Supporting Information

Enantioselective Total Synthesis of the *Securinega* Alkaloid (-)-Secu'amamine A

Peng Liu, Sungwoo Hong and Steven M. Weinreb*

Department of Chemistry, The Pennsylvania State University University Park, Pennsylvania 16802

E-mail: smw@chem.psu.edu

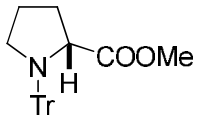
Table of Contents

Title page S1

Experimental details of compounds **5**, **11, 13-16, 18, 23-26, 28-35** S2-S21

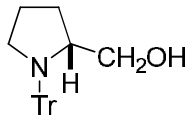
NMR spectra of compounds **5**, **11, 13-16, 18, 23-26, 28-35** S22-S70

General Methods. All non-aqueous reactions were carried out in oven- or flame-dried glassware under an argon atmosphere. All chemicals were purchased from commercial vendors and used as is, unless otherwise specified. Anhydrous tetrahydrofuran (THF) and dichloromethane (CH_2Cl_2) were obtained from a solvent purification system (Glass Contour). Reactions were magnetically stirred and monitored by thin layer chromatography (TLC) with 250 μm EMD 60 F_{254} precoated silica gel plates. Flash column chromatography was performed using EMD silica gel 60 (230-400 mesh). ^{1}H and ^{13}C NMR spectral data were recorded on Bruker DPX-300, CDPX-300, or DRX 400 MHz spectrometers. Chemical shifts are reported relative to chloroform (δ 7.24), dichloromethane (δ 5.32), benzene (δ 7.15) for ^{1}H NMR and chloroform (δ 77.0), dichloromethane (δ 53.8), benzene (δ 128.0) for ^{13}C NMR. Optical rotations were measured on a Rudoph Research Analytical Autopol II digital polarimeter.



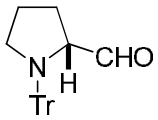
33

Synthesis of *N*-Trityl Ester 33. To a solution of D-proline (15.00 g, 130.30 mmol) in methanol (150 mL) was slowly added thionyl chloride (19.0 mL, 260.5 mmol) at 0 °C. When the addition was complete, the ice bath was removed. The reaction mixture was stirred for an additional 12 h at rt before the solvent and other volatile compounds were removed *in vacuo*. The resulting oil was dissolved in $CHCl_3$ (200 mL), and Et_3N (55 mL, 394.6 mmol) was added followed by trityl chloride (34.50 g, 123.80 mmol). The reaction mixture was stirred for 12 h at rt and hydrolyzed with a 2:1 mixture of saturated aqueous NH_4Cl solution and NH_4OH (28% in water). The aqueous layer was extracted with CH_2Cl_2. The combined organic extracts were dried over $MgSO_4$ and concentrated. The solid obtained was dissolved in THF and filtered. The filtrate was concentrated under reduced pressure. After recrystallization of the residue from Et_2O, compound **33** (40.6 g, 84%) was obtained as a slightly yellow solid: $[\alpha]_D^{20} = +40.1$ (c 2.74, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62-7.59 (m, 6H), 7.33-7.27 (m, 6H), 7.22-7.16 (m, 3H), 3.97 (dd, J = 8.9, 2.5 Hz, 1H), 3.73 (s, 3H), 3.50-3.46 (m, 1H), 2.92-2.89 (m, 1H), 1.70-1.50 (m, 2H), 1.14-0.95 (m, 2H); ^{13}C NMR (75 MHz, CDCl3) δ 177.1, 144.7, 129.2, 127.6, 126.1, 77.4, 62.7, 51.5, 49.9, 31.2, 24.2; HRMS-ES (*m/z*): $[M + Na]^+$ calcd for $C_{25}H_{25}NO_2Na$, 394.1783; found, 394.1793.



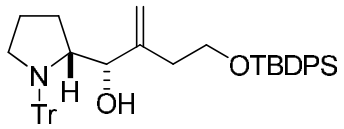
34

Synthesis of *N*-Trityl Alcohol 34. A solution of ester **33** (39.00 g, 105.00 mmol) in dry THF (150 mL) was added slowly via an addition funnel to a slurry of $LiAlH_4$ (3.19 g, 84.00 mmol) in dry THF (50 mL) at 0 °C. After the addition was complete, the reaction mixture was allowed to warm to rt and stirred for 12 h. The reaction mixture was cooled to 0 °C, and then carefully quenched with water (3.2 mL), 15% aqueous NaOH (3.2 mL), and water (9.6 mL). After being stirred for 30 min, the mixture was diluted with EtOAc, filtered through Celite and the phases were separated. The aqueous phase was saturated with NaCl and extracted with EtOAc. The combined organic phases were dried over $MgSO_4$, and concentrated to yield the title compound as a white foam (34.26 g, 95%), which was used without further purification. For analytical purposes, alcohol **34** was purified by flash column chromatography on silica gel (hexanes/EtOAc/Et_3N, 9/1/0.1) to give a white foam: $[\alpha]_D^{20} = -37.7$ (c 2.17, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.65-7.62 (m, 6H), 7.34-7.28 (m, 6H), 7.25-7.22 (m, 3H), 3,70-3.66 (m, 1H), 3.62 (d, J = 7.1 Hz, 1H), 3,57-3.54 (m, 1H), 3.27-3.24 (m, 1H), 3.10-3.04 (m, 1H), 2.40 (br s, 1H), 1.48-1.44 (m, 2H), 1.10-1.02 (m, 1H), 0.70-0.63 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 129.5, 127.5, 126.1, 77.6, 65.7, 61.1, 50.9, 29.0, 24.1; HRMS-ES (*m/z*): $[M + Na]^+$ calcd for $C_{24}H_{25}NONa$, 366.1834; found, 366.1824.



11

Synthesis of *N*-Trityl Aldehyde 11. To a solution of $(COCl)_2$ (13.0 mL, 148.5 mmol) in CH_2Cl_2 (120 mL) cooled at -78 °C was added dropwise a solution of DMSO (17.6 mL, 247.5 mmol) in CH_2Cl_2 (70 mL) via an addition funnel. After the addition was complete, the reaction was stirred for 30 min before a solution of the alcohol **34** (34.00 g, 99.00 mmol) in CH_2Cl_2 (90 mL) was added dropwise at -78 °C. After stirring the mixture for 1.5 h at this temperature, Et_3N (55.2 mL, 396.0 mmol) was added. The reaction mixture was further stirred for 1.5 h at -78 °C. and then quenched with a 2:1 mixture of a saturated aqueous NH_4Cl solution and NH_4OH (28% in water). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic extracts were dried over $MgSO_4$ and concentrated under reduced pressure. The solid obtained was dissolved in THF and filtered. The filtrate was concentrated *in vacuo* to give, after recrystallization from ether, the title compound (30.40 g, 90%) as a slightly yellow solid: $[\alpha]_D^{20} = +12.5$ (c 2.55, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.77 (d, J = 2.8 Hz, 1H), 7.50-7.47 (m, 6H), 7.21-7.16 (m, 6H), 7.12-7.07 (m, 3H), 3.71-3.65 (m, 1H), 3.24-3.16 (m, 1H), 2.88-2.80 (m, 1H), 1.55-1.47 (m, 1H), 1.38-1.30 (m, 1H), 1.07-1.00 (m, 1H), 0.74-0.69 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.4, 144.4, 129.4, 127.7, 126.4, 76.9, 50.5, 28.0, 24.2; HRMS-ES (*m/z*): $[M + Na]^+$ calcd for $C_{24}H_{23}NONa$, 364.1677; found, 364.1657.

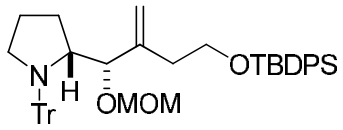


13

Synthesis of Alcohol 13. Magnesium turnings (5.991 g, 246.540 mmol) were flame dried, cooled under argon and then immersed in THF (20 mL). Two drops of 1,2-dibromomethane were added to the mixture which was then heated at reflux for 15 min. The heating was stopped and a solution of vinyl bromide **12** (24.00 g, 61.63 mmol) in THF (50 mL) was added dropwise via an addition funnel to keep a gentle reflux. After the addition was complete, the reaction mixture was stirred further at reflux for 30 min and then cooled to rt.

The resulting Grignard reagent was added via cannula to a solution of the aldehyde **11** (14.028 g, 41.088 mmol) in THF (50 mL) at -78 ^{o}C. The reaction mixture was stirred for 4 h at -78 ^{o}C and then quenched by the addition of saturated aqueous NH_4Cl (5 mL). The solution was diluted with EtOAc and H_2O. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous layer was further extracted with EtOAc. The combined organic phases were dried over $MgSO_4$ and concentrated *in vacuo*. Purification of the residue via flash chromatography on silica gel (hexanes/EtOAc/Et_3N, 9.5/0.5/0.1) afforded the desired alcohol **13** (25.449 g, 95%) as a white foam: $[\alpha]_D^{20} = +24.5$ (c 5.88, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, J = 6.6 Hz, 4H), 7.64 (d, J = 7.8 Hz, 6H), 7.54-7.45 (m, 6H), 7.39-7.21 (m, 9H), 5.23 (s, 1H), 4.91 (s, 1H), 4.45 (s, 1H), 3.71-3.67 (m, 1H), 3.63 (t, J = 7.1 Hz, 2H), 3.36-3.27 (m, 2H), 3.14-3.07 (m, 1H), 2.21-2.11 (m, 1H), 2.02-1.95 (m, 1H), 1.70-1.59 (m, 1H), 1.43-1.35 (m, 1H), 1.14 (s, 9H), 1.08-1.01 (m, 1H), 0.39-0.26 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$)

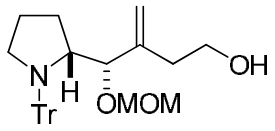
δ 145.4, 144.5, 135.5, 135.4, 133.8, 129.7, 129.5, 127.5, 127.4, 126.2, 110.2, 78.0, 75.6, 62.6, 62.4, 52.9, 36.2, 26.8, 25.2, 24.8, 19.1; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{44}H_{50}NO_2Si$, 652.3611; found, 652.3618.



14

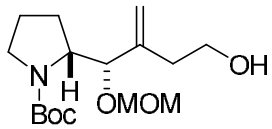
Synthesis of MOM Ether 14. To a solution of the alcohol **13** (25.00 g, 38.35 mmol) in DCM (200 mL) at rt was added DIPEA (33.4 mL, 191.9 mmol) followed by MOMBr (12.5 mL, 153.4 mmol). The reaction mixture was then heated at 50 ^oC and stirred for 24 h. The reaction mixture was concentrated under reduced pressure and the residue obtained was dissolved in THF and filtered. The filtrate was then concentrated and the resulting oil was subjected to silica gel chromatography (hexanes/EtOAc/Et_3N, 8/2/0.1) to give the title compound (24.00 g, 90%) as a slightly yellow oil: $[\alpha]_D^{20} = +38.8$ (c 5.73 , $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.75-7.71 (m, 4H), 7.67 (d, *J* = 7.2 Hz, 6H), 7.52-7.47 (m, 6H), 7.30 (t, *J* = 7.6 Hz, 6H), 7.21 (t, *J* = 7.2 Hz, 3H), 5.08 (s, 1H), 4.90 (d, *J* = 6.5 Hz, 1H), 4.87 (s, 1H), 4.81 (d, *J* = 6.5 Hz, 1H), 4.77 (s, 1H), 3.72 (s, 3H), 3.64 (t, *J* = 7.0 Hz, 2H), 3.56 (d, *J* = 8.1 Hz, 1H), 3.49-3.36 (m, 1H), 3.13-3.05 (m, 1H), 1.97-1.90 (m, 1H), 1.83-1.72 (m, 1H), 1.71-1.64 (m, 1H), 1.47-1.38 (m, 1H), 1.13 (s, 9H), 0.88-0.77 (m, 1H), 0.34-0.22 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.35, 144.15, 135.54, 135.51, 133.95, 133.87, 129.66, 129.53, 127.57, 127.47, 126.00, 111.47, 94.95,

82.69, 78.42, 62.84, 62.10, 55.84, 51.83, 36.16, 26.77, 25.34, 24.13, 19.15; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{46}H_{54}NO_3Si$, 696.3873; found, 696.3914.



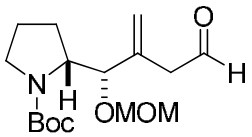
35

Synthesis of Alcohol 35. To a solution of silyl ether **14** (23.00 g, 33.05 mmol) in THF (100 mL) was added TBAF (1 M solution in THF, 49.6 mL, 49.6 mmol) at rt. After 12 h at rt, the reaction mixture was diluted with saturated aqueous NH_4Cl (60.0 mL) and then EtOAc and H_2O. NaCl was added to saturate the aqueous phase and the organic phase was separated. The aqueous phase was extracted with EtOAc. The combined organic extracts were dried over $MgSO_4$ and concentrated in *vacuo*. The crude material was purified by silica gel flash chromatography ((hexanes/EtOAc/Et_3N, 9/1/0.1) to give the title compound (14.370 g, 95%) as a slightly yellow oil: $[\alpha]_D^{20} = +28.4$ (c 9.5, $CHCl_3$); ^1H NMR (300 MHz, CD_2Cl_2) δ 7.64-7.59 (m, 6H), 7.31-7.25 (m, 6H), 7.22-7.13 (m, 3H), 5.08 (t, *J* = 1.6 Hz, 1H), 4.90 (d, *J* = 0.8 Hz, 1H), 4.87 (d, *J* = 6.5 Hz, 1H), 4.73 (d, *J* = 6.6 Hz, 1H), 4.68 (s, 1H), 3.61 (s, 3H), 3.50-3.46 (m, 3H), 3.42-3.32 (m, 1H), 3.06-2.98 (m, 1H), 1.99-1.89 (m, 1H), 1.68-1.58 (m, 3H), 1.40-1.29 (m, 1H), 0.77-0.69 (m, 1H), 0.32-0.22 (m, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 145.8, 145.2, 130.1, 127.9, 126.4, 112.5, 95.8, 83.0, 78.8, 63.2, 61.1, 56.1, 52.1, 37.2, 25.7, 24.4; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{30}H_{36}NO_3$, 458.2695; found, 458.2722.



15

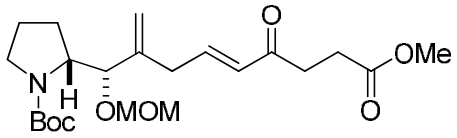
Synthesis of Carbamate Alcohol 15. To a solution of alcohol **35** (14.15 g, 30.92 mmol) in MeOH (200 mL) was added AcOH (14.0 mL). After the mixture was stirred at rt for 12 h, TEA (40 mL) was added followed by $(Boc)_2O$ (10.12 g, 46.38 mmol). The reaction mixture was stirred for an additional 6h at rt and the solvent was removed under reduced pressure. Purification of the residue via flash chromatography on silica gel (hexanes/EtOAc/Et_3N, 6/4/0.1) afforded the title compound (8.680 g, 89%) as a colorless oil: $[\alpha]_D^{20} = +125.2$ (c 6.15, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.16 (s, 0.5H), 5.07 (s, 0.5H), 5.00 (s, 1H), 4.51 (d, J = 6.7 Hz, 1H), 4.45 (d, J = 6.6 Hz, 1H), 4.32 (d, J = 5.0 Hz, 0.5 H), 3.94 (br s, 0.5 H), 3.82-3.70 (m, 3H), 3.45 (m, 0.5H), 3.34 (m, 0.5H), 3.29 (s, 1.5H), 3.26 (m, 1H), 3.25 (s, 1.5H), 2.91 (br s, 0.5 H), 2.44-2.35 (m, 0.5H), 2.31-2.16 (m, 1.5H), 2.07-1.93 (m, 2H), 1.71-1.66 (m, 2H), 1.45 (s, 4.5H), 1.38 (s, 4.5 H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.4, 153.9, 143.4, 143.2, 112.7, 112.2, 94.3, 94.2, 79.3, 79.0, 77.8, 77.3, 60.5, 60.1, 58.3, 58.2, 55.4, 55.2, 55.0, 46.9, 46.8, 36.0, 35.4, 28.2, 24.8, 24.7, 23.8, 23.5; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{16}H_{30}NO_5$, 316.2124; found, 316.2108.



16

Synthesis of Aldehyde 16. To a solution of the carbamate alcohol **15** (6.200 g, 19.660 mmol) in CH_2Cl_2 (200 mL) was added $NaHCO_3$ (7.430 g, 88.470 mmol) followed

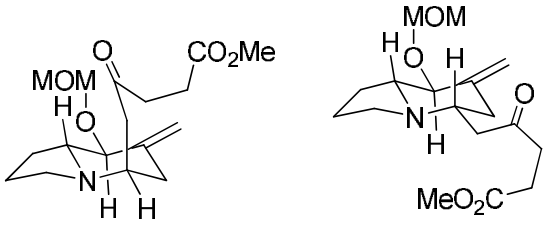
by Dess-Martin periodinane (12.50 g, 29.49 mmol). After 4 h, 10% $Na_2S_2O_3$ aqueous solution (200 mL) was added to the reaction mixture. After the solids dissolved, the mixture was extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to give a pale yellow oil. The aldehyde obtained was unstable and decomposed during chromatographic purification and therefore was used in crude form.



18

Synthesis of (*E*)-Enone 18. To a suspension of NaH (60% dispersion in mineral oil, 1.415 g, 35.390 mmol) in THF (15 mL) was slowly added a solution of the phosphonate **17** (9.364 g, 39.320 mmol) in THF (50 mL) at rt. After 45 min at rt, a solution of the crude aldehyde **16** (6.161 g, 19.660 mmol) in THF (40 mL) was added. The reaction mixture was stirred for 1.5 h and quenched with saturated aqueous NH_4Cl (5 mL). The resulting cloudy solution was diluted with EtOAc and H_2O. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous phase was then extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 and concentrated. The crude residue was chromatographed over silica gel to give the title compound (5.850 g, 70% based on carbamate alcohol **15**) as a slightly yellow oil: $[\alpha]_D^{20} = +87.5$ (c 6.40, $CHCl_3$); ^{1}H NMR (400 MHz , $CDCl_3$) δ 6.93-6.71 (m, 1H), 6.17 (d, J = 15.9 Hz, 1H), 5.20 (d, J = 26.3 Hz, 1H), 4.95 (d, J = 19.2 Hz, 1H), 4.54-4.45 (m, 3H), 3.93 (br s, 0.5H), 3.79 (br s, 0.5H), 3.67 (s, 3H), 3.54 (br s, 0.5H), 3.39 (br s, 0.5H), 3.32 (s, 2H), 3.30 (s, 2H), 3.08-3.02 (m, 0.5H), 2.95-2.88 (m, 3.5H), 2.65-2.59 (m, 2H), 2.01-

1.95 (m, 2H), 1.76-1.69 (m, 2H), 1.48 (s, 4.5H), 1.45 (s, 4.5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.96, 197.62g, 173.24, 154.44, 153.98, 144.57, 143.82, 143.75, 143.17, 131.43, 131.25, 113.64, 94.56, 94.30, 79.56, 78.99, 78.15, 77.72, 58.55, 58.45, 55.44, 55.36, 51.67, 47.01, 35.55, 35.27, 34.62, 34.40, 28.43, 27.69, 27.61, 25.18, 24.84, 24.16, 23.72; HRMS-ES (*m/z*): $\left[M + H\right]^+$ calcd for $C_{22}H_{36}NO_7$, 426.2492; found, 426.2468.



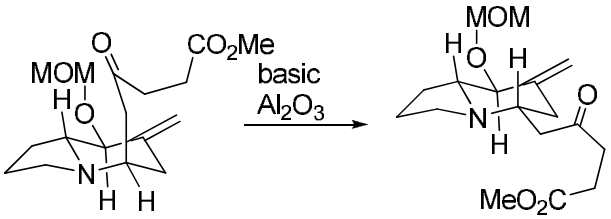
24 **23**

Synthesis of Indolizidines 24 and 23. To a solution of the enone **18** (71.0 mg, 0.167 mmol) in CH_2Cl_2 (6 mL) was added TFA (0.26 mL, 3.34 mmol). After the reaction mixture was stirred for 6 h at rt, the solvent was removed under reduced pressure and the resulting residue was evacuated under high vacuum for 1 h. The residue was then dissolved in CH_2Cl_2 (10 mL) and cooled to -78 °C. DIPEA (0.29 mL, 1.67 mmol) was slowly added along the inner wall of the flask. After 4 h at this temperature, the mixture was warmed to rt and the solvent was removed *in vacuo*. Purification of the residue by silica gel chromatography (hexanes/EtOAc/Et_3N, 4/6/0.1) afforded the major conjugate addition product **24** (32.0 mg, 59 %) and the minor isomer **23** (6.0 mg, 11%).

Major isomer **24** (pale yellow oil): $\left[\alpha\right]_D^{20} = +86.9$ (c 3.50, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.02 (s, 1H), 4.80 (d, J = 1.6 Hz, 1H), 4.64 (dd, J = 17.9 Hz, 6.8 Hz, 2H), 3.71 (d, J = 8.9 Hz, 1H), 3.63 (s, 3H), 3.56 (dd, J = 12.4 Hz, 5.8 Hz, 1H), 3.37 (s, 3H), 2.85 (td, J = 8.8 Hz, 3.6 Hz, 1H), 2.71-2.66 (m, 2H), 2.55-2.45 (m, 6H), 2.39 (dd, J =

16.0 Hz, 7.2 Hz, 1H), 2.13 (dd, J = 13.3 Hz, 1.9 Hz, 1H), 2.02-1.93 (m, 1H), 1.85-1.76 (m, 1H), 1.72-1.56 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.3, 173.1, 143.5, 108.7, 95.7, 79.9, 61.6, 55.8, 51.7, 51.3, 49.1, 38.1, 37.6, 37.5, 28.9, 27.6, 21.3; HRMS-ES (*m/z*): $\left[M + H\right]^+$ calcd for $C_{17}H_{28}NO_5$, 326.1907; found, 326.1946.

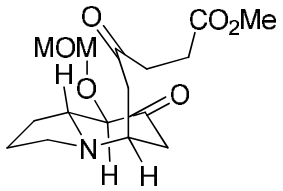
Minor isomer **23** (pale yellow oil): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.91 (d, J = 1.6 Hz, 1H), 4.78 (d, J = 1.7 Hz, 1H), 4.66 (dd, J = 11.4 Hz, 6.7 Hz, 2H), 3.79 (d, J = 9.0 Hz, 1H), 3.64 (s, 3H), 3.37 (s, 3H), 3.02 (td, J = 8.3 Hz, 2.3 Hz, 1H), 2.82-2.69 (m, 3H), 2.64-2.53 (m, 3H), 2.48-2.35 (m, 2H), 2.09-1.90 (m, 4H), 1.79-1.60 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.3, 173.5, 145.9, 107.0, 96.4, 80.5, 69.7, 58.8, 56.2, 52.2, 51.7, 48.3, 40.6, 38.4, 29.3, 28.0, 21.4; HRMS-ES (*m/z*): $\left[M + H\right]^+$ calcd for $C_{17}H_{28}NO_5$, 326.1907; found, 326.1964.



24 **23**

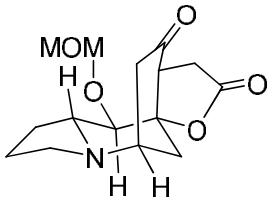
Isomerization of 24 to 23. To a solution of the major conjugate addition product **24** (15.0 mg, 0.0461 mmol) in CH_2Cl_2 (5 mL) was added basic alumina (EMD Chemicals TLC/GL AL OX60 F254, 2.00 g). The heterogenous mixture was stirred for 5 min and the solvent was removed *in vacuo*. The remaining solid was evacuated under high vacuum. After 12 h at rt, the alumina was washed with 10% isopropanol in $CHCl_3$ and filtered. The elutant was concentrated and the residue was purified by silica gel

chromatography (hexanes/EtOAc/Et_3N, 4/6/0.1) to afford the minor conjugate addition product **23** (12.0 mg, 80%).

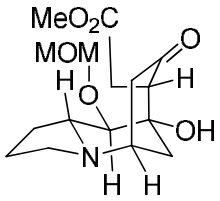


25

Synthesis of Diketo Ester 25. To a solution of the major indolizidine **24** (36.0 mg, 0.111 mmol) in THF (6 mL) and H_2O (2 mL) at 0 °C was added OsO_4 (4 wt% solution in water, 0.21 mL, 0.033 mmol).After the mixture was stirred for 15 min at this temperature, a solution of $NaIO_4$ (118.7 mg, 0.555 mmol) in H_2O (3 mL), and a solution of NMO (65.0 mg, 0.555 mmol) in H_2O (3 mL) were added. The reaction mixture was stirred for another 5 h at 0 °C. The mixture was then partitioned between EtOAc and H_2O and the aqueous phase was saturated with NaCl. The organic layer was separated and the aqueous layer was extracted with EtOAc. The combined organic phases were dried over Na_2SO_4 and concentrated. The residue was purified by silica gel chromatography (hexanes/EtOAc/Et_3N, 4/6/0.1) to give compound **25** (23.0 mg, 63%) as a colorless oil: $[\alpha]_D^{20} = +88.4$ (c 1.90, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.71 (d, J = 7.0 Hz, 1H), 4.62 (d, J = 7.0 Hz, 1H), 3.98-3.89 (m, 1H), 3.95 (d, J = 9.4 Hz, 1H), 3.62 (s, 3H), 3.36 (s, 3H), 2.98-2.86 (m, 2H), 2.81 (dd, J = 13.4 Hz, 6.4 Hz, 1H), 2.74-2.61 (m, 3H), 2.60-2.48 (m, 3H), 2.39 (dd, J = 16.5 Hz, 8.7 Hz, 1H), 2.20 (dd, J = 13.3 Hz, 2.0 Hz, 1H), 2.13-2.04 (m, 1H), 1.96-1.87 (m, 1H), 1.85-1.73 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.6, 206.9, 172.9, 96.1, 81.0, 62.4, 55.9, 52.9, 51.8, 49.4, 43.5, 40.8, 38.0, 30.0, 27.5, 22.1; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{16}H_{26}NO_6$, 328.1760; found, 328.1779.



26

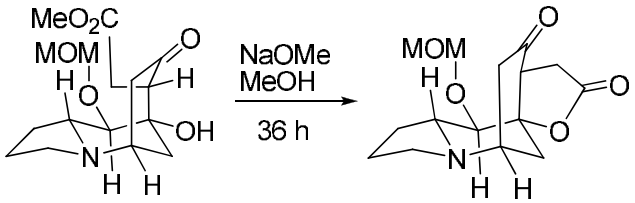


28

Synthesis of Tetracyclic γ-Lactone 26. To a solution of diketo ester **25** (95.0 mg, 0.290 mmol) in MeOH (20 mL) was added NaOMe (24.0 mg, 0.444 mmol). The solution was stirred for 12 h at rt and then quenched with saturated aqueous NH_4Cl (1.0 mL). The methanol solvent was removed under reduced pressure. The cloudy aqueous solution was diluted with H_2O and extracted with 15% isopropanol in $CHCl_3$. The combined organic phases were dried over Na_2SO_4 and concentrated. Purification of the residue by chromatography on silica gel (hexanes/EtOAc/Et_3N, 3/7/0.1) yielded compound **26** (64.0 mg, 75 %) as a colorless oil: $[\alpha]_D^{20} = +12.6$ (c 1.90, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.74 (d, *J* = 6.8 Hz, 1H), 4.61 (d, *J* = 6.8 Hz, 1H), 3.64 (d, *J* = 9.1 Hz, 1H), 3.58-3.53 (m, 1H), 3.47 (t, *J* = 10.6 Hz, 1H), 3.34 (s, 3H), 2.91-2.68 (m, 3H), 2.55 (d, *J* = 18.3 Hz, 1H), 2.48-2.33 (m, 3H), 2.26-2.19 (m, 1H), 2.11-2.03 (m, 1H), 1.93-1.84 (m, 1H), 1.81-1.67 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.4, 173.9, 97.4, 86.8, 79.9, 59.4, 56.1, 48.8, 48.7, 47.1, 36.6, 35.5, 29.8, 28.6, 21.5; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{15}H_{22}NO_5$, 296.1498; found, 296.1515.

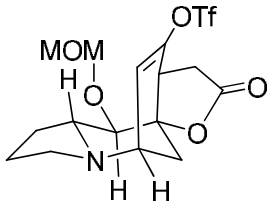
By further elution of the silica gel column with EtOAc/isopropanol/Et_3N (9.5/0.5/0.1), hydroxy ester **28** (14.2 mg, 15%) was obtained as a pale yellow oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.73 (d, *J* = 5.9 Hz, 1H), 4.46 (d, *J* = 5.9 Hz, 1H), 4.34 (s, 1H), 3.67 (s, 3H), 3.45 (d, *J* = 2.8 Hz, 1H), 3.41 (s, 3H), 3.22 (d, *J* = 9.8 Hz, 1H), 3.15 (dd, *J* = 10.1 Hz, 2.7 Hz, 1H), 2.93 (dd, *J* = 16.8 Hz, 10.1 Hz, 1H), 2.82 (td, *J* = 8.1 Hz,

4.1 Hz, 1H), 2.78 (dt, *J* = 16.5 Hz, 2.0 Hz, 1H), 2.69 (dd, *J* = 16.8 Hz, 2.9 Hz, 1H), 2.67-2.61 (m, 1H), 2.36-2.27 (m, 2H), 2.19 (dd, *J* =12.9 Hz, 2.7 Hz, 1H), 2.13 (dt, *J* = 12.1 Hz, 3.0 Hz 1H), 1.93-1.83 (m, 1H), 1.85-1.75 (m, 1H), 1.72-1.63 (m, 1H), 1.46-1.39 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.3, 174.2, 98.7, 89.6, 73.8, 59.4, 58.6, 56.1, 51.7, 50.7, 48.4, 41.0, 40.4, 29.4, 28.5, 21.3; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{16}H_{26}NO_6$, 328.1760; found, 328.1756.



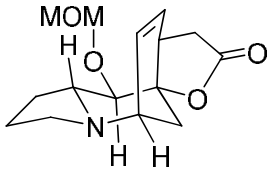
28 **26**

Transformation of Hydroxyl Ester 28 to Lactone 26. To a solution of hydroxy ester **28** (6.0 mg, 0.0306 mmol) in MeOH (5 mL) was added NaOMe (20.0 mg, 0.370 mmol). After stirring the mixture for 36 h at rt, the reaction was quenched with saturated aqueous NH_4Cl (1 mL). The solvent was removed under reduced pressure and the aqueous solution was diluted with H_2O and extracted with 15% isopropanol in $CHCl_3$. The combined organic phases were dried over Na_2SO_4 and concentrated. Purification of the residue by chromatography on silica gel (hexanes/EtOAc/Et_3N, 3/7/0.1) yielded lactone **26** (4.9 mg, 90 %).



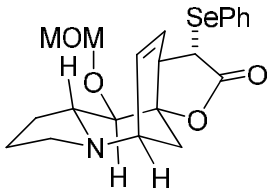
29

Synthesis of Enol Triflate 29. A solution of ketone **26** (95.0 mg, 0.322 mmol) in THF (10.0 mL) was added dropwise to a solution of KHMDS (0.5 M in toluene, 1.29 mL, 0.65 mmol) in THF (2 mL) at -78 ^{o}C. After 1 h at this temperature, a solution of *N*-phenyltriflimide (138.0 mg, 0.386 mmol) in THF (2 mL) was added slowly to the mixture. The reaction mixture was stirred at -78 ^{o}C for another 12 h and then quenched with saturated aqueous NH_4Cl (1 mL). The solution was diluted with EtOAc and H_2O and the aqueous phase was saturated with NaCl. The organic phase was then separated and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 and concentrated. The residue was purified by silica gel column chromatography (hexanes/EtOAc/Et_3N, 6/4/0.1) to give enol triflate **29** (117.0 mg, 85%) as a colorless oil: $[\alpha]_D^{20} = -10.7$ (c 3.55, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.93 (d, *J* = 6.4 Hz, 1H), 4.74 (d, *J* = 6.8 Hz, 1H), 4.62 (d, *J* = 6.8 Hz, 1H), 3.79-3.75 (m, 1H), 3.64 (d, *J* = 9.5 Hz, 1H), 3.54 (t, *J* = 10.5 Hz, 1H), 3.36 (s, 3H), 2.91 (dd, *J* = 8.4 Hz, 2.7 Hz, 1H), 2.85 (dd, *J* = 17.8 Hz, 9.6 Hz, 1H), 2.63 (dd, *J* = 17.6 Hz, 11.9 Hz, 1H), 2.38 (dd, *J* = 16.5 Hz, 8.3 Hz, 1H), 2.32-2.24 (m, 1H), 2.08 (dd, *J* = 12.4 Hz, 3.1 Hz, 1H), 2.05-1.99 (m, 1H), 1.91-1.83 (m, 1H), 1.79 (dd, *J* = 12.3 Hz, 3.0 Hz, 1H), 1.77-1.68 (m, 1H), 1.67-1.58 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.9, 149.7, 124.8, 120.6, 116.3, 115.4, 112.1, 97.3, 85.9, 78.8, 58.8, 56.0, 49.5, 48.6, 37.2, 32.9, 33.8, 27.8, 21.8; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{16}H_{21}NO_7SF_3$, 428.0991; found, 428.0967.



