Supporting Information

Sulfonylation-Induced *N*- to *O*-Acetyl Migration in 2-Acetamidoethanol Derivatives

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Contents

Experimental Procedures

Benzyl 2-acetamido-4,6-*O*-benzylidene-2-deoxy-α-D-glucopyranoside (1a)

The compound **1a** was prepared from D-(+)-glucosamine by a variation of the known procedures.[1] 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 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 onto 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, filtered white solid was washed well with MeOH and dried under vacuum to afford **1a** (67.7 g, 88%) as a white powder. **1a**: ^{1}H NMR (500 HMz, DMSO-d_6) δ 1.85 (s, 3H, CH_3CO), 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.

General procedures for the tosylation of 2-acetamidoethanol derivatives (Condition A or B)

Condition A: *p*-Toluenesulfonyl chloride (1.05 equiv) and pyridine (3.0 equiv) were added to a solution of substrate in CH_2Cl_2 at 0 °C, and the mixture was stirred at room temperature for 24 h. The solvent was removed under reduced pressure, and the crude product was purified by silica gel column chromatography. **Condition B:** *p*-Toluenesulfonyl chloride (2.0 equiv) was added to a solution of substrate in pyridine, and the mixture was refluxed for 30 h. The mixture was cooled to room temperature, and the solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatography.

Products of the tosylation of the compound **1a**

Benzyl 2-acetamido-4,6-*O*-benzylidene-2-deoxy-3-*O*-*p*-toluenesulfonyl-α-D-glucopyranoside (2a)
Benzyl 3-*O*-acetyl-4,6-*O*-benzylidene-2-deoxy-2-*p*-toluenesulfoneamido-α-D-glucopyranoside (3a)

The condition B was used for compound **1a** (100 mg, 0.25 mmol), and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3) to afford the product **2a** (46 mg, 33%) as a white solid and the product **3a** (74 mg, 53%) as a white solid. **2a**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 2.02 (s, 3H, CH_3CO), 2.28 (s, 3H, CH_3Ph), 3.68 (dd, J = 9.6, 9.6 Hz, 1H, H-4), 3.72 (dd, J = 10.7, 10.7 Hz, 1H, H-6a), 3.88 (ddd, J = 4.8, 9.8, 9.8 Hz, 1H, H-5), 4.21 (dd, J = 5.0, 10.4 Hz, 1H, H-6b), 4.46 (ddd, J = 3.6, 9.2, 10.4 Hz, 1H, H-2), 4.53 and 4.73 (AB, J = 11.9 Hz, 2H, OCH_2Ph), 4.92 (dd,

[1] (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.

J = 10.0, 10.0 Hz, 1H, H-3), 5.02 (d, *J* = 3.8 Hz, 1H, H-1), 5.37 (s, 1H, C*H*Ph), 6.00 (d, *J* = 9.0 Hz, 1H, N*H*), 6.97 (d, *J* = 8.0 Hz, 2H, Ts), 7.19–7.44 (m, 10H), 7.67 (d, *J* = 8.4 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.9 (*C*H_3Ph), 23.4 (*C*H_3CO), 52.5 (C-2), 63.6 (C-5), 68.9 (C-6), 70.6 (O*C*H_2Ph), 78.4 (C-3), 79.2 (C-4), 98.0 (C-1), 101.8 (*C*HPh), 126.4, 128.1, 128.3, 128.4, 128.6, 128.9, 129.2, 129.5, 133.9, 136.7, 136.8, 144.6, 170.6 (*C*=O); IR 3324, 1656, 1547, 1362, 1182, 1123, 1097 cm^{-1}; HRMS (FAB) calcd for $C_{29}H_{32}NO_8S$ ($M + H^+$), 554.1849, found 554.1848. **3a**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.72 (s, 3H, C*H*$_3$CO), 2.37 (s, 3H, C*H*$_3$Ph), 3.53 (ddd, *J* = 3.8, 10.2, 10.2 Hz, 1H, H-2), 3.56 (dd, *J* = 9.7, 9.7 Hz, 1H, H-4), 3.67 (dd, *J* = 10.3, 10.3 Hz, 1H, H-6a), 3.83 (ddd, *J* = 4.8, 9.8, 9.8 Hz, 1H, H-5), 4.15 (dd, *J* = 4.8, 10.4 Hz, 1H, H-6b), 4.37 and 4.62 (AB, *J* = 11.6 Hz, 2H, OC*H*$_2$Ph), 4.71 (d, *J* = 3.8 Hz, 1H, H-1), 5.00 (d, *J* = 10.2 Hz, 1H, N*H*), 5.24 (dd, *J* = 10.1, 10.1 Hz, 1H, H-3), 5.42 (s, 1H, C*H*Ph), 7.21 (d, *J* = 8.4 Hz, 2H, Ts), 7.26–7.39 (m, 10H), 7.62 (d, *J* = 8.4 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 20.8 (*C*H_3CO), 21.7 (*C*H_3Ph), 56.8 (C-2), 63.1 (C-5), 68.9 (C-6), 69.4 (C-3), 70.5 (O*C*H_2Ph), 79.4 (C-4), 98.0 (C-1), 101.7 (*C*HPh), 126.3, 127.1, 128.4, 128.6, 128.6, 128.9, 129.3, 129.9, 136.4, 137.0, 138.4, 143.7, 170.8 (*C*=O); IR 3356, 1734, 1498, 1337, 1234, 1163, 1090 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{31}NNaO_8S$ ($M + Na^+$), 576.1659, found 576.1662.

_N_-(4-Acetamidobenzyl)acetamide (14)

Acetic anhydride (3.4 mL, 36 mmol) and pyridine (6.6 mL, 82 mmol) were added to a solution of 4-aminobenzylamine (2.0 g, 16 mmol) in CH_2Cl_2 (100 mL), and the mixture was stirred for 40 h. After removal of the solvent and extra reagent under reduced pressure, the resulting yellow solid was washed with Et_2O to afford the product **14** (3.3 g, 99%) as a light yellow solid. **14**: ^{1}H NMR (500 HMz, CD_3OD) δ 1.97 (s, 3H, C*H*$_3$CO), 2.11 (s, 3H, C*H*$_3$CO), 4.30 (s, 2H, C*H*$_2$), 7.22 (d, *J* = 8.6 Hz, 2H), 7.49 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (126 MHz, CD_3OD) δ 22.7 (*C*H_3CO), 23.9 (*C*H_3CO), 43.9 (*C*H_2), 121.4, 129.3, 135.8, 139.2, 171.8 (*C*=O), 173.2 (*C*=O); HRMS (ESI) calcd for $C_{11}H_{14}N_2NaO_2$ ($M + Na^+$), 229.0947, found 229.0966.

***trans*-2-(Acetamido)cyclohexanol (1b)**

Sodium methoxide (356 mg, 6.6 mmol) was added to a solution of *trans*-2-aminocyclohexanol hydrochloride (1.0 g, 6.6 mmol) in MeOH (30 mL), and the mixture was stirred for 15 min. After addition of acetic anhydride (0.69 mL, 7.3 mmol), the resulting mixture was stirred at room temperature for 40 h, and then the solvent and extra reagent were removed under reduced pressure. $CHCl_3$ was added to the residue and filtrated. The combined filtrate was concentrated to dryness, and the resulting colorless paste was added hexane. The resulting suspension was filtrated to afford the product **1b** (1.03 g, quant.) as a white solid. **1b**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.11–1.38 (m, 4H), 1.64–1.77 (m, 2H), 1.94 (m, 1H), 2.01 (s, 3H, C*H*$_3$CO), 2.05 (m, 1H), 3.31 (ddd, *J* = 4.4, 10.0, 10.0, 1H, H-2), 3.60 (m, 1H, H-1), 5.64 (br s, 1H, O*H*), 6.16 (br s, 1H, N*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.5 (*C*H_3CO), 24.3 and 24.8 (C-4, C-5), 31.7 (C-3), 34.6 (C-6), 55.9 (C-2), 75.0 (C-1), 172.3 (*C*=O); IR 3275, 1646, 1562, 1450, 1376, 1140, 1069 cm^{-1}; HRMS (ESI) calcd for $C_8H_{16}NO_2$ ($M + H^+$), 158.1176, found 158.1197.

Products of the tosylation of the compound **1b**

***trans*-2-Acetamido-1-*O*-tosyl-cyclohexanol (2b)**

***trans*-1-*O*-Acetyl-2-(tosylamido)cyclohexanol (3b)**

***trans*-2-(Tosylamido)cyclohexanol (15b)**

***trans*-2-Acetamido-1-*O*-acetyl-cyclohexanol (16b)**

The condition A was used for compound **1b** (150 mg, 0.93 mmol), and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3 to 1:0) to afford the product **2b** (27 mg, 9%) as a white solid, the product **3b** (107 mg, 36%) as a white solid, the product **15b** (64 mg, 25%) as a white solid and the product **16b** (36 mg, 19%) as a white solid. The structures of the products **3b**,

15b and **16b** were confirmed by comparison to the reported data.[2] **2b**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.10–1.37 (m, 3H), 1.54 (m, 1H), 1.64 (m, 1H), 1.73 (m, 1H), 1.88 (m, 1H), 1.94 (s, 3H, C*H*$_3$CO), 2.19 (m, 1H), 2.46 (s, 3H, C*H*$_3$Ph), 3.85 (m, 1H, H-2), 4.34 (ddd, *J* = 4.6, 10.8, 10.8 Hz, 1H, H-1), 5.78 (d, *J* = 7.2 Hz, 1H, N*H*), 7.35 (d, *J* = 8.6 Hz, 2H, Ts), 7.78 (d, *J* = 8.0 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.9 (*C*H$_3$Ph), 23.7 (*C*H$_3$CO), 24.0 (C-4), 24.4 (C-5), 32.1 (C-6), 32.2 (C-3), 52.5 (C-2), 82.5 (C-1), 127.8, 130.1, 134.2, 145.1, 170.2 (*C*=O); IR 3436, 1638, 1561, 1370, 1188, 1175 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{21}NNaO_4S$ (M + Na$^+$), 334.1083, found 334.1075. **3b**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.14–1.38 (m, 4H), 1.64 (m, 1H), 1.69 (m, 1H), 1.76 (s, 3H, C*H*$_3$CO), 1.92 (m, 1H), 2.02 (m, 1H), 2.42 (s, 3H, C*H*$_3$Ph), 3.19 (m, 1H, H-2), 4.55 (ddd, *J* = 4.5, 10.2, 10.2 Hz, 1H, H-1), 4.95 (d, *J* = 7.6 Hz, 1H, N*H*), 7.29 (d, *J* = 8.0 Hz, 2H, Ts), 7.74 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.1 (*C*H$_3$CO), 21.7 (*C*H$_3$Ph), 23.9 and 24.4 (C-4, C-5), 31.3 (C-6), 33.7 (C-3), 57.1 (C-2), 74.3 (C-1), 127.1, 129.8, 138.8, 143.2, 171.6 (*C*=O); IR 3261, 1715, 1599, 1453, 1335, 1257, 1163, 1094, 1046 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{22}NO_4S$ (M + H$^+$), 312.1264, found 312.1277. **15b**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.05–1.28 (m, 4H), 1.58 (m, 1H), 1.65 (m, 1H), 1.73 (m, 1H), 2.01 (m, 1H), 2.43 (s, 3H, C*H*$_3$Ph), 2.76 (br s, 1H, O*H*), 2.86 (m, 1H, H-2), 3.31 (ddd, *J* = 4.3, 9.6, 9.6 Hz, 1H, H-1), 5.10 (d, *J* = 7.0 Hz, 1H, N*H*), 7.32 (d, *J* = 8.0 Hz, 2H, Ts), 7.80 (d, *J* = 8.4 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.8 (*C*H$_3$Ph), 24.0, 24.8, 32.0, 33.5, 59.9 (C-2), 73.5 (C-1), 127.3, 130.0, 137.5, 143.8; HRMS (ESI) calcd for $C_{13}H_{20}NO_3S$ (M + H$^+$), 270.1158, found 270.1129. **16b**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.14 (m, 1H), 1.19–1.35 (m, 2H), 1.44 (m, 1H), 1.66 (m, 1H), 1.74 (m, 1H), 1.89 (s, 3H, C*H*$_3$CO), 1.92 (m, 1H), 2.01 (s, 3H, C*H*$_3$CO), 2.03 (m, 1H), 3.83 (dddd, *J* = 4.2, 4.4, 10.2, 10.2 Hz, 1H, H-2), 4.61 (ddd, *J* = 4.5, 10.7, 10.7 Hz, 1H, H-1), 5.81 (br s, 1H, N*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.3 (*C*H$_3$CO), 23.5 (*C*H$_3$CO), 24.2 and 24.3 (C-4, C-5), 31.2 (C-6), 32.2 (C-3), 52.9 (C-2), 74.8 (C-1), 169.9 (*C*=O), 172.1 (*C*=O); IR 3289, 1732, 1646, 1559, 1375, 1246, 1042 cm^{-1}; HRMS (ESI) calcd for $C_{10}H_{17}NNaO_3$ (M + Na$^+$), 222.1101, found 222.1104.

Products of the tosylation of 2-acetamidoethanol (**1c**)

1-*O*-Acetyl-2-(tosylamido)ethanol (3c)

2-(Tosylamido)ethanol (15c)

2-Acetamido-1-*O*-acetyl-ethanol (16c)

The condition A was used for 2-acetamidoethanol (1.12 g, 10.9 mmol), and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3 to 1:0) to afford the product **3c** (1.01 g, 36%) as a colorless oil, the product **15c** (748 mg, 32%) as a white paste and the product **16c** (457 mg, 29%) as a colorless oil. **3c**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 2.00 (s, 3H, C*H*$_3$CO), 2.43 (s, 3H, C*H*$_3$Ph), 3.22 (dt, *J* = 5.4, 5.7 Hz, 1H, H-2), 4.09 (t, *J* = 5.3 Hz, 1H, H-1), 5.06 (t, *J* = 5.6 Hz, 1H, N*H*), 7.32 (d, *J* = 7.8 Hz, 2H, Ts), 7.76 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 20.9 (*C*H$_3$CO), 21.7 (*C*H$_3$Ph), 42.3 (C-2), 63.1 (C-1), 127.2, 130.0, 137.0, 143.9, 171.0 (*C*=O); IR 3237, 1736, 1719, 1596, 1441, 1331, 1252, 1160, 1091, 1051 cm^{-1}; HRMS (ESI) calcd for $C_{11}H_{15}NNaO_4S$ (M + Na$^+$), 280.0614, found 280.0603. **15c**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 2.42 (s, 3H, C*H*$_3$Ph), 3.08 (m, 1H, H-2), 3.61 (m, 1H, O*H*), 3.68 (m, 1H, H-1), 5.42 (br m, 1H, N*H*), 7.31 (d, *J* = 7.8 Hz, 2H, Ts), 7.76 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.7 (*C*H$_3$Ph), 45.4 (C-2), 61.4 (C-1), 127.3, 130.0, 136.7, 143.8; HRMS (ESI) calcd for $C_9H_{13}NNaO_3S$ (M + Na$^+$), 238.0508, found 238.0497. **16c**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.98 (s, 3H, C*H*$_3$CO), 2.06 (s, 3H, C*H*$_3$CO), 3.49 (m, 1H, H-2), 4.13 (m, 1H, H-1), 5.07 (br s, 1H, N*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.0 (*C*H$_3$CO), 23.3 (*C*H$_3$CO), 38.9 (C-2), 63.4 (C-1), 171.3

[2] For compound **3b**: (a) Liu, Y-K.; Li, R.; Yue, L.; Li, B-J.; Chen, Y-C.; Wu, Y.; Ding, L-S. *Org. Lett.* **2006**, *8*, 1521−1524. For compound **15b**: (b) Wang, Z.; Cui, Y-T.; Xu, Z-B.; Qu J. *J. Org. Chem.* **2008**, *73*, 2270−2274. (c) Fan, R-H.; Hou, X-L. *Org. Biomol. Chem.* **2003**, *1*, 1565−1567. For compound **16b**: (d) Miller, S. J.; Copeland, G. T.; Papaioannou, N.; Horstmann, T. E.; Ruel, E. M. *J. Am. Chem. Soc.* **1998**, *120*, 1629−1630. (e) Tabanella, S.; Valancogne, I.; Jackson, R. F. W. *Org. Biomol. Chem.* **2003**, *1*, 4254−4261.

(*C*=O); IR 3294, 1740, 1658, 1557, 1375, 1237, 1054 cm^{-1}; HRMS (ESI) calcd for $C_6H_{11}NNaO_3$ (M + Na^+), 168.0631, found 168.0625.

1-(Acetamidomethyl)cyclohexanol (1d)

Sodium methoxide (489 mg, 9.1 mmol) was added to a solution of 1-aminomethyl-1-cyclohexanol hydrochloride (1.5 g, 9.1 mmol) in MeOH (30 mL), and the mixture was stirred for 15 min. After addition of acetic anhydride (0.94 mL, 10 mmol), the resulting mixture was stirred at room temperature for 25 h, and then the solvent and extra reagent were removed under reduced pressure. CH_2Cl_2 was added to the residue, and the resulting suspension was filtrated. The combined filtrate was concentrated to afford the product **1d** (1.55 g, quant.) as a white solid. **1d**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.25–1.63 (m, 10H), 2.01 (s, 3H, C*H*$_3$CO), 2.80 (br s, 1H), 3.25 (d, *J* = 6.0 Hz, 2H, C*H*$_2$NHAc), 6.32 (br s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 22.2, 23.4, 25.9, 35.7, 49.3 (*C*H$_2$NHAc), 71.7 (C-1), 171.5 (*C*=O); IR 3391, 3290, 1652, 1552, 1421, 1292, 1274, 1166, 1127, 1044 cm^{-1}; HRMS (ESI) calcd for $C_9H_{17}NNaO_2$ (M + Na^+), 194.1151, found 194.1147.

Product of the tosylation of the compound **1d**

1-*O*-Acetyl-1-[(tosylamido)methyl]cyclohexanol (3d)

The condition A was used for compound **1d** (150 mg, 0.88 mmol), and the crude product was purified by silica gel column chromatography (AcOEt) to afford the product **3d** (148 mg, 52%) as a white solid. The condition B gave the poduct **3d** in 96% from the same amount of starting material. **3d**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.29 (m, 2H), 1.37–1.59 (m, 6H), 1.98 (s, 3H, C*H*$_3$CO), 2.08 (m, 2H), 2.42 (s, 3H, C*H*$_3$Ph), 3.25 (d, *J* = 6.6 Hz, 2H, C*H*$_2$NHTs), 5.53 (t, *J* = 6.6 Hz, 1H, N*H*), 7.29 (d, *J* = 7.8 Hz, 2H, Ts), 7.72 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.7 (*C*H$_3$Ph), 21.7 (C-3, C-5), 22.3 (*C*H$_3$CO), 25.4 (C-4), 32.8 (C-2, C-6), 49.4 (*C*H$_2$NHTs), 83.0 (C-1), 127.1, 129.9, 137.3, 143.4, 171.4 (*C*=O); IR 3284, 1734, 1599, 1450, 1328, 1236, 1163, 1094, 1064 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{23}NNaO_4S$ (M + Na^+), 348.1240, found 348.1246.

2-Acetamido-1,5-anhydro-4,6-*O*-benzylidene-2-deoxy-D-glucitol (1e)

Sodium methoxide (110 mg, 2.1 mmol) was added to a solution of 2-acetamido-3-*O*-acetyl-1,5-anhydro-4,6-*O*-benzylidene-2-deoxy-D-glucitol[3] (700 mg, 2.1 mmol) in MeOH (15 mL), and the mixture was stirred for 3 h at room temperature. The reaction mixture was quenched by the addition of the Amberlite IR-120 (H^+), and the resulting mixture was stirred for 5 min. The resin was filtered, and the filtrate was concentrated to dryness to afford the product **1e** (610 mg, quant.) as a white solid. **1e**: ^{1}H NMR (500 HMz, $CDCl_3$/CD_3OD = 95:5) δ 1.95 (s, 3H, C*H*$_3$CO), 3.15 (dd, *J* = 11.0, 11.0 Hz, 1H, H-1a), 3.32 (ddd, *J* = 5.2, 9.6, 9.6 Hz, 1H, H-5), 3.50 (dd, *J* = 9.2, 9.2 Hz, 1H, H-4), 3.62 (dd, *J* = 9.5, 9.5 Hz, 1H, H-3), 3.68 (dd, *J* = 10.4, 10.4 Hz, 1H, H-6a), 3.94 (ddd, *J* = 5.4, 10.4, 10.4 Hz, 1H, H-2), 4.05 (dd, *J* = 5.5, 11.1 Hz, 1H, H-1b), 4.26 (dd, *J* = 5.0, 10.6 Hz, 1H, H-6b), 5.52 (s, 1H, C*H*Ph), 7.30–7.50 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$/CD_3OD = 95:5) δ 22.9 (*C*H$_3$CO), 52.2 (C-2), 68.7 (C-1), 68.8 (C-6), 71.5 (C-5), 72.4 (C-3), 82.1 (C-4), 102.0 (*C*HPh), 126.4, 128.4, 129.4, 137.2, 172.3 (*C*=O); IR 3426, 3277, 1655, 1544, 1377, 1127, 1100 cm^{-1}; HRMS (FAB) calcd for $C_{15}H_{20}NO_5$ (M + H^+), 294.1336, found 294.1339.

Product of the tosylation of the compound **1e**

3-*O*-Acetyl-1,5-anhydro-4,6-*O*-benzylidene-2-deoxy-2-tosylamido-D-glucitol (3e)

The condition B was used for compound **1e** (50 mg, 0.17 mmol), and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:3) to afford the product **3e** (93 mg, 94%) as a white solid. **3e**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.70 (s, 3H, C*H*$_3$CO), 2.44 (s, 3H, C*H*$_3$Ph), 3.34 (dd, *J* = 11.3, 11.3 Hz, 1H, H-1a), 3.38 (ddd, *J* = 5.0, 9.6, 9.6 Hz, 1H, H-5), 3.49 (m, 1H, H-2), 3.56 (dd, *J* = 9.4,

[5] Hesek, D.; Lee, M.; Yamaguchi, T.; Noll, B. C.; Mobashery, S. *J. Org. Chem.* **2008**, *73*, 7349−7352.

9.4 Hz, 1H, H-4), 3.67 (dd, *J* = 10.3, 10.3 Hz, 1H, H-6a), 4.13 (dd, *J* = 5.4, 11.6 Hz, 1H, H-1b), 4.30 (dd, *J* = 5.0, 10.6 Hz, 1H, H-6b), 4.96 (dd, *J* = 9.7, 9.7 Hz, 1H, H-3), 5.32 (d, *J* = 6.8 Hz, 1H, N*H*), 5.45 (s, 1H, C*H*Ph), 7.33 (d, *J* = 7.8 Hz, 2H, Ts), 7.30–7.42 (m, 5H), 7.75 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 20.8 (*C*H_3CO), 21.7 (*C*H_3Ph), 54.6 (C-2), 68.8 (C-6), 70.8 (C-1), 71.6 (C-5), 73.0 (C-3), 79.0 (C-4), 101.7 (*C*HPh), 126.3, 127.2, 128.4, 129.4, 130.0, 137.0, 137.7, 143.8, 172.3 (*C*=O); IR 3287, 1745, 1455, 1227, 1167, 1088 cm^{-1}; HRMS (FAB) calcd for $C_{22}H_{26}NO_7S$ (M + H^+), 448.1424, found 448.1421.

Product of the tosylation of 2-acetamidophenol (**1f**)

1-*O*-Tosyl-2-(acetamido)phenol (2f)

The condition A was used for 2-acetamidophenol (1.0 g, 6.6 mmol), and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 1:1) to afford the product **2f** (1.98 g, 98%) as colorless crystals. The condition B gave the poduct **2f** in 98% from the same amount of starting material. **2f**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 2.07 (s, 3H, C*H*$_3$CO), 2.47 (s, 3H, C*H*$_3$Ph), 6.91 (d, *J* = 8.0 Hz, 1H, C-3), 6.99 (dd, *J* = 7.8, 7.8 Hz, 1H, C-4), 7.24 (dd, *J* = 7.8, 7.8 Hz, 1H, C-5), 7.35 (d, *J* = 8.0 Hz, 2H, Ts), 7.56 (br s, 1H, N*H*), 7.73 (d, *J* = 8.4 Hz, 2H, Ts), 8.19 (d, *J* = 8.2 Hz, 1H, C-6); ^{13}C NMR (126 MHz, $CDCl_3$) δ 22.0 (*C*H_3Ph), 24.8 (*C*H_3CO), 123.0, 123.0, 124.5, 128.0, 128.7, 130.3, 131.3, 131.9, 139.1, 146.5, 168.3 (*C*=O); IR 3400, 3302, 1695, 1675, 1600, 1525, 1442, 1372, 1307, 1194, 1170, 1088 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{15}NNaO_4S$ (M + Na^+), 328.0614, found 328.0599.

Crystal was examined under Infineum V8512 oil. A suitable sample was glued to a glass capillary and then transferred to the 100 K N_2 stream for compound **2f** of a Bruker SMART Apex CCD diffractometer. Preliminary unit cell parameters and crystal system analysis were determined from the centroids of reflections with I > 20σ(I) from three sets of 30 0.5° ω-scan frames (φ = 0, 120, 240°); final unit cell parameters were determined from the centroids of reflections with I > 10σ(I) from all data. An arbitrary, redundant, sphere of data was calculated using COSMO, included in the Apex2 suite of programs.[4] Data are uncorrected for absorption and Lorentz polarization effects. Structure solution and refinement utilized the programs of the SHELXTL software package.[5] Full detail of the X-ray structure determination is in the CIF file and ORTEP diagram is given in Figure S1.

***trans*-4-(Acetamido)cyclohexanol (1g)**

Sodium methoxide (1.07 g, 19.8 mmol) was added to a solution of *trans*-4-aminocyclohexanol hydrochloride (3.0 g, 19.8 mmol) in MeOH (100 mL), and the mixture was stirred for 15 min. After addition of acetic anhydride (2.06 mL, 21.8 mmol), the mixture was stirred at room temperature for 26 h, and then the solvent and extra reagent were removed under reduced pressure. $CHCl_3$ was added to the residue, and the resulting suspension was filtrated. The combined filtrate was concentrated to dryness, and the resulting colorless paste was added hexane. The resulting suspension was filtrated to afford the product **1g** (3.11 g, quant.) as a white solid. **1g**: ^{1}H NMR (500 HMz, CD_3OD) δ 1.20–1.40 (m, 4H), 1.84–1.98 (m, 4H), 1.90 (s, 3H, C*H*$_3$CO), 3.51 (m, 1H), 3.59 (m, 1H), 7.92 (br d, *J* = 7.0 Hz, 1H, N*H*); ^{13}C NMR (126 MHz, CD_3OD) δ 22.8 (*C*H_3CO), 31.6 and 34.9 (C-2, C-3, C-5 and C-6), 49.3 (C-4), 70.5 (C-1), 172.6 (*C*=O); IR 3277, 1636, 1562, 1456, 1371, 1328, 1148, 1112, 1065 cm^{-1}; HRMS (ESI) calcd for $C_8H_{16}NO_2$ (M + H^+), 158.1176, found 158.1171.

Products of the tosylation of the compound **1g**

***trans*-4-Acetamido-1-*O*-tosyl-cyclohexanol (2g)**

***trans*-4-(Tosylamido)cyclohexanol (15g)**

***trans*-4-Acetamido-1-*O*-acetyl-cyclohexanol (16g)**

The condition A was used for compound **1g** (450 mg, 2.9 mmol), and the crude product was purified by silica gel column chromatography (AcOEt) to afford the product **2g** (389 mg, 41%) as colorless

[4] Apex2. Bruker-AXS: Madison, WI, 2008; Vol. 58.

[5] Sheldrick, G. M., *Acta Crystallogr. A.* **2008**, *64*, 112−122.

crystals, the product **15g** (69 mg, 9%) as a white solid and the product **16g** (46 mg, 8%) as colorless crystals. **2g**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.06–1.27 (m, 2H), 1.48–1.66 (m, 2H), 1.84–2.03 (m, 4H), 1.89 (s, 3H, C*H*$_3$CO), 2.43 (s, 3H, C*H*$_3$Ph), 3.68 (m, 1H, H-4), 4.40 (ddd, *J* = 3.9, 10.6, 10.6 Hz, 1H, H-1), 5.69 (d, *J* = 7.4 Hz, 1H, N*H*), 7.32 (d, *J* = 8.1 Hz, 2H, Ts), 7.75 (d, *J* = 8.1 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.8 (*C*H$_3$Ph), 23.5 (*C*H$_3$CO), 30.2 and 30.9 (C-2, C-3, C-5, C-6), 46.8 (C-4), 80.4 (C-1), 127.6, 130.0, 134.4, 144.9, 169.7 (*C*=O); IR 3408, 3312, 1674, 1652, 1552, 1519, 1343, 1189, 1177, 1096 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{21}NNaO_4S$ (M + Na$^+$), 334.1083, found 334.1072. **15g**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.10–1.24 (m, 4H), 1.69–1.86 (m, 4H), 2.39 (s, 3H, C*H*$_3$Ph), 3.01 (m, 1H, H-4), 3.46 (m, 1H, H-1), 5.39 (d, *J* = 7.6 Hz, 1H, N*H*), 7.26 (d, *J* = 8.2 Hz, 2H, Ts), 7.72 (d, *J* = 8.2 Hz, 2H, Ts); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.6 (*C*H$_3$Ph), 31.3 and 33.5 (C-2, C-3, C-5, C-6), 51.9 (C-4), 69.1 (C-1), 126.9, 129.8, 138.2, 143.4; HRMS (ESI) calcd for $C_{13}H_{19}NNaO_3S$ (M + Na$^+$), 292.0978, found 292.0955. **16g**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.13–1.31 (m, 2H), 1.35–1.53 (m, 2H), 1.86–2.03 (m, 4H), 1.91 (s, 3H, C*H*$_3$CO), 1.99 (s, 3H, C*H*$_3$CO), 3.72 (dtt, *J* = 4.0, 7.7, 11.3 Hz, 1H, H-4), 4.61 (tt, *J* = 4.0, 10.8 Hz, 1H, H-1), 5.81 (br d, *J* = 7.2 Hz, 1H, N*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.5 (*C*H$_3$CO), 23.5 (*C*H$_3$CO), 30.1 and 30.6 (C-2, C-3, C-4, C-5), 47.3 (C-4), 72.1 (C-1), 169.7 (*C*=O), 170.9 (*C*=O); IR 3440, 3251, 1726, 1638, 1562, 1365, 1250, 1035 cm^{-1}; HRMS (ESI) calcd for $C_{10}H_{17}NNaO_3$ (M + Na$^+$), 222.1101, found 222.1087.

Product of the mesylation of the compound **1d**

1-*O*-Acetyl-1-[(mesylamido)methyl]cyclohexanol (19)

Methanesulfonyl chloride (90 μL, 1.2 mmol) was added to a solution of **1d** (100 mg, 0.58 mmol) in pyridine (1.0 mL) at 0 °C. The mixture was stirred at room temperature for 20 h, and then at 40 °C for 1 h. The solvent was removed under reduced pressure, and the crude product was purified by silica gel column chromatography (AcOEt/hexane, 2:3) to afford the product **19** (137 mg, 94%) as a white solid. **19**: ^{1}H NMR (500 HMz, $CDCl_3$) δ 1.33 (m, 2H), 1.43–1.63 (m, 6H), 2.07 (s, 3H, C*H*$_3$CO), 2.13 (m, 2H), 2.95 (s, 3H, C*H*$_3$SO$_2$), 3.48 (d, *J* = 6.6 Hz, 2H, C*H*$_2$NHMs), 5.15 (t, *J* = 6.2 Hz, 1H, N*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.7 (C-3, C-5), 22.3 (*C*H$_3$CO), 25.4 (C-4), 32.9 (C-2, C-6), 40.6 (Ms), 49.8 (*C*H$_2$NHMs), 83.1 (C-1), 171.5 (*C*=O); IR 3249, 1733, 1453, 1302, 1232, 1150 cm^{-1}; HRMS (ESI) calcd for $C_{10}H_{19}NNaO_4S$ (M + Na$^+$), 272.0927, found 272.0908.

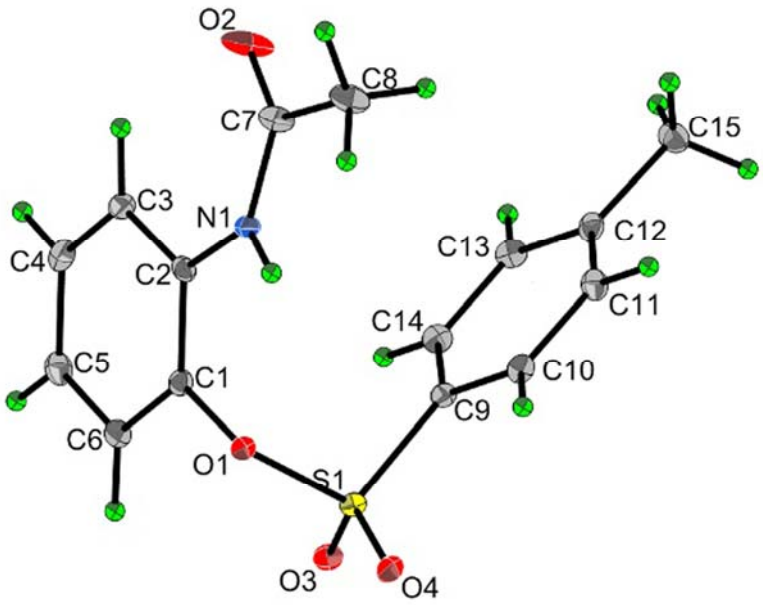
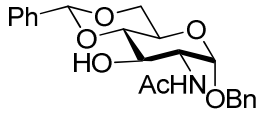


Figure S1. The ORTEP diagram of compound **2f**, shown at 30% probability level.



1a

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TY2-297

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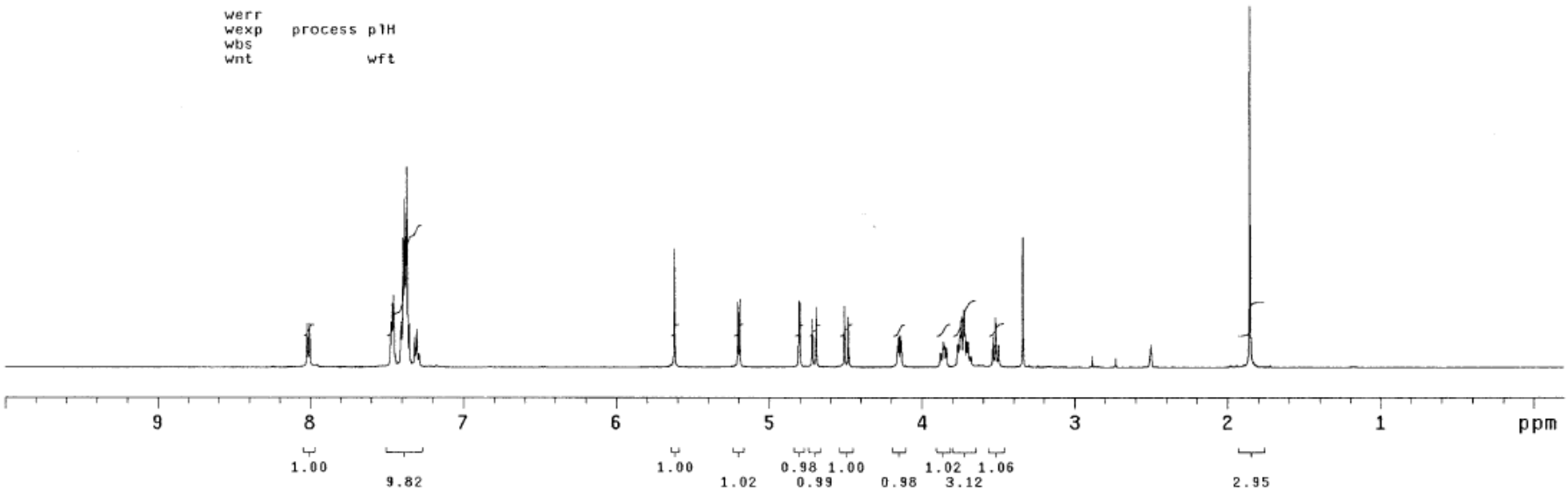

TY2-297

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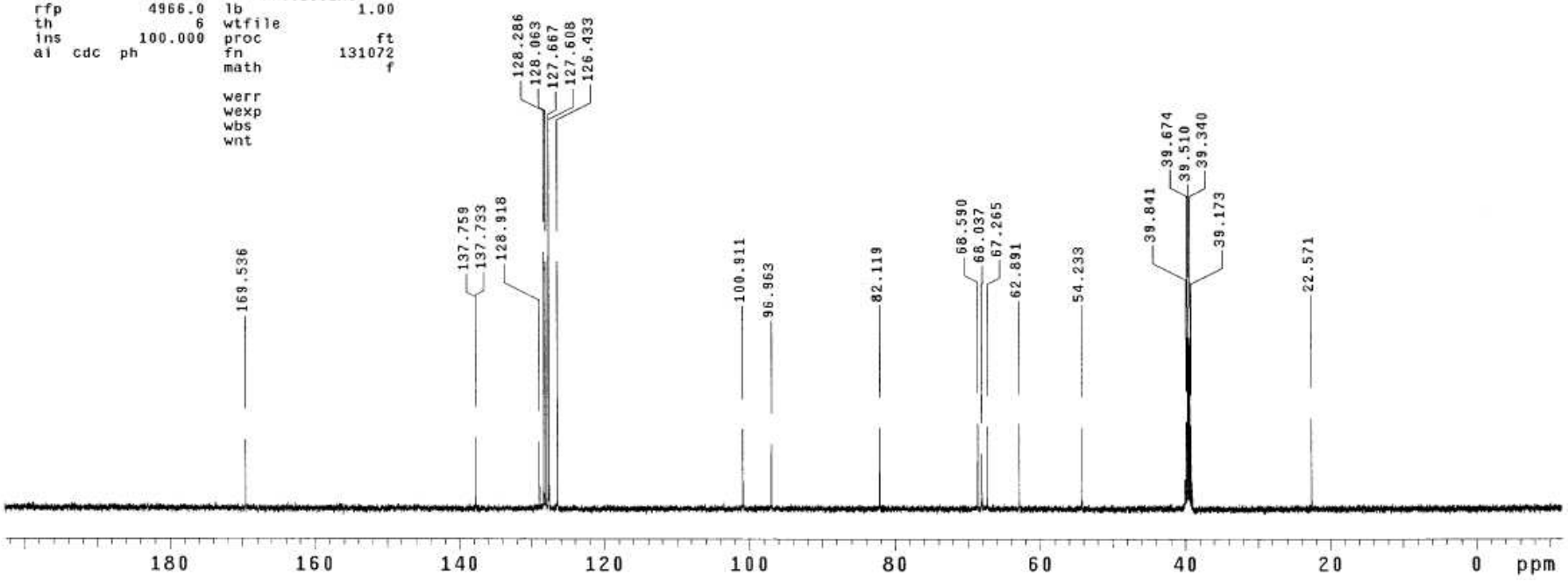

TY2-297

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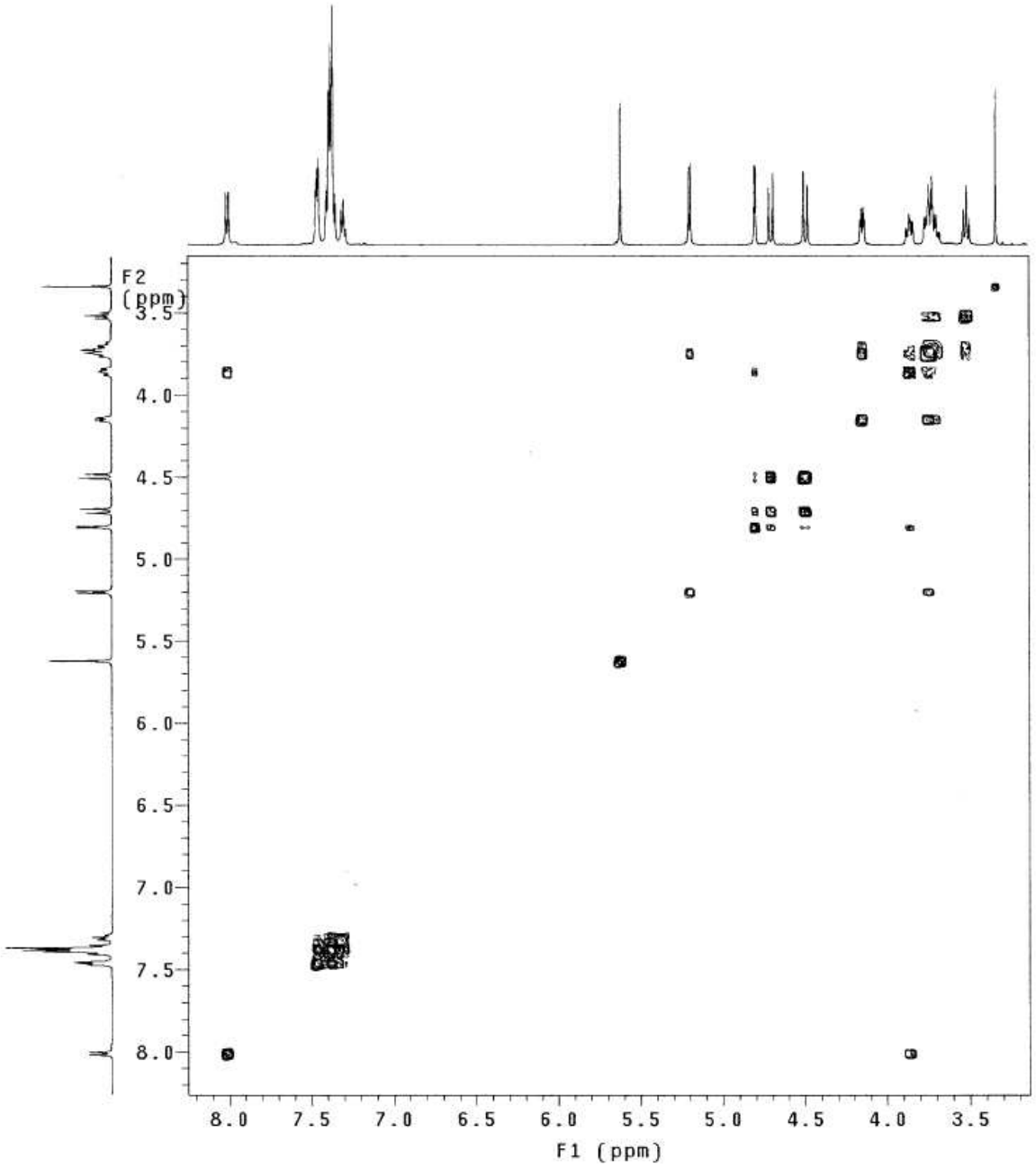

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 256 increments
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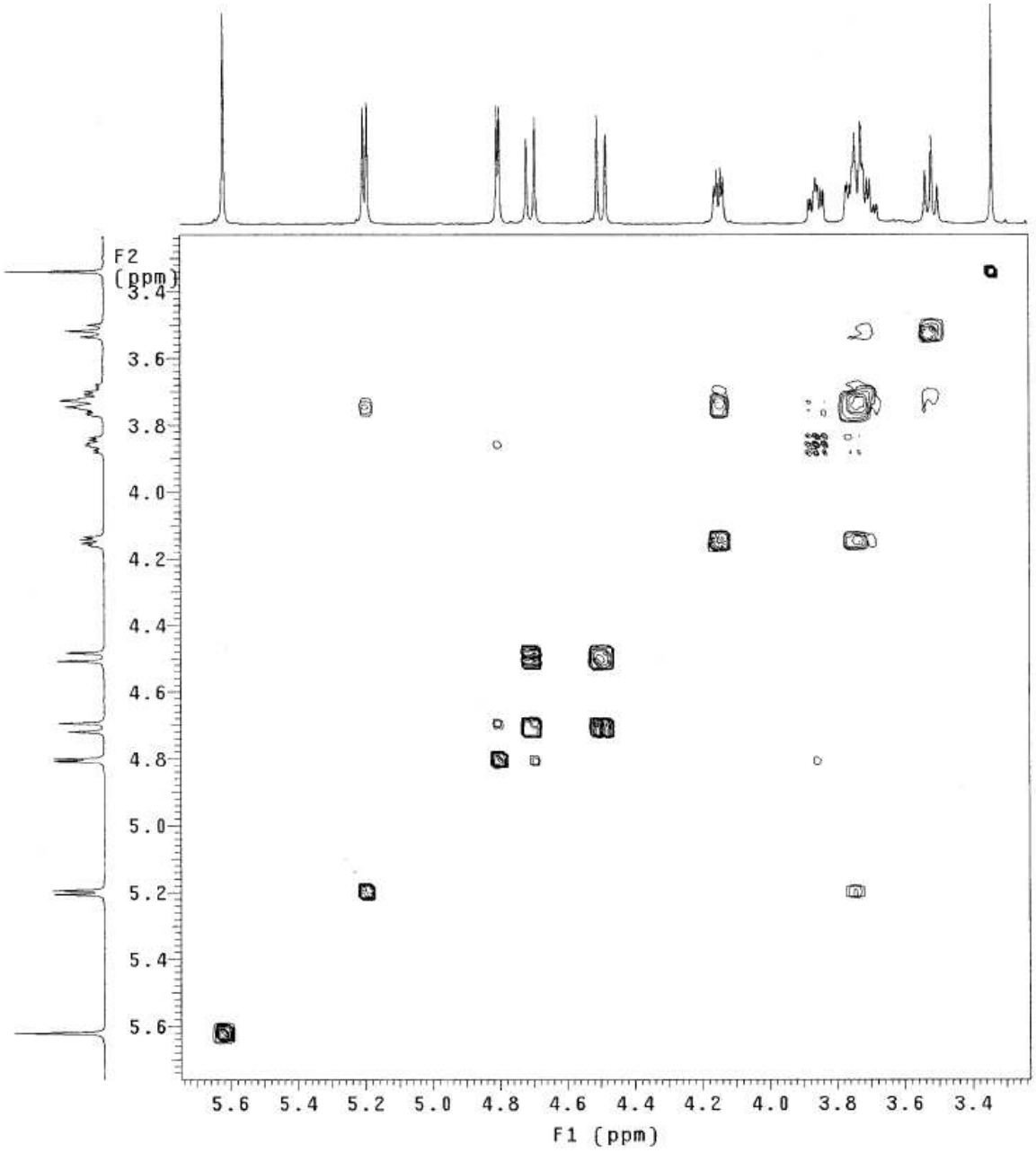

TY2-297

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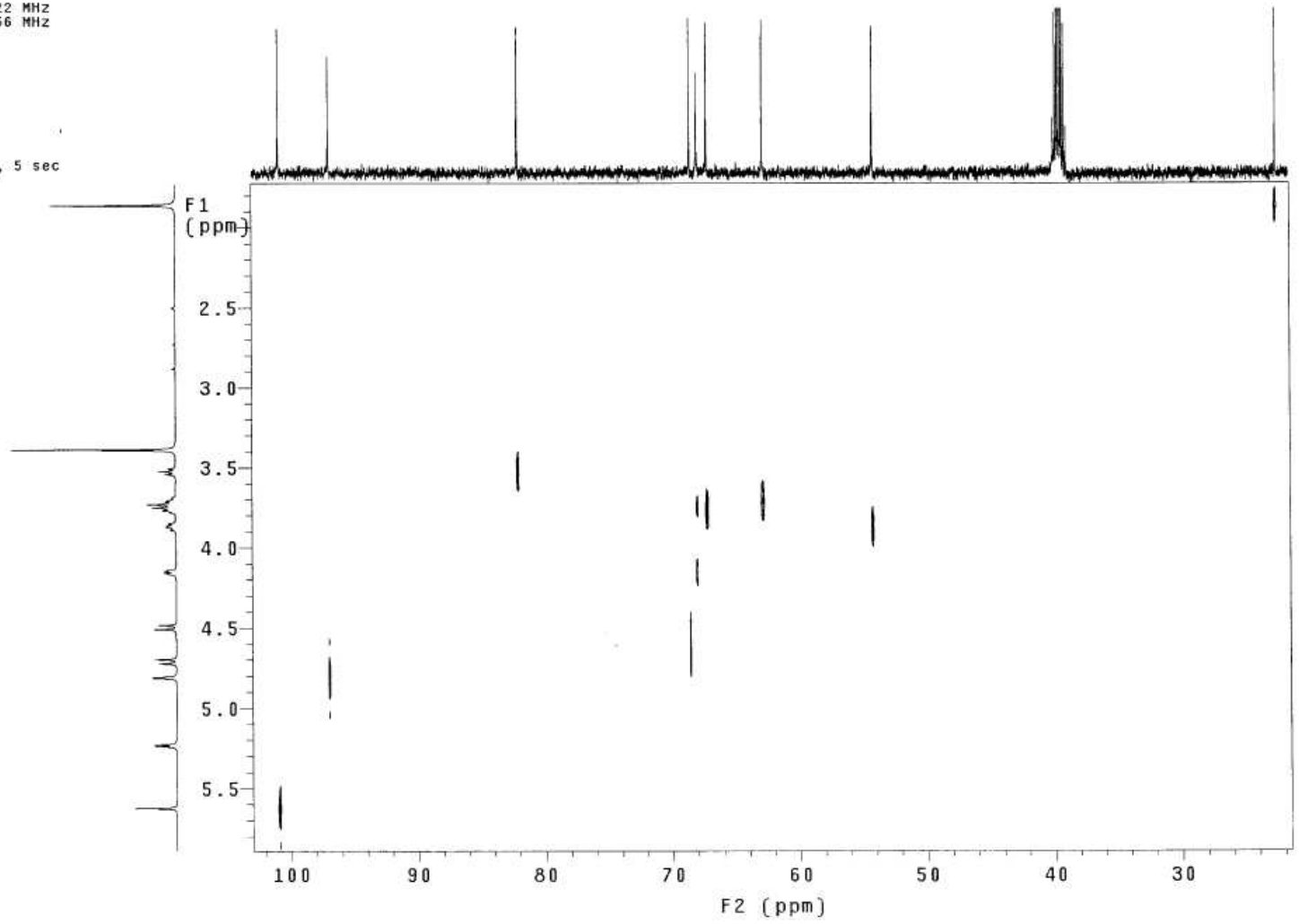

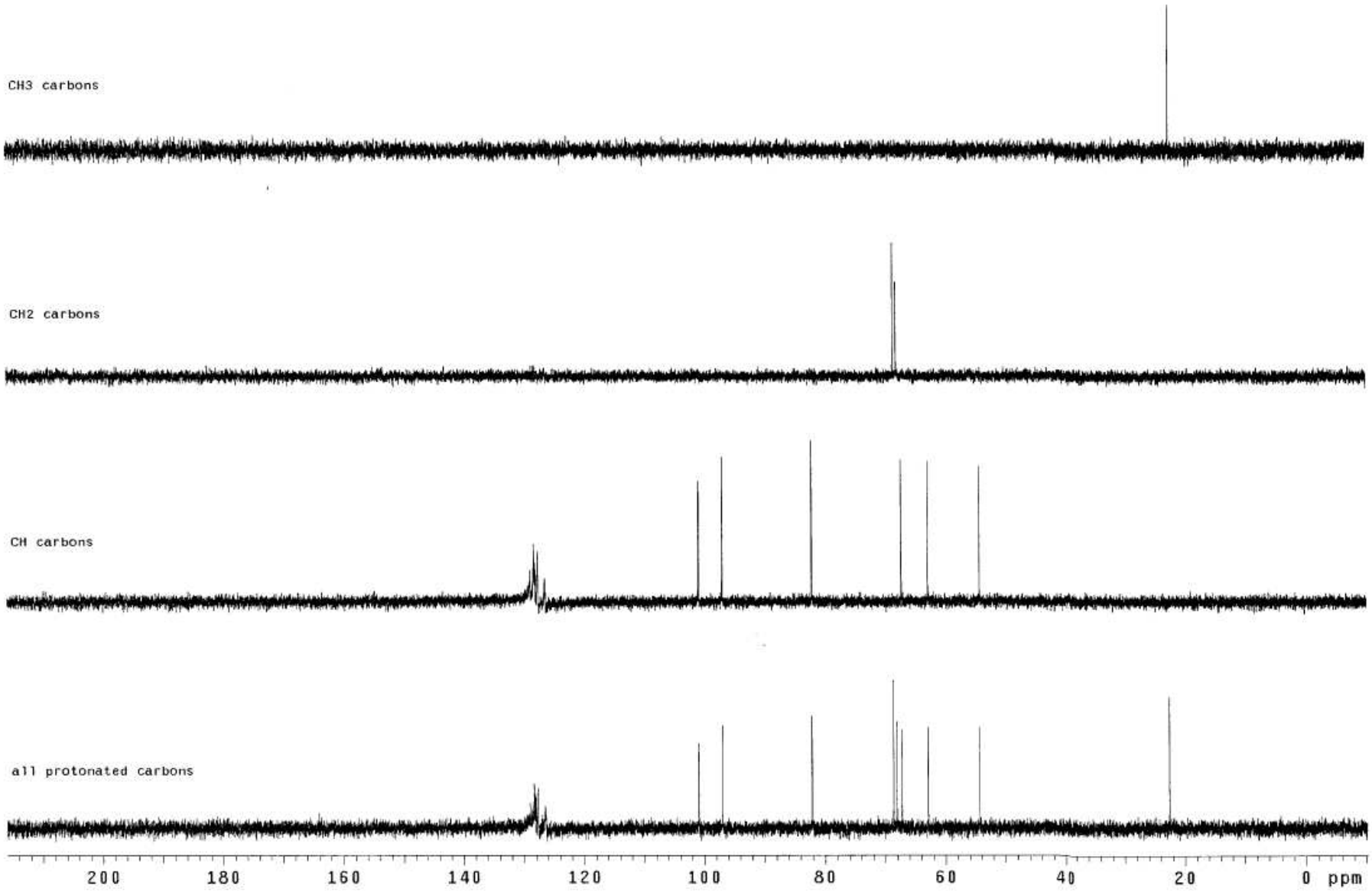
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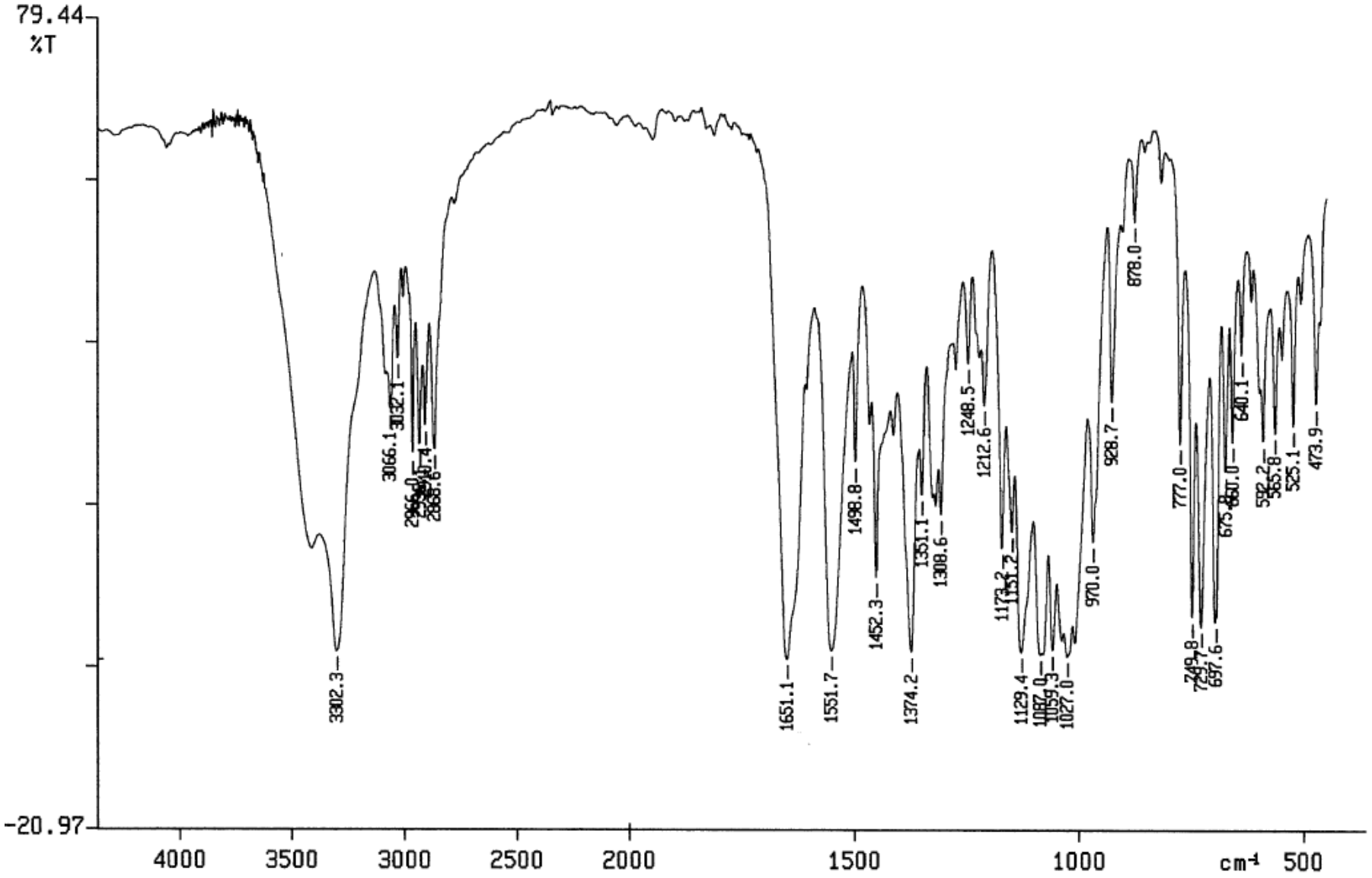
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TY2-318-8

