Electronic Supplementary Material (ESI) for ChemComm.

Programmed chemo-enzymatic synthesis of the oligosaccharide component of a synthetic carbohydrate-based antibacterial vaccine candidate

Stéphane Salamone,[ab] Catherine Guerreiro,[ab] Emmanuelle Cambon,[cde] Isabelle André,[cde] Magali Remaud-Siméon, [cde] and Laurence A. Mulard*[ab]

[a] Institut Pasteur, Unité de Chimie des Biomolécules, 28 rue du Dr Roux, 75724 Paris Cedex 15, France
E-mail: laurence.mulard@pasteur.fr, Tel : +33 (0) 1 40 61 38 20
[b] CNRS UMR 3523, Institut Pasteur, F-75015 Paris, France
[c] Université de Toulouse; INSA, UPS, INP; LISBP, 135 Avenue de Rangueil, F-31077 Toulouse, France
[d] CNRS, UMR5504, F-31400 Toulouse, France
[e] INRA, UMR792 Ingénierie des Systèmes Biologiques et des Procédés, F-31400 Toulouse, France

Content

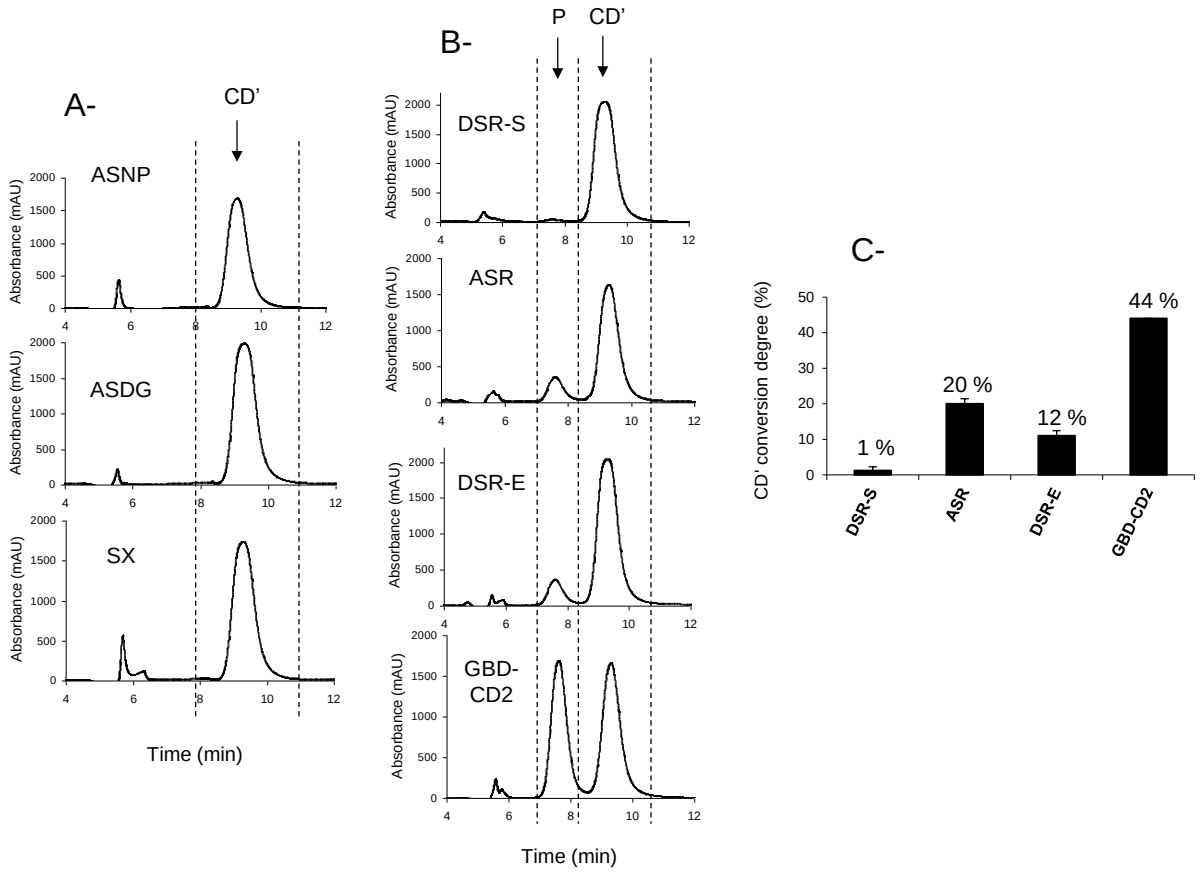


Figure S1: HPLC analysis with UV detection (λ = 220 nm) of CD' glucosylation reactions catalyzed by GH13 glucansucrases (panel A) and GH70 glucansucrases (panel B). Panel C: CD' conversion degree in reactions catalyzed by GH70 glucansucrases (reaction conditions: 30 °C, 146 mM Sucrose, 146 mM CD', 24 h, 1U/mL of enzyme). DSR-S: *Leuconostoc mesenteroides* NRRL B-512F dextransucrase ; ASR : *L. mesenteroides* NRRL B-1355 alternansucrase ; DSR-E: *L. mesenteroides* NRRL B-1299 dextransucrase; GBD-CD2: N(123)-glucan-binding domain-catalytic domain 2 (ΔN(123)-GBD-CD2) is a truncated form of the bifunctional glucansucrase DSR-E from *L. mesenteroides* NRRL B-1299

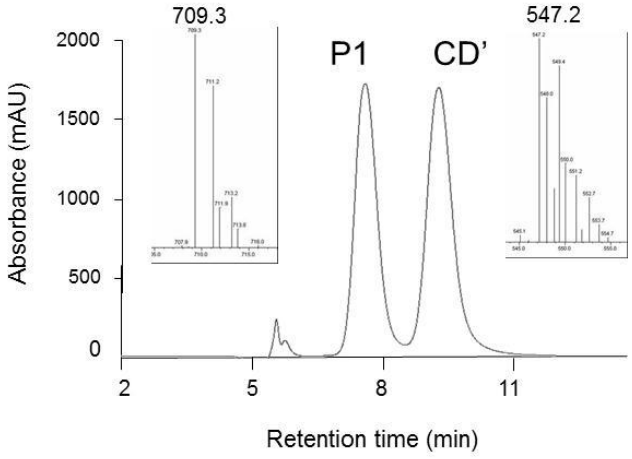


Figure S2: HPLC-MS analysis (λ = 220 nm) of the CD' glucosylation reaction catalyzed by GBD-CD2.

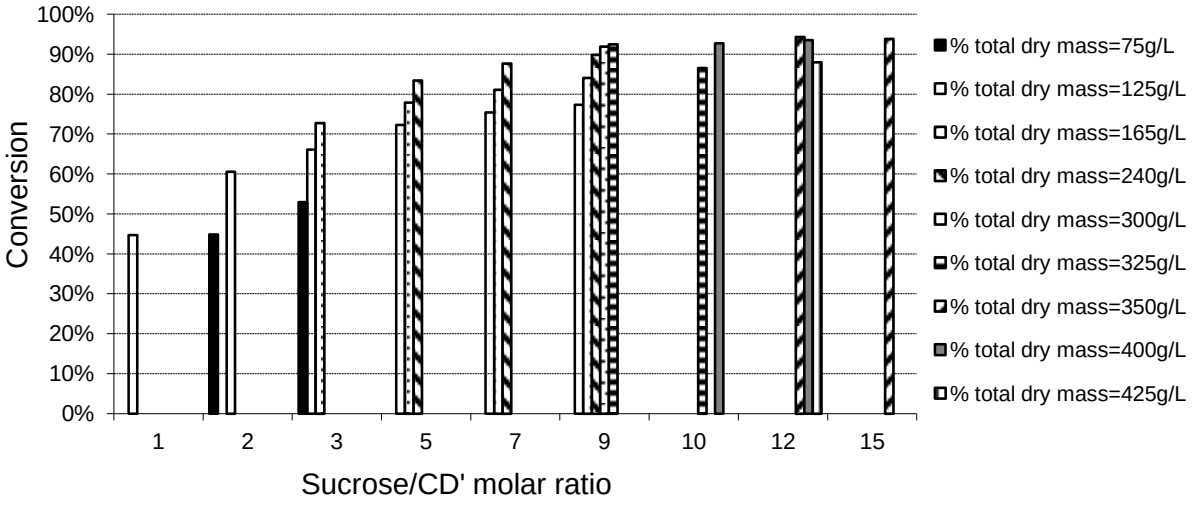
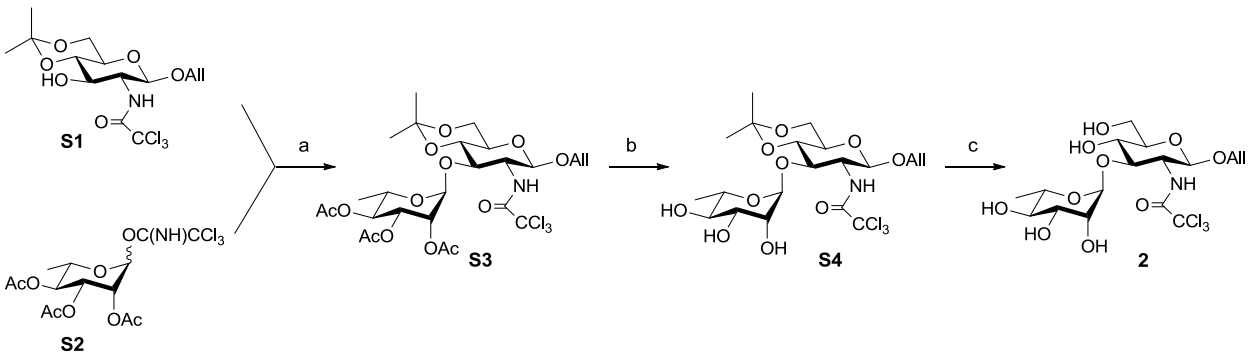


Figure S3: Conversion rate of CD' (**2**) into ECD' (**3**) using GBD-CD2 at different [Sucrose]/[CD'] ratios and initial total dry mass.



Scheme S1 Preparation of disaccharide acceptor **2**. *Reagents and conditions:* a) TMSOTf, DCM, -15°C, 93%; b) MeONa, MeOH, 86%; c) TFA 50% aq., DCM, 95%

Experimental data

- **Enzymatic glucosylation**
    - General methods

Enzymes. Glucansucrases from families 13 and 70 were tested in acceptor reactions with sucrose as donor and CD' (**2**) as acceptor. The selected enzymes from the Glycoside-Hydrolase (GH) family 13 include the amylosucrases of *Neisseria polysaccharea* (*Np*AS)[1] and *Deinococcus geothermalis* (*Dg*AS)[2] and the sucrose hydrolase from *Xanthomonas campestris* pv. c*ampestris* (*Xc*SH).[3] In the GH family 70, the native dextransucrase from *L. mesenteroides* NRRL B-512F (DSR-S),[4] one of its truncated recombinant form (DSR-S vardel Δ4N),[5] the recombinant form of the alternansucrase from *L. mesenteroides* NRRL B-1355 (ASR),[6] the native dextransucrase from *L. mesenteroides* NRRL B-1299 (DSR-E), which possesses two catalytic domains (CD1 and CD2 showing α-1,6 linkage and α-1,2 specificity, respectively[7] and the α-1,2-branching sucrase, a transglucosylase engineered from DSR-E (GBD-CD2)[8] that catalyzes exclusively the α-1,2 transfer of the glucosyl moiety from sucrose onto α-1,6 dextran chains were assayed.

Acceptor reaction assay. Acceptor reactions were performed in 1 mL of mixture containing equimolar amounts of sucrose and CD' (146 mM) in Tris-HCl 50 mM pH 7.5 for *Np*AS and *Xc*SH assays, Tris-HCl 50 mM pH 8 for *Dg*AS, sodium acetate buffer (AcONa) (20 mM, pH 5.4) for ASR assays, in AcONa (50 mM, [$CaCl_2$] = 0.5 g/L, pH 5.2) for DSR-S assays, in AcONa (20 mM, [$CaCl_2$] = 0.5 g/L, pH 5.4) for DSR-E and GBD-CD2 assays using 1 U/mL of enzyme at 30 °C for 24 h. One unit is defined as the amount of enzyme that catalyzes the release of 1 µmol of fructose/min at 30 °C from 146 mM sucrose (for *Np*AS, *Dg*AS and *Xc*SH) or 292 mM sucrose for DSR-S, GBD-CD2, DSR-E and ASR. All reactions were stopped by heating at 95 °C for 5 min. The final mixture was centrifuged at 18000 g for 10 min, filtered on a 0.22 µm membrane and diluted by 10 fold before HPLC analysis. To optimize the glucosylation reaction catalyzed by GBD-CD2, different initial molar sucrose/CD' ratios varying from 1 to 15 and initial sugar dry mass values (DM) varying from 75 to 425 g/L were assayed (Figure S2).

HPLC analysis of the enzymatic reactions. The HPLC analysis device consisted of a Dionex P680 series pump, a shodex RI 101 series refractometer, a Dionex UVD 340 UV/Vis detector, and an autosampler HTC-PAL. An Aminex HPX-87K column (300 × 7.8 mm) was employed at 65 °C eluting with CH_3CN/ultra-pure water (10:90, v/v). Glucose and fructose production and sucrose depletion were followed by RI detection. The conversion of CD' (**2**) and the formation of ECD' (**3**) were determined from their UV responses measured at λ = 220 nm. Sensitivity was identical in the 205-220 nm wavelength range.

Semi-preparative HPLC. ECD' (**3**) was isolated by semi-preparative octadecyl reverse-phase chromatography column (250 × 10 mm, 4 µm) with MeOH:ultrapure water (30:70, v/v) at a constant rate of 2 mL/min. Detection was carried out with UV. The purity of the isolated compound was checked by analytical RP-HPLC.

LC-MS analyses. ESI-MS used a MSQ® Plus Mass Spectrometer (Dionex - Thermoscientific) equipped with a source at 450 °C. Separation was achieved with a quadrupole and detection was in the positive mode.

NMR analysis. Heteronuclear multiple bond correlation spectroscopy (HMBC) experiments were recorded on a Bruker Avance 400 spectrometer (proton frequency of 400.13 MHz and ^{13}C frequency of 100.62 MHz) using the Bruker standard pulses program. The delay time for evolution of long-range ^{13}C-^{1}H couplings was set to 120 ms. Typical used parameters were 1200 Hz spectral width for ^{1}H and 22,130 Hz for ^{13}C, 16 scans and 196 experiments were accumulated. Samples were analyzed at 300.3 K in 5 mm o.d. tubes. Chemical shifts are given using an external reference (d6-trimethylsilylpropionic acid, sodium salt).

- Synthesis and characterization of ECD' (**3**)

Selecting a glucansucrase for CD' glucosylation: Acceptor reactions were first carried out with an equimolar ratio of sucrose and CD' (146 mM). Among the substrates, only CD' absorbs in UV. After 24 h, the UV-HPLC profiles of the reaction mixtures obtained with GH family 13 enzymes (*Np*AS, *Dg*AS and *Xc*SH) showed only one single peak corresponding to CD', indicating that none of these enzymes could glucosylate CD'(Figure S1-A). In contrast, an additional peak was observed on the profiles issued from the reactions catalyzed by the GH 70 family enzymes (Figure S1-B). LC-MS analysis of CD' glucosylation product synthesized by ASR, DSR-E and GBD-CD2 revealed a molecular weight of either 695.8 or 709.3 Da, corresponding to the ECD' sodium and potassium adducts, respectively (Figure 3). Identical MS and NMR spectra were observed for the glucosylation product isolated from the three reactions indicating that the same compound was produced irrespective of the enzyme used.

*Optimization of ECD' (**3**) production with GBD-CD2:* The best conversion was obtained with GBD-CD2 (Figure S1-C), which was selected to further optimize ECD' production by acting on temperature (20, 25 and 30 °C), reaction time (24 h and 48 h), total dry mass values from 75 to 425 g/L), and [Suc]/[CD'] molar ratios (from 1 to 15). Preliminary experiments served to identify 30 °C as the best performing temperature and showed the absence of evolution post 24 h (data not shown). Adopting these conditions, we showed that CD' conversion increased with the initial sucrose/CD' ratio regardless of total dry mass values (Figure S2). Furthermore, for a given [Suc]/[CD'] ratio, the increase of total dry mass value up to 350 g/L always favoured CD' conversion. Overall, the highest CD' conversion (94%) was obtained at initial [Suc]/[CD'] ratio of 12 and 350 g/L total dry mass. Preparative RP-HPLC of the crude material provided pure glucosylated CD' (ECD', **3**) and unreacted CD' (**2**).

*Gram synthesis and structural characterization of ECD' (**3**)*

Allyl α-D-glucopyranosyl-(1→4)-α-L-rhamnopyranosyl-(1→3)-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (ECD', 3). Sucrose (15.65 g, 45.7 mmol) was added to a solution of acceptor CD' (1.94 g, 3.8 mmol) in sodium acetate buffer (AcONa, 20 mM ; [$CaCl_2$] = 0.5 g/L, pH 5.4, 50 mL) containing GBD-CD2 (1 U/mL). The mixture was gently stirred at 30 °C for 24 h. At this time, HPLC analysis indicated that conversion had reached 94%. Preparative HLPC of the crude mixture (H_2O/MeOH, 100:0 → 70:30) furnished

a mixture of disaccharide CD' and glucosylation product. The mixture was purified by column chromatography (DCM/MeOH 100:0 → 70:30) to give first the unreacted acceptor CD' (140 mg, 0.27 mmol), then the enzymatic glucosylation product (2.22 g, 3.30 mmol, 87%, corrected yield 94%) both as white amorphous solids. The product of enzymatic glucosylation, identified as being the ECD' trisaccharide, was isolated as a white amorphous solid. Compound **3** had R_f = 0.51 (DCM/MeOH 70:30). $[\alpha]^{24}_D$ = + 8.7° (*c* 1.0, H_2O). ^{1}H NMR (D_2O) δ 5.98-5.88 (m, 1H, C*H*=CH_2), 5.37-5.31 (m, J_{trans} = 17.3 Hz, 1H, CH=C*H*$_2$), 5.29-5.26 (m, J_{cis} = 10.4 Hz, 1H, CH=C*H*$_2$), 5.05 (d, $J_{1,2}$ = 3.9 Hz, 1H, H-1$_E$), 4.91 (br s, 1H, H-1$_C$), 4.78 (overlapped with D_2O, 1H, H-1$_D$), 4.40-4.35 (m, 1H, -OCH_{2All}), 4.24-4.18 (m, 1H, -OCH_{2All}), 4.13 (dq, $J_{4,5}$ = 9.6 Hz, 1H, H-5$_C$), 4.03-3.95 (m, 2H, H-6a$_D$, H-5$_E$), 3.90-3.77 (m, 7H, H-2$_D$, H-6b$_D$, H-3$_D$, H-6a$_E$, H-6b$_E$, H-2$_C$, H-3$_C$), 3.71 (pt, J = 9.3 Hz, 1H, H-3$_E$), 3.61-3.56 (m, 2H, H-2$_E$, H-4$_D$), 3.54-3.45 (m, 3H, H-4$_E$, H-4$_C$, H-5$_D$), 1.33 (d, $J_{5,6}$ = 6.3 Hz, 3H, H-6$_C$). ^{13}C NMR (D_2O) δ 167.4 (C_{NTCA}), 135.6 (*C*H=CH_2), 121.3 (CH=*C*H$_2$), 103.7 (C-1$_C$, $^1J_{CH}$ = 170.3 Hz), 102.3 (C-1$_E$, $^1J_{CH}$ = 170.6 Hz), 101.6 (C-1$_D$, $^1J_{CH}$ = 163.5 Hz), 94.2 (CCl_3), 83.7 (2C, C-4$_C$, C-3$_D$), 78.7 (C-5$_D$), 75.3 (C-3$_E$), 74.4 (C-5$_E$), 74.1 (C-2$_E$), 73.5 (C-2$_C$), 73.4 (OCH_{2All}), 71.9 (C-4$_E$), 71.6 (C-3$_C$), 71.3 (C-4$_D$), 70.7 (C-5$_C$), 63.3 (C-6$_D$), 62.7 (C-6$_E$), 59.9 (C-2$_D$), 19.3 (C-6$_C$). HRMS (ESI^+) *m/z* calcd for $C_{23}H_{36}Cl_3NO_{15}Na$ $[M+Na]^+$: 694.1048. Found: 694.1044.

- **Chemical synthesis**

Abbreviations. Ac: acetyl; anhydr.: Anhydrous; All: Allyl; Ar: Argon; CSA: Camphorsulfonic acid; DBU: 1,8-Diazabicyclo[5.4.0]undec-7-ene; DCE: 1,2-Dichloroethane; DCM: Dichloromethane; DMF: *N*,*N*-Dimethylformamide; DMP: 2,2-Dimethoxypropane; HR-ESI-TOF-MS: High Resolution Electrospray Ionisation/Time-Of-Flight Mass Spectra; HR-MALDI-TOF-MS: High Resolution Matrix-Assisted Laser Desorption-Ionisation/Time-Of-Flight Mass Spectra; iPr: isopropylidene; [Ir]: 1,5-Cyclooctadiene-bis(methyldiphenylphosphine)-iridium hexafluorophosphate; MS: Molecular sieves; NMR: Nuclear Magnetic Resonance; NTCA: trichloroacetamide; RP-HPLC: Reverse Phase High Performance Liquid Chromatography; *p*TSA: *p*-Toluenesulfonic acid; TFA: Trifluoroacetic acid; THF: Tetrahydrofuran; TLC: Thin-Layer Chromatography; TMSOTf: Trimethylsilyl trifluoromethanesulfonate.

General. Reagents were purchased from Sigma-Aldrich, Fluka, Alfa Aesar or TCI Europe and were used as received. Solvents were obtained from VWR International and Carlo Erba and were used as received. Air and water sensitive reactions were performed in dried glassware under Ar. Anhydr. toluene, Et_2O, DCE, THF, DMF, CH_3CN, MeOH and pyridine were delivered on MS and were used as received. Sodium hydride (60% dispersion in mineral oil) was washed with anhydr. pentane under a stream of Ar before use. 4Å MS were activated before use by heating at 250°C under vacuum. Analytical TLC was performed with silica gel 60 F254, 0.25 mm pre-coated TLC plates (Merck). Compounds were visualized using UV254 and/or orcinol (1 mg·mL^{-1}) in 10% aq. H_2SO_4 with charring. Flash column chromatography was carried out using silica gel (Merck, particle size 40-63 µm or 15-40 µm). NMR spectra were recorded at 303 K on a Bruker Avance spectrometer at 400 MHz (^{1}H) and 100 MHz (^{13}C) equipped with a BBO probe. Elucidations of chemical structures were based on ^{1}H, COSY, DEPT-135, HSQC, ^{13}C, ^{13}C gated decoupling and HMBC experiments. Signals are reported as m (multiplet), s (singlet), d (doublet), t (triplet), pt (pseudo triplet), dd (doublet of

doublet), dq (doublet of quadruplet), br s (broad singlet), and coupling constants are reported in hertz (Hz). Spectra were recorded in $CDCl_3$, CD_3OD and D_2O. Chemical shifts are reported in ppm (δ) relative to residual solvent peak, $CHCl_3$ in the case of $CDCl_3$, MeOH in the case of CD_3OD, HOD and 4,4-dimethyl-4-silapentane-1-sulfonic acid (DSS) in the case of D_2O, at 7.26/77.16, 3.31/49.0, and 4.79/0.0 ppm for the ^1H and ^{13}C spectra, respectively. Of the two magnetically non-equivalent geminal protons at C-6, the one resonating at lower field is denoted H-6a, and the one at higher field is denoted H-6b. Sugar residues are serially lettered according to the lettering of the repeating unit of the *S. flexneri* 2a O-specific polysaccharide and identified by a subscript in the listing of signal assignments. HR-ESI-TOF-MS were recorded on a WATERS QTOF Micromass instrument in the positive-ion electrospray ionisation (ESI^+) mode. Solutions were prepared using 1:1 CH_3CN/H_2O containing 0.1% formic acid or MeOH/water containing 10 mM ammonium acetate in the case of sensitive compounds. HR-MALDI-TOF-MS were recorded on a 4800 MALDI TOF/TOF AB SCIEX instrument in the positive-ion reflector mode using 2,5-dihydroxybenzoic acid (DHB) in CH_3CN/TFA (0.1%) as the matrix. Optical rotations were obtained using the sodium D line at ambient temperature on a Bellingham + Stanley Ltd. ADP220 polarimeter. Analytical RP-HPLC (λ = 215 nm) of the final product was carried out using an Aeris Peptide 3.6 µm C_{18} 100 Å 2.1 × 100 mm analytical column, eluting with a 0-20% linear gradient of CH_3CN in 0.08% aq. TFA over 20 min at a flow rate of 0.3 $mL \cdot min^{-1}$.

Allyl (2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-2-deoxy-4,6-*O*-isopropylidene-2-trichloroacetamido-β-D-glucopyranoside (S3). To a solution of the known acceptor[9] **S1** (489 mg, 1.21 mmol) and the known donor[10] **S2** (665 mg, 1.53 mmol, 1.3 equiv.) in anhydr. DCM (12 mL) containing 4Å MS (530 mg), under Ar atmosphere and cooled to -15°C, was added TMSOTf (13 µL, 72 µmol, 0.06 equiv.). The reaction mixture was stirred 30 min then Et_3N (50 µL) was added and the mixture was filtered over a pad of Celite and concentrated to dryness. Chromatography of the residue (cyclohexane-EtOAc, 90:10 → 80:20) afforded fully protected disaccharide **S3** (760 mg, 1.12 mmol, 93%) as a white amorphous solid. R_f = 0.27 (cyclohexane-EtOAc 70:30). $[\alpha]^{24}_D$ = - 30.9° (*c* 1.0; $CHCl_3$). ^1H NMR ($CDCl_3$) δ7.00 (d, $J_{NH,2}$ = 7.2 Hz, 1H, NH), 5.88-5.78 (m, 1H, C*H*=CH_2), 5.28-5.16 (m, 4H, CH=CH_2, H-2_C, H-3_C), 5.02 (pt, *J* = 9.7 Hz, 1H, H-4_C), 5.01 (d, $J_{1,2}$ = 8.3 Hz, 1H, H-1_D), 4.79 (d, $J_{1,2}$ = 1.5 Hz, 1H, H-1_C), 4.39 (pt, *J* = 9.4 Hz, 1H, H-3_D), 4.33-4.28 (m, 1H, -OCH_{2All}), 4.18 (dq, 1H, H-5_C), 4.08-4.03 (m, 1H, -OCH_{2All}), 3.96 (dd, $J_{6a,6b}$ = 10.8 Hz, $J_{5,6a}$ = 5.4 Hz, 1H, H-$6a_D$), 3.80 (pt, $J_{5,6b}$ = 10.8 Hz, 1H, H-$6b_D$), 3.64 (pt, *J* = 9.4 Hz, 1H, H-4_D), 3.40-3.34 (m, 2H, H-2_D, H-5_D), 2.08 (s, 3H, H_{Ac}), 2.03 (s, 3H, H_{Ac}), 1.96 (s, 3H, H_{Ac}), 1.52 (s, 3H, H_{iPr}), 1.41 (s, 3H, H_{iPr}), 1.17 (d, $J_{5,6}$ = 6.3 Hz, 3H, H-6_C). ^{13}C NMR ($CDCl_3$) δ170.3, 170.0, 169.9 (3C, C_{Ac}), 162.2 (C_{NTCA}), 133.3 (*C*H=CH_2), 118.3 (CH=*C*H_2), 99.6 (C_{iPr}), 98.3 (C-1_C, $^1J_{CH}$ = 173.1 Hz), 98.2 (C-1_D, $^1J_{CH}$ = 165.2 Hz), 92.3 (CCl_3), 75.2 (C-3_D), 73.1 (C-4_D), 71.0 (C-4_C), 70.8 (-OCH_{2All}), 69.6 (C-3_C), 69.4 (C-2_C), 67.2 (C-5_D), 66.6 (C-5_C), 62.2 (C-6_D), 60.0 (C-2_D), 29.2 (C_{iPr}), 20.9, 20.8, 20.7 (3C, C_{Ac}), 19.3 (C_{iPr}), 17.4 (C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{26}H_{36}Cl_3NO_{13}Na$ $[M+Na]^+$: 698.1150. Found: 698.1158.

Allyl (α-L-rhamnopyranosyl)-(1→3)-2-deoxy-4,6-*O*-isopropylidene-2-trichloroacetamido-β-D-glucopyranoside (S4). To a solution of protected disaccharide **S3** (128 mg, 189 µmol) in anhydr. MeOH (3 mL) and under Ar atmosphere, was added MeONa (0.5 M, 115 µL, 58 µmol, 0.3 equiv.). The reaction mixture was stirred for 2 h, then quenched by addition of Dowex 50Wx8-200 and filtered over a pad of Celite. The solvent was removed under vacuum and the residue was purified by column chromatography (EtOAc) to give triol **S4** (90 mg, 163 µmol, 86%) as a white amorphous solid. R_f = 0.45 (EtOAc). $[\alpha]^{24}_D$ = - 64.5° (*c* 1.0; $CHCl_3$). ^{1}H NMR (MeOD) δ5.86-5.77 (m, 1H, C*H*=CH_2), 5.25-5.19 (m, J_{trans} = 17.3 Hz, 1H, CH=C*H*$_2$), 5.11-5.07 (m, J_{cis} = 10.5 Hz, 1H, CH=C*H*$_2$), 4.79 (d, $J_{1,2}$ = 1.5 Hz, 1H, H-1_C), 4.64-4.58 (m, 1H, H-1_D), 4.26-4.21 (m, 1H, -OCH_{2All}), 4.04-3.99 (m, 1H, -OCH_{2All}), 3.93 (dq, $J_{4,5}$ = 9.6 Hz, 1H, H-5_C), 3.88-3.82 (m, 3H, H-2_D, H-3_D, H-$6a_D$), 3.78 (pt, *J* = 10.3 Hz, 1H, H-$6b_D$), 3.72 (dd, $J_{2,3}$ = 3.3 Hz, 1H, H-2_C), 3.64-3.60 (m, $J_{4,5}$ = 9.4 Hz, 1H, H-4_D), 3.58-3.54 (m, 1H, H-3_C), 3.29-3.24 (m, 2H, H-4_C, H-5_D), 1.49 (s, 3H, H_{iPr}), 1.35 (s, 3H, H_{iPr}), 1.17 (d, $J_{5,6}$ = 6.2 Hz, 3H, H-6_C). ^{13}C NMR (MeOD) δ164.3 (C_{NTCA}), 135.0 (*C*H=CH_2), 117.5 (CH=*C*H_2), 102.5 (C-1_C), 101.7 (C-1_D), 100.8 (C_{iPr}), 94.0 (CCl_3), 78.2 (C-3_D), 74.2 (C-4_D), 73.8 (C-4_C), 72.3 (2C, C-2_C, C-3_C), 71.3 (-OCH_{2All}), 69.7 (C-5_C), 68.6 (C-5_D), 63.1 (C-6_D), 59.4 (C-2_D), 29.5 (C_{iPr}), 19.3 (C_{iPr}), 18.1 (C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{20}H_{30}Cl_3NO_{10}Na$ $[M+Na]^+$: 572.0833. Found: 572.0805.

Allyl (α-L-rhamnopyranosyl)-(1→3)-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (2). To a solution of triol **S4** (239 mg, 0.43 mmol) in DCM (10 mL) was added TFA (50% aq., 5 mL). The biphasic mixture was vigorously stirred for 30 min then repeatedly coevaporated with toluene. The residue was purified by column chromatography (EtOAc/MeOH, 98:2) to give pentaol **2** (208 mg, 0.41 mmol, 95%) as a white amorphous solid. R_f = 0.60 (DCM/MeOH 84:16). $[\alpha]^{23}_D$ = - 55.2° (*c* 1.0; H_2O). ^{1}H NMR (D_2O) δ6.03-5.94 (m, 1H, C*H*=CH_2), 5.42-5.36 (m, J_{trans} = 17.3 Hz, 1H, CH=C*H*$_2$), 5.35-5.31 (m, J_{cis} = 10.4 Hz, 1H, CH=C*H*$_2$), 4.96 (d, $J_{1,2}$ = 1.6 Hz, 1H, H-1_C), 4.84 (d, $J_{1,2}$ = 8.3 Hz, 1H, H-1_D), 4.46-4.40 (m, 1H, -OCH_{2All}), 4.29-4.24 (m, 1H, -OCH_{2All}), 4.07 (dq, $J_{4,5}$ = 9.8 Hz, 1H, H-5_C), 4.02 (dd, $J_{6a,6b}$ = 12.4 Hz, $J_{5,6a}$ = 2.0 Hz, 1H, H-$6a_D$), 3.96-3.83 (m, 4H, H-2_D, H-2_C, H-$6b_D$, H-3_D), 3.80 (dd, $J_{3,4}$ = 9.9 Hz, $J_{2,3}$ = 3.4Hz, 1H, H-3_C), 3.66-3.54 (m, 2H, H-4_D, H-5_D), 3.51 (pt, 1H, H-4_C), 1.30 (d, $J_{5,6}$ = 6.3 Hz, 3H, H-6_C). ^{13}C NMR (D_2O) δ167.4 (C_{NTCA}), 135.7 (*C*H=CH_2), 121.4 (CH=*C*H_2), 103.9 (C-1_C), 101.7 (C-1_D), 94.2 (CCl_3), 83.4 (C-3_D), 78.7 (C-5_D), 74.5 (C-4_C), 73.4 (-OCH_{2All}), 73.3 (C-2_C), 73.0 (C-3_C), 71.6 (C-5_C), 71.3 (C-4_D), 63.4 (C-6_D), 60.0 (C-2_D), 19.1 (C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{17}H_{26}Cl_3NO_{10}Na$ $[M+Na]^+$: 532.0520. Found: 532.0511.

Allyl (2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (5). To a solution of diol **4**[*] (826 mg, 0.68 mmol) in anhydr. CH_3CN (680 µL) and under Ar, were added trimethyl orthoacetate (260 µL, 2.04 mmol, 3.0 equiv.) and *p*-TSA (5 mg, 0.03 mmol, 0.04 equiv.). The reaction mixture was stirred for 35 min then 80% aq. AcOH (0.7 mL) was added. The reaction mixture was stirred for another 25 min then it

[*] To be described elsewhere

was diluted with DCM (60 mL) and rapidly washed with cold water (1 × 10 mL) and brine (1 × 10 mL). The aqueous phase was extracted with DCM (1 × 10 mL) and the combined organics were dried (Na_2SO_4), filtered and co-evaporated with toluene to give crude alcohol **5** (802 mg, 0.64 mmol, 94%) as a white amorphous solid. R_f = 0.20 (cyclohexane/EtOAc 70:30). $[\alpha]^{24}_D$ = + 7.8° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.40-7.23 (m, 29H, Ph, NH), 7.19-7.16 (m, 2H, Ph), 5.97-5.88 (m, 1H, C*H*=CH_2), 5.35-5.29 (m, J_{trans} = 17.2 Hz, 1H, CH=C*H*$_2$), 5.26-5.20 (m, J_{cis} = 10.3 Hz, 1H, CH=C*H*$_2$), 5.24 (bs, 1H, H-2_C), 5.11 (bs, 1H, H-1_C), 5.00 (d, $J_{1,2}$ = 3.6 Hz, 1H, H-1_E), 4.93 (d, *J* = 11.0 Hz, 1H, H_{Bn}), 4.86 (d, *J* = 10.9 Hz, 1H, H_{Bn}), 4.84-4.48 (m, 11H, 10H_{Bn}, H-1_D), 4.41-4.36 (m, 1H, -OCH_{2All}), 4.22 (pt, *J* = 7.0 Hz, 1H, H-3_D), 4.13-4.05 (m, 3H, H-5_E, OH, -OCH_{2All}), 4.00-3.75 (m, 8H, H-4_D, H-5_D, H-$6a_D$, H-$6b_D$, H-2_D, H-3_E, H-3_C, H-5_C), 3.70-3.56 (m, 4H, H-4_E, H-2_E, H-$6a_E$, H-$6b_E$), 3.40 (pt, *J* = 9.0 Hz, 1H, H-4_C), 2.15 (s, 3H, H_{Ac}), 1.36 (d, $J_{5,6}$ = 6.2 Hz, H-6_C). ^{13}C NMR ($CDCl_3$) δ 170.3 (C_{Ac}), 161.8 (C_{NTCA}), 138.7, 138.1, 138.0, 137.9, 137.6, 137.4 (6C_{quat}, Ph), 133.6 (*C*H=CH_2), 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 127.8, 127.7 (30C, Ph), 117.7 (CH=*C*H_2), 99.2 (C-1_E), 98.1 (C-1_D), 97.0 (C-1_C), 92.4 (CCl_3), 85.4 (C-4_C), 81.7 (C-3_E), 79.9 (C-2_E), 77.8 (C-4_E), 76.5 (C-4_D), 75.6 (C_{Bn}), 75.2 (C-3_D), 75.1 (C_{Bn}), 74.3 (C-5_D), 74.0, 73.6 (4C, C_{Bn}), 71.9 (C-2_C), 71.4 (C-5_E), 70.0 (OCH_{2All}), 69.2 (C-6_E), 68.6 (C-6_D), 68.3 (C-3_C), 67.6 (C-5_C), 55.4 (C-2_D), 21.1 (C_{Ac}), 17.8 (C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{67}H_{74}Cl_3NO_{16}Na$ $[M+Na]^+$: 1276.3971. Found: 1276.4047.

Allyl (3,4-di-*O*-benzyl-2-*O*-levulinoyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (7). To a solution of the crude acceptor **5** (505 mg, 402 µmol, 1.0 equiv.) and of the known rhamnosyl donor[11] **6** (280 mg, 477 µmol, 1.2 equiv.) in anhydr. Et_2O (8 mL) containing 4Å MS (500 mg), under Ar and cooled to -15 °C, was added TMSOTf (4 µL, 22 µmol, 0.05 equiv.). The reaction mixture was stirred for 40 min. Et_3N (10 µL) was added and the mixture was filtered over a pad of Celite and concentrated to dryness. The residue was purified by column chromatography (Toluene/EtOAc, 90:10 → 80:20) to give tetrasaccharide **7** (580 mg, 345 µmol, 86%) as a white amorphous solid. R_f = 0.27 (Toluene/EtOAc 86:14). $[\alpha]^{24}_D$ = + 9.8° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.39-7.16 (m, 41H, Ph, NH), 5.95-5.85 (m, 1H, C*H*=CH_2), 5.68 (bs, 1H, H-2_B), 5.31-5.26 (m, J_{trans} = 17.3 Hz, 1H, CH=C*H*$_2$), 5.21-5.17 (m, J_{cis} = 10.3 Hz, 1H, CH=C*H*$_2$), 5.14 (bs, 1H, H-2_C), 5.07 (d, $J_{1,2}$ = 2.4 Hz, 1H, H-1_C), 4.97-4.40 (m, 19H, 16H_{Bn}, H-1_D, H-1_B, H-1_E), 4.38-4.32 (m, 1H, -OCH_{2All}), 4.26 (bt, 1H, H-3_D), 4.10-4.05 (m, 1H, -OCH_{2All}), 3.99-3.69 (m, 14H, H-3_C, H-3_E, H-5_E, H-5_D, H-5_C, H-4_E, H-5_B, H-3_B, H-$6a_E$, H-$6b_E$, H-$6a_D$, H-$6b_D$, H-2_D, H-4_D), 3.48-3.43 (m, 2H, H-2_E, H-4_C), 3.38 (pt, *J* = 9.4 Hz, 1H, H-4_B), 2.77-2.68 (m, 4H, 2×CH_{2Lev}), 2.18 (s, 3H, CH_{3Lev}), 2.10 (s, 3H, H_{Ac}), 1.24 (d, $J_{5,6}$ = 6.3 Hz, 3H, H-6_B), 1.16 (d, $J_{5,6}$ = 6.2 Hz, 3H, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 206.5 (C_{Lev}), 171.9 (C_{Lev}), 170.0 (C_{Ac}), 161.8 (C_{NTCA}), 138.8, 138.6, 138.5, 138.4, 138.3, 138.0, 137.5 (8C_{quat}, Ph), 133.7 (*C*H=CH_2), 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4 (40C, Ph), 117.8 (CH=*C*H_2), 98.6 (C-1_E), 97.9 (C-1_D), 96.5 (C-1_C), 92.4 (CCl_3), 81.7 (C-3_E), 81.6 (C-2_E), 79.9 (C-4_B), 77.7 (C-4_E), 77.0 (C-3_B), 76.4 (C-4_D), 75.6 (2C, C_{Bn}), 75.2 (C-3_D), 75.0 (C_{Bn}), 74.3 (C-5_D), 73.9, 73.8, 73.5 (3C, C_{Bn}), 72.9 (C_{Bn}), 71.8 (C-5_E), 71.6 (C-

2_C), 70.1 (2C, C_{Bn}, OCH_{2All}), 69.2 (C-6_D), 68.8 (C-5_B), 68.6 (C-2_B), 68.3 (C-6_E), 68.0 (C-5_C), 56.3 (C-2_D), 38.1 (CH_{2Lev}), 30.0 (CH_{3Lev}), 28.3 (CH_{2Lev}), 21.2 (C_{Ac}), 18.5 (C-6_C), 18.1 (C-6_B). HRMS (ESI^+) *m/z* calcd for $C_{92}H_{102}Cl_3NO_{22}Na$ $[M+Na]^+$: 1700.5857. Found: 1700.5919.

Allyl (3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (8). To a solution of tetrasaccharide **7** (489 mg, 291 µmol) in anhydr. pyridine (3.6 mL), cooled to 0 °C and under Ar, were added AcOH (2.4 mL) and hydrazine hydrate (71 µL, 1.46 mmol, 5.0 equiv.), then the reaction mixture was allowed to warm to room temperature. After 70 min, cold water (15 mL) was added to the reaction mixture, which was then extracted with DCM (4 × 20 mL). The combined organics were dried (Na_2SO_4), filtered, evaporated and co-evaporated twice with toluene. The residue was purified by column chromatography (Toluene/EtOAc, 95:5 → 85:15) to give alcohol **8** (423 mg, 267 µmol, 92%) as a white foam. R_f = 0.22 (Toluene/EtOAc 86:14). $[\alpha]^{24}_D$ = - 6.1° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.39-7.13 (m, 41H, Ph, NH), 5.94-5.84 (m, 1H, C*H*=CH_2), 5.30-5.24 (m, J_{trans} = 17.2 Hz, 1H, CH=CH_2), 5.19-5.16 (m, J_{cis} = 10.5 Hz, 1H, CH=CH_2), 5.12 (bdd, $J_{2,3}$ = 3.2 Hz, 1H, H-2_C), 5.07 (d, $J_{1,2}$ = 2.0 Hz, 1H, H-1_C), 5.00 (d, $J_{1,2}$ = 3.0 Hz, 1H, H-1_E), 4.96 (d, $J_{1,2}$ = 1.7 Hz, 1H, H-1_B), 4.89 (d, *J* = 11.0 Hz, 1H, H_{Bn}), 4.86-4.48 (m, 14H, H_{Bn}), 4.81 (d, $J_{1,2}$ = 8.0 Hz, 1H, H-1_D), 4.41 (d, *J* = 12.1 Hz, 1H, H_{Bn}), 4.35-4.30 (m, 1H, -OCH_{2All}), 4.28-4.24 (m, 2H, H-3_D, H-2_B), 4.09-4.03 (m, 1H, -OCH_{2All}), 3.97 (bdd, 1H, H-3_C), 3.92-3.73 (m, 8H, H-4_D, H-$6a_D$, H-$6b_D$, H-5_D, H-5_C, H-2_D, H-3_E, H-5_E), 3.69-3.60 (m, 5H, H-4_E, H-$6a_E$, H-$6b_E$, H-5_B, H-3_B), 3.56 (pt, *J* = 9.1 Hz, 1H, H-4_C), 3.51-3.44 (m, 2H, H-4_B, H-2_E), 2.73 (bs, 1H, OH), 2.07 (s, 3H, H_{Ac}), 1.26 (d, $J_{5,6}$ = 6.2 Hz, 3H, H-6_B), 1.22 (d, $J_{5,6}$ = 6.2 Hz, 3H, H-6_C). ^{13}C NMR ($CDCl_3$) δ 169.9 (C_{Ac}), 161.6 (C_{NTCA}), 138.7, 138.6, 138.4, 138.2, 138.1, 137.9, 137.4 (8C_{quat}, Ph), 133.7 (*C*H=CH_2), 128.6, 128.5, 128.4, 128.3, 128.0, 127.9, 127.8, 127.7, 127.6 (40C, Ph), 117.8 (CH=*C*H_2), 102.6 (C-1_B), 98.7 (C-1_E), 97.7 (C-1_D), 95.6 (C-1_C), 92.4 (CCl_3), 81.6 (C-3_E), 81.2 (C-2_E), 80.0 (C-4_B), 79.6 (C-4_C), 79.2 (C-3_B), 78.5 (C-3_C), 77.7 (C-4_E), 75.9 (C-4_D), 75.6, 75.1, 74.9 (3C, C_{Bn}), 74.0 (C-5_D), 73.8, 73.7, 73.5 (3C, C_{Bn}), 73.2 (2C, C-3_D, C_{Bn}), 72.5 (C-2_C), 71.6 (C_{Bn}), 71.5 (C-5_E), 70.0 (OCH_{2All}), 69.5 (C-6_D), 68.6 (C-6_E), 68.4 (C-5_B), 68.3 (C-5_C), 68.2 (C-2_B), 54.9 (C-2_D), 21.0 (C_{Ac}), 18.6 (C-6_C), 18.1 (C-6_B). HRMS (ESI^+) *m/z* calcd for $C_{87}H_{96}Cl_3NO_{20}Na$ $[M+Na]^+$: 1602.5490. Found: 1602.5555.

Allyl (3,4-di-*O*-benzyl-2-*O*-levulinoyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (9). To a solution of the acceptor **8** (400 mg, 253 µmol, 1.0 equiv.) and the known rhamnosyl donor[11] **6** (191 mg, 325 µmol, 1.3 equiv.) in anhydr. Et_2O (5 mL) containing 4Å MS (680 mg), under Ar and cooled to -15 °C, was added TMSOTf (3 µL, 17 µmol, 0.07 equiv.). The reaction mixture was stirred for 1.5 h then allowed to warm to room temperature for 30 min, then Et_3N (10 µL) was added and the mixture was filtered over a pad of Celite and concentrated to dryness. The residue was purified by column chromatography (Toluene/EtOAc, 95:5 → 85:15) to give pentasaccharide **9** (437 mg, 218 µmol, 86%) as a

white amorphous solid. R_f = 0.50 (Toluene/EtOAc 80:20). $[\alpha]^{24}_D$ = + 2.9° (*c* 1.0, $CHCl_3$). ^1H NMR ($CDCl_3$) δ 7.36-7.13 (m, 51H, Ph, NH), 5.94-5.84 (m, 1H, C*H*=CH_2), 5.56 (dd, $J_{2,3}$ = 3.0 Hz, $J_{1,2}$ = 1.9 Hz, 1H, H-2_A), 5.31-5.25 (m, J_{trans} = 17.2 Hz, 1H, CH=C*H*$_2$), 5.19-5.16 (m, J_{cis} = 10.5 Hz, 1H, CH=C*H*$_2$), 5.14 (bs, 1H, H-2_C), 5.08 (d, $J_{1,2}$ = 2.4 Hz, H-1_C), 5.06 (bs, 1H, H-1_A), 4.94-4.78 (m, 8H, 5H_{Bn}, H-1_B, H-1_D, H-1_E), 4.75-4.47 (m, 14H, H_{Bn}), 4.40-4.30 (m, 4H, H-2_B, H-3_D, 1H_{Bn}, -OCH_{2All}), 4.10-4.04 (m, 1H, -OCH_{2All}), 3.97-3.37 (m, 20H, H-2_D, H-2_E, H-3_A, H-3_B, H-3_C, H-3_E, H-4_A, H-4_B, H-4_C, H-4_D, H-4_E, H-5_A, H-5_B, H-5_C, H-5_D, H-5_E, H-$6a_D$, H-$6b_D$, H-$6a_E$, H-$6b_E$), 2.68-2.47 (m, 4H, 2×CH_{2Lev}), 2.09, 2.08 (s, 6H, CH_{3Lev}, H_{Ac}), 1.30, 1.19, 1.15 (d, $J_{5,6}$ = 6.2 Hz, 9H, H-6_A, H-6_B, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 206.1 (C_{Lev}), 171.8 (C_{Lev}), 170.1 (C_{Ac}), 161.8 (C_{NTCA}), 138.9, 138.8, 138.7, 138.6, 138.3, 138.2, 138.1, 137.5 (10C_{quat}, Ph), 133.7 (*C*H=CH_2), 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.6, 127.5, 127.4 (50C, Ph), 117.9 (CH=*C*H_2), 99.4 (C-1_A), 97.9 (C-1_E), 96.2 (C-1_C), 92.4 (CCl_3), 56.6 (C-2_D), 38.1 (CH_{2Lev}), 29.8 (CH_{3Lev}), 28.2 (CH_{2Lev}), 21.2 (C_{Ac}), 18.7, 18.4, 18.1 (3C, C-6_A, C-6_B, C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{112}H_{124}Cl_3NO_{26}Na$ $[M+Na]^+$: 2026.7374. Found: 2026.7920.

(3,4-Di-*O*-benzyl-2-*O*-levulinoyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-α/β-D-glucopyranose (10). 1,5-Cyclooctadiene-bis(methyldiphenylphosphine)-iridium hexafluorophosphate (3 mg, 3.5 µmol, 0.03 equiv.) was dissolved in anhydr. THF (2 mL) under Ar. Hydrogen was bubbled through the solution for 15 min, causing the color to change from red to yellow. The solution was degassed by complete evaporation of the solvent under vacuum. The activated iridium complex was dissolved under Ar in anhydr. THF (2 mL) then a solution pentasaccharide **9** (248 mg, 124 µmol) in anhydr. THF (3 mL) was added. The reaction mixture was stirred for 2.5 h at room temperature then a solution of iodine (98 mg, 386 µmol, 3.1 equiv.) in THF/water (4.5 mL, 2:1) was added. After 4h20, the excess iodine was quenched by addition of a 10% sodium bisulphite aqueous solution (15 mL). The reaction mixture was partially concentrated under reduced pressure to remove the THF then the aqueous layer was extracted with DCM (3 × 20 mL). The combined organics were dried (Na_2SO_4), filtered and concentrated. Column chromatography of the residue (Toluene/EtOAc, 85:15 → 80:20) gave hemiacetal **10** (227 mg, 115 µmol, 93%) as a white foam. R_f = 0.43 (Toluene/EtOAc 75:25). β anomer: R_f = 0.26 (Toluene/EtOAc 75:25). ^1H NMR ($CDCl_3$) δ 7.41-6.99 (m, 51H, Ph, NH), 5.60 (bs, 1H, H-2_A), 5.25-4.37 (m, 26H, 20H_{Bn}, H-1_A, H-1_B, H-1_C, H-1_D, H-1_E, H-2_C), 4.29-3.40 (m, 22H, H-2_B, H-2_D, H-2_E, H-3_A, H-3_B, H-3_C, H-3_D, H-3_E, H-4_A, H-4_B, H-4_C, H-4_D, H-4_E, H-5_A, H-5_B, H-5_C, H-5_D, H-5_E, H-$6a_D$, H-$6b_D$, H-$6a_E$, H-$6b_E$), 2.72-2.57 (m, 4H, 2×CH_{2Lev}), 2.13, 2.12 (bs, 6H, CH_{3Lev}, H_{Ac}), 1.36-1.19 (m, 9H, H-6_A, H-6_B, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 206.2 (C_{Lev}), 171.8 (C_{Lev}), 170.1, 170.0 (C_{Ac}), 162.6, 161.7 (C_{NTCA}), 138.8-127.3 (Ph), 99.3 (C-1_A), 97.4 (C-1_E), 96.5 (C-1_C), 94.6, 91.3 (C-1_D), 92.5, 92.3 (CCl_3), 58.2, 55.4 (C-2_D), 38.1, 38.0 (CH_{2Lev}), 29.8, 29.7 (CH_{3Lev}), 28.2, 28.1 (CH_{2Lev}), 21.0 (C_{Ac}), 18.8, 18.7, 18.3, 18.2, 18.1, 18.0 (3C, C-6_A, C-6_B, C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{109}H_{120}Cl_3NO_{26}Na$ $[M+Na]^+$: 1986.7062. Found: 1986.7177.

