Supporting Information

Enantiospecific Synthesis of Pseudo-acarviosin as a Potential Anti-diabetic Agent

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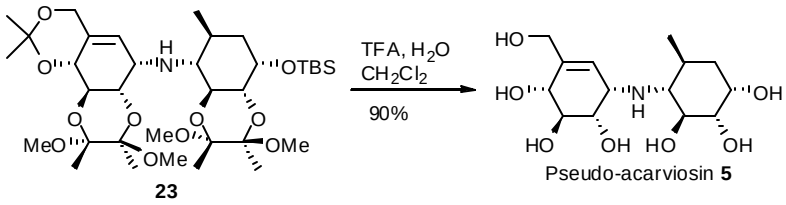
Contents

Experimental Procedures (synthetic chemistry)

Melting points were uncorrected. Optical rotations were obtained with a polarimeter operating at 589nm. Infrared spectra (IR) were recorded with a FT-IR spectrophotometer as thin film on potassium bromide discs. Nuclear magnetic resonance (NMR) spectra were measured at 300.13 MHz (^{1}H) or at 75.47 MHz (^{13}C) in $CDCl_3$ solutions, unless stated otherwise. All chemical shifts were recorded in ppm relative to tetramethylsilane (δ = 0.0). Spin-spin coupling constants (*J* value) recorded in Hz were measured directly from the spectra. All reactions were monitored by analytical thin-layer chromatography (TLC) on aluminium-precoated plates of silica gel with detection by spraying with 5% (w/v) dodecamolybdophosphoric acid in ethanol or 5% (w/v) ninhydrin in ethanol and subsequent heating. Silica gel (230–400 mesh) was used for flash chromatography. All reagents and solvents were general reagent grade unless otherwise stated. 2-Propanol was dried by sodium and distilled from its sodium salt under nitrogen. DMF was dried by magnesium sulfate, filtered and was then freshly distilled under reduced pressure. Acetonitrile was freshly distilled from P_2O_5 under nitrogen. THF was freshly distilled from Na/benzophenone ketyl under nitrogen. Dichloromethane and chloroform were freshly distilled from P_2O_5 under nitrogen. Diethyl ether was freshly distilled from K/benzophenone ketyl under nitrogen. Other reagents were purchased from commercial suppliers and were used without purification.

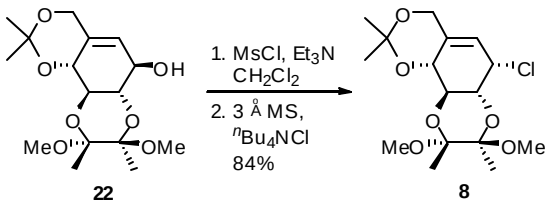
Pseudo-acarviosin 5.



23 Pseudo-acarviosin **5**

To a solution of amine **23** (985 mg, 1.40 mmol) in CH_2Cl_2 (6 mL) was added trifluoroacetic acid (TFA) (2 mL) and water (0.5 mL) at room temperature. The resultant solution was stirred for 7 days at room temperature. Concentration of the solution followed by flash chromatography ($CHCl_3$:MeOH, 2:1) yielded pseudo-acarviosin **5** (403 mg, 90%) as a colorless oil: $[\alpha]_D^{20}$ +43.7 (*c* 0.99, MeOH); R_f = 0.4 ($CHCl_3$:MeOH:32% aq. NH_3, 1.6:1.2:0.4); ^{1}H NMR (CD_3OD) δ 1.04 (3H, d, *J* = 6.3 Hz), 1.27–1.37 (1H, m), 1.80 (1H, dt, *J* = 14.4, 3.6 Hz), 2.12–2.23 (1H, m), 2.82 (1H, t, *J* = 10.5 Hz), 3.29 (1H, dd, *J* = 9.0, 3.0 Hz), 3.73–3.89 (5H, m), 4.01 (1H, brs), 4.08 & 4.18 (2H, ABq, *J* = 15.0 Hz), 5.74 (1H, d, *J* = 3.3 Hz); ^{13}C NMR (CD_3OD) δ 18.6 (CH_3), 28.2 (CH), 38.6 (CH_2), 56.9 (CH), 62.9 (CH_2), 67.6 (CH), 68.4 (CH), 69.8 (CH), 70.9 (CH), 71.5 (CH), 73.0 (CH), 76.3 (CH), 115.5 (CH), 147.9 (C); MS (FAB) *m/z* (relative intensity) 320 ($[M+H]^+$, 21), 93 (100); HRMS (FAB) calcd for $C_{14}H_{25}O_7N$ $[M+H]^+$ 320.1704, found 320.1708.

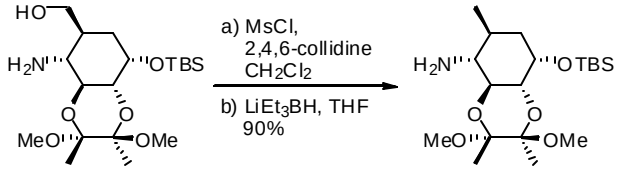
Allylic chloride 8.



22 **8**

To a solution of the ß-alcohol **22** (437 mg, 1.33 mmol) in CH_3CN (15 mL) and Et_3N (0.46 mL, 3.30 mmol) was added methanesulfonyl chloride (MsCl) (0.2 mL, 2.58 mmol) at 0 °C and the solution was stirred for 1 h at 0 °C. Then 3Å molecular sieves (ca. 1.5 g) was added and the mixture was stirred for 15 minute. Tetra-*n*-butylammonium chloride (1.8 g, 6.47 mmol) was then added. After stirred for 48 h at room temperature, the reaction mixture was diluted with diethyl ether (30 mL) and filtered through a thin pad of silica gel. The residue was washed with diethyl ether. Concentration of the filtrate followed by flash chromatography (hexane:Et_2O, 3:1) yielded allylic chloride **8** (391 mg, 84%) as a colorless oil: $[\alpha]_D^{20}$ –12.8 (*c* 1.20, $CHCl_3$); R_f = 0.43 (hexane:Et_2O, 1:1); IR (thin film) 2937, 1355, 1174, ; ^{1}H NMR δ 1.34 (3H, s), 1.37 (3H, s), 1.40 (3H, s), 1.50 (3H, s), 3.28 (3H, s), 3.31 (3H, s), 3.87 (1H, dd, *J* = 10.9, 4.2 Hz), 4.13 (1H, dd, *J* = 10.9, 8.1 Hz), 4.16 (1H, d, *J* = 14.1 Hz), 4.41 (1H, dd, *J* = 14.1, 1.5 Hz), 4.50 (1H, d, *J* = 8.1 Hz), 4.55 (1H, t, *J* = 4.2 Hz), 5.61 (1H, d, *J* = 4.2 Hz); ^{13}C NMR δ 18.0 (CH_3), 18.2 (CH_3), 20.7 (CH_3), 28.3 (CH_3), 48.5 (CH_3), 48.7 (CH_3), 55.6 (CH), 63.0 (CH_2), 66.7 (CH), 67.7 (CH), 70.6 (CH), 99.2 (C), 99.8 (C), 100.1 (C), 119.4 (CH), 135.7 (C); MS (ESI) *m/z* (relative intensity) 371 ([M+Na]$^+$, 25), 315 (12), 186 (100); HRMS (ESI) calcd for $C_{16}H_{25}O_6Cl_1$ [M+Na]$^+$ 371.1232, found 371.1239.

Pseudo-4-aminopyranose 9.

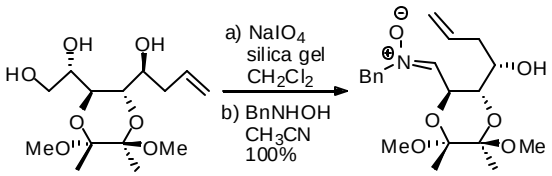


14 **9**

Methanesulfonyl chloride (MsCl) (0.37 mL, 4.84 mmol) and 2,4,6-collidine (0.98 mL, 7.45 mmol) were added to a solution of amine **14** (1.51 g, 3.72 mmol) in CH_2Cl_2 (100 mL) under N_2 at –30 °C. The reaction mixture was stirred for 18 h and quenched with saturated $NaHCO_3$ solution. The aqueous phase was extracted with EtOAc (2 × 50 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure to afford the mesylate as a colorless oil. To a solution of the mesylate in THF (100 mL) was added a 1M THF solution of $LiEt_3BH$ (7.40 mL, 7.45 mmol) dropwise at –30 °C under N_2. The reaction mixture was stirred for 1 h, then raised to room temperature, and stirred for a further 2 h. Water was then added slowly at 0 °C to destroy the excess of hydride and the

aqueous phase was extracted with EtOAc (2 × 50 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography (hexane:EtOAc, 1:1) to afford pseudo-4-aminopyranose **9** (1.38 g, 90%) as a colorless oil: $[\alpha]_D^{20}$ –112.5 (*c* 1.19, $CHCl_3$); R_f 0.40 ($CHCl_3$:MeOH, 15:1); IR (thin film) 3379, 2951, 1136, 1039, 828, 773 cm^{-1}; ^1H NMR δ 0.03 (3H, s), 0.08 (3H, s), 0.87 (9H, s), 0.99 (3H, d, *J* = 6.6 Hz), 1.19–1.31 (7H, m), 1.65 (1H, dt, *J* = 14.1, 3.9 Hz), 1.80–1.87 (1H, m), 2.14 (2H, br s), 2.43 (1H, t, *J* = 9.9 Hz), 3.21–3.22 (6H, 2s), 3.36 (1H, dd, *J* = 9.9, 2.4 Hz), 3.73 (1H, t, *J* = 9.9 Hz), 3.94–3.95 (1H, m); ^1H NMR ($CDCl_3$-D_2O) δ 0.03 (3H, s), 0.08 (3H, s), 0.87 (9H, s), 0.98 (3H, d, *J* = 6.6 Hz), 1.16–1.31 (7H, m), 1.65 (1H, dt, *J* = 14.1, 3.9 Hz), 1.79–1.90 (1H, m), 2.41 (1H, t, *J* = 9.9 Hz), 3.21–3.22 (6H, 2s), 3.36 (1H, dd, *J* = 10.2, 2.7 Hz), 3.74 (1H, t, *J* = 9.9 Hz), 3.94–3.95 (1H, m); ^{13}C NMR δ –4.8 (CH_3), –4.3 (CH_3), 17.9 (CH_3), 18.2 (CH_3), 18.6 (CH_3), 18.6 (C), 26.2 (CH_3), 32.2 (CH), 39.8 (CH_2), 47.9 (CH_3), 48.1 (CH_3), 58.1 (CH), 69.1 (CH), 70.6 (CH), 72.1 (CH), 99.4 (C), 99.6 (C); MS (ESI) *m/z* (relative intensity) 390 ($[MH]^+$, 100), 358 (8); HRMS (ESI) calcd for $C_{19}H_{39}O_5NSi$ $[MH]^+$ 390.2670, found 390.2669.

Nitrone 11.

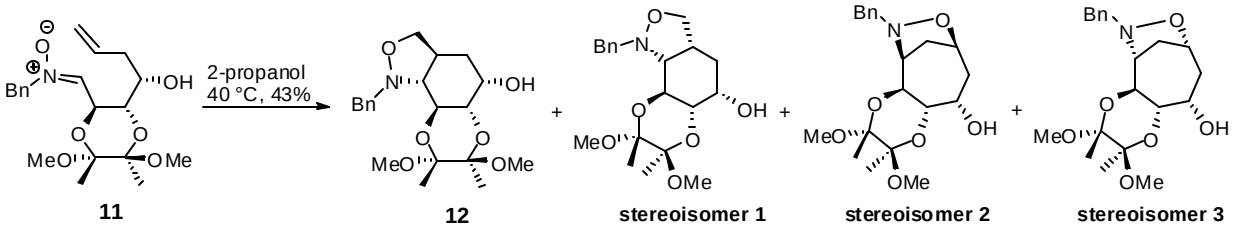


10 **11**

$NaIO_4$ (1.45 g, 20.3 mmol) was dissolved in a minimum amount of hot water (~80 °C) and to this solution was added silica gel (230–400 mesh, 10 g) with vigorous swirling and shaking. The mixture was suspended in CH_2Cl_2 (20 mL) and then a solution of alkene **10** (2.08 g, 6.78 mmol) in CH_2Cl_2 (10 mL) was added. After vigorous stirring at room temperature for 1 h, the mixture was filtered. The filtrate was concentrated under reduced pressure to give an aldehyde as a colorless oil. *N*-benzylhydroxylamine hydrochloride (1.19 g, 7.46 mmol) and $NaHCO_3$ (854 mg, 10.2 mmol) were added to a solution of the aldehyde in CH_3CN (80 mL). After stirring at room temperature for 30 min the reaction mixture was partitioned between EtOAc (50 mL) and water (50 mL). The aqueous layer was extracted with EtOAc (2 × 50 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and filtered. Concentration of the filtrate followed by flash chromatography (hexane:EtOAc, 1:2) gave nitrone **11** (2.08 g, 100% overall yield from **10**) as a colorless oil: $[\alpha]_D^{20}$ –112.8 (*c* 3.42, $CHCl_3$); R_f 0.48 (EtOAc); IR (thin film) 3268, 2946, 1209, 1128, 1031, 890, 704 cm^{-1}; ^1H NMR δ 1.26–1.29 (6H, 2s), 2.10 (1H, ddd, *J* = 14.7, 7.5, 0.9 Hz), 2.54 (1H, dtd, *J* = 14.1, 3.6, 1.8 Hz), 3.23–3.24 (6H, 2s), 3.47 (1H, dd, *J* = 9.6, 7.8 Hz), 3.74 (1H, td, *J* = 8.1, 3.6 Hz), 4.88 (2H, s), 4.96 (1H, dd, *J* = 9.9, 6.6 Hz), 5.04–5.10 (2H, m), 5.87 (1H, ddt, *J* = 17.1, 10.5, 6.9

Hz), 6.78 (1H, d, J = 6.6 Hz), 7.31–7.42 (5H, m); ^{13}C NMR δ 17.6, 17.8, 37.7, 48.5, 48.8, 67.4, 69.7, 70.8, 71.9, 98.4, 98.6, 117.1, 129.4, 129.7, 130.2, 131.5, 135.7, 139.4; MS (FAB) *m/z* (relative intensity) 380 ([MH]$^+$, 100), 348 (77), 91 (100); HRMS (FAB) calcd for $C_{20}H_{29}O_6N$ [MH]$^+$ 380.2068, found 380.2075.

Isoxazolidine 12, stereoisomer 1, stereoisomer 2 and stereoisomer 3.



A solution of nitrone **11** (2.22 g, 5.84 mmol) in 2-propanol (90 mL) was heated at 40 ^{o}C for 9 days. The solvent was removed under reduced pressure and flash chromatography (hexane:EtOAc, 1:2) of the residue gave firstly a mixture of **stereoisomer 3** and **stereoisomer 1** and secondly a mixture of isoxazolidine **12** and **stereoisomer 2** as colorless oils. Further flash chromatography (hexane:Et_2O, 1:5) of the mixture of **12** and **stereoisomer 2** afforded firstly **stereoisomer 2** (651 mg, 29%) as a colorless oil and secondly isoxazolidine **12** (952 mg, 43%) as a white solid. Flash chromatography ($CHCl_3$:EtOAc, 2:1) of the mixture of **stereoisomer 1** and **stereoisomer 3** afforded firstly **stereoisomer 3** (151 mg, 7%) as a white solid and secondly **stereoisomer 1** (412 mg, 19%) as a colorless oil.

Data for **12**: mp 193–194 ^{o}C; $[\alpha]_D^{20}$ –24.4 (*c* 1.77, $CHCl_3$); R_f 0.27 (Et_2O); IR (thin film) 3459, 2926, 1455, 1376, 1140, 1037, 755 cm^{-1}; ^{1}H NMR δ 1.30–1.34 (6H, 2s), 1.48 (1H, dd, J = 13.2, 11.7 Hz), 2.07 (1H, dt, J = 13.5, 3.3 Hz), 2.60–2.67 (2H, m), 2.92–3.04 (1H, m), 3.28–3.32 (6H, 2s), 3.61 (1H, dd, J = 11.1, 6 Hz), 3.71 (1H, dd, J = 9.3, 2.7 Hz), 3.84 (1H, d, J = 14.4 Hz), 3.98 (1H, t, J = 6.6 Hz), 4.11 (1H, q, J = 3 Hz), 4.27 (1H, t, J = 9.6 Hz), 4.50 (1H, d, J = 14.1 Hz), 7.23–7.40 (5H, m); ^{1}H NMR ($CDCl_3$-D_2O) δ 1.30–1.34 (6H, 2s), 1.48 (1H, td, J = 13.5, 2.4 Hz), 2.07 (1H, dt, J = 13.8, 3.3 Hz), 2.62 (2H, t, J = 10.2 Hz), 2.90–3.04 (1H, m), 3.27–3.31 (6H, 2s), 3.61 (1H, dd, J = 11.1, 6.3 Hz), 3.71 (1H, dd, J = 9.6, 3 Hz), 3.83 (1H, d, J = 14.1 Hz), 3.97 (1H, t, J = 6.9 Hz), 4.09 (1H, q, J = 2.7 Hz), 4.25 (1H, t, J = 9.6 Hz), 4.48 (1H, d, J = 14.4 Hz), 7.22–7.40 (5H, m); ^{13}C NMR δ 18.1 (CH_3), 18.2 (CH_3), 30.2 (CH_2), 42.0 (CH), 48.4 (CH_3), 64.9 (CH_2), 68.5 (CH), 69.4 (CH), 69.4 (CH_2), 70.4 (CH), 73.1 (CH), 99.8 (C), 100.8 (C), 127.5 (CH), 128.8 (CH), 129.3 (CH), 138.7 (C); MS (ESI) *m/z* (relative intensity) 402 ([M+Na]$^+$, 100), 380 ([MH]$^+$, 39); HRMS (ESI) calcd for $C_{20}H_{29}O_6N$ [M+Na]$^+$ 402.1887, found 402.1891; Anal. Calcd for $C_{20}H_{29}O_6N$: C, 63.31; H, 7.70; N, 3.69, found: C, 62.89; H, 7.66; N, 3.56.

Data for **stereoisomer 1**: $[\alpha]_D^{20}$ –85.0 (*c* 3.03, $CHCl_3$); R_f 0.27 ($CHCl_3$:EtOAc, 2:1); IR (thin film) 3452, 2949, 1121, 1035, 751 cm^{-1}; ^1H NMR δ 1.29–1.31 (6H, 2s), 1.81 (1H, ddd, *J* = 15.3, 6.3, 4.5 Hz), 2.07 (1H, dt, *J* = 15.3, 3.3 Hz), 2.62 (1H, br s), 2.88 (1H, br s), 3.13 (1H, t, *J* = 7.6 Hz), 3.25 (3H, s), 3.37 (3H, s), 3.56 (1H, dd, *J* = 11.1, 3.3 Hz), 3.93–4.18 (6H, m), 7.21–7.42 (5H, m); ^{13}C NMR δ 17.9 (CH_3), 18.2 (CH_3), 28.1 (CH_2), 38.3 (CH), 48.3 (CH_3), 61.9 (CH_2), 66.0 (CH), 67.1 (CH), 67.9 (CH), 70.7 (CH), 71.7 (CH_2), 99.6 (C), 100.1 (C), 127.4 (CH), 128.5 (CH), 129.0 (CH), 138.4 (C); MS (ESI) *m/z* (relative intensity) 402 ($[M+Na]^+$, 100), 380 ($[MH]^+$, 4); HRMS (ESI) calcd for $C_{20}H_{29}O_6N$ $[M+Na]^+$ 402.1887, found 402.1891.

Data for **stereoisomer 2**: $[\alpha]_D^{20}$ –146.8 (*c* 2.44, $CHCl_3$); R_f 0.47 (Et_2O); IR (thin film) 3445, 2947, 1454, 1377, 1125, 1040, 754 cm^{-1}; ^1H NMR δ 1.27–1.29 (6H, 2s), 1.83 (1H, dd, *J* = 15.6, 2.7 Hz), 2.03 (1H, dd, *J* = 15.6, 4.2 Hz), 2.29 (1H, br s), 2.43 (1H, d, *J* = 12.6 Hz), 2.60 (1H, br s), 3.15 (3H, s), 3.23 (3H, s), 3.38–3.41 (1H, m), 3.88 (2H, br s), 3.99 (1H, d, *J* = 5.7), 4.06–4.16 (2H, m), 4.53 (1H, d, *J* = 8.4 Hz), 7.22–7.46 (5H, m); ^1H NMR ($CDCl_3$-D_2O) δ 1.27–1.28 (6H, 2s), 1.83 (1H, dd, *J* = 15.9, 2.7 Hz), 2.02 (1H, ddd, *J* = 15.9, 5.7, 1.5 Hz), 2.31 (1H, dt, *J* = 12.6, 8.1 Hz), 2.47 (1H, d, *J* = 12.9 Hz), 3.16 (3H, s), 3.22 (3H, s), 3.40–3.44 (1H, dd, *J* = 7.5, 2.7 Hz), 3.83 (1H, d, *J* = 13.2 Hz), 3.89 (1H, dd, *J* = 9.3, 2.7 Hz), 3.96–4.04 (2H, m), 4.12 (1H, d, *J* = 9.3 Hz), 4.55 (1H, d, *J* = 9.3 Hz), 7.23–7.34 (3H, m), 7.41–7.43 (2H, m); ^{13}C NMR δ 17.9 (CH_3), 18.0 (CH_3), 31.1 (CH_2), 38.4 (CH_2), 48.9 (CH_3), 48.3 (CH_3), 62.6 (CH_2), 65.0 (CH), 69.3 (CH), 70.8 (CH), 75.7 (CH), 99.4 (C), 99.6 (C), 127.5 (CH), 128.5 (CH), 129.4 (CH), 137.9 (C); MS (ESI) *m/z* (relative intensity) 402 ($[M+Na]^+$, 100), 348 (15); HRMS (ESI) calcd for $C_{20}H_{29}O_6N$ $[M+Na]^+$ 402.1887, found 402.1881; Anal. Calcd for $C_{20}H_{29}O_6N$: C, 63.31; H, 7.70; N, 3.69, found: C, 62.75; H, 7.64; N, 3.51.

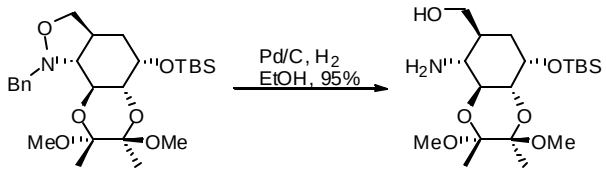
Data for **stereoisomer 3**: mp 201–202 °C; $[\alpha]_D^{20}$ –115.0 (*c* 1.97, $CHCl_3$); R_f 0.50 ($CHCl_3$:EtOAc, 2:1); IR (thin film) 3211, 2949, 2901, 1469, 1375, 1120, 1038, 940, 761, 705 cm^{-1}; ^1H NMR δ 1.13 (3H, s), 1.24 (3H, s), 1.61 (1H, ddd, *J* = 15.3, 3.6, 2.4 Hz), 2.23–2.36 (3H, m), 2.57 (3H, s), 3.16 (3H, s), 3.24 (1H, d, *J* = 7.8 Hz), 3.71 (2H, s), 2.79 (1H, d, *J* = 12 Hz), 3.87 (1H, t, *J* = 3 Hz), 4.33 (1H, d, *J* = 12 Hz), 4.67 (1H, br s), 7.24–7.39 (5H, m); ^{13}C NMR δ 17.7 (CH_3), 17.8 (CH_3), 32.8 (CH_2), 39.2 (CH_2), 47.2 (CH_3), 47.9 (CH_3), 63.7 (CH_2), 64.3 (CH), 70.9 (CH), 71.3 (CH), 72.4 (CH), 77.5 (CH), 99.9 (C), 100.3 (C), 128.2 (CH), 129.0 (CH), 129.9 (CH), 135.7 (C); MS (ESI) *m/z* (relative intensity) 402 ($[M+Na]^+$, 100), 380 ($[MH]^+$, 20); HRMS (ESI) calcd for $C_{20}H_{29}O_6N$ $[M+Na]^+$ 402.1887, found 402.1885; Anal. Calcd for $C_{20}H_{29}O_6N$: C, 63.31; H, 7.70; N, 3.69, found: C, 63.03; H, 7.69; N, 3.60.

Silyl ether 13.

12 **13**

A solution of cyclohexane **12** (490 mg, 1.29 mmol), imidazole (264 mg, 3.87 mmol) and *tert*-butyl dimethyl silyl chloride (TBSCl) (389 mg, 258 mmol) in dry DMF (2 mL) was stirred at room temperature for 24 h. The mixture was quenched with saturated $NaHCO_3$ solution and the aqueous phase was extracted with Et_2O (2 × 20 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography (hexane:Et_2O, 4:1) to afford silyl ether **13** (637 mg, 100%) as a colorless oil: $[\alpha]_D^{20}$ –3.34 (*c* 3.34, $CHCl_3$); R_f 0.39 (hexane:Et_2O, 4:1); IR (thin film) 2949, 2855, 1201, 1134, 1091, 1050, 830, 776 cm^{-1}; ^1H NMR δ 0.07 (3H, s), 0.13 (3H, s), 0.89 (9H, s), 1.27–1.29 (6H, 2s), 1.47 (1H, td, *J* = 12.9, 1.8 Hz), 1.83 (1H, dt, *J* = 13.2, 3.6 Hz), 2.62 (1H, t, *J* = 10.2 Hz), 2.95–3.07 (1H, m), 3.26–3.28 (6H, 2s), 3.56 (1H, dd, *J* = 9.6, 2.7 Hz), 3.63 (1H, dd, *J* = 11.1, 6.3 Hz), 3.83 (1H, d, *J* = 14.4 Hz), 3.97 (1H, t, *J* = 6.6 Hz), 4.08 (1H, q, *J* = 2.4 Hz), 4.24 (1H, t, *J* = 9.6 Hz), 4.52 (1H, d, *J* = 14.1 Hz), 7.22–7.40 (5H, m); ^{13}C NMR δ –4.9, –4.3, 18.0, 18.1, 18.6, 26.1, 32.4, 41.9, 48.0, 48.1, 65.2, 69.2, 69.5, 70.8, 73.1, 99.3, 100.1, 127.4, 128.7, 129.3, 139.0; MS (ESI) *m/z* (relative intensity) 494 ($[MH]^+$, 100); HRMS (ESI) calcd for $C_{26}H_{43}O_6NSi$ $[MH]^+$ 494.2932, found 494.2938.

Amine 14.

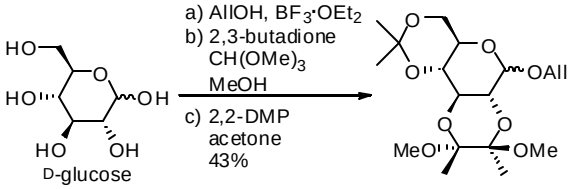


13 **14**

To a solution of silyl ether **13** (1.85 g, 3.75 mmol) in EtOH (100 mL) was added 10% Pd-on-charcoal (199 mg, 0.188 mmol) and the mixture was stirred under an atmosphere of H_2 (balloon). After stirring at room temperature under H_2 for 8 h, the mixture was filtered and the filtrate was concentrated. Flash chromatography ($CHCl_3$:MeOH, 8:1) of the residue gave amine **14** (1.44 g, 95%) as a colorless oil: $[\alpha]_D^{20}$ –119.1 (*c* 1.25, $CHCl_3$); R_f 0.27 (EtOAc:MeOH, 5:1); IR (thin film) 3363, 2951, 2855, 1472, 1376, 1254, 1127, 1038, 836, 774 cm^{-1}; ^1H NMR δ 0.04 (3H, s), 0.09 (3H, s), 0.88 (9H, s), 1.13 (1H, td, *J* = 14.1, 1.8 Hz), 1.25–1.28 (6H, 2s), 1.55 (1H, dt, *J* = 13.8, 3.9 Hz), 1.96–2.07 (1H, m), 2.63 (1H, t, *J* = 10.2 Hz), 2.86 (3H, br s),

3.20–3.21 (6H, 2s), 3.29 (1H, dd, *J* = 9.9, 2.4 Hz), 3.56–3.70 (3H, m), 3.97–3.98 (1H, m); ^{1}H NMR ($CDCl_3$-D_2O) δ 0.04 (3H, s), 0.09 (3H, s), 0.88 (9H, s), 1.14 (1H, td, *J* = 14.4, 2.1 Hz), 1.25–1.28 (6H, 2s), 1.55 (1H, dt, *J* = 13.8, 3.9 Hz), 1.94–2.07 (1H, m), 2.61 (1H, t, *J* = 10.2 Hz), 3.20–3.21 (6H, 2s), 3.29 (1H, dd, *J* = 9.9, 2.4 Hz), 3.55–3.70 (3H, m), 3.97–3.99 (1H, m); ^{13}C NMR δ –4.7, –4.3, 17.9, 18.2, 18.6, 26.1, 33.9, 38.1, 47.8, 48.2, 58.3, 68.6, 69.1, 71.7, 71.9, 99.6, 99.7; MS (ESI) *m/z* (relative intensity) 406 ($[MH]^+$, 100); HRMS (ESI) calcd for $C_{19}H_{39}O_6NSi$ $[MH]^+$ 406.2619, found 406.2624.

Allyl glucosides 15.

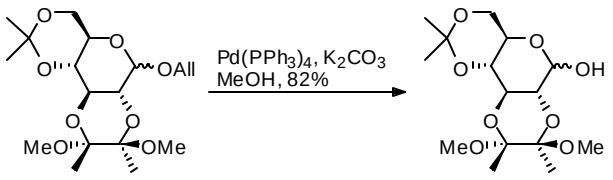


15

To a solution of allyl alcohol (160 mL) containing BF_3·OEt_2 (1.10 mL, 8.68 mmol) was added dry D-glucose (10.1 g, 56.1 mmol), and the mixture was heated under reflux for 12 h. The solvent was evaporated and the residue was dried for 1 h under reduced pressure to afford crude allyl glucopyranside (13.9 g). A suspension of the allyl glucopyranoside in methanol (120 mL) containing 2,3-butanedione (6.9 mL, 78.6 mmol), trimetyl orthoformate (18.4 mL, 168 mmol) was stirred at room temperature for 24 h. The solvent was evaporated and the residue was dried for 1 h under reduced pressure to give a crude oil (24.2 g).

The crude oil was dissolved in dry acetone (120 mL) and then 2,2-dimethoxypropane (DMP) (20.7 mL, 168 mmol) was added. The solution was stirred for 24 h at room temperature. The reaction mixture was then treated with powdered $NaHCO_3$ (7 g) and stirred for 5 h. The resultant mixture was filtered through a pad of silica gel that was eluted with EtOAc. Concentration of the filtrate and the eluant followed by flash chromatography (hexane:EtOAc, 3:1) afforded allyl glucosides **15** (9.06 g, 43%) as an oil: $[\alpha]_D^{20}$ –61.6 (*c* 1.31, $CHCl_3$); R_f = 0.5 (hexane:Et_2O, 1:1); IR (thin film) 2993, 2948, 1730, 1459, 1133, 1039 cm^{-1}; ^{1}H NMR δ 1.30 (6H, s), 1.32 (6H, s), 1.39 (3H, s), 1.40 (3H, s), 1.47 (3H, s), 1.48 (3H, s), 3.23–3.27 (12H, m), 3.60 (1H, t, *J* = 8.4 Hz), 3.67–3.90 (9H, m), 4.02–4.20 (5H, m), 4.32 (1H, ddd, *J* = 13.2, 4.8, 1.5 Hz), 4.56 (1H, d, *J* = 7.8 Hz), 4.84 (1H, d, *J* = 3.6 Hz), 5.14–5.22 (2H, m), 5.28–5.35 (2H, m), 5.83–6.00 (2H, m); ^{13}C NMR ($CDCl_3$) δ 18.1, 18.2, 18.2, 18.4, 19.6, 19.8, 29.8, 29.7, 48.4, 48.5, 48.5, 62.7, 62.9, 64.9, 67.5, 68.9, 68.9, 69.5, 70.5, 70.7, 70.9, 71.0, 72.1, 96.9, 99.8, 99.9, 100.1, 100.1, 100.2, 100.5, 100.9, 117.5, 118.6, 134.4, 134.5; MS (ESI) *m/z* (relative intensity) 397 ($[M]^+$, 100); HRMS (ESI) calcd for $C_{18}H_{30}O_8$ $[M]^+$ 397.1833, found 397.1832.

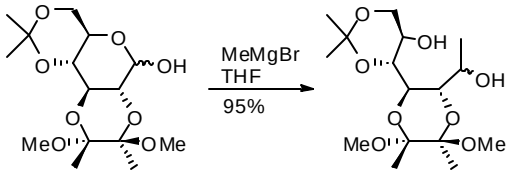
Lactols 16.