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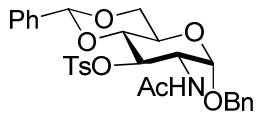
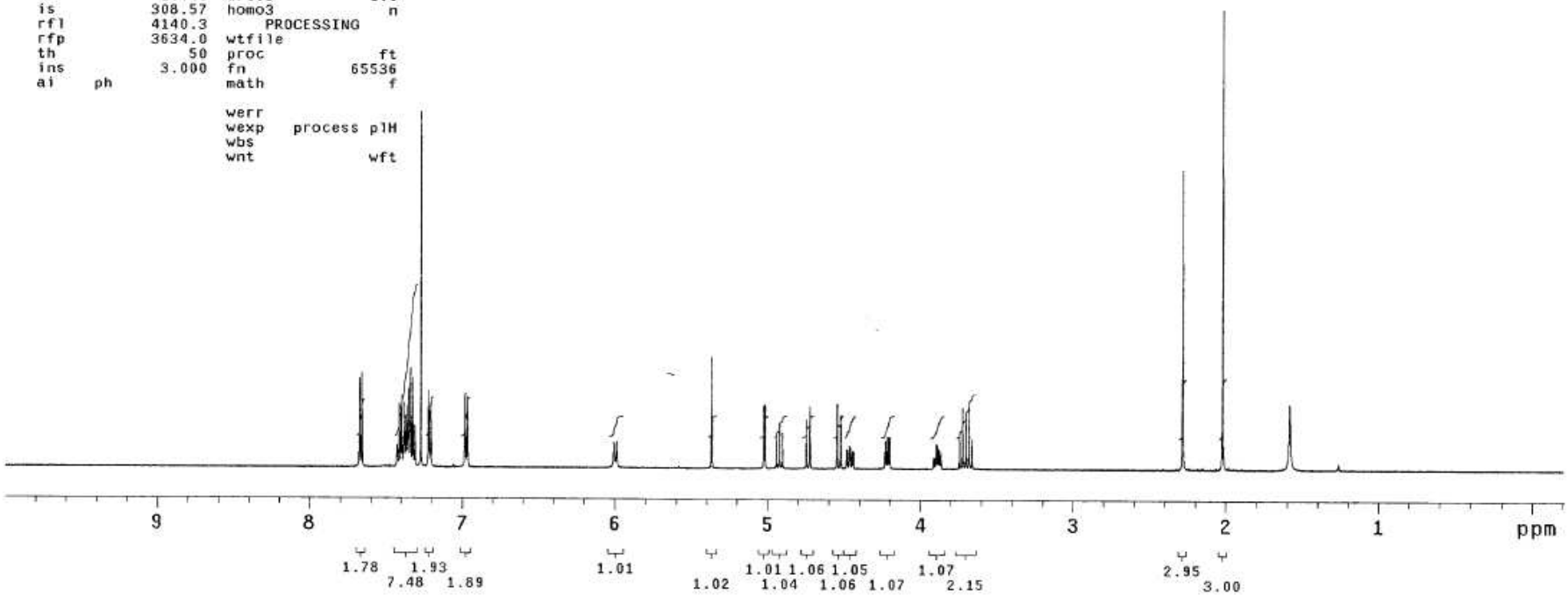
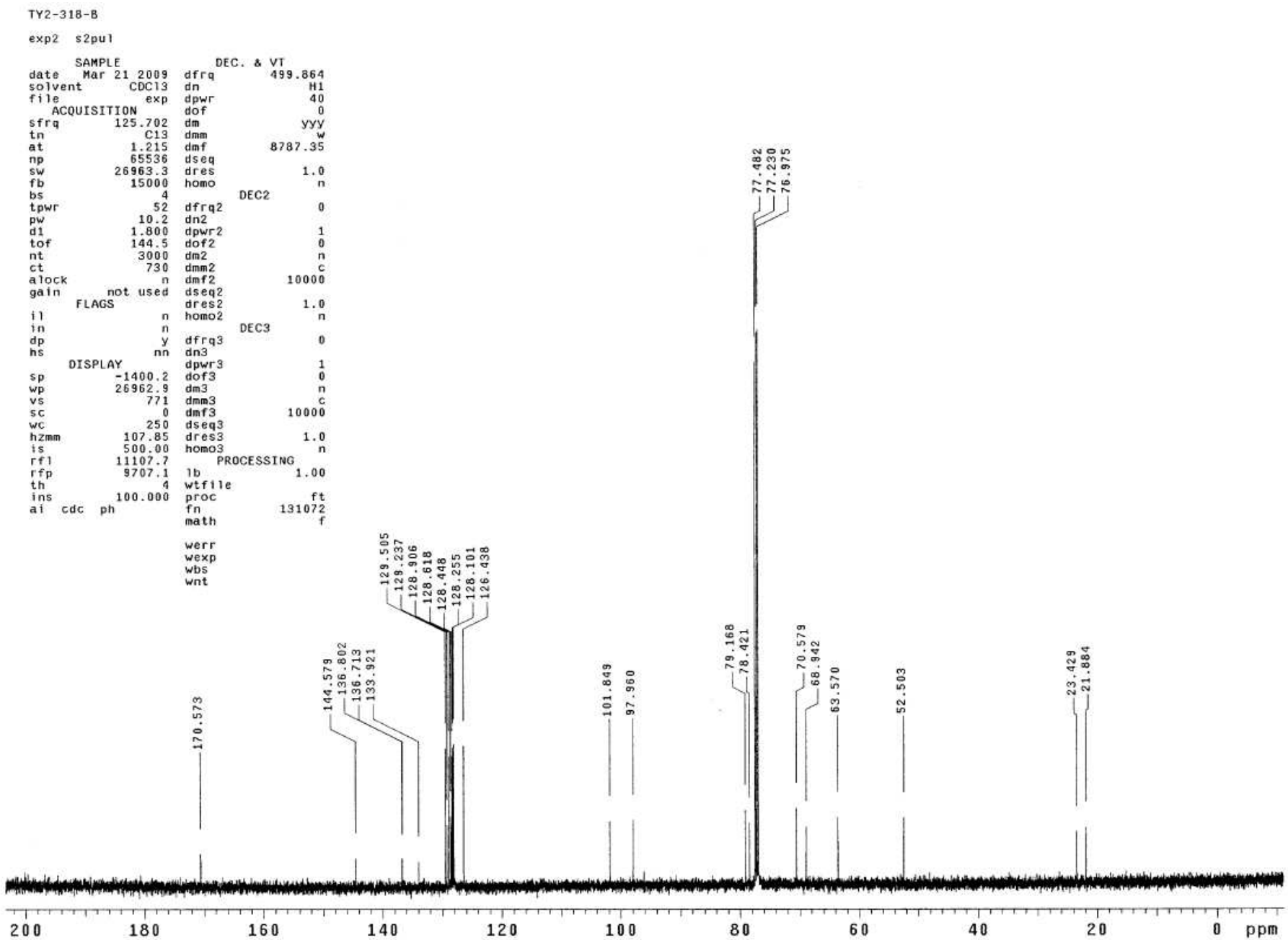


2a

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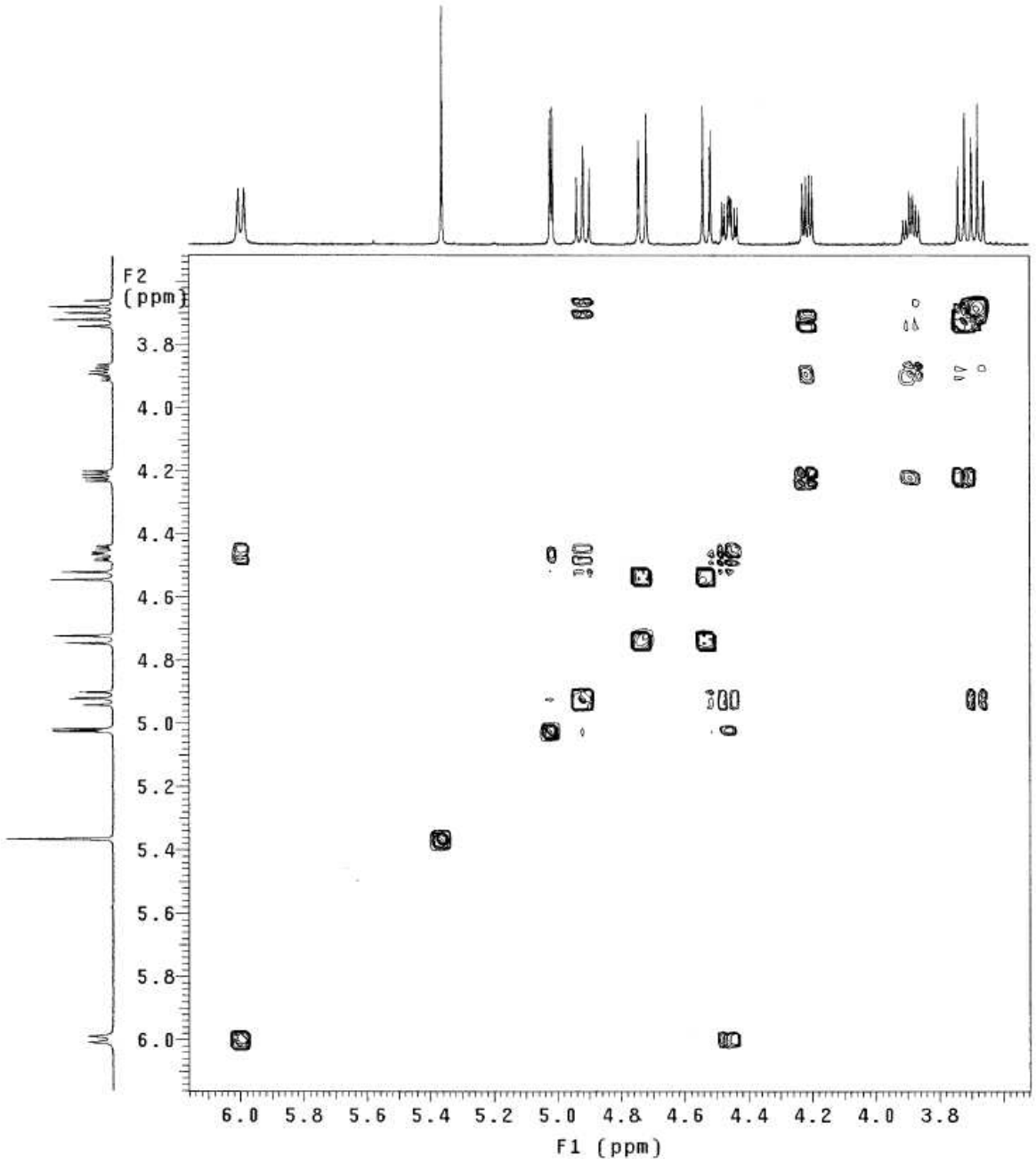
SAMPLE
date Mar 21 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 3000
ct 730
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1400.2
wp 26962.9
vs 771
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11107.7
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
170.573
144.579
136.802
136.713
133.921
129.505
129.237
128.906
128.618
128.448
128.255
128.101
126.438
101.849
97.960
79.168
78.421
77.482
77.230
76.975
70.579
68.942
63.570
52.503
23.429
21.884
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-318-8

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

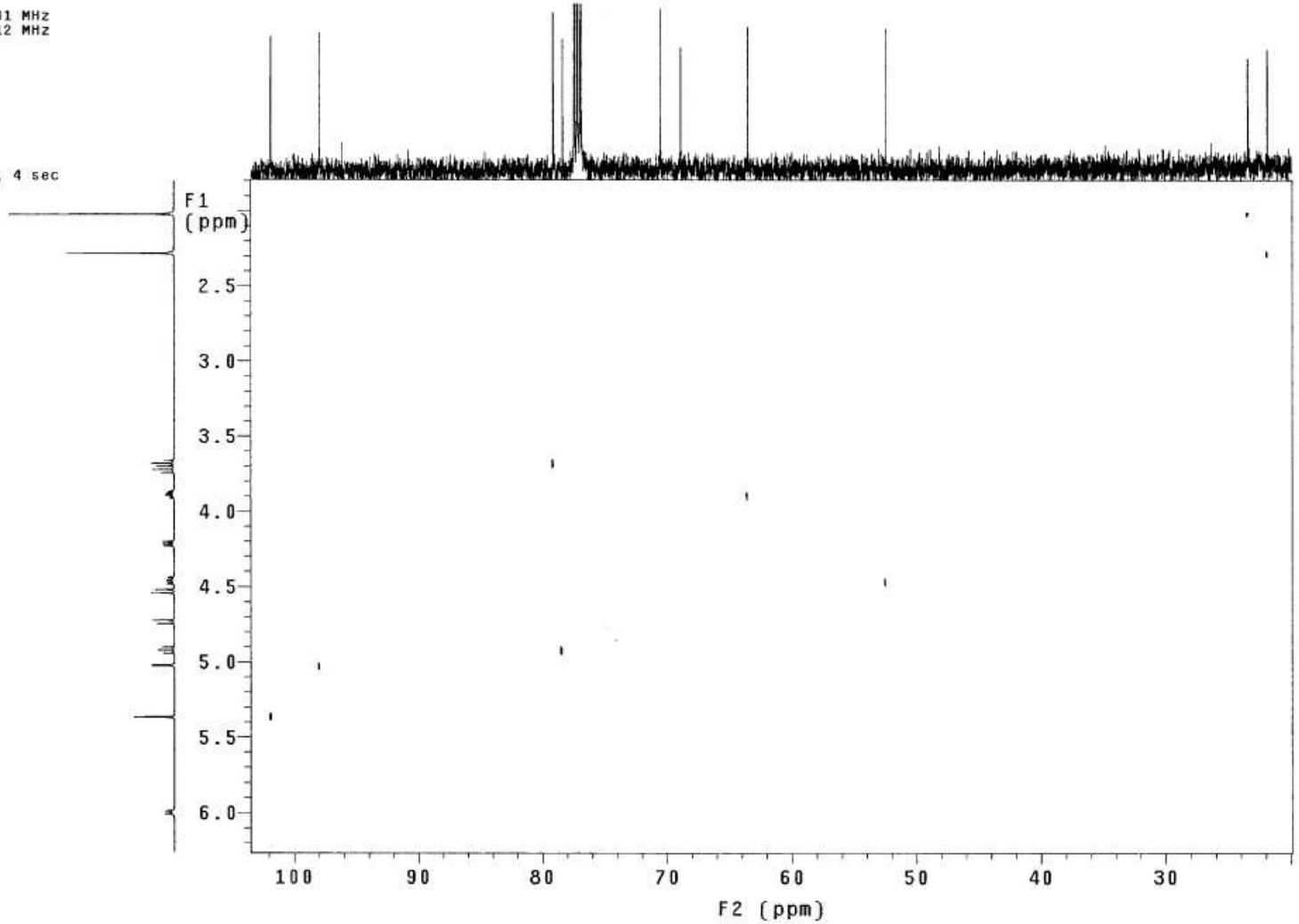


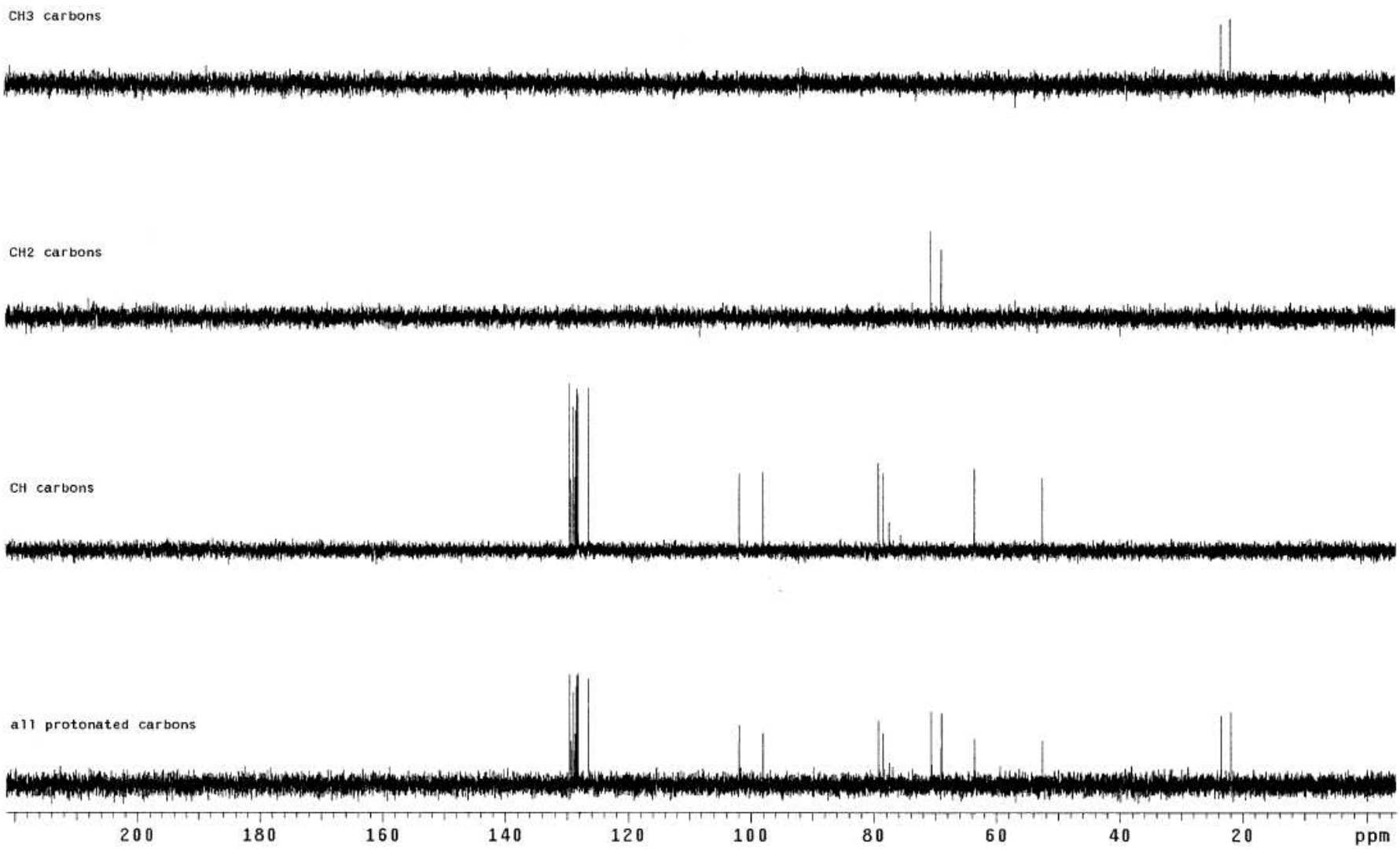
TY2-318-B

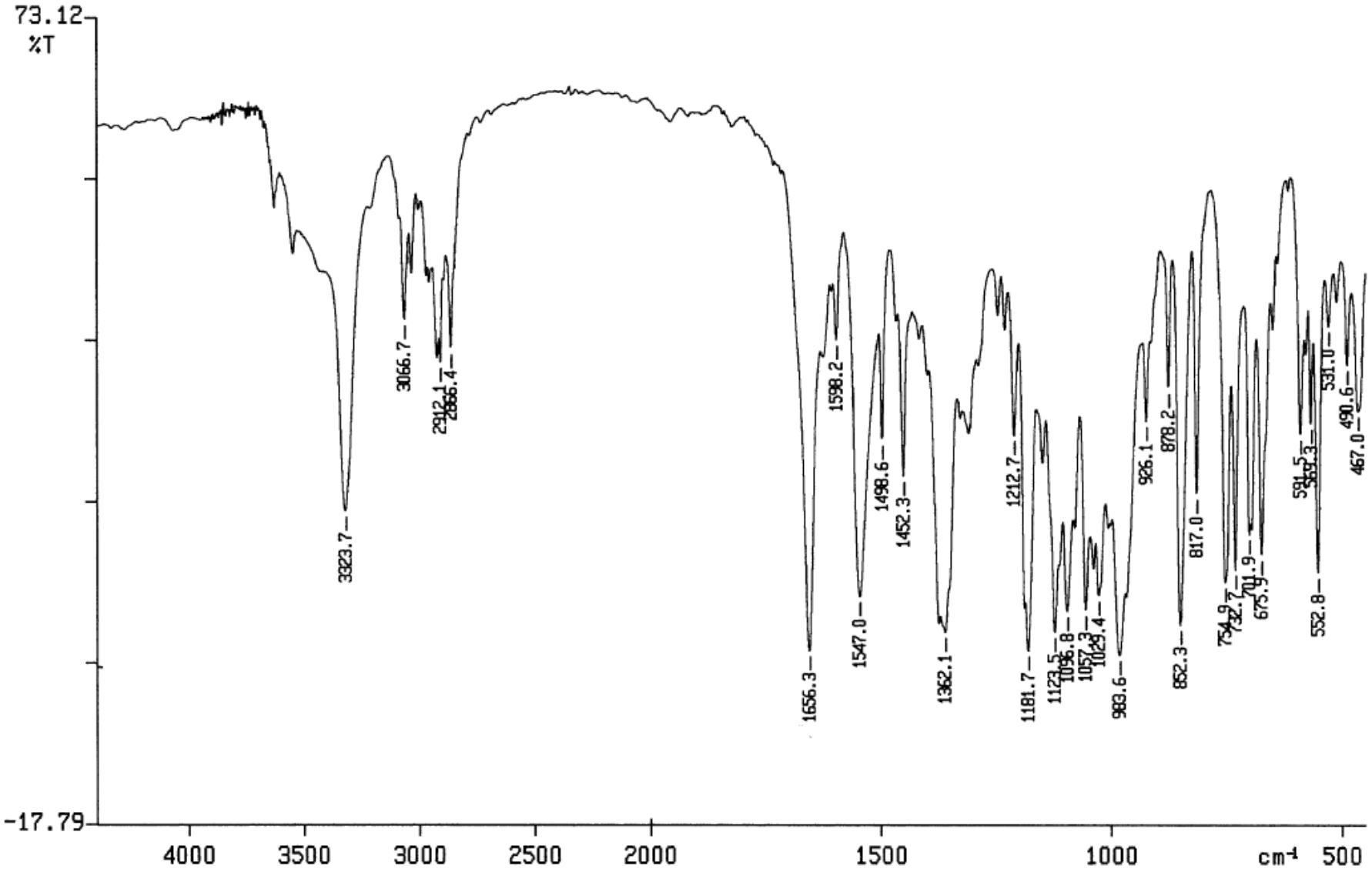
Pulse Sequence: hetcor

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: TY2-318-B-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, 125.6901591 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 7 hr, 32 min, 4 sec



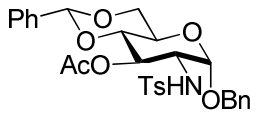

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


73.12
%T
-17.79
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3323.7
3066.7
2912.1
2866.4
1656.3
1598.2
1547.0
1498.6
1452.3
1362.1
1212.7
1181.7
1123.5
1096.8
1057.3
1029.4
983.6
926.1
878.2
852.3
817.0
754.9
732.7
711.9
675.9
591.5
569.3
552.8
531.0
490.6
467.0

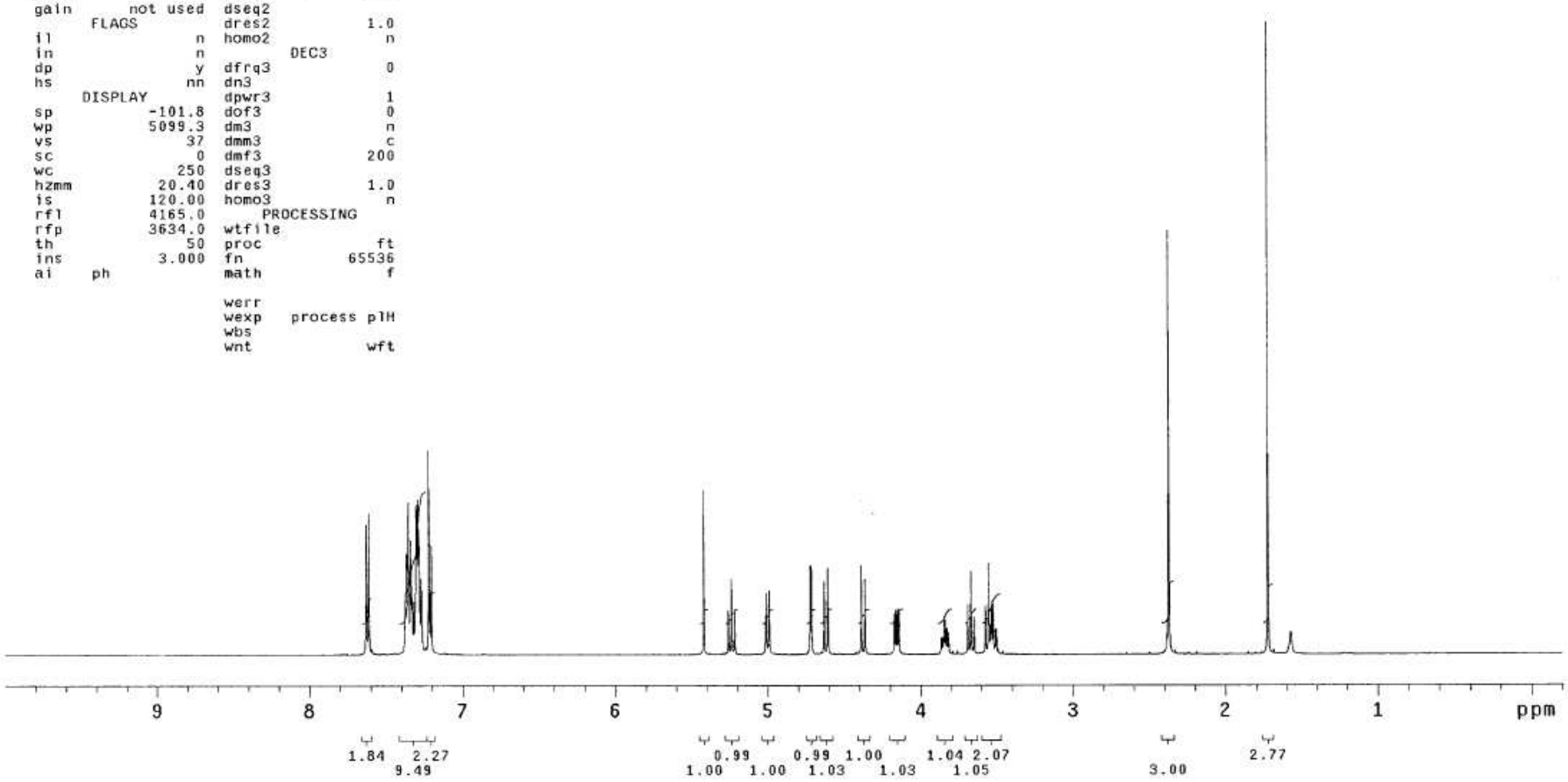
TY2-318-A

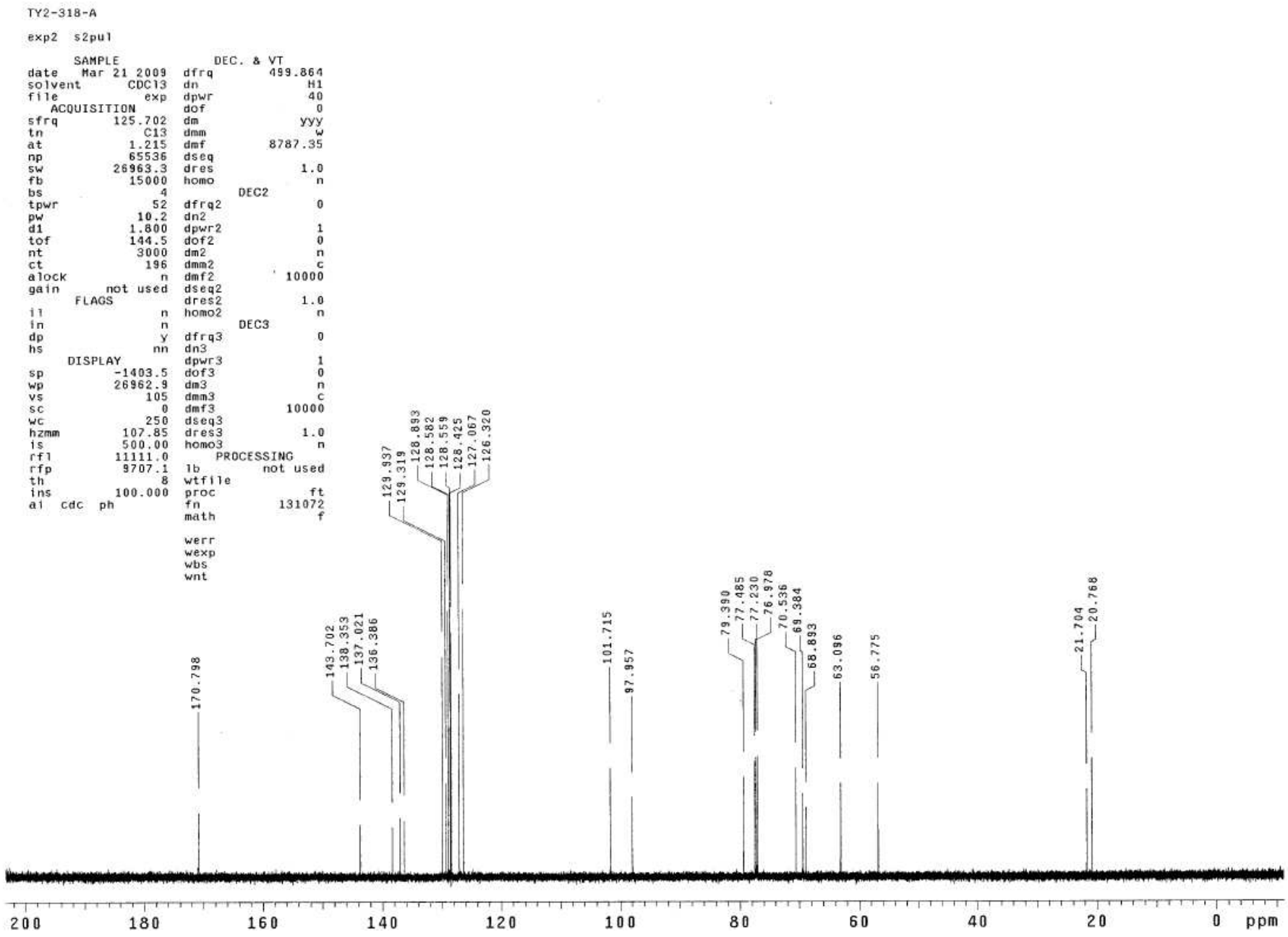
exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 21 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	-101.8	dof3	0
wp	5099.3	dm3	n
vs	37	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	120.00	homo3	n
rfl	4165.0	PROCESSING	
rfp	3634.0	wtfile	
th	50	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



3a

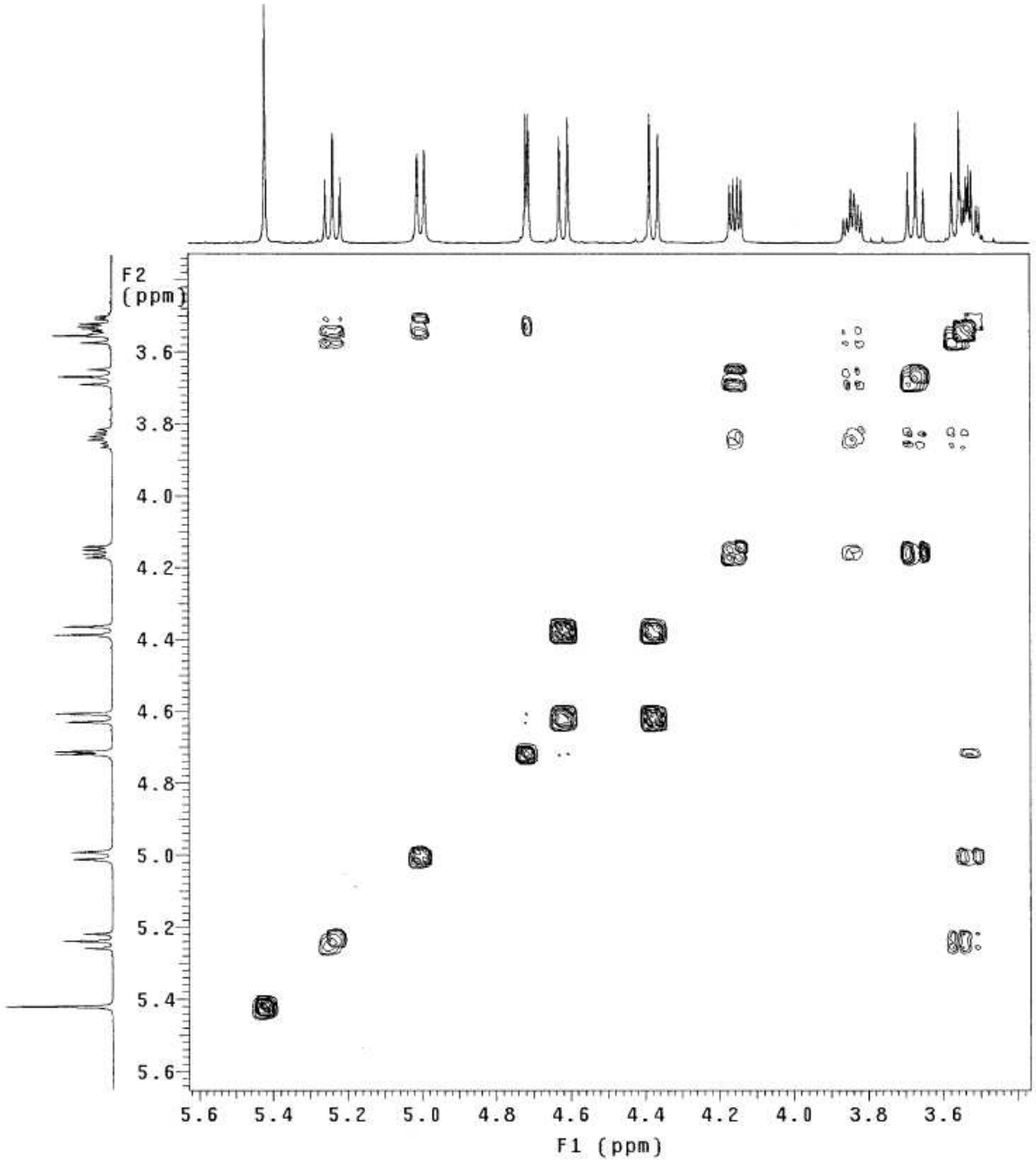



TY2-318-A
exp2 s2pul
SAMPLE
date Mar 21 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 3000
ct 196
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1403.5
wp 26962.9
vs 105
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11111.0
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
170.798
143.702
138.353
137.021
136.386
129.937
129.319
128.893
128.582
128.559
128.425
127.067
126.320
101.715
97.957
79.390
77.485
77.230
76.978
70.536
69.384
68.893
63.096
56.775
21.704
20.768
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-318-A

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, 499.8611956 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

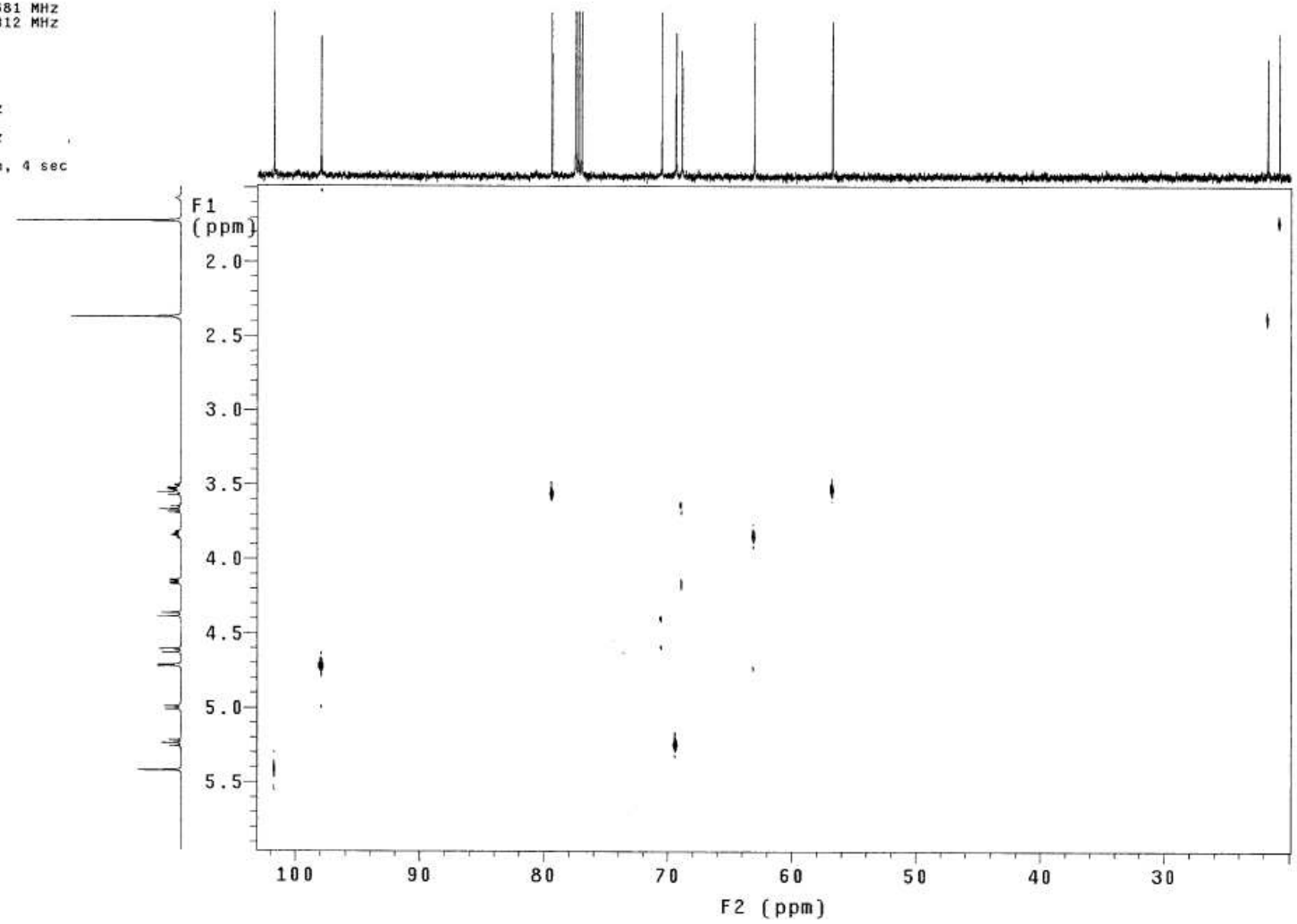


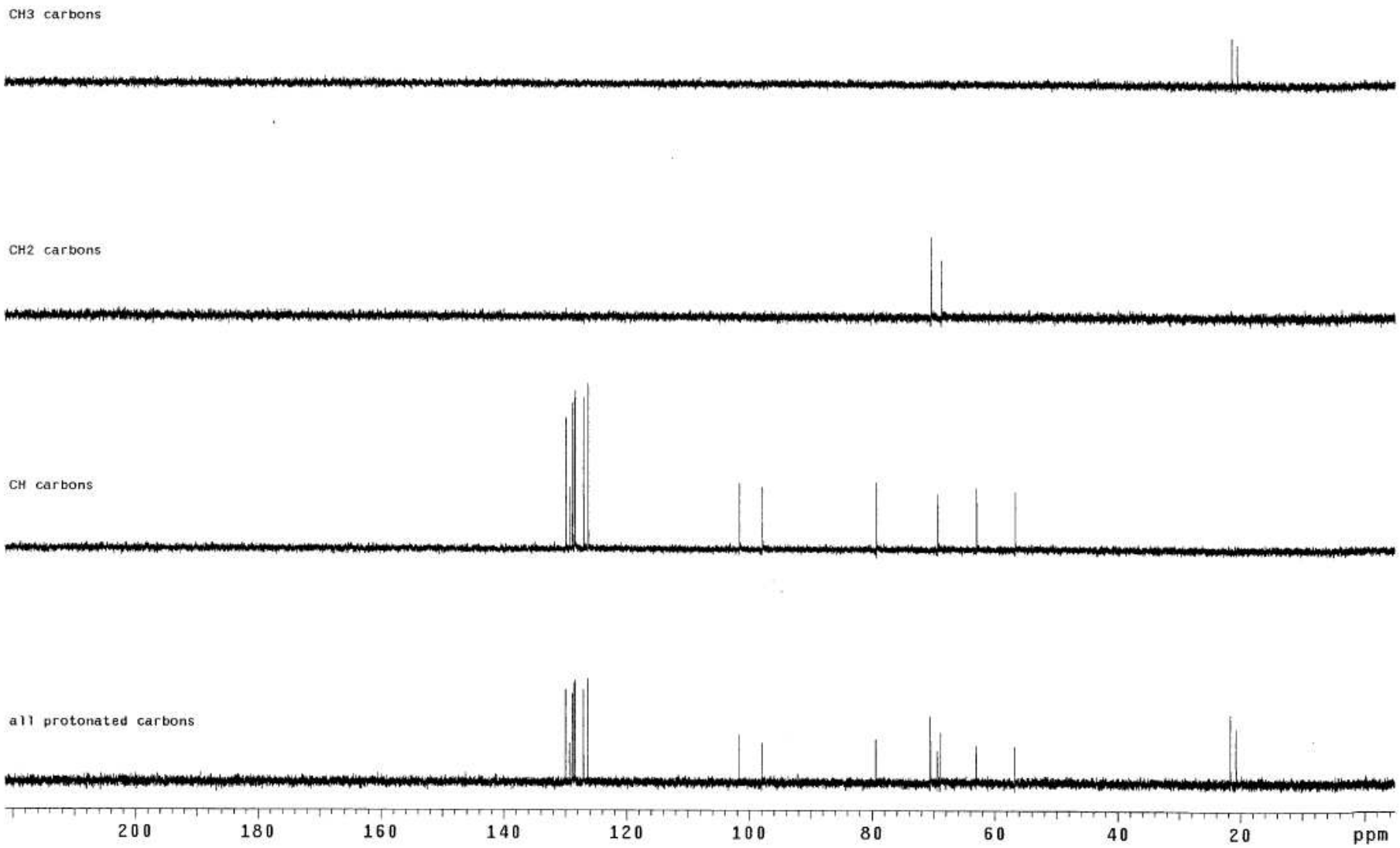
TY2-318-A

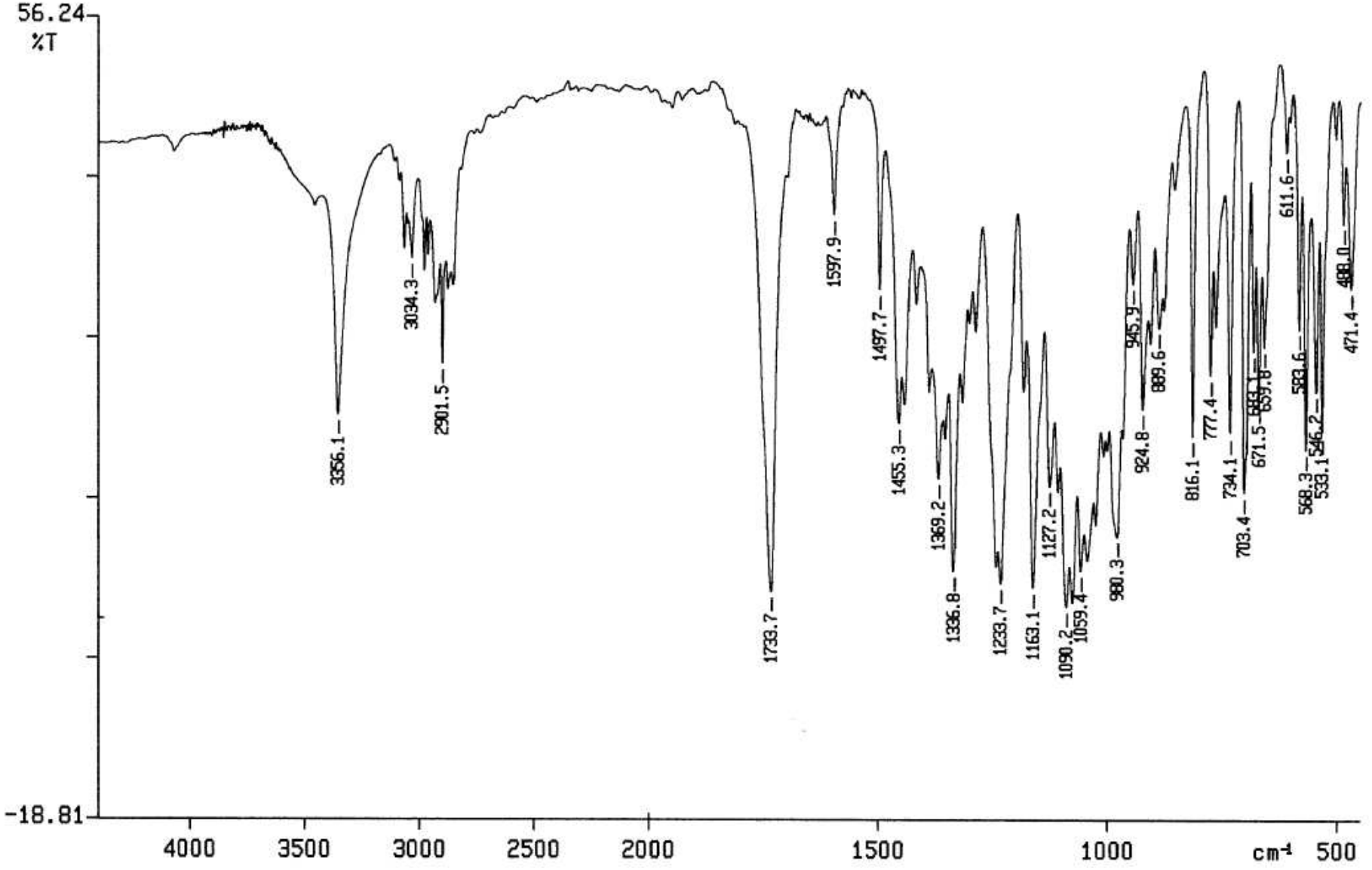
Pulse Sequence: hetcor

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, 125.6901681 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 7 hr, 32 min, 4 sec



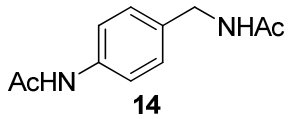

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


56.24
%T
-18.81
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3356.1
3034.3
2901.5
1733.7
1597.9
1497.7
1455.3
1369.2
1336.8
1233.7
1163.1
1127.2
1090.2
1059.4
980.3
945.9
924.8
889.6
816.1
777.4
734.1
703.4
671.5
659.8
611.6
583.6
568.3
546.2
533.1
471.4

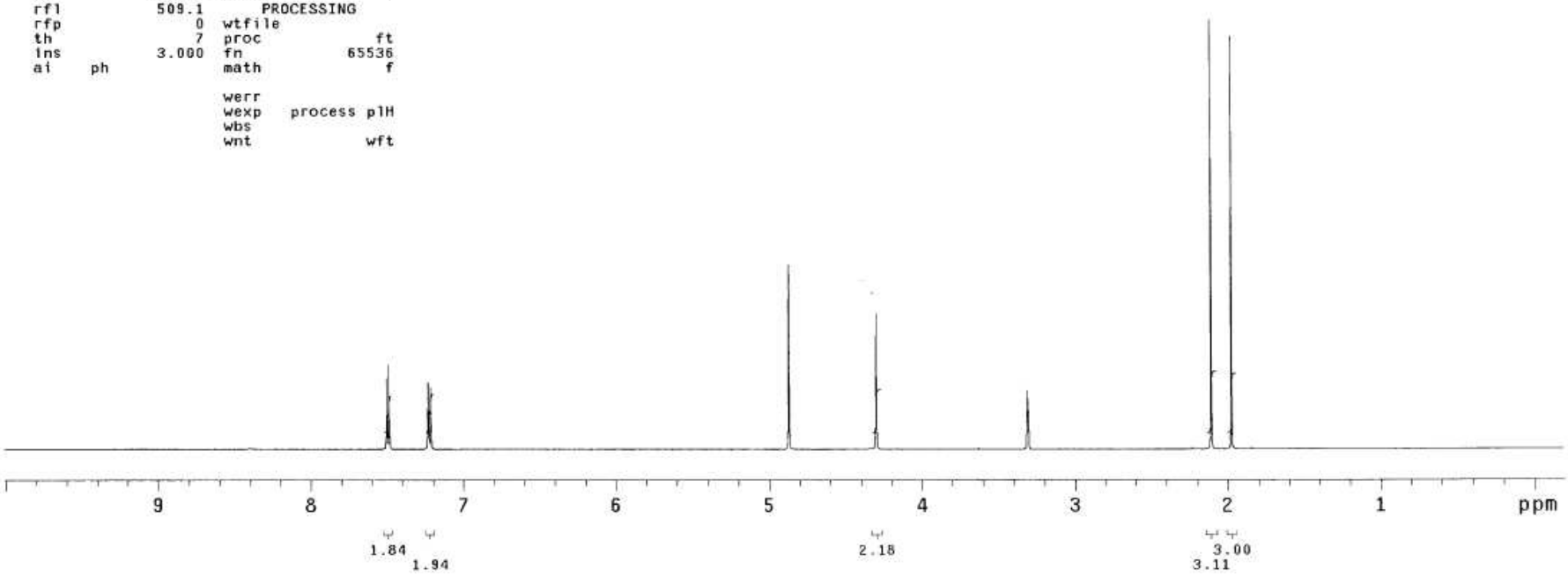
TY2-380

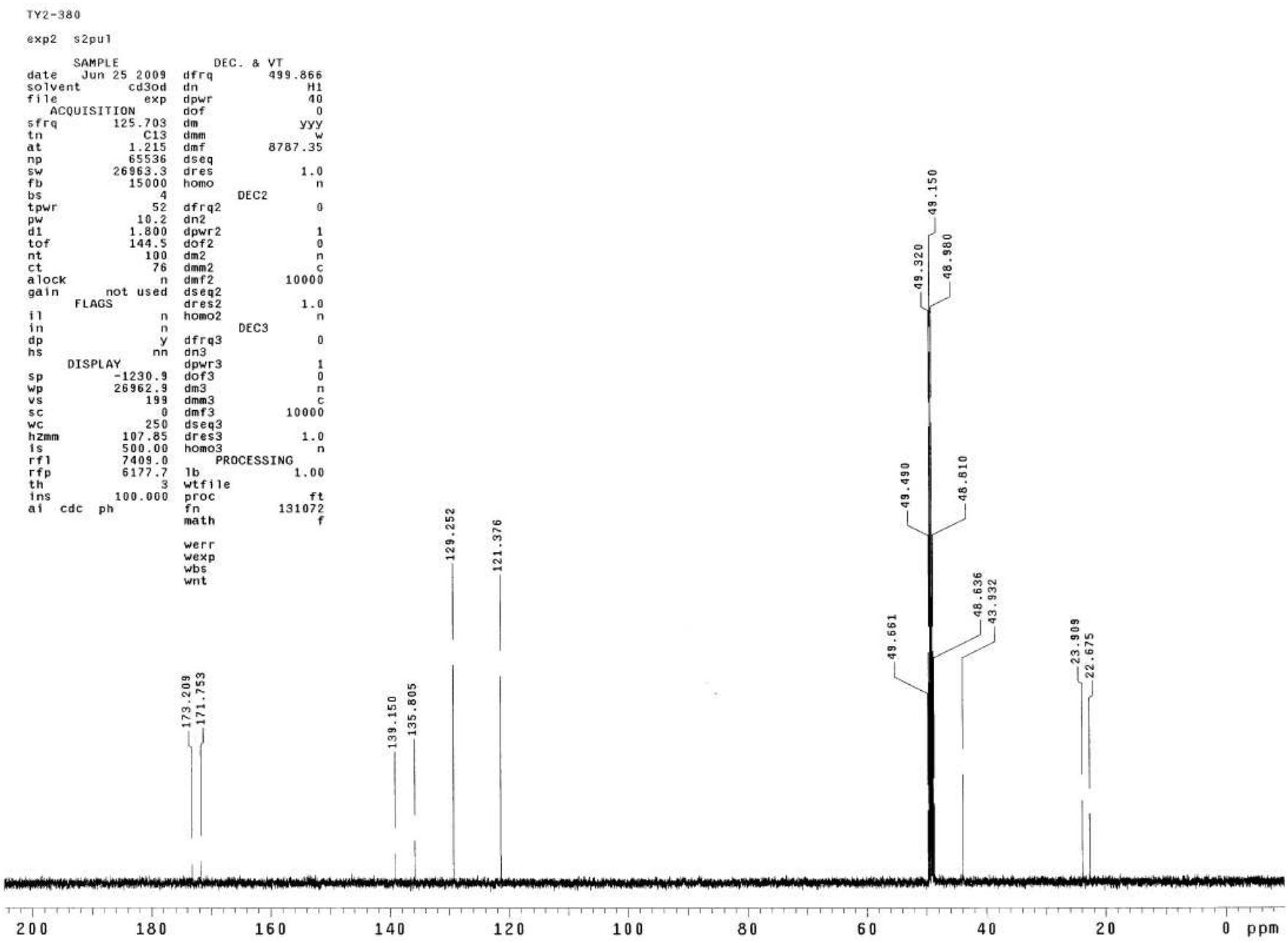
exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 25 2009	dfrq	499.866
solvent	CD3OD	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	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	-97.0	dof3	0
wp	5099.3	dm3	n
vs	15	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	148.04	homo3	n
rfl	509.1	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
lns	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



14

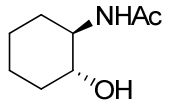



TY2-380
exp2 s2pul
SAMPLE
date Jun 25 2009
solvent cd3od
file exp
ACQUISITION
sfrq 125.703
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 100
ct 76
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1230.9
wp 26962.9
vs 199
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 7409.0
rfp 6177.7
th 3
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
173.209
171.753
139.150
135.805
129.252
121.376
49.661
49.490
49.320
49.150
48.980
48.810
48.636
43.932
23.909
22.675
200
180
160
140
120
100
80
60
40
20
0 ppm

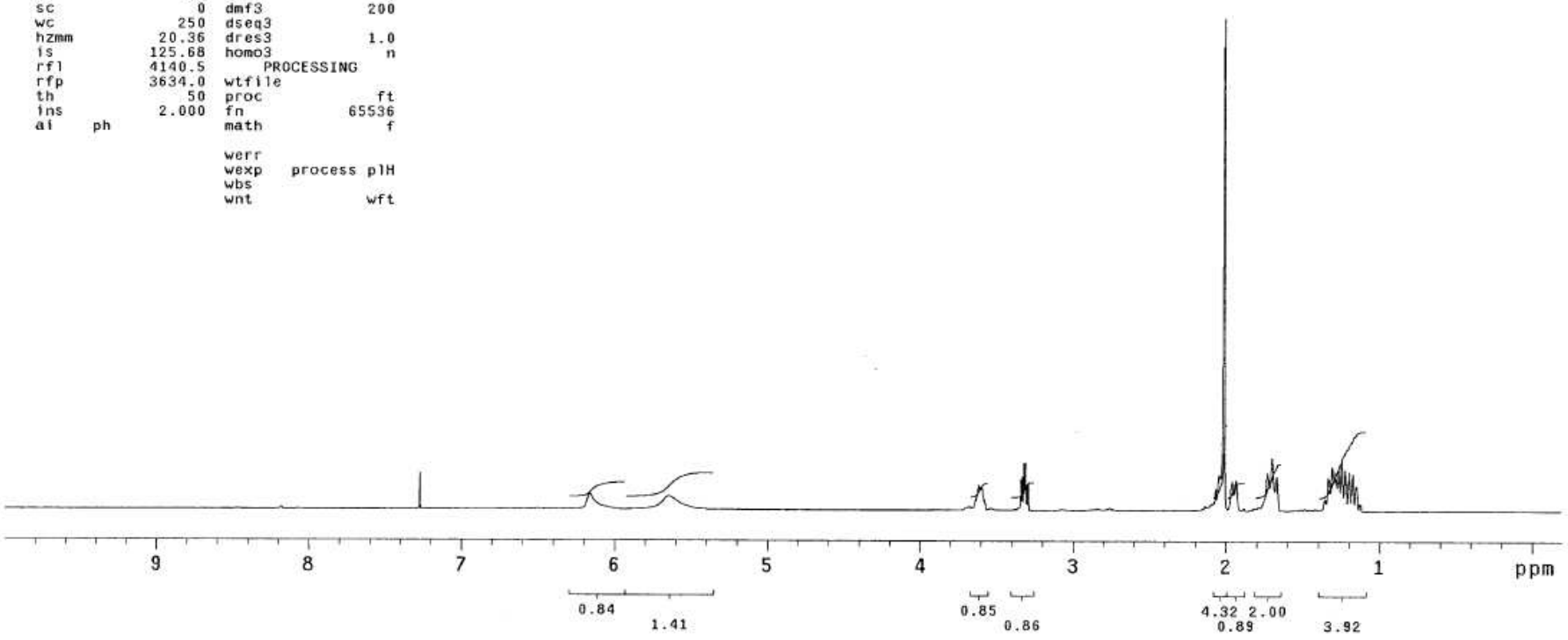
TY2-334

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	-94.4	dof3	0
wp	5090.8	dm3	n
vs	52	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.36	dres3	1.0
is	125.68	homo3	n
rfl	4140.5	PROCESSING	
rfp	3634.0	wtfile	
th	50	proc	ft
ins	2.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



