Electronic Supplementary Information for

Protecting Group Free Synthesis of Urea-linked Glycoconjugates: Efficient Synthesis of β-Urea glycosides in Aqueous Solution

Yoshiyasu Ichikawa,*[a] Takahiro Minami, Shohei Kusaba, Nobuyoshi Saeki, Yuta Tonegawa, Yumiko Tomita, Keiji Nakano,[a] Hiyoshizo Kotsuki[a], and Toshiya Masuda,[b]

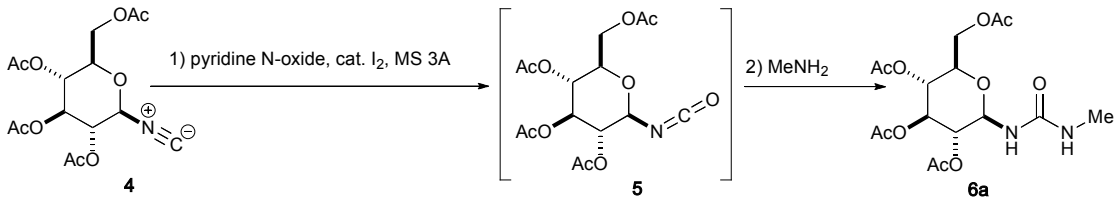
[a]Faculty of Science, Kochi University, Akebono-cho, Kochi 780-8520, Japan

[†]Faculty of Integrated Arts and Sciences, University of Tokushima, Tokushima 770-8502, Japan

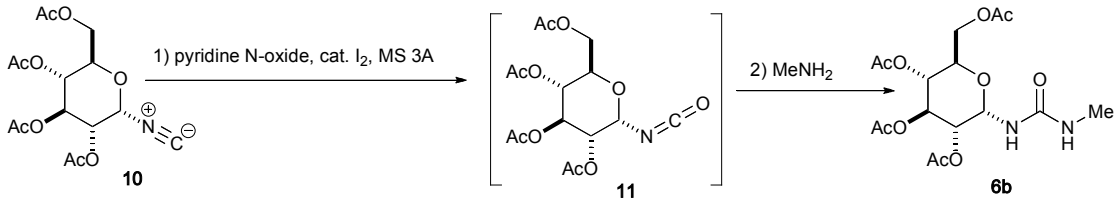
Tel: +81-88-844-8292, FAX: +81-88-844-8359

E-mail: ichikawa@kochi-u.ac.jp

General: Melting points (Mp) were recorded on a Yanaco MP-S3 melting point apparatus and were not corrected. Optical rotations were measured on a JASCO DIP-370 digital polarimeter. Infrared spectra (IR) were recorded on a JASCO FT/IR-460 spectrophotometer and were reported in wave number (cm^{-1}). Proton nuclear magnetic resonance (^1H NMR) spectra were recorded on JEOL LA 400 (400 MHz) spectrometers and JEOL LA 500 (500 MHz) spectrometers. Chemical shifts (δ) were reported in parts per million (ppm) relative to tetramethylsilane (TMS, δ 0.00, in $CDCl_3$), CD_3OD (δ 3.31), DOH (δ 4.80) as internal standards. Coupling constants (*J*) are given in Hz. Data were reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, br = broadened), coupling constant and assignment. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on JEOL LA 400 (100 MHz) spectrometers and JEOL LA 500 (125 MHz) spectrometers. Chemical shifts (δ) were reported in parts per million (ppm) relative to $CDCl_3$ (δ 77.0), CD_3OD (δ 49.9), DMSO (δ 40.5), and 1,4-dioxane (δ 67.19 in D_2O) as internal standards. Reactions were monitored by thin-layer chromatography on glass plates 0.25 mm coated with silica gel 60 F_{254} (MERCK 1.05715). Open-column chromatography was carried out with Silica gel 60 (particle size 0.063-0.200 mm, 70-230 mesh ASTM). Reactions were run under atmosphere of argon when the reactions were sensitive to moisture or oxygen. Dichloromethane, acetonitrile and toluene were stored over molecular sieves 3A. All other commercially available reagents were used as received.

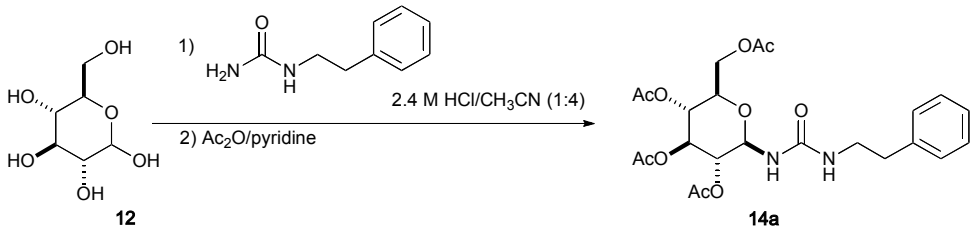


To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **4** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After being stirred at room temperature for 10 min, several drops of a solution methylamine in CH_2Cl_2 (prepared by extracting commercially available 40% aqueous methylamine with CH_2Cl_2 followed by drying on Na_2SO_4) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford *N*-methyl glucosylurea **6a** (21 mg, 90%) as a white solid: Mp 195−196 °C; $[\alpha]_D^{25}$ = +2.88 (*c* 1.00, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.02 (s, 3H), 2.03 (s, 3H), 2.05 (s, 3H), 2.08 (s, 3H), 2.76 (d, *J* = 4.5 Hz, 3H), 3.83 (ddd, *J* = 9.5, 4.5, 2.5 Hz, 1H), 4.10 (dd, *J* = 12.0, 2.5 Hz, 1H), 4.31 (dd, *J* = 12.0, 4.5 Hz, 1H), 4.72 (q, *J* = 4.5 Hz, 1H), 4.90 (t, *J* = 9.5 Hz, 1H), 5.07 (t, *J* = 9.5 Hz, 1H), 5.17 (t, *J* = 9.5 Hz, 1H), 5.31 (t, *J* = 9.5 Hz, 1H), 5.41 (d, *J* = 9.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.56, 20.71, 20.74, 26.9, 61.9, 68.3, 70.4, 72.8, 73.0, 80.1, 157.1, 169.6, 169.9, 170.6, 170.9.



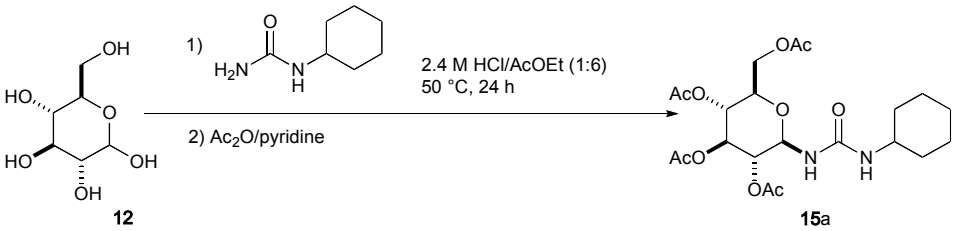
To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **10** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After

being stirred at room temperature for 10 min, several drops of a solution methylamine in CH_2Cl_2 was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford *N*-methyl glucosylurea **6b** (21 mg, 90%) as a white solid: Mp 204 ˚C (dec); $[\alpha]_D^{25}$ = +134.3 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3402, 3352, 2973, 2973, 2955, 2519, 2361, 1755 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.02 (s, 3H), 2.04 (s, 3H), 2.07 (s, 3H), 2.09 (s, 3H), 2.82 (d, *J* = 4.5 Hz, 3H), 4.10−4.15 (2H), 4.21 (dd, *J* = 12.0, 5.0 Hz, 1H), 5.04 (t, *J* = 10.0, 1H), 5.07 (dd, *J* = 10.0, 5.0 Hz, 1H), 5.32−5.37 (1H), 5.42 (t, *J* = 5.0 Hz, 1H); ^1H NMR (CD_3OD, 500 MHz) δ 2.00 (s, 6H), 2.01 (s, 3H), 2.03 (s, 3H), 2.71 (s, 3H), 3.89 (ddd, *J* = 10.0, 4.5, 2.0 Hz, 1H), 4.02 (dd, *J* = 12.0, 2.0 Hz, 1H), 4.27 (dd, *J* = 12.0, 4.5, 1H), 5.01 (t, *J* = 10.0, 1H), 5.01 (dd, *J* = 10.0, 5.5 Hz, 1H), 5.45 (t, *J* = 10.0 Hz, 1H), 5.70 (d, *J* = 5.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.5, 20.6, 26.7, 61.7, 66.9, 68.3, 68.9, 69.9, 76.6, 158.3, 169.2, 169.3, 170.1, 170.5. Anal. Calcd for $C_{16}H_{24}N_2O_{10}$: C, 47.52; H, 5.98; N, 6.93. Found: C, 47.43; H, 5.94; N, 6.78.



A solution of D-glucose **12** (300 mg, 1.67 mmol) and phenethylurea (547 mg, 3.33 mmol) in a mixture of CH_3CN (1.0 mL) and 2.4 N HCl (0.25 mL) was stirred at 50 °C for 1 day, and then was neutralized with solid $NaHCO_3$. The resulting reaction mixture was diluted with H_2O (ca. 2.0 mL) and washed with CH_2Cl_2. The separated aqueous layer was extracted with *n*-BuOH. The combined organic extracts were concentrated under reduced pressure to afford the solids.

The resulting crude product was dissolved in a mixture of pyridine (10 mL) and Ac_2O (5.0 mL). The solution was stirred at 50 °C for 3 hours, and diluted with saturated aqueous $NaHCO_3$. The separated aqueous layer was extracted with Et_2O. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:1 AcOEt/hexane) to afford phenetyl glucosylurea **14a** (520 mg, 63%, β:α = 90:10) as a white solid: Mp 58−59 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{26}$ = –1.11 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3367, 3028, 2942, 2358, 2338, 1750 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.01 (s, 3H), 2.03 (s, 3H), 2.04 (s, 3H), 2.07 (s, 3H), 2.75−2.85 (m, 2H), 3.38−3.50 (m, 2H), 3.79 (ddd, *J* = 10.5, 4.5, 2.0 Hz, 1H), 4.05 (dd, *J* = 12.5, 2.0 Hz, 1H), 4.32 (dd, *J* = 12.5, 4.5 Hz, 1H), 4.61 (t, *J* = 5.5 Hz, 1H), 4.88 (t, *J* = 9.5 Hz, 1H), 5.05 (t, *J* = 9.5 Hz, 1H), 5.13 (t, *J* = 9.5 Hz, 1H), 5.23 (d, *J* = 9.5 Hz, 1H) 5.30 (t, *J* = 9.5 Hz, 1H), 7.16−7.31 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.39, 20.49, 20.62, 20.94, 35.9, 41.4, 60.3, 61.7, 68.2, 70.4, 72.9, 80.0, 126.3, 128.4, 128.6, 138.7, 156.3, 169.5, 169,7, 170.5, 170.7; Anal. Calcd for $C_{23}H_{30}N_2O_{10}$: C, 55.86; H, 6.12; N, 5.67. Found: C, 55.71; H, 5.99; N, 5.62.



A solution of D-glucose **12** (300 mg, 1.67 mmol) and cyclohexylurea (474 mg, 3.33 mmol) in a mixture of AcOEt (1.5 mL) and 2.4 N HCl (0.25 mL) was stirred at 50 °C for 1 day. The reaction mixture was neutralized with solid $NaHCO_3$, diluted with H_2O (ca. 2.0 mL) and filtered to afford an aqueous filtrate and a solid. This solid, comprising urea glycoside and cyclohexylurea, was washed with CH_2Cl_2 to remove cyclohexylurea. The aqueous filtrate was washed with CH_2Cl_2 to remove cyclohexylurea and then extracted with *n*-BuOH. The combined *n*-BuOH extracts were concentrated under reduced pressure to afford crude urea glycoside. The two solids of crude urea glycoside were combined and dissolved in a mixture of pyridine (10 ml) and Ac_2O (5.0 ml). The resulting solution was stirred at 50 °C for 3 hours, and then treated with saturated aqueous $NaHCO_3$. The separated aqueous layer was extracted with Et_2O, and the combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (1:1 AcOEt/hexane) to afford cyclohexyl glucosylurea **15a** (537 mg, 68%, $\beta:\alpha \geq 98:2$) as a white solid; Mp 78–79 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{25} = -1.24$ (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3361, 2935, 2856, 2359,1753 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.03−1.20, 1.28−1.38, 1.56−1.62, 1.65−1.72, 1.86−1.92 (10H), 2.01 (s, 3H), 2.02 (s, 3H), 2.05 (s, 3H), 2.07 (s, 3H), 3.44−3.53 (1H), 3.82 (ddd, *J* = 10.0, 4.5, 2.0 Hz, 1H), 4.08 (dd, *J* = 12.5, 2.0 Hz, 1H), 4.32 (dd, *J* = 12.5, 4.5 Hz, 1H), 4.56 (d, *J* = 8.0 Hz, 1H), 4.90 (t, *J* = 9.5 Hz, 1H), 5.06 (t, *J* = 9.5 Hz, 1H), 5.15 (t, *J* = 9.5 Hz, 1H), 5.27 (d, *J* = 9.0 Hz, 1H), 5.30 (t, *J* = 9.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.44, 20.47, 20.6, 24.7, 25.4, 33.43, 33.45, 48.9, 61.8, 68.3, 70.4, 72.9, 80.0, 155.7, 169.5, 169.8, 170.6, 170.7. HRMS(ESI): m/z calcd for $C_{21}H_{33}N_2O_{10}$ $[M+H]^+$ 473.2155, found 473.2141; m/z calcd for $C_{21}H_{32}N_2O_{10}Na$ $[M+Na]^+$ 495.1955, found 495.1934.

Process for the work-up of cyclohexylurea glucoside (**15a**)

Reaction mixture (solution and precipitate)

Neutralized with solid $NaHCO_3$

add H_2O (ca. 2.0 mL)

Filtered

Solids
(a mixture of cyclohexylurea and urea glucoside)

washed with CH_2Cl_2 to remove cyclohexylurea

Filtrate (aqueous layer)

washed with CH_2Cl_2 to remove cyclohexylurea

Extracted with *n*-BuOH

Combined *n*-BuOH extracts

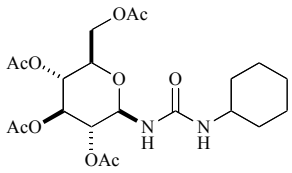
concentrated

Solids (urea glucoside)

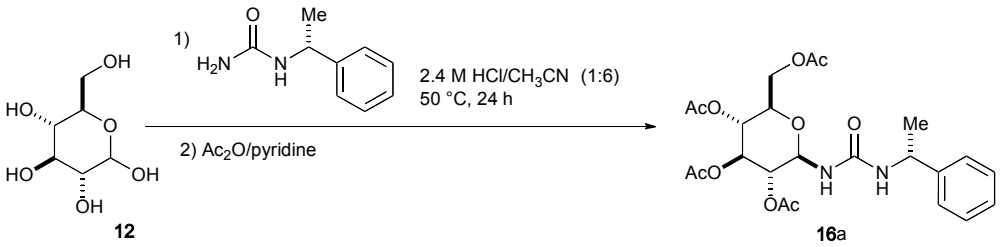
combined

Treated with Ac_2O/ pyridine

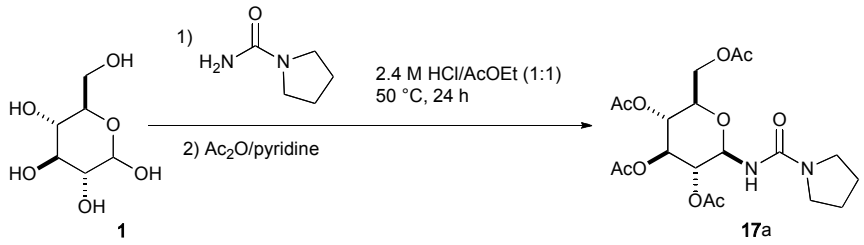
work-up



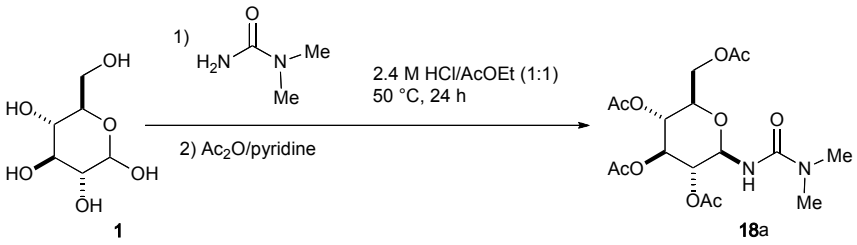
15a



A solution of D-glucose **12** (300 mg, 1.67 mmol) and (*R*)-methylbenzylurea (546 mg, 3.33 mmol) in a mixture of CH_3CN (1.5 mL) and 2.4 N HCl (0.25 mL) was stirred at 50 °C for 1 day. The reaction mixture was neutralized with solid $NaHCO_3$, diluted with H_2O (ca. 2.0 mL) and filtered to afford an aqueous filtrate and a solid. This solid, comprising urea glycoside and (*R*)-methylbenzylurea, was washed with CH_2Cl_2 to remove (*R*)-methylbenzylurea. The aqueous filtrate was washed with CH_2Cl_2 to remove (*R*)-methylbenzylurea and then extracted with *n*-BuOH. The combined *n*-BuOH extracts were concentrated under reduced pressure to afford crude urea glycoside. The two solids of crude urea glycoside were combined and dissolved in a mixture of pyridine (10 ml) and Ac_2O (5.0 ml). The resulting solution was stirred at 50 °C for 3 hours, and then treated with saturated aqueous $NaHCO_3$. The separated aqueous layer was extracted with Et_2O, and the combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:3 AcOEt/hexane) to afford (*R*)-methylbenzylurea glucoside **16a** (594 mg, 72%, $\beta:\alpha \geq 98:2$) as a white solid; Mp 132−133 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{24}$ = +2.76 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3361, 3061, 3030, 2971, 2359, 2341, 1752 $cm^{-1;}$ ^1H NMR ($CDCl_3$, 500 MHz) δ 1.45 (d, *J* = 7.0 Hz, 3H) 1.97 (s, 3H), 1.99 (s, 3H), 2.02 (s, 3H), 2.05 (s, 3H), 3.75 (ddd, *J* = 10.0, 4.0, 2.0 Hz, 1H), 4.03 (dd, *J* = 12.5, 2.5 Hz, 1H), 4.32 (dd, *J* = 12.5, 4.5 Hz, 1H), 4.79−4.86 (br, 1H), 4.86 (t, *J* = 9.5 Hz, 1H), 4.97 (d, *J* = 7.5 Hz, 1H), 5.03 (t, *J* = 9.5 Hz, 1H), 5.13 (t, *J* = 9.5 Hz, 1H), 5.24 (d, *J* = 9.5 Hz, 1H) 5.26 (t, *J* = 9.5 Hz, 1H), 7.23−7.35 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.47, 20.48, 20.54, 20.59, 22.9, 49.7, 61.7, 68.2, 70.3, 72.9, 73.0, 79.9, 125.7, 127.2, 128.5, 143.5, 155.8, 169.6, 169,8, 170.57, 170.63. HRMS(ESI): m/z calcd for $C_{23}H_{31}N_2O_{10}$ $[M+H]^+$ 495.1979, found 495.1991, m/z calcd for $C_{23}H_{31}N_2O_{10}Na$ $[M+Na]^+$ 517.1798, found 517.1790.

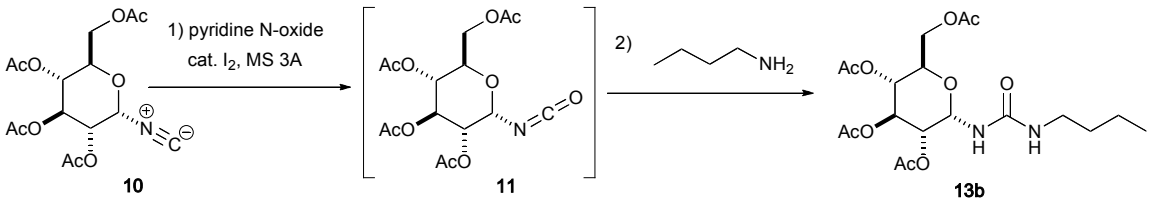


A solution of D-glucose **12** (300 mg, 1.67 mmol) and pyrrolidine urea (1.90 g, 16.7 mmol) dissolved in AcOEt (2.0 mL) and 2.4 N HCl (2.0 mL) was stirred at 50 °C for 1 day. The solution was neutralized with solid $NaHCO_3$, diluted with H_2O (ca. 2.0 mL) and filtered to afford an aqueous filtrate and a solid. The separated aqueous filtrate was washed with CH_2Cl_2 to remove excess pyrrolidine urea and then extracted with *n*-BuOH. The combined *n*-BuOH layer was concentrated under reduced pressure to afford crude solids, which were dissolved in pyridine (10 mL) and Ac_2O (5.0 mL). The solution was stirred at 50 °C for 3 hours, and then treated with saturated aqueous $NaHCO_3$. The separated aqueous layer was extracted with Et_2O, and the combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford pyrrolidine urea glucoside **17a** (303 mg, 40%, β:α = 95:5) as a white solid; Mp 184−185 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{24}$ = –2.20 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3361, 2935, 2856, 2359, 1753 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.85−1.93 (4H), 2.02 (s, 3H), 2.03 (s, 3H), 2.05 (s, 3H), 2.08 (s, 3H), 3.18−3.39 (4H), 3.83 (ddd, *J* = 10.5, 4.0, 2.5 Hz, 1H), 4.08 (dd, *J* = 12.5, 2.5 Hz, 1H), 4.33 (dd, *J* = 12.5, 4.5 Hz, 1H), 4.92 (t, *J* = 9.5 Hz, 1H), 5.07 (t, *J* = 9.5 Hz, 1H), 5.18 (t, *J* = 9.5 Hz, 1H), 5.28 (d, *J* = 9.0 Hz, 1H), 5.32 (t, *J* = 9.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.4, 20.6, 20.7, 20.8, 25.2, 45.2, 61.5, 68.1, 70.5, 72.6, 72.8, 80.1, 154.3, 169.4, 169.6, 170.4, 171.1. HRMS(ESI): m/z calcd for $C_{19}H_{29}N_2O_{10}$ $[M+H]^+$ 445.1822, found 445.1815; m/z calcd for $C_{19}H_{28}N_2O_{10}Na$ $[M+Na]^+$ 467.1642, found 462.1619.

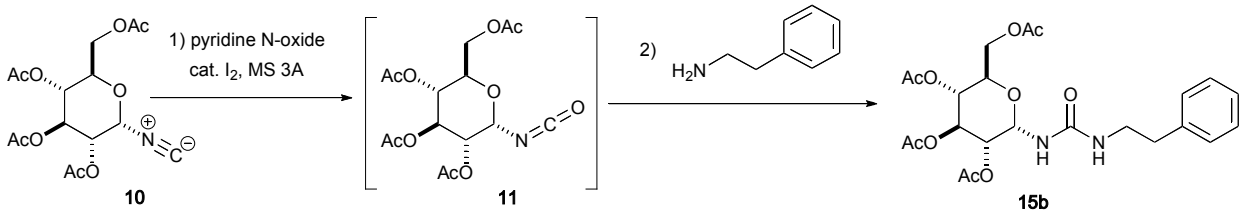


To a mixture of D-glucose **12** (300 mg, 1.67 mmol) and *N,N*-dimethyl urea (1.47 g, 16.7 mmol) in AcOEt (1.5 mL) was added 2.4 N HCl (1.5 mL). After being stirred at 50 °C for 1 day. The solution was neutralized with solid $NaHCO_3$, diluted with H_2O (ca. 2.0 mL) and washed with CH_2Cl_2. The separated aqueous layer was extracted with *n*-BuOH, and the combined *n*-BuOH layer was concentrated under reduced pressure to afford crude solids, which were dissolved in a mixture of pyridine (10 mL) and Ac_2O (5.0 mL). After stirring at 50 °C for 3 hours, the resulting reaction mixture was diluted with saturated aqueous $NaHCO_3$ and extracted with Et_2O. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford *N,N*-dimethyl glucosylurea **18a** (356 mg, 51%, β:α = 97:3) as a white solid; Mp 165−166 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{24}$ = –0.82 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3407, 2945, 2359, 1749, 1661 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.02 (s, 3H), 2.03 (s, 3H), 2.06 (s, 3H), 2.08 (s, 3H), 2.87 (s , 6H), 3.83 (ddd, *J* = 10.0, 4.0, 2.0 Hz, 1H), 4.08 (dd, *J* = 12.5, 2.0 Hz, 1H), 4.34 (dd, *J* = 12.5, 4.0 Hz, 1H), 4.92 (t, *J* = 9.5 Hz, 1H), 5.07 (t, *J* = 9.5 Hz, 1H), 5.16 (t, *J* = 9.5 Hz, 1H), 5.32 (t, *J* = 9.5 Hz, 1H), 5.47 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.3, 20.4, 20.5, 35.7, 61.4, 68.0, 70.5, 72.6, 72.7, 80.3, 156.1, 169.3, 169,5, 170.3, 171.0.

Anal. Calcd for $C_{17}H_{26}N_2O_{10}$: C, 48.80; H, 6.26; N, 6.70. Found: C, 48.84; H, 6.19; N, 6.69.

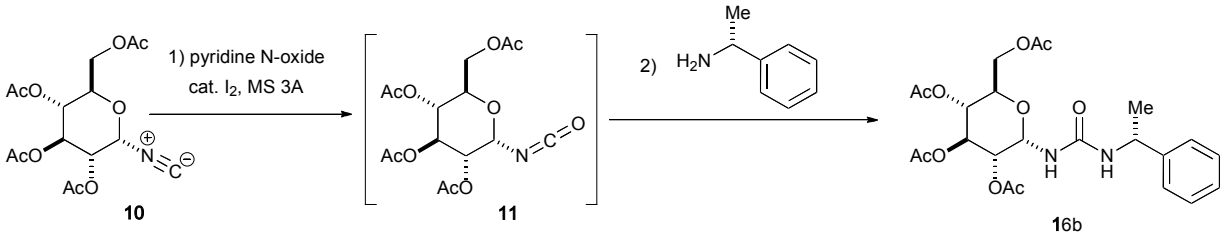


To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **10** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After being stirred at room temperature for 10 min, *n*-butylamine (0.017 mg, 0.17 mmol) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:1 AcOEt/hexane) to afford *n*-butylurea glucoside **13b** (22 mg, 88%) as a white solid; Mp 154−155 °C; $[\alpha]_D^{24}$ = +129.7 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3370, 3287, 2962, 2875, 1746 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.93 (t, *J* = 7.5 Hz, 3H), 1.35 (sept, *J* = 7.5 Hz, 2H), 1.50 (quint, *J* = 7.5 Hz, 2H), 2.02 (s, 3H), 2.04 (s, 3H), 2.08 (s, 3H), 2.09 (s, 3H), 3.17−3.29 (m,2H), 4.10 (dd, *J* = 12.0, 2.0 Hz, 1H), 4.12−4.16 (m, 1H), 4.23 (dd, *J* = 12.0, 4.5 Hz, 1H), 5.05 (t, *J* = 10.0, 1H), 5.07 (dd, *J* = 10.0, 5.0 Hz, 1H), 5.10 (d, *J* = 3.0 Hz, 1H), 5.30−5.33 (1H), 5.32 (t, *J* = 10.0 Hz, 1H), 5.40 (dd, *J* = 5.0, 3.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.7, 19.9, 20.51, 20.56, 20.66, 32.0, 39.9, 61.7, 66.9, 68.1, 68.8, 69.7, 157.5, 169.2, 169.3, 169.9, 170.4. HRMS(ESI): m/z calcd for $C_{19}H_{30}N_2O_{10}Na$ $[M+Na]^+$ 469.1798, found 469.1787.



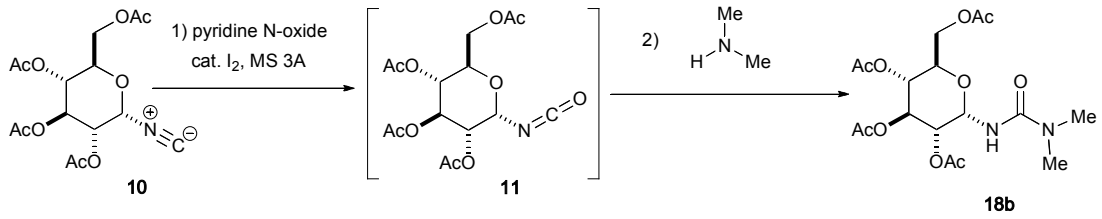
To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **10** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After

being stirred at room temperature for 10 min, phenethylamine (0.022 mg, 0.17 mmol) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:1 AcOEt/hexane) to afford phenethylurea glucoside **15b** (25 mg, 87%) as a white solid; Mp 154−155 °C; $[\alpha]_D^{25}$ = +130.4 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3396, 3280, 3084, 3063, 3028, 2952, 2891, 2362, 2344, 1742 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.01 (s, 3H), 2.02 (s, 3H), 2.06 (s, 3H), 2.07 (s, 3H), 2.81 (quint, *J* = 14.0, 7.0 Hz, 1H), 2.87 (quint, *J* = 14.0, 7.0 Hz, 1H), 3.44−3.54 (m, 2H), 3.73 (dd, *J* = 12.5, 2.0 Hz, 1H), 4.01 (ddd, *J* = 10.5, 4.0, 2.0 Hz, 1H), 4.16 (dd, *J* = 12.5, 4.0 Hz, 1H), 5.03 (t, *J* = 10.0 Hz, 1H), 5.05 (dd, *J* = 10.0, 5.5 Hz, 1H), 5.16 (d, *J* = 3.5 Hz, 1H), 5.30 (t, *J* = 10.0 Hz, 1H), 5.30−5.33 (1H), 5.34 (dd, *J* = 5.5, 3.5 Hz, 1H), 7.17−7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.43, 20.47, 20.58, 35.8, 41.3, 61.4, 66.7, 67.9, 68.7, 69.7, 76.9, 126.4, 128.4, 128.5, 138.6, 157.5, 169.1, 169.2, 169,9, 170.3. Anal. Calcd for $C_{23}H_{30}N_2O_{10}$: C, 55.86; H, 6.12; N, 5.67. Found: C, 55.60; H, 5.86; N, 5.67.



To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **10** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After being stirred at room temperature for 10 min, (*R*)-methylbenzyl amine (0.022 mg, 0.17 mmol) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and

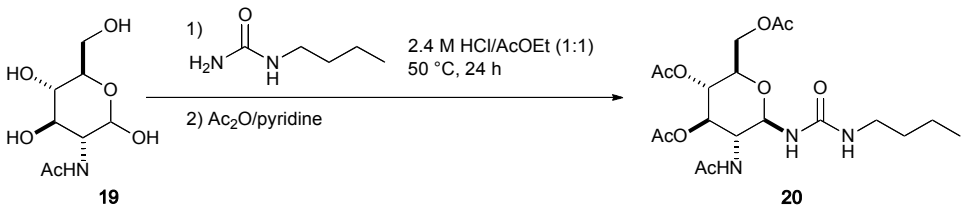
then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:3 AcOEt/hexane) to afford (*R*)-methylbenzyl glucosylurea **16b** (21 mg, 77%) as a white solid; Mp 118−119 °C; $[\alpha]_D^{24}$ = +1.13 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3480, 3086, 3062, 3030, 2977, 2937, 2901, 2875, 2728, 2480, 2405, 2263, 2121, 1750 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.49 (d, *J* = 7.0 Hz, 3H) 1.91 (s, 3H), 2.02 (s, 6H), 2.09 (s, 3H), 3.90 (brd, *J* = 12.5 Hz, 1H), 4.16 (dd, *J* = 12.5, 4.5 Hz, 1H), 4.92−4.97 (br, 1H), 5.02 (t, *J* = 10.0 Hz, 1H), 5.06 (dd, *J* = 9.0, 4.5 Hz, 1H), 5.17 (d, *J* = 3.5 Hz, 1H), 5.23−5.29 (br, 1H), 5.49 (t, *J* = 4.5 Hz, 1H) 5.57 (d, *J* = 7.0 Hz, 1H), 7.24−7.36 (m, 5H); ^1H NMR (CD_3OD, 500 MHz) δ 1.43 (d, *J* = 7.0 Hz, 3H) 1.90 (s, 3H), 1.99 (s, 3H), 2.01 (s, 3H), 2.03 (s, 3H), 3.85−3.93 (br, 1H), 4.04 (brd, *J* = 12.0 Hz, 1H), 4.27 (dd, *J* = 12.0, 4.5 Hz, 1H), 4.80−4.85 (br, 1H), 4.99 (dd, *J* = 10.0, 5.5 Hz, 1H), 5.01 (t, *J* = 10.0 Hz, 1H), 5.40 (t, *J* = 10.0 Hz, 1H), 5.67 (d, *J* = 5.5 Hz, 1H), 7.19−7.33 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.5, 22.7, 49.7, 61.4, 66.9, 68.0, 68.7, 69.8, 125.7, 127.3, 128.7, 143.7, 156.6, 169.2, 169.3, 170.0, 170.5. HRMS(ESI): m/z calcd for $C_{23}H_{31}N_2O_{10}$ $[M+H]^+$ 495.1979, found 495.2000; m/z calcd for $C_{23}H_{31}N_2O_{10}Na$ $[M+Na]^+$ 517.1798, found 517.1798.



To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **10** (20 mg, 0.056 mmol) and iodine (1.0 mg, 7.0 mol%). After being stirred at room temperature for 10 min, several drops of a solution *N,N*-dimethyl amine in CH_2Cl_2 (prepared by extraction of 40% aqueous methylamine with CH_2Cl_2 followed by drying on Na_2SO_4) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting

residue was purified by silica gel chromatography (AcOEt) to afford *N,N*-dimethyl glucosylurea **18b** (19 mg, 81%) as a white solid; Mp 69−70 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{24}$ = +73.9 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3416, 2961, 1752 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.03 (s, 3H), 2.04 (s, 6H), 2.08 (s, 3H), 2.98 (s , 6H), 4.00 (ddd, *J* = 9.5, 3.5, 2.5 Hz, 1H), 4.07 (dd, *J* = 12.5, 2.5 Hz, 1H), 4.34 (dd, *J* = 12.5, 3.5 Hz, 1H), 5.05 (t, *J* = 9.5 Hz, 1H), 5.10 (t, *J* = 6.0 Hz, 1H), 5.21 (dd, *J* = 9.5, 5.5 Hz, 1H), 5.28 (t, *J* = 9.5 Hz, 1H), 5.81 (t, *J* = 5.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.54, 20.56, 20.58, 20.7, 36.2, 61.6, 67.0, 68.4, 70.3, 76.0, 156.6, 168.8, 169.3, 170.3, 170.7. Anal. Calcd for $C_{17}H_{26}N_2O_{10}$: C, 48.80; H, 6.26; N, 6.70. Found: C, 48.68; H, 6.12; N, 6.66.

General Method A for the synthesis of β-urea glucosamides starting from N-acetyl-D-glucosamine (**19**)



A solution of *n*-butylurea (879 mg, 7.56 mmol) dissolved in a mixture of AcOEt (1.00 mL) and 2.4 N HCl (1.0 mL) was stirred at 50 °C for 10 min to result in a clear homogeneous solution. To this solution was added *N*-acetyl-D-glucosamine **19** (335 mg, 1.51 mmol) dissolved in 2.4 N HCl (0.50 mL), and the reaction mixture was heated at 50 °C for one day. The solution was treated with solid $NaHCO_3$ (500 mg) and water (1.0 mL), and filtered to afford an aqueous filtrate and a solid. The solid, comprising *n*-butylurea glycoside and *n*-butylurea, was washed with CH_2Cl_2 to remove *n*-butylurea. The aqueous filtrate was washed with CH_2Cl_2 (×4) and then extracted with *n*-BuOH (×6). The combined *n*-BuOH extracts were concentrated under reduced pressure to afford crude *n*-butylurea glycoside. The resulting two portions of crude *n*-butylurea glycoside were combined and dissolved in a mixture of pyridine (10 mL) and Ac_2O (5.0 mL). The resulting solution was stirred at room temperature for 12 hours, and then poured into water. After stirring for 30 minutes, the aqueous solution was extracted with AcOEt (×4). The combined extracts were washed with 1M aqueous $KHSO_4$, water and saturated aqueous $NaHCO_3$, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting solid (397 mg) was purified by recrystallization from hot AcOEt and hexane (55 mL AcOEt and 6 mL hexane, 1st crops, 171 mg; 27 mL AcOEt and 5 mL hexane, 2nd crops 64 mg). The residue obtained after recrystallization was purified by silica gel chromatography (25:1 CH_2Cl_2/ MeOH) to afford additional *n*-butylurea glycoside (97 mg, combined yield, 332 mg, 49%); Mp

154–155 °C; $[\alpha]_D^{25}$ –11.9 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3330, 1747, 1653, 1568, 1242 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.90 (t, *J* = 7.5 Hz, 3H), 1.26–1.36 (m, 2H), 1.40–1.50 (m, 2H), 1.88 (s, 1H), 1.95 (s, 3H), 2.04 (s, 3H), 2.06 (s, 3H), 2.08 (s, 3H), 3.07–3.19 (m, 2H), 3.77 (ddd, *J* = 9.0, 4.0, 2.0 Hz, 1H), 4.08 (dd, *J* = 12.0, 2.0 Hz, 1H), 4.12 (t, *J* = 9.0 Hz, 1H), 4.32 (dd, *J* = 12.0, 4.0 Hz, 1H), 4.85 (t, 5.5 Hz, 1H), 5.03–5.14 (m, 3H), 5.98 (d, *J* = 7.5 Hz, 1H), 6.33 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.7, 19.9, 20.62, 20.73, 20.79, 23.2, 31.9, 40.0, 53.4, 61.9, 67.9, 73.0, 73.1, 81.8, 156.7, 169.3, 170.7, 171.6, 171.9. HRMS(ESI): m/z calcd for $C_{19}H_{31}N_3O_9Na$ $[M+Na]^+$ 468.1958, found 468.1952.

Work-up process of *n*-butylurea glucosamide (**20)**

Reaction mixture (solution and precipitate)

Neutralized with solid $NaHCO_3$

add H_2O (ca. 1.0 mL)

CH_4

Solids (a mixture of *n*-butylurea and urea glucosamide)

Filtrate (aqueous layer)

washed with CH_2Cl_2 to remove *n*-butylurea

Extracted with *n*-BuOH

washed with CH_2Cl_2 to remove *n*-butylurea

Combined *n*-BuOH extracts

concentrated

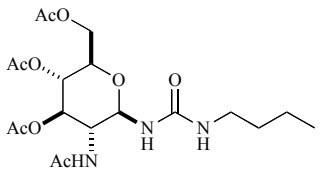
Solids (urea glucosamide)

Solids (urea glucosamide)

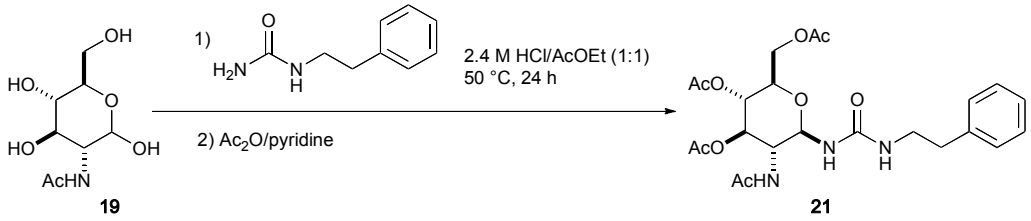
combined

Treated with Ac_2O and pyridine

work-up

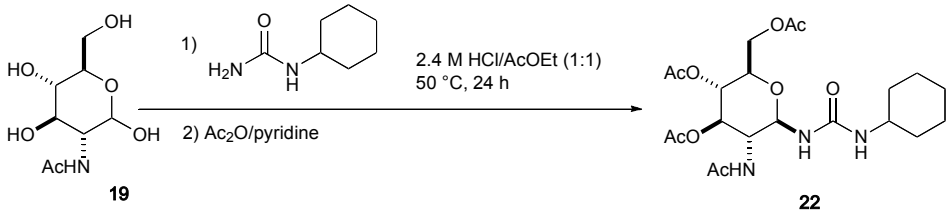


20



N-Acetyl-D-glucosamine **19** (335 mg, 1.51 mmol) was transformed into phenylethylurea glucosamide **21** (333 mg, 45%) by employing Method A starting β-phenylethylurea (1.20 g, 7.56 mmol), AcOEt (1.00 mL) and 2.4 N HCl (1.0 mL), pyridine (10 mL) and Ac_2O (5.0 mL) ; Mp 246 °C (dec.); $[\alpha]_D^{25}$ –10.5 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3333, 1747, 1653, 1567, 1238 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.91 (s, 3H), 1.96 (s, 1H), 2.03 (s, 3H), 2.05 (s, 3H), 2.08 (s, 3H), 2.72–2.80 (m, 2H), 3.30–3.45 (m, 2H), 3.76 (br, 1H), 4.07 (dd, *J* = 12.0, 2.0 Hz, 1H), 4.12 (t, *J* = 7.5 Hz, 1H), 4.32 (dd, *J* = 12.0, 4.5 Hz, 1H), 5.02–5.14 (m, 3H), 6.05 (d, *J* = 9.0 Hz, 1H), 6.41 (d, *J* = 9.0 Hz, 1H), 7.13–7.23 (m, 3H), 7.25–7.30 (m, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.6, 20.72, 20.79, 23.1, 36.1, 41.5, 53.4, 61.8, 67.9, 73.06, 73.11, 81.7, 126.4, 128.53, 128.69, 138.7, 156.7, 169.3, 170.7, 171.63, 171.89. HRMS(ESI): m/z calcd for $C_{23}H_{32}N_3O_9$ $[M+H]^+$ 494.2139, found 494.2136; m/z calcd for $C_{23}H_{31}N_3O_9Na$ $[M+Na]^+$ 516.1958, found 516.1953.

General Method B for the synthesis of β-urea glucosamides starting from N-acetyl-D-glucosamine (**19**)



A solution of cyclohexylurea (1.10 g, 7.56 mmol) dissolved in a mixture of AcOEt (1.00 mL) and 2.4 N HCl (0.50 mL) was stirred at 50 °C for 30 min to result in a clear homogeneous solution. To this solution was added *N*-acetyl-D-glucosamine **19** (335 mg, 1.51 mmol) dissolved in 2.4 N HCl (0.50 mL), and the reaction mixture was heated at 50 °C for one day. The solution was treated with solid $NaHCO_3$ (500 mg) and water (1.0 mL), and filtered to afford an aqueous filtrate and a solid. This solid, comprising urea glycoside and cyclohexylurea, was washed with CH_2Cl_2, and suspended in AcOEt (50 mL). The suspension was heated at refluxed for 30 min, and filtered. This procedure was repeated more two times to remove cyclohexylurea, giving crude urea glycoside. The aqueous filtrate was washed with CH_2Cl_2 (×4) and then extracted with *n*-BuOH (×6). The combined *n*-BuOH extracts were concentrated under reduced pressure to afford crude urea glycoside.

The two portions of crude urea glycoside were combined and dissolved in a mixture of pyridine (10 mL) and Ac_2O (5.0 mL). The resulting solution was stirred at room temperature for 12 hours, and then poured into water. After stirring for 30 minutes, the aqueous solution was extracted with AcOEt (×4). The combined extracts were washed with 1M aqueous $KHSO_4$, water and saturated aqueous $NaHCO_3$, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting solid (1.56 g) was purified by recrystallization from hot AcOEt (200 mL AcOEt, 1st crops, 353 mg; 200 mL AcOEt, 2nd crops 50 mg; 70 mL AcOEt, third crops 6

mg) to afford cyclohexylurea glucosamide **22** (combined yield, 409 mg, 57%): Mp 277 °C (dec.); $[\alpha]_D^{25}$ –9.3 (*c* 0.25, $CHCl_3$); IR (KBr) ν_{max} 3327, 1747, 1650, 1564, 1234 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.00–1.20 (m, 3H), 1.23– 1.39 (m, 2H), 1.59 (br, 1H), 1.68 (br, 1H), 1.77 (s, 3H), 1.88 (br, 2H), 1.95 (s, 3H), 2.04 (s, 3H), 2.06 (s, 3H), 2.09 (s, 3H), 3.49 (br, 1H), 3.76 (ddd, *J* = 10.0, 4.0, 2.0 Hz, 1H), 4.05–4.15 (m, 2H), 4.31 (dd, *J* = 12.0, 4.0 Hz, 1H), 4.56 (d, *J* = 9.0 Hz, 1H), 5.03 (t, *J* = 9.0 Hz, 1H), 5.06 (t, *J* = 9.0 Hz, 1H), 5.12 (t, *J* = 9.0 Hz, 1H), 5.86 (d, *J* = 8.0 Hz, 1H), 6.18 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.6, 20.7, 20.8, 23.2, 24.8, 25.4, 33.7, 53.6, 61.8, 67.7, 73.1, 82.2, 155.7, 169.3, 170.7, 171.8, 172.0. Anal. Calcd for $C_{21}H_{33}N_3O_9$: C, 53.49; H, 7.05; N, 8.91. Found: C, 53.19; H, 7.18; N, 8.76. HRMS(ESI): m/z calcd for $C_{21}H_{34}N_3O_9$ $[M+H]^+$ 472.2295, found 472.2299; m/z calcd for $C_{21}H_{33}N_3O_9Na$ $[M+Na]^+$ 494.2114, found 494.2122.