15 **16**

To a stirred solution of allyl glucosides **15** (9.06 g, 24.2 mmol) in MeOH (250 mL) was added a catalytic amount of $Pd(PPh_3)_4$ (130 mg, 0.11 mmol) under a nitrogen atmosphere. The pale yellow solution was stirred for 5 min, and K_2CO_3 (15 g, 108.5 mmol) was added. The reaction mixture was heated under reflux for 6 h, cooled, concentrated to half of its volume, and filtrated. The filtrate was evaporated under reduced pressure, and the residue was fractionated by flash chromatography on silica gel (hexane:EtOAc, 1:1) to furnish lactols **16** (6.64g, 82%) as a white solid: mp 196–198 °C; $[\alpha]_D^{20}$ –131 (*c* 1.47, $CHCl_3$); R_f = 0.5 (hexane:EtOAc, 1:1); IR (thin film) 3489, 2994, 2949, 1668, 1462, 1376, 1135 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.28–1.29 (12H, m), 1.36 (3H, s), 1.37 (3H, s), 1.46 (6H, s), 3.22 (3H, s), 3.24 (3H, s), 3.25 (3H, s), 3.26 (3H, s), 3.48 (1H, t, *J* = 9 Hz), 3.66–3.89 (10H, m), 4.04 (1H, t, *J* = 9.6 Hz), 4.81 (1H, d, *J* = 7.8 Hz), 5.16 (1H, d, *J* = 3.6 Hz); ^{13}C NMR ($CDCl_3$) δ 17.9 (CH_3), 17.8 (CH_3), 17.8 (CH_3), 18.1 (CH_3), 19.4 (CH_3), 19.5 (CH_3), 29.3 (CH_3), 29.4 (CH_3), 48.3 (CH_3), 48.4 (CH_3), 48.4 (CH_3), 48.5 (CH_3), 62.5 (CH_2), 62.8 (CH_2), 64.9 (CH), 66.9 (CH), 69.0 (CH), 69.7 (CH), 70.1 (CH), 70.9 (CH), 71.7 (CH), 71.9 (CH), 92.3 (CH), 95.5 (CH), 99.8 (C), 99.9 (C), 100.1 (C), 100.2 (C), 100.5 (C); MS (ESI) *m/z* (relative intensity) 357 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{15}H_{26}O_8$ $[M+Na]^+$ 357.1520, found 357.1518.

Diol 17.

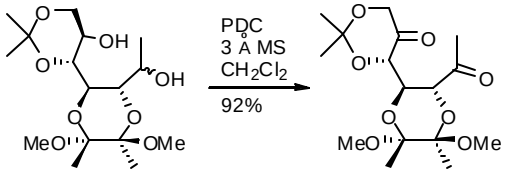


16 **17**

To a stirred solution of lactols **16** (6.64 g, 19.8 mmol) in THF (200 mL) at 0 °C was added methylmagnesium bromide (3 M solution in diethyl ether, 33 mL, 99.0 mmol) slowly. The temperature of the reaction was raised to room temperature and stirring was continued for 24 h. Saturated NH_4Cl (10 mL) was added to the reaction mixture at 0 °C. The reaction mixture was extracted with EtOAc (4 × 100 mL). The combined organic extracts were dried over $MgSO_4$ and filtered. The filtrate was concentrated to give a crude oil that could be used directly for the next step, or purified by flash column chromatography (EtOAc) to yield diol **17** (6.61 g, 95%) as an oil: $[\alpha]_D^{20}$ –16.1 (*c* 1.0, $CHCl_3$); R_f = 0.5 (EtOAc); IR (thin film) 3396, 2992, 2945, 1455, 1374, 1132 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.25–1.28 (11.1H, m), 1.38 (3H, s), 1.44 (3H, s), 1.48 (0.57H, s), 2.42 (0.6H, brs),

2.65 (1H, d, *J* = 7.5 Hz), 3.08 (1H, d, *J* = 3.6 Hz), 3.22 (3H, s), 3.24–3.25, (3.68H, m), 3.59 (1H, dd, *J* = 10.9, 8.4 Hz), 3.76 (1H, dd, *J* = 9.9, 1.8 Hz), 3.82–4.03 (4.9H, m), 4.15 (1H, dd, *J* = 9.9, 3 Hz); ^{13}C NMR ($CDCl_3$) δ 17.7, 17.8, 17.9, 18.0, 18.6, 19.4, 19.6, 19.7, 28.7, 28.7, 48.3, 48.4, 48.5, 48.7, 63.5, 63.9, 64.8, 65.2, 66.4, 67.6, 68.0, 71.0, 72.0, 73.3, 74.2, 77.7, 98.8, 99.2, 99.3, 99.5, 99.7, 100.1 ; MS (ESI) *m/z* (relative intensity) 373 ([M+Na]$^+$, 100); HRMS (ESI) calcd for $C_{16}H_{30}O_8$ [M+Na]$^+$ 373.1833, found 373.1841.

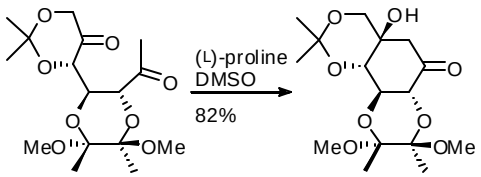
1,5-Diketone 18.



17 **18**

To a mixture of 3Å molecular sieves (ca. 12 g), pyridinium dichromate (PDC) (17 g, 45.1 mmol) in dry CH_2Cl_2 (140 mL) was added a solution of diol **17** (6.61 g, 18.8 mmol) in dry CH_2Cl_2 (50 mL) in one portion under nitrogen. The mixture was stirred for 144 h at room temperature. The mixture was filtered through a pad of Celite and the residue was washed with EtOAc. Concentration of the filtrate followed by flash column chromatography (hexane:Et_2O, 2:1) produced 1,5-diketone **18** (5.97 g, 92%) as white crystals: mp 94–96 °C; $[\alpha]_D^{20}$ –191 (*c* 1.31, $CHCl_3$); R_f = 0.6 (hexane:Et_2O, 1:2); IR (thin film) 2922, 1750, 1721, 1381 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.15 (3H, s), 1.26 (3H, s), 1.34 (3H, s), 1.41 (3H, s), 2.21 (3H, s), 3.19 (3H, s), 3.24 (3H, s), 3.81 (1H, d, *J* = 15.9 Hz), 4.22 (1H, d, *J* = 15.9 Hz), 4.28 (2H, s), 4.69 (1H, s); ^{13}C NMR ($CDCl_3$) δ 17.8 (CH_3), 18.0 (CH_3), 23.9 (CH_3), 25.0 (CH_3), 27.4 (CH_3), 48.7 (CH_3), 48.8 (CH_3), 66.6 (CH), 67.6 (CH_2), 72.8 (CH), 74.9 (CH), 99.3 (C), 99.8 (C), 101.5 (C), 207.7 (C), 207.8 (C); MS (ESI) *m/z* (relative intensity) 369 ([M+Na]$^+$, 100); HRMS (ESI) calcd for $C_{16}H_{26}O_8$ [M+Na]$^+$ 369.1520, found 369.1526.

ß-Hydroxy ketone 19.

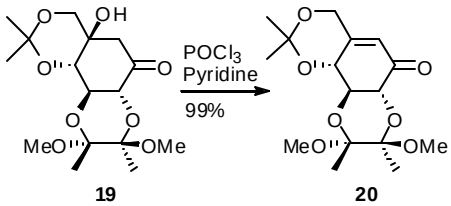


18 **19**

A suspension of 1,5-diketone **18** (3.02 g, 8.72 mmol) in 10 mL DMSO was added (L)-proline (230 mg, 2.00 mmol). After stirring for 6 days at room temperature, the solution was quenched with water (20 mL) and the resultant mixture was extracted with EtOAc (4 × 50 mL). The combined extracts were dried over $MgSO_4$, and filtered. Concentration of the filtrate

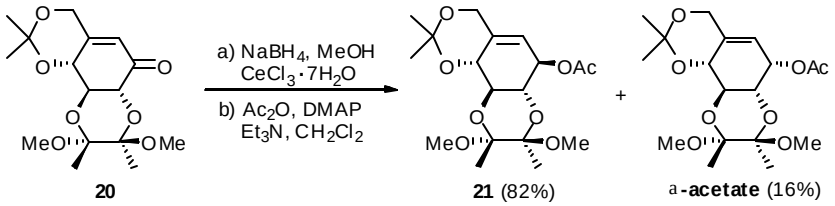
followed by flash chromatography (hexane:EtOAc, 1:1) gave ß-hydroxy ketone **19** (2.47 g, 82%) as white crystals: mp 197–198 °C; $[\alpha]_D^{20}$ –346 (*c* 0.37, $CHCl_3$); R_f = 0.4 (hexane:EtOAc, 1:1); IR (thin film) 3464, 2360, 1735, 1133 cm^{-1}; ^1H NMR δ 1.32 (3H, s), 1.36 (3H, s), 1.37 (3H, s), 1.47 (3H, s), 2.41 (1H, d, *J* =17.7 Hz), 2.52–2.59 (2H, m), 3.25 (3H, s), 3.30 (3H, s), 3.53 (1H, d, *J* =12.3 Hz), 3.68 (1H, d, *J* = 12.3 Hz), 3.91–3.99 (2H, m), 4.51–4.57 (1H, m); ^{13}C NMR δ 17.9 (CH_3), 18.0 (CH_3), 22.9 (CH_3), 25.5 (CH_3), 43.5 (CH_2), 48.4 (CH_3), 48.9 (CH_3), 69.1 (CH_2), 69.7 (CH), 70.7 (CH), 71.7 (C), 76.6 (CH), 99.4 (C), 100.2 (C), 101.5 (C), 202.7 (C); MS (ESI) *m/z* (relative intensity) 369 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{16}H_{26}O_8$ $[M+Na]^+$ 369.1520, found 369.1526; Anal. Calcd for $C_{16}H_{26}O_8$: C, 55.48; H, 7.57, found: C, 55.55; H, 7.69.

Enone 20.



$POCl_3$ (1.65 mL, 18.0 mmol) was slowly added to a solution of ß-hydroxy ketone **19** (1.25 g, 3.60 mmol) in pyridine (10 mL) at 0 °C. The resultant solution was warmed to room temperature and then stirred for 12 h. Toluene was added to precipitate out the pyridinium chloride salt. The mixture was then filtered through a pad of Celite and the residue was washed with EtOAc. Concentration of the filtrate followed by flash chromatography (hexane:EtOAc, 1:1) gave enone **20** (1.17 g, 99%) as yellow crystals: mp 128–129 °C; $[\alpha]_D^{20}$ –197 (*c* 1.28, $CHCl_3$); R_f = 0.46 (hexane:EtOAc, 1:1); IR (thin film) 2360, 1699, 1133 cm^{-1}; ^1H NMR δ 1.33 (3H, s), 1.39 (3H, s), 1.41 (3H, s), 1.51 (3H, s), 3.25 (3H, s), 3.29 (3H, s), 4.06 (1H, dd, *J* = 11.1, 8.7 Hz), 4.27 (1H, d, *J* = 11.1 Hz), 4.36 (1H, dt, *J* = 16.2, 1.5 Hz), 4.50 (1H, dt, *J* = 16.2, 1.5 Hz), 4.71 (1H, dq, *J* = 8.7, 1.5 Hz), 5.80 (1H, q, *J* = 1.5 Hz); ^{13}C NMR δ 17.9 (CH_3), 18.0 (CH_3), 22.4 (CH_3), 26.4 (CH_3), 48.5 (CH_3), 48.9 (CH_3), 62.1 (CH_2), 69.4 (CH), 72.0 (CH), 72.4 (CH), 99.4 (C), 100.5 (C), 100.8 (C), 121.4 (CH), 157.3 (C), 192.7 (C); MS (ESI) *m/z* (relative intensity) 351 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{16}H_{24}O_7$ $[M+Na]^+$ 351.1414, found 351.1416; Anal. Calcd for $C_{16}H_{24}O_7$: C, 58.53; H, 7.37, found: C, 58.21; H, 7.40.

ß-Acetate 21 and a**-acetate**.

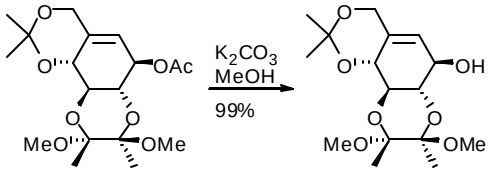


To a solution of enone **20** (1.31 g, 3.99 mmol) in MeOH (70 mL) was added $CeCl_3\,7H_2O$ (1.82 g, 4.80 mmol) at –20 °C. The mixture was stirred for 1 h and then $NaBH_4$ was added (225 mg, 5.94 mmol) in portions at –20 °C. Upon completion (TLC), the reaction was quenched by the addition of saturated NH_4Cl (10 mL), and the aqueous phase was extracted with EtOAc (4 × 70 mL). The combined organic extracts were washed with brine (2 × 20 mL), dried over $MgSO_4$ and filtered. Concentration of the filtrate gave an oil that was then put to the next step without further purification.

To a solution of the crude allylic alcohols (1.28 g) in dichloromethane (15 mL) was added triethylamine (3.3 mL, 23.7 mmol), acetic anhydride (0.7 mL, 7.41 mmol) and a catalytic amount of DMAP (48 mg, 0.39 mmol) at room temperature. The mixture was stirred for 24 h at room temperature. Concentration of the solution followed by flash chromatography (hexane:Et_2O, 1:1) afforded firstly ß-acetate **21** (1.18 g, 82% from **20**) and then α-acetate (0.26 g, 16% from **20**) as colorless oils: Data for ß-acetate **21**: $[\alpha]_D^{20}$ –229 (*c* 0.87, $CHCl_3$); R_f = 0.43 (hexane:EtOAc, 2:1); IR (thin film) 2993, 2951, 1740, 1545, 1454, 1374, 1137 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.29 (3H, s), 1.33 (3H, s), 1.39 (3H, s), 1.50 (3H, s), 2.07 (3H, s), 3.27 (3H, s), 3.28 (3H, s), 3.80–3.89 (2H, m), 4.10 (1H, d, *J* = 14.1 Hz), 4.43 (1H, dd, *J* = 13.9, 1.5 Hz), 4.53 (1H, brs), 5.39 (1H, s), 5.44–5.47 (1H, m); ^{13}C NMR ($CDCl_3$) δ 17.9 (CH_3), 18.0 (CH_3), 20.4 (CH_3), 21.4 (CH_3), 28.5 (CH_3), 48.1 (CH_3), 48.3 (CH_3), 63.0 (CH_2), 69.3 (CH), 69.4 (CH), 70.3 (CH), 72.0 (CH), 99.2 (C), 99.3 (C), 99.6 (C),119.0 (CH), 134.7 (C), 170.8 (C); MS (ESI) *m/z* (relative intensity) 395 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{18}H_{28}O_8$ $[M+Na]^+$ 395.1676, found 395.1680.

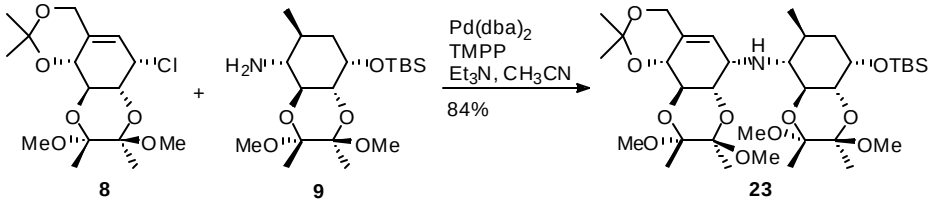
Data for α-acetate: $[\alpha]_D^{20}$ –9.36 (*c* 0.69, $CHCl_3$); R_f = 0.33 (hexane:EtOAc, 2:1); IR (thin film) 2933, 2951, 1741, 1138 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.26 (3H, s), 1.31 (3H, s), 1.41 (3H, s), 1.52 (3H, s), 2.09 (3H, s), 3.24 (3H, s), 3.28 (3H, s), 3.72 (1H, dd, *J* = 11.1, 4.5 Hz), 4.07 (1H, dd, *J* = 11.1, 7.8 Hz), 4.13 (1H, d, *J* = 14.7 Hz), 4.41 (1H, dd, *J* = 3.9, 2.7 Hz)), 4.43 (1H, d, *J* = 14.4 Hz), 5.37 (1H, t, *J* = 4.5 Hz), 5.57 (1H, d, *J* = 5.1 Hz); ^{13}C NMR ($CDCl_3$) δ 17.9 (CH_3), 18.3 (CH_3), 20.4 (CH_3), 21.6 (CH_3), 28.7 (CH_3), 48.4 (CH_3), 48.6 (CH_3), 63.3 (CH_2), 66.6 (CH), 67.1 (CH), 67.9 (CH), 70.4 (CH), 99.2 (C), 99.7 (C), 99.8 (C), 117.4 (CH), 137.9 (C), 171.4 (C); MS (ESI) *m/z* (relative intensity) 395 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{18}H_{28}O_8$ $[M+Na]^+$ 395.1676, found 395.1681.

ß-Alcohol 22.



To a solution of the ß-acetate **21** (980 mg, 2.63 mmol) in MeOH (10 mL) was added a catalytic amount of K_2CO_3 (18 mg, 0.13 mmol) and the mixture was stirred for 12 h at room temperature. The reaction mixture was diluted with EtOAc (100 mL) and washed with saturated NH_4Cl (10 mL). The aqueous layer was extracted with EtOAc (2 × 15 mL). The combined organic extracts were washed with brine (2 × 10 mL), dried over $MgSO_4$ and filtered. Concentration of the filtrate followed by flash chromatography (hexane:Et_2O, 1:1) gave ß-alcohol **22** (860 mg, 98%) as a colorless oil: $[\alpha]_D^{20}$ –201 (*c* 0.83, $CHCl_3$); R_f = 0.33 (hexane:EtOAc, 1:1); IR (thin film) 3469, 2994, 2950, 1738, 1455, 1376, 1140 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.33 (6H, s), 1.39 (3H, s), 1.51 (3H, s), 3.28 (3H, s), 3.29 (3H, s), 3.63 (1H, dd, *J* = 10.85, 7.8 Hz), 3.78 (1H, dd, *J* = 10.8, 7.8 Hz), 4.08 (1H, d, *J* = 13.5 Hz), 4.40-4.49 (2H, m), 4.55 (1H, d, *J* = 7.8 Hz), 5.43 (1H, s); ^{13}C NMR ($CDCl_3$) δ 18.1 (CH_3), 20.1 (CH_3), 29.0 (CH_3), 48.4 (CH_3), 48.5 (CH_3), 63.5 (CH_2), 69.9 (CH), 70.2 (CH), 70.3 (CH), 72.6 (CH), 99.2 (C), 99.3 (C), 99.6 (C), 122.7 (CH), 132.6 (C); MS (ESI) *m/z* (relative intensity) 353 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{16}H_{26}O_7$ $[M+Na]^+$ 353.1571, found 353.1576.

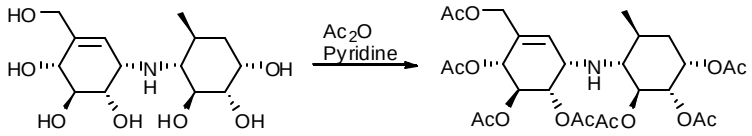
Protected pseudo-acarviosin 23.



To a mixture of chloride **8** (388 mg, 1.11**)** and amine **9** (1.29 g, 3.31 mmol) in CH_3CN (14 mL) was added bis(dibenzlideneacetone) palladium (0) (32 mg, 0.056 mmol), TMPP (18 mg, 0.11 mol) and Et_3N (0.7 mL, 5.02 mmol). The resultant solution was stirred for 48 h at room temperature under nitrogen. Concentration of the solution followed by flash chromatography (hexane:Et_2O, 5:1) gave firstly protected pseudo-acarviosin **23** (661 mg, 84%) as a colorless oil and then (hexane:EtOAc, 1:1) secondly recovered amine **9** (808 mg). Data for **23**: $[\alpha]_D^{20}$ –65.8 (*c* 0.93, $CHCl_3$); R_f = 0.55 (hexane:Et_2O, 1:1); IR (thin film) 2991, 2950, 1462, 1373, 1120 cm^{-1}; ^1H NMR δ .0.05 (3H, s), 0.08 (3H, s), 0.89 (9H, s), 0.94 (1H, d, *J* = 6.3 Hz), 1.07–1.17 (1H, m), 1.21 (3H, s), 1.22 (3H, s), 1.23 (3H, s), 1.31 (3H, s), 1.37 (3H, s), 1.49 (3H, s), 1.66 (1H, t, *J* = 3.6

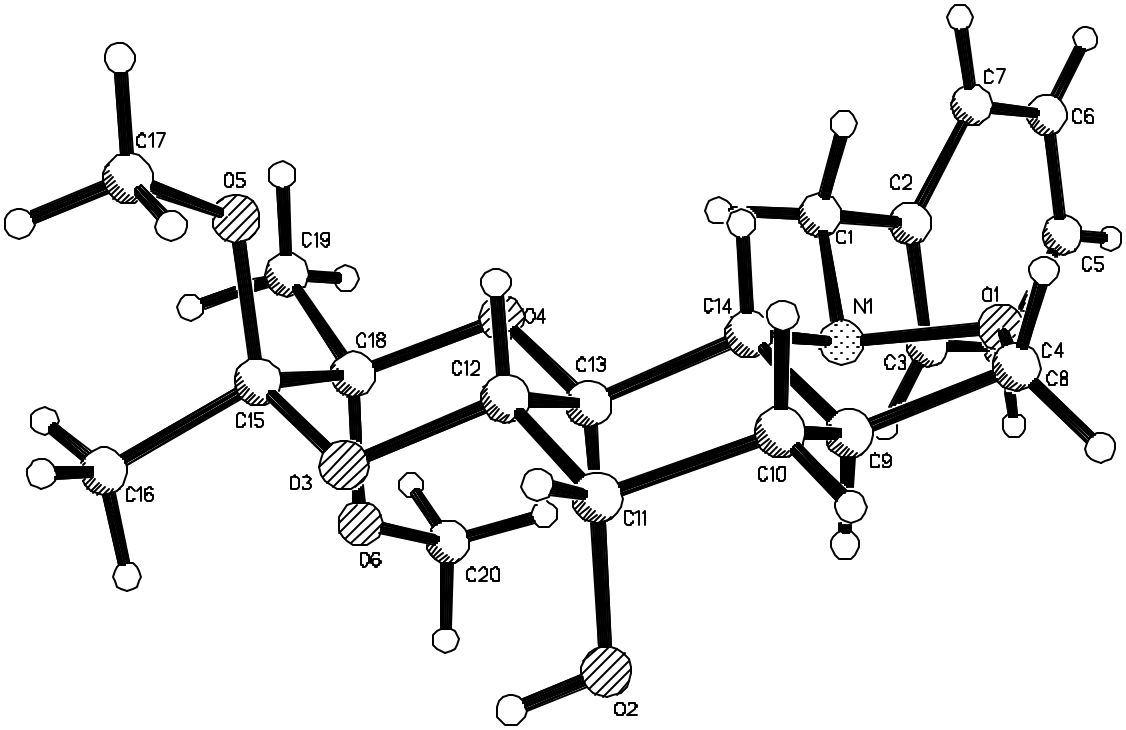
Hz), 1.71 (1H, t, *J* = 3.6 Hz), 2.09 (1H, t, *J* = 9.9 Hz), 3.21 (3H, s), 3.23 (3H, s), 3.29 (3H, s), 3.30 (3H, s) , 3.34 (1H, dd, *J* = 11.1, 2.4 Hz), 3.65 (1H, dd, *J* = 10.8, 5.1 Hz), 3.79 (1H, t, *J* = 9.9 Hz), 3.91–3.94 (2H, m), 4.04 (1H, d, *J* = 13.5 Hz), 4.19 (1H, dd, *J* = 7.8, 10.8 Hz), 4.38–4.43 (2H, m), 5.59 (1H, d, *J* = 4.5 Hz); ^{13}C NMR δ –4.7 (CH_3), –4.2 (CH_3), 17.9 (CH_3), 18.0 (CH_3), 18.4 (CH_3), 18.5, (CH_3), 18.8 (C), 19.6, (CH_3), 20.6 (CH_3), 26.3 (CH_3), 28.9 (CH_3), 32.3 (CH), 39.9 (CH_2), 47.9 (CH_3), 48.0 (CH_3), 48.4 (CH_3), 53.8 (CH), 60.7 (CH), 64.1 (CH_2), 67.9 (CH), 68.4 (CH), 69.2 (CH), 71.2 (CH), 72.9 (CH), 74.0 (CH), 99.0 (C), 99.2 (C), 99.4 (C), 99.6 (C), 122.7 (CH), 132.0 (C); MS (ESI) *m/z* (relative intensity) 702 ($[M+H]^+$, 100); HRMS (ESI) calcd for $C_{35}H_{63}N_1O_{11}Si_1$ $[M+H]^+$ 702.4243, found 702.4252.

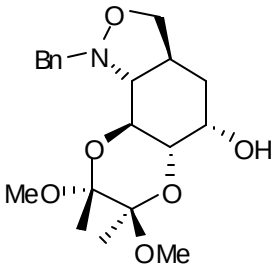
Heptaacetate 24.



Pseudo-acarviosin **5** **24**

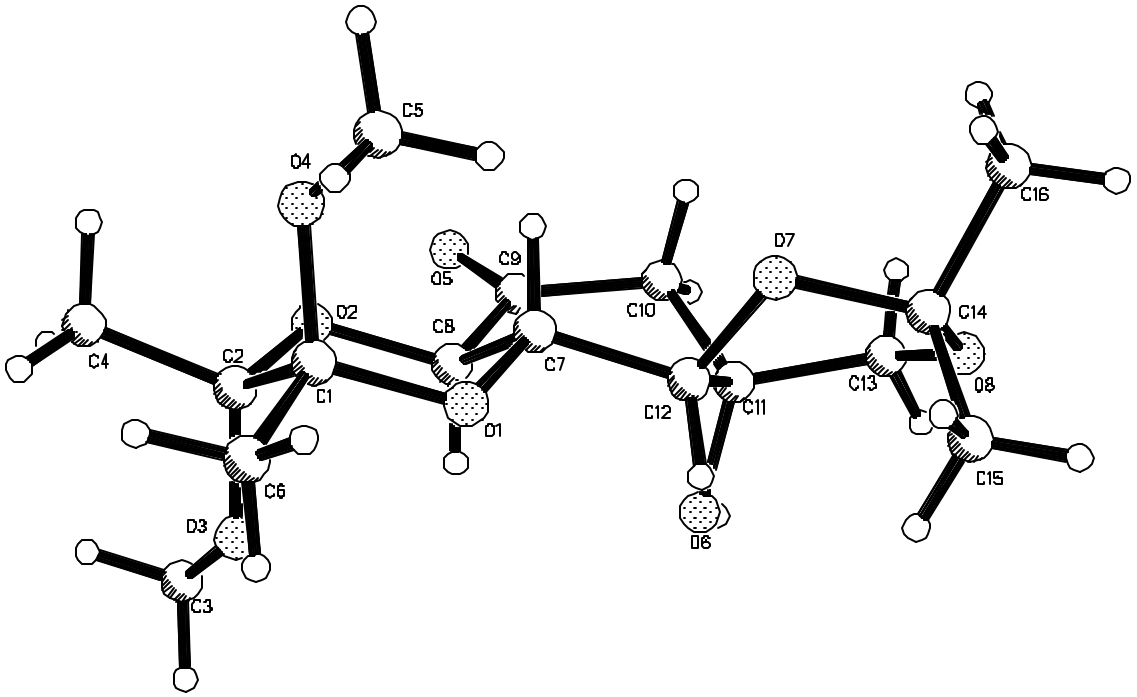
To a solution of amine **5** (20.2 mg, 0.063 mmol) in pyridine (2 mL) was added acetic anhydride (0.07 mL, 0.74 mmol) and a catalytic amount of DMAP (0.6 mg, 4.9 μmol) at room temperature. The mixture was stirred for 24 h at room temperature. Concentration of the solution followed by flash chromatography (hexane-Et_2O, 1:1) gave heptaacetate **24** (19.3 mg, 76%) as a colorless oil: $[\alpha]_D^{20}$ +65.3 (*c* 1.03, $CHCl_3$); IR (thin film) 2954, 1743, 1370, 1231, 1026 cm^{-1}; ^{1}H NMR δ 0.98 (3H, d, *J* = 6.3 Hz), 1.22–1.35 (1H, m), 1.66–1.76 (1H, m), 1.89 (1H, dt, *J* = 14.7, 3.9 Hz), 1.97 (3H, s), 2.00 (3H, s), 2.02 (3H, s), 2.03 (3H, s), 2.05 (3H, s), 2.10 (3H, s), 2.11 (3H, s), 2.26 (1H, t, *J* = 10.2 Hz), 3.76 (1H, t, *J* = 4.8 Hz), 4.36 and 4.66 (2H, ABq, *J* = 12.9 Hz), 4.83 (1H, dd, *J* = 10.2, 3.0 Hz), 4.94 (1H, dd, *J* = 10.5, 4.2 Hz), 5.19 (1H, t, *J* = 10.2 Hz), 5.30 (1H, d, *J* = 2.4 Hz), 5.60 (1H, d, *J* = 6.6 Hz), 5.68 (1H, dd, *J* = 10.2, 6.9 Hz), 6.00 (1H, d, *J* = 4.8 Hz); ^{13}C NMR δ 19.1 (CH_3), 21.1 (CH_3), 21.2 (CH_3), 21.2 (CH_3), 21.3 (CH_3), 21.3 (CH_3), 21.5 (CH_3), 33.1 (CH), 35.3 (CH_2), 52.7 (CH), 62.3 (CH), 63.6 (CH_2), 69.4 (CH), 70.3 (CH), 71.4 (CH), 71.6 (CH), 73.3 (CH), 75.4 (CH), 128.9 (CH), 134.2 (C), 170.6 (C), 170.7 (C), 170.7 (C), 170.7 (C), 170.8 (C), 170.9 (C), 171.6 (C); MS (FAB) *m/z* (relative intensity) 614 ($[M + H]^+$, 60), 93 (100).

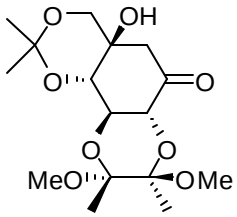




12

Fig. S1. X-ray crystallographic structure of isoxazoline **12**.





19

Fig. S2. X-ray crystallographic structure of **19**.

Experimental Procedures (*in vitro* enzyme inhibition assays and *in vivo* anti-diabetic studies)

Experimental animals

The rats were provided by and kept at the Laboratory Animal Services Center of The Chinese University of Hong Kong. All animal experiments conformed to the Guideline for Care and Use of Laboratory Animals and were approved by the Animal Research Ethics Committee of The Chinese University of Hong Kong. Three or four rats were housed in a wire-bottomed cage and acclimatized under the conditions of room temperature (25±3°C), humidity (55±5%), and light (12 h light-dark cycle). The animals were allowed free access to standard laboratory diet (Prolab 2500 rodent diet) and tap water.

Chemicals and reagents

α-Amylase (EC 3.2.1.23 from human saliva), streptozotocin (STZ), glucose, fructose, maltose, sucrose and starch were obtained from Sigma (St. Louis, MO, U.S.A.). All other reagents were of the highest analytical quality obtained from commercial sources.

Buffers and solutions

Starch, maltose, isomaltose, trehalose, lactose and sucrose were dissolved in 100 mM potassium phosphate buffer pH 6.3 as specific substrates for the digestive enzymes. One percent 3,5-dinitrosalicylic acid (DNS) and 12% sodium potassium tartrate were dissolved in 0.4 M NaOH as a stock solution. Bovine serum albumin (BSA) standard was prepared at a concentration of 10 mg/ml and stored at -30°C. STZ was dissolved in citrate buffer (28 mM of citric acid monohydrate and 255 mM of citric acid trisodium salt dihydrate, pH 4.5) at a concentration of 20 mg/ml. The solution was freshly prepared before use. Glucose was dissolved in distilled water at 0.4 g/ml for the oral glucose tolerance test. A brief sonication was performed to ensure complete dissolution. Fructose, maltose, and sucrose were also dissolved in distilled water at 0.4 g/ml for the carbohydrate loading test. A brief sonication was performed to ensure complete dissolution. A 1% (w/v) starch solution was obtained by dissolving soluble starch in distilled water by boiling. The glucose oxidase/peroxidase assay kit was purchased from Biosystems (CT, U.S.A.). The assay kit includes an assay reagent containing 70 mM of phosphate buffer, 5 mM of phenol, > 10 U/ml of glucose oxidase, > 1 U/ml of peroxidase, 0.4 mM of 4-aminoantipyrine, pH 7.5, and a glucose/urea/creatinine standard containing 100 mg/dl (5.55 mM) of glucose, 50 mg/dl of urea, and 2 mg/dl of creatinine. Acarbose obtained from Sequoia Research Products Ltd. (Pangbourne, UK) was dissolved at a concentration of 0.8 mg/ml for

the oral sugar tolerance test. The pseudo-acarviosin was dissolved to 100 mM in distilled water before used. Small aliquots were stored frozen at -30°C under nitrogen.

***In vitro* assessment of the enzyme inhibition activities**

α-Amylase assay

α-Amylase activity was determined by a modified method described previously (*S1*). α-Amylase (0.5 ml) with 0.5 ml 0.5% starch solution as substrate in 100 mM sodium phosphate buffer pH 6.9 was assayed at 37°C for 5 min and terminated by addition of 2 ml of the DNS reagent (1% DNS, 12% sodium potassium tartrate in 0.4 M NaOH). The reaction mixture was heated for 15 min at 100°C and immediately cooled on an ice bath to room temperature. Then the reaction mixture was diluted to 10 ml with distilled water. The absorbance at 540 nm was recorded. One unit of enzyme activity liberated 1.0 mg maltose from starch in 5 min at pH 6.9 at 37°C. To evaluate the inhibitory actions of the test compound, the compound was preincubated with α-amylase at 37°C for 30 min before 0.5 ml 0.5% starch solution was added.