30

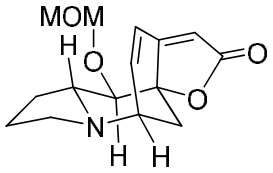
Synthesis of Alkene 30. To a solution of enol triflate **29** (71.0 mg, 0.166 mmol), DIPEA (0.12 mL, 0.689 mmol), $Pd(OAc)_2$ (0.4 mg, 0.002 mmol) and PPh_3 (1.0 mg, 0.004 mmol) in DMF (1.5 mL) was added formic acid (22.9 mg, 0.498 mmol). The solution was stirred at 60 ^oC for 1 h. During this period, the mixture became black. After cooling, the mixture was diluted with EtOAc and washed with brine. The organic phase was dried over Na_2SO_4 and concentrated. The residue was purified by silica gel column chromatography (hexanes/EtOAc/Et_3N, 3/7/0.1) to give alkene **30** (44.1 mg, 95%) as a colorless oil: $[\alpha]_D^{20} = -104.4$ (c 0.90, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 5.99 (dd, *J* = 9.7 Hz, 3.7 Hz, 1H), 5.82-5.78 (m, 1H), 4.75 (d, *J* = 6.7 Hz, 1H), 4.60 (d, *J* = 6.7 Hz, 1H), 3.61 (d, *J* = 9.4 Hz, 1H), 3.57 (dd, *J* = 5.7 Hz, 2.9 Hz, 1H), 3.34 (s, 3H), 3.20-3.09 (m, 1H), 2.88 (td, *J* = 8.5 Hz, 3.0 Hz, 1H), 2.73 (dd, *J* = 17.4 Hz, 9.3 Hz, 1H), 2.42-2.21 (m, 3H), 2.01 (dd, *J* = 12.3 Hz, 2.8 Hz, 1H), 1.96-1.95 (m, 1H), 1.89-1.81 (m, 1H), 1.75 (dd, *J* = 12.0 Hz, 3.0 Hz, 1H), 1.71-1.57 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.0, 130.8, 124.4, 97.5, 85.7, 79.8, 58.8, 55.9, 50.8, 48.5, 34.9, 34.7, 33.7, 27.7, 21.8; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{16}H_{22}NO_4$, 280.1549; found, 280.1552.



31

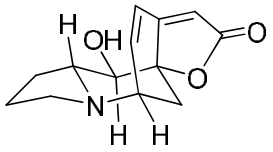
Synthesis of Selenide 31. To a solution of the lactone **30** (21.0 mg, 0.0752 mmol) in THF (3 mL) at -78 ^{o}C was added LDA (2.0 M in heptane/THF/ethylbenzne, 0.15 mL, 0.30 mmol). After stirring the mixture for 1 h at this temperature, a solution of PhSeCl (28.8 mg, 0.150 mmol) in THF (2 mL) was slowly added. The reaction mixture was stirred at -78 ^{o}C for another 12 h and then quenched with saturated aqueous NH_4Cl (1.0 mL). The mixture was diluted with EtOAc and H_2O, and NaCl was then added to saturate the aqueous phase. The organic phase was separated and the aqueous phase was extracted with EtOAc. The combined organic extracts were dried over Na_2SO_4 and concentrated. The residue was subjected to silica gel column chromatography (hexanes/EtOAc/Et_3N, 3/7/0.1) to provide the selenide (28.0 mg, 86%) as a colorless oil: $[\alpha]_D^{20} = -127.1$ (c 1.40, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72-7.68 (m, 2H), 7.38-7.26 (m, 3H), 6.06 (dd, *J* = 9.7 Hz, 3.7 Hz, 1H), 5.84 (ddd, *J* = 9.6 Hz, 5.7 Hz, 1.8 Hz, 1H), 4.48 (d, *J* = 6.8 Hz, 1H), 4.15 (d, *J* = 6.8 Hz, 1H), 3.63 (d, *J* = 12.1 Hz, 1H), 3.57 (dt, *J* = 6.0 Hz, 3.0 Hz, 1H), 3.50 (d, *J* = 9.5 Hz, 1H), 3.20 (s, 3H), 3.06 (ddd, *J* = 12.1 Hz, 3.7 Hz, 2.0 Hz, 1H), 2.86 (td, *J* = 8.5 Hz, 3.2 Hz, 1H), 2.36 (dd, *J* = 16.4 Hz, 8.6 Hz, 1H), 2.16 (td, *J* = 8.8 Hz, 6.4 Hz, 1H), 1.99 (dd, *J* = 12.1 Hz, 2.9 Hz, 1H), 1.94-1.88 (m, 1H), 1.82-1.76 (m, 1H), 1.68 (dd, *J* = 11.9 Hz, 3.0 Hz, 1H), 1.64-1.52 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.8, 136.7, 129.5, 129.2, 129.0, 125.9, 125.0, 97.0, 84.7, 79.5, 58.8, 55.7, 51.0, 48.5, 44.1,

41.3, 34.0, 27.7, 21.8; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{21}H_{26}NO_4Se$, 436.1027; found, 436.1019.



32

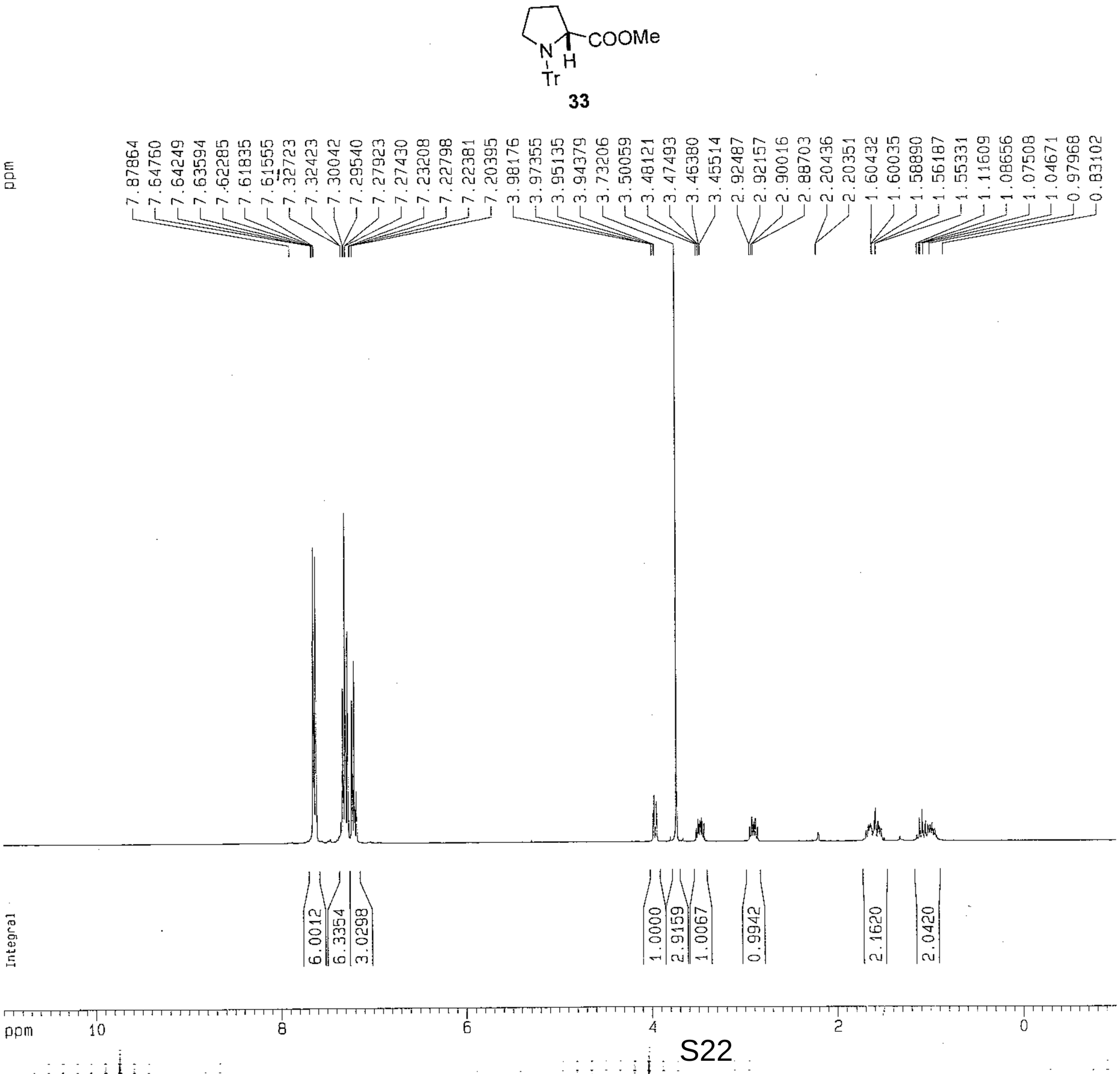
Synthesis of Diene Lactone 32. To a solution of selenide **31** (28.0 mg, 0.065 mmol) in MeOH (8 mL) and H_2O (4 mL) was added $NaHCO_3$ (11.0 mg, 0.131 mmol) followed by $NaIO_4$ (69.0 mg, 0.323 mmol) at rt. The reaction mixture was stirred for 1 h at this temperature and then methanol was removed *in vacuo*. The resulting cloudy solution was diluted with H_2O and EtOAc. The aqueous phase was saturated with NaCl and the organic phase was separated. The aqueous layer was subsequently extracted with EtOAc and 15% isopropanol in $CHCl_3$. The combined organic extracts were dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude material obtained was purified by silica gel column chromatography (hexanes/EtOAc/Et_3N, 3/7/0.1) to yield diene lactone **32** (15.0 mg, 84%) as a colorless oil: $[\alpha]_D^{20} = -468.6$ (c 0.70, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.77 (d, *J* = 9.5 Hz, 1H), 6.14 (dd, *J* = 9.5 Hz, 5.5 Hz, 1H), 5.83 (s, 1H), 4.75 (d, *J* = 6.9 Hz, 1H), 4.56 (d, *J* = 7.0 Hz, 1H), 3.88 (dd, *J* = 5.5 Hz, 2.8 Hz, 1H), 3.62 (d, *J* = 9.6 Hz, 1H), 3.33 (s, 3H), 2.98 (td, *J* = 8.6 Hz, 3.3 Hz, 1H), 2.54-2.43 (m, 2H), 2.33 (dd, *J* = 11.6 Hz, 2.8 Hz, 1H), 2.10-1.98 (m, 1H), 1.97 (dd, *J* = 11.6 Hz, 3.2 Hz, 1H), 1.91-1.84 (m, 1H), 1.74-1.65 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 162.0, 133.5, 124.3, 114.3, 98.1, 86.7, 80.4, 59.1, 56.2, 51.9, 48.5, 37.5, 28.2, 22.1; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{21}H_{20}NO_4$, 278.1392; found, 278.1385.



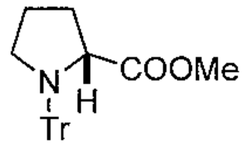
5

Synthesis of (-)-Secu'amamine A (5). To a solution of diene lactone **32** (14.0 mg, 0.050 mmol) in MeOH (6 mL) was added conc. HCl (0.60 mL). The reaction mixture was then stirred for 5 h at 60 ^{o}C. After cooling the mixture to rt, saturated aqueous $NaHCO_3$ (3 mL) was added and methanol was removed under reduced pressure. The resulting cloudy solution was then diluted with H_2O and 15% isopropanol in $CHCl_3$. The aqueous phase was saturated with NaCl, and the organic phase was separated. The aqueous layer was extracted with 15% isopropanol in $CHCl_3$. The combined organic layers were dried over Na_2SO_4 and concentrated. Purification of the residue via silica gel column chromatography (hexanes/EtOAc/Et_3N, 1/9/0.1) gave (-)-Secu'amamine A (**5**) (11.0 mg, 93 %) as a colorless oil: $[\alpha]_D^{20} = -511.3$ (c 0.15, $CHCl_3$); reported rotation: $[\alpha]_D^{20} = -479$ (c 0.15, $CHCl_3$); X-Ray quality crystals were prepared via slow evaporation from deuteriobenzene; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.76 (d, *J* = 9.6 Hz, 1H), 6.16 (dd, *J* = 9.5 Hz, 5.6 Hz, 1H), 5.83 (s, 1H), 3.90 (dt, *J* = 5.5 Hz, 2.8 Hz, 1H), 3.70 (d, *J* = 9.6 Hz, 1H), 2.98 (td, *J* = 8.6 Hz, 3.7 Hz, 1H), 2.68 (br s, 1H), 2.60-2.47 (m, 2H), 2.34 (dd, *J* = 11.6 Hz, 2.8 Hz, 1H), 2.09-2.03 (m, 1H), 1.98 (dd, *J* = 11.6 Hz, 3.2 Hz, 1H), 1.94-1.86 (m, 1H), 1.78-1.72 (m, 1H), 1.68-1.60 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.42, 162.08, 133.99, 124.34, 114.06, 87.05, 74.80, 59.53, 52.12, 48.60, 36.88, 28.24, 22.19; HRMS-ES (*m/z*): $[M + H]^+$ calcd for $C_{13}H_{16}NO_3$, 234.1130; found, 234.1150.

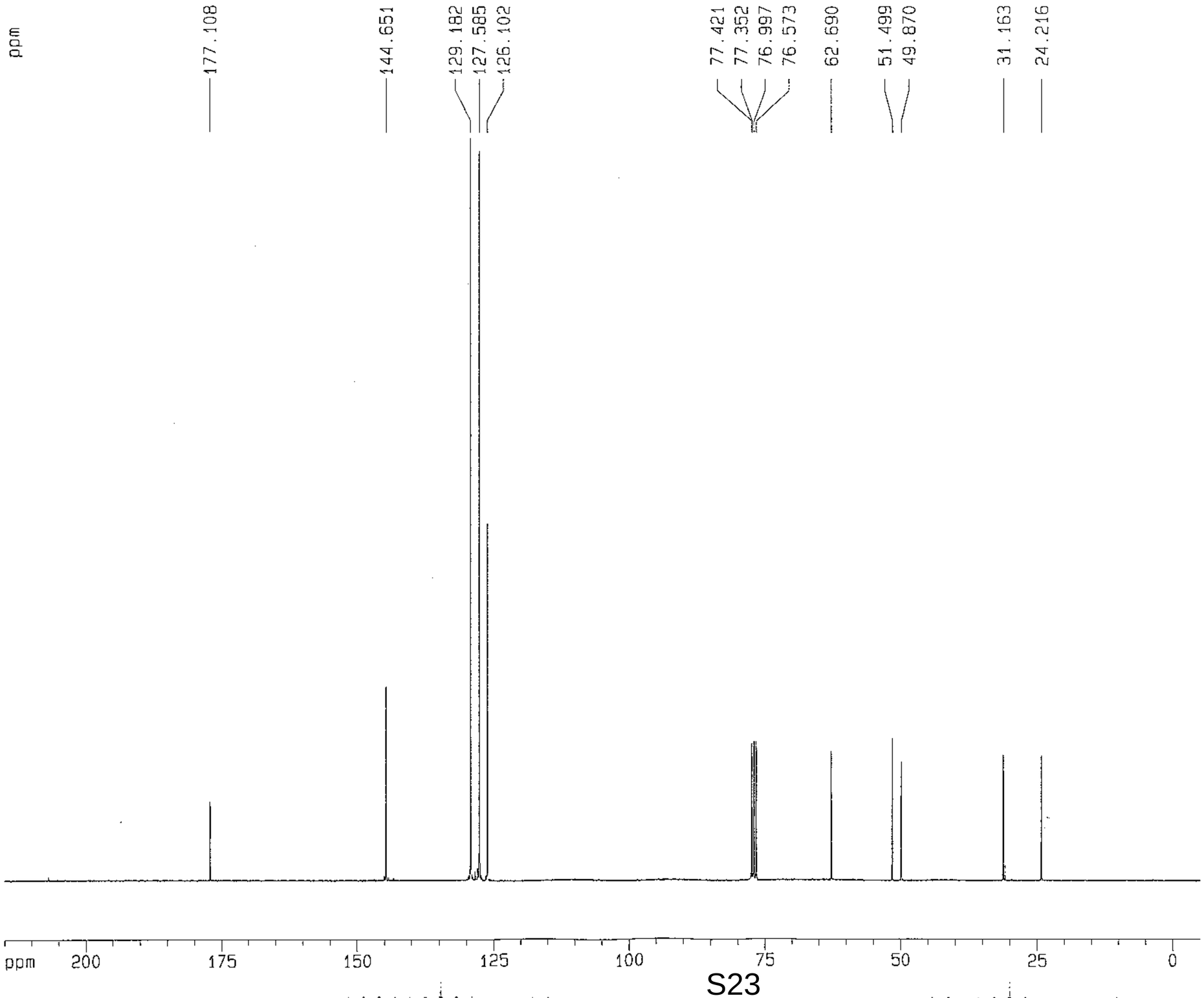
NMR Data of (-)-Secu'amamine A (5) in Deuteriobenzene: ^{1}H NMR (400 MHz, C_6D_6) δ 6.07 (d, *J* = 9.5 Hz, 1H), 5.54 (s, 1H), 5.43 (dd, *J* = 9.5 Hz, 5.6 Hz, 1H), 3.50 (d, *J* = 9.6 Hz, 1H), 3.18 (dt, *J* = 5.5 Hz, 3.0 Hz, 1H), 2.90 (br s, 1H), 2.53 (td, *J* = 8.3 Hz, 3.7 Hz, 1H), 2.38 (dt, *J* = 9.5 Hz, 7.0 Hz, 1H), 2.13 (td, *J* = 8.5 Hz, 7.1 Hz, 1H), 2.05 (dd, *J* = 11.4 Hz, 2.8 Hz, 1H), 1.96-1.88 (m, 1H), 1.65-1.54 (m, 1H), 1.52 (dd, *J* = 11.4 Hz, 3.9 Hz, 1H), 1.49-1.42 (m, 1H), 1.40-1.34 (m, 1H); ^{13}C NMR (75 MHz, C_6D_6) δ 172.31, 161.95, 133.54, 124.15, 114.82, 87.03, 75.42, 60.10, 52.30, 48.82, 37.14, 28.91, 22.77.



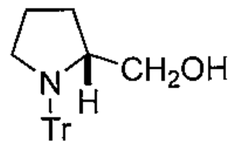

```
Current Data Parameters
NAME                  PL-Jan06-07
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20080106
Time                        21.03
INSTRUM                     spect
PROBHD       5 mm QNP 1H/1
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                             16
DS                              2
SWH                    6172.839 Hz
FIDRES                 0.094190 Hz
AQ                     5.3084660 sec
RG                         101.6
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                     1.00000000 sec

============ CHANNEL f1 =========
NUC1                           1H
P1                         11.70 usec
PL1                         0.00 dB
SFO1                 299.8718518 MHz

F2 - Processing parameters
SI                         32768
SF                   299.8700000 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
F1P                       11.000 ppm
F1                       3298.57 Hz
F2P                      -1.000 ppm
F2                      -299.87 Hz
PPMCM                   0.60000 ppm/cm
HZCM                  179.92200 Hz/cm
```



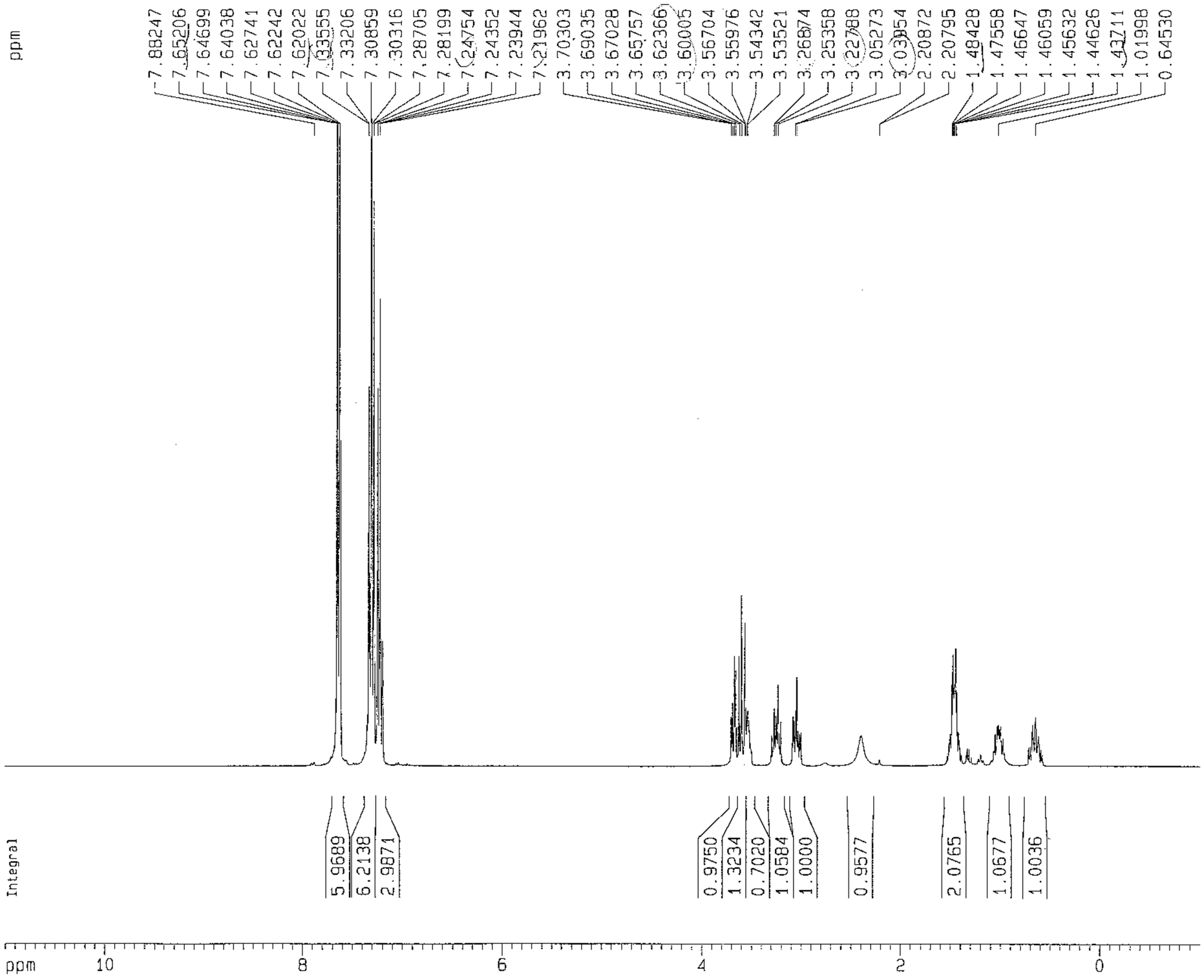
33




```
Current Data Parameters
NAME                  PL-Jan06-07
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20080106
Time                        23.14
INSTRUM                     spect
PROBHD           5 mm QNP 1H/1
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          2053
DS                             4
SWH                  18796.992 Hz
FIDRES                0.286819 Hz
AQ                   1.7433076 sec
RG                          1024
DW                     26.600 usec
DE                      6.00 usec
TE                      300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec

========== CHANNEL f1 ==========
NUC1                         13C
P1                        5.40 usec
PL1                      -6.00 dB
SFO1                75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                   115.00 usec
PL2                       0.00 dB
PL12                     20.00 dB
PL13                     20.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4023803 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                    16211.51 Hz
F2P                      -5.000 ppm
F2                     -377.01 Hz
PPMCM                  11.00000 ppm/cm
HZCM                  829.42621 Hz/cm
```



34



Current Data Parameters

NAME	PL-Jan04-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080104
Time	23.11
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	90.5
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

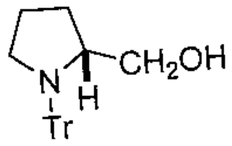
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

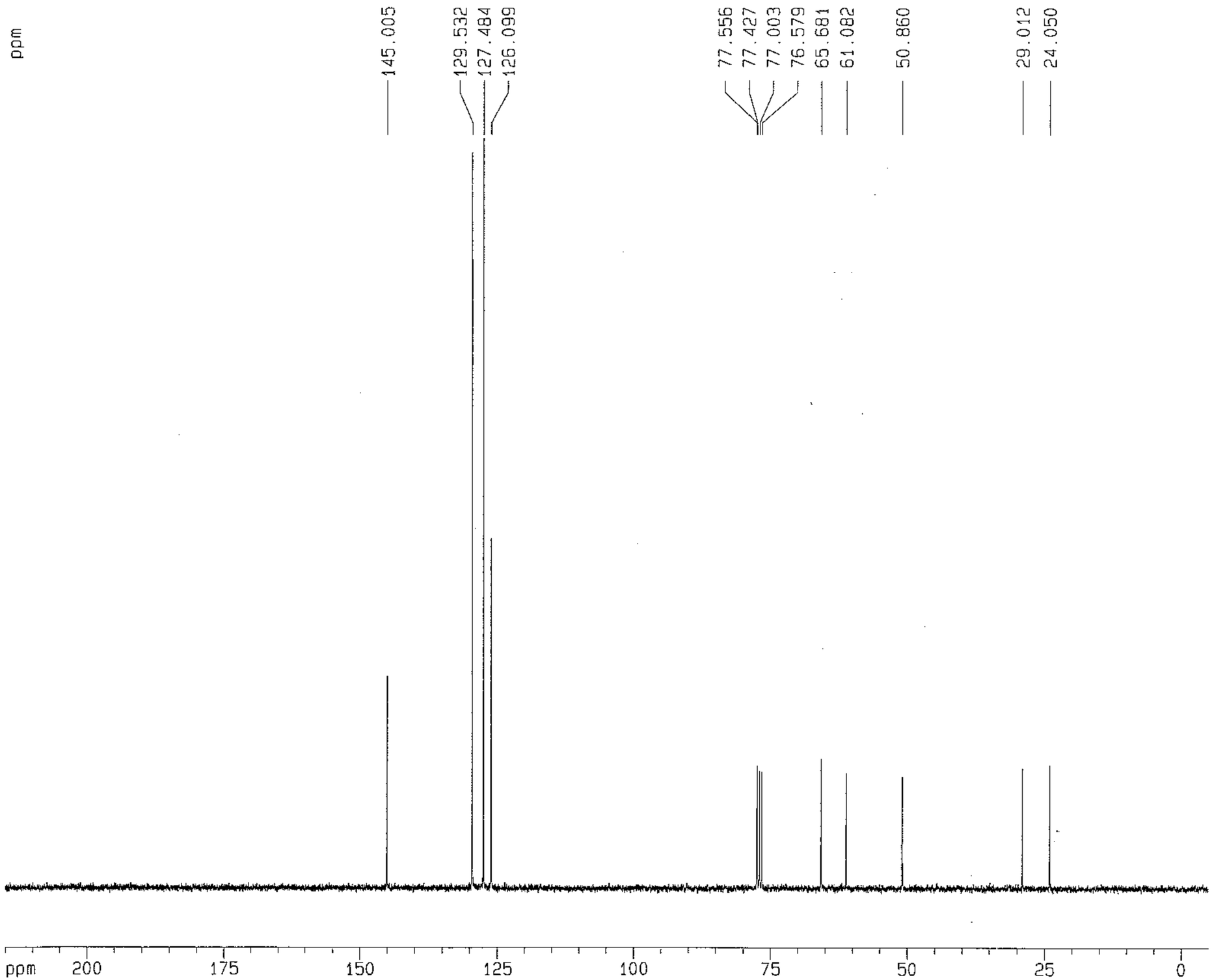
SI	32768
SF	299.8700000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

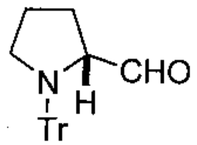
CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92200 Hz/cm



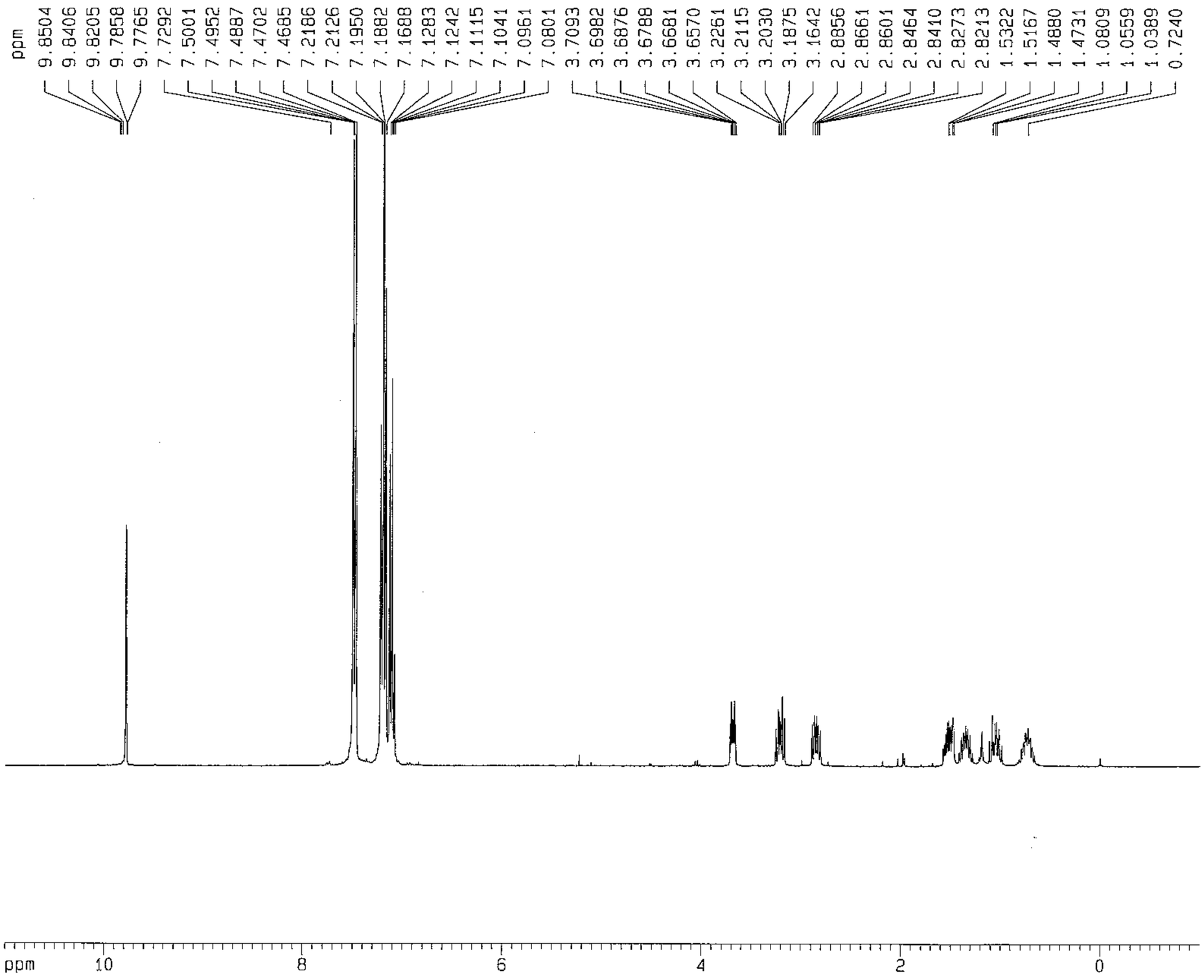
34




```
Current Data Parameters
NAME                 PL-Jan04-07
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20080104
Time                       23.19
INSTRUM                    spect
PROBHD     5 mm QNP 1H/1
PULPROG                   zgpg30
TD                         65536
SOLVENT                   CDCl3
NS                            67
DS                             4
SWH                  18796.992 Hz
FIDRES                0.286819 Hz
AQ                    1.7433076 sec
RG                        574.7
DW                       26.600 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
D12                   0.00002000 sec

============ CHANNEL f1 ============
NUC1                         13C
P1                         5.40 usec
PL1                       -6.00 dB
SFO1                 75.4106357 MHz

============ CHANNEL f2 ============
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    115.00 usec
PL2                        0.00 dB
PL12                      20.00 dB
PL13                      20.00 dB
SFO2                299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                   75.4023809 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
F1P                       215.000 ppm
F1                      16211.51 Hz
F2P                        -5.000 ppm
F2                       -377.01 Hz
PPMCM                   11.00000 ppm/cm
HZCM                   829.42621 Hz/cm
```



11



Current Data Parameters

NAME	Jun08-2006-Wein
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20060608
Time	8.53
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	724.1
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

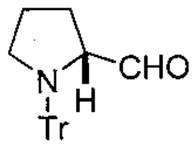
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

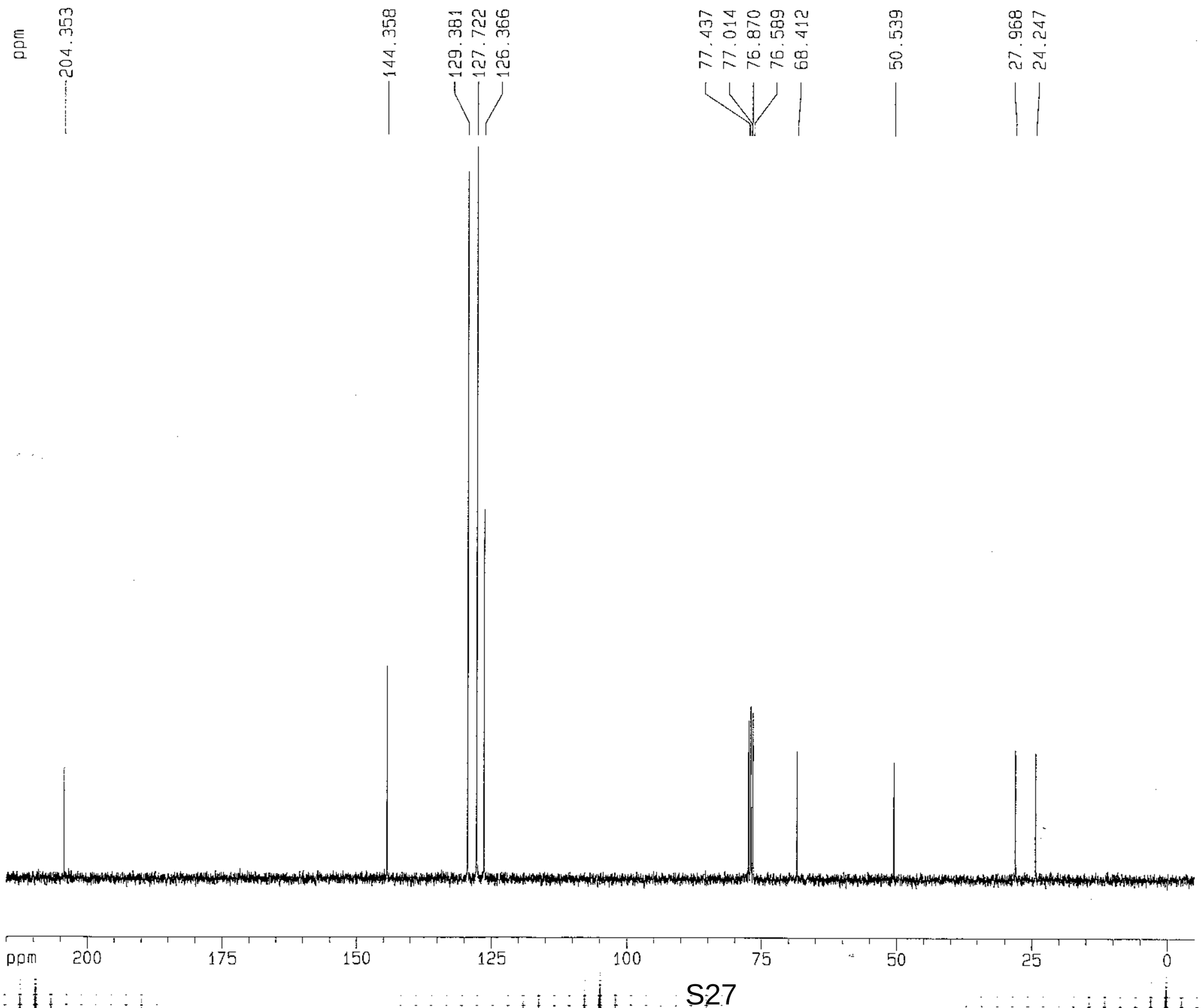
SI	32768
SF	299.8700318 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