Work-up process of cyclohexylurea glucosamide (**22**)

Reaction mixture (solution and precipitate)

Neutralized with solid $NaHCO_3$

add H_2O (ca. 1.0 mL)

Filtered

Solids
(a mixture of cyclohexylurea and urea glucosamide)

washed with CH_2Cl_2 to remove cyclohexylureaurea

Suspended in AcOEt
Heated at reflux for 30 min

Filtered

three cycles

Solids (urea glucosamide)

Filtrate (aqueous layer)

washed with CH_2Cl_2 to remove cyclohexylurea

Extracted with *n*-BuOH

Combined *n*-BuOH extracts

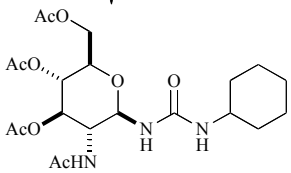
concentrated

Solids (urea glucosamide)

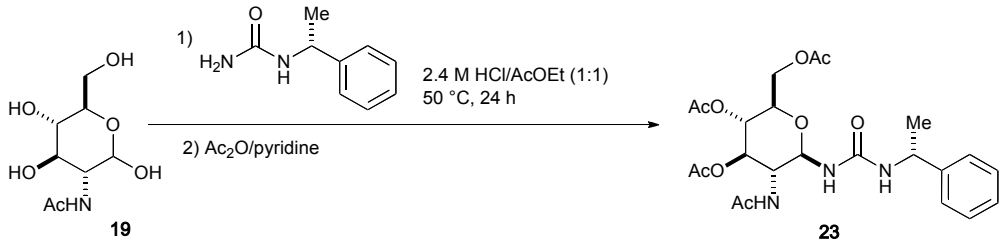
combined

Treated with Ac_2O and pyridine

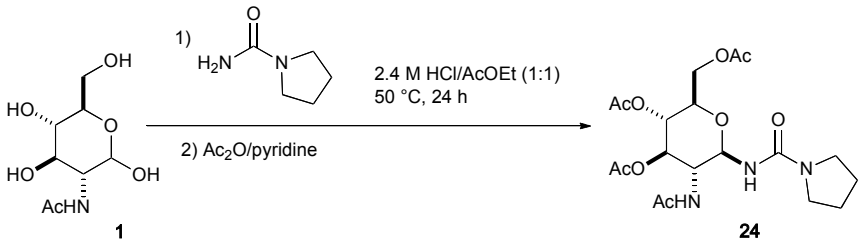
work-up



22

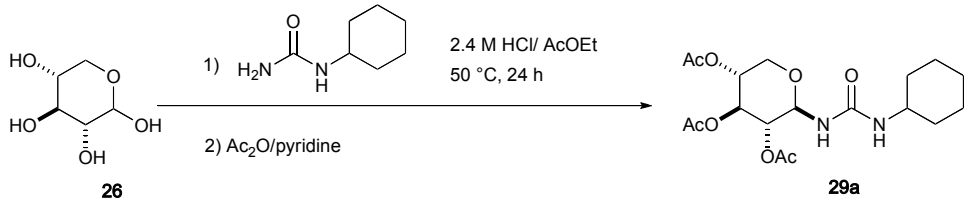


N-Acetyl-D-glucosamine **19** (335 mg, 1.51 mmol) was transformed into (*R*)-α-methylbenzylurea glycosamide **23** a white solid (361 mg, 48%) by employing Method A starting from (*R*)-α-methylbenzylurea (1.24 g, 7.56 mmol), AcOEt (1.50 mL), 2.4 N HCl (1.5 mL), pyridine (10 mL) and Ac_2O (5.0 mL); Mp 270 °C (dec.); $[\alpha]_D^{25}$ +13.4 (*c* 0.50, $CHCl_3$); IR (KBr) ν_{max} 3333, 3276, 1743, 1650, 1566, 1234 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.44 (d, *J* = 7.0 Hz, 3H), 1.68 (s, 3H), 1.96 (s, 1H), 2.03 (s, 3H), 2.05 (s, 3H), 2.06 (s, 3H), 3.71 (ddd, *J* = 9.5, 3.5, 2.0, 1H), 4.04 (dd, *J* = 12.0, 2.0 Hz, 1H), 4.10 (t, *J* = 9.5 Hz, 1H), 4.30 (dd, *J* = 12.0, 4.5 Hz, 1H), 4.86 (br, 1H), 4.98–5.07 (m, 3H), 5.11 (t, 9.5 Hz, 1H), 5.95 (d, *J* = 8.0 Hz, 1H), 6.06 (d, *J* = 8.0 Hz, 1H), 7.21–7.34 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.24, 20.73, 20.79, 22.8, 23.2, 49.8, 53.5, 61.8, 67.6, 73.07, 73.11, 82.3, 125.9, 127.2, 128.6, 148.5, 155.9, 169.2, 170.7, 171.8, 172.0. HRMS(ESI): m/z calcd for $C_{23}H_{32}N_3O_9$ $[M+H]^+$ 494.2139, found 494.2133; m/z calcd for $C_{23}H_{31}N_3O_9Na$ $[M+Na]^+$ 516.1958, found 516.1960.



N-Acetyl-D-glucosamine **19** (335 mg, 1.51 mmol) was transformed into pyrrolidineurea glucosamide **24** as a white solid (34 mg, 5%) by employing Method B starting from pyrrolidineurea (864 mg, 7.56 mmo), AcOEt (1.0 mL), 2.4 N HCl (1.0) , pyridine (10 mL) and Ac_2O (5.0 mL); Mp 181–182 °C; $[\alpha]_D^{25}$ –22.8 (*c* 0.77, $CHCl_3$); IR (KBr or NaCl) ν_{max} 3379, 3298, 2958, 1748, 1649, 1523, 1243 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.80 (br, 4H), 1.81 (s, 3H), 1.90 (s, 3H), 2.05 (s, 3H), 2.10 (s, 3H), 3.26 (br, 4H), 3.71 (br, 1H), 4.02 (dd, *J* = 13.0, 2.0 Hz, 1H), 4.08 (q, *J* = 9.5 Hz, 1H), 4.24 (dd, *J* = 13.0, 4.0 Hz, 1H), 4.95–5.04 (m, 2H), 5.07 (t, *J* = 9.5 Hz, 1H), 5.99 (d, *J* = 7.5 Hz, 1H), 6.18 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.6, 20.7, 20.8, 23.2, 25.3, 45.4, 53.4, 61.8, 67.7, 72.8, 73.0, 82.2, 155.0, 169.2, 170.7, 171.9, 172.0. HRMS(ESI): m/z calcd for $C_{19}H_{30}N_3O_9$ $[M+H]^+$ 444.1982, found 444.1983; m/z calcd for $C_{19}H_{29}N_3O_9Na$ $[M+Na]^+$ 466.1801, found 466.1796.

General method for the synthesis of urea xyloside starting from D-xylose (26)



26 **29a**

A solution of D-xylose (**26**) (300 mg, 2.00 mmol) and cyclohexylurea (570 mg, 3.99 mmol) in a mixture of AcOEt (1.80 mL) and 2.4 N HCl (0.30 mL) was stirred at 50 °C for 1 day. The precipitate, containing a mixture of urea xyloside and cyclohexylurea, was filtered, and washed with AcOEt to afford urea xyloside as a solid. The filtrate was neutralized with solid sodium bicarbonate, diluted with H_2O (1.0 mL), washed with CH_2Cl_2 and extracted with *n*-BuOH. The *n*-BuOH extracts were concentrated to afford urea xyloside as a solid. The two solids of urea xyloside were combined, and dissolved in a mixture of pyridine (10 mL) and Ac_2O (5.0 mL). The solution was stirred at 50 °C for 3 hours, and diluted with saturated aqueous $NaHCO_3$, The aqueous layer was extracted with CH_2Cl_2, and the combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue (1.56 g) was purified by silica gel chromatography (2:1 AcOEt/ hexane) to afford β-cyclohexylurea xyloside **29a** (713 mg, 89%) exclusively as a white solid; Mp 217–218 °C; $[\alpha]_D^{25}$ –3.82 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3331, 2933, 2852, 1748, 1651, 1561 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.06–1.19 (m, 3H), 1.29–1.41 (m, 2H), 1.55–1.71 (m, 3H), 1.85–1.95 (m, 2H), 2.03 (s, 6H), 2.07 (s, 3H), 3.44 (t, *J* = 11.0, Hz, 1H), 3.47–3.54 (br, 1H), 4.03 (dd, *J* = 11.5, 5.5 Hz, 1H), 4.64 (d, *J* = 8.0 Hz, 1H), 4.84 (t, *J* = 10.0 Hz, 1H), 4.98 (td, *J* = 10.0, 5.6 Hz, 1H), 5.06 (t, *J* = 10.0 Hz, 1H), 5.29 (t, *J* = 10.0, 1H), 5.37 (dd, *J* = 6.0, 3.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.7, 20.68, 20.74, 24.7, 25.5, 33.3, 33.4, 48.8, 64.0, 69.2, 70.5, 72.6, 80.5, 156.1, 169.8, 169.96, 170.96. HRMS(ESI): m/z calcd for $C_{18}H_{28}N_2O_8Na$ $[M+Na]^+$ 423.1743, found 423.1709.

Process for the work-up of cyclohexylurea xyloside (**29a**)

Reaction mixture (solution and precipitate)

Neutralized with solid $NaHCO_3$

add H_2O (ca. 1.0 mL)

Filtered

Solids
(a mixture of cyclohexylurea
and cyclohexylurea xyloside)

washed with AcOEt to
remove cyclohexylurea

Filtrate (aqueous layer)

washed with CH_2Cl_2 to
remove cyclohexylurea

Extracted with *n*-BuOH

Combined *n*-BuOH extracts

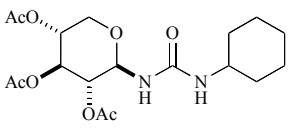
concentrated

Solids (cyclohexylurea xyloside)

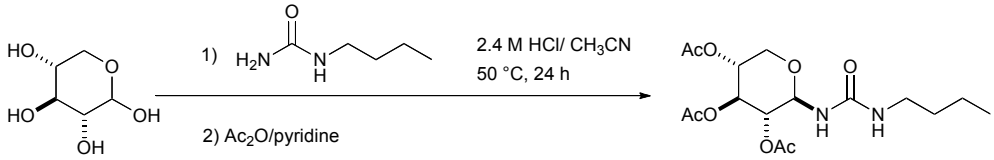
combined

Treated with Ac_2O/ pyridine

work-up

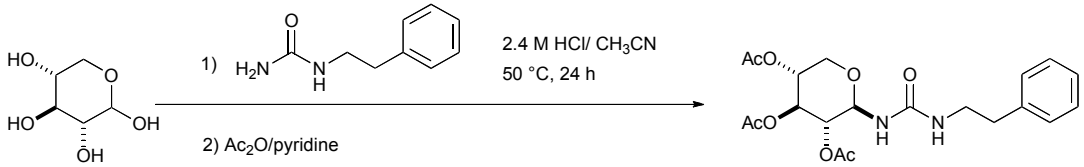


29a



26 **27a**

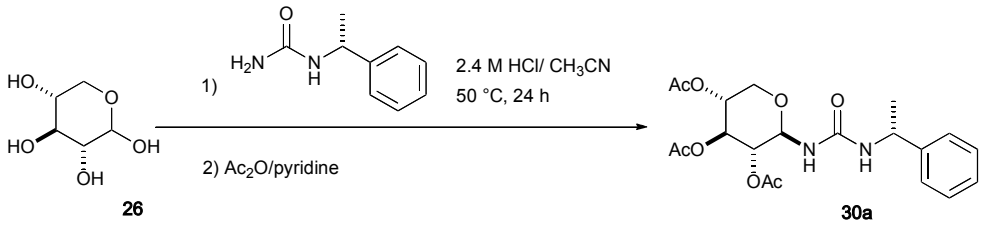
Starting from D-xylose **26** (300 mg, 2.00 mmol), *n*-butylurea (464 mg, 3.99 mmol), acetonitrile (1.00 mL), 2.4N HCl (0.25 mL), pyridine (10 mL) and Ac_2O (5.0 mL), *n*-butylurea glycoside **27a** was obtained as a white solid (547 mg, 73%, β:α = 98:2); Mp 145–146 °C (recrystallized from ether and hexane); $[\alpha]_D^{26}$ –1.73 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3343, 3297, 2960, 2931, 2877, 1758, 1648, 1576 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.90 (t, *J* = 7.0 Hz, 3H), 1.27–1.37 (m, 2H), 1.40–1.49 (m, 4H), 2.02 (d, *J* = 3.5 Hz, 6H), 2.07 (s, 3H), 3.44 (t, *J* = 11.5, 11.5 Hz, 1H), 4.06 (dd, *J* = 11.5, 5.5 Hz, 1H), 4.86 (t, *J* = 10.0, 9.5 Hz, 1H), 4.98 (td, *J* = 10.0, 5.5 Hz, 1H), 5.08 (t, *J* = 9.5, 9.5 Hz, 1H), 5.28 (t, *J* = 10.0, 9.5 Hz, 1H), 5.73 (d, *J* = 9.5 Hz, 1H); ^{13}C NMR (CD_3OD, 125 MHz) δ 14.2, 20.6, 21.0, 33.2, 40.5, 64.9, 70.5, 71.9, 74.4, 81.5, 159.7, 171.5. HRMS(ESI): m/z calcd for $C_{18}H_{28}N_2O_8Na$ $[M+Na]^+$ 423.1743, found 423.1709.



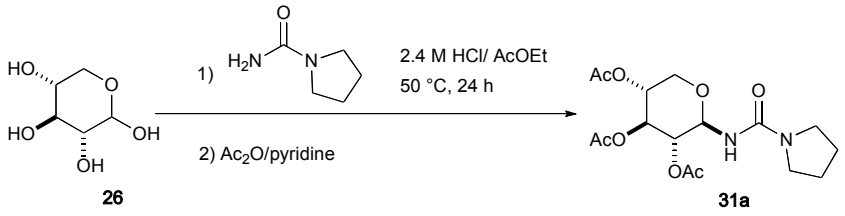
26 **28a**

D-Xylose **26** (300 mg, 2.00 mmol) was transformed into phenylethylurea glycoside **28a** (596 mg, 71%, β:α ≥ 98:2) by employing phenylethylurea (656 mg, 4.00 mmol), acetonitrile (1.20 mL), 2.4N HCl (0.30 mL), pyridine (3.2 mL) and Ac_2O (1.6 mL); Mp 72–73 °C (recrystallized from ether and hexane); $[\alpha]_D^{25}$ –6.57 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3370, 3288, 3028, 2943, 1755, 1651, 1568 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.03 (s, 6H), 2.05 (s, 3H), 2.70–2.85 (m, 2H), 3.38–3.50 (m, 3H), 4.04 (dd, *J* = 11.0, 5.5 Hz, 1H), 4.64 (t, *J* =5.5 Hz, 1H), 4.86 (t, *J* = 9.5, 1H), 4.96 (td, *J* = 9.5, 5.5 Hz, 1H), 5.02 (t, *J* = 9.5 Hz, 1H), 5.285 (t, *J* = 9.5 Hz, 1H), 5.295 (d, *J* = 9.5 Hz, 1H),

7.15–7.32 (m, 6H); ^{13}C NMR (CD_3OD, 125 MHz) δ 20.5, 20.6, 37.1, 42.4, 65.0, 70.5, 71.9, 74.4, 81.5, 127.3, 129.5, 129.8, 140.5, 159.6, 171.47, 171.50. HRMS(ESI): m/z calcd for $C_{20}H_{26}N_2O_8H$ $[M+H]^+$ 423.1767 found 423.1721.

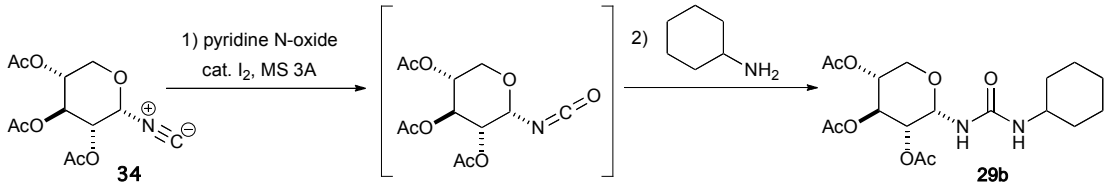


Starting from D-xylose **26** (300 mg, 2.00 mmol), (*R*)-α-methylbenzylurea (656 mg, 4.00 mmol), acetonitrile (1.80 mL), 2.4N HCl (0.30 mL), pyridine (2.4 mL) and Ac_2O (1.2 mL), (*R*)-α-methylbenzylurea glycoside **30a** was obtained as a white solid (660 mg, 78%, β:α ≥ 98:2); Mp 194–195 °C (recrystallized from ether and hexane); $[\alpha]_D^{26}$ +16.2 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3388, 3336, 2969, 2942, 1740, 1653, 1560 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.44 (d, *J* = 6.5 Hz, 3H), 1.99 (s, 3H), 2.01 (s, 6H), 3.36 (t, *J* = 10.5 Hz, 1H), 4.99 (t, *J* = 9.5 Hz, 1H), 4.83–4.87 (br, 1H), 4.94 (dt, *J* = 9.5, 5.5 Hz, 1H), 5.03 (t, *J* = 9.5 Hz, 1H), 5.15 (d, *J* = 17.5 Hz, 1H), 5.25 (t, *J* = 9.5 Hz, 1H), 5.47 (d, *J* = 9.5 Hz, 1H), 7.21–7.34 (m, 6H); ^{13}C NMR (CD_3OD, 125 MHz) δ 50.7, 50.6, 53.3, 80.8, 94.9, 100.5, 101.8, 104.4, 111.4, 156.8, 157.9, 159.5, 175.8, 188.9, 201.47, 201.50, 201.54. HRMS(ESI): m/z calcd for $C_{20}H_{26}N_2O_8H$ $[M+H]^+$ 423.1767, found 423.1732.

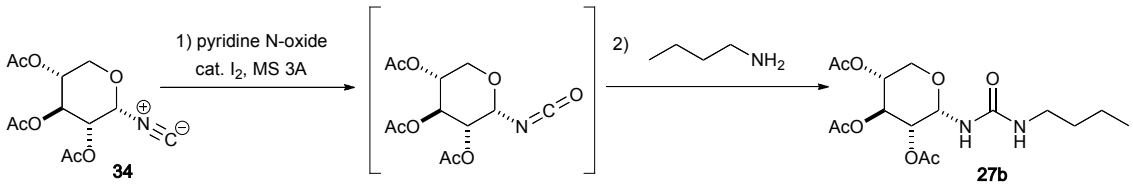


Starting from D-xylose **26** (300 mg, 2.00 mmol), pyrrolidineurea (456 mg, 4.00 mmol), ethyl acetate (1.80 mL), 2.4N HCl (0.30 mL), pyridine (1.6 mL) and Ac_2O (0.80 mL), pyrrolidineurea glycoside **31a** was obtained as a white solid (306 mg, 41%, β:α ≥ 98:2); Mp 111–113 °C (recrystallized from ether and hexane); $[\alpha]_D^{26}$ –7.60 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3392, 2974, 2876, 1752, 1653, 1535 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.84–1.94 (br, 4H), 2.04 (s, 3H), 2.05 (s, 3H), 2.06 (s, 3H), 3.20– 3.40(br, 3H),

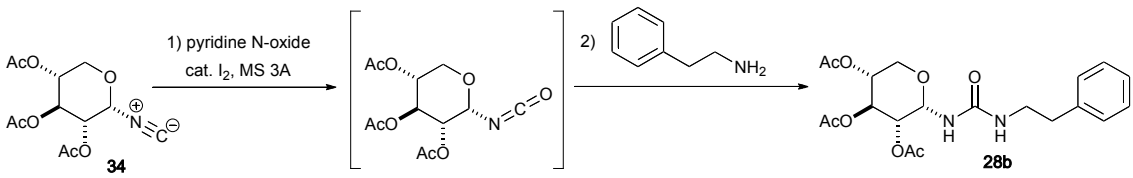
3.46 (t, *J* = 10.0 Hz, 1H), 4.05 (dd, *J* = 10.0, 5.5 Hz 1H), 4.85 (t, *J* = 10.0 Hz, 1H), 4.97 (dt, *J* = 10.0, 5.5 Hz, 1H), 5.08 (t, *J* = 10.0 Hz, 5H), 5.28 (d, *J* = 10.0 Hz, 1H), 5.32 (t, *J* = 10.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.69, 20.70, 20.9, 25.6, 45.5, 64.1, 69.3, 70.9, 72.3, 80.8, 154.7, 169.8, 169.9, 171.5. HRMS(ESI): m/z calcd for $C_{16}H_{24}N_2O_8Na$ $[M+Na]^+$ 395.1430, found 395.1440; m/z calcd for $C_{16}H_{24}N_2O_8H$ $[M+H]^+$ 373.1611, found 373.1599.



To a solution of α-xylopyranosyl isonitrile **34** (30 mg, 0.11 mmol), pyridine *N*-oxide (30 mg, 0.32 mmol) and powdered molecular sieves 3Å (50 mg) in acetonitrile (1.00 mL) under argon atmosphere was added iodine (2.0 mg, 0.0079 mmol). After being stirred at room temperature for 10 min, cyclohexylamine (0.36 mL, 0.32 mmol) was added. The resulting reaction mixture was stirred at room temperature for 20 min, and then filtered. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue (52 mg) was purified by silica gel chromatography (1:2 AcOEt/ hexane) to afford α-urea glycoside **29b** (38 mg, 90%) as a white solid. Mp 196–197 °C; $[\alpha]_D^{25}$ +34.5 (*c* 1.00, $CHCl_3$); IR (KBr or NaCl) ν_{max} 3335, 2933, 2855, 1752 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.05–1.95 (m, 10H), 2.06 (s, 3H), 2.07 (s, 3H), 2.12 (s, 3H), 3.55–3.65 (m, 1H), 3.85 (dd, *J* = 12.0, 6.0 Hz, 1H), 3.91 (dd, *J* = 12.0, 4.0 Hz, 1H), 4.82 (dt, *J* = 6.0, 4.0 Hz, 1H), 4.98 (dd, *J* = 6.0, 3.5 Hz, 1H), 5.23 (t, *J* = 6.0 Hz, 1H), 5.37 (dd, *J* = 6.0, 3.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.68, 20.70, 20.8, 24.7, 25.4, 33.4, 33.5, 48.8, 61.37, 67.35, 67.9, 68.8, 156.4, 169.2, 169.5, 169.6. HRMS(ESI): m/z calcd for $C_{18}H_{28}N_2O_8Na$ $[M+Na]^+$ 423.1743, found 423.1722.

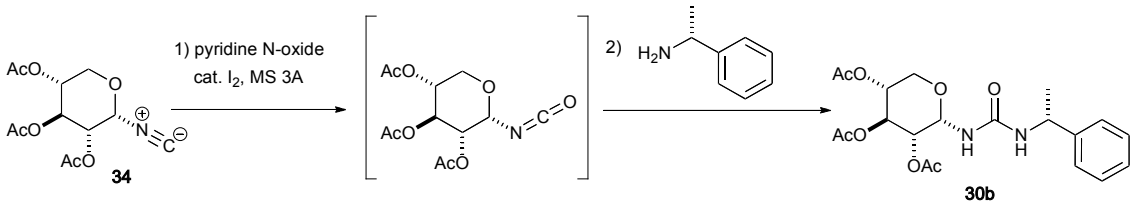


To a solution of α-xylopyranosyl isonitrile **34** ((25 mg, 0.088 mmol), pyridine *N*-oxide (25 mg, 0.26 mmol) and powdered molecular sieves 3Å (50 mg) in acetonitrile (1.00 mL) under argon atmosphere was added iodine (2.0 mg, 0.0079 mmol). After being stirred at room temperature for 10 min, *n*-butylamine (0.025 mL, 0.26 mmol) was added. The resulting reaction mixture was stirred at room temperature for 20 min, and then filtered. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue (32 mg) was purified by silica gel chromatography (1:2 AcOEt/ hexane) to afford α-urea glycoside **27b** (21 mg, 64%) as a white solid; Mp 181–182 °C; $[\alpha]_D^{24}$ +42.7 (*c* 0.95, $CHCl_3$); IR (KBr) ν_{max} 3341, 2960, 2933, 2873, 1749, 1643, 1567 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.90 (t, *J* = 7.0 Hz, 3H), 1.26–1.36 (m, 2H), 1.41–1.50 (m, 2H), 2.50 (s, 3H), 2.07 (s, 3H), 2.09 (s, 3H), 3.15–3.22 (m, 1H), 3.82 (dd, *J* = 12.0, 6.5 Hz, 1H), 3.89 (dd, *J* = 12.0, 4.0 Hz, 1H), 4.81 (dt, *J* = 6.5, 4.0 Hz, 1H), 4.88 (dd, *J* = 6.5, 3.5 Hz 1H), 5.15 (bt, *J* = 5.5 Hz, 1H), 5.23 (t, *J* = 6.5 Hz, 1H), 5.39 (dd, *J* = 6.5, 3.5 Hz), 5.49 (d, *J* = 6.5 Hz, 1H); ^{13}C NMR (CD_3OD, 125 MHz) δ 14.1, 20.58, 20.63, 20.7, 21.0, 40.5, 62.7, 69.0, 69.5, 70.2, 76.8, 159.6, 171.0, 171.3, 171.4; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.7, 19.9, 20.67, 20.74, 32.0, 40.0, 61.1, 67.6, 68.1, 68.8, 157.2, 169.3, 169.5, 169.6. HRMS(ESI): m/z calcd for $C_{16}H_{26}N_2O_8H$ $[M+H]^+$ 375.1767, found 375.1718.

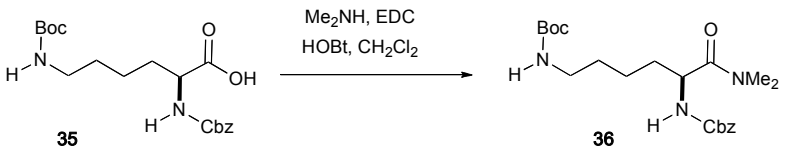


Starting from α-xylopyranosyl isonitrile **34** (25 mg, 0.088 mmol), pyridine *N*-oxide (25 mg, 0.26 mmol), iodine (1.3 mg, mg, 0.0051 mmol) and powdered molecular sieves 3Å (50 mg), acetonitrile (1.00 mL) and β-phenetylamine (0.033 mL, 0.26 mmol), α-urea

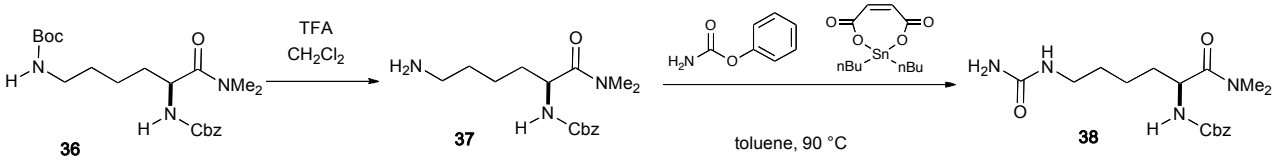
glycoside **28b** (17 mg, 47%) was obtained as a white solid; Mp 151–152 °C (recrystallized from ether and hexane); $[\alpha]_D^{27}$ +37.1 (*c* 0.93, $CHCl_3$)
IR (KBr) ν_{max} 3334, 2938, 1749, 1639, 1561 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.06 (s, 3H), 2.07 (s, 3H), 2.10 (s, 3H), 2.77–2.87 (m, 2H), 3.41–3.55 (m, 2H), 3.73–3.82 (m, 2H) 4.81 (dt, *J* = 6.5, 4.5 Hz, 1H), 4.87 (dd, *J* = 6.5, 4.5 Hz, 1H), 4.81 (dt, *J* = 6.5, 4.5 Hz, 1H), 4.98–5.04 (br, 1H), 5.15 (d, *J* = 6.5 Hz,1H), 5.22 (t, *J* = 6.5 Hz, 1H), 5.32 (dd, *J* = 6.5, 4.5 Hz, 1H), 7.16–7.33 (m, 6H); ^{13}C NMR (CD_3OD, 125 MHz) δ 20.6, 20.65, 20.69, 37.1, 42.3, 62.5, 69.1, 69.5, 70.2, 76.8, 127.3, 129.5, 129.8, 140.5, 159.5, 171.0, 171.2, 171.4 HRMS(ESI): m/z calcd for $C_{20}H_{26}N_2O_8H$ $[M+H]^+$ 423.1767, found 423.1735.



Starting from α-xylopyranosyl isonitrile **34** (50 mg, 0.12 mmol), pyridine *N*-oxide (50 mg, 0.53 mmol), iodine (3.0 mg, mg, 0.012 mmol) and powdered molecular sieves 3Å (50 mg), acetonitrile (2.00 mL) and (*R*)-α-methylbenzylamine (0.067 mL, 0.53 mmol), α-urea glycoside **30b** (48 mg, 65%) was obtained as a white solid; Mp 178–180 °C (recrystallized from ether and hexane); $[\alpha]_D^{27}$ +43.4 (*c* 0.65, $CHCl_3$); IR (KBr) ν_{max} 3317, 2969, 2871, 1745, 1645, 1558 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.48 (d, *J* = 6.5 Hz, 3H), 2.06 (s, 3H), 2.08 (s, 3H), 2.11 (s, 3H), 3.49 (d, *J* = 5.5 Hz, 1H), 3.72–3.80 (br, 1H), 3.88 (dd, *J* = 11.5, 3.5 Hz, 1H), 4.79 (dt, *J* = 6.5, 3.5 Hz, 1H), 4.86 (dd, *J* = 6.5, 3.5 Hz, 1H), 4.89–4.95 (br, 1H), 5.11 (d, *J* = 5.5 Hz 1H), 5.14–5.21(m, 2H), 5.42 (dd, *J* = 6.5, 3.5 Hz, 1H), 7.20–7.38 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 1.02, 20.7, 20.8, 22.9, 50.0, 61.5, 67.3, 67.9, 68.8, 76.5, 125.8, 127.4, 128.8, 143.6, 156.3, 169.2, 169.5, 169.6. HRMS(ESI): m/z calcd for $C_{20}H_{26}N_2O_8H$ $[M+H]^+$ 423.1767, found 423.1725.



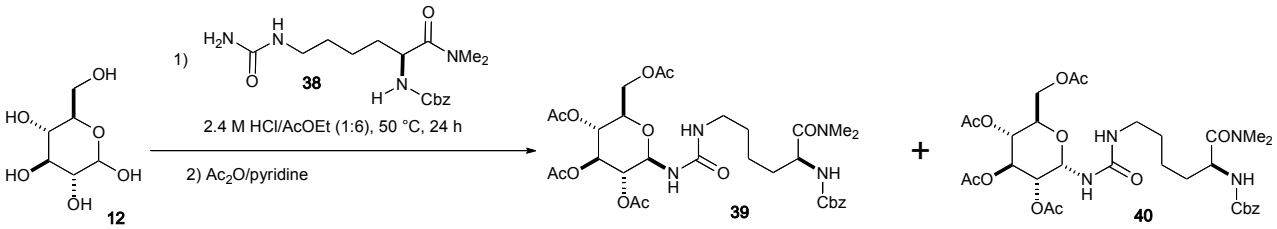
To a solution of carboxylic acid **35** (4.82 g, 10.5 mmol) in CH_2Cl_2 (100 mL) under argon atmosphere was added EDC (3.21 g, 16.8 mmol) and HOBt (1.42 g, 10.5 mmol). After being stirred at room temperature for 20 min, dimethylamine in CH_2Cl_2 was added. The resulting reaction mixture was stirred at room temperature for 5 hours, and then washed with 1 M $KHSO_4$, saturated aqueous $NaHCO_3$. Aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (2:1 AcOEt/hexane) to afford amide **36** (4.28 g, 87%) as a colorless oil: $[\alpha]_D^{24}$ = +15.7 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3335, 3033, 2974, 2935, 2866, 2484, 1710 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.43 (s, 9H), 1.34-1.74 (6H), 2.96 (s, 3H), 3.07 (s, 3H), 3.08−3.12 (2H), 4.58−4.62 (br, 1H), 4.66 (td, *J* = 8.5, 4.5 Hz, 1H), 5.09 (s, 2H), 5.71 (d, *J* = 8.5 Hz, 1H), 7.29−7.36 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 22.2, 28.3, 29.5, 32.7, 35,6, 37.0, 39.9, 40.1, 50.3, 50.4, 66.7, 78.9, 127.8, 127.9, 128.4, 136.3, 155.9, 156.0, 171.7; HRMS(ESI): m/z calcd for $C_{21}H_{33}N_3O_5Na$ $[M+Na]^+$ 467.1642, found 462.1619.



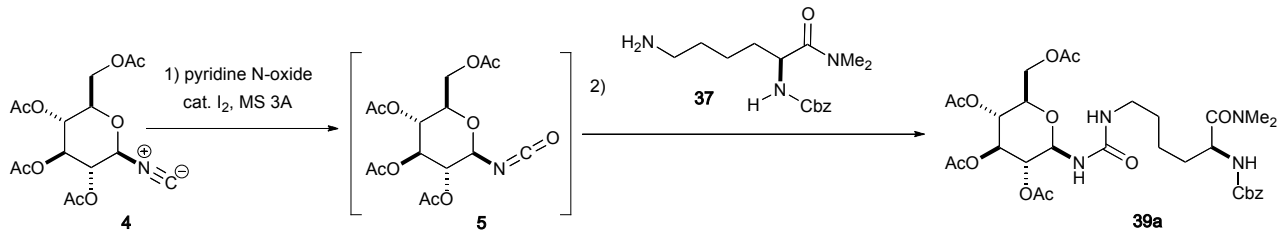
To a solution of Boc-protected **36** (5.51 g, 13.5 mmol) in CH_2Cl_2 (135 ml) was added TFA (18 ml) at room temperature. After stirring at room temperature for 30 min, the reaction mixture was concentrated. The resulting TFA sat was dissolved in CH_2Cl_2, and then washed with aqueous $NaHCO_3$ solution. The separated aqueous solution was extracted with CH_2Cl_2, and the combined organic layers were dried (Na_2SO_4) and then concentrated under reduced pressure to afford the amine **37** (2.55 g, 74%), which was used for the next reaction without further purification.

A solution of amine **37** (100 mg, 0.33 mmol), phenylcarbamate (134 mg, 0.98

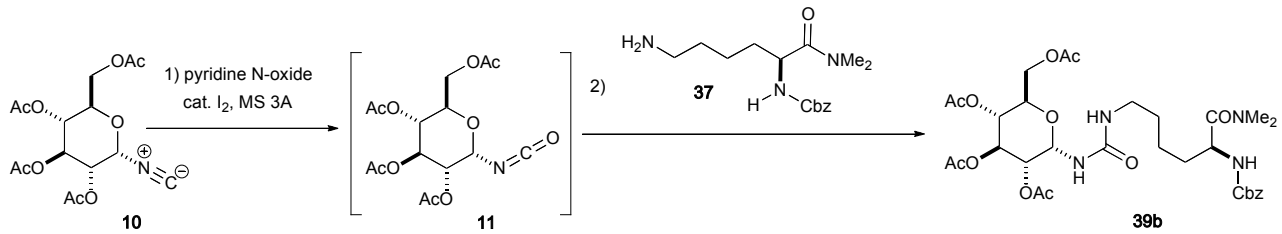
mmol) and di-*n*-butyltin maleate (11 mg, 0.033 mmol) dissolved in toluene (3.0 mL) was heated at 90 °C for 2 hours. After cooling, the resulting reaction mixture was concentrated under reduced pressure to afford the residue, which was purified by silica gel chromatography (7:1 CH_2Cl_2/MeOH) to afford urea **38** (92 mg, 80%) as a colorless oil; $[\alpha]_D^{25}$ = +11.0 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3360, 3034, 2938, 2864, 1711 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.33-1.70 (6H), 2.94 (s, 3H), 3.06 (s, 3H), 4.59−4.66 (1H), 4.63 (td, *J* = 8.5, 4.5 Hz, 1H), 5.07 (s,2H), 5.16−5.27 (br, 1H), 6.00 (d, *J* = 8.5 Hz, 1H), 7.29−7.36 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 22.2, 29.3, 31.9, 35.6, 36.9, 39.4, 49.9, 50.5, 66.4, 127.6, 127.8, 128.2, 136.1, 156.1, 159.8, 171.9. HRMS(ESI): m/z calcd for $C_{17}H_{26}N_4O_4Na$ $[M+Na]^+$ 373.1892, found 373.1871.



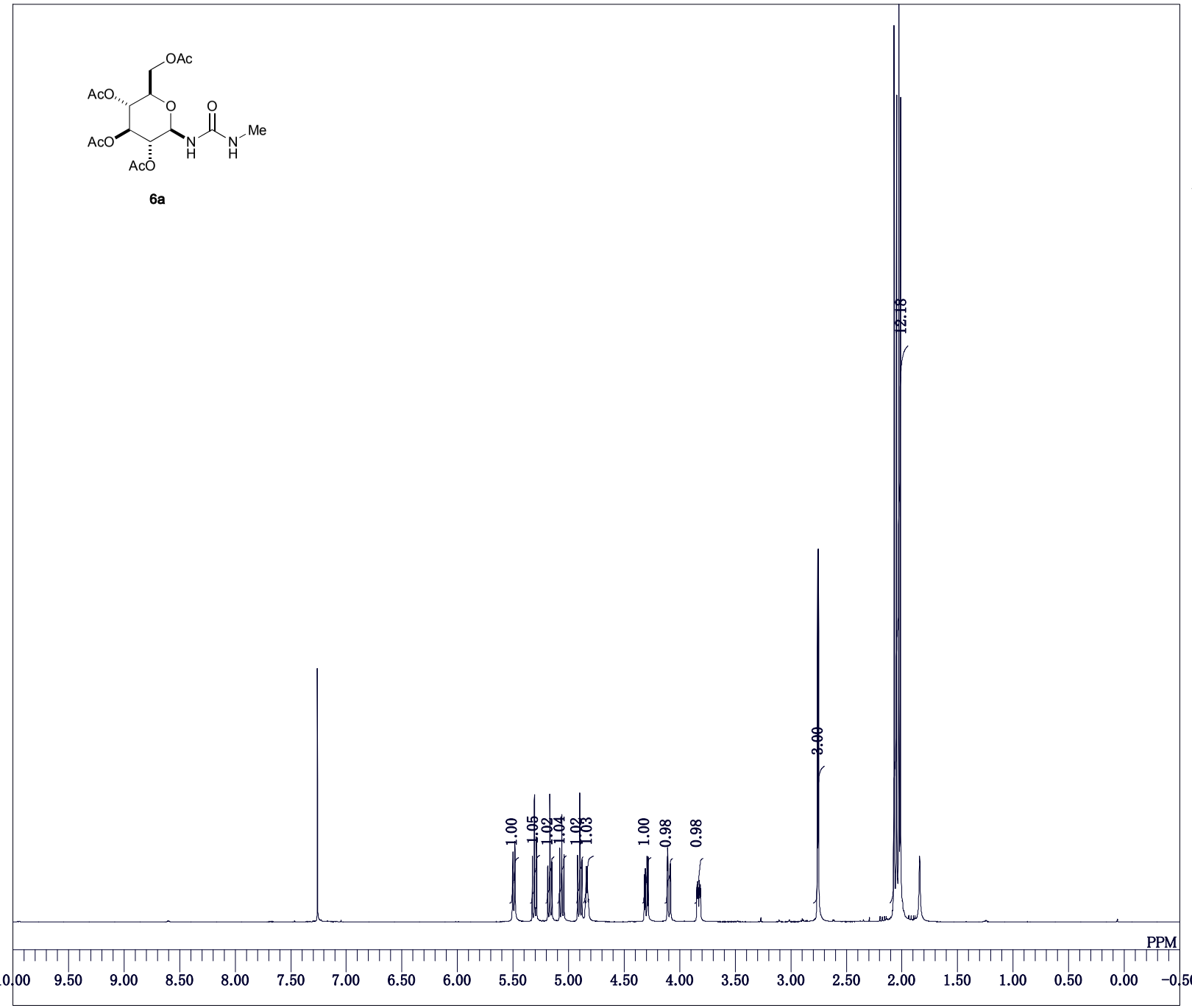
A solution of D-glucose **12** (100 mg, 0.56 mmol) and urea **38** (972 mg, 2.78 mmol) dissolved in a mixture of AcOEt (1.7 mL) and 2.4 N HCl (0.2 mL)was stirred at 50 °C for 1 day. The reaction mixture was neutralized with solid $NaHCO_3$, and was washed with CH_2Cl_2 to remove excess urea **38**. The aqueous layer was extracted with *n*-BuOH, and the combined extracts were concentrated under reduced pressure to afford the residue, which was dissolved in pyridine (2.0 mL) and Ac_2O (1.0 mL). The solution was stirred at 50 °C for 3 hours, and then diluted with saturated aqueous $NaHCO_3$. The separated aqueous layer was extracted with Et_2O, and combined organic layers were washed with brine, dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford glucosylurea **39** and **40** (202 mg, 51%, **39**: **40** = 95:5) as a white solid.



To a solution of pyridine *N*-oxide (16 mg, 0.17 mmol) and powdered molecular sieves 3A (60 mg) in acetonitrile (1.0 mL) under argon atmosphere was added glucosyl isonitrile **4** (20 mg, 0.06 mmol) and iodine (1.0 mg, 7.0 mol%). After being stirred at room temperature for 10 min, a solution of amine (52 mg, 0.17 mmol, dissolved in a small amount of CH_2Cl_2) was added. The resulting reaction mixture was stirred at room temperature for 30 min, and then filtered on Super Cell. The filtrate was poured into saturated aqueous $NaHSO_3$ solution, and aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried (Na_2SO_4) and then concentrated under reduced pressure. The resulting residue was purified by silica gel chromatography (AcOEt) to afford **39a** (32 mg, 83%) as a white solid; Mp 68−69 ˚C (recrystallized from AcOEt/hexane); $[\alpha]_D^{24}$ = +2.35 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3472, 3370, 3062, 3034, 2941, 2865, 2360, 2342, 2112, 1753 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.31−1.71 (6H), 2.01 (s, 3H), 2.03 (s, 3H), 2.04 (s, 3H), 2.05 (s, 3H), 2.96 (s, 3H), 3.07 (s, 3H), 3.11−3.18 (2H), 3.81 (ddd, *J* = 9.5, 4.5, 1.5 Hz, 1H), 4.07 (dd, *J* = 12.5, 1.5 Hz, 1H), 4.31 (dd, *J* = 12.5, 4.0 Hz, 1H), 4.64 (td, *J* = 8.5, 4.5 Hz, 1H), 4.88 (t, *J* = 9.5 Hz, 1H), 5.05 (t, *J* = 9.5 Hz, 1H), 5.10−5.12 (2H), 5.16 (t, *J* = 9.5 Hz, 1H), 5.29 (d, *J* = 9.5 Hz, 1H), 5.45 (d, *J* = 9.5 Hz, 1H), 5.88 (d, *J* = 8.5 Hz, 1H), 7.29−7.37 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.45, 20.49, 20.57, 22.2, 29.2, 32.4, 35.7, 37.0, 39.7, 50.3, 61.7, 66.7, 68.2, 70.4, 72.8, 73.0, 77.2, 79.9, 127.8, 127.9, 128.3, 136.2, 156.1, 156.5, 169.6, 169.7, 170.5, 170.6, 171.8. HRMS(ESI): m/z calcd for $C_{31}H_{45}N_4O_{13}$ $[M+H]^+$ 681.2983, found 681.2995.



Starting from glucosyl isonitrile **10** (20 mg, 0.06 mmol), amine **37** (52 mg, 0.17 mmol), pyridine *N*-oxide (16 mg, 0.17 mmol), powdered molecular sieves 3A (60 mg), acetonitrile (1.0 mL) and iodine (1.0 mg, 7.0 mol%), urea **39b** was obtained as a white solid (30 mg, 81%); Mp 62−63 °C; $[\alpha]_D^{24}$ = +18.5 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3379, 3034, 2942, 2866, 2360, 2342, 1752 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.34-1.75 (6H), 2.01 (s, 3H), 2.03 (s, 3H), 2.06 (s, 3H), 2.08 (s, 3H), 2.96 (s, 3H), 3.08 (s, 3H), 3.10−3.16 (1H), 3.24−3.32 (1H), 4.09−4.14 (2H), 4.23 (dd, *J* = 12.5, 5.0 Hz, 1H), 4.65 (td, *J* = 8.5, 4.5 Hz, 1H), 5.04 (t, *J* = 10.0 Hz, 1H), 5.08−5.10 (2H), 5.16 (br, 1H), 5.31 (d, *J* = 10.0 Hz, 1H), 5.40 (t, *J* = 5.5 Hz, 1H), 5.44 (t, *J* = 4.5 Hz, 1H), 5.77 (d, *J* = 8.5 Hz, 1H), 7.29−7.36 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.51, 20.54, 20.7, 22.2, 29.5, 32.5, 35.7, 37.0, 39.8, 50.4, 61.6, 66.8, 67.0, 68.1, 68.8, 69.8, 76.9, 127.9, 127.8, 128.0, 128.4, 136.2, 156.1, 157.4, 169.2, 169.4, 170.0, 170.5, 171.7. Anal. Calcd for $C_{21}H_{33}N_3O_9$: C, 54.70; H, 6.52; N, 8.23. Found: C, 54.63; H, 6.38; N, 8.10.



