Supporting Information

Inhibitors for Bacterial Cell Wall Recycling

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Contents

General Information

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) plates that were visualized using UV light and cerium sulfate or phosphomolybdic acid 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. IR spectra were obtained from KBr plates using a Perkin-Elmer Paragon 1000 FT-IR instrument. 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 spectrometer. Proton and Carbon chemical shifts were referenced to residual solvent peaks. NMR signal assignments for synthesized compounds were made 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 by FAB ionization, using a JEOL AX505HA mass spectrometer or by ESI ionization, using a BRUKER microTOF II mass spectrometer.

Crystals were examined under Infineum V8512 oil and placed on a MiTeGen mount, then transferred to the 296 K N_2 stream of a Bruker SMART Apex 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 four-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 submitted as Supporting Information.

Experimental Procedures

Cloning of the *nagZ* gene from *Pseudomonas aeruginosa* PAO1. Genomic DNA from *P. aeruginosa* PAO1 was used as the template for cloning. The PCR was accomplished using the following primers 5'-GATATACATATGCAAGGCTCTCTGATGCTC-3' (*Nde*I cut site underlined) and 5'-GATATAGGATCCTCAATCAATCAGTTGCGCAG-3' (*Bam*HI cut site underlined). The conditions used for the PCR were as follows: 30 cycles of denaturation at 94 °C for 40 s, followed by annealing of primers for 40 s at 58 °C, and extension for 3 min at 72 °C using pfu DNA polymerase. The reaction volume was 50 μL and contained 0.2 mM DNTPs, 0.5 μM of each primer, 50 ng of template DNA, and 1 μL of pfu DNA polymerase. The PCR reaction mixture was subjected to electrophoresis through a 1.5% agarose gel for 30 min at 100 V, and the product was excised and purified using a gel extraction kit (Qiagen). Double-digest reaction mixtures using both restriction endonucleases (*Nde*I and *Bam*HI) were carried out on both the PCR product and pET28a vector. The reaction volumes were 50 μL and consisted of approximately 5 μg of DNA and 1 U of each restriction enzyme (New England Biolabs) and were allowed to proceed for 3 h at 37 °C. The reaction mixtures were then subjected to electrophoresis through a 1.5% agarose gel, and the desired DNA was gel purified, as described above. A total of 0.5 μL of the digested plasmid DNA was then combined with 22 μL of the digested PCR product and ligated together using T4 DNA Ligase (New England Biolabs) for 1 h at 25 °C. A 14-μL portion of this ligation mixture were used to transform *E. coli* ultracompetent cells as described above, except plates containing 50 μg/mL of kanamycin were used. A colony was picked and cultured, and the plasmid DNA was harvested the following day, as described above. The entire insert was sequenced to ensure accuracy.

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

Protein expression in *E. coli* and purification of NagZ. DNA encoding NagZ in the pET28a expression vector was used to transform *E. coli* BL21 star (DE3) cells (Invitrogen). A colony was selected and cultured overnight in LB media containing 50 μg/mL of kanamycin. This culture was used to inoculate a 1 L culture that was grown to an OD_{600} of ~0.8 at 37 °C. At this point, protein expression was induced using 1 mM IPTG (isopropyl-β-D-1-thiogalactopyranoside, Fisher Scientific) for 2.5 h at 25 °C. Postinduction cells were harvested by centrifugation (Eppendorf 5810R) for 20 min at 3200 *g* and resuspended in 20 mL of nickel-column binding buffer (50 mM Na_2HPO_4, 0.5 M NaCl, 5 mM imidazole; pH 7.4). To the resuspended cells was added 1 mM PMSF and the cells were lysed by sonification. The cell debris was removed by centrifugation at 18500 *g* for 75 min, and the supernatant was loaded onto a 5 mL HisTrap FF column (GE Healthcare). The column was washed with 100 mL of washing buffer (50 mM Na_2HPO_4, 0.5 M NaCl, 60 mM imidazole; pH 7.4) and the protein was eluted with 30 mL of elution buffer (50 mM Na_2HPO_4, 0.5 M NaCl, 250 mM imidazole; pH 7.4). The purified enzyme was subsequently dialyzed overnight against PBS buffer (pH 7.4) containing 10% (w/v) glycerol, the enzyme was stored at 4 °C. The protein content from the column fractions was monitored by SDS-PAGE (Figure S1). The NagZ concentration was determined by measuring the absorbance of the solution at 280 nm and using a calculated extinction coefficient of 32 220 $M^{-1}cm^{-1}$.[3] Matrix-assisted laser desorption ionization (MALDI) mass spectrometric analysis revealed a molecular mass of 38 171 Da for NagZ, in agreement with the value deduced from the gene sequence (38 263 Da) (Figure S3A). The molecular mass of 37 594 Da for the NagZ from *E. coli* was not observed in the preparation.

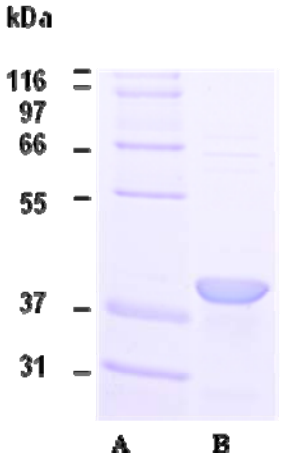


Figure S1. 12% SDS-PAGE analysis of the purified NagZ. (A) Mark12™ Molecular Weight Marker; (B) NagZ (5 μg) after dialysis.

Expression and purification of MltB. The gene encoding the lytic transglycosylase MltB (amino acids 21–361) was amplified from *Escherichia coli* K12 substrain MG1655 chromosome using high-fidelity *Pfu*Ultra II Fusion HS DNA Polymerase (Stratagene®) and the following oligonucleotide primers: mltB_fw_NdeI, 5'-AGATATACATATGAAGCCAAAACCTACTG-3', and mltB_rv_XhoI, 5'- ATCTCGAGCTGTACTCGCGCCAG-3'. The PCR product was cloned into pET-24a(+) vector from Novagene, to give a gene that codes for MltB (amino acids 21-361, S21M) with a C-terminal His6x tag preceded by two additional amino acids (LE). The protein (349 amino acids, 39,041 Da) was expressed in *E. coli* BL21 (DE3). Cells containing the plasmid were selected on agar supplemented with 50 μg/mL kanamycin. The transformants were inoculated overnight in 5 mL of Luria-Bertaini (LB) medium with 50 μg/mL kanamycin. This culture was used to inoculate 500 mL of LB medium supplemented with 50 μg/mL kanamycin in a 3-L Erlenmeyer flask, which was grown at 37 ºC, 120 rpm. Protein expression was induced at an OD_{600} of 0.8, with 0.4 mM IPTG and incubation was continued at 15 ºC for 12 h to minimize formation of inclusion bodies. Cells were harvested by centrifugation for 30 min at 3220 *g*, 4 °C, and the cell pellet was resuspended in 10 mL of 20 mM HEPES buffer, pH 7.0, supplemented with 0.5 M NaCl, 10% glycerol, 25 mM imidazole and 0.1% Brij-35. The protein was released from the cells by sonification on ice (10 cycles of 2 min sonification each, with 1 min rest in between sonification cycles, using a Branson 450 Sonifier). The cell extract was then centrifuged for 45 min at 18514 *g* at 4 °C. The supernatant was loaded onto a 5-mL HiTrap Chelating HP column (GE Healthcare). The

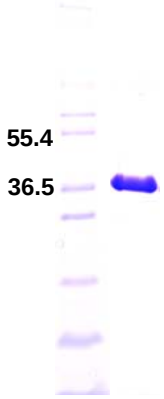


Figure S2. 12% SDS-PAGE gel showing purified MltB. Lane 1: Mark12™ Molecular Weight Marker; lane 2: MltB (10 μg) after dialysis.

[3] Généreux, C.; Dehareng, D.; Devreese, B.; Van Beeumen, J.; Frère, J. M.; Joris, B., *Biochem. J.* **2004,** *377*, 111-120.

column was washed with 10 mL of 20 mM HEPES buffer, pH 7.0, 0.5 M NaCl, 10% glycerol, 25 mM imidazole and 0.1% Brij-35. Elution was performed using a gradient from 0-100% of 20 mM HEPES buffer, pH 7.0, 0.5 M NaCl, 10% glycerol, 500 mM imidazole and 0.1% Brij-35. After dialysis against 50 mM HEPES buffer, pH 7.6, 0.20 M NaCl, and Brij-35, the protein concentration was determined from the absorbance at 280 nm, using the theoretical extinction coefficient at 280 nm (67 840 M^{-1} cm^{-1}). The protein content from the column fractions was monitored by SDS-PAGE (Figure S2). MALDI mass spectrometric analysis revealed a molecular mass of 39 263 Da for MltB, in agreement with the value deduced from the gene sequence (39 041 Da) (Figure S3B).

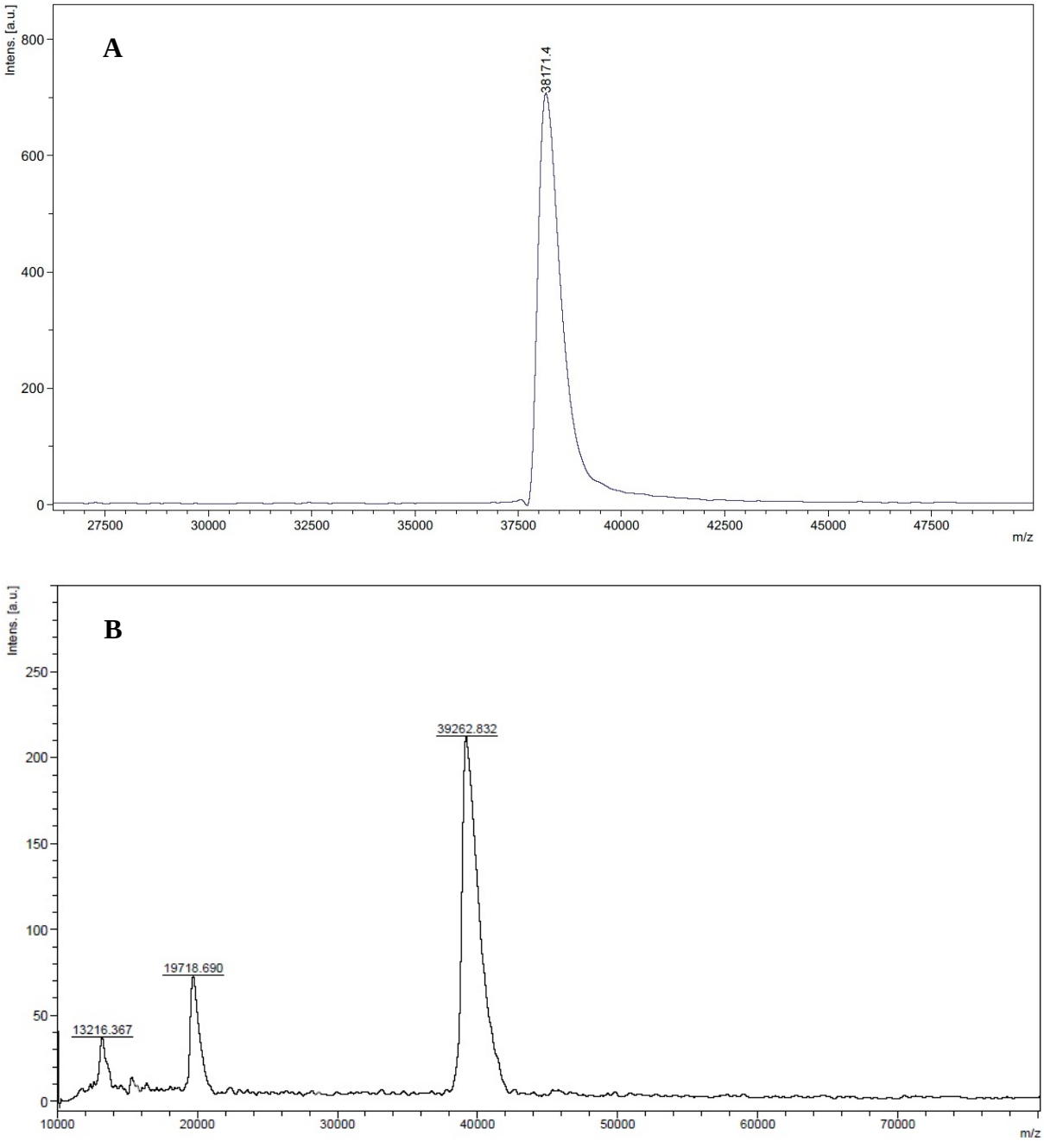


Figure S3. MALDI mass spectra of the purified NagZ (A) and of the purified MltB (B).

NagZ Inhibition Kinetics. Experiments to assess inhibition of NagZ by **3**, **4**, **5** and **6** were performed using 4-nitrophenyl 2-acetamido-2-deoxy-*β*-D-glucopyranoside (PNP-GlcNAc) as substrate, which was purchased from Sigma. Enzyme activity was measured by spectrophotometric monitoring of the release of 4-nitrophenolate ion at 400 nm. Standard reaction of NagZ (500 µL) was performed in PBS buffer pH 7.4 contained 40 nM enzyme and 0.1-1 mM substrate (Figure S4). The assays were performed in triplicate at 25 °C for 4 min.

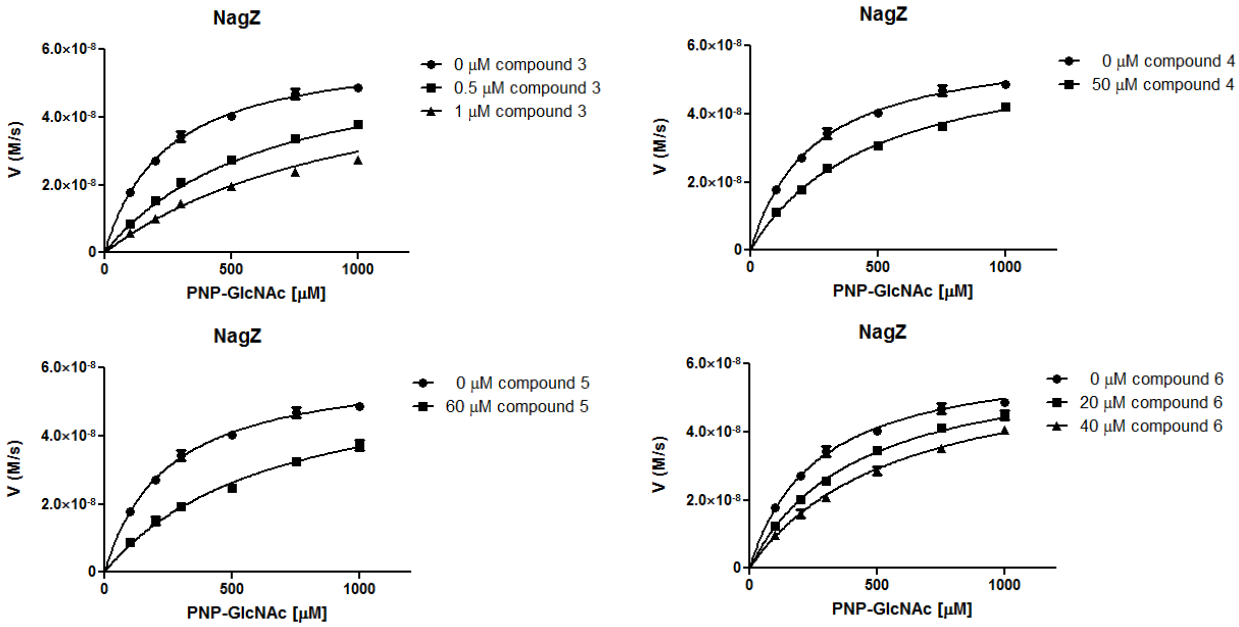
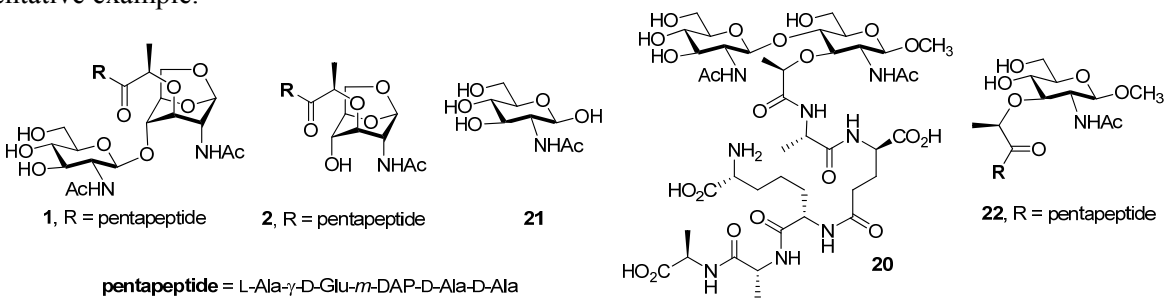


Figure S4. Inhibition kinetics of NagZ by compounds **3**, **4**, **5** and **6**.

Kinetics of Substrate Turnover by NagZ. The kinetics studies of NagZ and GlcNAc-anhMurNAc-pentapeptide (**1**) were performed by HPLC (Figure S5). The assays were carried out in PBS buffer (pH 7.4) at 25 °C with substrate concentrations ranging from 0.15 mM to 0.9 mM and 40 nM enzyme concentration. The reaction mixtures were incubated at 25 °C for 35 min. The reactions were stopped by the addition of two volumes of 0.075% TFA in water. The internal standard was *N*α,*N*ε-Diacetyl-L-Lys-D-Ala-D-Ala. Reaction products were separated and quantified on a C18 reversed-phase HPLC column (Sunfire C18, 3.5 µm, 4.6 × 150 mm; Waters) on a PerkinElmer series 200 System. The column was equilibrated with 0.05% trifluroacetic acid in water and compounds were 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 NagZ was quantified from the rate of product appearance. The kinetics studies of NagZ and GlcNAc-MurNAc-pentapeptide (**20**) were performed by HPLC using the same protocol (Figure S5). The t_R for **1** was 32 min, for **2** was 31 min, for **20** was 30.5 min and for **22** was 27 min. HPLC chromatograms of the NagZ reaction of **1** are shown in Figure S6 as a representative example.



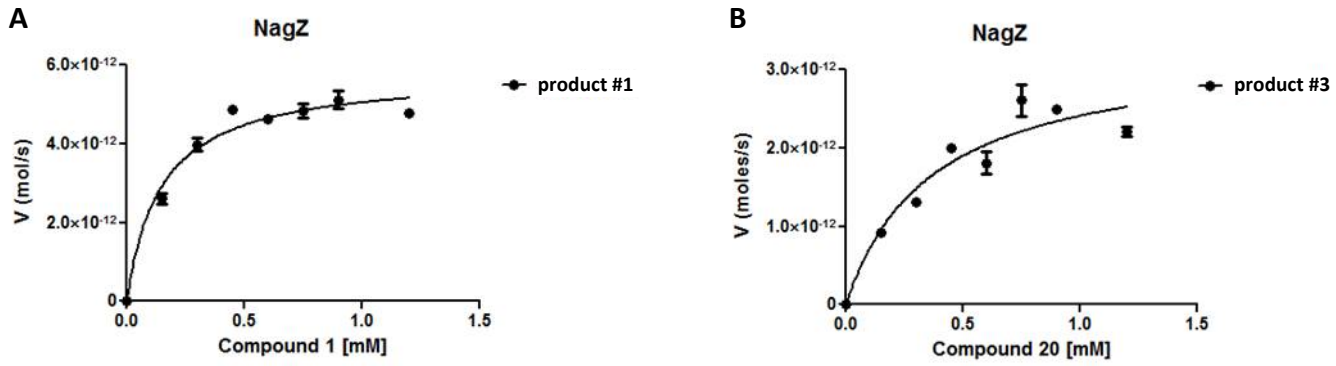


Figure S5. Kinetics studies of NagZ with GlcNAc-anhMurNAc-pentapeptide (**1**, **A**) and GlcNAc-MurNAc-pentapeptide (**20**, **B**)

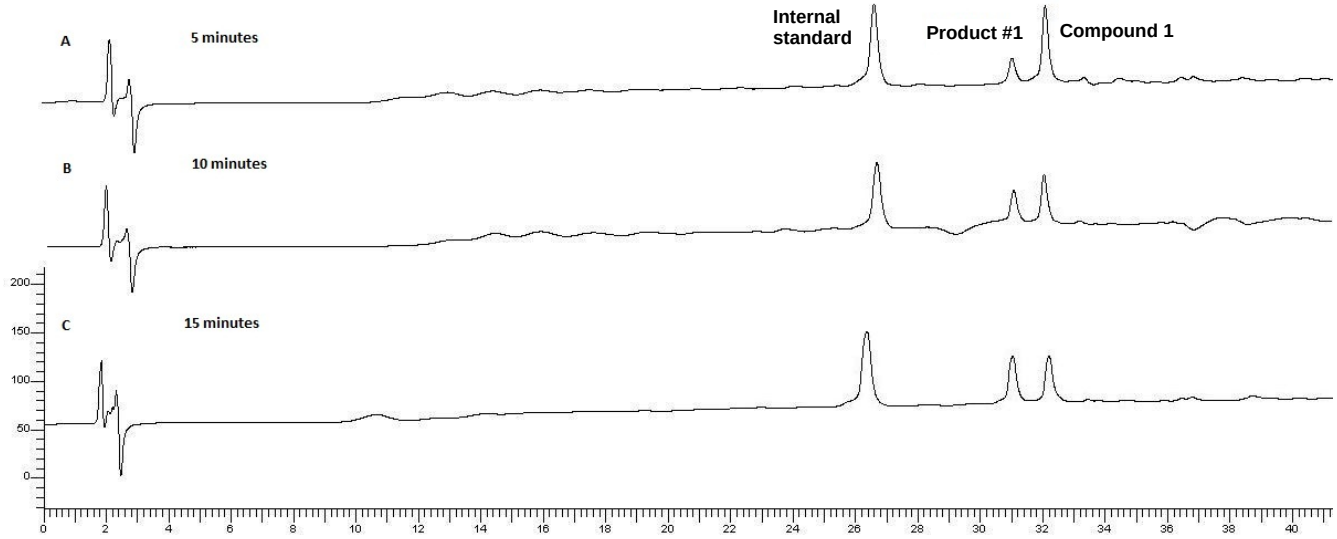


Figure S6. The NagZ reaction with compound **1**. The time course for conversion of compound **1** (retention time at 32 min) by NagZ to the product #1 (anhMurNAc pentapeptide, retention times at 31 min) were monitored at 5 min (A), 10 min (B), and 15 min (C) of incubation. The identity of product #1 was confirmed by LC/MS analysis. The second product #2 (GlcNAc) of NagZ reaction is not detectable at 205 nm.

The K_i, k_{cat} and K_m values were determined using a nonlinear regression with GraphPad Prism version 5.03 for Windows, GraphPad Software, San Diego California USA, www.graphpad.com.

ESI-MS. Characterization of the reaction products was performed using a Dionex Ultimate 3000 RSLC and Bruker MicrOTOF-QII electrospray ionization (ESI) mass spectrometer (Figure S7). The peaks were initially analyzed using positive ionization mode throughout the *m*/*z* of 100 – 1200. The charge states of the major ions were determined as the reciprocal of the spacing between two adjacent isotopic peaks differing in mass by 1 Da.[4] Analysis of the MS data and fragmentation pattern of the reaction products and of authentic compounds **2**, **21**, and **22** allowed us to confirm chemical structure of the reaction products (Figures S7 and S8).

[4] Henry, K. D.; McLafferty, F. W. *Org. Mass Spectrom.* **1990,** *25*, 490-492.

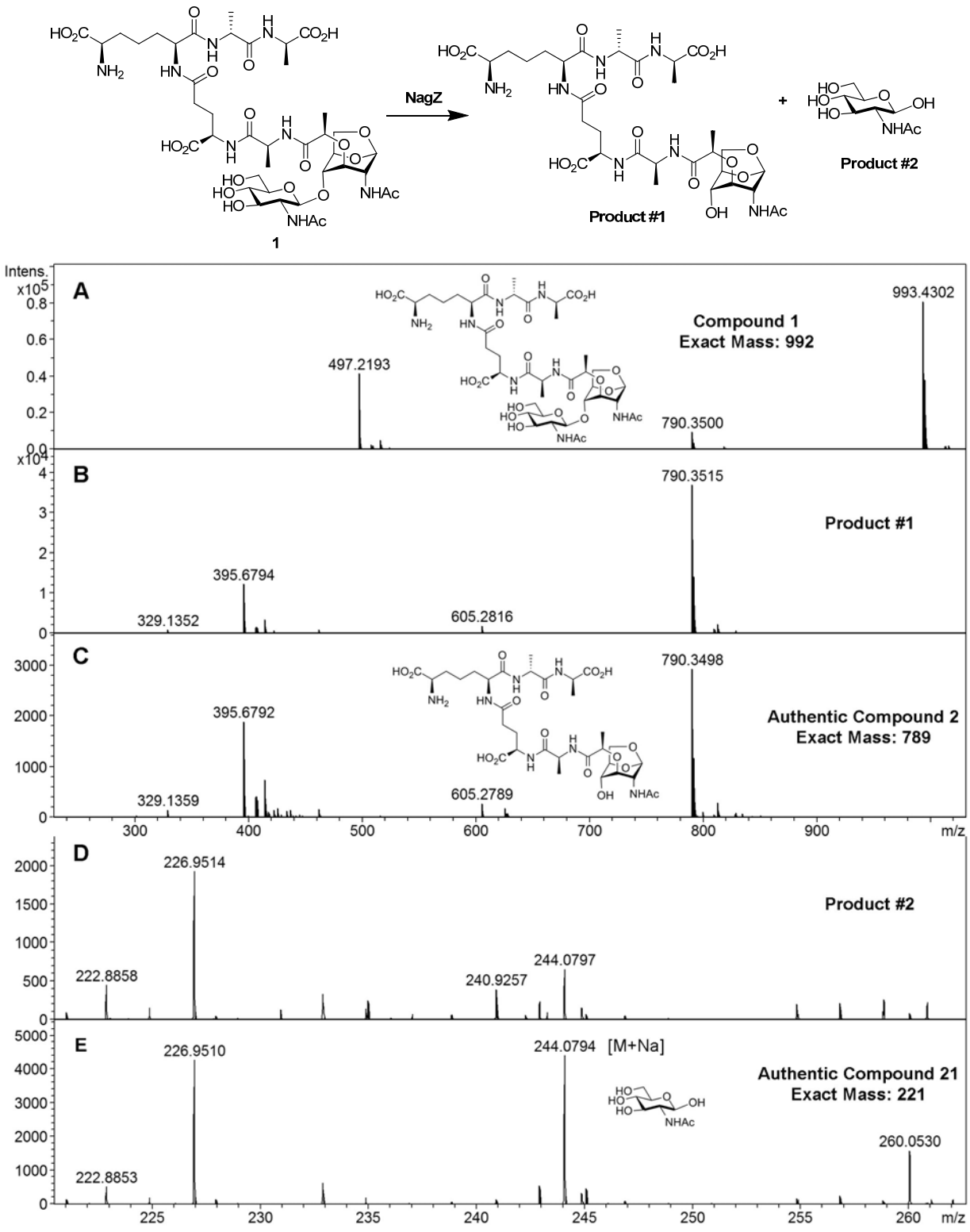


Figure S7. ESI-LC-MS analysis of the NagZ reaction products and comparison to authentic synthetic samples. The following spectra are shown: synthetic compound **1** (A); reaction product #1 (B); authentic compound **2** (C); reaction product #2 (D); and authentic compound **21** (E).

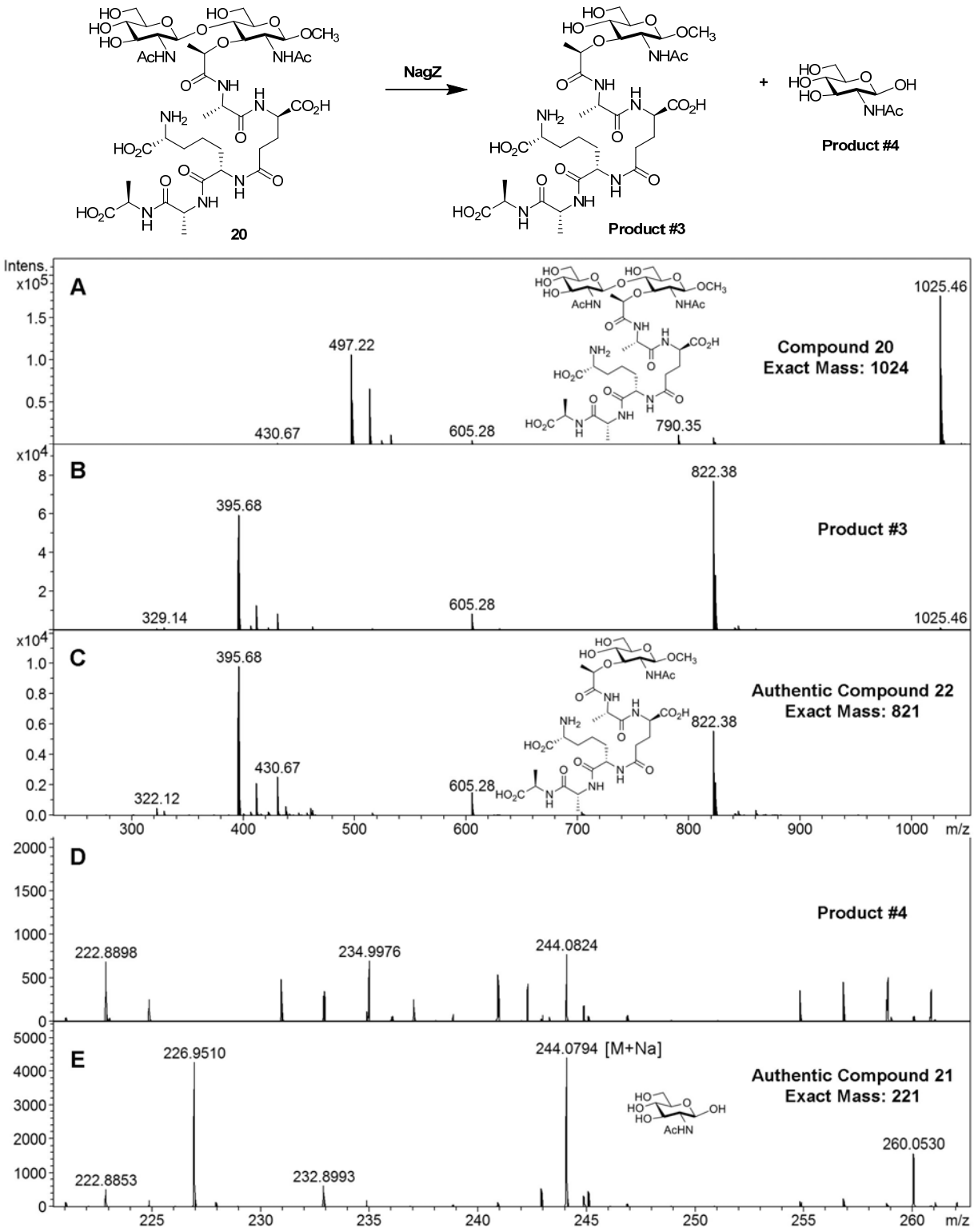


Figure S8. ESI-LC-MS analysis of the NagZ reaction products and comparison to authentic synthetic samples. The following spectra are shown: synthetic compound **20** (A); reaction product #3 (B); authentic compound **22** (C); reaction product #4 (D); and authentic compound **21** (E).

Evaluation of binding of compounds 3-6 to MltB. To evaluate binding of compounds **3-6** to MltB, we followed the extinction of the intrinsic fluorescence of MltB upon titration with increasing concentration of each compound. A total of 2 mL of a 0.25 μM solution of MltB in 50 mM HEPES buffer, pH 7.6, 0.20 M NaCl, and Brij-35 was titrated upon addition of aliquots of a 42 mM stock solution of compounds **3-6** in water. After each addition, the mixture was allowed to equilibrate at 25 °C with stirring (using a 7 × 2 mm stir bar) in a 3 mL fluorescence cell. The fluorescence spectra of the protein or protein-compound mixture were recorded with a Varian Cary Eclipse Fluorescence Spectrophotometer (Varian; λ_{exc} = 280 nm, λ_{em} = 290-450 nm, Excitation slit = 10 nm, Emission slit = 5 nm, PMT-V = 600 V). The MltB fluorescence emission spectrum presented a maximum at 334.06 nm (Figure S9). Upon titration of MltB with compounds **3-6** there was a decrease in the emission intensity at 334.06 nm, and a slight shift in the maximum of emission to a lower wavelength (Figure S9). Before fitting the binding data, the fluorescence intensity at 334.06 nm was corrected for dilution. For compounds **3** and **4**, the change in fluorescence intensity with increasing compound concentration was fit using equation S1 for a one-site saturation plus non-specific binding model (Figure S9.A-D; Table S1). For compounds **5** and **6**, the change in fluorescence intensity with increasing compound concentration was fit using equation S2 for a one-site saturation binding model (Figure S9.E-H; Table S1).

$$\Delta If = \frac{\Delta If_{\max} \cdot x}{K_d + x} + k_{ns} \cdot x \qquad (Equation\ S1)$$

$$\Delta If = \frac{\Delta If_{\max} \cdot x}{K_d + x} \qquad (Equation\ S2)$$

Table S1. Parameters obtained from the fit of the change of MltB intrinsic fluorescence upon titration with compounds **3-6** (Figure S7) using equation S1 (compounds **3** and **4**) or equation S2 (compounds **5** and **6**).

Compound	K_d **(μM)**	k_{ns} **(μM^{-1})**
Compound **3**	174 ± 9	0.128 ± 0.004
Compound **4**	1000 ± 200	0.060 ± 0.006
Compound **5**	189 ± 8	-
Compound **6**	1010 ± 20	-

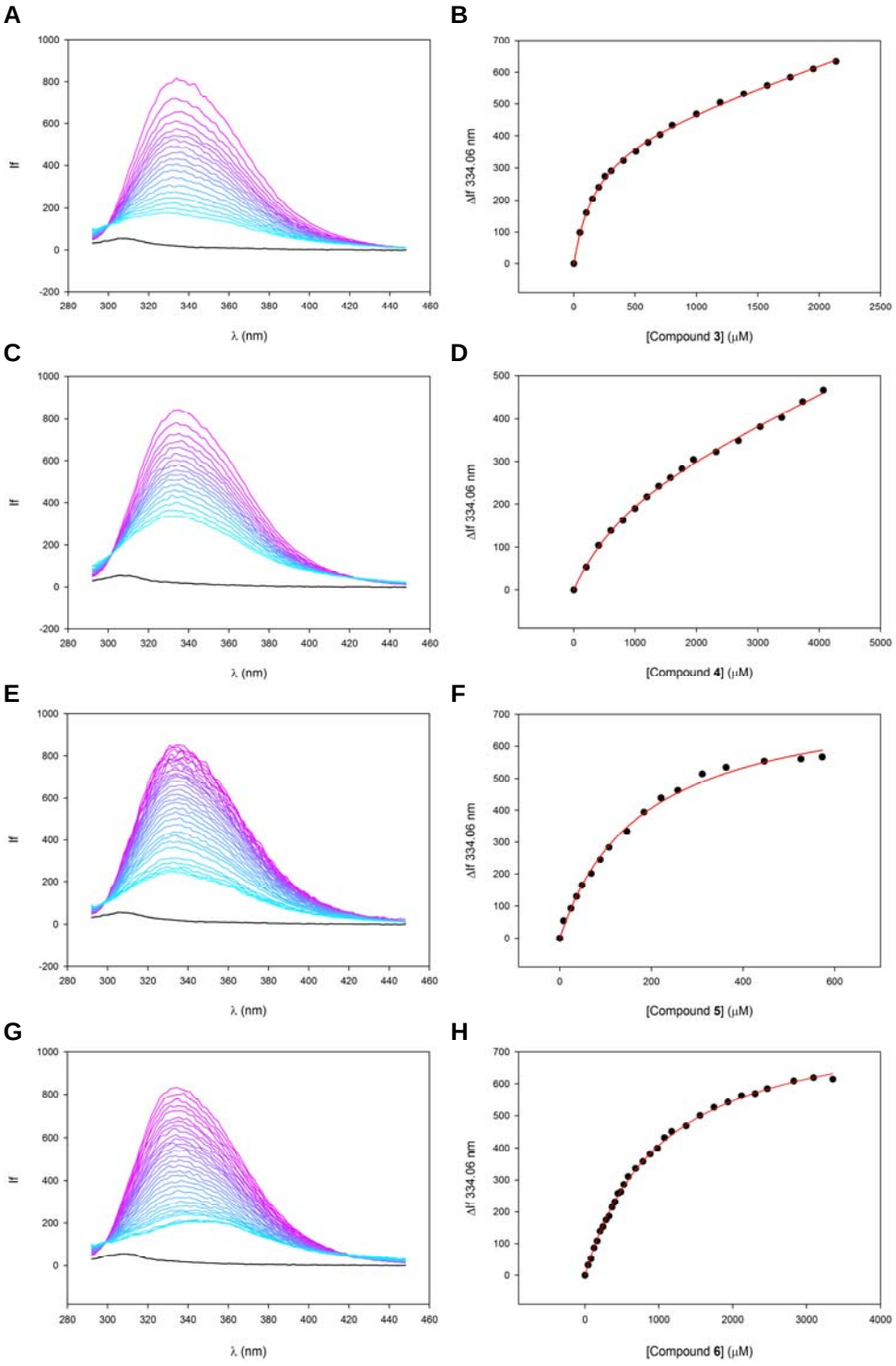
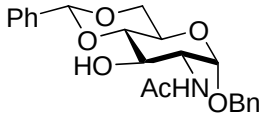


Figure S9. Left panels: fluorescence spectra of MltB titrated with increasing concentration of compounds **3-6** (pink: MltB in buffer, pink to cyan: MltB with increasing concentrations of the corresponding compound; black: buffer) **A.** Compound **3**. **C.** Compound **4**. **E.** Compound **5**. **G.** Compound **6**. Right panels: change in the intensity of fluorescence at 334.06 nm with increasing concentration of compounds **3-6**, where ΔI_f = - ($I_{f\,\text{final}}$ - $I_{f\,\text{initial}}$). **B.** Compound **3**. **D.** Compound **4**. **F.** Compound **5**. **H.** Compound **6**. In each case, the red line shows the best fit (to equation S1 for compounds **3** and **4** and to equation S2 for compounds **5** and **6**).

Synthetic Procedures

Compounds **1**, **2**, **20**, and **21** were prepared according to the literature methods developed by our laboratory.[5]

Benzyl 2-acetamido-4,6-*O*-benzylidene-2-deoxy-α-D-glucopyranoside (9)



The compound **9** was prepared from D-(+)-glucosamine by a variation of the known procedures.[6] Sodium methoxide (32.6 g, 0.60 mol) was added to a suspension of D-(+)-glucosamine hydrochloride (100 g, 0.46 mol) in MeOH (1 L), and the mixture was stirred for 30 min at 40 °C. After addition of acetic anhydride (79 mL, 0.83 mol), the resulting suspension was stirred vigorously for 22 h at 40 °C, and then cooled to 0 °C. After filtration of the reaction mixture, the filtered white solid was washed with cold MeOH and dried to afford *N*-acetyl D-glucosamine (100 g, 97%) as a white powder.

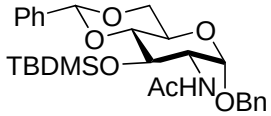
Acetyl chloride (27.4 mL, 0.39 mol) was slowly added to a suspension of *N*-acetyl D-glucosamine (85 g, 0.39 mol) in anhydrous benzyl alcohol (300 mL) under a nitrogen atmosphere. The suspension was stirred at room temperature for 2 h, heated to 50 °C for 4 h, and then cooled to room temperature. The resulting yellow solution was slowly poured into Et_2O (3 L) in ice-water bath, and the mixture was vigorously stirred for 2 h at 0 °C. The precipitate was recovered by filtration and dried under vacuum to afford benzyl 2-acetamido-2-deoxy-α-D-glucopyranoside (113 g, 94%) as a white solid.

Benzaldehyde dimethylacetal (58 mL, 0.39 mol) and *p*-toluenesulfonic acid monohydrate (1.8 g, 9.6 mmol) were added to a solution of benzyl 2-acetamido-2-deoxy-α-D-glucopyranoside (60 g, 0.19 mol) in anhydrous DMF (500 mL), and the mixture was stirred at 70 °C for 24 h. The resulting mixture was cooled to 0 °C and then triethylamine (8.1 mL, 56 mmol) was added. After stirring for 30 min at room temperature, the solvent and extra reagent were removed under reduced pressure to give a white solid. MeOH was added to the solid and the resulting suspension was stirred vigorously for 5 min. After filtration, the filtered white solid was washed well with MeOH and dried under vacuum to afford **2** (67.7 g, 88%) as a white powder. **2**: ^{1}H NMR (500 HMz, DMSO-d_6) δ 1.85 (s, 3H), 3.51 (dd, J = 8.5, 8.5 Hz, 1H, H-4), 3.63–3.80 (m, 3H, H-3, H-5, H-6a), 3.85 (ddd, J = 3.6, 8.2, 8.4 Hz, 1H, H-2), 4.14 (dd, J = 2.8, 8.6 Hz, 1H, H-6b), 4.48 and 4.70 (AB, J = 12.7 Hz, 2H, OCH_2Ph), 4.82 (d, J = 2.4 Hz, 1H, H-1), 5.19 (d, J = 5.8 Hz, 1H, O*H*), 5.61 (s, 1H, C*H*Ph), 7.26–7.49 (m, 10H), 8.00 (d, J = 8.0 Hz, 1H, N*H*); ^{13}C NMR (126 MHz, DMSO-d_6) δ 22.5, 54.2, 62.8, 67.2, 68.5, 82.1, 96.9, 100.9, 126.4, 127.6, 127.6, 128.0, 128.3, 137.7, 137.7, 169.4; IR 3399, 3302, 1651, 1552, 1452, 1374, 1129, 1087, 1059 cm^{-1}; HRMS (FAB) calcd for $C_{22}H_{25}NO_6$ (M + H$^+$) 400.1760, found 400.1763.

[5] (a) Hesek, D.; Lee, M.; Zhang, W.; Noll, B. C.; Mobashery, S. *J. Am. Chem. Soc.* **2009**, *131*, 5187-5193. (b) Lee, M.; Z hang, W.; Hesek, D.; Noll, B. C.; Boggess, B.; Mobashery, S. *J. Am.Chem. Soc.* **2009**, *131*, 8742-8743. (c) Lee, M.; He sek, D; Shah, I. M.; Oliver, A. G.;Dworkin, J.; Mobashery, M. *ChemBioChem* **2010,** *11*, 2525-2529.

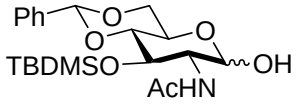
[6] (a) Berger, I.; Nazarov, A. A.; Hartinger, C. G.; Groessl, M.; Valiahdi, S-M.; Jakupec, M. A.; Keppler, B. K. *ChemMedChem* **2007**, *2*, 505−514. (b) Babic, A.; Pecar, S. *Tetrahedron: Asymmetry* **2008**, *19*, 2265−2271.

Benzyl 2-acetamido-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-α-D-glucopyranoside (10)



Imidazole (22.2 g, 0.33 mol) and *t*-butyldimethylsilyl chloride (25.8 g, 0.17 mol) were added to a solution of **9** (65 g, 0.16 mol) in anhydrous DMF (400 mL), and the reaction mixture was stirred at room temperature for 30 min and then 70 °C for 2.5 h under nitrogen atmosphere. After the mixture was cooled to room temperature, saturated $NaHCO_3$ (30 mL) was added. The mixture was concentrated to dryness, and the residue was dissolved in Et_2O then washed with water and brine. After drying over Na_2SO_4, the organic layer was concentrated under reduced pressure, and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3 to 1:1) to afford the product **10** (83.6 g, quant.) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ –0.03 (s, 3H), 0.03 (s, 3H), 0.83 (s, 9H), 1.96 (s, 3H), 3.56 (dd, *J* = 9.2, 9.2 Hz, 1H, H-4), 3.76 (dd, *J* = 10.3, 10.3 Hz, 1H, H-6a), 3.89 (ddd, *J* = 4.8, 10.0, 10.0 Hz, 1H, H-5), 3.90 (dd, *J* = 9.0, 9.8 Hz, 1H, H-3), 4.24 (dd, *J* = 4.8, 10.2 Hz, 1H, H-6b), 4.31 (ddd, *J* = 3.8, 9.9, 9.9 Hz, 1H, H-2), 4.49 and 4.73 (AB, *J* = 11.8 Hz, 2H, C*H*$_2$Ph), 4.89 (d, *J* = 3.8 Hz, 1H, H-1), 5.53 (s, 1H, C*H*Ph), 5.61 (d, *J* = 9.8 Hz, 1H, N*H*), 7.33–7.53 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ –4.8, –3.9, 18.2, 23.6, 25.8, 54.1 (C-2), 63.2 (C-5), 69.1 (C-6), 70.1 (*C*H$_2$Ph), 70.8 (C-3), 82.7 (C-4), 97.9 (C-1), 102.0 (*C*HPh), 126.4, 128.3, 128.4, 128.4, 128.8, 129.2, 137.1, 137.4, 169.8; HRMS (FAB) calcd for $C_{28}H_{40}NO_6Si$ (M + H$^+$) 574.2625, found 574.2615.

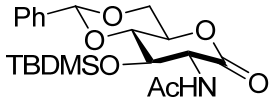
2-Acetamido-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-α,β-D-glucosamine (11)



α:β = 7:1

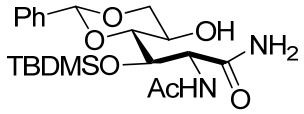
The compound **10** (40 g, 78 mmol, α:β = 5:1) was dissolved in THF (500 mL), and 10 wt% Pd/C (15 g) was added to the solution cautiously to avoid ignition. After stirring for 10 days under a hydrogen atmosphere at room temperature, the mixture was filtered through a layer of Celite, and the Celite pad was washed well with THF. The solution was removed under reduced pressure to afford the product **11** (33 g, quant., α:β = 7:1) as a white powder. ^{1}H NMR (500 HMz, $CDCl_3$) δ –0.10 (s, 21/8H, α), –0.08 (s, 3/8H, β), –0.04 (s, 21/8H, α), –0.01 (s, 3/8H, β), 0.75 (s, 63/8H, α), 0.76 (s, 9/8H, β), 1.94 (s, 21/8H, α), 1.98 (s, 3/8H, β), 3.38 (ddd, *J* = 4.8, 9.6, 9.6 Hz, 1/8H, H-5, β), 3.44 (dd, *J* = 9.3, 9.3 Hz, 1H, H-4), 3.66 (dd, *J* = 10.3, 10.3 Hz, 1H, H-6a, α and H-2, β), 3.72 (dd, *J* = 10.3, 10.3 Hz, 1/8H, H-6a, β), 3.79 (dd, *J* = 9.2, 9.2 Hz, 1/8H, H-3, β), 3.90 (dd, *J* = 9.4, 9.4 Hz, 7/8H, H-3, α), 3.98 (ddd, *J* = 5.0, 9.9, 9.9 Hz, 7/8H, H-5. α), 4.10 (ddd, *J* = 3.4, 9.8, 9.8 Hz, 7/8H, H-2, α), 4.16 (dd, *J* = 4.9, 10.1 Hz, 7/8H, H-6b, α), 4.26 (dd, *J* = 4.8, 10.4 Hz, 1/8H, H-6b, β), 4.61 (dd, *J* = 7.9, 7.9 Hz, 1/8H, H-1, β), 4.82 (d, *J* = 3.2 Hz, 7/8H, O*H*, α), 5.11 (dd, *J* = 3.6, 3.6 Hz, 7/8H, H-1, α), 5.42 (s, 1H, C*H*Ph), 5.57 (d, *J* = 7.6 Hz, 1/8H, O*H*, β), 5.84 (d, *J* = 9.6 Hz, 7/8H, N*H*, α), 6.16 (d, *J* = 7.2 Hz, 1/8H, N*H*, β), 7.24–7.42 (m, 5H); ^{13}C NMR for the α-isomer (The β-isomer was undetectable because it is the minor isomer.) (126 MHz, $CDCl_3$) δ –4.7, –3.9, 18.3, 23.7, 25.8, 54.9, 62.9, 69.2, 70.4, 82.8, 92.8, 102.1, 126.5, 128.3, 129.2, 137.4, 170.7; HRMS (FAB) calcd for $C_{21}H_{34}NO_6Si$ (M + H$^+$) 424.2155, found 424.2137.

2-Acetamido-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-D-glucono-1,5-lactone (12)



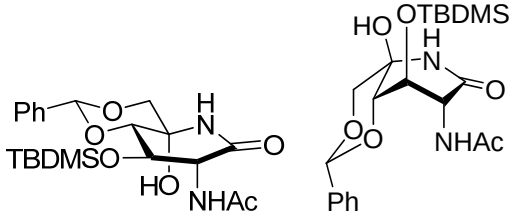
Molecular sieves 4 Å (20 g) and pyridinium chlorochromate (26.6 g, 124 mmol) were added to a solution of **11** (34.9 g, 82.4 mmol) in CH_2Cl_2 (300 mL), and the reaction mixture was stirred for 48 h at room temperature. After filtration through a short layer of silica gel, the resulting solution was concentrated *in vacuo* to afford the crude product **12** (35 g) as a light-brown foam. The crude product was used for next reaction without further purification. However, a portion of pure product (a white powder) was prepared by silica gel column chromatography (AcOEt) for the purpose of characterizarion of the compound. ^{1}H NMR (500 HMz, $CDCl_3$) δ –0.03 (s, 3H), –0.02 (s, 3H), 0.85 (s, 9H), 2.08 (s, 3H), 3.69 (dd, *J* = 7.2, 8.0 Hz, 1H, H-2), 3.73 (dd, *J* = 9.5, 9.5 Hz, 1H, H-4), 3.81 (dd, *J* = 10.0, 12.2 Hz, 1H, H-6a), 4.31 (dd, *J* = 8.0, 9.6 Hz, 1H, H-3), 4.49 (dd, *J* = 5.5, 12.5 Hz, 1H, H-6b), 4.51 (ddd, *J* = 5.4, 9.8, 9.8 Hz, 1H, H–5), 5.57 (s, 1H, C*H*Ph), 6.48 (d, *J* = 7.0 Hz, 1H, N*H*), 7.36–7.50 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ –4.7, –4.0, 18.3, 22.7, 25.9, 59.6, 68.0, 68.1, 72.3, 79.2, 102.1, 126.4, 128.4, 129.5, 136.8, 168.3, 170.9; HRMS (FAB) calcd for $C_{21}H_{32}NO_6Si$ (M + H^+) 422.1999, found 422.1985.

2-Acetamido-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-D-gluconamide (13)



Anhydrous methanolic ammonia (7 N, 412 mL, 2.88 mol) was added to a solution of the crude **12** (35 g) in anhydrous CH_2Cl_2 (600 mL). The reaction mixture was stirred for 3 h under a nitrogen atmosphere, followed by concentrated *in vacuo*. The crude product was purified by silica gel column chromatography (AcOEt) to afford the title compound (27.8 g, 77% from **12**) as a white powder. ^{1}H NMR (500 HMz, $CDCl_3$) δ 0.08 (s, 3H), 0.18 (s, 3H), 0.90 (s, 9H), 1.99 (s, 3H), 3.49 (brs, 1H, O*H*), 3.57 (dd, *J* = 10.5, 10.5 Hz, 1H, H-6a), 3.64 (dd, *J* = 4.0, 9.4 Hz, 1H, H-4), 3.86 (ddd, *J* = 5.3, 9.7, 9.7 Hz, 1H, H-5), 4.29 (dd, *J* = 5.4, 10.8 Hz, 1H, H-6b), 4.56 (dd, *J* = 3.6, 3.6 Hz, 1H, H-3), 4.69 (dd, *J* = 3.4, 6.8 Hz, 1H, H–2), 5.41 (s, 1H, C*H*Ph), 5.63 (brs, 1H, NH_2), 6.76 (brs, 1H, NH_2), 6.82 (d, *J* = 6.8 Hz, 1H, N*H*Ac), 7.33–7.46 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ –4.6, 18.4, 23.4, 26.1, 55.3 (C-2), 62.4 (C-5), 71.1 (C-3), 71.5 (C-6), 82.0 (C-4), 101.7 (*C*HPh), 126.5, 128.4, 129.3, 137.7, 171.3, 172.9; HRMS (FAB) calcd for $C_{21}H_{35}N_2O_6Si$ (M + H^+) 439.2264, found 439.2251.

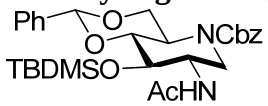
2-Acetamido-5-amino-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-D-glucono-1,5-lactam (8a), 2-Acetamido-5-amino-4,6-*O*-benzylidene-3-*O*-*tert*-butyldimethylsilyl-2-deoxy-L-idono-1,5-lactam (8b)



8a:**8b** = 2:3

Molecular sieves 4Å (10 g), 4-methylmorpholine *N*-oxide (3.23 g, 27.6 mmol) and tetra-*n*-propylammonium perruthenate (2.0 g, 5.7 mmol) were added to a solution of **13** (9.3 g, 21.2 mmol) in anhydrous CH_2Cl_2 (200 mL). The reaction mixture was stirred for 40 h under a nitrogen atmosphere**.** The mixture was filtered and the solution was concentrated *in vacuo*. The crude product was purified by silica gel column chromatography (AcOEt) to afford the mixture of **8a** and **8b** (5.65 g, **8a**:**8b** = 2:3, 61%) as a white powder. ^{1}H NMR (500 HMz, $CDCl_3$, of the mixture of **8a** and **8b**) δ –0.01 (s, 6/5H, A), 0.05 (s, 6/5H, A), 0.24 (s, 9/5H, B), 0.30 (s, 9/5H, B), 0.84 (s, 18/5H, A), 0.93 (s, 27/5H, B), 1.88 (s, 9/5H, B), 2.05 (s, 6/5H, A), 3.80 (d, *J* = 11.0 Hz, 2/5H, H-6a, A), 3.88 (d, *J* = 9.8 Hz, 2/5H, H-4, A), 3.97 (d, *J* = 12.4 Hz, 3/5H, H-6a, B), 4.00 (d, *J* = 11.0 Hz, 2/5H, H-6b, A), 4.03 (d, *J* = 12.2 Hz, 3/5H, H-6b, B), 4.18 (dd, *J* = 8.6, 8.6 Hz, 2/5H, H-2, A), 4.21 (dd, *J* = 0.7, 4.2 Hz, 3/5H, H-4, B), 4.27 (dd, *J* = 1.6, 4.0 Hz, 3/5H, H-3, B), 4.39 (dd, *J* = 8.6, 9.6 Hz, 2/5H, H-3, A), 4.67 (s, 2/5H, O*H*, A), 4.72 (dd, *J* = 1.6, 9.0 Hz, 3/5H, H-2, B), 4.73 (s, 3/5H, O*H*, B), 5.62 (s, 3/5H, C*H*Ph, B), 5.64 (s, 2/5H, C*H*Ph, A), 6.32 (d, *J* = 9.2 Hz, 3/5H, N*H*Ac, B), 6.54 (s, 2/5H, N*H*, A), 6.68 (s, 3/5H, N*H*, B), 6.83 (d, *J* = 8.6 Hz, 2/5H, N*H*Ac, A), 7.37–7.54 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ –5.3, –4.6, –4.6, –4.1, 18.1, 18.3, 23.1, 23.2, 25.8, 25.9, 52.7 (C-2, B), 58.4 (C-2, A), 69.7 (C-3, A), 72.0 (C-3, B), 73.2 (C-6, A), 73.5 (C-6, B), 74.7 (C-5), 76.0 (C-4, B), 76.8 (C-5), 81.1 (C-4, A), 101.8 (*C*HPh, B), 102.9 (*C*HPh, A), 126.1, 126.5, 128.4, 128.8, 129.6, 129.9, 136.6, 136.6, 168.8, 169.6, 170.9, 171.6; HRMS (FAB) calcd for $C_{21}H_{33}N_2O_6Si$ ($M + H^+$) 437.2108, found 437.2108.

2-Acetamido-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-3-*O*-*tert*-butyldimethylsilyl-1,2,5-trideoxy-D-glucitol (14)

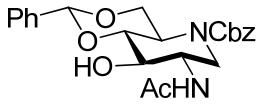


Borane dimethyl sulfide complex (2 M solution in THF, 45 mL, 90 mmol) was added dropwise to a solution of **8ab** (3.93 g, 9.0 mmol) in anhydrous CH_2Cl_2 (400 mL) at 0 °C, and the reaction mixture was stirred for 18 h at room temperature under a nitrogen atmosphere. The mixture was slowly quenched by the addition of MeOH (80 mL), and then stirred for another 2 h. After removal of the solvents under reduced pressure, the crude product (4.1 g) was used for the next reaction without purification. A portion of the pure product (a white powder) was obtained by silica gel column chromatography (AcOEt/MeOH, 4:1) for characterization of the compound. ^{1}H NMR (500 HMz, $CDCl_3$) δ –0.06 (s, 3H), 0.04 (s, 3H), 0.83 (s, 9H), 1.99 (s, 3H), 2.49 (dd, *J* = 11.1, 12.6 Hz, 1H), 2.75 (ddd, *J* = 4.6, 9.3, 10.3 Hz, 1H), 3.37 (dd, *J* = 9.0, 9.0 Hz, 1H), 3.38 (dd, *J* = 4.8, 12.7 Hz, 1H), 3.56−3.66 (m, 2H), 3.88 (m, 1H), 4.24 (dd, *J* = 4.7, 10.6 Hz, 1H), 5.34 (br d, *J* = 7.4 Hz, 1H), 5.51 (s, 1H), 7.32–7.52 (m, 5H); HRMS (FAB) calcd for $C_{21}H_{35}N_2O_4Si$ ($M + H^+$) 407.2366, found 407.2358.

Pyridine (5.4 mL, 54 mmol) and benzyloxycarbonyl chloride (1.92 mL, 13.5 mmol) were added to a solution of the crude product (obtained above, 4.1 g) in CH_2Cl_2 (200 mL), and the reaction mixture was stirred for 1 h under a nitrogen atmosphere. Saturated $NaHCO_3$ was added to the mixture, and the resulting mixture was washed with AcOEt. The combined organic layer was washed with water and brine, dried over Na_2SO_4, and then concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3) to afford **14** (1.41 g, 29% from **8ab**) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ –0.10 (s, 3H), –0.01 (s, 3H), 0.81 (s, 9H), 1.96 (s, 3H), 2.89 (dd, *J* = 10.3, 13.5 Hz, 1H, H-1a), 3.28 (ddd, *J* = 4.4, 10.0, 10.0 Hz, 1H, H-5), 3.64 (dd, *J* = 8.9, 8.9 Hz, 1H, H-3), 3.68 (dd, *J* = 8.4, 8.4 Hz, 1H, H-4), 3.84 (m, 1H, H-2), 4.35 (dd, *J* = 4.6, 13.6 Hz, 1H, H-1b), 4.42 (dd, *J* = 11.0, 11.0 Hz, 1H, H-6a), 4.79 (dd, *J* = 4.5, 11.5 Hz, 1H, H-6b), 5.08 and 5.15

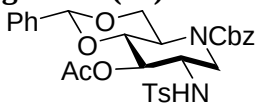
(AB, J = 12.5 Hz, 2H, CH_2Ph), 5.33 (d, J = 7.2 Hz, 1H, NHAc), 5.52 (s, 1H, CHPh), 7.30–7.49 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ –4.8, –3.8, 18.4, 23.7, 25.9, 47.8 (C-1), 53.0 (C-2), 55.2 (C-5), 67.7 (CH_2Ph), 69.6 (C-6), 75.2 (C-4), 81.7 (C-3), 102.2 (CHPh), 126.6, 128.3, 128.4, 128.4, 128.8, 129.3, 136.3, 137.5, 154.9, 170.3; HRMS (FAB) calcd for $C_{29}H_{41}N_2O_6Si$ (M + H^+) 541.2734, found 541.2726.