(3,4-Di-*O*-benzyl-2-*O*-levulinoyl–α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-α/β-D-glucopyranosyl *N*-(phenyl)trifluoroacetimidate (11). To a solution of hemiacetal **10** (352 mg, 179 µmol) in acetone (4.5 mL) and under Ar were added first cesium carbonate (68 mg, 209 µmol, 1.2 equiv.), then 2,2,2-trifluoro-*N*-(phenyl)acetimidoyl chloride (74 mg, 357 µmol, 2.0 equiv.). After 2 h, volatiles were evaporated and the residue was purified by flash chromatography (Toluene/EtOAc, 98:2 → 85:15) to give imidate **11** (316 mg, 148 µmol, 83%) as a white foam. NMR data indicated that donor **11** was present as a 1:0.3 mix with the corresponding oxazoline. Compound **11** had R_f = 0.63 (Toluene/EtOAc 85:15). ^{1}H NMR ($CDCl_3$) δ 7.36-7.10 (m, 53H, Ph), 6.88 (d, $J_{NH,2}$ = 8.5 Hz, 1H, NH), 6.77 (d, J = 7.4 Hz, 2H, Ph), 6.41 (br s, 1H, H-1_D), 5.54 (dd, $J_{1,2}$ = 1.8 Hz, $J_{2,3}$ = 3.0 Hz, 1H, H-2_A), 5.17 (bs, 1H, H-1_C), 5.12 (m, 1H, H-2_C), 5.05 (d, 1H, H-1_A), 4.95-4.34 (m, 24H, 20H$_{Bn}$, H-1_B, H-1_E, H-2_B, H-2_D), 4.16 (pt, $J_{2,3}$ = $J_{3,4}$ = 9.3 Hz, 1H, H-3_D), 4.00-3.27 (m, 19H, H-2_E, H-3_A, H-3_B, H-3_C, H-3_E, H-4_A, H-4_B, H-4_C, H-4_D, H-4_E, H-5_A, H-5_B, H-5_C, H-5_D, H-5_E, H-$6a_D$, H-$6b_D$, H-$6a_E$, H-$6b_E$), 2.67-2.51 (m, 4H, 2×CH_{2Lev}), 2.10 (bs 3H, CH_{3Lev}), 2.08 (s, 3H, H_{Ac}), 1.30-1.18 (m, 9H, H-6_A, H-6_B, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 206.2 (C_{Lev}), 171.8 (C_{Lev}), 170.1 (C_{Ac}), 163.0 (C=NPh), 161.9 (C_{NTCA}), 143.1, 138.9-127.5, 119.4 (Ph), 99.5 (C-1_A), 97.2 (C-1_C), 92.3 (CCl_3), 81.7 (C-3_E), 76.0 (C-3_D), 72.0 (C-2_C), 69.5 (C-2_A), 54.5 (C-2_D), 38.2 (CH_{2Lev}), 29.9 (CH_{3Lev}), 28.3 (CH_{2Lev}), 21.1 (C_{Ac}), 18.4, 18.3, 18.1 (3C, C-6_A, C-6_B, C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{117}H_{128}Cl_3F_3N_3O_{26}$ $[M+NH_4]^+$: 2152.7803. Found: 2152.4663, $C_{109}H_{122}Cl_3N_2O_{25}$ $[M\text{-}HOC(NPh)CF_3+NH_4]^+$: 1963.7402. Found: 1963.6669, $C_{110}H_{126}Cl_3N_2O_{26}$ $[M\text{-}OC(NPh)CF_3+OMe+NH_4]^+$: 1995.7665. Found: 1995.6724.

2-Azidoethyl (3,4-di-*O*-benzyl-2-*O*-levulinoyl–α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (12). A solution of donor **11** (53 mg, 27 µmol) and azidoethanol (100 µL, 121 mg, 1.39 mmol) in anhydr. toluene (4 mL) containing 4Å MS (140 mg) was stirred under Ar for 30 min. After cooling to -10 °C, TMSOTf (0.3 µL, 0.05 equiv.) was added. The reaction mixture was stirred for 1h at this temperature, then Et_3N (10 µL) was added and the mixture was filtered over a pad of Celite and concentrated to dryness. The residue was purified by column chromatography (Toluene/EtOAc, 100:0 → 85:15) to give azide **12** (41 mg, 20 µmol, 81%) as a white foam. R_f = 0.41 (Toluene/EtOAc 80:20). $[\alpha]^{24}_D$ = + 3.0° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.38-7.15 (m, 51H, Ph, NH), 5.58 (dd, $J_{2,3}$ = 2.8 Hz, $J_{1,2}$ = 1.9 Hz, 1H, H-2_A), 5.17 (bs, 1H, H-2_C), 5.11 (d, $J_{1,2}$ = 2.3 Hz, 1H, H-1_C), 5.09 (bs, 1H, H-1_A), 4.97-4.91 (m, 5H, 2H$_{Bn}$, H-1_B, H-1_D, H-1_E), 4.85-4.35 (m, 20H, 18H$_{Bn}$, H-2_B, H-3_D), 4.04-3.34 (m, 24H, H-2_D, H-2_E, H-3_A, H-3_B, H-3_C, H-3_E, H-4_A, H-4_B, H-4_C, H-4_D, H-4_E, H-5_A, H-5_B, H-5_C, H-5_D, H-5_E, H-$6a_D$, H-$6b_D$, H-$6a_E$, H-$6b_E$, -$OCH_2CH_2N_3$), 2.70-2.52 (m, 4H, 2×CH_{2Lev}), 2.11 (bs, 6H, CH_{3Lev}, H_{Ac}), 1.33, 1.22, 1.18 (d, $J_{5,6}$ = 6.2 Hz, 9H, H-6_A, H-6_B, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 206.1 (C_{Lev}), 171.7 (C_{Lev}), 170.0 (C_{Ac}), 161.9 (C_{NTCA}), 138.8-127.3 (Ph), 99.3 (C-1_A), 98.8, 98.4 (2C, C-1_D, C-1_E), 96.1 (C-1_C), 92.3 (CCl_3), 56.7 (C-2_D), 50.7 (CH_2N_3), 38.1 (CH_{2Lev}), 29.8 (CH_{3Lev}), 28.1 (CH_{2Lev}), 21.1 (C_{Ac}), 18.6, 18.3, 18.1

(3C, C-6_A, C-6_B, C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{111}H_{123}Cl_3N_4O_{26}Na$ $[M+Na]^+$: 2055.7338. Found: 2055.7261.

2-Azidoethyl (3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (13). To a solution of pentasaccharide **12** (1.36 g, 0.67 mmol) in anhydr. pyridine (8.0 mL), cooled to 0 °C and under Ar, were added acetic acid (5.3 mL) and hydrazine hydrate (160 µL, 3.29 mmol, 4.9 equiv.), then the reaction mixture was allowed to warm to room temperature. After 1h30, cold water (25 mL) was added to the reaction mixture, which was then extracted with DCM (3 × 60 mL). The combined organics were dried (Na_2SO_4), filtered, evaporated and co-evaporated twice with Toluene. The residue was purified by column chromatography (Toluene/EtOAc, 92:8 → 85:15) to give alcohol **13** (1.16 g, 0.60 mmol, 90%) as a white foam. R_f = 0.35 (Toluene/EtOAc 80:20). $[\alpha]^{24}_D$ = - 0.9° (*c* 1.0, $CHCl_3$). ^1H NMR ($CDCl_3$) δ 7.42-7.18 (m, 51H, Ph, NH), 5.19 (bs, 1H, H-2_C), 5.15-5.13 (m, 2H, H-1_A, H-1_C), 5.01 (bs, 1H, H-1_B), 4.98-4.43 (m, 23H, 20H_{Bn}, H-1_D, H-1_E, H-2_B), 4.37 (bt, 1H, H-3_D), 4.17 (dd, $J_{2,3}$ = 3.0, $J_{1,2}$ = 1.7 Hz, H-2_A), 4.05-3.35 (m, 24H, H-2_D, H-2_E, H-3_A, H-3_B, H-3_C, H-3_E, H-4_A, H-4_B, H-4_C, H-4_D, H-4_E, H-5_A, H-5_B, H-5_C, H-5_D, H-5_E, H-$6a_D$, H-$6b_D$, H-$6a_E$, H-$6b_E$, -$OCH_2CH_2N_3$), 2.34 (bs, 1H, OH), 2.12 (s, 3H, H_{Ac}), 1.33, 1.26, 1.21 (d, $J_{5,6}$ = 6.2 Hz, 9H, H-6_A, H-6_B, H-6_C). ^{13}C NMR (partial, $CDCl_3$) δ 170.1 (C_{Ac}), 161.9 (C_{NTCA}), 138.9-127.4 (Ph), 101.1 (2C, C-1_A, C-1_B), 98.9, 98.4 (2C, C-1_D, C-1_E), 96.1 (C-1_C), 92.4 (CCl_3), 56.5 (C-2_D), 50.8 (CH_2N_3), 21.1 (C_{Ac}), 18.7, 18.3, 18.2 (3C, C-6_A, C-6_B, C-6_C). HRMS (ESI^+) *m/z* calcd for $C_{106}H_{117}Cl_3N_4O_{24}Na$ $[M+Na]^+$: 1957.7021. Found: 1957.7002.

2-Azidoethyl (3,4-di-*O*-benzyl-2-*O*-levulinoyl–α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (14). A solution of acceptor **13** (81 mg, 42 µmol, 1.2 equiv.) and donor **11** (74 mg, 35µmol, 1.0 equiv.) in anhydr. toluene (2 mL) containing 4Å MS (100 mg), under Ar was stirred at room temperature for 15 min. After cooling to -10 °C for 15 min, TMSOTf (1.3 µL, 0.2 equiv.) was added. The reaction mixture was stirred at 0 °C for 1 h. Et_3N (10 µL) was added and the mixture was filtered over a pad of Celite and concentrated to dryness. The residue was purified by column chromatography (Toluene/EtOAc, 100:0 → 85:15) to give the expected decasaccharide **14** (111 mg, 29 µmol, 82%) as a white foam. R_f = 0.27 (Toluene/EtOAc 86:14). $[\alpha]^{24}_D$ = + 4.9° (*c* 1.0, $CHCl_3$). ^1H NMR ($CDCl_3$) δ 7.35-6.95 (m, 102H, Ph, NH), 5.56 (bs, 1H, H-2_A), 5.12-5.07 (m, 5H, H-$1_{A'}$, H-1_C, H-$1_{C'}$, H-2_C, H-$2_{C'}$), 5.03 (br s, 1H, H-1_A), 4.96-4.17 (m, 51H, 40H_{Bn}, H-1_B, H-$1_{B'}$, H-1_D, H-$1_{D'}$, H-1_E, H-$1_{E'}$, H-$2_{A'}$, H-2_B, H-$2_{B'}$, H-3_D, H-$3_{D'}$), 4.00-3.31 (m, 44H, H-2_D, H-$2_{D'}$, H-2_E, H-$2_{E'}$, H-3_A, H-$3_{A'}$, H-3_B, H-$3_{B'}$, H-3_C, H-$3_{C'}$, H-3_E, H-$3_{E'}$, H-4_A, H-$4_{A'}$, H-4_B, H-$4_{B'}$, H-4_C, H-$4_{C'}$, H-4_D, H-$4_{D'}$, H-4_E, H-$4_{E'}$, H-5_A, H-$5_{A'}$, H-5_B, H-$5_{B'}$, H-5_C, H-

$5_{C'}$, H-5_D, H-$5_{D'}$, H-5_E, H-$5_{E'}$, H-$6a_D$, H-$6a_{D'}$, H-$6b_D$, H-$6b_{D'}$, H-$6a_E$, H-$6a_{E'}$, H-$6b_E$, H-$6b_{E'}$, -$OCH_2CH_2N_3$), 2.66-2.50 (m, 4H, 2×CH_{2Lev}), 2.09 (s 3H, CH_{3Lev}), 2.07 (s, 6H, H_{Ac}), 1.32-1.11 (m, 18H, H-6_A, H-6_B, H-6_C, H-$6_{A'}$, H-$6_{B'}$, H-$6_{C'}$). ^{13}C NMR (partial, $CDCl_3$) δ 206.1 (C_{Lev}), 171.8 (C_{Lev}), 170.1 (2C, C_{Ac}), 161.9 (C_{NTCA}), 161.4 (C_{NTCA}), 139.1-127.2 (Ph), 100.2 (2C, C-1_B, C-$1_{B'}$), 99.5 (C-1_A), 96.3 (2C, C-1_C, C-$1_{C'}$), 92.7 (CCl_3), 92.4 (CCl_3), 58.7 (C-2_D), 56.9 (C-$2_{D'}$), 50.8 (CH_2N_3), 38.2 (CH_{2Lev}), 29.8 (CH_{3Lev}), 28.2 (CH_{2Lev}), 21.1 (2C, C_{Ac}), 18.8-18.1 (6C, C-6_A, C-6_B, C-6_C, C-$6_{A'}$, C-$6_{B'}$, C-$6_{C'}$). HRMS (MALDI$^+$) *m/z* calcd for $C_{215}H_{235}Cl_6N_5O_{49}Na$ $[M+Na]^+$: 3903.4080. Found: 3803.3596, $C_{215}H_{235}Cl_6N_5O_{49}K$ $[M+K]^+$: 3919.5012. Found: 3919.4165.

2-Azidoethyl (3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (15). The fully protected **14** (94 mg, 24 µmol, 1.0 equiv.) was dissolved under Ar in anhydr. pyridine (1.0 mL), cooled to 0 °C, and acetic acid (260 µL) followed by hydrazine hydrate (9.2 mg, 7.0 equiv.) were added. The reaction mixture was allowed to warm to room temperature and stirred for 1 h. Cold water (10 mL) was added to the reaction mixture, which was then extracted with DCM (3 × 10 mL). The combined organics were dried (Na_2SO_4), filtered, evaporated and co-evaporated twice with toluene. The residue was purified by column chromatography (Toluene/EtOAc, 100:0 → 85:15) to give alcohol **15** (81 mg, 22 µmol, 89%) as a white foam. R_f = 0.39 (Toluene/EtOAc 82:18). $[\alpha]^{24}_D$ = + 4.0° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.44-7.01 (m, 102H, Ph, NH), 5.21-5.16 (m, 6H, H-1_A, H-$1_{A'}$, H-1_C, H-$1_{C'}$, H-2_C, H-$2_{C'}$), 5.07-4.21 (m, 52H, 40H_{Bn}, H-1_B, H-$1_{B'}$, H-1_D, H-$1_{D'}$, H-1_E, H-$1_{E'}$, H-2_A, H-$2_{A'}$, H-2_B, H-$2_{B'}$, H-3_D, H-$3_{D'}$), 4.07-3.38 (m, 44H, H-2_D, H-$2_{D'}$, H-2_E, H-$2_{E'}$, H-3_A, H-$3_{A'}$, H-3_B, H-$3_{B'}$, H-3_C, H-$3_{C'}$, H-3_E, H-$3_{E'}$, H-4_A, H-$4_{A'}$, H-4_B, H-$4_{B'}$, H-4_C, H-$4_{C'}$, H-4_D, H-$4_{D'}$, H-4_E, H-$4_{E'}$, H-5_A, H-$5_{A'}$, H-5_B, H-$5_{B'}$, H-5_C, H-$5_{C'}$, H-5_D, H-$5_{D'}$, H-5_E, H-$5_{E'}$, H-$6a_D$, H-$6a_{D'}$, H-$6b_D$, H-$6b_{D'}$, H-$6a_E$, H-$6a_{E'}$, H-$6b_E$, H-$6b_{E'}$, -$OCH_2CH_2N_3$), 2.15 (s, 6H, H_{Ac}), 1.40-1.20 (m, 18H, H-6_A, H-6_B, H-6_C, H-$6_{A'}$, H-$6_{B'}$, H-$6_{C'}$). ^{13}C NMR (partial, $CDCl_3$) δ 170.0 (2C, C_{Ac}), 161.9 (C_{NTCA}), 161.3 (C_{NTCA}), 139.1-127.2 (Ph), 101.1 (2C, C-1_A, C-$1_{A'}$), 96.3 (2C, C-1_C, C-$1_{C'}$), 100.2 (2C, C-1_B, C-$1_{B'}$), 98.9, 98.5 (4C, C-1_D, C-$1_{D'}$, C-1_E, C-$1_{E'}$), 92.7 (CCl_3), 92.4 (CCl_3), 58.7 (C-2_D), 56.8 (C-$2_{D'}$), 50.7 (CH_2N_3), 21.1 (2C, C_{Ac}), 18.8-18.1 (6C, C-6_A, C-6_B, C-6_C, C-$6_{A'}$, C-$6_{B'}$, C-$6_{C'}$). HRMS (MALDI$^+$) *m/z* calcd for $C_{210}H_{229}Cl_6N_5O_{47}Na$ $[M+Na]^+$: 3806.3745. Found: 3806.0535, $C_{210}H_{229}Cl_6N_5O_{47}K$ $[M+K]^+$: 3822.3484. Found: 3822.0601.

2-Azidoethyl (3,4-di-*O*-benzyl-2-*O*-levulinoyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-

***O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-(2-*O*-acetyl-α-L-rhamnopyranosyl)-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (16).** To a solution of acceptor **15** (103 mg, 27 μmol, 1.0 equiv.) and donor **11** (87 mg, 41 μmol, 1.5 equiv.) in anhydr. toluene (1.5 mL) containing 4Å MS (160 mg), under Ar at -30 °C, was added TMSOTf (1.0 μL, 5.5 μmol, 0.2 equiv.). The reaction mixture was stirred at -30 °C for 1 h, then at room temperature for 15 min. Et_3N was added and the mixture was filtered over a pad of Celite and concentrated to dryness. The residue was purified by flash chromatography (Toluene/EtOAc, 100:0 → 85:15) to give pentadecasaccharide **16** (121 mg, 21 μmol, 78%) as a white foam. R_f = 0.51 (Toluene/EtOAc 85:15). $[\alpha]^{24}_D$ = + 3.5° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.40-7.04 (m, 153H, Ph, NH), 5.54 (bs, 1H, H-2_A), 5.14-5.00 (m, 9H, H-1_A, H-$1_{A'}$, H-$1_{A''}$, H-1_C, H-$1_{C'}$, H-$1_{C''}$, H-2_C, H-$2_{C'}$, H-$2_{C''}$), 4.93-4.12 (m, 75H, 60H_{Bn}, H-1_B, H-$1_{B'}$, H-$1_{B''}$, H-1_D, H-$1_{D'}$, H-$1_{D''}$, H-1_E, H-$1_{E'}$, H-$1_{E''}$, H-2_B, H-$2_{B'}$, H-$2_{B''}$, H-3_D, H-$3_{D'}$, H-3_D''), 4.00-3.27 (m, 64H, H-2_D, H-$2_{D'}$, H-$2_{D''}$, H-2_E, H-$2_{E'}$, H-$2_{E''}$, H-3_A, H-$3_{A'}$, H-$3_{A''}$, H-3_B, H-$3_{B'}$, H-$3_{B''}$, H-3_C, H-$3_{C'}$, H-$3_{C''}$, H-3_E, H-$3_{E'}$, H-$3_{E''}$, H-4_A, H-$4_{A'}$, H-$4_{A''}$, H-4_B, H-$4_{B'}$, H-$4_{B''}$, H-4_C, H-$4_{C'}$, H-$4_{C''}$, H-4_D, H-$4_{D'}$, H-$4_{D''}$, H-4_E, H-$4_{E'}$, H-$4_{E''}$, H-5_A, H-$5_{A'}$, H-$5_{A''}$, H-5_B, H-$5_{B'}$, H-$5_{B''}$, H-5_C, H-$5_{C'}$, H-$5_{C''}$, H-5_D, H-$5_{D'}$, H-$5_{D''}$, H-5_E, H-$5_{E'}$, H-$5_{E''}$, H-$6a_D$, H-$6a_{D'}$, H-$6a_{D''}$, H-$6b_D$, H-$6b_{D'}$, H-$6b_{D''}$, H-$6a_E$, H-$6a_{E'}$, H-$6a_{E''}$, H-$6b_E$, H-$6b_{E'}$, H-$6b_{E''}$, -$OCH_2CH_2N_3$), 2.65-2.50 (m, 4H, 2×CH_{2Lev}), 2.06 (s, 6H, H_{Ac}), 2.04 (s, 3H, H_{Ac}), 1.33-1.16 (m, 27H, H-6_A, H-6_B, H-6_C, H-$6_{A'}$, H-$6_{B'}$, H-$6_{C'}$, H-$6_{A''}$, H-$6_{B''}$, H-$6_{C''}$). ^{13}C NMR (partial, $CDCl_3$) δ 206.1 (C_{Lev}), 171.8 (C_{Lev}), 170.1 (3C, C_{Ac}), 161.9 (C_{NTCA}), 161.4 (C_{NTCA}), 161.3 (C_{NTCA}), 139.1-127.3 (Ph), 92.7 (2C, CCl_3), 92.4 (CCl_3), 69.5 (C-2_A), 58.7 (2C, C-2_D, C-$2_{D'}$), 56.8 (C-$2_{D''}$), 50.7 (CH_2N_3), 38.2 (CH_{2Lev}), 29.9 (CH_{3Lev}), 28.2 (CH_{2Lev}), 21.1 (3C, C_{Ac}), 18.8-18.1 (9C, C-6_A, C-6_B, C-6_C, C-$6_{A'}$, C-$6_{B'}$, C-$6_{C'}$, C-$6_{A''}$, C-$6_{B''}$, C-$6_{C''}$). HRMS (MALDI$^+$) *m/z* calcd for $C_{319}H_{347}Cl_9N_6O_{72}Na$ $[M+Na]^+$: 5752.0806. Found: 5752.4351, $C_{319}H_{347}Cl_9N_6O_{72}K$ $[M+K]^+$: 5767.0508. Found: 5767.5322.

2-Azidoethyl (3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-α-L-rhamnopyranosyl-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-α-L-rhamnopyranosyl-(1→3)-(4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→2)-(3,4-di-*O*-benzyl-α-L-rhamnopyranosyl)-(1→3)-[2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl)-(1→4)]-α-L-rhamnopyranosyl-(1→3)-4,6-di-*O*-benzyl-2-deoxy-2-trichloroacetamido-β-D-glucopyranoside (17). To a solution of the fully protected pentadecasaccharide **16** (105 mg, 18 μmol, 1.0 equiv.) in MeOH/DCM (4:3, 7 mL) was added methanolic sodium methoxide (25% w/w, 200 μL) and the mixture was stirred at 60 °C for 3 h, and then at room temperature overnight. Dowex 50Wx8-200 (H^+ form) was added. The suspension was filtered over a pad of Celite, and the filtrate was concentrated to dryness. The residue was purified by flash

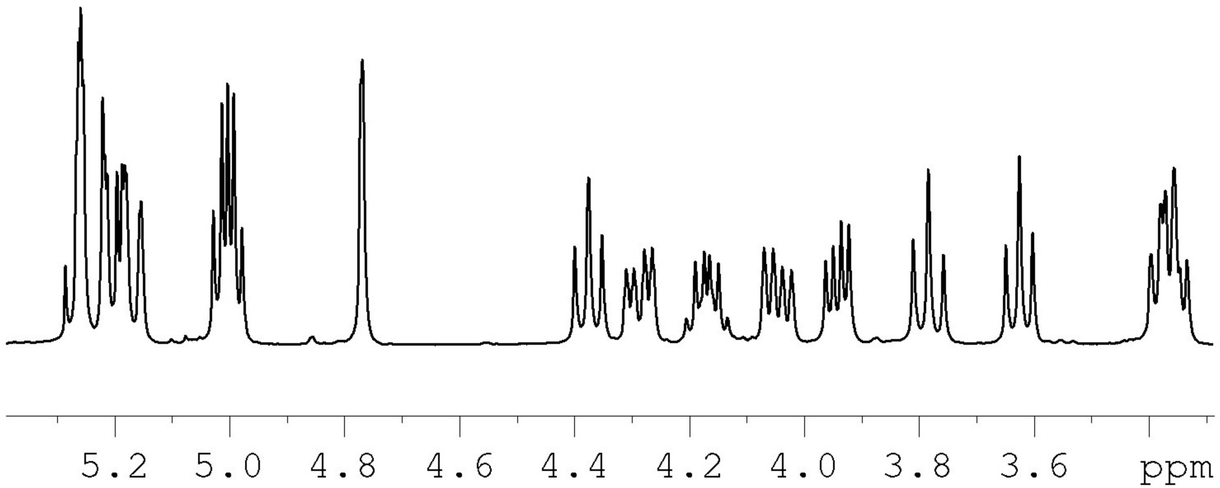
chromatography (Toluene/EtOAc 100:0 → 81:15) to give tetraol **17** (73 mg, 13.3 μmol, 74%) as a white foam. R_f = 0.34 (Toluene/EtOAc 85:15). $[\alpha]^{24}_D$ = - 7.2° (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.43-6.92 (m, 153H, Ph, NH), 5.54 (bs, 1H, H-2_A), 5.19-4.12 (m, 81H, 60H_{Bn}, H-1_A, H-$1_{A'}$, H-$1_{A''}$, H-1_B, H-$1_{B'}$, H-$1_{B''}$, H-1_C, H-$1_{C'}$, H-$1_{C''}$, H-1_D, H-$1_{D'}$, H-$1_{D''}$, H-1_E, H-$1_{E'}$, H-$1_{E''}$, H-2_C, H-$2_{C'}$, H-$2_{C''}$, H-3_D, H-$3_{D'}$, H-$3_{D''}$), 4.00-3.27 (m, 67H, H-2_B, H-$2_{B'}$, H-$2_{B''}$, H-2_D, H-$2_{D'}$, H-$2_{D''}$, H-2_E, H-$2_{E'}$, H-$2_{E''}$, H-3_A, H-$3_{A'}$, H-$3_{A''}$, H-3_B, H-$3_{B'}$, H-$3_{B''}$, H-3_C, H-$3_{C'}$, H-$3_{C''}$, H-3_E, H-$3_{E'}$, H-$3_{E''}$, H-4_A, H-$4_{A'}$, H-$4_{A''}$, H-4_B, H-$4_{B'}$, H-$4_{B''}$, H-4_C, H-$4_{C'}$, H-$4_{C''}$, H-4_D, H-$4_{D'}$, H-$4_{D''}$, H-4_E, H-$4_{E'}$, H-$4_{E''}$, H-5_A, H-$5_{A'}$, H-$5_{A''}$, H-5_B, H-$5_{B'}$, H-$5_{B''}$, H-5_C, H-$5_{C'}$, H-$5_{C''}$, H-5_D, H-$5_{D'}$, H-$5_{D''}$, H-5_E, H-$5_{E'}$, H-$5_{E''}$, H-$6a_D$, H-$6a_{D'}$, H-$6a_{D''}$, H-$6b_D$, H-$6b_{D'}$, H-$6b_{D''}$, H-$6a_E$, H-$6a_{E'}$, H-$6a_{E''}$, H-$6b_E$, H-$6b_{E'}$, H-$6b_{E''}$, -$OCH_2CH_2N_3$), 1.32-1.03 (m, 27H, H-6_A, H-6_B, H-6_C, H-$6_{A'}$, H-$6_{B'}$, H-$6_{C'}$, H-$6_{A''}$, H-$6_{B''}$, H-$6_{C''}$). ^{13}C NMR (partial, $CDCl_3$) δ 162.0 (C_{NTCA}), 161.9 (2C, C_{NTCA}), 139.0-127.3 (Ph), 92.6 (2C, CCl_3), 92.5 (CCl_3), 50.9 (CH_2N_3), 18.6-18.3 (9C, C-6_A, C-6_B, C-6_C, C-$6_{A'}$, C-$6_{B'}$, C-$6_{C'}$, C-$6_{A''}$, C-$6_{B''}$, C-$6_{C''}$). HRMS (MALDI$^+$) *m/z* calcd for $C_{308}H_{335}Cl_9N_6O_{67}Na$ $[M+Na]^+$: 5527.0088. Found: 5527.5210.

2-Aminoethyl α-L-rhamnopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→3)-[α-D-glucopyranosyl-(1→4)]-α-L-rhamnopyranosyl-(1→3)-2-acetamido-2-deoxy-β-D-glucopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→3)-[α-D-glucopyranosyl-(1→4)]-α-L-rhamnopyranosyl-(1→3)-2-acetamido-2-deoxy-β-D-glucopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→3)-[α-D-glucopyranosyl-(1→4)]-α-L-rhamnopyranosyl-(1→3)-2-acetamido-2-deoxy-β-D-glucopyranoside (1). To a degassed solution of pentadecasaccharide **17** (53 mg, 9.6 μmol) in *t*BuOH/DCM/water (7/1/1, 5.0 mL), was added Pd/C (52 mg) and the suspension was stirred for 4 days under a hydrogen atmosphere. After this time, RP-HPLC analysis of the reaction mixture revealed a peak with a retention time corresponding to that of the target pentadecasaccharide **1**, albeit as a minor peak. The suspension was filtered and the crude material was dissolved in *t*BuOH/water (1/1, 5.0 mL). Pd/C (23 mg) was added and the suspension was stirred for a day under a hydrogen atmosphere. After this time, RP-HPLC analysis of the reaction mixture revealed the absence of any major progress. $Pd(OH)_2$/C (25 mg) was added and the suspension was stirred for 3 more days, at which time RP-HPLC analysis of the reaction mixture indicated that most of the intermediates had been converted into the target pentadecasaccharide **1**, an observation that was supported by MS analysis. The suspension was filtered over a 0.2 μm filter. Evaporation of the volatiles, freeze-drying and purification of the residue by RP-HPLC (λ = 215 nm) using a Kromasil 5 μm C18 100 Å 10 × 250 mm semi-preparative column eluting with a 0-20% linear gradient of CH_3CN in 0.08% aq. TFA over 20 min at a flow rate of 5.5 mL•min^{-1}, gave the known pentadecasaccharide **1** (12.6 mg, 5.1 μmol, 52%) as a white powder following repeated freeze-drying. NMR data obtained for the fully deprotected aminoethyl glycoside **1** were identical to those of a reference compound prepared by chemical synthesis.[12] In addition, compound **1** had $[\alpha]^{24}_D$ = -10.0° (*c* 1.0, water). HRMS (MALDI$^+$) *m/z* calcd for $C_{98}H_{166}N_4O_{67}Na$ $[M+Na]^+$: 2493.9602. Found: 2493.8005, $C_{98}H_{166}N_4O_{67}K$ $[M+K]^+$: 2509.9343. Found: 2509.7732. RP-HPLC (λ = 215 nm): t_R = 6.40 min.

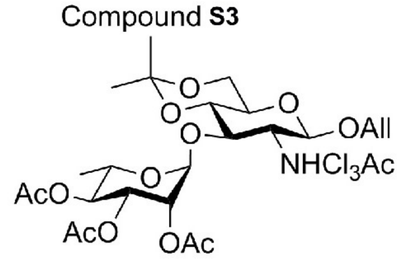
References

1. G. Potocki De Montalk, M. Remaud-Siméon, R. M. Willemot, V. Planchot and P. Monsan, *J. Bacteriol.*, 1999, **181**, 375-381.
2. S. Emond, S. Mondeil, K. Jaziri, I. André, P. Monsan, M. Remaud-Siméon and G. Potocki-Véronèse, *FEMS Microbiol. Lett.*, 2008, **285**, 25-32.
3. E. Champion, M. Remaud-Siméon, L. K. Skov, J. S. Kastrup, M. Gajhede and O. Mirza, *Acta Crystallogr., Sect. D: Biol. Crystallogr.*, 2009, **65**, 1309-1314.
4. J. F. Robyt and T. F. Walseth, *Carbohydr. Res.*, 1979, **68**, 95-111.
5. C. Moulis, A. Arcache, P.-C. Escalier, M. Rinaudo, P. Monsan, M. Remaud-Siméon and G. Potocki-Veronèse, *FEMS Microbiol. Lett.*, 2006, **261**, 203-210.
6. G. Joucla, S. Pizzut, P. Monsan and M. Remaud-Siméon, *FEBS Lett.*, 2006, **580**, 763-768.
7. S. Bozonnet, M. Dols-Laffargue, E. Fabre, S. Pizzut, M. Remaud-Simeon, P. Monsan and R.-M. Willemot, *J. Bacteriol.*, 2002, **184**, 5753-5761.
8. E. Fabre, S. Bozonnet, A. Arcache, R. M. Willernot, M. Vignon, P. Monsan and M. Remaud-Simeon, *J. Bacteriol.*, 2005, **187**, 296-303.
9. J. Boutet, T. H. Kim, C. Guerreiro and L. A. Mulard, *Tetrahedron Lett.*, 2008, **49**, 5339-5342.
10. A. Esswein and R. R. Schmidt, *Liebigs Ann. Chem.*, 1988, 675-678.
11. J. Boutet and L. A. Mulard, *Eur. J. Org. Chem.*, 2008, 5526-5542.
12. F. Bélot, C. Guerreiro, F. Baleux and L. A. Mulard, *Chem. Eur. J.*, 2005, **11**, 1625-1635.

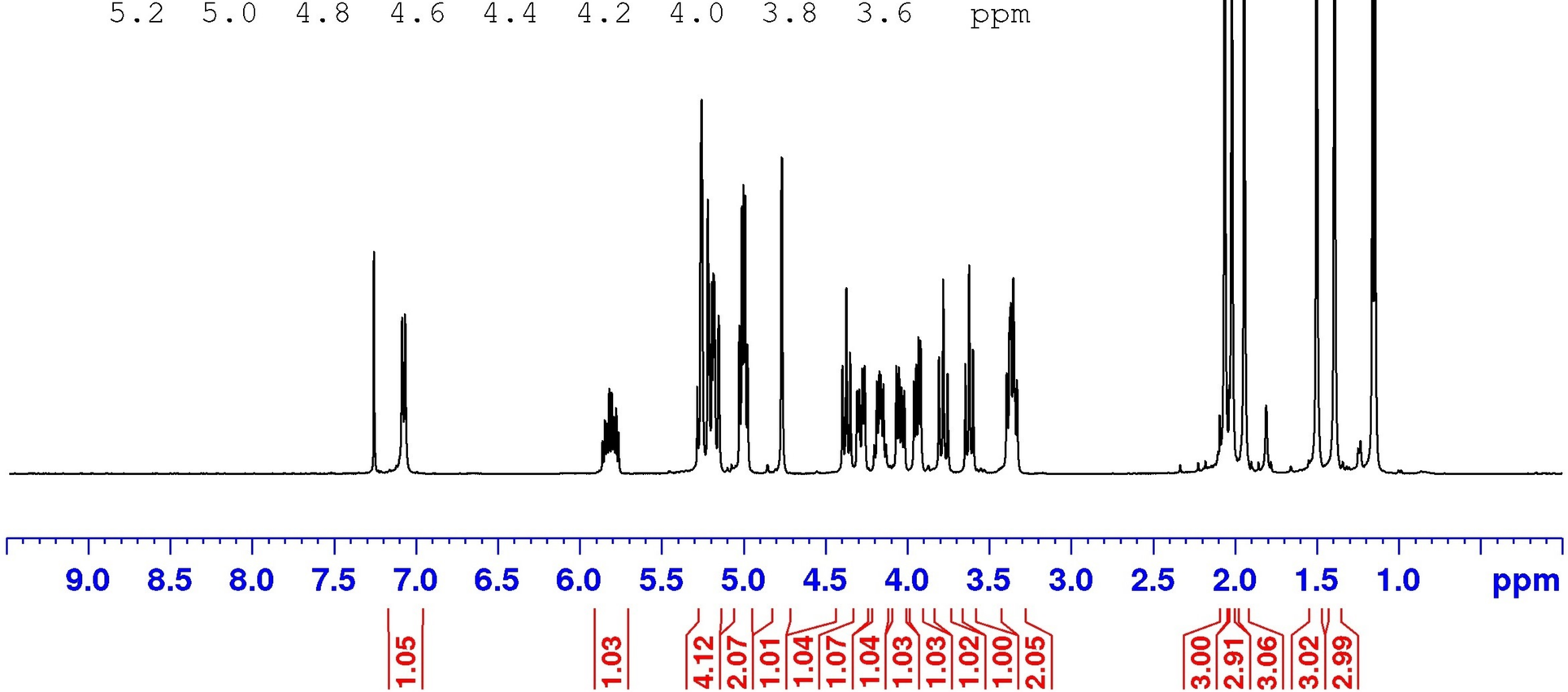
7.26 7.09 7.07 5.26 5.26 5.26 5.22 5.22 5.21 5.20 5.19 5.18 5.18 5.16 5.15 5.03 5.01 5.00 4.99 4.98 4.77 4.40 4.38 4.35 4.26 4.07 3.95 3.94 3.92 3.81 3.78 3.65 3.62 3.60 3.38 3.37 3.36 2.07 2.02 1.95 1.50 1.39 1.16 1.15