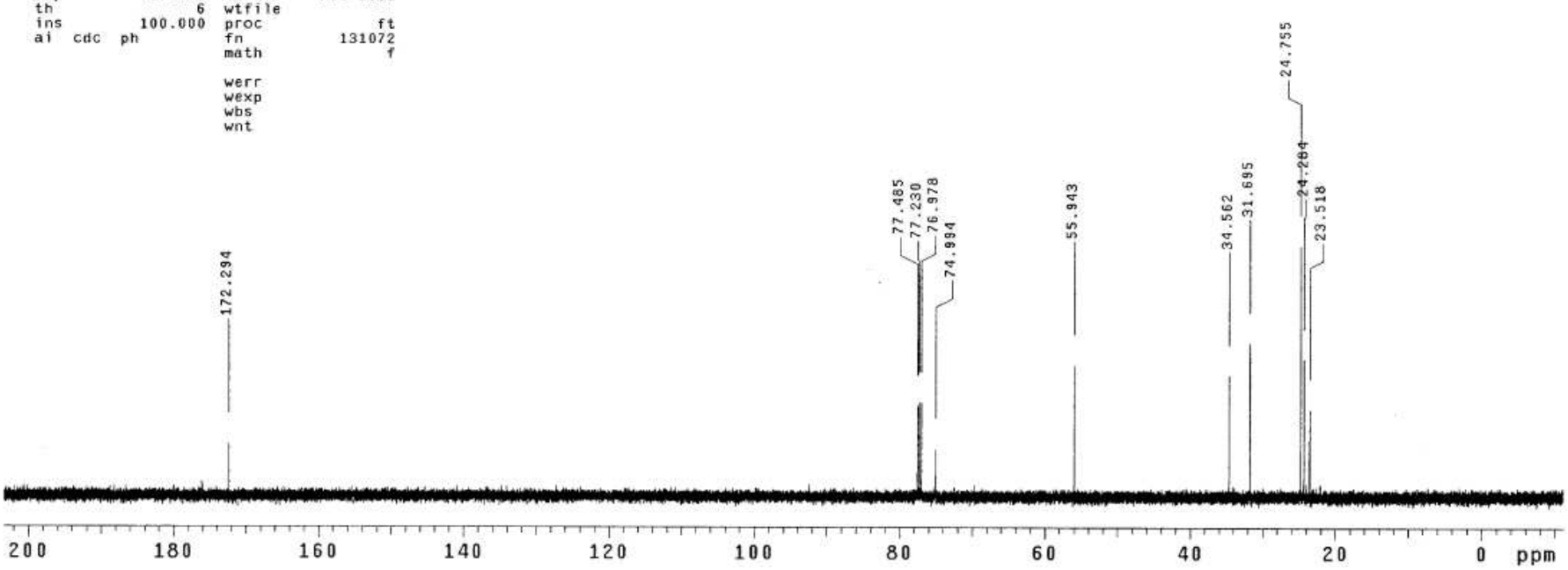
1b



TY2-334

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	84	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	89	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	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

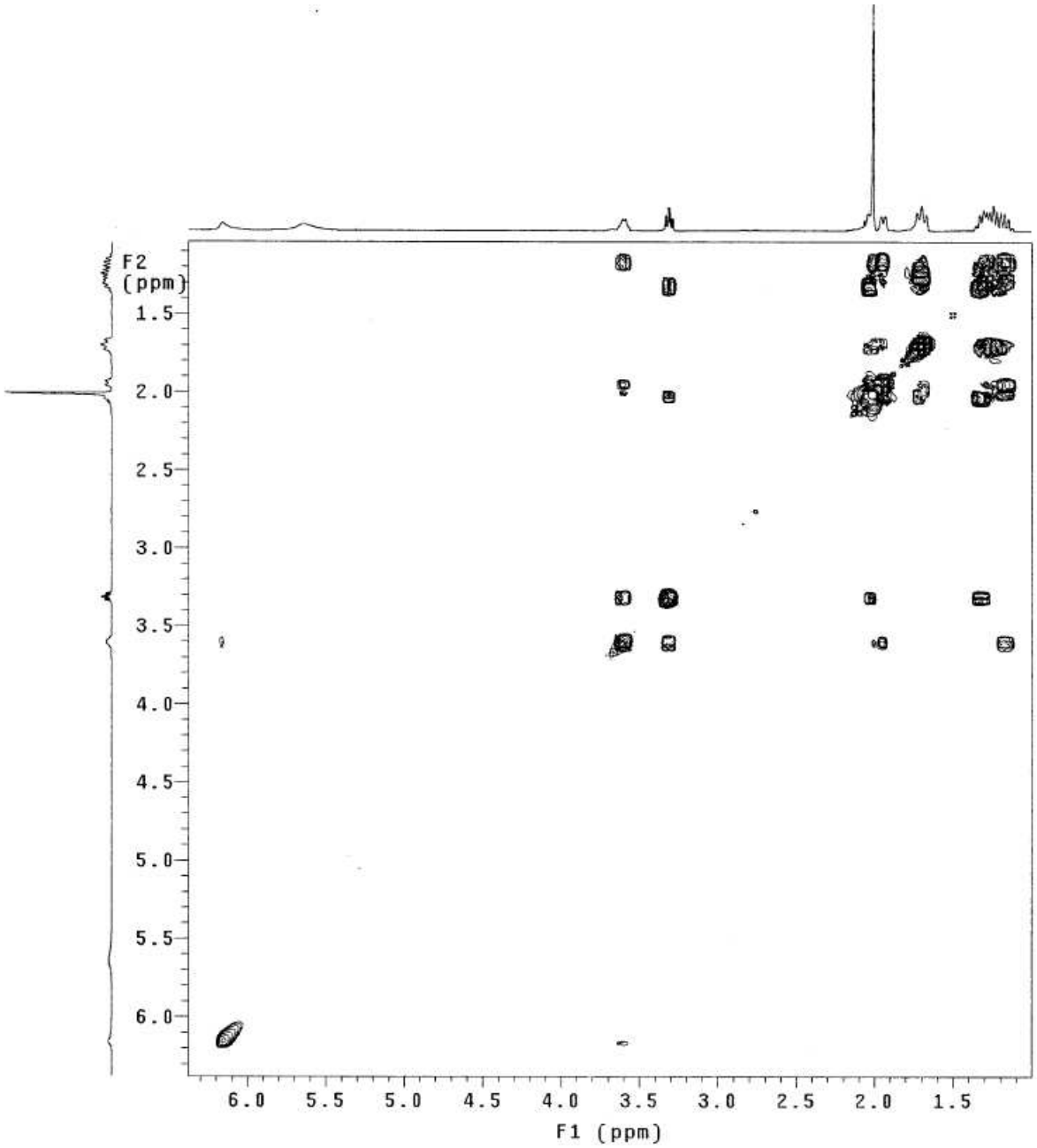


TY2-334

Pulse Sequence: relayh

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

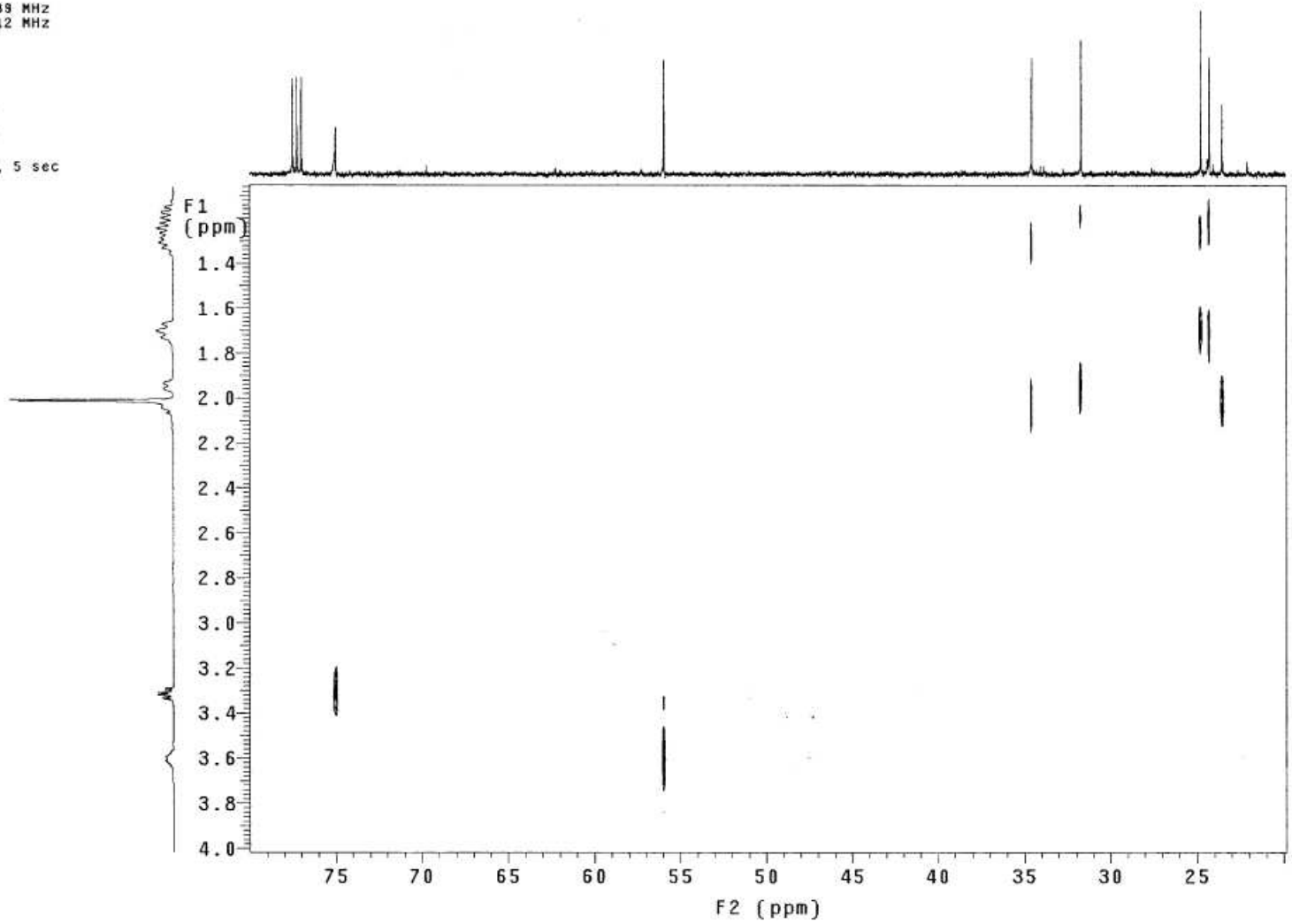
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611711 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

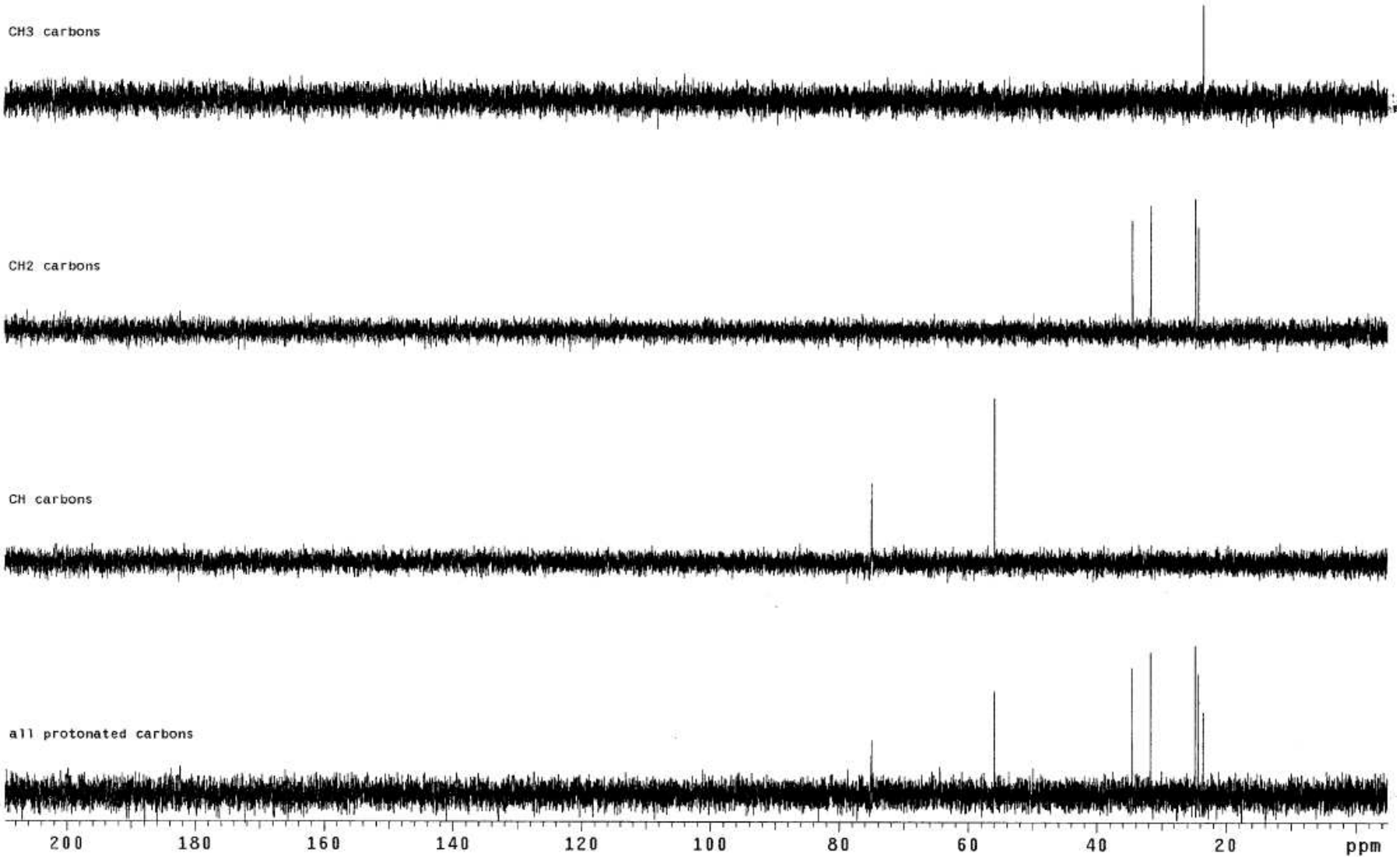


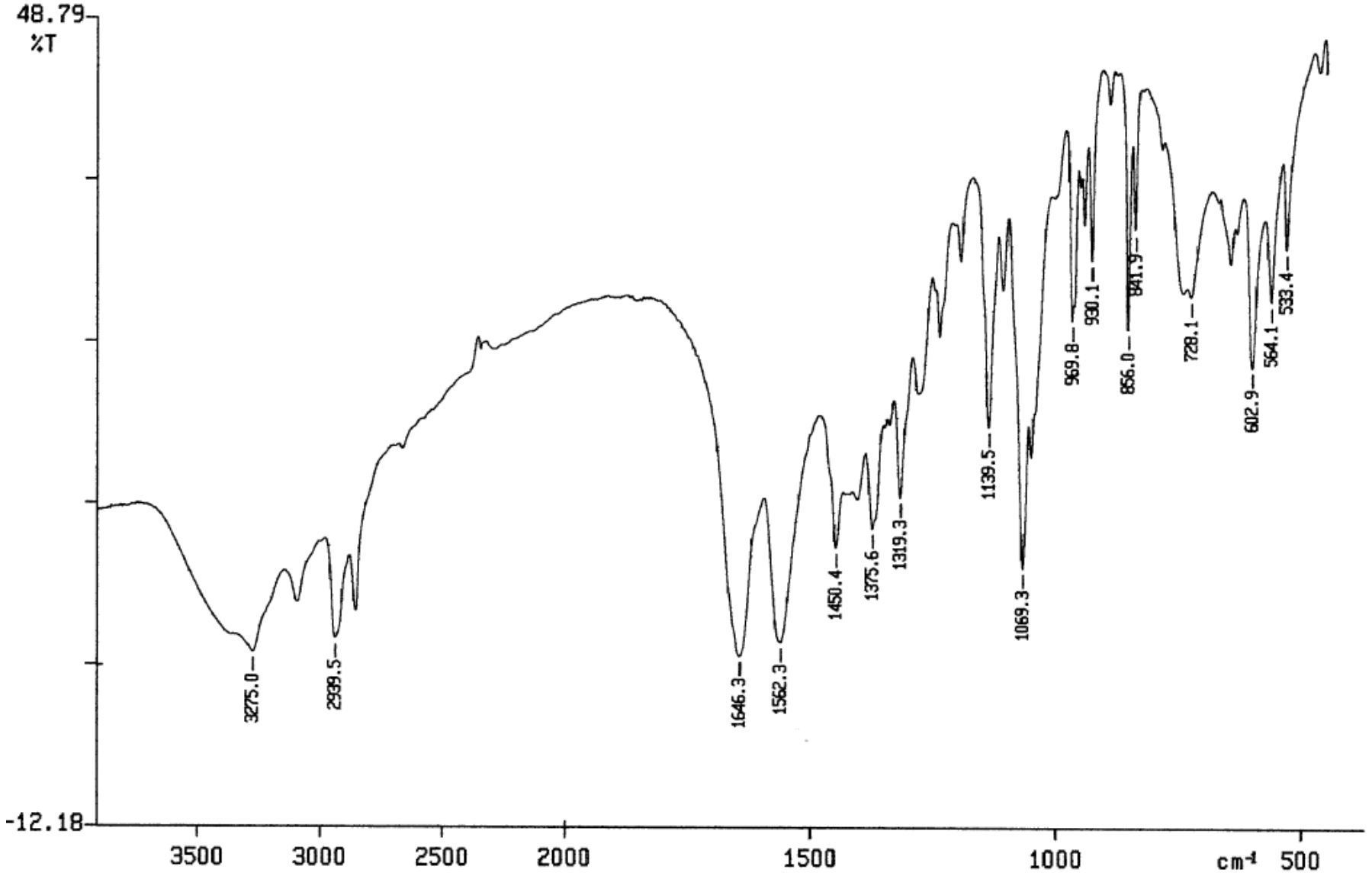
TY2-334

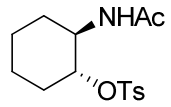
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

 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, 125.6901689 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 3 hr, 46 min, 5 sec



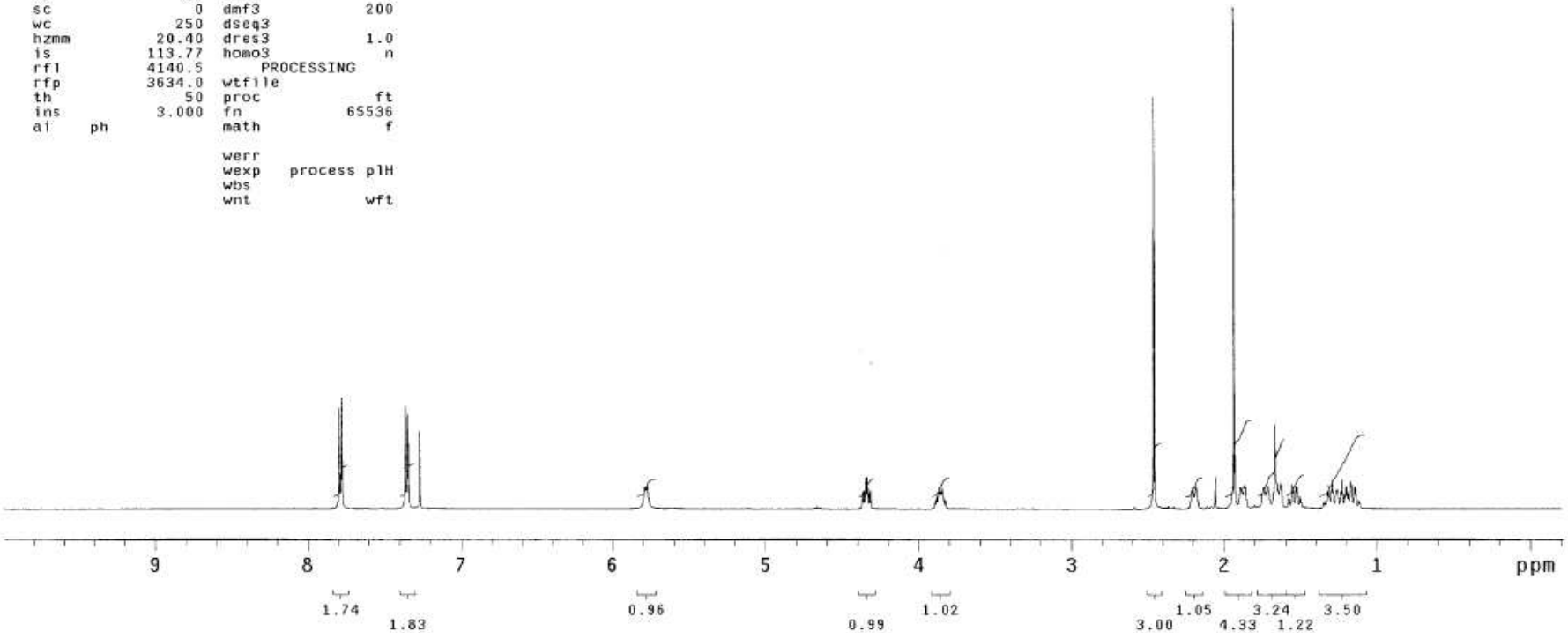

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


48.79
%T
-12.18
3500
3000
2500
2000
1500
1000
cm-1
500
3275.0
2939.5
1646.3
1562.3
1450.4
1375.6
1319.3
1139.5
1069.3
969.8
930.1
856.0
841.9
728.1
602.9
564.1
533.4



2b

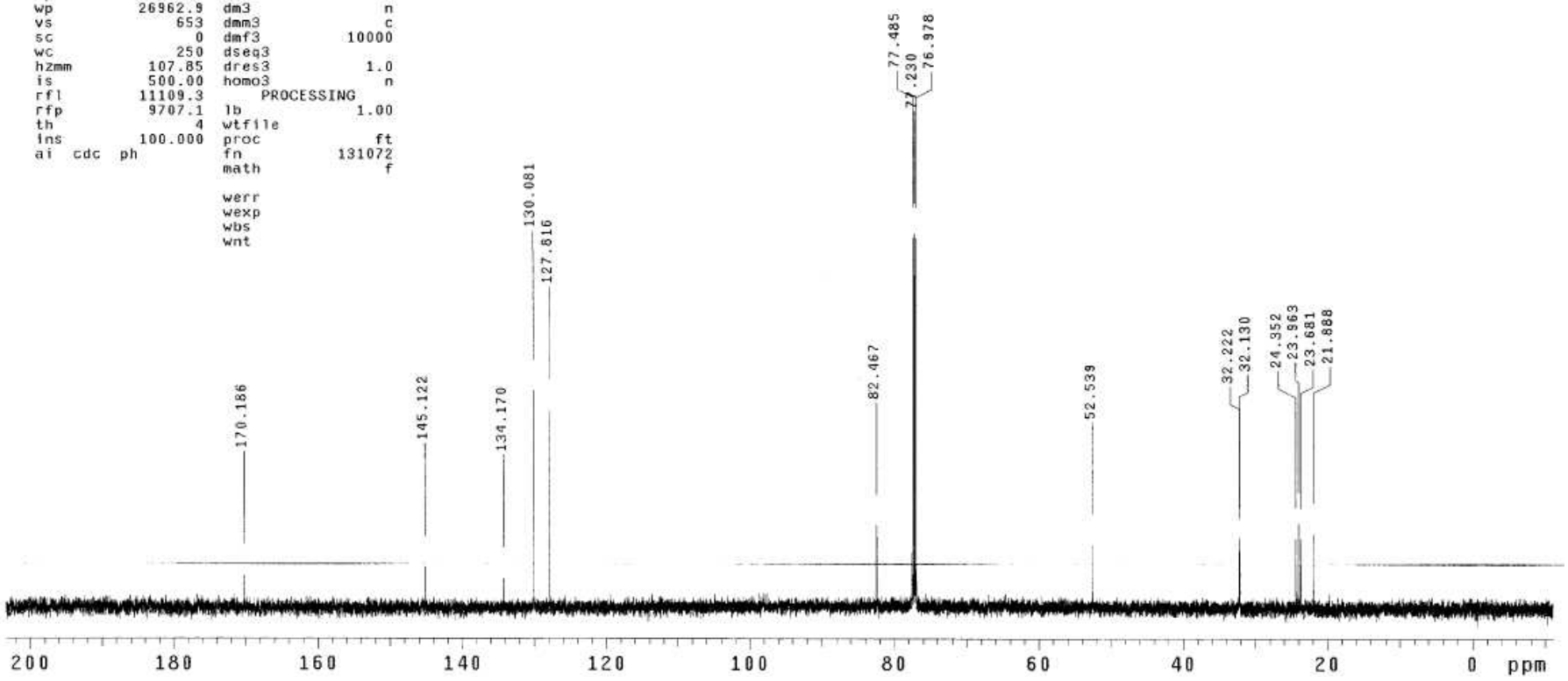
```
TY2-360-C-crystal

exp1  s2pul

      SAMPLE
date    Jun  3 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          -103.0
wp          5099.3
vs              28
sc               0
wc             250
hzmm         20.40
is           113.77
rfl          4140.5
rfp          3634.0
th              50
ins          3.000
al    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 p1H
wbs
wnt             wft
```



TY2-360-C-crystal

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 3 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	278	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	653	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	1.00
th	4	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

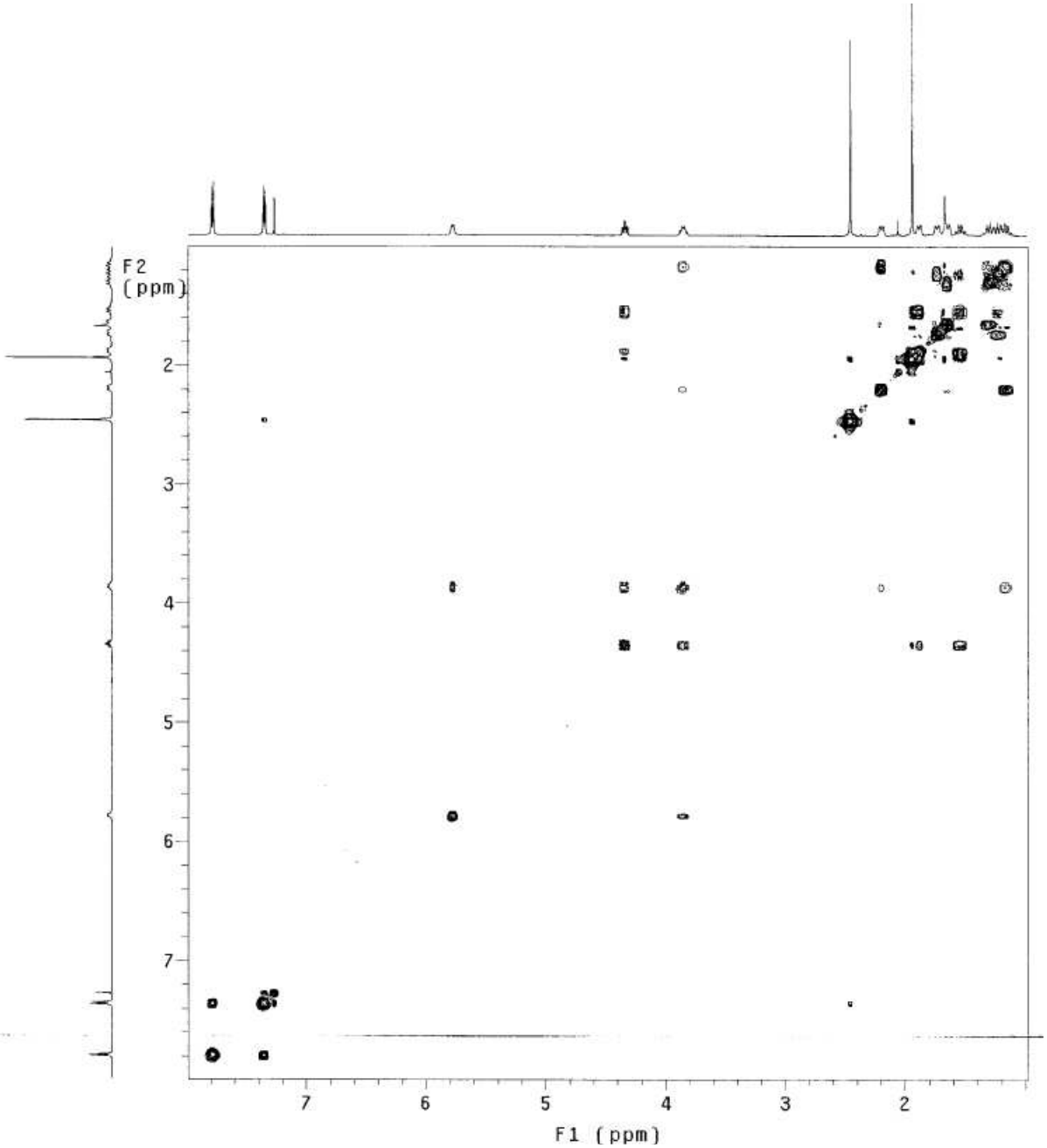


TY2-360-C-crystal

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, 499.8611711 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

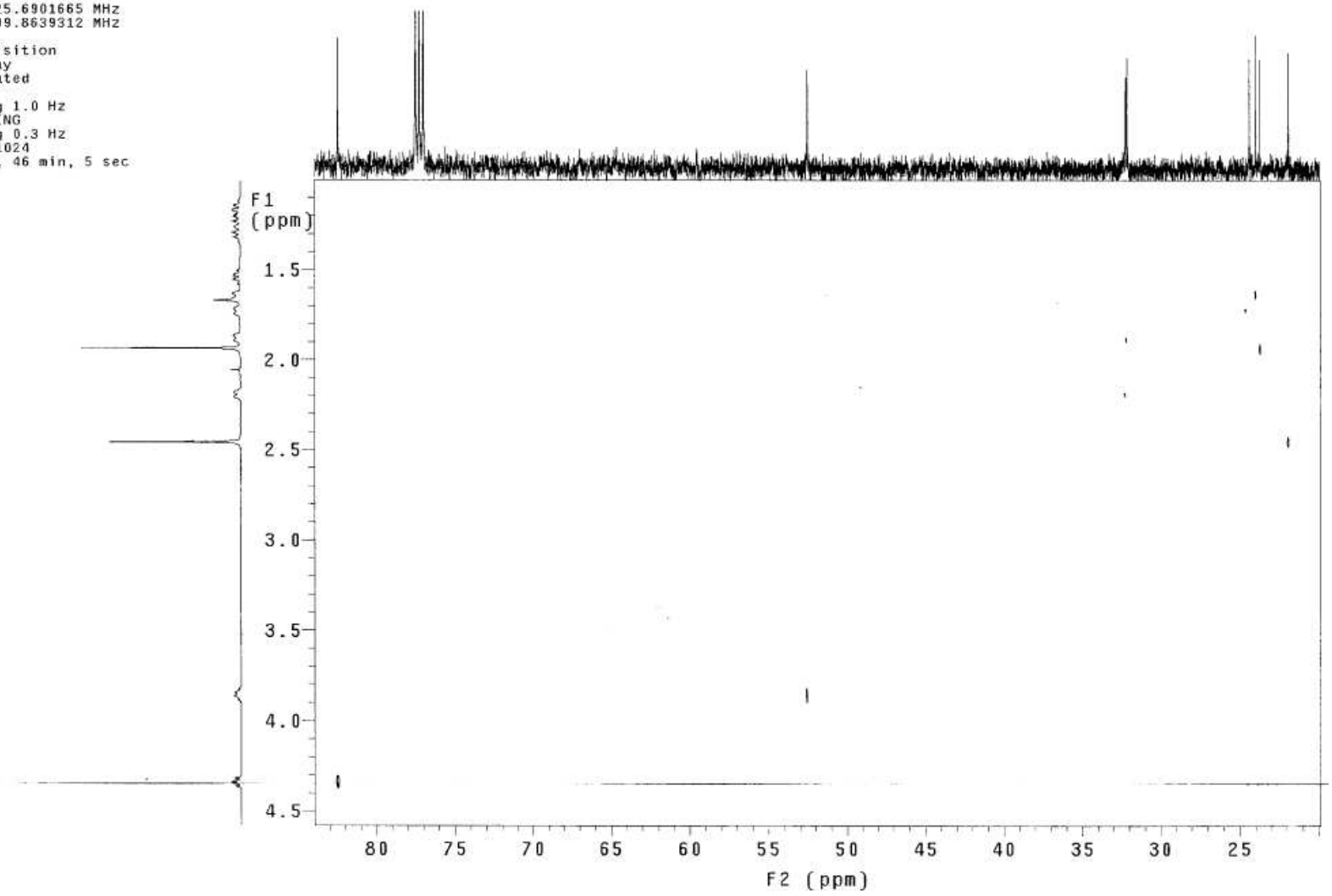


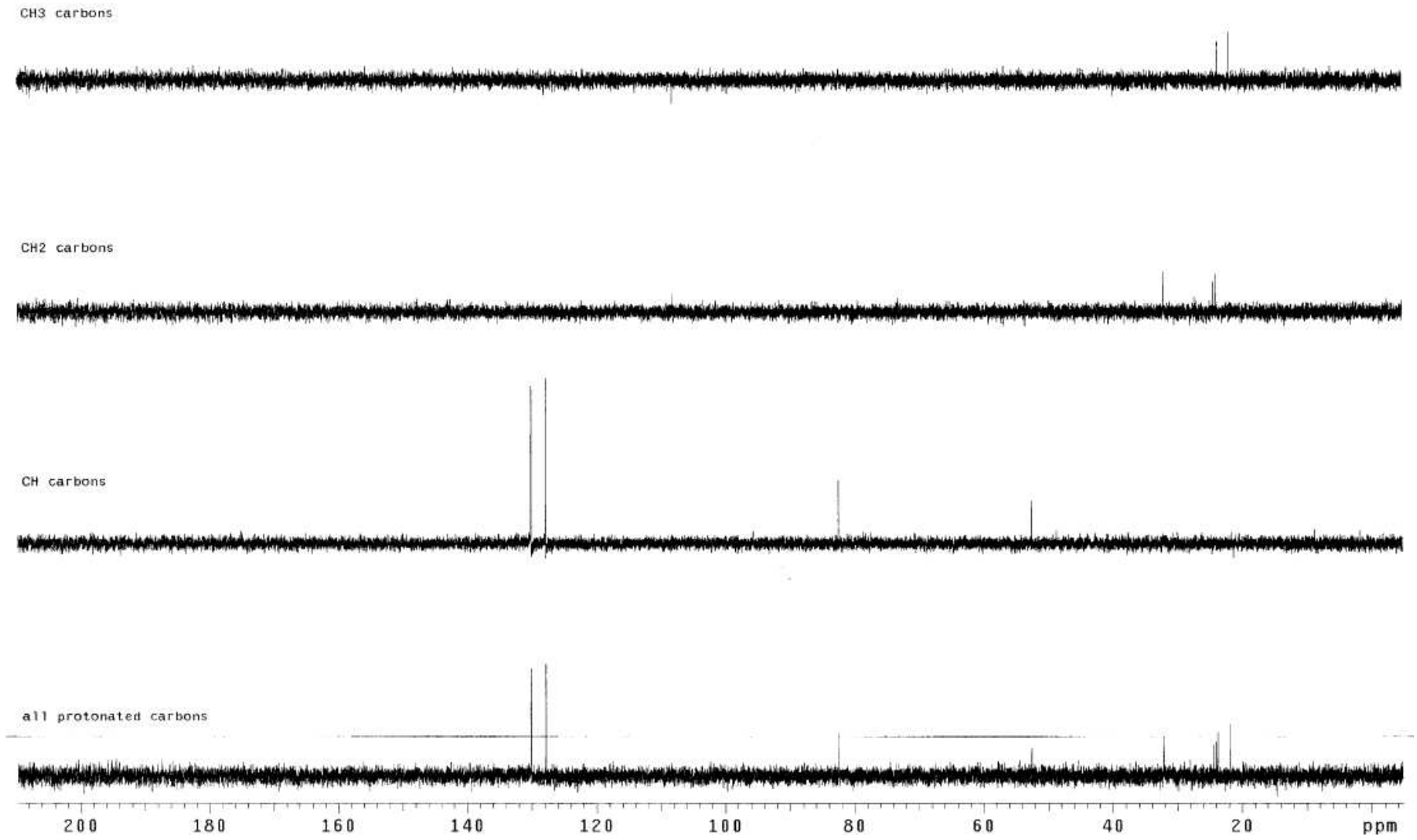
TY2-360-C-crystal

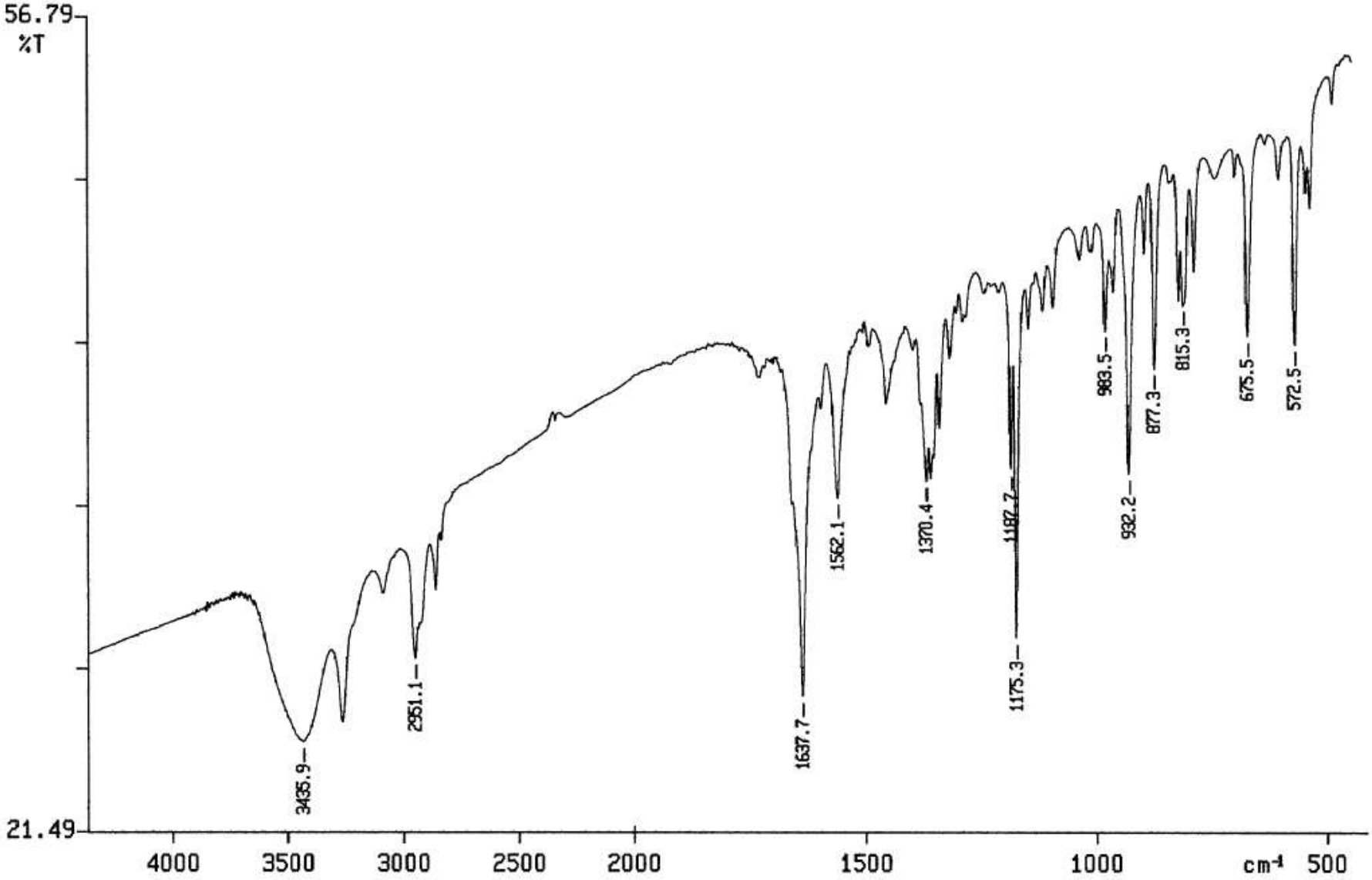
Pulse Sequence: hetcor

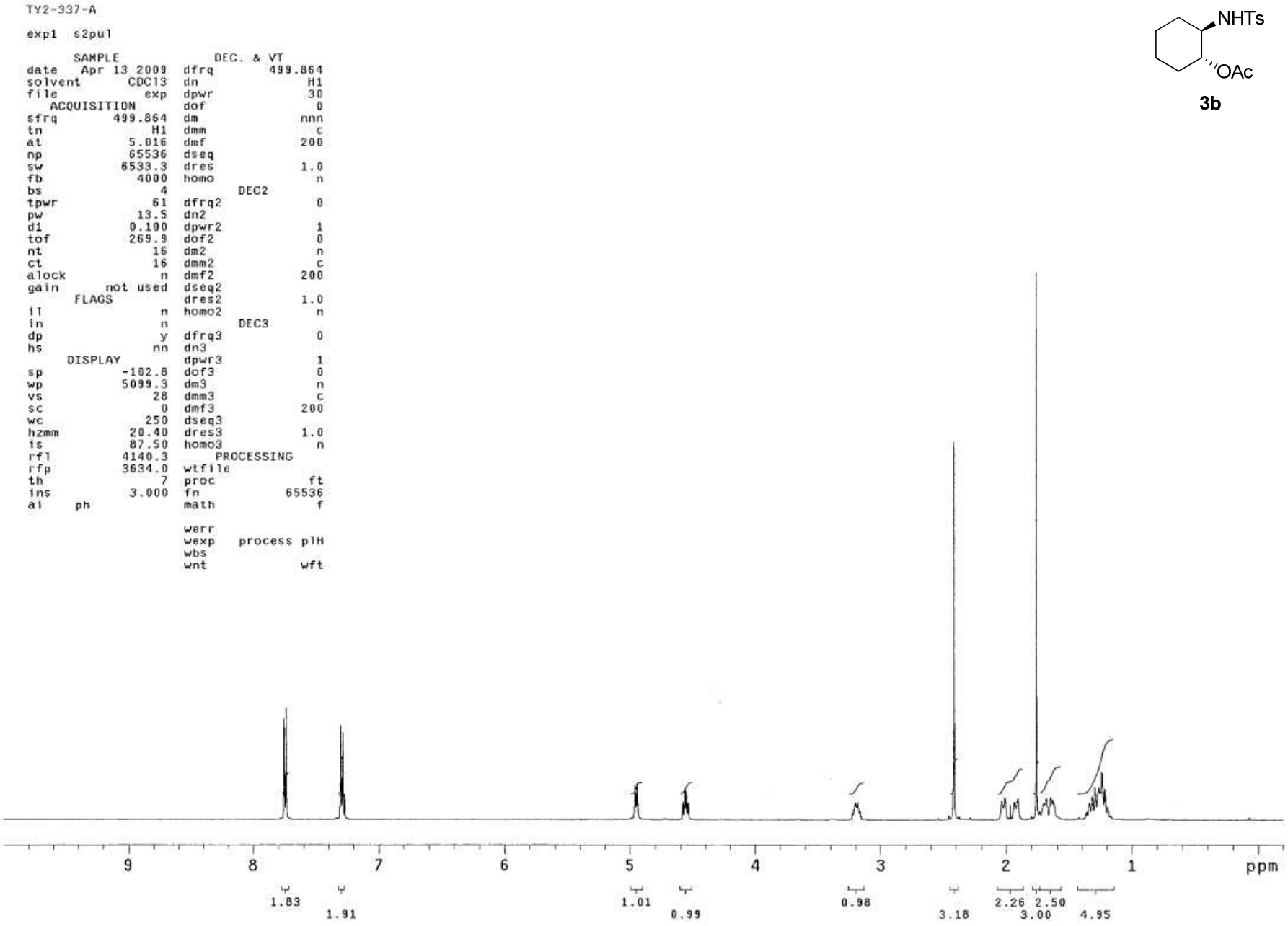
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

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, 125.6901665 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

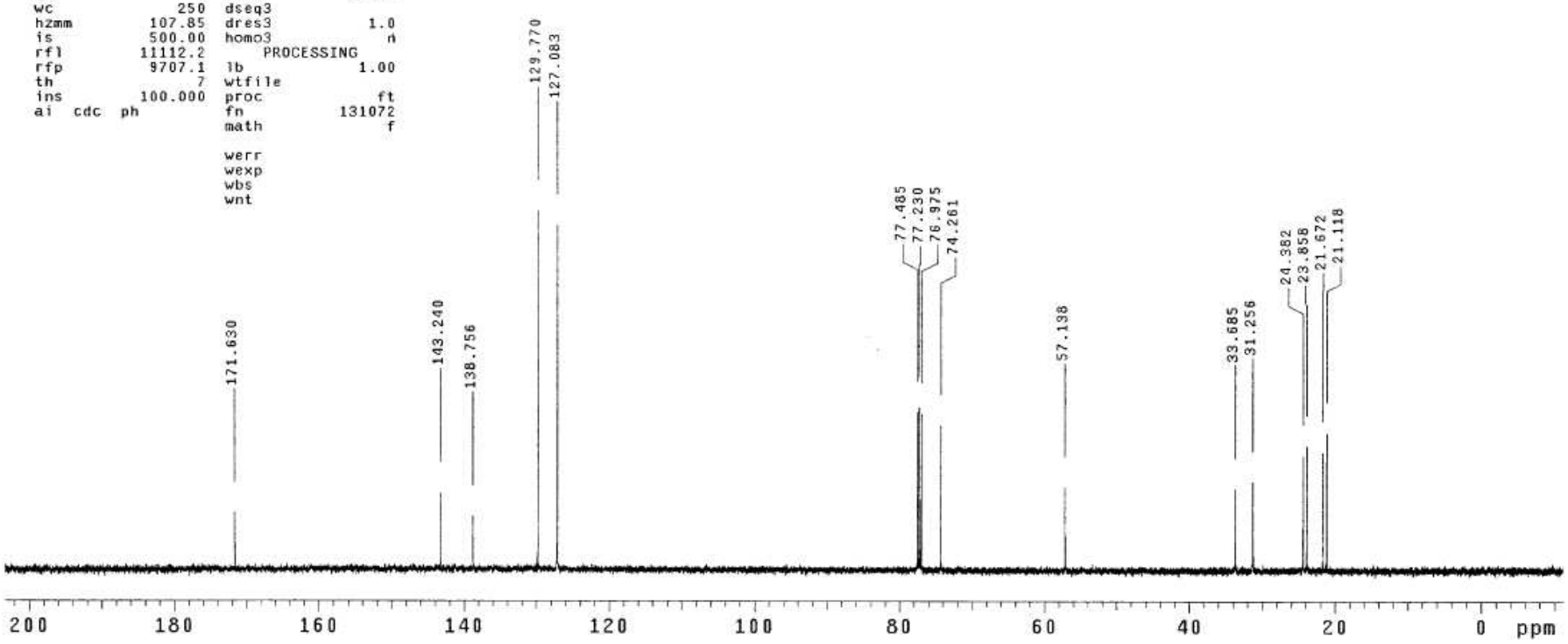

56.79
%T
21.49
3435.9
2951.1
1637.7
1562.1
1370.4
1187.2
1175.3
983.5
932.2
877.3
815.3
675.5
572.5
4000
3500
3000
2500
2000
1500
1000
cm-1
500


TY2-337-A
expl s2pul
SAMPLE
date Apr 13 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 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -102.8
wp 5099.3
vs 28
sc 0
wc 250
hzmm 20.40
is 87.50
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
NHTs
OAc
3b
9
8
7
6
5
4
3
2
1
ppm
1.83
1.91
1.01
0.99
0.98
3.18
2.26
2.50
3.00
4.95

TY2-337-A

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 13 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	99	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	-1404.8	dof3	0
wp	26962.9	dm3	n
vs	168	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11112.2	PROCESSING	
rfp	9707.1	lb	1.00
th	7	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

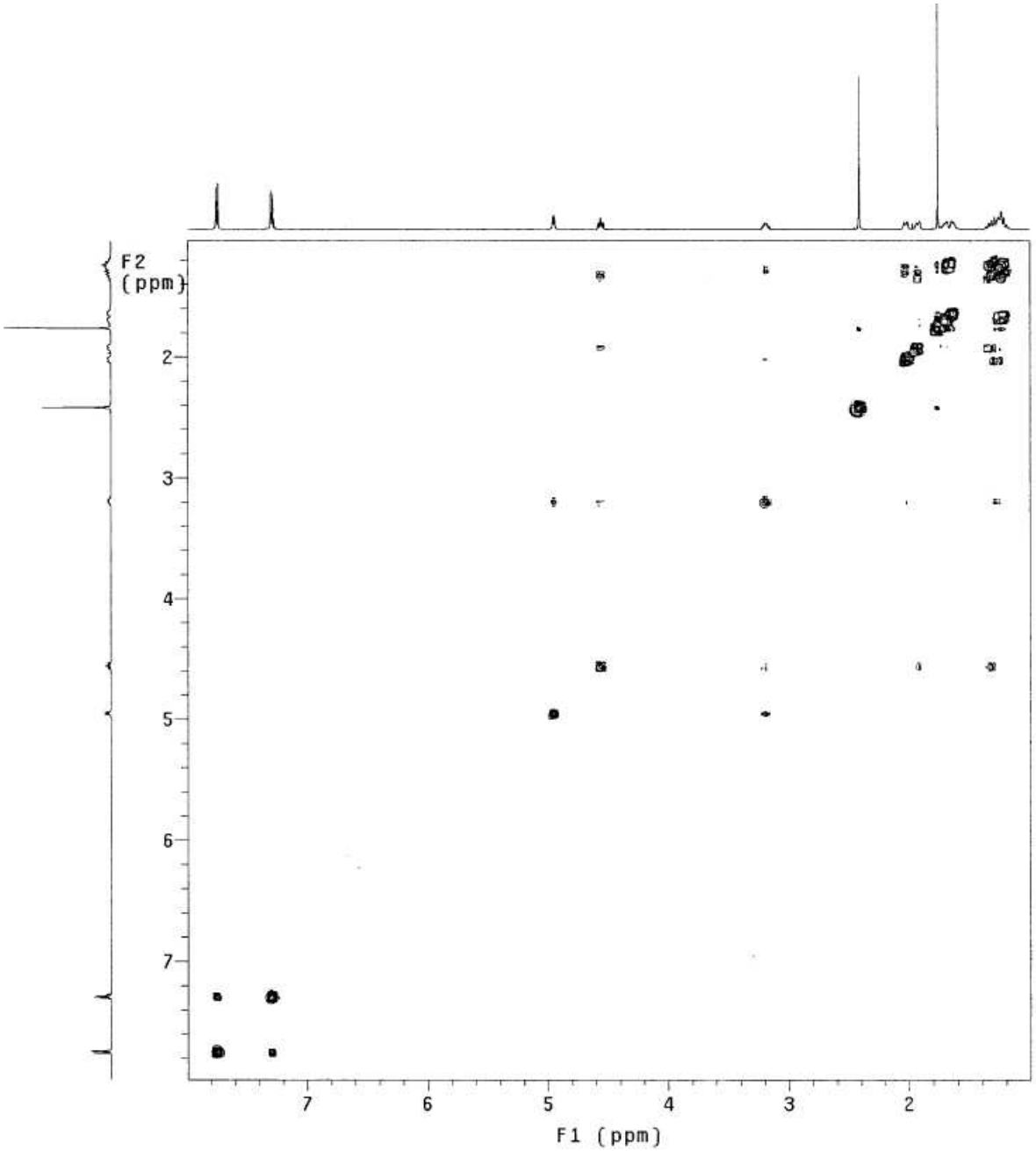


TY2-337-A

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

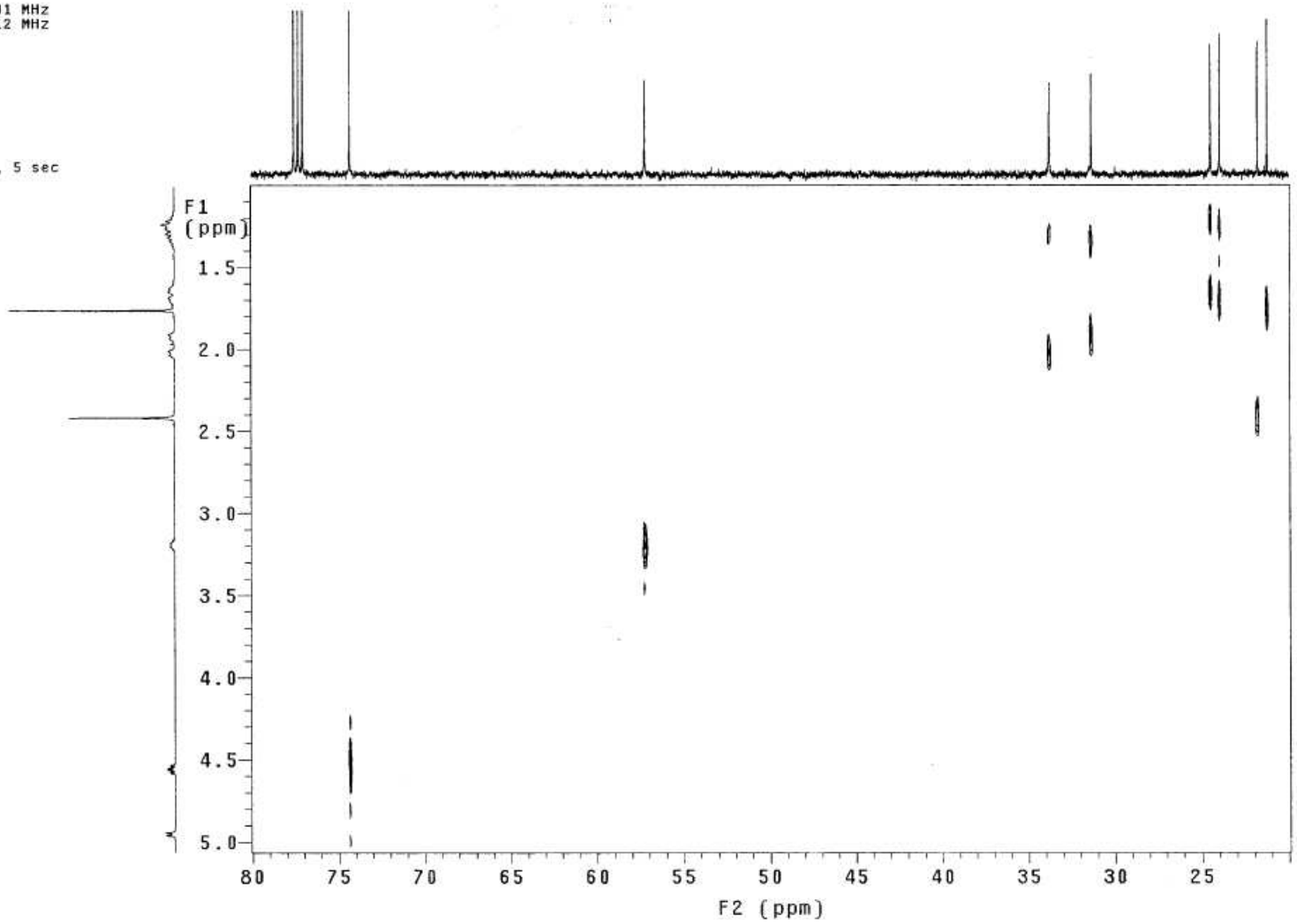


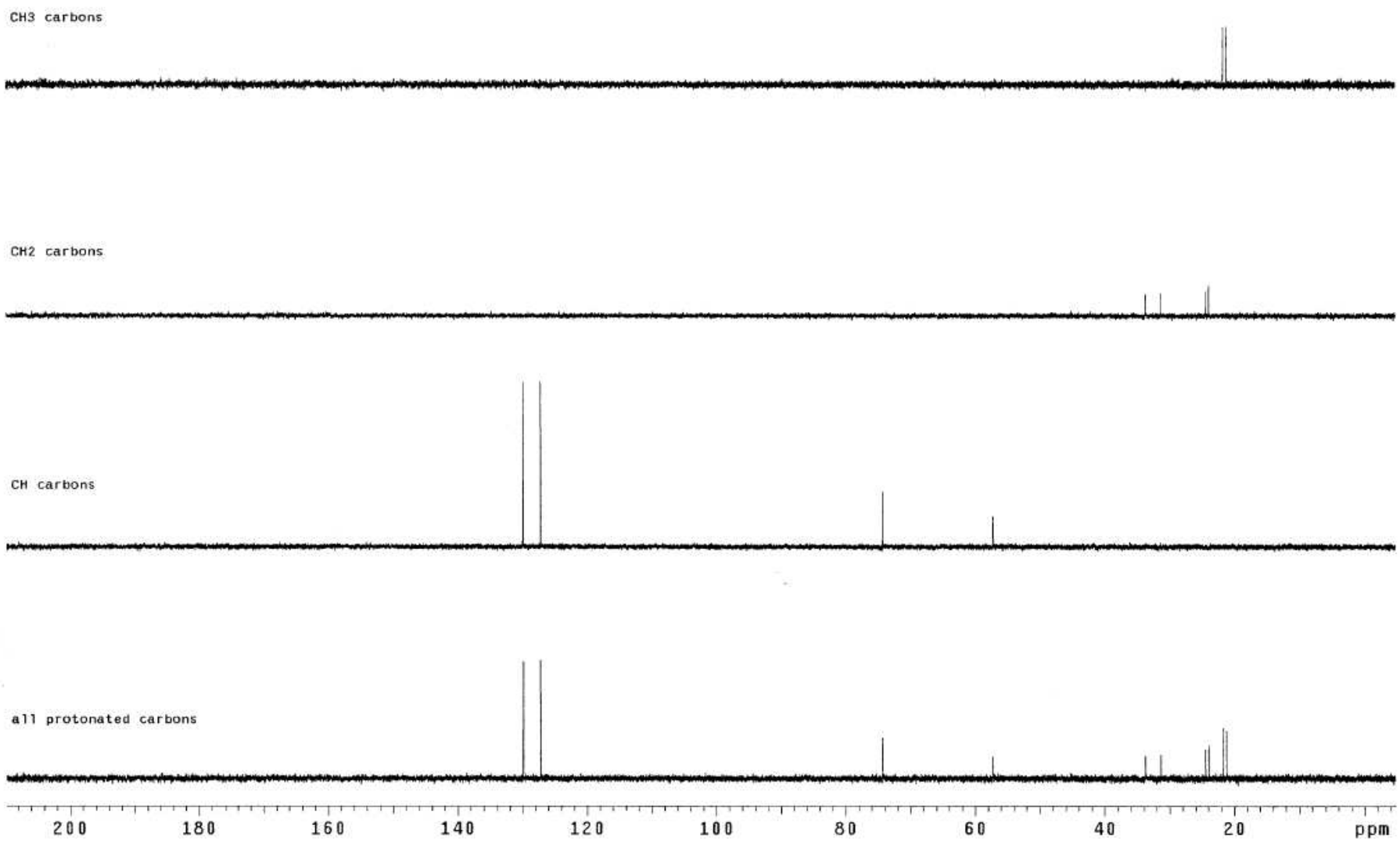
TY2-337-A

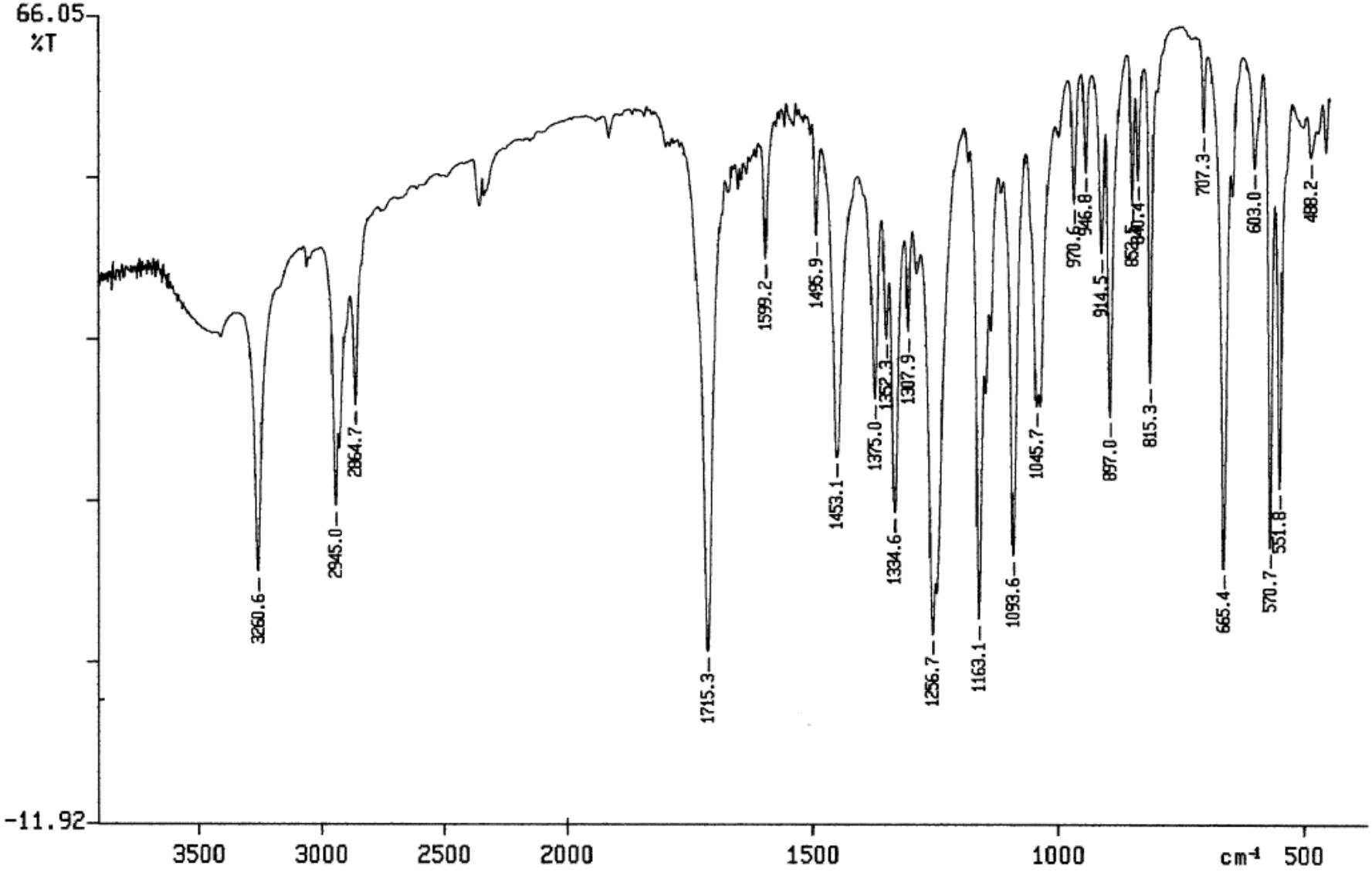
Pulse Sequence: hetcor

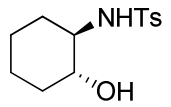
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