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EXMOD	proton.jxp
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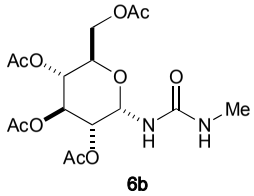
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50



6b

DFILE	sou4117data_proton-1-1.jdf
COMNT	single_pulse
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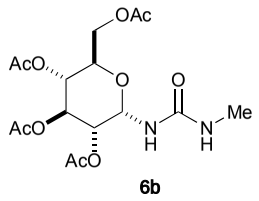
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	18.7 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



6b

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EXMOD	proton.jxp
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OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
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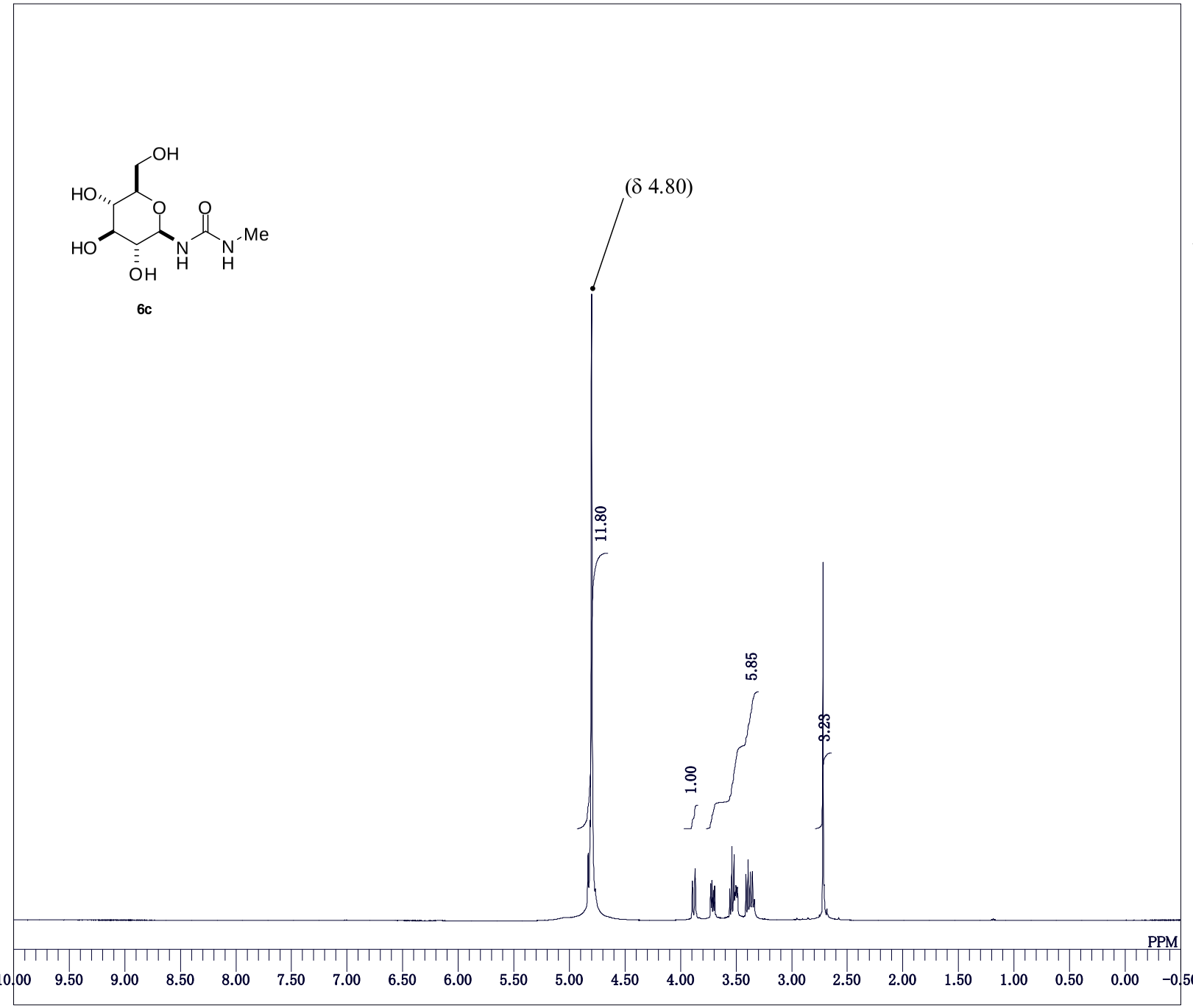
SLVNT	CD3OD
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



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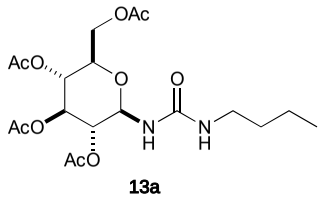
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	D2O
EXREF	4.80 ppm
BF	0.12 Hz
RGAIN	50



13a

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EXMOD	proton.jxp
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OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
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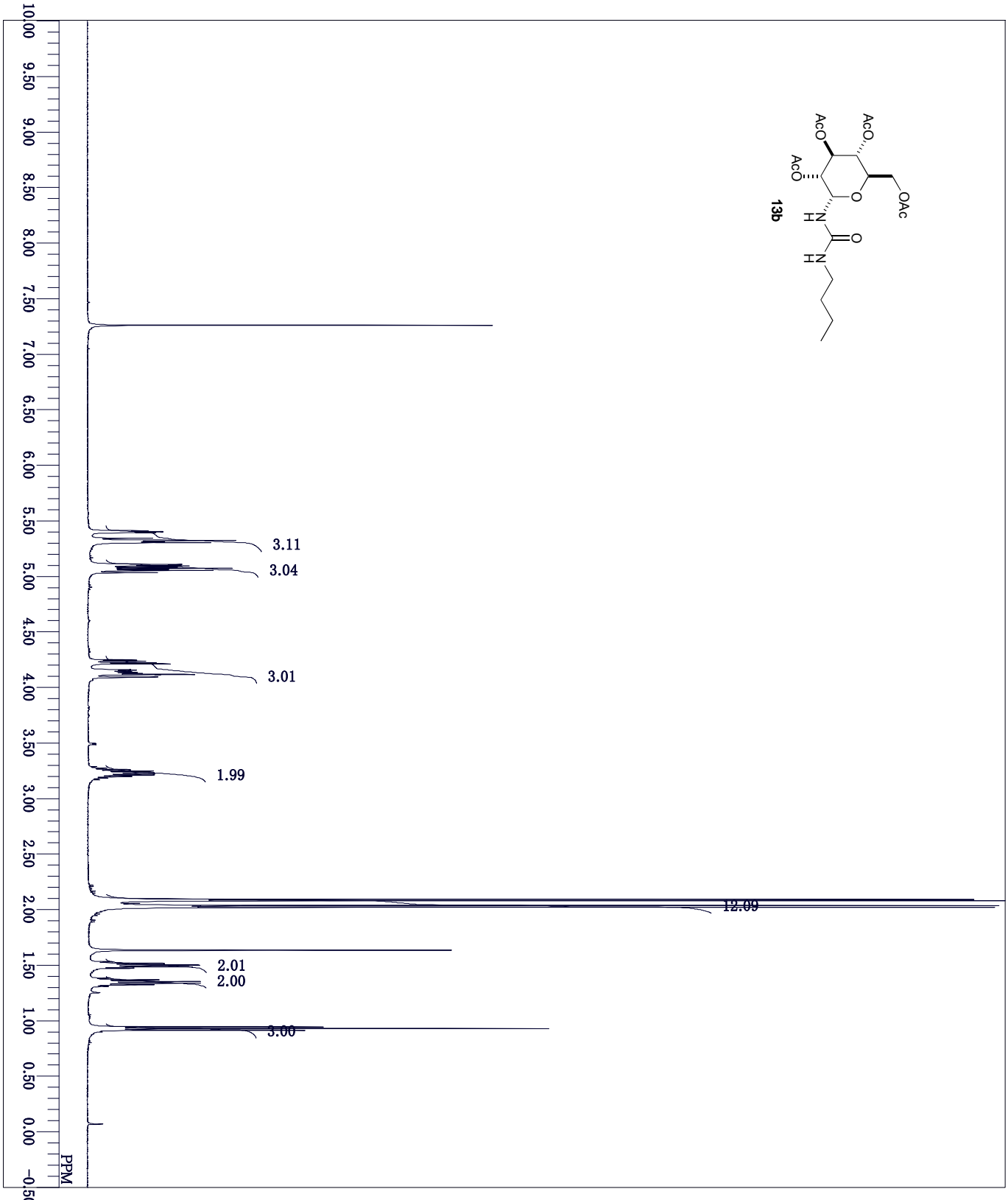
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50



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FREQU 9384.38 Hz
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CTEMP 19.9 c
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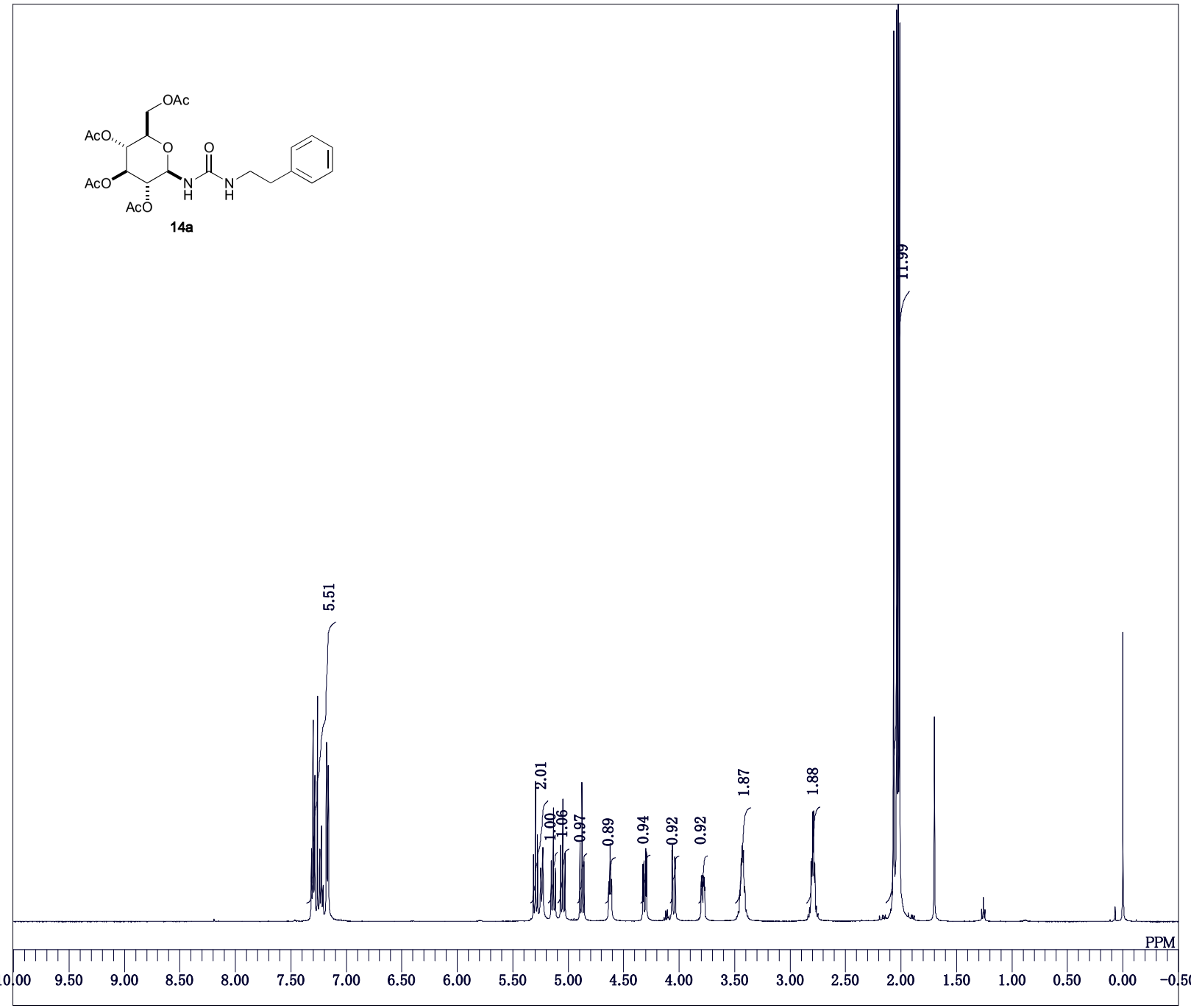
RGAIN 50



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OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
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CTEMP	21.1 c
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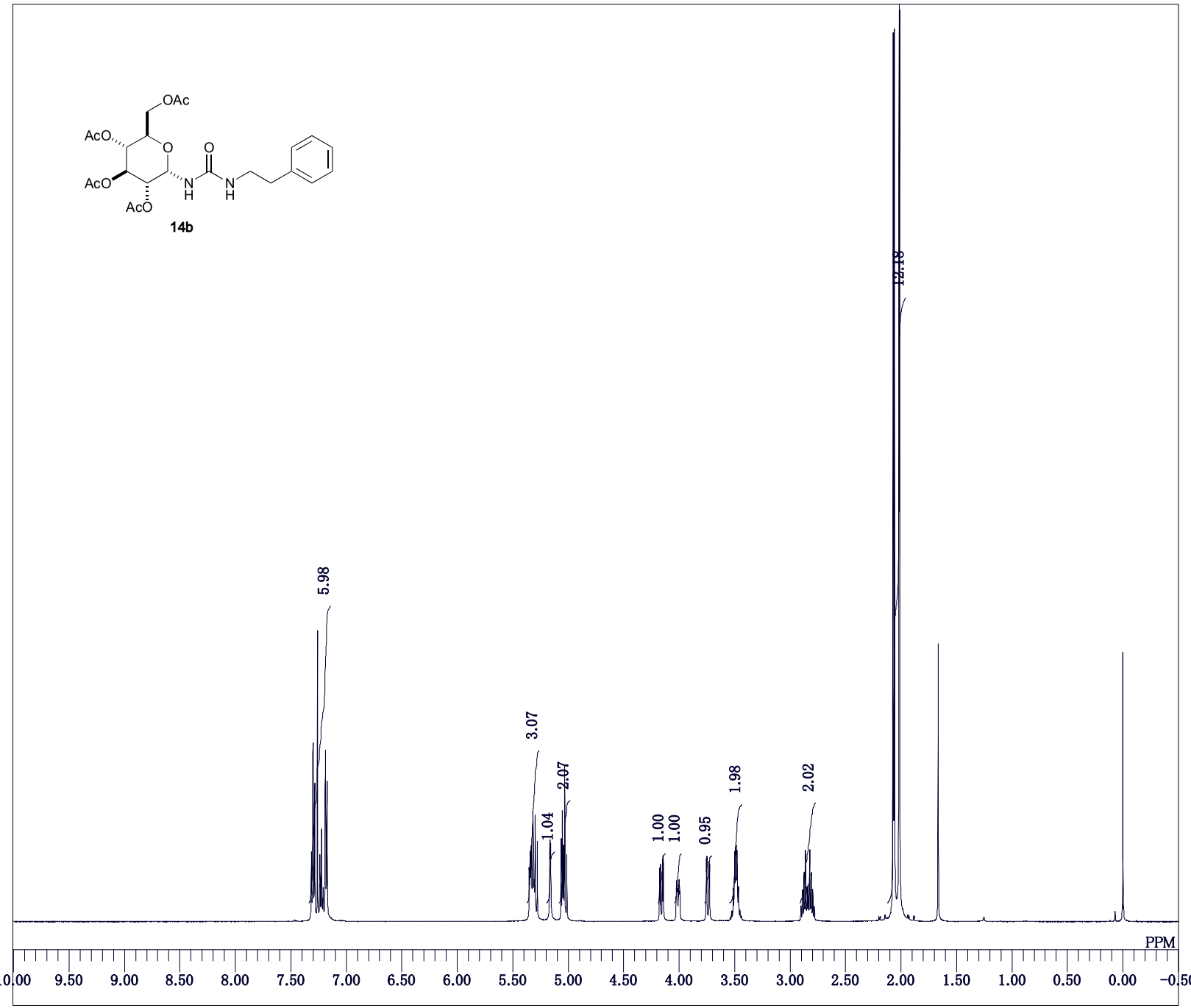
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



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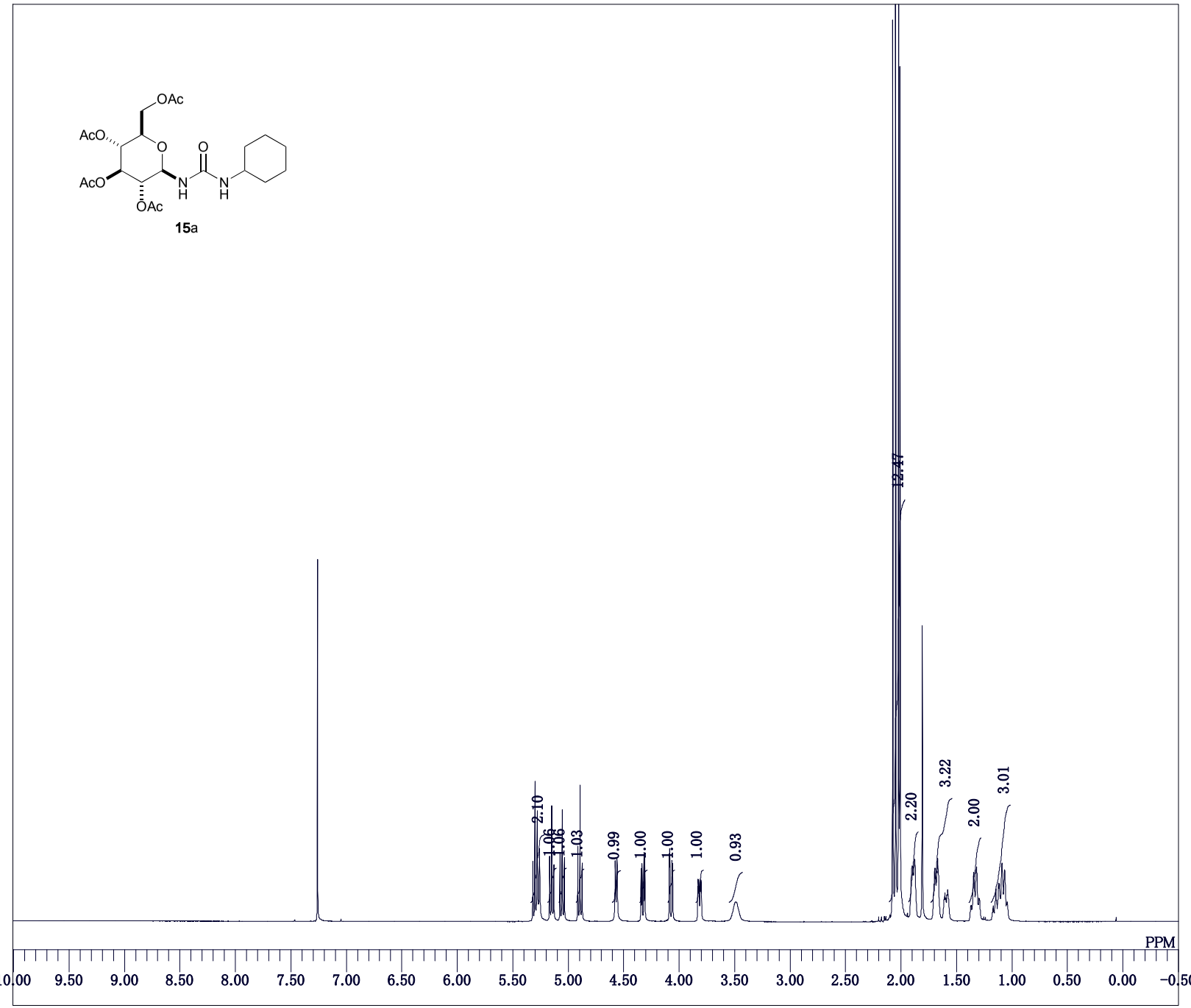
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



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EXMOD	proton.jxp
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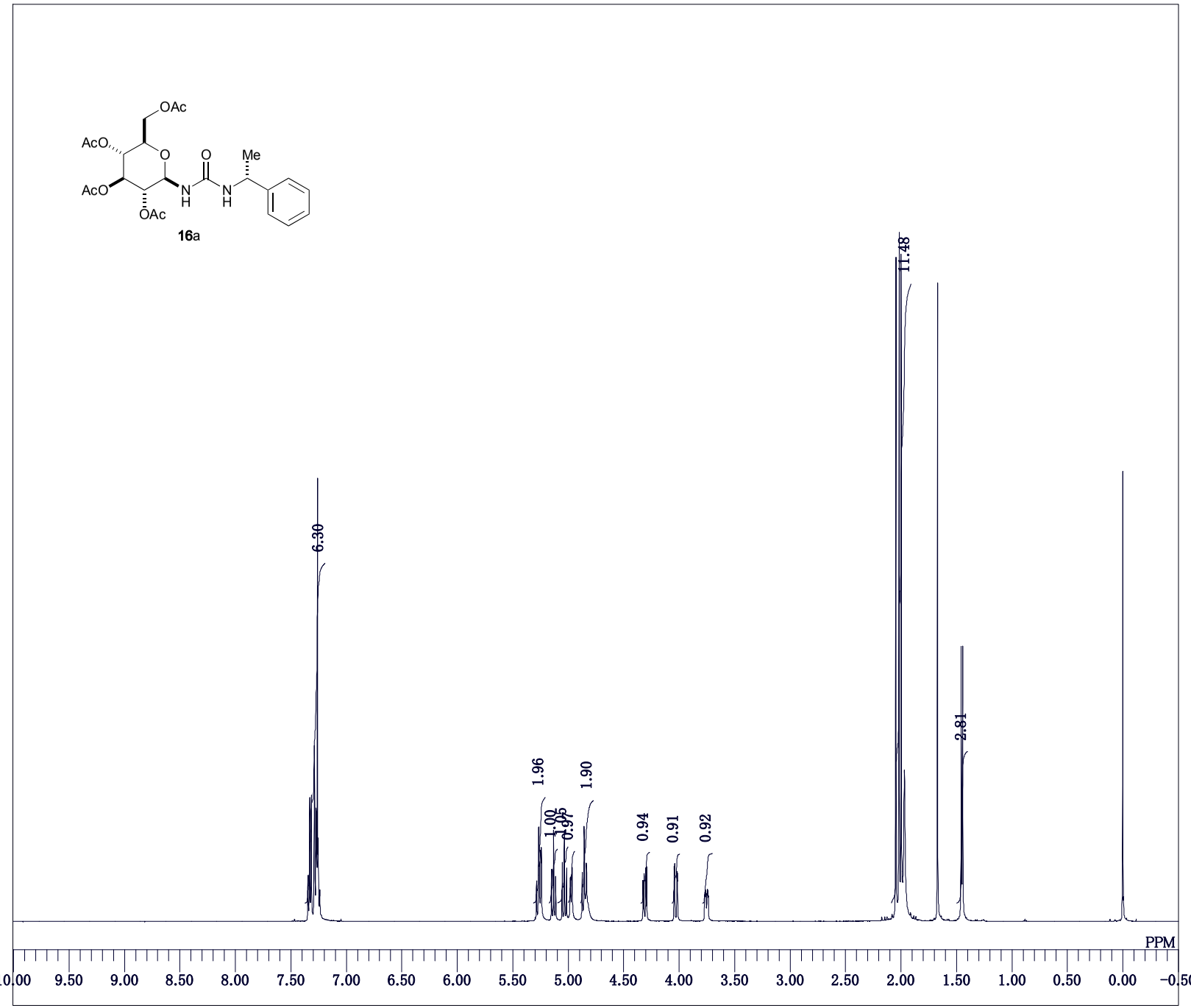
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



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OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
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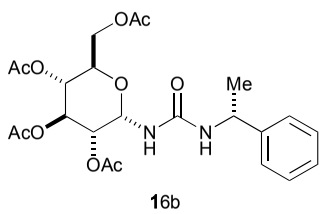
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50



16b

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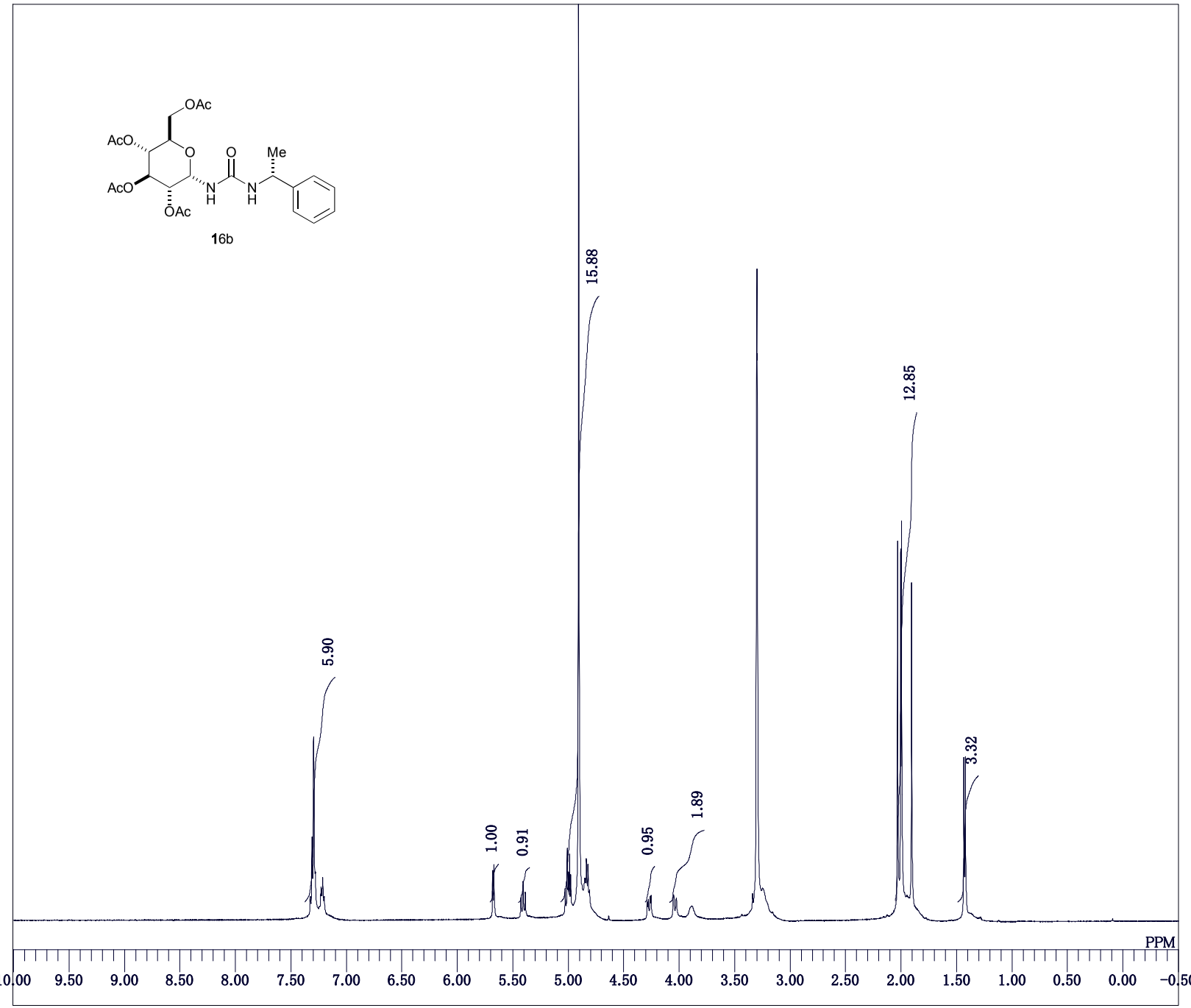
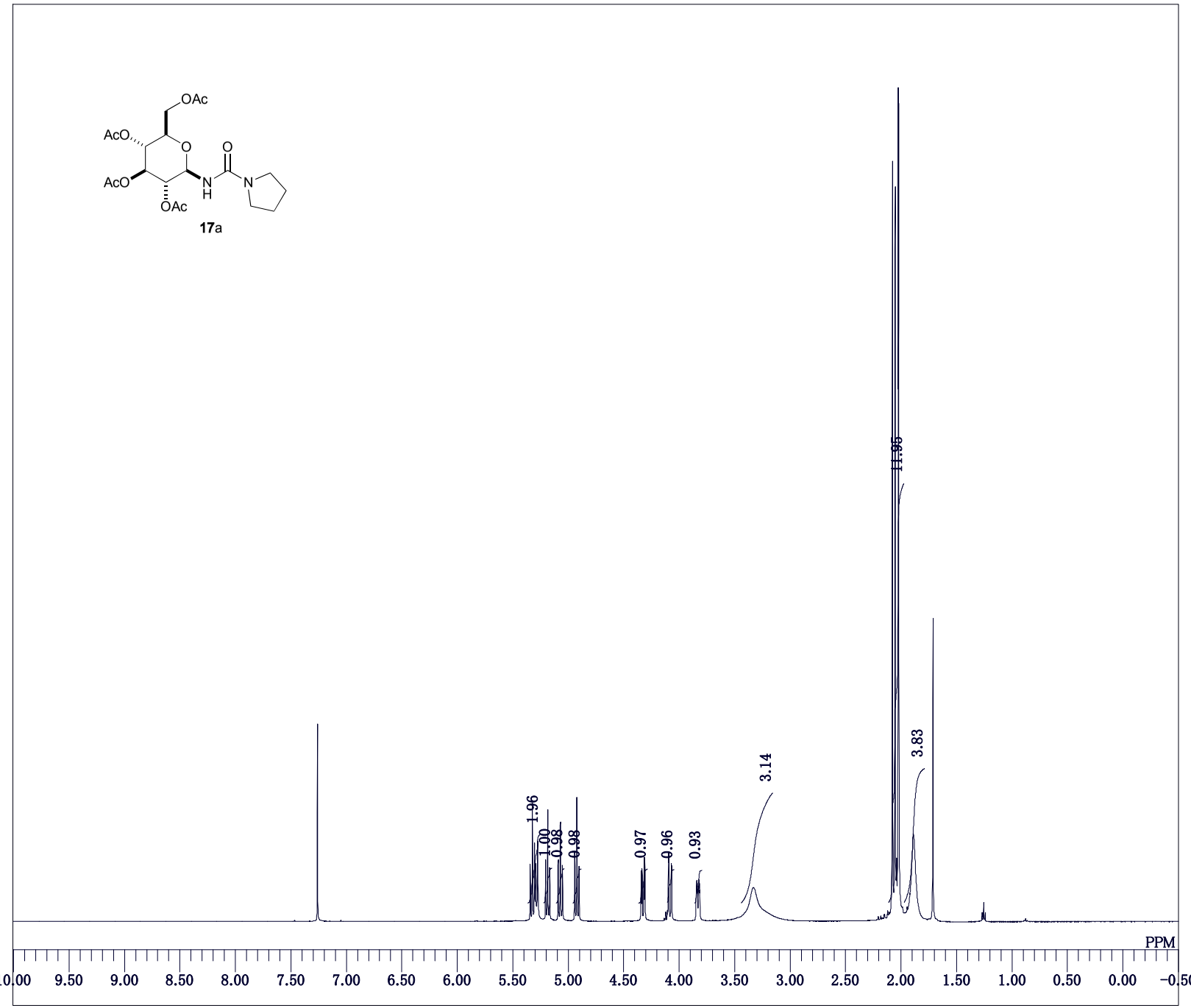
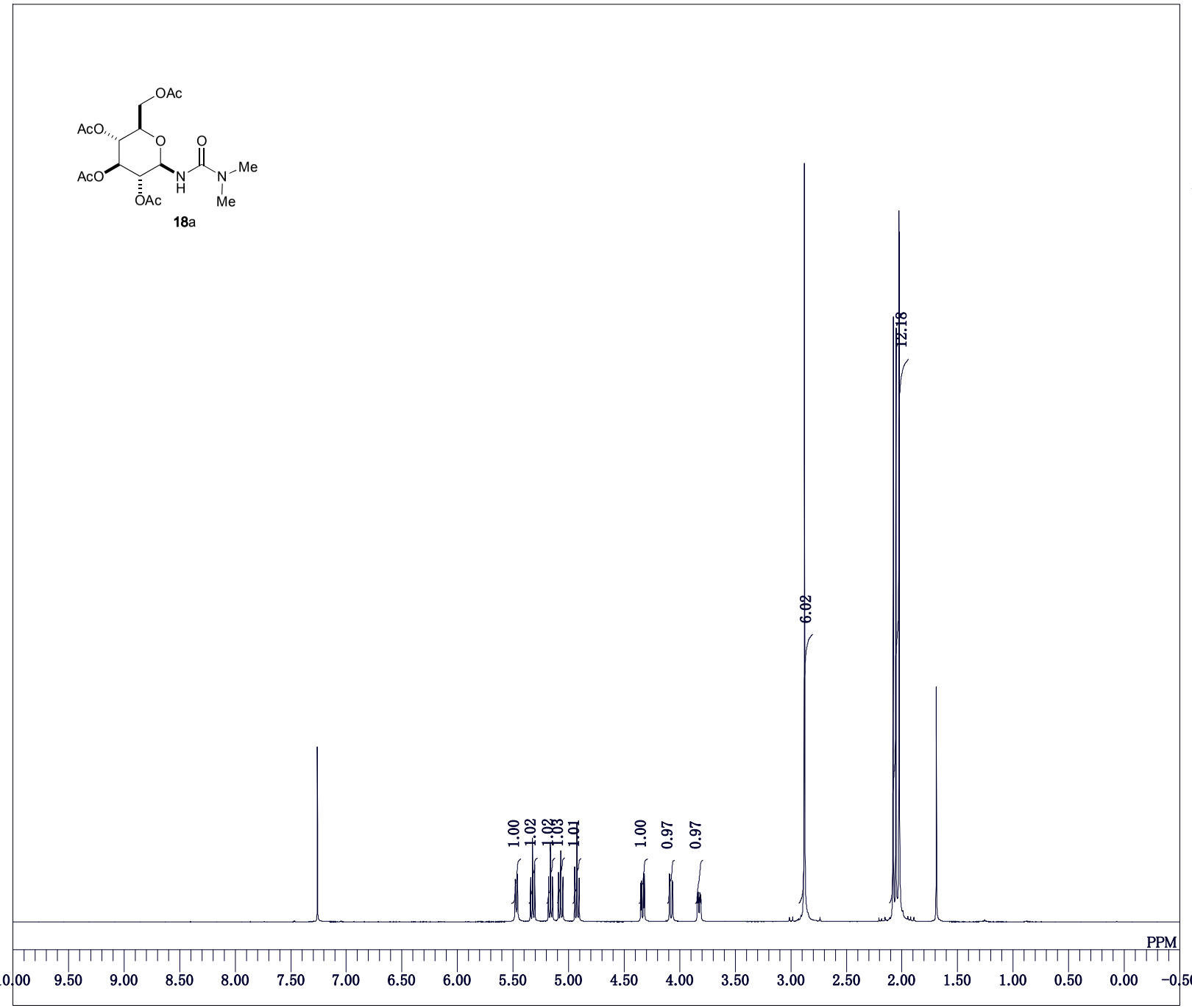
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



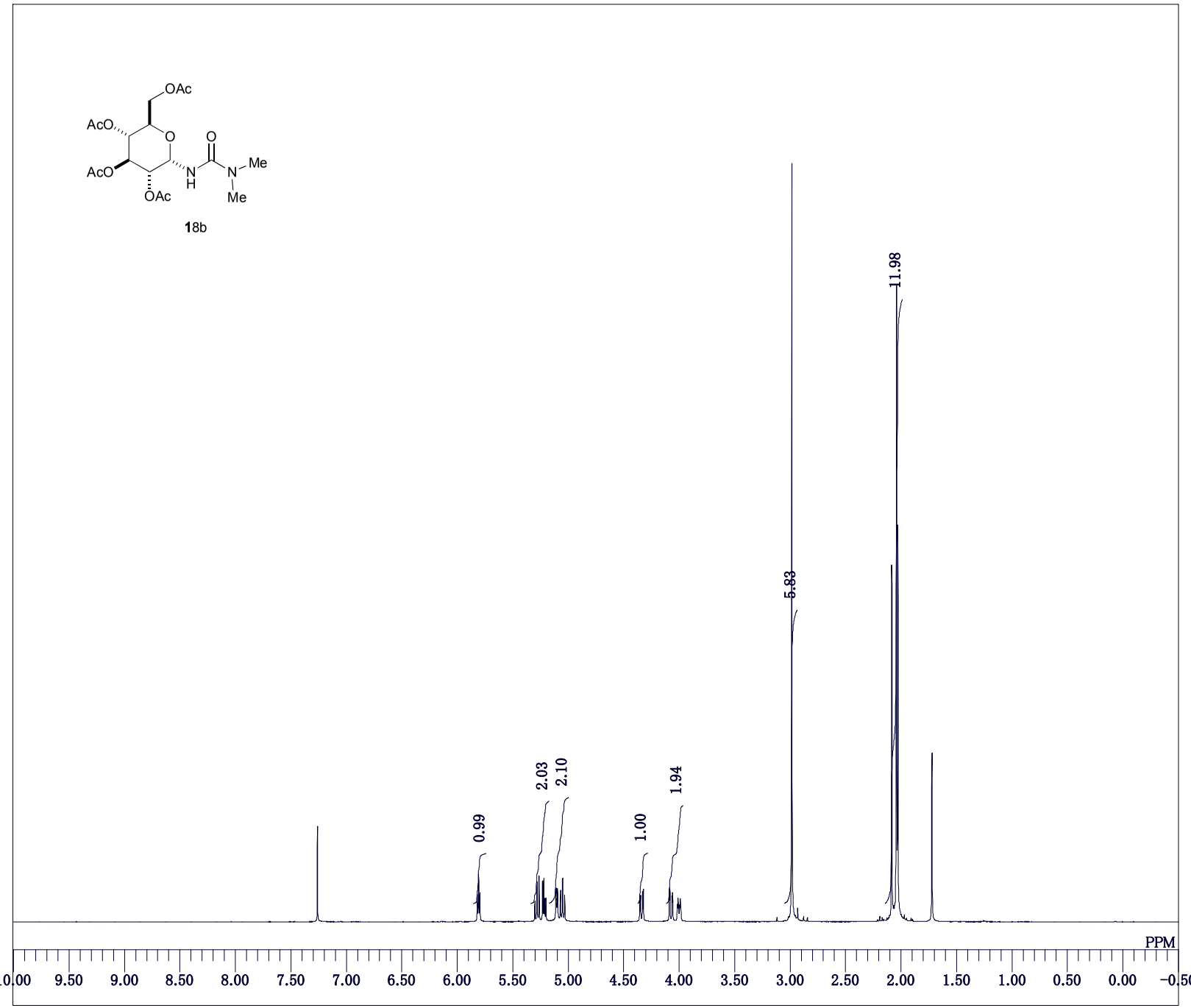
DFILE	sou4115CD3ODdata_proton-5-1.jdf
COMNT	single_pulse
DATIM	2011-11-25 14:38:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.7 c
SLVNT	CD3OD
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



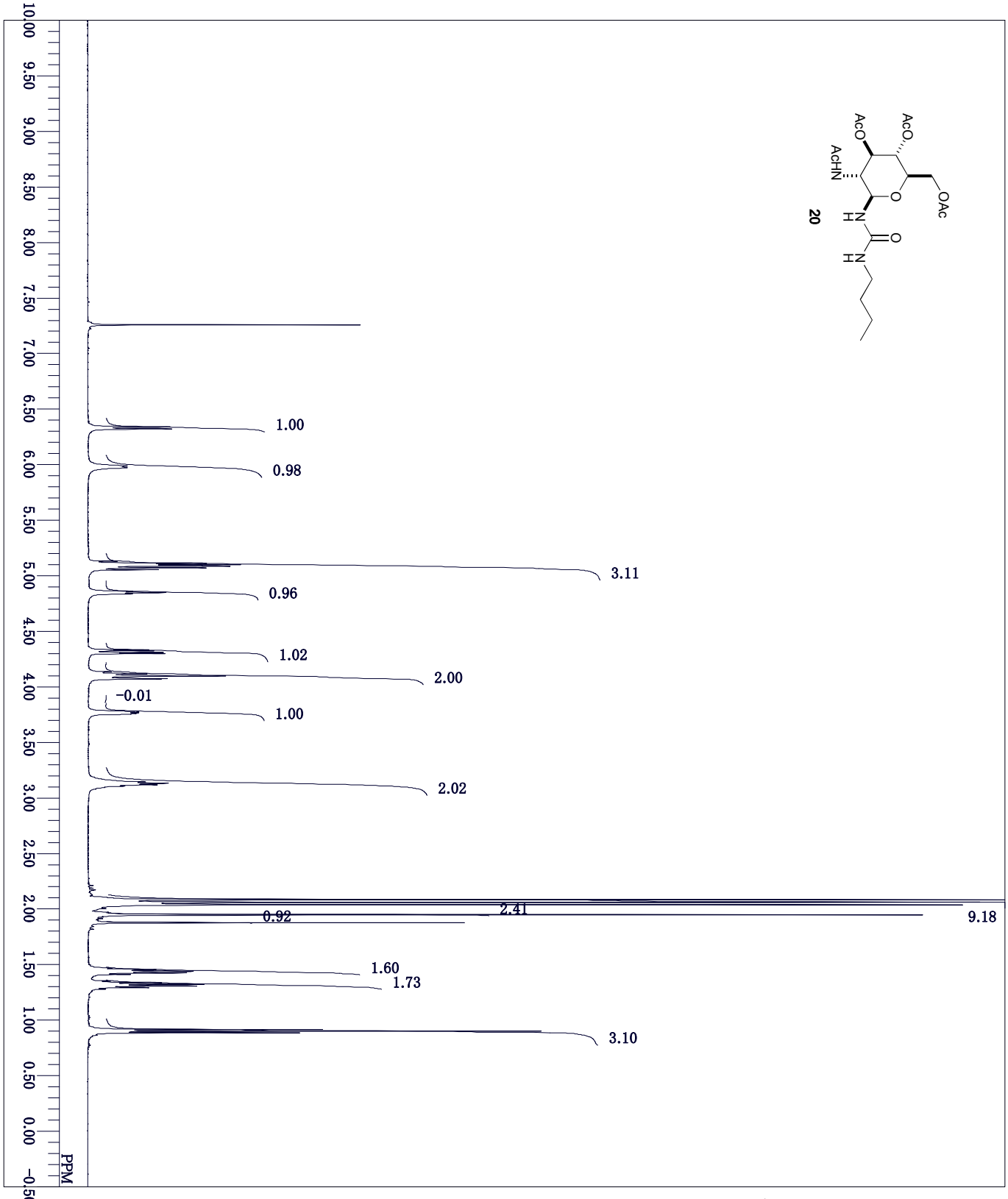
DFILE	sou3074data_proton-1-1.jdf
COMNT	single_pulse
DATIM	2011-08-26 10:31:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50



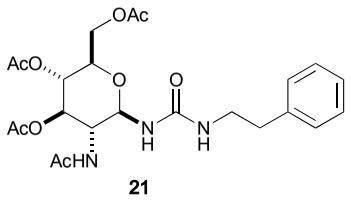
DFILE	sou3112data_proton-1-1.jdf
COMNT	single_pulse
DATIM	2011-08-26 10:40:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50



DFILE	sou4116data_proton-4-1.jdf
COMNT	single_pulse
DATIM	2011-11-30 21:01:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.8 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50

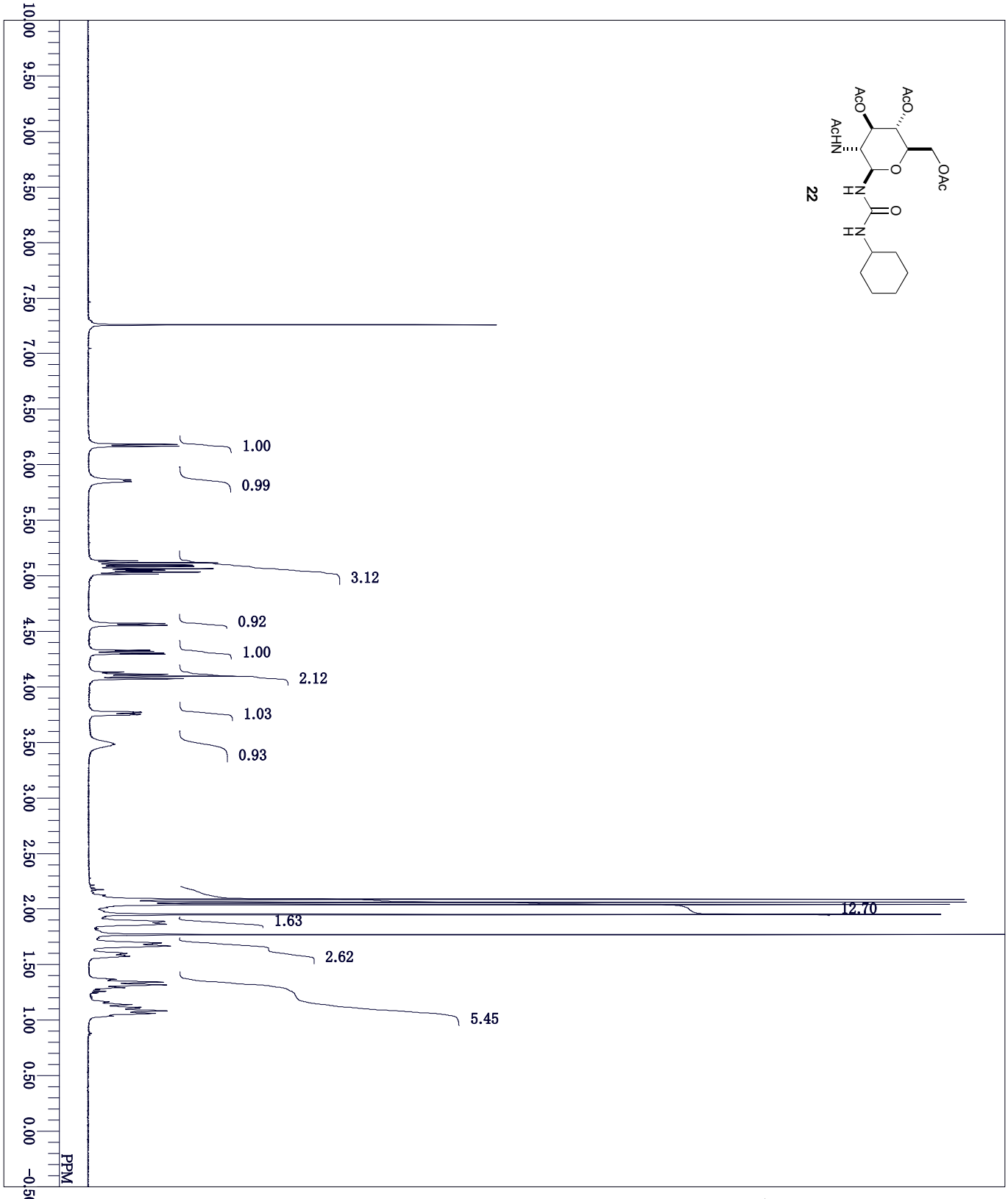


DFILE TON1146_proton-1-1.jdf
COMNT single_pulse
DATIM 2013-01-29 17:45:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.8 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50