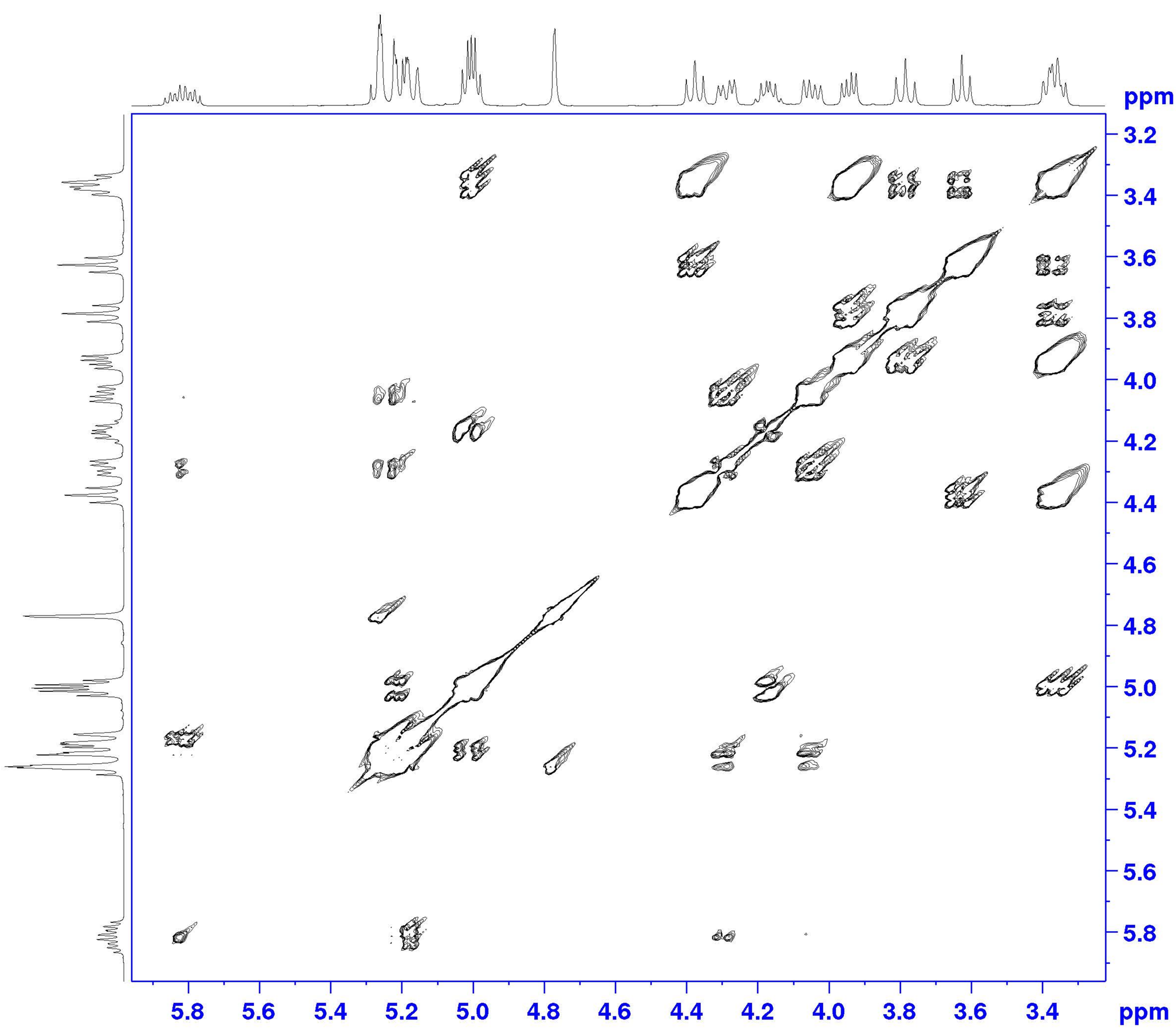


Compound **S3**

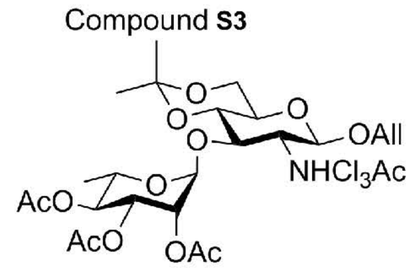



```
F2 - Acquisition Parameters
Date_              20121015
Time                 18.05
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              4006.410 Hz
FIDRES           0.061133 Hz
AQ               8.1788931 sec
RG                     57
DW                124.800 usec
DE                  6.00 usec
TE                 303.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.75 usec
PL1                -6.00 dB
SFO1         400.1318006 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300091 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```





Compound **S3**



F2 - Acquisition Parameters

Date_	20121015
Time	12.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3063.726 Hz
FIDRES	1.495960 Hz
AQ	0.3342336 sec
RG	64
DW	163.200 usec
DE	6.50 usec
TE	303.0 K
D0	0.00000300 sec
D1	1.34557998 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00032640 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1316532 MHz

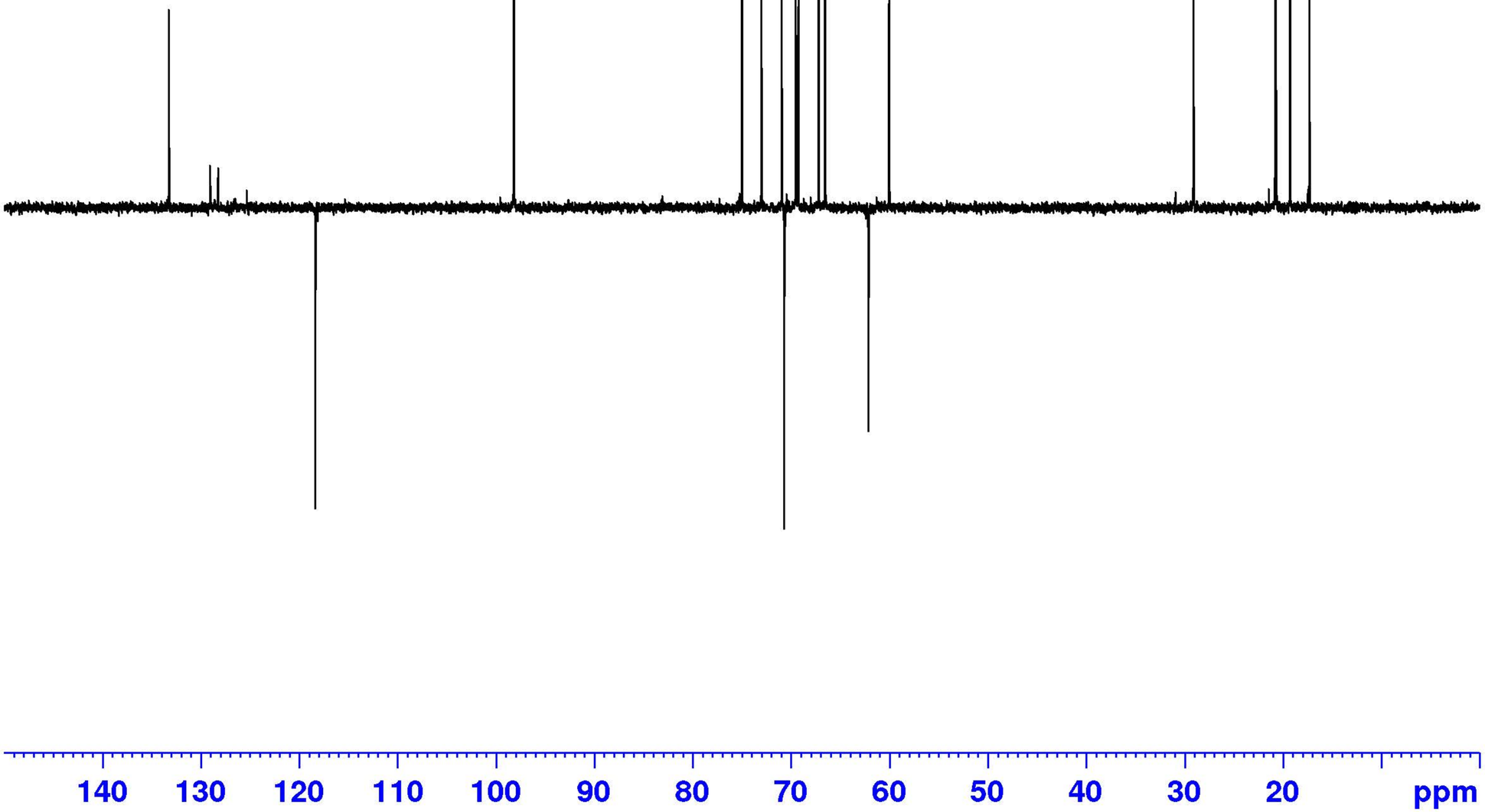
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

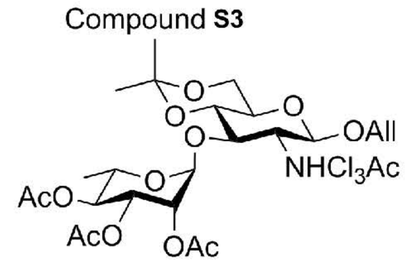
F1 - Acquisition parameters

TD	512
SFO1	400.1317 MHz
FIDRES	5.983839 Hz
SW	7.657 ppm
FnMODE	QF

133.22
118.34
98.17
98.14
74.96
72.98
70.92
70.68
69.49
69.22
67.16
66.50
62.11
59.99
29.07
20.77
20.70
20.68
19.24
17.29



Compound **S3**



F2 - Acquisition Parameters

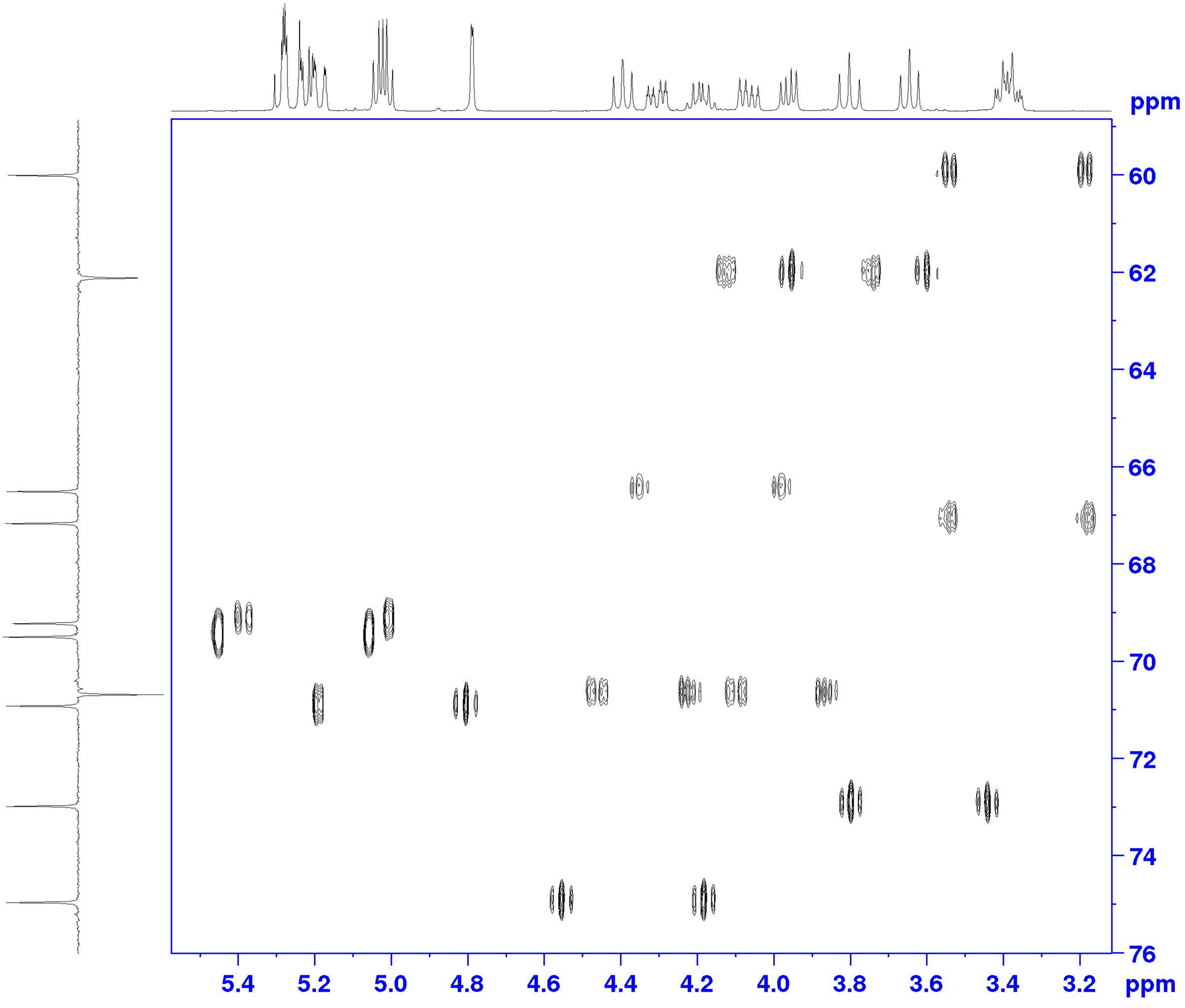
Date_	20121015
Time	13.26
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

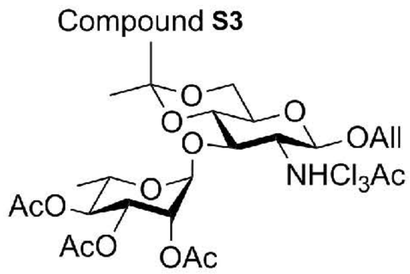
======== CHANNEL f1 ========

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

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

CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
P4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz





F2 - Acquisition Parameters

Date_	20121015
Time	13.29
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpnd
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	3019.324 Hz
FIDRES	1.474279 Hz
AQ	0.3391488 sec
RG	5160.6
DW	165.600 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.47951996 sec
D4	0.00172414 sec
D11	0.03000000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1316387 MHz

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

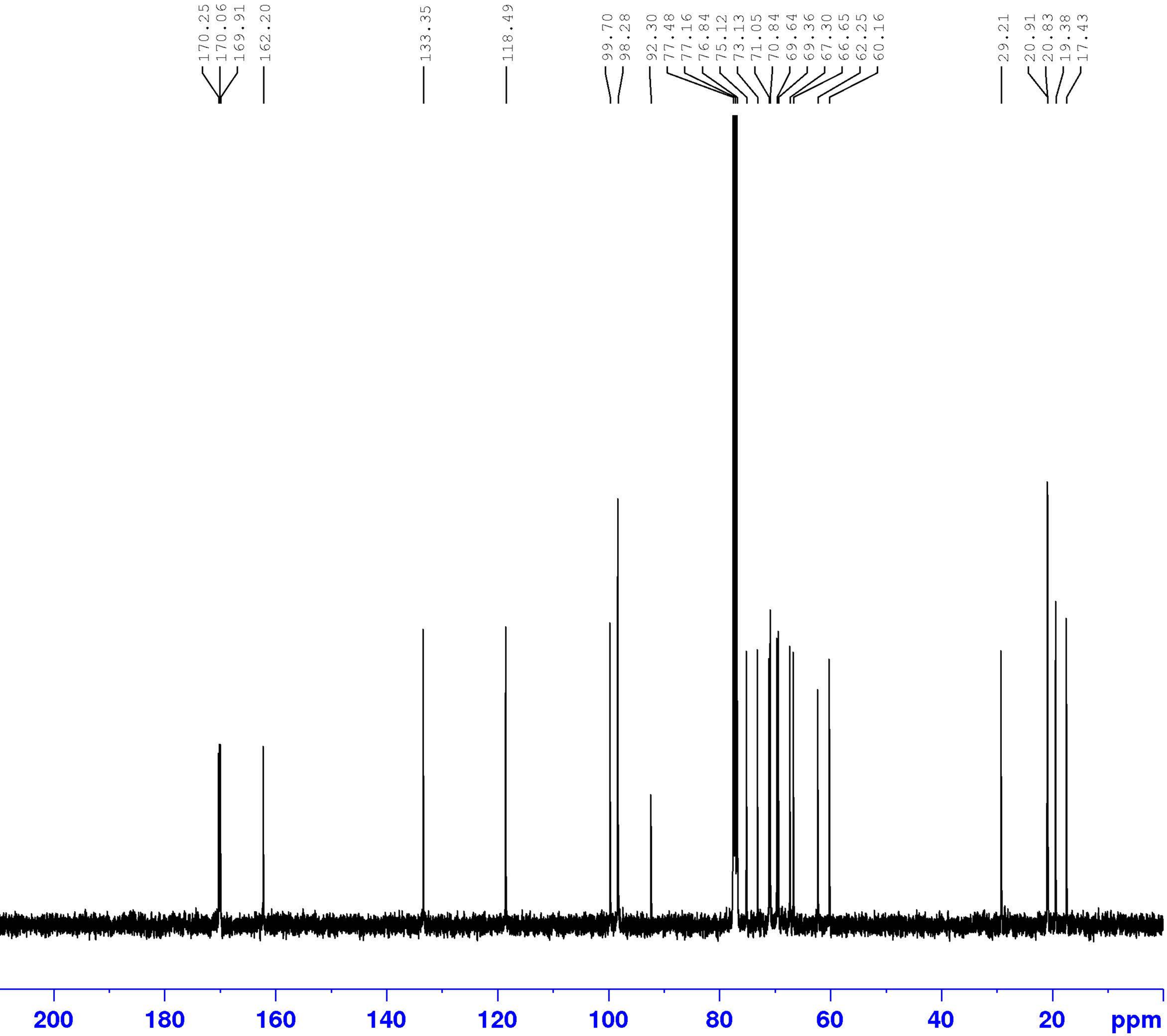
NUC2	13C
P3	5.90 usec
P4	11.80 usec
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz

====== GRADIENT CHANNEL =====

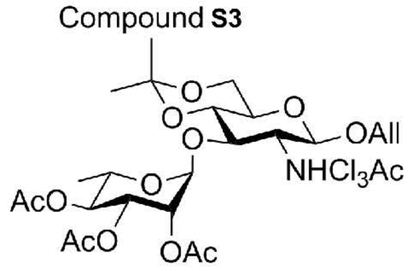
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	256
SFO1	100.6208 MHz
FIDRES	70.749016 Hz
SW	180.000 ppm
FnMODE	Echo-Antiecho



Compound **S3**



F2 - Acquisition Parameters

Date_	20121015
Time	18.50
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1408
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	13004
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

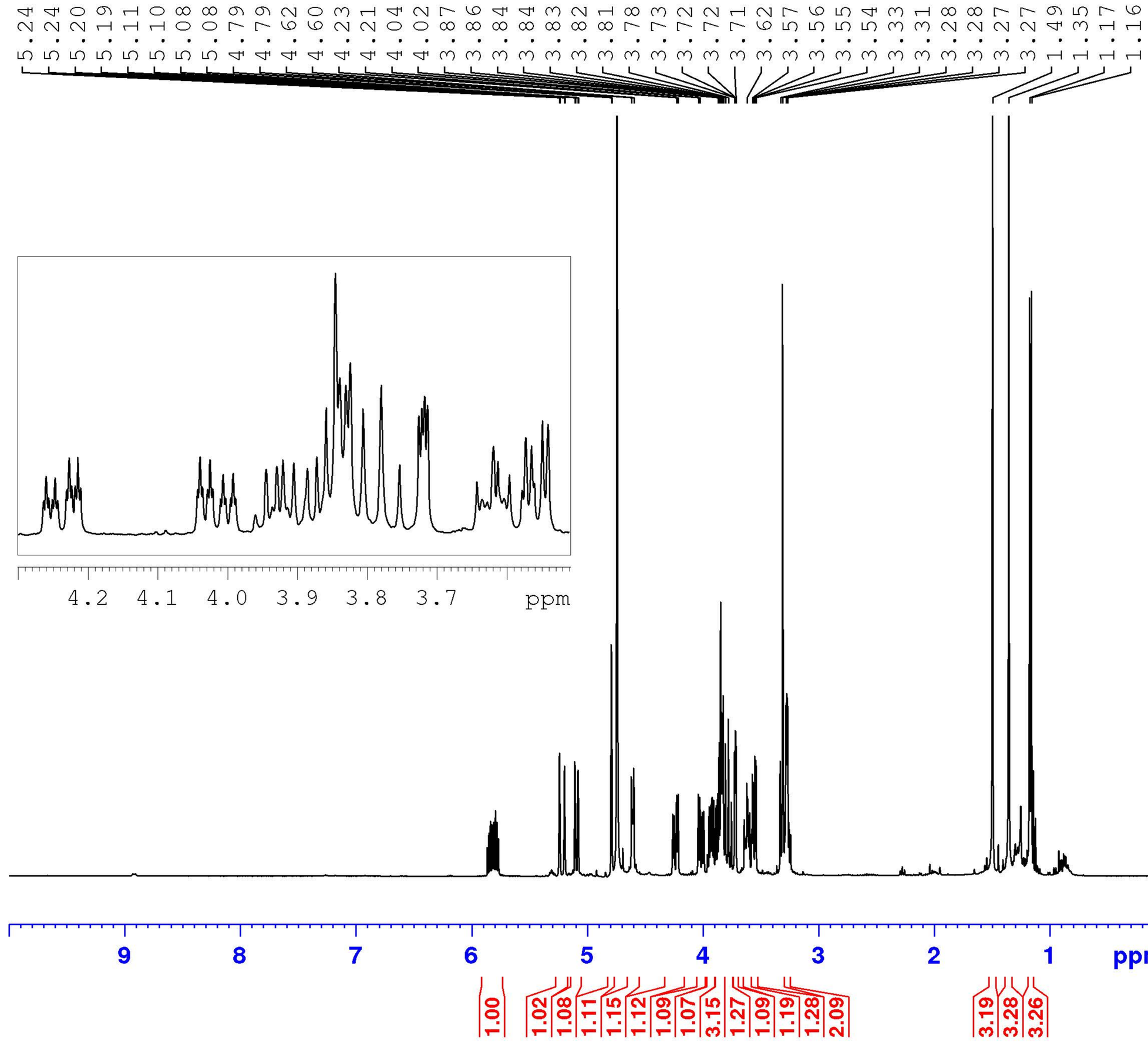
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

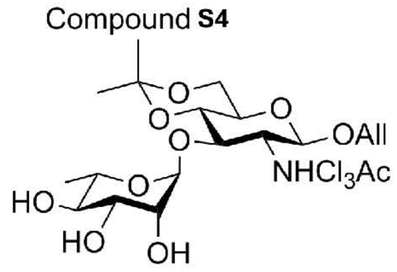
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127547 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Compound **S4**



F2 - Acquisition Parameters

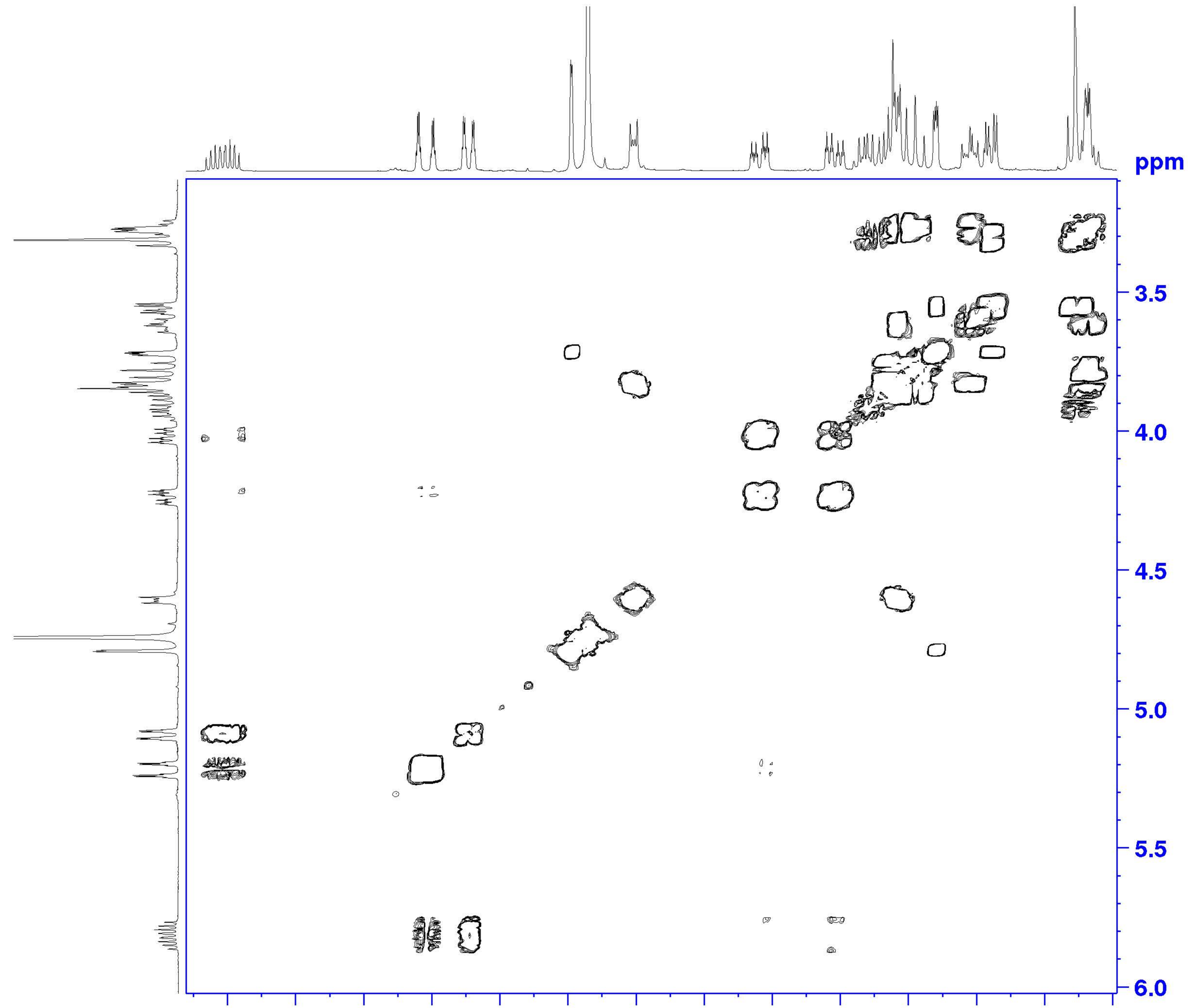
Date_	20101013
Time	11.57
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	45.3
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

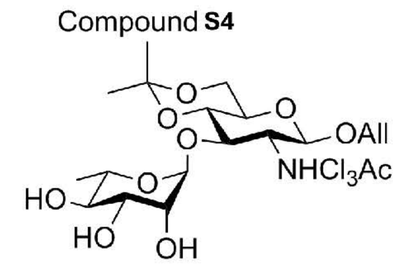
======== CHANNEL f1 ========

NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300227 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





F2 - Acquisition Parameters

Date_	20101013
Time	11.59
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	MeOD
NS	1
DS	8
SWH	2380.952 Hz
FIDRES	1.162574 Hz
AQ	0.4300800 sec
RG	35.9
DW	210.000 usec
DE	6.50 usec
TE	303.0 K
d0	0.00000300 sec
D1	1.24932396 sec
d11	0.03000000 sec
d12	0.00002000 sec
d13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00042000 sec

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

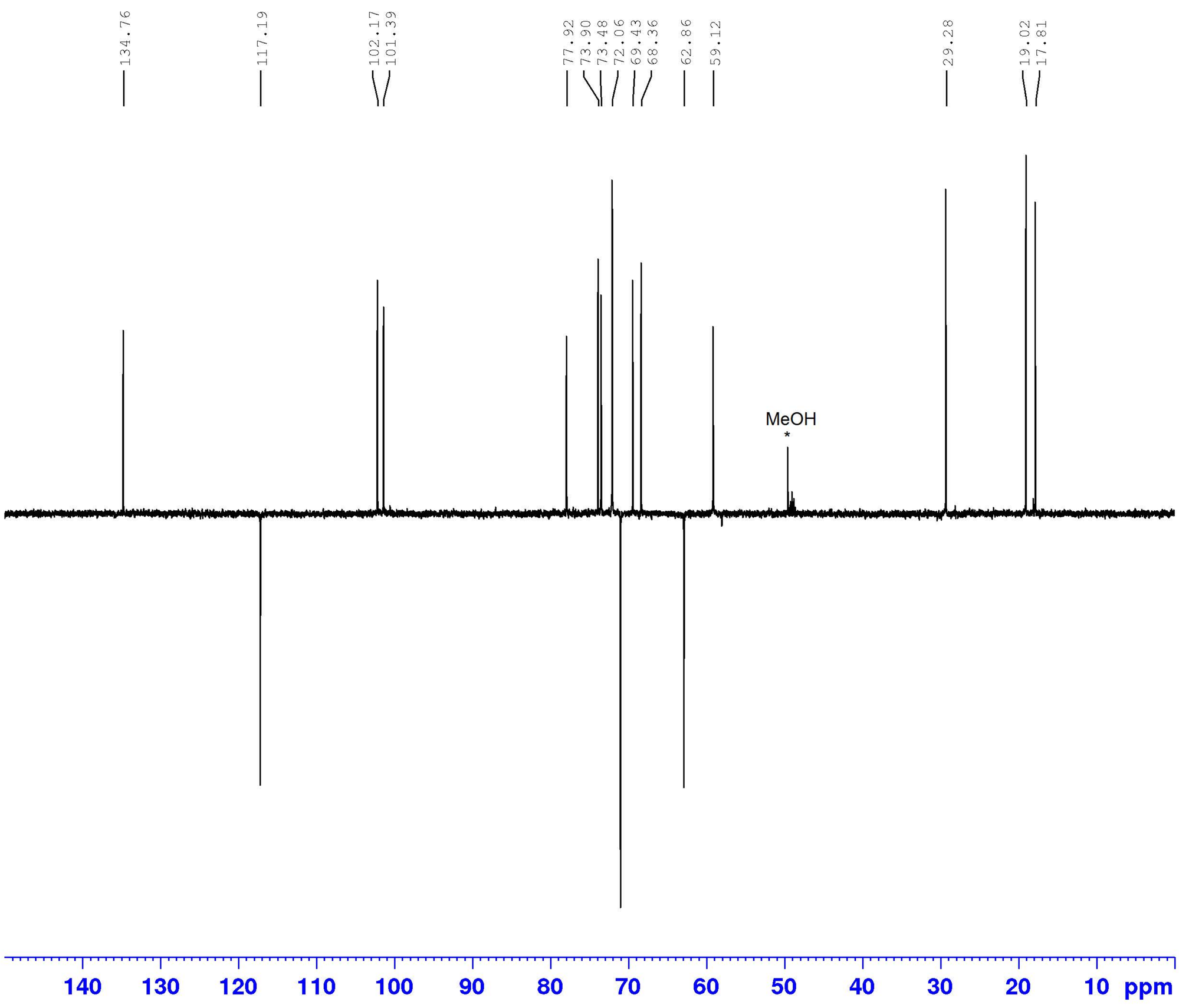
NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1313327 MHz

====== GRADIENT CHANNEL =====

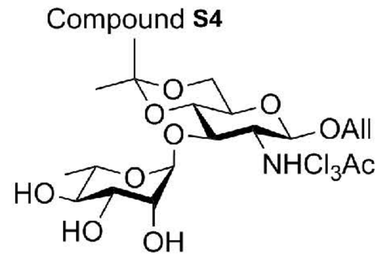
GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	400.1313 MHz
FIDRES	18.601191 Hz
SW	5.950 ppm
FnMODE	QF



Compound **S4**



F2 - Acquisition Parameters

Date_	20101013
Time	23.25
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
d2	0.00344828 sec
d12	0.00002000 sec
DELTA	0.00000751 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

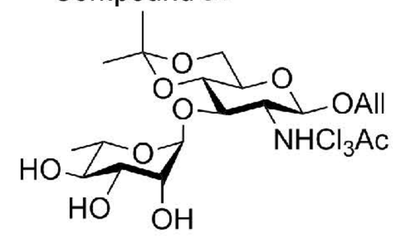
======== CHANNEL f2 ========

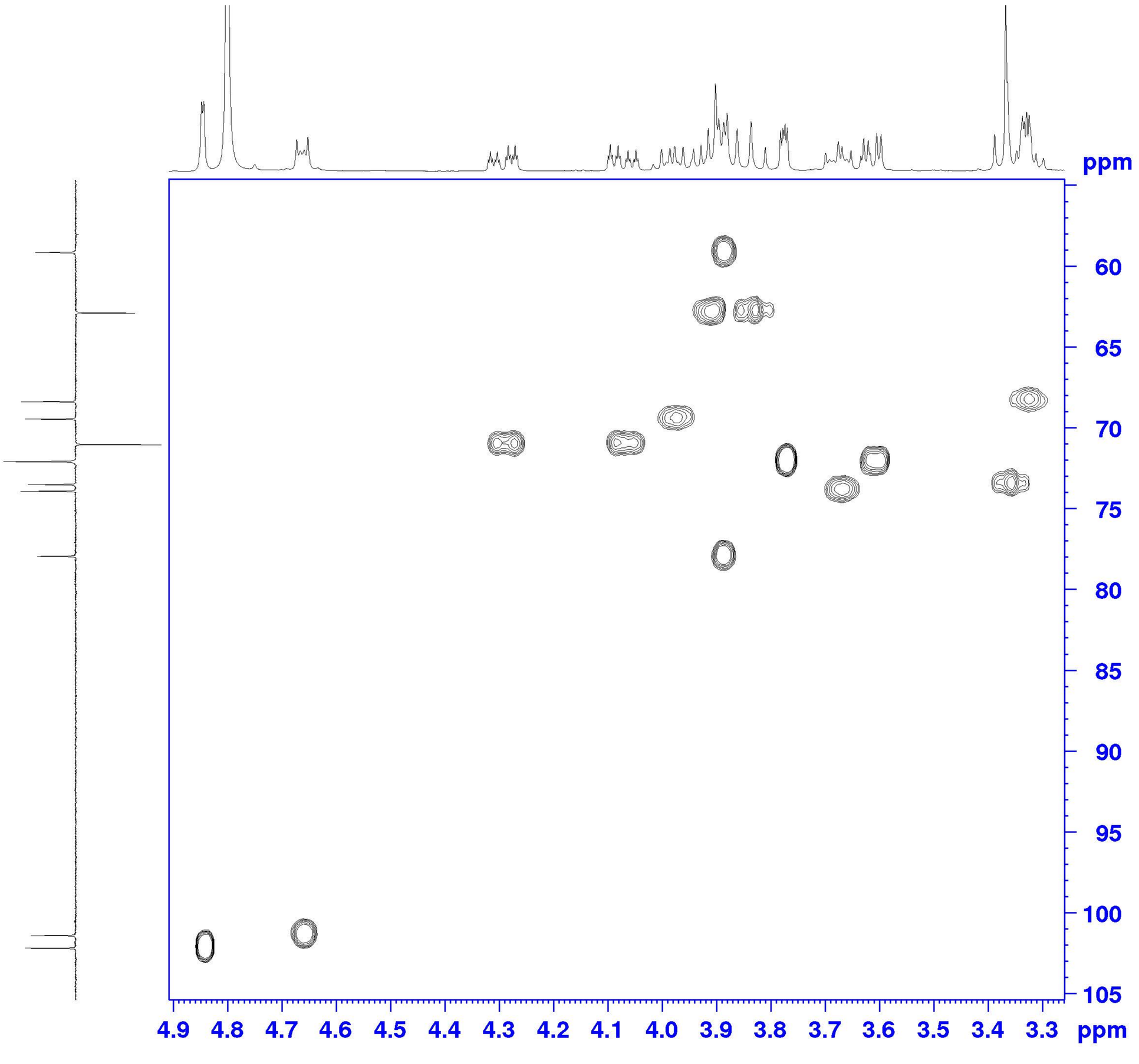
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
p4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

F1 - Acquisition parameters

TD	128
SFO1	400.1316 MHz
FIDRES	7.812500 Hz
SW	2.499 ppm
FnMODE	QF

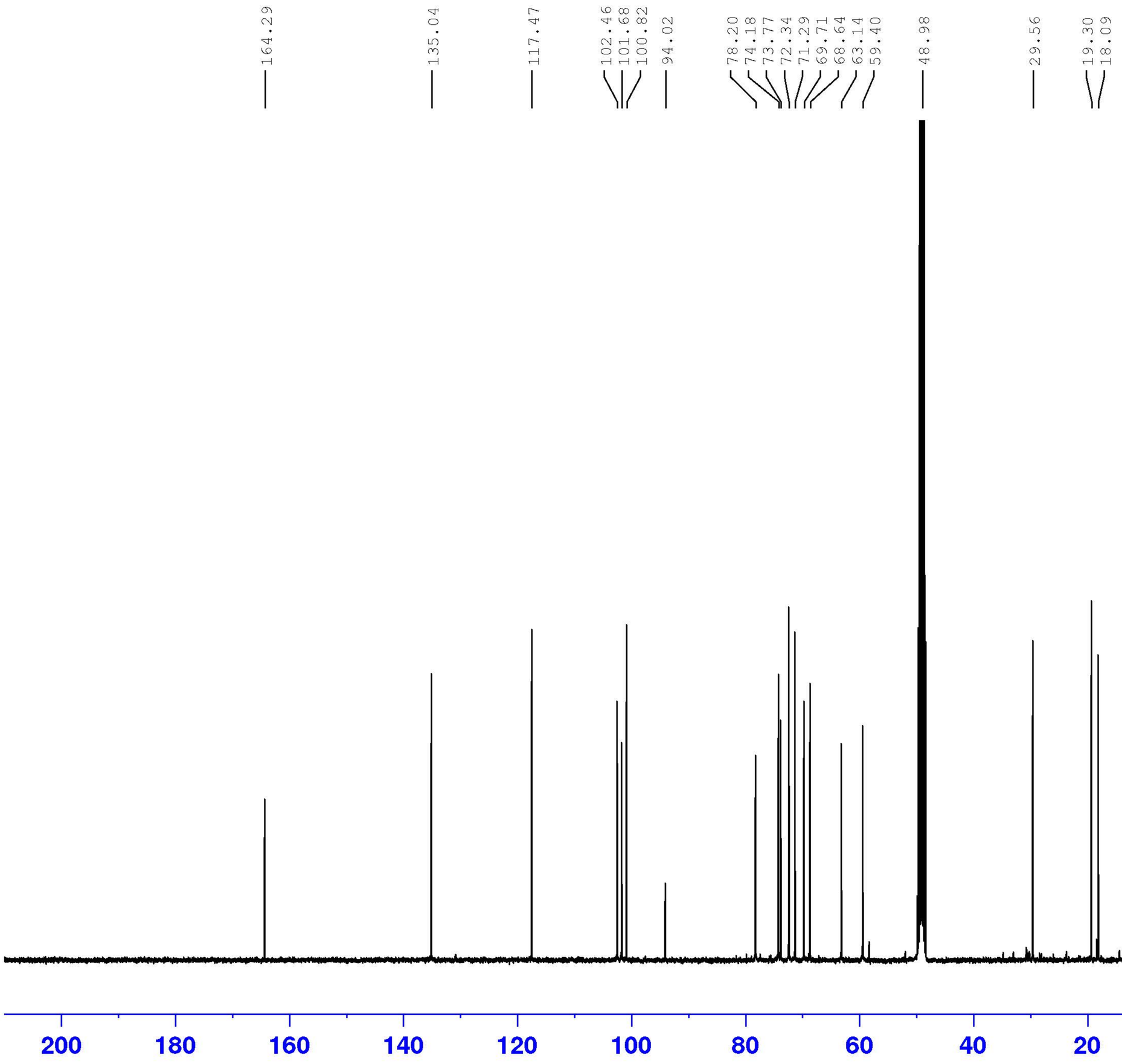
Compound **S4**



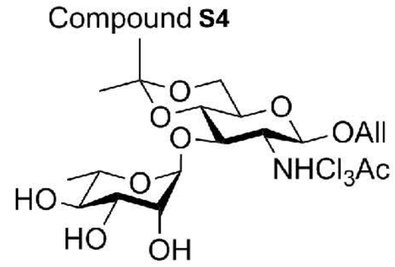


F2 - Acquisition Parameters

Date_	20101013
Time	23.28
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	MeOD
NS	2
DS	16
SWH	2380.952 Hz
FIDRES	1.162574 Hz
AQ	0.4300800 sec
RG	10321.3
DW	210.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.26161301 sec
d4	0.00172414 sec
d11	0.03000000 sec
D16	0.00020000 sec
DELTA	0.00121950 sec
DELTA1	0.00046614 sec
DELTA2	0.00147414 sec
IN0	0.00002925 sec
ST1CNT	0
ZGOPTNS	
========	CHANNEL f1 ========
NUC1	1H
P1	6.75 usec
p2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1313327 MHz
========	CHANNEL f2 ========
CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz



Compound **S4**



F2 - Acquisition Parameters

Date_	20101014	
Time	3.23	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	MeOD	
NS	4096	
DS	4	
SWH	26178.010	Hz
FIDRES	0.399445	Hz
AQ	1.2517376	sec
RG	456.1	
DW	19.100	usec
DE	6.50	usec
TE	303.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	

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

NUC1	13C	
P1	5.90	usec
PL1	-6.00	dB
SFO1	100.6248425	MHz

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

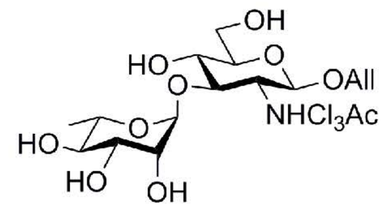
CPDPRG[2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-6.00	dB
PL12	20.00	dB
PL13	20.00	dB
SFO2	400.1316005	MHz

F2 - Processing parameters

SI	32768	
SF	100.6126340	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

5.42 5.42 5.38 5.37 5.35 5.34 5.32 5.32 4.97 4.96 4.85 4.83 4.79 4.41 4.29 4.27 4.05 4.04 4.03 4.01 4.00 3.94 3.92 3.91 3.91 3.90 3.89 3.87 3.86 3.85 3.84 3.83 3.82 3.81 3.79 3.78 3.64 3.61 3.59 3.58 3.53 3.51 3.48 1.31 1.30

Compound **2**



F2 - Acquisition Parameters

Date_	20101127
Time	7.54
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	D2O
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	181
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324710 MHz

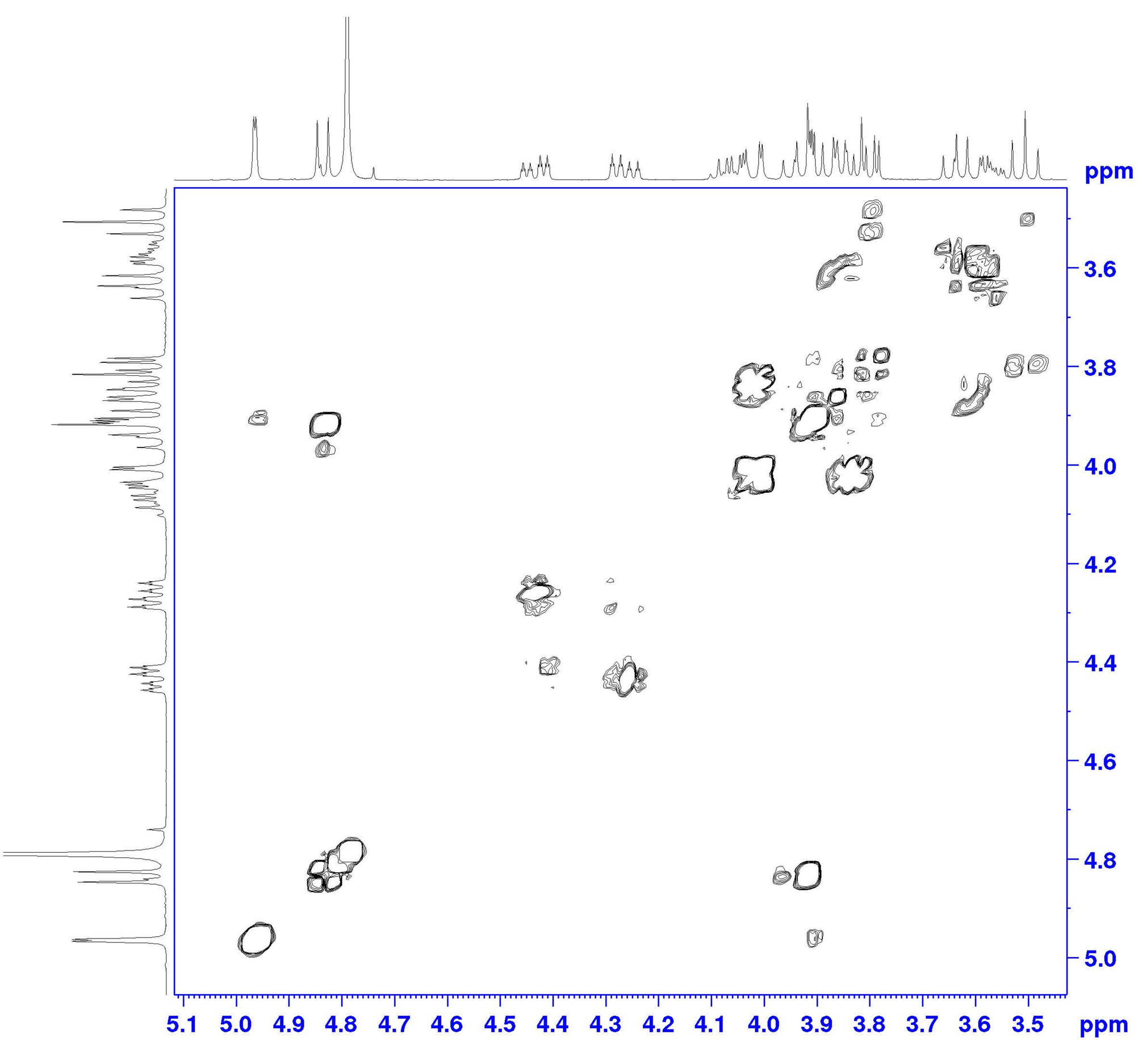
F2 - Processing parameters

SI	32768
SF	400.1299642 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

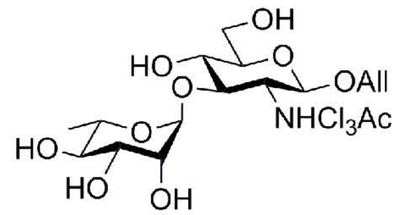
4.4 4.2 4.0 3.8 3.6 ppm

9 8 7 6 5 4 3 2 1 ppm

1.00 2.10 1.05 1.16 1.06 1.08 2.18 2.15 1.95 1.32 1.08 1.09 1.07 3.14



Compound **2**



F2 - Acquisition Parameters

Date_	20101127
Time	7.55
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	D2O
NS	1
DS	8
SWH	2314.815 Hz
FIDRES	1.130281 Hz
AQ	0.4423680 sec
RG	64
DW	216.000 usec
DE	6.50 usec
TE	303.0 K
d0	0.00000300 sec
D1	1.23703599 sec
d11	0.03000000 sec
d12	0.00002000 sec
d13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00043200 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1313888 MHz

====== GRADIENT CHANNEL =====

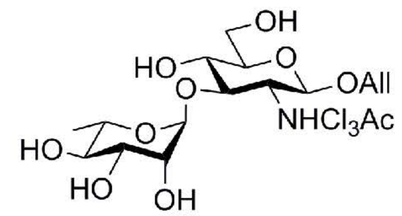
GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	400.1314 MHz
FIDRES	18.084490 Hz
SW	5.785 ppm
FnMODE	QF

133.13
118.75
101.32
99.07
80.81
76.10
71.92
70.83
70.66
70.38
69.01
68.70
60.77
57.41
16.49

Compound **2**



F2 - Acquisition Parameters

Date_	20101127
Time	9.57
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	D2O
NS	2048
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
d2	0.00344828 sec
d12	0.00002000 sec
DELTA	0.00000751 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

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

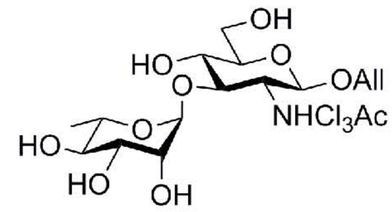
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
p4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

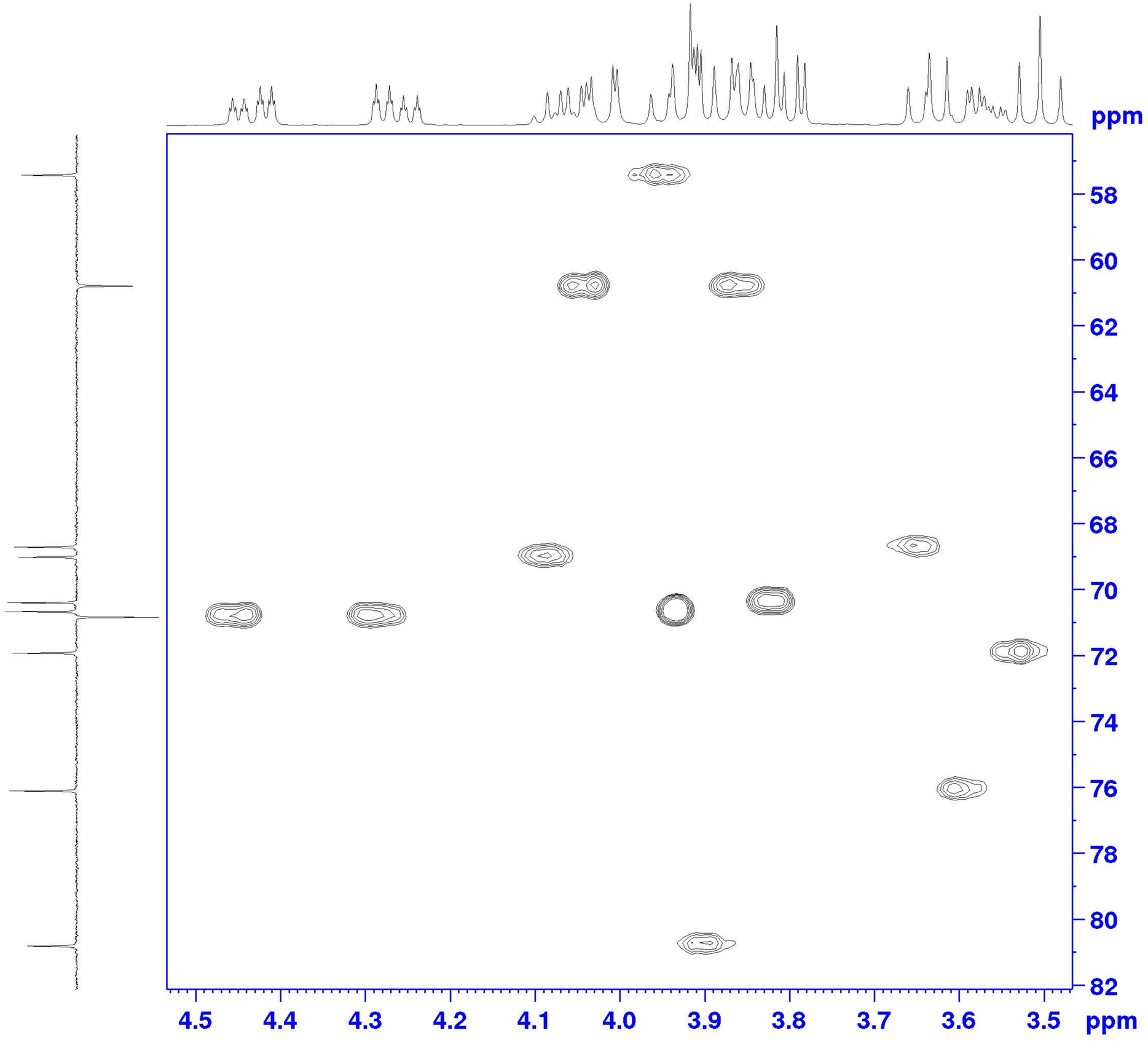
F1 - Acquisition parameters

TD	128
SFO1	400.1316 MHz
FIDRES	7.812500 Hz
SW	2.499 ppm
FnMODE	QF

130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **2**





F2 - Acquisition Parameters

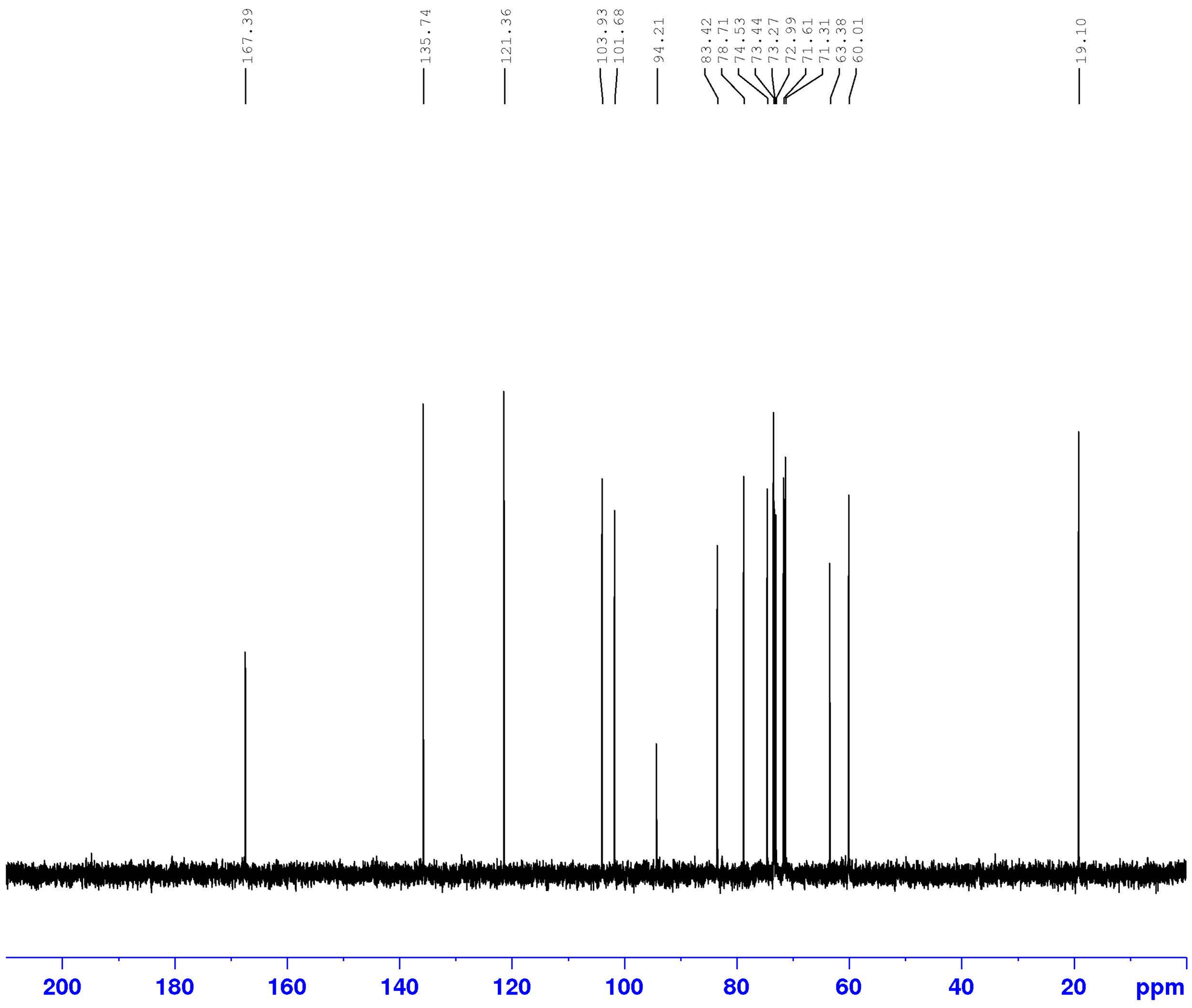
Date_	20101127
Time	9.59
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	D2O
NS	8
DS	16
SWH	2314.815 Hz
FIDRES	1.130281 Hz
AQ	0.4423680 sec
RG	18390.4
DW	216.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.24932504 sec
d4	0.00172414 sec
d11	0.03000000 sec
D16	0.00020000 sec
DELTA	0.00121950 sec
DELTA1	0.00046614 sec
DELTA2	0.00147414 sec
IN0	0.00002925 sec
ST1CNT	0
ZGOPTNS	

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

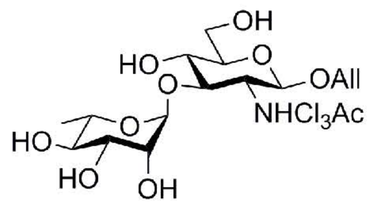
NUC1	1H
P1	6.75 usec
p2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1313888 MHz

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

CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500



Compound **2**



F2 - Acquisition Parameters

Date_	20101127
Time	14.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	4096
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	362
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

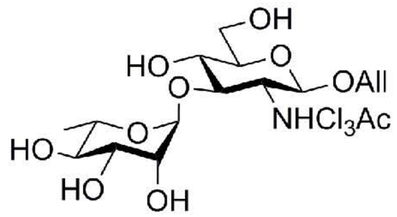
======== CHANNEL f2 ========

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6125063 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **2**



F2 - Acquisition Parameters

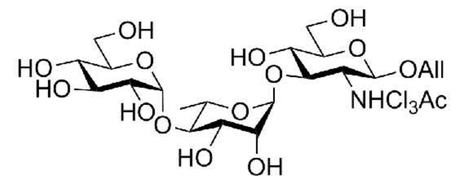
Date_	20101127
Time	14.48
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	D2O
NS	8
DS	16
SWH	2314.815 Hz
FIDRES	1.130281 Hz
AQ	0.4423680 sec
RG	16384
DW	216.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
CNST13	10.0000000
d0	0.00000300 sec
D1	0.87712002 sec
d2	0.00344828 sec
d6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002240 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	6.75 usec
p2	13.50 usec
PL1	-6.00 dB
SFO1	400.1313888 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	5.90 usec
PL2	-6.00 dB
SFO2	100.6228138 MHz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec

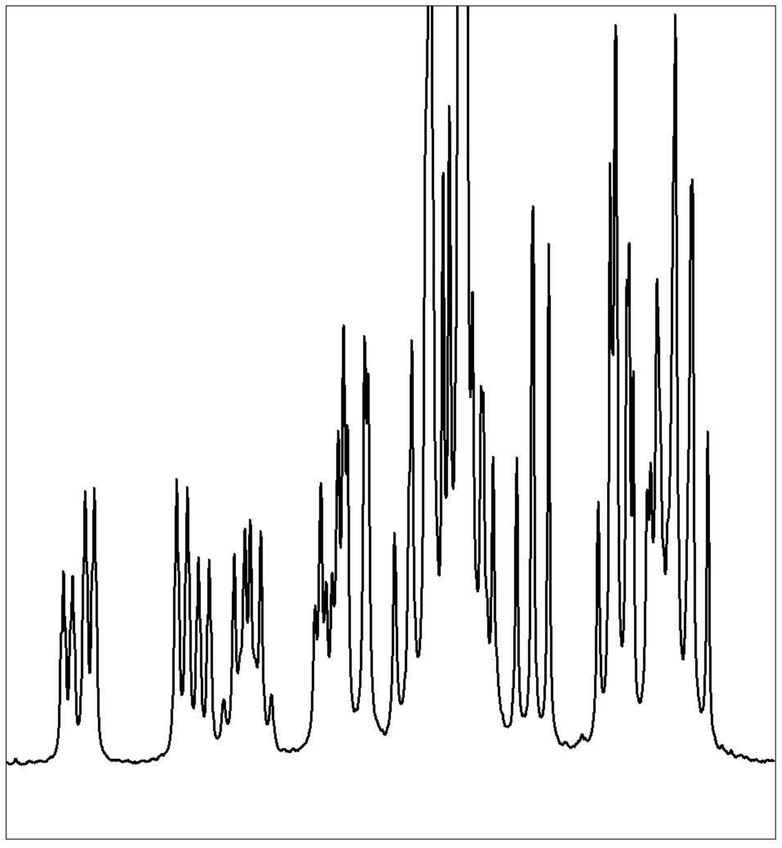
F1 - Acquisition parameters

TD	256
SFO1	100.6228 MHz
FIDRES	87.193077 Hz
SW	221.833 ppm
FnMODE	QF

5.366 5.362 5.323 5.319 5.292 5.289 5.266 5.263 5.056 5.046 4.911 4.790 4.773 4.235 3.995 3.987 3.982 3.956 3.951 3.886 3.859 3.839 3.830 3.810 3.805 3.796 3.783 3.780 3.765 3.730 3.706 3.683 3.592 3.583 3.566 3.564 3.557 3.531 3.521 3.494 3.471 3.469 3.446 1.345 1.329

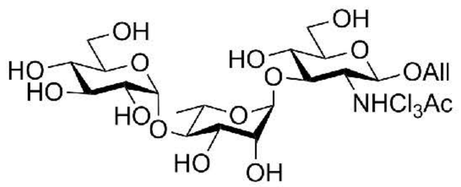
Compound **3**

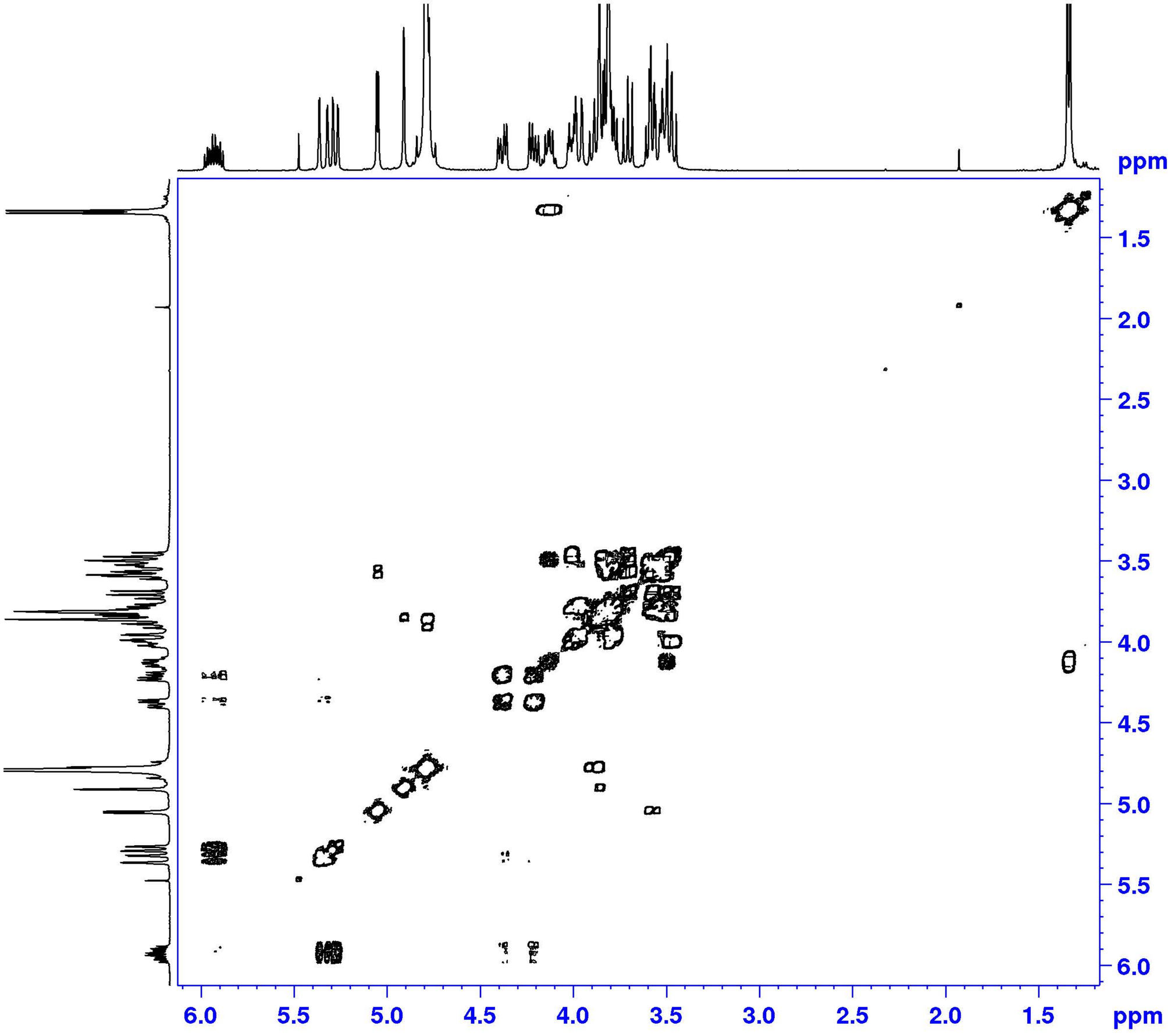





```
F2 - Acquisition Parameters
Date_              20110829
Time                 22.28
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT               D2O
NS                     16
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9583745 sec
RG                    128
DW                60.400 usec
DE                 6.00 usec
TE                 999.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 6.75 usec
PL1               -6.00 dB
SFO1        400.1324708 MHz

F2 - Processing parameters
SI                 32768
SF          400.1299637 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Compound **3**





F2 - Acquisition Parameters

Date_	20110829
Time	22.30
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	D2O
NS	1
DS	8
SWH	2281.022 Hz
FIDRES	1.113780 Hz
AQ	0.4489216 sec
RG	64
DW	219.200 usec
DE	6.50 usec
TE	999.9 K
d0	0.00000300 sec
D1	1.23089194 sec
d11	0.03000000 sec
d12	0.00002000 sec
d13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00043840 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1313857 MHz

====== GRADIENT CHANNEL =====

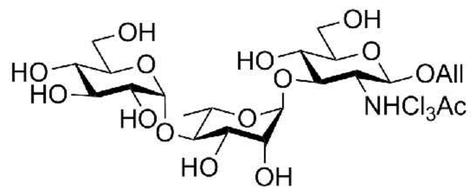
GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