2-Acetamido-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-1,2,5-trideoxy-D-glucitol (7)



TBAF (1 M solution in THF, 3.27 mL, 3.27 mmol) was added to a solution of **14** (1.36 g, 2.52 mmol) in THF (100 mL), and the mixture was stirred for 24 h. After removal of the solvent, the crude product was purified by silica gel column chromatography (AcOEt/CH_2Cl_2, 10:1) to afford the product **7** (1.07 g, quant.) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ 2.02 (s, 3H, CH_3CO), 2.84 (dd, J = 9.9, 13.5 Hz, 1H, H-1a), 3.31 (ddd, J = 4.5, 9.8, 9.8 Hz, 1H, H-5), 3.69 (dd, J = 8.6, 8.6 Hz, 1H, H-3), 3.71 (dd, J = 8.1, 8.1 Hz, 1H, H-4), 3.88 (m, 1H, H-2), 4.40 (dd, J = 11.0, 11.0 Hz, 1H, H-6a), 4.46 (dd, J = 4.7, 13.7 Hz, 1H, H-1b), 4.82 (dd, J = 4.5, 11.5 Hz, 1H, H-6b), 5.10 and 5.17 (AB, J = 12.2 Hz, 2H, OCH_2Ph), 5.59 (s, 1H, CHPh), 5.61 (d, J = 5.6 Hz, 1H, NH), 7.31–7.51 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.5, 47.4 (C-1), 52.2 (C-2), 55.0 (C-5), 67.8 (OCH_2Ph), 69.4 (C-6), 74.5 (C-3), 80.9 (C-4), 101.8 (CHPh), 126.5, 128.3, 128.5, 128.5, 128.9, 129.5, 136.1, 137.4, 155.0 (C=O), 171.6 (C=O); IR 3393, 3283, 1716, 1694, 1654, 1550, 1427, 1374, 1252, 1143, 1089 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{27}N_2O_6$ (M + H^+) 427.1864, found 427.1860.

3-*O*-Acetyl-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-1,2,5-trideoxy-2-tosylamido-D-glucitol (15)



p-Toluenesulfonyl chloride (23 mg, 0.122 mmol) was added to a solution of **7** (26 mg, 0.061 mmol) in pyridine, and the mixture was refluxed for 30 h. The mixture was cooled to room temperature, and the solvent was removed under vacuum. The crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3) to afford **15** (27 mg, 76%) as a white solid. The colorless crystals for X-ray analysis were prepared from the recrystallization (MeOH/H_2O, 100:1). ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.75 (s, 3H, CH_3CO), 2.41 (s, 3H, CH_3Ph), 2.85 (dd, J = 11.4, 13.8 Hz, 1H, H-1a), 3.31 (ddd, J = 4.6, 10.2, 10.2 Hz, 1H, H-5), 3.37 (m, 1H, H-2), 3.69 (dd, J = 9.5, 9.5 Hz, 1H, H-4), 4.47 (dd, J = 5.1, 13.9 Hz, 1H, H-1b), 4.52 (dd, J = 11.1, 11.1 Hz, 1H, H-6a), 4.74 (dd, J = 4.6, 11.8 Hz, 1H, H-6b), 4.89 (dd, J = 9.6, 9.6 Hz, 1H, H-3), 5.09 and 5.14 (AB, J = 12.3 Hz, 2H, OCH_2Ph), 5.27 (d, J = 7.0 Hz, 1H, NH), 5.48 (s, 1H, CHPh), 7.24 (d, J = 8.0 Hz, 2H), 7.28–7.46 (m, 10H), 7.68 (d, J = 8.2 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 20.8 (CH_3CO), 21.7 (CH_3Ph), 50.4 (C-1), 53.9 (C-2), 55.7 (C-5), 67.7 (OCH_2Ph), 69.2 (C-6), 74.4 (C-3), 78.1 (C-4), 101.1 (CHPh), 126.1, 127.1, 128.3, 128.4, 128.5, 128.9, 129.2, 129.9, 136.1, 137.3, 138.0, 143.7, 154.3 (C=O), 172.2 (C=O); IR 3257, 1740, 1703, 1683, 1598, 1441, 1376, 1334, 1244, 1158, 1092, 1034 cm^{-1}; HRMS (ESI) calcd for $C_{30}H_{32}N_2NaO_8S$ (M + Na^+) 603.1772, found 603.1764.

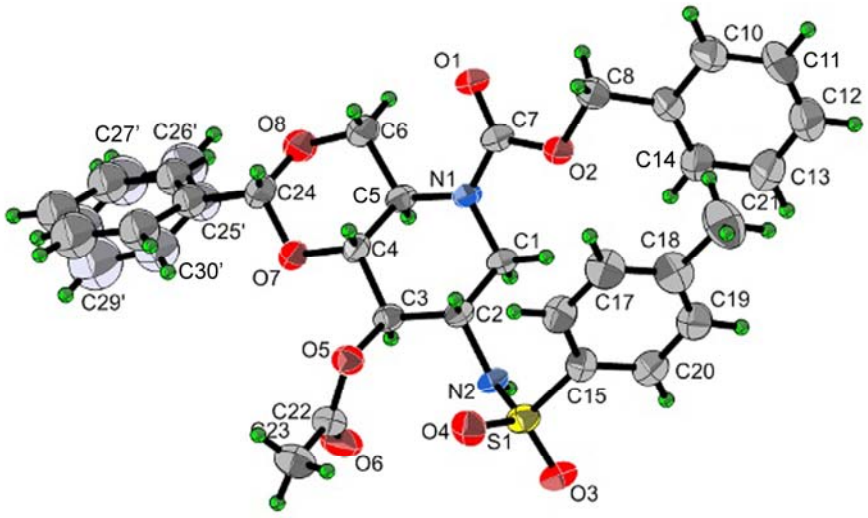
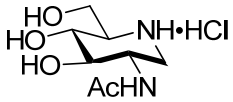


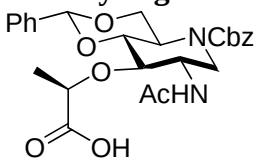
Figure S10. The ORTEP diagram of compound **15**, shown at 30% probability level. The disordered phenyl group (C25'-C30') was modeled as two partial occupancy, rigid-body six-membered rings. The occupancies of the two sites were summed to unity giving an approximate 0.54:0.46 ratio.

2-Acetamido-1,2,5-trideoxy-1,5-imino-D-glucitol hydrochloride (3)



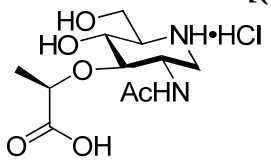
The compound **7** (50 mg, 117 μmol) was dissolved in *i*-PrOH (5 mL), and 10 wt% Pd/C (50 mg) was added to the solution cautiously to avoid ignition. After the addition of conc.HCl (29 μL, 0.35 mmol), the mixture was stirred for 17 h under hydrogen atmosphere and then the suspension was filtered through a layer of Celite. The Celite pad was washed well with *i*-PrOH, and the filtrate was concentrated under reduced pressure to afford the product **3** (28 mg, quant.) as a white powder. ^{1}H NMR (500 HMz, D_2O) δ 2.01 (s, 3H, C*H*$_3$CO), 2.97 (dd, *J* = 12.5, 12.5 Hz, 1H, H-1a), 3.21 (ddd, *J* = 3.4, 5.0, 10.3 Hz, 1H, H-5), 3.48 (dd, *J* = 4.8, 12.8 Hz, 1H, H-1b), 3.60 (dd, *J* = 9.7, 9.7 Hz, 1H, H-3), 3.66 (dd, *J* = 9.8, 9.8 Hz, 1H, H-4), 3.88 (dd, *J* = 5.2, 12.8 Hz, 1H, H-6a), 3.93 (dd, *J* = 3.0, 12.8 Hz, 1H, H-6b), 4.05 (ddd, *J* = 4.8, 10.4, 12.0 Hz, 1H, H-2); ^{13}C NMR (126 MHz, D_2O) δ 22.1 (*C*H$_3$CO), 44.2 (C-1), 48.2 (C-2), 57.7 (C-6), 60.0 (C-5), 68.5 (C-4), 73.5 (C-3), 174.7 (*C*=O); HRMS (FAB) calcd for $C_8H_{17}N_2O_4$ (M + H$^+$) 205.1188, found 205.1189.

2-Acetamido-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-3-*O*-[(1*R*)-1-carboxy]ethyl-1,2,5-trideoxy-D-glucitol (16)



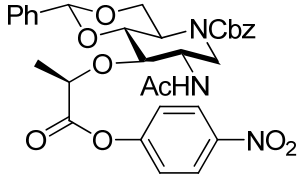
NaH (302 mg, 7.5 mmol, 60% dispersion in oil) was added to a solution of **7** (643 mg, 1.5 mmol) in anhydrous THF (15 mL), and the mixture was stirred at 60 °C for 30 min, and then (*S*)-2-chloropropionic acid (262 μL, 3.0 mmol) was added dropwise at 60 °C over 1 h. After stirring at 60 °C for 4 h, the mixture was stirred at room temperature for 16 h. MeOH was added to the mixture at 0 °C, and the resulting mixture was concentrated under reduced pressure. CH_2Cl_2 and AcOEt were added to the residue, and the resulting suspension was washed with 1 M HCl, water, and then brine. The organic layer was dried over Na_2SO_4, and then concentrated to dryness. The residue was washed with MeOH and AcOEt to afford the product **16** (684 mg, 91%) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$/CD_3OD, 95:5) δ 1.42 (d, *J* = 7.0 Hz, 3H, OCHC*H*$_3$), 2.00 (s, 3H, C*H*$_3$CO), 2.44 (dd, *J* = 11.0, 13.2 Hz, 1H, H-1a), 3.20 (ddd, *J* = 4.5, 10.1, 10.1 Hz, 1H, H-5), 3.45 (dd, *J* = 9.0, 10.0 Hz, 1H, H-3), 3.61 (ddd, *J* = 4.6, 10.5, 10.5 Hz, 1H, H-2), 3.81 (dd, *J* = 9.2, 9.2 Hz, 1H, H-4), 4.52 (dd, *J* = 11.1, 11.1 Hz, 1H, H-6a), 4.60 (q, *J* = 7.1 Hz, 1H, OC*H*CH$_3$), 4.81 (dd, *J* = 4.6, 11.8 Hz, 1H, H-6b), 4.94 (dd, *J* = 4.6, 13.4 Hz, 1H, H-1b), 5.08 and 5.11 (AB, *J* = 12.6 Hz, 2H, OC*H*$_2$Ph), 5.58 (s, 1H, C*H*Ph), 7.25–7.46 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$/CD_3OD, 95:5) δ 18.8 (OCH*C*H$_3$), 23.0 (*C*H$_3$CO), 48.2 (C-1), 51.2 (C-2), 55.9 (C-5), 67.5 (O*C*H$_2$Ph), 69.3 (C-6), 75.2 (O*C*HCH$_3$), 79.8 (C-3), 82.2 (C-4), 101.0 (*C*HPh), 125.8, 127.9, 128.2, 128.5, 128.7, 129.1, 136.2, 137.5, 154.7 (*C*=O), 172.5 (*C*=O), 177.5 (*C*=O); HRMS (ESI) calcd for $C_{26}H_{31}N_2O_8$ (M + H$^+$) 499.2075, found 499.2057.

2-Acetamido-3-*O*-[(1*R*)-1-carboxy]ethyl-1,2,5-trideoxy-1,5-imino-D-glucitol hydrochloride (4)



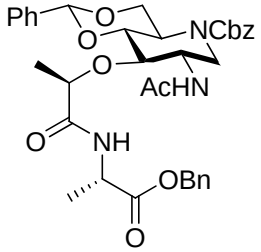
Compound **16** (40 mg, 80 μmol) was dissolved in *i*-PrOH (3 mL) and 10 wt% Pd/C (40 mg) was added to the solution cautiously to avoid ignition. After the addition of conc.HCl (13 μL, 0.16 mmol), the mixture was stirred for 18 h under hydrogen atmosphere and the suspension was filtered through a layer of Celite. The Celite pad was washed well with *i*-PrOH, and the filtrate was concentrated under reduced pressure to afford the product **4** (25 mg, quant.) as a white powder. ^{1}H NMR (500 HMz, D_2O) δ 1.41 (d, *J* = 7.0 Hz, 3H, OCHC*H*$_3$), 1.97 (s, 3H, C*H*$_3$CO), 2.95 (dd, *J* = 12.6, 12.6 Hz, 1H, H-1a), 3.21 (ddd, *J* = 3.6, 4.4, 10.5 Hz, 1H, H-5), 3.49 (dd, *J* = 4.8, 12.8 Hz, 1H, H-1b), 3.61 (dd, *J* = 9.7, 9.7 Hz, 1H, H-3), 3.74 (dd, *J* = 9.8, 9.8 Hz, 1H, H-4), 3.87 (dd, *J* = 4.9, 12.7 Hz, 1H, H-6a), 3.92 (dd, *J* = 2.9, 12.9 Hz, 1H, H-6b), 4.06 (ddd, *J* = 4.8, 11.3, 11.3 Hz, 1H, H-2), 4.44 (q, *J* = 6.9 Hz, 1H, OC*H*CH$_3$); ^{13}C NMR (126 MHz, D_2O) δ 18.6 (OCH*C*H$_3$), 22.4 (*C*H$_3$CO), 44.1 (C-1), 47.6 (C-2), 57.6 (C-6), 60.0 (C-5), 69.3 (C-4), 77.6 (O*C*HCH$_3$), 82.3 (C-3), 174.5 (*C*=O), 177.4 (*C*=O); HRMS (ESI) calcd for $C_{11}H_{21}N_2O_6$ (M + H$^+$) 277.1394, found 277.1404.

2-Acetamido-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-1,2,5-trideoxy-3-*O*-[(1*R*)-1-(4-nitrophenoxy)carbonyl]ethyl-D-glucitol (17)



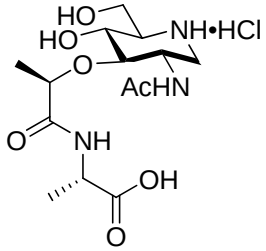
Pyridine (0.50 mL, 6.21 mmol) and *p*-nitrophenyl trifluoroacetate (773 mg, 3.29 mmol) were added to a solution of **16** (410 mg, 0.82 mmol) in anhydrous CH_2Cl_2 (15 mL), and then the mixture was added TEA (0.23 mL, 1.64 mmol) and stirred for 23 h. After the addition of water, the mixture was extracted with AcOEt. The combined organic layer was washed with water and brine, then dried over Na_2SO_4, and then concentrated to dryness. The resulting yellow residue was washed with AcOEt to afford the product **17** (367 mg, 72%) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.64 (d, *J* = 7.0 Hz, 3H, OCHC*H*$_3$), 1.97 (s, 3H, C*H*$_3$CO), 2.49 (dd, *J* = 10.9, 13.5 Hz, 1H, H-1a), 3.25 (ddd, *J* = 4.6, 10.1, 10.1 Hz, 1H, H-5), 3.52 (dd, *J* = 8.8, 10.2 Hz, 1H, H-3), 3.73 (ddd, *J* = 4.4, 10.4, 14.8 Hz, 1H, H-2), 3.81 (dd, *J* = 9.4, 9.4 Hz, 1H, H-4), 4.58 (dd, *J* = 11.1, 11.1 Hz, 1H, H-6a), 4.88 (dd, *J* = 4.6, 11.8 Hz, 1H, H-6b), 4.94 (dd, *J* = 5.0, 13.8 Hz, 1H, H-1b), 4.95 (q, *J* = 7.0 Hz, 1H, OC*H*CH$_3$), 5.11 and 5.15 (AB, *J* = 12.5 Hz, 2H, OC*H*$_2$Ph), 5.65 (s, 1H, C*H*Ph), 7.25–7.51 (m, 12H), 8.30 (d, *J* = 9.2 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.9 (OCH*C*H$_3$), 23.5 (*C*H$_3$CO), 48.4 (C-1), 51.2 (C-2), 55.9 (C-5), 67.5 (O*C*H$_2$Ph), 69.5 (C-6), 75.2 (O*C*HCH$_3$), 80.3 (C-3), 82.3 (C-4), 101.1 (*C*HPh), 122.5, 125.8, 125.9, 128.1, 128.3, 128.6, 128.8, 128.8, 129.3, 136.4, 137.6, 145.9, 154.7 (*C*=O), 171.5 (*C*=O), 173.9 (*C*=O); HRMS (ESI) calcd for $C_{32}H_{34}N_3O_{10}$ (M + H$^+$) 620.2239, found 620.2222.

_N_-{2-_O_-[2-Acetamido-4,6-_O_-benzylidene-1,5-(benzyloxycarbonyl)imino-1,2,5-trideoxy-D-glucitol-3-yl]-D-lactoyl}-L-alanine benzyl ester (18)



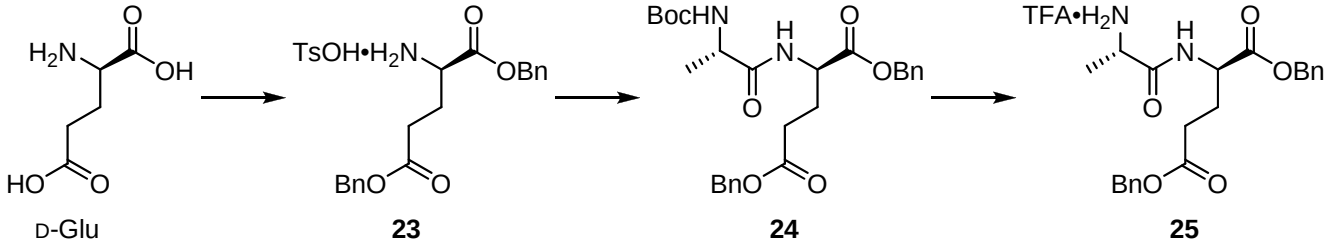
Triethylamine (13 μL, 89 μmol) was added to a solution of L-alanine benzyl ester hydrochloride (19 mg, 89 μmol) in THF/CH_2Cl_2 (6 mL, 1:1), and the mixture was stirred for 15 min. Compound **17** (50 mg, 81 μmol) was added to the mixture, and then the mixture was stirred for 40 h. The volatiles were removed under reduced pressure, and the resulting residue was washed with AcOEt to afford the product **18** (45 mg, 85%) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.37 (d, *J* = 6.8 Hz, 3H, OCHC*H*$_3$), 1.43 (d, *J* = 7.2 Hz, 3H, NHCHC*H*$_3$), 1.98 (s, 3H, C*H*$_3$CO), 2.58 (dd, *J* = 10.4, 13.4 Hz, 1H, H-1a), 3.22 (ddd, *J* = 4.5, 10.2, 10.2 Hz, 1H, H-5), 3.44 (dd, *J* = 9.1, 9.1 Hz, 1H, H-3), 3.76 (m, 1H, H-2), 3.80 (dd, *J* = 8.8, 9.6 Hz, 1H, H-4), 4.40 (q, *J* = 6.7 Hz, 1H, OC*H*CH$_3$), 4.48 (dd, *J* = 11.1, 11.1 Hz, 1H, H-6a), 4.57 (dq, *J* = 7.2, 7.2 Hz, 1H, NHC*H*CH$_3$), 4.70 (dd, *J* = 4.7, 13.5 Hz, 1H, H-1b), 4.82 (dd, *J* = 4.6, 11.6 Hz, 1H, H-6b), 5.09 and 5.15 (AB, *J* = 12.4 Hz, 2H, NCOOC*H*$_2$Ph), 5.19 (s, 2H, CHCOOC*H*$_2$Ph), 5.57 (s, 1H, C*H*Ph), 6.52 (d, *J* = 7.2 Hz, 1H, CON*H*CH), 7.29–7.49 (m, 15H), 7.52 (d, *J* = 4.0 Hz, 1H, N*H*Ac); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.1 (NHCH*C*H$_3$), 19.8 (OCH*C*H$_3$), 23.5 (*C*H$_3$CO), 48.0 (C-1), 48.5 (NH*C*HCH$_3$), 51.3 (C-2), 55.5 (C-5), 67.5 (CHCOO*C*H$_2$Ph), 67.5 (NCOO*C*H$_2$Ph), 69.5 (C-6), 76.8 (O*C*HCH$_3$), 80.6 (C-3), 81.7 (C-4), 101.3 (*C*HPh), 126.1, 128.1, 128.3, 128.4, 128.6, 128.7, 128.8, 128.9, 129.3, 135.4, 136.4, 137.7, 154.8 (*C*=O), 171.5 (*C*=O), 172.6 (*C*=O), 174.1 (*C*=O); HRMS (ESI) calcd for $C_{36}H_{42}N_3O_9$ (M + H$^+$) 660.2916, found 660.2883.

***N*-{2-*O*-[2-Acetamido-1,2,5-trideoxy-1,5-imino-D-glucitol-3-yl]-D-lactoyl}-L-alanine hydrochloride (5)**

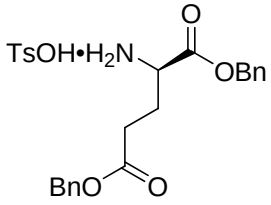


The compound **18** (30 mg, 45 μmol) was dissolved in *i*-PrOH (3 mL), and 10 wt% Pd/C (30 mg) was added to the solution cautiously to avoid ignition. After the addition of conc.HCl (7.6 μL, 91 μmol), the mixture was stirred for 40 h under hydrogen atmosphere and filtered through a layer of Celite. The Celite pad was washed well with *i*-PrOH, and the filtrate was concentrated under reduced pressure to afford the product **5** (17 mg, quant.) as a white powder. ^{1}H NMR (500 HMz, D_2O) δ 1.37 (d, *J* = 6.8 Hz, 3H, CHC*H*$_3$), 1.42 (d, *J* = 7.4 Hz, 3H, CHC*H*$_3$), 1.95 (s, 3H, C*H*$_3$CO), 2.95 (dd, *J* = 12.6, 12.6 Hz, 1H, H-1a), 3.23 (ddd, *J* = 3.4, 4.6, 10.8 Hz, 1H, H-5), 3.46 (dd, *J* = 5.0, 12.8 Hz, 1H, H-1b), 3.56 (dd, *J* = 9.1, 10.3 Hz, 1H, H-3), 3.76 (dd, *J* = 9.1, 10.9 Hz, 1H, H-4), 3.89 (dd, *J* = 4.8, 12.8 Hz, 1H, H-6a), 3.93 (dd, *J* = 3.4, 12.8 Hz, 1H, H-6b), 4.15 (ddd, *J* = 4.9, 10.3, 12.3 Hz, 1H, H-2), 4.30 (q, *J* = 6.9 Hz, 1H, C*H*CH$_3$), 4.32 (q, *J* = 7.4 Hz, 1H, C*H*C*H*$_3$); ^{13}C NMR (126 MHz, D_2O) δ 16.1 (CH*C*H$_3$), 18.7 (CH*C*H$_3$), 22.2 (*C*H$_3$CO), 44.2 (C-1), 47.8 (C-2), 48.7 (NH*C*HCH$_3$), 57.6 (C-6), 59.9 (C-5), 67.7 (C-4), 78.5 (O*C*HCH$_3$), 82.0 (C-3), 174.3 (*C*=O), 175.7 (*C*=O), 176.5 (*C*=O); HRMS (ESI) calcd for $C_{14}H_{25}N_3NaO_7$ (M + Na^+) 370.1585, found 370.1576.

Scheme S1. Synthesis of Compound **25**



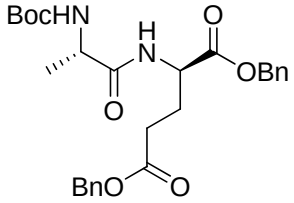
Dibenzyl D-glutamate *p*-toluenesulfonate (23)



A mixture of D-glutamic acid (1.5 g, 10.2 mmol), *p*-toluenesulfonic acid monohydrate (2.33 g, 12.2 mmol) and benzyl alcohol (26 mL) in benzene (80 mL) was refluxed for 26 h with the removal of water in a Dean-Stark apparatus. The clear solution was cooled to room temperature and concentrated to half the volume under reduced pressure. The resulting solution was poured into Et_2O (200 mL) in an ice-water bath, and the mixture was stirred for 2 h at that temperature. The precipitate was recovered by filtration, and washed with Et_2O to afford the title compound (3.90 g, 77%) as a white solid. ^{1}H NMR

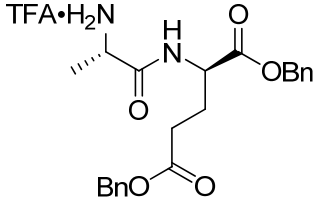
(500 HMz, $CDCl_3$) δ 2.18 (m, 2H, CHCH_2CH$_2$), 2.24 (CH_3Ph), 2.39 (dt, *J* = 7.2, 17.6 Hz, 1H, CHCH$_2$CH_2), 2.51 (dt, *J* = 7.4, 17.8 Hz, 1H, CHCH$_2$CH_2), 1.37 (d, *J* = 6.8 Hz, 3H, CHCH_3), 1.42 (d, *J* = 7.4 Hz, 3H, CHCH_3), 4.14 (m, 1H, C*H*CH$_2$CH$_2$), 4.87−5.06 (m, 4H, CH_2Ph), 6.97 (d, *J* = 7.8 Hz, 2H, Ts), 7.14−7.32 (m, 10H), 7.71 (d, *J* = 8.0 Hz, 2H, Ts), 8.30 (d, *J* = 3.0 Hz, 1H, N*H*)); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.5 (*C*H$_3$Ph), 25.3 (CH*C*H$_2$CH$_2$), 29.5 (CHCH$_2$*C*H$_2$), 52.6 (*C*HCH$_2$CH$_2$), 66.6 and 68.1 (*C*H$_2$Ph), 126.2, 128.3, 128.3, 128.5, 128.6, 128.7, 128.7, 129.1, 134.8, 135.9, 140.6, 141.2, 169.0 (*C*=O), 172.2 (*C*=O); HRMS (ESI) calcd for $C_{19}H_{22}NO_4$ ($M + H^+$) 328.1543, found 328.1546.

Dibenzyl *N*-[*N*-(*tert*-butoxycarbonyl)-L-alanyl]-D-glutamate (24)



Triethylamine (0.34 mL, 2.4 mmol) and 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide hydrochloride (280 mg, 1.4 mmol) were added to a solution of **23** (300 mg, 0.60 mmol) in CH_2Cl_2 (10 mL), and the mixture was stirred for 10 min. Boc-L-alanine (114 mg, 0.60 mmol) was added to the mixture, and the mixture was stirred at room temperature for 40 h. After the addition of water, the mixture was washed with AcOEt. The combined organic layer was washed with water and brine, and dried over Na_2SO_4. The solvent was removed under reduced pressure, and the resulting residue was purified by silica gel column chromatography (AcOEt/hexane, 1:3 to 1:2) to afford the title compound (214 mg, 71%) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.34 (d, *J* = 7.2 Hz, 3H, CHCH_3), 1.44 (s, 9H, *t*-Bu), 2.03 (m, 1H, CHCH_2CH$_2$), 2.26 (m, 1H, CHCH_2CH$_2$), 2.41 (m, 2H, CHCH$_2$CH_2), 4.20 (br s, 1H, C*H*CH$_3$), 4.65 (dt, *J* = 5.0, 8.0 Hz, 1H, C*H*CH$_2$CH$_2$), 4.97 (br s, 1H, N*H*CHCH$_3$), 5.08 and 5.11 (AB, *J* = 12.5 Hz, 2H, CH_2Ph), 5.14 and 5.17 (AB, *J* = 12.3 Hz, 2H, CH_2Ph), 6.92 (br s, 1H, N*H*CHCH$_2$), 7.30−7.39 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.3 (CH*C*H$_3$), 27.3 (CH*C*H$_2$CH$_2$), 28.4 (C(*C*H$_3$)$_3$), 30.2 (CHCH$_2$*C*H$_2$), 50.3 (*C*HCH$_3$), 51.8 (*C*HCH$_2$CH$_2$), 66.7 and 67.5 (*C*H$_2$Ph), 80.4 (*C*(CH$_3$)$_3$), 128.4, 128.5, 128.7, 128.7, 128.8, 135.3, 135.9, 155.7 (*C*=O), 171.6 (*C*=O), 172.7 (*C*=O), 172.8 (*C*=O); HRMS (ESI) calcd for $C_{27}H_{35}N_2O_7$ ($M + H^+$) 499.2439, found 499.2421.

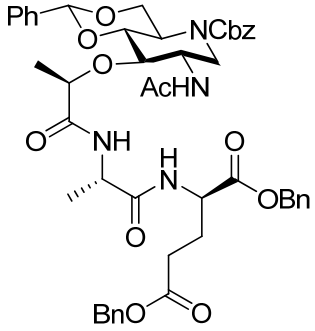
Dibenzyl *N*-(L-alanyl)-D-glutamate trifluoroacetate (25)



Trifluoroacetic acid (0.26 mL, 3.4 mmol) was slowly added to a solution of **24** (170 mg, 0.34 mmol) in CH_2Cl_2 (10 mL), and the mixture was stirred at room temperature for 40 h. The solvent and extra reagent were removed under vacuum to afford the title compound (174 mg, quant.) as a colorless oil. ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.47 (d, *J* = 6.6 Hz, 3H, CHCH_3), 2.01 (m, 1H, CHCH_2CH$_2$), 2.16 (m, 1H, CHCH_2CH$_2$), 2.38 (t, *J* = 7.1 Hz, 2H, CHCH$_2$CH_2), 4.27 (br s, 1H, C*H*CH$_3$), 4.65 (dt, *J* = 5.6, 7.5 Hz, 1H, C*H*CH$_2$CH$_2$), 5.04 and 5.07 (AB, *J* = 12.2 Hz, 2H, CH_2Ph), 5.06 and 5.12 (AB, *J* = 12.1 Hz, 2H, CH_2Ph), 7.24−7.35 (m, 10H), 7.84 (d, *J* = 7.4 Hz, 1H, N*H*CHCH$_2$), 7.89 (br s, 2H); ^{13}C NMR (126

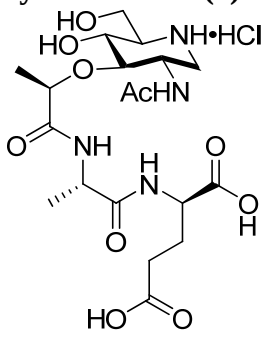
MHz, $CDCl_3$) δ 17.2 (CH*C*H_3), 26.6 (CH*C*H_2CH$_2$), 30.2 (CHCH$_2$*C*H$_2$), 49.9 (*C*HCH$_3$), 52.3 (*C*HCH$_2$CH$_2$), 67.1 and 68.0 (*C*H_2Ph), 128.4, 128.5, 128.6, 128.8, 128.9, 134.9, 135.6, 170.4 (*C*=O), 171.6 (*C*=O), 173.1 (*C*=O); HRMS (ESI) calcd for $C_{22}H_{27}N_2O_5$ (M + H^+) 399.1914, found 399.1888.

Dibenzyl *N*-{2-*O*-[2-acetamido-4,6-*O*-benzylidene-1,5-(benzyloxycarbonyl)imino-1,2,5-trideoxy-D-glucitol-3-yl]-D-lactoyl}-L-alanyl-D-glutamate (19)



Triethylamine (15 μL, 107 μmol) was added to a solution of amine salt **25** (54 mg, 107 μmol) in THF/CH_2Cl_2 (6 mL, 1:1), and the mixture was stirred for 15 min. Compound **17** (60 mg, 97 μmol) was added to the mixture and the mixture was stirred for 40 h. The solvents and extra reagent were removed under reduced pressure, and the resulting residue was washed with AcOEt to afford the title compound (77 mg, 90%) as a white solid. ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.33 (d, *J* = 7.0 Hz, 3H, CHC*H*$_3$), 1.37 (d, *J* = 6.8 Hz, 3H, CHC*H*$_3$), 1.99 (s, 3H, C*H*$_3$CO), 2.04 (m, 1H, CHC*H*$_2$CH$_2$), 2.24 (m, 1H, CHC*H*$_2$CH$_2$), 2.36 (dt, *J* = 7.1, 17.4 Hz, 1H, CHCH$_2$C*H*$_2$), 2.45 (dt, *J* = 7.5, 17.0 Hz, 1H, CHCH$_2$C*H*$_2$), 2.63 (dd, *J* = 10.3, 13.3 Hz, 1H, H-1a), 3.23 (ddd, *J* = 4.6, 10.2, 10.2 Hz, 1H, H-5), 3.52 (dd, *J* = 9.1, 9.1 Hz, 1H, H-3), 3.76 (m, 1H, H-2), 3.79 (dd, *J* = 9.3, 9.3 Hz, 1H, H-4), 4.39−4.49 (m, 3H, H-6a, OC*H*CH$_3$, and NHC*H*CH$_3$), 4.63 (m, 2H, H-1b and C*H*CH$_2$CH$_2$), 4.80 (dd, *J* = 4.4, 11.6 Hz, 1H, H-6b), 5.05−5.19 (m, 6H, NCOOC*H*$_2$Ph, CHCOOC*H*$_2$Ph, and CH$_2$COOC*H*$_2$Ph), 5.56 (s, 1H, C*H*Ph), 6.59 (d, *J* = 7.2 Hz, 1H, CON*H*CHCH$_3$), 7.07 (d, *J* = 7.8 Hz, 1H, CON*H*CHCH$_2$), 7.26–7.48 (m, 20H), 7.69 (d, *J* = 4.0 Hz, 1H, N*H*Ac); ^{13}C NMR (126 MHz, $CDCl_3$) δ 17.7 (CH*C*H$_3$), 19.8 (CH*C*H$_3$), 23.5 (*C*H$_3$CO), 27.1 (CH*C*H$_2$CH$_2$), 30.3 (CHCH$_2$*C*H$_2$), 47.7 (C-1), 49.0 (NH*C*HCH$_3$), 51.4 (C-2), 52.0 (*C*HCH$_2$CH$_2$), 55.2 (C-5), 66.9 (*C*H$_2$Ph), 67.5 (*C*H$_2$Ph), 67.6 (*C*H$_2$Ph), 69.5 (C-6), 76.7 (O*C*HCH$_3$), 80.2 (C-3), 81.9 (C-4), 101.3 (*C*HPh), 126.1, 128.1, 128.3, 128.3, 128.5, 128.6, 128.6, 128.7, 128.8, 128.8, 129.2, 135.2, 135.7, 136.4, 137.7, 154.9 (*C*=O), 171.6 (*C*=O), 171.6 (*C*=O), 171.9 (*C*=O), 172.7 (*C*=O), 174.6 (*C*=O); HRMS (ESI) calcd for $C_{48}H_{54}N_4NaO_{12}$ (M + Na^+) 901.3630, found 901.3602.

***N*-{2-*O*-[2-Acetamido-1,2,5-trideoxy-1,5-imino-D-glucitol-3-yl]-D-lactoyl}-L-alanyl-D-glutamine hydrochloride (6)**

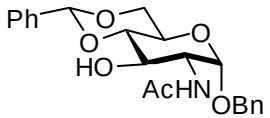


Compound **19** (45 mg, 51 μmol) was dissolved in *i*-PrOH (3 mL), and 10 wt% Pd/C (45 mg) was added to the solution cautiously to avoid ignition. After the addition of conc. HCl (8.5 μL, 102 μmol), the mixture was stirred for 40 h under hydrogen atmosphere and was filtered through a layer of Celite. The Celite pad was washed well with *i*-PrOH, and the filtrate was concentrated under reduced pressure to afford **6** (26 mg, quant.) as a white powder. ^{1}H NMR (500 HMz, D_2O) δ 1.33 (d, *J* = 6.8 Hz, 3H, CHC*H*$_3$), 1.37 (d, *J* = 7.4 Hz, 3H, CHC*H*$_3$), 1.91 (s, 3H, C*H*$_3$CO), 1.95 (m, 1H, CHC*H*$_2$CH$_2$), 2.19 (m, 1H, CHC*H*$_2$CH$_2$), 2.43 (t, *J* = 7.1 Hz, 2H, CHCH$_2$C*H*$_2$), 2.91 (dd, *J* = 12.5, 12.5 Hz, 1H, H-1a), 3.21 (ddd, *J* = 3.5, 4.6, 10.7 Hz, 1H, H-5), 3.46 (dd, *J* = 4.8, 12.8 Hz, 1H, H-1b), 3.53 (dd, *J* = 9.7, 9.7 Hz, 1H, H-3), 3.74 (dd, *J* = 9.1, 10.7 Hz, 1H, H-4), 3.87 (dd, *J* = 4.8, 13.0 Hz, 1H, H-6a), 3.90 (dd, *J* = 3.3, 12.9 Hz, 1H, H-6b), 4.11 (ddd, *J* = 4.9, 10.5, 12.1 Hz, 1H, H-2), 4.26 (q, *J* = 7.2 Hz, 1H, NHC*H*CH$_3$), 4.31 (q, *J* = 6.8 Hz, 1H, OC*H*CH$_3$), 4.41 (dd, *J* = 5.0, 9.6 Hz, 1H, C*H*CH$_2$CH$_2$); ^{13}C NMR (126 MHz, D_2O) δ 16.9 (CH*C*H$_3$), 18.7 (CH*C*H$_3$), 22.2 (*C*H$_3$CO), 25.9 (CH*C*H$_2$CH$_2$), 30.0 (CHCH$_2$*C*H$_2$), 44.1 (C-1), 47.7 (C-2), 49.7 (NH*C*HCH$_3$), 51.9 (*C*HCH$_2$CH$_2$), 57.6 (C-6), 59.9 (C-5), 67.7 (C-4), 78.2 (O*C*HCH$_3$), 81.7 (C-3), 174.3 (*C*=O), 174.7 (*C*=O), 175.0 (*C*=O), 175.5 (*C*=O), 177.0 (*C*=O); HRMS (ESI) calcd for $C_{19}H_{32}N_4NaO_{10}$ (M + Na^+) 499.2011, found 499.2026.

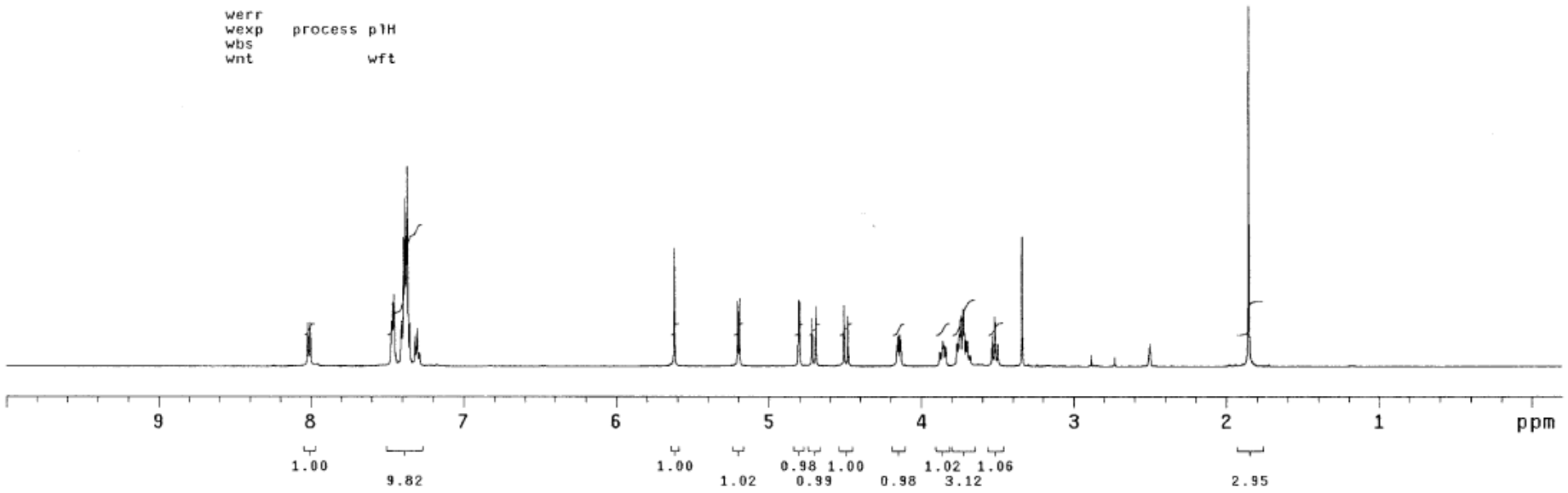
TY2-297

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 22 2009	dfrq	499.866
solvent	DMSO	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.866	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	-99.5	dof3	0
wp	5099.3	dm3	n
vs	29	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	91.80	homo3	n
rfl	1752.7	PROCESSING	
rfp	1249.7	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



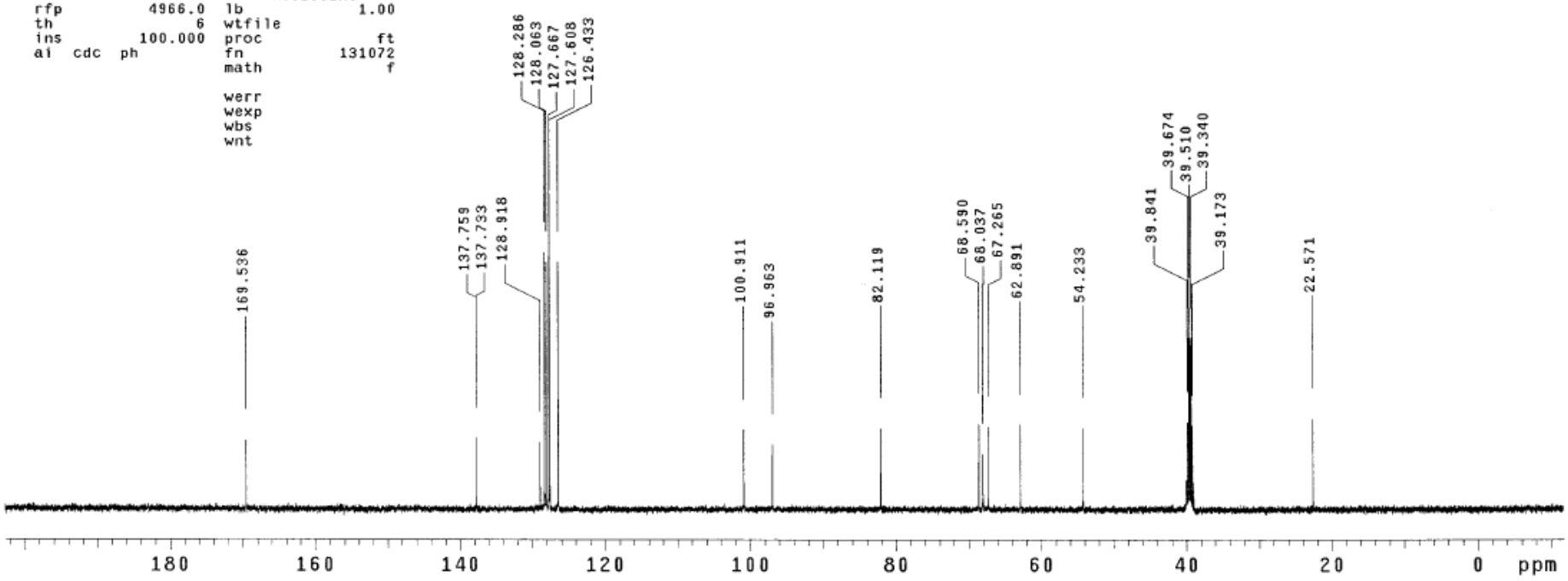
9



TY2-297

exp2 s2pul

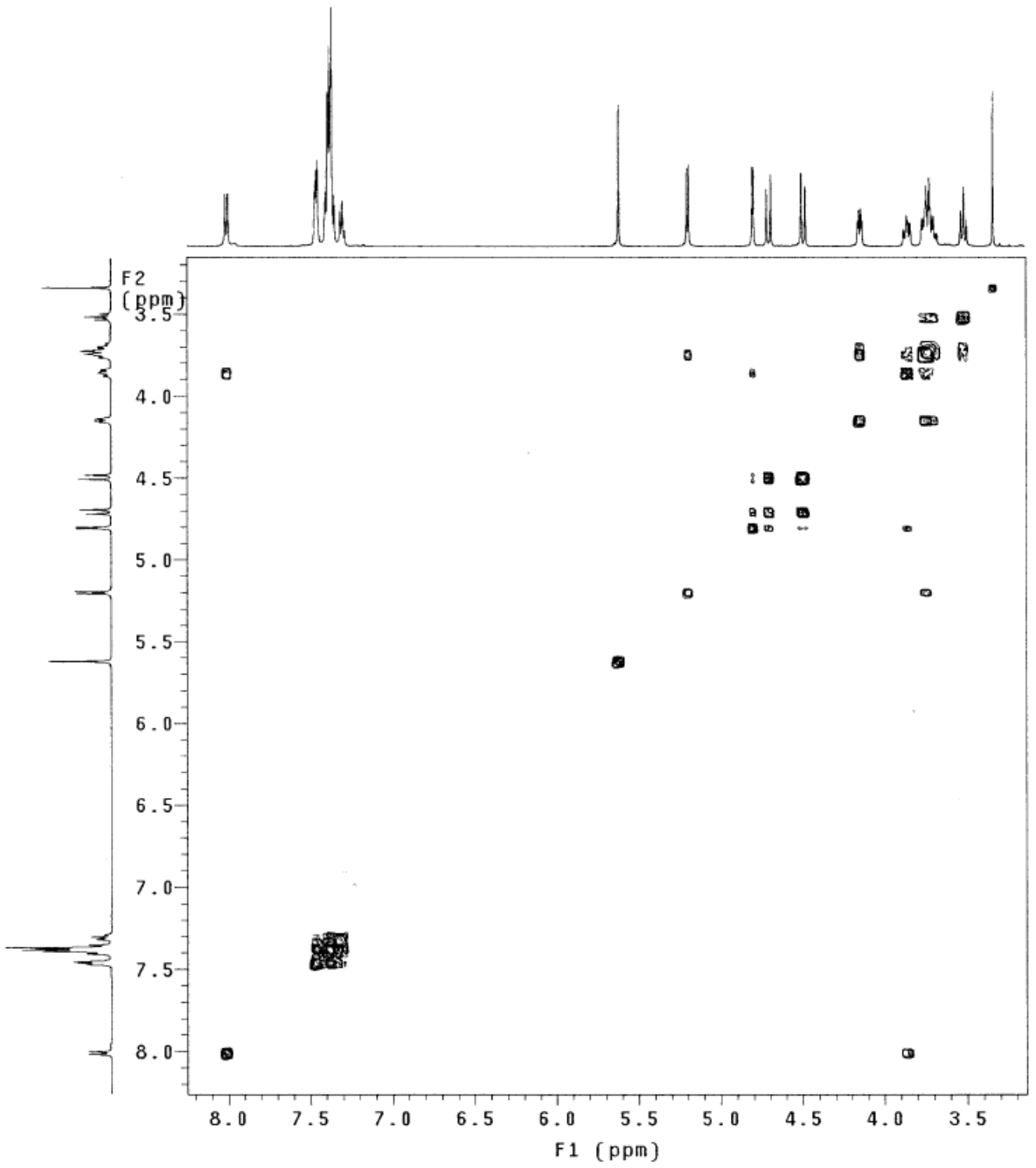
SAMPLE		DEC. & VT	
date	Jun 20 2009	dfrq	499.866
solvent	DMSO	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1000	dm2	n
ct	48	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	-1480.6	dof3	0
wp	26962.9	dm3	n
vs	98	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	6447.1	PROCESSING	
rfp	4966.0	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-297

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

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 32 repetitions
 256 increments
OBSERVE H1, 499.8635420 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

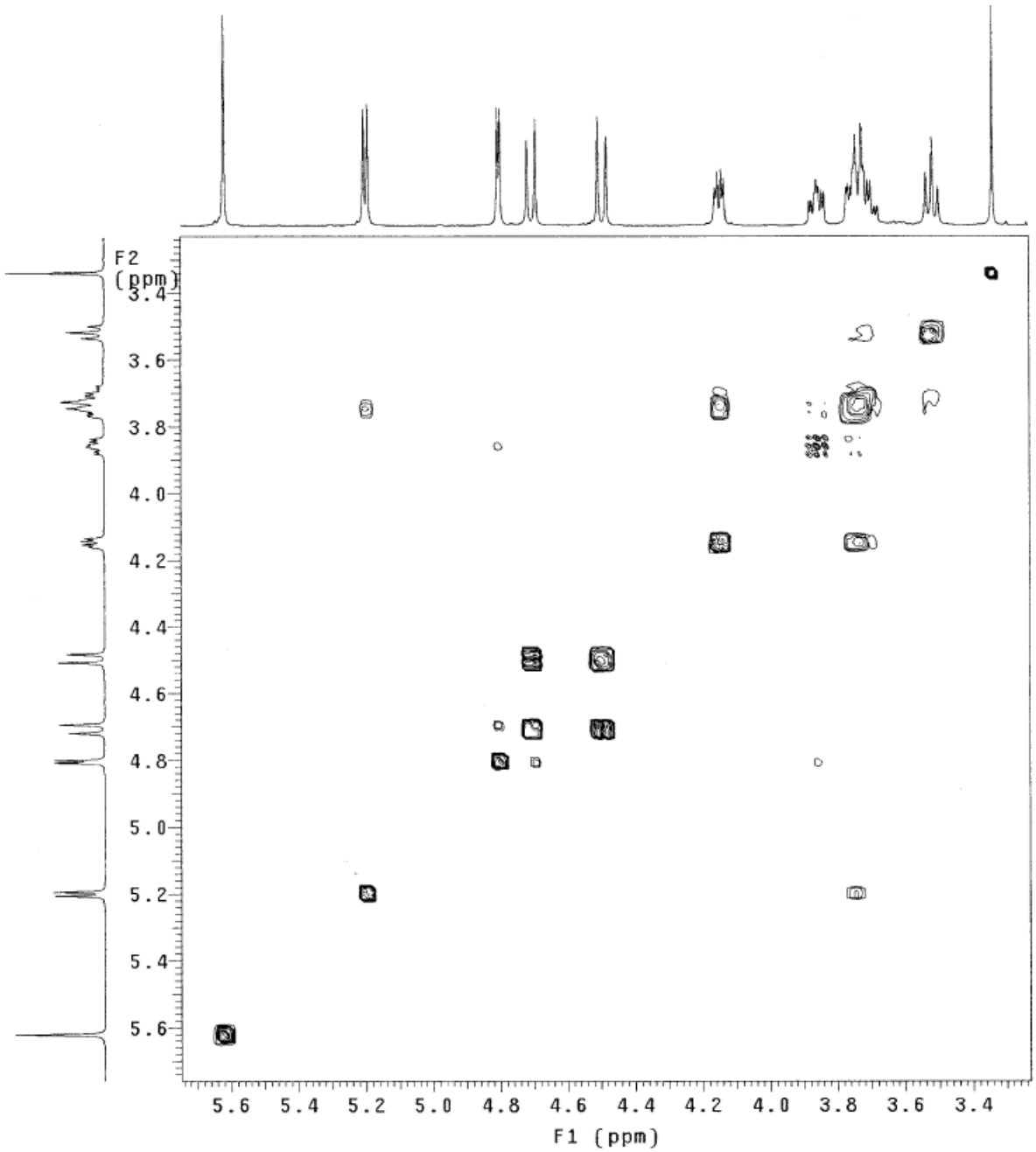


TY2-297

Pulse Sequence: relayh

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

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8635420 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

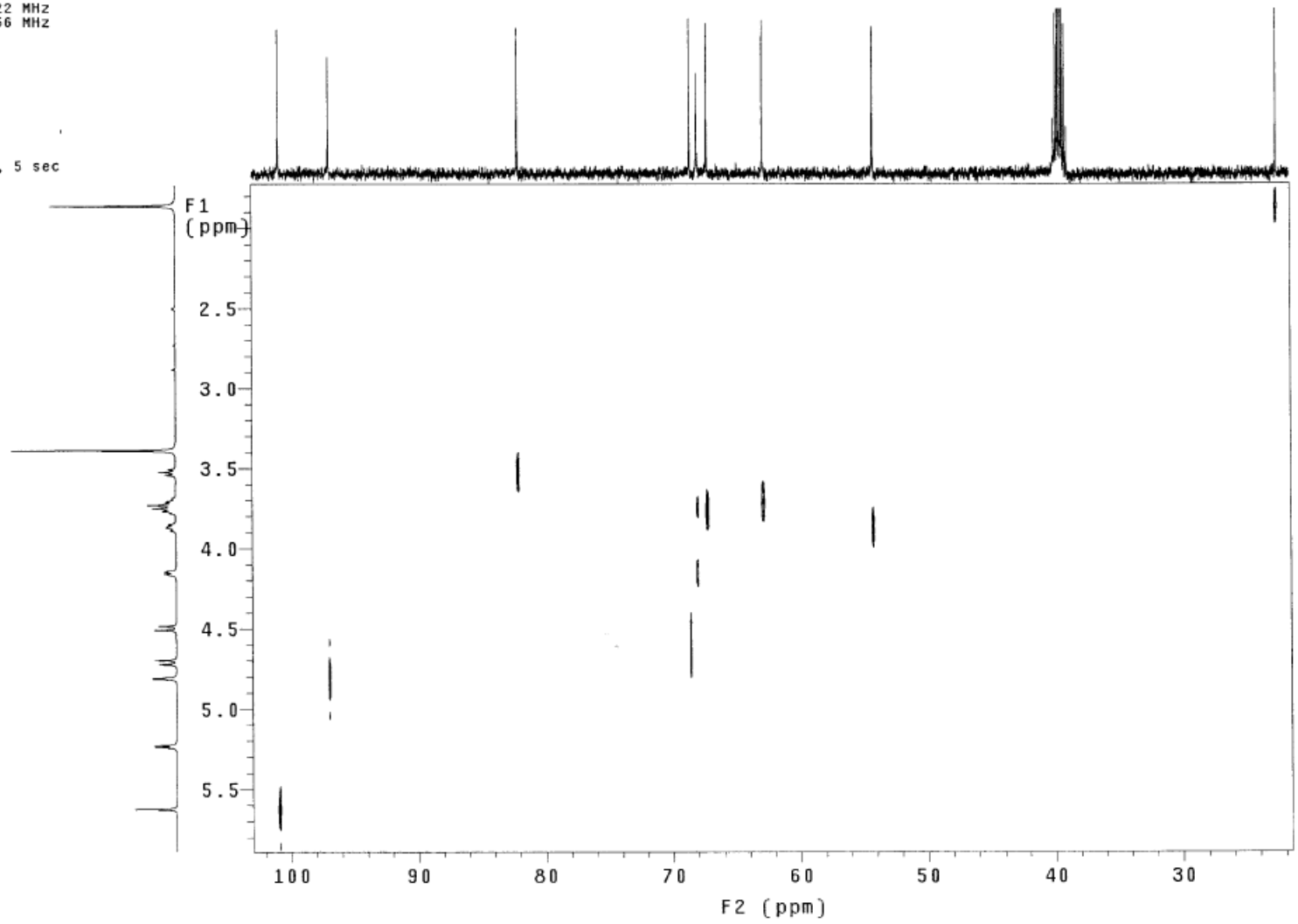


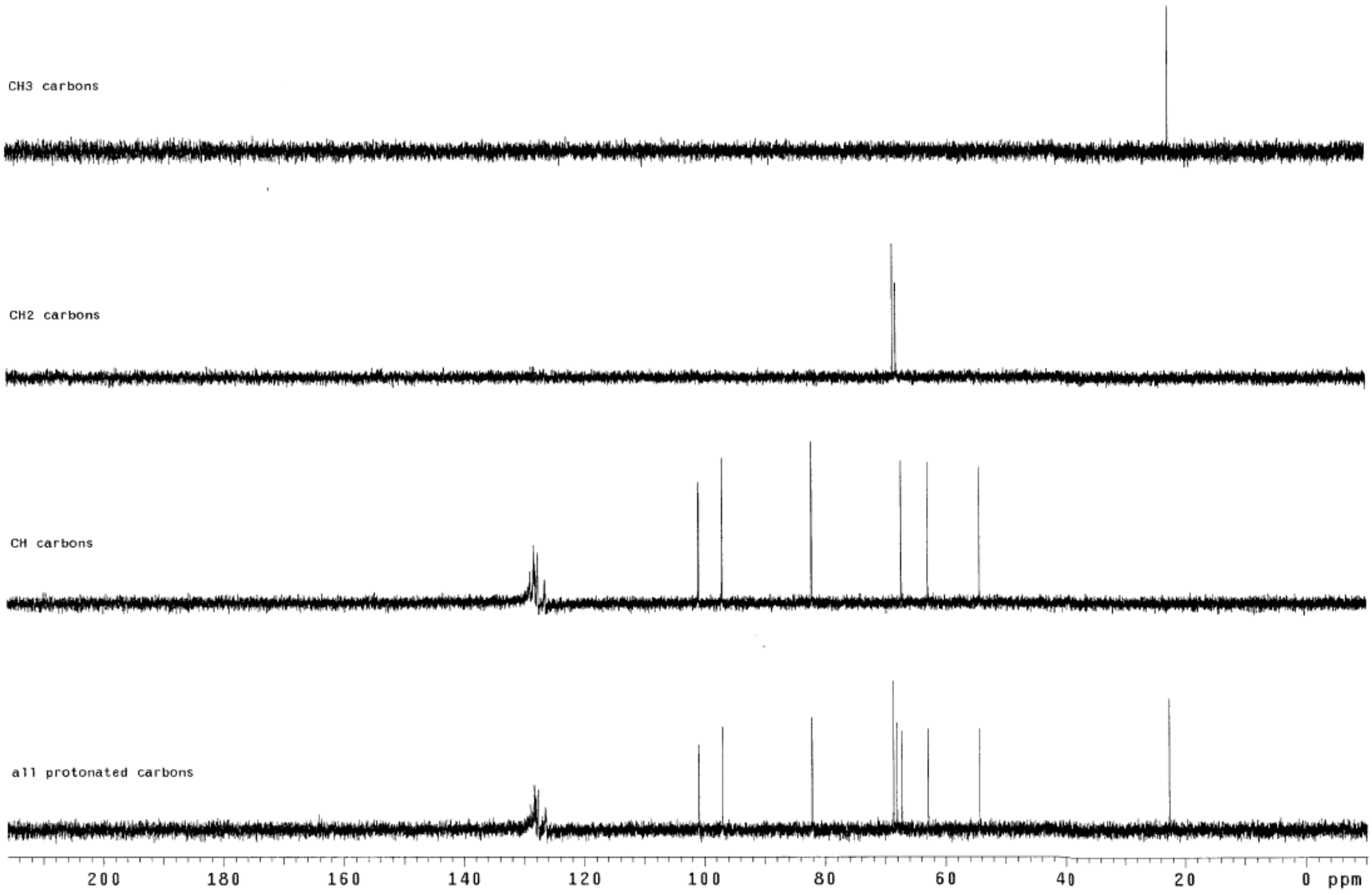
TY2-297

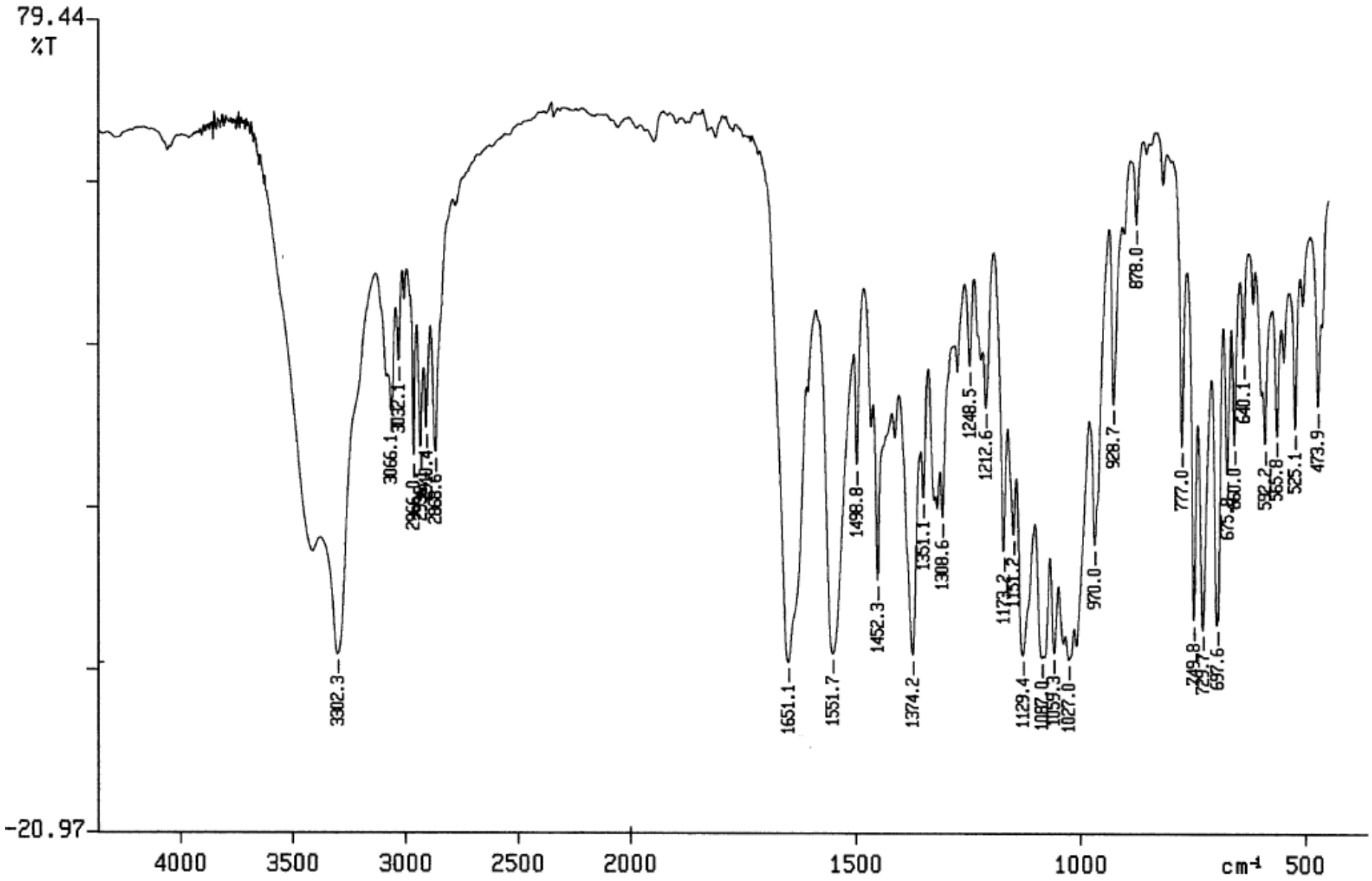
Pulse Sequence: hetcor

Solvent: DMSO
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
32 repetitions
256 increments
OBSERVE C13, 125.6908422 MHz
DECOUPLE H1, 499.8663056 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, 46 min, 5 sec