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, 125.6901591 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 3 hr, 46 min, 5 sec



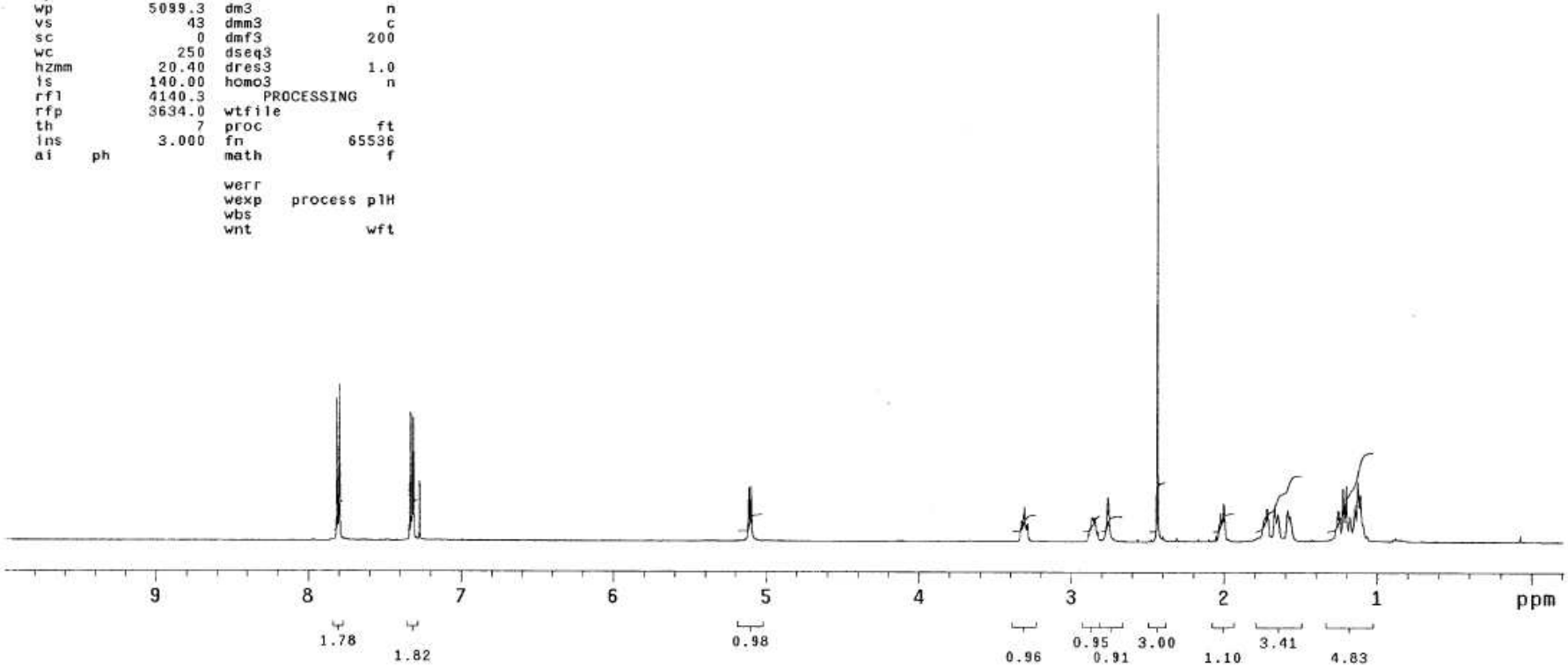

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


66.05
%T
-11.92
3260.6
2945.0
2864.7
1715.3
1599.2
1495.9
1453.1
1375.0
1352.3
1334.6
1307.9
1256.7
1163.1
1093.6
1045.7
970.6
946.8
914.5
897.0
852.5
840.4
815.3
707.3
665.4
603.0
570.7
551.8
488.2
3500
3000
2500
2000
1500
1000
cm-1
500



15b

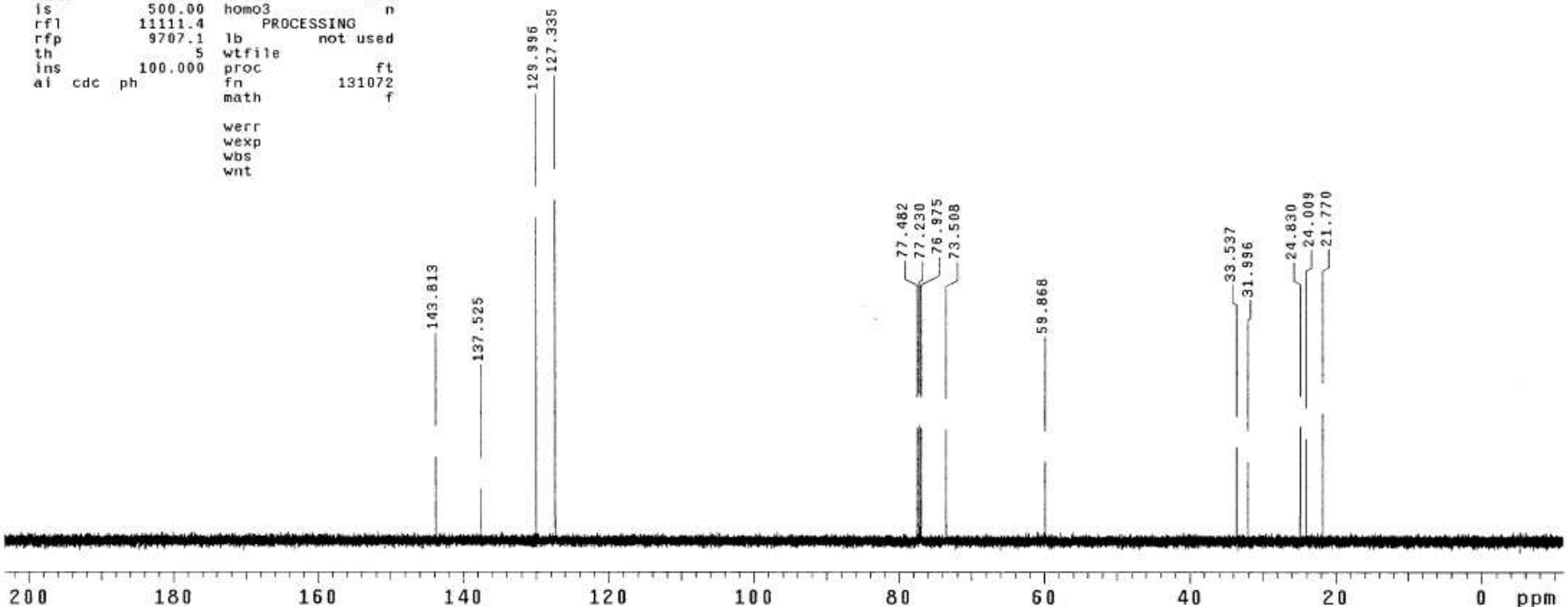
```
TY2-337-B

exp1  s2pul

      SAMPLE
date    Apr 13 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              16
ct              16
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp          -102.8
wp          5099.3
vs              43
sc               0
wc             250
hzmm         20.40
is           140.00
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 p1H
wbs
wnt             wft
```



TY2-337-B

exp2 s2pul

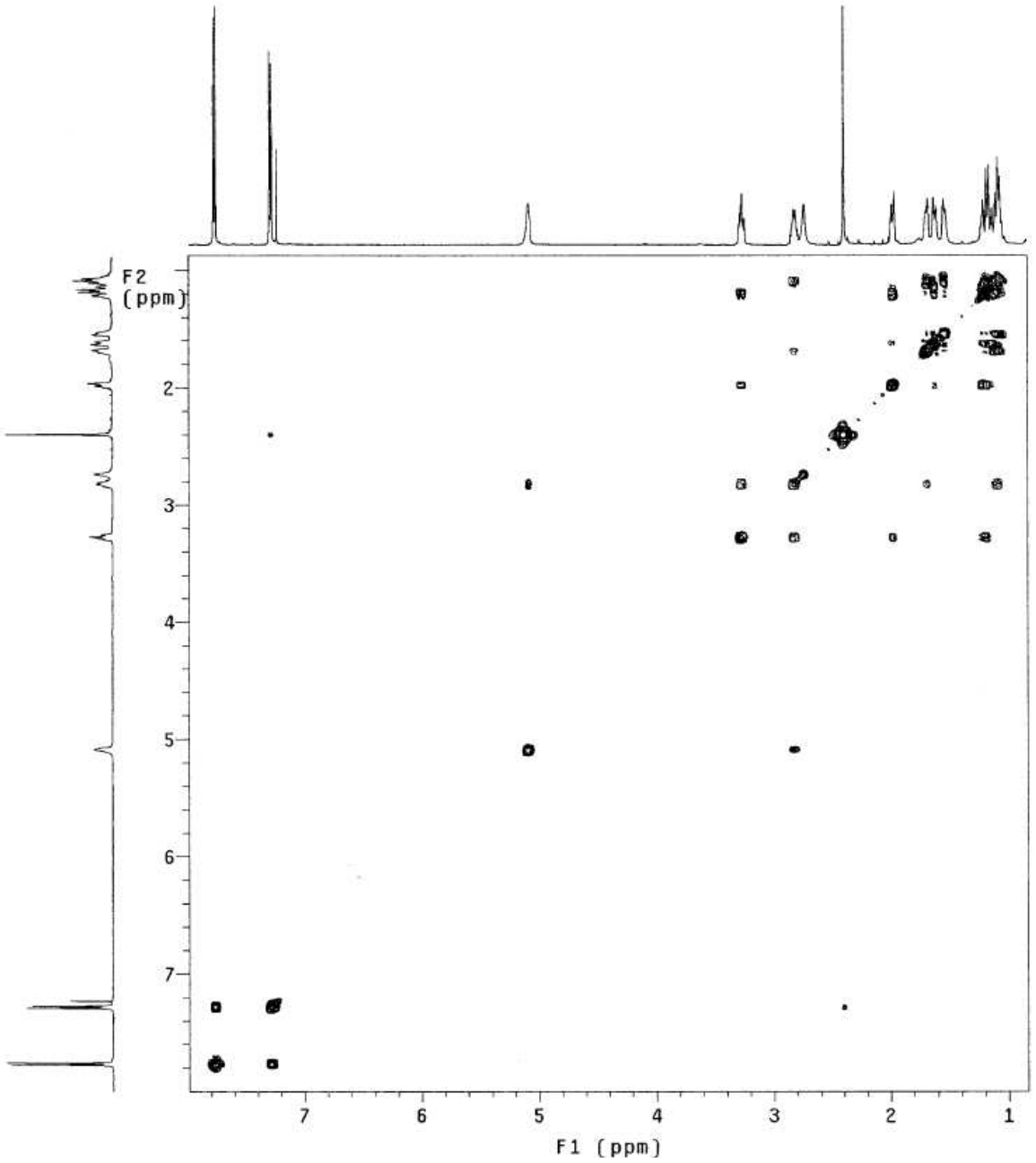
SAMPLE		DEC. & VT	
date	Apr 13 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	3000	dm2	n
ct	78	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	-1403.9	dof3	0
wp	26962.9	dm3	n
vs	78	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11111.4	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	



TY2-337-B

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

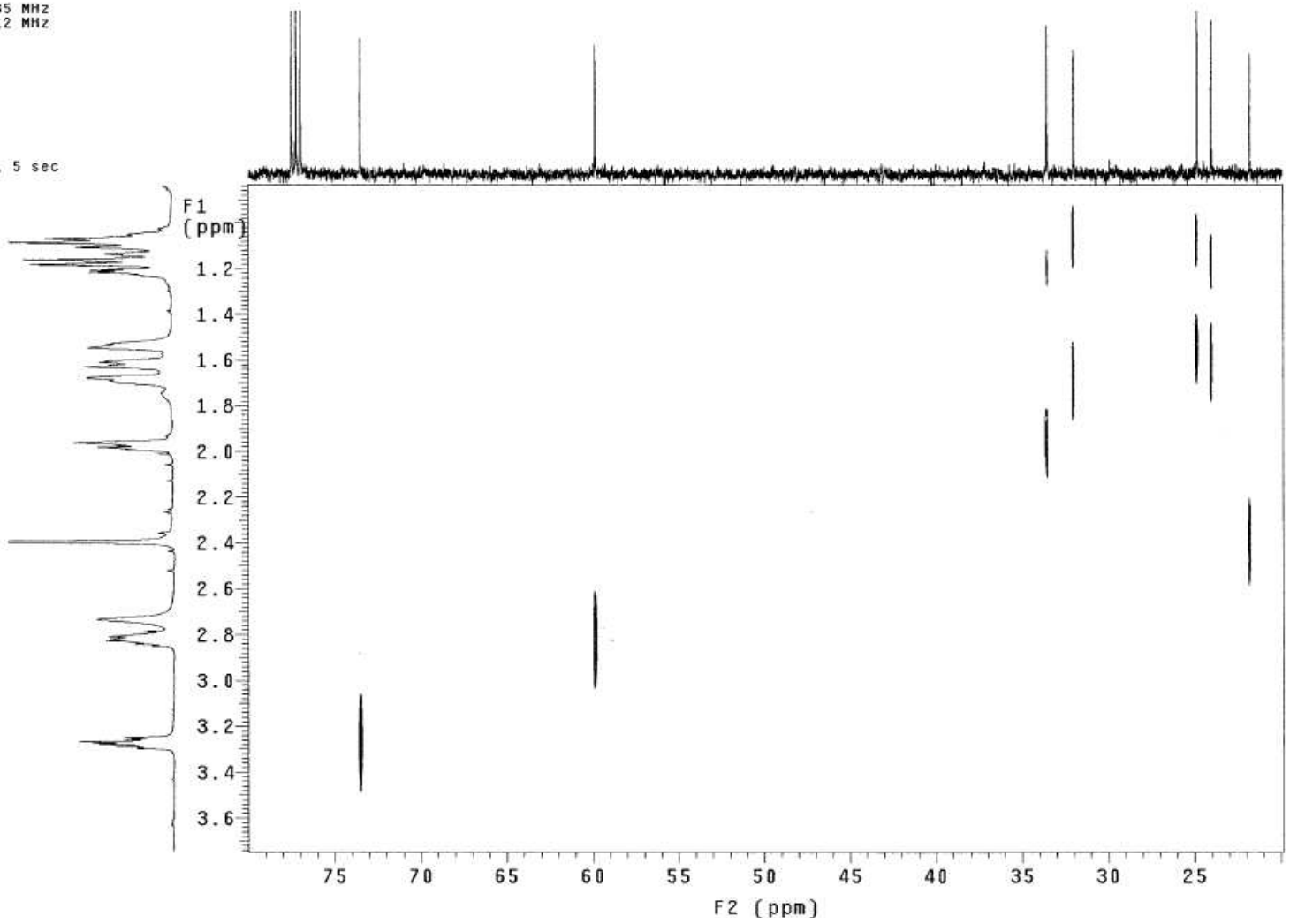
 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width 6533.3 Hz
 2D Width 6533.3 Hz
 32 repetitions
 256 increments
OBSERVE H1, 499.8611905 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

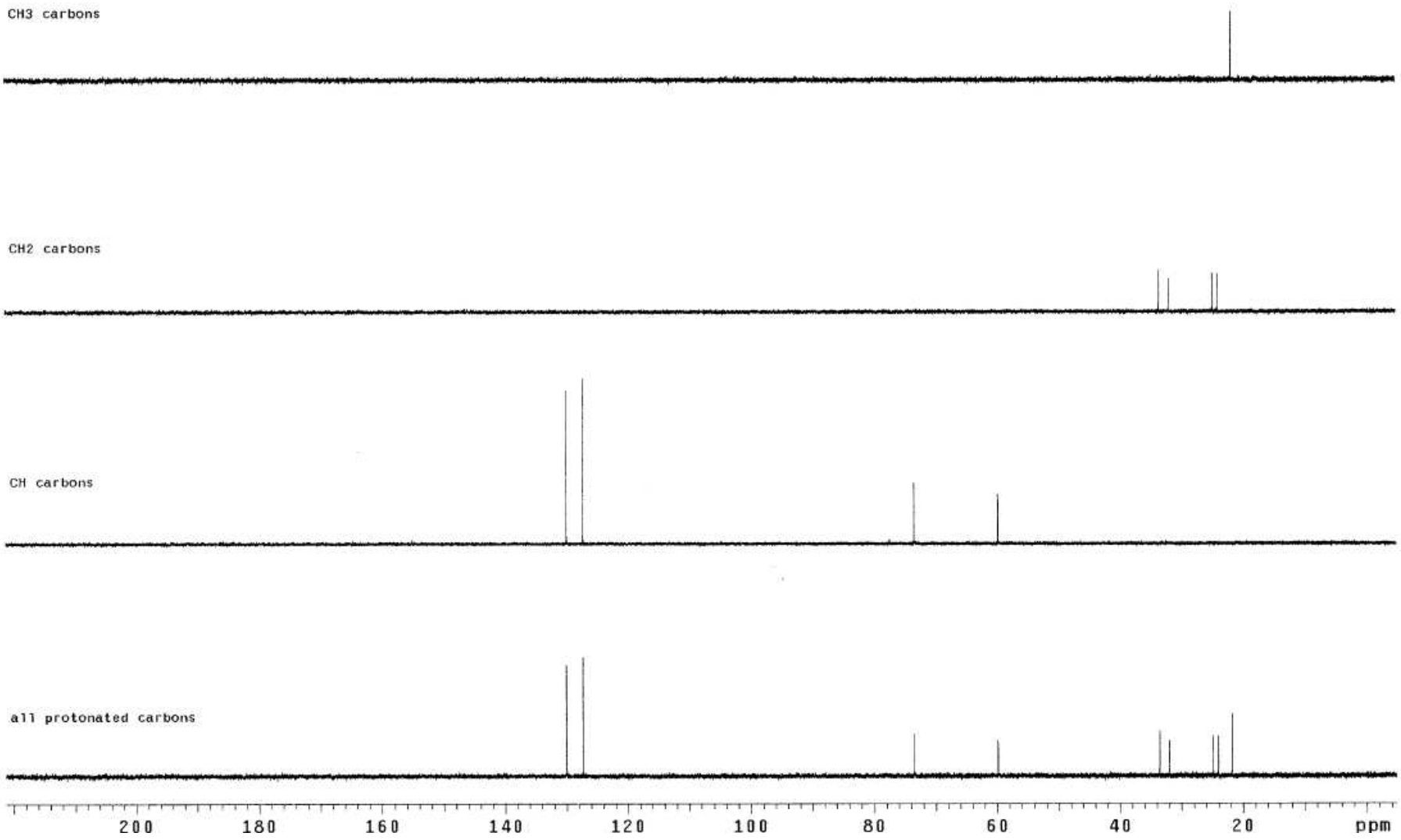


TY2-337-B

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, 125.6901685 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

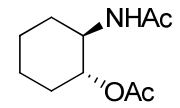



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

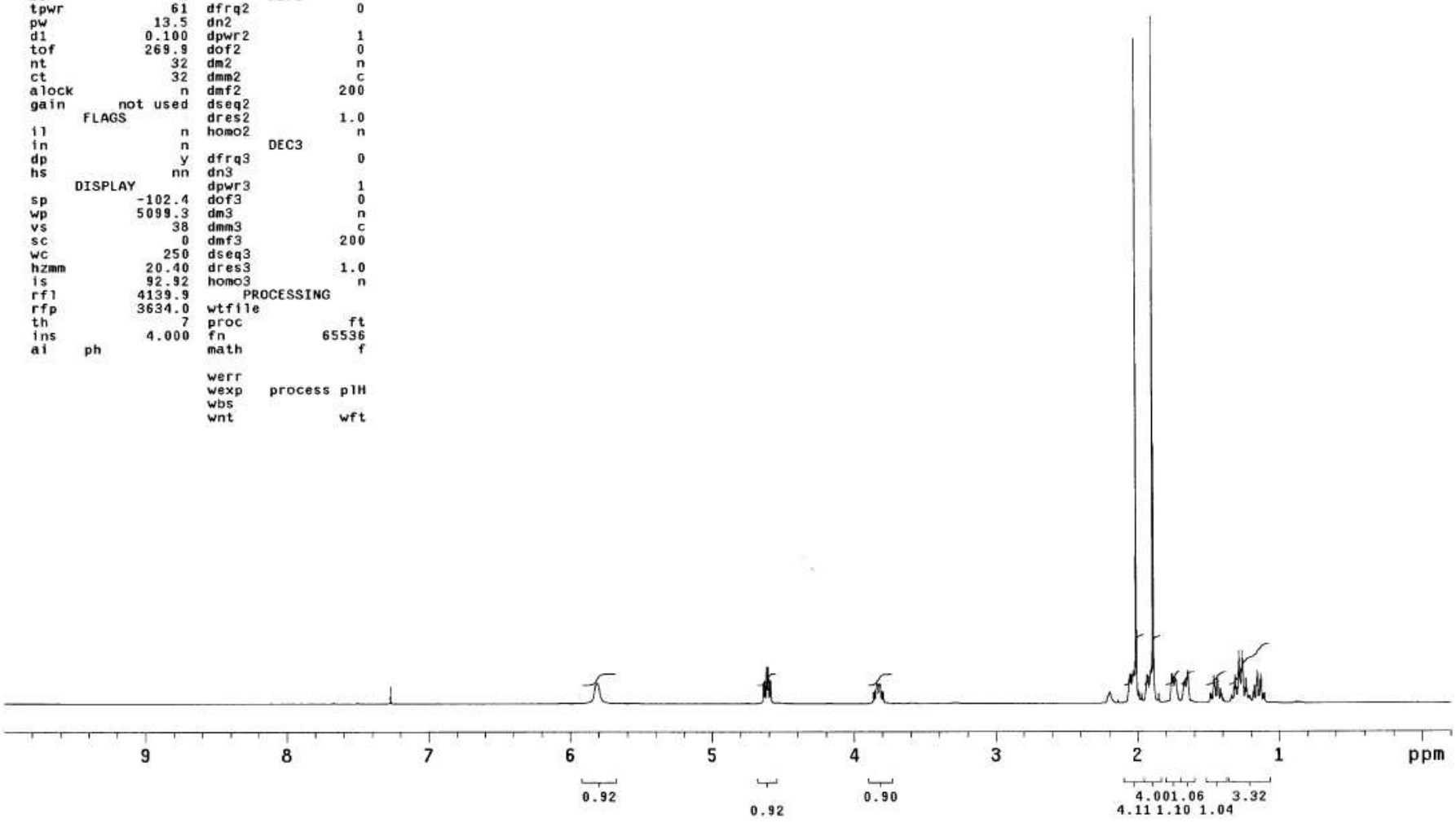
TY2-378

expl s2pul

SAMPLE		DEC. & VT	
date	Jun 29 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	-102.4	dof3	0
wp	5099.3	dm3	n
vs	38	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	92.92	homo3	n
rfl	4139.9	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	4.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



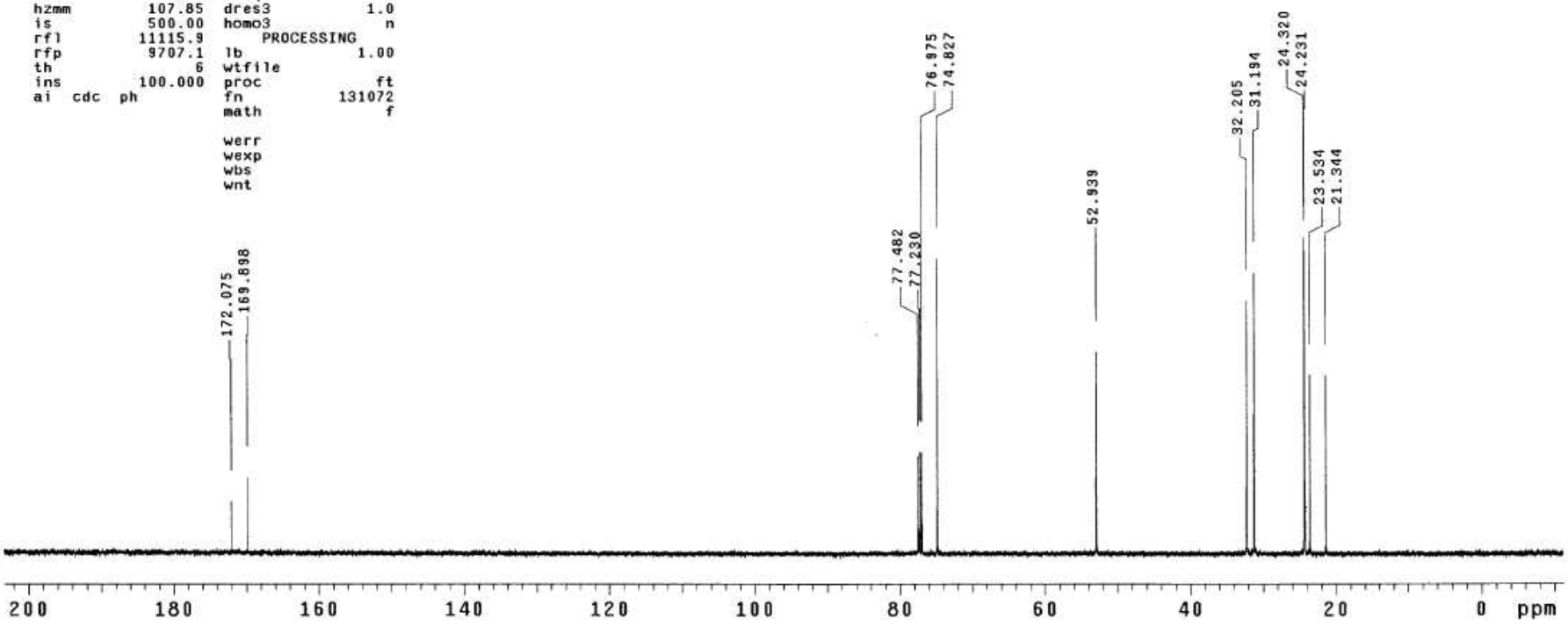
16b



TY2-378

exp2 s2pul

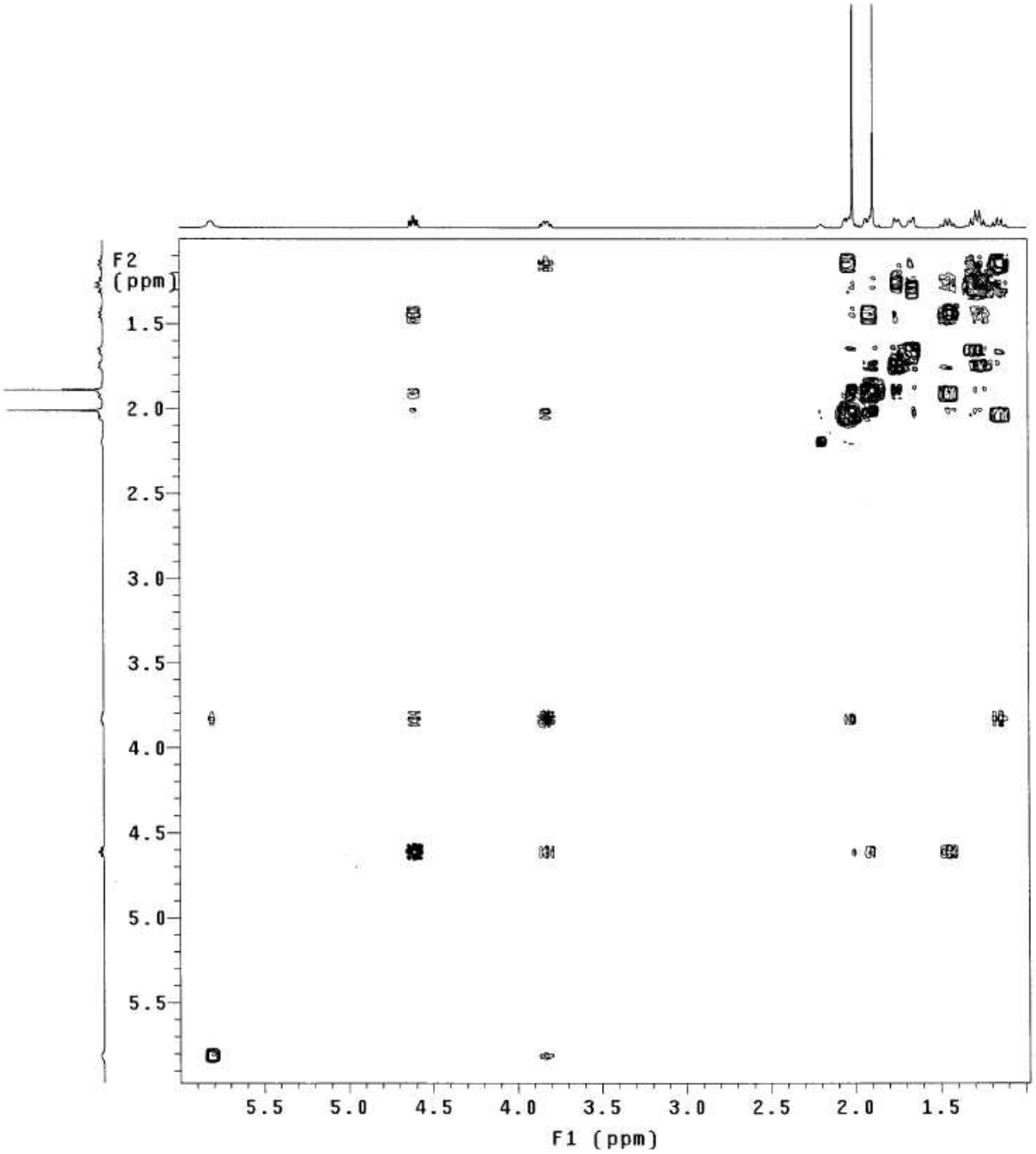
SAMPLE		DEC. & VT	
date	Jun 29 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	3000	dm2	n
ct	139	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	-1408.5	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	11115.9	PROCESSING	
rfp	9707.1	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-378

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, 499.8611705 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

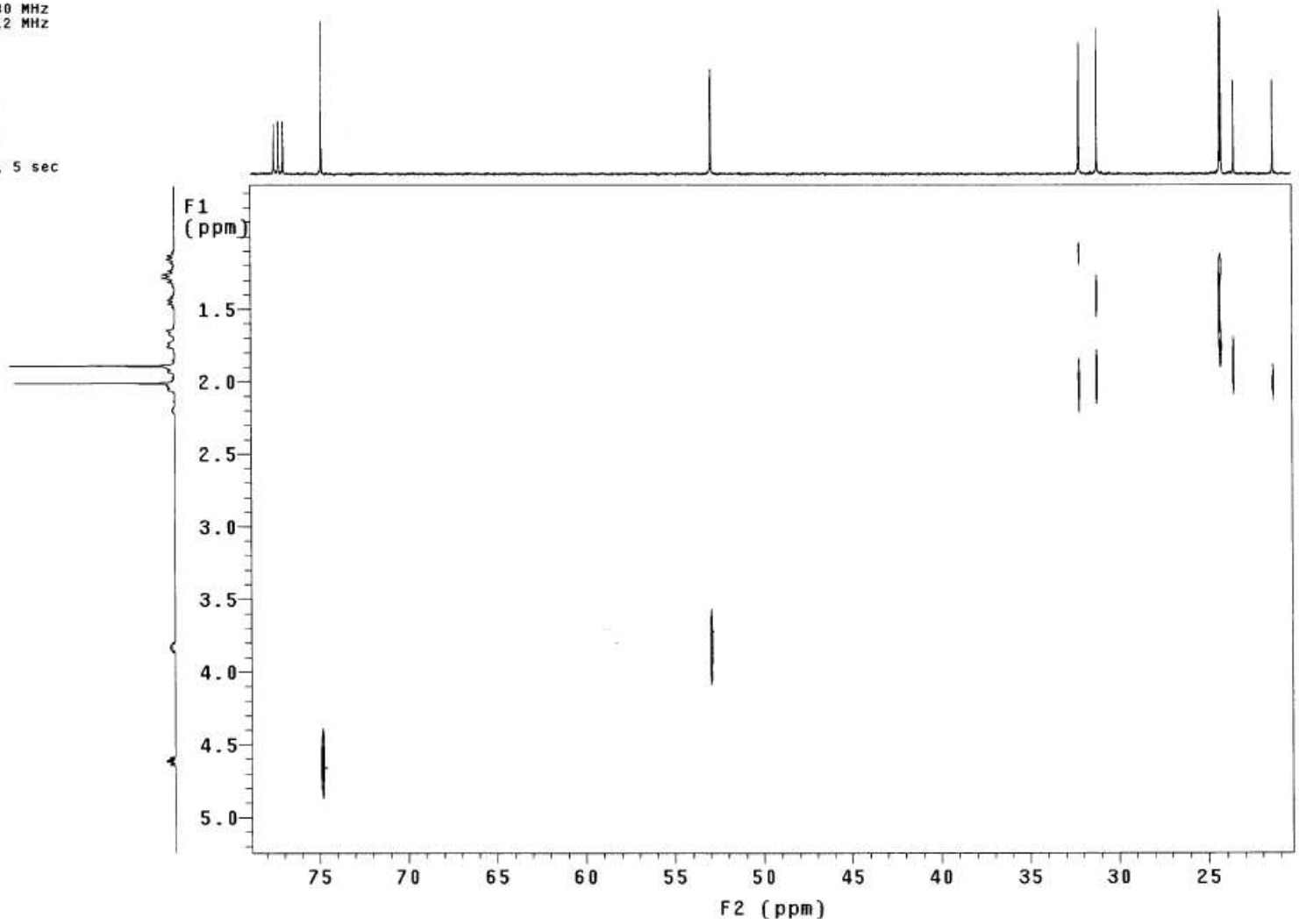


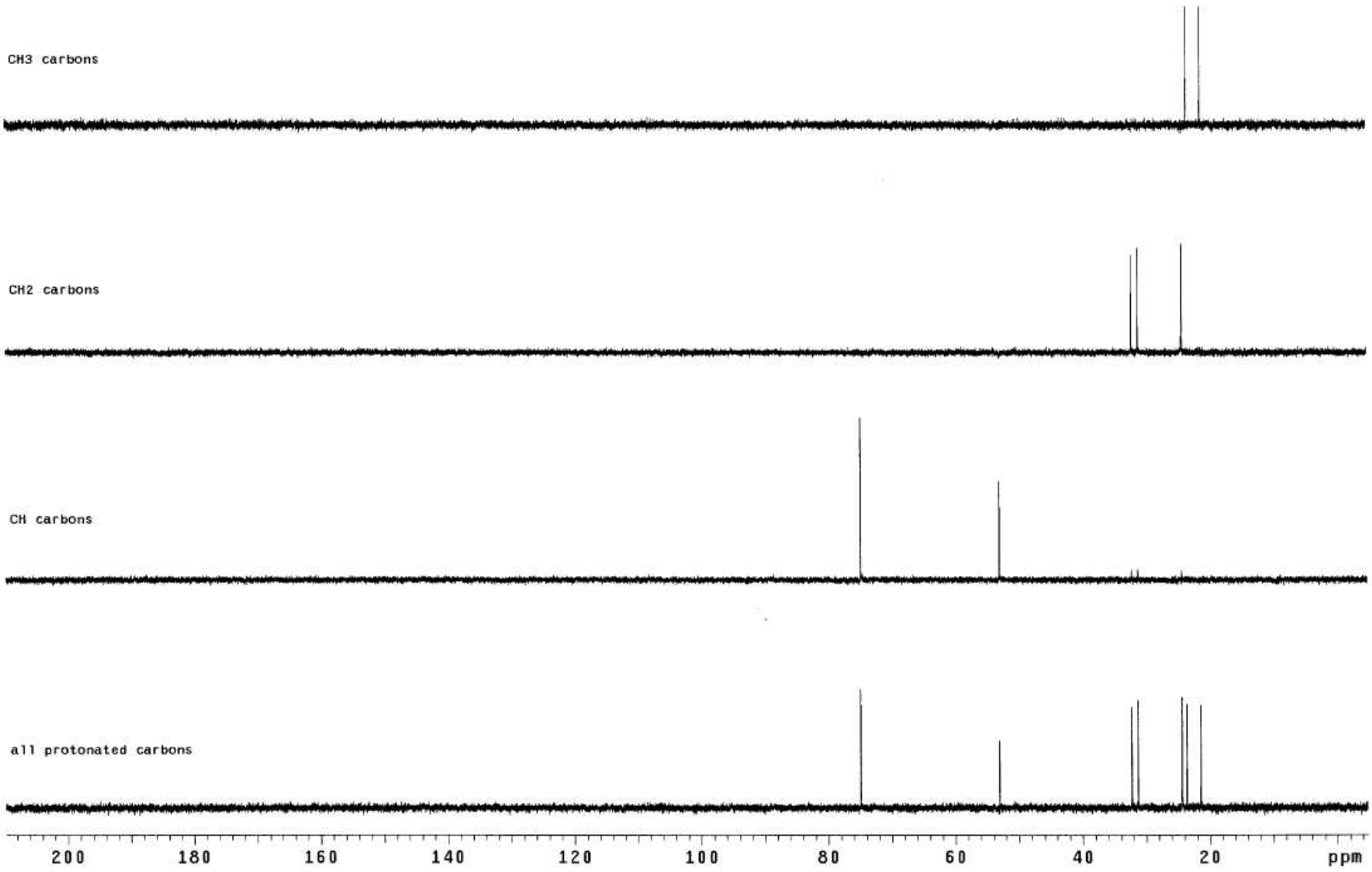
TY2-378

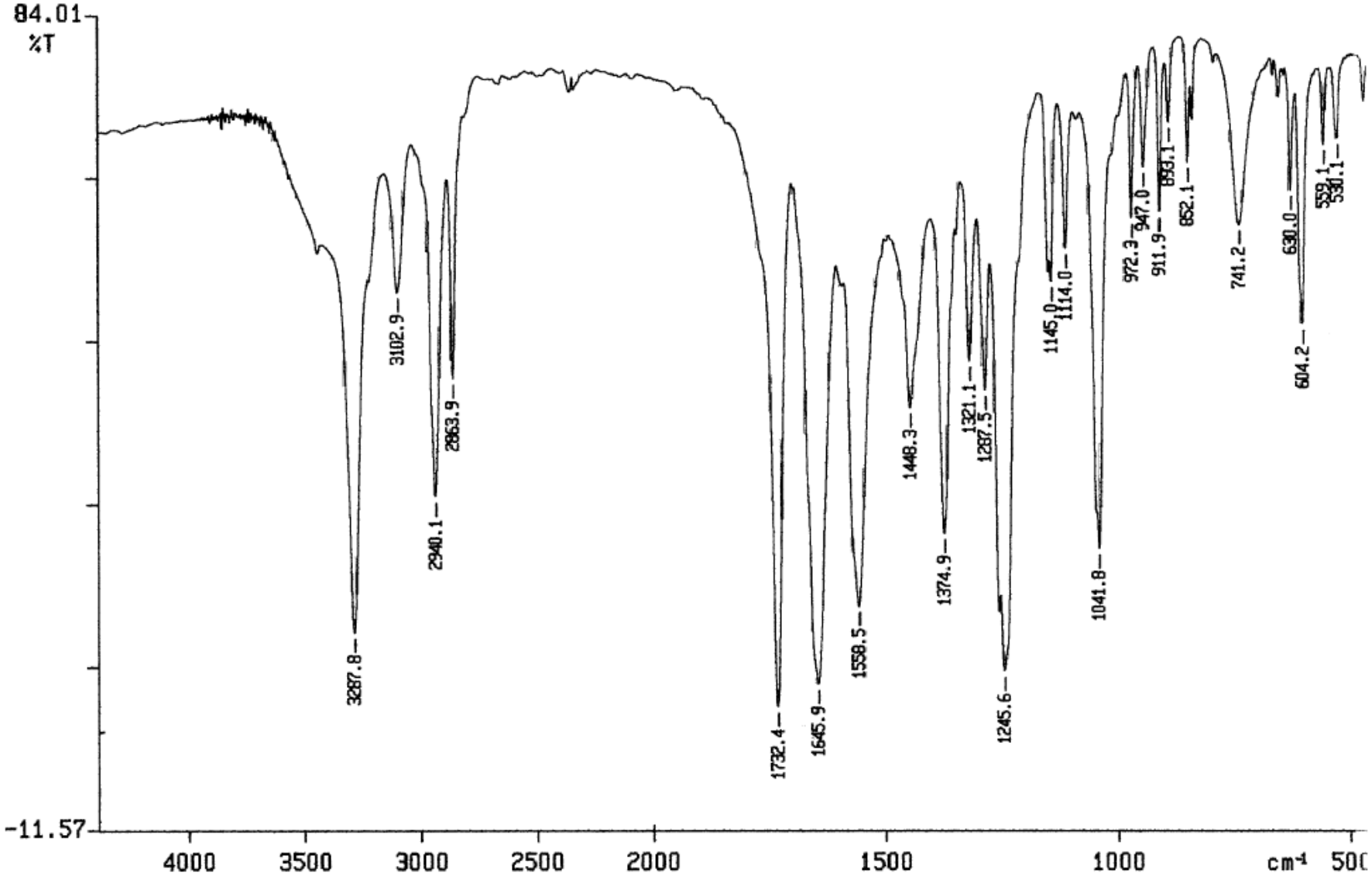
Pulse Sequence: hetcor

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, 125.6901730 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



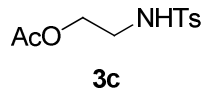

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


84.01
%T
-11.57
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3287.8
3102.9
2940.1
2863.9
1732.4
1645.9
1558.5
1448.3
1374.9
1321.1
1287.5
1245.6
1145.0
1114.0
1041.8
972.3
947.0
911.9
893.1
852.1
741.2
630.0
604.2
559.1
530.1

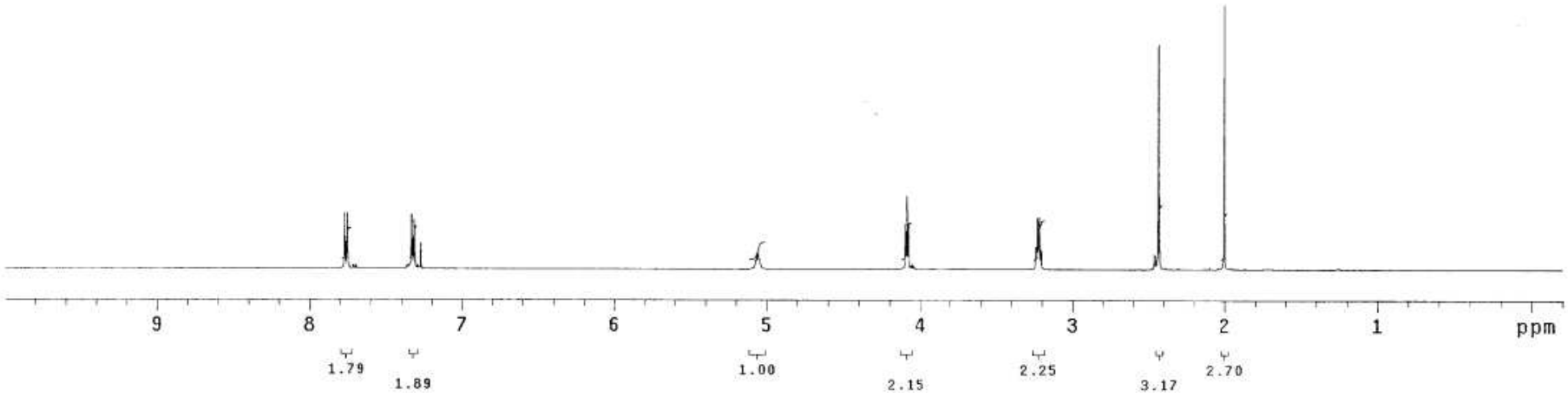
TY2-344

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 23 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	-102.2	dof3	0
wp	5099.3	dm3	n
vs	15	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	117.48	homo3	n
rfl	4139.7	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



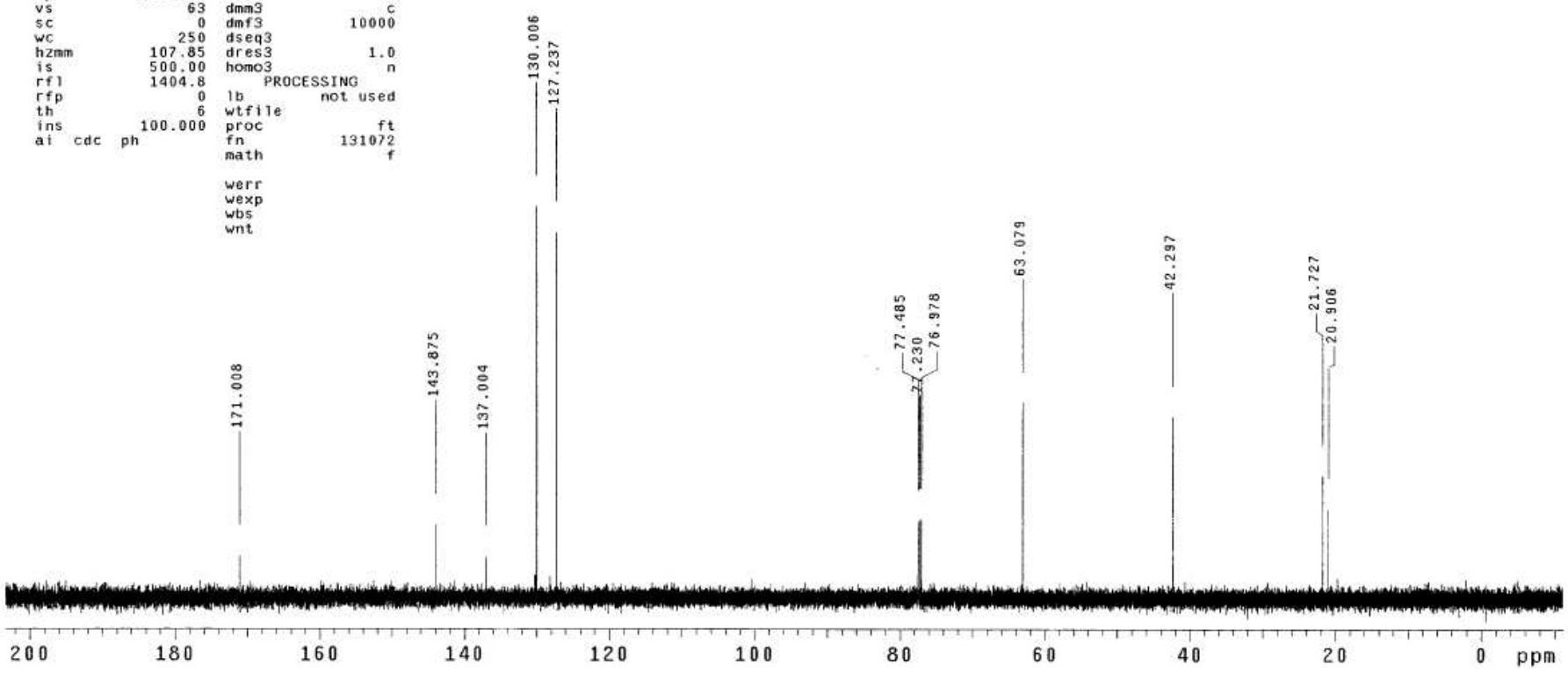
3c



TY2-344

exp2 s2pul

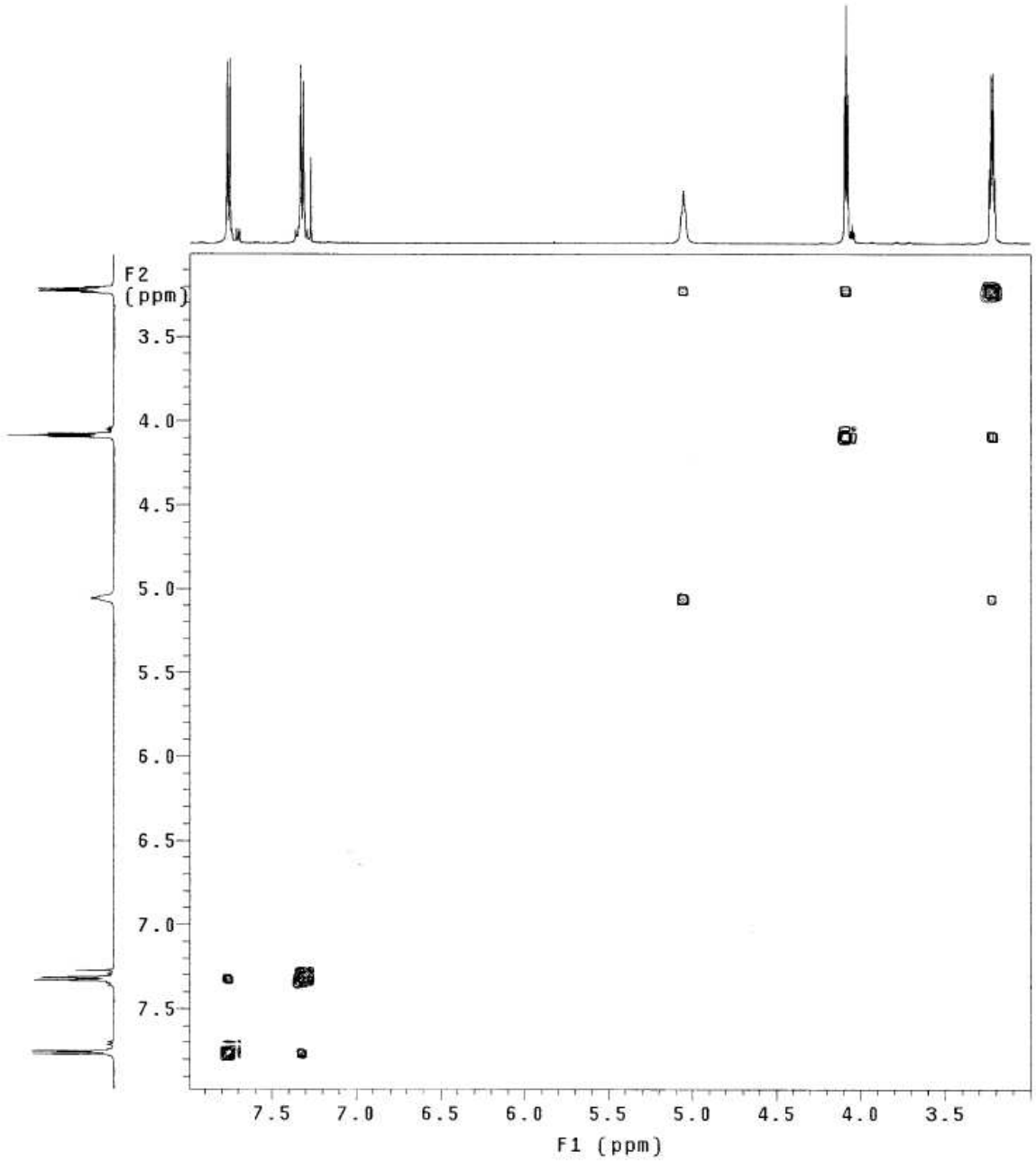
SAMPLE		DEC. & VT	
date	Apr 23 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	2000	dm2	n
ct	60	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	-1404.3	dof3	0
wp	26962.9	dm3	n
vs	63	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	1404.8	PROCESSING	
rfp	0	lb	not used
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-344