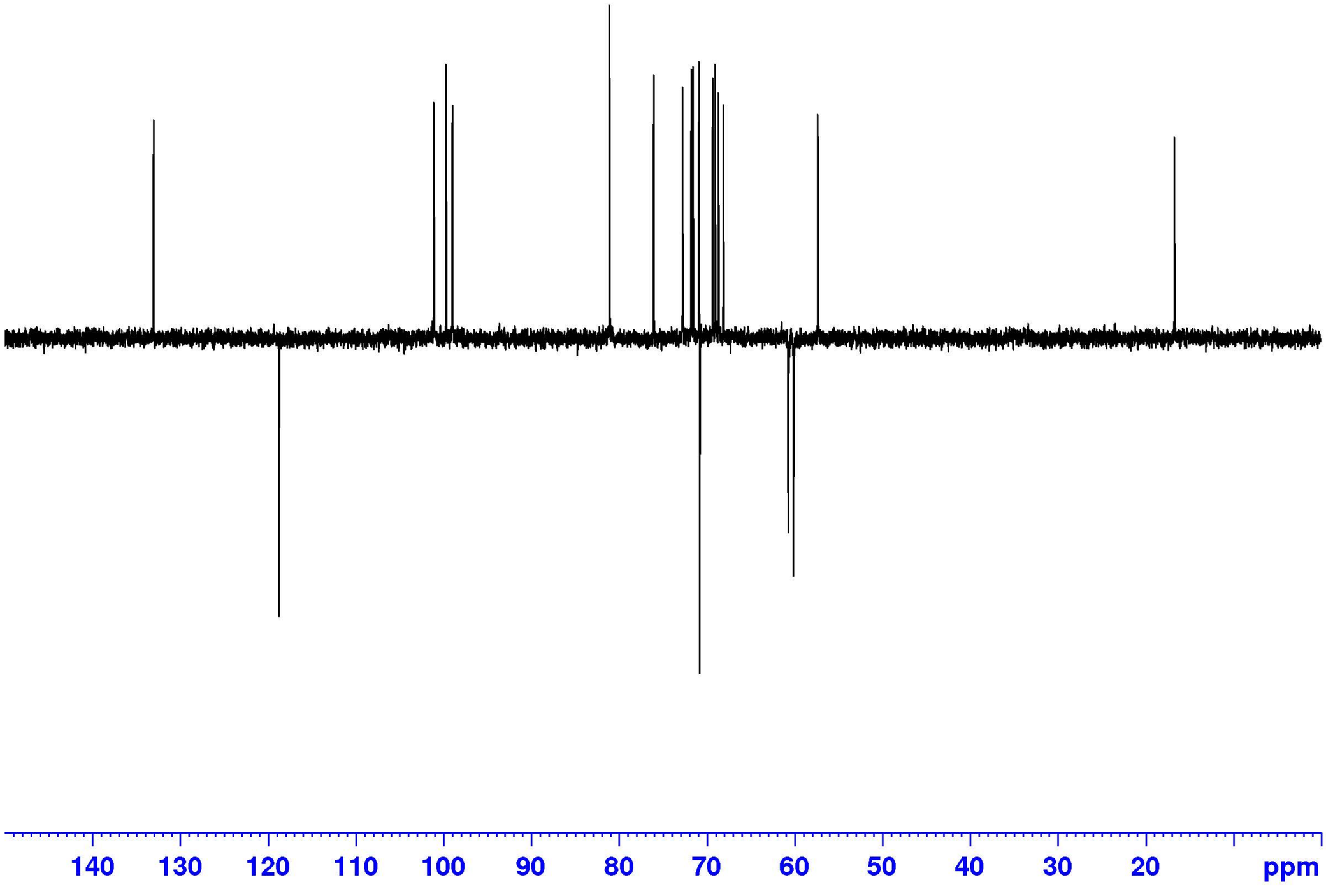
F1 - Acquisition parameters

TD	128
SFO1	400.1314 MHz
FIDRES	17.820484 Hz
SW	5.701 ppm
FnMODE	QF

133.03
118.73
101.07
99.68
98.98
81.08
76.02
72.73
71.74
71.53
70.86
70.78
69.30
69.02
68.64
68.08
60.68
60.09
57.31
16.67

Compound **3**





F2 - Acquisition Parameters

Date_	20110829
Time	23.33
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	D2O
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
D1	2.00000000 sec
d2	0.00344828 sec
d12	0.00002000 sec
DELTA	0.00000751 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

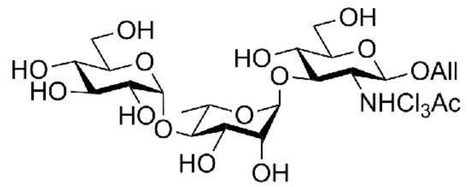
======== CHANNEL f2 ========

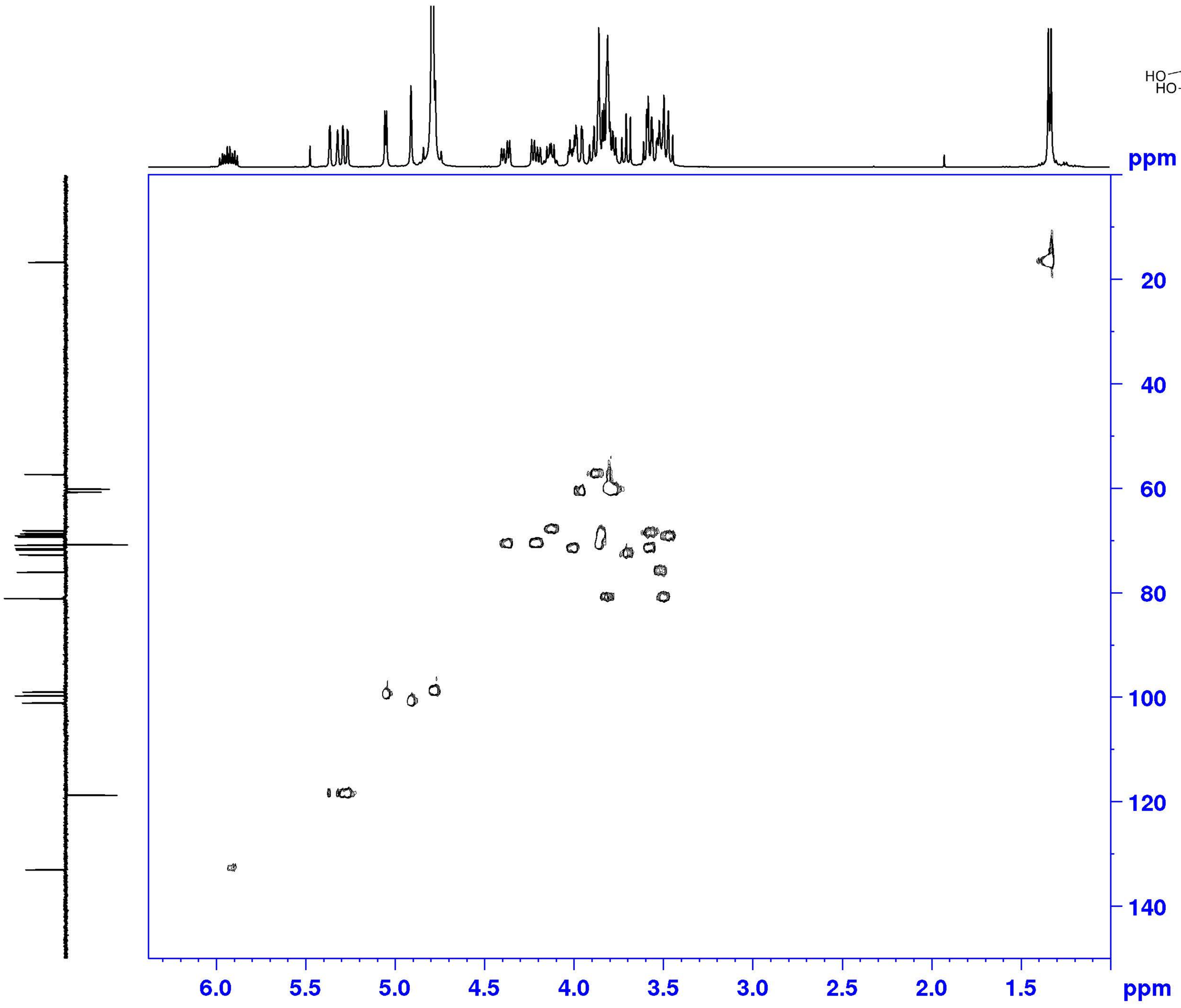
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
p4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

F1 - Acquisition parameters

TD	128
SFO1	400.1316 MHz
FIDRES	7.812500 Hz
SW	2.499 ppm
FnMODE	QF

Compound **3**





F2 - Acquisition Parameters

Date_	20110829
Time	23.35
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	D2O
NS	4
DS	16
SWH	2281.022 Hz
FIDRES	1.113780 Hz
AQ	0.4489216 sec
RG	18390.4
DW	219.200 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.24318099 sec
d4	0.00172414 sec
d11	0.03000000 sec
D16	0.00020000 sec
DELTA	0.00121950 sec
DELTA1	0.00046614 sec
DELTA2	0.00147414 sec
IN0	0.00002760 sec
ST1CNT	0
ZGOPTNS	

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

NUC1	1H
P1	6.75 usec
p2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1313857 MHz

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

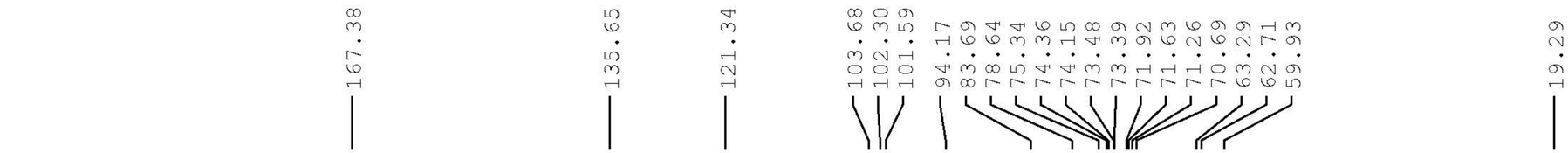
CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

====== GRADIENT CHANNEL =====

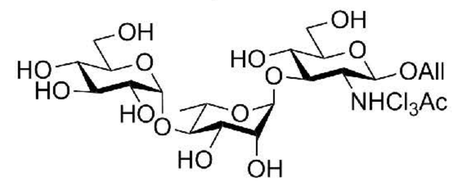
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

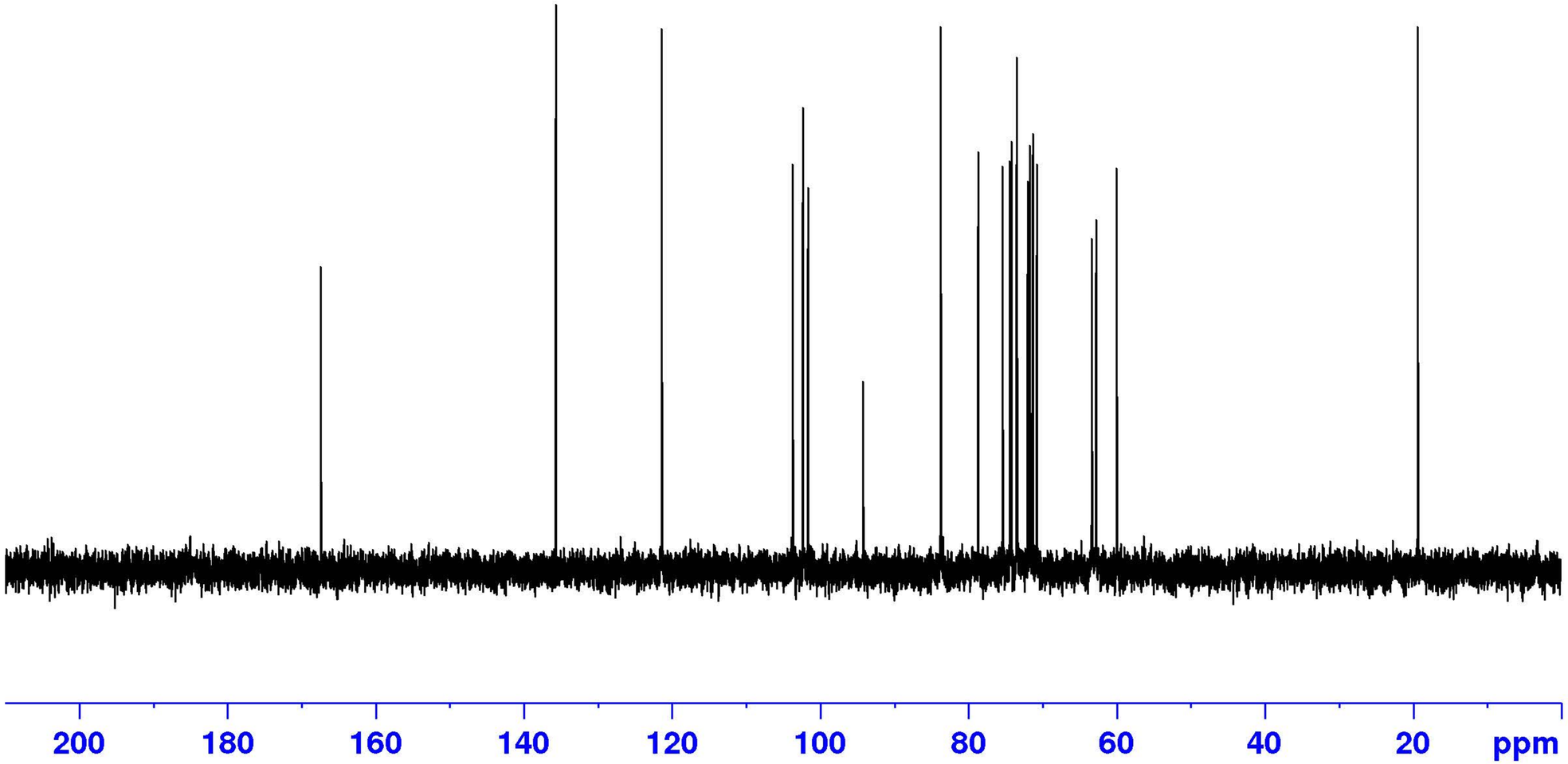
F1 - Acquisition parameters

TD	128
SFO1	100.6208 MHz
FIDRES	141.530792 Hz
SW	180.042 ppm
FnMODE	Echo-Antiecho



Compound **3**





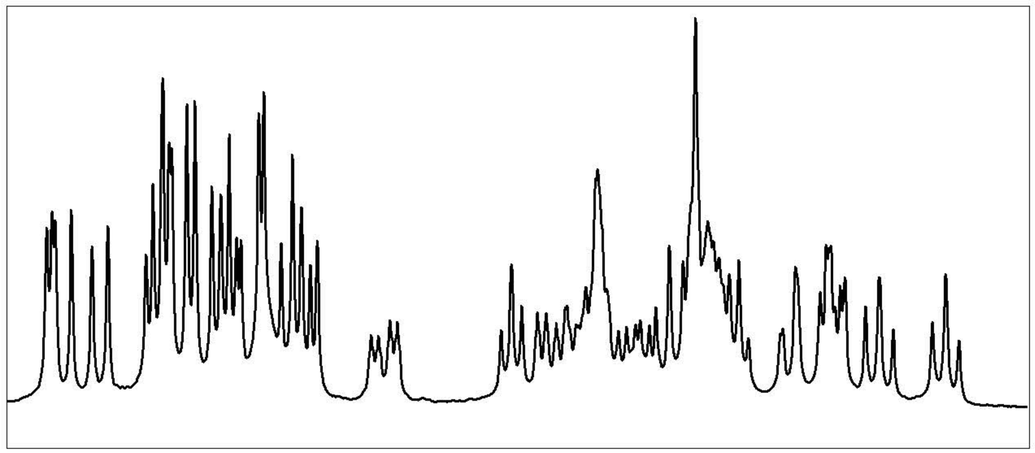
F2 - Acquisition Parameters

Date_	20110830
Time	2.42
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	3072
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	13004
DW	19.100 usec
DE	6.50 usec
TE	999.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz
======== CHANNEL f2 ========	
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

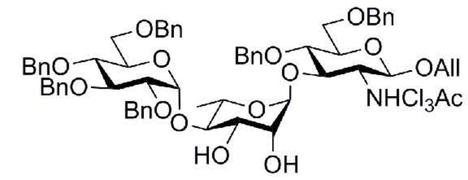
F2 - Processing parameters

SI	32768
SF	100.6125063 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

7.385 7.368 7.349 7.341 7.329 7.320 7.312 7.305 7.290 7.283 7.178 7.169 7.160 5.166 5.164 4.950 4.940 4.935 4.908 4.845 4.768 4.751 4.740 4.735 4.709 4.695 4.666 4.651 4.637 4.624 4.617 4.586 4.577 4.547 4.528 4.513 4.485 4.008 4.005 3.837 3.815 3.806 3.613 1.334 1.318



Compound **4**



F2 - Acquisition Parameters

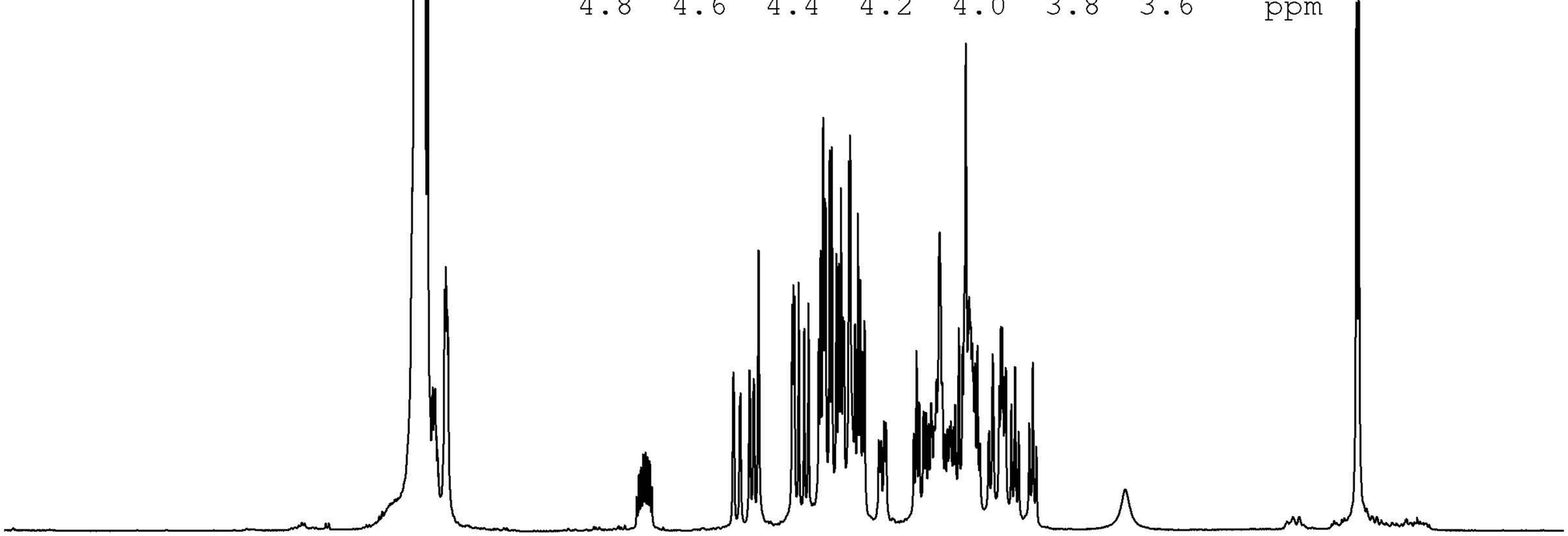
Date_	20120222
Time	22.03
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	35.9
DW	60.400 usec
DE	6.00 usec
TE	999.9 K
D1	1.00000000 sec
TD0	1

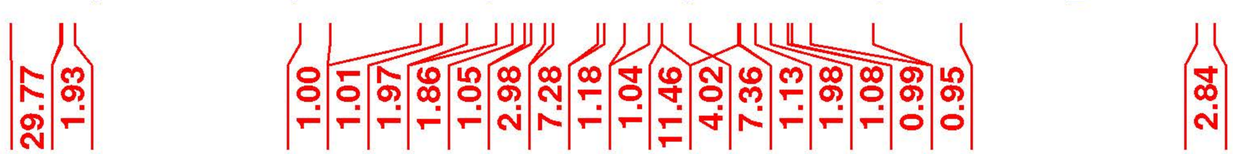
======== CHANNEL f1 ========

NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

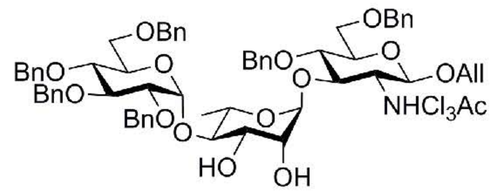
F2 - Processing parameters

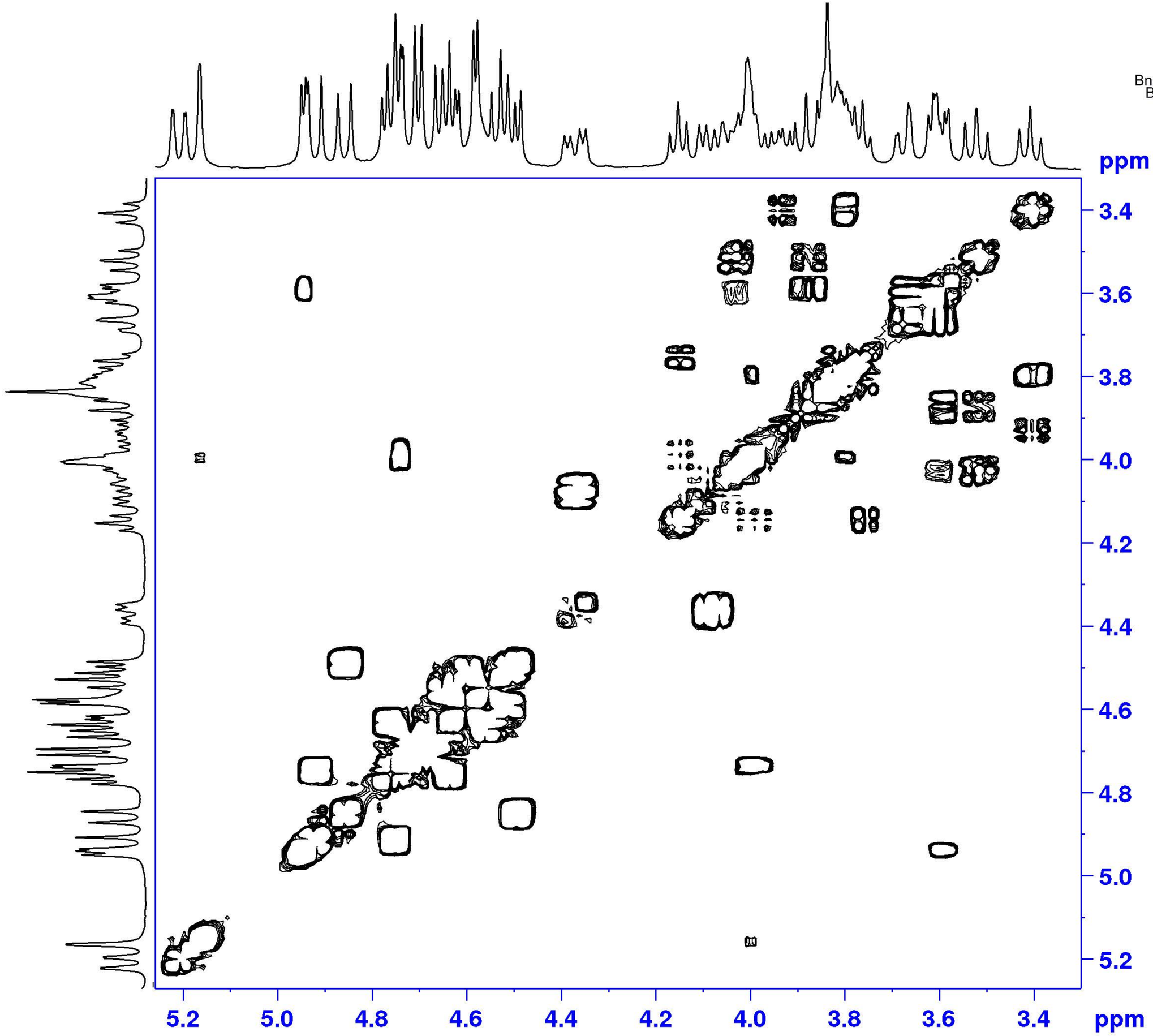
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





Compound **4**





F2 - Acquisition Parameters

Date_	20120222
Time	22.05
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3378.378 Hz
FIDRES	1.649599 Hz
AQ	0.3031040 sec
RG	16
DW	148.000 usec
DE	6.50 usec
TE	999.9 K
D0	0.00000300 sec
D1	1.37629998 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00029600 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1317932 MHz

====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

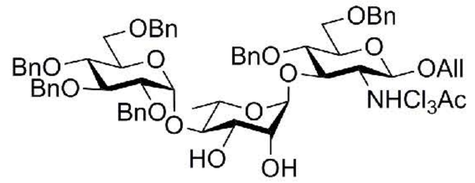
TD	128
SFO1	400.1318 MHz
FIDRES	26.393581 Hz
SW	8.443 ppm
FnMODE	QF

133.62
128.58
128.52
128.48
128.46
128.19
128.05
128.00
127.94
127.88
127.80
127.72
117.62

99.42
98.80
98.46
85.93
81.73
79.85
77.88
77.34
76.35
75.66
75.17
75.06
74.41
74.09
73.61
73.57
73.48
71.39
70.78
69.92
69.84
69.29
68.69
67.09
55.21

17.75

Compound **4**



F2 - Acquisition Parameters

Date_	20120222
Time	22.57
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	800
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

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

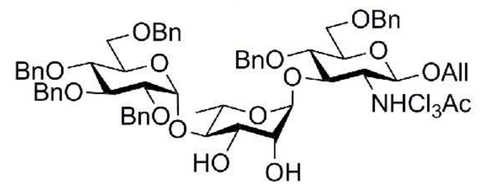
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
P4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

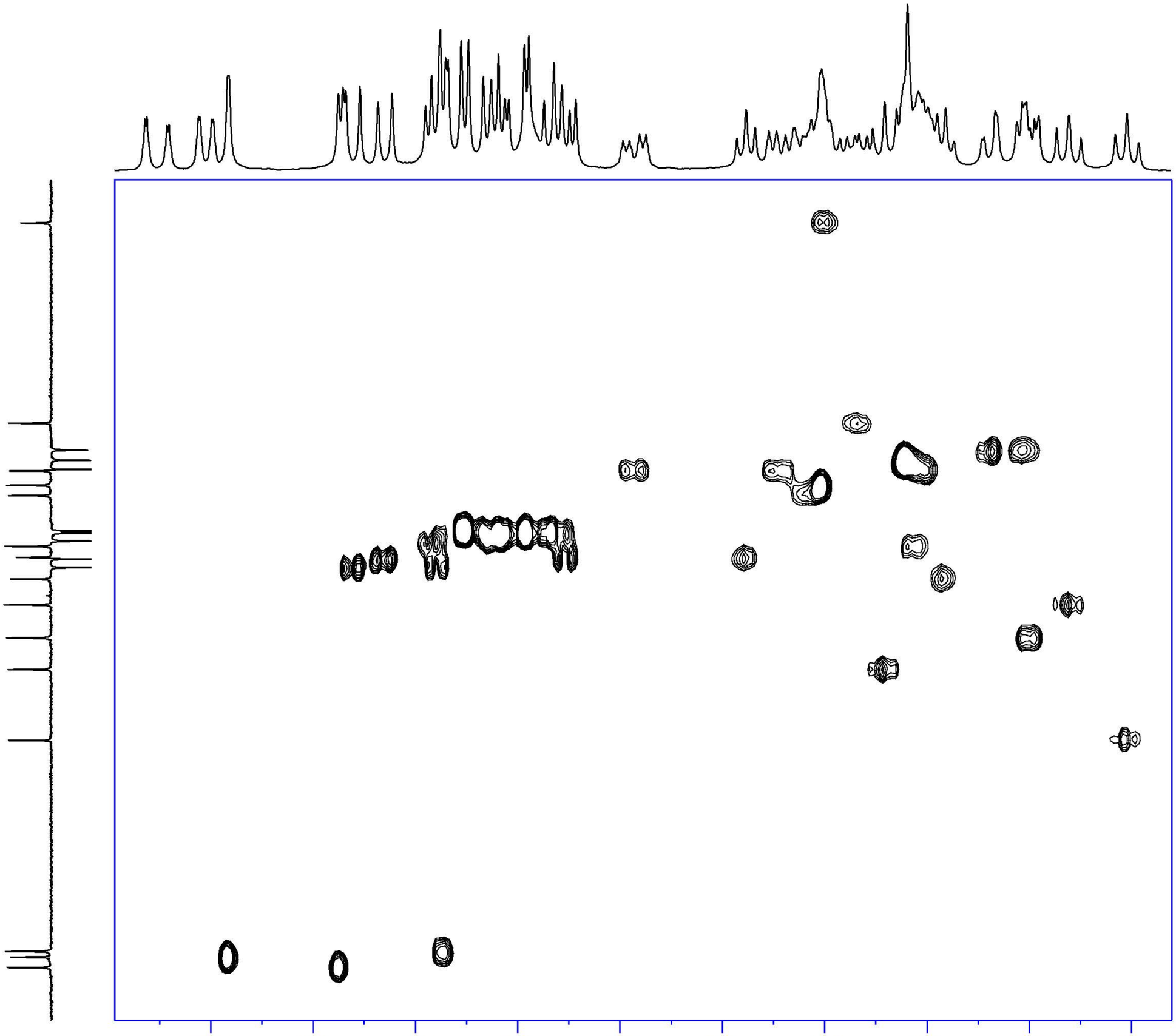
F2 - Processing parameters

SI	32768
SF	100.6127655 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

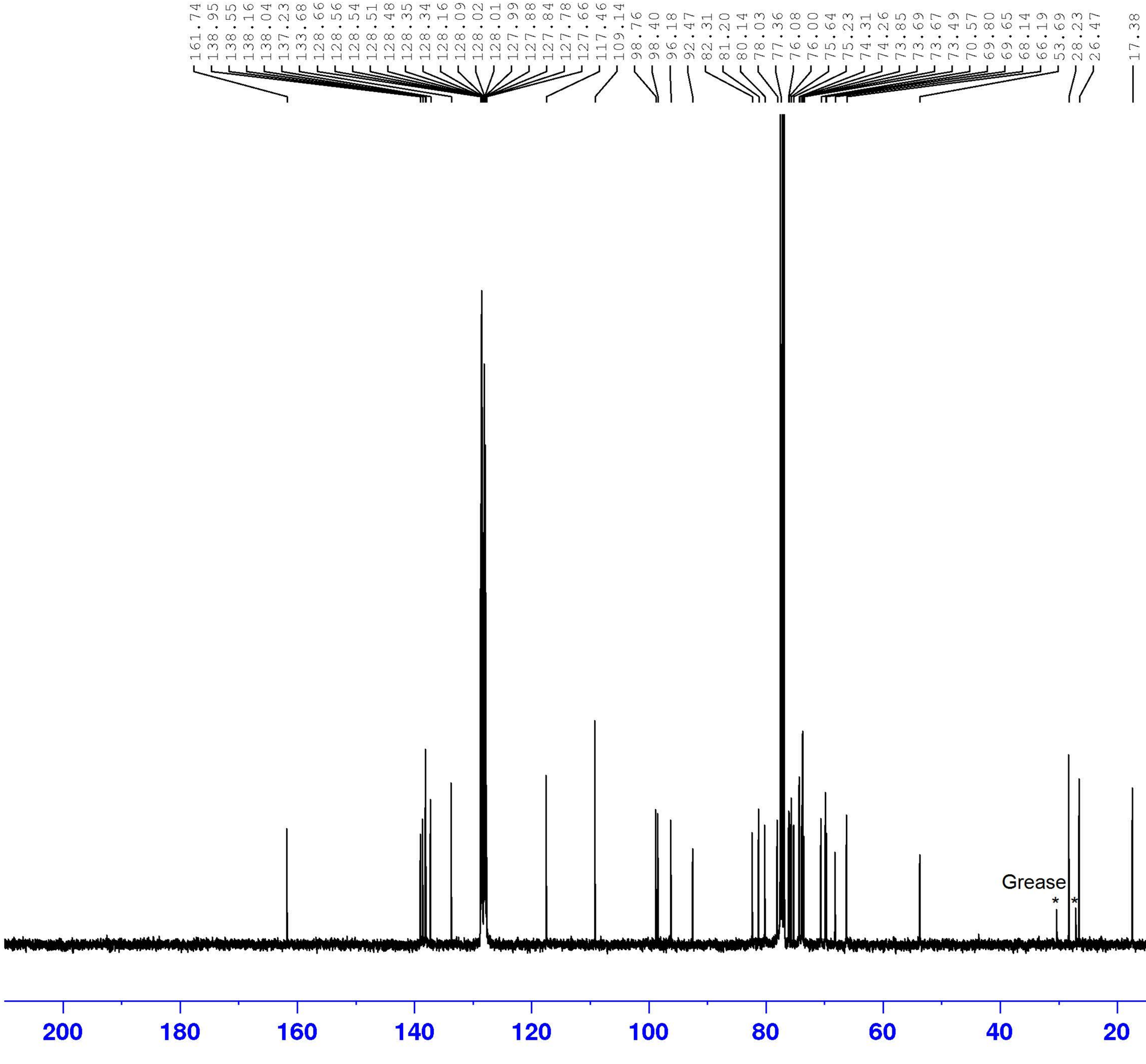
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **4**

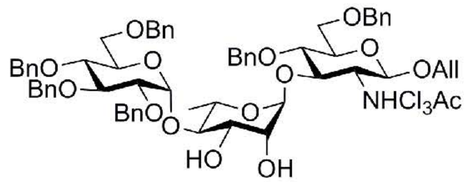





```
F2 - Acquisition Parameters
Date_            20120222
Time                22.59
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                  3000
SOLVENT             CDCl3
NS                     4
DS                    16
SWH             3378.378 Hz
FIDRES          1.126126 Hz
AQ              0.4440000 sec
RG                   6502
DW                148.000 usec
DE                  6.00 usec
TE                 999.9 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.33679998 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
IN0            0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.75 usec
P2                 13.50 usec
P28              1000.00 usec
PL1                -6.00 dB
SFO1         400.1317932 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                  5.90 usec
P14               500.00 usec
P24              2000.00 usec
PCPD2              75.00 usec
PL0               120.00 dB
PL2                -6.00 dB
PL12               23.00 dB
SFO2         100.6208180 MHz
SP3                 6.74 dB
SP7                 6.74 dB
SPNAM[3] Crp60,0.5,20.1
SPNAM[7]    Crp60comp.4
SPOAL3            0.500
SPOAL7            0.500
SPOFFS3  0 Hz
SPOFFS7  0 Hz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   128
SFO1          100.6208 MHz
FIDRES       141.498032 Hz
SW             180.000 ppm
FnMODE    Echo-Antiecho
```



Compound **4**



F2 - Acquisition Parameters

Date_	20111207
Time	7.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	23170.5
DW	19.100 usec
DE	6.50 usec
TE	999.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

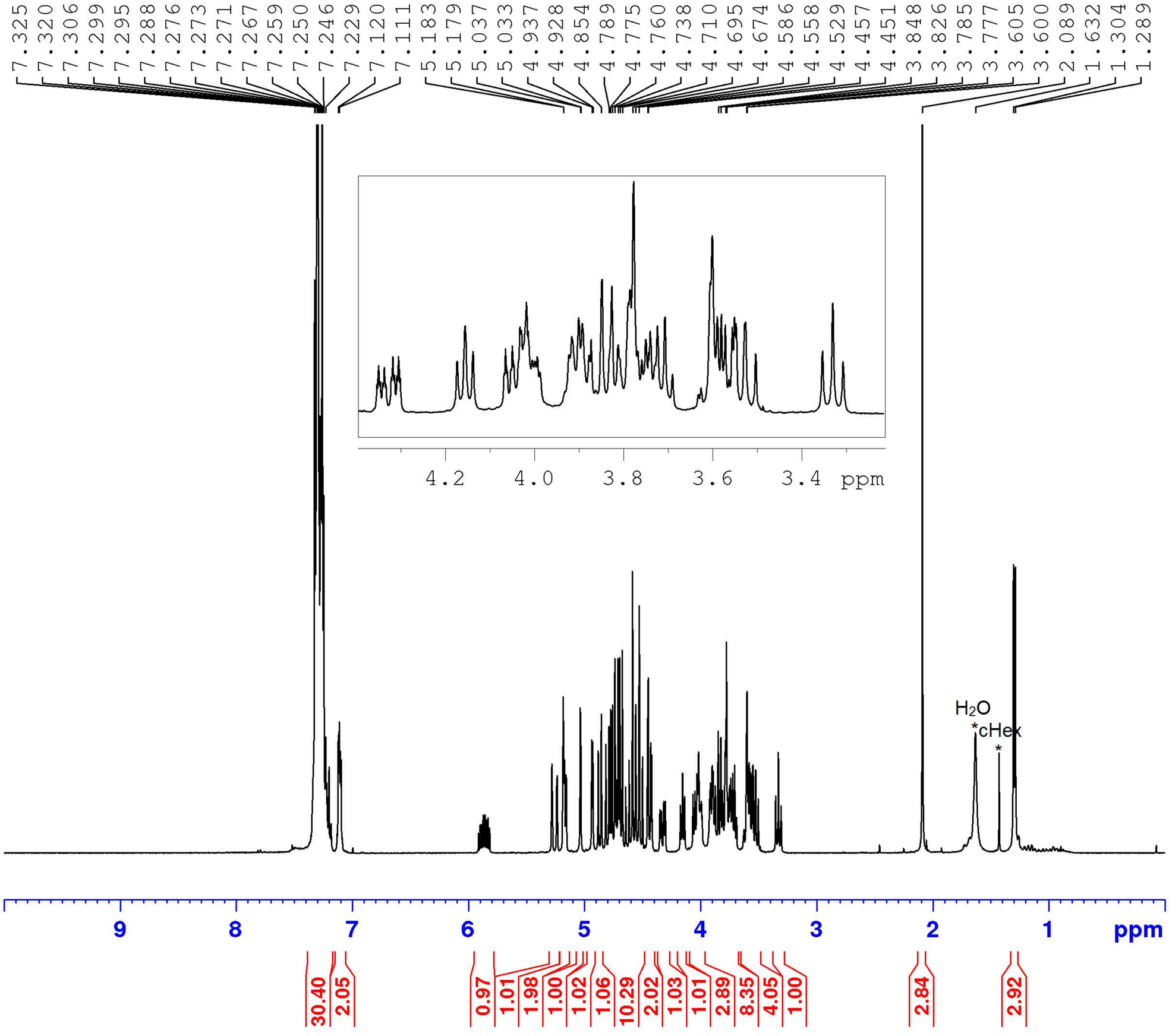
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

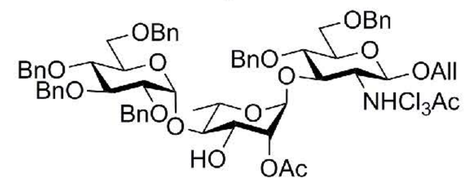
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127592 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Compound **5**



F2 - Acquisition Parameters

Date_	20111208
Time	16.13
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	114
DW	60.400 usec
DE	6.00 usec
TE	999.9 K
D1	1.00000000 sec
TD0	1

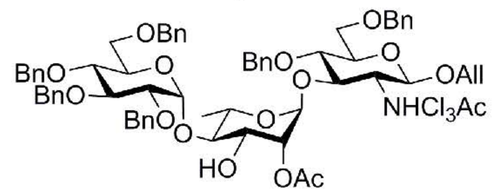
======== CHANNEL f1 ========

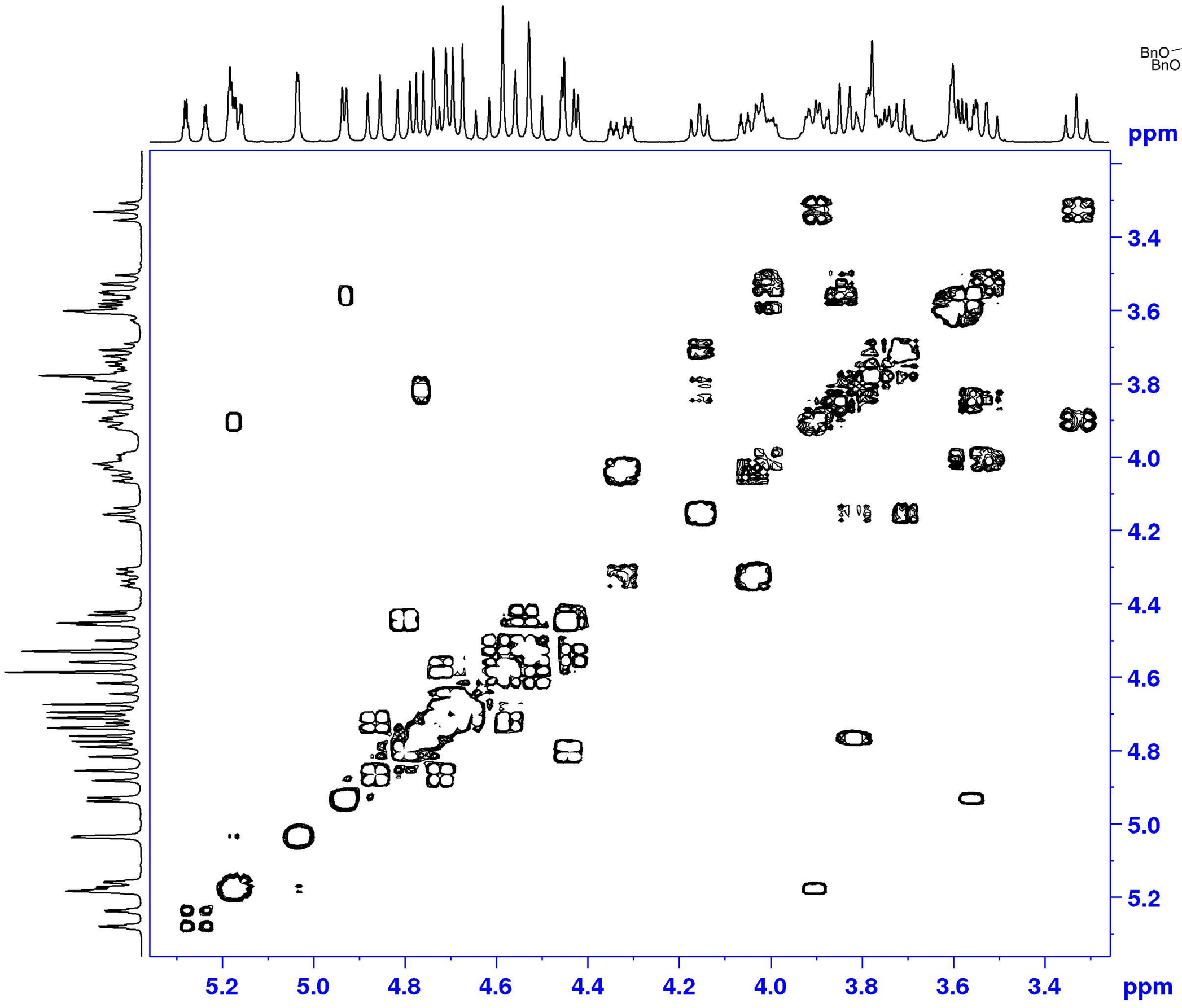
NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Compound **5**





F2 - Acquisition Parameters

Date_	20111208
Time	16.15
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3188.775 Hz
FIDRES	1.557019 Hz
AQ	0.3211264 sec
RG	64
DW	156.800 usec
DE	6.50 usec
TE	999.9 K
D0	0.00000300 sec
D1	1.35786796 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00031360 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1317330 MHz

====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	400.1317 MHz
FIDRES	24.912308 Hz
SW	7.969 ppm
FnMODE	QF

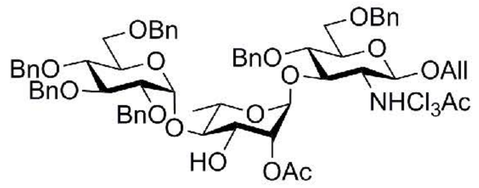
133.61
128.53
128.44
128.41
128.10
128.03
127.97
127.94
127.92
127.87
127.84
127.78
127.77
127.67
117.70

99.15
98.08
97.04

85.37
81.72
79.91
77.78
76.49
75.61
75.20
75.09
74.25
73.99
71.84
71.37
69.98
69.20
68.57
68.30
67.53
55.39

21.07
17.81

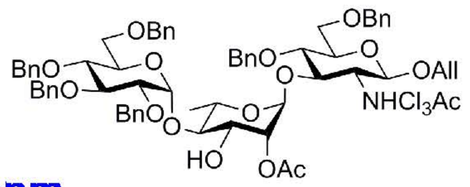
Compound **5**

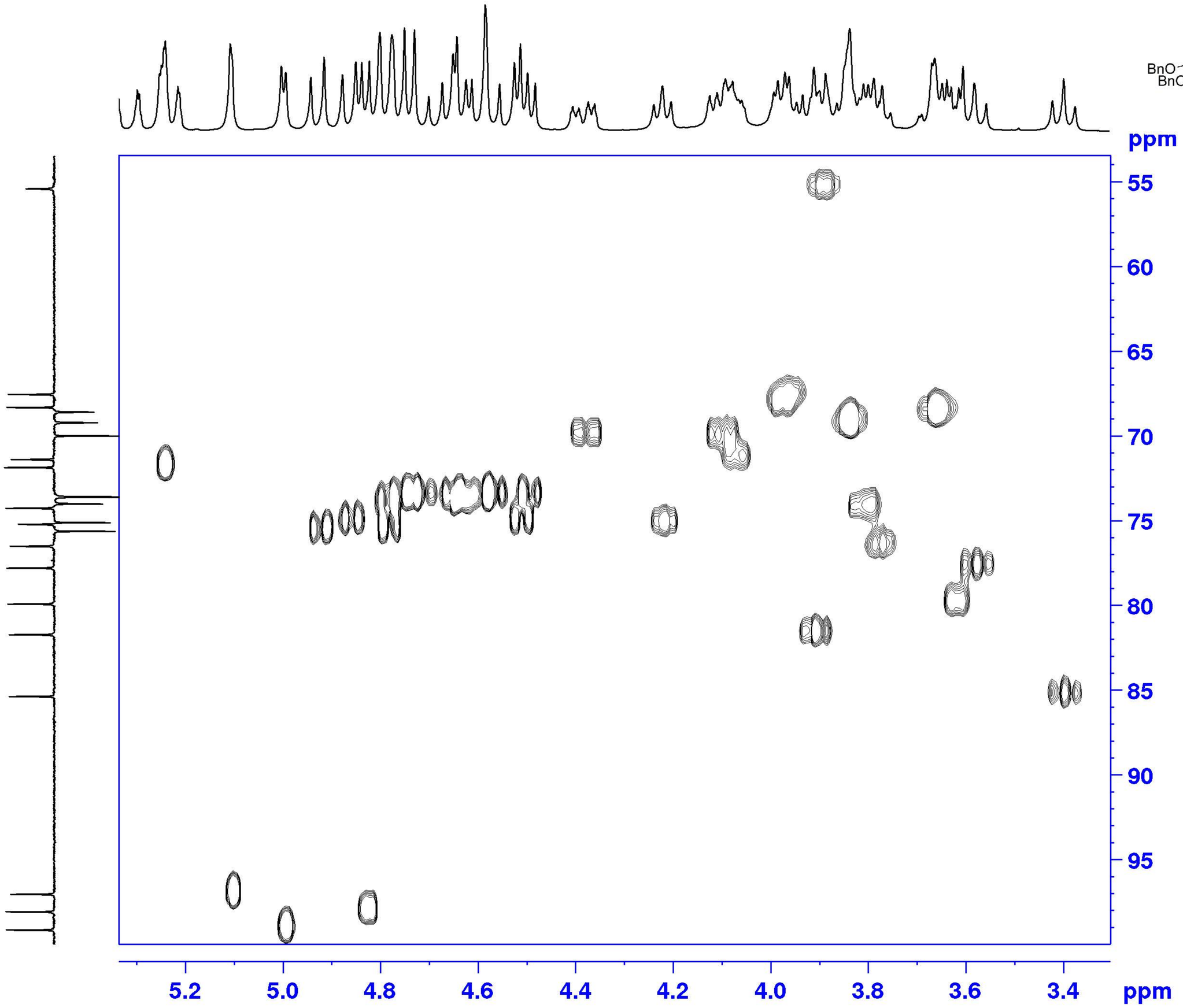



```
F2 - Acquisition Parameters
Date_              20111209
Time                   7.07
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG          deptsp135
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664256 sec
RG                   16384
DW                  20.850 usec
DE                   6.00 usec
TE                  999.9 K
CNST2           145.0000000
D1               2.00000000 sec
D2               0.00344828 sec
D12              0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    5.90 usec
P12                2000.00 usec
PL0                 120.00 dB
PL1                  -6.00 dB
SFO1           100.6228298 MHz
SP2                   6.74 dB
SPNAM[2]      Crp60comp.4
SPOAL2               0.500
SPOFFS2   0 Hz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
P3                    6.75 usec
P4                   13.50 usec
PCPD2                80.00 usec
PL2                  -6.00 dB
PL12                 20.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **5**





F2 - Acquisition Parameters

Date_	20111209
Time	7.10
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	2
DS	16
SWH	3378.378 Hz
FIDRES	1.649599 Hz
AQ	0.3031040 sec
RG	5160.6
DW	148.000 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.38858902 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1318230 MHz

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

CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

====== GRADIENT CHANNEL =====

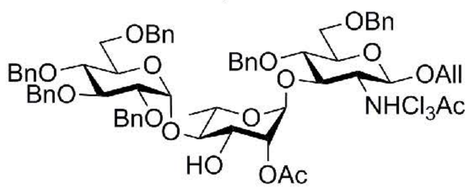
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	100.6208 MHz
FIDRES	141.498032 Hz
SW	180.000 ppm
FnMODE	Echo-Antiecho

170.31
161.78
138.69
138.10
138.01
137.91
137.62
137.43
133.64
128.56
128.50
128.46
128.43
128.12
128.05
127.99
127.97
127.94
127.89
127.86
127.80
127.69
117.72
99.17
98.11
97.07
92.42
85.39
81.74
79.94
77.81
76.52
75.63
75.22
75.12
74.28
74.01
73.61
71.87
71.40
70.00
69.23
68.61
68.33
67.56
55.41
21.09
17.83

Compound **5**

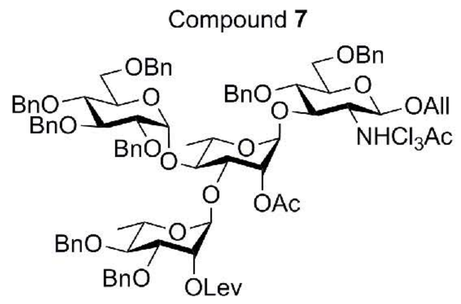


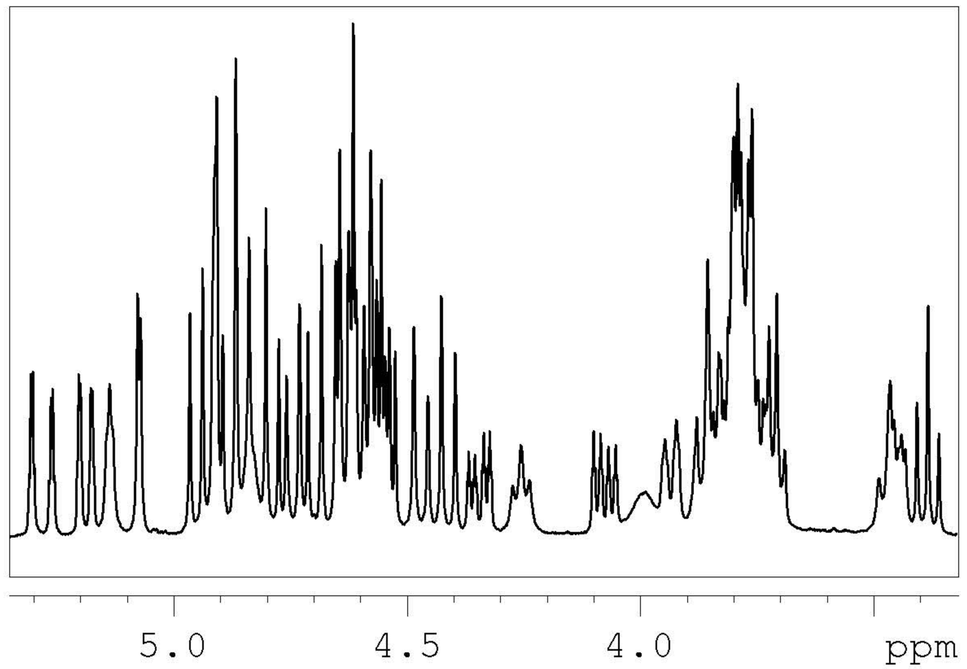