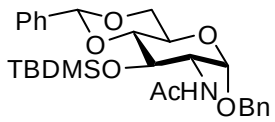

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
0 ppm


79.44
%T
-20.97
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3302.3
3066.1
3032.1
1651.1
1551.7
1498.8
1452.3
1374.2
1351.1
1308.6
1248.5
1212.6
1173.2
1129.4
1087.0
1059.3
1027.0
970.0
928.7
878.0
777.0
749.8
697.6
640.1
565.8
525.1
473.9

TY2-194

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 16 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	4	dm2	n
ct	4	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	-111.4	dof3	0
wp	5116.5	dm3	n
vs	25	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.47	dres3	1.0
is	400.00	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

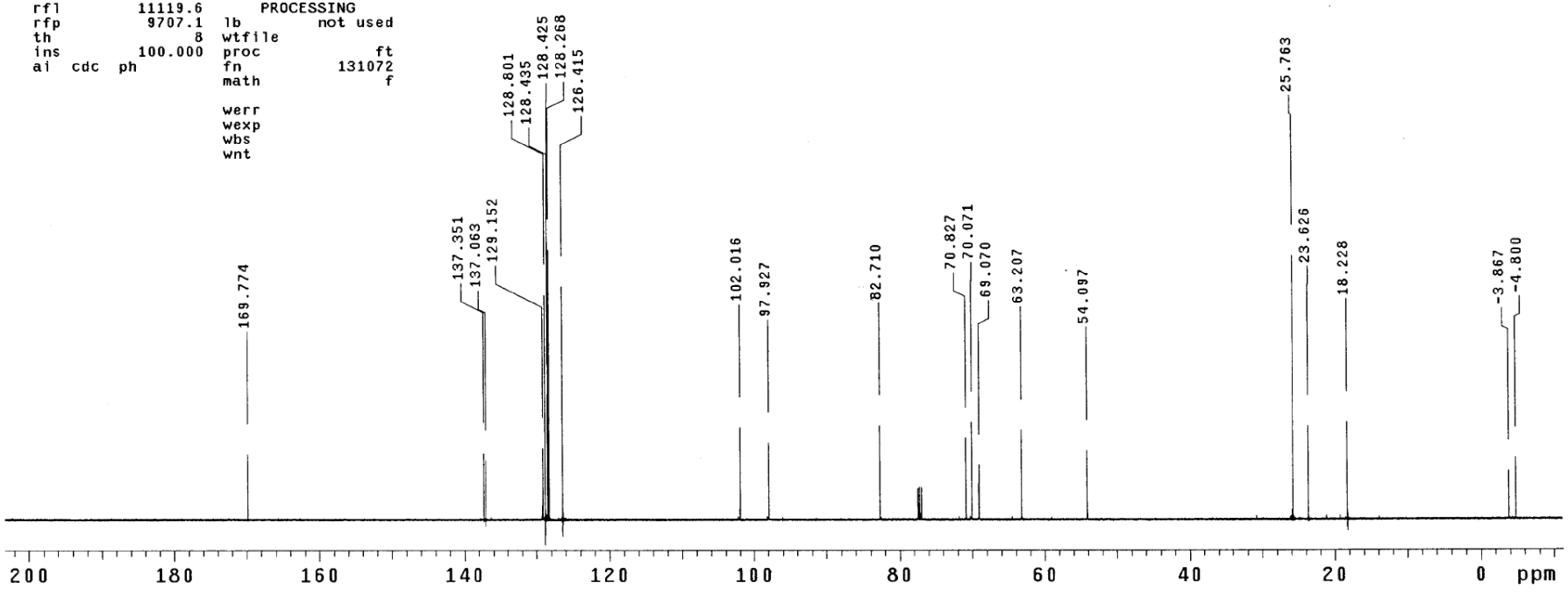


10

TY2-194

exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 16 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	1000	dm2	n
ct	399	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	-1412.2	dof3	0
wp	26962.9	dm3	n
vs	24	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11119.6	PROCESSING	
rfp	9707.1	lb	not used
th	8	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

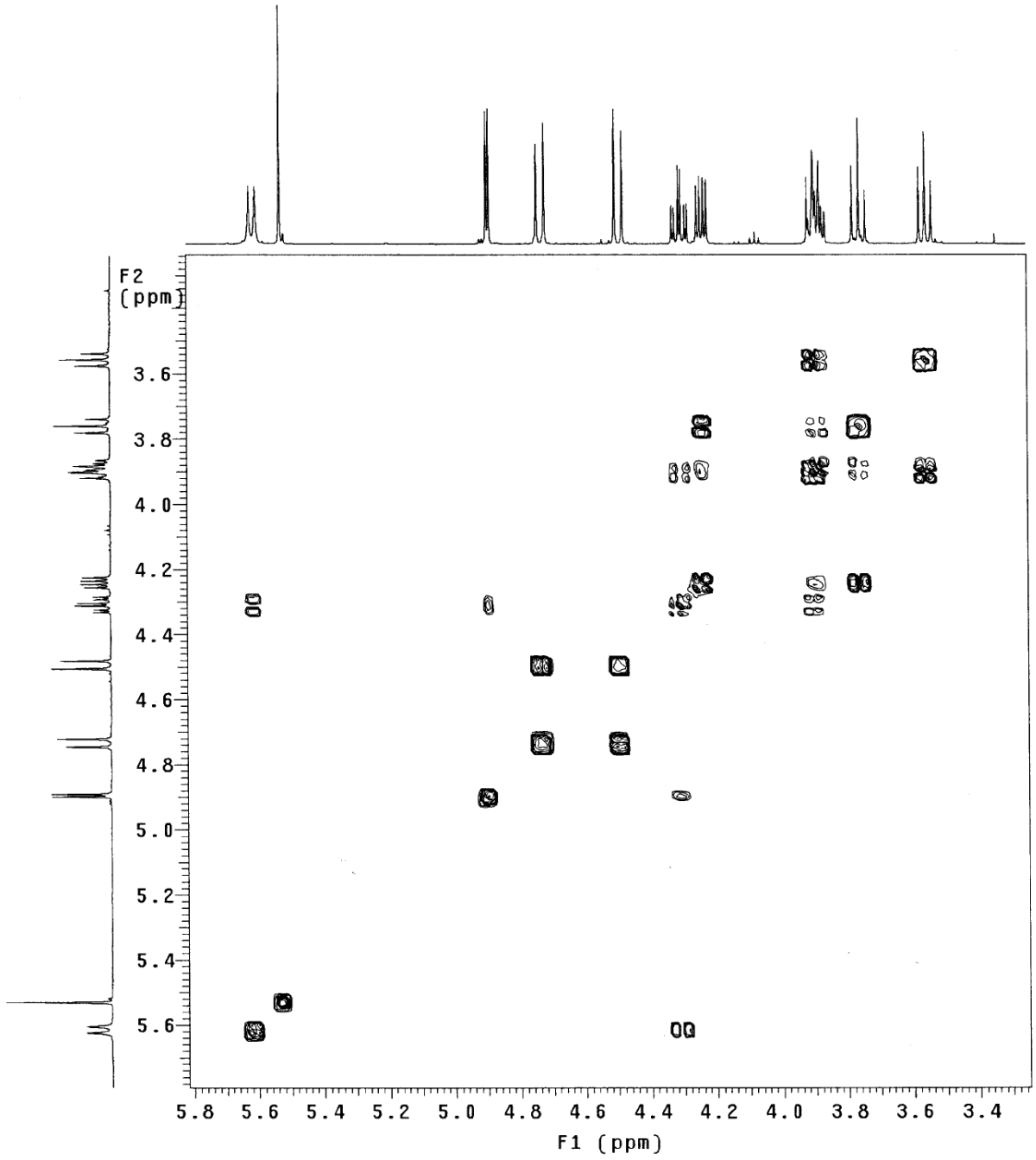


TY2-194

Pulse Sequence: relayh

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

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
16 repetitions
256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

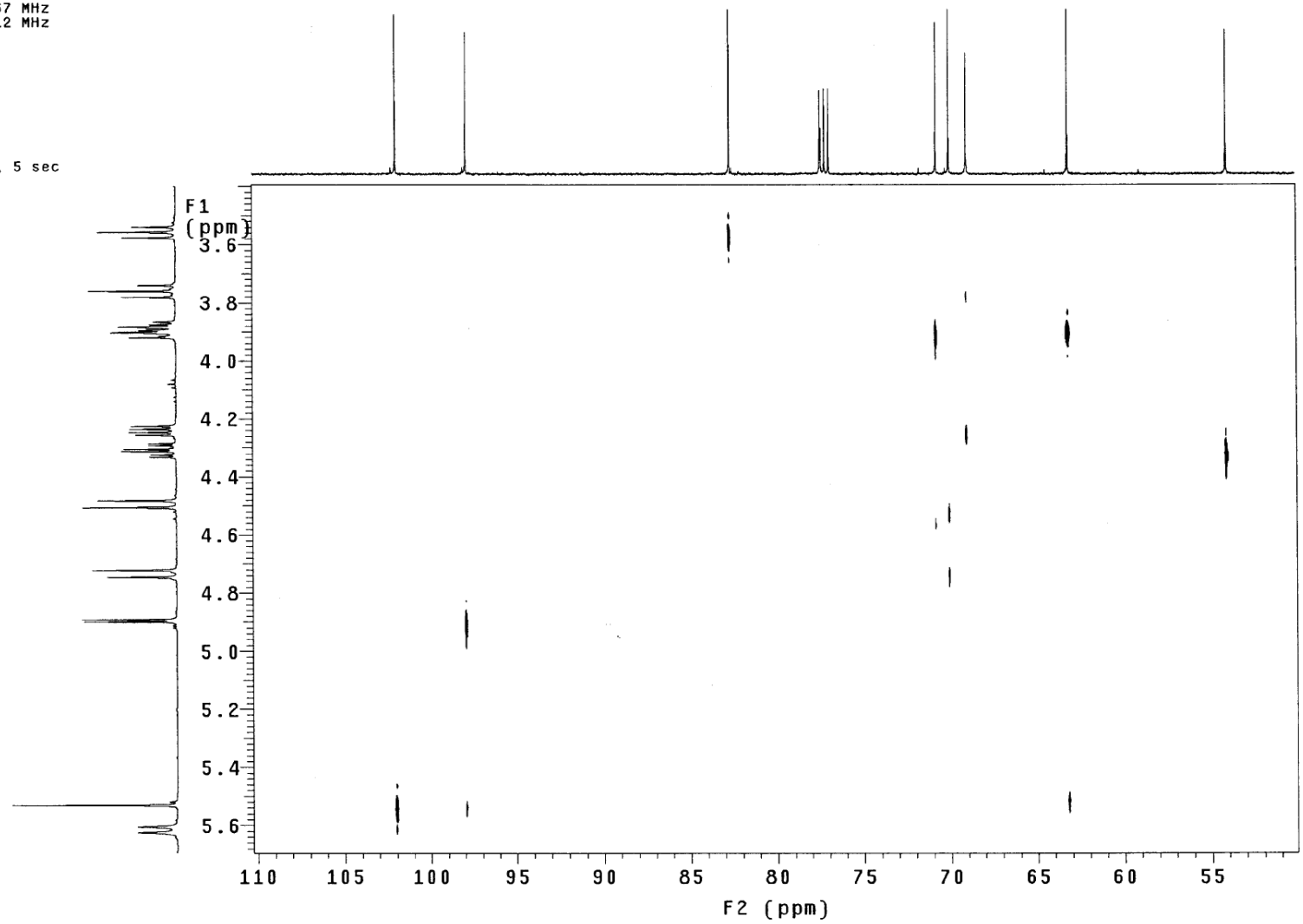


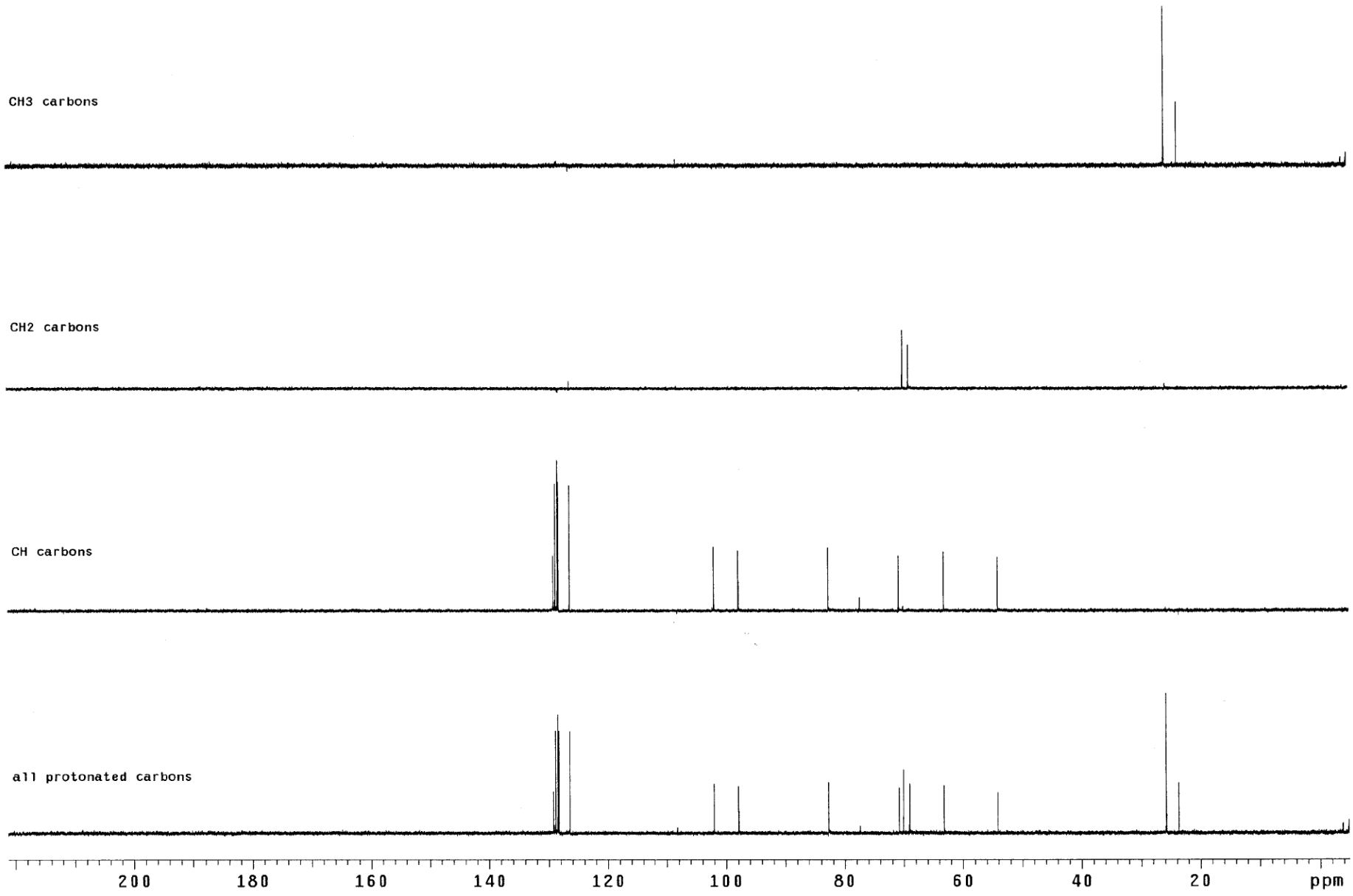
TY2-194

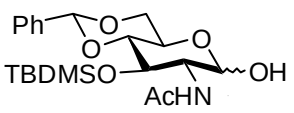
Pulse Sequence: hetcor

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
32 repetitions
256 increments
OBSERVE C13, 125.6901767 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 3 hr, 46 min, 5 sec




CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm



11

TY2-175

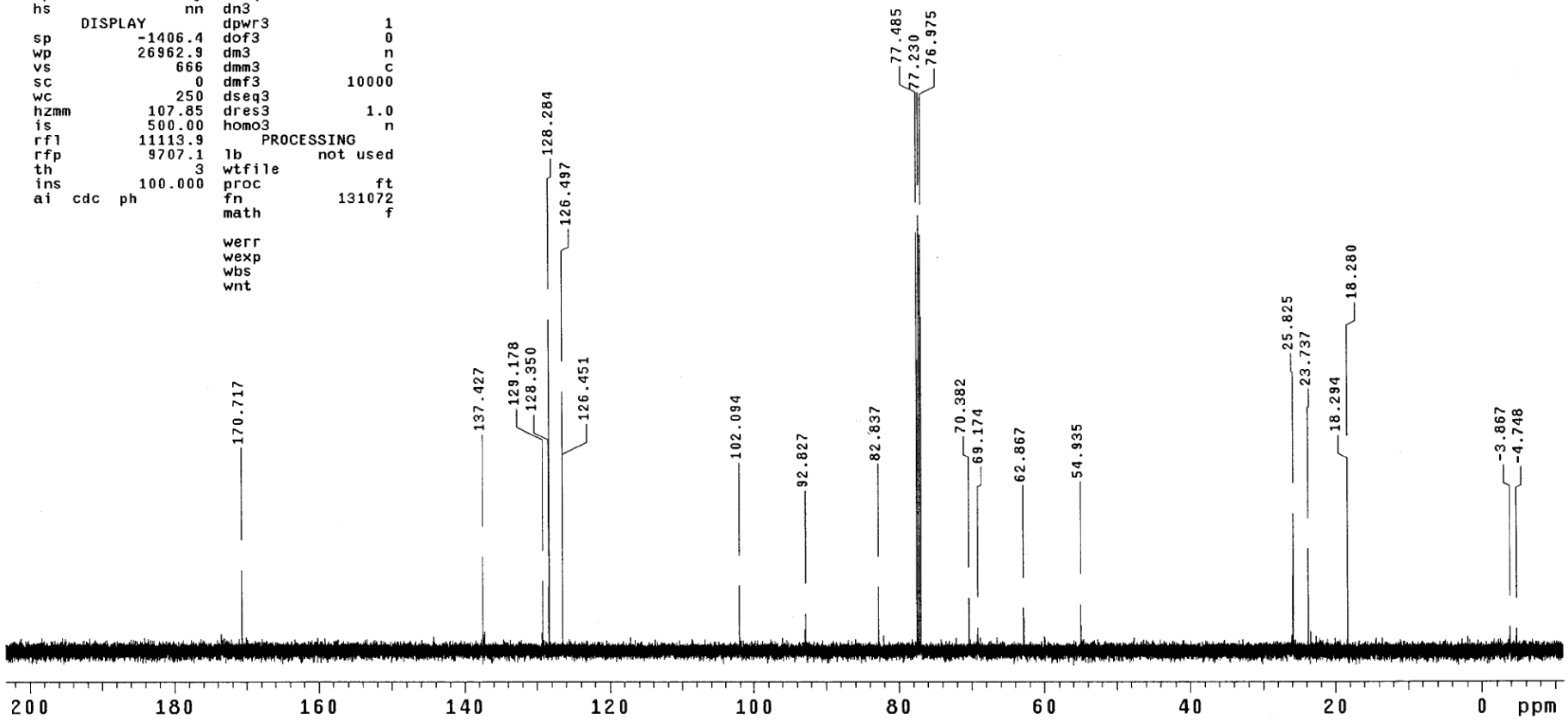
exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 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	-184.7	dof3	0
wp	5185.1	dm3	n
vs	50	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.74	dres3	1.0
is	412.79	homo3	n
rfl	4179.4	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	9.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

TY2-175

exp2 s2pul

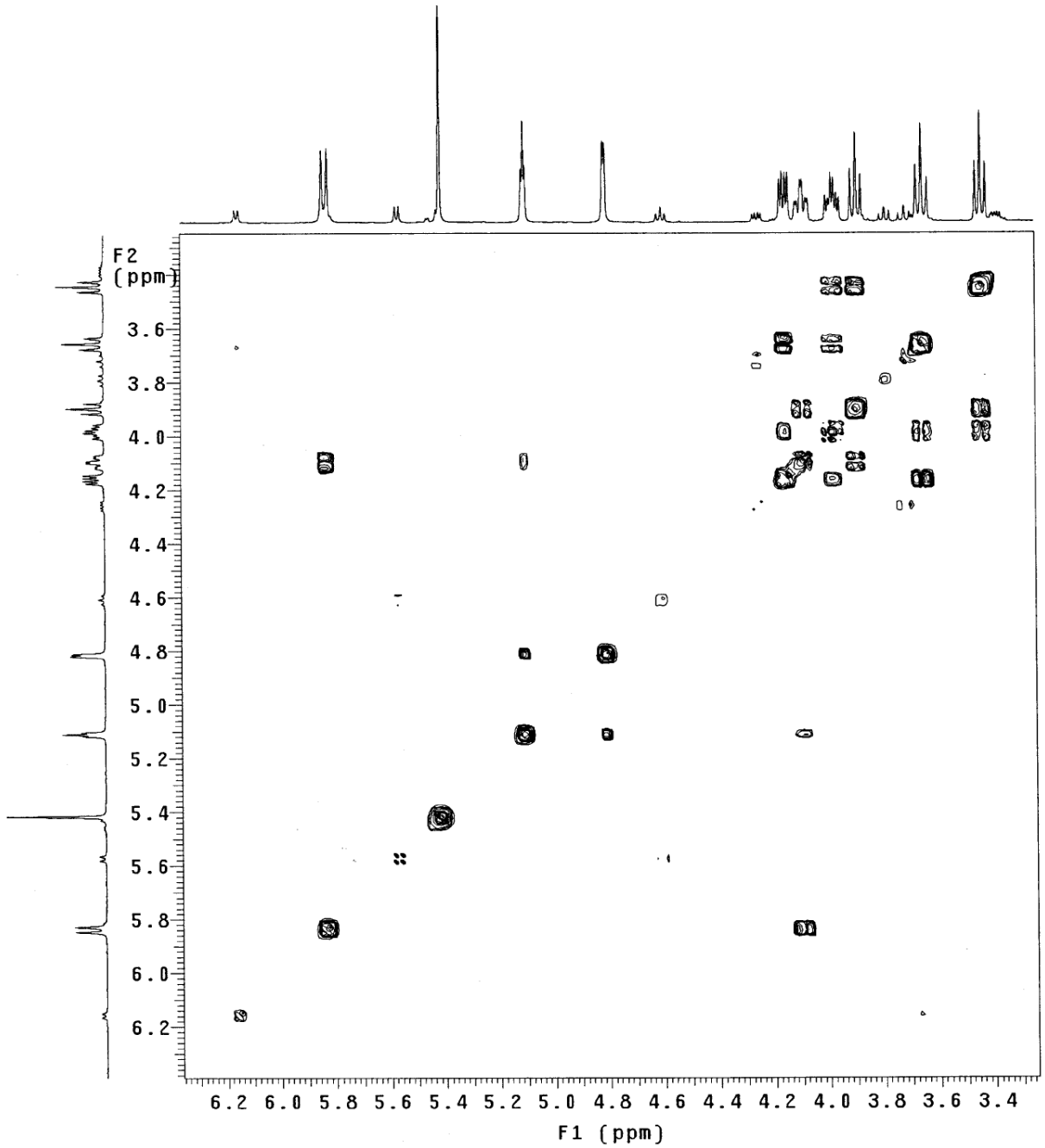
SAMPLE		DEC. & VT	
date	Oct 5 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	3000	dm2	n
ct	1296	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	-1406.4	dof3	0
wp	26962.9	dm3	n
vs	666	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11113.9	PROCESSING	
rfp	9707.1	lb	not used
th	3	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-175

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

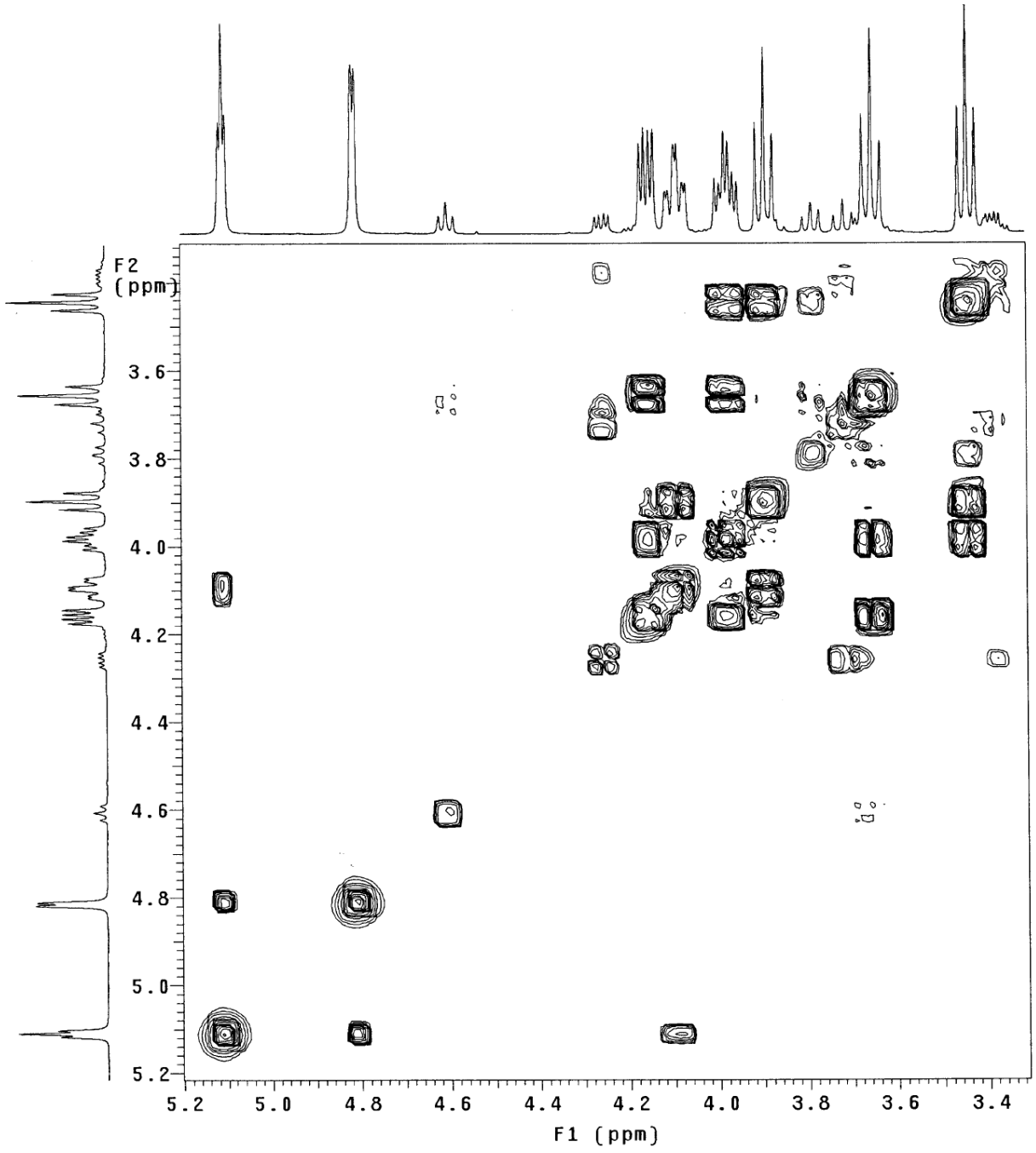
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8612100 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 50 min, 56 sec



TY2-175

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

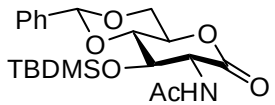
 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 8 repetitions
 256 increments
OBSERVE H1, 499.8612100 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 50 min, 56 sec



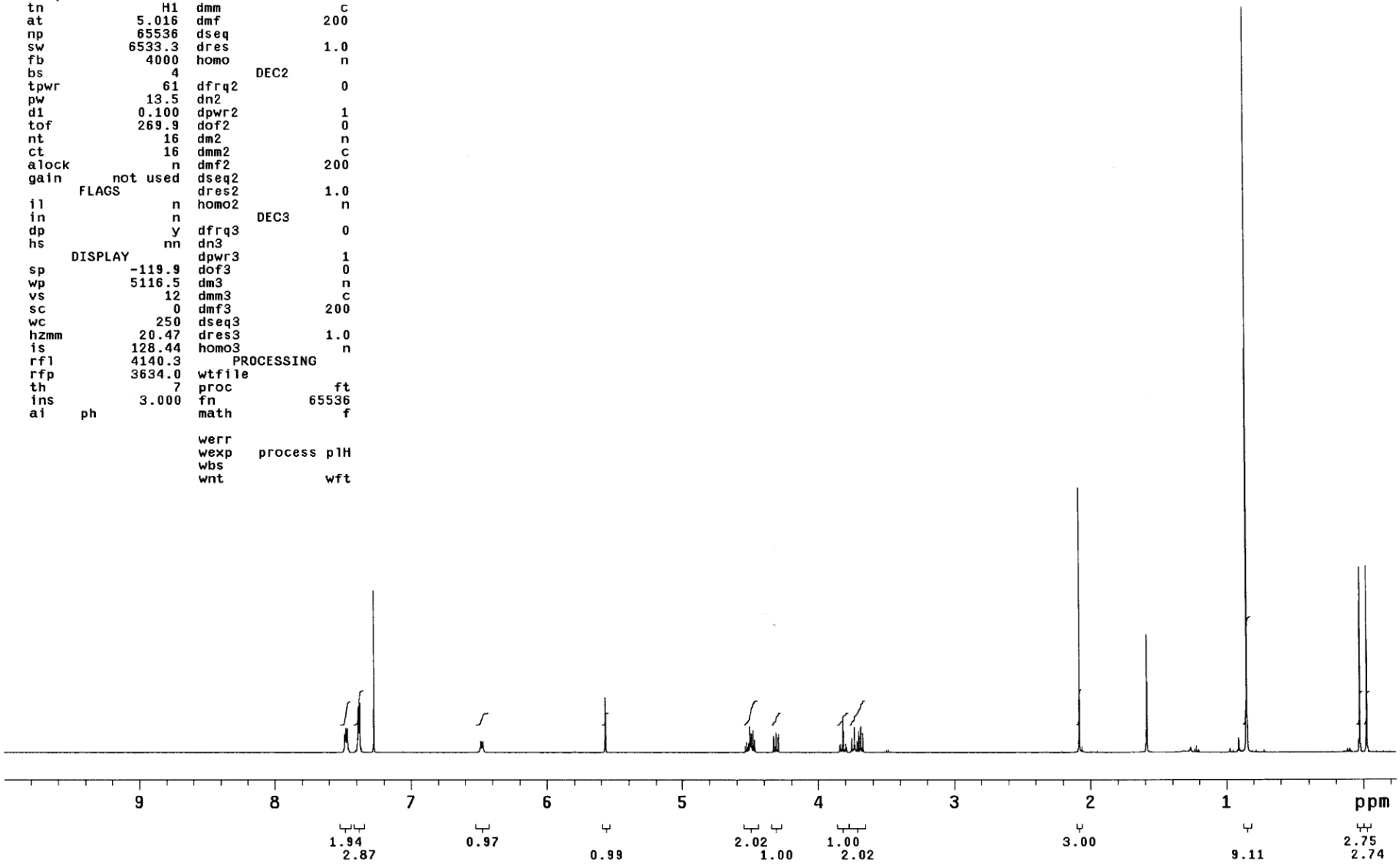
TY2-172PDC

exp1 s2pul

SAMPLE		DEC. & VT	
date	Sep 29 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	-119.9	dof3	0
wp	5116.5	dm3	n
vs	12	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.47	dres3	1.0
is	128.44	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



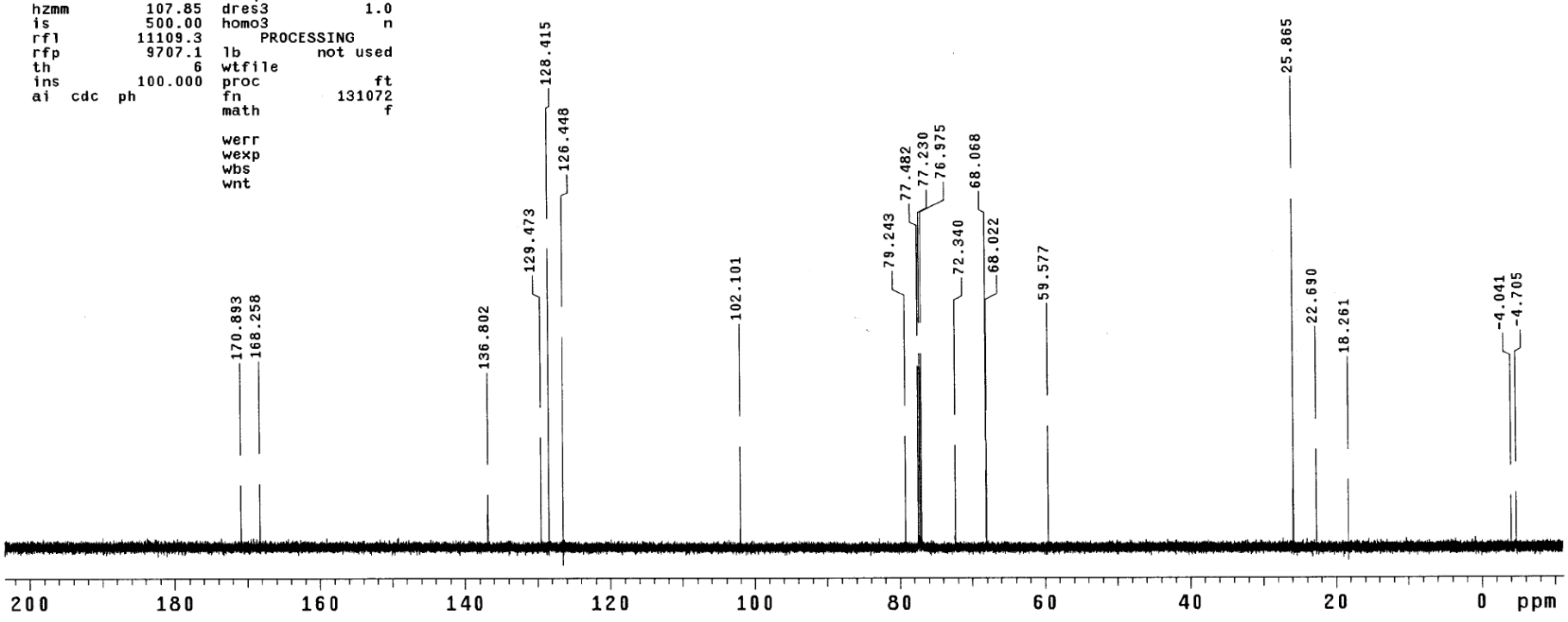
12



TY2-177

exp2 s2pul

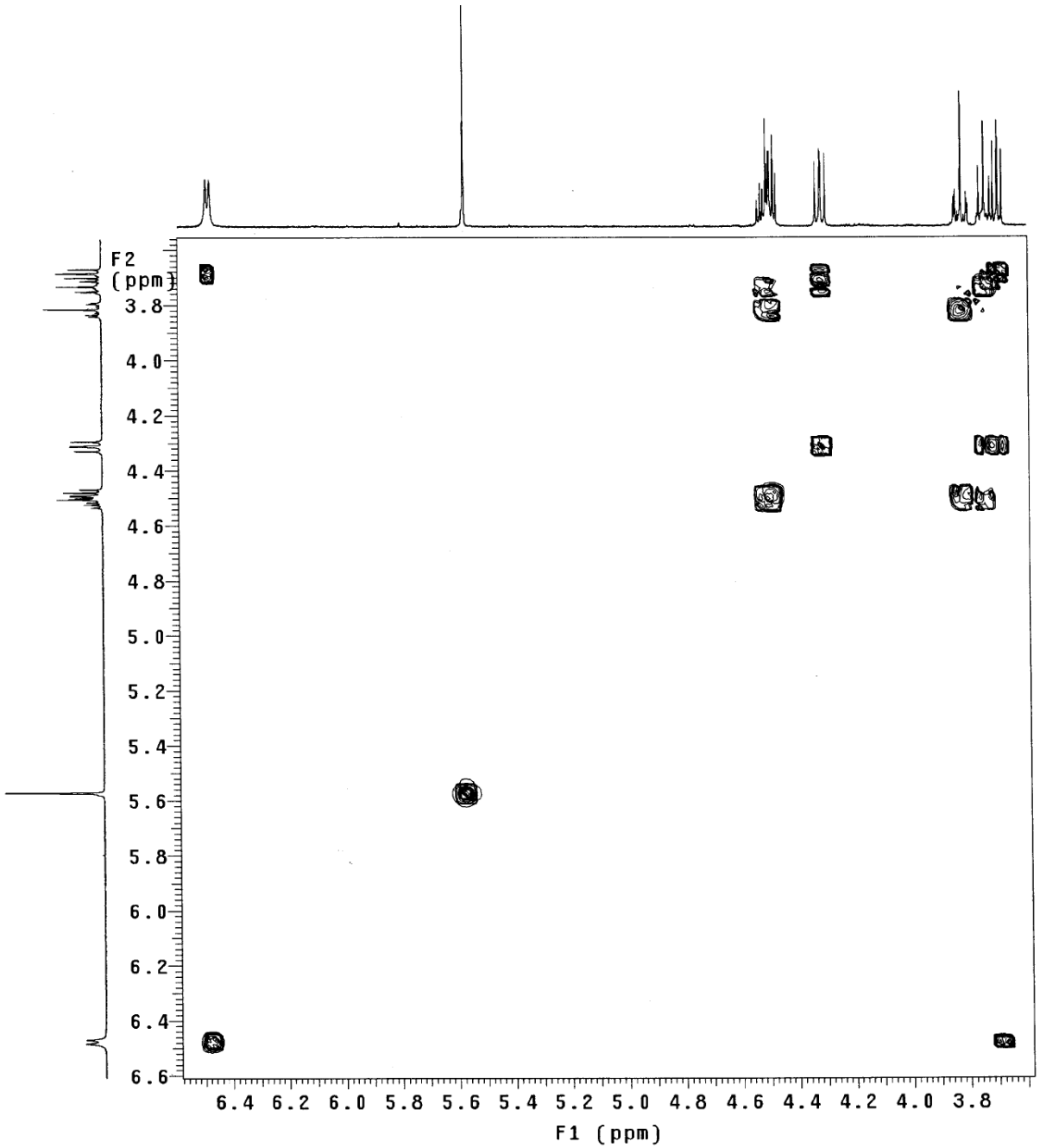
SAMPLE		DEC. & VT	
date	Oct 1 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	2000	dm2	n
ct	421	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	-1401.9	dof3	0
wp	26962.9	dm3	n
vs	160	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11109.3	PROCESSING	
rfp	9707.1	lb	not used
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

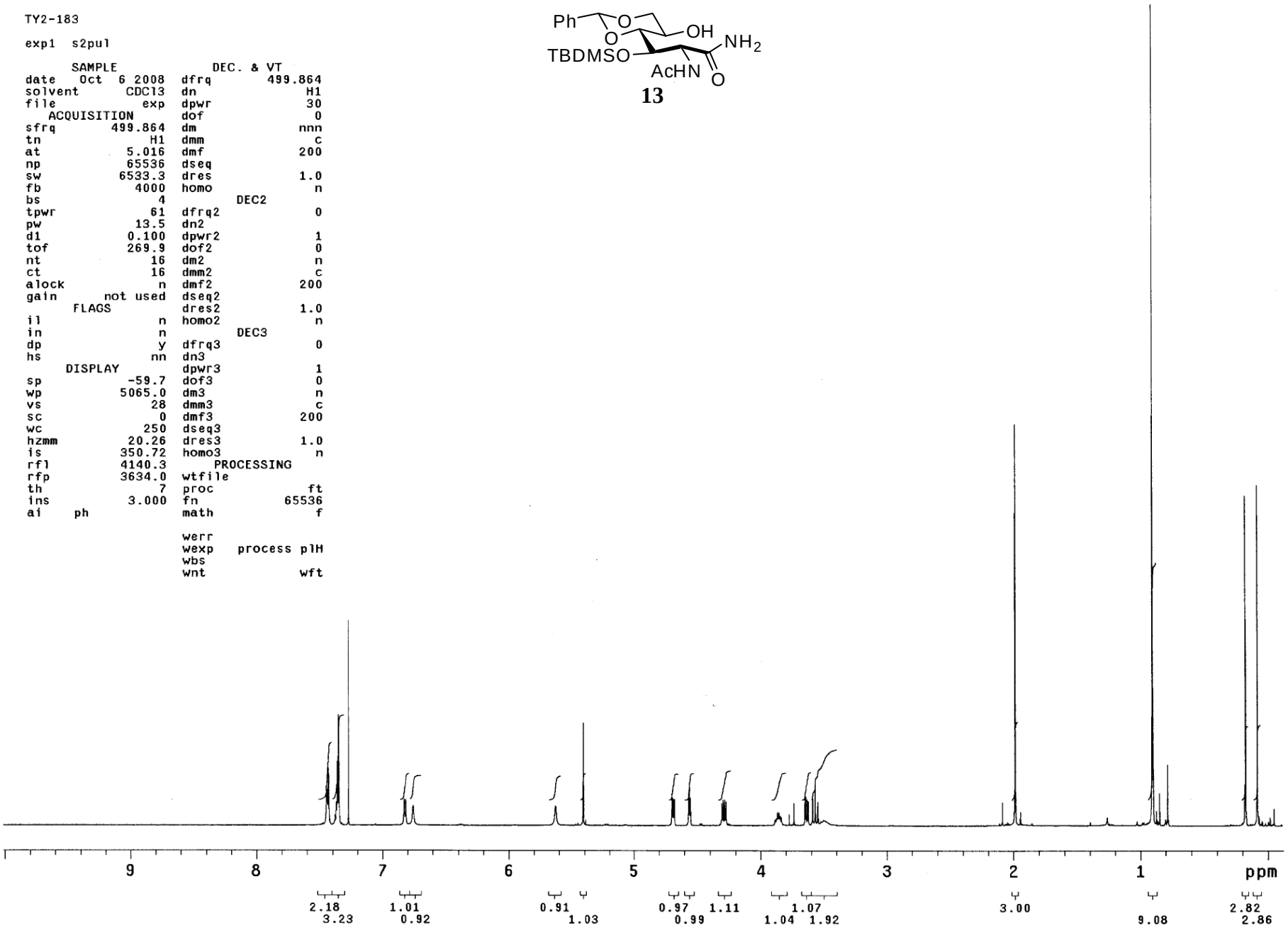


TY2-172

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

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 16 repetitions
 256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

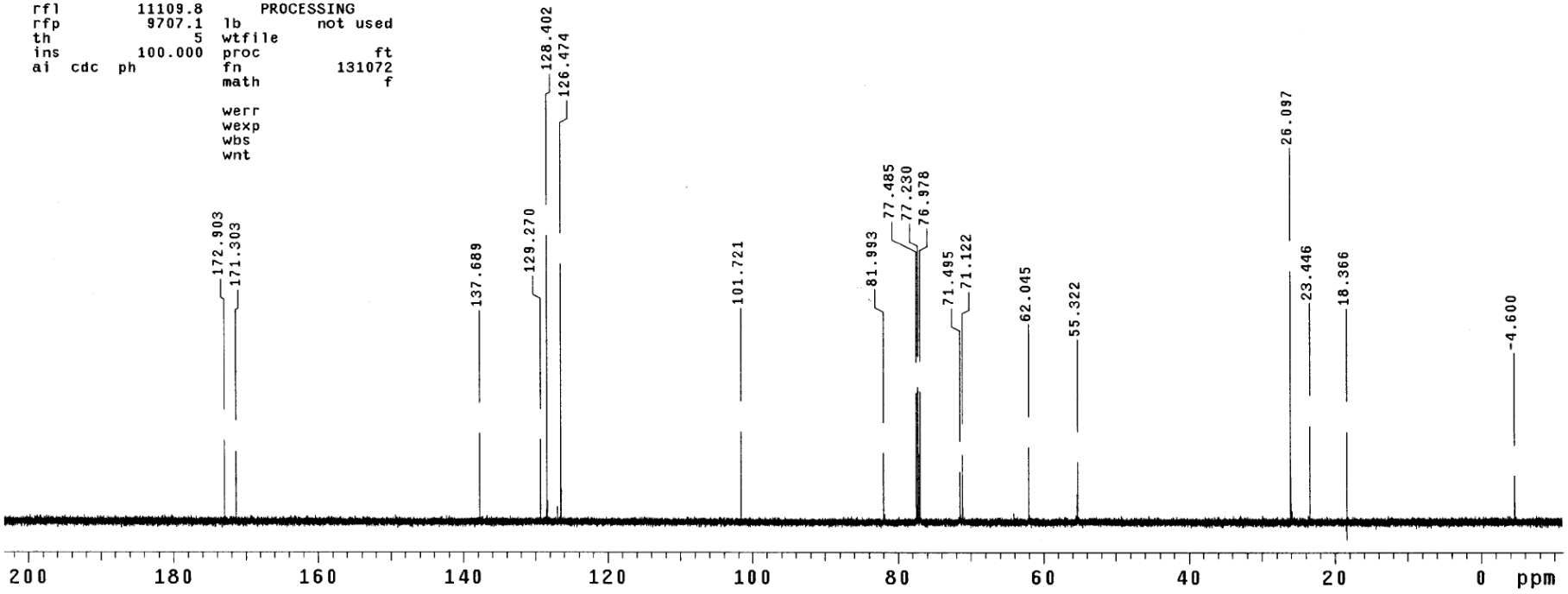



TY2-183
exp1 s2pul
SAMPLE
date Oct 6 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -59.7
wp 5065.0
vs 28
sc 0
wc 250
hzmm 20.26
is 350.72
rfl 4140.3
rfp 3634.0
th 7
ins 3.000
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
Ph
O
O
OH
NH2
TBDMSO
AcHN
O
13
9
8
7
6
5
4
3
2
1
ppm
2.18
3.23
1.01
0.92
0.91
1.03
0.97
0.99
1.11
1.07
1.04
1.92
3.00
9.08
2.82
2.86

TY2-183

exp3 s2pul

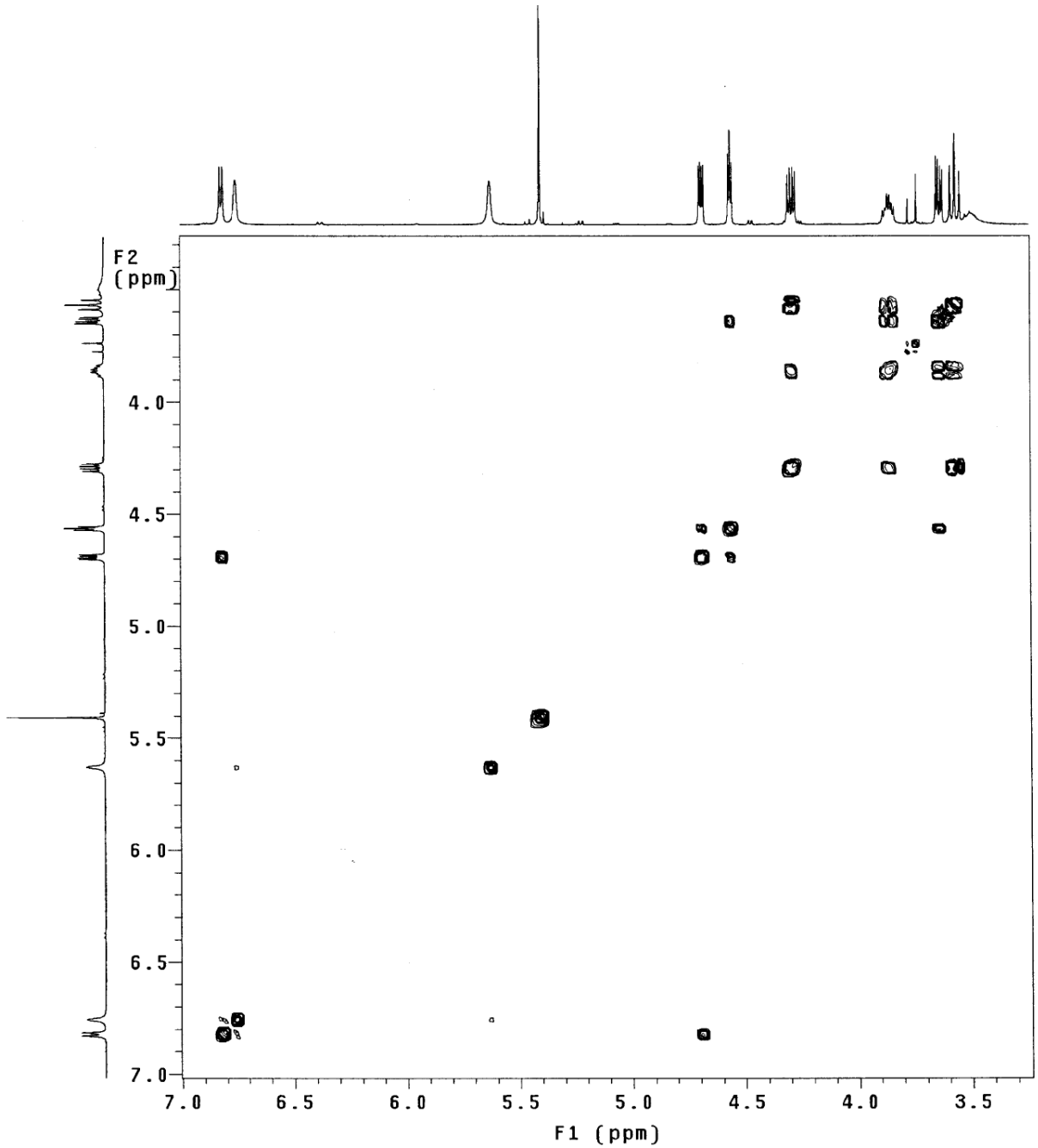
SAMPLE		DEC. & VT	
date	Oct 6 2008	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	2000	dm2	n
ct	331	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	-1402.3	dof3	0
wp	26962.9	dm3	n
vs	124	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11109.8	PROCESSING	
rfp	9707.1	lb	not used
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-183

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

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 8 repetitions
 256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 50 min, 56 sec

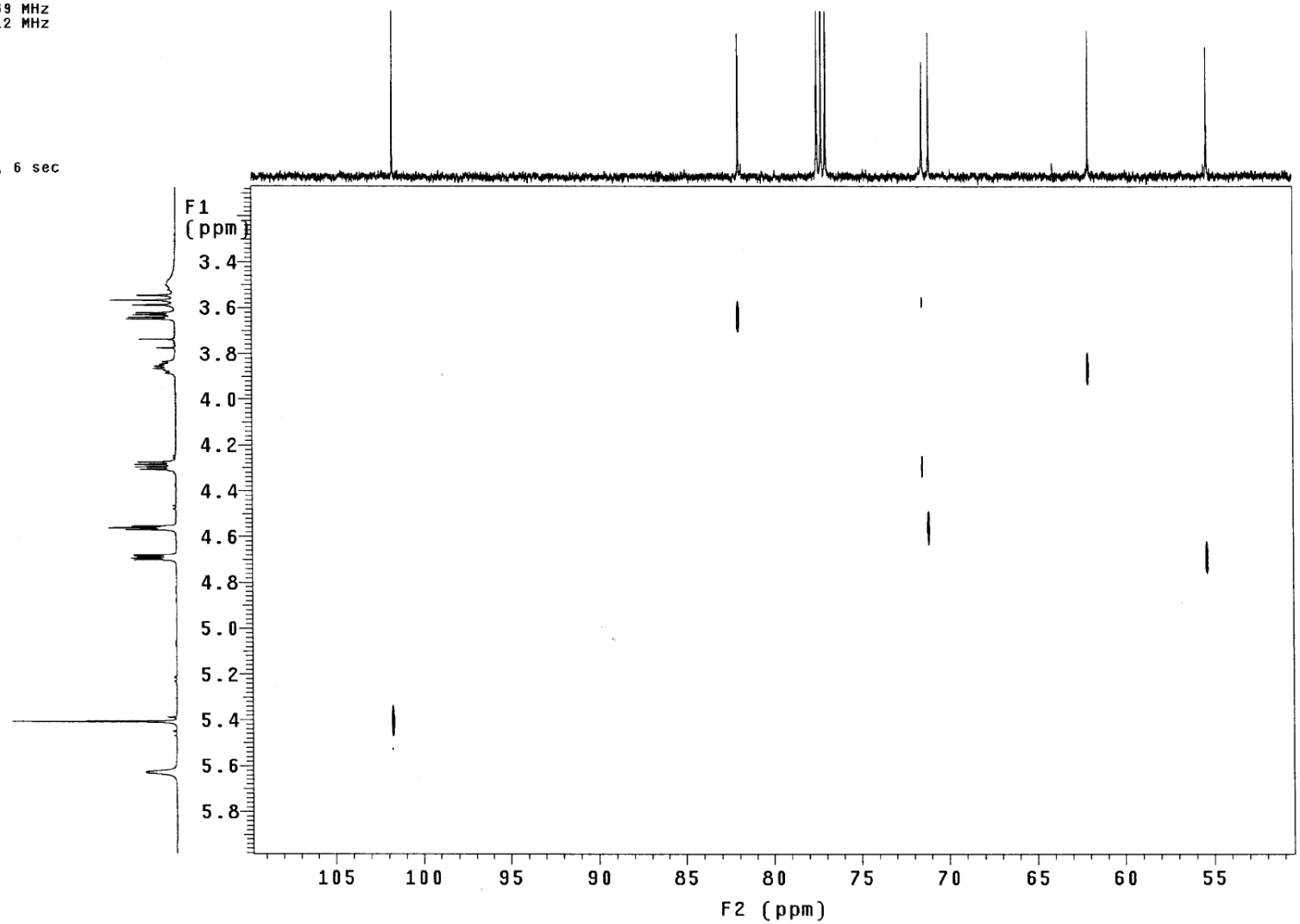


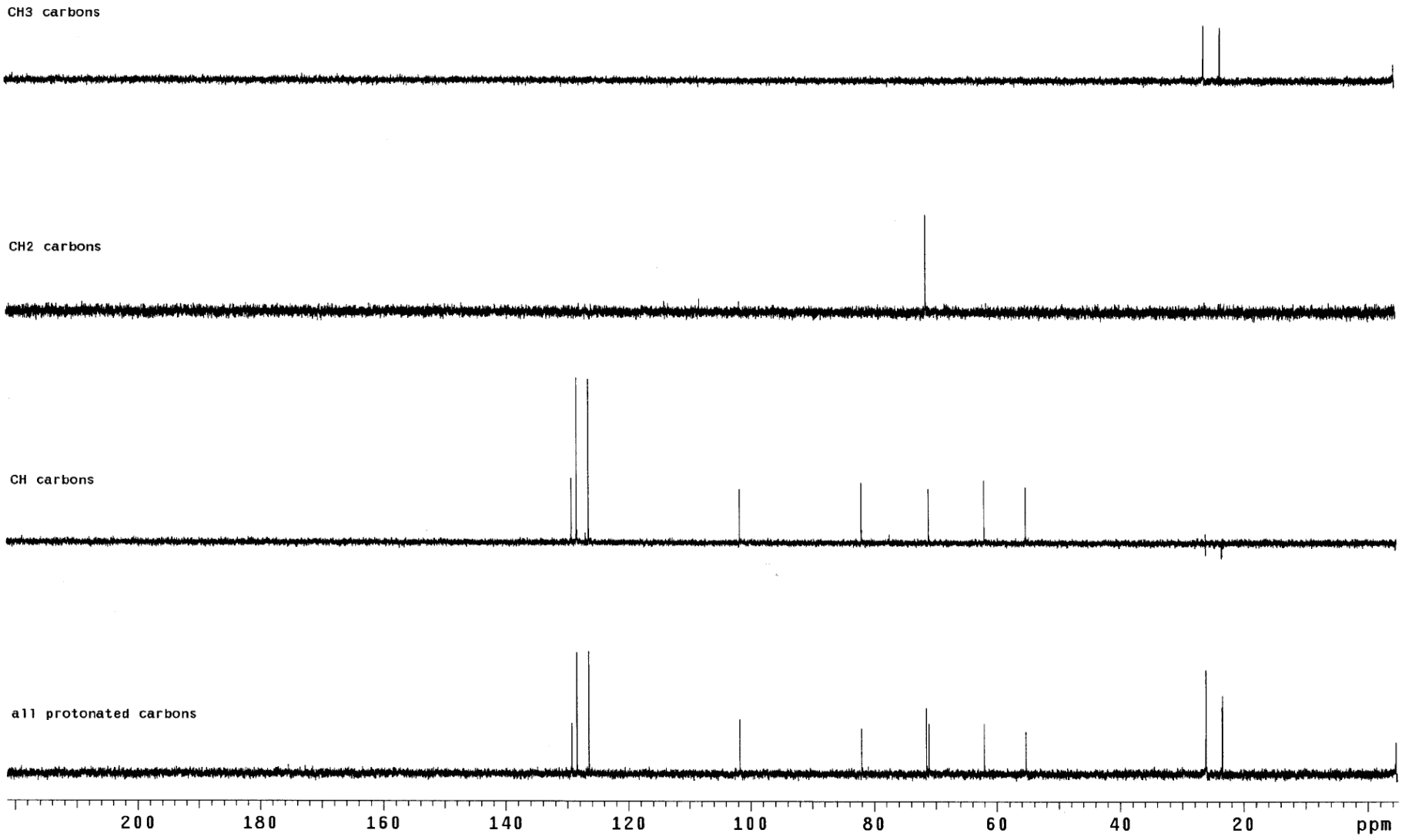
TY2-183

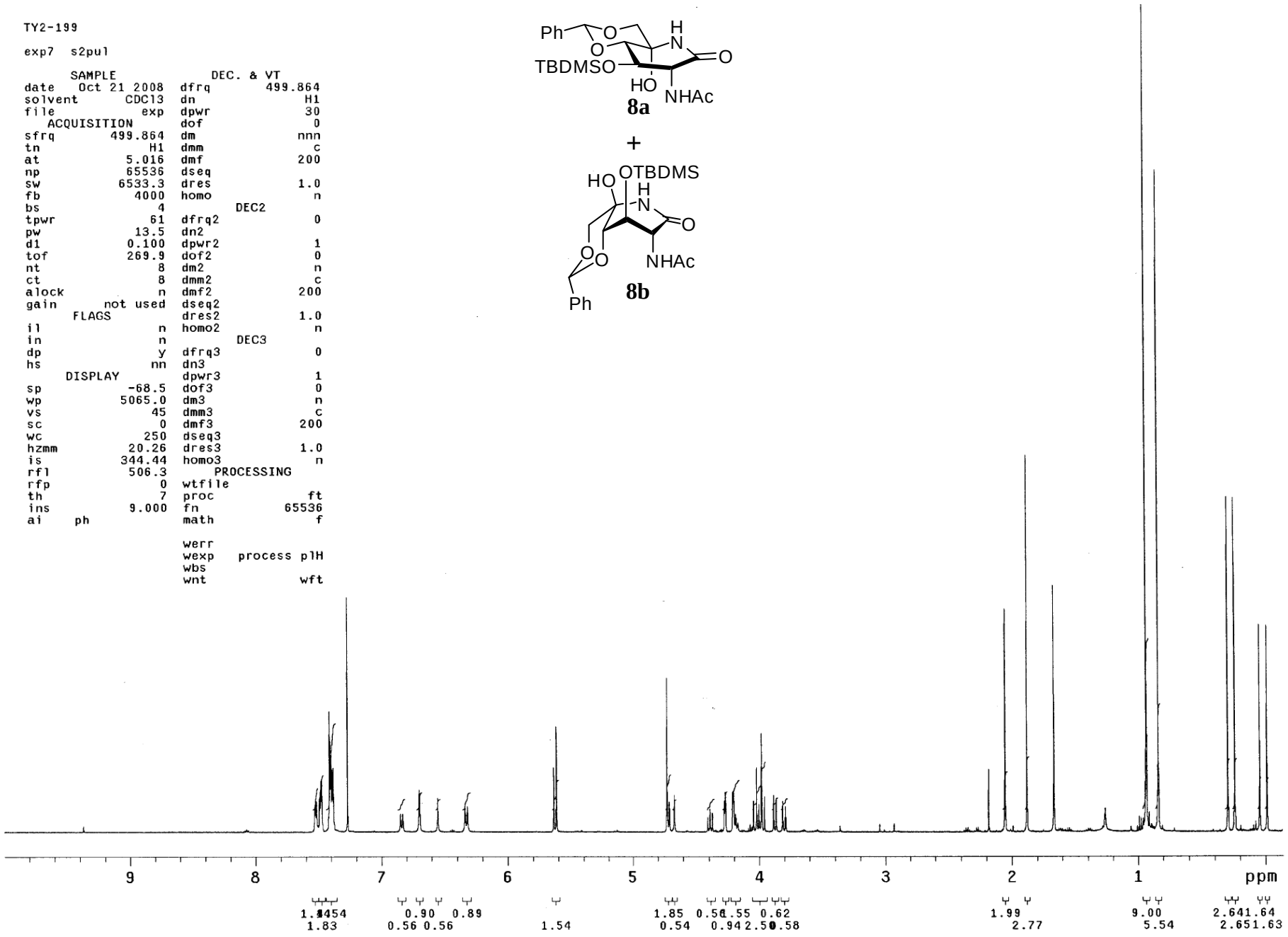
Pulse Sequence: hetcor

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
16 repetitions
256 increments
OBSERVE C13, 125.6901669 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 1 hr, 53 min, 6 sec



