Supporting Information

Total Synthesis of *N*-Acetylglucosamine-1,6-anhydro-*N*-acetylmuramylpentapeptide and Evaluation of Its Turnover by AmpD from *Escherichia coli*

*Dusan Hesek, Mijoon Lee, Weilie Zhang, Bruce C. Noll and Shahriar Mobashery**

Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN 46556

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Experimental Data for Compounds

General Procedures. All organic reagents were purchased from either Sigma-Aldrich Chemical Company or Acros Organics, unless otherwise stated. All reactions were performed under an atmosphere of nitrogen unless noted otherwise. Reactions were monitored by thin-layer chromatography (TLC) carried out on Whatman reagents 0.25 mm silica gel 60-F plates that were visualized using UV light and/or aqueous cerium sulfate staining, followed by heating. Flash chromatography was carried out with silica gel 60, 230-400 mesh (0.040-0.063 mm particle size) purchased from EM Science. NMR spectra, including ^{1}H, ^{13}C, DEPT, H-H COSY, and H-C HETCOR experiments, were recorded on a Varian UnityPlus 300, or a Varian INOVA-500, or Varian DirectDrive 600 spectrometer. Proton and Carbon chemical shifts were referenced to residual solvent peaks. NMR signal assignments for synthesized compounds were performed on the basis of H-H COSY, H-C HETCOR, and DEPT experiments. High-resolution mass spectra were obtained at the Department of Chemistry and Biochemistry, University of Notre Dame via FAB ionization, using a JEOL AX505HA mass spectrometer.

Analytical high performance liquid chromatography (HPLC) was performed on Waters 2414 instrument with SunFire C18 reversed-phased column (Waters) or delta-pak C18 reversed-phased column (Waters) using a linear gradient of 2-15% acetonitrile in water supplemented with 0.1% TFA over 40 min at 1 mL/min. Detection of the samples was by UV at 205 nm. Preparative HPLC purifications were performed using delta-pak C18 reversed-phased column, 100 Å pore size, 19 × 300 mm.

Crystals were examined under Infineum V8512 oil and placed on a MiTeGen mount, then transferred to the 100 K N_2 stream (or 296 K) of either a Bruker SMART Apex CCD diffractometer or Bruker X8-Apex II CCD diffractometer. Unit cell parameters were determined from reflections with $I > 10\sigma(I)$ harvested from three orthogonal sets of 30 0.5° ω scans. Data collection strategy was calculated using COSMO, included in the Apex2 suite of programs[1] to maximize coverage of reciprocal space in a minimum amount of time. Average 4-fold redundancy of measurements was sought. Data were corrected for Lorentz and polarization effects, as well as for absorption. Structure solution and refinement utilized the programs of the SHELXTL software package.[2] Full details of the X-ray structure determinations are in the CIF files included as Supporting Information.

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[1] Apex2. Bruker-AXS: Madison, WI, 2008; Vol. 58.
[2] Sheldrick, G. M., *Acta Crystallogr. A.* **2008,** *64*, 112-122.

Kinetic studies. The assays were carried out in 20 mM sodium phosphate buffer, pH 7.0, at 25 °C with substrate concentrations ranging from 50 μM to 3.0 mM and 1.5 μM of AmpD. The reaction mixtures were incubated at 25 °C for 30 min. The reactions were stopped by the addition of 2 volume of 0.075% TFA in water. Reaction products were separated and quantified on a C18 reversed-phase HPLC column (Symmetry Shield RP18, 5 μm, 3.9 mm by 150 mm; Waters) on a PerkinElmer series 200 System. The column was equilibrated with 0.05% trifluoroacetic acid in water and eluted with a linear acetonitrile gradient from 0 to 15% over 40 min with a flow rate of 1 mL/min. The column effluent was monitored at 205 nm. The catalytic activity of the AmpD was quantified from the rate of substrate disappearance and of pentapeptide appearance.

4-*O*-Benzyl-3,6-di-*O*-*tert*-butyldimethylsilyl-D-(–)-glucal (4b). The procedure was adapted from that reported by Bartolozzi *et al*.[3] A solution of **3** (37 g, 0.10 mol) in anhydrous DMF (400 mL), was treated with NaH (8.0 g, 0.20 mol, 60% in oil), in three portions under vigorous stirring. The resulting mixture was stirred at 35-40 ºC for 30 min, followed by dropwise addition of a solution of benzyl bromide (17 mL, 0.14 mol) in DMF (10 mL), at the end of which the solution was stirred for 24 h. A total of 200 mL of hexanes were added and the biphasic mixture was vigorously stirred for 15 min and then the layers were allowed to separate. The upper layer was dried over $MgSO_4$, filtered, and concentrated in vacuo, and the residue was purified by column chromatography on silica gel (hexanes/Et_2O, 5:1), to afford the main fraction of **4b** (9.3 g) as a colorless oil in 20% yield. The lower phase was evaporated to dryness. The residue was carefully diluted with water, neutralized with AcOH (10%), and extracted with EtOAc (2 × 250 mL). The combined organic solution was washed with water, dried over $MgSO_4$, filtered, and was evaporated to dyness under reduced pressure. The residue was chromatographed on silica gel using a gradient elution of 20% – 95% Et_2O in hexanes to provide the main compound as **4a** in 65% (23 g). When this reaction was performed in anhydrous THF instead of DMF, compound **4b** was obtained as the major product in 89%. **Compound 4b:** ^{1}H NMR (500 MHz, $CDCl_3$), δ 0.07, 0.09, 0.10 and 0.11 (4×s, 12H, $SiCH_3$), 0.91 and 0.93 (2×s, 18H, Si-C$(CH_3)_3$), 3.66 (dd, *J* = 8.1, 6.1 Hz, 1H, H-4), 3.85 (dd, *J* = 11.5, 2.3 Hz, 1H, H-6a), 3.90 (m, 1H, H-5), 3.97 (dd, *J* = 11.4, 4.6 Hz, 1H, H-6b), 4.35 (m, 1H, H-3), 4.65 (dd, *J* = 6.2, 2.6 Hz, 1H, H-2), 4.74 and 4.83 (AB, *J* = 11.2 Hz, 2H, OC*H*$_2$Ph), 6.32 (dd, *J* = 6.2, 1.2 Hz, 1H, H-1), 7.28 - 7.39 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$), ẟ-5.2 (q, $SiCH_3$), -5.0 (q, $SiCH_3$), -4.5 (q, $SiCH_3$), -4.3 (q, $SiCH_3$), 18.0 and 18.5 (2×s, Si-*C*$(CH_3)_3$), 25.9 and 26.1 (2×q, Si-C(*C*H$_3$)$_3$), 62.1 (t, C-6), 69.1 (d, C-3), 74.0 (t, O*C*H$_2$Ph), 76.6 (d, C-4), 78.2 (d, C-5), 103.3 (d, C-2), 127.8, 127.9, 128.5, 138.5, 143.6 (d, C-1); HRMS (FAB), calcd for

[3] Bartolozzi, A.; Pacciani, S.; Benvenuti, C.; Cacciarini, M.; Liguori, F.; Menichetti, S.; Nativi, C. *J. Org. Chem.* **2003,** *68*, 8529-8533.

$C_{25}H_{43}O_4Si_2$ ($M–H^+$), 463.2700, found 463.2702. **4-*O*-benzyl-6-*O*-*tert*-butyldimethylsilyl-D-(–)-glucal (4a):** ^1H NMR (600 MHz, $CDCl_3$), δ 0.09 (s, 6H, $SiCH_3$), 0.91 (s, 9H, Si-C(CH_3)$_3$), 2.41 (d, J = 5.6 Hz, 1H, $C_{(3)}$-OH), 3.67 (dd, J = 8.3, 6.3 Hz, 1H, H-4), 3.85 (dt, J = 8.3, 2.6 Hz, 1H, H-5), 3.95 (br. s, 2H, H-6), 4.28 (br. s, 1H, H-3), 4.70 (dd, J = 6.1, 2.2 Hz, 1H, H-2), 4.78 and 4.80 (AB, J = 11.7 Hz, 2H, OCH_2Ph), 6.34 (d, J = 6.1 Hz, 1H, H-1), 7.26 - 7.42 (m, 5H). ^{13}C NMR (151 MHz, $CDCl_3$), δ -5.5 (q, $SiCH_3$), -5.2 (q, $SiCH_3$), 25.8 (q, $SiCH_3$), 62.3 (t, C-6), 68.0 (d, C-3), 73.6 (t, OCH_2Ph), 77.0 (d, C-4), 77.5 (d, C-5), 102.2 (d, C-2), 127.8, 127.9, 128.5, 138.4, 144.4 (d, C-1); HRMS (FAB), calcd for $C_{19}H_{29}O_4Si$ ($M–H^+$), 349.1835, found 349.1847.

4-*O*-Benzyl-D-(–)-glucal (5). A solution of compound **4b** (16 g, 34 mmol) in anhydrous THF (140 mL) was treated with dropwise addition of tetrabutylammonium fluoride (70 mL, 1.0 M in THF) in an ice-water bath. The stirring was continued at room temperature until the starting material was consumed (4 h). The mixture was cooled again in the ice-water bath and was treated with acetic acid (5 mL) and the solvent was removed in vacuo and the residue was taken up in CH_2Cl_2 (50 mL), washed with aq. $NaHCO_3$, and the organic layer was dried over $MgSO_4$, filtered, and concentrated. The crude product was crystallized from hexanes/EtOAc (5/1). The crude mother liquids from crystallization were further purified by column chromatography on silica gel using Et_2O to give the desired product (7.2 g, 90%, combined from recrystallization and column chromatography) as a white solid. ^1H NMR (500 MHz, CD_3CN) δ 3.68 (dd, J = 9.0, 6.4 Hz, 1H, H-4), 3.85-4.00 (m, 4H, H-5, H-6, OH), 4.36 (d, J = 6.6 Hz, 1H, H-3), 4.71 (dd, J = 6.2, 2.2 Hz, 1H, H-2), 4.80 and 4.95 (AB, J = 11.5 Hz, 2H, OCH_2Ph), 6.33 (d, J = 5.8 Hz, 1H, H-1), 7.32 (m, 5H); ^{13}C NMR (126 MHz, acetone-d_6), δ 61.7 (t, C-6), 69.5 (d, C-3), 74.0 (t, OCH_2Ph), 77.7 (d, C-4), 78.9 (d, C-5), 104.9 (d, C-2), 128.0, 128.4, 128.9, 139.9, 144.1 (d, C-1).

1,6-Anhydro-4-*O*-benzyl-2-iodo-2-deoxy-β-D-glucopyranose (6). We used a variation of the procedure of Tailler *et al*[4] for the transformation of compound **5** to **6**. 4-Benzyl-D-glucal (**5**, 2.4 g, 10 mmol) in acetonitrile (30 mL) was mixed with 4 Å molecular sieves (1 g) and $(Bu_3Sn)_2O$ (4.8 g, 8.0 mmol) and the resulting mixture was refluxed for 2 h. After cooling to room temperature, iodine (3.0 g, 12 mmol) was added in several portions, followed by the addition of propylene oxide (0.6 mL), and the mixture was stirred for 20 h at room temperature. Workup was carried out by filtration and evaporation of the volatile compounds *in vacuo*. The residue was then taken up into EtOAc and the organic phase was treated with aqueous sodium thiosulfate. The organic layer was separated, dried over $MgSO_4$, filtered, and the solvent was evaporated to dryness. The crude product was then purified by column chromatography on silica gel using hexanes/Et_2O, 3/1 to 1/1 to give compound **6** as a white solid (3.3 g,

[4] Tailler, D.; Jacquinet, J. C.; Noirot, A. M.; Beau, J. M. *J. Chem. Soc., Perkin Trans. 1* **1992**, 3163-3164.

91%). Crystals were grown from mixed solvents of hexanes and Et_2O and used for determination of X-ray crystal structure. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.77 (d, *J* = 6.6 Hz, 1H, OH), 3.43 (t, 1H, H-4), 3.69 (dd, *J* = 7.5, 5.5 Hz, 1H, H-6), 3.93 (m, 1H, H-2), 4.07 (d, *J* = 7.4 Hz, 1H, H-6), 4.26 (m, 1H, H-3), 4.63 and 4.75 (AB, *J* = 12.0 Hz, 2H, OC*H*$_2$Ph), 4.63 (m, 1H, H-5), 5.75 (s, 1H, H-1), 7.40 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 26.4 (d, C-2), 66.3 (t, C-6), 71.5 (t, O*C*H$_2$Ph), 72.6 (d, C-3), 75.1 (d, C-5), 78.6 (d, C-4), 103.5 (d, C-1), 127.8, 127.9, 128.5, 137.4; HRMS (FAB), calcd for $C_{13}H_{16}IO_4$ ($M+H^+$), 363.0093, found 363.0074.

4-*O*-Benzyl-1,6:2,3-dianhydro-β-D-mannopyranose (7). A mixture of compound **6**[4] (3.6 g, 10 mmol) and K_2CO_3 (1.5 g, 11 mmol) in acetonitrile (150 mL) was stirred for 2 h at 60 ºC. The mixture was filtered through a layer of Celite and the residue was washed with acetonitrile. The combined filtrates were evaporated to yield compound **7** (2.0 g, 85%), which was pure by NMR, thus used directly in the next step. Use of Ag_2CO_3 gave reaction outcome to produce compound **7** in 84%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.19 (dt, *J* = 3.8, 0.8 Hz, 1H, H-3), 3.45 (t, *J* = 3.5 Hz, 1H, H-2), 3.65-3.72 (m, 3H, H-5, H-6), 4.51 (dd, *J* = 5.8, 0.8 Hz, 1H, H-4), 4.73 (s, 2H, OC*H*$_2$Ph), 5.71 (d, *J* = 3.2 Hz, 1H, H-1), 7.20-7.39 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 47.7 (d, C-3), 54.3 (d, C-2), 65.7 (t, C-6), 71.5 (d, C-4), 72.1 (t, O*C*H$_2$Ph), 73.6 (d, C-5), 97.5 (d, C-1), 127.8, 128.1, 128.6, 137.2; HRMS (FAB), calcd for $C_{13}H_{13}O_4$ (M^+), 233.0814, found 233.0797.

1,6:2,3-Dianhydro-β-D-mannopyranose (10). A mixture of compound **9** (2.7 g, 10 mmol) and Ag_2CO_3 (2.4 g) in acetonitrile (150 mL) was stirred for 2 h at 60 ºC. The mixture was filtered through a layer of Celite and the residue was washed with acetonitrile. The combined filtrates were concentrated and the residue was purified by column chromatography (hexanes/Et_2O, 1/5, to Et_2O) to yield compound **10** (1.3 g, 92%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.84 (m, 1H, $C_{(4)}$-OH), 3.12 (m, 1H, H-3), 3.43 (t, *J* = 3.5 Hz, 1H, H-2), 3.73 (m, 2H, H-6), 3.90 (s, 1H, H-4), 4.41 (m, 1H, H-5), 5.68 (d, *J* = 3.0 Hz, 1H, H-1); ^{13}C NMR (126 MHz, $CDCl_3$) δ 49.2 (d, C-3), 54.1 (d, C-2), 65.5 (t, C-6), 66.9 (d, C-4), 74.1 (d, C-5), 97.6 (d, C-1); HRMS (FAB), calcd for $C_6H_9O_4$ ($M+H^+$), 145.0501, found 145.0481.

4-*O*-Benzyl-1,6:3,4-dianhydro-β-D-altropyranose (11). A solution of **10** (4.3 g, 30 mmol) in anhydrous THF (50 mL) was treated with NaH (1.2 g, 30 mmol, 60% in oil), which was added in two portions under vigorous stirring. The resulting mixture was stirred at room temperature for 30 min, followed by dropwise addition of benzyl bromide (3.6 mL, 30 mmol). After stirring at room temperature for 1 h, the volume of the reaction was reduced and a portion of hexanes was added to the solution. The layers were separated and the bottom layer was concentrated in vacuo to dryness. The residue was purified by column chromatography on silica gel (hexanes/Et_2O, 1/1 to Et_2O), to afford the desired

product as a colorless oil, which crystallized on standing (6.0 g, 85%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.05 (dd, *J* = 4.0, 2.4 Hz, 1H, H-3), 3.10 (d, *J* = 4.0 Hz, 1H, H-4), 3.62 (d, *J* = 2.8 Hz, 1H, H-2), 3.85 (dd, *J* = 7.4, 4.4 Hz, 1H, H-6a), 4.10 (d, *J* = 7.6 Hz, 1H, H-6b), 4.65 and 4.73 (AB, d, *J* = 12.2 Hz, 2H, OC*H*$_2$Ph), 4.67 (m, 1H, H-5), 5.31 (t, *J* = 2.6 Hz, 1H, H-1), 7.29 - 7.38 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 49.5 (d, C-3), 50.0 (d, C-4), 67.2 (t, C-6), 69.8 (d, C-5), 71.9 (t, O*C*H$_2$Ph), 72.0 (d, C-2), 98.0 (d, C-1), 127.9, 128.0, 128.4, 137.0; HRMS (FAB), calcd for $C_{13}H_{13}O_4$ (M^+), 233.0814, found 233.0818.

1,6-Anhydro-2-azido-4-*O*-benzyl-2-deoxy-β-D-glucopyranose (8). $TMSN_3$ (4.0 mL, 33 mmol) and $BF_3{\cdot}Et_2O$ (5.7 mL, 45 mmol) were added to a stirred solution of epoxide **7** (2.3 g, 10 mmol) in anhydrous CH_2Cl_2 (80 mL). The resultant solution was stirred 20 h at room temperature, then was heated under reflux for 1.5 h. The mixture was poured into saturated K_2CO_3 in ice-water bath. Layers were separated and the aqueous phase was extracted with CH_2Cl_2. The combined organic layer was dried over Na_2SO_4, filtered and the solvent was evaporated. Column chromatography on silica gel eluting with Et_2O afforded compound **8** (1.9 g, 70%) as a yellow oil, which solidified on standing. The product was recrystallized from Et_2O:hexanes. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.47 (br. s, 1H, $C_{(3)}$-OH), 3.24 (d, *J* = 3.2 Hz, 1H, H-2), 3.39 (m, 1H, H-4), 3.70 (dd, *J* = 7.1, 5.7 Hz, 1H, H-6a), 3.90 (t, *J* = 3.2 Hz, 1H, H-3), 3.95 (d, *J* = 7.6 Hz, 1H, H-6b), 4.62 (d, *J* = 5.4 Hz, 1H, H-5), 4.69 (s, 2H, OC*H*$_2$Ph), 5.48 (s, 1H, H-1), 7.30 - 7.41 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 62.6 (d, C-2), 66.2 (t, C-6), 70.3 (d, C-3), 71.8 (t, O*C*H$_2$Ph), 75.0 (d, C-5), 78.4 (d, C-4), 101.0 (d, C-1), 127.9, 128.1, 128.6, 137.3; HRMS (FAB), calcd for $C_{13}H_{14}N_3O_4$ ($M–H^+$), 276.0984, found 276.1000.

2-Acetamido-1,6-anhydro-4-*O*-benzyl-2-deoxy-β-D-glucopyranose (12). Compound **8** (1.0 g, 3.6 mmol) was dissolved in MeOH (10 mL) and 5% Pd/C (0.1 g) was added, and the reaction mixture was stirred vigorously under a hydrogen atmosphere at room temperature for 1 h. The reaction mixture was filtered through a layer of Celite and was washed with MeOH. The combined filtrate was evaporated to dryness and the residues was dissolved in CH_2Cl_2 (10 mL). Acetic anhydride (7 mL) and pyridine (5 mL) were added to the solution, which was allowed to stir for 20 h at room temperature. The solution was evaporated to dryness and the residue was dissolved in CH_2Cl_2 and was washed with water. The organic layer was dried over $MgSO_4$, filtered, concentrated to dryness and the crude product was subjected to column chromatography to afford the desired compound (1.0 g, 85% from compound **8**). ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.90 (s, 3H), 2.06 (s, 3H), 3.30 (s, 1H, H-4), 3.73 (dd, *J* = 7.2, 6.1 Hz, 1H, H-6a), 3.96 (d, *J* = 7.7 Hz, 1H, H-6b), 4.10 (d, *J* = 9.7 Hz, 1H, H-2), 4.52 (d, *J* = 5.5 Hz, 1H, H-5), 4.68 and 4.75 (AB, *J* = 12.1 Hz, 2H, O*CH*$_2$Ph), 4.81 (s, 1H, H-3), 5.31 (s, 1H, H-1), 6.12 (d, *J* = 9.7 Hz, 1H, NH), 7.29-7.36 (m, 5H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 21.2 (q), 23.3 (q), 48.9 (d, C-2), 65.3 (t, C-6),

69.7 (d, C-3), 71.5 (t, O*C*H$_2$Ph), 74.1 (d, C-4), 74.4 (d, C-5), 100.4 (d, C-1), 128.1, 128.3, 128.7, 137.4, 169.3 (C=O), 169.9 (C=O); HRMS (FAB), calcd for $C_{17}H_{22}NO_6$ (M+H$^+$), 336.1447, found 336.1453.

2-Acetamido-1,6-anhydro-2-deoxy-β-D-glucopyranose (13). Compound **12** (1.0 g, 3.0 mmol) in MeOH (10 mL) was stirred in the presence of 10% Pd/C (0.15 g) at 55 ºC in an atmosphere of hydrogen for 4 h. The reaction mixture was filtered through a layer of Celite and the residue was washed with MeOH. Concentration of the combined filtrate provided the desired product (0.66 g, 90%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.94 (s, 3H), 2.01 (s, 3H), 3.54 (d, *J* = 1.2 Hz, 1H, H-4), 3.70 (dd, *J* = 7.5, 5.9 Hz, 1H, H-6a), 3.92 (d, *J* = 8.6 Hz, 1H, H-2), 4.00 (d, *J* = 7.2 Hz, 1H, H-6b), 4.48 (d, *J* = 5.6 Hz, 1H, H-5), 4.57 (m, 1H, H-3), 5.25 (s, 1H, H-1), 6.87 (d, *J* = 9.6 Hz, 1H, NH); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.1 (q), 22.9 (q), 49.1 (d, C-2), 65.1 (t, C-6), 68.3 (d, C-4), 72.3 (d, C-3), 75.8 (d, C-5), 100.4 (d, C-1), 170.0 (C=O), 170.5 (C=O); HRMS (FAB), calcd for $C_{10}H_{16}NO_6$ (M+H$^+$), 246.0978, found 246.0974.

2-Acetamido-1,6-anhydro-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose (14). To a stirred solution of compound **8** (1.4 g, 5.0 mmol) in anhydrous dioxane (25 mL) was added NaH (1.3 g, 33 mmol, 60% in oil). The mixture was kept for 10 min at 45 ºC, and then the temperature was decreased to room temperature. (*S*)-2-Chloropropionic acid (1.3 g, 12 mmol) was added and the stirring was continued for 2 h at 90 ºC, at which point the organic solvent was evaporated to dryness. A 50 mL portion of water was carefully added to the residue to decompose the excess of sodium hydride. The solution was extracted once with hexanes/EtOAc (1:1) to remove the mineral oil and it was filtered over a layer of charcoal. The solution was acidified in ice-water temperature with 2.5 M hydrochloric acid until pH 3 was reached, and the resulting precipitate was immediately extracted with a few portions of CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, rapidly dried over sodium sulfate, followed by evaporation to dryness. The residue was chromatographed on silica column, using $CHCl_3$/acetone (3/1) to provide 1,6-anhydro-2-azido-4-*O*-benzyl-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose. (1.2 g, 69%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (d, *J* = 6.8 Hz, 3H), 3.32 (s, 1H, H-4), 3.46 (s, 1H, H-2), 3.59 (s, 1H, H-3), 3.72 (t, *J* = 6.5 Hz, 1H, H-6a), 3.85 - 4.02 (m, 2H, H-6b, Lac-α-H), 4.65 and 4.72 (AB, *J* = 12.0 Hz, 2H, OC*H*$_2$Ph), 4.65 (br. s., 1H, H-5), 5.49 (s, 1H, H-1), 7.26 - 7.41 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.4 (q), 60.3 (d, C-2), 65.3 (t, C-6), 71.6 (t, O*CH*$_2$Ph), 74.1 (d, C-5), 74.3 (d, Lac-α-C), 75.4 (d, C-4), 77.1 (d, C-3), 100.4 (d, C-1), 127.9, 128.2, 128.6, 137.0, 177.1 (C=O); HRMS (FAB), calcd for $C_{16}H_{20}N_3O_6$ (M+H$^+$), 350.1352, found 350.1353.

The sample obtained above (1.2 g, 3.4 mmol) was dissolved in MeOH (20 mL) and 5% Pd/C (0.12 g) was added and the mixture was stirred vigorously under an atmosphere of hydrogen at room temperature for 1 h. The mixture was filtered through a layer of Celite and the residue was washed with

MeOH. The combined filtrate was concentrated to dryness and the residue was dissolved in CH_2Cl_2 (10 mL). The mixture was treated with acetic anhydride (10 mL) and was allowed to stir for 20 h at room temperature. Volatiles were removed *in vacuo* and the resultant crude compound was taken up into a mixture of CH_2Cl_2 and water. The solution was stirred for 1 h and layers were separated. The organic layer was dried over $MgSO_4$, filtered, and concentrated to dryness and the residue was subjected to column chromatography to afford 2-acetamido-1,6-anhydro-4-*O*-benzyl-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose (1.1 g, 85%). 2-amido-1,6-anhydro-4-*O*-benzyl-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose: ^{1}H NMR (500 MHz, CD_3OD) δ 1.34 (d, *J* = 7.0 Hz, 3H, Lac-β-CH_3), 3.16 (d, *J* = 6.8 Hz, 1H, H-2), 3.48 (t, *J* = 6.3 Hz, 1H, H-3), 3.55 (d, *J* = 5.6 Hz, 1H, H-4), 3.67 (dd, *J* = 7.4, 5.4 Hz, 1H, H-6a), 3.90 (d, *J* = 7.4 Hz, 1H, H-6b), 4.24 (q, *J* = 6.8 Hz, 1H, Lac-α-H), 4.62 and 4.74 (2d, *J* = 11.8 Hz, 2H, OC*H*$_2$Ph), 4.69 (d, *J* = 5.2 Hz, 1H, H-5), 5.17 (br. s, 2H, NH_2), 5.54 (s, 1H, H-1), 7.30 - 7.46 (m, 5H); ^{13}C NMR (126 MHz, CD_3OD) δ 20.2 (q), 56.9 (d, C-2), 68.4 (t, C-6), 72.8 (t, O*C*H$_2$Ph), 77.2 (2d, C-3 and C-5), 79.0 (d, Lac-α-C), 82.3 (d, C-4), 100.7 (d, C-1), 129.2, 129.5, 129.8, 139.0, 181.5 (C=O); HRMS (FAB), calcd for $C_{16}H_{22}NO_6$ ($M+H^+$), 324.1447, found 324.1457. 2-acetamido-1,6-anhydro-4-*O*-benzyl-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (d, *J* = 6.8 Hz, 3H, Lac-β-CH_3), 1.96 (s, 3H), 3.47 and 3.56 (2s, 2H, H-3 and H-4), 3.56 (s, 1H, H-3), 3.77 (dd, *J* = 7.2, 5.8 Hz, 1H, H-6a), 4.11 (d, *J* = 9.0 Hz, 1H, H-2), 4.16 (d, *J* = 7.6 Hz, 1H, H-6b), 4.25 (q, *J* = 6.9 Hz, 1H, Lac-α-H), 4.61 and 4.69 (AB, *J* = 11.8 Hz, 2H, OC*H*$_2$Ph), 4.65 (d, *J* = 5.4 Hz, 1H, H-5), 5.44 (s, 1H, H-1), 6.39 (d, *J* = 9.0 Hz, 1H, NH), 7.29 - 7.40 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 17.6 (q), 22.8 (q), 48.2 (d, C-2), 65.3 (t, C-6), 71.5 (t, O*C*H$_2$Ph), 74.1 (d, C-5), 74.2 (d, Lac-α-C), 75.7 and 75.9 (2d, C-3 and C-4), 100.6 (d, C-1), 127.7, 128.2, 128.6, 136.9, 170.3 (C=O), 173.7 (C=O); HRMS (FAB), calcd for $C_{18}H_{24}NO_7$ ($M+H^+$), 360.1553, found 360.1555.

The above compound (1.0 g, 2.7 mmol) was dissolved in MeOH (10 mL) and the solution was stirred in the presence of 10% Pd/C (0.15 g) at 55 ºC under an atmosphere of hydrogen for 4 h. The mixture was filtered through a layer of Celite and the residue was washed with MeOH. Concentration of the combined filtrate provided compound **14** (0.69 g, 92%). ^{1}H NMR (500 MHz, CD_3CN) δ 1.37 (d, *J* = 6.8 Hz, 3H, Lac-β-CH_3), 1.93 (s, 3H), 3.37 (s, 1H, H-3), 3.67 (m, 2H, H-4 and H-6a), 3.90 (d, *J* = 8.4 Hz, 1H, H-2), 4.11 (d, *J* = 7.4 Hz, 1H, H-6b), 4.24 (q, *J* = 6.8 Hz, 1H, Lac-α-H), 4.50 (d, *J* = 5.2 Hz, 1H, H-5), 5.31 (s, 1H, H-1), 6.79 (d, *J* = 8.6 Hz, 1H, NH); ^{13}C NMR (126 MHz, CD_3CN) δ 18.8 (q), 23.1 (q), 50.6 (d, C-2), 66.2 (t, C-6), 69.8 (d, C-4), 74.9 (d, Lac-α-C), 77.2 (d, C-5), 79.9 (d, C-3), 101.6 (d, C-1), 171.4 (C=O), 175.1 (C=O); HRMS (FAB), calcd for $C_{18}H_{24}NO_7$ ($M+H^+$), 366.1553, found 366.1547.

3,4,6-tri-*O*-Acetyl-2-deoxy-2-(2,2,2-trichloroethoxycarbonylamino)-β-D-glucopyranosyl-(1→4)-2-acetamido-1,6-anhydro-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose (20). Compounds **16**[5] (1.5 g, 2.4 mmol) and **14** (0.55 g, 2.0 mmol) were dissolved in anhydrous CH_2Cl_2 (15 mL) and acetonitrile (5 mL), 4-Å activated molecular sieves (1 g) were added, and the suspension was stirred under argon for 2 h. The catalyst TfOH (0.3 mL) was added in two portions, and the solution was stirred at room temperature for 1 h. The reaction was quenched by the addition of triethylamine (0.3 mL) and it was filtered through a layer of Celite. The combined filtrate was concentrated to dryness and the residue was purified by flash chromatography (CH_3Cl/acetone/MeOH, 6:2:0.05) to give **20** (0.77 g) in 52% and **23** (0.38 g) in 26%. **Compound 20:** ^{1}H NMR (500 MHz, CD_3CN) δ 1.33 (d, *J* = 6.8 Hz, 3H), 1.91 (s, 3H), 1.94 (s, 3H), 1.98 (s, 3H), 2.01 (s, 3H), 3.08 - 3.18 (m, 2H), 3.23 (s, 1H), 3.55 (d, *J* = 5.0 Hz, 1H), 3.60 (d, *J* = 3.8 Hz, 1H), 3.63 (t, *J* = 6.5 Hz, 1H), 3.78 (dd, *J* = 19.3, 9.0 Hz, 1H), 3.90 (d, *J* = 9.8 Hz, 1H), 3.94 - 4.00 (m, 1H), 4.01 - 4.12 (m, 2H), 4.18 - 4.32 (m, 2H), 4.45 (d, *J* = 5.4 Hz, 1H), 4.59 (d, *J* = 12.2 Hz, 1H), 4.87 (d, *J* = 12.2 Hz, 1H), 5.05 (t, *J* = 9.8 Hz, 1H), 5.21 (s, 1H), 5.37 (t, *J* = 10.1 Hz, 1H), 5.92 (d, *J* = 8.8 Hz, 1H), 6.55 (d, *J* = 9.0 Hz, 1H), 6.71 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 8.5, 18.2, 20.4, 20.5, 22.9, 46.7, 50.2, 54.8, 61.5, 68.1, 68.4, 71.8, 72.3, 74.1, 75.8, 78.5, 92.0, 95.4, 100.5, 154.3, 169.4, 170.2, 170.7, 171.3. **Compound 23:** ^{1}H NMR (500 MHz, CD_3OD) δ 1.37 (d, *J* = 7.0 Hz, 3H), 1.98 (s, 3H), 1.99 (s, 3H), 2.01 (s, 3H), 2.04 (s, 3H), 3.35 (s, 1H), 3.63 (s, 1H), 3.68 (m, *J* = 12.6 Hz, 1H), 3.83 (dd, *J* = 10.5, 8.9 Hz, 1H), 3.92 - 4.00 (m, 2H), 4.08 - 4.15 (m, 2H), 4.28 - 4.33 (m, 1H), 4.36 (q, *J* = 6.9 Hz, 1H), 4.49 (d, *J* = 5.6 Hz, 1H), 4.64 and 4.88 (AB, *J* = 12.2 Hz, 2H), 5.05 (t, *J* = 9.7 Hz, 1H), 5.27 (s, 1H), 5.34 (m, *J* = 19.7 Hz, 1H), 5.85 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (126 MHz, CD_3OD) δ 18.8, 20.7, 20.8, 22.9, 26.4, 52.2, 56.3, 63.0, 66.5, 69.8, 70.4, 73.7, 74.0, 74.9, 75.6, 77.8, 80.3, 93.7, 102.2, 156.5, 171.4, 171.7, 172.4, 172.5, 172.7; HRMS (FAB), calcd for $C_{26}H_{36}Cl_3N_2O_{16}$ ($M+H^+$), 737.1130, found 737.1118.

***tert*-Butyldimethylsilyl 3,4,6-tri-*O*-benzyl-2-dimethylmaleimido-2-deoxy-β-D-glycopyranoside (25).** Two methods were used.

Method A. A solution of compound **24**[6] (4.5 g, 10 mmol) in anhydrous CH_3CN (120 mL), was treated with NaH (1.6 g, 40 mmol, 60% in oil), in three portions under vigorous stirring in an ice-water bath. The resulting mixture was stirred at room temperature for 30 min, followed by dropwise addition of a solution of benzyl bromide (5.0 mL, 41 mmol) in CH_3CN (10 mL), and the resulted mixture was stirred for an additional 1 h at room temperature. After stirring at 35 °C for 24 h, the solution was cooled and

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[6] Aly, M. R. E.; Castro-Palomino, J. C.; Ibrahim, E. S. I.; El-Ashry, E. S. H.; Schmidt, R. R., *Eur. J. Org. Chem.* **1998**, 2305-2316.

the reaction was quenched with Amberlite 120 (H^+). After filtration, the solvent was removed under reduced pressure and the residue was carefully diluted with water, neutralized with AcOH (10%), and extracted with EtOAc. The combined organic solution was washed with water, dried over $MgSO_4$, filtered, and was evaporated to dryness under reduced pressure. The residue was chromatographed on silica gel using a gradient elution of 20% - 95% Et_2O in hexanes to provide the desired compound in 25% (1.6 g). ^{1}H NMR (600 MHz, $CDCl_3$) δ -0.04 and 0.07 (2×s, 6H, $SiCH_3$), 0.76 (s, 9H, Si-C$(CH_3)_3$), 1.34 (s, 6H), 3.60 (m, 1H), 3.73 (m, 3H), 3.91 (dd, *J* = 10.8, 8.2 Hz, 1H), 4.25 (dd, *J* = 10.8, 8.8 Hz, 1H), 4.46 (d, *J* = 12.3 Hz, 1H), 4.59 (m, 1H), 4.66 (m, 2H), 4.84 (t, *J* = 11.9 Hz, 2H), 5.18 (d, *J* = 8.2 Hz, 1H), 7.14-7.31 (m, Ar-H, 15H); ^{13}C NMR (151 MHz, $CDCl_3$) δ -5.6, -4.1, 8.6, 22.7, 25.4, 57.8, 68.9, 73.4, 74.6, 74.9, 75.0, 79.6, 79.6, 93.4, 127.3, 127.5, 127.6, 127.8, 127.9 , 128.0 , 128.2 , 128.3 , 128.4 , 138.1 , 138.6, 172.1.

Method B. This procedure was carried out under scrupulously dry condition. To a solution of **24** (4.5 g, 10 mmol) in anhydrous CH_2Cl_2 (120 mL) and anhydrous n-Hexane (120 mL) were added benzyl 2,2,2-trichloroacetimidate (11 mL, 60 mmol) and activated 4-Å molecular sieves (10 g), and the mixture was stirred at room temperature for 0.5 h. Then, the mixture was cooled in an ice-water bath, and a catalytic amount of trifluoromethanesulfonic acid was added dropwise (2 × 0.8 mL). The reaction was monitored by TLC. When the starting material was consumed completely (approximately 3 h), the reaction was quenched by addition of Et_3N (2 mL) and additional cyclohexane (20 mL) was added. After the mixture was stirred for 0.5 h at room temperature, 2,2,2-trichloroacetamide was filtered and washed with hexanes/EtOAc (4/1). The filtrate was concentrated to dryness, and the residue was chromatographed (hexane to hexanes/Et_2O, 8/1) to give **25** as a viscous colorless oil (3.7 g, 56%).

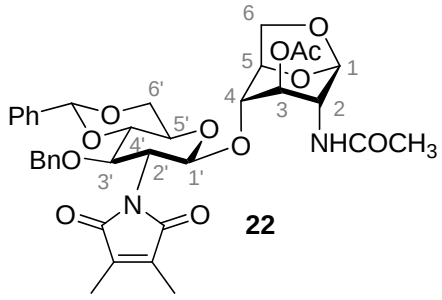
3,4,6-tri-*O*-Benzyl-2-dimethylmaleimido-2-deoxy-β-D-glycopyranosyl-(1→4)-1,6:2,3-dianhydro-β-D-mannopyranose (21). Compound **25** (3.4 g, 5.0 mmol) in dry THF (40 mL) was treated by the dropwise addition of tetrabutylammonium fluoride (6.3 mL, 1.0 M in THF) at room temperature. The mixture was stirred until starting material was consumed (4 h). Subsequently, acetic acid (0.4 mL) was added, and the solvent was removed in vacuo, the residue was taken up in CH_2Cl_2 (50 mL) and washed with satd $NaHCO_3$, and the organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated. The crude product was chromatographed (hexane/Et_2O, 4/1) to give 3,4,6-tri-*O*-benzyl-2-dimethylmaleimido-2-deoxy-β-D-glycopyranose in 77% yield (2.1 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.79 (br. s., 6H), 3.70 (m, 3H), 3.90 (dd, *J* = 10.8, 8.6 Hz, 1H), 4.29 (dd, *J* = 10.8, 8.2 Hz, 1H), 4.40-4.60 (m, 6H), 4.83 (dd, *J* = 11.6, 8.6 Hz, 1H), 5.21 (d, *J* = 8.6 Hz, 1H), 7.12-7.27 (m, 15H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 15.2 , 57.3 , 65.2 , 65.8 , 68.5 , 73.4 , 74.7, 74.8, 76.7, 79.3 , 92.9 , 127.3 , 127.7 , 127.8 , 127.9 , 128.2 , 128.4, 128.9 , 137.7 , 137.8 , 173.2.

Sample obtained above (1.1 g, 2.0 mmol) in anhydrous CH_2Cl_2 (20 mL) was treated with 2,2,2-trichloroacetonitrile (0.5 mL, 25 mmol) and a catalytic amount of DBU. When the starting material was consumed completely (2 h), the reaction mixture was filtered through a small layer of silica gel, which was washed with CH_2Cl_2. The filtrate was concentrated under scrupulously dry conditions, and the residue (compound **17**) was kept under high vacuum for 0.5 h over P_2O_5. Derivative **17** was introduced into the next reaction step without further purification. Glycosylation of compounds **10** and **17** was carried out under the same condition for preparation of compound **20** to give compound **21** in 85%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.79 (br. s., 6H), 3.27 (d, *J* = 3.1 Hz, 1H), 3.35 (m, 1H), 3.57 - 3.66 (m, 3H), 3.71 - 3.81 (m, 3H), 3.86 (s, 1H), 4.01 (dd, *J* = 10.7, 8.5 Hz, 1H), 4.11 - 4.19 (m, 2H), 4.44 (d, *J* = 12.1 Hz, 1H), 4.54 - 4.68 (m, 3H), 4.80 - 4.87 (m, 2H), 5.26 (d, *J* = 8.4 Hz, 1H), 5.59 (d, *J* = 2.9 Hz, 1H), 7.08 - 7.39 (m, 15H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 8.6 (q), 48.2 (d), 54.2 (d), 55.4 (d), 65.4 (t), 68.3 (t), 71.4 (d), 73.3 (d and t), 74.8 (t), 74.9 (t), 75.0 (d), 79.1 (d), 79.6 (d), 97.3 (d), 97.5 (d), 127.3, 127.6, 127.9, 128.0, 128.2, 128.3, 137.7, 138.1, 171.6; MS (FAB) [M–H$^+$] 682.

Introduction of Azide. $TMSN_3$ (0.8 mL, 6.6 mmol) and $BF_3{\cdot}Et_2O$ (1.1 mL, 9.0 mmol) were added to a stirred solution of epoxide **21** (1.37 g, 2.0 mmol) in anhydrous CH_2Cl_2 (15 mL). The resultant solution was stirred 20 h at room temperature, then was heated under reflux for 1.5 h. The mixture was poured into saturated K_2CO_3 in ice-water bath. Layers were separated and the aqueous phase was extracted with CH_2Cl_2. The combined organic layer was dried over Na_2SO_4, filtered and the solvent was evaporated. Column chromatography on silica gel afforded compound **27** (0.82 g, 70%) as a major product along with compounds **28a** and **28b** (positions of N_3 group in compounds **28a** and **28b** were not assigned). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.83 (br. s., 6H), 3.66 - 3.86 (m, 3H), 3.88 - 4.06 (m, 2H), 4.29 (dd, *J* = 10.5, 8.7 Hz, 1H), 4.41 - 4.92 (m, 6H), 5.26 (d, *J* = 9.4 Hz, 1H), 7.12 - 7.50 (m, 15H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 9.0 (q), 55.4 (d), 68.4 (t), 73.8 (t), 75.2 (t), 75.3 (t), 77.4 (d), 79.2 (d), 79.8 (d), 86.0 (d), 127.7, 128.0, 128.1, 128.2, 128.6, 128.7, 138.0, 138.1, 138.5, 171.5; HRMS (FAB), calcd for $C_{33}H_{33}N_4O_6$ (M–H$^+$), 581.2400, found 581.2407. **Compound 28a:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.74 (br. s., 6H), 3.17 (d, *J* = 6.6 Hz, 1H), 3.32 (d, *J* = 5.5 Hz, 1H), 3.37 - 3.75 (m, 6H), 3.93 (dd, *J* = 10.9, 8.1 Hz, 1H), 4.01 - 4.21 (m, 3H), 4.31 - 4.82 (m, 6H), 5.11 (d, *J* = 8.6 Hz, 1H), 5.18 (s, 1H), 6.96 - 7.37 (m, 15H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 8.9, 55.7, 65.3, 67.3, 68.9, 72.7, 73.7, 74.8, 75.2, 76.3, 79.8, 80.1, 85.1, 98.6, 102.0, 127.7, 128.1, 128.3, 128.5, 128.7, 137.5, 137.6, 138.3 ; MS (FAB) [M–H$^+$] 725. **Compound 28b:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.80 (br. s., 6H), 3.23 (d, *J* = 6.6 Hz, 1H), 3.38 (d, *J* = 5.4 Hz, 1H), 3.48 (dd, *J* = 10.1, 7.5 Hz, 1H), 3.53 (t, *J* = 9.4 Hz, 1H), 3.60 (dd, *J* = 7.6, 5.6 Hz, 1H), 3.64 - 3.78 (m, 4H), 3.99 (dd, *J* = 10.6, 8.6 Hz, 1H), 4.12 (dd, *J* = 10.7, 8.7 Hz, 1H), 4.16 - 4.26 (m, 1H), 4.38 - 4.59 (m, 4H), 4.82 (dd, *J* = 11.6, 6.2 Hz, 2H), 5.17 (d, *J* = 8.4 Hz, 1H), 5.24 (s, 1H), 7.05 - 7.41

(m, 15H); ^{13}C NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 8.9 (q), 55.7, 65.2 (d), 67.3 (t), 68.8 (t), 72.7 (d), 73.7 (t), 74.8 (d), 75.2 (t), 75.2 (d), 76.3 (d), 79.8 (d), 80.1 (d), 85.1 (d), 98.6 (d), 102.0, 127.7, 128.1, 128.3, 128.4, 128.6, 128.7, 137.5, 137.6, 138.3, 171.9; MS (FAB) [M–H$^+$] 725.

Azido 3,4,6-tri-*O*-acetyl-2-dimethylmaleimido-2-deoxy-β-D-glycopyranoside (30). Compound **29** was subjected to the same condition described for compound **27** to give compound **30** in 80%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.83 (s, 3H), 1.89 (s, 9H), 1.94 (s, 3H), 2.02 (s, 3H), 3.85 (ddd, *J* = 10.2, 4.6, 2.3 Hz, 1H, H-5), 3.91 (dd, *J* = 10.6, 9.6 Hz, 1H, H-2), 4.09 (dd, *J* = 12.4, 2.2 Hz, 1H, H-6), 4.24 (dd, *J* = 12.5, 4.7 Hz, 1H, H-6), 5.05 (t, *J* = 9.8 Hz, 1H, H-4), 5.41 (d, *J* = 9.4 Hz, 1H, H-1), 5.54 (dd, *J* = 10.6, 9.2 Hz, 1H, H-3); ^{13}C NMR (126 MHz, , $CDCl_3$) δ 8.8 (q), 20.4 (q), 20.6 (q), 20.7 (q), 53.8 (d, C-2), 61.7 (t, C-6), 68.4 (d, C-4), 70.5 (d, C-3), 73.9 (d, C-5), 85.6 (d, C-1), 169.4, 170.0, 170.6; HRMS (FAB), calcd for $C_{18}H_{22}NO_9$ (M–N_3^+), 396.1295, found 396.1312.



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3-*O*-Benzyl-4,6-*O*-benzylidene-2-deoxy-2-dimethylmaleimido-β-D-glucopyranosyl-(1→4)-2-acetamido-1,6-anhydro-2-deoxy-β-D-glucopyranose (22). Compounds **13** (1.2 g, 5.0 mmol) and **18**[7] (3.4 g, 5.5 mmol) were dissolved in anhydrous CH_2Cl_2 (50 mL), 4-Å molecular sieves (5 g) was added, and the suspension was stirred under argon for 2 h. The catalyst TfOH (0.5 mL) was added in two portions, and the solution was stirred at room temperature for 1 h. The reaction was quenched by adding triethylamine (0.1 mL) and it was filtered through a layer of Celite. The combined filtrate was concentrated to dryness and the residue was purified by flash chromatography (CH_2Cl_2/MeCN/MeOH, 10:3:0.5) to give **22** (2.6 g) in 75%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.84 (br. s, 6H), 2.05 (s, 3H), 2.11 (s, 3H), 3.57 (td, *J* = 9.8, 5.0 Hz, 1H, H-5′), 3.63 (br. s, 1H, H-4), 3.65 (dd, *J* = 7.6, 5.8 Hz, 1H, H-6a), 3.78 (t, *J* = 9.2 Hz, 1H, H-4′), 3.83 (t, *J* = 10.4 Hz, 1H, H-6′a), 3.99 (d, *J* = 7.4 Hz, 1H, H-6b), 4.03 (d, *J* = 10.2 Hz, 1H, H-2), 4.07 (dd, *J* = 10.6, 8.6 Hz, 1H, H-2′), 4.12 (d, *J* = 5.4 Hz, 1H, H-5), 4.35 (dd, *J* = 10.6, 5.0 Hz, 1H, H-6′b), 4.49 (dd, *J* = 10.4, 9.0 Hz, 1H, H-3′), 4.52 and 4.85 (AB, *J* = 12.5 Hz, 2H, OC*H*$_2$Ph), 4.73 (s, 1H, H-3), 5.03 (d, *J* = 8.4 Hz, 1H, H-1′), 5.13 (s, 1H, H-1), 5.60 (s, 1H, C*H*Ph), 5.99

[7] Hesek, D.; Lee, M.; Morio, K.-I.; Mobashery, S. *J. Org. Chem.* **2004**, *69*, 2137–2146.

(d, J = 10.0 Hz, 1H, NH), 7.11 - 7.55 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 8.7, 21.0, 22.9 (3 × q), 48.4 (d, C-2), 55.8 (d, C-2′), 64.4 (t, C-6), 66.2 (d, C-5′), 68.5 (t, C-6′), 70.7 (d, C-3), 72.3 (d, C-5), 73.1 (d, C-4), 74.2 (t, O*C*H$_2$Ph), 74.4 (d, C-3′), 82.6 (d, C-4′), 96.8 (d, C-1′), 100.9 (d, C-1), 101.3 (d, *C*HPh), 126.1, 127.5, 128.2, 128.3, 129.1, 137.2, 138.3, 169.1, 169.8, 172.3; HRMS (FAB), calcd for $C_{36}H_{41}N_2O_{12}$ ($M+H^+$), 693.2660, found 693.2658.

2-Acetamido-3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-β-D-glucopyranosyl-(1→4)-2-acetamido-1,6-anhydro-2-deoxy-β-D-glucopyranose (31). NaOH (3.0 g of solid pellets, 75 mmol) was added to a stirred solution of **22** (2.1 g, 3.0 mmol) in a mixture of 1,4-dioxane/water (4:1, 150 mL) at 10 °C under a nitrogen atmosphere. The mixture was sonicated for 15 min and then the mixture was allowed to warm to room temperature and was let stir for an additional 3 h. The pH was adjusted to 3 by the addition of 0.5 N HCl and the resulting reaction mixture was stirred for 10 h at room temperature. Evaporation of the mixture to dryness gave a yellow oily residue, which was treated with MeOH (100 mL). The precipitated inorganic material was filtered off and washed well with cold MeOH. The filtrate was evaporated to dryness. The residue was taken up into pyridine (15 mL) and the solution was treated with an excess of acetic anhydride (20 mL), followed by stirring at room temperature for 20 h. The solution was evaporated to dryness. The crude product was purified with column chromatography using EtOAc/MeCN (3/2) to give **31** (1.0 g, 55%) as a white powder. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.94 (s, 3H), 2.01 (s, 3H), 2.03 (s, 3H), 2.09 (s, 3H), 3.41 (dt, J = 9.5, 4.7 Hz, 1H, H-5′), 3.64 - 3.83 (m, 5H, H-4, H-3′, H-4′, H-6a, H-6′a), 4.03 (d, J = 7.4 Hz, 1H, H-6b), 4.06 (d, J = 9.2 Hz, 1H, H-2), 4.09 (m, 1H, H-2′), 4.30 (dd, J = 10.5, 5.1 Hz, 1H, H-6′b), 4.55 (d, J = 5.4 Hz, 1H, H-5), 4.60 (d, J = 8.2 Hz, 1H, H-1′), 4.67 and 4.88 (AB, J = 12.1 Hz, 2H, OC*H*$_2$Ph), 4.73 (s, 1H, H-3), 5.24 (s, 1H, H-1), 5.53 (s, 1H, C*H*Ph), 6.65 (d, J = 9.0 Hz, 1H, NH), 7.00 (d, J = 10.0 Hz, 1H, NH′), 7.18 - 7.53 (m, 14H), 8.57 (d, J = 4.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.0, 22.8, 23.6 (3 × q), 48.5 (d, C-2), 54.9 (d, C-2′), 64.6 (t, C-6), 66.2 (d, C-5′), 68.6 (t, C-6′), 71.2 (d, C-3), 72.1 (d, C-5), 72.8 (d, C-4), 74.0 (t, O*C*H$_2$Ph), 77.26 and 81.9 (2d, C-3′ and C-4′), 100.0 (d, C-1′), 100.8 (d, C-1), 101.2 (d, *C*HPh), 124.3, 126.0, 127.9, 128.2, 128.3, 128.4, 129.1, 137.2, 137.3, 148.5, 169.4, 171.1, 172.2; HRMS (FAB), calcd for $C_{32}H_{39}N_2O_{11}$ ($M+H^+$), 627.2554, found 627.2572**.**

2-Acetamido-3-*O*-benzyl-4,6-*O*-benzylidene-2-deoxy-β-D-glucopyranosyl-(1→4)-2-acetamido-1,6-anhydro-2-deoxy-3-*O*-[(1*R*)-1-carboxyethyl]-β-D-glucopyranose (33). A solution of NaOMe (86 mg, 1.6 mmol) in 1 mL anhydrous MeOH was added dropwise to a suspension of **31** (1.0 g, 1.6 mmol) in anhydrous MeOH (20 mL). The mixture was stirred at room temperature for 1 h under an atmosphere of nitrogen and then after neutralization with acetic acid (0.5 mL)**,** the solution was evaporated to dryness. The residue (compound **32**) was dissolved in anhydrous 1,4-dioxane/DMF (1/1, 10 mL), 4-Å

molecular sieves (1 g) were added, and the suspension was stirred under nitrogen for 1 h. After addition of NaH (0.19 g, 4.8 mmol, 60% dispersion in oil), the mixture was kept for 30 min at 45 ºC, and then the temperature was brought to room temperature. (*S*)-2-Chloropropionic acid (0.87 g, 8.0 mmol) was added and the stirring was continued for 2 h at 90 ºC. The organic solvent was evaporated to dryness. A 50 mL portion of water was carefully added to the residue to decompose the excess of sodium hydride. The solution was extracted once with hexanes/EtOAc (1/1) to remove the mineral oil and the solution was filtered over a layer of charcoal. The solution was brought to pH 3.0 by the addition of 2.5 M hydrochloric acid at the ice-water temperature. The resulting precipitate was immediately extracted with CH_2Cl_2. The combined organic layer was washed with water, rapidly dried over Na_2SO_4, and concentrated to dryness. The residue was purified by column chromatography on silica column, using $CHCl_3$/acetone (3/1) to afford compound **33** (0.50 g, 48%). **32:** ^{1}H NMR (500 MHz, DMF-*d6*) δ 2.01 (s, 3H), 2.07 (s, 3H), 3.55 (td, *J* = 9.6, 5.0 Hz, 1H, H-5′), 3.62 (s, 1H, H-3), 3.67 (t, *J* = 6.4 Hz, 2H, H-6a), 3.79 (m, 1H, H-4), 3.84 (t, *J* = 9.3 Hz, 1H, H-4′), 3.91 (td, *J* = 9.9, 3.6 Hz, 2H, H-3′ and , H-6′a), 3.98 (d, *J* = 9.8 Hz, 1H, H-2), 4.09 (q, *J* = 9.2 Hz, 1H, H-2′), 4.28 (d, *J* = 7.0 Hz, 1H, H-6b), 4.29 - 4.33 (dd, *J* = 10.2, 4.9 Hz, 1H, H-6′b), 4.77 and 4.89 (AB, *J* = 11.7 Hz, 2H, OC*H*$_2$Ph), 4.79 (s, 1H, H-5), 4.86 (d, *J* = 8.4 Hz, 1H, H-1′), 5.20 (s, 1H, H-1), 5.79 (s, 1H, C*H*Ph), 7.07 (d, *J* = 10.0 Hz, 1H, NH), 7.25 - 7.56 (m, 10H), 8.43 (d, *J* = 9.3 Hz, 1H, NH′); ^{13}C NMR (126 MHz, DMF-*d6*) δ 22.2, 23.2 (2 × q), 51.3 (d, C-2), 55.1 (d, C-2′), 64.5 (t, C-6), 66.2 (d, C-5′), 68.4 (t, C-6′), 71.2 (d, C-3), 72.3 (d, C-5), 74.0 (t, O*C*H$_2$Ph), 75.3 (d, C-4), 78.9 (d, C-3′), 81.9 (d, C-4′), 99.9 (d, C-1′), 100.9 (d, *C*HPh), 101.4 (d, C-1), 126.3, 127.6, 127.7, 128.3, 129.0, 138.3, 139.4, 169.8, 172.0; HRMS (FAB), calcd for $C_{30}H_{37}N_2O_{12}$ (M+H$^+$), 585.2448, found 585.2444. **33:** ^{1}H NMR (500 MHz, CD_3CN) δ 1.34 (d, *J* = 6.8 Hz, 3H), 1.92 (s, 3H), 2.01 (s, 3H), 3.36 (m, 1H, H-3), 3.43 (td, *J* = 9.6, 5.1 Hz, 1H, H-5′), 3.64 - 3.84 (m, 5H, H-6a, H-3′, H-4′, H-4, H-6′a), 3.93 (dd, *J* = 10.0, 8.4 Hz, 1H, H-2′), 4.01 (s, 1H, H-2), 4.11 (q, *J* = 6.8 Hz, 1H, Lac-α-H), 4.12 (t, *J* = 6.9 Hz, 1H, H-6b), 4.27 (dd, *J* = 10.4, 5.0 Hz, 1H, H-6′b), 4.56 (d, *J* = 5.6 Hz, 1H, H-5), 4.59 (d, *J* = 8.4 Hz, 1H, H-1′), 4.66 and 4.82 (AB, *J* = 11.7 Hz, 2H, OC*H*$_2$Ph), 5.22 (s, 1H, H-1), 5.63 (s, 1H, C*H*Ph), 7.25 - 7.51 (m, 10H); ^{13}C NMR (126 MHz, CD_3CN) δ 18.9, 23.0, 23.7 (3 × q), 49.9 (d, C-2), 55.8 (d, C-2′), 65.7 (t, C-6), 67.1 (d, C-5′), 69.3 (t, C-6′), 73.8 (d, C-5), 74.3 (d, C-4), 75.0 (t, O*C*H$_2$Ph), 75.3 (d, Lac-α-C), 78.7 (d, C-3), 79.2 (d, C-3′), 82.8 (d, C-4′), 101.3 (d, C-1′), 101.9 (d, C-1), 102.0 (d, *C*HPh), 127.2, 128.7, 129.0, 129.3, 130.0, 138.9, 139.9, 171.5, 173.0, 175.4; HRMS (FAB), calcd for $C_{33}H_{41}N_2O_{12}$ (M+H$^+$), 658.2660, found 658.2660.

Compound 36. EDCI (0.18 g, 0.94 mmol) was added to a mixture of *N*-hydroxysuccinimde (0.11 g, 0.96 mmol) and **33** (0.5 g, 0.76 mmol) in CH_2Cl_2 (5 mL) in an ice-water bath. The mixture was stirred at room temperature 20 h. Meanwhile, the Boc-protected pentapeptide (0.85 g, 0.91 mmol) in CH_2Cl_2 (5 mL) was treated with trifluoroacetic acid (2 mL) in an ice-water bath. Temperature was gradually

increased to room temperature over 1 h. The reaction mixture was evaporated to dryness under reduced pressure and the residue was dissolved in toluene. This was followed by evaporation to dryness. The residue (**34**) was dissolved in iPr_2NEt (0.4 mL, 2.3 mmol) and DMF (5 mL), and the solution was then added to the NHS-ester of the anhydrosugar, prepared above. The resulting mixture was stirred at room temperature 20 h. The mixture was diluted with CH_2Cl_2 and water was added. Layers were separated. The organic layer was dried over $MgSO_4$, filtered, concentrated and the sample was subjected to column chromatography on silica gel (CH_2Cl_2/MeCN/MeOH, 10:3:0.5) to give the title compound (0.84 g, 75%). ^{1}H NMR (500 MHz, CD_3CN) δ 1.32 (d, *J* = 7.2 Hz, 3H), 1.36 (2d, 6H), 1.40 (d, *J* = 7.4 Hz, 3H), 1.41 - 1.97 (m, 8H), 1.99 (s, 3H), 2.05 (s, 3H), 2.15 - 2.29 (m, 2H), 3.42 (s, 1H, H-3), 3.50 (td, *J* = 9.7, 5.0 Hz, 1H, H-5′), 3.71 - 3.91 (m, 5H, H-5, H-6a H-3′, H-4′, H-6′a), 3.96 - 4.04 (m, 2H, H-2, H-2′), 4.04 - 4.14 (m, 3H), 4.24 (d, *J* = 7.6 Hz, 1H, H-6b), 4.28 - 4.45 (m, 6H), 4.31 (m, 1H, H-6′b), 4.65 (d, *J* = 8.4 Hz, 1H, H-1′), 4.63 - 4.68 (m, 1H, H-4), 4.73 and 4.88 (2d, *J* = 11.8 Hz, 1H), 5.09 - 5.19 (m, 4H), 5.24 (s, 2H), 5.34 (s, 1H, H-1), 5.69 (s, 1H, C*H*Ph), 6.77 (d, *J* = 9.4 Hz, 1H), 6.92 (d, *J* = 10.0 Hz, 1H), 7.30 - 7.58 (m, 15H), 7.95 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (126 MHz, CD_3CN) δ 17.6, 17.9, 18.0, 18.7, 22.9, 23.8, (6 × q), 22.9, 27.7, 31.7, 31.7, 32.0 (5 × t), 48.4 (d, C-2), 49.3, 49.9, 50.6, 52.1, 55.5 (5 × d), 55.8 (d, C-2′), 62.7 (d), 65.4 (t, C-6), 67.0 (d, C-5′), 67.3 (t), 67.6 (t), 68.2 (t), 69.2 (t, C-6′), 73.5 (d, C-5), 74.6 (d, C-4), 75.0 (t, O*C*H_2Ph), 76.9 (d, Lac-α-C), 79.2 (d, C-3′), 79.4 (d, C-3), 82.7 (d, C-4′), 100.8 (d, C-1′), 101.9 (d, C-1 and *C*HPh), 127.2, 128.6, 128.9, 129.1, 129.2, 129.3, 129.4, 129.5, 129.7, 130.0, 136.8, 137.1, 137.2, 138.9, 139.8, 171.1, 171.4, 172.3, 172.9, 173.2, 173.5, 173.9, 174.0, 174.3; FAB MS *m/z* 1441.96 $[M+H]^+$.

***N*-Acetyl-β-D-glucosamine-(1→4)-1,6-anhydro-β-D-*N*-acetylmuramyl-L-Ala-γ-D-Glu-*meso*-DAP-D-Ala-D-Ala (1).** Compound **36** (0.50 g, 0.34 mmol) in AcOH (5 mL) and stirred at 60 ºC for 2 h. After removal of AcOH, the residue was dissolved in MeOH (5 mL) and stirred in the presence of 10% Pd/C (0.1 g) under an atmosphere of hydrogen at 50 ºC for 3 h. The reaction mixture was filtered through a layer of Celite and the residue was washed with MeOH. The combined filtrate was concentrated to dryness under reduced pressure. The crude product was subjected to HPLC purification to afford compound **1** (0.22 g, 66%). Preparative HPLC purifications were performed on delta-pak C18 reversed-phased column, 100 Å pore size, 19 × 300 mm using a linear gradient of 5-15% acetonitrile in water supplemented with 0.1% TFA over 0.5 h. ^{1}H NMR (600 MHz, D_2O) δ 1.32 - 1.46 (4d, 12H), 1.46 - 1.56 (m, 2H), 1.73 - 2.03 (m, 5H), 2.04, 2.06 (2s, 6H), 2.22 – 2.45 (m, 3H), 3.45 (m, 2H, H-5′, H-4′), 3.53 - 3.62 (m, 2H, H-3′, H-3), 3.68 - 3.84 (m, 3H, H-6′a, H-2′, H-6a), 3.90 (d, *J* = 12.0 Hz, 1H, H-6′b), 3.99 (d, *J* = 12.0 Hz, 2H, H-2, H-4), 4.06 (t, *J* = 6.2 Hz, 1H), 4.15 - 4.44 (m, 6H), 4.66 (d, *J* = 8.5 Hz, 1H, H-1′), 4.70 (d, *J* = 5.0 Hz, 1H, H-5), 5.44 (s, 1H, H-1); ^{13}C NMR (151 MHz, D_2O) δ 15.9, 16.3 16.7, 17.9, 20.7, 22.2 (6 × q), 21.8, 26.4, 29.2, 30.1, 31.1 (5 × t), 48.8 (d, C-2), 48.5, 49.4, 51.7, 52.5, 53.8 (6 × d),

55.4 (d, C-2′), 60.5 (t, C-6′), 64.7 (t, C-6), 69.7 (t, C-4′), 73.2 (t, C-3′), 73.4 (C-5), 74.1 (C-4), 75.9 (d, C-5′), 76.0 (d, Lac-α-C), 77.2 (d, C-3), 99.8 (d, C-1), 100.4 (d, C-1′), 171.8, 173.5, 173.8, 174.4, 174.6, 175.0, 175.3, 175.4, 176.0 (10 × C=O); HRMS (FAB), calcd for $C_{40}H_{65}N_8O_{21}$ ($M+H^+$), 993.4264, found 993.4230.

***N*-Acetyl-β-D-glucosamine-(1→4)-1,6-anhydro-β-D-*N*-acetylmuramyl-L-Ala-γ-D-Glu-L-Lys-D-Ala-D-Ala (2).** This material was prepared in the same manner as described for **1**, with the exception that pentapeptide **35**[8] was used in place of **34**. ^{1}H NMR (500 MHz, CD_3OD) δ 1.34 - 1.48 (4d, 16H), 1.51 (br. s, 1H), 1.63 - 1.74 (m, 1H), 1.78 - 1.93 (m, 2H), 2.04 (s, 3H), 2.07 (s, 3H), 2.20 - 2.35 (m, 3H), 2.95 (m, 2H), 3.28 - 3.39 (m, 2H, H-5′, H-4′), 3.49 (dd, *J* = 10.3, 8.3 Hz, 1H, H-3′), 3.55 (s, 1H, H-3), 3.71 (dd, *J* = 11.9, 5.3 Hz, 1H, H-6′a), 3.74 - 3.80 (m, 2H, H-2′, H-6a), 3.84 - 3.94 (m, 2H, H-4, H-6′b), 3.99 (s, 1H, H-2), 4.14 (q, *J* = 6.8 Hz, 1H, Lac-α-H), 4.23 - 4.43 (m, 7H), 4.53 (d, *J* = 8.4 Hz, 1H, H-1′), 4.65 (d, *J* = 5.0 Hz, 1H, H-5), 5.33 (s, 1H, H-1); ^{13}C NMR (126 MHz, CD_3OD) δ 18.1, 18.3, 18.7, 18.9, 21.2, 22.9, 23.7 (6 × q), 28.3, 29.6 32.4, 32.9, 40.6 (5 × t), 49.4 (d, C-2), 50.7 (d), 51.0 (d), 51.2 (d), 54.1 (d), 55.6 (d), 57.2 (d, C-2′), 62.7 (t, C-6), 66.1 (t, C-6′), 72.1 (d, C-4′), 74.8 (d, C-5), 75.4 (d, C-3′), 75.6 (d, C-4), 77.5 (d, Lac-α-C), 78.3 (d, C-5′), 79.8 (d, C-3), 101.9 (d, C-1′), 102.1 (d, C-1), 173.5, 174.4, 174.6, 175.1, 175.6, 175.7, 175.8, 176.6, 177.7; HRMS (FAB), calcd for $C_{39}H_{65}N_8O_{19}$ ($M+H^+$), 949.4366, found 949.4387.

Table S1. Crystallographic Details

	4a	**5**	**6**	**8**	**11**	**30**
chemical formula	$C_{19}H_{30}O_4Si$	$C_{13}H_{16}O_4$, $0.28(C_7H_8)$	$C_{13}H_{15}IO_4$	$C_{13}H_{15}N_3O_4$	$C_{13}H_{14}O_4$	$C_{18}H_{22}N_4O_9$
formula weight	350.52	262.29	362.15	277.28	234.24	438.40
space group	$P2_12_12_1$	$P2_12_12_1$	$P1$	$P2_12_12_1$	$P2_1$	$P2_12_12$
a (Å)	6.6344(2)	4.5700(1)	6.1718(4)	6.7203(3)	10.9272(12)	17.5877(2)
b (Å)	10.9383(2)	13.9439(3)	7.5275(5)	10.1187(4)	19.943(2)	21.7941(2)
c (Å)	27.1190(6)	21.6237(5)	13.9663(6)	18.5765(8)	11.0323(11)	5.7479(1)
α (°)	90.00	90.00	91.5770(10)	90.00	90.00	90.00
β (°)	90.00	90.00	92.433(5)	90.00	110.217(6)	90.00
γ (°)	90.00	90.00	91.599(5)	90.00	90.00	90.00
V Å^3	1968.00(8)	1377.94(5)	647.74(7)	1263.22(9)	2256.1(4)	2203.22(5)
Z	4	4	2	4	8	4
T (°C)	296(2)	296(2)	100(2)	100(2)	100(2)	291(2)
λ (Å)	1.54178	1.54178	0.71073	1.54178	0.71073	1.54178
D_{obsd} (g cm^{-3})	1.183	1.264	1.857	1.458	1.379	1.322
μ (cm^{-1})	1.202	0.748	2.476	0.923	0.102	0.919
R1(F^2, $I > 2\sigma(I)$)	0.0265	0.0504	0.0160	0.0259	0.0820	0.0324
wR2(F^2)	0.0686	0.1500	0.0584	0.0660	0.2494	0.0899
S	1.055	0.935	1.303	1.106	1.098	1.045

$$wR2 = \sqrt{\frac{\sum[w(F_o^2 - F_c^2)^2]}{\sum[w(F_o^2)^2]}} \; ; \; R1 = \frac{\sum \| F_o| - |F_c \|}{\sum |F_o|} \; ; \; GooF = S = \sqrt{\frac{\sum[w(F_o^2 - F_c^2)^2]}{(n-p)}}$$

n= number of reflections, *p*= number of parameters refined

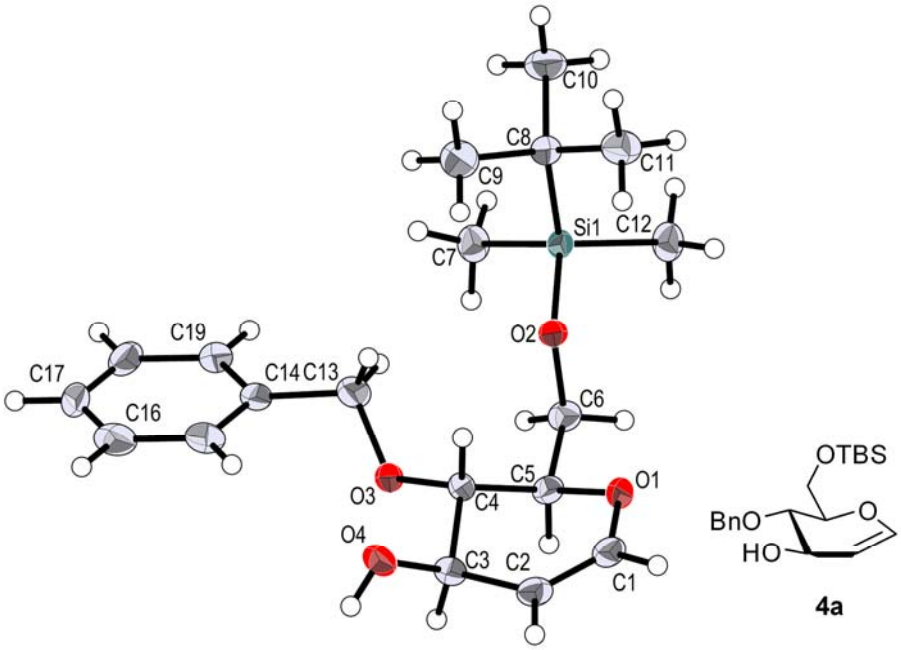


Figure S1. The molecular structure of compound **4a**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii.

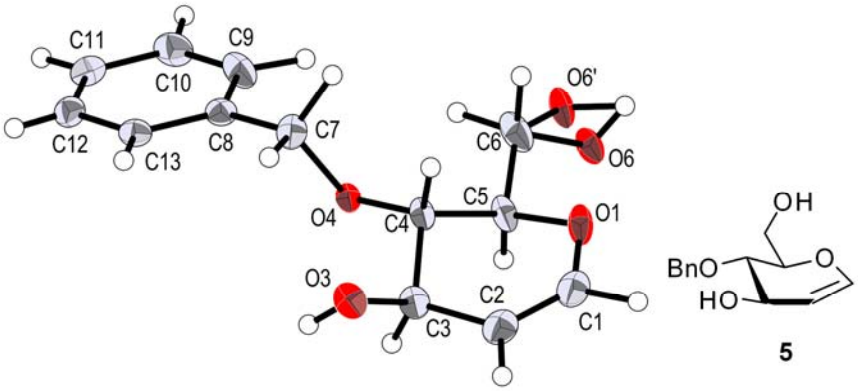


Figure S2. The molecular structure of compound **5**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii. Disorder is present in the position of O6. Two sites were modeled, with site occupancy for the first at 0.79(3). One hydrogen is shared between these atomic positions. The disordered, partially present molecule of toluene has been omitted.

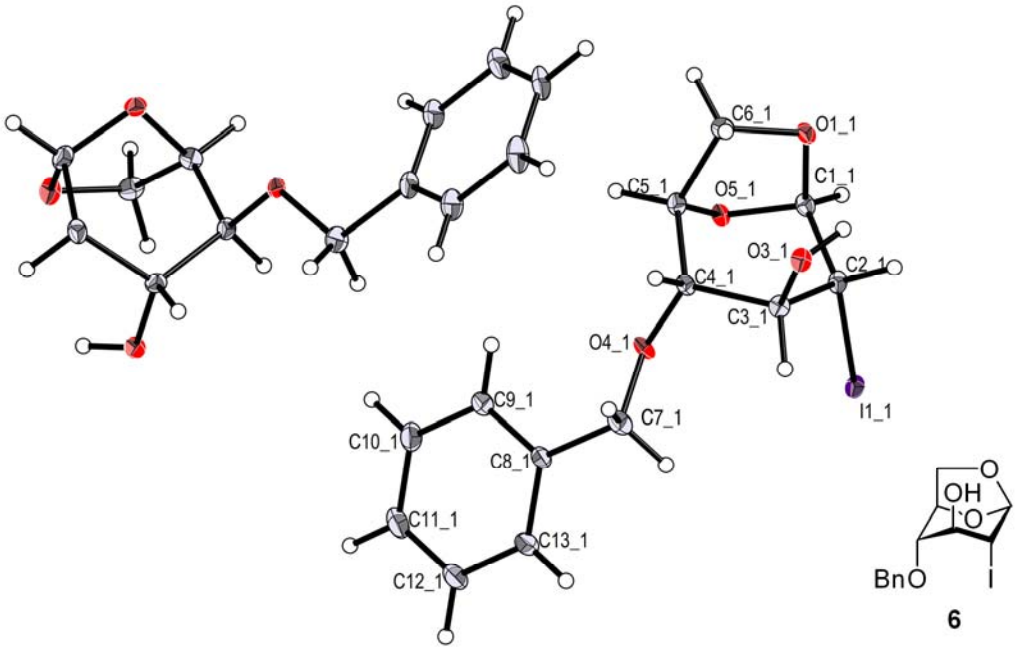


Figure S3. The molecular structure of compound **6**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii. Two independent molecules are present in the unit cell.

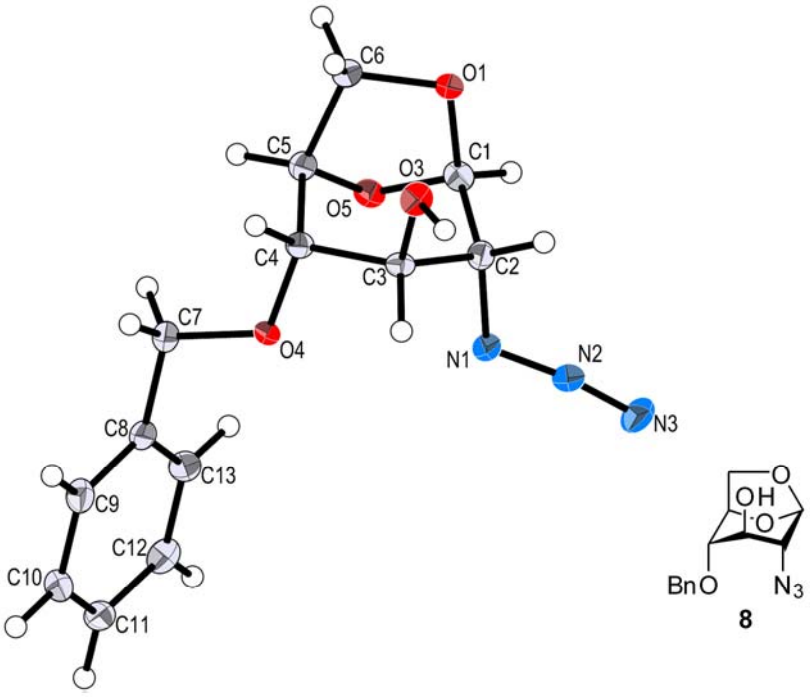


Figure S4. The molecular structure of compound **8**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii.

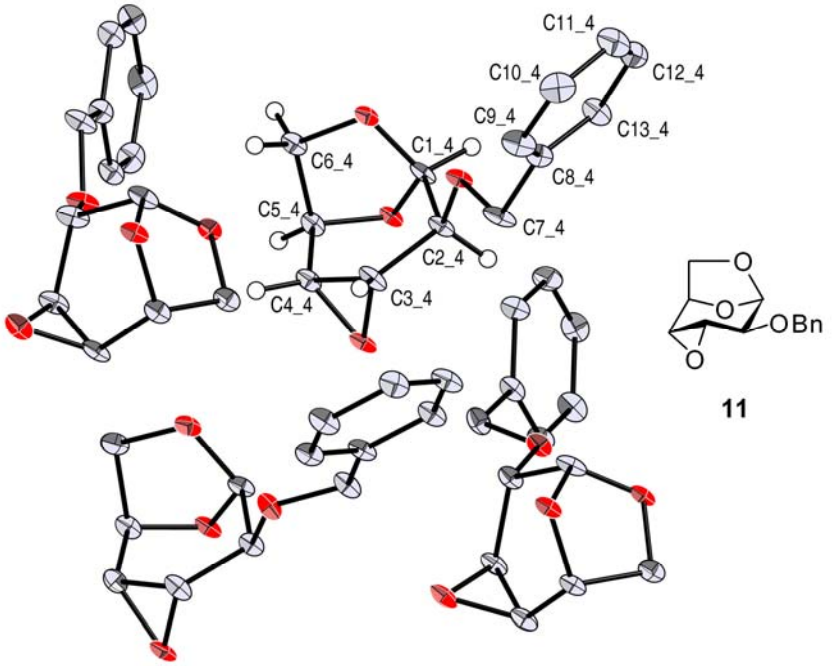


Figure S5. The molecular structure of compound **11**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii. Four independent molecules are present in the unit cell.