Preparation of intestinal enzymes

Digestive enzyme preparations from the small intestine of rats were used as the source of various digestive enzymes (maltase, sucrase, trehalase, isomaltase, glycoamylase, and lactase) (*S2*). Male Sprague-Dawley rats (200-250 g) were fasted overnight before sacrificed for experiments. The mucosal layer of the small intestine was removed and homogenized in 5 times its volume of buffer: 0.5 M NaCl, 0.5 M KCl, 5 mM EDTA, pH 7.0. The homogenate was centrifuged at 20,000g for 30 min and the pellet was washed by suspension and recentrifuged three times in fresh cold saline. The final pellet was homogenized in 5 times its volume of 0.9 % NaCl and centrifuged at 200g for 10 min. The cloudy supernatant, which contained 70-90 % enzyme activity, was further diluted to 8~10 mg/ml protein with 0.9 % NaCl. The preparation was snap frozen and stored at -80°C in aliquots until use.

Protein assay

The protein concentration was measured by the Lowry method (*S3*). Briefly, prior to the assay, the assay reagent was freshly prepared by mixing 2% Na_2CO_3 and 0.5% $CuSO_4$ in 1% sodium tartrate solution in a ratio of 50:1. BSA standards and the samples were diluted 1 time by 1 M NaOH solution. The diluted standard or sample (0.2 ml) in a test tube was mixed with 1 ml assay reagent for 10 min at room temperature. Then, 0.1 ml Folin reagent was added. After 30 min, the absorbance was measured at 750 nm. The protein concentration of the sample was calculated from the standard curve generated from the BSA standards.

Assays of intestinal enzymes

Various sugars were used as the specific substrates for the enzymes at 40 mM final concentration in 100 mM potassium phosphate buffer pH 6.3. The enzyme activity in each case was assayed at 37°C for 30 min. The reactions were stopped by heating at 80°C for 3 min. Another set of mixture was heat inactivated at 80°C for 3 min as blank. The reaction mixture was centrifuged and the supernatant was used to measure the glucose concentration.

Enzyme inhibition reversibility studies

The enzymes were mixed with the test compound for 30 min at 37°C. Then half aliquot of the enzyme-inhibitor mixture or the enzyme alone control was dialyzed against the assay buffer for 24 h. The other half aliquot in parallel was kept at 4°C for 24 h without dialysis. The enzyme activity was then determined as described above.

Kinetics of enzyme inhibition

The kinetic parameters of the enzyme inhibition were determined by the Lineweaver-Burk transformation method at different concentrations of substrate and inhibitor (*S4*). In mixed inhibition, the inhibitor can bind to the free enzyme as well as to the enzyme-substrate complex (*S5*). As a result, two inhibitor constants, K_i and K_I, can be defined, where K_i is the dissociation constant of the enzyme-inhibitor complex, and K_I is the dissociation constant of the enzyme-substrate-inhibitor complex. Since the Lineweaver-Burk plot crosses to the left of the 1/V axis but above the 1/[S] axis, it belongs to the so-called competitive-noncompetitive type of inhibition where $K_I > K_i$. K_i can be calculated from the slope of the inhibited curve where:

$$\text{Slope} = \frac{K_m}{V_{max}} \left(1 + \frac{[I]}{K_i}\right)$$

K_I can be calculated from the y-intercept of the inhibited curve where:

$$\text{y-intercept} = \frac{1}{V_{max}} \left(1 + \frac{[I]}{K_I}\right)$$

***In vivo* assessment of the anti-diabetic activity**

Induction of experimental diabetes in rats

For diabetic studies, a neonatal STZ-induced (n-STZ) rat model of type 2 diabetes was employed as described previously (*S6*). In general, the n-STZ model is induced by injecting Wistar rats on the day of their birth (n0-STZ). The n0-STZ rats

exhibit insulin deficient acute diabetes mellitus 3-5 days after birth, but subsequently the plasma glucose and insulin values are no longer significantly different from those of the controls. It is only by 8 weeks of age and thereafter the n0-STZ rats show mild hyperglycemia, mild hypoinsulinemia and glucose intolerance. In this study, the n0-STZ rats were used. Briefly, neonatal Wistar rats were injected with STZ intraperitoneally (100 mg/kg body weight) on the first day of their birth. STZ was dissolved in 50 mM citrate buffer (pH 4.5) at 20 mg/ml freshly prepared before use. Control animals were manipulated in parallel without STZ administration. All animals were weaned 21 days after birth. They were housed in stainless steel wired cages and were given standard laboratory diet and tap water *ad libitum*. The animals were used for the subsequent experiments at 12 weeks of age. The diabetic condition was confirmed by a 2 h fasting plasma glucose level at 140 mg/dl or over in the adult rats.

Carbohydrate loading test

The n0-STZ rats at the age of week 10 to 12 were used in the carbohydrate loading test (*S7*). The test compound was administrated orally with each carbohydrate (glucose, fructose, sucrose, maltose and starch) and the plasma glucose level of rats was determined.

The diabetic rats were firstly randomized into various groups (n=6): negative control (glucose, fructose, sucrose, maltose or starch solution, 5 ml/kg), positive control (acarbose in carbohydrate solution, 5 ml/kg, 4 mg/kg body weight), and treatment groups (pseudo-acarviosin in carbohydrate solution, 5 ml/kg, at various doses). The procedure of the carbohydrate loading test was as follows. Prior to the experiment, the diabetic rats were fasted for 14-16 h. Then, a 200 µl blood sample was collected from the tail vein of each rat in a 1.5 ml microfuge tube containing heparin (1250 U/ml, 5 µl). This time point was designated as time zero. After the blood sample was collected, the carbohydrate solution with or without the test compound was immediately introduced orally to the rats by oral intubation. Blood was collected every 20 min over a period of 2 h. The plasma samples were collected by centrifugation at 4000g for 5 min and transferred to another tube and stored on ice. The plasma glucose level was measured by the enzymatic glucose oxidase/peroxidase method. Rats were kept unfed throughout the whole experimental period.

Plasma glucose level determination

The plasma glucose level was measured by an enzymatic glucose oxidase/peroxidase assay kit (Biosystems) performed on 48-well plates. To 5 µl of plasma sample in a well, the assay reagent (1ml) pre-warmed at 37^{o}C was added and the mixture was incubated at 37^{o}C for 5 min. In each plate, two blanks and two standards were included by adding 5 µl of distilled water and 5 µl of glucose standard solution respectively. The absorbance values of the plasma samples and the standards were measured at 500 nm against the blank on a microplate reader.

References for supporting information.

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S2. B. L. Rhinehart, K. M. Robinson, P. S. Liu, A. J. Payne, M. E. Wheatley, S. R. Wagner, *J. Pharmacol. Exp. Ther.* **241**, 915 (1987).

S3. O. H. Lowry, N. J. Rosebrough, A. L. Farr, R. J. Randall, *J. Biol. Chem.* **193**, 265 (1951).

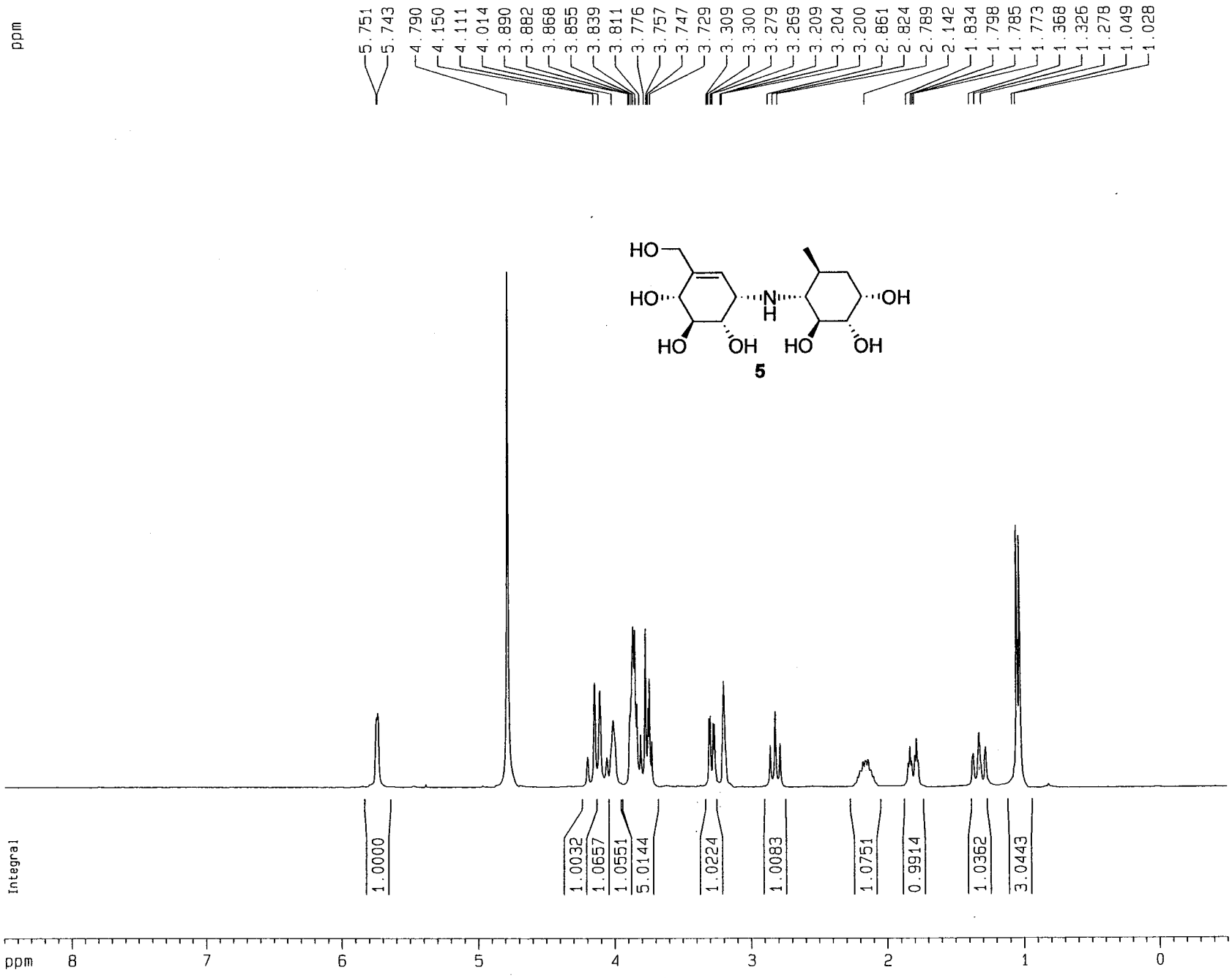
S4. L. D. Kong, Y. Zhang, X. Pan, R. X. Tan and C. H. K. Cheng, *Cell. Mol. Life Sci.* **57**, 500 (2000).

S5. T. Palmer, *Understanding Enzymes* 3rd edn. (Ellis Horwood, London, 1991), pp. 139-165.

S6. O. Blondel, D. Bailbe, B. Portha, *Diabetes* **38**, 610 (1989).

S7. J. M. Li, C. T. Che, C. B. S. Lau, P. S. Leung, C. H. K. Cheng, *J. Pharmacol. Exp. Ther.* **320**, 38 (2006).

Solvent: CD_3OD



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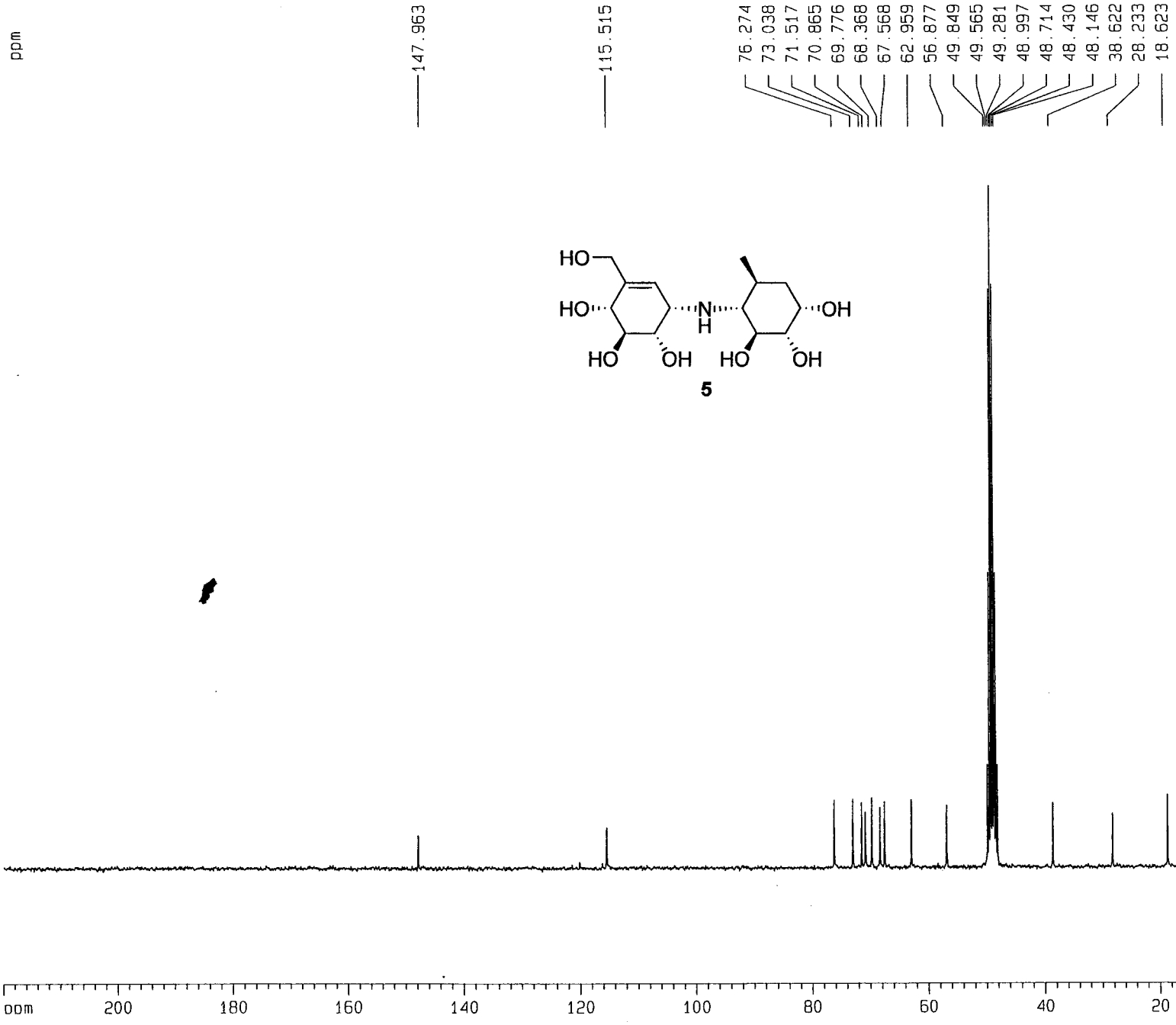

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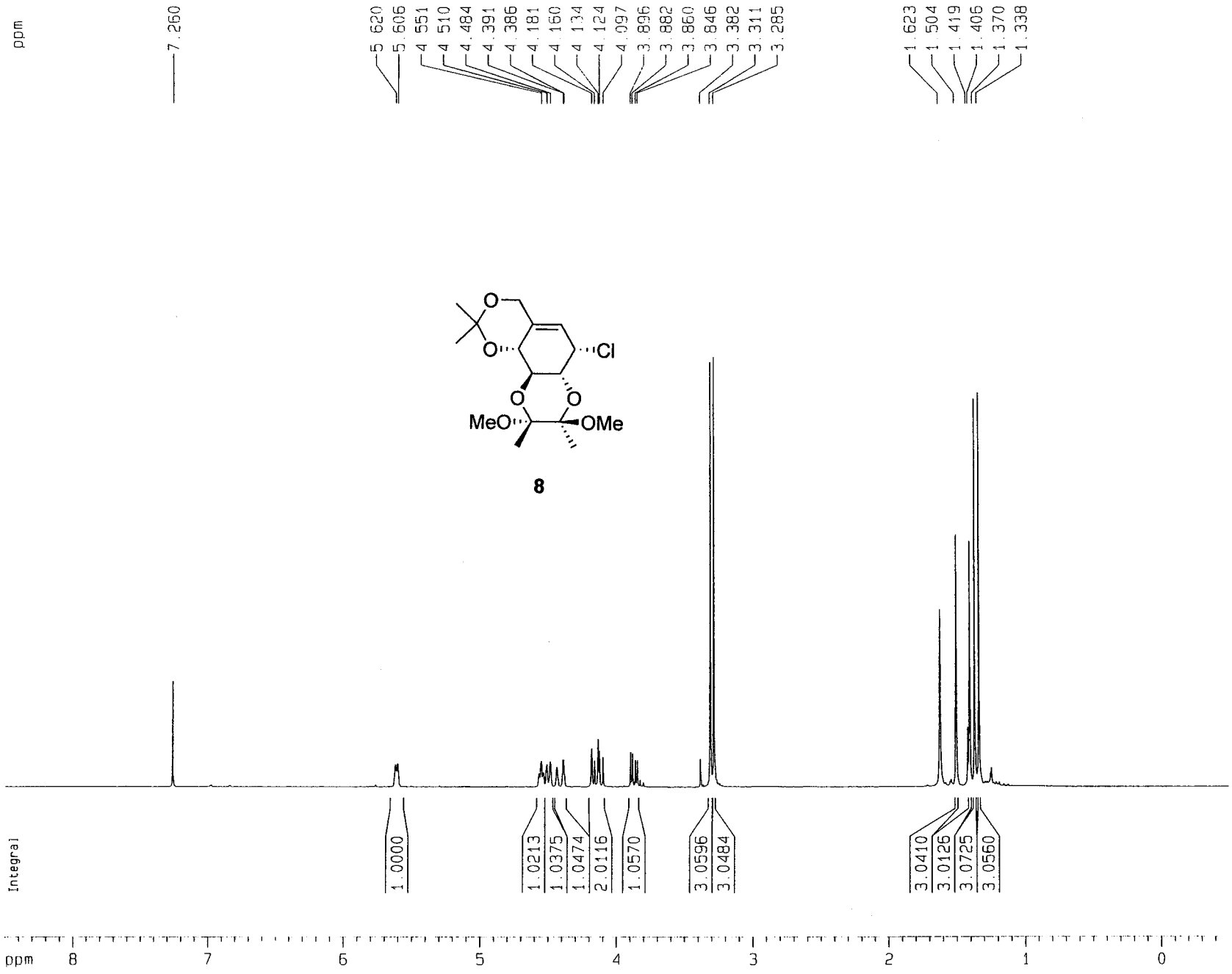

8

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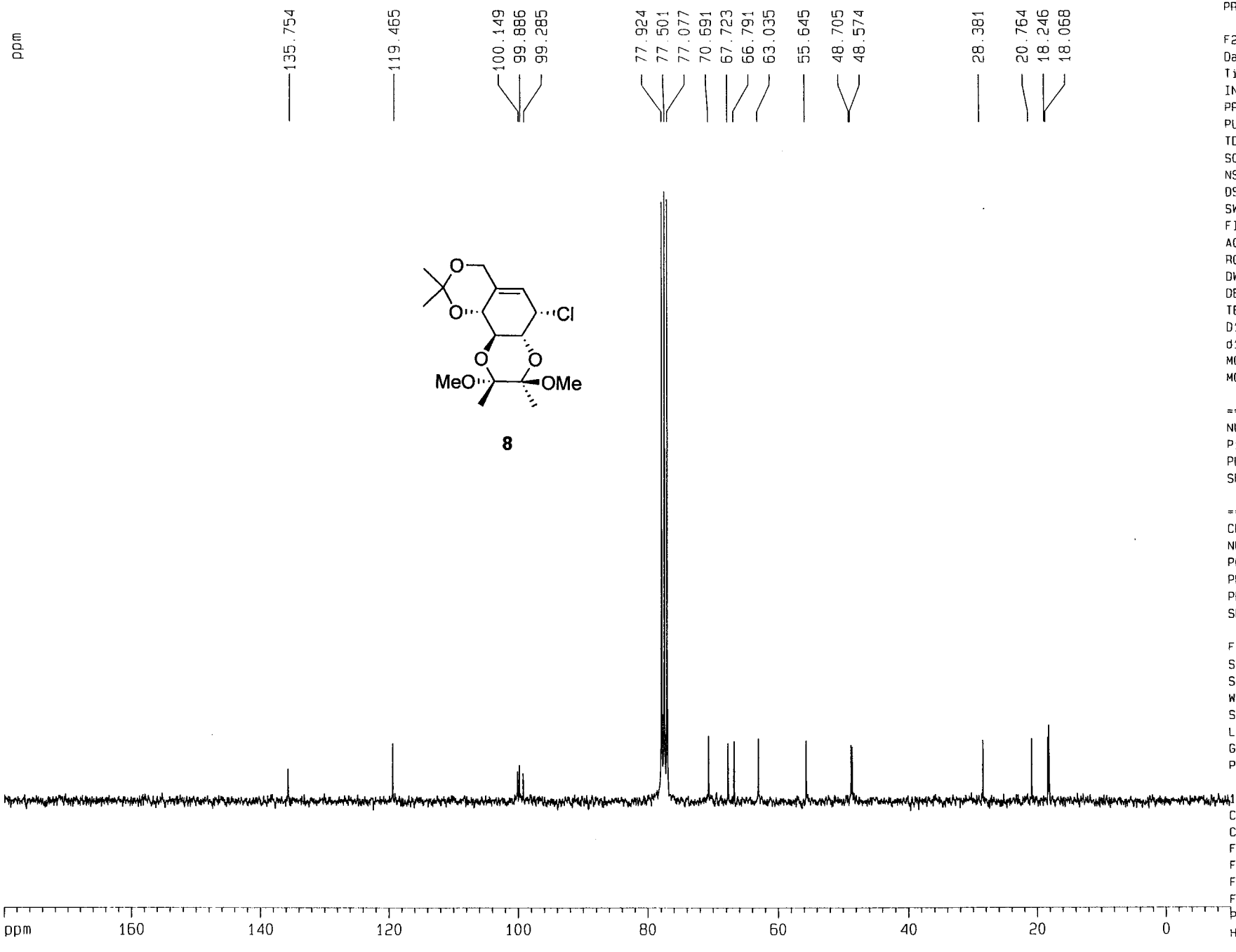

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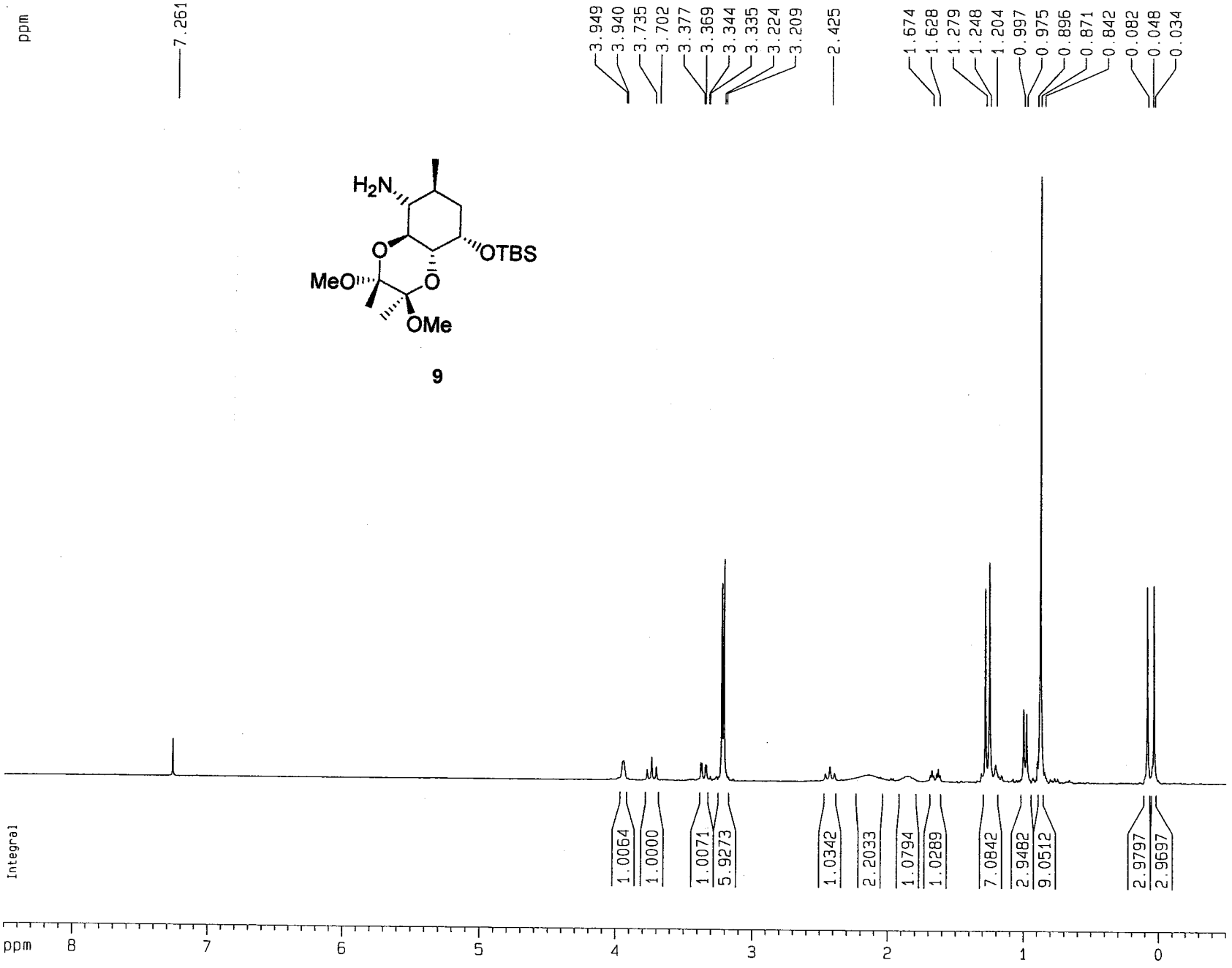

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1D NMR plot parameters

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F2	-150.07 Hz
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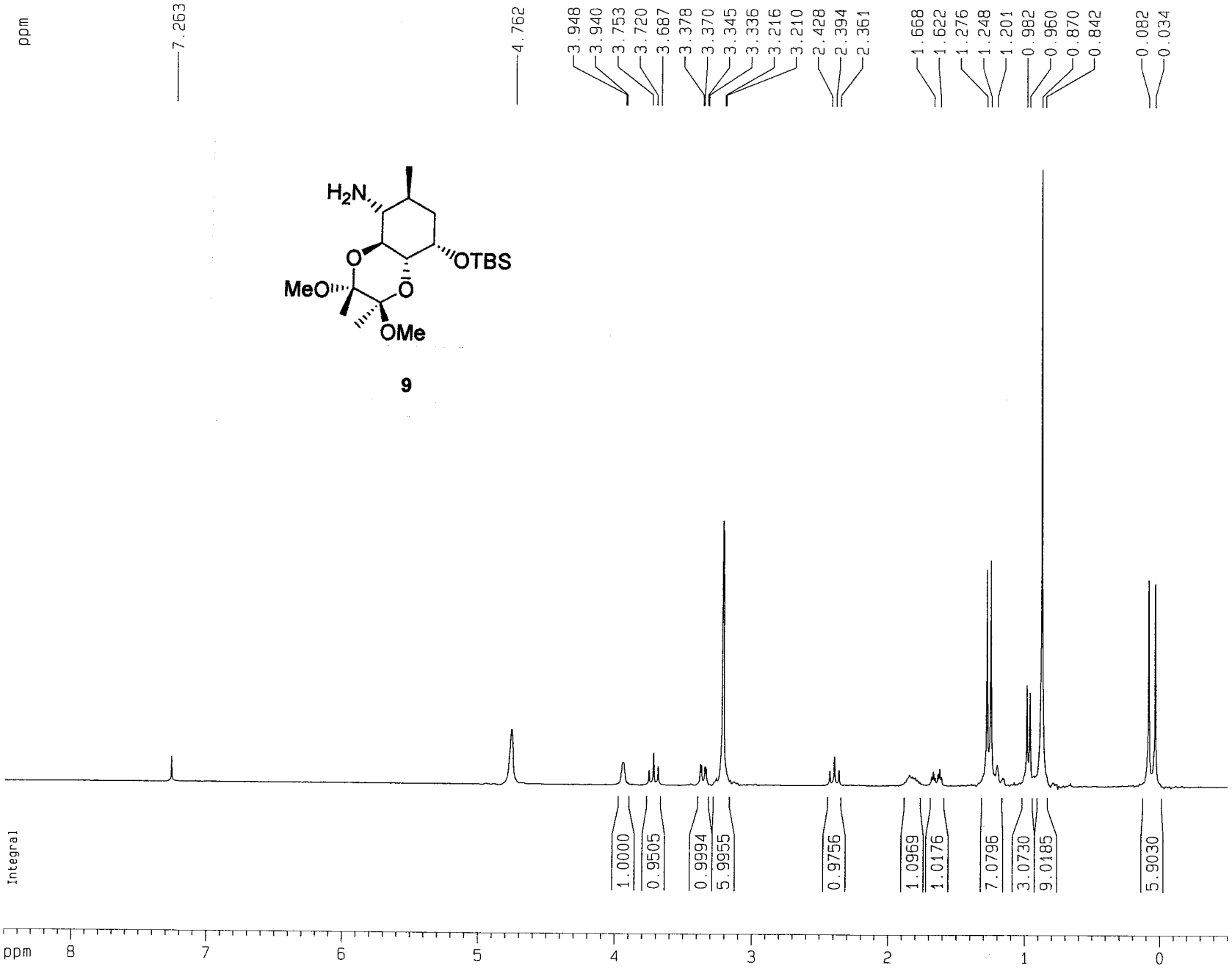
HZCM	122.78046 Hz/cm

Solvent: $CDCl_3$ with D_2O



Current Data Parameters

NAME	wwf65b_d20
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050618
Time	14.55
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	114
DW	55.600 usec
DE	79.43 usec
TE	295.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

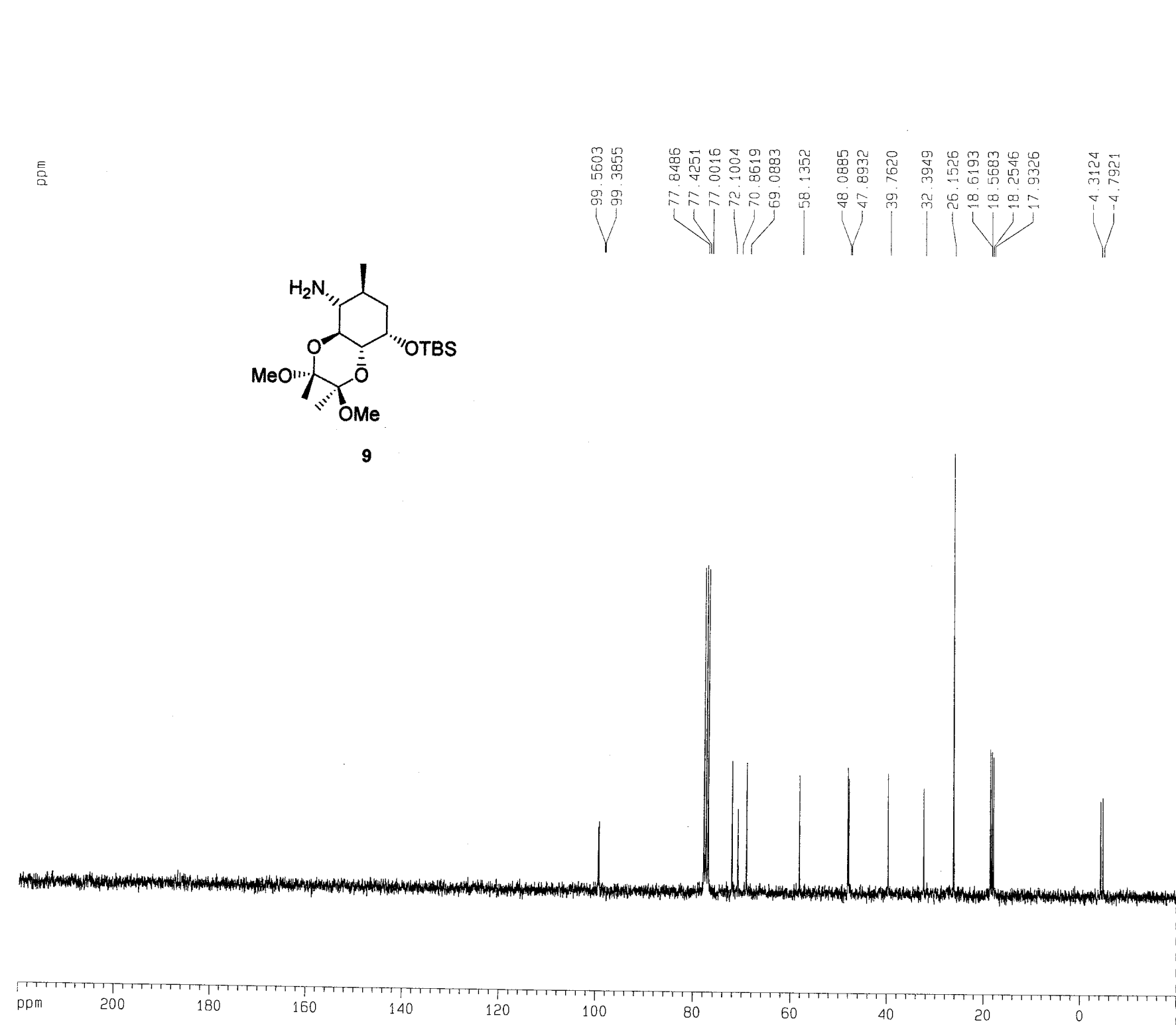
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.10 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf65carbon
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20050618
Time	14.41
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	347
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	912.3
DW	18.900 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

