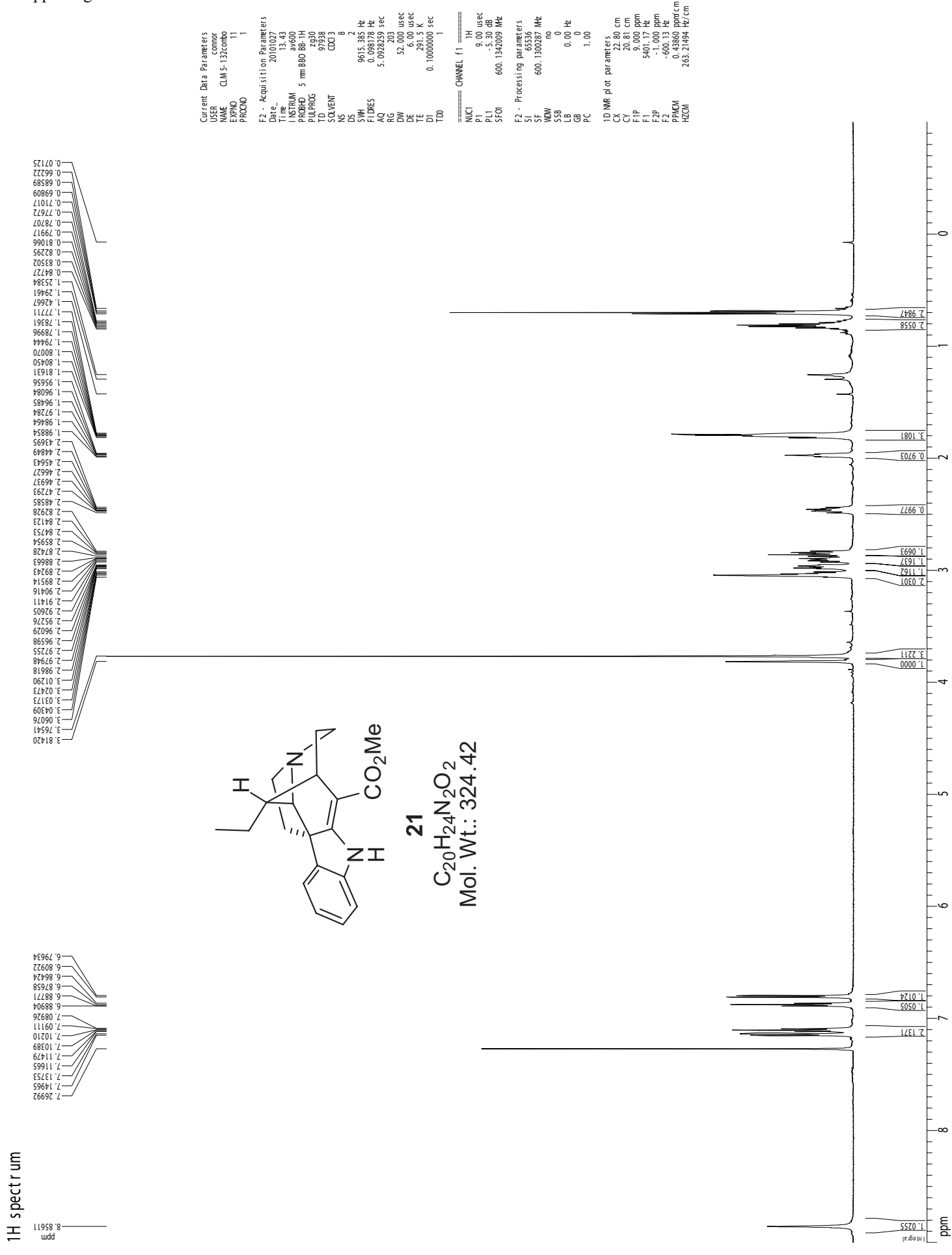

1H spectrum
Current Data Parameters
USER connor
NAME CLM-5-134f1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101028
Time 16.03
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200272 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.000 ppm
F2 -500.22 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
CO2Me
N
N
H
2b
C20H22N2O2
Mol. Wt.: 322.40
ppm
integral