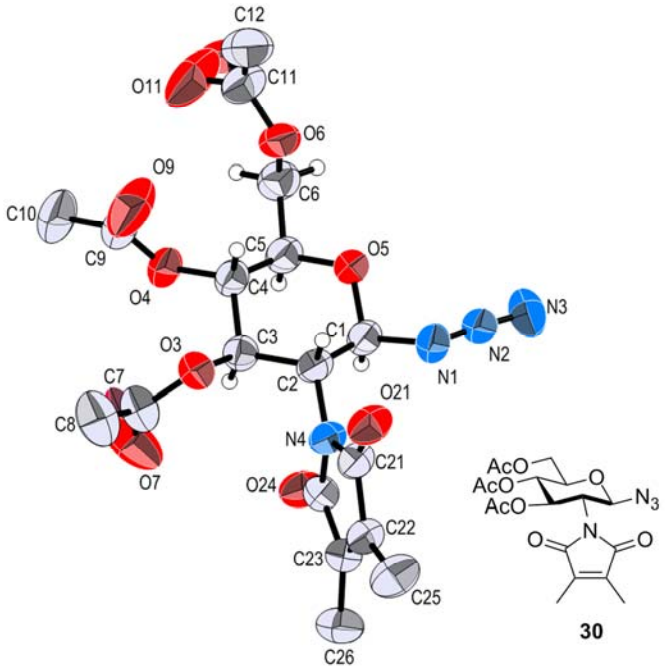
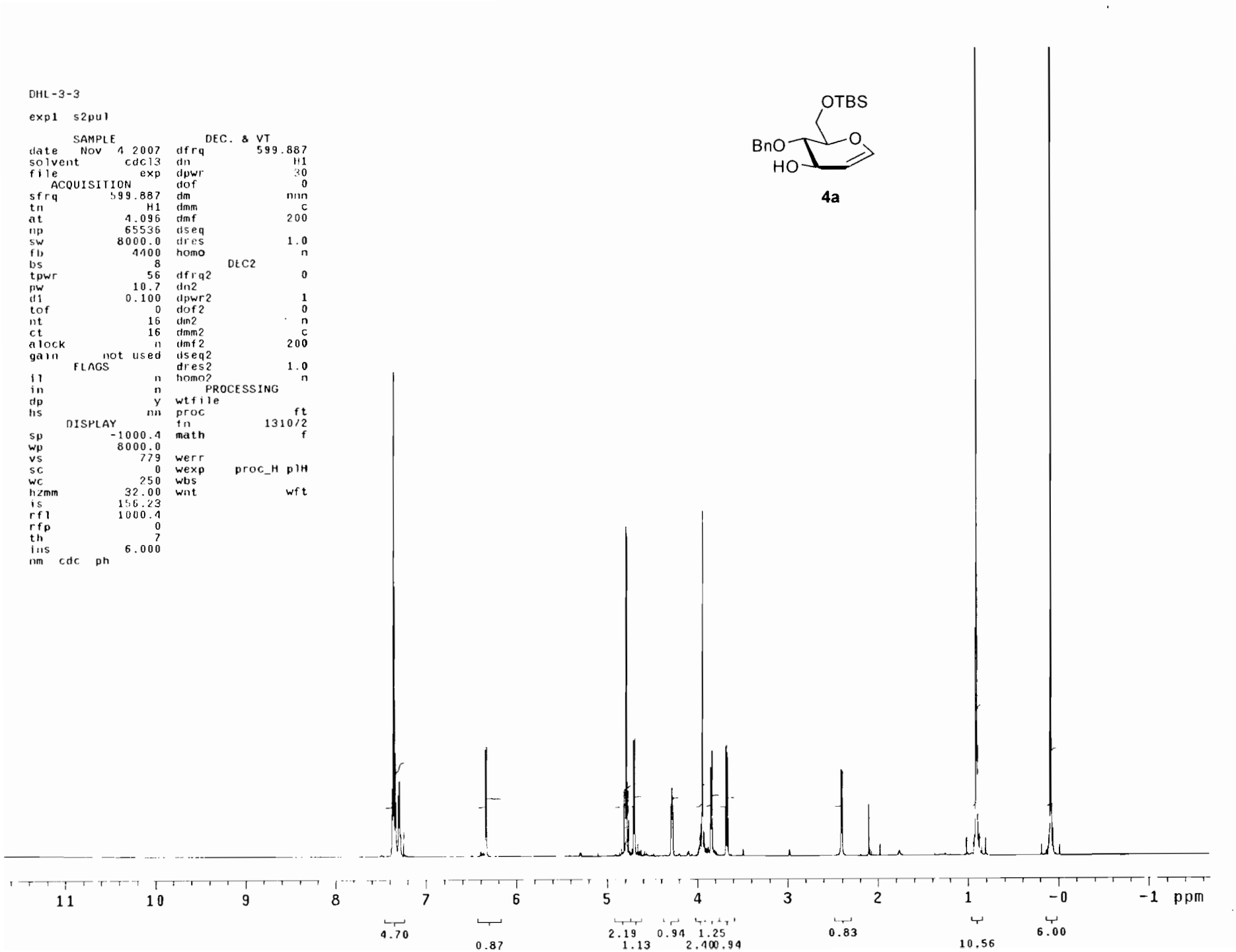


Figure S6. The molecular structure of compound **30**, showing the atom-numbering scheme. The ORTEP diagram is shown at 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radii.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fb 4400
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pw 10.7
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ct 16
alock n
gain not used
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in n
dp y
hs nn
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sc 0
wc 250
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nm cdc ph
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dfrq 599.887
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
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proc ft
fn 1310/2
math f
werr
wexp proc_H plH
wbs
wnt wft
OTBS
BnO
HO
O
4a
11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
4.70
0.87
2.19
1.13
0.94
1.25
2.40
0.94
0.83
10.56
6.00

DHE-3-3

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Ambient temperature
UNITYplus-600 "tesla"

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Width 4793.9 Hz
2D Width 4793.9 Hz
8 repetitions
256 increments
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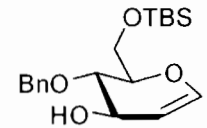
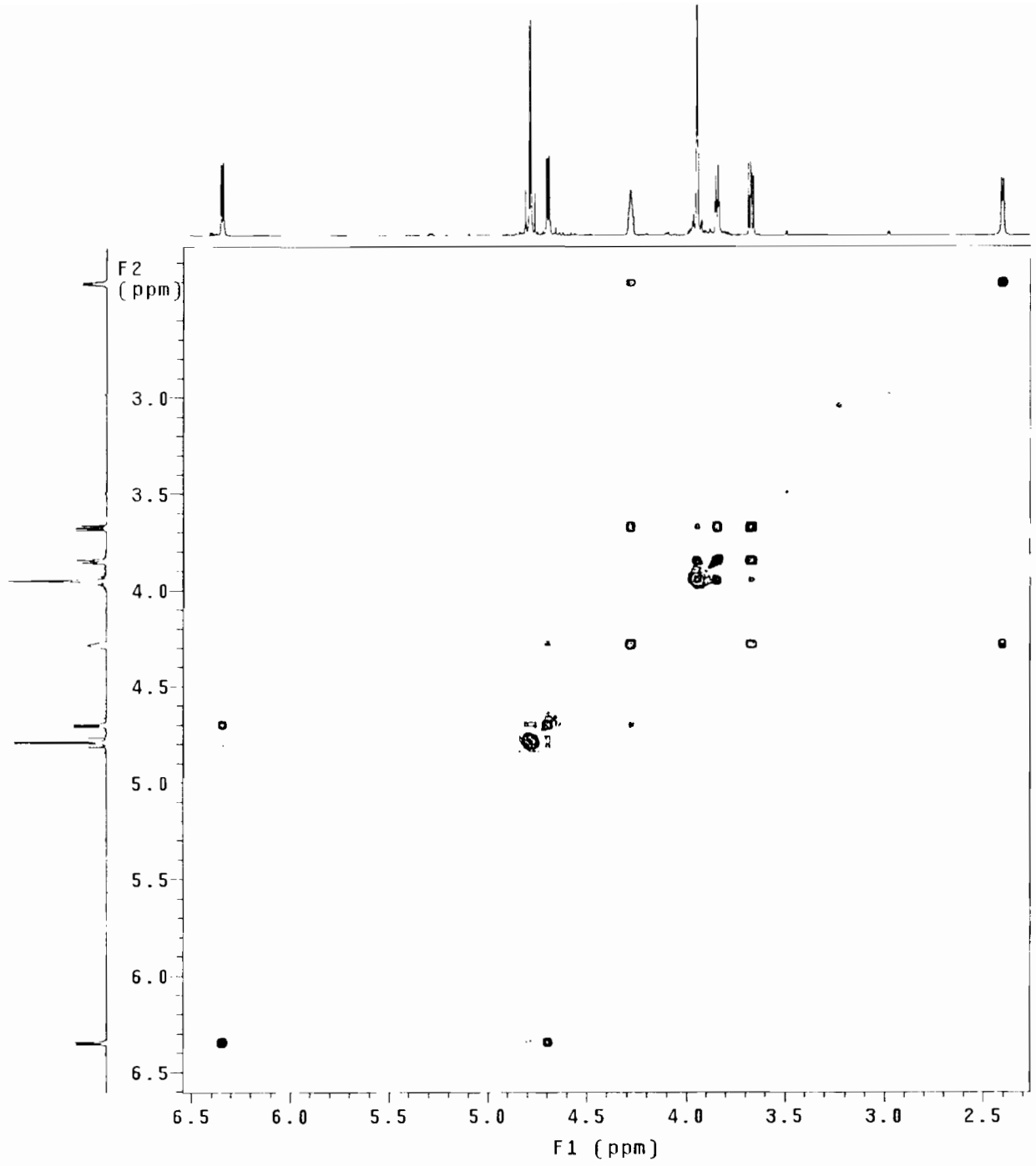
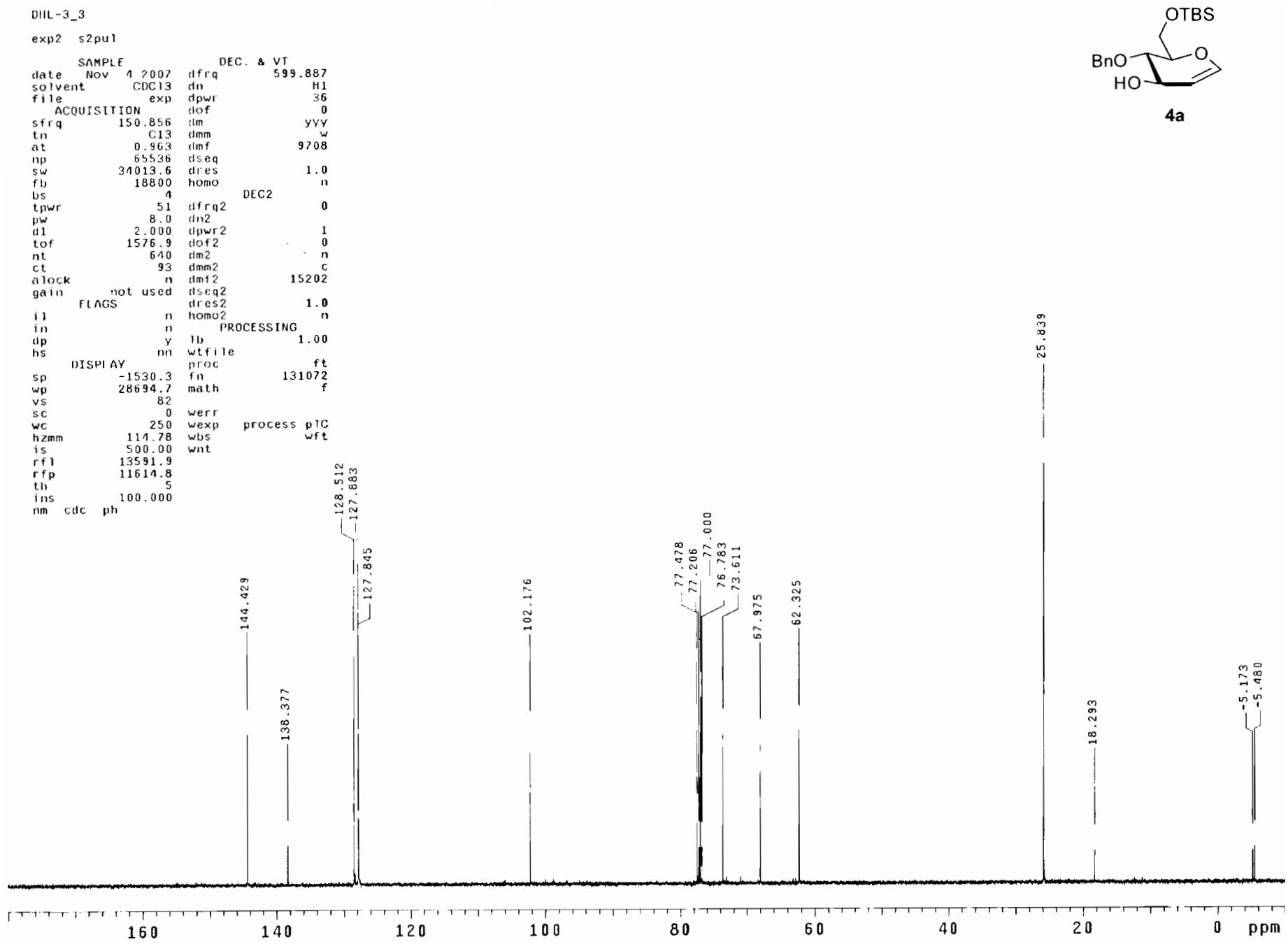


4a

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tpwr 51
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gain not used
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in n
dp y
hs nn
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is 500.00
rfl 13591.9
rfp 11614.8
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ins 100.000
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dfrq 599.887
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dmm w
dmf 9708
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 15202
dseq2
dres2 1.0
homo2 n
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wnt
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BnO
HO
O
4a
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128.512
127.883
127.845
102.176
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67.975
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120
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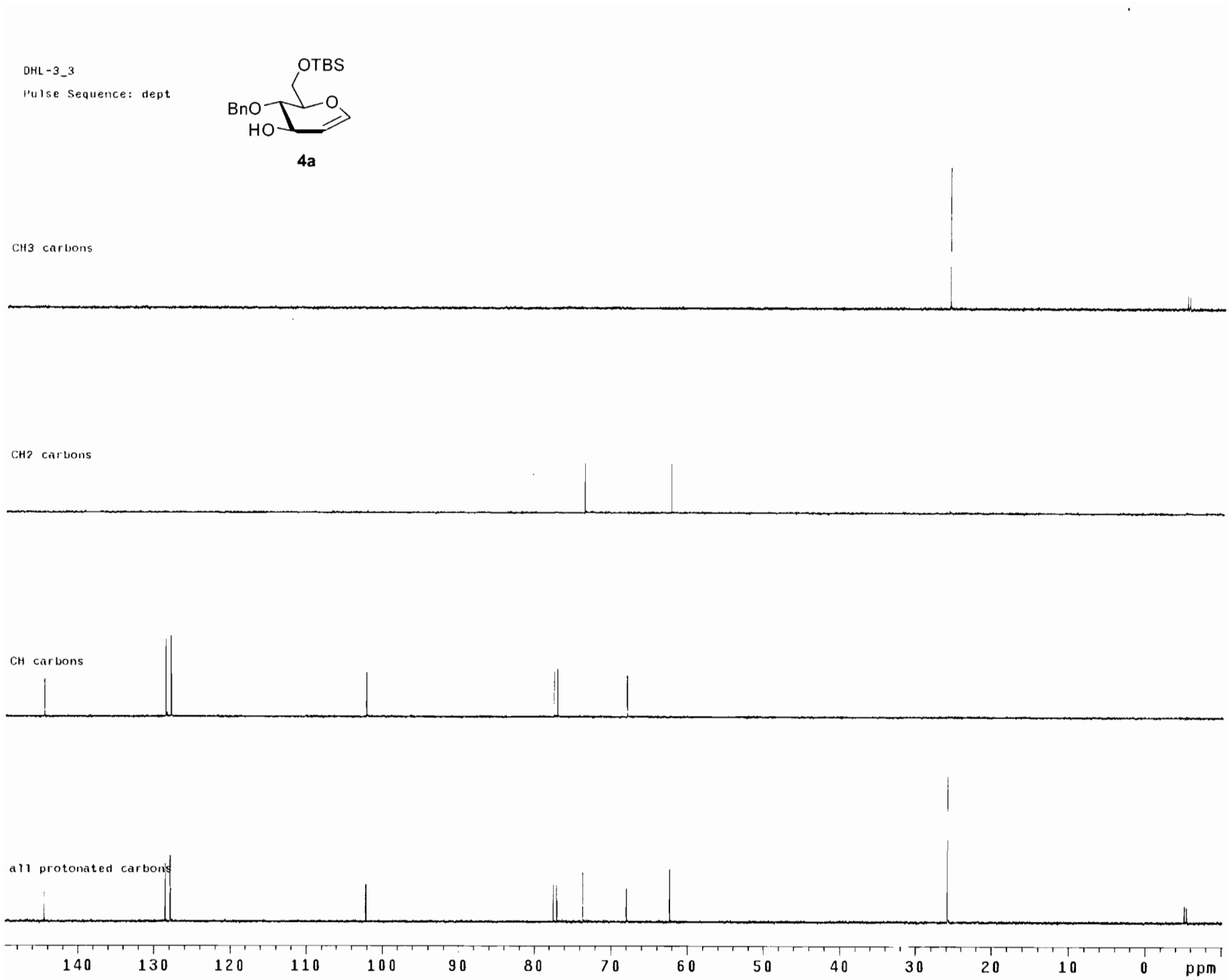
60
40
20
0
ppm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O
HO
4a
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CH2 carbons
CH carbons
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60
50
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30
20
10
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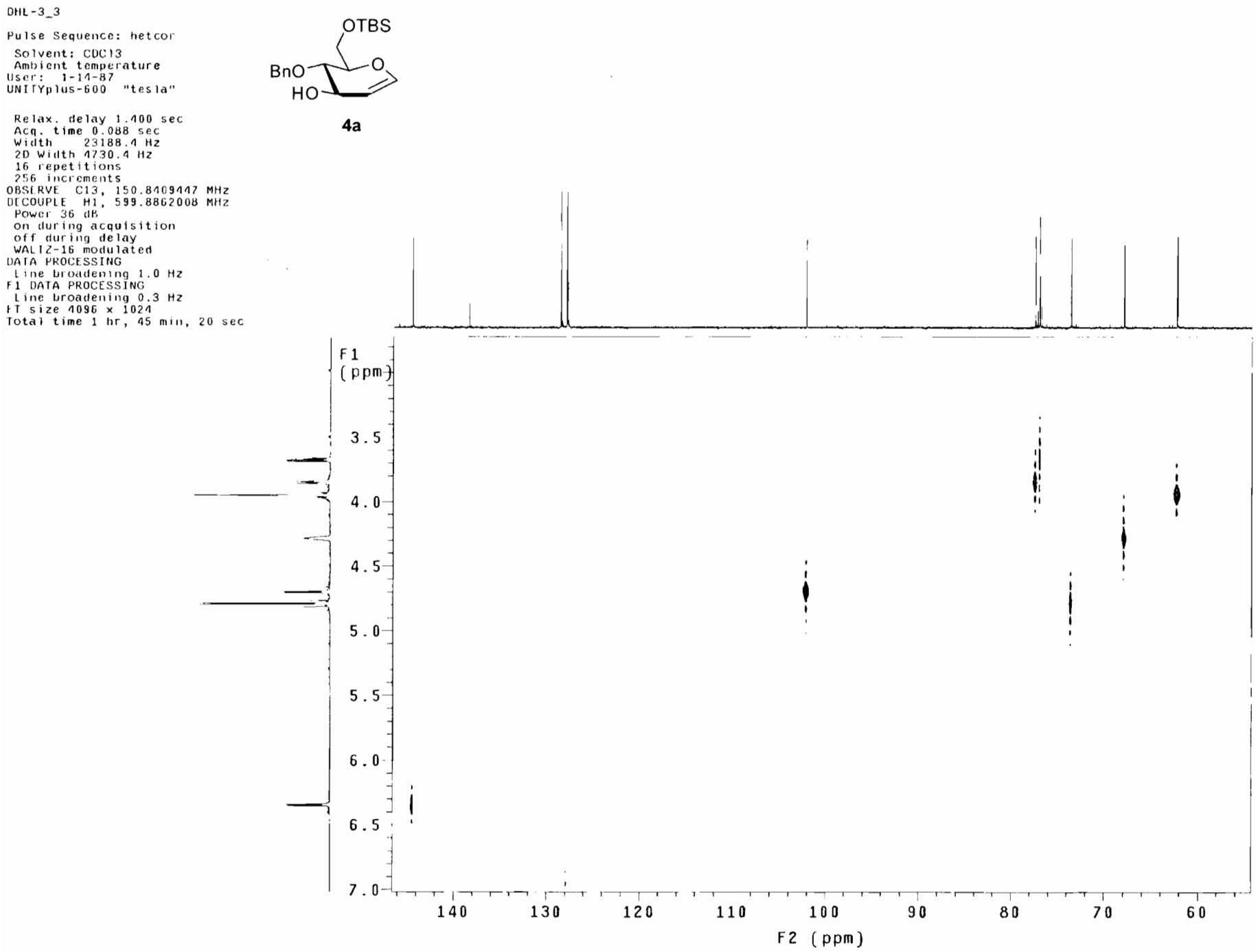
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Ambient temperature
User: 1-14-87
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Acq. time 0.088 sec
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2D Width 4730.4 Hz
16 repetitions
256 increments
OBSERVE C13, 150.8409447 MHz
DECOUPLE H1, 599.8862008 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 1 hr, 45 min, 20 sec
OTBS
BnO
O
HO
4a
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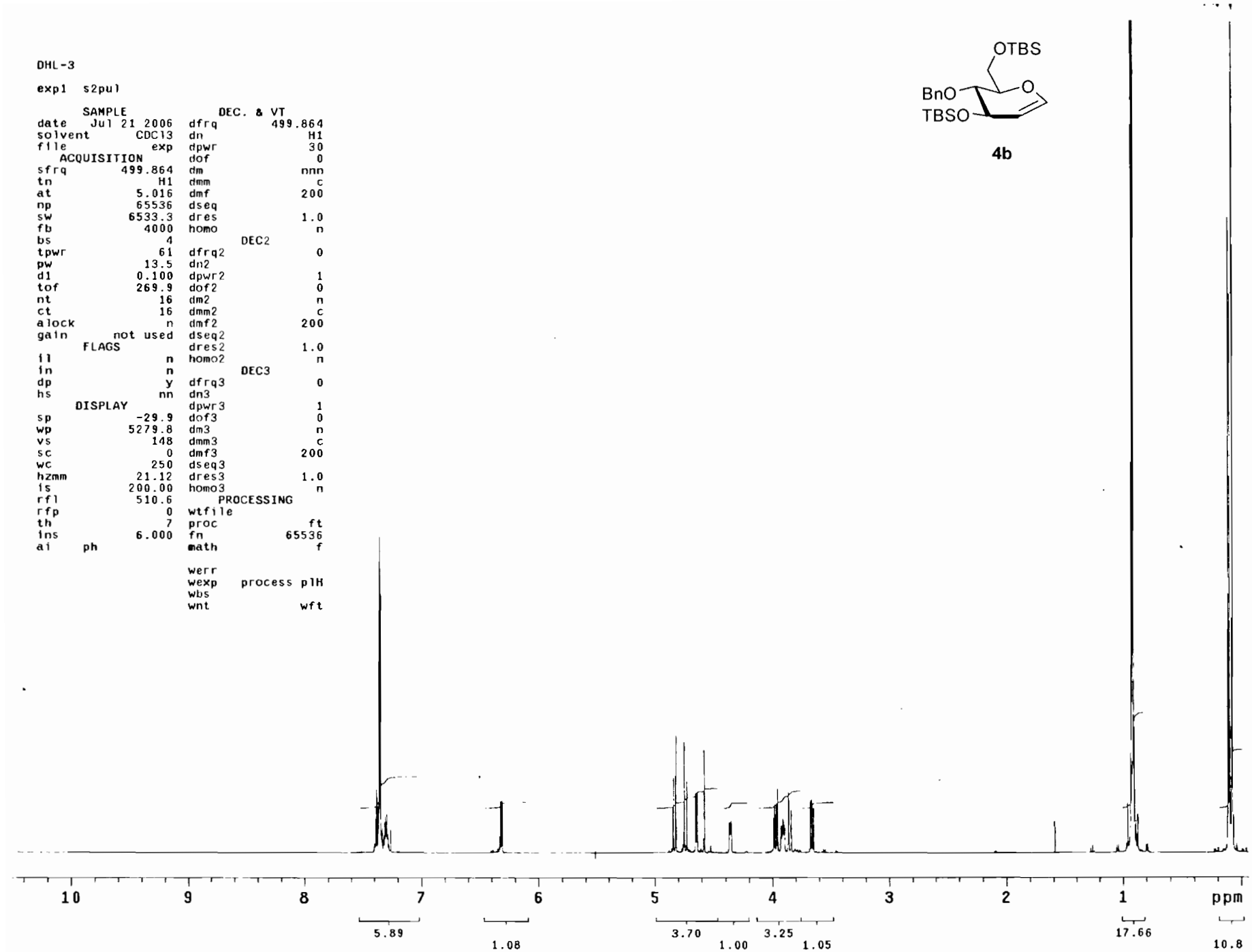
F2 (ppm)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dres2 1.0
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DEC3
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dpwr3 1
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dm3 n
dmm3 c
dmf3 200
dseq3
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homo3 n
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math f
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wexp process plH
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wnt wft
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BnO
TBSO
O
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7
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4
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2
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ppm
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10.8

DHL-3

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 Ambient temperature
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 Acq. time 0.130 sec
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 2D Width 3923.5 Hz
 4 repetitions
 256 increments
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 Sine bell 0.033 sec
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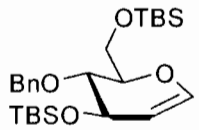
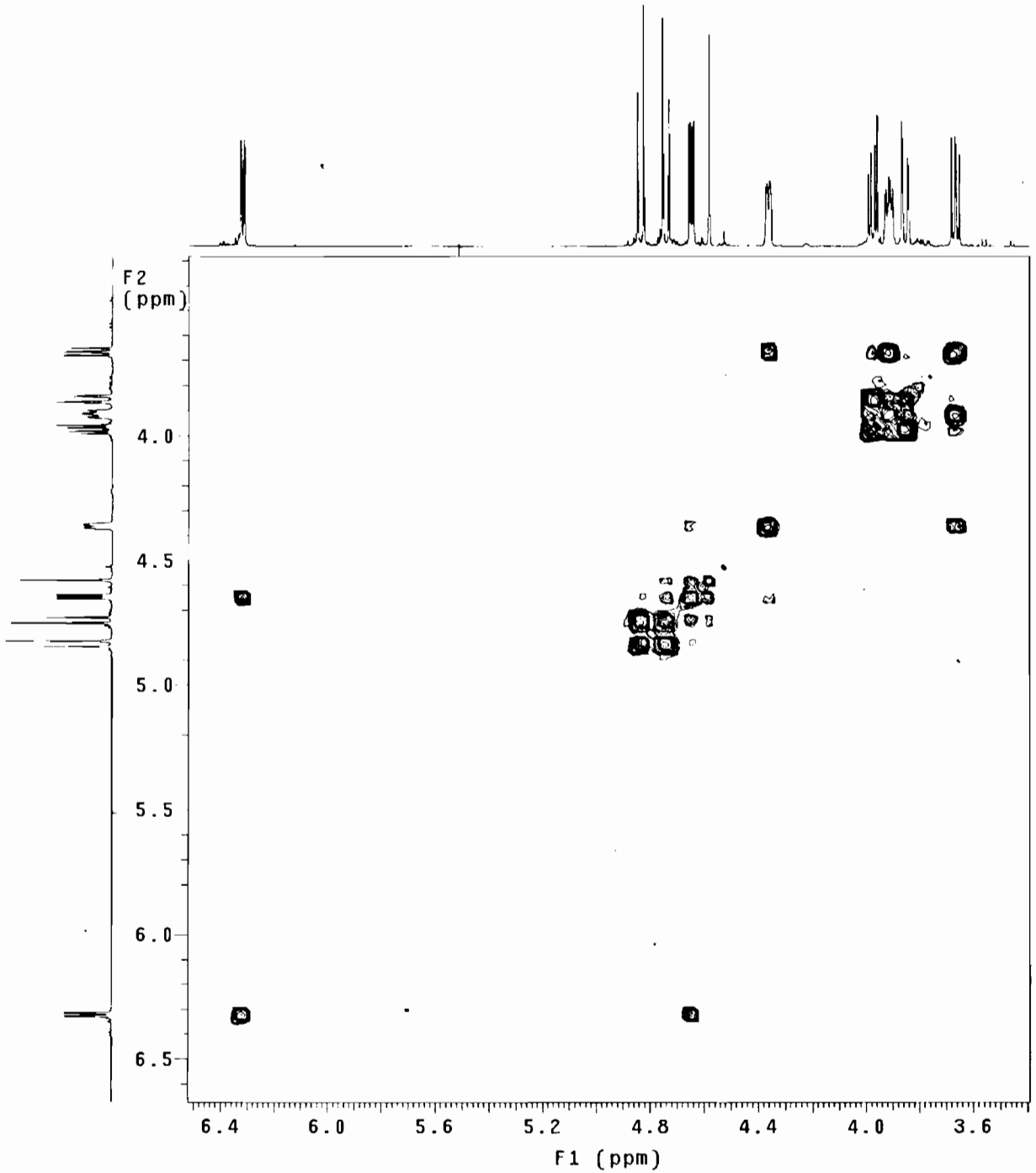
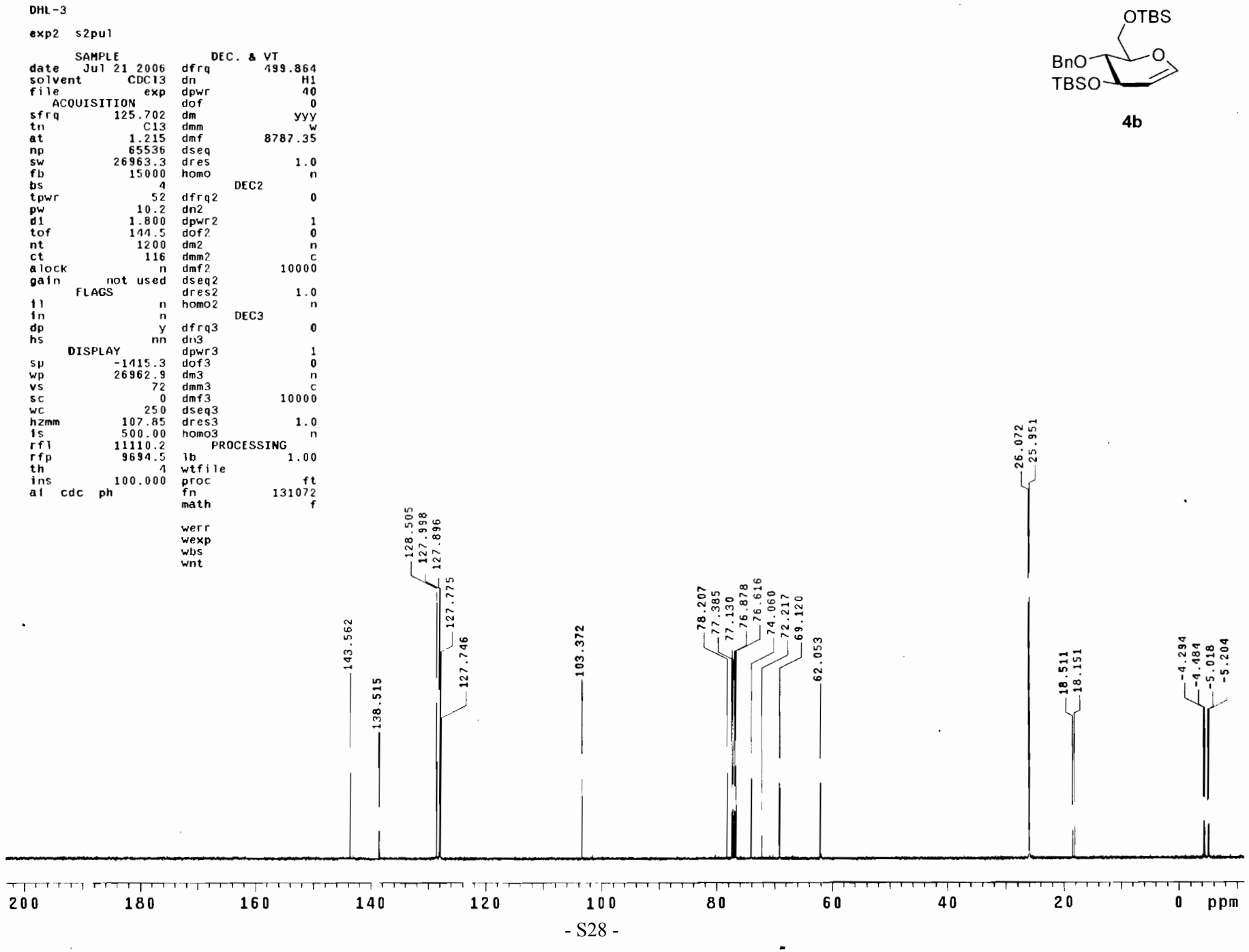
Total time 25 min, 20 sec



4b

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dpwr3 1
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TBSO
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77.130
76.878
76.616
74.060
72.217
69.120
62.053
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20
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ppm

DHL-3

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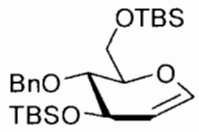

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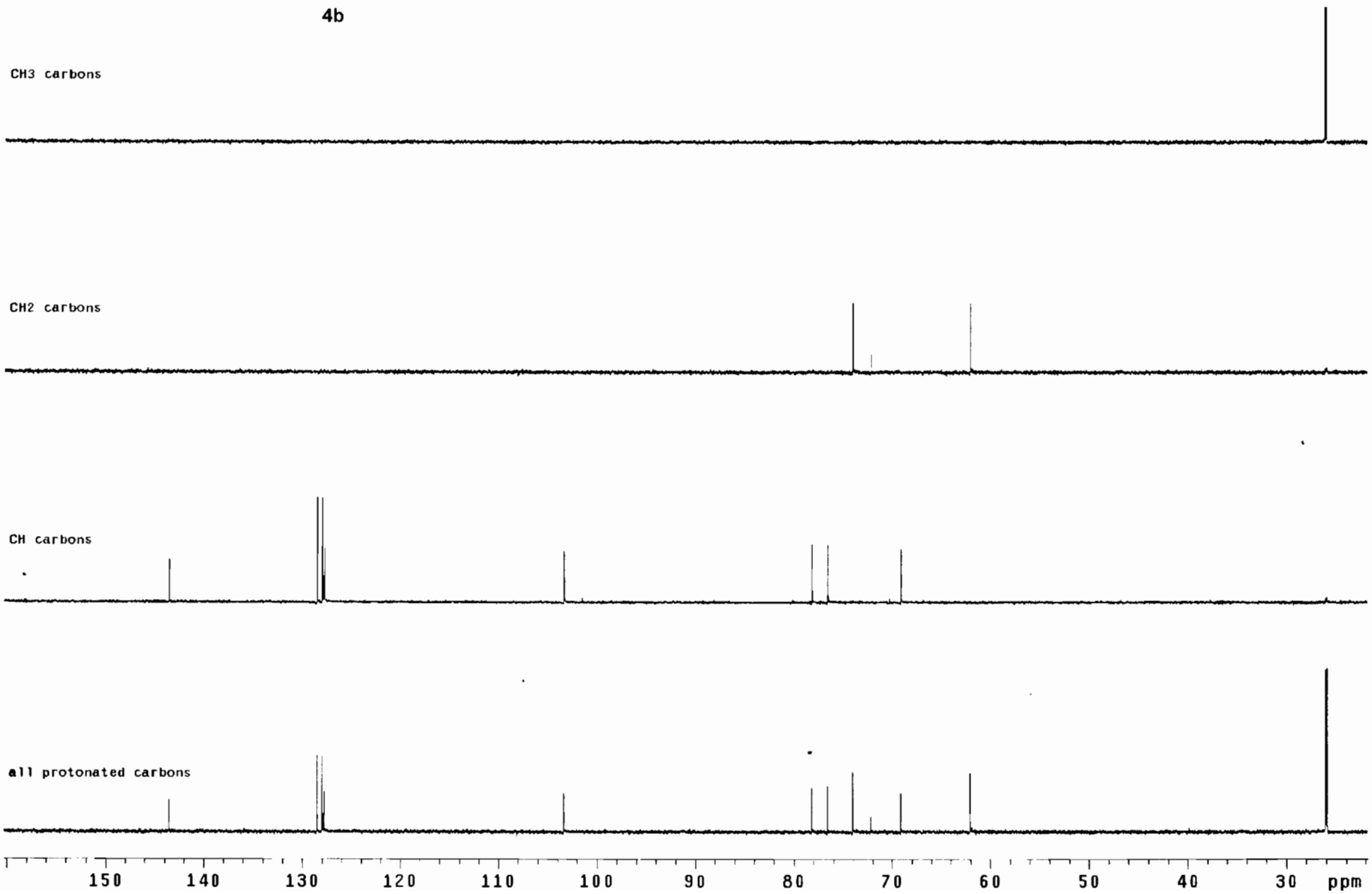

DHL-3

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Ambient temperature
User: 1-14-87
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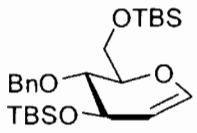
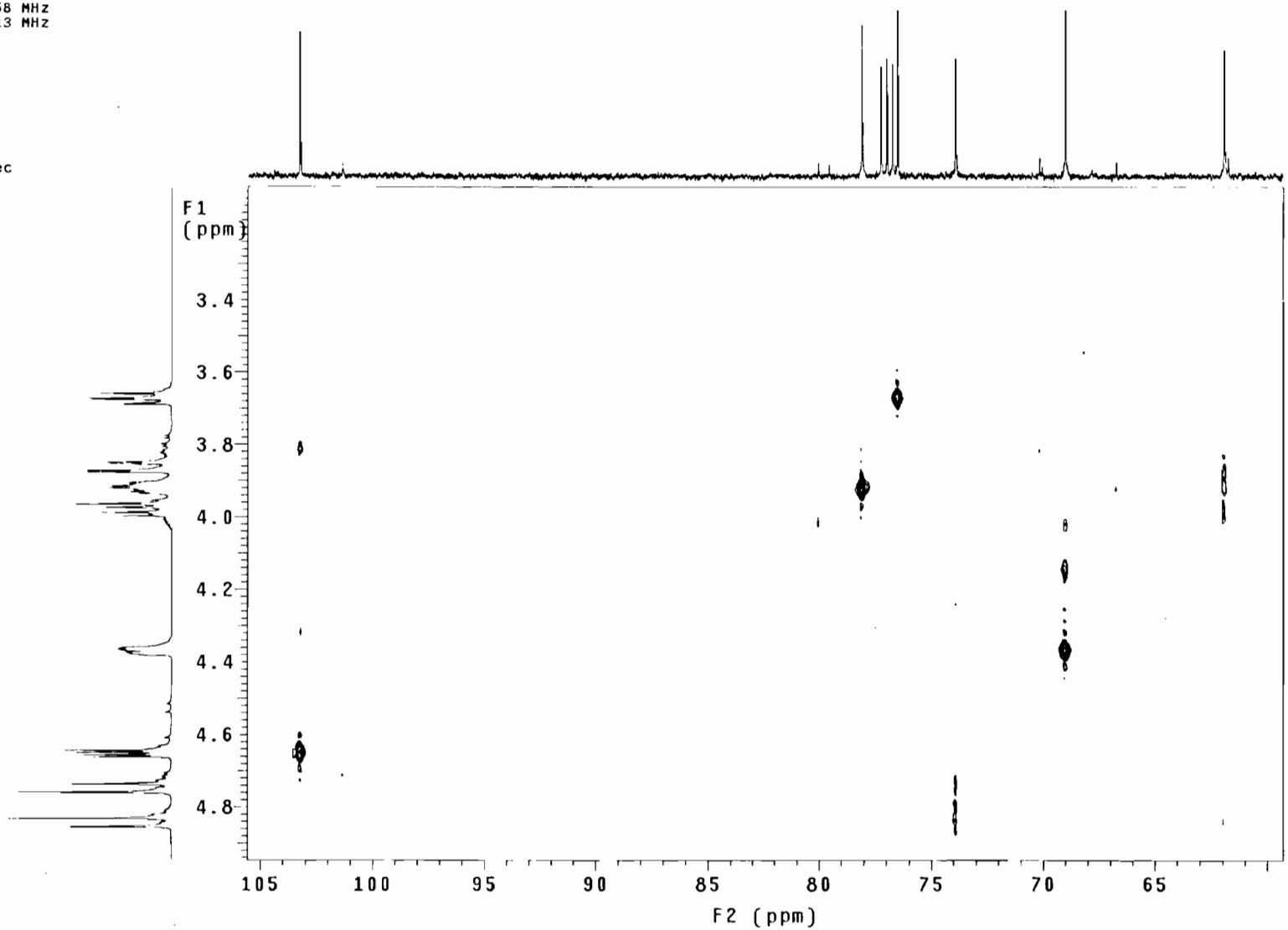
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 4026.4 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6901958 MHz
DECOUPLE H1, 499.8630813 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 28 min, 33 sec



4b



DHL-7

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tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	909.7	dof3	0
wp	3966.5	dm3	n
vs	158	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.87	dres3	1.0
is	200.00	homo3	n
rfl	463.5	PROCESSING	
rfp	0	wtfile	
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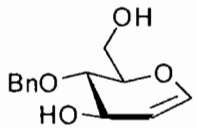
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



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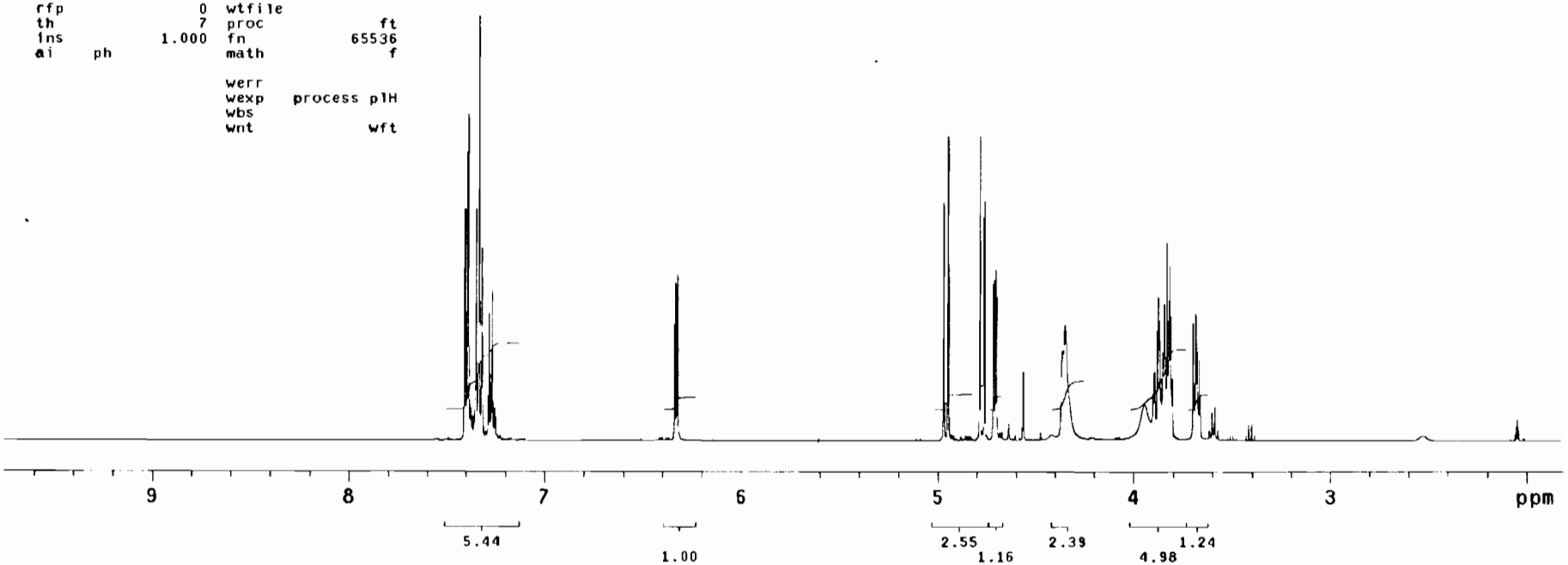

DHL-7

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Solvent: CD3CN
Ambient temperature
INOVA-500 "inova5"

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COSY 90-90
Acq. time 0.236 sec
Width 2168.3 Hz
2D Width 2168.3 Hz
4 repetitions
128 increments
OBSERVE H1, 499.8637722 MHz
DATA PROCESSING
Sine bell 0.118 sec
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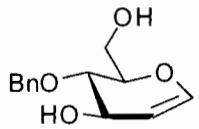
Sine bell 0.059 sec
FT size 1024 x 1024
Total time 13 min, 39 sec



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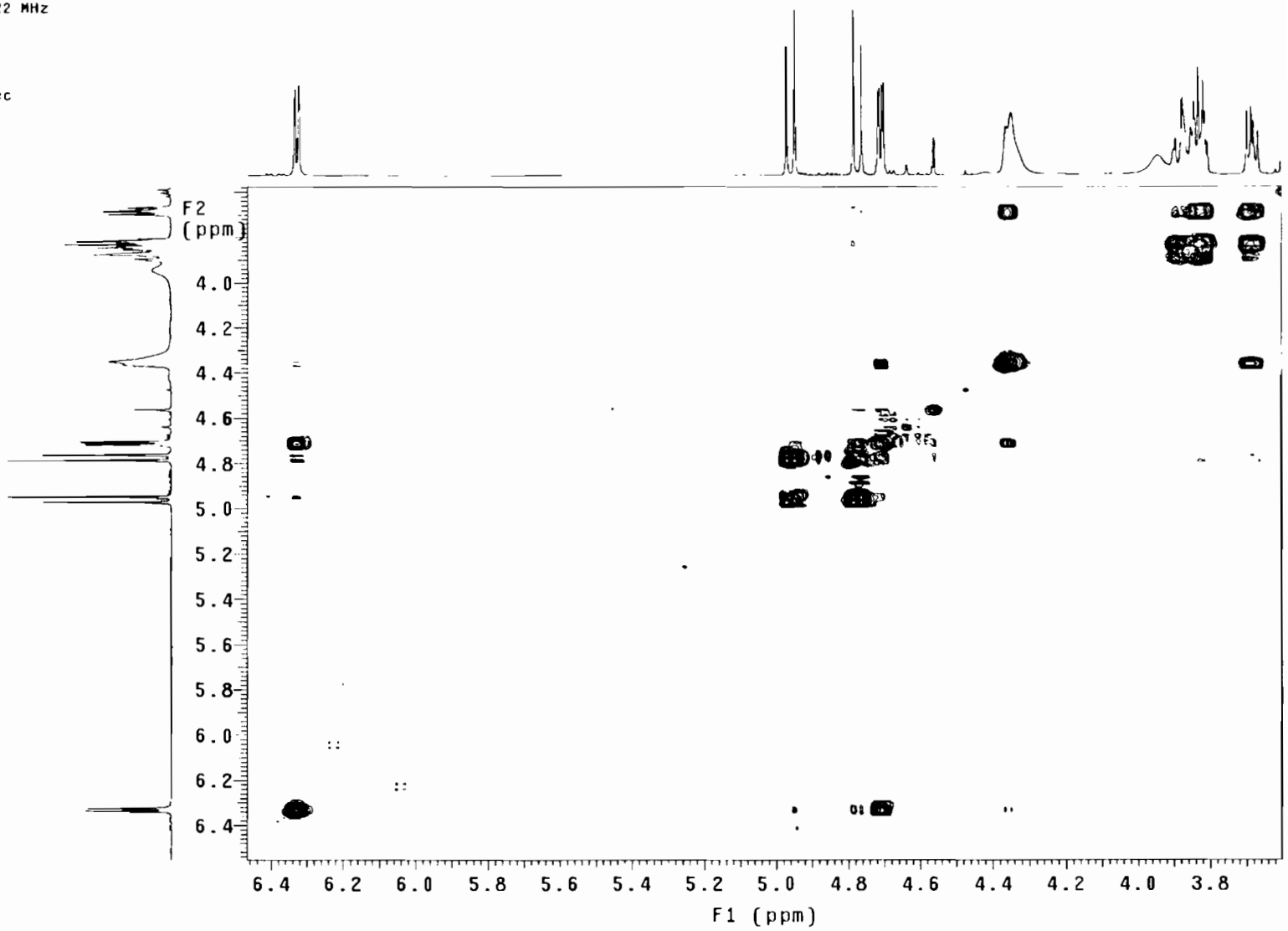

DHL-7

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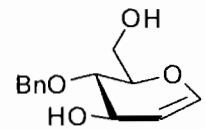
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1200	dm2	n
ct	44	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
fl	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	3378.4	dof3	0
wp	21683.8	dm3	n
vs	27	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	86.74	dres3	1.0
fs	500.00	homo3	n
rfl	5091.8	PROCESSING	
rfp	3758.2	lb	1.00
th	8	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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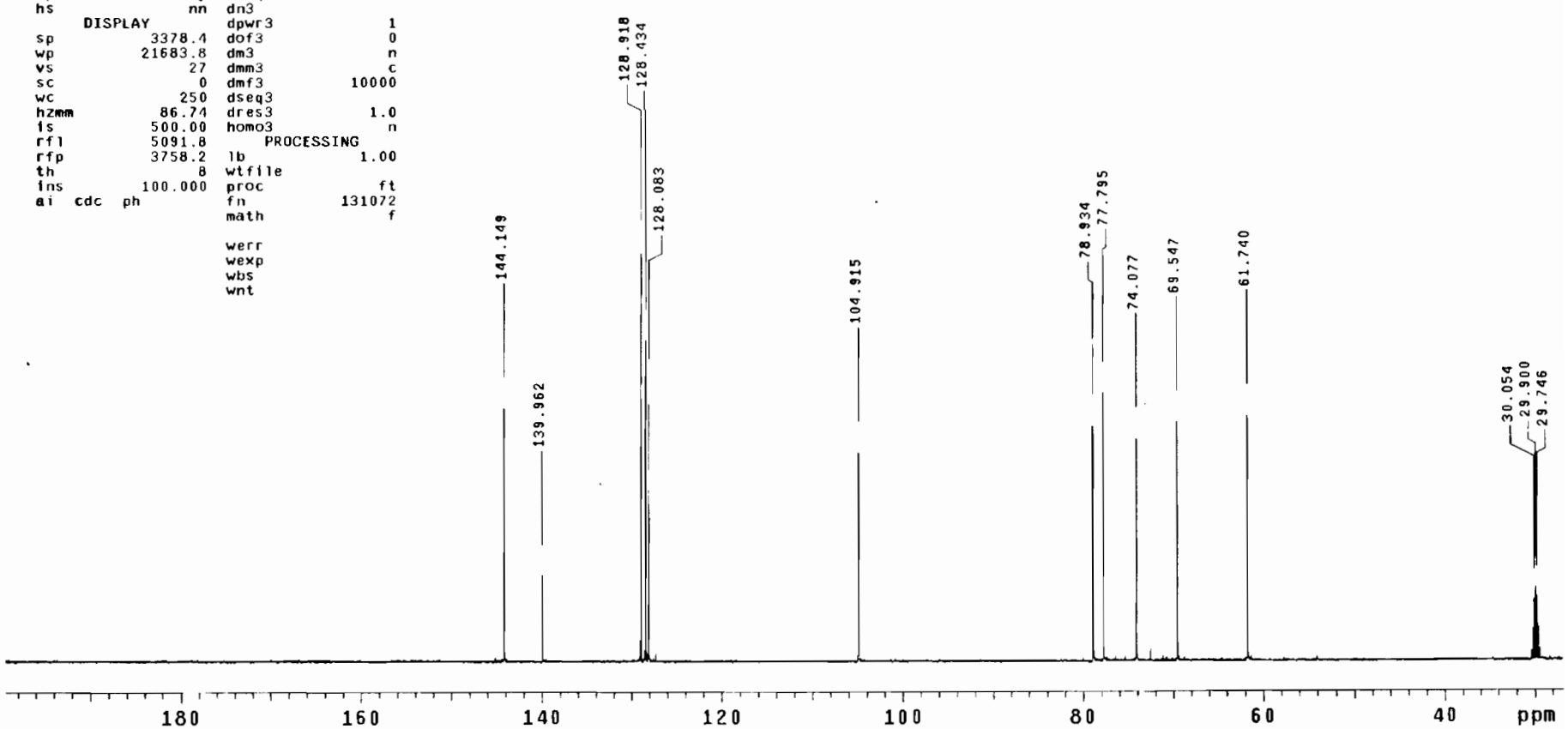

DHL-7

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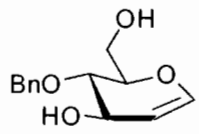

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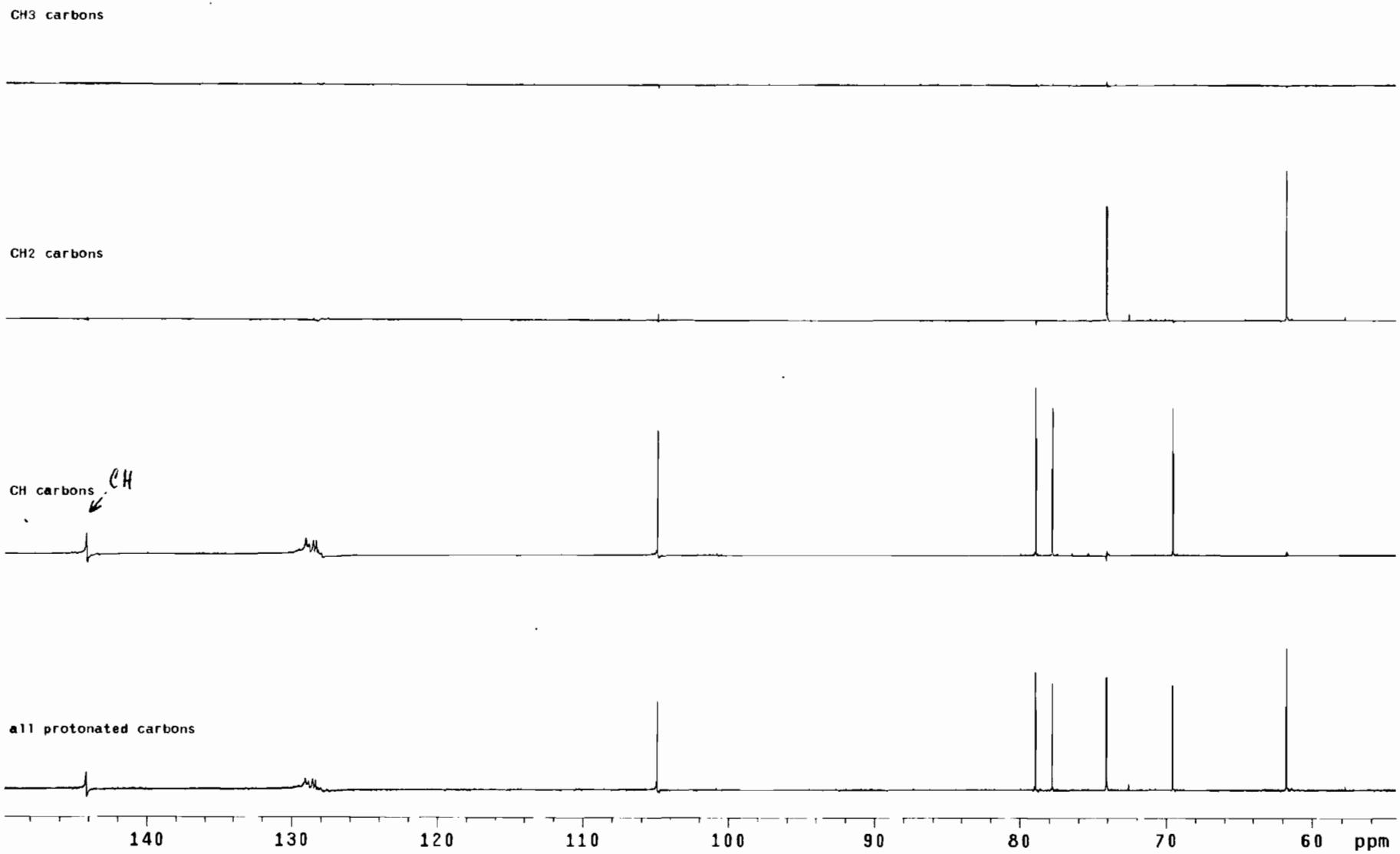

DHL-7

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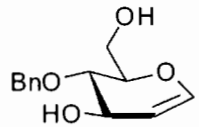
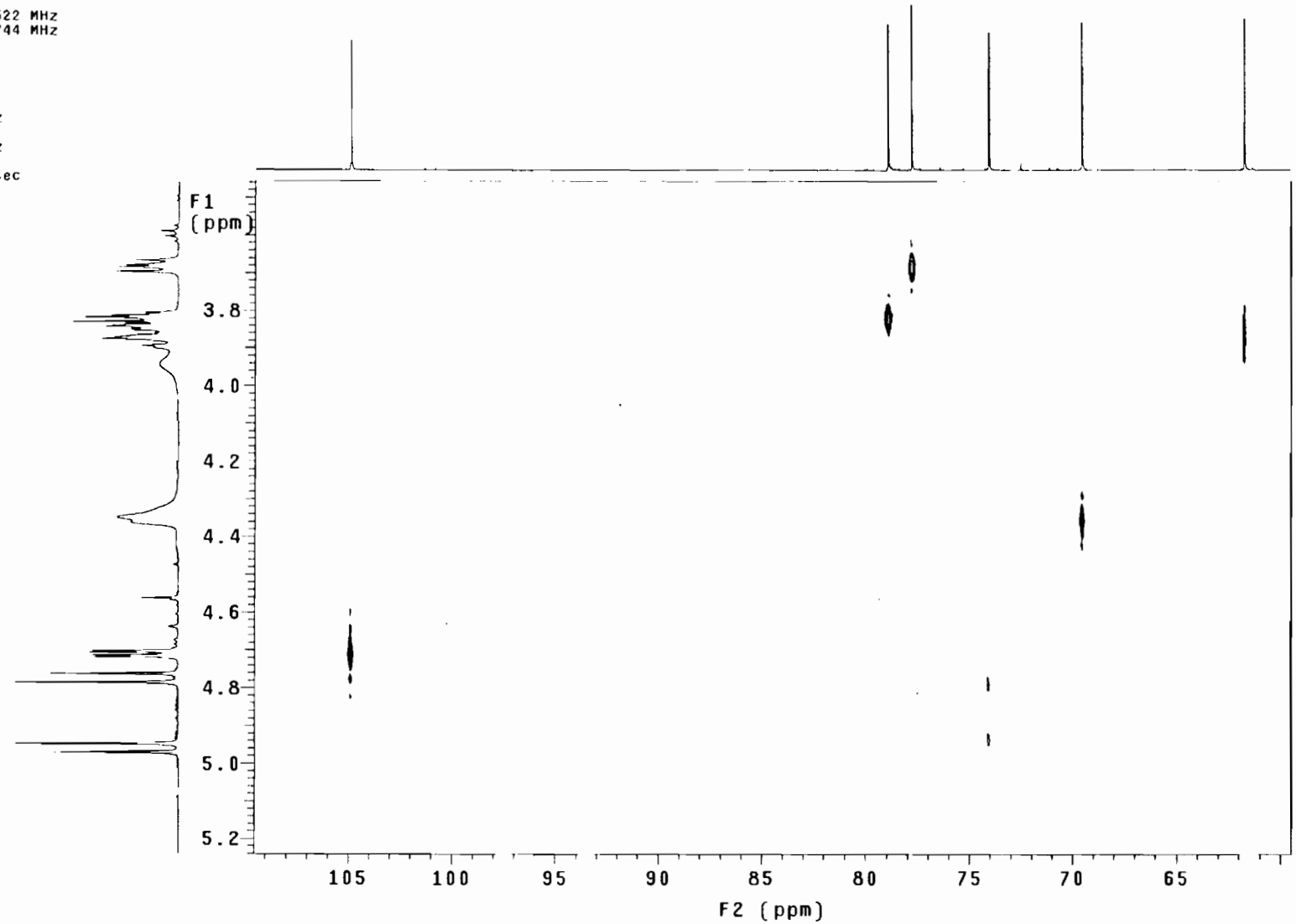
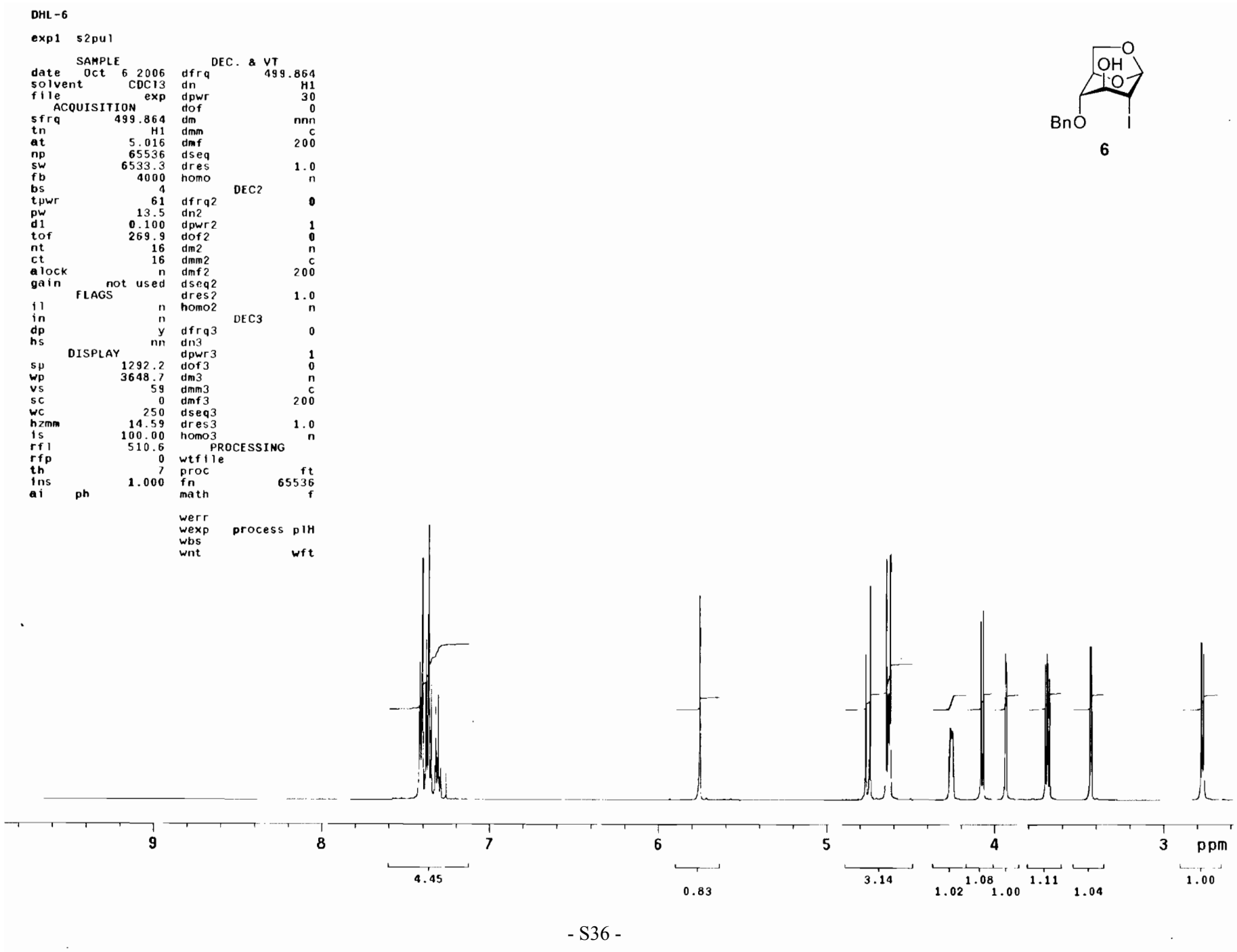
Ambient temperature
User: 1-14-87
File: DHL-7-HC
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2131.7 Hz
4 repetitions
128 increments
OBSERVE C13, 125.6907522 MHz
DECOUPLE H1, 499.8664744 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 14 min, 19 sec



5

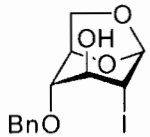



DHL-6
exp1 s2pul
SAMPLE
date Oct 6 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1292.2
wp 3648.7
vs 59
sc 0
wc 250
hzmm 14.59
is 100.00
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
O
OH
O
BnO
I
6
9
8
7
6
5
4
3
ppm
4.45
0.83
3.14
1.02
1.08
1.00
1.11
1.04
1.00

DHL-6

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.203 sec
Width 2526.4 Hz
2D Width 2526.4 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.101 sec
F1 DATA PROCESSING
Sine bell 0.051 sec
FT size 1024 x 1024
Total time 53 min, 34 sec



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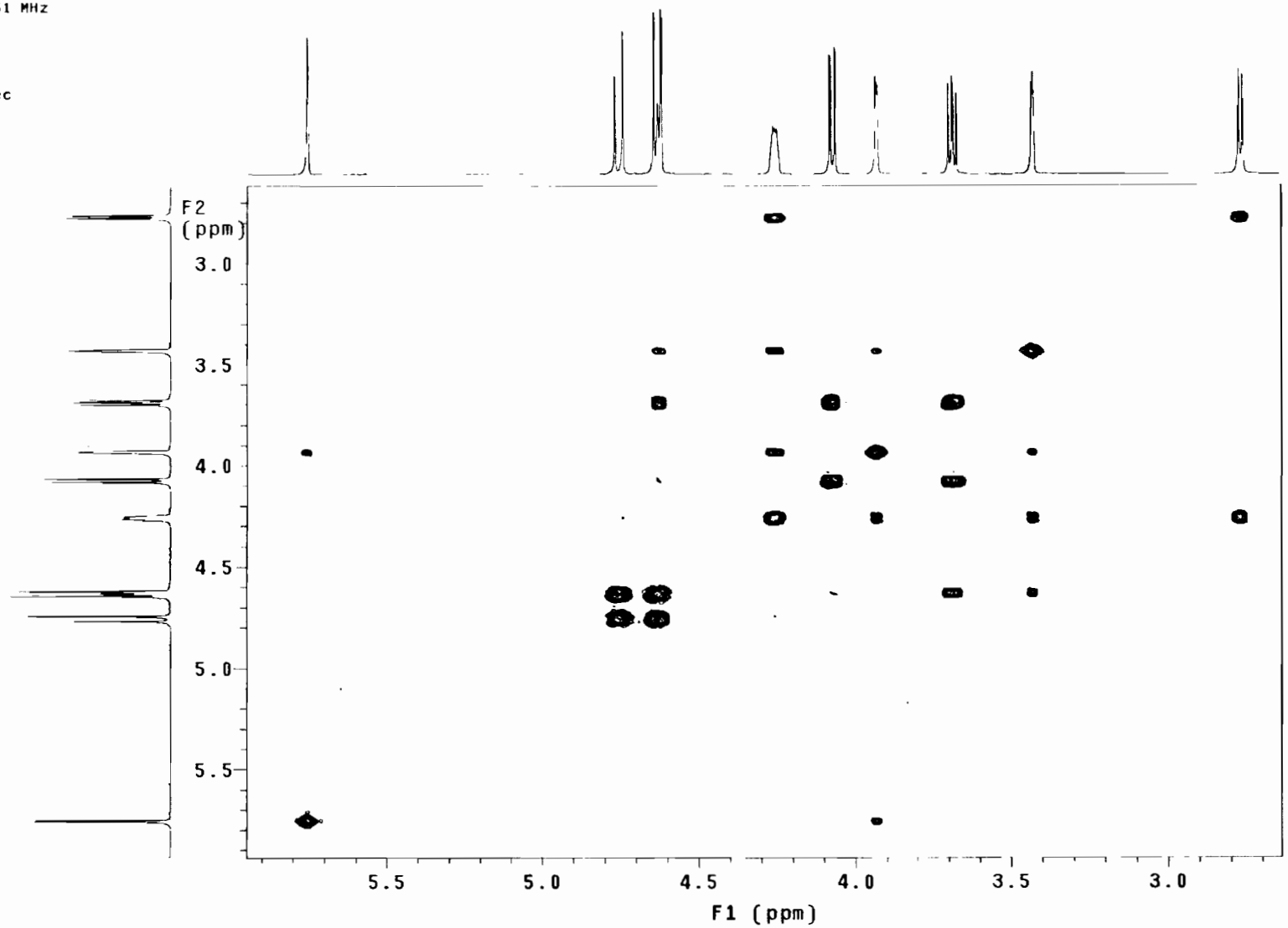
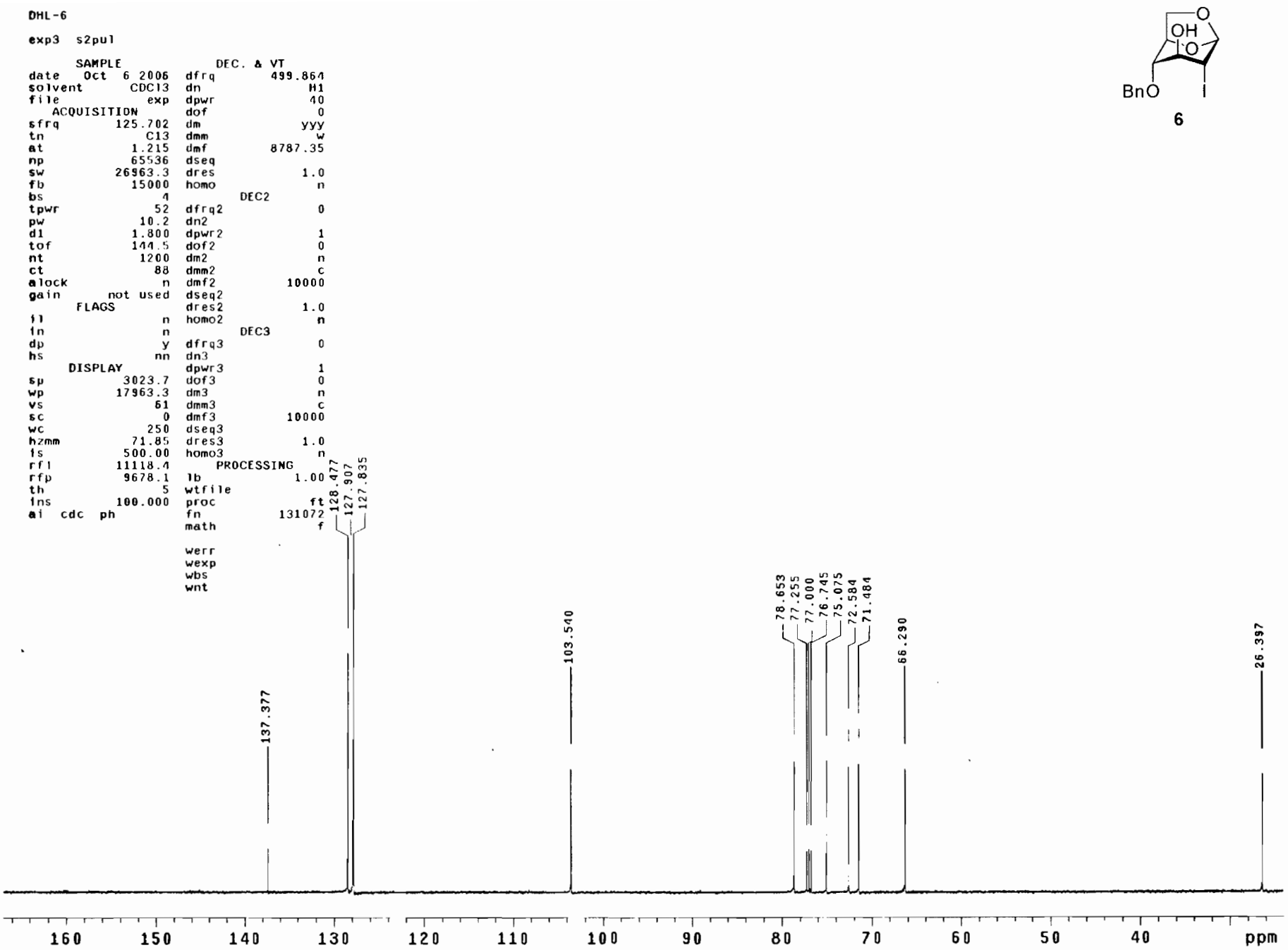
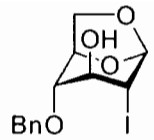


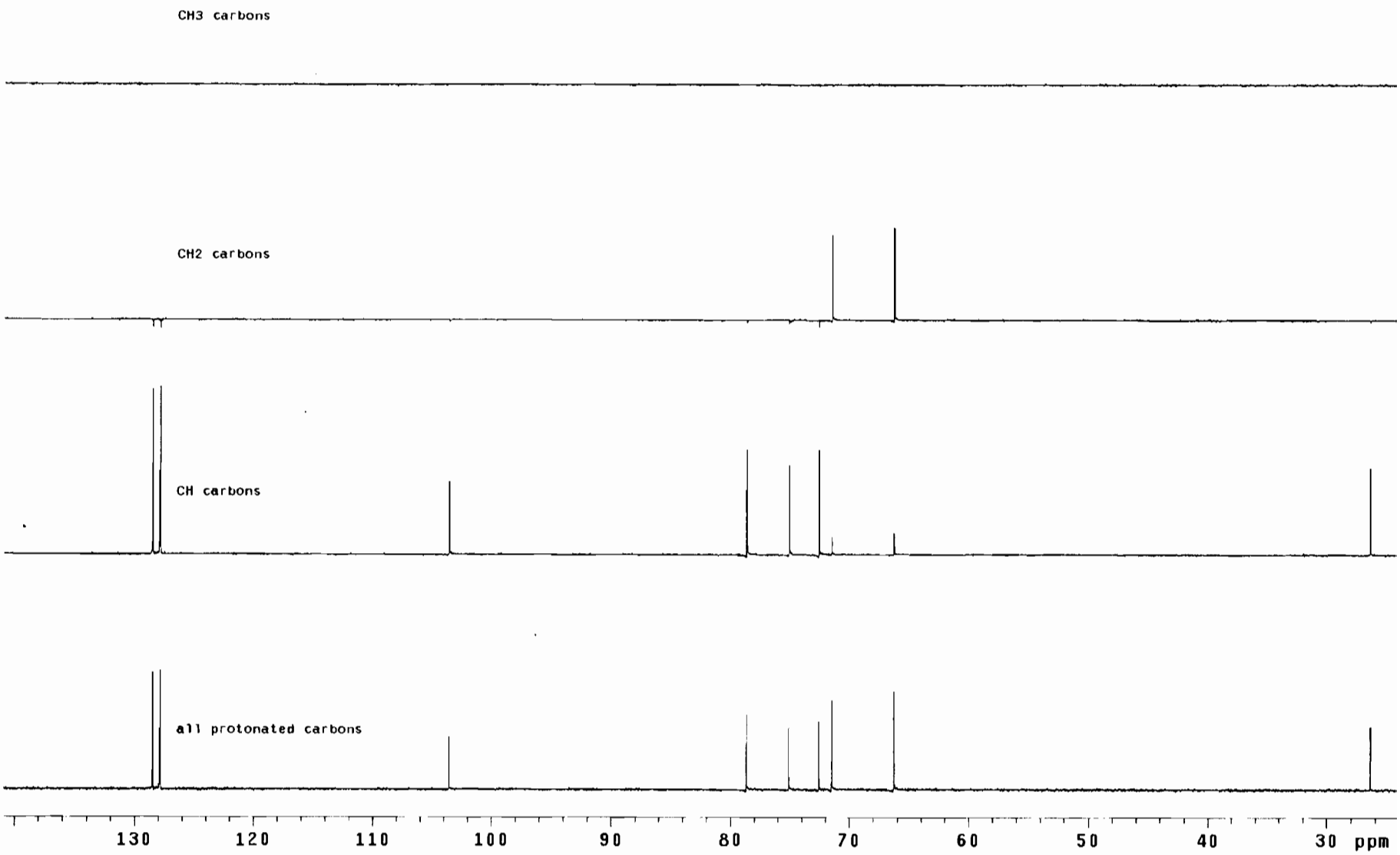

DHL-6
exp3 s2pul
SAMPLE
date Oct 6 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 88
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 3023.7
wp 17963.3
vs 61
sc 0
wc 250
hzmm 71.85
is 500.00
rfl 11118.4
rfp 9678.1
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
128.477
127.907
127.835
137.377
103.540
78.653
77.255
77.000
76.745
75.075
72.584
71.484
66.290
26.397
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm
OH
O
O
BnO
I
6

