Supporting Information for:

Convergency and Divergency as Strategic Elements in Total Synthesis: The Total Synthesis of (–)-Drupacine and the Formal Total Synthesis of (±)-Cephalotaxine, (–)-Cephalotaxine and (+)-Cephalotaxine

Qi Liu, Eric M. Ferreira, Brian M. Stoltz*

The Arnold and Mabel Beckman Laboratories of Chemical Synthesis, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125, USA

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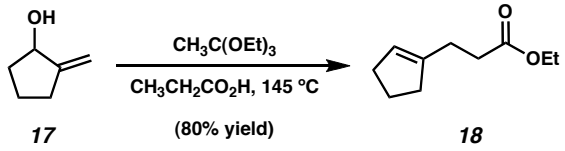
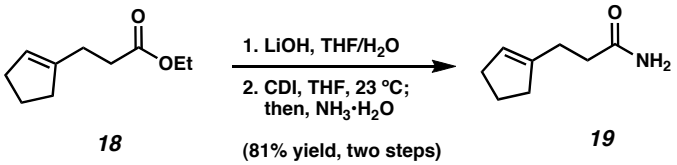
Materials and Methods. Unless stated otherwise, reactions were performed in flame-dried glasswares under a nitrogen or an argon atmosphere, using anhydrous solvents (either freshly distilled or passed through activated alumina columns). All other commercially obtained reagents were used as received. Reaction temperatures were controlled by a temperature modulator. Thin-layer chromatography (TLC) was conducted with silica gel pre-coated plates (0.25 mm) and visualized using a combination of UV, *p*-anisaldehyde and ceric ammonium molybdate. Preparative TLC was conducted with silica gel pre-coated plates (0.50 mm, 20 × 20 cm). Silica gel (particle size 0.032-0.063 mm) was used for flash column chromatography. Analytical chiral HPLC was performed with a Chiralcel AD, AS, OJ or OD-H normal phase column (each is 4.6 mm × 25 cm). ^{1}H NMR spectra were recorded at 300 MHz or at 500 MHz and are reported relative to residual solvent peaks ($CDCl_3$, δ 7.26; DMSO-d_6, δ 2.49). Data for ^{1}H NMR spectra is reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), and integration. ^{13}C NMR spectra were recorded at 75 MHz or at 125 MHz and are reported relative to residual solvent peaks ($CDCl_3$, δ 77.3; DMSO-d_6, δ 39.5). Data for ^{13}C NMR spectra is reported in terms of chemical shift. IR spectra were recorded on a spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Optical rotations were measured with a polarimeter. High resolution mass spectra were obtained.

Preparative Procedures.

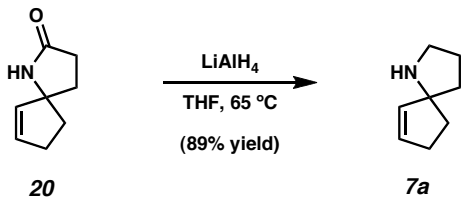


Ester 18. The allylic alcohol **17** (12.3 g, 125 mmol) was dissolved in triethylorthoacetate (150 mL, 818 mmol), and the solution was treated with propionic acid (3.0 mL, 40 mmol). The reaction was heated to 145 °C with distillative removal of ethanol (ca. 23 mL). After distillation was complete, the reaction was stirred at 145 °C for an additional 1 h, and then cooled to room temperature and diluted with Et_2O (300 mL). The resulting solution was stirred with 1.0 M aqueous $KHSO_4$ (300 mL) for 8 h. The phases were separated, and the aqueous phase was extracted with Et_2O (3 × 300 mL). The organic layers were combined, washed with saturated $NaHCO_3$ (300 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash chromatography (30:1 hexanes/ Et_2O) to give ester **18** (16.6 g, 80% yield) as a clear oil. R_f 0.62 (4:1 hexanes/ Et_2O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.32 (m, 1H), 4.11 (q, J = 7.2 Hz, 2H), 2.47-2.41 (m, 2H), 2.38-2.32 (m, 2H), 2.30-2.19 (m, 4H), 1.83 (quintet, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 173.7, 143.2, 124.1, 60.5, 35.3, 33.1, 32.6, 26.6, 23.6, 14.4; IR (neat): 2943, 1736, 1445, 1184, 1038 cm^{-1}; HRMS-EI (m/z): $[M]^+$ calc'd for $C_{10}H_{16}O_2$, 168.1150; found, 168.1155.

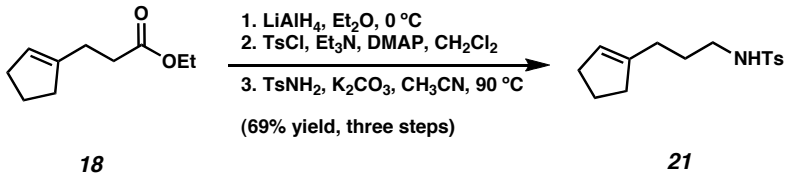


Amide 19. To a solution of ethyl ester **18** (3.0 g, 17.8 mmol) in THF (30 mL) at room temperature was added a solution of LiOH (2.13 g, 89.0 mmol) in H_2O (30 mL). The mixture was heated to 50 °C and stirred for 15 h. The mixture was cooled to room temperature, and the volatile solvent was removed by rotary evaporation. The aqueous residue was acidified to pH = 0 with 3 N HCl. The white solid (2.1 g, 84.2%) was collected by vacuum filtration. R_f 0.35 (4:1 hexanes/ EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 11.5 (br s, 1H), 5.37 (app. quintet, J = 1.8 Hz, 1H), 2.55-2.50 (m, 2H), 2.42-2.36 (m, 2H), 2.33-2.22 (m, 4H), 1.86 (app. quintet, J = 7.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 180.2, 142.8, 124.3, 35.4, 32.8, 32.7, 26.3, 23.6; IR (film): 3100 (br), 2896, 1706, 1446, 1302 cm^{-1}.

To a solution of acid (4.65 g, 33.2 mmol) in THF (100 mL) was treated with 1,1'-carbonyldiimidazole (CDI, 5.43 g, 33.5 mmol) at room temperature. After stirring for 1 h, the solution was cooled to 0 °C and 28% ammonium hydroxide solution (12 mL) was added in one portion. The solution was sparged with argon to remove excess ammonia and the volatile solvent was removed by rotary evaporation. The resulting white solid was suspended in H_2O (100 mL) and collected by vacuum filtration to give the amide **19** (4.5 g, 97.5% yield) as a white powder. R_f 0.15 (1:1 hexanes/ EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.72 (br s, 1H), 5.58 (br s, 1H), 5.38 (br s, 1H), 2.39 (app. s, 4H), 2.32-2.22 (m, 4H), 1.86 (app. quintet, J = 7.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 175.5, 143.3, 124.5, 35.3, 34.4, 32.6, 27.0, 23.6; IR (film): 3382, 3183, 1657, 1632, 1416 cm^{-1}; HRMS-EI (m/z): $[M]^+$ calc'd for $C_8H_{13}NO$, 139.0997; found, 139.0996.



Spiroamine 7a. To a suspension of lithium aluminum hydride (417 mg, 11.0 mmol) in THF (10 mL) at 0 °C was added a solution of spirolactam **20** (504 mg, 3.68 mmol) in THF (8 mL) dropwise over 2 min. The reaction mixture was heated to 70 °C and stirred for 8 h. The mixture was cooled to 0 °C and quenched with H_2O (0.42 mL), 3 N NaOH (0.42 mL) and H_2O (0.85 mL) sequentially. The mixture was allowed to warm to room temperature and stirred vigorously for 4 h. The resulting white precipitate was removed by vacuum filtration and the filtrate was concentrated in vacuo to provide amine **7a** (402 mg, 89% yield) as a clear oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.65 (dt, J_1 = 5.7 Hz, J_2 = 2.4 Hz, 1H), 5.55 (dt, J_1 = 5.7 Hz, J_2 = 2.1 Hz, 1H), 2.88 (td, J_1 = 6.9 Hz, J_2 = 1.8 Hz, 2H), 2.36-2.15 (m, 3H), 1.87-1.59 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.4, 131.1, 74.2, 46.0, 38.6, 37.6, 31.3, 25.7; IR (film): 3307, 2952, 1676, 1449, 1058 cm^{-1}; HRMS-EI (*m*/*z*): [M]$^+$ calc'd for $C_8H_{13}N$, 123.1048; found, 123.1049.



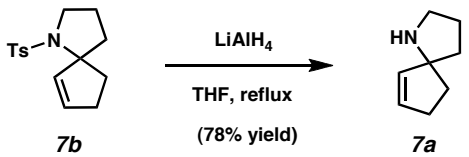
Sulfonamide 21. To a suspension of lithium aluminum hydride (LAH, 5.7 g, 150 mmol) in Et_2O (100 mL) at 0 °C, a solution of ester **18** (16.5 g, 98 mmol) in Et_2O (50 mL) was added dropwise via an addition funnel. After the addition was complete, the reaction was warmed to room temperature and allowed to stir for 19 h. The reaction was then cooled

to 0 °C and quenched by careful addition of H_2O (ca. 50 mL). The resulting mixture was diluted with Et_2O (300 mL) and stirred vigorously with 20% aqueous solution of sodium potassium tartrate (300 mL) for 4 h. The phases were then separated, and the aqueous phase was extracted with Et_2O (4 × 300 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated to yield the alcohol (11.1 g, 88% yield) as a clear oil. The alcohol was taken to the next step without further purification. R_f 0.15 (1:1 hexanes/ CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.32 (m, 1H), 3.59 (m, 1H), 2.57 (br, 1H), 2.26-2.20 (m, 4H), 2.10 (m, 2H), 1.87-1.80 (m, 2H), 1.70-1.66 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.4, 123.8, 62.9, 35.2, 32.6, 30.9, 27.6, 23.6; IR (neat): 3306, 2948, 1652, 1445, 1059 cm^{-1}.

To a solution of alcohol (9.2 g, 73 mmol) in CH_2Cl_2 (100 mL) was added Et_3N (8.12 g, 11.2 mL, 80 mmol), *p*-toluenesulfonyl chloride (TsCl, 15.3 g, 80 mmol), and *N,N*-dimethyl-4-aminopyridine (DMAP, 100 mg, 0.82 mmol) successively at 0 °C. The reaction was allowed to warm to room temperature and stirred for 16 h. The reaction was poured into saturated aqueous NH_4Cl (300 mL). Phases were separated and the aqueous phase was extracted with CH_2Cl_2 (3 × 200 mL). The organic layers were combined, washed with H_2O (200 mL), brine (200 mL), dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by flash chromatography (4:1 hexanes/ CH_2Cl_2) to give tosylate (17.1 g, 84% yield) as a clear oil. R_f 0.25 (2:1 hexanes/ CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 5.20 (m, 1H), 3.98 (t, *J* = 6.3 Hz, 2H), 2.41 (s, 3H), 2.20-2.17 (m, 2H), 2.10-2.02 (m, 4H), 1.78-1.73 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 145.0, 142.7, 133.3, 130.0, 128.1, 124.6, 70.5,

35.0, 32.6, 27.1, 26.9, 23.5, 21.8; IR (film): 2950, 1598, 1445, 1361, 1176, 1097 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_{15}H_{20}O_3S$, 280.1133; found, 280.1144.

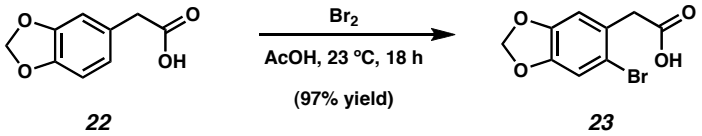
To a solution of tosylate (14.0 g, 49.9 mmol) in CH_3CN (200 mL) was added *p*-toluenesulfonamide (17.1 g, 100 mmol) and K_2CO_3 (13.8 g, 100 mmol) at room temperature. The resulting mixture was heated at 90 °C for 30 h. After cooled the reaction to room temperature, the volatiles were removed in vacuo. The residue was partitioned between H_2O (500 mL) and CH_2Cl_2 (300 mL). The aqueous phase was extracted with CH_2Cl_2 (2 × 300 mL). The combined organic layers were washed with H_2O (2 × 300 mL), 3 M NaOH (4 × 200 mL), H_2O (200 mL), dried over $MgSO_4$, filtered and concentrated to a yellow oil. The oil was purified by flash chromatography (4:1 hexanes/ EtOAc) to give sulfonamide **21** (13.0 g, 93% yield). R_f 0.20 (4:1 hexanes/ EtOAc); ^1H NMR (300 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.7 Hz, 2H), 7.28 (d, *J* = 8.7 Hz, 2H), 5.23 (m, 1H), 5.03 (t, *J* = 6.0 Hz, 1H), 2.89 (q, *J* = 6.3 Hz, 2H), 2.40 (s, 3H), 2.24-2.19 (m, 2H), 2.13-2.07 (m, 2H), 2.03-1.99 (m, 2H), 1.83-1.75 (m, 2H), 1.63-1.56 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.5, 143.4, 137.2, 129.9, 127.3, 124.4, 43.2, 35.1, 32.6, 28.2, 27.7, 23.6, 21.7; IR (film): 3284, 2945, 1598, 1438, 1325, 1163, 1095 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{15}H_{21}NO_2S$, 280.1371; found, 280.1372.



7b *7a*

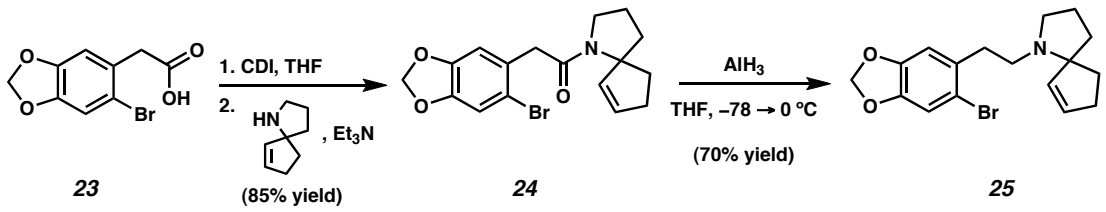
Spiroamine 7a. To a solution of cyclic sulfonamide **7b** (140 mg, 0.5 mmol) in THF (5 mL) was added lithium aluminum hydride (76 mg, 2.0 mmol) at 0 °C. The solution was heated at 70 °C for 12 h. After cooled to room temperature, the reaction was quenched

with H_2O (76 μL), 3 M NaOH (76 μL) and H_2O (228 μL) and stirred for 1 h. The resulting white precipitate was removed by filtration and washed with ether. The filtrate was dried over Na_2SO_4, filtered and concentrated in vacuo to afford the spiroamine **7a** (48 mg, 78% yield) as clear oil. The spiroamine was taken to the next step without further purification.



22 *23*

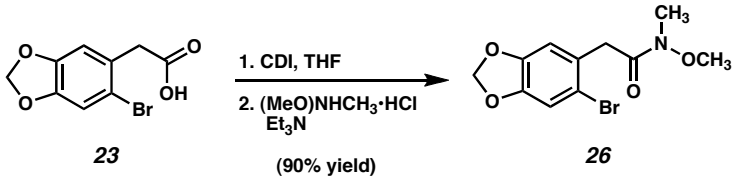
Acid 23. To a solution of 3,4-(methylenedioxy)phenylacetic acid (**22**) (900 mg, 5.0 mmol) in acetic acid (5.0 mL) was added Br_2 (1.6 g, 10.0 mmol) dropwise at room temperature. The resulting solution was stirred at room temperature for 18 h. The reaction was quenched by slow addition of 10% $Na_2S_2O_3$ solution (ca. 1 mL) until the red color disappeared. The mixture was poured into ice water (200 mL) and extracted with Et_2O (4 × 100 mL). The organic layers were combined, washed with brine (100 mL), dried over Na_2SO_4, filtered and concentrated to give the desired acid **23** (1.25, 97%) as a white powder. The acid was taken to the next step without further purification. R_f 0.33 (EtOAc); ^{1}H NMR (300 MHz, DMSO-d_6): δ 12.4 (s, 1H), 7.16 (s, 1H), 6.97 (s, 1H), 6.02 (s, 2H), 3.60 (s, 2H); ^{13}C NMR (300 MHz, DMSO-d_6): δ 171.6, 147.2, 147.0, 128.1, 114.9, 112.1, 111.6, 101.9; IR (film): 3400 (br), 1699, 1502, 1488, 1409, 1225 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_9H_7BrO_4$, 257.9528; found, 257.9536.



Tertiary amine 25. To a solution of acid **23** (520 mg, 2.0 mmol) in THF (5.0 mL) was treated with 1,1'-carbonyldiimidazole (CDI, 325 mg, 2.0 mmol) at room temperature. After stirring for 15 min, a solution of spiroamine **7a** (259 mg, 2.1 mmol) in THF (2.0 mL) and triethylamine (212 mg, 2.1 mmol) were added. The resulting solution was stirred at room temperature for 1 h. The reaction was poured into saturated aqueous NH_4Cl (50 mL) and extracted with Et_2O (4 × 50 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash chromatography (7:3 hexanes/ EtOAc) to give amide **24** (620 mg, 84.7% yield) as a white powder. R_f 0.30 (1:1 hexanes/ EtOAc); ^1H NMR (300 MHz, $CDCl_3$) showed this compound as a mixture of rotamers in 2:1 ratio, and it is taken to the next step without further characterization.

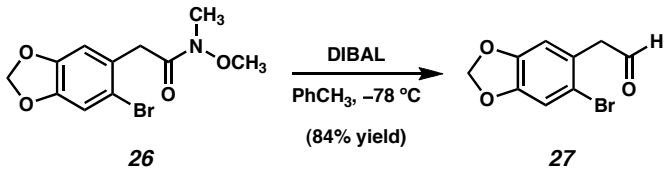
At 0 °C, THF (2.0 mL) was slowly added to a round-bottomed flask charged with aluminum chloride ($AlCl_3$, 16 mg, 0.12 mmol). After the mixture was stirred at this temperature for 5 min, a solution of lithium aluminum hydride (LAH, 14 mg, 0.36 mmol) in THF (1.0 mL) was added dropwise via a syringe. The resulting mixture was stirred for 30 min at room temperature and then cooled to –78 °C. A solution of amide (36.3 mg, 0.1 mmol) in THF (2.0 mL) was added slowly. The reaction was stirred at –78 °C for 45 min, warmed to room temperature and stirred for additional 2 h. The reaction was cooled to 0 °C and quenched by slow addition of 1 N HCl (5.0 mL). The mixture was diluted with H_2O (20 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The organic layers were

combined, washed with 1 N NaOH (20 mL), brine (30 mL), dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by preparative TLC (9:1 CH_2Cl_2/MeOH) to yield amine **25** (25 mg, 70% yield) as a lightly yellow oil. R_f 0.20 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.96 (s, 1H), 6.71 (s, 1H), 5.92 (2, 2H), 5.80 (dt, J_1 = 5.7 Hz, J_2 = 2.4 Hz, 1H), 5.56 (dt, J_1 = 5.7 Hz, J_2 = 2.1 Hz, 1H), 2.98-2.92 (m, 1H), 2.84-2.77 (m, 2H), 2.50-2.42 (m, 2H), 2.30 (tt, J_1 = 6.6 Hz, J_2 = 2.1 Hz, 2H), 1.94-1.82 (m, 4H), 1.63 (app. quintet, J = 6.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.5, 147.0, 134.3, 133.1, 133.0, 114.6, 112.8, 110.5, 101.8, 78.5, 51.4, 49.8, 38.2, 36.3, 31.7, 29.8, 21.5; IR (film): 2956, 1503, 1478, 1229, 1041 cm^{-1}; HRMS-FAB (*m*/*z*): [M + H]$^+$ calc'd for $C_{17}H_{20}BrNO_2$, 350.0756; found, 350.0748.



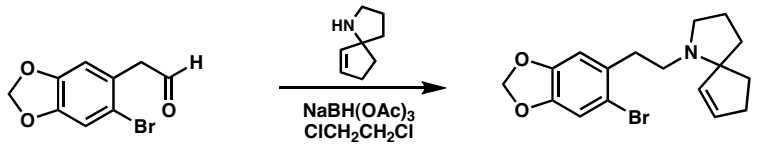
Weinreb amide 26. To a solution of acid **23** (260 mg, 1.0 mmol) in THF (4.0 mL) was treated with 1,1'-carbonyldiimidazole (CDI, 163 mg, 1.0 mmol) at room temperature. After stirring for 15 min, a solution of Weinreb amine hydrochloride salt (108 mg, 1.1 mmol) and triethylamine (303 mg, 3.0 mmol) were added. The resulting solution was stirred at room temperature for 4 h. The reaction was poured into saturated aqueous NH_4Cl (30 mL) and extracted with CH_2Cl_2 (4 × 30 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash chromatography (3:2 hexanes/ EtOAc) to give amide **26** (270 mg, 90% yield) as a white powder. R_f 0.65 (EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.00 (s, 1H), 6.78 (s, 1H), 5.95 (s, 2H), 3.82 (s, 2H), 3.72 (s, 3H), 3.21 (s, 3H); ^{13}C NMR (75 MHz,

$CDCl_3$): δ 171.7, 147.7, 147.6, 128.0, 115.5, 112.9, 111.2, 102.0, 61.6, 39.6, 32.6; IR (film): 2968, 1667, 1503, 1481, 1233, 1037 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{11}H_{12}BrNO_4$, 302.0028; found, 302.0021.



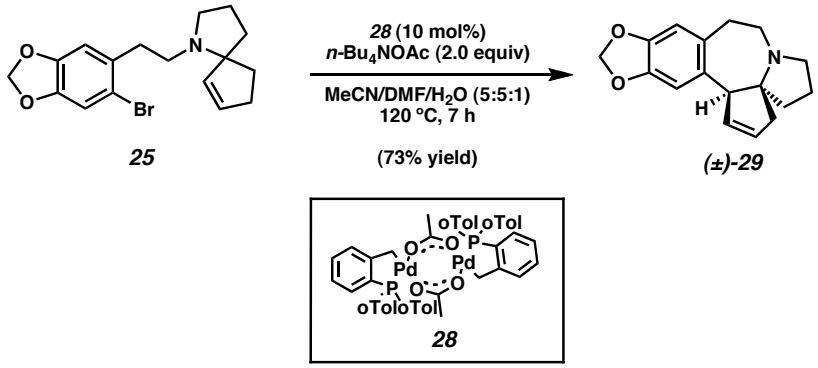
26 *27*

Aldehyde 27. To a solution of Weinreb amide **26** (55 mg, 0.18 mmol) in CH_2Cl_2 (1.0 mL) at –78 °C was added a solution of DIBAL (39 mg, 0.275 mmol) in CH_2Cl_2 (1.0 mL) dropwise. The resulting solution was stirred at –78 °C for 30 min and quenched carefully with MeOH (ca. 0.2 mL). The reaction was poured into CH_2Cl_2 (50 mL), washed with 1 N HCl (10 mL) and brine (10 mL) and the organic layer was quickly passed through a pad of silica gel and concentrated to give the aldehyde **27** (37 mg, 84% yield) as a clear oil. This compound was not stable at room temperature and was taken to the next step immediately. R_f 0.30 (1:4 EtOAc/hexanes); ^1H NMR (500 MHz, $CDCl_3$): δ 9.73 (t, *J* = 2.0 Hz, 1H), 7.08 (s, 1H), 6.72 (s, 1H), 6.01 (s, 2H), 3.78 (d, *J* = 2.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 198.6, 148.3, 148.0, 125.5, 115.6, 113.2, 111.3, 102.2, 50.6; IR (film): 2904, 1724, 1503, 1478, 1233, 1038 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_9H_7BrO_3$, 241.9579; found, 241.9569.



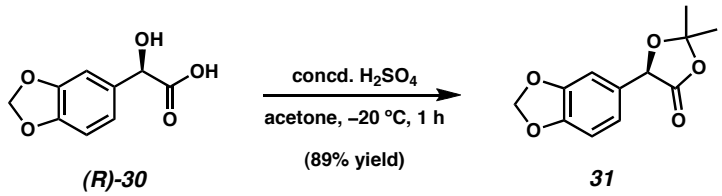
27 *25*

Tertiary amine 25. To a solution of aldehyde **27** (150 mg, 0.62 mmol) and spiroamine **7a** (84 mg, 0.68 mmol) in 1,2-dichloromethane was added sodium triacetoxyborohydride (197 mg, 0.93 mmol) at room temperature. The resulting solution was stirred at room temperature for 24 h and then poured into saturated $NaHCO_3$ (50 mL). The aqueous was extracted with ether (3 × 70 mL). The organic layers were combined, dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash chromatography (1: 9 $MeOH/CH_2Cl_2$) to give amine **25** (183 mg, 85% yield) as a yellow oil.



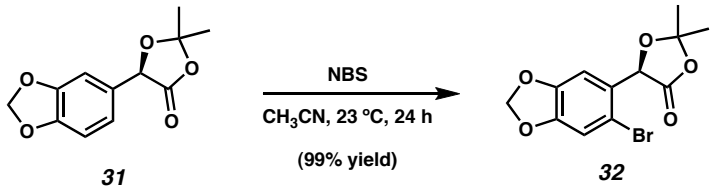
Olefin 29. The amine **25** (70 mg, 0.2 mmol) was dissolved in a mixture of solvents ($DMF/CH_3CN/H_2O$ = 3 mL: 3 mL: 0.6 mL). The solution was degassed with Argon for 15 min and then treated with trans-Di-μ-acetatobis[2-(di-*o*-tolylphosphino)benzyl] dipalladium(II) (21 mg, 0.02 mmol) and tetra-n-butyl ammonium acetate (120 mg, 0.4 mmol). The resulting solution was heated at 120 °C for 7 h. The reaction was cooled to room temperature and filtered through a short pad of celite. The filtrate was concentrated in vacuo. The residue was dissolved in Et_2O (20 mL) and extracted with 1 N NaOH (20 mL). The aqueous phase was extracted with Et_2O (3 × 20 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by flash chromatography (EtOAc) to give the olefin **29** (39 mg, 72.5% yield) as a white powder. R_f 0.10 (EtOAc); R_f 0.25 (85:17:1 EtOAc/MeOH/Et_3N); ^1H NMR (300

MHz, $CDCl_3$): δ 6.65 (s, 1H), 6.59 (s, 1H), 5.88 (dd, J_1 = 2.4 Hz, J_2 = 1.5 Hz, 2H), 5.79 (ddd, J_1 = 5.7 Hz, J_2 = 5.4 Hz, J_3 = 2.7 Hz, 1H), 5.52 (ddd, J_1 = 5.7 Hz, J_2 = 4.8 Hz, J_3 = 2.7 Hz, 1H), 3.87 (m, 1H), 3.18 (ddd, J_1 = 14.1 Hz, J_2 = 12.9 Hz, J_3 = 7.2 Hz, 1H), 3.07 (ddd, J_1 = 10.5 Hz, J_2 = 8.7 Hz, J_3 = 4.2 Hz, 1H), 2.74 (ddd, J_1 = 18.0 Hz, J_2 = 4.5 Hz, J_3 = 2.7 Hz, 1H), 2.53 (dd, J_1 = 11.1 Hz, J_2 = 7.2 Hz, 1H), 2.43-2.28 (m, 2H), 2.05-1.93 (m, 3H), 1.80-1.67 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.5, 146.1, 132.5, 132.4, 132.0, 128.9, 111.0, 110.0, 100.9, 68.3, 62.5, 53.8, 49.2, 43.4, 34.9, 30.8, 20.1; IR (film): 2940, 2875, 1502, 1485, 1225, 1039 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{17}H_{19}NO_2$, 270.1484; found, 270.1490.



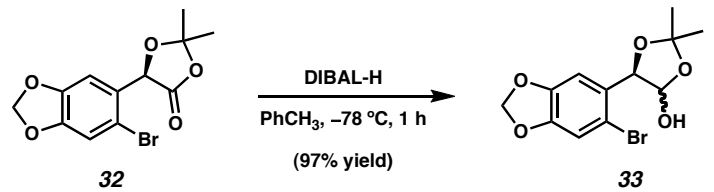
1,3-dioxolanone 31. To a solution of α-hydroxy carboxylic acid (**30**) (1.88 g, 9.6 mmol) in acetone (10 mL) was added concentrated H_2SO_4 (1.0 mL) dropwise at –20 °C. The resulting mixture was stirred at –20 °C for 1 h. The reaction was poured into saturated aqueous $NaHCO_3$ (50 mL) with crushed ice and extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated to a yellowish oil. The oil was purified by flash chromatography (4:1 hexanes/ EtOAc) to give dioxolanone **31** (2.0 g, 89% yield) as a clear oil. R_f 0.18 (4:1 hexanes/ EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.92-6.88 (m, 2H), 6.80 (dd, J_1 = 7.8 Hz, J_2 = 0.9 Hz, 1H), 5.93 (s, 2H), 5.27 (s, 1H), 1.67 (s, 3H), 1.62 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.7, 148.5, 148.3, 128.4, 120.9, 110.9, 108.6, 107.2, 101.6, 76.1, 27.4, 26.2; IR (film): 2994, 1794, 1505, 1492, 1446, 1388, 1242, 1118, 1038 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for

$C_{12}H_{12}O_5$, 236.0685; found, 236.0675; $[\alpha]^{25.2}_D$ –70.6° (*c* 1.0, CH_2Cl_2); ee% = 97.5%, Chiralcel OD-H, 1% *i*-PrOH/hexanes, 1 mL/min, T_R = 17.3 min, $T_{R(minor)}$ = 21.4 min.



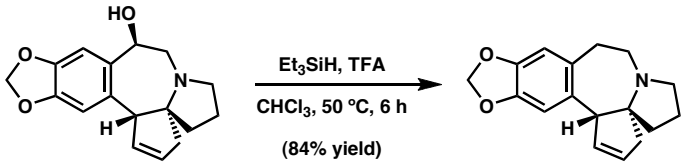
31 *32*

Aryl bromide 32. To a solution of dioxolanone **31** (1.2 g, 5.1 mmol) in acetonitrile (15 mL) was added *N*-bromosuccinamide (1.0 g, 5.6 mmol) at room temperature. The resulting solution was stirred at room temperature in the dark for 24 h. The solution was passed through a pad of silica gel and eluted with CH_2Cl_2 (ca. 100 mL). The filtrate was concentrated to dryness and the residue was purified by flash chromatography (2:1 hexanes/CH_2Cl_2) to give the aryl bromide **32** (1.6 g, 99% yield) as a yellow oil. R_f 0.35 (2:1 hexanes/CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.04 (s, 1H), 6.84 (s, 1H), 5.99 (dd, J_1 = 5.1 Hz, J_2 = 1.5 Hz, 1H), 5.73 (s, 1H), 1.73 (s, 3H), 1.66 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.8, 149.5, 148.1, 126.7, 115.3, 113.4, 110.9, 108.7, 102.5, 76.0, 27.2, 25.9; IR (film): 2992, 1794, 1504, 1482, 1388, 1238, 1119, 1036 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_{12}H_{11}BrO_5$, 313.9790; found, 313.9794; $[\alpha]^{24.7}_D$ –61.3° (*c* 1.0, CH_2Cl_2); ee% = 97.7%, Chiralcel AD, 1% *i*-PrOH/hexanes, 1 mL/min, T_R = 17.7 min, $T_{R(minor)}$ = 29.4 min.



32 *33*

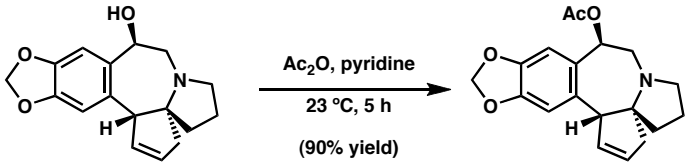
Hemiacetal 33. To a solution of aryl bromide **32** (314 mg, 1.0 mmol) in toluene (4 mL) was added DIBAL (227 mg, 232 μL, 1.6 mmol) dropwise at –78 °C. After stirred at this temperature for 30 min, the reaction was quenched by slow addition of 1 N HCl (2 mL) at –78 °C. The solution was allowed to warm to room temperature and stirred for additional 30 min. The solution was diluted with H_2O (20 mL) and extracted with Et_2O (3 × 20 mL). The combined organic layers were passed through a short pad of silica gel and concentrated in vacuo. The resulting residue was purified by flash chromatography (4:1 hexanes/EtOAc) to give hemiacetal **33** (305 mg, 97% yield) as a mixture of diastereomers in about 1:1 ratio. R_f 0.18 (4:1 hexanes/EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.15 (s, 1H), 6.99 (s, 1H), 6.98 (s, 1H), 6.97 (s, 1H), 5.99-5.95 (m, 4H), 5.77 (dd, J_1 = 5.1 Hz, J_2 = 3.6 Hz, 1H), 5.33-5.28 (m, 3H), 3.58 (d, J = 3.6 Hz, 1H), 2.73 (d, J = 5.4 Hz, 1H), 1.66 (s, 3H), 1.64 (s, 3H), 1.59 (s, 3H), 1.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 148.3, 147.9, 147.7, 131.5, 129.3, 128.7, 128.5, 125.5, 120.2, 112.9, 112.7, 112.2, 110.1, 109.1, 107.9, 102.1, 101.9, 94.3, 84.3, 81.1, 29.0, 27.8, 27.6, 26.5; IR (film): 3436, 2987, 1502, 1477, 1241, 1038 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_{12}H_{13}BrO_5$, 315.9946; found, 315.9940.



15 *(+)-29*

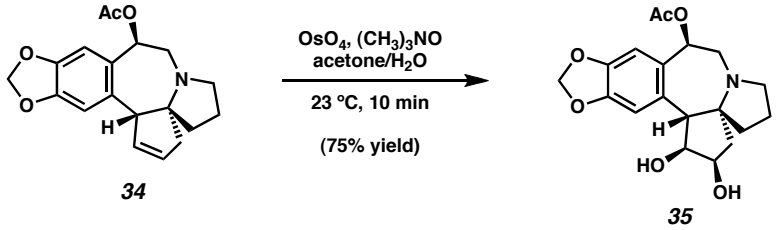
Olefin (+)-29. To a solution of alcohol **15** (8.0 mg, 0.028 mmol) in $CHCl_3$ (1.0 mL) was added trifluoroacetic acid (80 μL, 1 mmol) and Et_3SiH (100 μL, 0.63 mmol). The resulting solution was heated at 50 °C for 6 h and then cooled to room temperature. The

reaction was poured into saturated $NaHCO_3$ (5 mL) and the aqueous was extracted with CH_2Cl_2 (5 × 10 mL). The organics were combined, dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by preparative TLC (10% MeOH/ CH_2Cl_2) to give the olefin (+)-**29** (6.4 mg, 84% yield) as a clear oil. The spectra data are identical to those of (±)-**29** presented above. $[\alpha]^{27.3}_D$ +190.5° (*c* 0.15, $CHCl_3$).