```
F2 - Acquisition Parameters
Date_            20111209
Time                8.29
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1246
DS                    4
SWH           26178.010 Hz
FIDRES         0.399445 Hz
AQ             1.2517376 sec
RG               20642.5
DW                19.100 usec
DE                 6.50 usec
TE                999.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  5.90 usec
PL1                -6.00 dB
SFO1         100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               20.00 dB
PL13               20.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127663 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

cHex
Grease
*
*
*

200 180 160 140 120 100 80 60 40 20 ppm

7.363 7.350 7.347 7.341 7.336 7.324 7.320 7.316 7.311 7.309 7.300 7.296 7.283 7.275 7.270 7.262 7.253 7.249 7.243 7.236 7.231 7.223 7.185 4.911 4.907 4.866 4.801 4.643 4.614 4.577 4.554 3.798 3.789 3.782 3.766 3.759 2.732 2.721 2.169 2.093 1.616 1.248 1.232 1.170 1.154





F2 - Acquisition Parameters

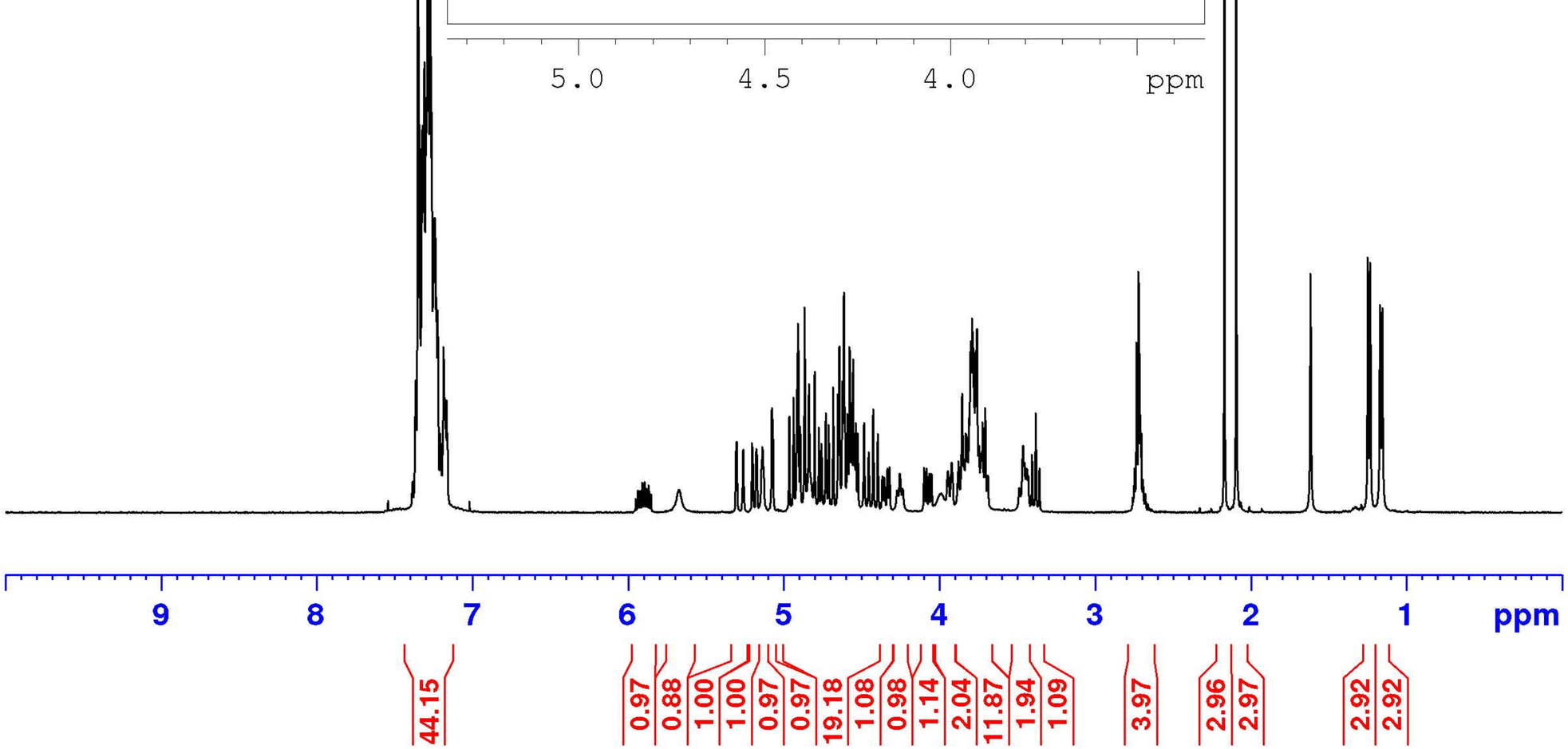
Date_	20120821
Time	10.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	161.3
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

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

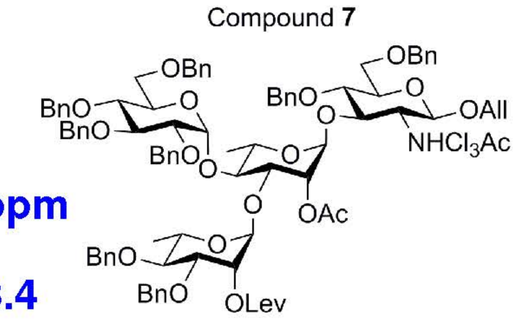
NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



Compound **7**



ppm

3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0

5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 ppm

F2 - Acquisition Parameters

Date_	20111210
Time	20.02
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3289.474 Hz
FIDRES	1.606188 Hz
AQ	0.3112960 sec
RG	14.3
DW	152.000 usec
DE	6.50 usec
TE	999.9 K
D0	0.00000300 sec
D1	1.36810803 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00030400 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1317424 MHz

====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	400.1317 MHz
FIDRES	25.699013 Hz
SW	8.221 ppm
FnMODE	QF

133.67
128.53
128.45
128.42
128.39
128.31
128.28
128.21
128.11
128.02
127.92
127.88
127.84
127.78
127.73
127.62
127.55
127.50
127.37
117.83

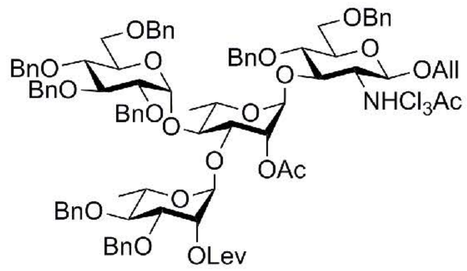
97.85
96.47
81.63
79.89
77.52
76.33
75.60
75.22
75.01
74.27
73.92
73.76
73.51
72.89
71.77
71.54
70.09
69.11
68.77
68.57
68.28
68.01
56.33
53.53

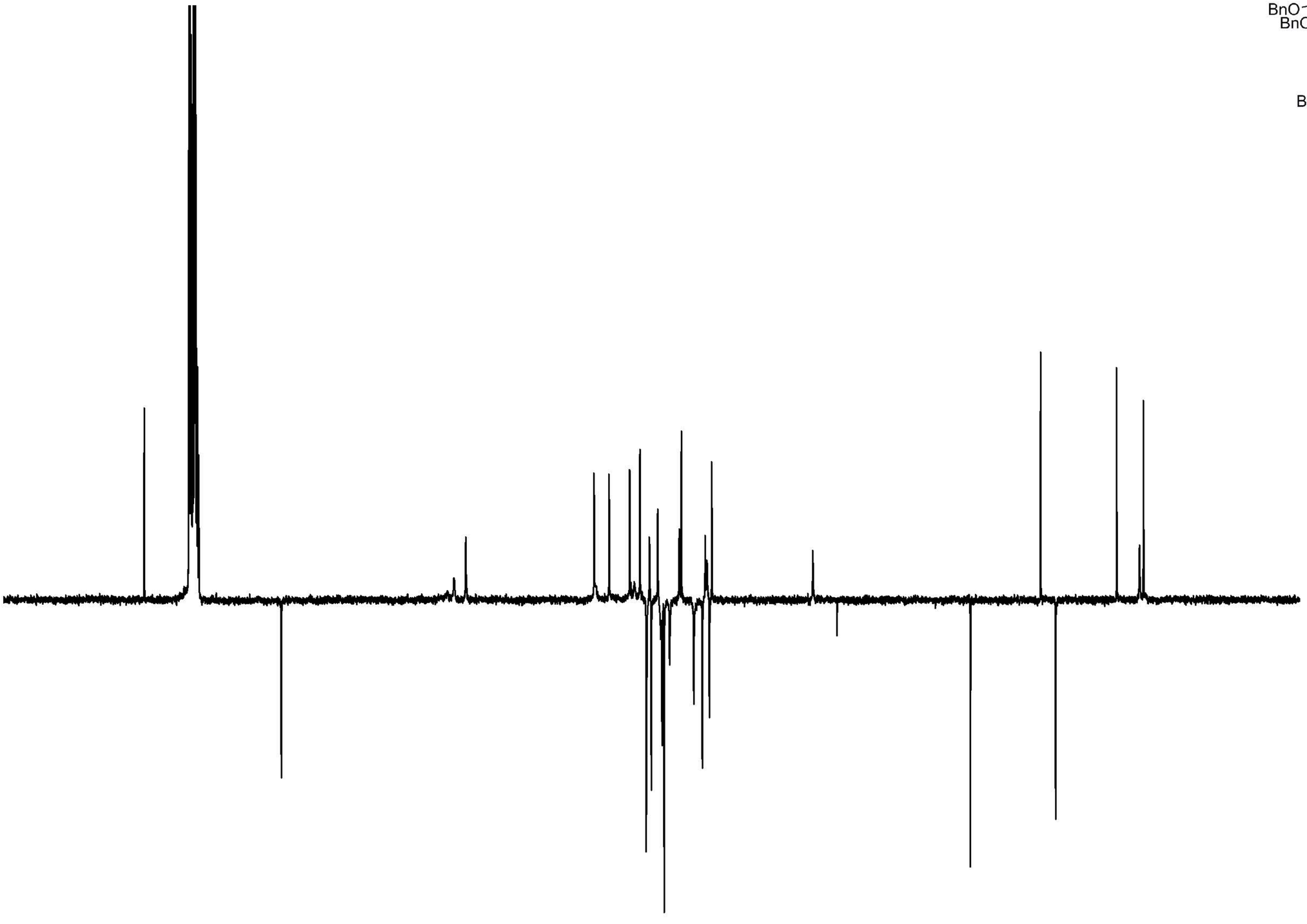
38.10

29.96
28.23

21.17
18.52
18.07

Compound **7**





F2 - Acquisition Parameters

Date_	20111210	
Time	21.07	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	deptsp135	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	23980.814	Hz
FIDRES	0.365918	Hz
AQ	1.3664256	sec
RG	16384	
DW	20.850	usec
DE	6.00	usec
TE	999.9	K
CNST2	145.0000000	
D1	2.00000000	sec
D2	0.00344828	sec
D12	0.00002000	sec
TD0	1	

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

NUC1	13C	
P1	5.90	usec
P12	2000.00	usec
PL0	120.00	dB
PL1	-6.00	dB
SFO1	100.6228298	MHz
SP2	6.74	dB
SPNAM[2]	Crp60comp.4	
SPOAL2	0.500	
SPOFFS2	0 Hz	

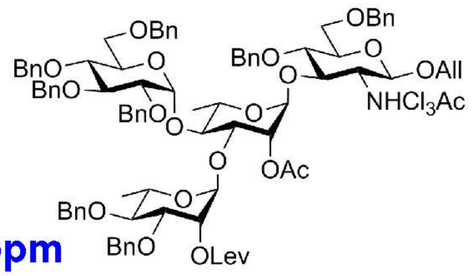
======== CHANNEL f2 ========

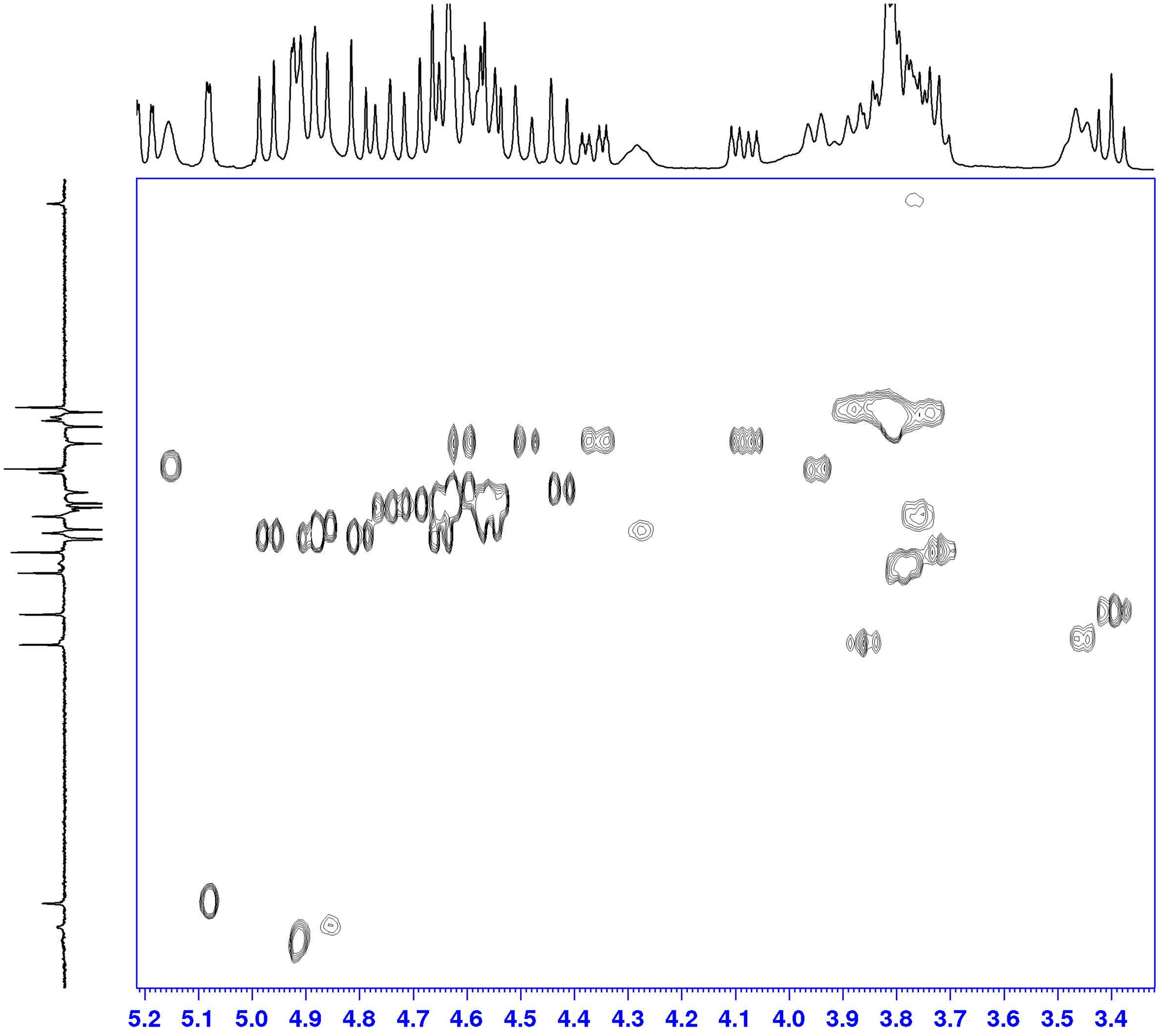
CPDPRG[2	waltz16	
NUC2	1H	
P3	6.75	usec
P4	13.50	usec
PCPD2	80.00	usec
PL2	-6.00	dB
PL12	20.00	dB
SFO2	400.1316005	MHz

F2 - Processing parameters

SI	32768	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

Compound **7**





F2 - Acquisition Parameters

Date_	20111210
Time	21.09
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	2
DS	16
SWH	3289.474 Hz
FIDRES	1.606188 Hz
AQ	0.3112960 sec
RG	2298.8
DW	152.000 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.38039696 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1317424 MHz

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

CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

====== GRADIENT CHANNEL =====

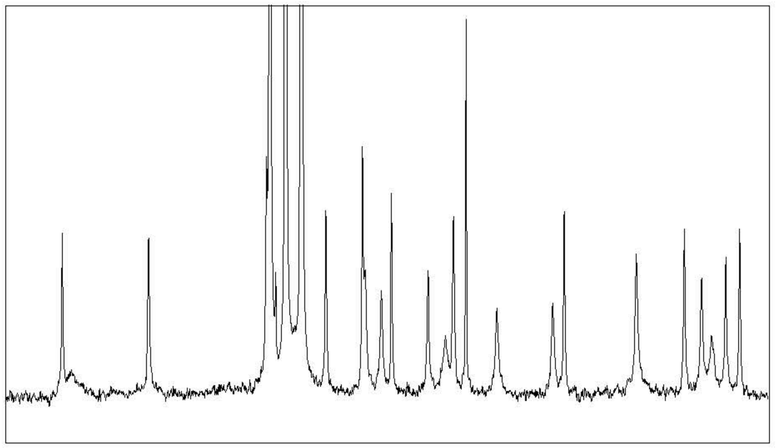
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

F1 - Acquisition parameters

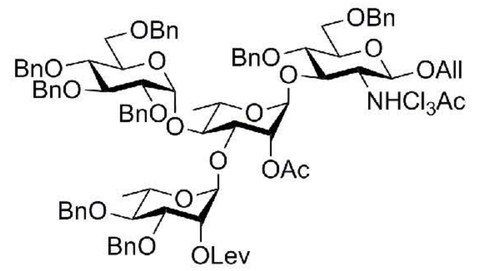
TD	128
SFO1	100.6208 MHz
FIDRES	141.498032 Hz
SW	180.000 ppm
FnMODE	Echo-Antiecho

206.45

171.89
170.00
161.84
138.84
138.65
138.54
138.39
138.31
138.03
137.51
133.70
128.55
128.47
128.44
128.41
128.34
128.30
128.24
128.13
128.04
127.95
127.90
127.87
127.80
127.75
127.65
127.57
127.52
127.40
117.85
97.87
96.50
92.39
81.66
79.92
77.55
77.37
76.36
75.62
75.24
75.03
74.30
73.95
73.79
73.54
72.92
71.79
71.57
70.11
69.15
68.80
68.60
68.32
68.04
56.34
53.55
38.12
29.98
28.26
21.19
18.55
18.09



Compound **7**



F2 - Acquisition Parameters

Date_	20111211
Time	0.09
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	6502
DW	19.100 usec
DE	6.50 usec
TE	999.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

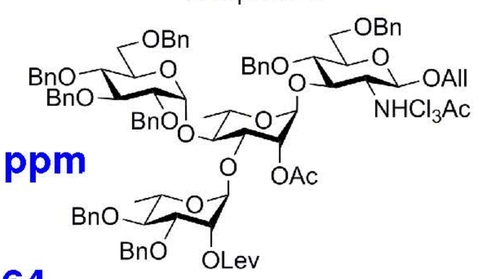
F2 - Processing parameters

SI	32768
SF	100.6127664 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

CH_2Cl_2
*

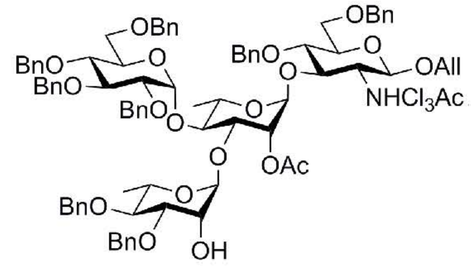
200 180 160 140 120 100 80 60 40 20 ppm

Compound **7**

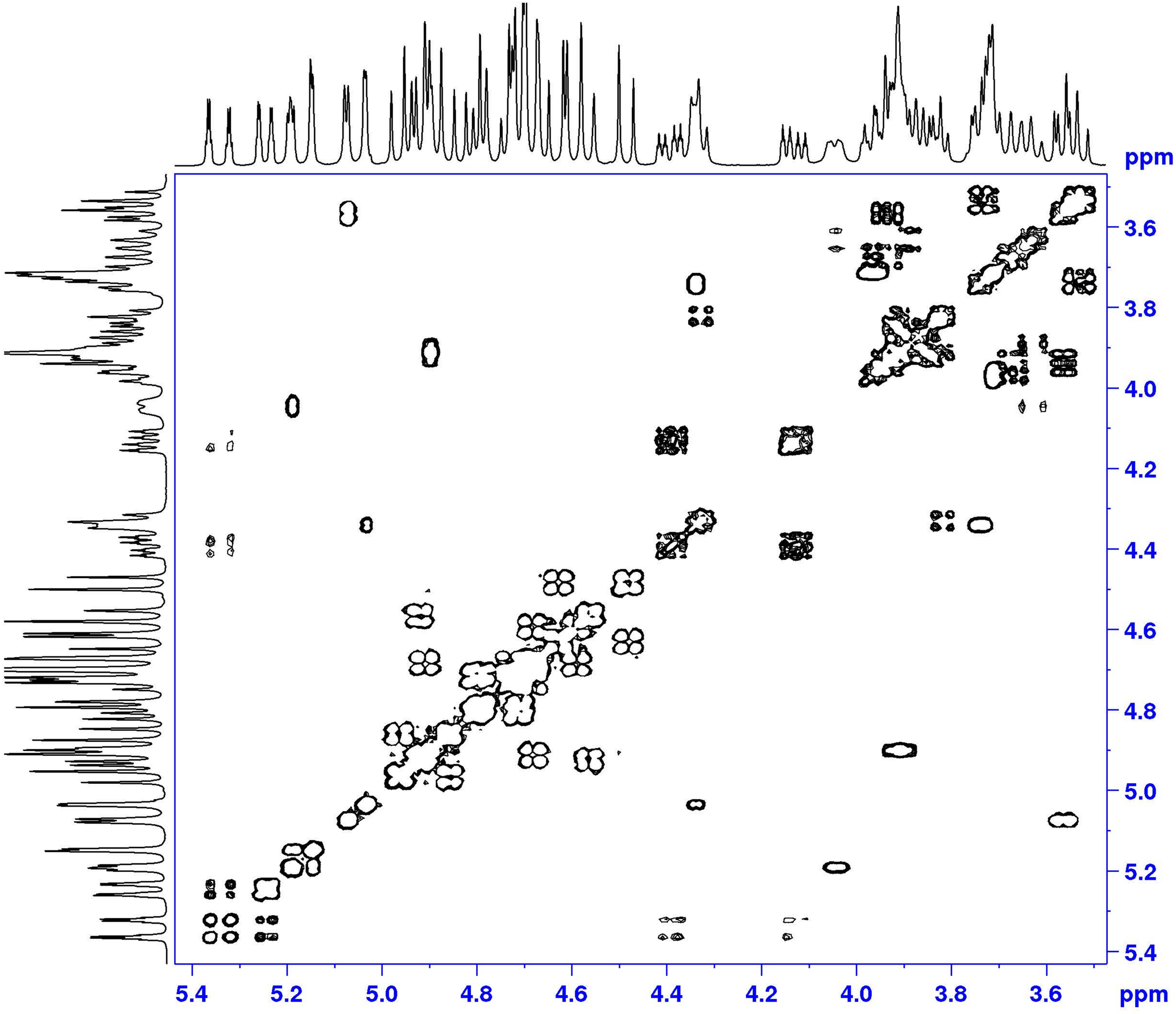



```
F2 - Acquisition Parameters
Date_              20111211
Time                   0.12
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG     hmbcgplpndqf
TD                     2048
SOLVENT               CDCl3
NS                       16
DS                       16
SWH                3289.474 Hz
FIDRES             1.606188 Hz
AQ                0.3112960 sec
RG                     16384
DW                 152.000 usec
DE                   6.00 usec
TE                   999.9 K
CNST2            145.0000000
CNST13            10.0000000
D0                0.00000300 sec
D1                1.00819194 sec
D2                0.00344828 sec
D6                0.05000000 sec
D16               0.00020000 sec
IN0               0.00002240 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.75 usec
P2                    13.50 usec
PL1                   -6.00 dB
SFO1          400.1317424 MHz

======== CHANNEL f2 ========
NUC2                     13C
P3                     5.90 usec
PL2                   -6.00 dB
SFO2          100.6228138 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPNAM[3]          SINE.100
GPZ1                  50.00 %
GPZ2                  30.00 %
GPZ3                  40.10 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       128
```

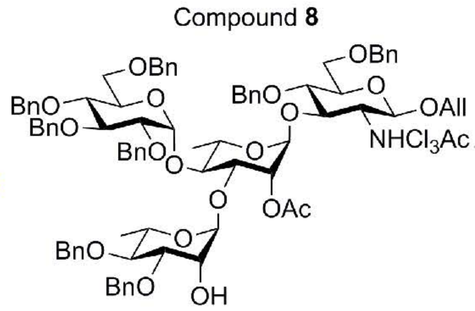
Compound **8**




```
F2 - Acquisition Parameters
Date_              20120906
Time                 22.13
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 100000
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8278.146 Hz
FIDRES            0.082781 Hz
AQ                6.0400000 sec
RG                    22.6
DW                  60.400 usec
DE                   6.00 usec
TE                  303.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   6.75 usec
PL1                 -6.00 dB
SFO1          400.1324708 MHz

F2 - Processing parameters
SI                   32768
SF            400.1300000 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



Compound **8**

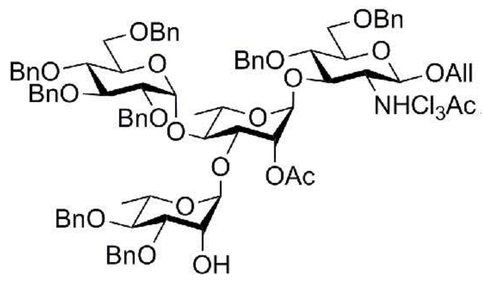



```
F2 - Acquisition Parameters
Date_              20120906
Time                  22.14
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                    2048
SOLVENT               CDCl3
NS                       2
DS                       8
SWH               3205.128 Hz
FIDRES             1.565004 Hz
AQ                0.3194880 sec
RG                     11.3
DW                 156.000 usec
DE                    6.50 usec
TE                   303.0 K
D0              0.00000300 sec
D1              1.35991597 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00031200 sec

======== CHANNEL f1 ========
NUC1                    1H
P0                   6.75 usec
P1                   6.75 usec
P17               2500.00 usec
PL1                 -6.00 dB
PL10                 6.57 dB
SFO1          400.1317466 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     512
SFO1            400.1317 MHz
FIDRES          6.260016 Hz
SW                8.010 ppm
FnMODE                 QF
```

133.70
128.58
128.47
128.45
128.44
128.39
128.36
128.03
127.99
127.94
127.93
127.89
127.84
127.81
127.79
127.69
127.67
117.75
102.62
98.63
97.71
95.63
81.61
81.15
80.02
79.59
79.24
78.53
77.74
77.36
75.85
75.62
75.09
74.87
74.03
73.77
73.73
73.49
73.27
73.24
72.48
71.63
71.54
69.99
69.47
68.57
68.42
68.32
68.27
54.86

21.02
18.58
18.07

Compound **8**



F2 - Acquisition Parameters

Date_	20120907	
Time	0.42	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	deptsp135	
TD	65536	
SOLVENT	CDCl3	
NS	2000	
DS	4	
SWH	23980.814	Hz
FIDRES	0.365918	Hz
AQ	1.3664256	sec
RG	16384	
DW	20.850	usec
DE	6.00	usec
TE	303.0	K
CNST2	145.0000000	
D1	2.00000000	sec
D2	0.00344828	sec
D12	0.00002000	sec
TD0	1	

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

NUC1	13C	
P1	5.90	usec
P12	2000.00	usec
PL0	120.00	dB
PL1	-6.00	dB
SFO1	100.6228298	MHz
SP2	6.74	dB
SPNAM[2]	Crp60comp.4	
SPOAL2	0.500	
SPOFFS2	0 Hz	

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

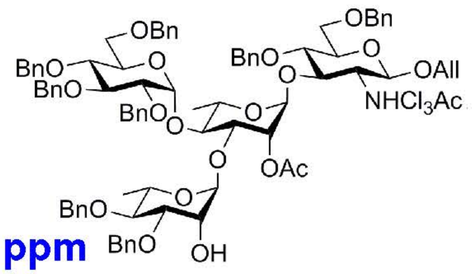
CPDPRG[2	waltz16	
NUC2	1H	
P3	6.75	usec
P4	13.50	usec
PCPD2	80.00	usec
PL2	-6.00	dB
PL12	20.00	dB
SFO2	400.1316005	MHz

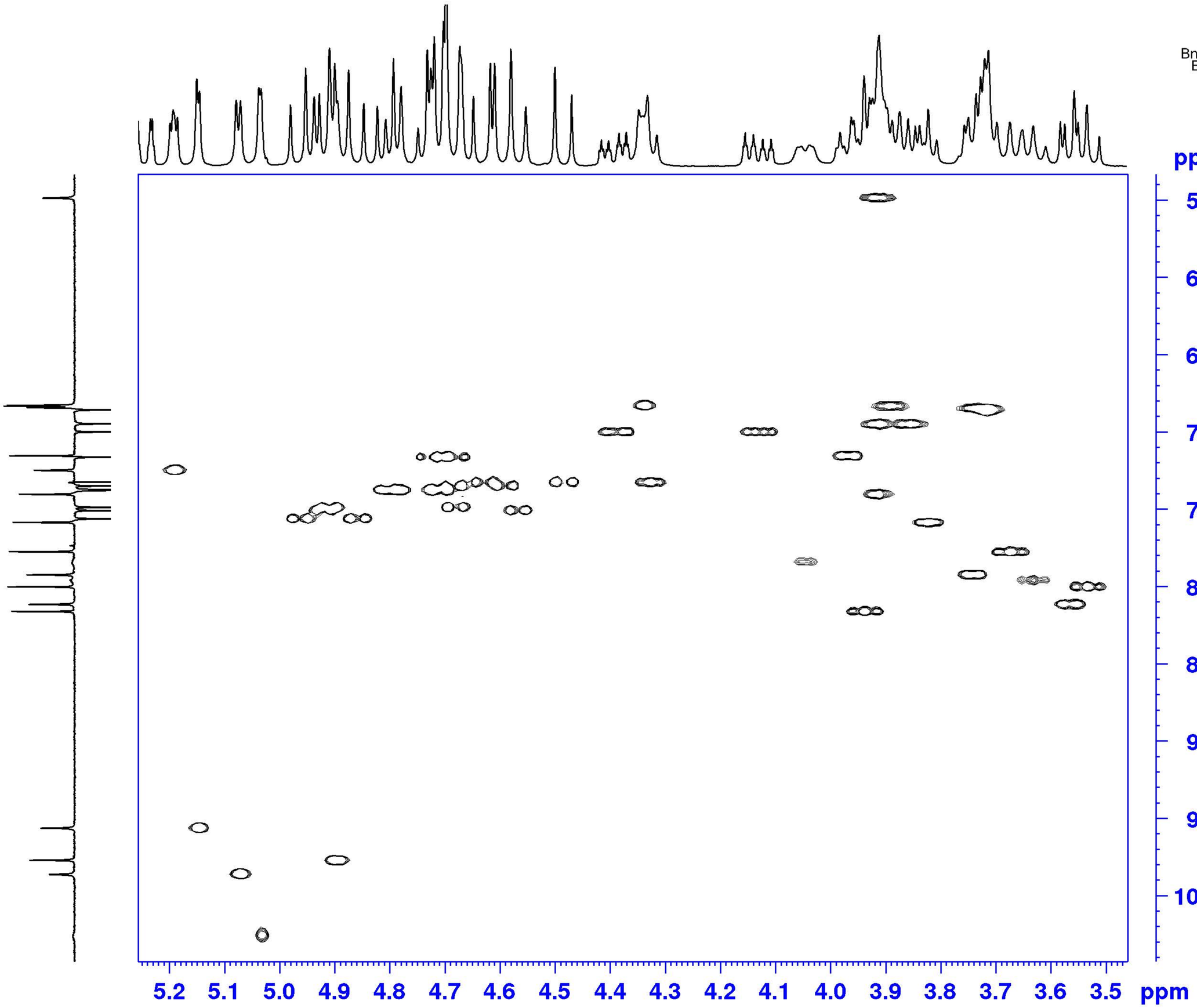
F2 - Processing parameters

SI	32768	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **8**





F2 - Acquisition Parameters

Date_	20120907
Time	0.44
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	3205.128 Hz
FIDRES	1.565004 Hz
AQ	0.3194880 sec
RG	2298.8
DW	156.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.37220502 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1317466 MHz

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

CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

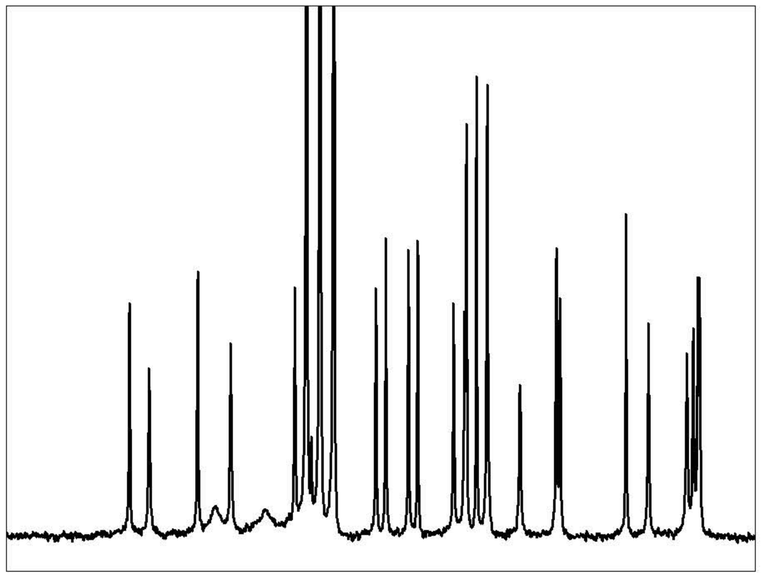
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

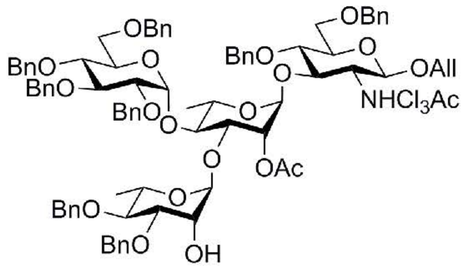
F1 - Acquisition parameters

TD	512
SFO1	100.6208 MHz
FIDRES	35.374508 Hz
SW	180.000 ppm
FnMODE	Echo-Antiecho

169.94
161.63
138.73
138.66
138.39
138.24
138.14
137.89
137.40
133.70
128.57
128.47
128.45
128.44
128.39
128.36
128.03
127.99
127.94
127.89
127.84
127.82
127.79
127.69
127.67
117.75
98.63
97.71
95.63
92.42
81.61
81.15
80.02
79.25
77.75
77.47
77.36
77.16
76.84
75.85
75.62
75.09
74.87
74.03
73.77
73.73
73.49
73.24
72.48
71.63
71.54
69.99
69.47
68.57
68.42
68.32
68.27
54.85
21.02
18.58
18.07



Compound **8**



F2 - Acquisition Parameters

Date_	20120907
Time	6.22
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	5000
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	9195.2
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

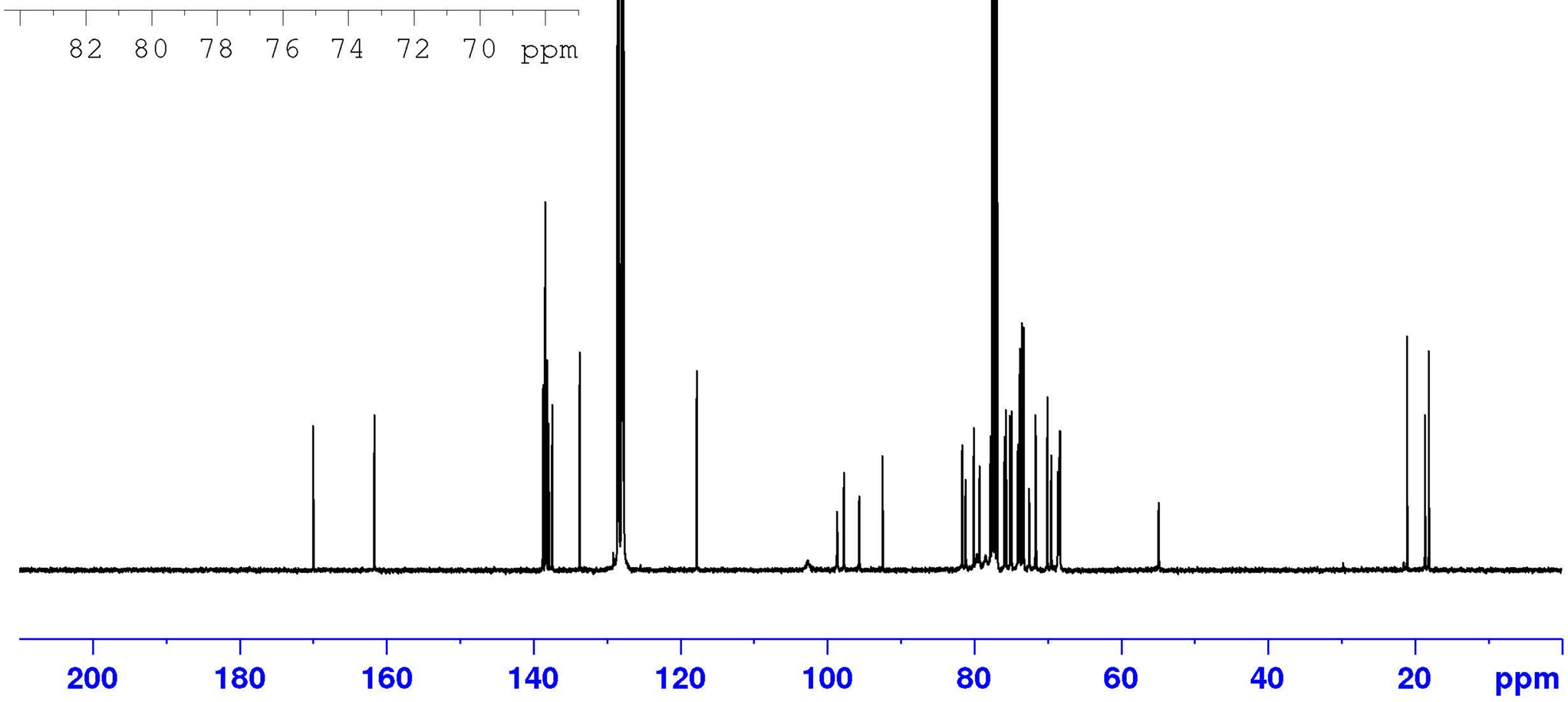
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

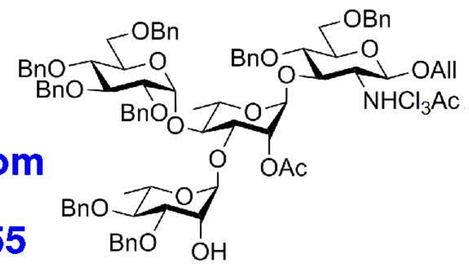
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Compound **8**



ppm

55
60
65
70
75
80
85
90
95
100
105

5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
ppm

F2 - Acquisition Parameters

Date_	20120907
Time	6.24
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	16
DS	16
SWH	3205.128 Hz
FIDRES	1.565004 Hz
AQ	0.3194880 sec
RG	16384
DW	156.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	1.00000000 sec
D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002240 sec

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
PL1	-6.00 dB
SFO1	400.1317466 MHz

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

NUC2	13C
P3	5.90 usec
PL2	-6.00 dB
SFO2	100.6228138 MHz

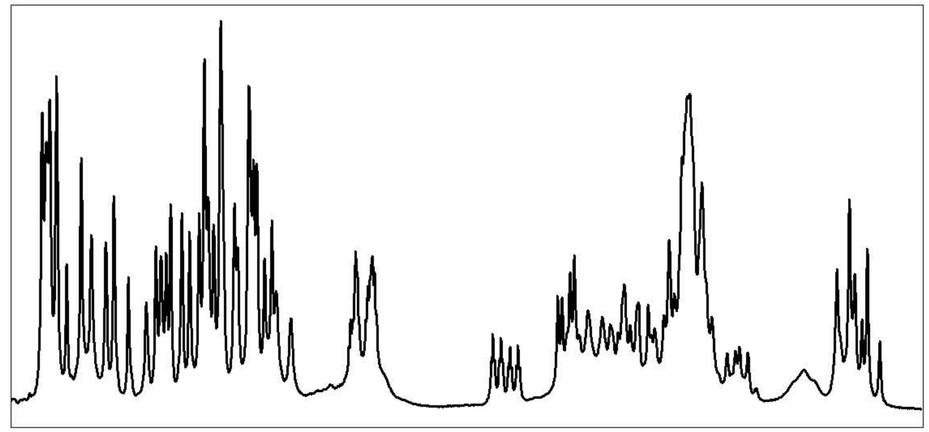
====== GRADIENT CHANNEL =====

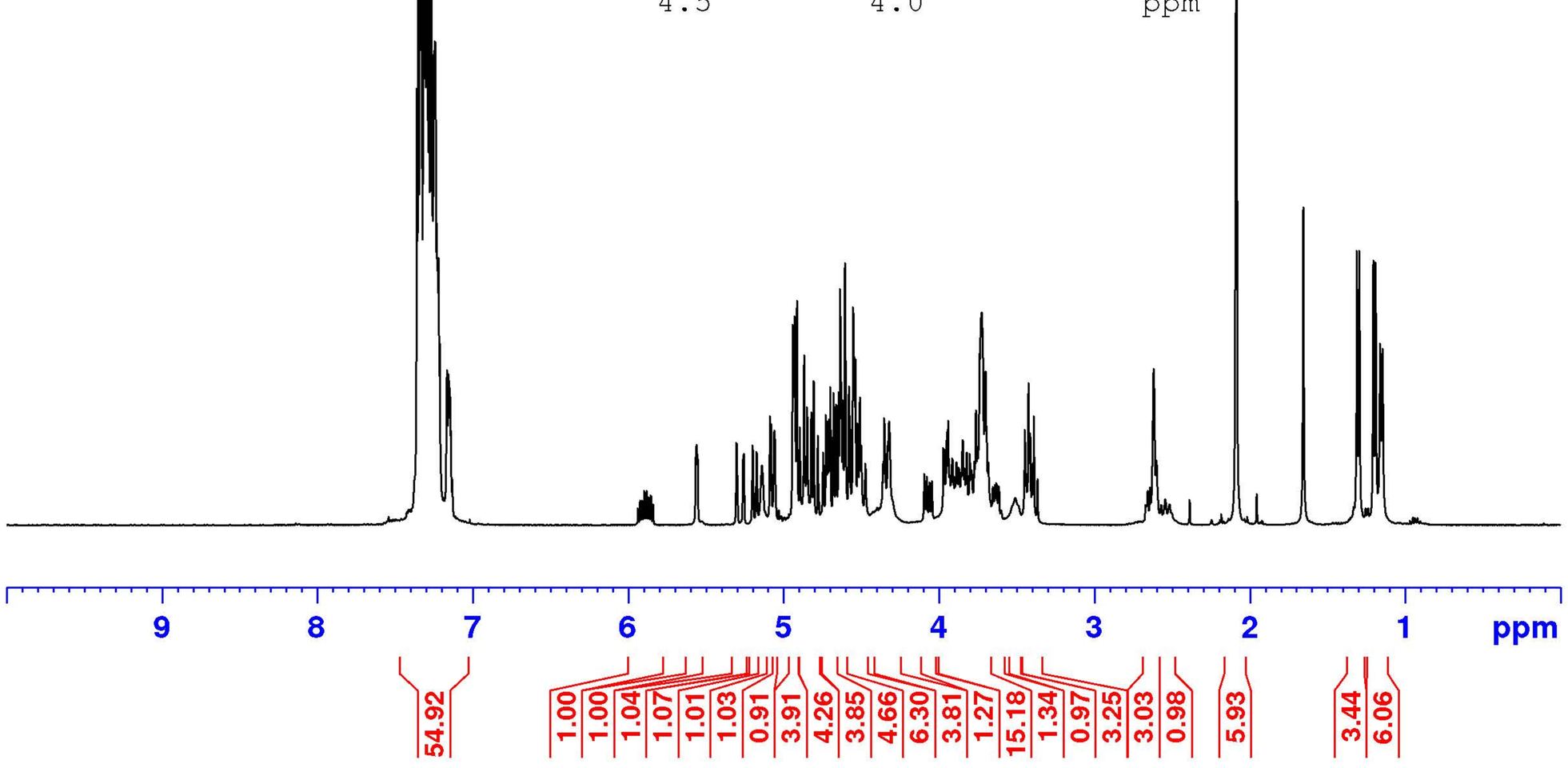
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec

F1 - Acquisition parameters

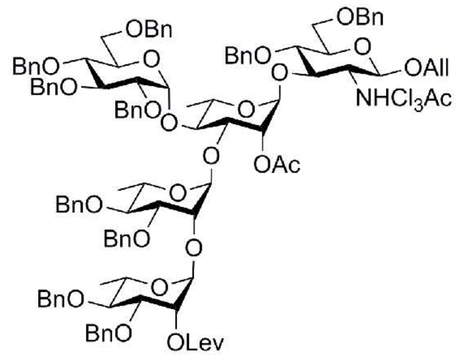
TD	512

7.361 7.350 7.340 7.332 7.326 7.311 7.305 7.299 7.292 7.283 7.272 7.267 7.262 7.256 7.250 7.243 7.232 7.227 7.219 7.214 7.166 4.941 4.933 4.927 4.914 4.868 4.636 4.606 4.553 4.544 4.538 3.738 3.728 3.724 3.701 2.618 2.090 2.084 1.655 1.311 1.295 1.205 1.190 1.161 1.146



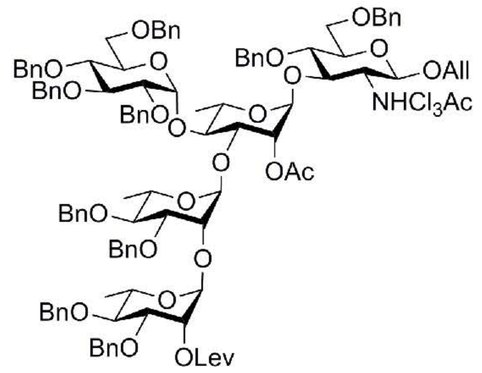


Compound **9**




```
F2 - Acquisition Parameters
Date_              20111214
Time                 17.51
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             8278.146 Hz
FIDRES             0.126314 Hz
AQ              3.9583745 sec
RG                   90.5
DW                60.400 usec
DE                 6.00 usec
TE                999.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.75 usec
PL1                -6.00 dB
SFO1          400.1324708 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300000 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

Compound **9**



F2 - Acquisition Parameters

Date_	20111214
Time	17.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3188.775 Hz
FIDRES	1.557019 Hz
AQ	0.3211264 sec
RG	64
DW	156.800 usec
DE	6.50 usec
TE	999.9 K
D0	0.00000300 sec
D1	1.35786796 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00031360 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1316912 MHz

====== GRADIENT CHANNEL =====

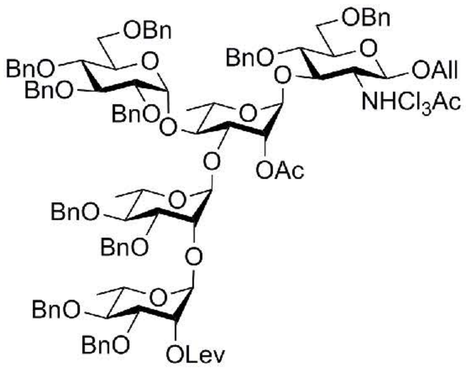
GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	128
SFO1	400.1317 MHz
FIDRES	24.912308 Hz
SW	7.969 ppm
FnMODE	QF

128.36
128.33
128.28
128.18
128.09
128.03
127.97
127.90
127.87
127.82
127.72
127.69
127.66
127.63
127.60
127.54
127.51
127.37
127.26
117.77
99.29
97.81
96.09
81.64
81.20
79.99
79.81
78.80
77.60
77.51
77.28
76.29
75.55
75.34
75.24
74.94
74.80
74.68
74.25
73.96
73.74
73.47
72.95
71.87
71.59
71.57
70.06
69.33
69.10
68.83
68.29
68.26
68.04
56.55
38.06
29.76
28.11
21.07
18.61
18.29
18.04

Compound **9**



F2 - Acquisition Parameters

Date_	20111215
Time	13.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	1500
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

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

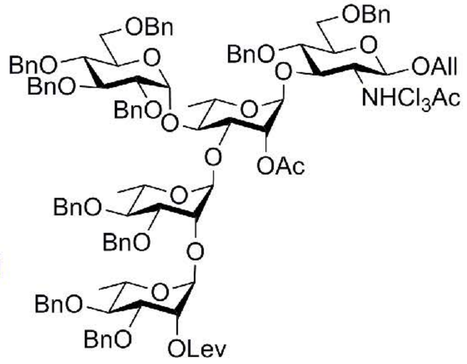
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
P4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