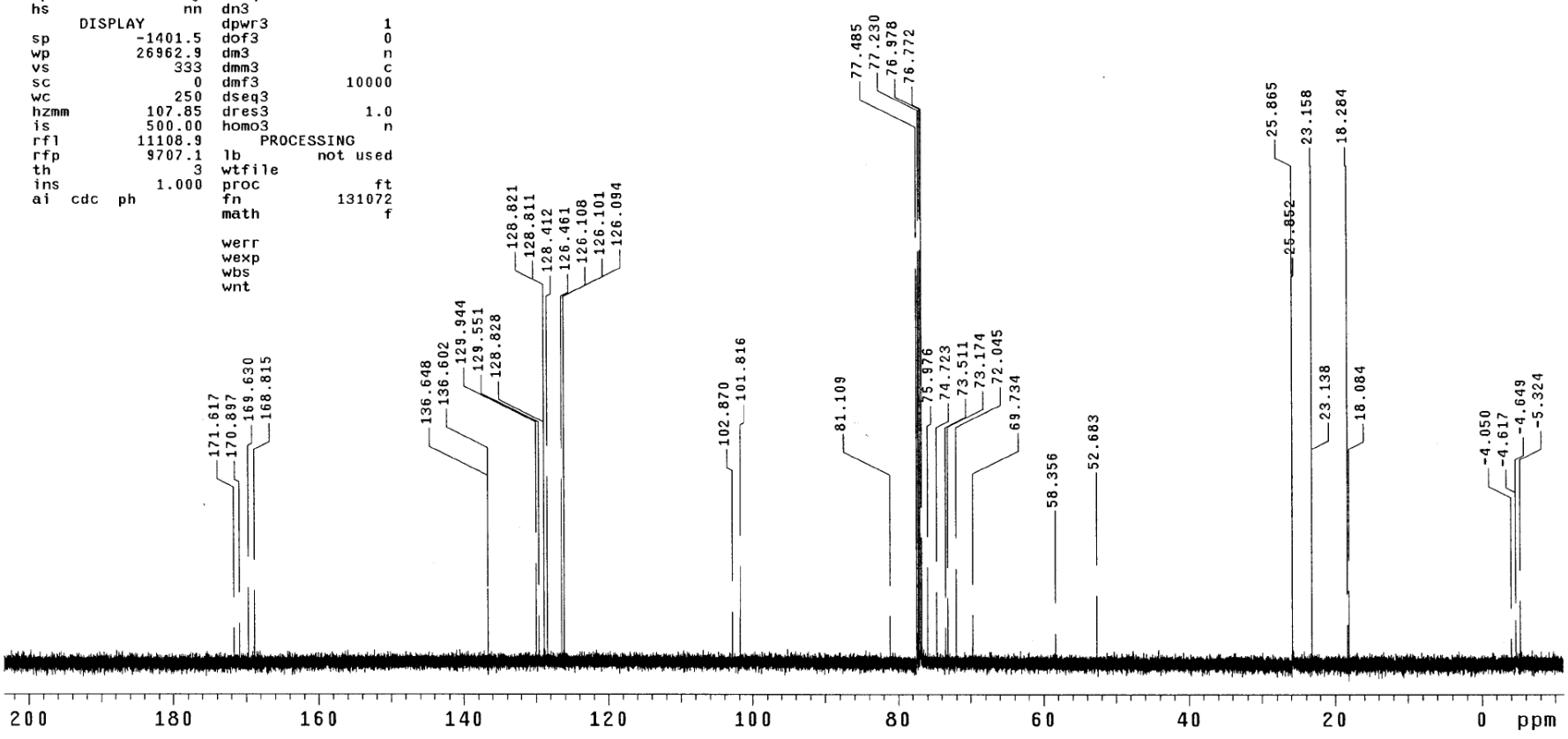

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


TY2-199
exp7 s2pul
SAMPLE
date Oct 21 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 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -68.5
wp 5065.0
vs 45
sc 0
wc 250
hzmm 20.26
is 344.44
rfl 506.3
rfp 0
th 7
ins 9.000
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
Ph
O
O
TBDMSO
H
N
O
HO
NHAc
8a
+
HO
OTBDMS
H
N
O
O
O
NHAc
Ph
8b
9
8
7
6
5
4
3
2
1
ppm
1.83
0.56
0.56
1.54
0.54
0.94
2.77
5.54
2.65
1.63

TY2-199

exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 21 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	3000	dm2	n
ct	896	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	-1401.5	dof3	0
wp	26962.9	dm3	n
vs	333	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11108.9	PROCESSING	
rfp	9707.1	lb	not used
th	3	wtfile	
ins	1.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

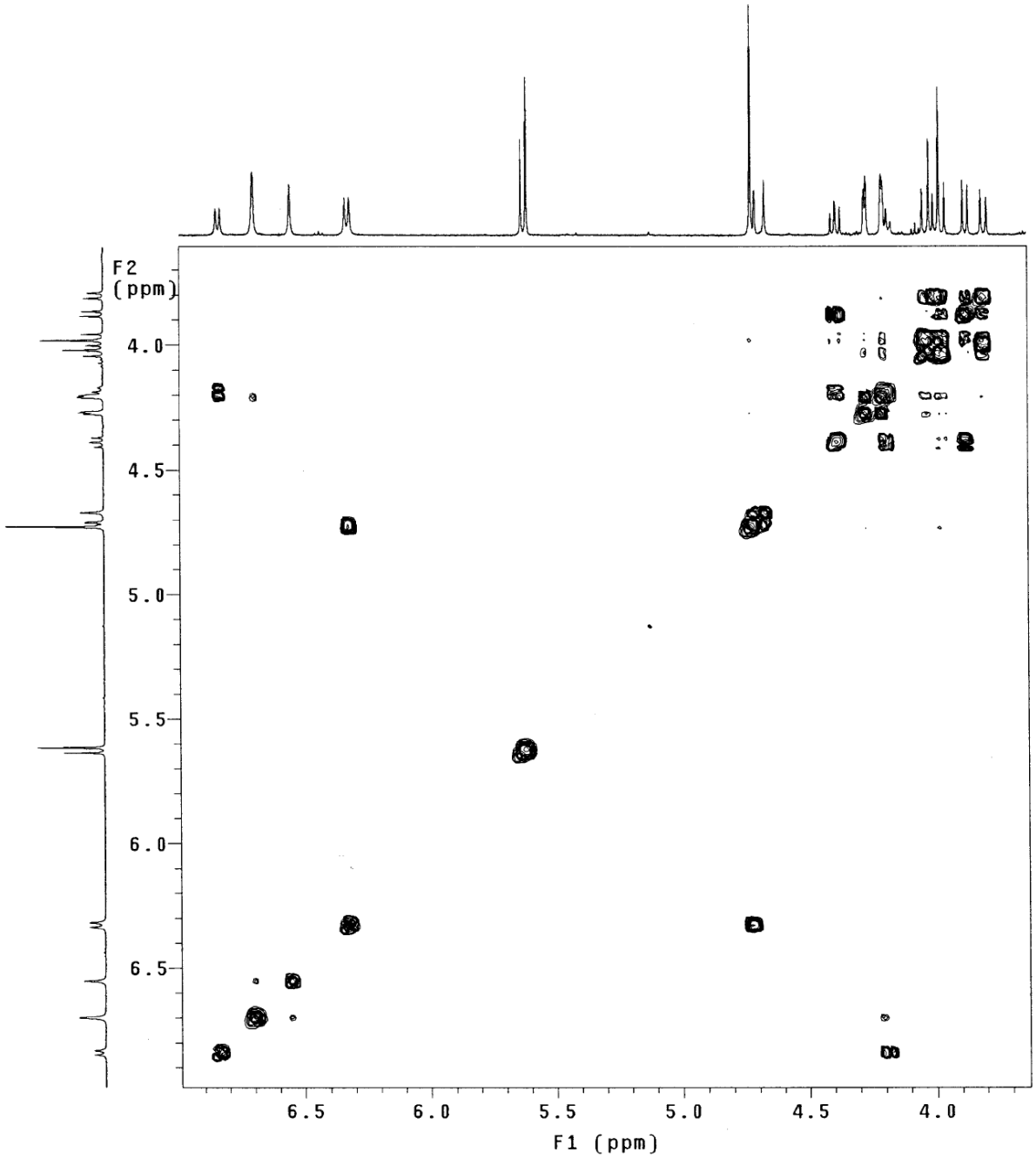


TY2-199

Pulse Sequence: relayh

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

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
16 repetitions
256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

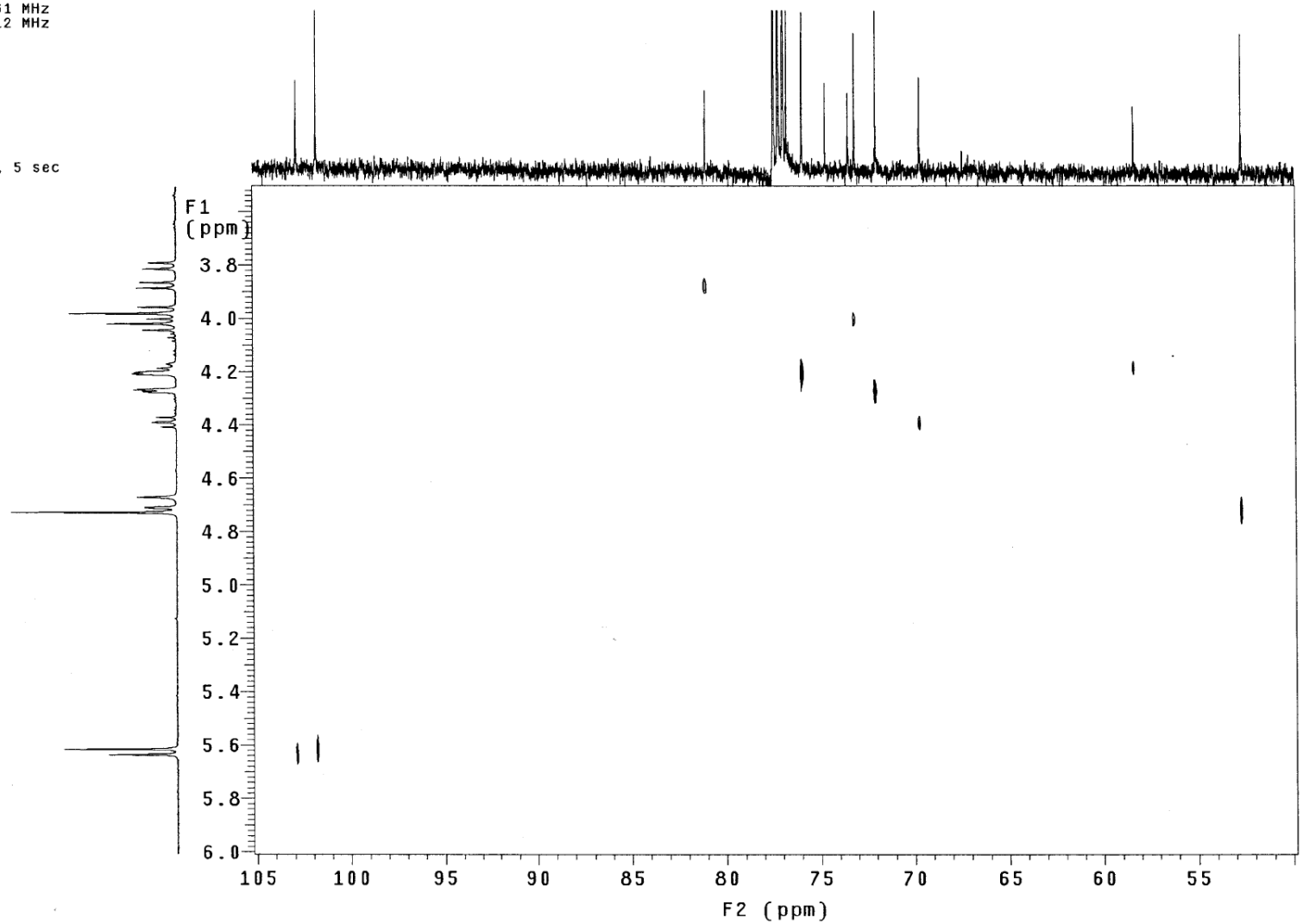


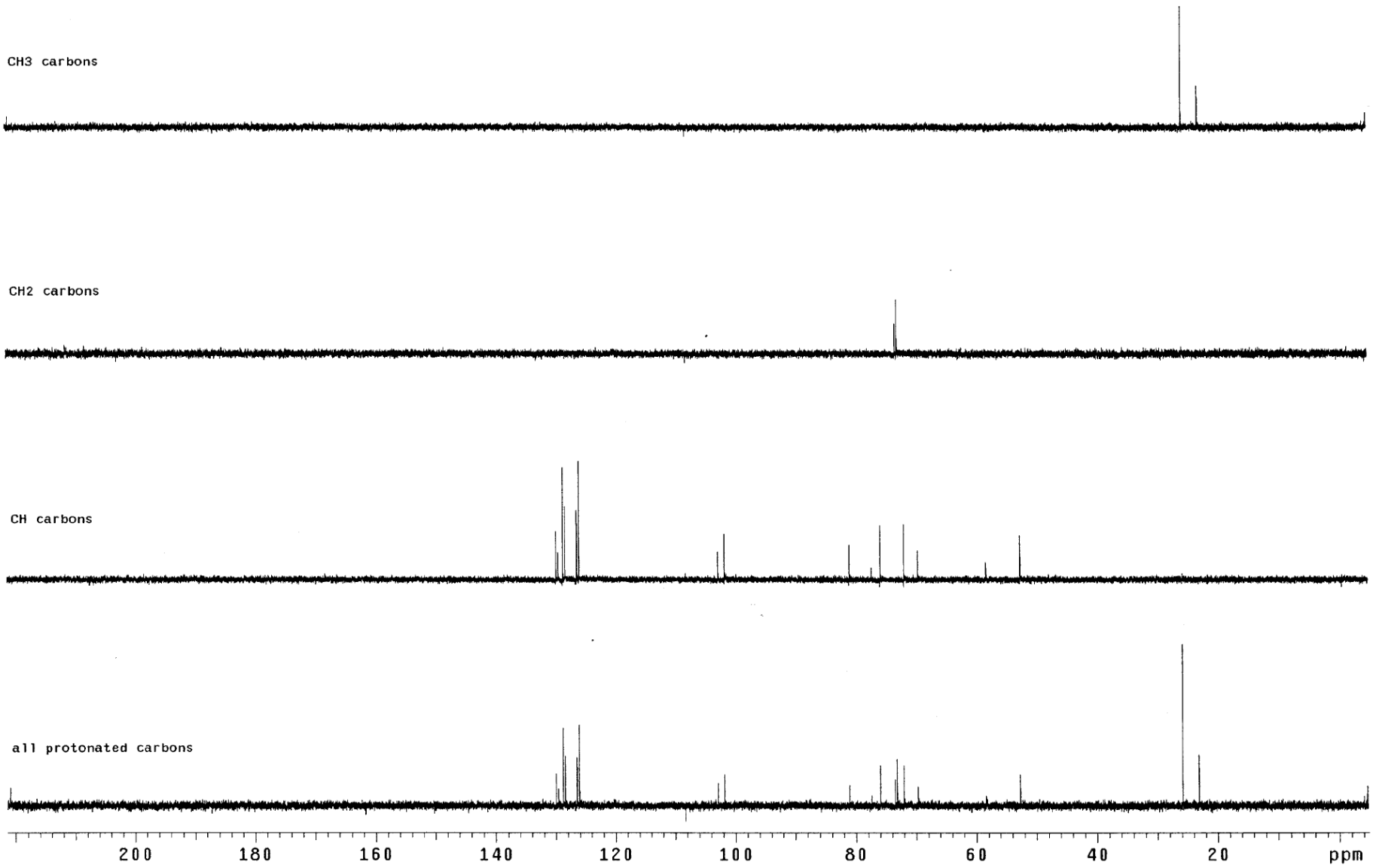
TY2-199

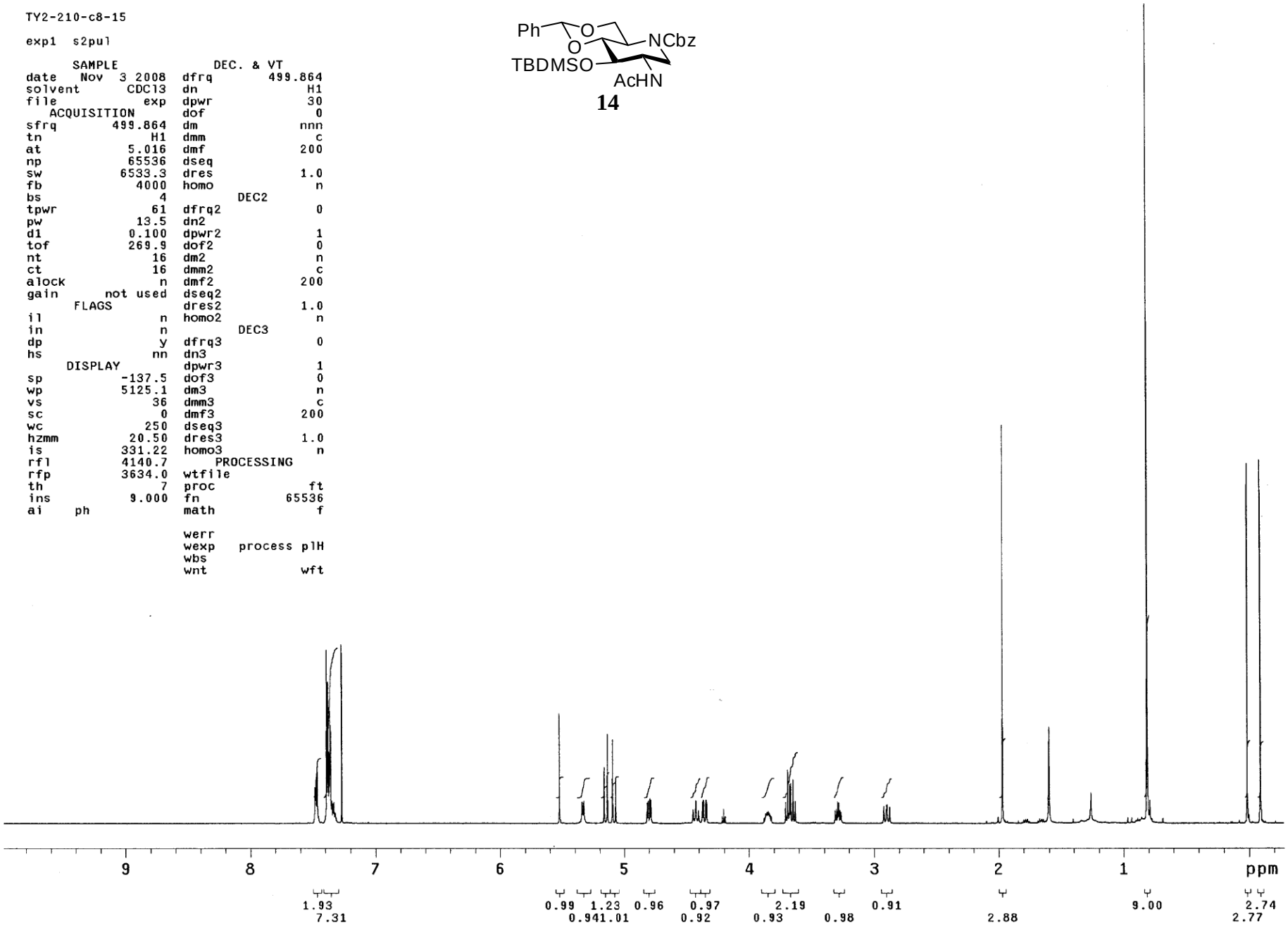
Pulse Sequence: hetcor

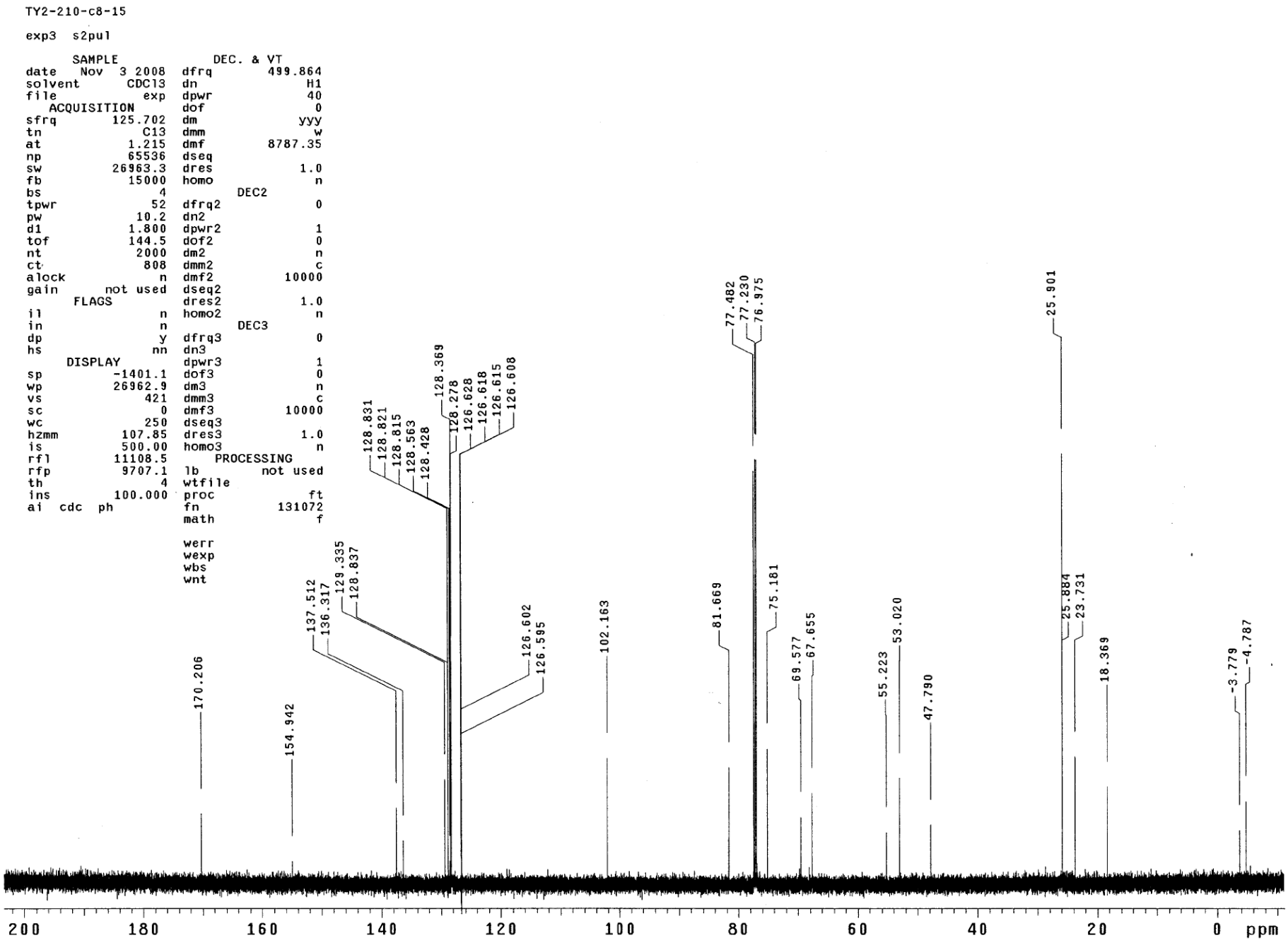
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE C13, 125.6901661 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 3 hr, 46 min, 5 sec




CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

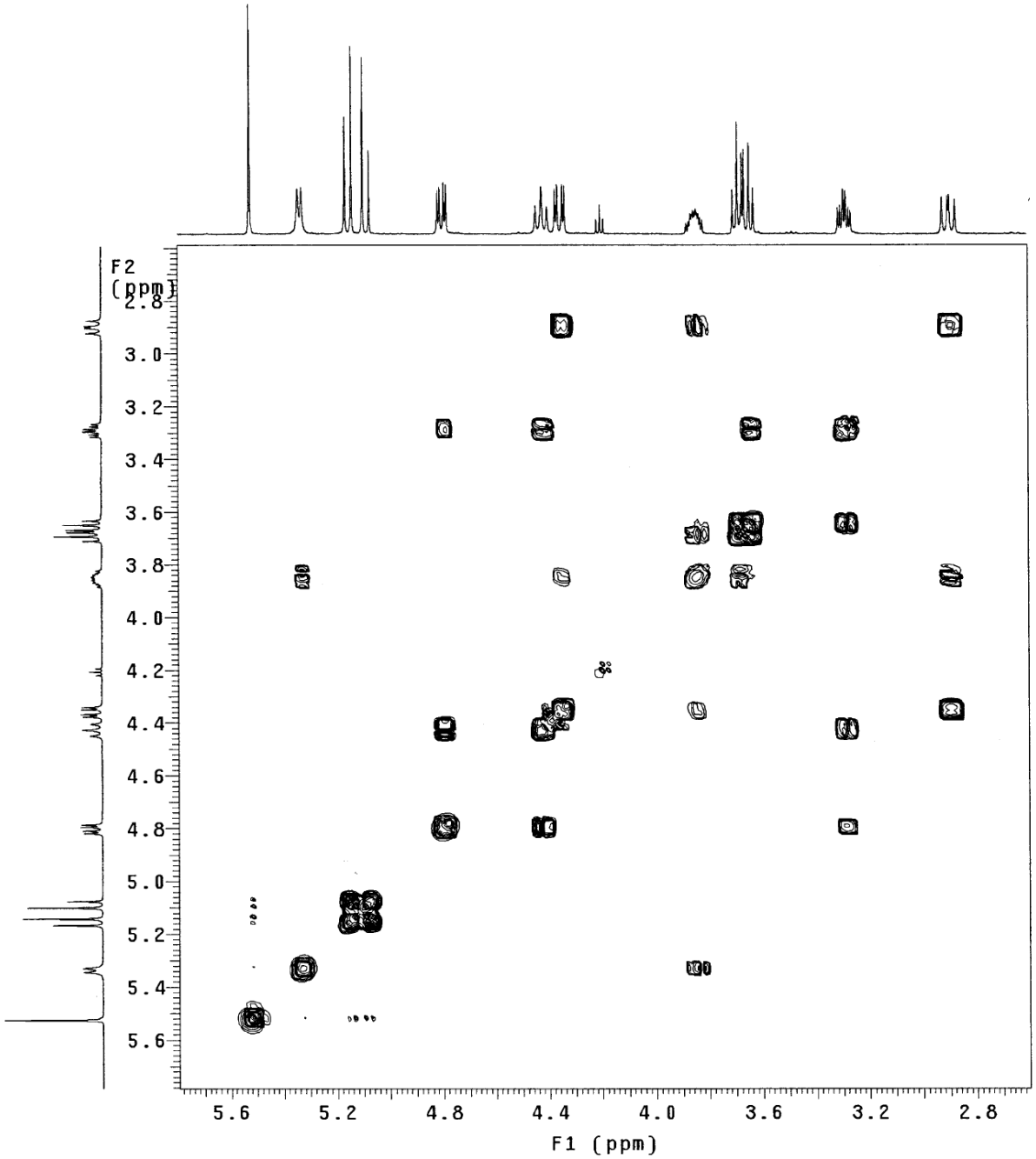

TY2-210-c8-15
exp1 s2pul
SAMPLE
date Nov 3 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -137.5
wp 5125.1
vs 36
sc 0
wc 250
hzmm 20.50
is 331.22
rfl 4140.7
rfp 3634.0
th 7
ins 9.000
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
Ph
O
O
NCbz
TBDMSO
AcHN
14
9
8
7
6
5
4
3
2
1
ppm
1.93
7.31
0.99
1.23
0.96
0.97
2.19
0.91
9.00
2.74
0.94
1.01
0.92
0.93
0.98
2.88
2.77


TY2-210-c8-15
exp3 s2pul
SAMPLE
date Nov 3 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 2000
ct 808
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1401.1
wp 26962.9
vs 421
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11108.5
rfp 9707.1
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 not used
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
170.206
154.942
137.512
136.317
129.335
128.837
128.831
128.821
128.815
128.563
128.428
128.369
128.278
126.628
126.618
126.615
126.608
126.602
126.585
102.163
81.669
77.482
77.230
76.975
75.181
69.577
67.655
55.223
53.020
47.790
25.901
25.884
23.731
18.369
-3.779
-4.787
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-210-c8-15

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

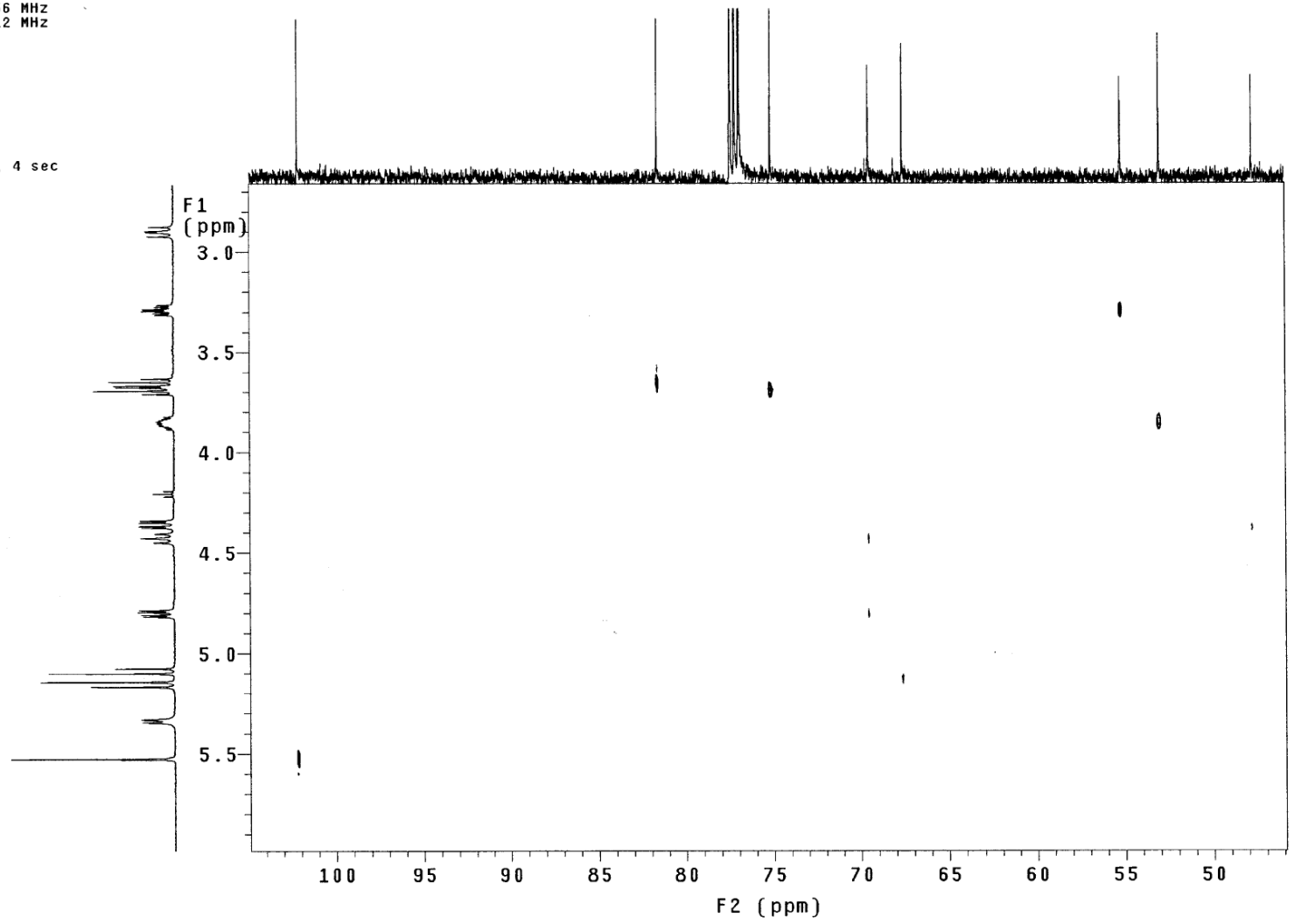
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
16 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

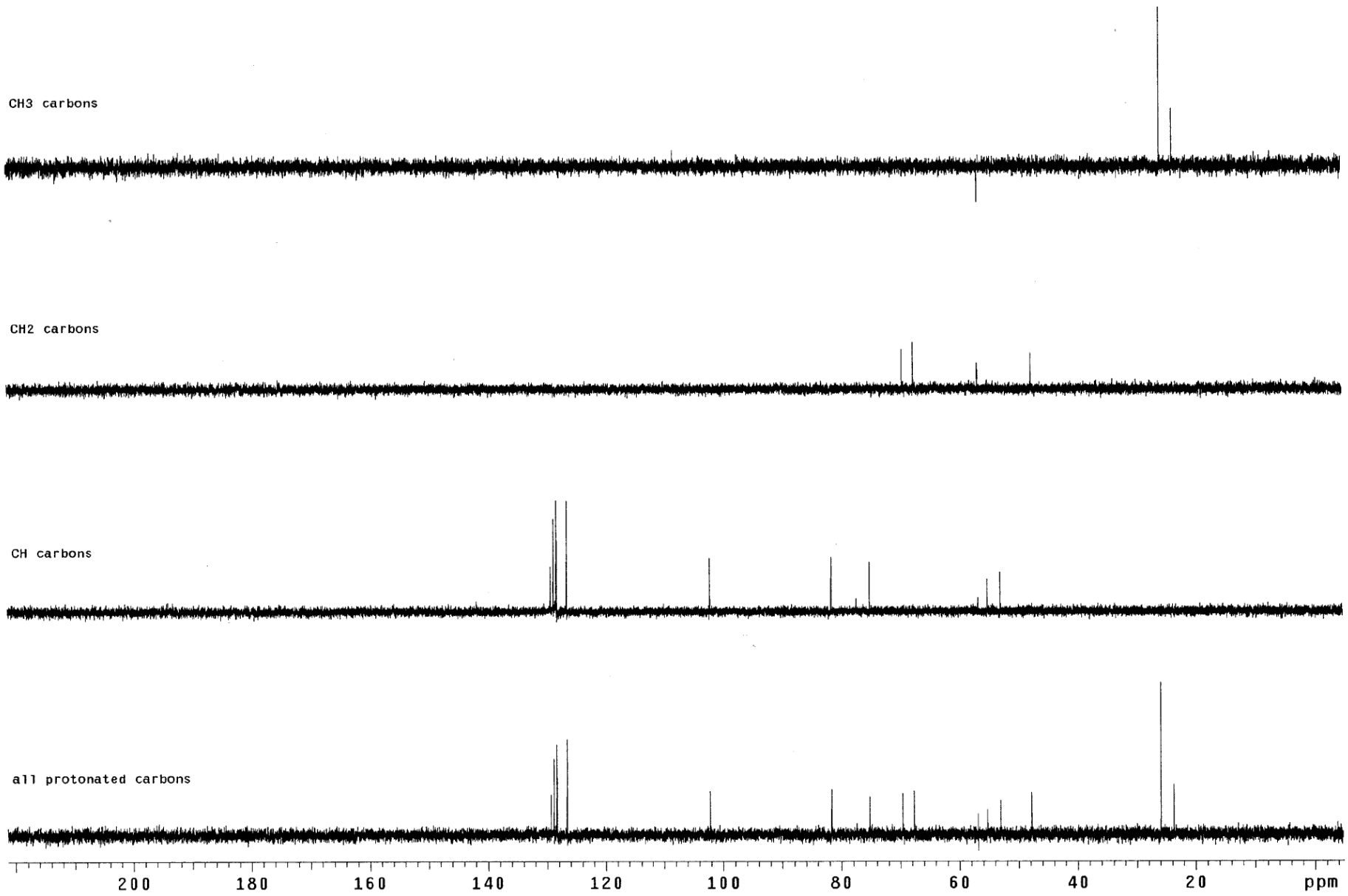


TY2-210-c8-15

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
 64 repetitions
 256 increments
OBSERVE C13, 125.6901656 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 7 hr, 32 min, 4 sec

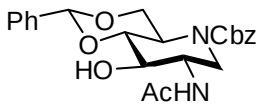



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

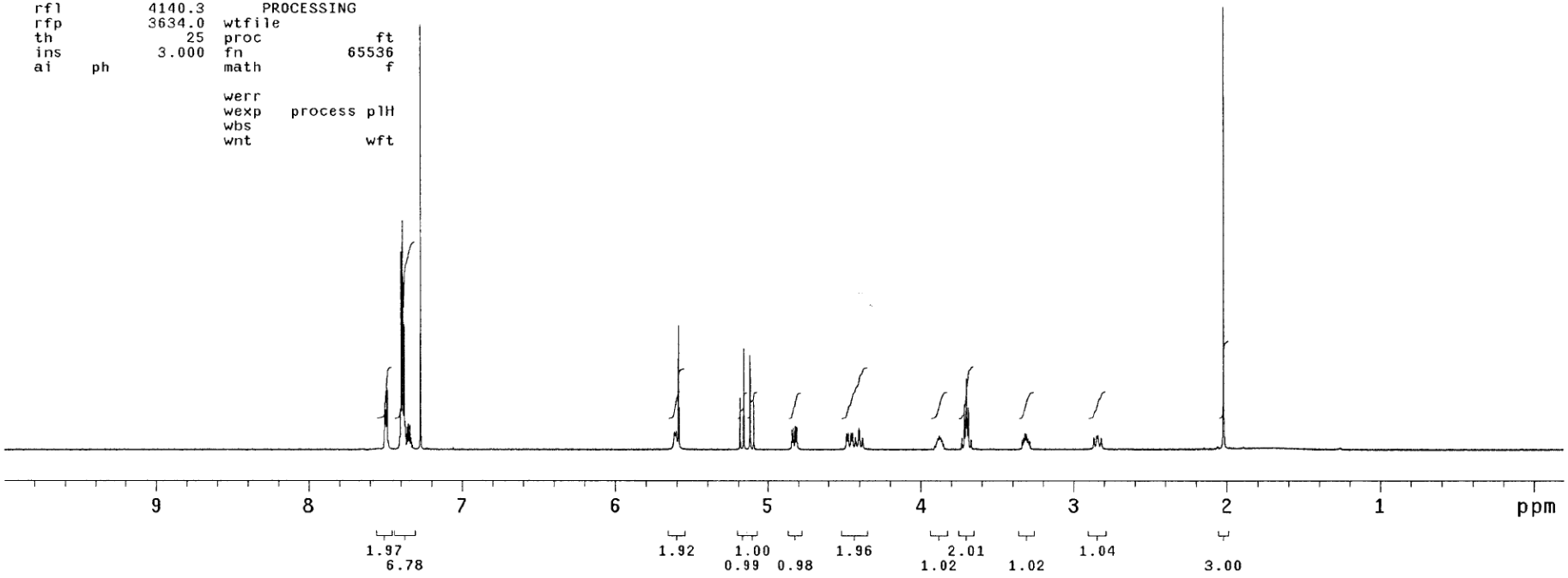
TY2-298

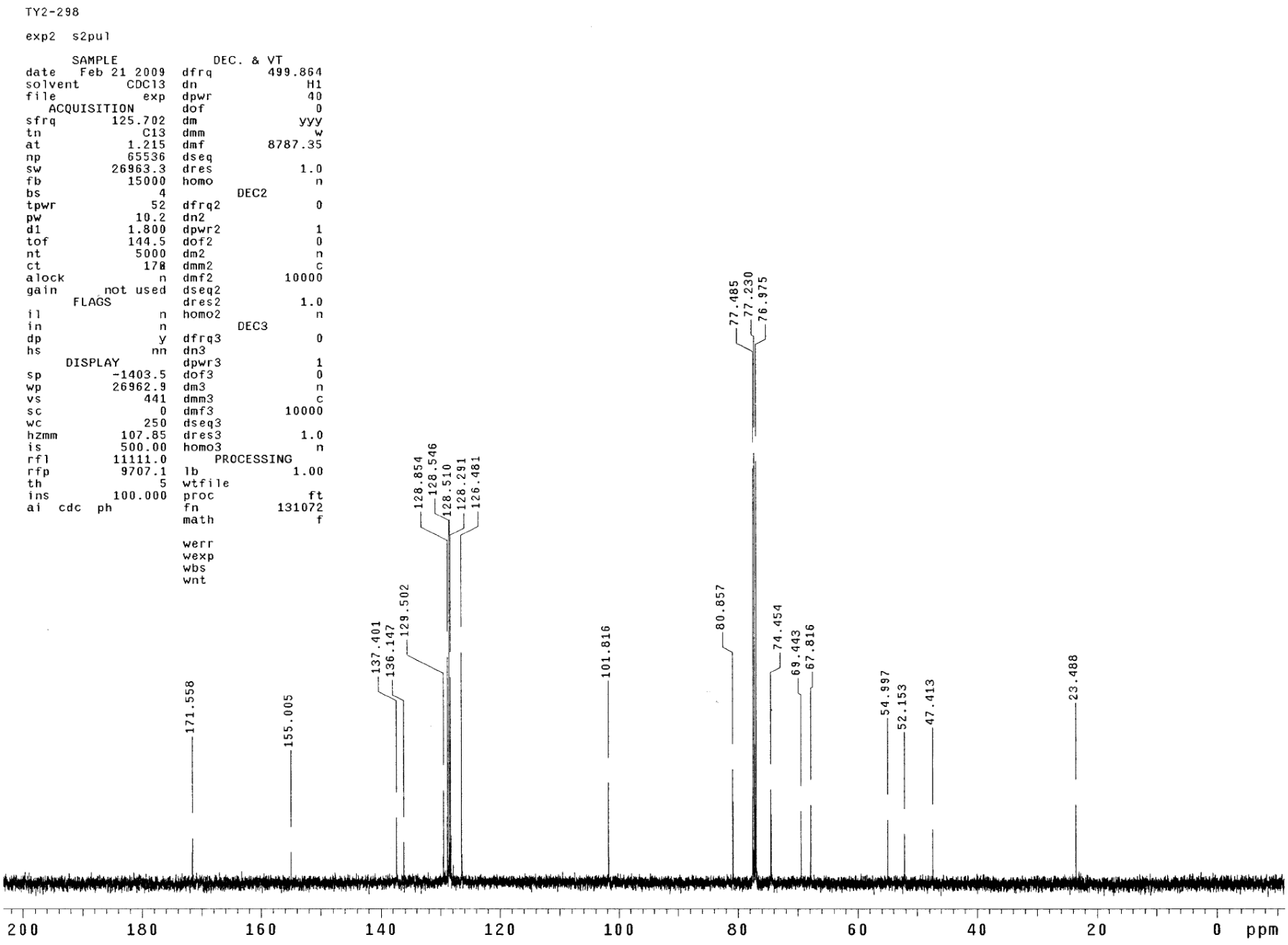
exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 20 2009	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	-94.2	dof3	0
wp	5090.8	dm3	n
vs	23	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.36	dres3	1.0
is	233.40	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	25	proc	ft
ins	3.000	fn	65536
ai	ph	math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



7



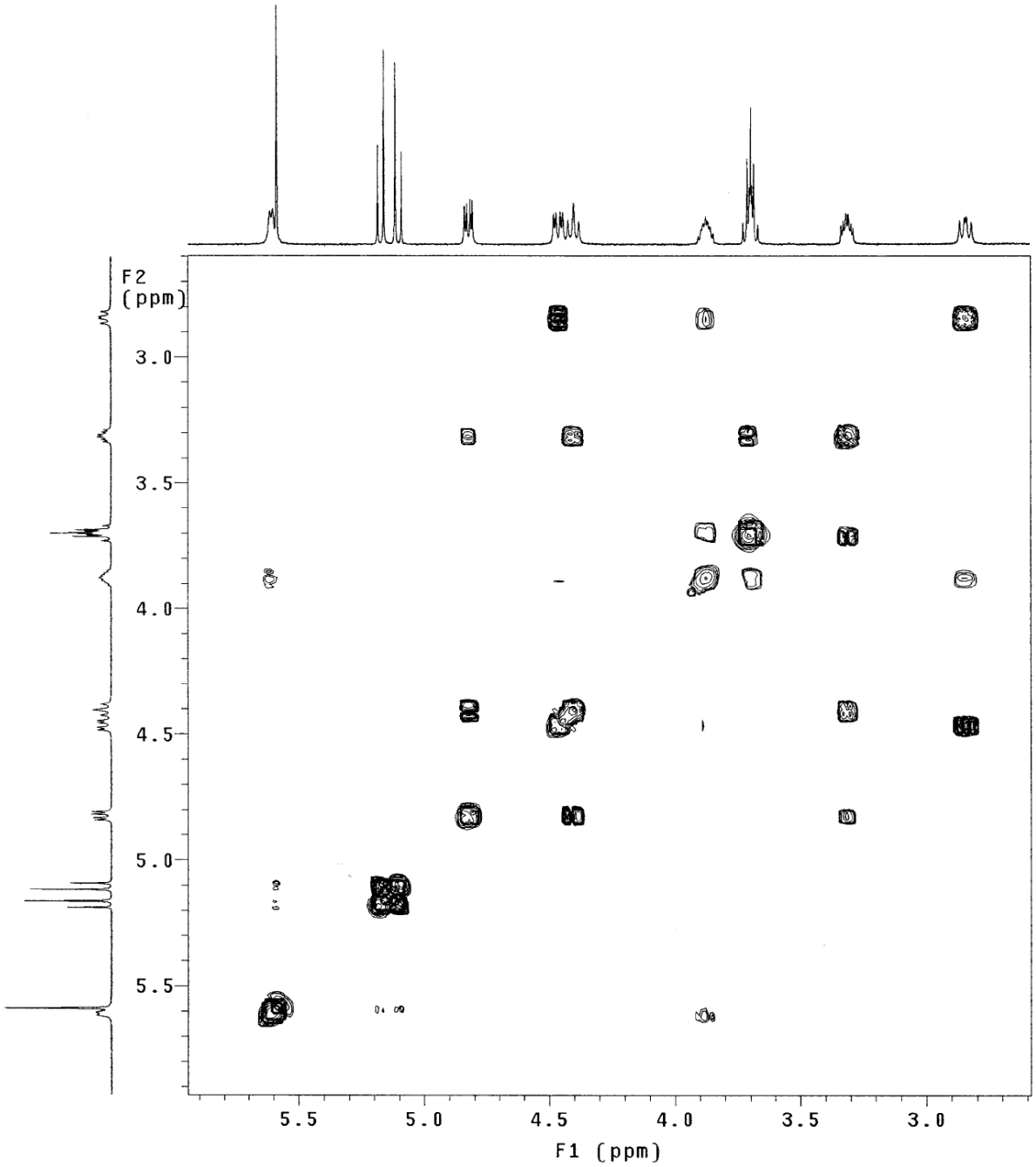

TY2-298
exp2 s2pul
SAMPLE
date Feb 21 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 5000
ct 178
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1403.5
wp 26962.9
vs 441
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11111.0
rfp 9707.1
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
171.558
155.005
137.401
136.147
129.502
128.854
128.546
128.510
128.291
126.481
101.816
80.857
77.485
77.230
76.975
74.454
69.443
67.816
54.997
52.153
47.413
23.488
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-298

Pulse Sequence: relayh

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

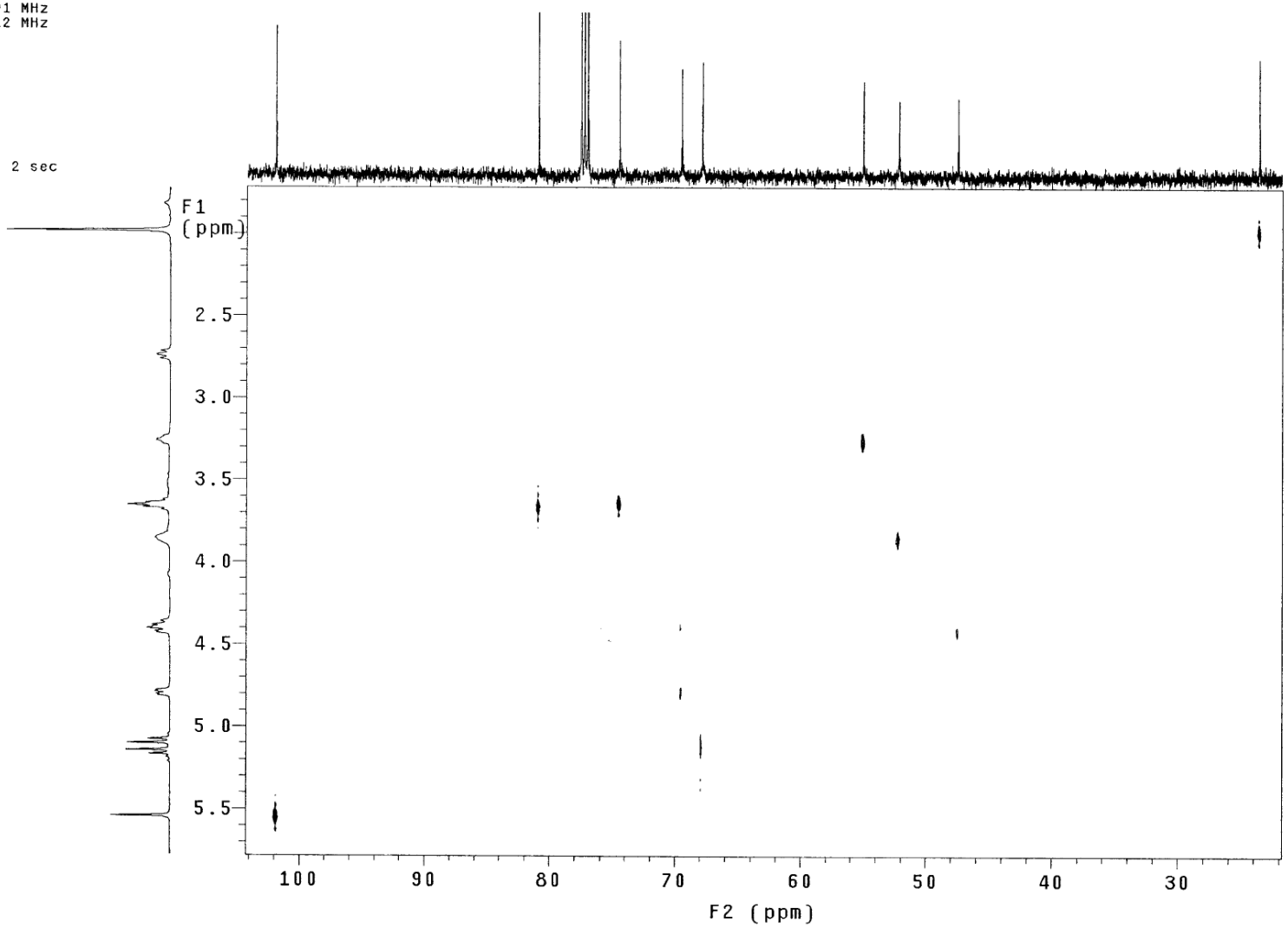
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

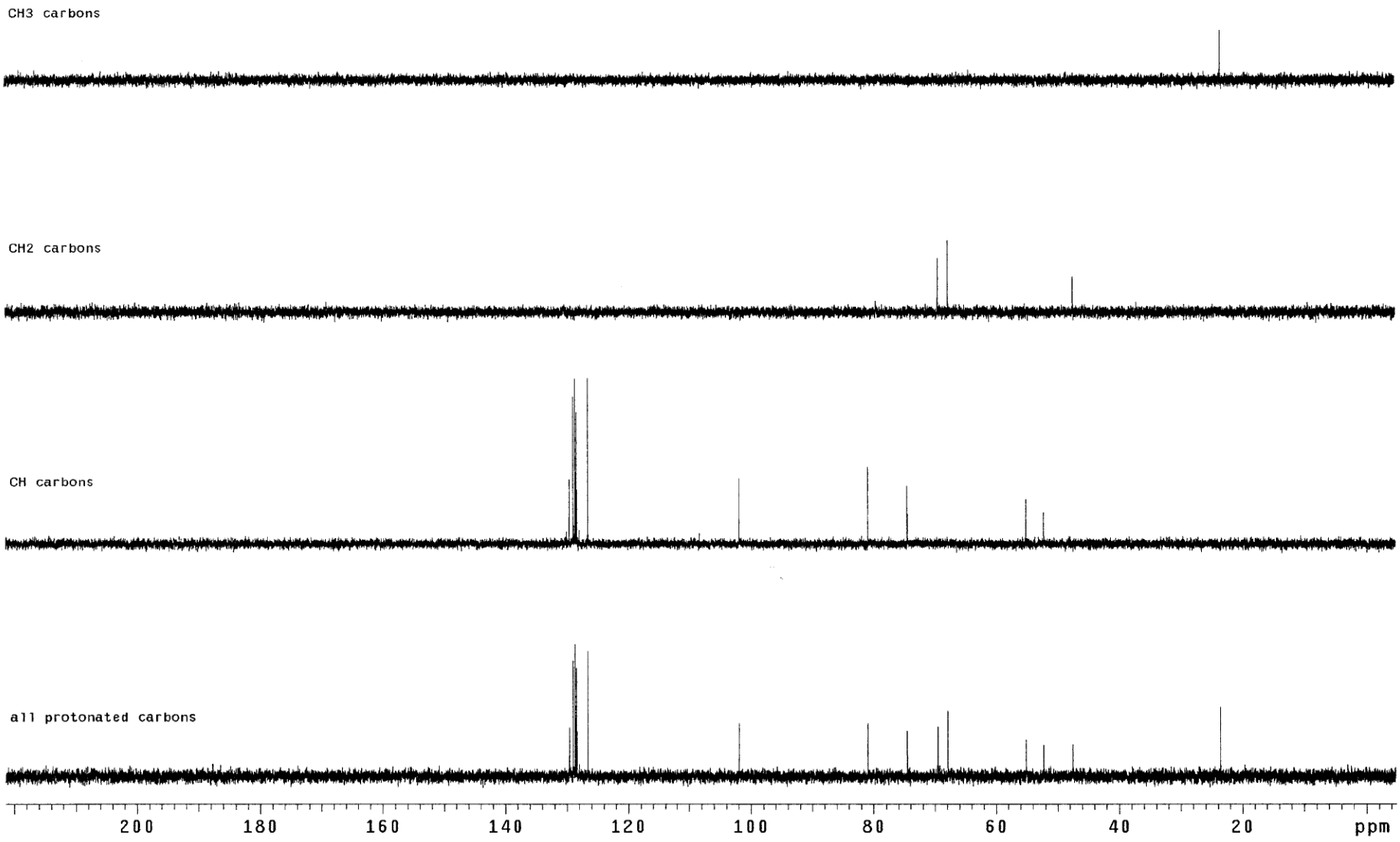


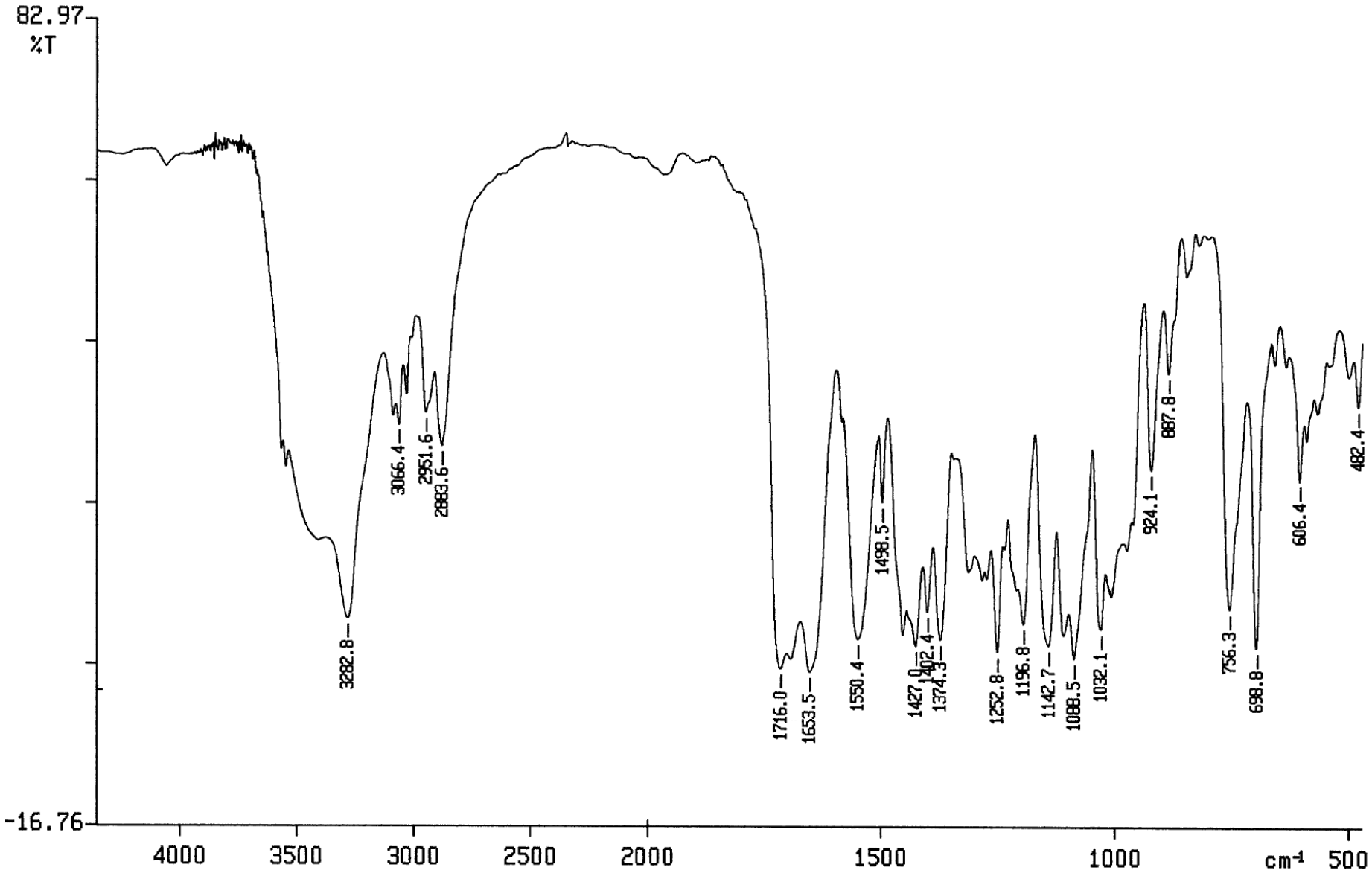
TY2-298

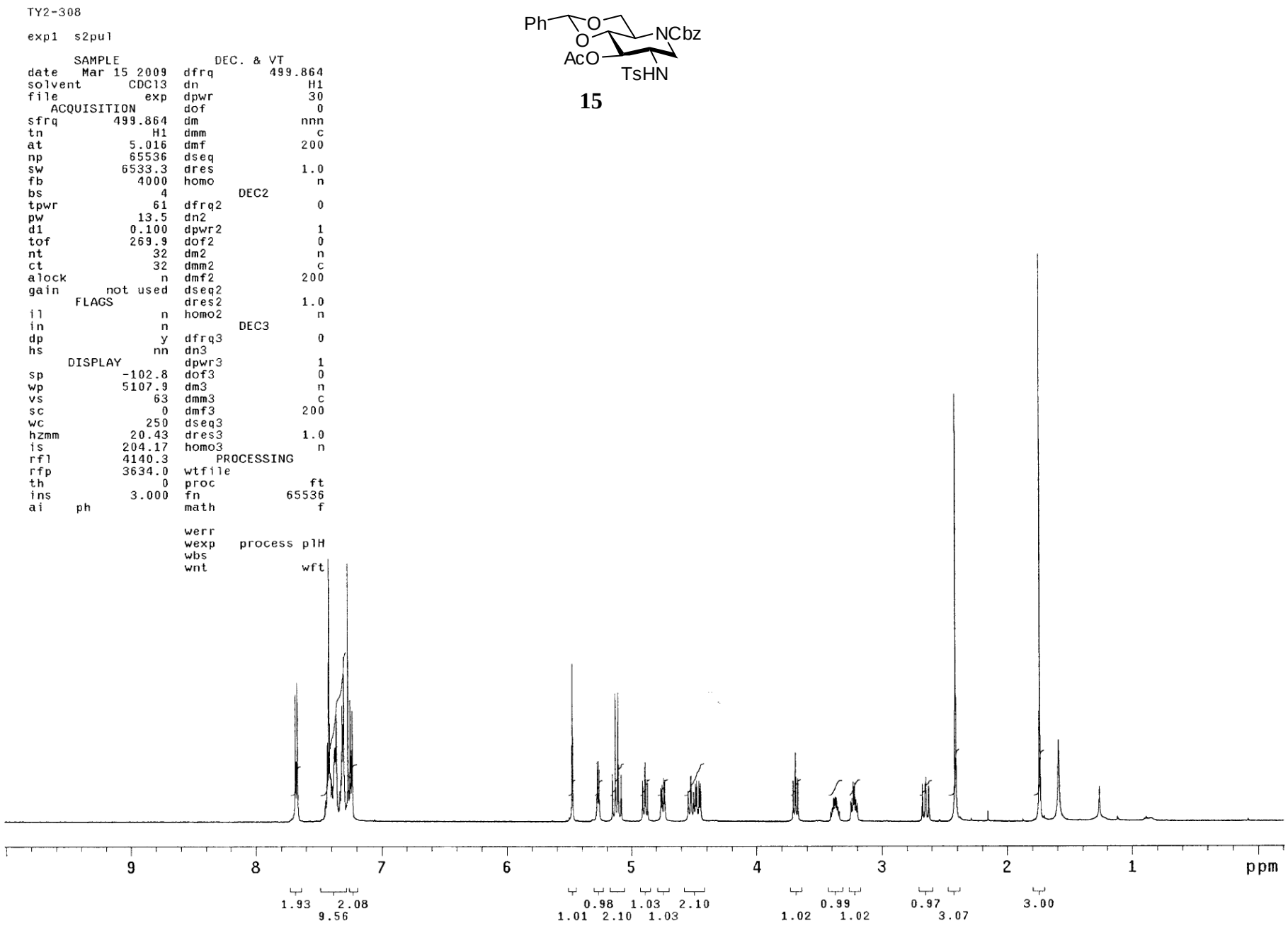
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