11



Current Data Parameters

NAME	PL-Jan10-07
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20080110
Time	14.32
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	47
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1024
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

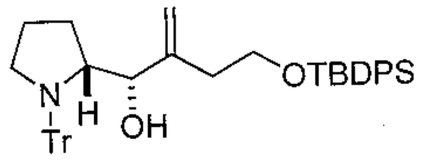
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

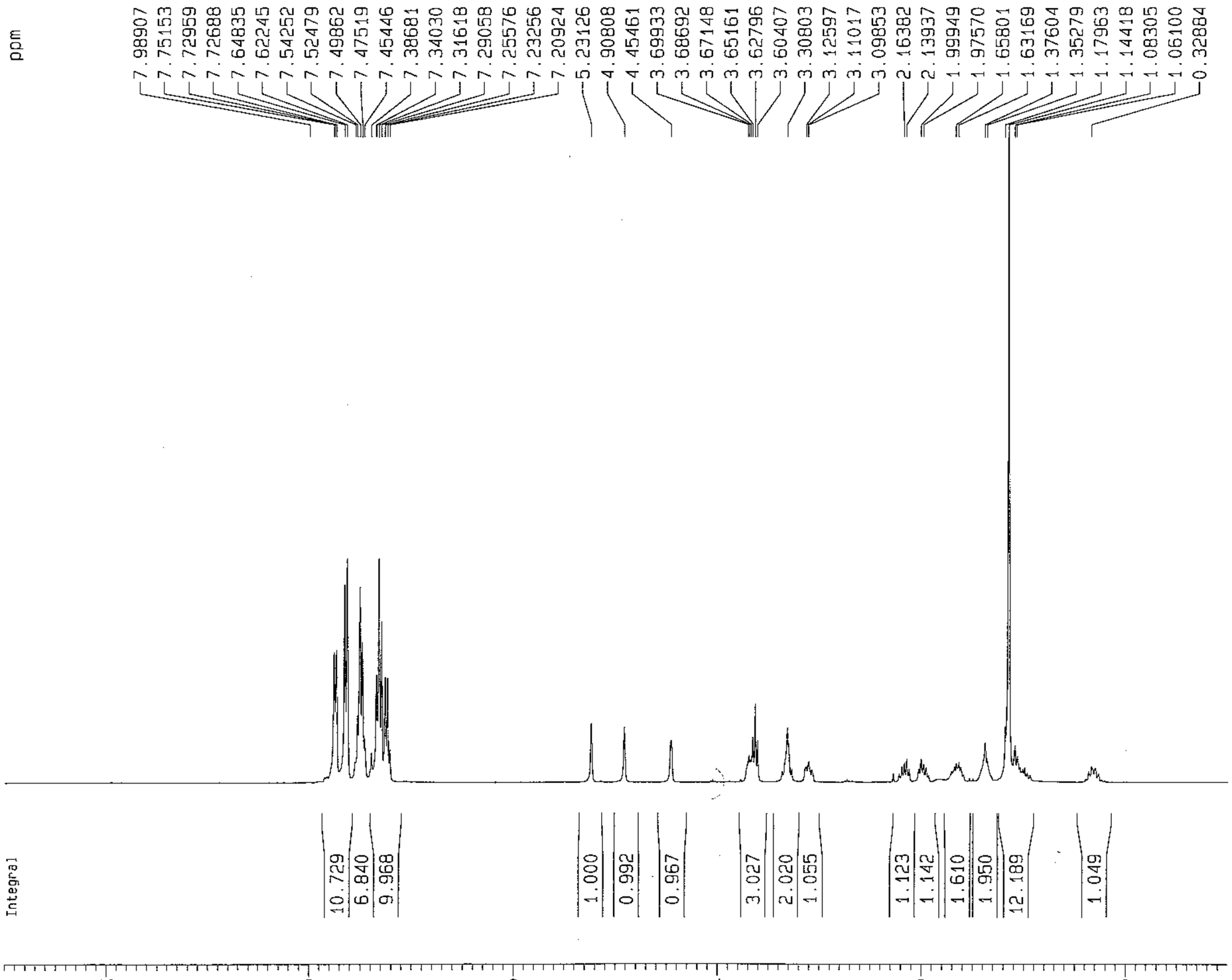
SI	32768
SF	75.4023780 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

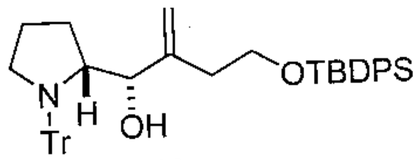
CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



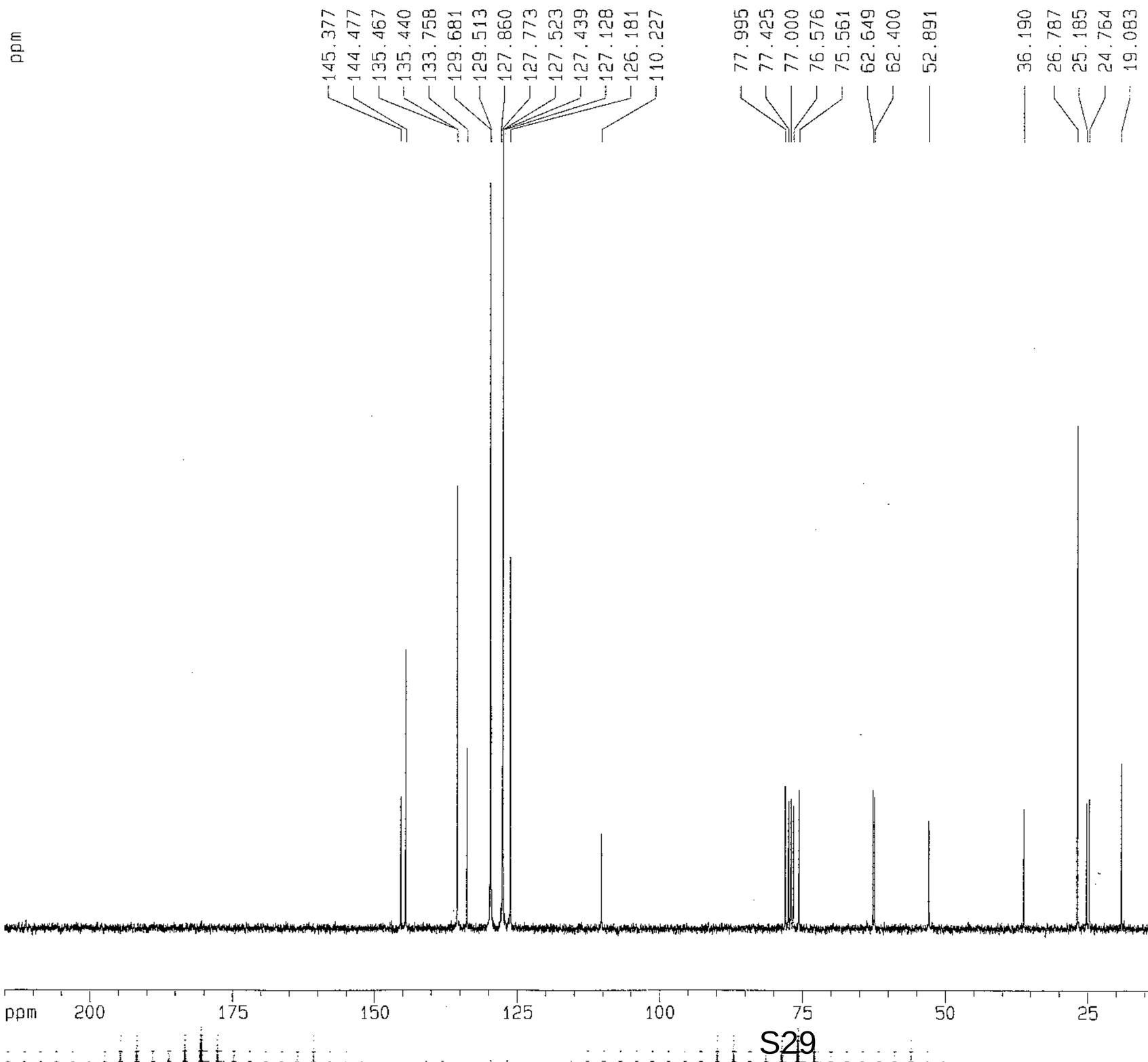
13



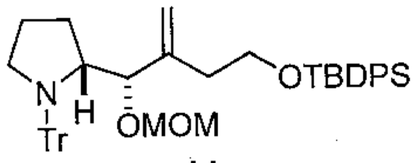

```
Current Data Parameters
NAME        PL-Aug-08-06
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20060808
Time              10.13
INSTRUM           spect
PROBHD    5 mm QNP 1H/1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                71.8
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec

========== CHANNEL f1 =========
NUC1                 1H
P1                11.70 usec
PL1                0.00 dB
SFO1        299.8718518 MHz

F2 - Processing parameters
SI                32768
SF          299.8700000 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P               11.000 ppm
F1                3298.57 Hz
F2P               -1.000 ppm
F2                -299.87 Hz
PPMCM             0.60000 ppm/cm
HZCM            179.92200 Hz/cm
```



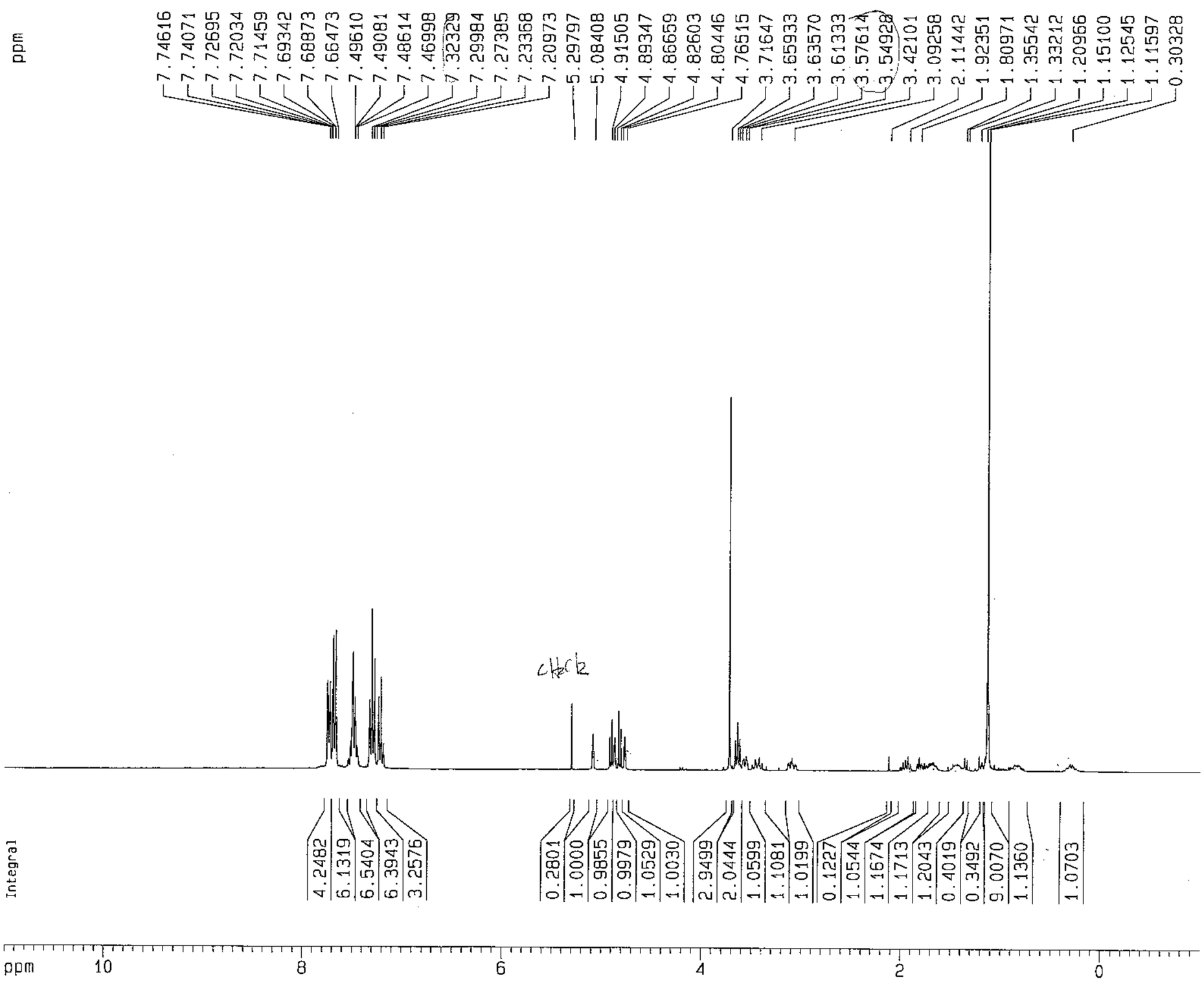
13



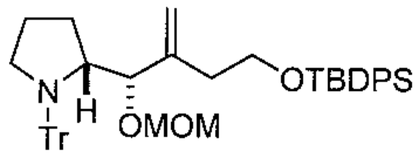

```
Current Data Parameters
NAME                  PL-Jan15-07
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20080115
Time                        21.30
INSTRUM                     spect
PROBHD           5 mm GNP 1H/1
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            62
DS                             4
SWH                  18796.992 Hz
FIDRES                0.286819 Hz
AQ                    1.7433076 sec
RG                          1024
DW                      26.600 usec
DE                       6.00 usec
TE                       300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec

========== CHANNEL f1 ==========
NUC1                         13C
P1                        5.40 usec
PL1                      -6.00 dB
SFO1                75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                   115.00 usec
PL2                       0.00 dB
PL12                     20.00 dB
PL13                     20.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4023918 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      215.000 ppm
F1                     16211.51 Hz
F2P                       -5.000 ppm
F2                      -377.01 Hz
PPMCM                  11.00000 ppm/cm
HZCM                 829.42627 Hz/cm
```



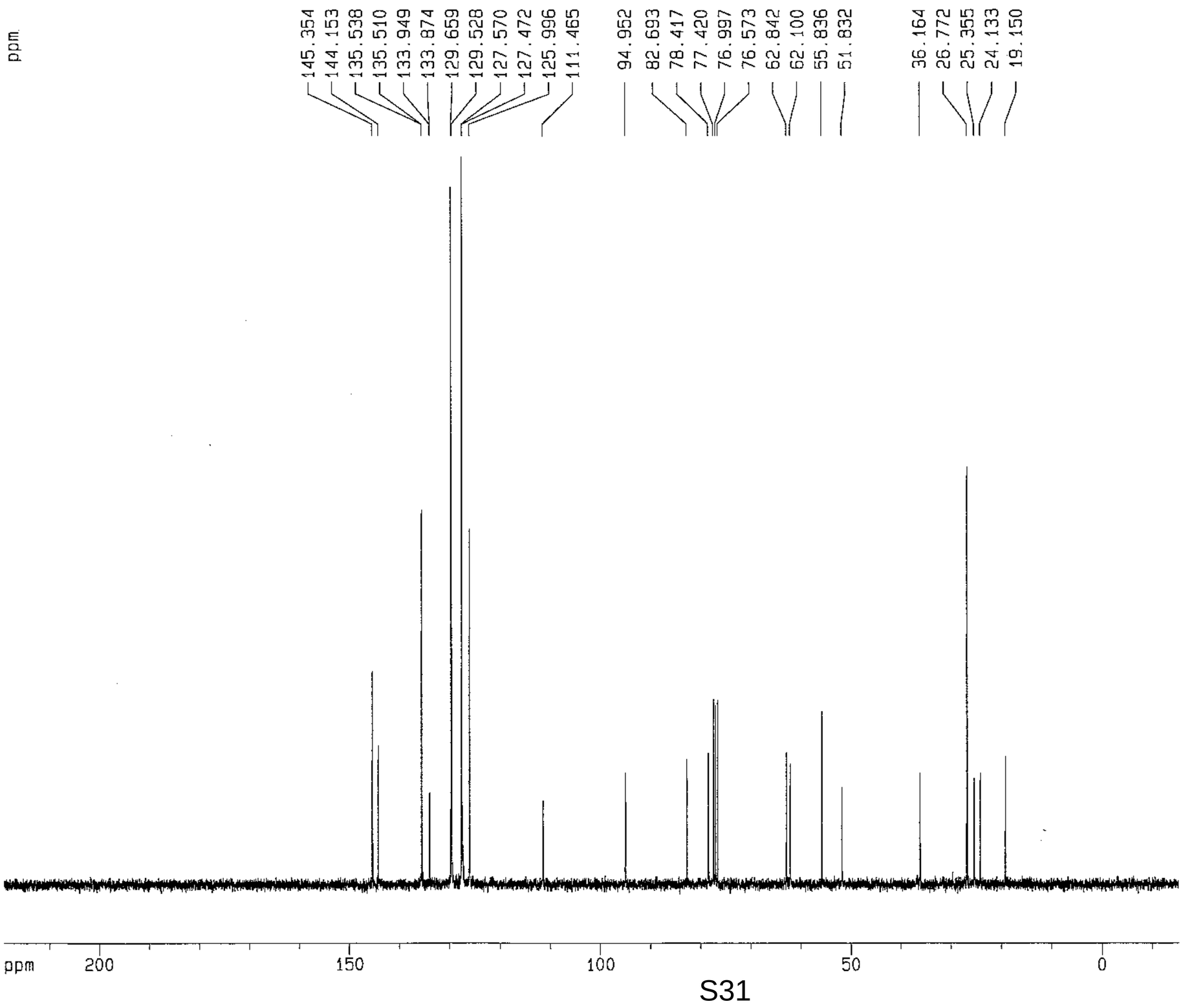
14



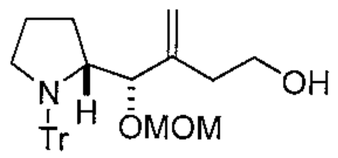

```
Current Data Parameters
NAME                  PL-Jul24-07
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070724
Time                        15.13
INSTRUM                     spect
PROBHD              5 mm Multinu
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                             16
DS                              2
SWH                    6172.839 Hz
FIDRES                 0.094190 Hz
AQ                     5.3084660 sec
RG                          71.8
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                    1.00000000 sec

========== CHANNEL f1 ========
NUC1                          1H
P1                         9.60 usec
PL1                       -6.00 dB
SFO1                300.1318534 MHz

F2 - Processing parameters
SI                         32768
SF                  300.1300000 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
F1P                        11.000 ppm
F1                       3301.43 Hz
F2P                       -1.000 ppm
F2                       -300.13 Hz
PPMCM                    0.60000 ppm/cm
HZCM                   180.07800 Hz/cm
```



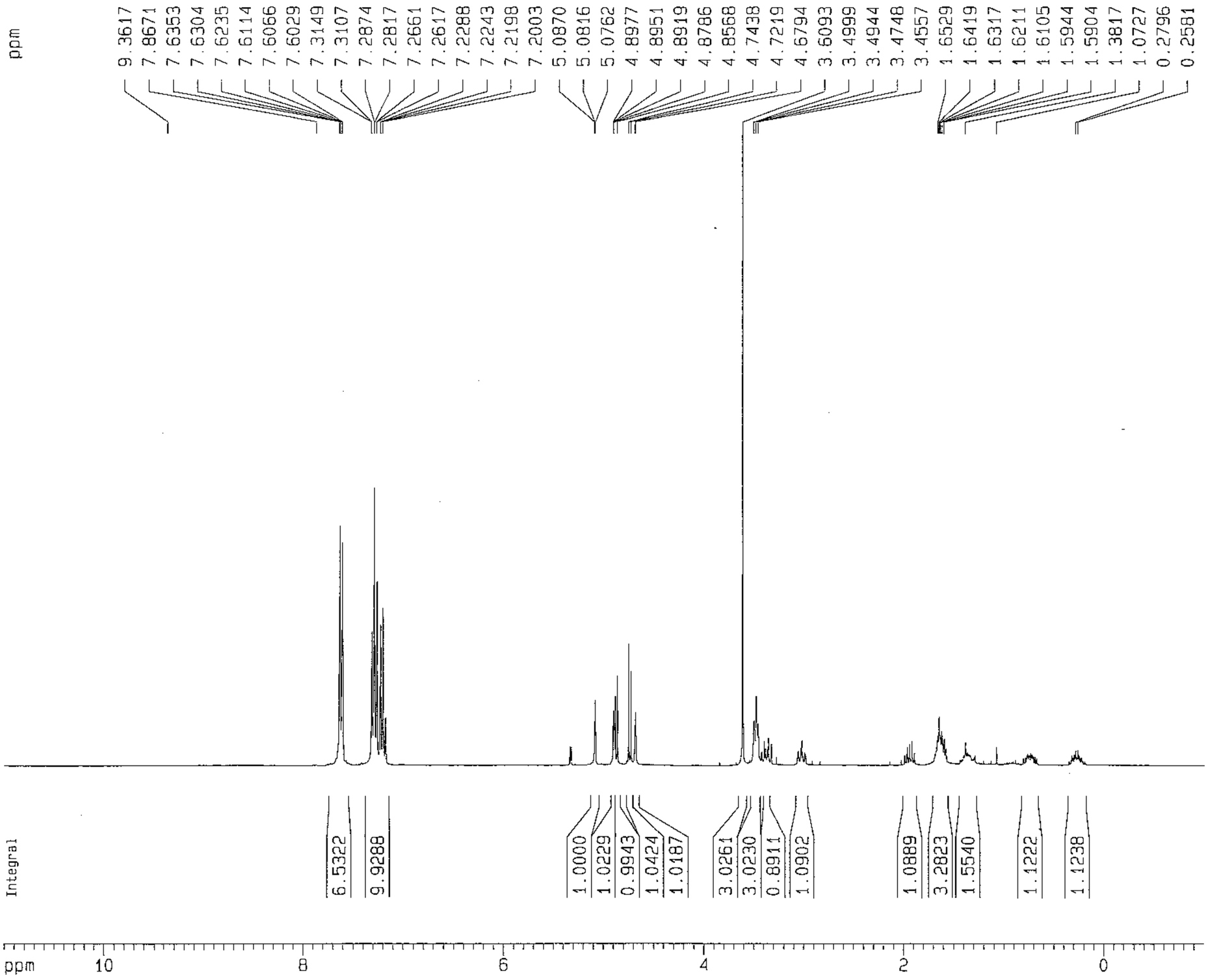
14



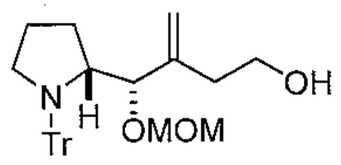

```
Current Data Parameters
NAME          PL-Jul24-07
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_          20070724
Time              15.20
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  103
DS                    4
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AQ             1.7400308 sec
RG                16384
DW               26.550 usec
DE                6.00 usec
TE               300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

========== CHANNEL f1 ==========
NUC1                 13C
P1                11.80 usec
PL1                0.00 dB
SFO1          75.4760200 MHz

========== CHANNEL f2 ==========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             110.00 usec
PL2                0.00 dB
PL12              17.50 dB
PL13              17.50 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF           75.4677583 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
F1P              219.154 ppm
F1             16539.10 Hz
F2P              -19.167 ppm
F2             -1446.52 Hz
PPMCM          11.91610 ppm/cm
HZCM          899.28101 Hz/cm
```



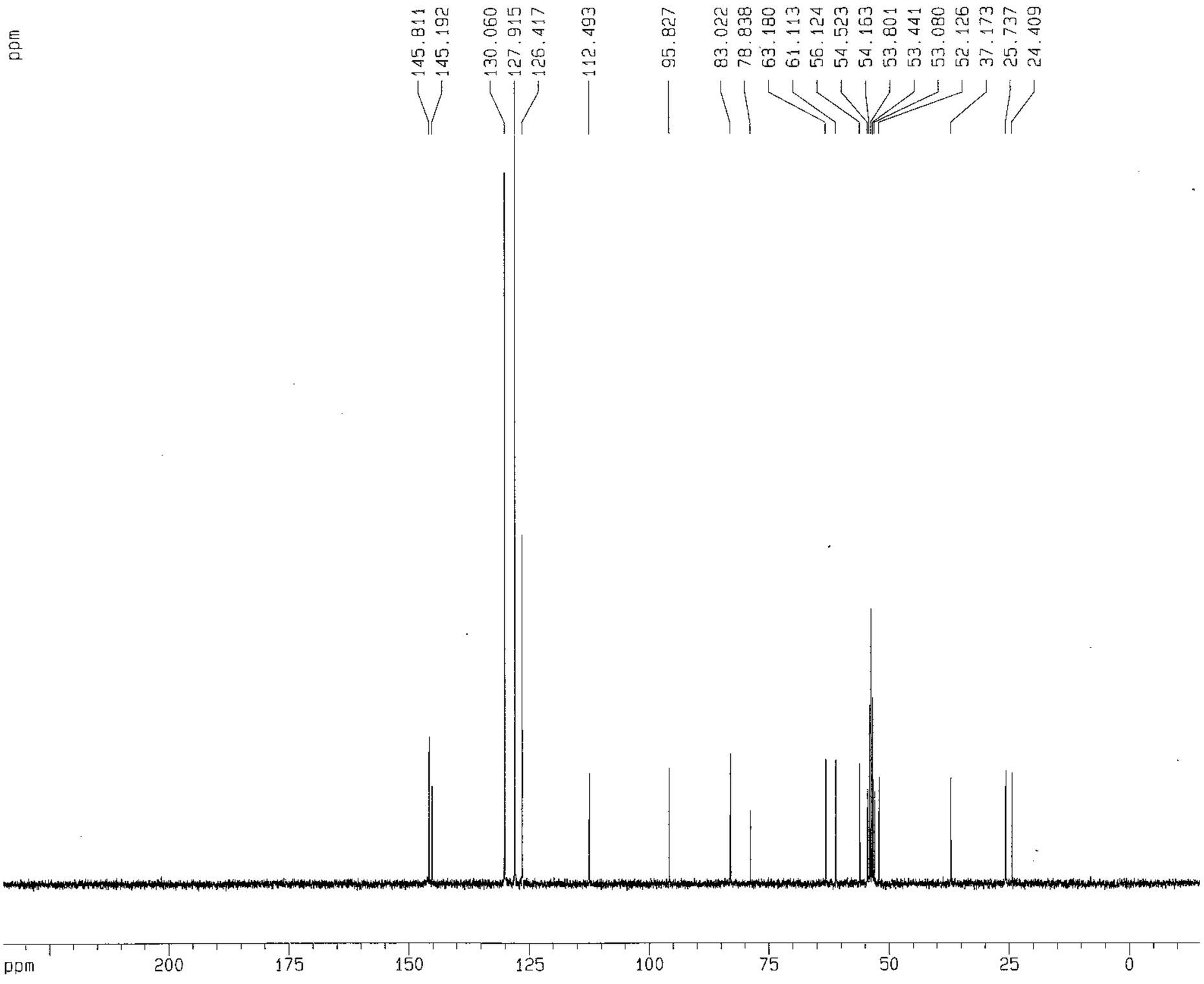
35




```
Current Data Parameters
NAME                  PL-Jul21-07
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070721
Time                        17.35
INSTRUM                     spect
PROBHD           5 mm QNP 1H/1
PULPROG                     zg30
TD                         65536
SOLVENT                    CD2Cl2
NS                             16
DS                              2
SWH                   6172.839 Hz
FIDRES                0.094190 Hz
AQ                    5.3084660 sec
RG                            114
DW                     81.000 usec
DE                      6.00 usec
TE                      300.0 K
D1                    1.00000000 sec

============ CHANNEL f1 ========
NUC1                           1H
P1                     11.70 usec
PL1                     0.00 dB
SFO1                299.8718518 MHz

F2 - Processing parameters
SI                         32768
SF                  299.8700143 MHz
WDW                           no
SSB                            0
LB                      0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                   11.000 ppm
F1                   3298.57 Hz
F2P                  -1.000 ppm
F2                   -299.87 Hz
PPMCM                 0.60000 ppm/cm
HZCM                179.92201 Hz/cm
```



35



Current Data Parameters

NAME	PL-Jul21-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20070721
Time	17.55
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	162
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	362
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

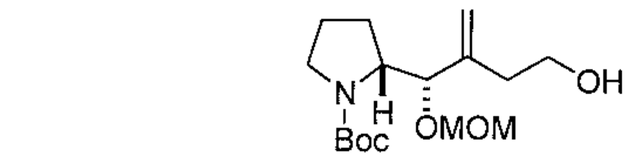
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

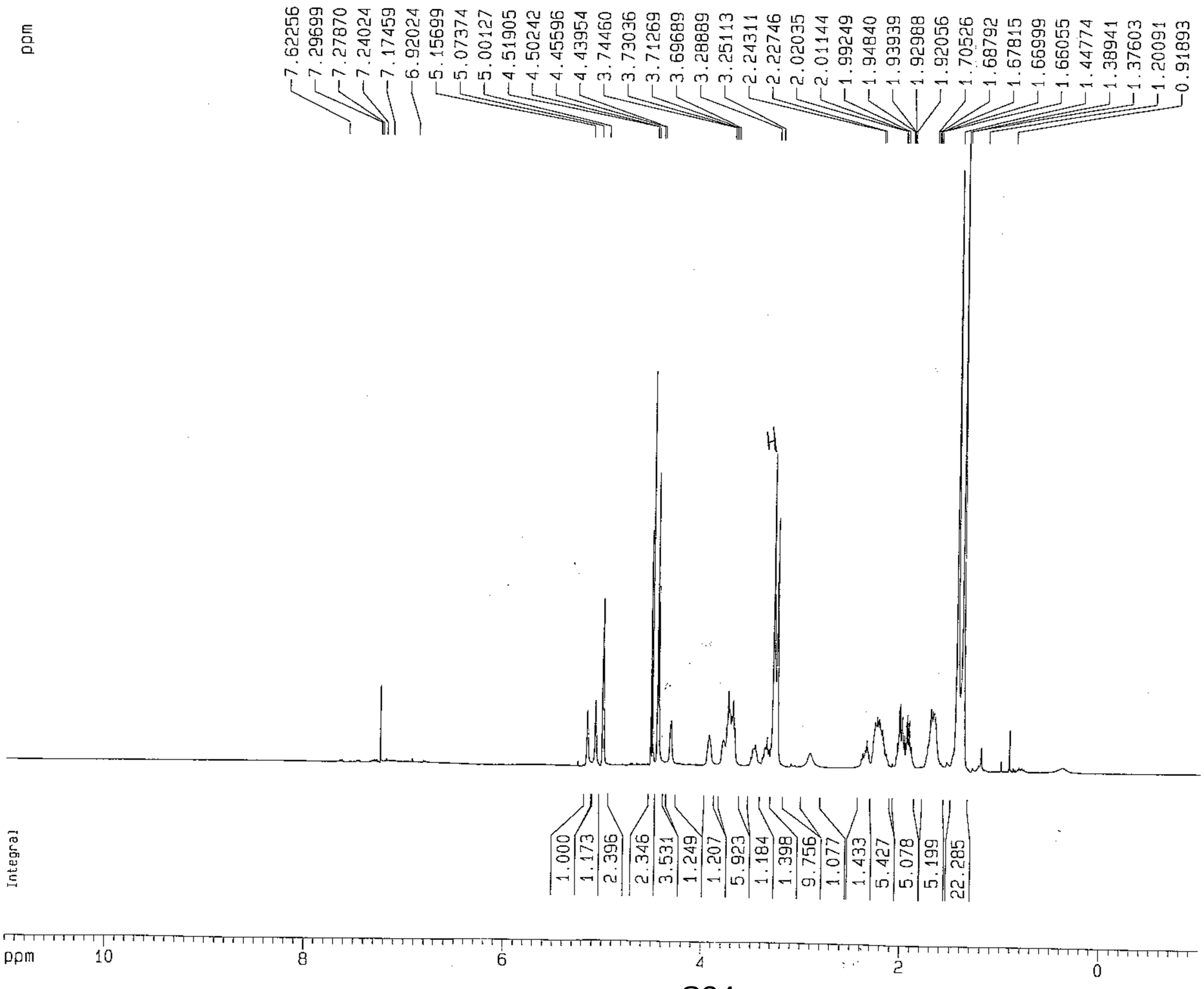
SI	32768
SF	75.4023468 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	234.574 ppm
F1	17687.44 Hz
F2P	-14.715 ppm
F2	-1109.55 Hz
PPMCM	12.46446 ppm/cm
HZCM	939.84955 Hz/cm



15



Current Data Parameters

NAME	PL-Jul27-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20070727
Time	15.19
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	143.7
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

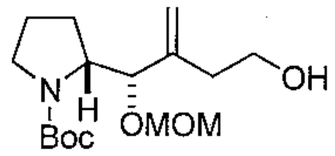
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