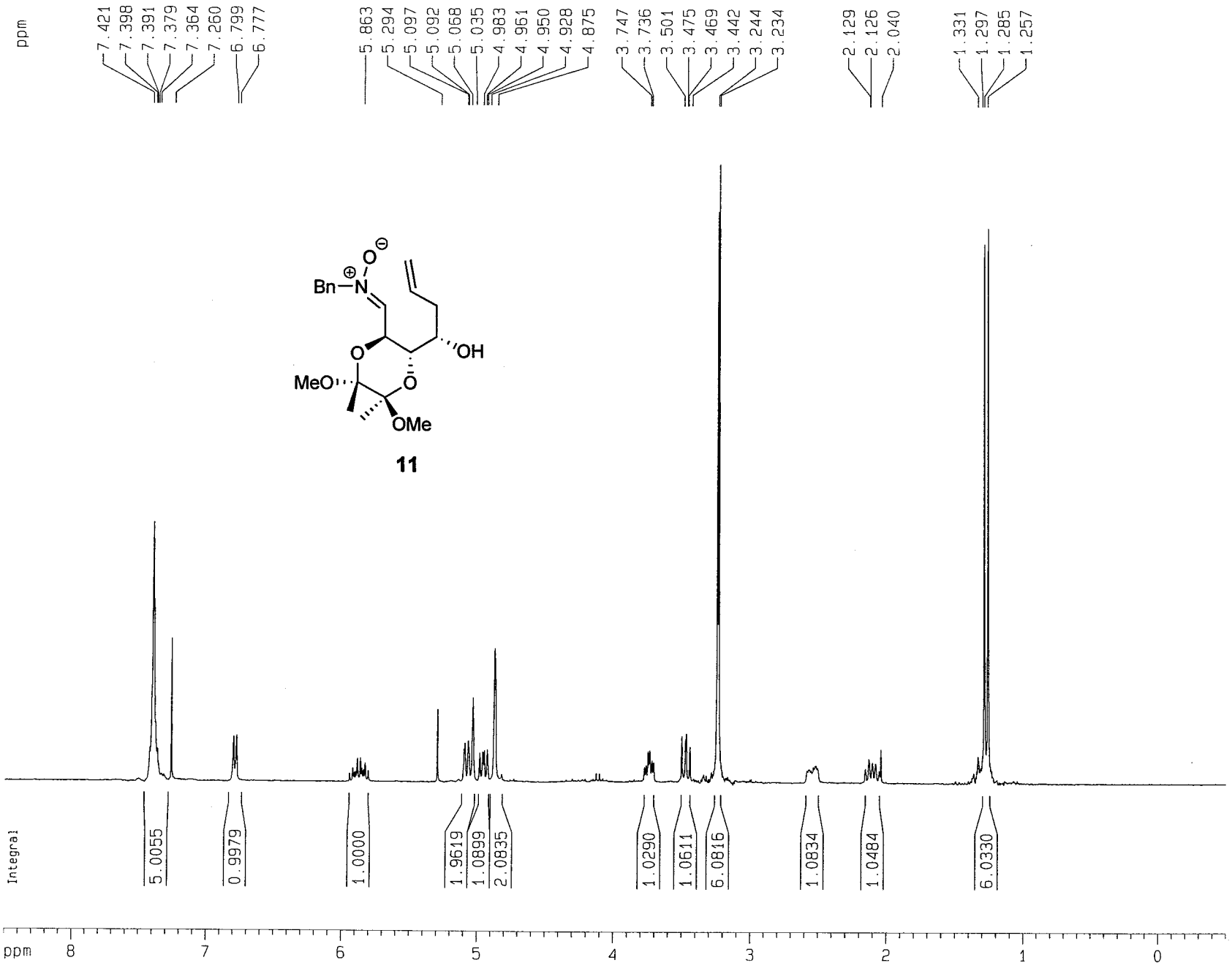
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677196 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	8.81 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-20.000 ppm
F2	-1509.36 Hz
PPMCM	10.43478 ppm/cm
HZCM	787.48926 Hz/cm



Current Data Parameters

NAME	wwf53
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050304
Time	20.44
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	228.1
DW	55.600 usec
DE	79.43 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

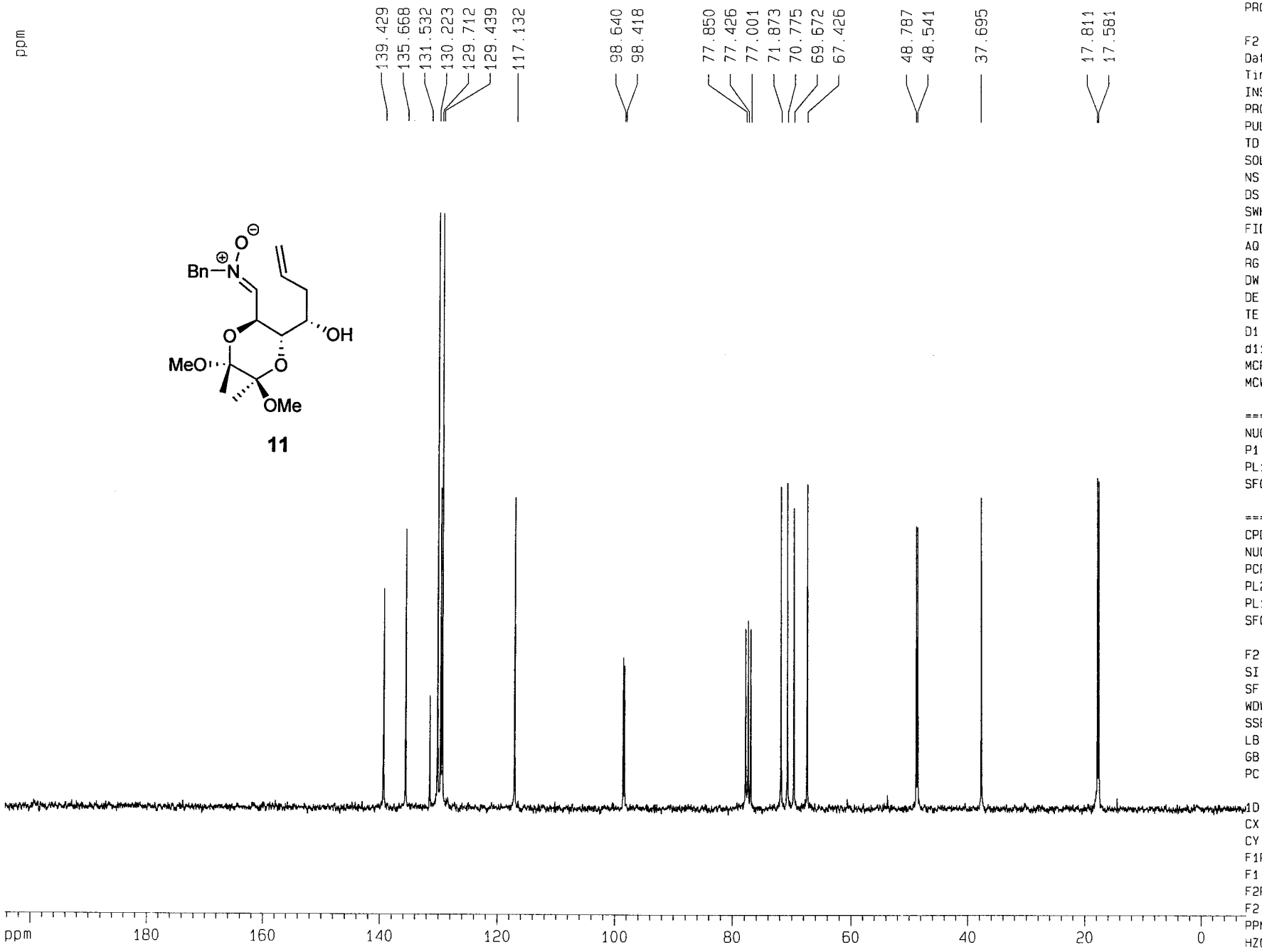
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.27 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf53-carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050304
Time	20.56
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	188
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	8192
DW	18.900 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

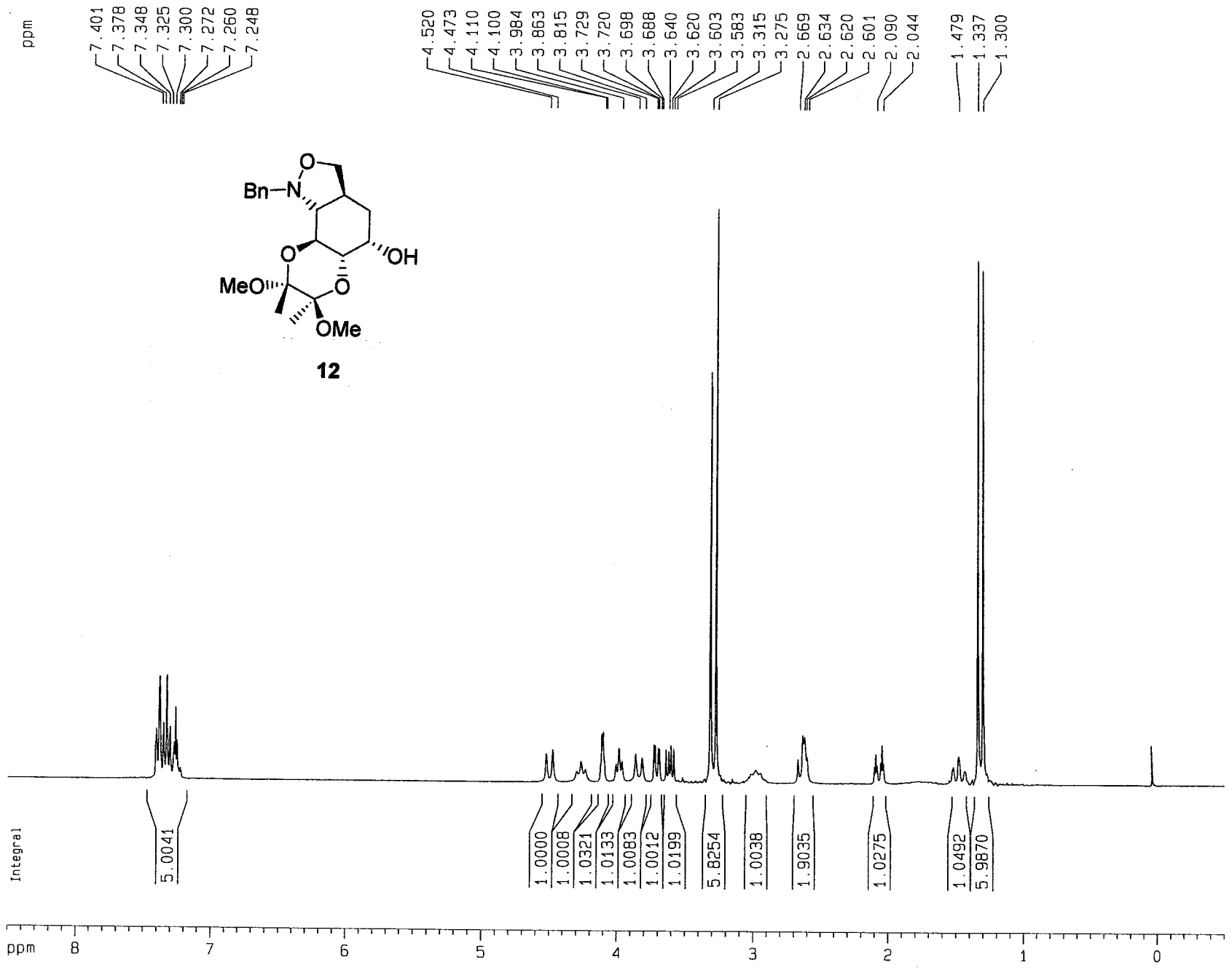
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677276 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	11.12 cm
F1P	204.375 ppm
F1	15423.75 Hz
F2P	-7.698 ppm
F2	-580.97 Hz
PPMCM	9.22060 ppm/cm
HZCM	695.85754 Hz/cm



Current Data Parameters

NAME	wwf55
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050317
Time	13.25
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	161.3
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

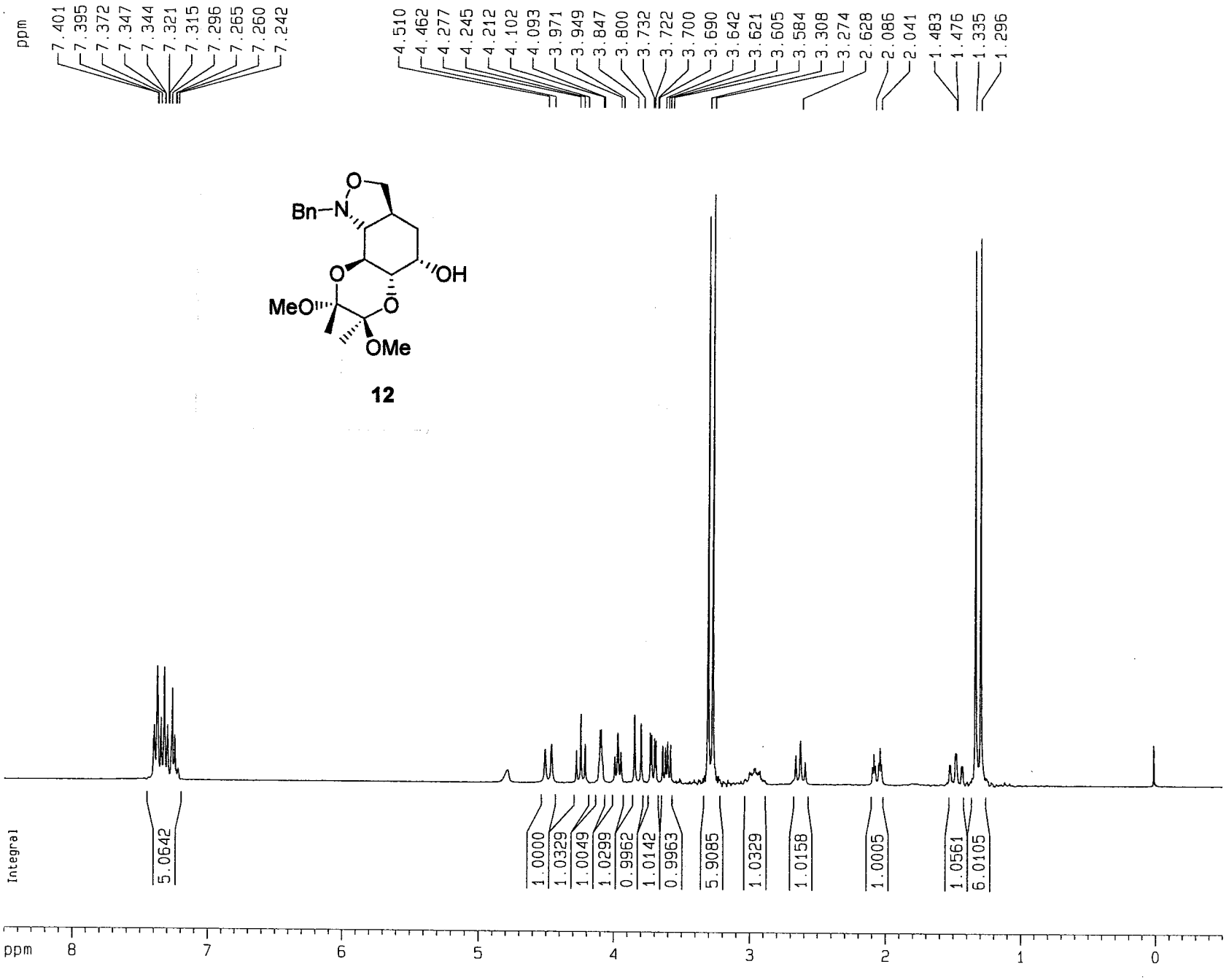
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.44 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm

Solvent: $CDCl_3$ with D_2O



Current Data Parameters

NAME	wwf55-d20
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050317
Time	13.28
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	161.3
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

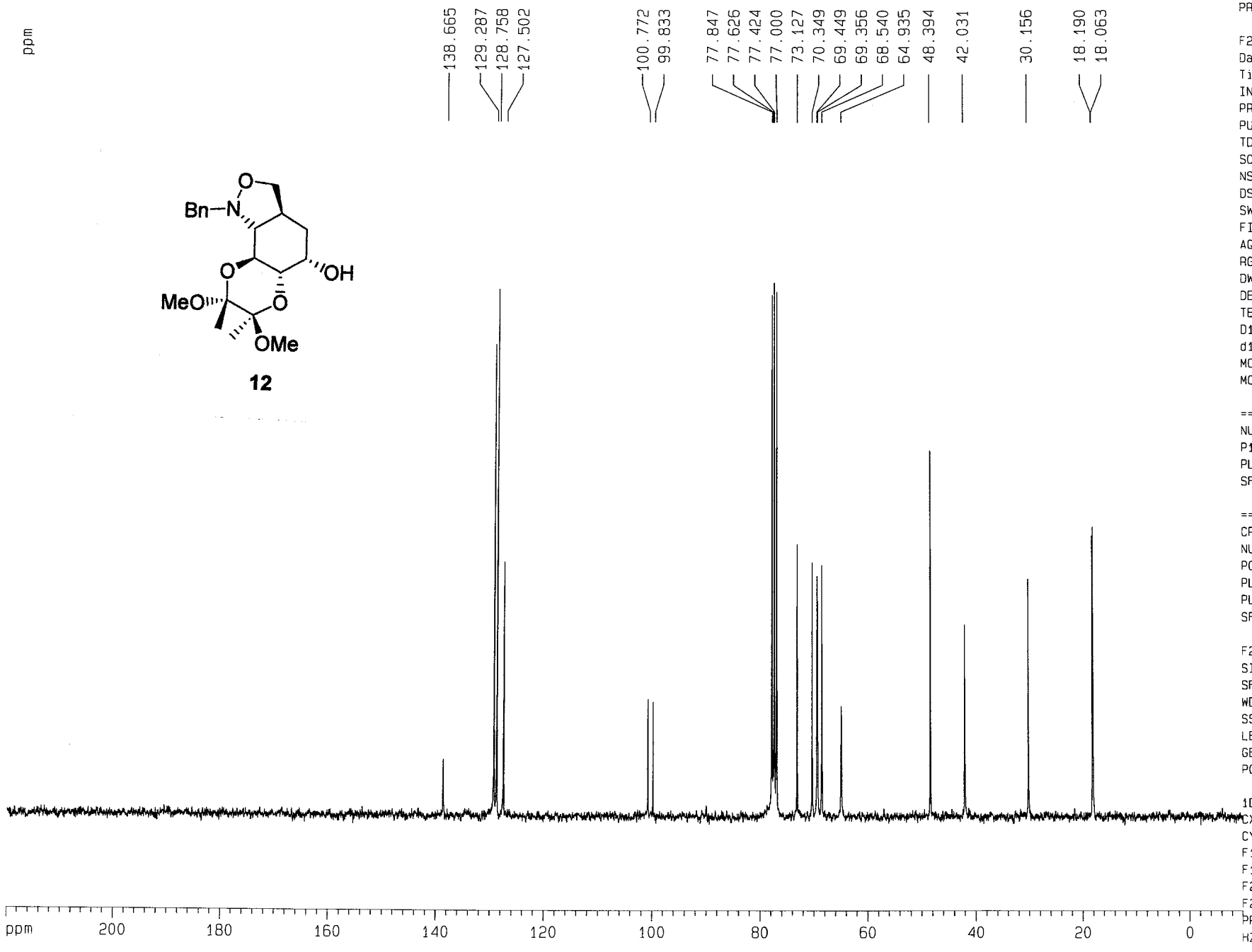
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.66 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf55cat2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050317
Time	13.53
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	1379
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	1448.2
DW	18.900 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

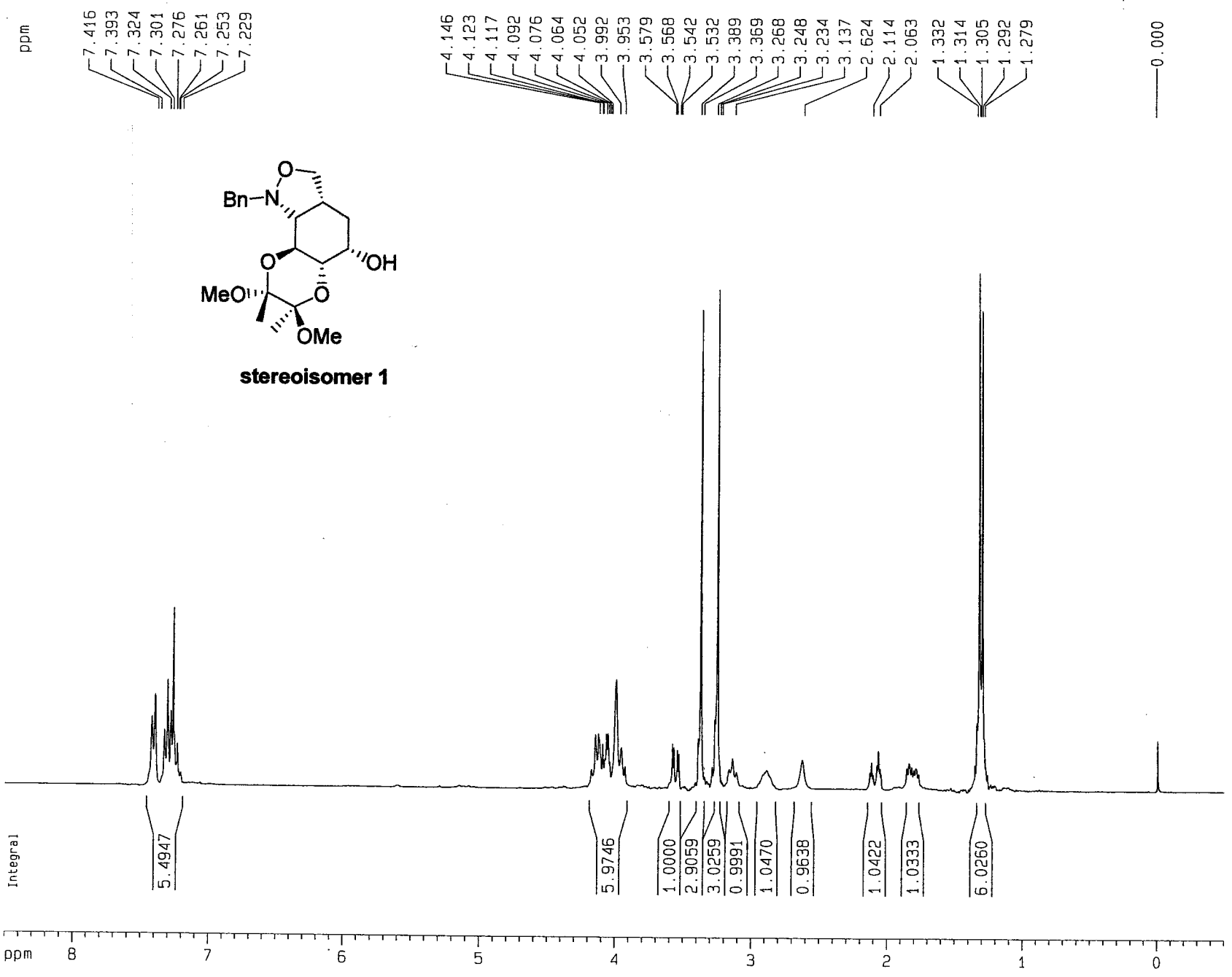
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677204 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	9.75 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67719 Hz/cm



Current Data Parameters

NAME	wwf57
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050412
Time	18.51
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	71.8
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

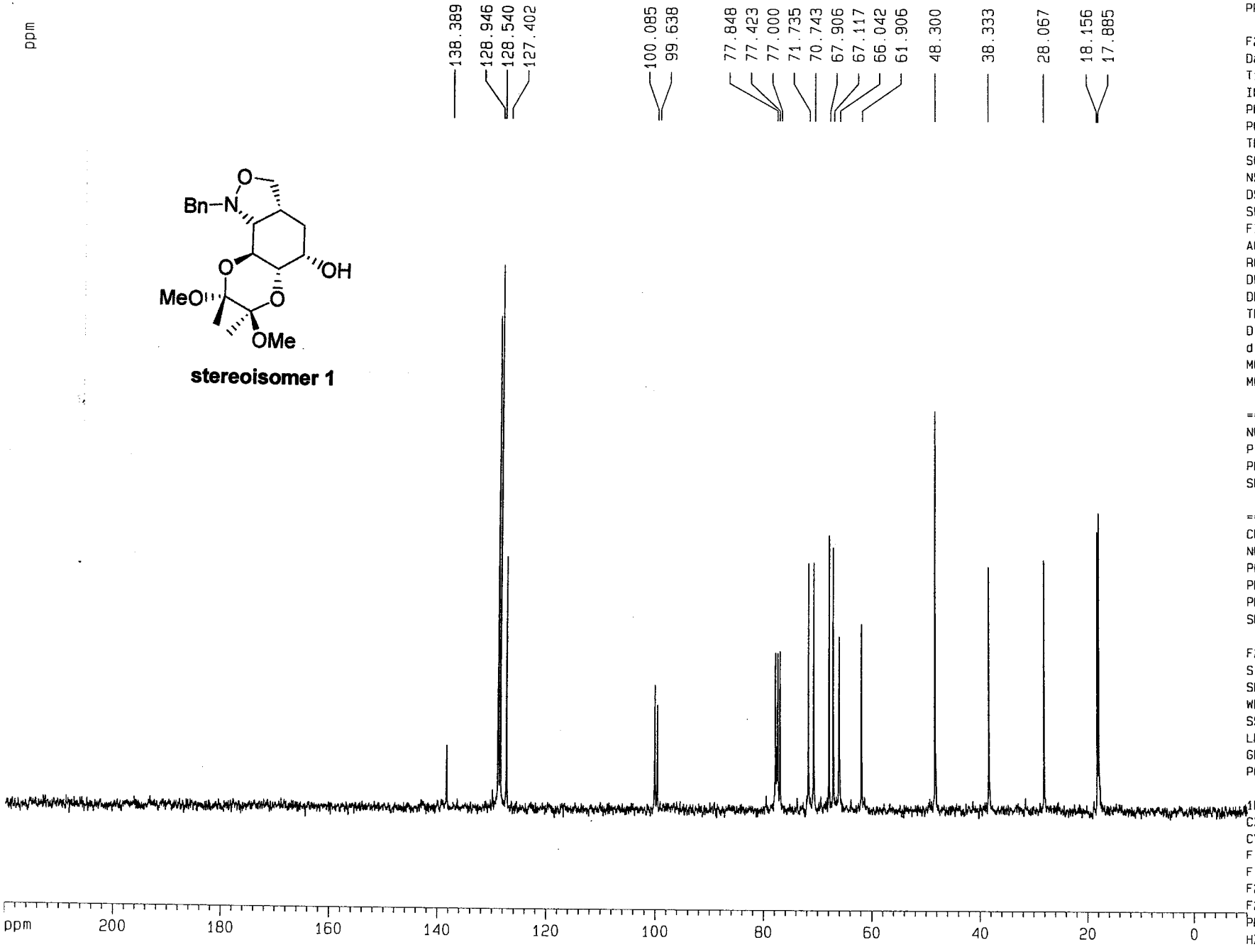
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	9.56 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf57carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050412
Time	19.03
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	180
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	1024
DW	18.900 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

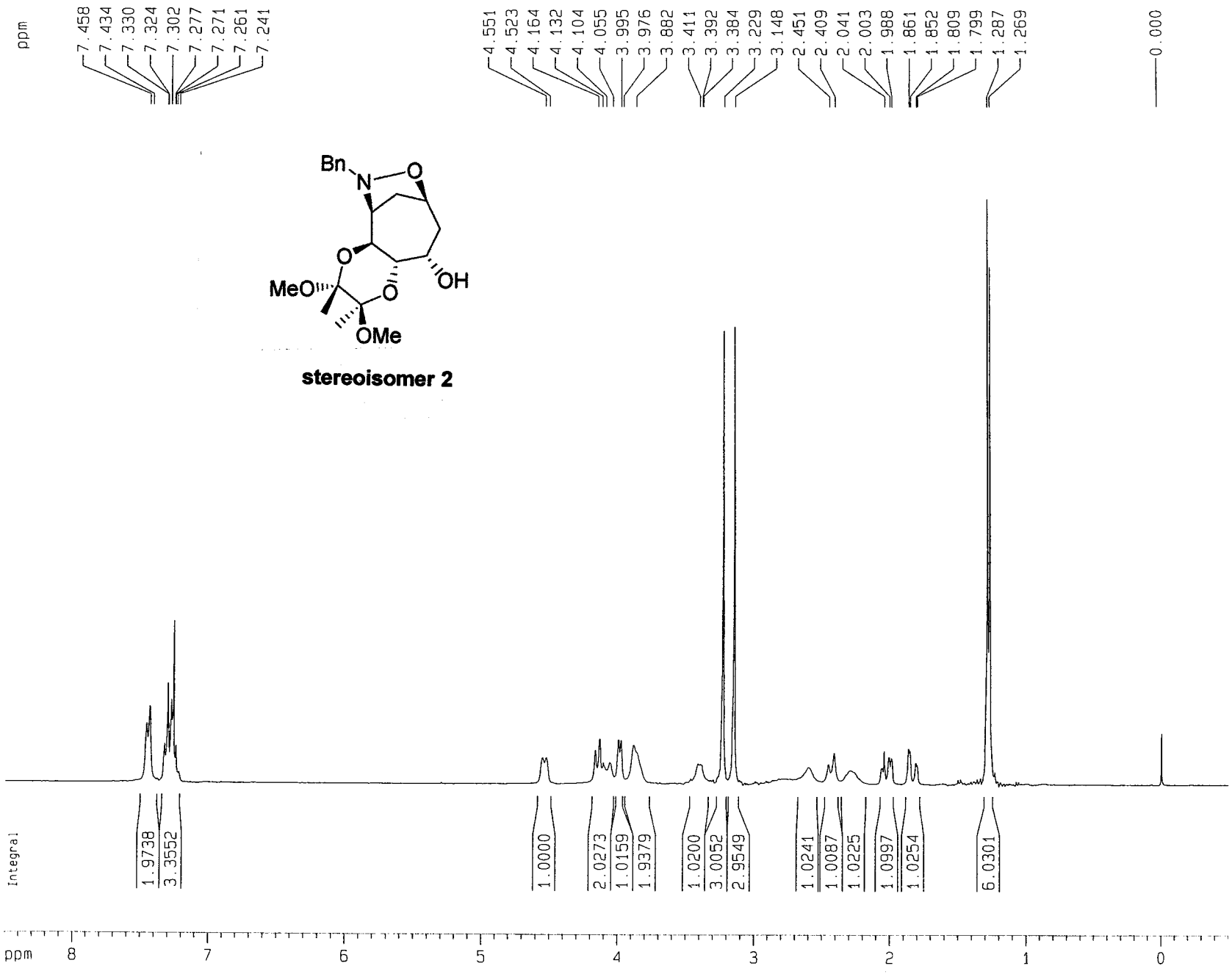
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677244 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	10.05 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67725 Hz/cm



Current Data Parameters

NAME	wwf54c
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050407
Time	18.50
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	80.6
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