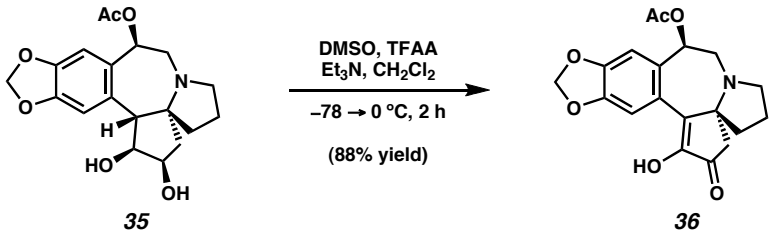


15 ***34***

***anti*-Acetate 34**. The alcohol **15** (67 mg, 0.235 mmol) was dissolved in pyridine (2 mL) and cooled to 0 °C. The solution was treated with acetic anhydride (0.3 mL) and stirred at room temperature for 5 h. The reaction was poured into saturated aqueous NH_4Cl (20 mL) and extracted with CH_2Cl_2 (5 × 50 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by flash chromatography (1:4 hexanes/EtOAc) to give acetate **34** (70 mg, 90% yield) as a white semi solid. R_f 0.40 (EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.85 (s, 1H), 6.66 (s, 1H), 6.33 (dd, J_1 = 9.9 Hz, J_2 = 7.2 Hz, 1H) 5.94-5.90 (m, 3H), 5.62-5.58 (m, 1H), 3.92 (t, J = 2.4 Hz, 1H), 3.08-3.00 (m, 1H), 2.89-2.73 (m, 3H), 2.49-2.40 (m, 1H), 2.15 (s, 3H), 2.10-2.03 (m, 1H), 1.98-1.89 (m, 3H), 1.80-1.70 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.1, 147.0, 146.7, 131.7, 131.4, 129.9, 129.1, 111.2, 104.3, 101.2, 68.8, 68.7, 61.7, 53.1, 52.9, 42.3, 35.5, 21.3, 20.2; IR (film): 2882, 1741, 1502, 1485, 1369, 1236, 1038 cm^{-1}; HRMS-EI (*m*/*z*): $[M]^+$ calc'd for $C_{19}H_{21}NO_4$, 327.1471; found, 327.1468; $[\alpha]^{24.7}_D$ +42.2° (*c* 1.0, $CHCl_3$).

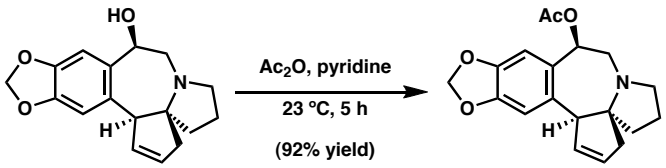


***anti*-Diol 35**. To a solution of olefin **34** (40 mg, 0.122 mmol) in acetone (2.0 mL) was added trimethylamine *N*-oxide (13.8 mg, 0.184 mmol, 1.5 equiv) at room temperature. A freshly made solution of OsO_4 (1.55 mg, 0.006 mmol, 5 mol%) in H_2O (1.0 mL) was added and the reaction was stirred at room temperature for 10 min. Sodium sulfite (Na_2SO_3, 0.3 g) was added to the solution and the mixture was stirred at room temperature for 30 min. Most volatiles were removed in vacuo and the residue was partitioned between H_2O (10 mL) and CH_2Cl_2 (10 mL). The aqueous phase was further extracted with CH_2Cl_2 (4 × 10 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash chromatography (9:1 CH_2Cl_2/MeOH) to give diol **35** (33 mg, 75% yield) as a white powder. R_f 0.40 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.89 (s, 1H), 6.65 (s, 1H), 6.02 (dd, J_1 = 10.2 Hz, J_2 = 7.8 Hz, 1H), 5.93 (s, 2H), 4.50 (dd, J_1 = 9.3 Hz, J_2 = 3.9 Hz, 1H), 4.29 (t, J = 3.6 Hz, 1H), 3.12 (d, J = 9.6 Hz, 1H), 3.05-2.98 (m, 1H), 2.89-2.77 (m, 2H), 2.51-2.35 (m, 3H), 2.20 (s, 3H), 2.14-2.03 (m, 2H), 1.77-1.68 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.4, 147.2, 146.8, 130.2, 129.8, 112.3, 104.7, 101.3, 77.9, 72.5, 68.7, 67.0, 59.8, 53.7, 52.3, 43.9, 31.1, 21.3, 19.7; IR (film): 3400, 2929, 1741, 1503, 1486, 1371, 1237, 1038 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{19}H_{23}NO_6$, 362.1604; found, 362.1588; $[\alpha]^{25.6}_D$ –6.3° (*c* 1.0, CH_2Cl_2).

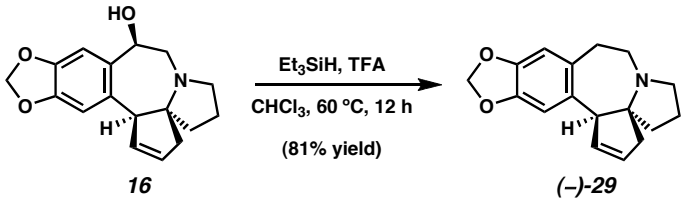


35 ***36***

***anti*-Dione 36**. DMSO (60 mg, 55 μL, 0.77 mmol) was dissolved in CH_2Cl_2 (3 mL). At –78 °C, trifluoroacetic anhydride (TFAA, 46 mg, 30 μL, 0.22 mmol) was added slowly. The resulting solution was stirred at –78 °C for 10 min and then treated with a solution of diol **35** (20 mg, 0.055 mmol) in CH_2Cl_2 (1 mL) dropwise. The reaction was allowed to stir at –78 °C for 1.5 h and then treated with Et_3N (90 mg, 124 μL, 0.88 mmol). The solution was warmed to 0 °C and stirred for 30 min. The reaction was quenched with water (15 mL) and diluted with CH_2Cl_2 (20 mL). The aqueous phase was further extracted with CH_2Cl_2 (4 × 20 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by preparative TLC (12:1 CH_2Cl_2/MeOH) to yield dione **36** (17.3 mg, 88% yield) as a white solid. R_f 0.70 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.85 (s, 1H), 6.77 (s, 1H), 6.15 (t, J = 4.5 Hz, 1H), 6.00 (dd, J_1 = 6.3 Hz, J_2 = 1.5 Hz, 2H), 3.45 (A part of ABX, J_1 = 16.2 Hz, J_2 = 3.3 Hz, 1H), 3.23-3.16 (m, 2H), 3.04-2.98 (m, 1H), 2.55 (q, J = 18.0 Hz, 2H), 2.19 (s, 3H), 2.03-1.93 (m, 2H), 1.81-1.68 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 200.8, 170.7, 148.9, 147.7, 147.5, 144.7, 130.8, 124.1, 109.7, 109.0, 102.0, 71.2, 69.9, 52.4, 50.1, 48.9, 39.6, 24.6, 21.7; IR (film): 3400, 2927, 1733, 1707, 1505, 1486, 1370, 1238 cm^{-1}; HRMS-FAB (m/z): $[M + H]^+$ calc'd for $C_{19}H_{19}NO_6$, 358.1291; found, 358.1285; $[\alpha]^{24.8}_D$ –152.2° (*c* 0.7, $CHCl_3$).

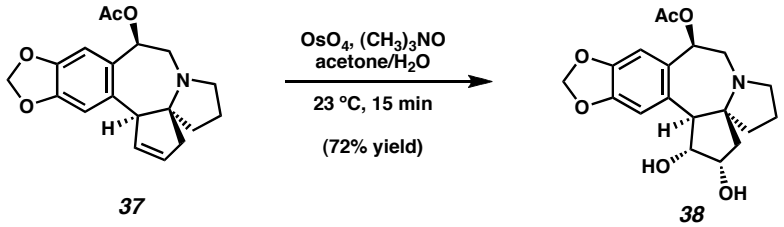


***syn*-Acetate 37**. The crude product of Heck reaction was dissolved in CH_2Cl_2 (3 mL) and pyridine (1 mL) and cooled to 0 °C. The solution was treated with acetic anhydride (1 mL) and stirred at room temperature for 5 h. The reaction was poured into saturated aqueous NH_4Cl (20 mL) and extracted with CH_2Cl_2 (5 × 15 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by flash chromatography (3:7 hexanes/EtOAc→1:9 hexanes/EtOAc) to give acetate **37** (116 mg, 65% yield over two steps) as a white semi solid. R_f 0.24 (EtOAc); R_f 0.45 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.83 (s, 1H), 6.72 (s, 1H), 5.93-5.91 (m, 2H), 5.86 (q, J = 4.5 Hz, 1H), 5.75 (m 2H), 3.85 (d, J = 1.5 Hz), 3.31 (dd, J_1 = 14.1 Hz, J_2 = 8.7 Hz, 1H), 2.89-2.82 (m, 1H), 2.75-2.62 (m, 2H), 2.47 (d, J = 17.7 Hz, 1H), 2.15 (app. dd, J_1 = 17.7 Hz, J_2 = 1.8 Hz, 1H), 2.01 (s, 3H), 1.93-1.66 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 147.7, 146.6, 134.8, 133.7, 129.5, 127.7, 111.2, 111.0, 101.4, 75.1, 71.5, 59.1, 52.4, 51.9, 38.9, 38.6, 21.7, 20.7; IR (film): 2959, 1728, 1506, 1489, 1370, 1234, 1041 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{19}H_{21}NO_4$, 328.1549; found, 328.1563; $[\alpha]^{24.9}_D$ –68.3° (*c* 1.0, $CHCl_3$).



Olefin (–)-29. To a solution of alcohol **16** (5.6 mg, 0.019 mmol) in $CHCl_3$ (1.0 mL) was added trifluoroacetic acid (80 μL, 1 mmol) and Et_3SiH (100 μL, 0.63 mmol). The

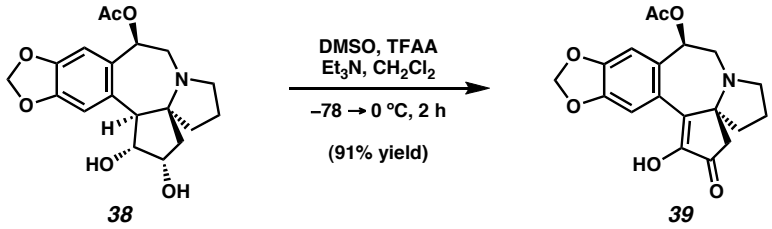
resulting solution was heated at 60 °C for 12 h and then cooled to room temperature. The reaction was poured into saturated $NaHCO_3$ (5 mL) and the aqueous was extracted with CH_2Cl_2 (5 × 10 mL). The organics were combined, dried over Na_2SO_4, filtered and concentrated to dryness. The residue was purified by preparative TLC (15% MeOH/ CH_2Cl_2) to give the olefin (–)-**29**(4.0 mg, 81% yield) as a clear oil. The spectra data are identical to those of (±)-**29** presented above. $[\alpha]^{22.8}_D$ –201.2° (*c* 0.19, $CHCl_3$).



37 *38*

***syn*-Diol 38**. To a solution of olefin **37** (78 mg, 0.238 mmol) in acetone (3.0 mL) was added trimethylamine *N*-oxide (26.9 mg, 0.359 mmol, 1.5 equiv) at room temperature. A freshly made solution of OsO_4 (3 mg, 0.012 mmol, 5 mol%) in H_2O (1.0 mL) was added and the reaction was stirred at room temperature for 10 min. Sodium sulfite (Na_2SO_3, 0.5 g) was added to the solution and the mixture was stirred at room temperature for 30 min. Most volatiles were removed in vacuo and the residue was partitioned between H_2O (20 mL) and CH_2Cl_2 (20 mL). The aqueous phase was further extracted with CH_2Cl_2 (4 × 20 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by flash chromatography (9:1 CH_2Cl_2/MeOH) to give diol **38** (58 mg, 72% yield) as a white powder. R_f 0.35 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.85 (s, 1H), 6.67 (s, 1H), 5.99 (dd, J_1 = 8.7 Hz, J_2 = 3.0 Hz, 1H), 5.91 (dd, J_1 = 3.6 Hz, J_2 = 1.8 Hz, 2H), 4.52 (dd, J_1 = 10.2 Hz, J_2 = 4.5 Hz, 1H), 4.24 (t, *J*

= 4.2 Hz, 1H), 3.45 (dd, J_1 = 15.3 Hz, J_2 = 9.0 Hz, 1H), 3.17 (d, J = 10.2 Hz, 1H), 2.91 (app. q, J = 4.2 Hz, 1H), 2.74 (dd, J_1 = 15.0 Hz, J_2 = 2.7 Hz, 1H), 2.52 (q, J = 7.5 Hz, 2H), 2.20-2.10 (m, 3H), 2.00 (s, 3H), 1.83-1.67 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.1, 148.0, 146.8, 133.9, 128.4, 113.7, 113.4, 101.6, 80.4, 76.2, 72.3, 68.2, 60.3, 52.8, 51.1, 42.3, 34.3, 21.8, 20.4; IR (film): 3413, 2930, 1735, 1506, 1489, 1371, 1232 cm^{-1}; HRMS-FAB (*m*/*z*): $[M + H]^+$ calc'd for $C_{19}H_{23}NO_6$, 362.1604; found, 362.1591; $[\alpha]^{24.4}_D$ –9.6° (*c* 1.0, CH_2Cl_2).

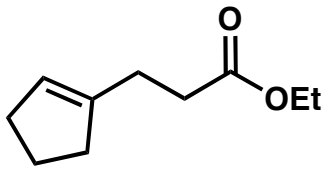


38 ***39***

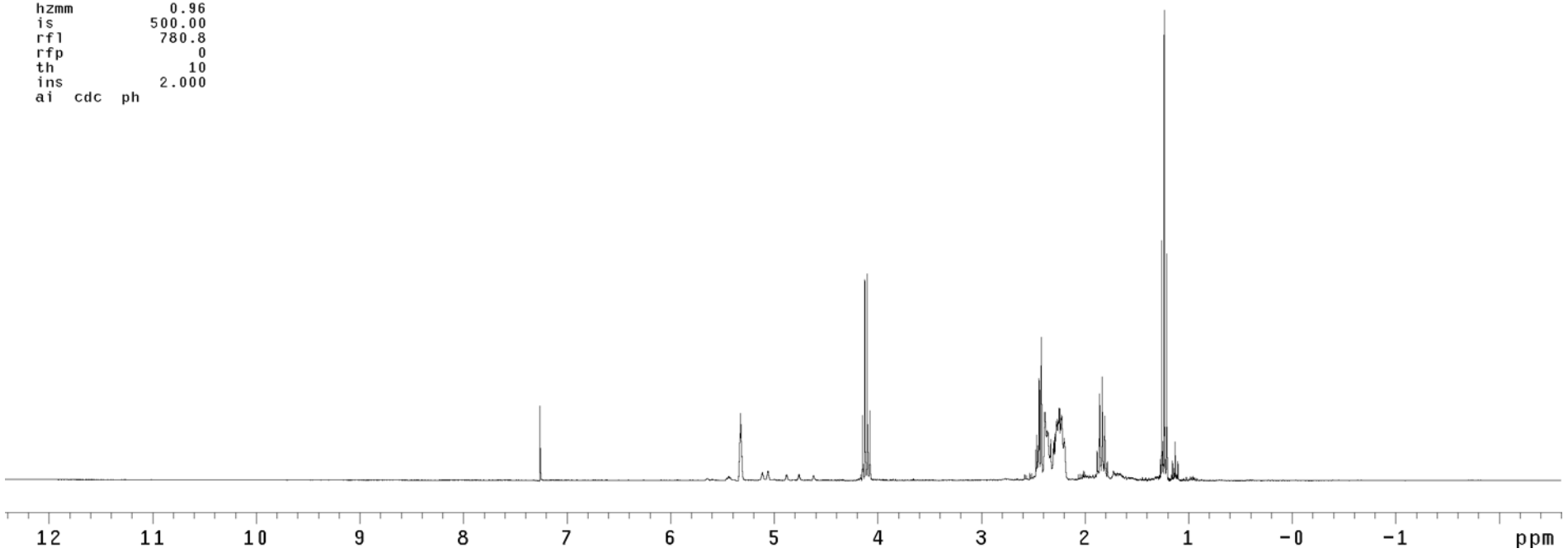
***syn*-Dione 39**. DMSO (120 mg, 109 μL, 1.54 mmol) was dissolved in CH_2Cl_2 (5 mL). At –78 °C, trifluoroacetic anhydride (TFAA, 92.4 mg, 61 μL, 0.44 mmol) was added slowly. The resulting solution was stirred at –78 °C for 10 min and then treated with a solution of diol **38** (40 mg, 0.11 mmol) in CH_2Cl_2 (2 mL) dropwise. The reaction was allowed to stir at –78 °C for 1.5 h and then treated with Et_3N (178 mg, 245 μL, 1.76 mmol). The solution was warmed to 0 °C and stirred for 30 min. The reaction was quenched with water (15 mL) and diluted with CH_2Cl_2 (20 mL). The aqueous phase was further extracted with CH_2Cl_2 (4 × 20 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by preparative TLC (12:1 CH_2Cl_2/MeOH) to yield dione **39** (36 mg, 91% yield) as a white powder. R_f 0.60 (9:1 CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.92 (s, 1H), 6.86 (s, 1H), 6.35 (dd, J_1 = 10.2 Hz, J_2 = 6.0 Hz, 1H), 6.01 (d, J = 6.6 Hz, 2H), 3.29 (A part of ABX, J_1 =

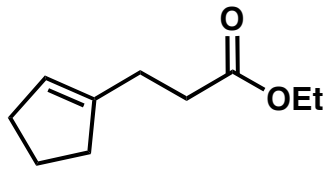
15.0 Hz, J_2 = 6.0 Hz, 1H), 3.12 (B part of ABX, J_1 = 15.0 Hz, J_2 = 10.0 Hz, 1H), 3.10-3.05 (m, 1H), 2.90-2.84 (m, 1H), 2.58 (q, J = 18.6 Hz, 2H), 2.04 (s, 3H), 1.87-1.69 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 201.3, 170.6, 148.7, 148.0, 147.7, 143.8, 130.6, 124.9, 111.2, 109.8, 101.9, 71.9, 69.5, 50.9, 50.3, 47.2, 40.0, 24.8, 21.5; IR (film): 3307, 2929, 1725, 1704, 1504, 1487, 1383, 1234 cm^{-1}; HRMS-FAB (m/z): $[M + H]^+$ calc'd for $C_{19}H_{19}NO_6$, 358.1291; found, 358.1297; $[\alpha]^{25.0}_D$ +131.8° (*c* 1.0, CH_2Cl_2).

```
QL-III-179 on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            30
   /QL-III-179.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.869  dmm              c
tn              H1  dmf            200
at            1.995     PROCESSING
np            17984  wtfile
sw           4506.5  proc             ft
fb        not used  fn        not used
bs               4
tpwr            55  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct              12
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -780.5
wp          4506.3
vs              53
sc               0
wc             250
hzmm          0.96
is          500.00
rfl           780.8
rfp              0
th              10
ins          2.000
ai  cdc  ph
```



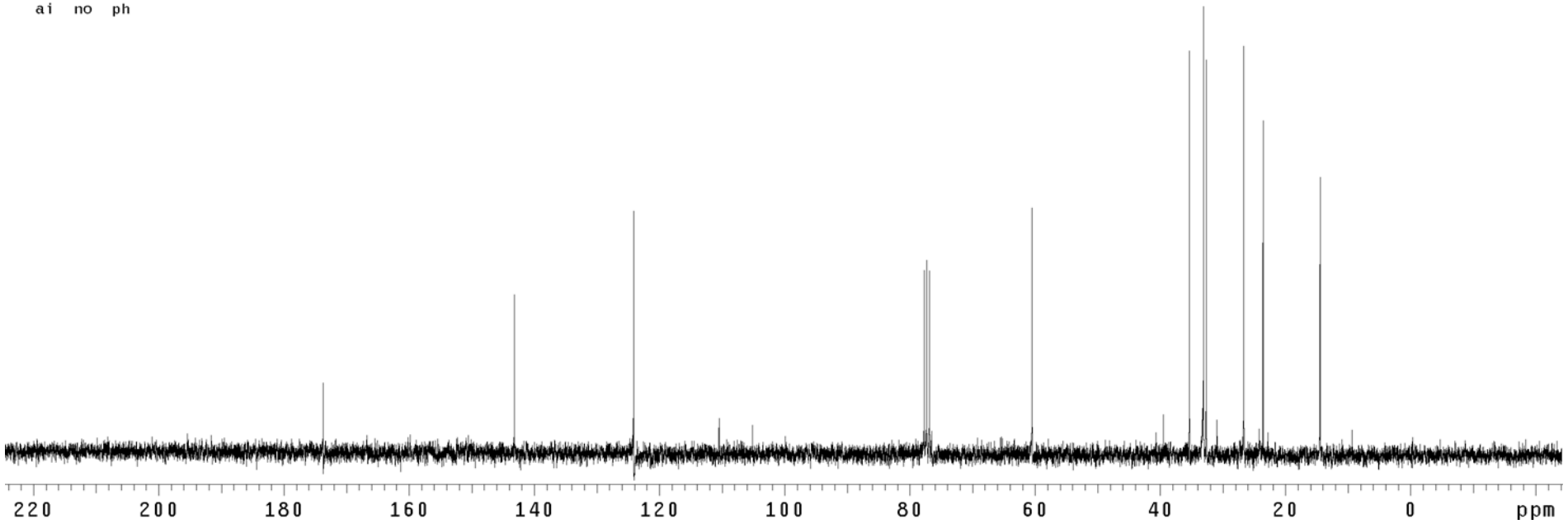
18

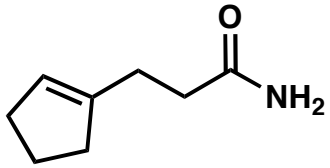




18

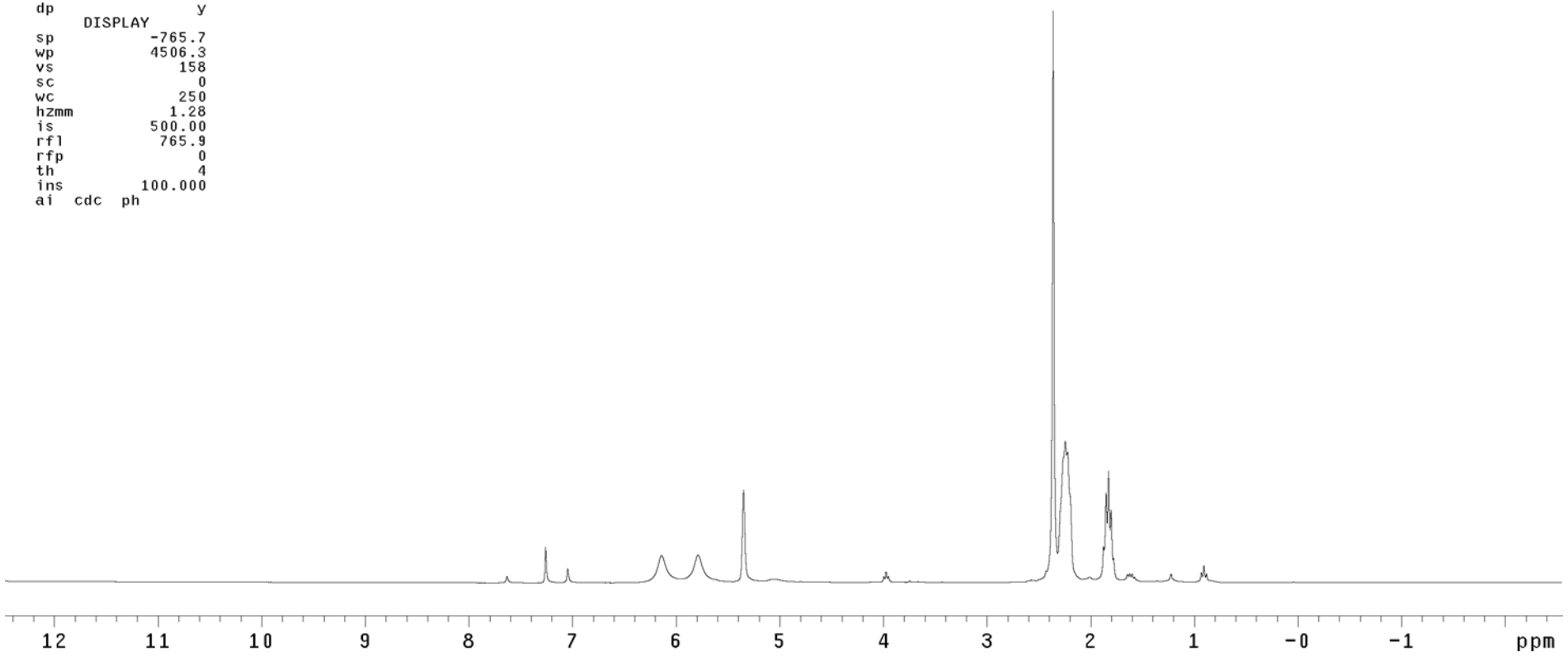
```
QL-III-179 on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            36
/QL-III-179_C13.fi~ dof              0
                 d  dm             yyy
   ACQUISITION      dmm              w
sfrq         75.409  dmf           6500
tn              C13      PROCESSING
at            1.815  lb           1.00
np            68106  wtfile
sw          18761.7  proc           ft
fb            10400  fn       not used
bs                8
tpwr             59  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct              144
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1829.7
wp          18761.4
vs              134
sc                0
wc              250
hzmm          20.12
is           500.00
rfl          1830.0
rfp               0
th               14
ins          100.000
ai  no  ph
```

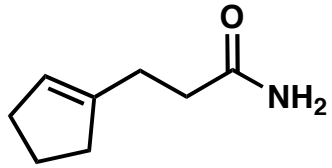




19

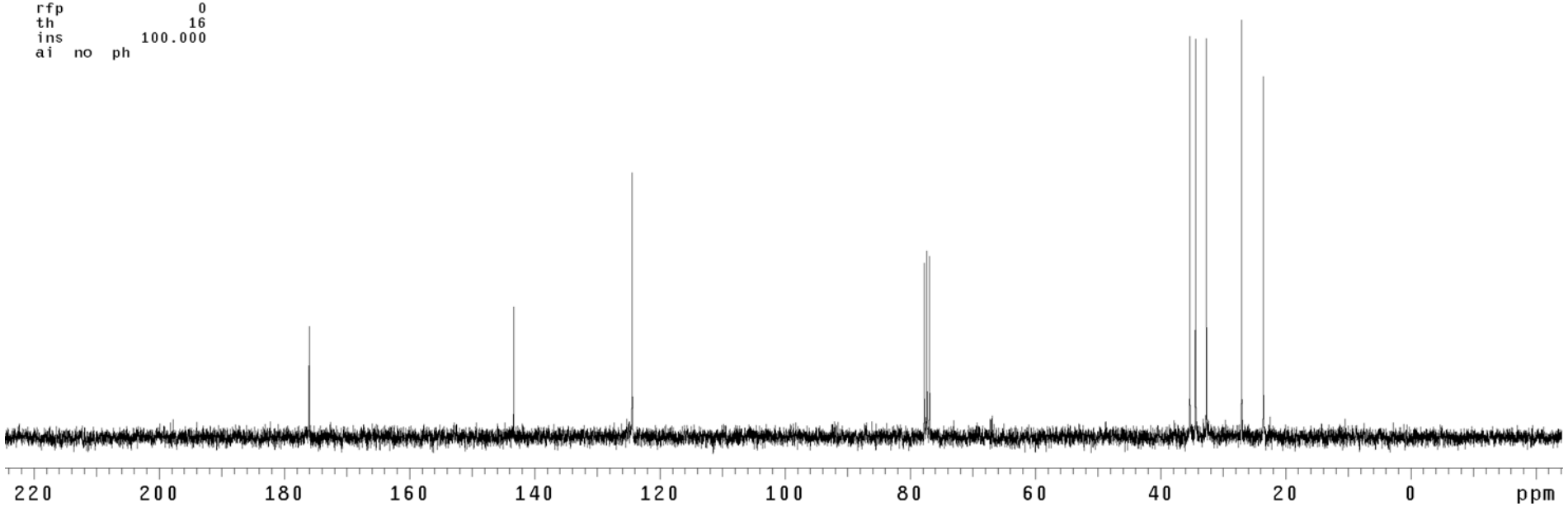
```
QL-I-271 on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
     /QL-I-271.fid  dof              0
   ACQUISITION      dm            nnn
sfrq       299.818  dmm             c
tn              H1  dmf           200
at           1.995      PROCESSING
np           17984  lb            2.00
sw          4506.5  wtfile
fb        not used  proc            ft
bs               4  fn       not used
tpwr            56
pw             7.0  werr
d1           1.000  wexp
tof              0  wbs
nt             256  wnt
ct              36
alock            n
gain     not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs             158
sc               0
wc             250
hzmm          1.28
is          500.00
rfl           765.9
rfp              0
th               4
ins        100.000
ai  cdc  ph
```



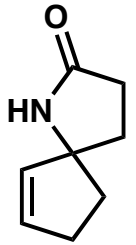


19

```
QL-I-271 on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr             35
 /QL-I-271_C13.fid  dof               0
   ACQUISITION      dm              yyy
sfrq         75.396  dmm               w
tn              C13  dmf            7300
at            1.815      PROCESSING
np            68106  lb            1.00
sw          18761.7  wtfile
fb            10400  proc            ft
bs                8  fn        not used
tpwr             62
pw              8.7  werr
d1                0  wexp
tof               0  wbs
nt             1024  wnt
ct              144
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs               64
sc                0
wc              250
hzmm          20.12
is           500.00
rfl          1827.5
rfp               0
th               16
ins         100.000
ai  no  ph
```



```
QL-I-259 on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            30
     /QL-I-259.fid  dof              0
   ACQUISITION      dm            nnn
sfrq       299.869  dmm             c
tn              H1  dmf           200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb       not used  fn       not used
bs              4
tpwr           55  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct             16
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -780.5
wp         4506.3
vs            112
sc              0
wc            250
hzmm         4.00
is         500.00
rfl         780.8
rfp             0
th             20
ins       100.000
ai  cdc  ph
```



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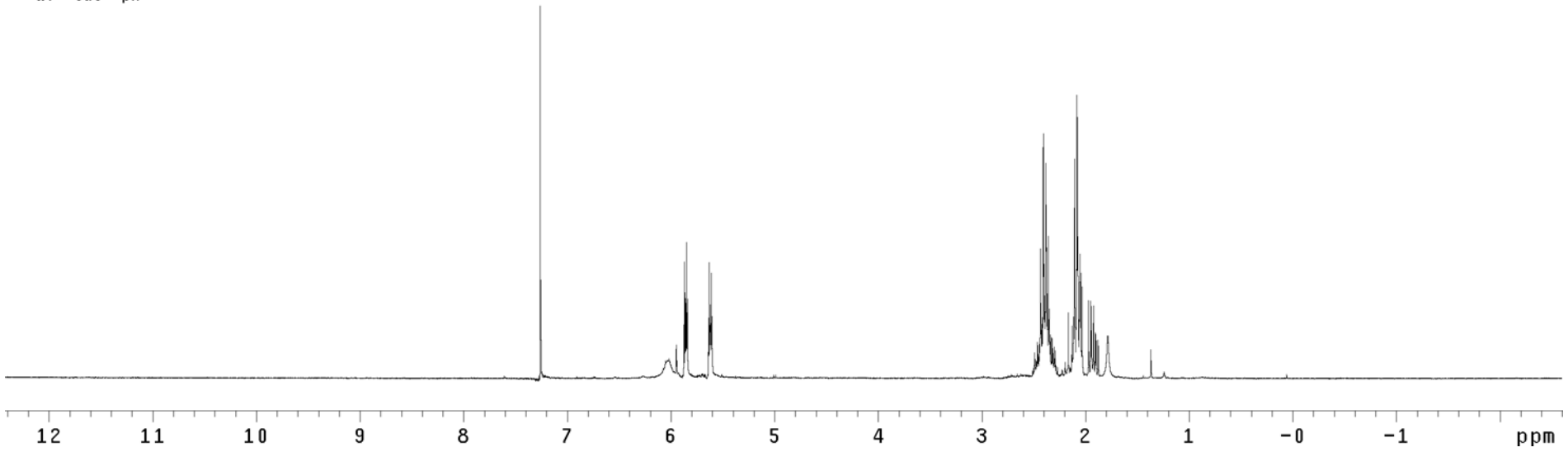


QL-I-259 after column on hg2

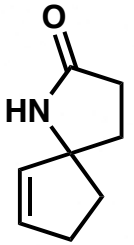
exp1 std13c

SAMPLE		DEC. & VT	
date	Oct 23 2005	dfrq	299.818
solvent	CDCl3	dn	H1
file	/home/qil/hg2~	dpwr	35
	/QL-I-259C13.fid	dof	0
	ACQUISITION	dm	yyy
sfrq	75.396	dmm	w
tn	C13	dmf	7900
at	1.815		PROCESSING
np	68106	lb	1.00
sw	18761.7	wtfile	
fb	10400	proc	ft
bs	4	fn	not used
tpwr	62		
pw	8.7	werr	
d1	0	wexp	
tof	0	wbs	
nt	1024	wnt	
ct	184		
alock	n		
gain	not used		