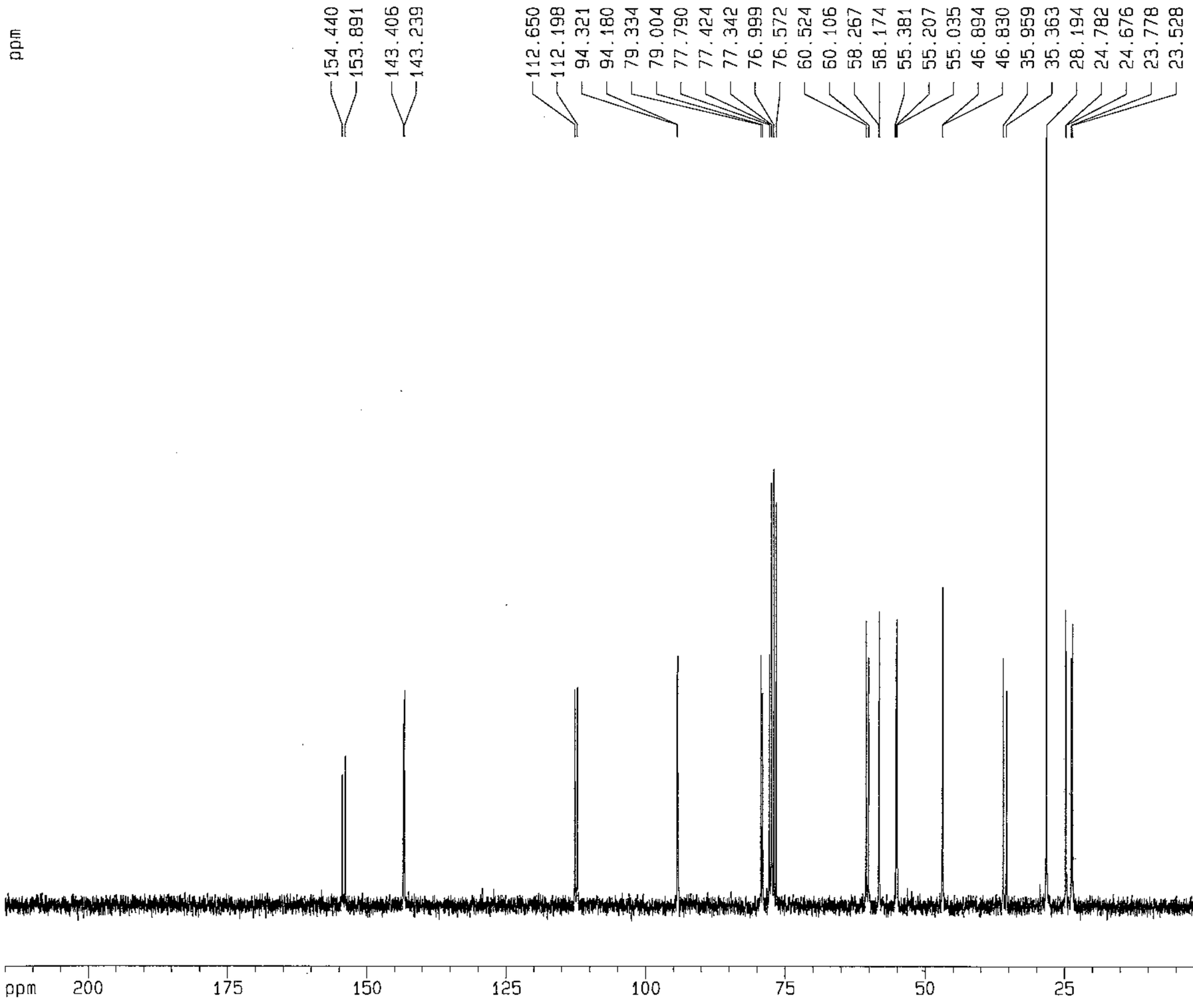
SI	32768
SF	400.1300166 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm

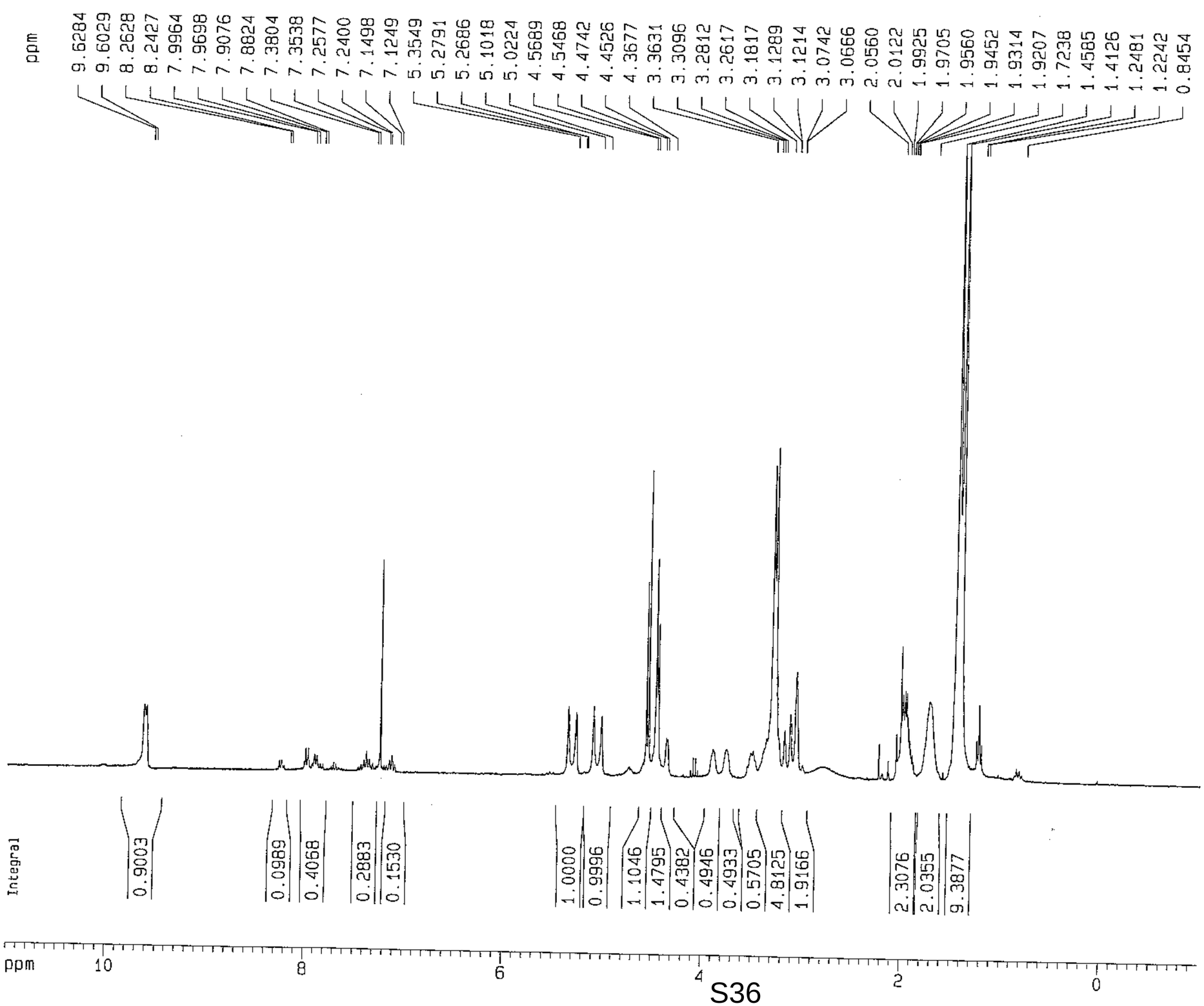


15




```
Current Data Parameters
NAME                  PL-Sep10-07
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070910
Time                        10.40
INSTRUM                     spect
PROBHD           5 mm QNP 1H/1
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            112
DS                              4
SWH                   18796.992 Hz
FIDRES                 0.286819 Hz
AQ                    1.7433076 sec
RG                           2048
DW                         26.600 usec
DE                          6.00 usec
TE                         300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
D12                   0.00002000 sec

============ CHANNEL f1 ============
NUC1                          13C
P1                           5.40 usec
PL1                         -6.00 dB
SFO1                 75.4106357 MHz

============ CHANNEL f2 ============
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      115.00 usec
PL2                          0.00 dB
PL12                        20.00 dB
PL13                        20.00 dB
SFO2                299.8711995 MHz

F2 - Processing parameters
SI                            32768
SF                   75.4023878 MHz
WDW                              EM
SSB                               0
LB                             1.00 Hz
GB                                0
PC                             1.40

1D NMR plot parameters
CX                            20.00 cm
F1P                          215.000 ppm
F1                         16211.51 Hz
F2P                           -5.000 ppm
F2                          -377.01 Hz
PPMCM                     11.00000 ppm/cm
HZCM                     829.42627 Hz/cm
```

16



Current Data Parameters

NAME	PL-Sep28-07
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20070928
Time	23.16
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	181
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

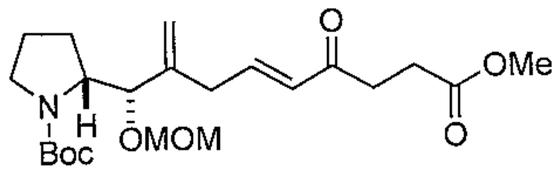
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

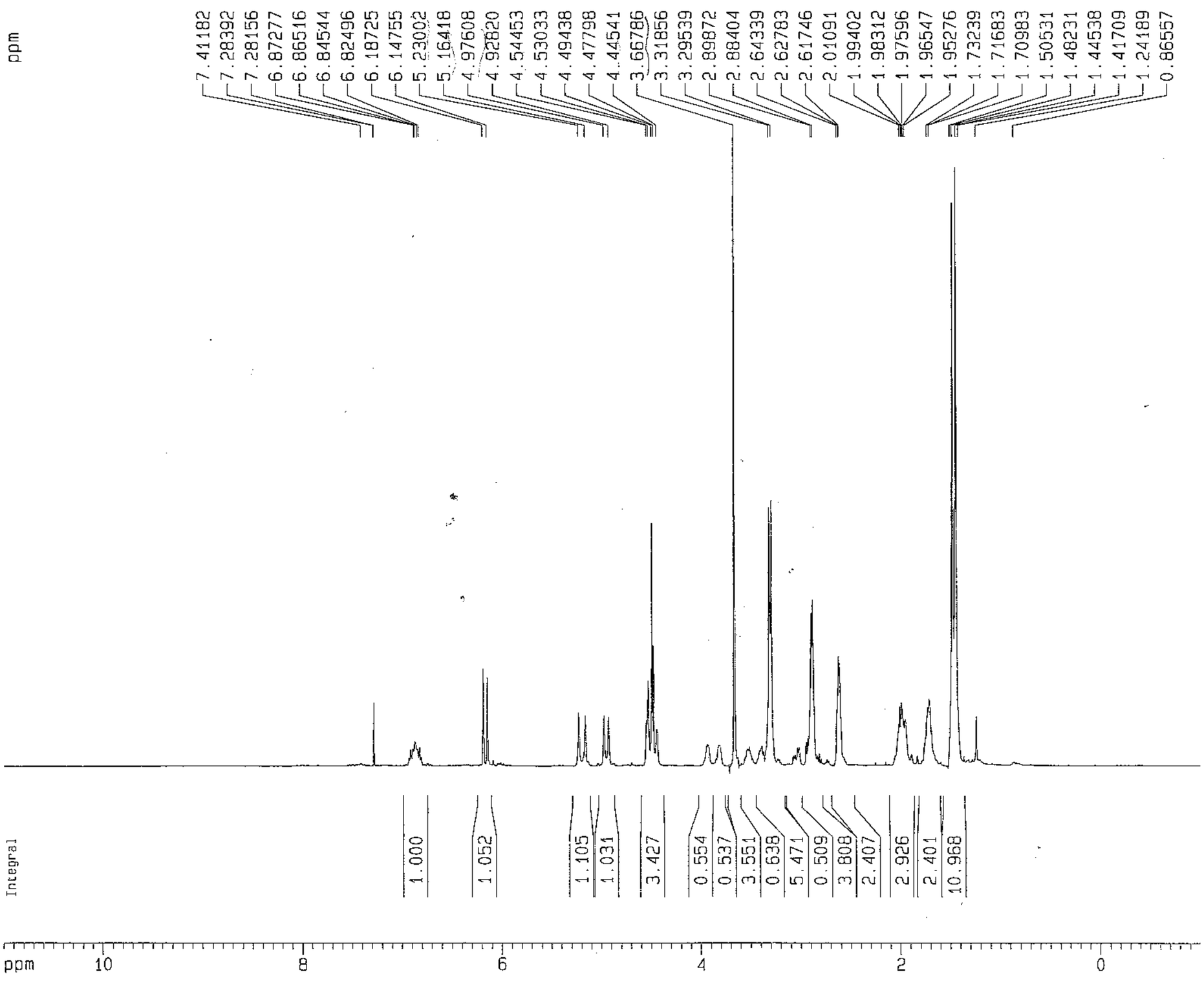
SI	32768
SF	299.8700161 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



18



Current Data Parameters

NAME	PL-Aug21-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20070822
Time	0.00
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	45.3
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

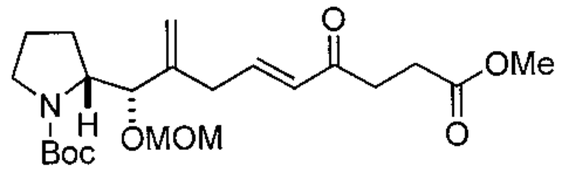
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

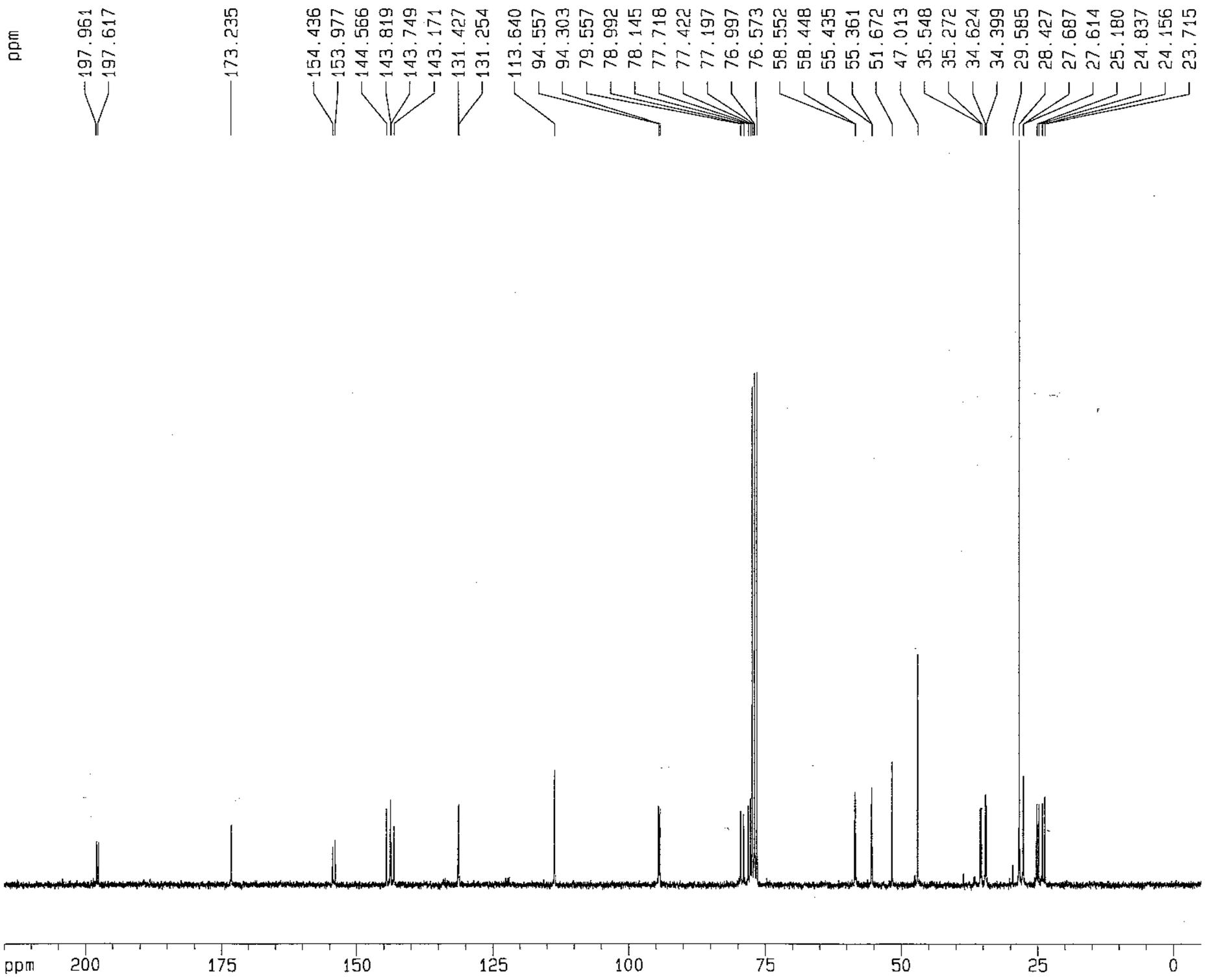
SI	32768
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

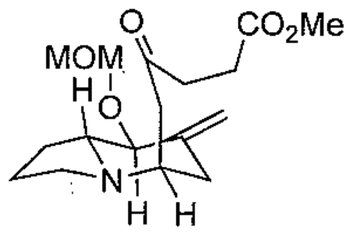
CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm



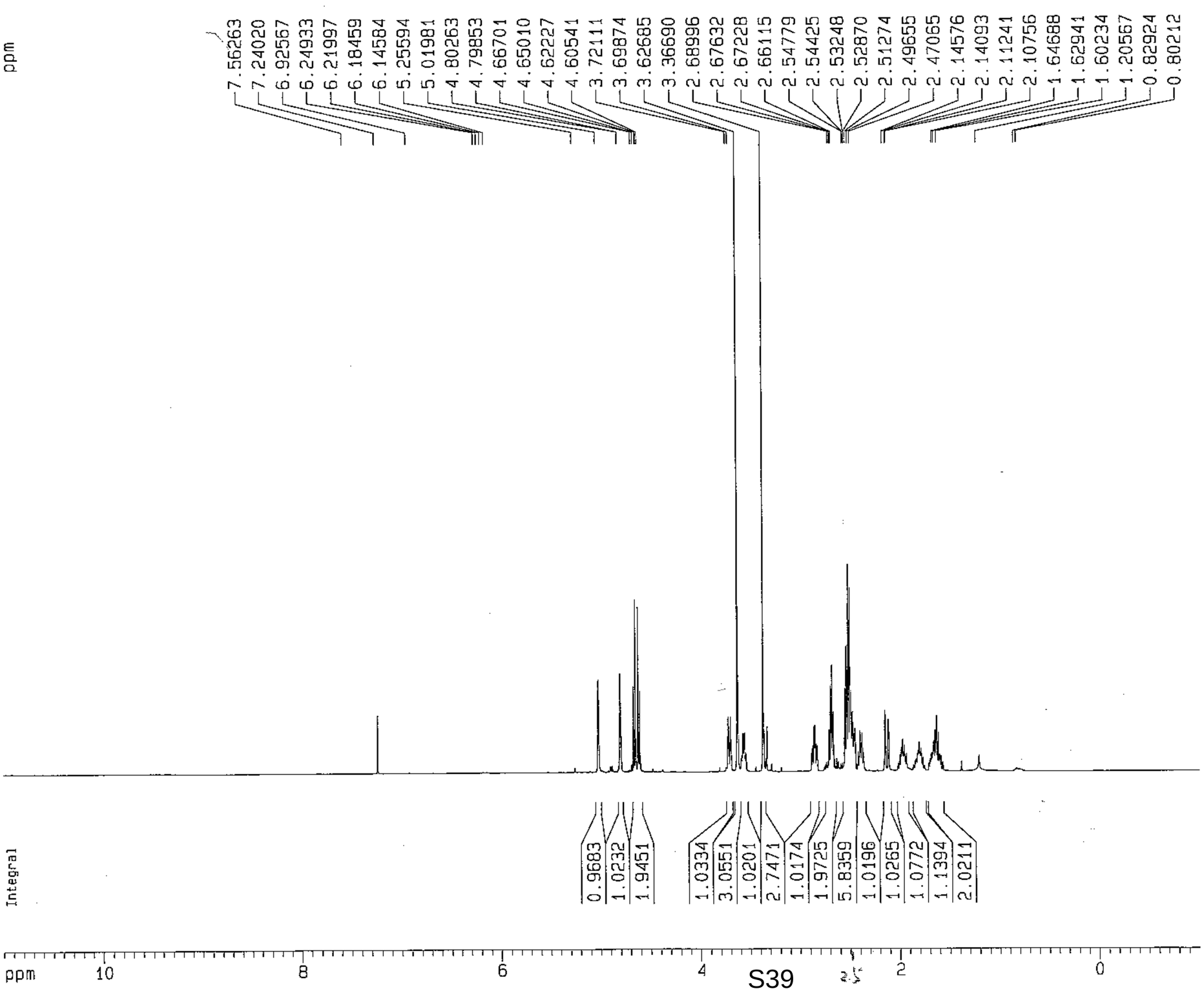
18




```
Current Data Parameters
NAME          PL-Aug23-07
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070823
Time               16.39
INSTRUM            spect
PROBHD  5 mm QNP 1H/1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1400
DS                    4
SWH          18796.992 Hz
FIDRES       0.286819 Hz
AQ           1.7433076 sec
RG                 1024
DW              26.600 usec
DE               6.00 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1               5.40 usec
PL1             -6.00 dB
SFO1        75.4106357 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2          115.00 usec
PL2              0.00 dB
PL12            20.00 dB
PL13            20.00 dB
SFO2       299.8711995 MHz

F2 - Processing parameters
SI                 32768
SF          75.4023792 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               215.000 ppm
F1              16211.51 Hz
F2P               -5.000 ppm
F2              -377.01 Hz
PPMCM             11.00000 ppm/cm
HZCM            829.42621 Hz/cm
```



24



Current Data Parameters

NAME	PL-Feb05-08
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20080205
Time	12.55
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
AQ	3.9584243 sec
RG	28.5
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

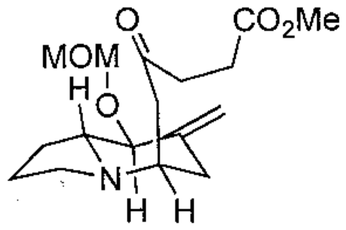
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300174 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

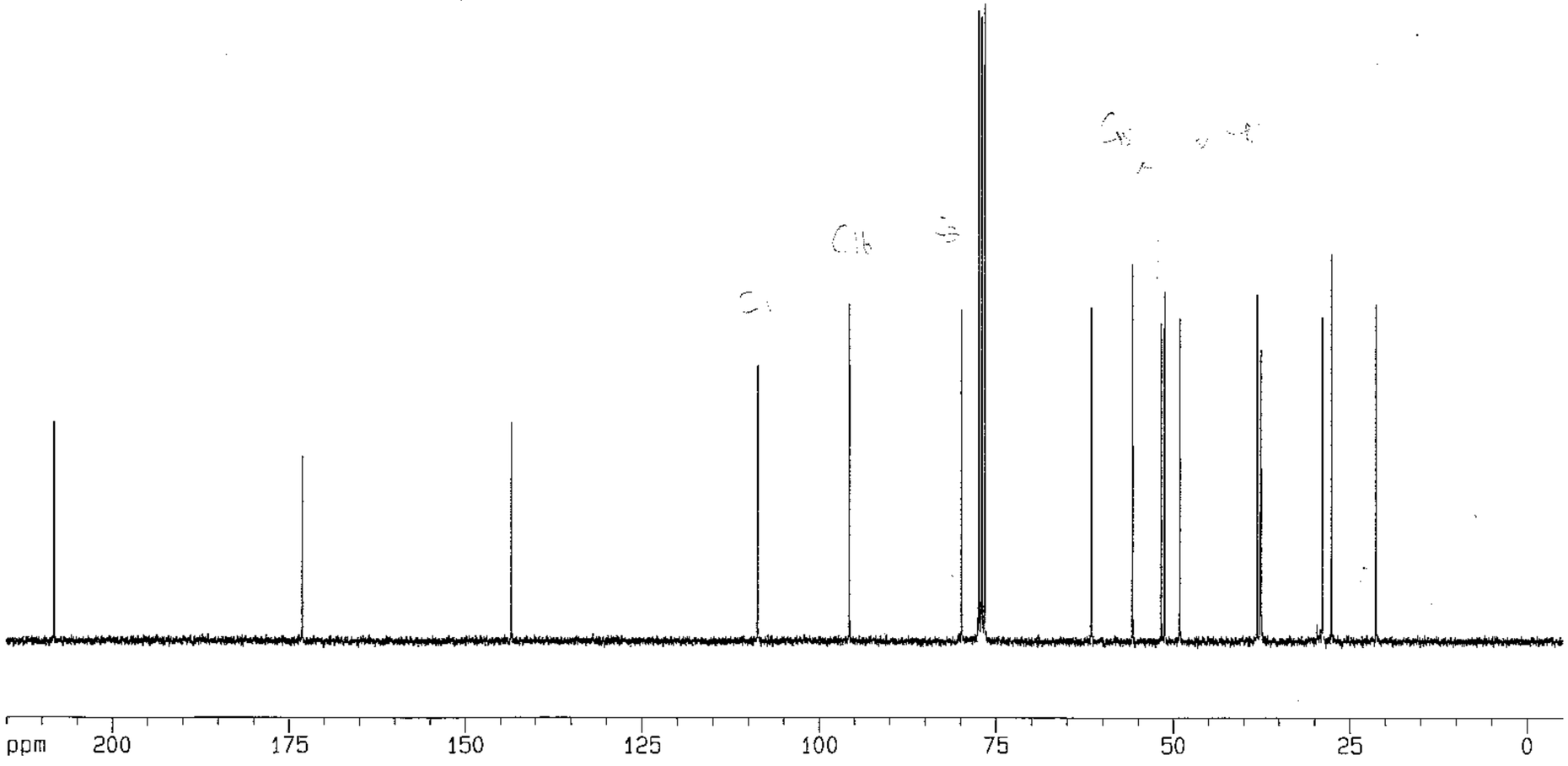
1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm

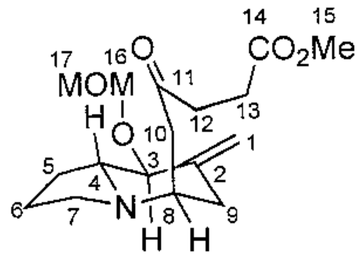


24

ppm
208.334
173.115
143.467
108.657
95.731
79.861
77.428
77.004
76.580
61.588
55.789
51.740
51.287
49.103
38.126
37.632
37.523
28.889
27.551
21.312

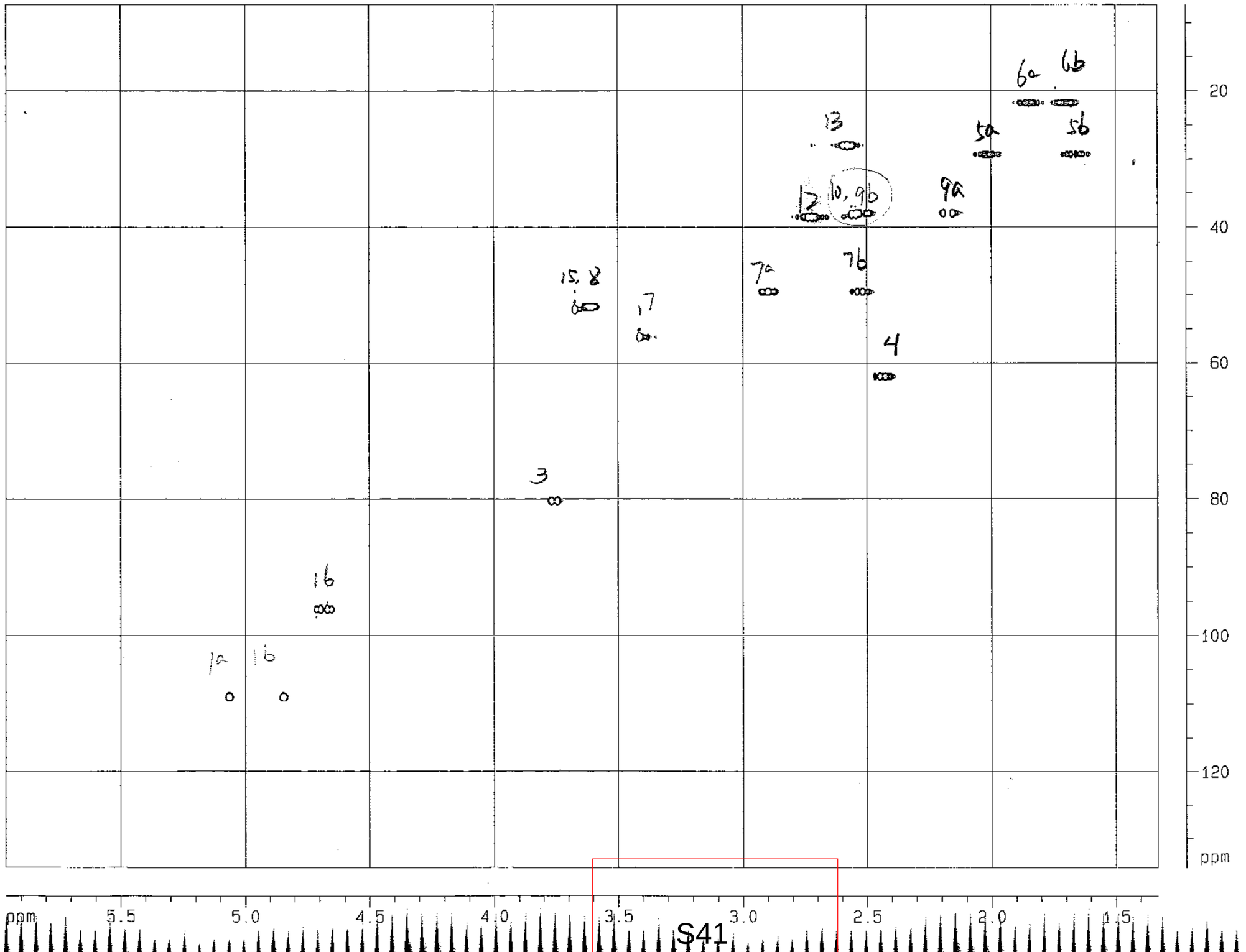



```
Current Data Parameters
NAME          PL-Oct07-07
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_          20071008
Time               0.01
INSTRUM           spect
PROBHD  5 mm QNP 1H/1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1113
DS                    4
SWH          18796.992 Hz
FIDRES        0.286819 Hz
AQ            1.7433076 sec
RG                 2048
DW               26.600 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

========== CHANNEL f1 ==========
NUC1                13C
P1                5.40 usec
PL1              -6.00 dB
SFO1        75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2          115.00 usec
PL2              0.00 dB
PL12            20.00 dB
PL13            20.00 dB
SFO2       299.8711995 MHz

F2 - Processing parameters
SI                32768
SF         75.4023769 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX              20.00 cm
F1P            215.000 ppm
F1           16211.51 Hz
F2P             -5.000 ppm
F2            -377.01 Hz
PPMCM         11.00000 ppm/cm
HZCM         829.42615 Hz/cm
```



24

HMQC



Current Data Parameters

NAME	PL-Oct08-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20071008
Time	18.18
INSTRUM	spect
PROBHD	5 mm 681 1H-B
PULPROG	inv4gptp
TD	2048
SOLVENT	CDCl3
NS	4
DS	80
SWH	2626.545 Hz
FIDRES	1.279563 Hz
AQ	0.3898064 sec
RG	7299.2
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.98631298 sec
d2	0.00344828 sec
d4	0.00172414 sec
d11	0.03000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
d20	0.00052414 sec
d21	0.00224428 sec
INO	0.00001490 sec

CHANNEL f1

NUC1	1H
P1	7.10 usec
p2	14.20 usec
PL1	0.00 dB
SFO1	400.1317545 MHz

CHANNEL f2

CPDPRG2	garp
NUC2	13C
P3	17.00 usec
p4	34.00 usec
PCPD2	64.00 usec
PL2	-6.00 dB
PL12	6.30 dB
SFO2	100.6202332 MHz

GRADIENT CHANNEL

GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPX1	17.00 %
GPX2	20.00 %
GPX3	25.00 %
GPY1	17.00 %
GPY2	20.00 %
GPY3	25.00 %
GPZ1	17.00 %
GPZ2	20.00 %
GPZ3	25.00 %
P16	1000.00 usec

F1 - Acquisition parameters

ND0	4
TD	256
SFO1	100.6202 MHz
FIDRES	65.541107 Hz
SW	166.751 ppm

F2 - Processing parameters

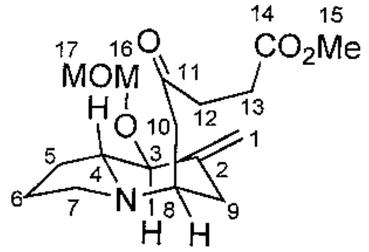
SI	2048
SF	400.1300000 MHz
WDW	GSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	1024
MC2	TPPI
SF	100.6127290 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