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, 499.8611703 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

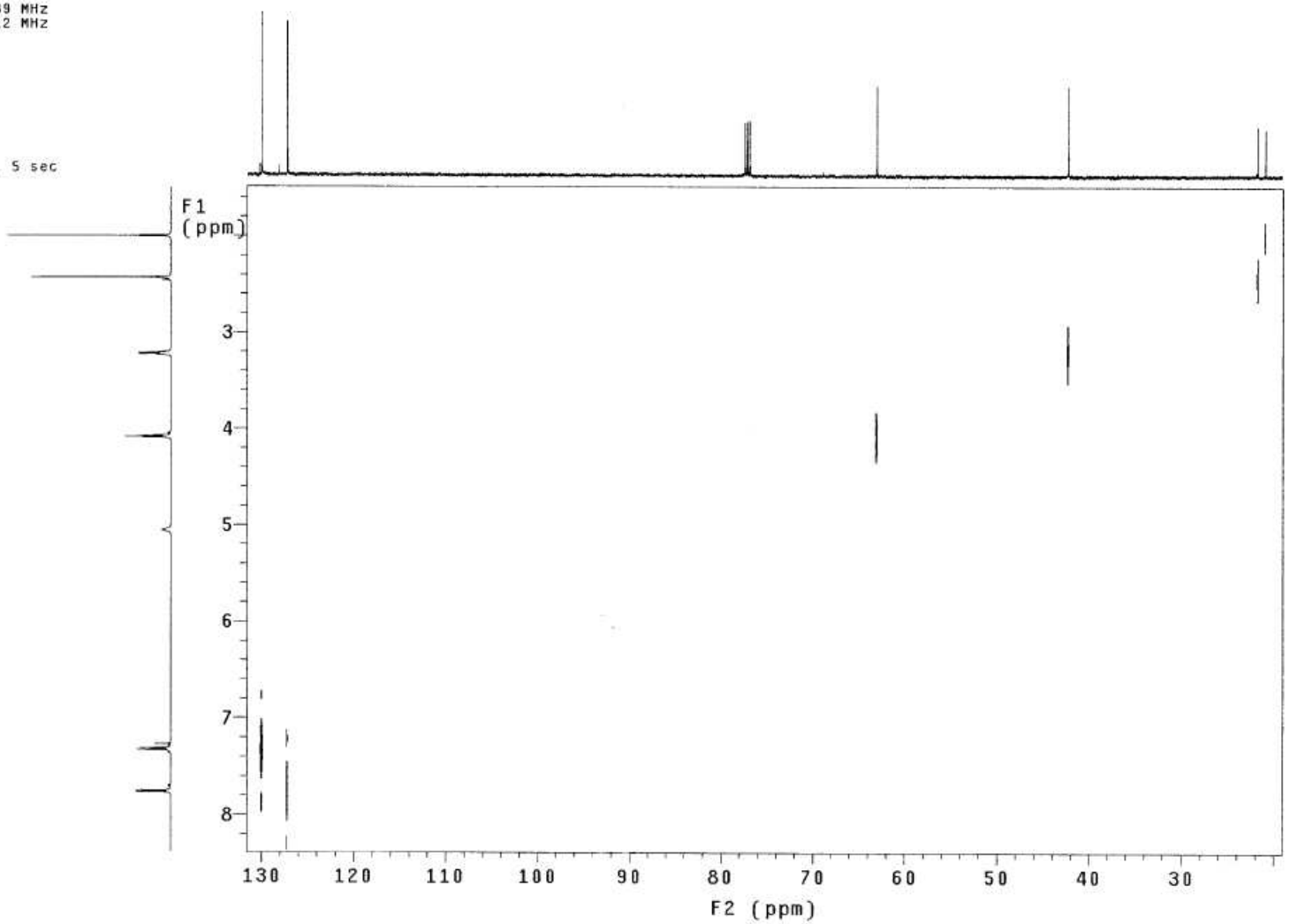


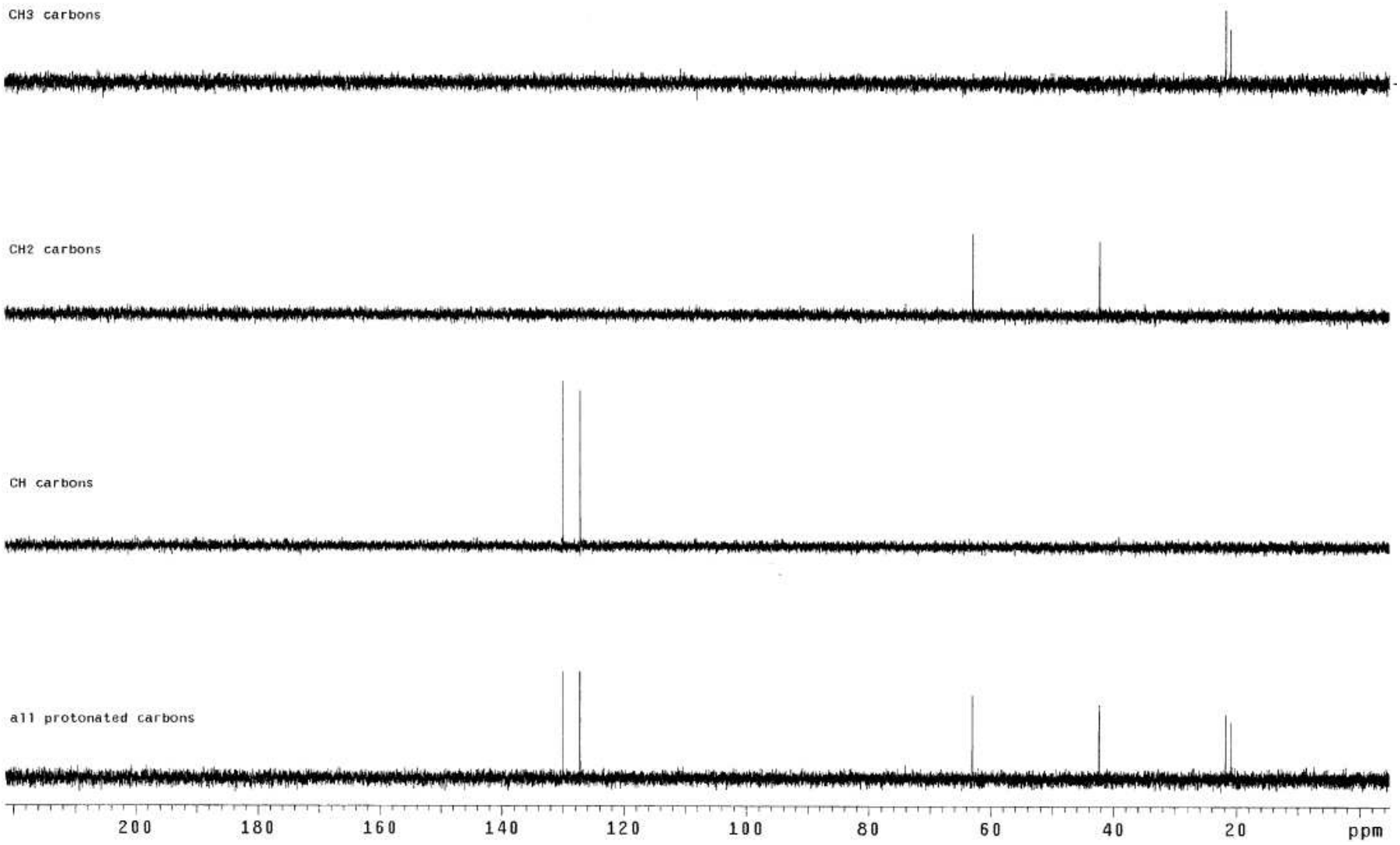
TY2-344

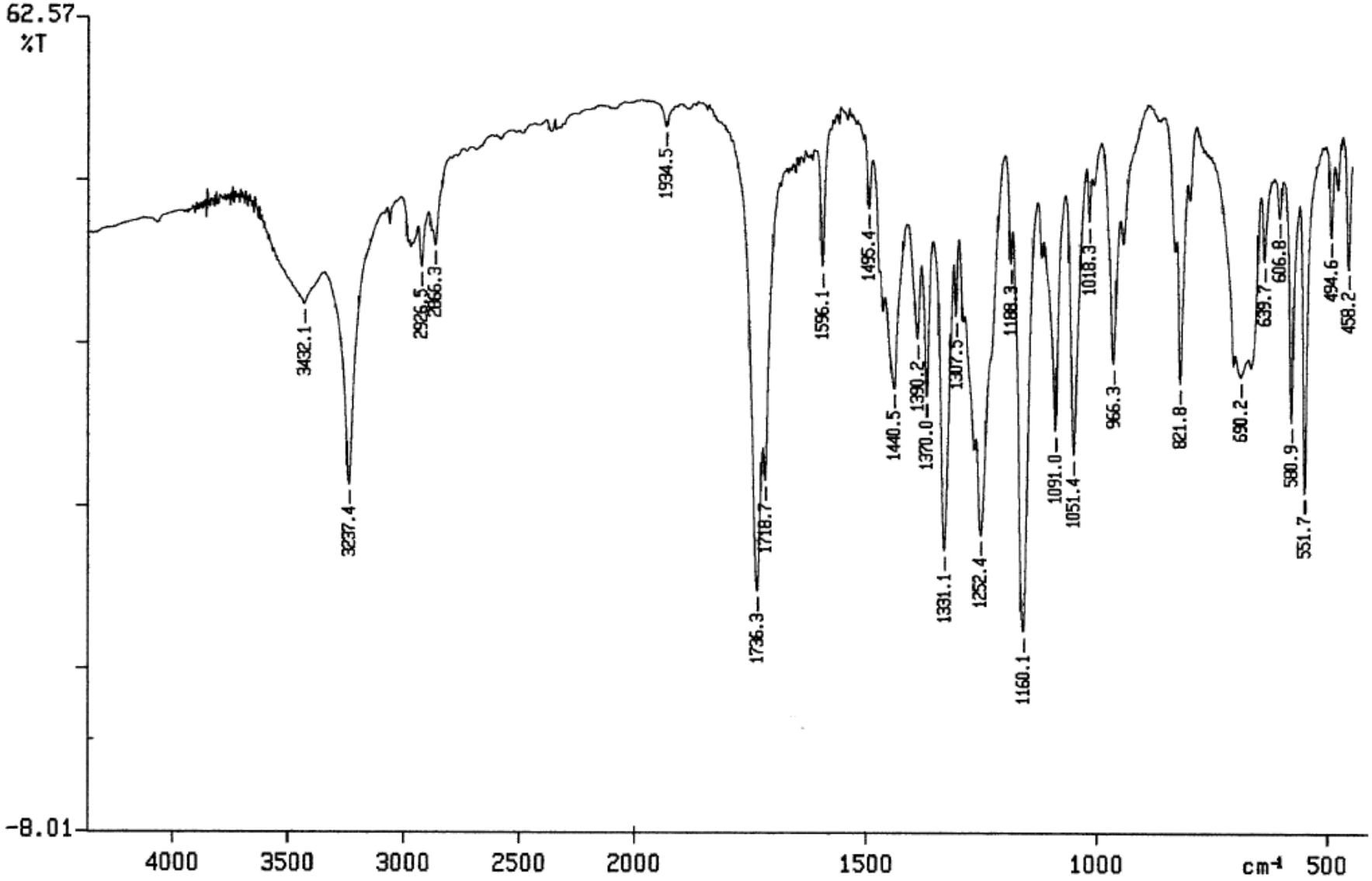
Pulse Sequence: hetcor

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, 125.6901689 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 3 hr, 46 min, 5 sec



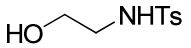

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


62.57
%T
-8.01
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3432.1
3237.4
2926.5
2866.3
1934.5
1736.3
1718.7
1596.1
1495.4
1440.5
1390.2
1370.0
1331.1
1307.5
1252.4
1188.3
1160.1
1091.0
1051.4
1018.3
966.3
821.8
690.2
639.7
606.8
580.9
551.7
494.6
458.2

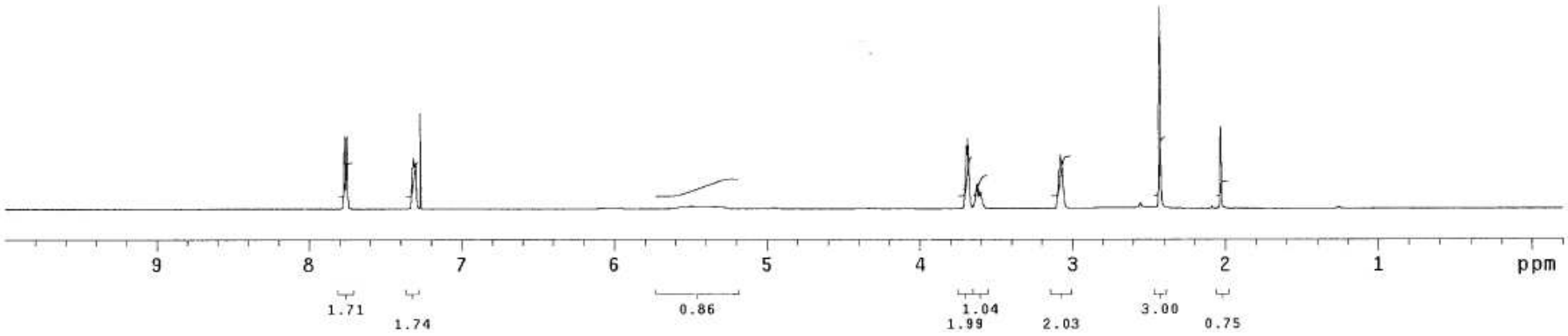
TY2-362-B-c15-22

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 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	-103.0	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	136.39	homo3	n
rfl	4140.5	PROCESSING	
rfp	3634.0	wtfile	
th	50	proc	ft
ins	3.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



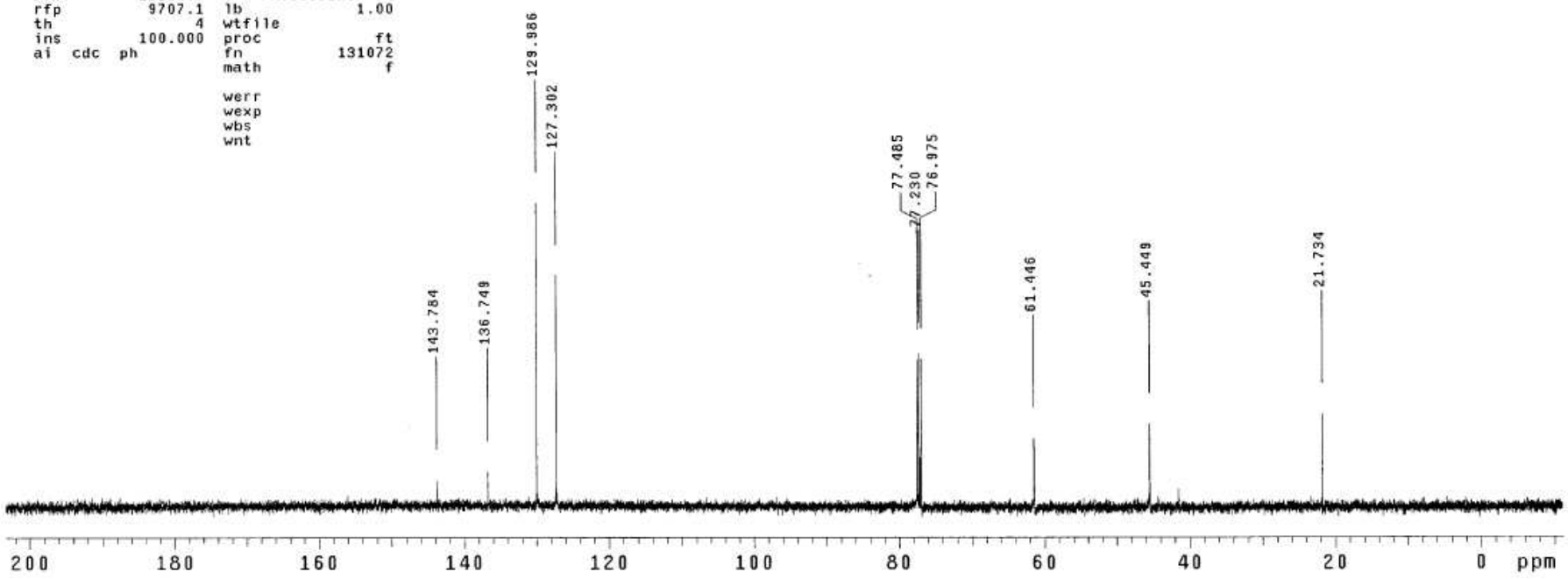
15c



TY2-362-B-c15-22

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 6 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	2000	dm2	n
ct	84	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	-1403.5	dof3	0
wp	26962.9	dm3	n
vs	207	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11111.0	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	

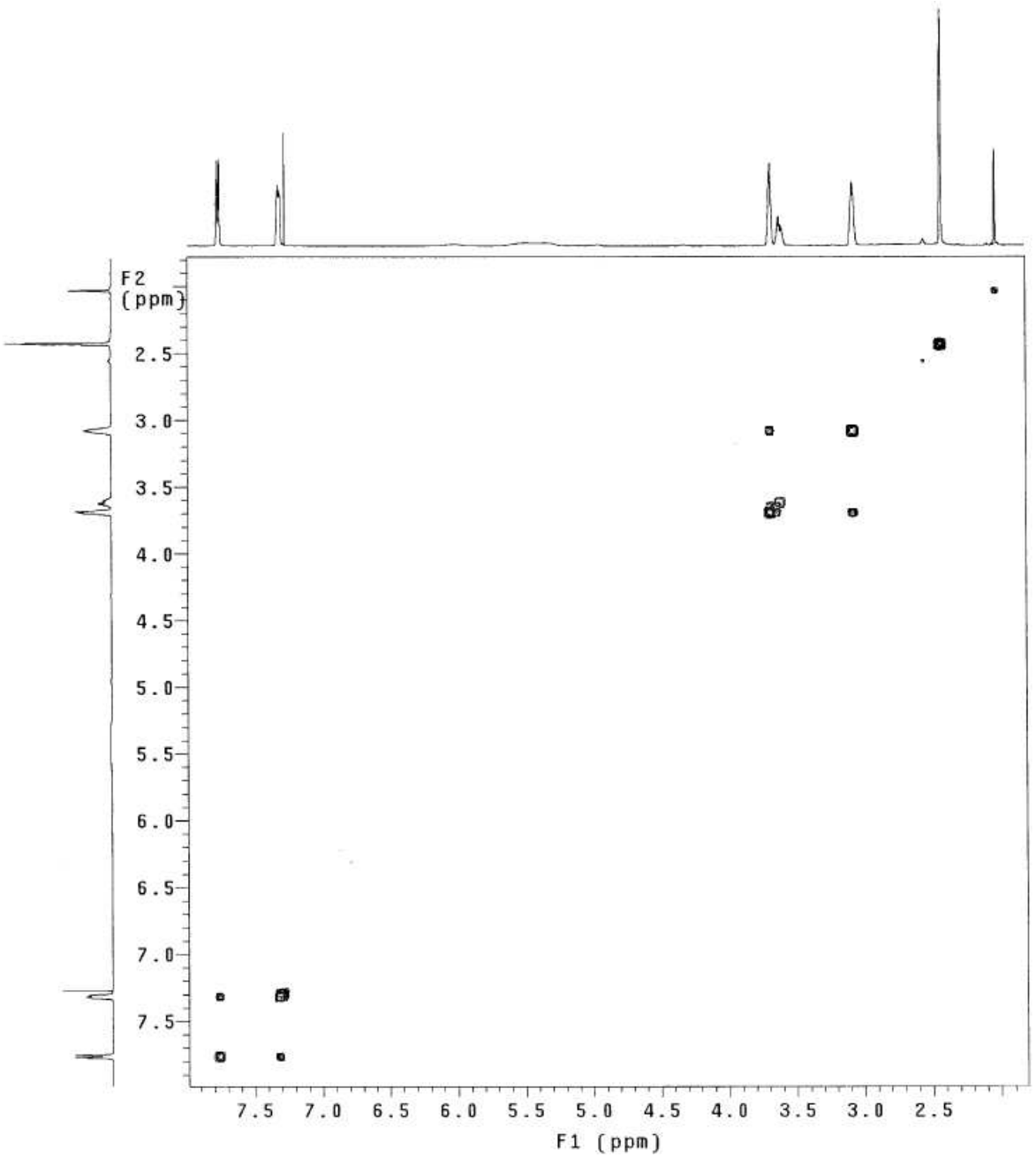


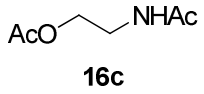
TY2-362-B-c15-22

Pulse Sequence: relayh

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

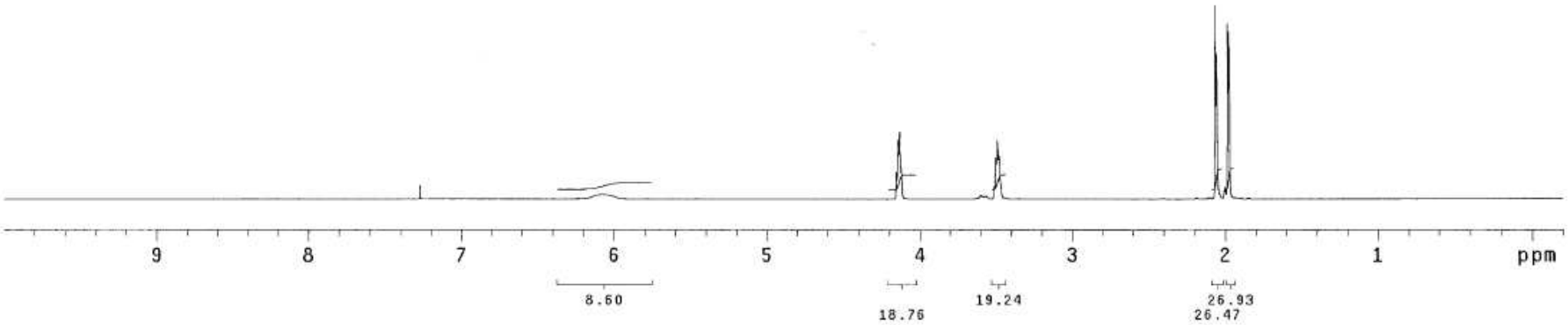
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611711 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec





16c

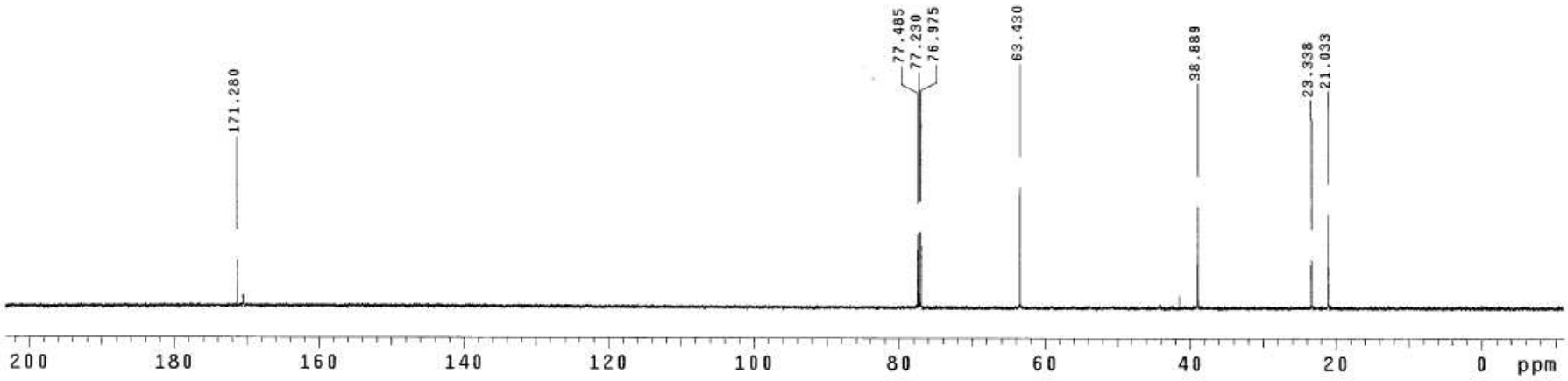
```
TY2-362-C-c24-28

expl  s2pul

      SAMPLE
date   Jun  6 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                4
ct                4
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -102.2
wp           5099.3
vs               16
sc                0
wc              250
hzmm          20.40
is            31.00
rfl          4139.7
rfp          3634.0
th              50
ins         100.000
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
```



TY2-362-C-c24-28

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 6 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	300	dm2	n
ct	257	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	-1407.2	dof3	0
wp	26962.9	dm3	n
vs	123	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11114.7	PROCESSING	
rfp	9707.1	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

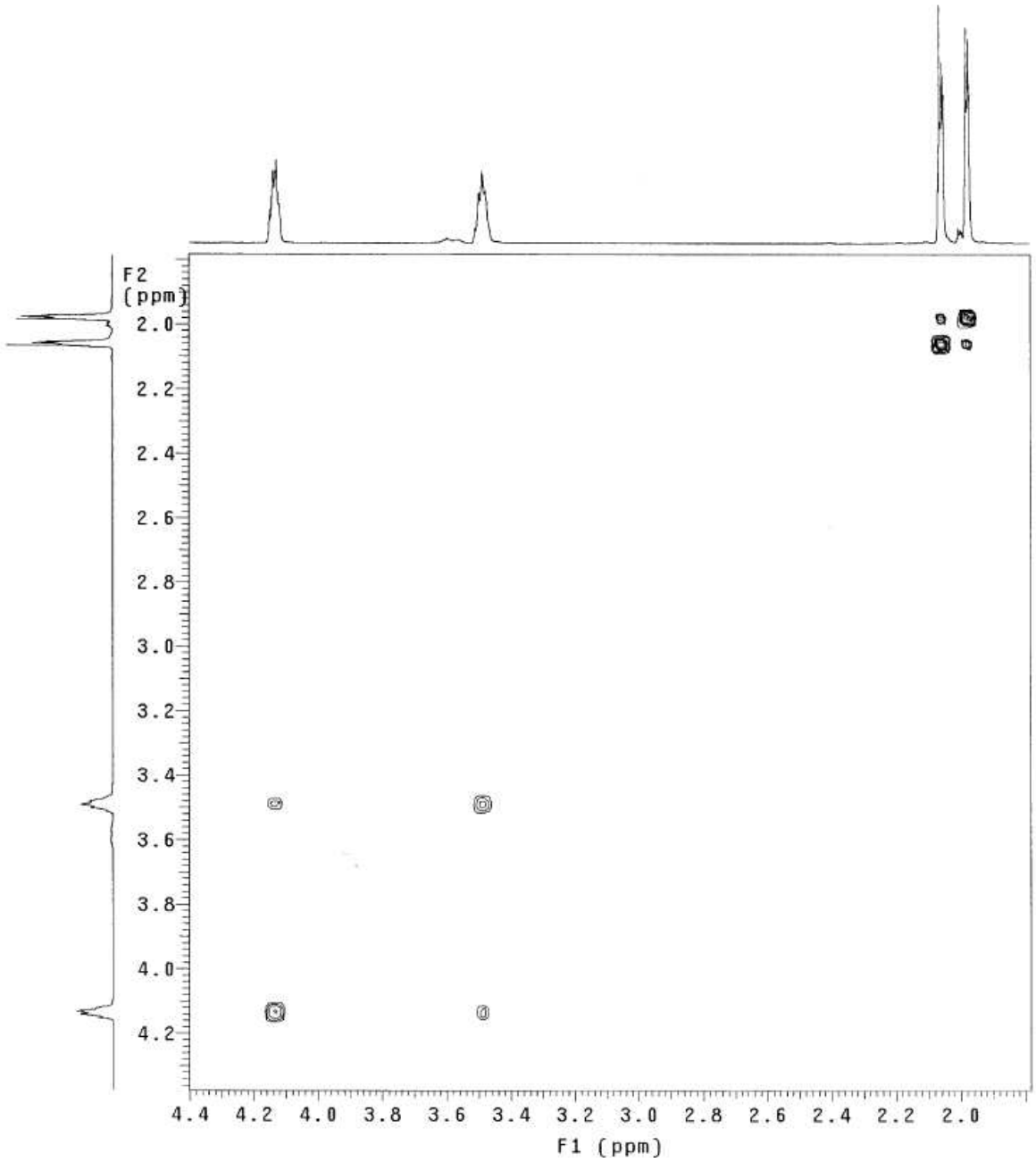


TY2-362-C-c24-28

Pulse Sequence: relayh

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

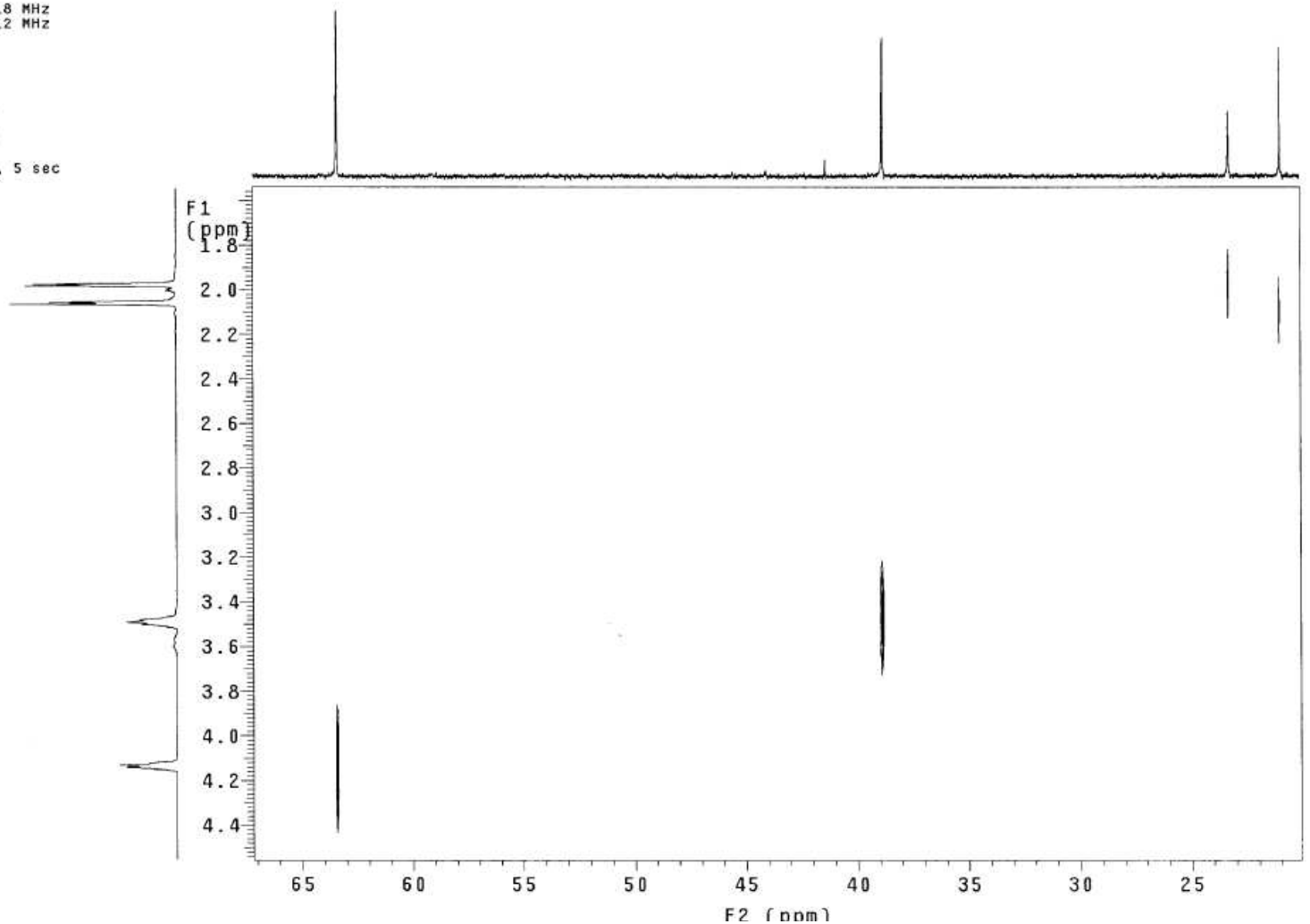
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611703 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

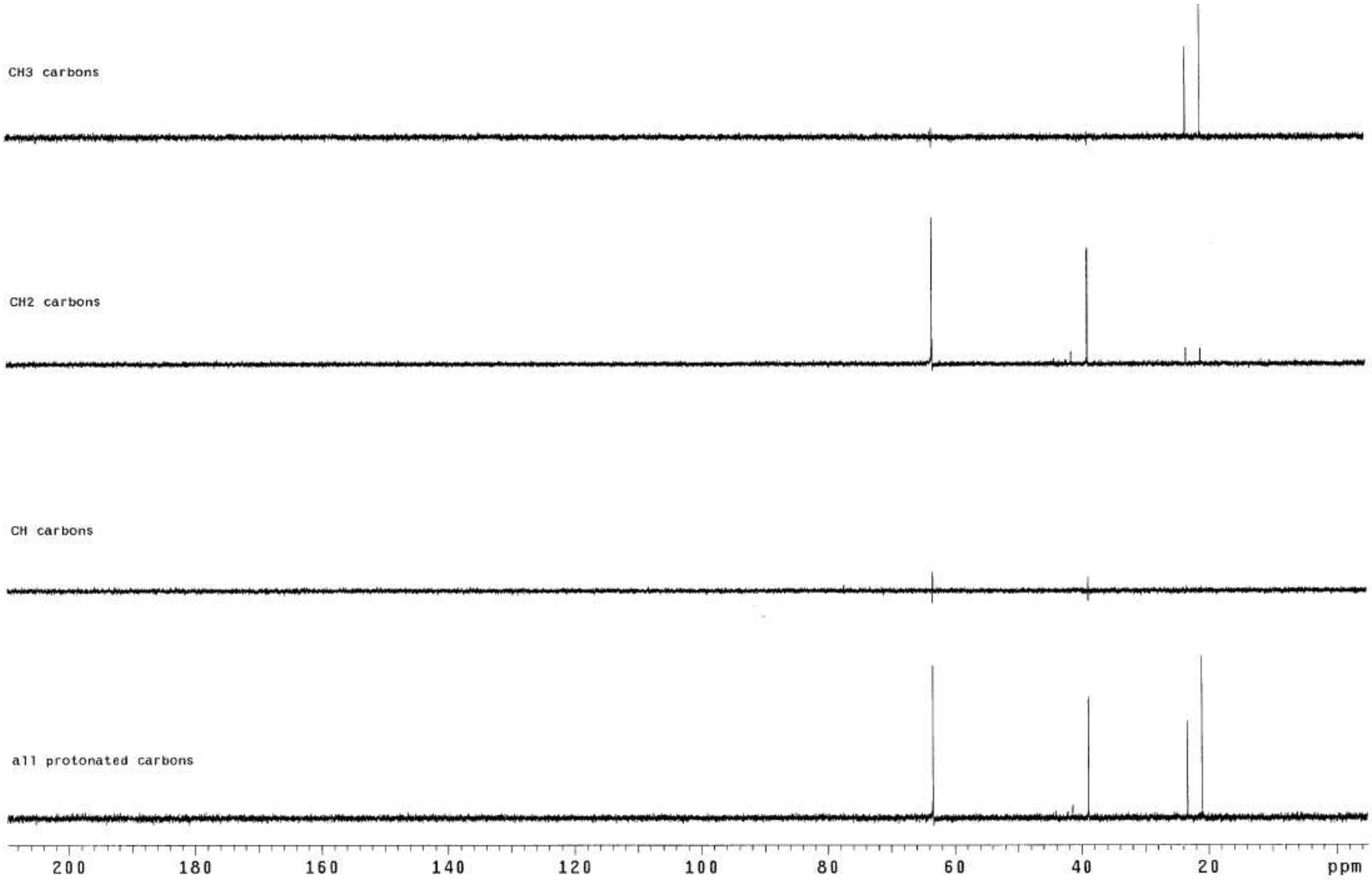


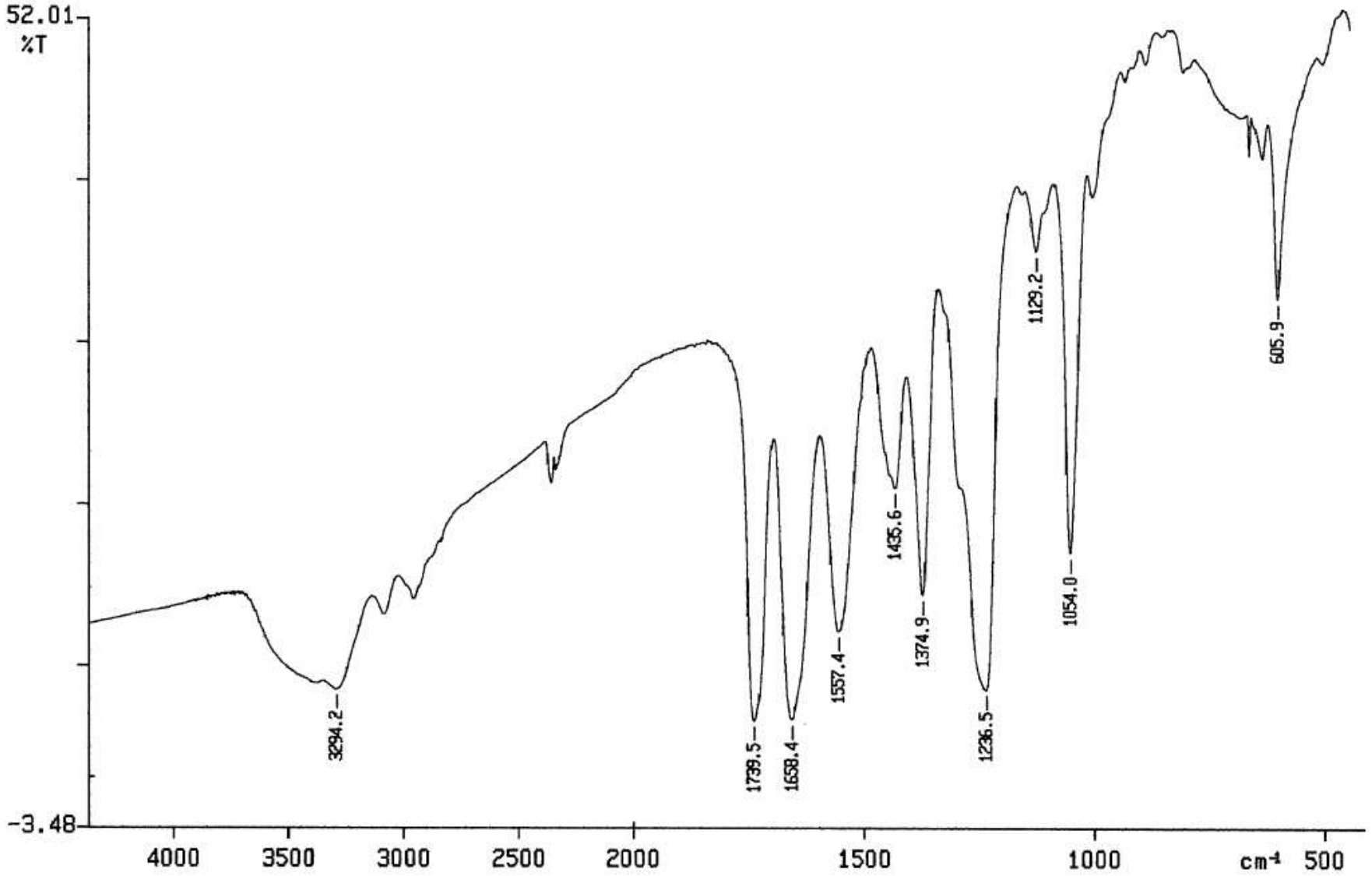
TY2-362-C-c24-28

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, 125.6901718 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



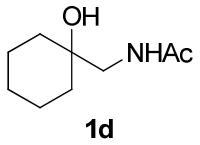

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


52.01
%T
-3.48
3294.2
1739.5
1658.4
1557.4
1435.6
1374.9
1236.5
1129.2
1054.0
605.9
4000
3500
3000
2500
2000
1500
1000
cm-1
500

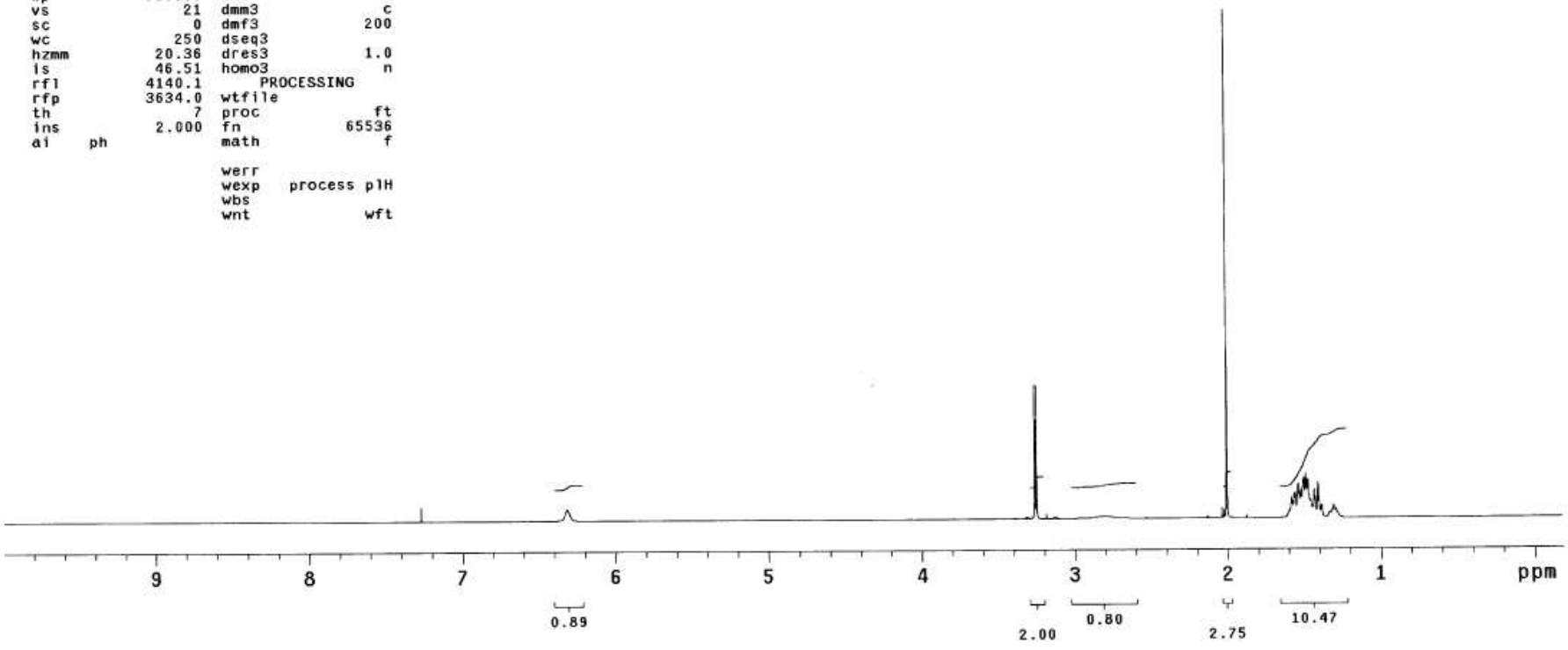
TY2-393

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 2 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	-94.0	dof3	0
wp	5090.8	dm3	n
vs	21	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.36	dres3	1.0
is	46.51	homo3	n
rfl	4140.1	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



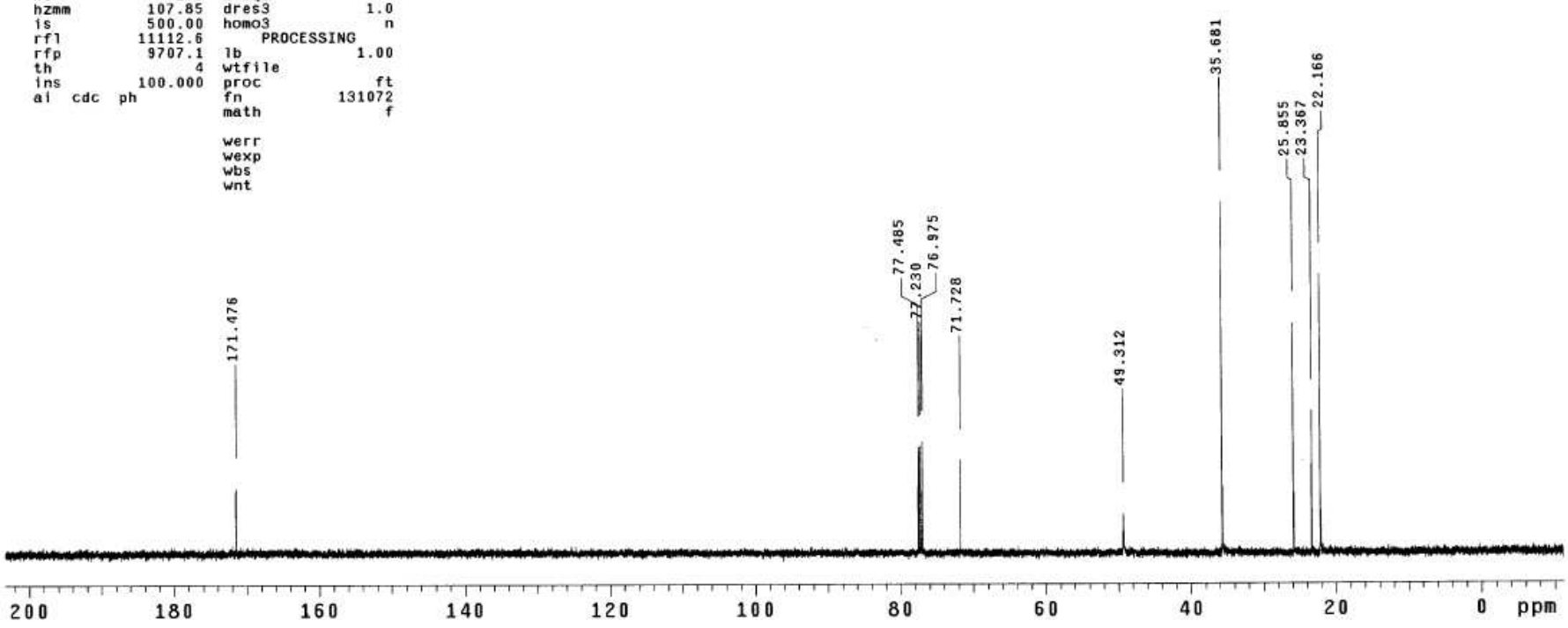
1d

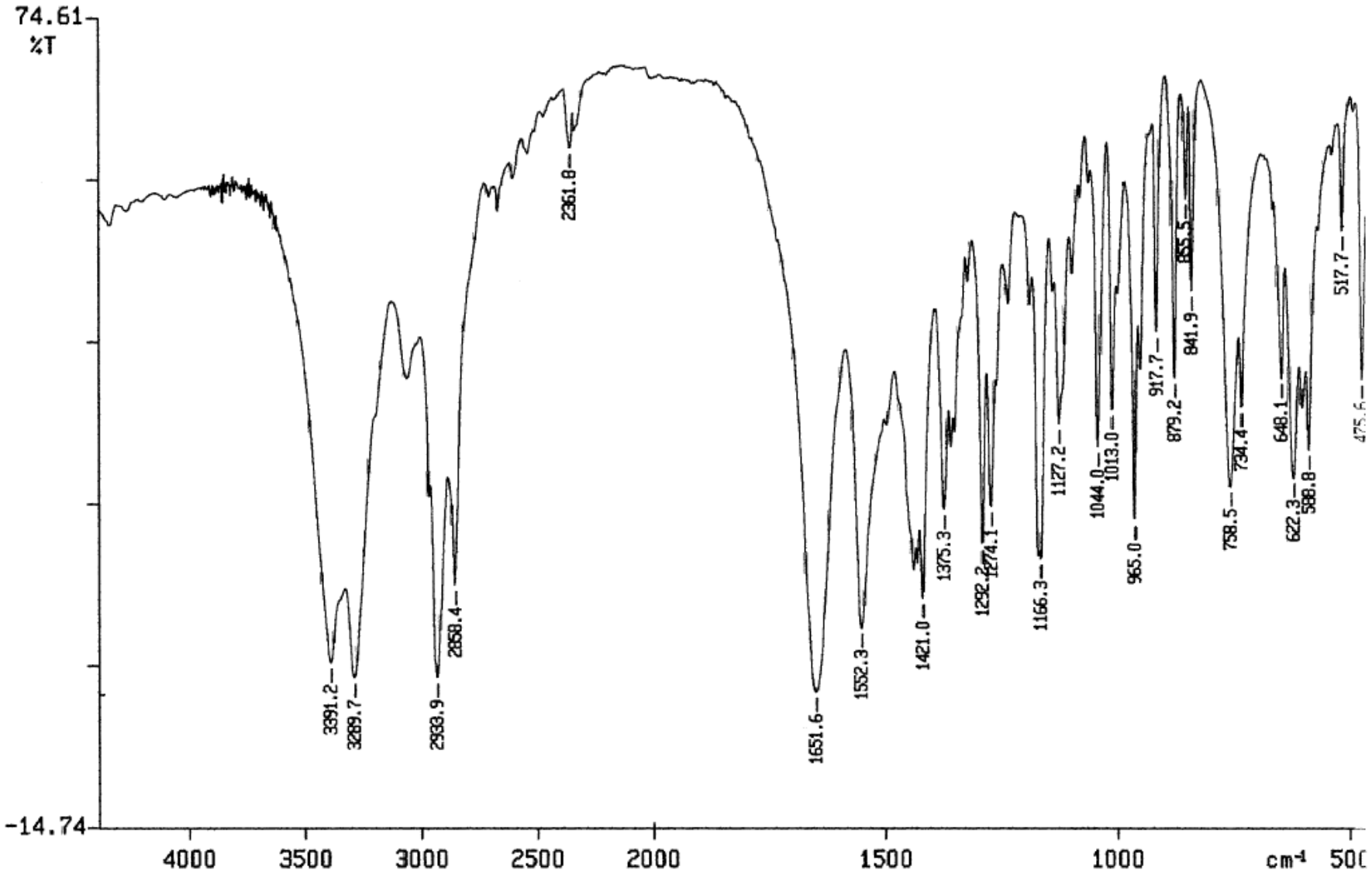


TY2-393

exp3 s2pul

SAMPLE		DEC. & VT	
date	Jul 2 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	1000	dm2	n
ct	52	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	-1405.2	dof3	0
wp	26962.9	dm3	n
vs	112	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11112.6	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	

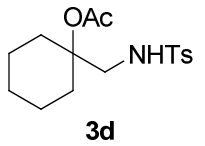



74.61
%T
-14.74
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3391.2
3289.7
2938.9
2858.4
2361.8
1651.6
1532.3
1421.0
1375.3
1292.2
1274.1
1166.3
1127.2
1044.0
1013.0
965.0
917.7
879.2
855.5
841.9
758.5
734.4
648.1
622.3
588.8
517.7

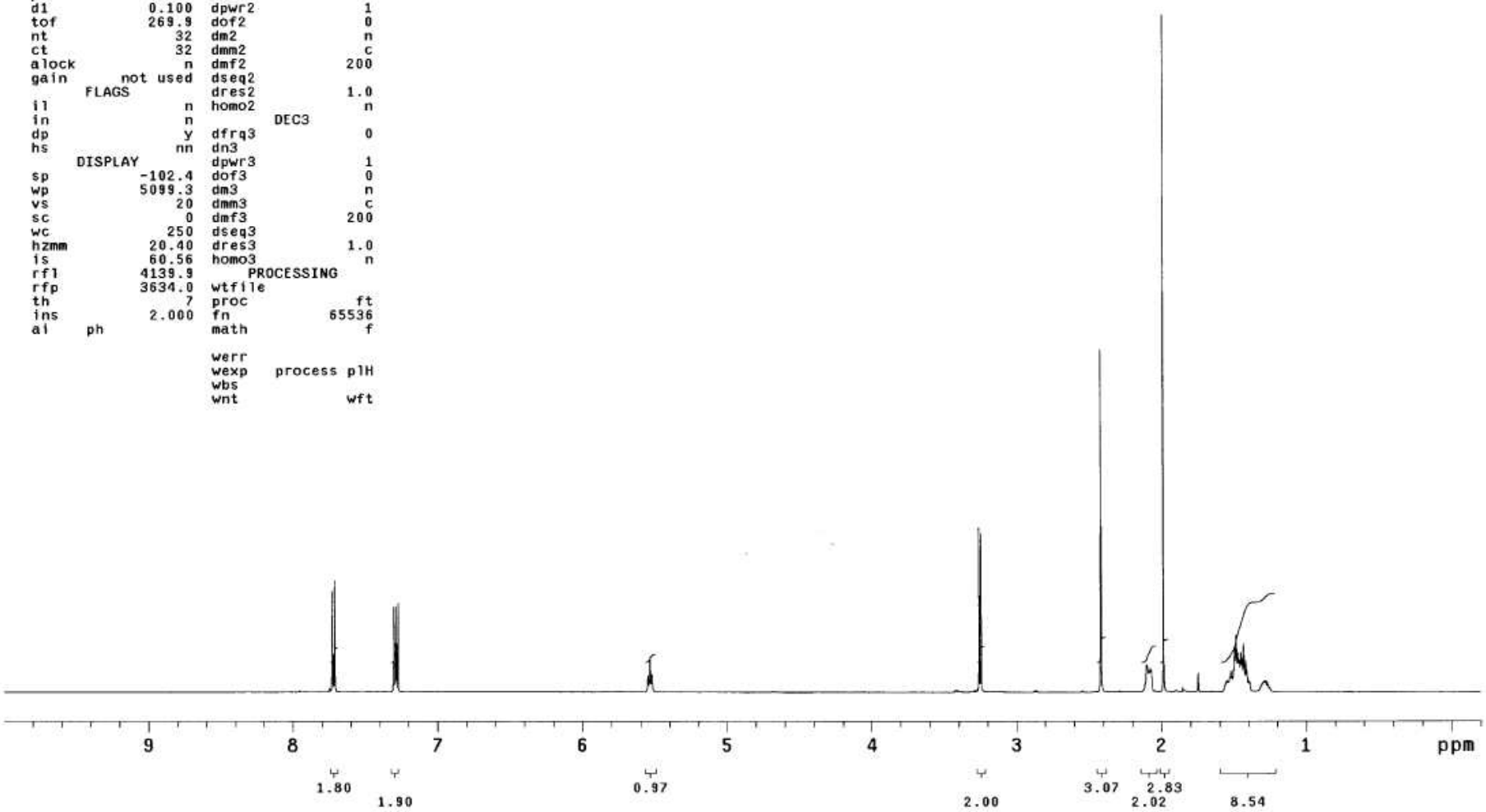
TY2-398-c2

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 7 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	-102.4	dof3	0
wp	5099.3	dm3	n
vs	20	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	60.56	homo3	n
rfl	4139.9	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



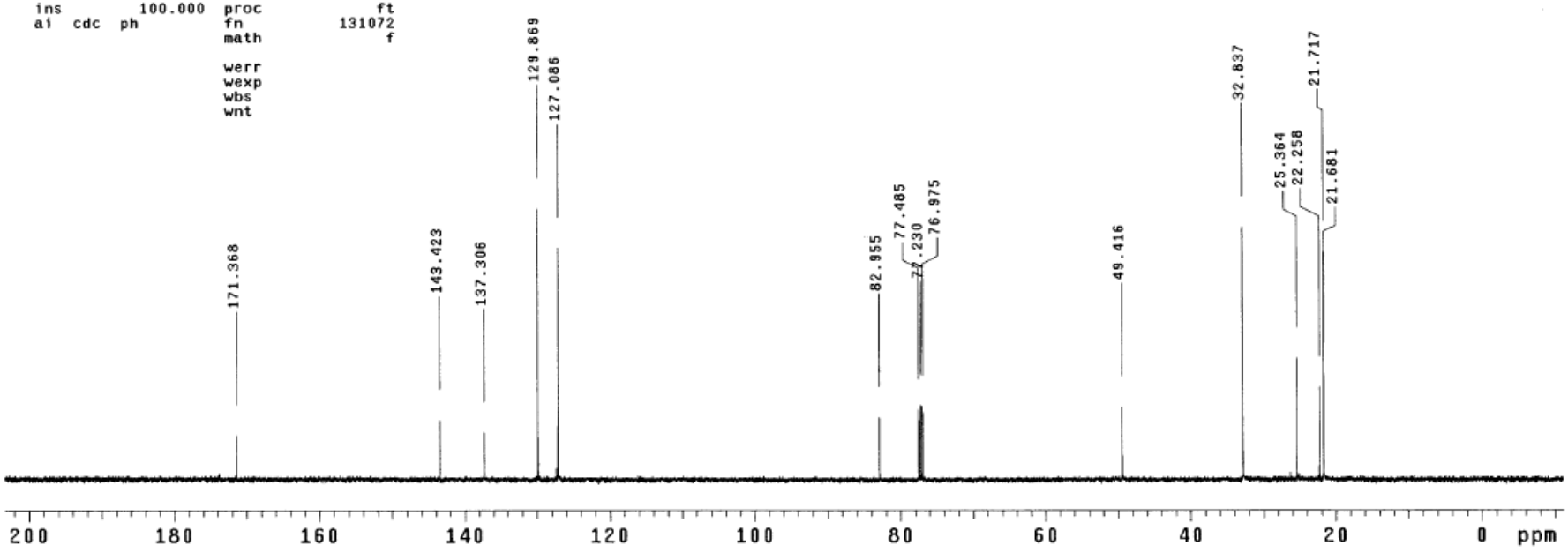
3d



TY2-398-c2

exp3 s2pul

SAMPLE		DEC. & VT	
date	Jul 7 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	1000	dm2	n
ct	60	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	70	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	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