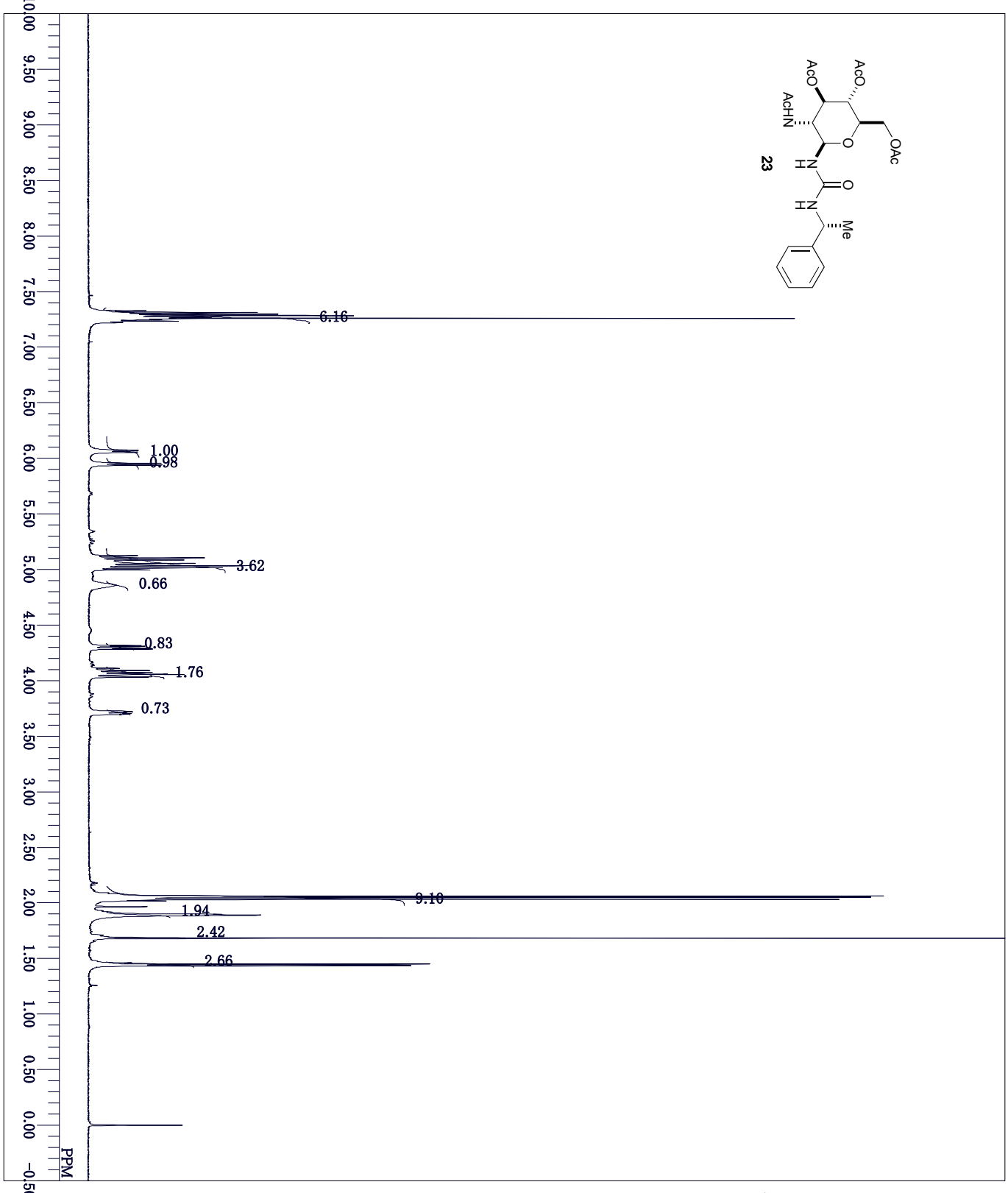


21

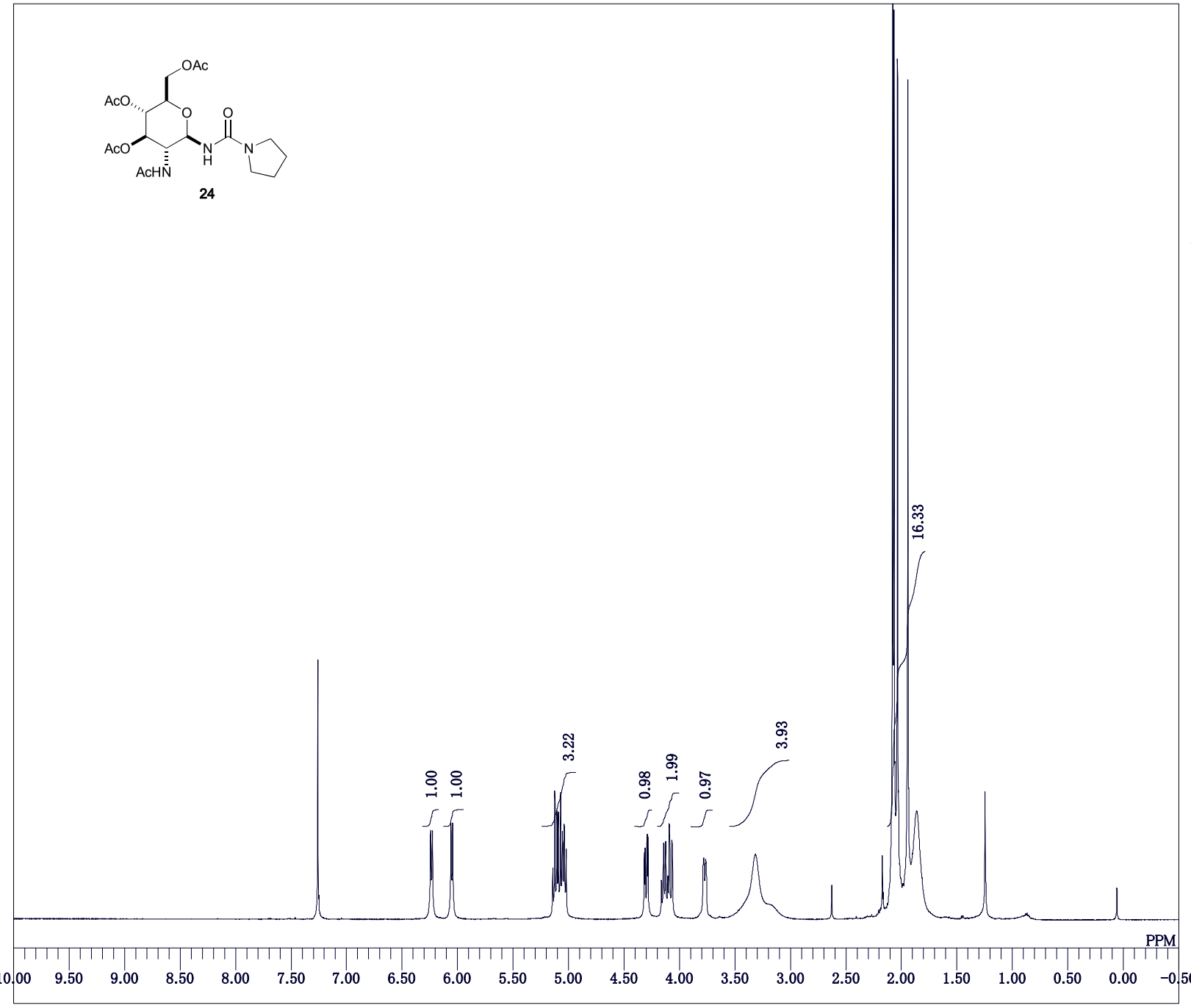
DFILE	TON1138_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-01-29 17:29:43
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	50

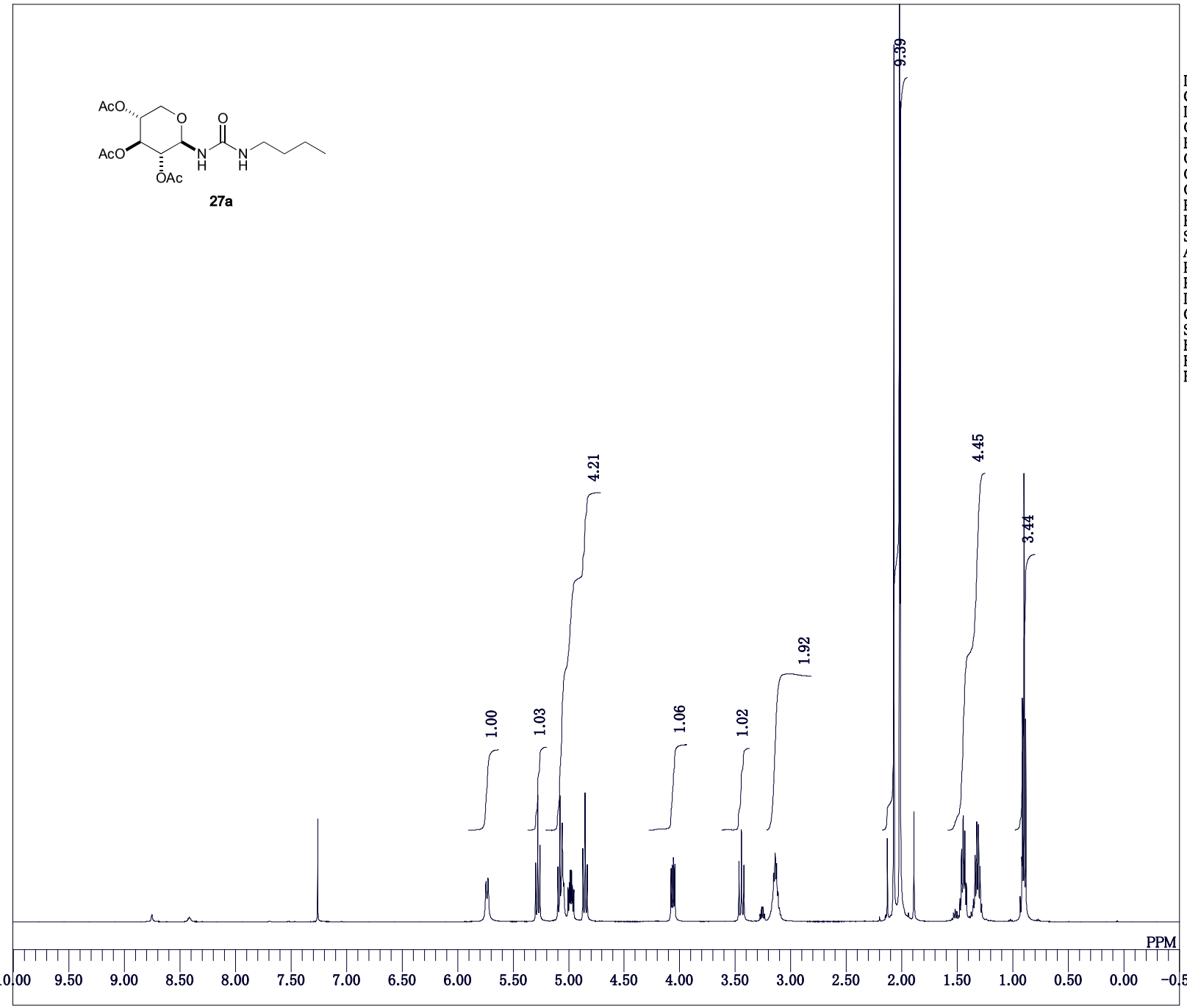


DFILE TON1130_proton-2-1.jdf
COMNT single_pulse
DATIM 2013-02-07 15:56:00
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.4 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50

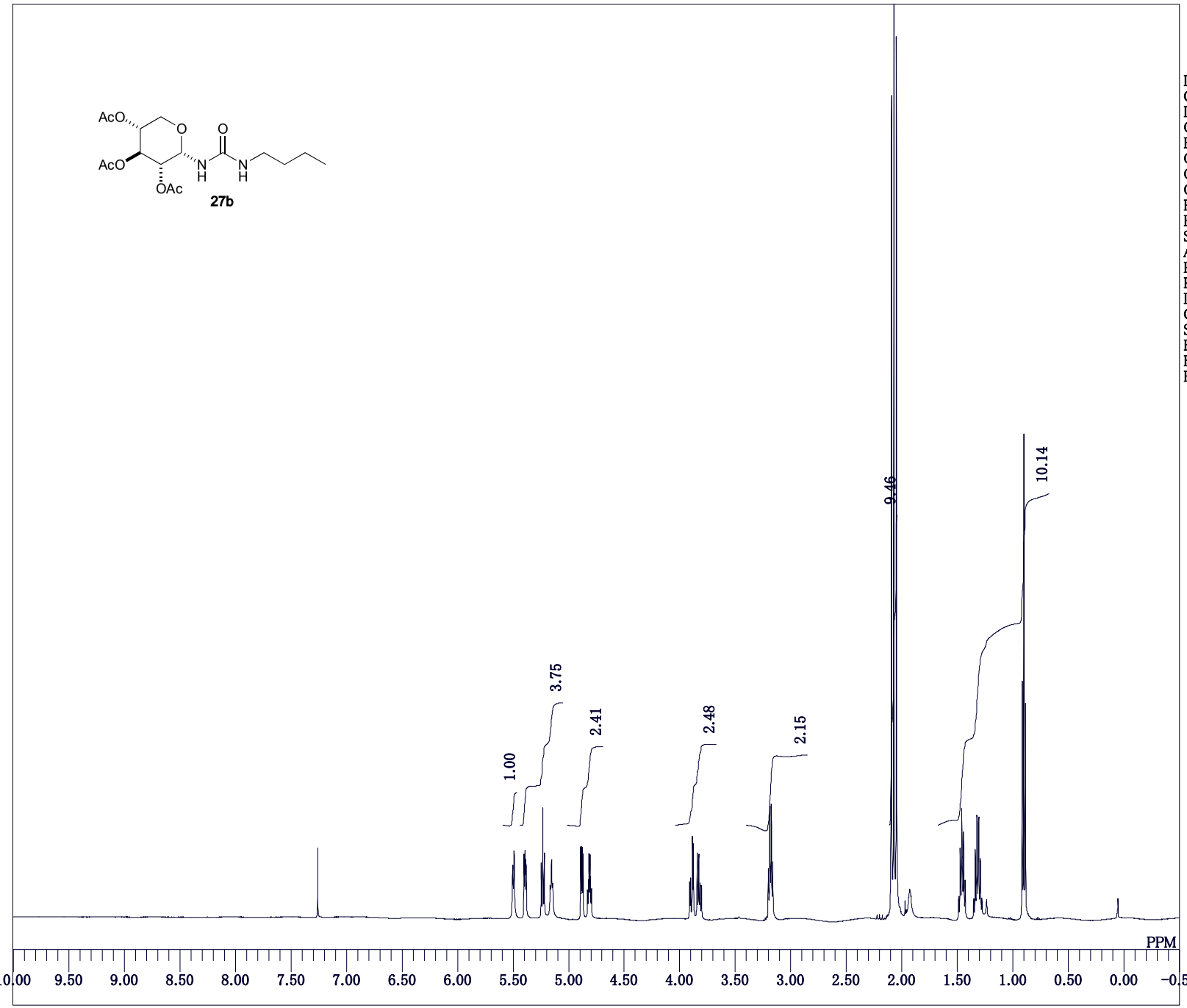


DFILE TON1142_proton-1-1.jdf
COMNT single_pulse
DATIM 2013-01-29 17:37:22
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.6 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50

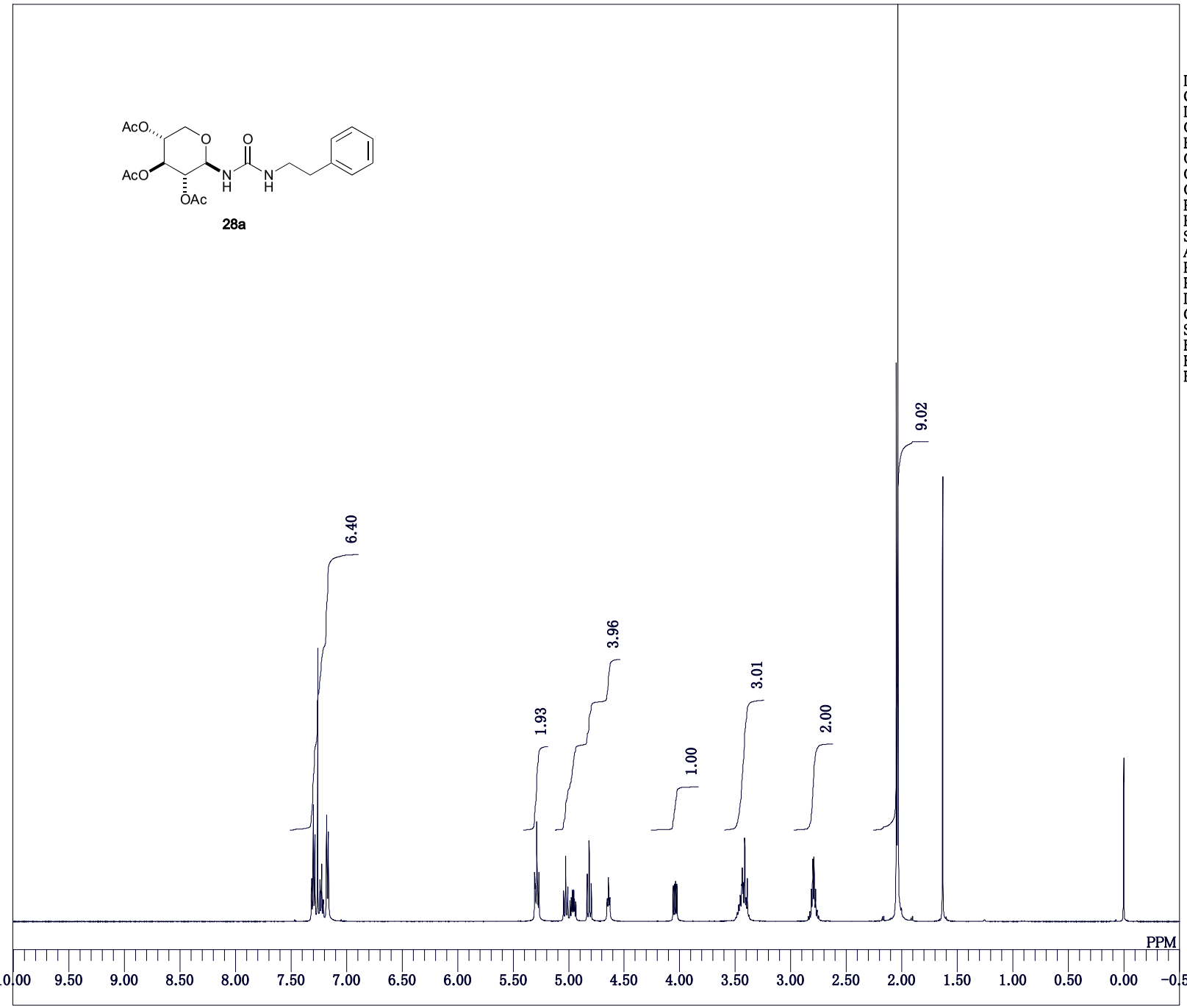

OAc
AcO
AcO
AcHN
O
O
N
H
N
24
DFILE TON1_proton-1-1.jdf
COMNT single_pulse
DATIM 2013-02-12 09:12:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.5 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50
1.00
1.00
3.22
0.98
1.99
0.97
3.93
16.33
PPM
10.00 9.50 9.00 8.50 8.00 7.50 7.00 6.50 6.00 5.50 5.00 4.50 4.00 3.50 3.00 2.50 2.00 1.50 1.00 0.50 0.00 -0.50



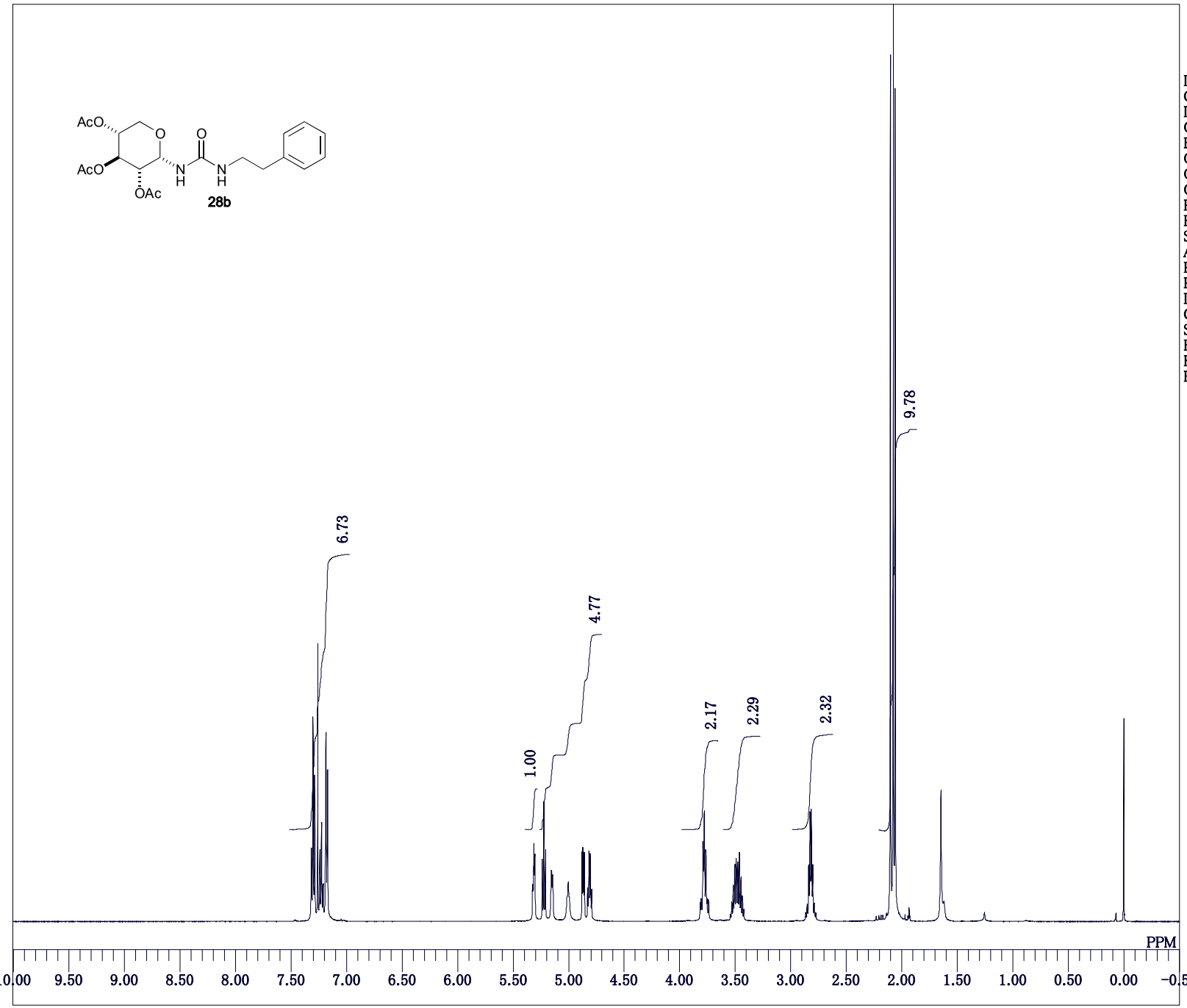
DFILE	SAE1056_proton-1-1.jdf
COMNT	single_pulse
DATIM	2012-02-25 10:34:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.5 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	36



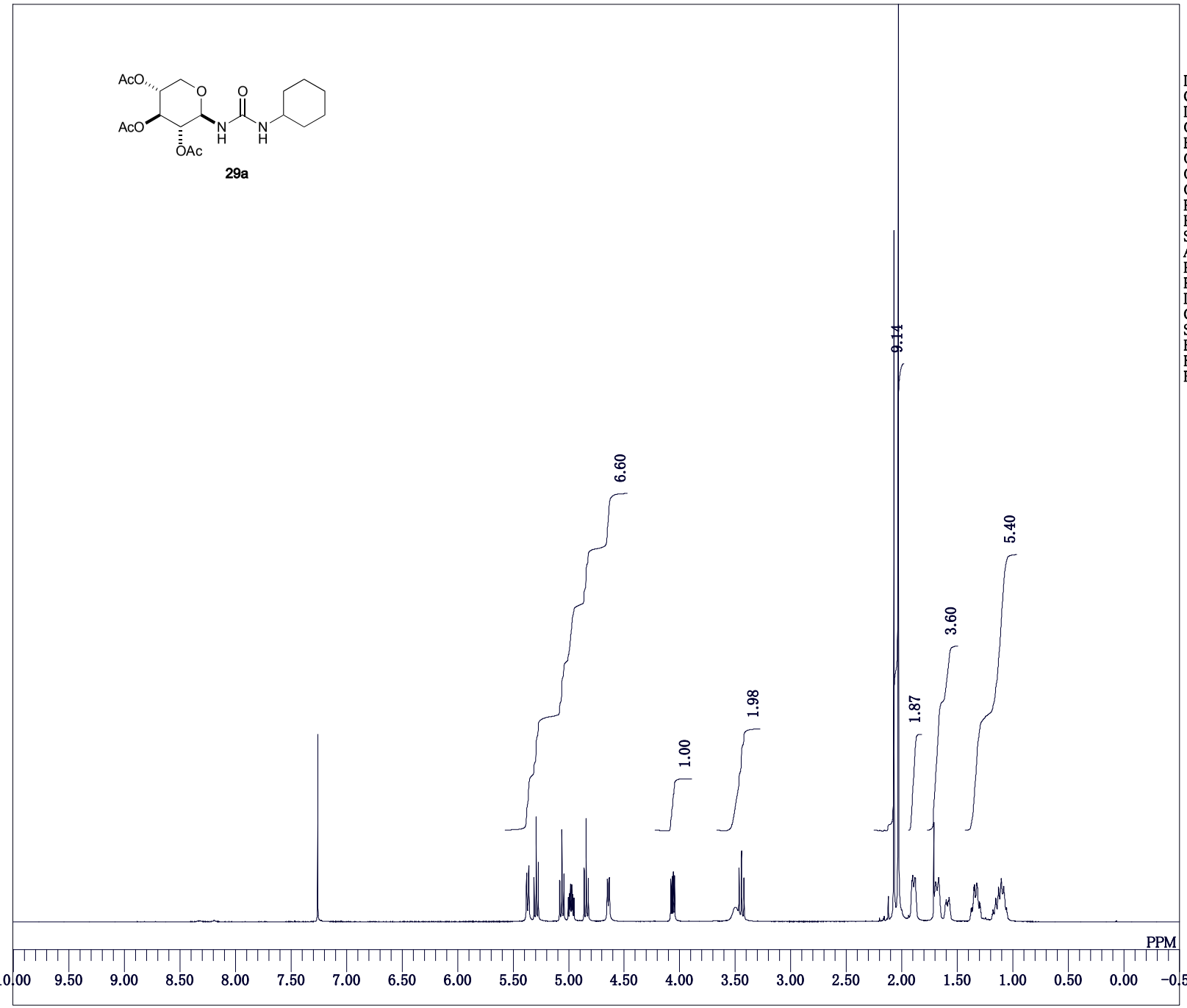
DFILE	SAE2057_proton-1-1.jdf
COMNT	single_pulse
DATIM	2012-07-09 22:11:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50

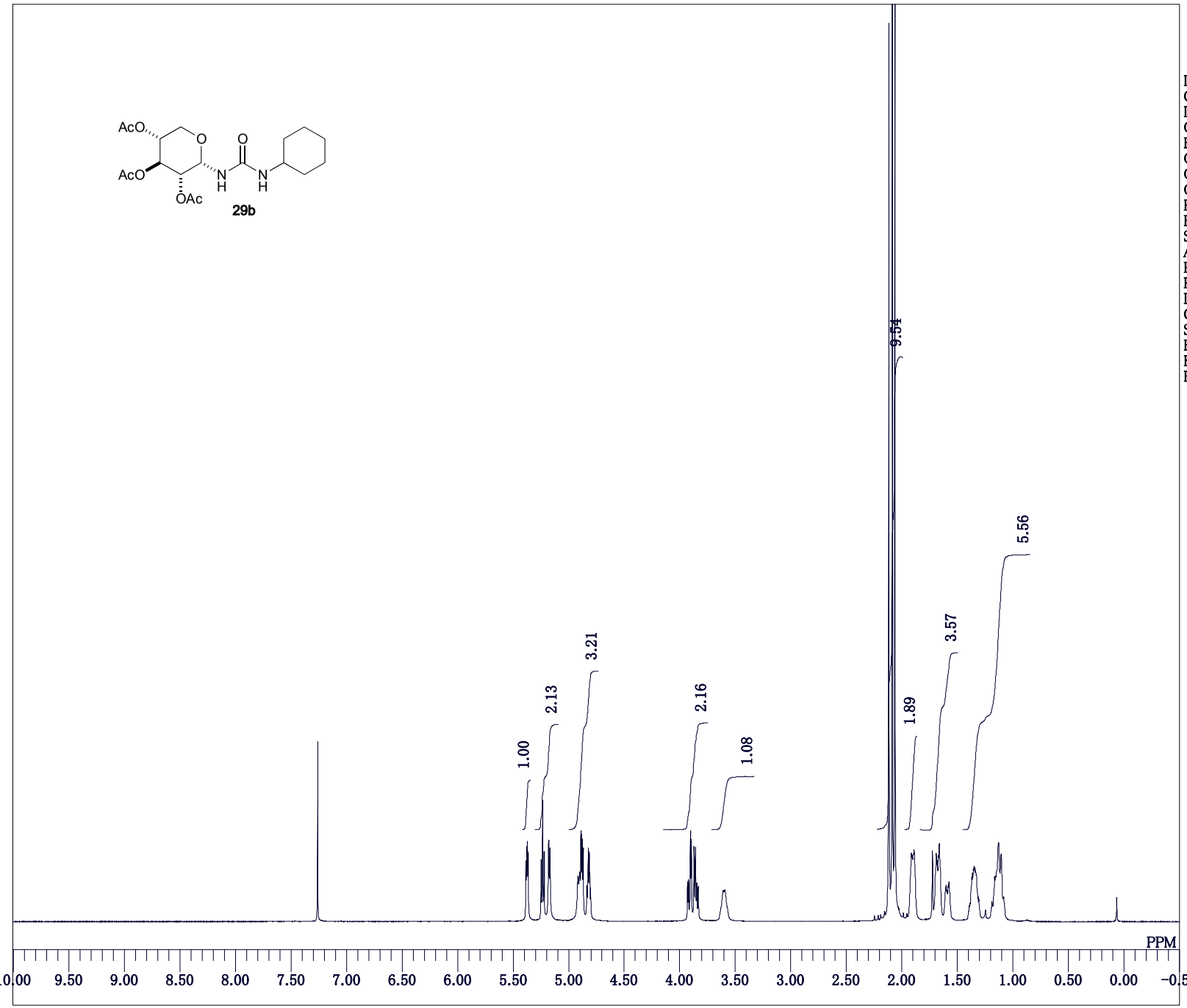


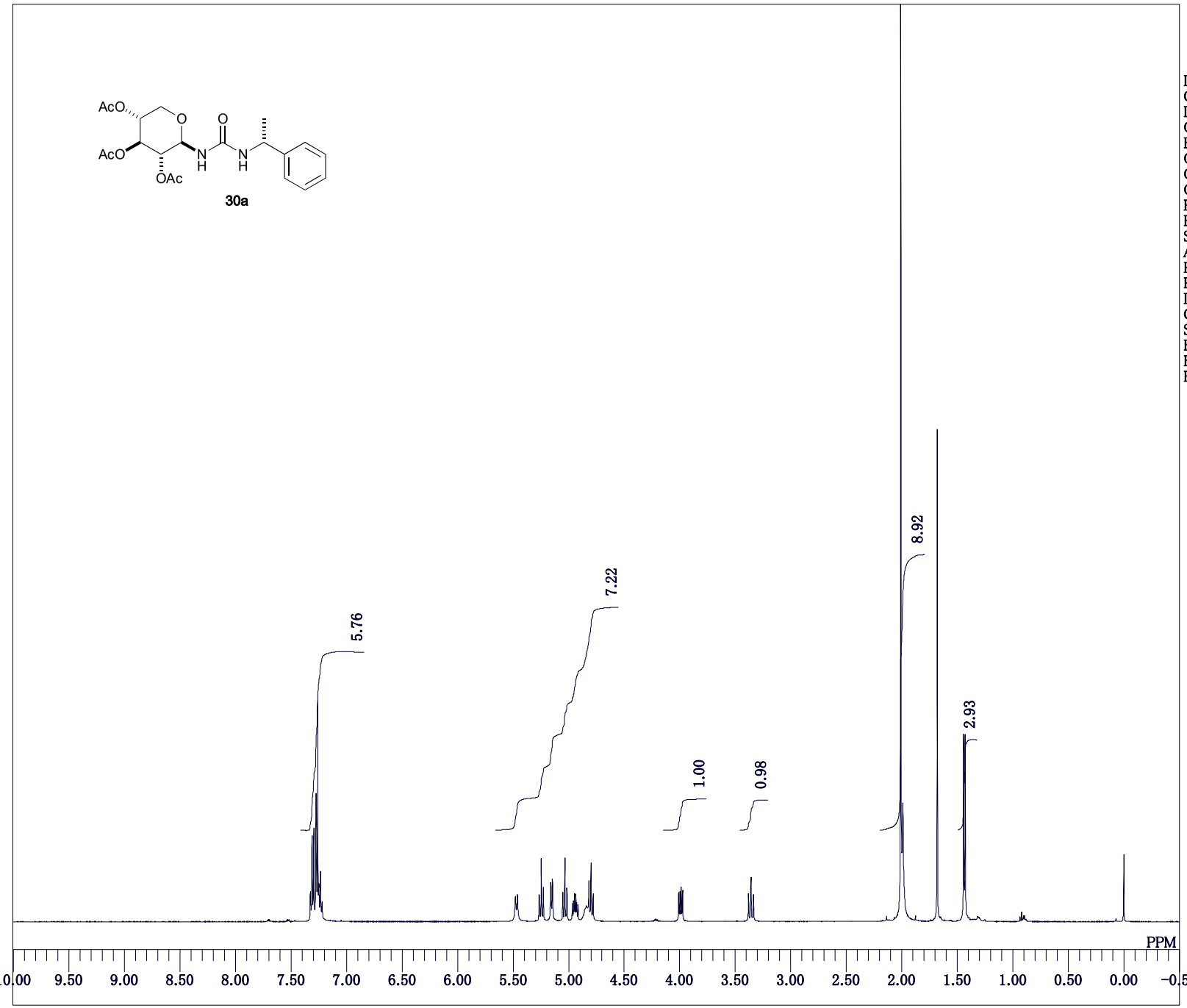
DFILE	SAE1191_proton-2-1.jdf
COMNT	single_pulse
DATIM	2012-04-09 17:27:59
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



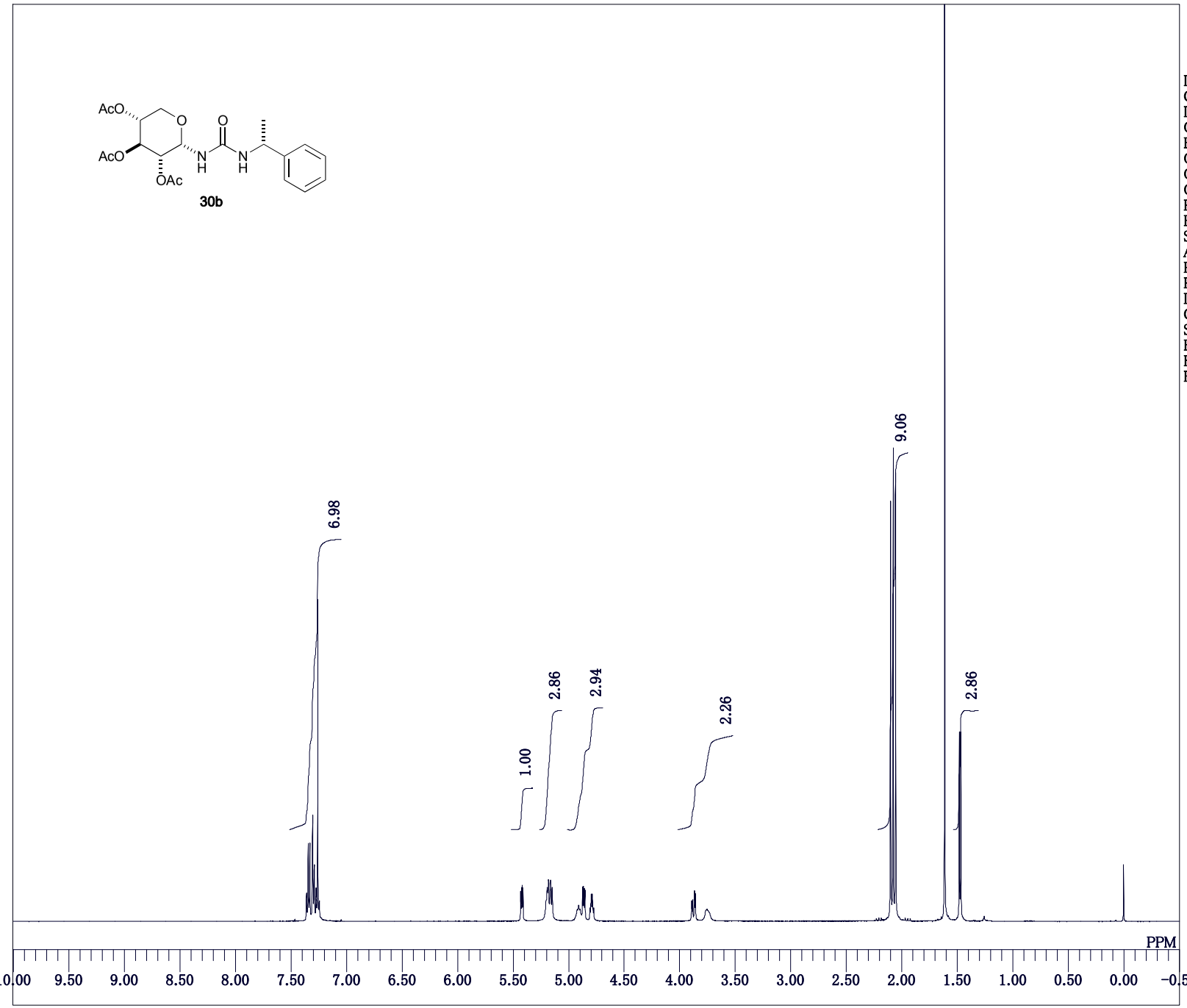
DFILE	SAE2055_proton-6-1.jdf
COMNT	single_pulse
DATIM	2012-07-12 17:40:42
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50


AcO
AcO
OAc
O
O
N
H
N
H
29a
DFILE SAE1047_proton-2-1.jdf
COMNT single_pulse
DATIM 2012-02-19 12:11:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.3 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 1.00 Hz
RGAIN 50
6.60
1.00
1.98
9.14
1.87
3.60
5.40
PPM
10.00 9.50 9.00 8.50 8.00 7.50 7.00 6.50 6.00 5.50 5.00 4.50 4.00 3.50 3.00 2.50 2.00 1.50 1.00 0.50 0.00 −0.50

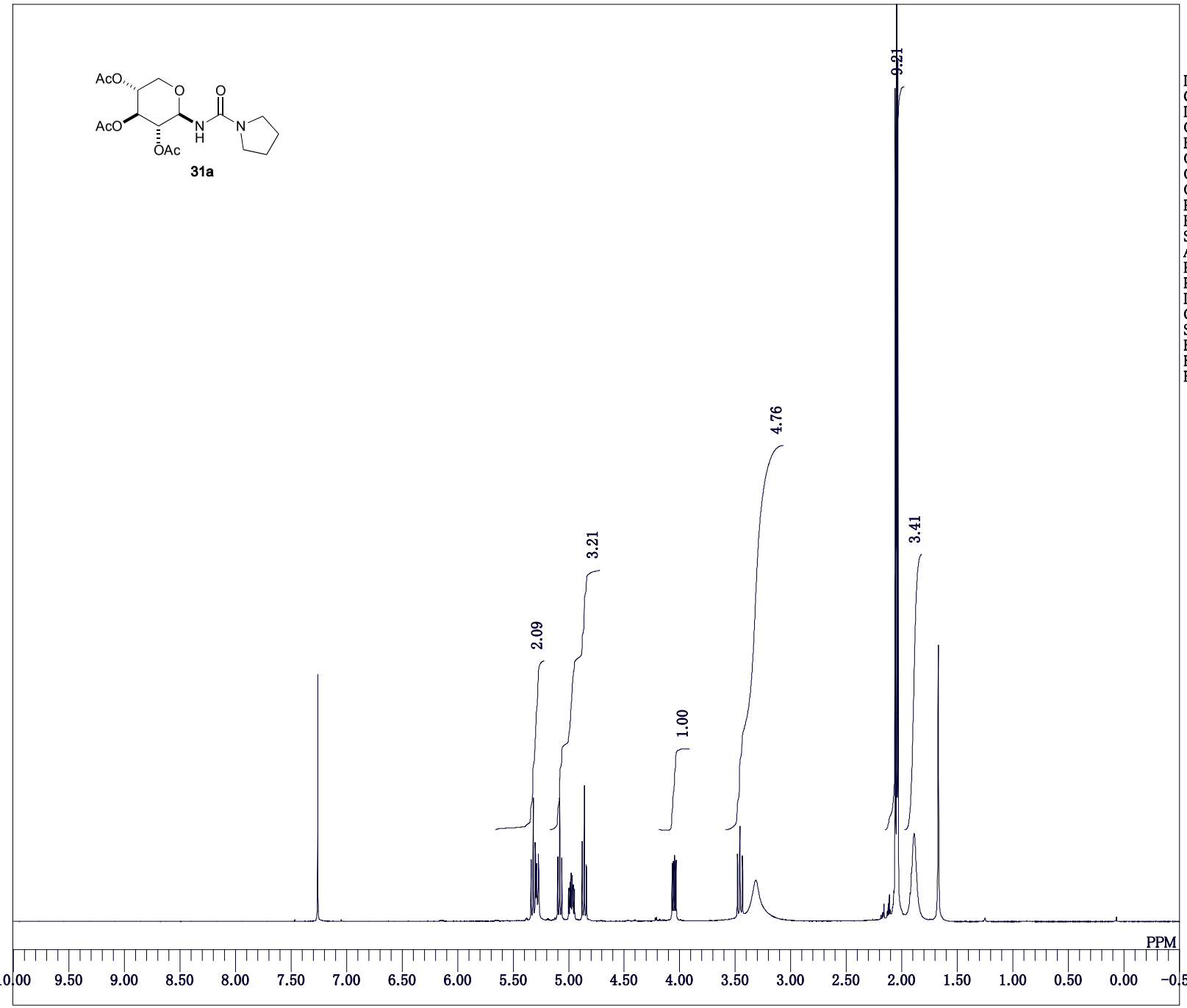

AcO
AcO
OAc
O
N
H
O
N
H
29b
DFILE sou4118_proton-1-1.jdf
COMNT single_pulse
DATIM 2011-11-22 14:33:28
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16400
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 16.6 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 1.00 Hz
RGAIN 50
1.00
2.13
3.21
2.16
1.08
9.54
1.89
3.57
5.56
PPM
10.00
9.50
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
−0.50



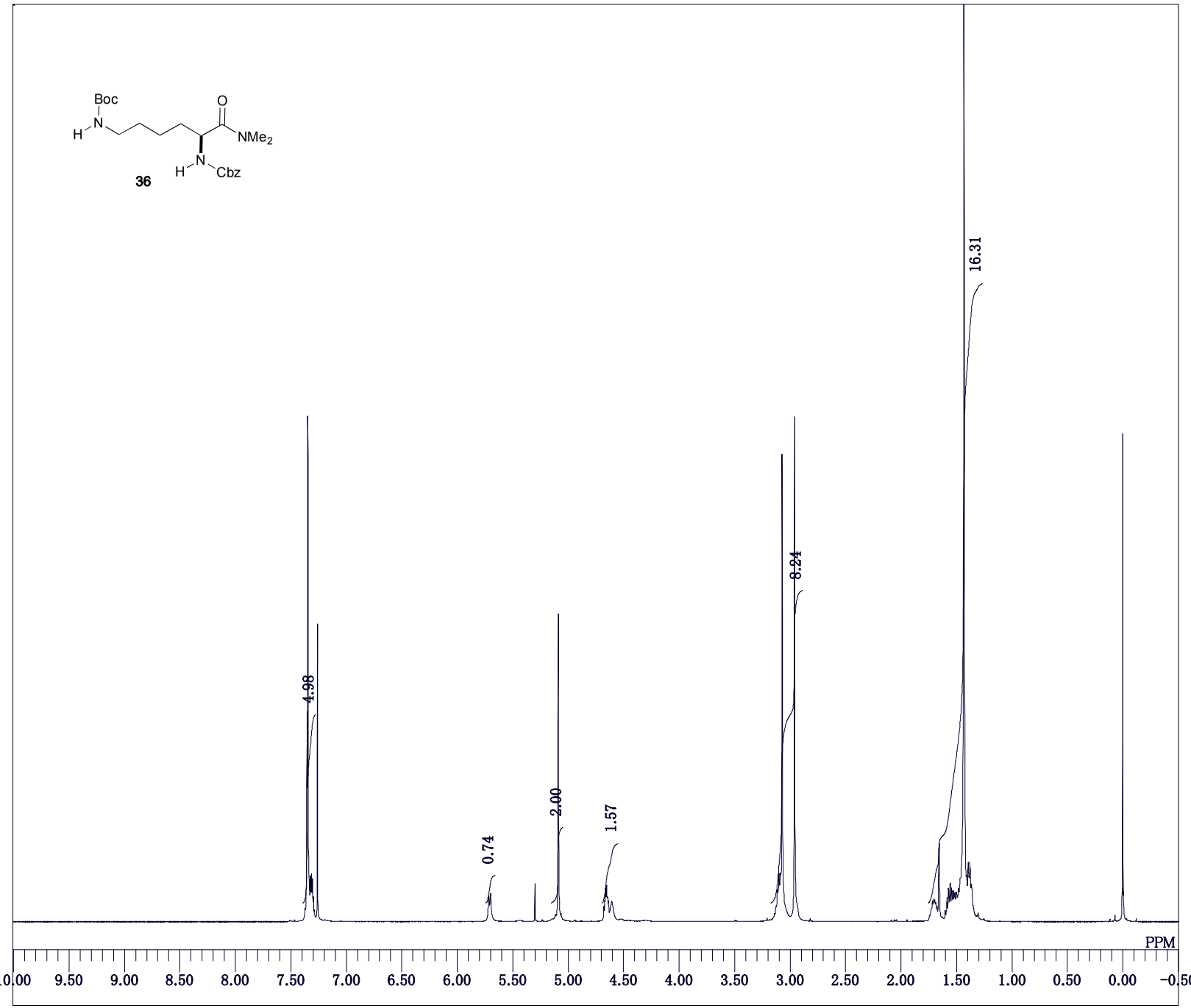
DFILE	SAE20-1_proton-2-1.jdf
COMNT	single_pulse
DATIM	2012-06-11 11:15:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