 Relax. delay 1.500 sec
 Acq. time 0.111 sec
 Width   18403.5 Hz
 2D Width 6533.3 Hz
 128 repetitions
 256 increments
OBSERVE C13, 125.6901591 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 15 hr, 4 min, 2 sec

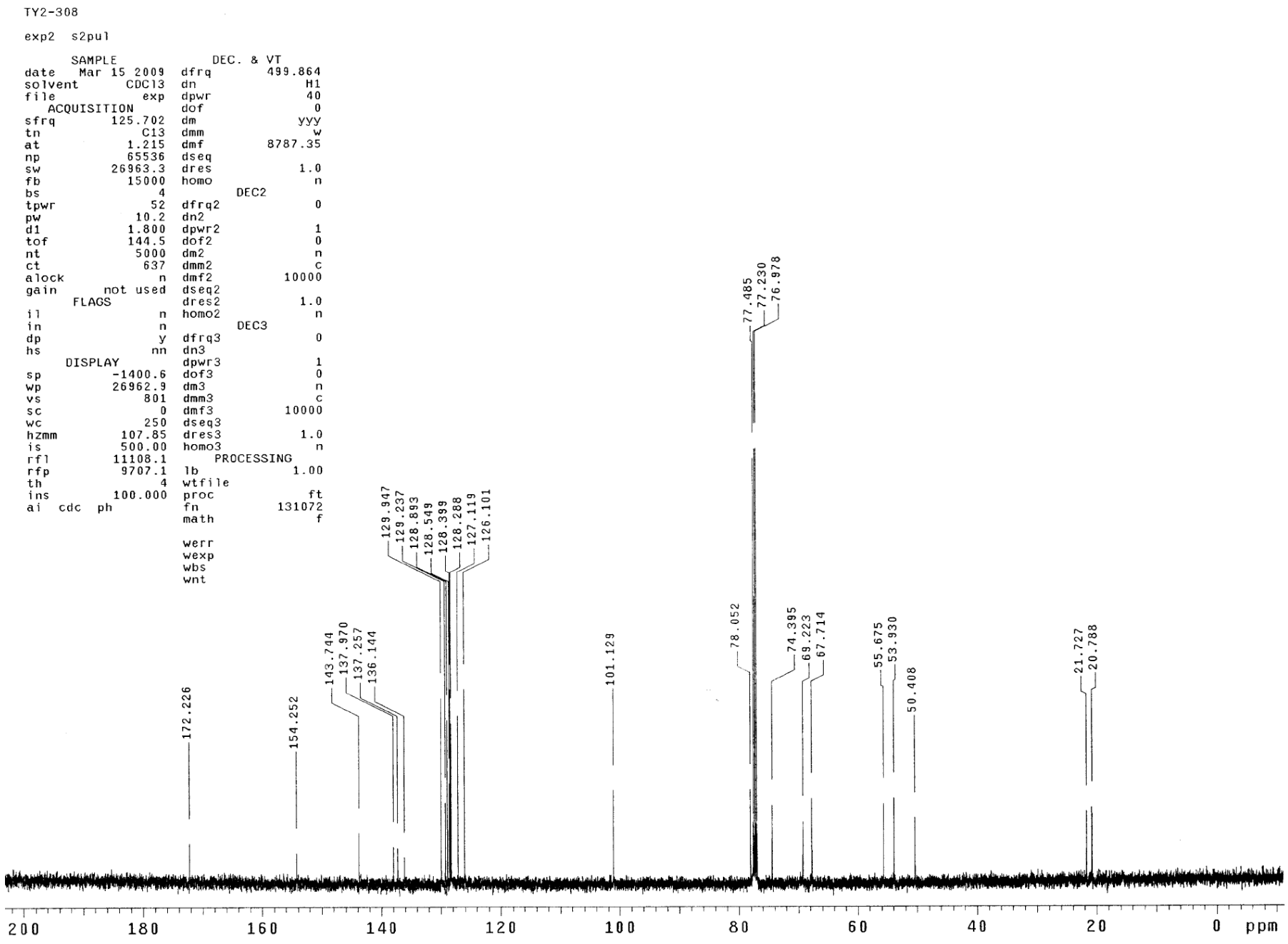



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


82.97
%T
-16.76
3282.8
3066.4
2951.6
2883.6
1716.0
1653.5
1550.4
1498.5
1427.0
1402.4
1374.3
1252.8
1196.8
1142.7
1088.5
1032.1
924.1
887.8
756.3
698.8
606.4
482.4
4000
3500
3000
2500
2000
1500
1000
cm-1
500


TY2-308
exp1 s2pul
SAMPLE
date Mar 15 2009
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 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -102.8
wp 5107.9
vs 63
sc 0
wc 250
hzmm 20.43
is 204.17
rfl 4140.3
rfp 3634.0
th 0
ins 3.000
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
Ph
O
O
NCbz
AcO
TsHN
9
8
7
6
5
4
3
2
1
ppm
1.93
2.08
9.56
0.98
1.03
2.10
1.01
2.10
1.03
0.99
1.02
1.02
0.97
3.07
3.00

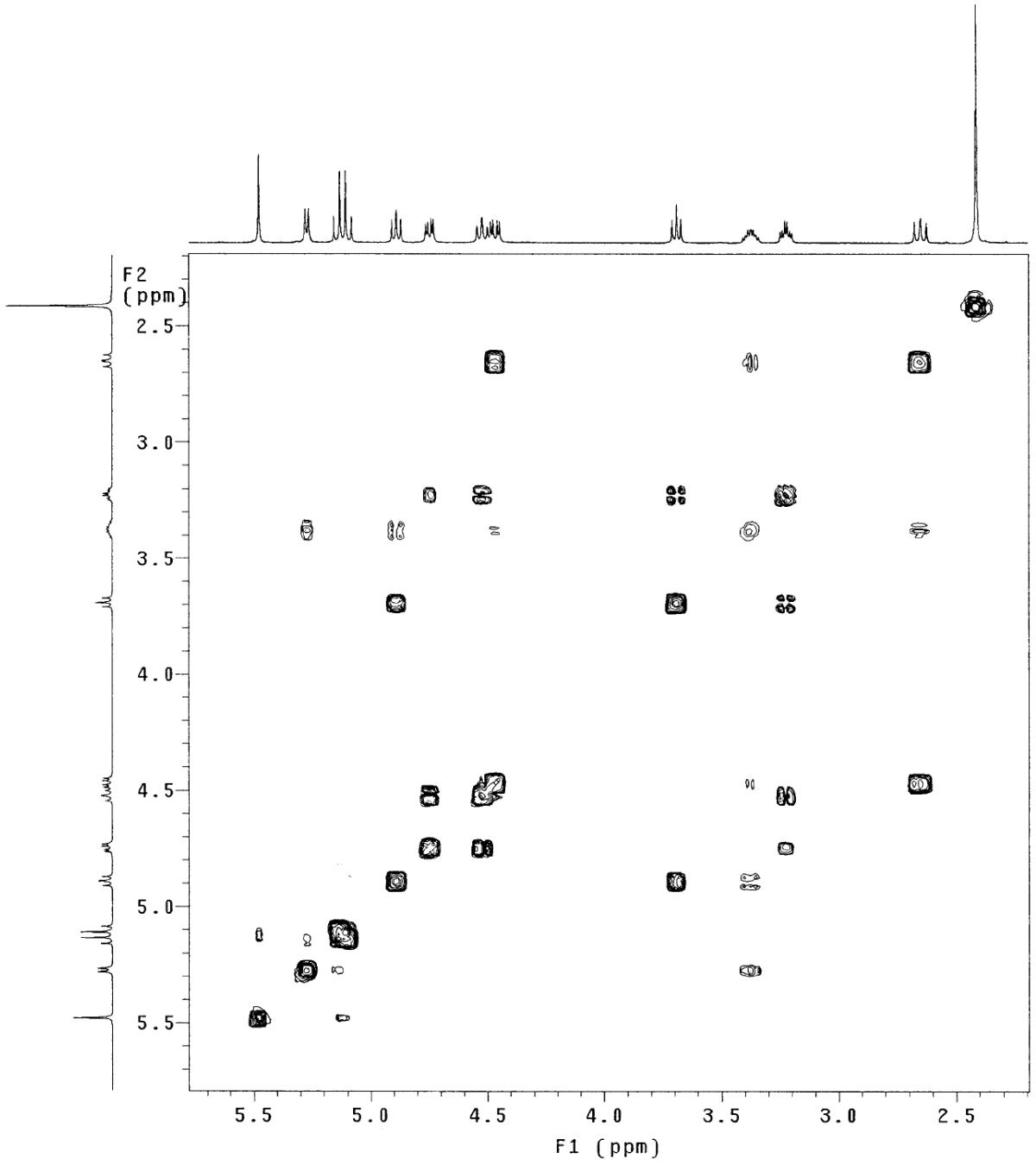
15


TY2-308
exp2 s2pul
SAMPLE
date Mar 15 2009
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 5000
ct 637
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1400.6
wp 26962.9
vs 801
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11108.1
rfp 9707.1
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
77.485
77.230
76.978
129.947
129.237
128.893
128.549
128.399
128.288
127.119
126.101
143.744
137.970
137.257
136.144
172.226
154.252
101.129
78.052
74.395
69.223
67.714
55.675
53.930
50.408
21.727
20.788
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-308

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

 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 64 repetitions
 256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 6 hr, 46 min, 8 sec

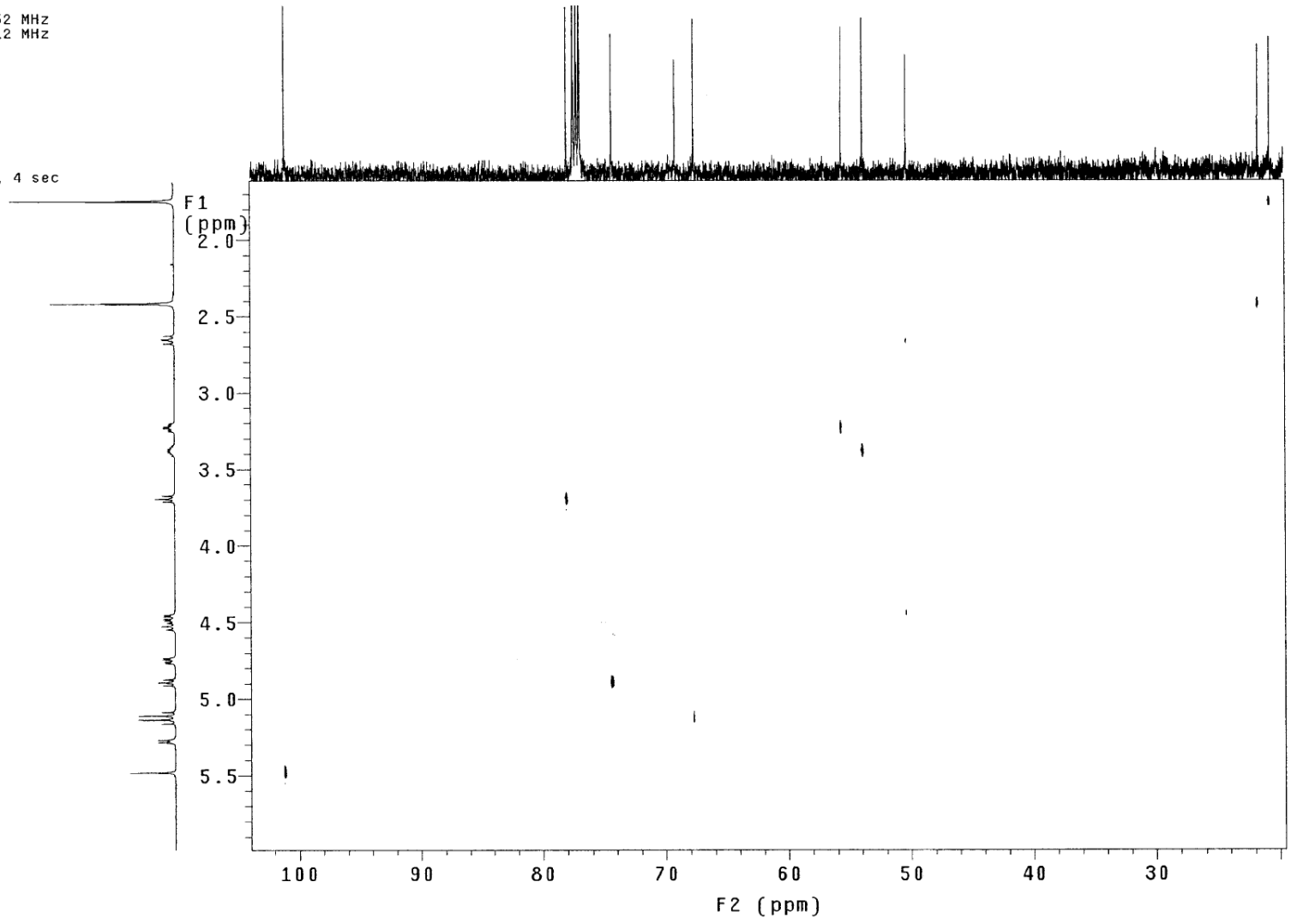


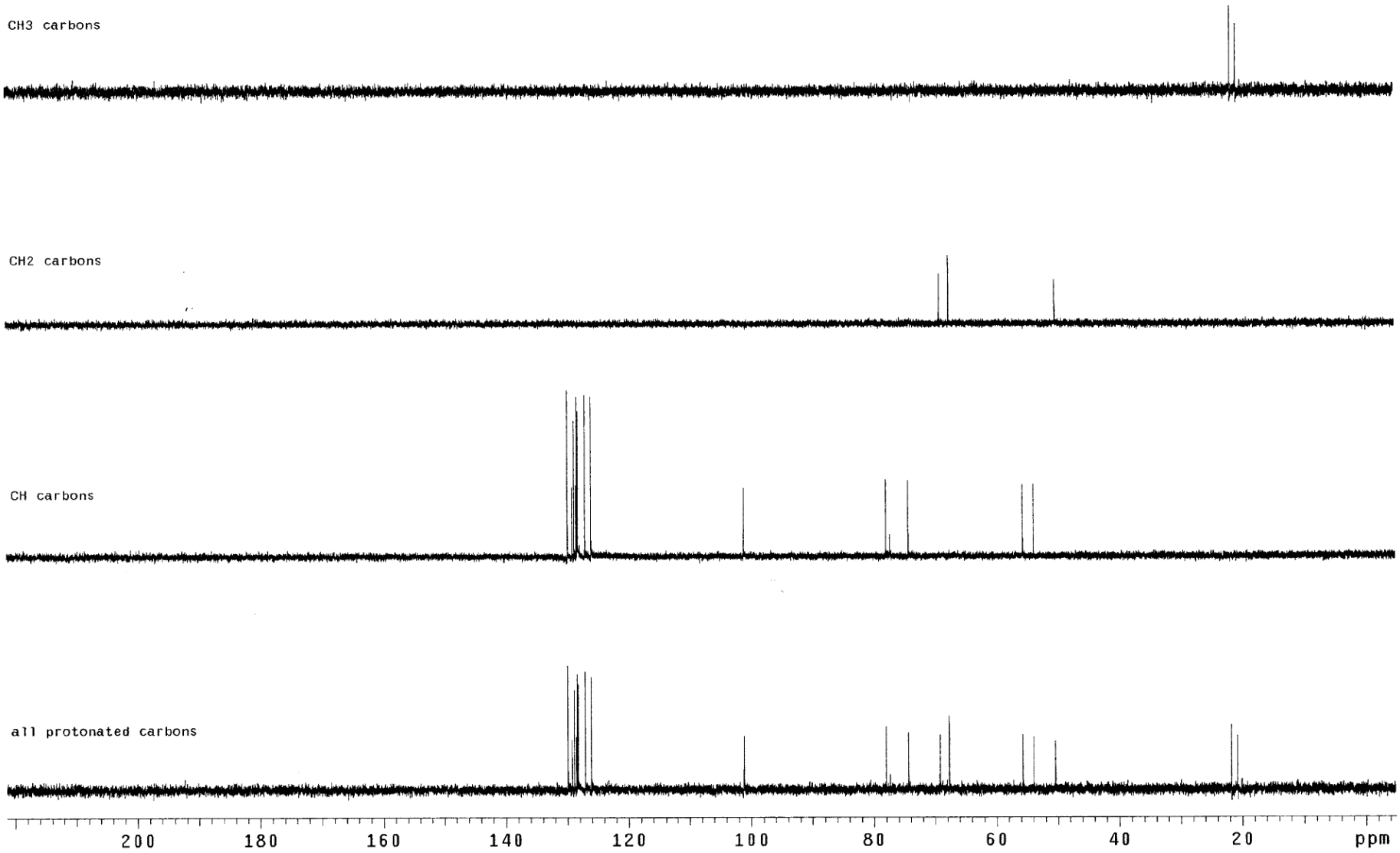
TY2-308

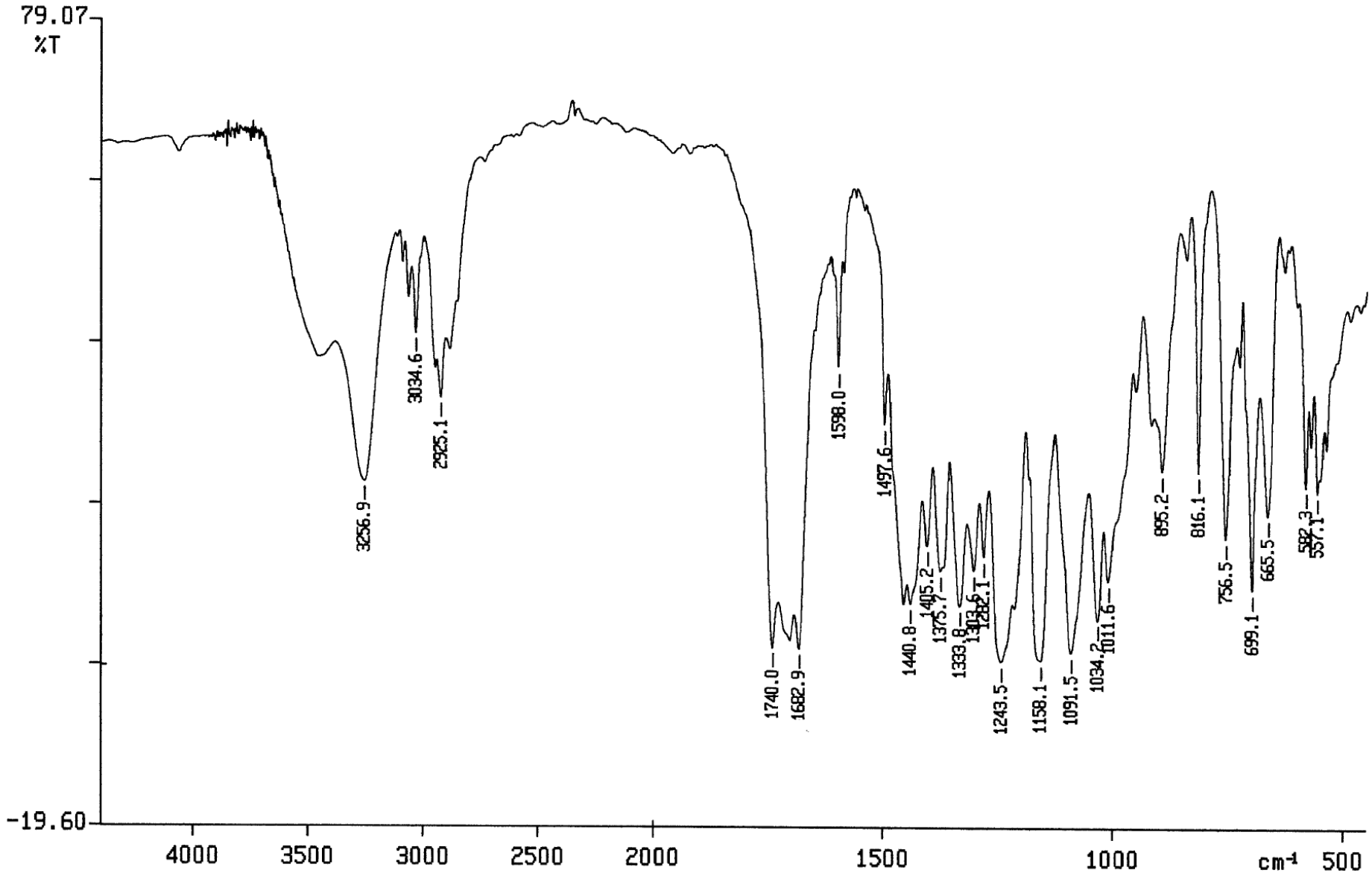
Pulse Sequence: hetcor

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: TY2-308-CH
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
64 repetitions
256 increments
OBSERVE C13, 125.6901652 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 7 hr, 32 min, 4 sec



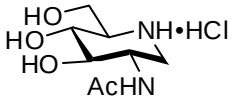

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


79.07
%T
-19.60
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3256.9
3034.6
2925.1
1740.0
1682.9
1598.0
1497.6
1440.8
1405.2
1375.7
1333.8
1243.5
1158.1
1091.5
1034.2
1011.6
895.2
816.1
756.5
699.1
665.5
582.3
557.1

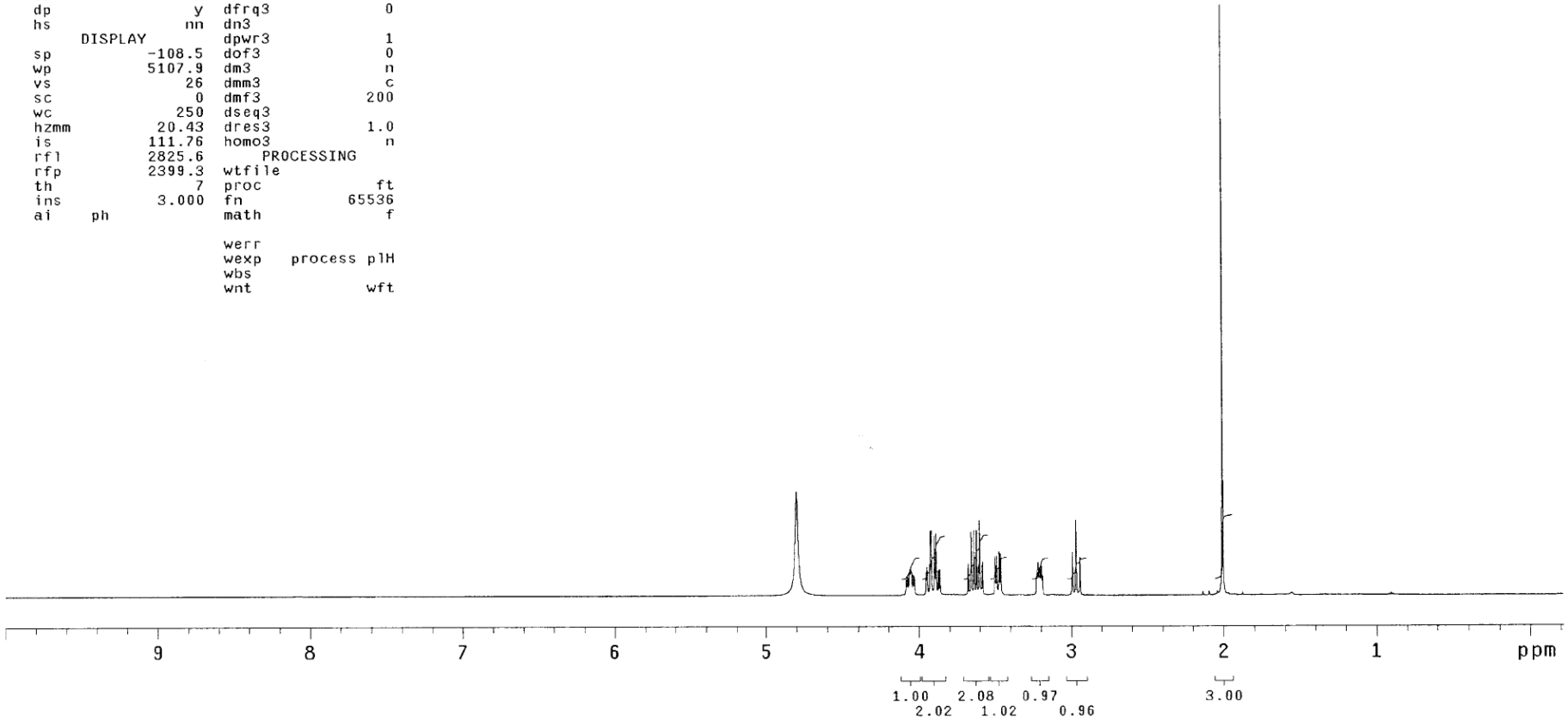
TY2-352

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 19 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.865	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	-108.5	dof3	0
wp	5107.9	dm3	n
vs	26	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	111.76	homo3	n
rfl	2825.6	PROCESSING	
rfp	2399.3	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



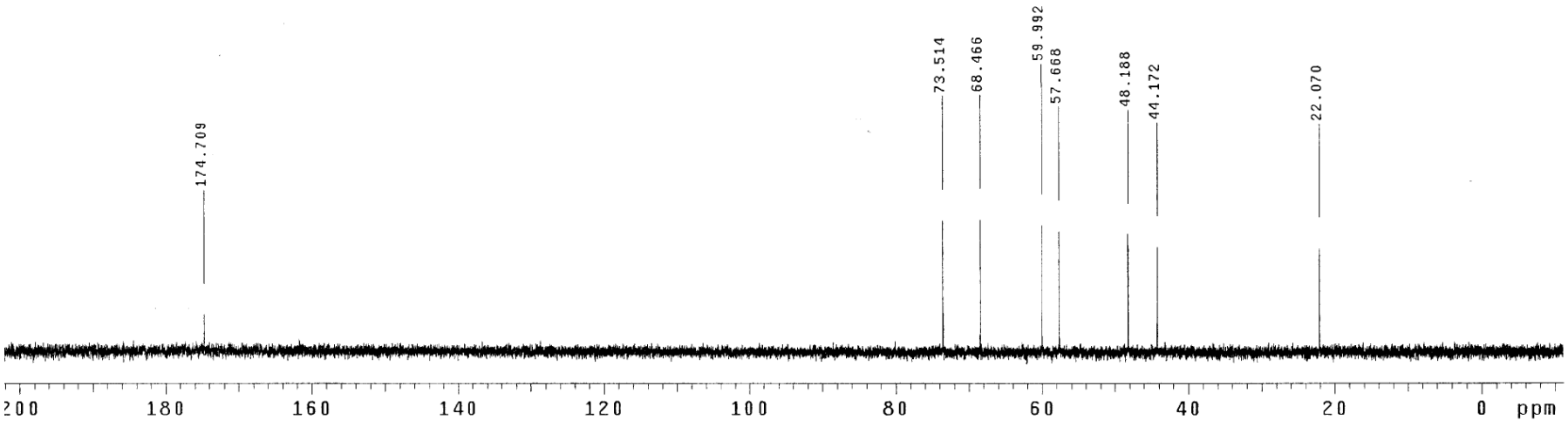
3



TY2-352

exp2 s2pul

SAMPLE		DEC. & VT	
date	May 19 2009	dfrq	499.865
solvent	D2O	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	5000	dm2	n
ct	96	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	-1394.6	dof3	0
wp	26962.9	dm3	n
vs	242	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	1395.0	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	

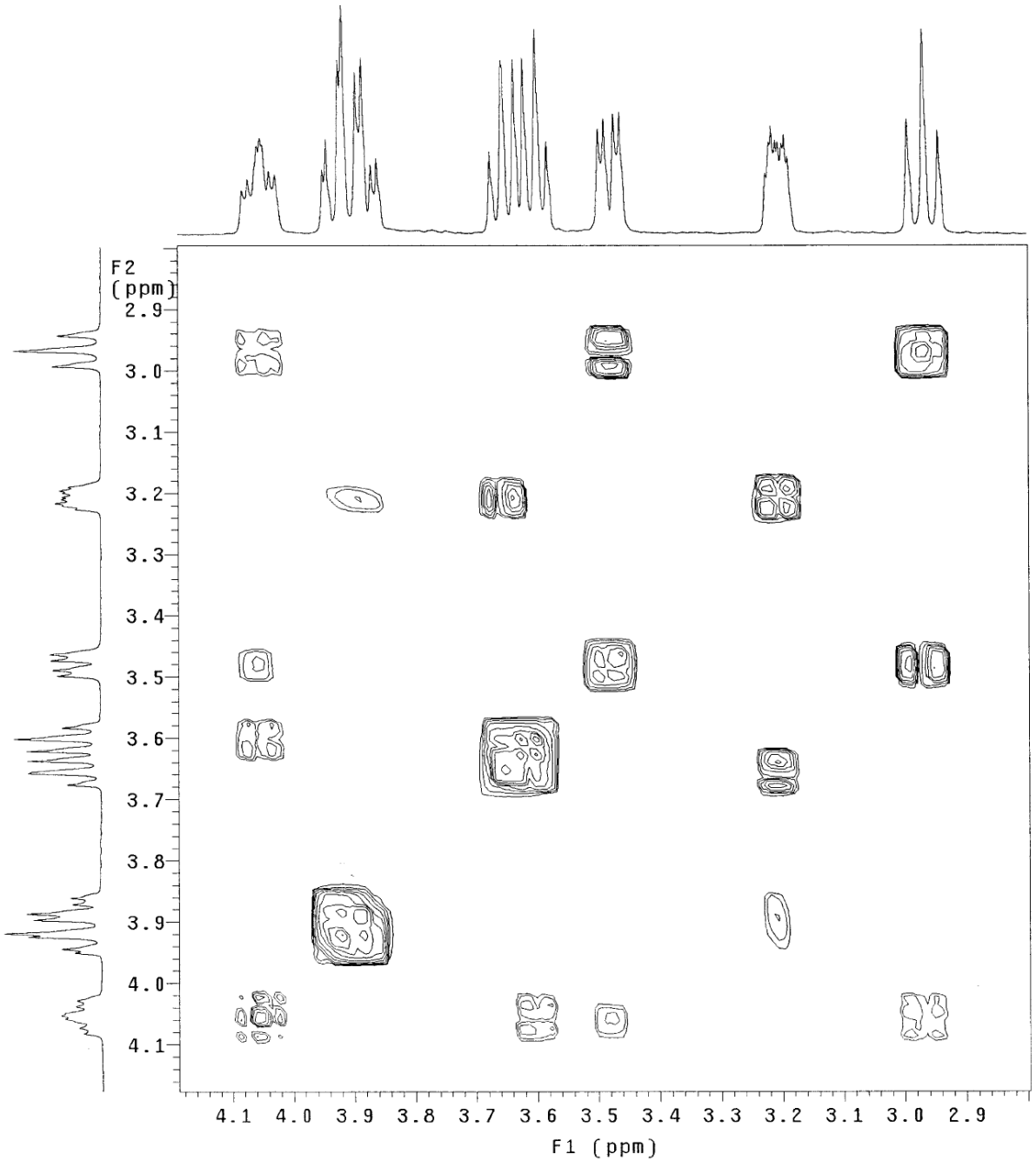


TY2-352

Pulse Sequence: relayh

Solvent: D2O
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

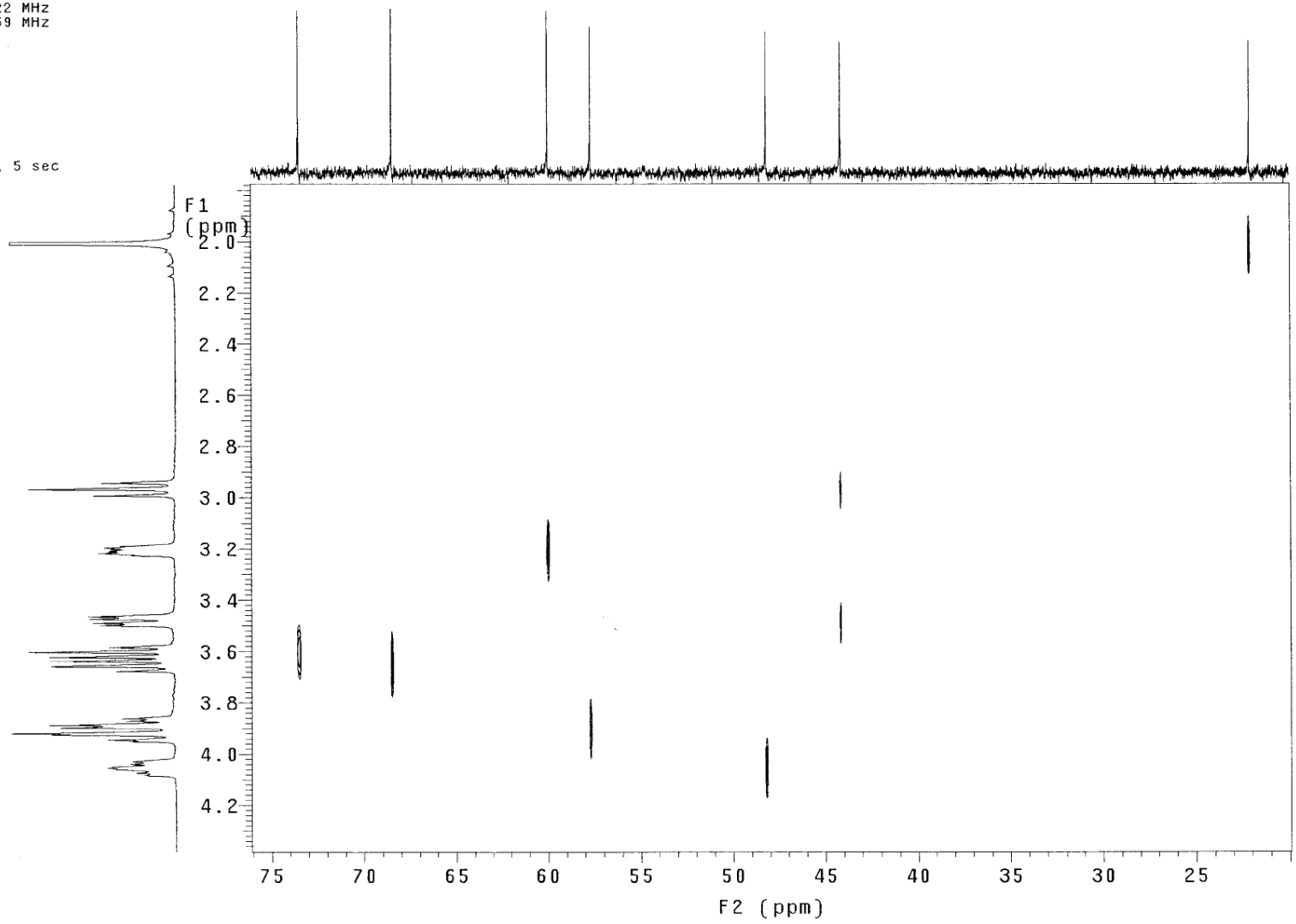
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8623759 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

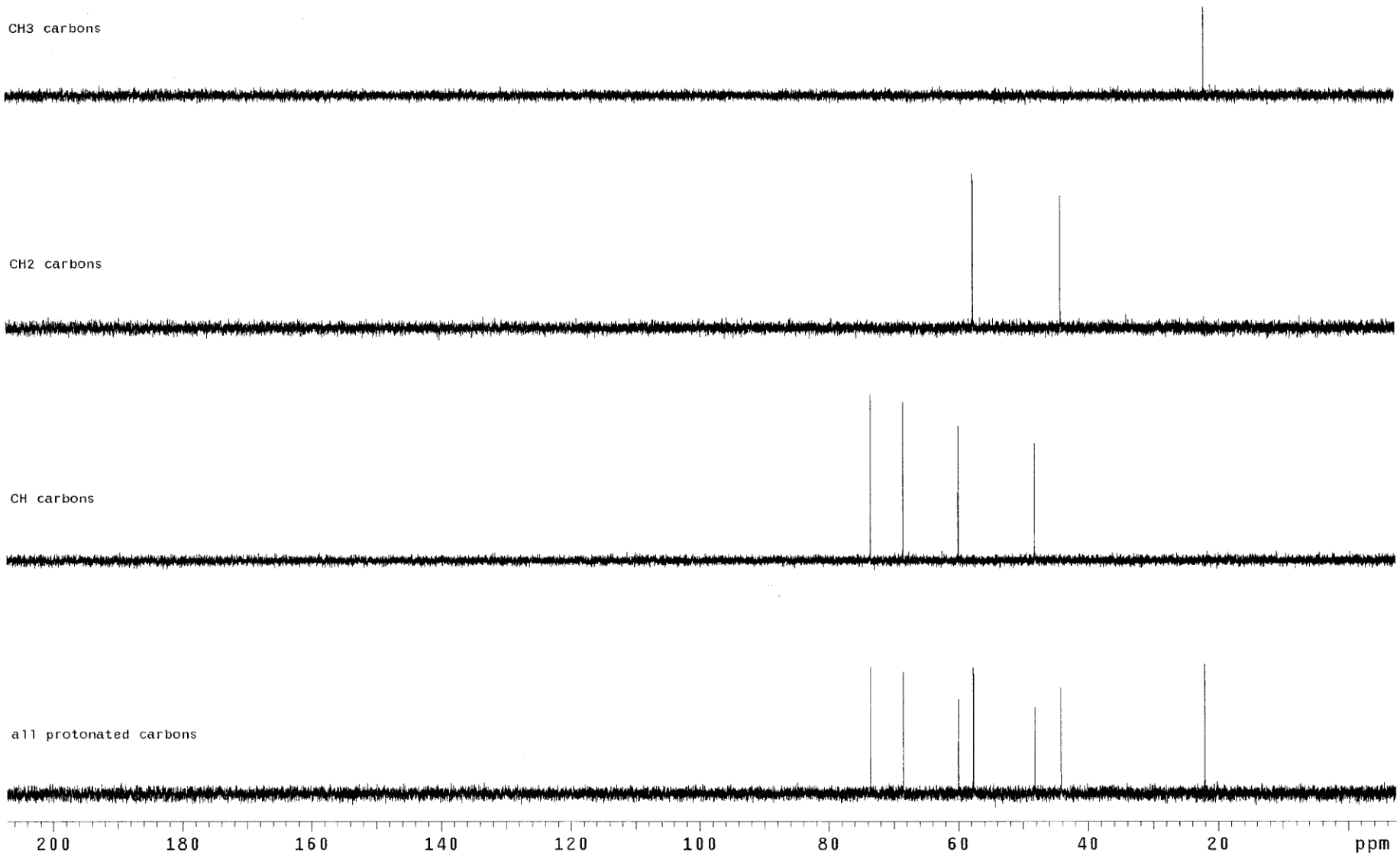


TY2-352

Pulse Sequence: hetcor
 Solvent: D2O
 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
 32 repetitions
 256 increments
OBSERVE C13, 125.6904822 MHz
DECOUPLE H1, 499.8652159 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, 46 min, 5 sec

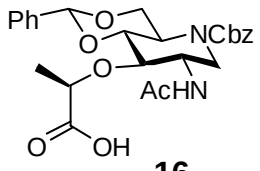



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

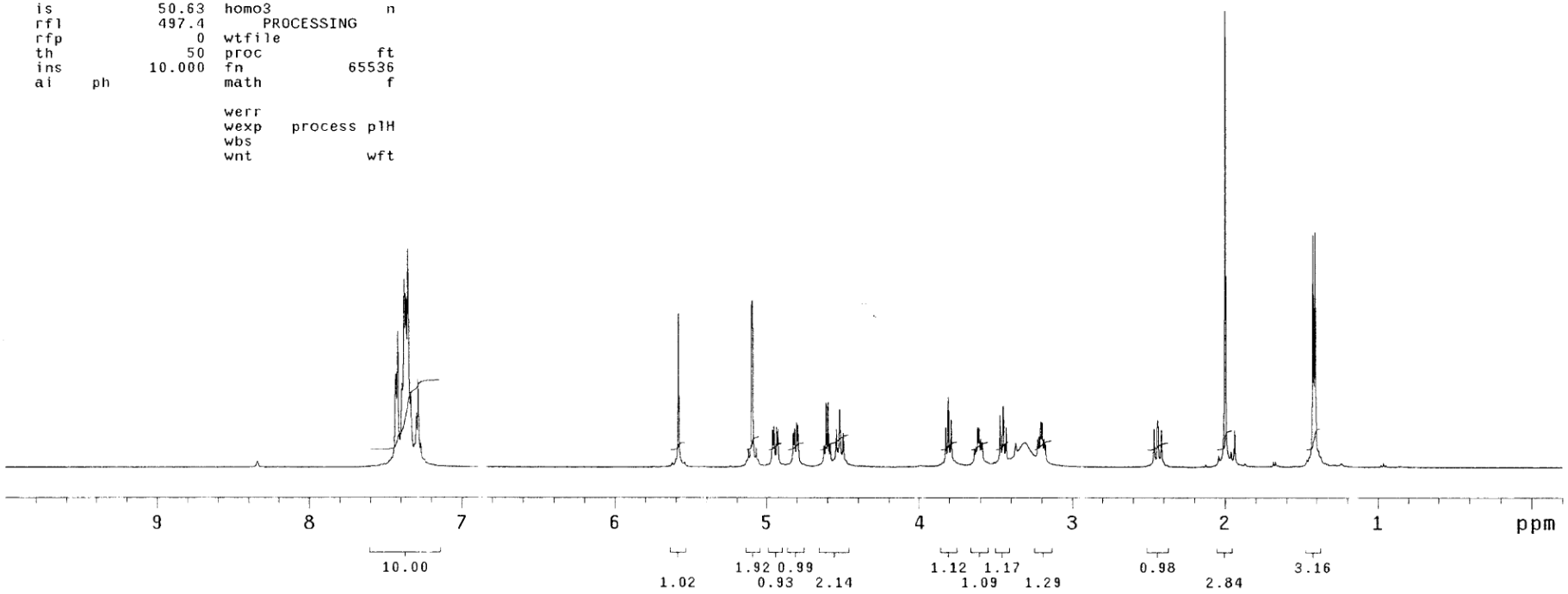
TY2-339

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 19 2009	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	-102.4	dof3	0
wp	5099.3	dm3	n
vs	50	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	50.63	homo3	n
rfl	497.4	PROCESSING	
rfp	0	wtfile	
th	50	proc	ft
ins	10.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



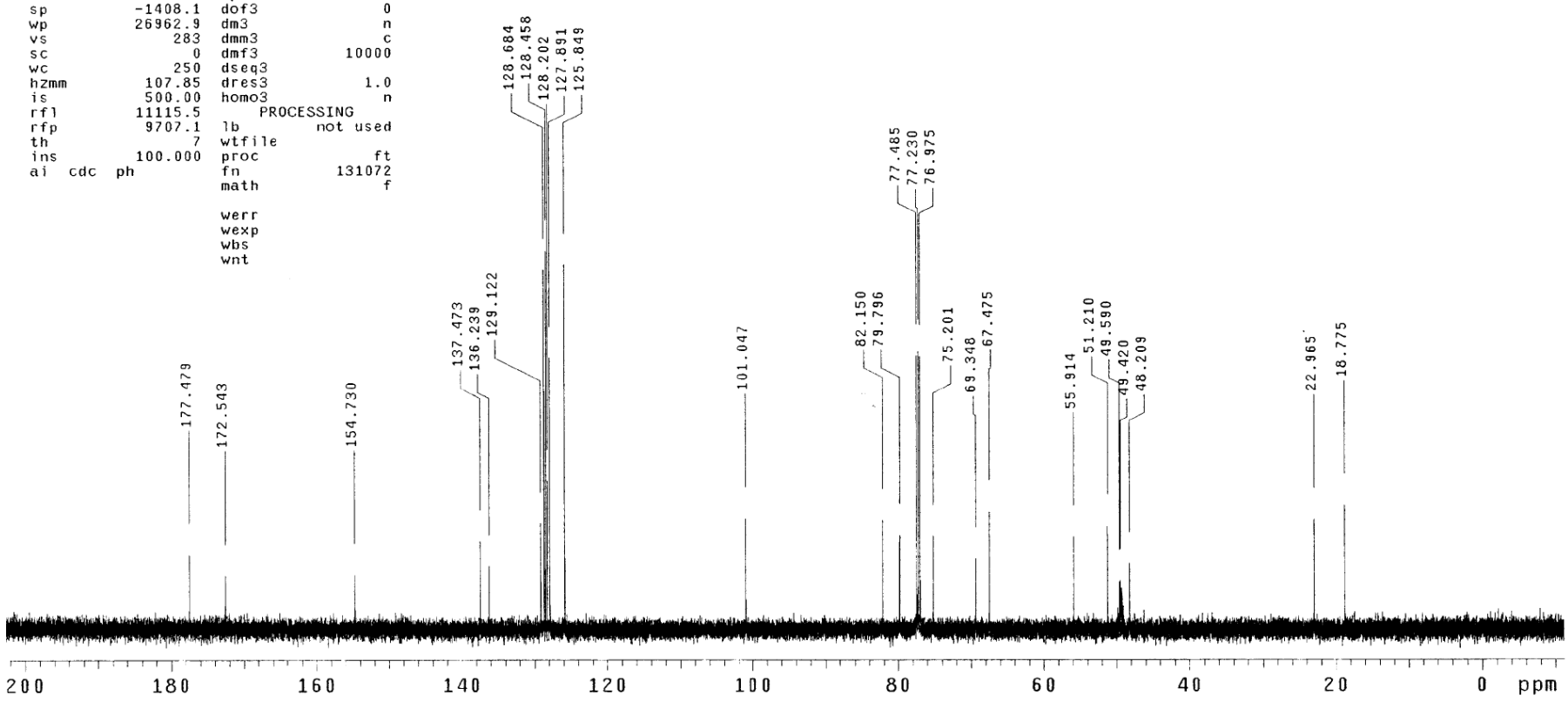
16



TY2-339

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 19 2009	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	5000	dm2	n
ct	264	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	-1408.1	dof3	0
wp	26962.9	dm3	n
vs	283	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11115.5	PROCESSING	
rfp	9707.1	lb	not used
th	7	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

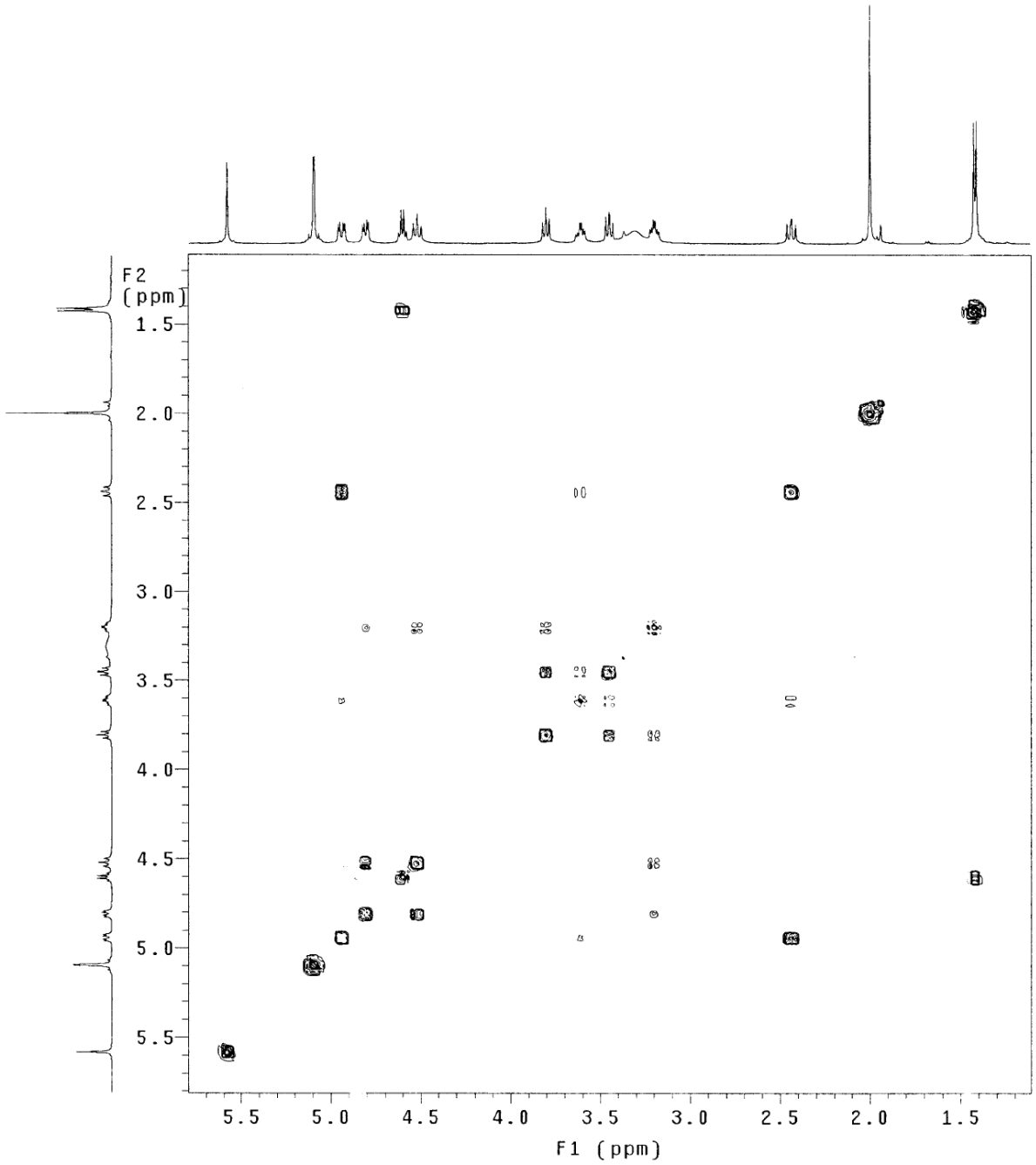


TY2-339

Pulse Sequence: relayh

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

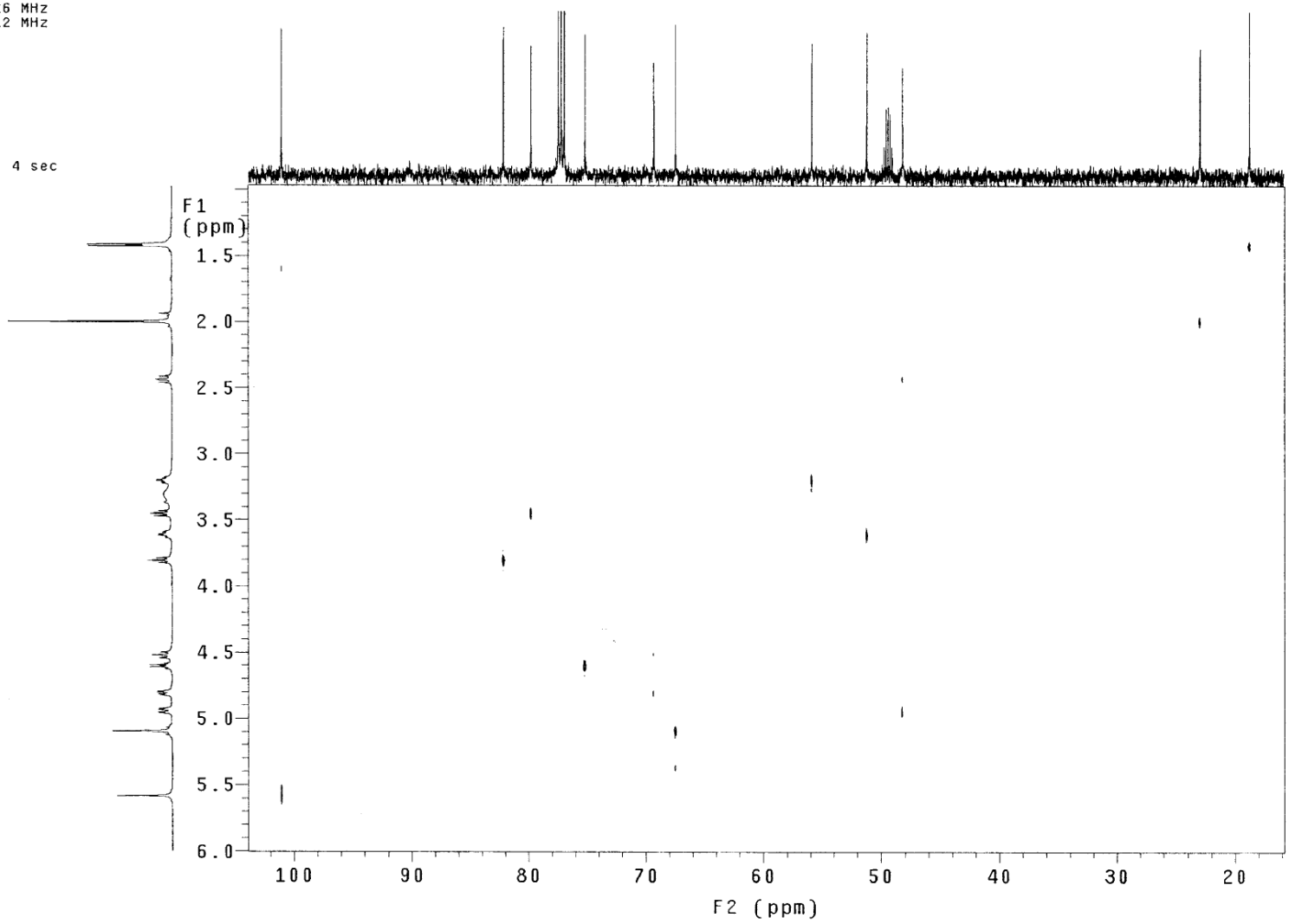
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611619 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

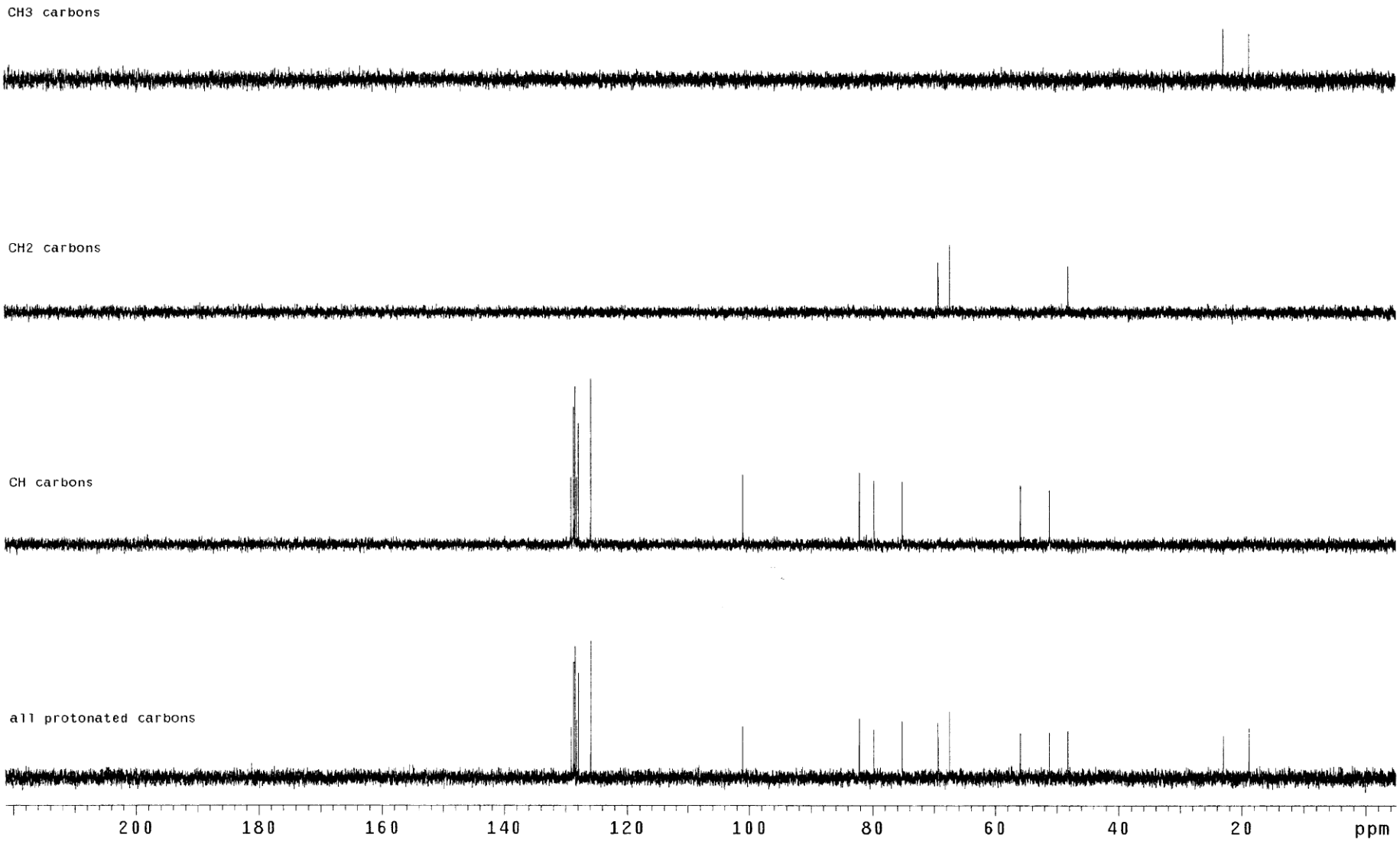


TY2-339

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
 64 repetitions
 256 increments
OBSERVE C13, 125.6901726 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 7 hr, 32 min, 4 sec