FLAGS	
il	n
in	n
dp	y

DISPLAY	
sp	-1827.2
wp	18761.4
vs	106
sc	0
wc	250
hzmm	75.05
is	500.00
rfl	1827.5
rfp	0
th	15
ins	100.000

ai no ph



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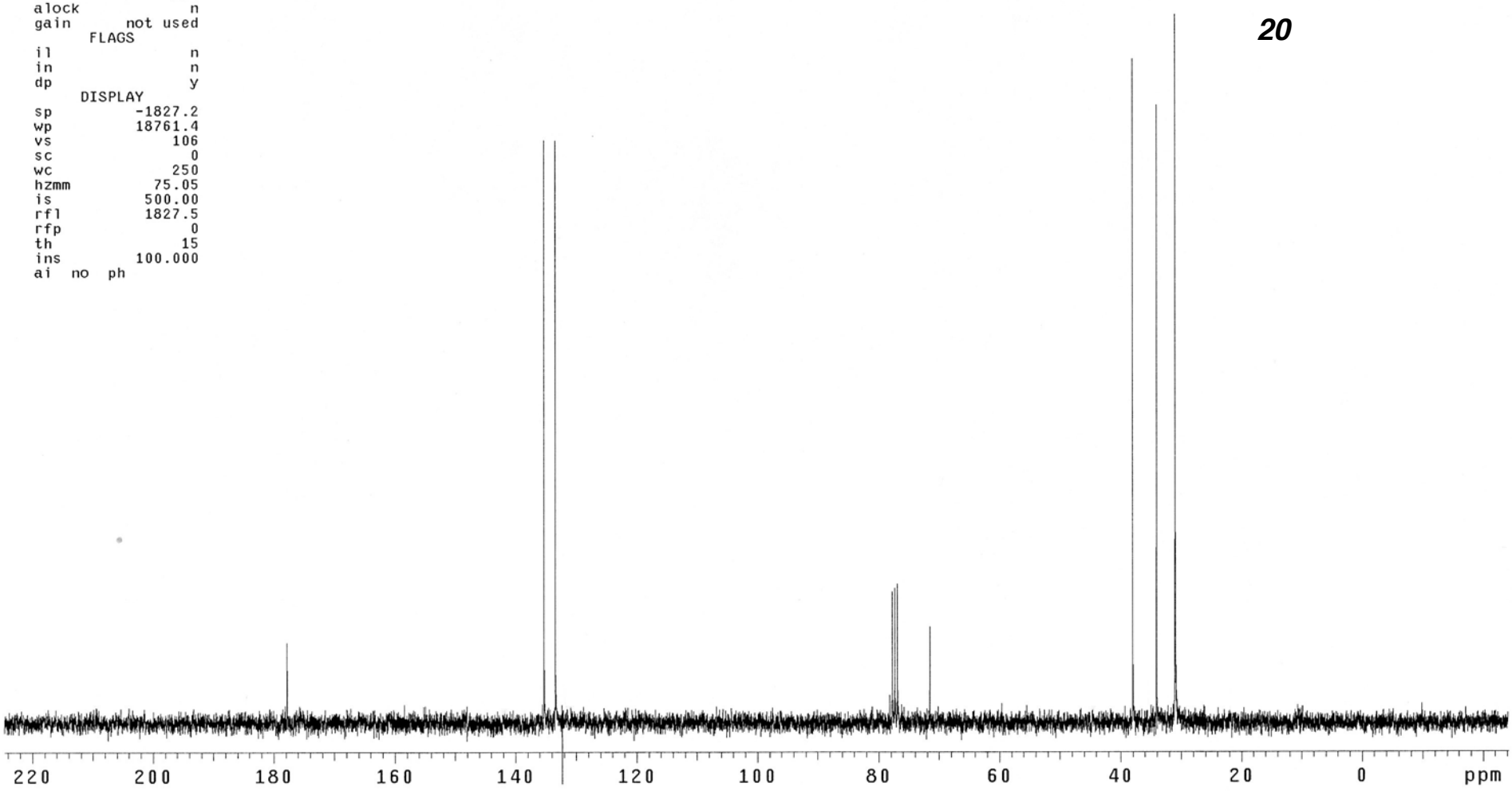
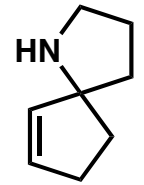


```
QL-IV-033 on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            30
    /QL-IV-033.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.869  dmm              c
tn              H1  dmf            200
at            1.995     PROCESSING
np            17984  wtfile
sw           4506.5  proc            ft
fb        not used  fn        not used
bs               4
tpwr            55  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct              36
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -780.5
wp          4506.3
vs             228
sc               0
wc             250
hzmm          1.17
is          500.00
rfl           780.8
rfp              0
th              11
ins           1.000
ai  cdc  ph
```



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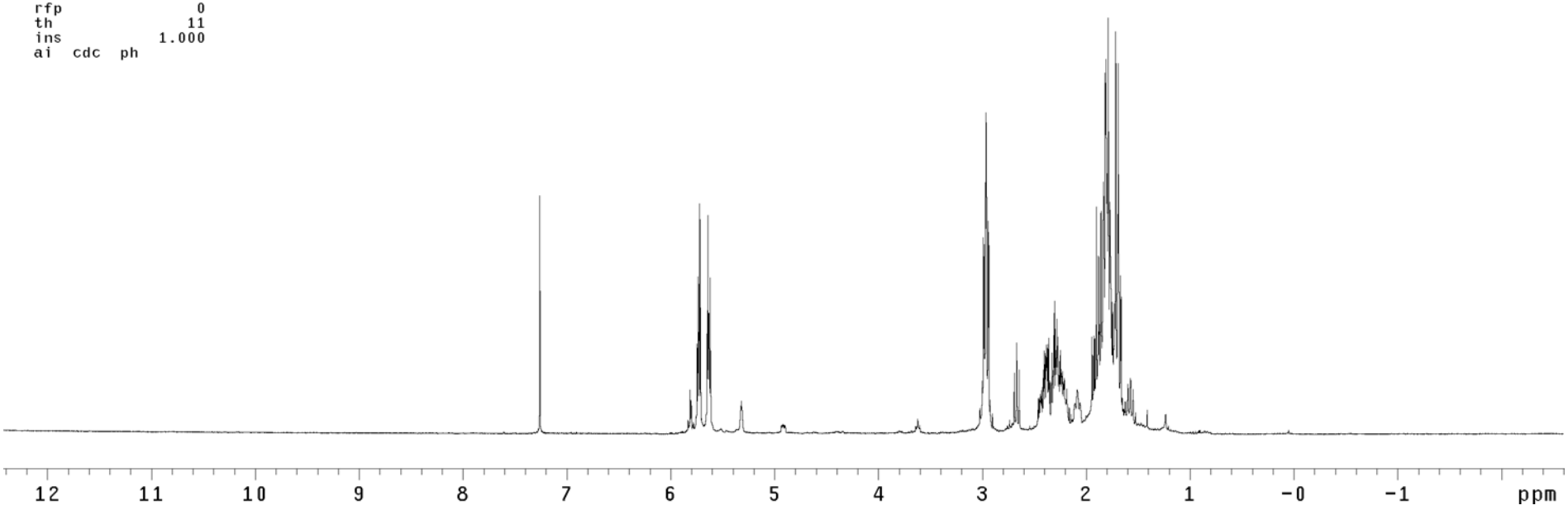
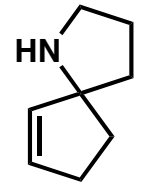
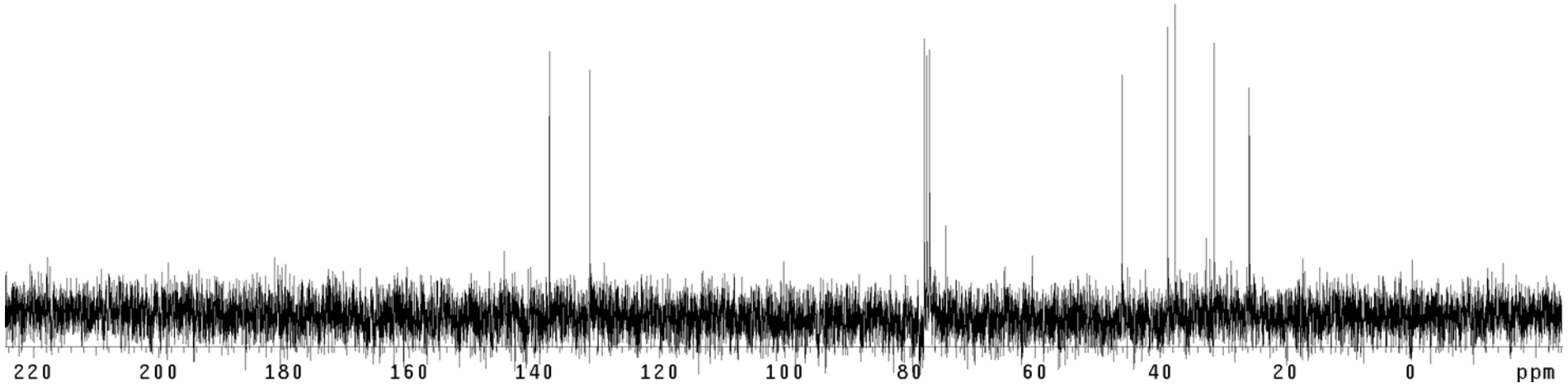


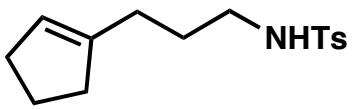
```
QL-IV-033 on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/ql1/hg1~ dpwr             36
/QL-IV-033_C13.fid  dof               0
   ACQUISITION      dm              yyy
sfrq         75.409  dmm               w
tn             C13  dmf            6500
at           1.815      PROCESSING
np           68106  lb            1.00
sw         18761.7  wtfile
fb           10400  proc             ft
bs               8  fn        not used
tpwr            59
pw              8.7  werr
d1                0  wexp
tof               0  wbs
nt             1024  wnt
ct               64
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp         -1829.7
wp         18761.4
vs             270
sc               0
wc             250
hzmm          4.10
is          500.00
rfl         1830.0
rfp              0
th              15
ins         100.000
ai  no  ph
```



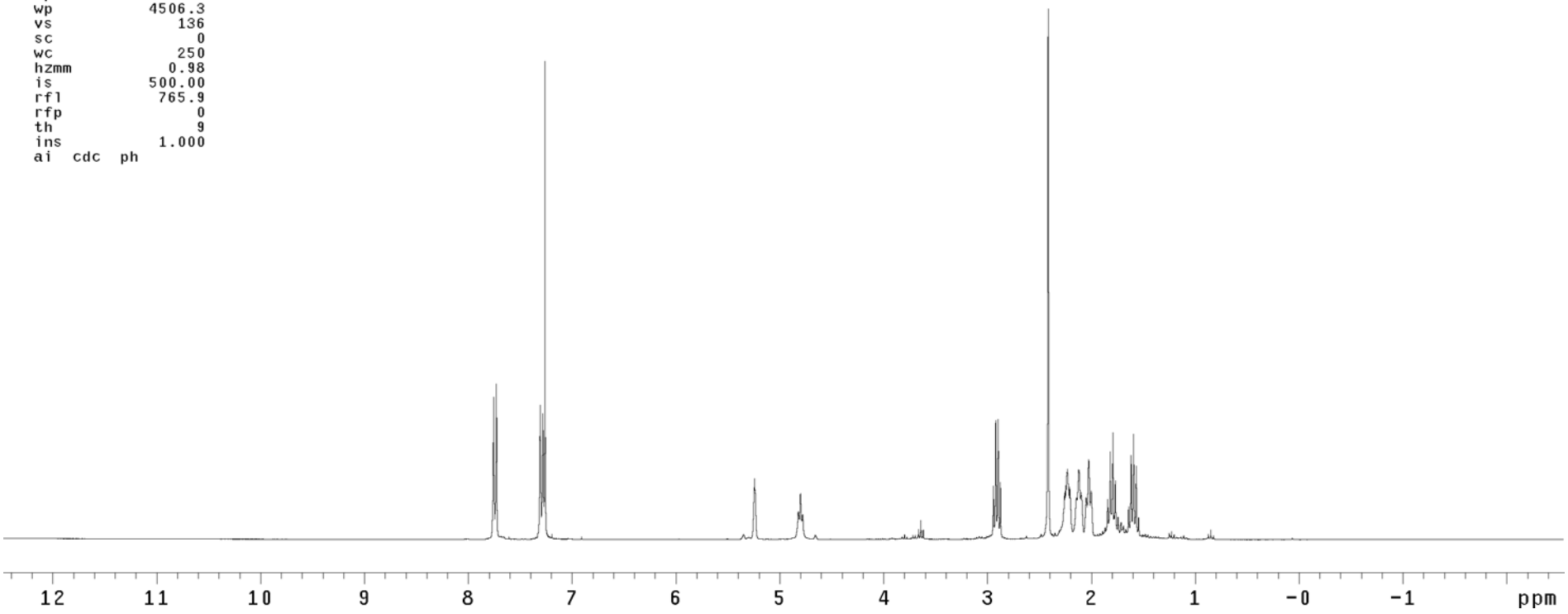
7a

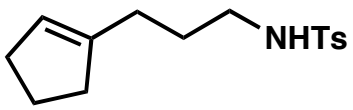


```
QL-I-195 on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
     /QL-I-195.fid  dof              0
   ACQUISITION      dm            nnn
sfrq       299.818  dmm             c
tn              H1  dmf           200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb       not used  fn       not used
bs              4
tpwr           56  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct             52
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -765.7
wp         4506.3
vs            136
sc              0
wc            250
hzmm         0.98
is         500.00
rfl         765.9
rfp             0
th              9
ins         1.000
ai  cdc  ph
```



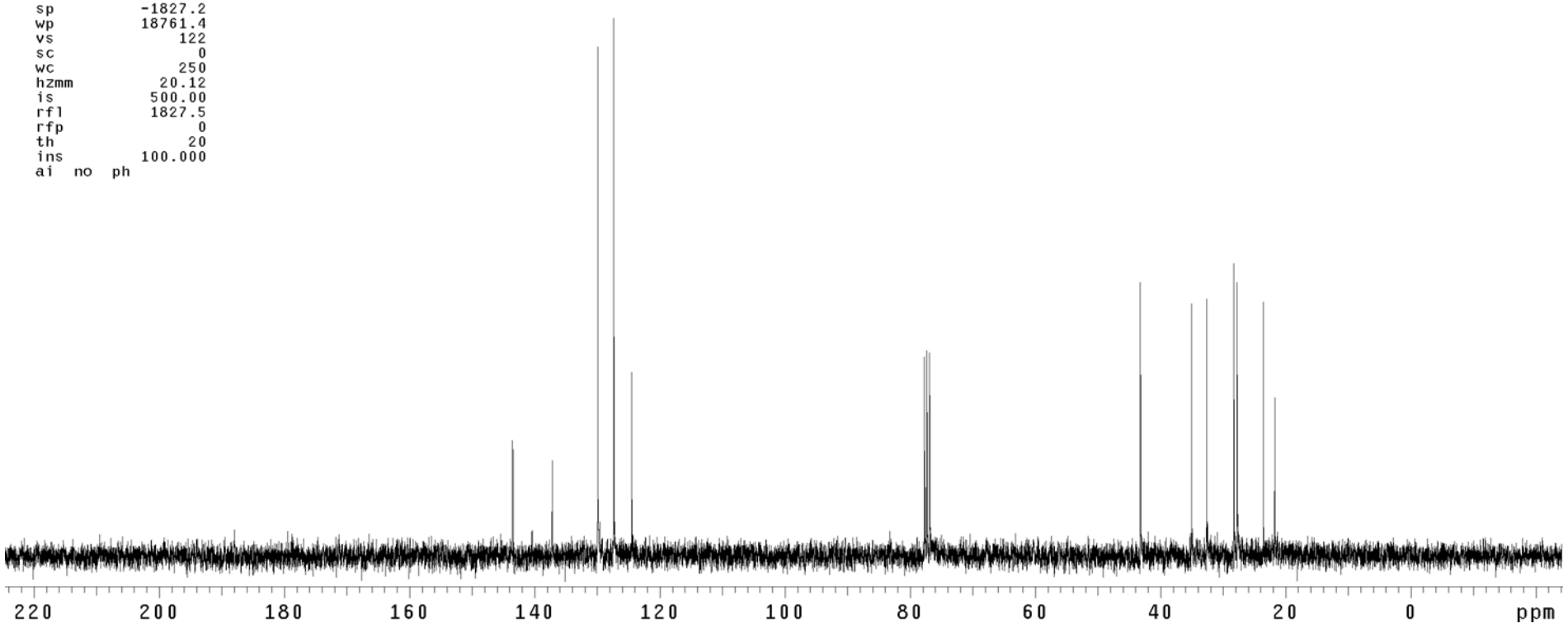
21



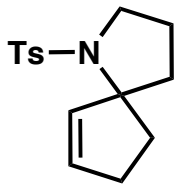


21

```
QL-I-195 on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date   Aug 24 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr             35
 /QL-I-195_C13.fid  dof               0
   ACQUISITION      dm             yyy
sfrq         75.396  dmm              w
tn              C13  dmf           7300
at            1.815      PROCESSING
np            68106  lb            1.00
sw          18761.7  wtfile
fb            10400  proc            ft
bs                8  fn        not used
tpwr             62
pw              8.7  werr
d1                0  wexp
tof               0  wbs
nt             1024  wnt
ct               72
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs              122
sc                0
wc              250
hzmm          20.12
is           500.00
rfl          1827.5
rfp               0
th               20
ins         100.000
ai  no  ph
```



```
QL-I-205 after column on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date   Sep 30 2005  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/ql1/hg2~ dpwr            30
     /QL-I-205.fid  dof              0
   ACQUISITION      dm            nnn
sfrq       299.818  dmm             c
tn              H1  dmf           200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb       not used  fn       not used
bs              4
tpwr           56  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct             12
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -765.7
wp         4506.3
vs             80
sc              0
wc            250
hzmm         4.00
is         500.00
rfl         765.9
rfp             0
th             20
ins       100.000
ai  cdc  ph
```



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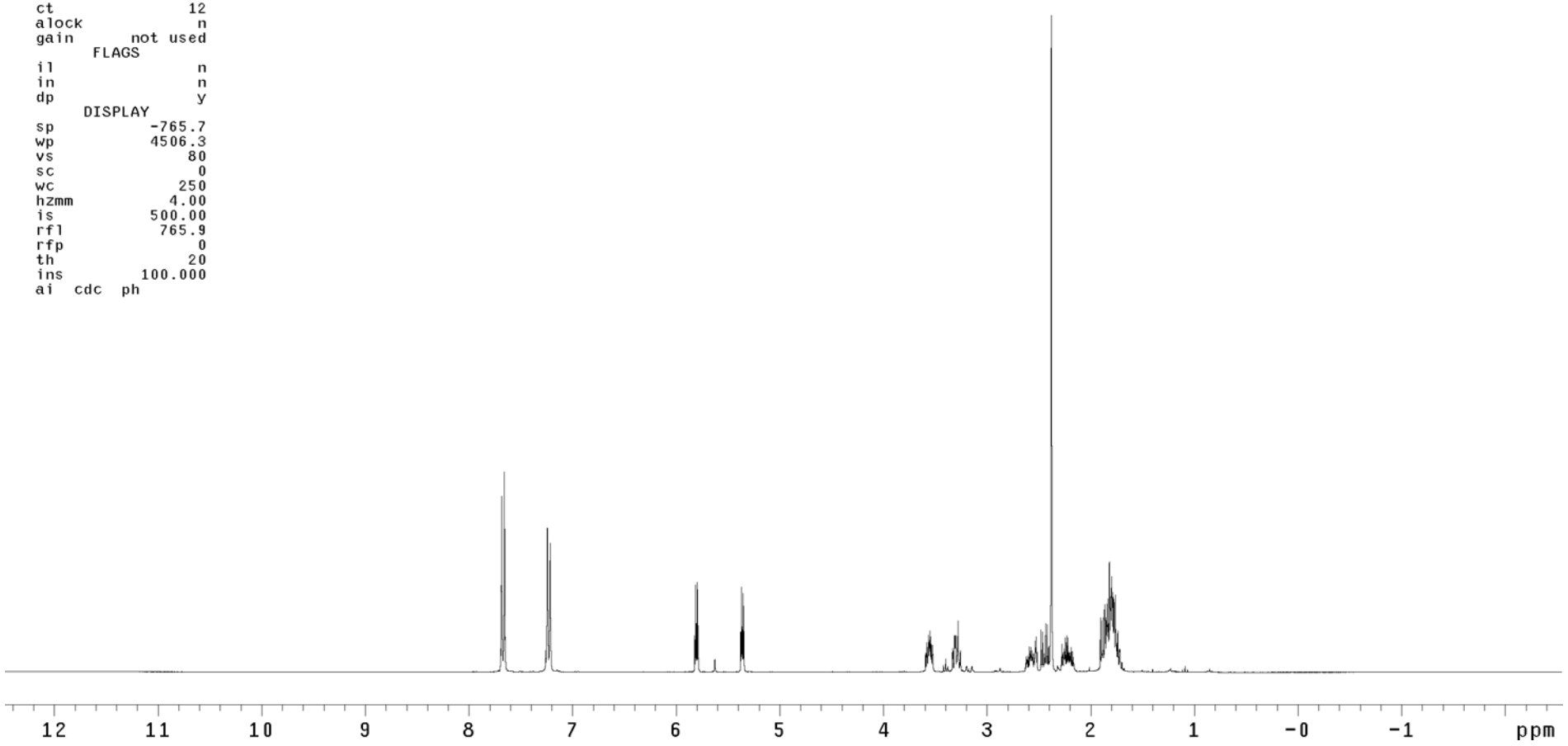
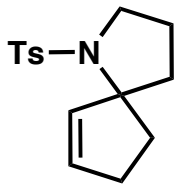


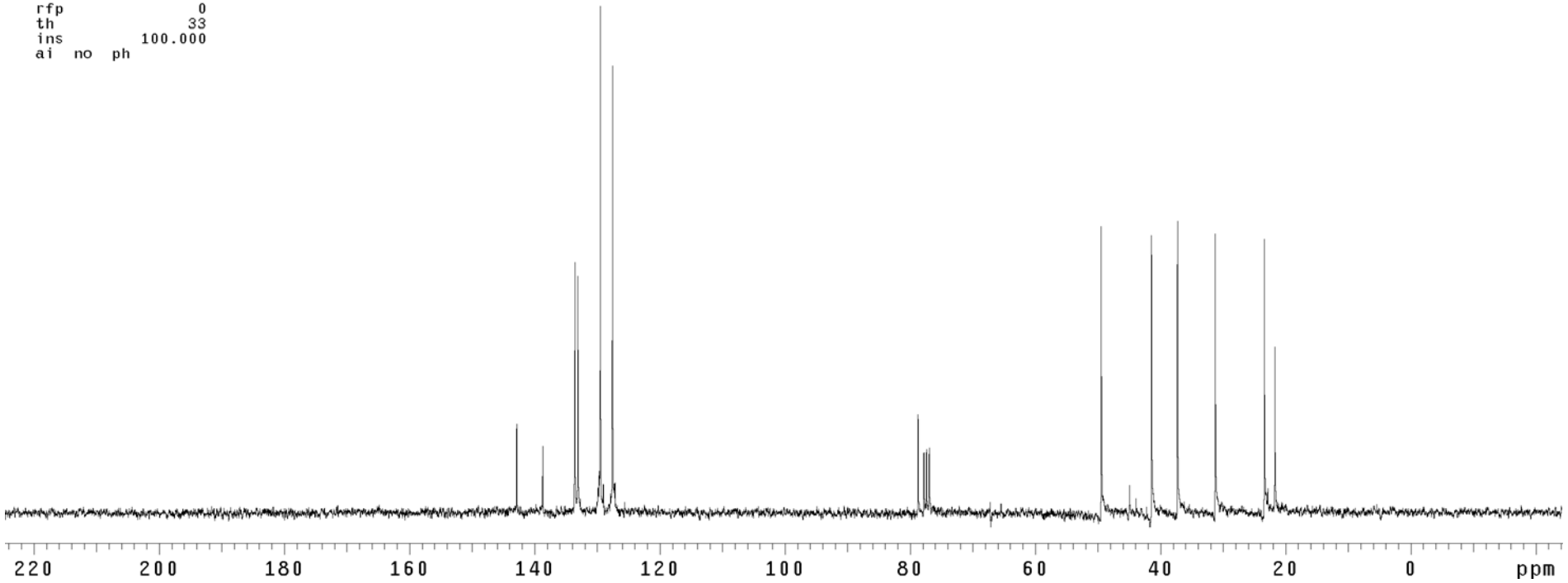
QL-I-205 after column on hg2

exp1 std13c

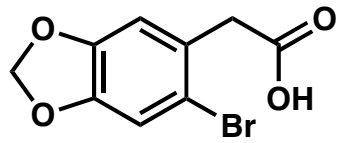
SAMPLE		DEC. & VT	
date	Sep 30 2005	dfrq	299.818
solvent	CDCl3	dn	H1
file	/home/qil/hg2~ /QL-I-205_C13.fid	dpwr	35
		dof	0
ACQUISITION		dm	yyy
sfrq	75.396	dmm	w
tn	C13	dmf	7900
at	1.815	PROCESSING	
np	68106	lb	3.00
sw	18761.7	wtfile	
fb	10400	proc	ft
bs	4	fn	not used
tpwr	62		
pw	8.7	werr	
d1	0	wexp	
tof	0	wbs	
nt	1024	wnt	
ct	204		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1827.2		
wp	18761.4		
vs	131		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	1827.5		
rfp	0		
th	33		
ins	100.000		
ai no ph			



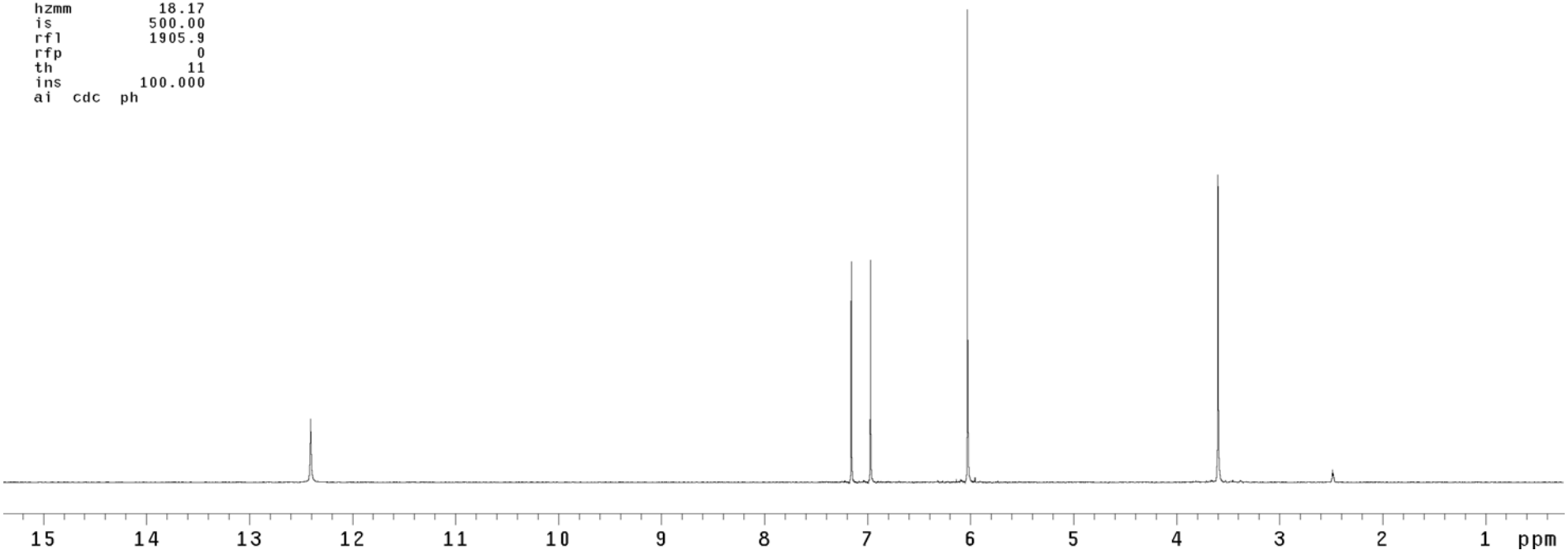
7b



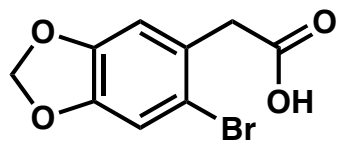
```
QL-II-051 on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Jul 11 2006  dfrq       299.870
solvent       DMSO  dn              H1
file /home/qil/hg1~ dpwr            30
    /QL-II-051.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.870  dmm              c
tn              H1  dmf            200
at           1.997      PROCESSING
np           26982  wtfile
sw          6756.8  proc            ft
fb       not used  fn       not used
bs              16
tpwr            55  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt               4
ct               4
alock            n
gain     not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp            71.2
wp          4543.4
vs              24
sc               0
wc             250
hzmm         18.17
is          500.00
rfl         1905.9
rfp              0
th              11
ins         100.000
ai  cdc  ph
```



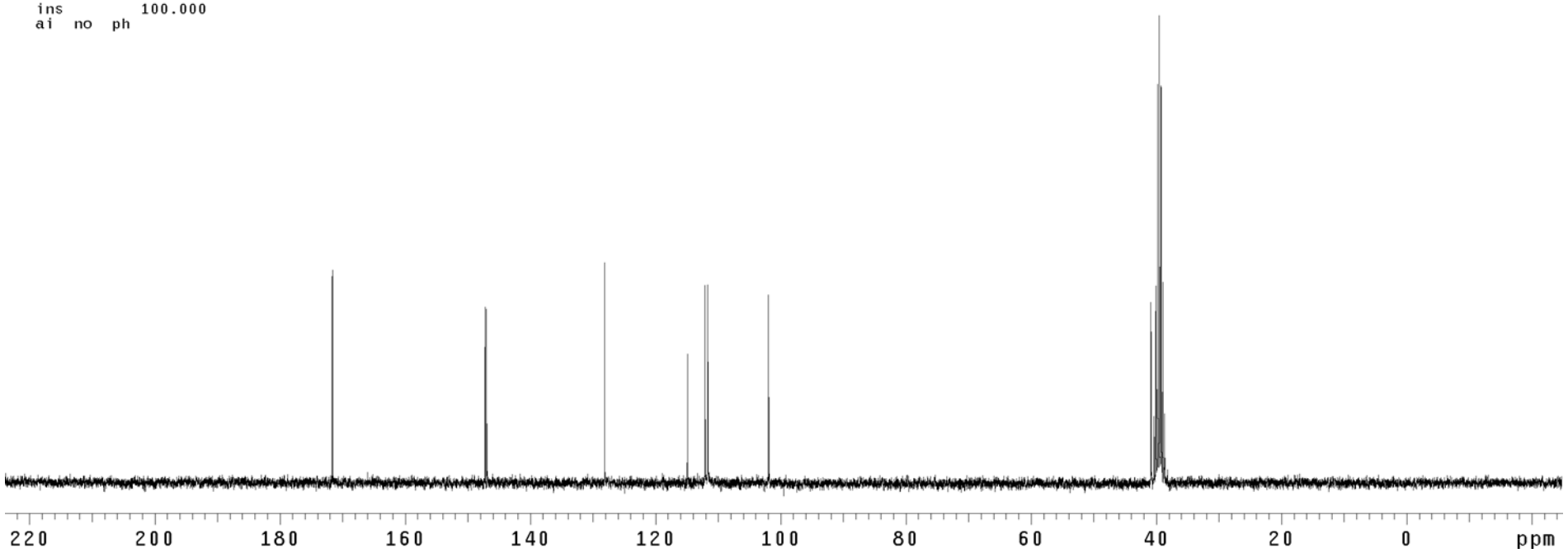
23



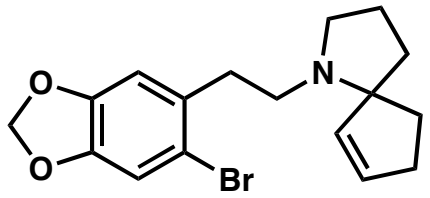
```
QL-II-051 on hg2

exp1  std13c

      SAMPLE
date   Jul 11 2006
solvent       DMSO
file /home/qil/hg1~
/QL-II-051_C13.fid
   ACQUISITION
sfrq        75.410
tn             C13
at             1.815
np            68106
sw          18761.7
fb            10400
bs                8
tpwr             59
pw              8.7
d1                0
tof               0
nt             1024
ct              312
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1877.2
wp          18761.4
vs               70
sc                0
wc              250
hzmm           2.86
is           500.00
rfl          4855.9
rfp          2978.4
th               20
ins         100.000
ai  no  ph

      DEC. & VT
dfrq        299.870
dn               H1
dpwr             36
dof               0
dm              yyy
dmm               w
dmf            6500
     PROCESSING
lb             1.00
wtfile
proc             ft
fn        not used

werr
wexp
wbs
wnt
```



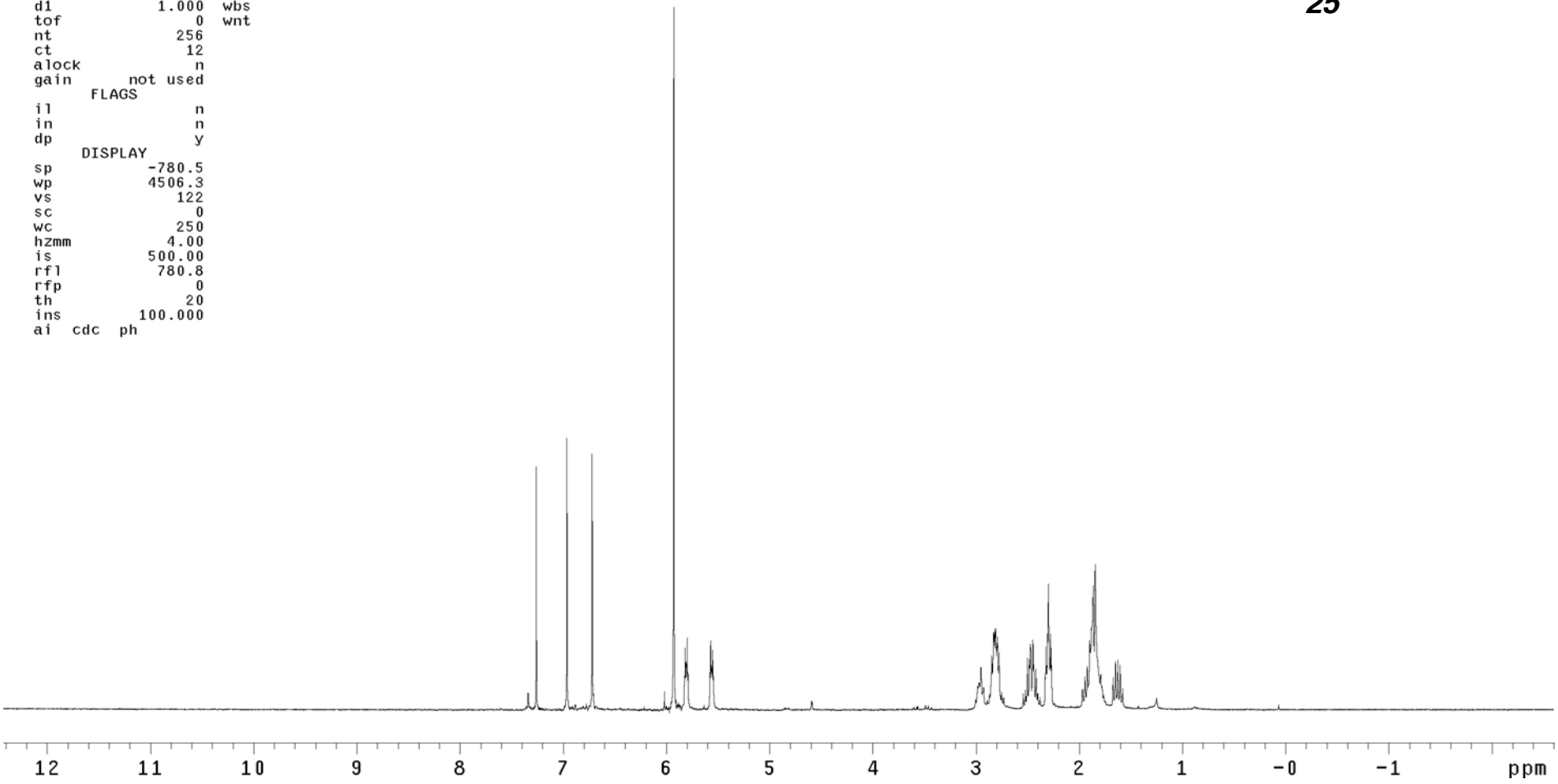
23



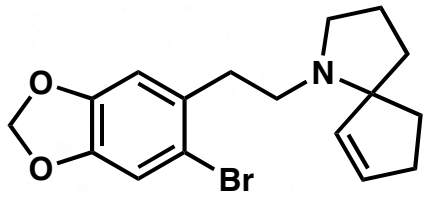
```
QL-II-273 retake on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 30 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            30
    /QL-II-273.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.869  dmm              c
tn              H1  dmf            200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb       not used  fn       not used
bs              4
tpwr           55  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct             12
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -780.5
wp         4506.3
vs            122
sc              0
wc            250
hzmm         4.00
is         500.00
rfl         780.8
rfp             0
th             20
ins       100.000
ai  cdc  ph
```