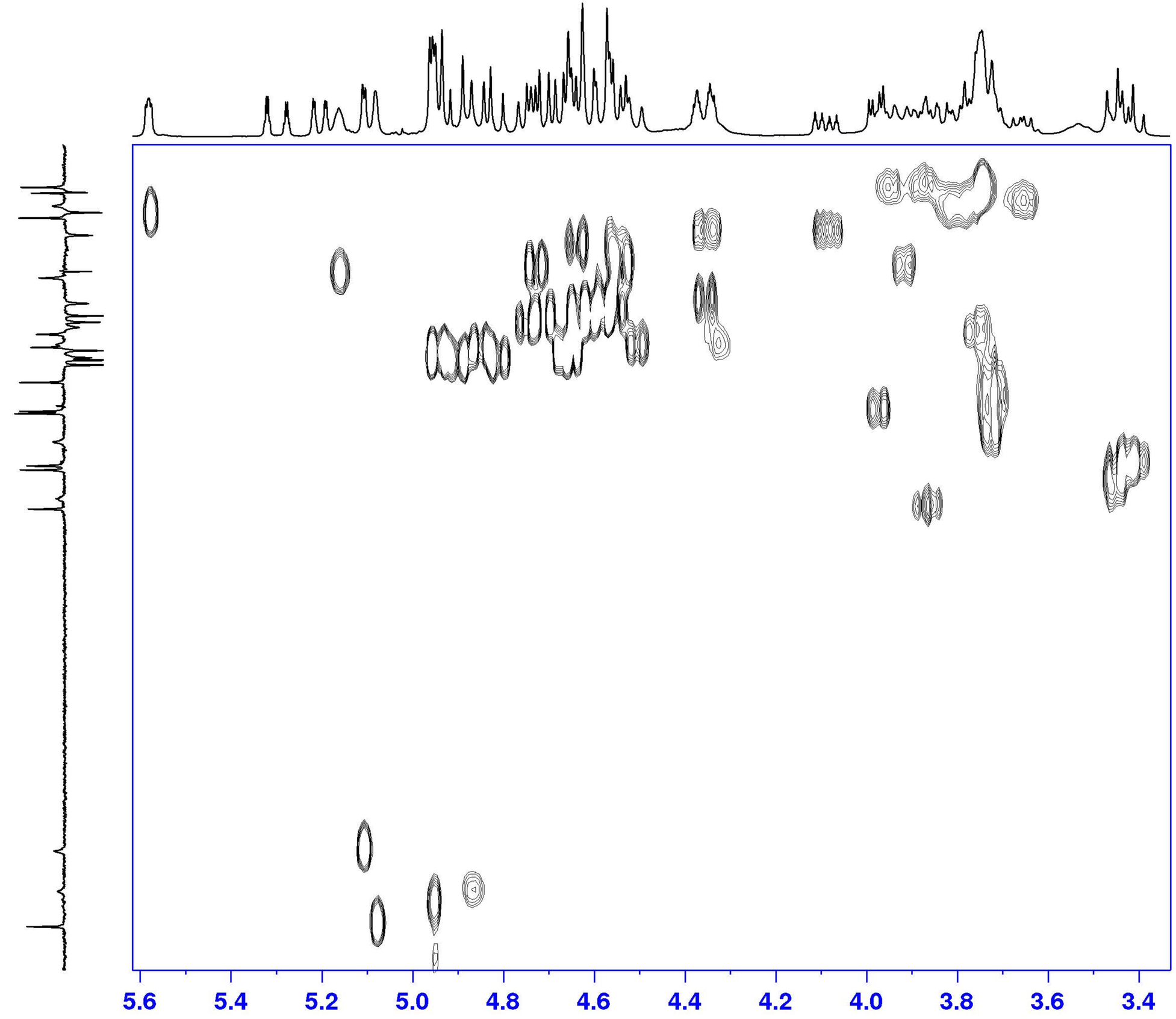
F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

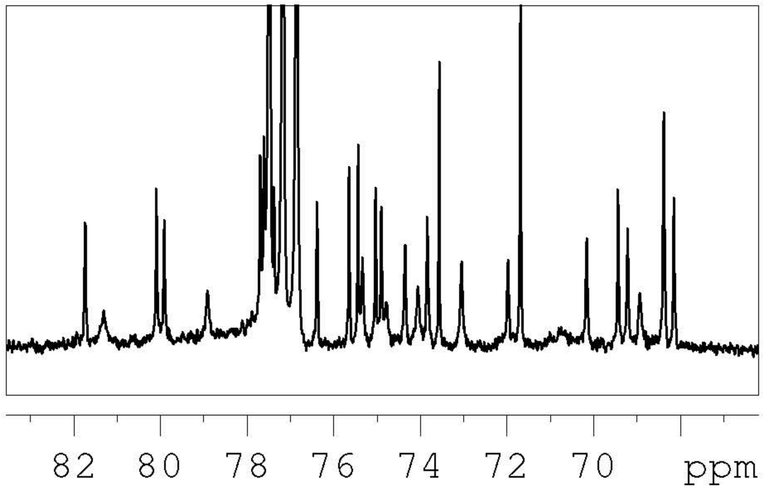
Compound **9**



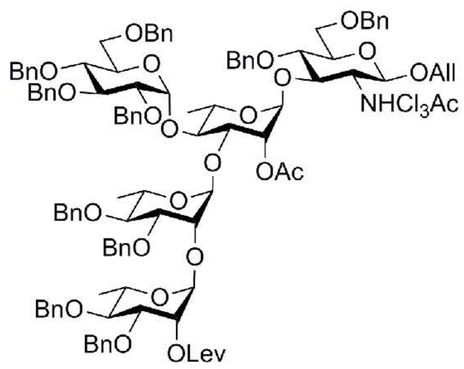



```
Current Data Parameters
NAME                  SS281
EXPNO                    14
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111215
Time                  13.49
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                     2048
SOLVENT                CDCl3
NS                        4
DS                       16
SWH                3140.704 Hz
FIDRES             1.533547 Hz
AQ                0.3260416 sec
RG                     4096
DW                 159.200 usec
DE                   6.00 usec
TE                   999.9 K
CNST2            145.0000000
D0                0.00000300 sec
D1                1.36606097 sec
D4                0.00172414 sec
D11               0.03000000 sec
D16               0.00020000 sec
IN0               0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.75 usec
P2                    13.50 usec
P28                 1000.00 usec
PL1                   -6.00 dB
SFO1           400.1317104 MHz

======== CHANNEL f2 ========
CPDPRG[2                garp
NUC2                     13C
P3                     5.90 usec
P14                  500.00 usec
P24                 2000.00 usec
PCPD2                 75.00 usec
PL0                  120.00 dB
PL2                   -6.00 dB
PL12                  23.00 dB
SFO2           100.6208180 MHz
SP3                    6.74 dB
SP7                    6.74 dB
SPNAM[3] Crp60,0.5,20.1
SPNAM[7]    Crp60comp.4
SPOAL3                0.500
SPOAL7                0.500
SPOFFS3  0 Hz
SPOFFS7  0 Hz

====== GRADIENT CHANNEL =====
GPNAM[1]          SINE.100
GPNAM[2]          SINE.100
GPZ1                 80.00 %
GPZ2                 20.10 %
P16                1000.00 usec
```

171.80
170.05
161.81
138.85
138.76
138.72
138.63
138.35
138.33
138.21
138.09
137.53
133.71
128.60
128.53
128.50
128.47
128.45
128.41
128.37
128.27
128.18
128.12
128.05
127.99
127.95
127.91
127.81
127.78
127.75
127.72
127.68
127.63
127.60
127.46
127.35
117.85
99.38
92.44
81.73
80.08
79.90
77.69
77.60
77.37
76.37
75.64
75.43
75.33
75.02
74.89
74.34
73.83
73.56
71.97
71.68
70.15
69.43
69.20
68.37
68.13
38.15
29.85
28.20
21.16
18.38
18.13

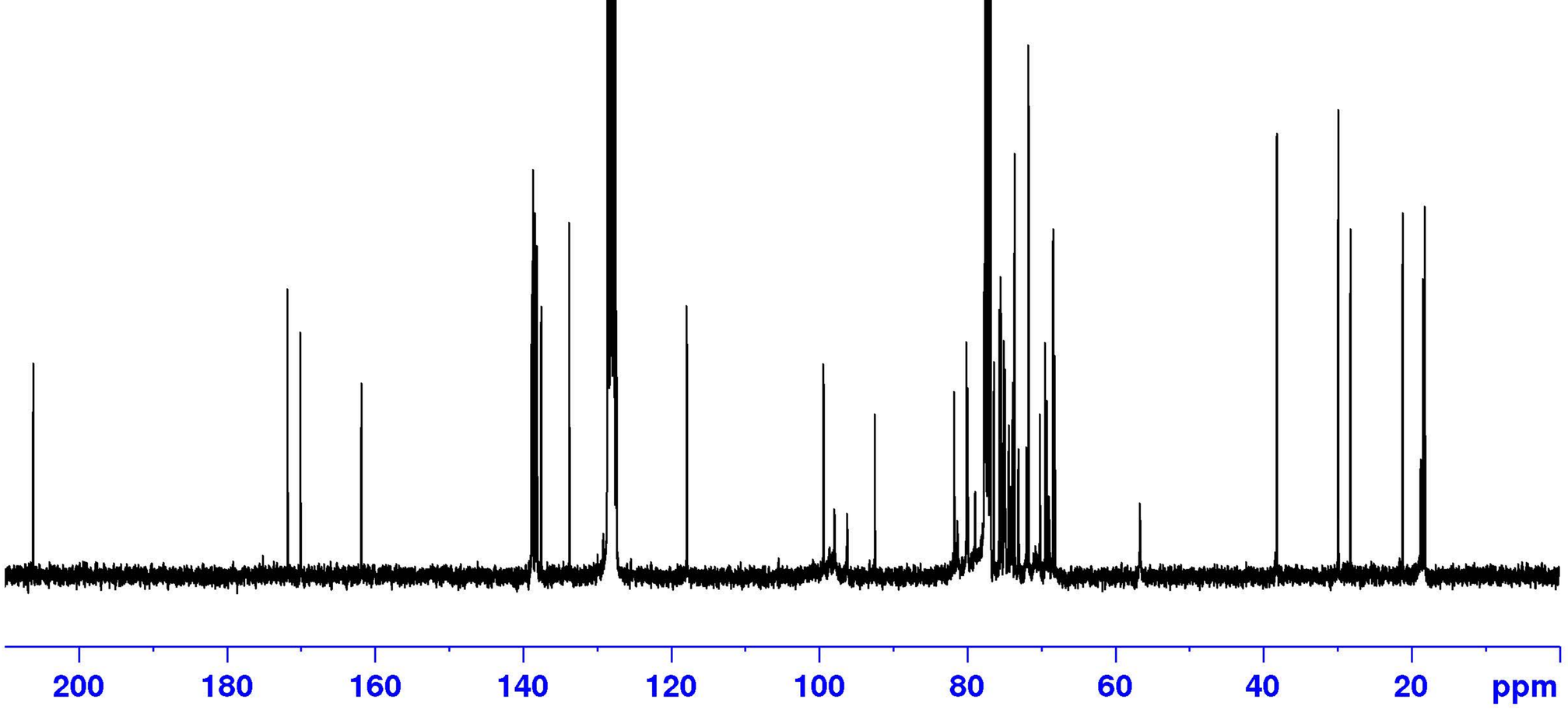


Compound **9**

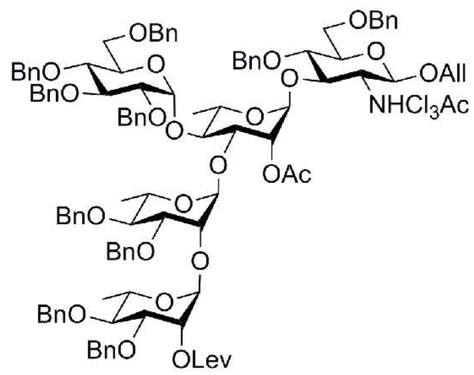


F2 - Acquisition Parameters

Date_	20111216
Time	0.34
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	6000
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	9195.2
DW	19.100 usec
DE	6.50 usec
TE	999.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz
======== CHANNEL f2 ========	
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz



Compound **9**



F2 - Acquisition Parameters

Date_	20111216
Time	0.37
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	16
DS	16
SWH	3140.704 Hz
FIDRES	1.533547 Hz
AQ	0.3260416 sec
RG	16384
DW	159.200 usec
DE	6.00 usec
TE	999.9 K
CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	0.99385601 sec
D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002240 sec

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

NUC1	1H
P1	6.75 usec
P2	13.50 usec
PL1	-6.00 dB
SFO1	400.1316999 MHz

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

NUC2	13C
P3	5.90 usec
PL2	-6.00 dB
SFO2	100.6228138 MHz

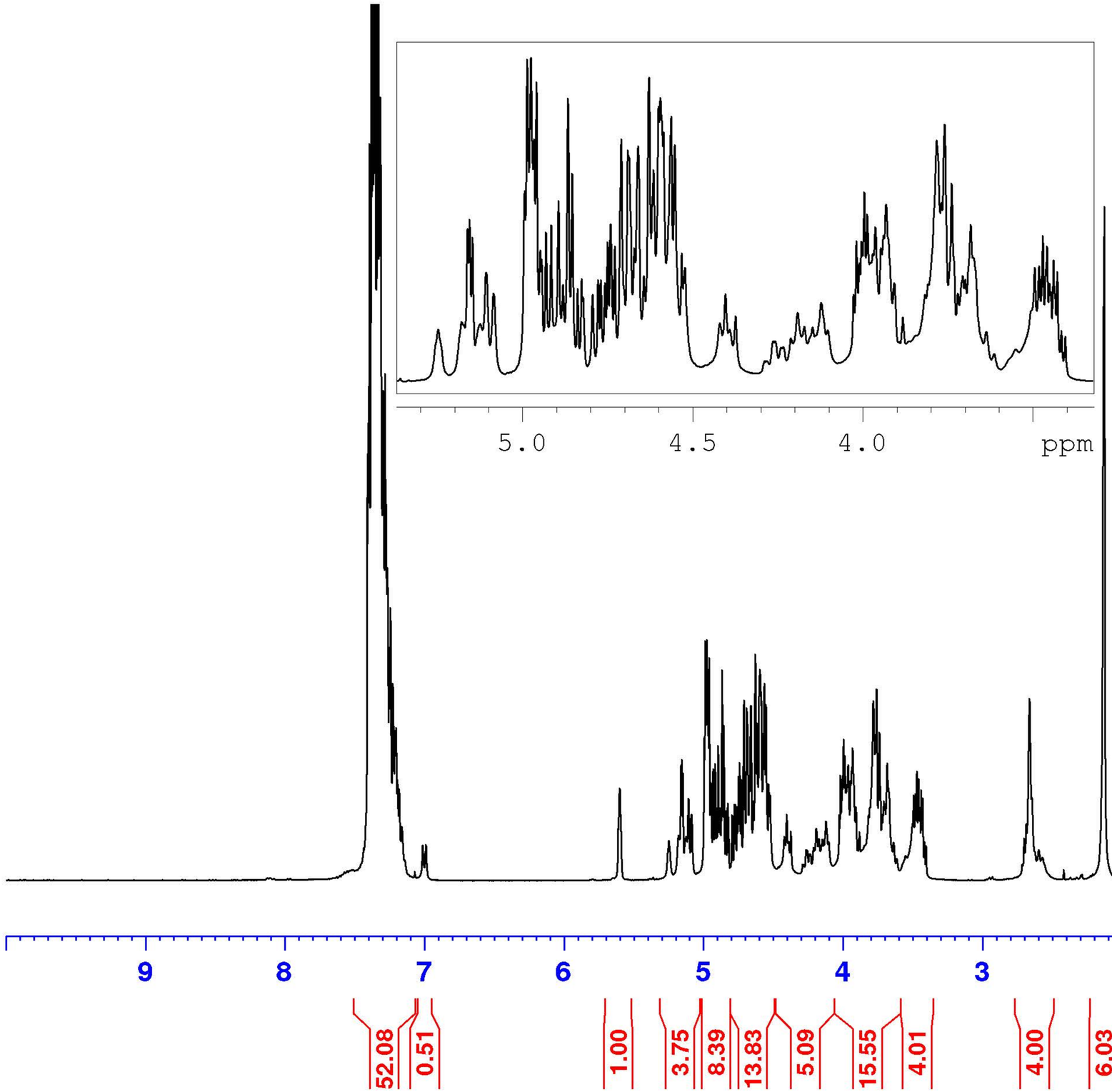
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec

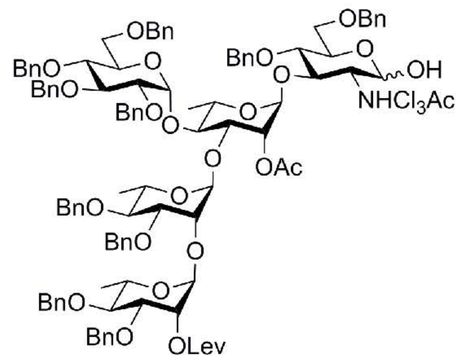
F1 - Acquisition parameters

TD	128
SFO1	100.6228 MHz
FIDRES	174.386154 Hz
SW	221.833 ppm
FnMODE	QF

7.408 7.404 7.399 7.394 7.381 7.370 7.354 7.346 7.337 7.332 7.323 7.322 7.318 7.313 7.295 7.283 7.277 7.264 7.259 7.253 7.244 7.227 4.986 4.975 4.966 4.959 4.866 4.709 4.628 4.600 4.595 4.585 4.563 4.552 3.782 3.759 2.663 2.130 2.125 1.356 1.340 1.325 1.249 1.234 1.222



Compound **10**



F2 - Acquisition Parameters

Date_	20120419
Time	3.39
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	28.5
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

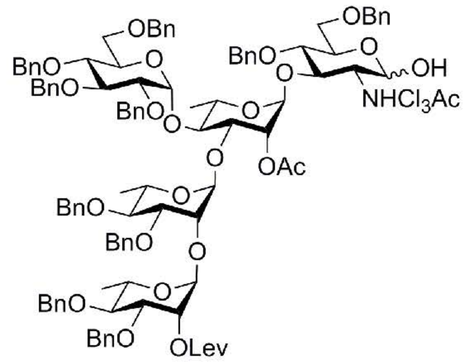
======== CHANNEL f1 ========

NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Compound **10**



F2 - Acquisition Parameters

Date_	20120419
Time	3.41
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	3324.468 Hz
FIDRES	1.623275 Hz
AQ	0.3080192 sec
RG	12.7
DW	150.400 usec
DE	6.50 usec
TE	300.0 K
D0	0.00000300 sec
D1	1.37220395 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00030080 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1318012 MHz

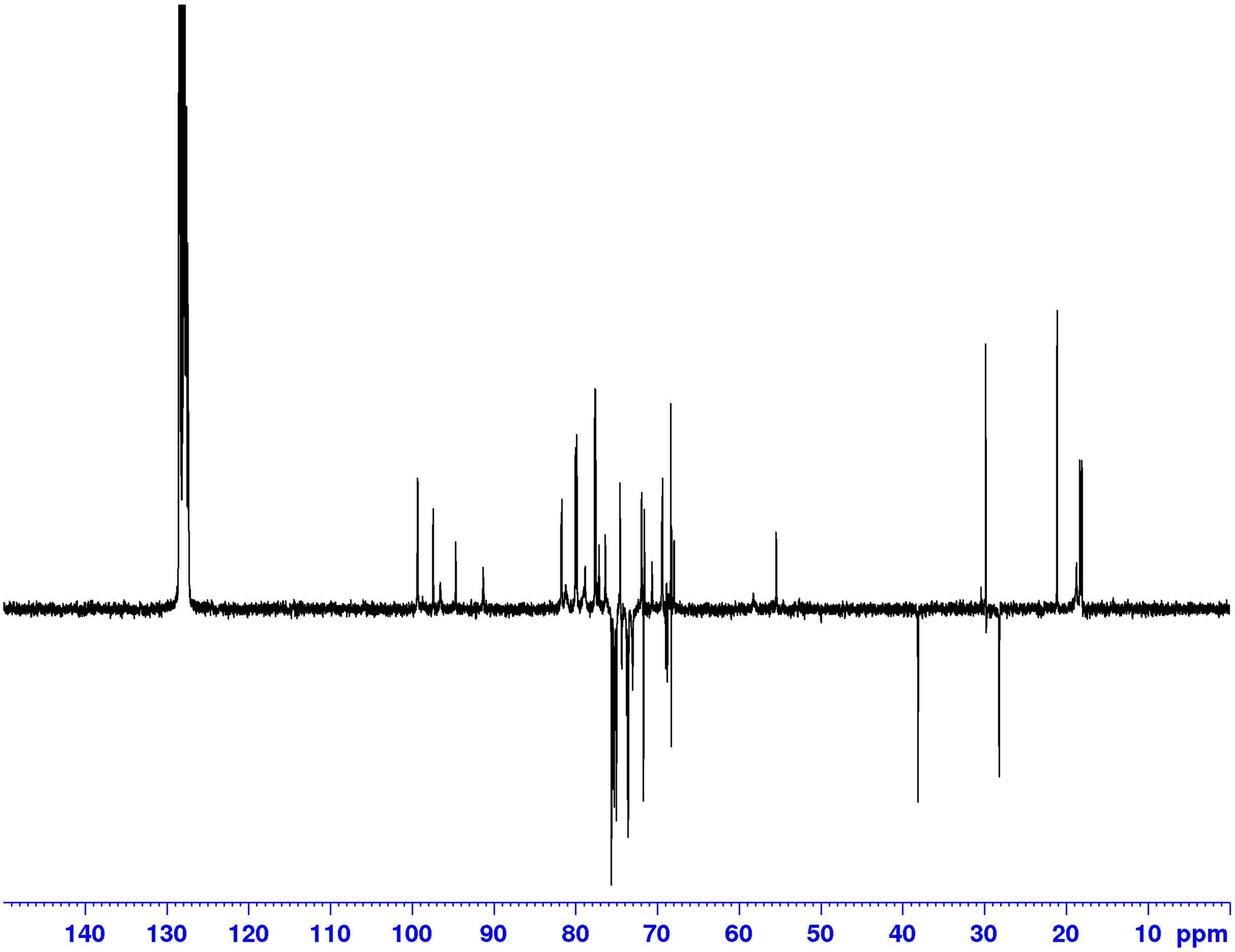
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

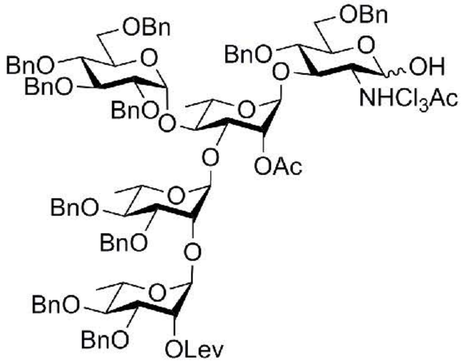
F1 - Acquisition parameters

TD	128
SFO1	400.1318 MHz
FIDRES	25.972406 Hz
SW	8.308 ppm
FnMODE	QF

128.52 128.49 128.45 128.41 128.40 128.37 128.34 128.32 128.21 128.08 128.00 127.92 127.90 127.85 127.80 127.77 127.71 127.67 127.64 127.59 127.42 127.32 99.30 97.40 96.52 94.65 91.29 81.74 81.67 81.20 80.00 79.83 78.80 77.62 77.53 77.34 77.12 76.34 74.54 74.33 73.74 73.00 71.91 71.65 71.57 70.63 69.42 69.35 68.94 68.87 68.76 68.65 68.33 68.23 67.94 58.26 55.43 49.95 30.38 29.80 29.75 21.08 18.69 18.29 18.24 18.08 18.03

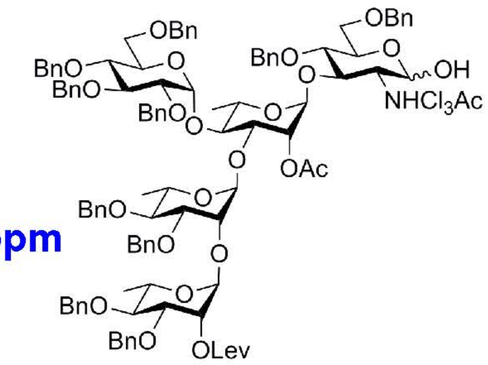


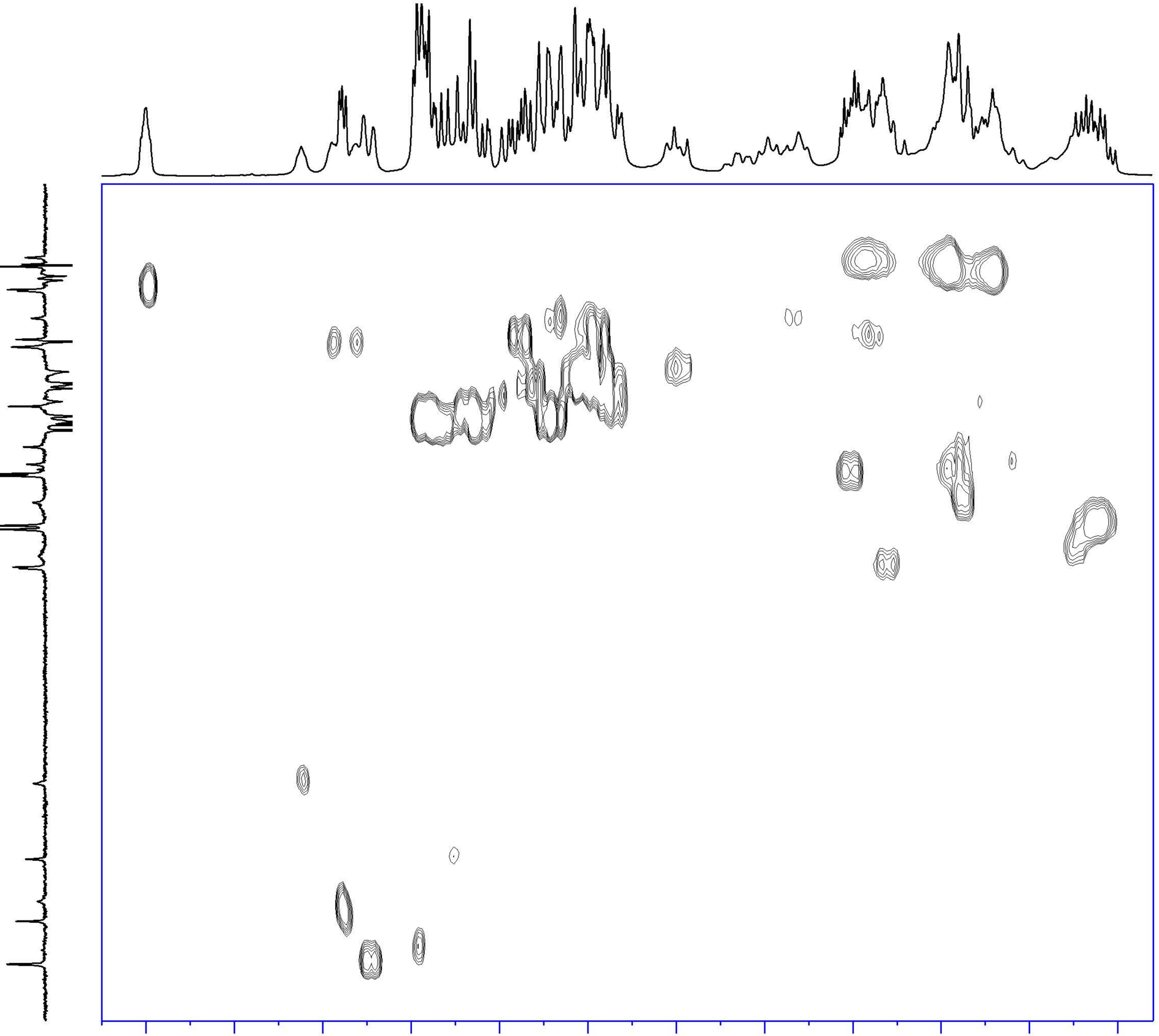
Compound **10**




```
F2 - Acquisition Parameters
Date_           20120419
Time                4.39
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG         deptsp135
TD                  65536
SOLVENT             CDCl3
NS                    900
DS                      4
SWH             23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664256 sec
RG                  16384
DW                 20.850 usec
DE                  6.00 usec
TE                 300.0 K
CNST2         145.0000000
D1              2.00000000 sec
D2              0.00344828 sec
D12             0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                    5.90 usec
P12                2000.00 usec
PL0                 120.00 dB
PL1                  -6.00 dB
SFO1          100.6228298 MHz
SP2                   6.74 dB
SPNAM[2]     Crp60comp.4
SPOAL2               0.500
SPOFFS2   0 Hz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
P3                    6.75 usec
P4                   13.50 usec
PCPD2                80.00 usec
PL2                  -6.00 dB
PL12                 20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127690 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

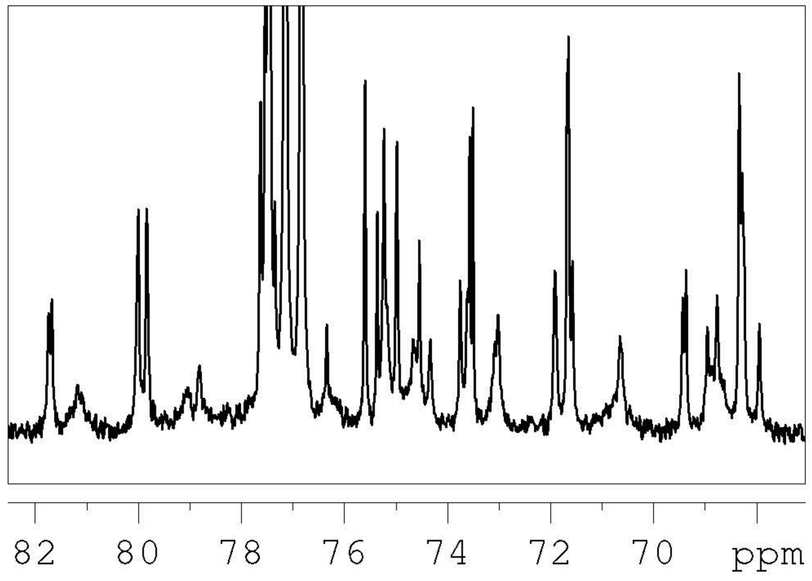
Compound **10**

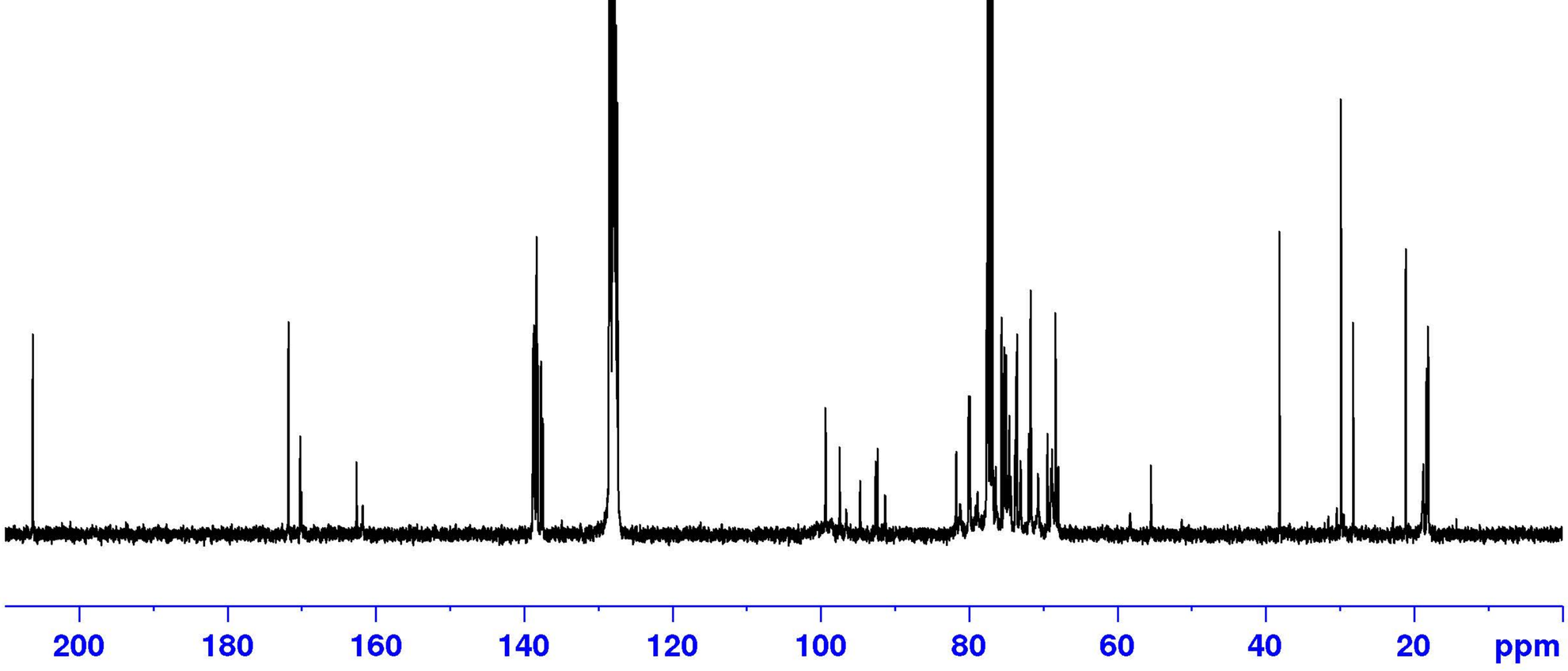




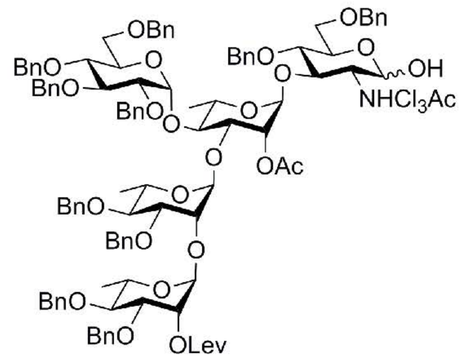
F2 - Acquisition Parameters	
Date_	20120419
Time	4.41
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	2
DS	16
SWH	3324.468 Hz
FIDRES	1.623275 Hz
AQ	0.3080192 sec
RG	5160.6
DW	150.400 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.38449299 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	
======== CHANNEL f1 ========	
NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1318012 MHz
======== CHANNEL f2 ========	
CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

171.75
138.83
138.76
138.66
138.63
138.59
138.55
138.52
138.37
138.27
138.17
138.12
137.74
137.67
137.65
137.45
128.54
128.51
128.48
128.44
128.40
128.36
128.34
128.32
128.21
128.08
127.99
127.92
127.85
127.80
127.77
127.71
127.67
127.64
127.58
127.43
127.32
99.33
80.00
79.84
77.62
77.53
77.46
77.34
77.14
76.82
75.60
75.36
75.23
74.98
74.54
73.56
73.50
71.67
71.64
68.33
68.28
38.11
38.08
29.80
28.17
28.14
21.08
18.29
18.24
18.08
18.03





Compound **10**



F2 - Acquisition Parameters

Date_	20120419
Time	8.44
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4200
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	14596.5
DW	19.100 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

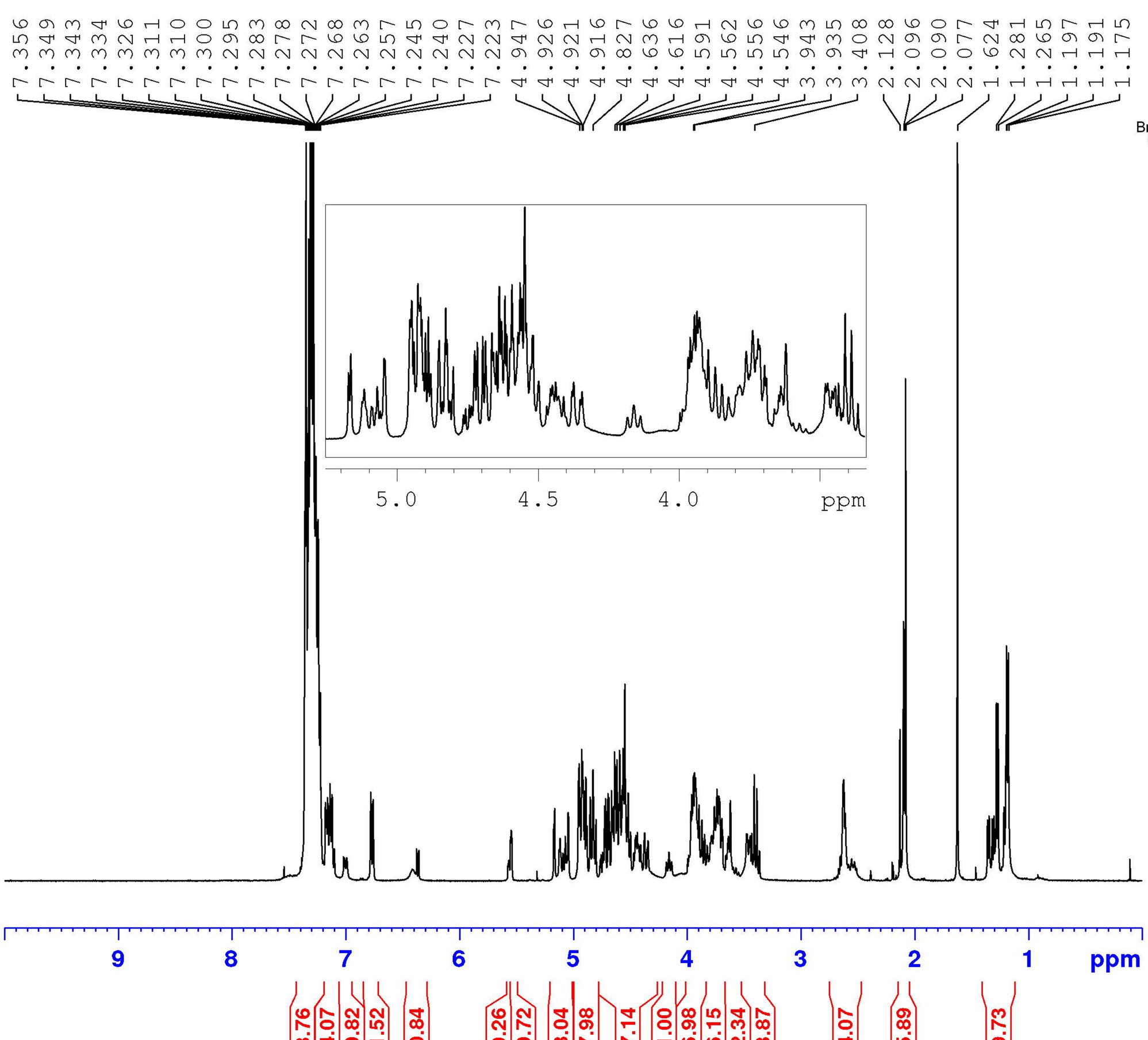
NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

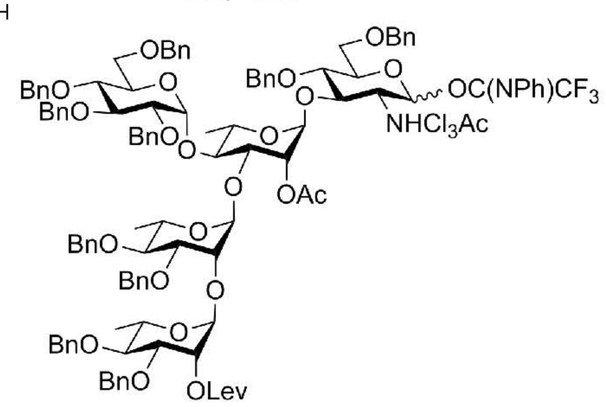
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

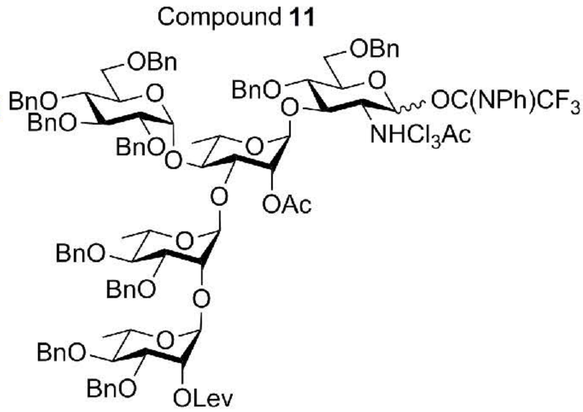


Compound **11**




```
F2 - Acquisition Parameters
Date_              20140729
Time                  12.56
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9583745 sec
RG                      114
DW                 60.400 usec
DE                  6.00 usec
TE                  303.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                  7.10 usec
PL1                -6.00 dB
SFO1          400.1324708 MHz

F2 - Processing parameters
SI                    32768
SF            400.1300000 MHz
WDW                      EM
SSB          0
LB                  0.30 Hz
GB           0
PC                  1.00
```

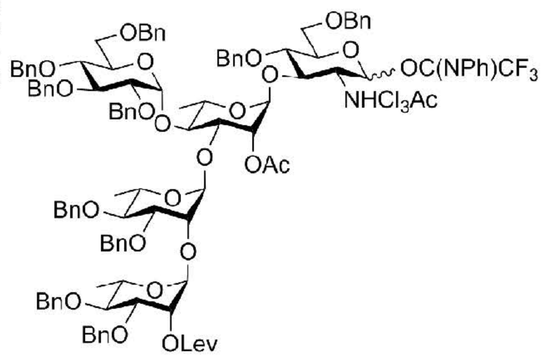
Compound **11**

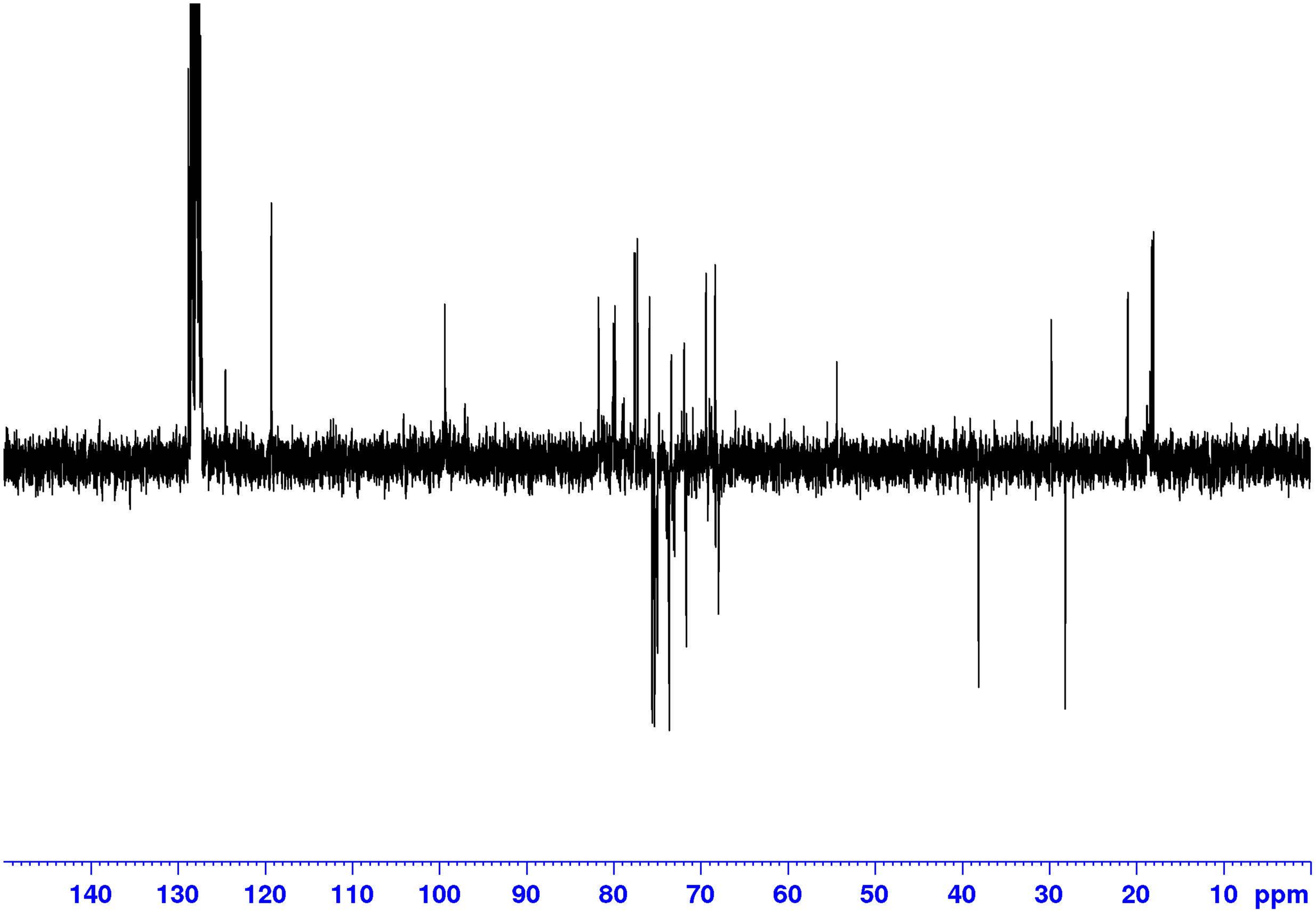



```
F2 - Acquisition Parameters
Date_              20140729
Time                 23.36
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                   2048
SOLVENT              CDCl3
NS                      1
DS                      8
SWH             3140.704 Hz
FIDRES          1.533547 Hz
AQ              0.3260416 sec
RG                     64
DW               159.200 usec
DE                 6.50 usec
TE                303.0 K
D0           0.00000300 sec
D1           1.35377204 sec
D11          0.03000000 sec
D12          0.00002000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00031840 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                 7.10 usec
P1                 7.10 usec
P17             2500.00 usec
PL1               -6.00 dB
PL10               6.13 dB
SFO1        400.1316452 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                    512
SFO1          400.1316 MHz
FIDRES        6.134187 Hz
SW               7.849 ppm
FnMODE                QF
```

128.76
128.52
128.45
128.40
128.35
128.28
128.26
128.14
128.00
127.97
127.94
127.91
127.87
127.83
127.79
127.75
127.73
127.67
127.61
127.55
127.50
127.38
127.31
124.53
119.24
99.33
81.69
79.98
79.80
78.78
77.58
77.51
77.22
75.83
73.34
71.87
69.36
68.95
68.31
68.22
54.32
29.71
20.93
18.38
18.18
17.98

Compound **11**





F2 - Acquisition Parameters

Date_	20140730
Time	1.19
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	1500
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

NUC1	13C
P1	6.25 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	12.00 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

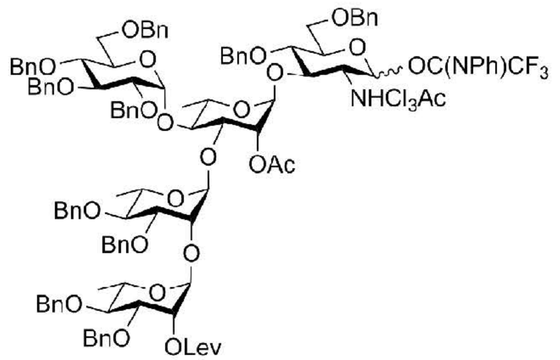
======== CHANNEL f2 ========

CPDPRG[2	waltz16
NUC2	1H
P3	7.10 usec
P4	14.20 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

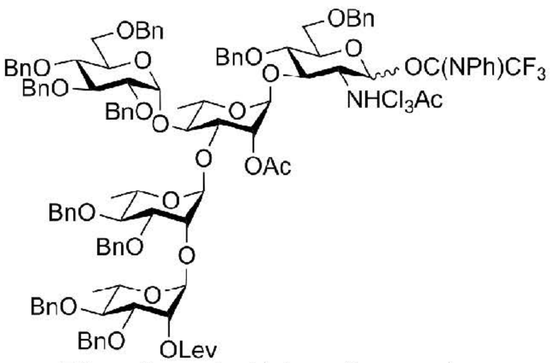
Compound **11**




```
F2 - Acquisition Parameters
Date_              20140730
Time                  1.22
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                    2048
SOLVENT               CDCl3
NS                       4
DS                      16
SWH              3140.704 Hz
FIDRES           1.533547 Hz
AQ              0.3260416 sec
RG                 18390.4
DW                159.200 usec
DE                  6.00 usec
TE                303.0 K
CNST2         145.0000000
D0              0.00000300 sec
D1              1.36606097 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
IN0             0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                    1H
P1                  7.10 usec
P2                 14.20 usec
P28              1000.00 usec
PL1                -6.00 dB
SFO1         400.1316452 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                   13C
P3                  6.25 usec
P14               500.00 usec
P24              2000.00 usec
PCPD2             75.00 usec
PL0               120.00 dB
```

206.15
171.82
170.09
162.99
161.92
143.11
138.91
138.78
138.70
138.67
138.42
138.37
138.26
137.85
137.59
128.91
128.66
128.59
128.54
128.49
128.42
128.40
128.28
128.11
128.09
128.05
128.02
127.97
127.93
127.87
127.81
127.75
127.70
127.64
127.53
127.46
124.69
119.39
99.47
92.30
81.83
80.12
79.94
78.93
77.73
77.65
75.97
75.69
75.39
75.07
74.88
73.79
73.71
73.48
73.39
72.02
71.75
69.50
68.45
68.06
54.47
38.20
29.85
28.26
21.07
18.32
18.12

Compound **11**



F2 - Acquisition Parameters

Date_	20140730
Time	5.09
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3000
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	13004
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	6.25 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

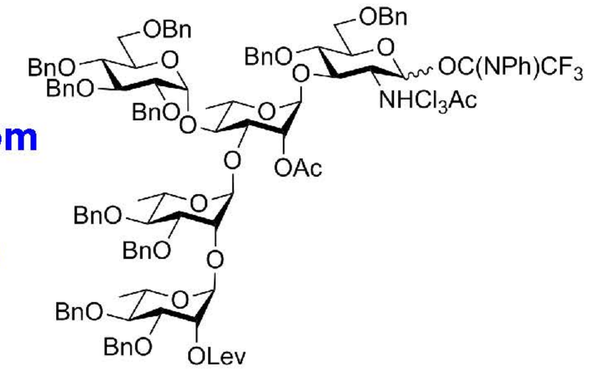
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127544 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

200 180 160 140 120 100 80 60 40 20 ppm

Compound **11**




```
F2 - Acquisition Parameters
Date_            20140730
Time                5.12
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG     hmbcgplpndqf
TD                  2048
SOLVENT             CDCl3
NS                    16
DS                    16
SWH             3140.704 Hz
FIDRES          1.533547 Hz
AQ              0.3260416 sec
RG                  16384
DW               159.200 usec
DE                 6.00 usec
TE                303.0 K
CNST2         145.0000000
CNST13         10.0000000
D0             0.00000300 sec
D1             0.99385601 sec
D2             0.00344828 sec
D6             0.05000000 sec
D16            0.00020000 sec
IN0            0.00002240 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.10 usec
P2                14.20 usec
PL1               -6.00 dB
SFO1         400.1316452 MHz