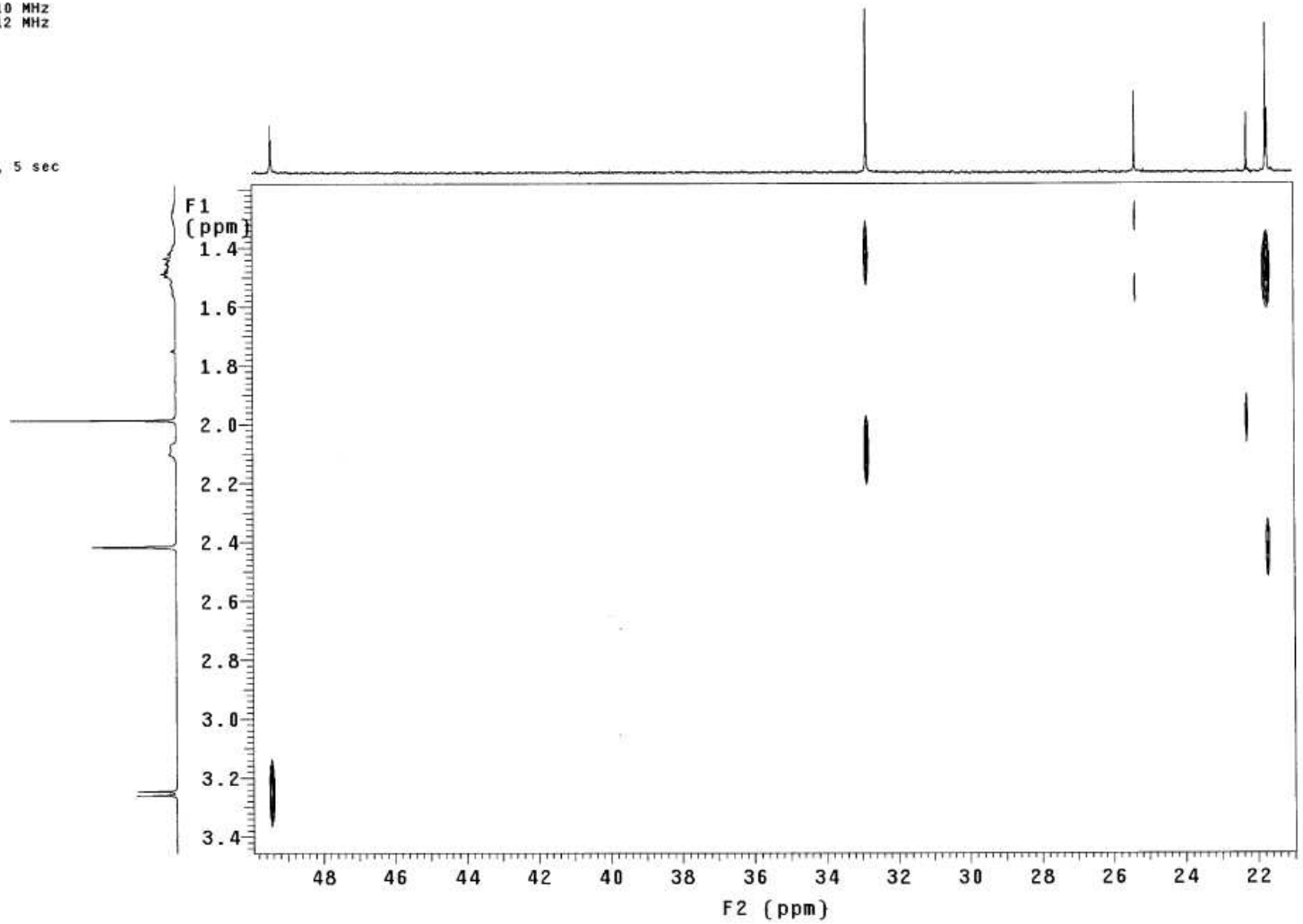


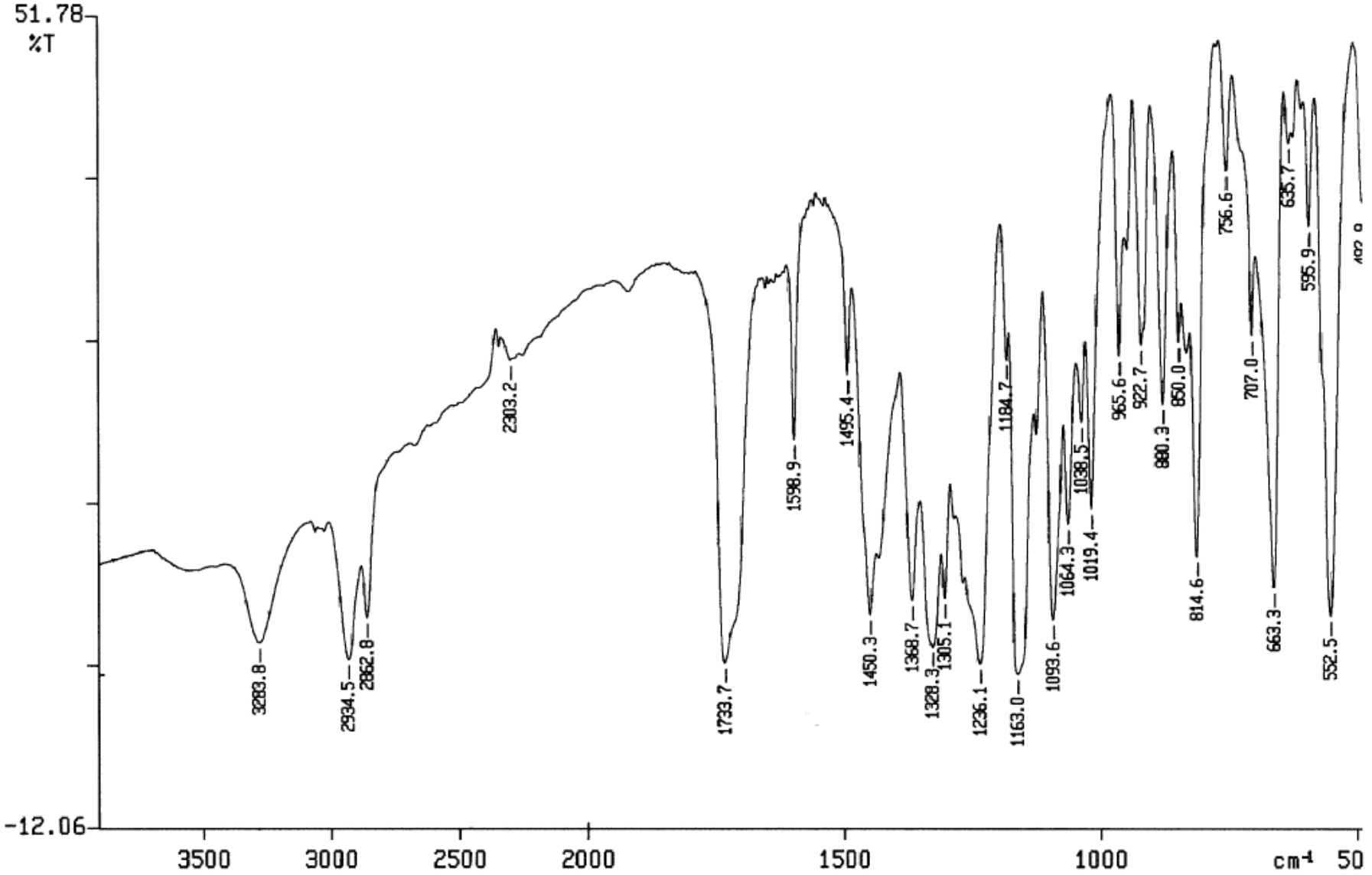
TY2-398-c2

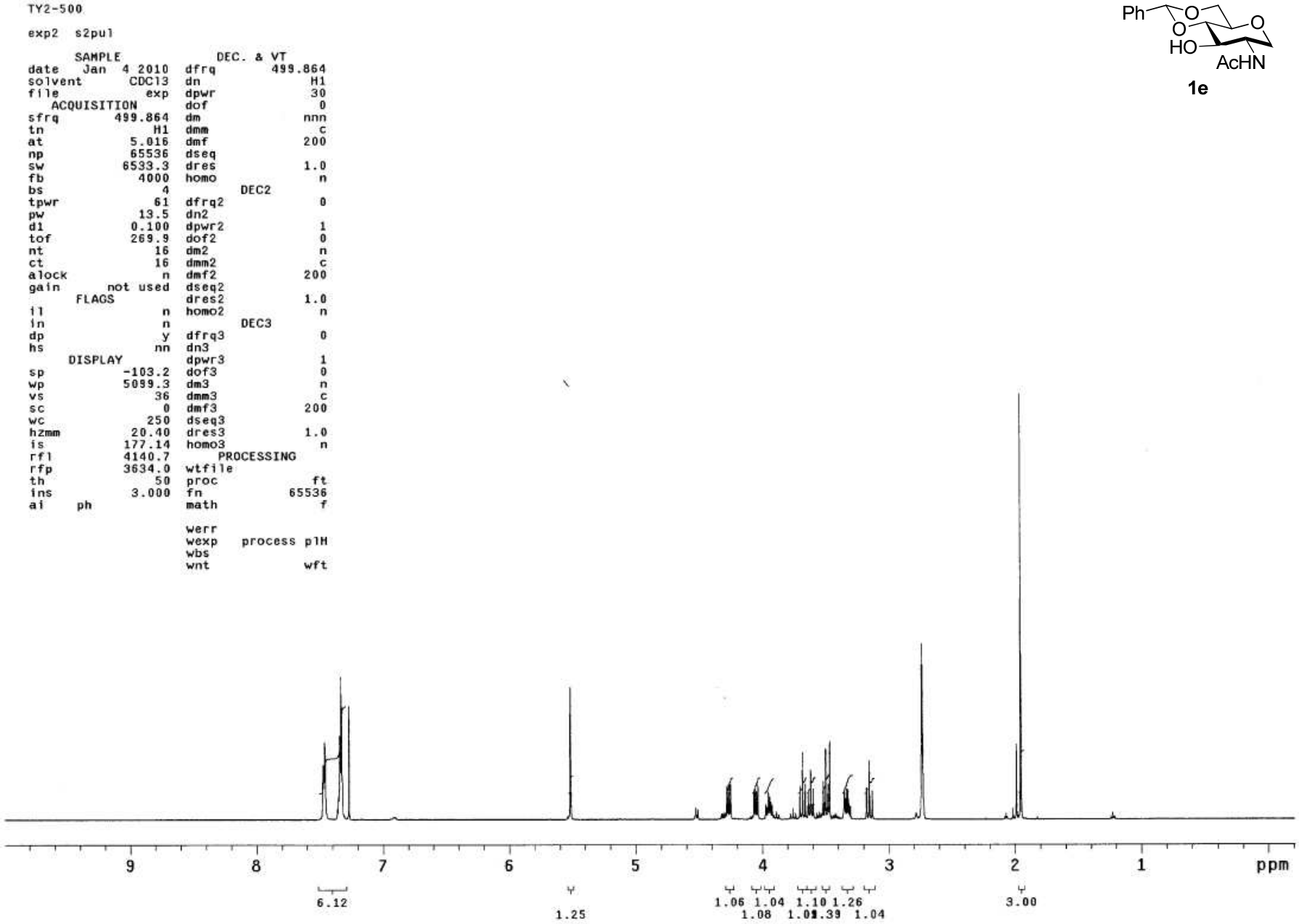
Pulse Sequence: hetcor

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, 125.6901710 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



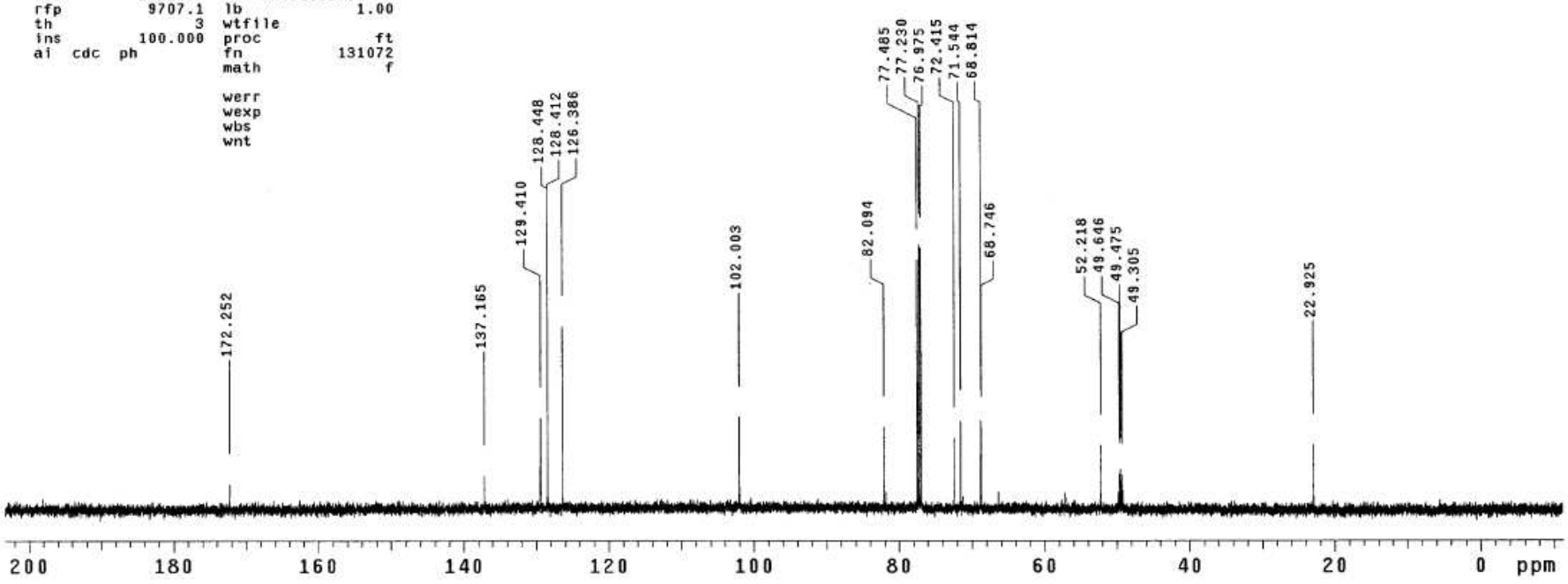

51.78
%T
-12.06
3283.8
2934.5
2862.8
2303.2
1733.7
1598.9
1495.4
1450.3
1368.7
1328.3
1305.1
1236.1
1184.7
1163.0
1093.6
1064.3
1038.5
1019.4
965.6
922.7
880.3
850.0
814.6
756.6
707.0
663.3
635.7
595.9
552.5
3500
3000
2500
2000
1500
1000
cm-1
50


TY2-500
exp2 s2pul
SAMPLE
date Jan 4 2010
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 -103.2
wp 5099.3
vs 36
sc 0
wc 250
hzmm 20.40
is 177.14
rfl 4140.7
rfp 3634.0
th 50
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 p1H
wbs
wnt wft
Ph
O
O
O
HO
AcHN
1e
9
8
7
6
5
4
3
2
1
ppm
6.12
1.25
1.06
1.04
1.10
1.26
1.08
1.01
1.39
1.04
3.00

TY2-500

exp3 s2pul

SAMPLE		DEC. & VT	
date	Jan 4 2010	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	182	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	-1407.2	dof3	0
wp	26962.9	dm3	n
vs	313	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11114.7	PROCESSING	
rfp	9707.1	lb	1.00
th	3	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

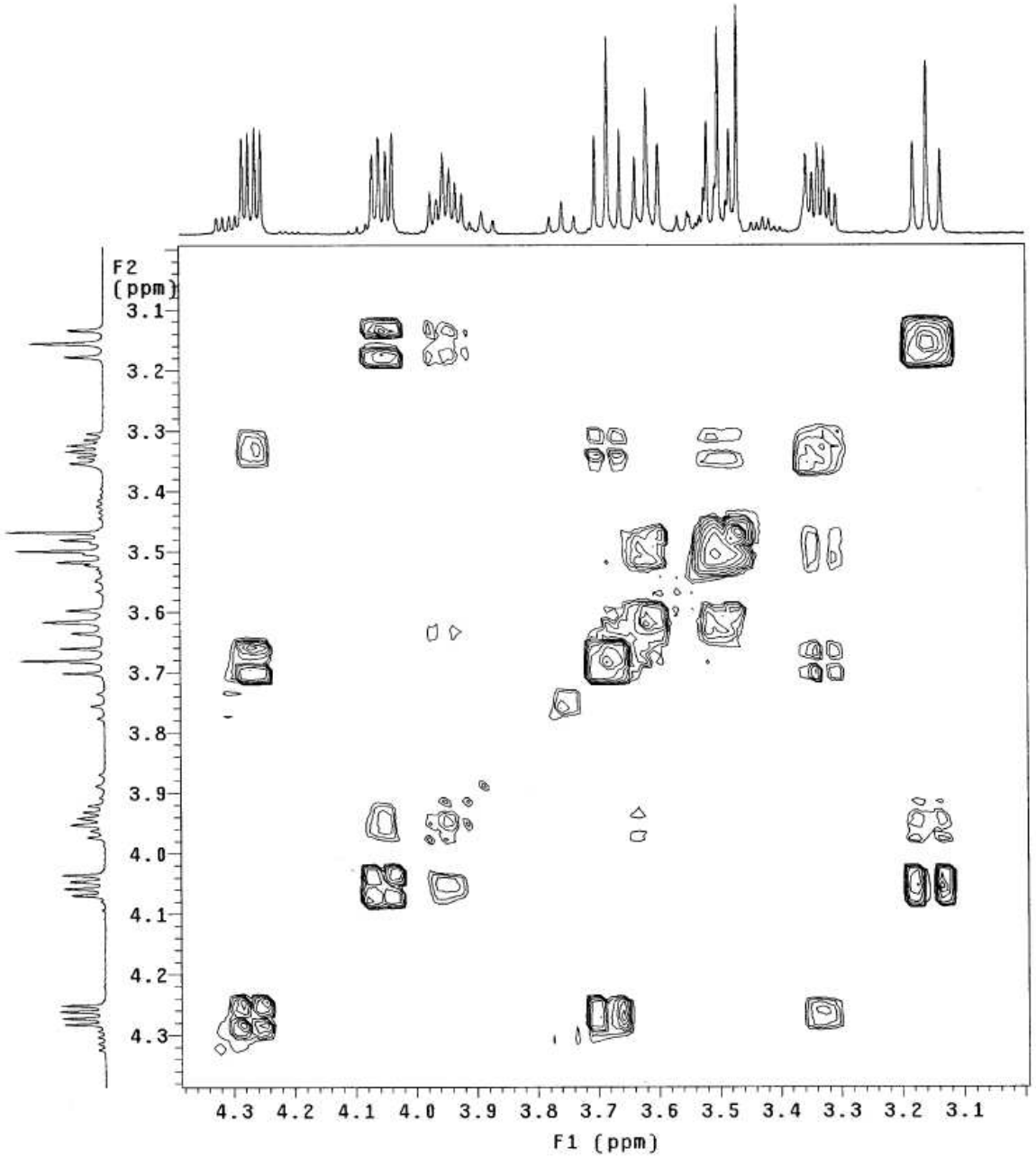


TY2-500

Pulse Sequence: relayh

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

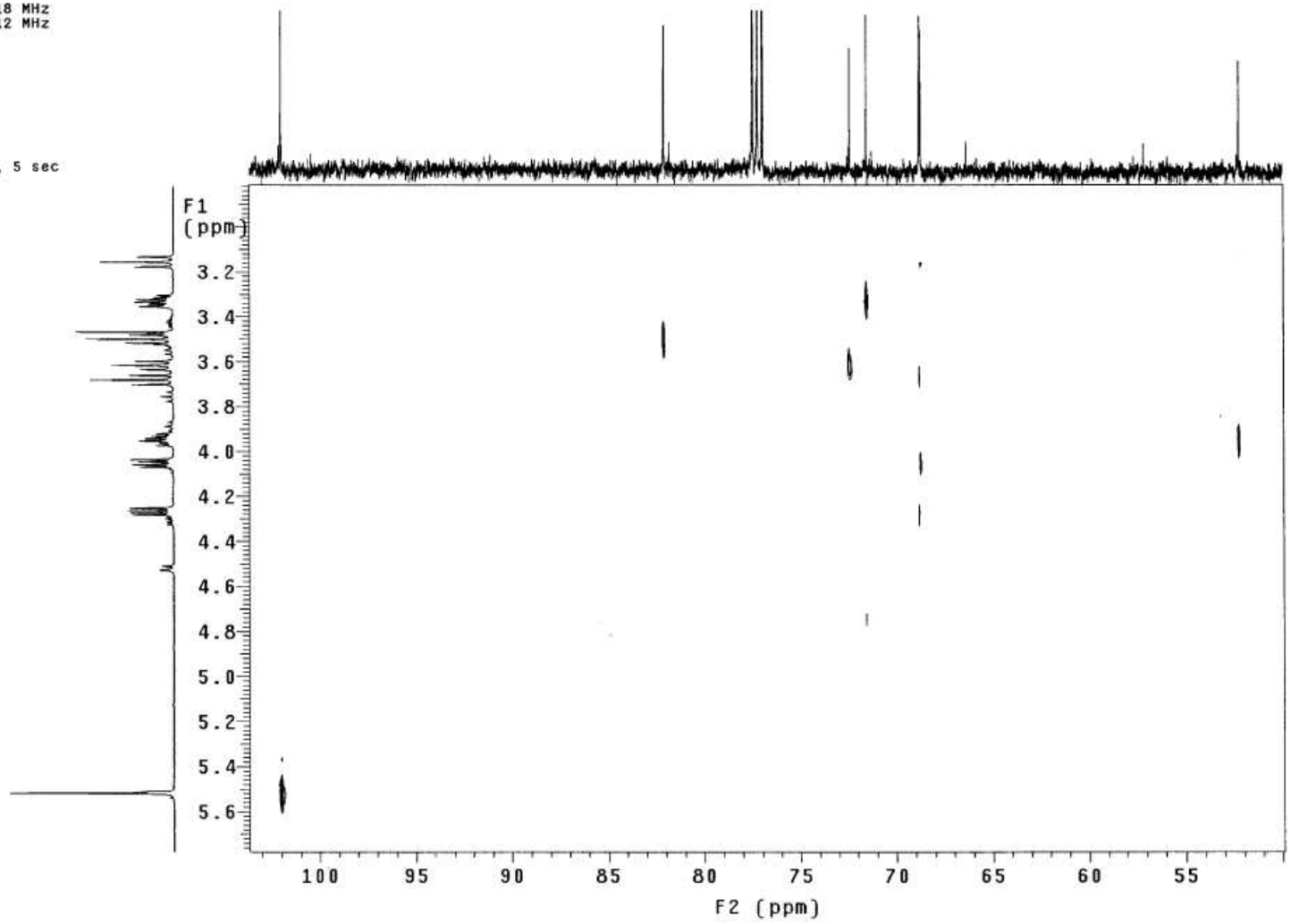
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
32 repetitions
256 increments
OBSERVE H1, 499.8611713 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 3 hr, 23 min, 10 sec

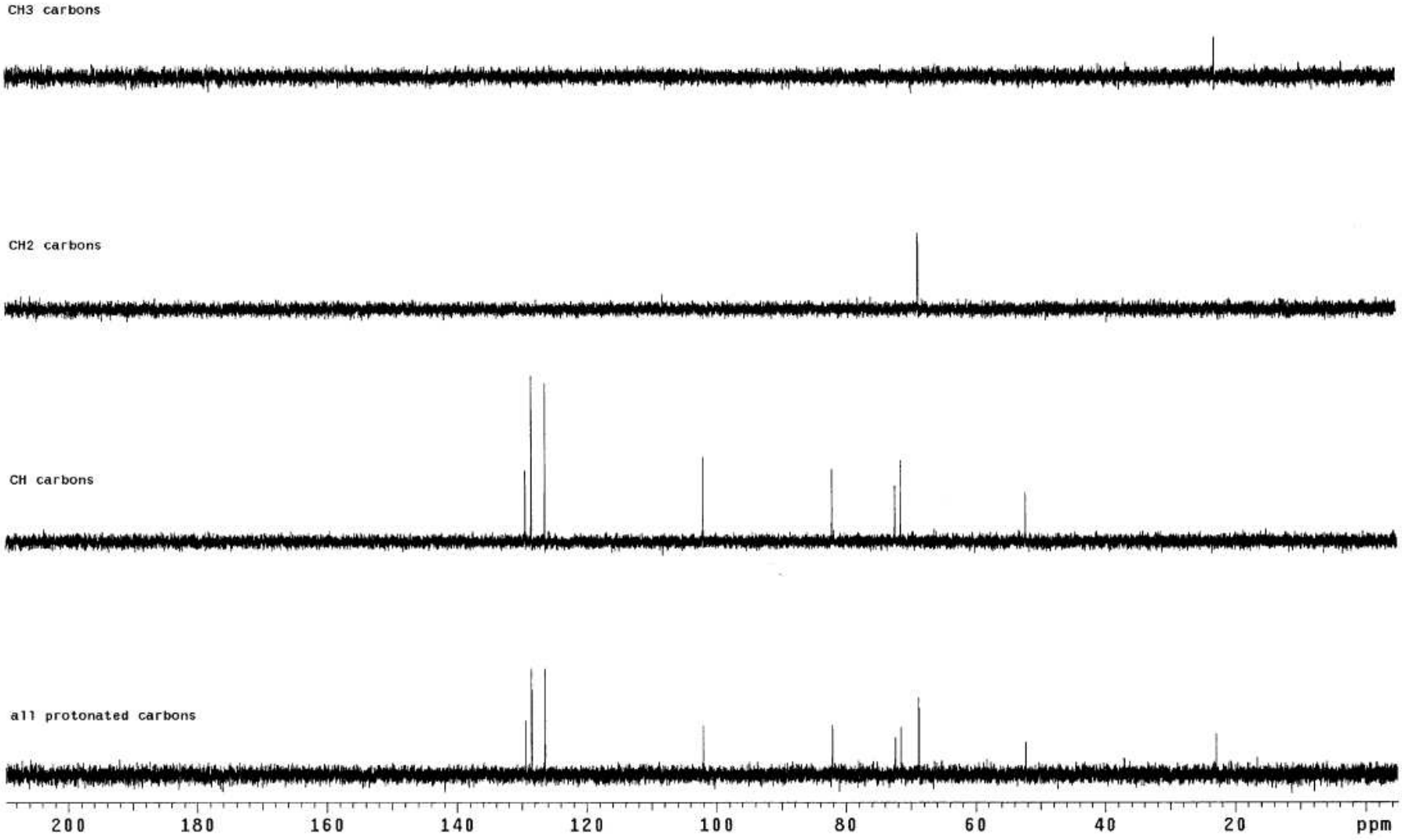


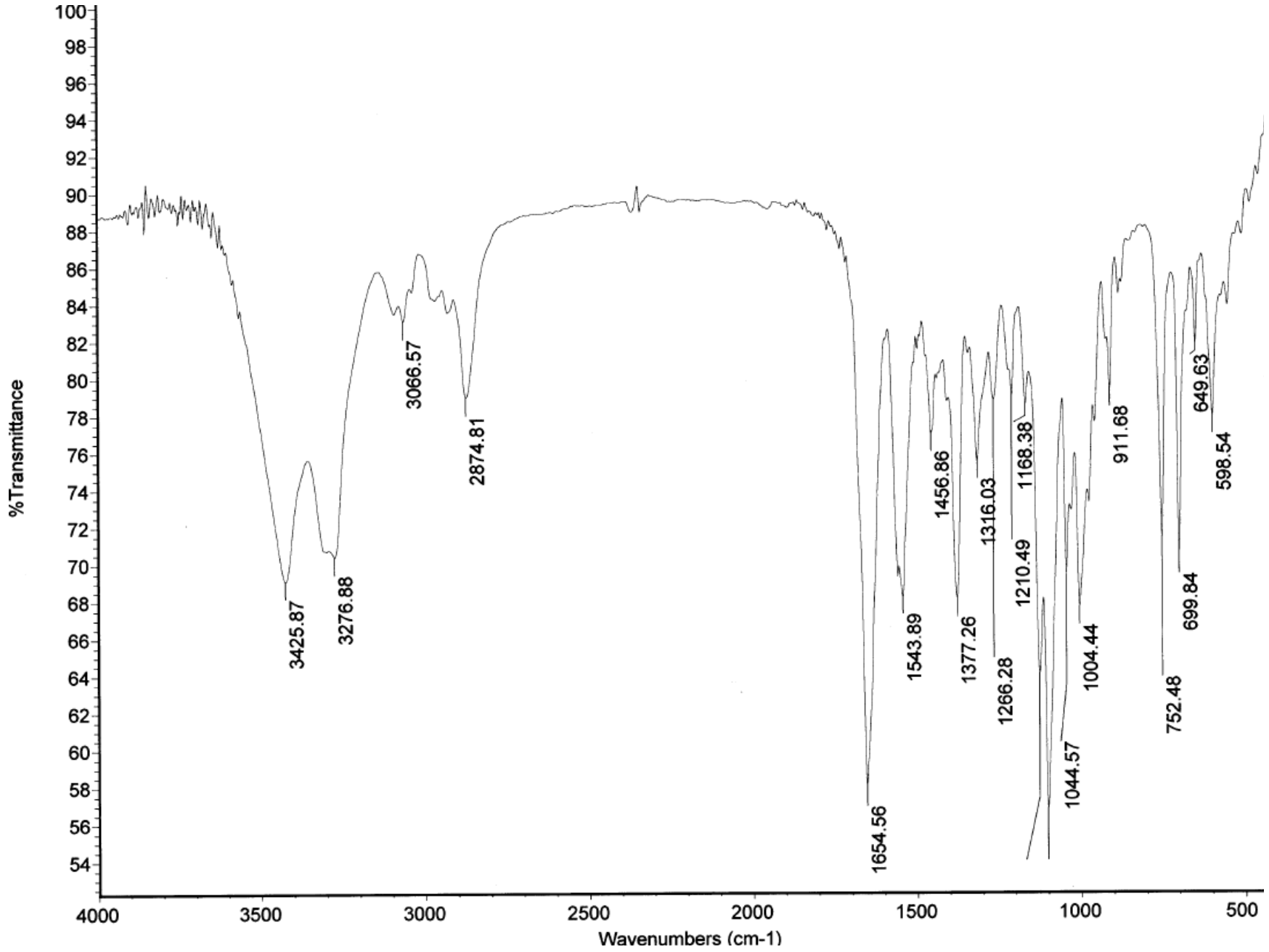
TY2-500

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, 125.6901718 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



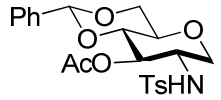

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


%Transmittance
Wavenumbers (cm-1)
3425.87
3276.88
3066.57
2874.81
1654.56
1543.89
1456.86
1377.26
1316.03
1266.28
1210.49
1168.38
1044.57
1004.44
911.68
752.48
699.84
649.63
598.54

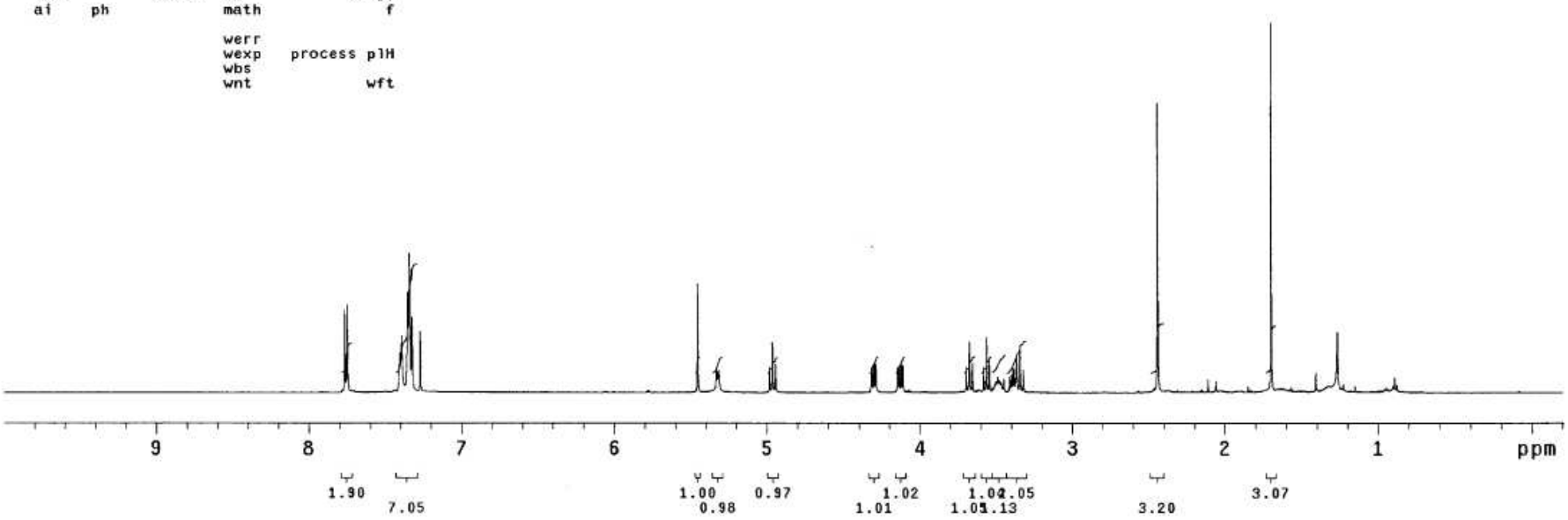
TY2-501

exp2 s2pul

SAMPLE		DEC. & VT	
date	Dec 23 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	22	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	127.97	homo3	n
rfl	4140.3	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



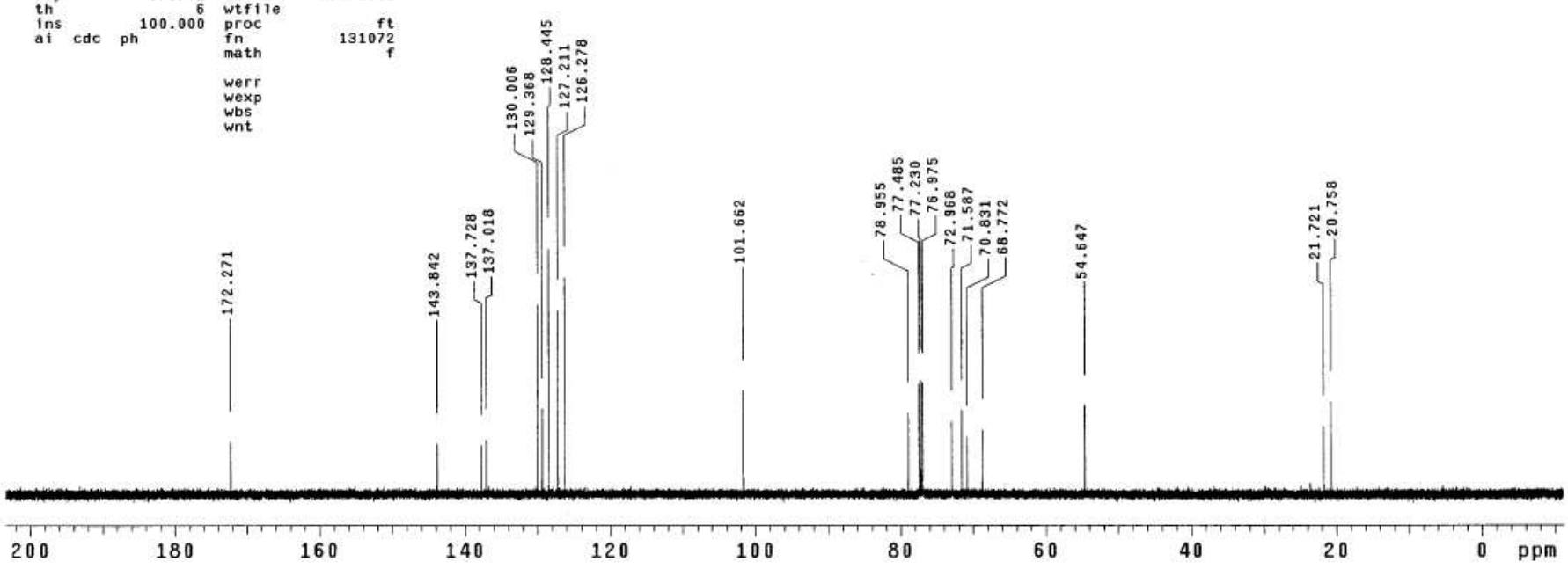
3e



TY2-501

exp3 s2pul

SAMPLE		DEC. & VT	
date	Dec 23 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	2000	dm2	n
ct	164	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	-1403.5	dof3	0
wp	26962.9	dm3	n
vs	85	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11111.0	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	

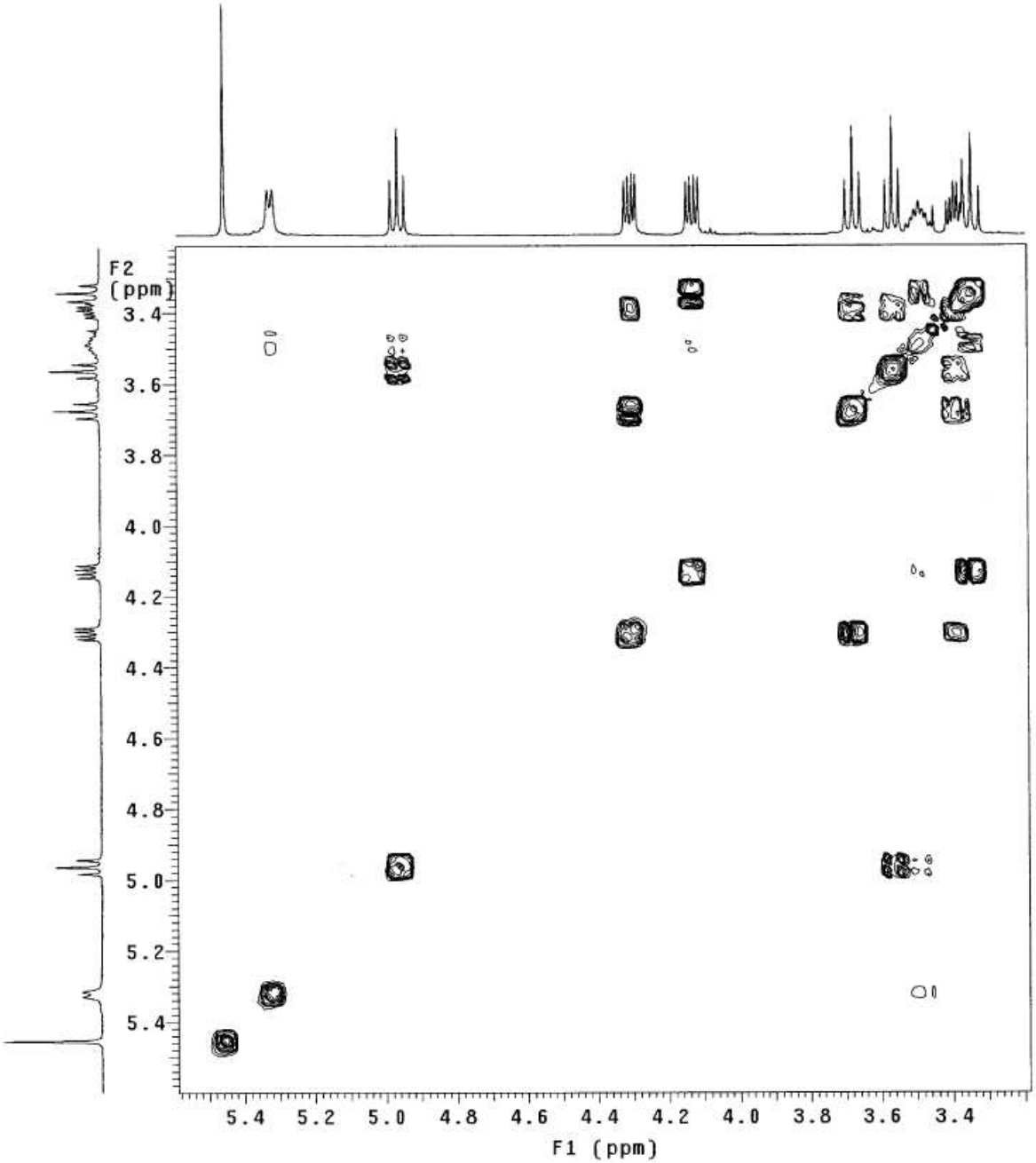


TY2-501

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

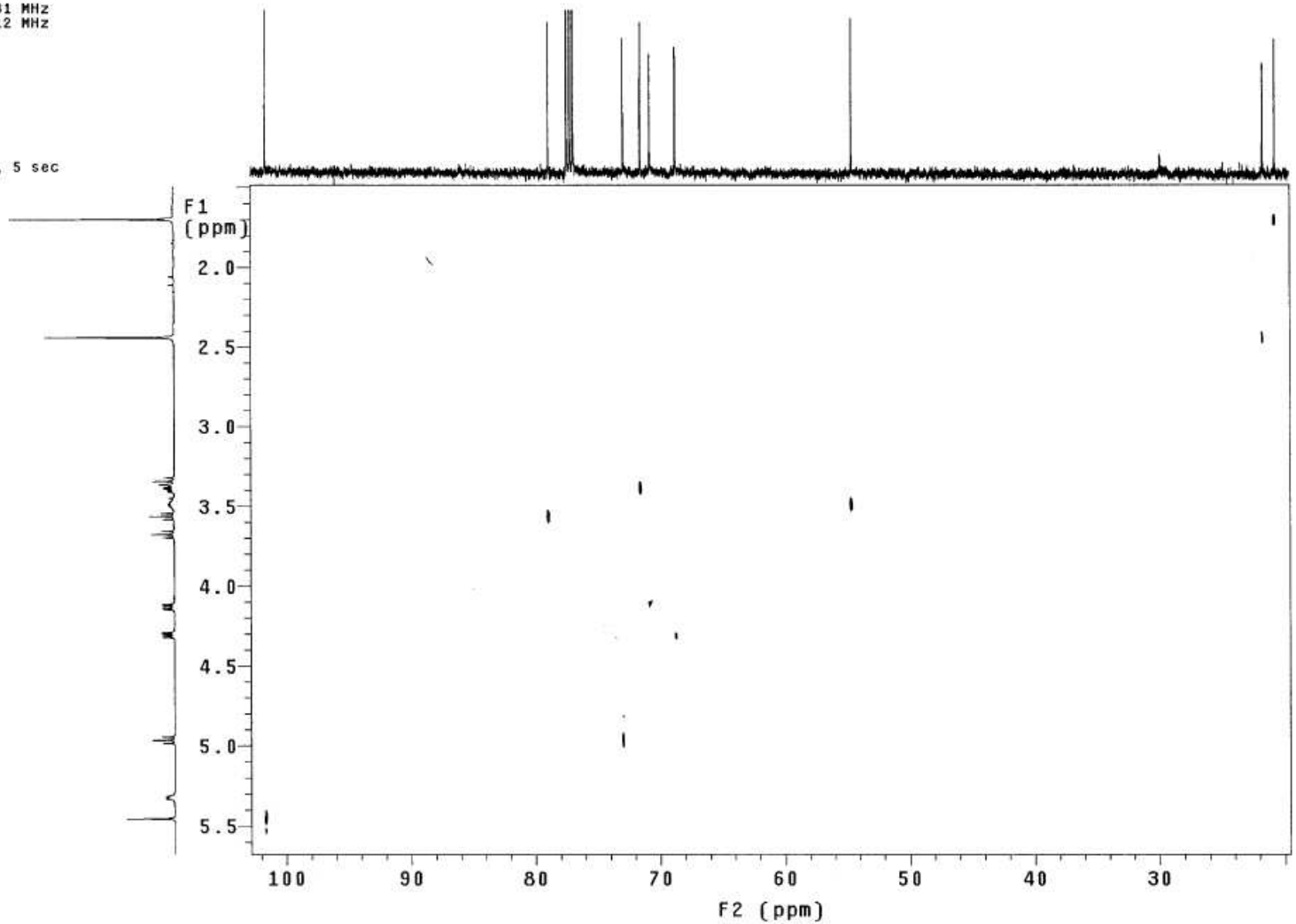


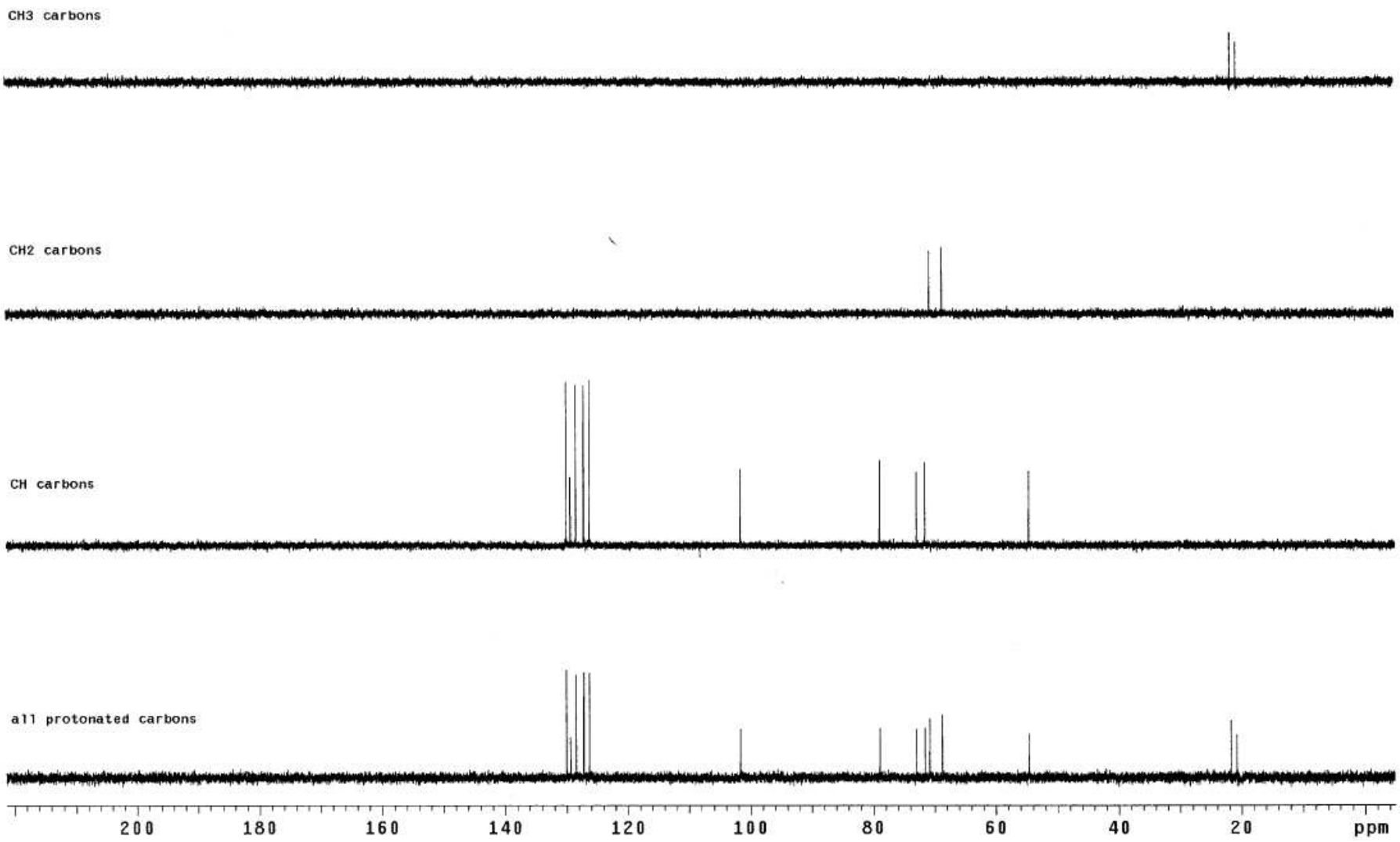
TY2-501

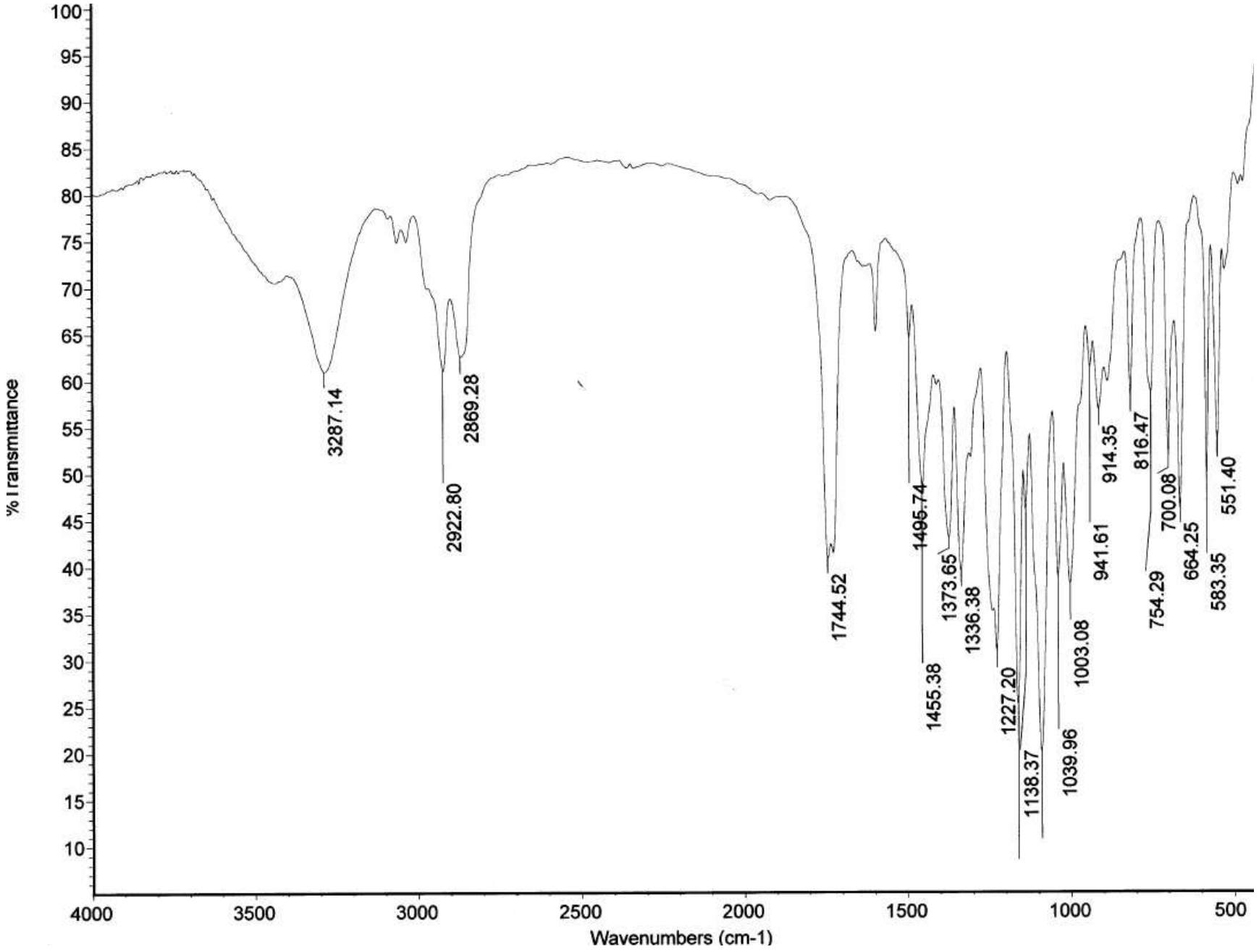
Pulse Sequence: hetcor

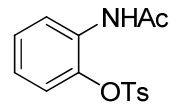
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

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, 125.6901681 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



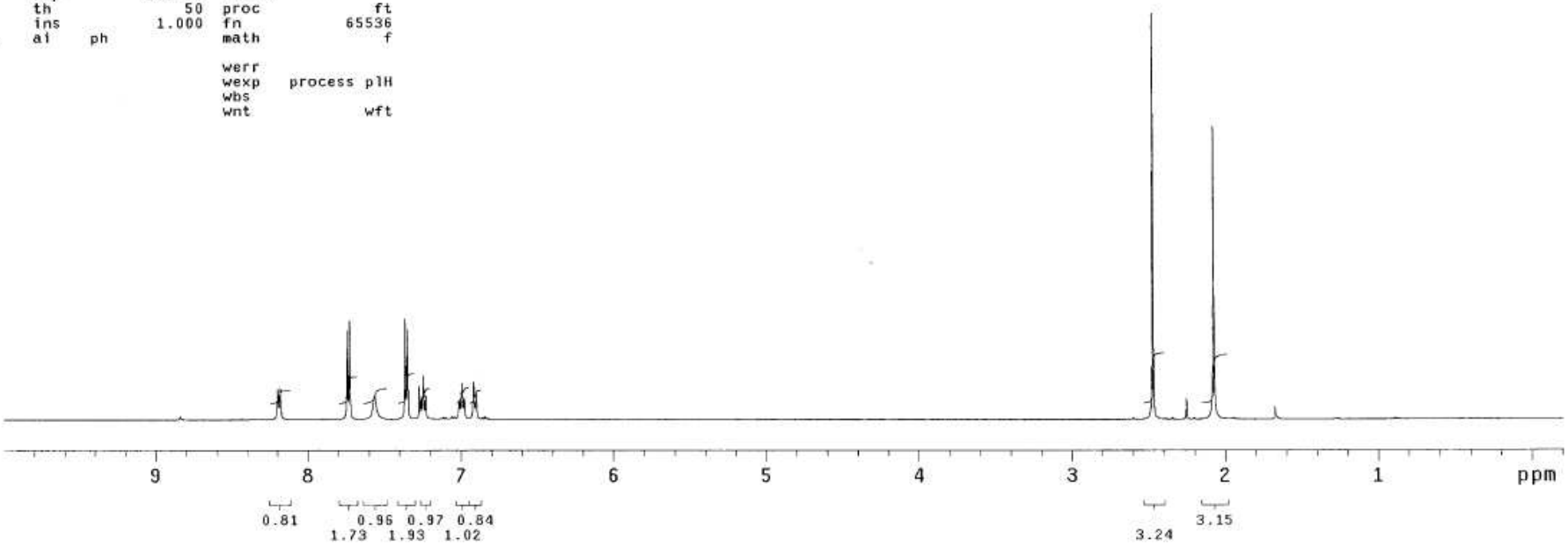

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

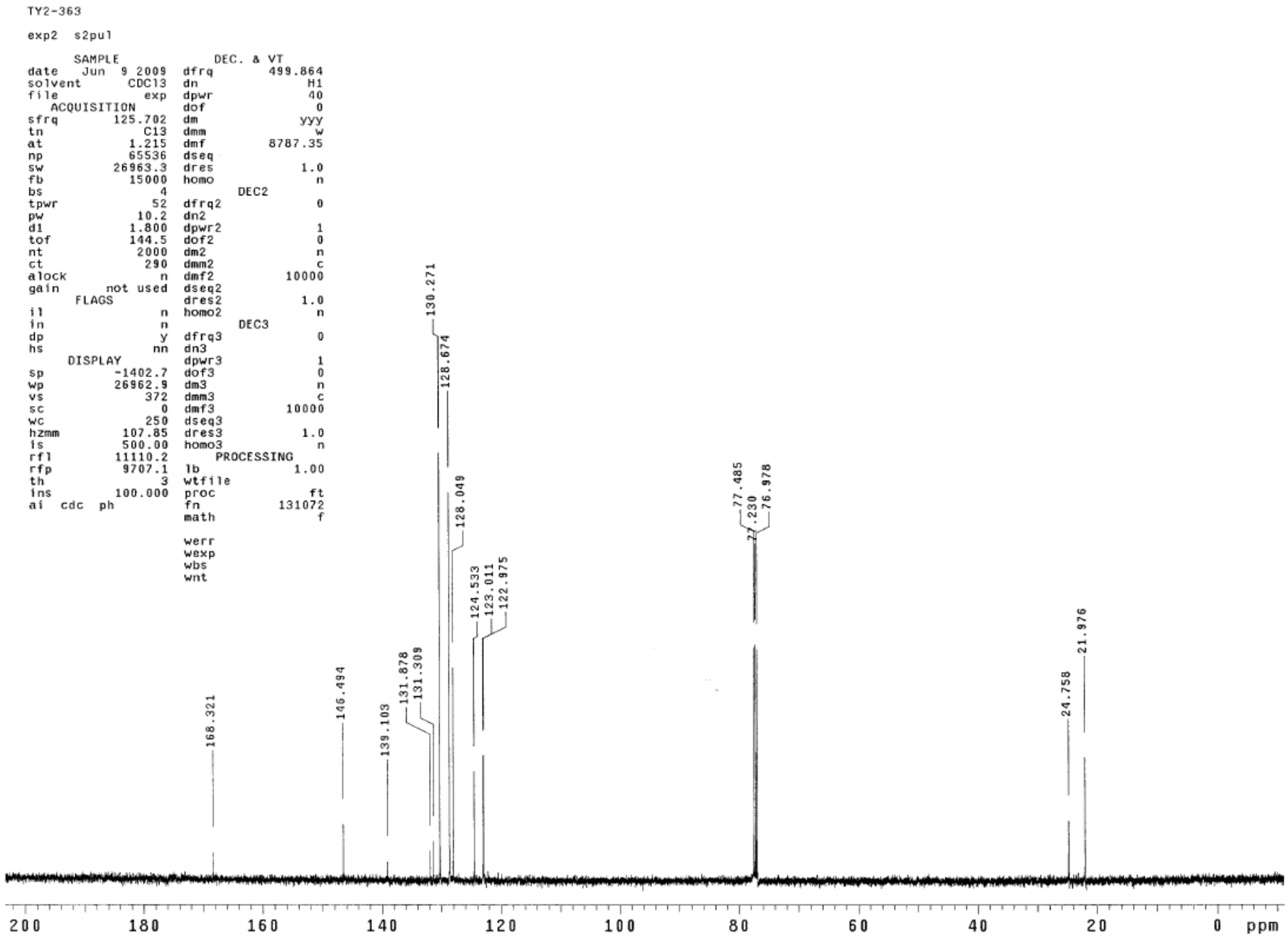

% Transmittance
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3287.14
2922.80
2869.28
1744.52
1496.74
1455.38
1373.65
1336.38
1227.20
1138.37
1039.96
1003.08
941.61
914.35
816.47
754.29
700.08
664.25
583.35
551.40