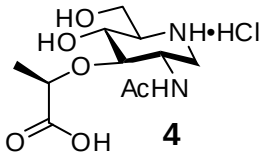



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

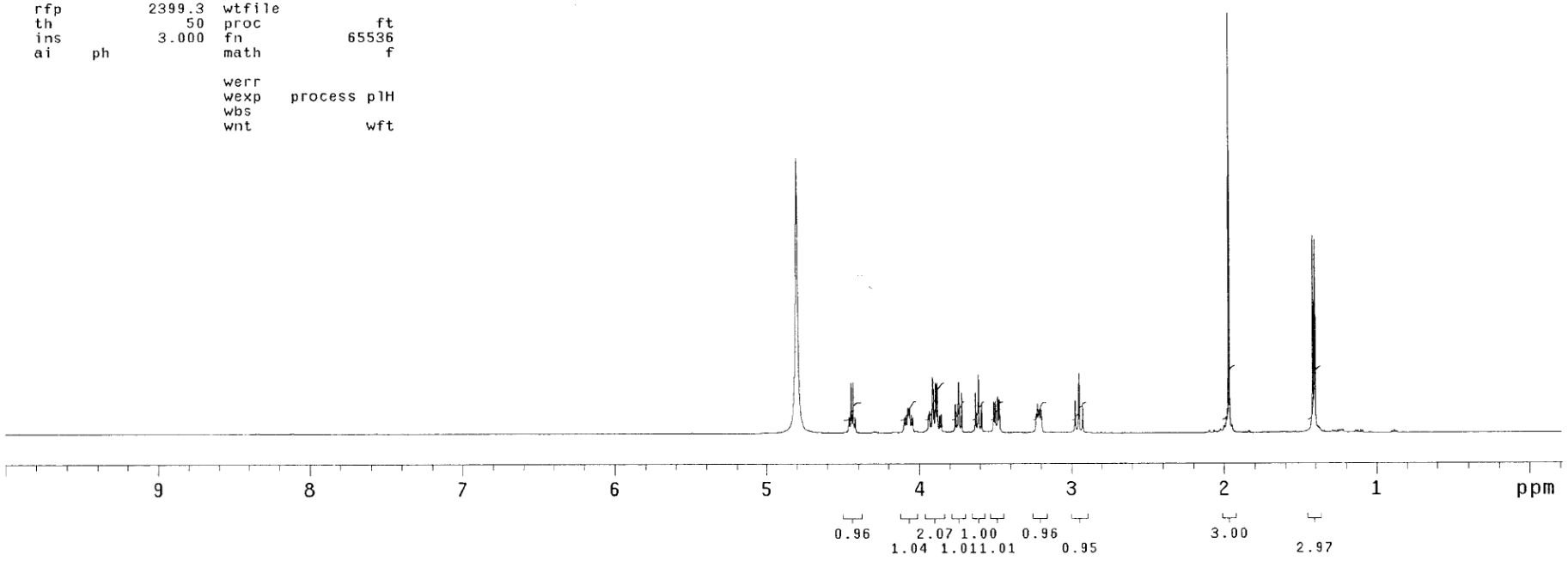
TY2-356

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 23 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.865	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	-106.7	dof3	0
wp	5107.9	dm3	n
vs	52	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	205.33	homo3	n
rfl	2832.4	PROCESSING	
rfp	2399.3	wtfile	
th	50	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



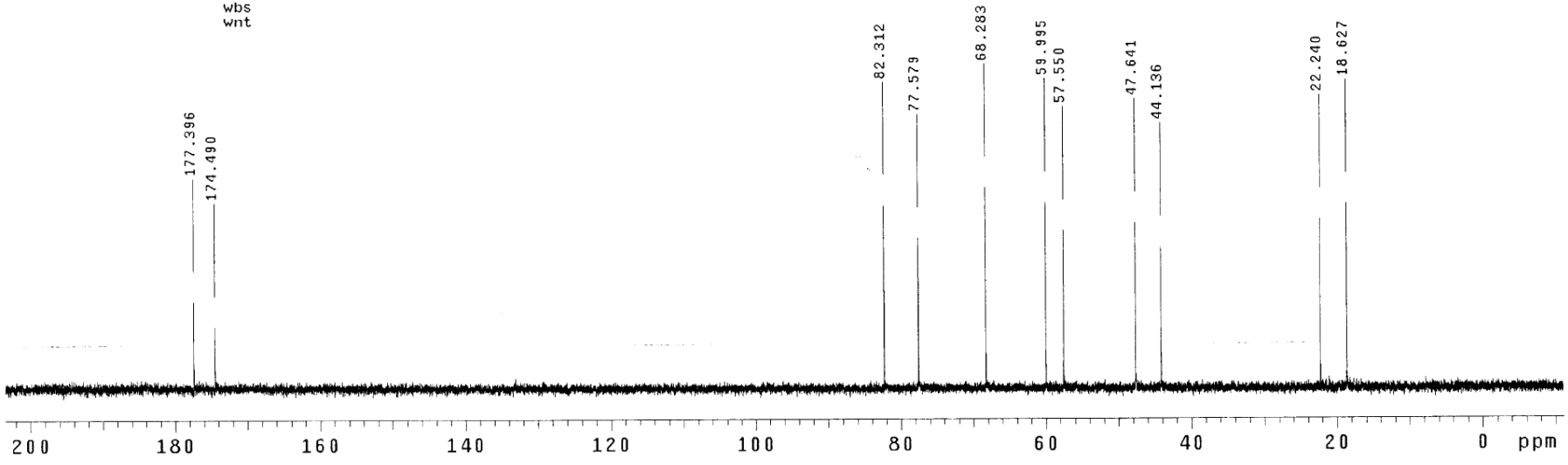
4



TY2-356

exp2 s2pul

SAMPLE		DEC. & VT	
date	May 23 2009	dfrq	499.865
solvent	D2O	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	2000	dm2	n
ct	133	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	-1394.6	dof3	0
wp	26962.9	dm3	n
vs	249	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	1395.0	PROCESSING	
rfp	0	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

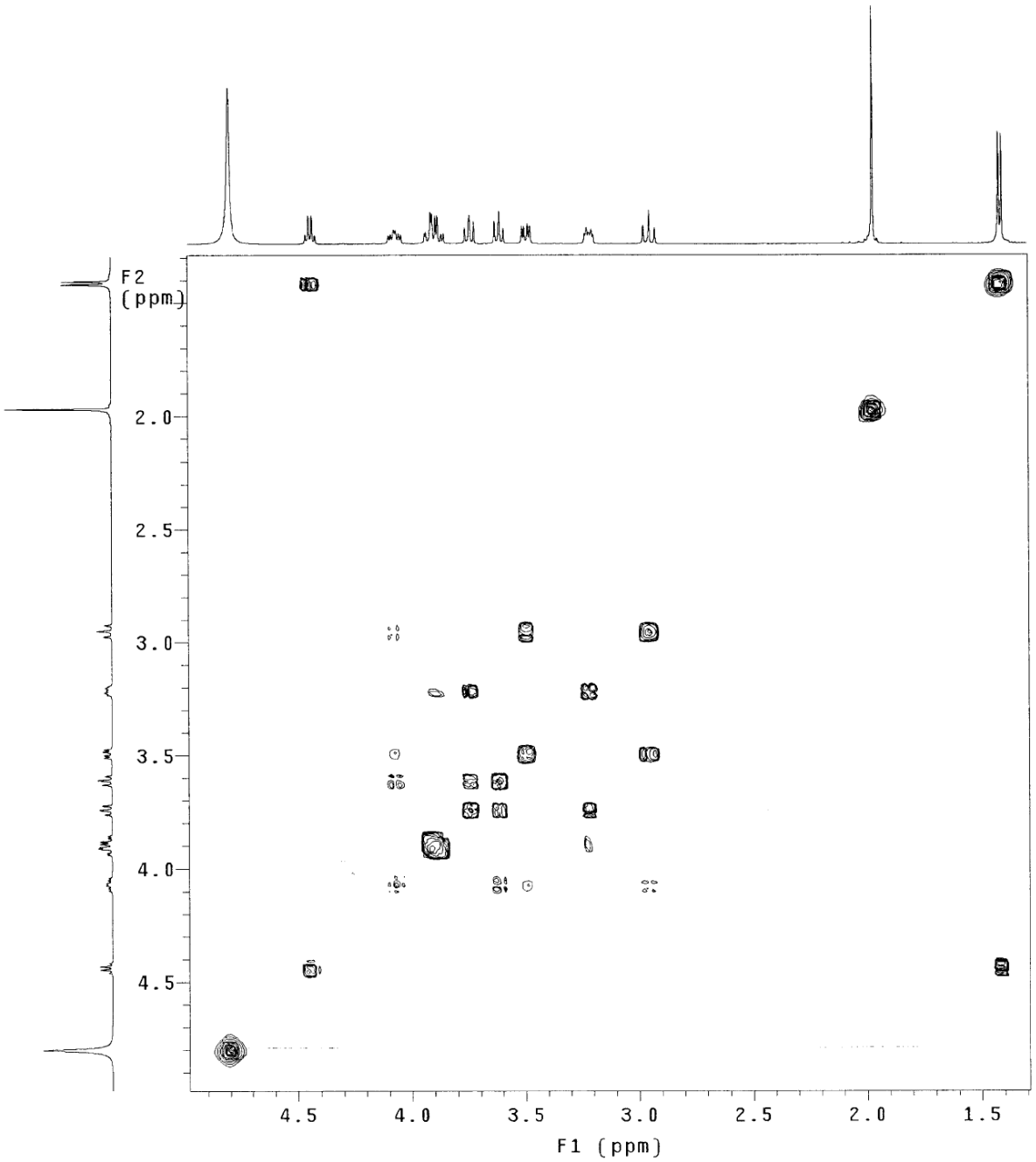


TY2-356

Pulse Sequence: relayh

Solvent: D2O
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

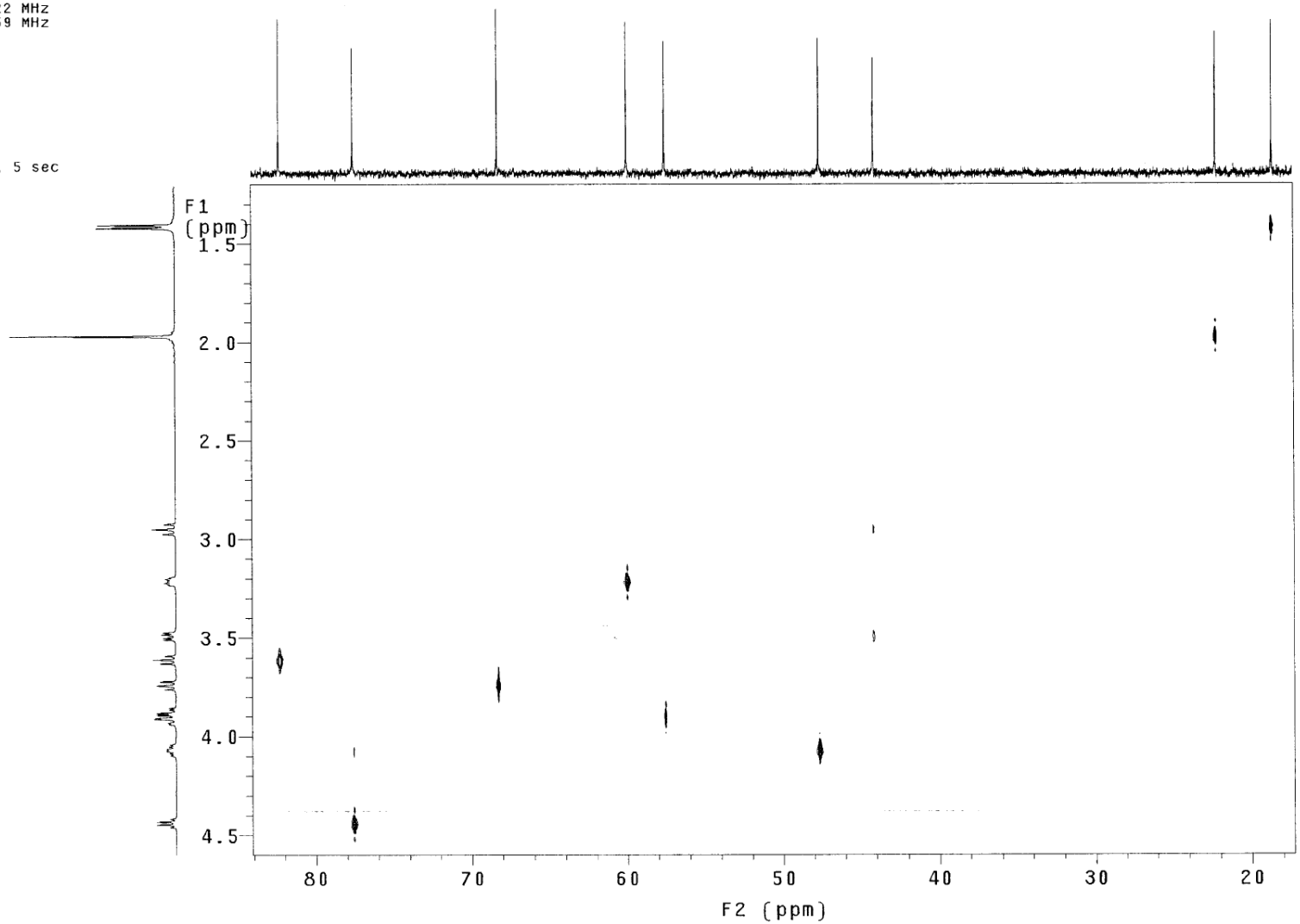
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8623823 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

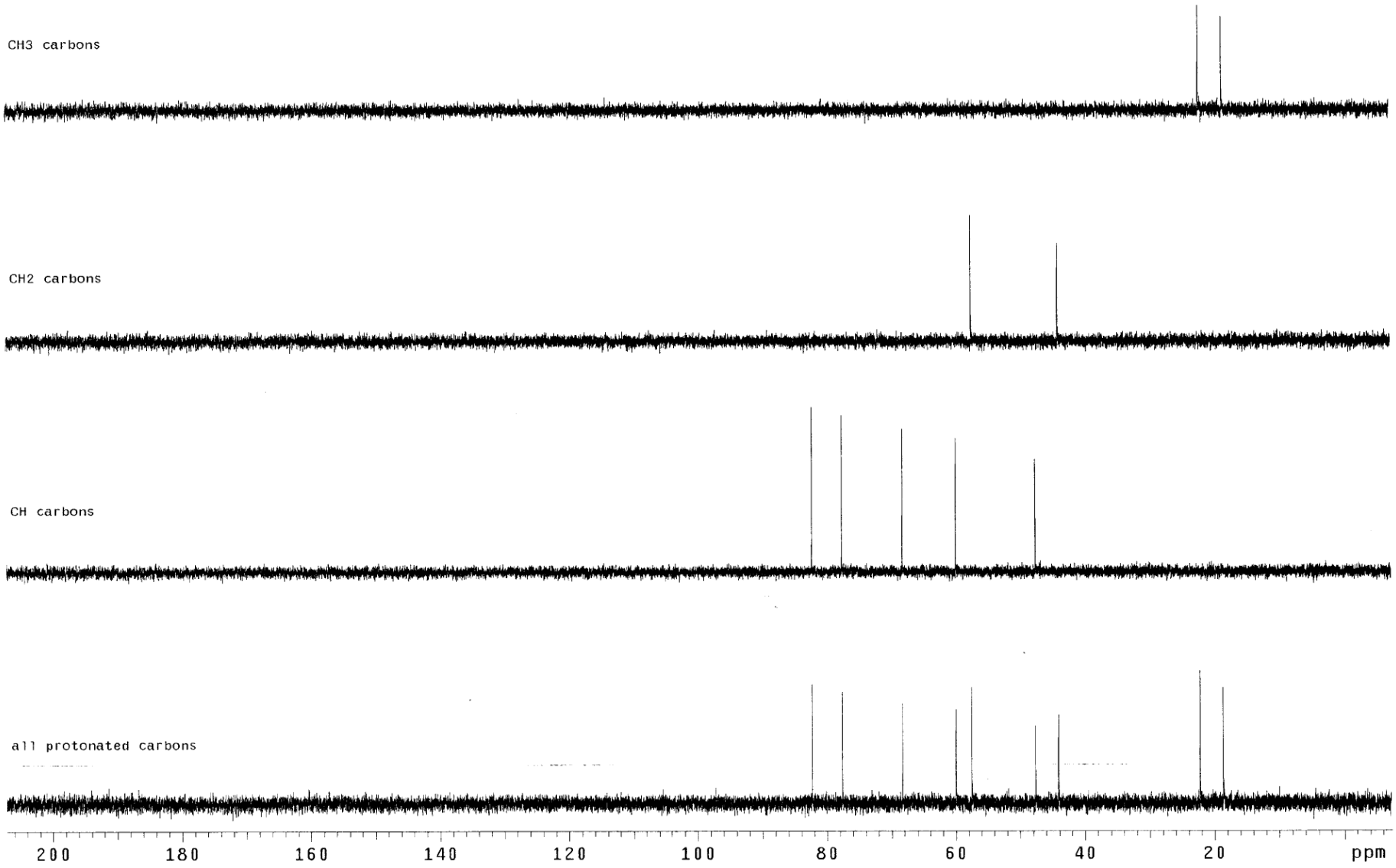


TY2-356

Pulse Sequence: hetcor
 Solvent: D2O
 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
 32 repetitions
 256 increments
OBSERVE C13, 125.6904822 MHz
DECOUPLE H1, 499.8652159 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, 46 min, 5 sec

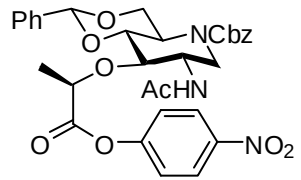



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

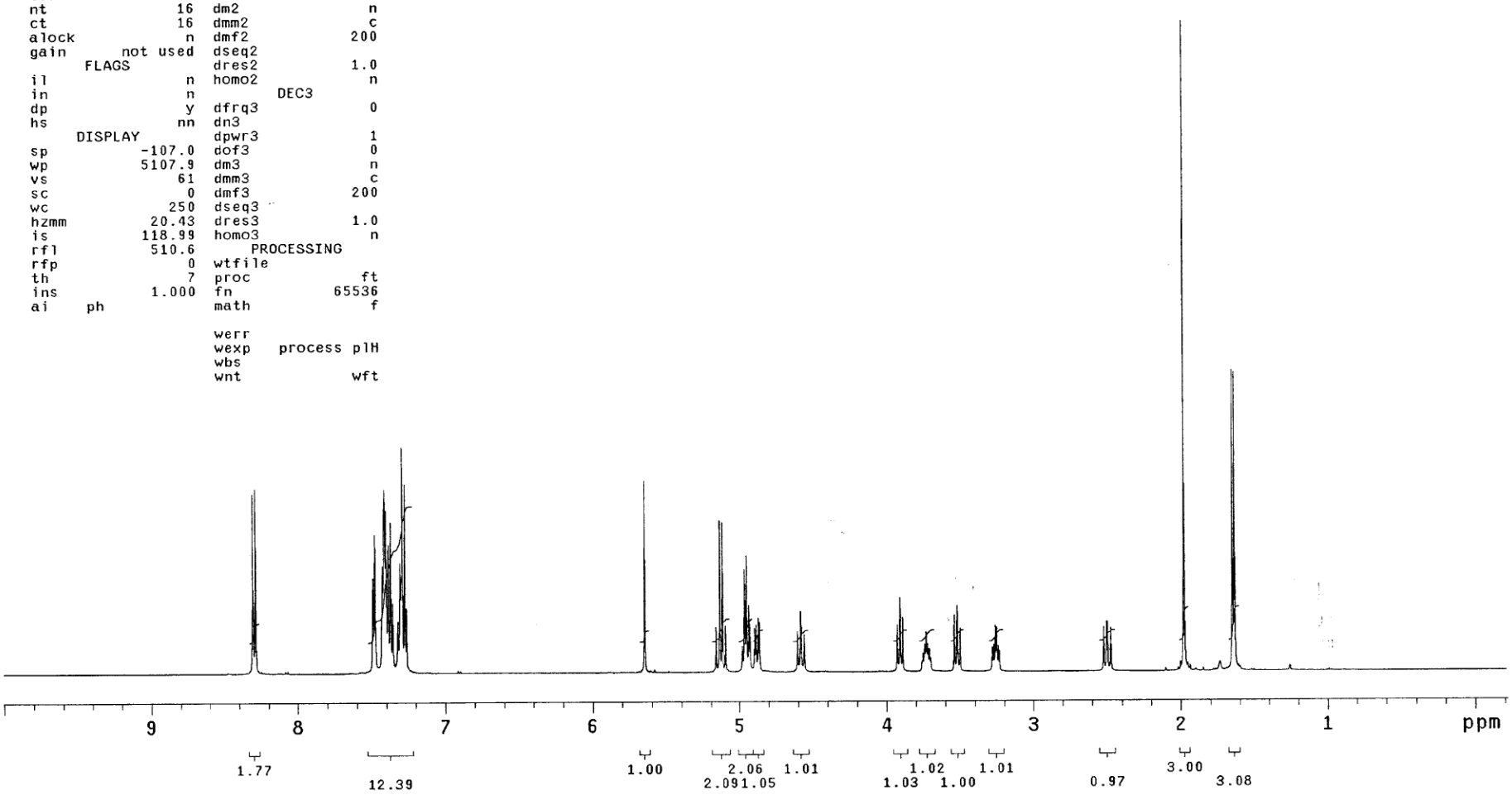
TY2-332

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 11 2009	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	-107.0	dof3	0
wp	5107.9	dm3	n
vs	61	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	118.99	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



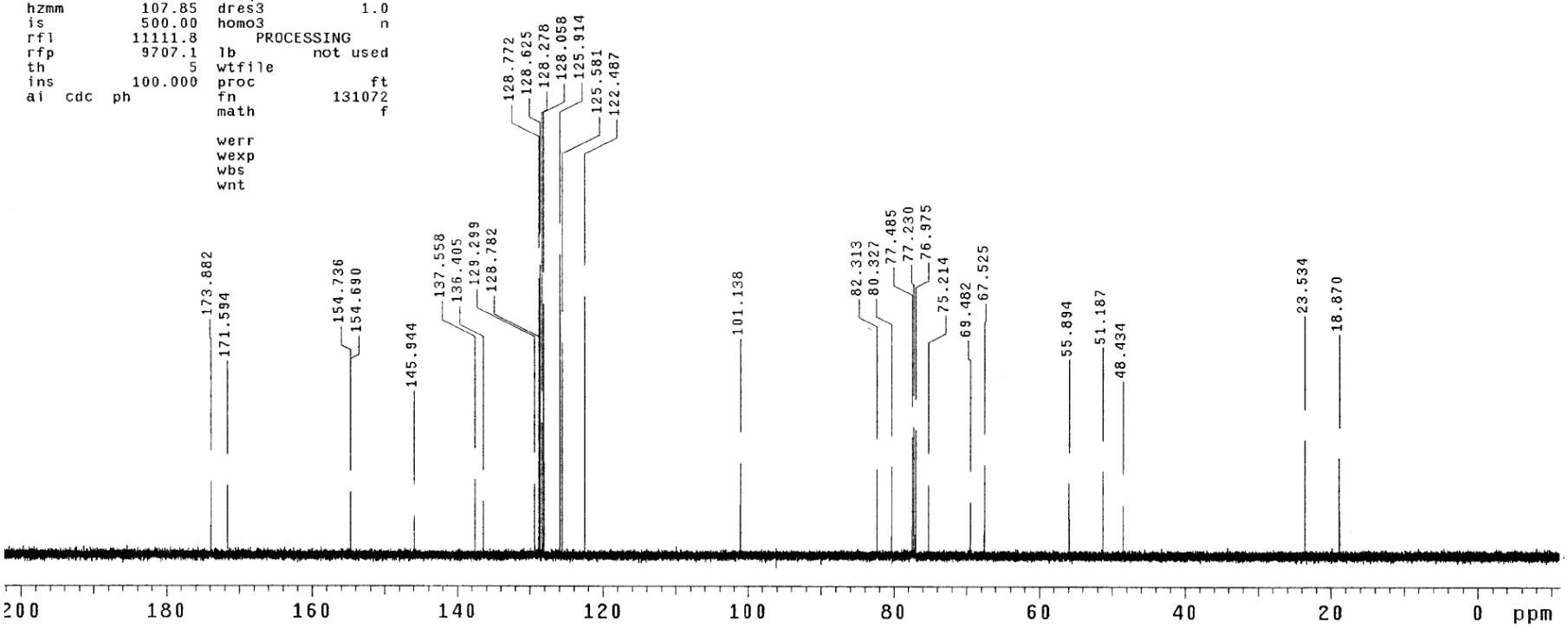
17



TY2-332

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 11 2009	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	3000	dm2	n
ct	203	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	-1404.4	dof3	0
wp	26962.9	dm3	n
vs	106	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11111.8	PROCESSING	
rfp	9707.1	lb	not used
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

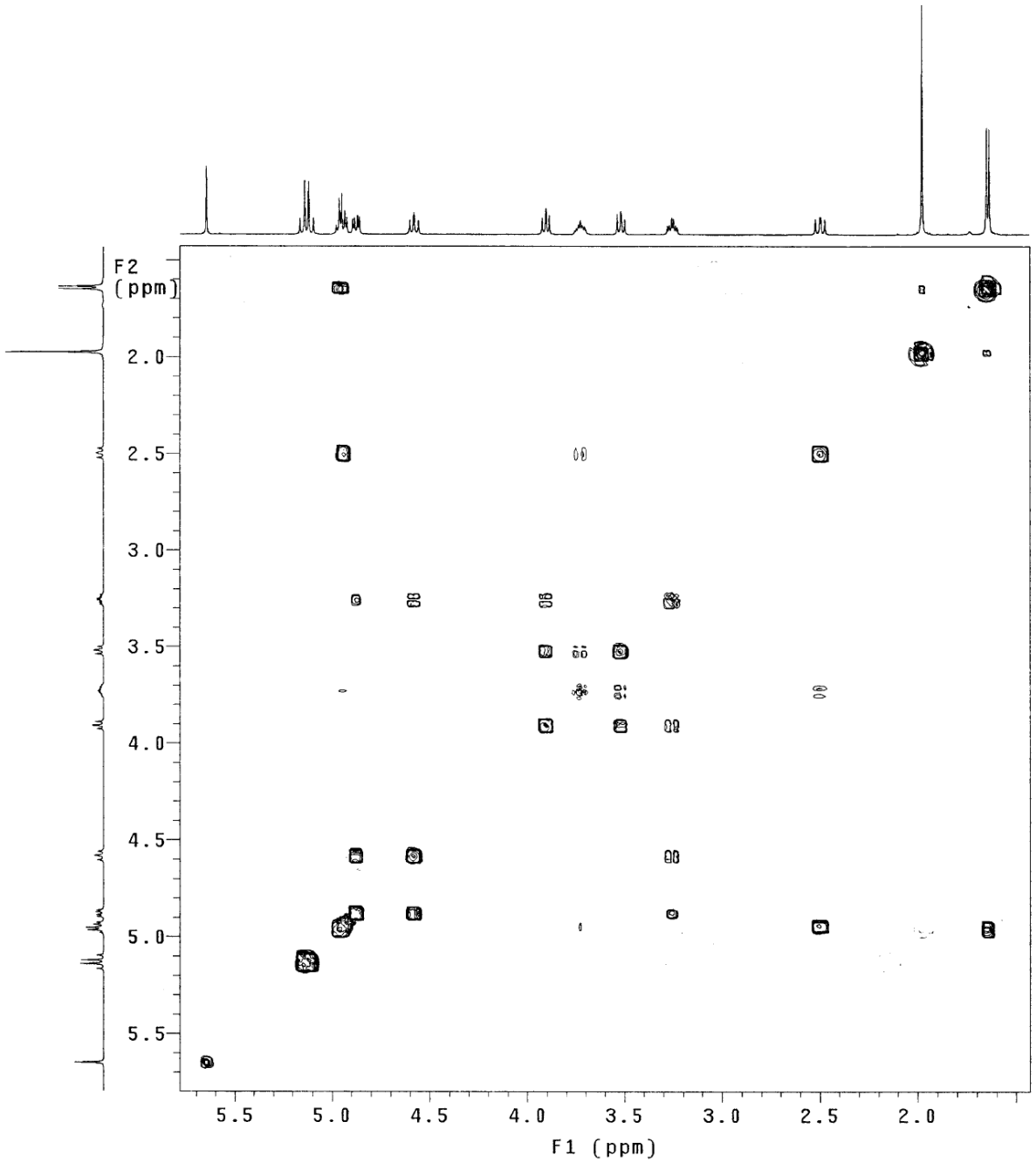


TY2-332

Pulse Sequence: relayh

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

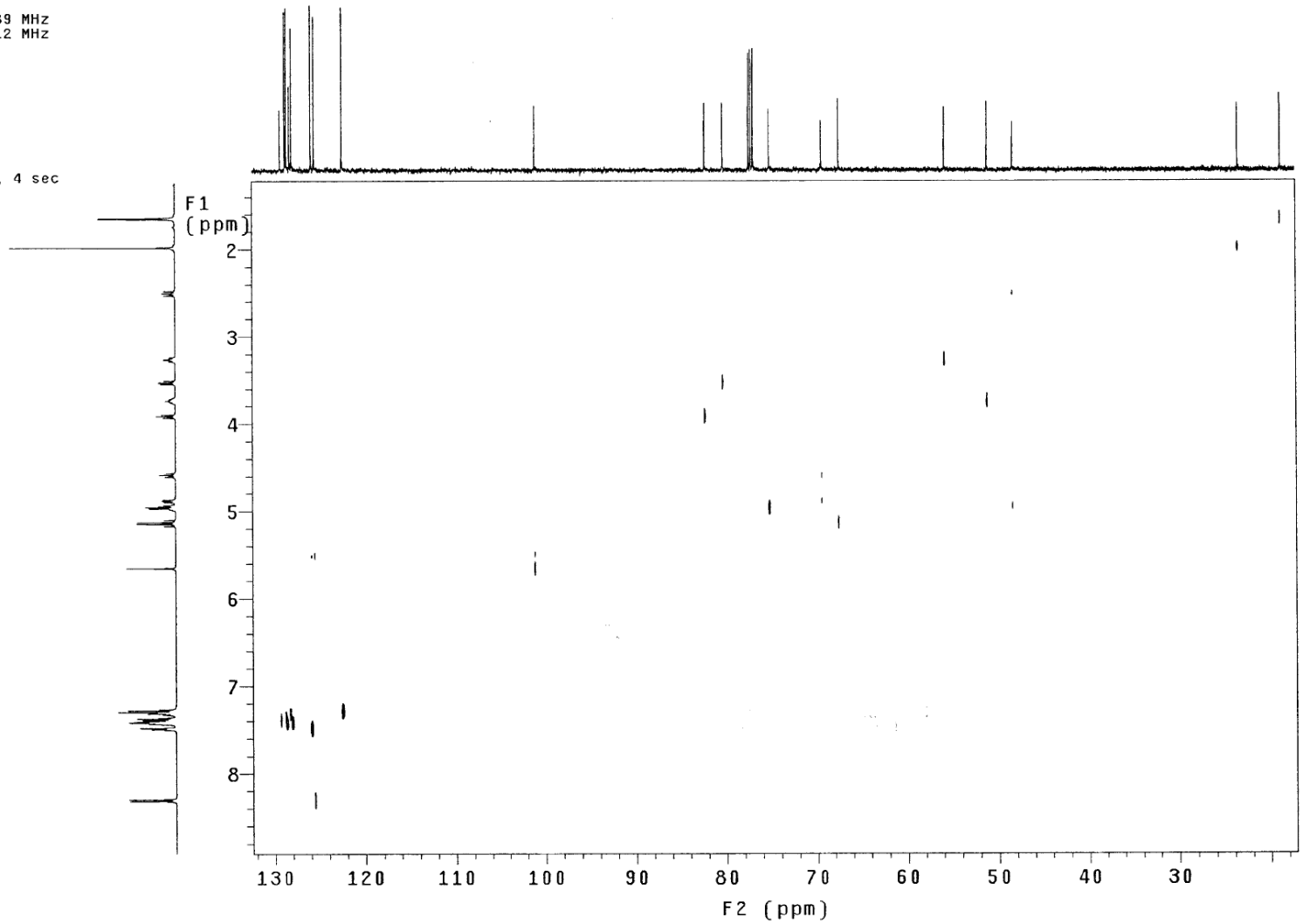
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

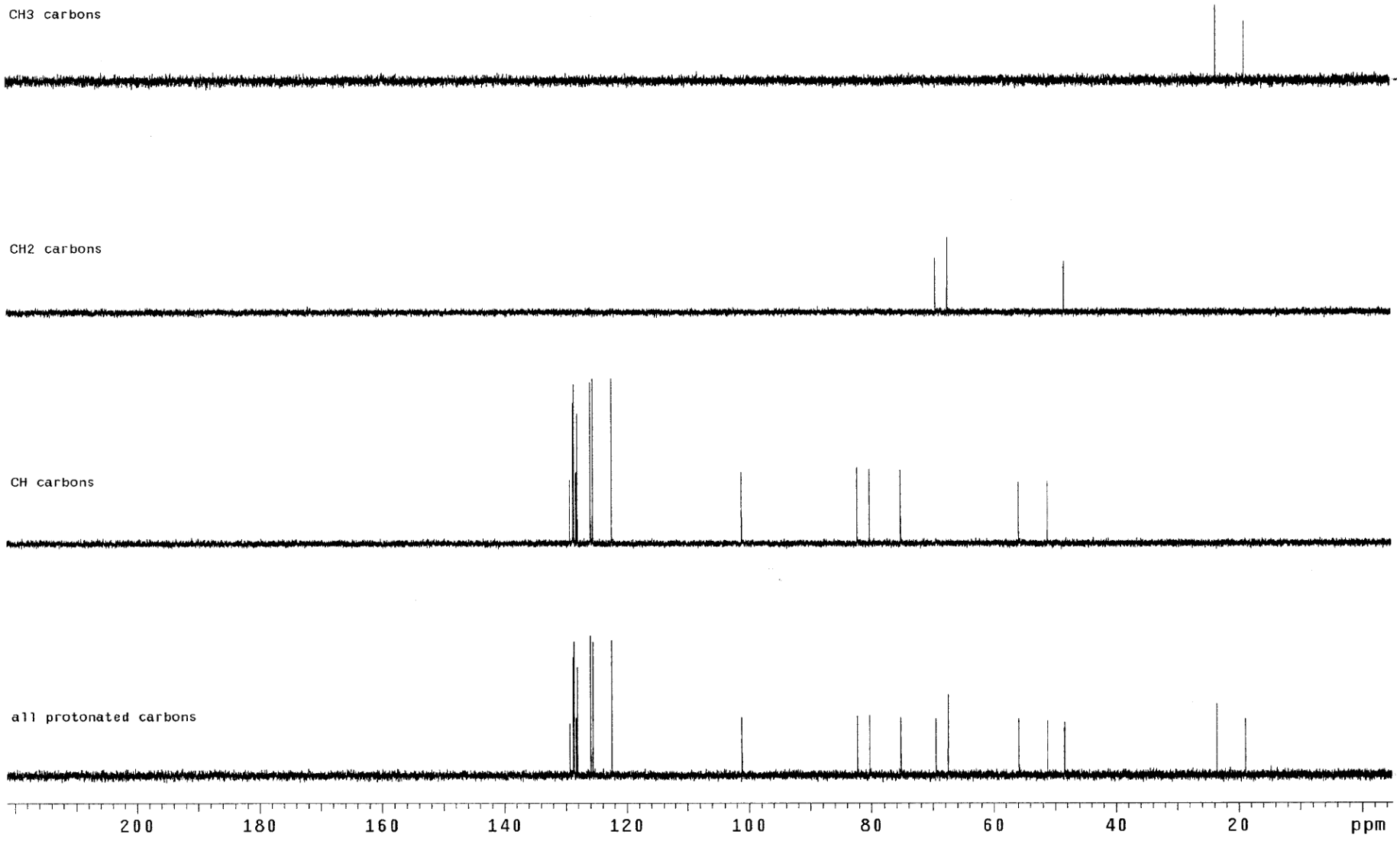


TY2-332

Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
File: TY2-332-CH
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
 64 repetitions
 256 increments
OBSERVE C13, 125.6901689 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 7 hr, 32 min, 4 sec

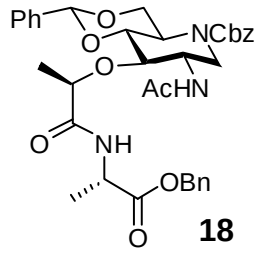



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

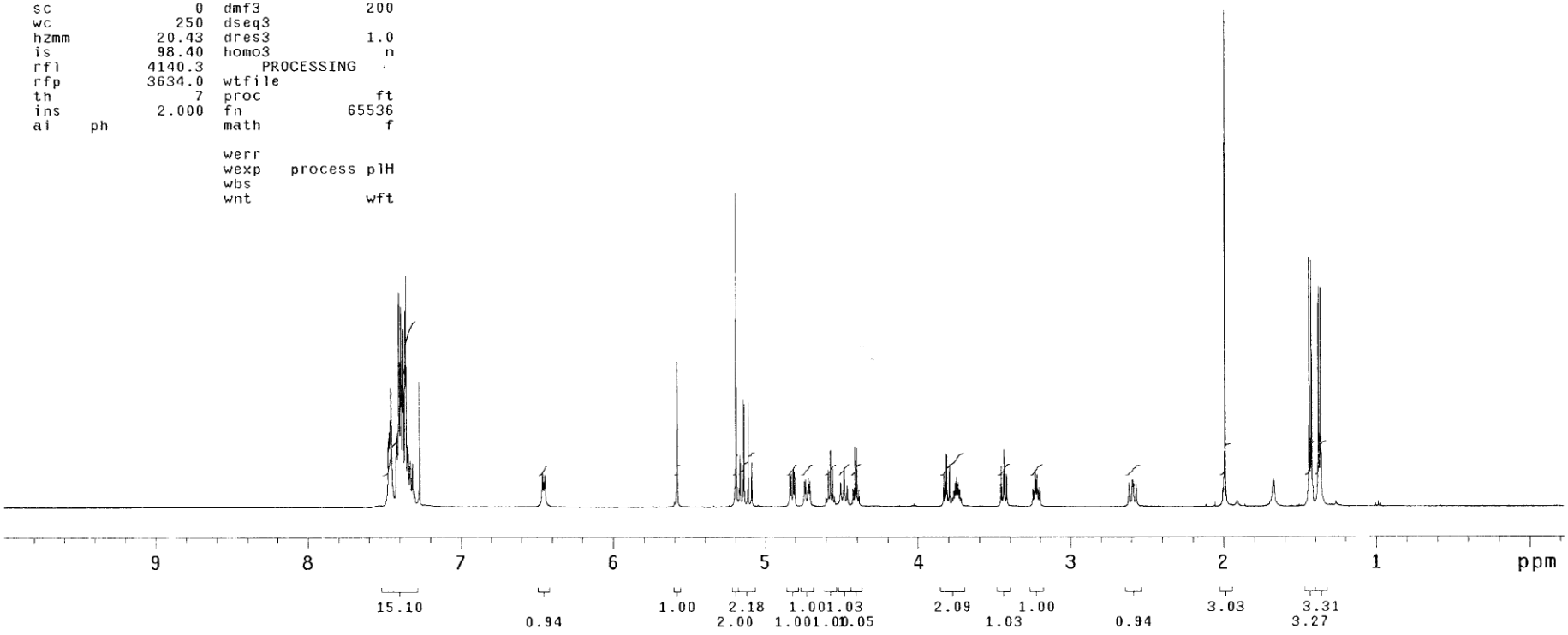
TY2-347

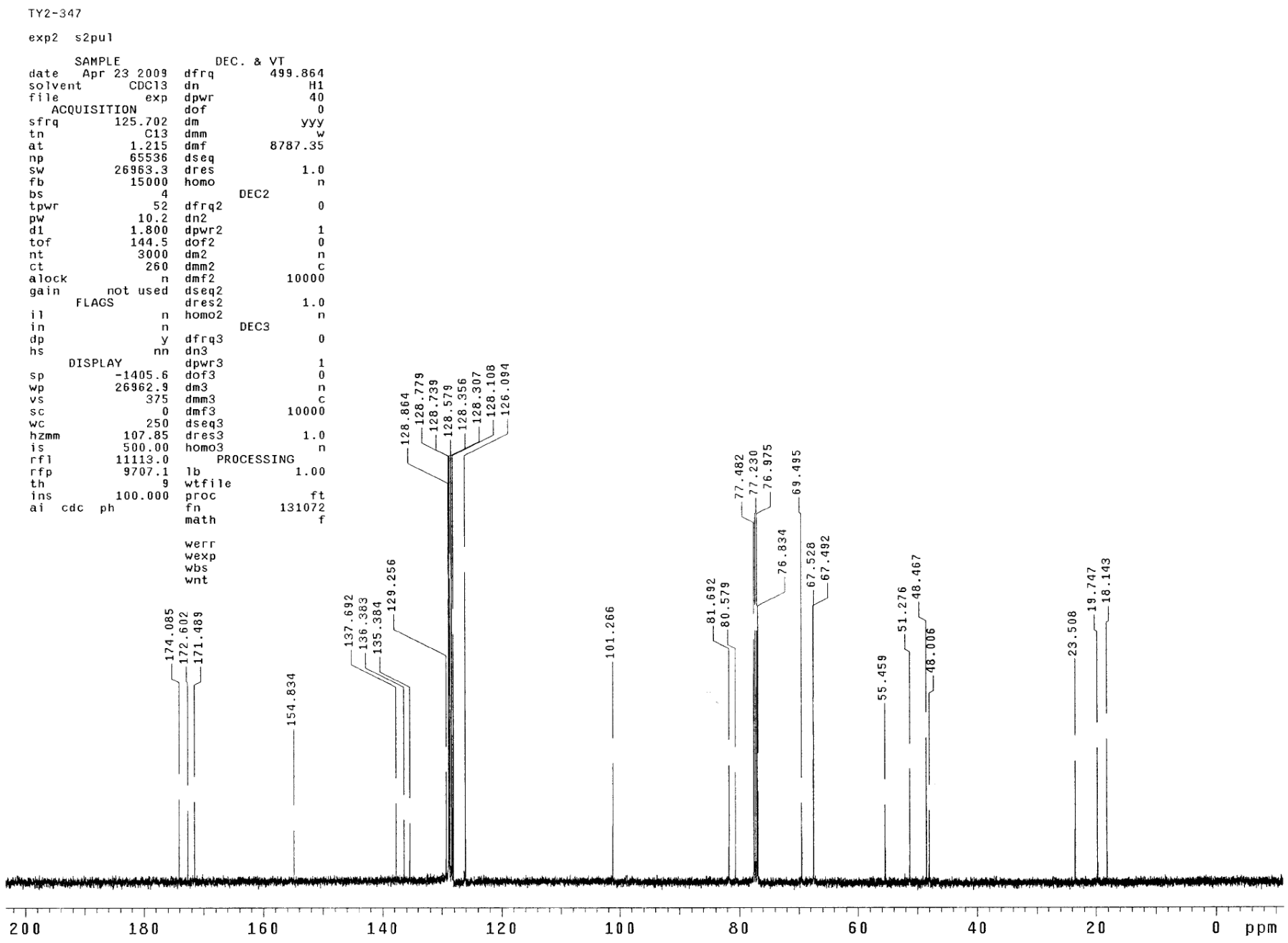
exp1 s2pul

SAMPLE		DEC. & VT	
date	May 8 2009	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	-111.4	dof3	0
wp	5107.9	dm3	n
vs	36	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	98.40	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	2.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



18



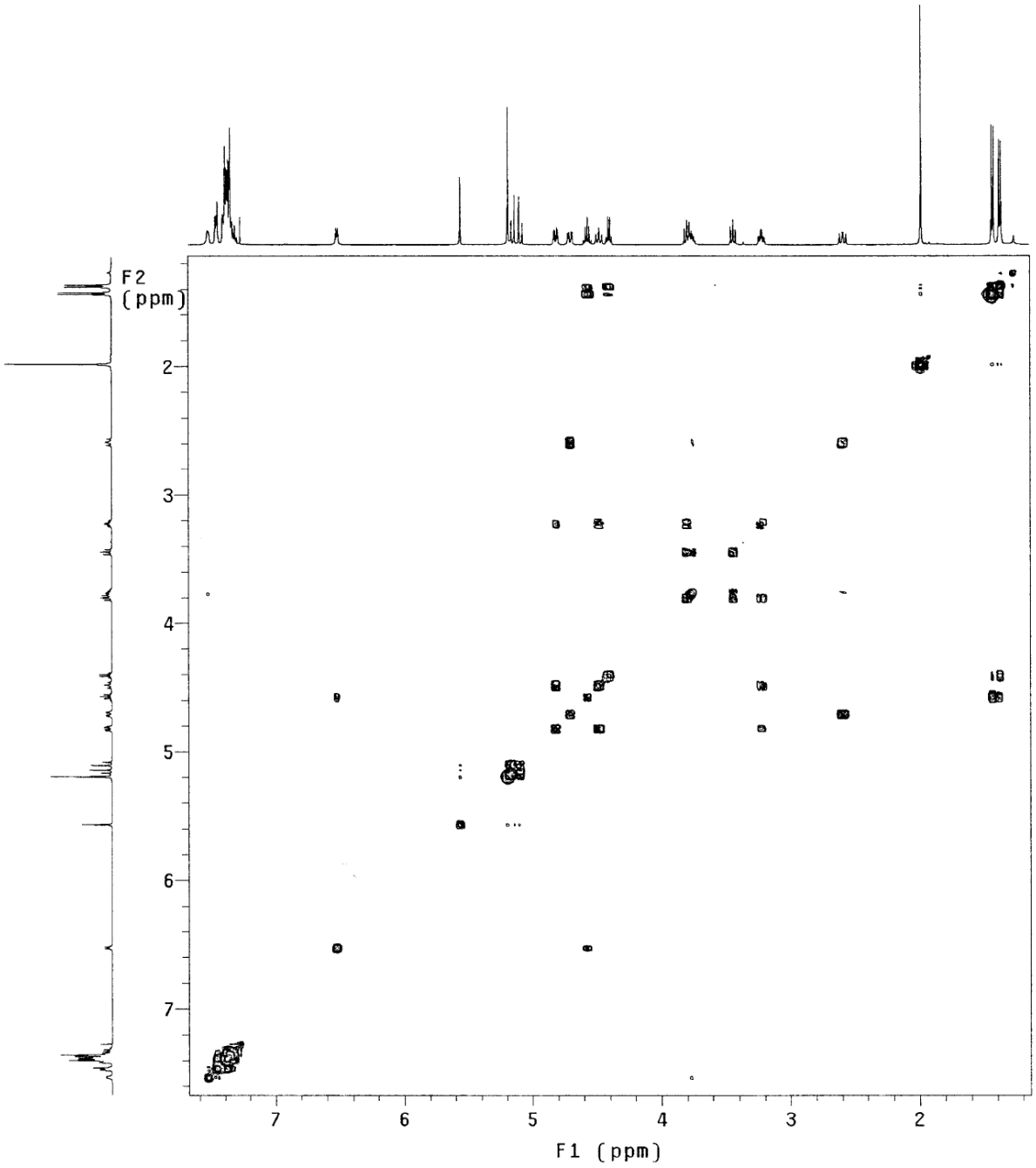

TY2-347
exp2 s2pul
SAMPLE
date Apr 23 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 3000
ct 260
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1405.6
wp 26962.9
vs 375
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11113.0
rfp 9707.1
th 9
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
174.085
172.602
171.489
154.834
137.692
136.383
135.384
129.256
128.864
128.779
128.739
128.579
128.356
128.307
128.108
126.094
101.266
81.692
80.579
77.482
77.230
76.975
76.834
69.495
67.528
67.492
55.459
51.276
48.467
48.006
23.508
19.747
18.143
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-347

Pulse Sequence: relayh

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

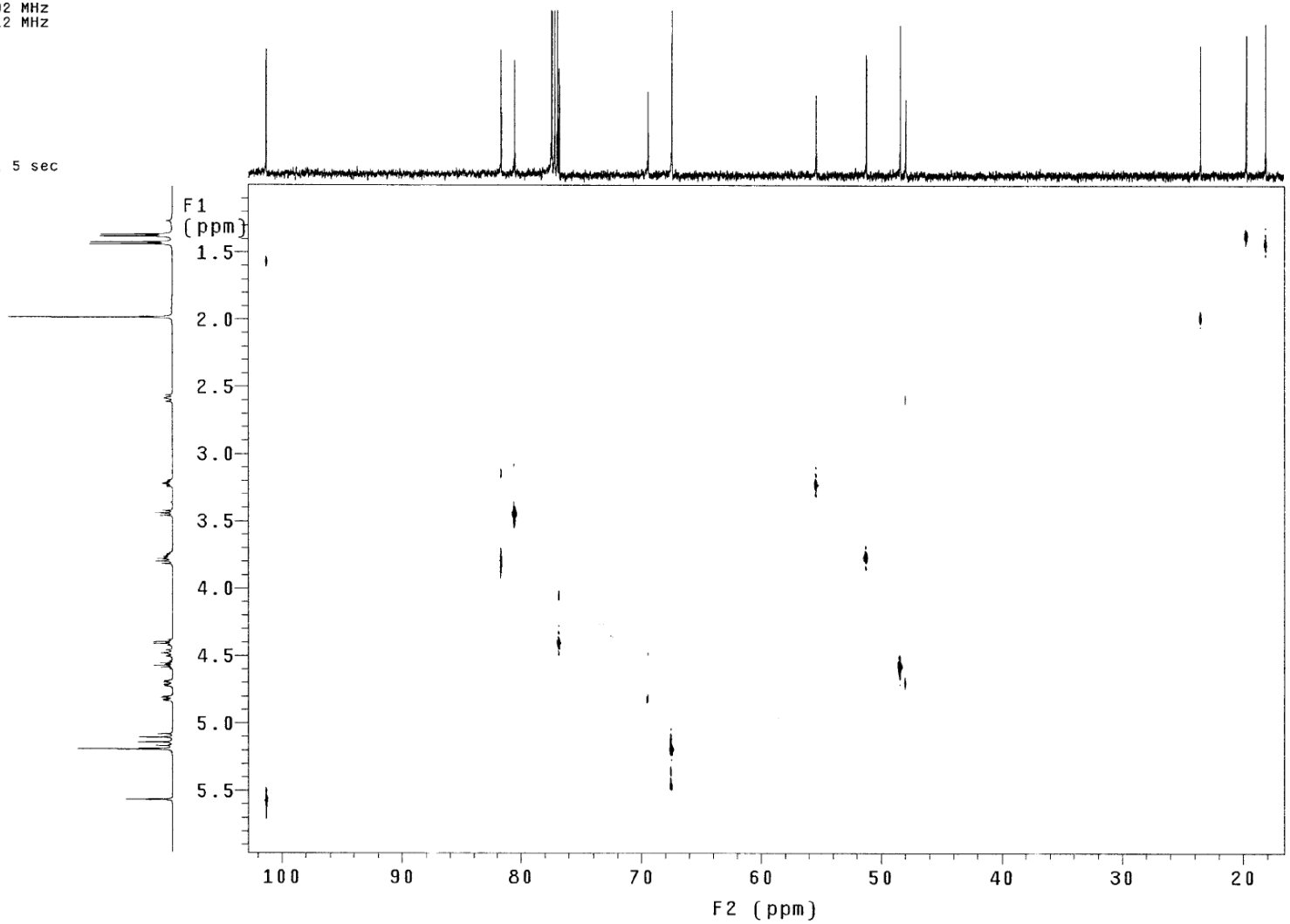
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611707 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

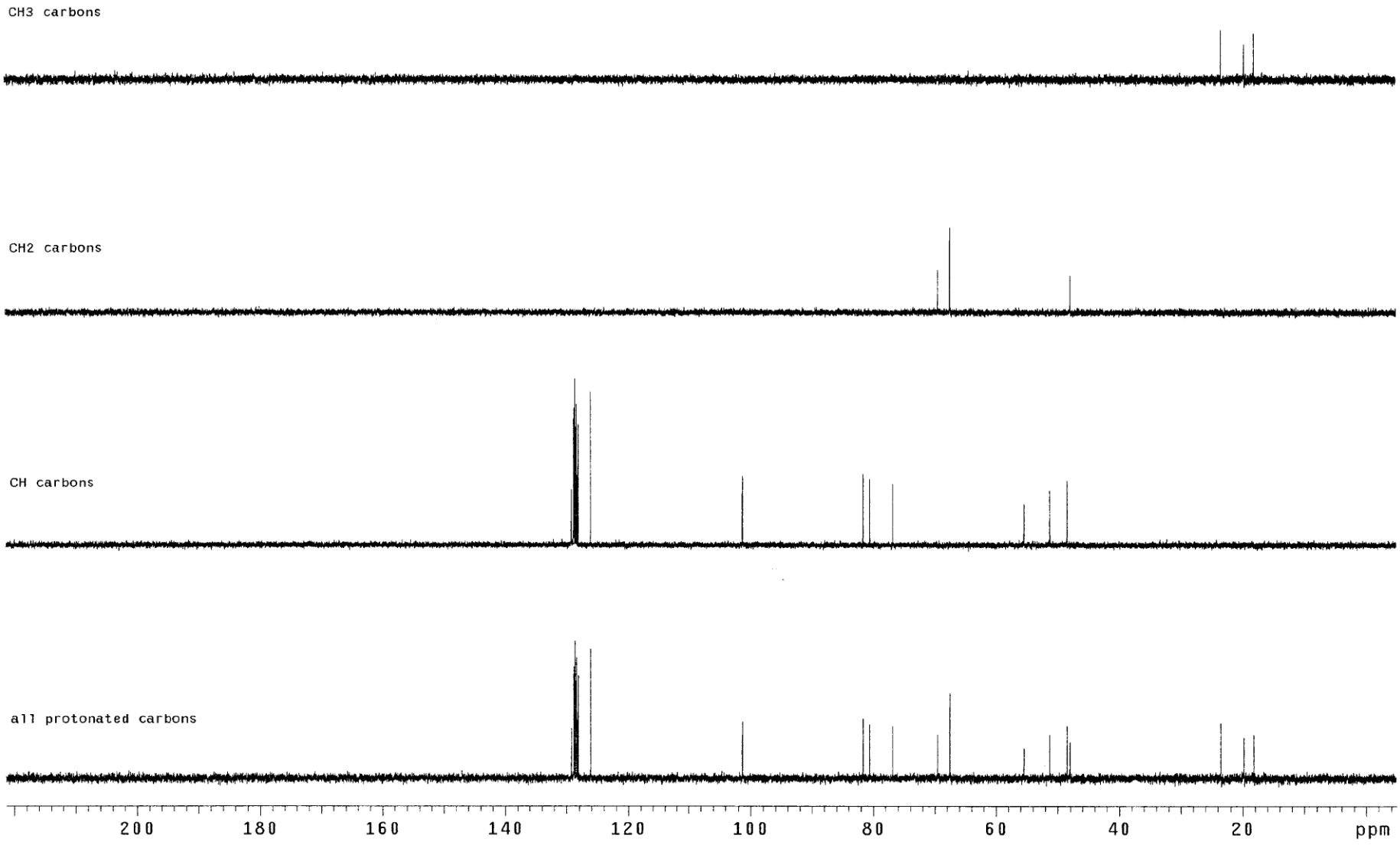


TY2-347

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
 32 repetitions
 256 increments
OBSERVE C13, 125.6901702 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 3 hr, 46 min, 5 sec

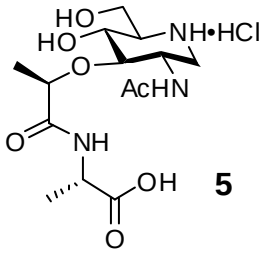



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

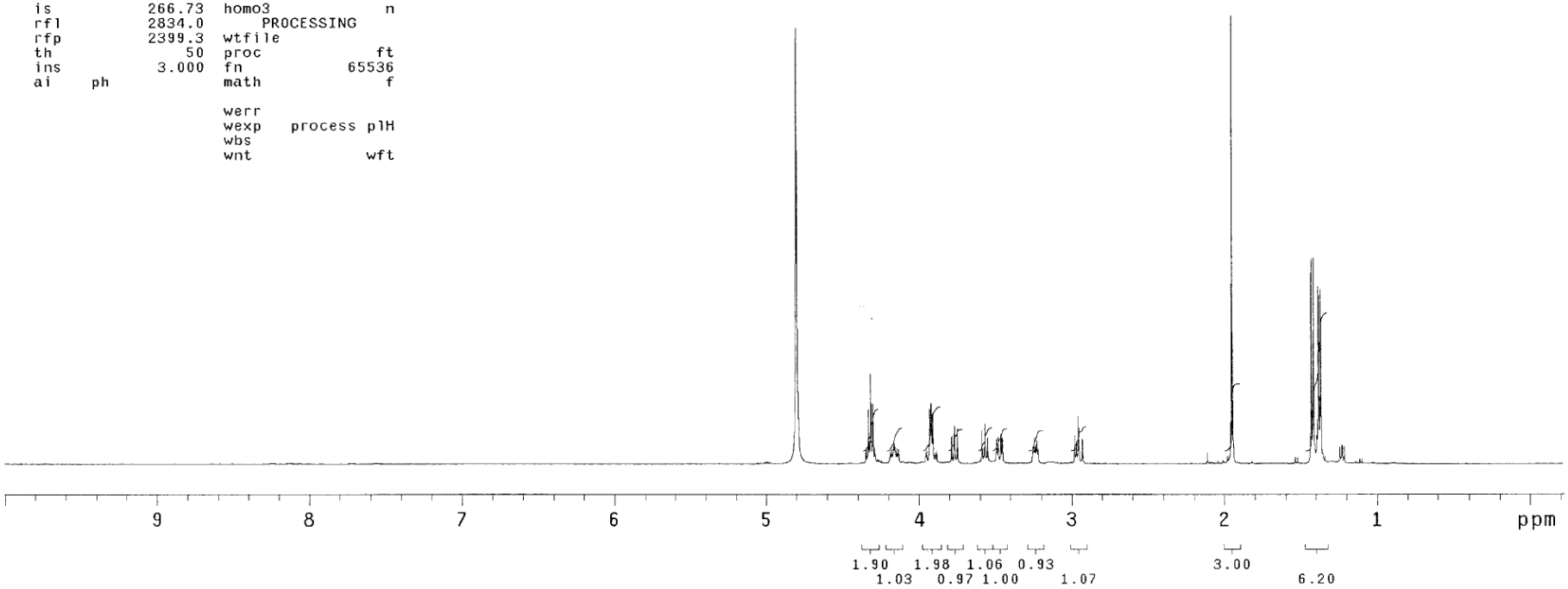
TY2-357

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 28 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.865	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	-108.3	dof3	0
wp	5107.9	dm3	n
vs	41	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	266.73	homo3	n
rfl	2834.0	PROCESSING	
rfp	2399.3	wtfile	
th	50	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



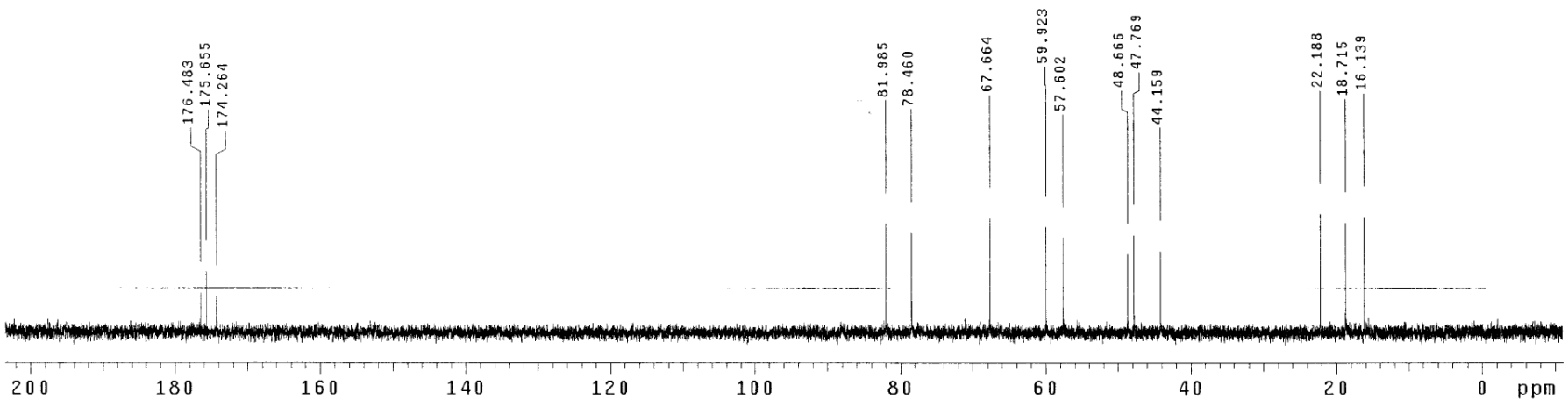
5



TY2-357

exp2 s2pul

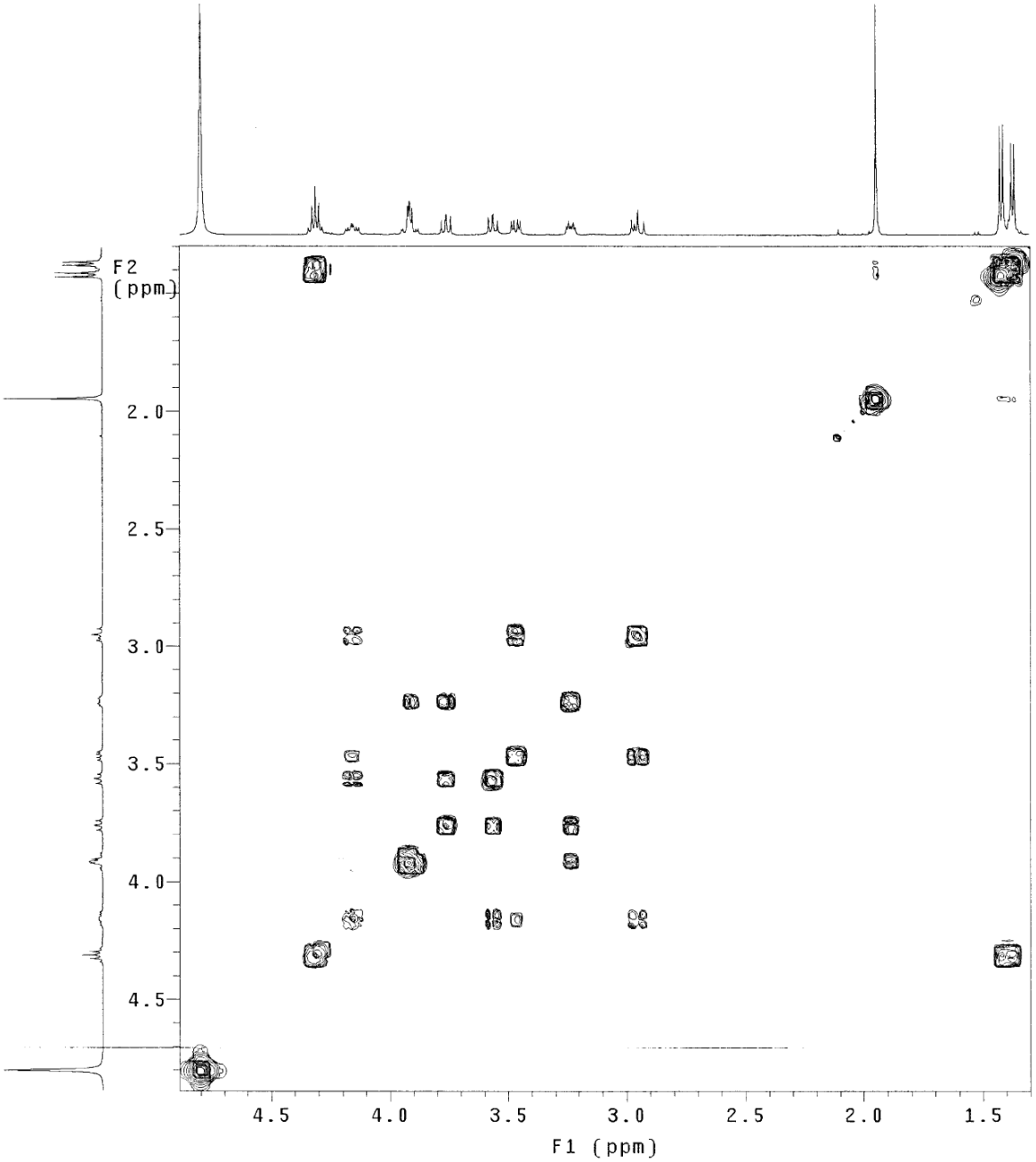
SAMPLE		DEC. & VT	
date	May 28 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	3000	dm2	n
ct	160	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	-1394.6	dof3	0
wp	26962.9	dm3	n
vs	363	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	1395.0	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	