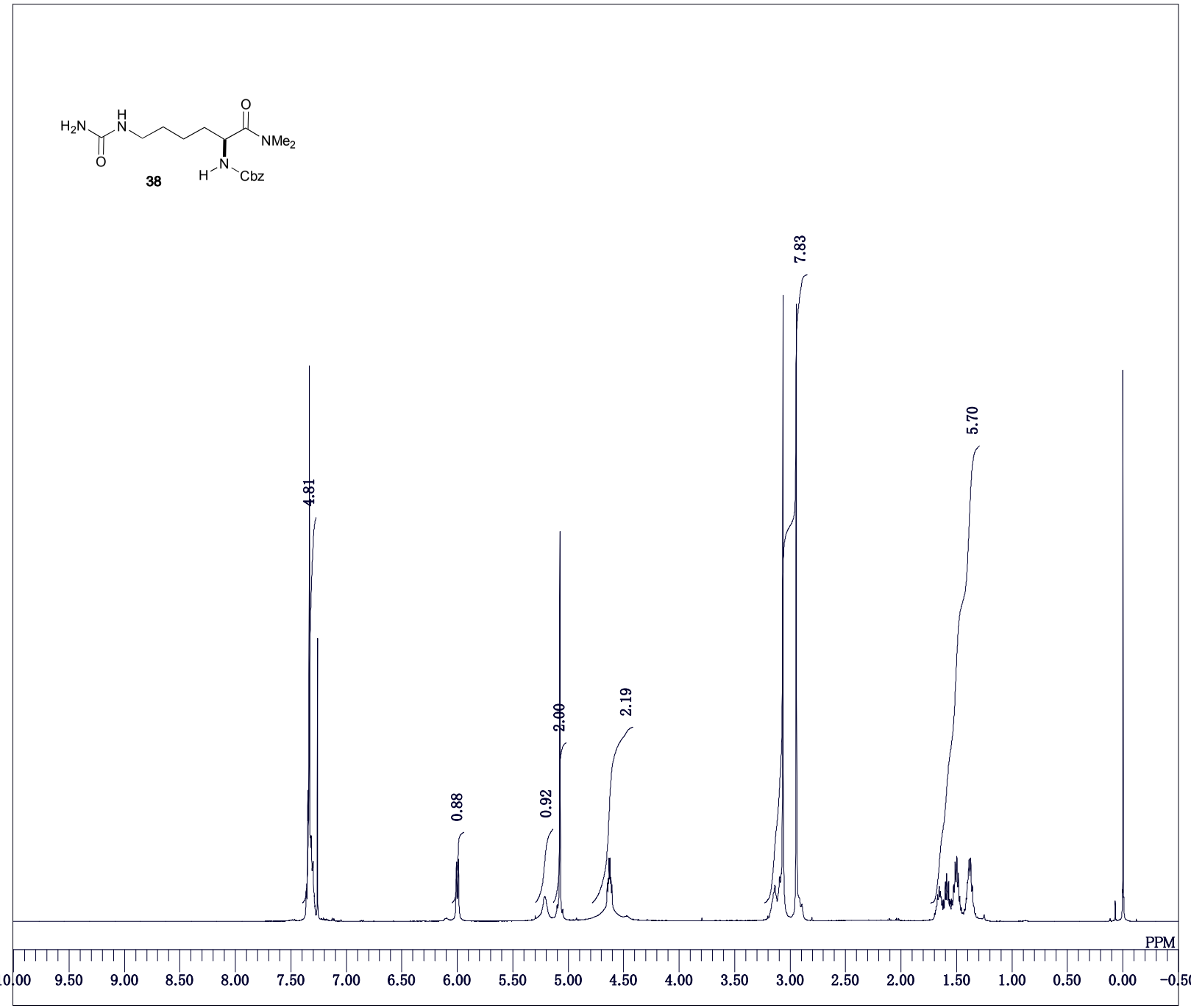
DFILE	SAE2017_proton-5-1.jdf
COMNT	single_pulse
DATIM	2012-07-31 21:48:55
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



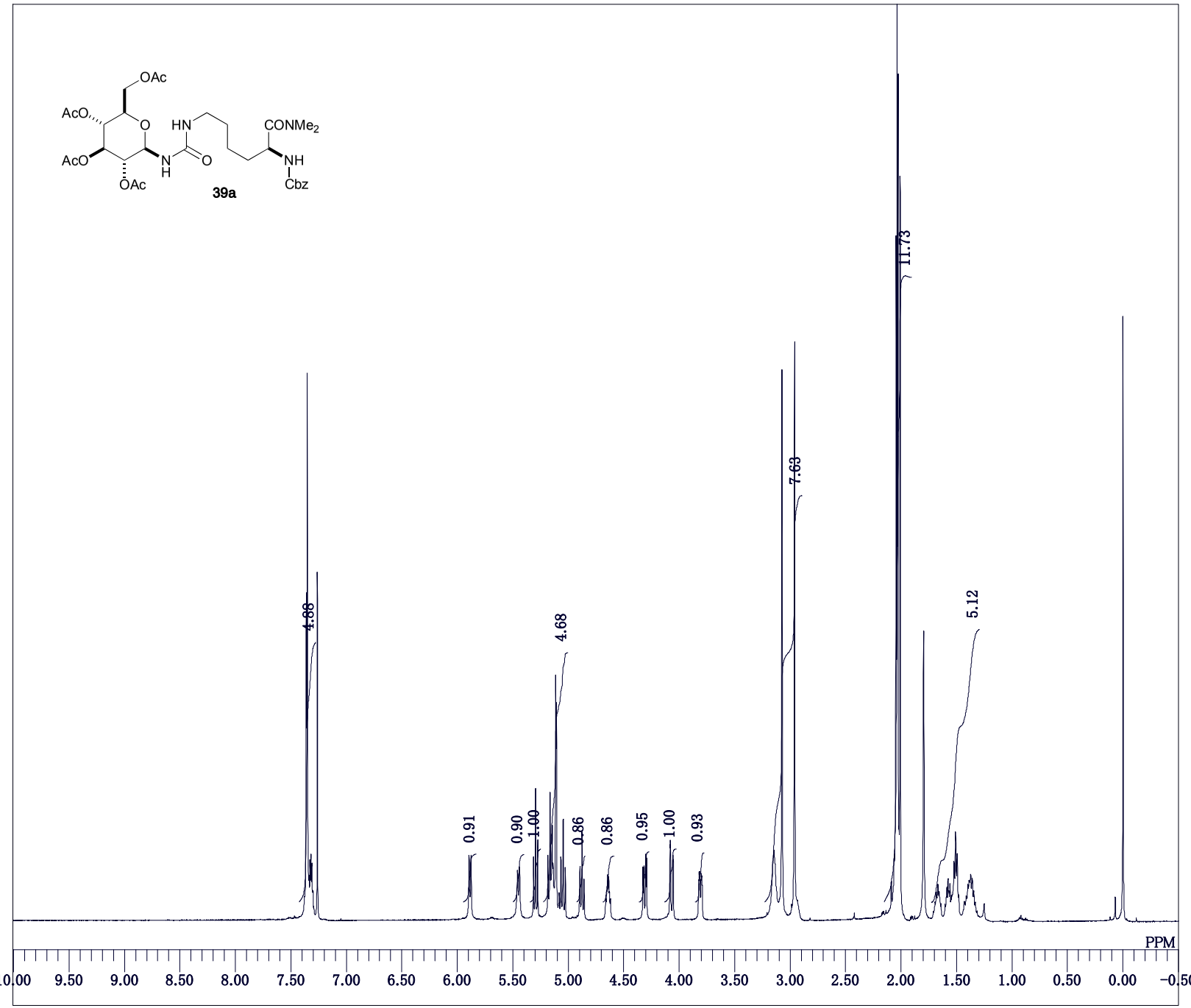
DFILE	SAE2051_proton-1-1.jdf
COMNT	single_pulse
DATIM	2012-07-04 15:52:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



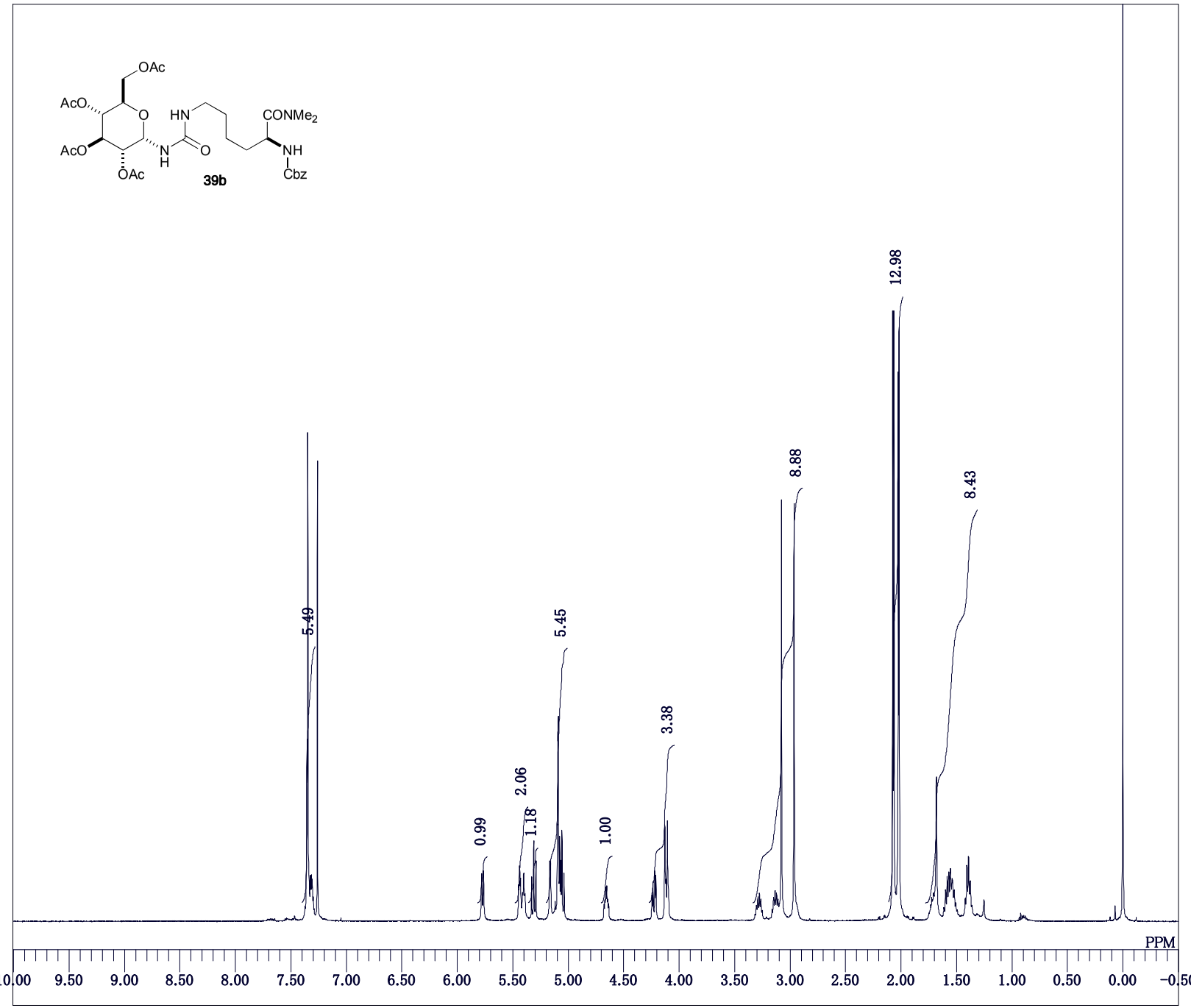
DFILE	sou3009data_proton-4-1.jdf
COMNT	single_pulse
DATIM	2011-11-25 14:57:13
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



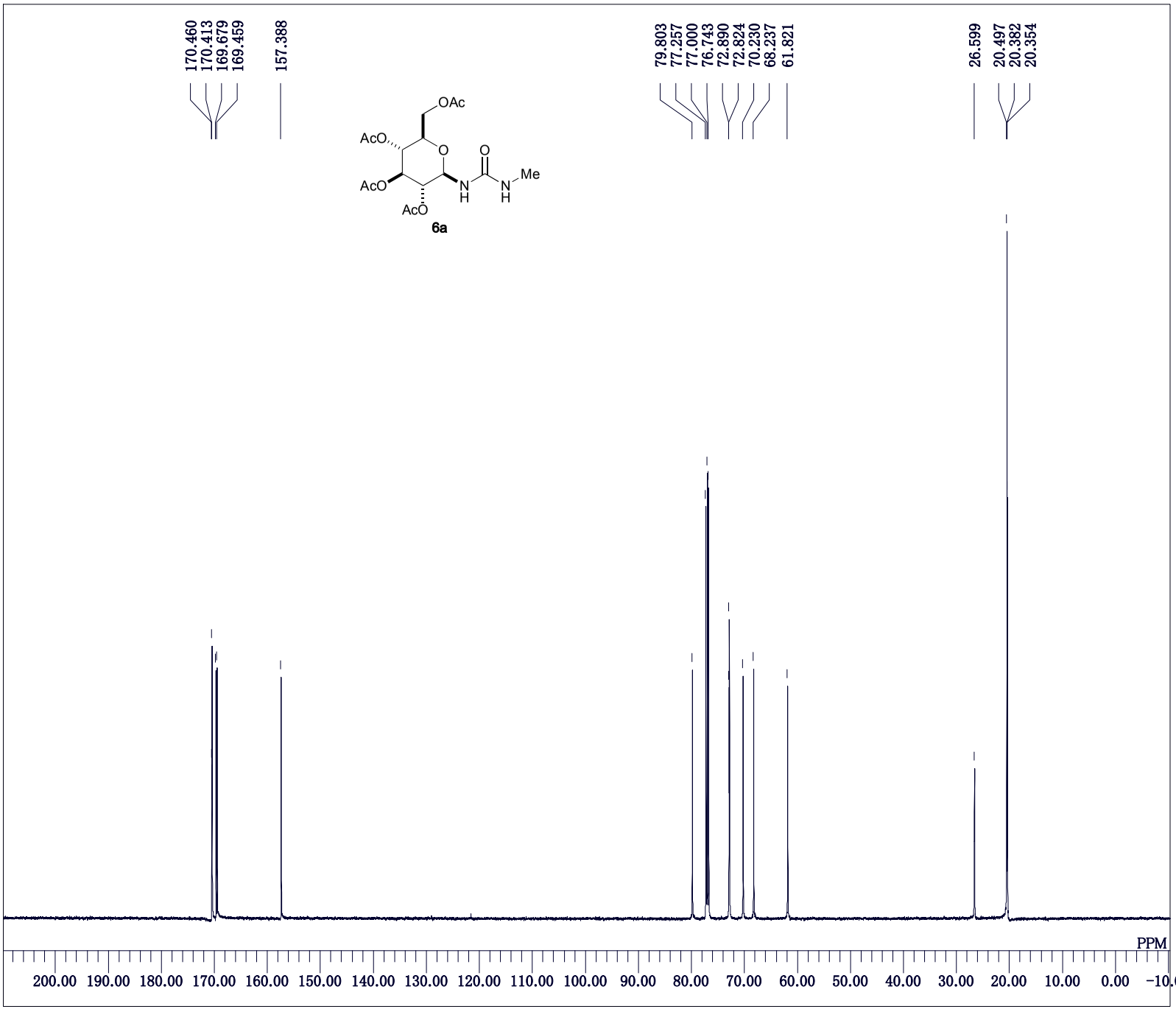
DFILE	sou4074data_proton-2-1.jdf
COMNT	single_pulse
DATIM	2011-11-25 14:48:08
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



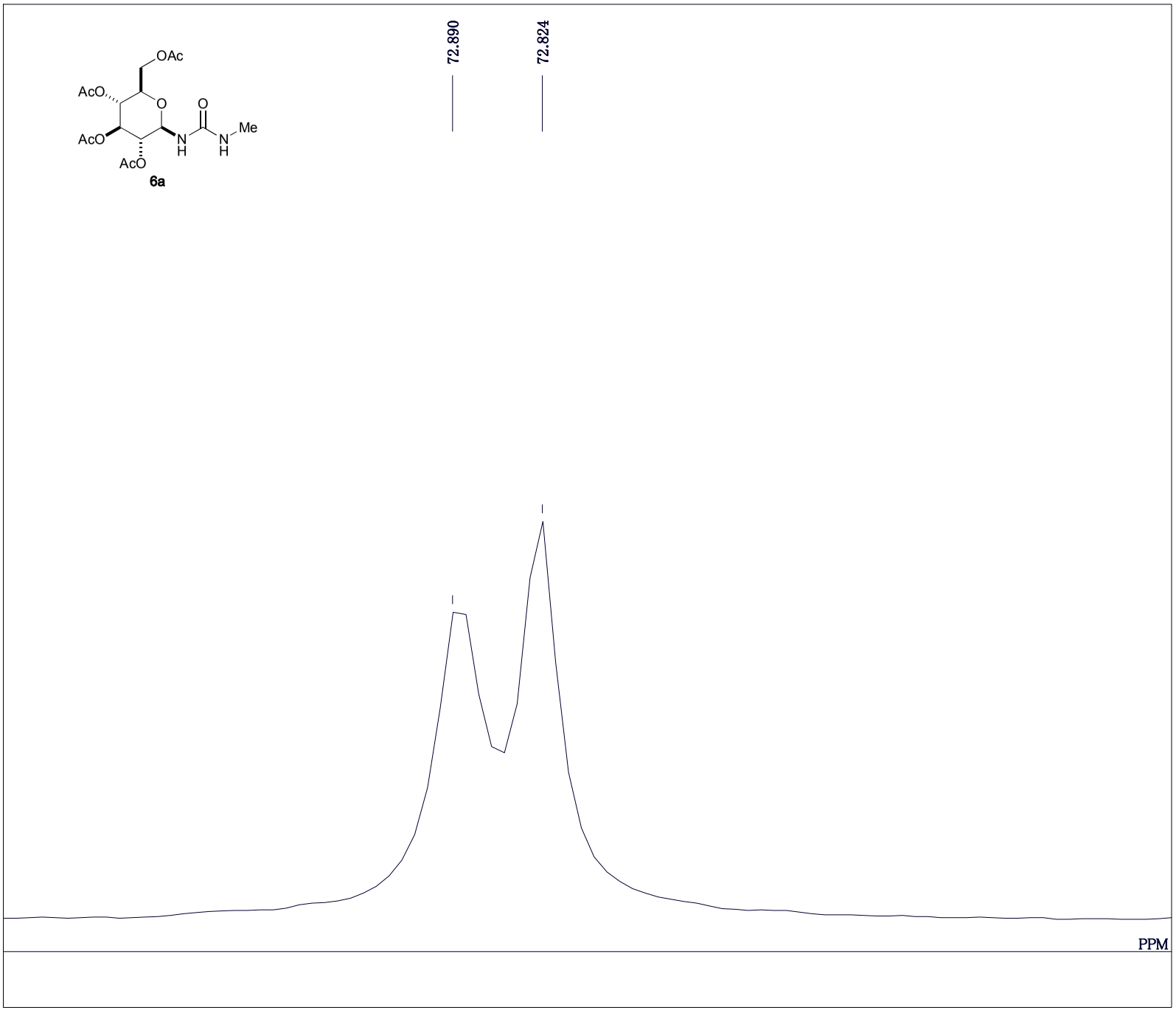
DFILE	sou3015data_proton-3-1.jdf
COMNT	single_pulse
DATIM	2011-11-30 20:51:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.9 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



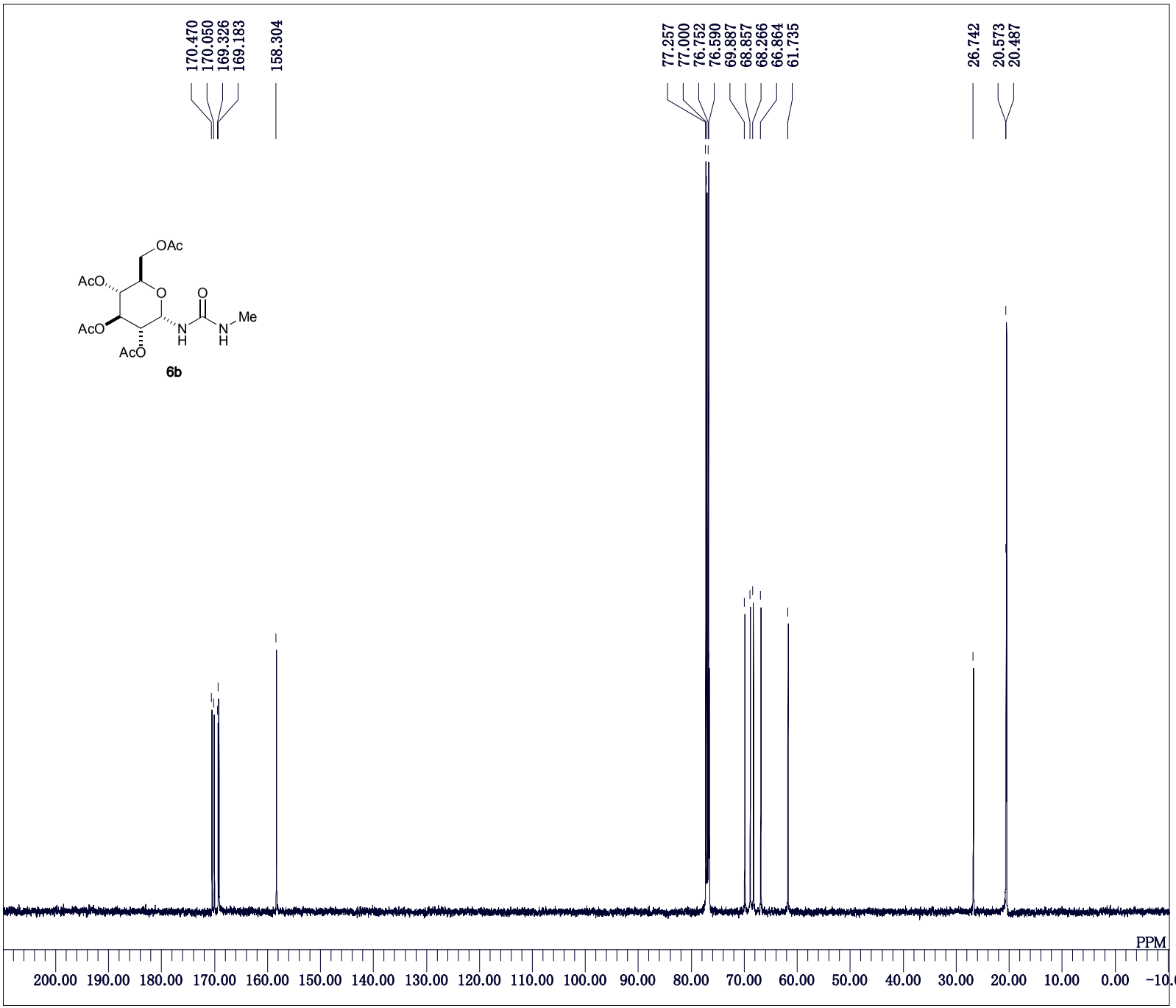
DFILE	sou3012data_proton-2-1.jdf
COMNT	single_pulse
DATIM	2011-11-22 10:02:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16400
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	16.8 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	1.00 Hz
RGAIN	50



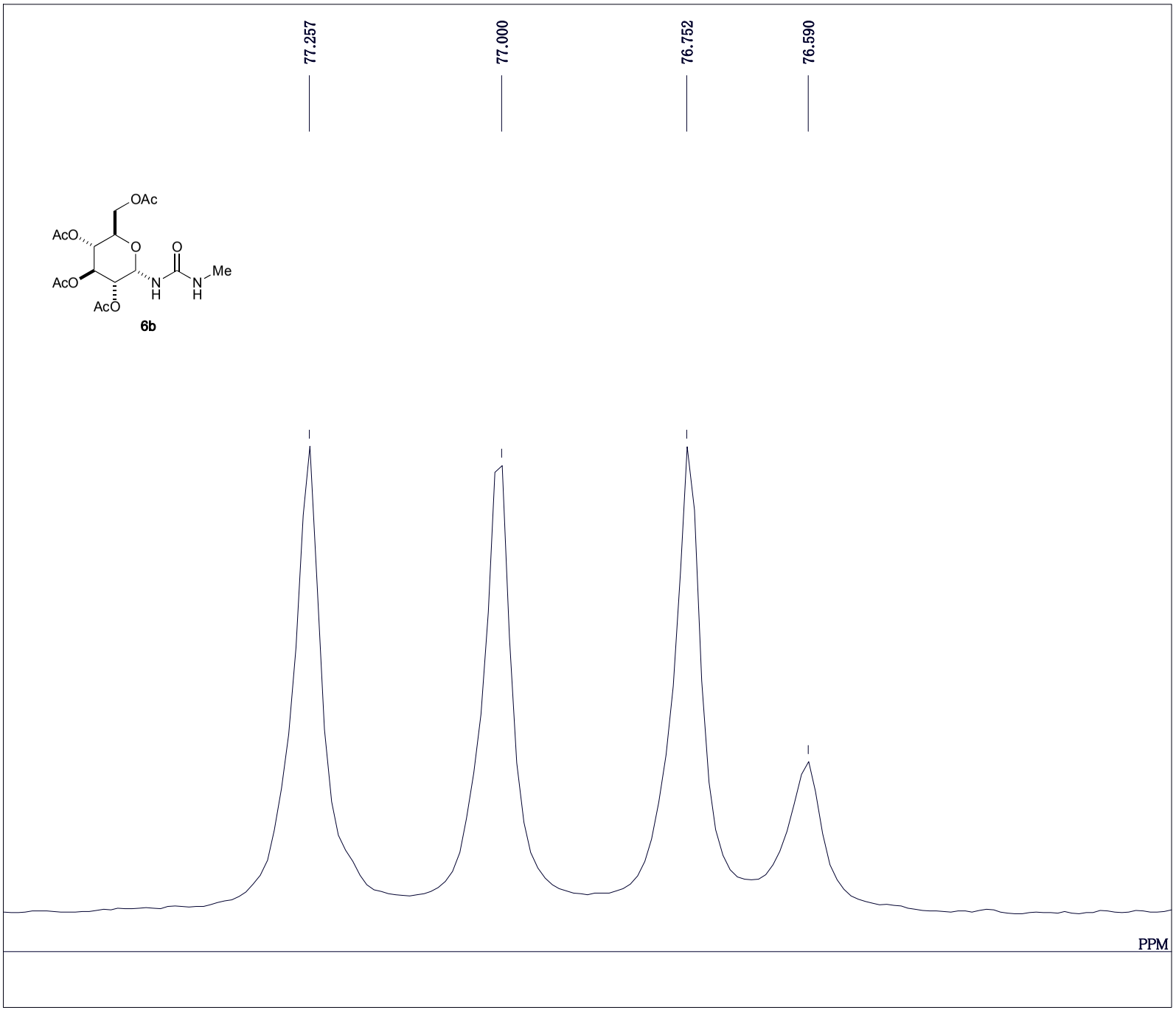
DFILE	sou3080data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 15:13:33
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



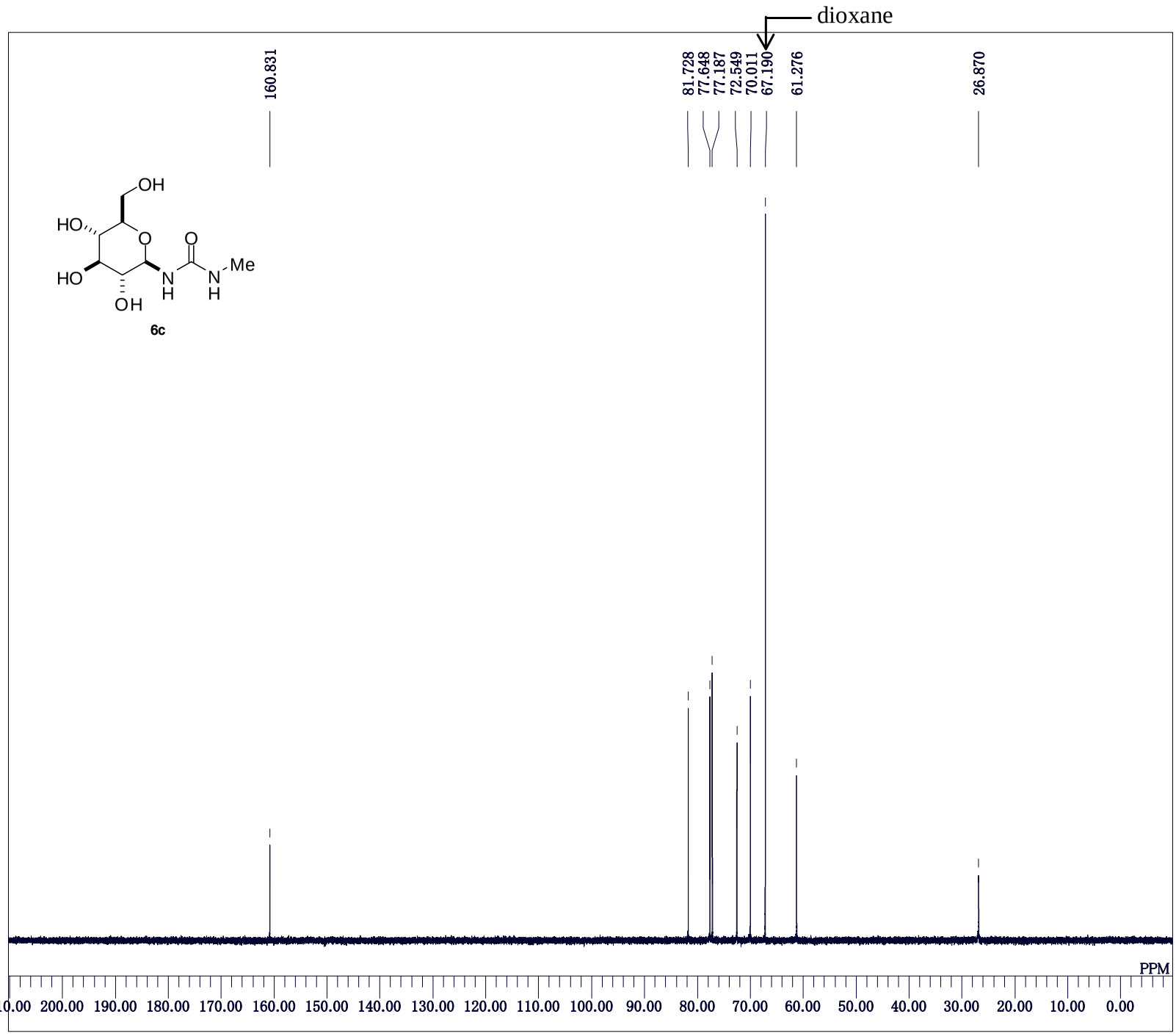
DFILE	sou3080data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 15:13:33
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



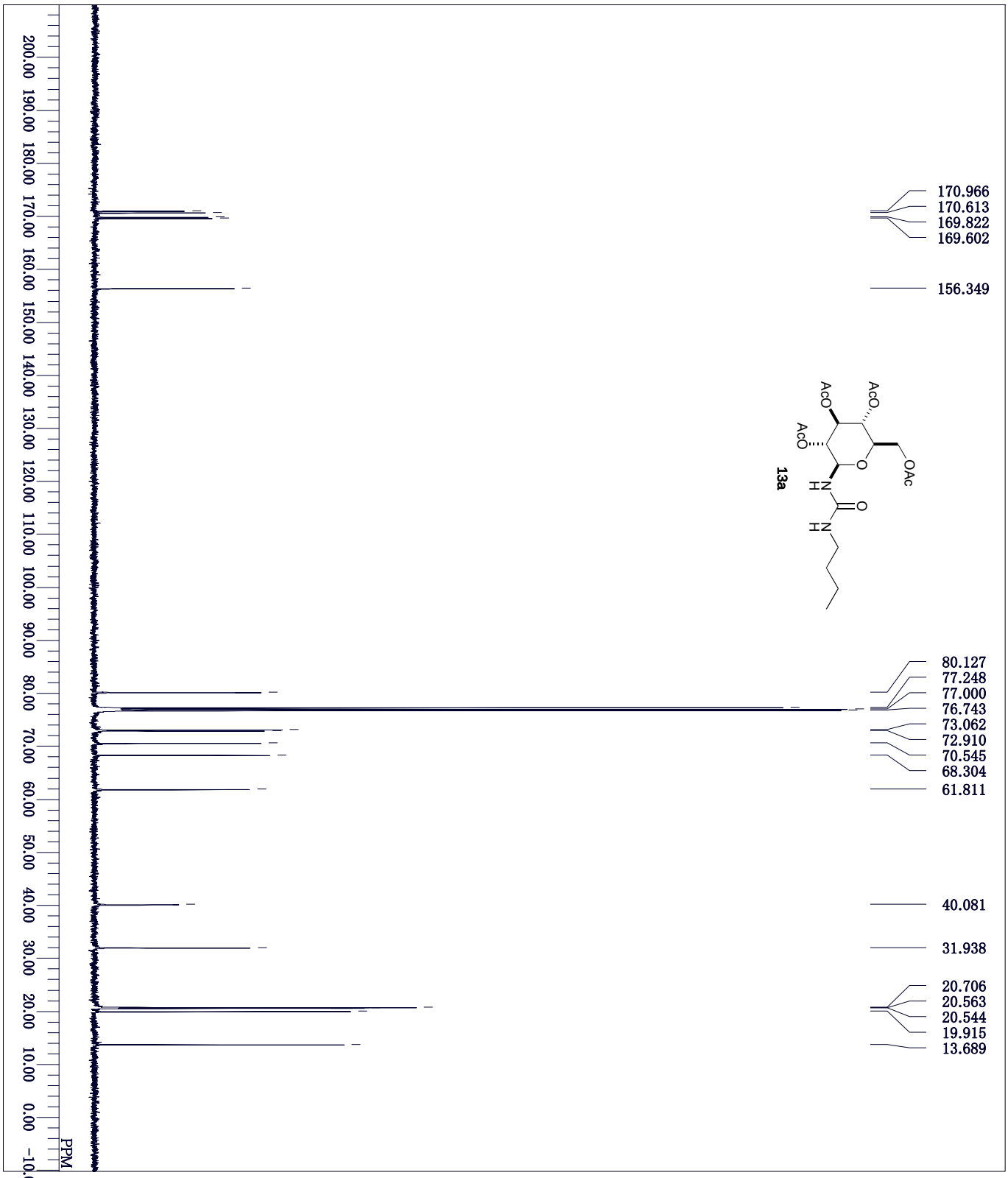
DFILE	sou4117data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-14 10:32:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



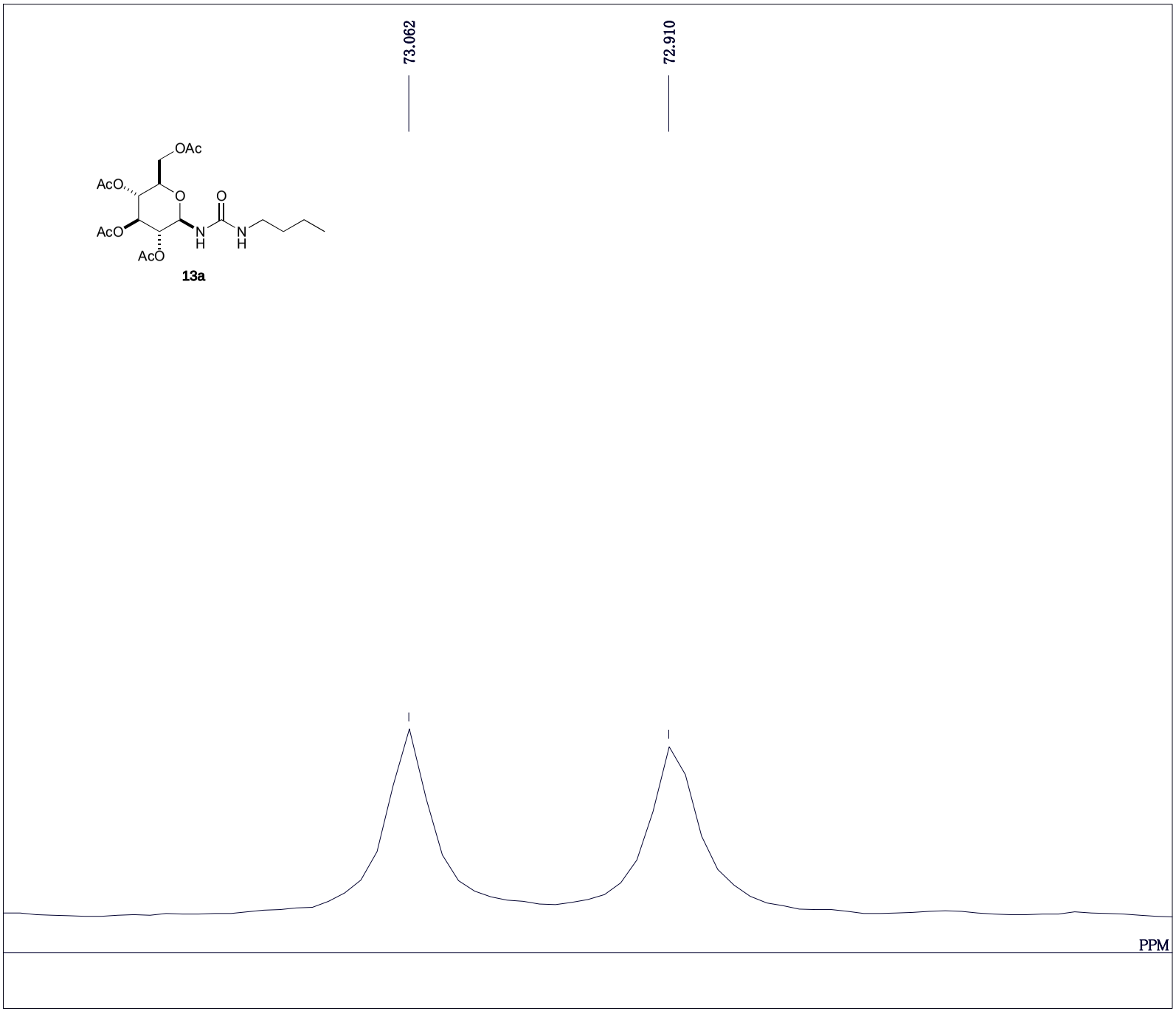
DFILE	sou4117data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-14 10:32:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



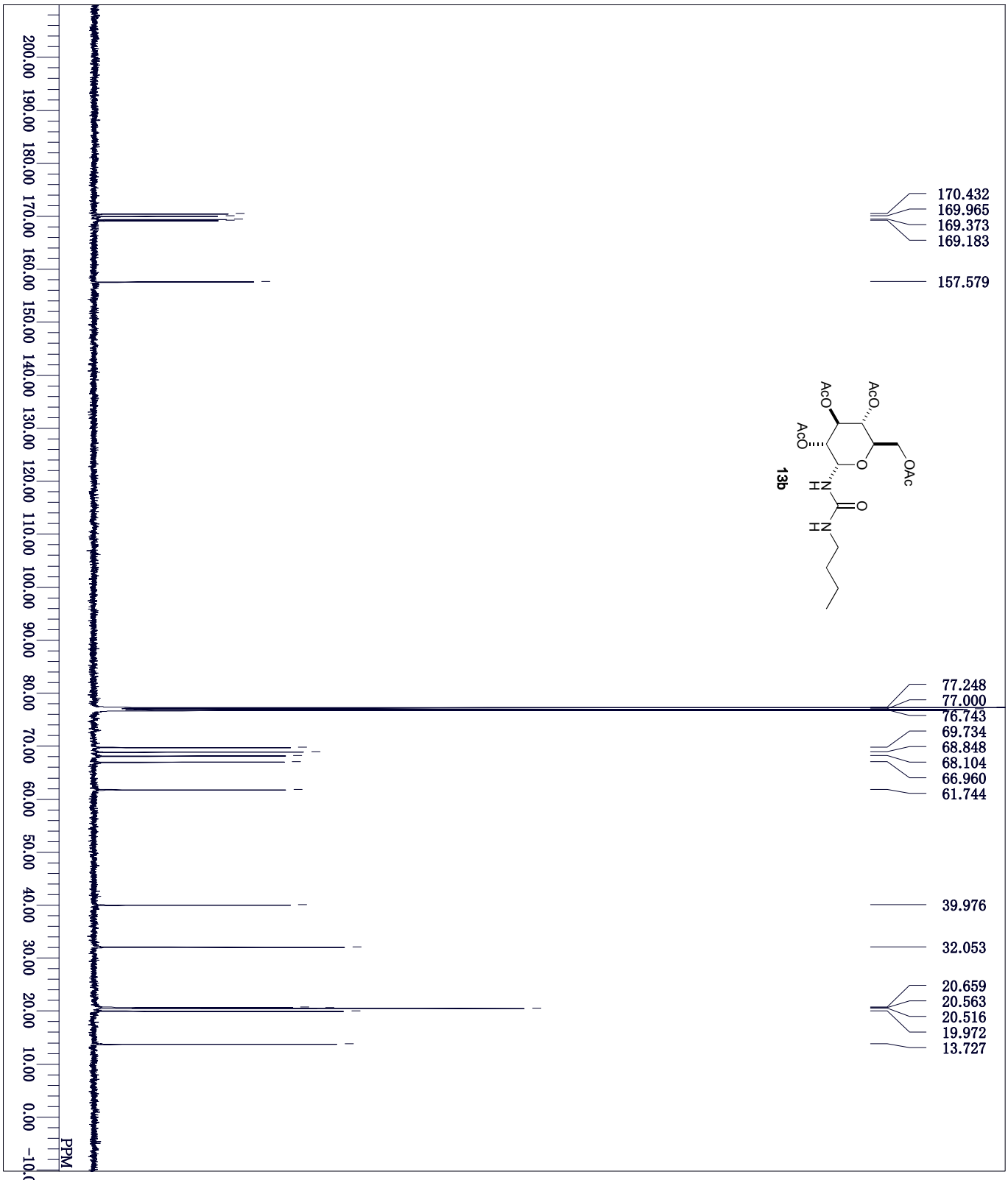
DFILE	SAE2159-2-carbon-dioxane.als
COMNT	
DATIM	Mon Feb 17 21:13:56 2014
OBNUC	13C
EXMOD	bcm
OBFRQ	100.40 MHz
OBSET	0.00 KHz
OBFIN	135500.00 Hz
POINT	1048576
FREQU	27100.27 Hz
SCANS	420
ACQTM	1.2091 sec
PD	1.7909 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	D2O
EXREF	67.19 ppm
BF	0.12 Hz
RGAIN	29



DFILE sou3140data_Carbon-2-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2011-11-09 10:18:24
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 50

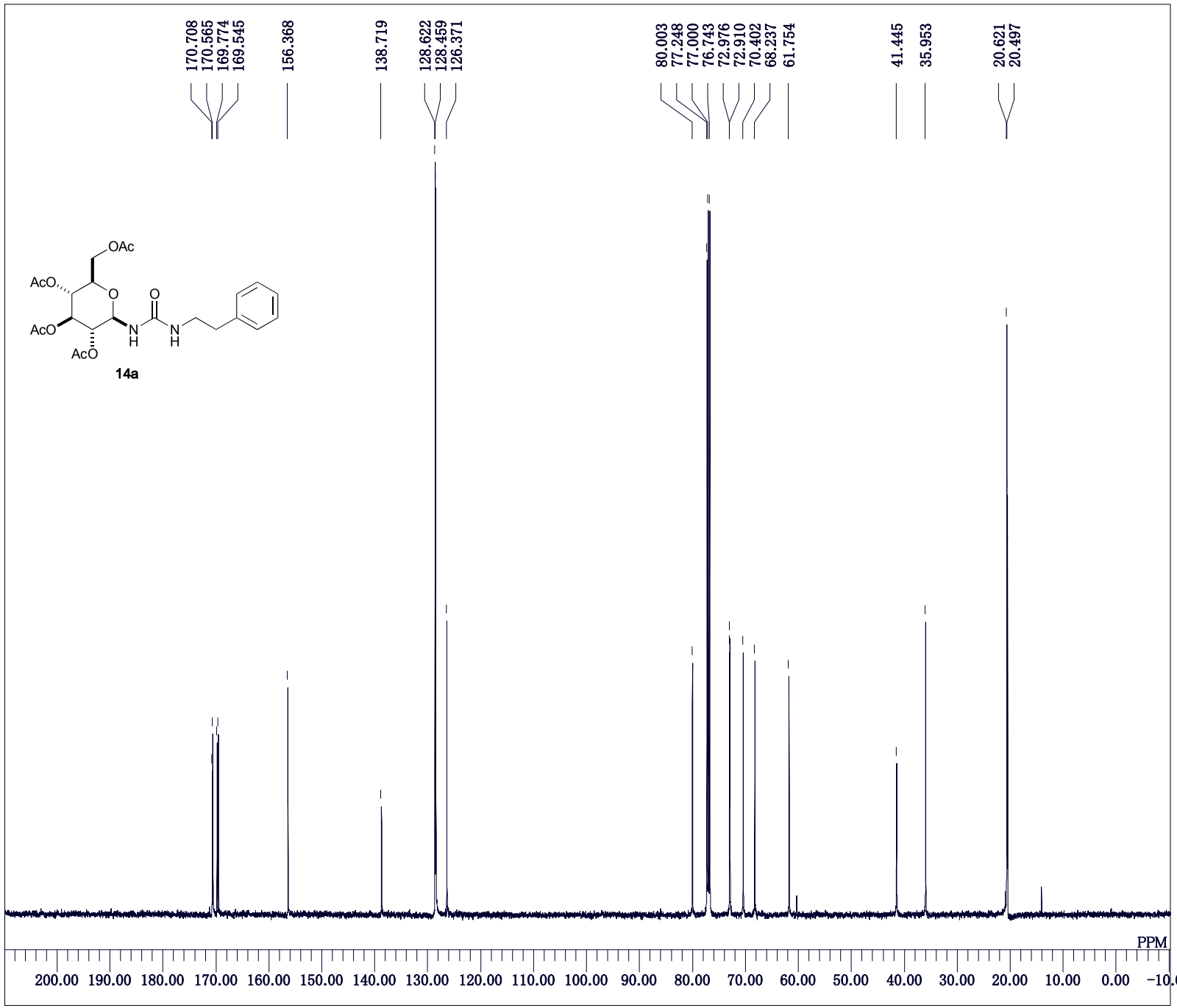


DFILE	sou3140data_Carbon-2-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-09 10:18:24
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50