DHL-6

Pulse Sequence: dept



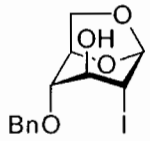
6



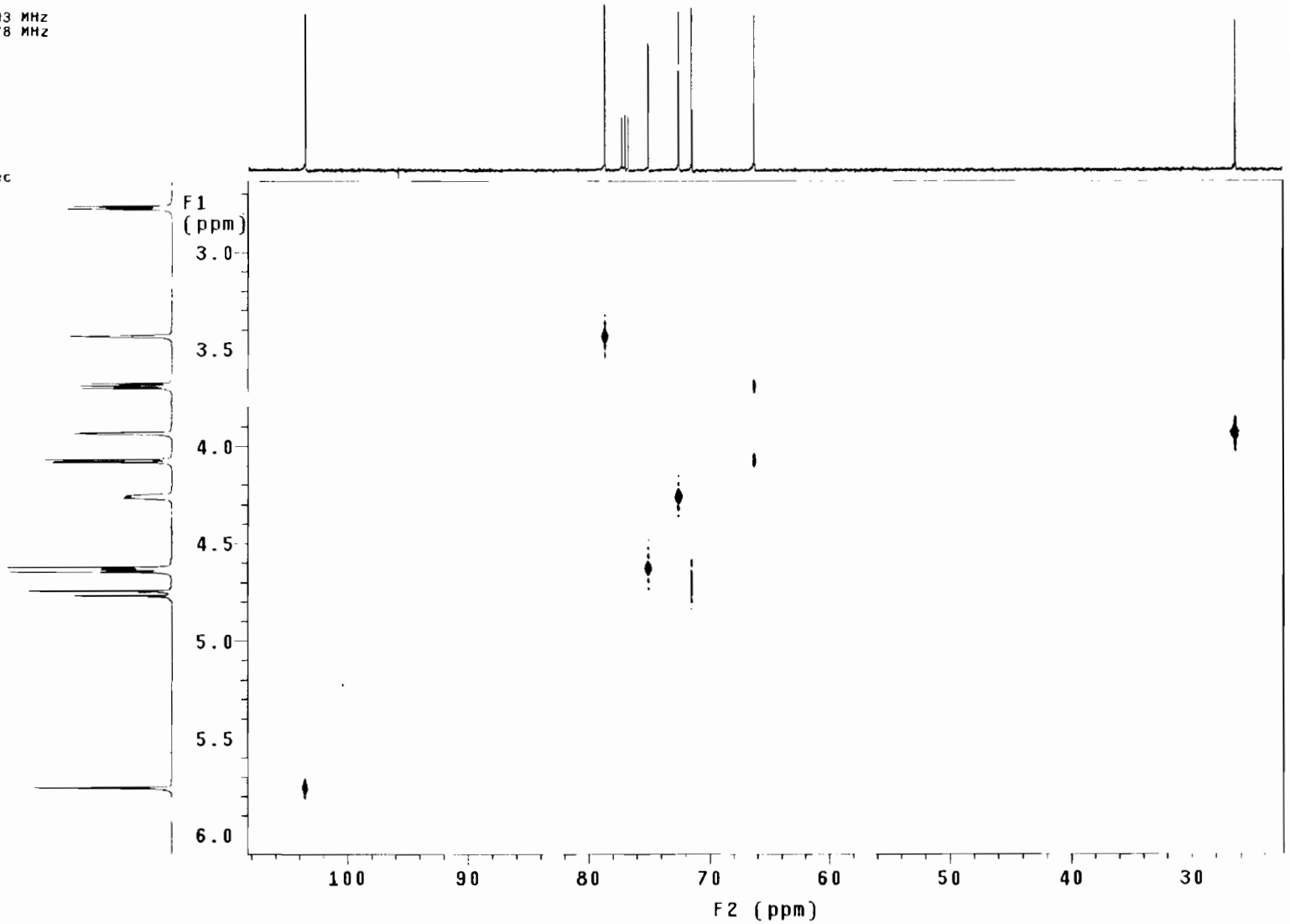
DHL-6

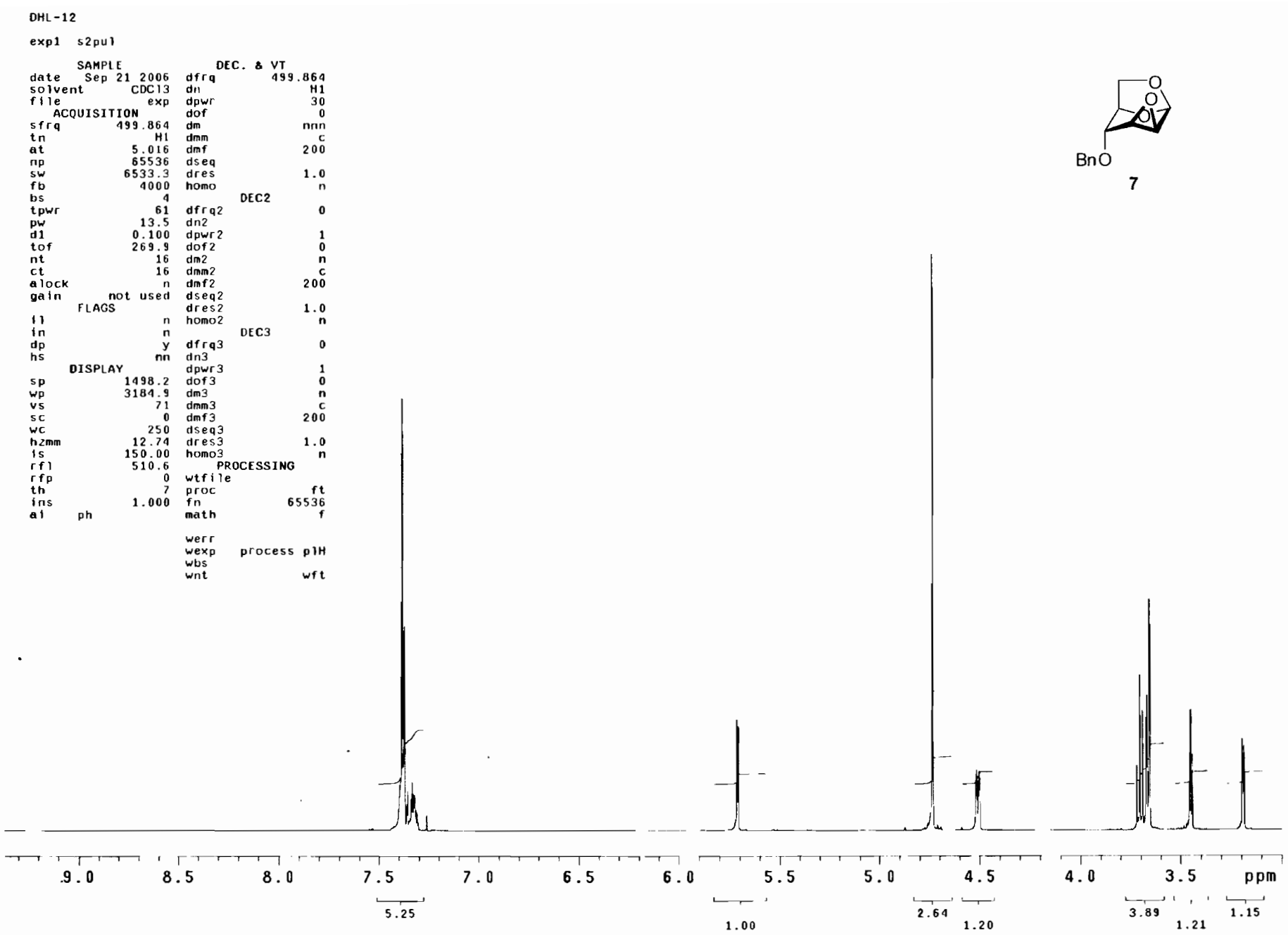
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
File: DHL-6-HC
INOVA-500 "inova5"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width 18403.5 Hz
 2D Width 2651.4 Hz
 8 repetitions
 128 increments
OBSERVE C13, 125.6902043 MHz
DECOUPLE H1, 499.8637978 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
F1 DATA PROCESSING
 Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 25 sec



6

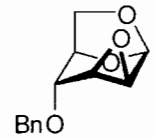



DHL-12
exp1 s2pul
SAMPLE
date Sep 21 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1498.2
wp 3184.9
vs 71
sc 0
wc 250
hzmm 12.74
is 150.00
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
O
O
O
BnO
7
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
ppm
5.25
1.00
2.64
1.20
3.89
1.21
1.15

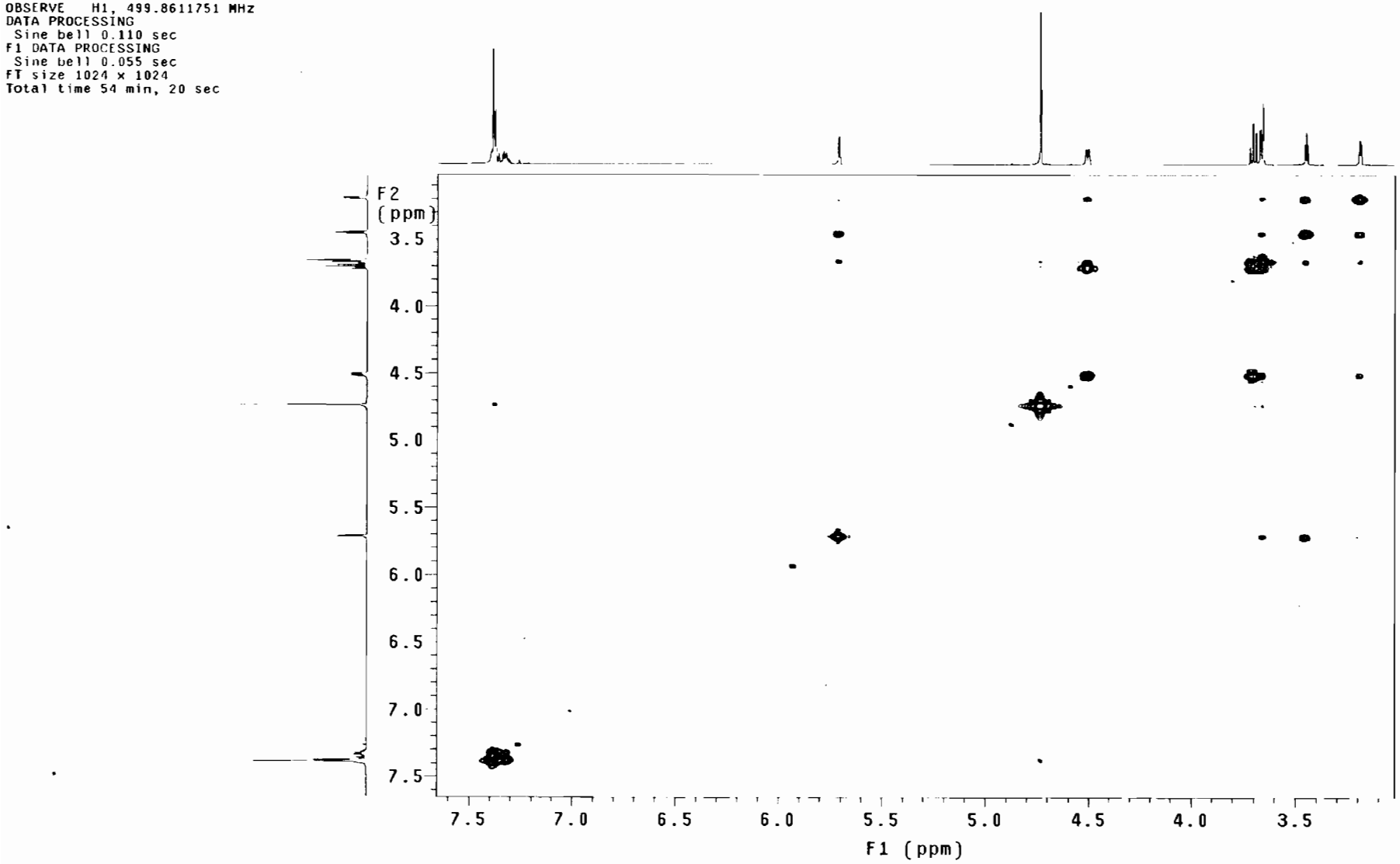
DHL-12

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.221 sec
Width 2317.8 Hz
2D Width 2317.8 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.110 sec
F1 DATA PROCESSING
Sine bell 0.055 sec
FT size 1024 x 1024
Total time 54 min, 20 sec



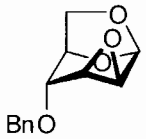
7



DHL-12

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.221 sec
Width 2317.8 Hz
2D Width 2317.8 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.110 sec
F1 DATA PROCESSING
Sine bell 0.055 sec
FT size 1024 x 1024
Total time 54 min, 20 sec



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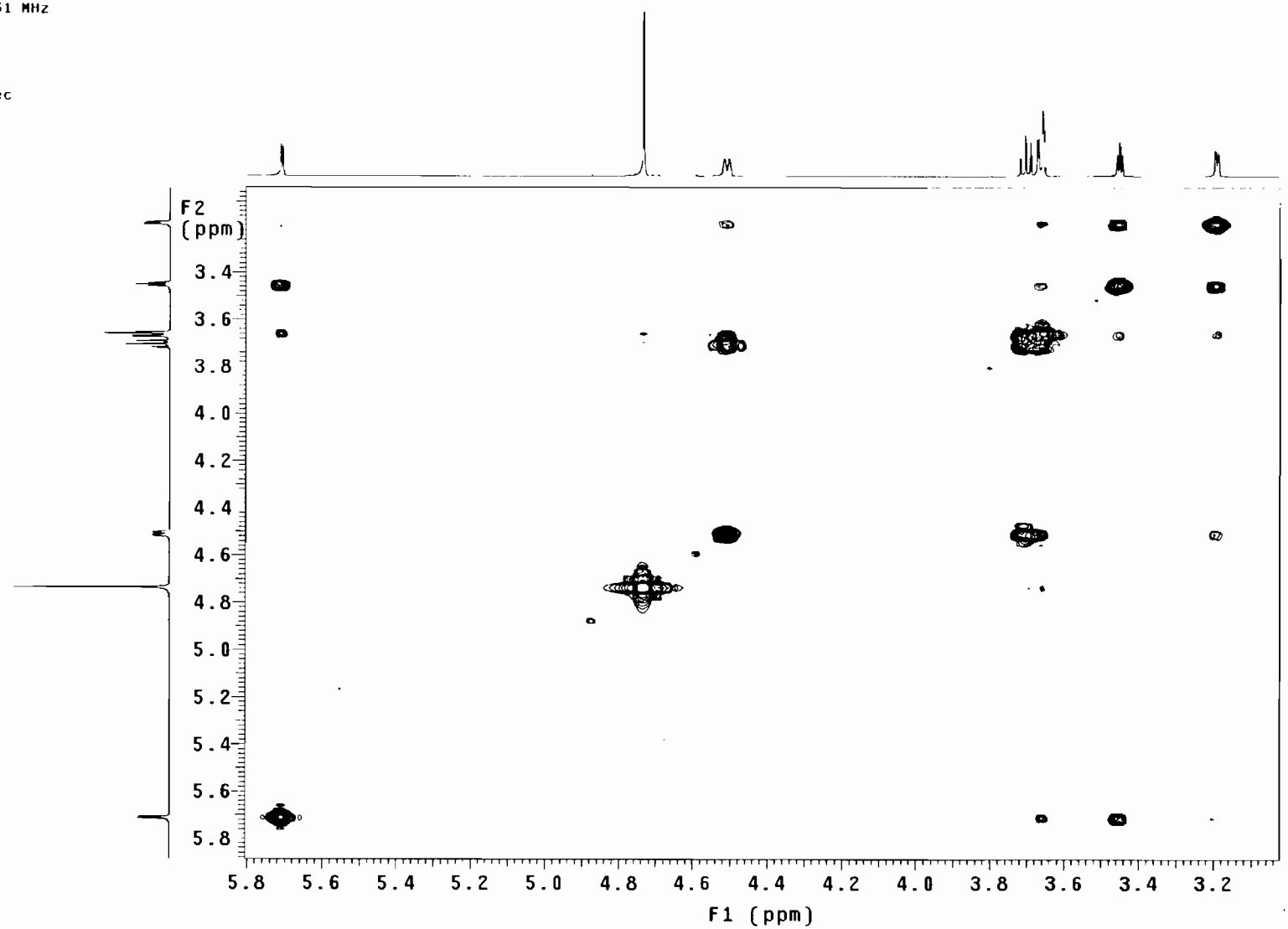
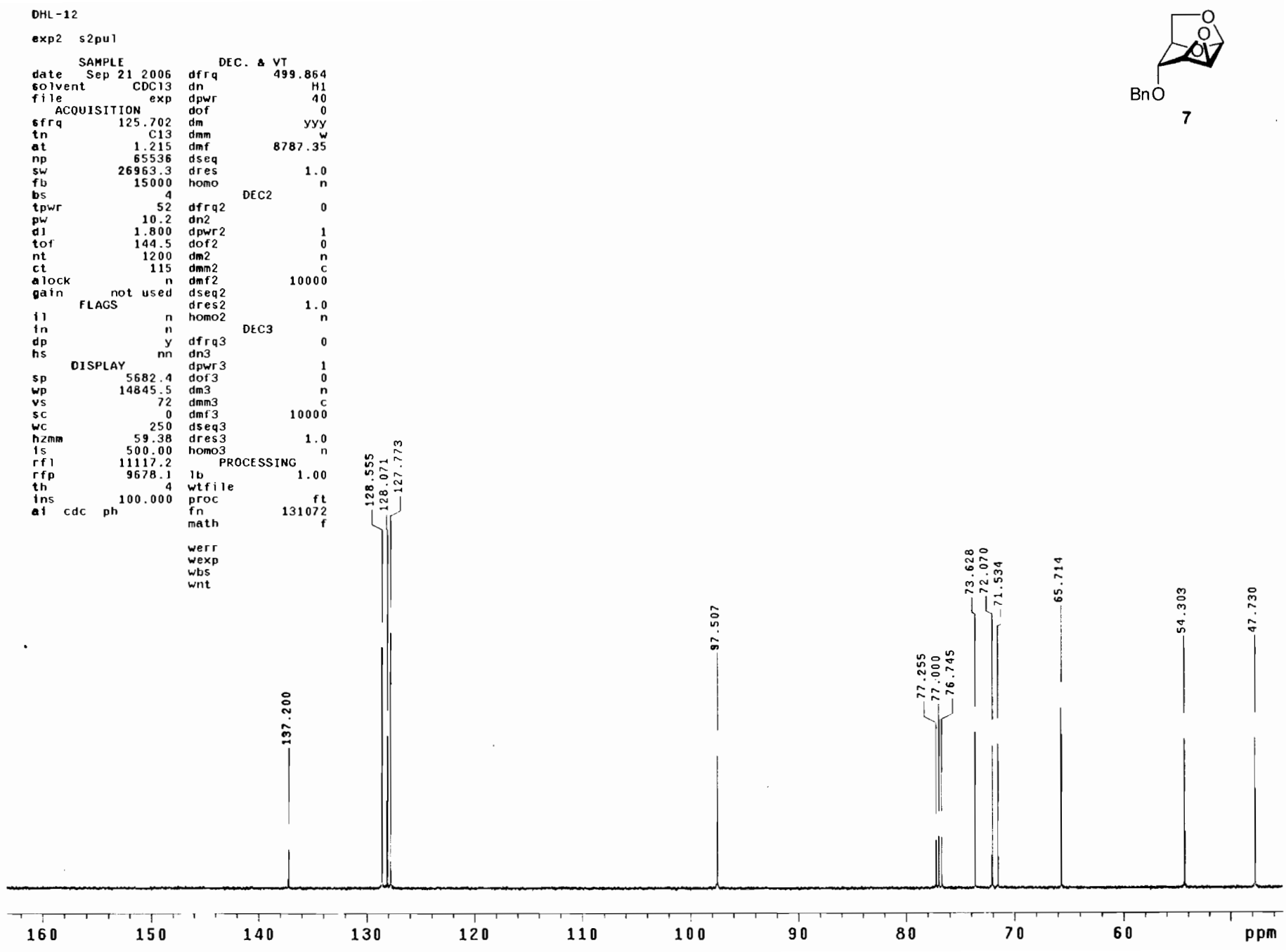
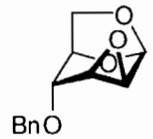



DHL-12
exp2 s2pul
SAMPLE
date Sep 21 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 115
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 5682.4
wp 14845.5
vs 72
sc 0
wc 250
hzmm 59.38
is 500.00
rfl 11117.2
rfp 9678.1
th 4
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
BnO
7
128.555
128.071
127.773
137.200
97.507
77.255
77.000
76.745
73.628
72.070
71.534
65.714
54.303
47.730
160
150
140
130
120
110
100
90
80
70
60
ppm

DHL-12

Pulse Sequence: dept



7

CH3 carbons

CH2 carbons

CH carbons

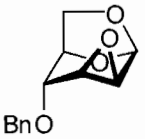
all protonated carbons

140 130 120 110 100 90 80 70 60 50 ppm

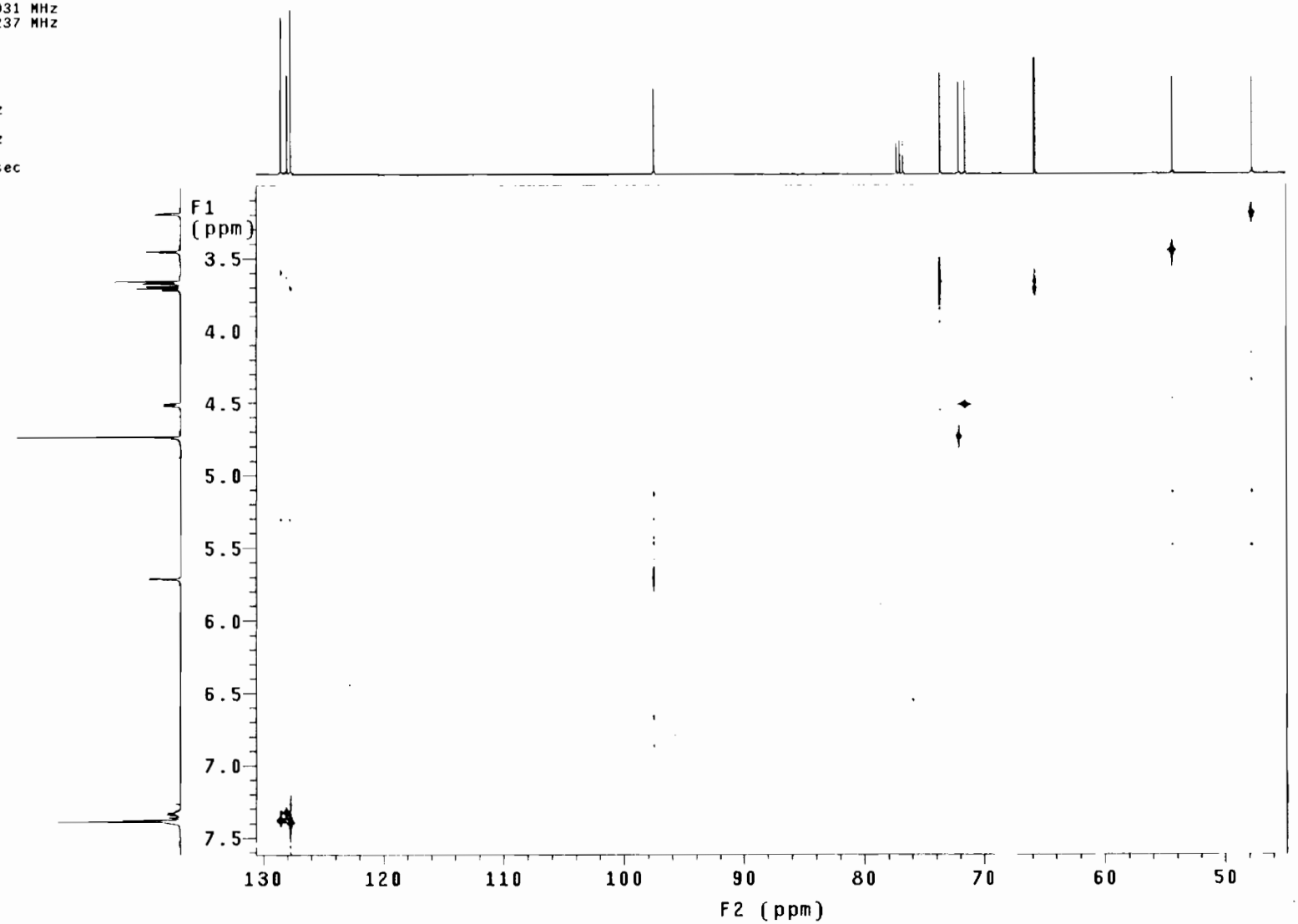
DHL-12

Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2352.2 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902031 MHz
DECOUPLE H1, 499.8638237 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 56 sec



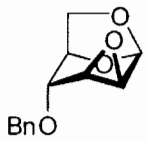
7



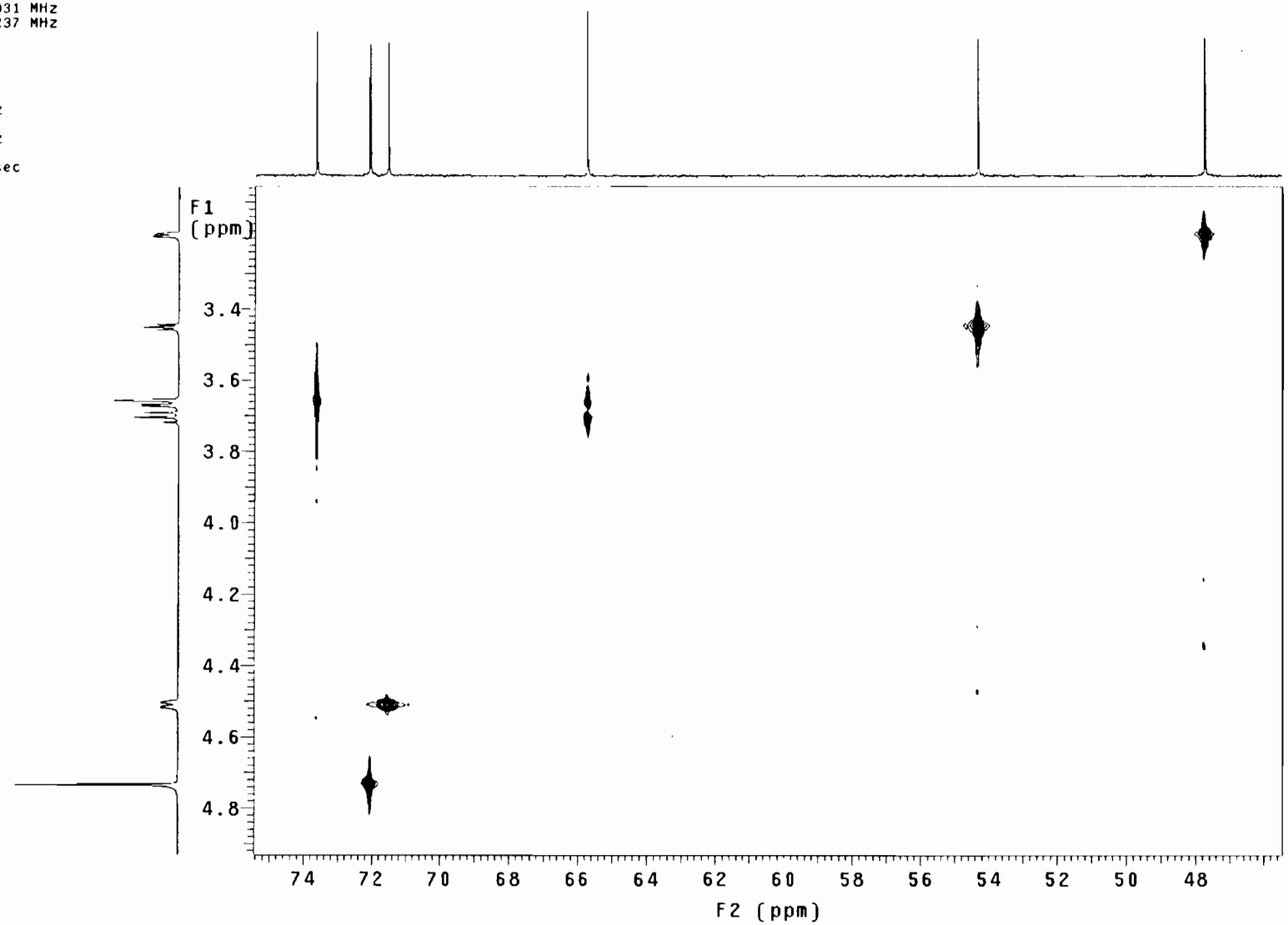
DHL-12

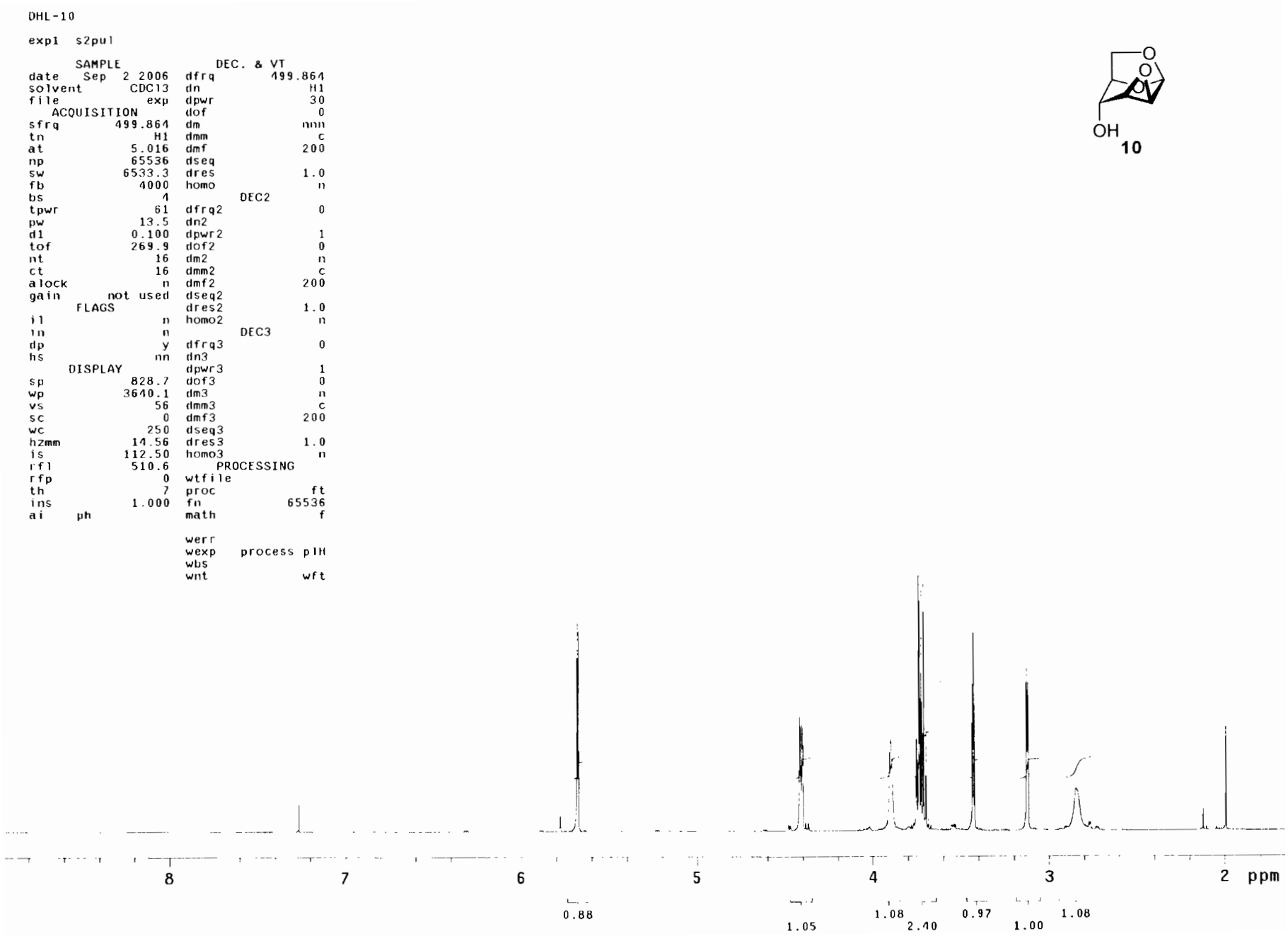
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2352.2 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902031 MHz
DECOUPLE H1, 499.8638237 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 56 sec



7



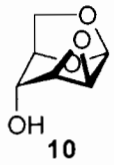

DHL-10
exp1 s2pul
SAMPLE
date Sep 2 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 828.7
wp 3640.1
vs 56
sc 0
wc 250
hzmm 14.56
is 112.50
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
OH
10
8
7
6
5
4
3
2 ppm
0.88
1.05
1.08
2.40
0.97
1.00
1.08

DHL-10

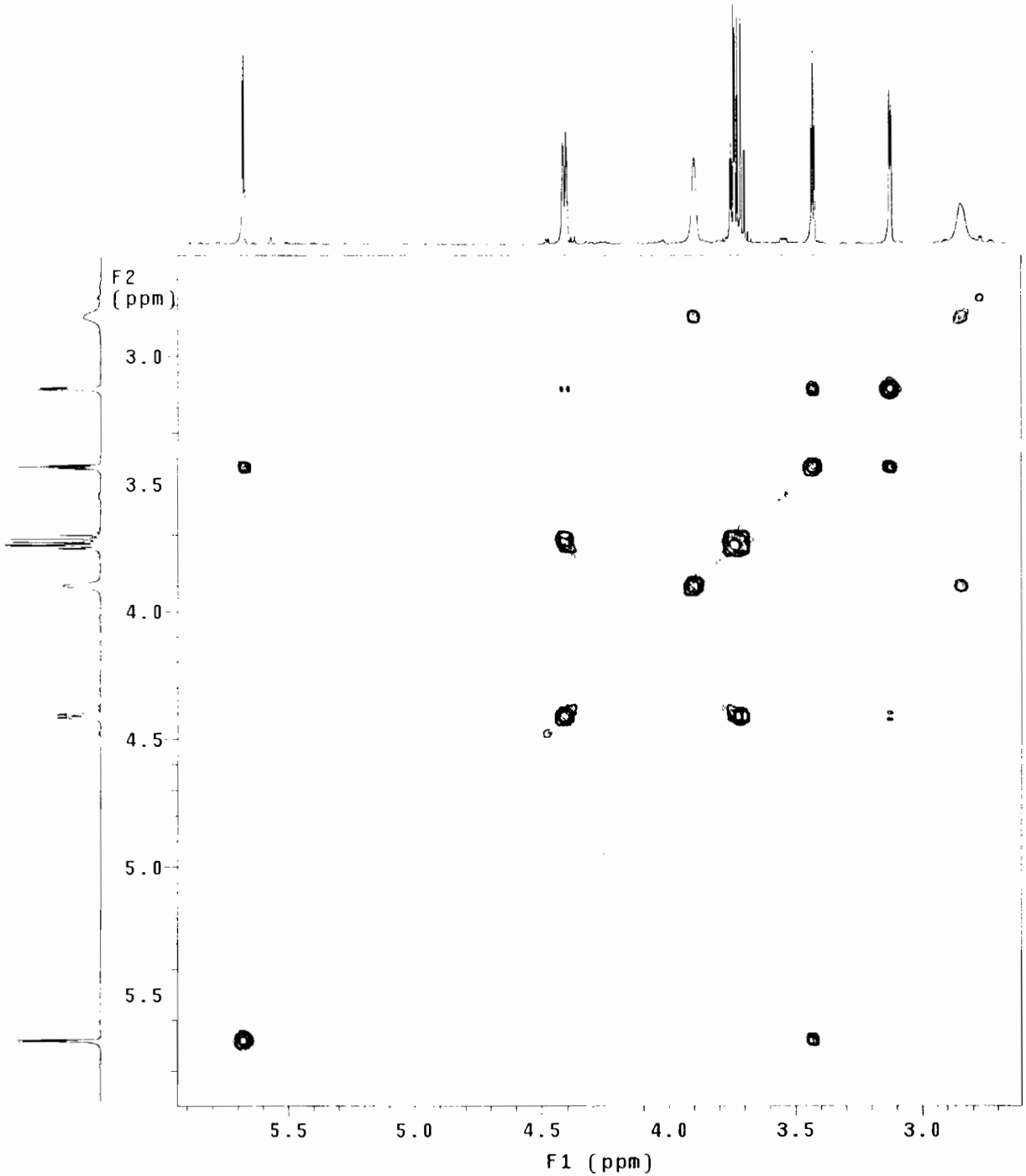
Pulse Sequence: relayh

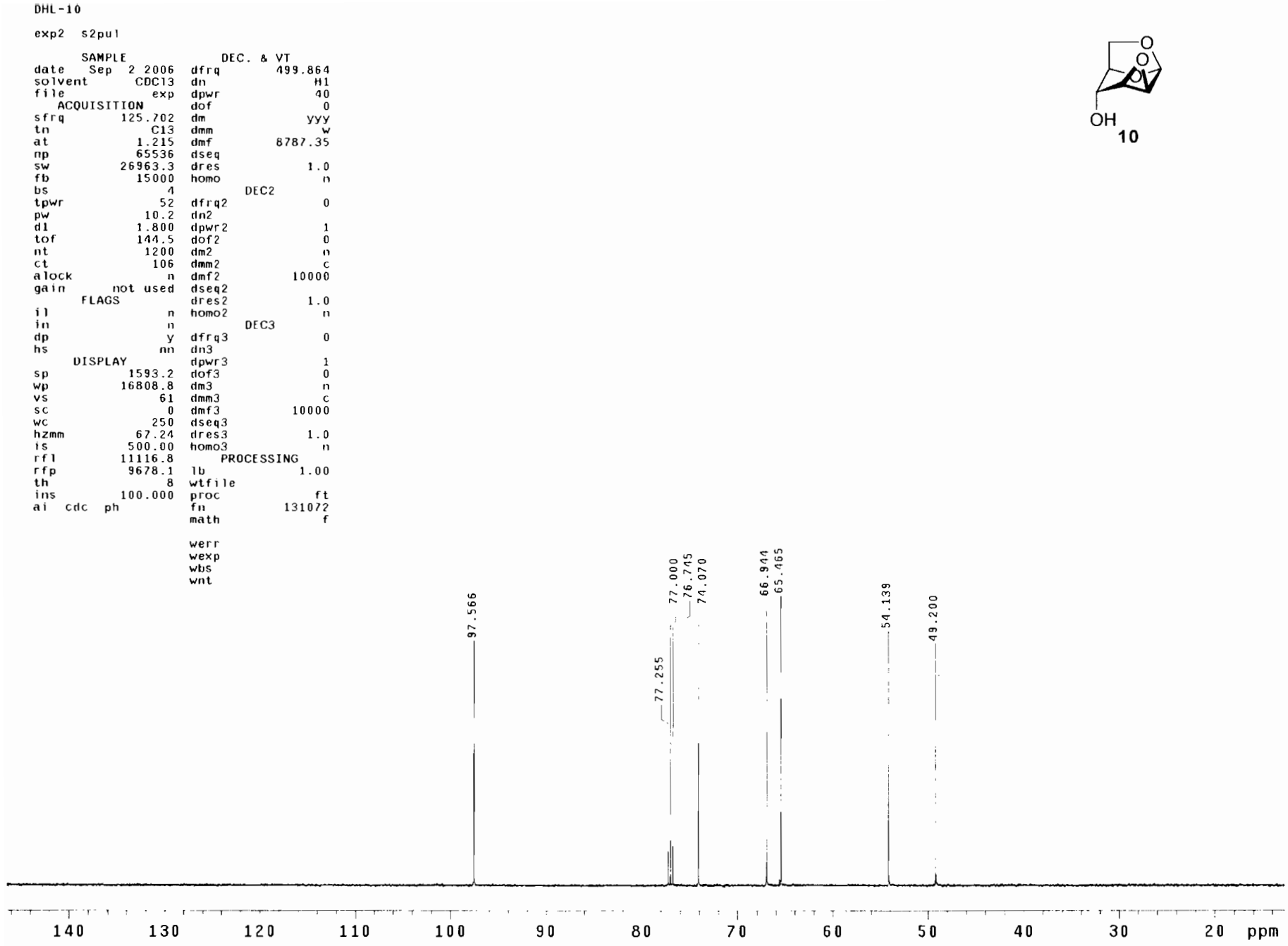
Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

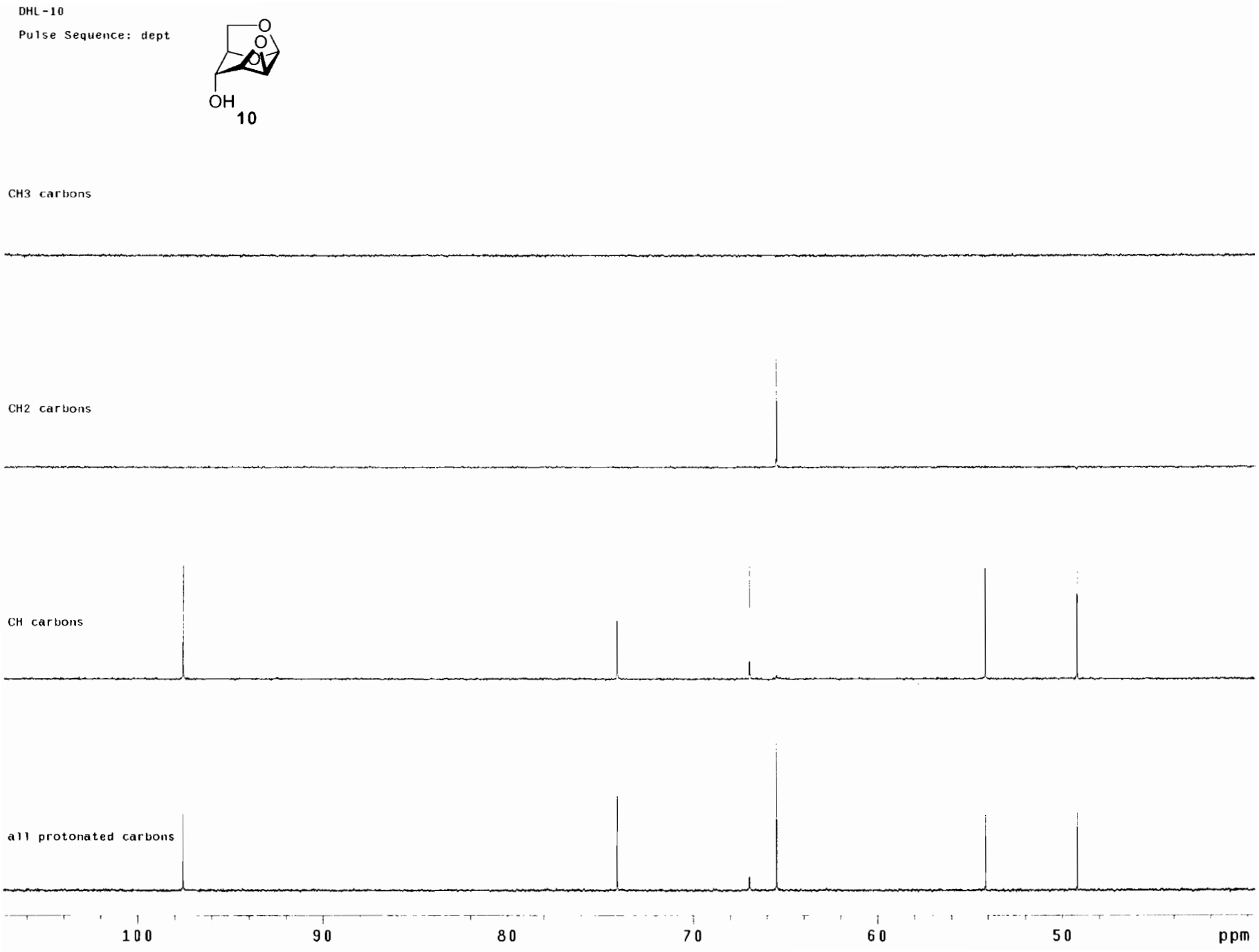
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.153 sec
Width 1669.2 Hz
2D Width 1669.2 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.077 sec
F1 DATA PROCESSING
Sine bell 0.038 sec
FT size 512 x 512
Total time 26 min, 28 sec



10



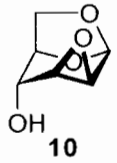

DHL-10
exp2 s2pul
SAMPLE
date Sep 2 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 106
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1593.2
wp 16808.8
vs 61
sc 0
wc 250
hzmm 67.24
is 500.00
rfl 11116.8
rfp 9678.1
th 8
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
O
O
OH
10
97.566
77.255
77.000
76.745
74.070
66.944
65.465
54.139
49.200
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


DHL-10
Pulse Sequence: dept
OH
10
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
100
90
80
70
60
50
ppm

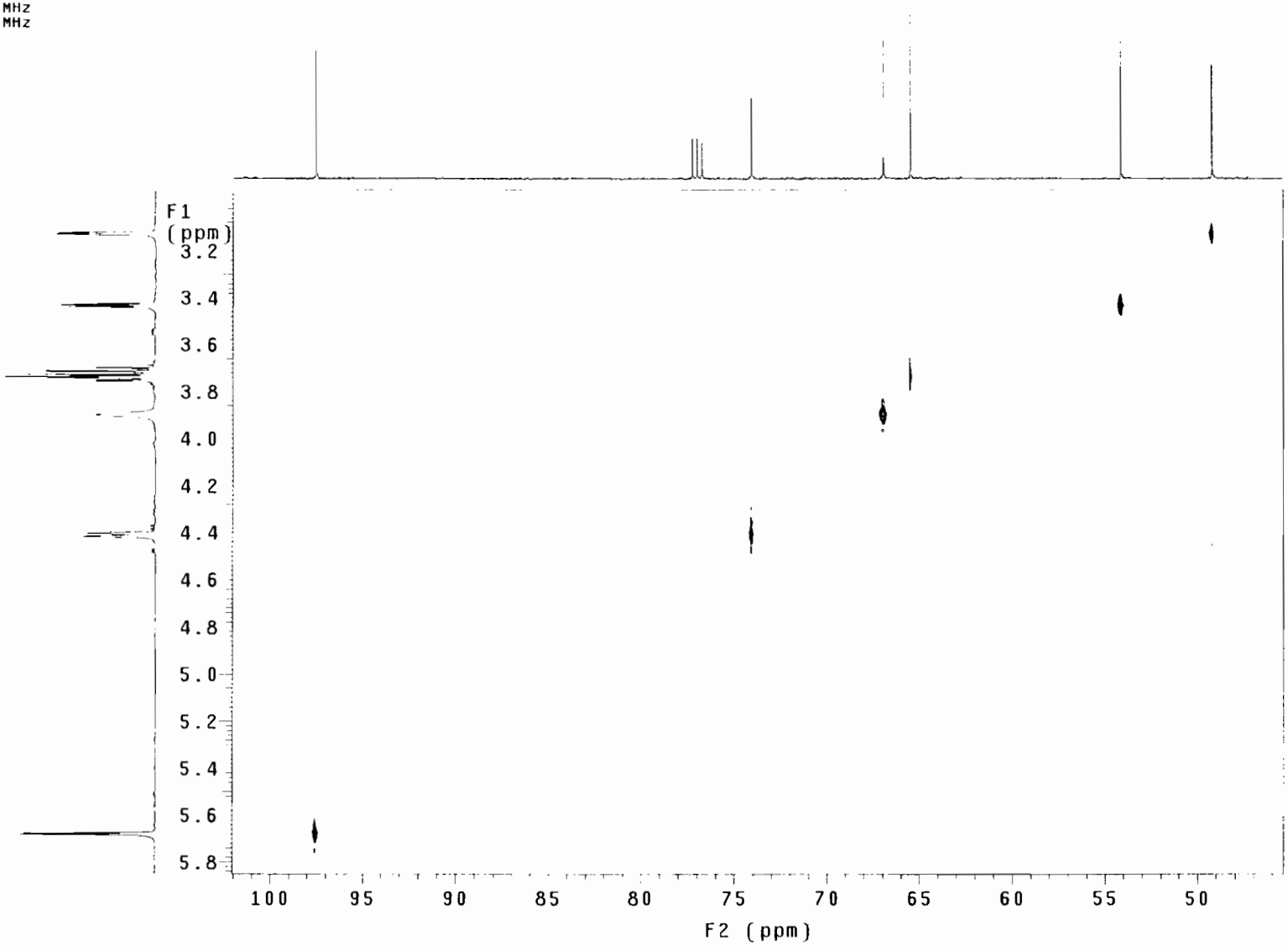
DHL-10

Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width 18403.5 Hz
 2D Width 1631.1 Hz
 4 repetitions
 256 increments
OBSERVE C13, 125.6902027 MHz
DECOUPLE H1, 499.8633086 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
F1 DATA PROCESSING
 Line broadening 0.3 Hz
FT size 4096 x 256
Total time 29 min, 21 sec



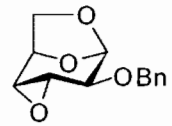
10



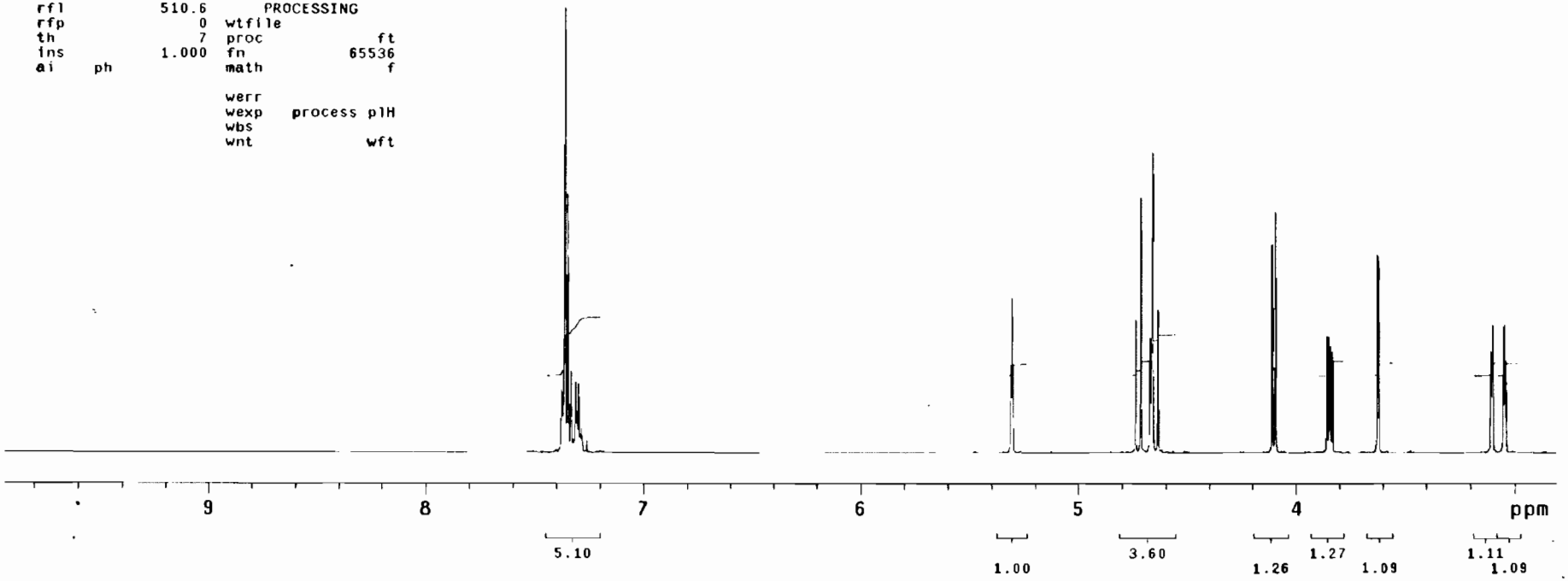
DHL-11

exp1 s2pul

SAMPLE		DEC. & VT	
date	Sep 21 2006	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1403.9	dof3	0
wp	3562.7	dm3	n
vs	58	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	14.25	dres3	1.0
is	100.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



11

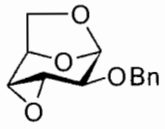


DHL-11

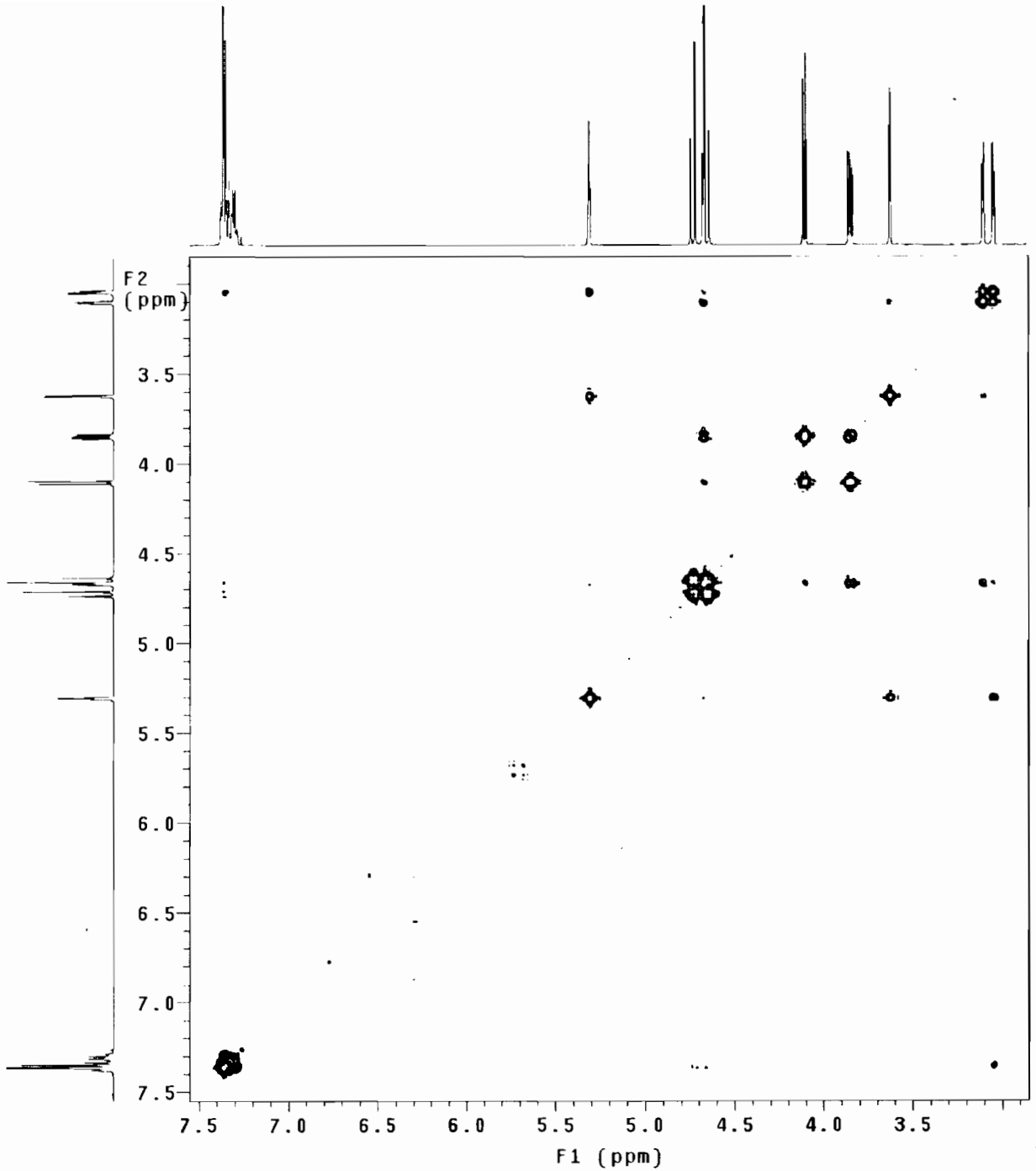
Pulse Sequence: relayh

Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.218 sec
Width 2352.1 Hz
2D Width 2352.1 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.109 sec
F1 DATA PROCESSING
Sine bell 0.054 sec
FT size 1024 x 1024
Total time 27 min, 12 sec



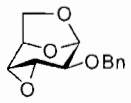
11



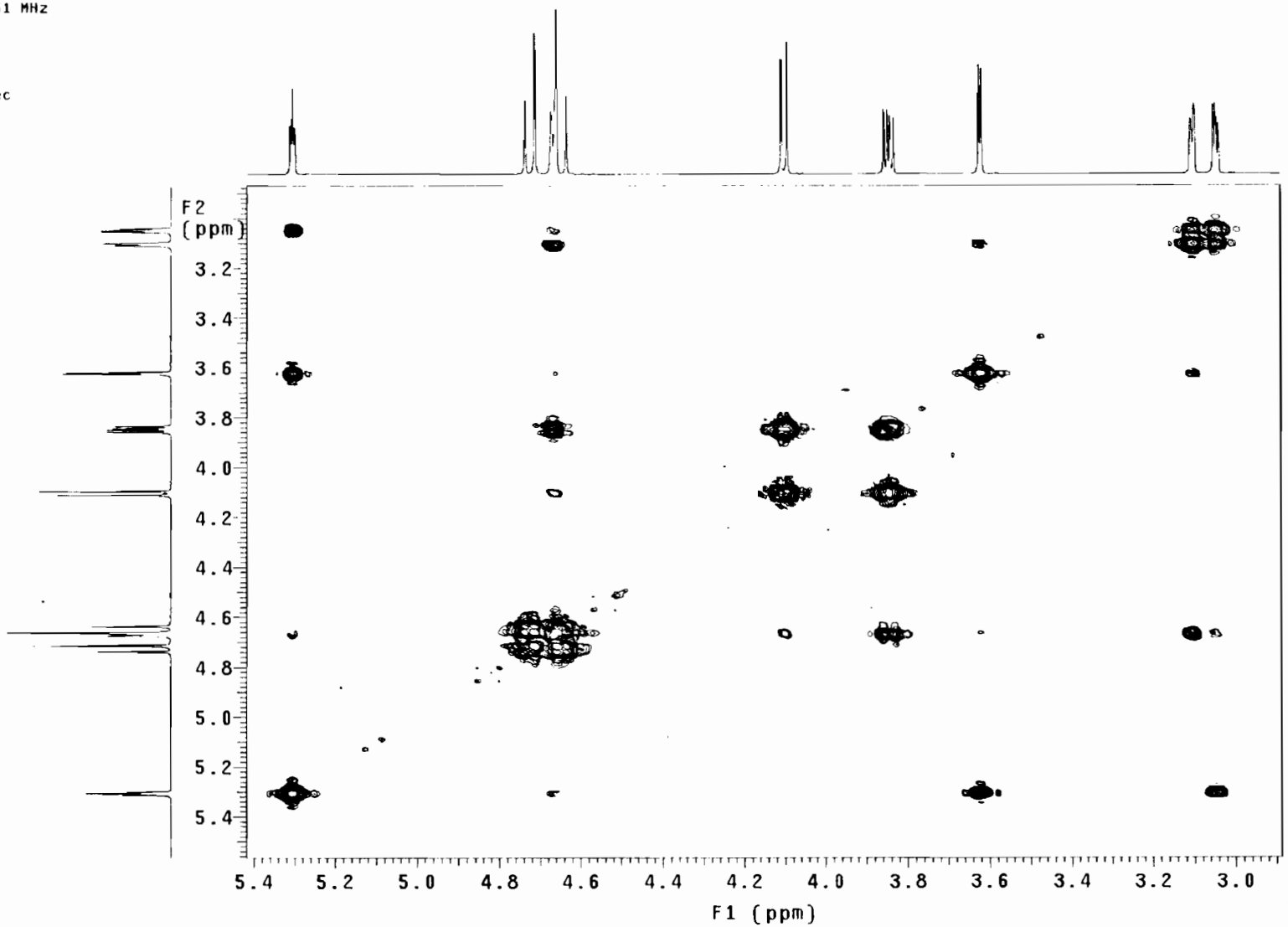
OHL-11

Pulse Sequence: relayh
 Solvent: CDCl3
 Ambient temperature
INOVA-500 "inova5"

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.218 sec
 Width 2352.1 Hz
 2D Width 2352.1 Hz
 4 repetitions
 256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
 Sine bell 0.109 sec
F1 DATA PROCESSING
 Sine bell 0.054 sec
FT size 1024 x 1024
Total time 27 min, 12 sec



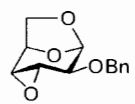
11



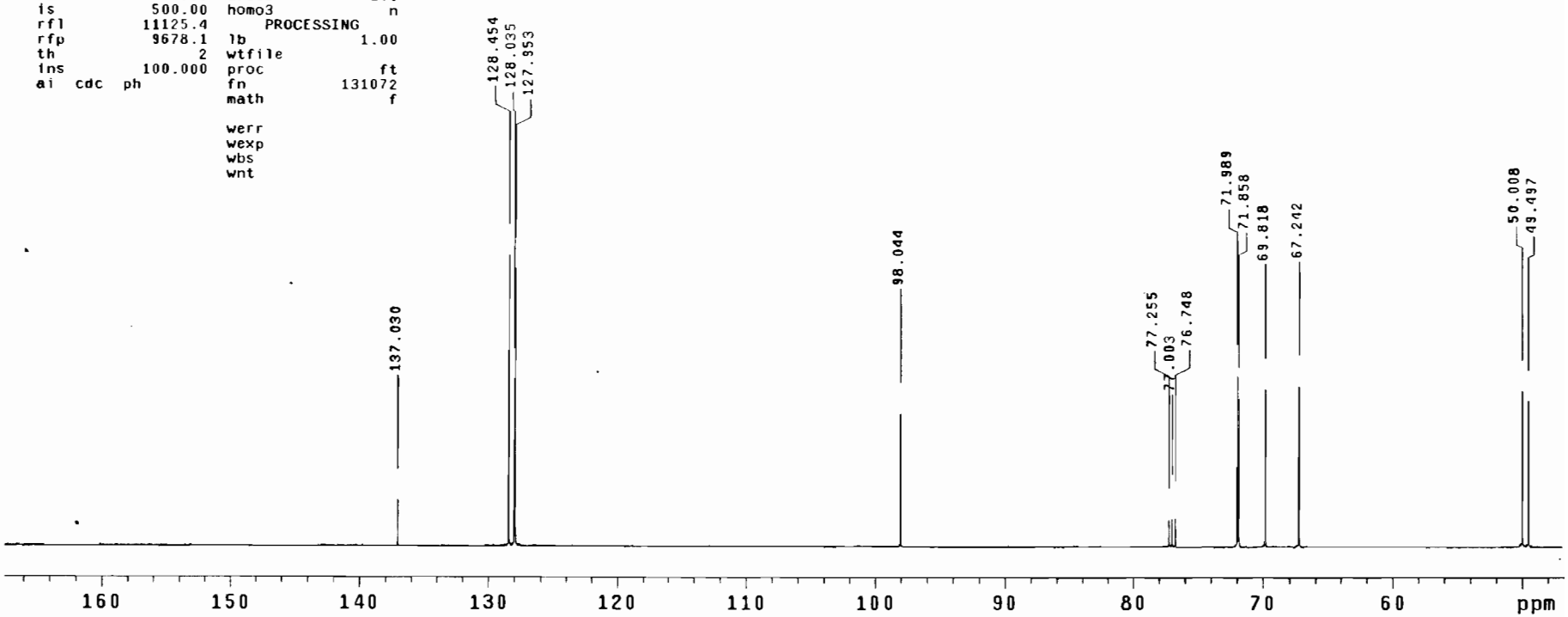
DHL-11

exp2 s2pul

SAMPLE		DEC. & VT	
date	Sep 21 2006	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1200	dm2	n
ct	100	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	5886.8	dof3	0
wp	15164.4	dm3	n
vs	30	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	60.66	dres3	1.0
is	500.00	homo3	n
rfl	11125.4	PROCESSING	
rfp	9678.1	lb	1.00
th	2	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

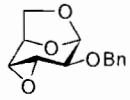


11



DHL-11

Pulse Sequence: dept



11

CH3 carbons

CH2 carbons

CH carbons

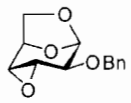
all protonated carbons

150 140 130 120 110 100 90 80 70 60 50 ppm

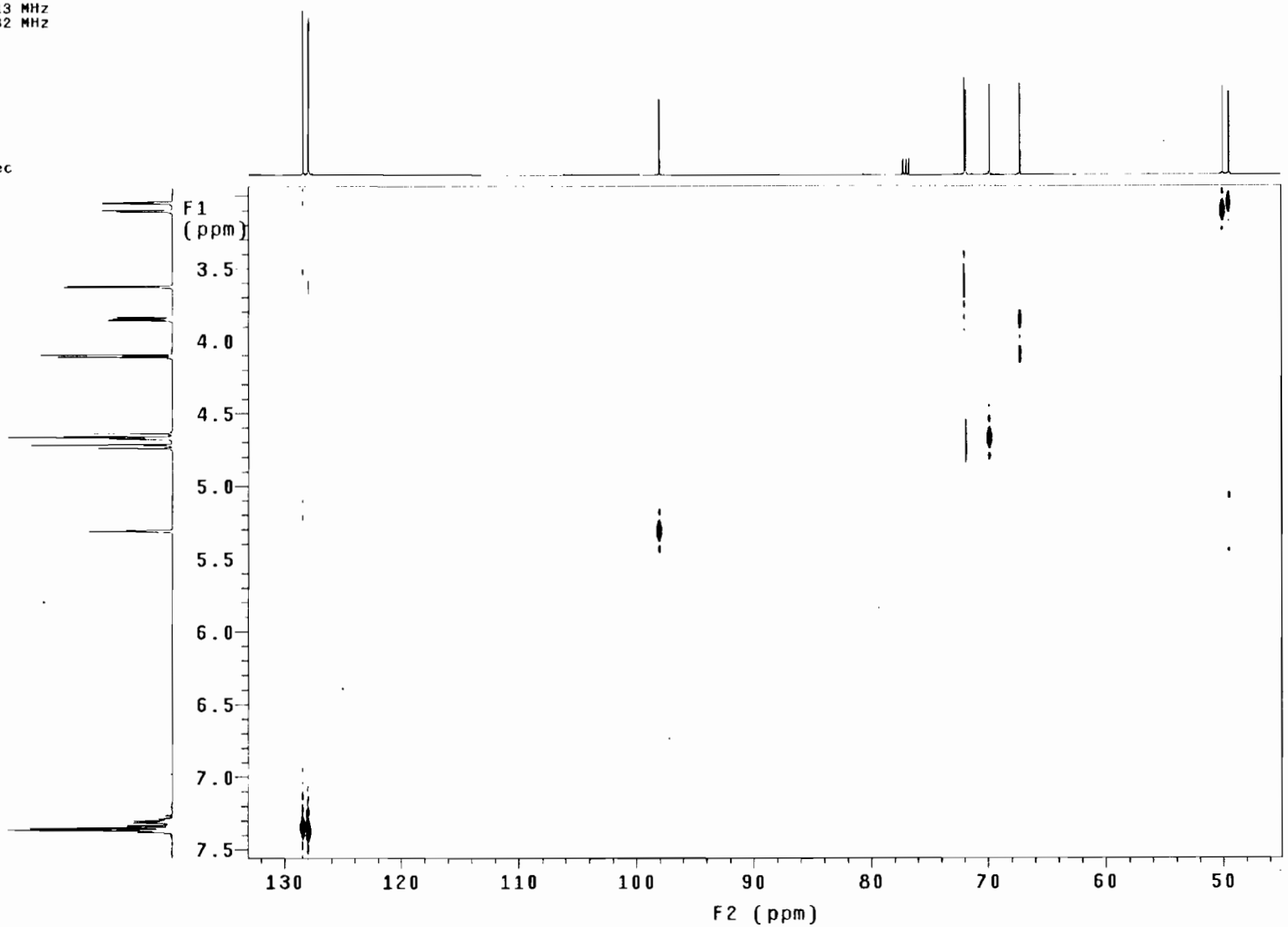
DHL-11

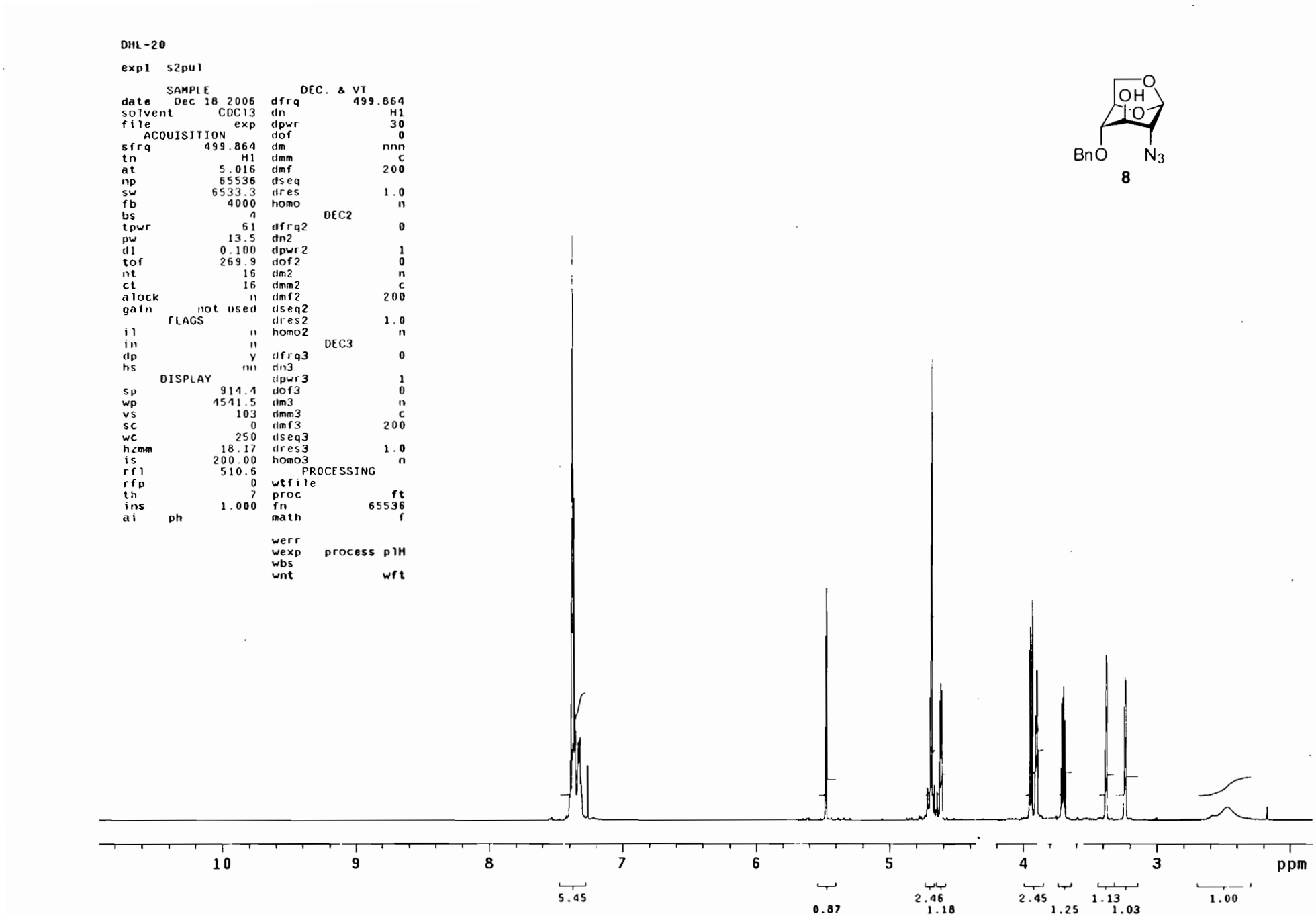
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width 18403.5 Hz
 2D Width 2345.5 Hz
 4 repetitions
 256 increments
OBSERVE C13, 125.6902113 MHz
DECOUPLE H1, 499.8637982 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
F1 DATA PROCESSING
 Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 57 sec



11

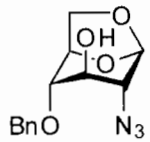



DHL-20
expl s2pul
SAMPLE
date Dec 18 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 914.4
wp 4541.5
vs 103
sc 0
wc 250
hzmm 18.17
is 200.00
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
OH
O
BnO
N3
8
10
9
8
7
6
5
4
3
ppm
5.45
0.87
2.46
1.18
2.45
1.25
1.13
1.03
1.00

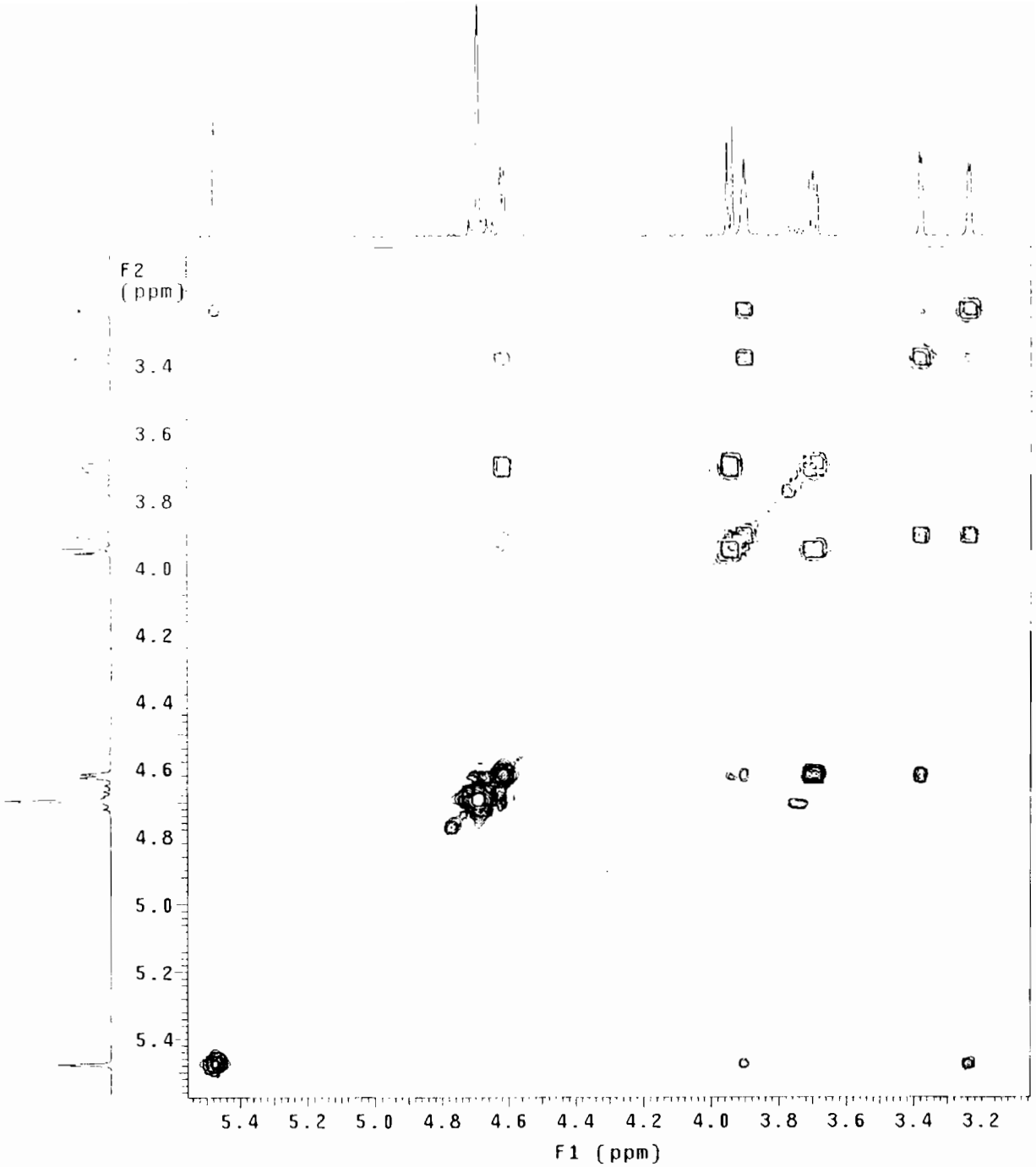
DHL-20

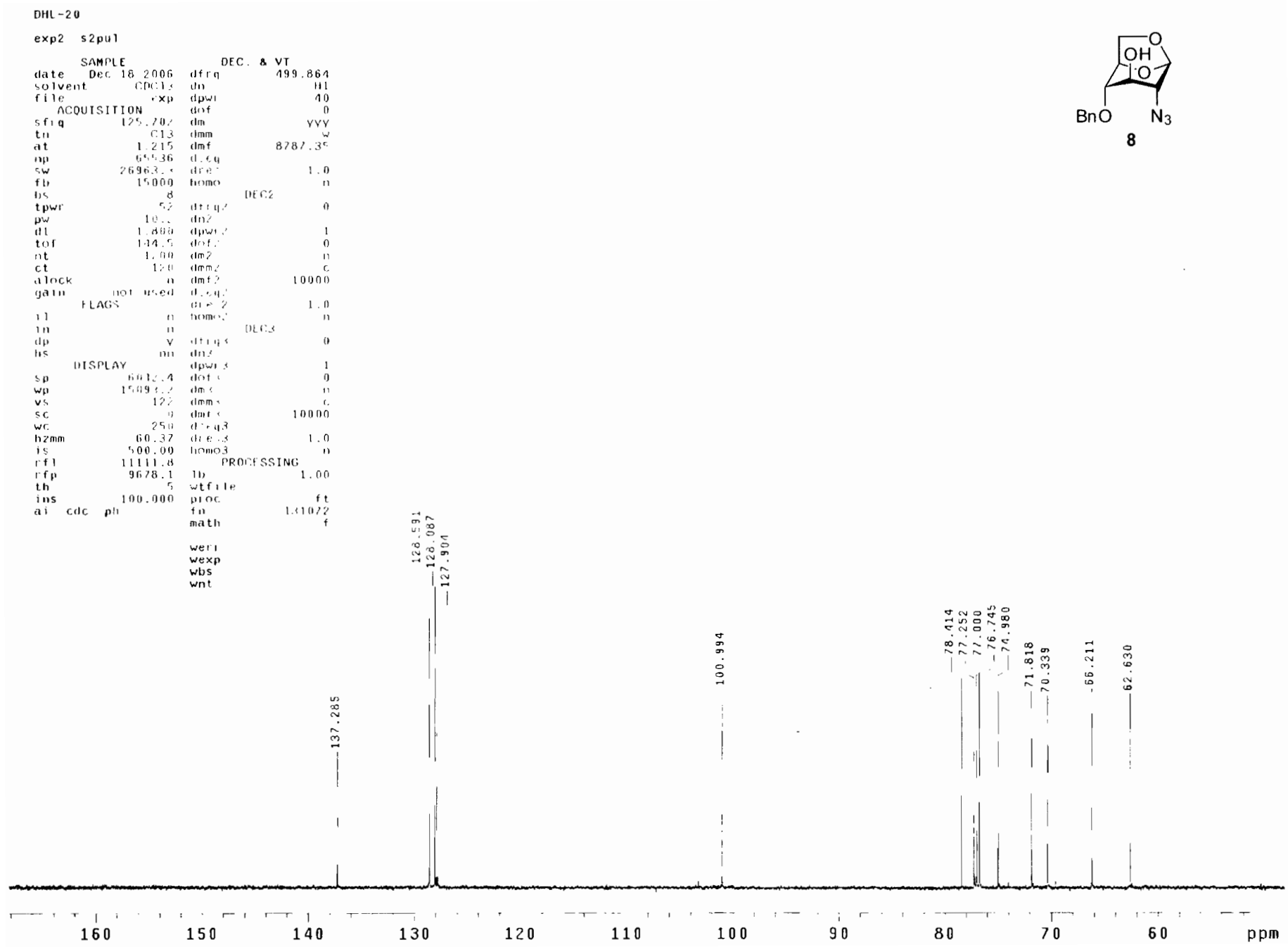
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.190 sec
Width 2697.4 Hz
2D Width 2697.4 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.095 sec
F1 DATA PROCESSING
Sine bell 0.047 sec
FT size 1024 x 1024
Total time 53 min, 1 sec