TY2-357

Pulse Sequence: relayh
 Solvent: D2O
 Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

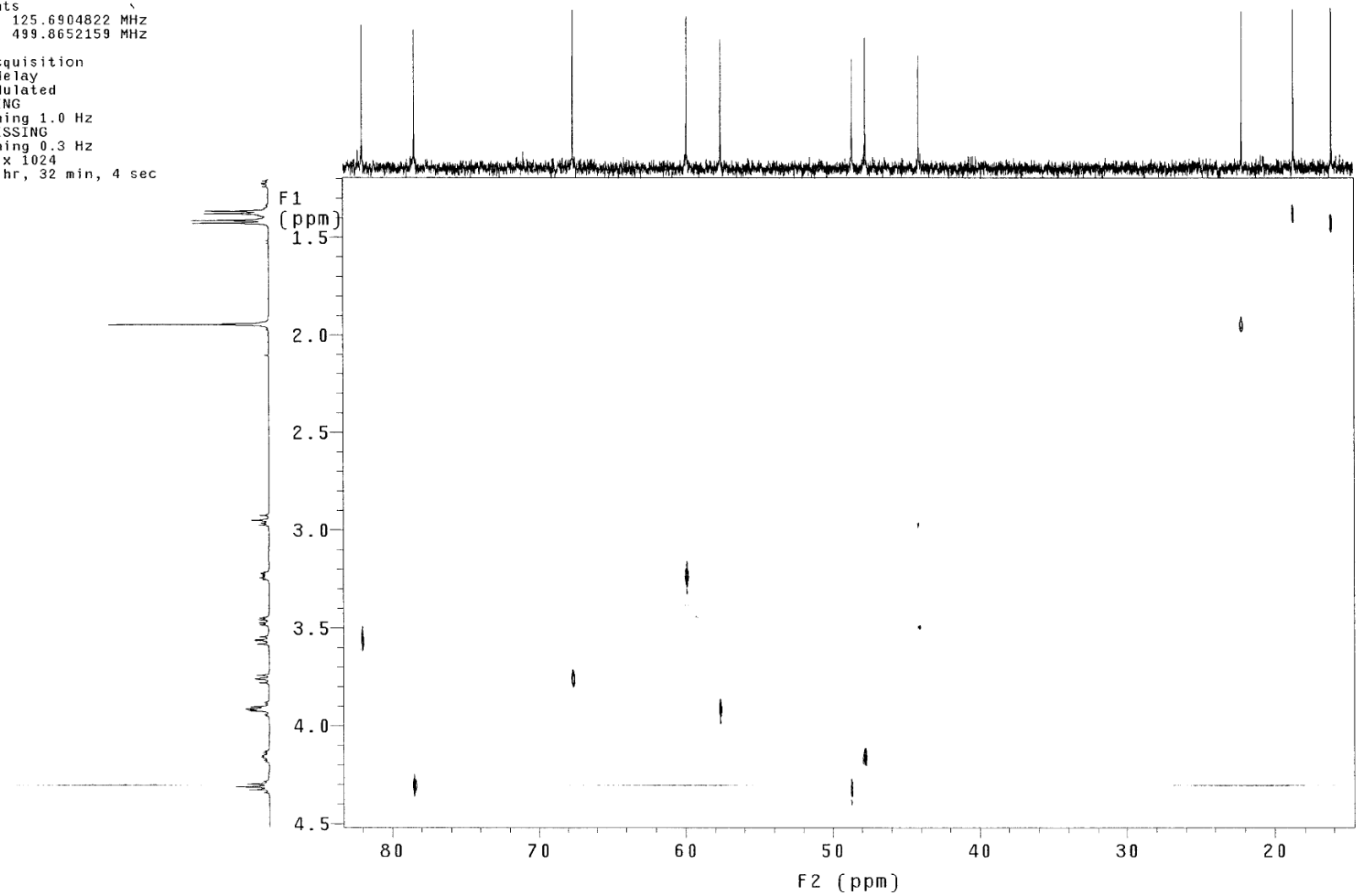
 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 64 repetitions
 256 increments
OBSERVE H1, 499.8623841 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 6 hr, 46 min, 8 sec

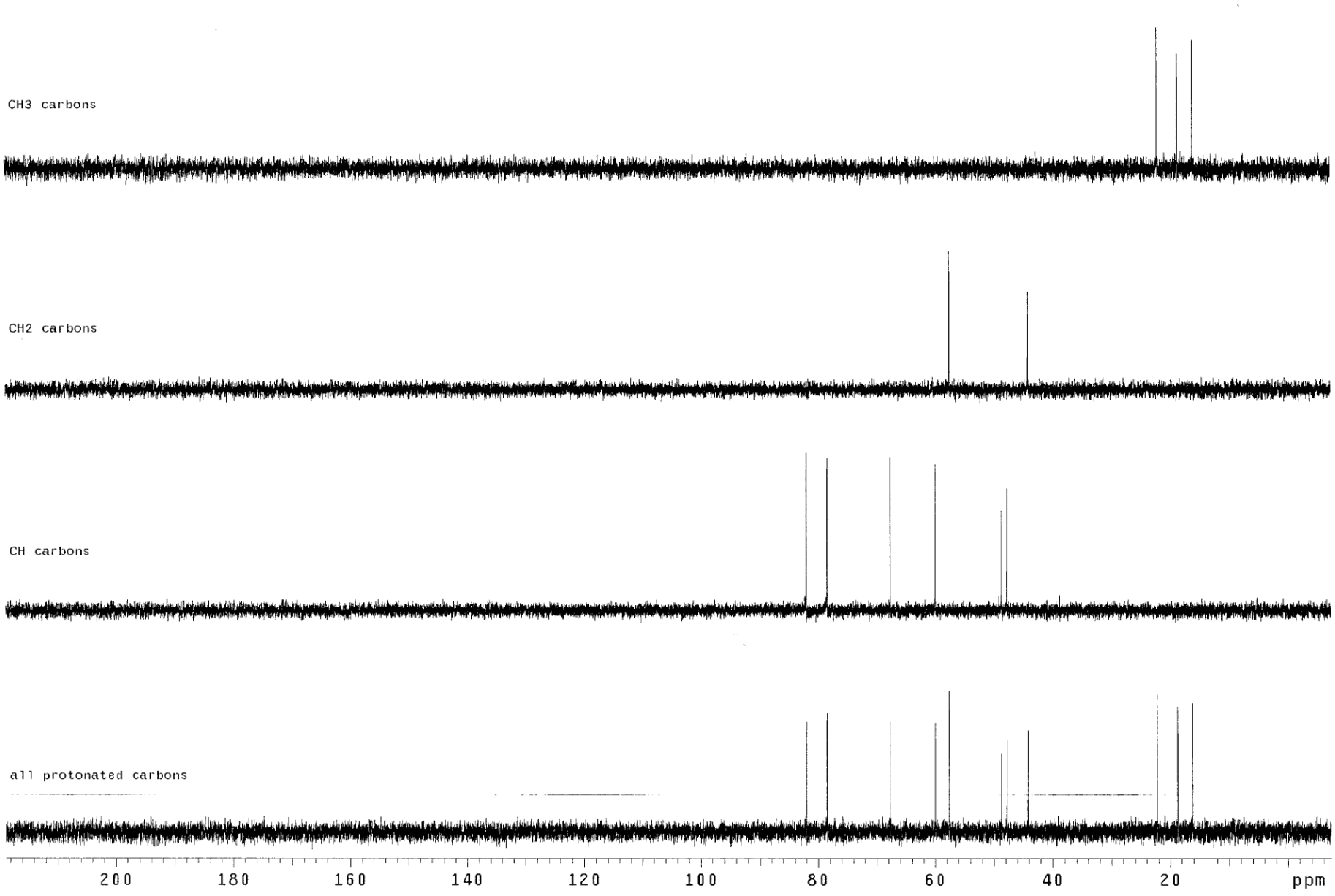


TY2-357

Pulse Sequence: hetcor
 Solvent: D2O
 Ambient temperature
User: 1-14-87
File: TY2-357-CH
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
 64 repetitions
 256 increments
OBSERVE C13, 125.6904822 MHz
DECOUPLE H1, 499.8652159 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, 32 min, 4 sec

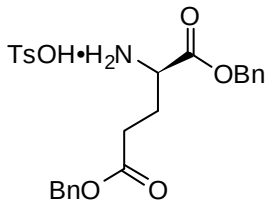


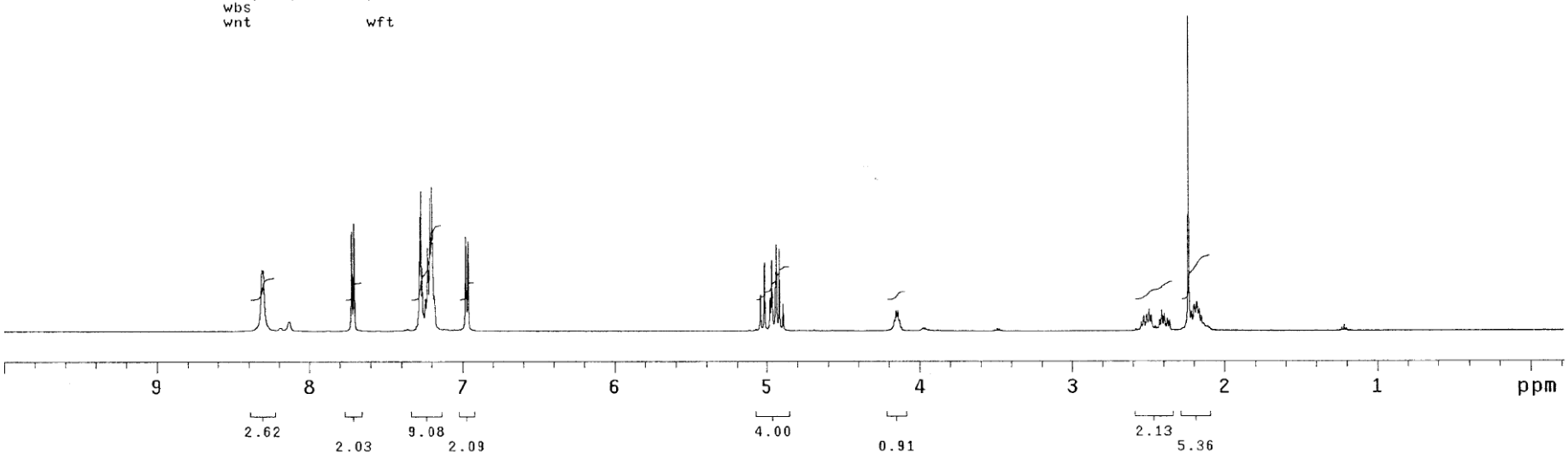

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

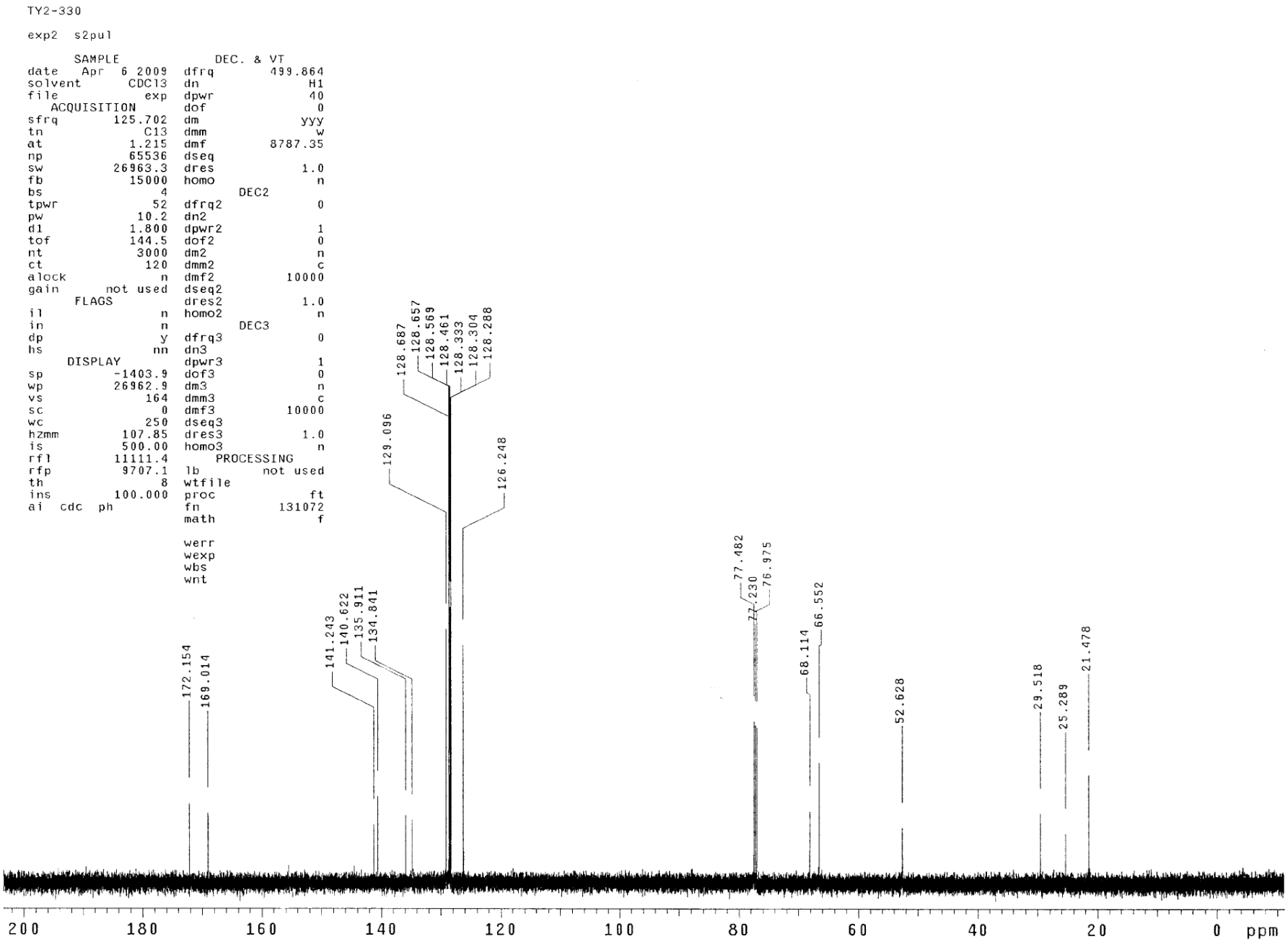
TY2-330

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 6 2009	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	-107.0	dof3	0
wp	5107.9	dm3	n
vs	36	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	81.48	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	4.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft





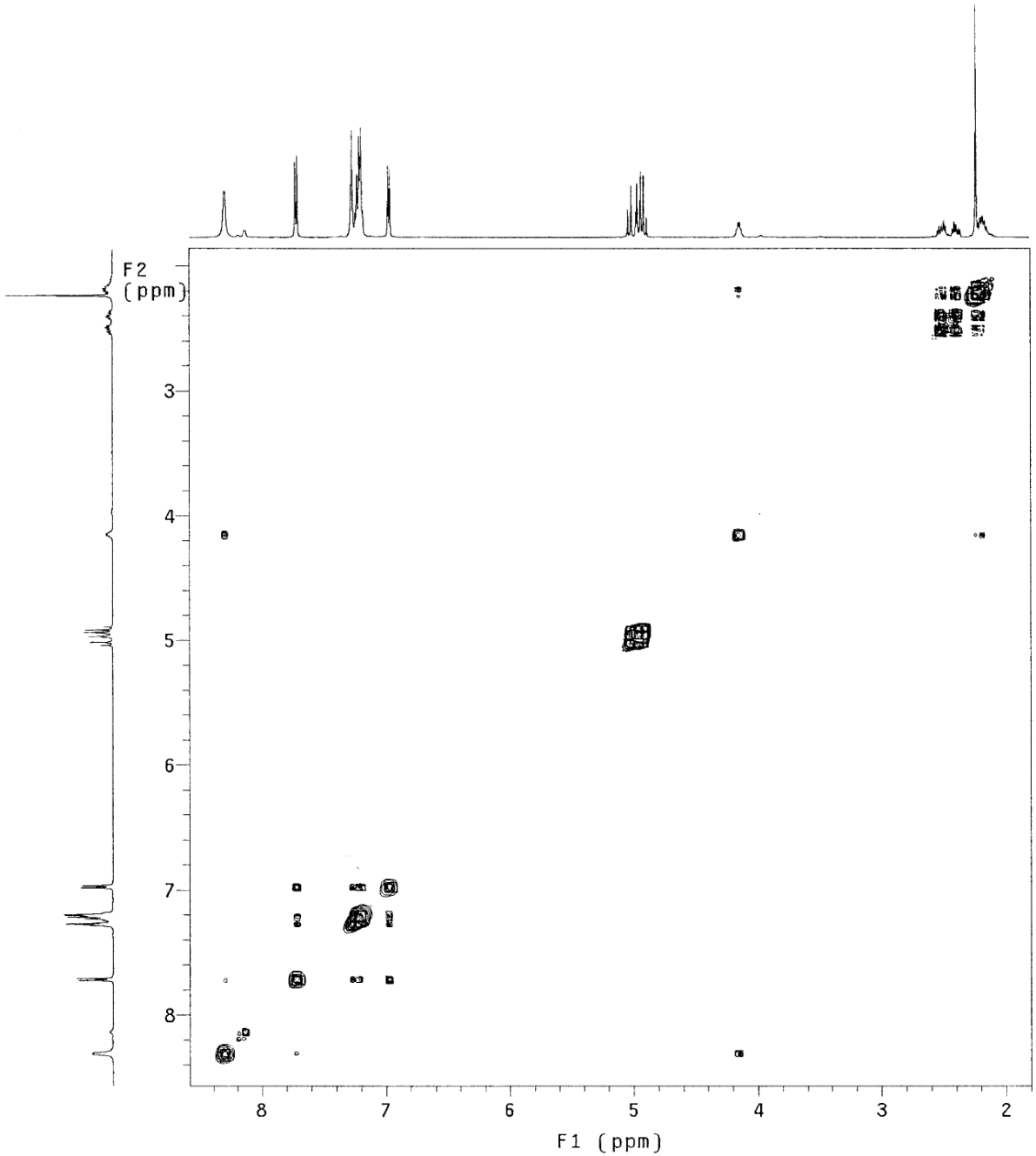

TY2-330
exp2 s2pul
SAMPLE
date Apr 6 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 3000
ct 120
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1403.9
wp 26962.9
vs 164
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11111.4
rfp 9707.1
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 not used
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
172.154
169.014
141.243
140.622
135.911
134.841
129.096
128.687
128.657
128.569
128.461
128.333
128.304
128.288
126.248
77.482
77.230
76.975
68.114
66.552
52.628
29.518
25.289
21.478
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-330

Pulse Sequence: relayh

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

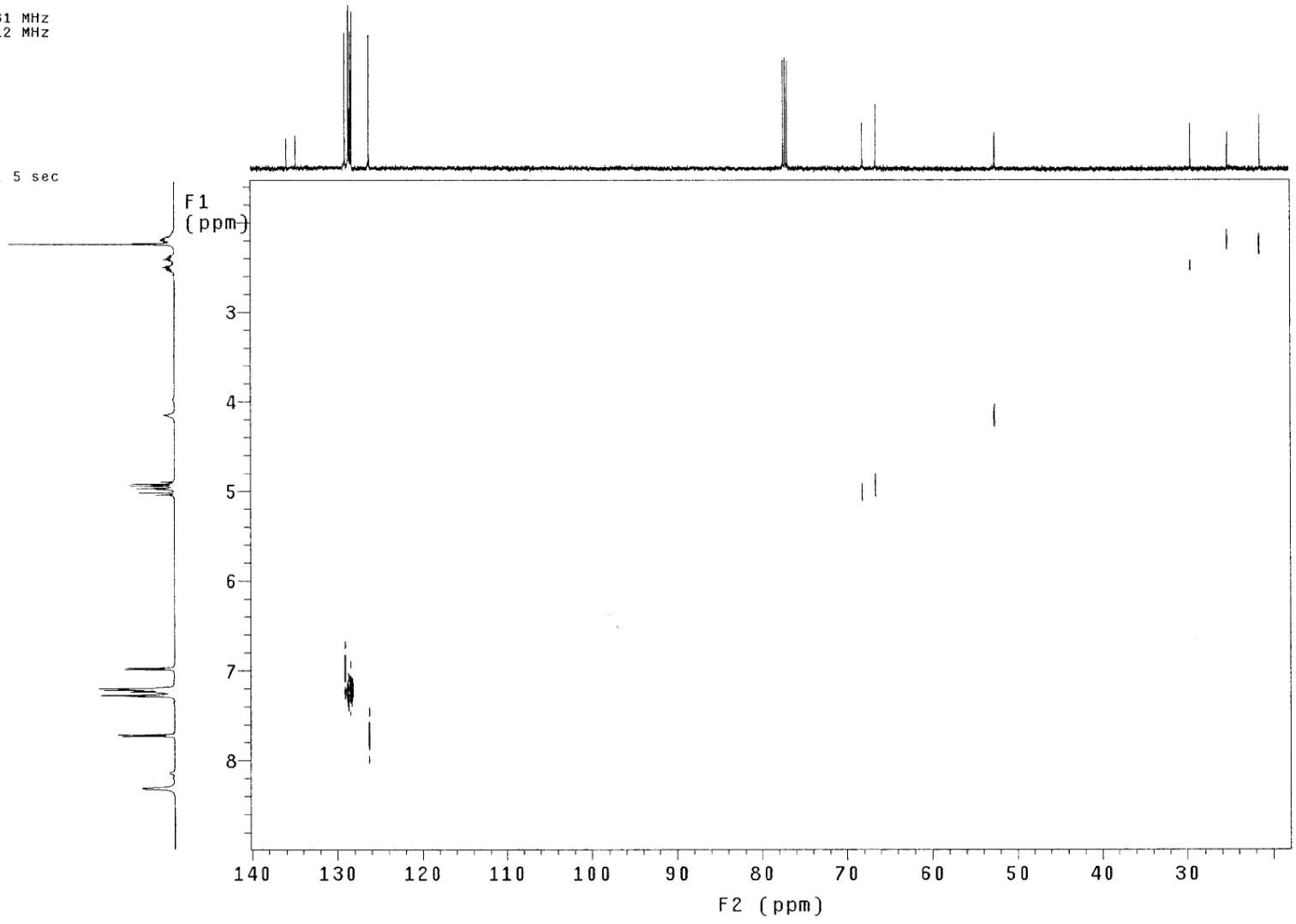
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

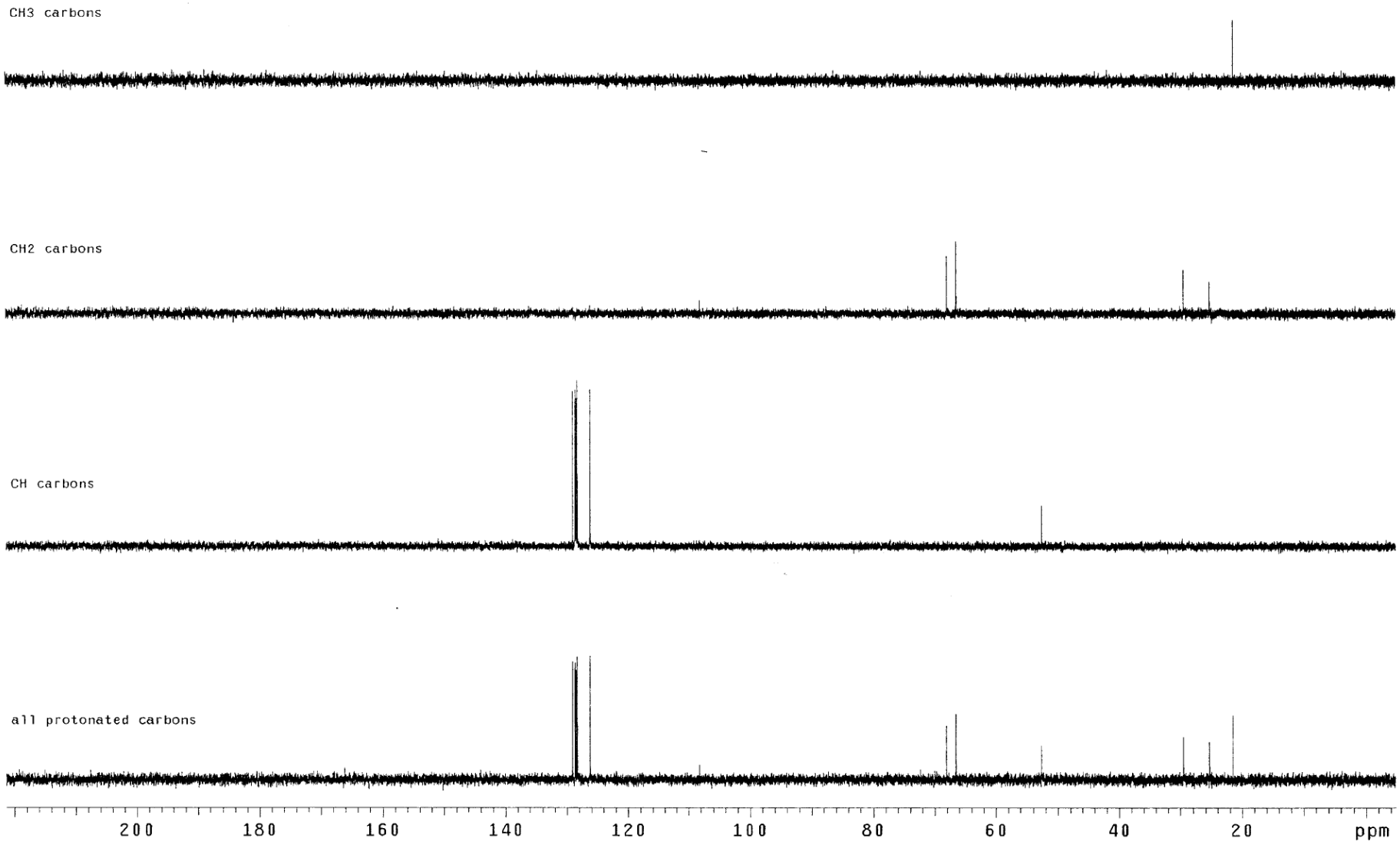


TY2-330

Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
File: TY2-330-CH
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
 32 repetitions
 256 increments
OBSERVE C13, 125.6901681 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 3 hr, 46 min, 5 sec

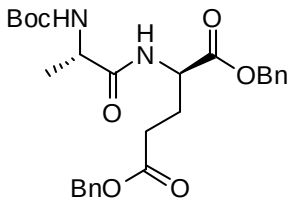


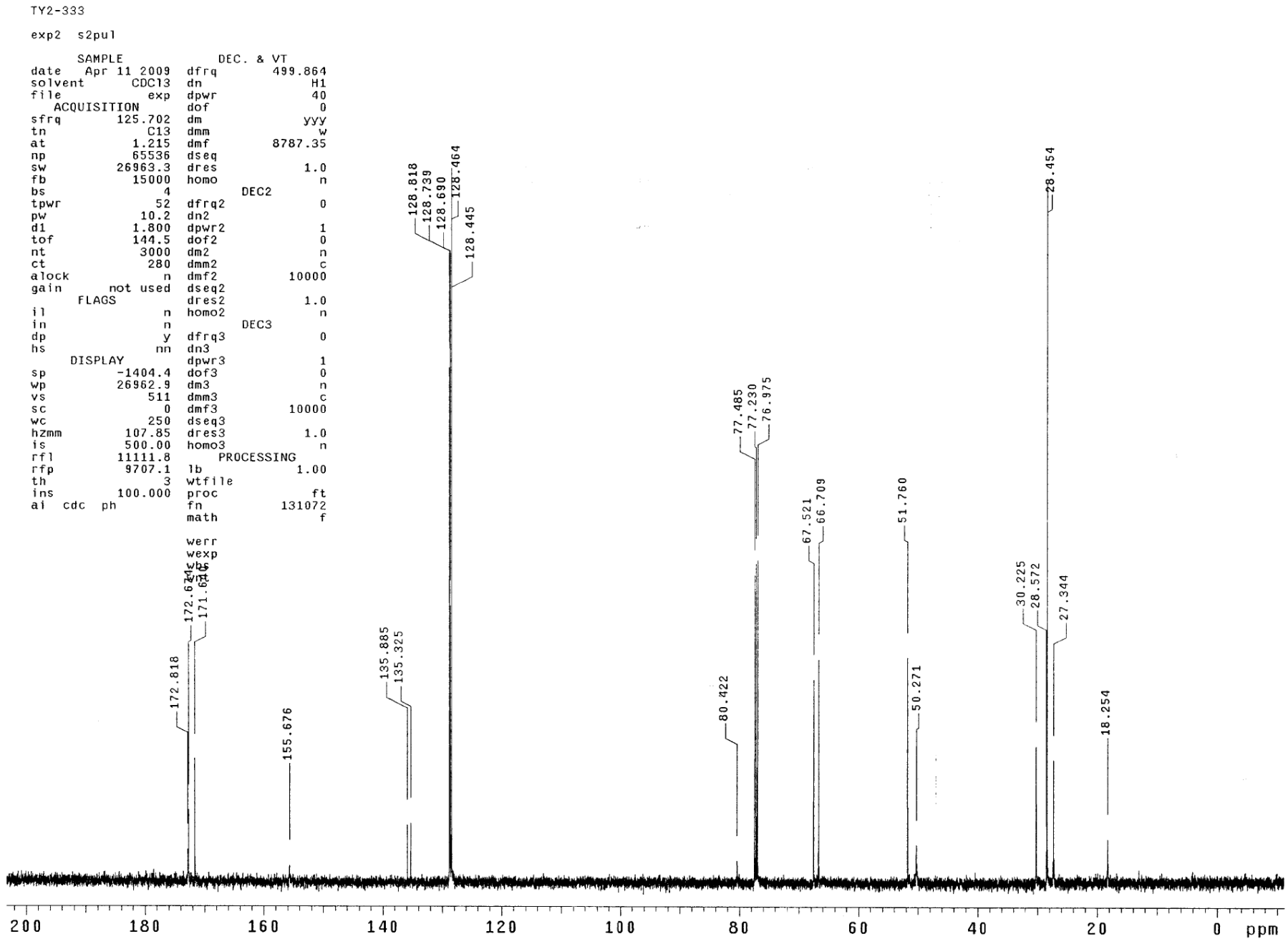

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

TY2-333

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 11 2009	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	-102.8	dof3	0
wp	5099.3	dm3	n
vs	31	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	172.93	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	2.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



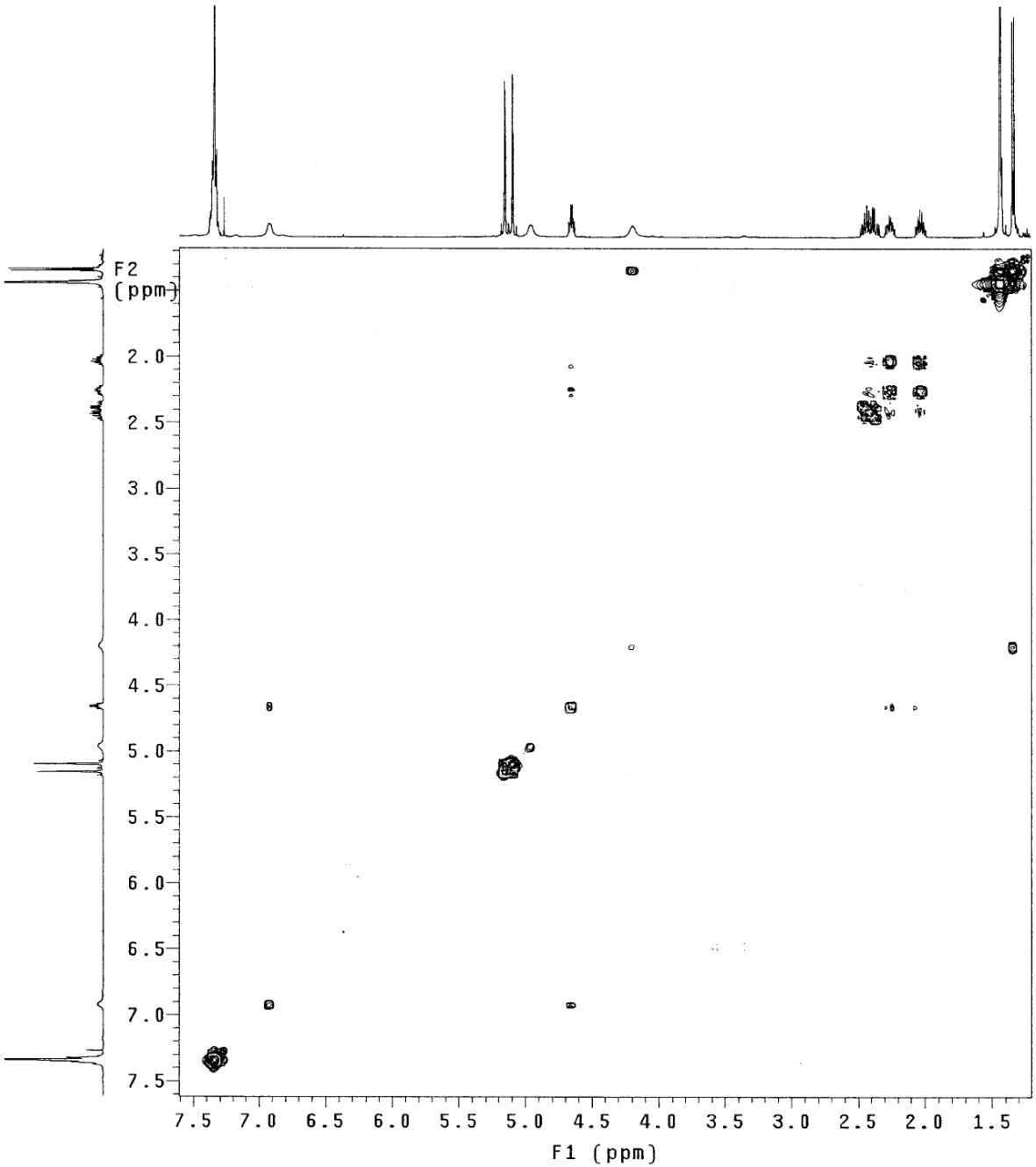

TY2-333
exp2 s2pul
SAMPLE
date Apr 11 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 3000
ct 280
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1404.4
wp 26962.9
vs 511
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11111.8
rfp 9707.1
th 3
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
172.818
155.676
135.885
135.325
80.422
77.485
77.230
76.975
67.521
66.709
51.760
50.271
30.225
28.572
28.454
27.344
18.254
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-333

Pulse Sequence: relayh

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

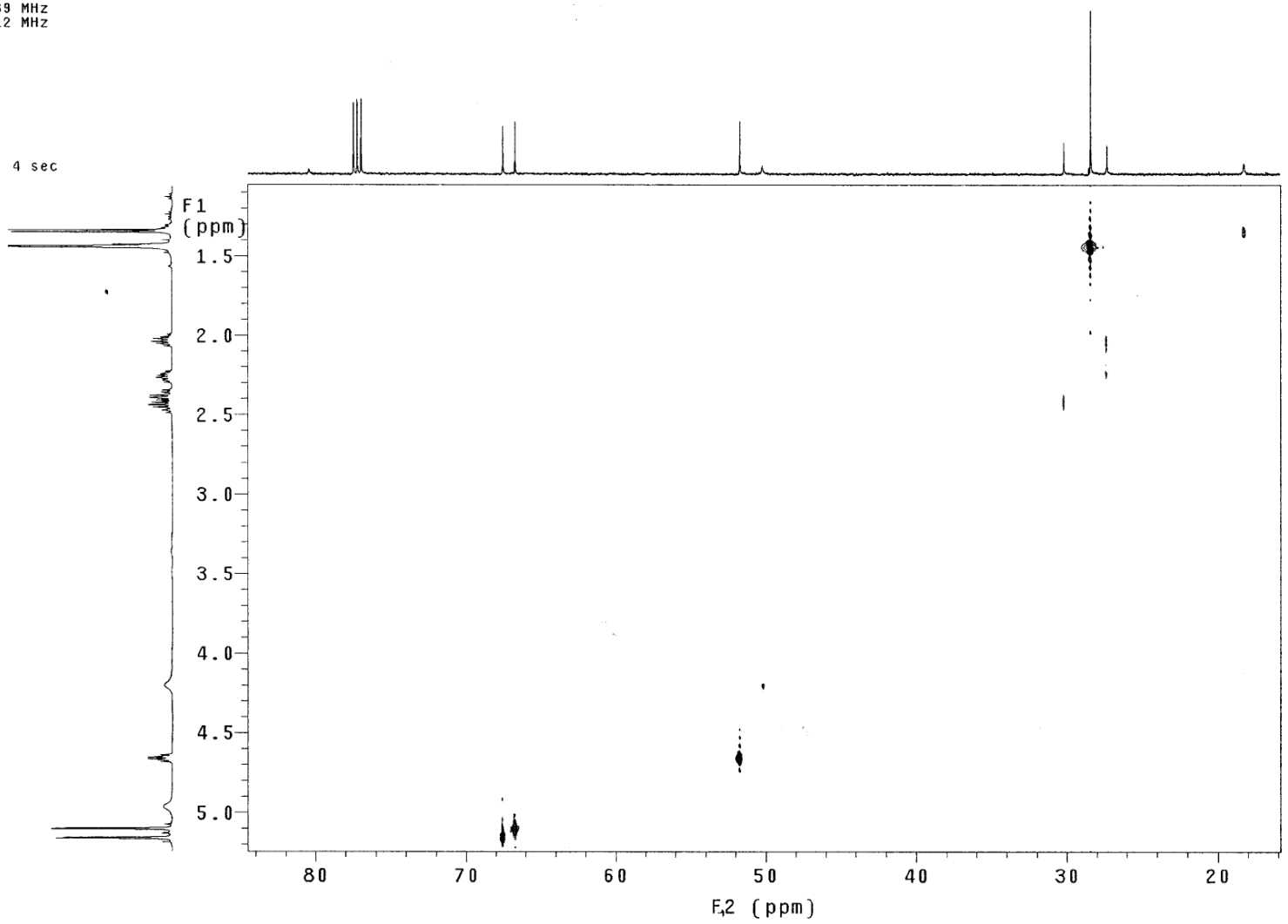
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611709 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

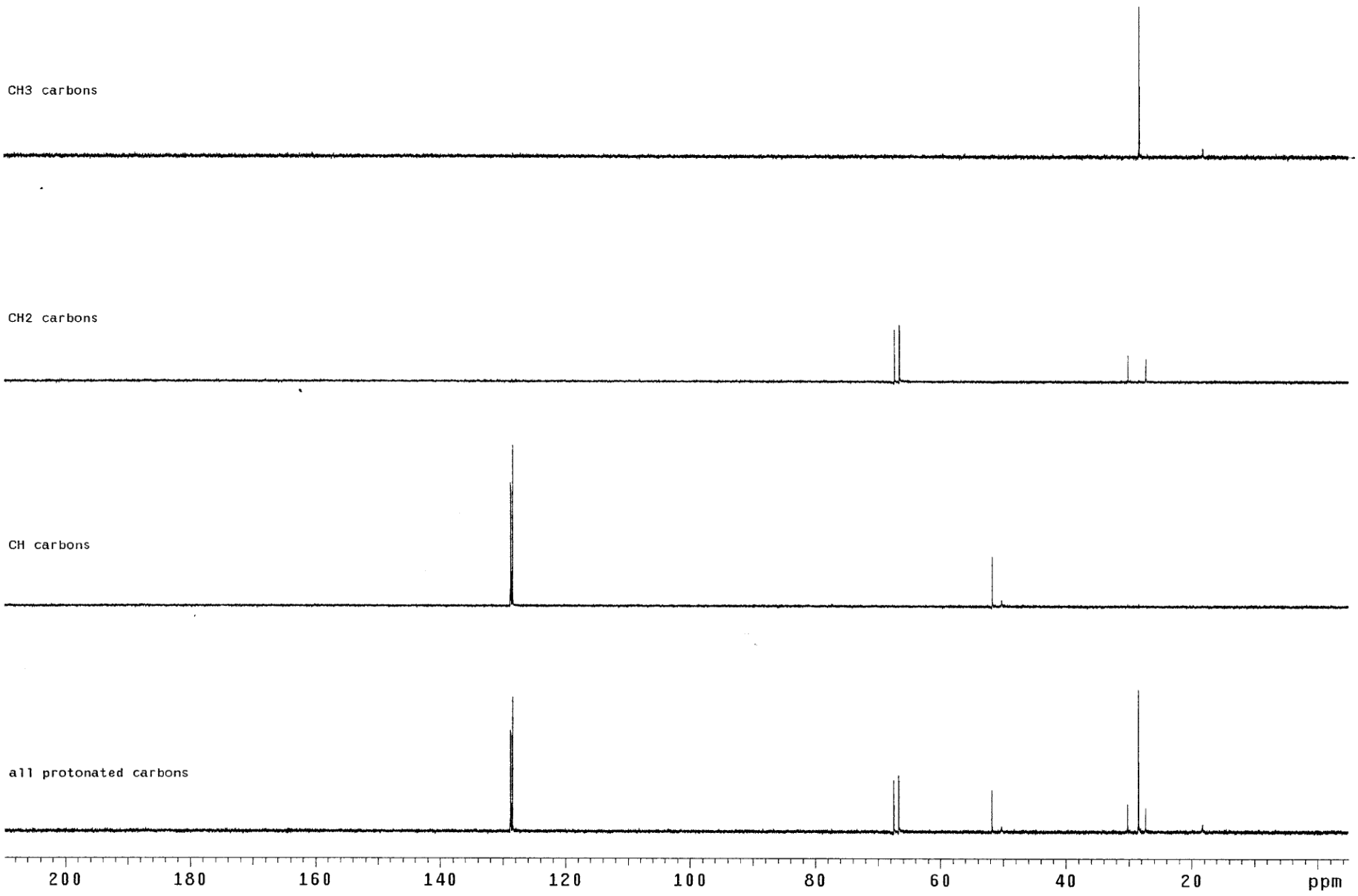


TY2-333

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
 64 repetitions
 256 increments
OBSERVE C13, 125.6901689 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 7 hr, 32 min, 4 sec

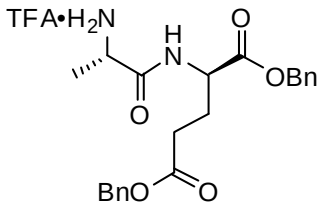


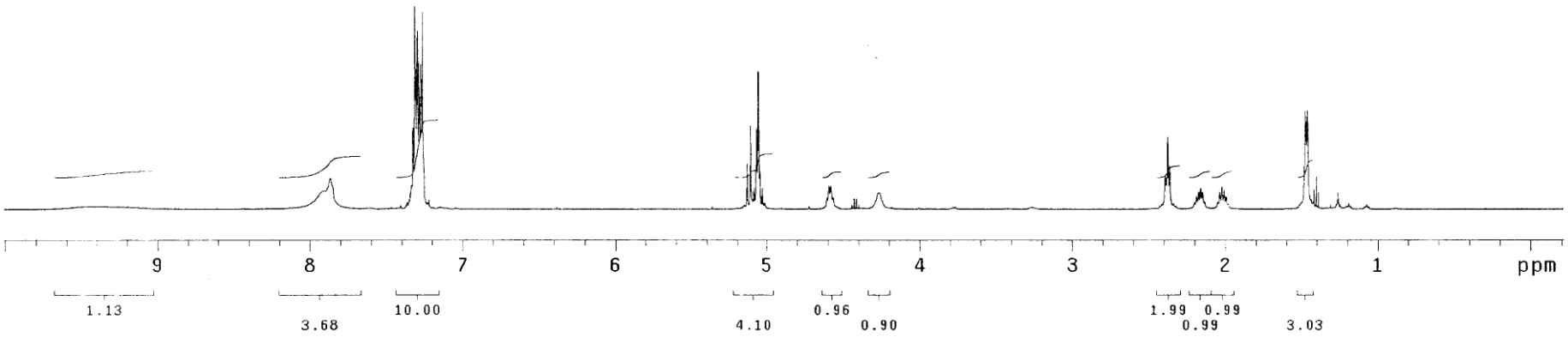

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

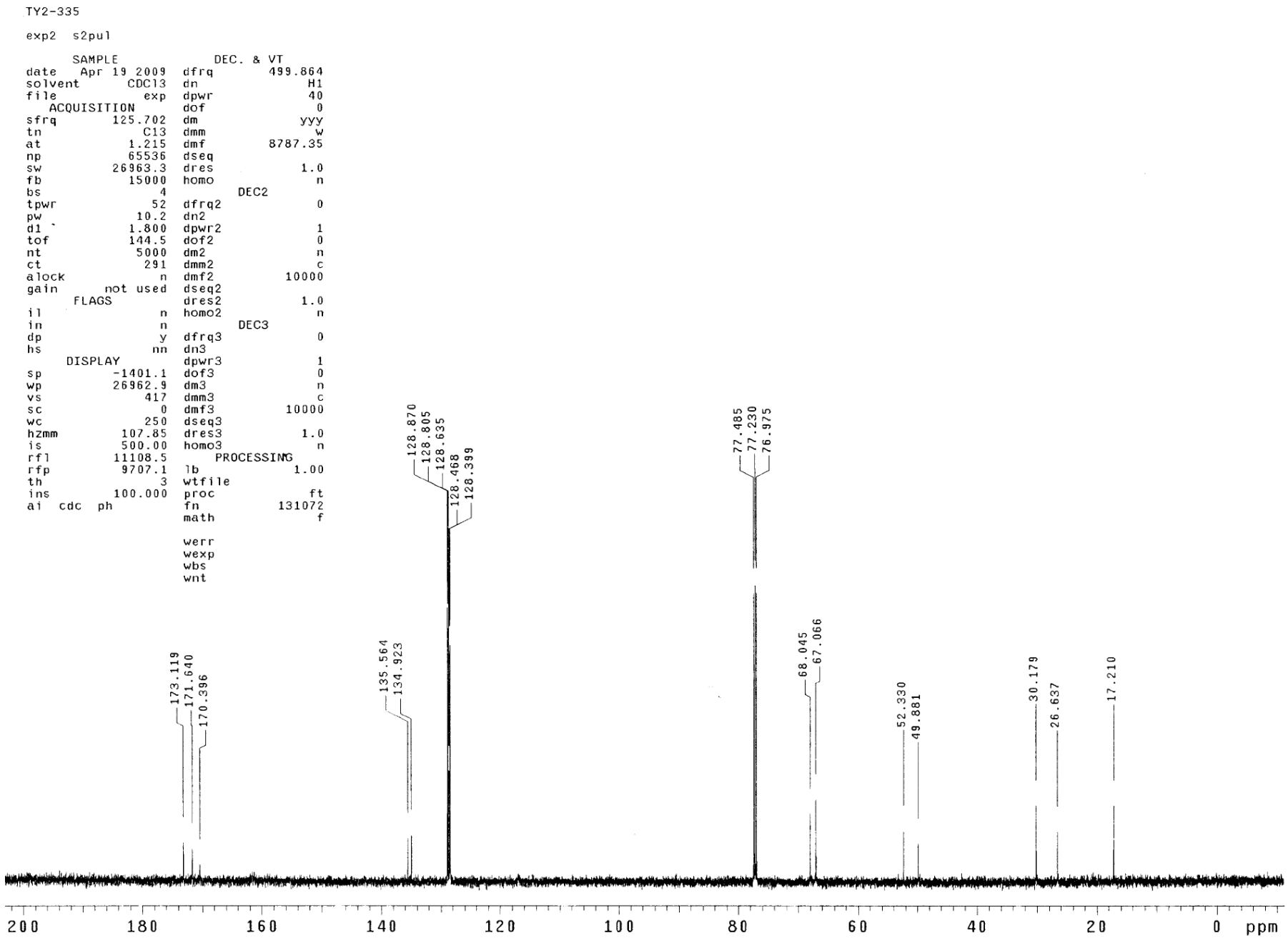
TY2-335

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 19 2009	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	4	dm2	n
ct	4	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	-107.0	dof3	0
wp	5107.9	dm3	n
vs	33	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	44.70	homo3	n
rfl	510.6	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	10.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft





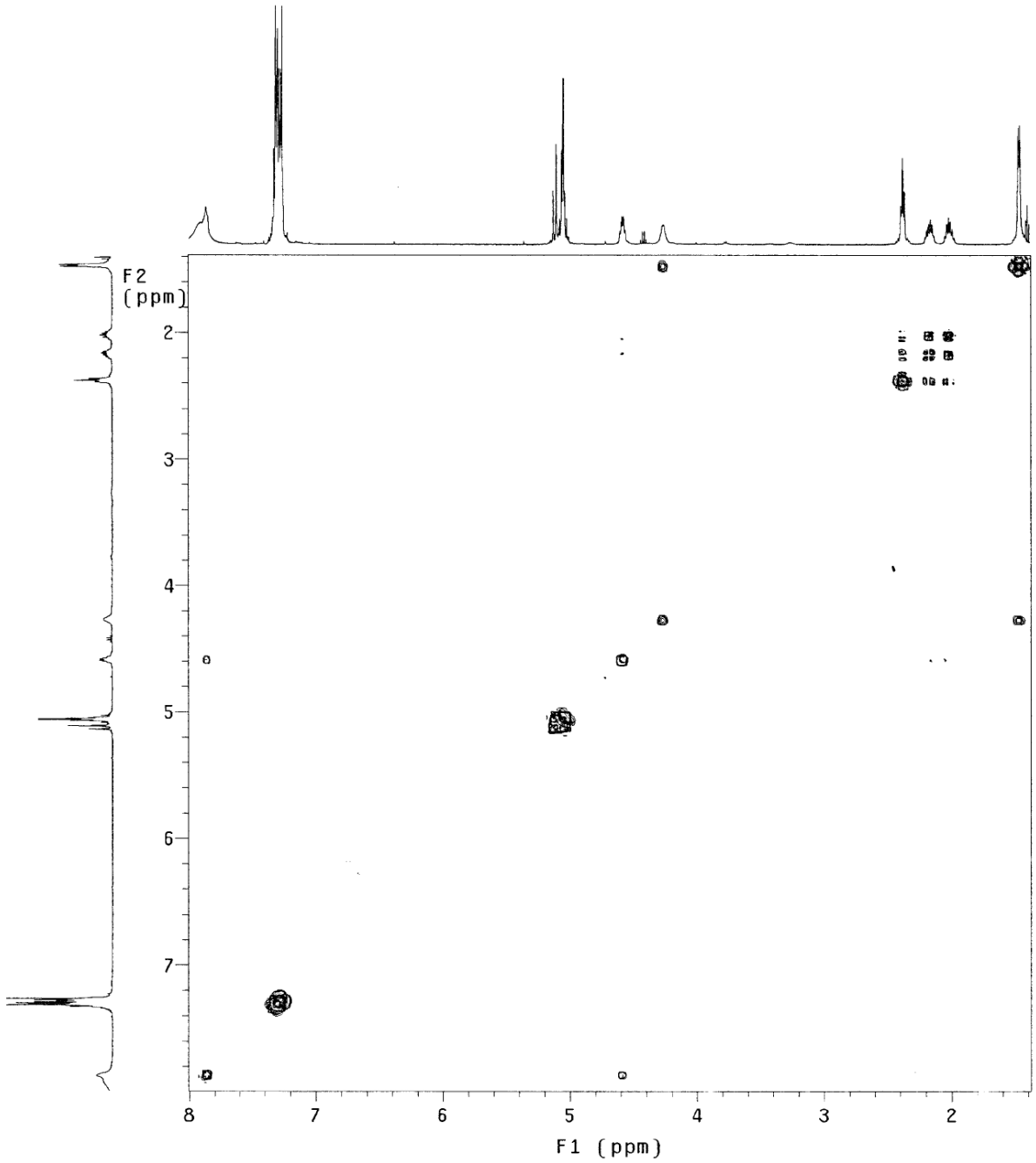

TY2-335
exp2 s2pul
SAMPLE
date Apr 19 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 5000
ct 291
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1401.1
wp 26962.9
vs 417
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11108.5
rfp 9707.1
th 3
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
173.119
171.640
170.396
135.564
134.923
128.870
128.805
128.635
128.468
128.399
77.485
77.230
76.975
68.045
67.066
52.330
49.881
30.179
26.637
17.210
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-335

Pulse Sequence: relayh

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

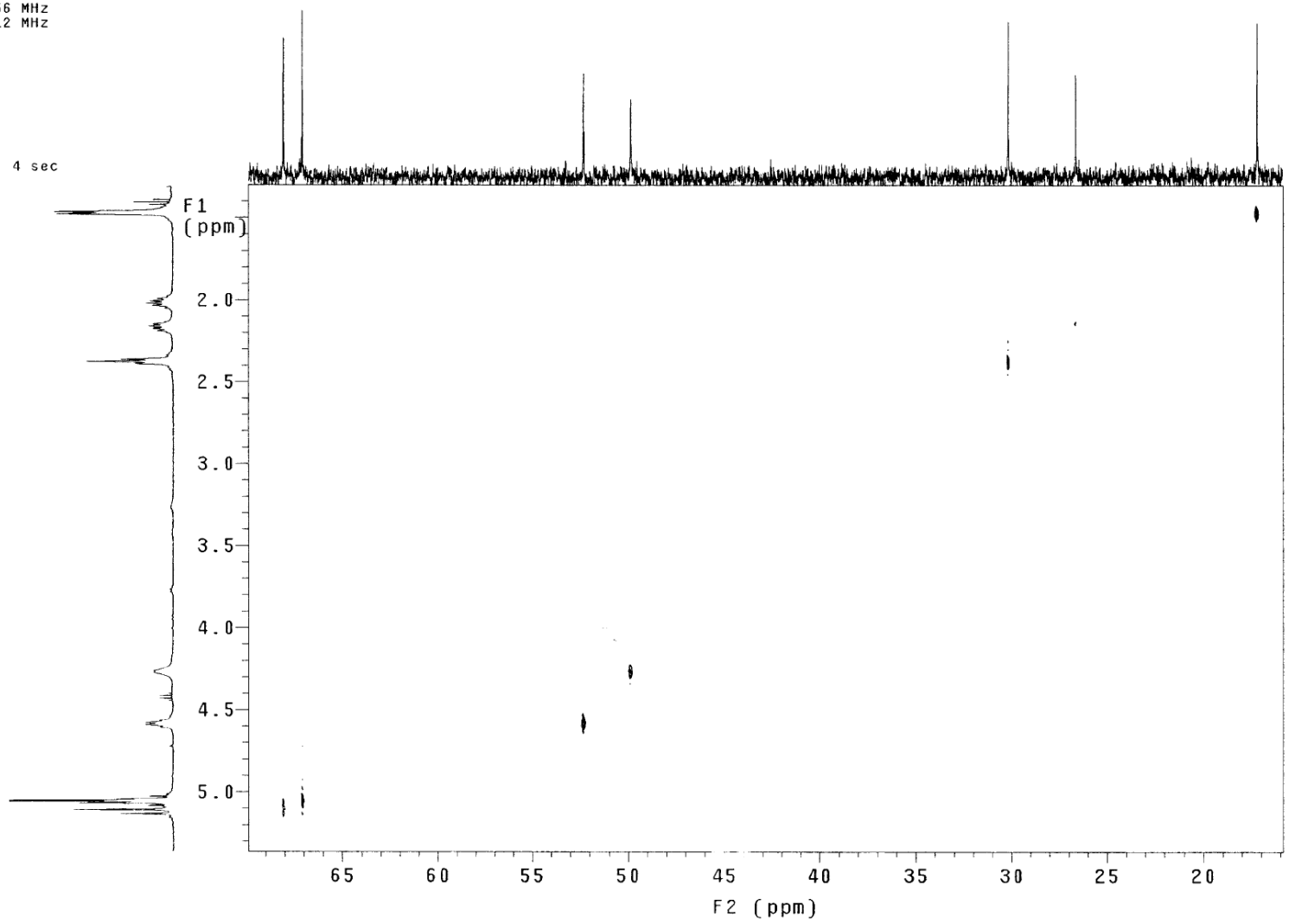
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

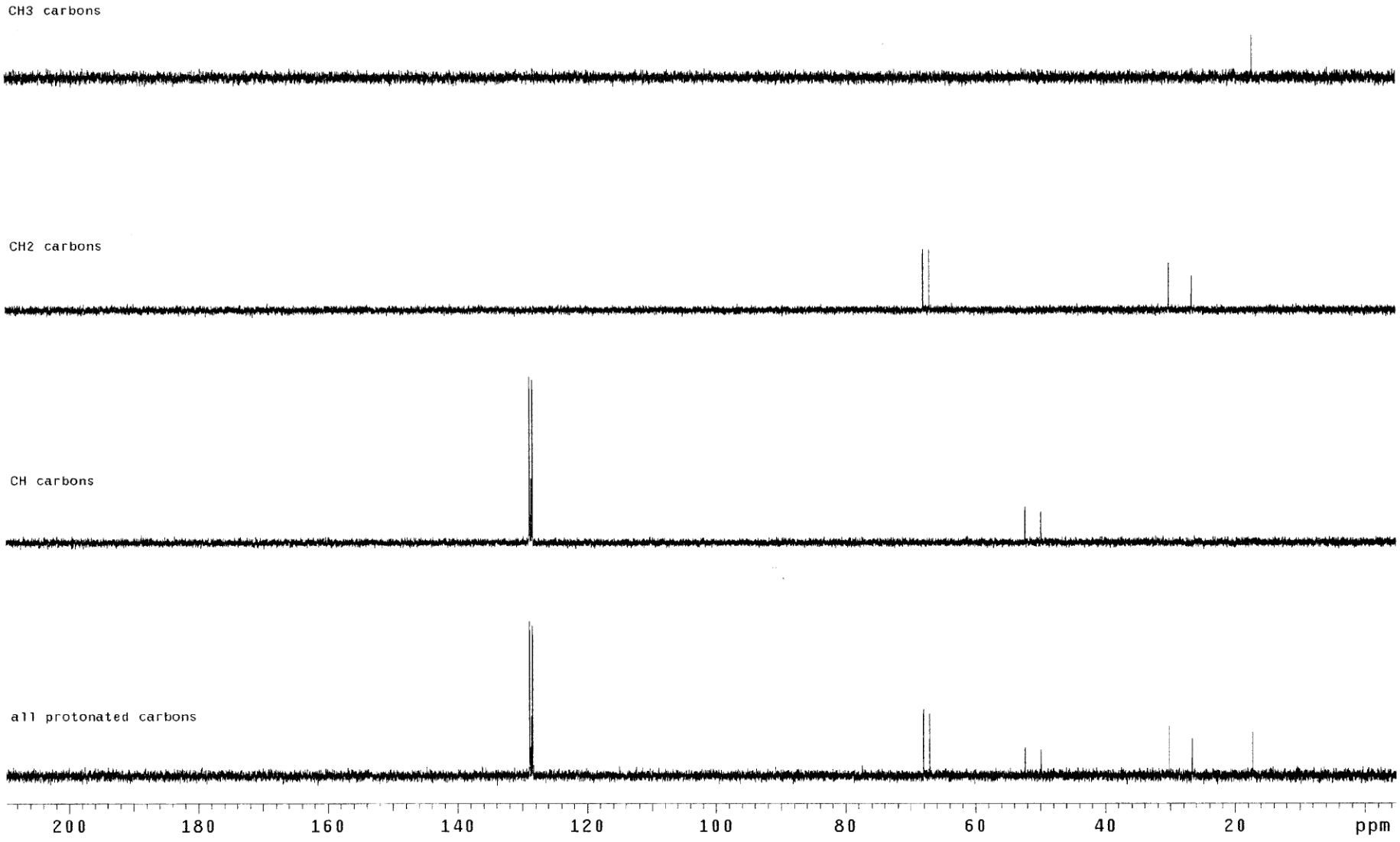


TY2-335

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
 64 repetitions
 256 increments
OBSERVE C13, 125.6901656 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 7 hr, 32 min, 4 sec