======== CHANNEL f2 ========
NUC2                 13C
P3                 6.25 usec
PL2               -6.00 dB
SFO2         100.6228138 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPNAM[3]        SINE.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   512
SFO1          100.6228 MHz
FIDRES       43.596539 Hz
SW             221.833 ppm
FnMODE              QF
```

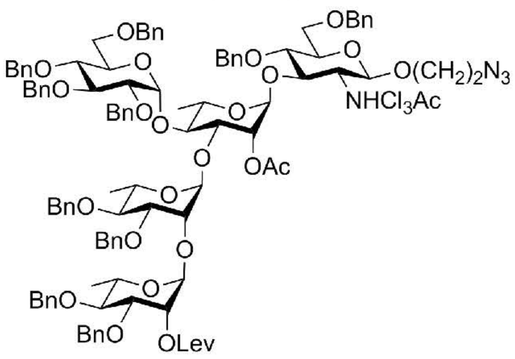
ppm

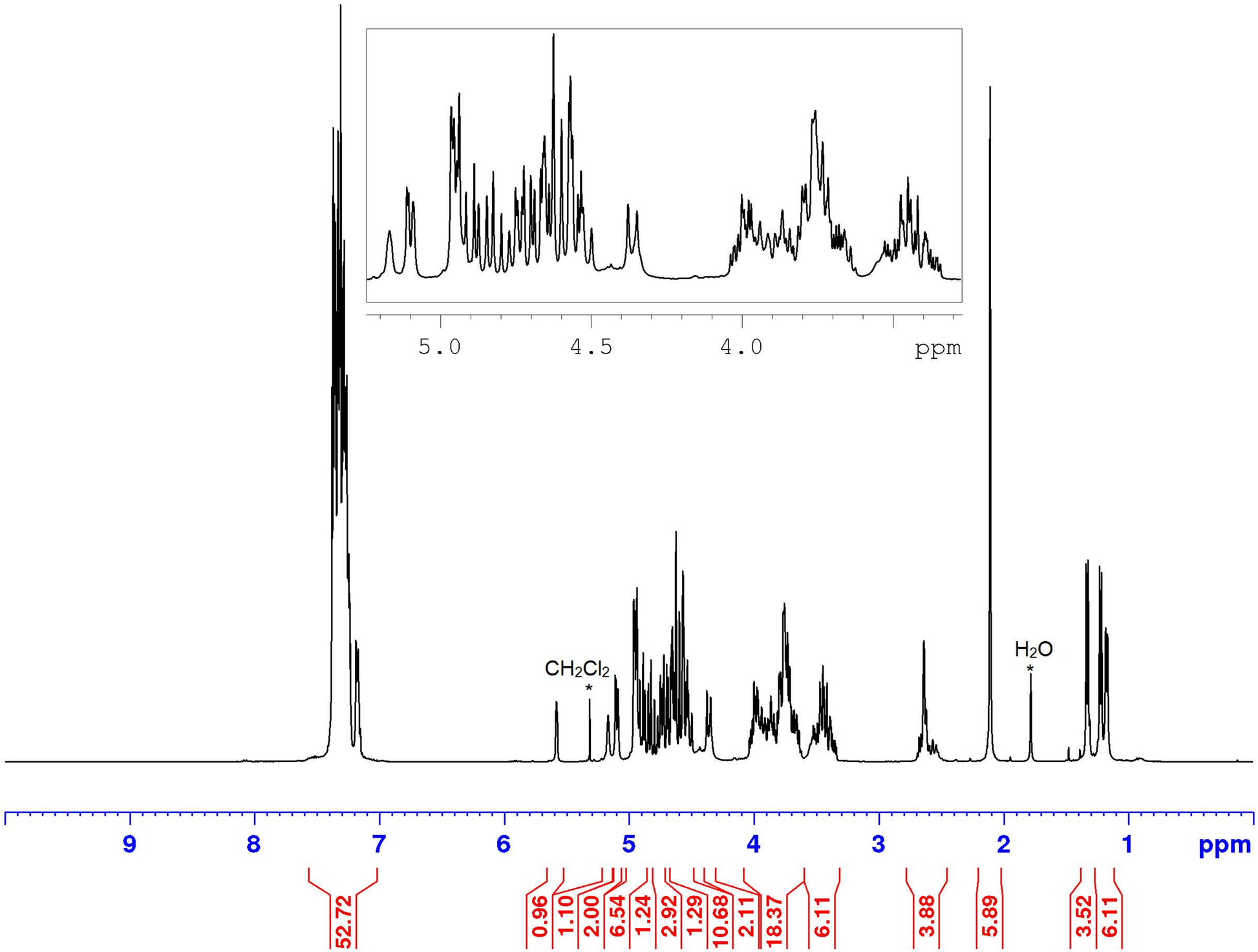
68 70 72 74 76 78 80 82

5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 ppm

7.381 7.370 7.361 7.354 7.346 7.336 7.331 7.315 7.311 7.303 7.299 7.291 7.286 7.283 7.280 7.274 7.261 7.252 7.246 7.239 7.233 7.189 7.170 4.964 4.955 4.945 4.938 4.660 4.655 4.625 4.598 4.573 4.569 4.562 3.766 3.756 3.731 2.640 2.110 1.339 1.324 1.232 1.216 1.183 1.168

Compound **12**





F2 - Acquisition Parameters

Date_	20120619
Time	17.49
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	35.9
DW	60.400 usec
DE	6.00 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1

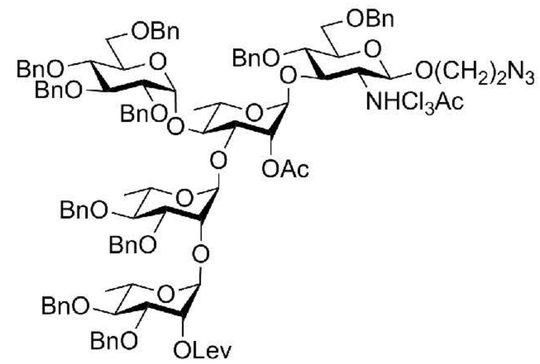
======== CHANNEL f1 ========

NUC1	1H
P1	6.75 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

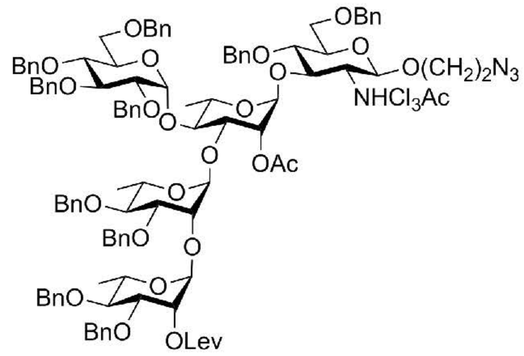
Compound **12**




```
F2 - Acquisition Parameters
Date_            20120619
Time                17.51
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                  2048
SOLVENT             CDCl3
NS                     1
DS                     8
SWH              3289.474 Hz
FIDRES           1.606188 Hz
AQ               0.3112960 sec
RG                    18
DW                152.000 usec
DE                  6.50 usec
TE                299.3 K
D0             0.00000300 sec
D1             1.36810803 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00030400 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                  6.75 usec
P1                  6.75 usec
P17              2500.00 usec
PL1                -6.00 dB
PL10                6.57 dB
SFO1          400.1317587 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                    128
SFO1           400.1318 MHz
FIDRES        25.699013 Hz
SW                8.221 ppm
FnMODE                QF
```

128.42 128.40 128.37 128.36 128.32 128.22 128.15 128.08 128.00 127.95 127.91 127.86 127.78 127.75 127.71 127.64 127.59 127.55 127.39 127.29 99.27 98.81 96.12 81.67 81.27 80.04 79.82 78.83 77.62 77.55 77.36 76.27 75.58 75.38 75.29 74.97 74.72 74.41 74.10 73.79 73.51 72.98 71.89 71.65 71.62 70.60 69.37 69.04 68.90 68.33 68.30 68.25 68.09 56.69 50.70 38.08 29.79 28.15 21.11 18.63 18.33 18.10

Compound **12**



F2 - Acquisition Parameters

Date_	20120620	
Time	1.23	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	deptsp135	
TD	65536	
SOLVENT	CDCl3	
NS	800	
DS	4	
SWH	23980.814	Hz
FIDRES	0.365918	Hz
AQ	1.3664256	sec
RG	16384	
DW	20.850	usec
DE	6.00	usec
TE	299.7	K
CNST2	145.0000000	
D1	2.00000000	sec
D2	0.00344828	sec
D12	0.00002000	sec
TD0	1	

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

NUC1	13C	
P1	5.90	usec
P12	2000.00	usec
PL0	120.00	dB
PL1	-6.00	dB
SFO1	100.6228298	MHz
SP2	6.74	dB
SPNAM[2]	Crp60comp.4	
SPOAL2	0.500	
SPOFFS2	0 Hz	

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

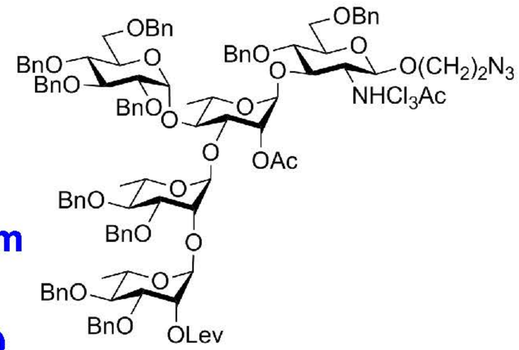
CPDPRG[2	waltz16	
NUC2	1H	
P3	6.75	usec
P4	13.50	usec
PCPD2	80.00	usec
PL2	-6.00	dB
PL12	20.00	dB
SFO2	400.1316005	MHz

F2 - Processing parameters

SI	32768	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Compound **12**



ppm

50

55

60

65

70

75

80

85

90

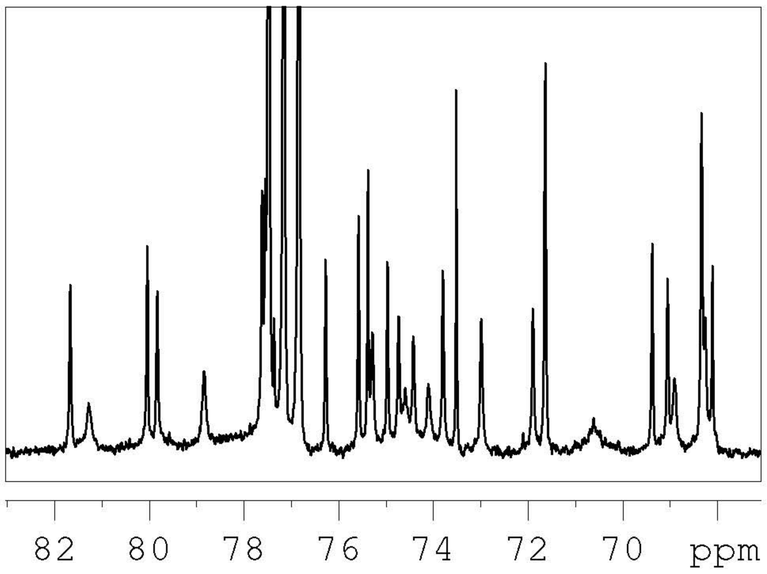
95

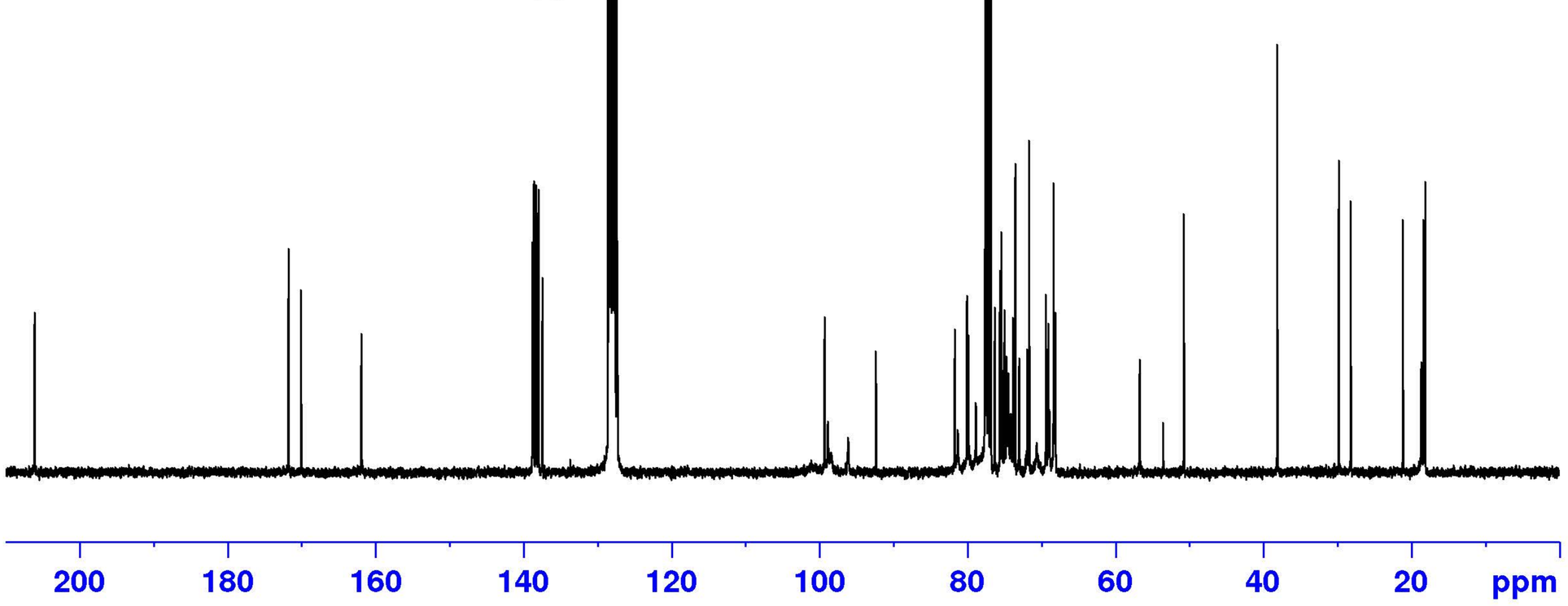
100

F2 - Acquisition Parameters	
Date_	20120620
Time	1.26
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	3360.215 Hz
FIDRES	1.640730 Hz
AQ	0.3047424 sec
RG	3649.1
DW	148.800 usec
DE	6.00 usec
TE	299.7 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.38654101 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	
======== CHANNEL f1 ========	
NUC1	1H
P1	6.75 usec
P2	13.50 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1317920 MHz
======== CHANNEL f2 ========	
CPDPRG[2	garp
NUC2	13C
P3	5.90 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	23.00 dB
SFO2	100.6208180 MHz
SP3	6.74 dB
SP7	6.74 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

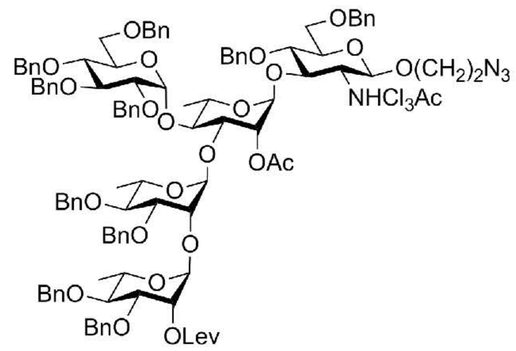
5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 ppm

171.74
170.04
161.89
138.80
138.71
138.66
138.59
138.58
138.30
138.27
138.15
137.96
137.42
128.56
128.48
128.42
128.40
128.36
128.31
128.22
128.15
128.08
128.00
127.95
127.91
127.86
127.77
127.75
127.71
127.63
127.58
127.55
127.39
127.29
99.27
92.33
81.67
80.04
79.83
77.62
77.55
77.47
77.36
77.16
76.84
76.27
75.58
75.38
74.96
74.72
73.79
73.51
72.98
71.89
71.62
69.37
69.04
68.32
68.25
68.09
50.70
38.08
29.78
28.15
21.11
18.33
18.10





Compound **12**



F2 - Acquisition Parameters

Date_	20120620
Time	5.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	4500
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	16384
DW	19.100 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

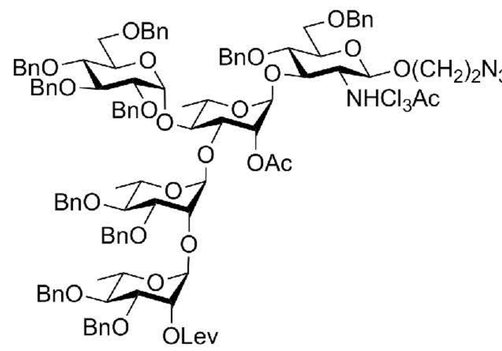
======== CHANNEL f2 ========

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

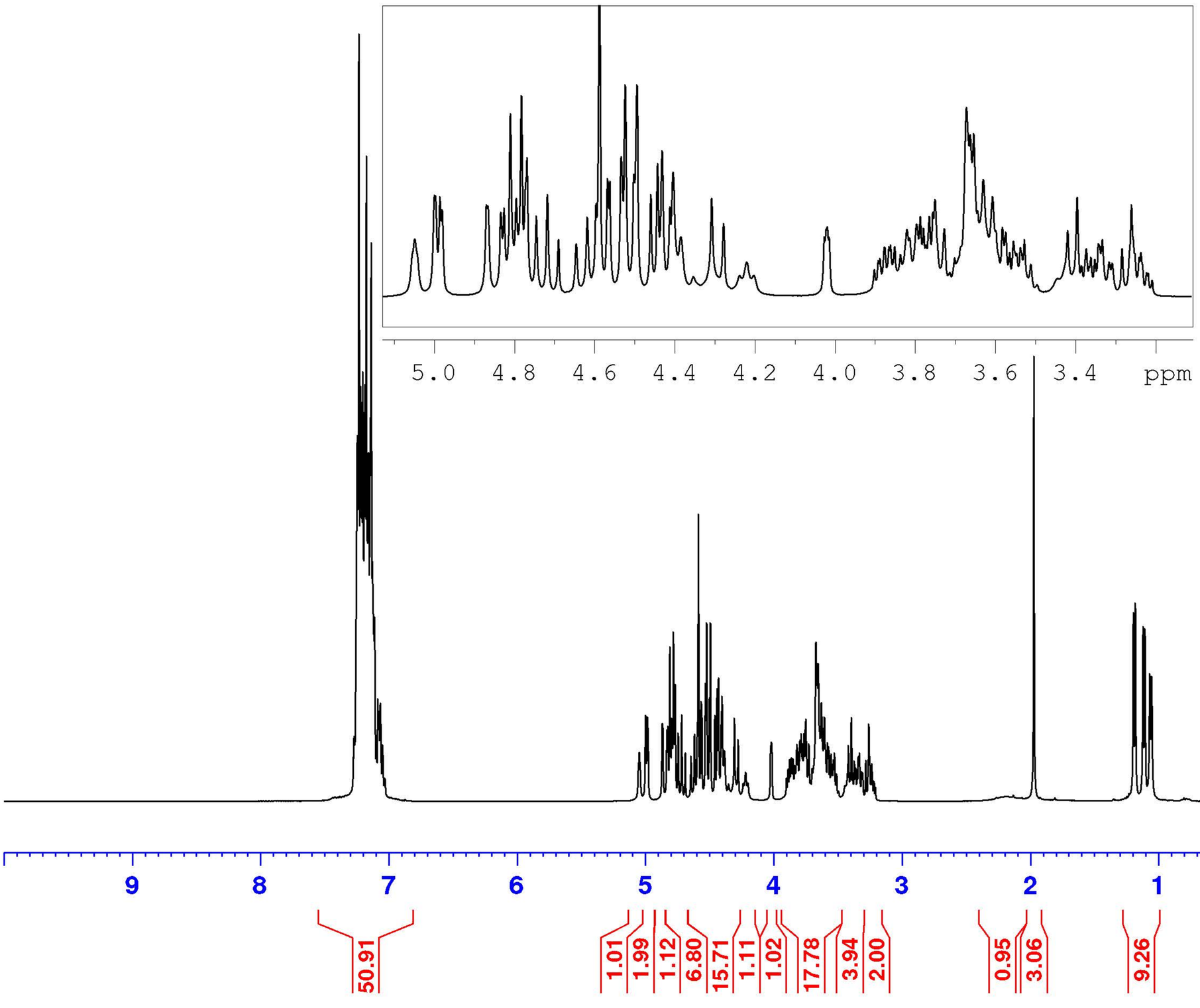
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **12**

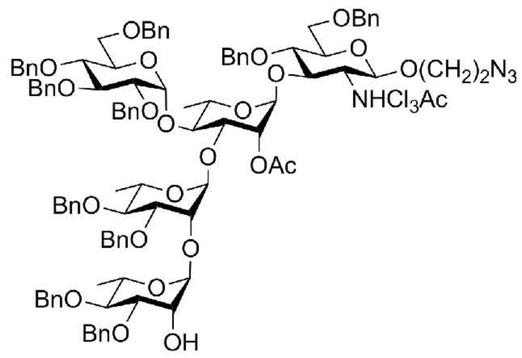


F2 - Acquisition Para	
Date_	201206
Time	5.
INSTRUM	spe
PROBHD	5 mm PABBO B
PULPROG	hmbcgplpnd
TD	20
SOLVENT	CDC
NS	
DS	
SWH	3360.2
FIDRES	1.6407
AQ	0.30474
RG	163
DW	148.8
DE	6.
TE	299
CNST2	145.00000
CNST13	10.00000
D0	0.000003
D1	1.014335
D2	0.003448
D6	0.050000
D16	0.000200
IN0	0.000022
======== CHANNEL f1 =	
NUC1	
P1	6.
P2	13.
PL1	-6.
SFO1	400.13179
======== CHANNEL f2 =	
NUC2	1
P3	5.
PL2	-6.
SFO2	100.62281
====== GRADIENT CHANN	
GPNAM[1]	SINE.1
GPNAM[2]	SINE.1
GPNAM[3]	SINE.1
GPZ1	50.
GPZ2	30.
GPZ3	40.
P16	1000.

7.250 7.246 7.235 7.232 7.220 7.213 7.202 7.188 7.184 7.175 7.162 7.157 7.148 7.140 7.136 7.129 7.123 7.117 7.112 7.086 7.066 4.809 4.782 4.768 4.587 4.567 4.561 4.532 4.522 4.501 4.493 4.442 4.430 4.403 3.671 3.661 3.652 3.628 1.971 1.196 1.181 1.121 1.106 1.068 1.053

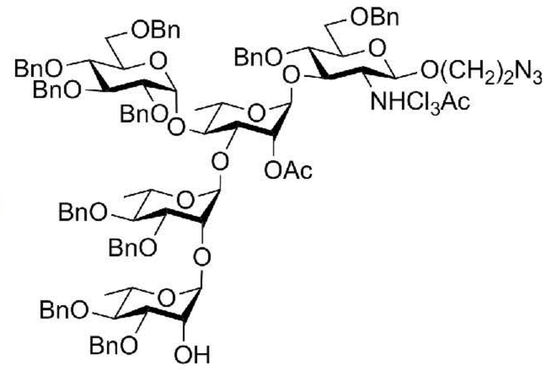


Compound **13**




```
F2 - Acquisition Parameters
Date_              20120822
Time                 18.51
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8278.146 Hz
FIDRES             0.126314 Hz
AQ                3.9583745 sec
RG                     35.9
DW                 60.400 usec
DE                  6.00 usec
TE                  303.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.75 usec
PL1                -6.00 dB
SFO1          400.1324708 MHz

F2 - Processing parameters
SI                    32768
SF            400.1300586 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

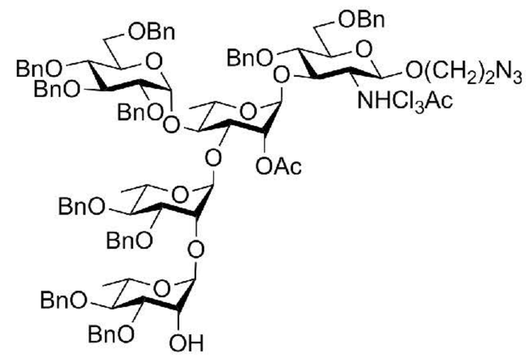
Compound **13**




```
F2 - Acquisition Parameters
Date_              20120822
Time                 18.53
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG        cosygpppqf
TD                    2048
SOLVENT               CDCl3
NS                       1
DS                       8
SWH               3360.215 Hz
FIDRES             1.640730 Hz
AQ                0.3047424 sec
RG                      16
DW                 148.800 usec
DE                   6.50 usec
TE                  303.0 K
D0              0.00000300 sec
D1              1.37425196 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00029760 sec

======== CHANNEL f1 ========
NUC1                     1H
P0                    6.75 usec
P1                    6.75 usec
P17                2500.00 usec
PL1                  -6.00 dB
PL10                  6.57 dB
SFO1          400.1317973 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPZ1                10.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1              400.1318 MHz
FIDRES           26.251680 Hz
SW                    8.398 ppm
FnMODE                   QF
```

128.33
128.31
128.00
127.96
127.92
127.90
127.87
127.81
127.79
127.73
127.71
127.63
127.58
127.55
127.52
127.44
127.35
101.00
98.82
98.37
96.02
81.67
81.24
80.13
80.06
79.57
78.83
77.59
77.29
76.22
75.55
75.33
75.26
75.03
74.70
74.50
74.39
74.05
73.78
73.49
73.11
72.08
71.93
71.61
70.60
69.11
68.86
68.29
68.22
68.15
68.01
56.46
50.68
21.05
18.63
18.20
18.08

Compound **13**



F2 - Acquisition Parameters

Date_	20120823
Time	6.21
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	800
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	6.74 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

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

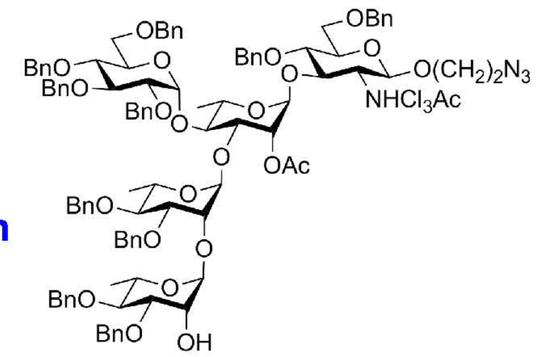
CPDPRG[2	waltz16
NUC2	1H
P3	6.75 usec
P4	13.50 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

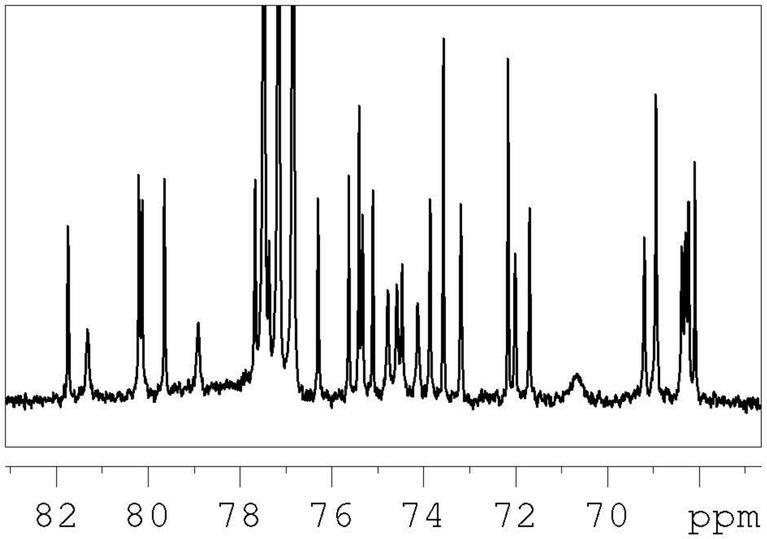
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

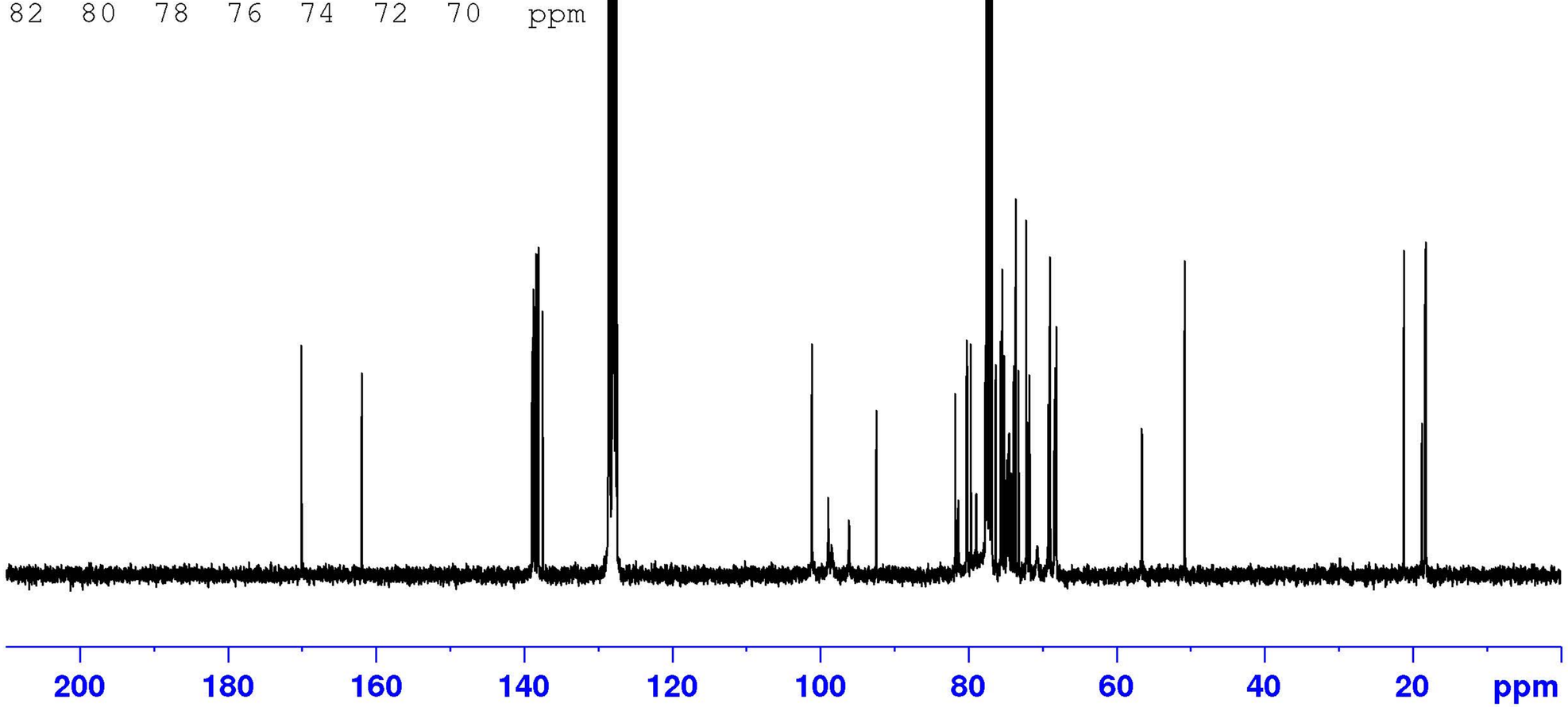
Compound **13**



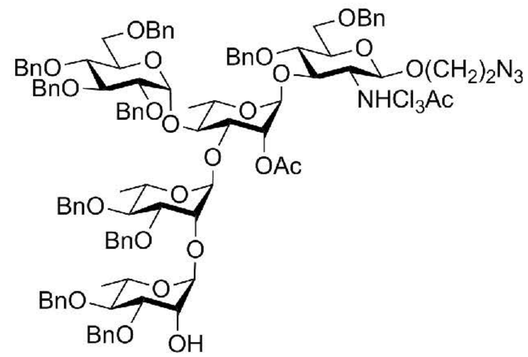

```
F2 - Acquisition Parameters
Date_            20120823
Time                6.23
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                  2048
SOLVENT             CDCl3
NS                     2
DS                    16
SWH             2893.519 Hz
FIDRES          1.412851 Hz
AQ             0.3538944 sec
RG                  6502
DW               172.800 usec
DE                 6.00 usec
TE                303.0 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.33738899 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
IN0            0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                  1H
P1                 6.75 usec
P2                13.50 usec
P28             1000.00 usec
PL1               -6.00 dB
SFO1         400.1317677 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                 5.90 usec
P14              500.00 usec
P24             2000.00 usec
PCPD2             75.00 usec
PL0              120.00 dB
PL2               -6.00 dB
PL12              23.00 dB
SFO2         100.6208180 MHz
SP3                6.74 dB
SP7                6.74 dB
SPNAM[3] Crp60,0.5,20.1
SPNAM[7]    Crp60comp.4
SPOAL3               0.500
SPOAL7               0.500
SPOFFS3  0 Hz
SPOFFS7  0 Hz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec
```

161.90
138.90
138.79
138.74
138.67
138.62
138.42
138.34
138.17
138.04
137.45
128.61
128.52
128.48
128.45
128.40
128.38
128.07
128.03
128.00
127.94
127.88
127.87
127.80
127.78
127.70
127.65
127.62
127.59
127.51
127.43
101.08
92.38
81.74
80.20
80.13
79.64
77.67
77.48
77.36
77.16
76.84
76.29
75.63
75.41
75.33
75.10
74.58
74.46
73.85
73.56
73.18
72.16
72.00
71.69
69.19
68.93
68.37
68.29
68.22
68.08
56.53
50.75
21.12
18.70
18.28
18.16





Compound **13**



F2 - Acquisition Parameters

Date_	20120823
Time	9.23
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	11585.2
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

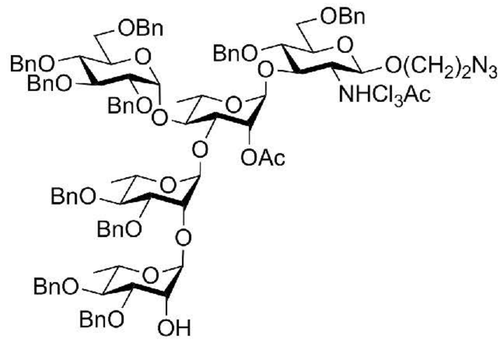
======== CHANNEL f2 ========

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

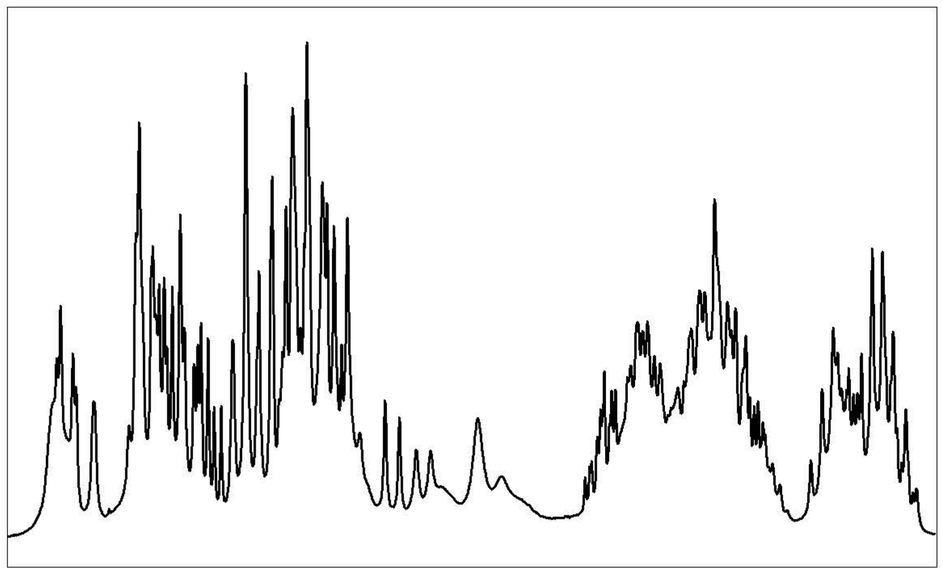
SI	32768
SF	100.6127617 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **13**

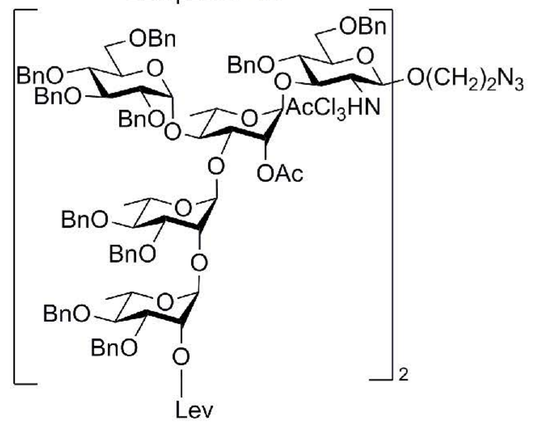


F2 - Acquisition Para	
Date_	201208
Time	9.
INSTRUM	spe
PROBHD	5 mm PABBO B
PULPROG	hmbcgplpnd
TD	20
SOLVENT	CDC
NS	
DS	
SWH	2893.5
FIDRES	1.4128
AQ	0.35389
RG	163
DW	172.8
DE	6.
TE	303
CNST2	145.00000
CNST13	10.00000
D0	0.000003
D1	0.965183
D2	0.003448
D6	0.050000
D16	0.000200
IN0	0.000022
======== CHANNEL f1 =	
NUC1	
P1	6.
P2	13.
PL1	-6.
SFO1	400.13176
======== CHANNEL f2 =	
NUC2	1
P3	5.
PL2	-6.
SFO2	100.62281
====== GRADIENT CHANN	
GPNAM[1]	SINE.1
GPNAM[2]	SINE.1
GPNAM[3]	SINE.1
GPZ1	50.
GPZ2	30.
GPZ3	40.
P16	1000.

7.35 7.34 7.34 7.32 7.32 7.31 7.31 7.30 7.30 7.28 7.28 7.27 7.26 7.25 7.23 7.23 7.21 7.19 7.18 7.17 7.16 4.94 4.94 4.85 4.71 4.66 4.63 4.62 4.59 4.55 4.54 4.53 4.50 3.73 2.09 2.07 1.32 1.30 1.27 1.26 1.21 1.20 1.19 1.17 1.12

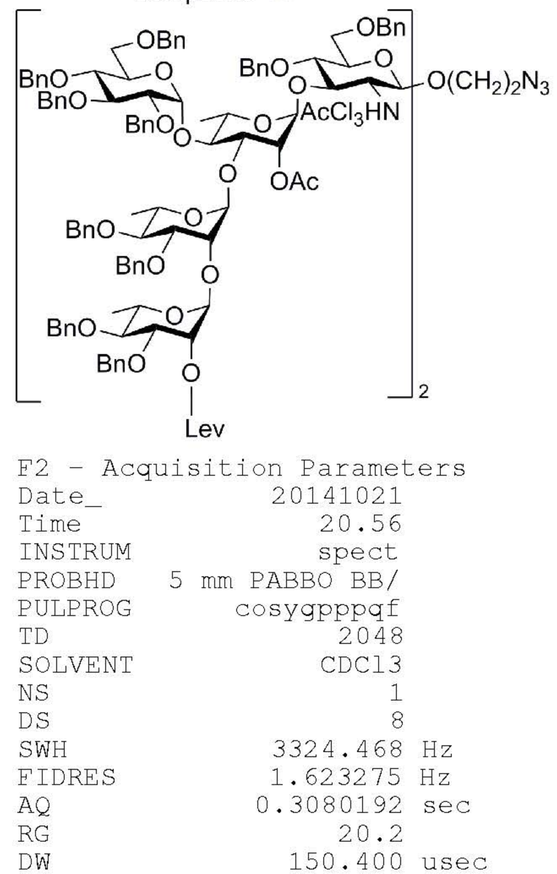


Compound **14**




```
F2 - Acquisition Parameters
Date_              20141021
Time                 20.54
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9583745 sec
RG                   35.9
DW                60.400 usec
DE                 6.00 usec
TE                303.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.10 usec
PL1               -6.00 dB
SFO1        400.1324708 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300093 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

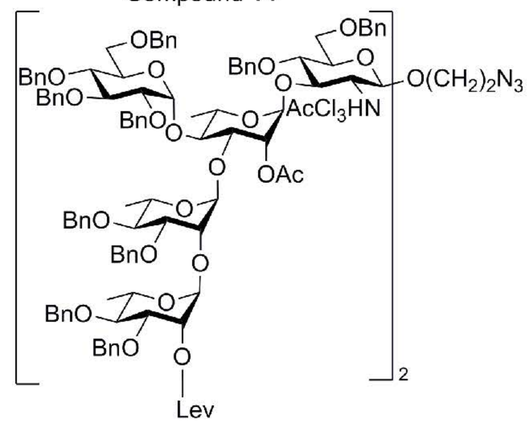
Compound **14**




```
F2 - Acquisition Parameters
Date_              20141021
Time                 20.56
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                   2048
SOLVENT              CDCl3
NS                      1
DS                      8
SWH             3324.468 Hz
FIDRES          1.623275 Hz
AQ             0.3080192 sec
RG                   20.2
DW              150.400 usec
DE                6.50 usec
TE                303.0 K
D0           0.00000300 sec
D1           1.37220395 sec
D11          0.03000000 sec
D12          0.00002000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00030080 sec

======== CHANNEL f1 ========
NUC1                   1H
P0                 7.10 usec
P1                 7.10 usec
P17             2500.00 usec
PL1               -6.00 dB
PL10               6.13 dB
SFO1       400.1317800 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]       SINE.100
GPZ1              10.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                   512
SFO1          400.1318 MHz
FIDRES        6.493102 Hz
SW               8.308 ppm
FnMODE                QF
```

129.1 128.6 128.5 128.4 128.4 128.4 128.3 128.3 128.3 128.3 128.2 128.0 127.9 127.9 127.9 127.8 127.8 127.7 127.7 127.6 127.6 127.5 127.5 127.4 127.3 127.2 127.1 125.3 99.4 96.3 81.7 81.6 80.9 80.2 80.0 79.8 79.3 77.6 77.6 77.5 77.3 77.0 76.5 76.3 75.1 75.0 74.9 74.8 74.5 74.2 71.9 71.8 71.6 69.4 68.5 68.3 68.1 67.9 67.8 56.8 29.7 28.1 21.1 21.0 18.7 18.2 18.2 18.1 18.0

Compound **14**

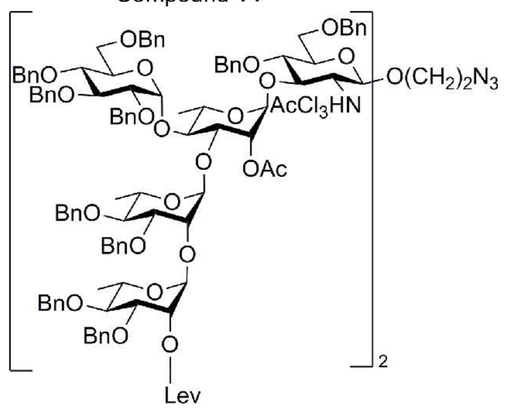


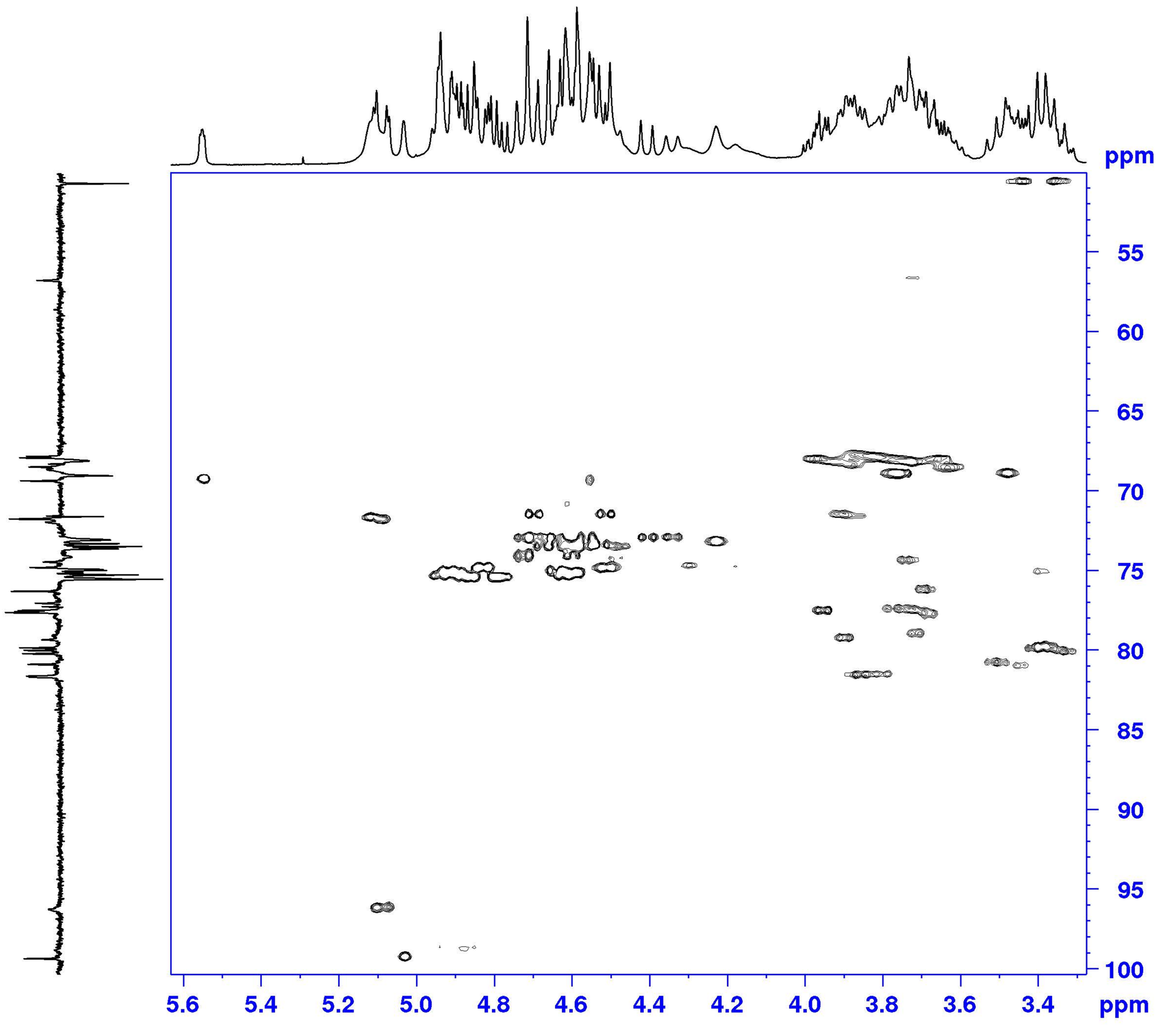
F2 - Acquisition Parameters

Date_	20141021
Time	23.11
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	6.25 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	12.00 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz
======== CHANNEL f2 ========	
CPDPRG[2	waltz16
NUC2	1H
P3	7.10 usec
P4	14.20 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
SFO2	400.1316005 MHz

130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **14**



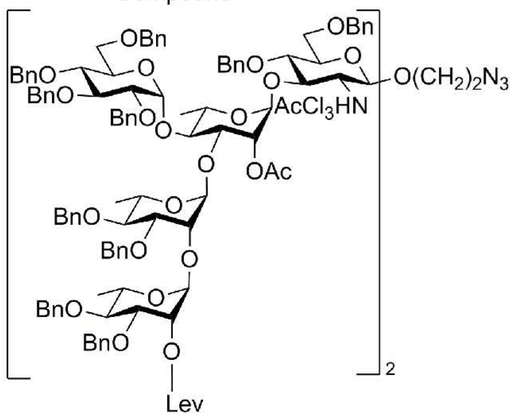


F2 - Acquisition Parameters

Date_	20141021
Time	23.13
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	3324.468 Hz
FIDRES	1.623275 Hz
AQ	0.3080192 sec
RG	6502
DW	150.400 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.38449299 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
P2	14.20 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1317800 MHz
======== CHANNEL f2 ========	
CPDPRG[2	garp
NUC2	13C
P3	6.25 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	22.50 dB
SFO2	100.6208180 MHz
SP3	12.00 dB
SP7	12.00 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

206.1
171.8
170.1
161.4
138.9
138.8
138.8
138.7
138.7
138.7
138.6
138.5
138.5
138.4
138.2
138.0
138.0
137.8
137.4
128.7
128.6
128.5
128.5
128.4
128.4
128.4
128.3
128.2
128.2
128.1
128.0
128.0
127.9
127.9
127.9
127.8
127.8
127.7
127.6
127.6
127.5
127.2
92.7
77.7
77.7
77.6
77.4
76.4
75.6
75.6
75.3
75.1
74.9
73.7
73.6
73.4
73.1
71.8
71.7
69.1
68.4
68.0
50.8
38.2
29.8
28.2
21.2
21.1
18.3
18.2
18.1

Compound **14**



F2 - Acquisition Parameters

Date_	20141022	
Time	4.01	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	4096	
DS	4	
SWH	26178.010	Hz
FIDRES	0.399445	Hz
AQ	1.2517376	sec
RG	13004	
DW	19.100	usec
DE	6.50	usec
TE	303.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	

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

NUC1	13C	
P1	6.25	usec
PL1	-6.00	dB
SFO1	100.6248425	MHz

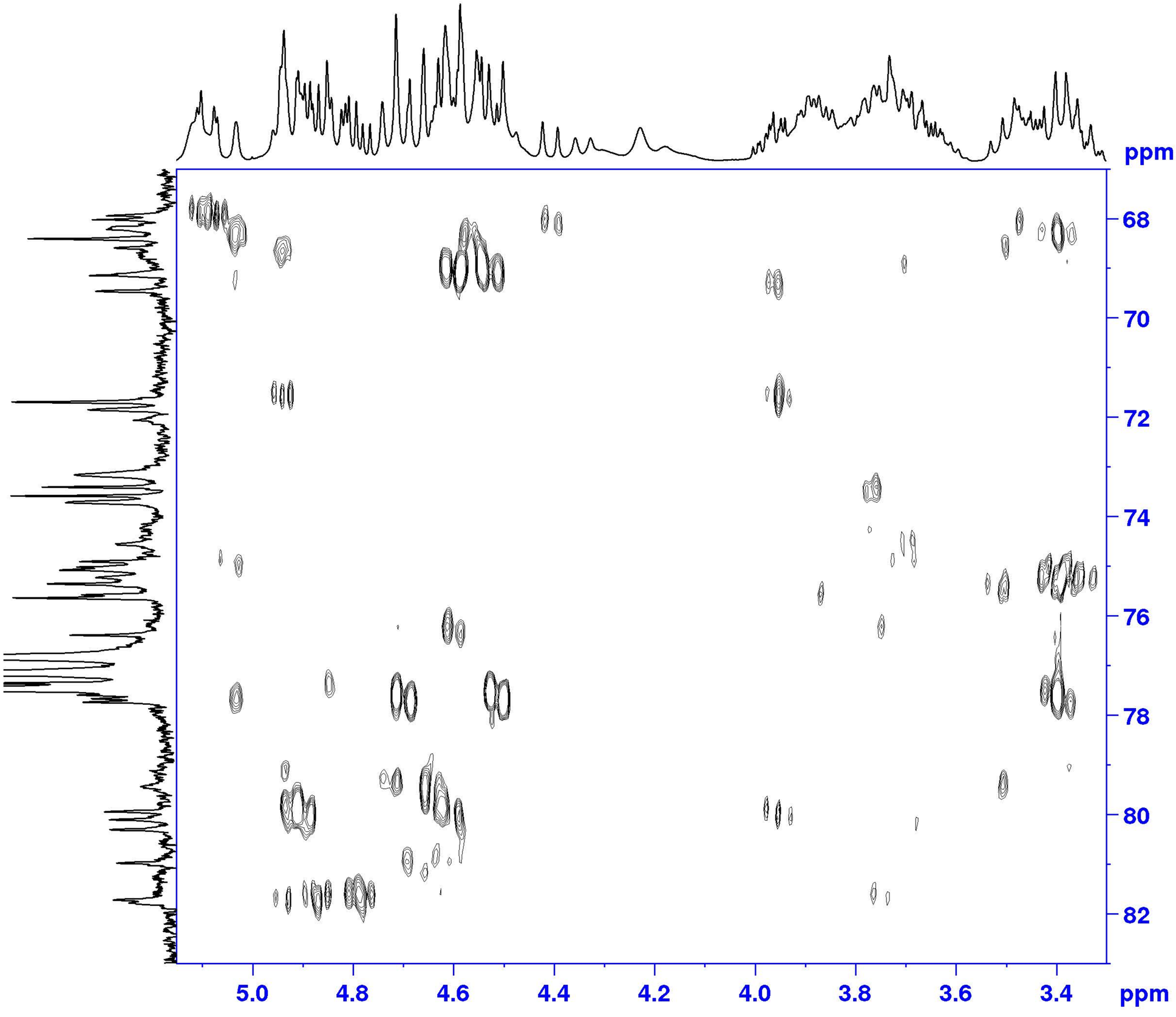
======== CHANNEL f2 ========

CPDPRG[2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-6.00	dB
PL12	15.04	dB
PL13	20.00	dB
SFO2	400.1316005	MHz

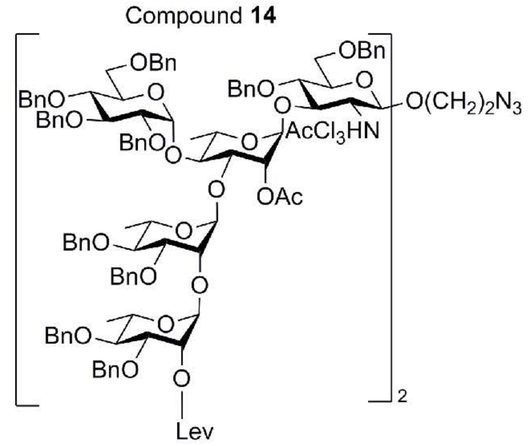
F2 - Processing parameters

SI	32768	
SF	100.6127600	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

200 180 160 140 120 100 80 60 40 20 ppm

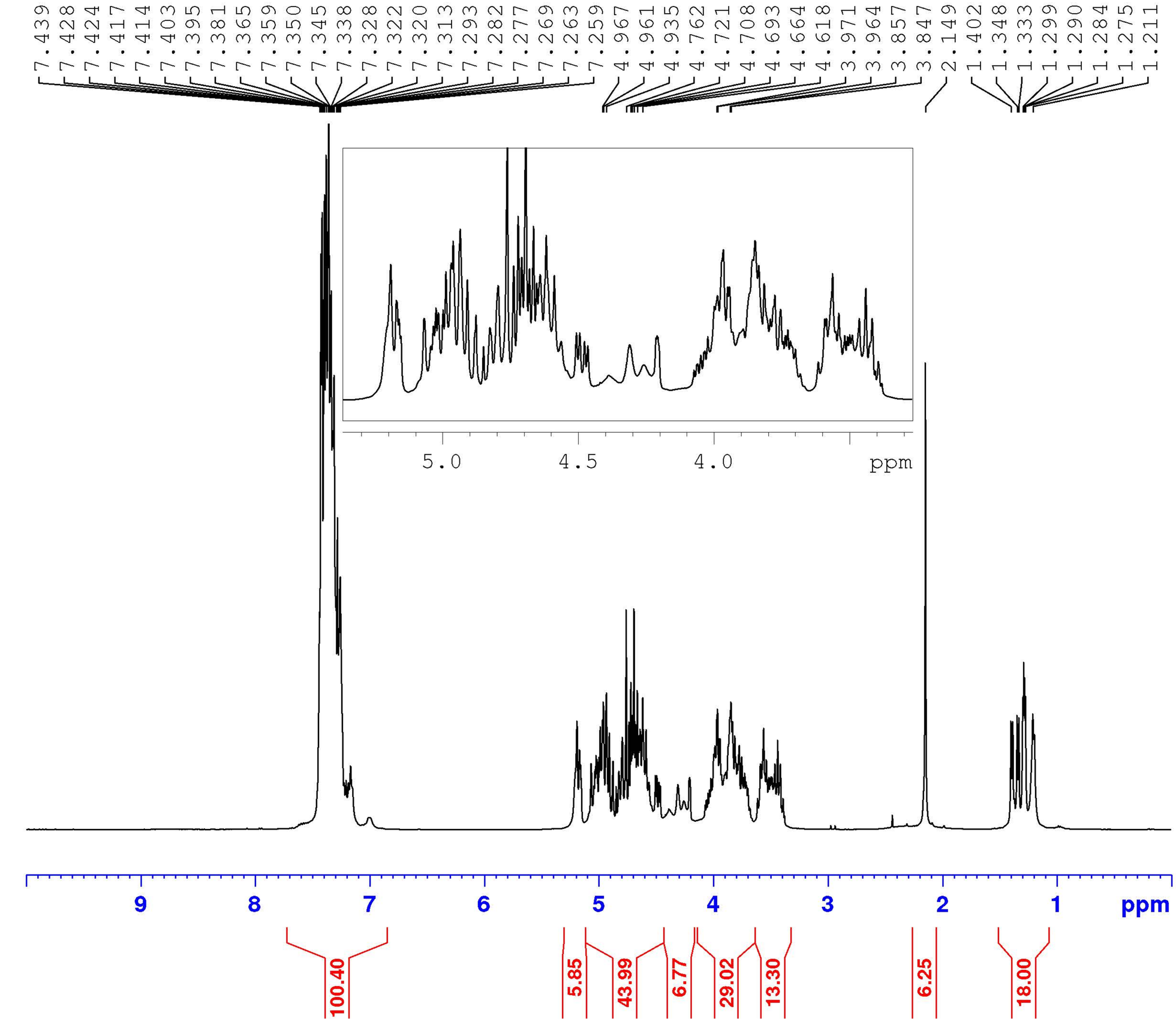


Compound **14**

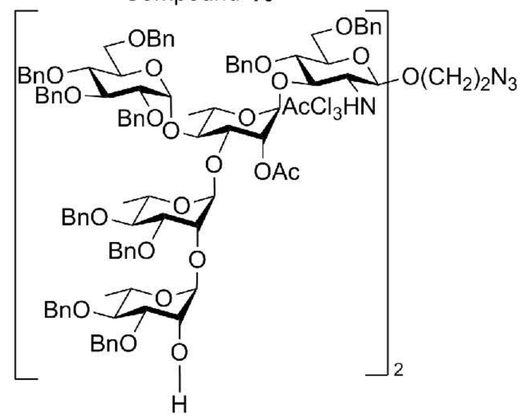


F2 - Acquisition Parameters

Date_	20141022
Time	4.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	3324.468 Hz
FIDRES	1.623275 Hz
AQ	0.3080192 sec
RG	16384
DW	150.400 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	1.01228797 sec
D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002240 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	-6.00 dB
SFO1	400.1317800 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	6.25 usec
PL2	-6.00 dB
SFO2	100.6228138 MHz
====== GRADIENT CHANNEL =====	
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec

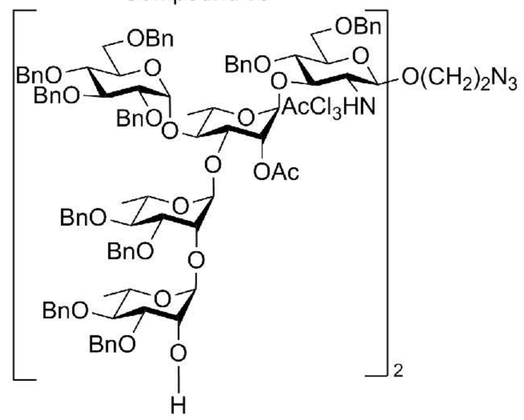


Compound **15**