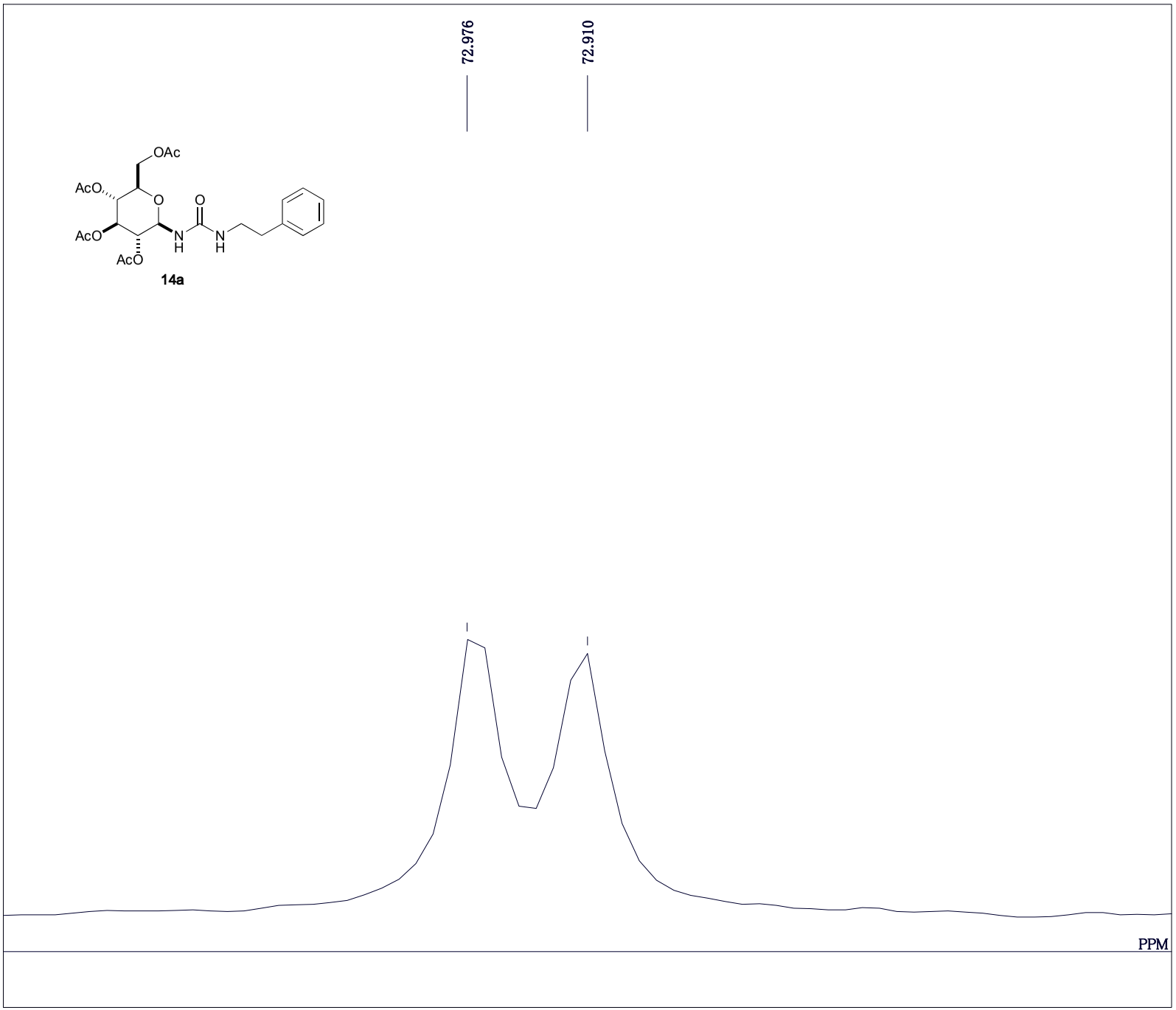


sou4107data_Carbon-1-1.jdf
single pulse decoupled gated NOE
2011-11-19 09:59:16
13C
carbon.jxp
125.77 MHz
7.87 KHz
4.21 Hz
32780
39308.18 Hz
512
0.8336 sec
2.0000 sec
2.72 usec
1H
19.1 c
CDCL3
77.00 ppm
1.00 Hz
50

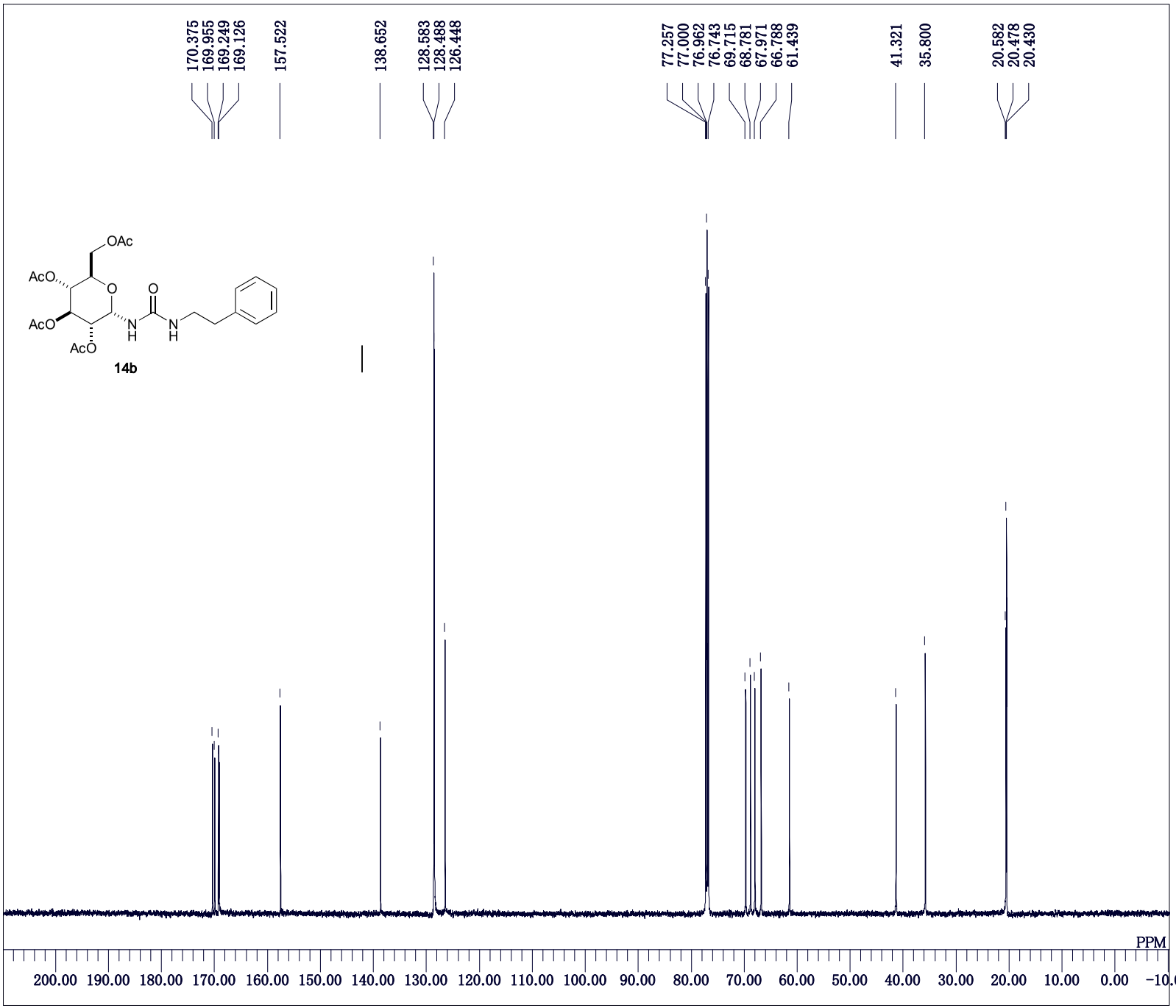
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



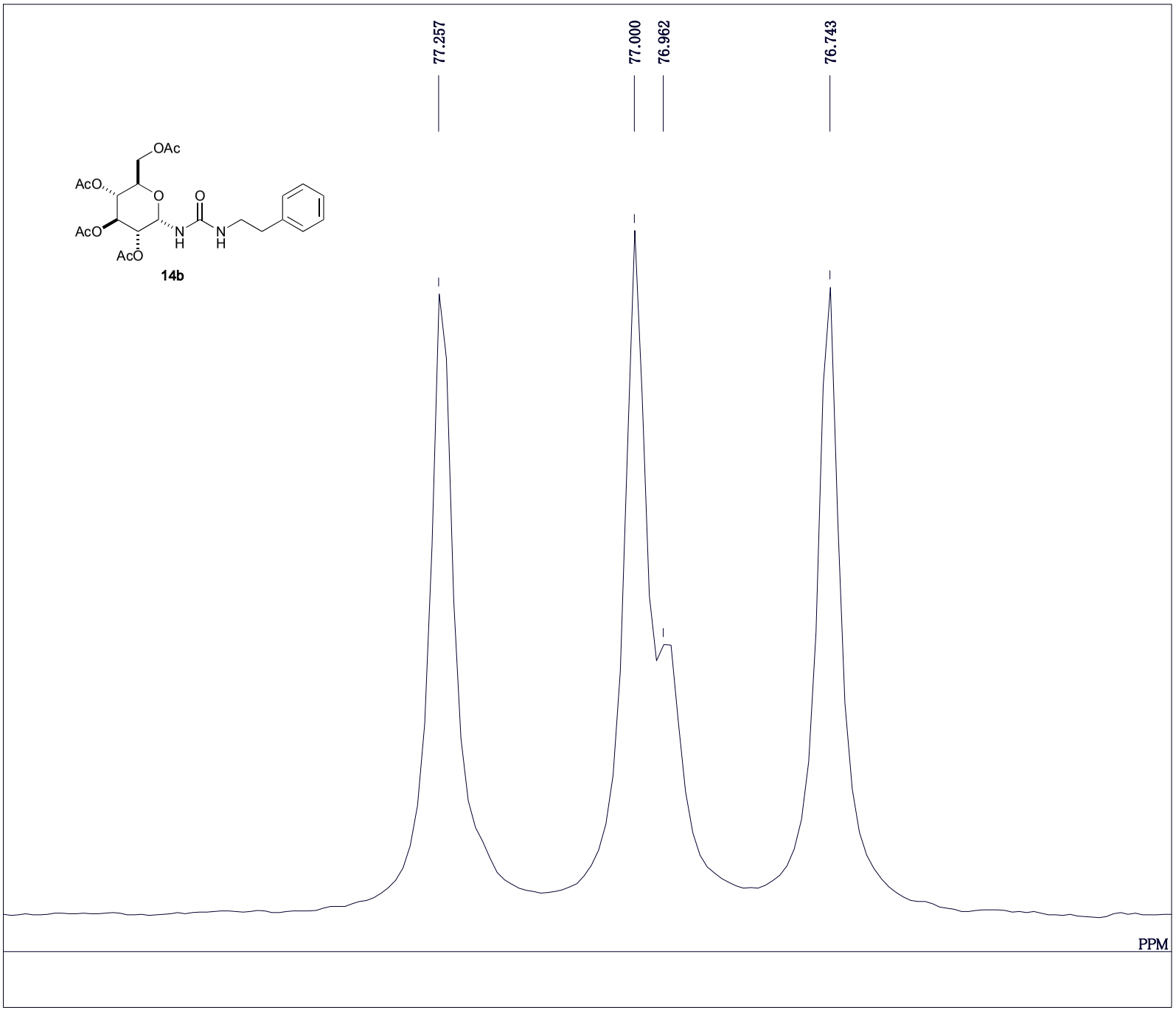
DFILE	sou3138data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-09 17:50:10
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



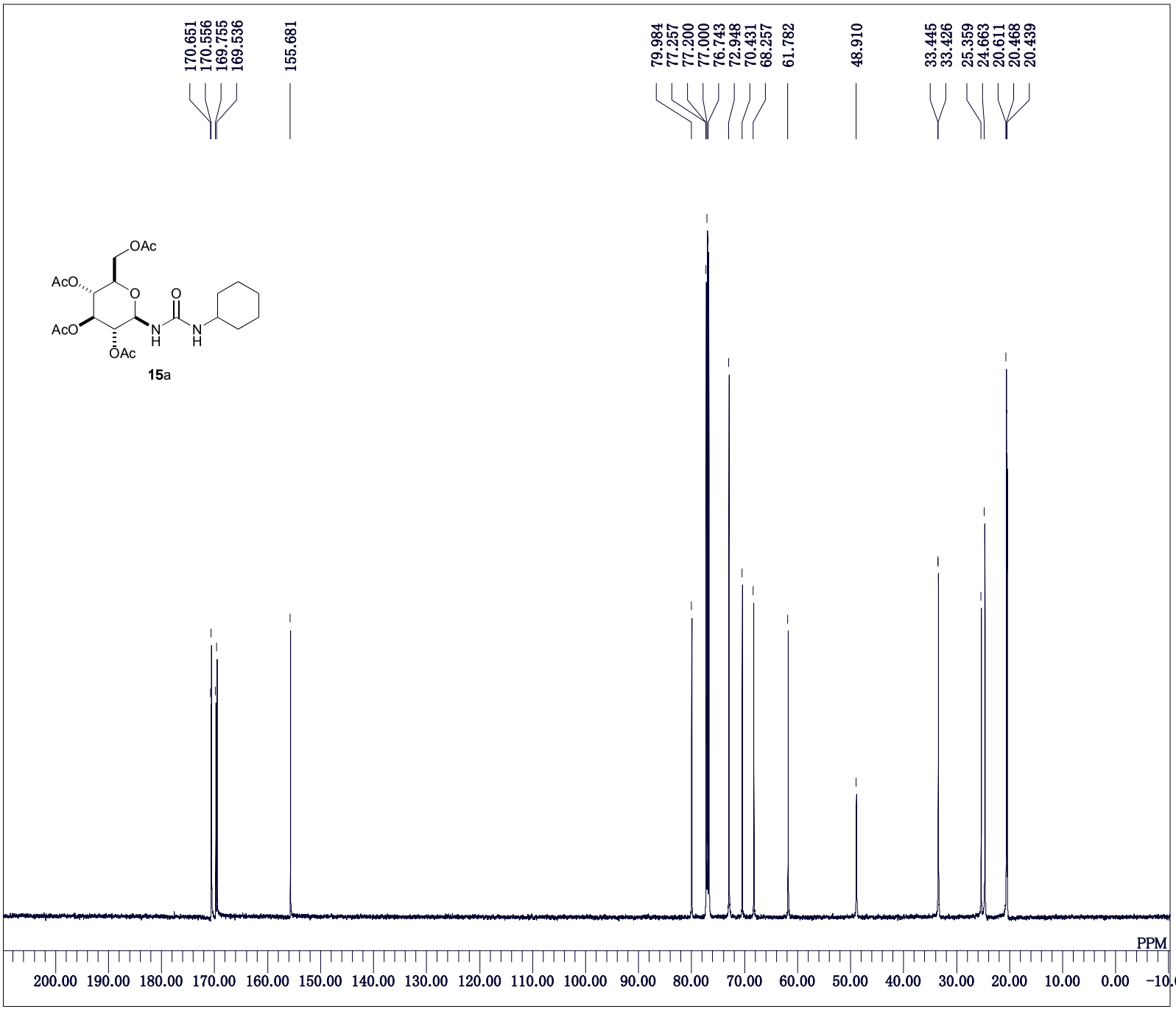
DFILE	sou3138data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-09 17:50:10
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



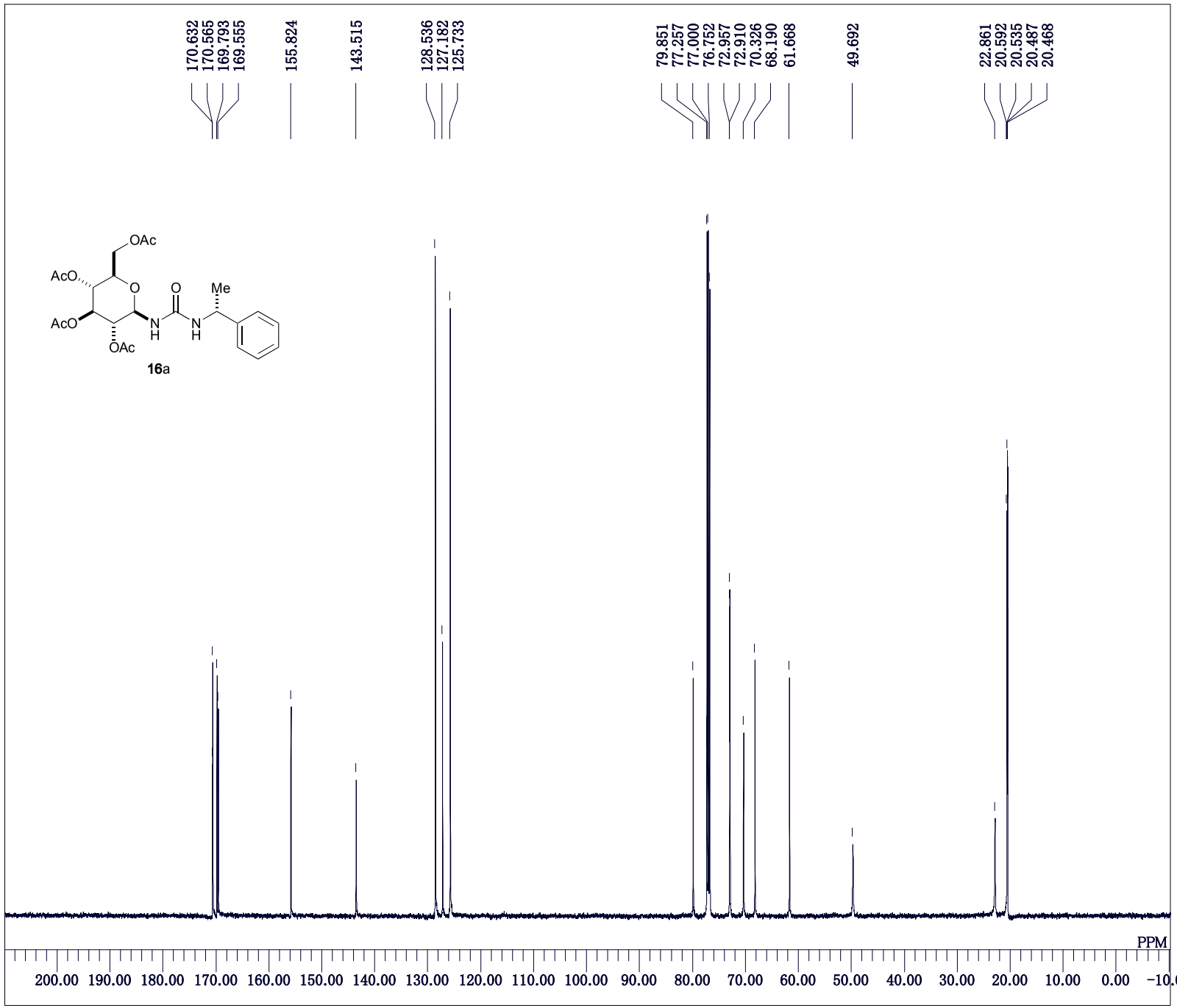
DFILE	sou4108data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-19 11:11:51
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



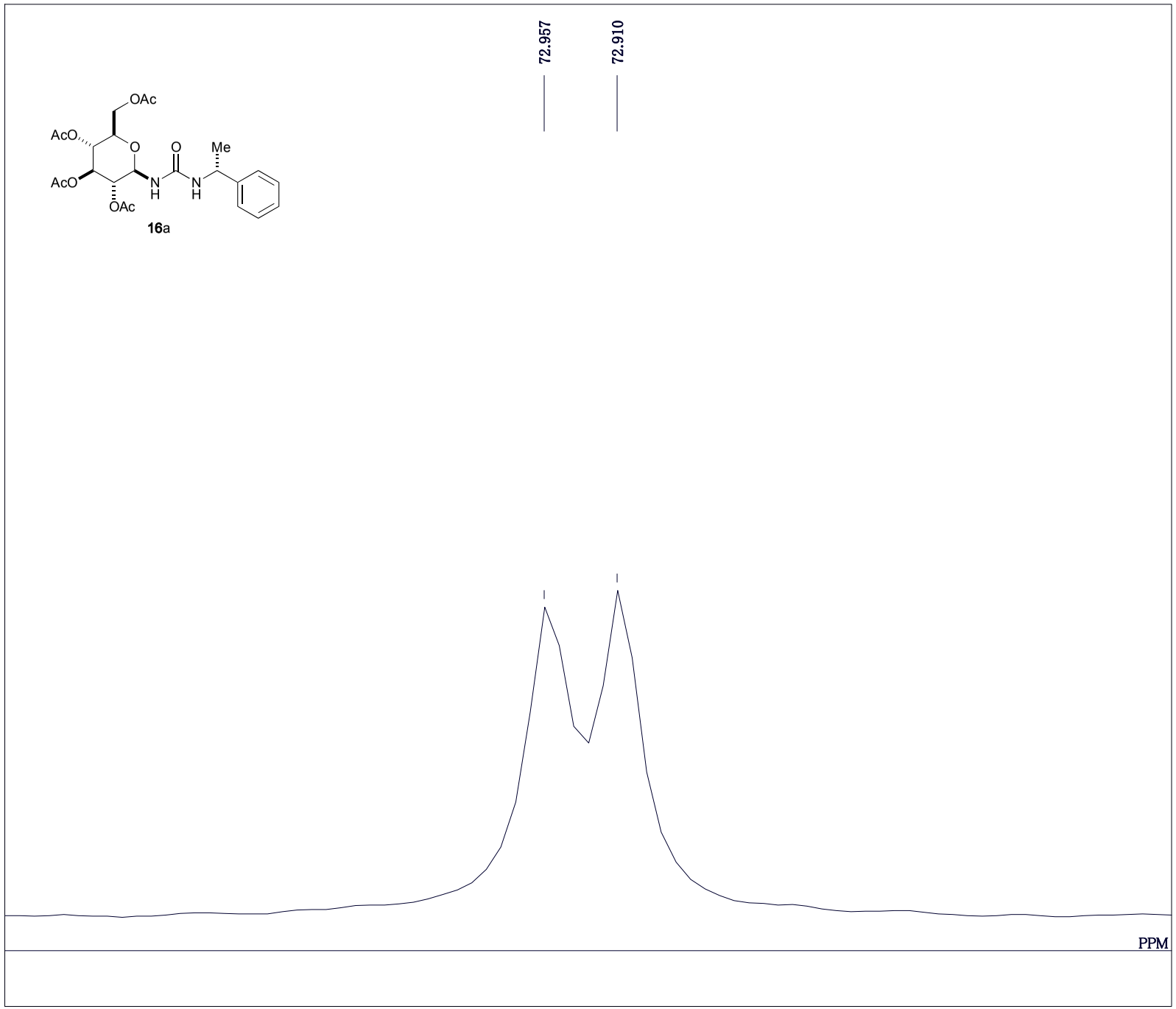
DFILE	sou4108data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-19 11:11:51
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



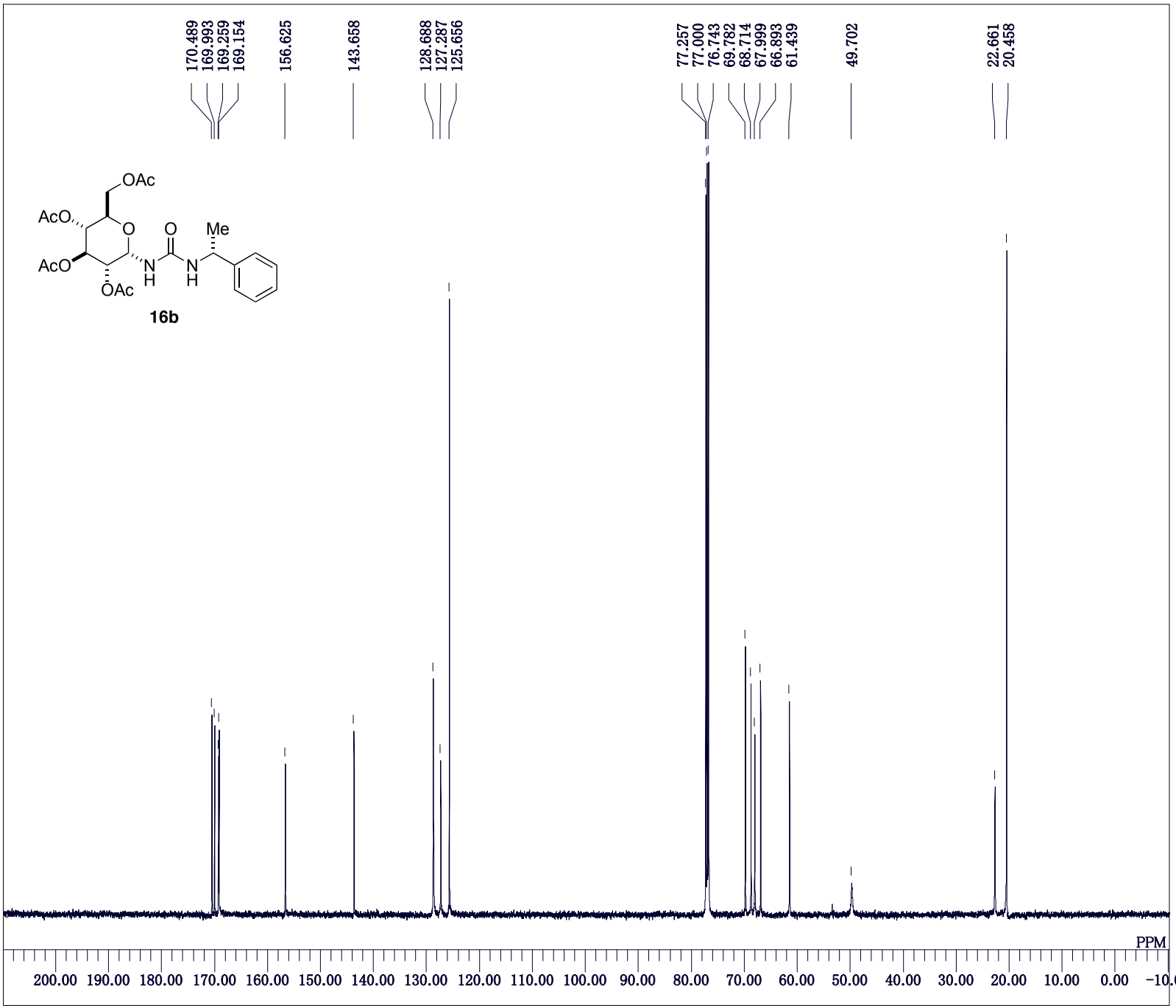
DFILE	sou3142data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-27 14:24:25
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



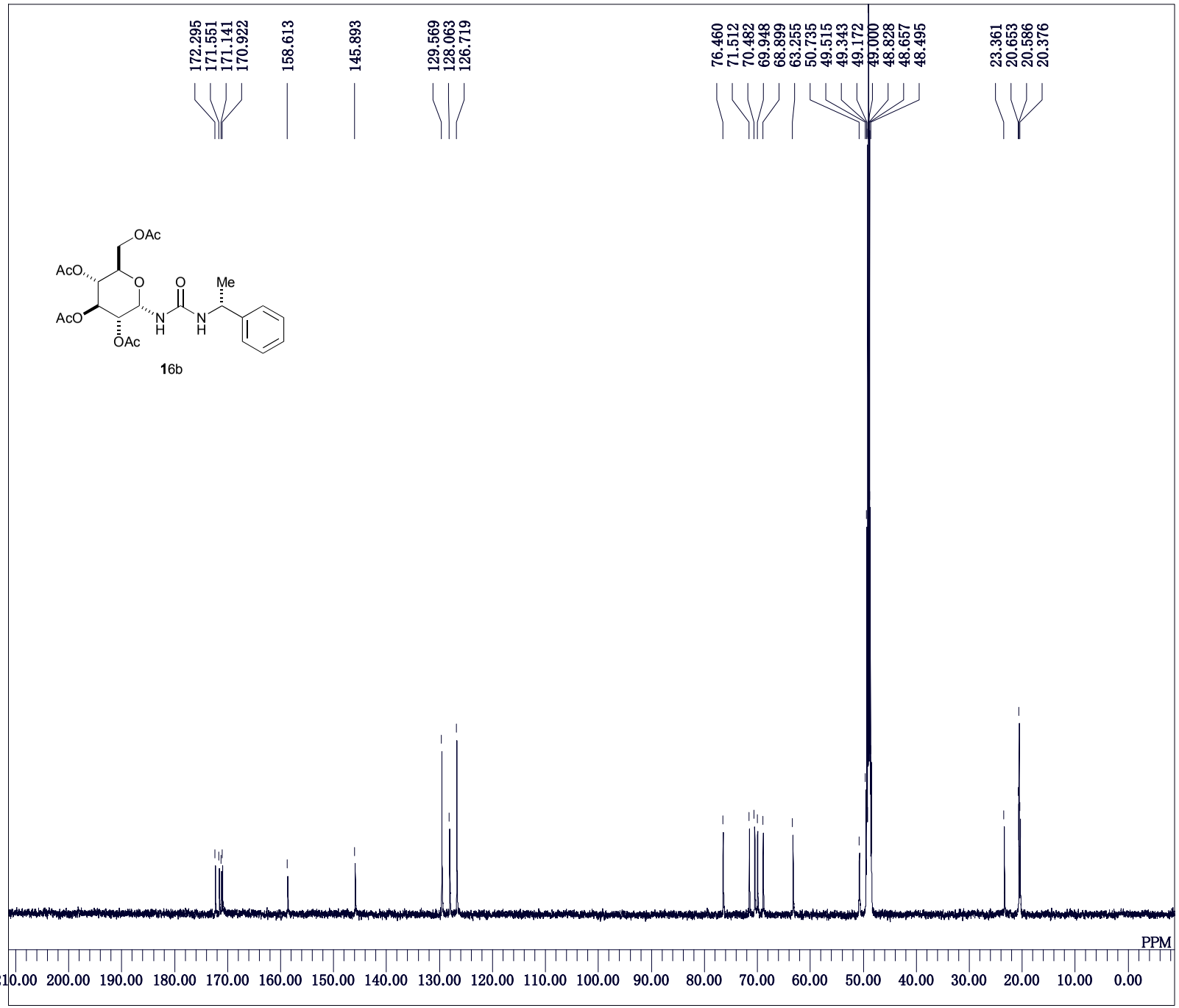
DFILE	sou3058data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-27 10:39:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



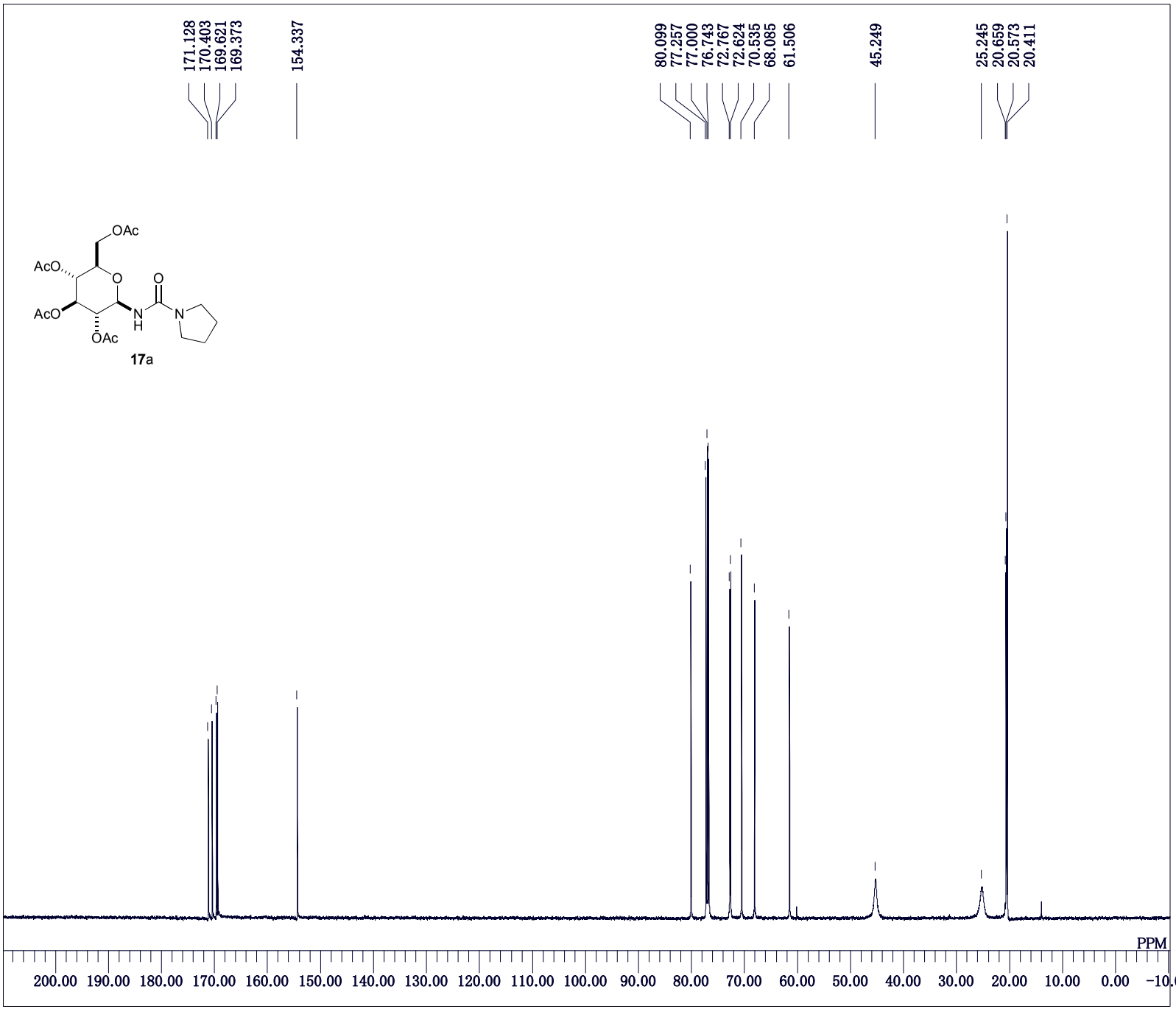
DFILE	sou3058data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-27 10:39:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



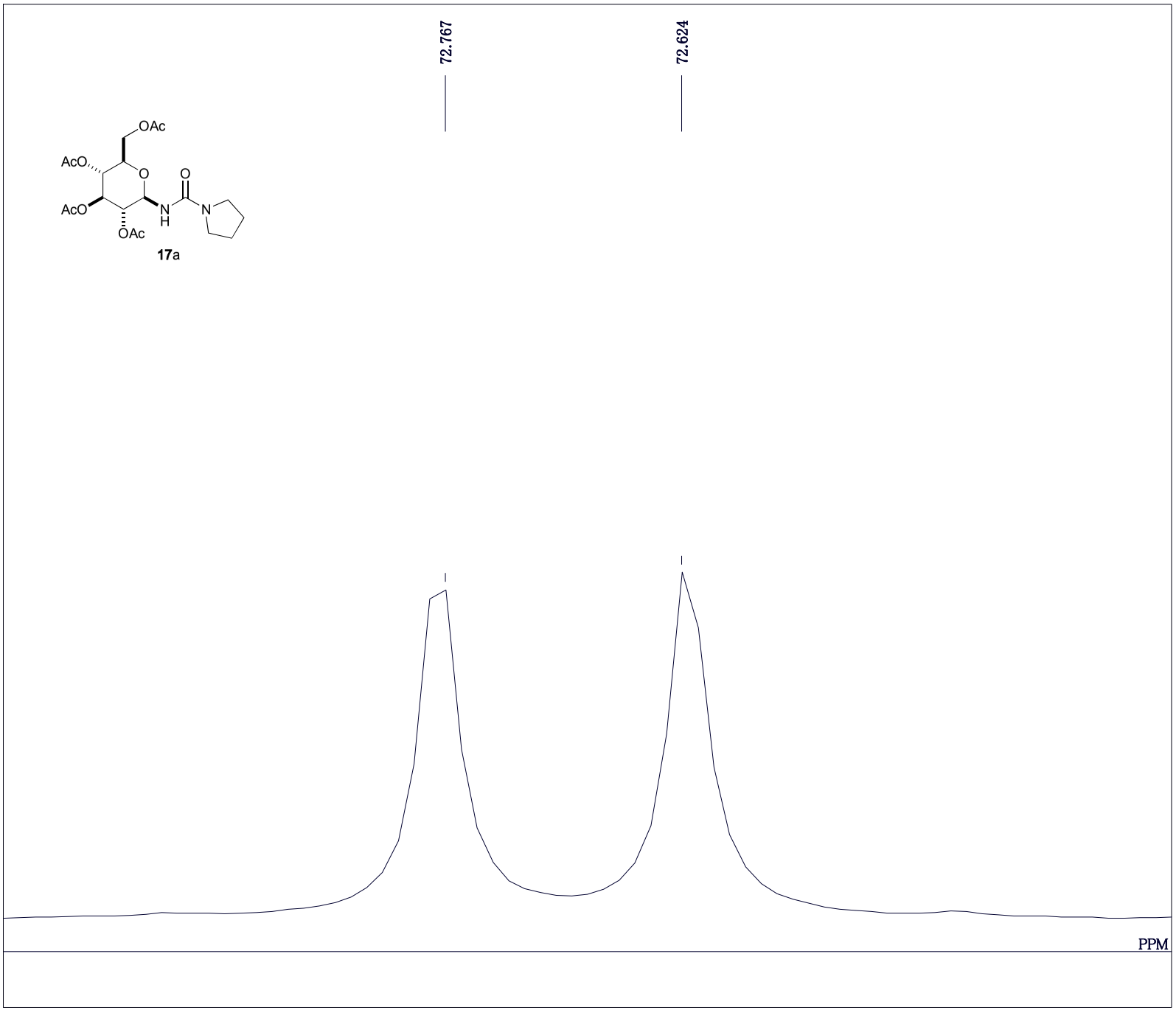
DFILE	sou4115data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-19 10:38:07
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



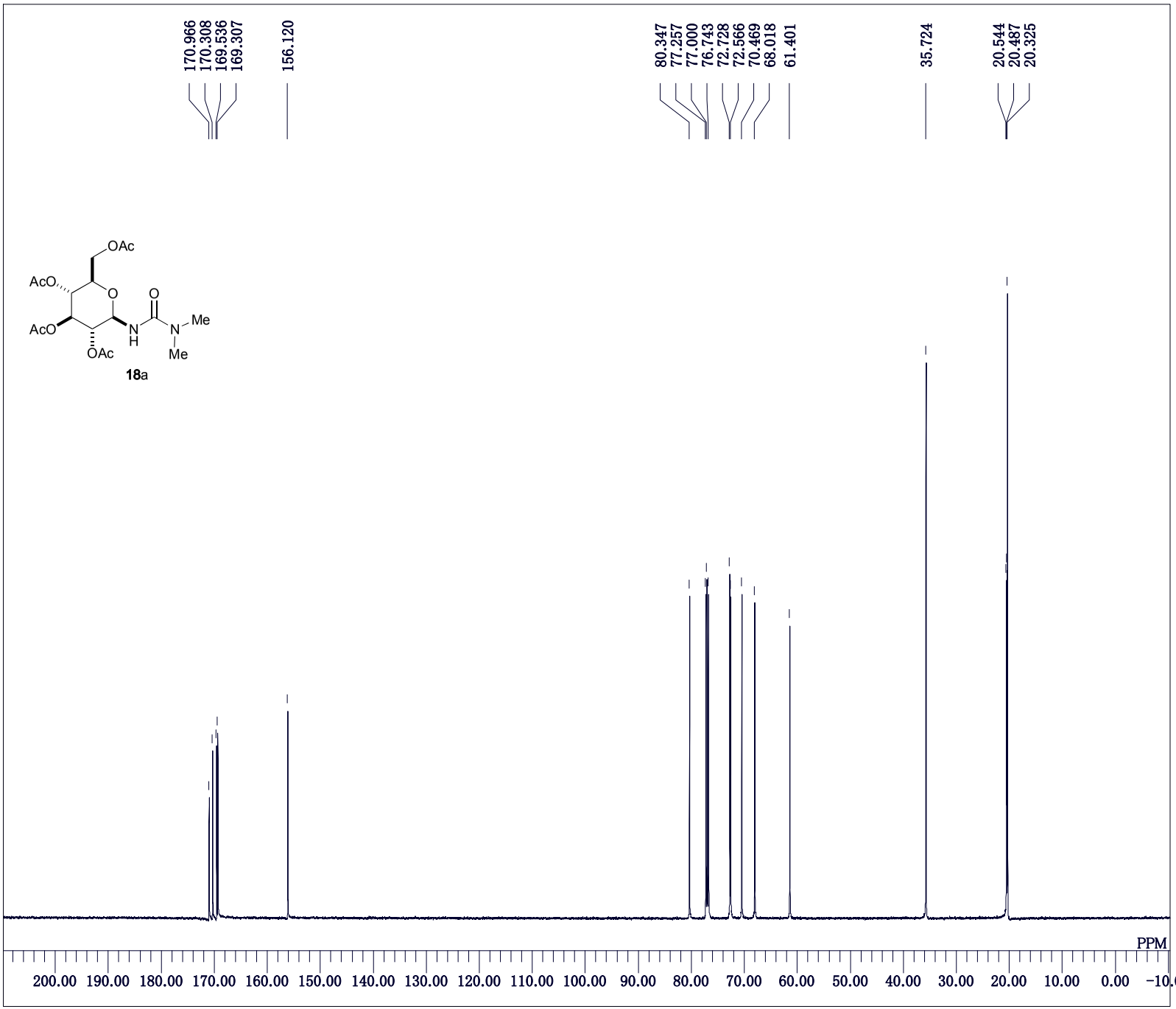
DFILE	sou4115CD3ODdata_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-12-05 14:44:26
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	1.00 Hz
RGAIN	50



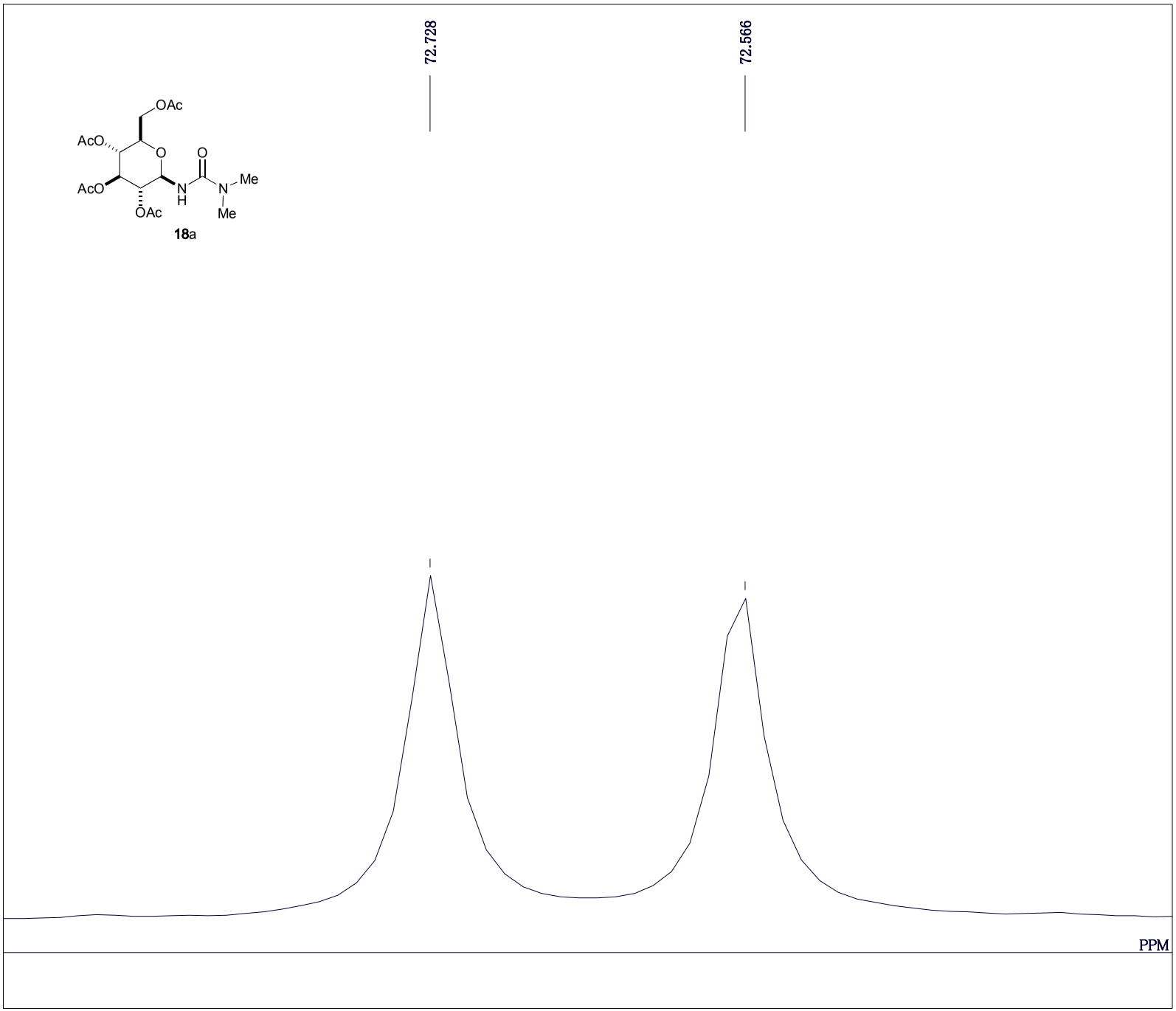
DFILE	sou3074data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 11:15:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