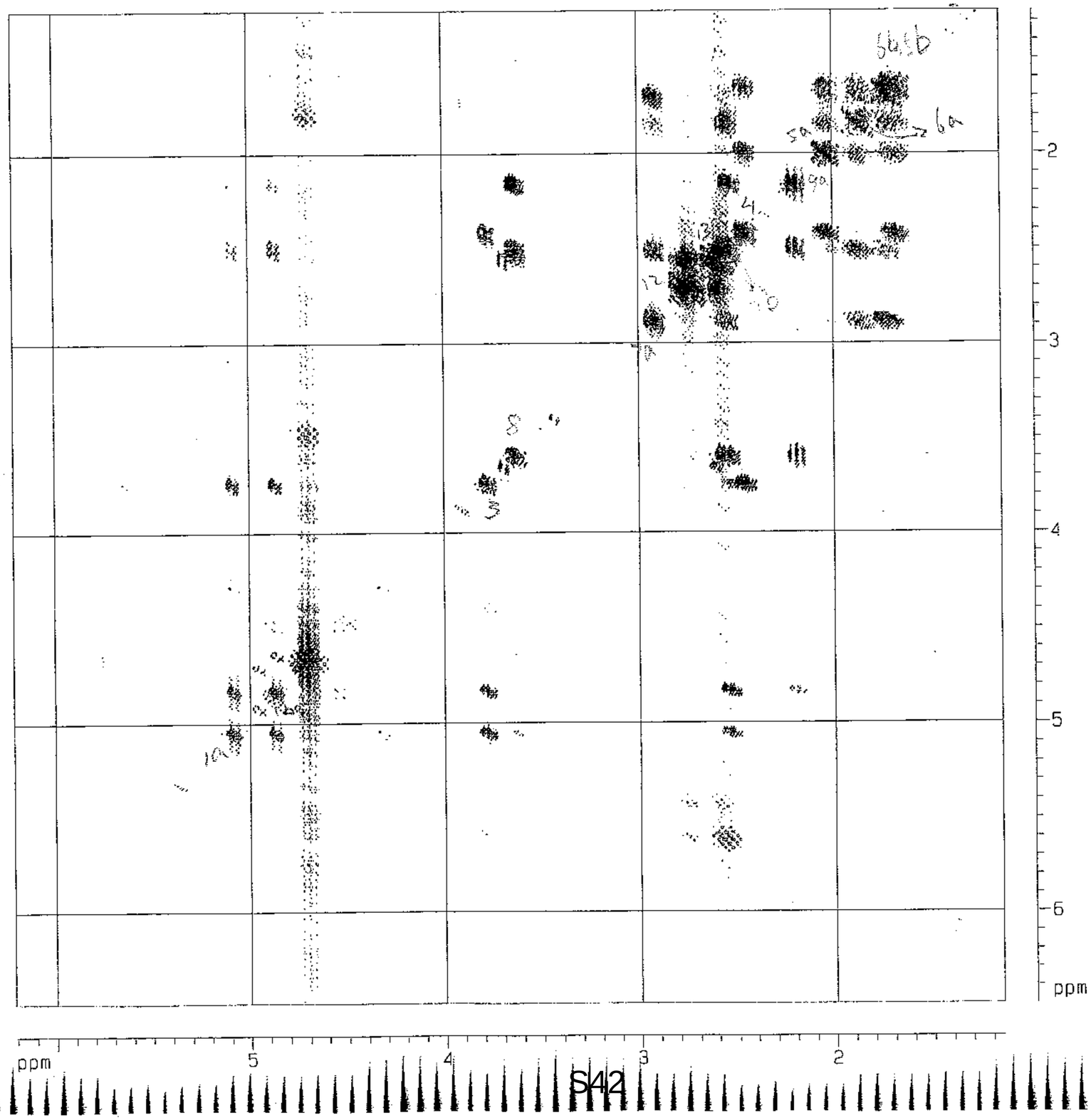
2D NMR plot parameters

CX2	20.00 cm
CX1	15.00 cm
F2PLO	5.562 ppm
F2LO	2365.39 Hz
F2PHI	1.331 ppm
F2HI	538.55 Hz
F1PLO	134.353 ppm
F1LO	13517.58 Hz
F1PHI	7.326 ppm
F1HI	737.07 Hz
F2PPMCM	0.23153 ppm/cm
F2HZCM	92.64037 Hz/cm
F1PPMCM	6.46646 ppm/cm
F1HZCM	92.03412 Hz/cm



24

COSY



Current Data Parameters

NAME	PL-Oct08-07
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20071008
Time	21.16
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	ejb.cosygsmftp
TD	2048
SOLVENT	CDCl3
NS	4
DS	6
SWH	2620.545 Hz
FIDRES	1.279563 Hz
AQ	0.3908084 sec
RG	5792.6
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
D0	0.00000300 sec
D1	4.00000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
d20	0.00120300 sec
IN0	0.00015080 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	0.00 dB
SFO1	400.1317545 MHz

========== GRADIENT CHANNEL =======

P16	1000.00 usec

F1 - Acquisition parameters

NDO	2
TD	650
SFO1	400.1318 MHz
FIDRES	4.031608 Hz
SW	6.549 ppm

F2 - Processing parameters

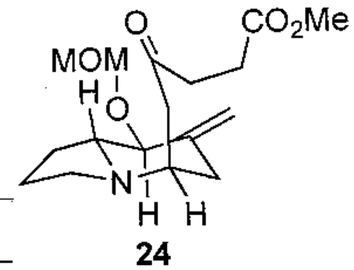
SI	2048
SF	400.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	1024
MC2	TPPI
SF	400.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

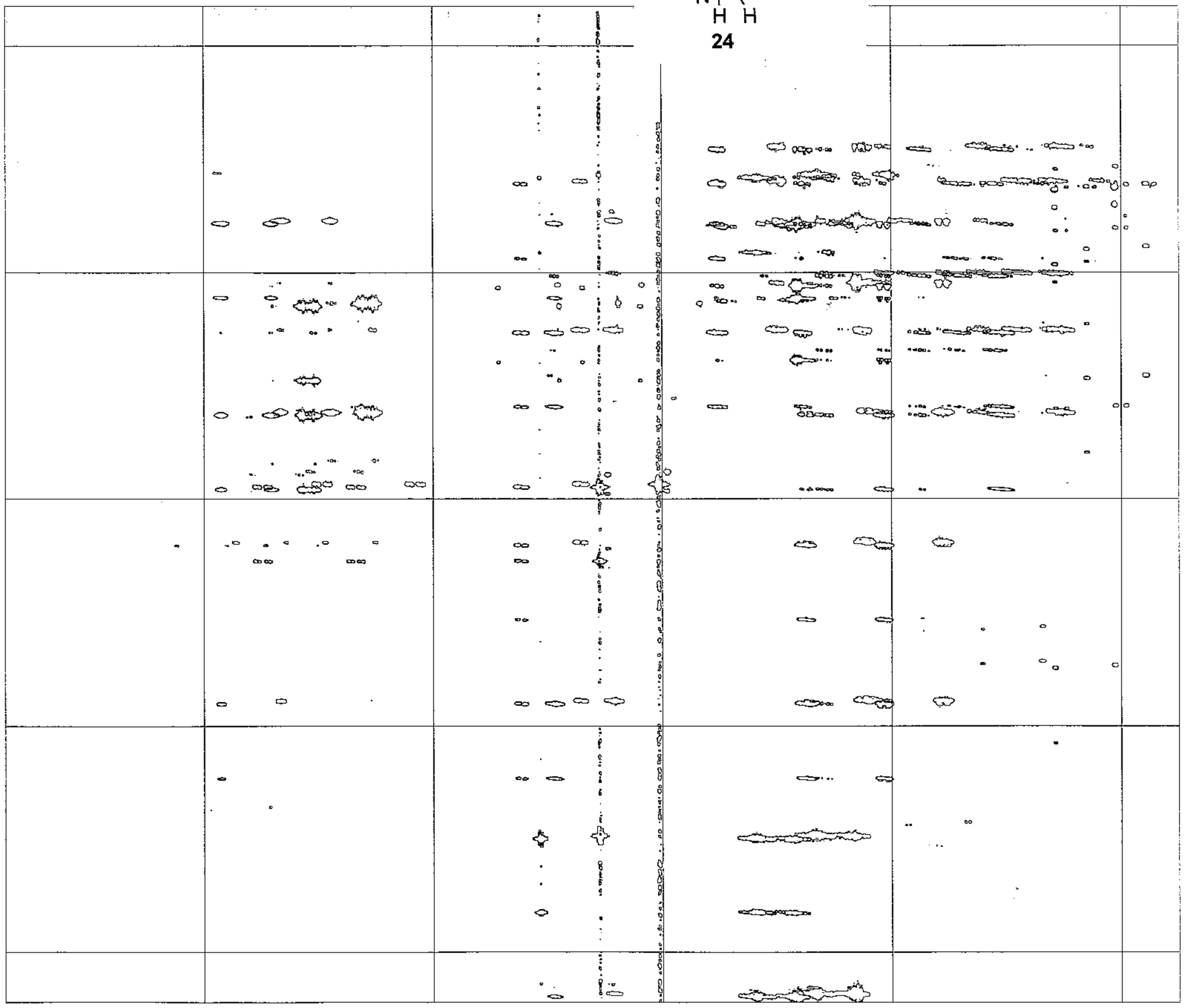
2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	6.211 ppm
F2LO	2485.20 Hz
F2PHI	1.146 ppm
F2HI	458.37 Hz
F1PLO	6.496 ppm
F1LO	2599.08 Hz
F1PHI	1.245 ppm
F1HI	498.04 Hz
F2PPMCM	0.33769 ppm/cm
F2HZCM	135.12186 Hz/cm
F1PPMCM	0.35008 ppm/cm
F1HZCM	140.06950 Hz/cm



24

HMBC



Current Data Parameters

NAME	PL-Oct08-07
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20071006
Time	19.00
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	inv4gslplrnd
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	2620.545 Hz
FIDRES	1.279553 Hz
AQ	0.3908084 sec
RG	16384
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
d1	1.50000000 sec
d2	0.00344828 sec
d6	0.06500000 sec
d13	0.00000300 sec
D16	0.00020000 sec
IN0	0.00002240 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	7.10 usec
p2	14.20 usec
PL1	0.00 dB
SFO1	400.1317546 MHz

========== CHANNEL f2 ==========

NUC2	13C
P3	17.00 usec
PL2	-6.00 dB
SFO2	100.6227903 MHz

========== GRADIENT CHANNEL ========

P16	1000.00 usec

F1 - Acquisition parameters

ND0	2
TD	512
SFO1	100.6228 MHz
FIDRES	45.596539 Hz
SW	221.833 ppm

F2 - Processing parameters

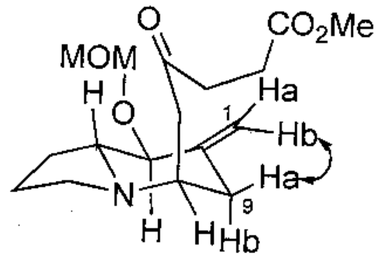
SI	2048
SF	400.1301597 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	1024
MC2	QF
SF	100.6126736 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

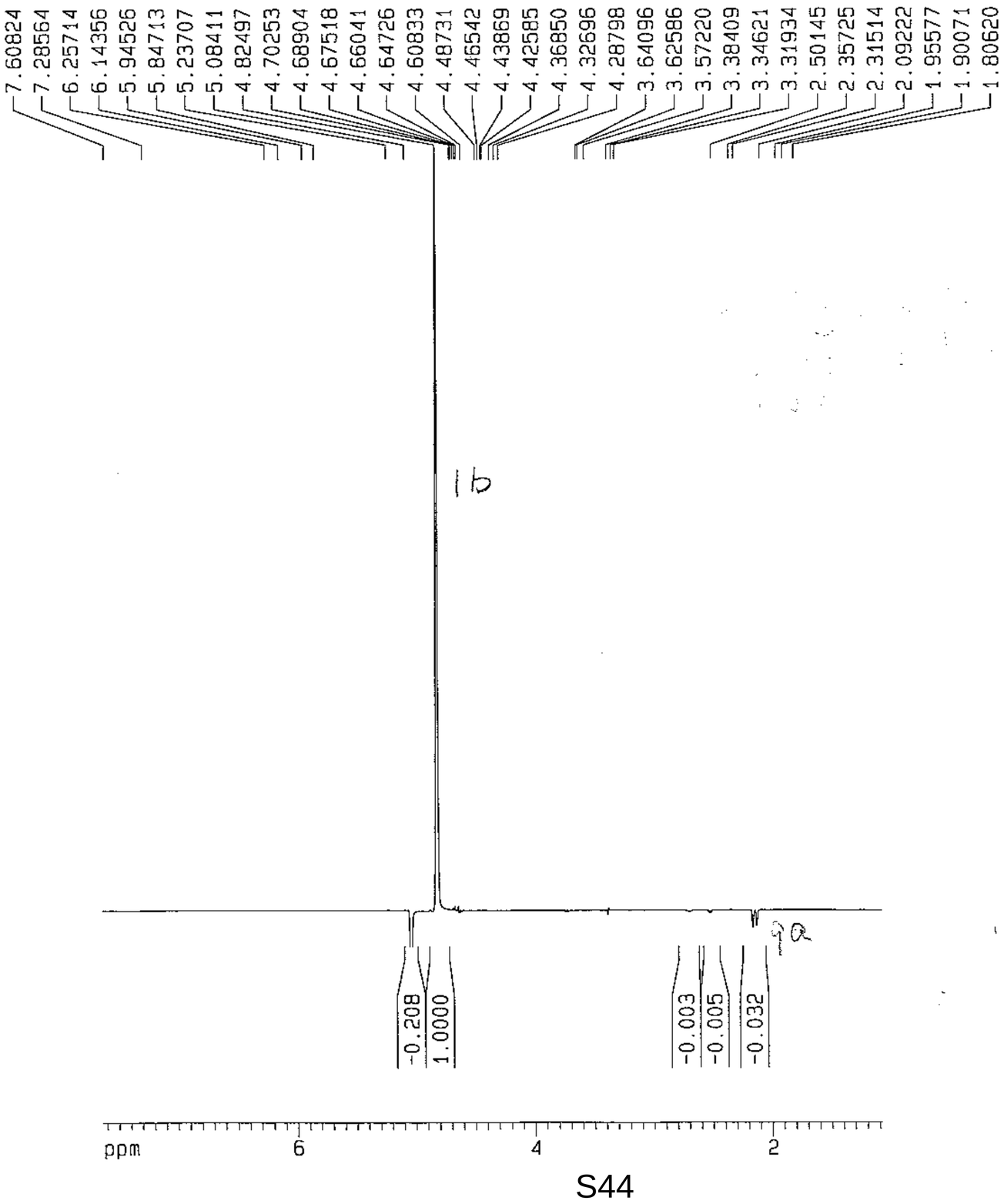
2D NMR plot parameters

CX2	20.00 cm
CX1	17.00 cm
F2PLO	5.889 ppm
F2LO	2348.52 Hz
F2PHI	0.746 ppm
F2HI	298.66 Hz
F1PLO	211.261 ppm
F1LO	21255.57 Hz
F1PHI	-8.960 ppm
F1HI	-891.47 Hz
F2PPMCM	0.25615 ppm/cm
F2HZCM	102.49308 Hz/cm
F1PPMCM	12.94834 ppm/cm
F1HZCM	1301.70723 Hz/cm



NOESY

24



Current Data Parameters

NAME	PL-Oct08-07
EXPNO	5
PROCNO	11

F2 - Acquisition Parameters

Date_	20071009
Time	0.31
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	noesygptp
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	2620.545 Hz
FIDRES	1.279563 Hz
AQ	0.3908084 sec
RG	35.9
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	4.00000000 sec
D8	0.80000001 sec
D16	0.00020000 sec
d20	0.39880002 sec
IN0	0.00019080 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	0.00 dB
SFO1	400.1317546 MHz

========== GRADIENT CHANNEL ========

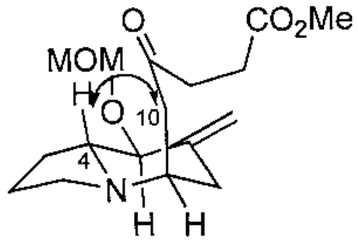
GPNAM1	sine.100
GPNAM2	sine.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	40.00 %
GPZ2	-40.00 %
P16	1000.00 usec

F2 - Processing parameters

SI	2048
SF	400.1300092 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

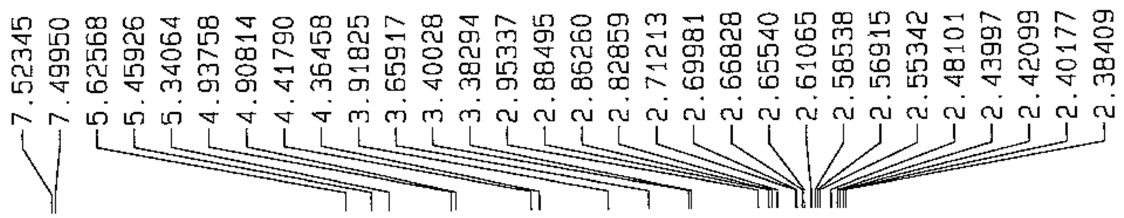
1D NMR plot parameters

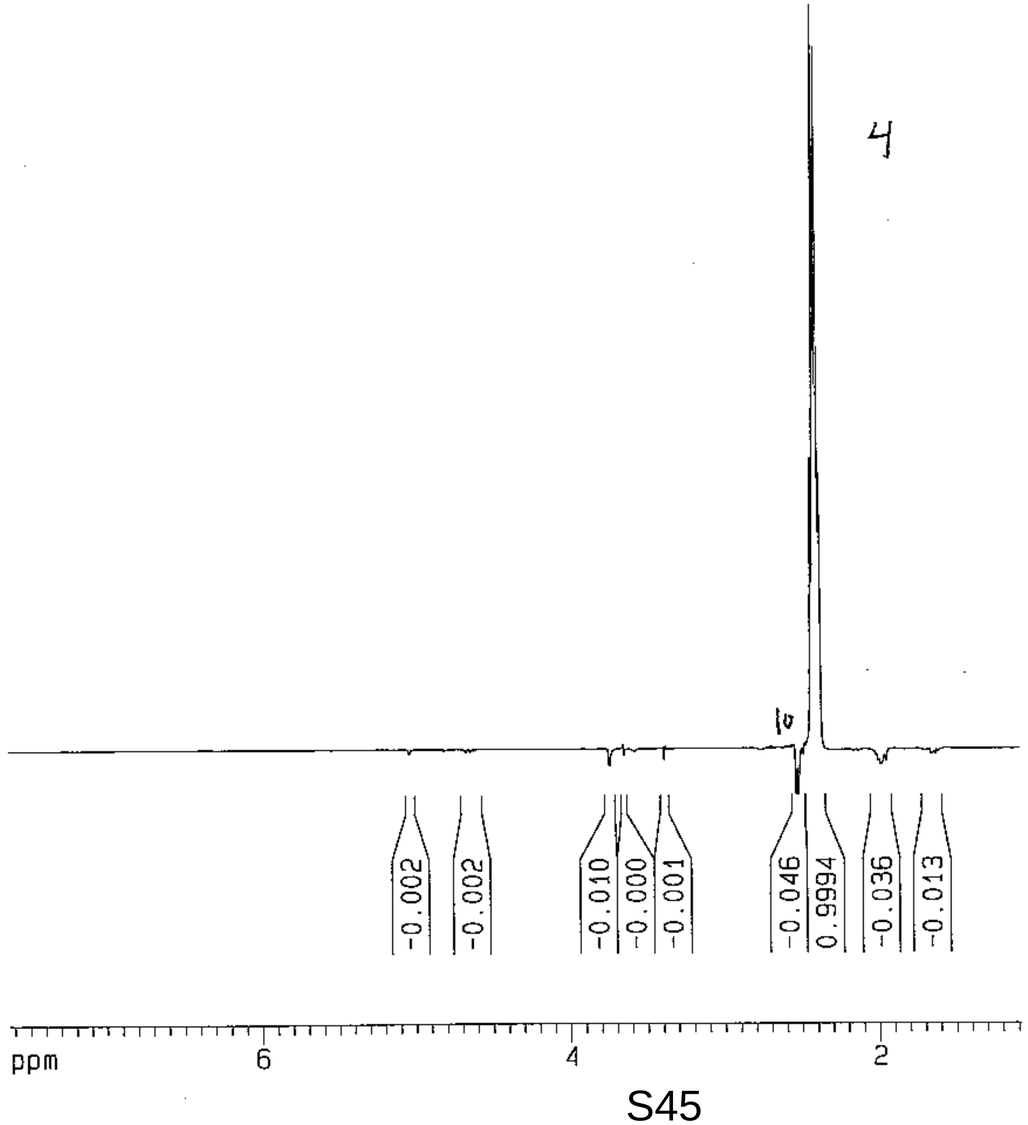
CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm



24

NOESY





Current Data Parameters

NAME	PL-Oct08-07
EXPNO	5
PROCNO	5

F2 - Acquisition Parameters

Date_	20071009
Time	0.31
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	noesygptp
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	2620.545 Hz
FIDRES	1.279563 Hz
AQ	0.3908084 sec
RG	35.9
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	4.00000000 sec
D8	0.80000001 sec
D16	0.00020000 sec
d20	0.39880002 sec
IN0	0.00019080 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	0.00 dB
SFO1	400.1317546 MHz

========== GRADIENT CHANNEL ========

GPNAM1	sine.100
GPNAM2	sine.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	40.00 %
GPZ2	-40.00 %
P16	1000.00 usec

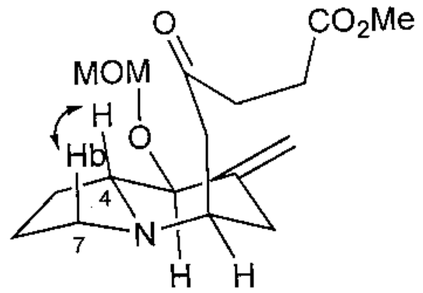
F2 - Processing parameters

SI	2048
SF	400.1300092 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

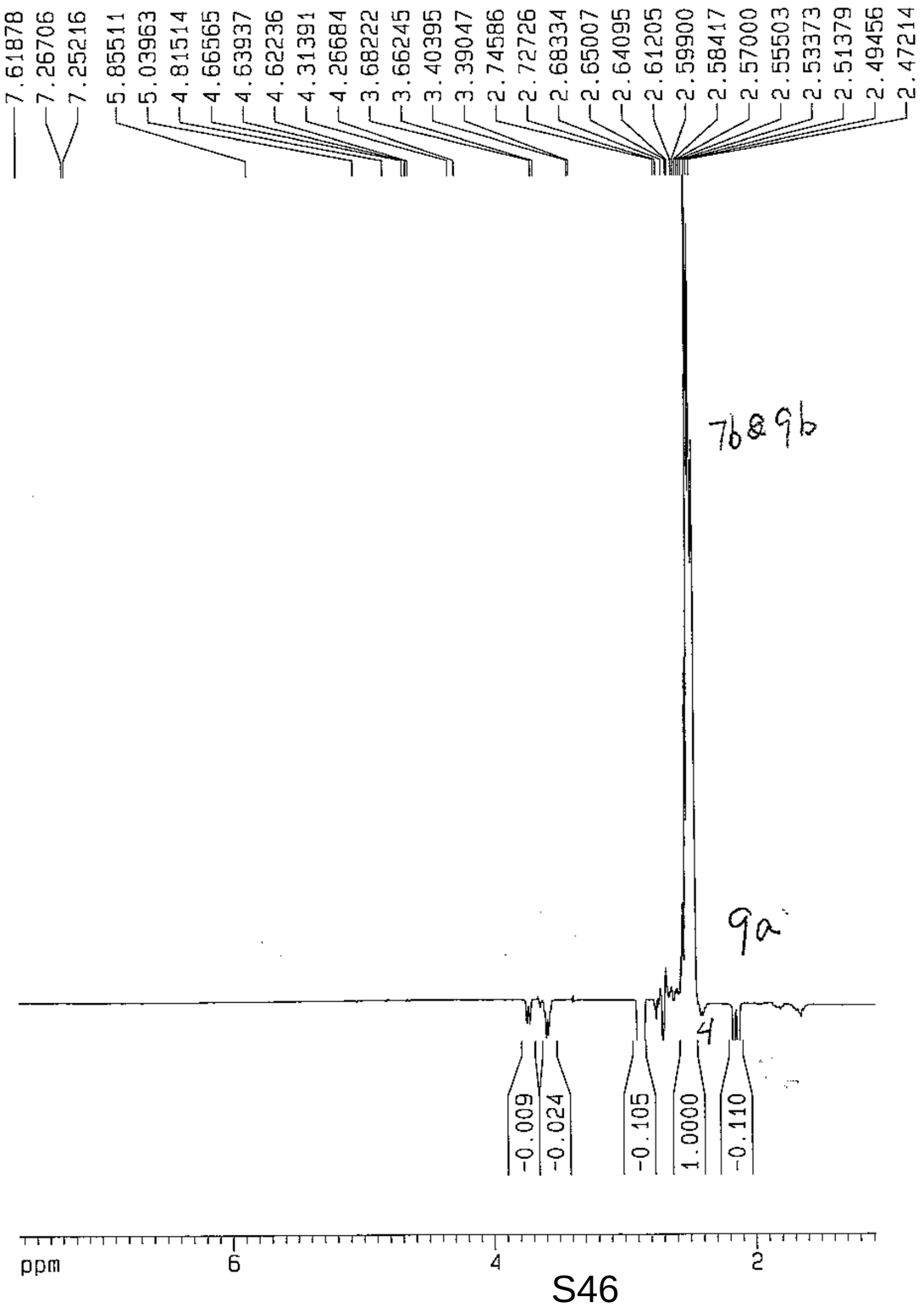
1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm

NOESY



24



Current Data Parameters

NAME	PL-Oct08-07
EXPNO	5
PROCNO	29

F2 - Acquisition Parameters

Date_	20071009
Time	0.31
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	noesygptp
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	2620.545 Hz
FIDRES	1.279563 Hz
AQ	0.3908084 sec
RG	35.9
DW	190.800 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	4.00000000 sec
D8	0.80000001 sec
D16	0.00020000 sec
d20	0.39880002 sec
IN0	0.00019080 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	0.00 dB
SFO1	400.1317546 MHz

========== GRADIENT CHANNEL ========

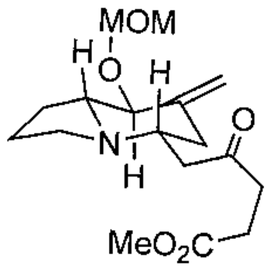
GPNAM1	sine.100
GPNAM2	sine.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	40.00 %
GPZ2	-40.00 %
P16	1000.00 usec

F2 - Processing parameters

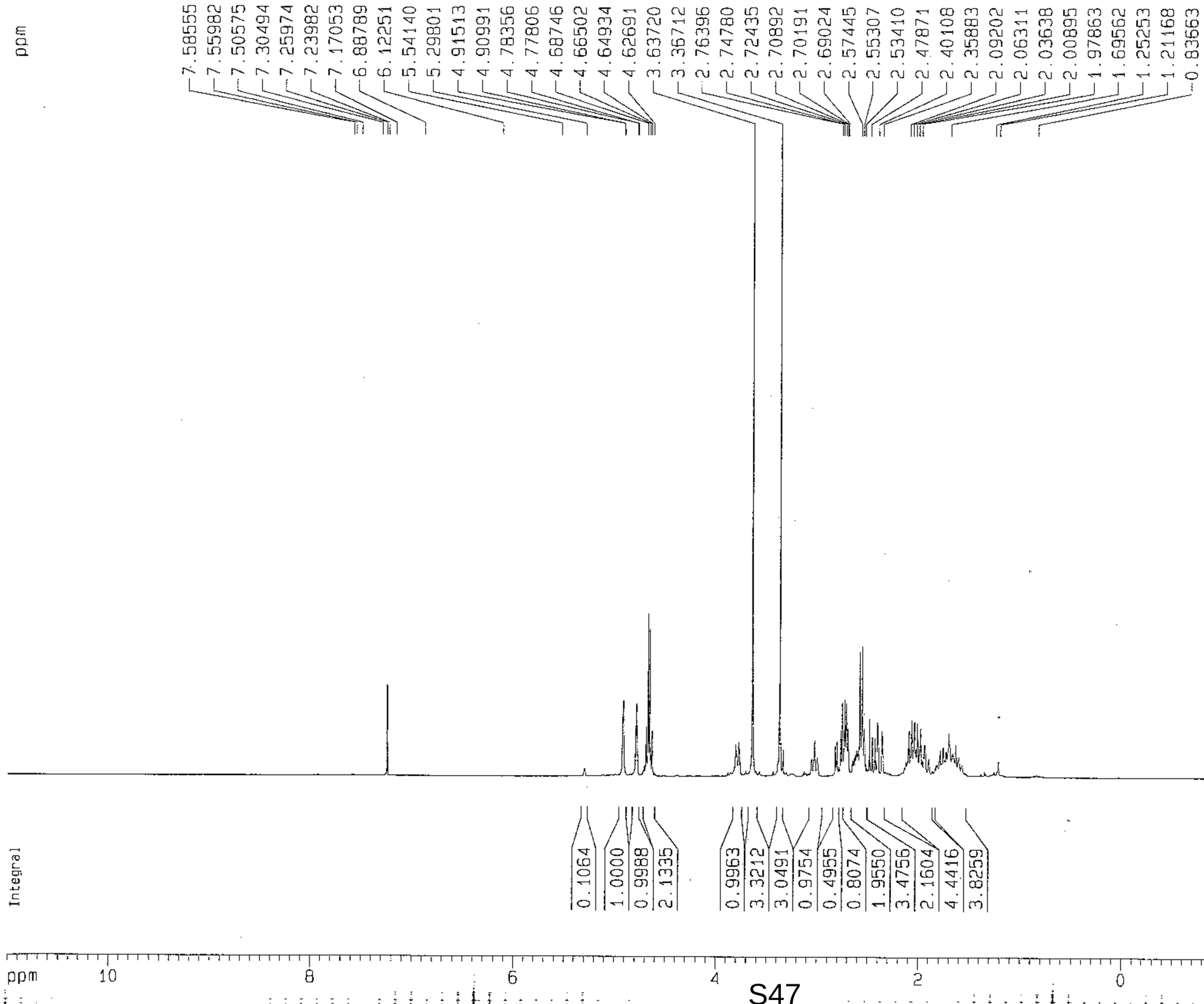
SI	2048
SF	400.1300092 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm



23



Current Data Parameters

NAME	PL-Feb02-08
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080202
Time	14.06
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	128
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

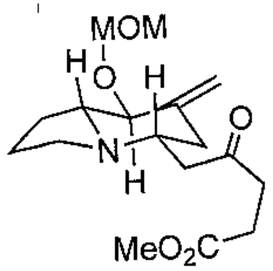
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

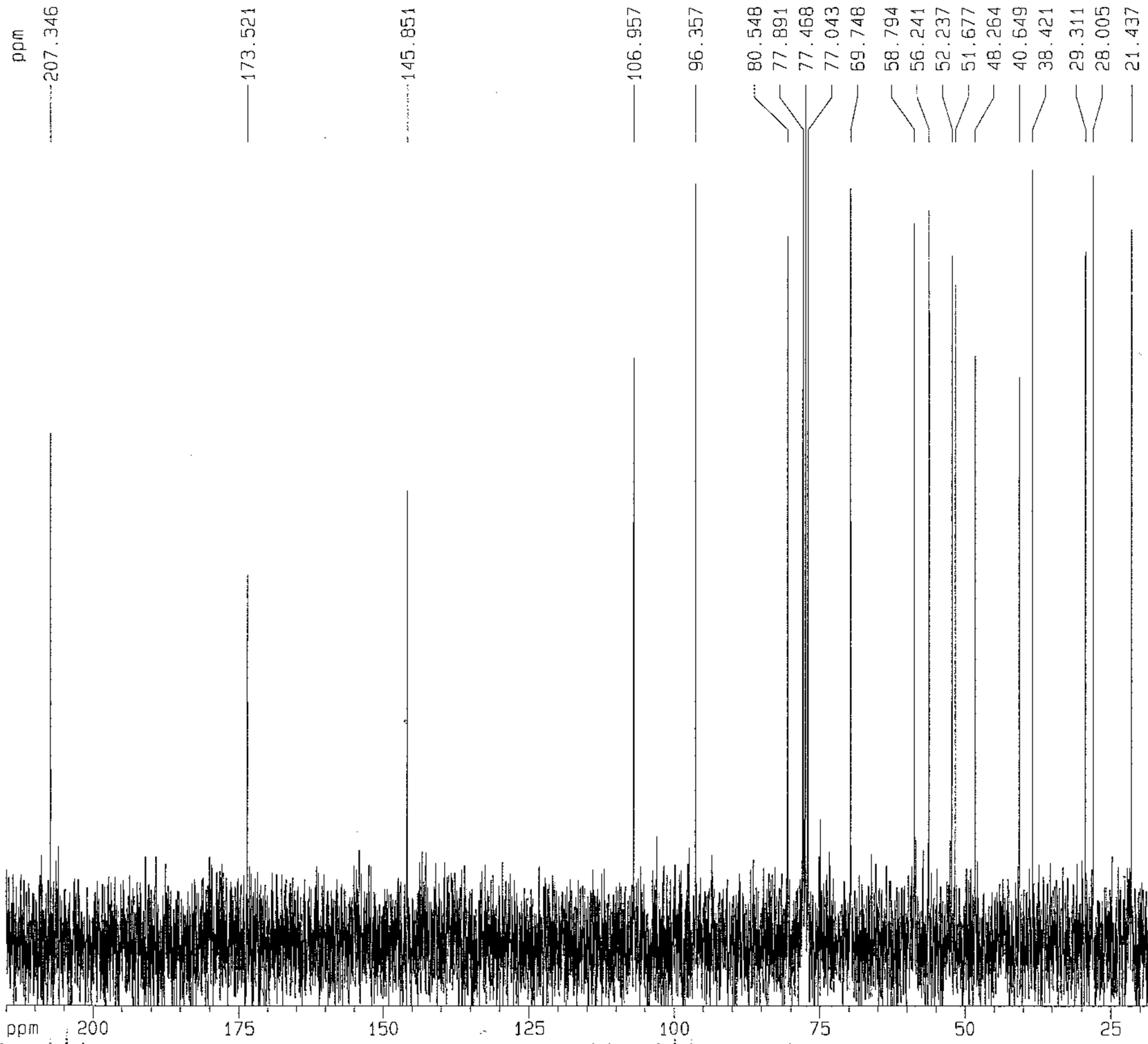
SI	32768
SF	299.8700161 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



23



Current Data Parameters

NAME	PL-Feb02-08
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080202
Time	14.14
INSTRUM	spect
PROBHD	5 mm GNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	55
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1024
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

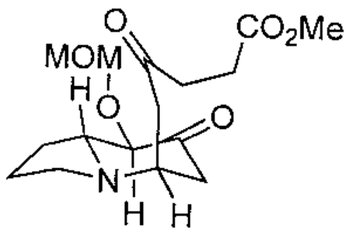
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

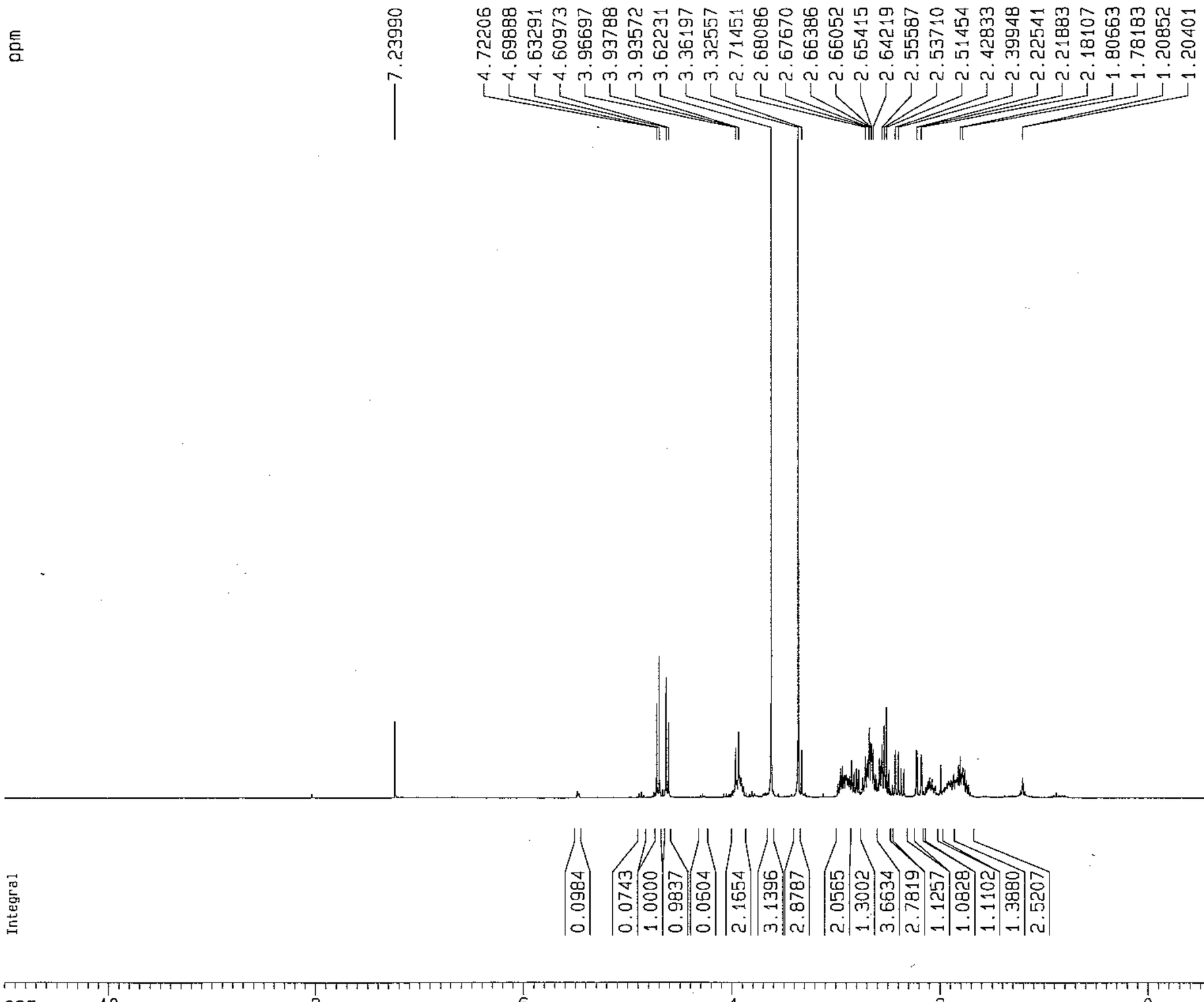
SI	32768
SF	75.4023410 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.50 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42578 Hz/cm



25



Current Data Parameters

NAME	PL-Feb10-08
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080210
Time	15.43
INSTRUM	spect
PROBHD	5 mm GNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	114
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

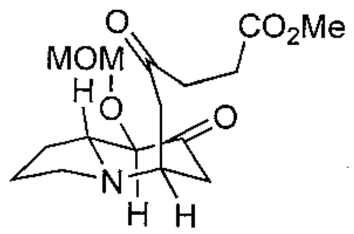
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

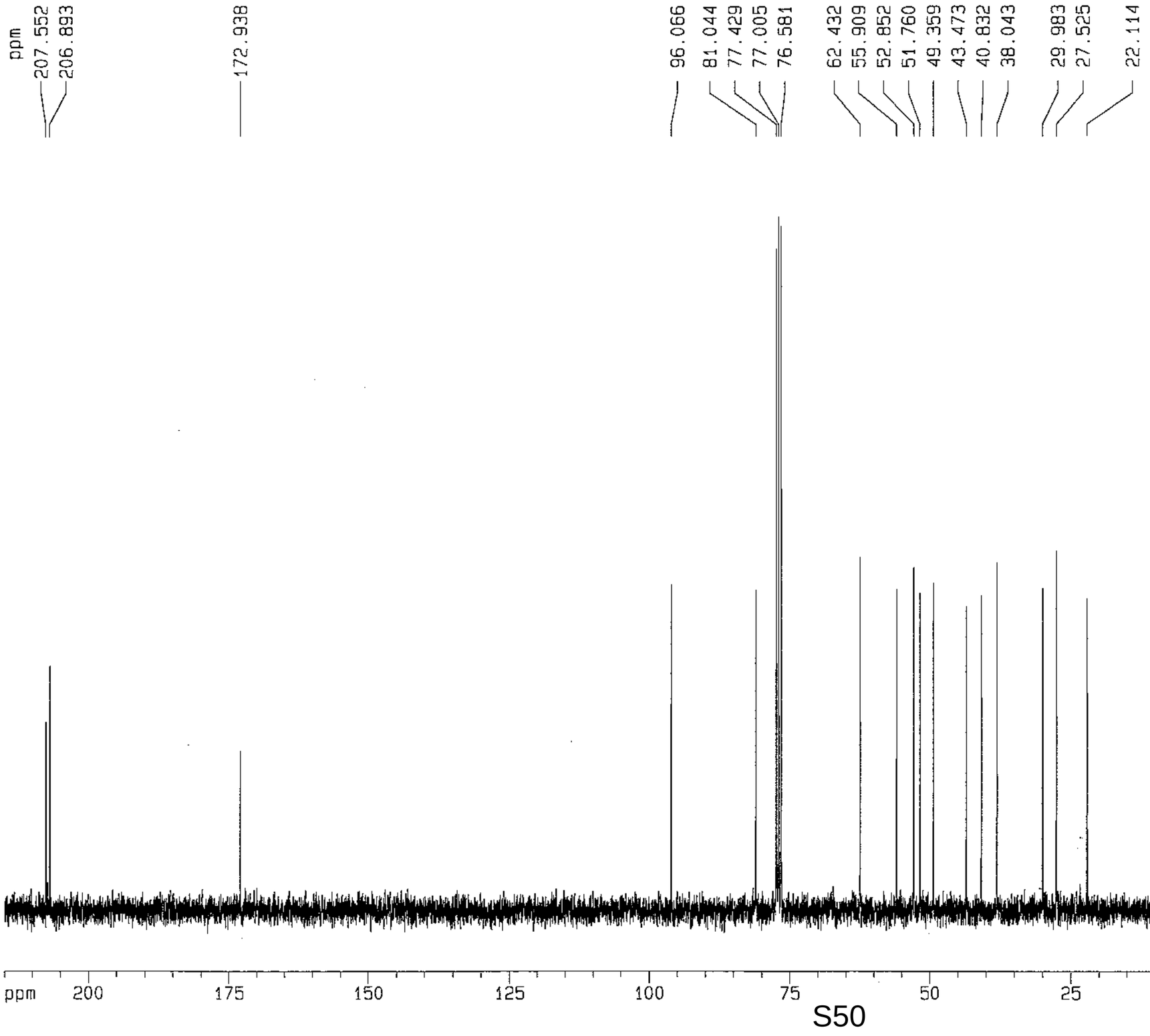
SI	32768
SF	299.8700159 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



25



Current Data Parameters

NAME	PL-Feb10-08
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20080210
Time	15.50
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	119
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	2048
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

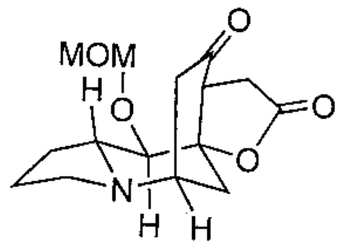
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

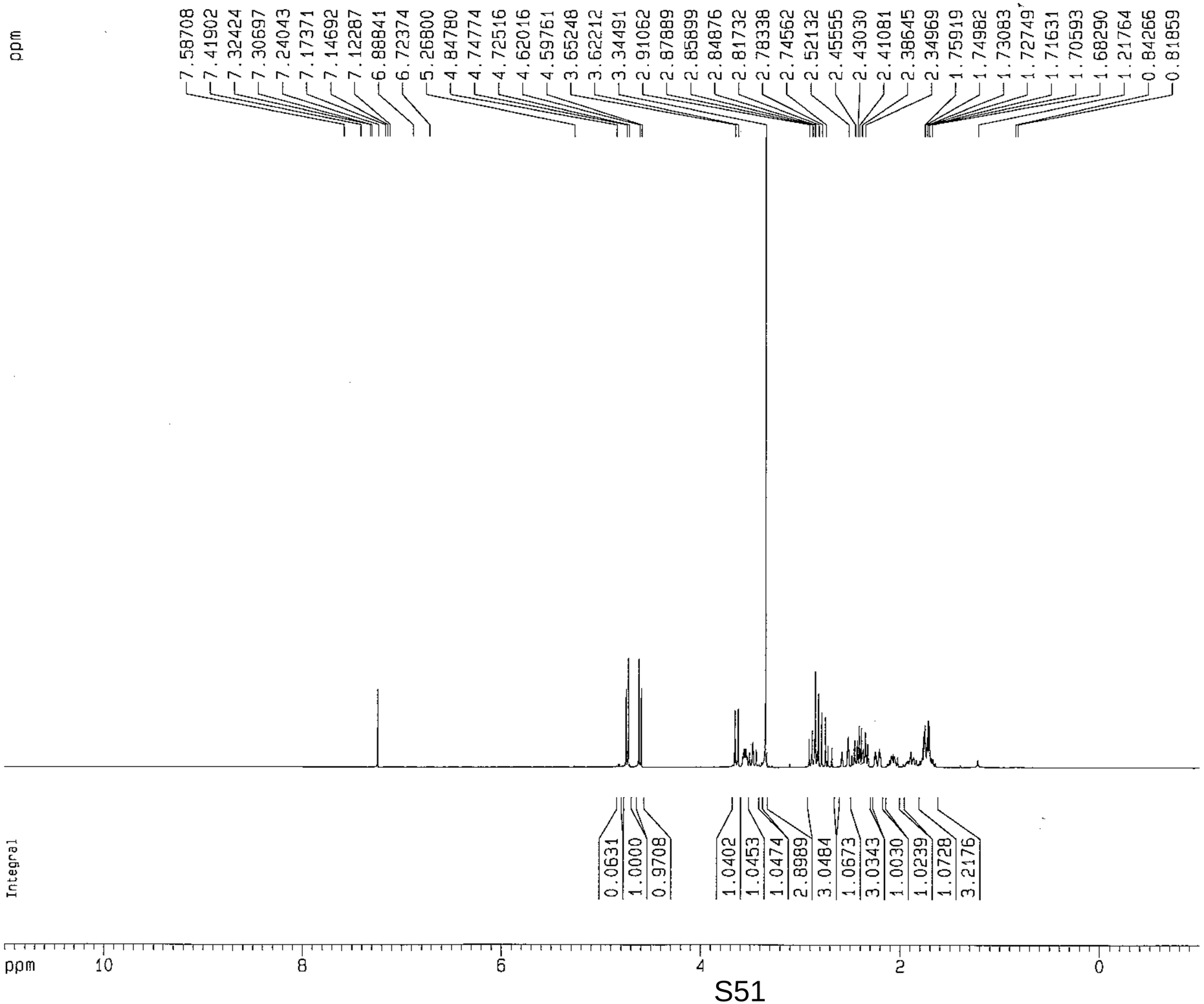
SI	32768
SF	75.4023769 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



26



Current Data Parameters

NAME	PL-Feb15-08
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080215
Time	10.05
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	203.2
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

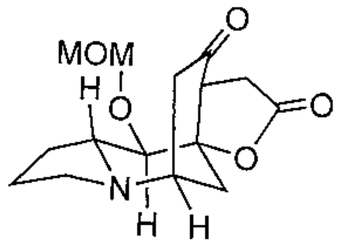
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

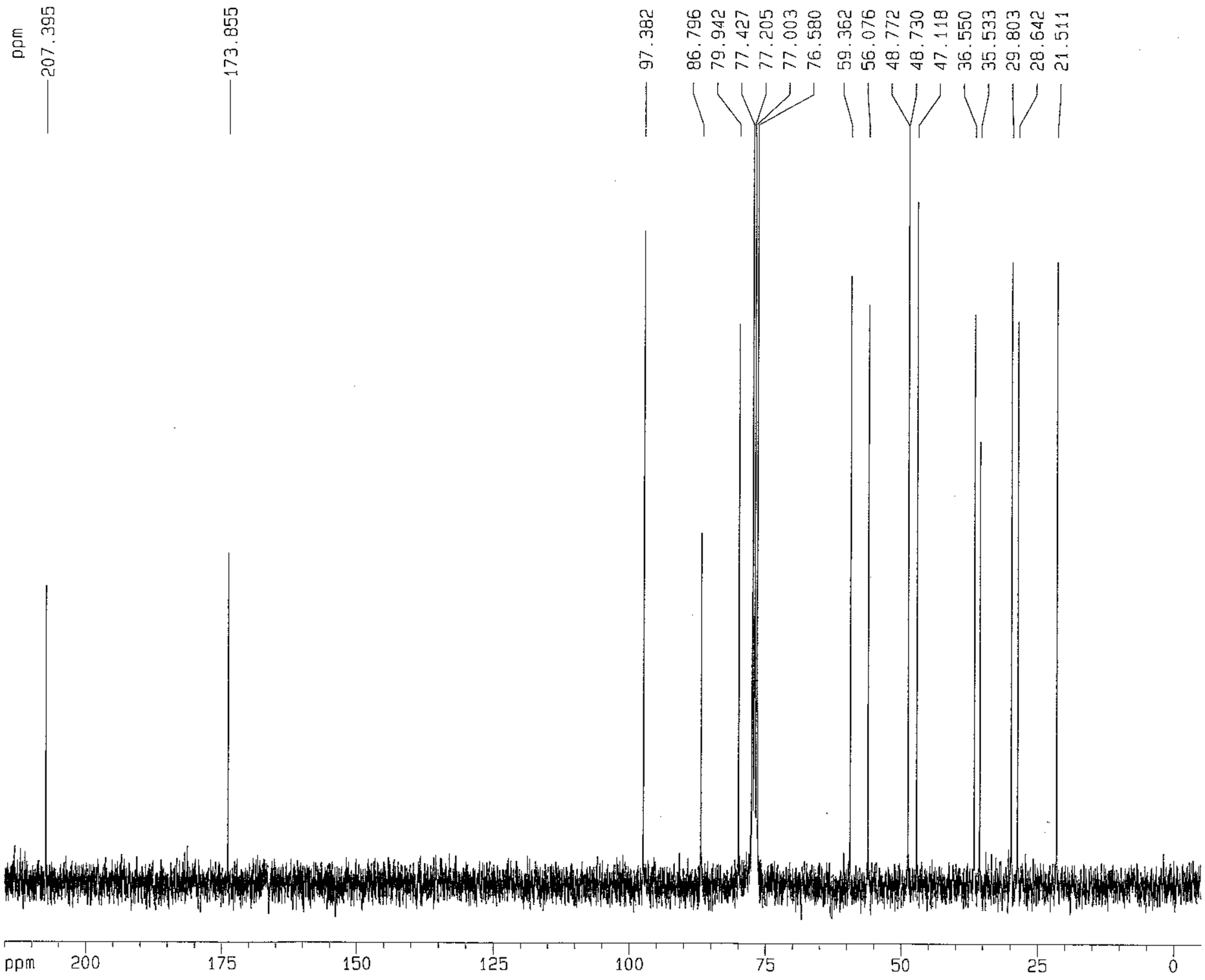
SI	32768
SF	299.8700157 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

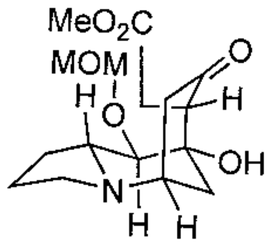
CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



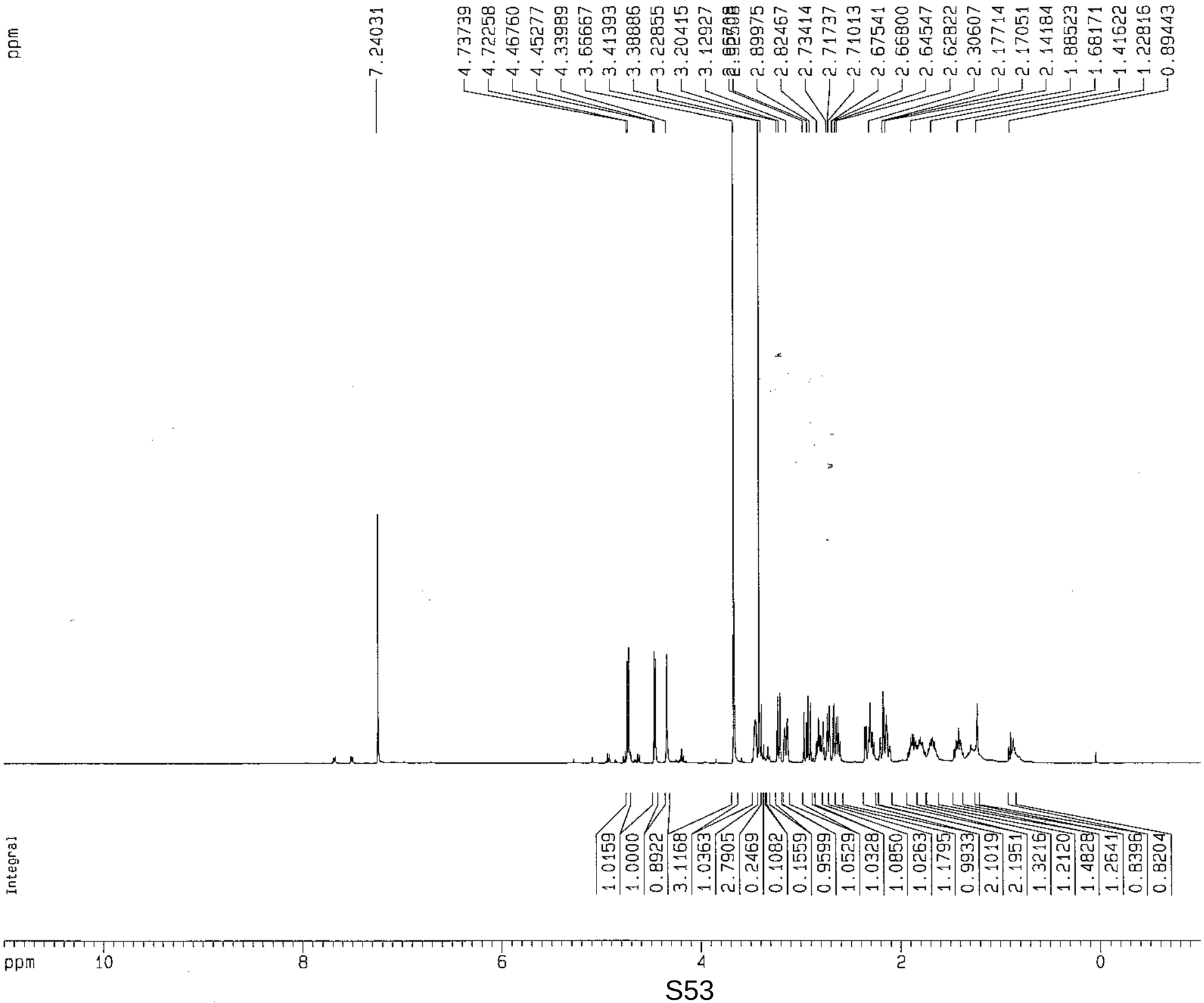
26




```
Current Data Parameters
NAME                  PL-Nov18-07
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20071118
Time                        22.56
INSTRUM                     spect
PROBHD       5 mm QNP 1H/1
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          2160
DS                             4
SWH                  18796.992 Hz
FIDRES                0.286819 Hz
AQ                   1.7433076 sec
RG                          1024
DW                     26.600 usec
DE                      6.00 usec
TE                      300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec

========== CHANNEL f1 ==========
NUC1                         13C
P1                        5.40 usec
PL1                      -6.00 dB
SFO1                75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                   115.00 usec
PL2                       0.00 dB
PL12                     20.00 dB
PL13                     20.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4023746 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                     16211.51 Hz
F2P                      -5.000 ppm
F2                      -377.01 Hz
PPMCM                  11.00000 ppm/cm
HZCM                  829.42615 Hz/cm
```



28



Current Data Parameters

NAME	PL-Mar30-08
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080330
Time	20.41
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	114
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

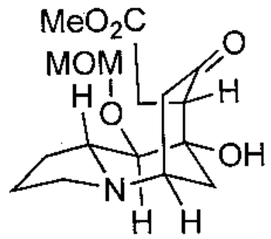
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

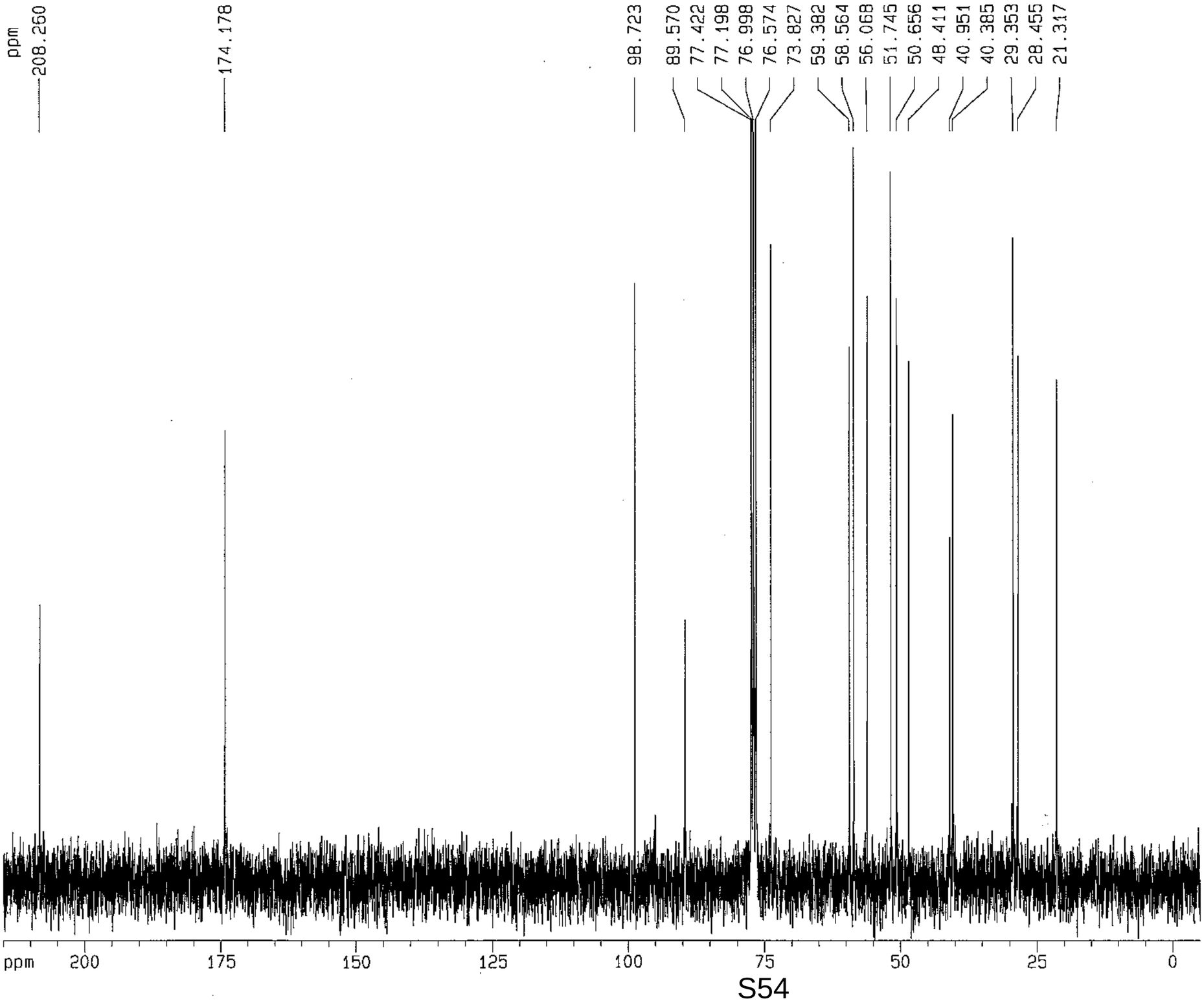
SI	32768
SF	400.1300169 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm



28



Current Data Parameters

NAME	PL-Mar14-08
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080314
Time	12.45
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	514
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1625.5
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

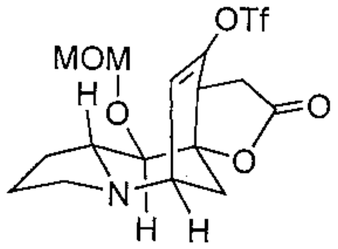
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

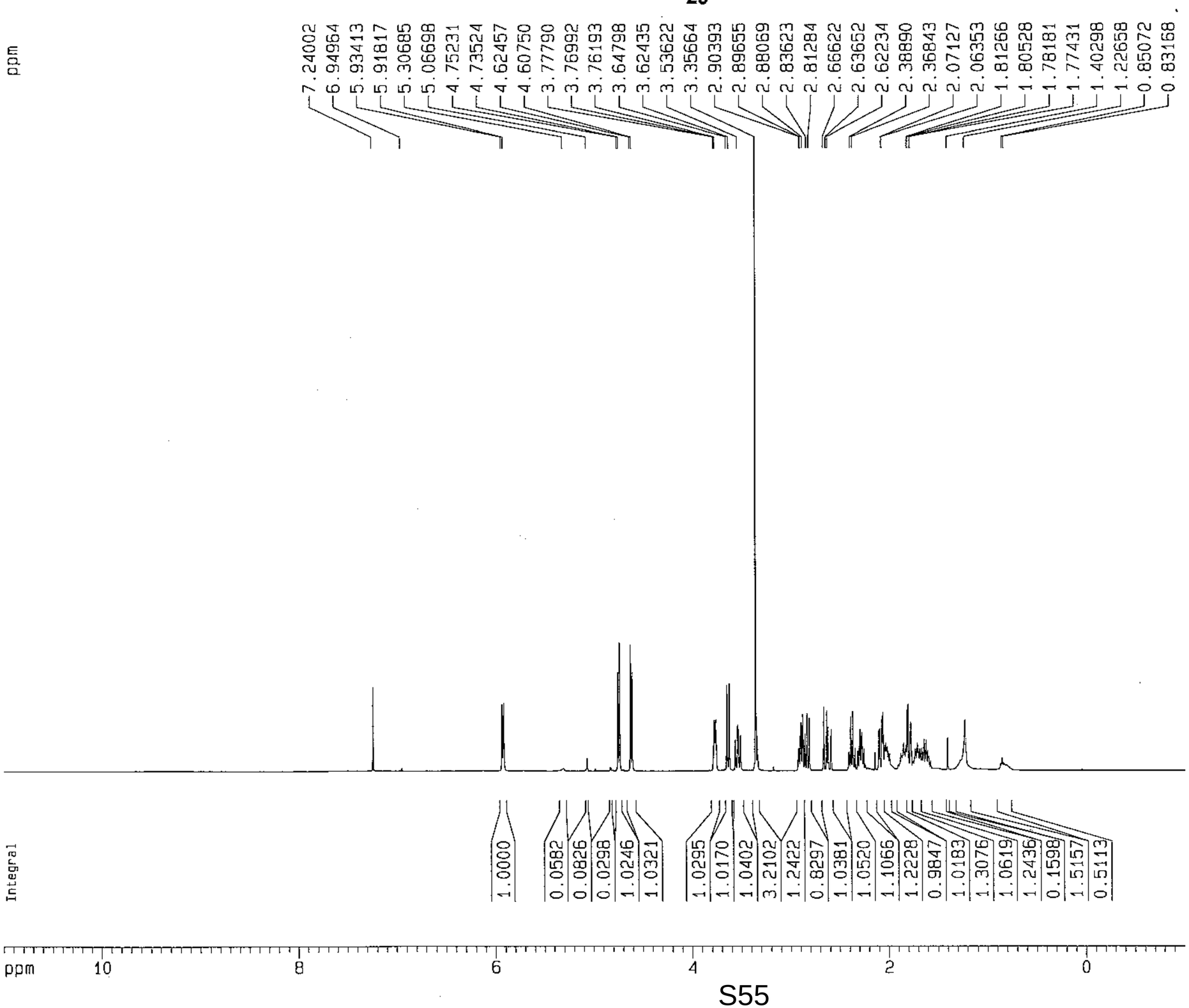
SI	32768
SF	75.4023763 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



29



Current Data Parameters

NAME	PL-Dec19-07
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20071219
Time	17.33
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	24576
SOLVENT	CDCl3
NS	16
DS	0
SWH	8278.146 Hz
FIDRES	0.336839 Hz
AQ	1.4844404 sec
RG	322.5
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

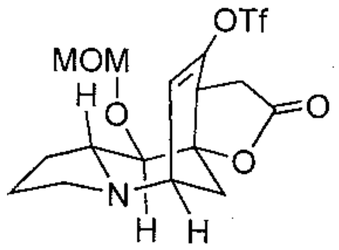
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

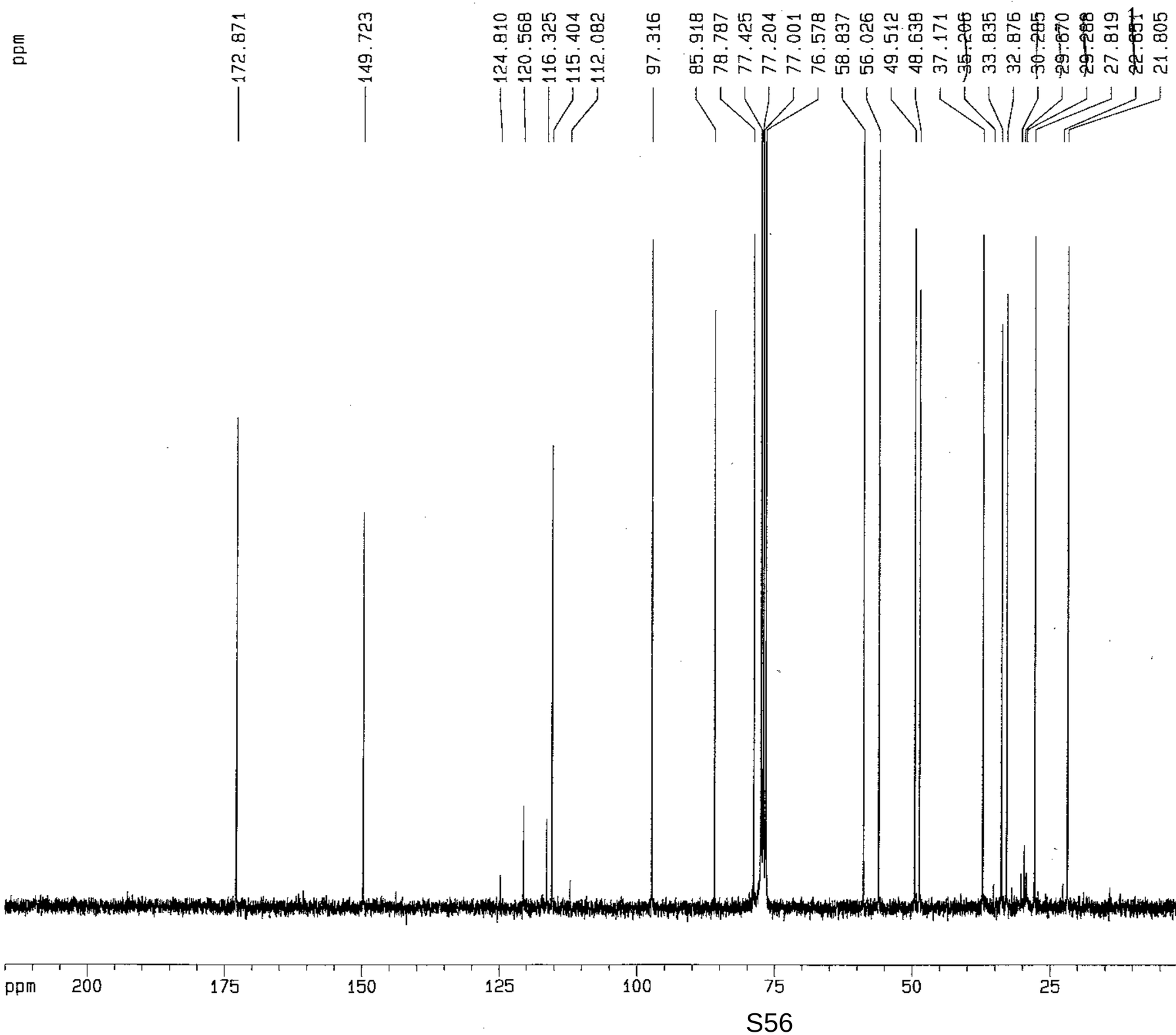
SI	32768
SF	400.1300174 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07802 Hz/cm



29



Current Data Parameters

NAME	PL-Dec19-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20071220
Time	9.46
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	9747
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	16384
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	11.80 usec
PL1	0.00 dB
SFO1	75.4760200 MHz

========== CHANNEL f2 ==========

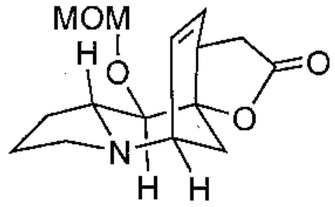
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677509 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

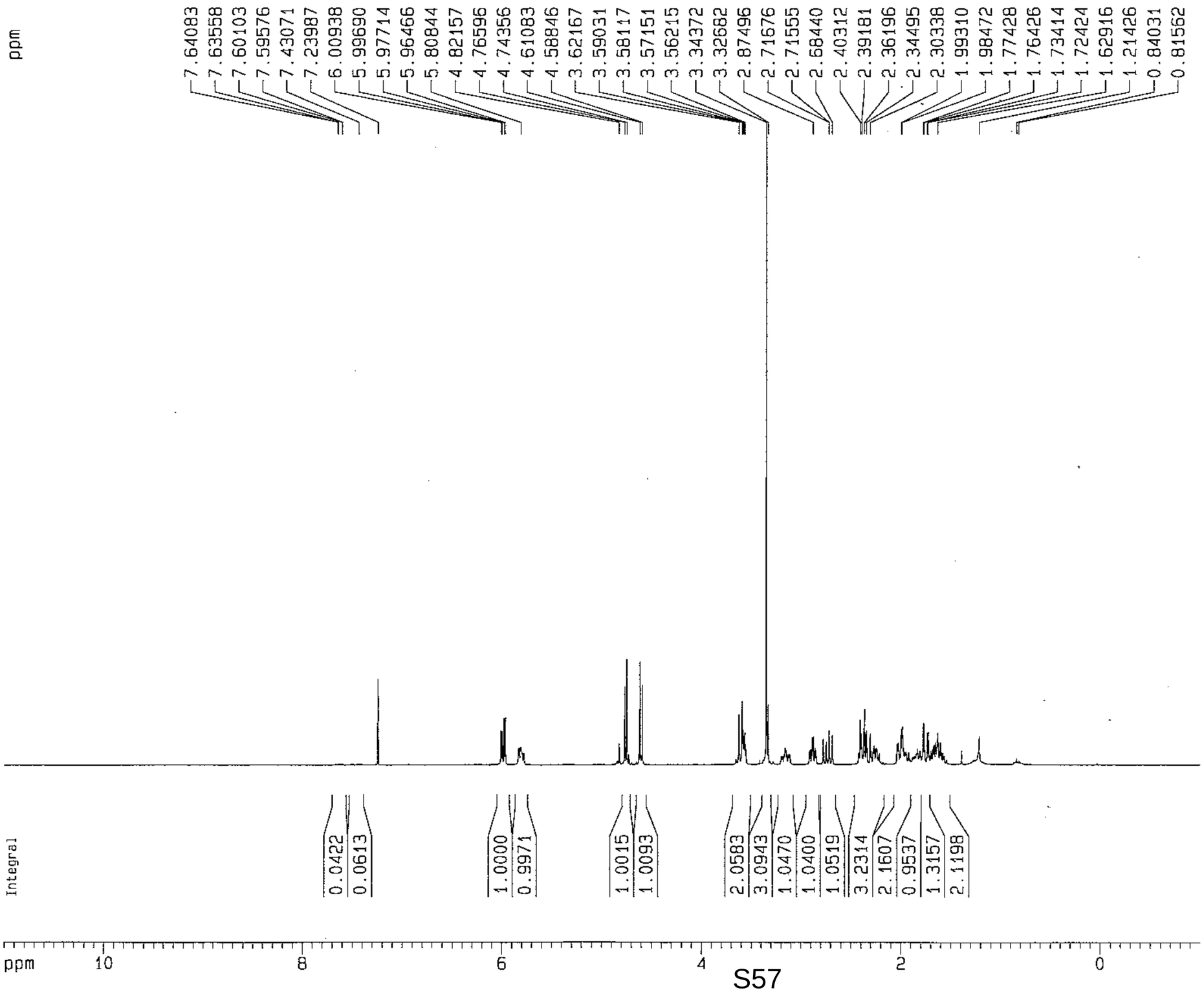
1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16225.57 Hz
F2P	-5.000 ppm
F2	-377.34 Hz
PPMCM	11.00000 ppm/cm
HZCM	830.14526 Hz/cm



30

$C_{15}H_{21}NO_4$



Current Data Parameters

NAME	PL-Mar08-08
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080308
Time	13.17
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	228.1
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

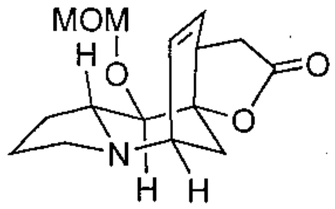
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

SI	32768
SF	299.8700161 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

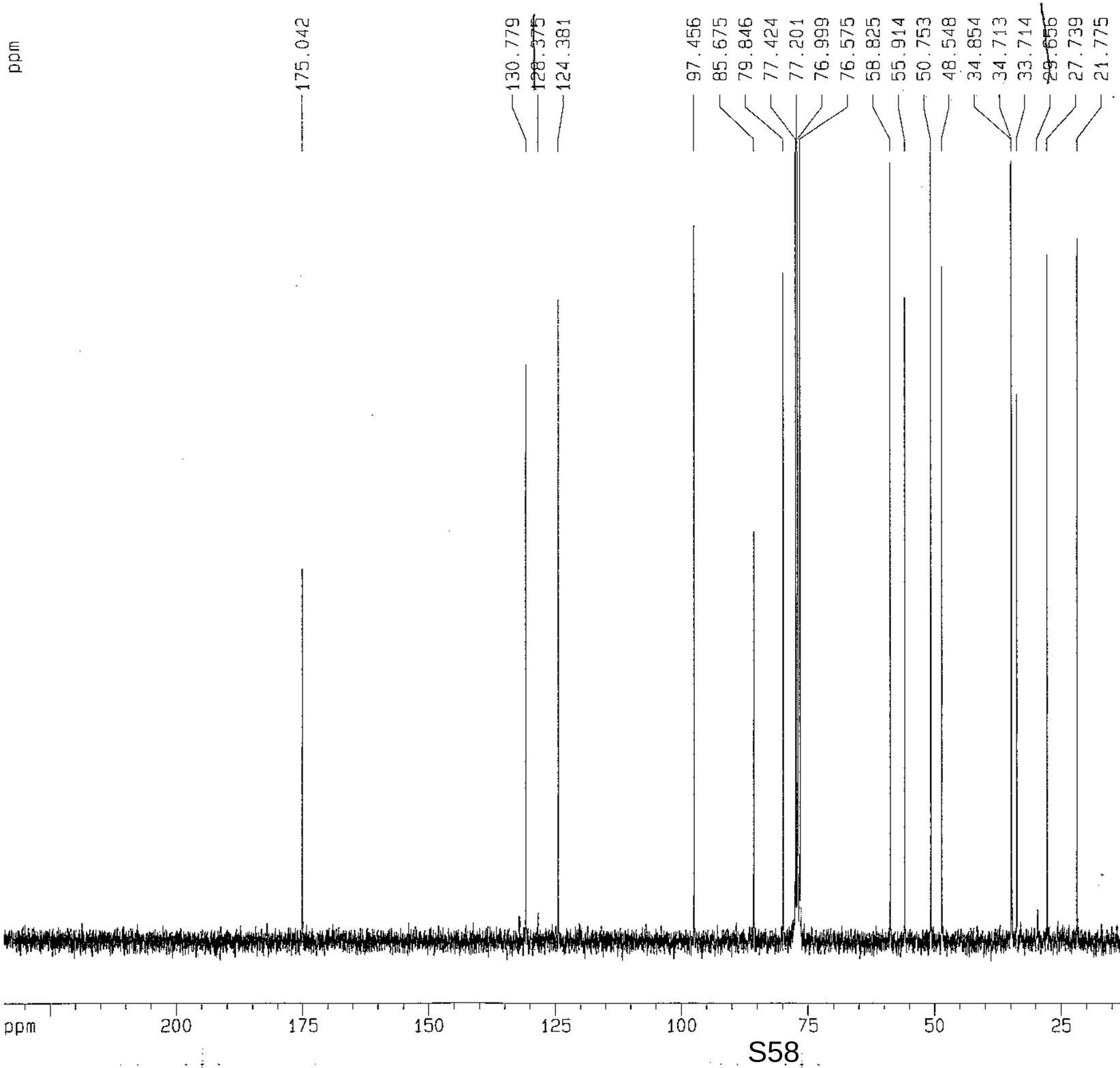
1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm

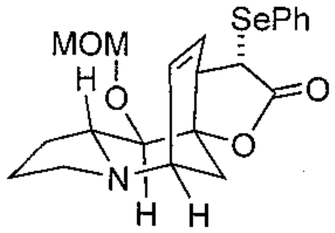


30

C15

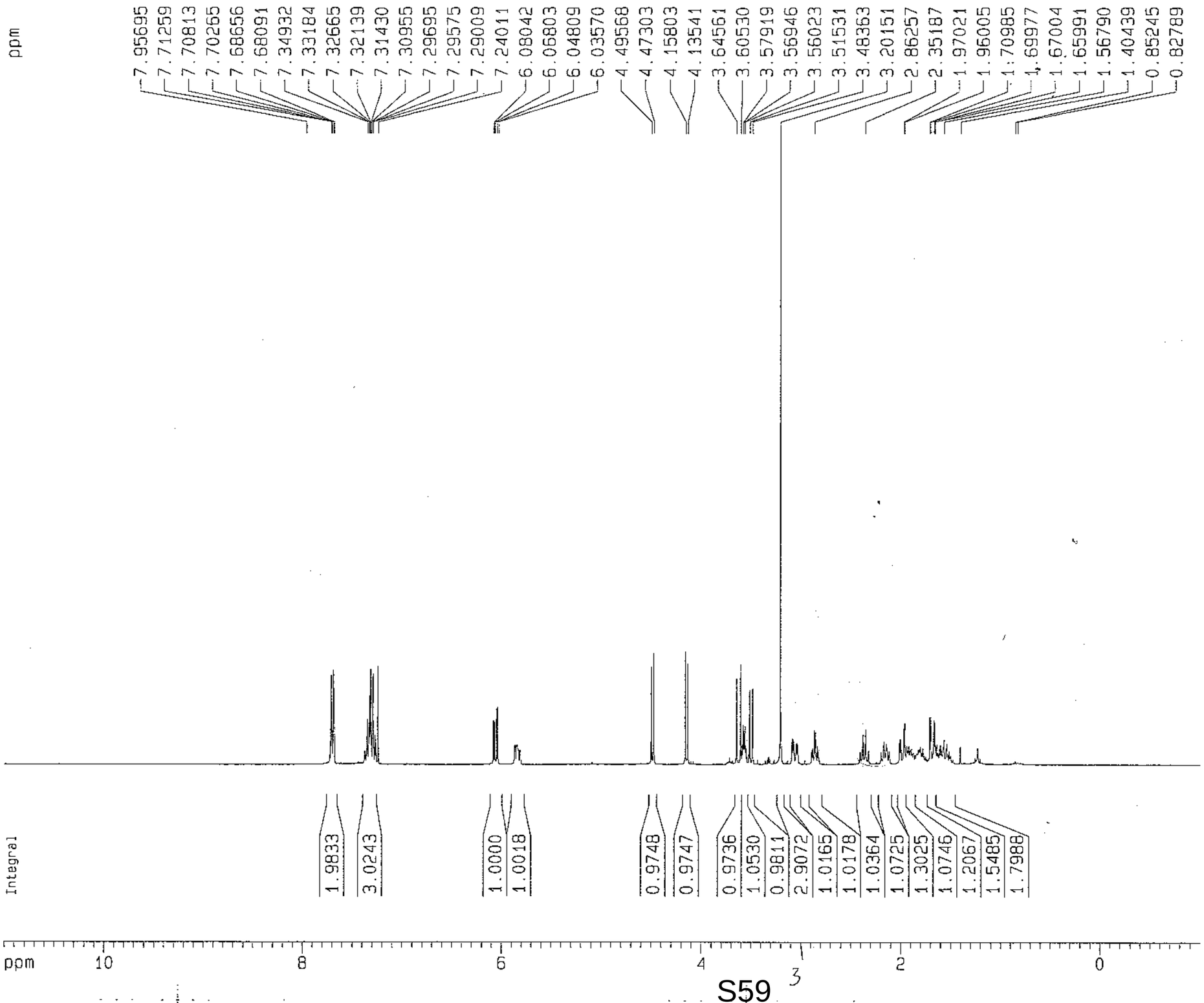



```
Current Data Parameters
NAME                  PL-Dec29-07
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20071229
Time                        13.53
INSTRUM                     spect
PROBHD          5 mm GNP 1H/1
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1377
DS                             4
SWH                  18796.992 Hz
FIDRES                0.286819 Hz
AQ                   1.7433076 sec
RG                          1024
DW                     26.600 usec
DE                      6.00 usec
TE                      300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec

========== CHANNEL f1 ==========
NUC1                         13C
P1                         5.40 usec
PL1                       -6.00 dB
SFO1                 75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    115.00 usec
PL2                        0.00 dB
PL12                      20.00 dB
PL13                      20.00 dB
SFO2                299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                   75.4023757 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
F1P                       234.190 ppm
F1                      17658.51 Hz
F2P                       -15.099 ppm
F2                      -1138.48 Hz
PPMCM                    12.46446 ppm/cm
HZCM                    939.84961 Hz/cm
```



31

$C_{21}H_{25}NO_4Se$



Current Data Parameters

NAME	PL-Jan04-07
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080104
Time	23.05
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	256
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

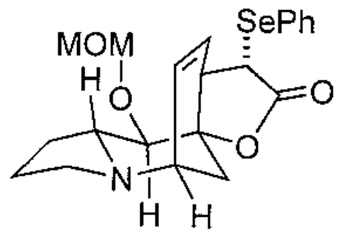
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

SI	32768
SF	299.8700159 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

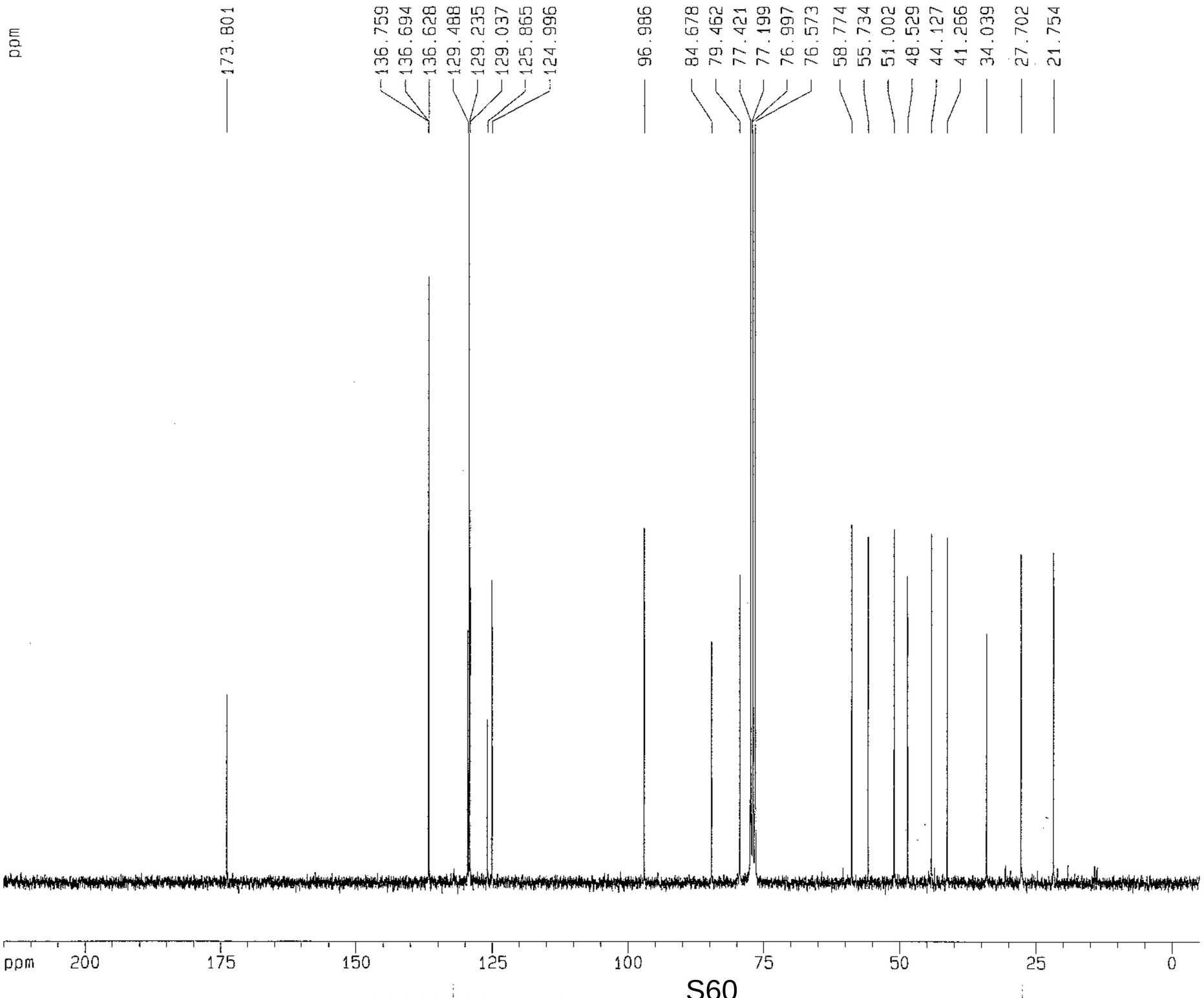
1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



$C_{21}H_{25}NO_4Se$

31



Current Data Parameters

NAME	PL-Jan03-07
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20080103
Time	21.14
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2556
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	512
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

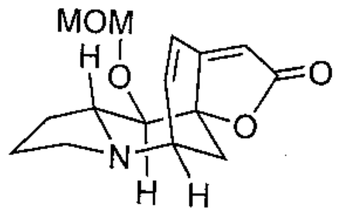
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

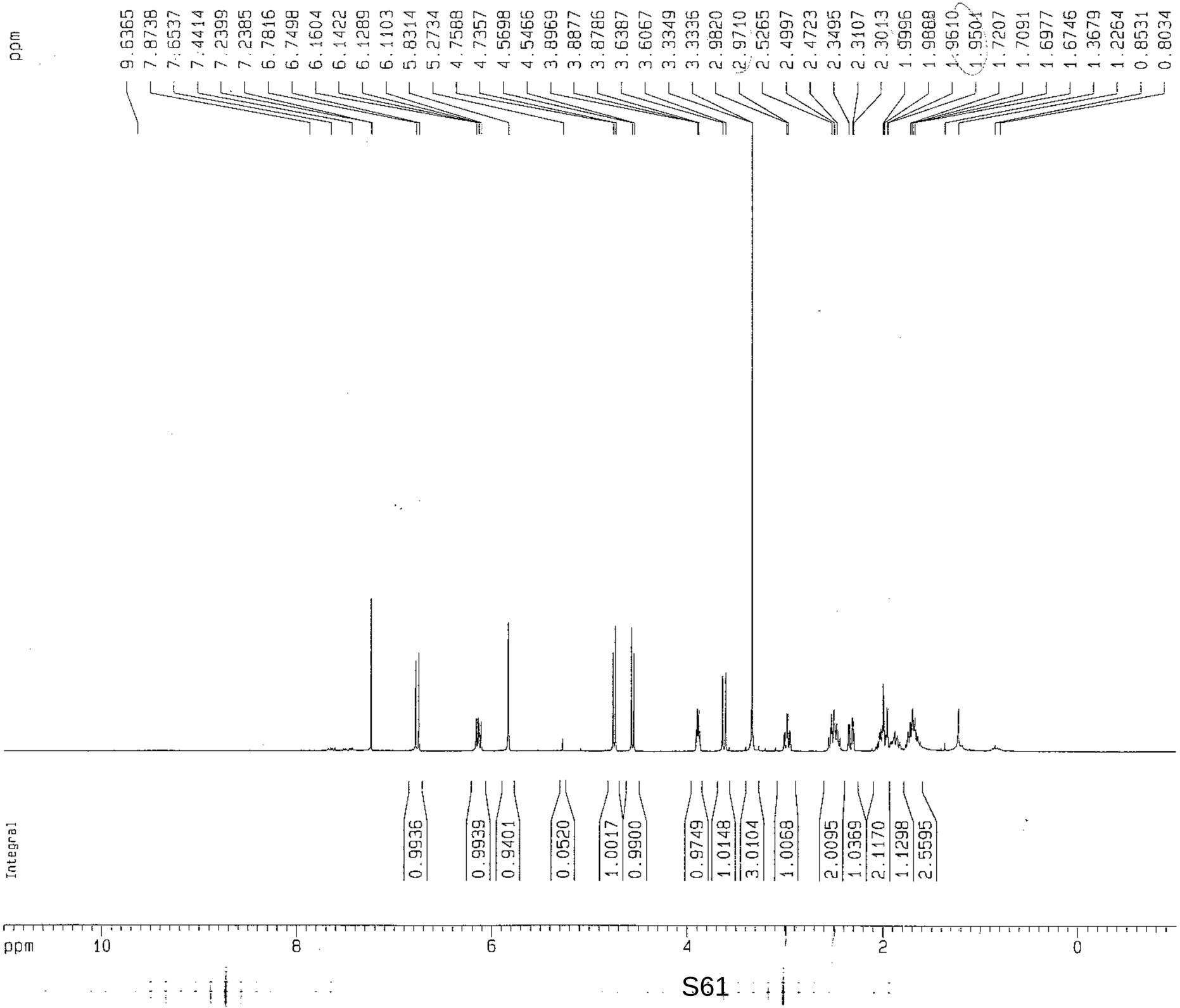
SI	32768
SF	75.4023757 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



32



Current Data Parameters

NAME	PL-Jan06-07
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20080107
Time	0.19
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	456.1
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

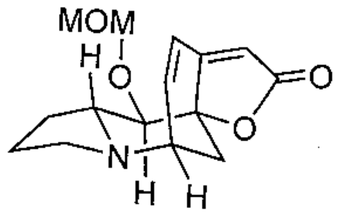
NUC1	1H
P1	11.70 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

F2 - Processing parameters

SI	32768
SF	299.8700163 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

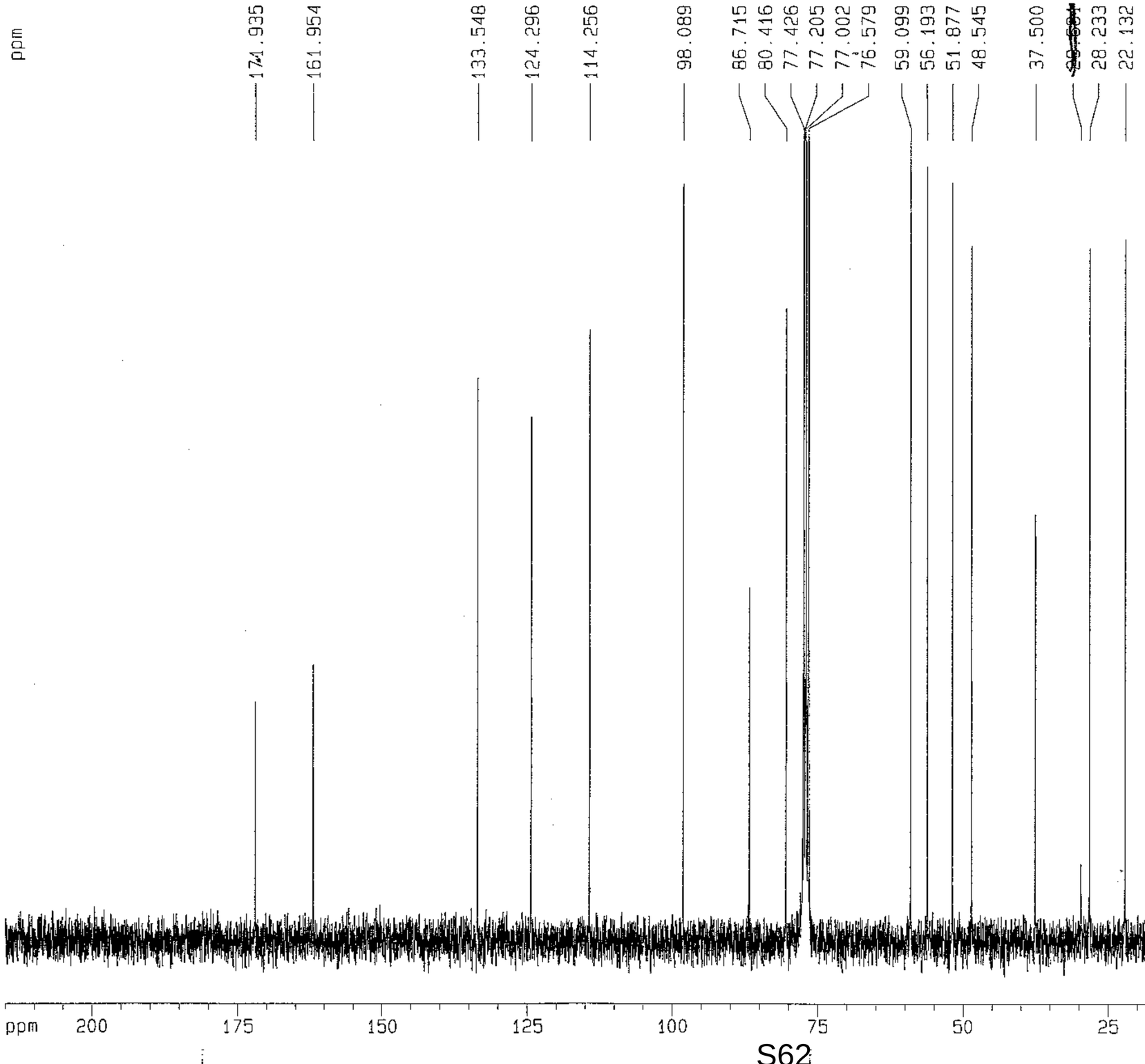
1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm



32

$C_{15}H_{19}NO_4$



Current Data Parameters

NAME	PL-Jan06-07
EXPNO	7
PROCNO	1

F2 - Acquisition Parameters

Date_	20080107
Time	2.40
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2024
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1024
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

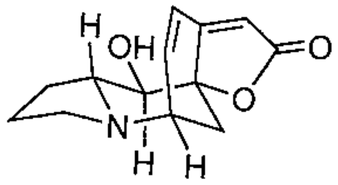
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

SI	32768
SF	75.4023746 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

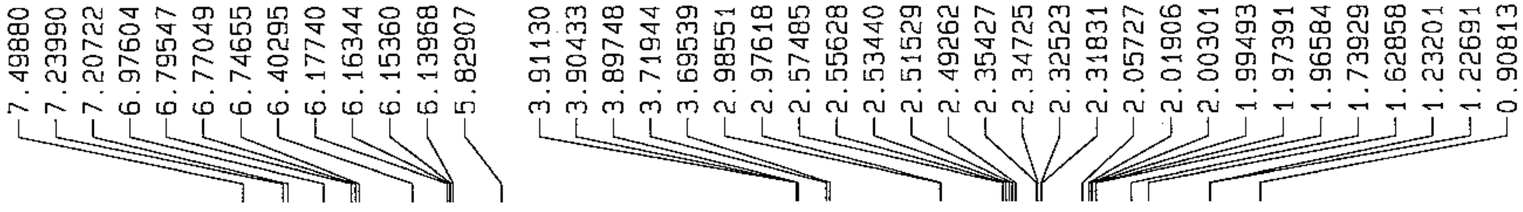
1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



5

$C_{13}H_{15}NO_3$



Current Data Parameters

NAME	PL-Jan07-08
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080108
Time	14.41
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

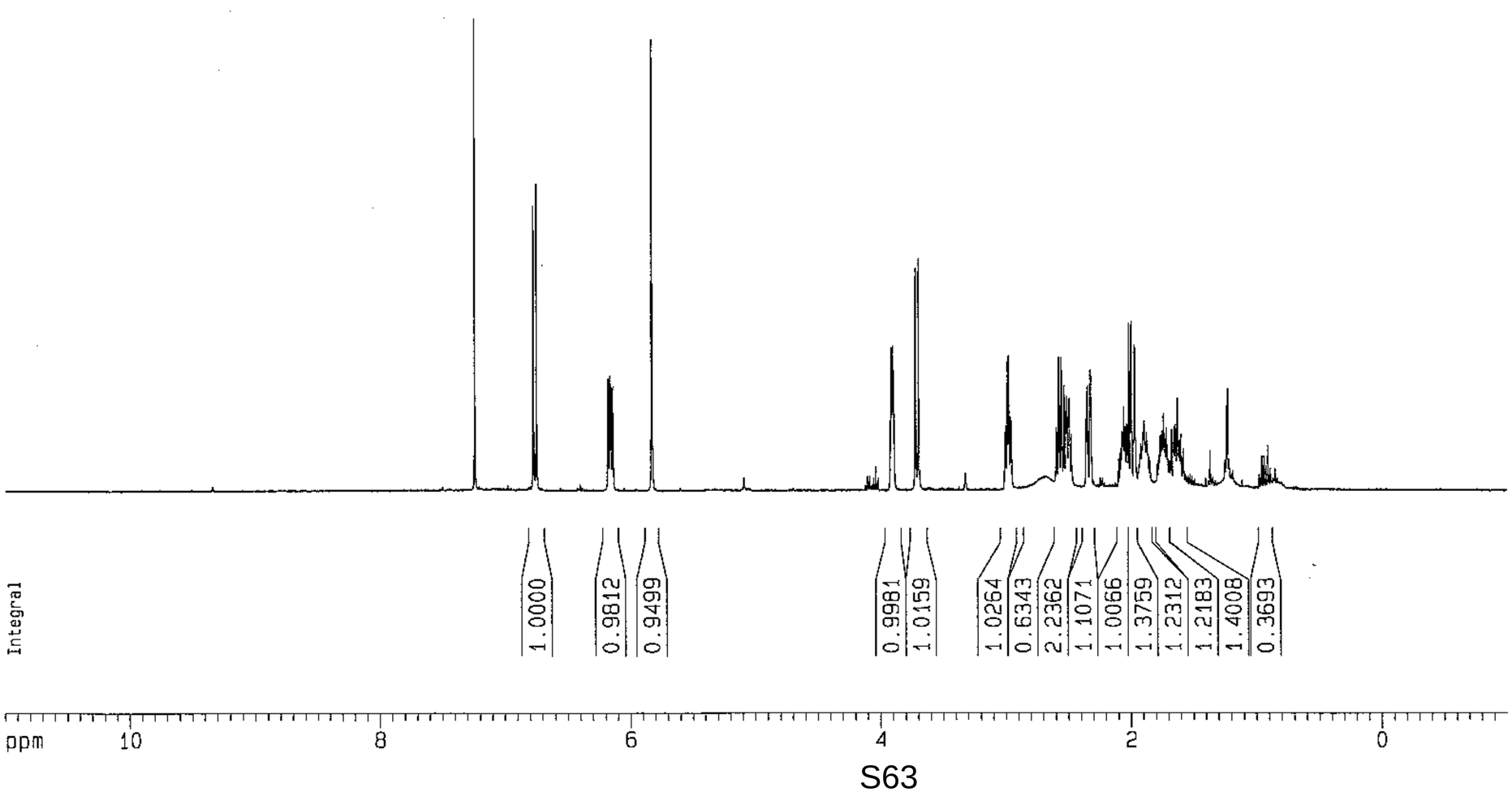
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

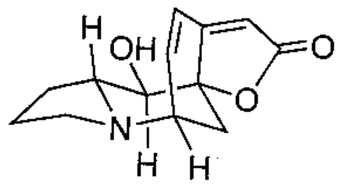
F2 - Processing parameters

SI	32768
SF	400.1300176 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

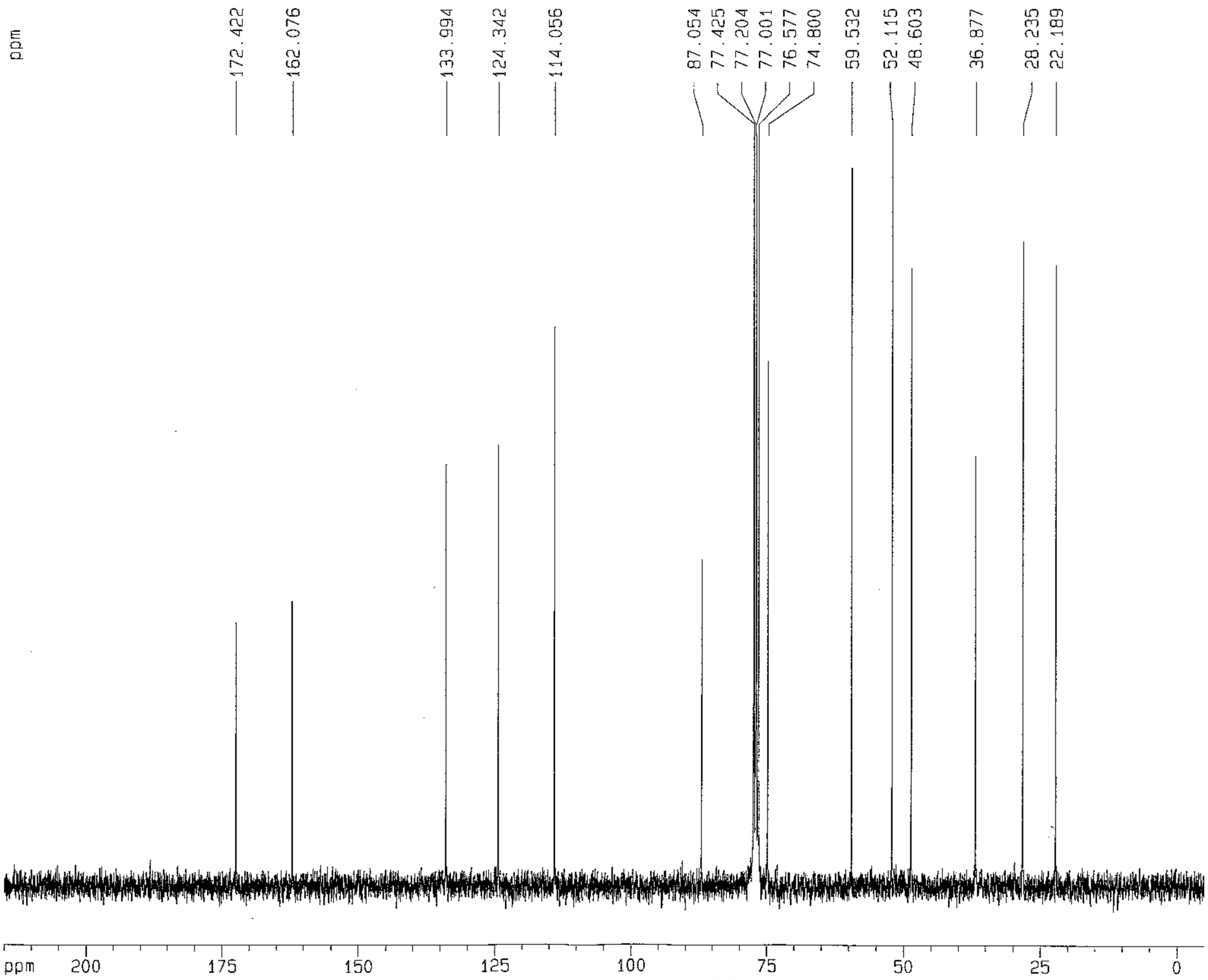
CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm





5

$C_{13}H_{15}NO_3$



Current Data Parameters

NAME	PL-Jan08-07
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080108
Time	18.54
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2632
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	1824.6
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	5.40 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

========== CHANNEL f2 ==========

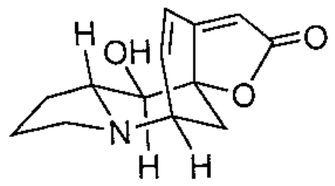
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

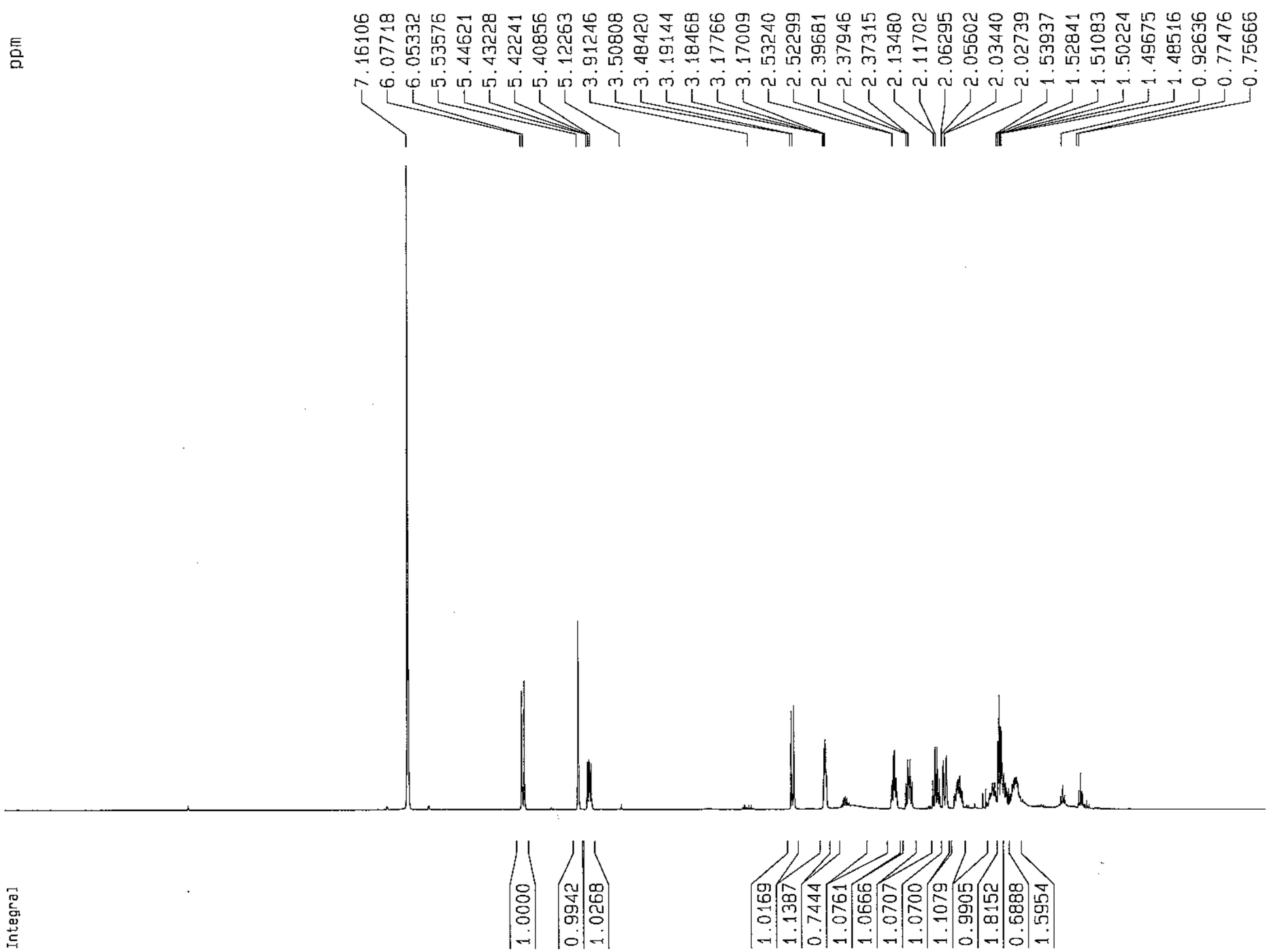
SI	32768
SF	75.4023752 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42615 Hz/cm



5



Current Data Parameters

NAME	PL-Apr05-08
EXPNO	1
PROCNO	3

F2 - Acquisition Parameters

Date_	20080405
Time	16.56
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

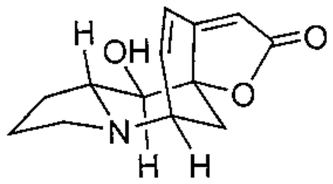
NUC1	1H
P1	6.45 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

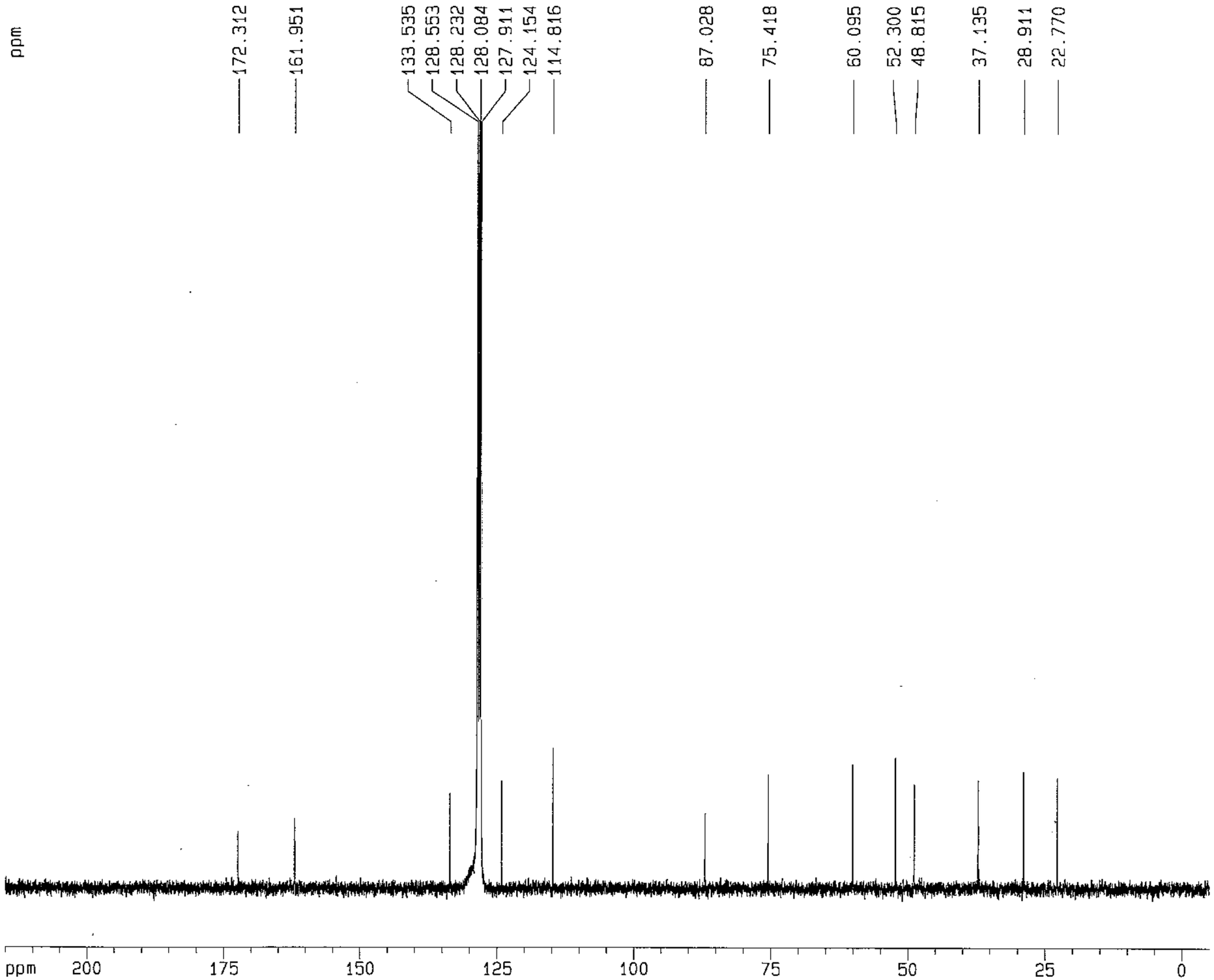
SI	32768
SF	400.1300436 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

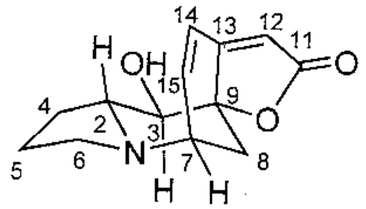
CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07802 Hz/cm



5

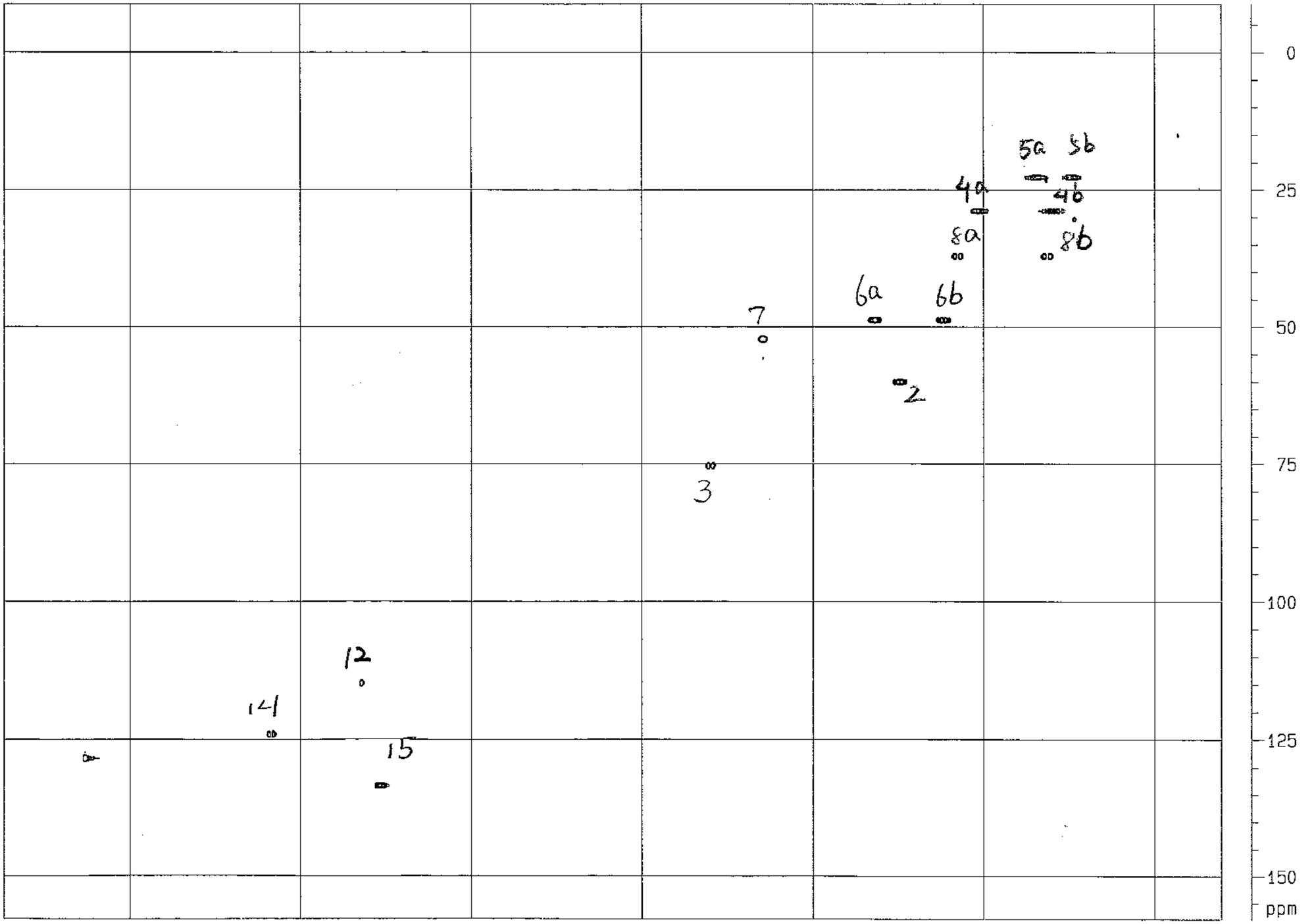



```
Current Data Parameters
NAME                PL-Apr06-08
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080406
Time                      11.15
INSTRUM                   spect
PROBHD       5 mm QNP 1H/1
PULPROG                 zgpg30
TD                       65536
SOLVENT                   C6D6
NS                        1191
DS                           4
SWH                18796.992 Hz
FIDRES              0.286819 Hz
AQ                  1.7433076 sec
RG                        2048
DW                      26.600 usec
DE                       6.00 usec
TE                      300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec

========== CHANNEL f1 ==========
NUC1                        13C
P1                         5.40 usec
PL1                       -6.00 dB
SFO1                75.4106357 MHz

========== CHANNEL f2 ==========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    115.00 usec
PL2                        0.00 dB
PL12                      20.00 dB
PL13                      20.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4023410 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
F1P                       215.000 ppm
F1                      16211.50 Hz
F2P                        -5.000 ppm
F2                       -377.01 Hz
PPMCM                  11.00000 ppm/cm
HZCM                  829.42578 Hz/cm
```



5

HMQC



Current Data Parameters

NAME	PL-Apr05-06
EXPNO	3
PROCNO	3

F2 - Acquisition Parameters

Date_	20060405
Time	18.12
INSTRUM	spect
PROBHD	5 mm BBi 1H-6
PULPROG	inv4gptp
TD	2048
SOLVENT	C606
NS	4
DS	60
SWH	2853.861 Hz
FIDRES	1.393487 Hz
AQ	0.3588586 sec
RG	18380.4
DW	175.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.99831298 sec
d2	0.00344828 sec
d4	0.00172414 sec
d11	0.03000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
d20	0.00052414 sec
d21	0.00224428 sec
IN0	0.00001490 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	6.81 usec
p2	13.62 usec
PL1	0.00 dB
SFO1	400.1316713 MHz

========== CHANNEL f2 ==========

CPDPRG2	garp
NUC2	13C
P3	17.00 usec
p4	34.00 usec
PCPD2	64.00 usec
PLE	-6.00 dB
PL12	6.30 dB
SFO2	100.6202332 MHz

========== GRADIENT CHANNEL ==========

GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPX1	17.00 %
GPX2	20.00 %
GPX3	25.00 %
GPY1	17.00 %
GPY2	20.00 %
GPY3	25.00 %
GPZ1	17.00 %
GPZ2	20.00 %
GPZ3	25.00 %
P16	1000.00 usec

F1 - Acquisition parameters

NDO	4
TD	256
SFO1	100.6202 MHz
FIDRES	65.541107 Hz
SW	166.751 ppm

F2 - Processing parameters

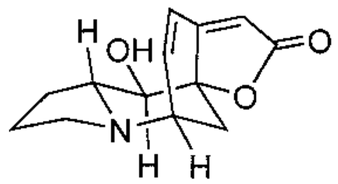
SI	2048
SF	400.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	1024
MC2	TPPI
SF	100.6127290 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

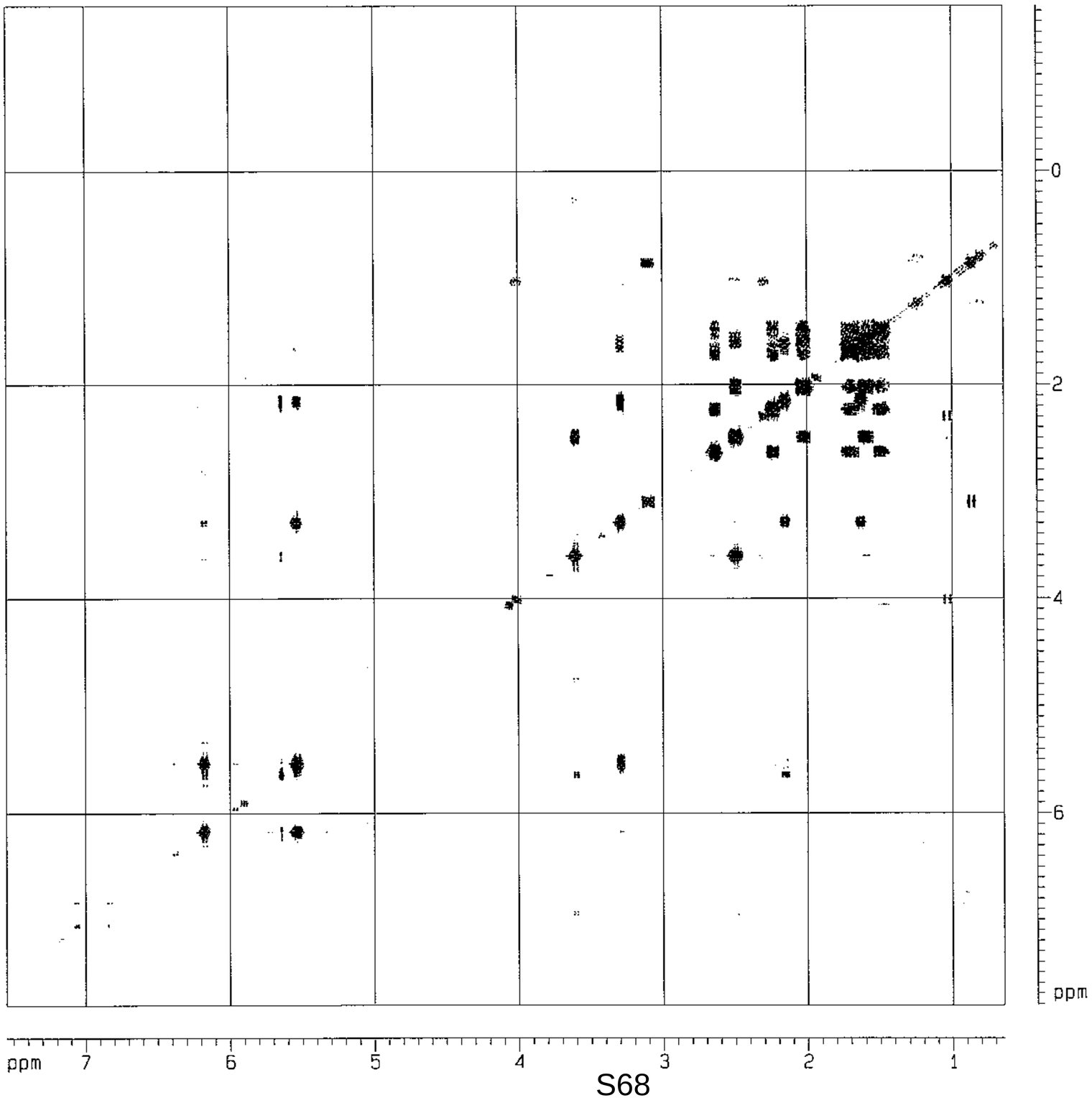
2D NMR plot parameters

CX2	20.00 cm
CX1	15.00 cm
F2PLO	7.743 ppm
F2LO	3098.28 Hz
F2PHI	0.611 ppm
F2HI	244.40 Hz
F1PLO	157.967 ppm
F1LO	15893.45 Hz
F1PHI	-6.797 ppm
F1HI	-885.07 Hz
F2PPMCM	0.35562 ppm/cm
F2HZCM	142.69406 Hz/cm
F1PPMCM	11.11756 ppm/cm
F1HZCM	1116.56624 Hz/cm



5

COSY



Current Data Parameters

NAME	PL-Apr05-08
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20080406
Time	0.49
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	ajb.cosygsmftp
TD	2048
SOLVENT	C6D6
NS	4
DS	8
SWH	2853.881 Hz
FIDRES	1.393497 Hz
AQ	0.3588596 sec
RG	2896.3
DW	175.200 usec
DE	6.00 usec
TE	300.0 K
D0	0.00000300 sec
D1	4.00000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
d20	0.00120300 sec
IN0	0.00009360 sec

CHANNEL f1

NUC1	1H
P1	6.81 usec
P2	13.62 usec
PL1	0.00 dB
SFO1	400.1316713 MHz

GRADIENT CHANNEL

P16	1000.00 usec

F1 - Acquisition parameters

ND0	2
TD	650
SFO1	400.1317 MHz
FIDRES	8.218277 Hz
SW	13.350 ppm

F2 - Processing parameters

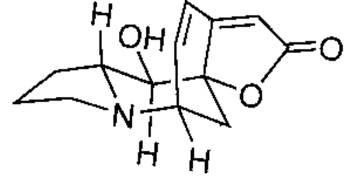
SI	2048
SF	400.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	1024
MC2	TPPI
SF	400.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

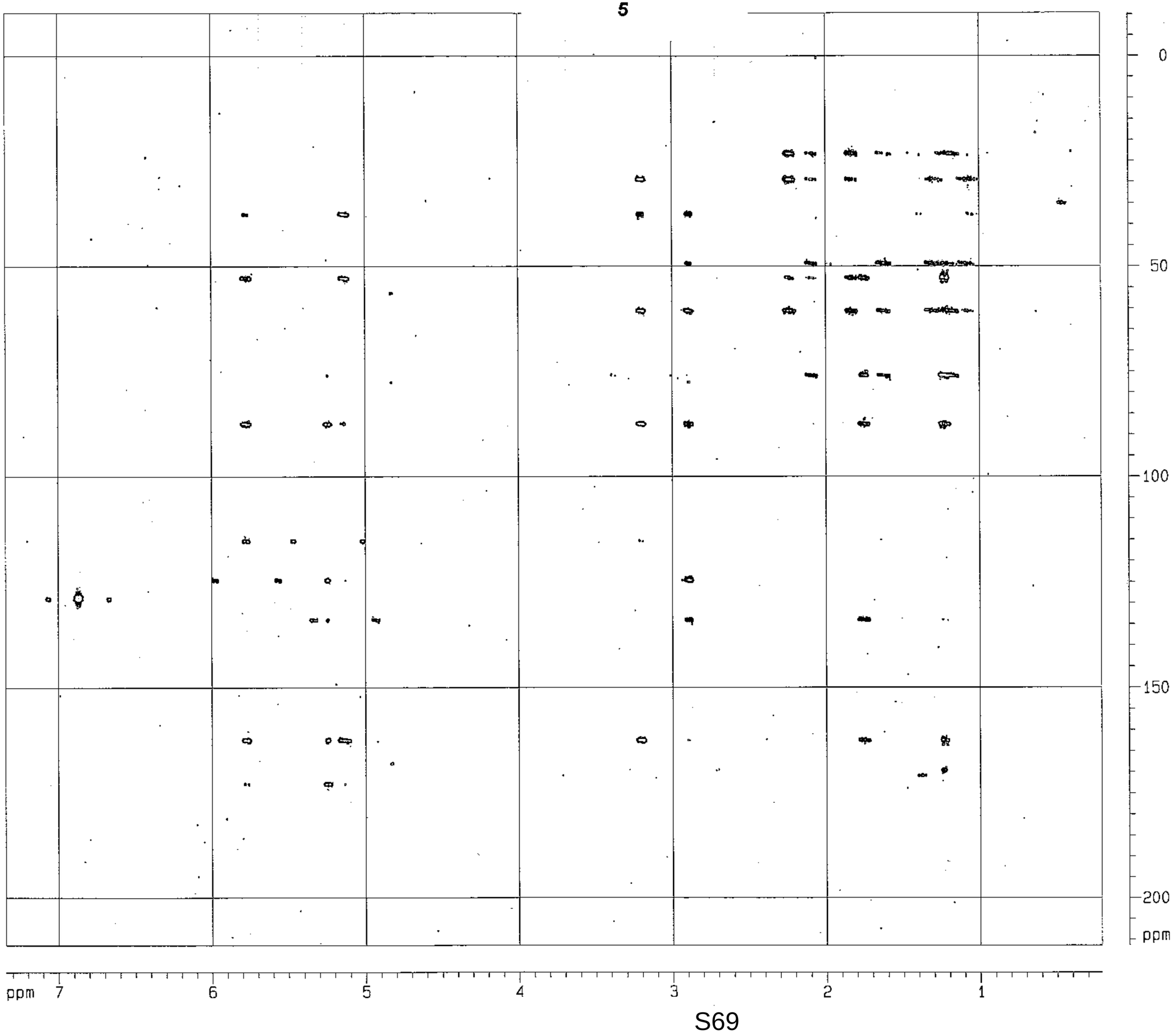
2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PL0	7.548 ppm
F2L0	3020.25 Hz
F2PHI	0.642 ppm
F2HI	256.94 Hz
F1PL0	7.814 ppm
F1L0	3126.80 Hz
F1PHI	-1.546 ppm
F1HI	-618.78 Hz
F2PPMCM	0.46040 ppm/cm
F2HZCM	184.22026 Hz/cm
F1PPMCM	0.62408 ppm/cm
F1HZCM	249.70508 Hz/cm



5

HMBC



Current Data Parameters

NAME	PL-Apr05-08
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20080406
Time	5.00
INSTRUM	spect
PROBHD	5 mm BBI 1H-B
PULPROG	inv4gslplrnd
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	2853.881 Hz
FIDRES	1.393497 Hz
AQ	0.3588596 sec
RG	8192
DW	175.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.50000000 sec
d2	0.00344828 sec
D6	0.06500000 sec
d13	0.00000300 sec
D16	0.00020000 sec
IN0	0.00002240 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	6.81 usec
p2	13.62 usec
PL1	0.00 dB
SFO1	400.1316713 MHz

========== CHANNEL f2 ==========

NUC2	13C
P3	17.00 usec
PL2	-6.00 dB
SFO2	100.6227903 MHz

========== GRADIENT CHANNEL =======

P16	1000.00 usec

F1 - Acquisition parameters

NDO	2
TD	512
SFO1	100.6228 MHz
FIDRES	43.596539 Hz
SW	221.833 ppm

F2 - Processing parameters

SI	2046
SF	400.1301597 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

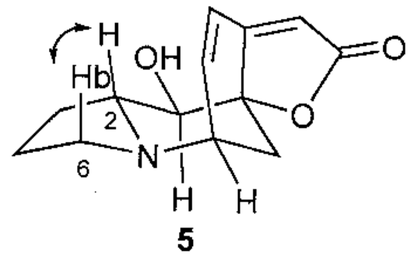
F1 - Processing parameters

SI	1024
MC2	QF
SF	100.6126736 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

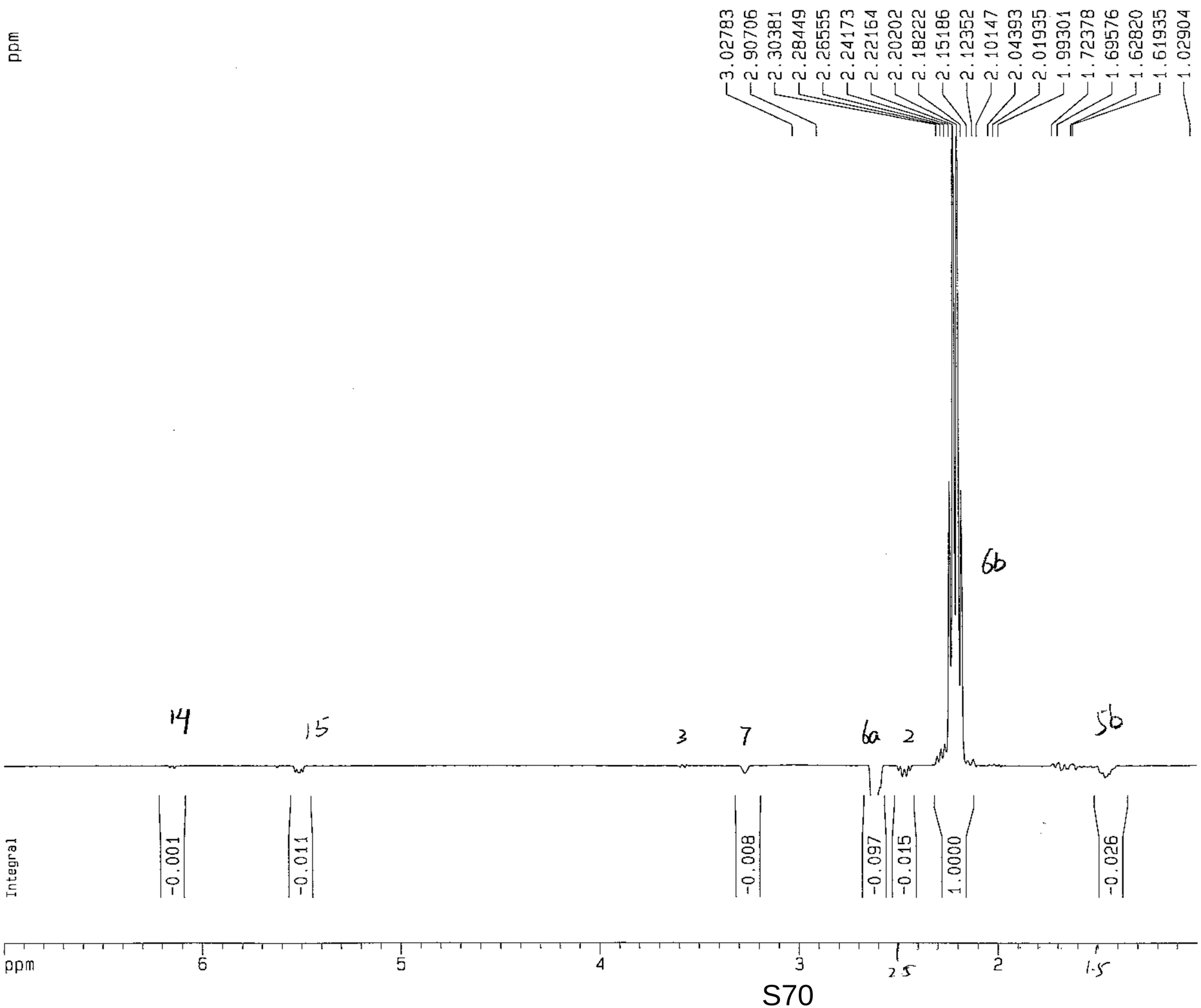
CX2	20.00 cm
CX1	17.00 cm
F2PLO	7.344 ppm
F2LO	2938.57 Hz
F2PHI	0.212 ppm
F2HI	84.69 Hz
F1PLO	211.478 ppm
F1LO	21277.37 Hz
F1PHI	-10.377 ppm
F1HI	-1044.06 Hz
F2PPMCM	0.35662 ppm/cm
F2HZCM	142.69406 Hz/cm
F1PPMCM	13.05030 ppm/cm
F1HZCM	1313.02515 Hz/cm

7ppm- 1ppm



5

NOESY




```
Current Data Parameters
NAME          PL-Apr05-08
EXPNO                   4
PROCNO                 11

F2 - Acquisition Parameters
Date_           20080405
Time               18.54
INSTRUM            spect
PROBHD     5 mm BBI 1H-B
PULPROG       noesygptp
TD                  2048
SOLVENT             C6D6
NS                     8
DS                     8
SWH           2853.881 Hz
FIDRES        1.393497 Hz
AQ            0.3588596 sec
RG                   128
DW              175.200 usec
DE                6.00 usec
TE               300.0 K
d0           0.00000300 sec
D1           4.00000000 sec
D8           0.80000001 sec
D16          0.00020000 sec
d20          0.39880002 sec
IN0          0.00012496 sec

============ CHANNEL f1 =============
NUC1                 1H
P1                6.81 usec
P2               13.62 usec
PL1               0.00 dB
SFO1       400.1316713 MHz

============ GRADIENT CHANNEL ========
GPNAM1        sine.100
GPNAM2        sine.100
GPX1              0.00 %
GPX2              0.00 %
GPY1              0.00 %
GPY2              0.00 %
GPZ1             40.00 %
GPZ2            -40.00 %
P16           1000.00 usec

F2 - Processing parameters
SI                2048
SF         400.1300092 MHz
WDW              QSINE
SSB                  2
LB                0.00 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
F1P               7.000 ppm
F1             2800.91 Hz
F2P               1.000 ppm
F2              400.13 Hz
PPMCM            0.30000 ppm/cm
HZCM           120.03900 Hz/cm
```