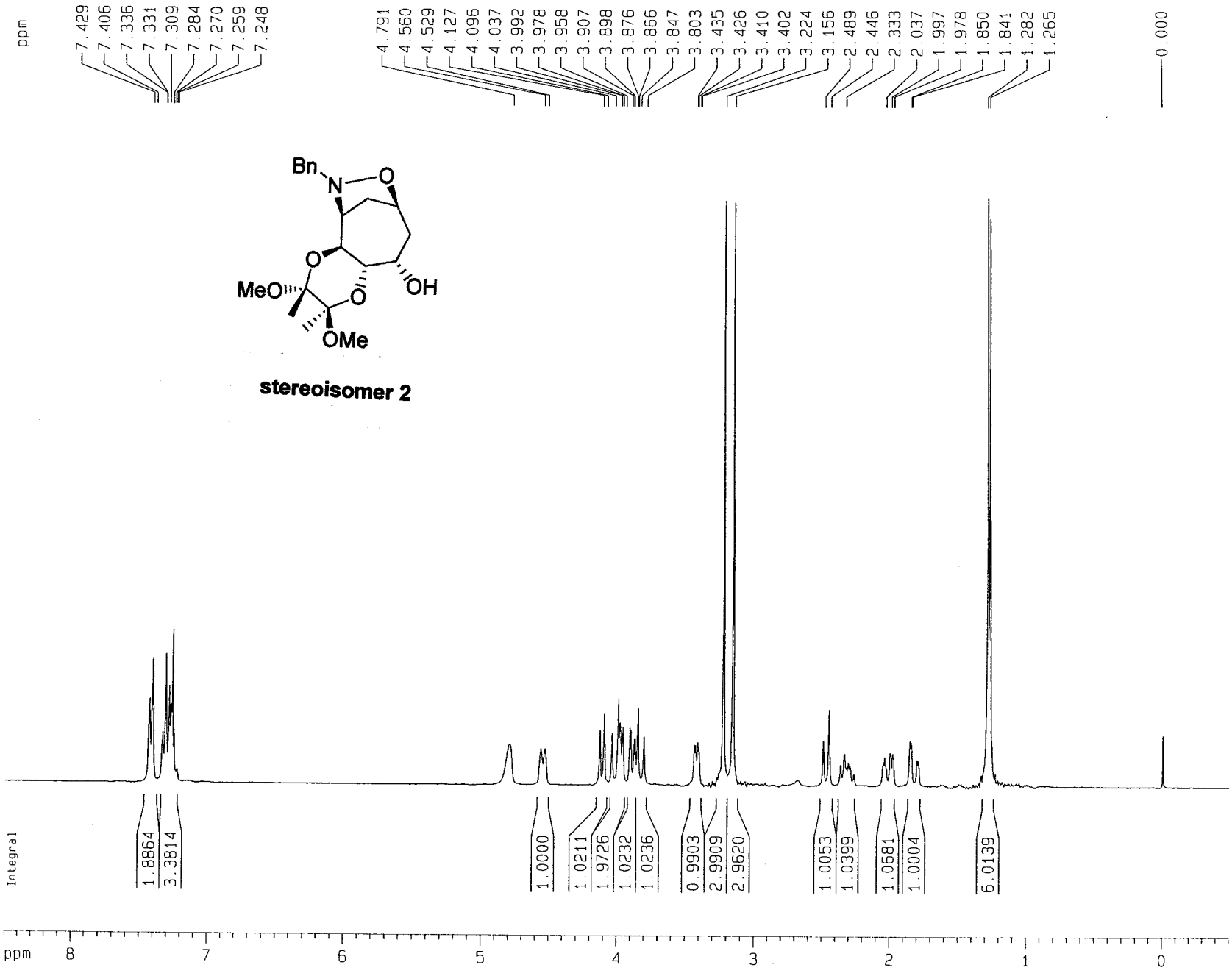
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.62 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm

Solvent: $CDCl_3$ with D_2O



Current Data Parameters

NAME	wwf54c-d20
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050407
Time	18.54
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	71.8
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

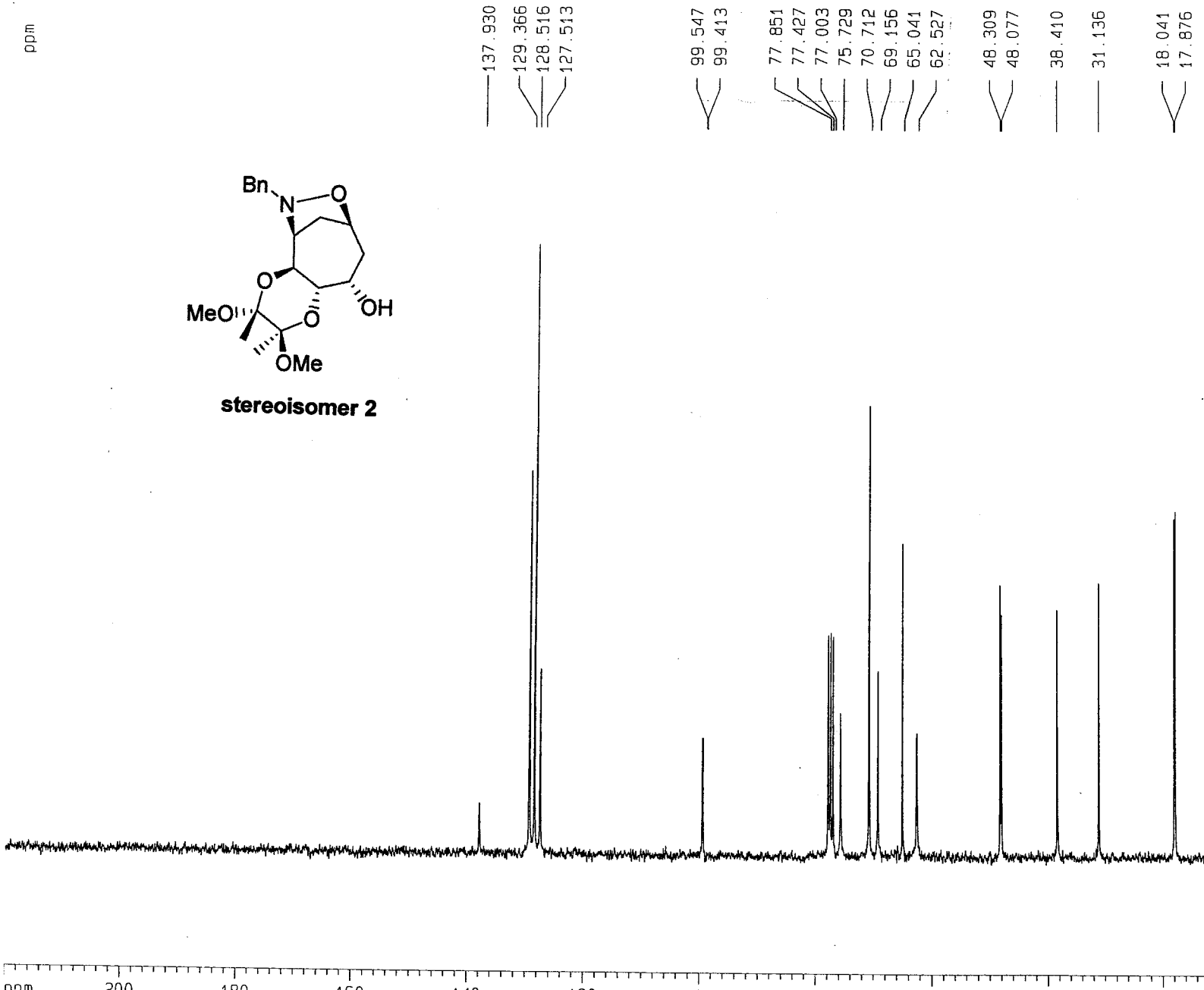
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.59 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf54-carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050330
Time	20.31
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	443
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	1824.6
DW	18.900 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

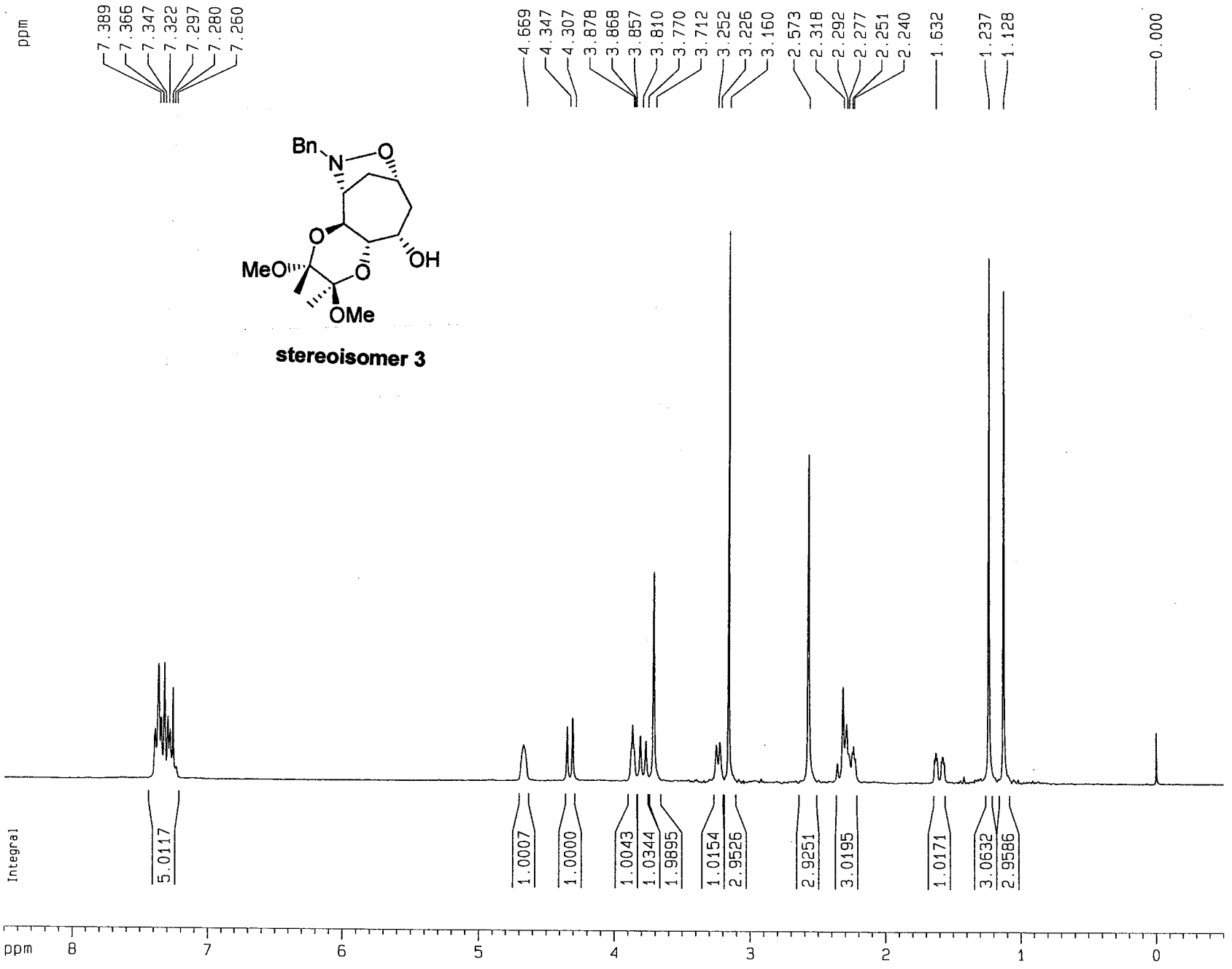
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677196 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	10.68 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67719 Hz/cm



Current Data Parameters

NAME	wwf56
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050409
Time	13.49
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	80.6
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

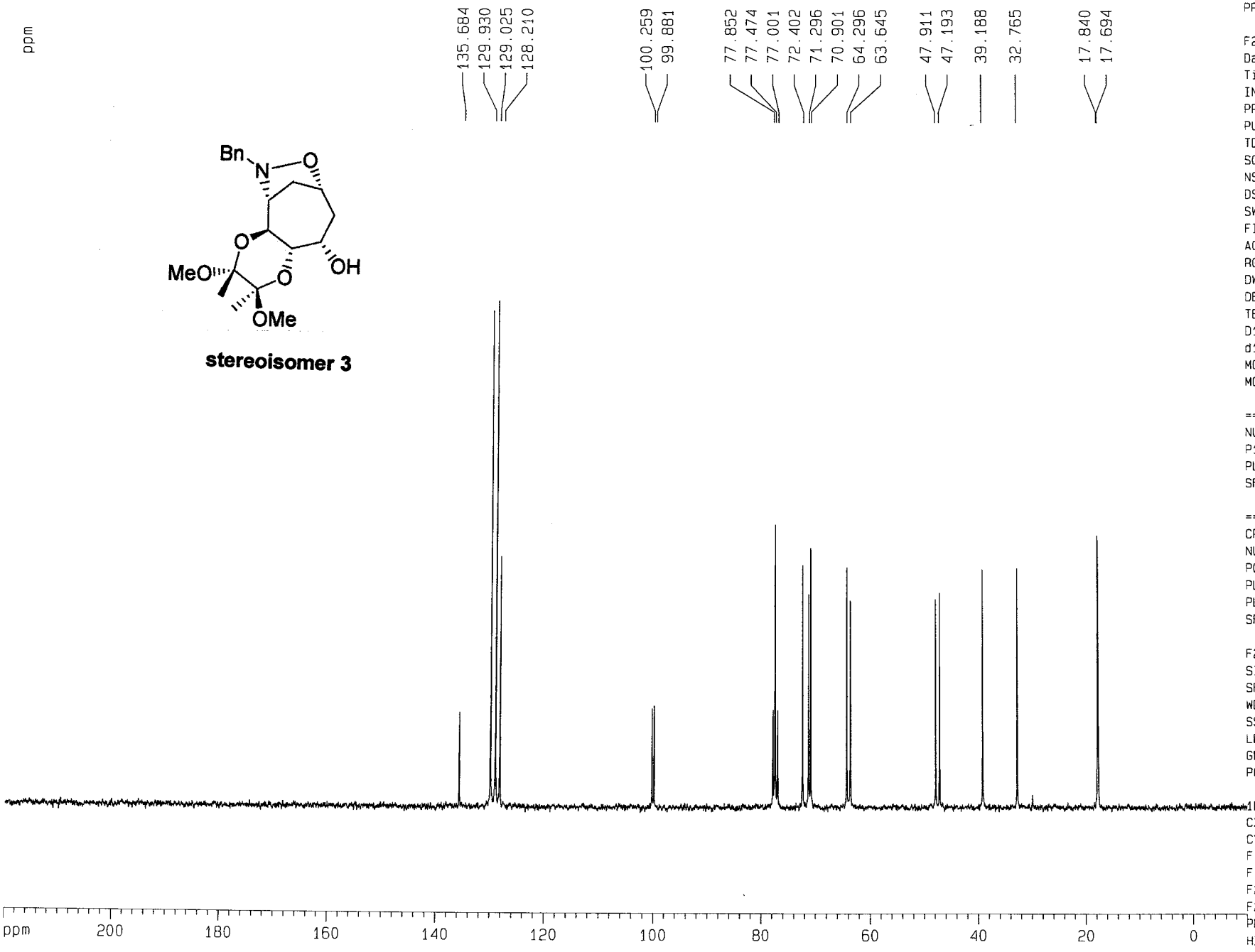
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.08 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf56carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050409
Time	13.55
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	142
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	8192
DW	18.900 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

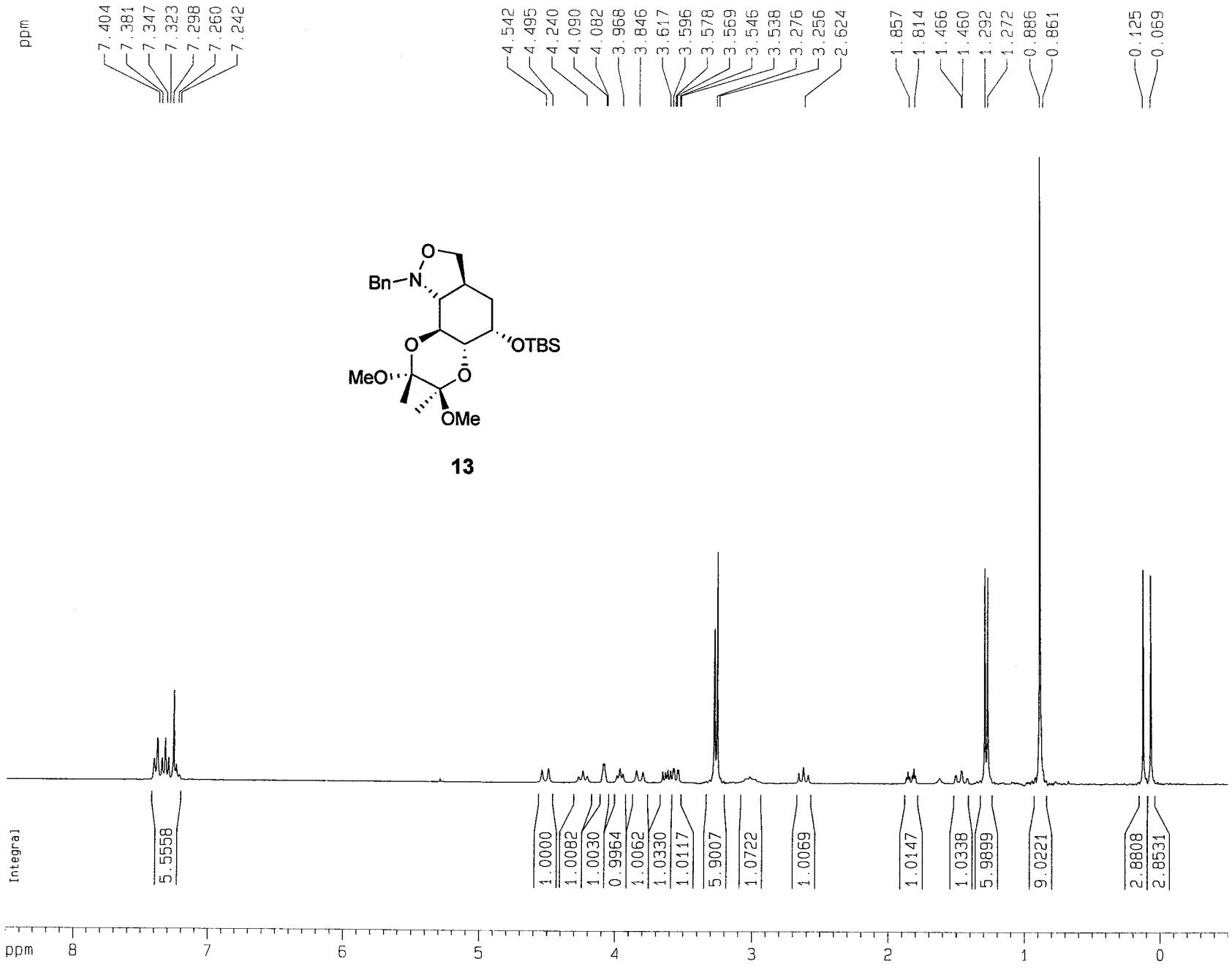
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677297 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	9.42 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67725 Hz/cm



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Current Data Parameters

NAME	wwf59d
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050321
Time	13.22
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	64
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

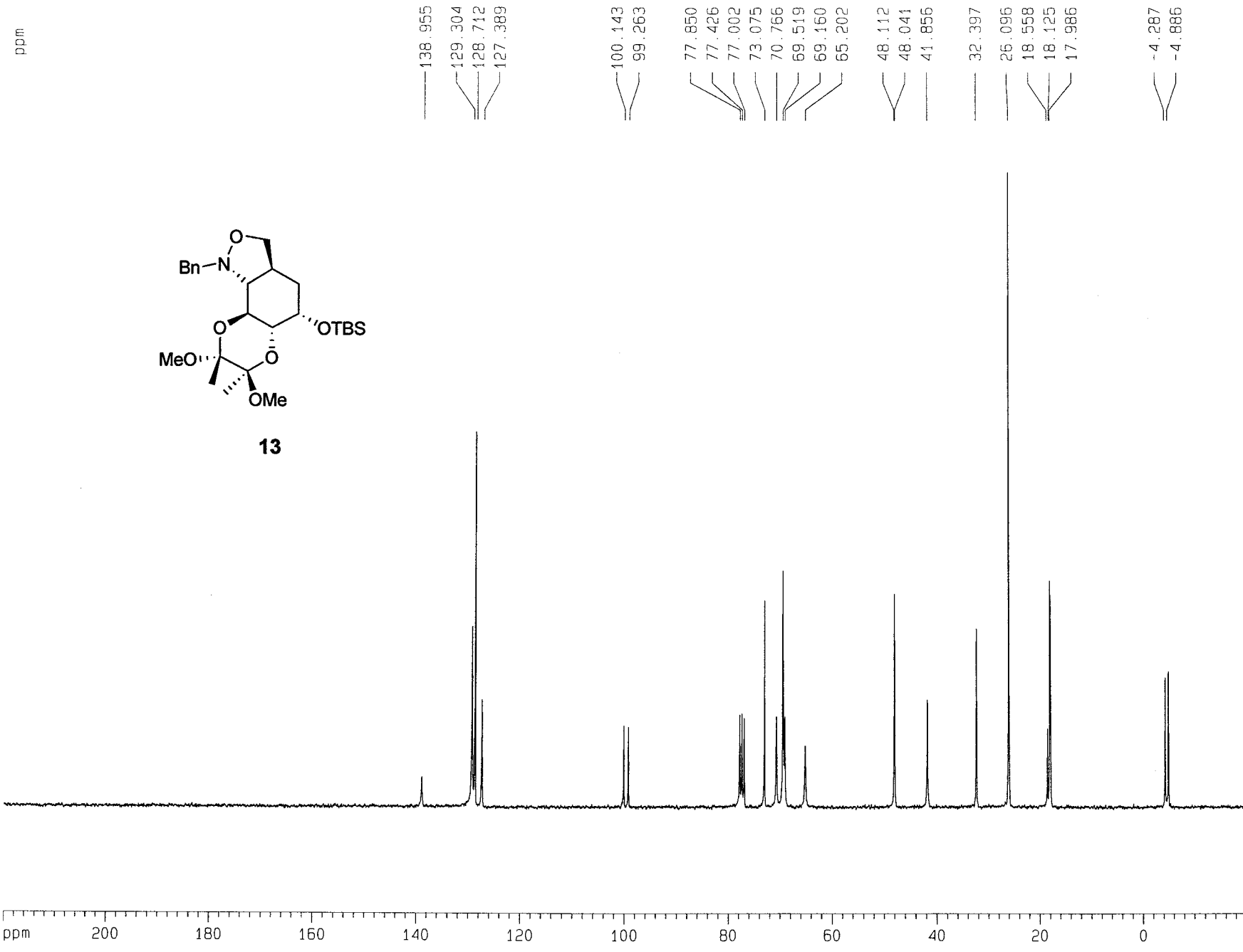
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.31 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf59car3
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050401
Time	12.06
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	644
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	9195.2
DW	18.900 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

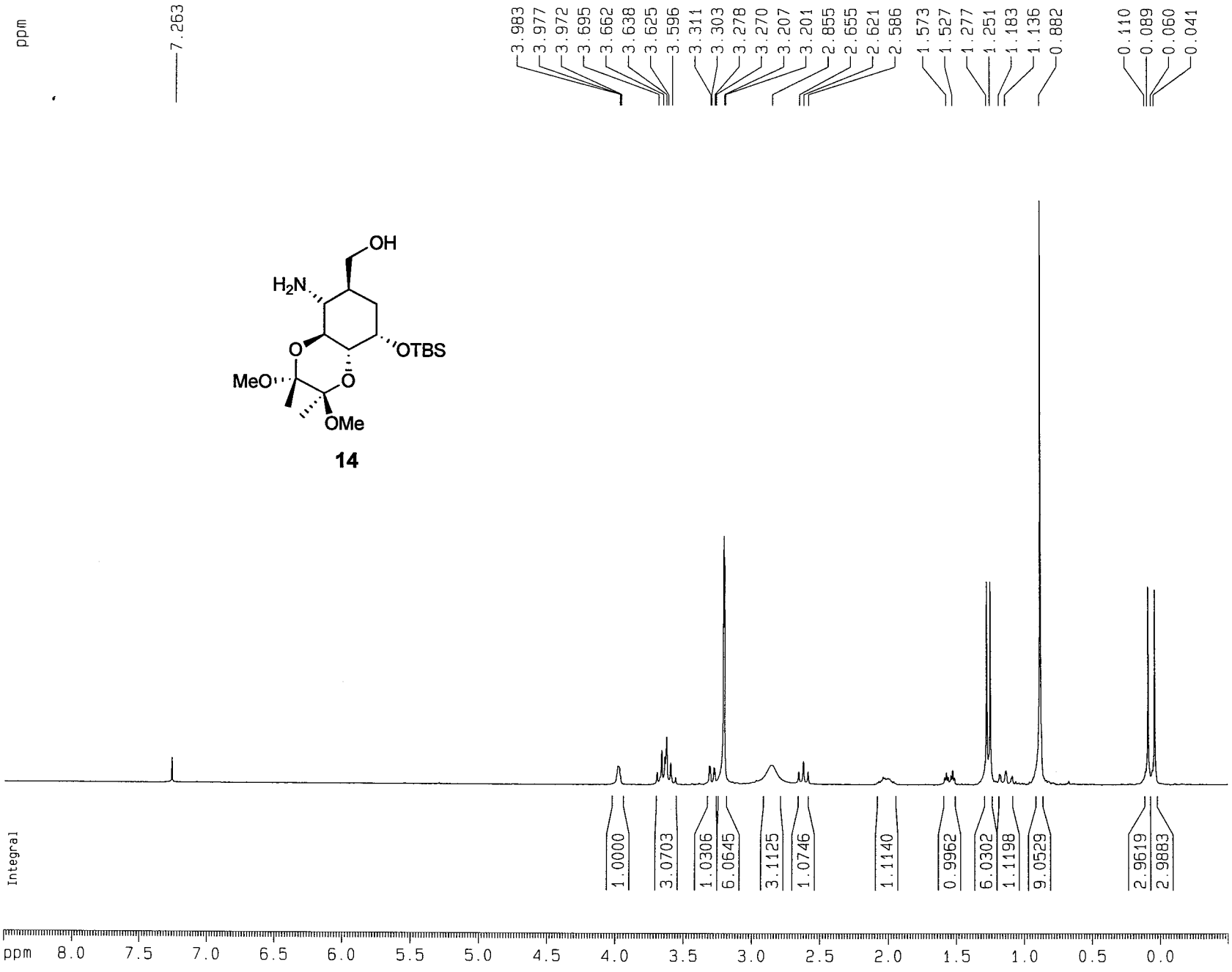
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677248 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	11.80 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-20.000 ppm
F2	-1509.36 Hz
PPMCM	10.43478 ppm/cm
HZCM	787.48938 Hz/cm



Current Data Parameters

NAME	wwf78
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050622
Time	18.35
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	114
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SF01	300.1312000 MHz

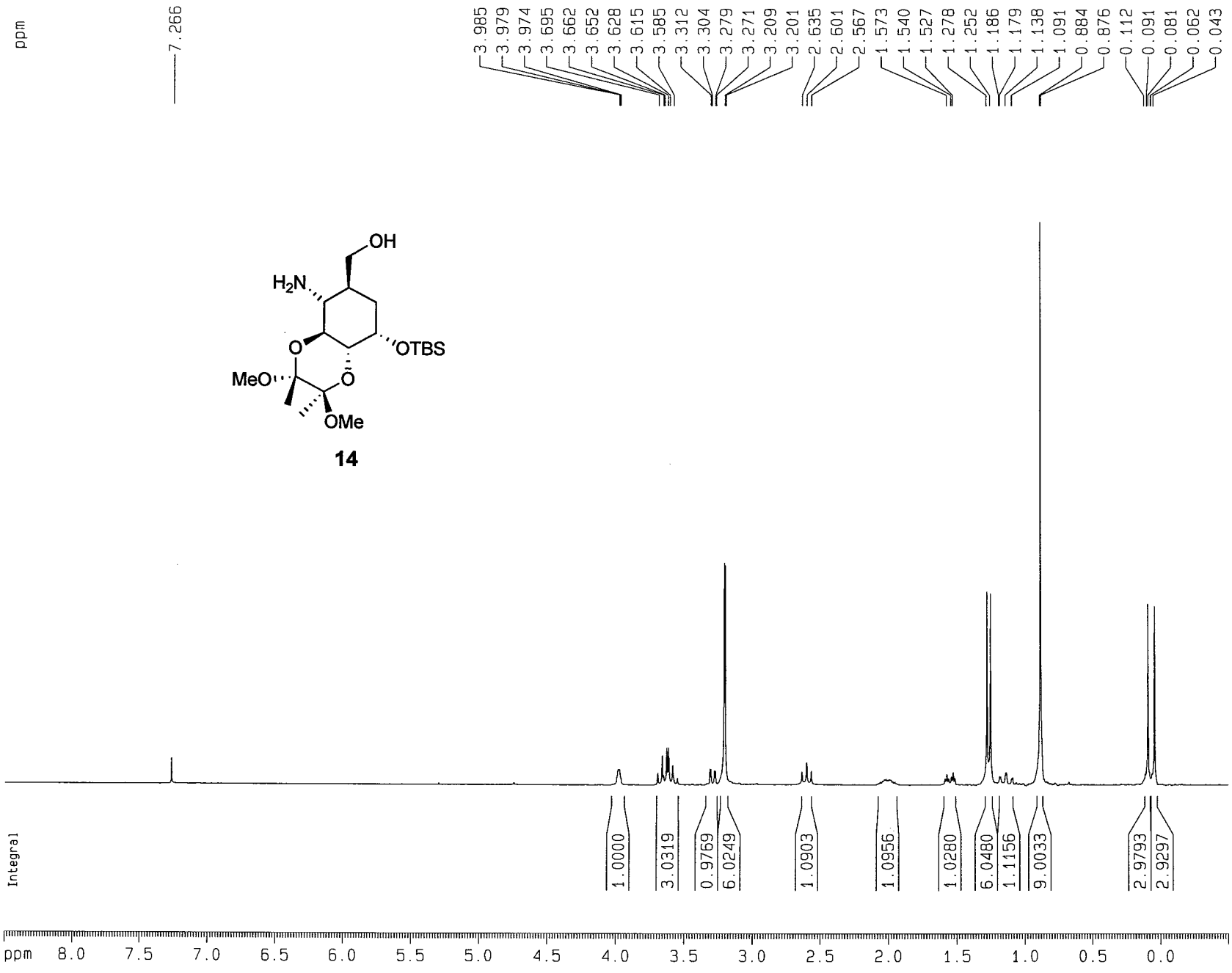
F2 - Processing parameters

SI	32768
SF	300.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.66 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm

Solvent: $CDCl_3$ with D_2O



Current Data Parameters

NAME	wwf78d20
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050622
Time	18.40
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	114
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

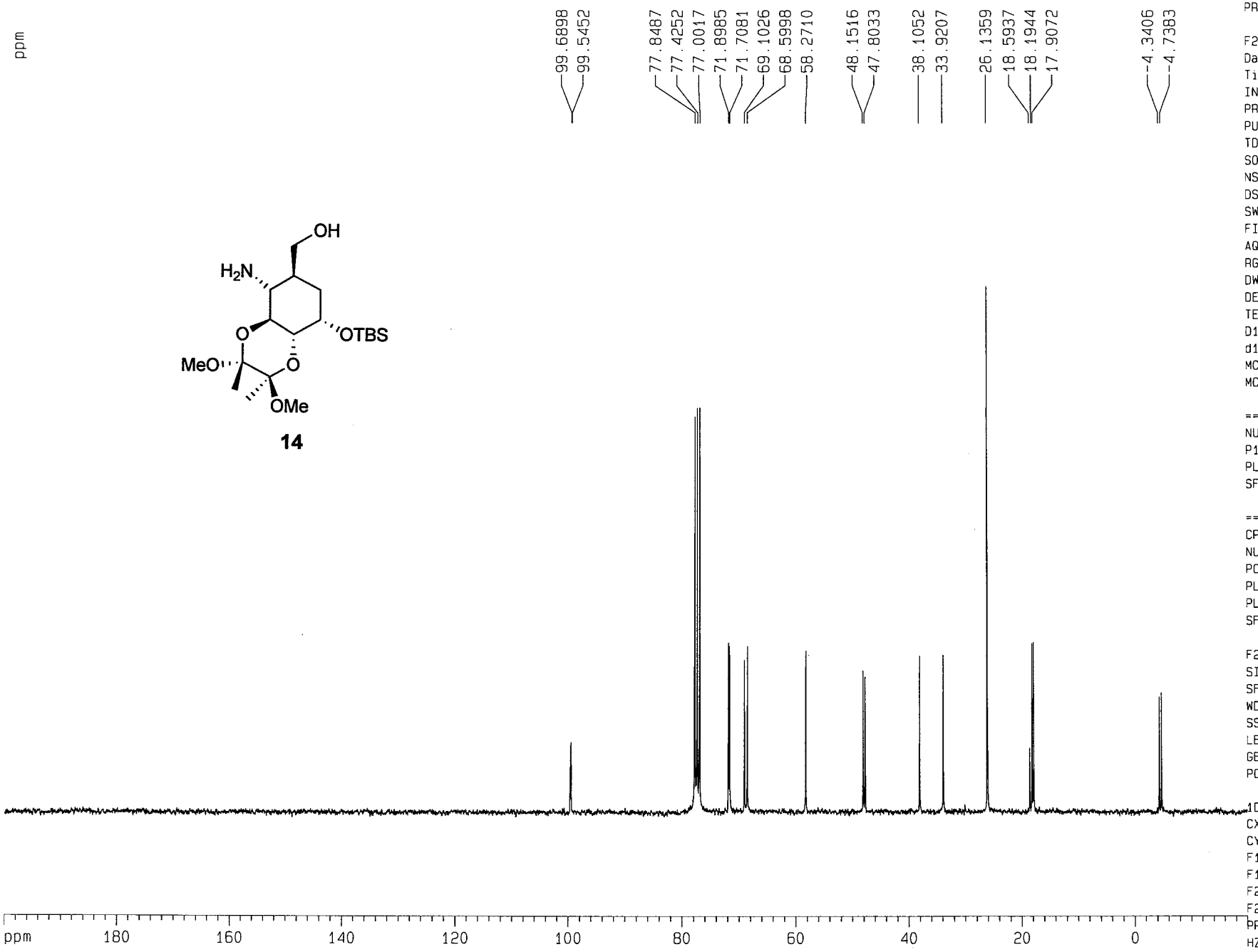
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SF01	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.25 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	wwf78carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050622
Time	18.50
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	2675
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	2298.8
DW	18.900 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