2f

```
TY2-363

exp1  s2pul

      SAMPLE          DEC. & VT
date   Jun  9 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             34   dmm3              c
sc              0   dmf3            200
wc            250   dseq3
hzmm        20.40   dres3           1.0
is          123.23  homo3             n
rfl         4140.1       PROCESSING
rfp         3634.0  wtfile
th             50   proc             ft
ins         1.000   fn            65536
ai    ph            math              f

                   werr
                   wexp   process plH
                   wbs
                   wnt             wft
```



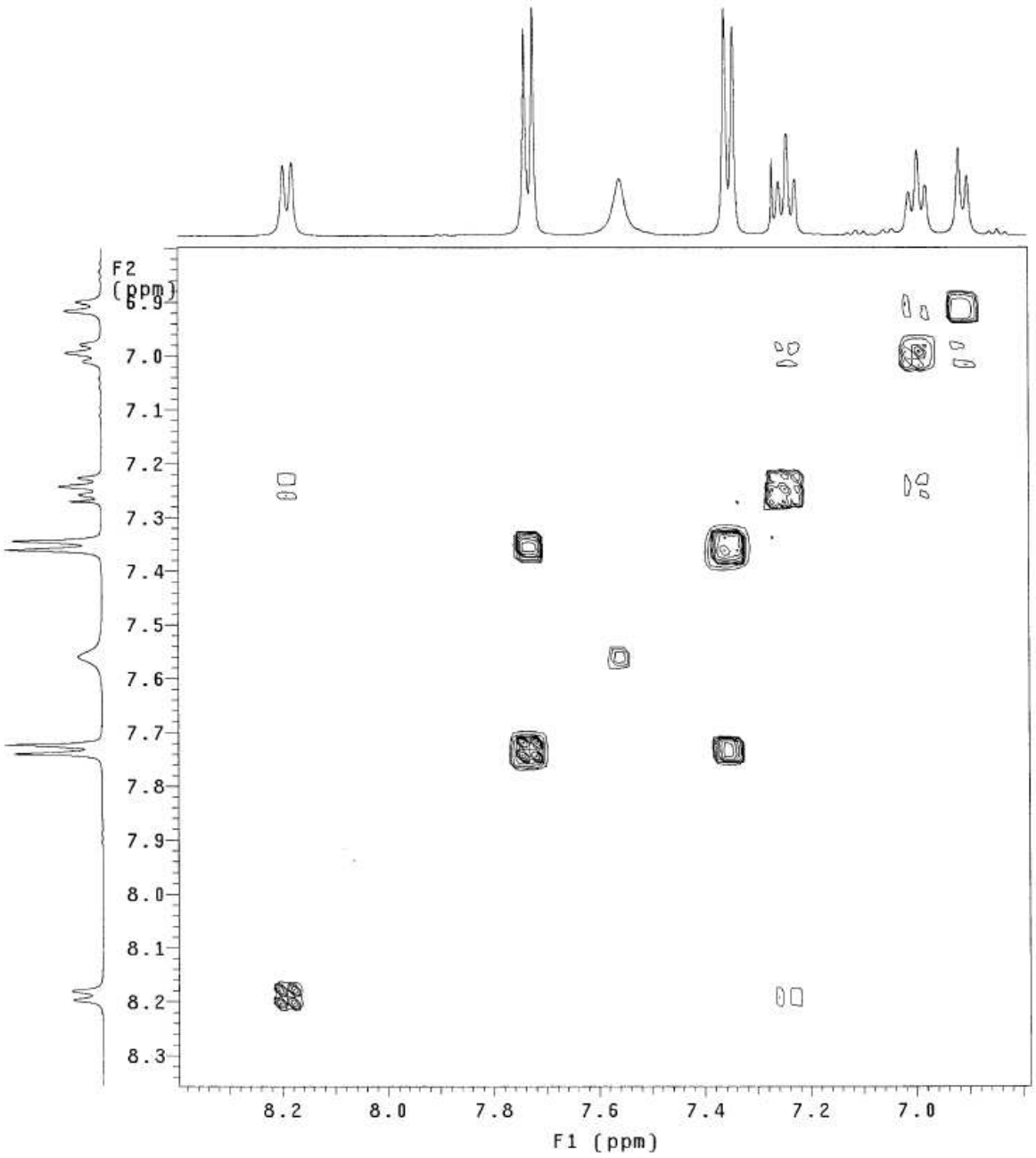

TY2-363
exp2 s2pul
SAMPLE
date Jun 9 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 2000
ct 290
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1402.7
wp 26962.9
vs 372
sc 0
wc 250
hzmm 107.85
is 500.00
rfl 11110.2
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
168.321
146.494
139.103
131.878
131.309
130.271
128.674
128.049
124.533
123.011
122.975
77.485
77.230
76.978
24.758
21.976
200
180
160
140
120
100
80
60
40
20
0
ppm

TY2-363

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
258 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, 24 min, 46 sec

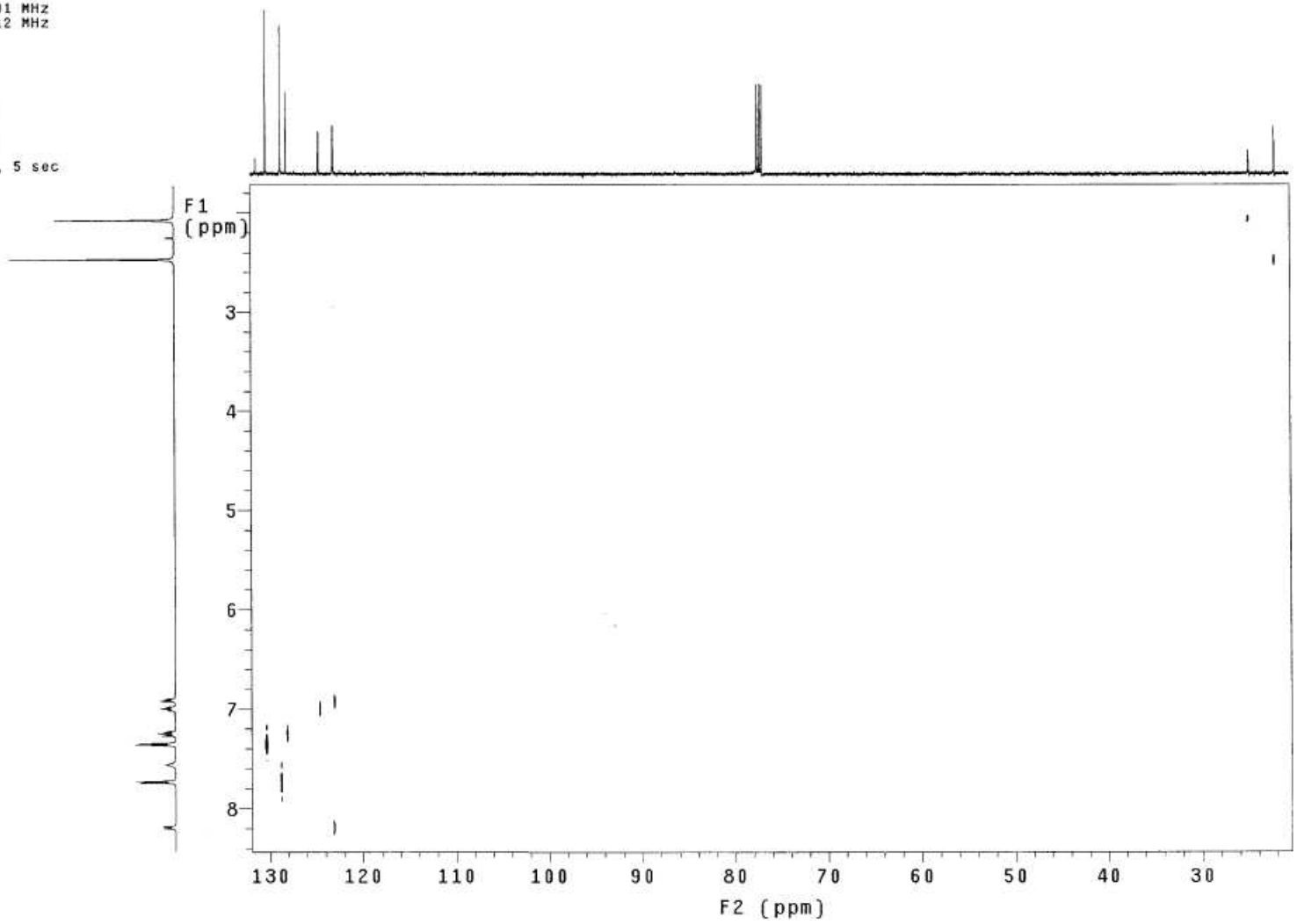


TY2-363

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, 125.6901591 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 3 hr, 46 min, 5 sec

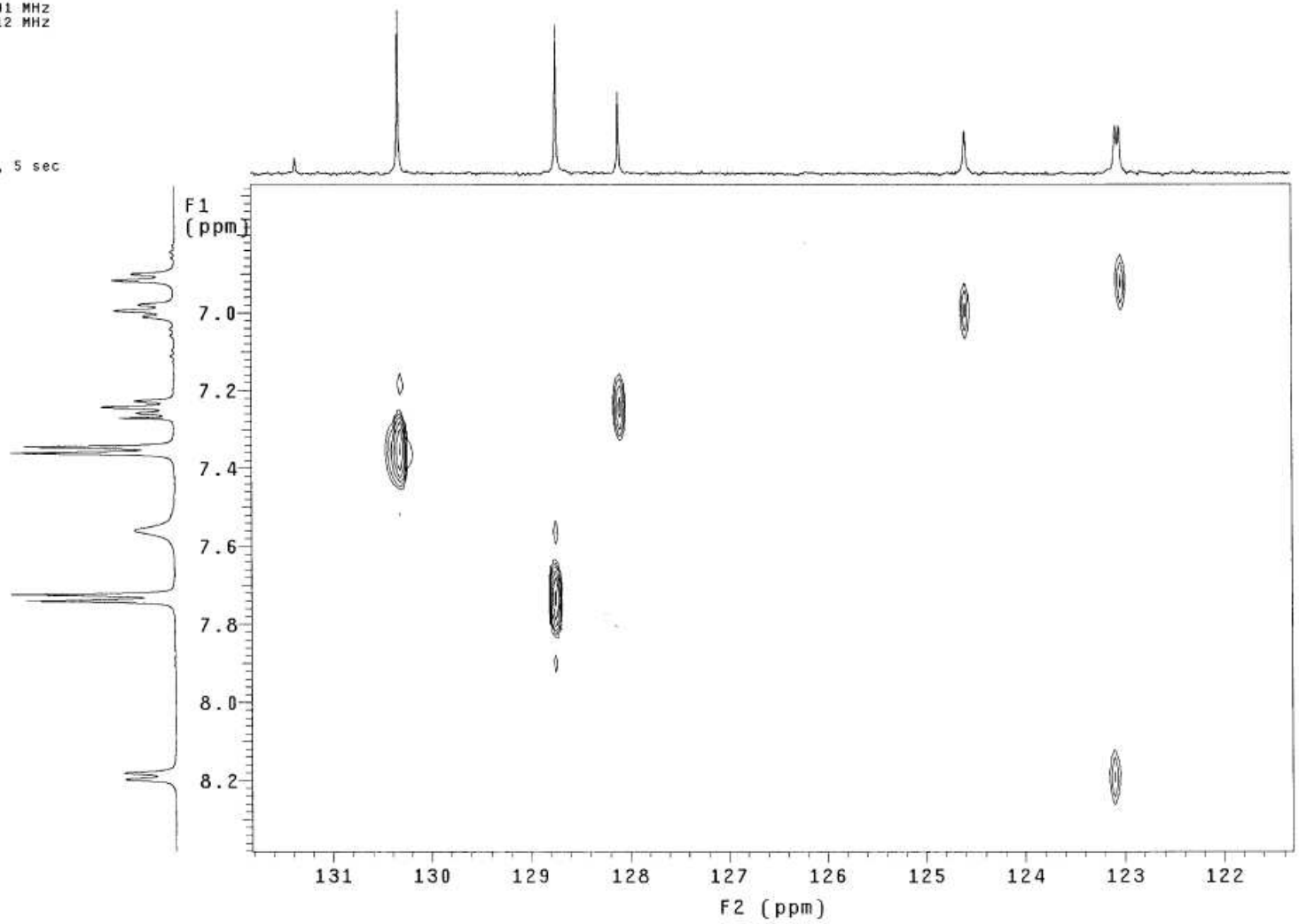


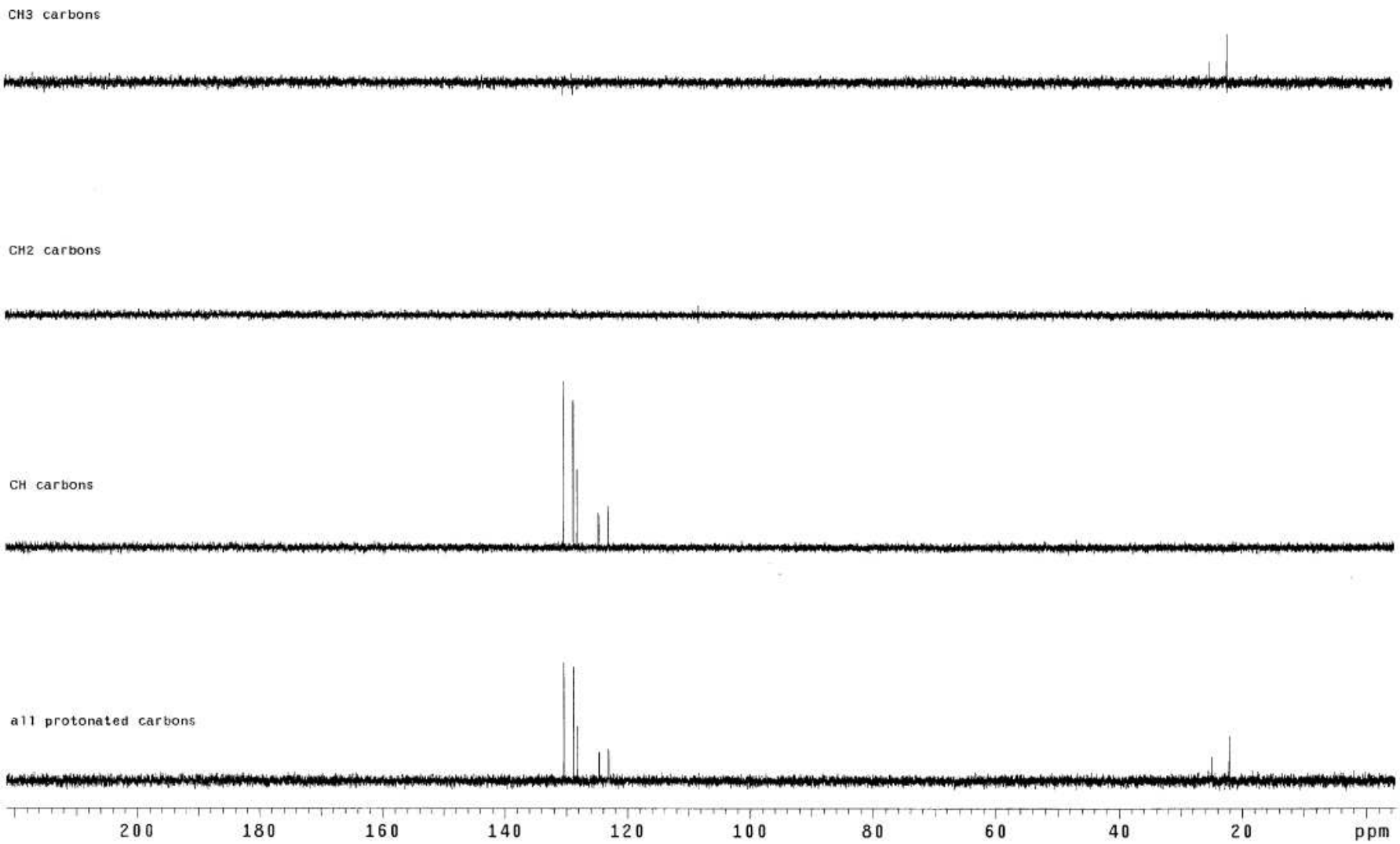
TY2-363

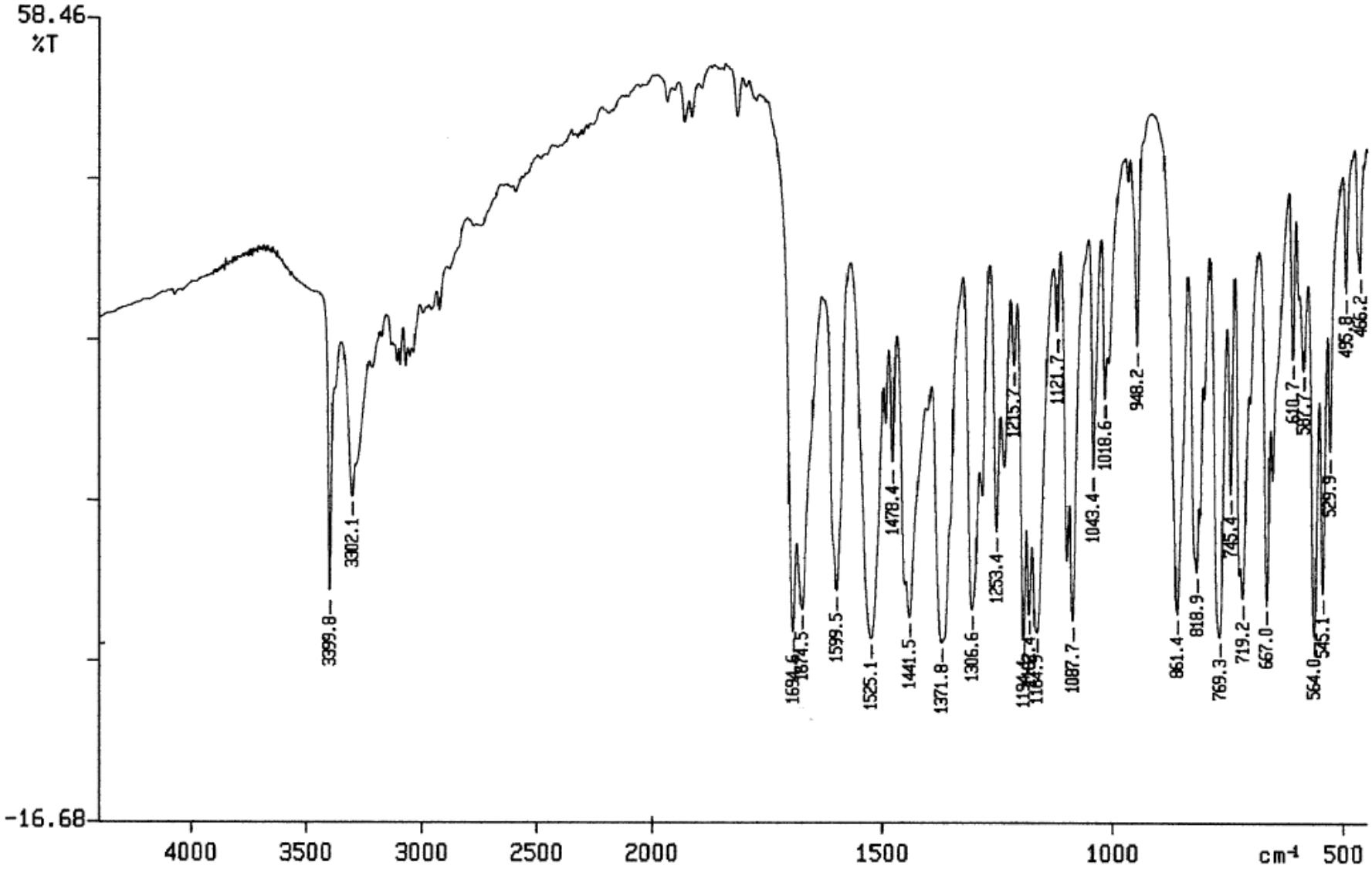
Pulse Sequence: hetcor

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, 125.6901591 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 3 hr, 46 min, 5 sec



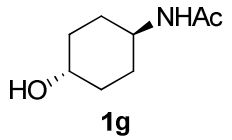

CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm


58.46
%T
-16.68
4000
3500
3000
2500
2000
1500
1000
cm-1
500
3399.8
3302.1
1599.5
1525.1
1478.4
1441.5
1371.8
1306.6
1253.4
1121.7
1087.7
1043.4
1018.6
948.2
861.4
818.9
769.3
745.4
719.2
667.0
564.0
545.1
529.9
495.8

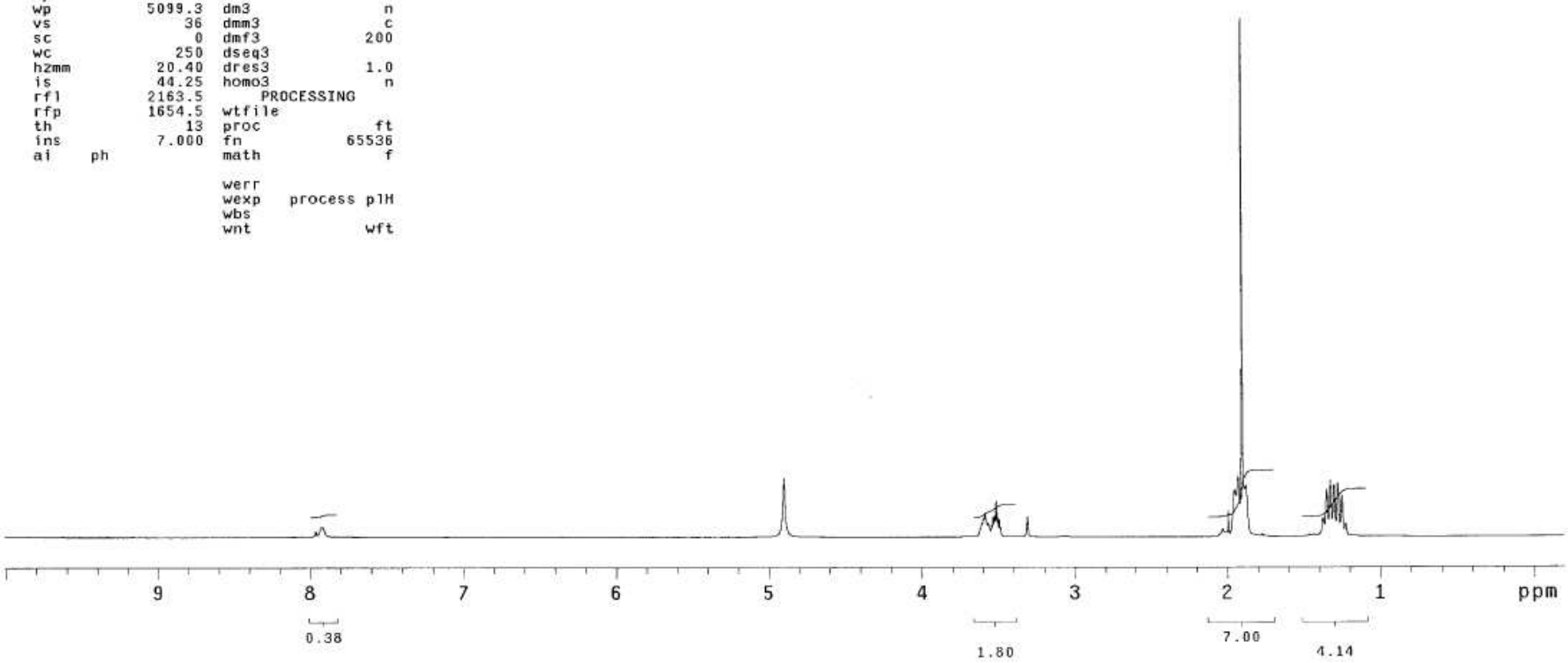
TY2-375

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jun 21 2009	dfrq	499.866
solvent	CD3OD	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	-96.8	dof3	0
wp	5099.3	dm3	n
vs	36	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	44.25	homo3	n
rfl	2163.5	PROCESSING	
rfp	1654.5	wtfile	
th	13	proc	ft
ins	7.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



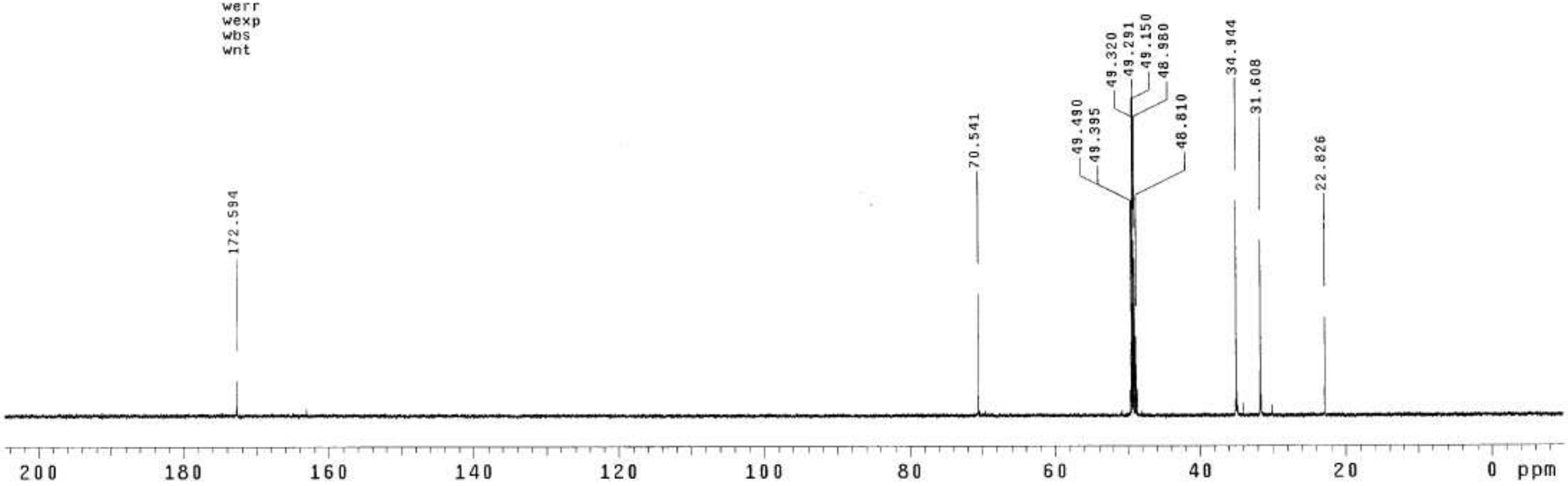
1g

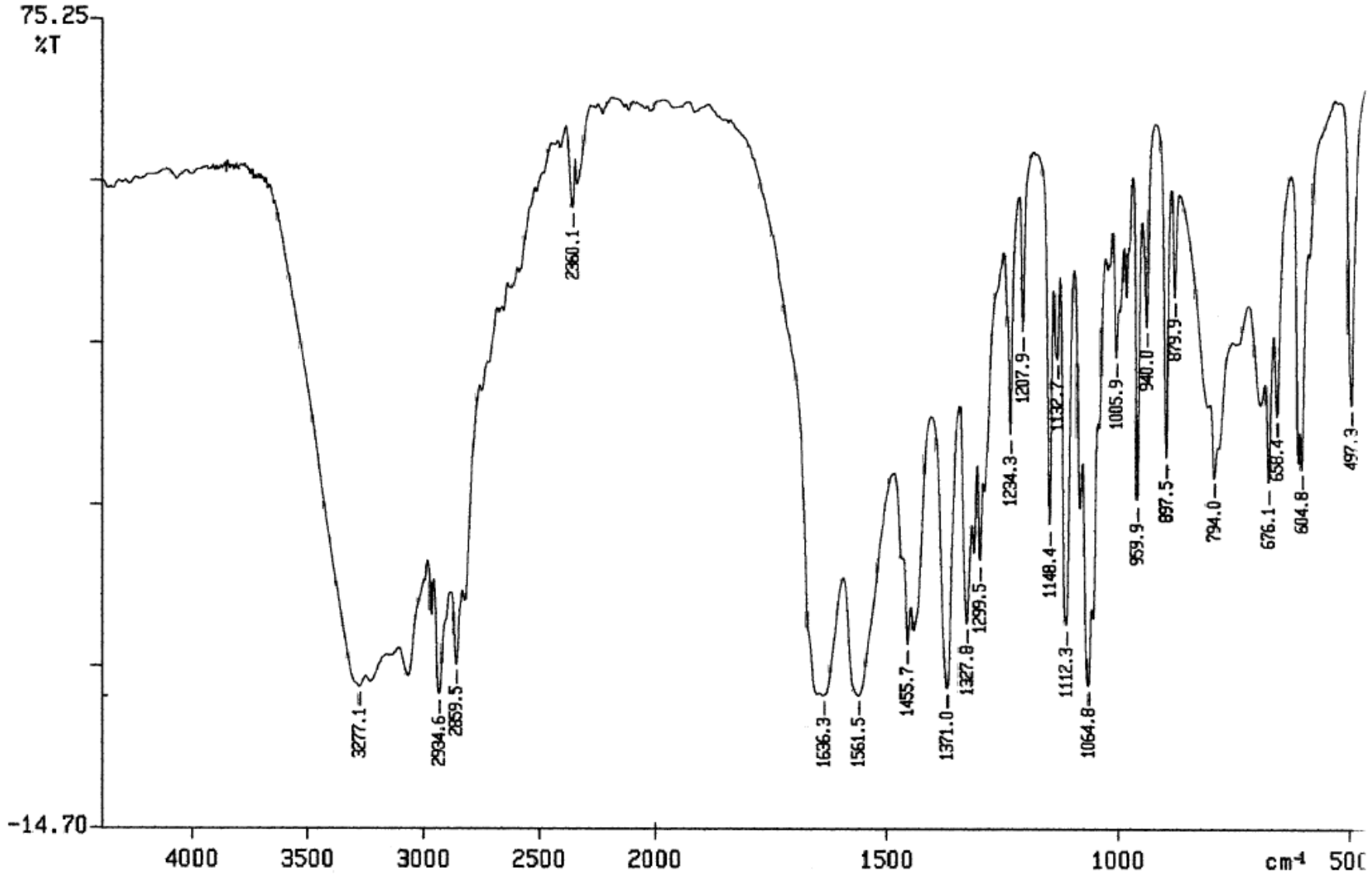


TY2-376

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 21 2009	dfrq	499.866
solvent	cd3od	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1000	dm2	n
ct	58	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	-1234.2	dof3	0
wp	26962.9	dm3	n
vs	61	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	7412.3	PROCESSING	
rfp	6177.7	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

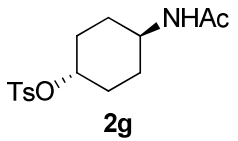



75.25
%T
-14.70
4000
3500
3000
2500
2000
1500
1000
cm-1
3277.1
2934.6
2859.5
2360.1
1636.3
1561.5
1455.7
1371.0
1327.8
1299.5
1234.3
1207.9
1148.4
1112.3
1064.8
1005.9
959.9
940.0
897.5
879.9
794.0
676.1
658.4
604.8
497.3

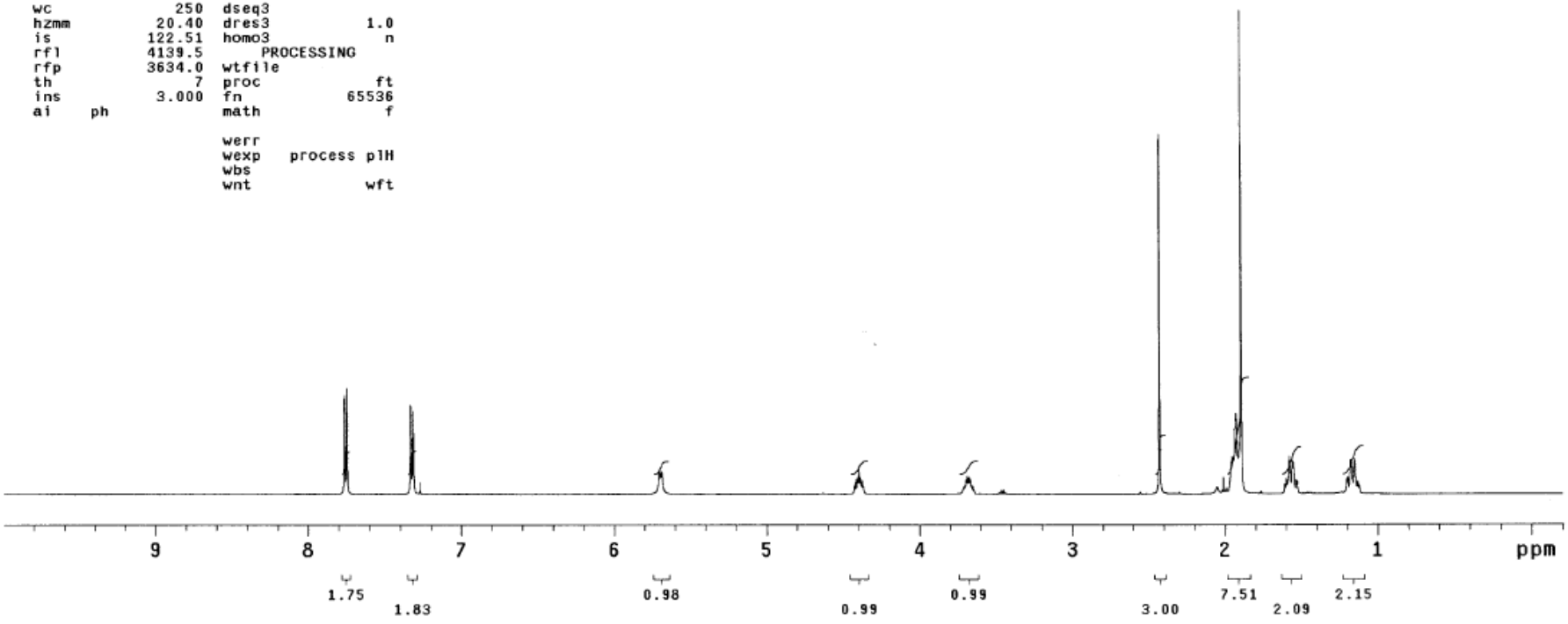
TY2-381-c5-24-recrystalization

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 7 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	-102.0	dof3	0
wp	5099.3	dm3	n
vs	33	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	122.51	homo3	n
rfl	4139.5	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



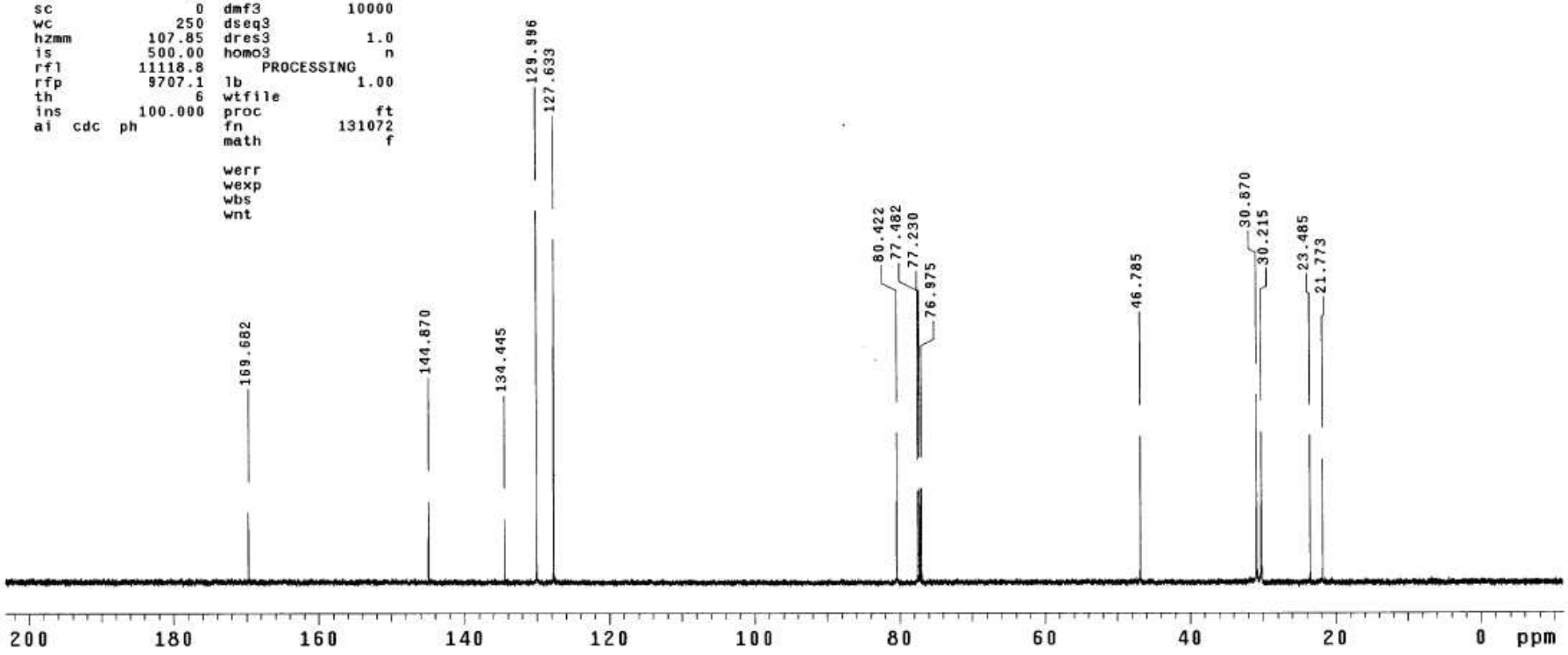
2g



TY2-381-c5-24-recrystalization

exp3 s2pul

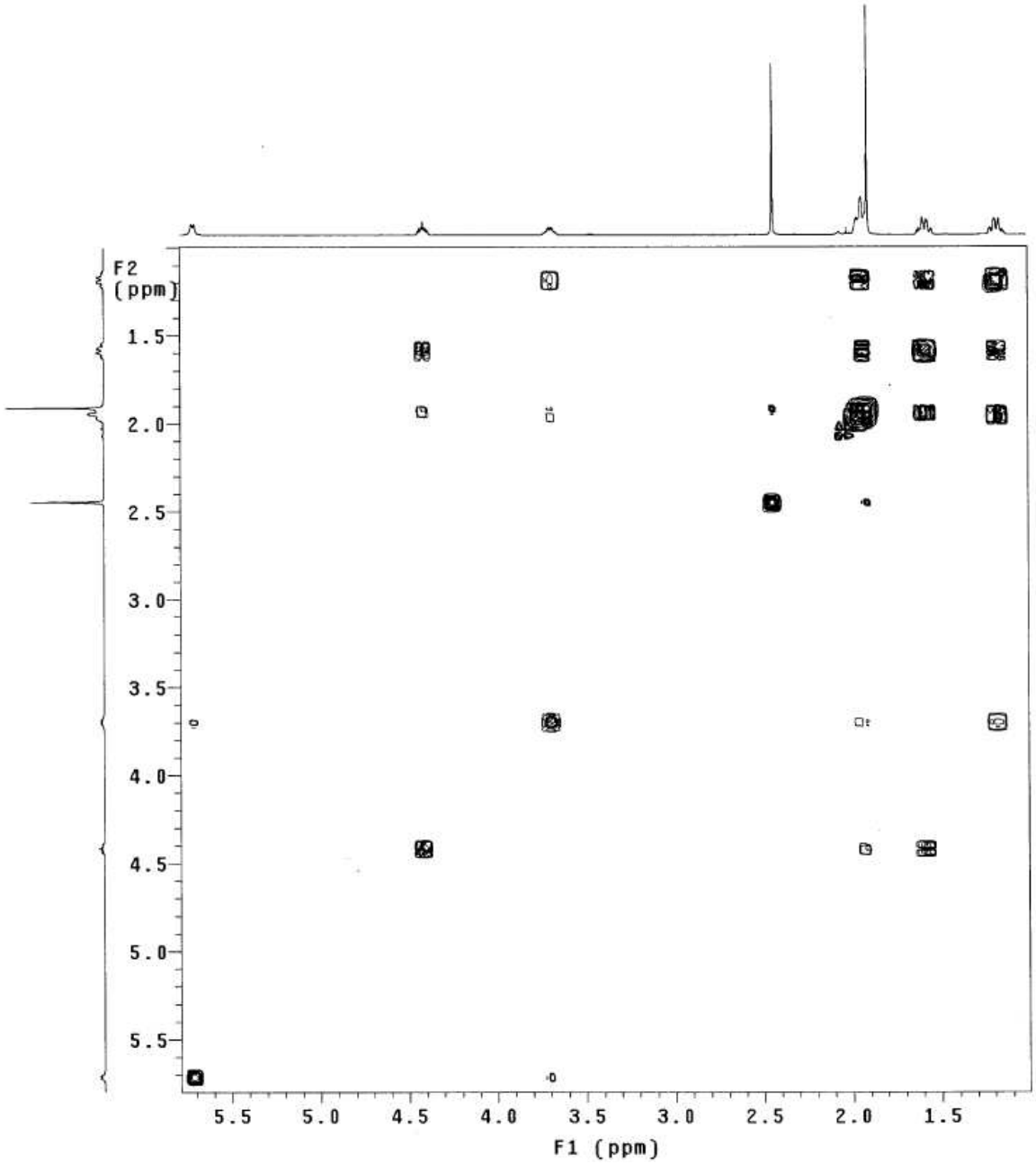
SAMPLE		DEC. & VT	
date	Jul 7 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1000	dm2	n
ct	67	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	-1411.3	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	11118.8	PROCESSING	
rfp	9707.1	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-381-c5-24-recrystalization

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

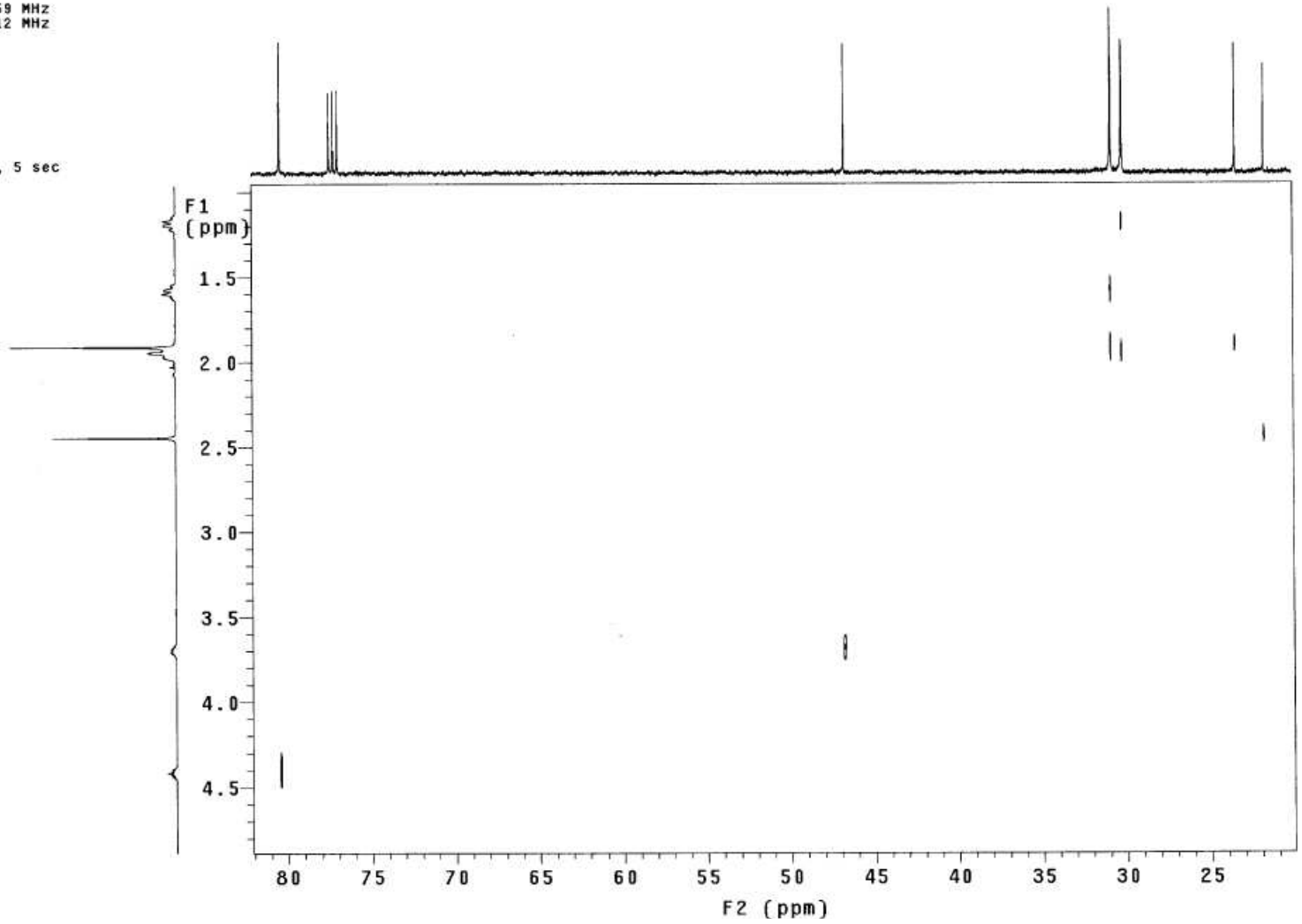


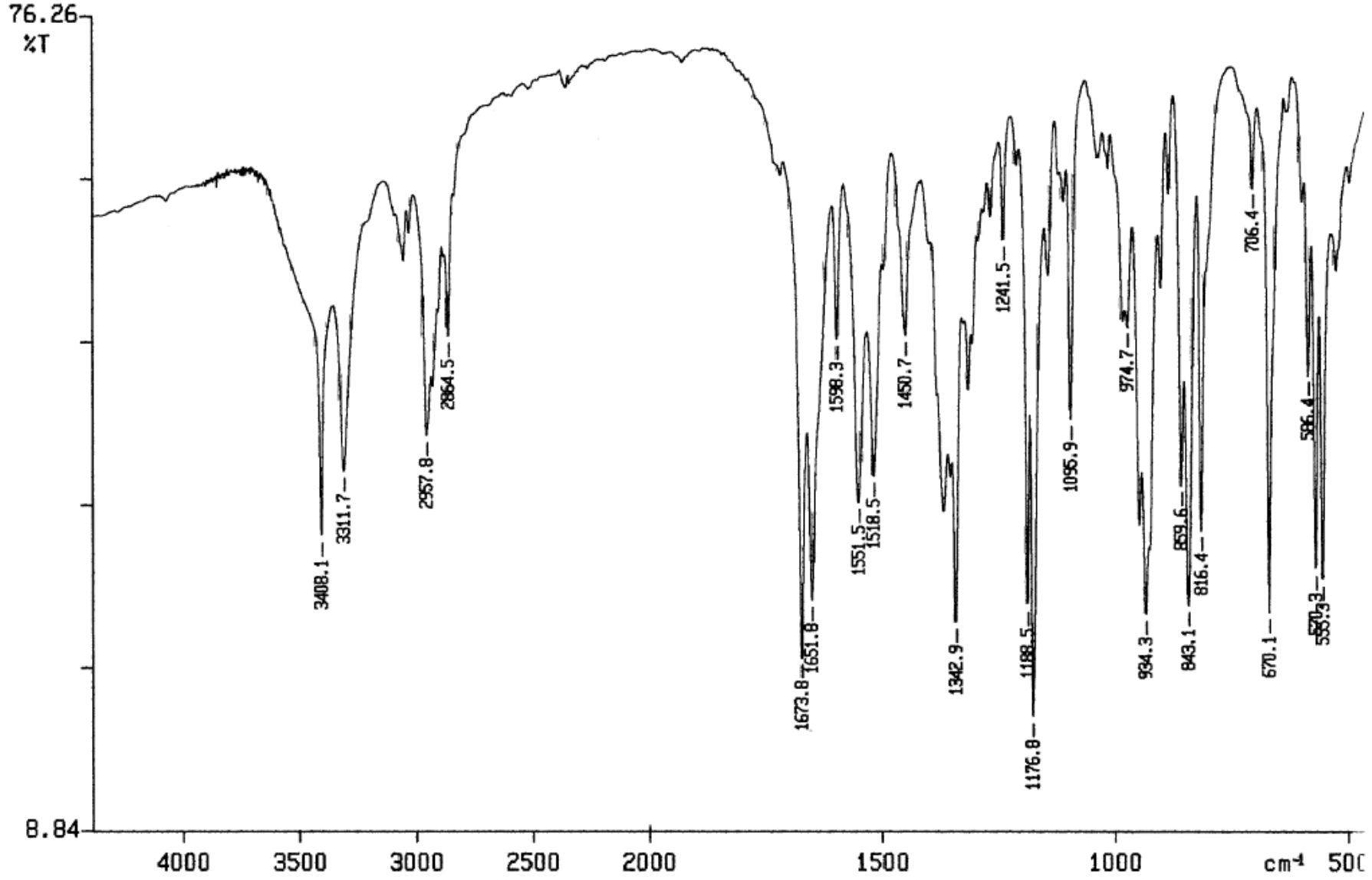
TY2-381-c5-24-recrystalization

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, 125.6901759 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

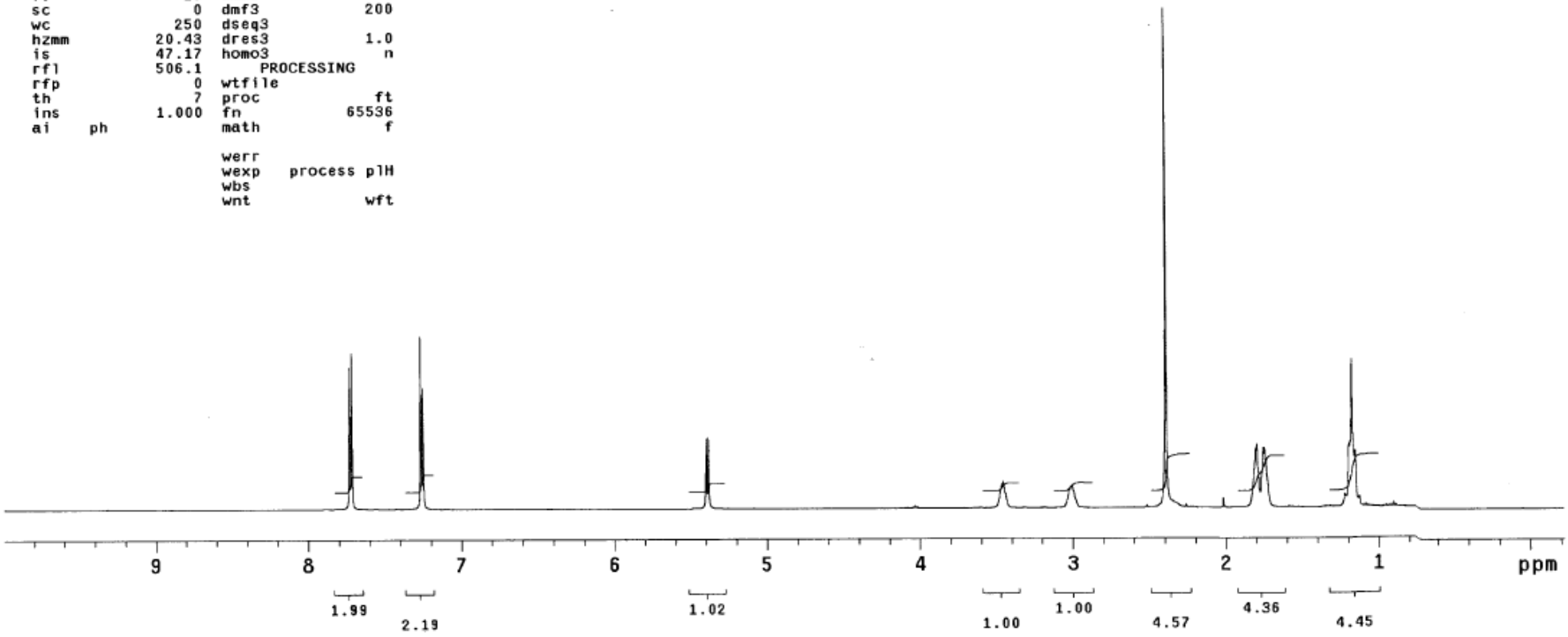


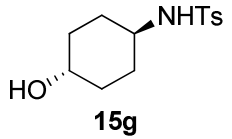

76.26
%T
8.84
3408.1
3311.7
2957.8
2864.5
1673.8
1651.8
1598.3
1551.5
1518.5
1450.7
1342.9
1241.5
1188.5
1176.8
1095.9
974.7
934.3
859.6
843.1
816.4
706.4
670.1
586.4
570.3
555.3
4000
3500
3000
2500
2000
1500
1000
cm-1
500

TY2-411

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 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	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	-111.2	dof3	0
wp	5107.9	dm3	n
vs	28	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	47.17	homo3	n
rfl	506.1	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process plH
		wbs	
		wnt	wft