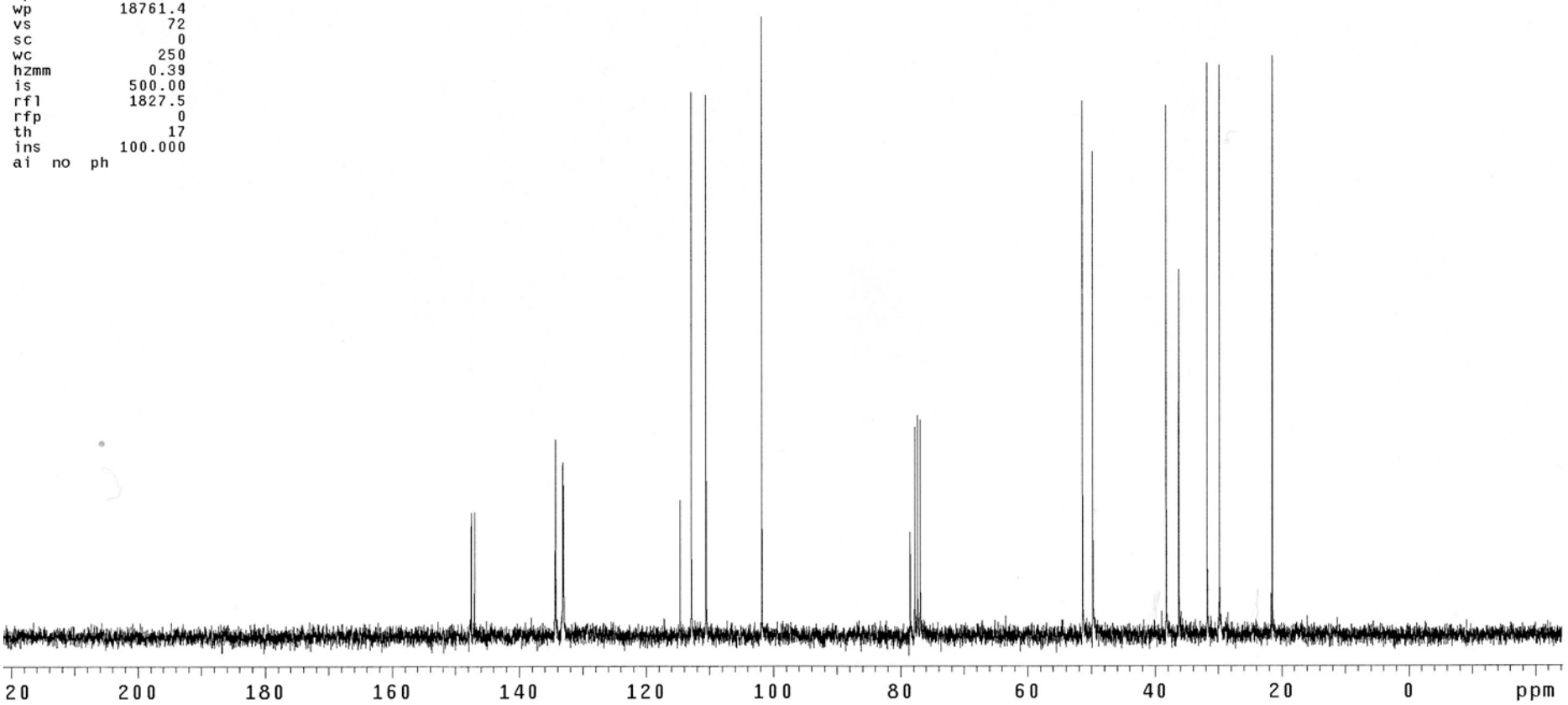
25



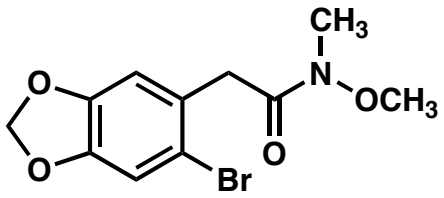
```
QL-II-271 after column on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date    Mar  7 2006  dfrq       299.818
solvent      CDCl3   dn              H1
file /home/qil/hg2~  dpwr            35
/QL-II-271_C13.fid   dof              0
   ACQUISITION       dm             yyy
sfrq         75.396  dmm              w
tn              C13  dmf           7300
at            1.815      PROCESSING
np            68106  lb           1.00
sw          18761.7  wtfile
fb            10400  proc           ft
bs                8  fn       not used
tpwr             62
pw              8.7  werr
d1                0  wexp
tof               0  wbs
nt             1024  wnt
ct                0
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs               72
sc                0
wc              250
hzmm           0.39
is           500.00
rfl          1827.5
rfp               0
th               17
ins         100.000
ai  no  ph
```



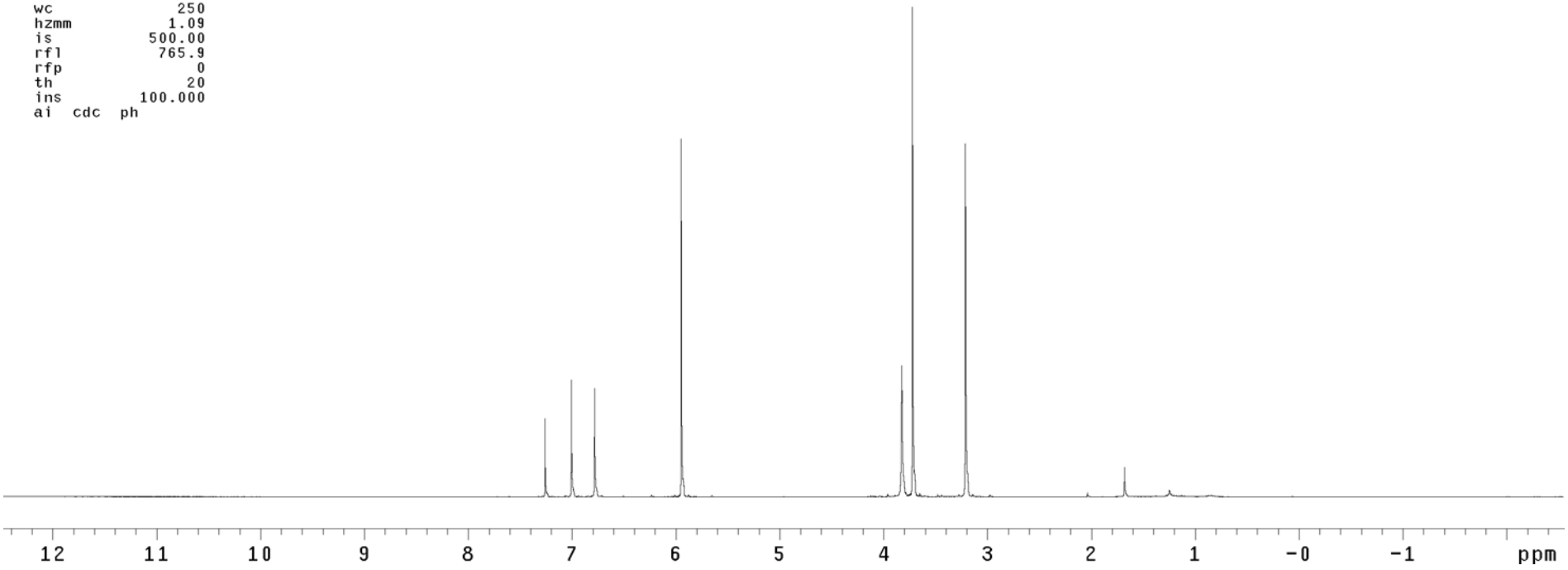
25



```
QL-IV-129 after FCC on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Sep 14 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
    /QL-IV-129.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc             ft
fb       not used  fn       not used
bs              4
tpwr           56  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct              0
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -765.7
wp         4506.3
vs             49
sc              0
wc            250
hzmm         1.09
is         500.00
rfl         765.9
rfp             0
th             20
ins       100.000
ai  cdc  ph
```



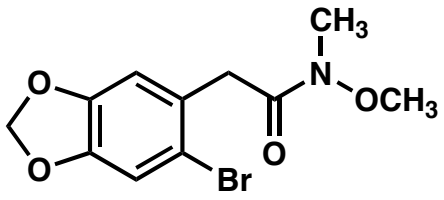
26



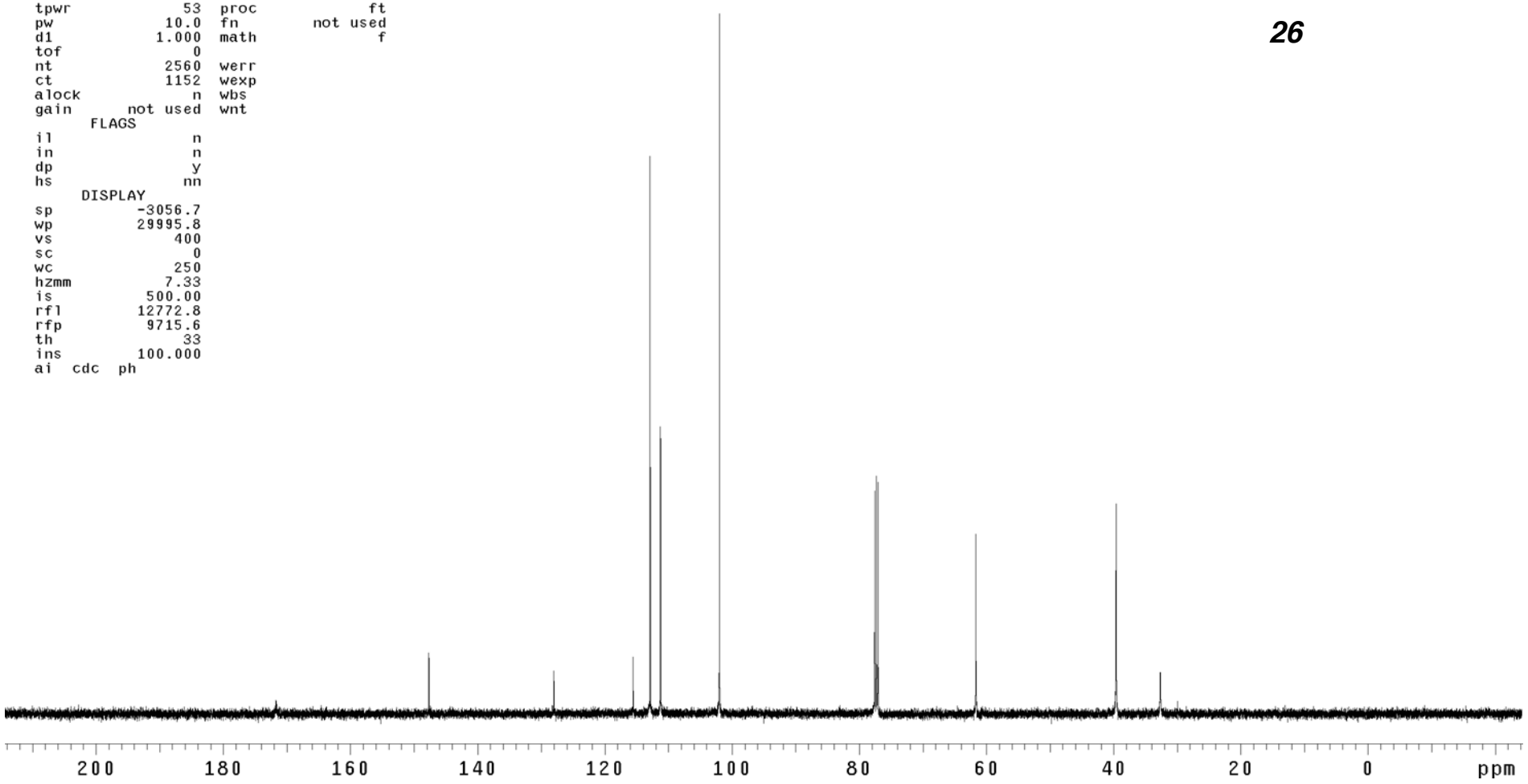
STANDARD CARBON PARAMETERS

exp1 s2pul

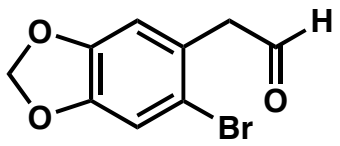
SAMPLE		DEC. & VT	
date	Sep 14 2006	dfrq	499.852
solvent	CDCl3	dn	H1
file	/home/qil/var~	dpwr	41
500/QL-IV-129_C13.~		dof	0
	fid	dm	yyy
ACQUISITION		dmm	w
sfrq	125.699	dmf	11696
tn	C13	dseq	
at	1.300	dres	1.0
np	78020	homo	n
sw	29996.3	PROCESSING	
fb	17000	lb	0.50
bs	64	wtfile	
tpwr	53	proc	ft
pw	10.0	fn	not used
d1	1.000	math	f
tof	0		
nt	2560	werr	
ct	1152	wexp	
alock	n	wbs	
gain	not used	wnt	
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-3056.7		
wp	29995.8		
vs	400		
sc	0		
wc	250		
hzmm	7.33		
is	500.00		
rfl	12772.8		
rfp	9715.6		
th	33		
ins	100.000		
ai cdc ph			



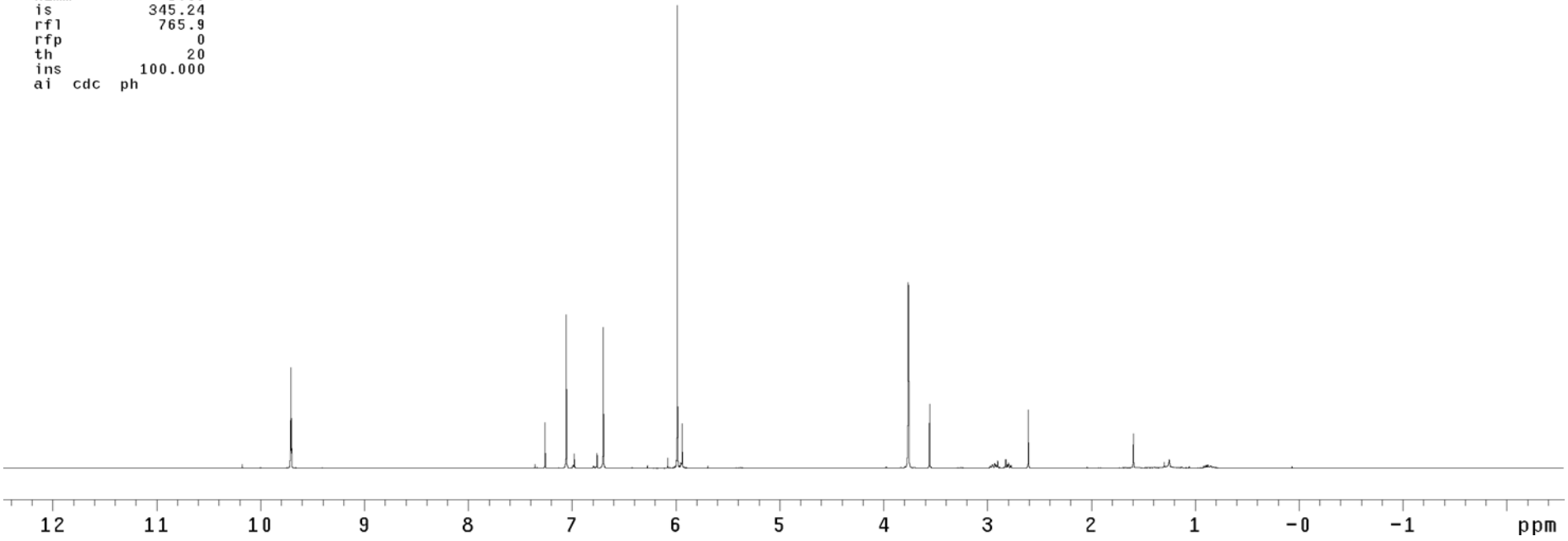
26



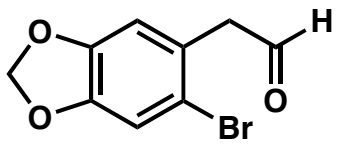
```
QL-IV-131 after FCC on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Sep 19 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
    /QL-IV-131.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at            1.995      PROCESSING
np            17984  wtfile
sw           4506.5  proc            ft
fb        not used  fn        not used
bs               4
tpwr            56  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs              18
sc               0
wc             250
hzmm          3.06
is          345.24
rfl           765.9
rfp              0
th              20
ins         100.000
ai  cdc  ph
```



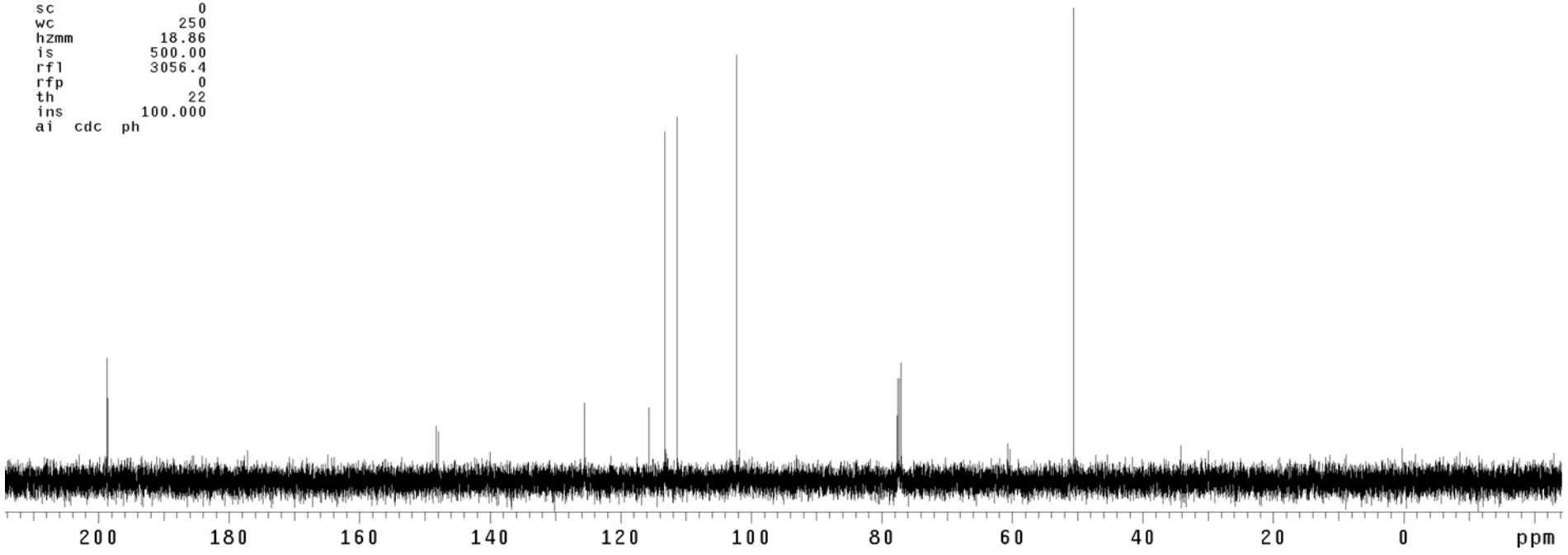
27

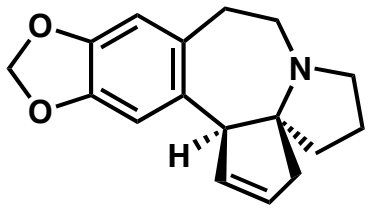


```
QL-IV-131 on I500

exp1  s2pul

      SAMPLE            DEC. & VT
date   Sep 14 2006  dfrq       499.852
solvent      CDCl3  dn              H1
file /home/ql/var~  dpwr            41
500/QL-IV-131_C13.~ dof              0
               fid  dm             yyy
   ACQUISITION      dmm              w
sfrq       125.699  dmf          11696
tn              C13  dseq
at            1.300  dres           1.0
np            78020  homo             n
sw         29996.3       PROCESSING
fb            17000  lb            0.50
bs                4  wtfile
tpwr             53  proc             ft
pw             10.0  fn        not used
d1            1.000  math             f
tof               0
nt             1024  werr
ct               12  wexp
alock             n  wbs
gain      not used  wnt
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp          -3055.9
wp          29995.8
vs              137
sc                0
wc              250
hzmm          18.86
is           500.00
rfl          3056.4
rfp               0
th               22
ins         100.000
ai  cdc  ph
```



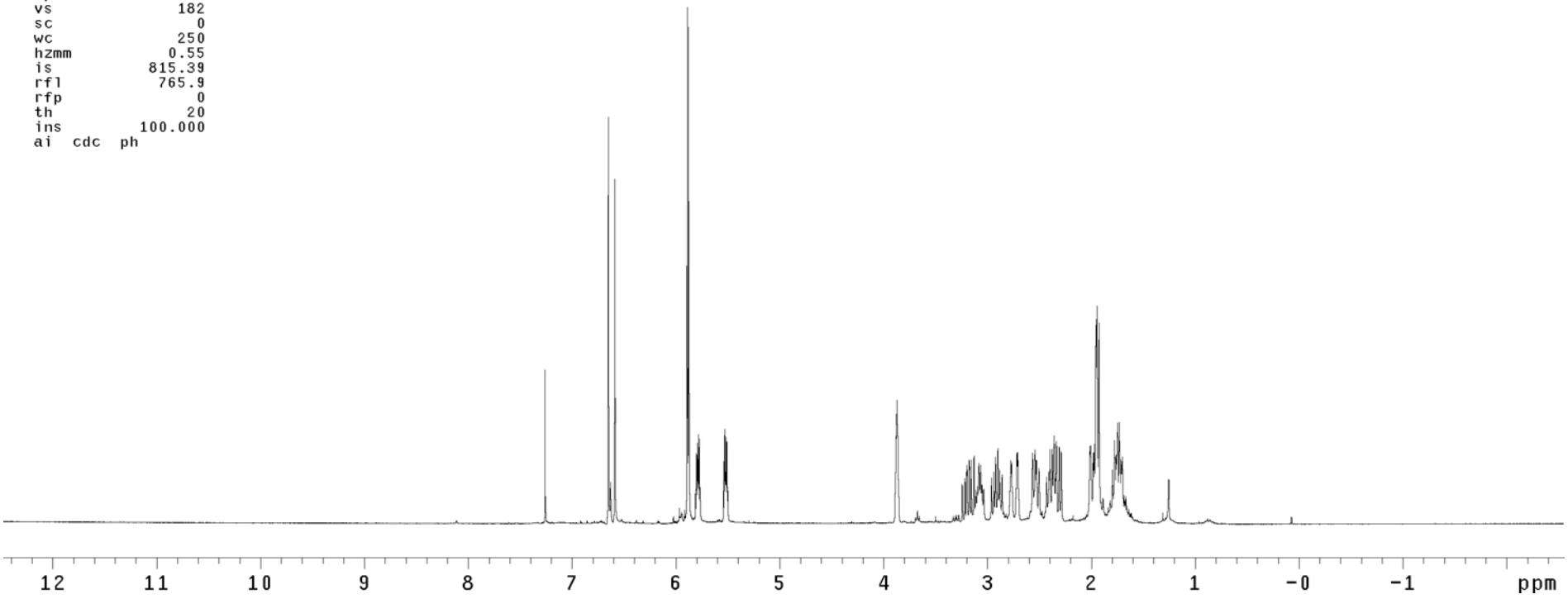
27





29

```
QL-II-295 after column on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date    Mar 15 2006  dfrq        299.818
solvent      CDCl3  dn                H1
file /home/qil/hg2~ dpwr              30
    /QL-II-295.fid  dof                0
   ACQUISITION      dm               nnn
sfrq        299.818  dmm                c
tn               H1  dmf              200
at             1.995      PROCESSING
np             17984  wtfile
sw            4506.5  proc              ft
fb         not used  fn         not used
bs                4
tpwr             56  werr
pw              7.0  wexp
d1            1.000  wbs
tof               0  wnt
nt              256
ct                0
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp           -765.7
wp           4506.3
vs              182
sc                0
wc              250
hzmm           0.55
is           815.39
rfl           765.9
rfp               0
th               20
ins          100.000
ai  cdc  ph
```

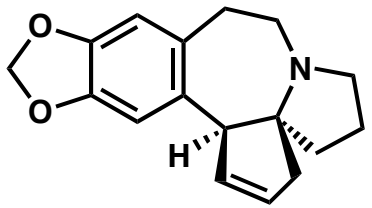


13C OBSERVE

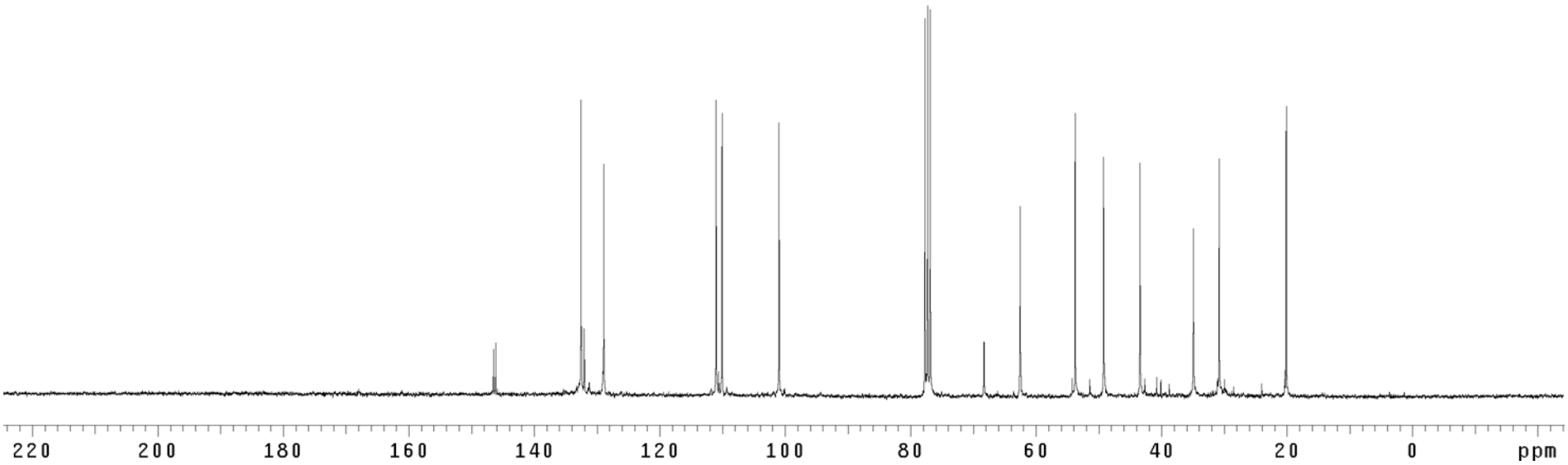
exp1 std13c

SAMPLE		DEC. & VT	
date	Mar 16 2006	dfrq	299.868
solvent	CDCl3	dn	H1
file	/home/qil/hg1~	dpwr	36
	/QL-II-295_C13.fid	dof	0
ACQUISITION		dm	yyy
sfrq	75.409	dmm	w
tn	C13	dmf	6500
at	1.815	PROCESSING	
np	68106	lb	2.00
sw	18761.7	wtfile	
fb	10400	proc	ft
bs	16	fn	not used
tpwr	59		
pw	8.7	werr	
d1	0	wexp	
tof	0	wbs	
nt	25600	wnt	
ct	0		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1823.9		
wp	18761.4		
vs	1416		
sc	0		
wc	250		
hzmm	6.11		
is	500.00		
rfl	1824.2		
rfp	0		
th	18		
ins	100.000		

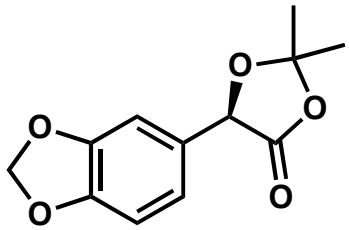
ai no ph



29



```
QL-III-133 spot1 after column on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date   Apr 28 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/ql1/hg2~ dpwr            30
   /QL-III-133.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at            1.995     PROCESSING
np            17984  wtfile
sw           4506.5  proc             ft
fb        not used  fn        not used
bs               4
tpwr            56  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct               8
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs              28
sc               0
wc             250
hzmm          4.00
is          500.00
rfl           765.9
rfp              0
th              20
ins         100.000
ai  cdc  ph
```



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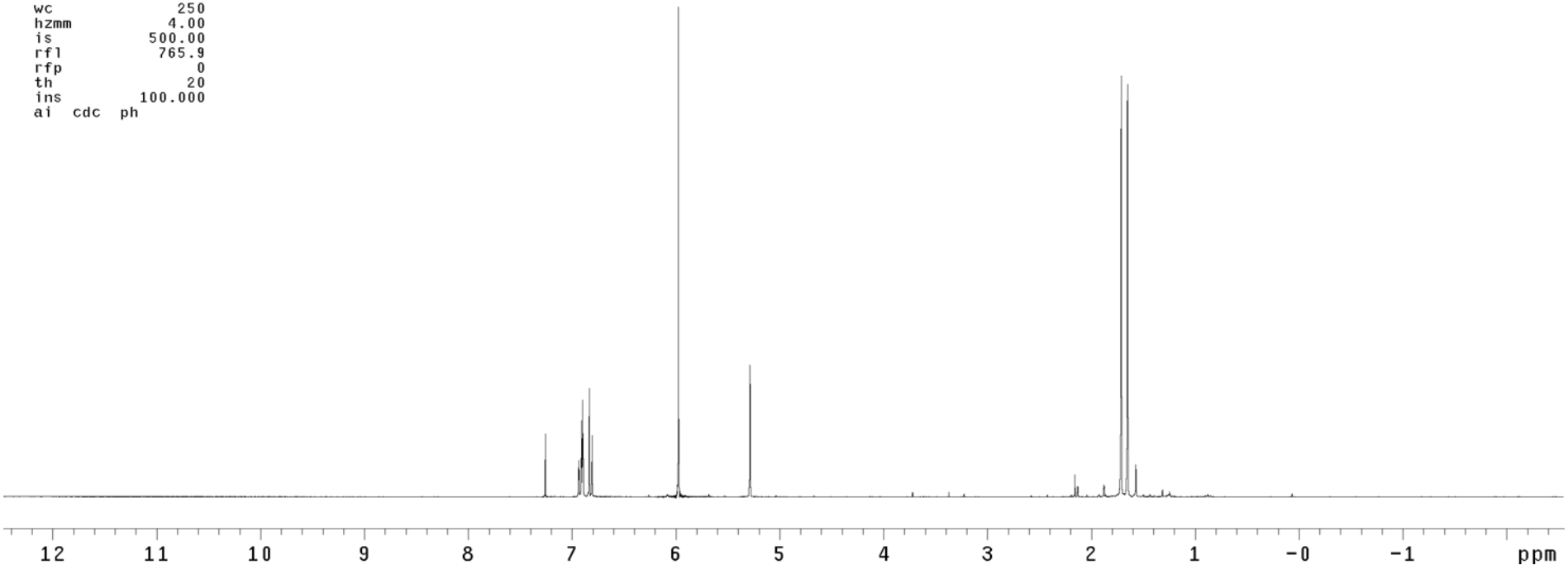
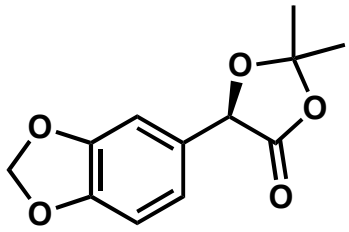