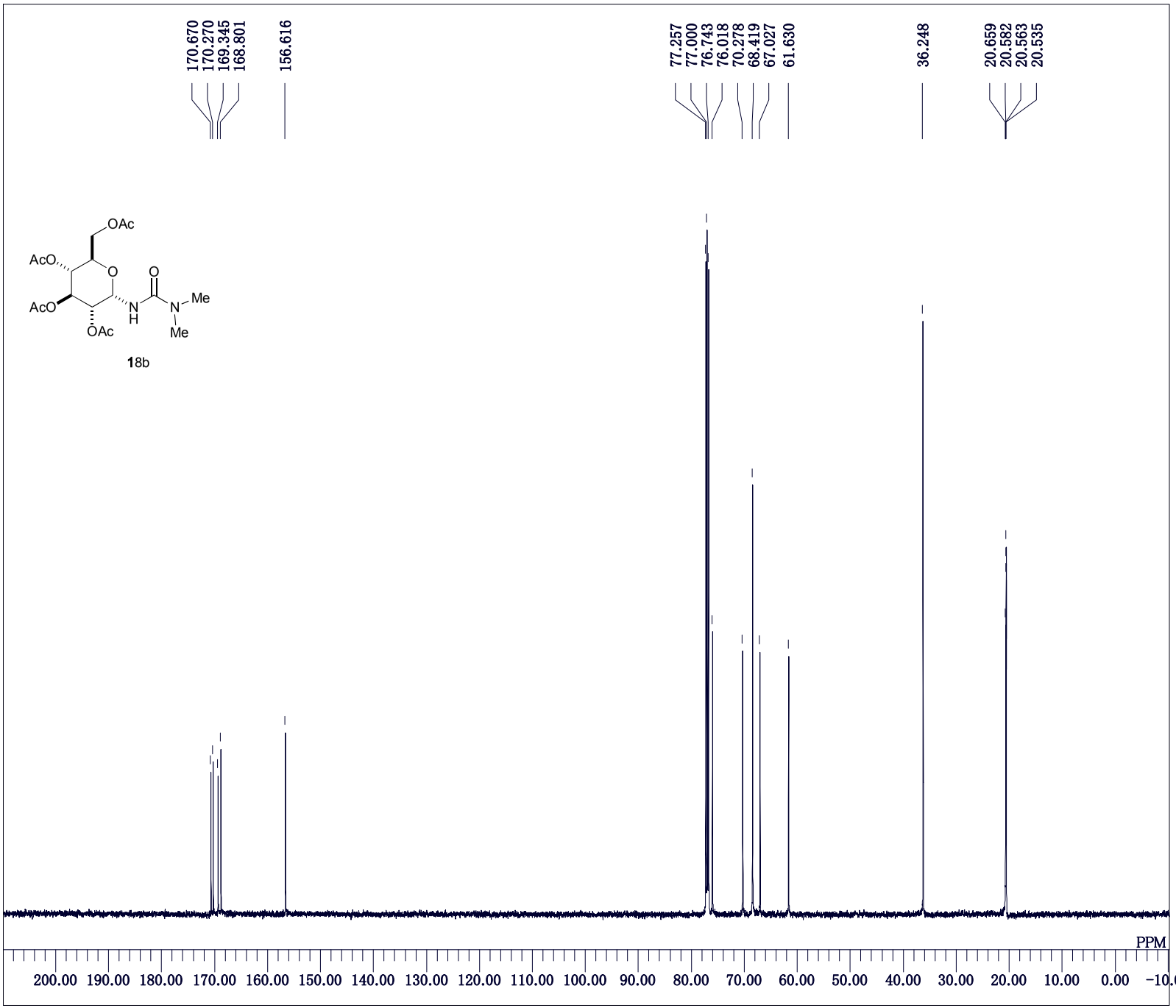
DFILE	sou3074data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 11:15:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



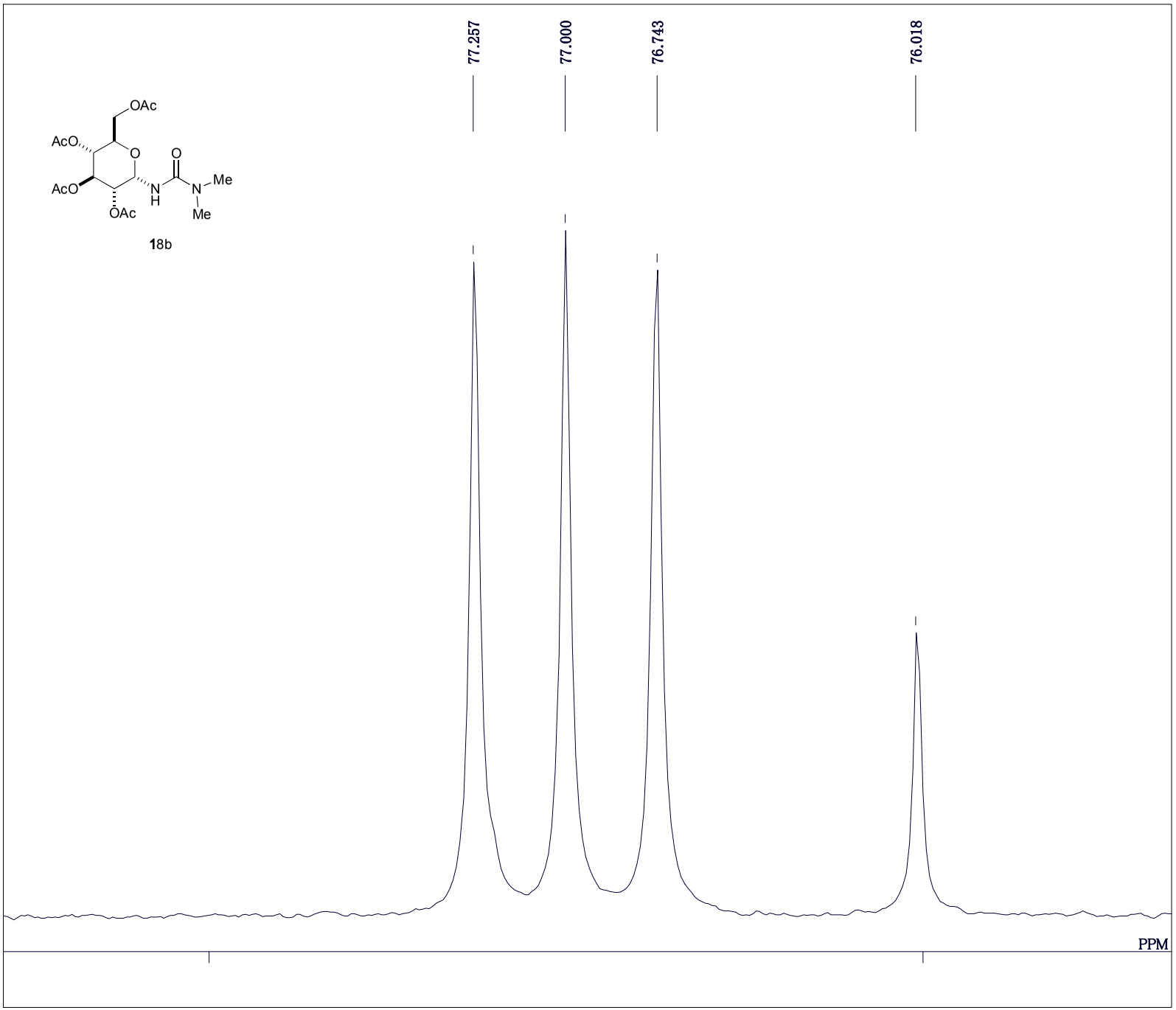
DFILE	sou3112data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 21:52:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



DFILE	sou3112data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-08-29 21:52:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



DFILE	sou4116data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-30 21:10:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



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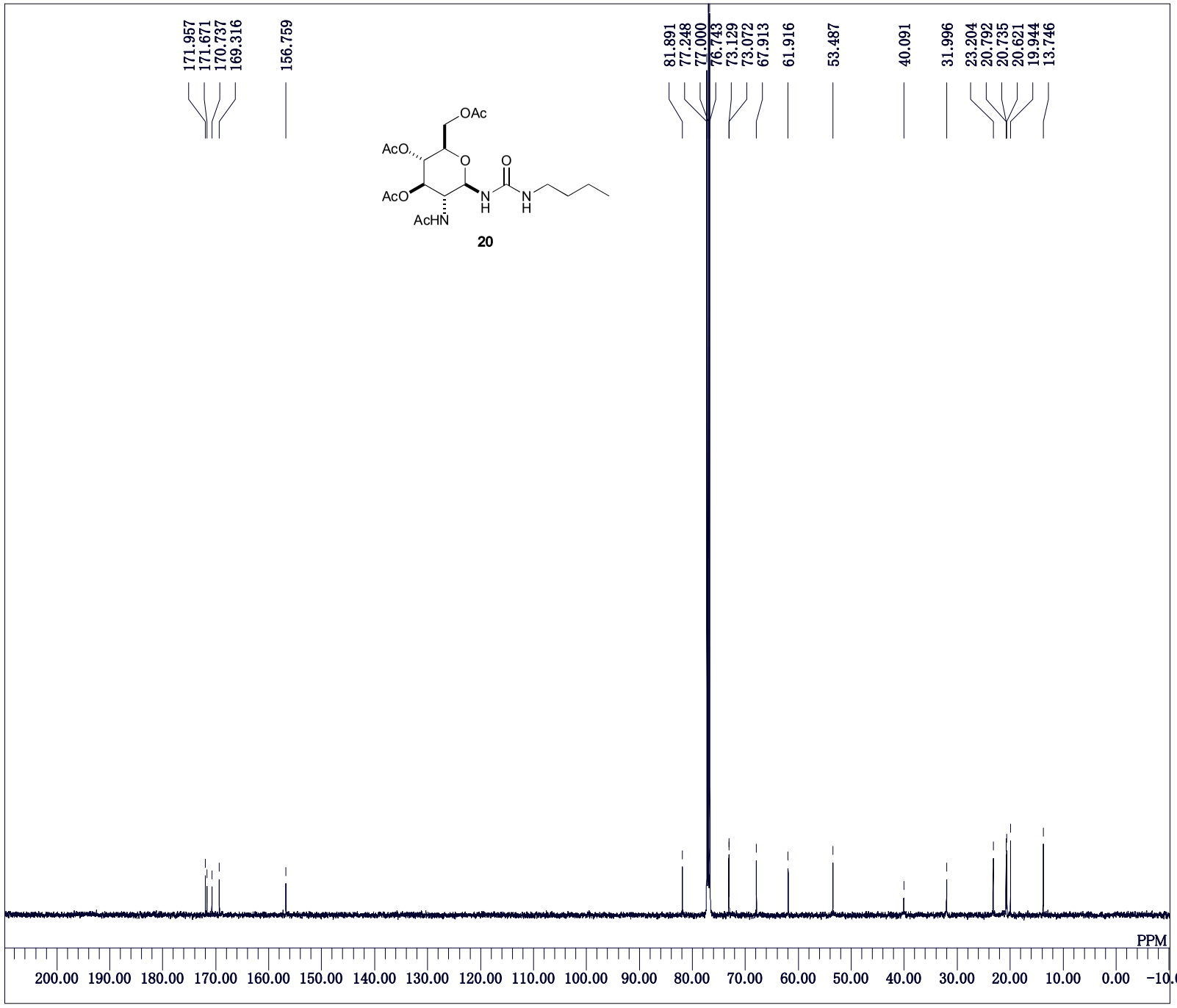
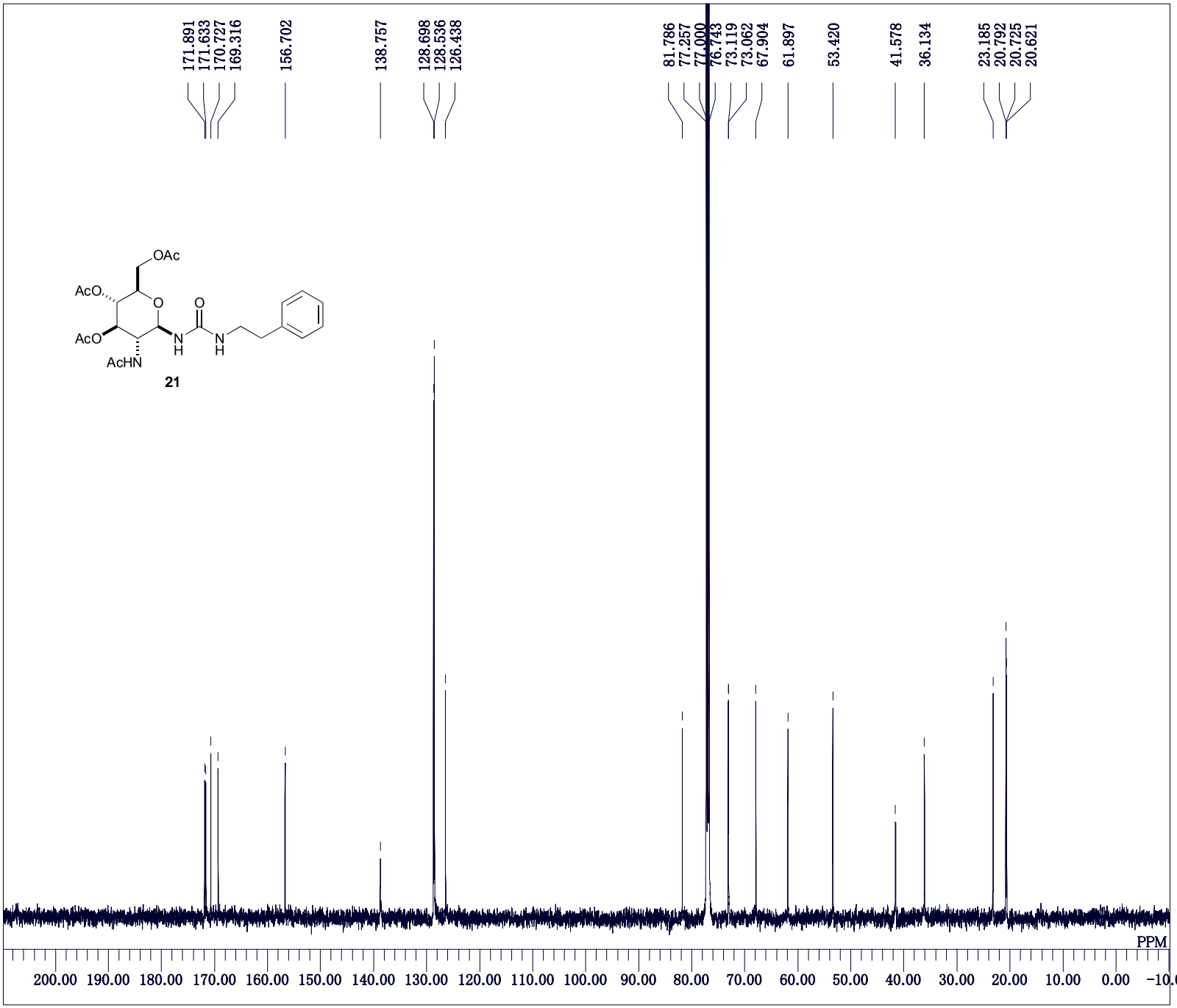
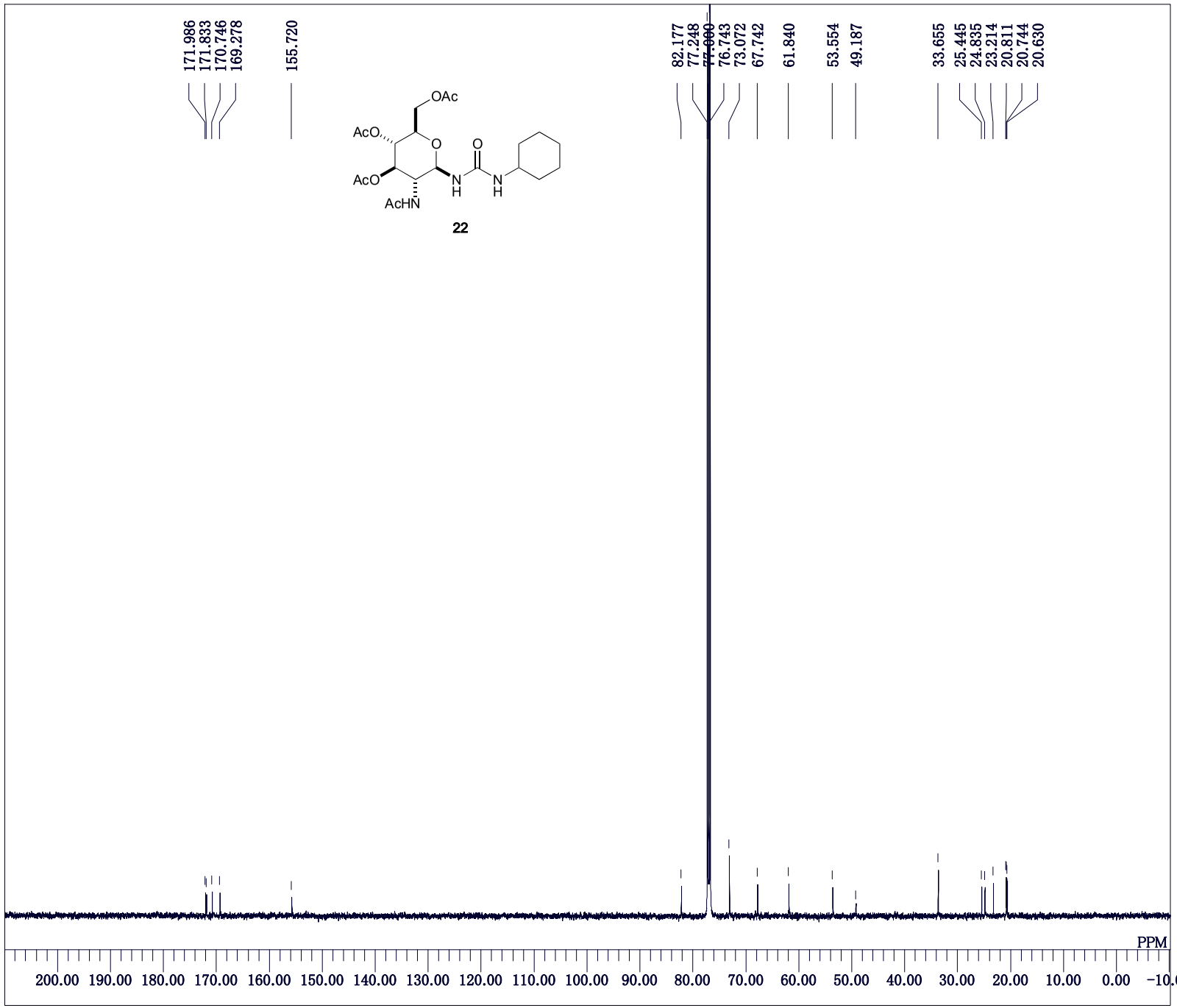
DFILE	sou4116data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-30 21:10:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



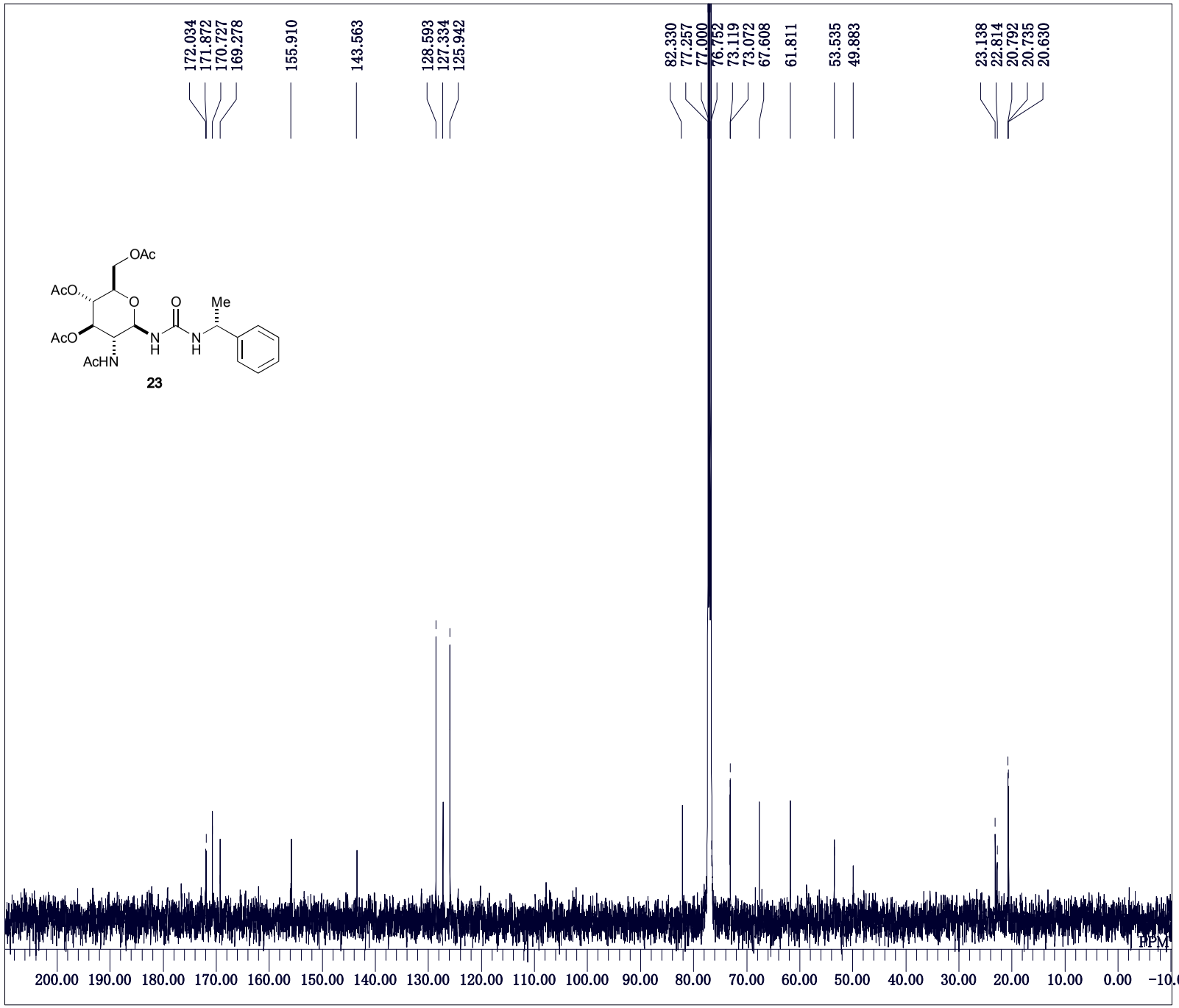
DFILE	TON1146_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-02-05 15:31:31
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



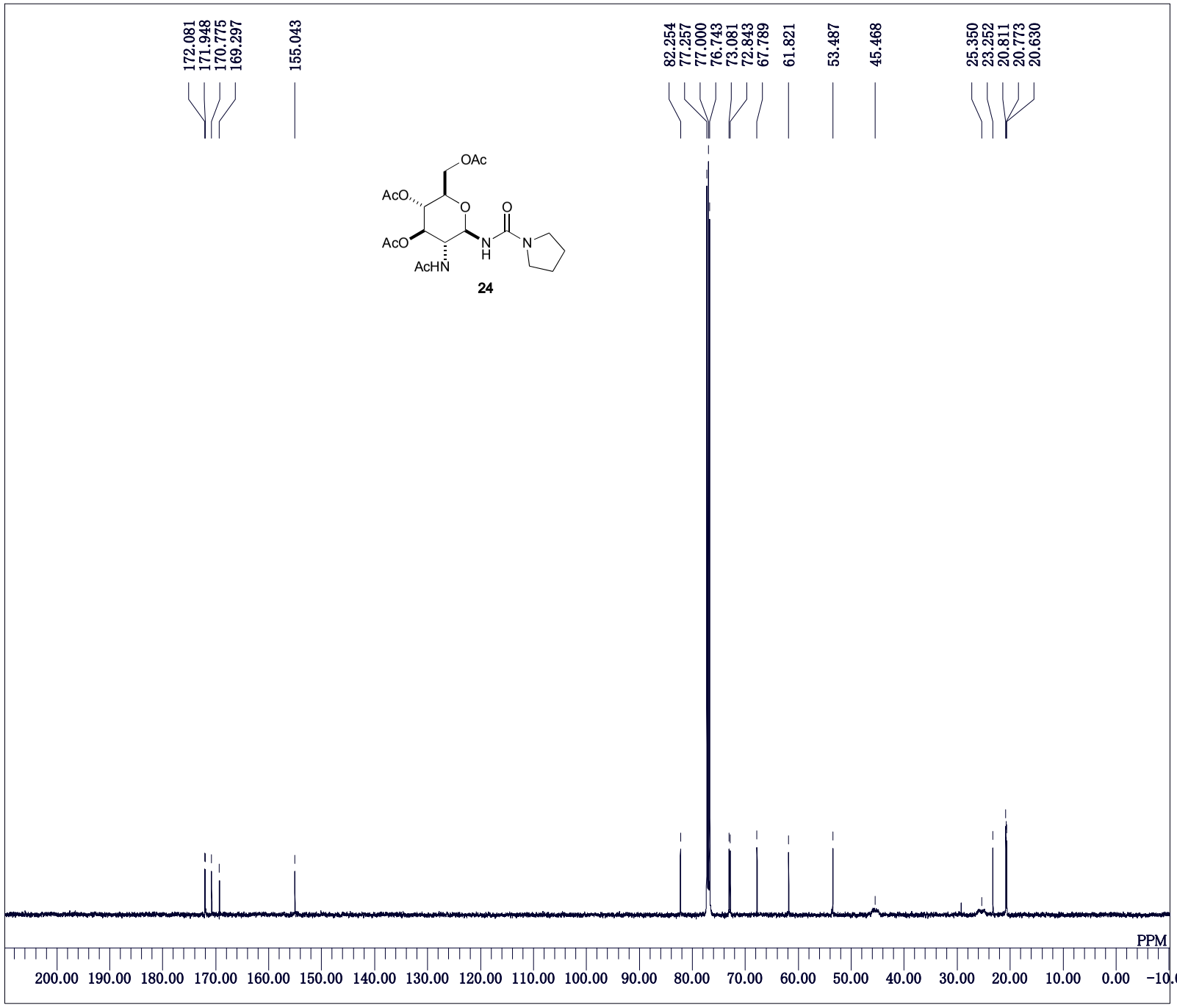
DFILE	TON1138_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-02-07 21:20:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	16.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



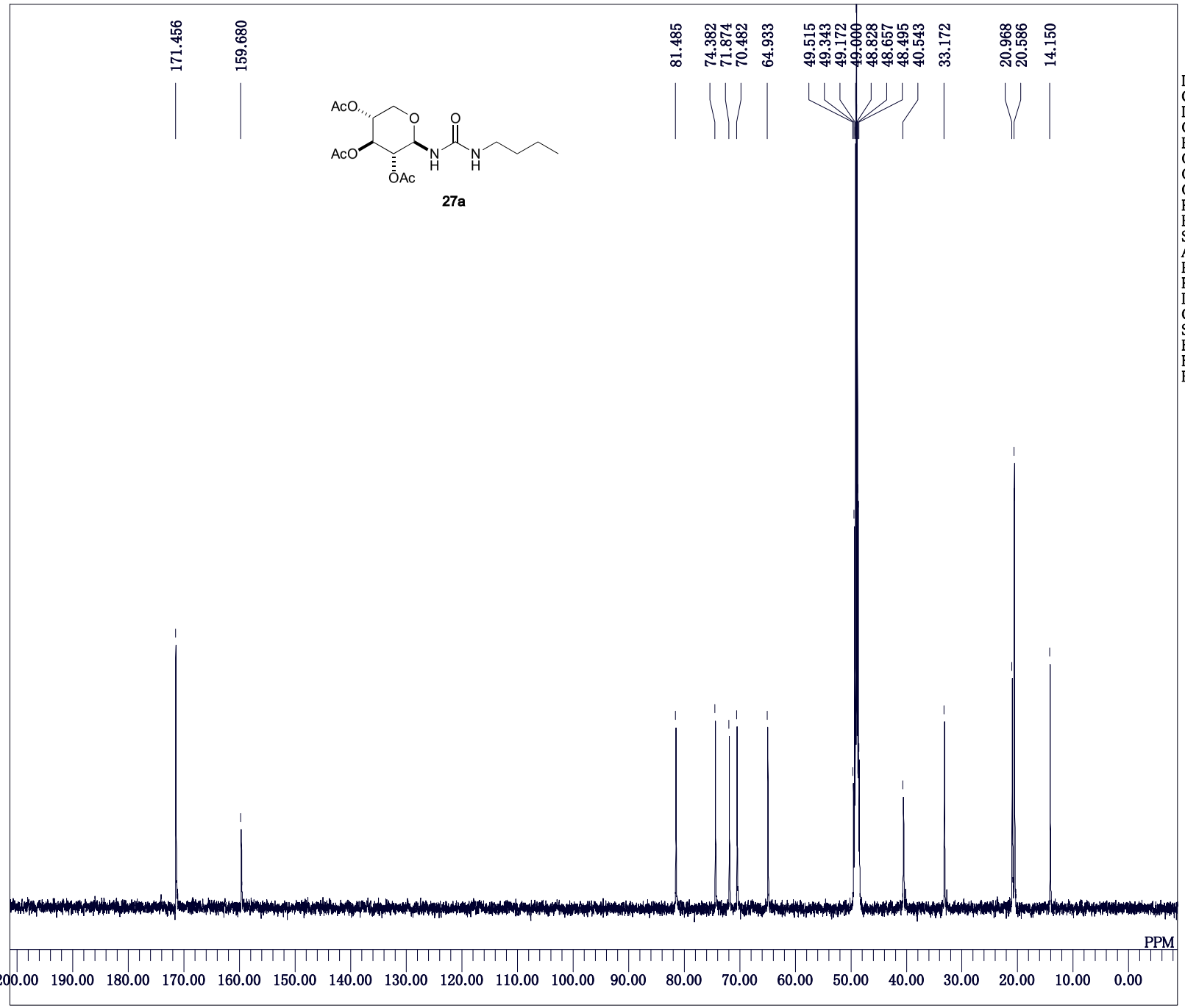
DFILE	sou3094data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-22 19:34:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



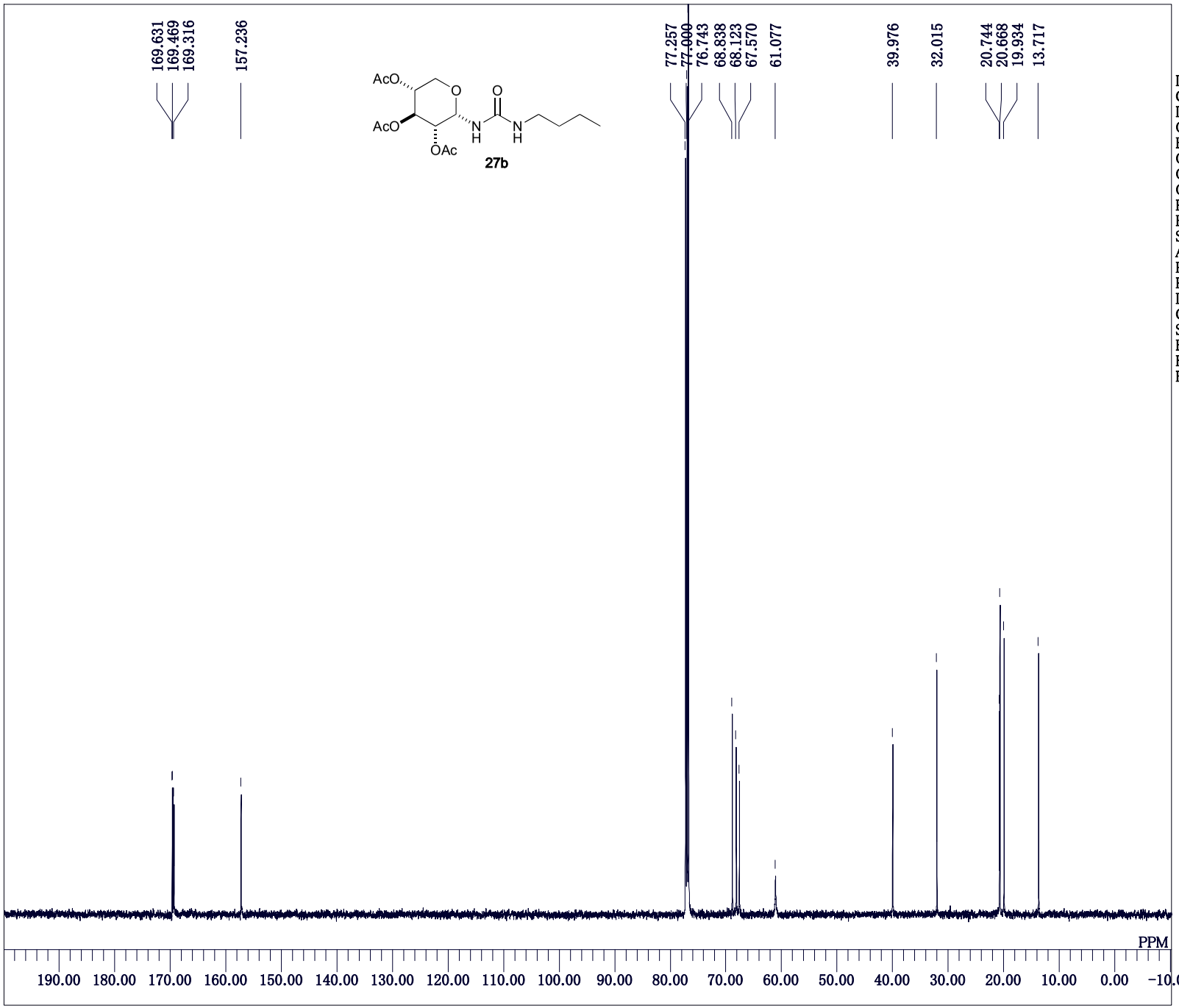
DFILE	TON1142_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-02-07 22:23:05
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



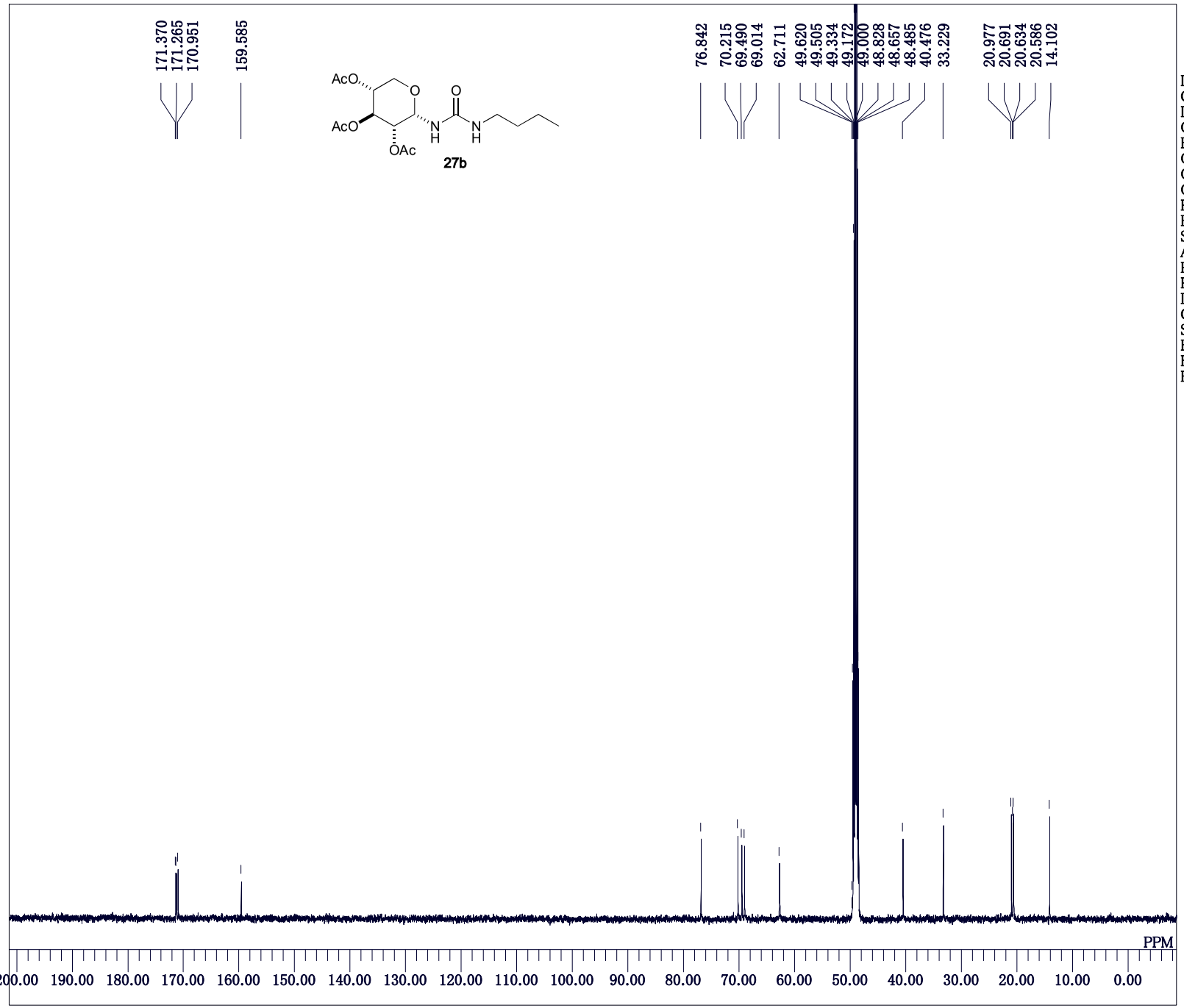
DFILE	TON1_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-02-12 09:13:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



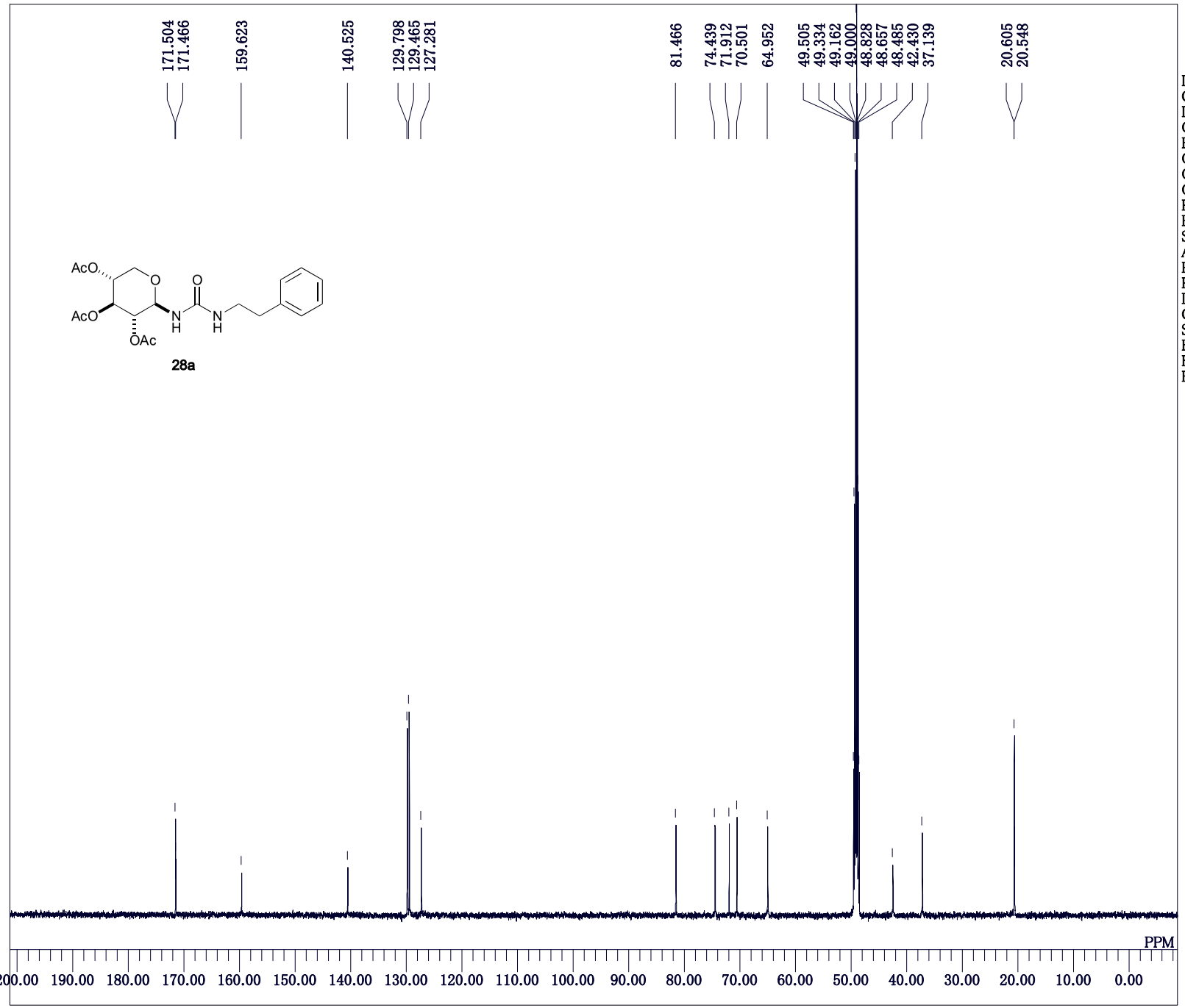
DFILE	SAE1056_Carbon-2-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-02-20 22:27:39
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	256
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	16.9 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	1.00 Hz
RGAIN	50



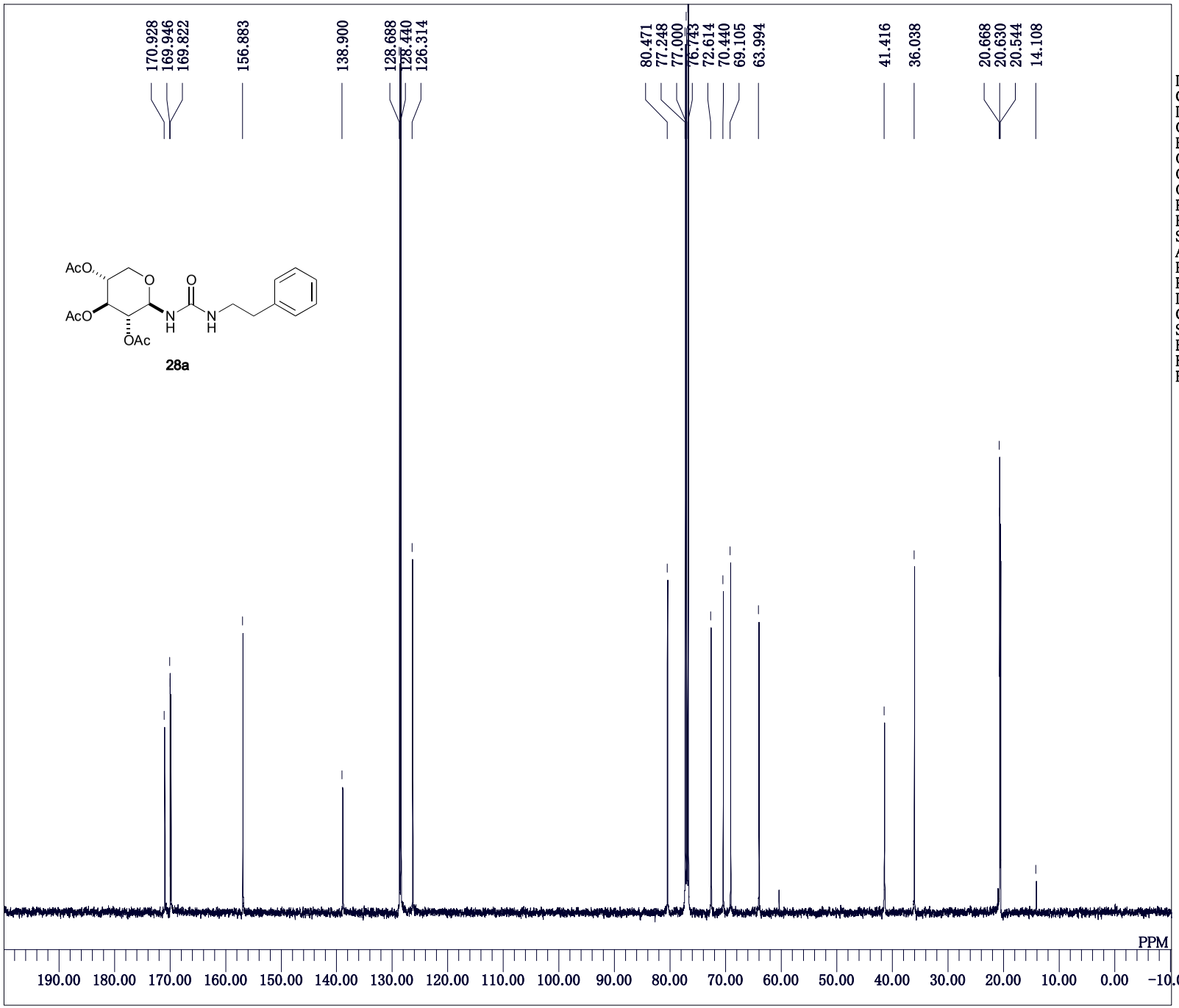
DFILE	SAE2057_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-07-12 15:47:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



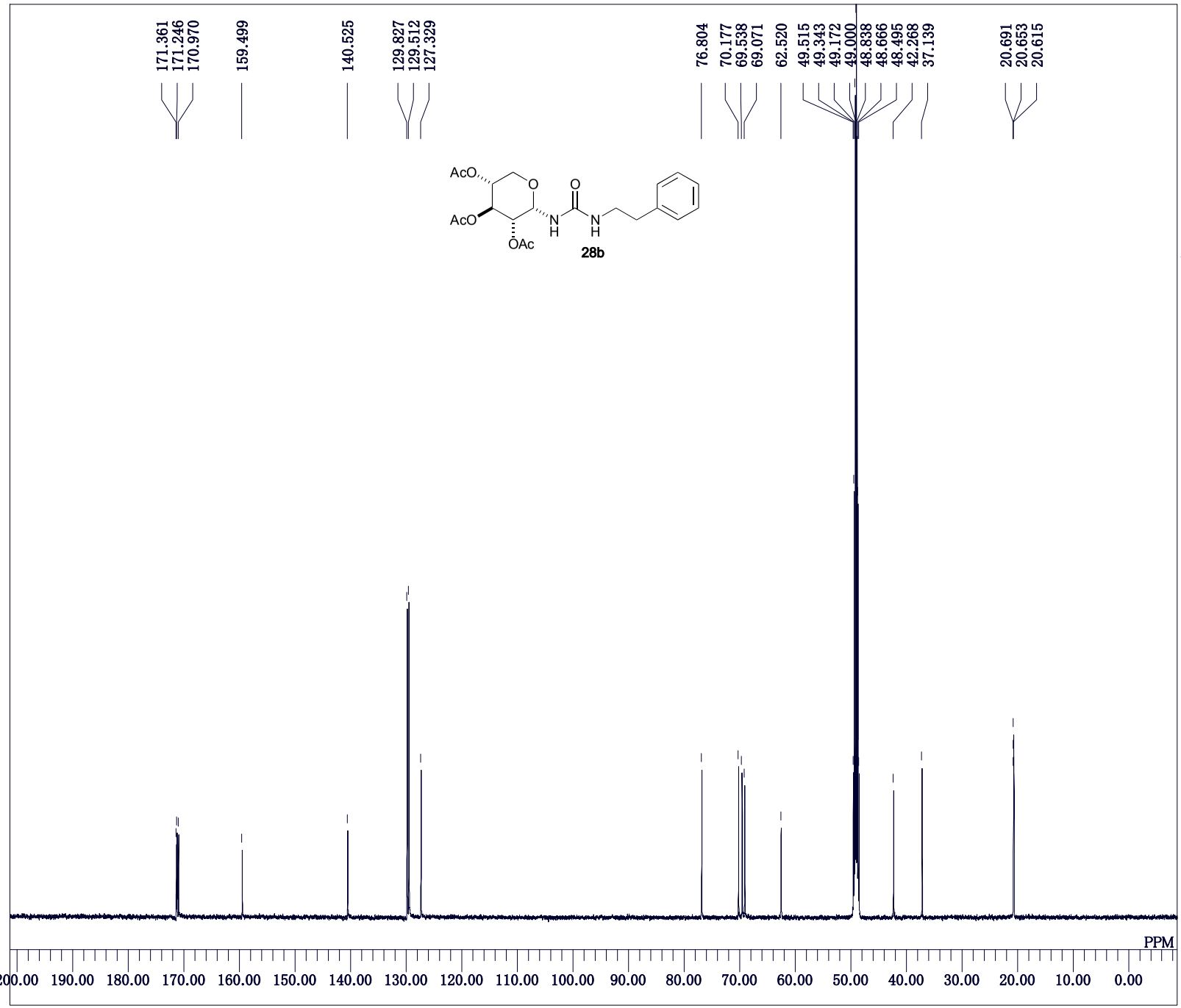
DFILE	SAE2057_Carbon-2-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-07-13 14:52:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	1.00 Hz
RGAIN	50



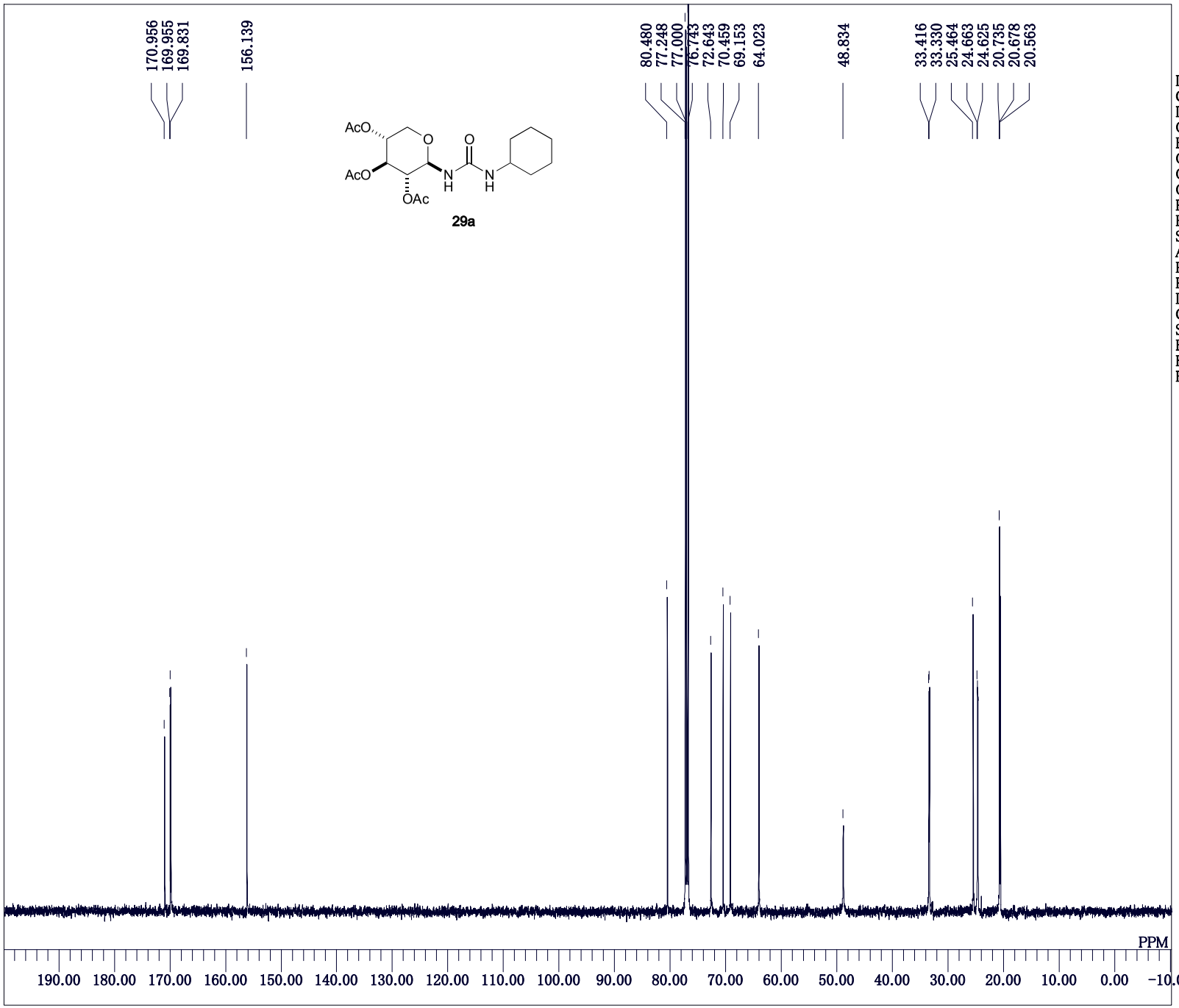
DFILE	SAE1191_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-07-05 09:29:32
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	1.00 Hz
RGAIN	50



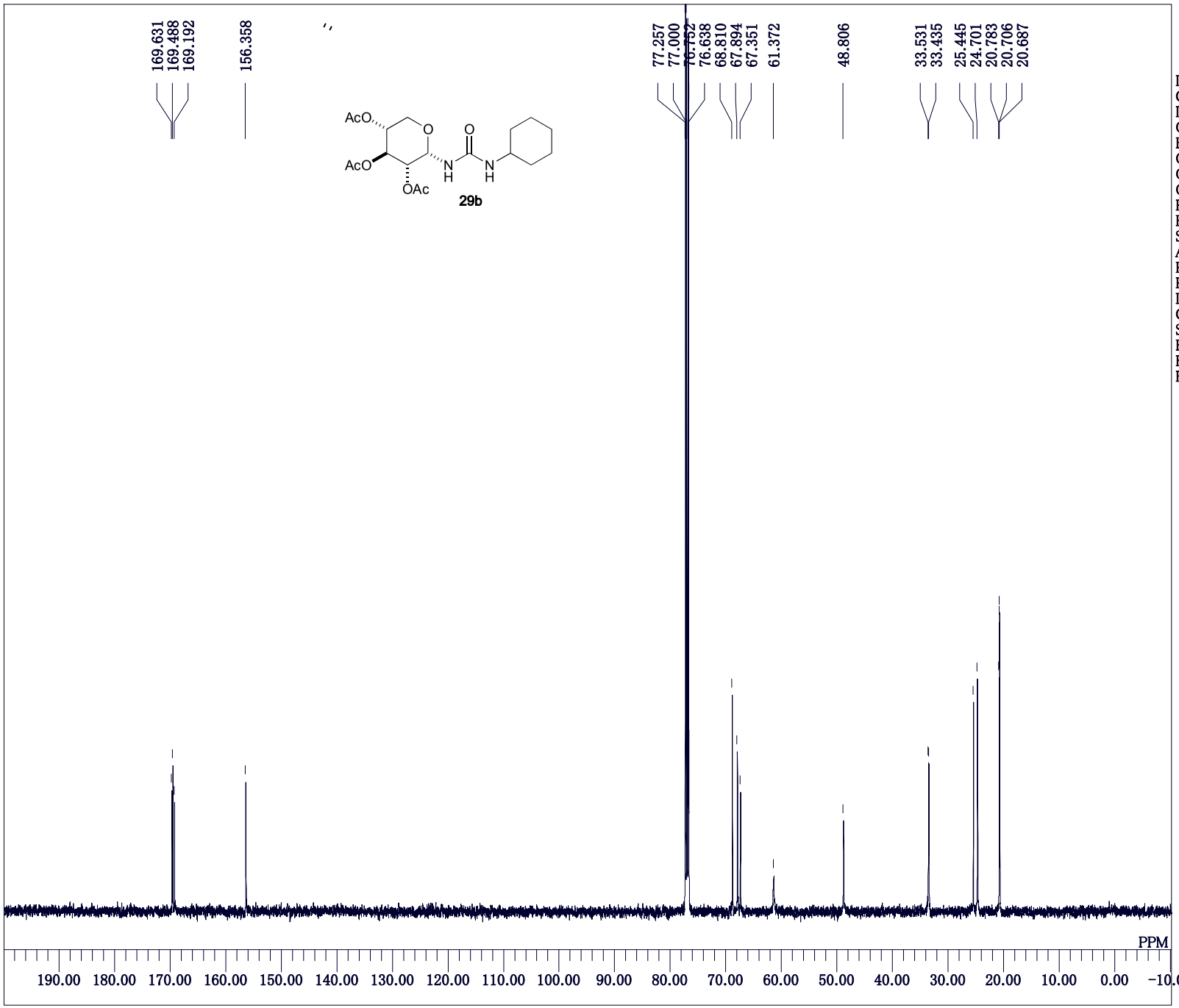
DFILE	SAE1191_Carbon-2-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-08-04 15:17:22
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50

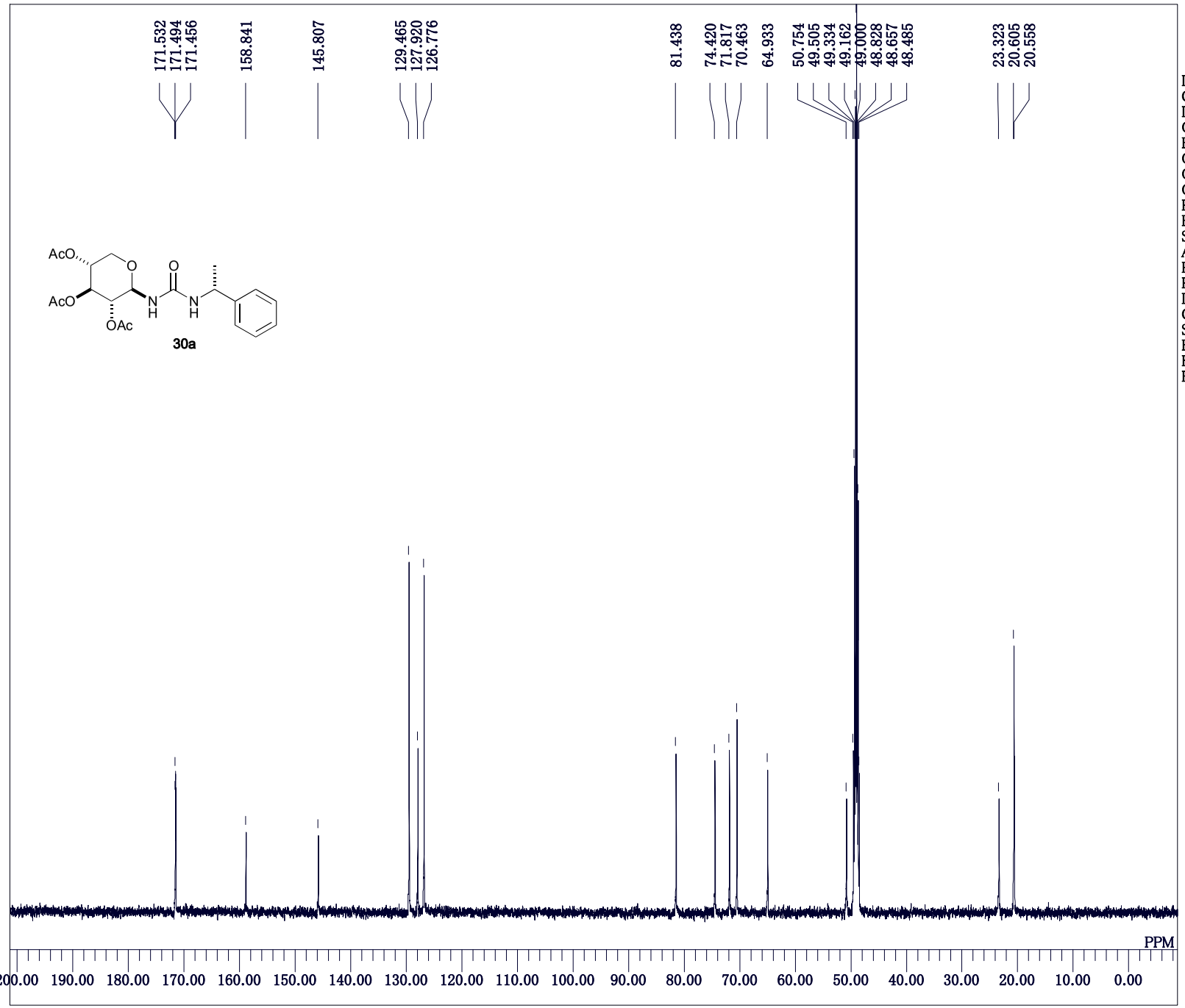


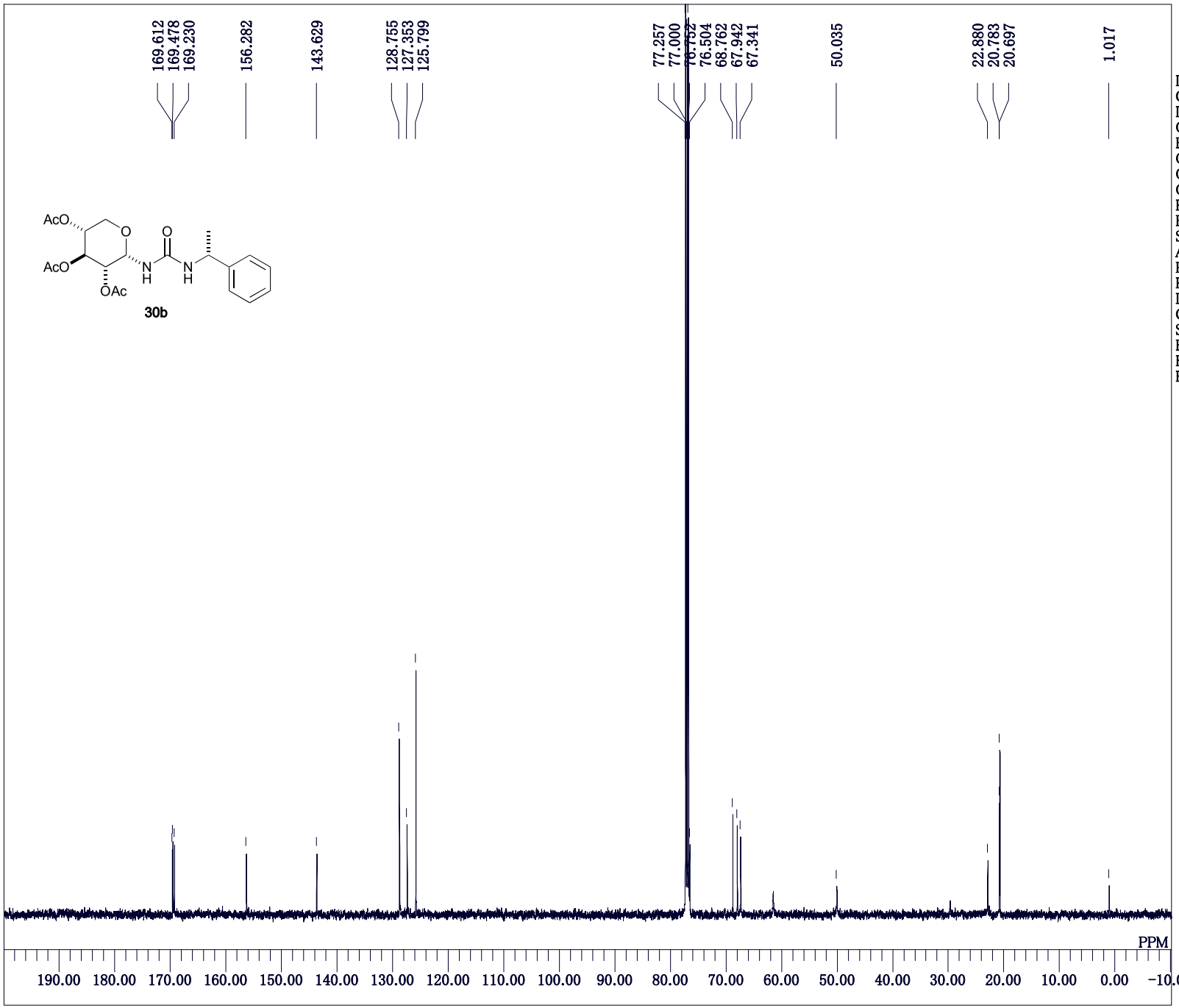
DFILE	SAE2055-13C_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-06-27 18:16:31
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	1.00 Hz
RGAIN	50



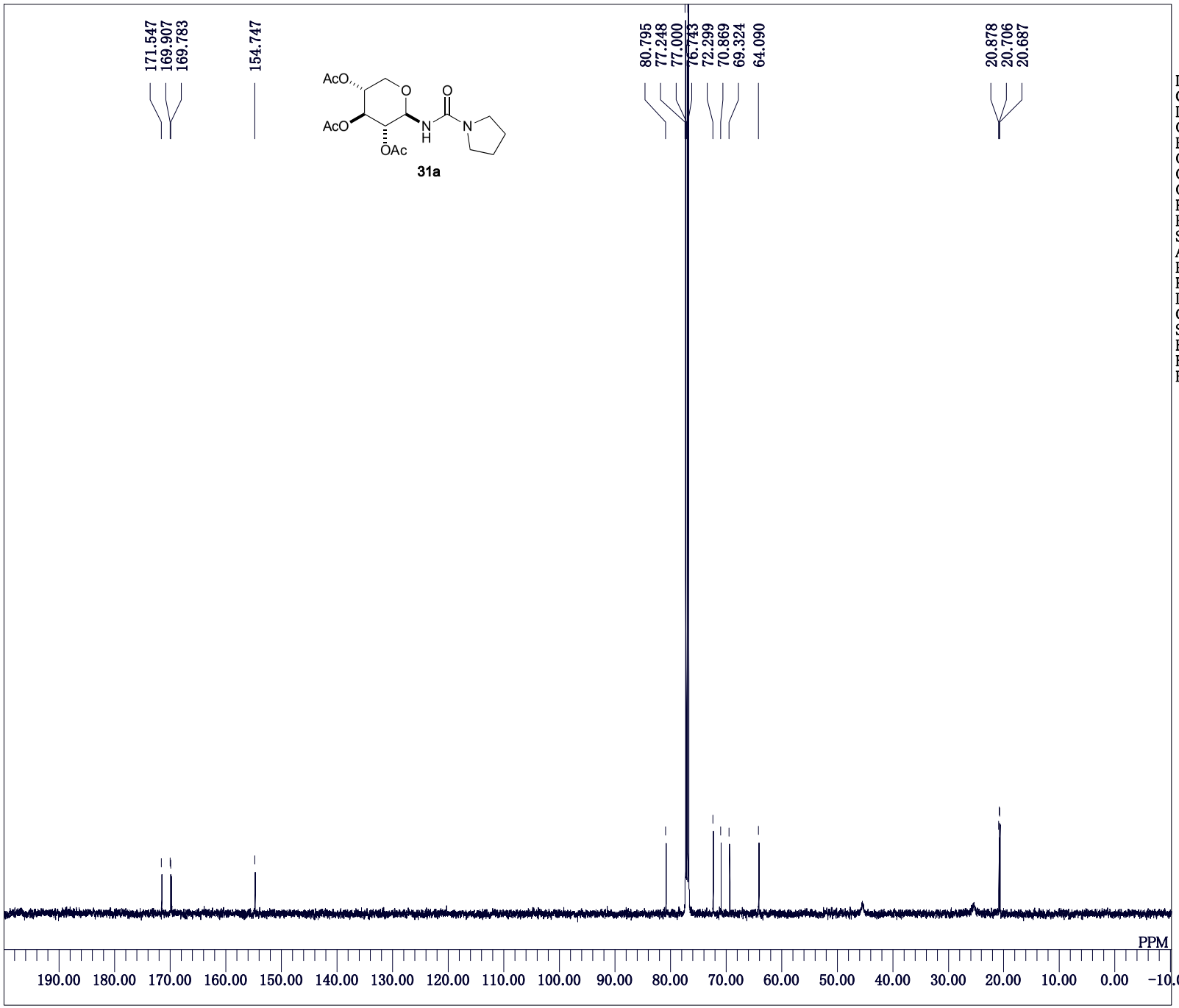
DFILE	SAE1047_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-02-20 22:07:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	256
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50


169.631
169.488
169.192
156.358
77.257
77.000
76.752
76.638
68.810
67.894
67.351
61.372
48.806
33.531
33.435
25.445
24.701
20.783
20.706
20.687
AcO
AcO
OAc
O
O
N
H
N
H
29b
DFILE sou4118_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2012-02-19 12:35:36
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 17.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 50
PPM
190.00 180.00 170.00 160.00 150.00 140.00 130.00 120.00 110.00 100.00 90.00 80.00 70.00 60.00 50.00 40.00 30.00 20.00 10.00 0.00 -10.00

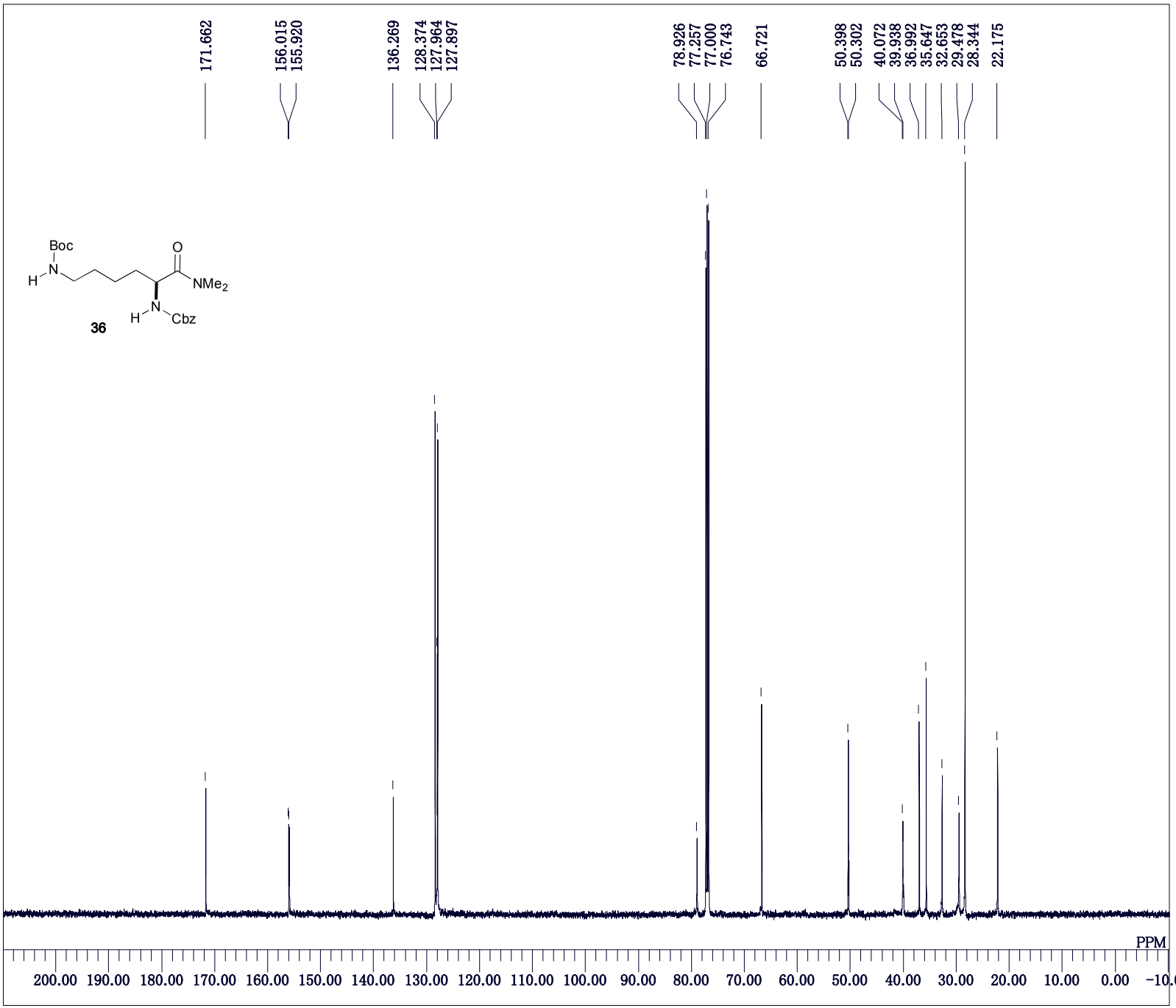

171.532
171.494
171.456
158.841
145.807
129.465
127.920
126.776
81.438
74.420
71.817
70.463
64.933
50.754
49.505
49.334
49.162
49.000
48.828
48.657
48.485
23.323
20.605
20.558
AcO
AcO
OAc
O
N
H
N
H
O
30a
DFILE SAE2047-13C_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2012-07-04 20:18:35
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 1.00 Hz
RGAIN 50
PPM
200.00 190.00 180.00 170.00 160.00 150.00 140.00 130.00 120.00 110.00 100.00 90.00 80.00 70.00 60.00 50.00 40.00 30.00 20.00 10.00 0.00



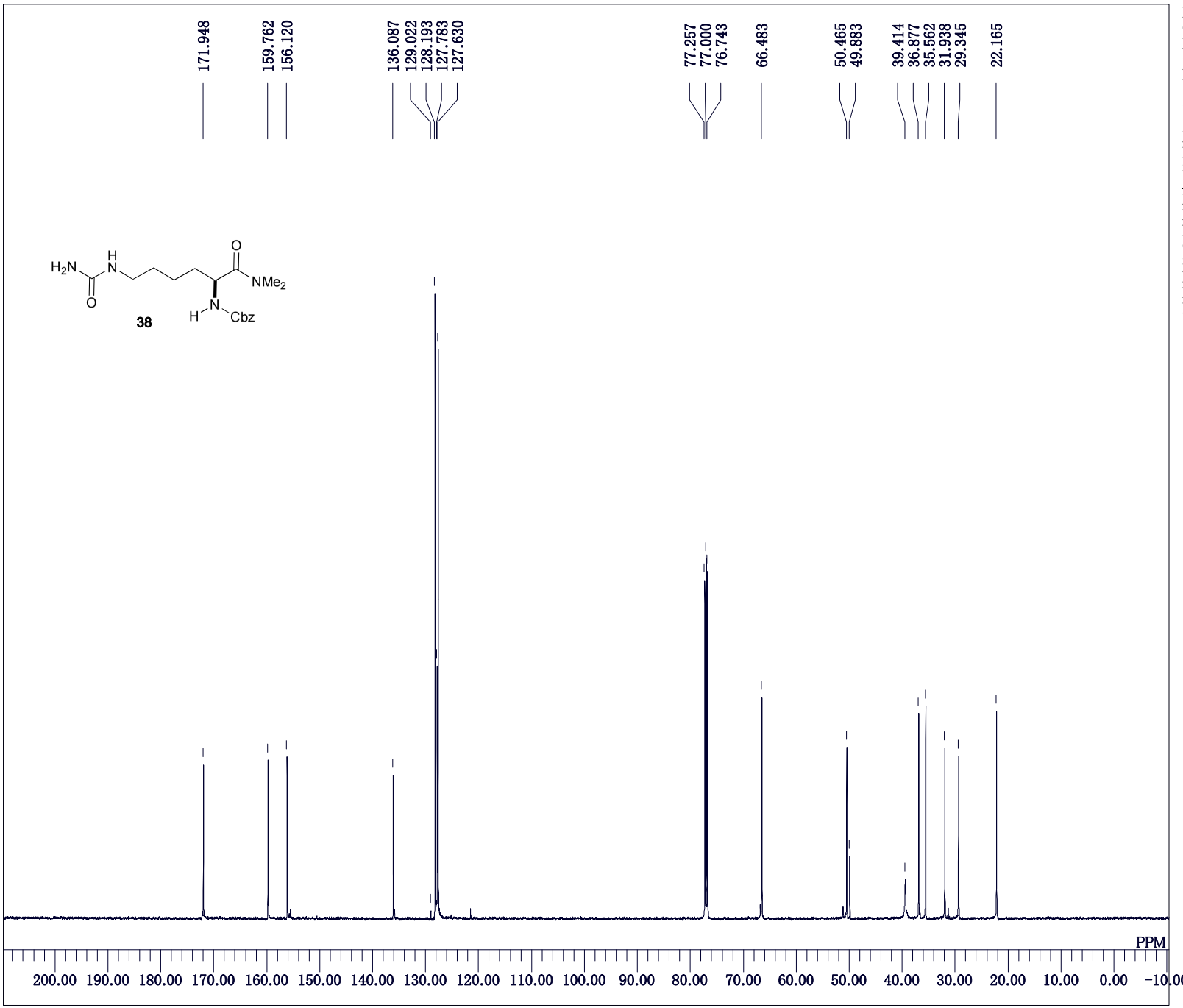
DFILE	SAE2017-13C_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-06-21 14:12:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



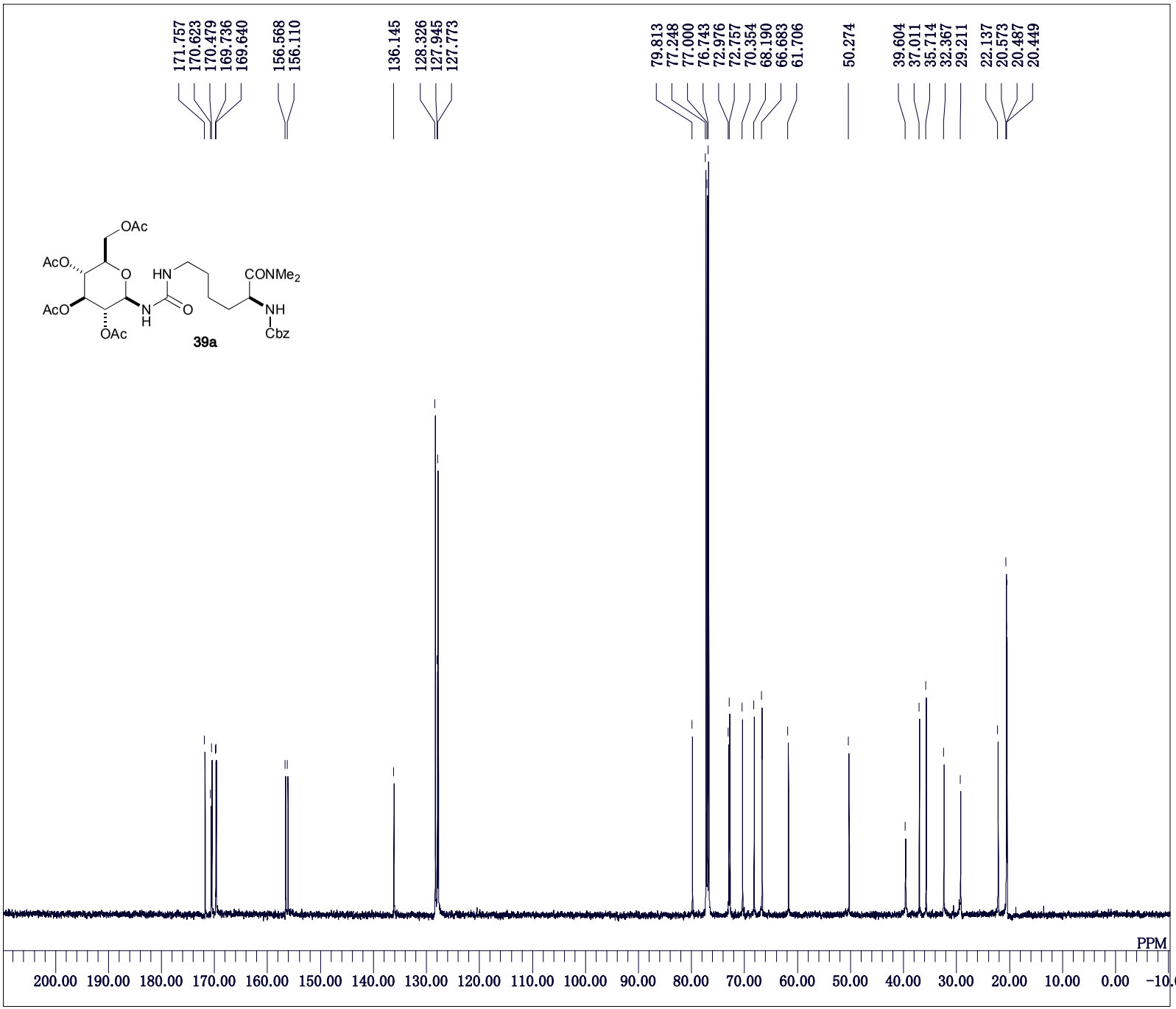
DFILE	SAE2051-13c_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2012-07-04 16:01:36
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



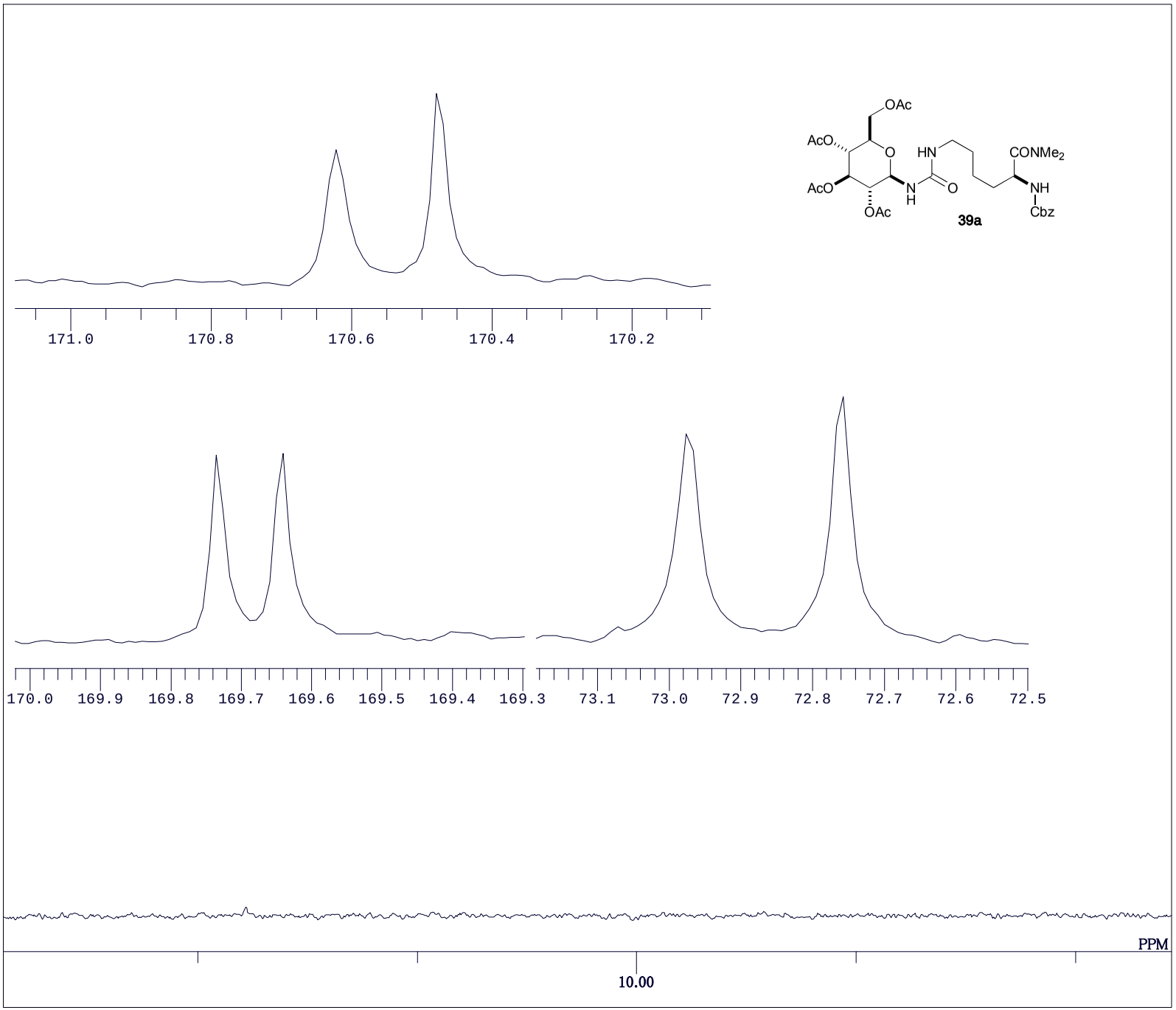
DFILE	sou3009data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-11 11:52:38
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



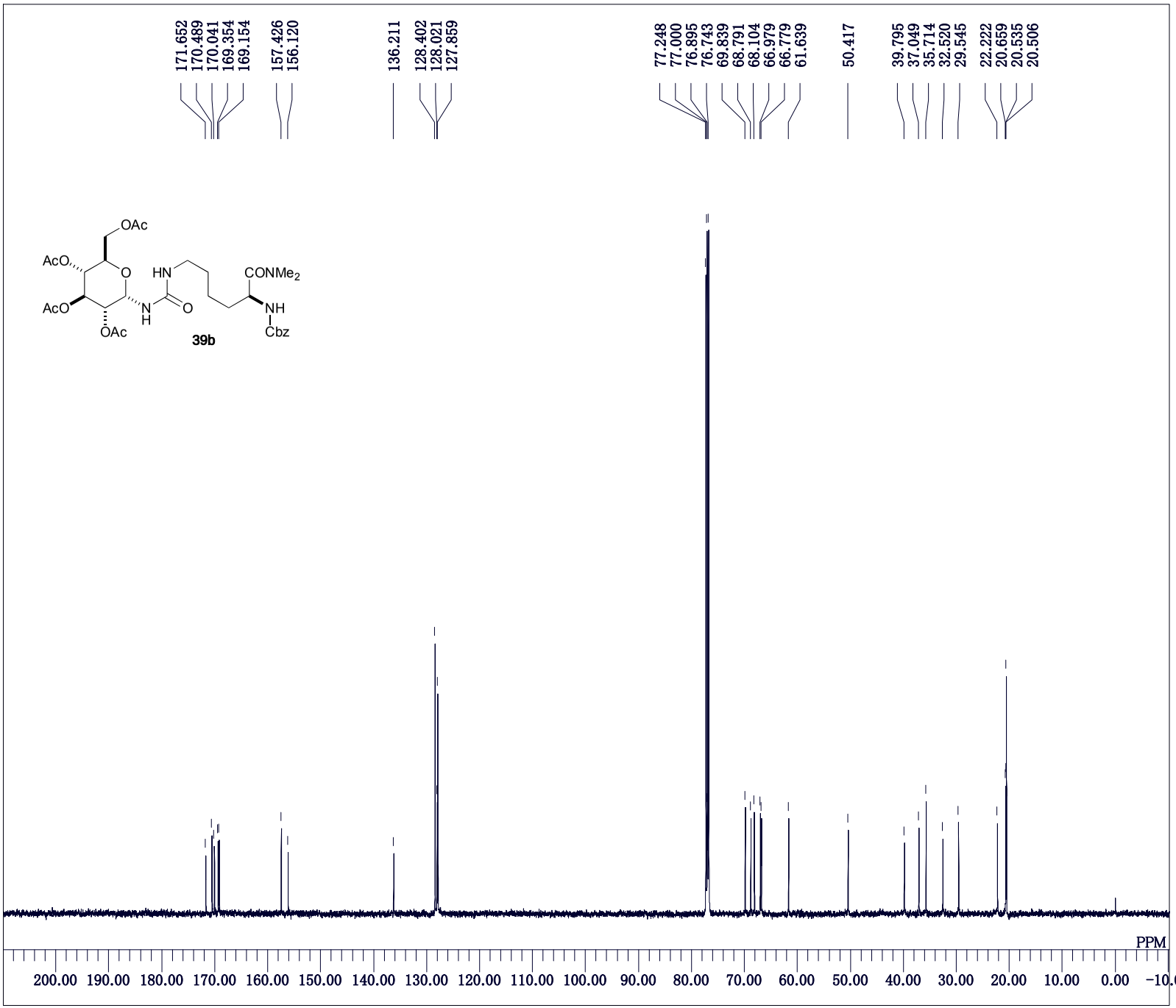
DFILE	sou4074_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-11 12:25:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



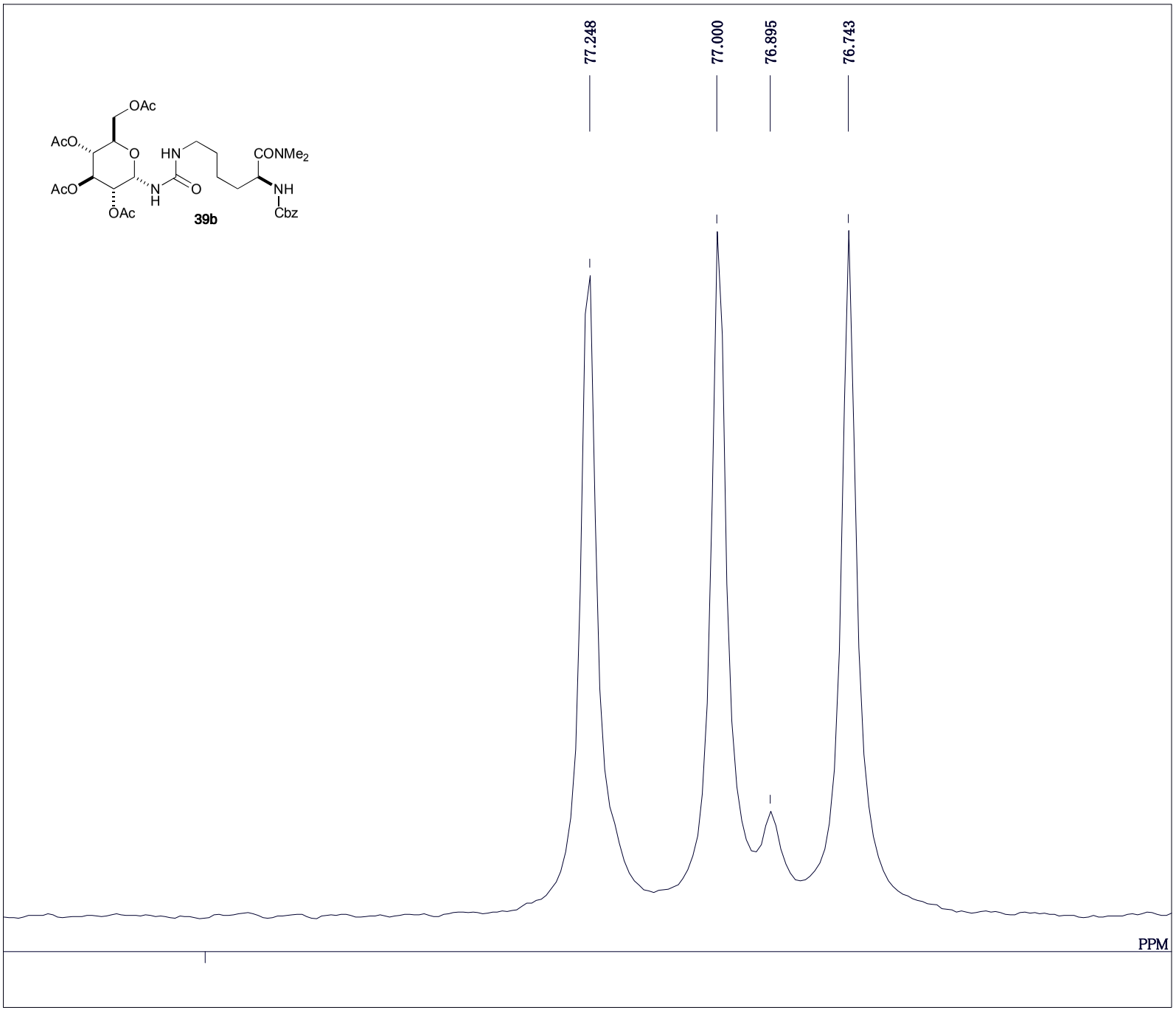
DFILE	sou3015data_Carbon-3-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-30 15:58:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



DFILE	sou3015data_Carbon-3-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-30 15:58:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



DFILE	sou3012data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-14 15:43:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50



DFILE	sou3012data_Carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2011-11-14 15:43:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32780
FREQU	39308.18 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50