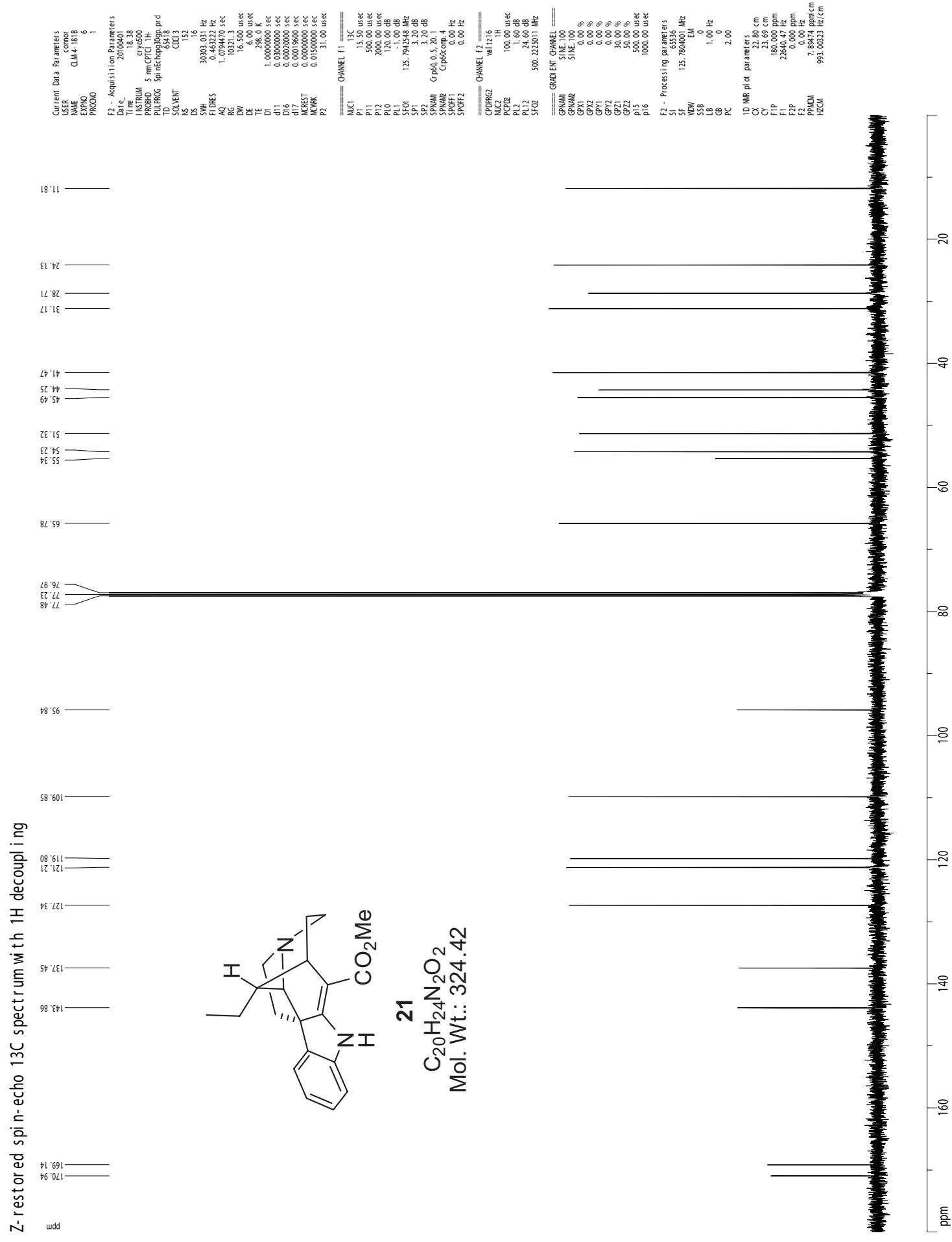


2b

$C_{20}H_{22}N_2O_2$

Mol. Wt.: 322.40


1H spectrum
Current Data Parameters
USER connor
NAME CLM5-132combo
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101027
Time 13.43
INSTRUM av600
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 97938
SOLVENT CDCl3
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928359 sec
RG 203
DW 52.000 usec
DE 6.00 usec
TE 291.5 K
D1 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -5.30 dB
SFO1 600.1342009 MHz
F2 - Processing parameters
SI 65536
SF 600.1300287 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.80 cm
CY 20.81 cm
F1P 9.000 ppm
F1 5401.17 Hz
F2P -1.000 ppm
F2 -600.13 Hz
PPMCM 0.43860 ppm/cm
HZCM 263.21494 Hz/cm
21
C20H24N2O2
Mol. Wt.: 324.42
CO2Me
H
N
N
H
integral
ppm


Z-restored spin-echo 13C spectrum with 1H decoupling
ppm
Current Data Parameters
USER connor
NAME CLM4-1818
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100401
Time 18.38
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65418
SOLVENT CDCl3
NS 152
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 31.00 usec
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7804001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 23.69 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00323 Hz/cm
11.81
24.13
28.71
31.17
41.47
44.25
45.49
51.32
54.23
55.34
65.78
76.97
77.23
77.48
95.84
109.85
119.80
121.21
127.34
137.45
143.86
169.14
170.94
H
N
N
H
CO2Me
21
C20H24N2O2
Mol. Wt.: 324.42
ppm