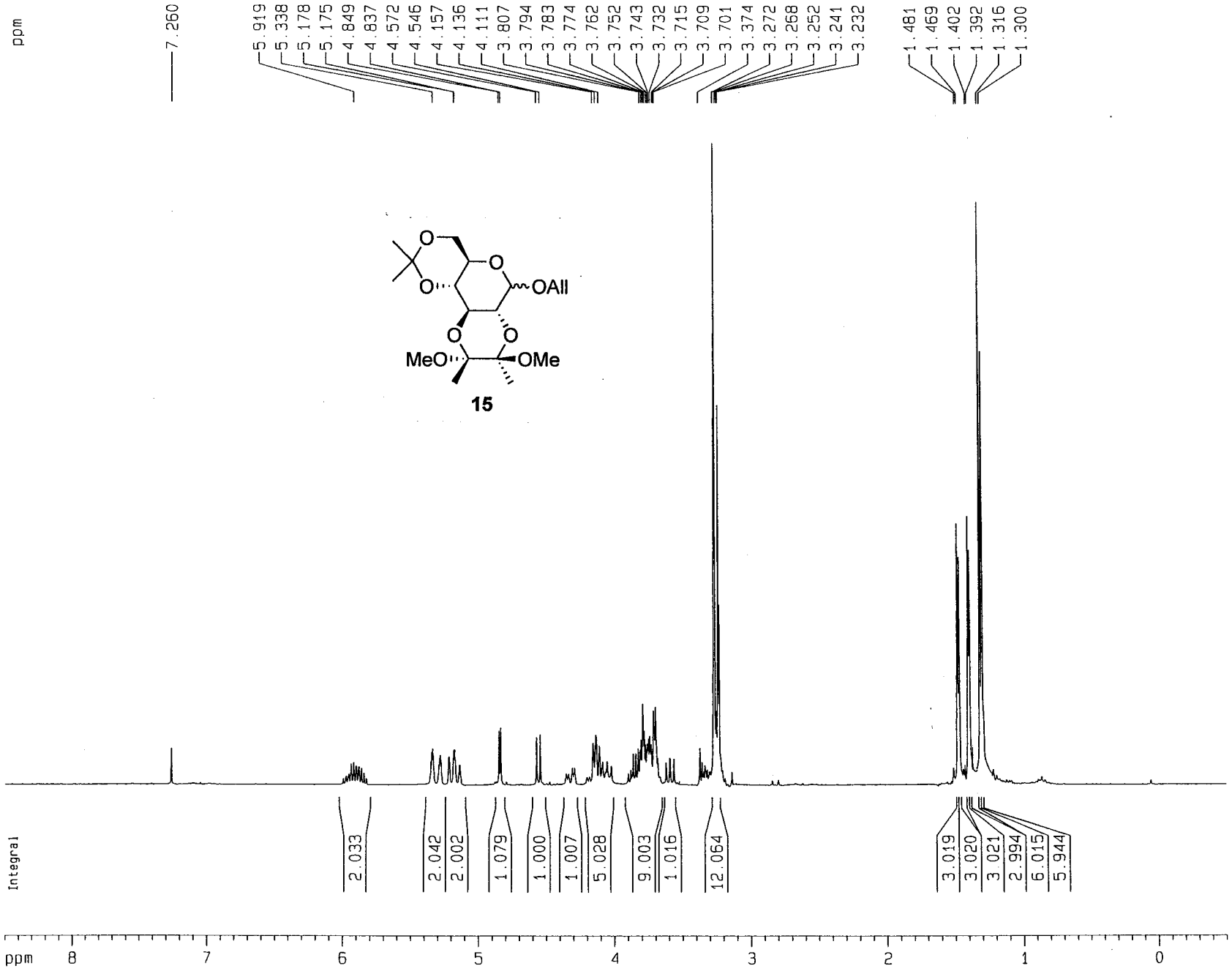
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677200 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	9.72 cm
F1P	200.000 ppm
F1	15093.54 Hz
F2P	-20.000 ppm
F2	-1509.35 Hz
PPMCM	9.56522 ppm/cm
HZCM	721.86517 Hz/cm



Current Data Parameters

NAME	GLal103
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050718
Time	13.05
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	64
DW	55.600 usec
DE	79.43 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

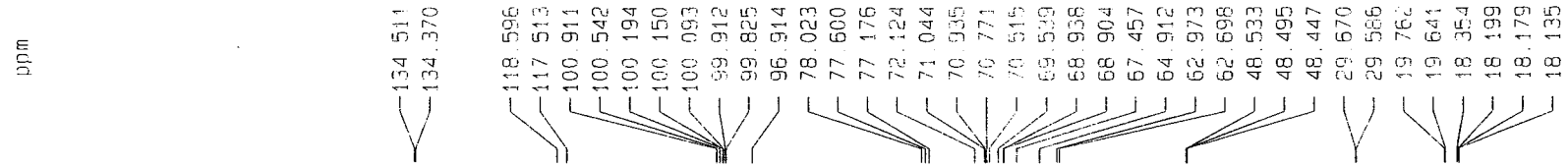
F2 - Processing parameters

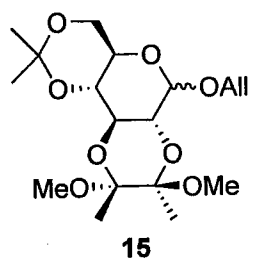
SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.54 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm

C13





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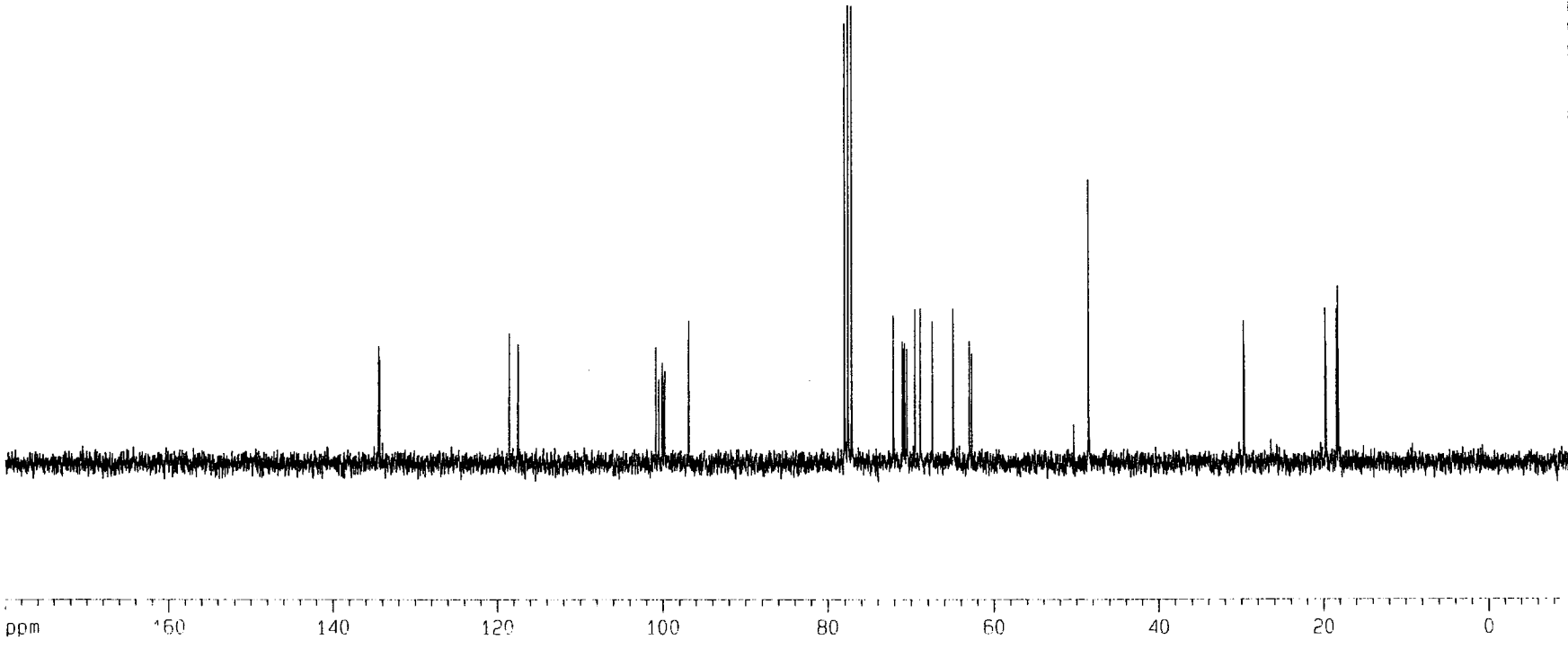


Current Data Parameters

NAME	GLal103c13
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20050718
Time	13.10
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	114
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	8192
DW	18.900 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

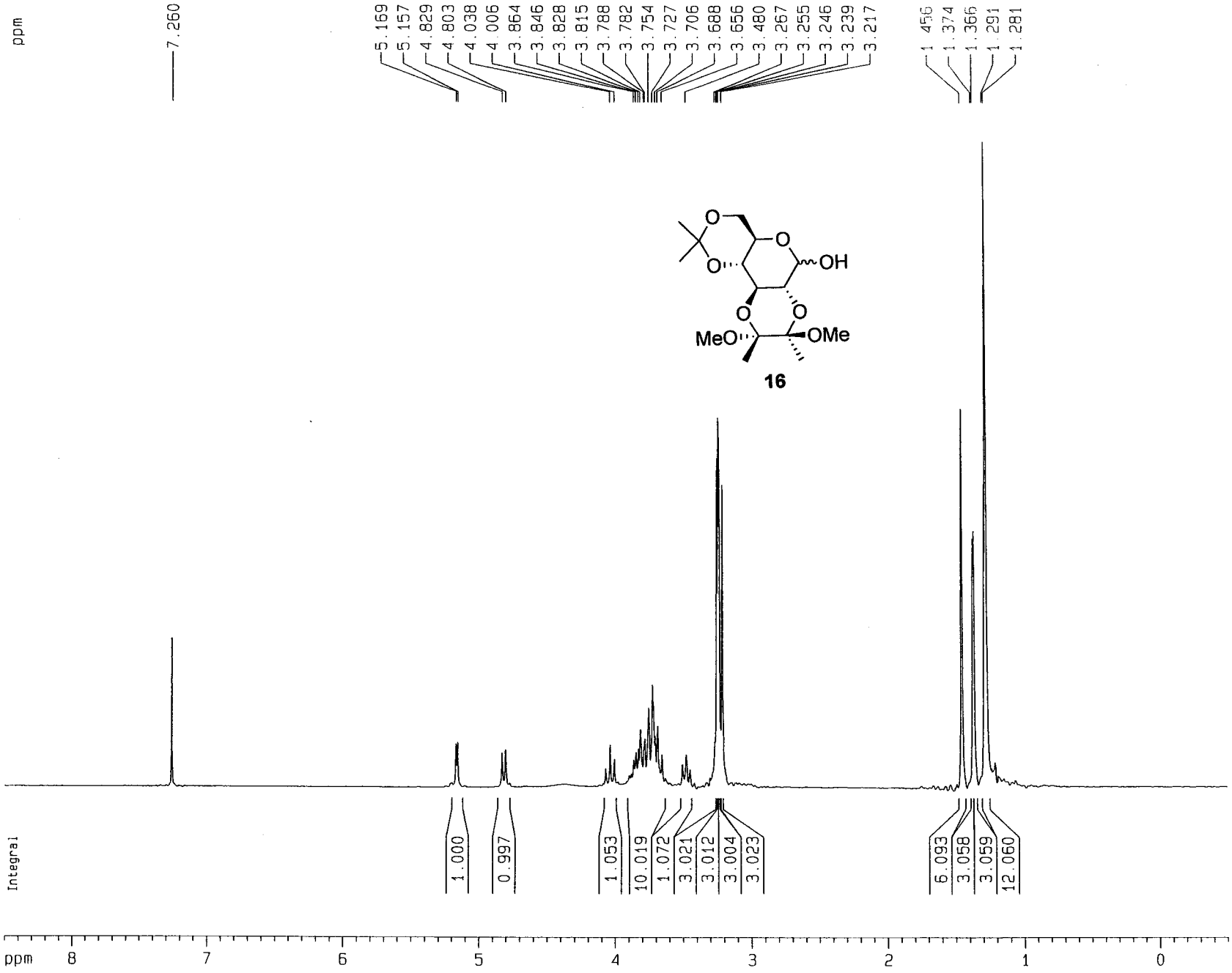
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677070 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	6.73 cm
F1P	180.000 ppm
F1	13584.19 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	8.26087 ppm/cm
HZCM	623.42889 Hz/cm



Current Data Parameters

NAME	GL37-h1-data
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060619
Time	19.09
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	4803.074 Hz
FIDRES	0.146578 Hz
AQ	3.4111989 sec
RG	28.5
DW	104.100 usec
DE	148.71 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

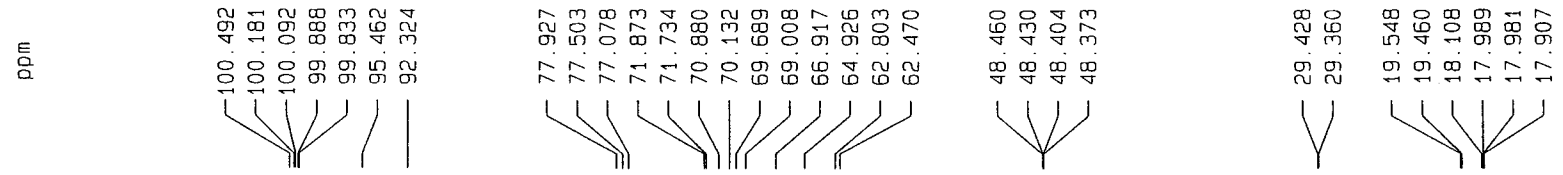
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

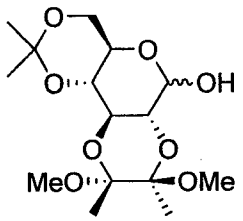
F2 - Processing parameters

SI	32768
SF	300.1300067 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.51 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm





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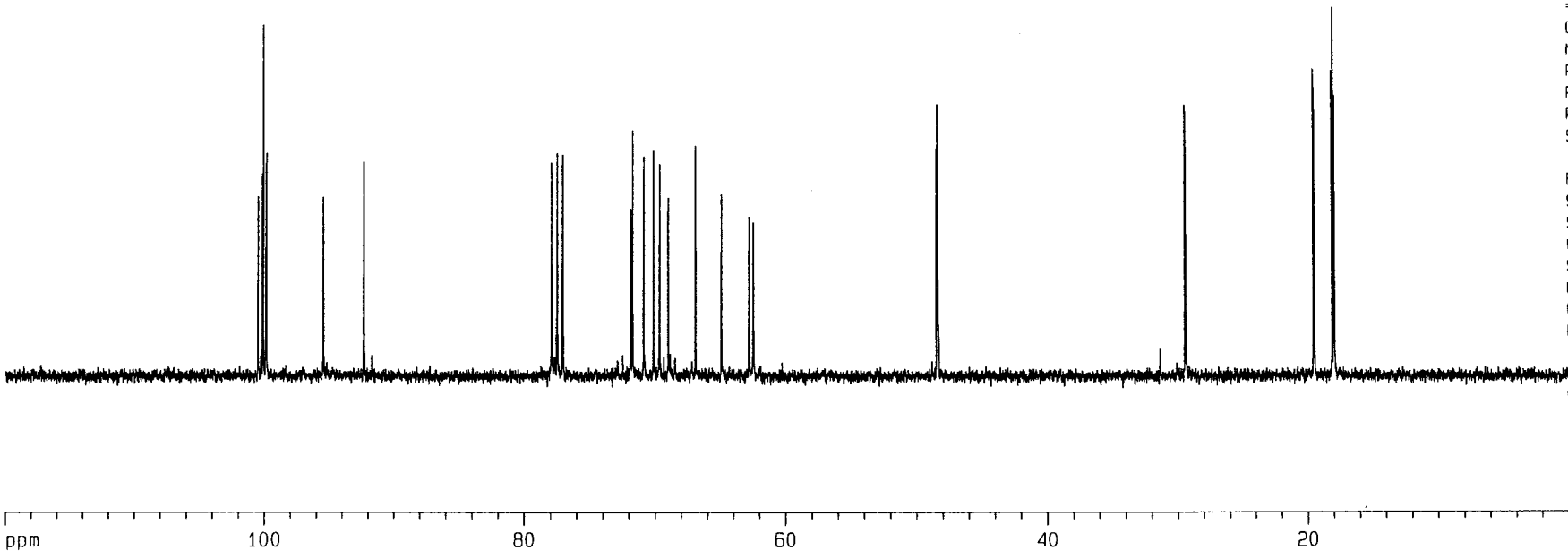


Current Data Parameters

NAME	GL37c13
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20060619
Time	19.15
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	143
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	10321.3
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

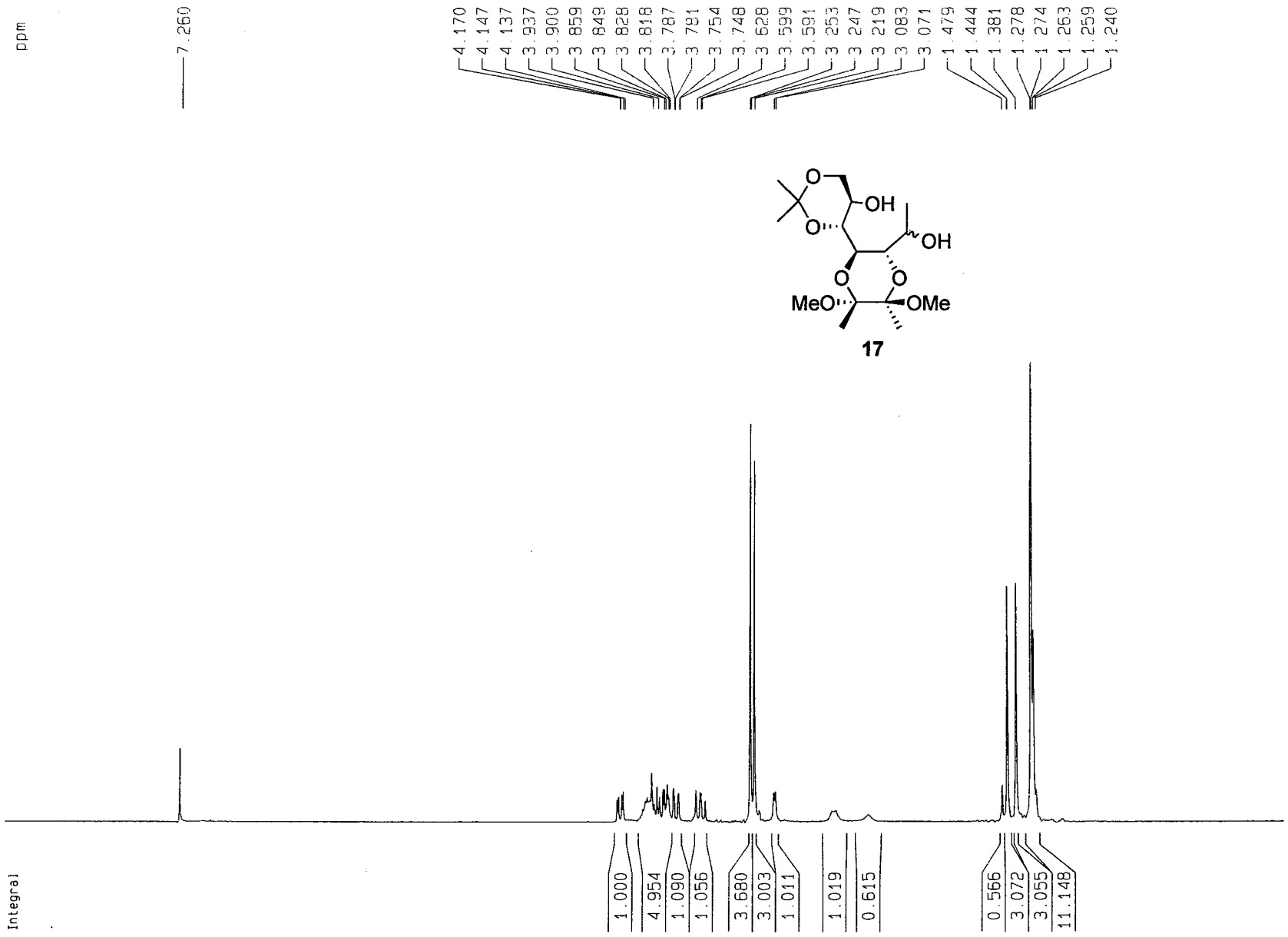
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677174 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	5.41 cm
F1P	120.000 ppm
F1	9056.13 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	5.21739 ppm/cm
HZCM	393.74463 Hz/cm



Current Data Parameters

NAME	GL39
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050421
Time	10.44
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	12288
SOLVENT	CDCl3
NS	4
DS	0
SWH	4803.074 Hz
FIDRES	0.390875 Hz
AQ	1.2792308 sec
RG	64
DW	104.100 usec
DE	148.71 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

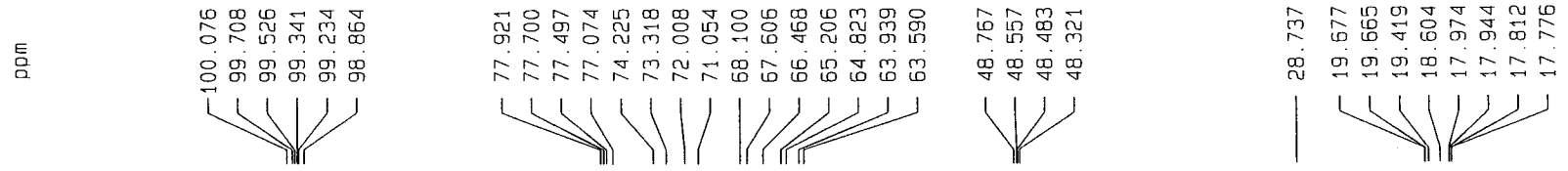
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

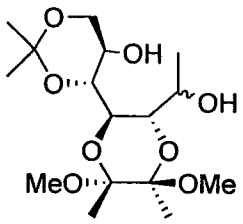
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	7.88 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm





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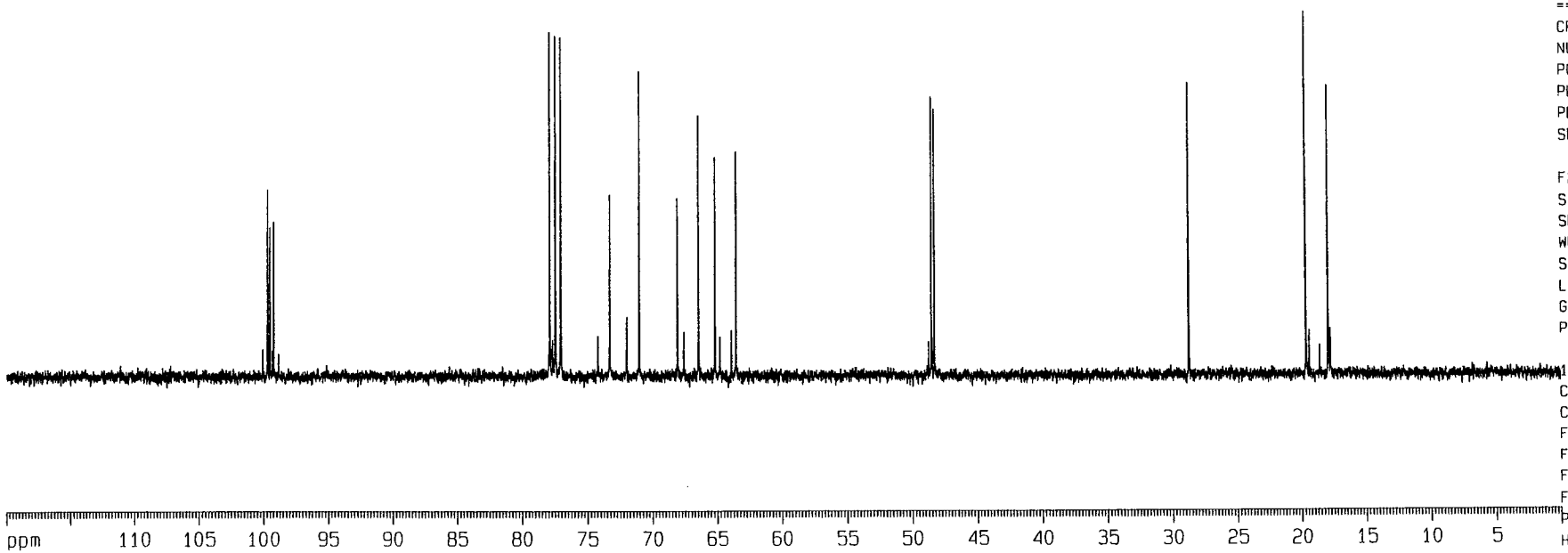


Current Data Parameters

NAME	GL39c13
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20050421
Time	10.50
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	189
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	8192
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

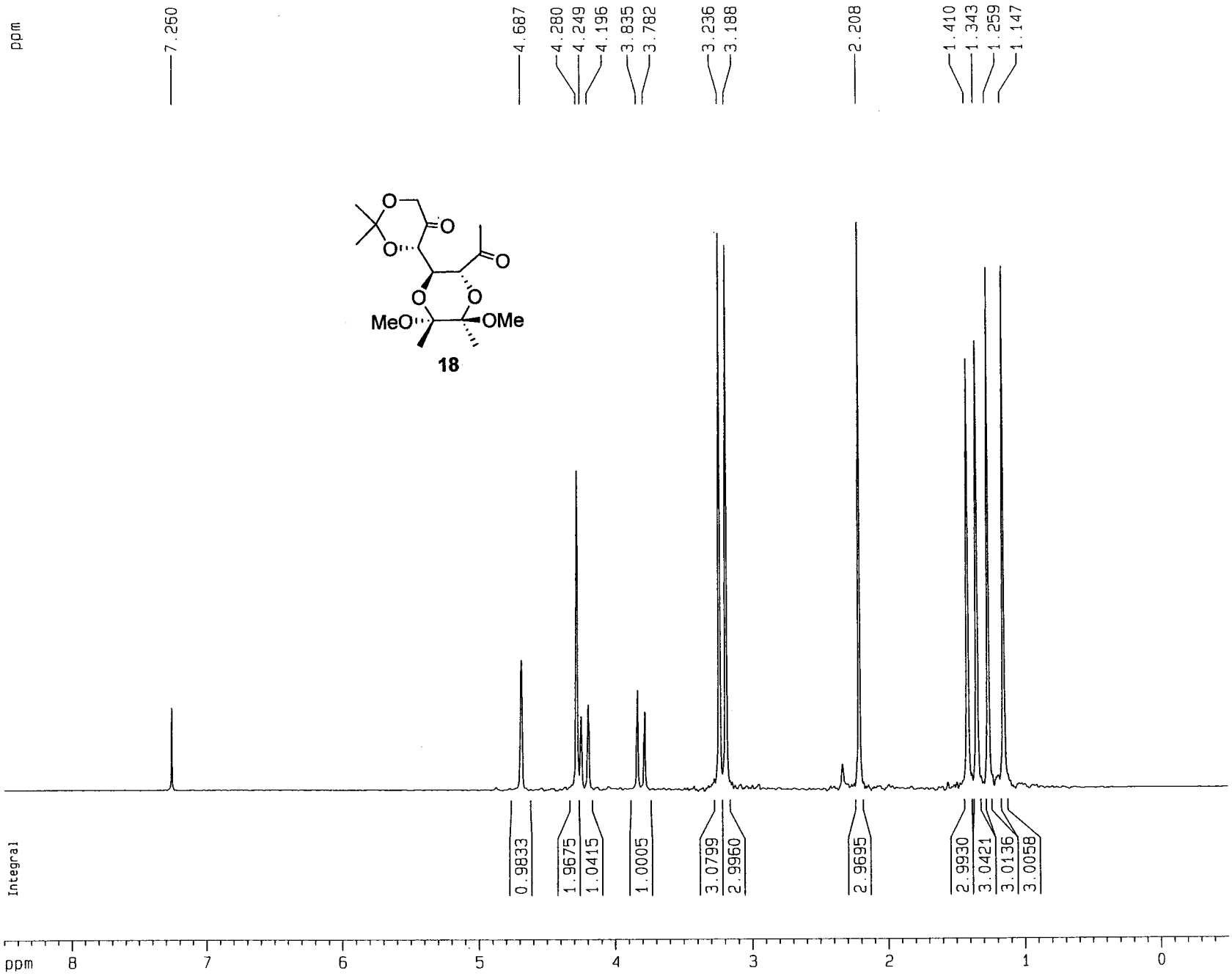
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677150 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	5.34 cm
F1P	120.000 ppm
F1	9056.12 Hz
F2P	-0.000 ppm
F2	-0.00 Hz
PPMCM	5.21739 ppm/cm
HZCM	393.74460 Hz/cm



Current Data Parameters

NAME	GL40-h1-data
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060703
Time	19.47
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	C6D6
NS	4
DS	0
SWH	4803.074 Hz
FIDRES	0.146578 Hz
AQ	3.4111989 sec
RG	28.5
DW	104.100 usec
DE	148.71 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300060 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.18 cm
F1P	8.500 ppm
F1	2551.11 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78047 Hz/cm

ppm

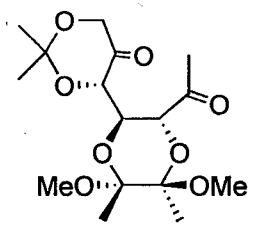
207.746
207.640

101.468
99.767
99.314

77.926
77.501
77.078
74.852
72.814
67.521
66.623

48.802
48.698

27.406
24.957
23.928
18.040
17.776



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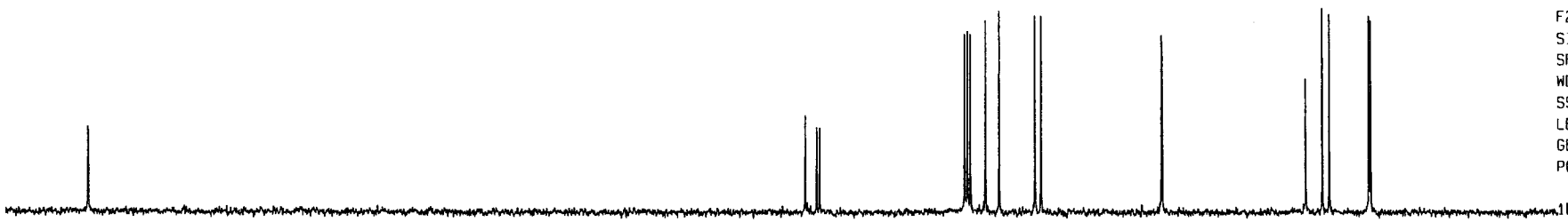


ppm 200 180 160 140 120 100 80 60 40 20 0

Current Data Parameters

NAME	GL40c13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060605
Time	19.33
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	229
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	3251
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