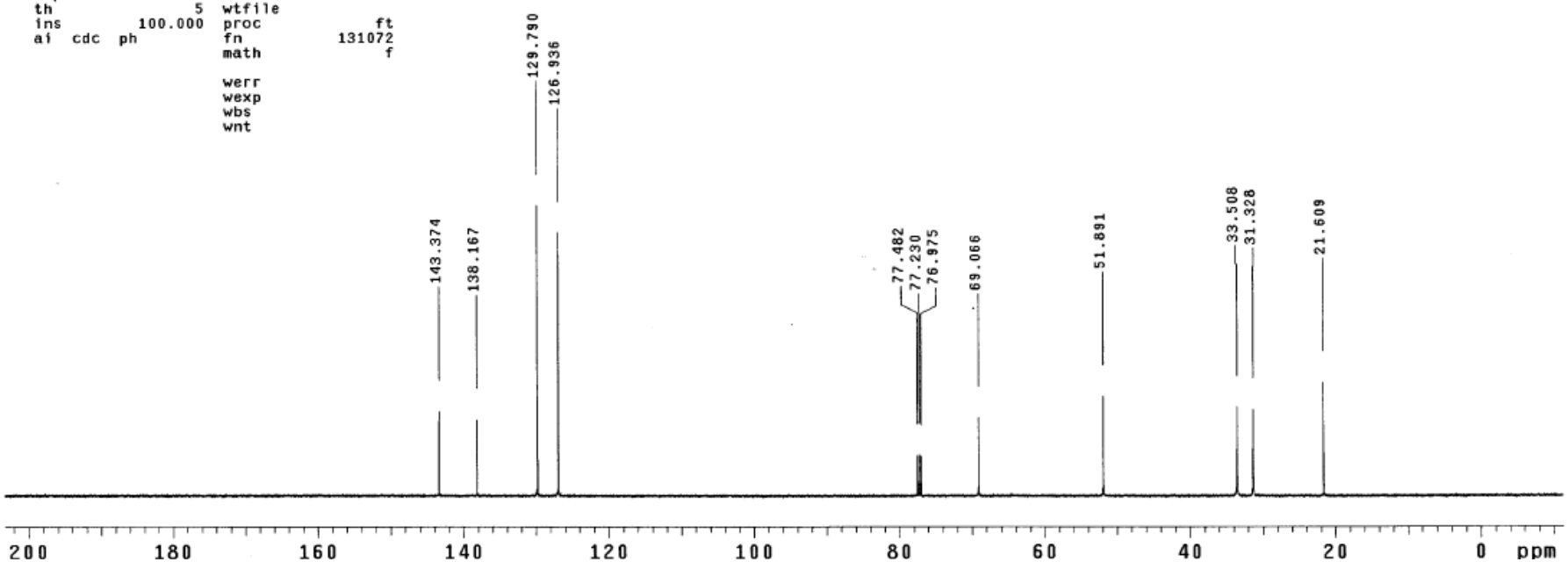


15g

TY2-411

exp3 s2pul

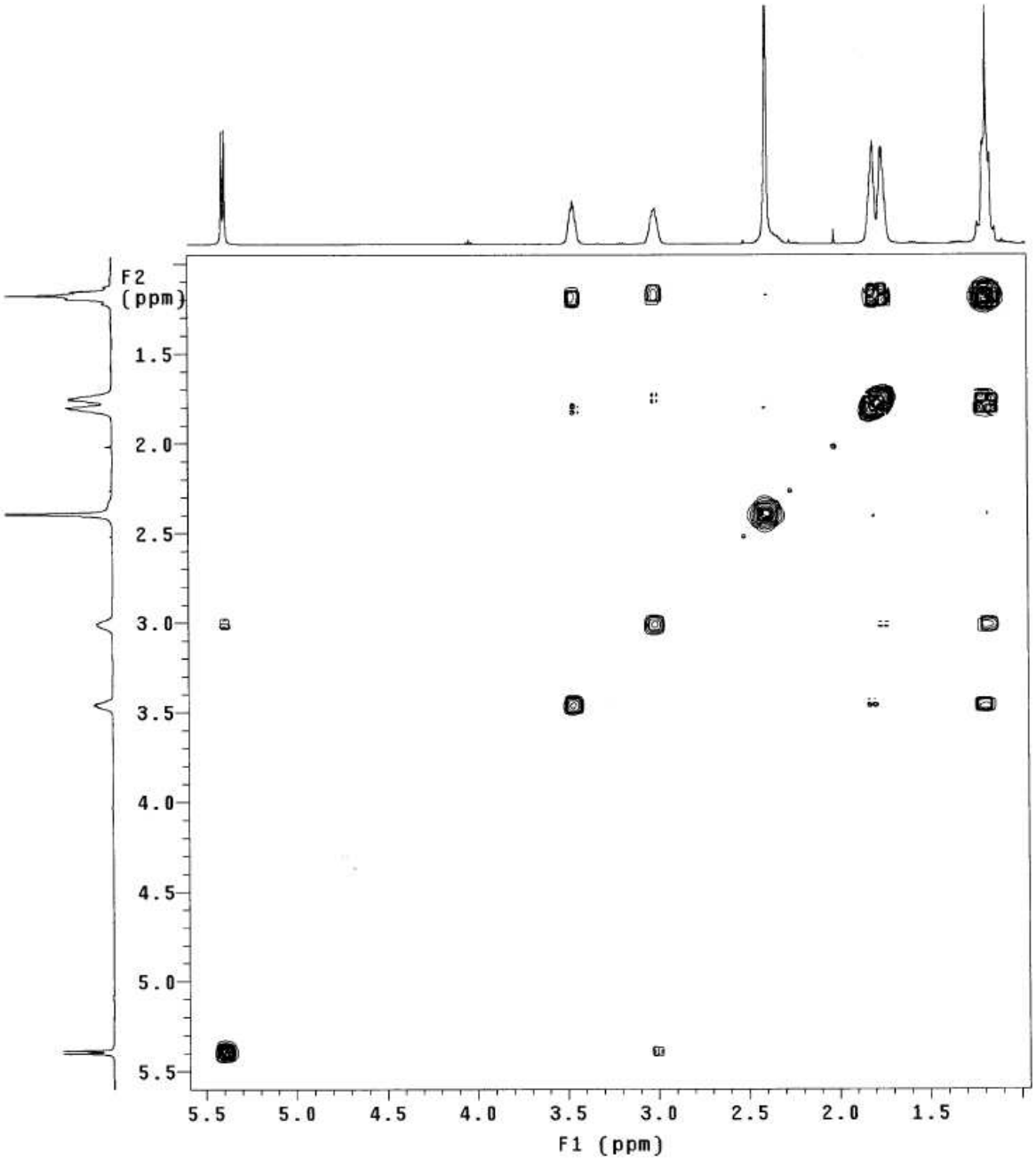
SAMPLE		DEC. & VT	
date	Jul 20 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	256	dm2	n
ct	67	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	-1417.1	dof3	0
wp	26962.9	dm3	n
vs	41	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11124.6	PROCESSING	
rfp	9707.1	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-411

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

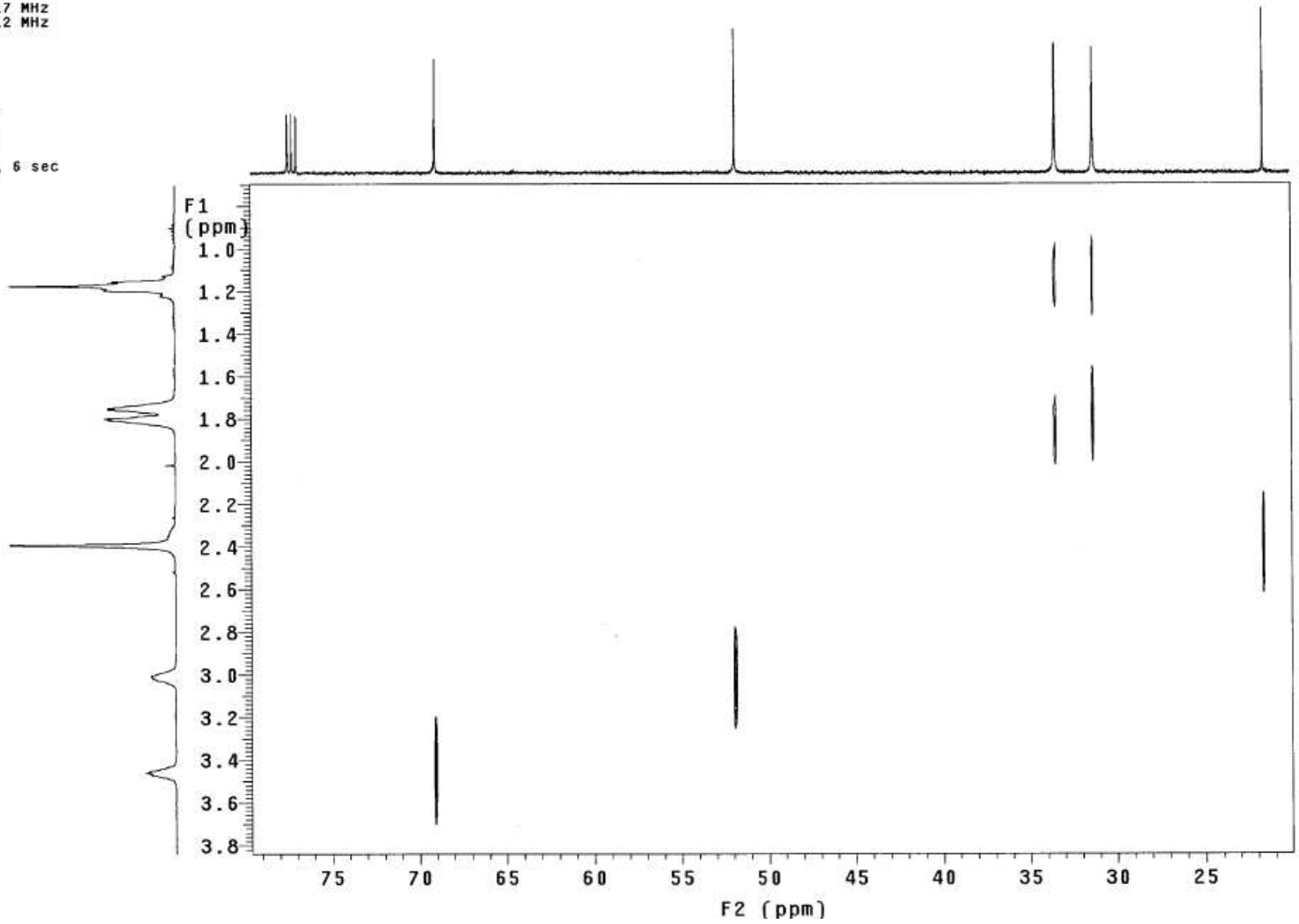
 Relax. delay 1.300 sec
 COSY 90-90
 Acq. time 0.157 sec
 Width   6533.3 Hz
 2D Width 6533.3 Hz
 16 repetitions
 256 increments
OBSERVE   H1, 499.8611707 MHz
DATA PROCESSING
 Sine bell 0.078 sec
F1 DATA PROCESSING
 Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

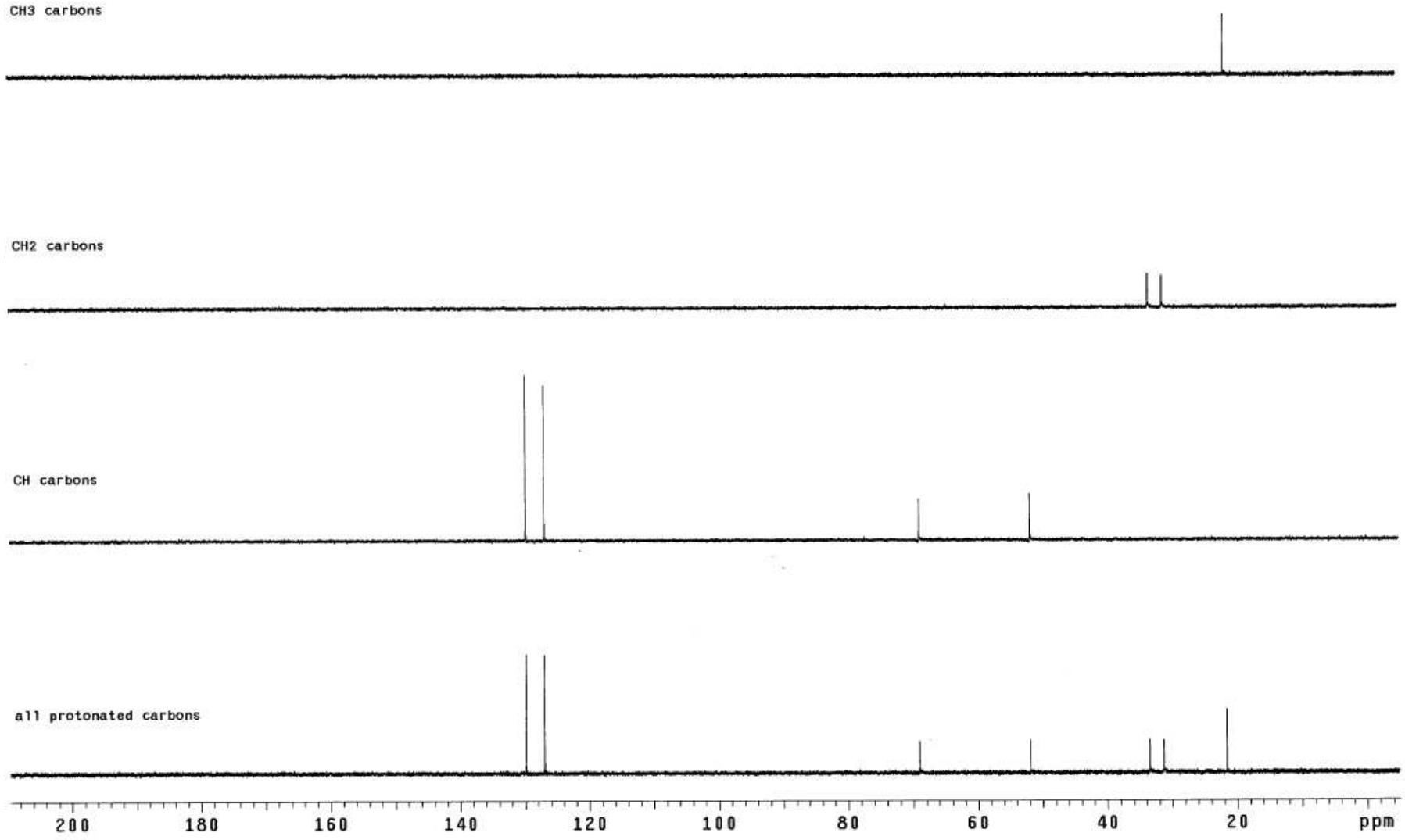


TY2-411

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, 125.6901817 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

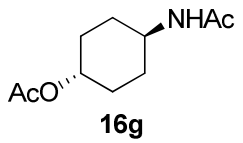



CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
200
180
160
140
120
100
80
60
40
20
ppm

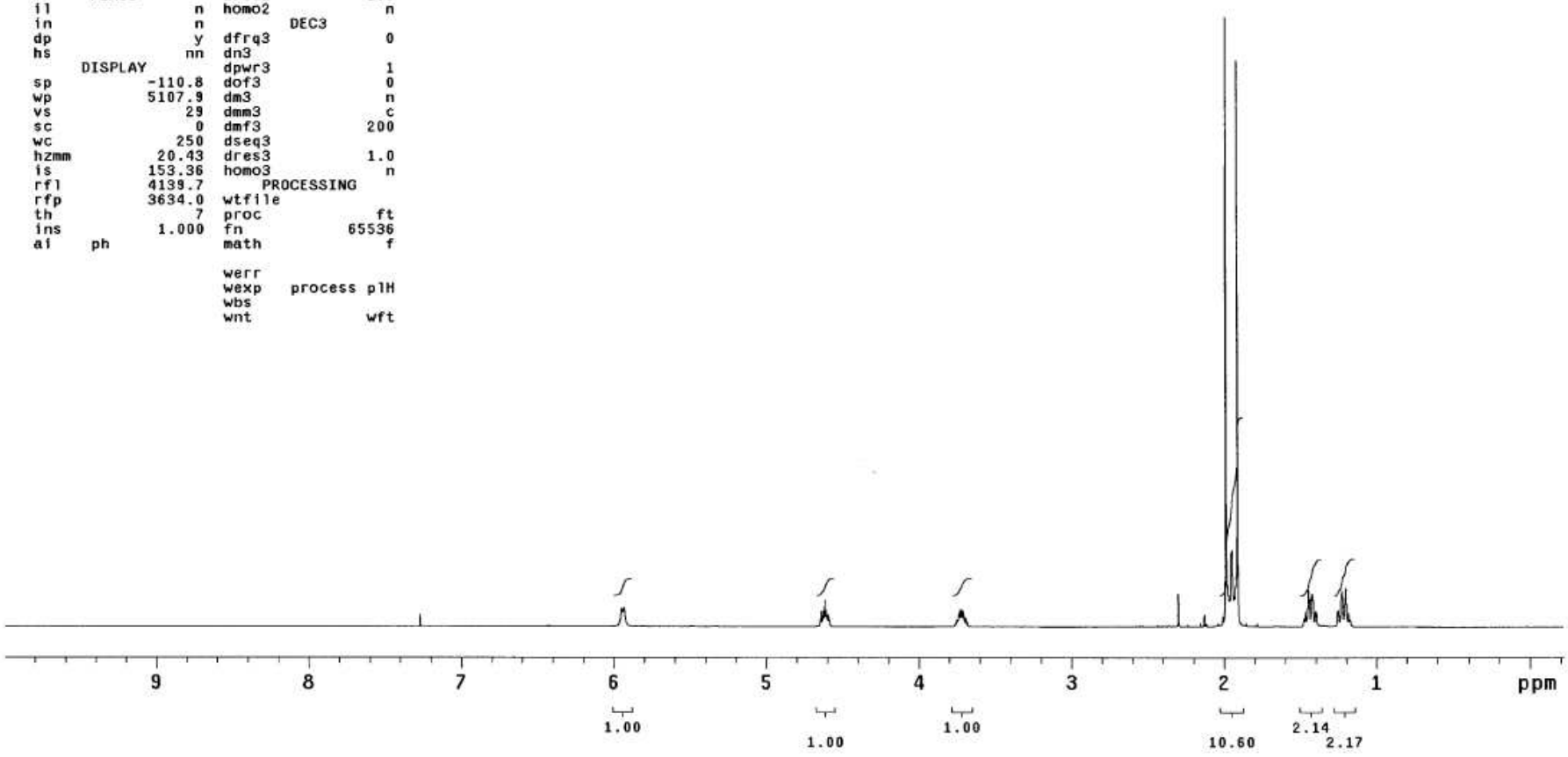
TY2-415

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 21 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	-110.8	dof3	0
wp	5107.9	dm3	n
vs	29	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.43	dres3	1.0
is	153.36	homo3	n
rfl	4139.7	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	1.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



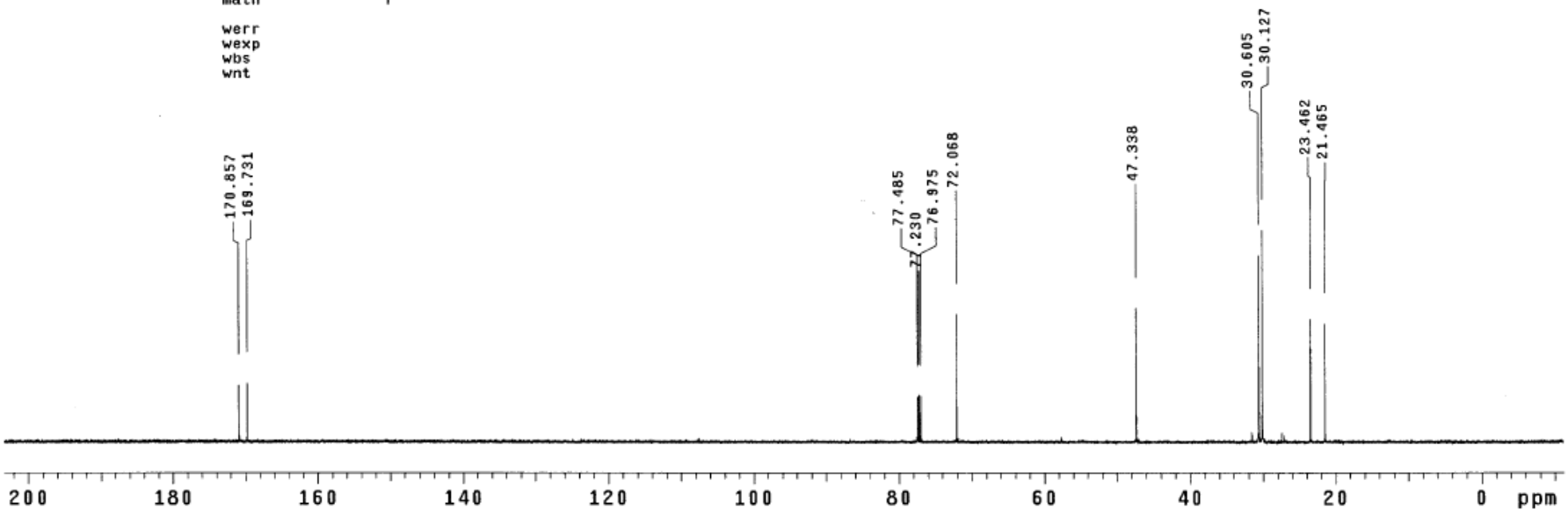
16g



TY2-415

exp3 s2pul

SAMPLE		DEC. & VT	
date	Jul 21 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	512	dm2	n
ct	69	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	-1411.8	dof3	0
wp	26962.9	dm3	n
vs	47	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	107.85	dres3	1.0
is	500.00	homo3	n
rfl	11119.2	PROCESSING	
rfp	9707.1	lb	1.00
th	6	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

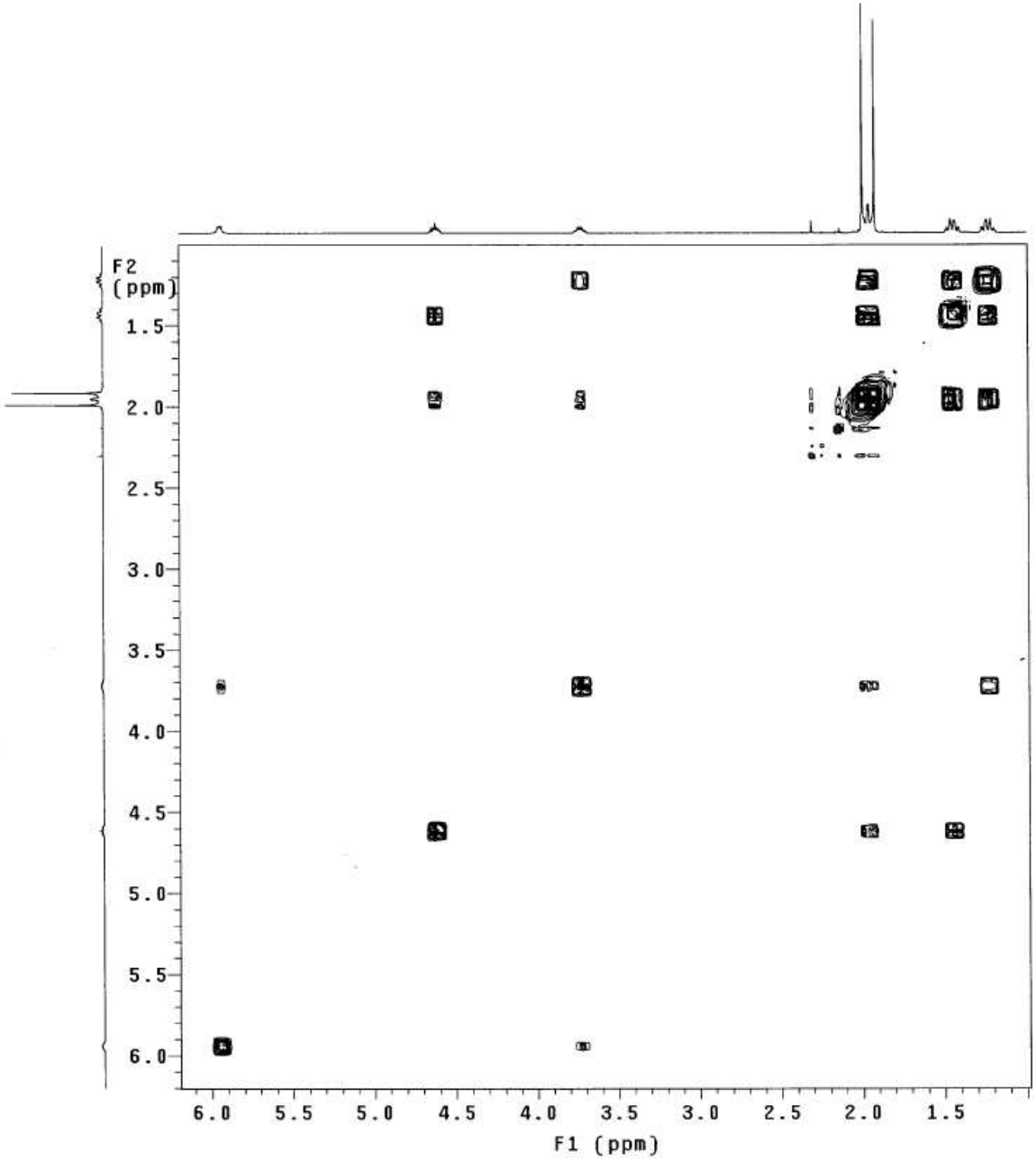


TY2-415

Pulse Sequence: relayh

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

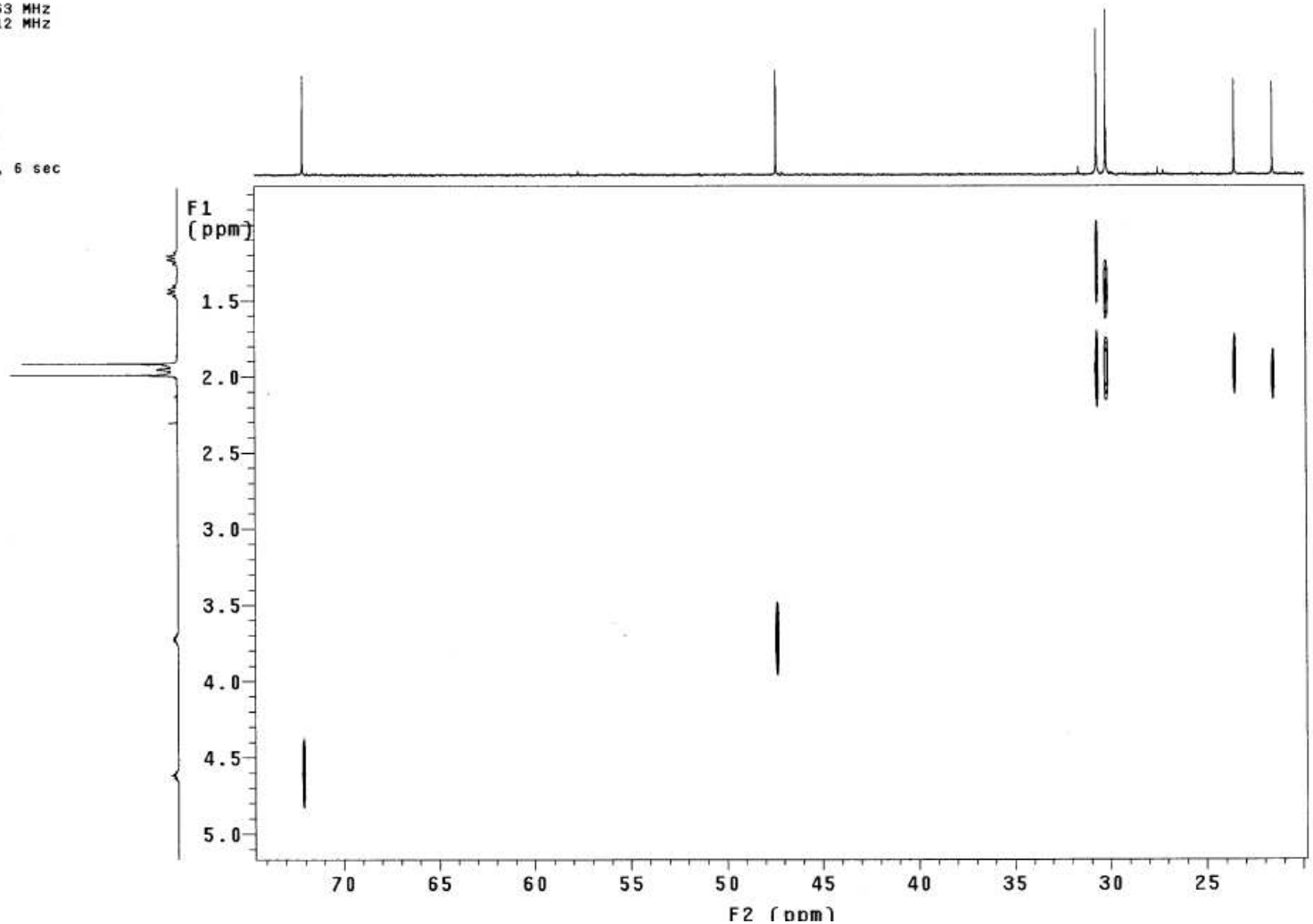
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.157 sec
Width 6533.3 Hz
2D Width 6533.3 Hz
16 repetitions
256 increments
OBSERVE H1, 499.8611703 MHz
DATA PROCESSING
Sine bell 0.078 sec
F1 DATA PROCESSING
Sine bell 0.039 sec
FT size 2048 x 2048
Total time 1 hr, 41 min, 40 sec

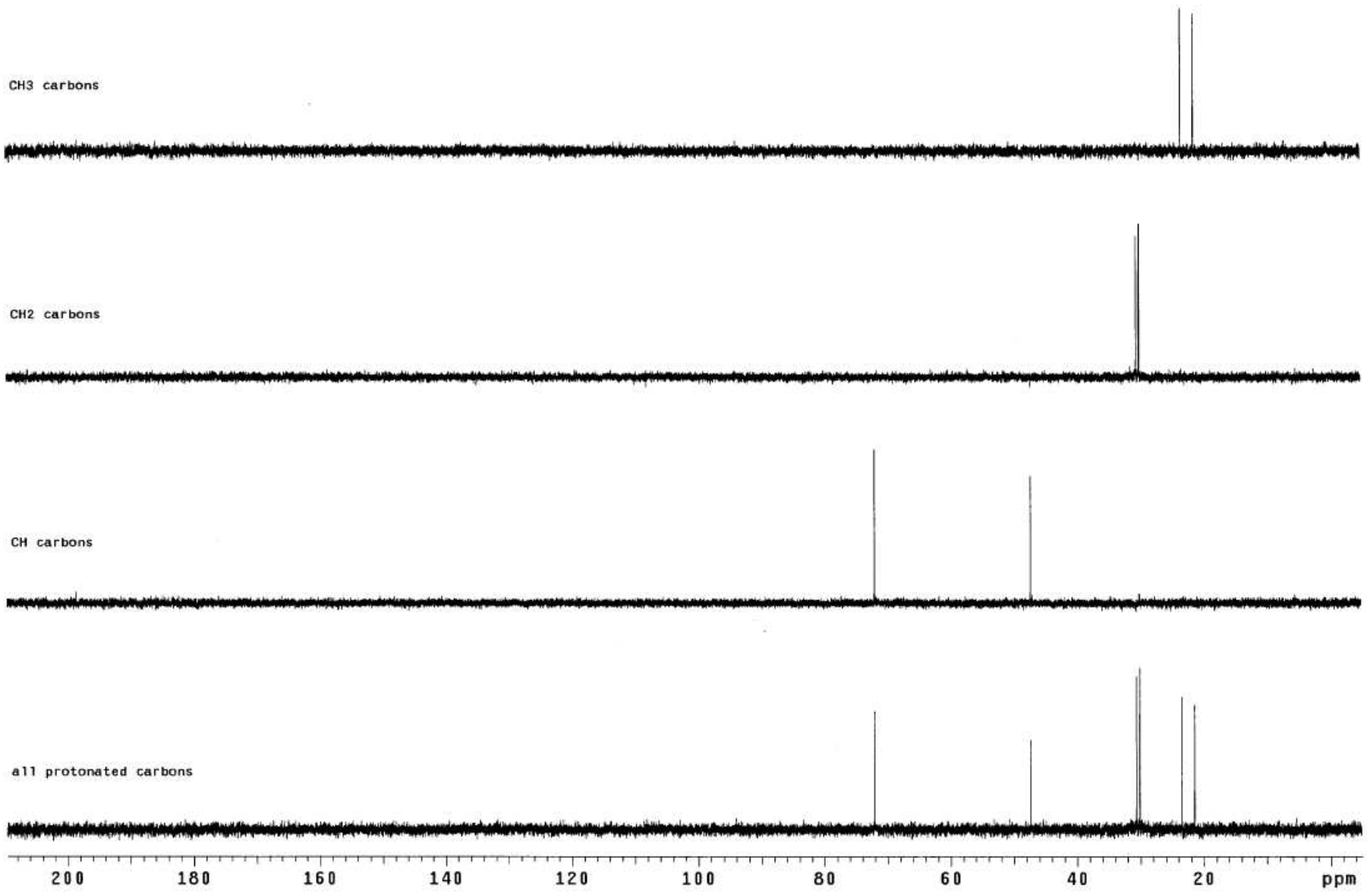


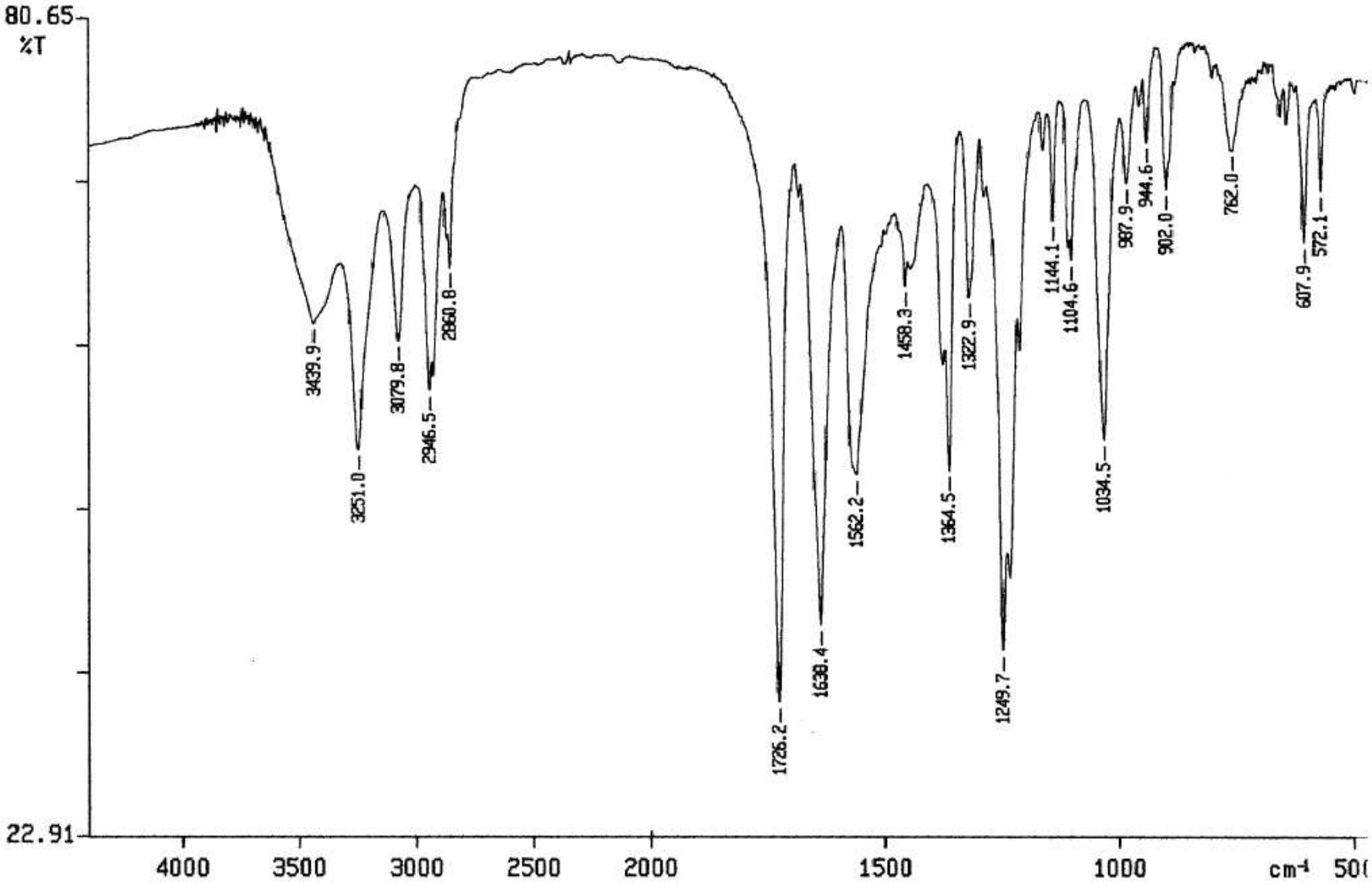
TY2-415

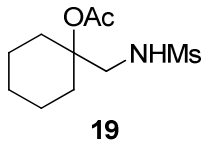
Pulse Sequence: hetcor
 Solvent: CDCl3
 Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"

 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, 125.6901763 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


80.65
%T
22.91
3439.9
3251.0
3079.8
2946.5
2860.8
1726.2
1638.4
1562.2
1458.3
1364.5
1322.9
1249.7
1144.1
1104.6
1034.5
987.9
944.6
902.0
762.0
607.9
572.1
4000
3500
3000
2500
2000
1500
1000
cm-1
500

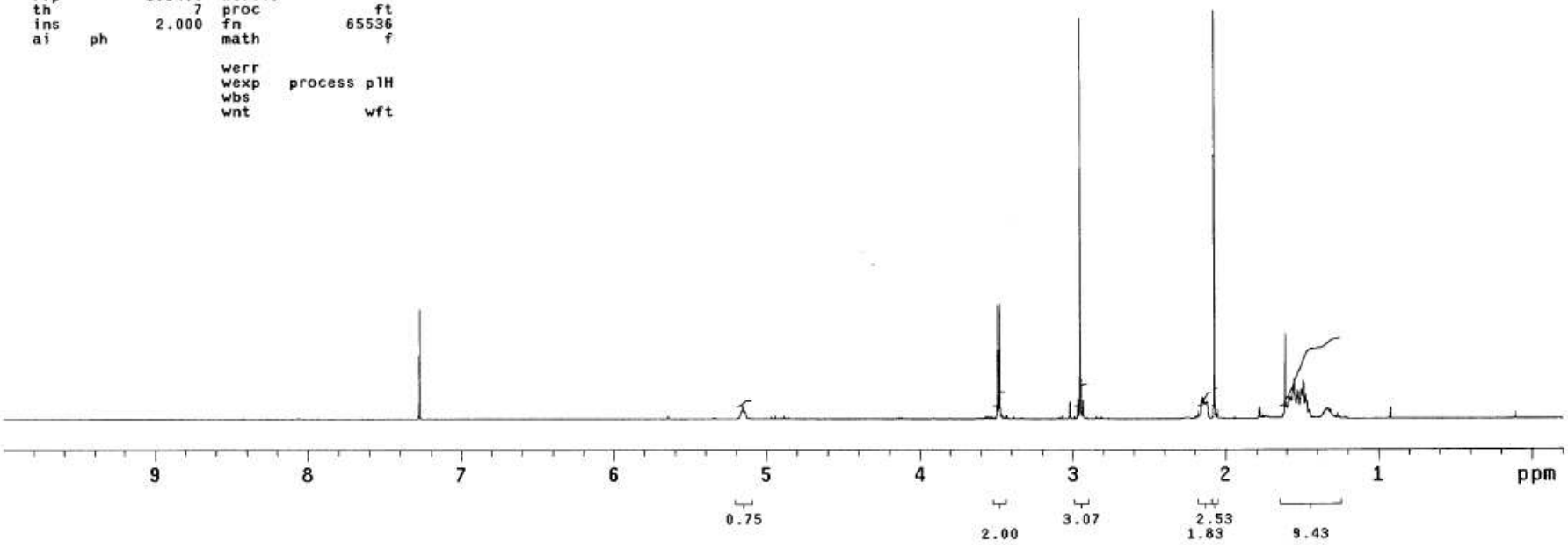


19

TY2-416

exp2 s2pul

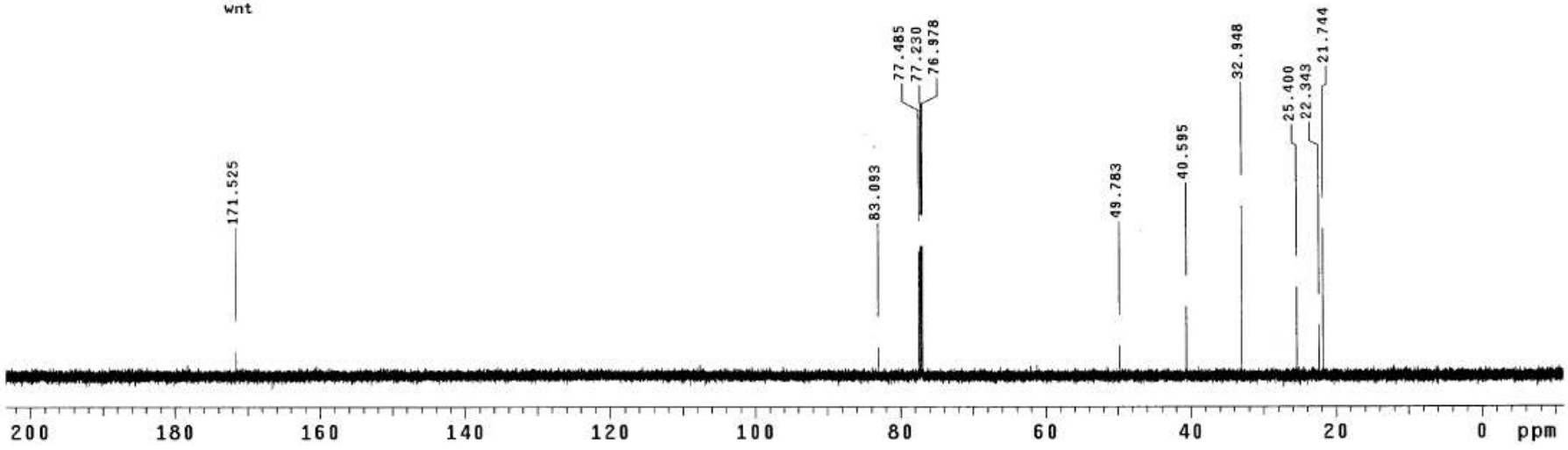
SAMPLE		DEC. & VT	
date	Jul 18 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.864	dm	nnn
tn	H1	dmm	c
at	5.016	dmf	200
np	65536	dseq	
sw	6533.3	dres	1.0
fb	4000	homo	n
bs	4	DEC2	
tpwr	61	dfrq2	0
pw	13.5	dn2	
d1	0.100	dpwr2	1
tof	269.9	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
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	-102.2	dof3	0
wp	5099.3	dm3	n
vs	21	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	20.40	dres3	1.0
is	69.57	homo3	n
rfl	4139.7	PROCESSING	
rfp	3634.0	wtfile	
th	7	proc	ft
ins	2.000	fn	65536
ai ph		math	f
		werr	
		wexp	process p1H
		wbs	
		wnt	wft



TY2-416

exp3 s2pul

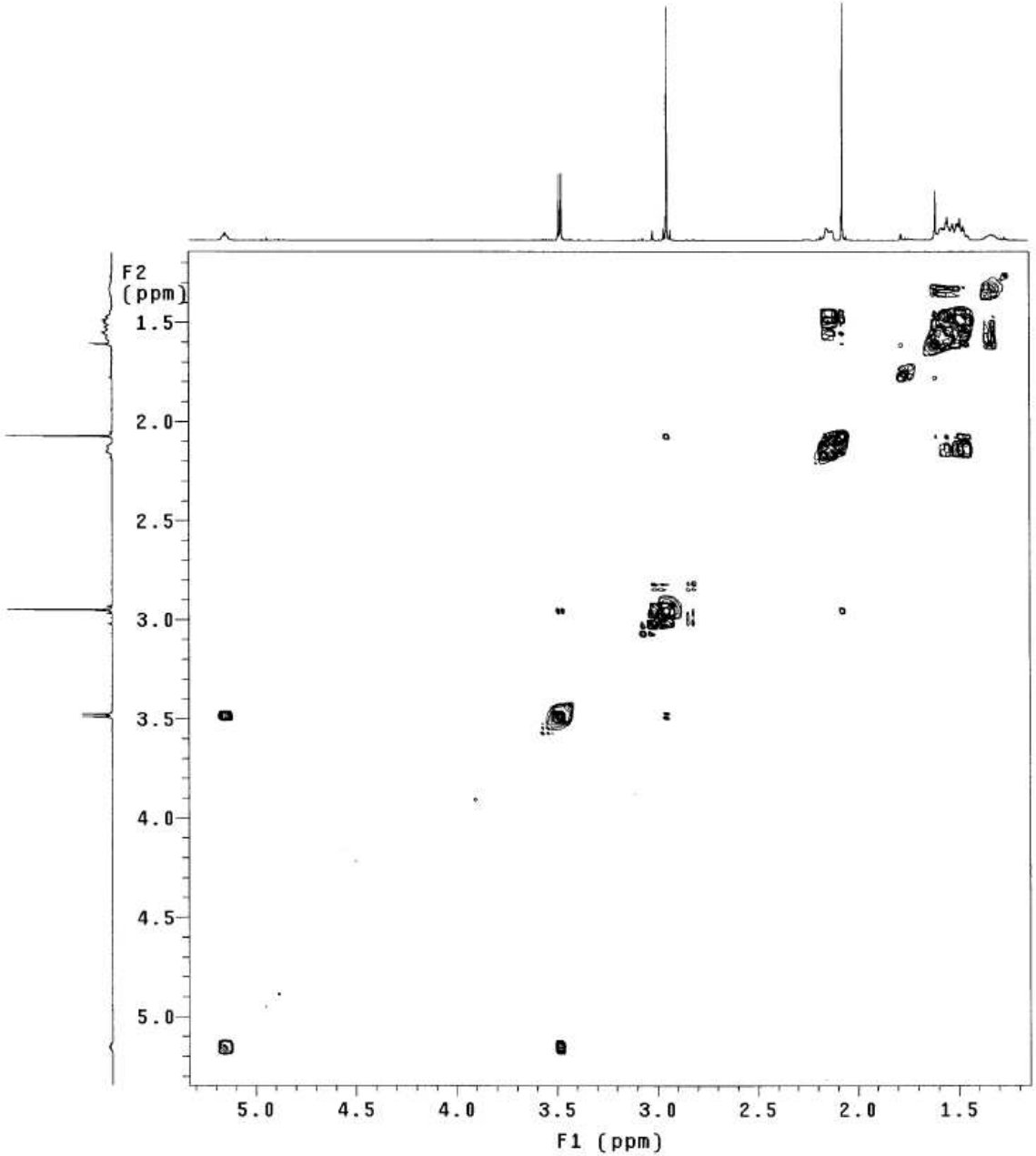
SAMPLE		DEC. & VT	
date	Jul 18 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	1000	dm2	n
ct	122	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	95	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	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	



TY2-416

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, 499.8611703 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

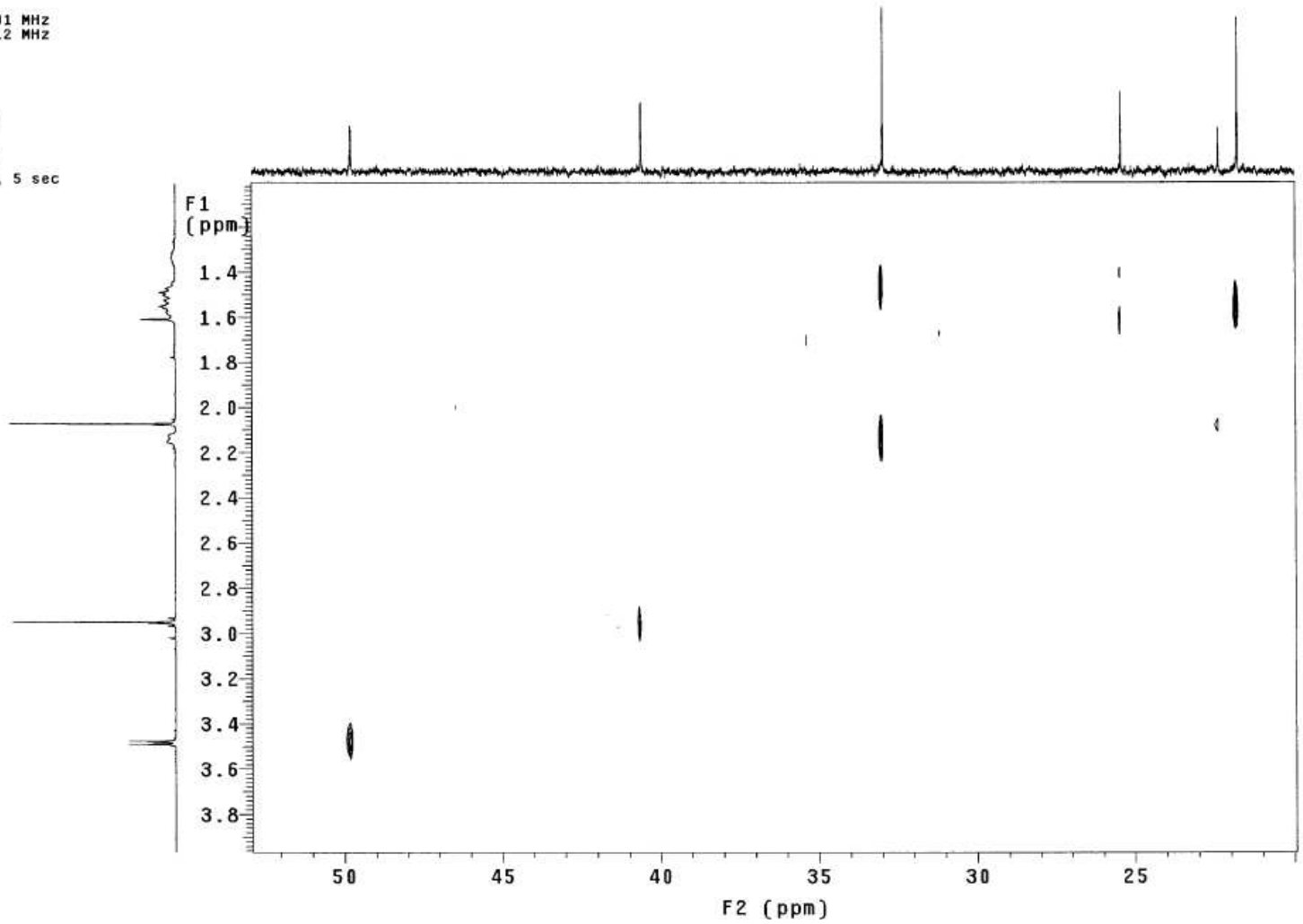


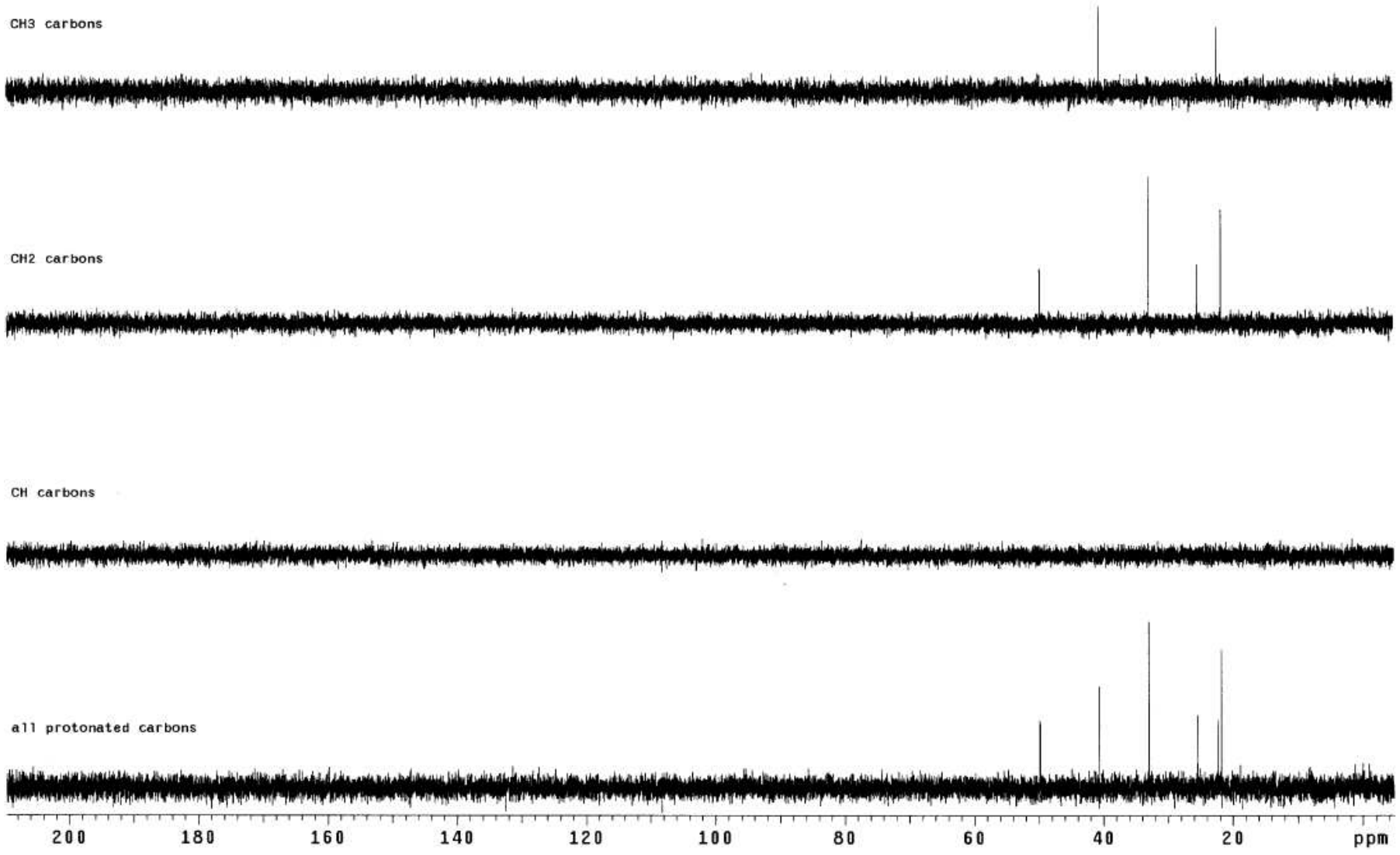
TY2-416

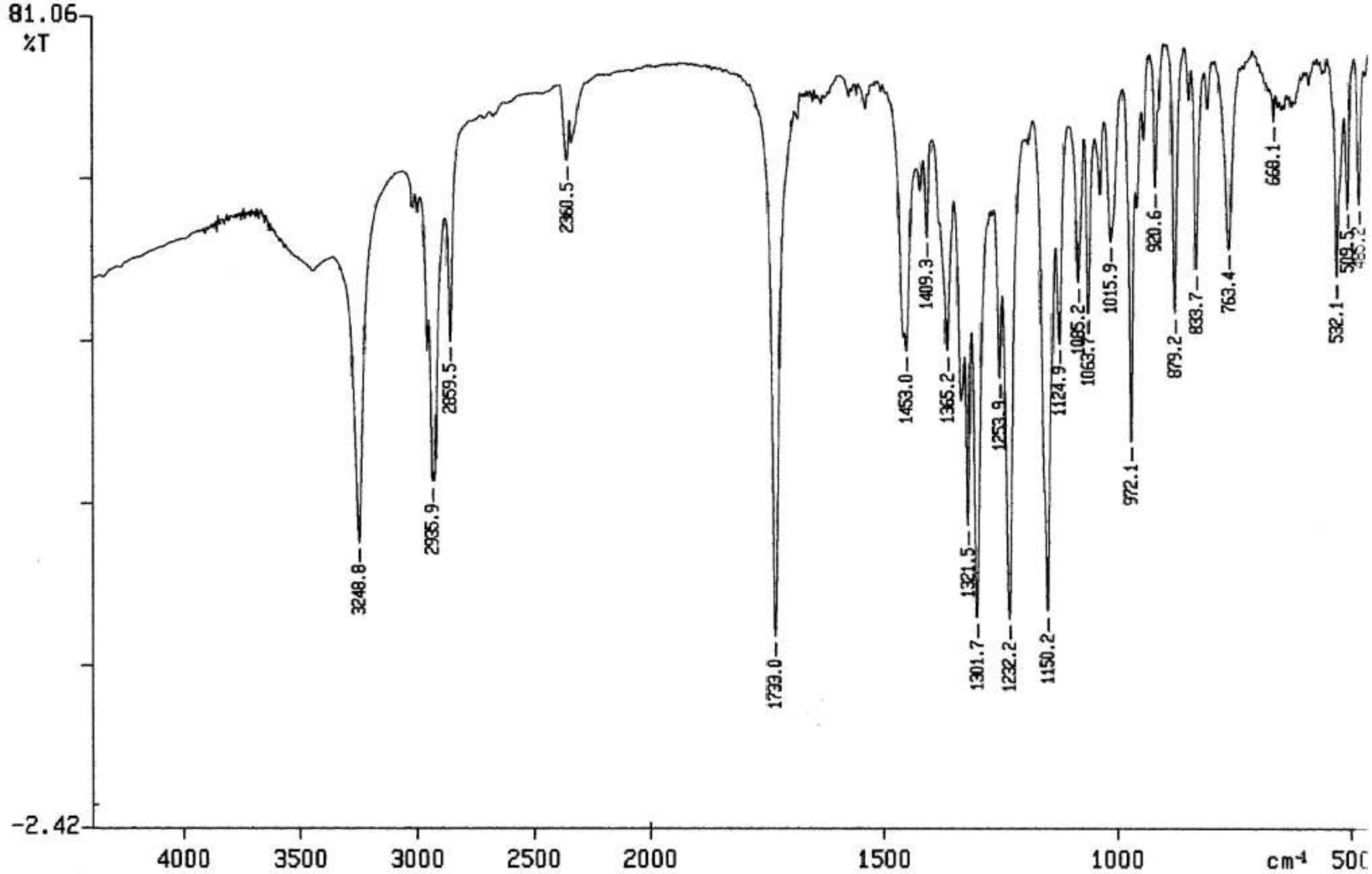
Pulse Sequence: hetcor

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: TY2-416-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, 125.6901591 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 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


81.06
%T
-2.42
4000
3500
3000
2500
2000
1500
1000
cm-1
3248.8
2935.9
2859.5
2360.5
1733.0
1453.0
1409.3
1365.2
1321.5
1301.7
1253.9
1232.2
1150.2
1124.9
1085.2
1063.7
1015.9
972.1
920.6
879.2
833.7
763.4
668.1
532.1
509.5