QL-II-153 after extraction on hg1

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 1 2006	dfrq	299.868
solvent	CDCl3	dn	H1
file	/home/qil/hg1~	dpwr	36
	/QL-II-153_C13.fid	dof	0
ACQUISITION		dm	yyy
sfrq	75.409	dmm	w
tn	C13	dmf	6500
at	1.815	PROCESSING	
np	68106	lb	1.00
sw	18761.7	wtfile	
fb	10400	proc	ft
bs	4	fn	not used
tpwr	59		
pw	8.7	werr	
d1	0	wexp	
tof	0	wbs	
nt	1024	wnt	
ct	72		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1823.9		
wp	18761.4		
vs	18		
sc	0		
wc	250		
hzmm	8.68		
is	500.00		
rfl	1824.2		
rfp	0		
th	20		
ins	100.000		

ai no ph



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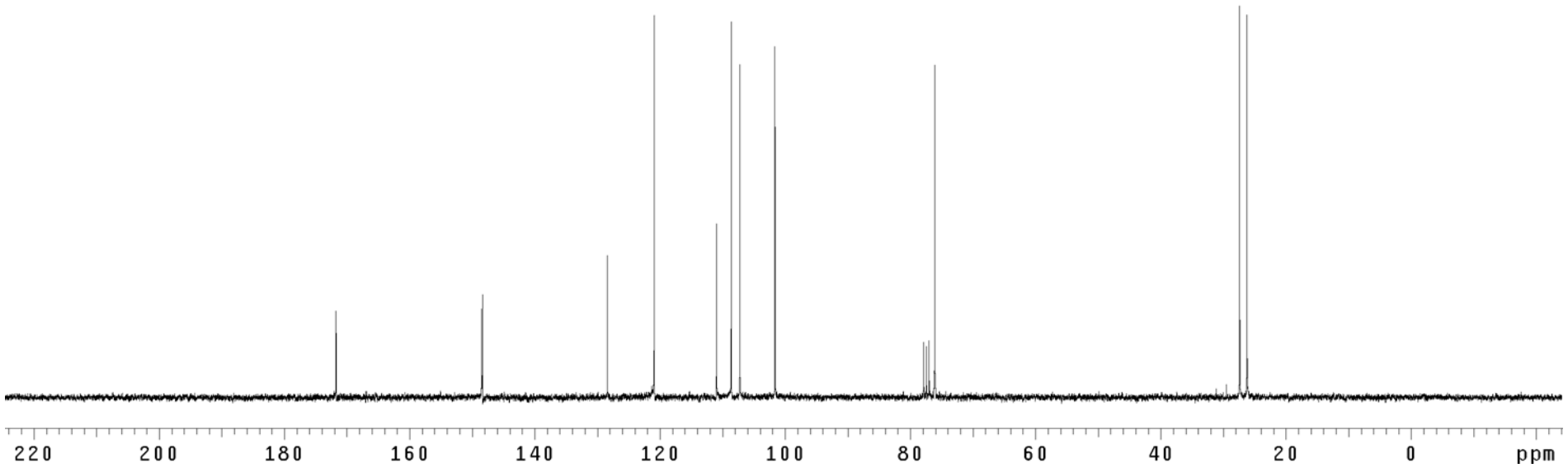
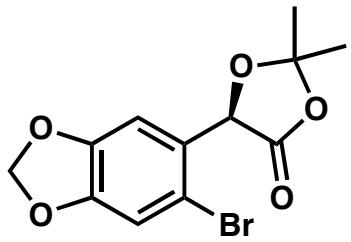
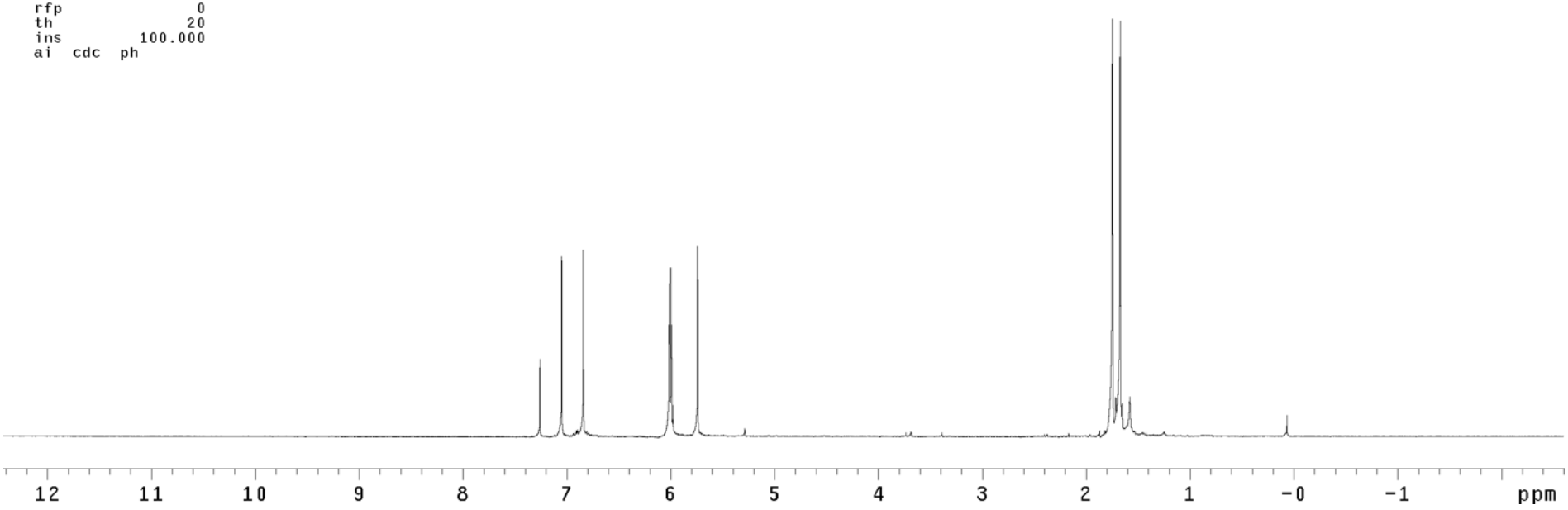


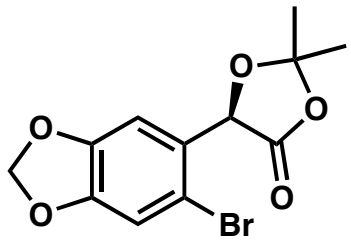
```
QL-III-139 on hg1

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 14 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            30
   /QL-III-139.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.869  dmm              c
tn              H1  dmf            200
at            1.995     PROCESSING
np            17984  wtfile
sw           4506.5  proc            ft
fb        not used  fn        not used
bs               4
tpwr            55  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct              32
alock            n
gain     not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -780.5
wp          4506.3
vs              84
sc               0
wc             250
hzmm          0.51
is          500.00
rfl          780.8
rfp              0
th              20
ins         100.000
ai  cdc  ph
```



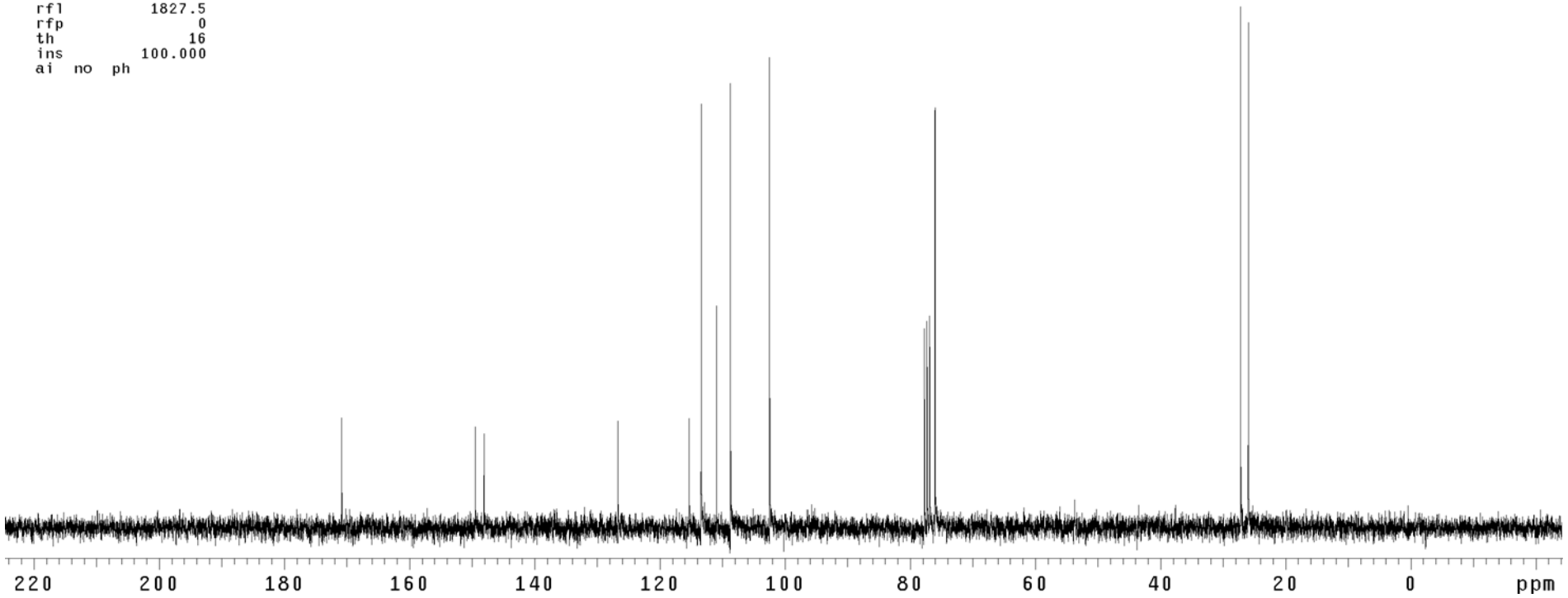
(R)-32



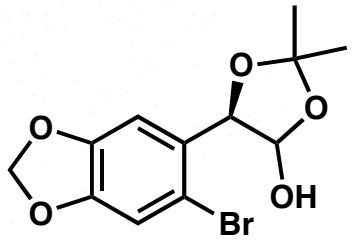
```
QL-II-281 pure on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date   Mar 12 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            35
/QL-II-281-C13.fid  dof              0
   ACQUISITION      dm             yyy
sfrq        75.396  dmm              w
tn             C13  dmf           7300
at           1.815      PROCESSING
np           68106  lb           1.00
sw         18761.7  wtfile
fb           10400  proc           ft
bs               8  fn       not used
tpwr            62
pw             8.7  werr
d1               0  wexp
tof              0  wbs
nt            1024  wnt
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp         -1827.2
wp         18761.4
vs              74
sc               0
wc             250
hzmm         17.75
is          500.00
rfl         1827.5
rfp              0
th              16
ins         100.000
ai  no  ph
```



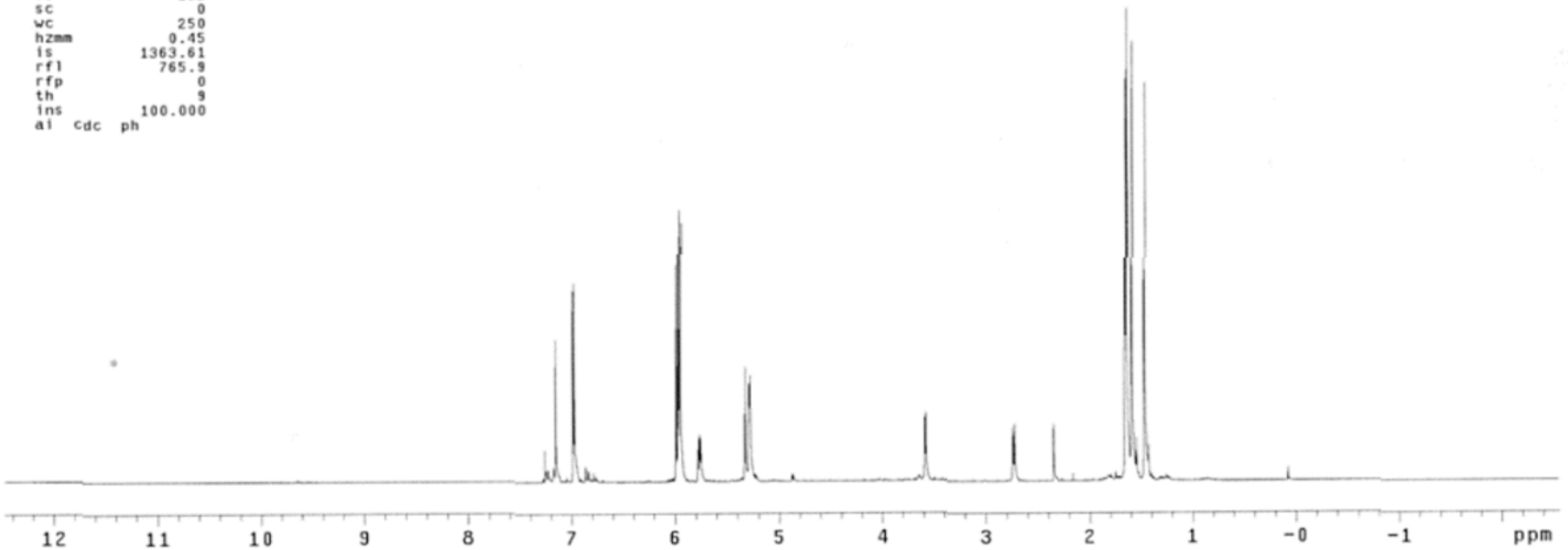
(R)-32



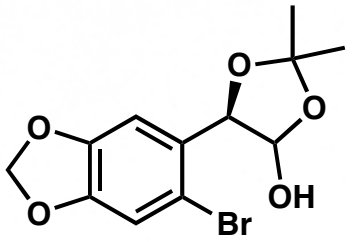
```
QL-III-147 after extraction crude on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date    May  2 2006  dfrq        299.818
solvent      CDCl3   dn               H1
file /home/qil/hg2~  dpwr             30
/QL-III-147crude.f~  dof               0
                id   dm              nnn
   ACQUISITION       dmm               c
sfrq        299.818  dmf             200
tn               H1      PROCESSING
at             1.995  wtfile
np             17984  proc            ft
sw            4506.5  fn         not used
fb         not used
bs                4  werr
tpwr             56  wexp
pw              7.0  wbs
d1            1.000  wnt
tof               0
nt              256
ct               12
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp           -765.7
wp           4506.3
vs              153
sc                0
wc              250
hzmm           0.45
is          1363.61
rfl            765.9
rfp               0
th                9
ins         100.000
al  cdc  ph
```



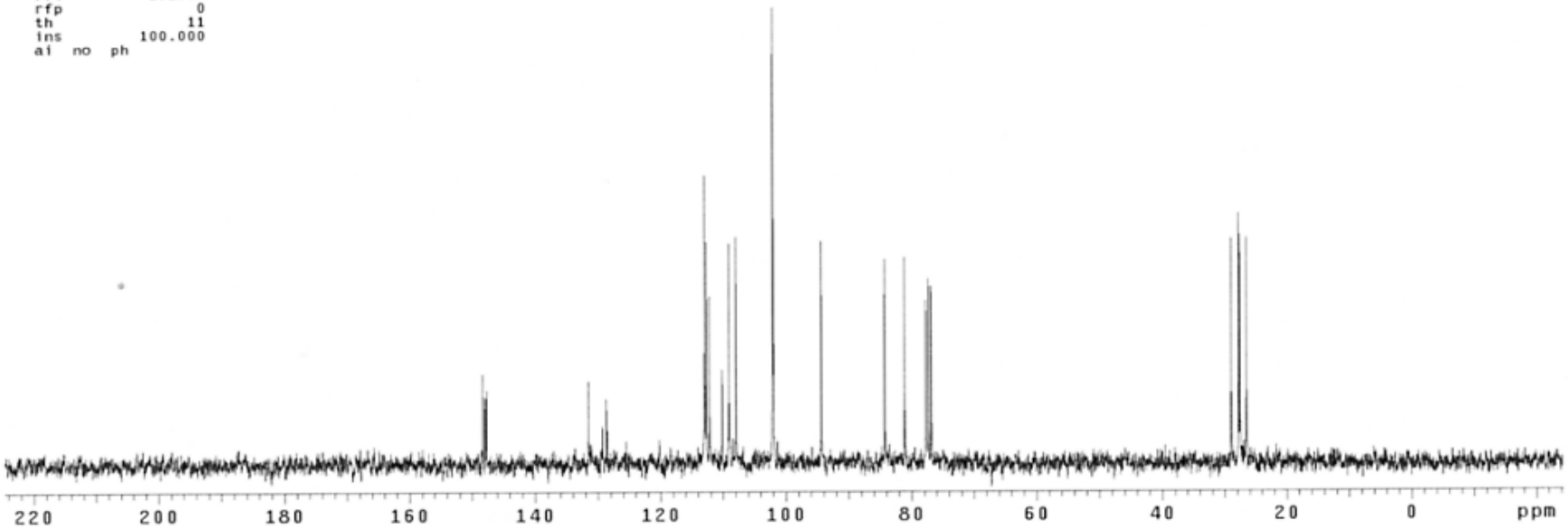
33



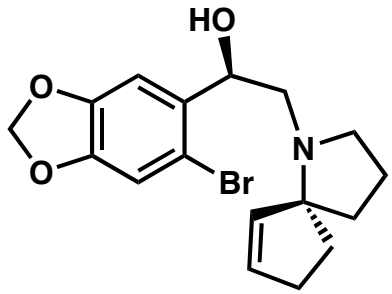
```
QL-III-147crude on hg2

exp1  std13c

      SAMPLE                DEC. & VT
date    May  2 2006  dfrq        299.818
solvent      CDCl3   dn              H1
file /home/qil/hg2~  dpwr            35
/QL-III-147crude_C~  dof              0
             13.fid  dm               yyy
   ACQUISITION       dmm              w
sfrq         75.396  dmf           7300
tn               C13       PROCESSING
at             1.815  lb            3.00
np             68106  wtfile
sw           18761.7  proc            ft
fb             10400  fn        not used
bs                 8
tpwr              62  werr
pw               8.7  wexp
d1                 0  wbs
tof                0  wnt
nt              1024
ct               144
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
     DISPLAY
sp           -1827.2
wp           18761.4
vs               149
sc                 0
wc               250
hzmm            6.70
is            500.00
rfl           1827.5
rfp                0
th                11
ins           100.000
ai  no  ph
```



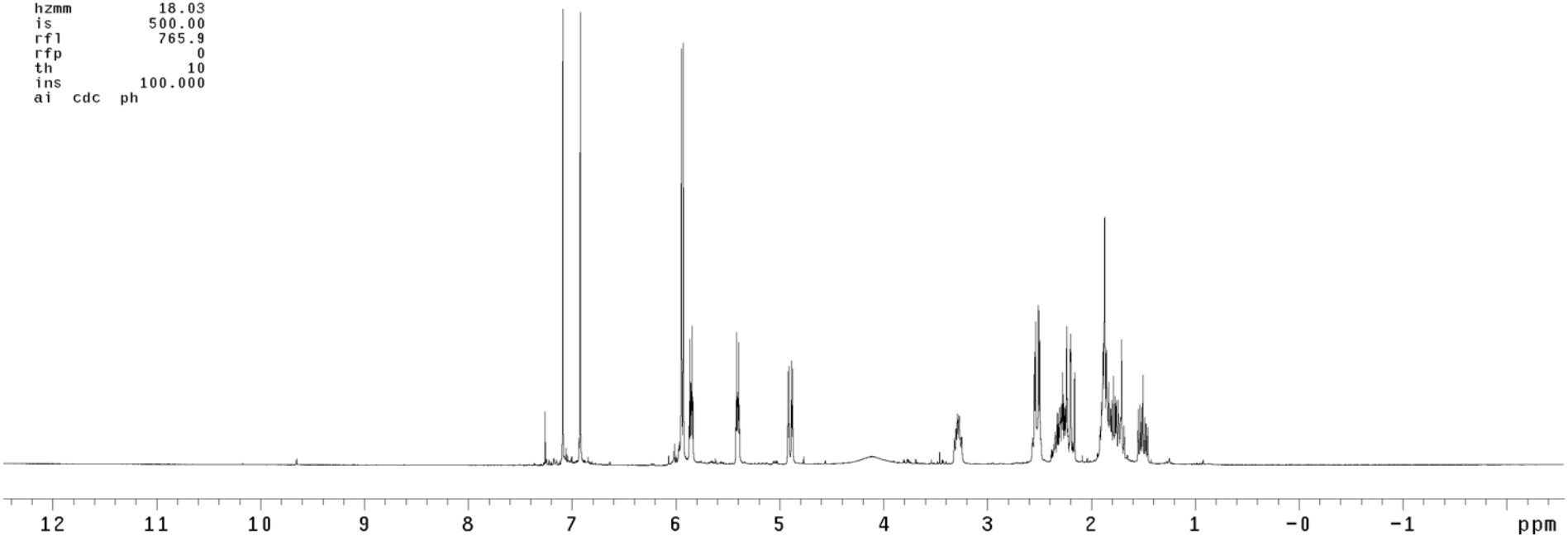
33



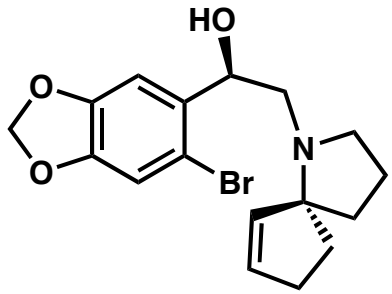
```
QL-II-297 diastereomer 1  on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date    Mar 17 2006  dfrq        299.818
solvent      CDCl3  dn               H1
file /home/qil/hg2~ dpwr             30
/QL-II-297_diaster~ dof               0
        eomer1.fid  dm              nnn
   ACQUISITION      dmm               c
sfrq        299.818  dmf             200
tn               H1      PROCESSING
at            1.995  wtfile
np            17984  proc             ft
sw           4506.5  fn         not used
fb         not used
bs               16  werr
tpwr             56  wexp
pw              7.0  wbs
d1            5.000  wnt
tof               0
nt              256
ct                0
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp           -765.7
wp           4506.3
vs              142
sc                0
wc              250
hzmm          18.03
is           500.00
rfl           765.9
rfp               0
th               10
ins         100.000
ai  cdc  ph
```



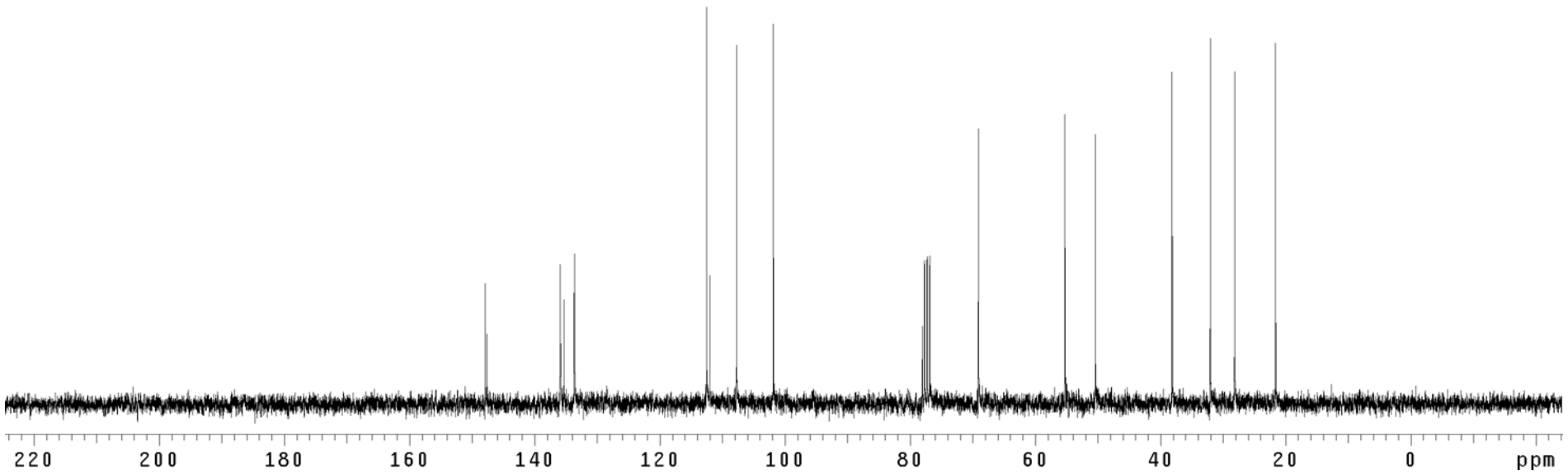
9



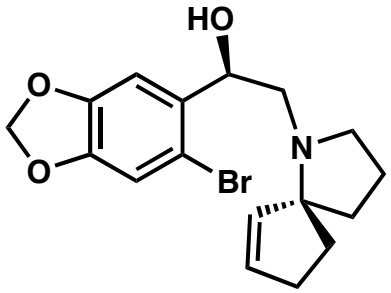
```
QL-II-297 diastereomer 1 on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date    Mar 17 2006  dfrq       299.818
solvent      CDCl3   dn               H1
file /home/qil/hg2~  dpwr             35
/QL-II-297diastere~  dof               0
     omer1_C13.fid   dm              yyy
   ACQUISITION       dmm               w
sfrq         75.396  dmf            7300
tn              C13       PROCESSING
at            1.815  lb             1.00
np            68106  wtfile
sw          18761.7  proc             ft
fb            10400  fn         not used
bs                8
tpwr             62  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct                0
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs               71
sc                0
wc              250
hzmm           1.97
is           500.00
rfl          1827.5
rfp               0
th               20
ins         100.000
ai  no  ph
```



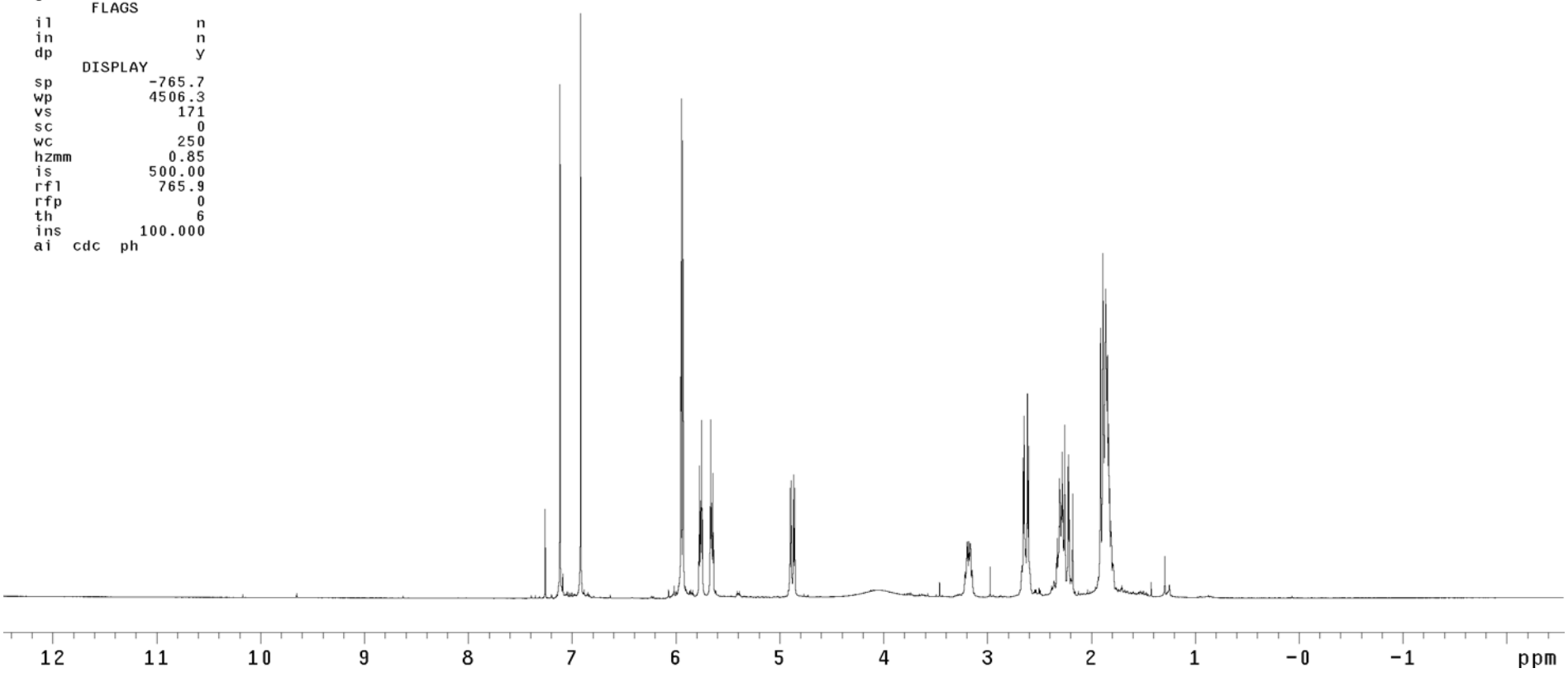
9



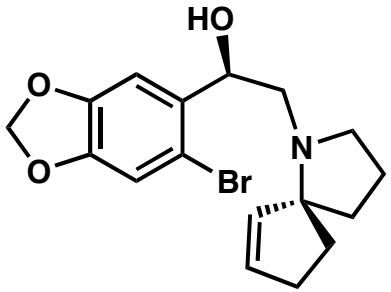
```
QL-II-297 diastereomer 2 the lower spot
after column on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date    Mar 17 2006  dfrq        299.818
solvent      CDCl3  dn               H1
file /home/qil/hg2~ dpwr             30
/QL-II-297diastere~ dof               0
         omer2.fid  dm              nnn
   ACQUISITION      dmm               c
sfrq       299.818  dmf             200
tn              H1      PROCESSING
at           1.995  wtfile
np           17984  proc             ft
sw          4506.5  fn         not used
fb        not used
bs               4  werr
tpwr            56  wexp
pw             7.0  wbs
d1           5.000  wnt
tof              0
nt             256
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs             171
sc               0
wc             250
hzmm          0.85
is          500.00
rfl          765.9
rfp              0
th               6
ins        100.000
ai  cdc  ph
```



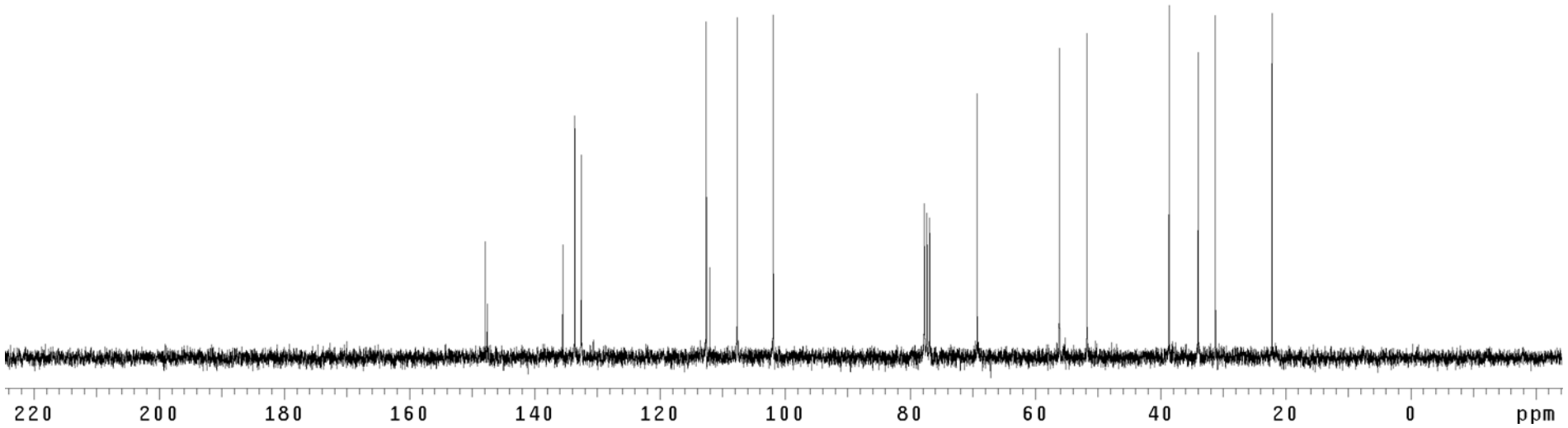
10



```
QL-II-297 diastereomer 2 the lower spot
after column on hg2

exp1  std13c

      SAMPLE            DEC. & VT
date    Mar 17 2006  dfrq       299.818
solvent      CDCl3   dn              H1
file /home/qil/hg2~  dpwr            35
/QL-II-297diastere~  dof              0
     omer2_C13.fid   dm             yyy
   ACQUISITION       dmm              w
sfrq         75.396  dmf           7300
tn              C13       PROCESSING
at            1.815  lb            1.00
np            68106  wtfile
sw          18761.7  proc            ft
fb            10400  fn        not used
bs                8
tpwr             62  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct                0
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs              116
sc                0
wc              250
hzmm           1.13
is           500.00
rfl          1827.5
rfp               0
th               20
ins         100.000
ai  no  ph
```



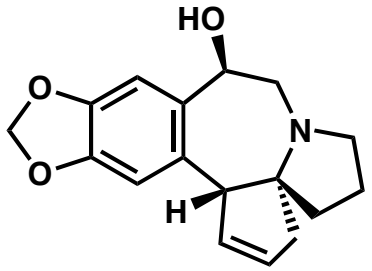
10



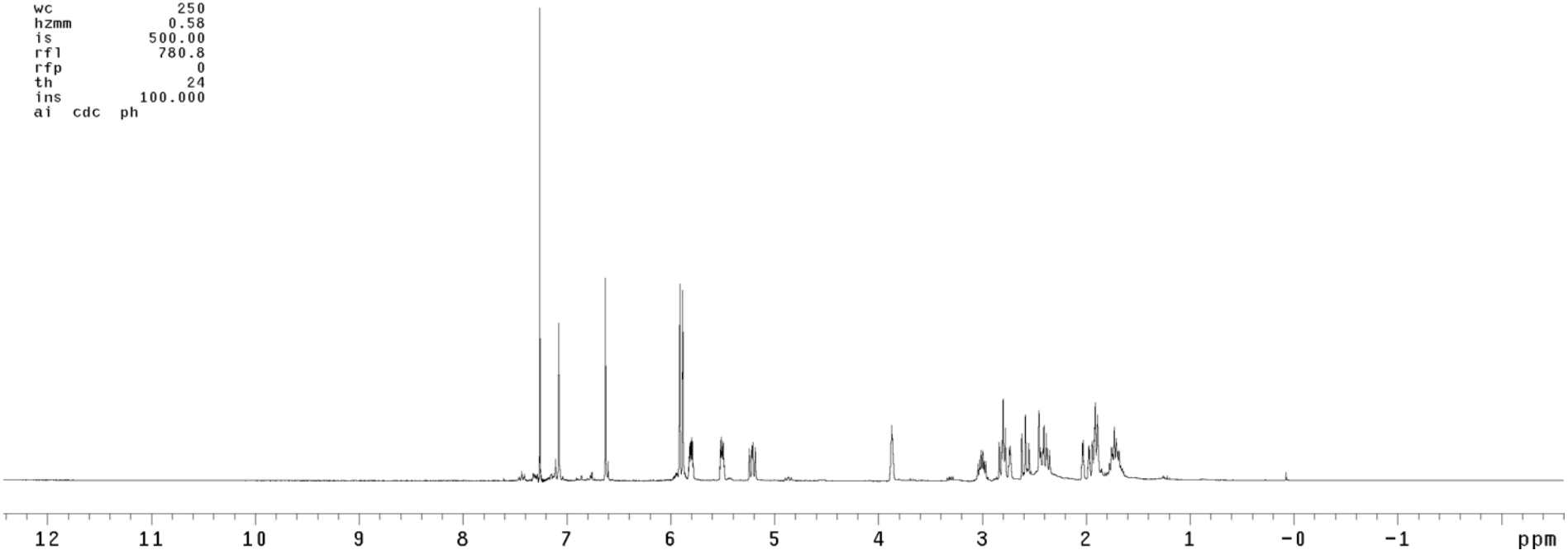
STANDARD 1H OBSERVE

exp1 std1h

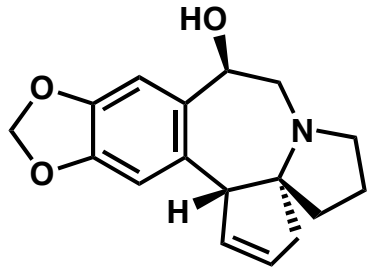
SAMPLE		DEC. & VT	
date	Aug 15 2006	dfrq	299.869
solvent	CDCl3	dn	H1
file	/home/qil/hg1~	dpwr	30
	/QL-IV-055.fid	dof	0
ACQUISITION		dm	nnn
sfrq	299.869	dmm	c
tn	H1	dmf	200
at	1.995	PROCESSING	
np	17984	wtfile	
sw	4506.5	proc	ft
fb	not used	fn	not used
bs	4		
tpwr	55	werr	
pw	7.0	wexp	
d1	1.000	wbs	
tof	0	wnt	
nt	256		
ct	64		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-780.5		
wp	4506.3		
vs	92		
sc	0		
wc	250		
hzmm	0.58		
is	500.00		
rfl	780.8		
rfp	0		
th	24		
ins	100.000		
ai cdc ph			



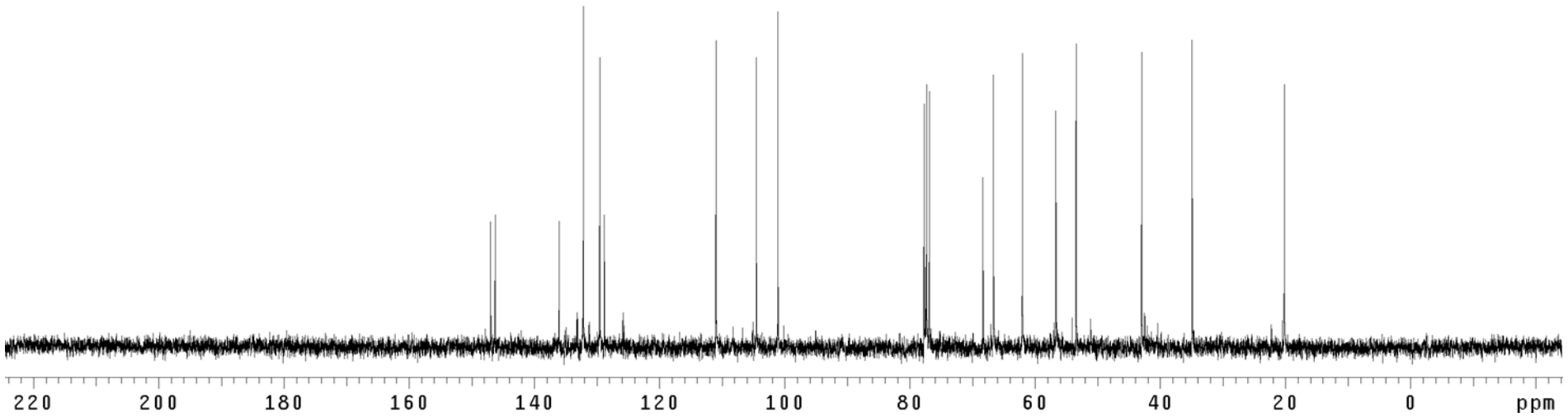
15



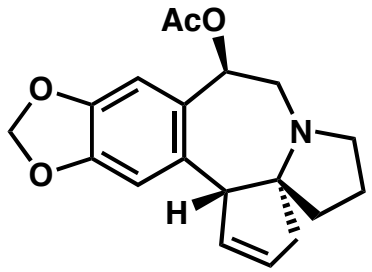
```
13C OBSERVE

exp1  std13c

      SAMPLE            DEC. & VT
date    Aug 15 2006  dfrq        299.869
solvent      CDCl3    dn              H1
file /home/qil/hg1~   dpwr            36
/QL-IV-055_C13.fid    dof              0
   ACQUISITION        dm             yyy
sfrq         75.409   dmm              w
tn             C13    dmf           6500
at           1.815        PROCESSING
np           68106    lb            1.00
sw         18761.7    wtfile
fb           10400    proc             ft
bs              16    fn         not used
tpwr            59
pw             8.7    werr
d1               0    wexp
tof              0    wbs
nt            1024    wnt
ct             224
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp         -1829.7
wp         18761.4
vs             163
sc               0
wc             250
hzmm         19.13
is          500.00
rfl         1830.0
rfp              0
th              22
ins         100.000
ai  no  ph
```



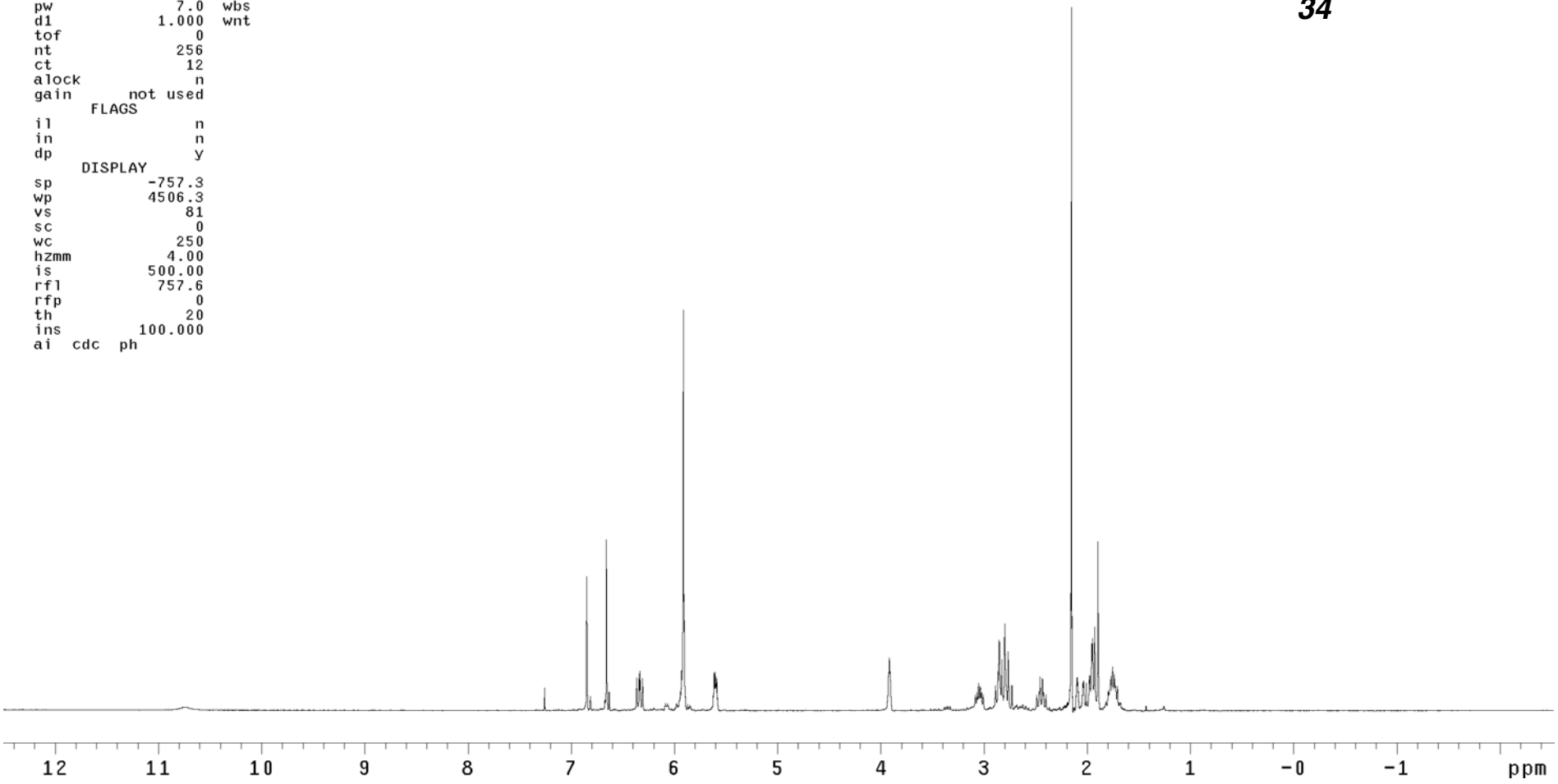
15



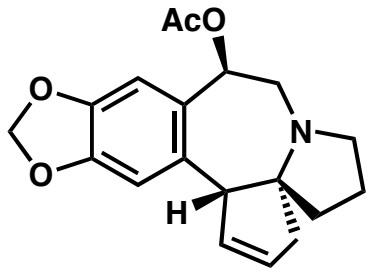
```
QL-III-049 spot1 after column on hg1

exp1  std1h

      SAMPLE            DEC. & VT
date   Mar 27 2006  dfrq       299.868
solvent      CDCl3  dn              H1
file /home/ql/hg1~ dpwr             30
/QL-III-049spot1.f~ dof              0
                id  dm             nnn
   ACQUISITION      dmm              c
sfrq       299.868  dmf            200
tn              H1      PROCESSING
at           1.995  wtfile
np           17984  proc            ft
sw          4506.5  fn        not used
fb        not used
bs               4  werr
tpwr            55  wexp
pw             7.0  wbs
d1           1.000  wnt
tof              0
nt             256
ct              12
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -757.3
wp          4506.3
vs              81
sc               0
wc             250
hzmm          4.00
is          500.00
rfl          757.6
rfp              0
th              20
ins         100.000
ai  cdc  ph
```



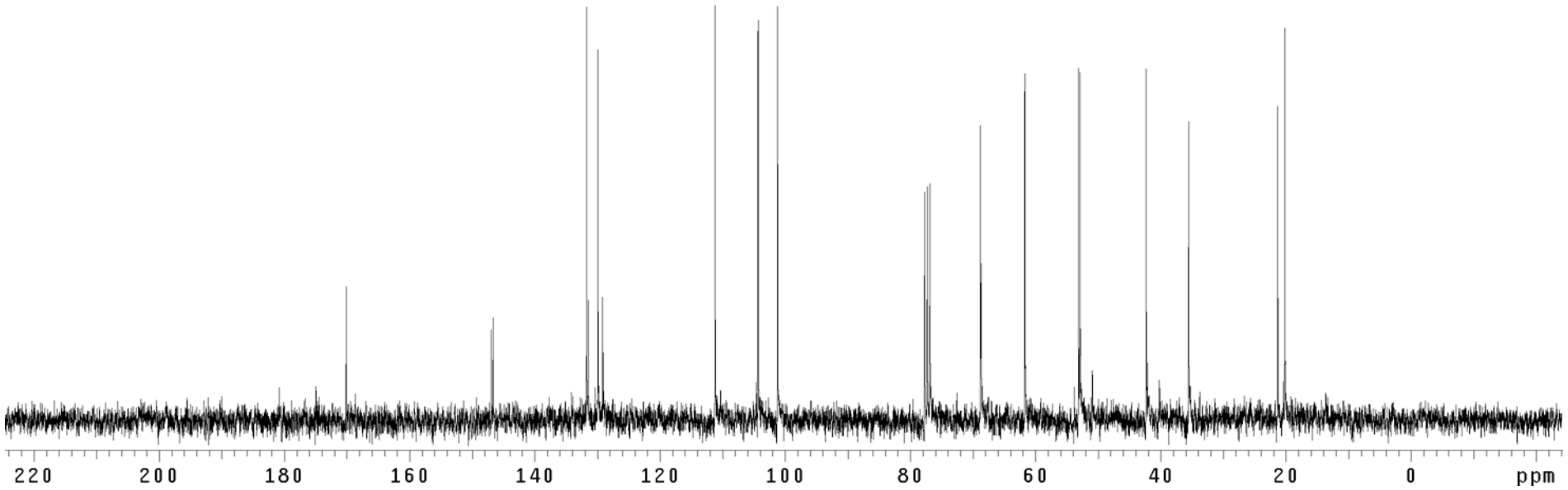
34



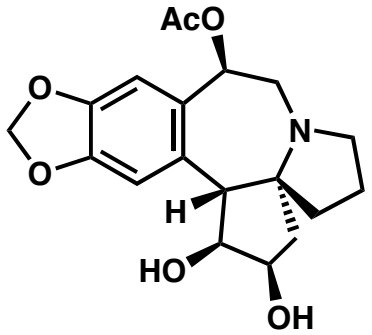
```
QL-III-049 spot1 after column on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Mar 27 2006  dfrq       299.868
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            36
/QL-III-049spot1_C~ dof              0
           13.fid  dm             yyy
   ACQUISITION      dmm              w
sfrq        75.409  dmf           6500
tn             C13       PROCESSING
at           1.815  lb            1.50
np           68106  wtfile
sw         18761.7  proc            ft
fb           10400  fn        not used
bs               8
tpwr            59  werr
pw             8.7  wexp
d1               0  wbs
tof              0  wnt
nt            1024
ct             144
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp         -1823.9
wp         18761.4
vs             398
sc               0
wc             250
hzmm         75.05
is          500.00
rfl         1824.2
rfp              0
th              12
ins         100.000
ai  no  ph
```



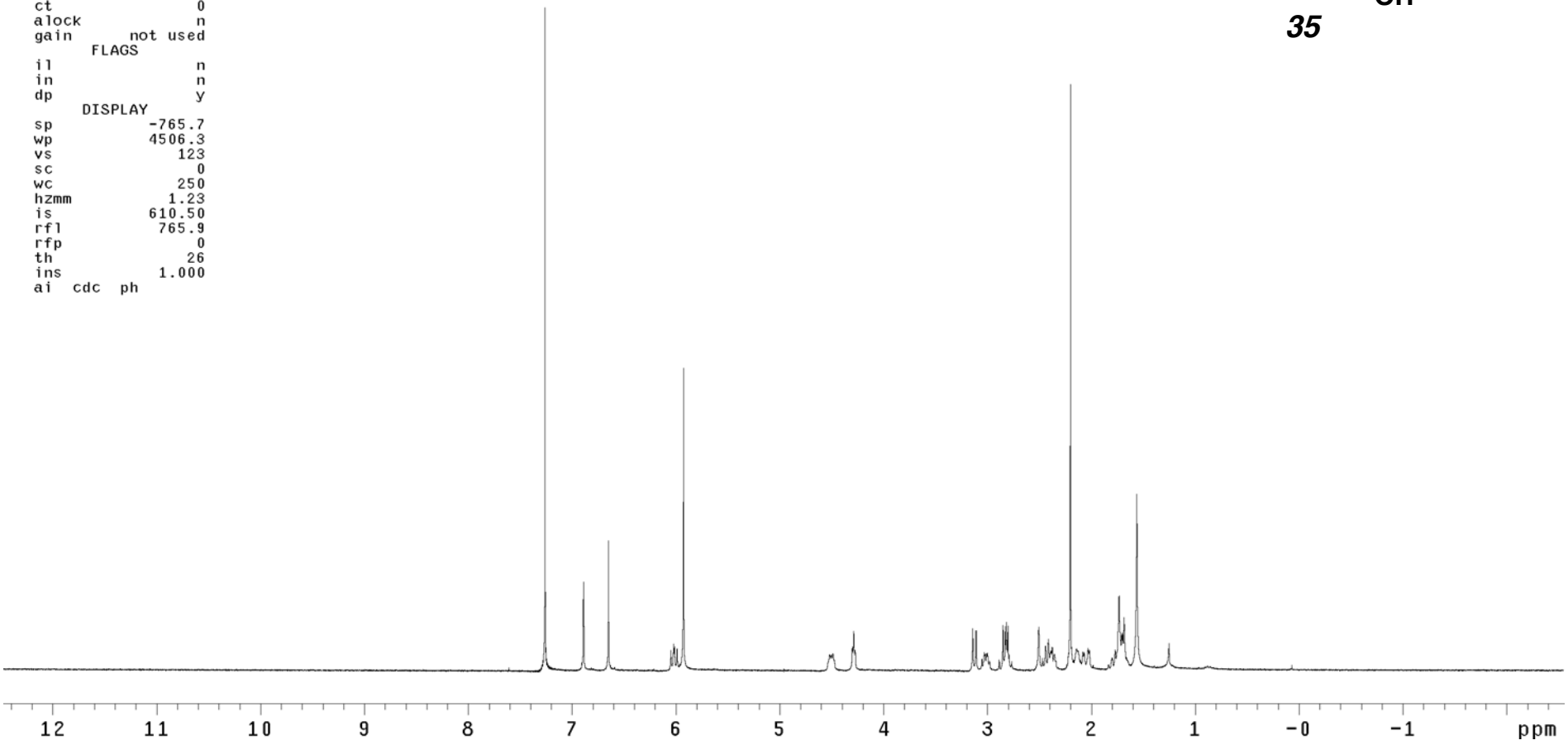
34



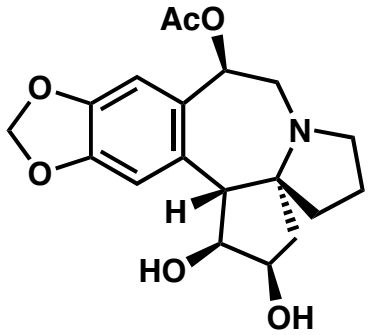
```
QL-IV-105 after column on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug 20 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
    /QL-IV-105.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at            1.995     PROCESSING
np            17984  wtfile
sw           4506.5  proc            ft
fb        not used  fn        not used
bs               4
tpwr            56  werr
pw             7.0  wexp
d1           1.000  wbs
tof              0  wnt
nt             256
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs             123
sc               0
wc             250
hzmm          1.23
is          610.50
rfl           765.9
rfp              0
th              26
ins           1.000
ai  cdc  ph
```



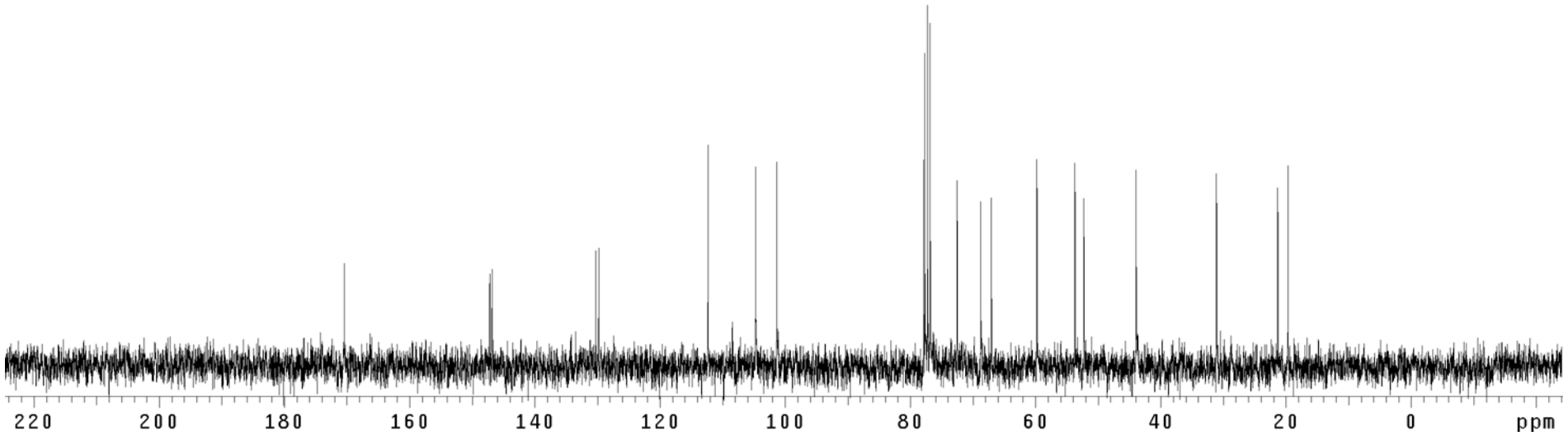
35



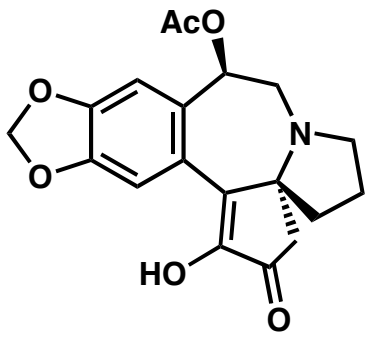
```
QL-III-095 after column on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Apr 14 2006  dfrq       299.868
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            36
/QL-III-095_C13.fi~ dof              0
                 d  dm             yyy
   ACQUISITION      dmm              w
sfrq         75.409  dmf           6500
tn              C13       PROCESSING
at            1.815  lb            1.60
np            68106  wtfile
sw          18761.7  proc            ft
fb            10400  fn        not used
bs                8
tpwr             59  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct                0
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1823.9
wp          18761.4
vs              304
sc                0
wc              250
hzmm          23.57
is           500.00
rfl          1824.2
rfp               0
th               23
ins          100.000
ai  no  ph
```



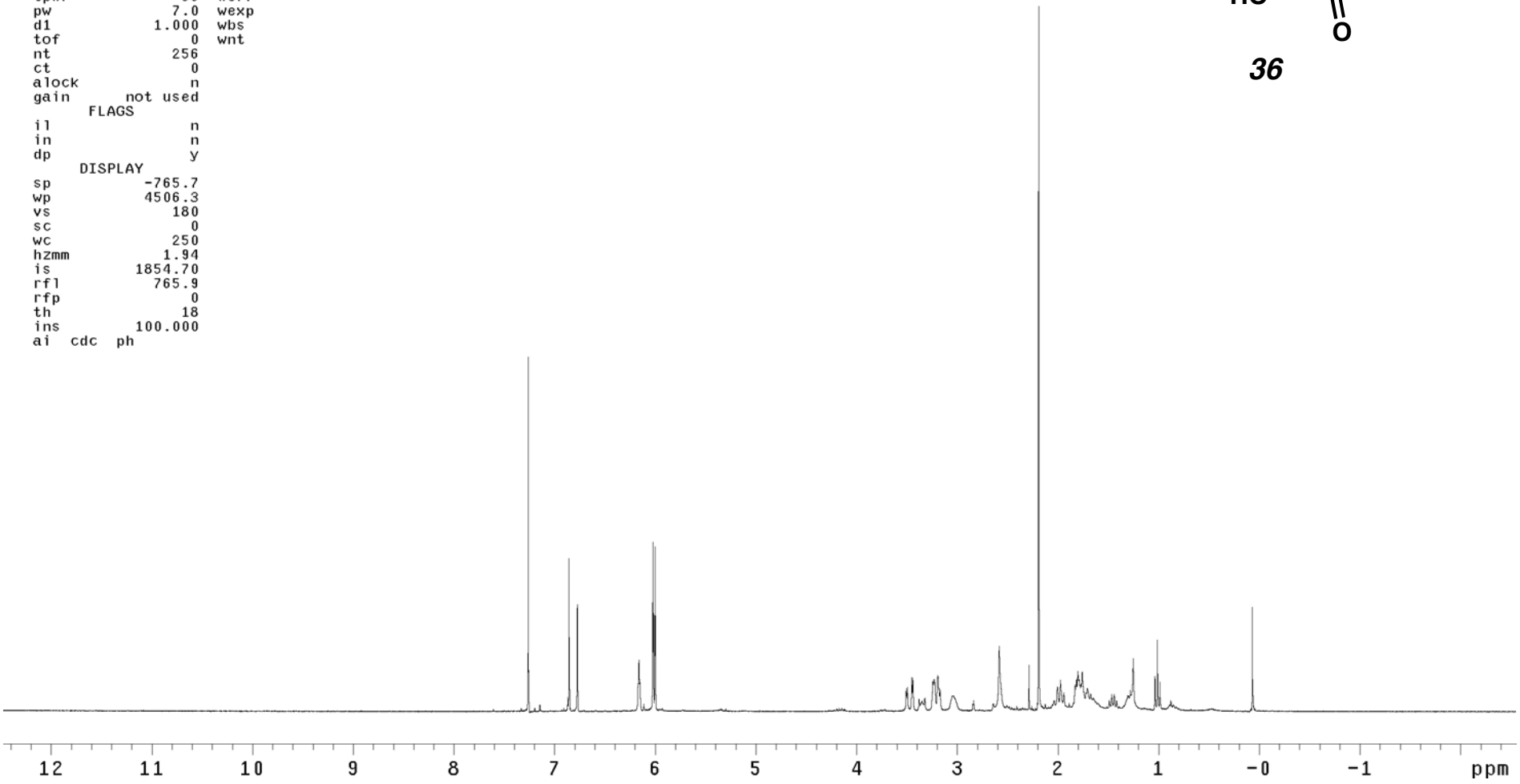
35



```
QL-IV-069 on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug  4 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
    /QL-IV-069.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb       not used  fn       not used
bs              4
tpwr           56  werr
pw            7.0  wexp
d1          1.000  wbs
tof             0  wnt
nt            256
ct              0
alock           n
gain     not used
      FLAGS
il              n
in              n
dp              y
     DISPLAY
sp         -765.7
wp         4506.3
vs            180
sc              0
wc            250
hzmm         1.94
is        1854.70
rfl         765.9
rfp             0
th             18
ins       100.000
ai  cdc  ph
```



36



13C OBSERVE

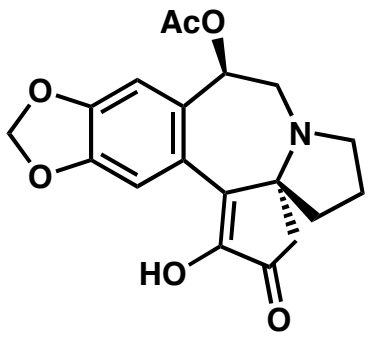
exp1 std13c

SAMPLE		DEC. & VT	
date	Aug 3 2006	dfrq	299.869
solvent	CDCl3	dn	H1
file	/home/qil/hg1~/QL-IV-069_C13.fid	dpwr	36
		dof	0
ACQUISITION		dm	yyy
sfrq	75.409	dmm	w
tn	C13	dmf	6500
at	1.815	PROCESSING	
np	68106	lb	1.00
sw	18761.7	wtfile	
fb	10400	proc	ft
bs	16	fn	not used
tpwr	59		
pw	8.7	werr	
d1	0	wexp	
tof	0	wbs	
nt	205600	wnt	
ct	0		
alock	n		
gain	not used		

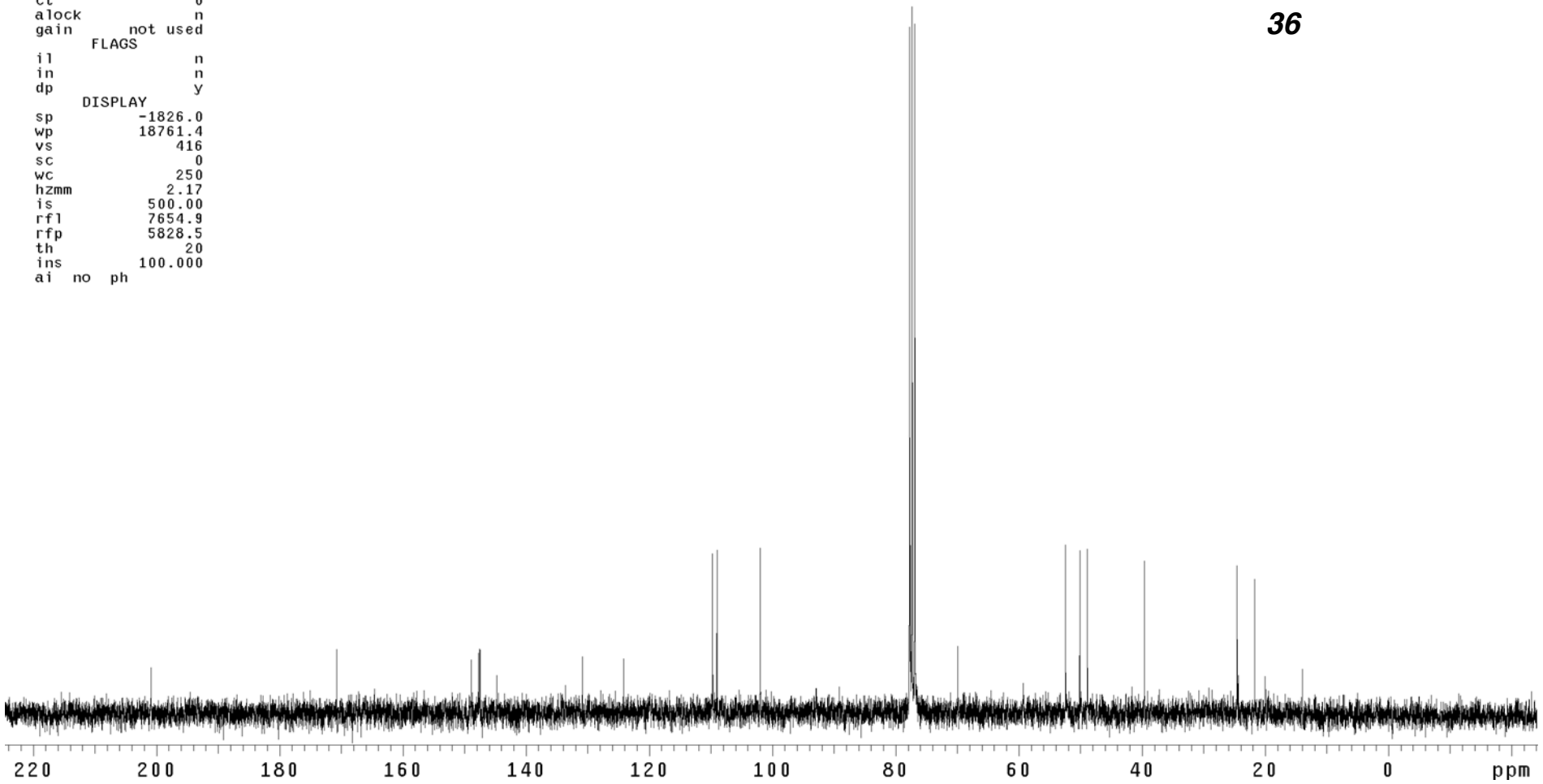
FLAGS	
il	n
in	n
dp	y

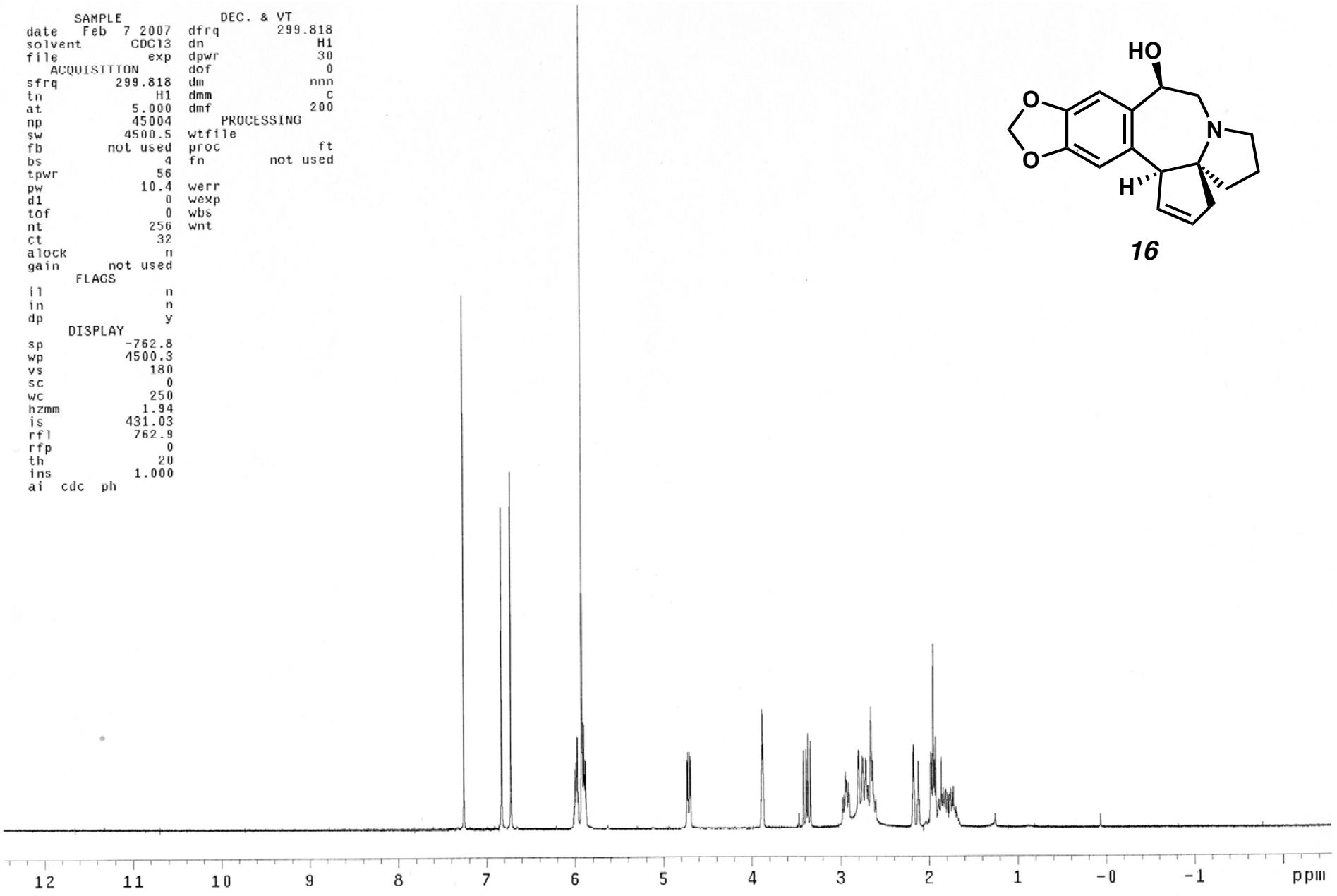
DISPLAY	
sp	-1826.0
wp	18761.4
vs	416
sc	0
wc	250
hzmm	2.17
is	500.00
rfl	7654.9
rfp	5828.5
th	20
ins	100.000

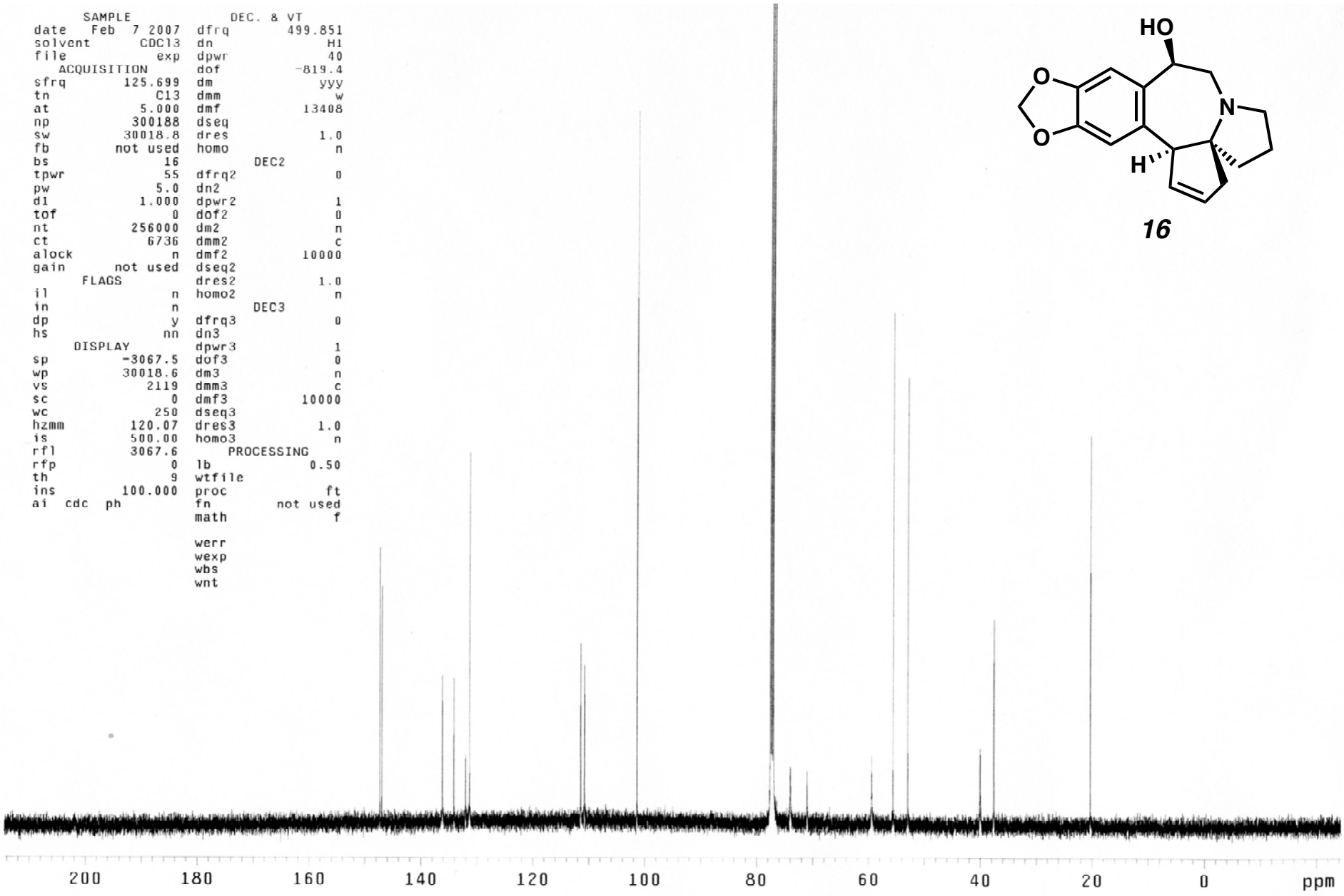
ai no ph



36



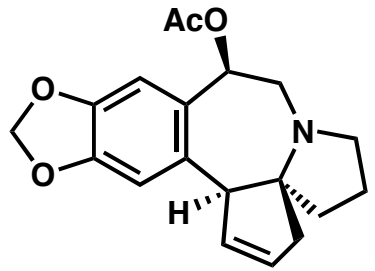

SAMPLE
date Feb 7 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 299.818
tn H1
at 5.000
np 45004
sw 4500.5
fb not used
bs 4
tpwr 56
pw 10.4
d1 0
tof 0
nt 256
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -762.8
wp 4500.3
vs 180
sc 0
wc 250
hzmm 1.94
is 431.03
rfl 762.9
rfp 0
th 20
ins 1.000
ai cdc ph
DEC. & VT
dfrq 299.818
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
HO
O
O
N
H
16
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


SAMPLE
date Feb 7 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.699
tn C13
at 5.000
np 300188
sw 30018.8
fb not used
bs 16
tpwr 55
pw 5.0
d1 1.000
tof 0
nt 256000
ct 6736
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -3067.5
wp 30018.6
vs 2119
sc 0
wc 250
hzmm 120.07
is 500.00
rfl 3067.6
rfp 0
th 9
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.851
dn H1
dpwr 40
dof -819.4
dm yyy
dmm w
dmf 13408
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
HO
O
O
N
H
16
200
180
160
140
120
100
80
60
40
20
0
ppm

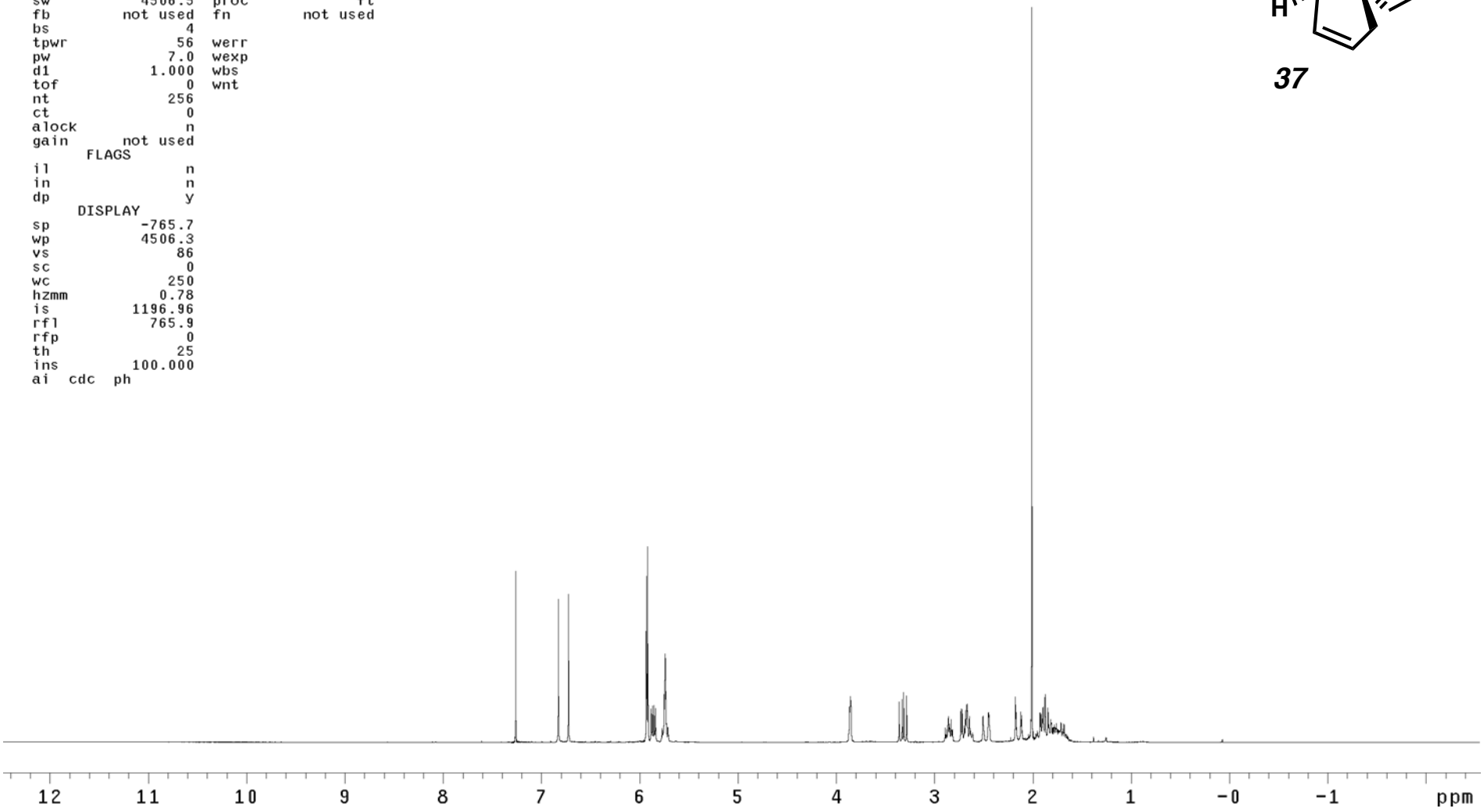
QL-IV-085 after column on hg2

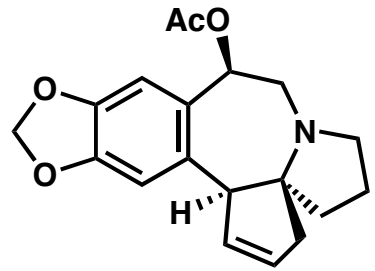
exp1 std1h

SAMPLE		DEC. & VT	
date	Aug 6 2006	dfrq	299.818
solvent	CDCl3	dn	H1
file	/home/qil/hg2~ /QL-IV-085.fid	dpwr	30
		dof	0
ACQUISITION		dm	nnn
sfrq	299.818	dmm	c
tn	H1	dmf	200
at	1.995	PROCESSING	
np	17984	wtfile	
sw	4506.5	proc	ft
fb	not used	fn	not used
bs	4		
tpwr	56	werr	
pw	7.0	wexp	
d1	1.000	wbs	
tof	0	wnt	
nt	256		
ct	0		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-765.7		
wp	4506.3		
vs	86		
sc	0		
wc	250		
hzmm	0.78		
is	1196.96		
rfl	765.9		
rfp	0		
th	25		
ins	100.000		
ai cdc ph			



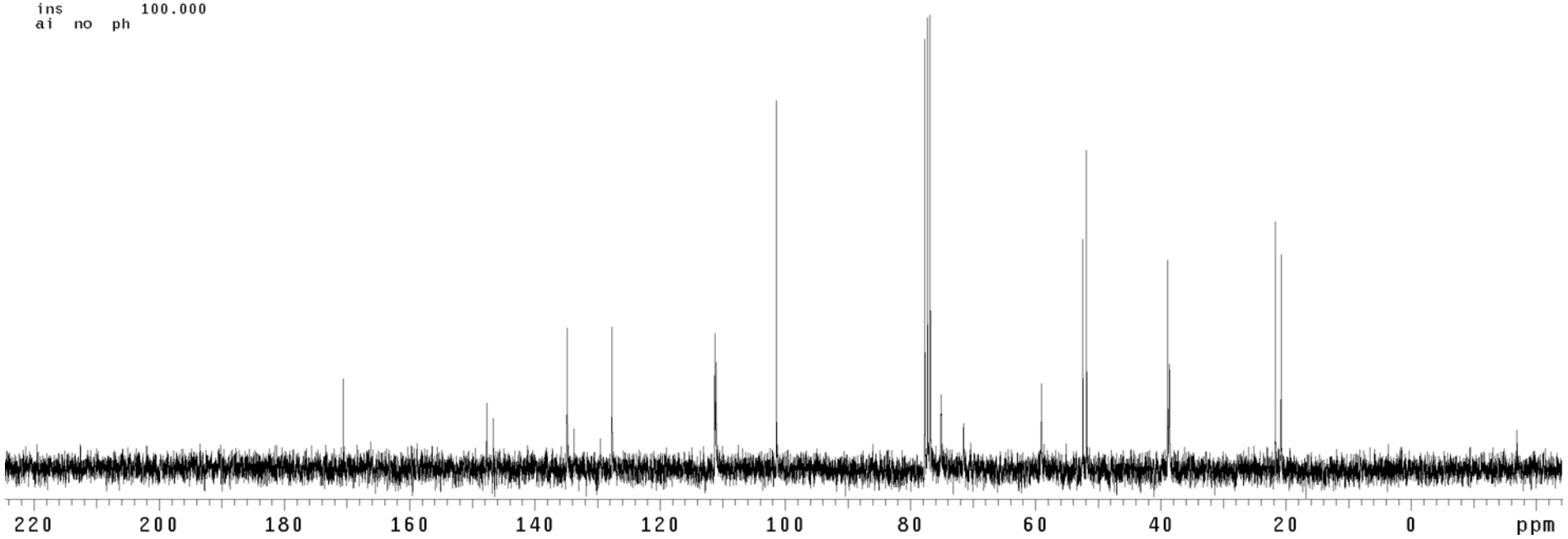
37



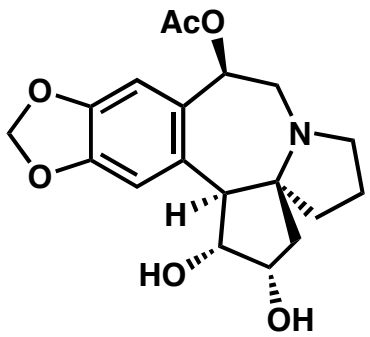


37

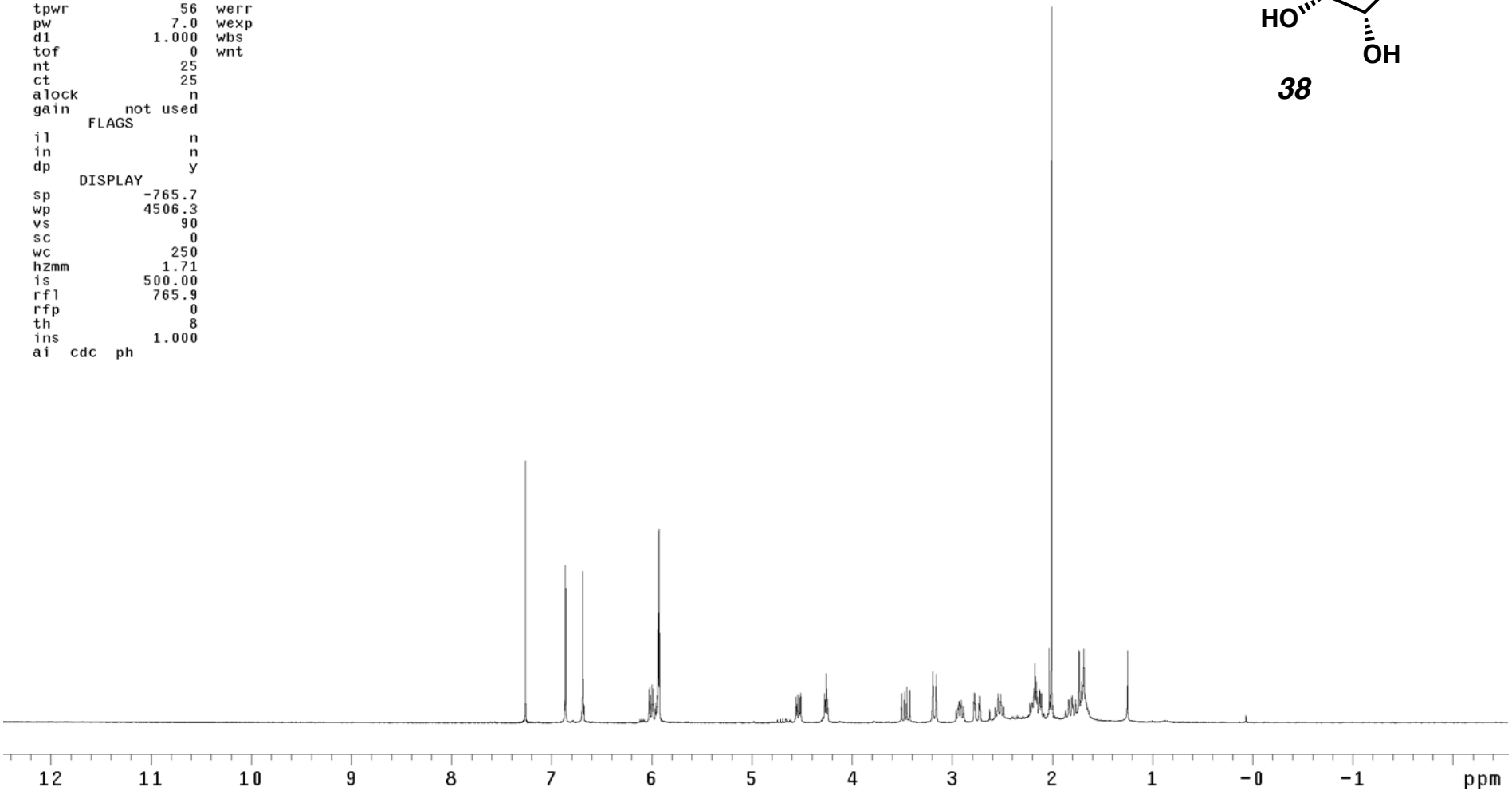
```
QL-III-049 spot2 after column on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Mar 27 2006  dfrq       299.868
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            36
/QL-III-049spot2_C~ dof              0
           13.fid  dm             yyy
   ACQUISITION      dmm              w
sfrq        75.409  dmf           6500
tn             C13       PROCESSING
at           1.815  lb            1.00
np           68106  wtfile
sw         18761.7  proc            ft
fb           10400  fn        not used
bs               8
tpwr            59  werr
pw             8.7  wexp
d1               0  wbs
tof              0  wnt
nt            1024
ct             624
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp         -1823.9
wp         18761.4
vs             221
sc               0
wc             250
hzmm          2.06
is          500.00
rfl         1824.2
rfp              0
th              24
ins         100.000
ai  no  ph
```



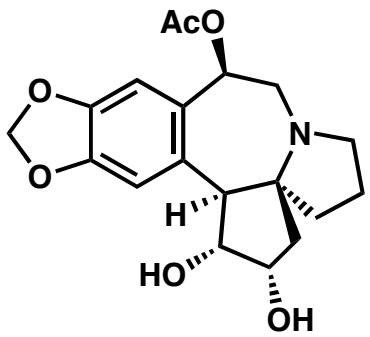
```
QL-IV-089 after column on hg2

exp1  std1h

      SAMPLE           DEC. & VT
date   Aug  8 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
    /QL-IV-089.fid  dof              0
   ACQUISITION      dm             nnn
sfrq       299.818  dmm              c
tn              H1  dmf            200
at           1.995      PROCESSING
np           17984  wtfile
sw          4506.5  proc            ft
fb        not used  fn        not used
bs                4
tpwr             56  werr
pw              7.0  wexp
d1            1.000  wbs
tof               0  wnt
nt               25
ct               25
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -765.7
wp          4506.3
vs              90
sc               0
wc             250
hzmm          1.71
is          500.00
rfl           765.9
rfp               0
th                8
ins           1.000
ai  cdc  ph
```



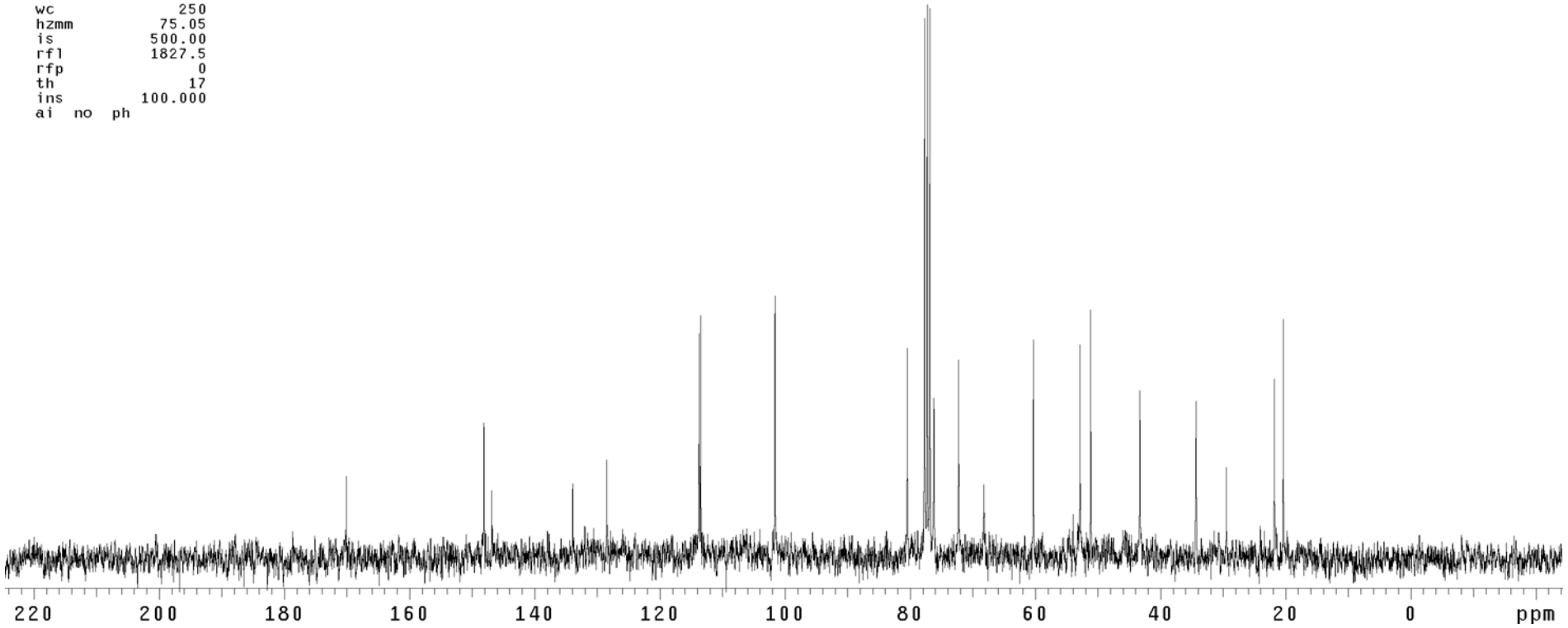
38

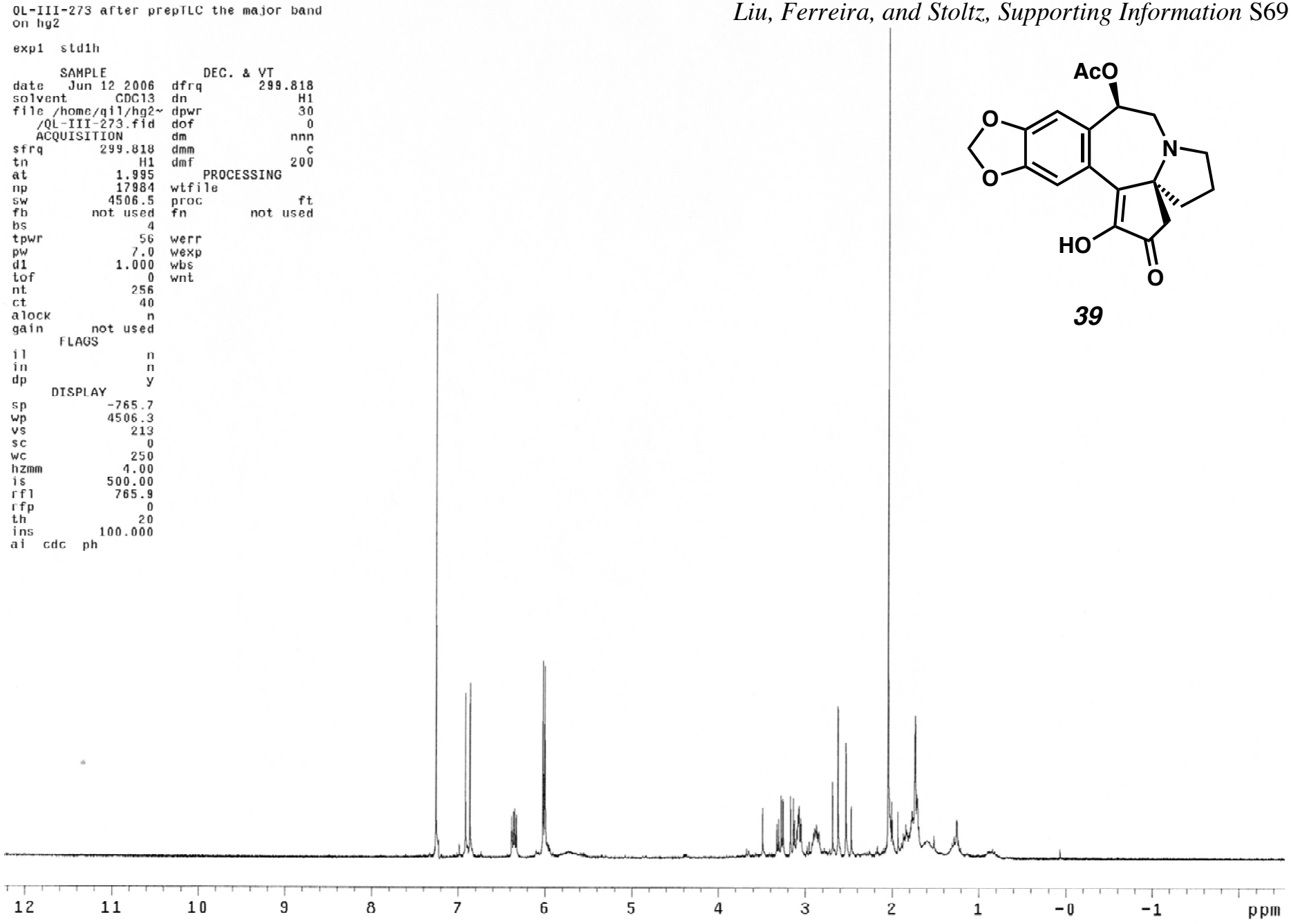


```
QL-III-269 after extraction on hg2

exp1  std13c

      SAMPLE           DEC. & VT
date   Jun 11 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            35
/QL-III-269_C13cru~ dof              0
              de.fid dm             yyy
   ACQUISITION       dmm              w
sfrq         75.396  dmf           7300
tn              C13       PROCESSING
at            1.815  lb            3.00
np            68106  wtfile
sw          18761.7  proc             ft
fb            10400  fn        not used
bs                8
tpwr             62  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct              632
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1827.2
wp          18761.4
vs              430
sc                0
wc              250
hzmm          75.05
is           500.00
rfl          1827.5
rfp               0
th               17
ins          100.000
ai  no  ph
```



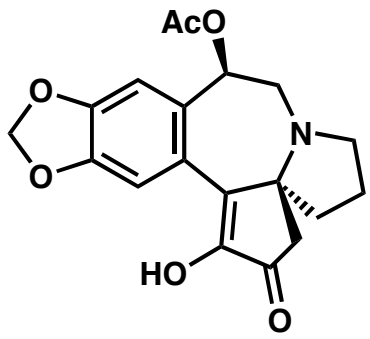
38



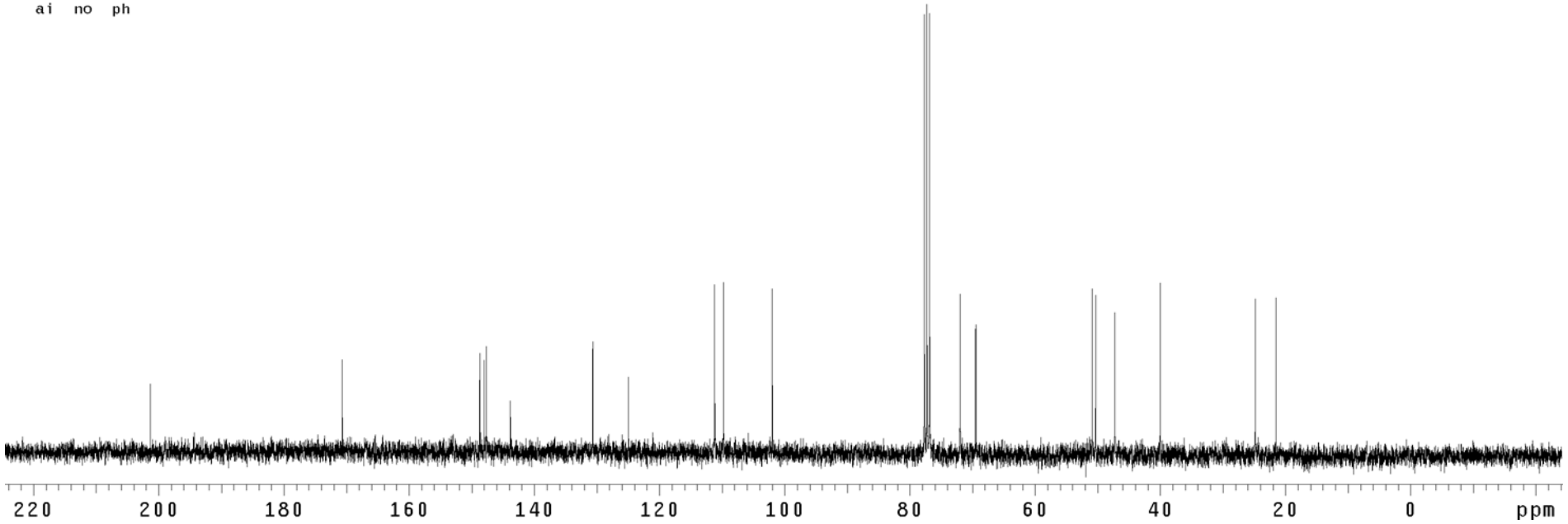

QL-III-273 after prepTLC the major band
on hg2
exp1 std1h
SAMPLE
date Jun 12 2006
solvent CDCl3
file /home/qil/hg2~
/QL-III-273.fid
ACQUISITION
sfrq 299.818
tn H1
at 1.995
np 17984
sw 4506.5
fb not used
bs 4
tpwr 56
pw 7.0
d1 1.000
tof 0
nt 256
ct 40
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -765.7
wp 4506.3
vs 213
sc 0
wc 250
hzmm 4.00
is 500.00
rfl 765.9
rfp 0
th 20
ins 100.000
ai cdc ph
DEC. & VT
dfrq 299.818
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
AcO
O
O
N
HO
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

39

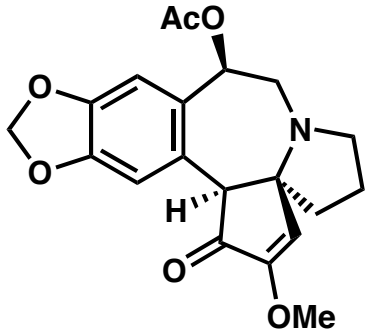
```
QL-II-271 on hg1

exp1  std13c

      SAMPLE           DEC. & VT
date   Jun 12 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr            36
/QL-III-273_C13.fi~ dof              0
                  d  dm             yyy
   ACQUISITION       dmm              w
sfrq         75.409  dmf           6500
tn              C13       PROCESSING
at            1.815  lb            1.00
np            68106  wtfile
sw          18761.7  proc            ft
fb            10400  fn        not used
bs               16
tpwr             59  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt             1024
ct                0
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1829.7
wp          18761.4
vs             1043
sc                0
wc              250
hzmm          31.85
is           500.00
rfl          1830.0
rfp               0
th               19
ins          100.000
ai  no  ph
```



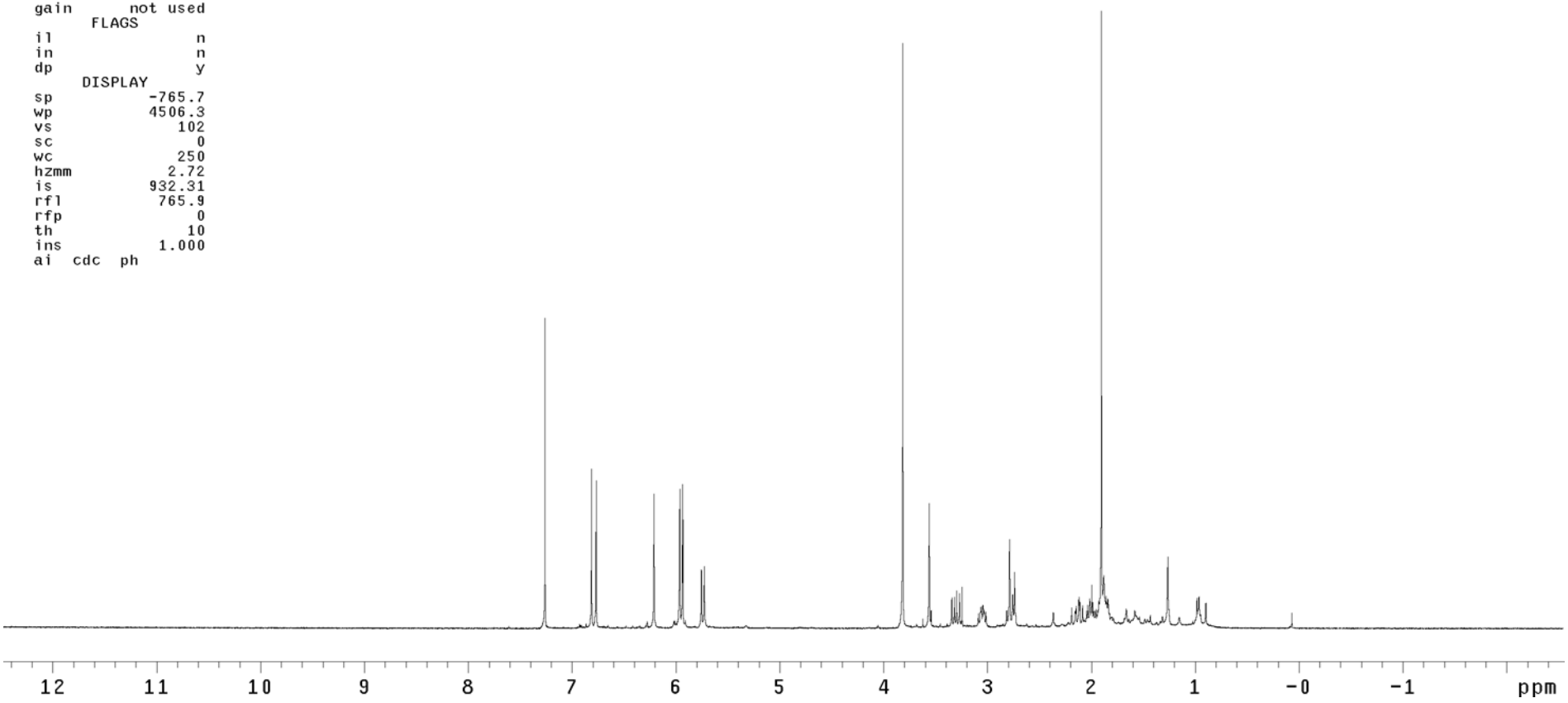
39



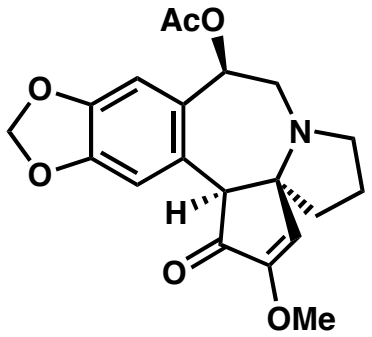
```
QL-IV-109 band2 after prepTLC on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date   Aug 21 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/qil/hg2~ dpwr            30
/QL-IV-109band2.fi~ dof              0
                  d  dm            nnn
   ACQUISITION       dmm             c
sfrq       299.818  dmf           200
tn              H1       PROCESSING
at           1.995  wtfile
np           17984  proc            ft
sw          4506.5  fn        not used
fb        not used
bs               4  werr
tpwr            56  wexp
pw             7.0  wbs
d1           1.000  wnt
tof              0
nt             256
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs             102
sc               0
wc             250
hzmm          2.72
is          932.31
rfl           765.9
rfp              0
th              10
ins           1.000
ai  cdc  ph
```



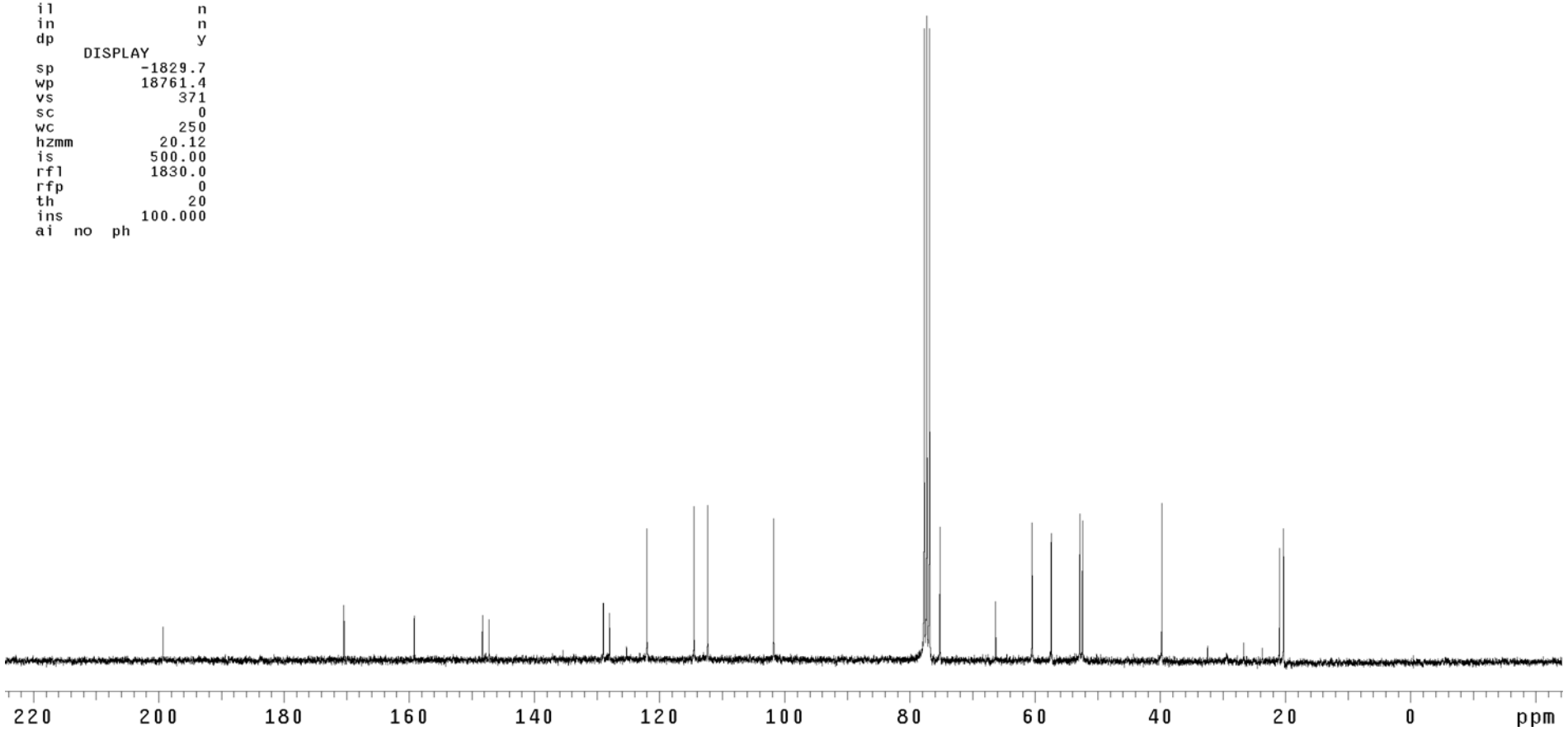
40



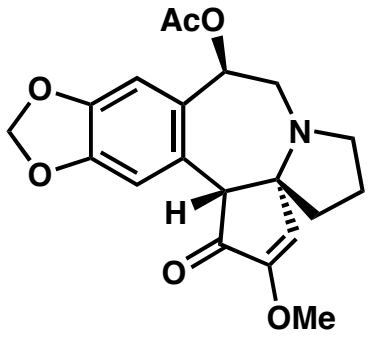
```
QL-III-275 major band after 2nd prepTLC
on hg1

exp1  std13c

      SAMPLE            DEC. & VT
date   Jun 13 2006  dfrq       299.869
solvent      CDCl3  dn              H1
file /home/qil/hg1~ dpwr             36
/QL-III-275_C13.fi~ dof               0
                  d  dm              yyy
   ACQUISITION       dmm              w
sfrq         75.409  dmf           6500
tn              C13      PROCESSING
at            1.815  lb            1.00
np            68106  wtfile
sw          18761.7  proc            ft
fb            10400  fn        not used
bs               16
tpwr             59  werr
pw              8.7  wexp
d1                0  wbs
tof               0  wnt
nt           256000
ct                0
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1829.7
wp          18761.4
vs              371
sc                0
wc              250
hzmm          20.12
is           500.00
rfl          1830.0
rfp               0
th               20
ins         100.000
ai  no  ph
```



40



```
QL-IV-109 band1 after prepTLC on hg2

exp1  std1h

      SAMPLE            DEC. & VT
date   Aug 22 2006  dfrq       299.818
solvent      CDCl3  dn              H1
file /home/ql/hg2~ dpwr            30
/QL-IV-109band1.fi~ dof             0
                  d  dm            nnn
   ACQUISITION       dmm             c
sfrq       299.818  dmf           200
tn              H1       PROCESSING
at           1.995  wtfile
np           17984  proc            ft
sw          4506.5  fn        not used
fb        not used
bs               4  werr
tpwr            56  wexp
pw             7.0  wbs
d1           1.000  wnt
tof              0
nt             256
ct               0
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp          -765.7
wp          4506.3
vs             178
sc               0
wc             250
hzmm          2.32
is         5428.24
rfl           765.9
rfp              0
th              44
ins           1.000
ai  cdc  ph
```



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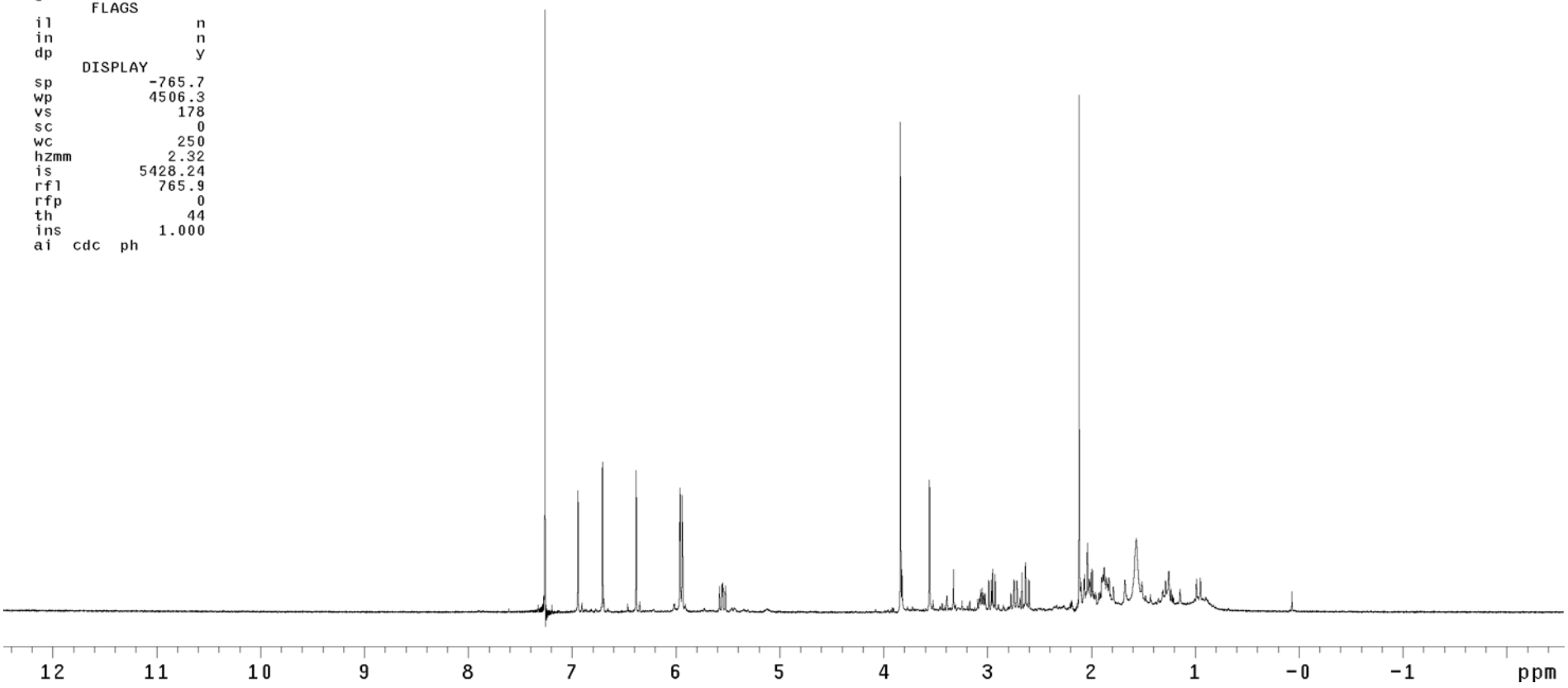


STANDARD CARBON PARAMETERS

exp1 s2pul

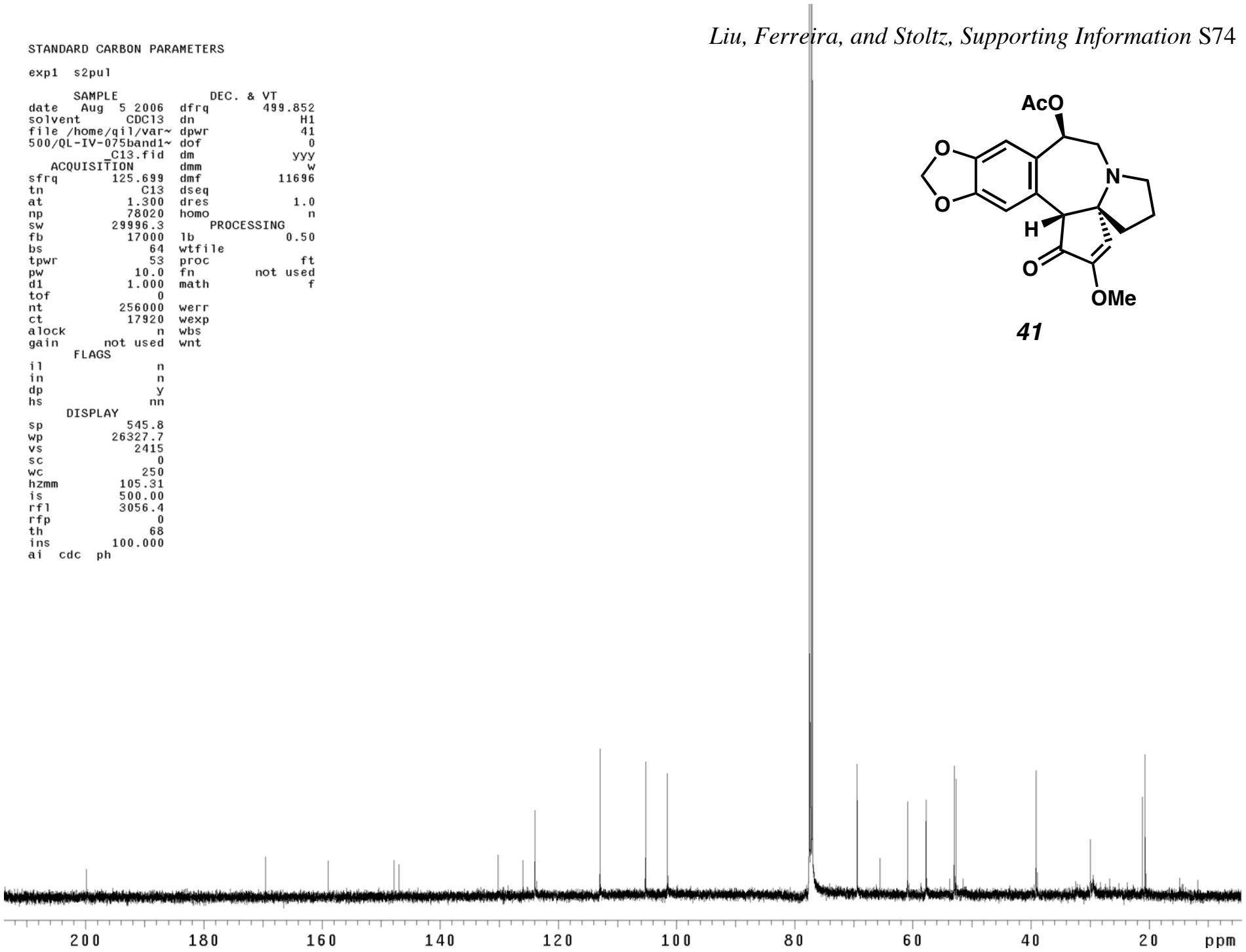
SAMPLE		DEC. & VT	
date	Aug 5 2006	dfrq	499.852
solvent	CDCl3	dn	H1
file	/home/qil/var~	dpwr	41
500/QL-IV-075band1~	_C13.fid	dof	0
		dm	yyy
ACQUISITION		dmm	w
sfrq	125.699	dmf	11696
tn	C13	dseq	
at	1.300	dres	1.0
np	78020	homo	n
sw	29996.3	PROCESSING	
fb	17000	lb	0.50
bs	64	wtfile	
tpwr	53	proc	ft
pw	10.0	fn	not used
d1	1.000	math	f
tof	0		
nt	256000	werr	
ct	17920	wexp	
alock	n	wbs	
gain	not used	wnt	

FLAGS	
il	n
in	n
dp	y
hs	nn

DISPLAY	
sp	545.8
wp	26327.7
vs	2415
sc	0
wc	250
hzmm	105.31
is	500.00
rfl	3056.4
rfp	0
th	68
ins	100.000

ai cdc ph

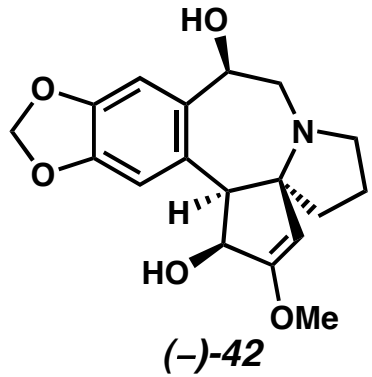
41



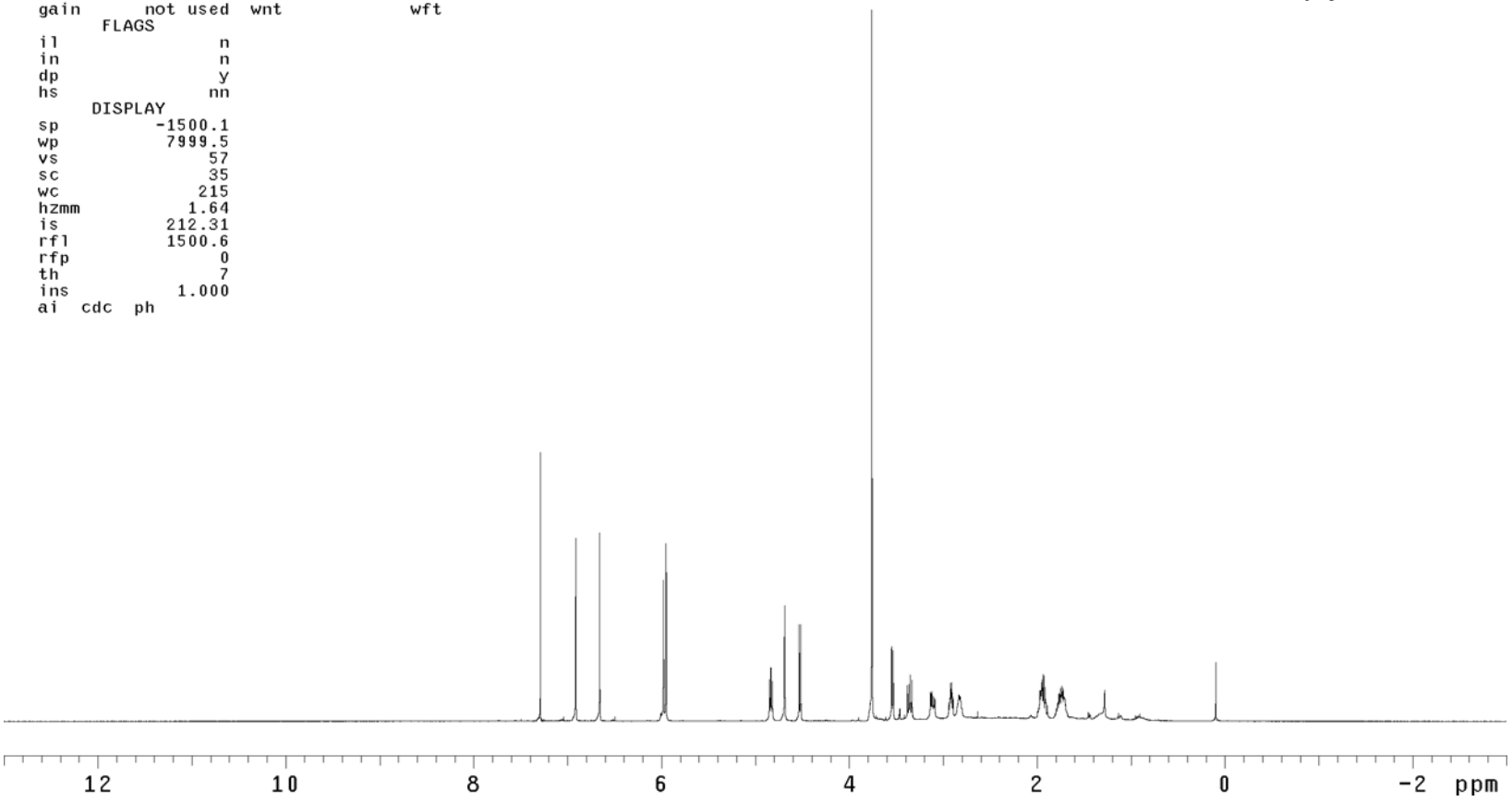
```
QL-IV-111 after prepTLC on I500

exp1  s2pul

      SAMPLE           DEC. & VT
date   Aug 22 2006  dfrq       499.851
solvent      CDCl3  dn              H1
file /home/ql/var~ dpwr            47
 500/QL-IV-111.fid  dof         -823.0
   ACQUISITION      dm             nnn
sfrq       499.852  dmm             c
tn              H1  dmf          9900
at           1.892  dseq
np           30272  dres          1.0
sw          8000.0  homo            n
fb            4000      PROCESSING
bs               4  wtfile
tpwr            57  proc           ft
pw             8.0  fn        not used
d1               0  math            f
tof              0
nt             256  werr
ct              88  wexp
alock            n  wbs
gain     not used   wnt            wft
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp         -1500.1
wp          7999.5
vs              57
sc              35
wc             215
hzmm          1.64
is          212.31
rfl         1500.6
rfp              0
th               7
ins          1.000
ai  cdc  ph
```



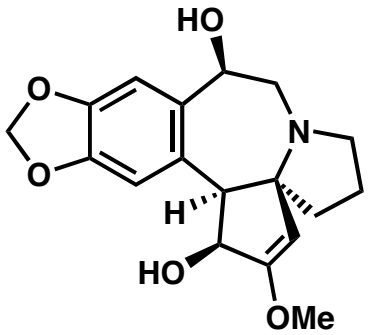
(–)-42



```
13C OBSERVE

exp1  std13c

      SAMPLE            DEC. & VT
date   Aug 23 2006  dfrq        299.869
solvent      CDCl3  dn               H1
file /home/qil/hg1~ dpwr             36
/QL-IV-111_C13.fid  dof               0
   ACQUISITION      dm              yyy
sfrq         75.409  dmm               w
tn              C13  dmf            6500
at            1.815      PROCESSING
np            68106  lb             1.00
sw          18761.7  wtfile
fb            10400  proc             ft
bs               16  fn         not used
tpwr             59
pw              8.7  werr
d1            1.000  wexp
tof               0  wbs
nt           256000  wnt
ct                0
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -1826.6
wp          18761.4
vs             2475
sc                0
wc              250
hzmm          75.05
is           500.00
rfl          7655.4
rfp          5828.5
th               18
ins         100.000
ai  no  ph
```



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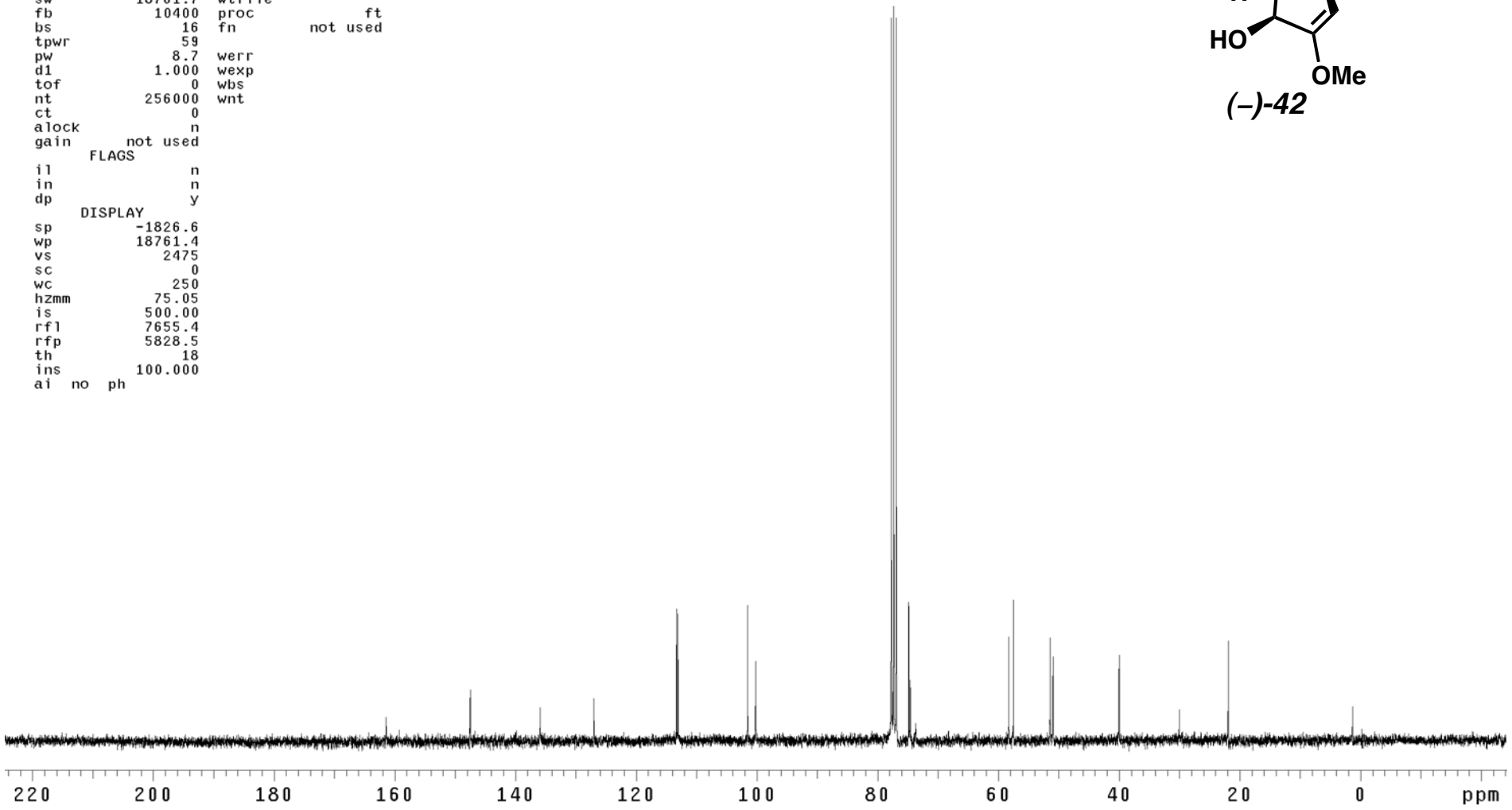
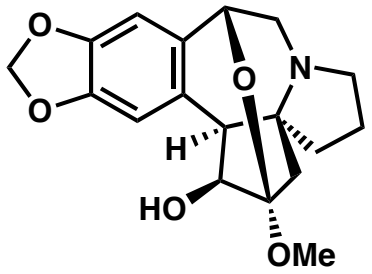


```
QL-IV-113 after FCC on I500

exp1  s2pul

      SAMPLE           DEC. & VT
date   Aug 29 2006  dfrq       499.851
solvent      CDCl3  dn              H1
file /home/qil/var~ dpwr             47
 500/QL-IV-113.fid  dof          -823.0
   ACQUISITION      dm              nnn
sfrq       499.852  dmm               c
tn              H1  dmf            9900
at           1.892  dseq
np           30272  dres            1.0
sw          8000.0  homo              n
fb            4000      PROCESSING
bs               4  wtfile
tpwr            57  proc             ft
pw             8.0  fn         not used
d1               0  math              f
tof              0
nt             256  werr
ct             108  wexp
alock            n  wbs
gain     not used   wnt             wft
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp           156.1
wp          4747.1
vs              98
sc              35
wc             215
hzmm         22.05
is         2086.70
rfl         1500.6
rfp              0
th               7
ins          1.000
ai  cdc  ph
```



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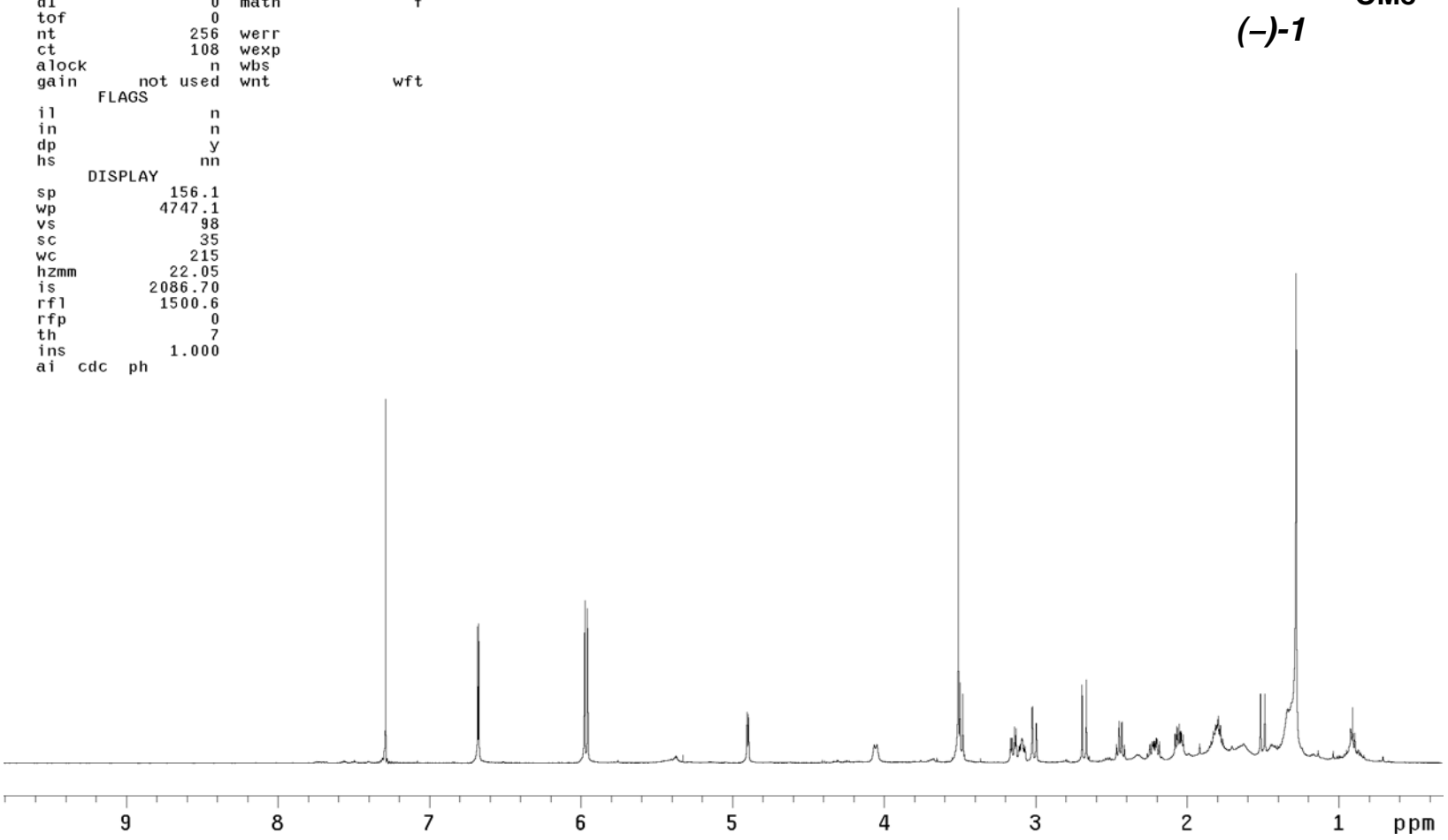

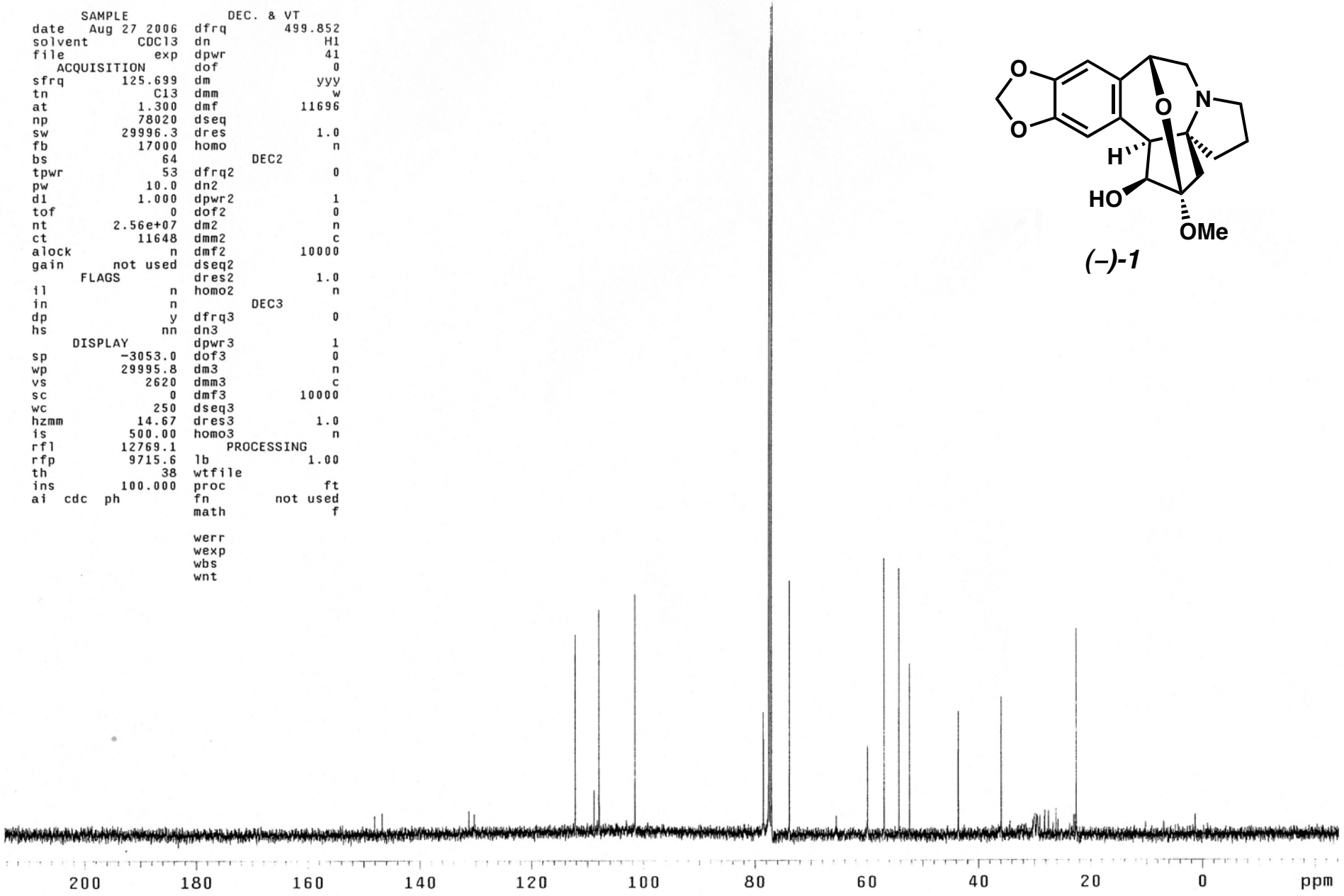
SAMPLE
date Aug 27 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 125.699
tn C13
at 1.300
np 78020
sw 29996.3
fb 17000
bs 64
tpwr 53
pw 10.0
d1 1.000
tof 0
nt 2.56e+07
ct 11648
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -3053.0
wp 29995.8
vs 2620
sc 0
wc 250
hzmm 14.67
is 500.00
rfl 12769.1
rfp 9715.6
th 38
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.852
dn H1
dpwr 41
dof 0
dm yyy
dmm w
dmf 11696
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
O
O
N
H
HO
OMe
(–)-1
200
180
160
140
120
100
80
60
40
20
0
ppm