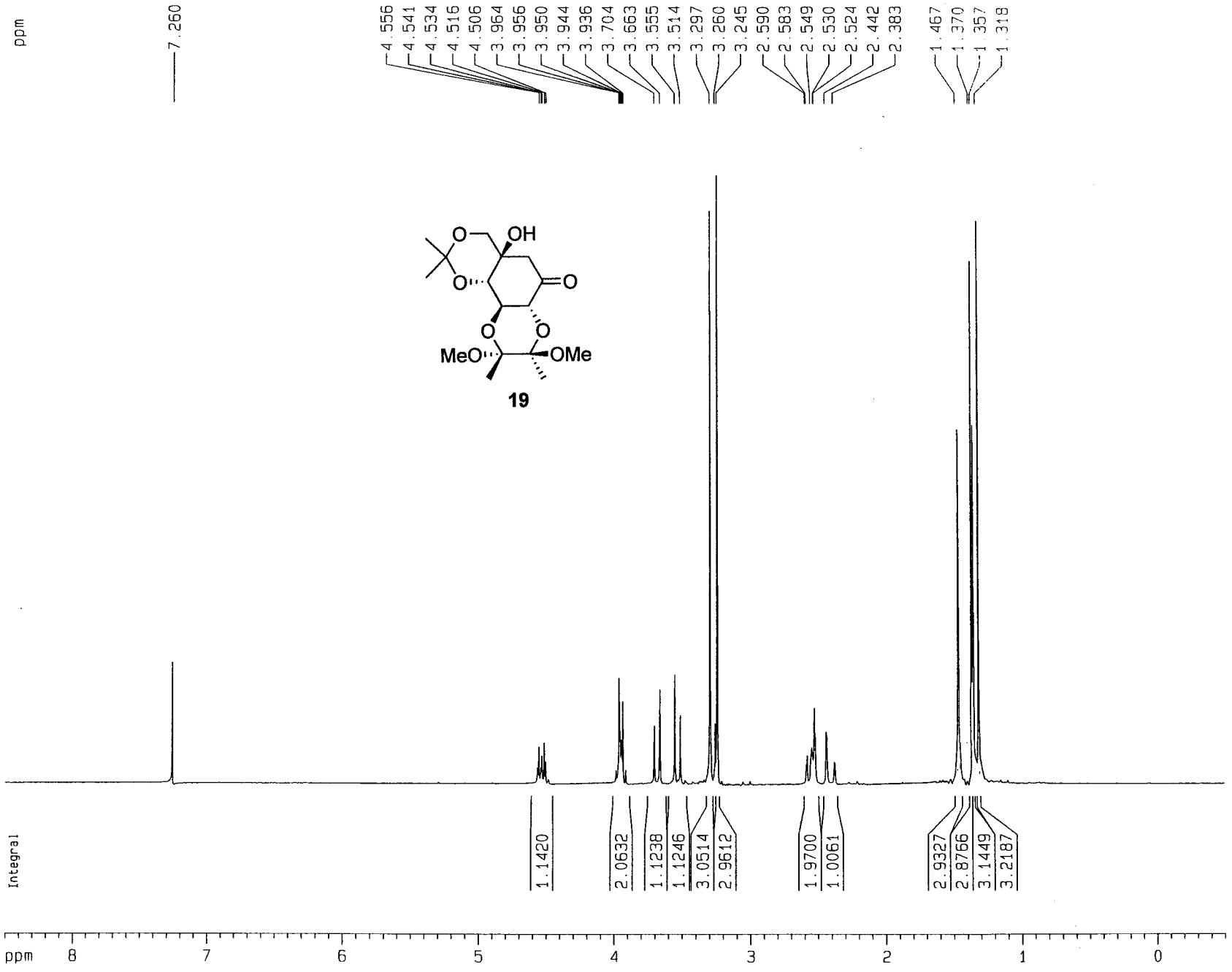
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677146 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	3.00 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67712 Hz/cm



Current Data Parameters

NAME	GL41-h1-data
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20060717
Time	19.09
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	181
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

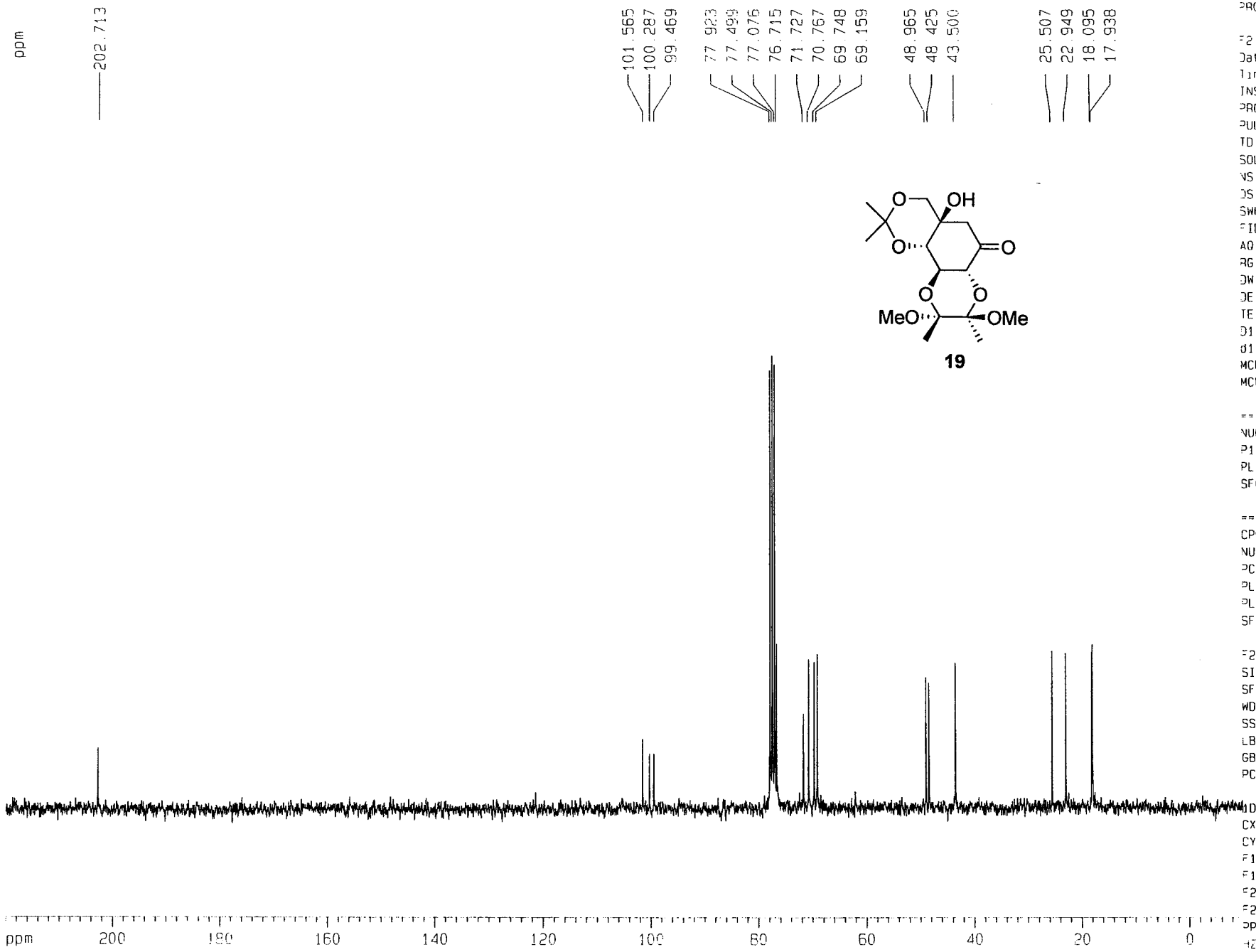
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.35 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.01 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters
NAME GL41-c13
EXPNO 1
PROCNO 1

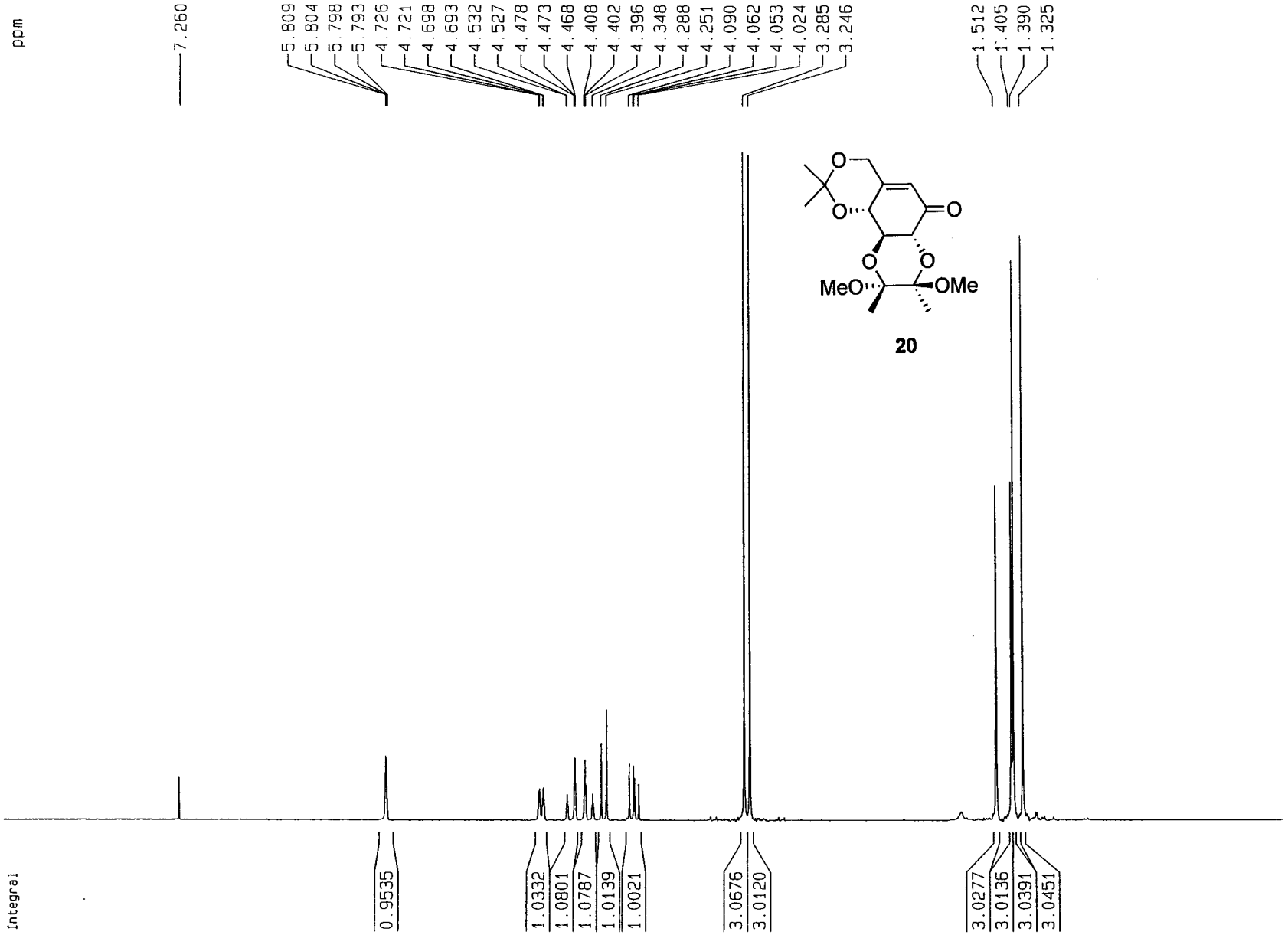
F2 - Acquisition Parameters
Date_ 20060718
Time 19.41
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 503
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 13004
DW 22.050 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677133 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 8.41 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm



Current Data Parameters

NAME	GL42-h1data
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20060728
Time	11.38
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	4
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	64
DW	55.600 usec
DE	79.43 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

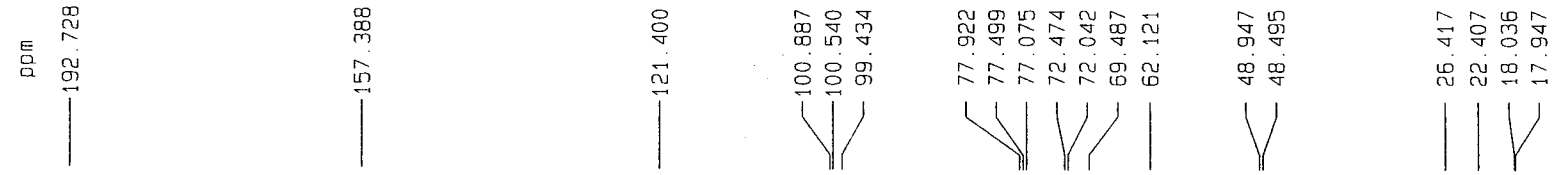
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SF01	300.1312000 MHz

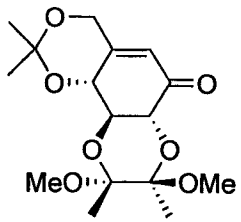
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.50 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm





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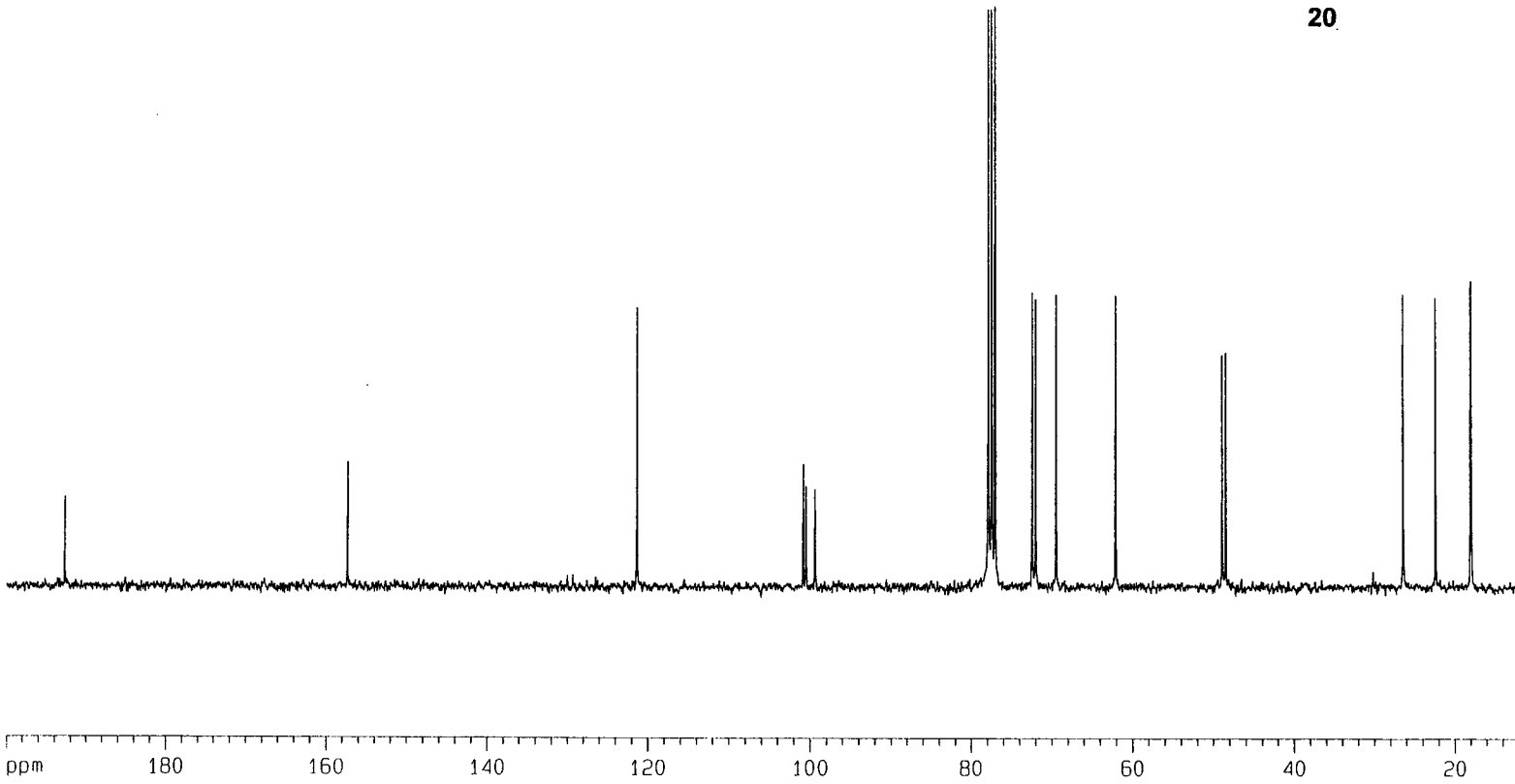


Current Data Parameters

NAME	GL421c13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050511
Time	18.54
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	1822
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	8192
DW	22.050 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

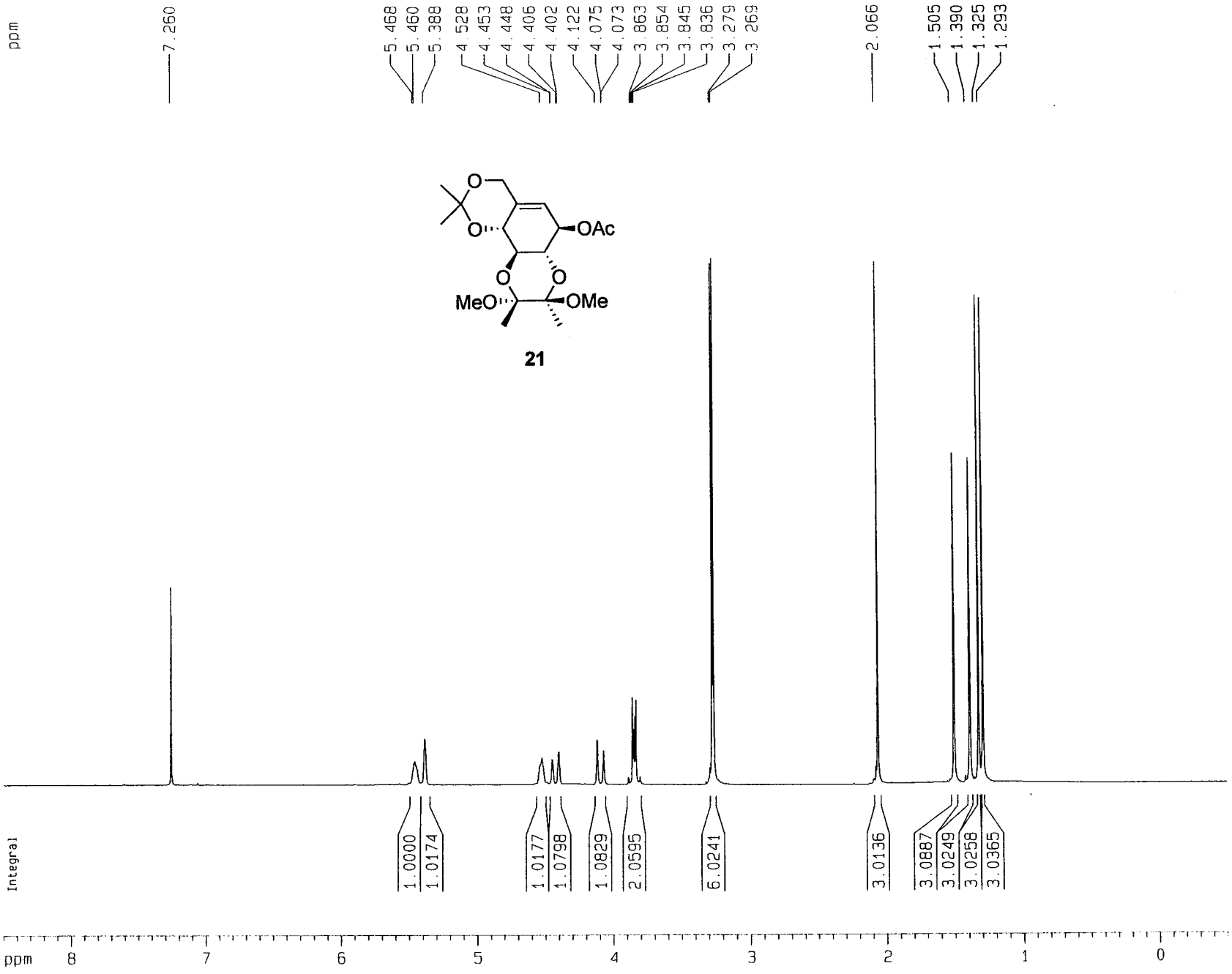
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677139 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	7.94 cm
F1P	200.000 ppm
F1	15093.54 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	9.13043 ppm/cm
HZCM	689.05304 Hz/cm



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Current Data Parameters

NAME	GL45betaOAc
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050623
Time	18.19
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	574.7
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

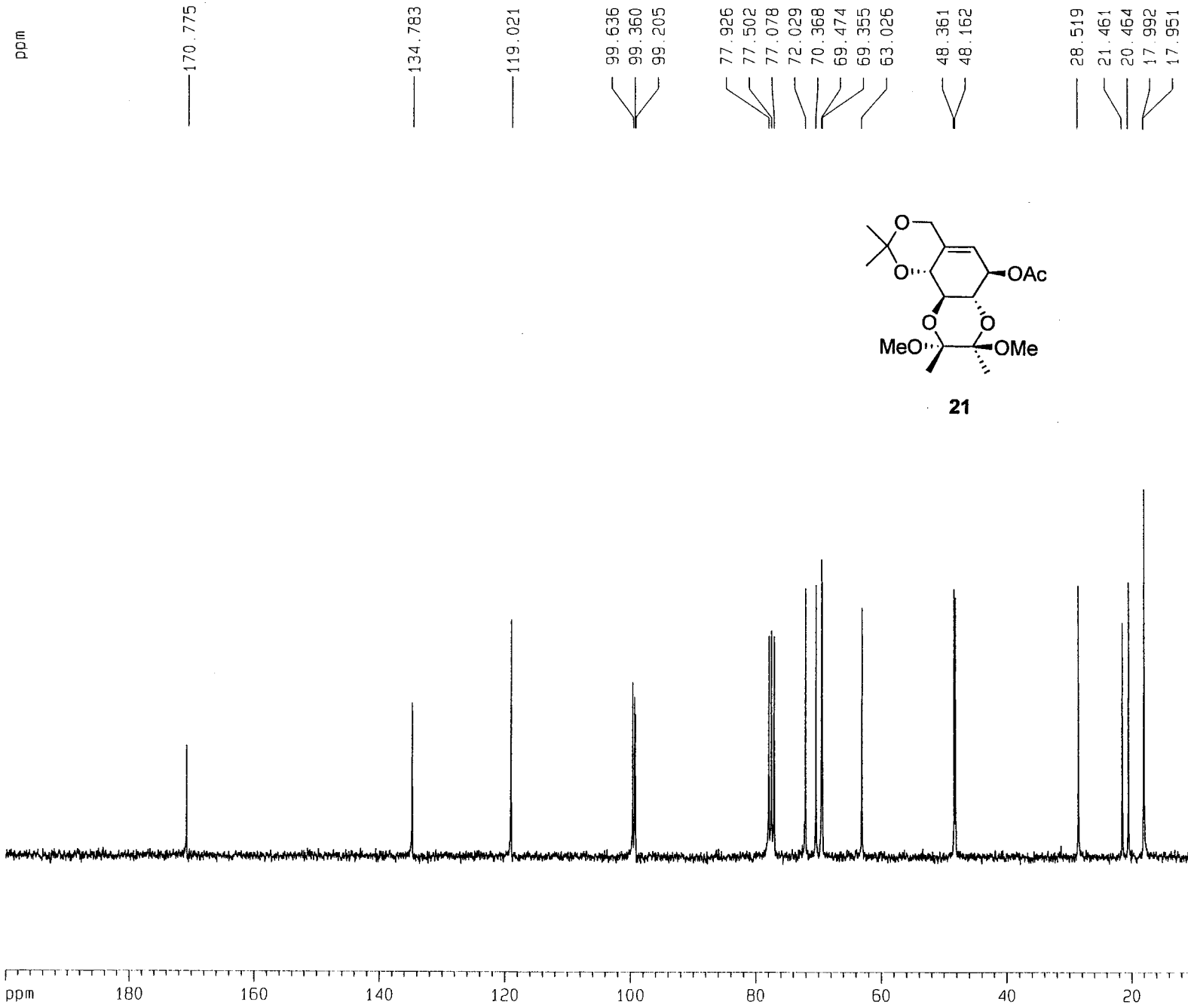
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300052 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	9.77 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	GL45betaOAcC13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050623
Time	18.32
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	292
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	8192
DW	18.900 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

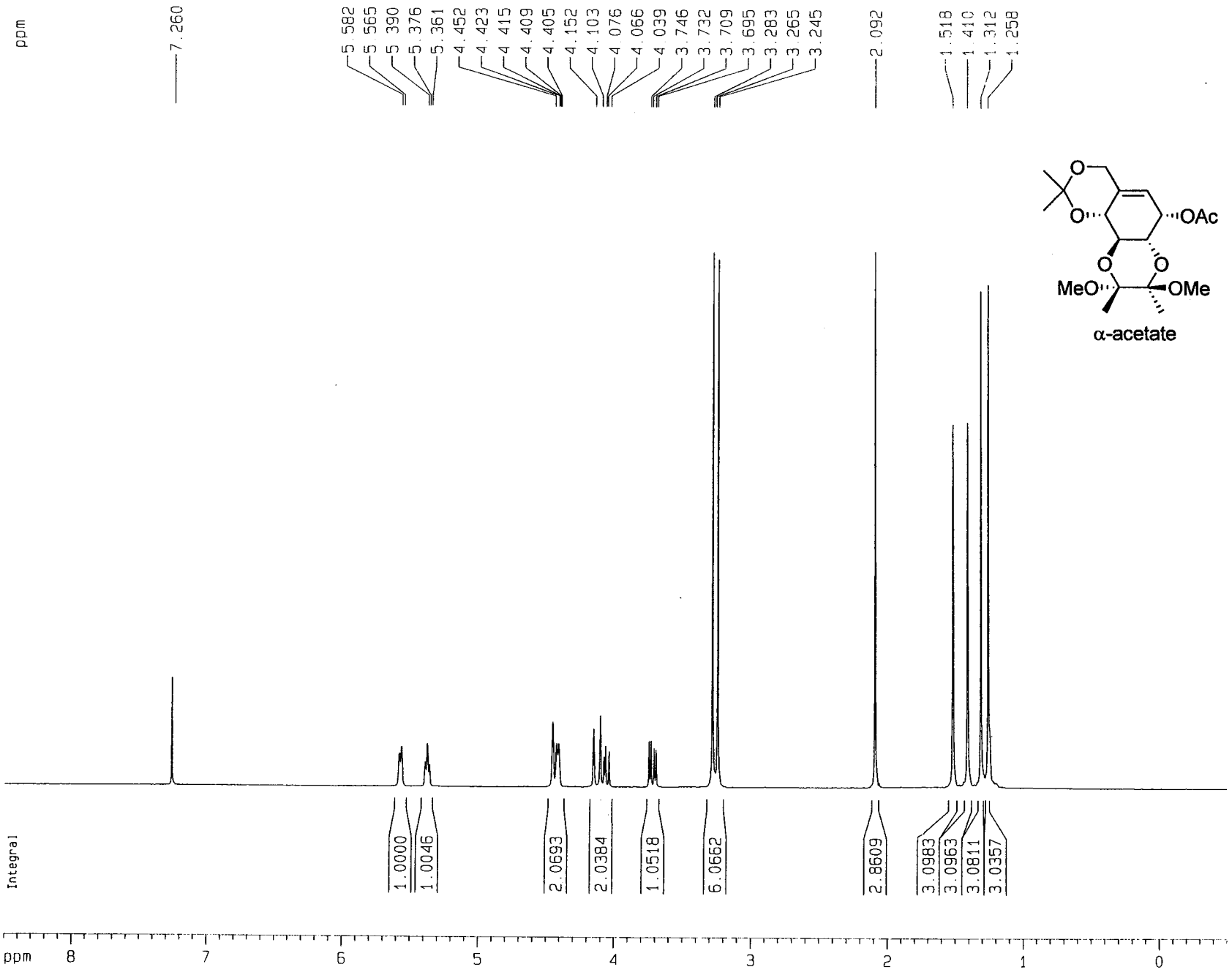
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677165 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	6.45 cm
F1P	200.000 ppm
F1	15093.54 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	9.13043 ppm/cm
HZCM	689.05310 Hz/cm



Current Data Parameters

NAME	GL46data
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050521
Time	14.07
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	181
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

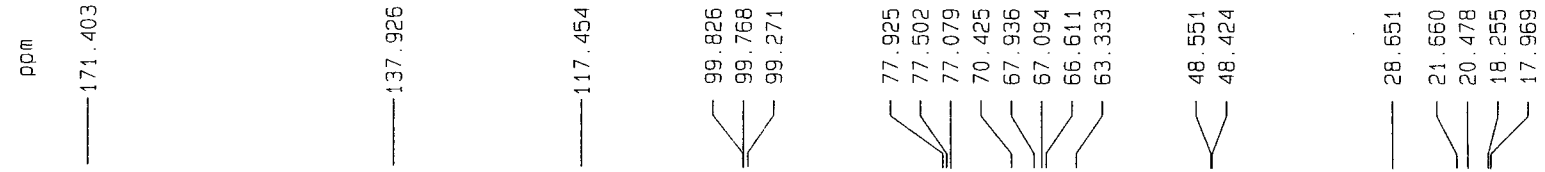
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

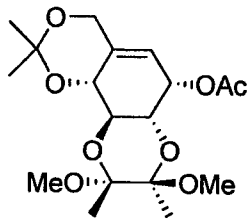
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

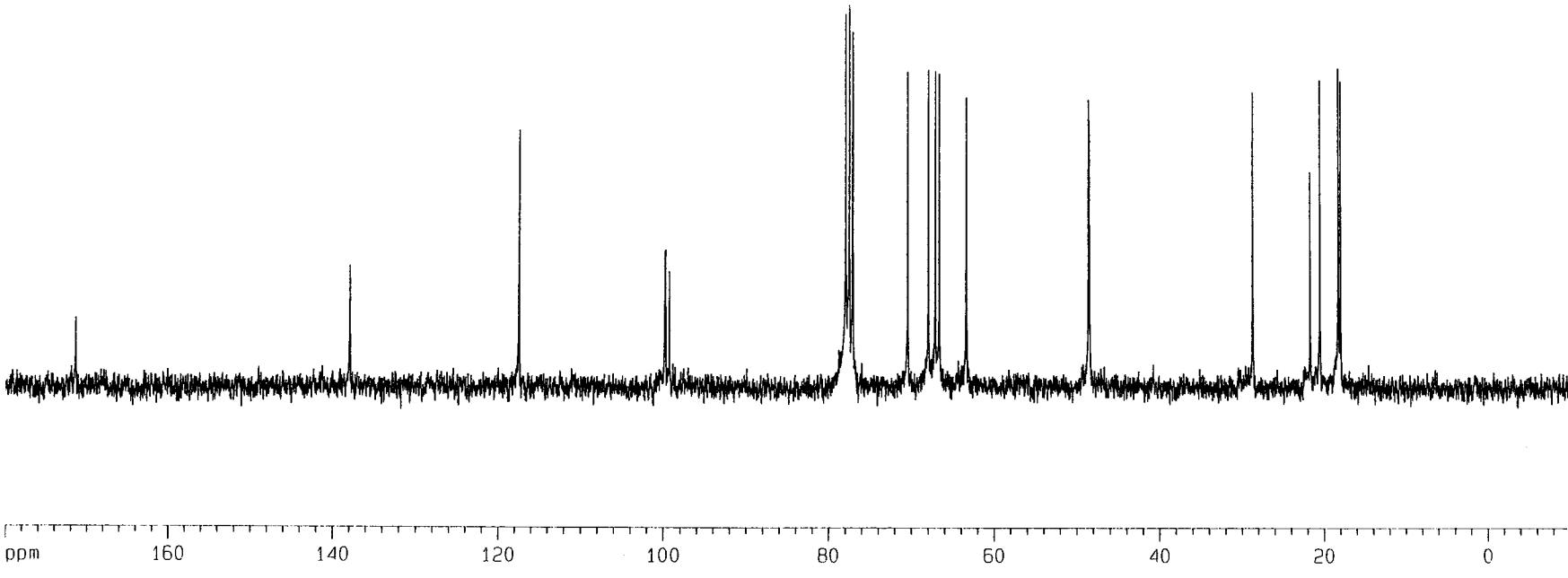
1D NMR plot parameters

CX	22.00 cm
CY	9.91 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm





α-acetate



Current Data Parameters

NAME	GL46alphac13
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20050520
Time	10.17
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	1172
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	11585.2
DW	22.050 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

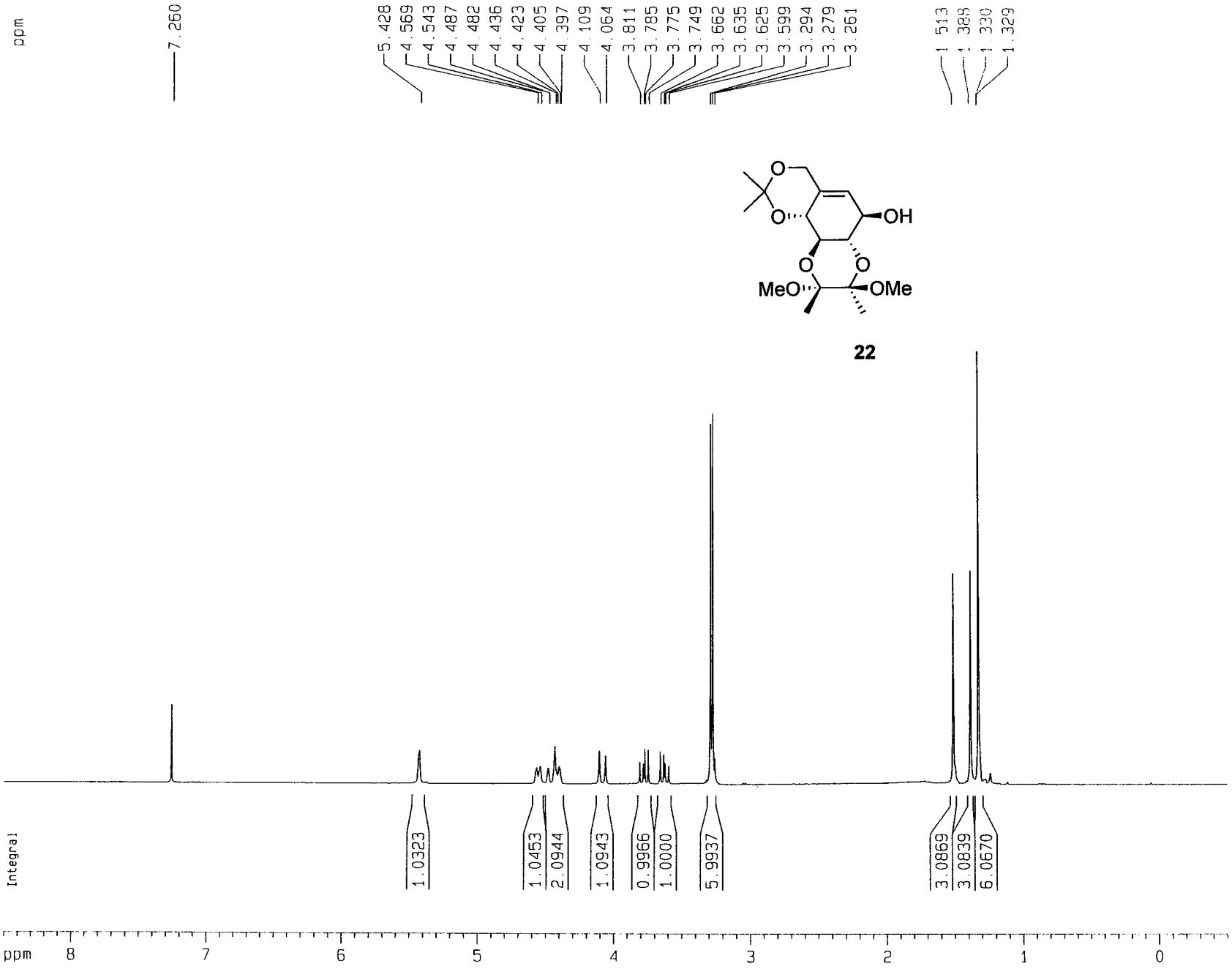
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677136 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	5.67 cm
F1P	180.000 ppm
F1	13584.19 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	8.26087 ppm/cm
HZCM	623.42896 Hz/cm



Current Data Parameters

NAME	GL43beta1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050826
Time	11.54
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	161.3
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

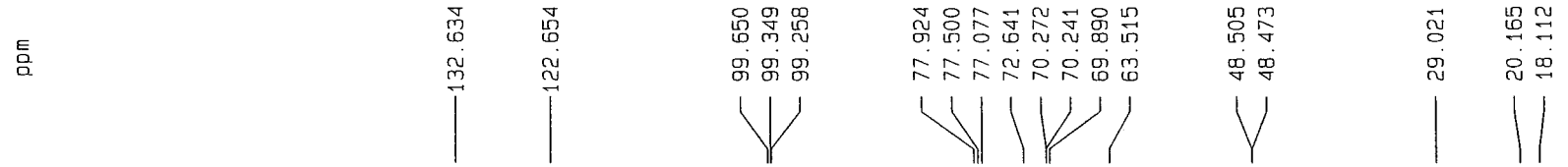
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

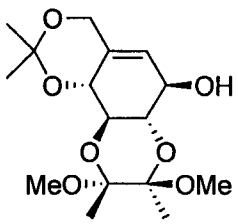
F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	7.85 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm





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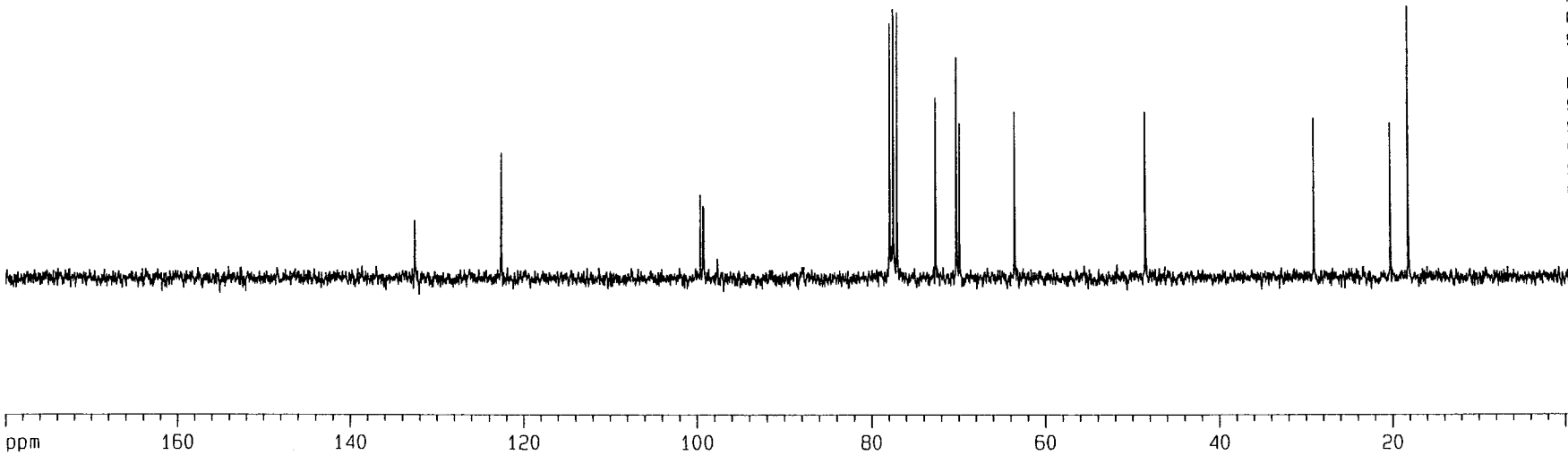
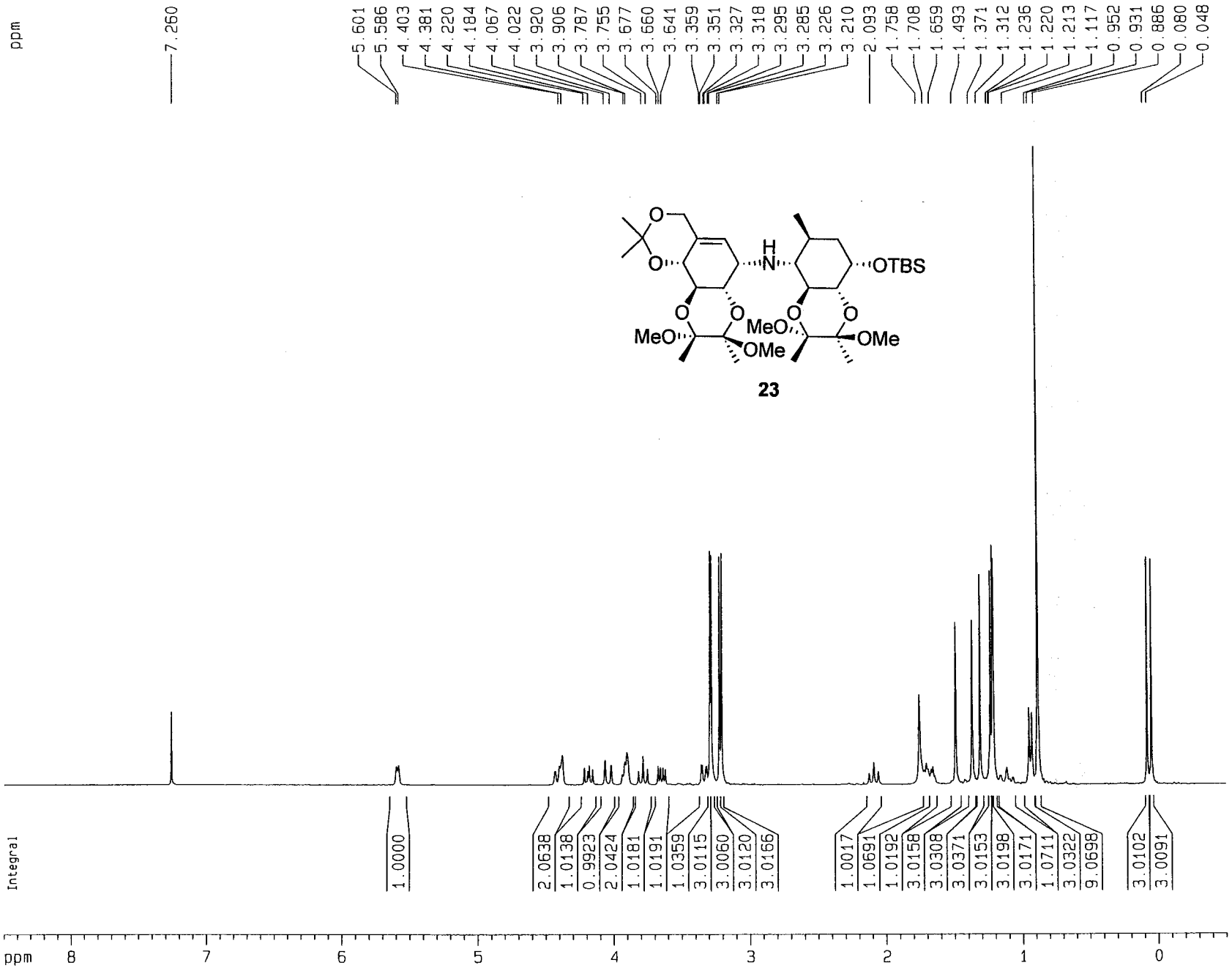



```
Current Data Parameters
NAME                  GL43-c13
EXPNO                        1
PROCNO                       2

F2 - Acquisition Parameters
Date_                 20060807
Time                     18.12
INSTRUM                dpx300
PROBHD   5 mm BBO BB-1H
PULPROG                  zgdc
TD                      65536
SOLVENT                 CDCl3
NS                        139
DS                          0
SWH                 26455.027 Hz
FIDRES               0.403672 Hz
AQ                   1.2386804 sec
RG                       8192
DW                     18.900 usec
DE                      6.00 usec
TE                       0.0 K
D1                1.00000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      3.00 usec
PL1                     -6.00 dB
SFO1               75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                   120.00 dB
PL12                   19.00 dB
SFO2              300.1315007 MHz

F2 - Processing parameters
SI                        65536
SF                 75.4677141 MHz
WDW                          EM
SSB                           0
LB                       2.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       23.00 cm
CY                        4.01 cm
F1P                    180.000 ppm
F1                   13584.19 Hz
F2P                     -0.500 ppm
F2                     -37.73 Hz
PPMCM                 7.84783 ppm/cm
HZCM                592.25751 Hz/cm
```



Current Data Parameters

NAME	GL57dataok
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050916
Time	13.20
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	8992.806 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	128
DW	55.600 usec
DE	79.43 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

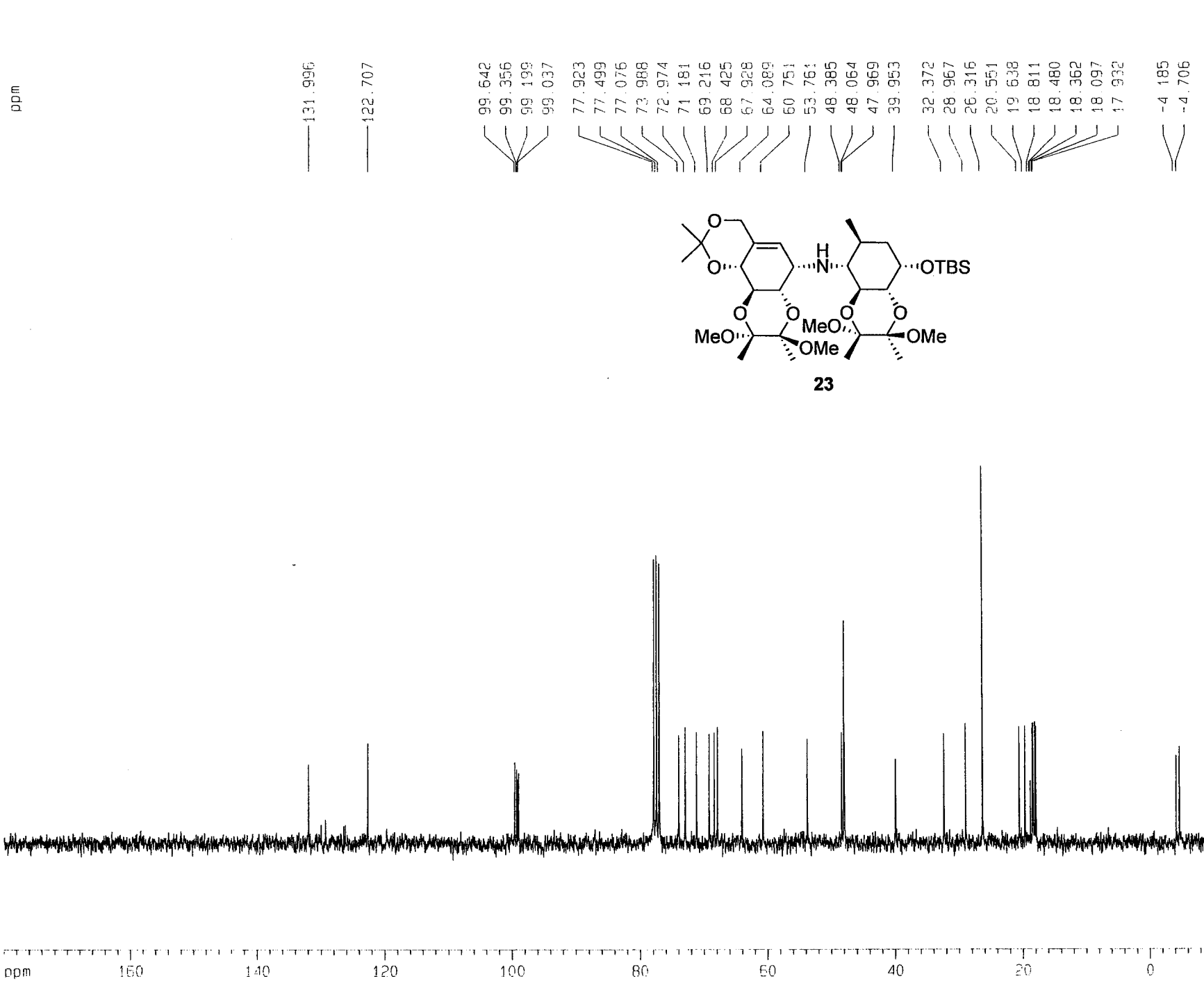
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SF01	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.74 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



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Current Data Parameters

NAME	GL57c13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050714
Time	20.42
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	71
DS	0
SWH	26455.027 Hz
FIDRES	0.403672 Hz
AQ	1.2386804 sec
RG	8192
DW	18.900 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

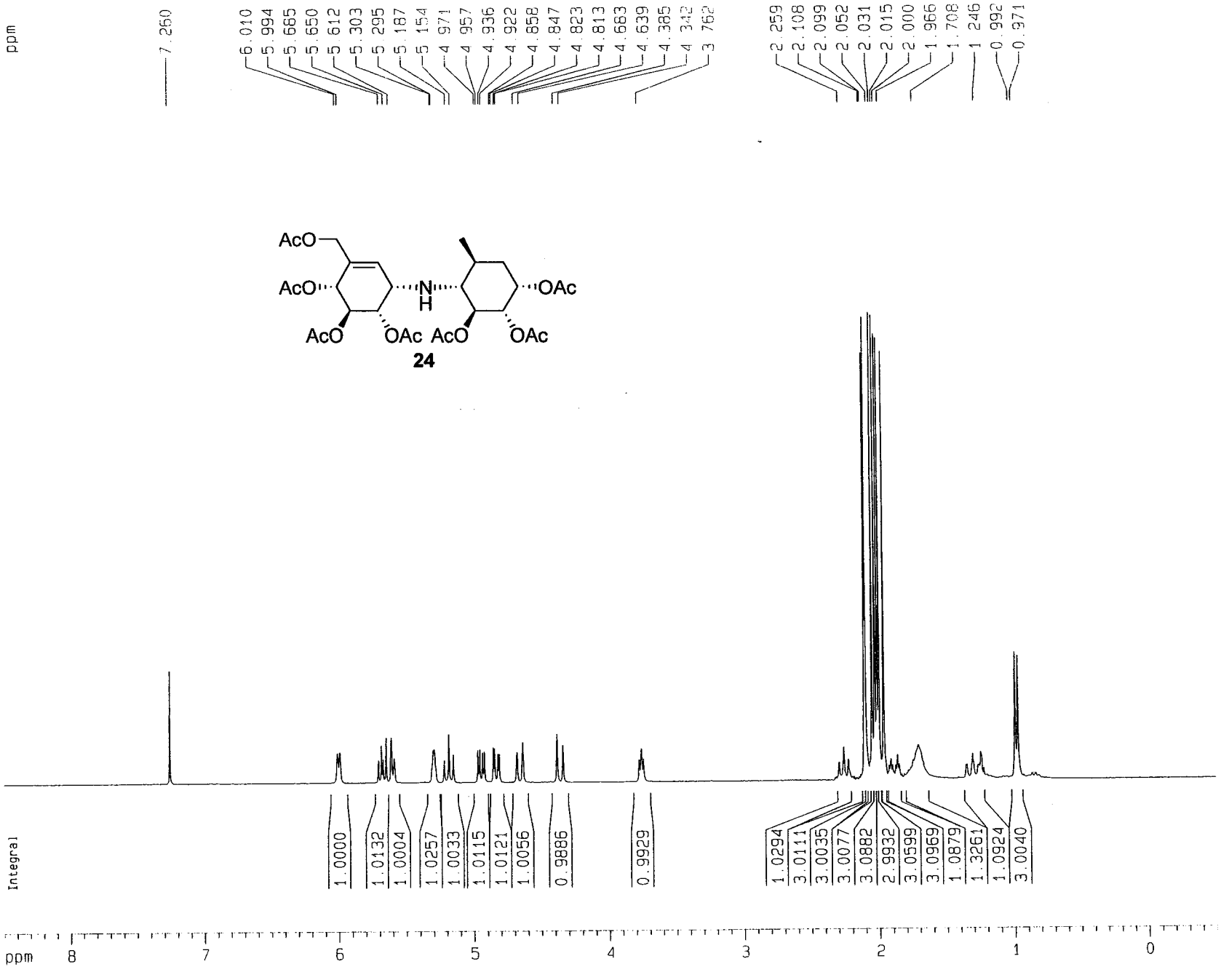
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

F2 - Processing parameters

SI	65536
SF	75.4677145 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	7.16 cm
F1P	180.000 ppm
F1	13584.19 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	8.26087 ppm/cm
HZCM	623.42896 Hz/cm



Current Data Parameters

NAME	GL59-h1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20070214
Time	15.39
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	4803.074 Hz
FIDRES	0.146578 Hz
AQ	3.4111989 sec
RG	161.3
DW	104.100 usec
DE	148.71 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

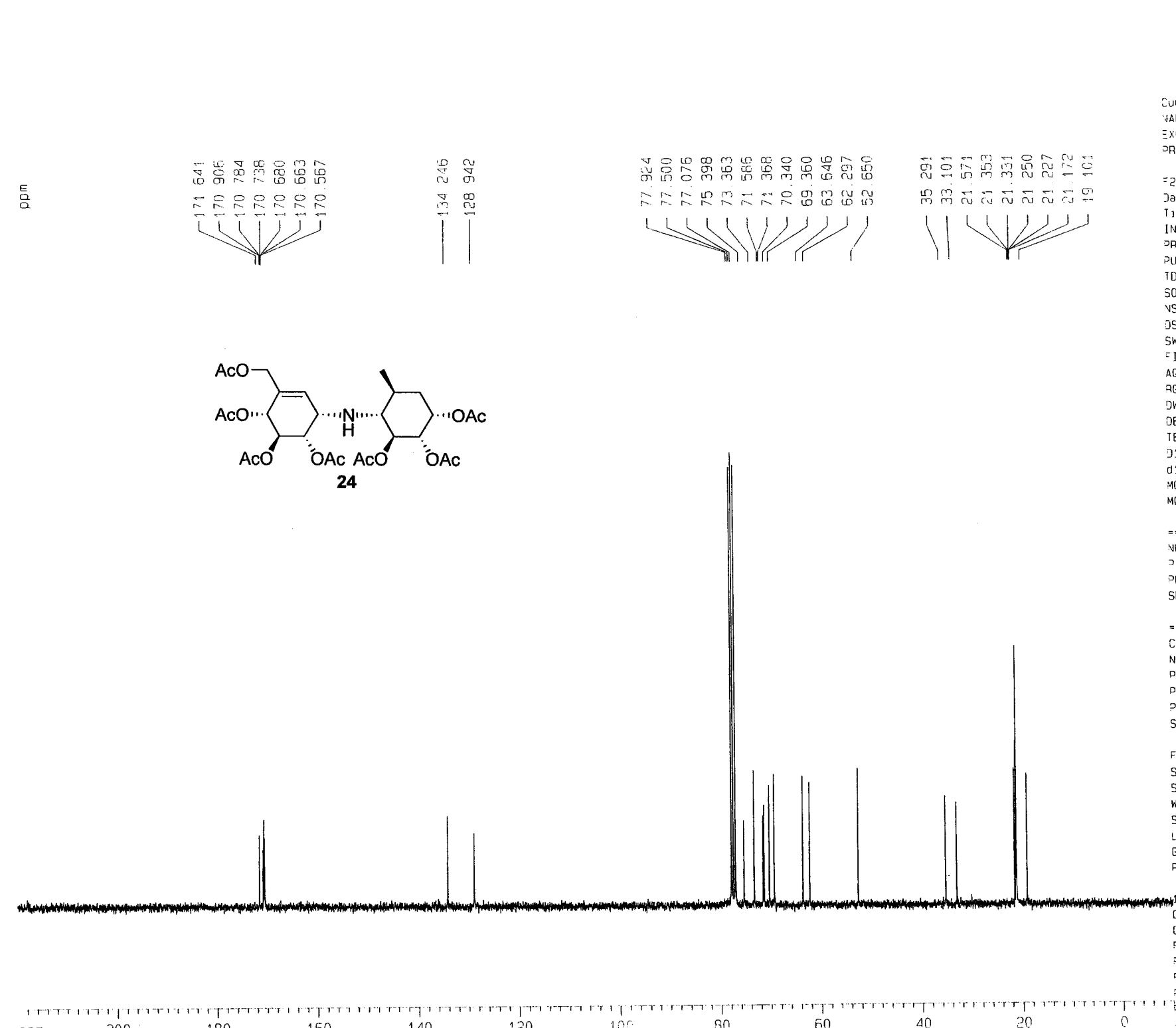
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	8.45 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm



Current Data Parameters

NAME	GL59-c13
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

Date_	20070214
Time	15.46
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	MeOD
NS	1409
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	1290.2
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

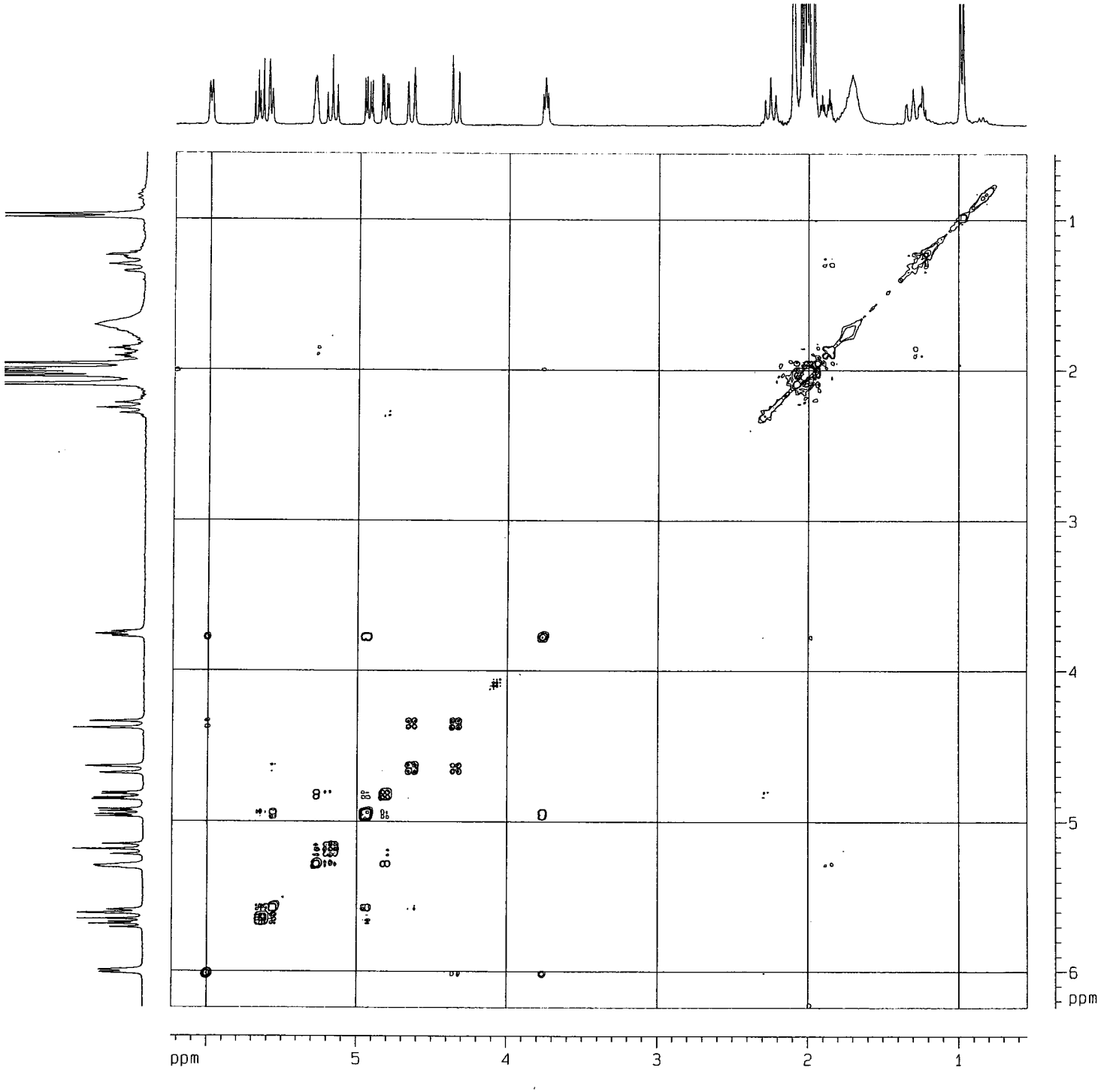
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

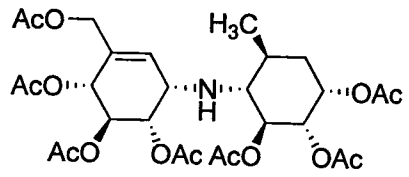
F2 - Processing parameters

SI	65536
SF	75.4677133 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	8.96 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67712 Hz/cm





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Current Data Parameters

NAME	GL59noesy
EXPNO	61
PROCNO	1

F2 - Acquisition Parameters

Date_	20070215
Time	21.29
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	C6D6
NS	32
DS	16
SWH	1707.650 Hz
FIDRES	1.667627 Hz
AQ	0.2998772 sec
RG	181
DW	292.800 usec
DE	6.00 usec
TE	0.0 K
d0	0.00028106 sec
D1	2.00000000 sec
D8	1.10000002 sec
IN0	0.00058580 sec
MCREST	0.00000000 sec
MCWRK	1.00000000 sec
ST1CNT	0

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

NUC1	1H
P1	9.30 usec
PL1	-2.00 dB
SFO1	300.1310327 MHz

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.131 MHz
FIDRES	6.668231 Hz
SW	5.688 ppm
FnMODE	States-TPPI

F2 - Processing parameters

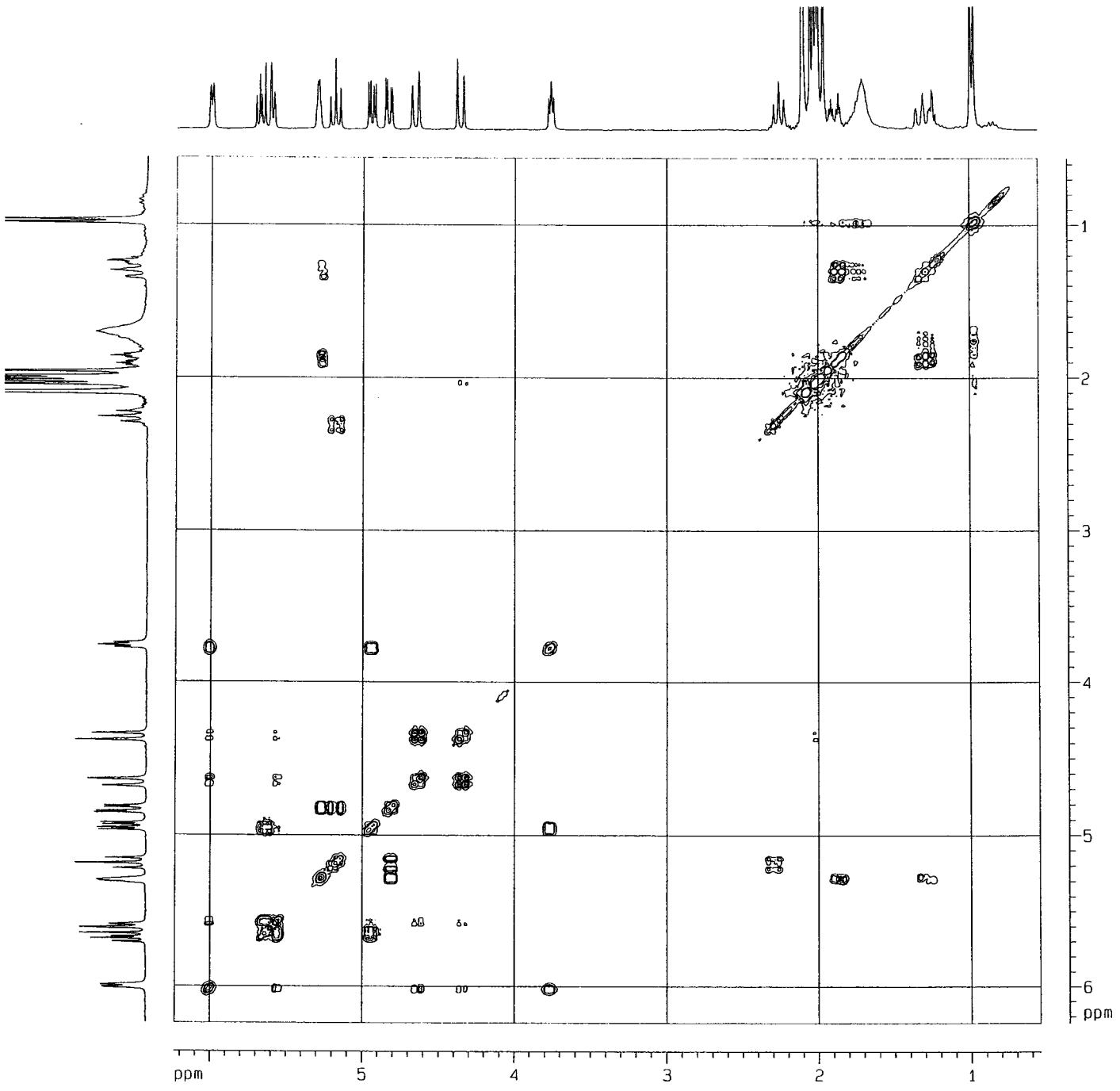
SI	512
SF	300.1300150 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

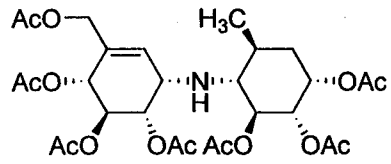
F1 - Processing parameters

SI	512
MC2	States-TPPI
SF	300.1300118 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	6.236 ppm
F2LO	1871.47 Hz
F2PHI	0.546 ppm
F2HI	163.82 Hz
F1PLO	6.245 ppm
F1LO	1874.40 Hz
F1PHI	0.558 ppm
F1HI	167.33 Hz
F2PPMCM	0.37931 ppm/cm
F2HZCM	113.84335 Hz/cm
F1PPMCM	0.37918 ppm/cm
F1HZCM	113.80448 Hz/cm





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Current Data Parameters

NAME	GL59cosy
EXPNO	301
PROCNO	1

F2 - Acquisition Parameters

Date_	20070216
Time	11.40
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	cosyqf45
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	1707.650 Hz
FIDRES	1.667627 Hz
AQ	0.2998772 sec
RG	161.3
DW	292.800 usec
DE	6.00 usec
TE	0.0 K
d0	0.00000300 sec
D1	3.00000000 sec
IN0	0.00058580 sec
MCREST	0.00000000 sec
MCWRK	3.00000000 sec

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

NUC1	1H
P1	9.30 usec
PL1	-2.00 dB
SFO1	300.1310327 MHz

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	300.131 MHz
FIDRES	6.668231 Hz
SW	5.688 ppm
FnMODE	QF

F2 - Processing parameters

SI	512
SF	300.1300150 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	512
MC2	QF
SF	300.1300118 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	6.236 ppm
F2LO	1871.47 Hz
F2PHI	0.546 ppm
F2HI	163.82 Hz
F1PLO	6.245 ppm
F1LO	1874.40 Hz
F1PHI	0.558 ppm
F1HI	167.33 Hz
F2PPMCM	0.37931 ppm/cm
F2HZCM	113.84335 Hz/cm
F1PPMCM	0.37918 ppm/cm
F1HZCM	113.80448 Hz/cm