```
F2 - Acquisition Parameters
Date_            20120907
Time                23.38
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 100000
SOLVENT             CDCl3
NS                     256
DS                       2
SWH             8278.146 Hz
FIDRES          0.082781 Hz
AQ            6.0400000 sec
RG                    20.2
DW                60.400 usec
DE                 6.00 usec
TE                303.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.75 usec
PL1                -6.00 dB
SFO1         400.1324708 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Compound **15**



F2 - Acquisition Parameters

Date_	20120907
Time	23.40
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	3396.739 Hz
FIDRES	1.658564 Hz
AQ	0.3014656 sec
RG	10.1
DW	147.200 usec
DE	6.50 usec
TE	303.0 K
D0	0.00000300 sec
D1	1.37834799 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00029440 sec

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

NUC1	1H
P0	6.75 usec
P1	6.75 usec
P17	2500.00 usec
PL1	-6.00 dB
PL10	6.57 dB
SFO1	400.1317802 MHz

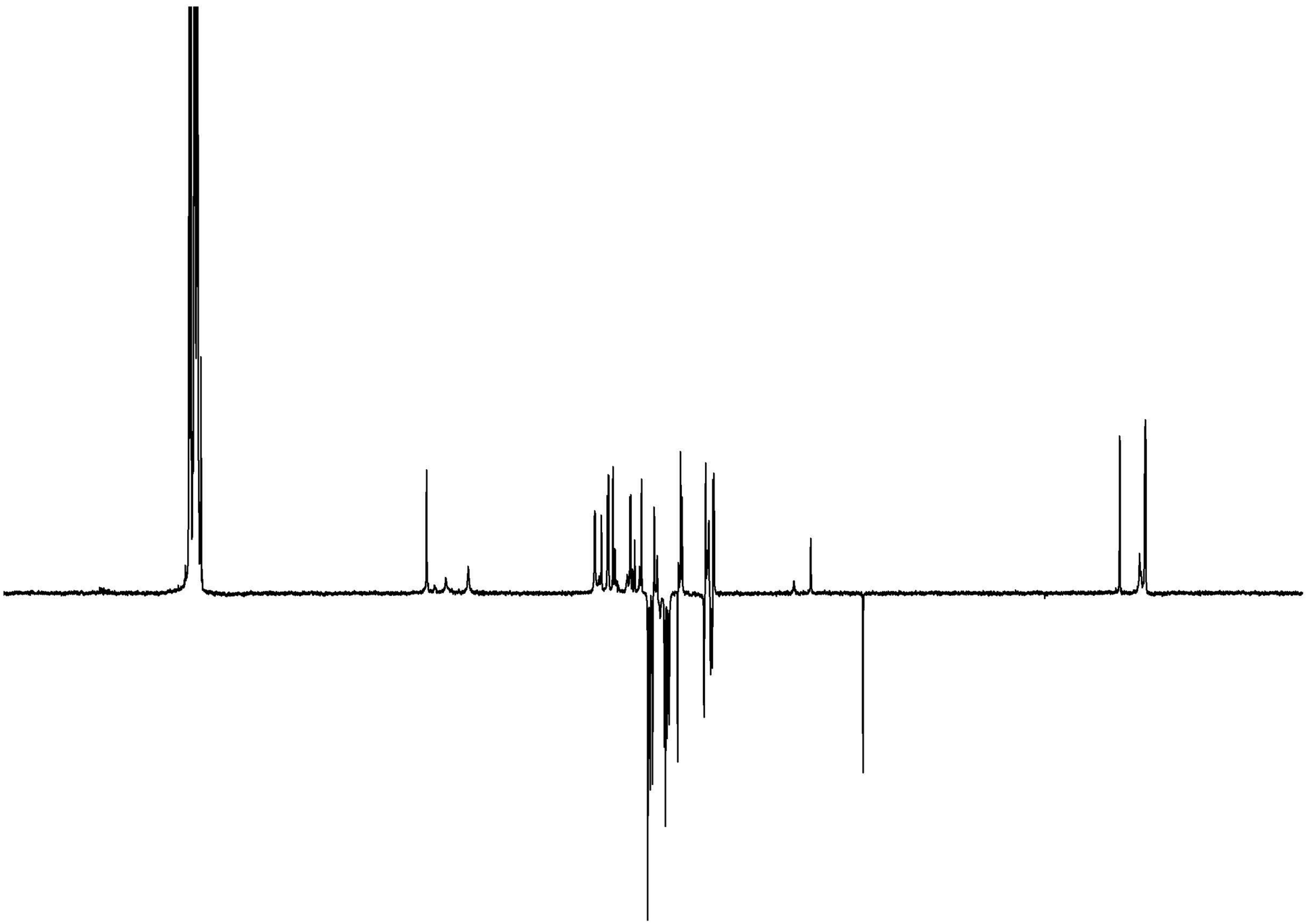
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPZ1	10.00 %
P16	1000.00 usec

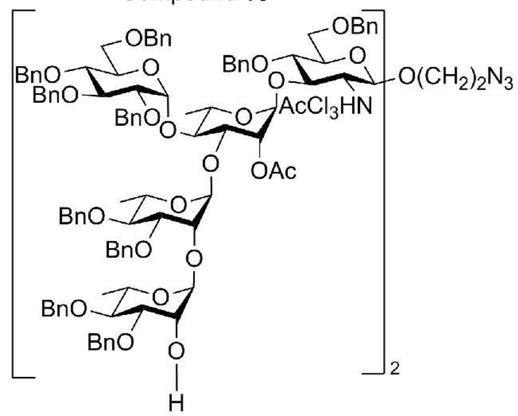
F1 - Acquisition parameters

TD	512
SFO1	400.1318 MHz
FIDRES	6.634256 Hz
SW	8.489 ppm
FnMODE	QF

128.85 128.63 128.58 128.56 128.49 128.45 128.41 128.39 128.34 128.14 128.03 127.99 127.94 127.87 127.84 127.79 127.74 127.69 127.65 127.61 127.56 127.51 127.44 127.27 127.19 127.06 101.13 98.93 96.33 81.74 81.68 81.17 80.95 80.28 80.18 80.13 79.63 79.39 79.11 79.04 77.97 77.66 77.57 77.36 77.11 76.53 76.32 74.85 74.51 72.08 71.81 71.63 68.92 68.73 68.55 68.07 67.98 58.72 56.77 21.12 21.08 18.81 18.65 18.24 18.19 18.15 18.09

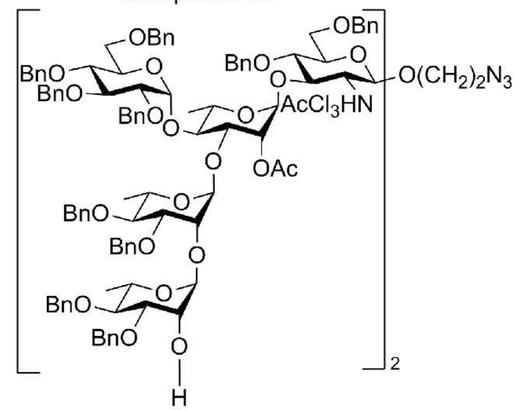


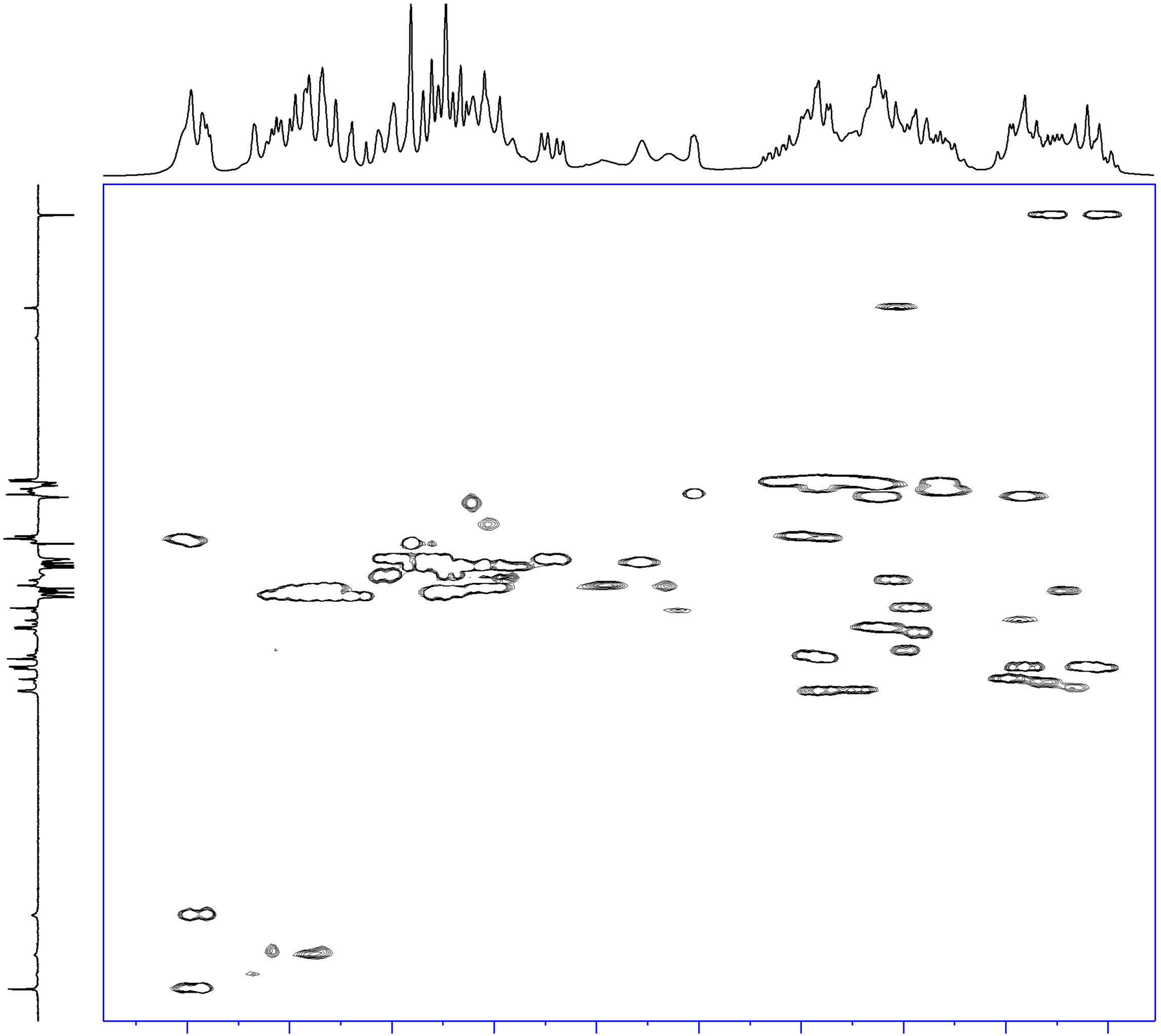
Compound **15**




```
F2 - Acquisition Parameters
Date_              20120908
Time                  7.27
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG          deptsp135
TD                   65536
SOLVENT              CDCl3
NS                    6000
DS                       4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ               1.3664256 sec
RG                   16384
DW                  20.850 usec
DE                   6.00 usec
TE                  303.0 K
CNST2           145.0000000
D1               2.00000000 sec
D2               0.00344828 sec
D12              0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    5.90 usec
P12                2000.00 usec
PL0                 120.00 dB
PL1                  -6.00 dB
SFO1           100.6228298 MHz
SP2                   6.74 dB
SPNAM[2]     Crp60comp.4
SPOAL2               0.500
SPOFFS2   0 Hz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
P3                    6.75 usec
P4                   13.50 usec
PCPD2                80.00 usec
PL2                  -6.00 dB
PL12                 20.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF             100.6127690 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

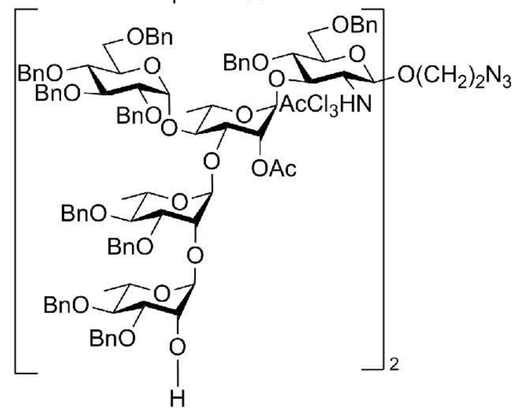
Compound **15**






```
F2 - Acquisition Parameters
Date_            20120908
Time                7.30
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                  2048
SOLVENT             CDCl3
NS                    16
DS                    16
SWH             3396.739 Hz
FIDRES           1.658564 Hz
AQ              0.3014656 sec
RG                  45.3
DW                147.200 usec
DE                  6.00 usec
TE                303.0 K
CNST2          145.0000000
D0              0.00000300 sec
D1              1.39063704 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
IN0             0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                  1H
P1                  6.75 usec
P2                 13.50 usec
P28              1000.00 usec
PL1                -6.00 dB
SFO1          400.1317802 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                  5.90 usec
P14               500.00 usec
P24              2000.00 usec
PCPD2              75.00 usec
PL0               120.00 dB
PL2                -6.00 dB
PL12               23.00 dB
SFO2          100.6208180 MHz
SP3                 6.74 dB
SP7                 6.74 dB
SPNAM[3] Crp60,0.5,20.1
SPNAM[7]    Crp60comp.4
SPOAL3              0.500
SPOAL7              0.500
SPOFFS3   0 Hz
SPOFFS7   0 Hz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPNAM[2]        SINE.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec
```

Compound **15**



F2 - Acquisition Parameters

Date_	20120909
Time	5.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	20000
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	9195.2
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	5.90 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

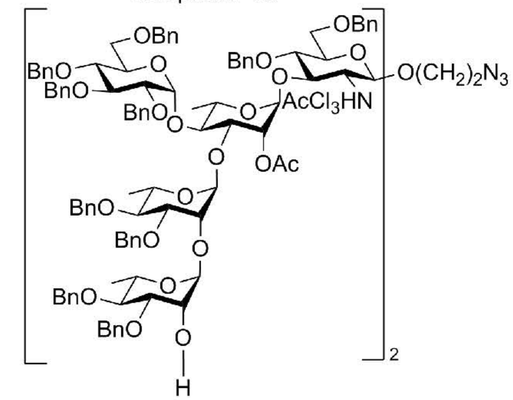
======== CHANNEL f2 ========

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	20.00 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

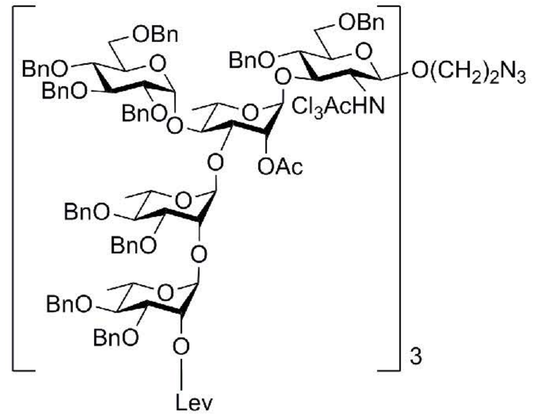
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **15**




```
F2 - Acquisition Parameters
Date_              20120909
Time                   5.55
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG     hmbcgplpndqf
TD                     2048
SOLVENT               CDCl3
NS                       32
DS                       16
SWH              3396.739 Hz
FIDRES           1.658564 Hz
AQ              0.3014656 sec
RG                     16384
DW               147.200 usec
DE                 6.00 usec
TE                 303.0 K
CNST2          145.0000000
CNST13          10.0000000
D0              0.00000300 sec
D1              1.01843202 sec
D2              0.00344828 sec
D6              0.05000000 sec
D16             0.00020000 sec
IN0             0.00002240 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   6.75 usec
P2                  13.50 usec
PL1                 -6.00 dB
SFO1          400.1317802 MHz

======== CHANNEL f2 ========
NUC2                   13C
P3                   5.90 usec
PL2                 -6.00 dB
SFO2          100.6228138 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPNAM[3]         SINE.100
GPZ1                50.00 %
GPZ2                30.00 %
GPZ3                40.10 %
P16               1000.00 usec
```

Compound **16**



F2 - Acquisition Parameters

Date_	20140802
Time	16.13
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	57
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

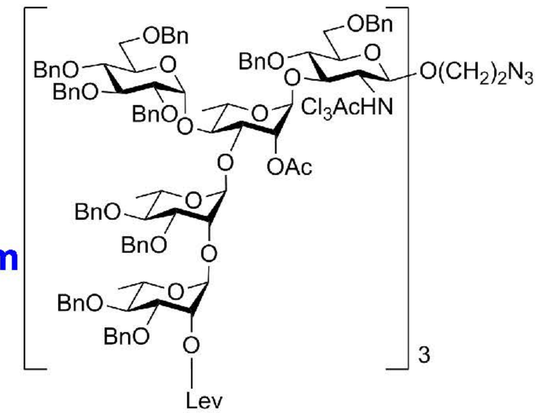
======== CHANNEL f1 ========

NUC1	1H
P1	7.10 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

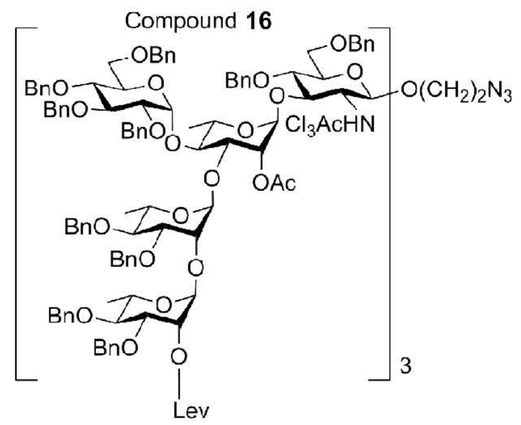
SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Compound **16**

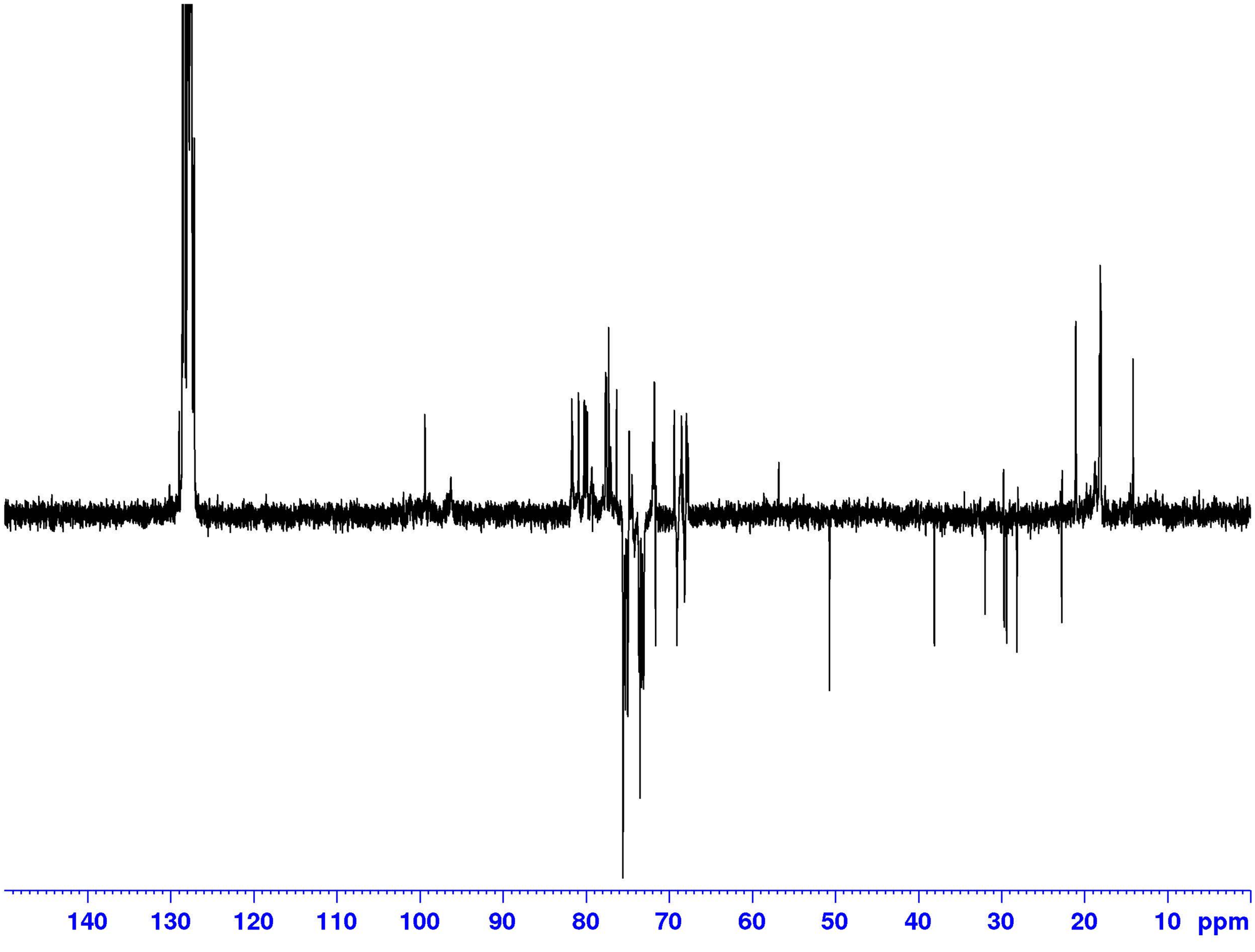



```
F2 - Acquisition Parameters
Date_              20140802
Time                 16.15
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                    2048
SOLVENT               CDCl3
NS                       4
DS                       8
SWH               3787.879 Hz
FIDRES            1.849550 Hz
AQ                0.2703360 sec
RG                    25.4
DW                 132.000 usec
DE                   6.50 usec
TE                  303.0 K
D0              0.00000300 sec
D1              1.40906799 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00026400 sec

======== CHANNEL f1 ========
NUC1                    1H
P0                   7.10 usec
P1                   7.10 usec
P17               2500.00 usec
PL1                 -6.00 dB
PL10                 6.13 dB
SFO1          400.1315756 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPZ1               10.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                     512
SFO1            400.1316 MHz
FIDRES          7.398201 Hz
SW                 9.467 ppm
FnMODE                QF
```

Compound **16**



128.96 128.60 128.55 128.50 128.41 128.38 128.32 128.28 128.26 128.14 127.97 127.93 127.87 127.84 127.79 127.70 127.68 127.58 127.55 127.45 127.38 127.26 127.13 99.35 96.22 81.67 80.87 80.19 79.99 79.83 79.28 77.62 77.49 77.23 77.01 76.29 75.45 75.11 74.78 74.44 74.13 73.60 71.94 71.74 71.59 69.35 69.03 68.48 68.29 68.10 67.91 67.82 67.68 56.77 38.06 31.94 29.70 29.66 29.36 28.11 22.70 22.67 21.01 19.72 18.72 18.20 18.15 18.06 18.01 14.11



F2 - Acquisition Parameters

Date_	20140802
Time	22.10
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	5120
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664256 sec
RG	16384
DW	20.850 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

NUC1	13C
P1	6.25 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-6.00 dB
SFO1	100.6228298 MHz
SP2	12.00 dB
SPNAM[2]	Crp60comp.4
SPOAL2	0.500
SPOFFS2	0 Hz

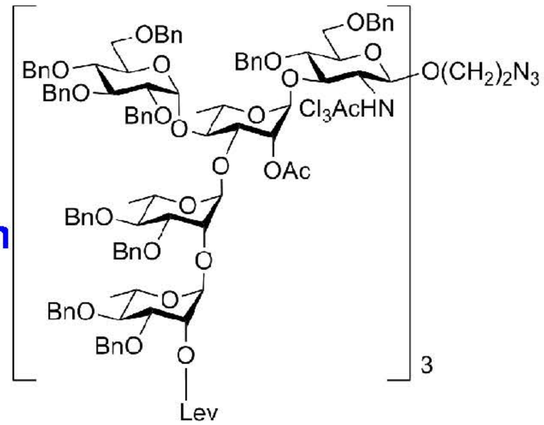
======== CHANNEL f2 ========

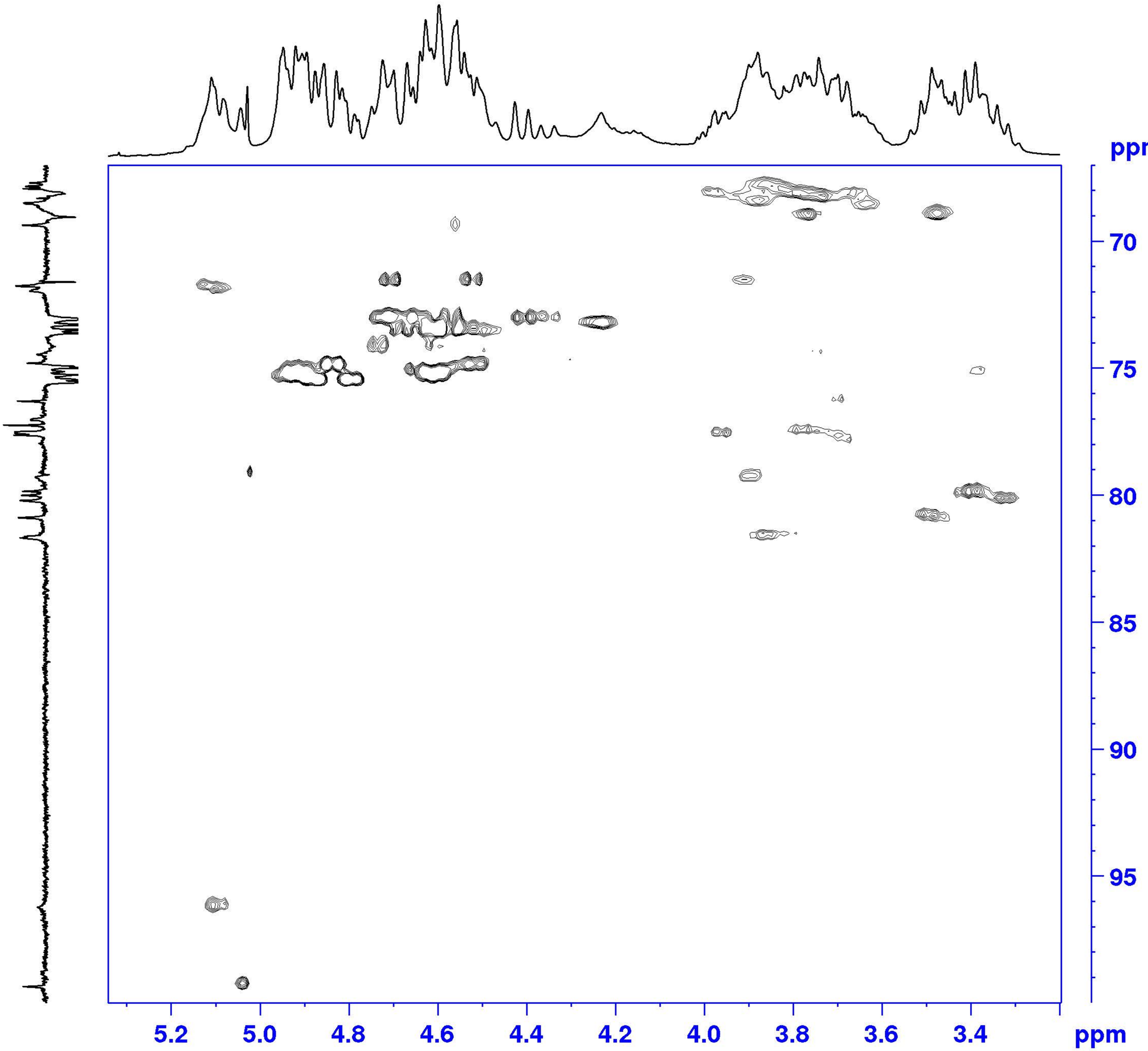
CPDPRG[2	waltz16
NUC2	1H
P3	7.10 usec
P4	14.20 usec
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **16**

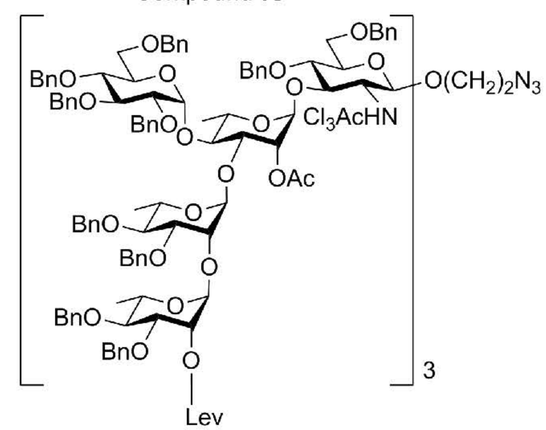


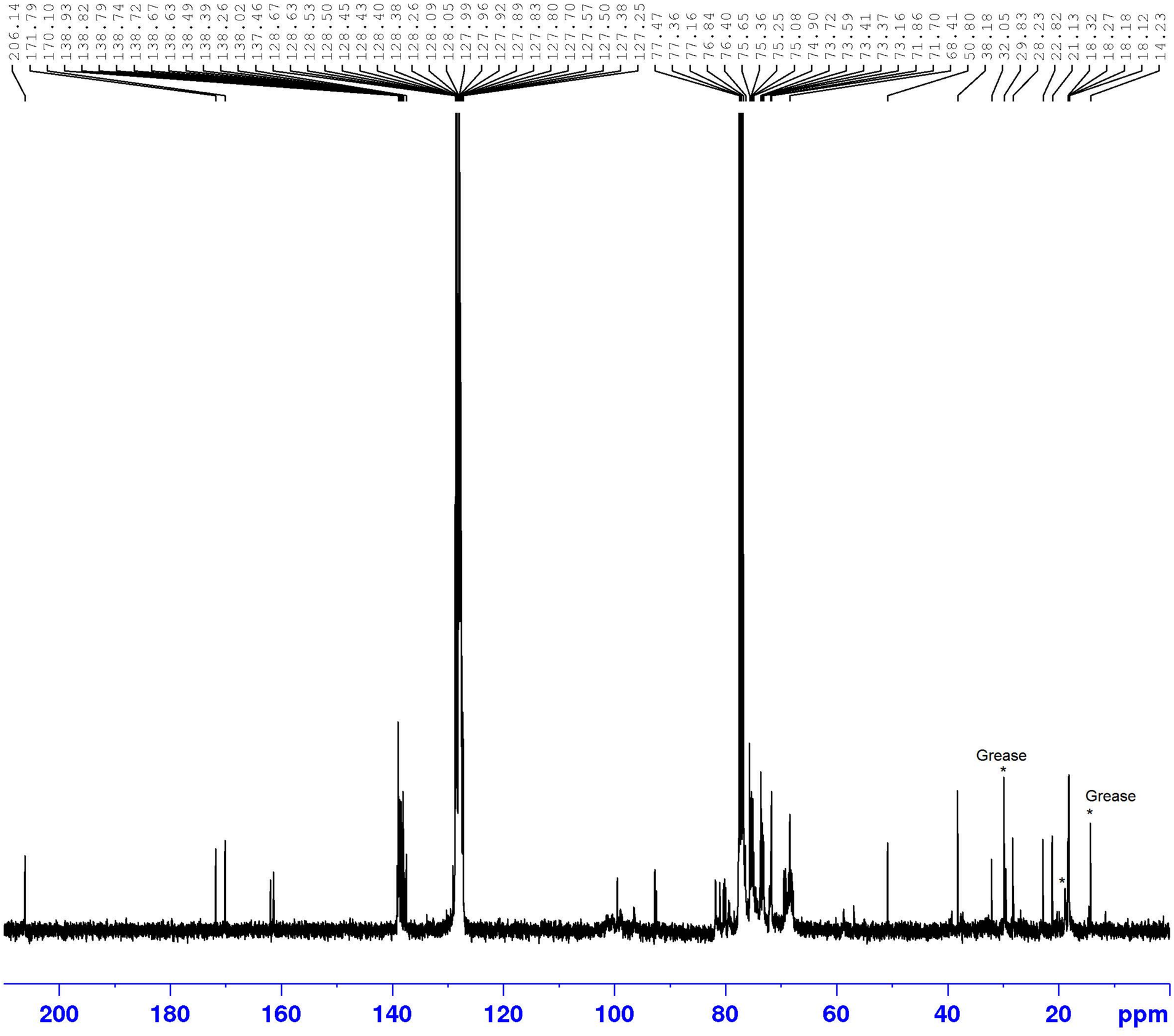



```
F2 - Acquisition Parameters
Date_            20140802
Time                22.13
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG     hsqcetgpsp.2
TD                  2048
SOLVENT              CDC13
NS                       8
DS                      16
SWH              3787.879 Hz
FIDRES           1.849550 Hz
AQ               0.2703360 sec
RG                    8192
DW                132.000 usec
DE                  6.00 usec
TE                 303.0 K
CNST2         145.0000000
D0             0.00000300 sec
D1             1.42135704 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
IN0            0.00002760 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.10 usec
P2                 14.20 usec
P28              1000.00 usec
PL1                -6.00 dB
SFO1          400.1315756 MHz

======== CHANNEL f2 ========
CPDPRG[2              garp
NUC2                  13C
P3                  6.25 usec
P14               500.00 usec
P24              2000.00 usec
PCPD2              75.00 usec
PL0               120.00 dB
PL2                -6.00 dB
PL12               22.50 dB
SFO2          100.6208180 MHz
SP3                12.00 dB
SP7                12.00 dB
SPNAM[3] Crp60,0.5,20.1
SPNAM[7]     Crp60comp.4
SPOAL3               0.500
SPOAL7               0.500
SPOFFS3  0 Hz
SPOFFS7  0 Hz

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    512
SFO1           100.6208 MHz
FIDRES         35.374508 Hz
SW               180.000 ppm
FnMODE     Echo-Antiecho
```

206.14
171.79
170.10
138.93
138.82
138.79
138.74
138.72
138.67
138.63
138.49
138.39
138.26
138.02
137.46
128.67
128.63
128.53
128.50
128.45
128.43
128.40
128.38
128.26
128.09
128.05
127.99
127.96
127.92
127.89
127.83
127.80
127.70
127.57
127.50
127.38
127.25
77.47
77.36
77.16
76.84
76.40
75.65
75.36
75.25
75.08
74.90
73.72
73.59
73.41
73.37
73.16
71.86
71.70
68.41
50.80
38.18
32.05
29.83
28.23
22.82
21.13
18.32
18.27
18.18
18.12
14.23

Compound **16**





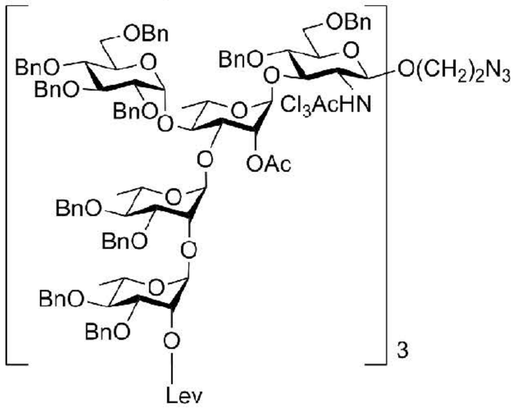
F2 - Acquisition Parameters

Date_	20140807
Time	6.48
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	12288
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	4597.6
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	6.25 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz
======== CHANNEL f2 ========	
CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound **16**



ppm

65
70
75
80
85
90
95
100

5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 ppm

F2 - Acquisition Parameters

Date_	20140803
Time	15.24
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	32
DS	16
SWH	3787.879 Hz
FIDRES	1.849550 Hz
AQ	0.2703360 sec
RG	16384
DW	132.000 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
CNST13	5.0000000
D0	0.00000300 sec
D1	2.04915190 sec
D2	0.00344828 sec
D6	0.10000000 sec
D16	0.00020000 sec
IN0	0.00002240 sec

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

NUC1	1H
P1	7.10 usec
P2	14.20 usec
PL1	-6.00 dB
SFO1	400.1315756 MHz

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

NUC2	13C
P3	6.25 usec
PL2	-6.00 dB
SFO2	100.6228138 MHz

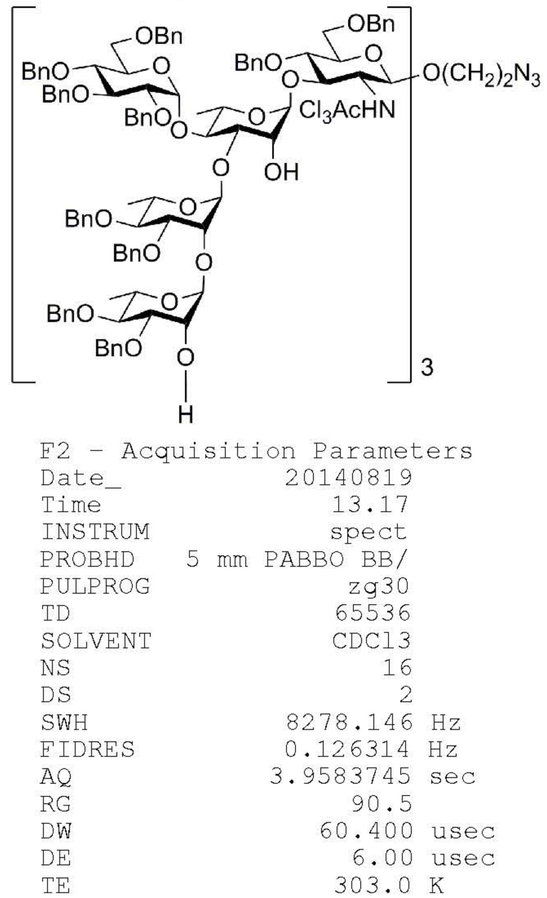
====== GRADIENT CHANNEL =====

GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPNAM[3]	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	512
SFO1	100.6228 MHz
FIDRES	43.596539 Hz
SW	221.833 ppm
FnMODE	QF

Compound **17**



F2 - Acquisition Parameters

Date_	20140819
Time	13.17
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9583745 sec
RG	90.5
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

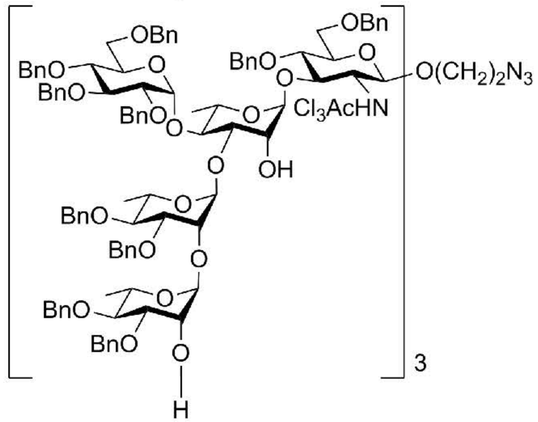
======== CHANNEL f1 ========

NUC1	1H
P1	7.10 usec
PL1	-6.00 dB
SFO1	400.1324708 MHz

F2 - Processing parameters

SI	32768
SF	400.1300437 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

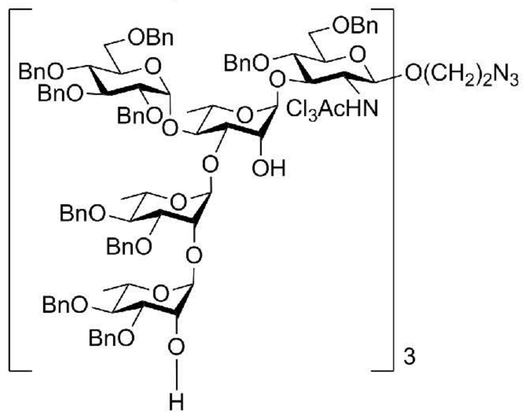
Compound **17**




```
F2 - Acquisition Parameters
Date_              20140819
Time                 13.19
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpppqf
TD                    2048
SOLVENT               CDCl3
NS                       1
DS                       8
SWH                3140.704 Hz
FIDRES             1.533547 Hz
AQ                0.3260416 sec
RG                       64
DW                 159.200 usec
DE                   6.50 usec
TE                   303.0 K
D0              0.00000300 sec
D1              1.35377204 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00031840 sec

======== CHANNEL f1 ========
NUC1                    1H
P0                   7.10 usec
P1                   7.10 usec
P17                2500.00 usec
PL1                  -6.00 dB
PL10                 6.13 dB
SFO1          400.1316688 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]        SINE.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     128
SFO1            400.1317 MHz
FIDRES          24.536747 Hz
SW                 7.849 ppm
FnMODE                 QF
```

129.04 128.96 128.75 128.61 128.48 128.45 128.42 128.34 128.27 128.23 128.11 128.03 128.00 127.96 127.92 127.90 127.86 127.80 127.77 127.73 127.70 127.61 127.53 127.45 127.40 127.33 127.27 127.25 127.19 100.89 99.98 99.75 99.65 99.50 99.25 98.46 81.66 81.44 81.10 80.15 79.97 79.57 79.08 78.02 77.82 77.69 77.57 77.22 76.73 76.12 75.95 75.07 74.54 71.72 71.63 71.48 70.14 68.82 68.29 68.00 67.71 57.81 56.05 18.48 18.40 18.30 18.24 18.18 18.11

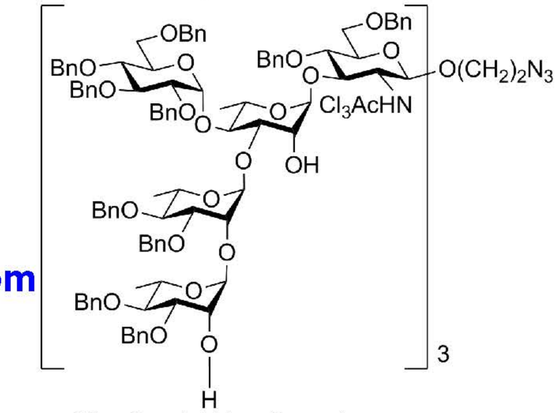
Compound **17**




```
F2 - Acquisition Parameters
Date_            20140819
Time                22.18
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG        deptsp135
TD                 65536
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664256 sec
RG                 16384
DW                20.850 usec
DE                 6.00 usec
TE                303.0 K
CNST2        145.0000000
D1             2.00000000 sec
D2             0.00344828 sec
D12            0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.25 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -6.00 dB
SFO1         100.6228298 MHz
SP2                12.00 dB
SPNAM[2]     Crp60comp.4
SPOAL2               0.500
SPOFFS2   0 Hz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
P3                  7.10 usec
P4                 14.20 usec
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               15.04 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound **17**



ppm

55 60 65 70 75 80 85 90 95 100

5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 ppm

F2 - Acquisition Parameters

Date_	20140819
Time	22.21
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	hsqcetgpsp.2
TD	2048
SOLVENT	CDCl3
NS	16
DS	16
SWH	3140.704 Hz
FIDRES	1.533547 Hz
AQ	0.3260416 sec
RG	18390.4
DW	159.200 usec
DE	6.00 usec
TE	303.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.36606097 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
IN0	0.00002760 sec
ZGOPTNS	

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

NUC1	1H
P1	7.10 usec
P2	14.20 usec
P28	1000.00 usec
PL1	-6.00 dB
SFO1	400.1316688 MHz

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

CPDPRG[2	garp
NUC2	13C
P3	6.25 usec
P14	500.00 usec
P24	2000.00 usec
PCPD2	75.00 usec
PL0	120.00 dB
PL2	-6.00 dB
PL12	22.50 dB
SFO2	100.6208180 MHz
SP3	12.00 dB
SP7	12.00 dB
SPNAM[3]	Crp60,0.5,20.1
SPNAM[7]	Crp60comp.4
SPOAL3	0.500
SPOAL7	0.500
SPOFFS3	0 Hz
SPOFFS7	0 Hz

====== GRADIENT CHANNEL =====

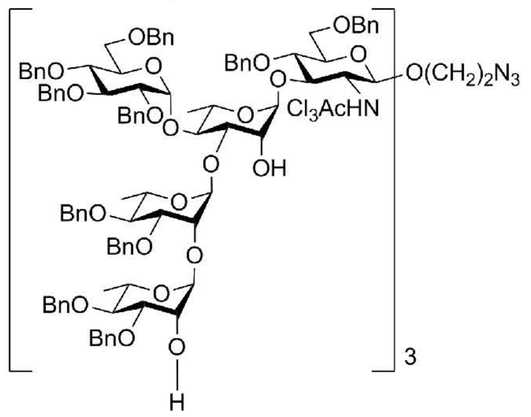
GPNAM[1]	SINE.100
GPNAM[2]	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec

F1 - Acquisition parameters

TD	512
SFO1	100.6208 MHz
FIDRES	35.374508 Hz
SW	180.000 ppm
FnMODE	Echo-Antiecho

161.99 138.99 138.88 138.75 138.65 138.51 138.40 138.25 138.01 137.96 137.85 137.76 137.51 129.19 129.10 128.90 128.75 128.62 128.60 128.56 128.49 128.47 128.42 128.38 128.26 128.18 128.15 128.11 128.05 128.01 127.95 127.92 127.85 127.76 127.67 127.60 127.47 127.40 127.34 92.57 92.47 81.24 80.30 80.13 79.72 76.26 75.69 75.47 75.13 74.03 73.78 73.68 73.62 73.45 73.24 73.16 72.16 70.07 69.14 68.44 68.14 50.93 29.84 18.63 18.55 18.44 18.39 18.33 18.26

Compound **17**



F2 - Acquisition Parameters

Date_	20140820
Time	7.01
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	5120
DS	4
SWH	26178.010 Hz
FIDRES	0.399445 Hz
AQ	1.2517376 sec
RG	13004
DW	19.100 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	6.25 usec
PL1	-6.00 dB
SFO1	100.6248425 MHz

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

CPDPRG[2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	15.04 dB
PL13	20.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127541 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

200 180 160 140 120 100 80 60 40 20 ppm