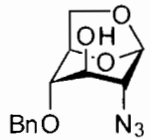
8



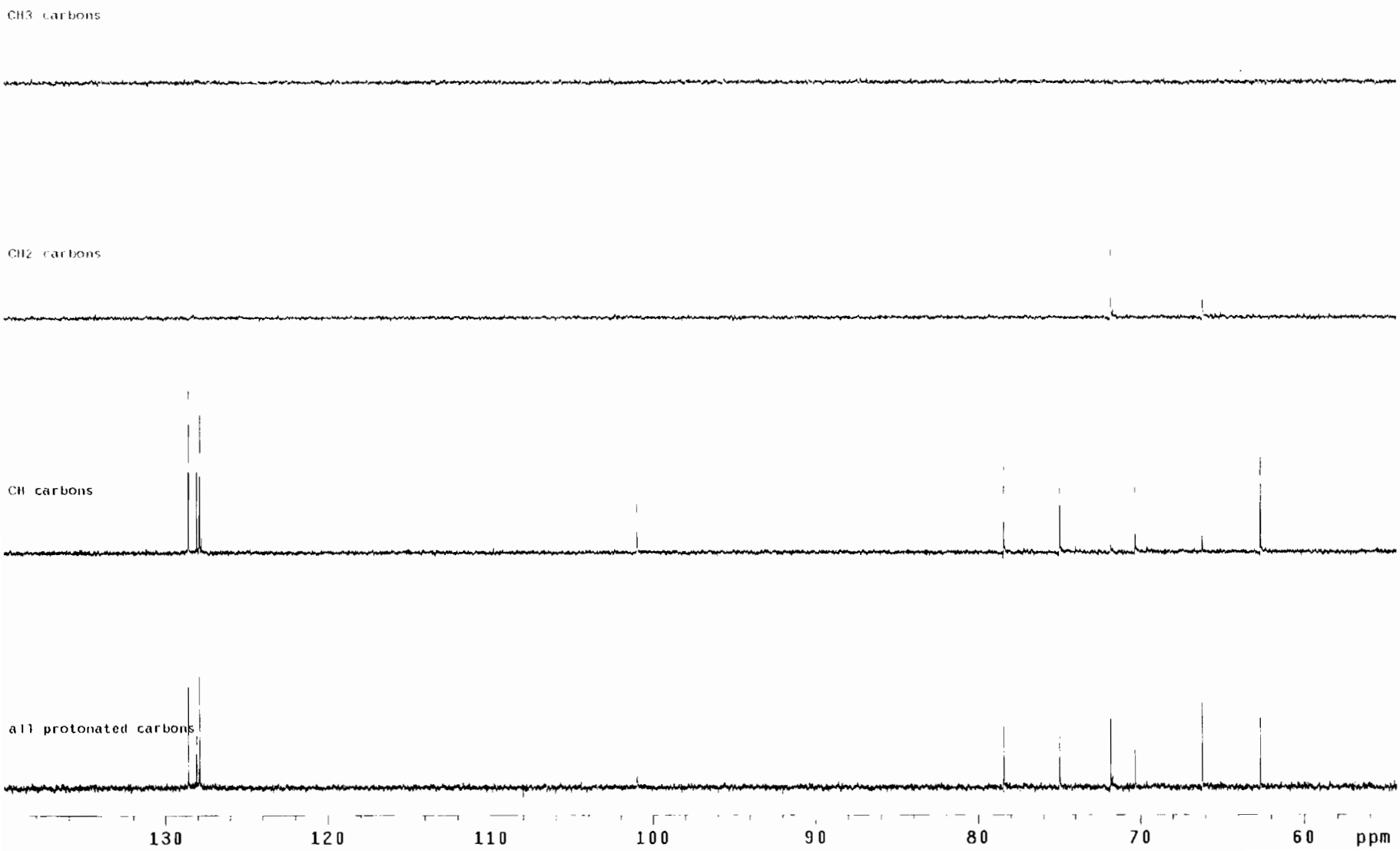

DHL-20
exp2 s2pul
SAMPLE
date Dec 18 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 125.70
tn C13
at 1.215
np 65536
sw 26963.
fb 15000
bs 8
tpwr 52
pw 10.
dl 1.688
tof 144.5
nt 1.00
ct 120
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 6612.4
wp 15893.2
vs 122
sc 0
wc 250
hzmm 60.37
is 500.00
rfl 11111.8
rfp 9678.1
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
d.cq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
d.cq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dfrq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
128.591
128.087
127.904
137.285
100.994
78.414
77.252
77.000
76.745
74.980
71.818
70.339
66.211
62.630
BnO
OH
O
N3
8
160
150
140
130
120
110
100
90
80
70
60
ppm

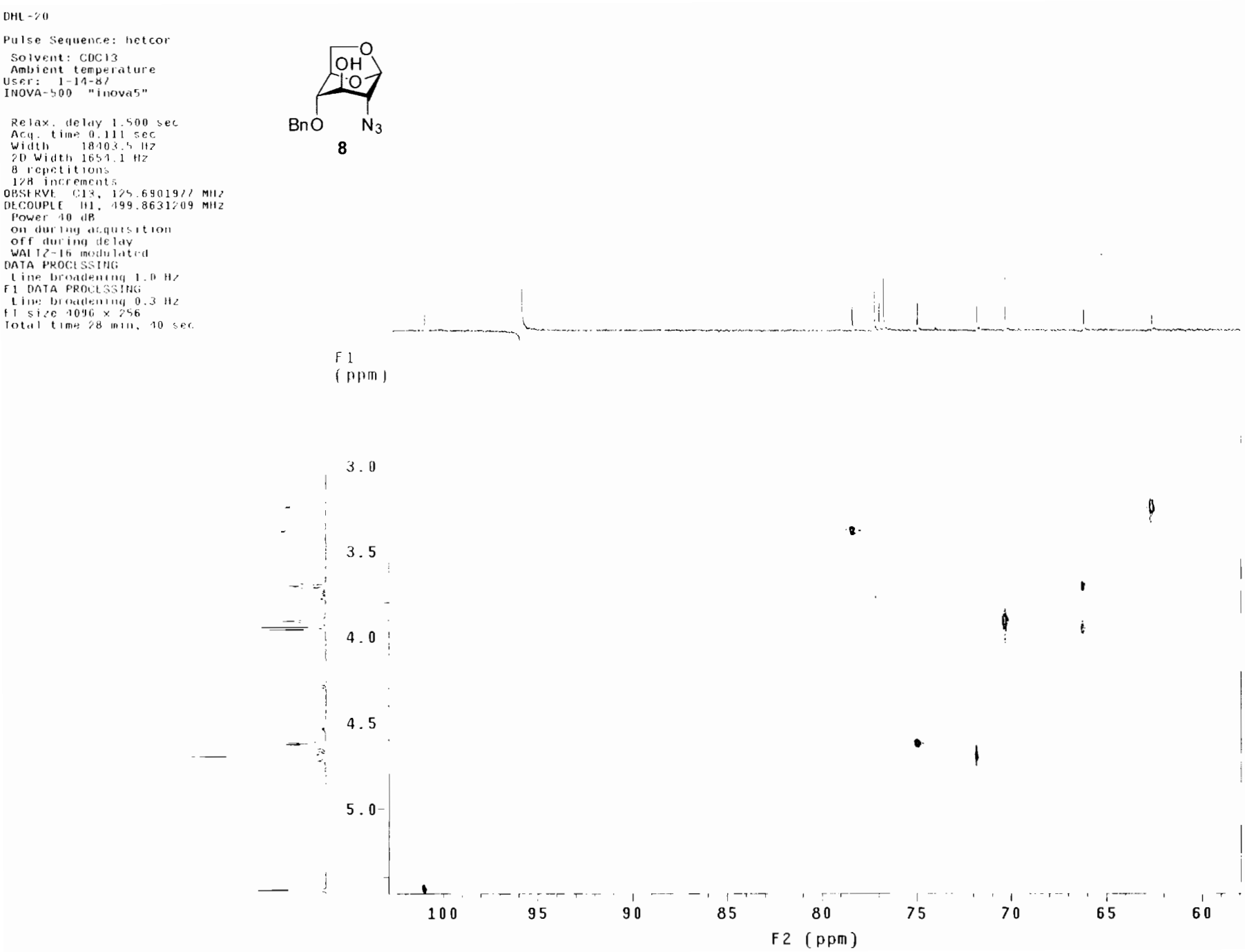
DHL-20

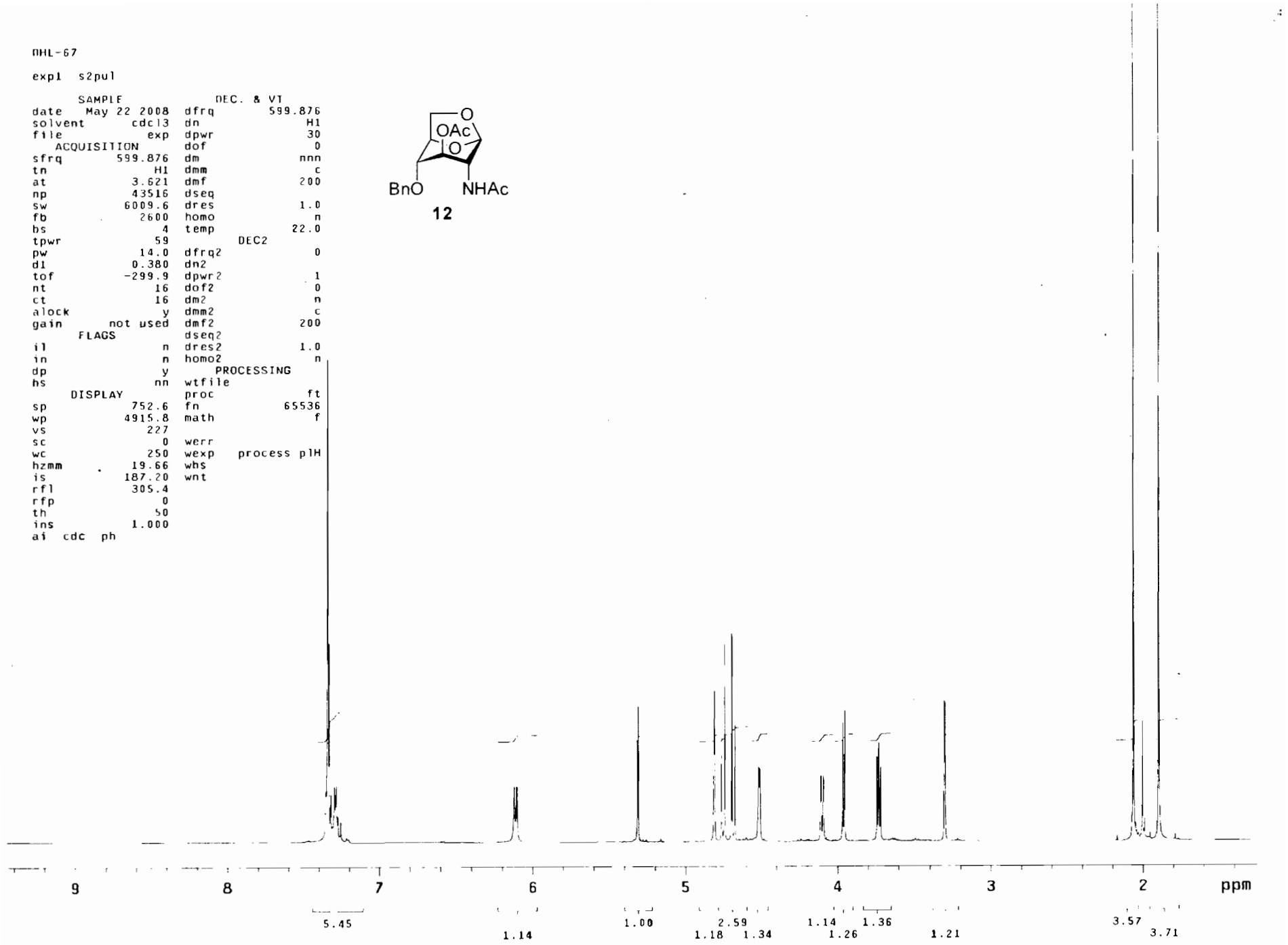
Pulse Sequence: dept



8




DHL-20
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 1654.1 Hz
8 repetitions
128 increments
OBSERVE C13, 125.6901977 MHz
DECOUPLE H1, 499.8631209 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
f1 size 4096 x 256
Total time 28 min, 40 sec
OH
O
O
BnO
N3
8
F1
(ppm)
3.0
3.5
4.0
4.5
5.0
100
95
90
85
80
75
70
65
60
F2 (ppm)


MHL-67
expl s2pul
SAMPLE
date May 22 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 599.876
tn H1
at 3.621
np 43516
sw 6009.6
fb 2600
bs 4
tpwr 59
pw 14.0
dl 0.380
tof -299.9
nt 16
ct 16
alock y
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 752.6
wp 4915.8
vs 227
sc 0
wc 250
hzmm 19.66
is 187.20
rfl 305.4
rfp 0
th 50
ins 1.000
ai cdc ph
DEC. & VT
dfrq 599.876
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 22.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
whs
wnt
O
OAc
O
BnO
NHAc
12
9
8
7
6
5
4
3
2
ppm
5.45
1.14
1.00
2.59
1.18
1.34
1.14
1.26
1.36
1.21
3.57
3.71

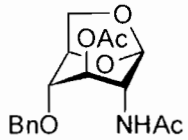
DHL-71

File: afs/nd.edu/user26/dhesek/Private/DHL/DHL-71_HH.fid

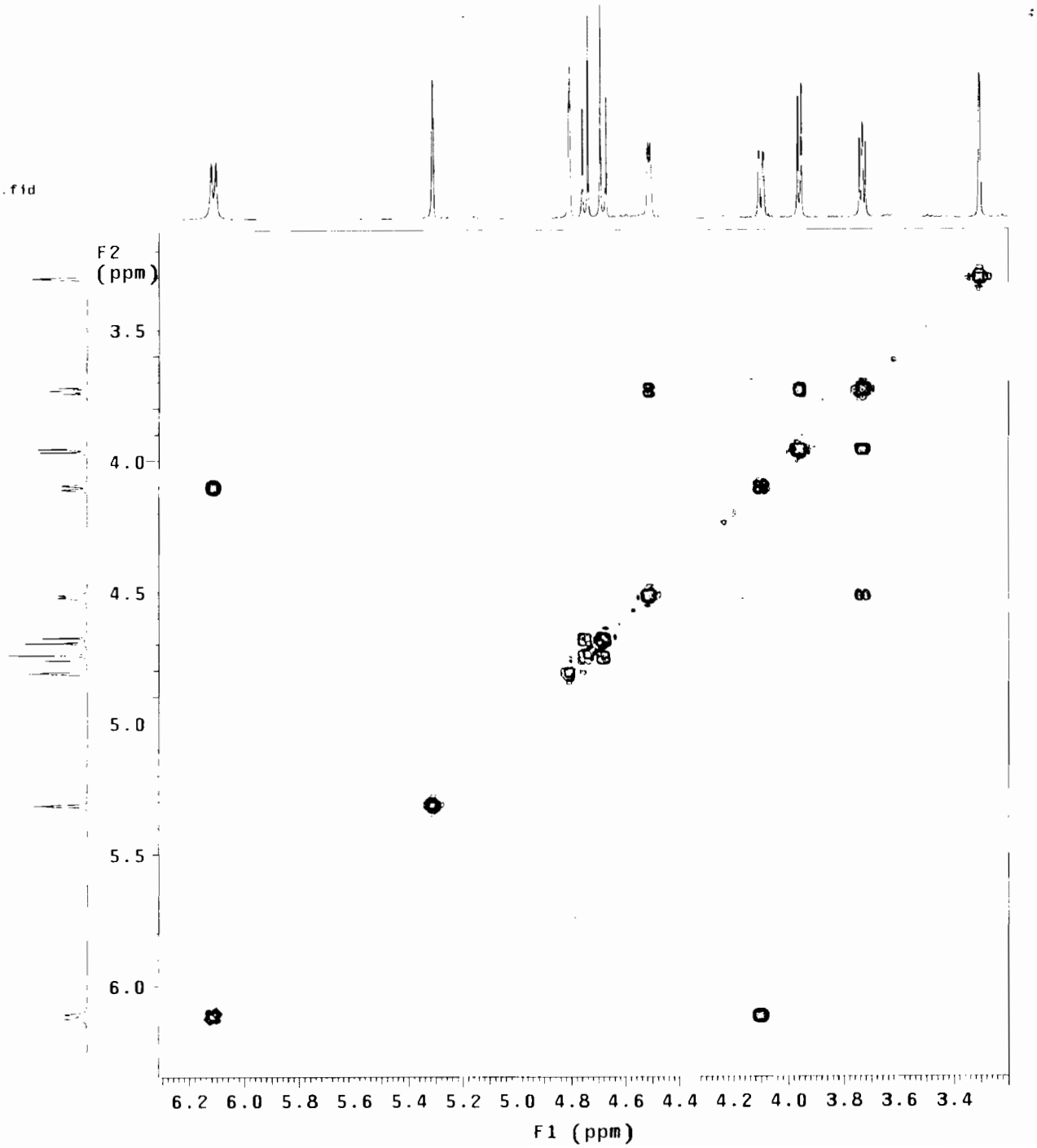
Pulse Sequence: COSY

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
File: DHL-71_HH
VNMRS-600 "nmr600"

Relax. delay 1.000 sec
Acq. time 0.128 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
4 repetitions
256 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Sine bell 0.064 sec
F1 DATA PROCESSING
Sine bell 0.043 sec
FT size 2048 x 2048
Total time 20 min, 15 sec



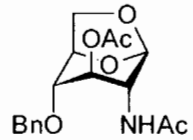
12



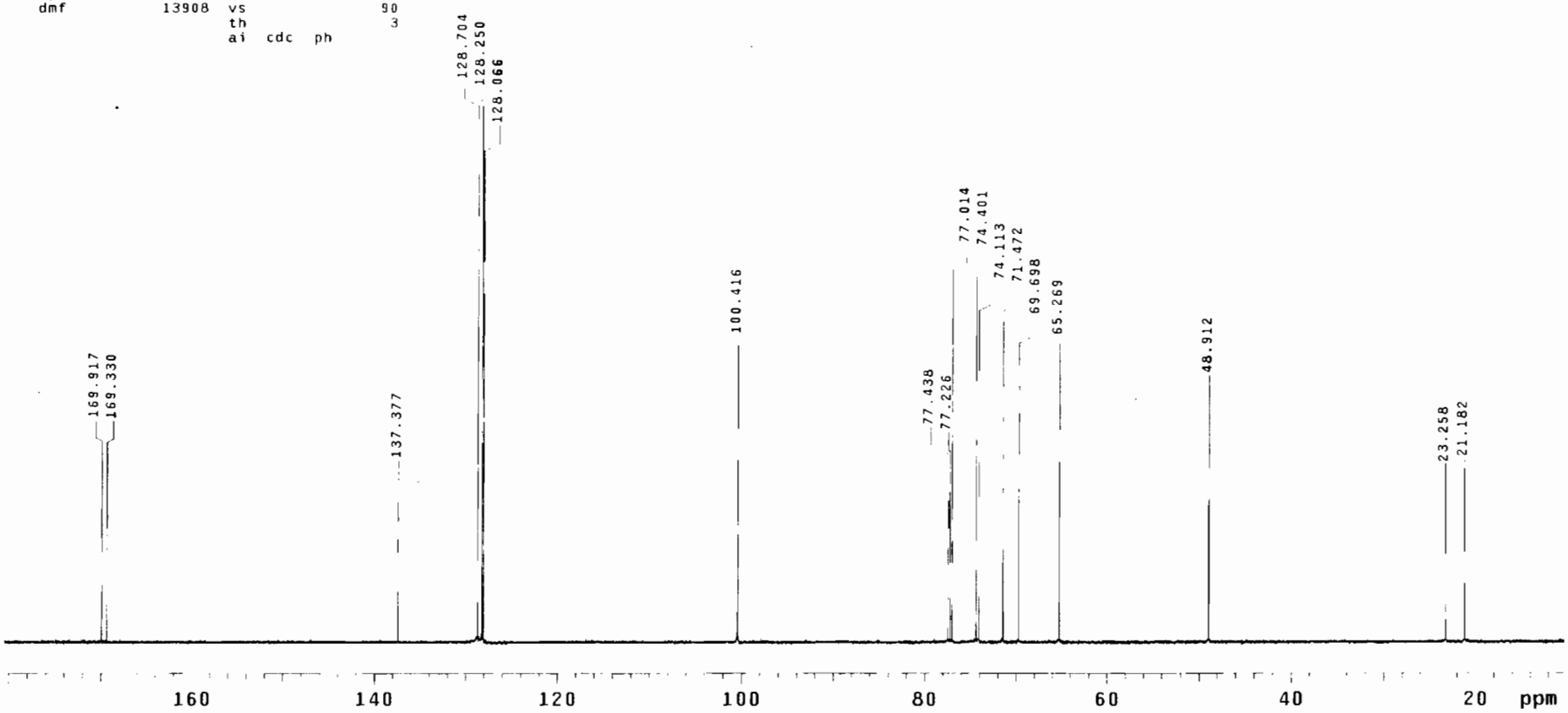
DHL-71

exp3 Carbon

SAMPLE		SPECIAL	
date	May 22 2008	temp	22.0
solvent	cd3od	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	36764.7	pw90	7.500
at	1.783	alfa	10.000
np	131072	**FLAGS**	
fb	17000	il	n
bs	4	in	n
d1	1.220	dp	y
nt	512	hs	nn
ct	40	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	262144
sfrq	150.855	**DISPLAY**	
tof	1542.7	sp	1544.1
tpwr	58	wp	25669.8
pw	7.500	rfl	13586.1
DECOUPLER		rfp	11649.3
dn	H1	rp	-117.2
dof	0	lp	30.3
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	44	sc	0
dmf	13908	vs	90
		th	3
		ai cdc ph	

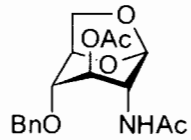


12

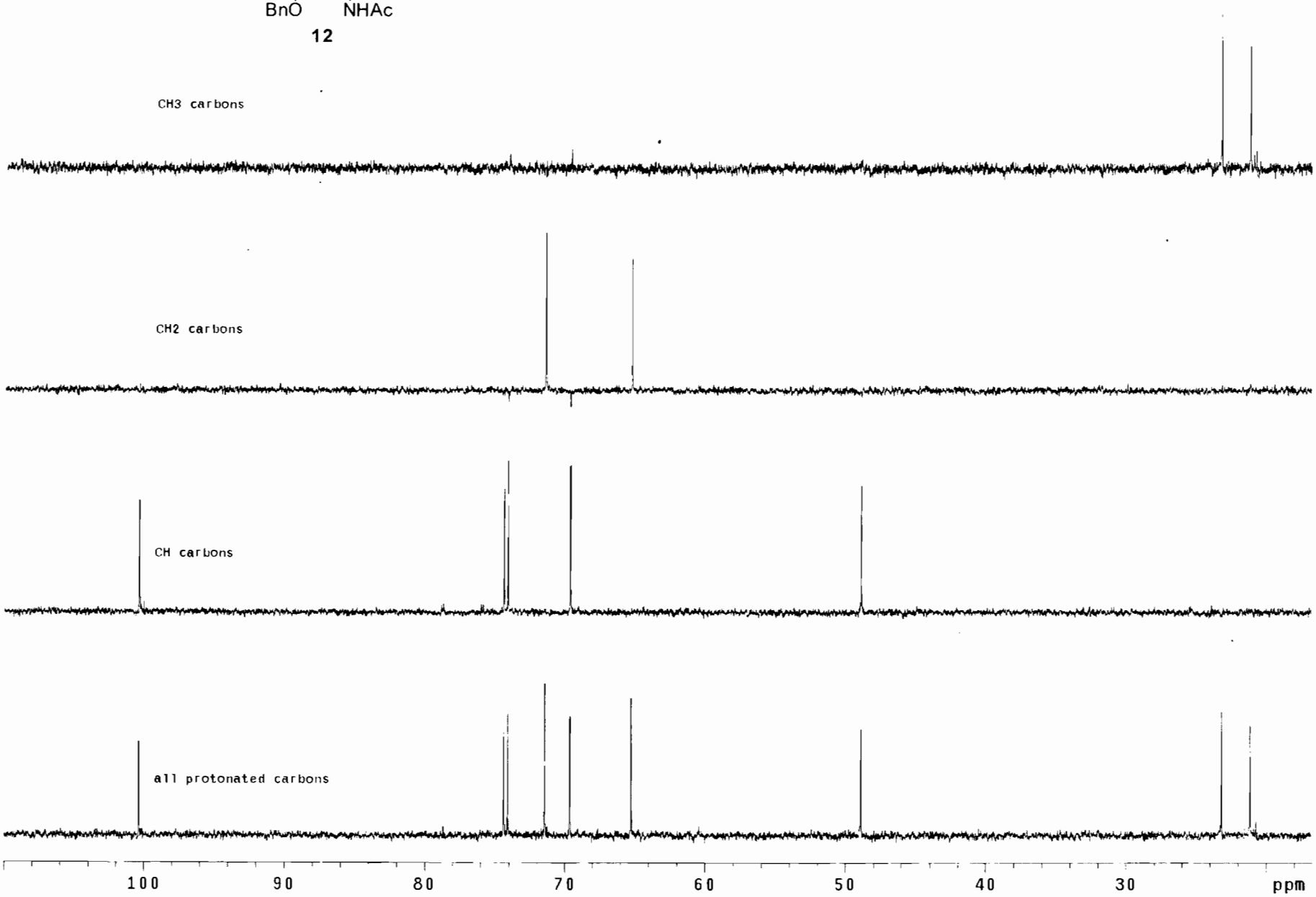


DHL-71

Pulse Sequence: dept



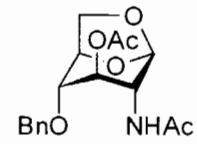
12



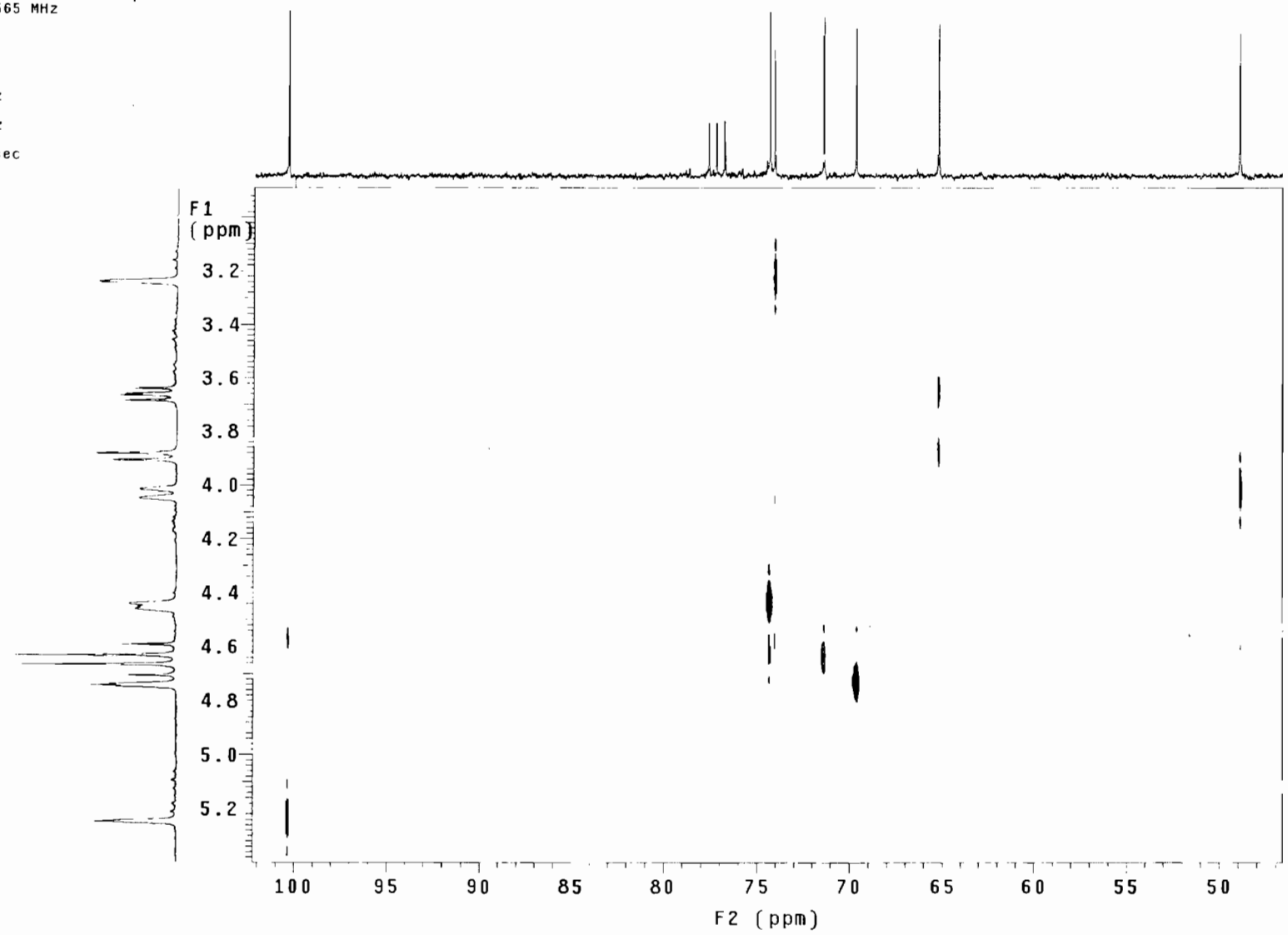
DHL-71

Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr3a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.179 sec
Width 11471.2 Hz
2D Width 1790.8 Hz
4 repetitions
128 increments
OBSERVE C13, 75.4216934 MHz
DECOUPLE H1, 299.9482565 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 14 min, 57 sec



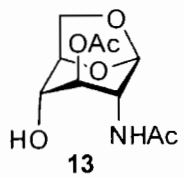
12



DHL-72

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 27 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPlAY		dpwr3	1
sp	502.5	dof3	0
wp	5004.8	dm3	n
vs	68	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.02	dres3	1.0
is	100.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

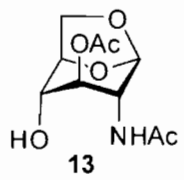


13

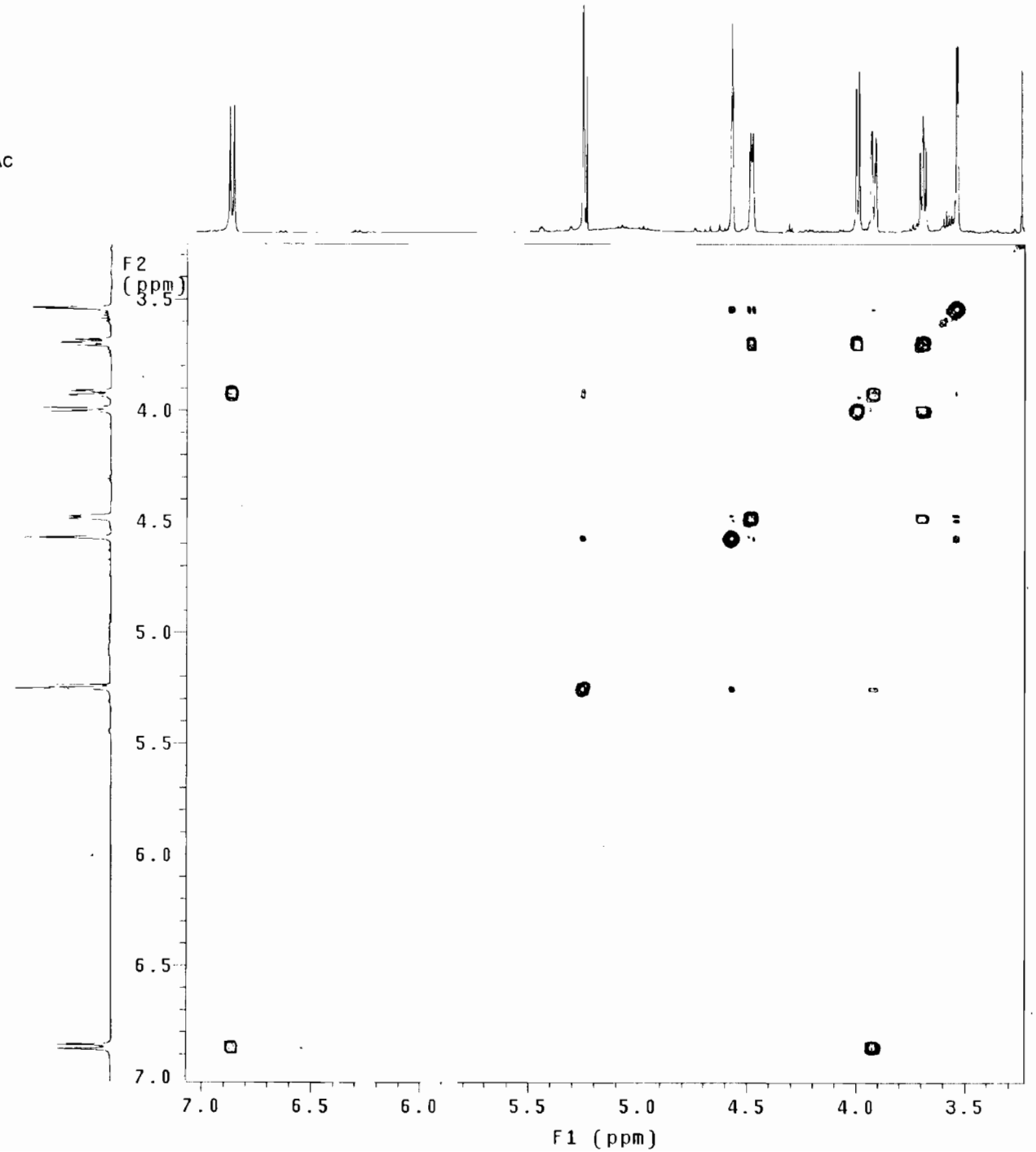
DHL-72

Pulse Sequence: relayh
 Solvent: CDCl3
 Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.172 sec
 Width 2970.3 Hz
 2D Width 2970.3 Hz
 4 repetitions
 256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
 Sine bell 0.086 sec
F1 DATA PROCESSING
 Sine bell 0.043 sec
FT size 1024 x 1024
Total time 26 min, 14 sec



13

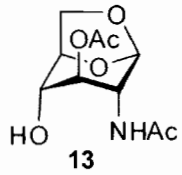


DHL - 7L

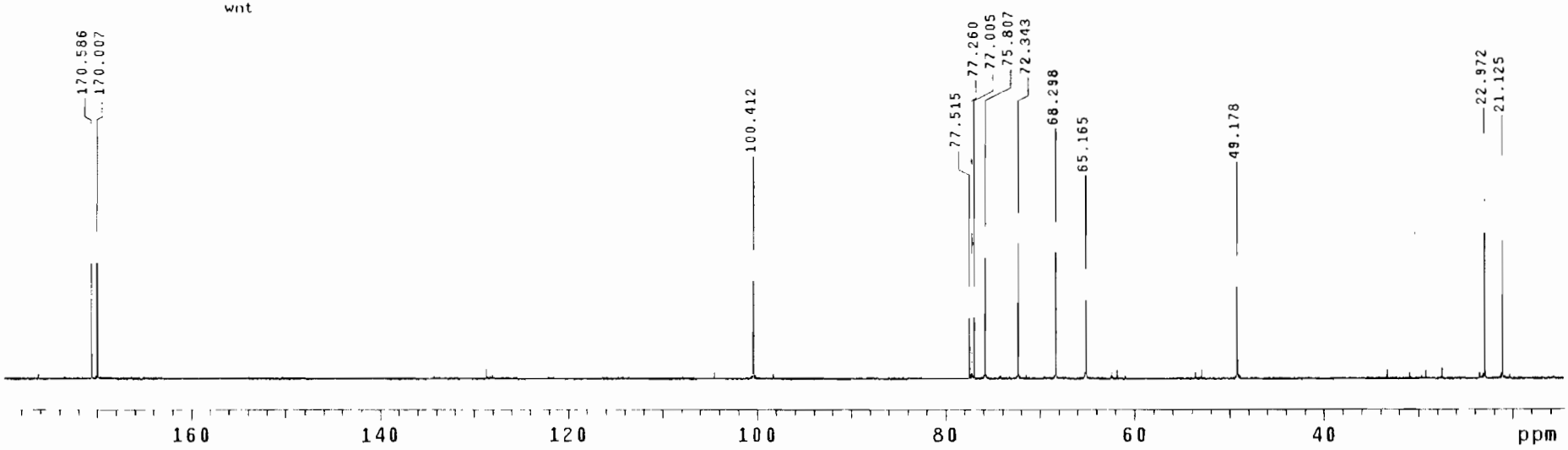
STANDARD PROTON PARAMETERS

exp2 s2pul

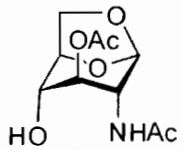
SAMPLE		DEC. & VT	
date	May 27 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	640	dm2	n
ct	161	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1833.2	dof3	0
wp	20768.4	dm3	n
vs	61	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	83.07	dres3	1.0
is	500.00	homo3	n
rfl	11130.7	PROCESSING	
rfp	9710.8	lb	1.00
th	7	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



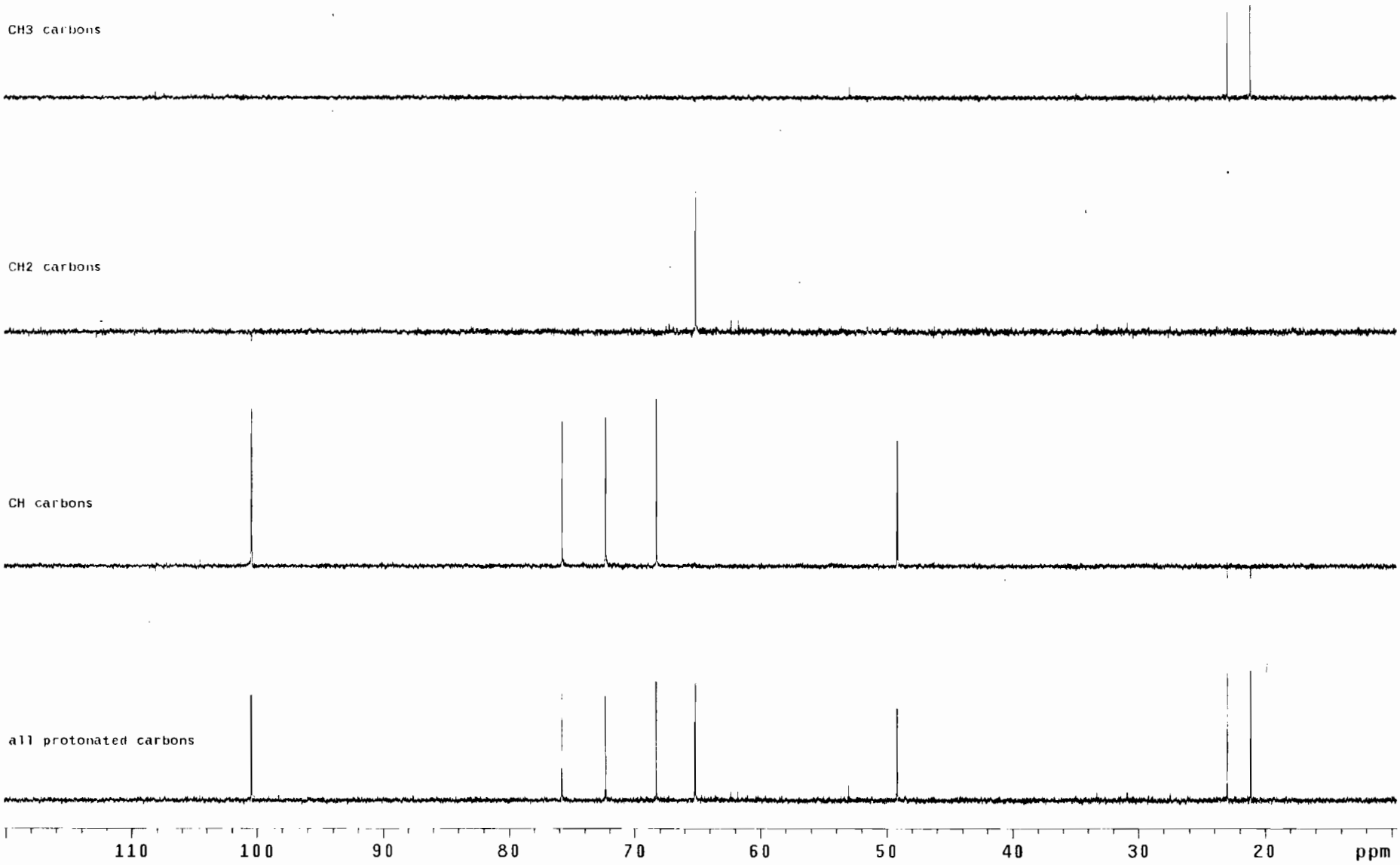
13



DHL-72
STANDARD PROTON PARAMETERS
Pulse Sequence: dept



13



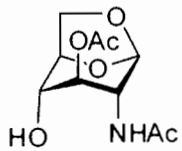
DHL-7L

STANDARD PROTON PARAMETERS

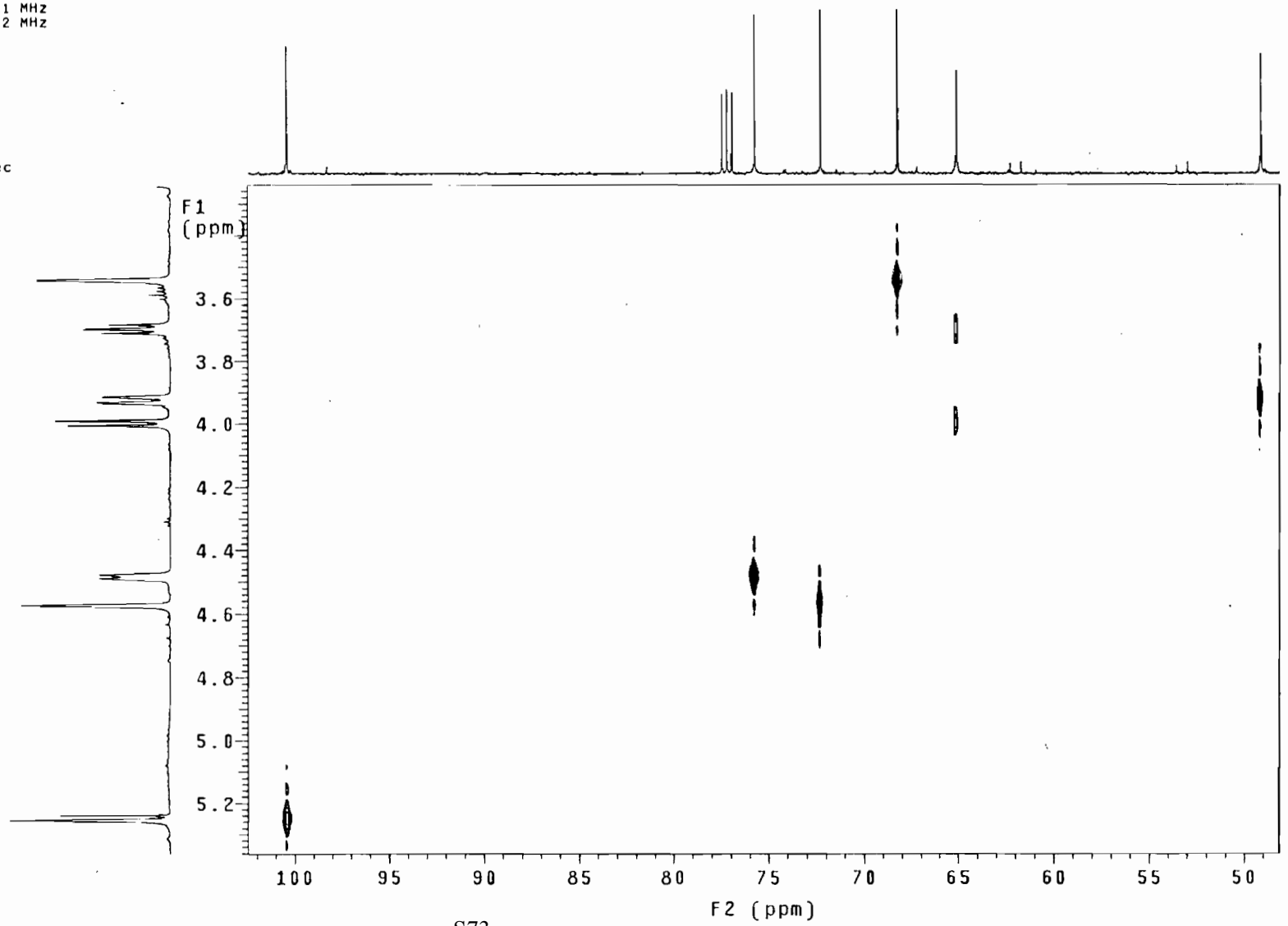
Pulse Sequence: hetcor

Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 6533.3 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6901841 MHz
DECOUPLE H1, 499.8639312 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 28 min, 21 sec



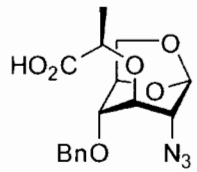
13

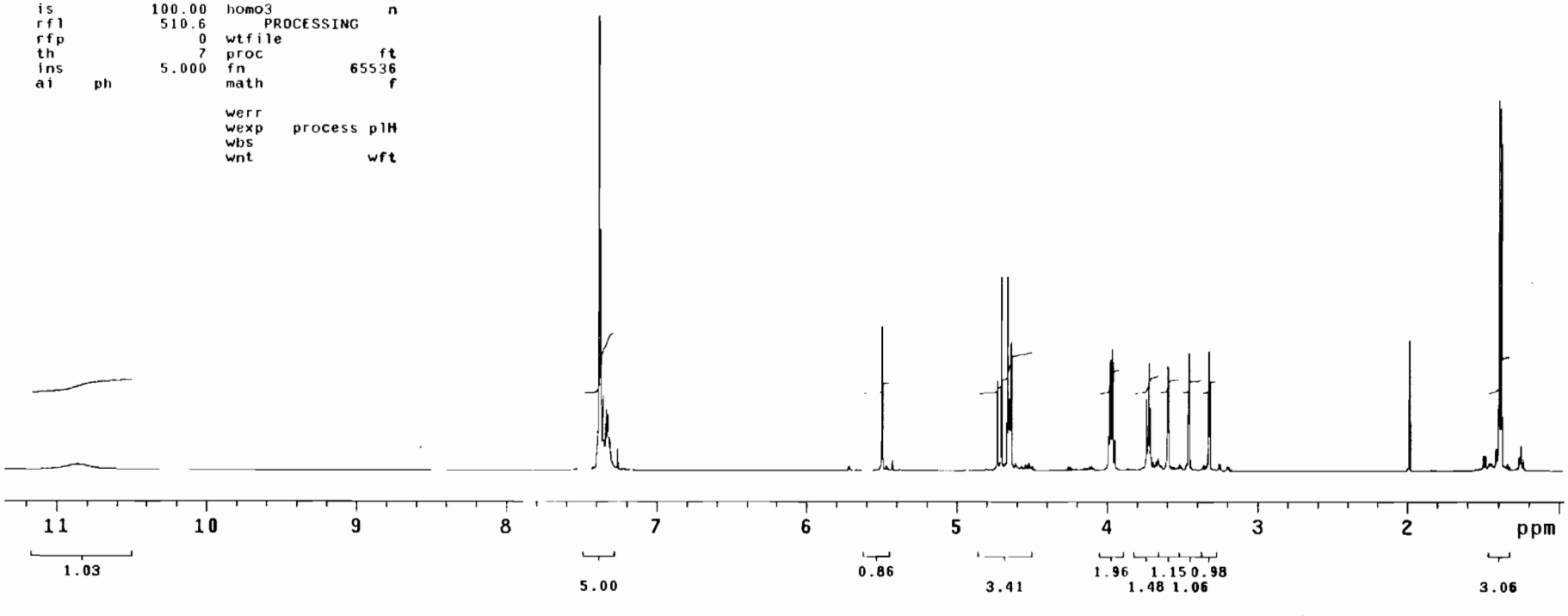


DHL-21

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jan 27 2007	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	485.3	dof3	0
wp	5185.1	dm3	n
vs	55	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.74	dres3	1.0
is	100.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	5.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

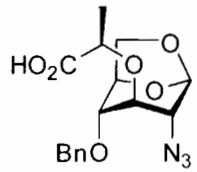


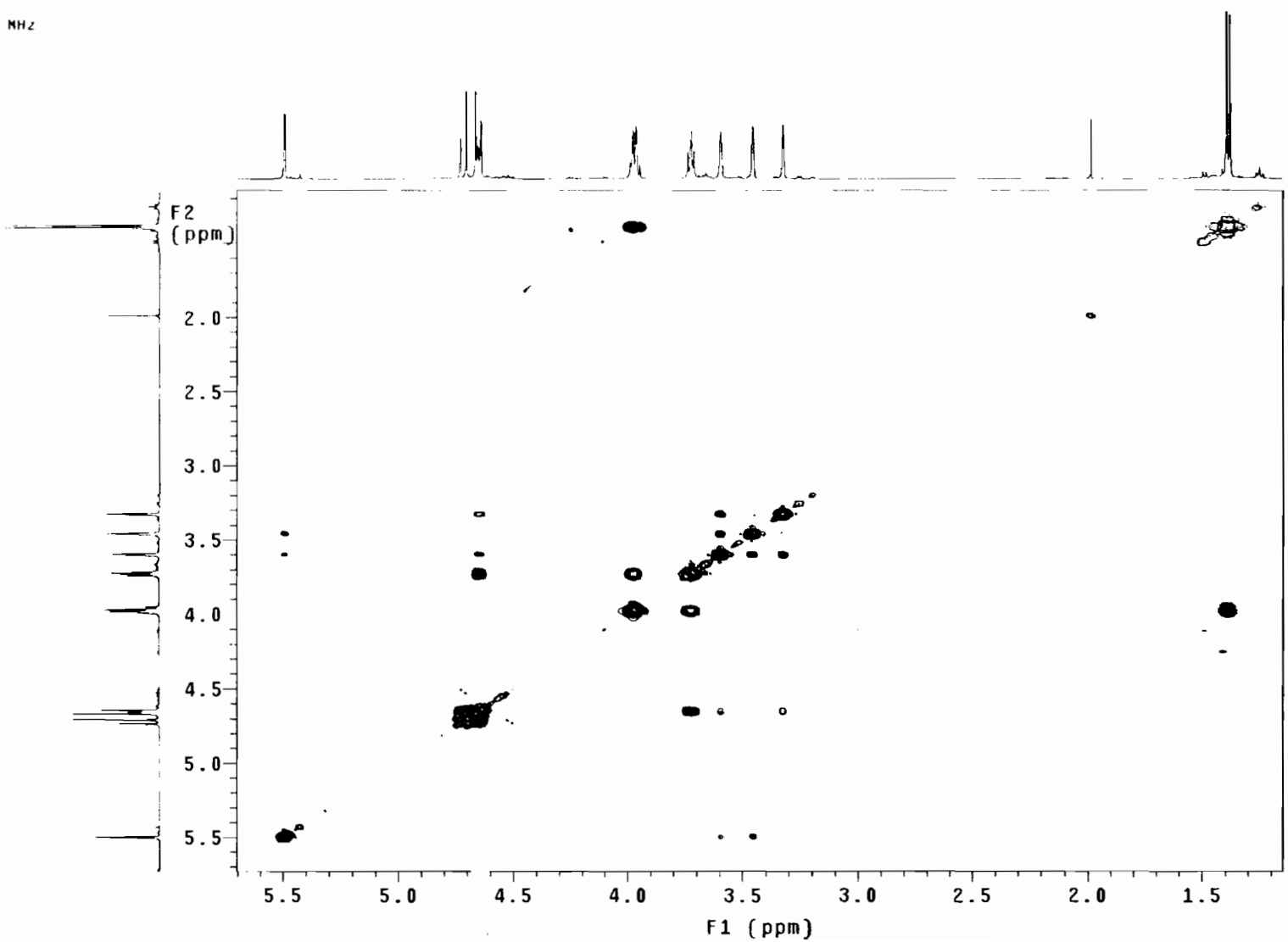


DHL-21

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 3262.2 Hz
2D Width 3262.2 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 1024 x 1024
Total time 51 min, 36 sec

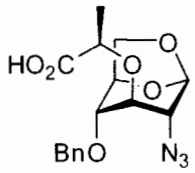


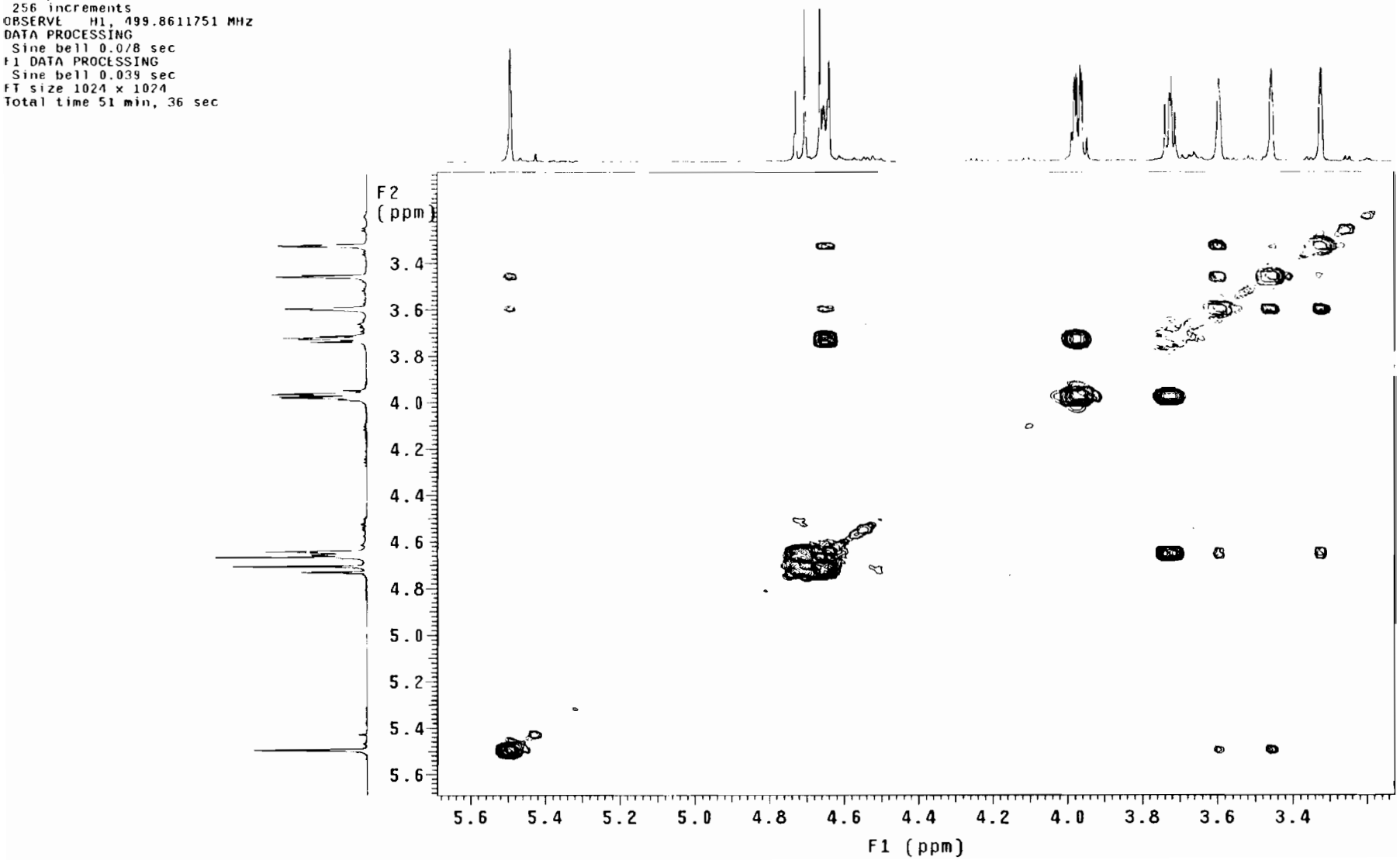


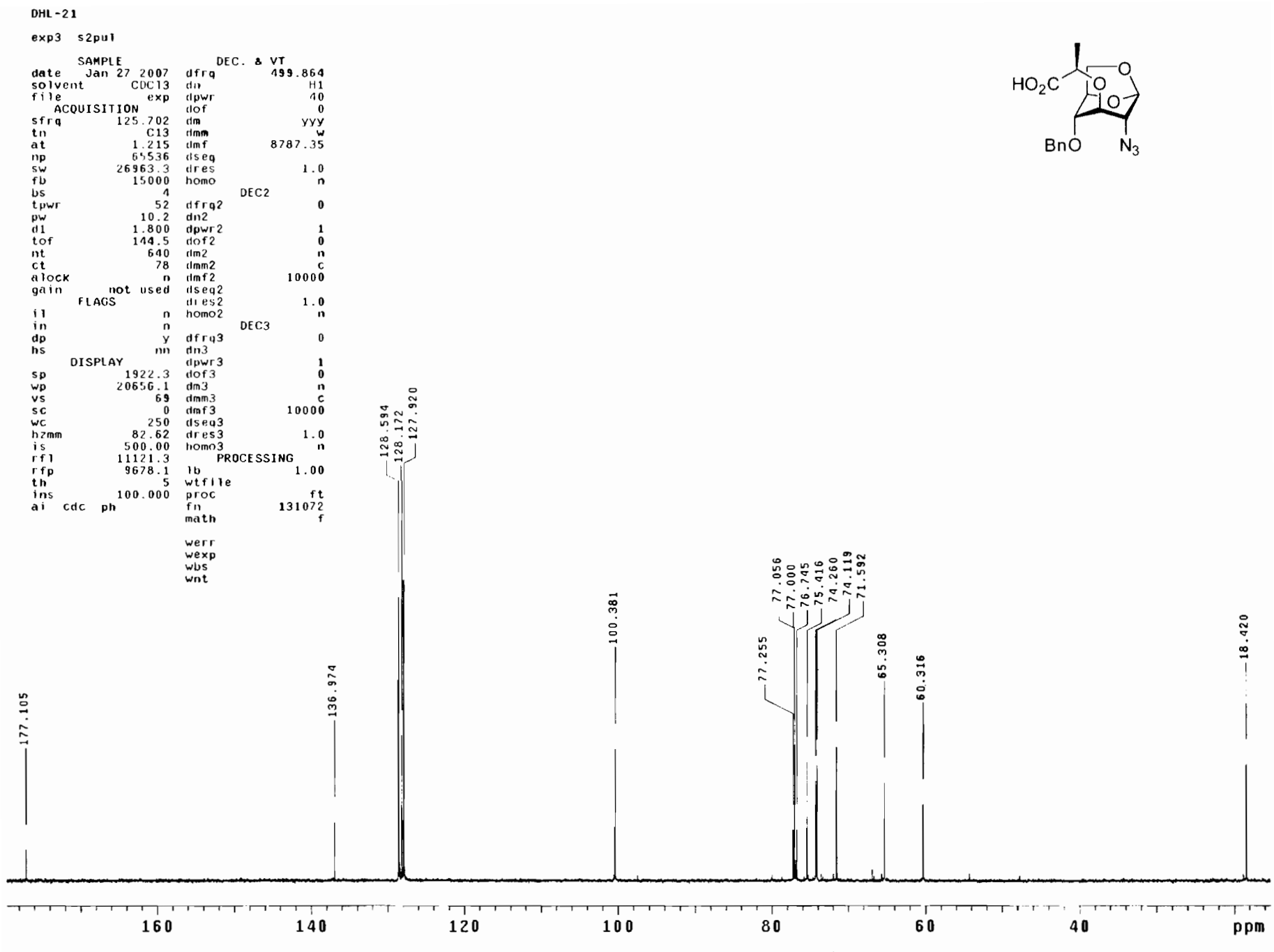
DHL-21

Pulse Sequence: relayh
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 3262.2 Hz
 2D Width 3262.2 Hz
 8 repetitions
 256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 1024 x 1024
Total time 51 min, 36 sec

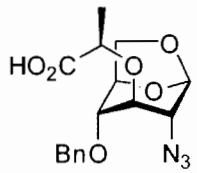





DHL-21
exp3 s2pul
SAMPLE
date Jan 27 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 78
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1922.3
wp 20656.1
vs 69
sc 0
wc 250
hzmm 82.62
is 500.00
rfl 11121.3
rfp 9678.1
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HO2C
O
O
O
BnO
N3
177.105
136.974
128.594
128.172
127.920
100.381
77.255
77.056
77.000
76.745
75.416
74.260
74.119
71.592
65.308
60.316
18.420
160
140
120
100
80
60
40
ppm

DHL-21

Pulse Sequence: dept



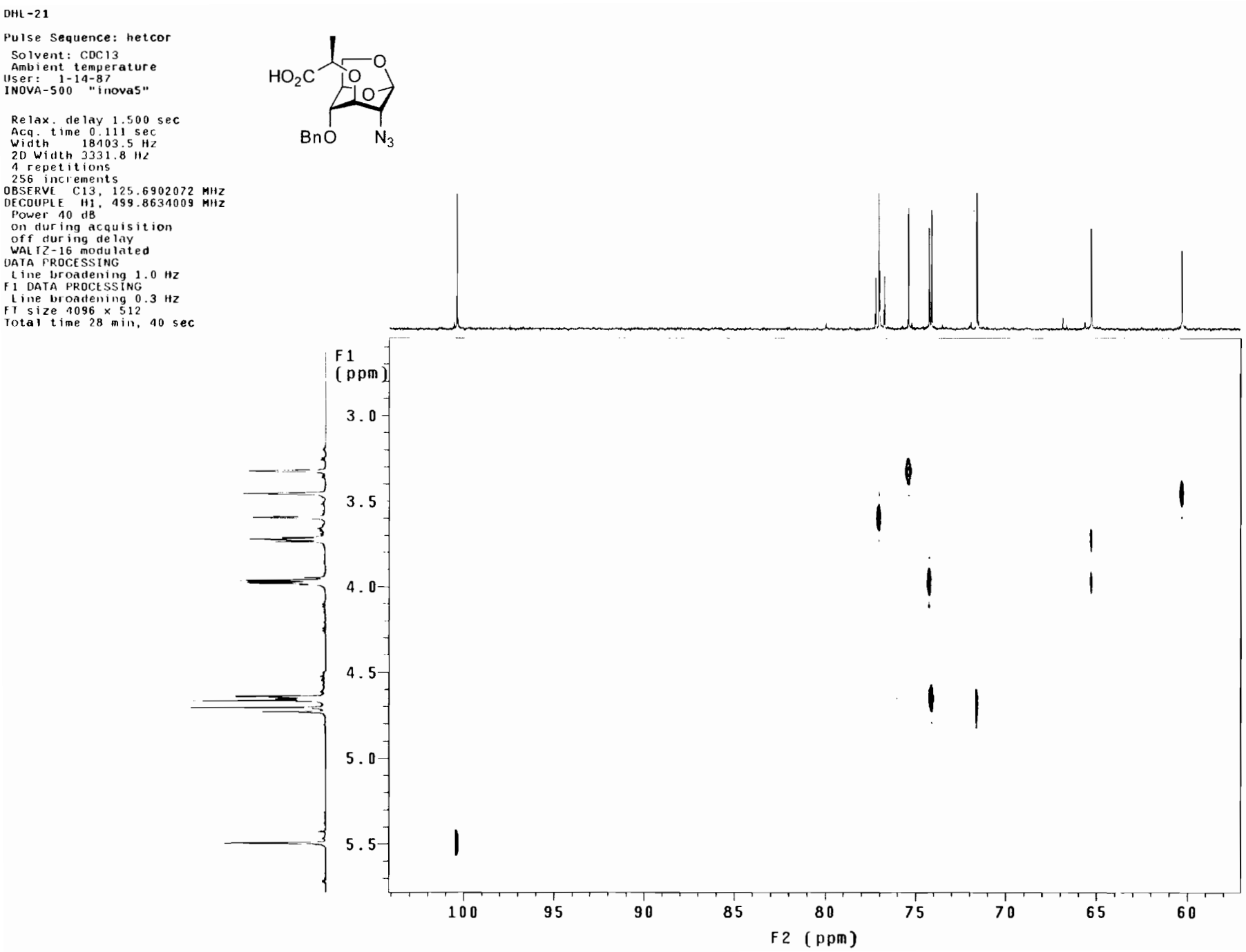
CH3 carbons

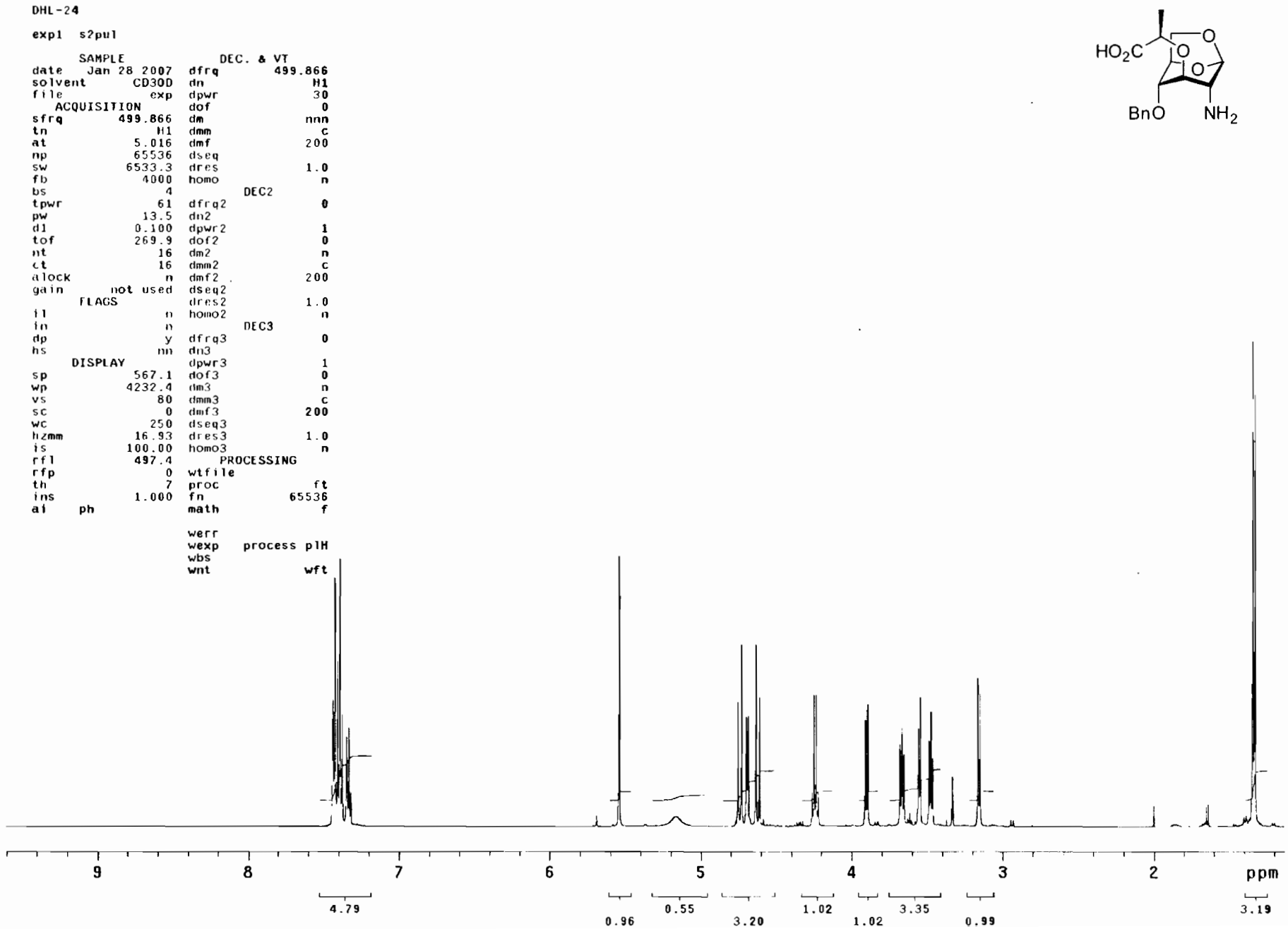
CH2 carbons

CH carbons

all protonated carbons

140 130 120 110 100 90 80 70 60 50 40 30 ppm

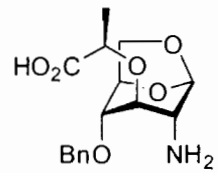

DHL-21
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3331.8 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902072 MHz
DECOUPLE H1, 499.8634009 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 40 sec
HO2C
BnO
N3
F1 (ppm)
3.0
3.5
4.0
4.5
5.0
5.5
100
95
90
85
80
75
70
65
60
F2 (ppm)

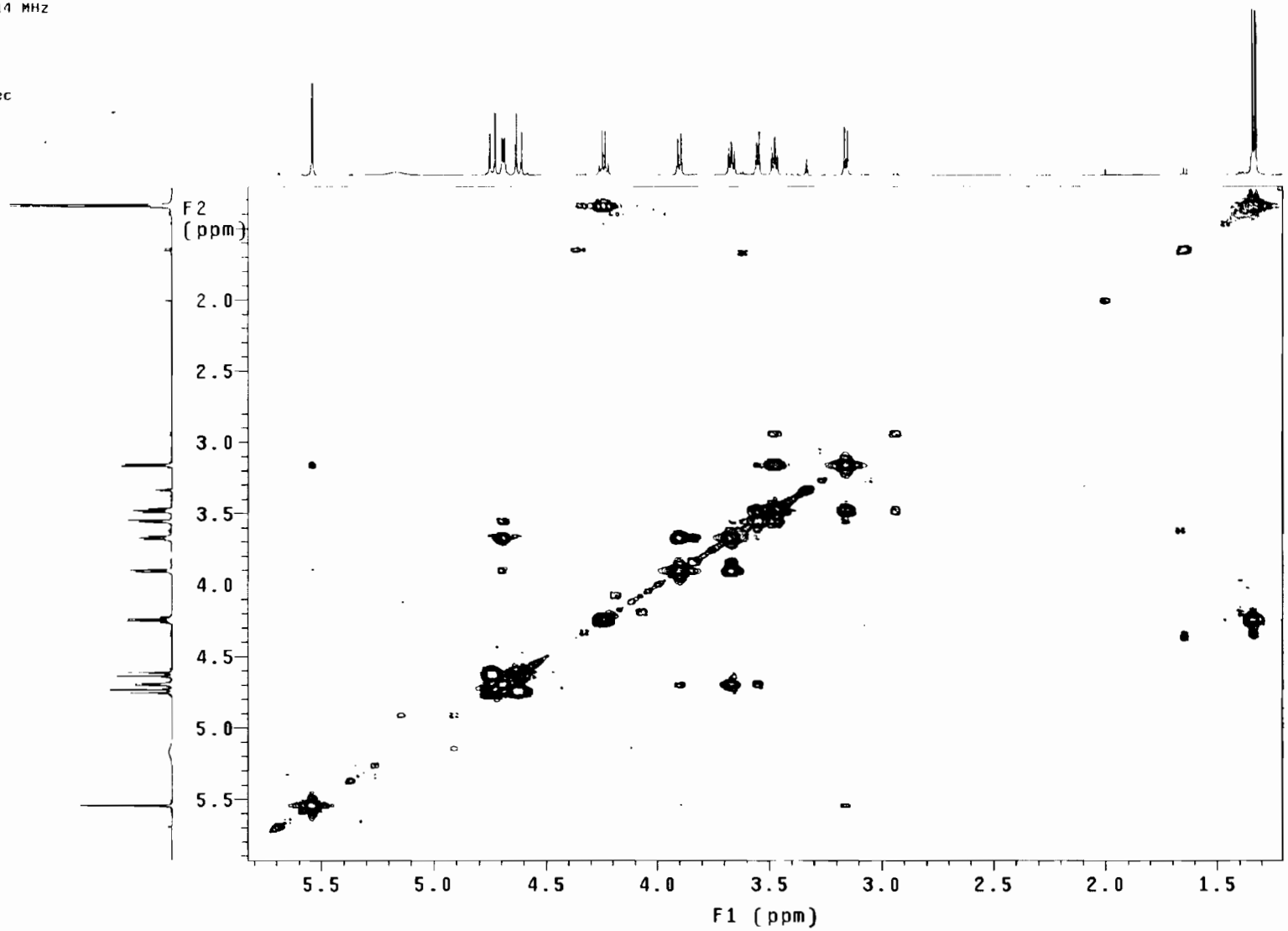

DHL-24
expl s2pul
SAMPLE
date Jan 28 2007
solvent CD3OD
file exp
ACQUISITION
sfrq 499.866
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 567.1
wp 4232.4
vs 80
sc 0
wc 250
hzmm 16.93
is 100.00
rfl 497.4
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
HO2C
O
O
O
BnO
NH2
9
8
7
6
5
4
3
2
ppm
4.79
0.96
0.55
3.20
1.02
1.02
3.35
0.99
3.19

DHL-24

Pulse Sequence: relayh
Solvent: CD3OD
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.159 sec
Width 3219.4 Hz
2D Width 3219.4 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8631314 MHz
DATA PROCESSING
Sine bell 0.080 sec
F1 DATA PROCESSING
Sine bell 0.040 sec
FT size 1024 x 1024
Total time 25 min, 56 sec

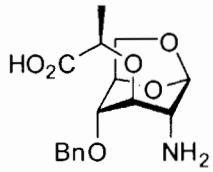


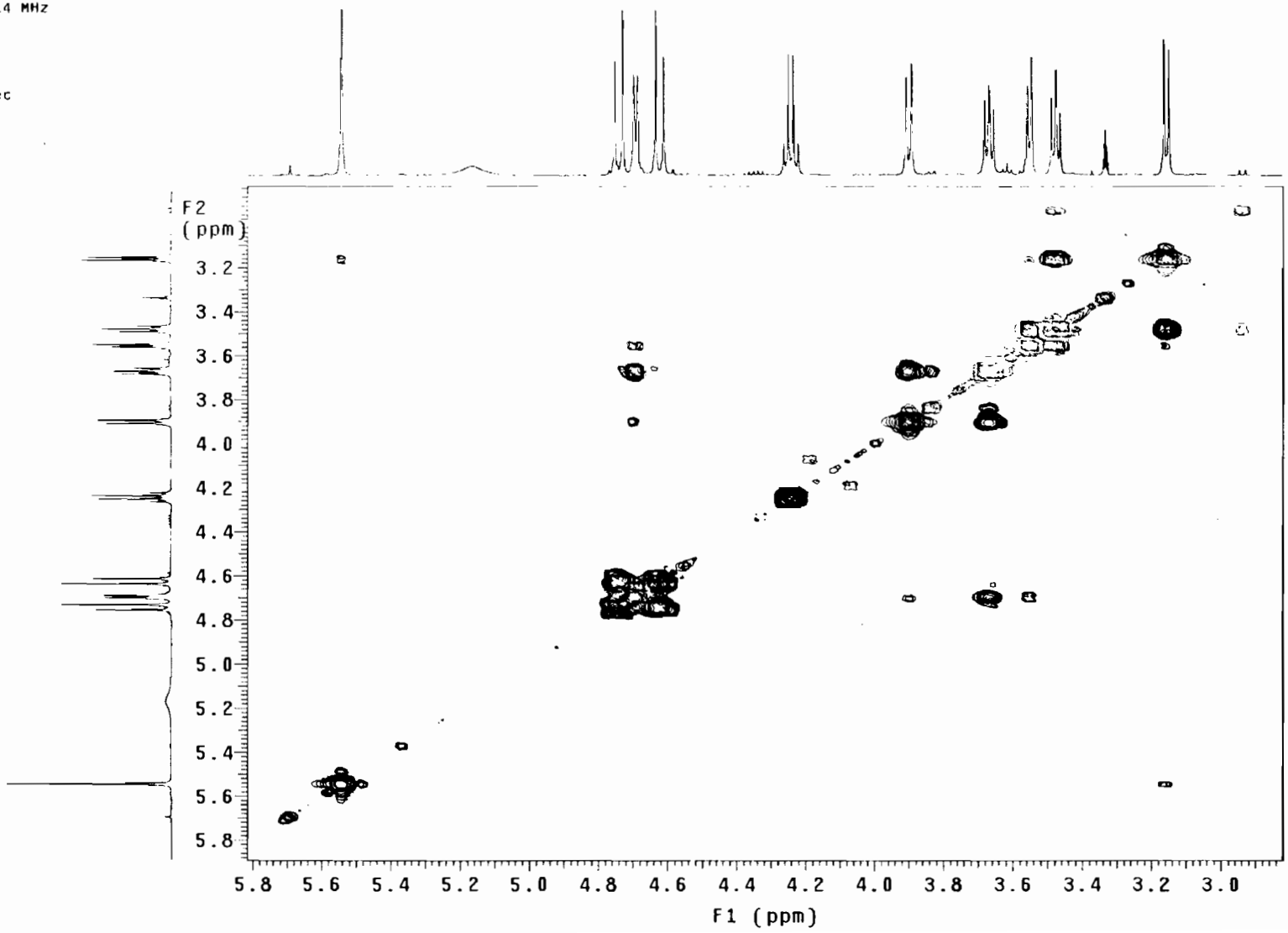


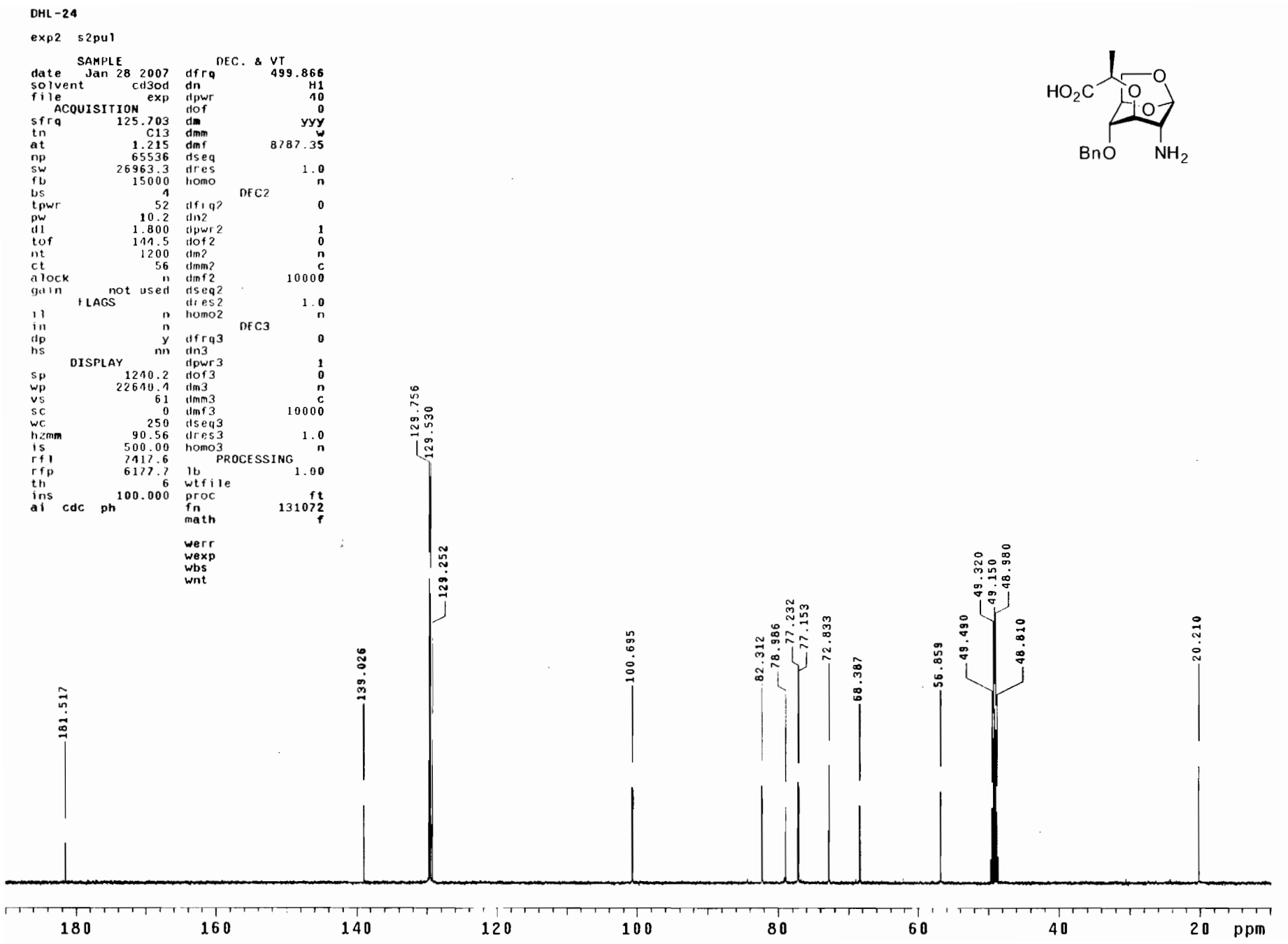
DHL-24

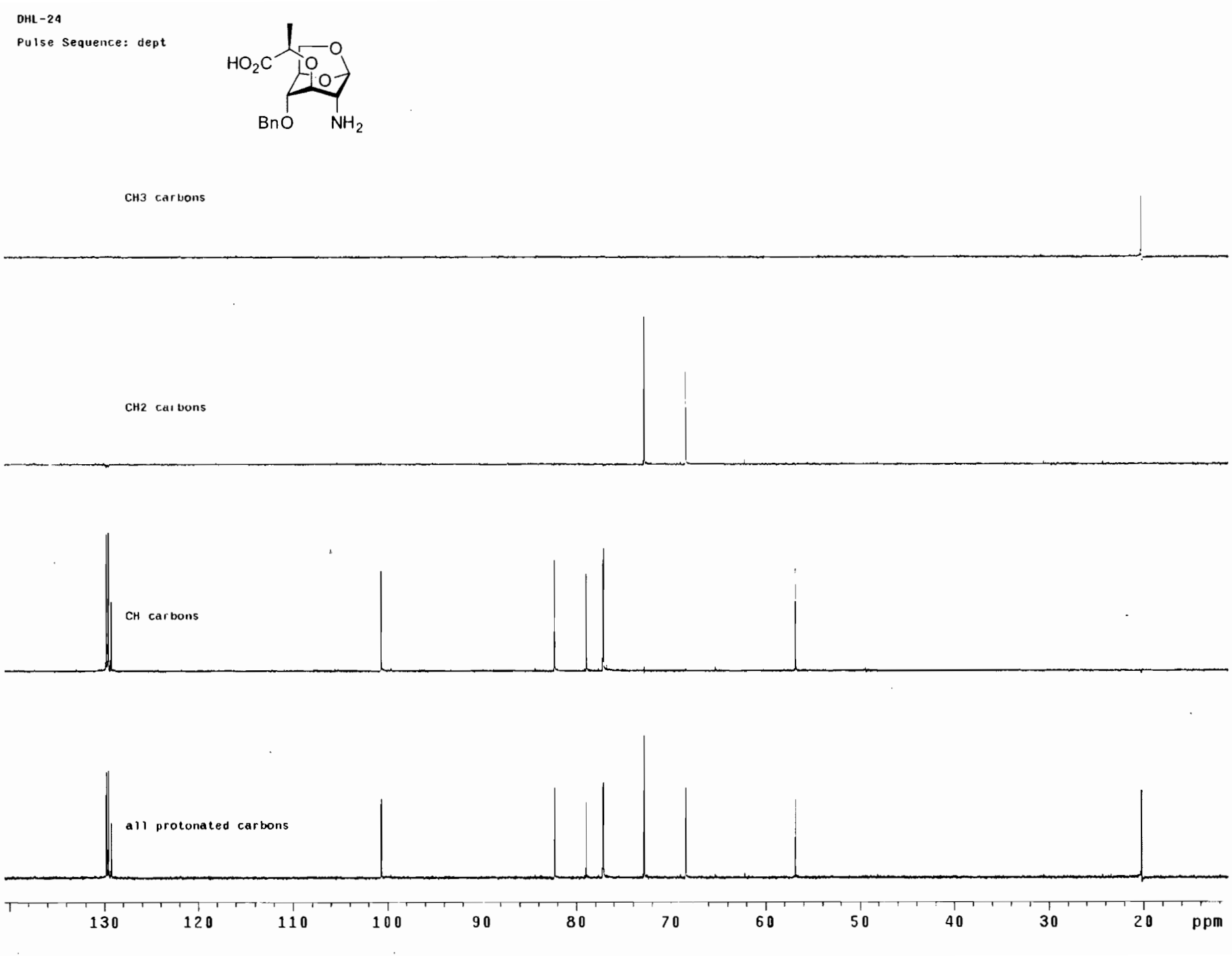
Pulse Sequence: relayh
Solvent: CD3OD
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.159 sec
Width 3219.4 Hz
2D Width 3219.4 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8631314 MHz
DATA PROCESSING
Sine bell 0.080 sec
F1 DATA PROCESSING
Sine bell 0.040 sec
FT size 1024 x 1024
Total time 25 min, 56 sec






DHL-24
exp2 s2pul
SAMPLE
date Jan 28 2007
solvent cd3od
file exp
ACQUISITION
sfrq 125.703
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 56
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1240.2
wp 22640.4
vs 61
sc 0
wc 250
hzmm 90.56
is 500.00
rfl 7417.6
rfp 6177.7
th 6
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
181.517
139.026
129.756
129.530
129.252
100.695
82.312
78.986
77.232
77.153
72.833
68.387
56.859
49.490
49.320
49.150
48.980
48.810
20.210
HO2C
O
O
O
BnO
NH2
180
160
140
120
100
80
60
40
20
ppm

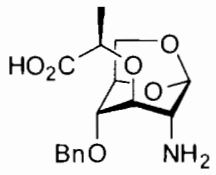

DHL-24
Pulse Sequence: dept
HO2C
O
O
O
BnO
NH2
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm

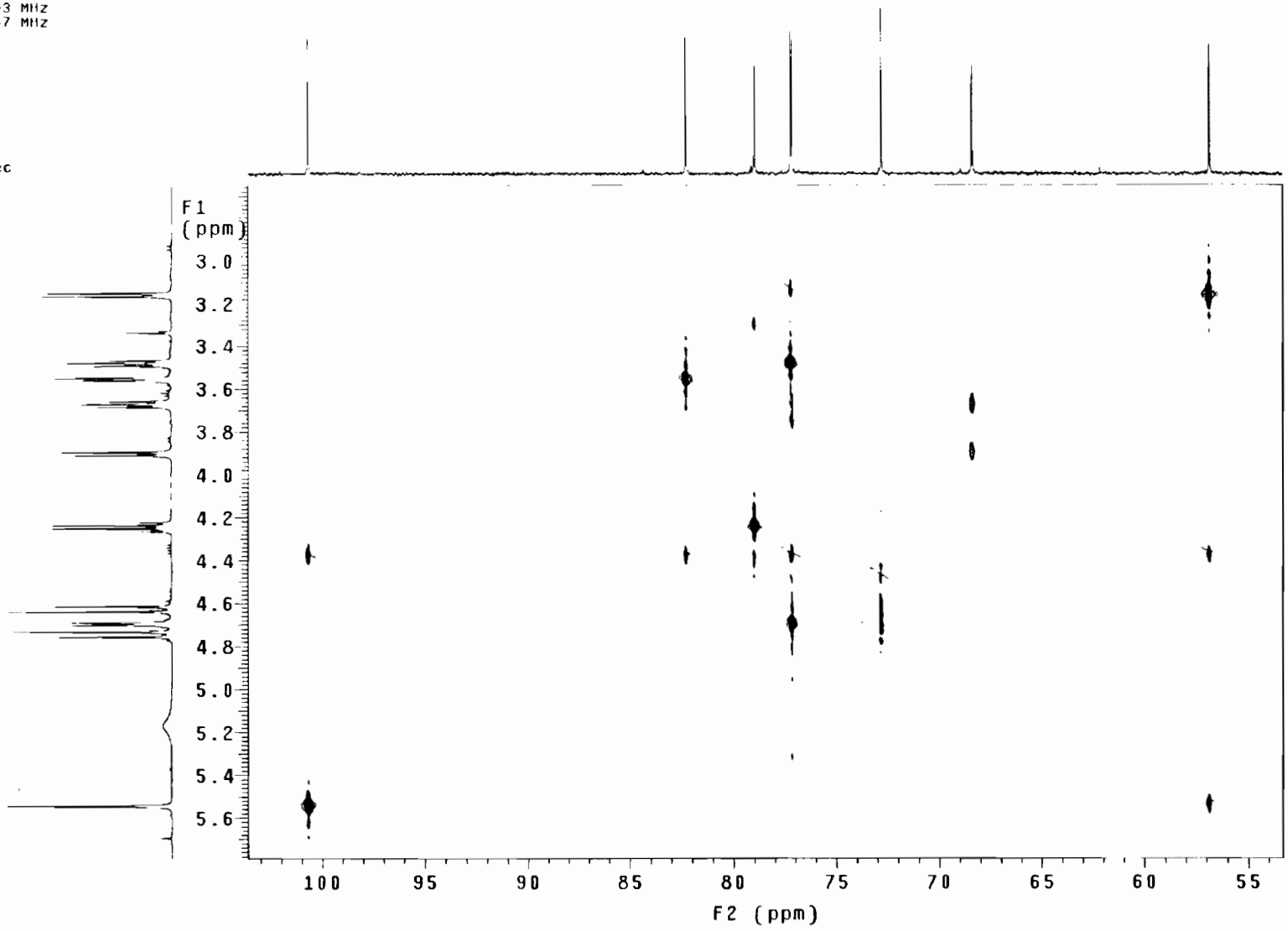
DHL-24

Pulse Sequence: hetcor

Solvent: cd3od
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3236.6 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6904993 MHz
DECOUPLE H1, 499.8653167 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 41 sec



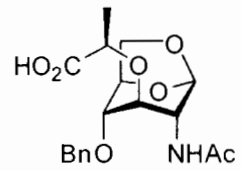


DHL-26-2

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jan 31 2007	dfrq	299.948
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.949	dm	nnn
tn	H1	dmm	c
at	3.620	dmf	200
np	32768	dseq	
sw	4525.4	dres	1.0
fb	2600	homo	n
bs	4	**PROCESSING**	
tpwr	59	wtfile	
pw	14.0	proc	ft
d1	0.380	fn	65536
tof	481.6	math	f
nt	16		
ct	16	werr	
alock	n	wexp	process plH
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	319.2		
wp	3324.1		
vs	151		
sc	0		
wc	250		
hzmm	13.30		
is	125.39		
rfl	281.3		
rfp	0		
th	62		
ins	1.000		

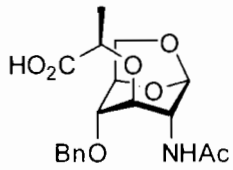
nm cdc ph

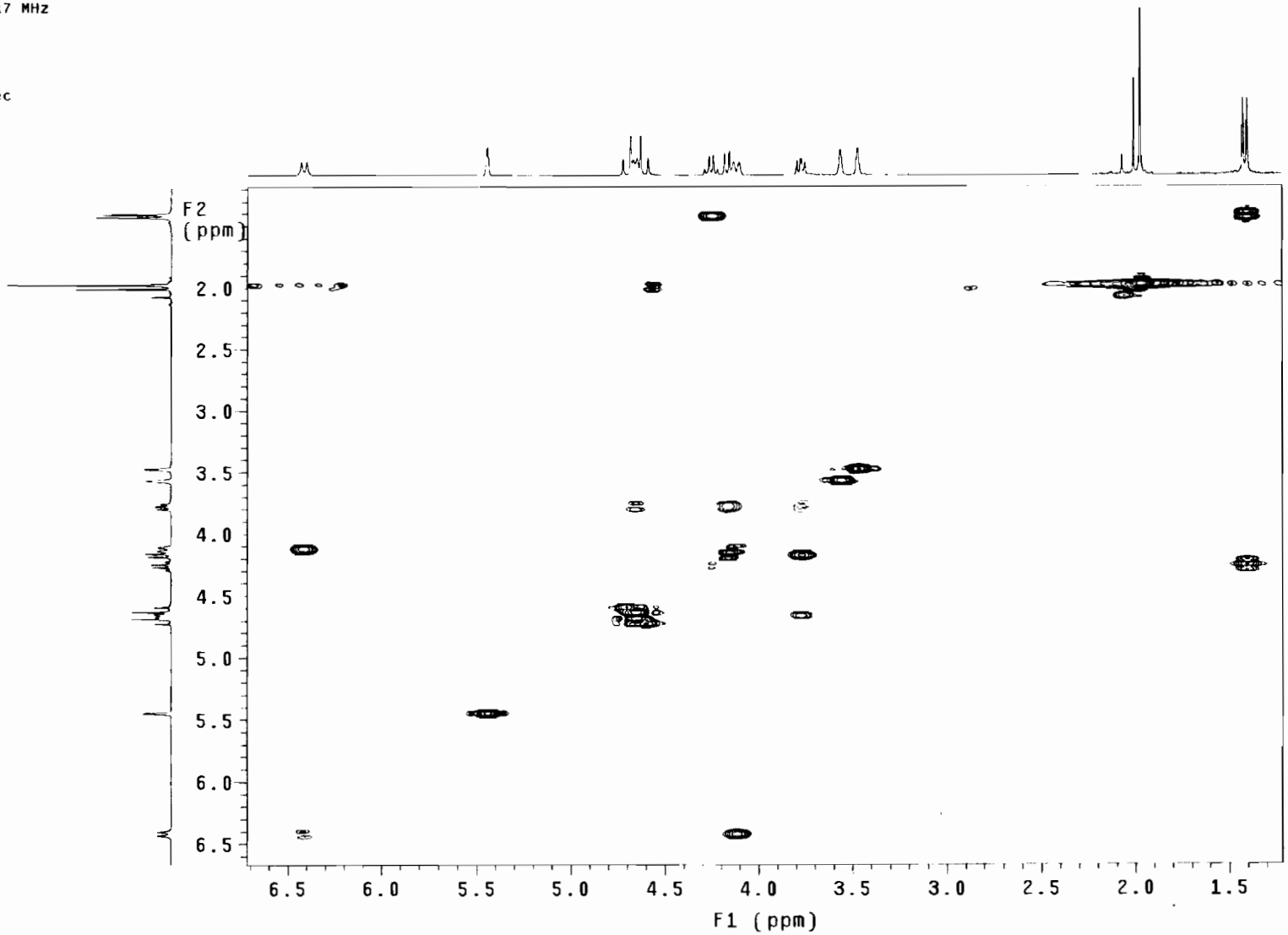


DHL-26-2

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr3a.chem.nd.edu"

Relax. delay 1.400 sec
COSY 90-90
Acq. time 0.125 sec
Width 2045.6 Hz
2D Width 2045.6 Hz
4 repetitions
128 increments
OBSERVE H1, 299.9468517 MHz
DATA PROCESSING
Sine bell 0.063 sec
F1 DATA PROCESSING
Sine bell 0.063 sec
FT size 2048 x 1024
Total time 13 min, 29 sec

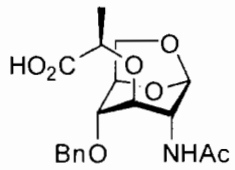


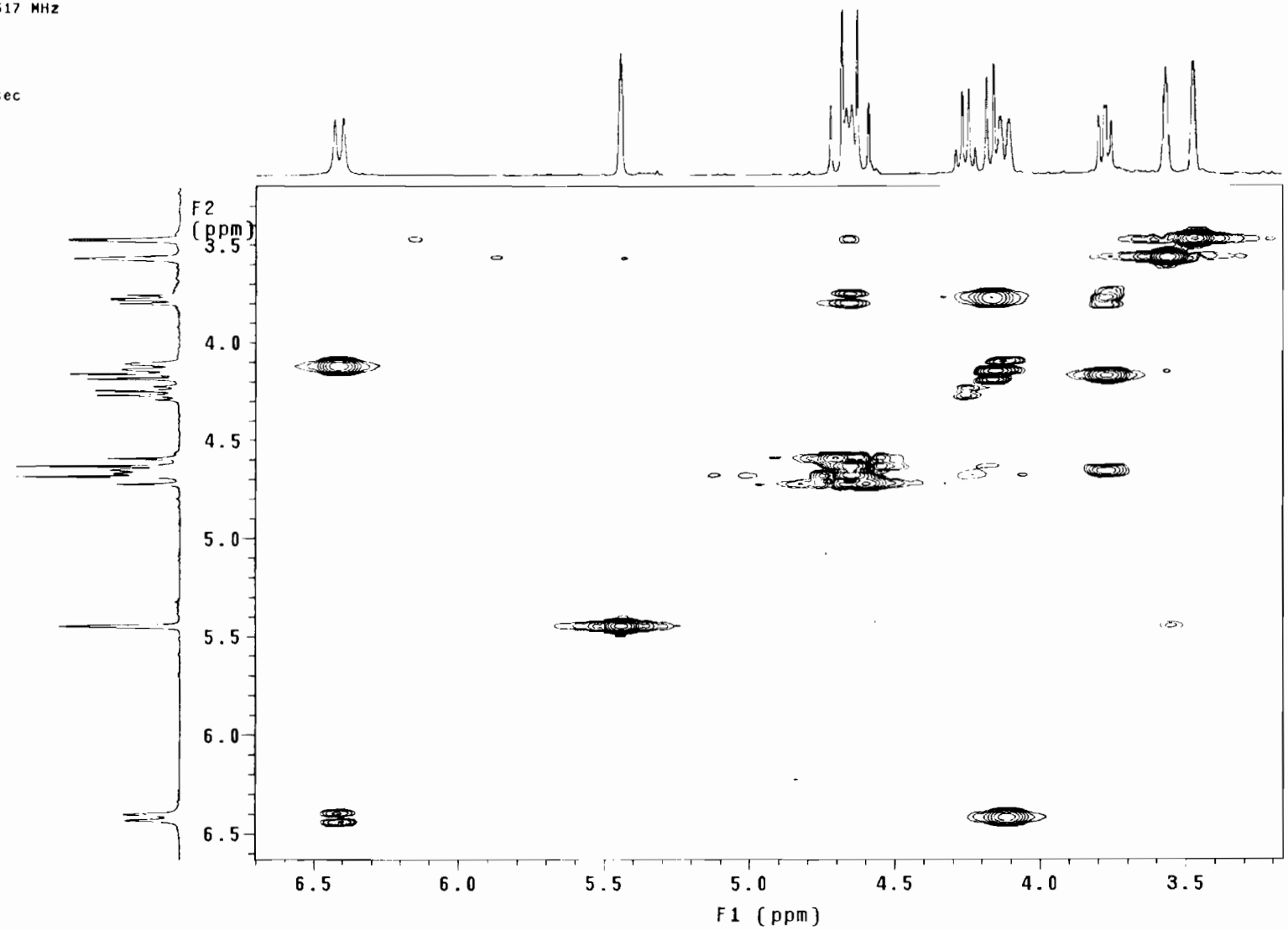


DHL-26-2

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr3a.chem.nd.edu"

Relax. delay 1.400 sec
COSY 90-90
Acq. time 0.125 sec
Width 2045.6 Hz
2D Width 2045.6 Hz
4 repetitions
128 increments
OBSERVE H1, 299.9468517 MHz
DATA PROCESSING
Sine bell 0.063 sec
F1 DATA PROCESSING
Sine bell 0.063 sec
FT size 2048 x 1024
Total time 13 min, 29 sec

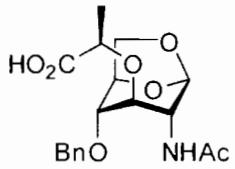


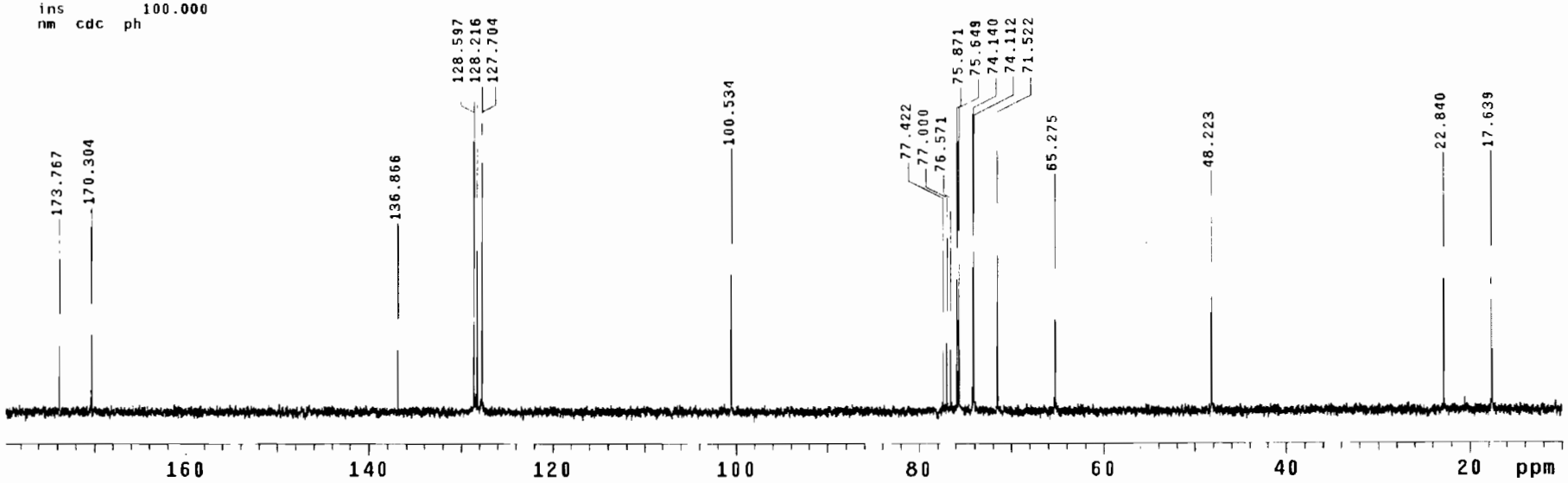


DHL-26-2

exp2 s2pul

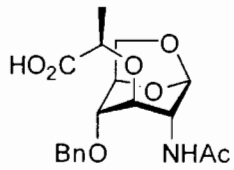
SAMPLE		DEC. & VT	
date	Jan 30 2007	dfrq	299.949
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	272.7
sfrq	75.429	dm	yyy
tn	C13	dmm	w
at	0.957	dmf	8033
np	32768	dseq	
sw	17116.0	dres	1.0
fb	9400	homo	n
bs	4	PROCESSING	
tpwr	59	lb	1.00
pw	8.3	wtfile	
d1	2.000	proc	ft
tof	0	fn	65536
nt	1200	math	f
ct	47		
alock	n	werr	
gain	not used	wexp	process plC
FLAGS		wbs	wft
fl	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	748.0		
wp	12797.1		
vs	52		
sc	0		
wc	250		
hzmm	51.19		
is	500.00		
rfl	6836.0		
rfp	5807.5		
th	7		
ins	100.000		
nm cdc ph			





DHL-26-2

Pulse Sequence: dept



CH3 carbons

CH2 carbons

CH carbons

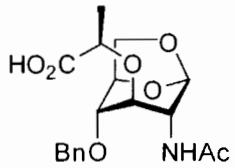
all protonated carbons

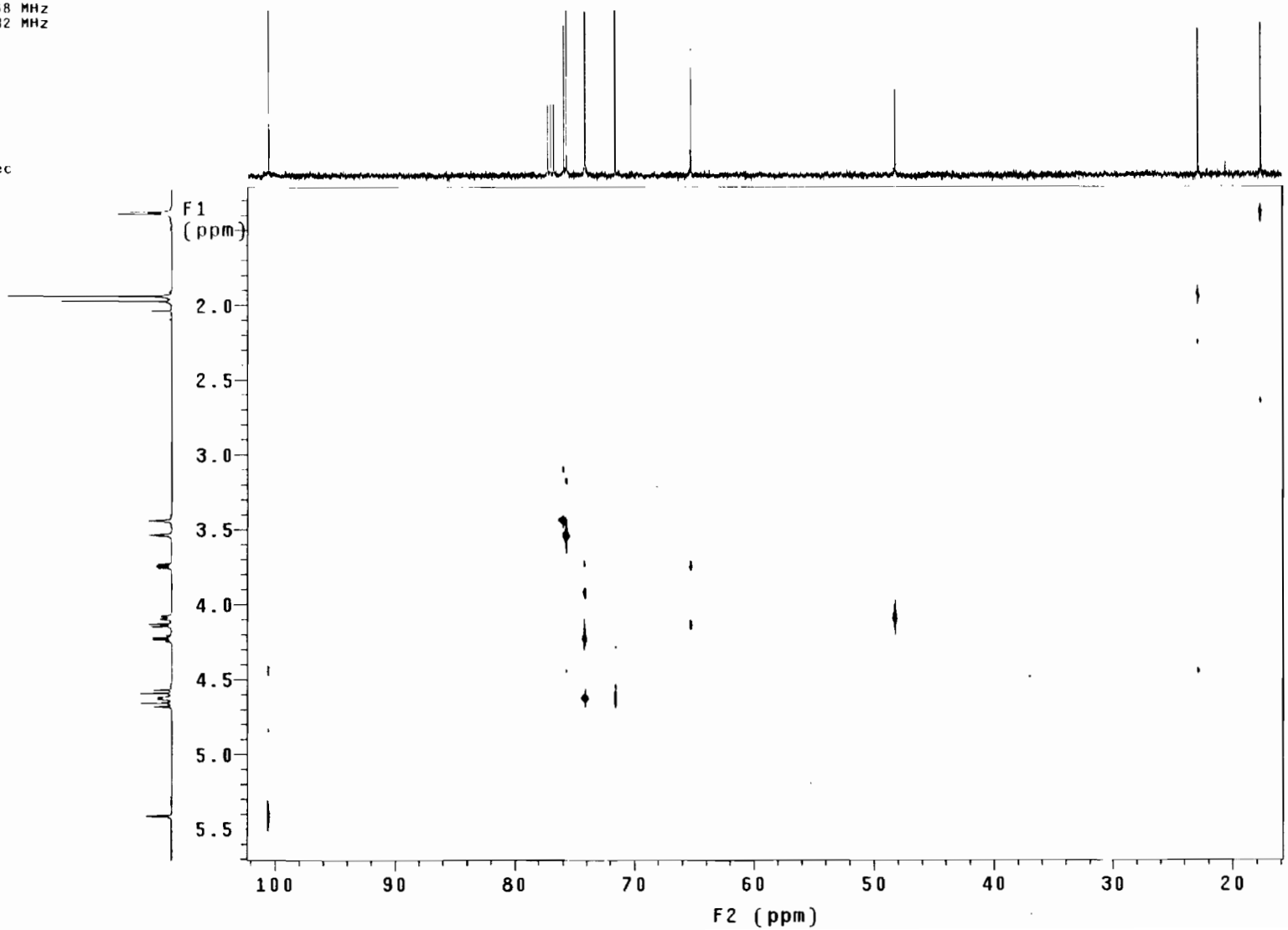
130 120 110 100 90 80 70 60 50 40 30 20 ppm

DHL-26-2

Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3382.4 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6902068 MHz
DECOUPLE H1, 499.8633942 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 57 min, 13 sec

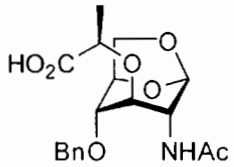


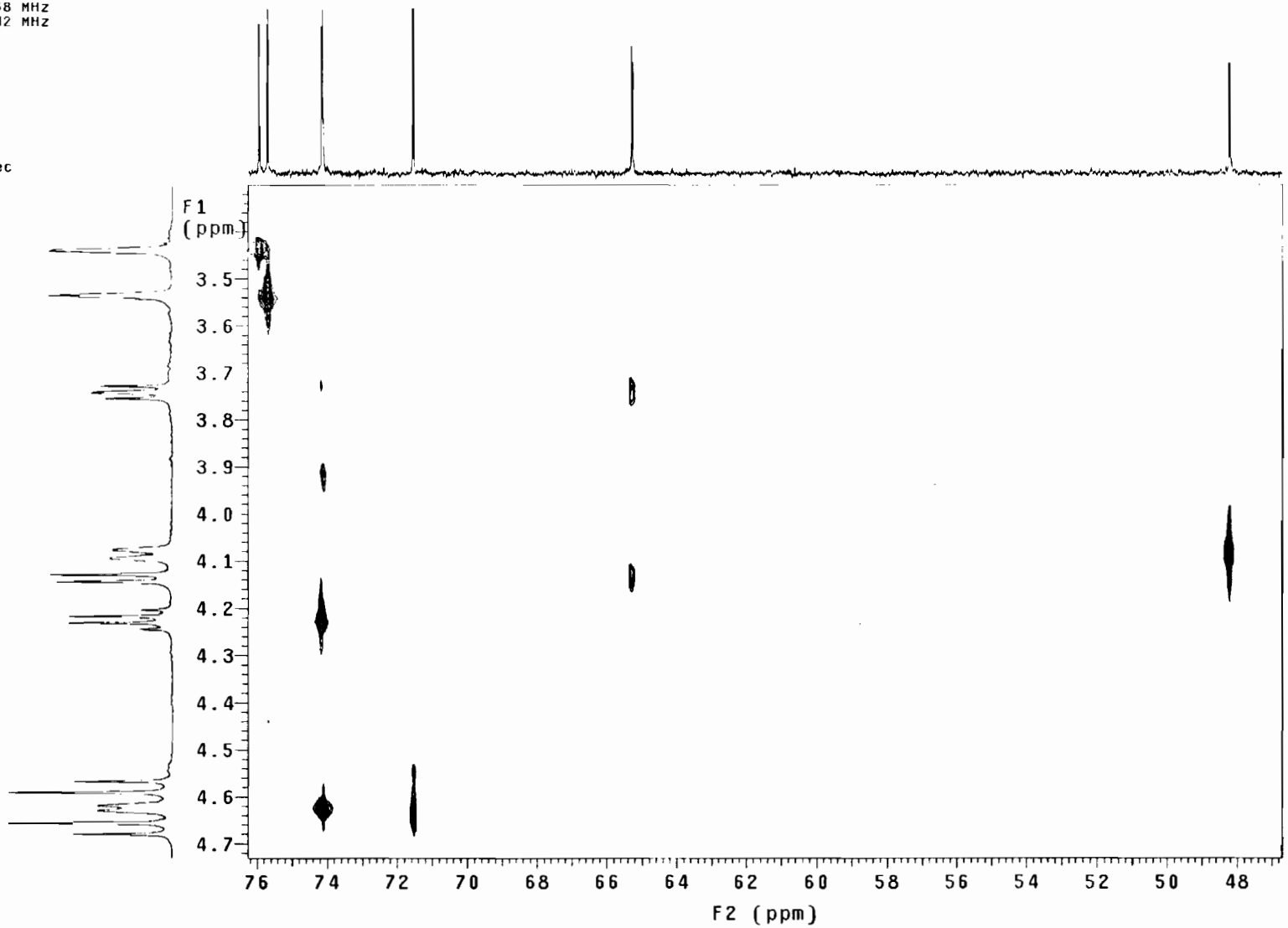


DHL-26-2

Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3382.4 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6902068 MHz
DECOUPLE H1, 499.8633942 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 57 min, 13 sec

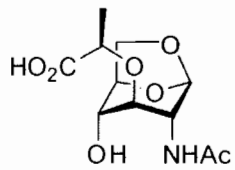




DHL27

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 23 2007	dfrq	499.866
solvent	CD3CN	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.867	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	562.0	dof3	0
wp	3863.2	dm3	n
vs	94	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.45	dres3	1.0
is	145.68	homo3	n
rfl	519.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



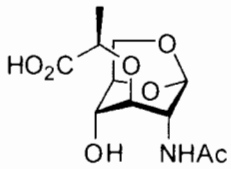
14

DHL27

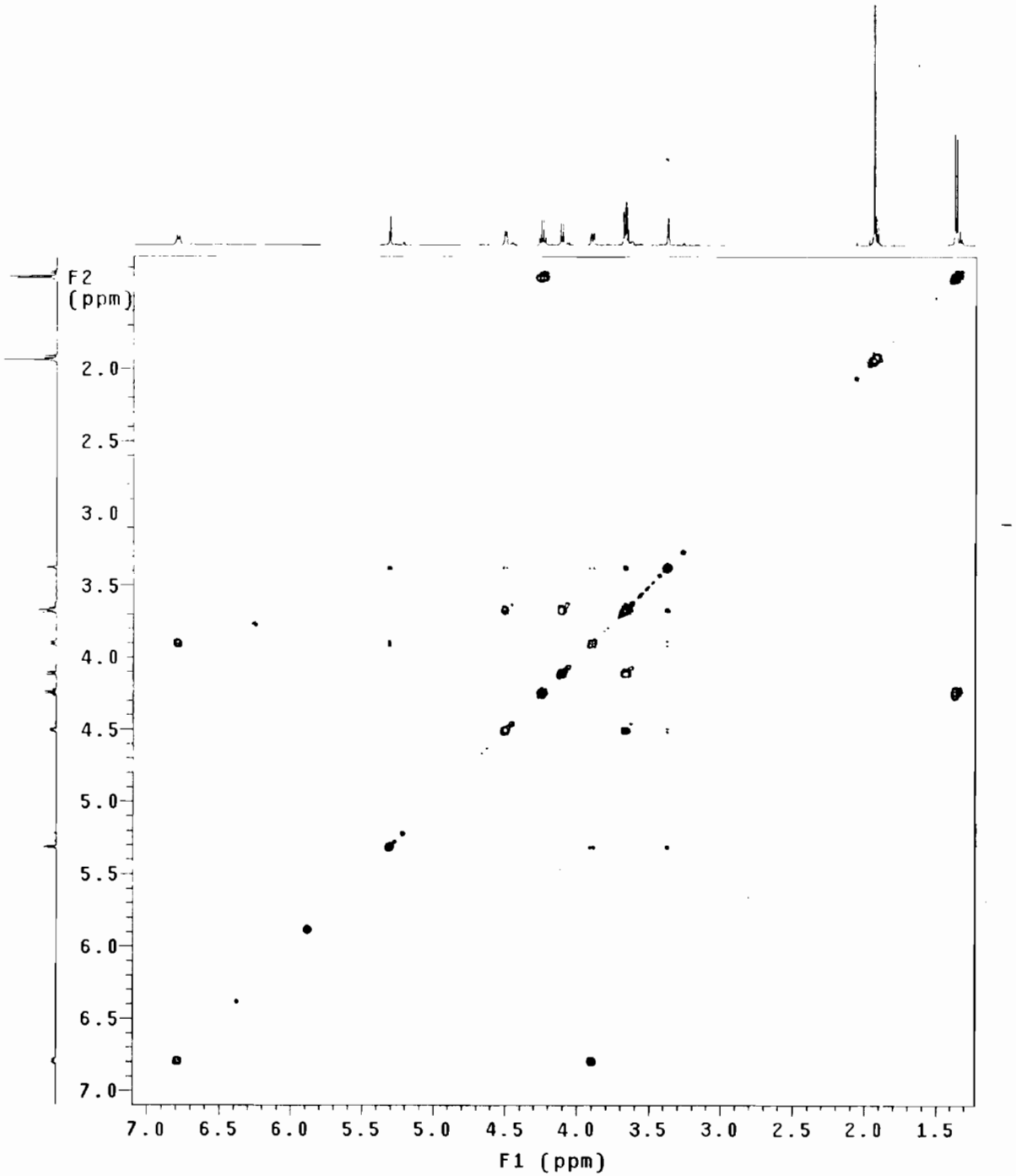
Pulse Sequence: relayh

Solvent: CD3CN
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.174 sec
Width 2939.4 Hz
2D Width 2939.4 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8638284 MHz
DATA PROCESSING
Sine bell 0.087 sec
F1 DATA PROCESSING
Sine bell 0.044 sec
FT size 1024 x 1024
Total time 26 min, 16 sec



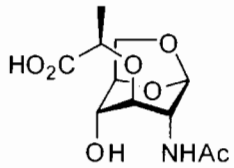
14



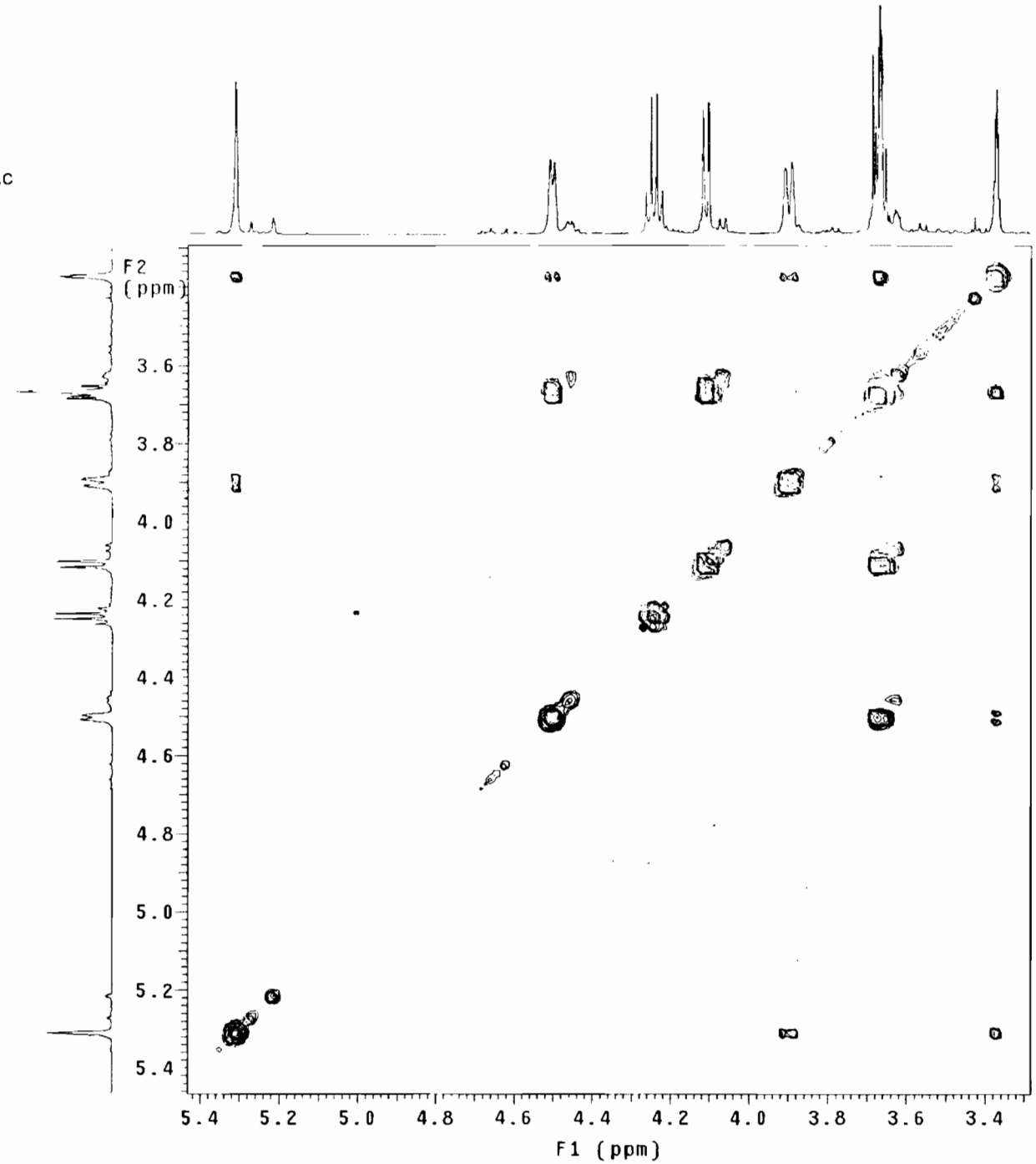
DHL27

Pulse Sequence: relayh
Solvent: CD3CN
Ambient temperature
INOVA-500 "inova5"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.174 sec
Width 2939.4 Hz
2D Width 2939.4 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8638284 MHz
DATA PROCESSING
Sine bell 0.087 sec
FI DATA PROCESSING
Sine bell 0.044 sec
FT size 1024 x 1024
Total time 26 min, 16 sec



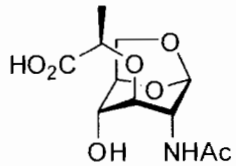
14



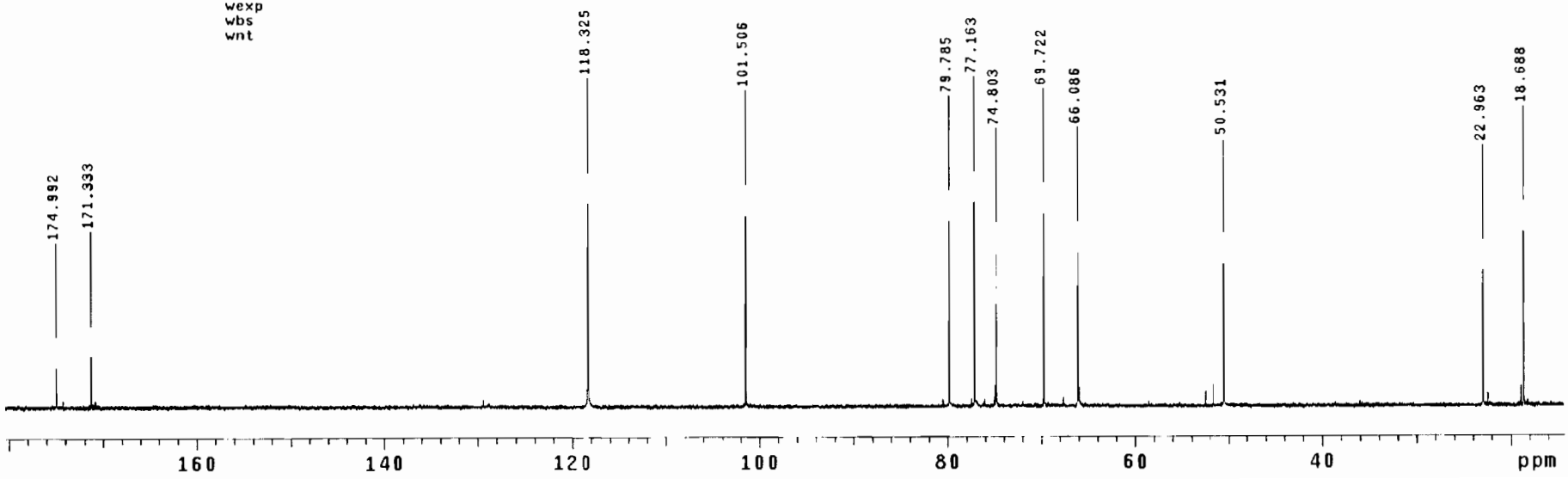
DHL27

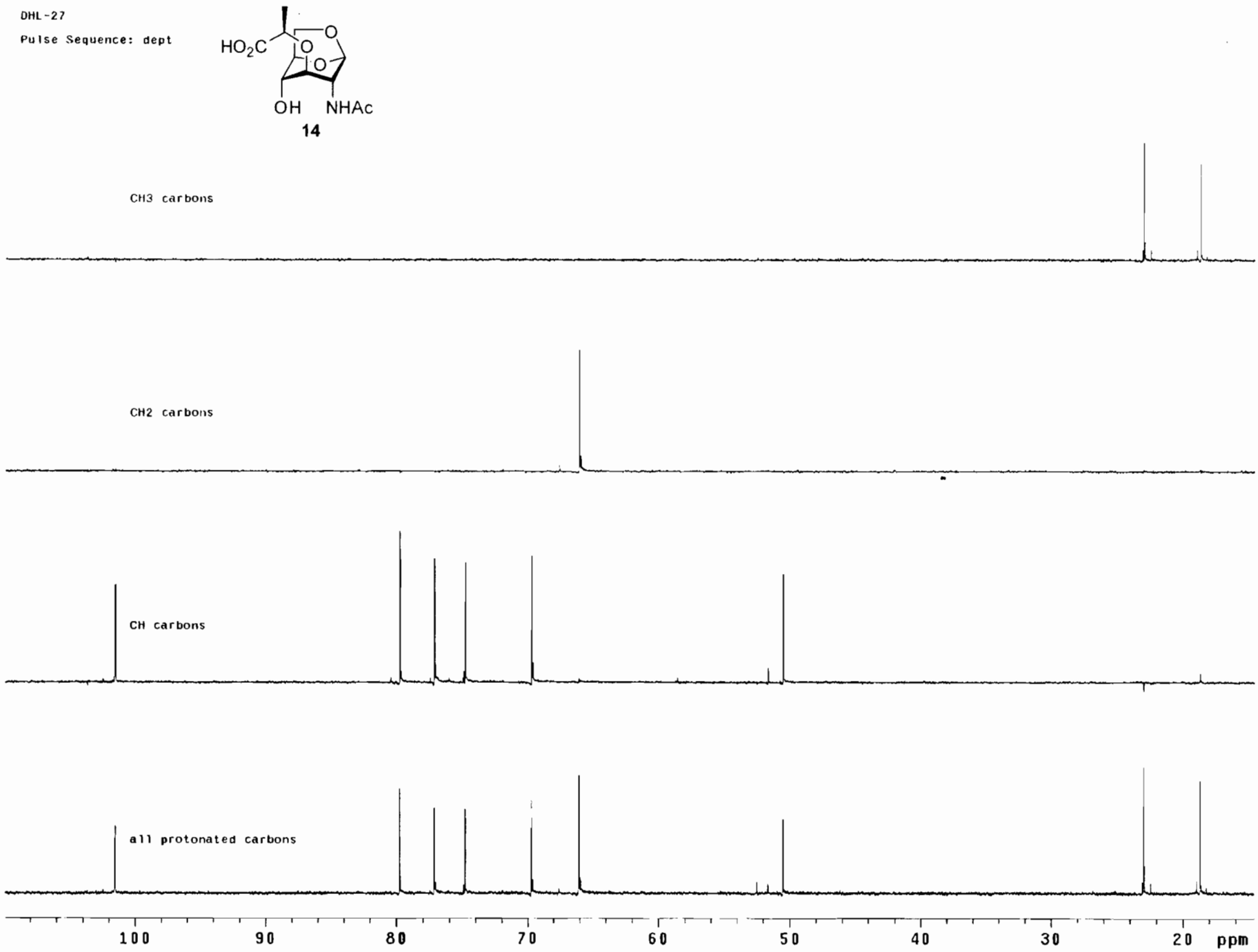
exp2 s2pul

SAMPLE		DEC. & VT	
date	Feb 23 2007	dfrq	499.866
solvent	cd3cn	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1200	dm2	n
ct	81	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1804.6	dof3	0
wp	20868.4	dm3	n
vs	63	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	83.47	dres3	1.0
is	500.00	homo3	n
rfl	1477.0	PROCESSING	
rfp	163.4	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



14

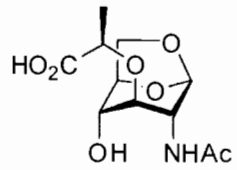



DHL-27
Pulse Sequence: dept
HO2C
O
O
O
OH
NHAc
14
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
100
90
80
70
60
50
40
30
20
ppm

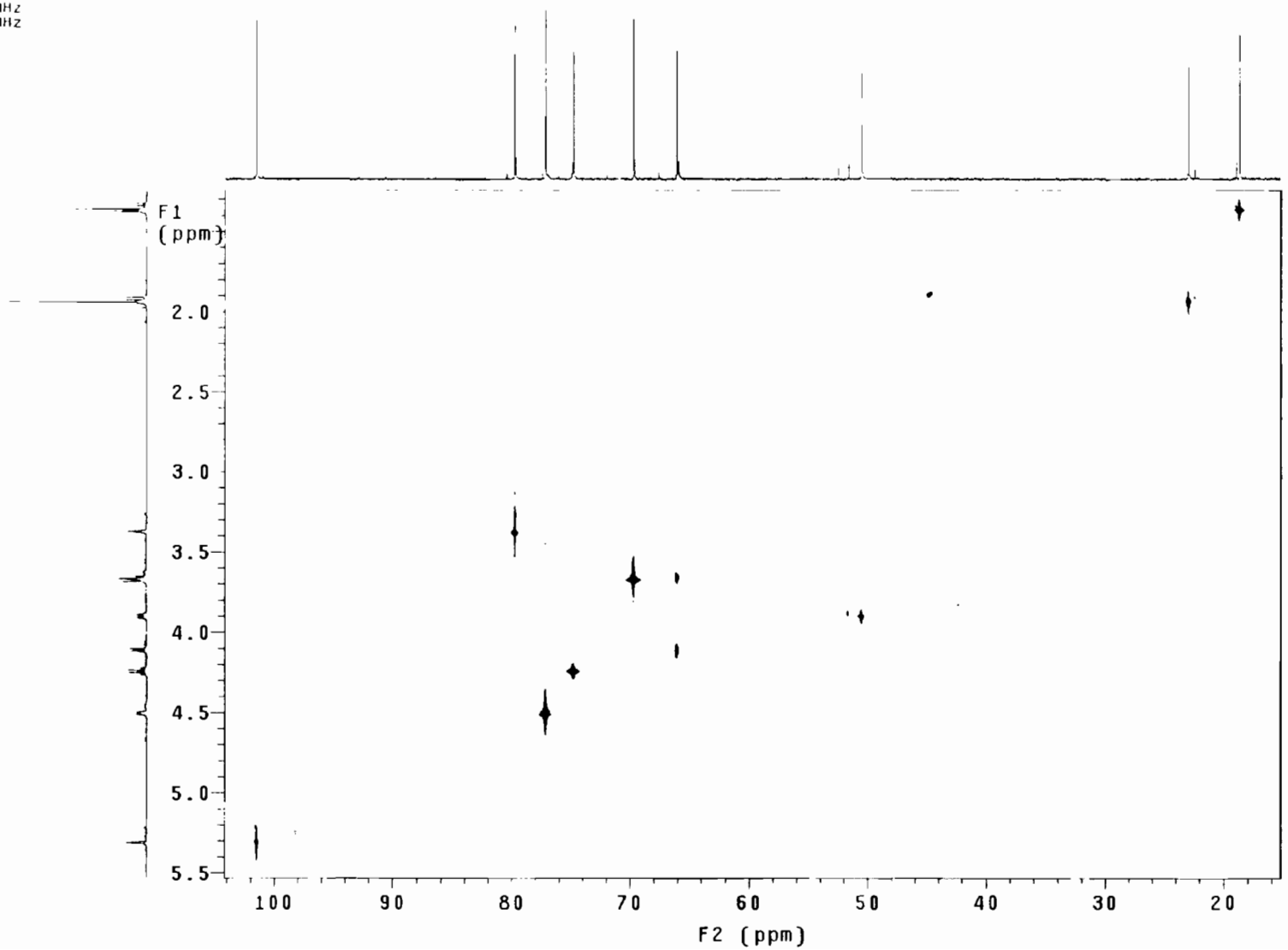
DHL-27

Pulse Sequence: hetcor
Solvent: cd3cn
Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2900.3 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6907427 MHz
DECOUPLE H1, 499.8658787 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 57 min, 26 sec



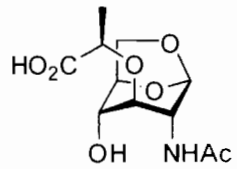
14



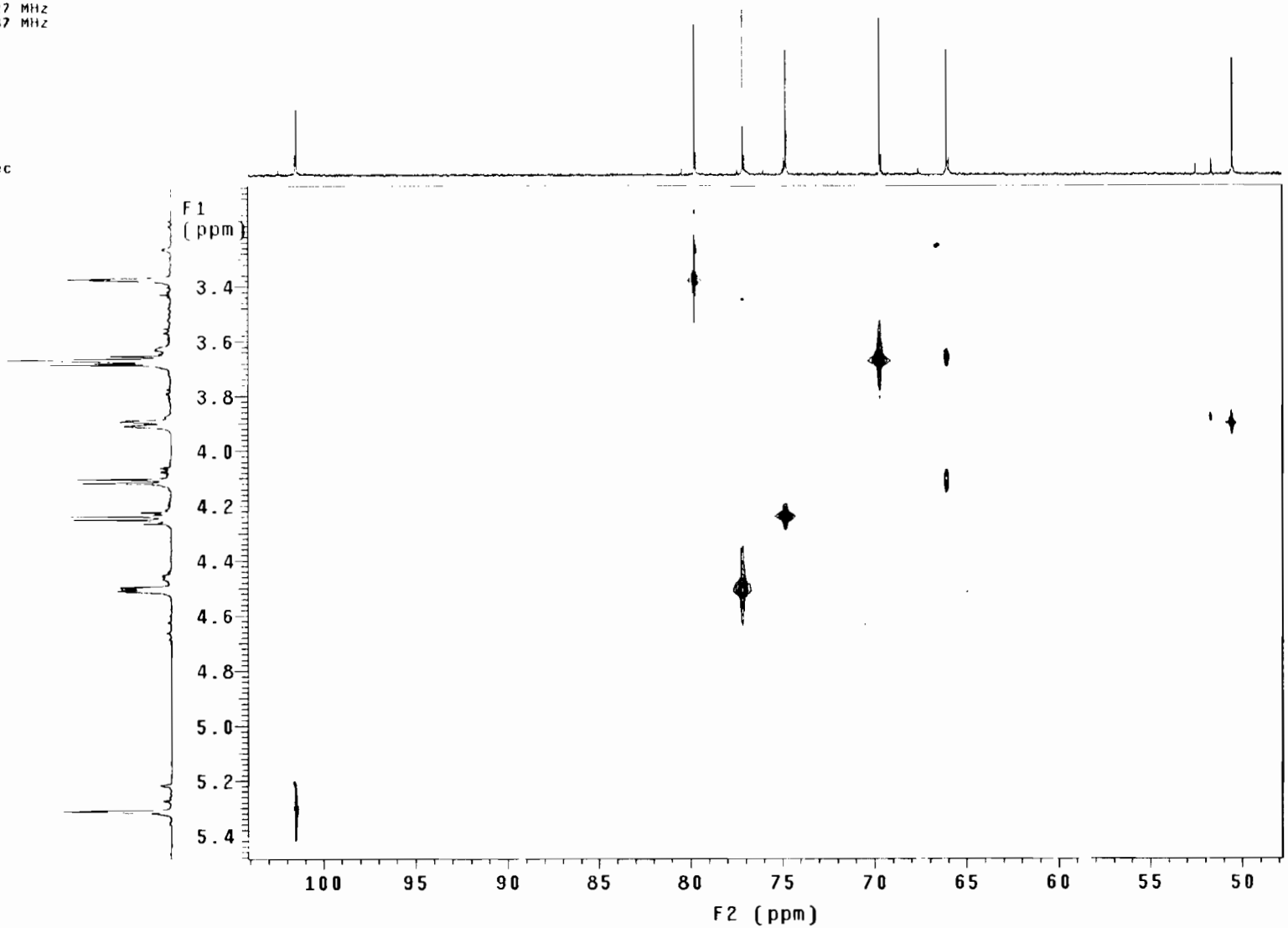
DHL-27

Pulse Sequence: hetcor
 Solvent: cd3cn
 Ambient temperature
User: 1-14-87
INOVA-500 "inova5"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width 18403.5 Hz
 2D Width 2900.3 Hz
 8 repetitions
 256 increments
OBSERVE C13, 125.6907427 MHz
DECOUPLE H1, 499.8658787 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
F1 DATA PROCLSSING
 Line broadening 0.3 Hz
FT size 4096 x 512
Total time 57 min, 26 sec



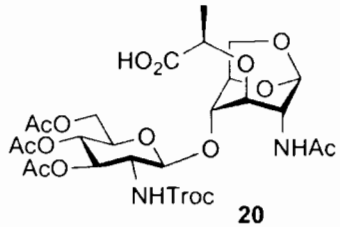
14



DHI-47

exp3 s2pul

SAMPLE		DEC. & VT	
date	Feb 1 2008	dfrq	499.866
solvent	CD3CN	dn	H1
file	/afs/nd.edu/u~	dpwr	30
ser26/dhesek/Priva~		dof	0
te/DHL/DHL-47-H.fi~		dm	nnn
	d	dmm	c
ACQUISITION		dmf	200
sfrq	499.867	dseq	
tn	H1	dres	1.0
at	5.016	homo	n
np	65536	DEC2	
sw	6533.3	dfrq2	0
fb	4000	dn2	
bs	4	dpwr2	1
tpwr	61	dof2	0
pw	13.5	dm2	n
d1	0.100	dmm2	c
tof	269.9	dmf2	200
nt	16	dseq2	
ct	16	dres2	1.0
alock	n	homo2	n
gain	not used	DEC3	
FLAGS		dfrq3	0
il	n	dn3	
in	n	dpwr3	1
dp	y	dof3	0
hs	nn	dm3	n
DISPLAY		dmm3	c
sp	635.2	dmf3	200
wp	3854.4	dseq3	
vs	254	dres3	1.0
sc	0	homo3	n
wc	250	PROCESSING	
hzmm	15.42	wtfile	
is	400.00	proc	ft
rfl	523.8	fn	65536
rfp	0	math	f
th	7		
ins	1.000	werr	
ai ph		wexp	process plH
		wbs	
		wnt	wft



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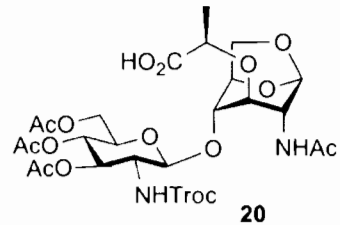
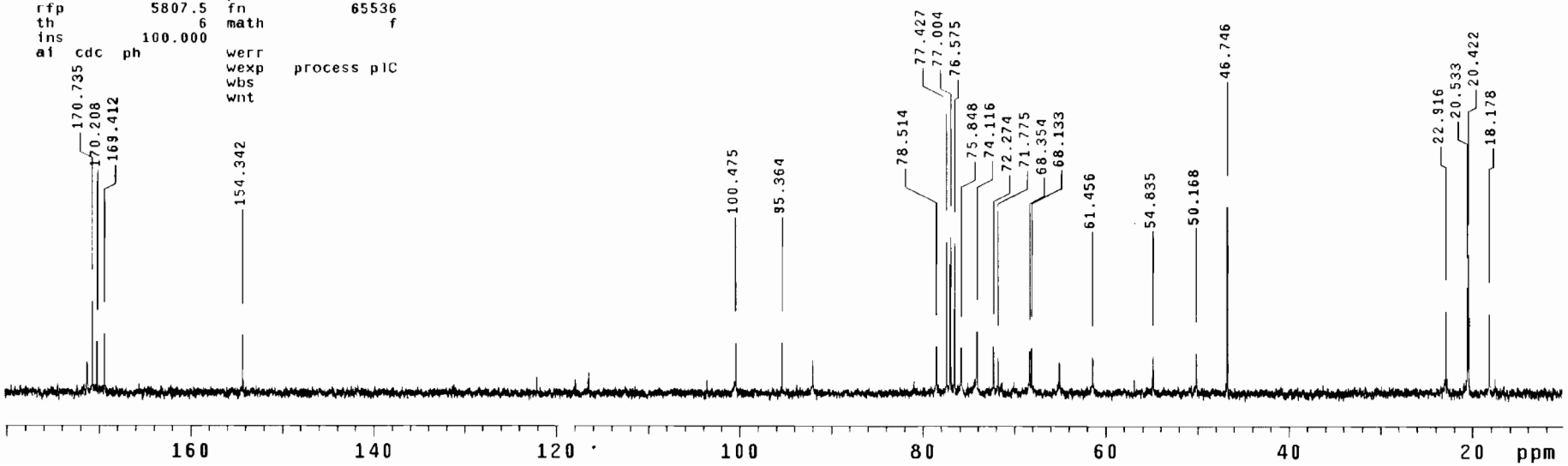
DHL-47

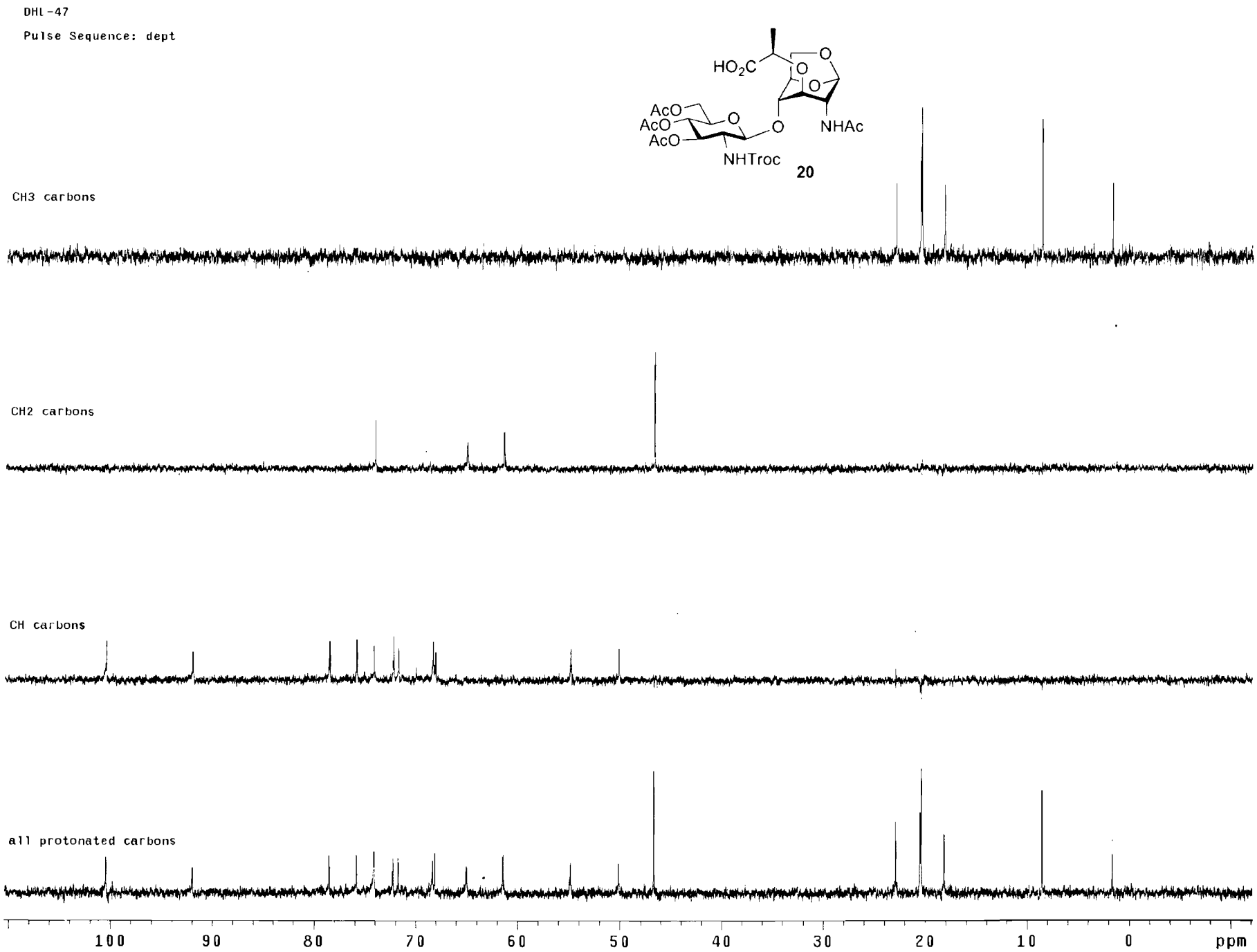
exp3 s2pul

SAMPLE		DEC. & VT	
date	Jan 31 2008	dfrq	299.949
solvent	CDCl3	dn	H1
file	/afs/nd.edu/u~	dpwr	40
ser26/dhesek/Priva~		dof	272.7
te/DHL/DHL-47-C.fi~		dm	yyy
	d	dmm	w
ACQUISITION		dmf	8033
sfrq	75.429	dseq	
tn	C13	dres	1.0
at	0.957	homo	n
np	32768	DEC2	
sw	17116.0	dfrq2	undefined
fb	9400	dn2	undefined
bs	4	dpwr2	undefined
tpwr	59	dof2	undefined
pw	8.3	dm2	undefined
d1	2.000	dmm2	undefined
tof	0	dmf2	undefined
nt	640	dseq2	undefined
ct	172	dres2	undefined
alock	n	homo2	undefined
gain	not used	DEC3	
FLAGS		dfrq3	undefined
il	n	dn3	undefined
in	n	dpwr3	undefined
dp	y	dof3	undefined
hs	nn	dm3	undefined
DISPLAY		dmm3	undefined
sp	770.4	dmf3	undefined
wp	12819.2	dseq3	undefined
vs	585	dres3	undefined
sc	0	homo3	undefined
wc	250	PROCESSING	
hzmm	51.28	lb	1.00
is	500.00	wtfile	
rfl	6836.6	proc	ft
rfp	5807.5	fn	65536
th	6	math	f
ins	100.000		
ai cdc ph		werr	
		wexp	process pIC
		wbs	
		wnt	



20

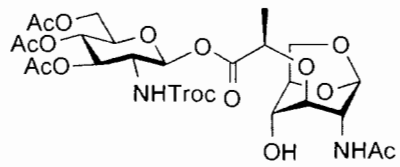



DHL-47
Pulse Sequence: dept
HO2C
AcO
AcO
AcO
NHTroc
NHAc
20
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
100
90
80
70
60
50
40
30
20
10
0
ppm

DHL-48_3

exp1 Proton

SAMPLE		SPECIAL	
date	Feb 17 2008	temp	22.0
solvent	cd3od	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	9615.4	pw90	11.100
at	3.408	alfa	10.000
np	65536	FLAGS	
fb	4000	il	n
bs	4	in	n
ss	2	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	131072
TRANSMITTER		DISPLAY	
tn	H1	sp	777.2
sfrq	599.879	wp	4561.9
tof	599.8	rfl	1208.5
tpwr	61	rfp	0
pw	11.100	rp	-56.3
DECOUPLER		lp	5.8
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	184
dpwr	38	th	50
dmf	35088	ai cdc ph	

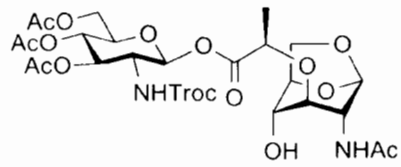


23

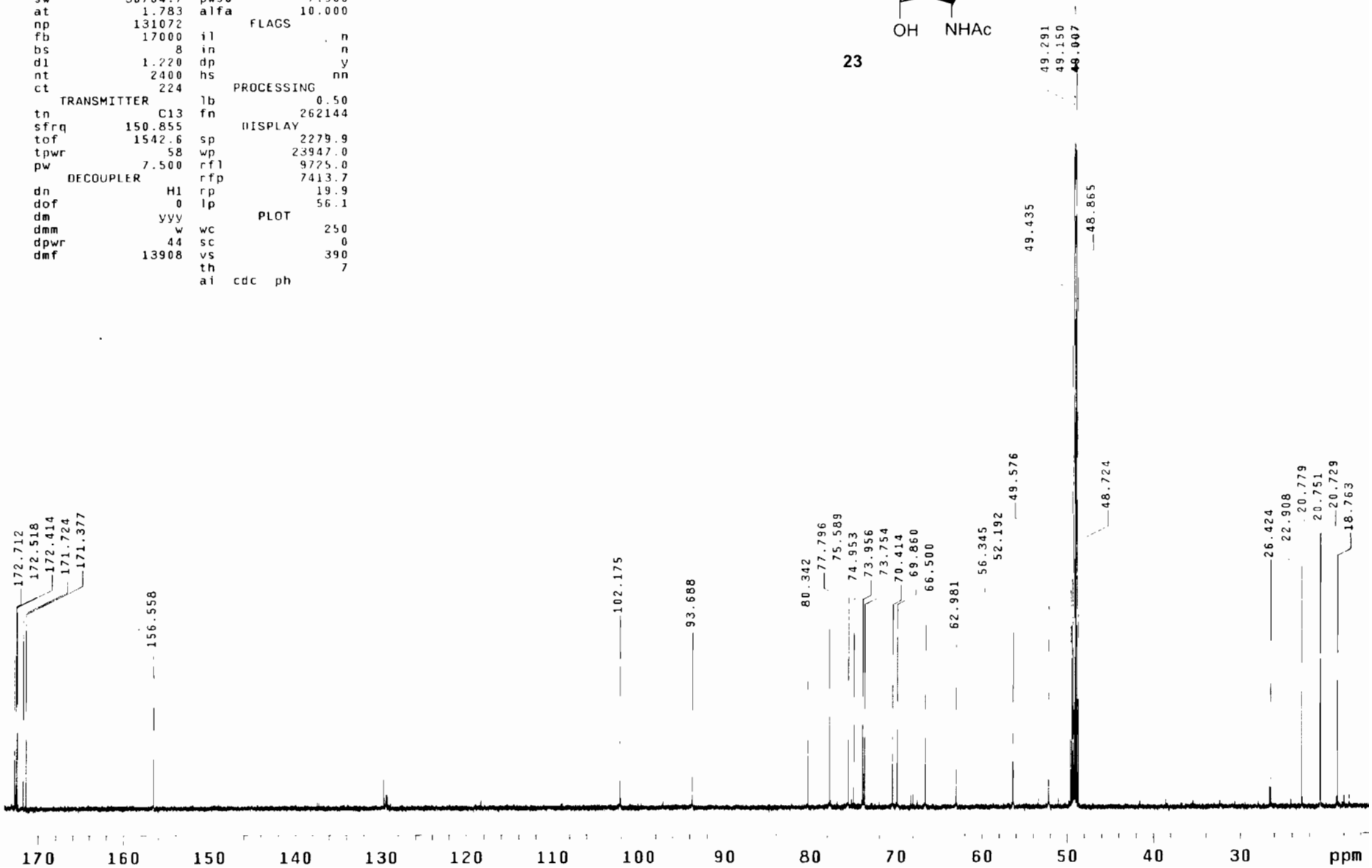
DHL-48_3

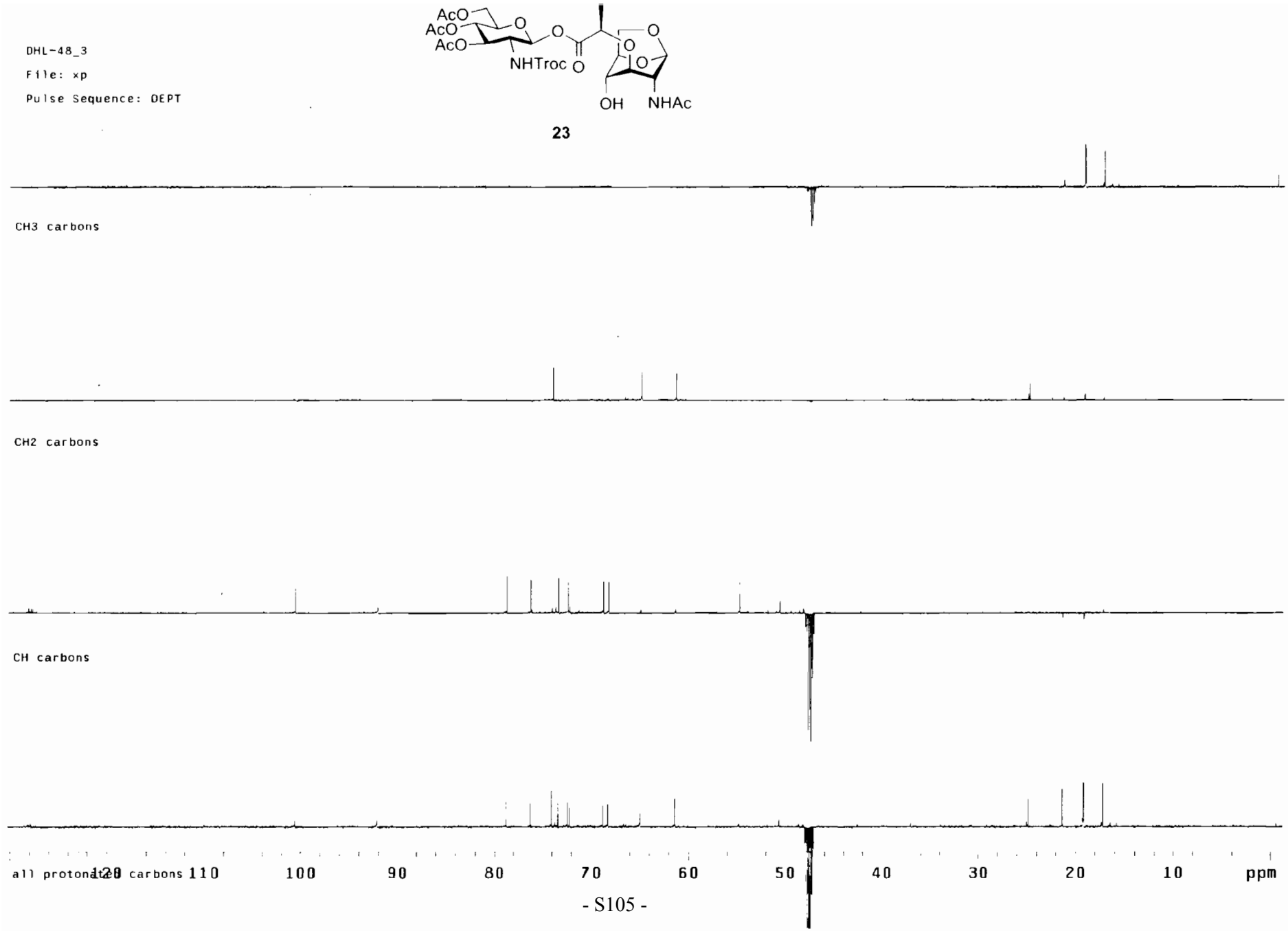
exp2 Carbon

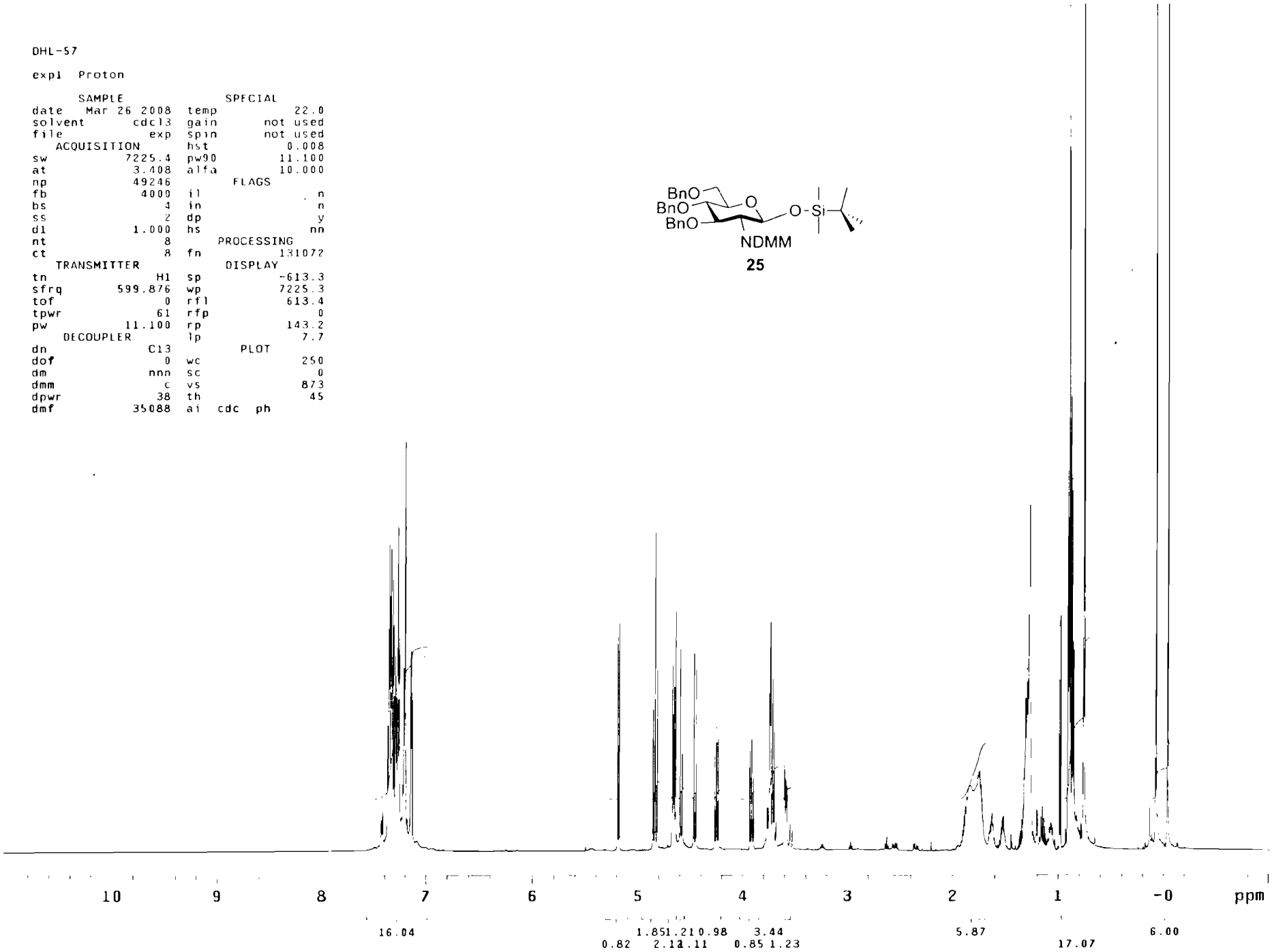
SAMPLE		SPECIAL	
date	Feb 17 2008	temp	22.0
solvent	cd3od	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	36764.7	pw90	7.500
at	1.783	alfa	10.000
np	131072	FLAGS	
fb	17000	il	n
bs	8	in	n
d1	1.220	dp	y
nt	2400	hs	nn
ct	224	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	262144
sfrq	150.855	DISPLAY	
tof	1542.6	sp	2279.9
tpwr	58	wp	23947.0
pw	7.500	rfl	9725.0
DECOUPLER		rfp	7413.7
dn	H1	rp	19.9
dof	0	lp	56.1
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	13908	vs	390
		th	7
		ai	cdc ph



23



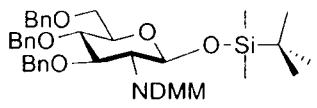

DHL-48_3
File: xp
Pulse Sequence: DEPT
AcO
AcO
AcO
NHTroc
O
OH
NHAc
23
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
120
110
100
90
80
70
60
50
40
30
20
10
ppm


DHL-57
expl Proton
SAMPLE
date Mar 26 2008
solvent cdcl3
file exp
ACQUISITION
sw 7225.4
at 3.408
np 49246
fb 4000
bs 4
ss 2
dl 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 599.876
tof 0
tpwr 61
pw 11.100
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 38
dmf 35088
SPECIAL
temp 22.0
gain not used
spin not used
hst 0.008
pw90 11.100
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 131072
DISPLAY
sp -613.3
wp 7225.3
rfl 613.4
rfp 0
rp 143.2
lp 7.7
PLOT
wc 250
sc 0
vs 873
th 45
ai cdc ph
BnO
BnO
BnO
O
O-Si
NDMM
25
10
9
8
7
6
5
4
3
2
1
-0
ppm
16.04
0.82
2.12
1.85
1.21
1.11
0.98
0.85
3.44
1.23
5.87
17.07
6.00

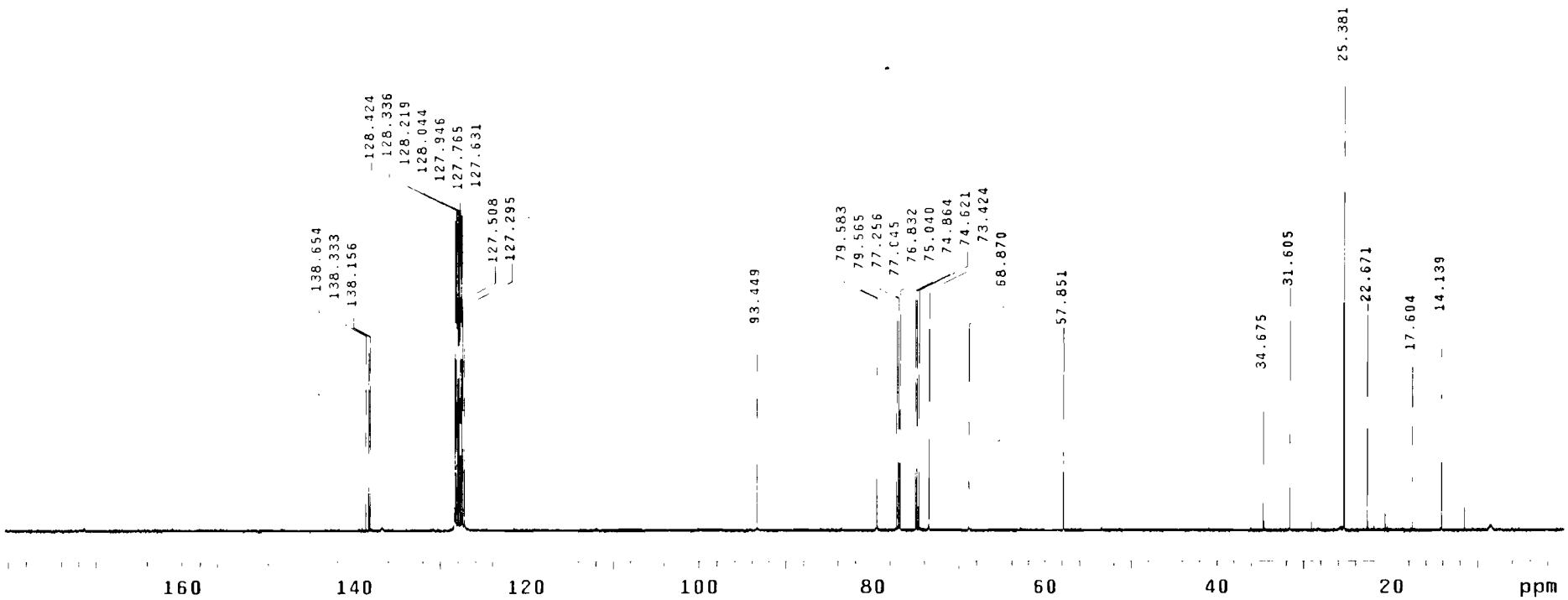
DHL-57

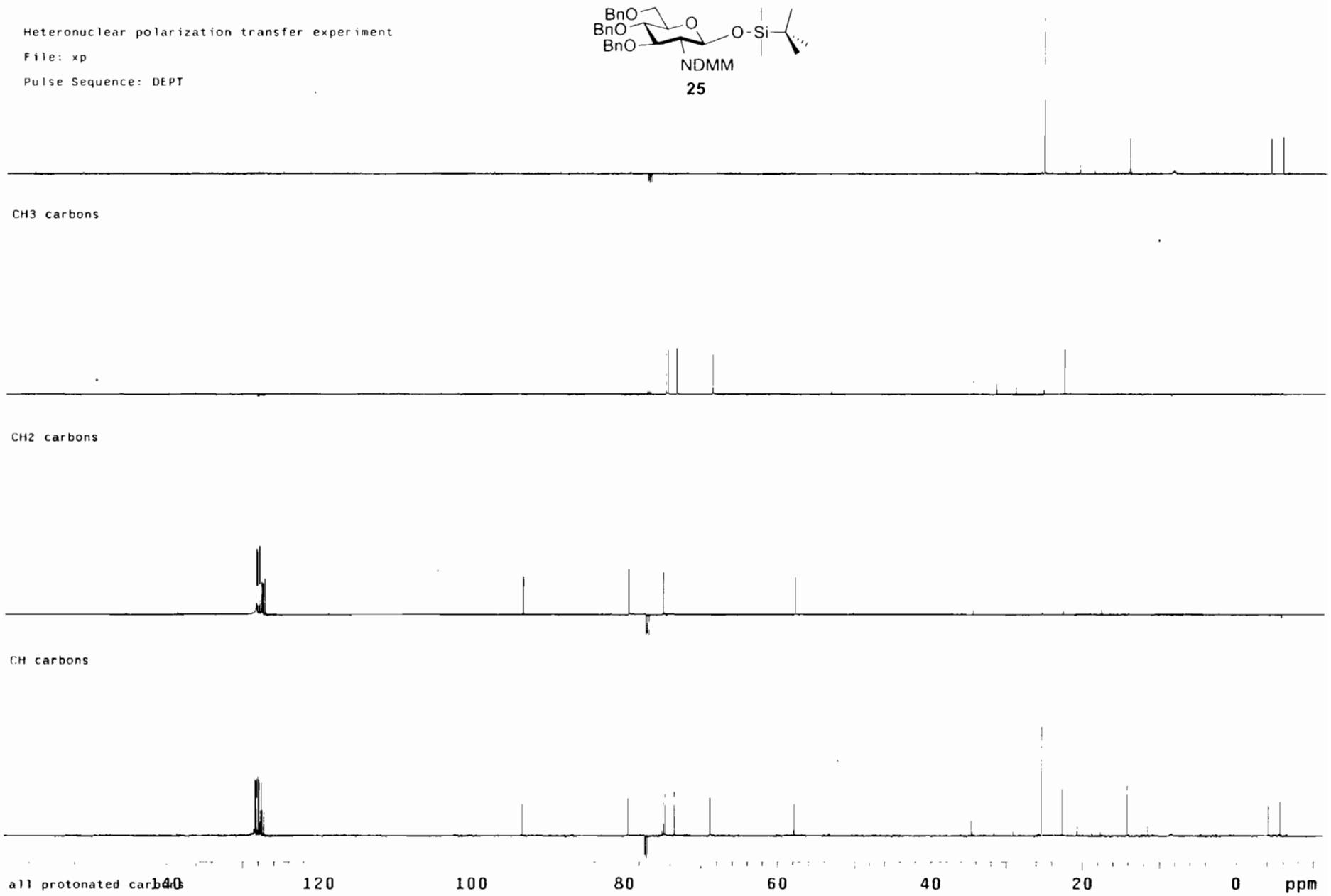
exp2 Carbon

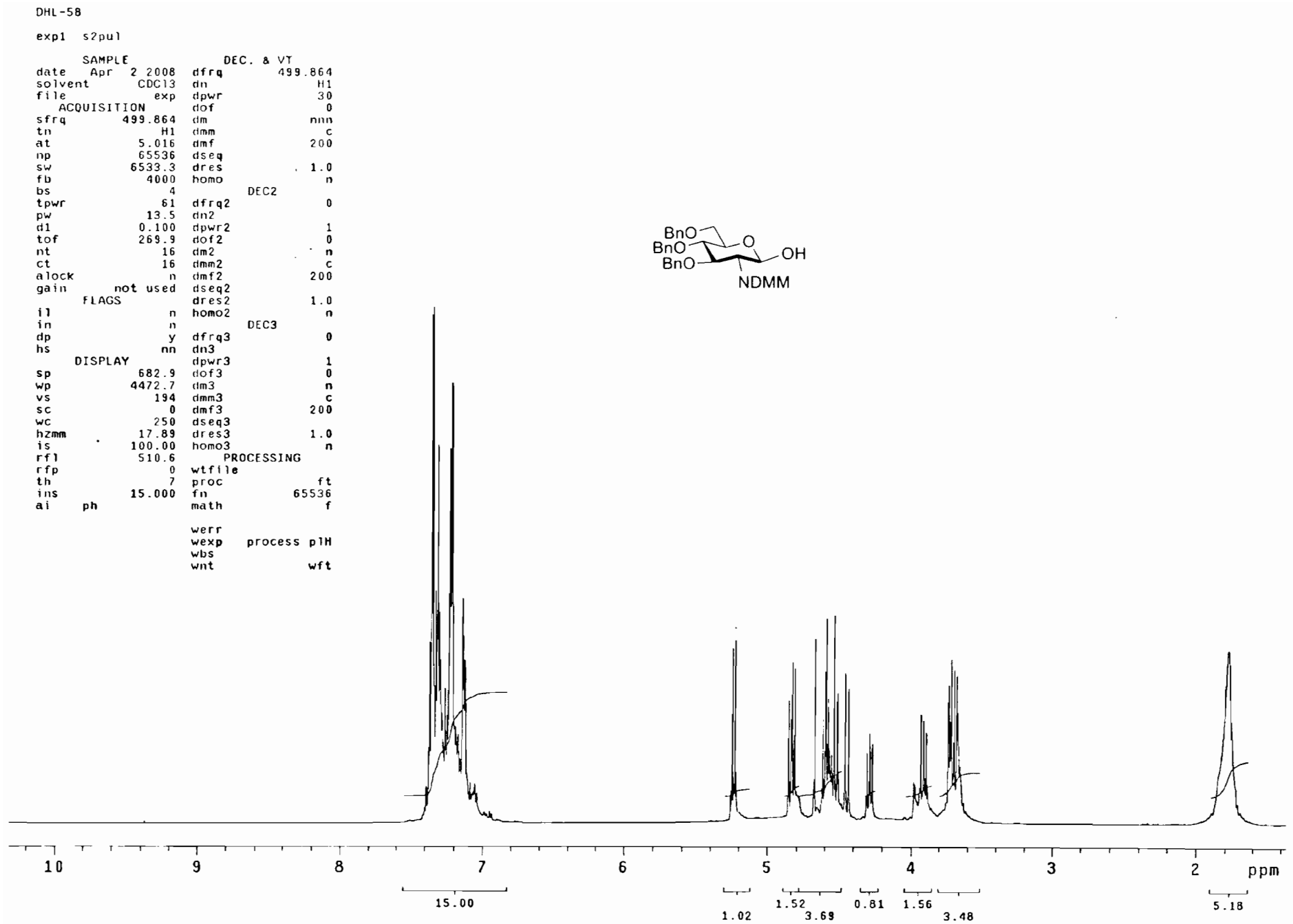
SAMPLE		SPECIAL	
date	Mar 26 2008	temp	22.0
solvent	cdcl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	7.500
at	1.783	alfa	10.000
np	108694	FLAGS	
fb	17000	il	n
bs	4	in	n
d1	1.220	dp	y
nt	256	hs	nn
ct	68	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	262144
sfrq	150.852	DISPLAY	
tof	-720.0	sp	1.6
tpwr	58	wp	27215.3
pw	7.500	rfl	1668.5
DECOUPLER		rfp	0
dn	H1	rp	-128.4
dof	0	lp	-4.3
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	13908	vs	151
		th	5
		ai cdc ph	

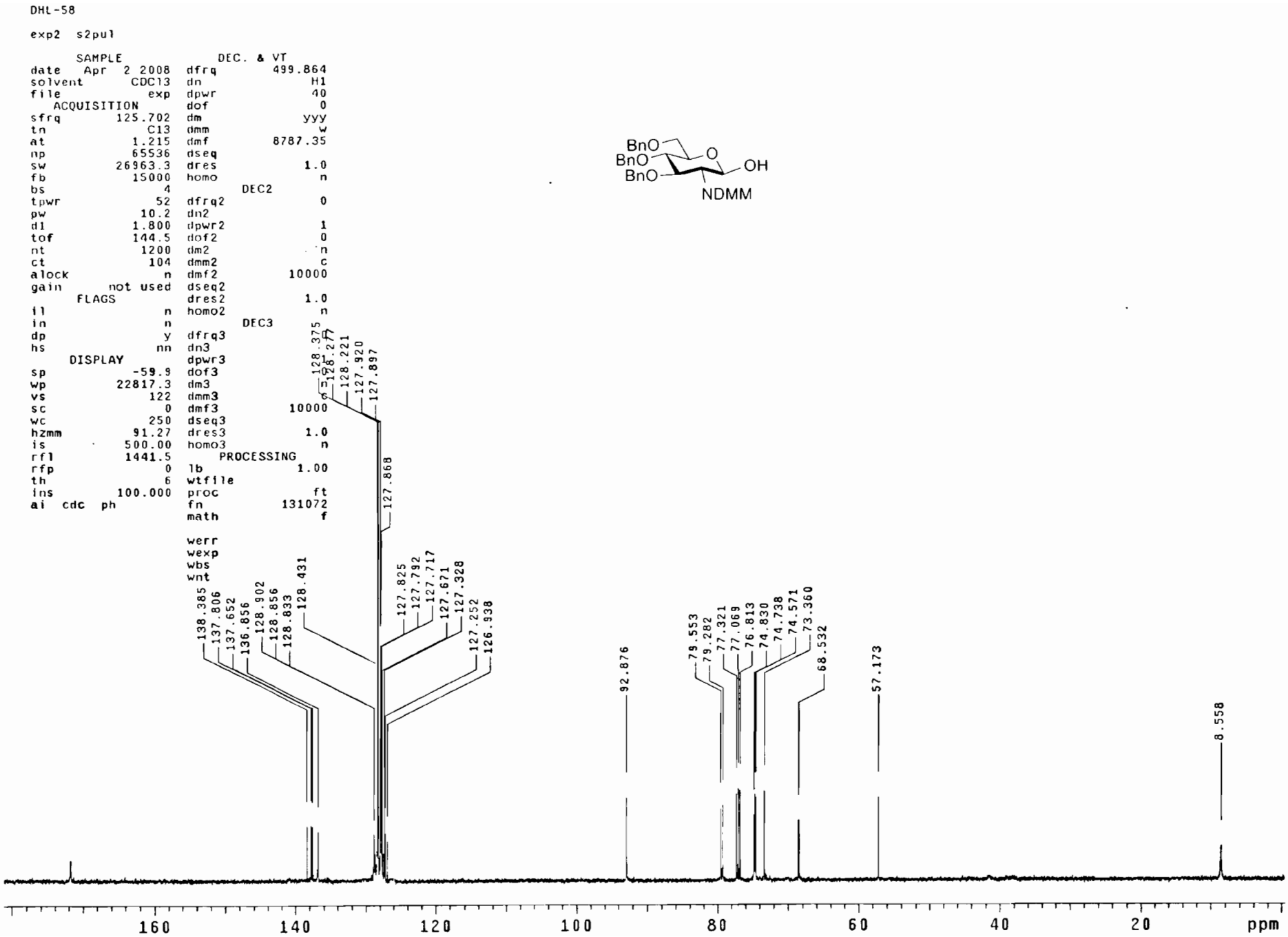


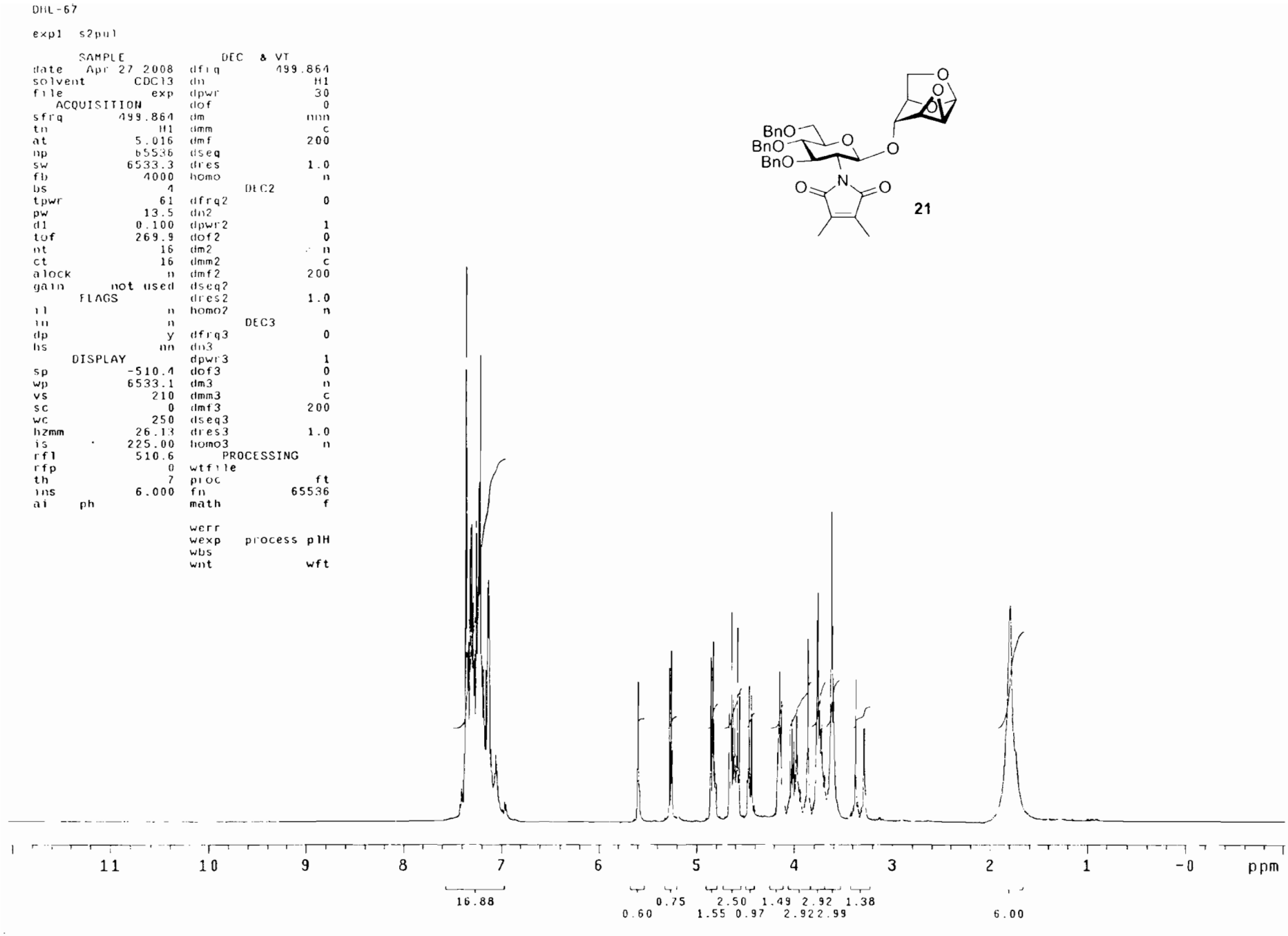
25

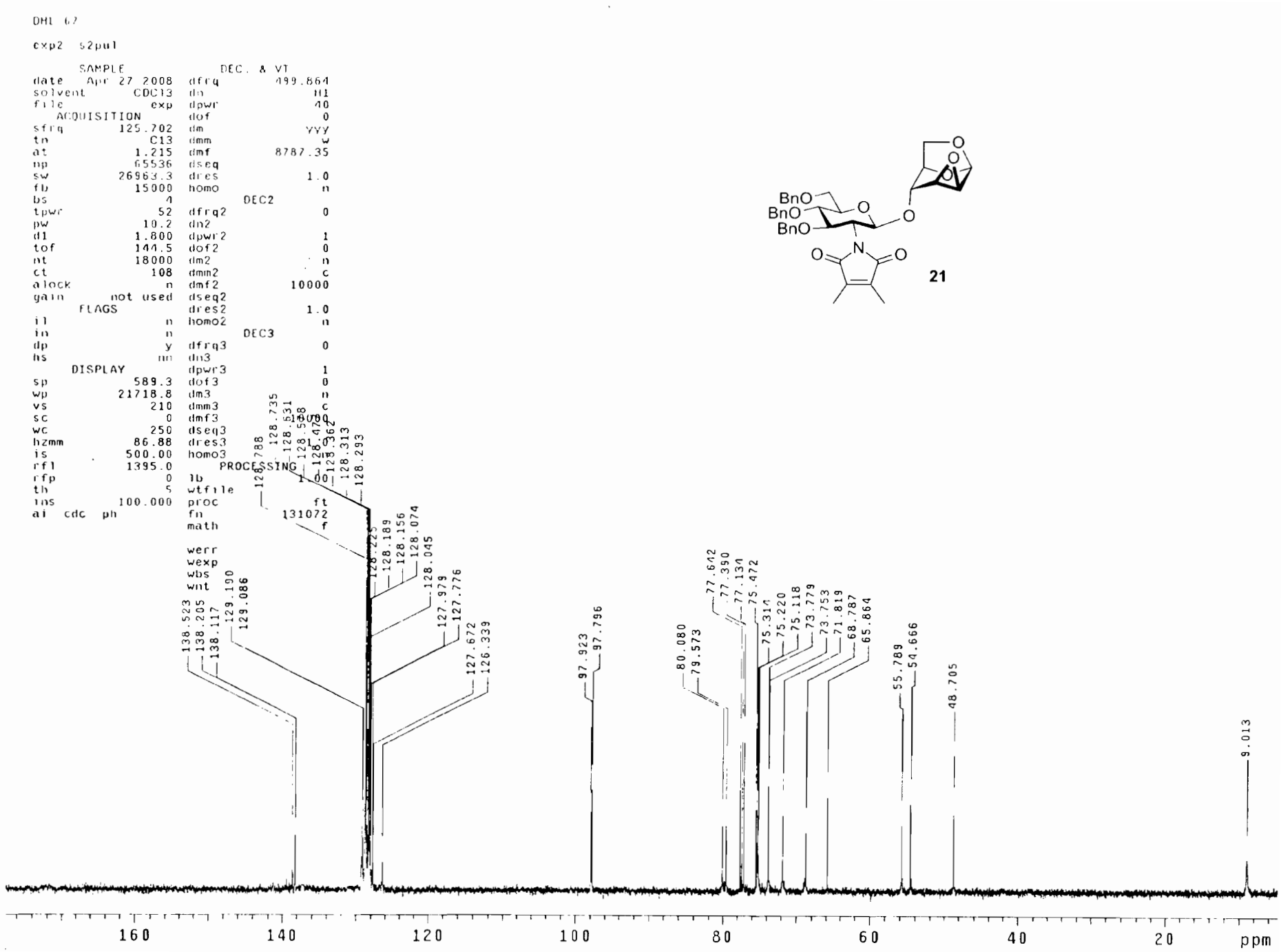



Heteronuclear polarization transfer experiment
File: xp
Pulse Sequence: DEPT
BnO
BnO
BnO
O
O-Si
NDMM
25
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
140
120
100
80
60
40
20
0
ppm


DHL-58
exp1 s2pul
SAMPLE
date Apr 2 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 682.9
wp 4472.7
vs 194
sc 0
wc 250
hzmm 17.89
is 100.00
rfl 510.6
rfp 0
th 7
ins 15.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
BnO
BnO
BnO
O
OH
NDMM
10
9
8
7
6
5
4
3
2
ppm
15.00
1.02
1.52
3.69
0.81
1.56
3.48
5.18

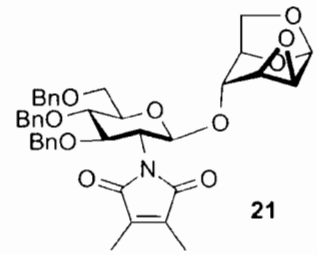

DHL-58
exp2 s2pul
SAMPLE
date Apr 2 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 104
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -59.9
wp 22817.3
vs 122
sc 0
wc 250
hzmm 91.27
is 500.00
rfl 1441.5
rfp 0
th 6
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
BnO
BnO
BnO
O
OH
NDMM
138.385
137.806
137.652
136.856
128.902
128.856
128.833
128.431
128.375
128.271
128.221
127.920
127.897
127.868
127.825
127.792
127.717
127.671
127.328
127.252
126.938
92.876
79.553
79.282
77.321
77.069
76.813
74.830
74.738
74.571
73.360
68.532
57.173
8.558
160
140
120
100
80
60
40
20
ppm


DHL-67
expl s2pul
SAMPLE
date Apr 27 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -510.4
wp 6533.1
vs 210
sc 0
wc 250
hzmm 26.13
is 225.00
rfl 510.6
rfp 0
th 7
ins 6.000
ai ph
DEC & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
BnO
BnO
BnO
O
O
O
N
O
O
21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
16.88
0.60
0.75
1.55
2.50
0.97
1.49
2.92
2.99
1.38
6.00


DHL 67
exp2 s2pul
SAMPLE
date Apr 27 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 18000
ct 108
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 589.3
wp 21718.8
vs 210
sc 0
wc 250
hzmm 86.88
is 500.00
rfl 1395.0
rfp 0
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm c
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3
homo3
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
BnO
BnO
BnO
O
O
O
O
O
N
O
O
21
138.523
138.205
138.117
129.190
129.086
128.788
128.735
128.631
128.588
128.470
128.362
128.313
128.293
128.225
128.189
128.156
128.074
128.045
127.979
127.776
127.672
126.339
97.923
97.796
80.080
79.573
77.642
77.390
77.134
75.472
75.314
75.220
75.118
73.779
73.753
71.819
68.787
65.864
55.789
54.666
48.705
9.013
160
140
120
100
80
60
40
20
ppm

DHL-67

Pulse Sequence: dept



21

CH3 carbons

CH2 carbons

CH carbons

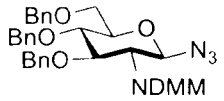
all protonated carbons

95 90 85 80 75 70 65 60 55 50 ppm

DHL-68_1

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 18 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	32	dm2	n
ct	32	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	502.5	dof3	0
wp	4996.3	dm3	n
vs	203	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	100.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	6.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



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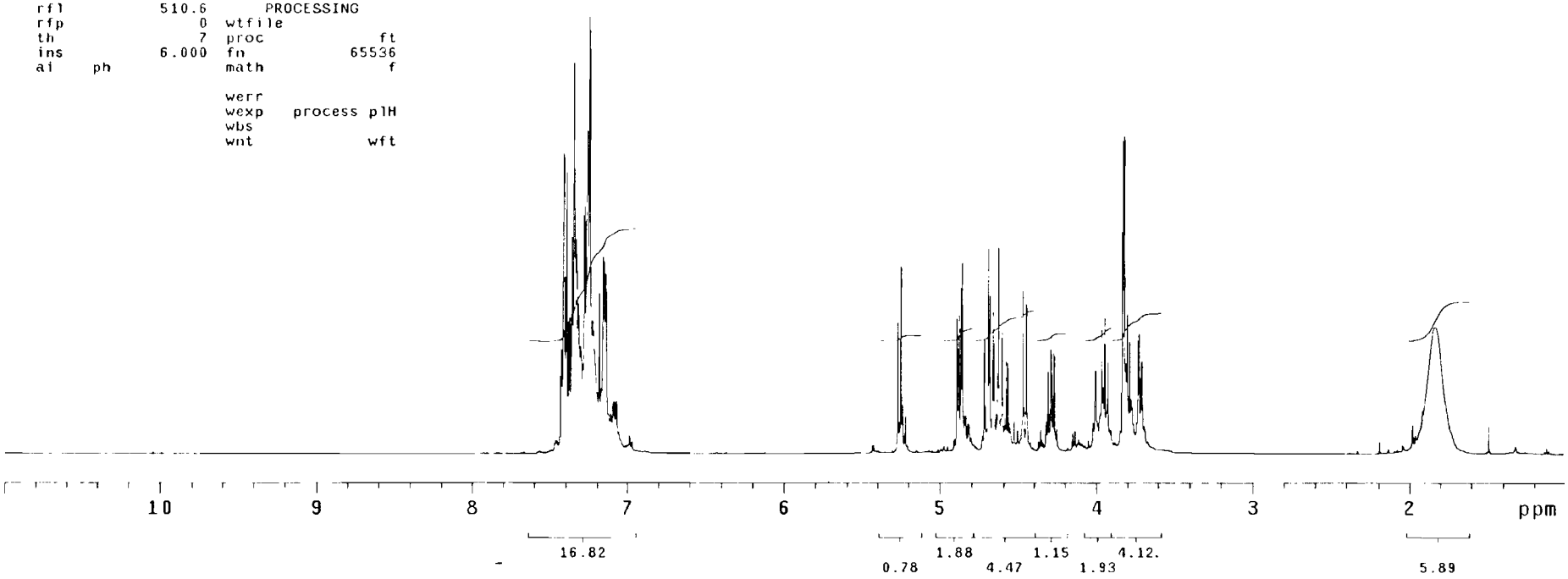
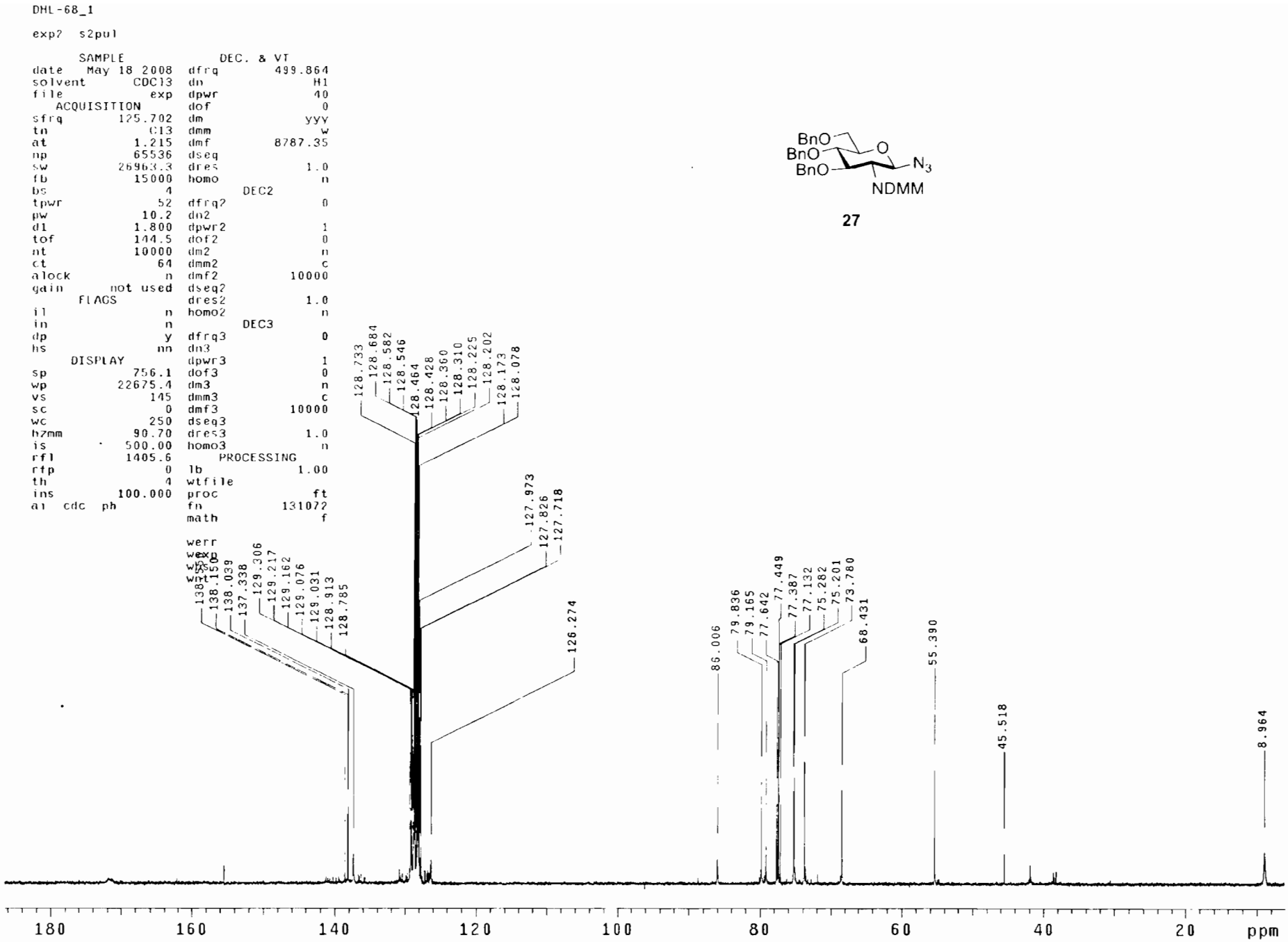
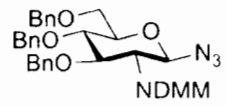



DHL-68_1
exp2 s2pul
SAMPLE
date May 18 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 10000
ct 64
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 756.1
wp 22675.4
vs 145
sc 0
wc 250
hzmm 90.70
is 500.00
rfl 1405.6
rfp 0
th 4
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
BnO
BnO
BnO
O
N3
NDMM
27
138.150
138.039
137.338
129.306
129.217
129.162
129.076
129.031
128.913
128.785
128.733
128.684
128.582
128.546
128.464
128.428
128.360
128.310
128.225
128.202
128.173
128.078
127.973
127.826
127.718
126.274
86.006
79.836
79.165
77.642
77.449
77.387
77.132
75.282
75.201
73.780
68.431
55.390
45.518
8.964
180
160
140
120
100
80
60
40
20
ppm

DHL-68_1

Pulse Sequence: dept



27

CH3 carbons

CH2 carbons

CH carbons

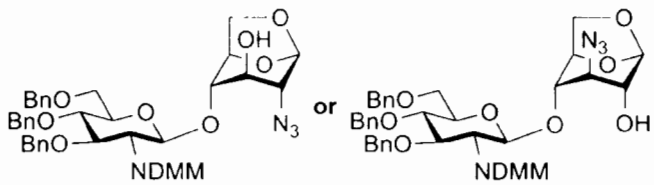
all protonated carbons

95 90 85 80 75 70 65 60 55 ppm

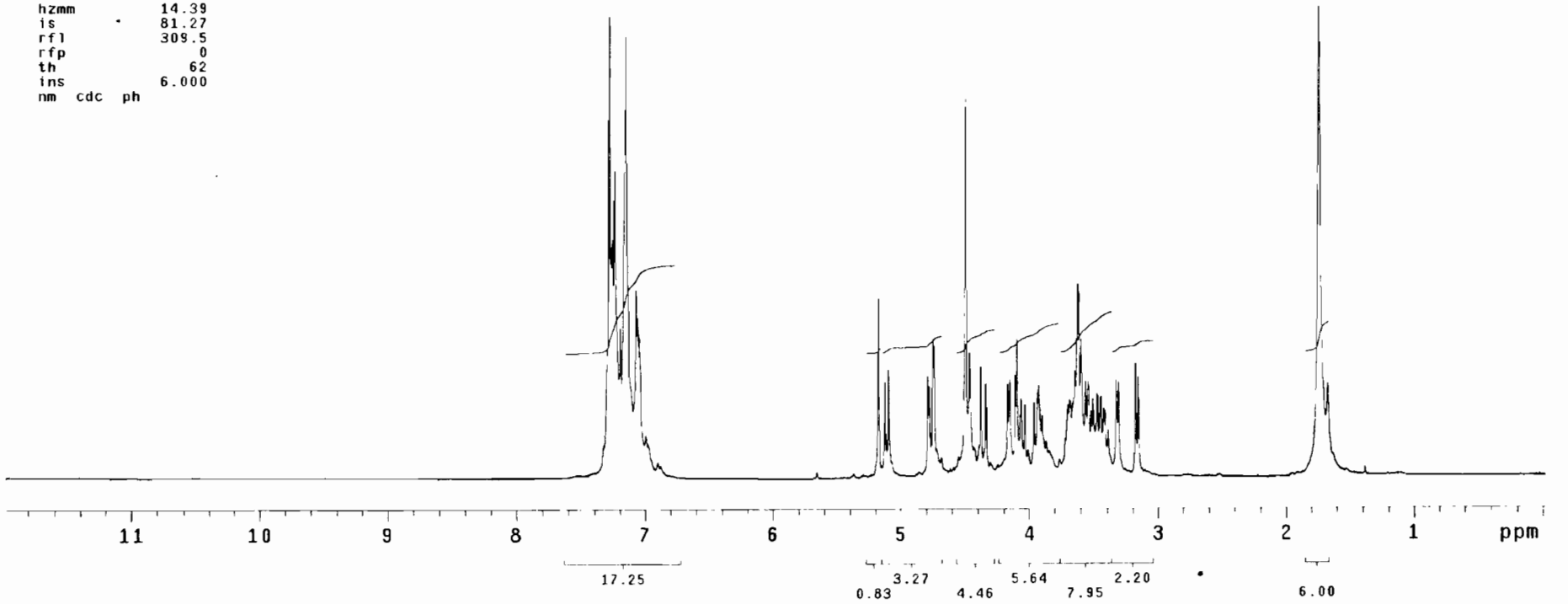
DHL-68_3

exp3 s2pul

SAMPLE		DEC. & VT	
date	May 14 2008	dfrq	299.948
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.949	dm	nnn
tn	H1	dmm	c
at	3.620	dmf	200
np	32768	dseq	
sw	4525.4	dres	1.0
fb	2600	homo	n
bs	4	PROCESSING	
tpwr	59	wtfile	
pw	14.0	proc	ft
d1	0.380	fn	65536
tof	481.6	math	f
nt	16		
ct	16	werr	
alock	n	wexp	process plH
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-6.4		
wp	3597.6		
vs	76		
sc	0		
wc	250		
hzmm	14.39		
is	81.27		
rfl	309.5		
rfp	0		
th	62		
ins	6.000		
nm cdc ph			



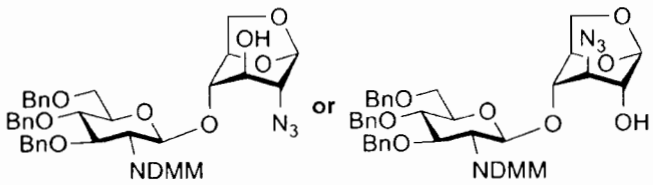
28a



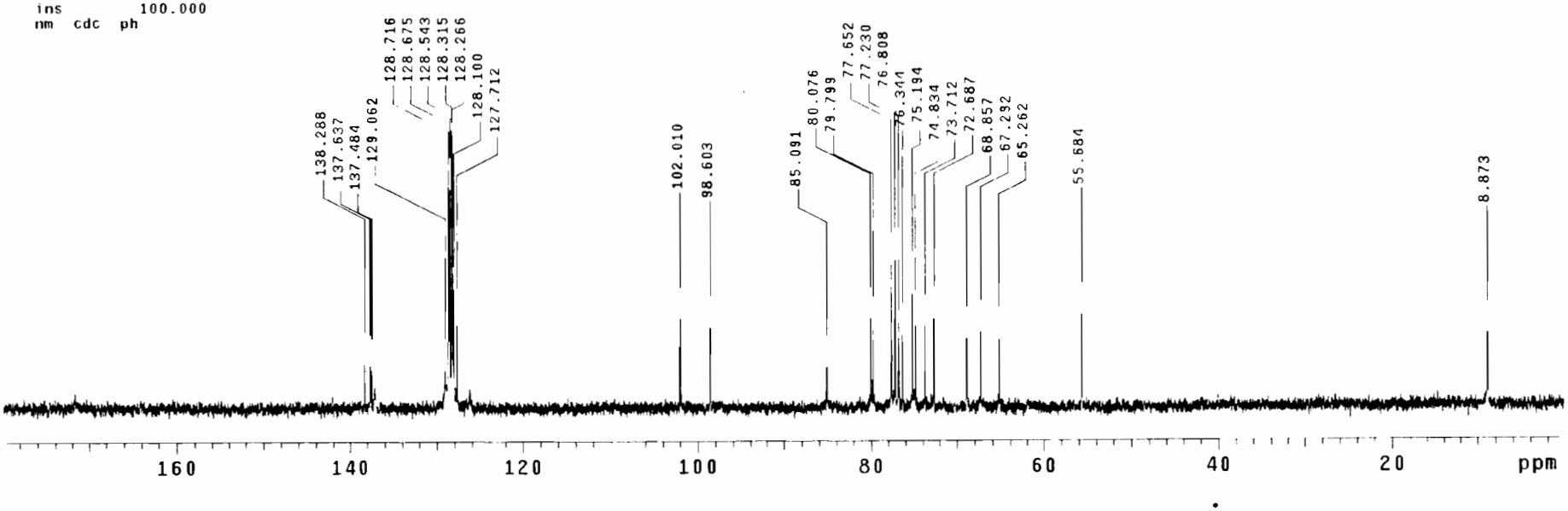
DHL-68_3

exp4 s2pul

SAMPLE		DEC. & VT	
date	May 14 2008	dfrq	299.949
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	272.7
sfrq	75.429	dm	yyy
tn	C13	dmm	w
at	0.957	dmf	8033
np	32768	dseq	
sw	17116.0	dres	1.0
fb	9400	homo	n
bs	4	PROCESSING	
tpwr	59	lb	1.00
pw	8.3	wtfile	
d1	2.000	proc	ft
tof	0	fn	65536
nt	640	math	f
ct	196		
alock	n	werr	
gain	not used	wexp	process plC
FLAGS		wbs	wft
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	5.8		
wp	13561.9		
vs	44		
sc	0		
wc	250		
hzmm	54.25		
is	500.00		
rfl	6830.8		
rfp	5824.8		
th	5		
ins	100.000		
nm cdc ph			



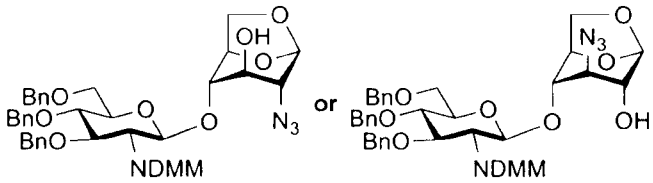
28a



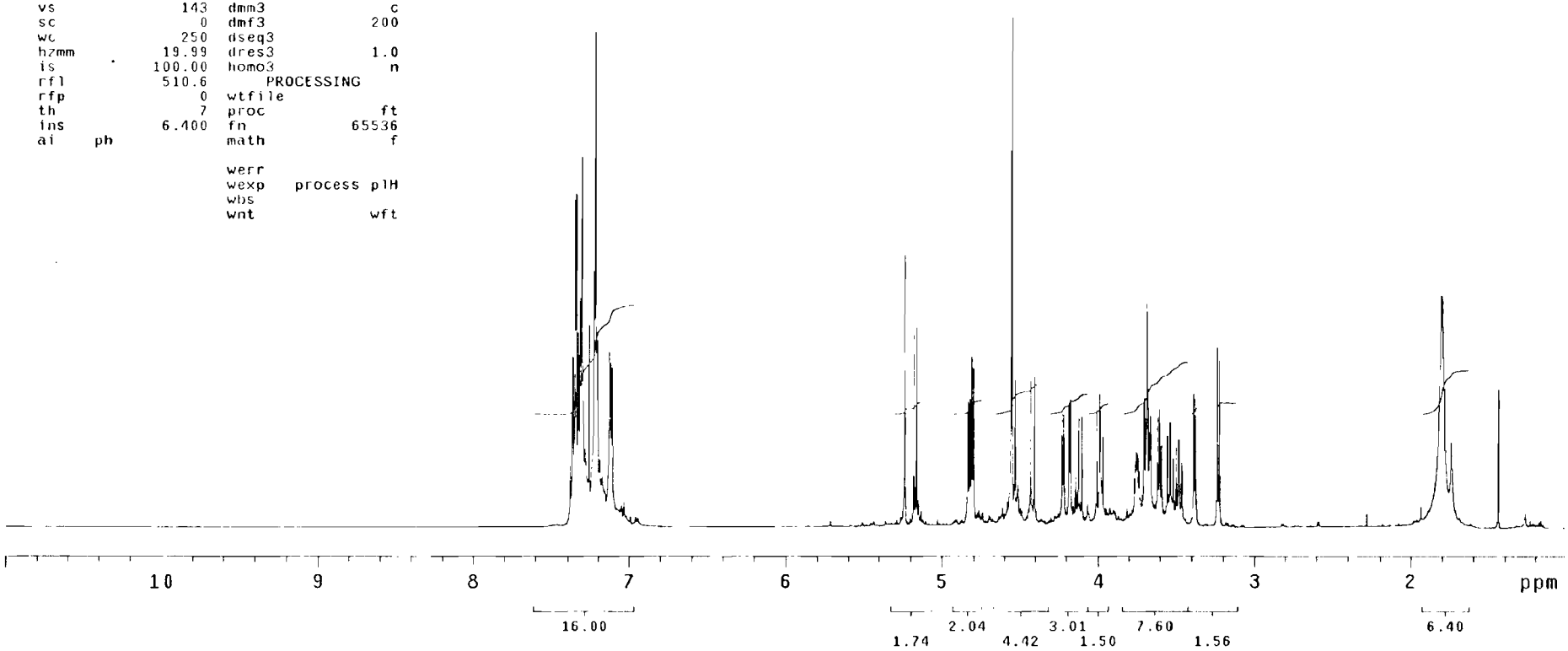
DHL-68_4

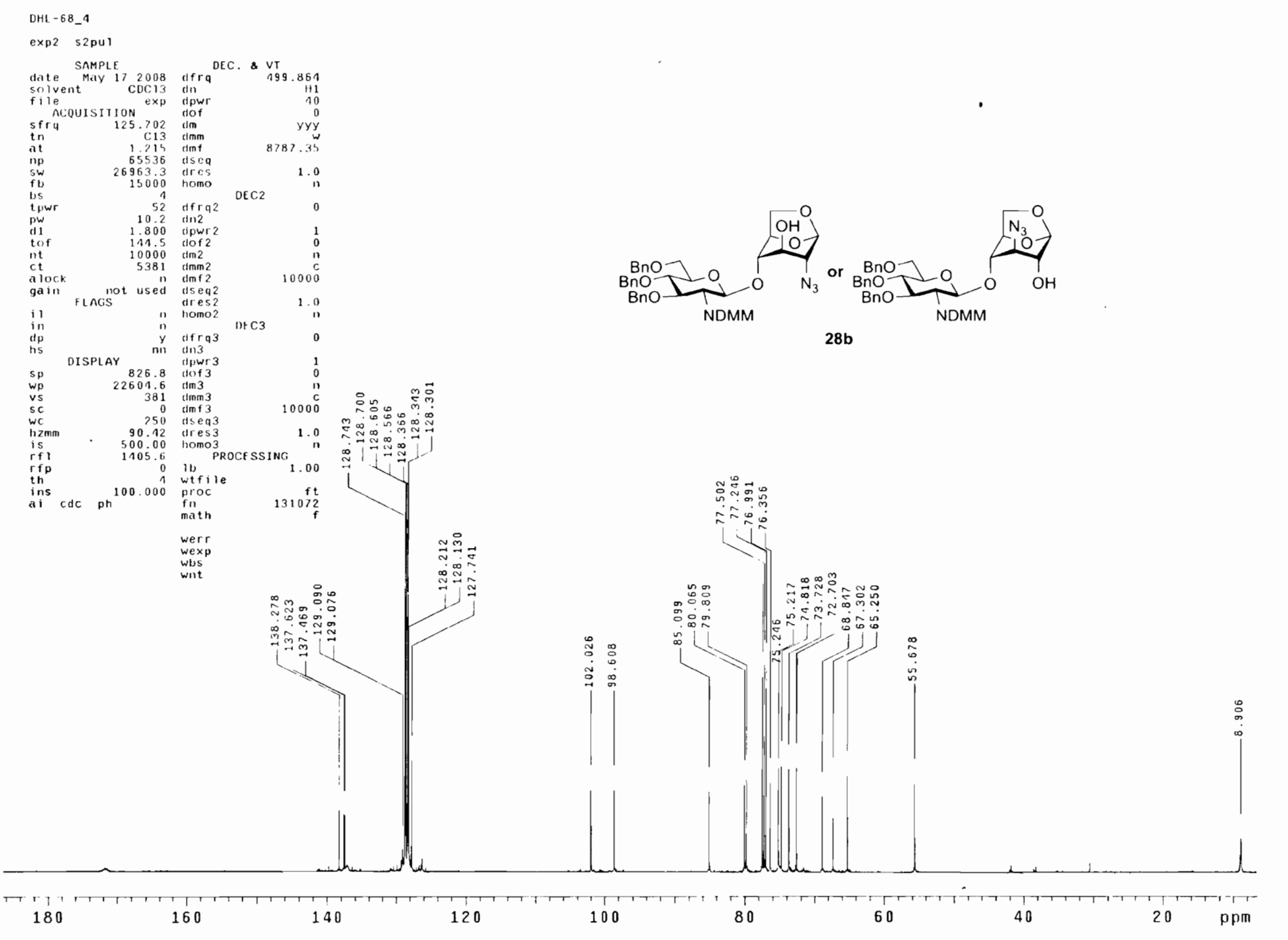
exp1 s2pul

SAMPLE		DEC. & VT	
date	May 17 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	32	dm2	n
ct	32	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	502.5	dof3	0
wp	4996.3	dm3	n
vs	143	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	100.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	6.400	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



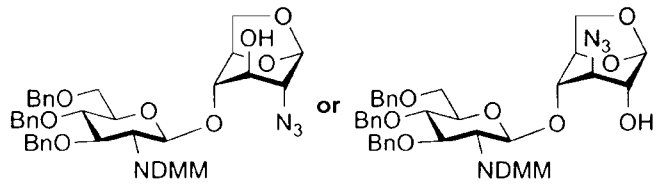
28b




DHL-68_4
exp2 s2pul
SAMPLE
date May 17 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 10000
ct 5381
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 826.8
wp 22604.6
vs 381
sc 0
wc 250
hzmm 90.42
is 500.00
rfl 1405.6
rfp 0
th 4
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
BnO
BnO
BnO
O
NDMM
OH
N3
or
28b
128.743
128.700
128.605
128.566
128.366
128.343
128.301
128.212
128.130
127.741
138.278
137.623
137.469
129.090
129.076
102.026
98.608
85.099
80.065
79.809
77.502
77.246
76.991
76.356
75.246
75.217
74.818
73.728
72.703
68.817
67.302
65.250
55.678
8.906
180
160
140
120
100
80
60
40
20
ppm

DHL-68_4

Pulse Sequence: dept



28b

CH3 carbons

CH2 carbons

CH carbons

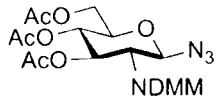
all protonated carbons

105 100 95 90 85 80 75 70 65 60 55 ppm

DHL-77

expl s2pul

SAMPLE		DEC. & VT	
date	Jun 5 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	791.0	dof3	0
wp	2940.1	dm3	n
vs	46	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	11.76	dres3	1.0
is	200.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

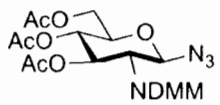


30

DHL-77

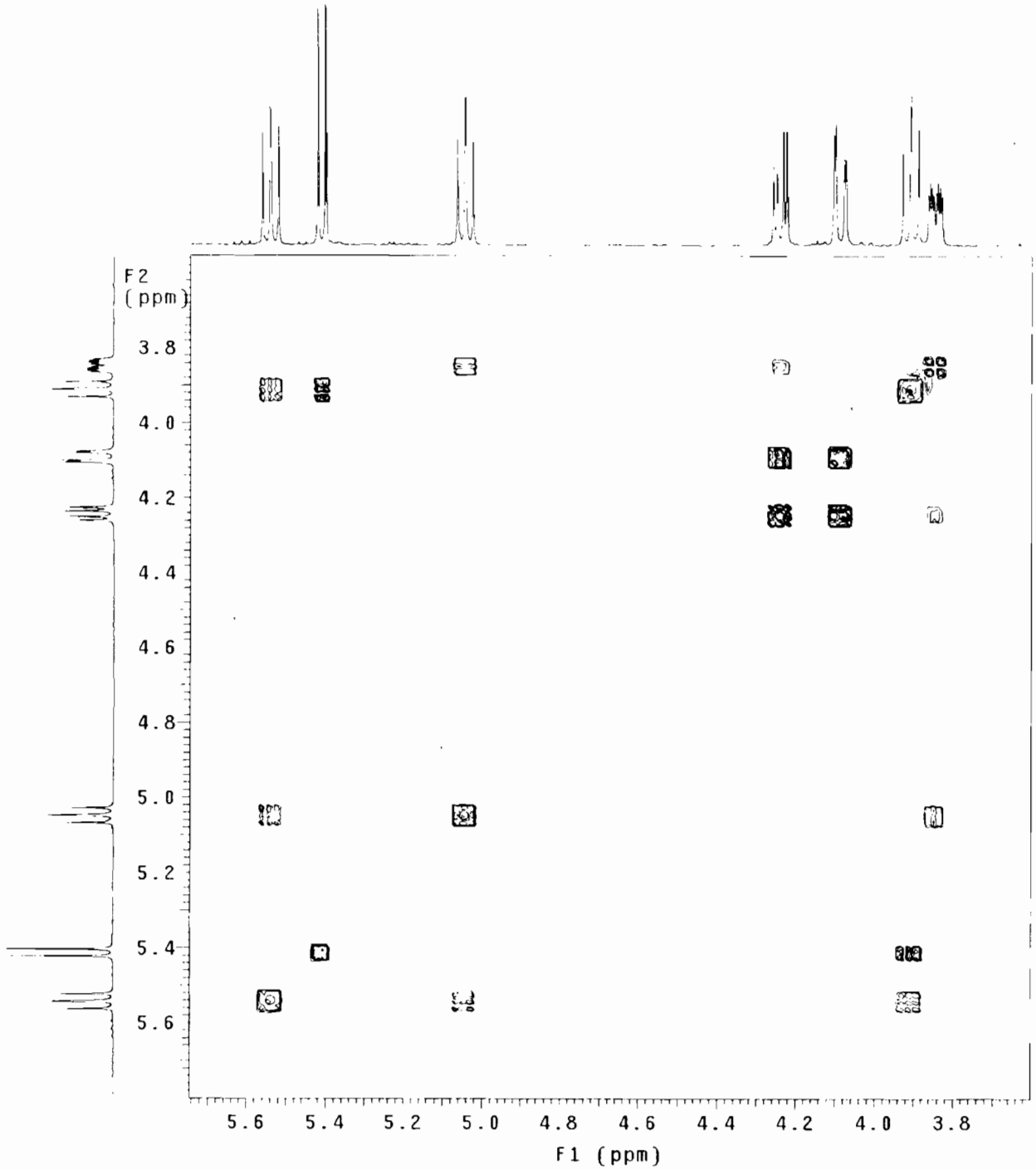
Pulse Sequence: relayh
 Solvent: CDCl3
 Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.219 sec
 Width 2334.9 Hz
 2D Width 2334.9 Hz
 4 repetitions
 256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
 Sine bell 0.110 sec
F1 DATA PROCESSING
 Sine bell 0.055 sec
FT size 1024 x 1024
Total time 27 min, 14 sec



NDMM

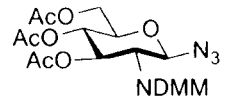
30



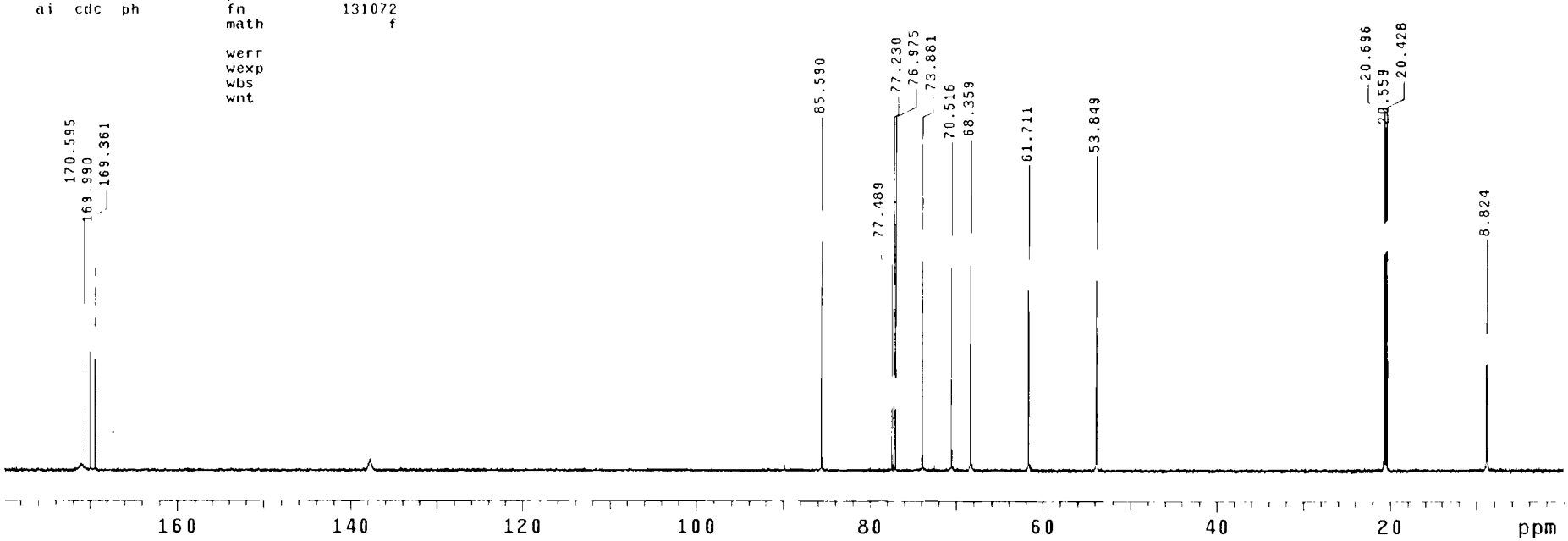
DHL-77

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 5 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	64	dm2	n
ct	60	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
fl	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-0.6	dof3	0
wp	22604.6	dm3	n
vs	64	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	90.42	dres3	1.0
is	500.00	homo3	n
rfl	11125.0	PROCESSING	
rfp	9707.1	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

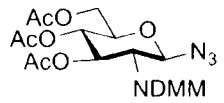


30



DHL-77

Pulse Sequence: dept



30

CH3 carbons

CH2 carbons

CH carbons

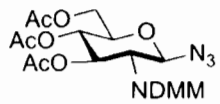
all protonated carbons

100 90 80 70 60 50 40 30 20 10 ppm

DHL-77

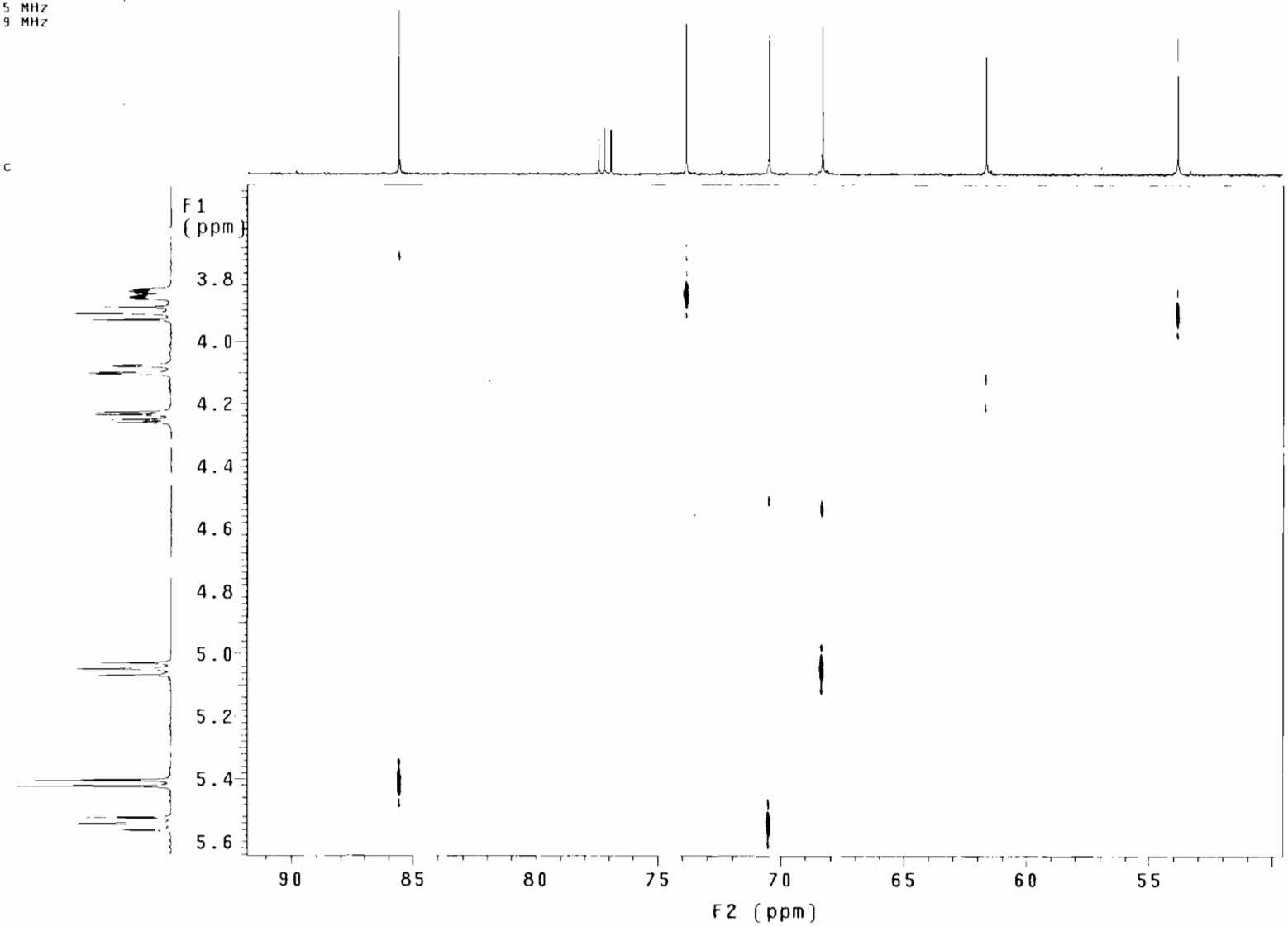
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

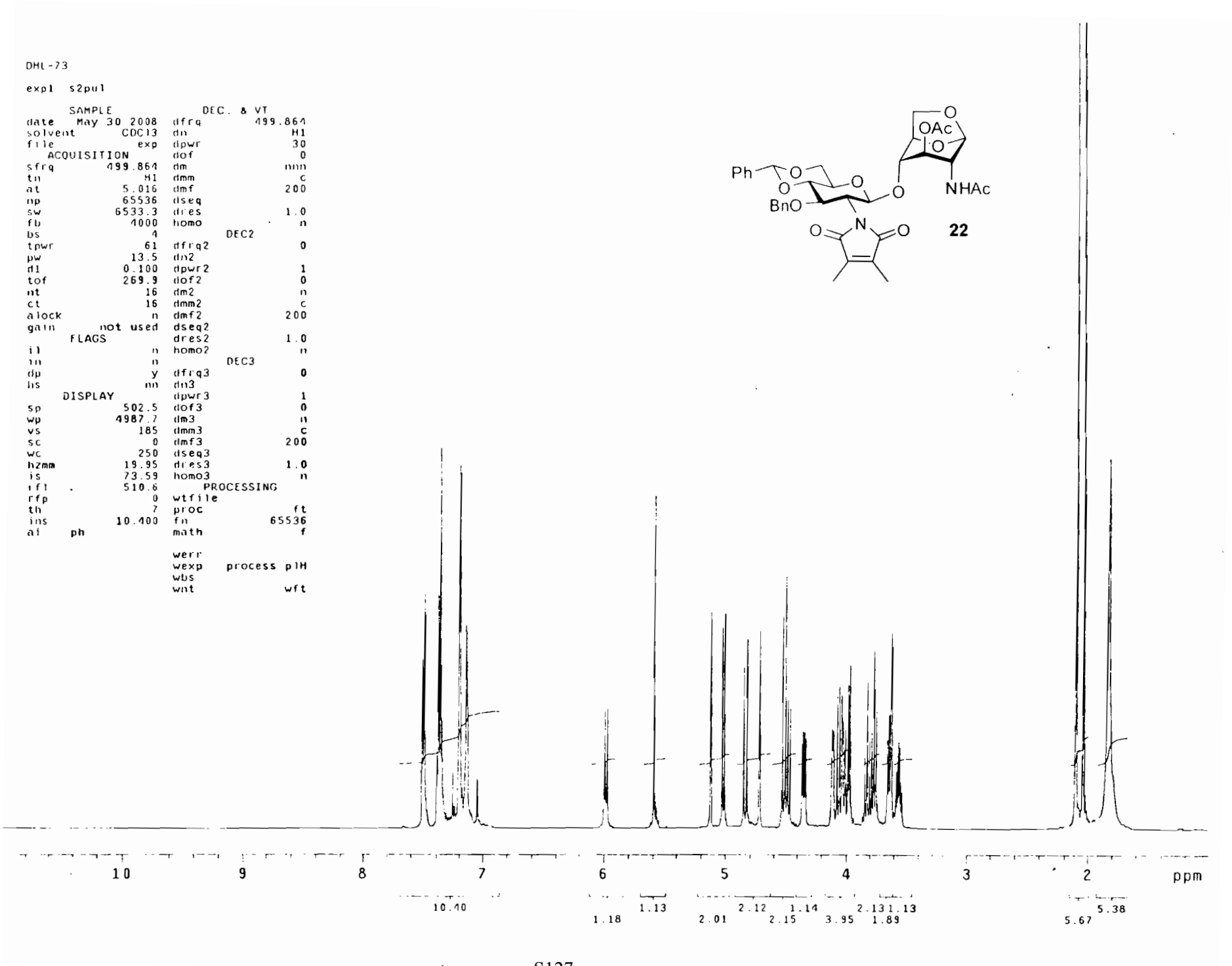
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2030.3 Hz
4 repetitions
128 increments
OBSERVE C13, 125.6901825 MHz
DECOUPLE H1, 499.8630359 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
F1 DATA PROCESSING
line broadening 0.3 Hz
FT size 4096 x 512
Total time 14 min, 20 sec



NDMM

30

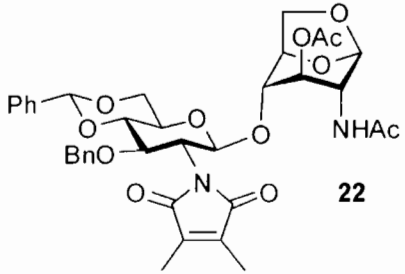



DHL-73
expl s2pul
SAMPLE
date May 30 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 502.5
wp 4987.7
vs 185
sc 0
wc 250
hzmm 19.95
is 73.59
rfl 510.6
rfp 0
th 7
ins 10.400
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
Ph
BnO
OAc
NHAc
22
10
9
8
7
6
5
4
3
2
ppm
10.40
1.18
1.13
2.01
2.12
2.15
1.14
3.95
2.13
1.89
1.13
5.67
5.38

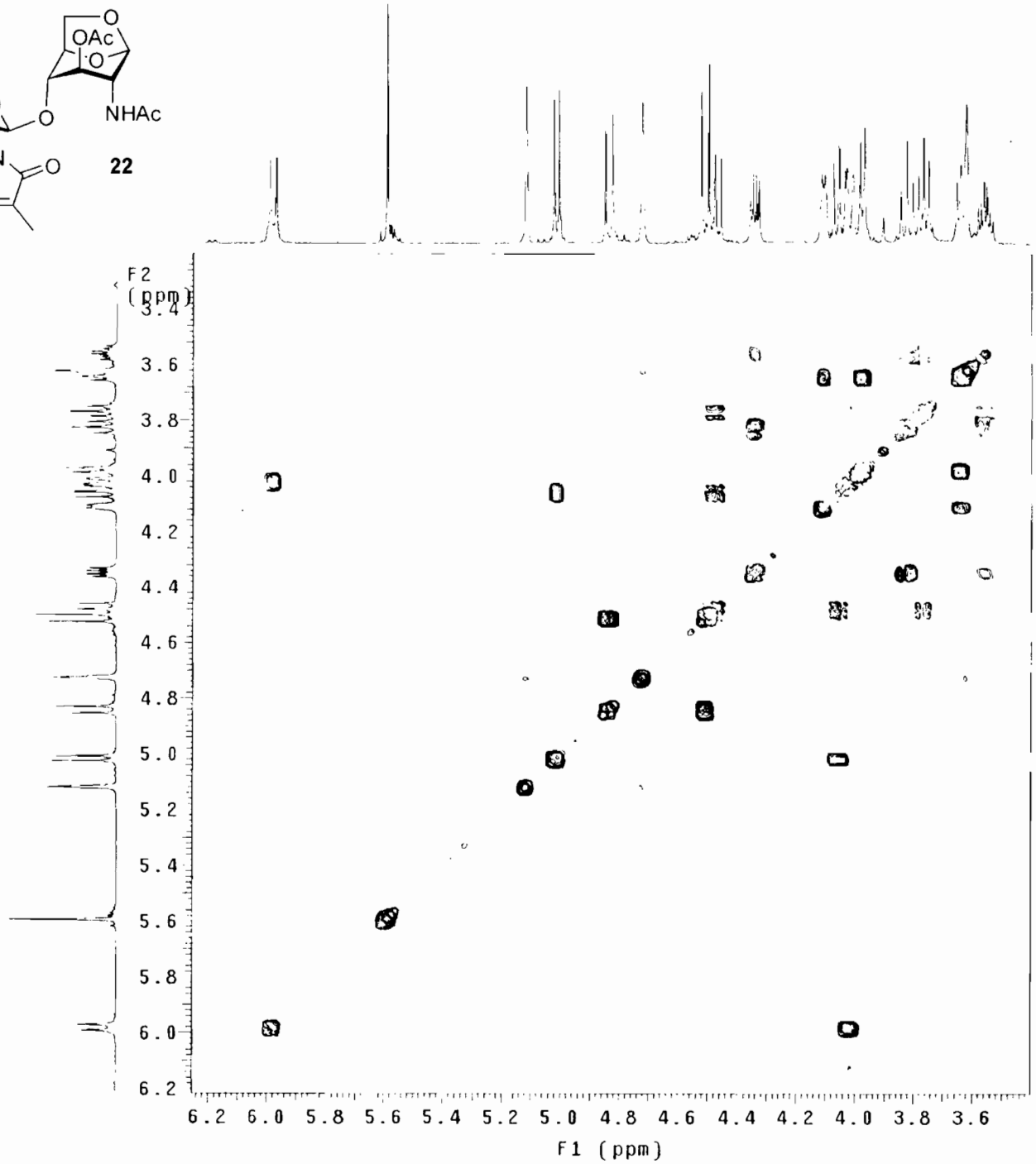
DHL-73

Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.161 sec
Width 3172.1 Hz
2D Width 3172.1 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.081 sec
F1 DATA PROCESSING
Sine bell 0.040 sec
FT size 1024 x 1024
Total time 51 min, 48 sec



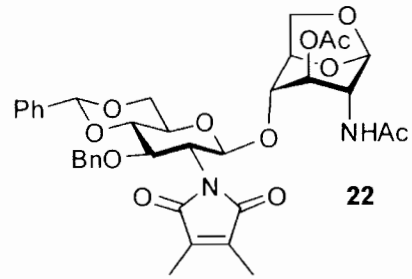
22



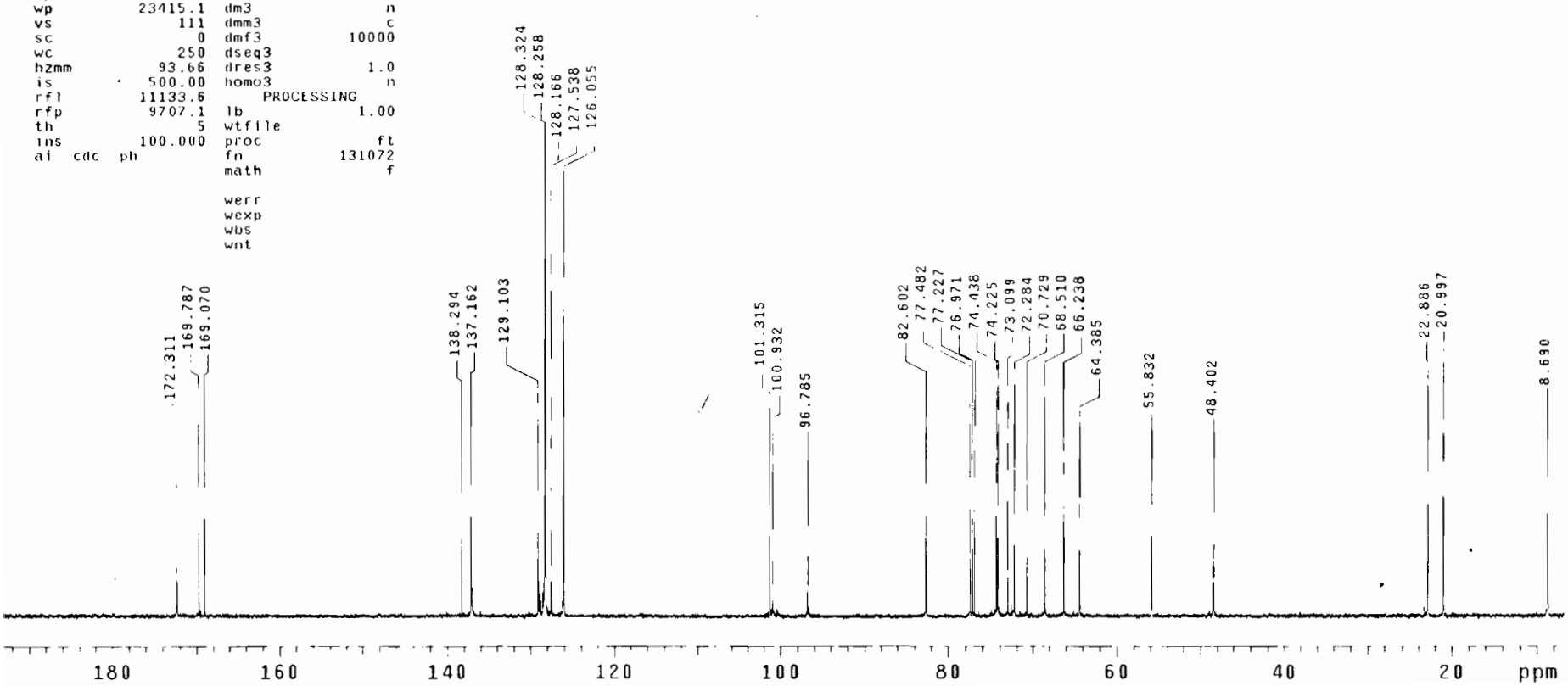
DHI-73

exp2 s2pul

SAMPLE		DEC. & VT	
date	May 30 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	640	dm2	n
ct	146	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	837.5	dof3	0
wp	23415.1	dm3	n
vs	111	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	93.66	dres3	1.0
is	500.00	homo3	n
rfl	11133.6	PROCESSING	
rfp	9707.1	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



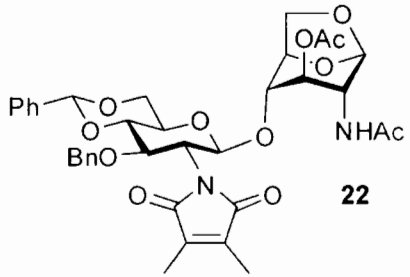
22



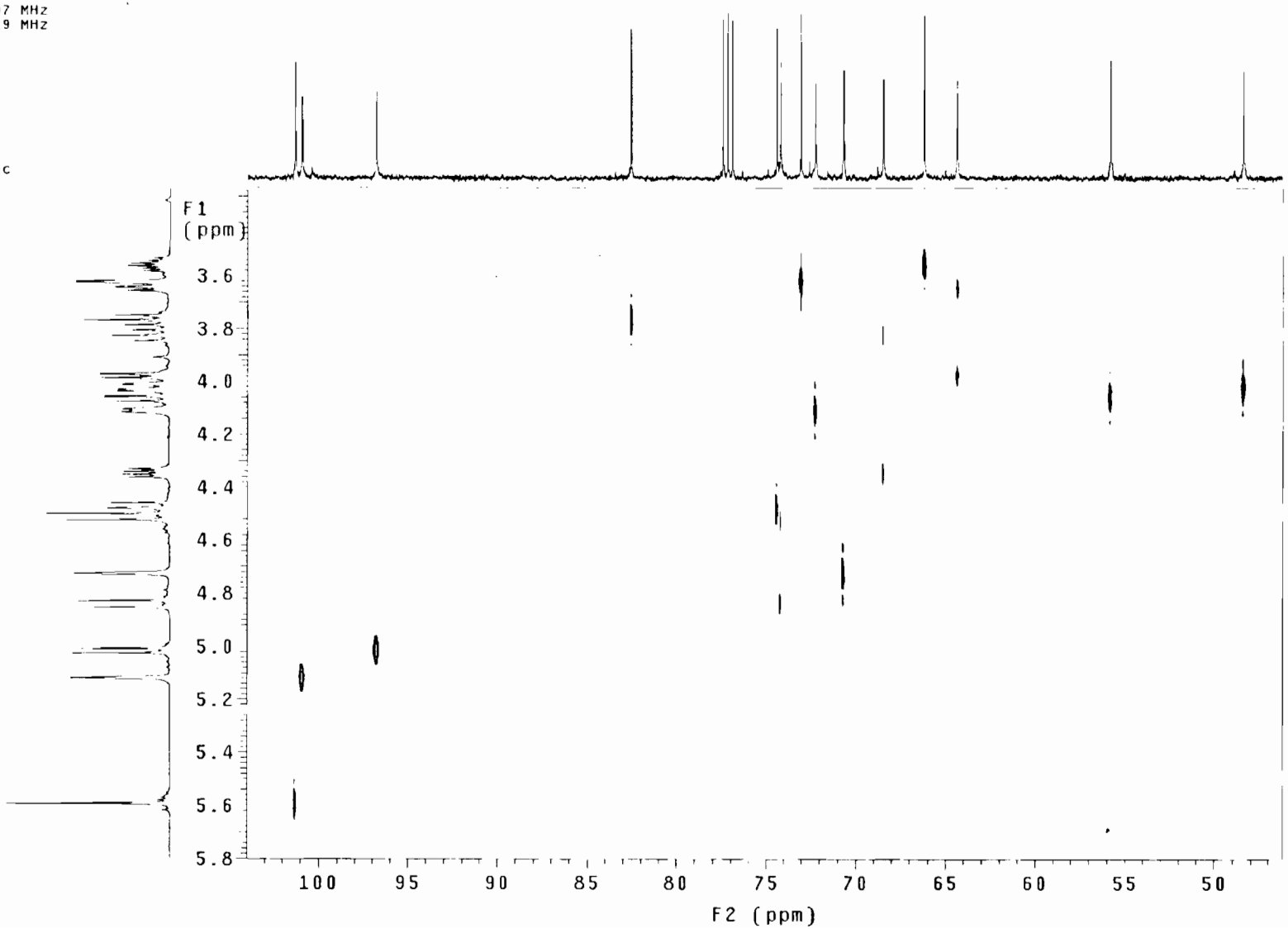
DHL-73

Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3408.0 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6901907 MHz
DECOUPLE H1, 499.8635019 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 39 sec



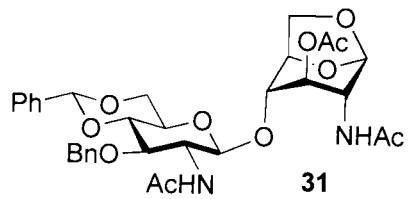
22



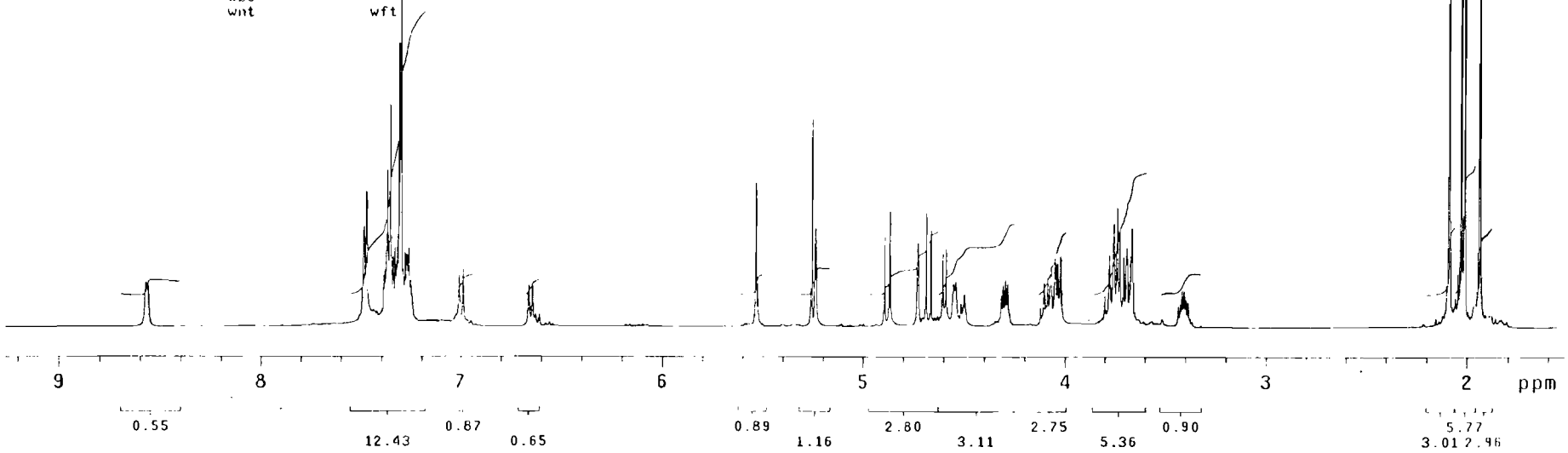
DHL-75

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 1 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	760.1	dof3	0
wp	3871.8	dm3	n
vs	120	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.49	dres3	1.0
is	400.00	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



31

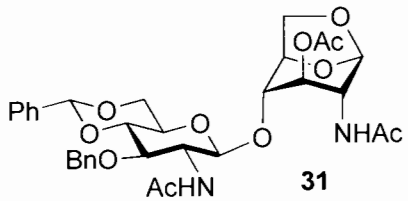


DHL-75

Pulse Sequence: relayh

Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.147 sec
Width 3494.5 Hz
2D Width 3494.5 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.073 sec
F1 DATA PROCESSING
Sine bell 0.037 sec
FT size 1024 x 1024
Total time 51 min, 10 sec



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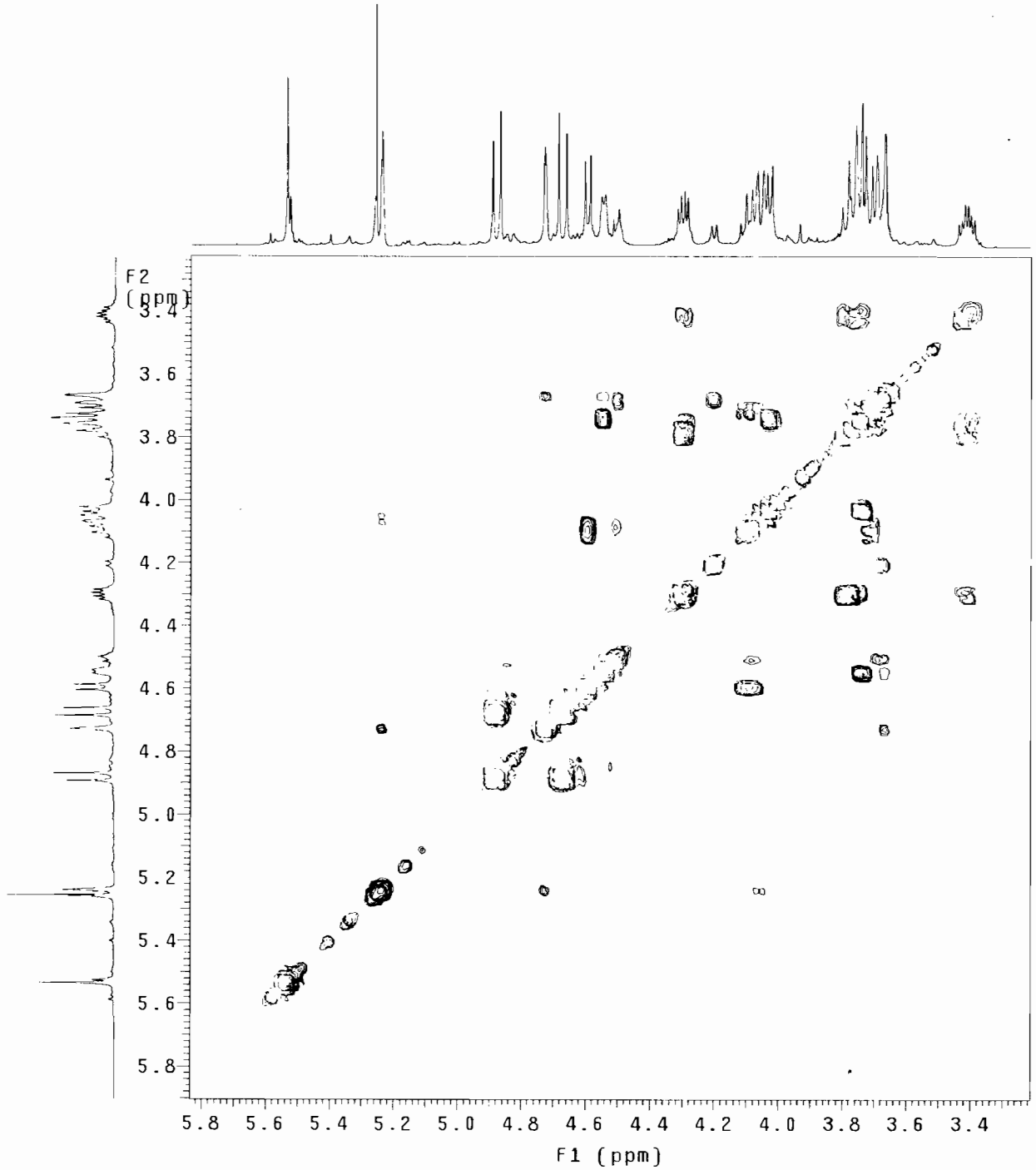
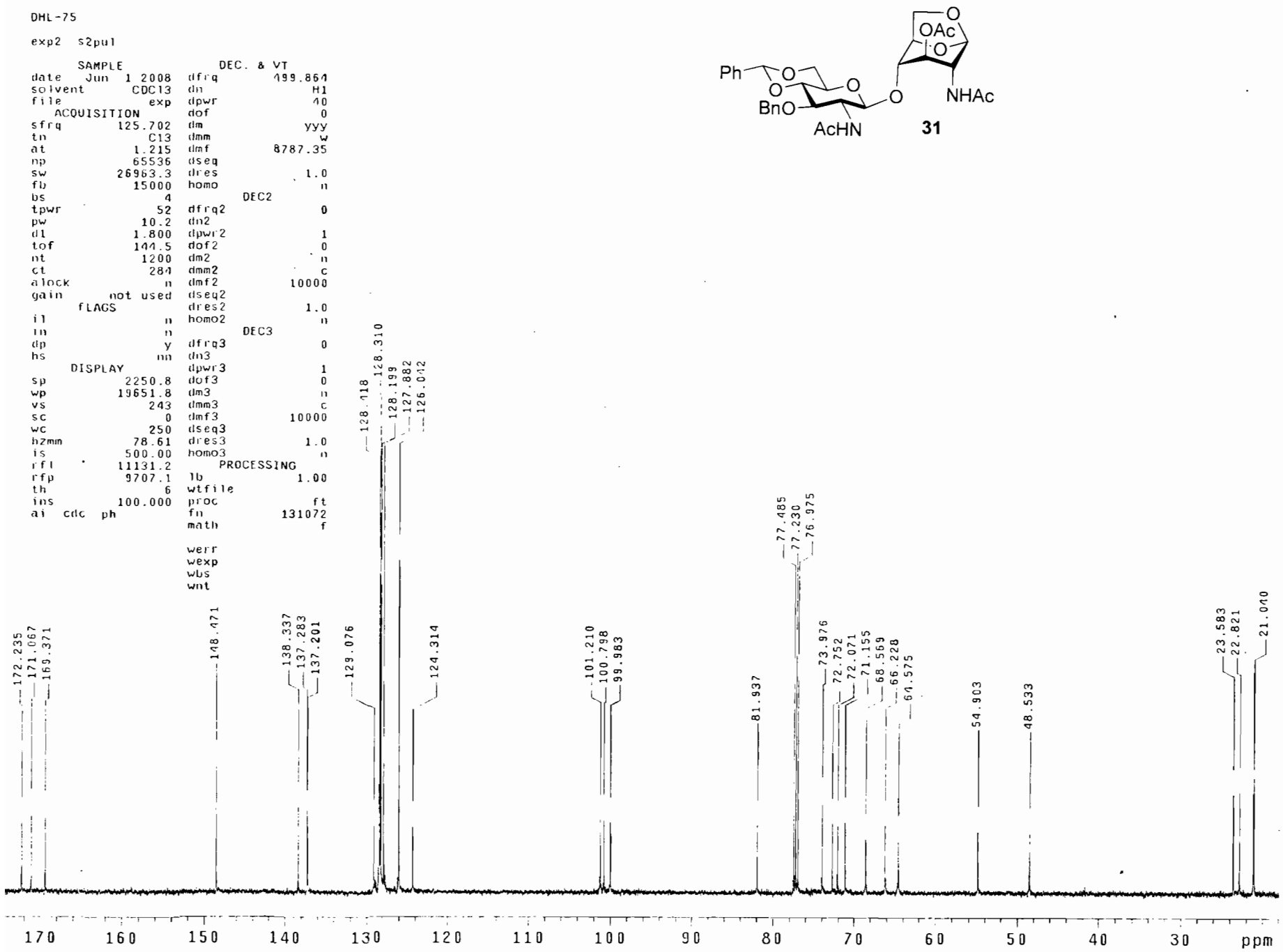
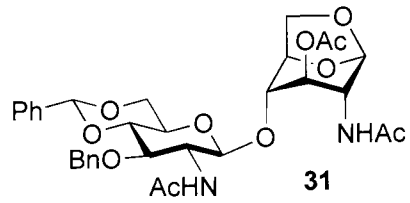



DHL-75
exp2 s2pul
SAMPLE
date Jun 1 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 1200
ct 284
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2250.8
wp 19651.8
vs 243
sc 0
wc 250
hzmm 78.61
is 500.00
rfl 11131.2
rfp 9707.1
th 6
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Ph
O
OAc
BnO
AcHN
NHAc
31
172.235
171.067
169.371
148.471
138.337
137.283
137.201
129.076
128.718
128.310
128.199
127.882
126.042
124.314
101.210
100.798
99.983
81.937
77.485
77.230
76.975
73.976
72.752
72.071
71.155
68.569
66.228
64.575
54.903
48.533
23.583
22.821
21.040
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

DHL-75

Pulse Sequence: dept



31

CH3 carbons

CH2 carbons

CH carbons

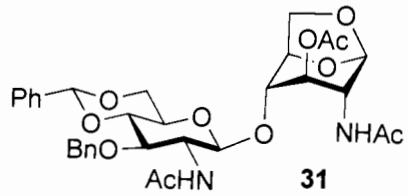
all protonated carbons

100 90 80 70 60 50 40 30 ppm

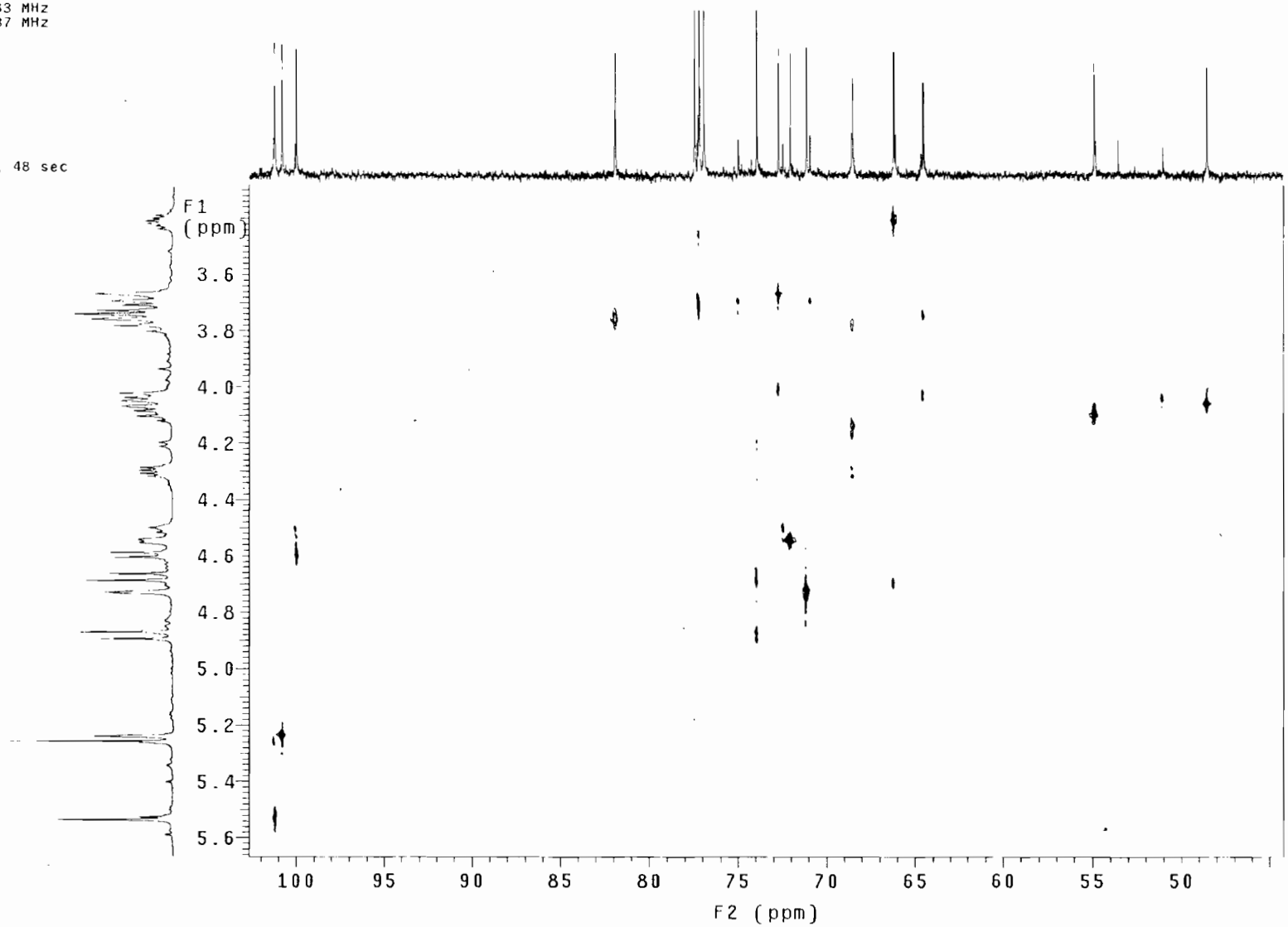
DHL-75

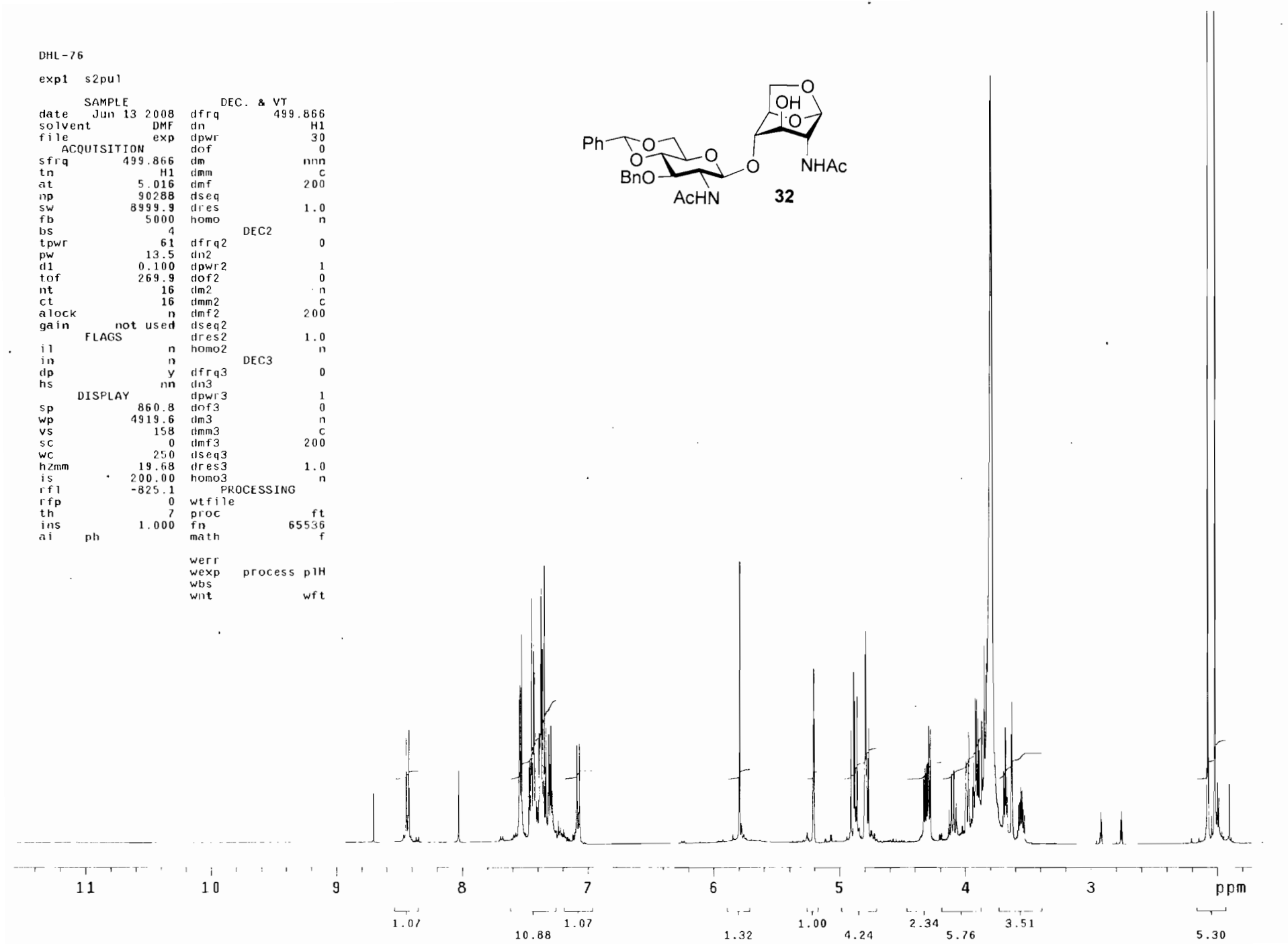
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width   18403.5 Hz
 2D Width 3461.4 Hz
 8 repetitions
 512 increments
OBSERVE C13, 125.6901883 MHz
DECOUPLE H1, 499.8637637 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
F1 DATA PROCESSING
 Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 1 hr, 56 min, 48 sec



31



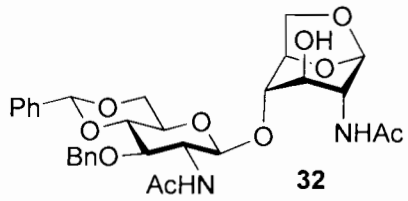

DHL-76
exp1 s2pul
SAMPLE
date Jun 13 2008
solvent DMF
file exp
ACQUISITION
sfrq 499.866
tn H1
at 5.016
np 90288
sw 8999.9
fb 5000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 860.8
wp 4919.6
vs 158
sc 0
wc 250
hzmm 19.68
is 200.00
rfl -825.1
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
Ph
O
OH
BnO
AcHN
NHAc
32
11
10
9
8
7
6
5
4
3
ppm
1.07
10.88
1.07
1.32
1.00
4.24
2.34
5.76
3.51
5.30

DHL-76

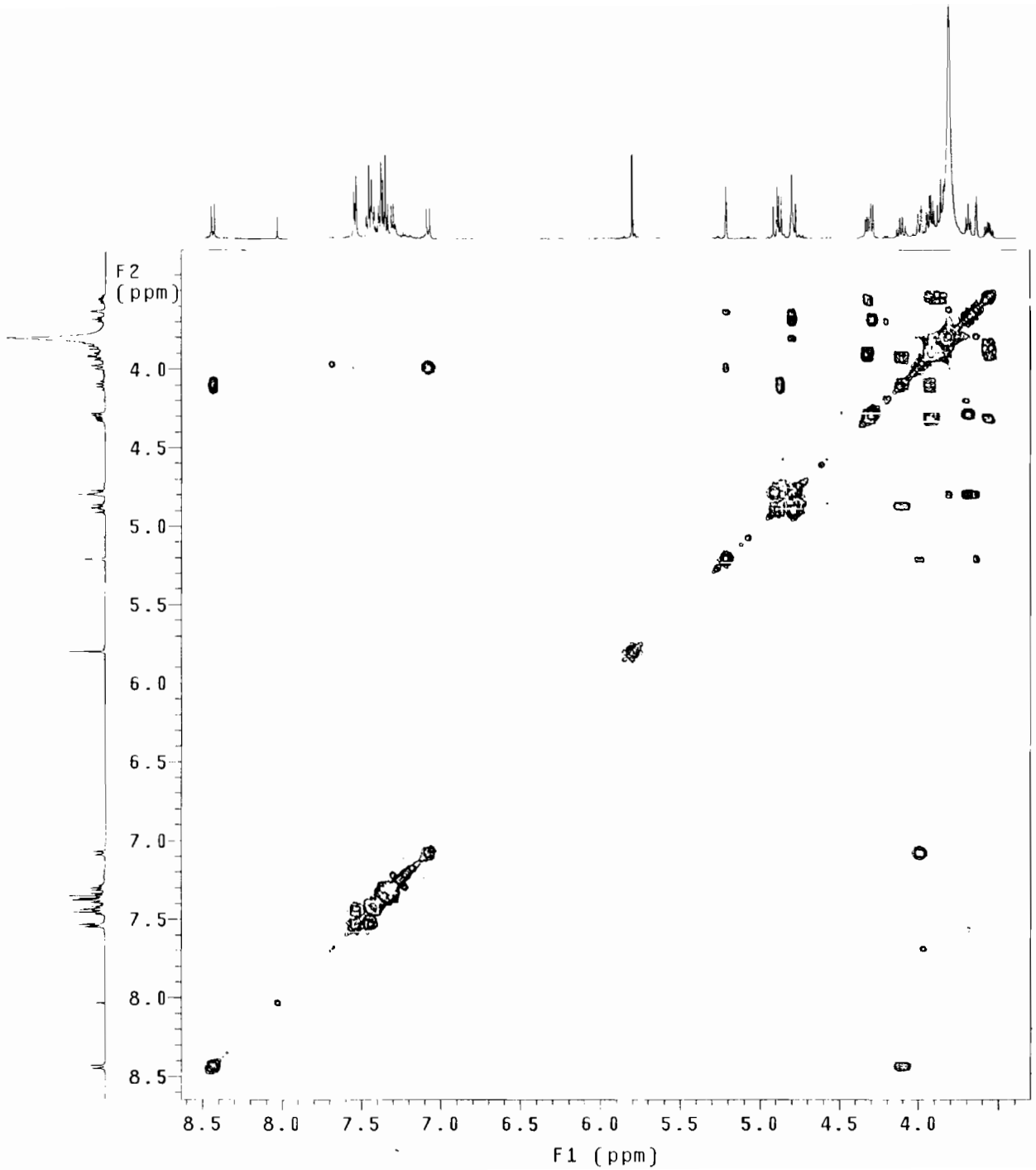
Pulse Sequence: relayh

Solvent: DMF
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.142 sec
Width 3606.9 Hz
2D Width 3606.9 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8607706 MHz
DATA PROCESSING
Sine bell 0.071 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 3 hr, 28 min, 9 sec



32

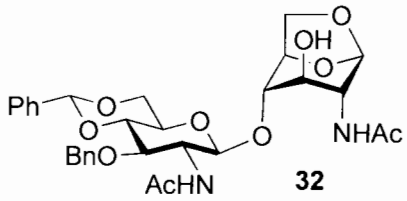


DHL-76

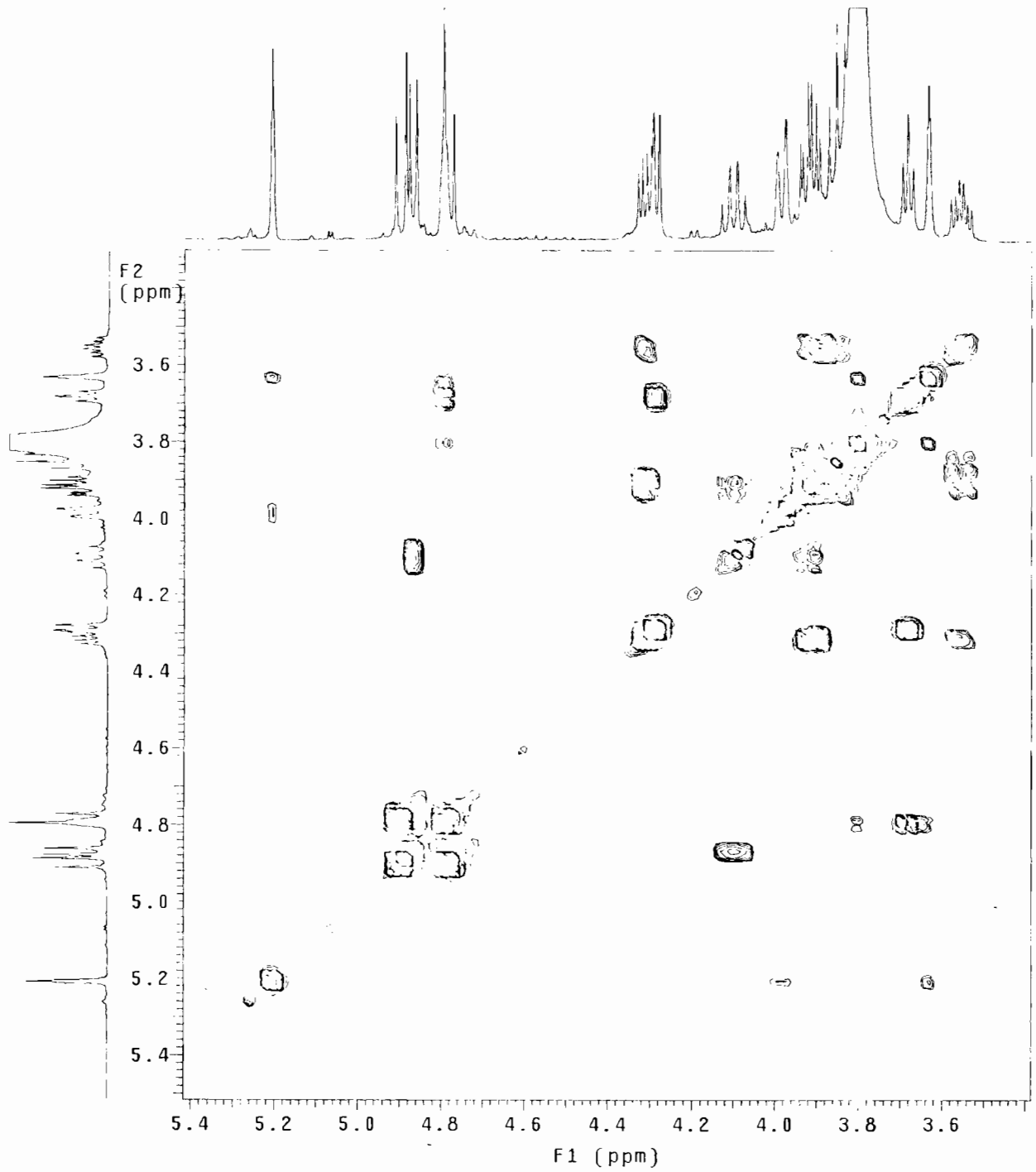
Pulse Sequence: relayh

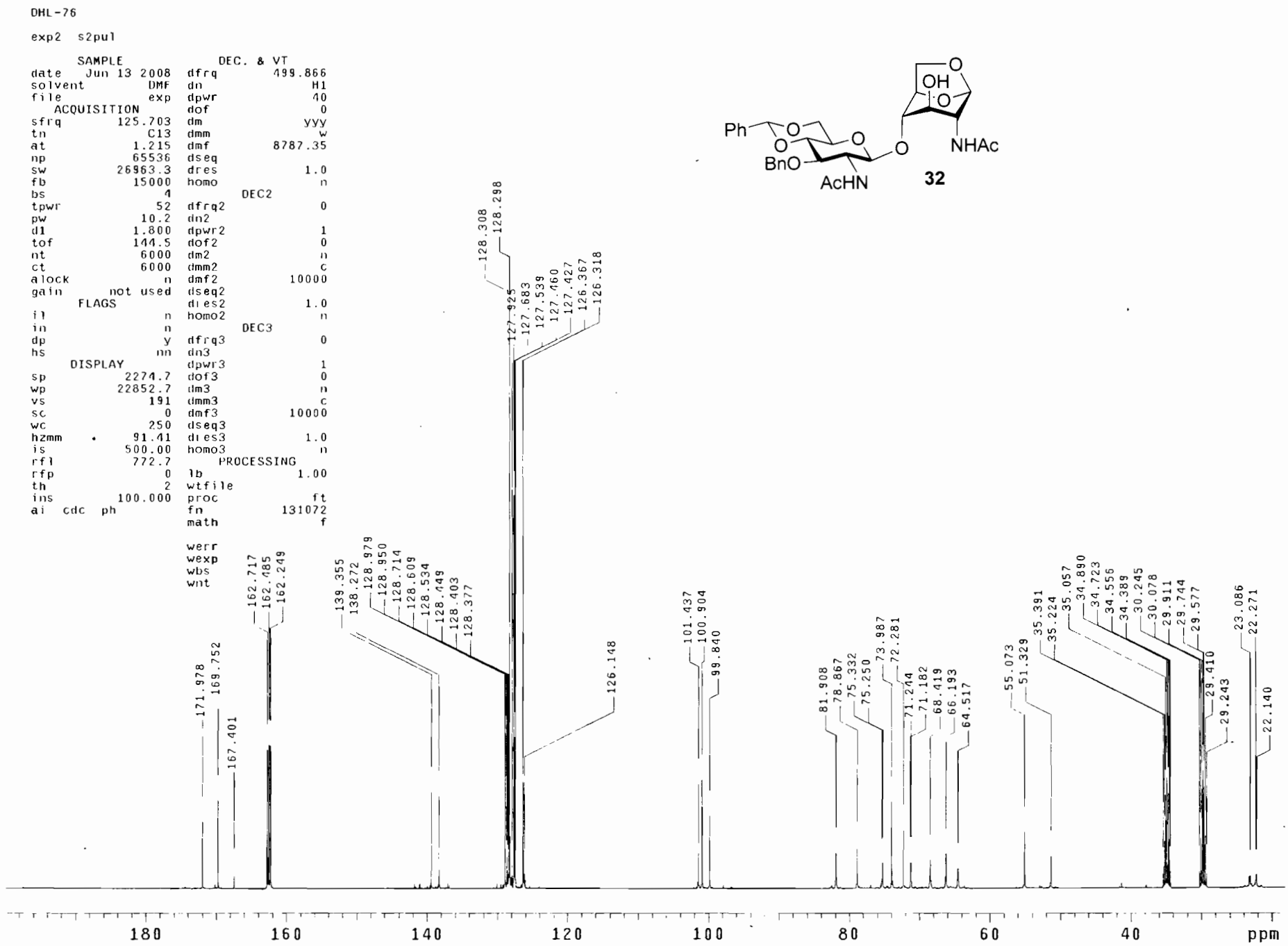
Solvent: DMF
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

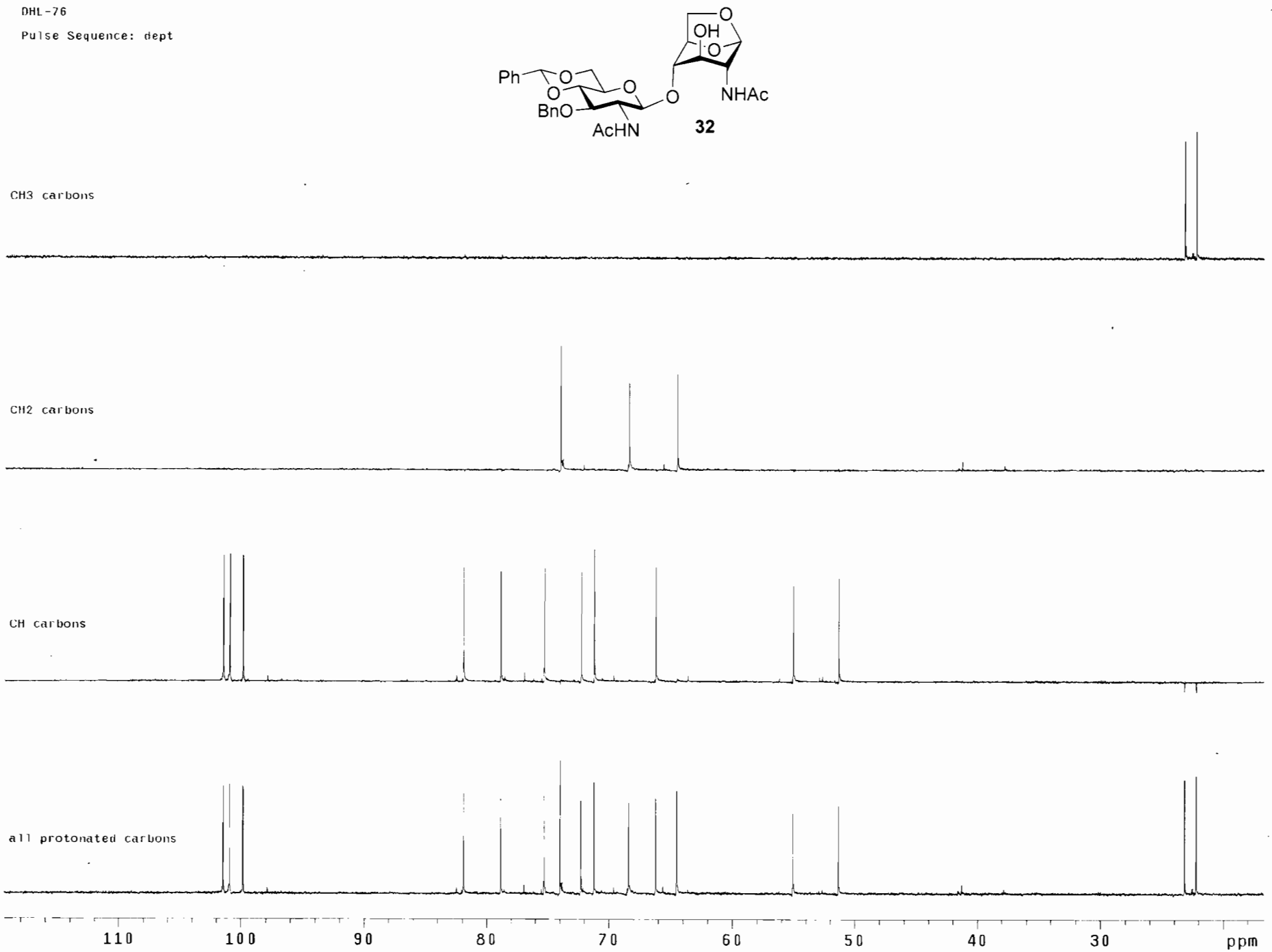
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.142 sec
Width 3606.9 Hz
2D Width 3606.9 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8607706 MHz
DATA PROCESSING
Sine bell 0.071 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 3 hr, 28 min, 9 sec



32




DHL-76
exp2 s2pul
SAMPLE
date Jun 13 2008
solvent DMF
file exp
ACQUISITION
sfrq 125.703
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 6000
ct 6000
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2274.7
wp 22852.7
vs 191
sc 0
wc 250
hzmm 91.41
is 500.00
rfl 772.7
rfp 0
th 2
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Ph
BnO
AcHN
NHAc
OH
32
128.308
128.298
127.925
127.683
127.539
127.460
127.427
126.367
126.318
171.978
169.752
167.401
162.717
162.485
162.249
139.355
138.272
128.979
128.950
128.714
128.609
128.534
128.449
128.403
128.377
126.148
101.437
100.904
99.840
81.908
78.867
75.332
75.250
73.987
72.281
71.244
71.182
68.419
66.193
64.517
55.073
51.329
35.391
35.224
35.057
34.890
34.723
34.556
34.389
30.245
30.078
29.911
29.744
29.577
29.410
29.243
23.086
22.271
22.140
180
160
140
120
100
80
60
40
ppm

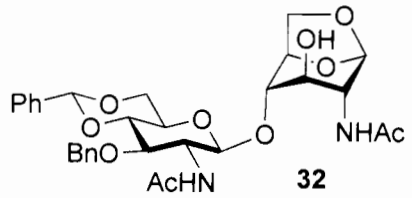

DHL-76
Pulse Sequence: dept
Ph
O
O
O
BnO
AcHN
O
O
OH
O
NHAc
32
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
110
100
90
80
70
60
50
40
30
ppm

DHL-76

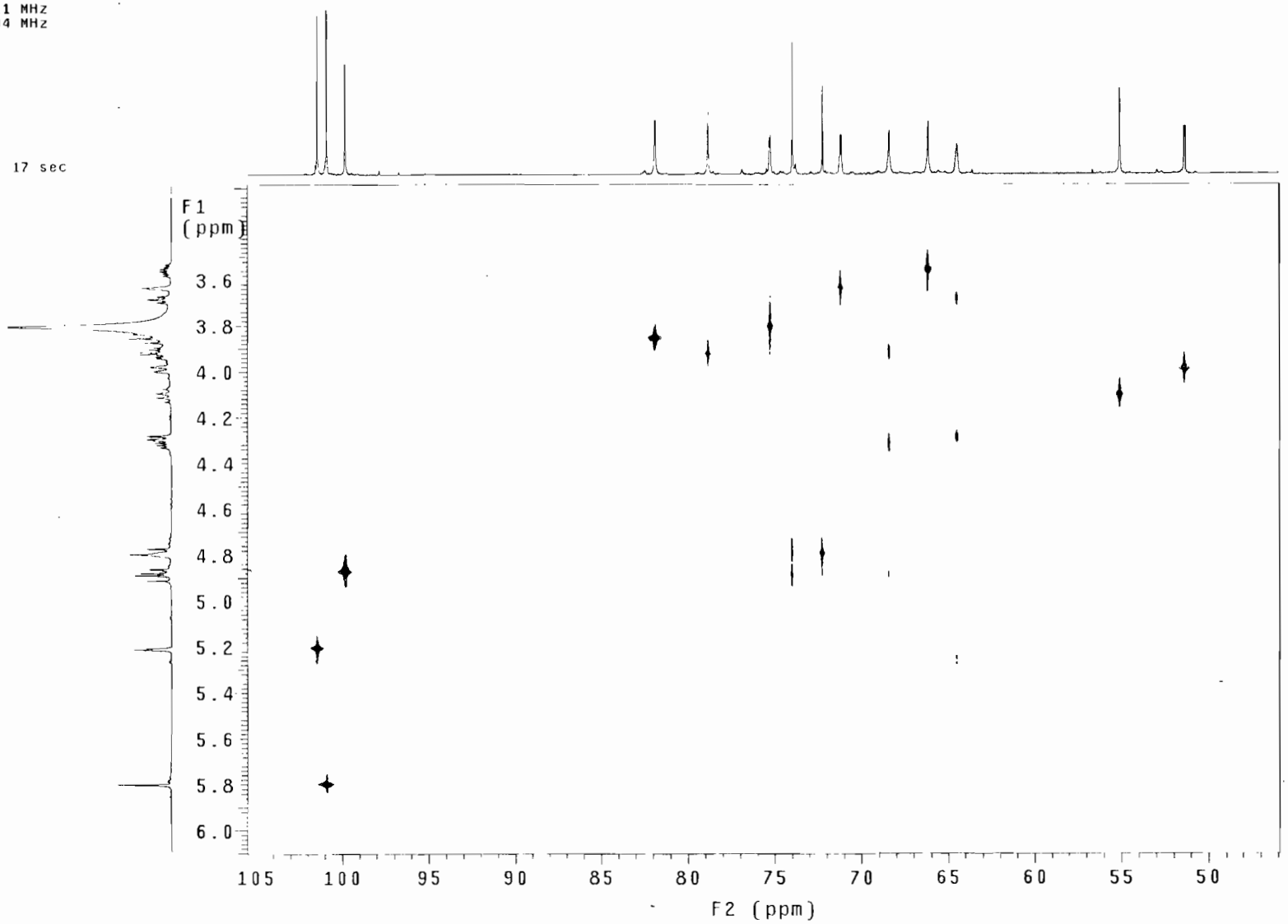
Pulse Sequence: hetcor

Solvent: DMF
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3534.8 Hz
16 repetitions
512 increments
OBSERVE C13, 125.6900811 MHz
DECOUPLE H1, 499.8634194 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 3 hr, 53 min, 17 sec



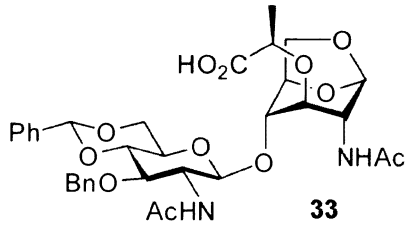
32



DHL-82

expl s2pul

SAMPLE		DEC. & VT	
date	Jun 16 2008	dfrq	499.866
solvent	CD3CN	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.867	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	506.4	dof3	0
wp	4764.6	dm3	n
vs	114	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.06	dres3	1.0
is	143.64	homo3	n
rfl	1493.6	PROCESSING	
rfp	969.7	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



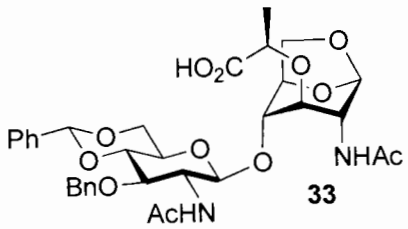
33

OHL-82

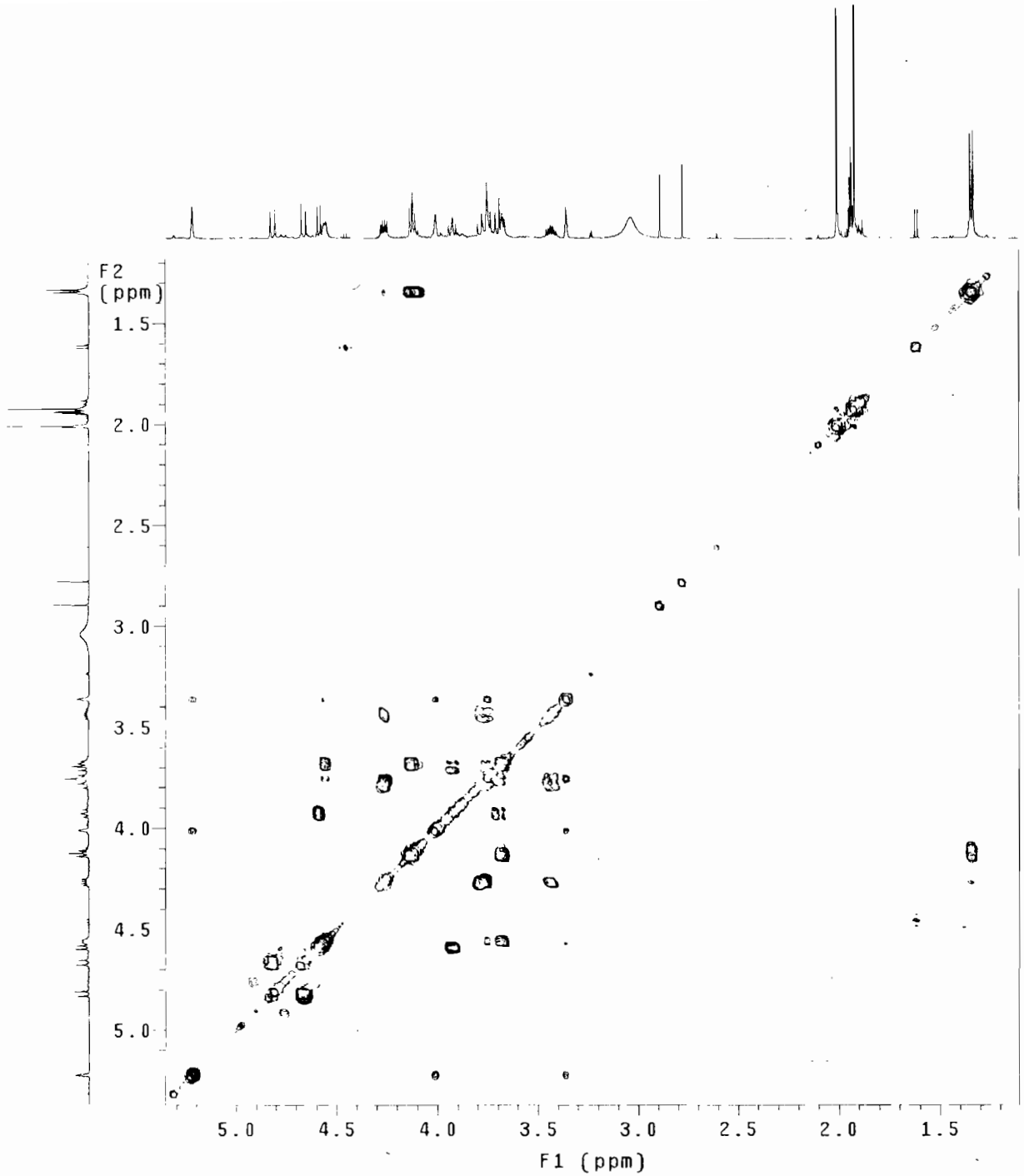
Pulse Sequence: relayh

Solvent: CD3CN
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.138 sec
Width 3708.5 Hz
2D Width 3708.5 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8638327 MHz
DATA PROCESSING
Sine bell 0.069 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 3 hr, 27 min, 21 sec



33

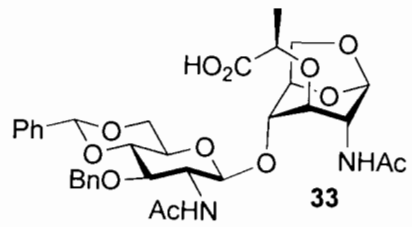


DHL-82

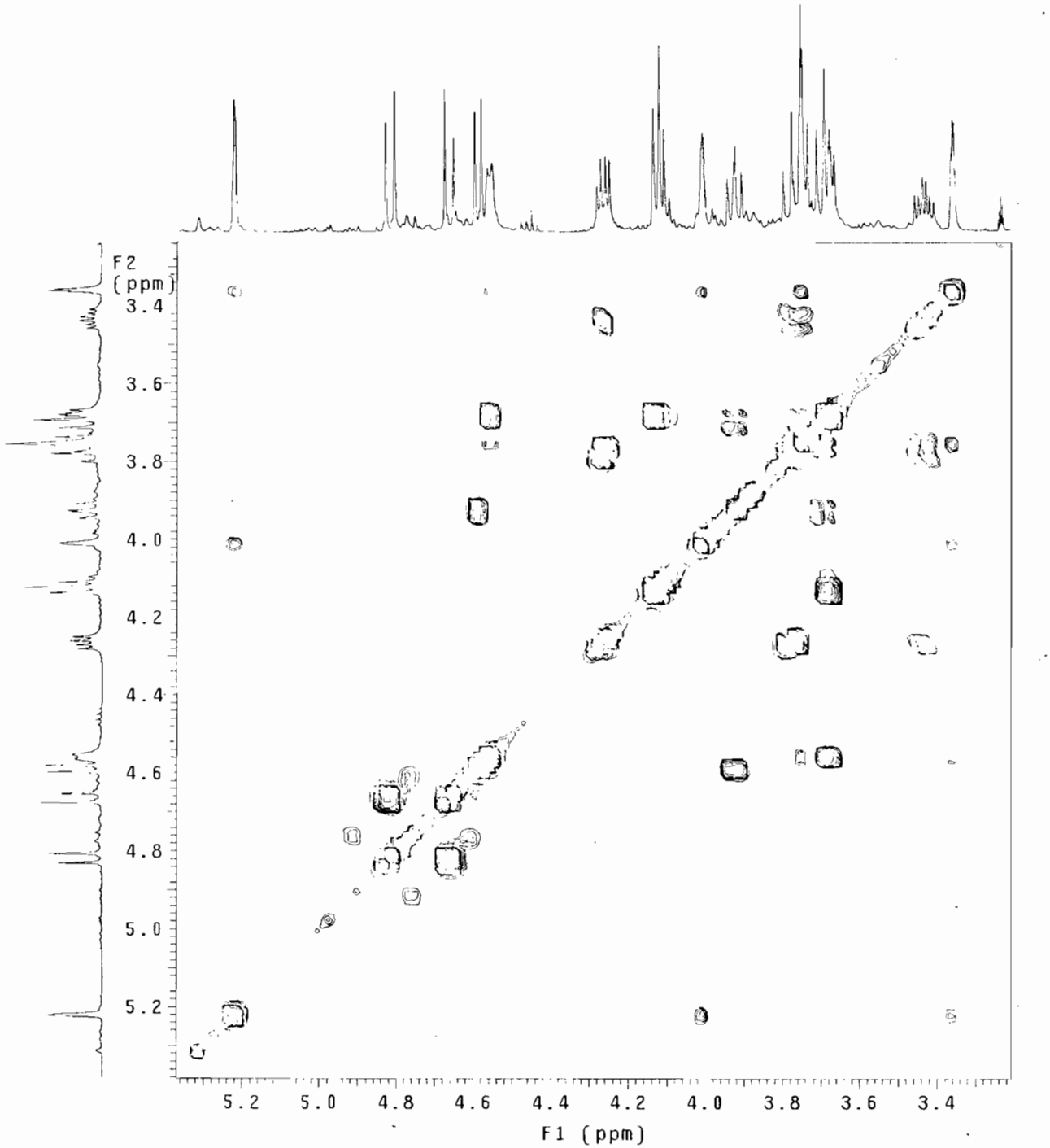
Pulse Sequence: relayh

Solvent: CD3CN
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.138 sec
Width 3708.5 Hz
2D Width 3708.5 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8638327 MHz
DATA PROCESSING
Sine bell 0.069 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 3 hr, 27 min, 21 sec



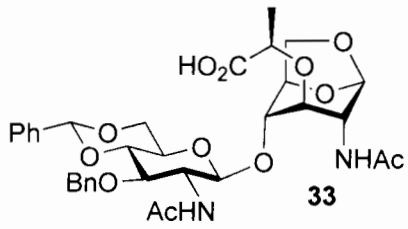
33



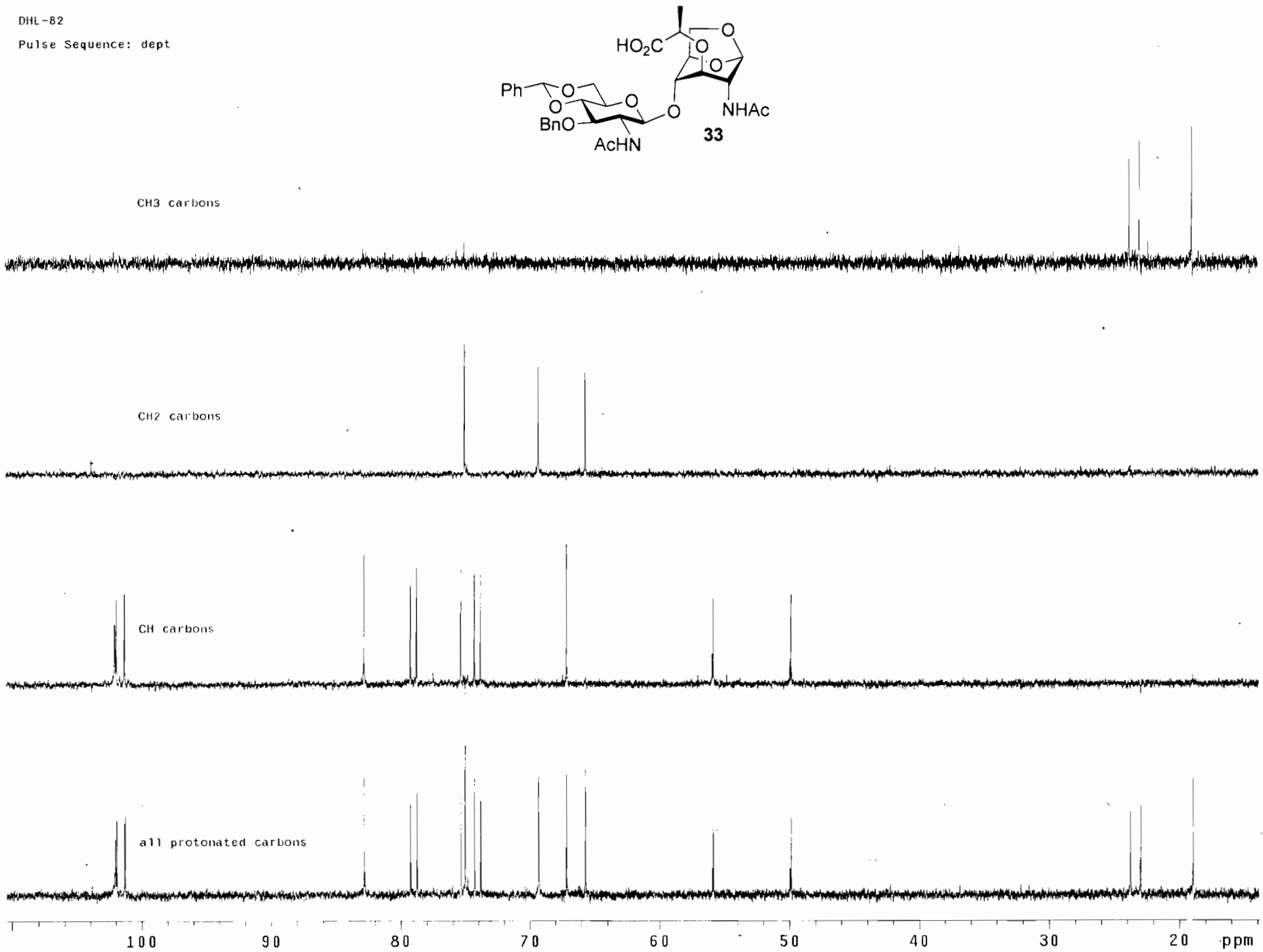
DHL-82

exp7 s2pul

SAMPLE		DEC. & VT	
date	Jun 16 2008	dfrq	499.866
solvent	cd3cn	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	6000	dm2	n
ct	5145	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1686.3	dof3	0
wp	21577.3	dm3	n
vs	319	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	86.31	dres3	1.0
is	500.00	homo3	n
rfl	1290.0	PROCESSING	
rfp	0	lb	1.00
th	4	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



33

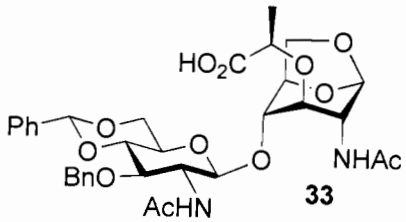

DHL-82
Pulse Sequence: dept
HO2C
Ph
BnO
AcHN
NHAc
33
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
100
90
80
70
60
50
40
30
20
ppm

DHL-82

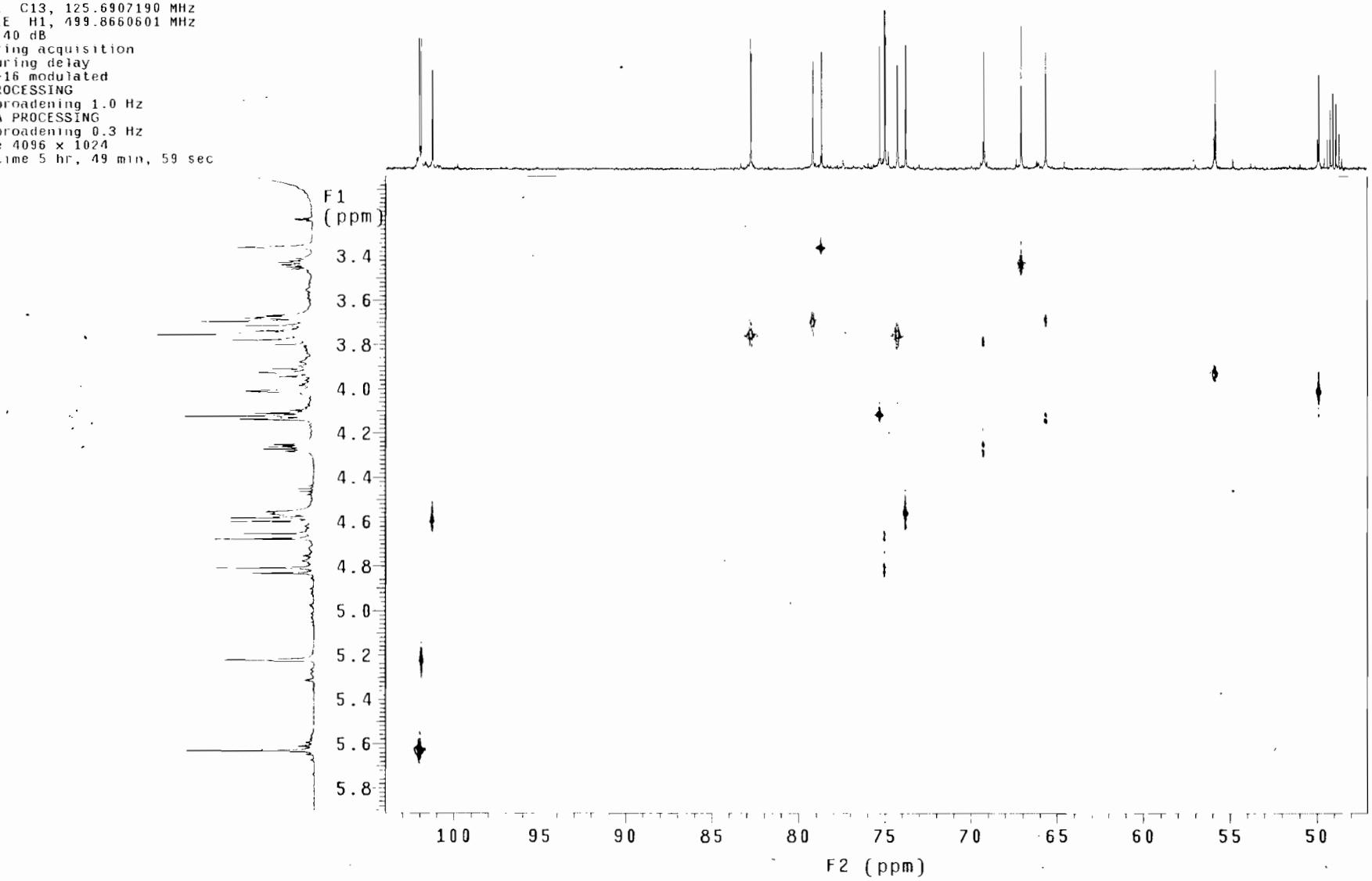
Pulse Sequence: hetcor

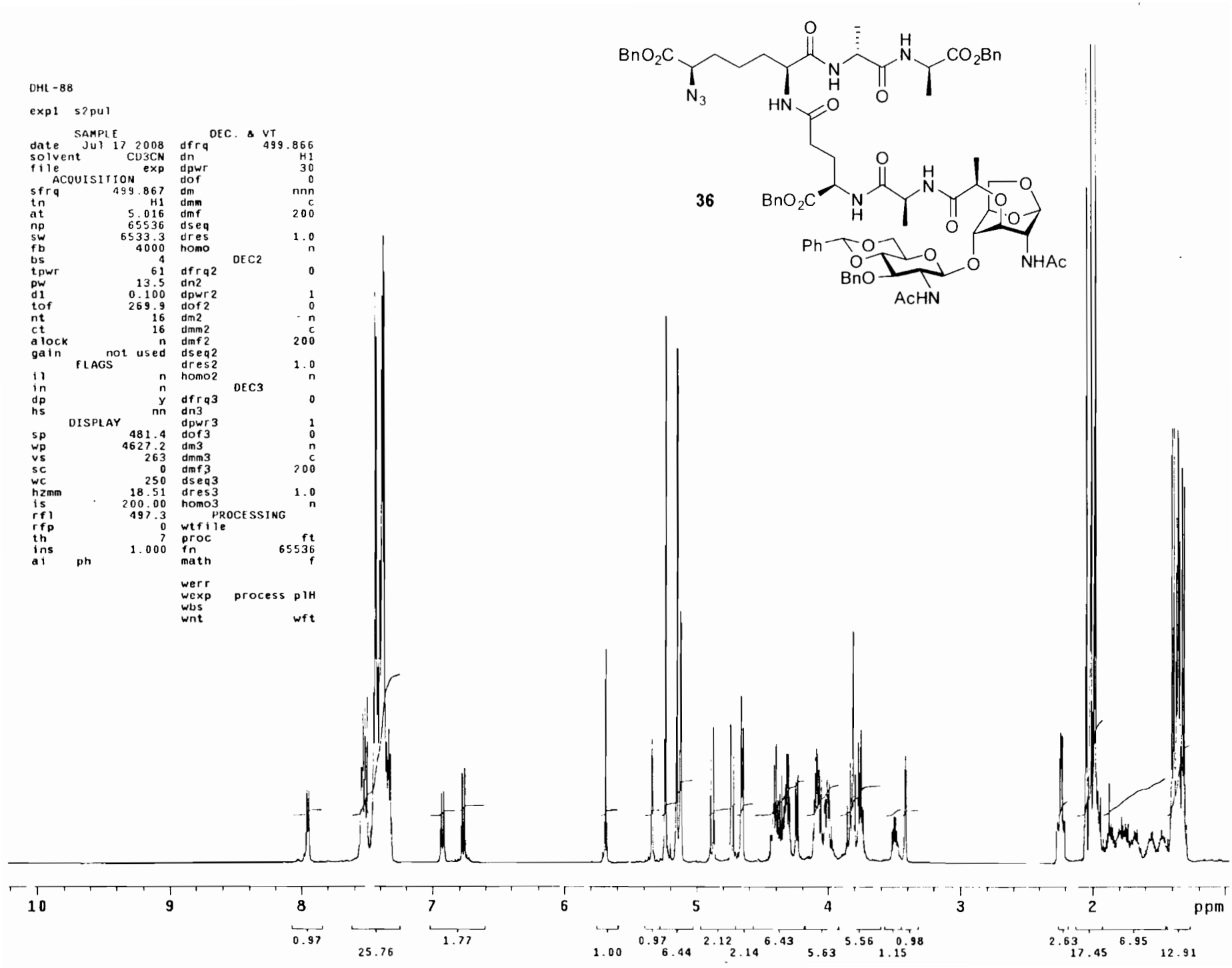
Solvent: cd3cn
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3511.2 Hz
24 repetitions
512 increments
OBSERVE C13, 125.6907190 MHz
DECOUPLE H1, 499.8660601 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 5 hr, 49 min, 59 sec



33



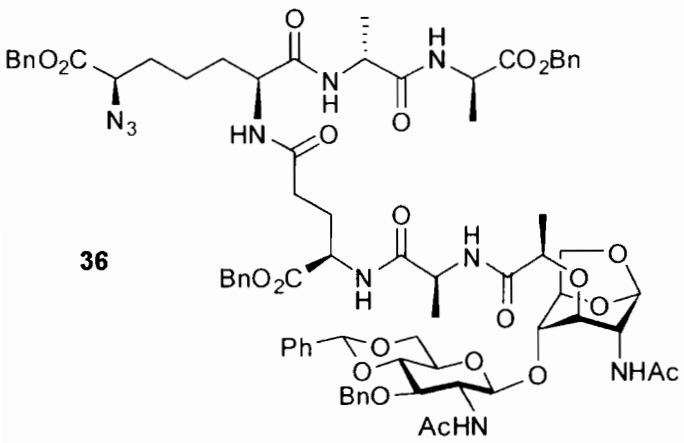

DHL-88
expl s2pul
SAMPLE
date Jul 17 2008
solvent CD3CN
file exp
ACQUISITION
sfrq 499.867
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 481.4
wp 4627.2
vs 263
sc 0
wc 250
hzmm 18.51
is 200.00
rfl 497.3
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
36
10
9
8
7
6
5
4
3
2
ppm
0.97
25.76
1.77
1.00
0.97
6.44
2.12
2.14
6.43
5.63
5.56
1.15
0.98
2.63
17.45
6.95
12.91

DHL-88

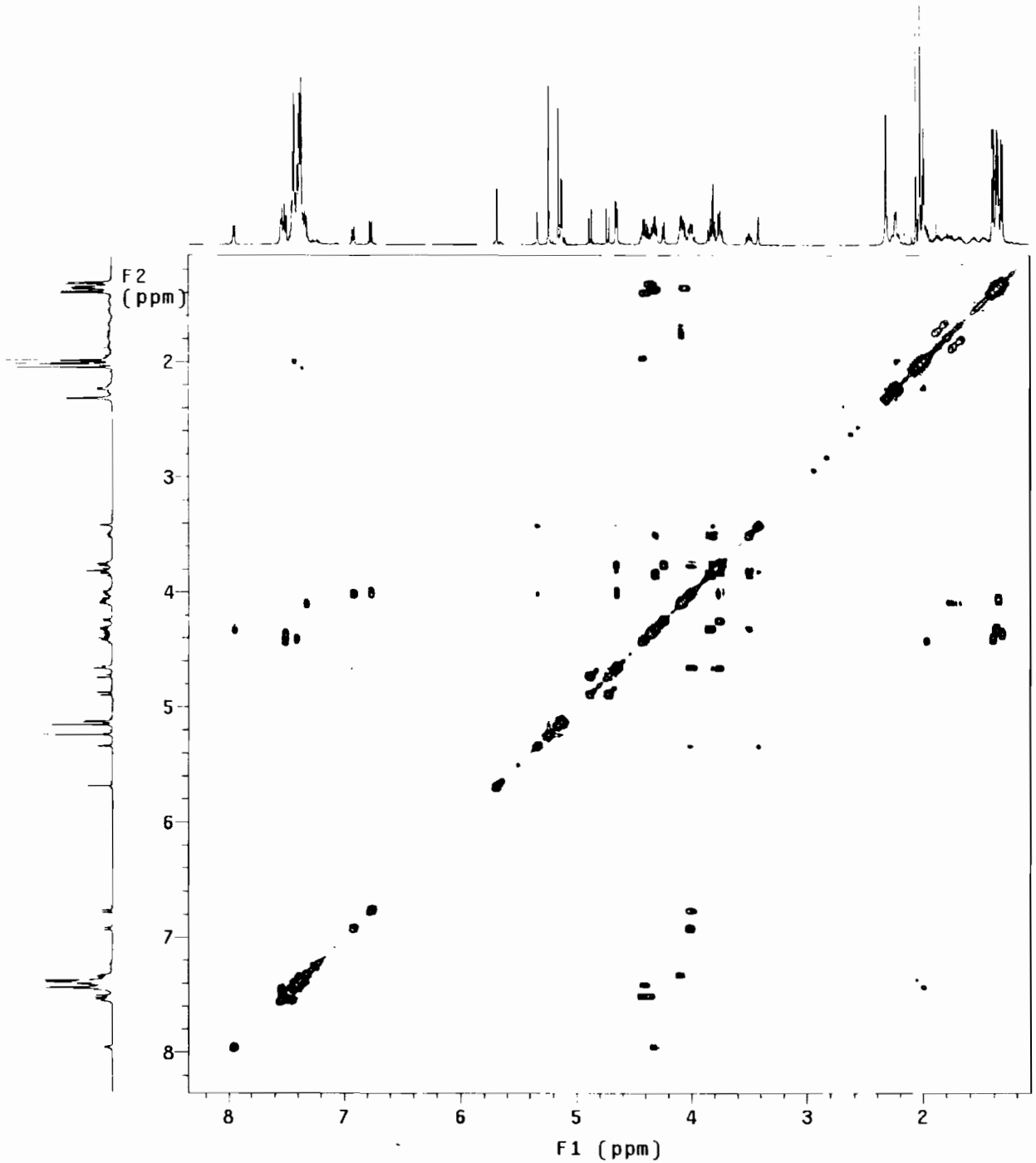
Pulse Sequence: relayh

Solvent: CD3CN
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.141 sec
Width 3640.0 Hz
2D Width 3640.0 Hz
8 repetitions
512 increments
OBSERVE H1, 499.8638062 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 1 hr, 44 min, 2 sec



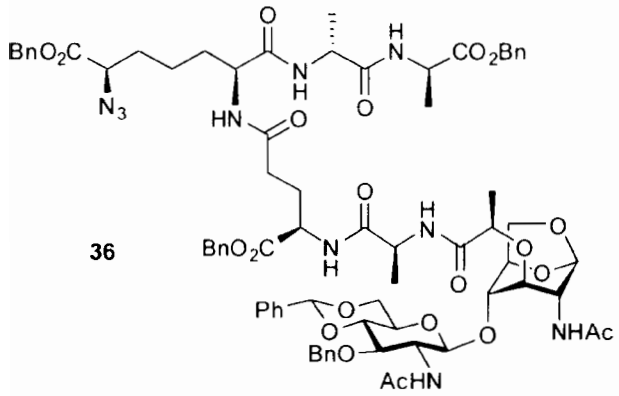
36



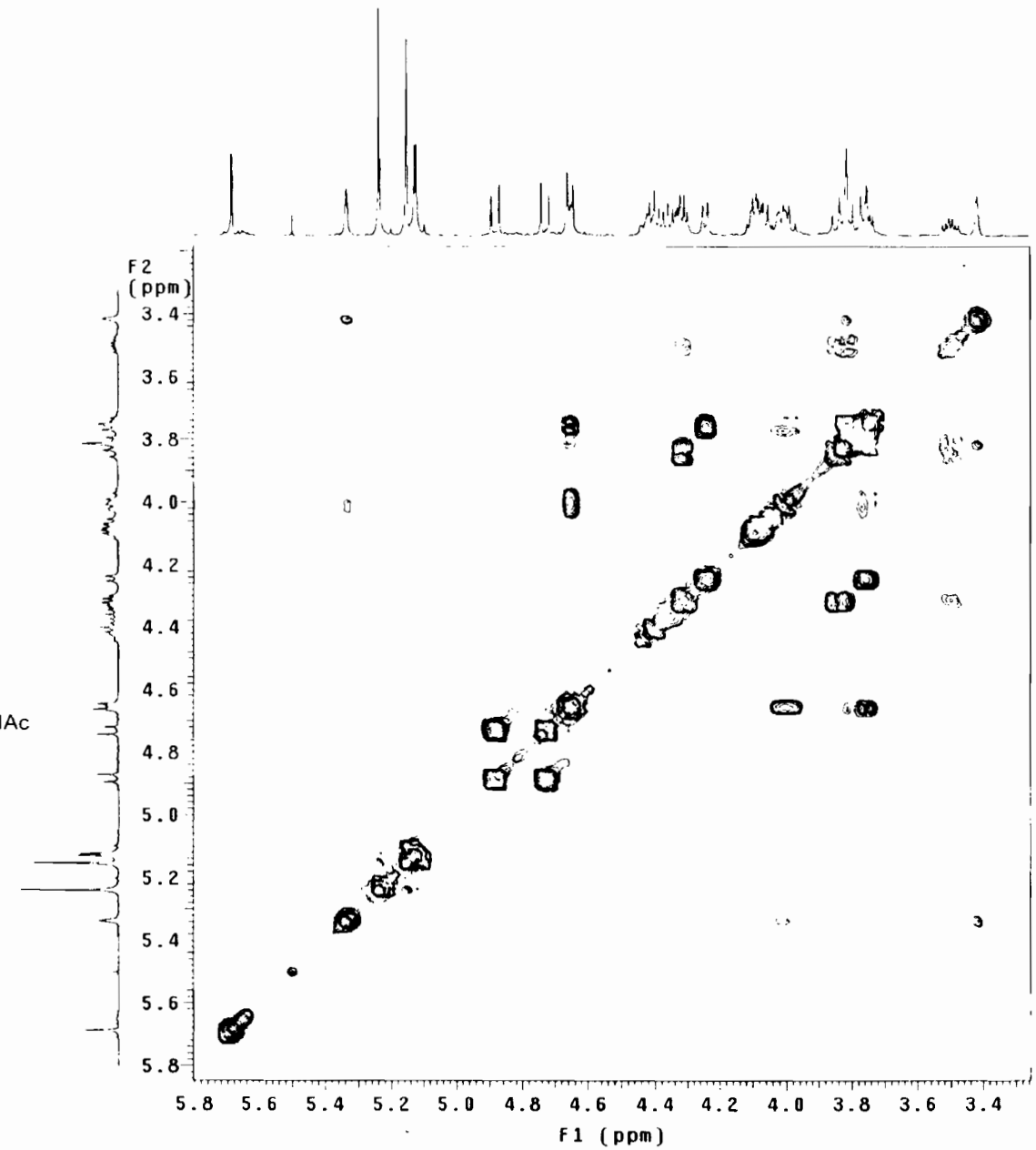
DHL-88

Pulse Sequence: relayh
Solvent: CD3CN
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.141 sec
Width 3640.0 Hz
2D Width 3640.0 Hz
8 repetitions
512 increments
OBSERVE H1, 499.8638062 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 1 hr, 44 min, 2 sec



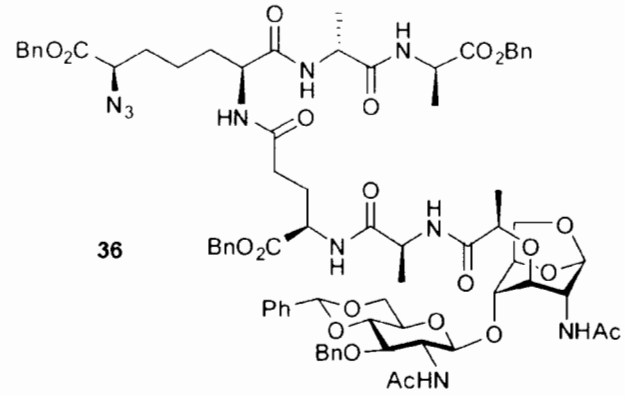
36

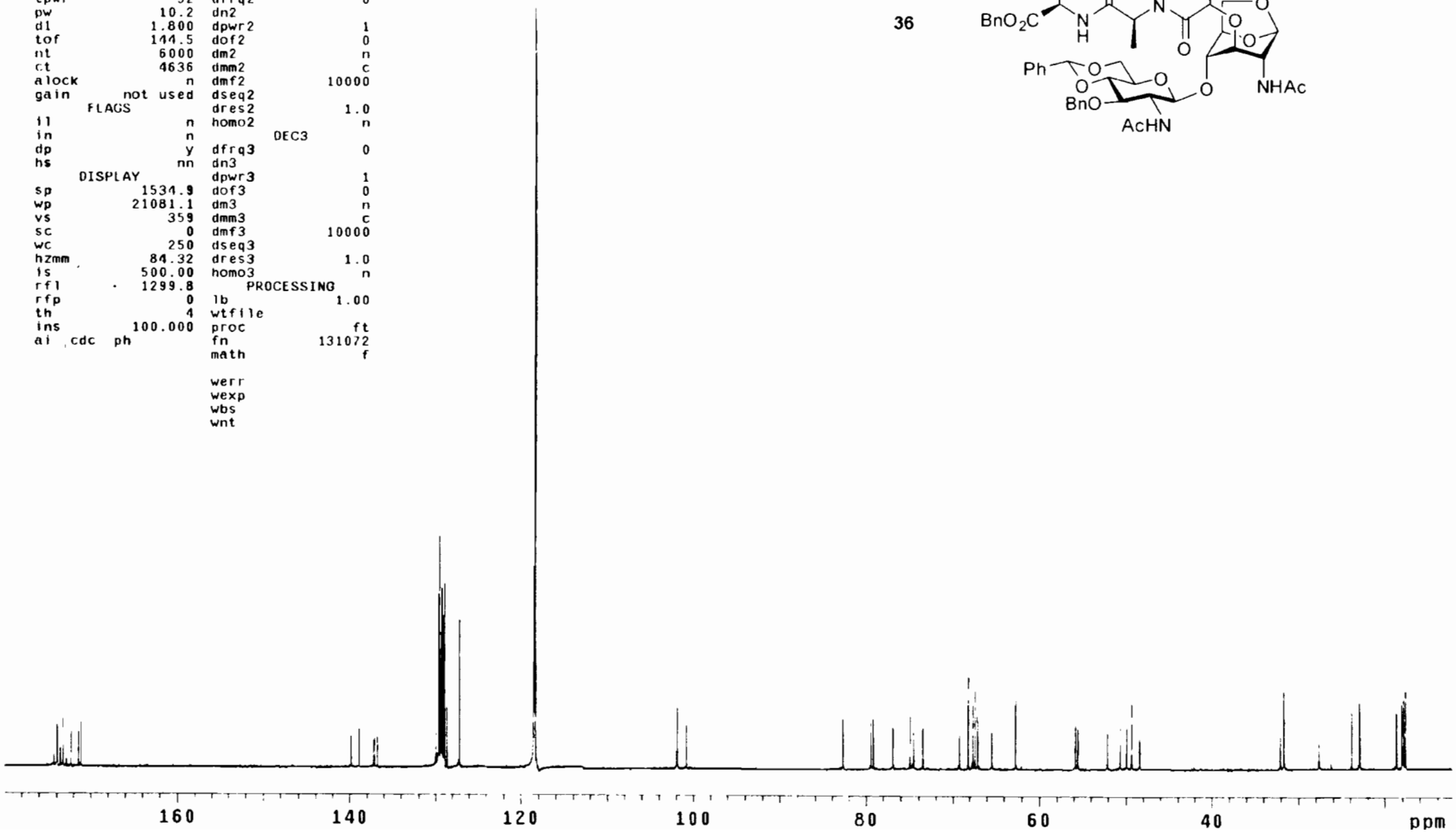


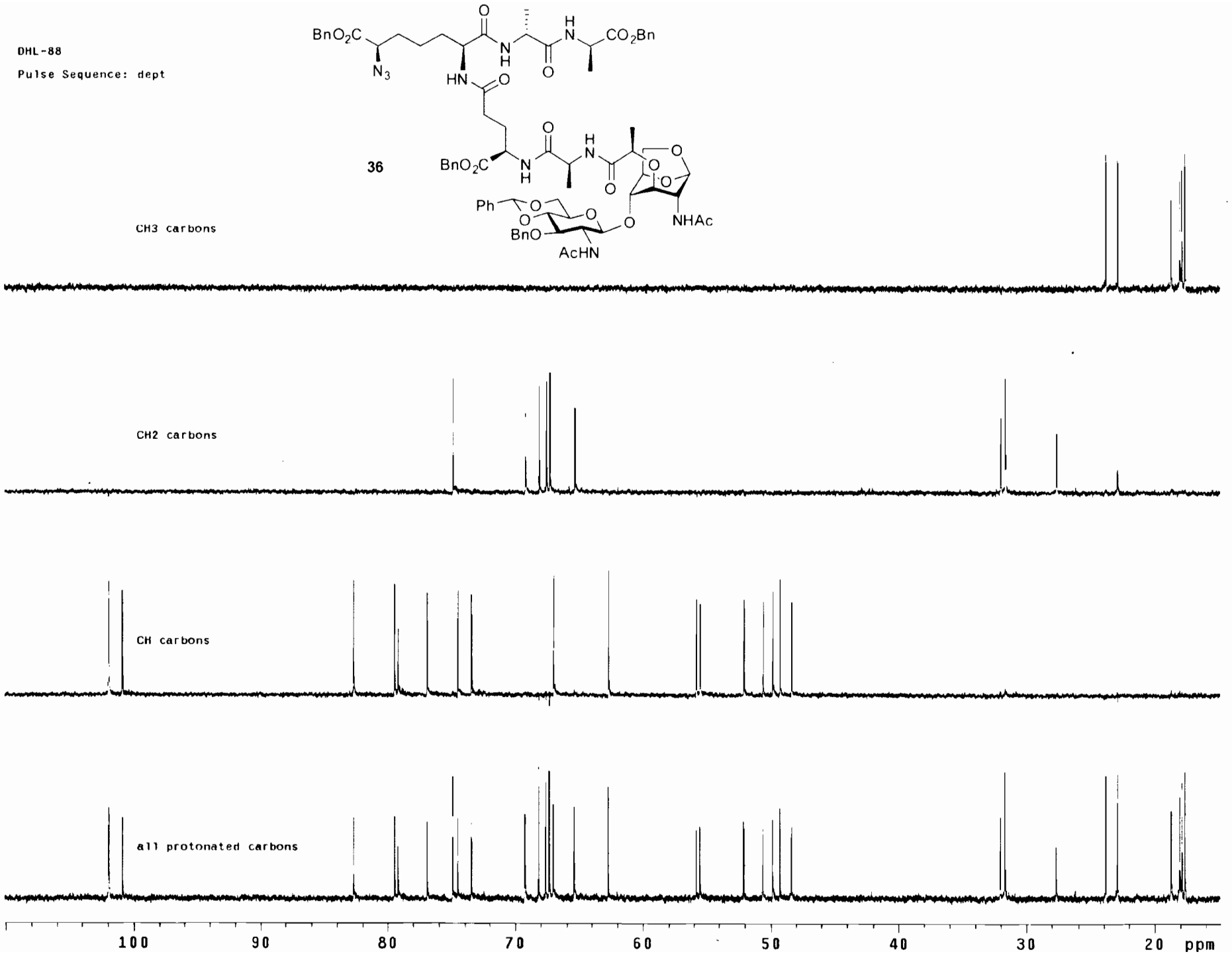
DHL-88

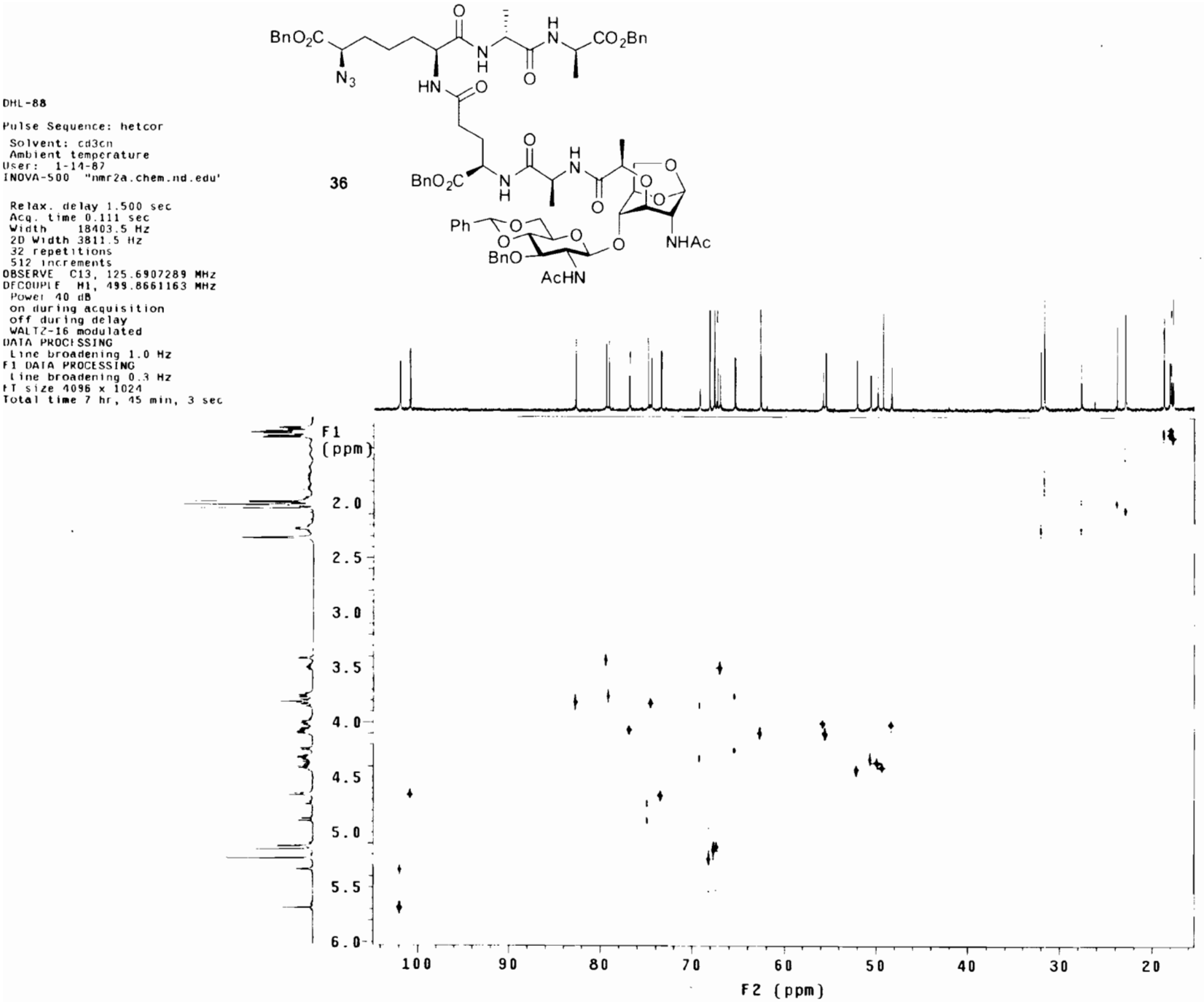
exp7 s2pul

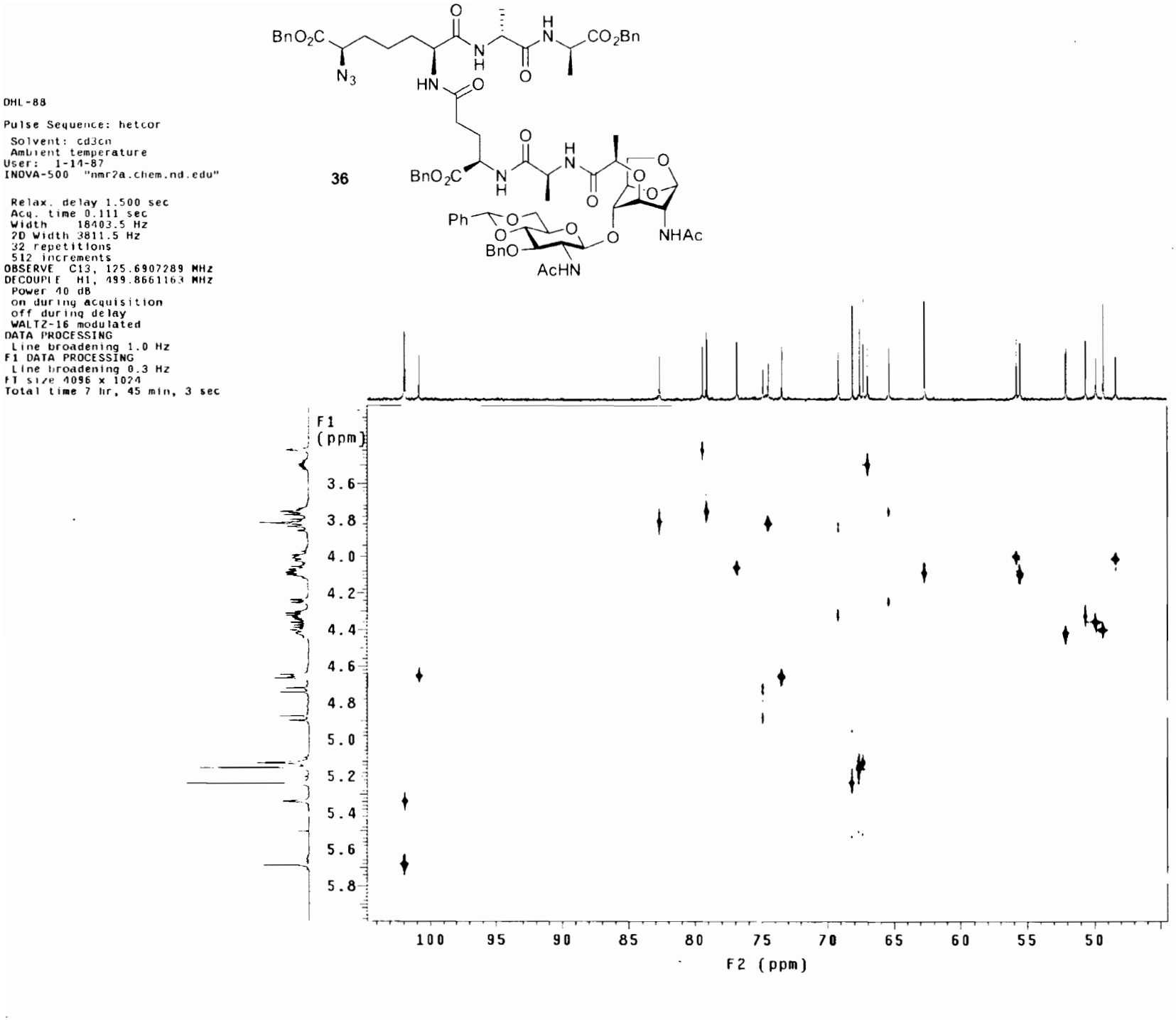
SAMPLE		DEC. & VT	
date	Jul 17 2008	dfrq	499.866
solvent	cd3cn	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	64	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	6000	dm2	n
ct	4636	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1534.9	dof3	0
wp	21081.1	dm3	n
vs	359	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	84.32	dres3	1.0
is	500.00	homo3	n
rfl	1299.8	PROCESSING	
rfp	0	lb	1.00
th	4	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	






DHL-88
Pulse Sequence: dept
36
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
100
90
80
70
60
50
40
30
20
ppm

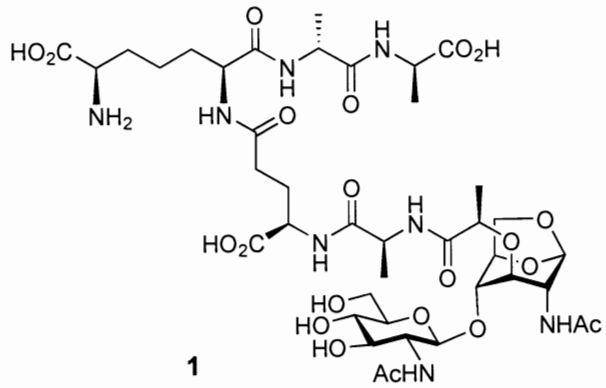

DHL-88
Pulse Sequence: hetcor
Solvent: cd3cn
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3811.5 Hz
32 repetitions
512 increments
OBSERVE C13, 125.6907289 MHz
DECOUPLE H1, 499.8661163 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 7 hr, 45 min, 3 sec
36
F1 (ppm)
F2 (ppm)


DHL-88
Pulse Sequence: hetcor
Solvent: cd3cn
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3811.5 Hz
32 repetitions
512 increments
OBSERVE C13, 125.6907289 MHz
DECOUPLE H1, 499.8661163 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 7 hr, 45 min, 3 sec
36
F1 (ppm)
F2 (ppm)

DHL-89

exp3 Proton

SAMPLE		SPECIAL	
date	Oct 17 2008	temp	22.0
solvent	d2o	gain	not used
file	/afs/nd.edu/u~	spin	not used
ser26/dhesek/Priva~		hst	0.008
te/DHL/DHL-89H.fid		pw90	10.400
ACQUISITION		alfa	10.000
sw	4807.7	**FLAGS**	
at	3.408	il	n
np	32768	in	n
fb	4000	dp	y
bs	4	hs	nn
d1	0.600	**PROCESSING**	
nt	32	fn	65536
ct	32	**DISPLAY**	
TRANSMITTER		sp	649.8
tn	H1	wp	3635.8
sfrq	599.877	rfl	2495.0
tof	-299.9	rfp	2885.4
tpwr	61	rp	19.9
pw	10.400	lp	3.7
DECOUPLER		**PLOT**	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	1592
dmm	c	th	9
dpwr	38	ai cdc ph	
dmf	35088		



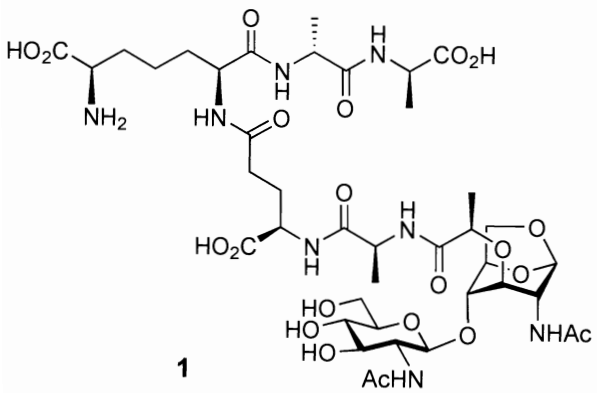
1

Std proton

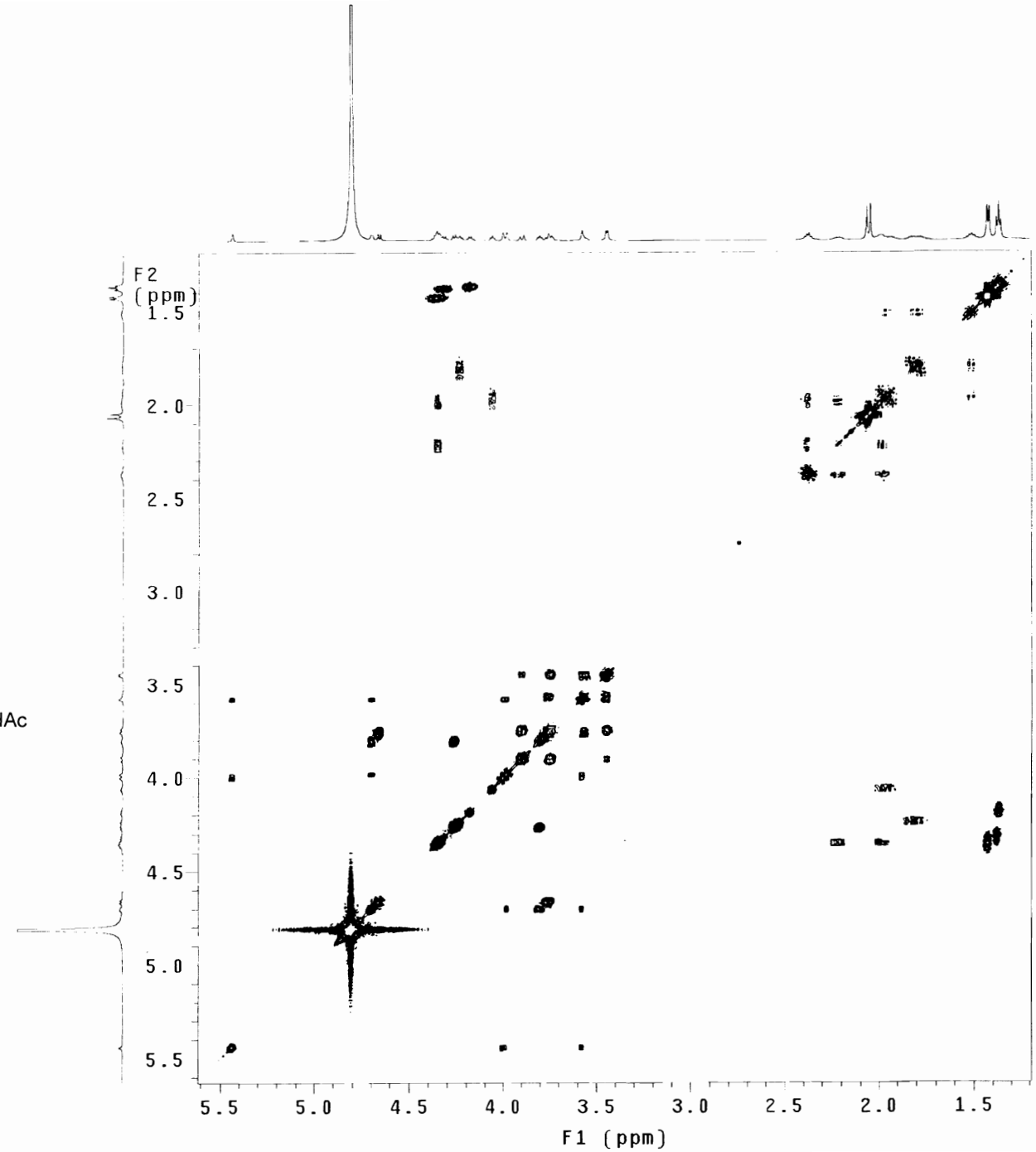
File: xp

Pulse Sequence: gCOSY
Solvent: d2o
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 1.300 sec
Acq. time 0.213 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
12 repetitions
512 increments
OBSERVE H1, 599.8743040 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.191 sec
FT size 8192 x 8192
Total time 2 hr, 42 min, 32 sec



1

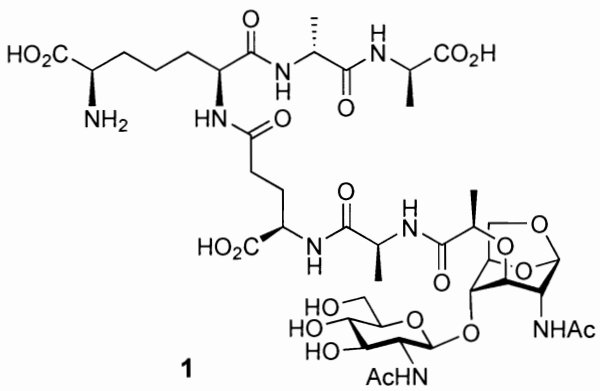


Std proton

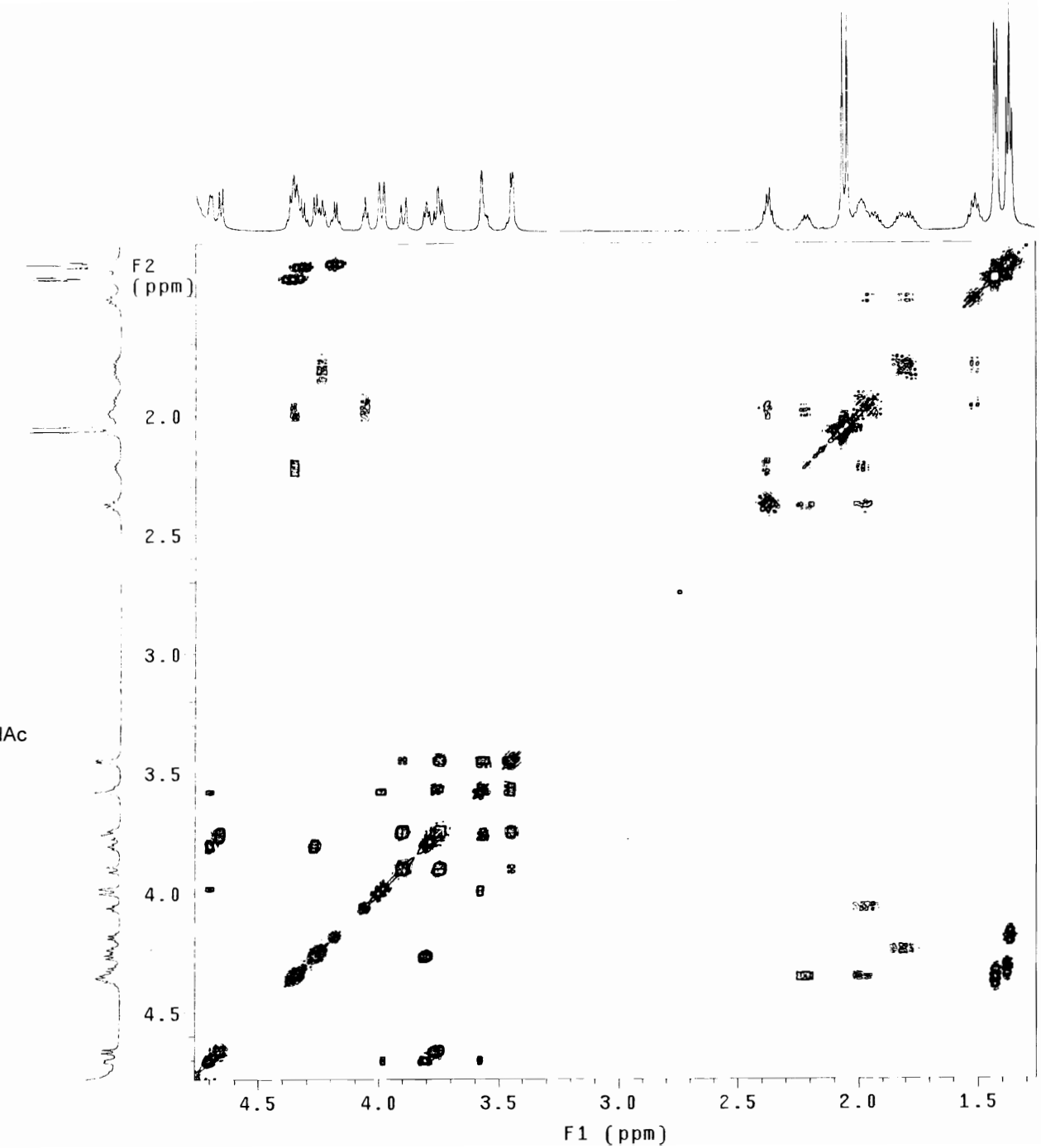
File: xp

Pulse Sequence: gCOSY
Solvent: d2o
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 1.300 sec
Acq. time 0.213 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
12 repetitions
512 increments
OBSERVE H1, 599.8743040 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.191 sec
FT size 8192 x 8192
Total time 2 hr, 42 min, 32 sec



1

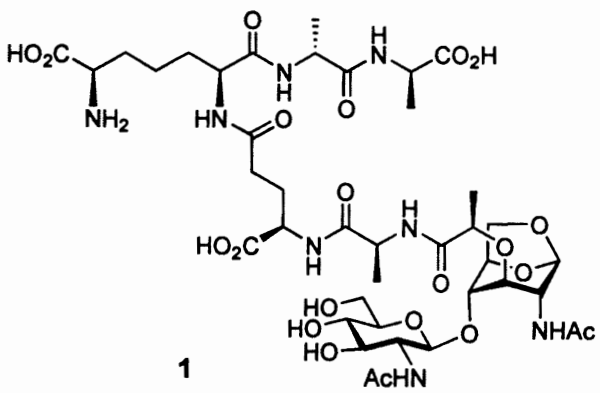


Std proton

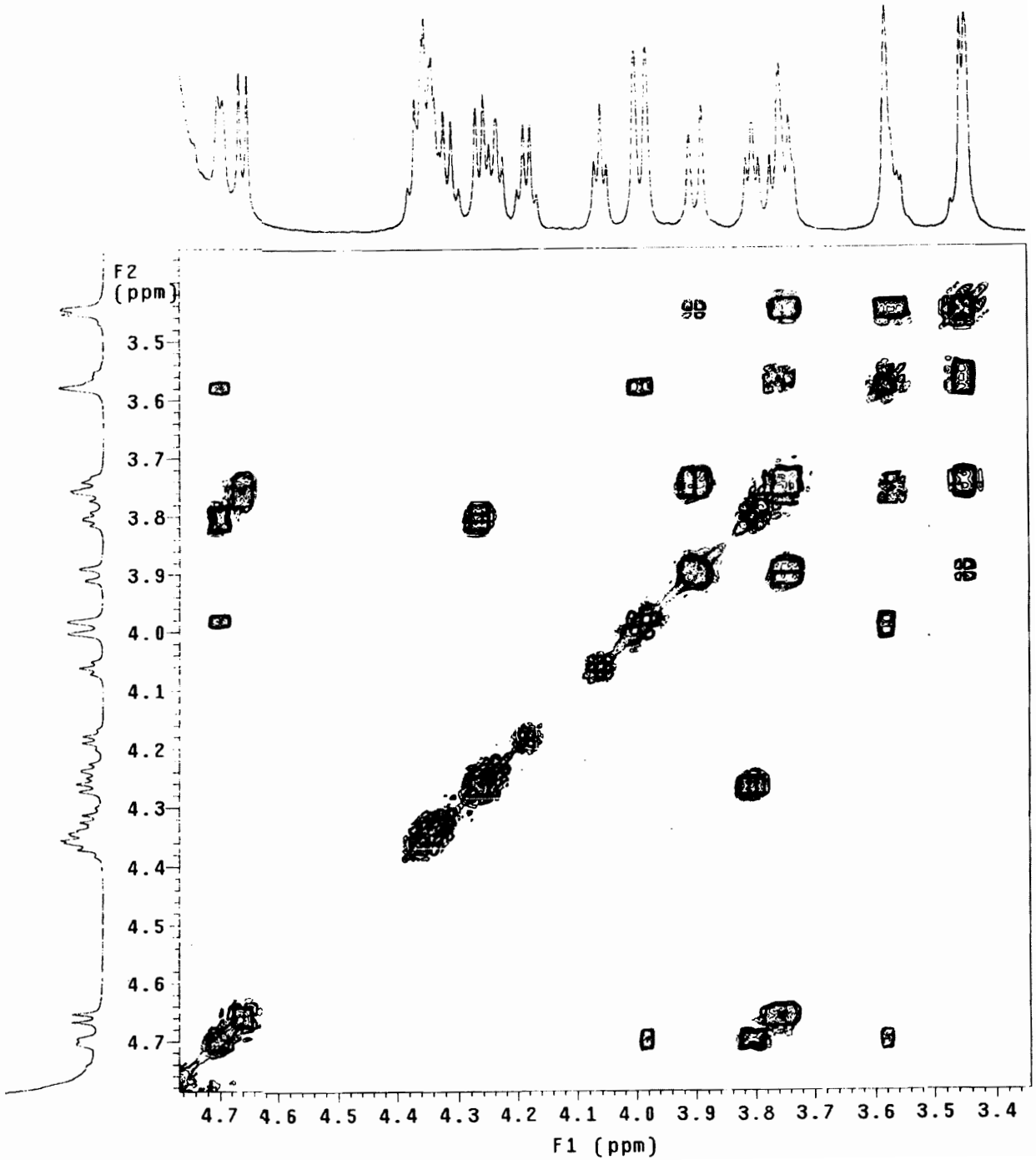
File: xp

Pulse Sequence: gCOSY
Solvent: d2o
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 1.300 sec
Acq. time 0.213 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
12 repetitions
512 increments
OBSERVE H1, 599.8743040 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.191 sec
FT size 8192 x 8192
Total time 2 hr, 42 min, 32 sec



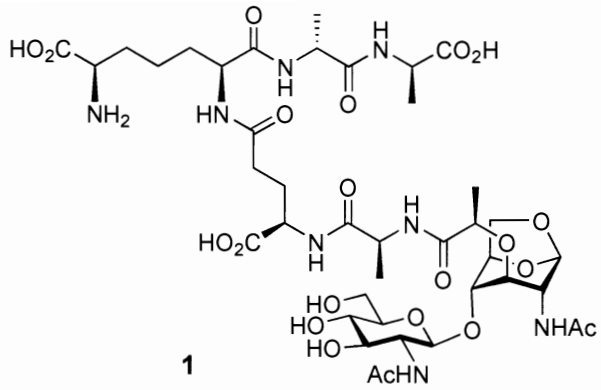
1



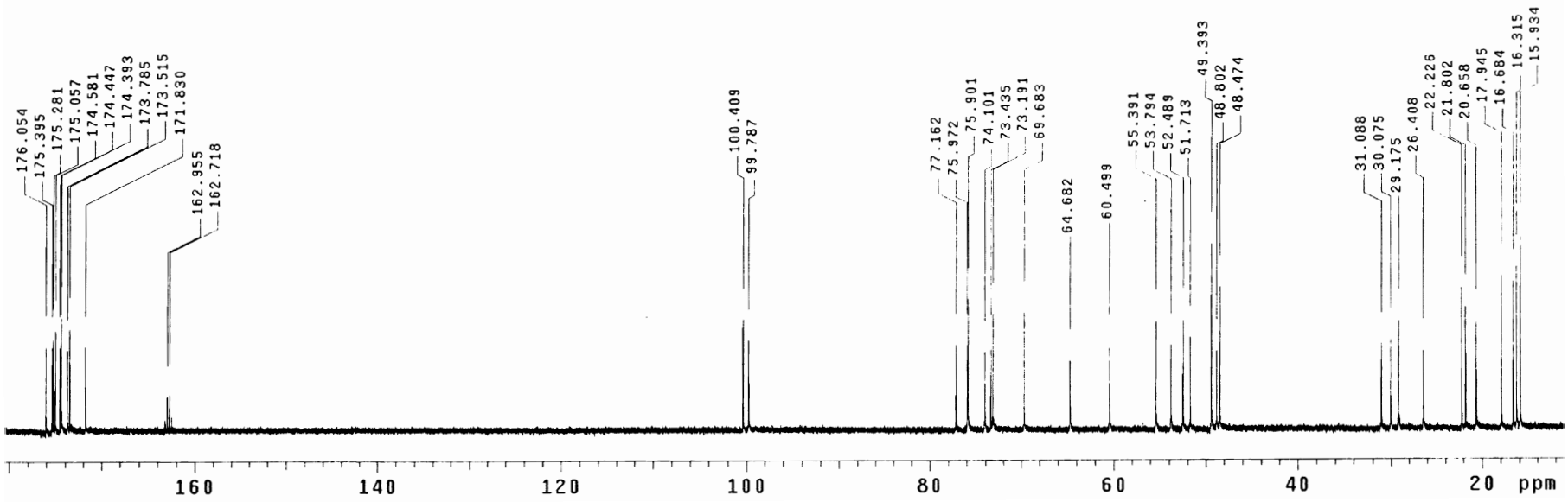
DHL-89

exp4 Carbon

SAMPLE		SPECIAL	
date	Oct 17 2008	temp	22.0
solvent	d2o	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	7.500
at	1.783	alfa	10.000
np	108694	**FLAGS**	
fb	17000	il	n
bs	4	in	n
d1	1.220	dp	y
nt	20000	hs	nn
ct	20000	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	262144
sfrq	150.852	**DISPLAY**	
tof	-719.9	sp	1678.3
tpwr	58	wp	25524.3
pw	7.500	rfl	1668.4
DECOUPLER		rfp	0
dn	H1	rp	135.4
dof	0	lp	-0
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	44	sc	0
dmf	15094	vs	4182
		th	5
		ai cdc ph	



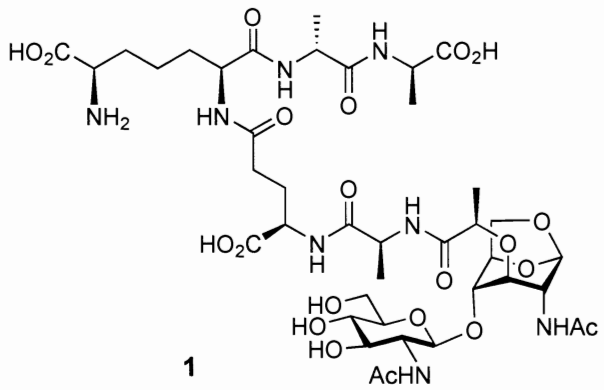
1



DHL-89

File: xp

Pulse Sequence: DEPT



1

CH3 carbons

CH2 carbons

CH carbons

all protonated carbons

100 90 80 70 60 50 40 30 20 ppm

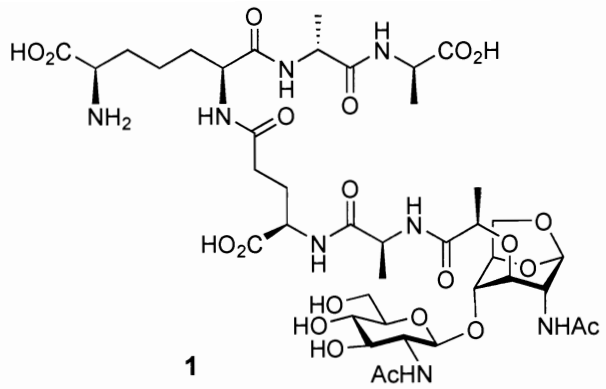
DHL-89

File: xp

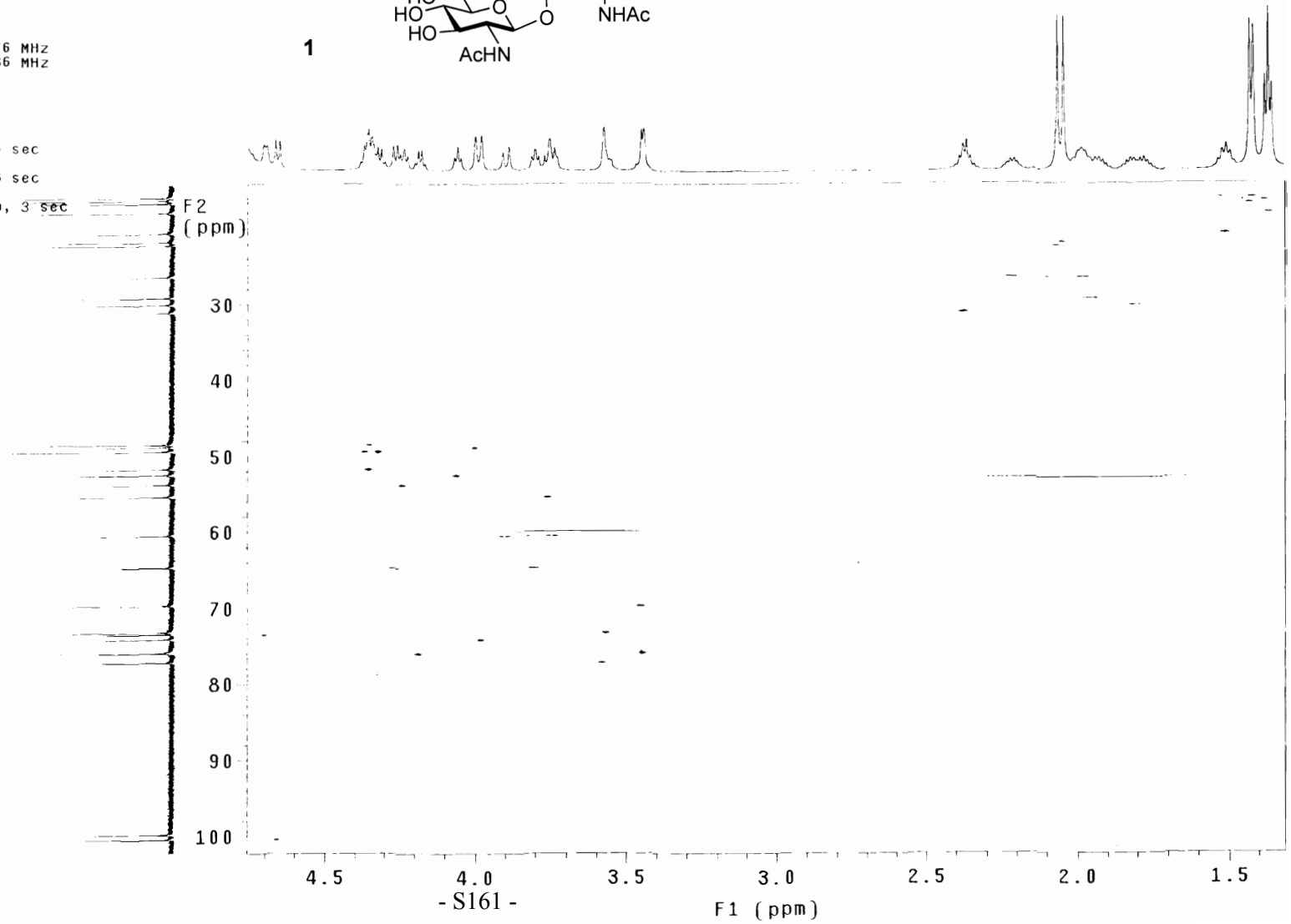
Pulse Sequence: HETCOR

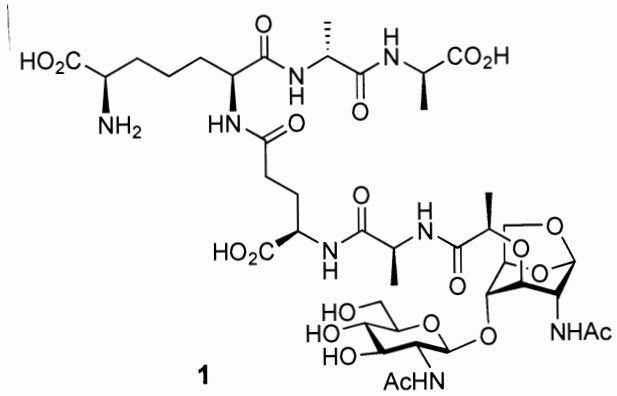
Solvent: d2o
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 1.313 sec
Acq. time 0.187 sec
Width 21929.8 Hz
2D Width 4799.0 Hz
36 repetitions
2 x 512 increments
OBSERVE C13, 150.8385376 MHz
DECOUPLE H1, 599.8770986 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 0.086 sec
F1 DATA PROCESSING
Gauss apodization 0.098 sec
FT size 8192 x 4096
Total time 16 hr, 11 min, 3 sec



1





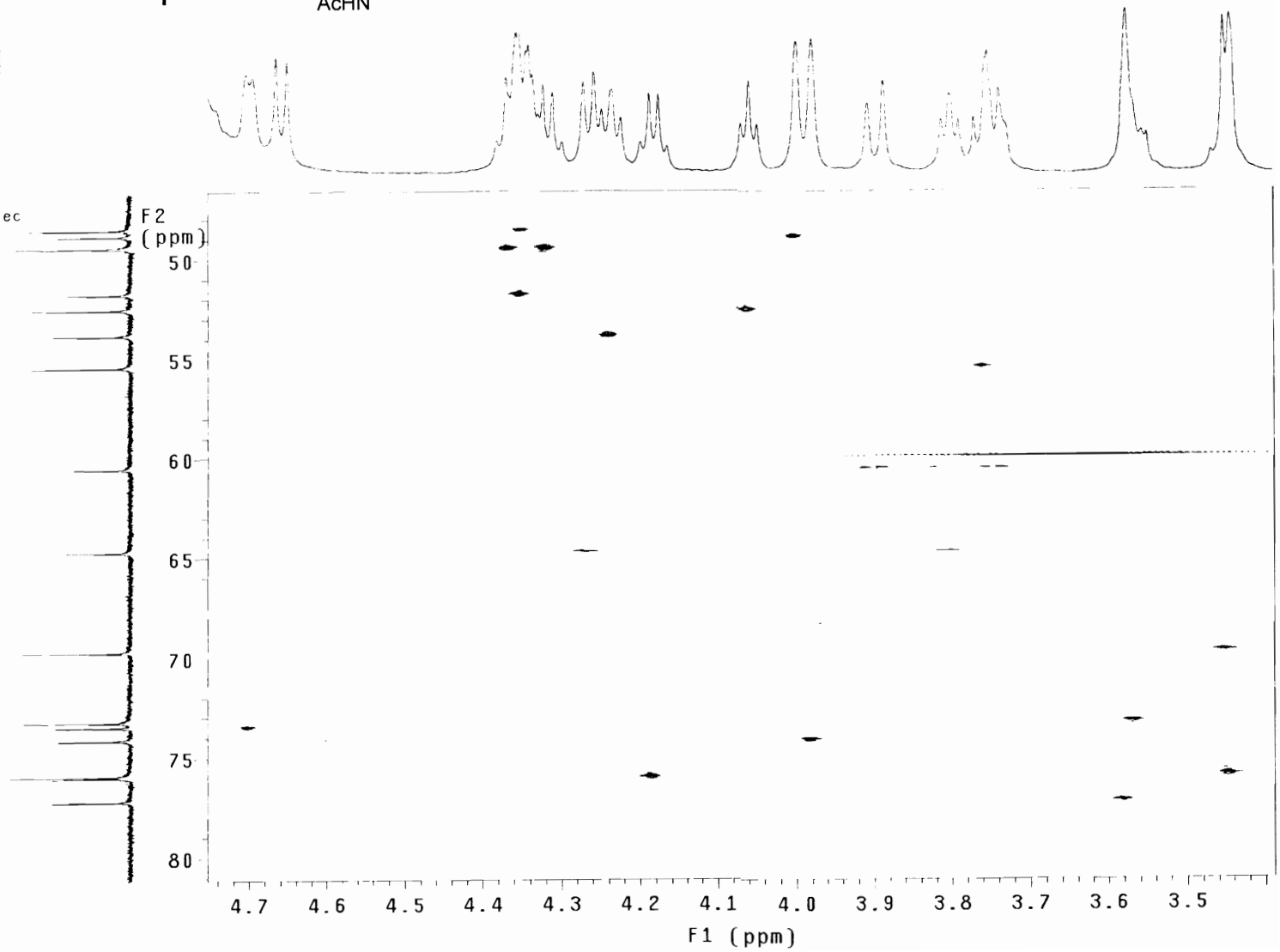
1

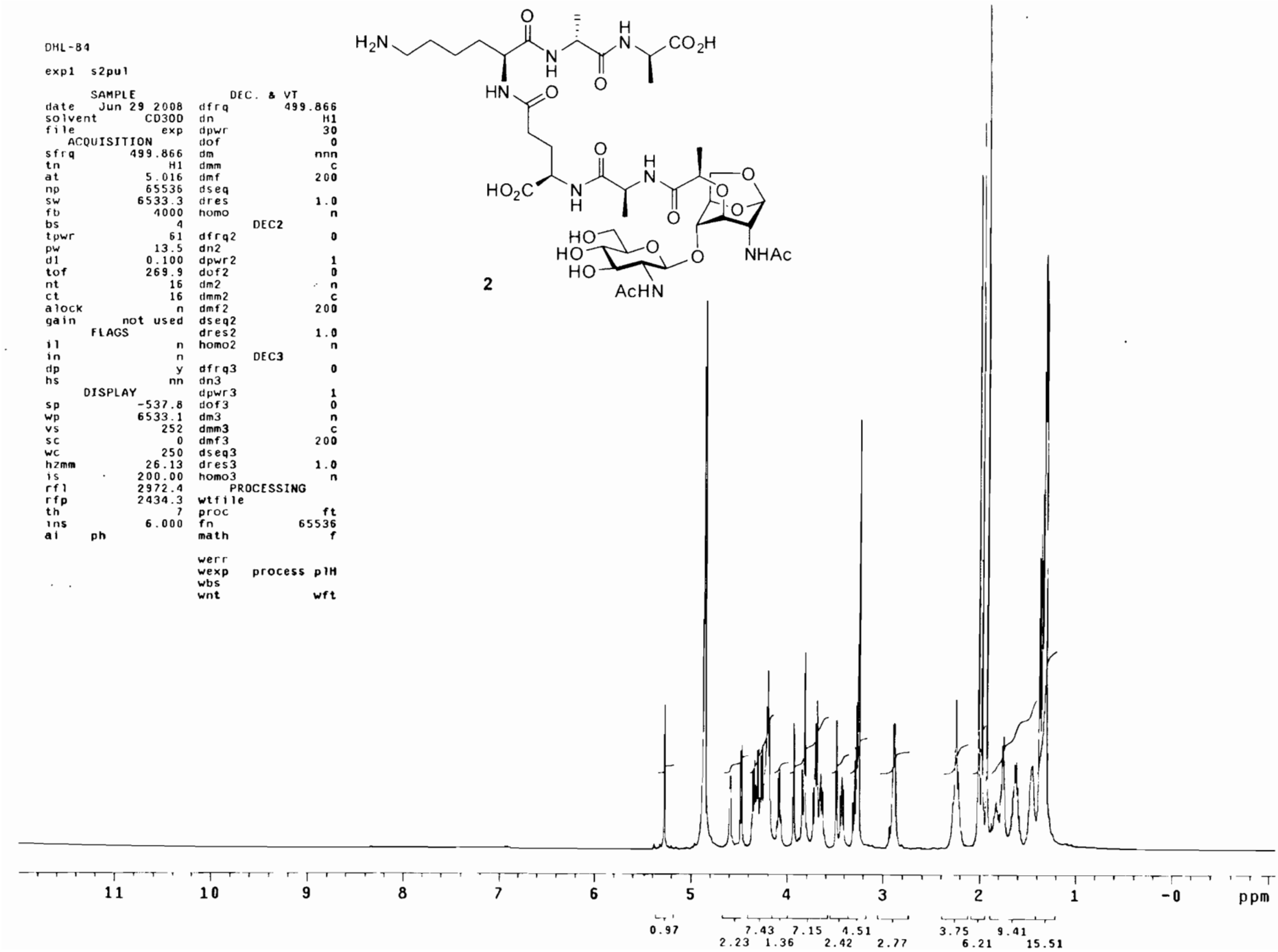
```
DHL-89

File: xp

Pulse Sequence: HETCOR
 Solvent: d2o
 Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600  "nmr600"

 Relax. delay 1.313 sec
 Acq. time 0.187 sec
 Width    21929.8 Hz
 2D Width 4799.0 Hz
 36 repetitions
 2 x 512 increments
OBSERVE  C13, 150.8385376 MHz
DECOUPLE  H1, 599.8770986 MHz
 Power 44 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Gauss apodization 0.086 sec
F1 DATA PROCESSING
 Gauss apodization 0.098 sec
FT size 8192 x 4096
Total time 16 hr, 11 min, 3 sec
```



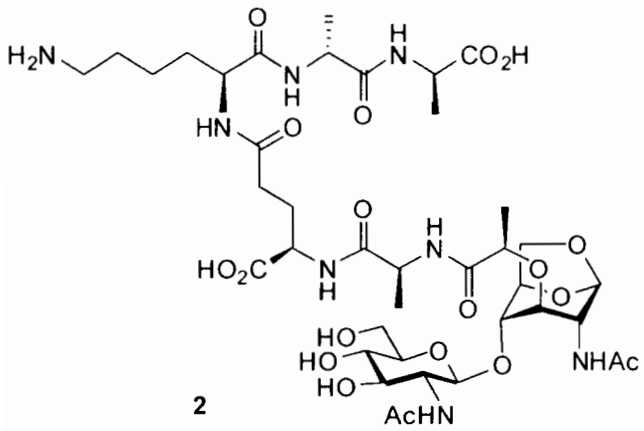

DHL-84
exp1 s2pul
SAMPLE
date Jun 29 2008
solvent CD3OD
file exp
ACQUISITION
sfrq 499.866
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -537.8
wp 6533.1
vs 252
sc 0
wc 250
hzmm 26.13
is 200.00
rfl 2972.4
rfp 2434.3
th 7
ins 6.000
ai ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
2
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.97
2.23
1.36
7.43
7.15
2.42
4.51
2.77
3.75
6.21
9.41
15.51

DHL-84

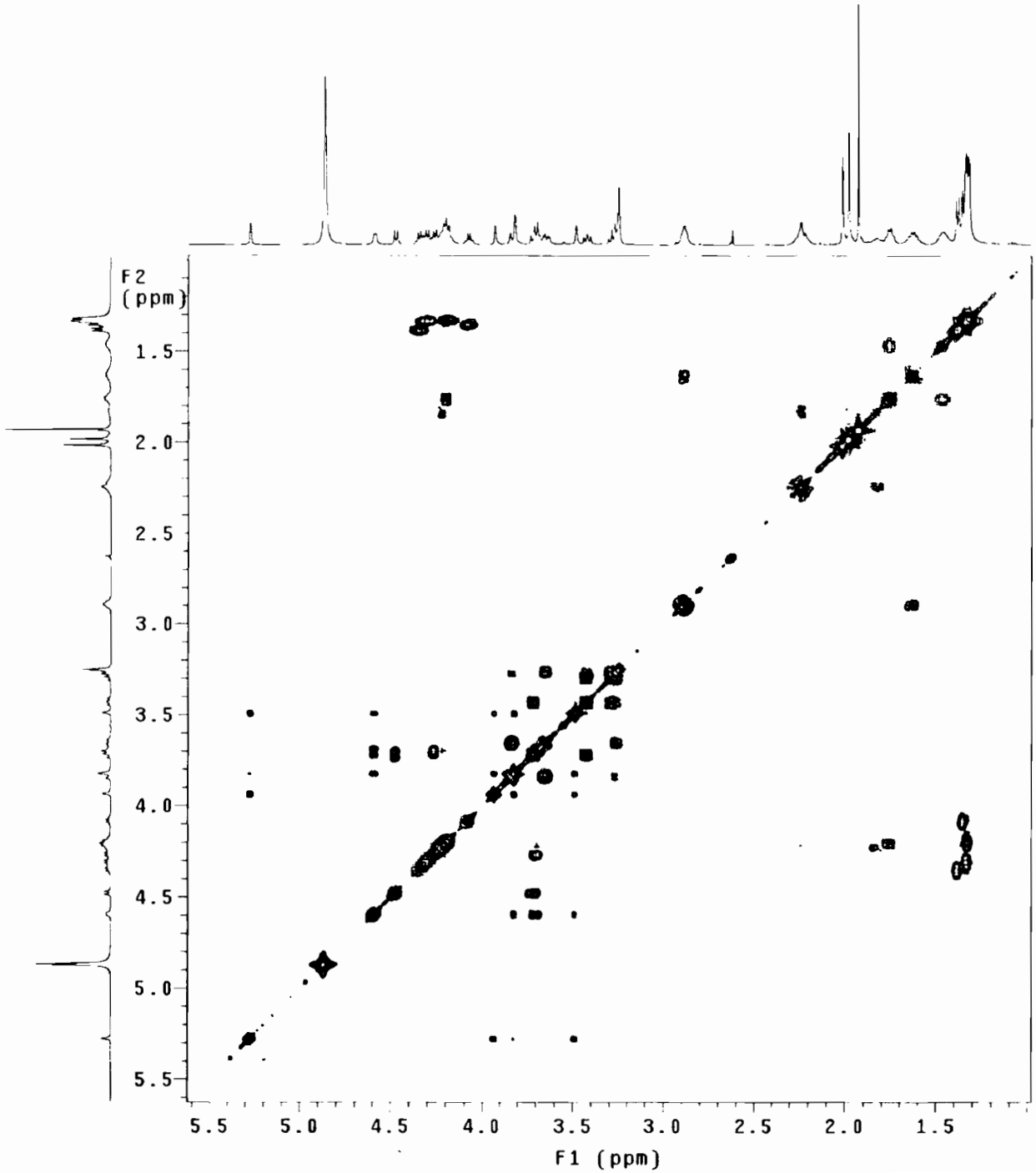
Pulse Sequence: relayh

Solvent: CD3OD
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.220 sec
Width 2326.5 Hz
2D Width 2326.5 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8631721 MHz
DATA PROCESSING
Sine bell 0.110 sec
F1 DATA PROCESSING
Sine bell 0.055 sec
FT size 1024 x 1024
Total time 3 hr, 44 min, 9 sec



2

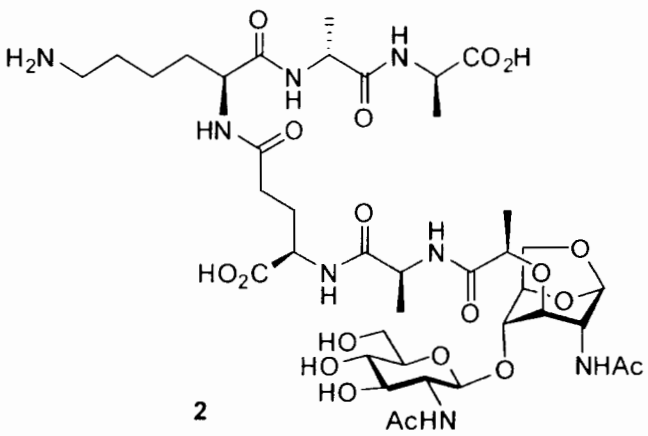


DHL-84

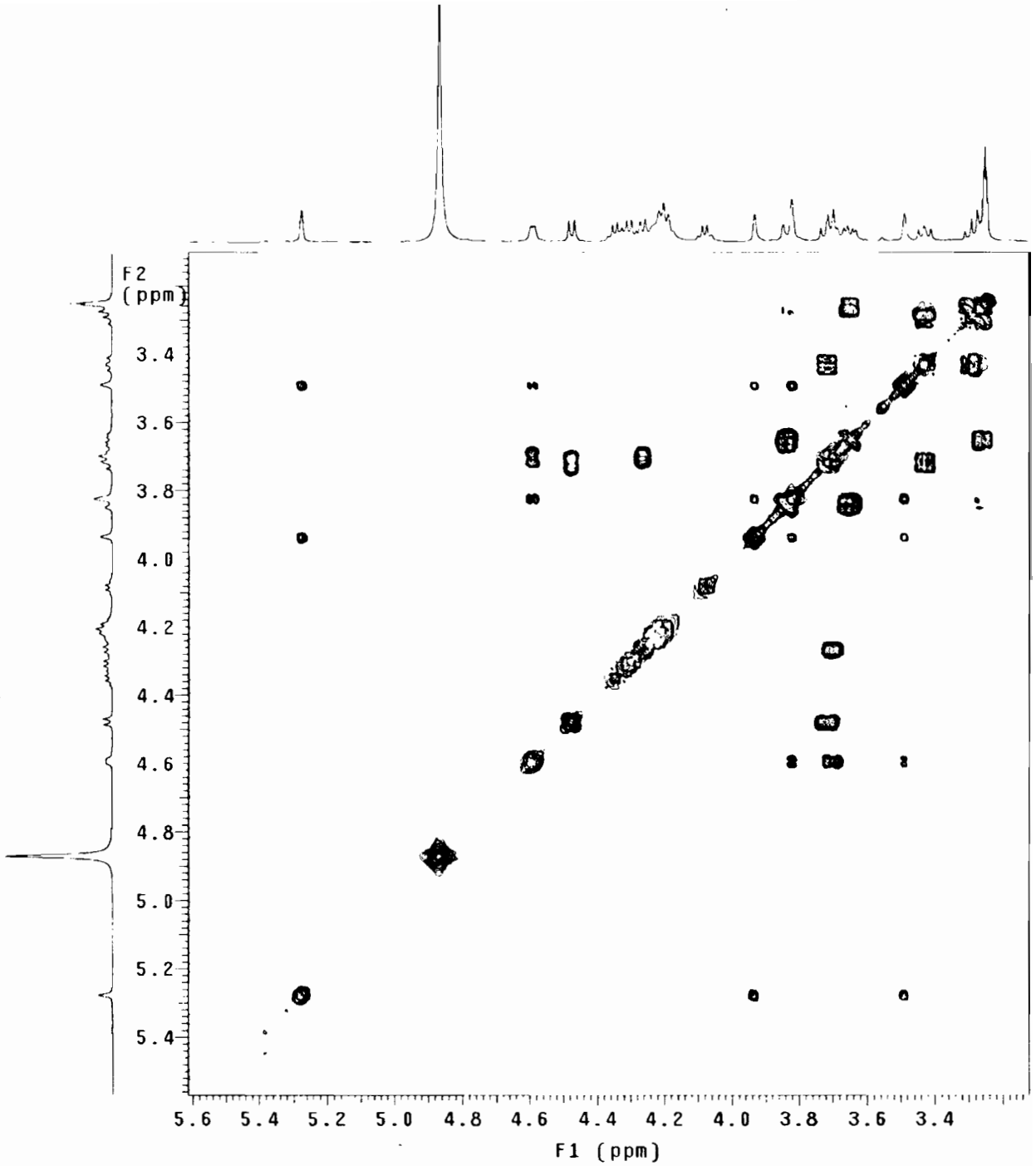
Pulse Sequence: relayh

Solvent: CD3OD
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.220 sec
Width 2326.5 Hz
2D Width 2326.5 Hz
16 repetitions
512 increments
OBSERVE H1, 499.8631721 MHz
DATA PROCESSING
Sine bell 0.110 sec
F1 DATA PROCESSING
Sine bell 0.055 sec
FT size 1024 x 1024
Total time 3 hr, 44 min, 9 sec



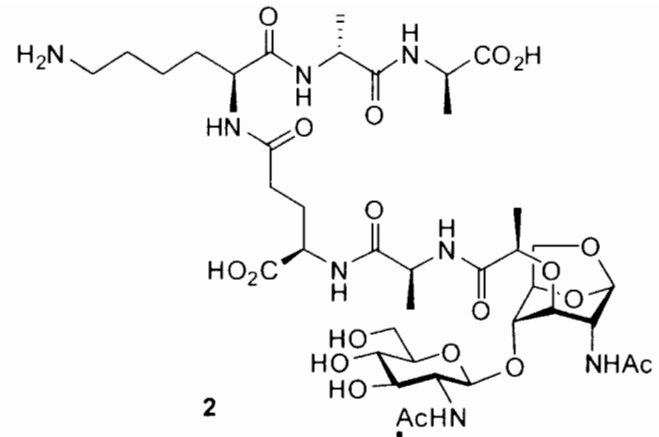
2



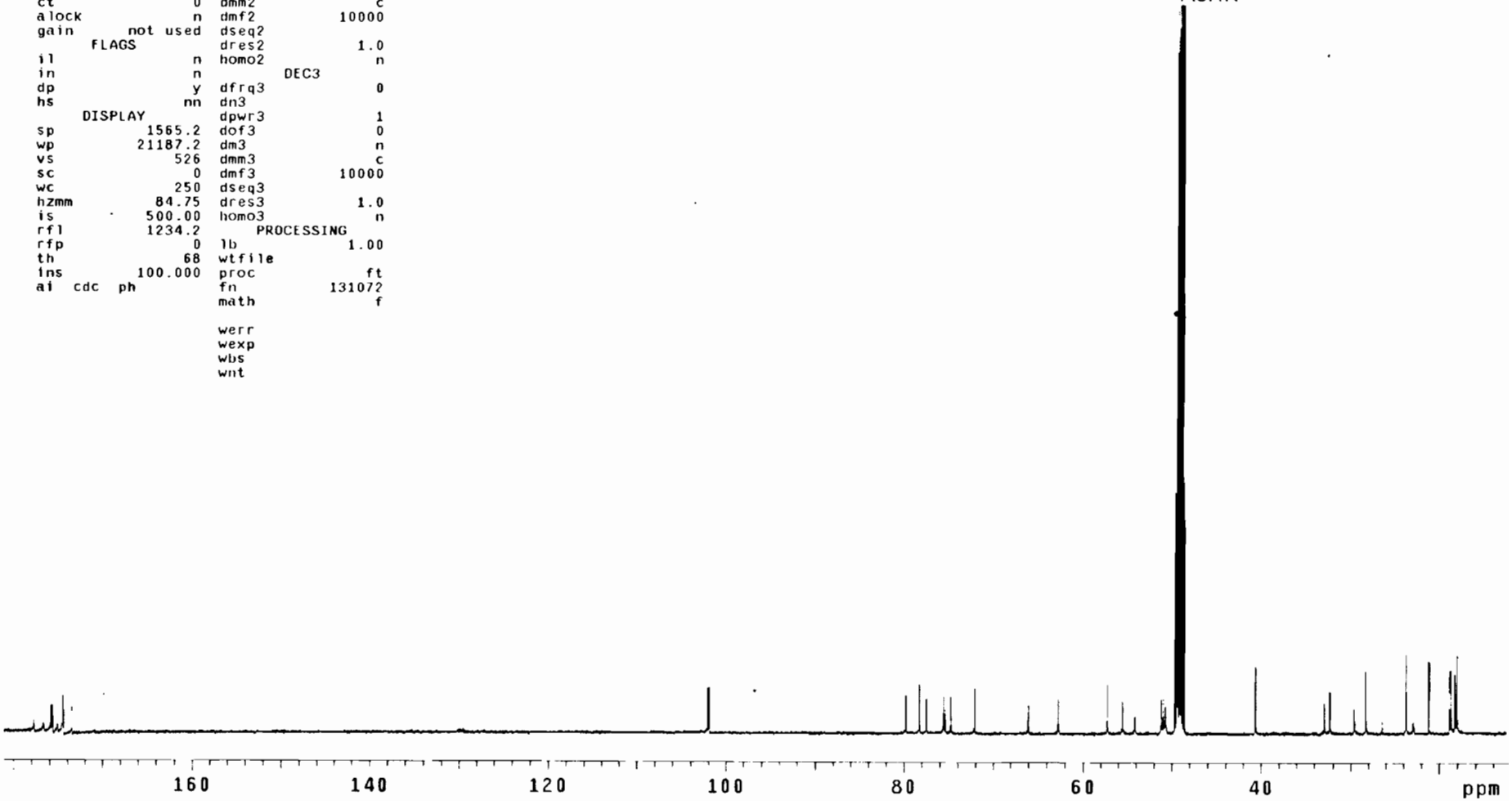
DHL-84

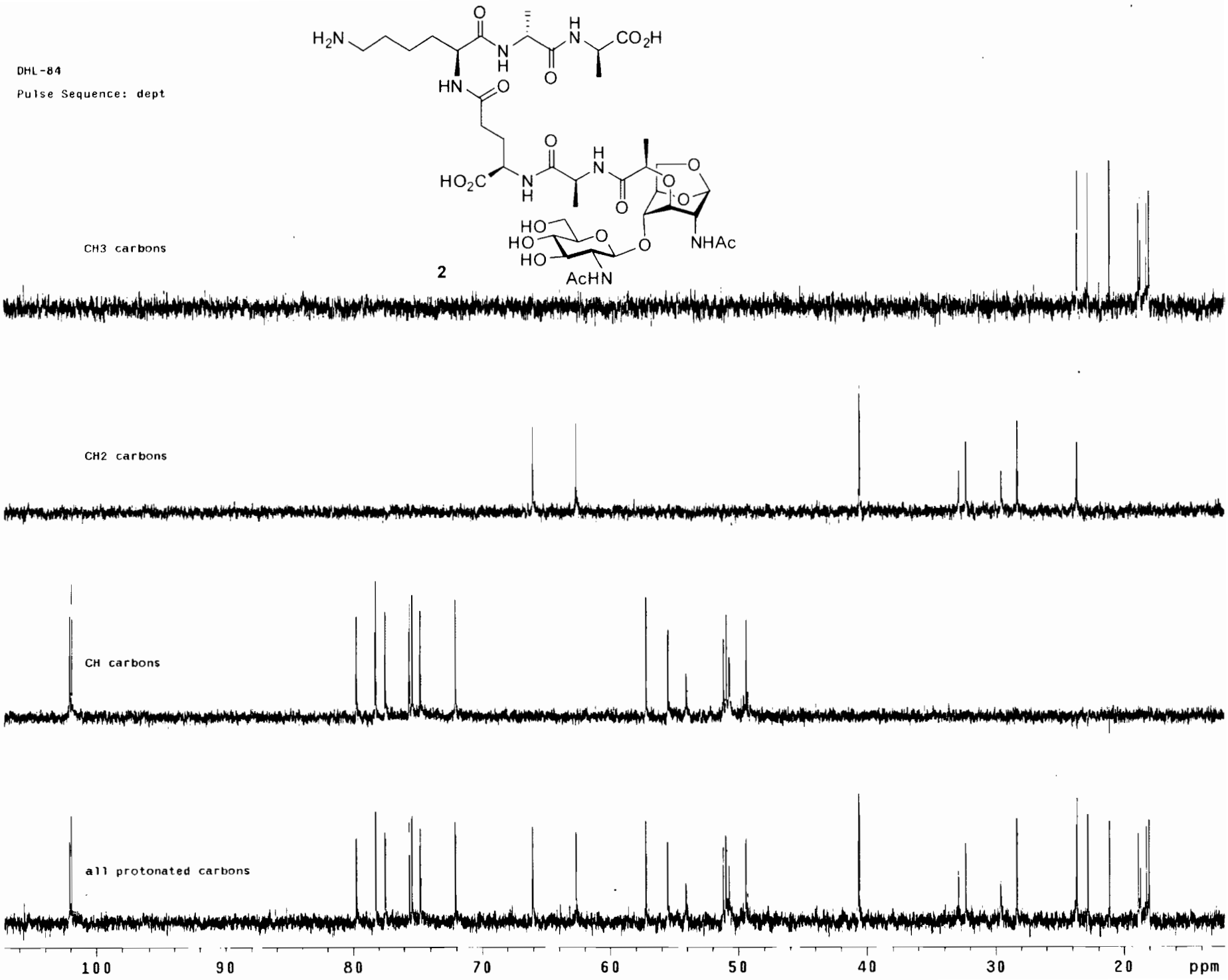
exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 29 2008	dfrq	499.866
solvent	cd3od	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	7000	dm2	n
ct	0	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1565.2	dof3	0
wp	21187.2	dm3	n
vs	526	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	84.75	dres3	1.0
is	500.00	homo3	n
rfl	1234.2	PROCESSING	
rfp	0	lb	1.00
th	68	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



2



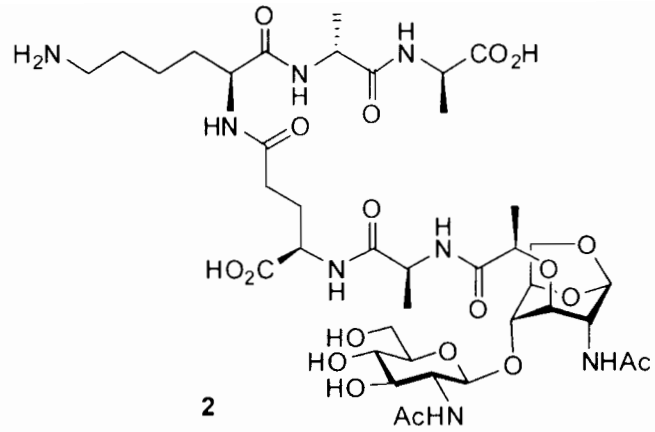

DHL-84
Pulse Sequence: dept
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
2
100
90
80
70
60
50
40
30
20
ppm

DHL-84

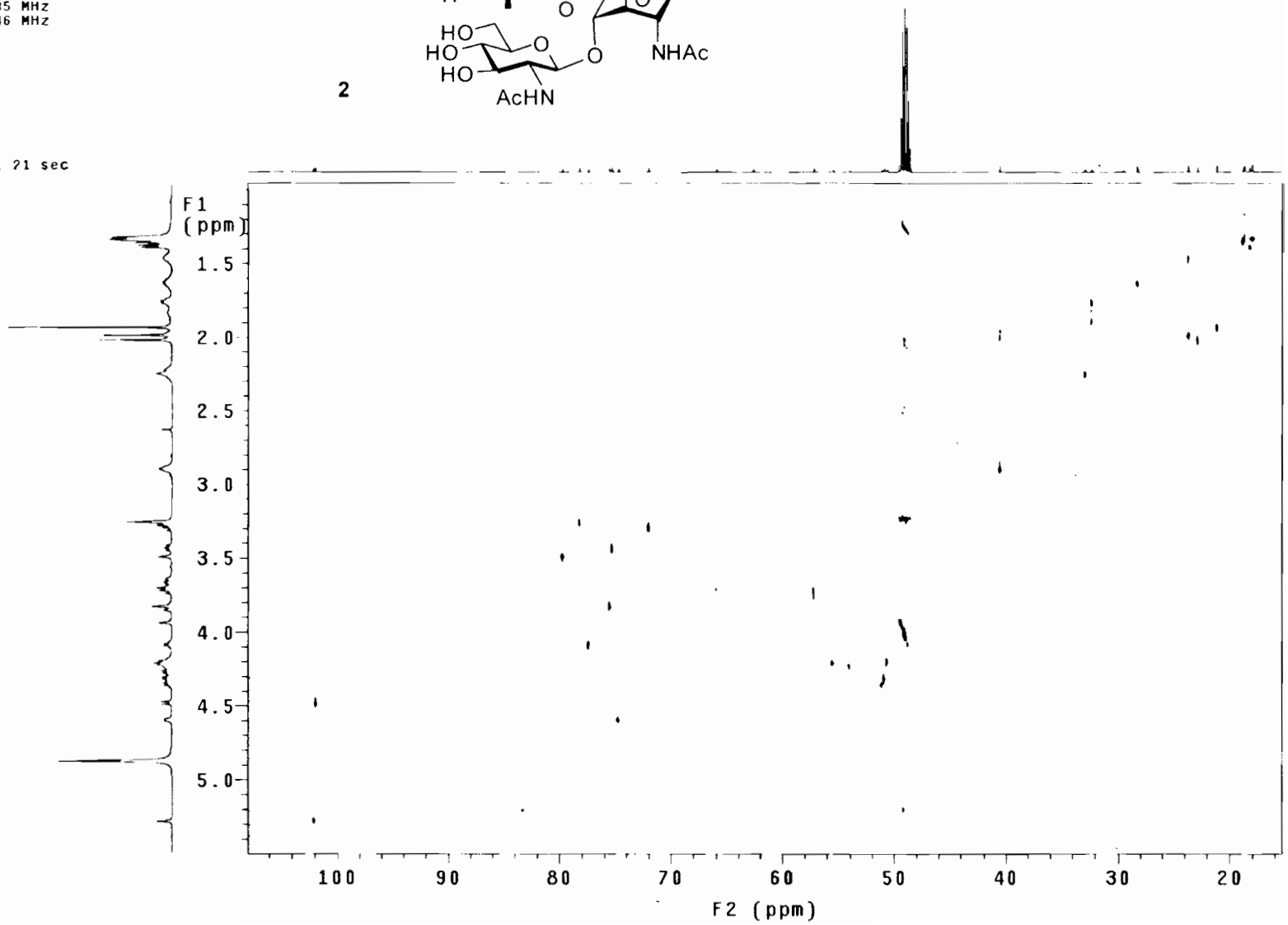
Pulse Sequence: hetcor

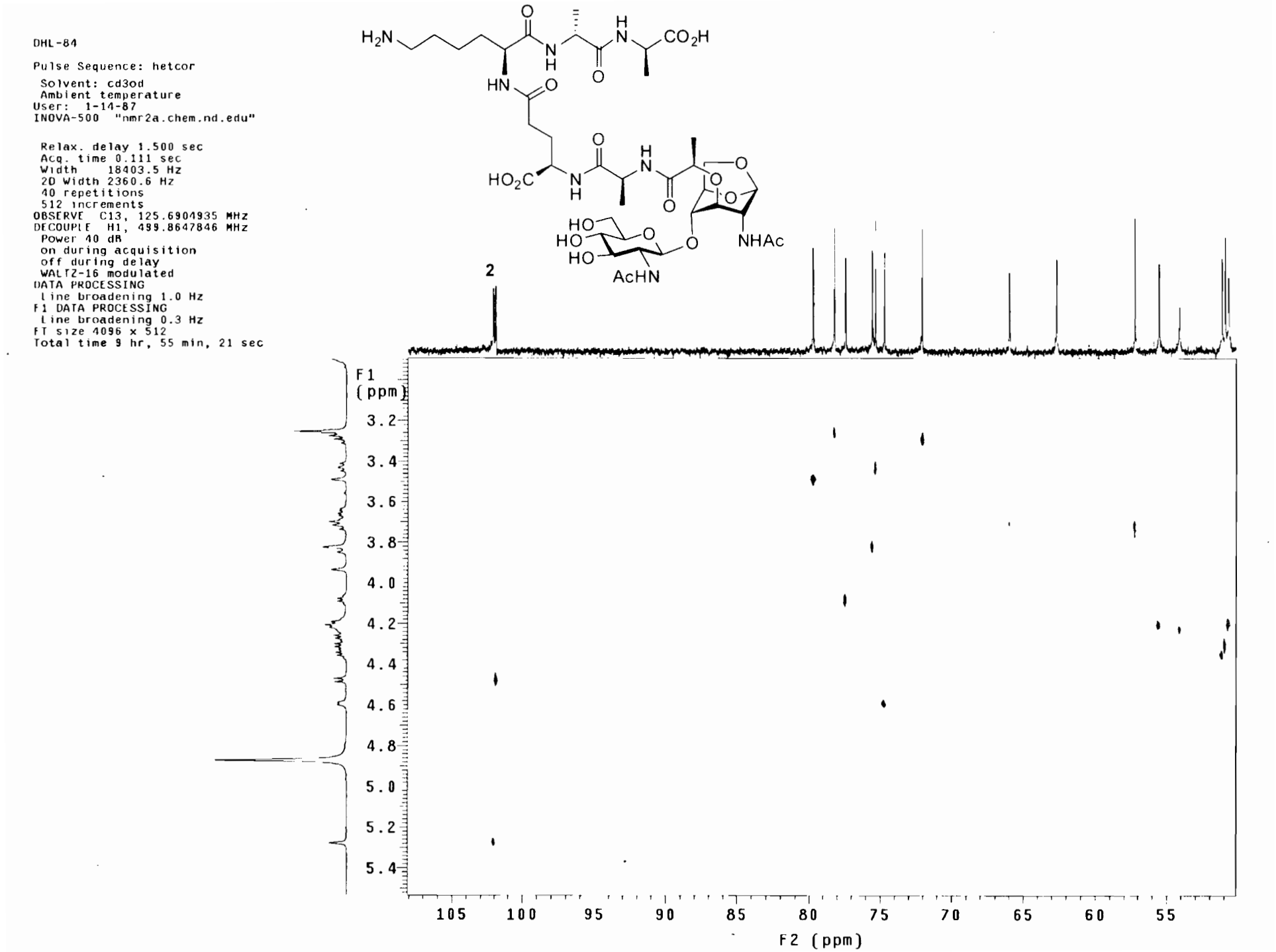
Solvent: cd3od
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2360.6 Hz
40 repetitions
512 increments
OBSERVE C13, 125.6904935 MHz
DECOUPLE H1, 499.8647846 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 9 hr, 55 min, 21 sec



2




DHL-84
Pulse Sequence: hetcor
Solvent: cd3od
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2360.6 Hz
40 repetitions
512 increments
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Power 40 dB
on during acquisition
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WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 9 hr, 55 min, 21 sec
2
NHAc
AcHN
F1 (ppm)
3.2
3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
105
100
95
90
85
80
75
70
65
60
55
F2 (ppm)