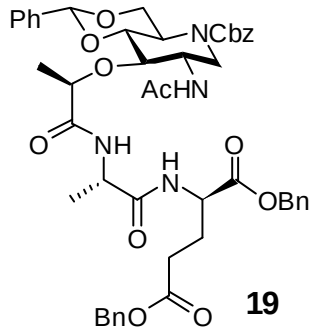



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

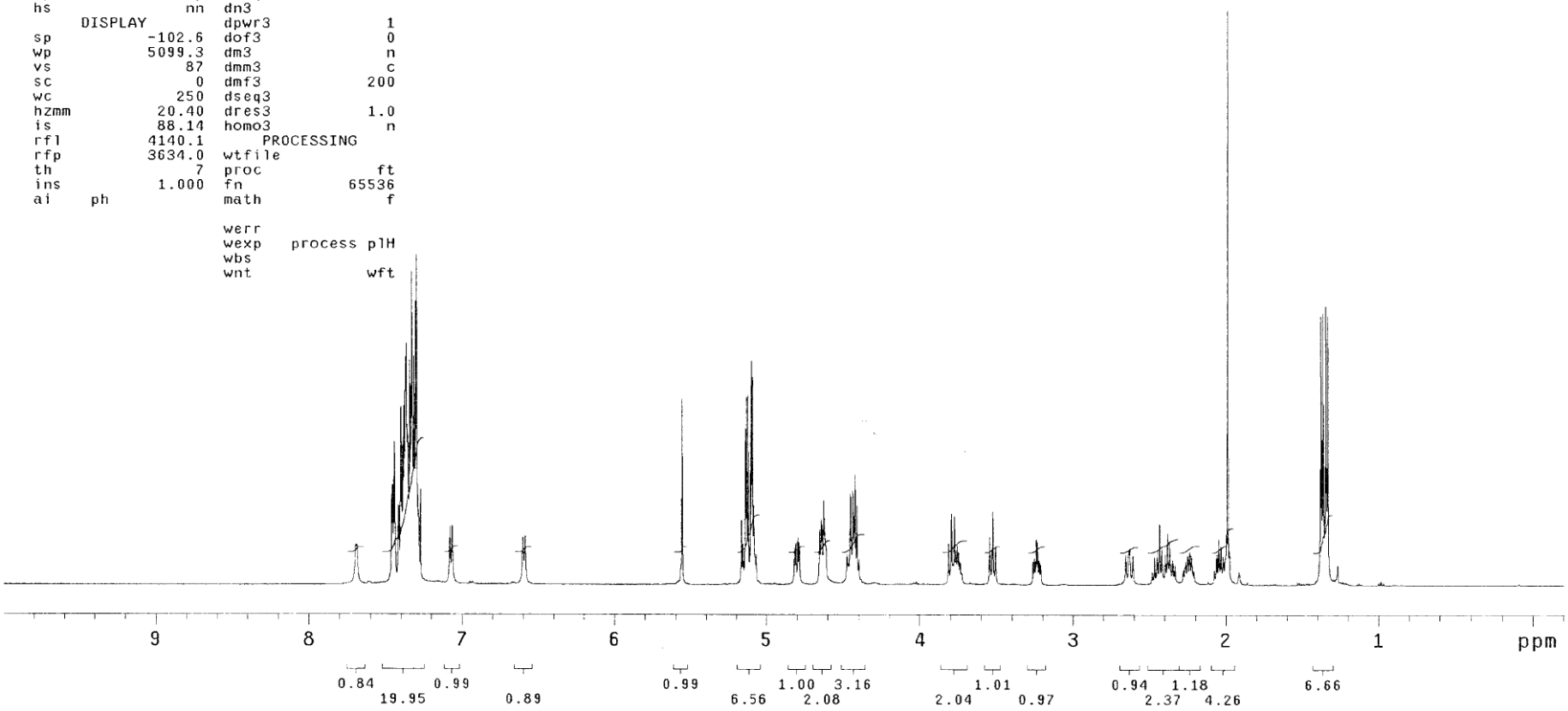
TY2-348

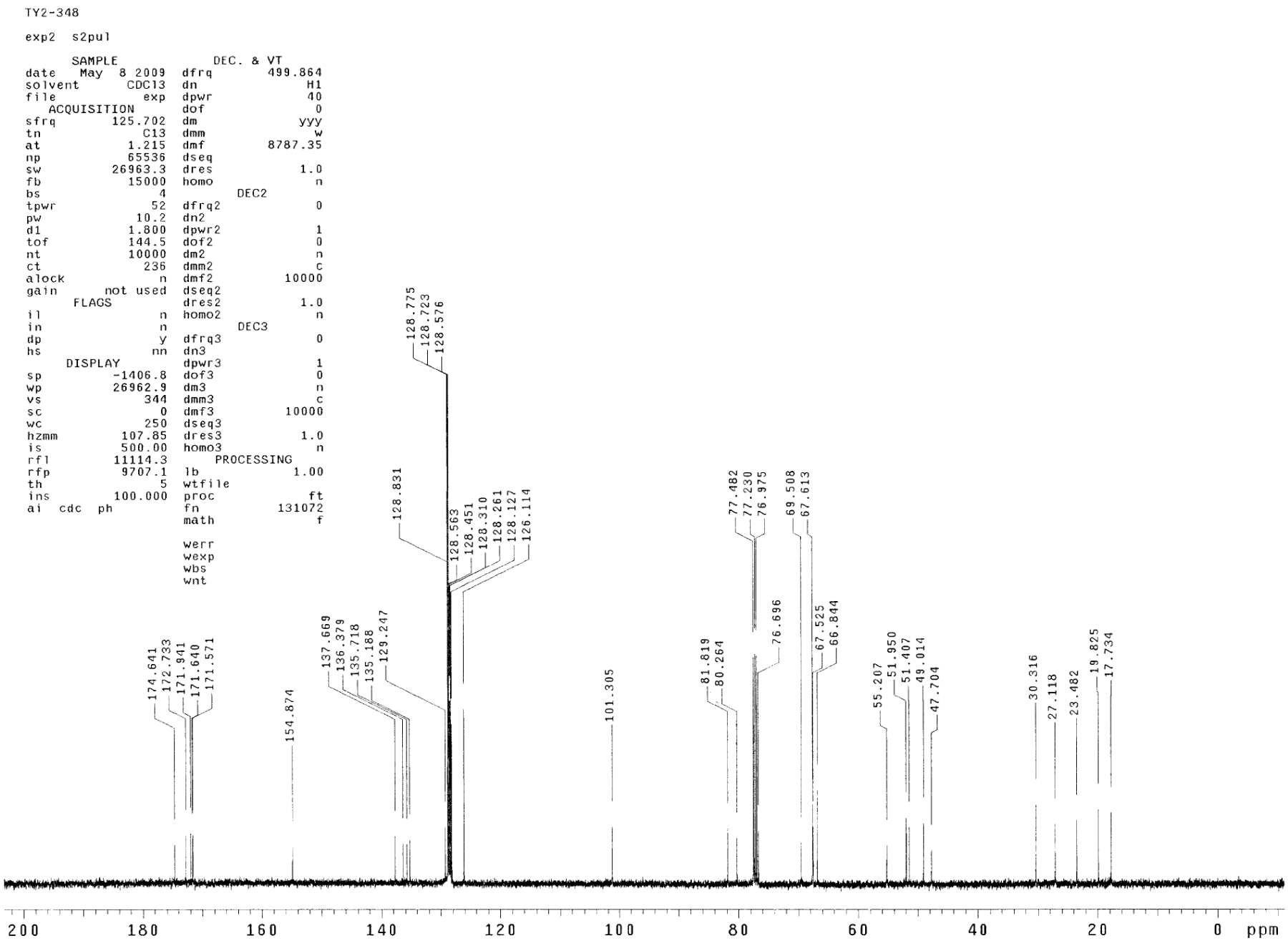
exp1 s2pul

SAMPLE		DEC. & VT	
date	May 8 2009	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	-102.6	dof3	0
wp	5099.3	dm3	n
vs	87	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	88.14	homo3	n
rfl	4140.1	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



19



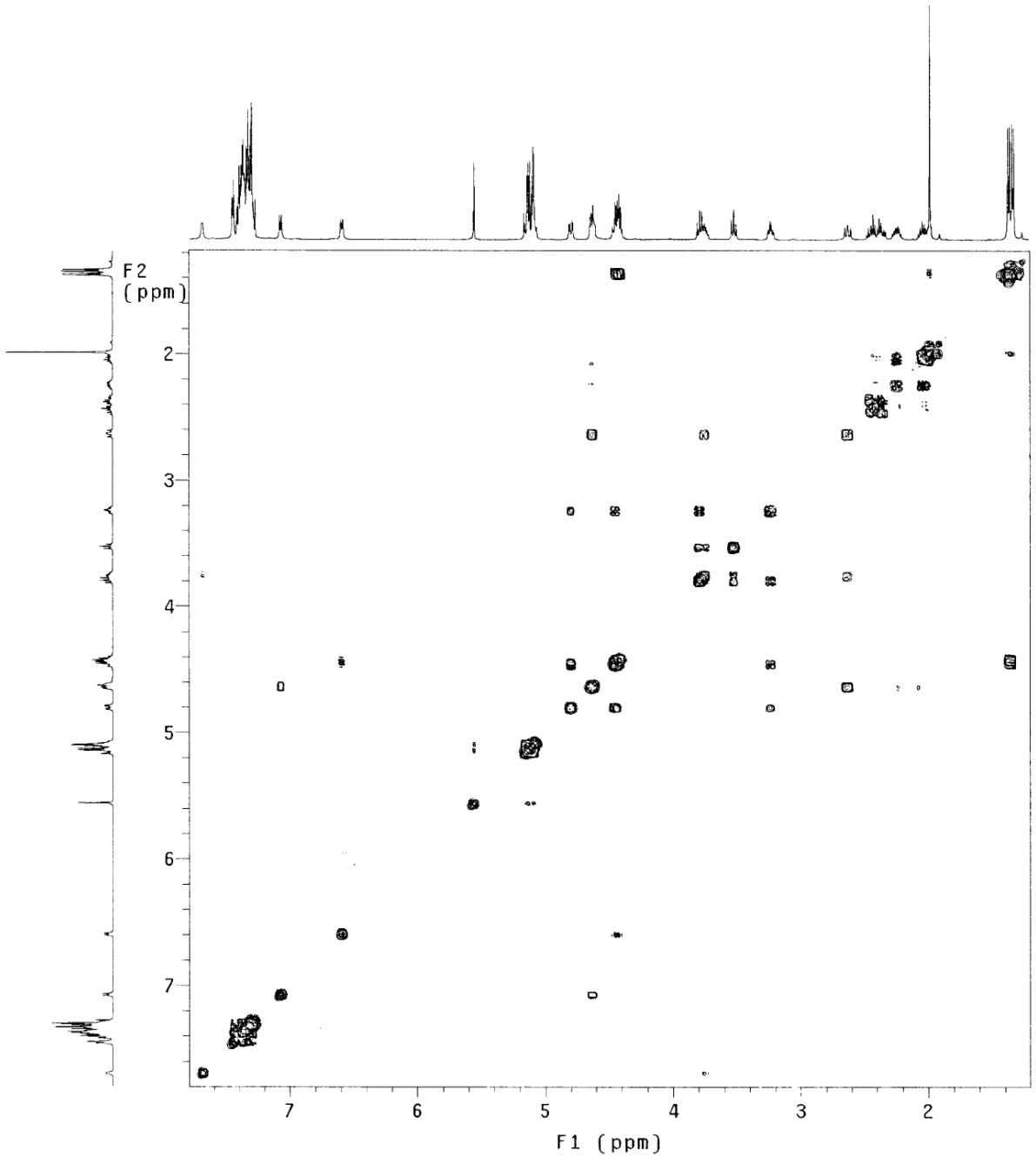

TY2-348
exp2 s2pul
SAMPLE
date May 8 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 10000
ct 236
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1406.8
wp 26962.9
vs 344
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11114.3
rfp 9707.1
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
174.641
172.733
171.941
171.640
171.571
154.874
137.669
136.379
135.718
135.188
129.247
128.831
128.775
128.723
128.576
128.563
128.451
128.310
128.261
128.127
126.114
101.305
81.819
80.264
77.482
77.230
76.975
76.696
69.508
67.613
67.525
66.844
55.207
51.950
51.407
49.014
47.704
30.316
27.118
23.482
19.825
17.734
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-348

Pulse Sequence: relayh

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

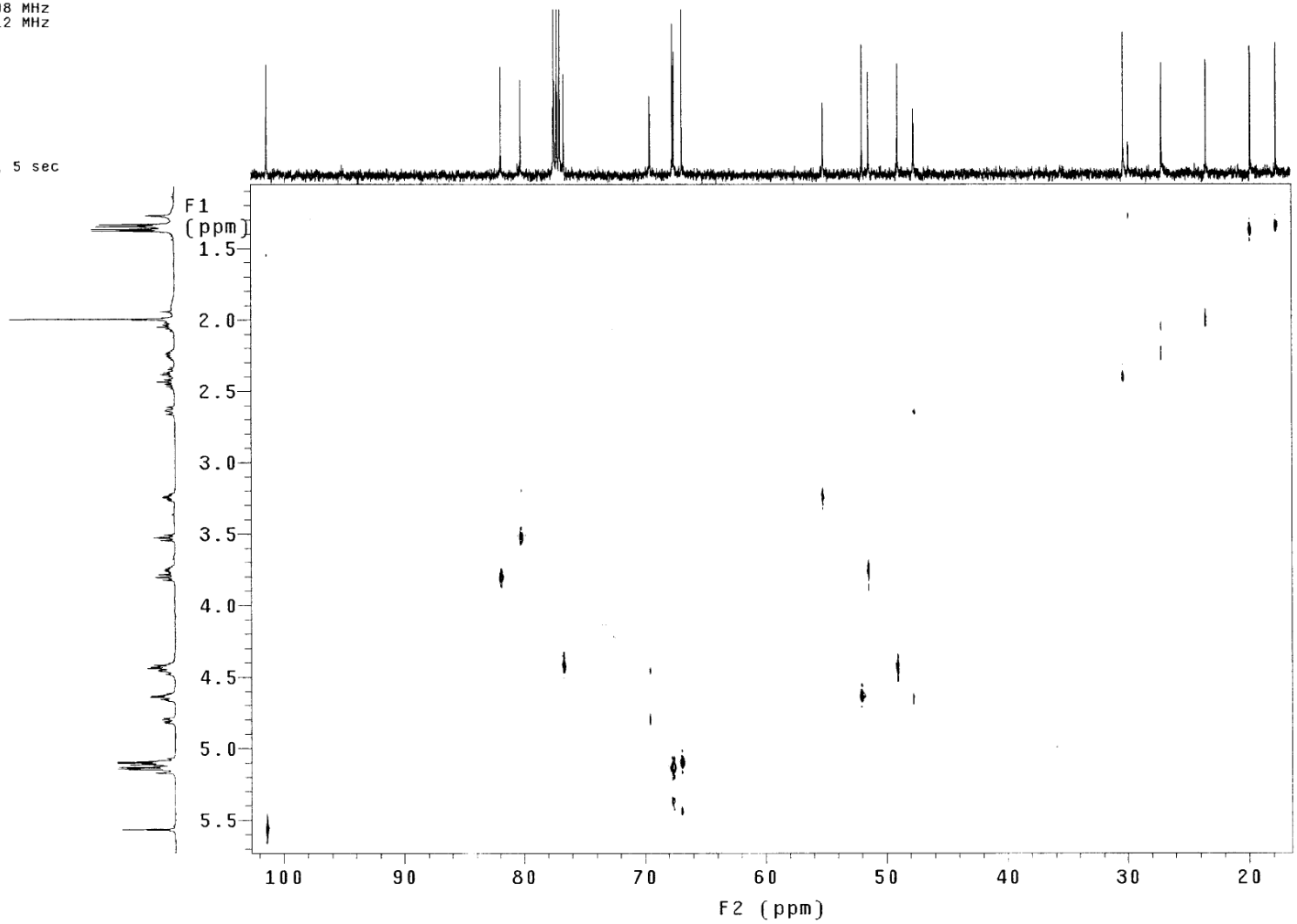
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
64 repetitions
256 increments
OBSERVE H1, 499.8611707 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 6 hr, 46 min, 8 sec

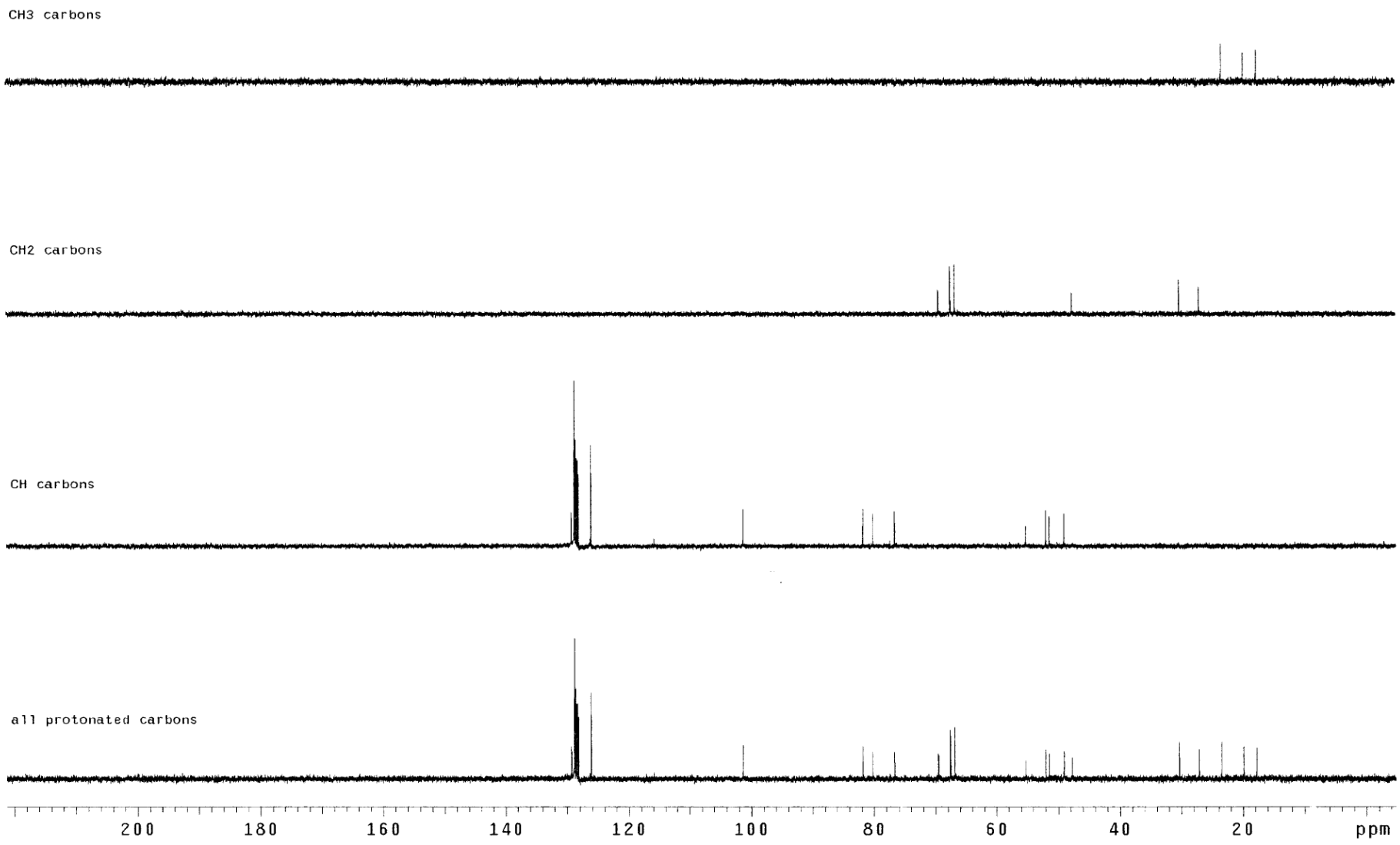


TY2-348

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
 32 repetitions
 256 increments
OBSERVE C13, 125.6901698 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 3 hr, 46 min, 5 sec

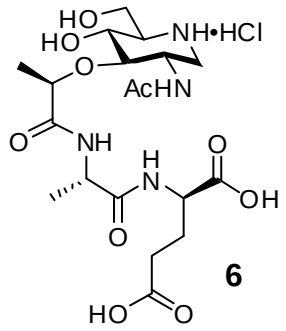



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

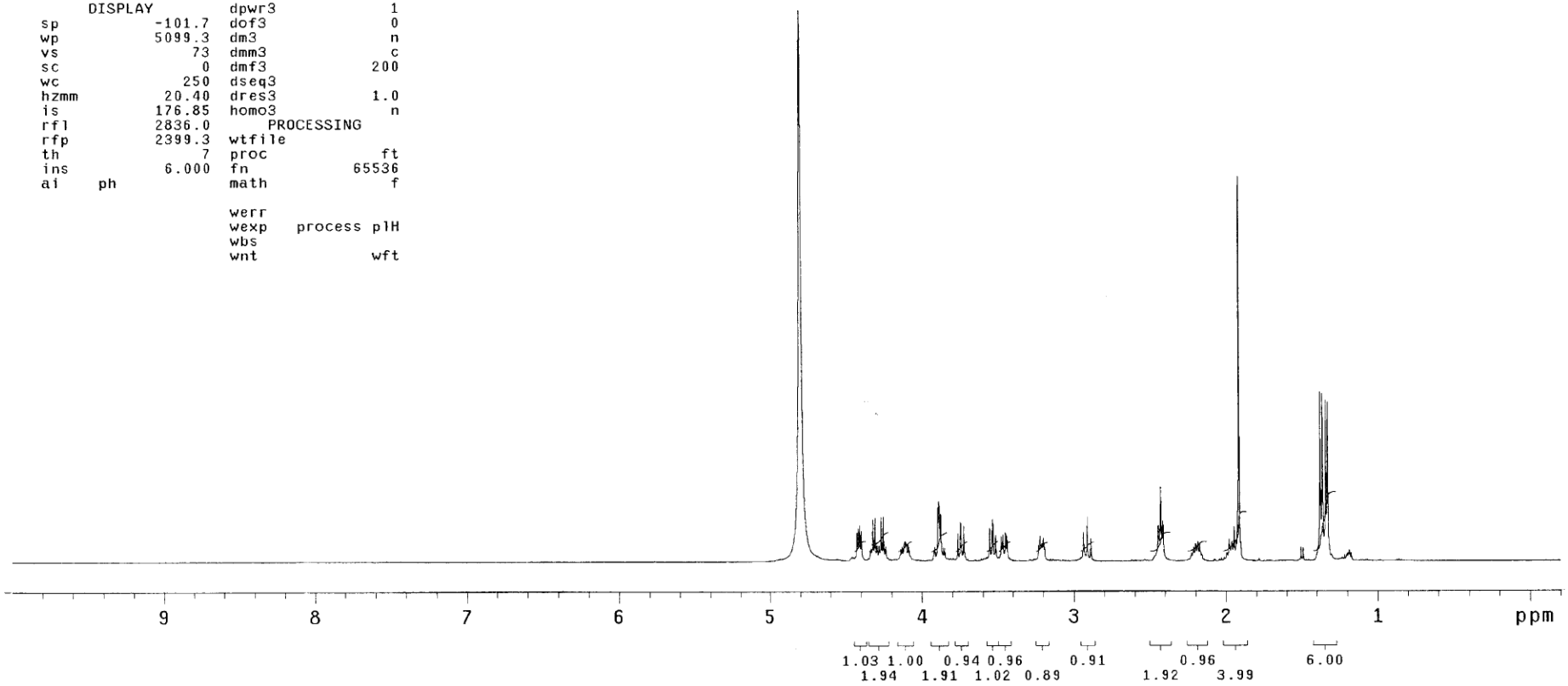
STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 1 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.865	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	-101.7	dof3	0
wp	5099.3	dm3	n
vs	73	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	176.85	homo3	n
rfl	2836.0	PROCESSING	
rfp	2399.3	wtfile	
th	7	proc	ft
ins	6.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



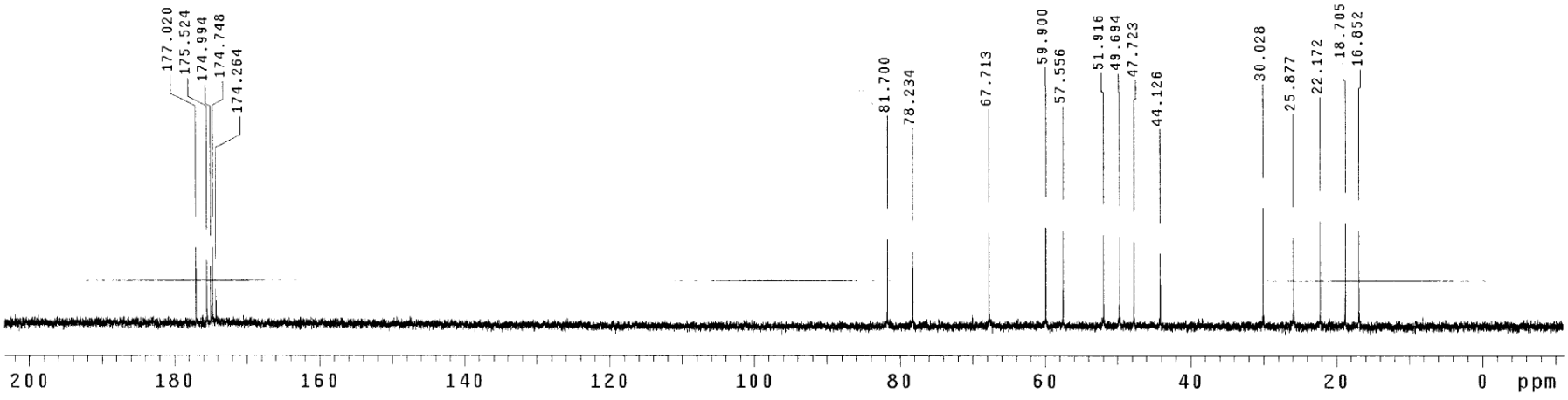
6



TY2-358

exp2 s2pul

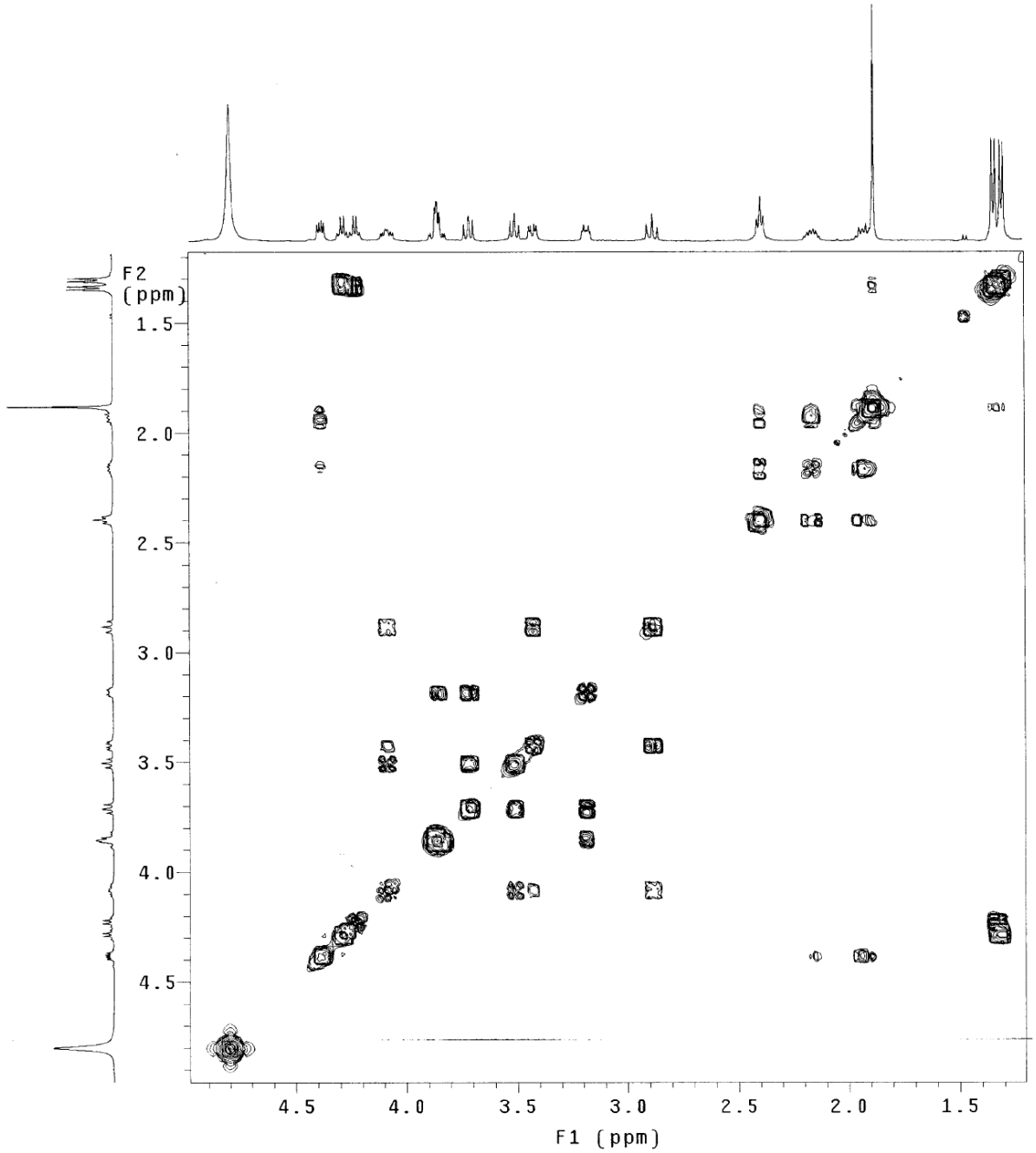
SAMPLE		DEC. & VT	
date	May 29 2009	dfrq	499.865
solvent	D2O	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	3000	dm2	n
ct	392	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	-1394.6	dof3	0
wp	26962.9	dm3	n
vs	361	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	1395.0	PROCESSING	
rfp	0	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-358

Pulse Sequence: relayh
 Solvent: D2O
 Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

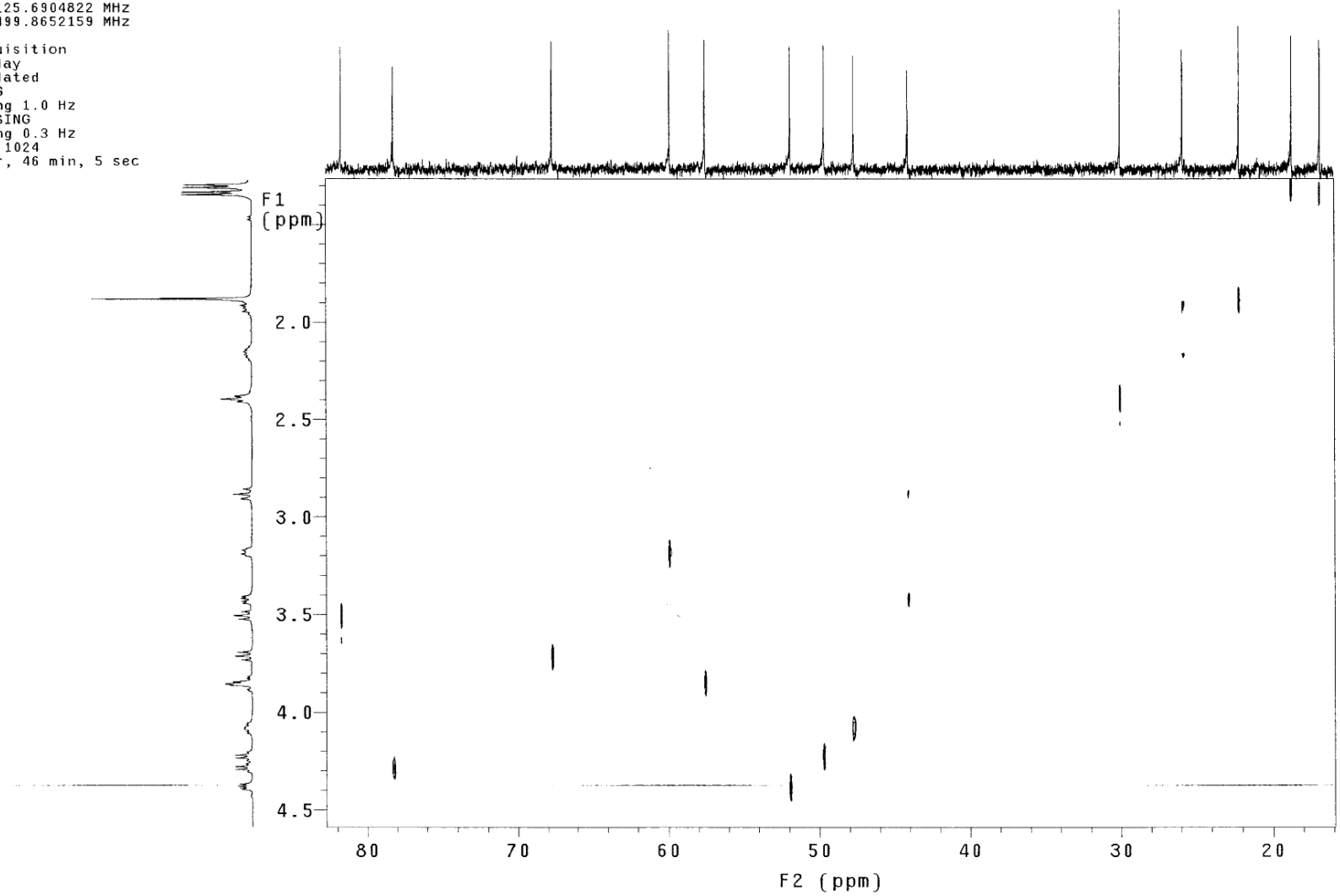
 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 32 repetitions
 256 increments
OBSERVE H1, 499.8623875 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

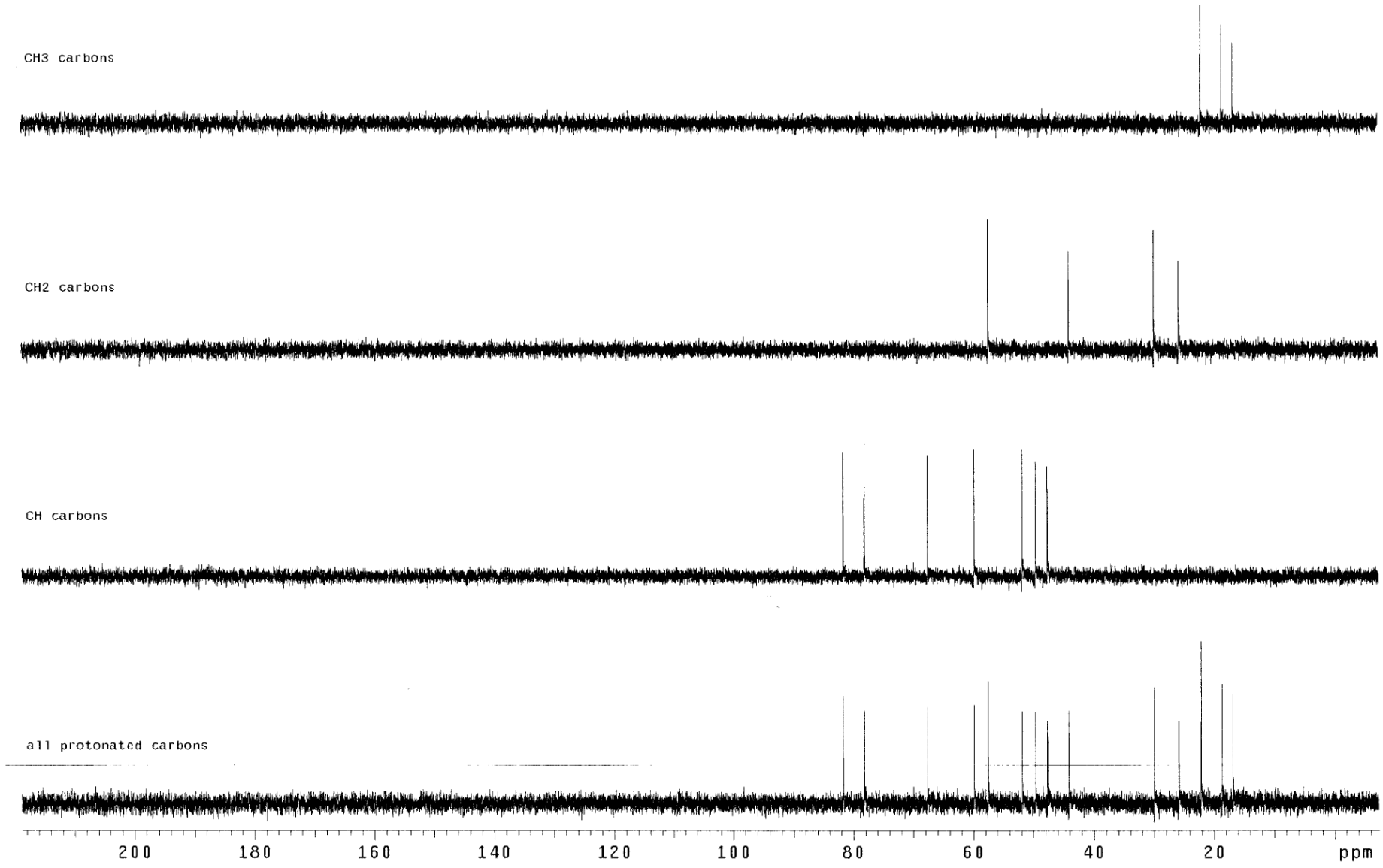


TY2-358

Pulse Sequence: hetcor
 Solvent: D2O
 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
 32 repetitions
 256 increments
OBSERVE C13, 125.6904822 MHz
DECOUPLE H1, 499.8652159 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, 46 min, 5 sec

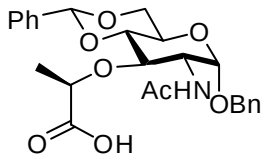


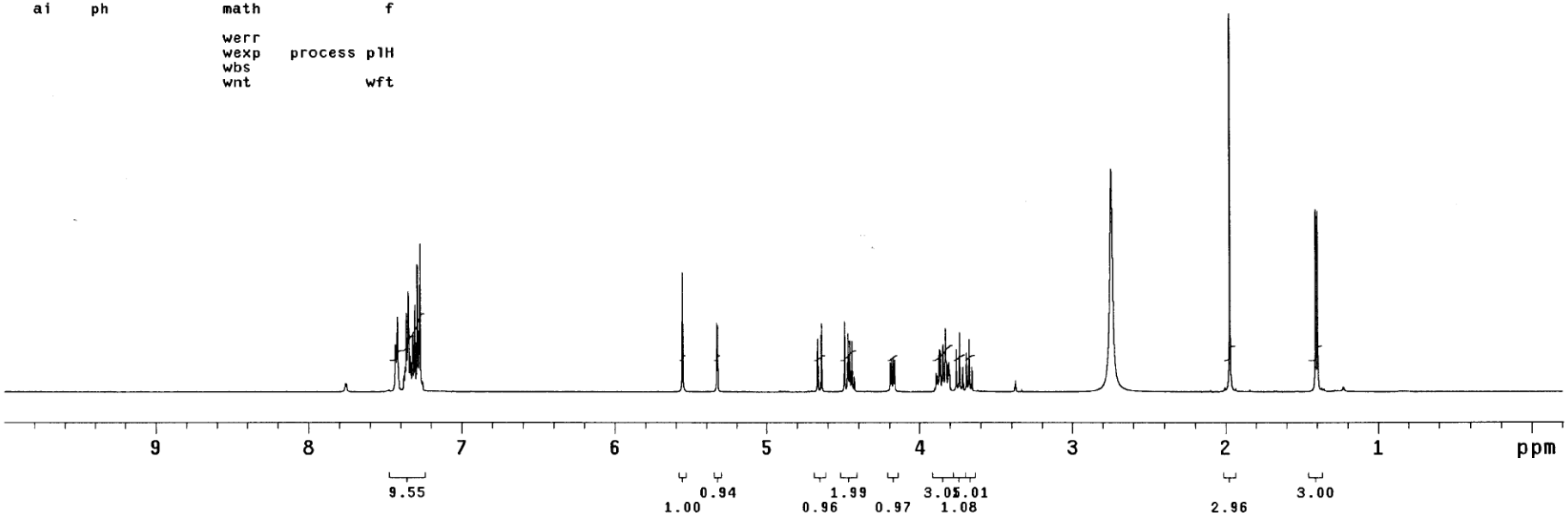

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

TY2-430

exp2 s2pul

SAMPLE		DEC. & VT	
date	Aug 17 2009	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	-102.6	dof3	0
wp	5099.3	dm3	n
vs	29	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	45.41	homo3	n
rfl	4140.1	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	3.000	fn	65536
ai	ph	math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft

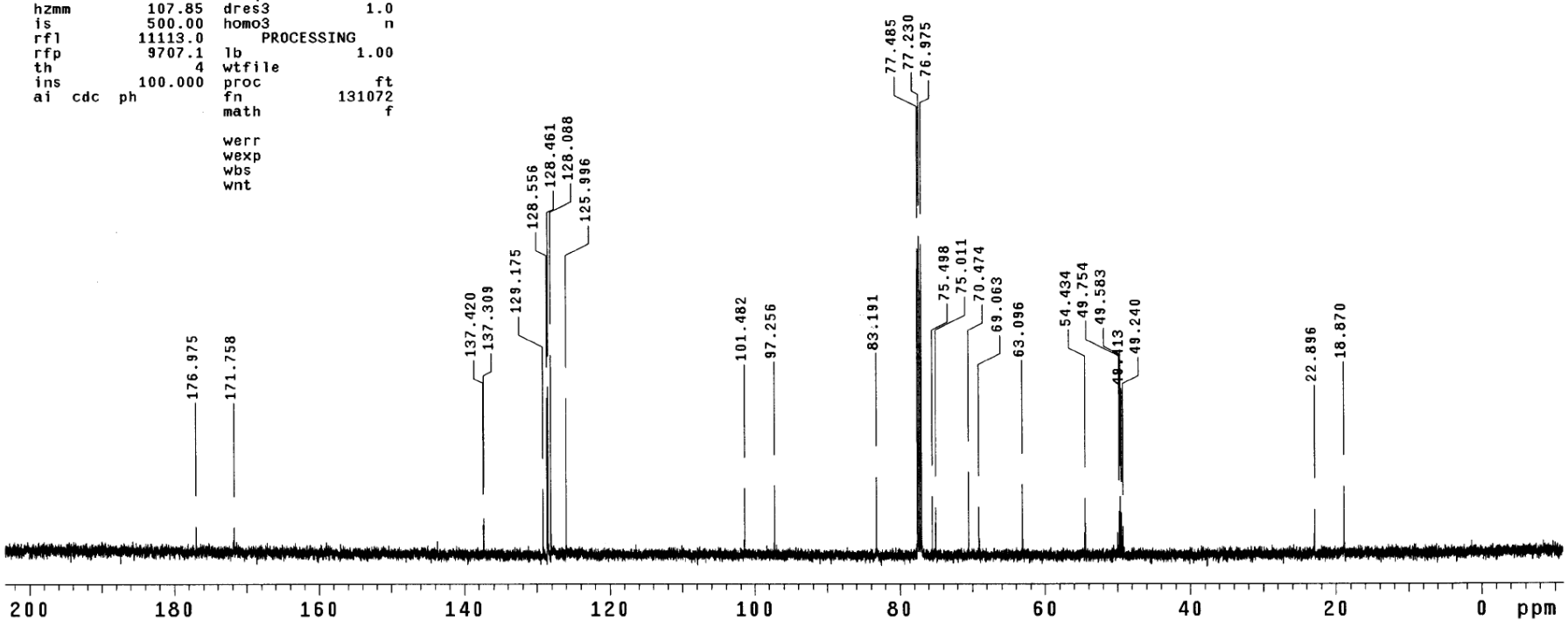




TY2-430

exp3 s2pul

SAMPLE		DEC. & VT	
date	Aug 17 2009	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	3000	dm2	n
ct	316	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	-1405.6	dof3	0
wp	26962.9	dm3	n
vs	422	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11113.0	PROCESSING	
rfp	9707.1	lb	1.00
th	4	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

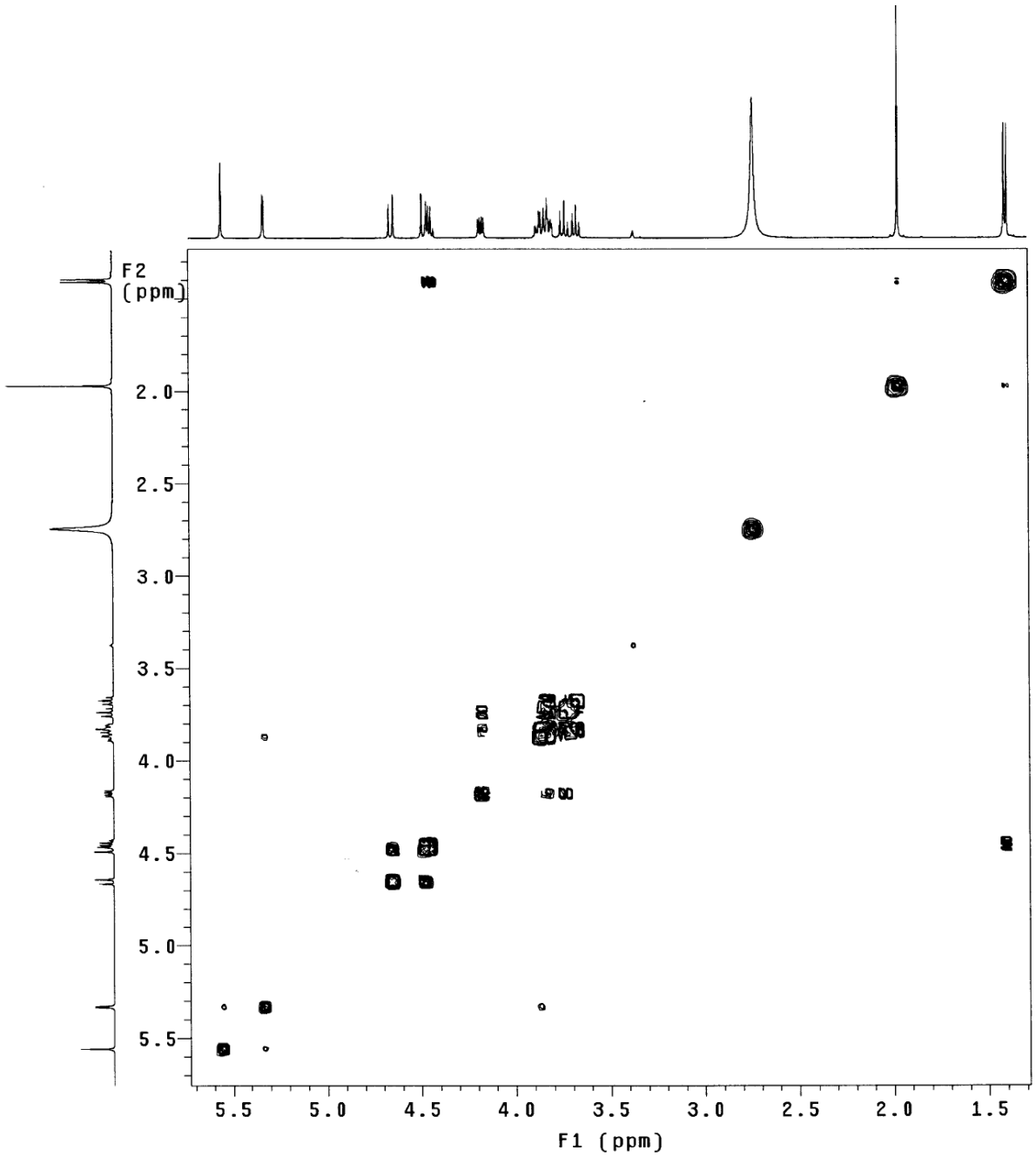


TY2-430

Pulse Sequence: relayh

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

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611707 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

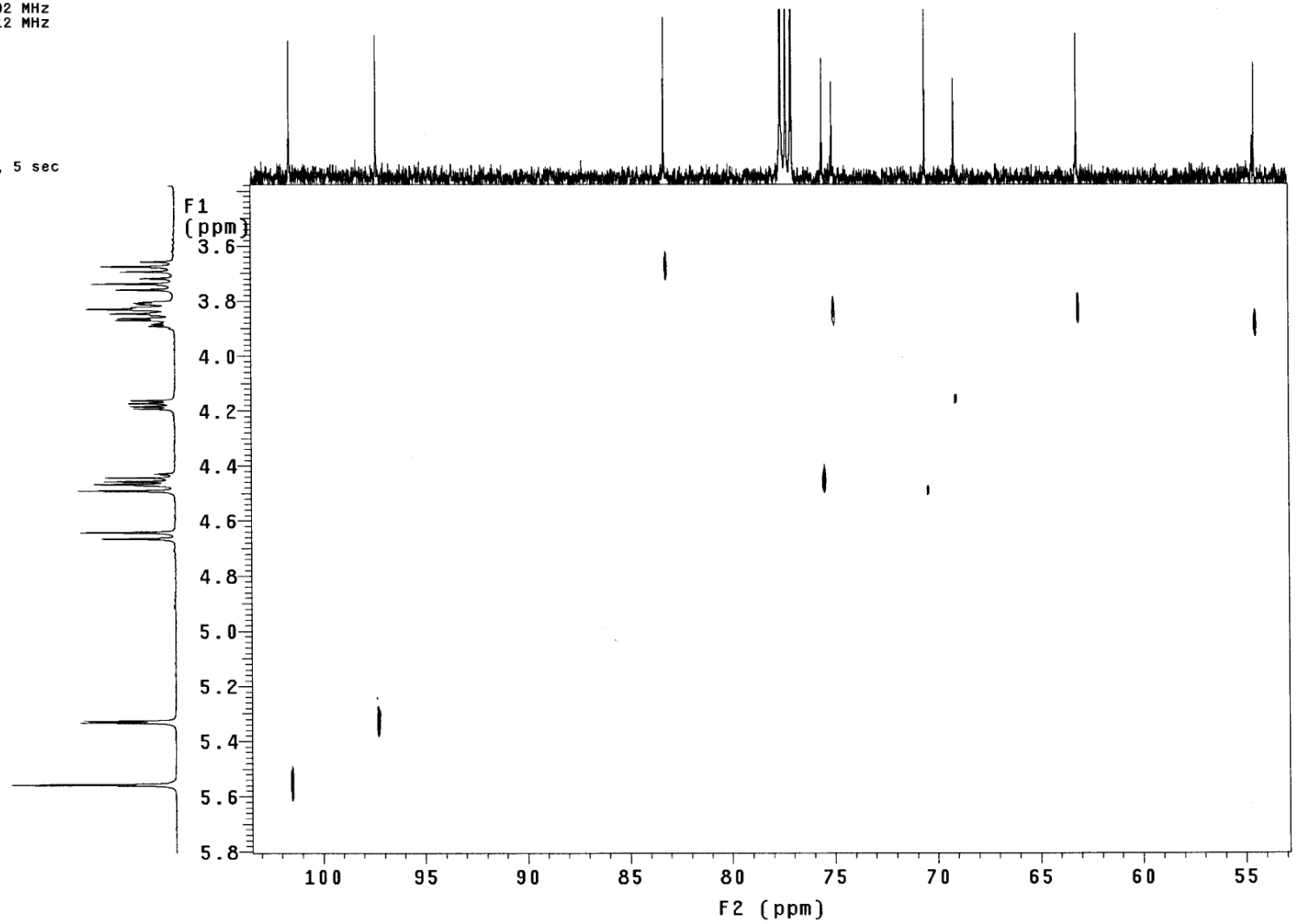


TY2-430

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
32 repetitions
256 increments
OBSERVE C13, 125.6901702 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 3 hr, 46 min, 5 sec



TY2-430

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

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 Line broadening 1.0 Hz
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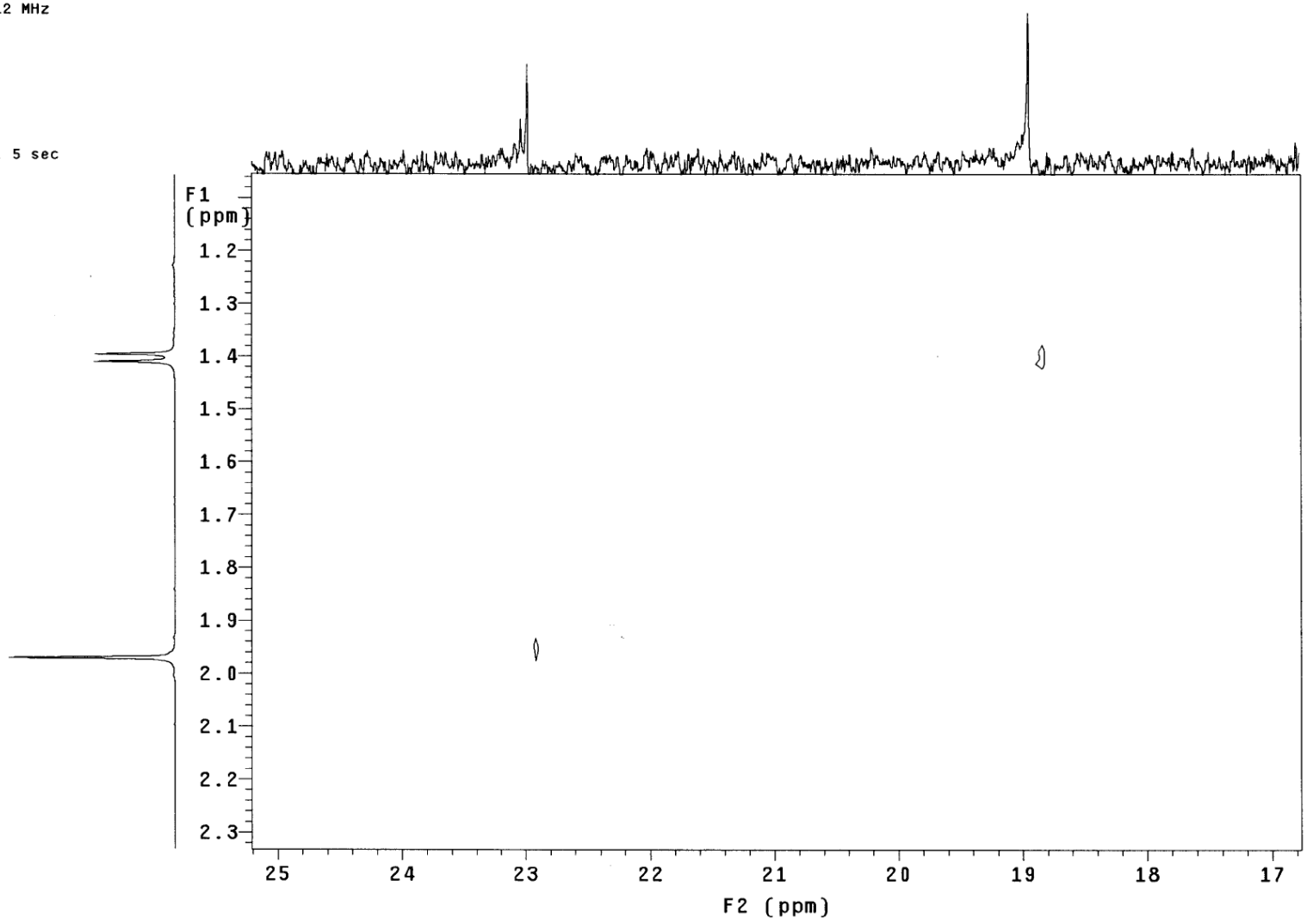
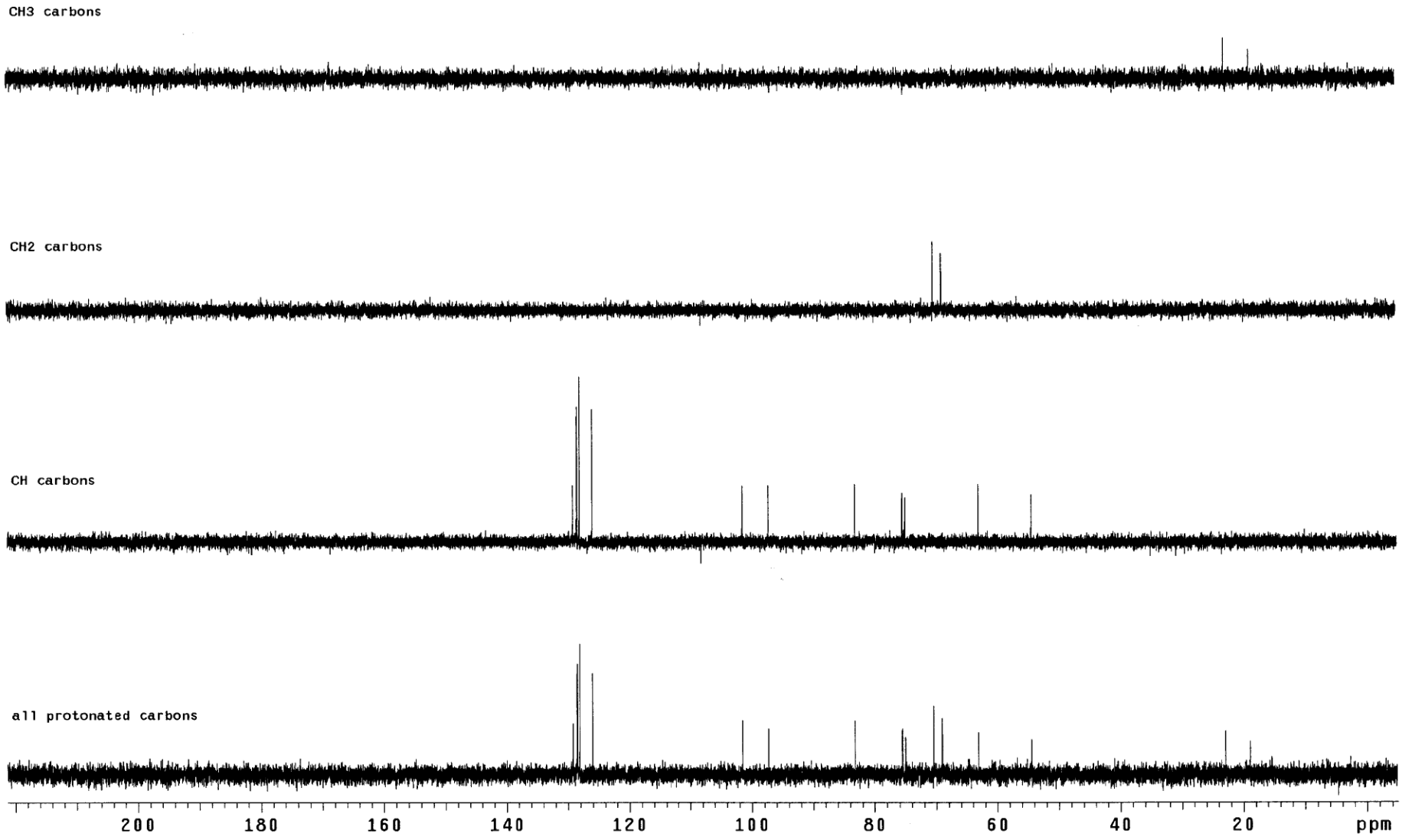
 Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 3 hr, 46 min, 5 sec




CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm