Supporting Information

Direct Synthesis of C-Glycosides from Unprotected 2-*N*-Acyl-Aldohexoses via Aldol Condensation-oxa-Michael Reactions with Unactivated Ketones

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General

For thin layer chromatography (TLC), Merck silica gel 60 F254 aluminum sheets were used. Flash column chromatography was performed using Yamazen silica gel flash column chromatography. ^{1}H NMR and ^{13}C NMR were recorded on a Bruker Avance 400. Proton chemical shifts are given in relative to the residual proton signals of the deuterated solvent in CD_3OD (δ 3.31 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in CD_3OD (δ 49.0 ppm). High-resolution mass spectra were recorded on a Thermo Scientific LTQ Orbitrap ESI ion trap mass spectrometer. Optical rotations were measured on a Jasco P2200 polarimeter.

1. Reaction of *N*-acetyl-D-mannosamine (1) with acetone

Reaction using L-proline and *N*,*N*-diisopropylethylamine as catalyst (Table 1, entry 6)
To a mixture of L-proline (24.0 mg, 0.209 mmol) in DMSO (1.0 mL), acetone (615 μL, 8.36 mmol) and *N*,*N*-diisopropylethylamine (36.0 μL, 0.209 mmol) were added at room temperature (25 °C), and the mixture was stirred for 5 min. To this mixture, *N*-acetyl-D-mannosamine monohydrate (100.0 mg, 0.418 mmol) was added and the resulting mixture was stirred at the same temperature for 96 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 92:8 to 84:16 over 10 min and 84:16 for 25 min) to give **2a** (72.8 mg, 62%).

Reaction using L-proline and *N*,*N*-diisopropylethylamine as catalyst (two times size reaction of Table 1, entry 6)
To a mixture of L-proline (48.0 mg, 0.418 mmol) in DMSO (1.0 mL), acetone (1.23 mL, 16.7 mmol) and *N*,*N*-diisopropylethylamine (73.0 μL, 0.418 mmol) were added at room temperature (25 °C), and the mixture was stirred for 5 min. To this mixture, *N*-acetyl-D-mannosamine monohydrate (200.0 mg, 0.836 mmol) was added and the resulting mixture was stirred at the same temperature for 96 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 92:8 to 84:16 over 10 min and 84:16 for 25 min) to give **2a** (131.0 mg, 60%). Compound **2a** was crystallized from acetone.

Reaction using L-proline, *N*,*N*-diisopropylethylamine, and *cis*-4-hydroxycyclohexanecarboxylic acid as catalyst (Table 1, entry 12)
A mixture of L-proline (24.0 mg, 0.209 mmol), DMSO (1.0 mL), acetone (615 μL, 8.36 mmol), *N*,*N*-diisopropylethylamine (81.0 μL, 0.627 mmol), cis-4-hydroxycyclohexanecarboxylic acid (60.0 mg, 0.418 mmol), and *N*-acetyl-D-mannosamine monohydrate (100.0 mg, 0.418 mmol) were stirred at room temperature (25 °C) for 48 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 90:10 to 83:17 over 10 min and 83:17 for 25 min) to give **2a** (51.0 mg, 44%).

Reaction using pyrrolidine and boric acid as catalyst (Table 1, entry 18)
A mixture of pyrrolidine (17.0 μL, 0.209 mmol), H_3BO_3 (26.0 mg, 0.416 mmol), DMSO (1.0 mL), and acetone (615 μL, 8.36 mmol) was stirred at room temperature (25 °C) for 5 min. To this mixture, *N*-acetyl-D-mannosamine monohydrate (100.0 mg, 0.418 mmol) was added and the resulting mixture was stirred at the same temperature for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 92:8 to 83:17 over 10 min and 83:17 for 25 min) to give **2b** (62.6 mg, 53%).

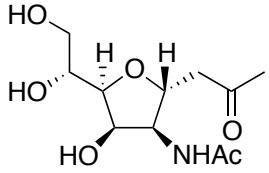
Transformation of 2a to 2b under the pyrrolidine-boric acid conditions
To a solution of **2a** (30.0 mg, 0.115 mmol) in DMSO (100 μL) was added H_3BO_3 (7.0 mg, 0.115 mmol) followed by pyrrolidine (5.0 μl, 0.57 mmol) at room temperature (25 °C), and the resulting mixture was stirred at the same temperature. Initially, the mixture was a clear colorless solution. After 1 h, the reaction mixture turned to pale yellow and the TLC analysis

showed that **2a** was consumed and **2b** was formed. The mixture was purified silica gel flash column chromatography (CH_2Cl_2/MeOH = 92:8 to 83:17 over 10 min and 83:17 for 25 min) to give **2b** (18.0 mg, 60%).

When compound **2a** was treated with pyrrolidine alone or H_3BO_3 alone in DMSO at room temperature (25 °C) under the same conditions, no changes of **2a** was detected after 1 h.

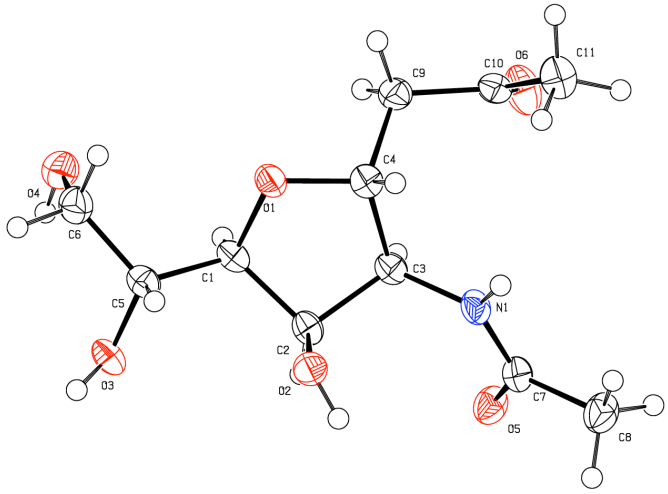
Compound 2a

N-((2*R*,3*S*,4*R*,5*S*)-5-((*R*)-1,2-dihydroxyethyl)-4-hydroxy-2-(2-oxopropyl)tetrahydrofuran-3-yl)acetamide



R_f = 0.32 (CH_2Cl_2/MeOH = 5:1).

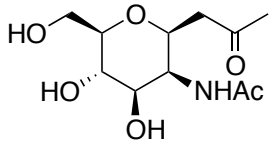
Colorless crystals (crystallized from acetone); mp 157 °C. $[\alpha]_D^{25}$ +59.3 (c 1.04, MeOH). ^{1}H NMR (400MHz, CD_3OD): δ 4.26 (dd, *J* = 4.0 Hz, 3.2 Hz, 1H), 4.23 (ddd, *J* = 9.6 Hz, 6.4 Hz, 5.2 Hz, 1H), 4.17 (dd, *J* = 9.6 Hz, 4.0 Hz, 1H), 3.94 (dd, *J* = 8.4 Hz, 3.2 Hz, 1H), 3.88 (ddd, *J* = 8.4 Hz, 6.0 Hz, 3.2 Hz, 1H), 3.73 (dd, *J* = 11.6 Hz, 3.2 Hz, 1H), 3.56 (dd, *J* = 11.6 Hz, 6.0 Hz, 1H), 2.704 (d, *J* = 6.4 Hz, 1H), 2.702 (d, *J* = 5.2 Hz, 1H), 2.17 (s, 3H), 1.99 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.8, 173.6, 82.1, 77.3, 72.2, 71.4, 64.9, 59.0, 48.4, 30.6, 22.6. HRMS (ESI): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1273.



X-ray crystal structure of **2a** (CCDC 1422504)

Compound 2b[S1, S2]

N-((2*S*,3*S*,4*R*,5*S*,6*R*)-4,5-dihydroxy-6-(hydroxymethyl)-2-(2-oxopropyl)tetrahydro-2*H*-pyran-3-yl)acetamide



R_f = 0.27 (CH_2Cl_2/MeOH = 5:1).
Colorless gum. $[\alpha]_D^{25}$ –29.0 (c 1.00, MeOH). Lit. $[\alpha]_D$ –28.5 (c 0.45, H_2O).[S2] ^{1}H NMR (400MHz, CD_3OD): δ 4.30 (dd, *J* = 4.4 Hz, 1.2 Hz, 1H), 4.03 (ddd, *J* = 7.2 Hz, 5.2 Hz, 1.2 Hz, 1H), 3.78 (d, *J* = 3.6 Hz, 2H), 3.69 (dd, *J* = 9.6 Hz, 4.4 Hz, 1H), 3.50 (t, *J* = 9.6 Hz, 1H), 3.22 (dt, *J* = 9.6 Hz, 3.6 Hz, 1H), 2.65 (dd, *J* = 17.2 Hz, 7.2 Hz, 1H), 2.54 (dd, *J* = 17.2 Hz, 5.2 Hz, 1H), 2.14 (s, 3H), 2.05 (s, 3H). ^{13}C NMR (400 MHz, CD_3OD): δ 208.6, 174.5, 82.3, 75.1, 74.8, 68.3, 62.2, 54.1, 45.9, 30.4, 22.6. HRMS (ESI): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1275.

2. Reaction of *N*-acetyl-D-glucosamine (5) with acetone

Reaction using L-proline and *N*,*N*-diisopropylethylamine as catalyst (Table 2, entry 4)
A mixture of D-proline (78.0 mg, 0.68 mmol), DMSO (3.0 mL), acetone (1.99 mL, 27.1 mmol), and *N*,*N*-diisopropylethylamine (118 μL, 0.68 mmol) was stirred at room temperature (25 °C) for 5 min. To this mixture, *N*-acetyl-D-glucosamine (300 mg, 1.36 mmol) was added and the resulting mixture was stirred at the same temperature for 96 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 90:10 to 83:17 over 10 min and 83:17 for 25 min) to give a mixture of **6a** and **6b** (7.7 mg, 2%).

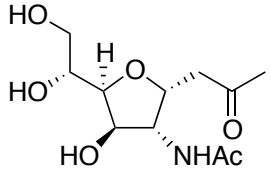
Because the reactions of *N*-acetyl-D-mannosamine (**1**) to give **2** (Table 1) were performed using *N*-acetyl-D-mannosamine monohydrate, the reaction of *N*-acetyl-D-glucosamine (**5**) described in Table 2, entry 4 was also performed with addition of H_2O (1 equiv to **5**; H_2O was premixed with **5** prior to the addition to the reaction mixture). No improvement in the product formation in this reaction was observed.

Reaction using pyrrolidine and boric acid (1 equiv) as catalyst (Table 2, entry 6)
A mixture of pyrrolidine (111 μL, 1.36 mmol), H_3BO_3 (168.0 mg, 2.71 mmol), DMSO (6.0 mL), acetone (3.99 mL, 54.3 mmol), and *N*-acetyl-D-glucosamine (600 mg, 2.71 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 86:14 to 78:22 over 10 min and 78:22 for 25 min) to give **6a** (156.0 mg, 22%, **6a-1**/**6a-2** = 1:3).

Reaction using pyrrolidine and boric acid (2 equiv) as catalyst (Table 2, entry 7)
A mixture of pyrrolidine (19.0 μL, 0.23 mmol), H_3BO_3 (56.0 mg, 0.90 mmol), DMSO (1.0 mL), acetone (0.66 mL, 9.0 mmol), and *N*-acetyl-D-glucosamine (100 mg, 0.45 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 86:14 to 78:22 over 10 min and 78:22 for 25 min) to give **6b** (79.0 mg, 66%).

Compound 6a-1

N-((2*R*,3*R*,4*R*,5*S*)-5-((*R*)-1,2-dihydroxyethyl)-4-hydroxy-2-(2-oxopropyl)tetrahydrofuran-3-yl)acetamide

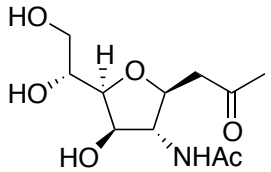


R_f = 0.30 (CH_2Cl_2/MeOH = 5:1).

Colorless oil (**6a-1**:**6a-2** = 3:1). ^{1}H NMR (400MHz, CD_3OD) (**6a-1** extracted from **6a-1**:**6a-2** = 3:1): δ 4.15 (dd, *J* = 4.0 Hz, 2.0 Hz, 1H), 4.04 (dt, *J* = 8.0 Hz, 2.0 Hz, 1H), 3.92-3.87 (m, 2H), 3.76 (dd, *J* = 8.4 Hz, 4.0 Hz, 1H), 3.74 (dd, *J* = 3.2 Hz, 1.2 Hz, 1H), 3.58 (dd, *J* = 11.2 Hz, 5.6 Hz, 1H), 2.94 (dd, *J* = 16.8 Hz, 8.0 Hz, 1H), 2.84 (dd, *J* = 11.2 Hz, 5.6 Hz, 1H), 2.94 (dd, *J* = 16.8 Hz, 8.0 Hz, 1H), 2.84 (dd, *J* = 16.8 Hz, 4.8 Hz, 1H), 2.16 (s, 3H), 1.96 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.6, 173.2, 82.2, 80.5, 77.7, 71.2, 65.1, 64.1, 49.1, 30.5, 22.5. HRMS (ESI) (**6a-1**:**6a-2** = 3:1): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1275.

Compound 6a-2

N-((2*S*,3*R*,4*R*,5*S*)-5-((*R*)-1,2-dihydroxyethyl)-4-hydroxy-2-(2-oxopropyl)tetrahydrofuran-3-yl)acetamide

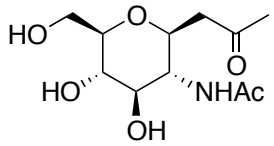


R_f = 0.24 (CH_2Cl_2/MeOH = 5:1).

Colorless oil (**6a-1**:**6a-2** = 1:1). ^{1}H NMR (400MHz, CD_3OD) (**6a-2** extracted from **6a-1**:**6a-2** = 1:1): δ 4.63 (ddd, *J* = 8.4 Hz, 5.6 Hz, 4.4 Hz, 1H), 4.32 (dd, *J* = 4.4 Hz, 1.2 Hz, 1H), 4.18 (dd, *J* = 3.6 Hz, 1.2 Hz, 1H), 3.94-3.89 (m, 2H), 3.75 (dd, *J* = 11.2 Hz, 3.0 Hz, 1H), 3.58 (dd, *J* = 11.2 Hz, 5.6 Hz, 1H), 2.73 (dd, *J* = 16.4 Hz, 8.4 Hz, 1H), 2.60 (dd, *J* = 16.4 Hz, 5.6 Hz, 1H), 2.16 (s, 3H), 1.97 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD) (**6a-2** extracted from **6a-1**:**6a-2** = 1:1): δ 209.1, 173.2, 81.0, 77.3, 76.2, 71.1, 65.4, 60.6, 44.4, 30.4, 22.4. HRMS (ESI) (**6a-1**:**6a-2** = 1:1): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1291.

Compound 6b[S1,S3]

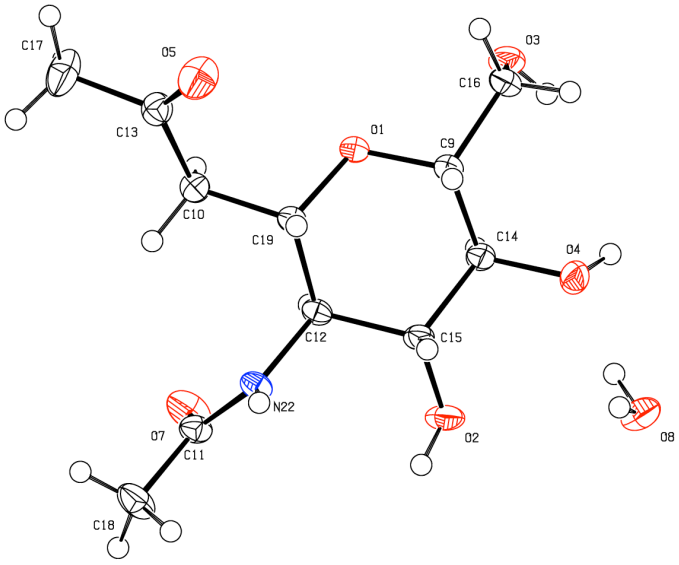
N-((2*S*,3*R*,4*R*,5*S*,6*R*)-4,5-dihydroxy-6-(hydroxymethyl)-2-(2-oxopropyl)tetrahydro-2*H*-pyran-3-yl)acetamide



R_f = 0.22 (CH_2Cl_2/MeOH = 5:1).

Colorless crystals (crystallized from CH_2Cl_2-MeOH); mp 141 °C. ^{1}H NMR (400MHz, CD_3OD): δ 3.80 (dd, *J* = 12.0 Hz, 2.4 Hz, 1H), 3.75 (ddd, *J* = 9.6 Hz, 8.0 Hz, 4.0 Hz, 1H),

3.63 (dd, J = 12.0 Hz, 5.6 Hz, 1H), 3.62 (t, J = 9.6 Hz, 1H), 3.40 (dd, J = 9.6 Hz, 8.4 Hz, 1H), 3.32 (dd, J = 9.6 Hz, 8.4 Hz, 1H), 3.22 (ddd, J = 9.6 Hz, 5.6 Hz, 2.4 Hz, 1H), 2.67 (dd, J = 16.4 Hz, 8.0 Hz, 1H), 2.60, (dd, J = 16.4 Hz, 8.0 Hz, 1H), 2.16, (s, 3H), 1.96 (s, 3H). ^{13}C NMR (100 MHz, D_2O): δ 209.6, 173.8, 81.7, 77.1, 76.3, 72.2, 62.8, 56.7, 47.3, 30.6, 22.8. HRMS (ESI): calcd for $C_{11}H_{19}NO_6Na$ ([M + Na]$^+$) 284.1110, found 284.1105.



X-ray crystal structure of **6b** (CCDC 1422505)

3. Reaction of *N*-valeryl-D-glucosamine (7) with acetone

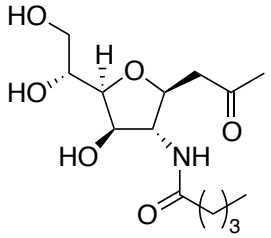
Reaction using D-proline and *N*,*N*-diisopropylethylamine as catalyst (Scheme 4)

To a mixture of D-proline (22.0 mg, 0.19 mmol) in DMSO (1.0 mL), acetone (558 µL, 7.6 mmol) and *N*,*N*-diisopropylethylamine (33.0 µL, 0.19 mmol) were added at room temperature (25 °C), and the mixture was stirred for 5 min. To this mixture, *N*-valeryl-D-glucosamine (100.0 mg, 0.38 mmol) was added and the resulting mixture was stirred at the same temperature for 96 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 90:10 to 83:17 over 10 min and 83:17 for 25 min) to give **8a** (13.8 mg, 12%).

Reaction using pyrrolidine and boric acid as catalyst (Scheme 4)

A mixture of pyrrolidine (16.0 µL, 0.19 mmol), H_3BO_3 (23.0 mg, 0.38 mmol), DMSO (1.0 mL), and acetone (559 µL, 7.6 mmol) was stirred at room temperature (25 °C) for 5 min. To this mixture, *N*-valeryl-D-glucosamine (100.0 mg, 0.38 mmol) was added and the resulting mixture was stirred at the same temperature for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 90:10 to 83:17 over 10 min and 83:17 for 25 min) to give **8b** (85.1 mg, 74%).

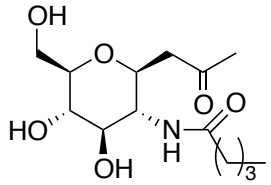
Compound 8a



R_f = 0.49 (CH_2Cl_2/MeOH = 5:1).

Colorless gum. ^{1}H NMR (400MHz, CD_3OD): δ 4.64 (ddd, *J* = 8.0 Hz, 5.4 Hz, 4.4. Hz, 1H), 4.34 (dd, 4.4.Hz, 1.2 Hz, 1H), 4.16 (dd, *J* = 3.6 Hz, 1.2 Hz, 1H), 3.92 (dd, *J* = 8.4 Hz, 3.6 Hz, 1H), 3.88 (ddd, *J* = 8.4 Hz, 5.6 Hz, 3.2 Hz, 1H), 3.76 (dd, *J* = 11.2 Hz, 3.2 Hz, 1H), 3.58 (dd, *J* = 11.2 Hz, 5.6 Hz, 1H), 2.73 (dd, 1H, *J* = 16.4 Hz, 8.0 Hz, 1H), 2.59 (dd, *J* = 16.4 Hz, 5.4 Hz, 1H), 2.28-2.20 (m, 2H), 2.16 (s, 3H), 1.64-1.55 (m, 2H), 1.40-1.30 (m, 2H), 0.96-0.92 (m, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.0, 176.3, 81.0, 77.4, 76.2, 71.2, 65.5, 60.5, 44.5, 36.5, 30.4, 29.3, 23.4, 14.1. HRMS (ESI): calcd for $C_{14}H_{26}NO_6$ ($[M + H]^+$) 304.1755, found 304.1760.

Compound 8b



R_f = 0.42 (CH_2Cl_2/MeOH = 5:1).

Pale yellow solid. ^{1}H NMR (400MHz, CD_3OD): δ 3.80 (dd, *J* = 12.0 Hz, 2.4 Hz, 1H), 3.75 (ddd, *J* = 10.0 Hz, 8.4 Hz, 3.2 Hz, 1H), 3.63 (dd, *J* = 10.0 Hz, 9.6 Hz, 1H), 3.63 (dd, *J* = 12.0 Hz, 5.2 Hz, 1H), 3.40 (dd, *J* = 10.0 Hz, 8.4 Hz, 1H), 3.31 (dd, *J* = 10.0 Hz, 8.4 Hz, 1H), 3.22 (ddd, *J* = 9.6 Hz, 5.2 Hz, 2.4 Hz, 1H), 2.67 (dd, *J* = 16.4 Hz, 8.4 Hz, 1H), 2.57 (dd, *J* = 16.4 Hz, 3.2 Hz, 1H), 2.21 (dt, *J* = 2.4 Hz, 7.6 Hz, 2H), 2.16 (s, 3H), 1.63-1.55 (m, 2H), 1.40-1.31 (m, 2H), 0.93 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.5, 176.8, 81.7, 77.1, 76.3, 72.3, 62.9, 56.5, 47.3, 37.1, 30.7, 29.0, 23.4, 14.1. HRMS (ESI): calcd for $C_{14}H_{26}NO_6$ ($[M + H]^+$) 304.1755, found 304.1756.

4. Reaction of *N*-acetyl-D-galactosamine (9) with acetone

Reaction using L-proline and *N*,*N*-diisopropylethylamine as catalyst in MeOH (Scheme 6)

A mixture of D-proline (26.0 mg, 0.23 mmol), MeOH (1.0 mL), acetone (660 μL, 9.0 mmol), *N*,*N*-diisopropylethylamine (39.0 μL, 0.23 mmol), and *N*-acetyl-D-galactosamine (100 mg, 0.45 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 91:9 to 84:16 over 10 min and 84:16 for 25 min) to give **10a** (103 mg, 88%, **10a-1**:**10a-2** = 1:1).

Reaction using D-proline and *N*,*N*-diisopropylethylamine as catalyst in DMSO

A mixture of D-proline (16.0 mg, 0.14 mmol), DMSO (1.0 mL), acetone (398 μL, 5.4 mmol), diisopropylethylamine (24.0 μL, 0.14 mmol), and *N*-acetyl-D-galactosamine (60.0 mg, 0.27 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 91:9 to 84:16 over 10 min and 84:16 for 25 min) to give **10a** (19.0 mg, 26%, **10a-1**:**10a-2** = 1.4:1).

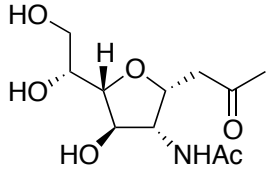
Reaction using D-proline and *N*,*N*-diisopropylethylamine as catalyst in MeOH at 60 °C (Scheme 6)

A mixture of D-proline (26.0 mg, 0.23 mmol), MeOH (1.0 mL), acetone (0.66 mL, 9.0 mmol), *N*,*N*-diisopropylethylamine (39.0 μL, 0.23 mmol), and *N*-acetyl-D-galactosamine (100 mg, 0.45 mmol) was stirred at 60 °C for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 91:9 to 84:16 over 10 min and 84:16 for 25 min) to give **10b** (38.0 mg, 32%).

Reaction using pyrrolidine and boric acid as catalyst at 25 °C

A mixture of pyrrolidine (19.0 μL, 0.23 mmol), H_3BO_3 (28.0 mg, 0.45 mmol), DMSO (1.0 mL), acetone (0.66 mL, 9.0 mmol), and *N*-acetyl-D-galactosamine (100 mg, 0.45 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 91:9 to 84:16 over 10 min and 84:16 for 25 min) to give **10a-1** (17.0 mg, 15%) and **10a-2** (17.0 mg, 15%), **10b** (7.0 mg, 6%).

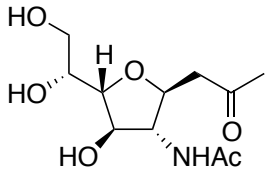
Compound 10a-1



R_f = 0.29 (CH_2Cl_2/MeOH = 5:1).

Colorless gum. $[\alpha]_D^{25}$ +7.1 (c 0.17, MeOH). ^{1}H NMR (400MHz, CD_3OD): δ 4.46 (ddd, *J* = 8.0 Hz, 5.2 Hz, 4.4 Hz, 1H), 4.21 (dd, *J* = 4.4 Hz, 2.0 Hz, 1H), 4.08 (dd, *J* = 3.6 Hz, 2.0 Hz, 1H), 3.75 (dd, *J* = 3.6 Hz, 2.4 Hz, 1H), 3.73 (ddd, *J* = 6.8 Hz, 5.6 Hz, 2.4 Hz, 1H), 3.60 (dd, *J* = 10.8 Hz, 5.6 Hz, 1H), 3.57 (dd, *J* = 10.8 Hz, 6.8 Hz, 1H), 2.71 (dd, *J* = 16.8 Hz, 8.0 Hz, 1H), 2.61 (dd, *J* = 16.8 Hz, 5.2 Hz, 1H), 2.16 (s, 3H), 1.97 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.0, 172.6, 86.4, 79.2, 77.1, 72.6, 64.4, 60.2, 44.2, 30.3, 22.7. HRMS (ESI): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1275.

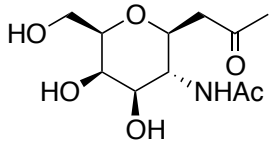
Compound 10a-2



R_f = 0.25 (CH_2Cl_2/MeOH = 5:1).

Colorless gum. $[\alpha]_D^{25}$ –10.5 (c 0.38, MeOH). ^{1}H NMR (400MHz, CD_3OD): δ 4.19 (t, *J* = 7.2 Hz, 1H), 4.14 (ddd, *J* = 7.2 Hz, 6.8 Hz, 4.4 Hz, 1H), 4.09 (dd, *J* = 7.2 Hz, 6.8 Hz, 1H), 3.83 (dd, *J* = 7.2 Hz, 2.8 Hz, 1H), 3.65 (ddd, *J* = 6.8 Hz, 5.6 Hz, 2.8 Hz, 1H), 3.58 (d, *J* = 5.6 Hz, 1H), 3.58 (d, *J* = 6.8 Hz, 1H), 2.84 (dd, *J* = 16.8 Hz, 7.2 Hz, 1H), 2.79 (d, *J* = 16.8 Hz, 4.4 Hz, 1H), 2.17 (s, 3H), 1.96 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.9, 173.8, 83.8, 78.8, 76.6, 72.6, 64.4, 62.4, 48.6, 30.6, 22.7. HRMS (ESI): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1288.

Compound 10b[S1]



R_f = 0.21 (CH_2Cl_2/MeOH = 5:1).

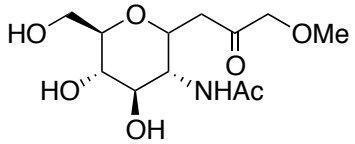
Colorless solid. $[\alpha]_D^{25}$ +19.4 (c 0.73, MeOH). ^{1}H NMR (400MHz, CD_3OD): δ 3.91 (t, *J* = 10.4 Hz, 1H), 3.87 (dd, *J* = 3.2 Hz, 0.8 Hz, 1H), 3.70 (ddd, *J* = 10.4 Hz, 8.8 Hz, 3.2 Hz, 1H), 3.663 (d, *J* = 6.8 Hz, 1H), 3.661 (d, *J* = 5.6 Hz, 1H), 3.53 (dd, *J* = 10.4 Hz, 3.2 Hz, 1H), 3.45 (ddd, *J* = 6.8 Hz, 5.6 Hz, 0.8 Hz, 1H), 2.72 (dd, *J* = 16.6 Hz, 8.8 Hz, 1H), 2.62 (dd, *J* = 16.6 Hz, 3.2 Hz, 1H), 2.16 (s, 3H), 1.96 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 209.7, 174.1, 80.2, 76.7, 74.0, 70.0, 62.6, 53.2, 47.3, 30.7, 22.9. HRMS (ESI): calcd for $C_{11}H_{20}NO_6$ ($[M + H]^+$) 262.1285, found 262.1289.

5. Reaction of *N*-acetyl-D-glucosamine (5) with methoxyacetone

Reaction of *N*-acetyl-D-glucosamine (5) with methoxyacetone using pyrrolidine and boric acid as catalyst (Scheme 7)

A mixture of H_3BO_3 (28.0 mg, 0.45 mmol) and *N*-acetyl-D-glucosamine (100 mg, 0.45 mmol) in DMSO (1.0 mL) was stirred at room temperature (25 °C) for 15 min. To the mixture, methoxyacetone (0.83 mL, 9.0 mmol) and pyrrolidine (19.0 μL, 0.23 mmol) were added and the resulting mixture was stirred at the same temperature for 36 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 86:14 to 79:21 over 10 min and 79:21 for 25 min) to give **11** (86.0 mg, 66%, α-isomer:β-isomer =1:1).

Compound 11



R_f = 0.32 (a 1:1 mixture of α- and β-isomers, CH_2Cl_2/MeOH = 5:1).

Colorless gum. ^{1}H NMR (400MHz, CD_3OD) (α-isomer:β-isomer =1:1): δ 4.68-4.62 (m, 1H x 1/2), 4.34-4,31 (m, 1H x 1/2), 4.19-4.03 (m, 2H + 1H +1H x 1/2), 3.95-3.71 (m, 3H + 1H x 1/2), 3.61-3.55 (m, 1H), 3.38 (s, 3H), 2.92 (dd, *J* = 16.2 Hz, 8.4 Hz, 1H x 1/2), 2.77 (dd, *J* = 16.2 Hz, 4.4 Hz, 1H x 1/2), 2.72 (dd, *J* = 16.2 Hz, 8.4 Hz, 1H x 1/2), 2.77 (dd, *J* = 16.2 Hz, 5.4 Hz, 1H x 1/2), 1.98 (s, 3H x 1/2), 1.96 (s, 3H x 1/2). ^{13}C NMR (100 MHz, CD_3OD) (α-

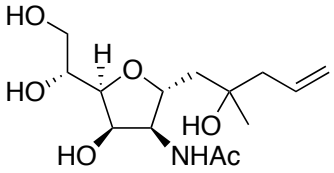
isomer:β-isomer =1:1): δ 208.5, 208.2, 173.2, 82.2, 81.0, 80.3, 78.7, 78.5, 77.6, 77.2, 76.1, 71.2, 71.1, 65.4, 65.1, 64.1, 60.6, 59.5, 44.7, 40.0, 22.5, 22.4. HRMS (ESI): calcd for $C_{12}H_{22}NO_7$ ([M + H]$^+$) 292.1391, found 262.1394.

6. Allylation reaction of 2a

Allylation reaction of 2a with allyl bromide and indium (Scheme 8)

To a solution of **2a** (50.0 mg, 0.191 mmol) in DMF (4.0 mL) and water (0.5 mL) were added allyl bromide (165 μL, 1.91 mmol) and In (22.0 mg, 0.191 mmol) at room temperature (25°C). The resulting mixture was stirred at the same temperature for 18 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 95:5 to 88:12 over 10 min and 88:12 for 25 min) to give **12** (100.3 mg, 88%).

Compound 12

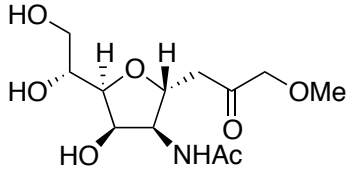


Colorless gum. ^{1}H NMR (400MHz, CD_3OD): δ 5.95-5.81(m, 1H), 5.09-5.02 (m, 2H), 4.27-4.21 (m, 1H), 4.12-4.05 (m, 2H), 3.95-3.85 (m, 2H), 3.78-3.73 (m, 1H), 3.59 (dd, *J* = 11.4 Hz, 5.4 Hz, 1H), 2.35-2.19 (m, 2H), 2.00 (s, 3H), 1.74-1.61 (m, 2H), 1.19 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 173.5, 135.9, 135.7, 118.1, 118.0, 82.1, 82.0, 77.8, 77.6, 72.9, 72.8, 71.6, 71.44, 71.40, 65.10, 65.07, 60.0, 47.6, 45.9, 45.7, 27.5, 27.0, 22.6. HRMS (ESI): calcd for $C_{14}H_{26}NO_6$ ([M + H]$^+$) 304.1755, found 304.1760.

7. Reaction of *N*-acetyl-D-mannosamine (1) with methoxyacetone

A mixture of L-proline (24.0 mg, 0.21 mmol), DMSO (1.0 mL), methoxyacetone (78 μL, 8.4 mmol), *N*,*N*-diisopropylethylamine (36.0 μL, 0.21 mmol), and *N*-acetyl-D-mannosamine monohydrate (100.0 mg, 0.42 mmol) was stirred at room temperature (25 °C) for 24 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 90:10 to 83:17 over 10 min and 83:17 for 25 min) to give compound **13** (11.0 mg, 8.8%).

Compound 13



R_f = 0.38 (CH_2Cl_2/MeOH = 5:1).

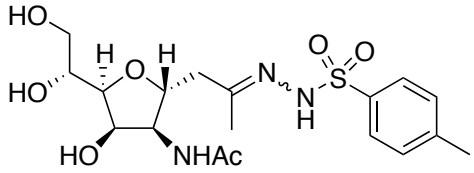
Colorless gum. ^{1}H NMR (400MHz, CD_3OD): δ 4.32-4.18 (m, 3H), 4.14 (s, 2H), 3.94 (dd, *J* = 8.0 Hz, 3.2 Hz, 1H), 3.87 (ddd, *J* = 8.0 Hz, 5.6 Hz, 3.2 Hz, 1H), 3.72 (dd, *J* = 11.2 Hz, 3.2 Hz, 1H), 3.56 (dd, *J* = 11.2 Hz, 5.6 Hz, 1H), 3.38 (s, 3H), 2.70 (dd, *J* = 16.0 Hz, 7.2 Hz, 1H), 2.63

(dd, J = 16.0 Hz, 3.2 Hz, 1H), 1.99 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 208.6, 173.6, 82.2, 78.8, 77.3, 72.2, 71.4, 64.9, 59.4, 59.1, 43.9, 22.6. HRMS (ESI): calcd for $C_{12}H_{22}NO_7$ ($[M + H]^+$) 292.1391, found 262.1397.

8. Reaction of 2a with sulfonyl hydrazide

To a solution of **2a** (50.0 mg, 0.19 mmol) in DMSO (1.0 mL) was added *p*-toluenesulfonyl hydrazide (46.0 mg, 0.25 mmol) at room temperature (25 °C) and the mixture was stirred at 40°C for 16 h. The mixture was purified by silica gel flash column chromatography (CH_2Cl_2/MeOH = 93:7 to 85:15 over 10 min and 85:15 for 25 min) to give **14a** (15.4 mg, 19%) and **14b** (41.5 mg, 50%).

Compound 14 (14a and 14b)



Compound 14a

R_f = 0.44 (CH_2Cl_2/MeOH 8:1).

Colorless gum. ^{1}H NMR (400MHz, CD_3OD): δ 7.80 (d, J = 8.2 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 4.24 (t, J = 3.2 Hz, 1H), 4.09 (dd, J = 9.6 Hz, 3.2 Hz, 1H), 4.05 (ddd, J = 9.6 Hz, 7.6 Hz, 2.8 Hz, 1H), 3.92 (dd, J = 8.4 Hz, 3.2 Hz, 1H), 3.83 (ddd, J = 8.4 Hz, 6.2 Hz, 3.0 Hz, 1H), 3.70 (dd, J = 11.4 Hz, 3.0 Hz, 1H), 3.53 (dd, J = 11.4 Hz, 6.2 Hz, 1H), 2.55 (dd, J = 14.8 Hz, 7.6 Hz, 1H), 2.49 (dd, J = 14.8 Hz, 2.8 Hz, 1H), 2.43 (s, 3H), 2.01 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 173.7, 158.9, 145.2, 137.3, 130.5, 129.1, 82.3, 78.9, 71.8, 71.4, 65.0, 59.0, 36.1, 24.0, 22.6, 21.5. HRMS (ESI): calcd for $C_{18}H_{28}N_3O_7S$ ($[M + H]^+$) 430.1642, found 430.1596.

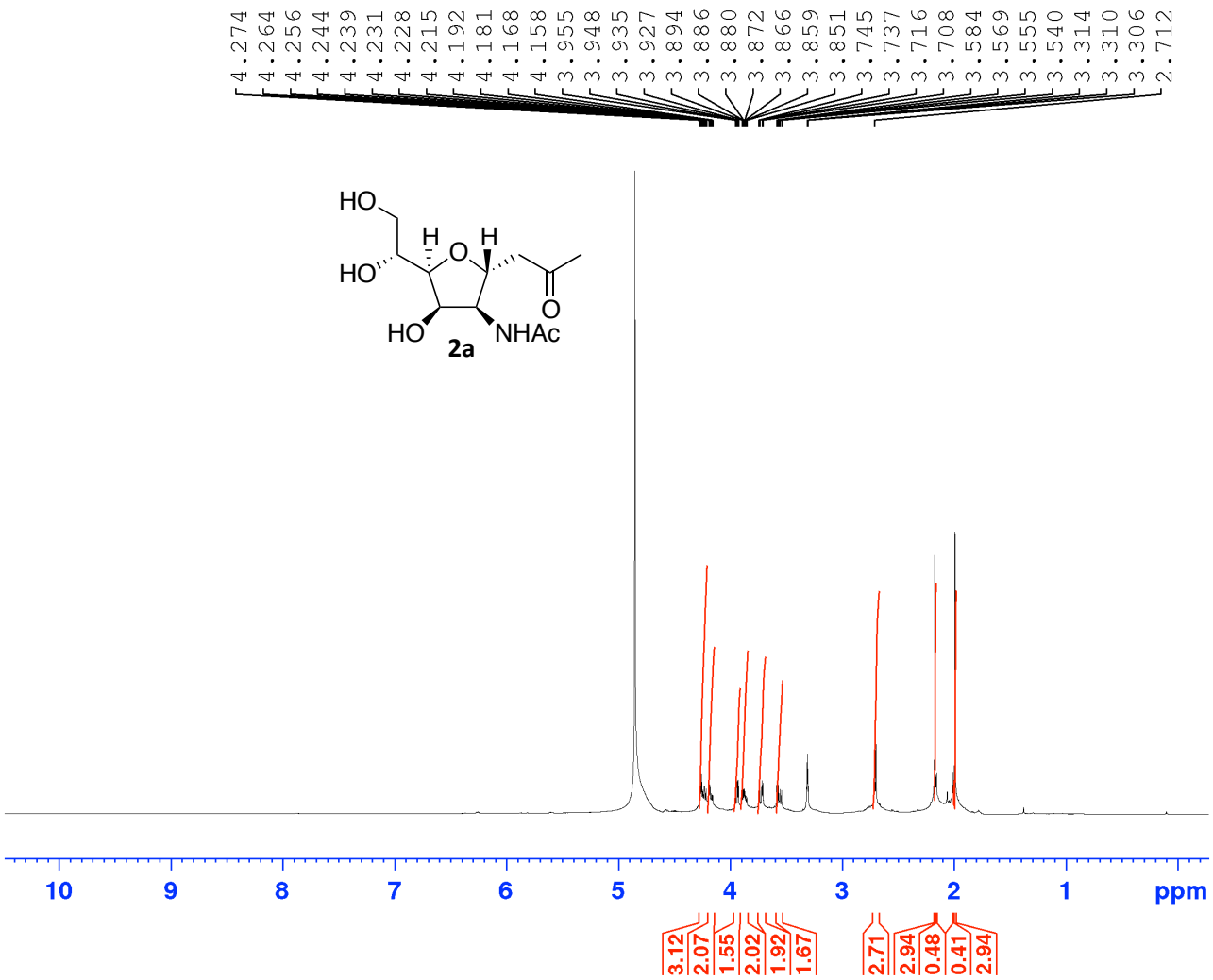
Compound 14b

R_f = 0.31 (CH_2Cl_2/MeOH 8:1).

Colorless gum. ^{1}H NMR (400MHz, CD_3OD): δ 7.80 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 4.25 (m, 1H), 4.10-4.02 (m, 2H), 3.84-3.77 (m, 2H), 3.68-3.62 (m, 1H), 3.50-3.45 (m, 1H), 2.49-2.34 (m, 2H), 2.42 (s, 3H), 2.42 (s,), 1.99 (s, 3H), 1.85 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD): δ 173.7, 159.0, 145.2, 137.4, 130.5, 129.0, 81.8, 78.7, 72.0, 71.4, 65.0, 59.5, 43.3, 22.7, 21.5, 17.3. HRMS (ESI): calcd for $C_{18}H_{28}N_3O_7S$ ($[M + H]^+$) 430.1642, found 430.1599.

9. References

(S1) N. Bragnier, M-C. Scherrmann, Synthesis **2005**, 814.
(S2) H.-W. Hagedorn, H. Merten, R. Brossmer, *Carbohydr. Res.* **1992**, *236*, 89.
(S3) F. Peri, L. Cipolla, B. La Ferla, P. Dumy, F. Nicotra, *Glycoconjugate J.* **1999**, *16*, 399.

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PROCNO                 1

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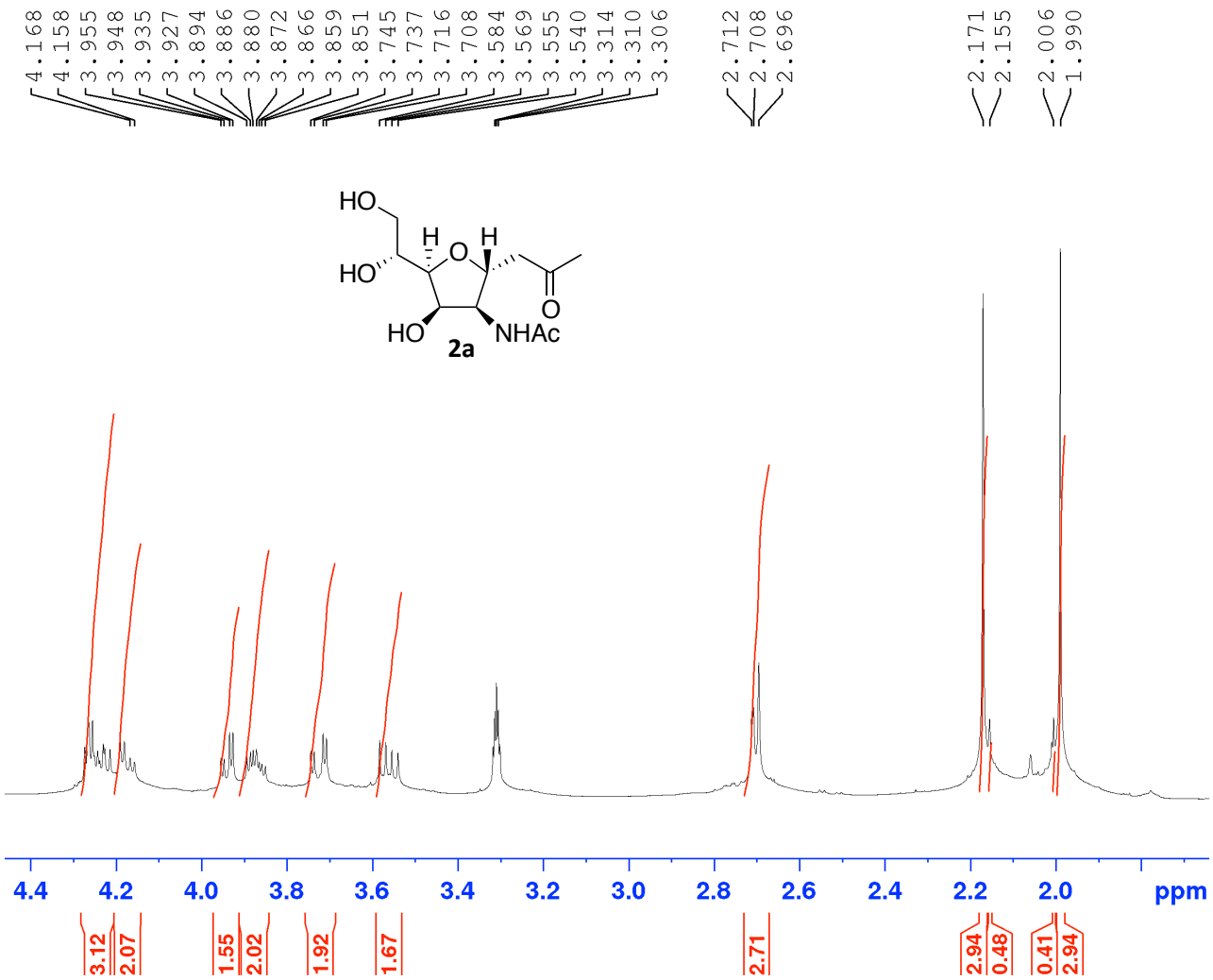


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Current Data Parameters
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EXPNO                  3
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FIDRES          0.122266 Hz
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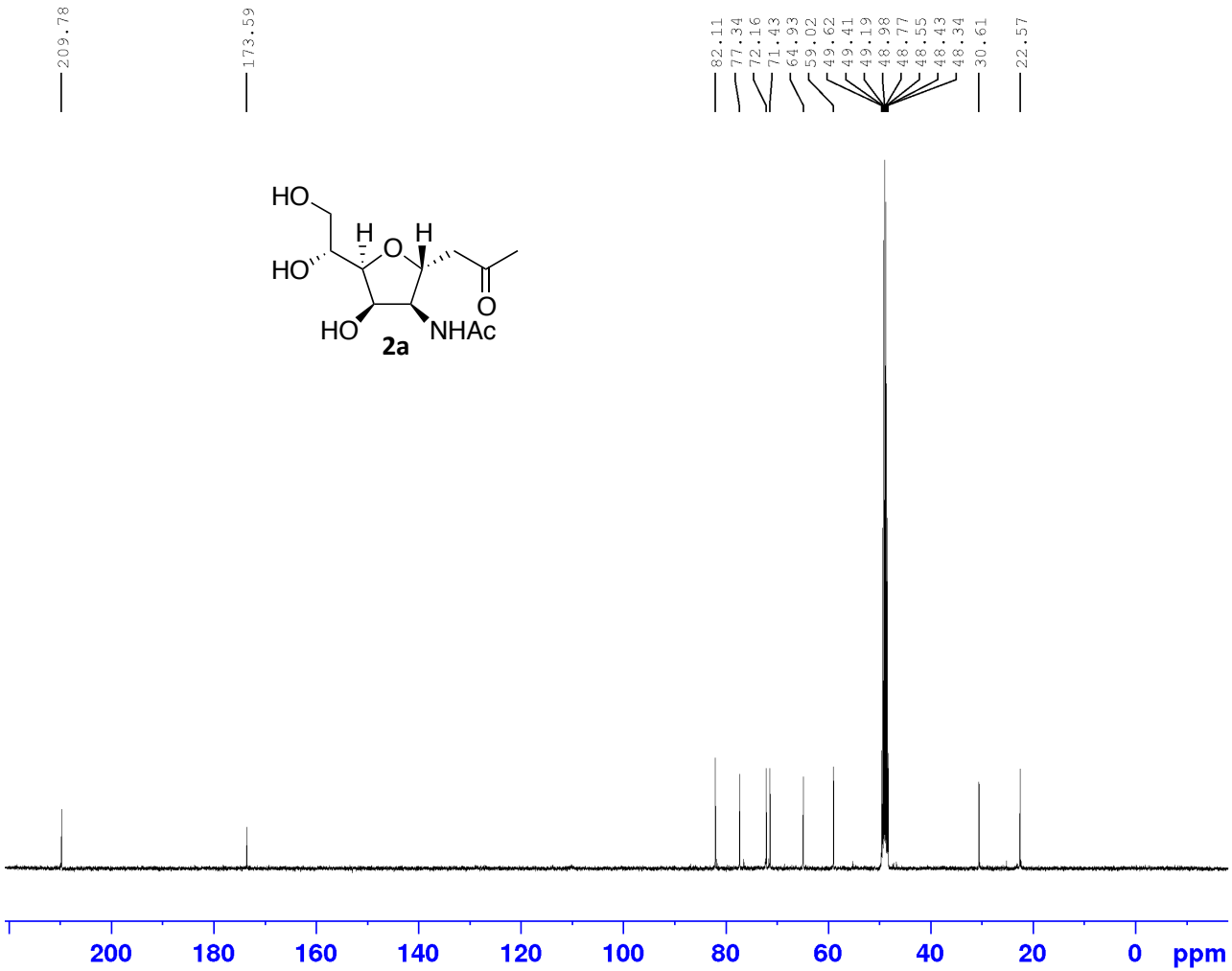


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TD0                    1

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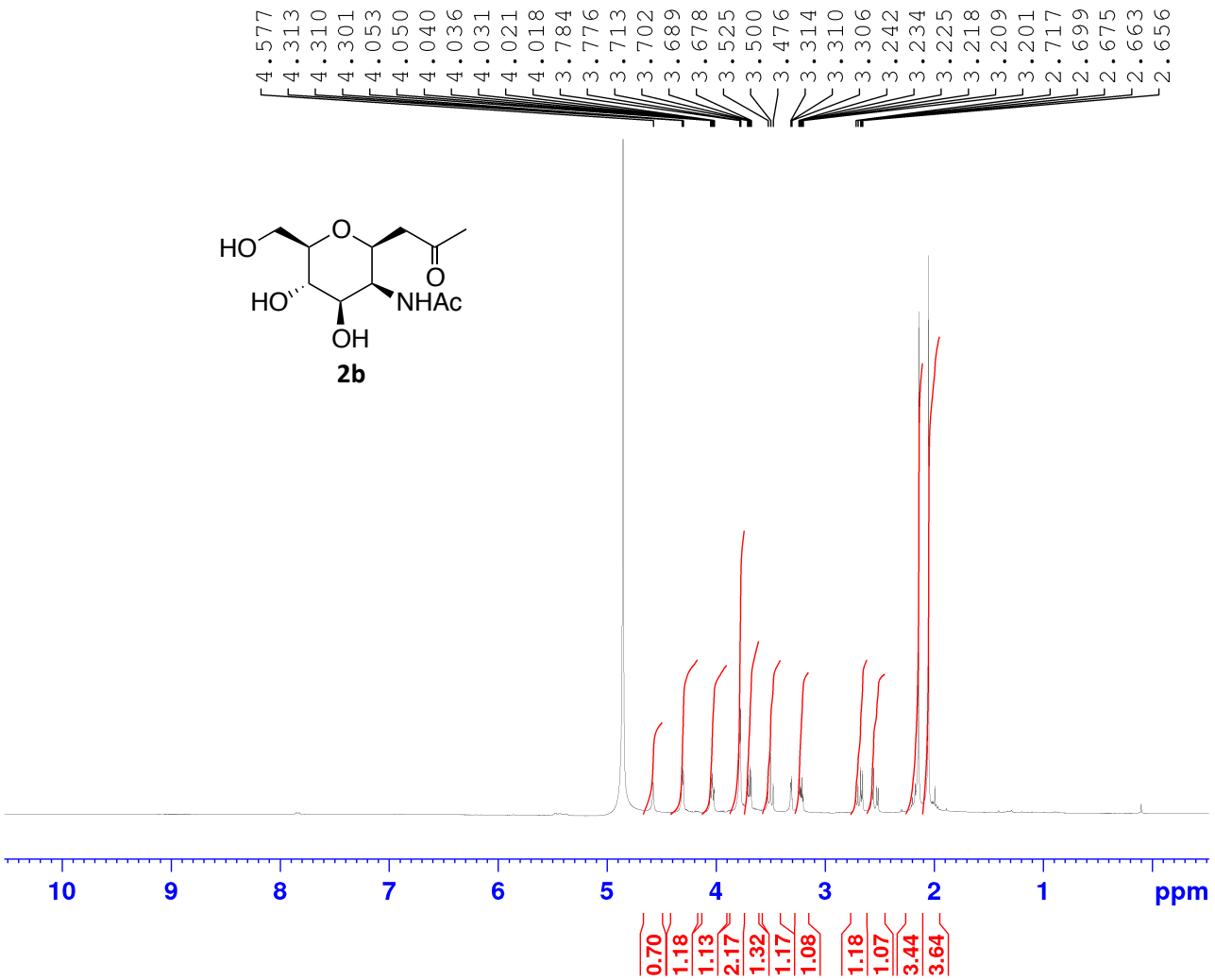


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SOLVENT             MeOD
NS                    16
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FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
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DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                   1H
P1                 15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
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WDW                   EM
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GB        0
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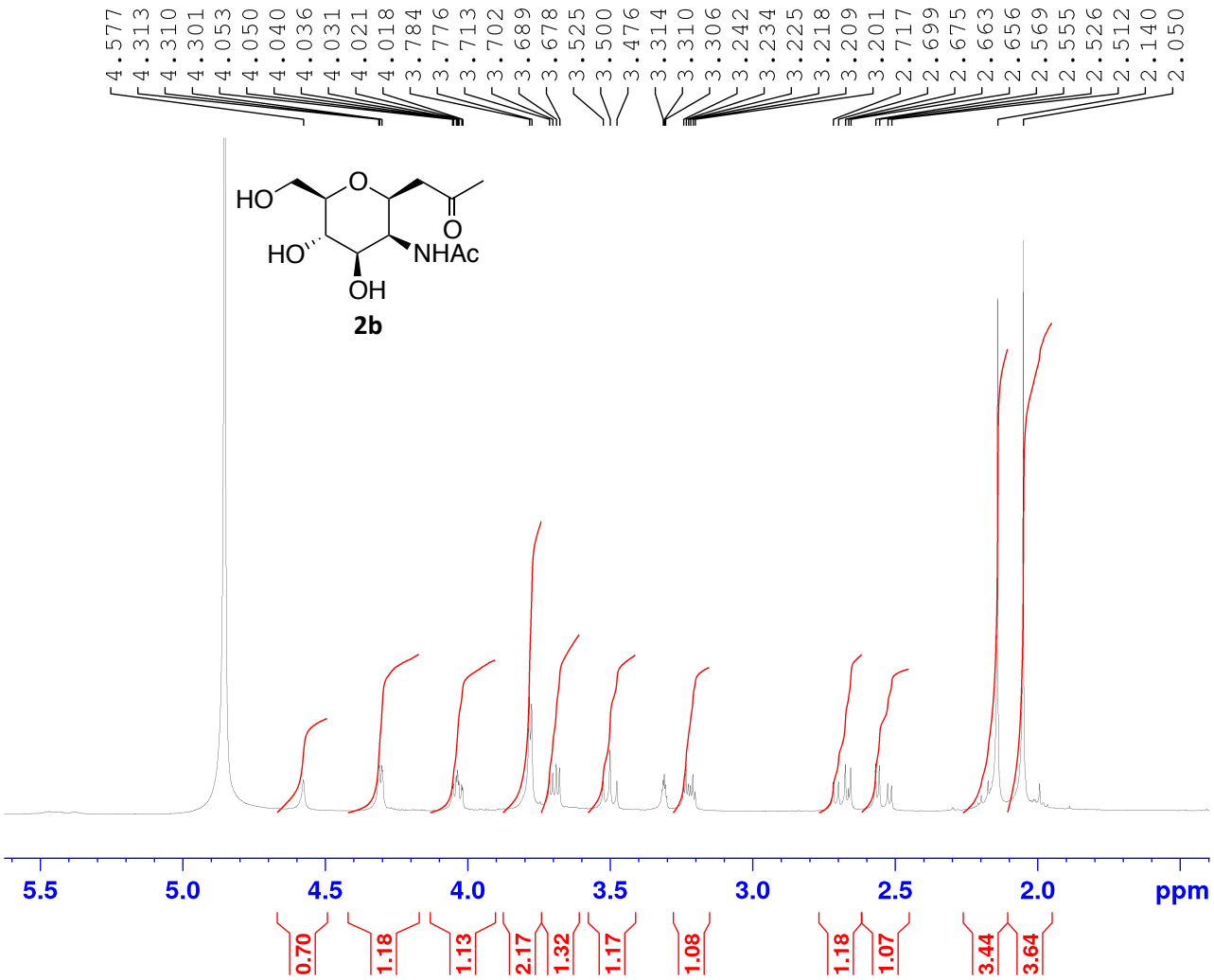


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PROCNO                  1

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PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ               4.0894465 sec
RG                   31.13
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
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NUC1                   1H
P1                  15.00 usec
PLW1           8.00000000 W

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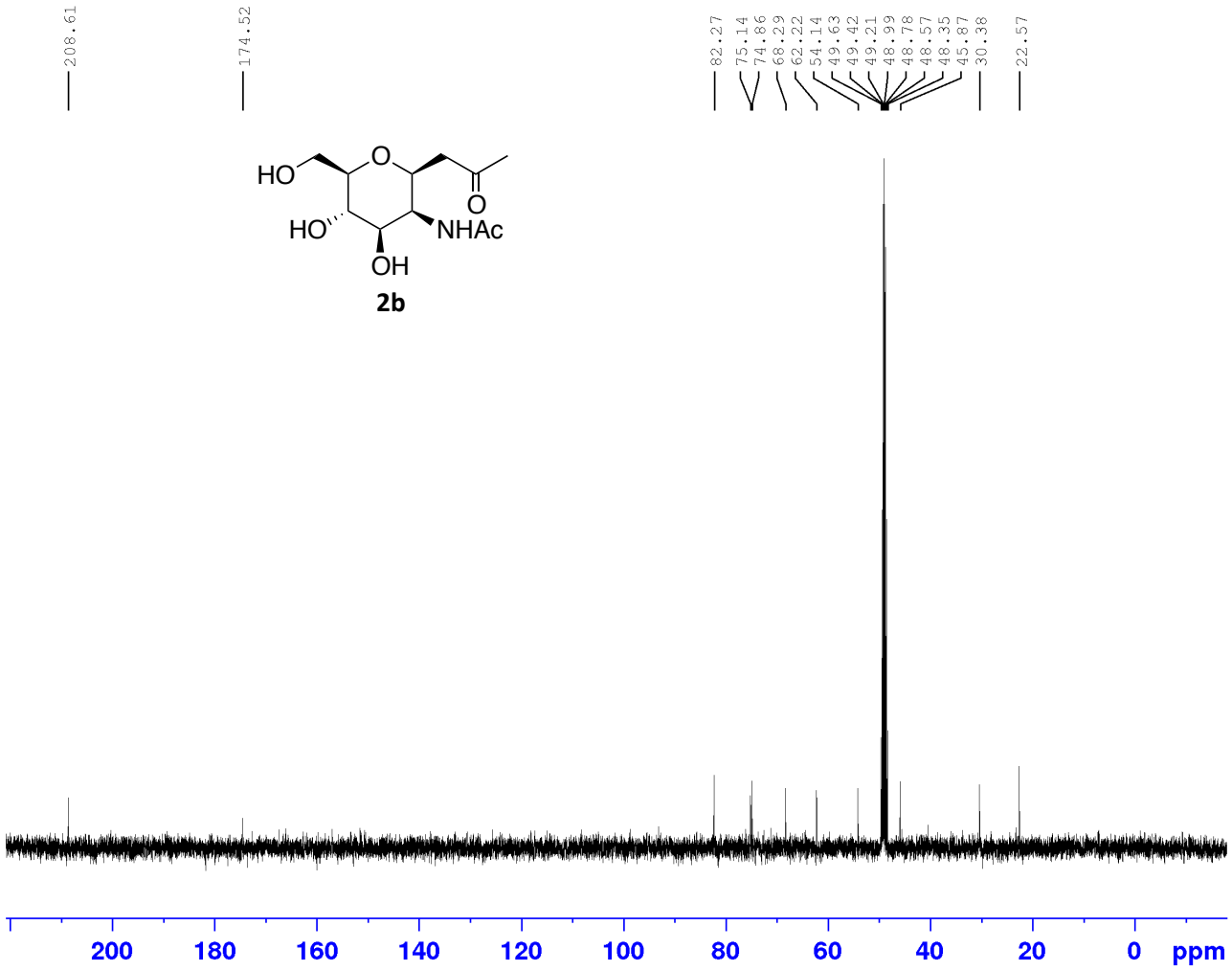


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PULPROG          zgpg30
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SOLVENT            MeOD
NS                   32
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               301.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

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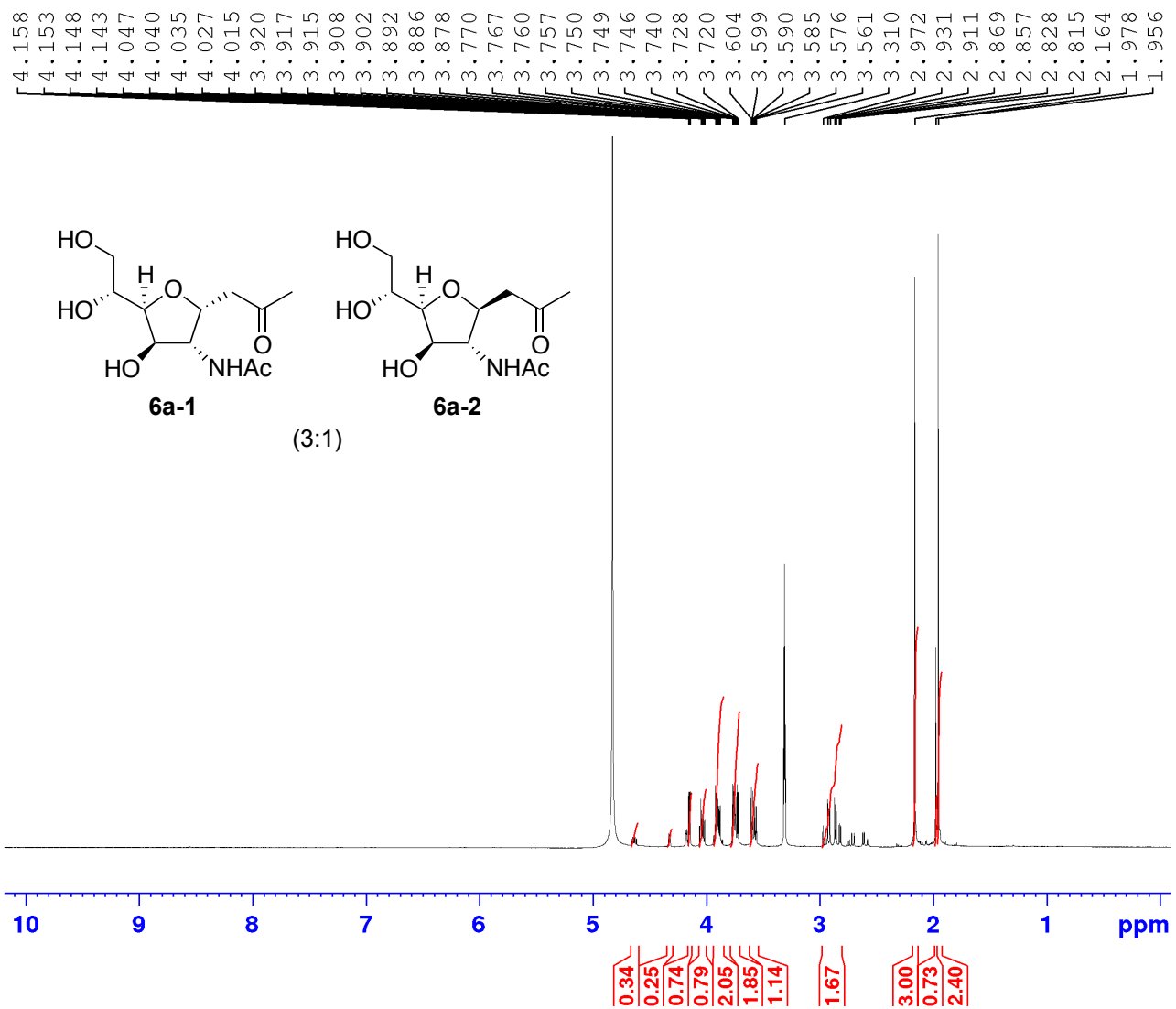


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PROCNO                  1

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PULPROG              zg30
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SOLVENT              MeOD
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  62.88
DW                 62.400 usec
DE                  6.50 usec
TE                 301.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
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NUC1                   1H
P1                  15.00 usec
PLW1          8.00000000 W

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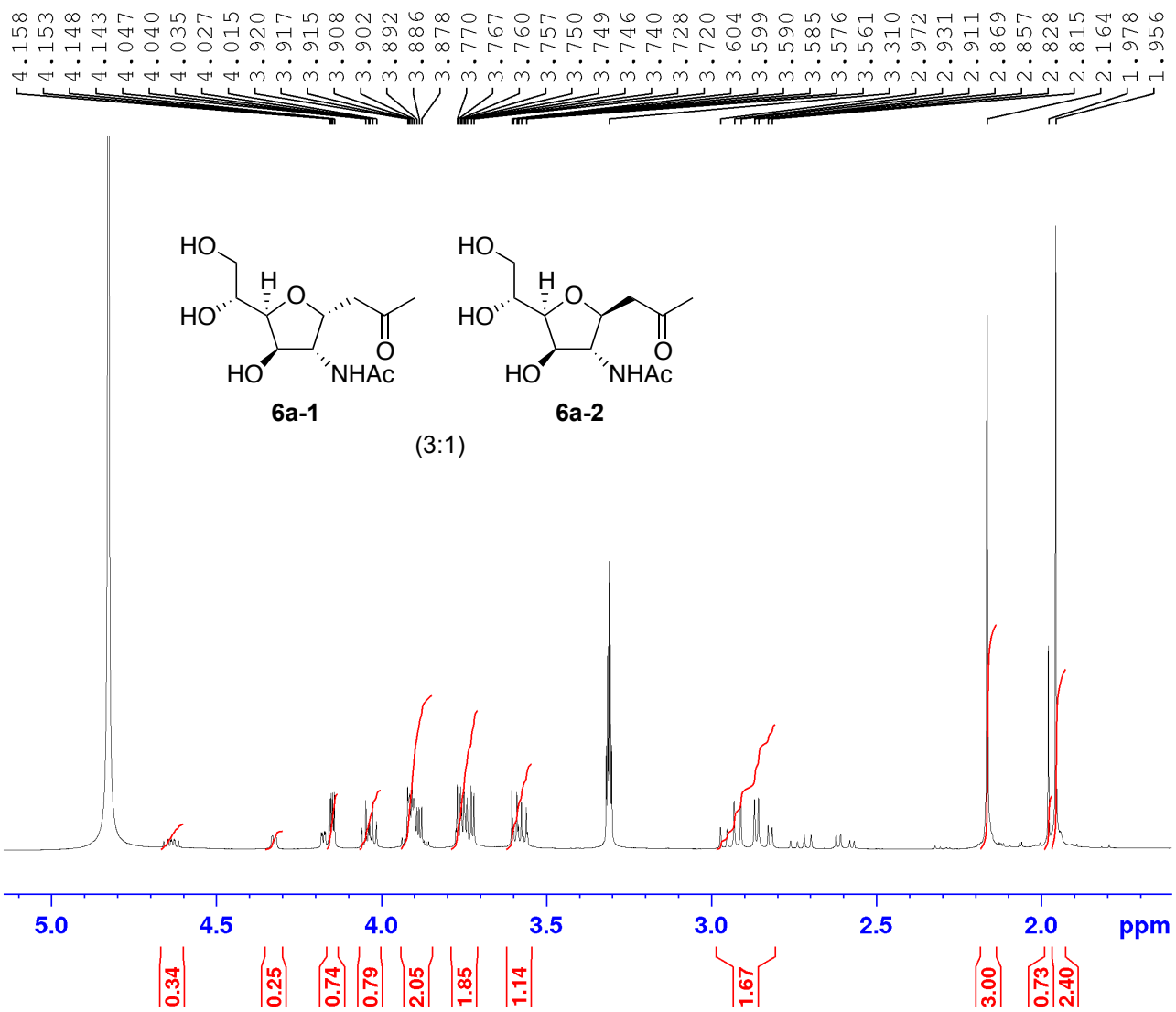


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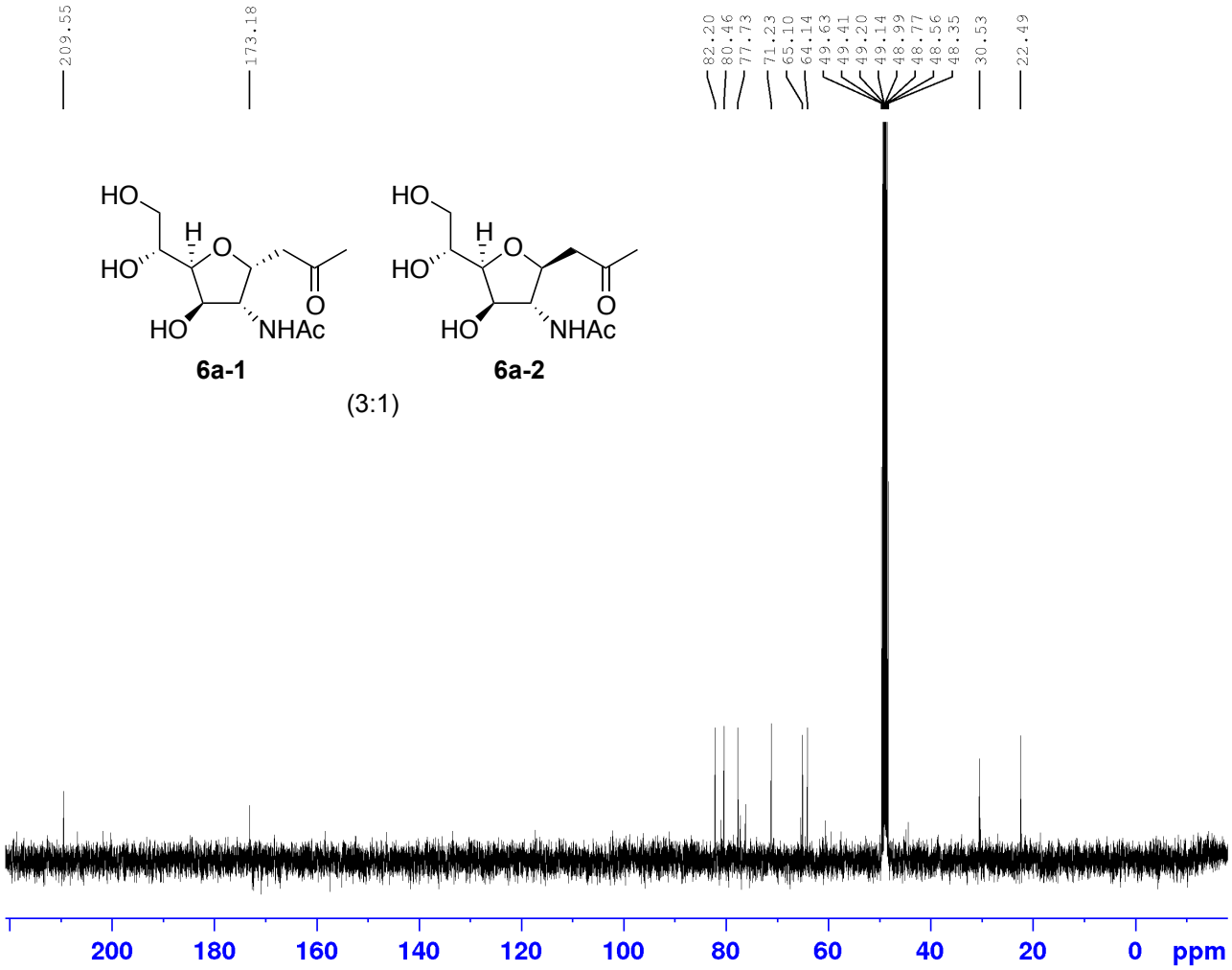
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130322
Time               13.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 62.88
DW                62.400 usec
DE                 6.50 usec
TE                301.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300075 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



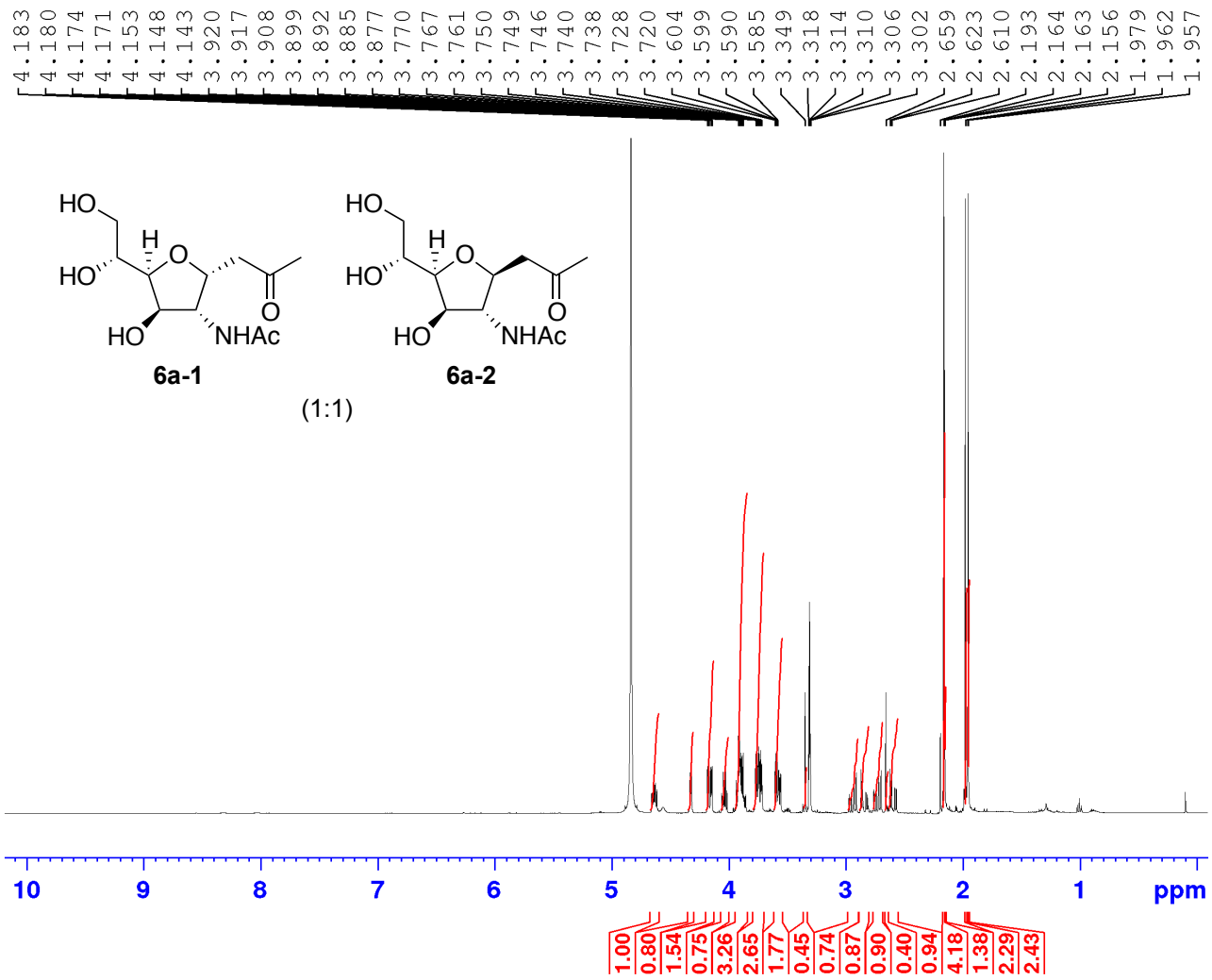

```
Current Data Parameters
NAME           SLJIII162
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130322
Time               13.53
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                  128
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE                301.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6126286 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

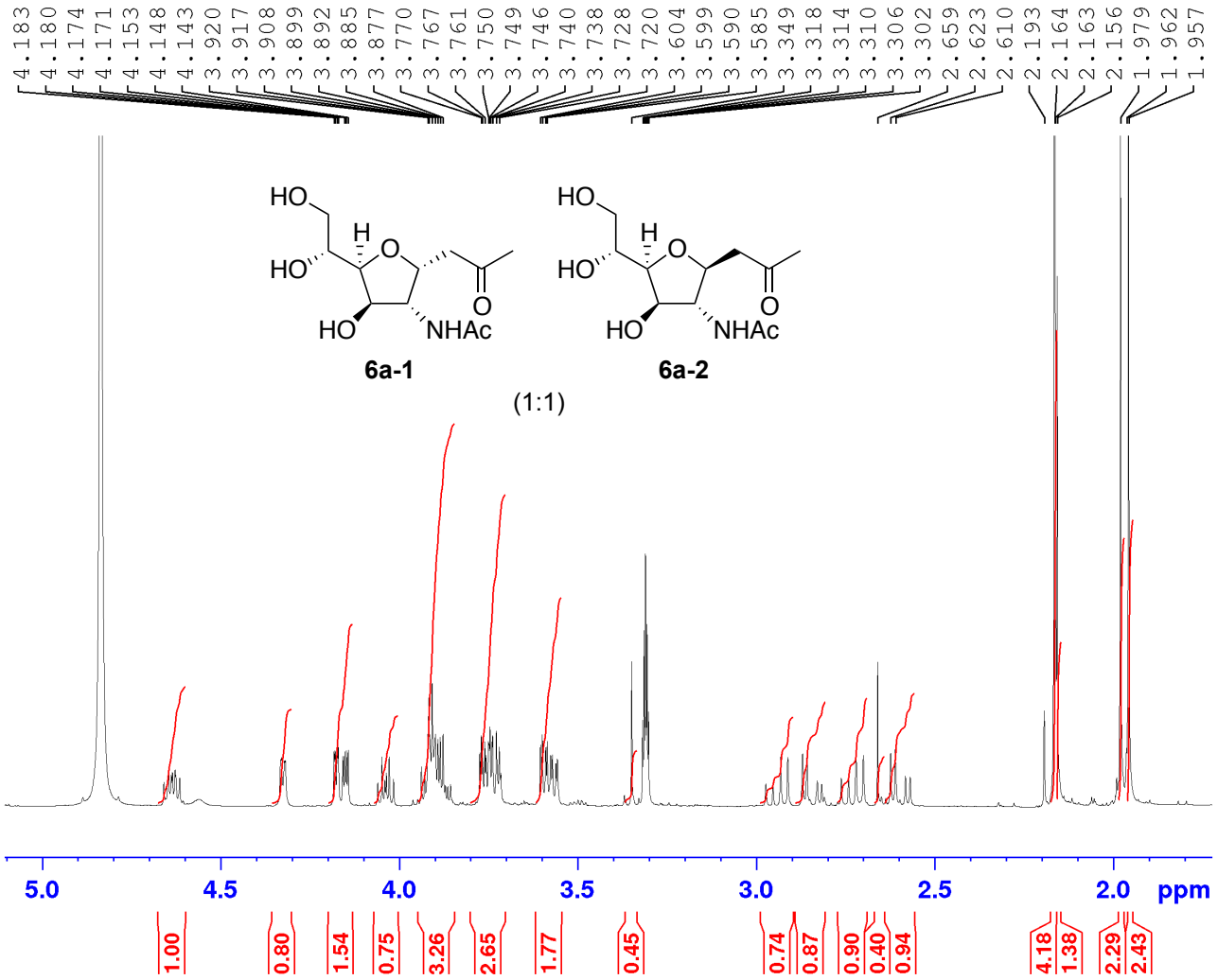



```
Current Data Parameters
NAME           SLJIII237
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130906
Time               14.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
DW                62.400 usec
DE                 6.50 usec
TE                299.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300076 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

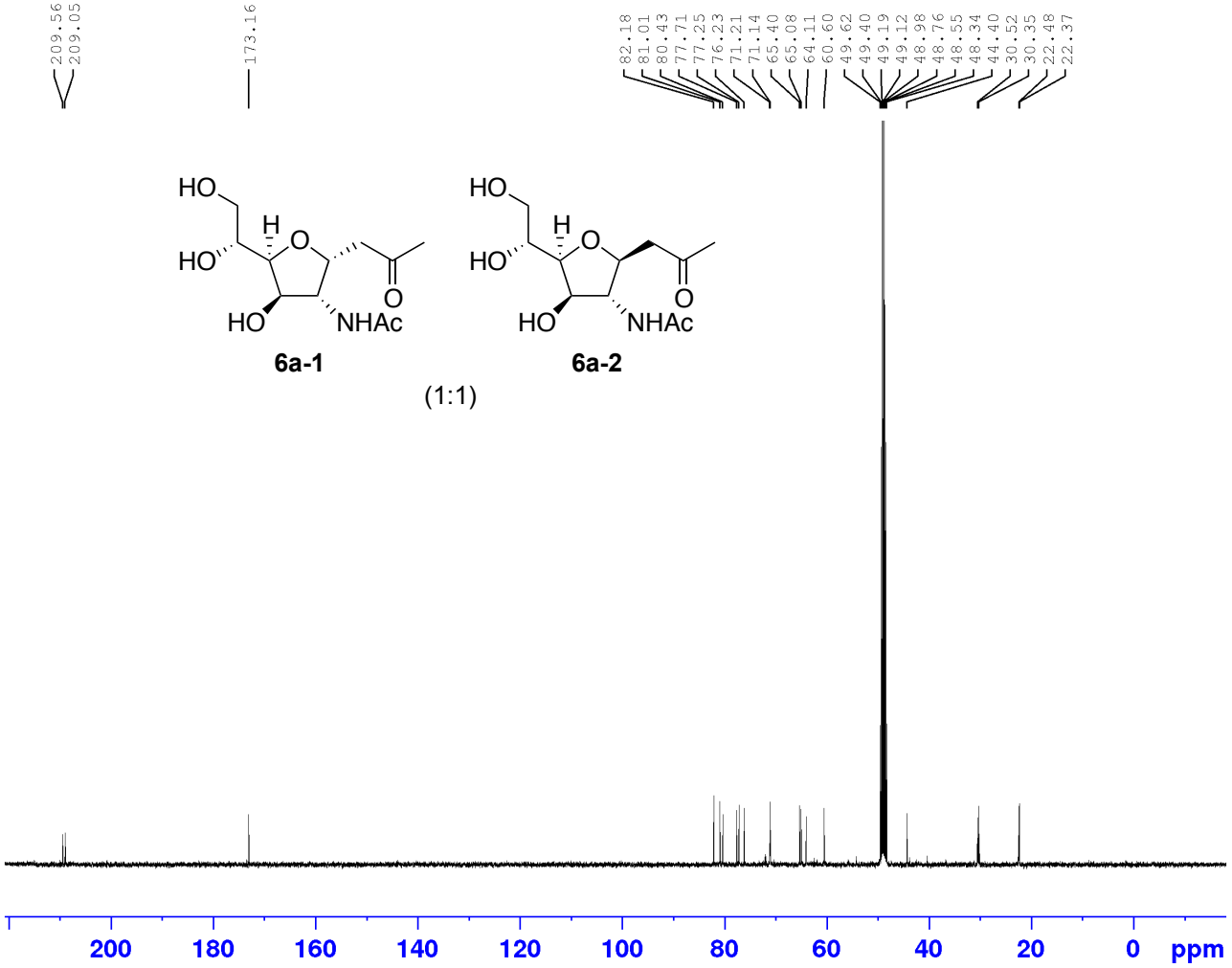



```
Current Data Parameters
NAME           SLJIII237
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130906
Time               14.39
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            MeOD
NS                   16
DS                    2
SWH             8012.820 Hz
FIDRES             0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
DW                 62.400 usec
DE                  6.50 usec
TE                299.9 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300076 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



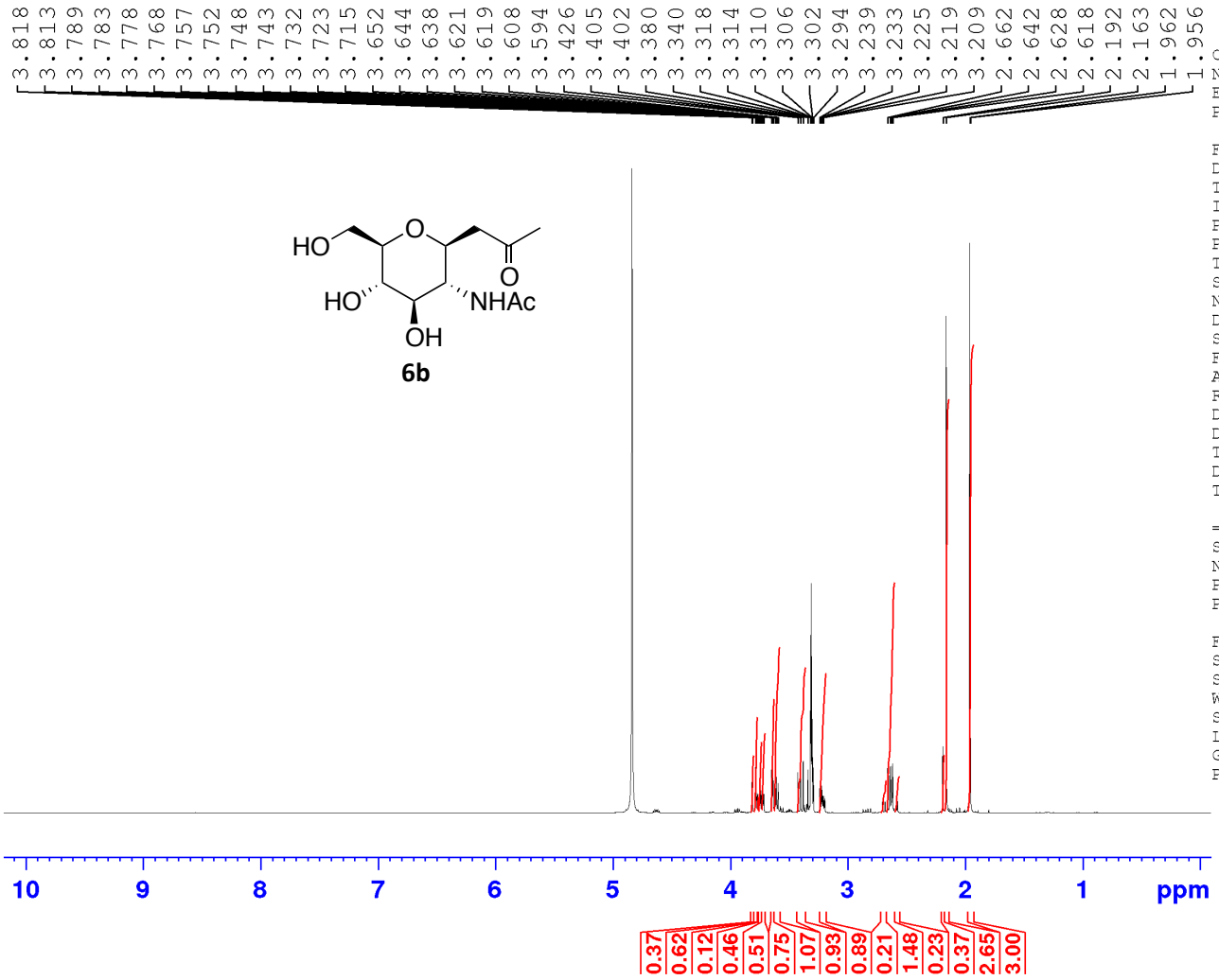

```
Current Data Parameters
NAME           SLJIII237
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130910
Time               17.17
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               301.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6126307 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

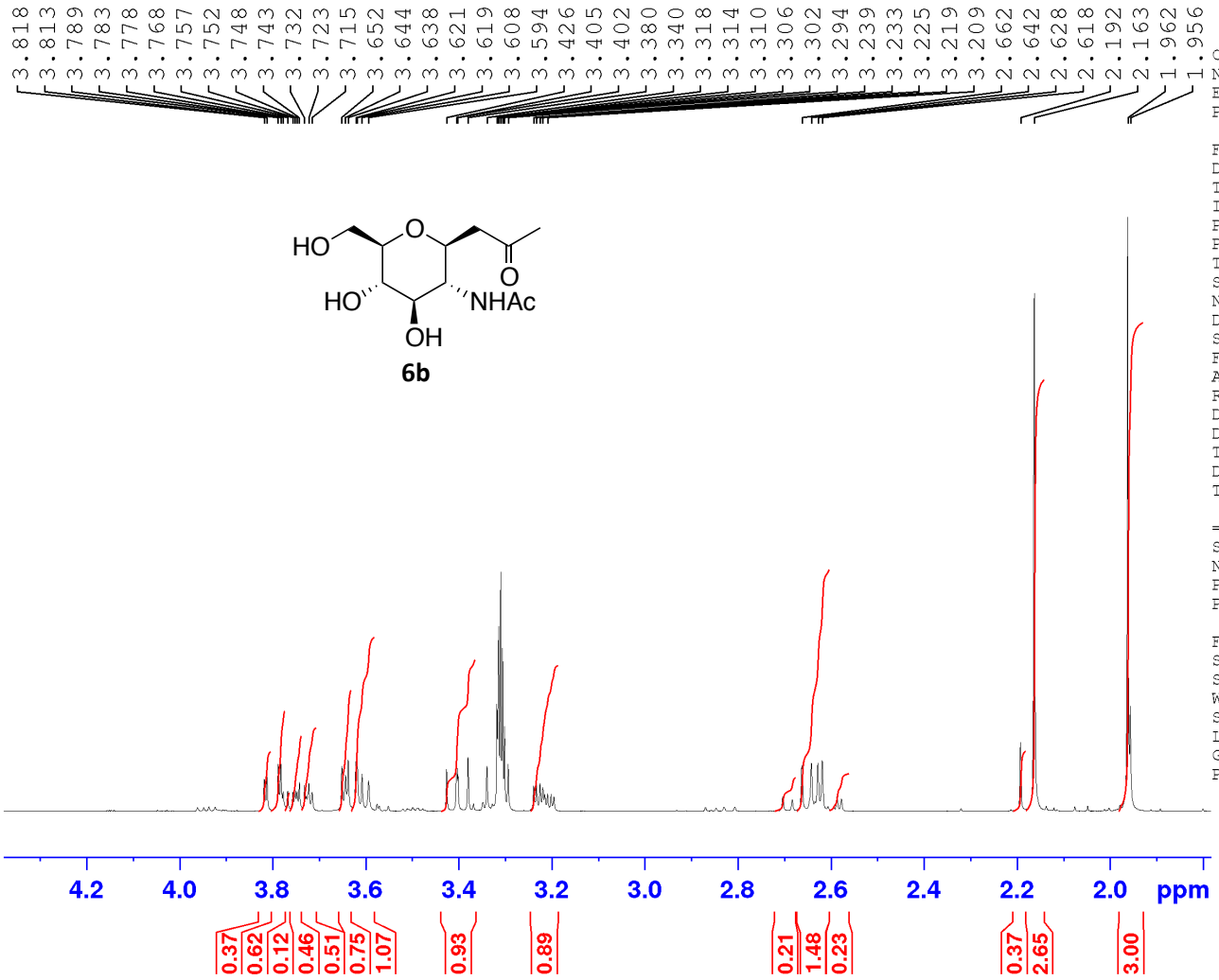



```
Current Data Parameters
NAME            SLJIII162
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130906
Time                15.39
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  62.88
DW                62.400 usec
DE                 6.50 usec
TE                299.8 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300075 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

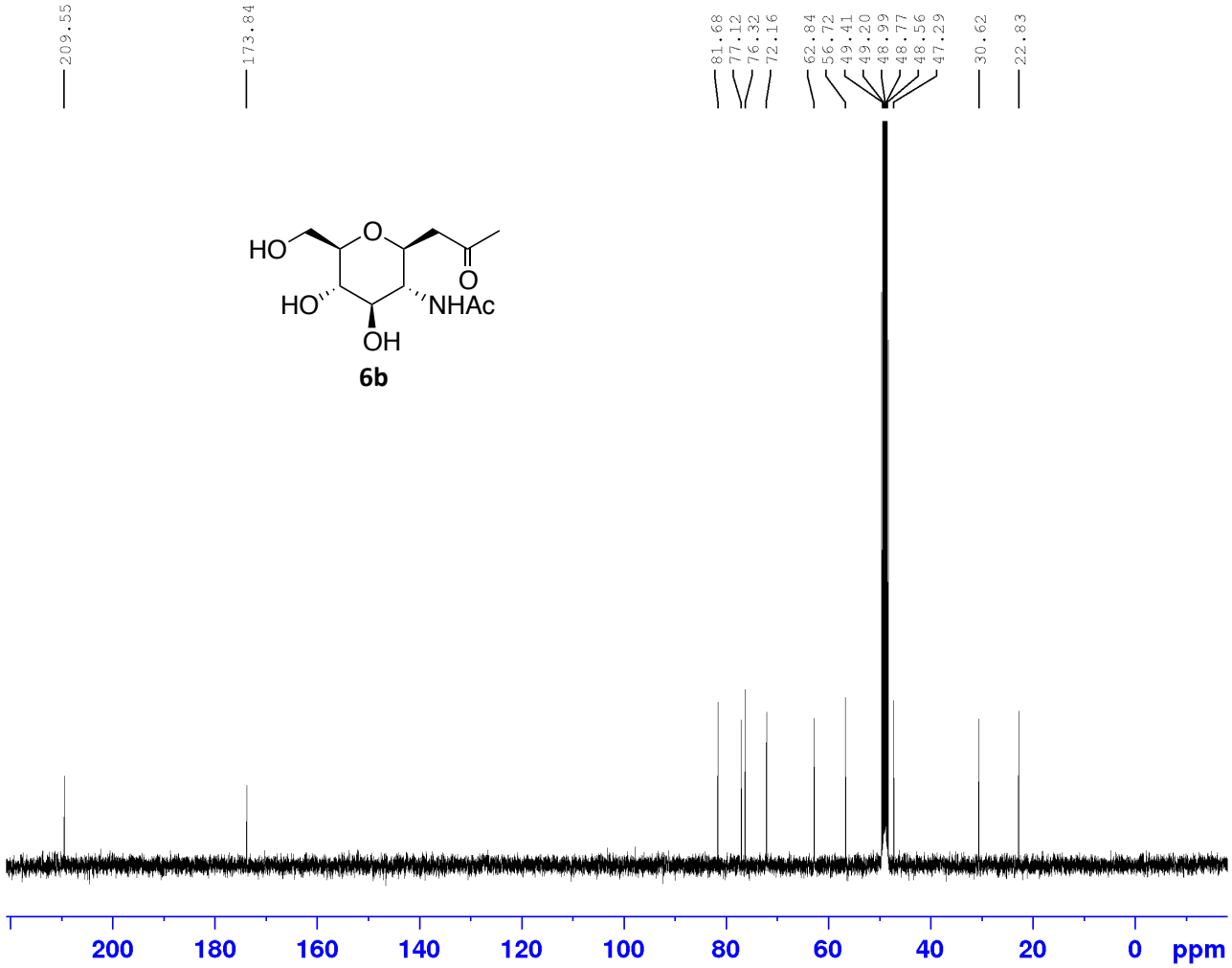



```
Current Data Parameters
NAME            SLJIII162
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130906
Time                15.39
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  62.88
DW                62.400 usec
DE                 6.50 usec
TE                299.8 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300075 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



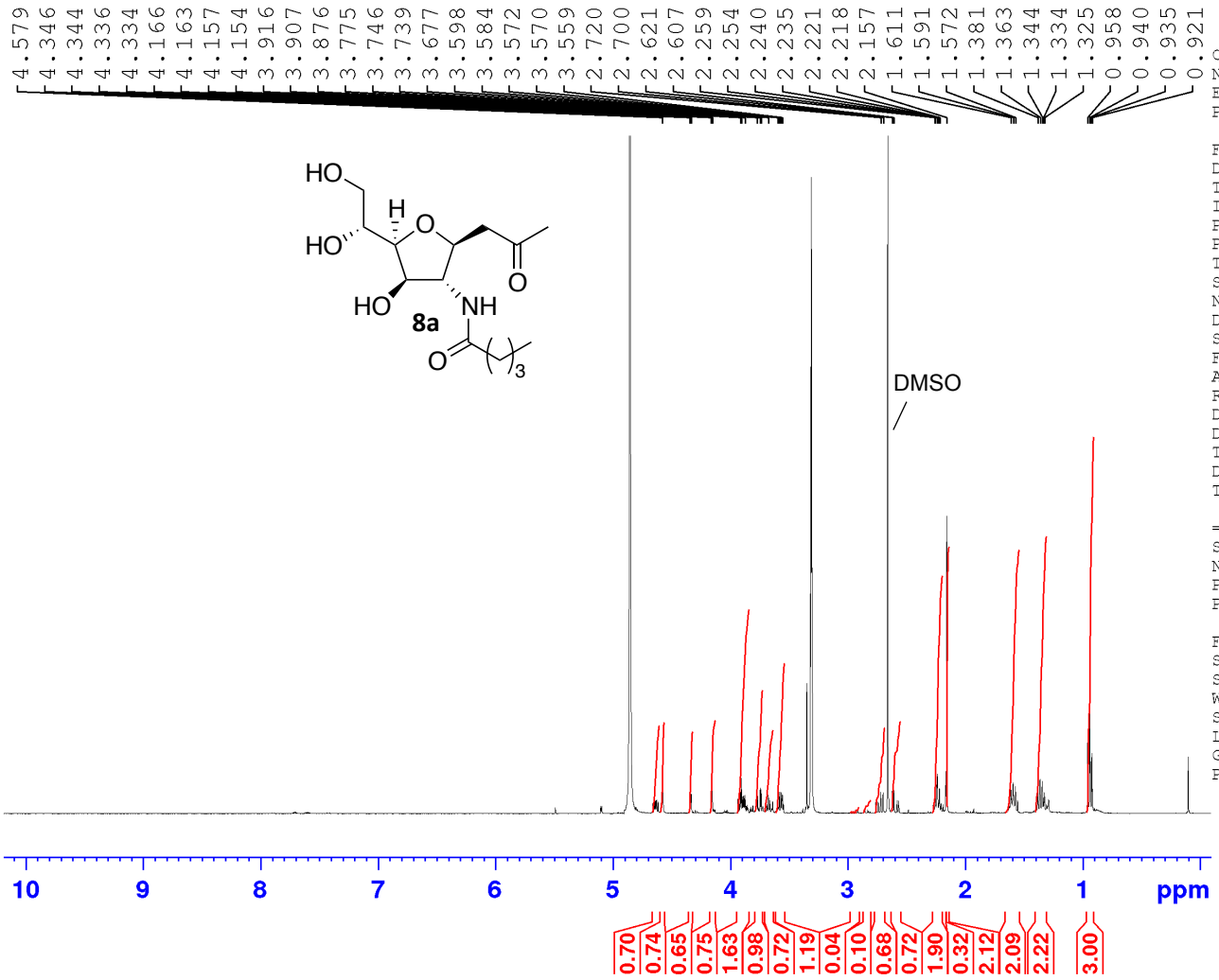

```
Current Data Parameters
NAME            SLJIII162
EXPNO                  26
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150828
Time                11.41
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             MeOD
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE                299.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6126281 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

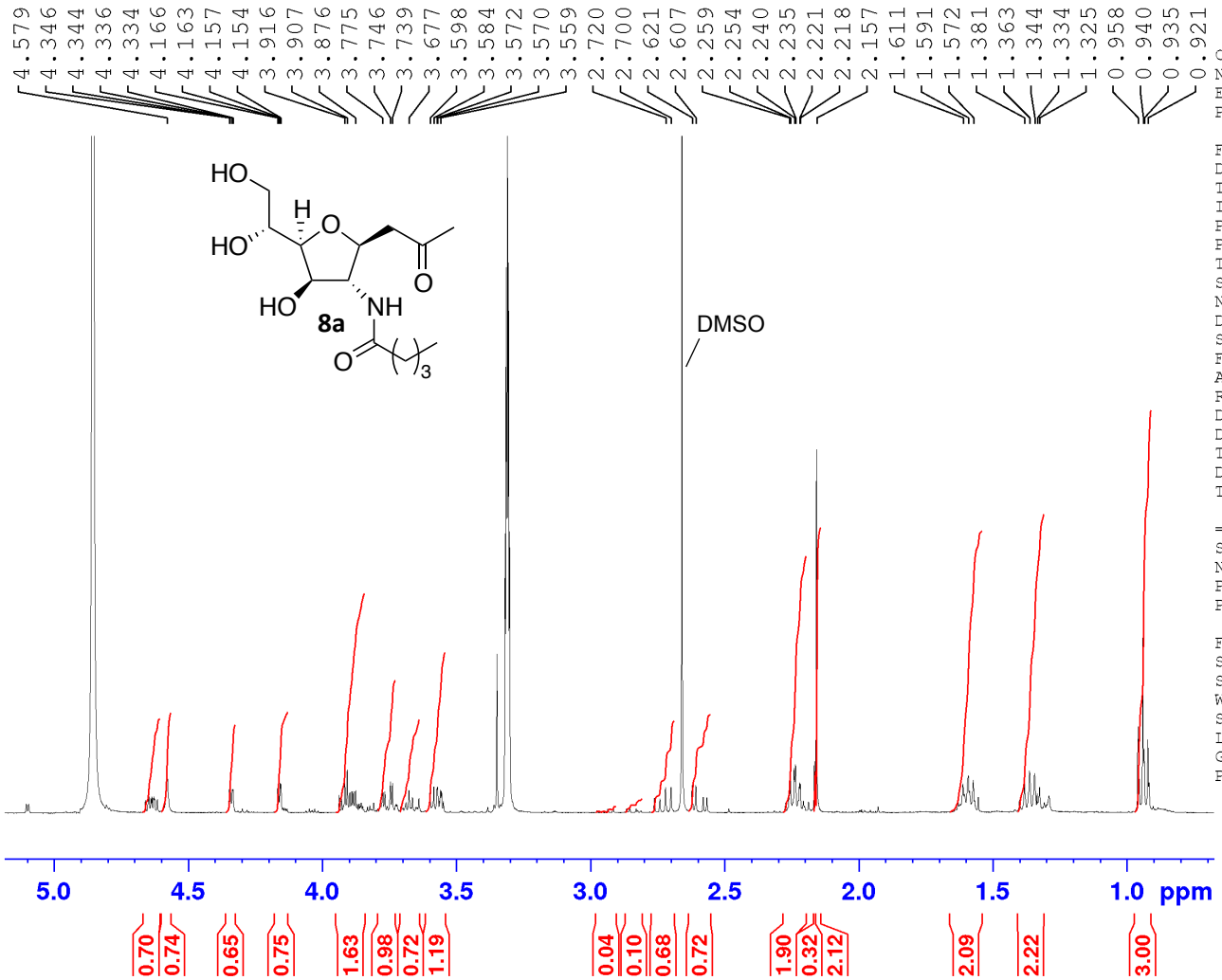



```
Current Data Parameters
NAME            SLJVII477
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140220
Time                14.12
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  71.01
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300077 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

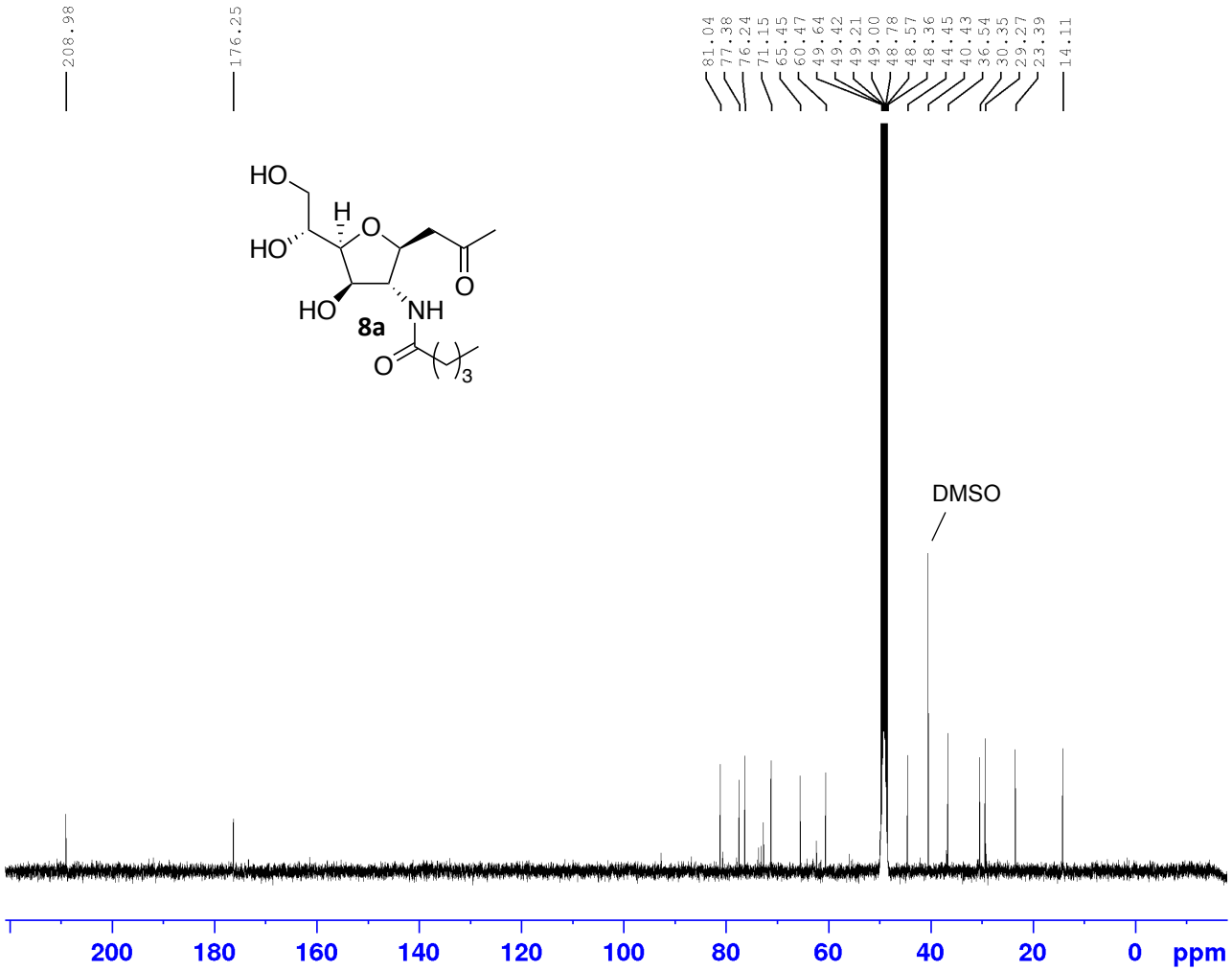



```
Current Data Parameters
NAME            SLJVII477
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140220
Time               14.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 71.01
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



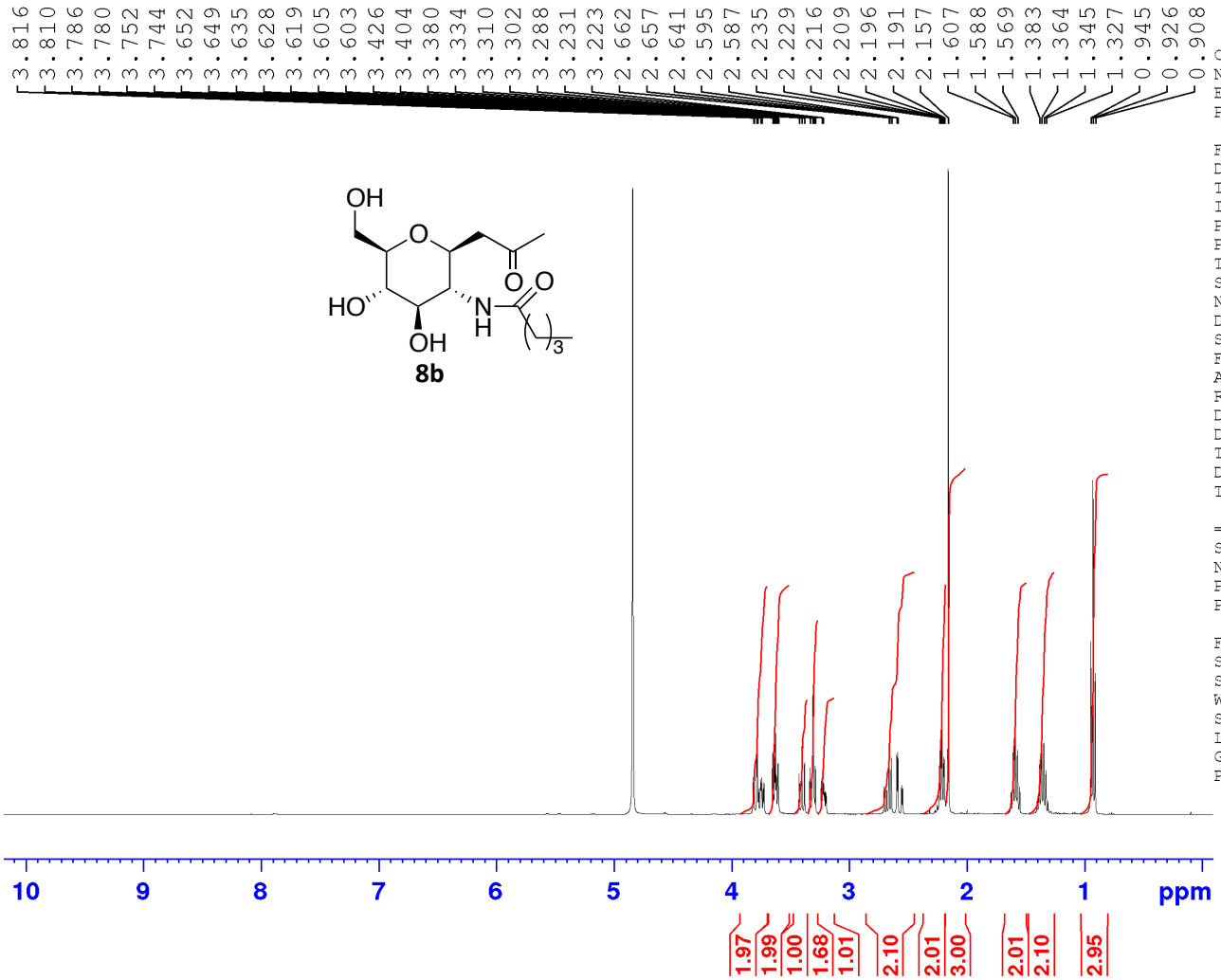

```
Current Data Parameters
NAME           SLJVII477
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140221
Time                8.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                16384
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE                299.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6126271 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

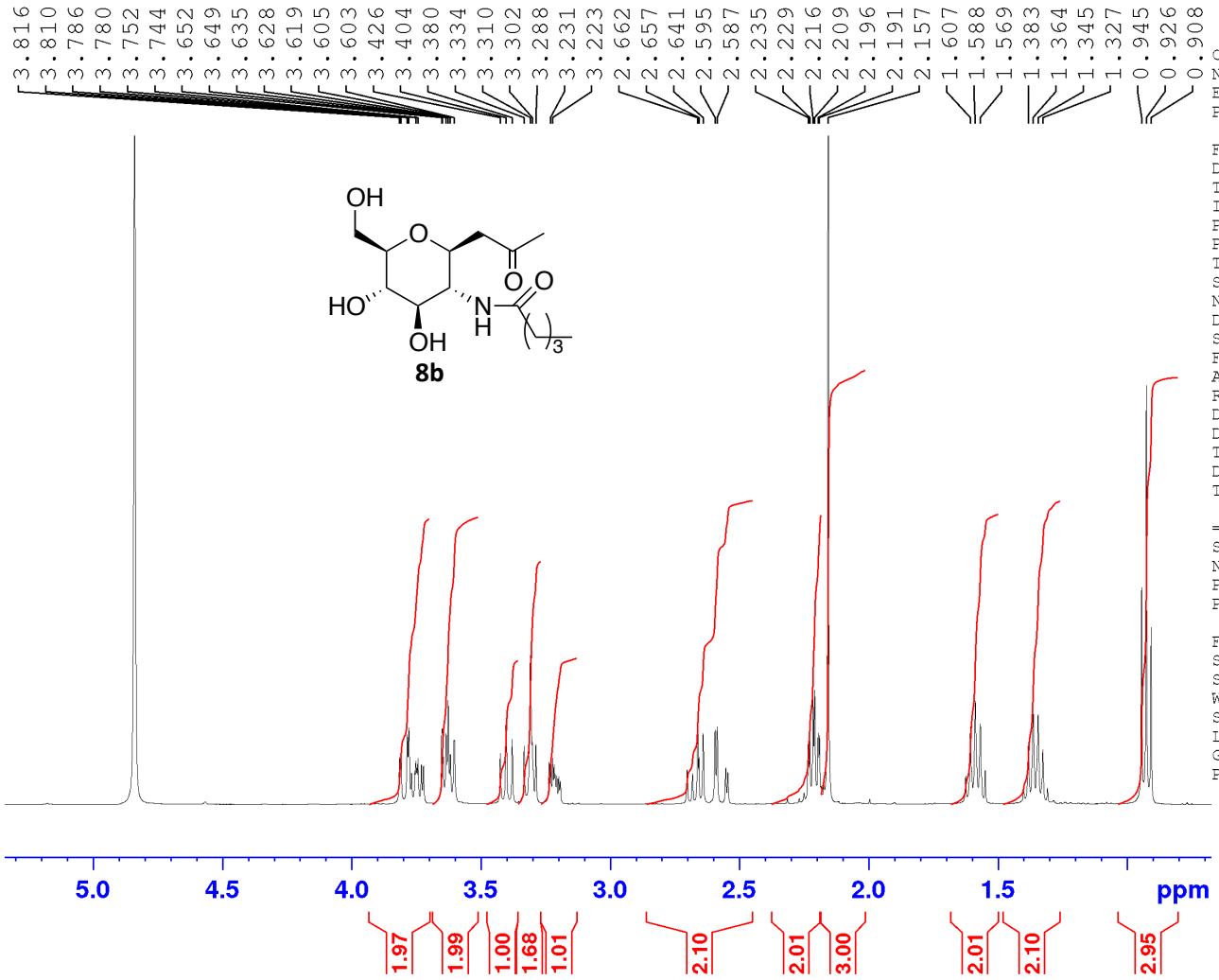



```
Current Data Parameters
NAME                 SLJX754
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20150713
Time                 14.02
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT               MeOD
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                   31.13
DW                  62.400 usec
DE                   6.50 usec
TE                  298.4 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                  15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                   65536
SF            400.1300091 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

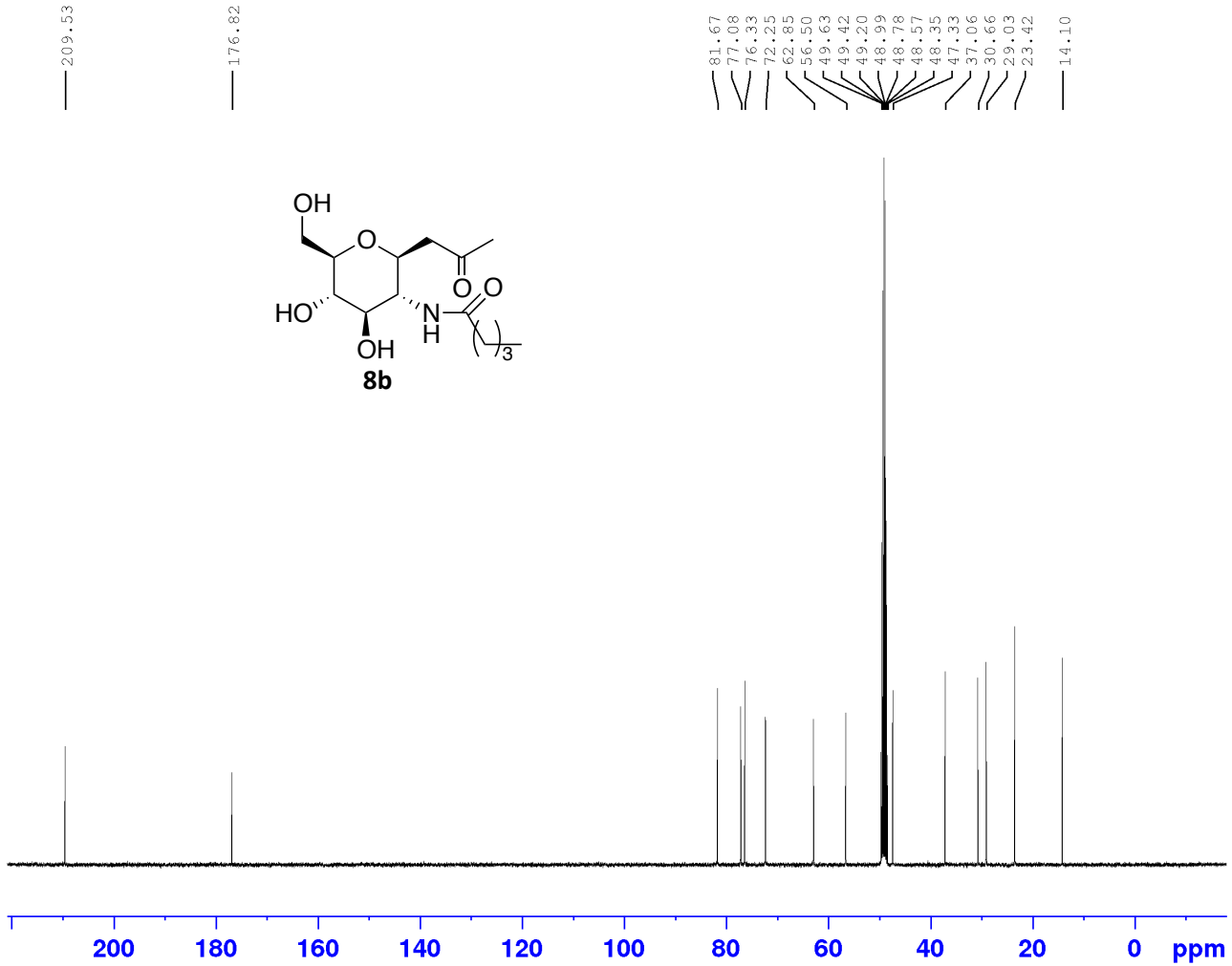



```
Current Data Parameters
NAME                  SLJX754
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150713
Time                  14.02
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894465 sec
RG                    31.13
DW                   62.400 usec
DE                    6.50 usec
TE                   298.4 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                       1H
P1                      15.00 usec
PLW1             8.00000000 W

F2 - Processing parameters
SI                        65536
SF              400.1300091 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



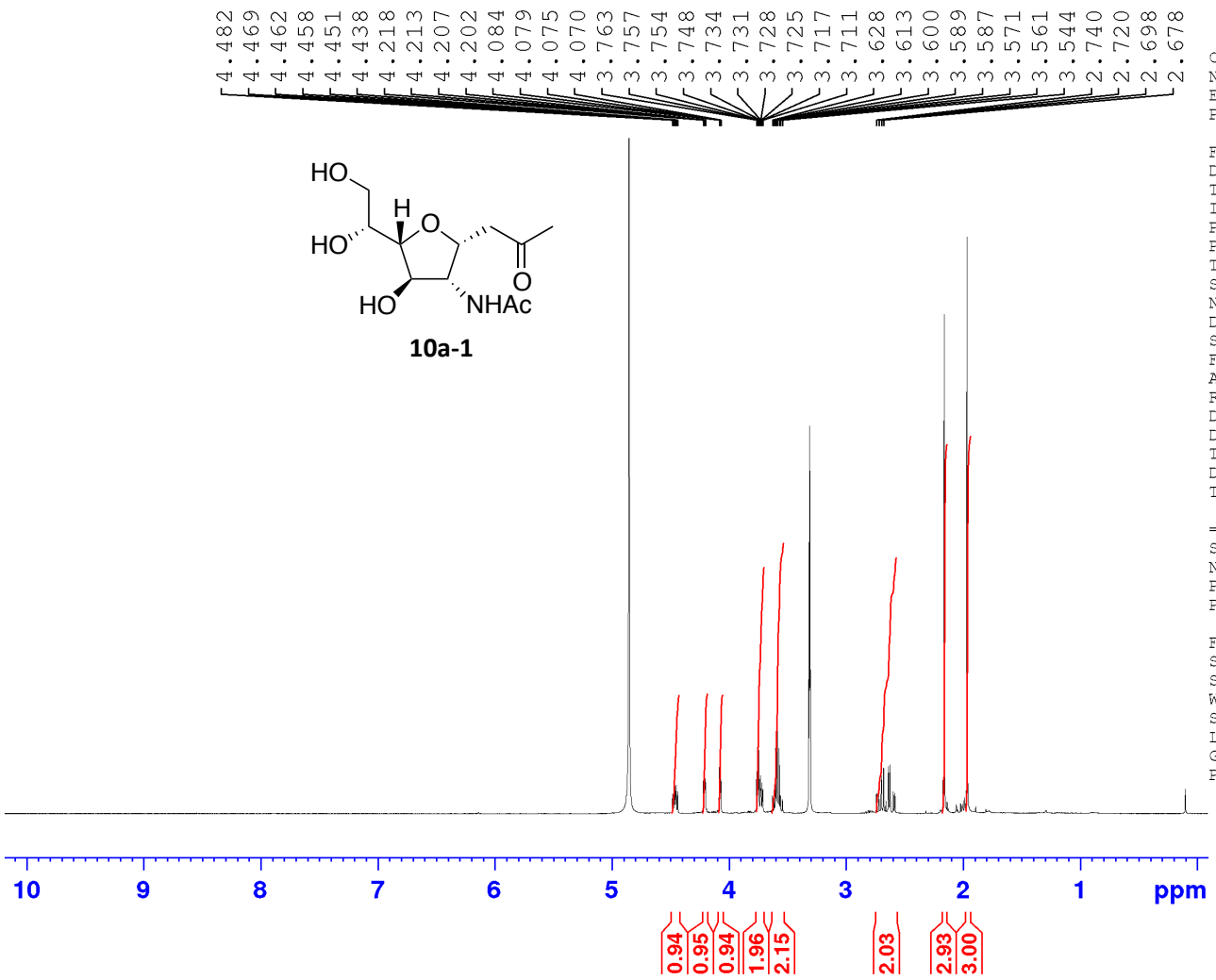

```
Current Data Parameters
NAME             SLJX754
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150713
Time               17.12
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             MeOD
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               299.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6126300 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

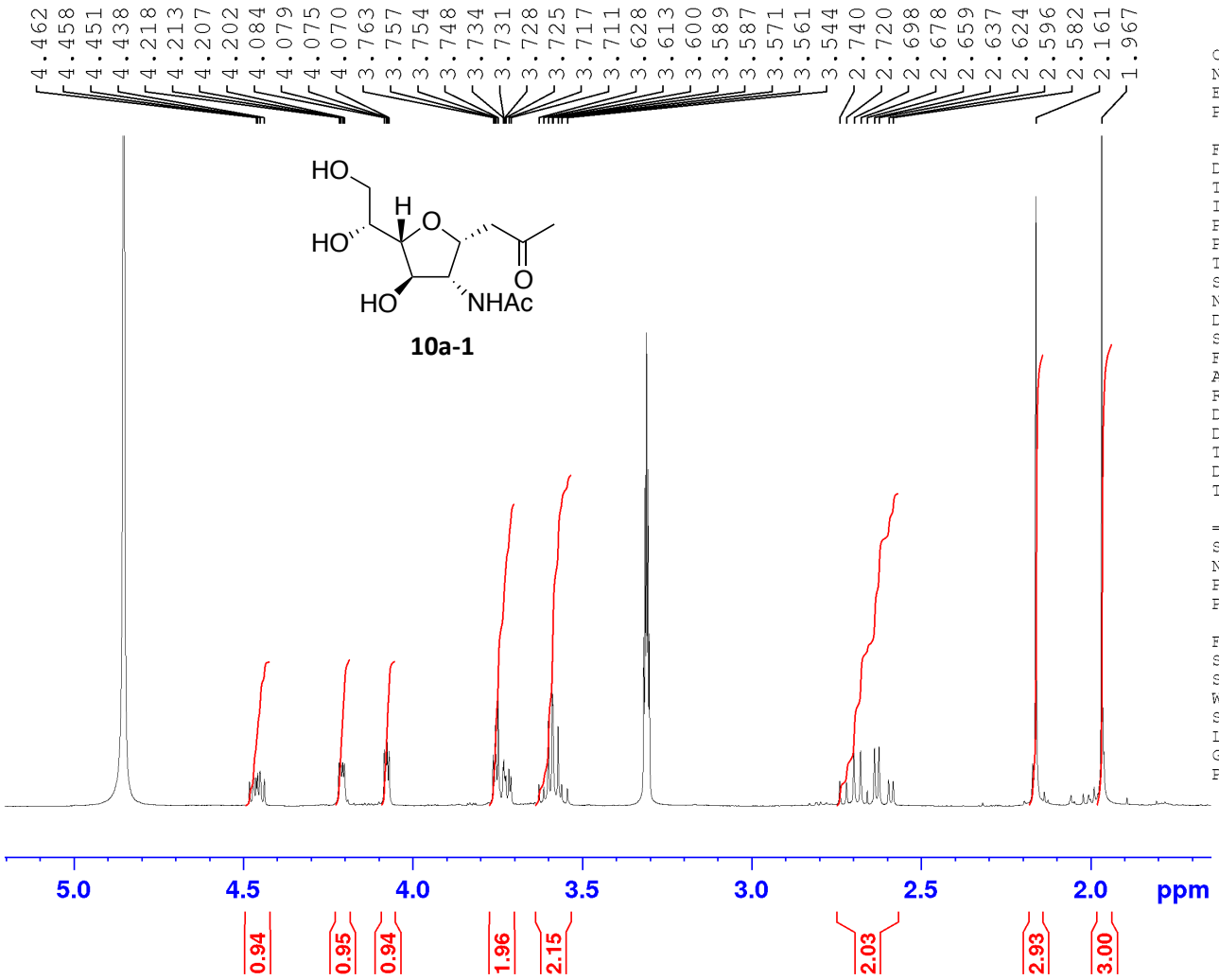



```
Current Data Parameters
NAME              SLJIV250
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20130925
Time                 10.41
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT               MeOD
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                   71.01
DW                  62.400 usec
DE                   6.50 usec
TE                  298.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                  15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                   65536
SF            400.1300075 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

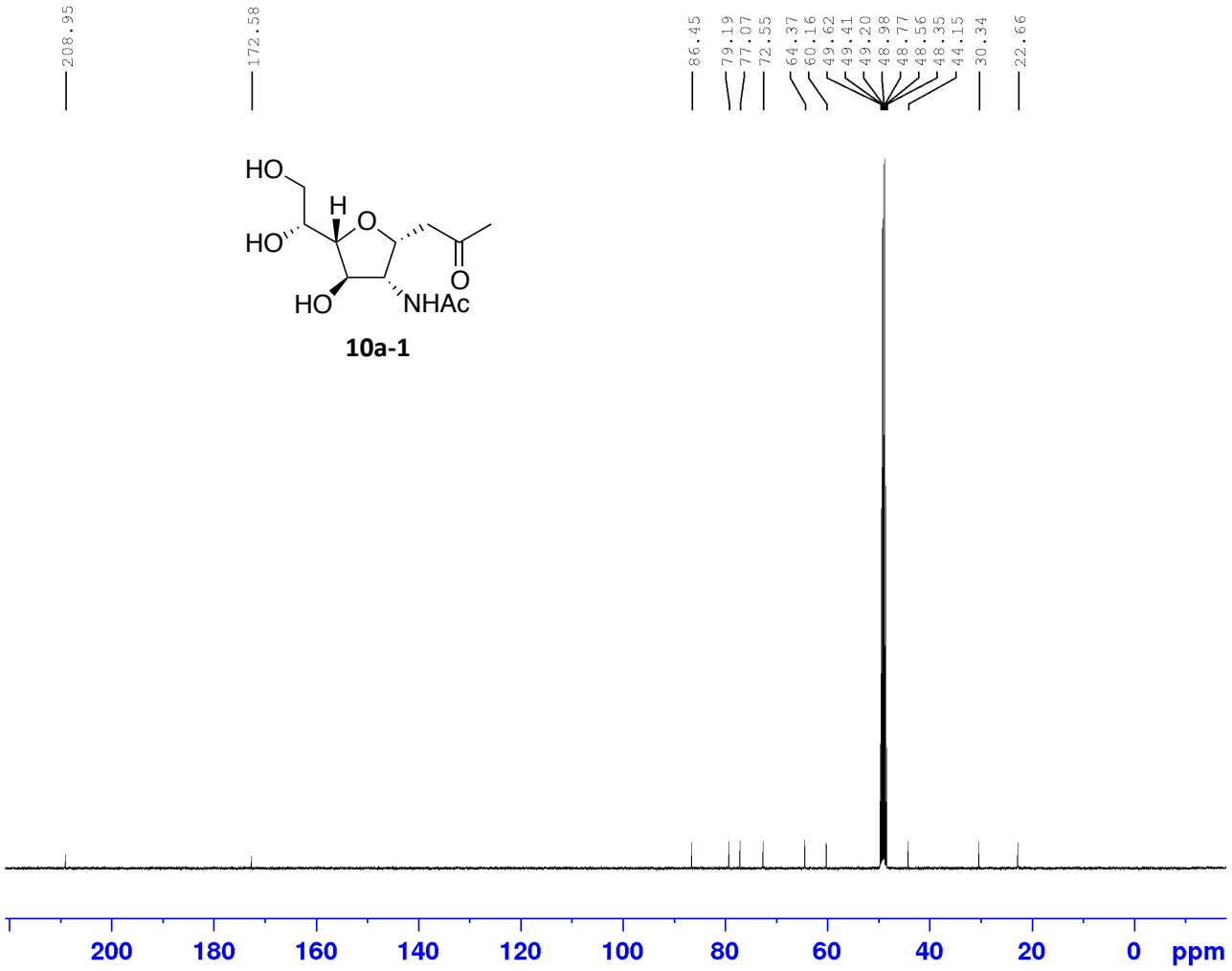



```
Current Data Parameters
NAME              SLJIV250
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20130925
Time                 10.41
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                   71.01
DW                 62.400 usec
DE                  6.50 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                    1H
P1                  15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                  65536
SF           400.1300075 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



10a-1

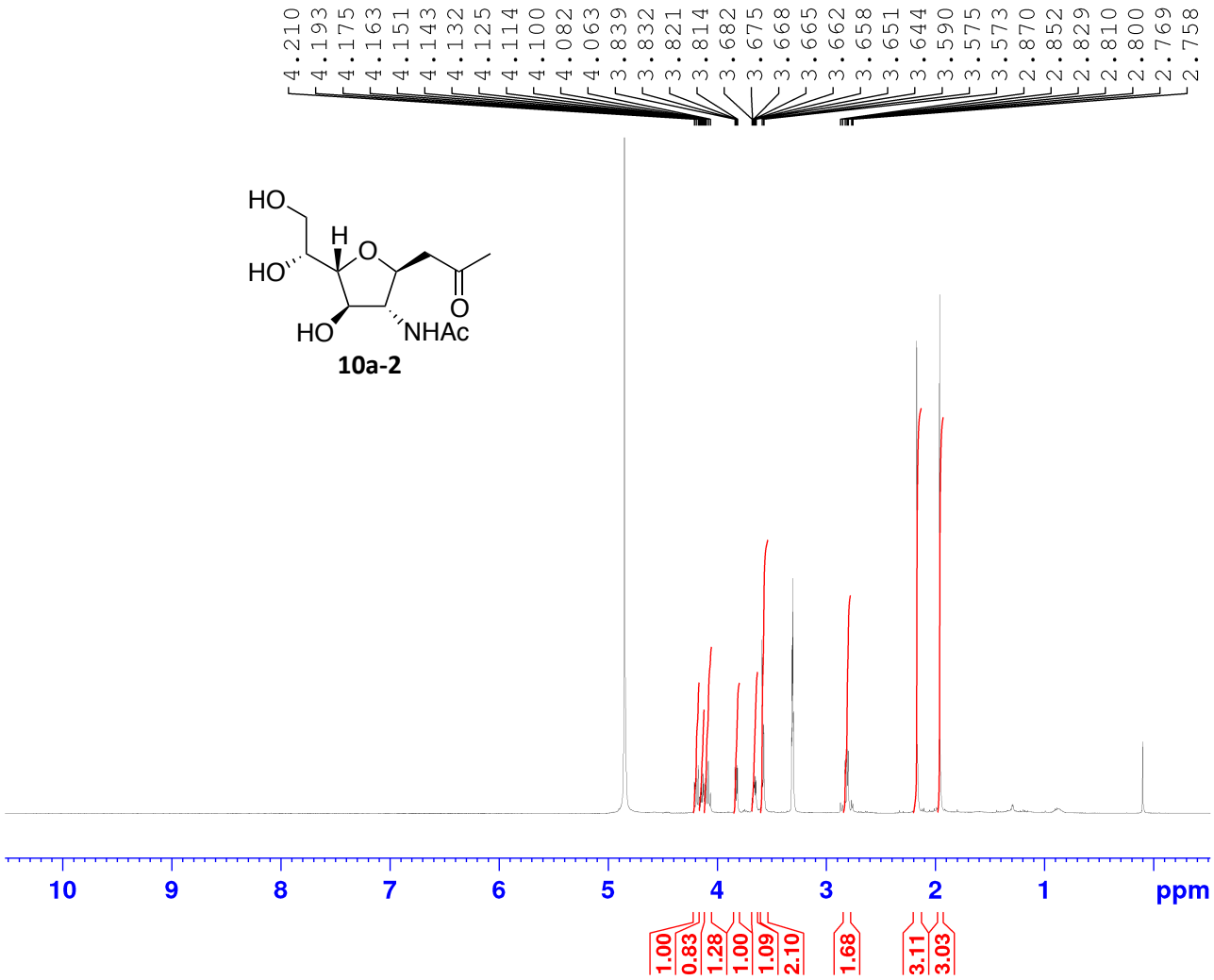
```
Current Data Parameters
NAME                  SLJIV250
EXPNO                        8
PROCNO                       1

F2 - Acquisition Parameters
Date_                20130925
Time                    13.01
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                   MeOD
NS                        2048
DS                           4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                  1.3631488 sec
RG                      195.88
DW                      20.800 usec
DE                       6.50 usec
TE                      299.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             100.6228293 MHz
NUC1                      13C
P1                      10.00 usec
PLW1            70.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                   80.00 usec
PLW2             8.00000000 W
PLW12            0.28125000 W
PLW13            0.28125000 W

F2 - Processing parameters
SI                       32768
SF                100.6126286 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

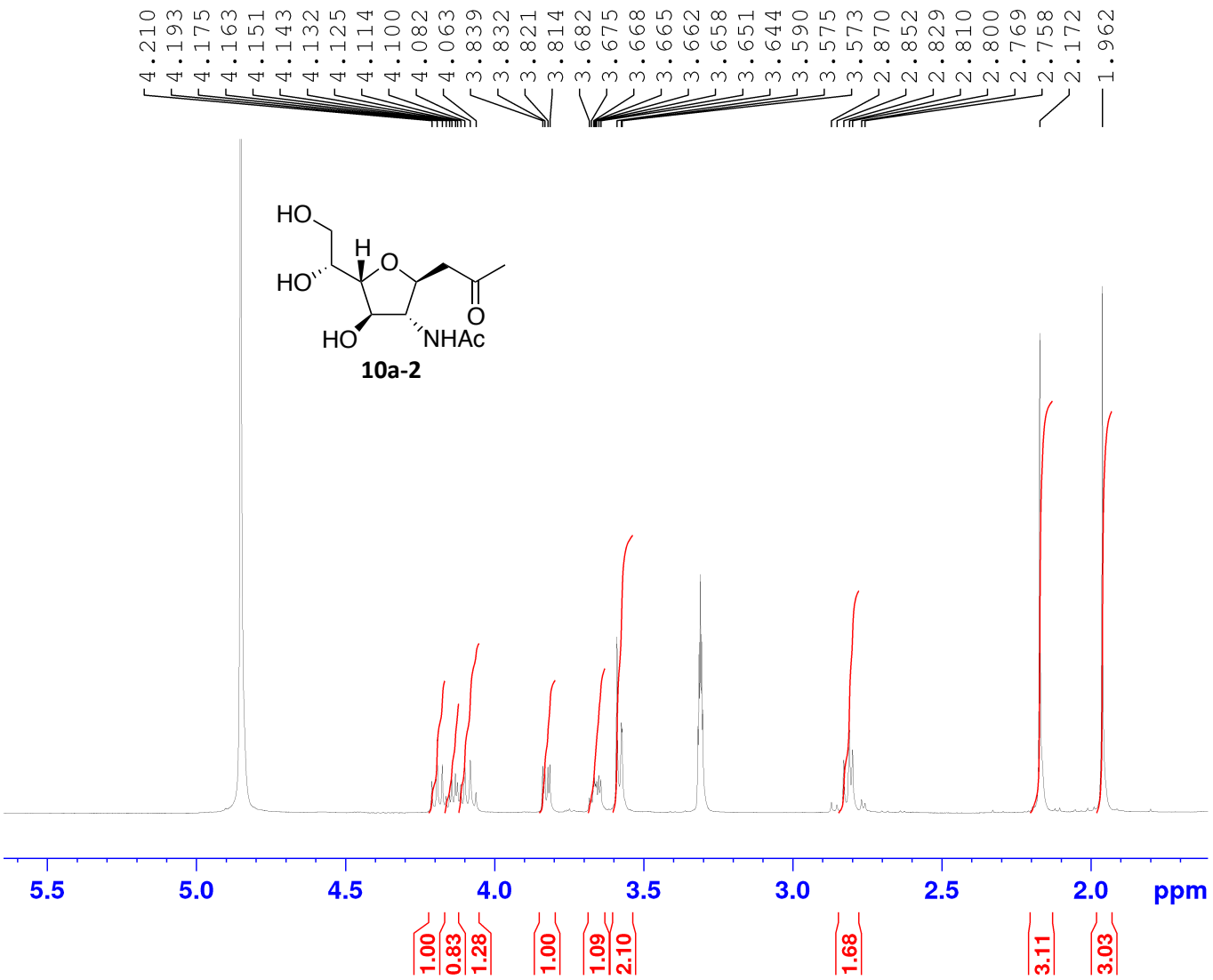



```
Current Data Parameters
NAME           SLJXII903
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150104
Time               17.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 71.01
DW                62.400 usec
DE                 6.50 usec
TE                298.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

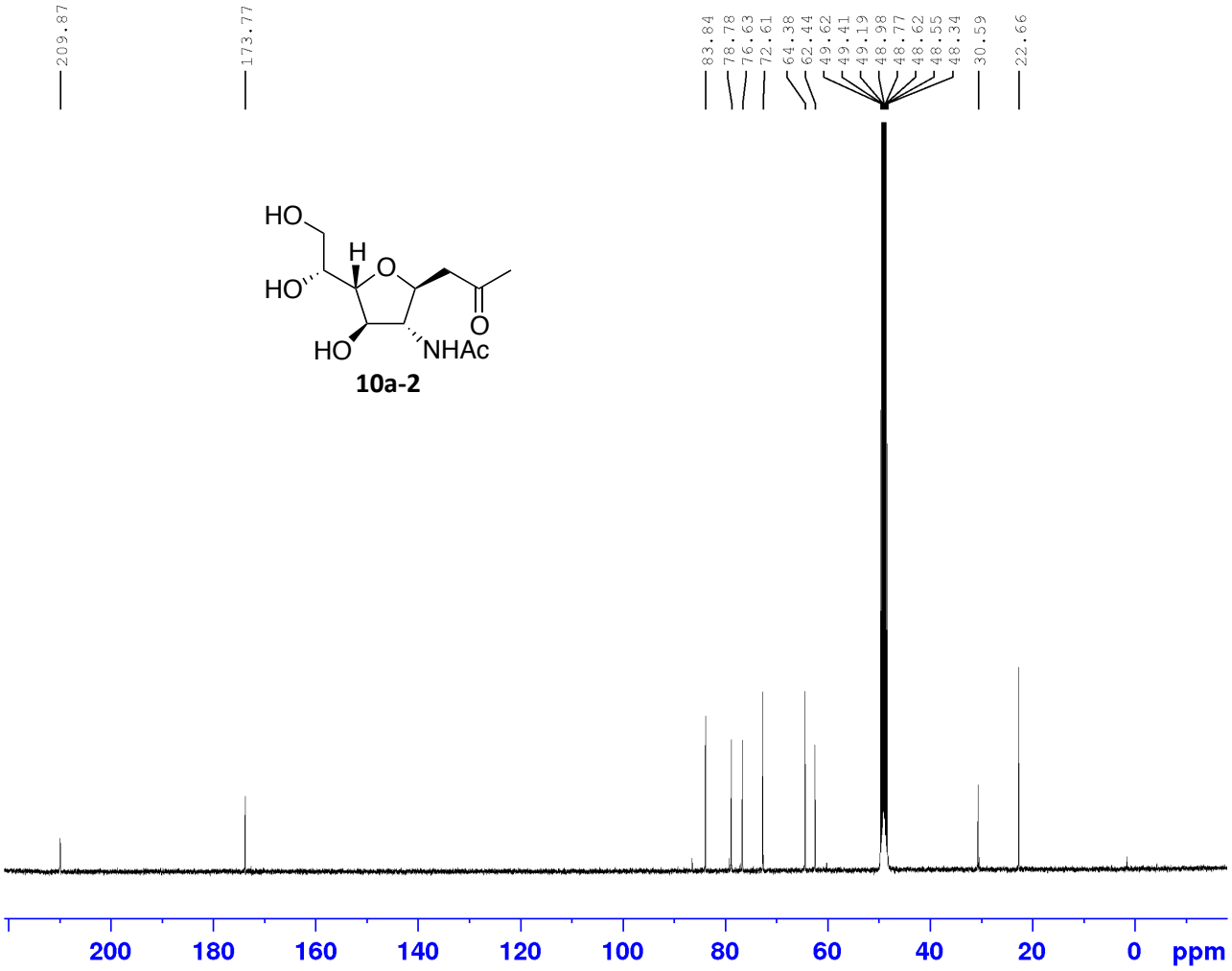



```
Current Data Parameters
NAME           SLJXII903
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150104
Time               17.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 71.01
DW                62.400 usec
DE                 6.50 usec
TE                298.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300077 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



10a-2

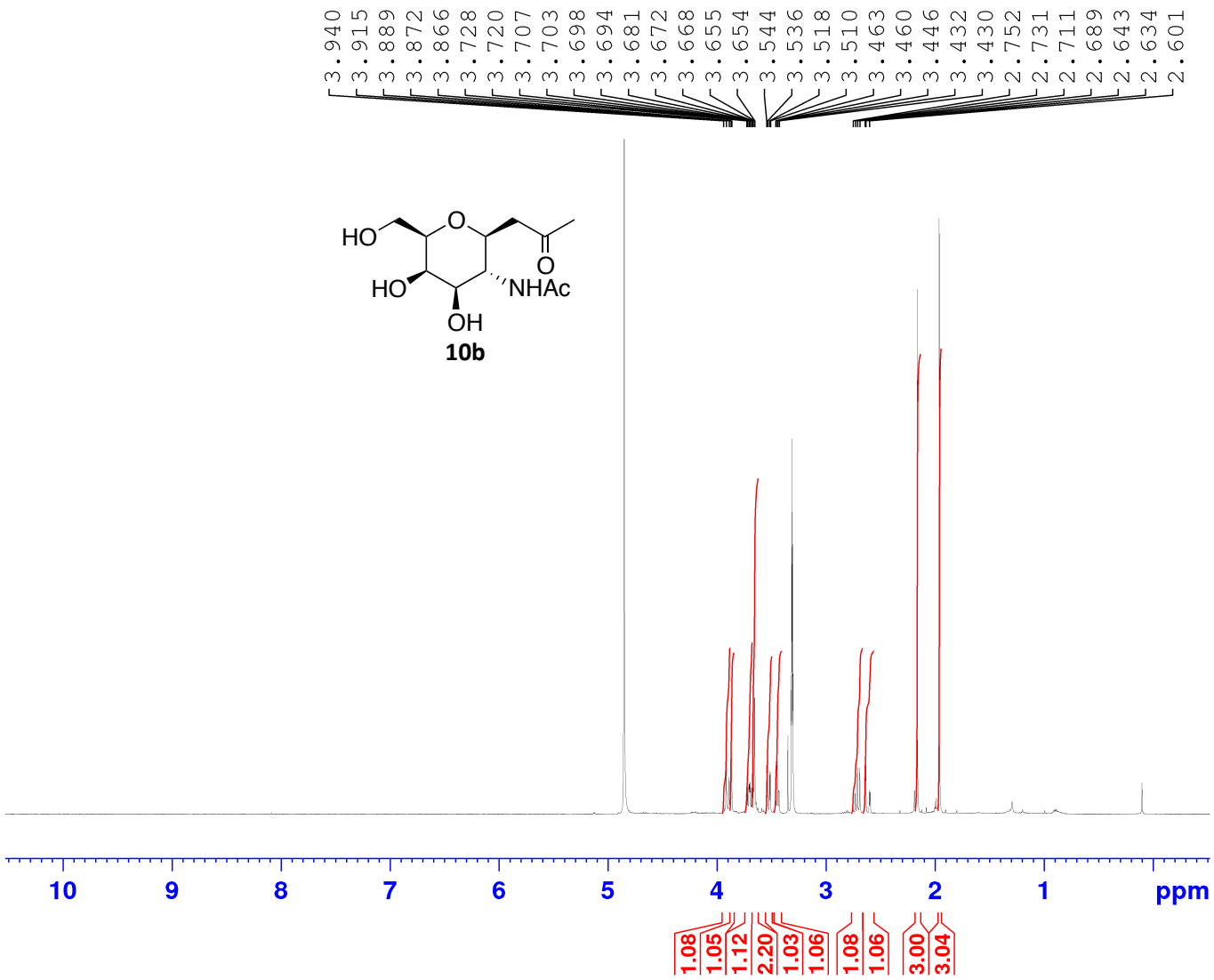
```
Current Data Parameters
NAME           SLJXII903
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150106
Time                8.11
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                14336
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               299.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6126294 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

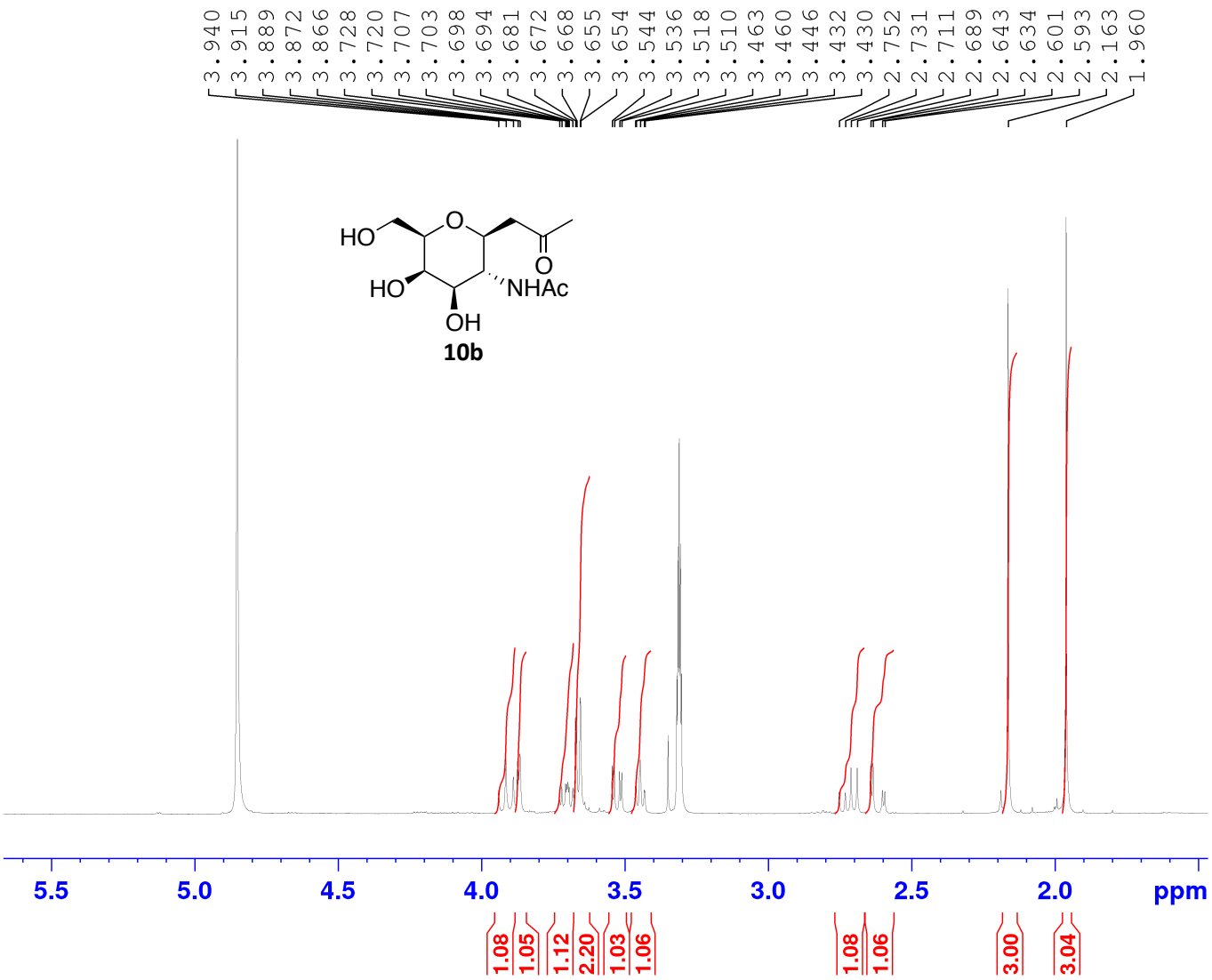



```
Current Data Parameters
NAME          SLJXIII991
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150223
Time               16.21
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 77.81
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

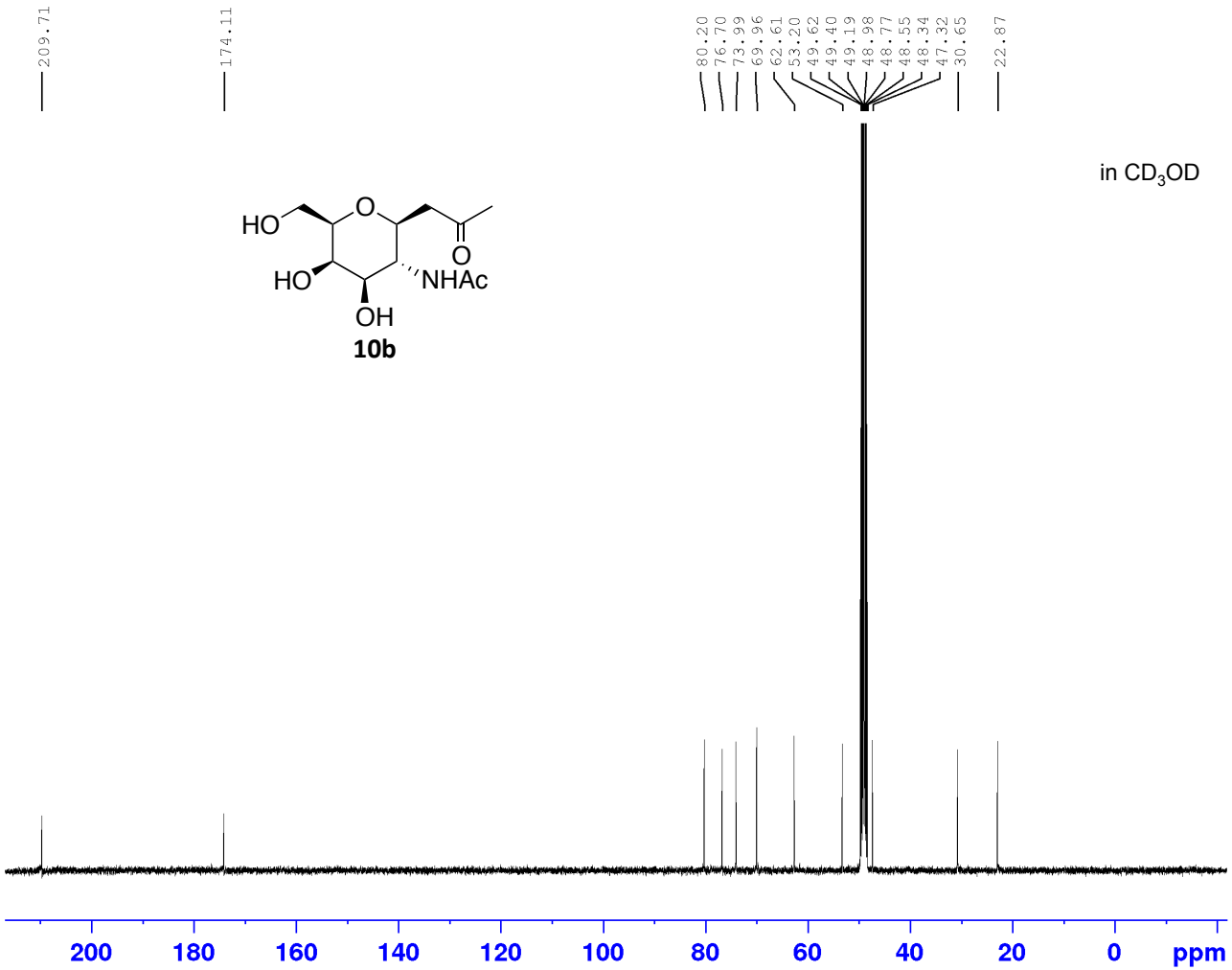



```
Current Data Parameters
NAME          SLJXIII991
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150223
Time               16.21
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 77.81
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



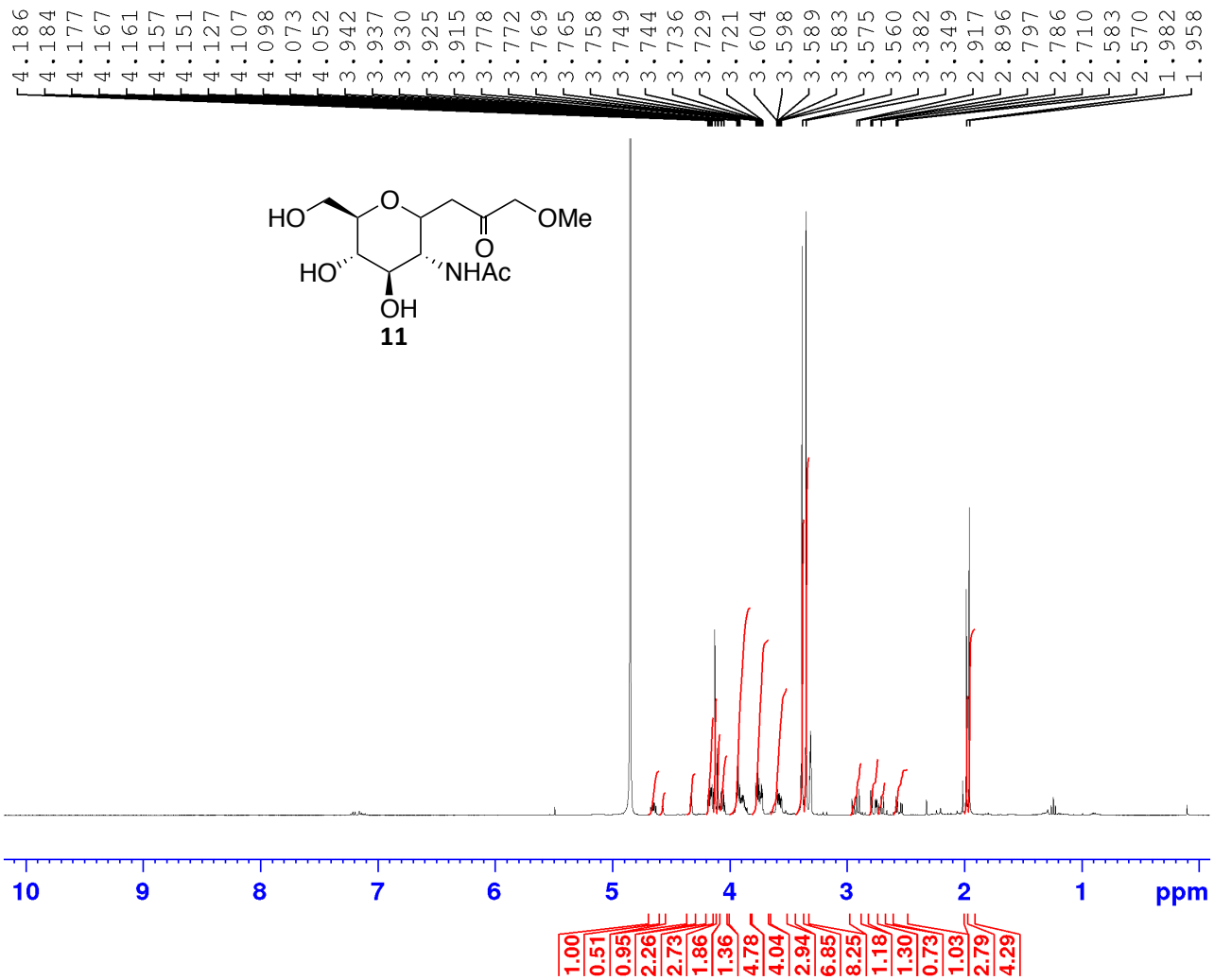

```
Current Data Parameters
NAME                   991
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150224
Time                 8.13
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10000
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                 195.88
DW                 20.800 usec
DE                  6.50 usec
TE                 299.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                  32768
SF           100.6130256 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

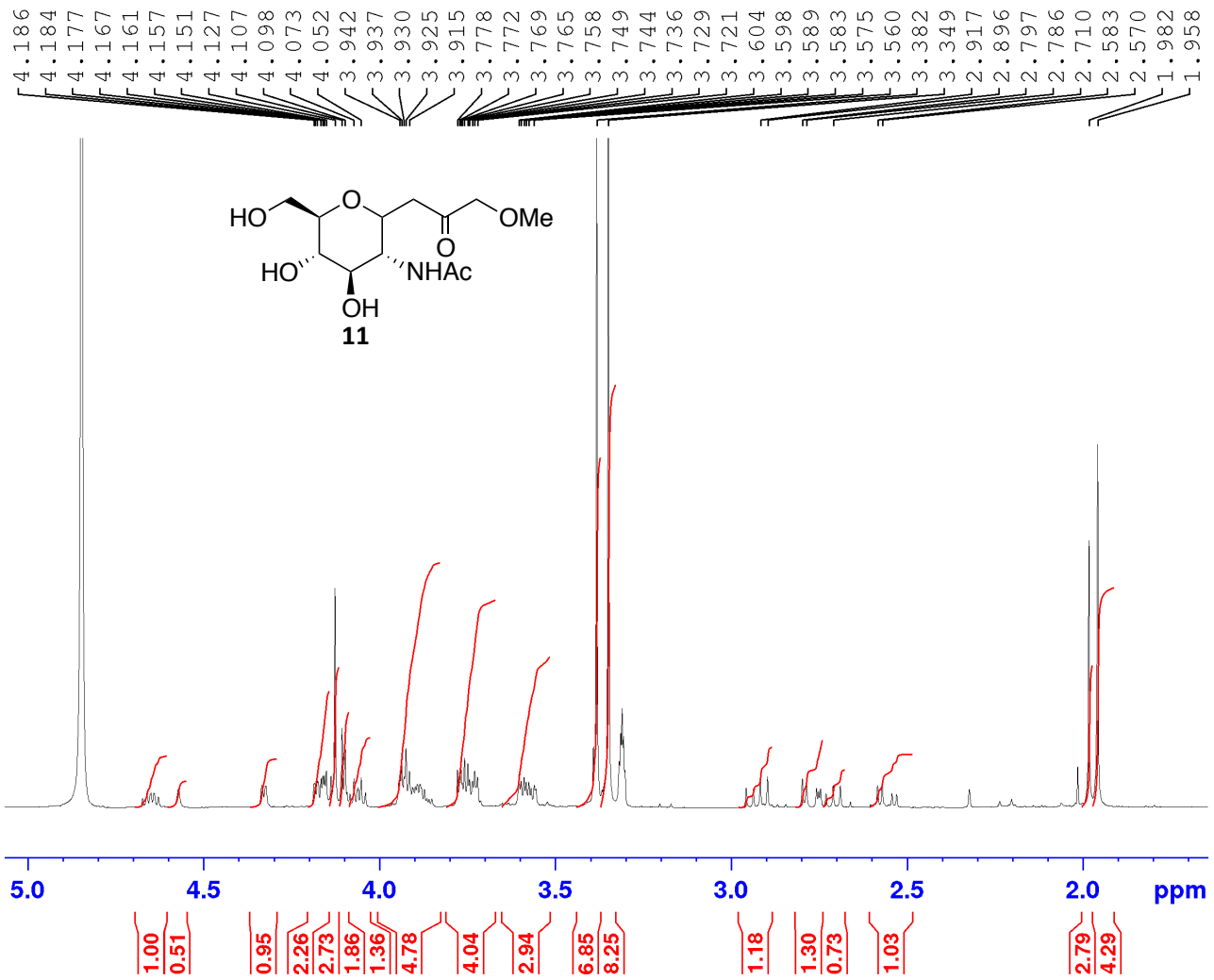



```
Current Data Parameters
NAME          SLJXIII953
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150715
Time               15.43
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                 31.13
DW                62.400 usec
DE                 6.50 usec
TE                298.6 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300074 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

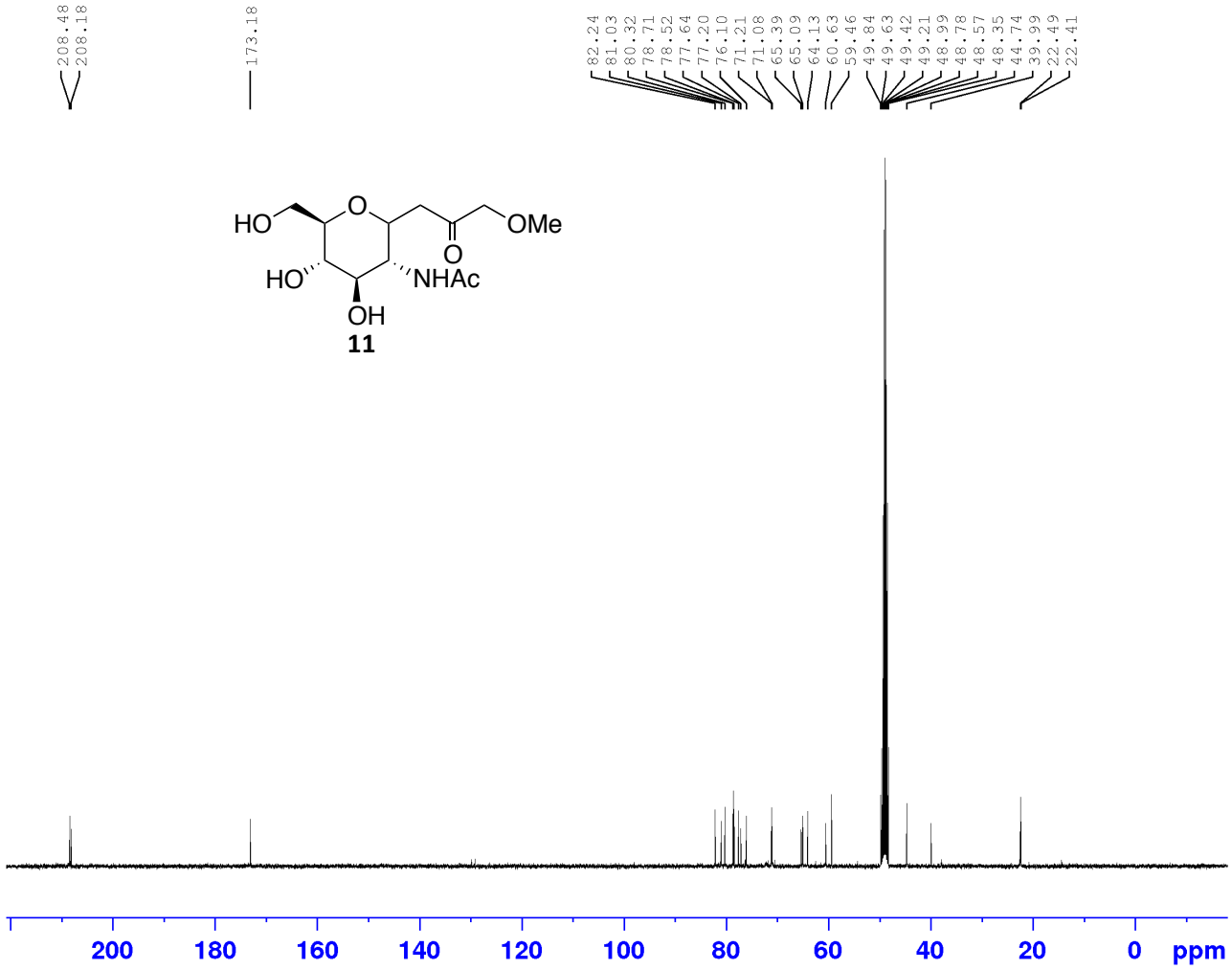



```
Current Data Parameters
NAME          SLJXIII953
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150715
Time               15.43
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
DW                62.400 usec
DE                 6.50 usec
TE                298.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300074 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



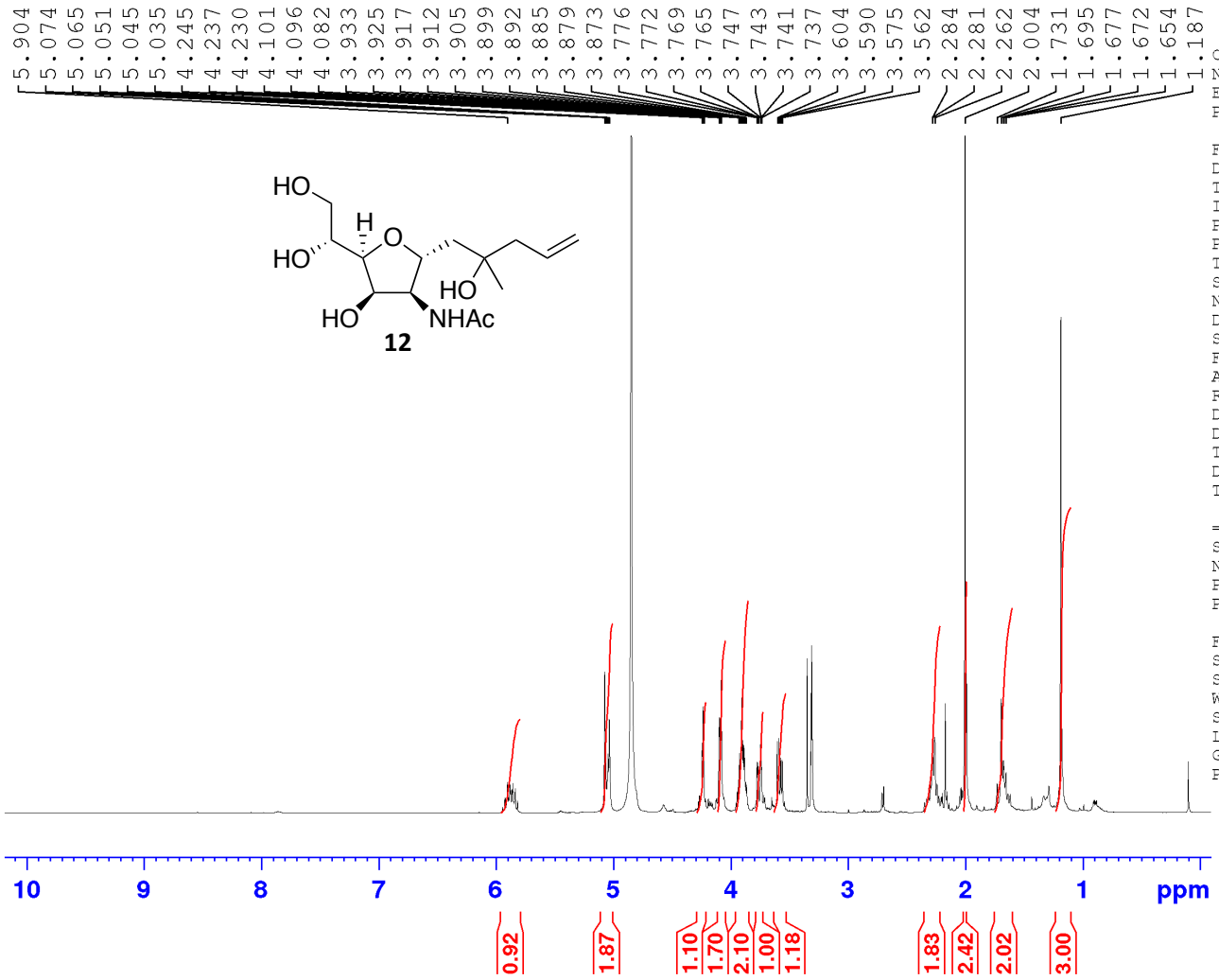

```
Current Data Parameters
NAME          SLJXIII953
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150715
Time               16.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT            MeOD
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE                299.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6126292 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

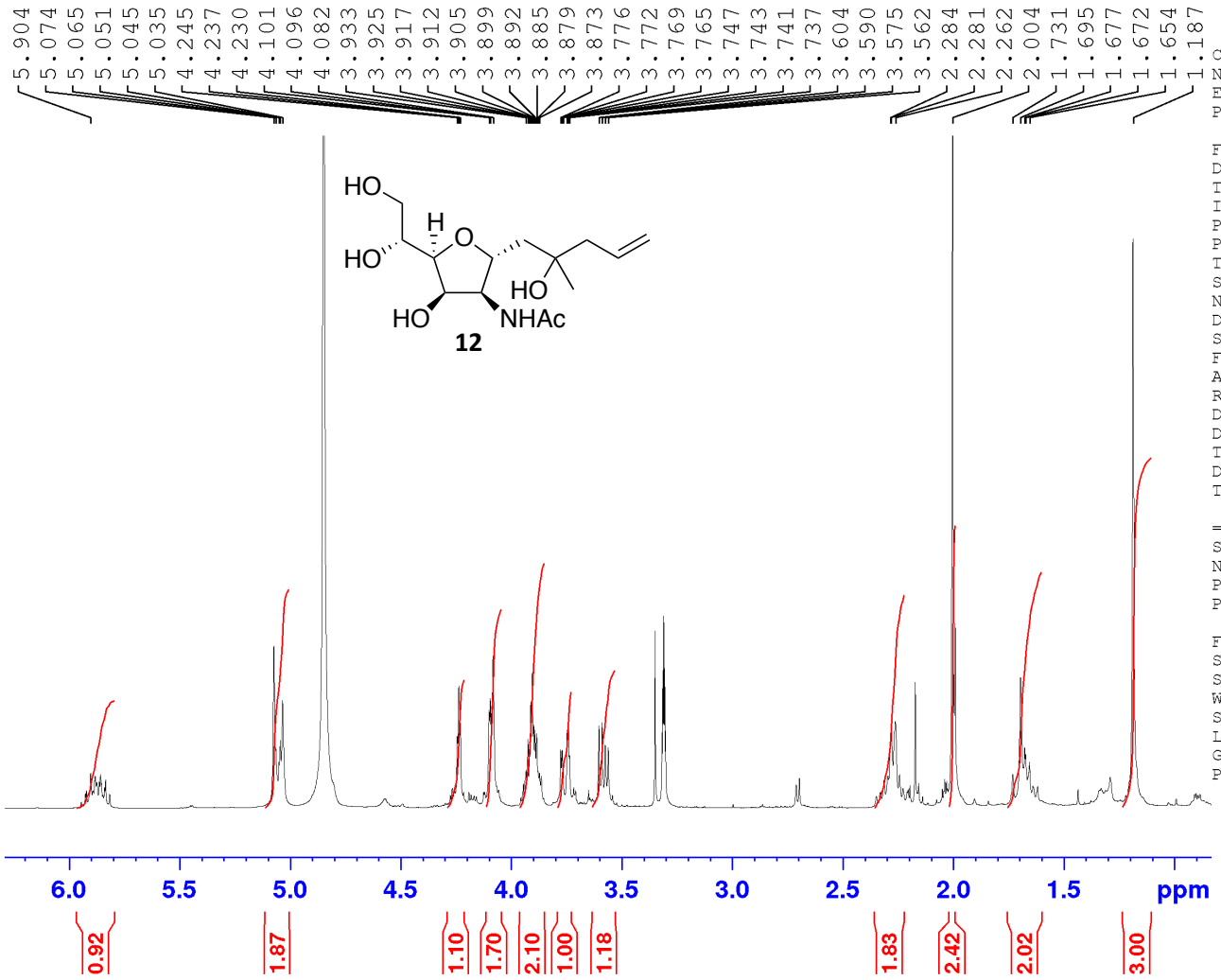



```
Current Data Parameters
NAME            SLJXII942
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150126
Time                16.31
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     32
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  31.13
DW                 62.400 usec
DE                  6.50 usec
TE                 298.5 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                  65536
SF           400.1300077 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

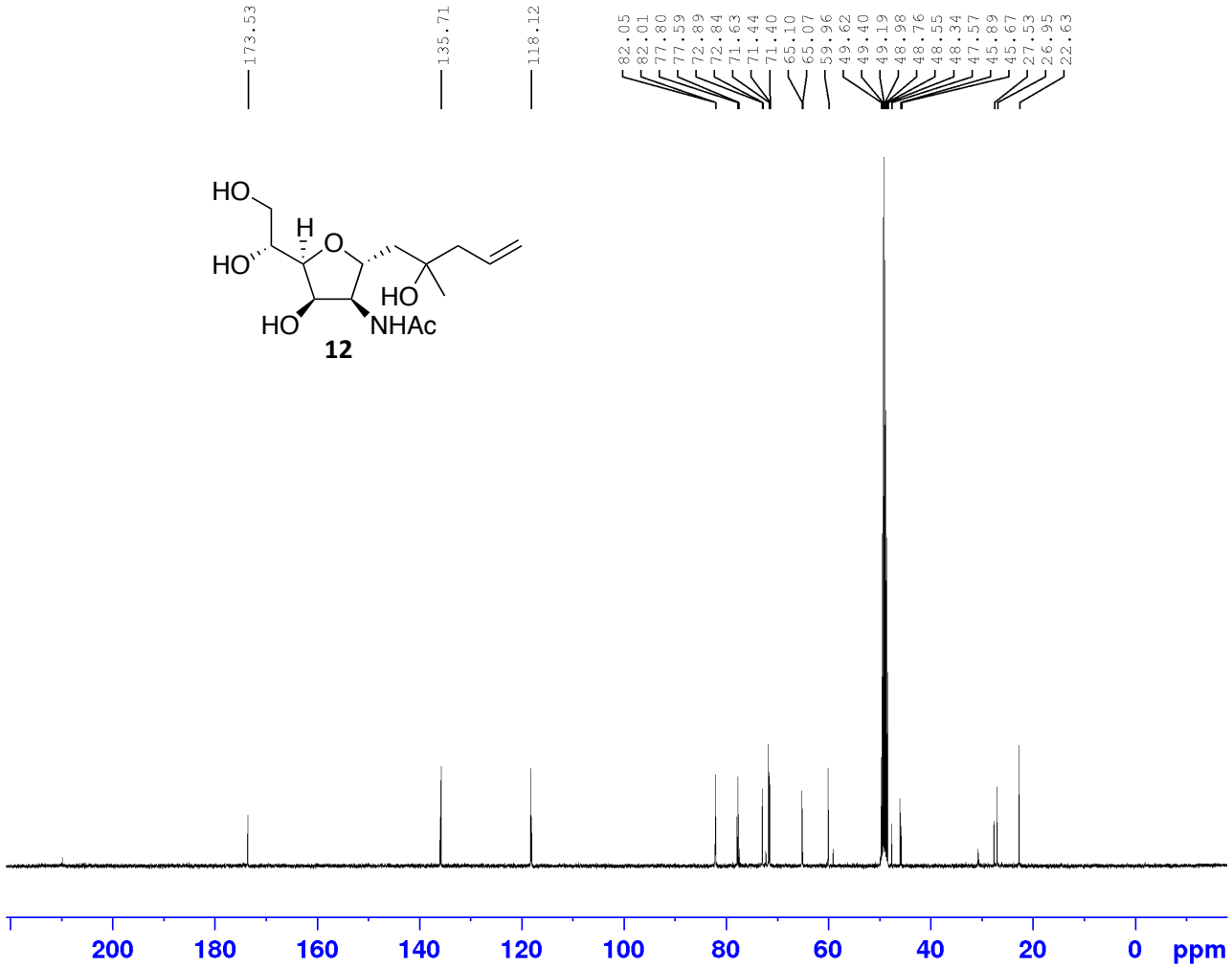



```
Current Data Parameters
NAME            SLJXII942
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150126
Time                16.31
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT              MeOD
NS                     32
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  31.13
DW                62.400 usec
DE                 6.50 usec
TE                 298.5 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                  65536
SF            400.1300077 MHz
WDW                    EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



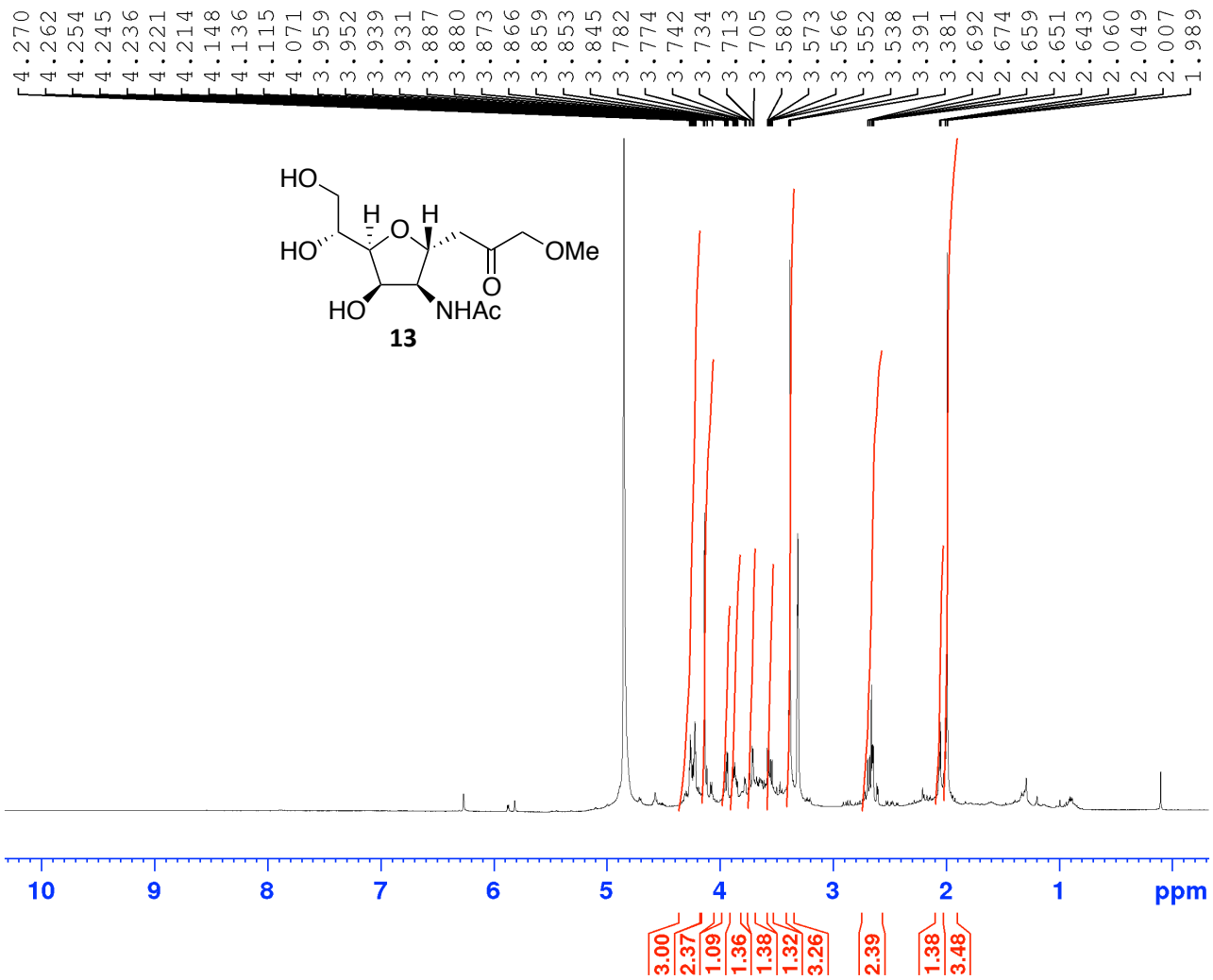

```
Current Data Parameters
NAME            SLJXII942
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150128
Time                14.21
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             MeOD
NS                  2048
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               299.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF           100.6126314 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

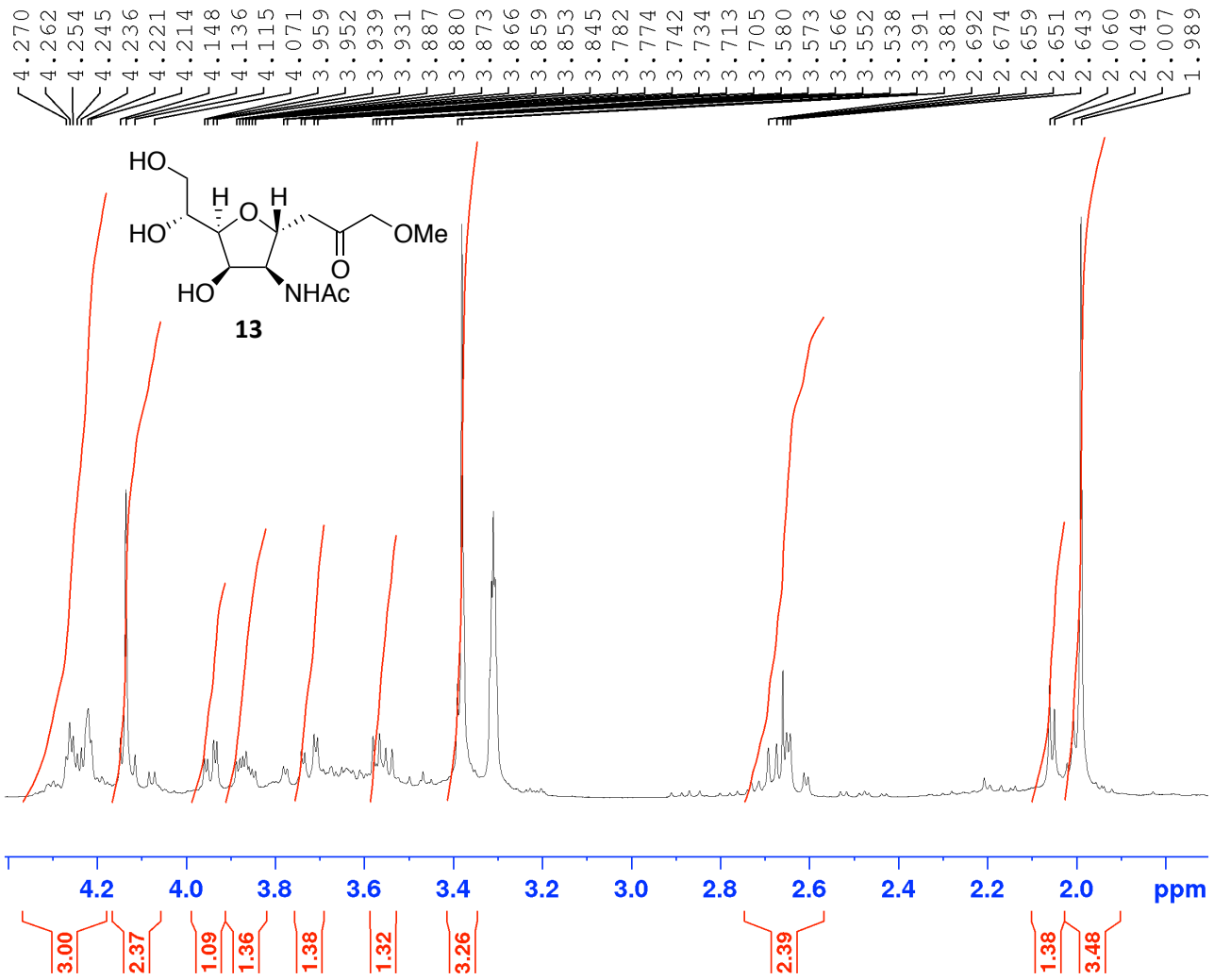



```
Current Data Parameters
NAME           SLJvii493
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140718
Time               14.53
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            MeOD
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES            0.122266 Hz
AQ             4.0894465 sec
RG                 49.09
DW                62.400 usec
DE                 6.50 usec
TE                298.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                    65536
SF            400.1300077 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

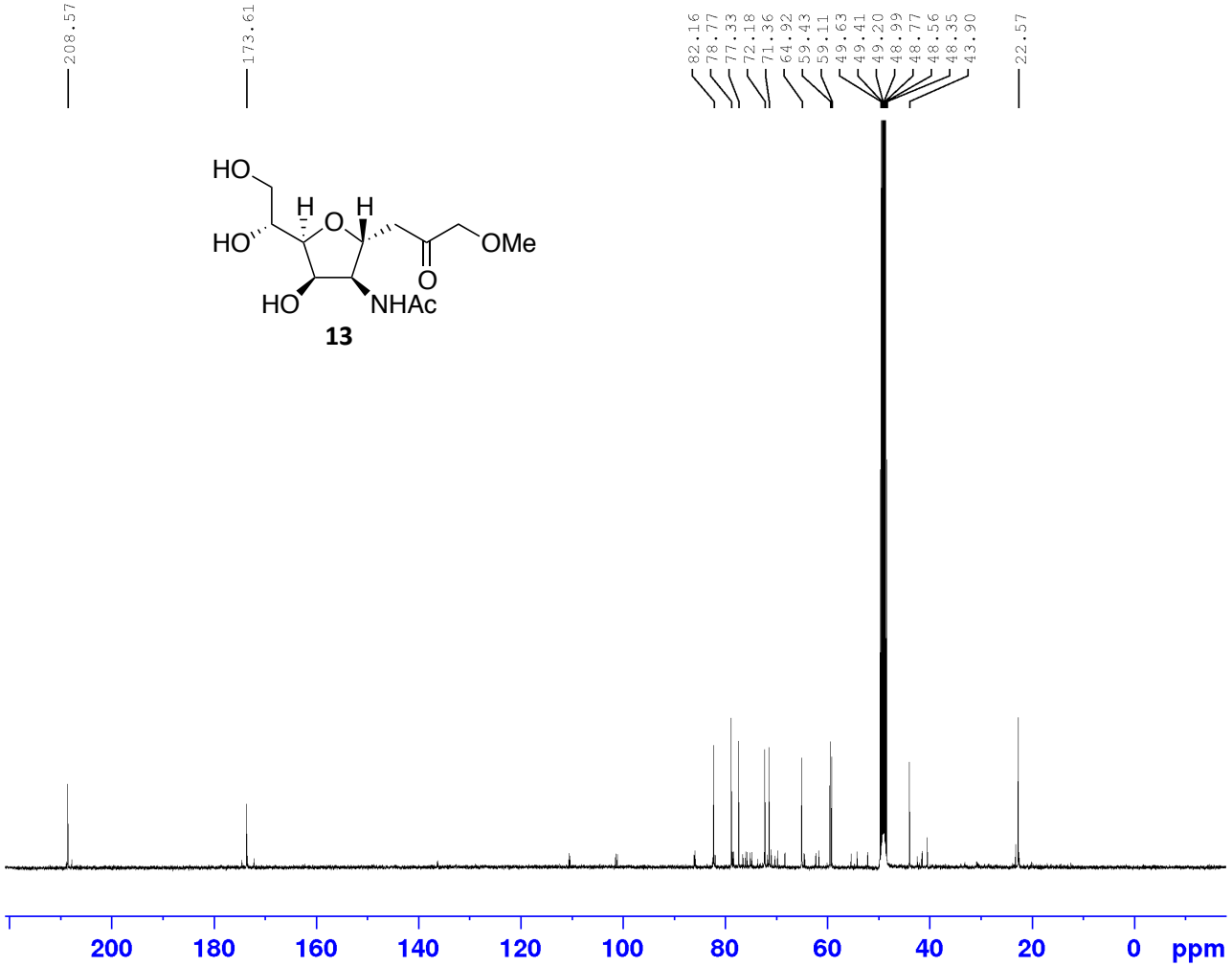



```
Current Data Parameters
NAME           SLJvii493
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140718
Time               14.53
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT            MeOD
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                 49.09
DW                62.400 usec
DE                 6.50 usec
TE                298.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                15.00 usec
PLW1          8.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300077 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



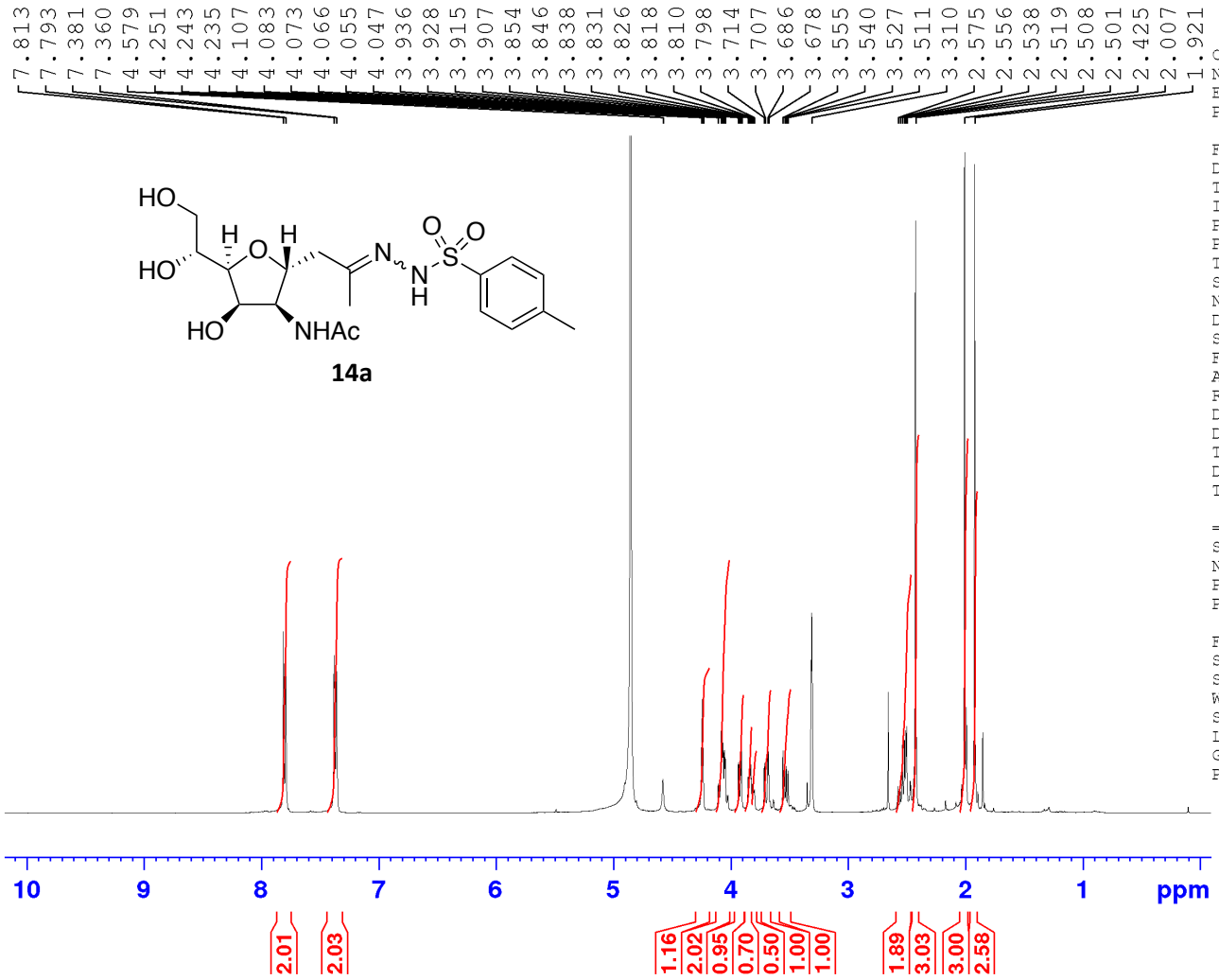

```
Current Data Parameters
NAME           SLJvii493
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140719
Time                8.51
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                16384
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               299.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          8.00000000 W
PLW12         0.28125000 W
PLW13         0.28125000 W

F2 - Processing parameters
SI                 32768
SF          100.6126290 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

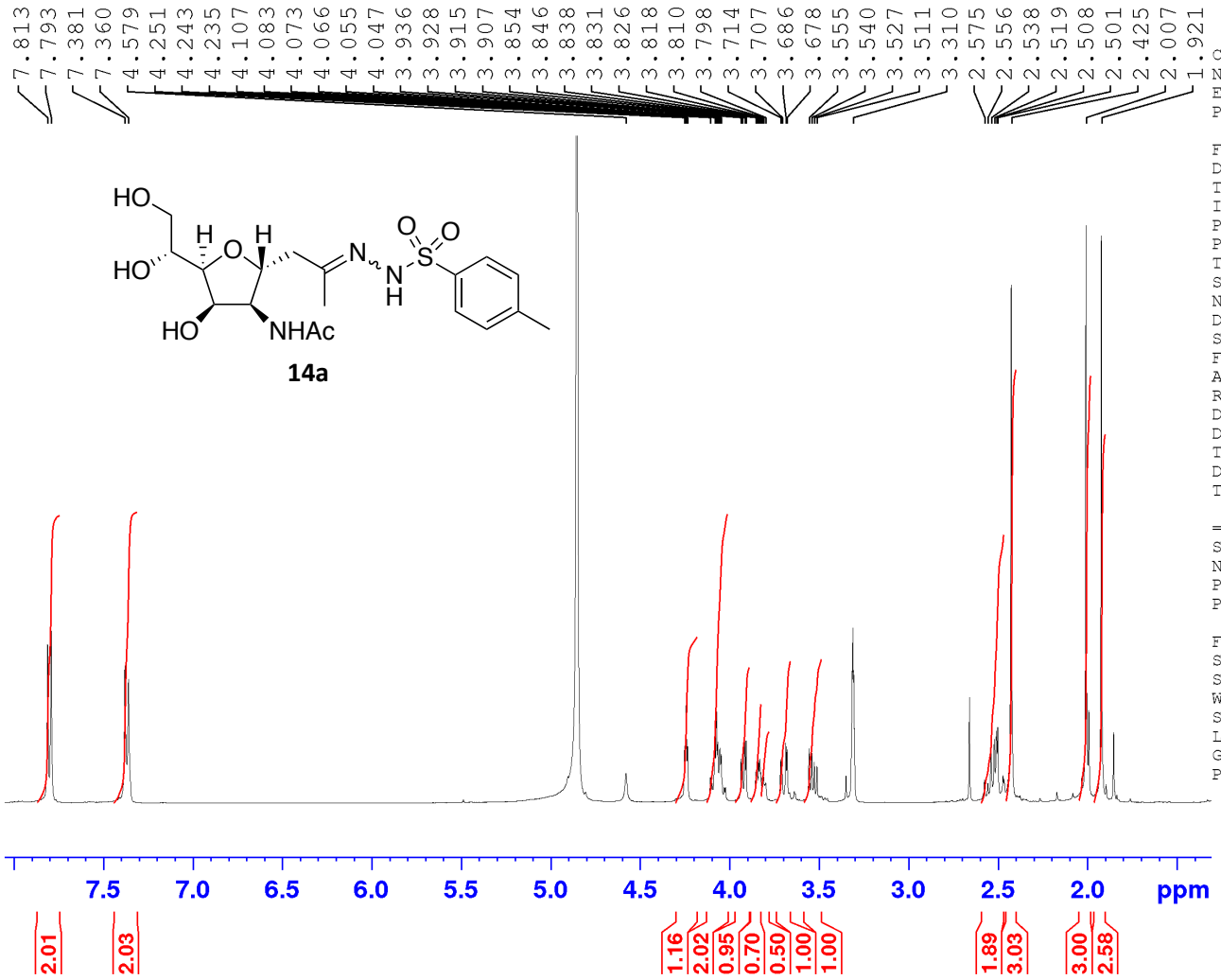



```
Current Data Parameters
NAME          SLJXIII1021
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150430
Time               14.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

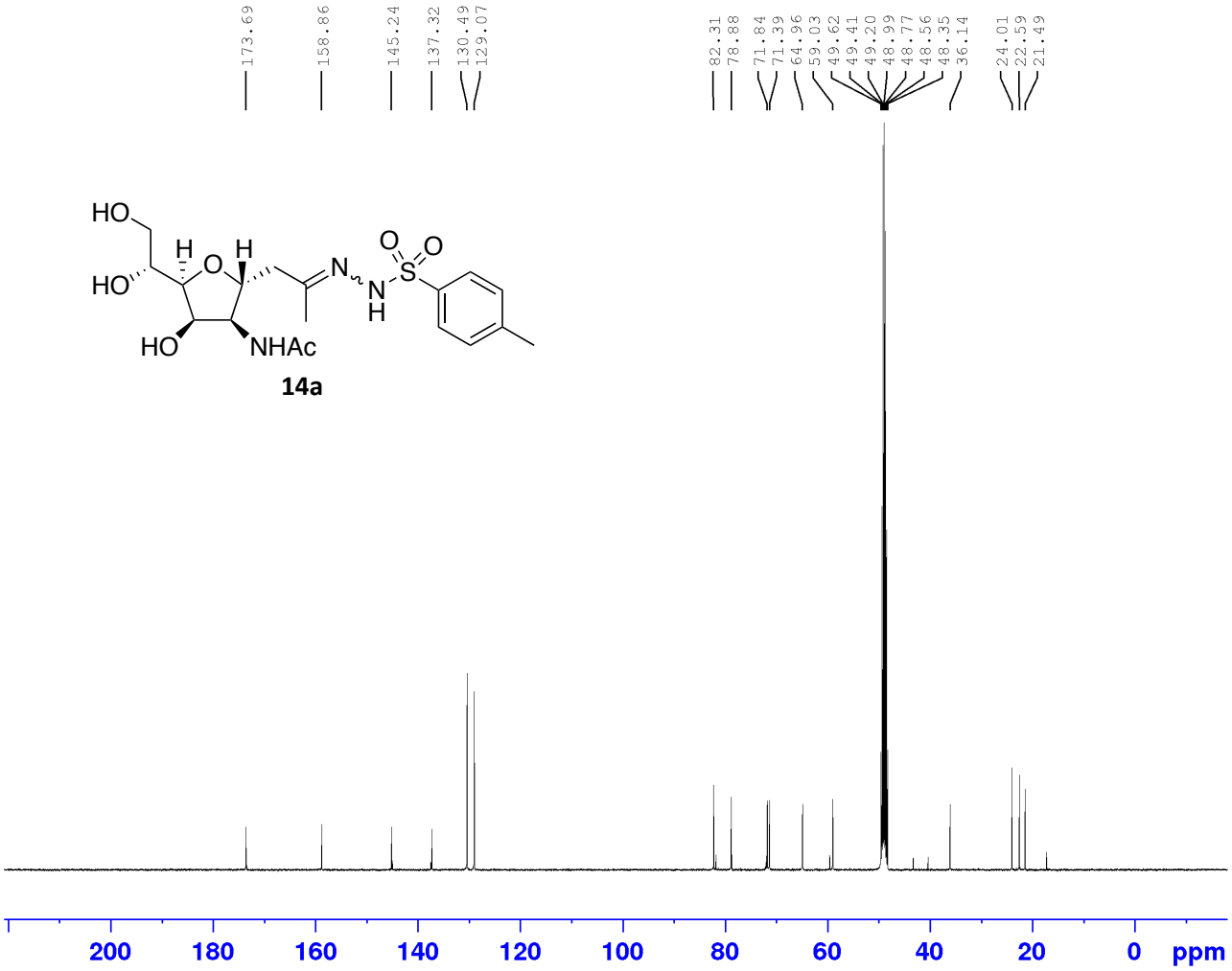



```
Current Data Parameters
NAME          SLJXIII1021
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150430
Time               14.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 31.13
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300077 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



14a

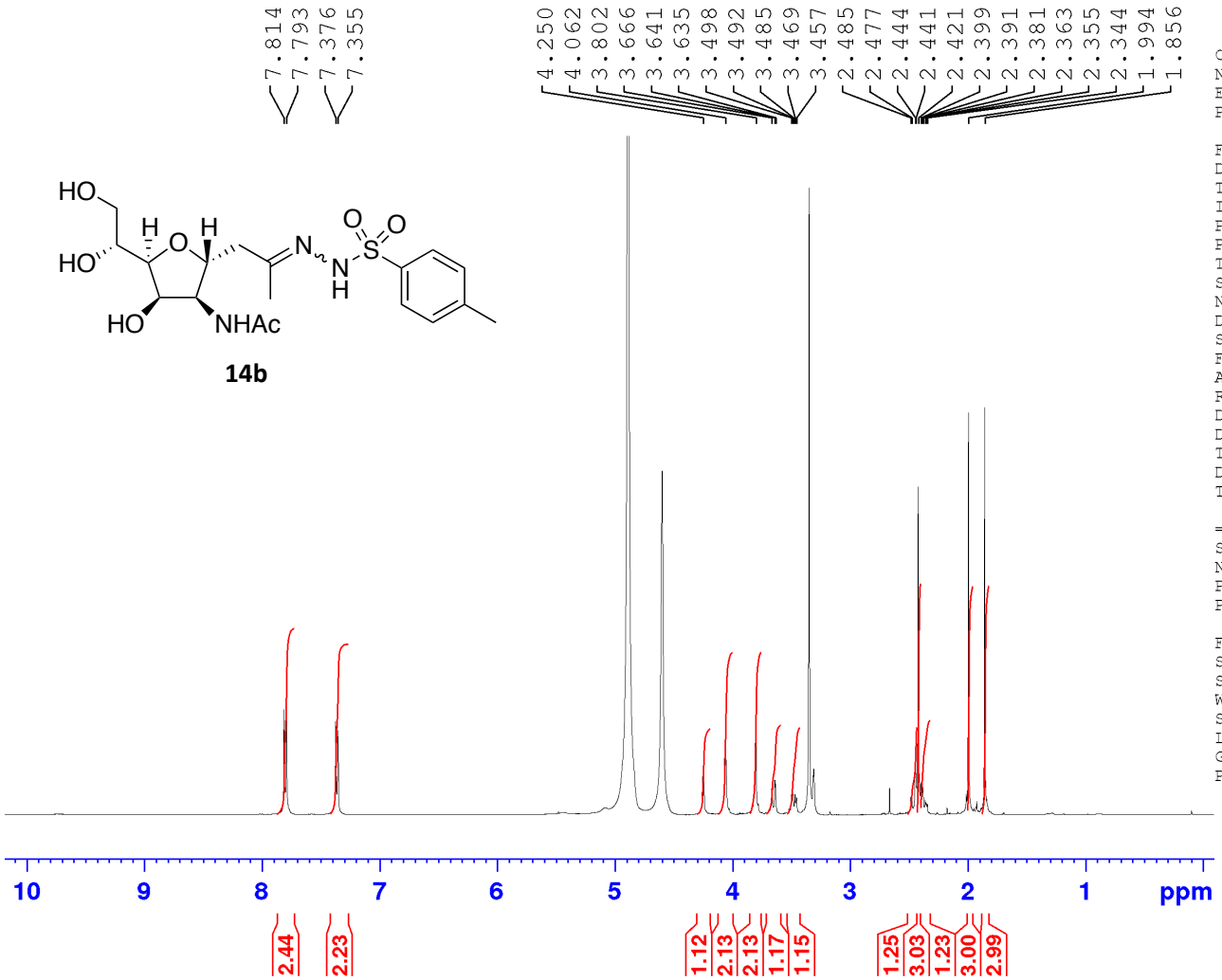
```
Current Data Parameters
NAME         SLJXIII1021
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150501
Time               6.24
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT           MeOD
NS               12288
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE               299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               10.00 usec
PLW1        70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2         8.00000000 W
PLW12        0.28125000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6126301 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

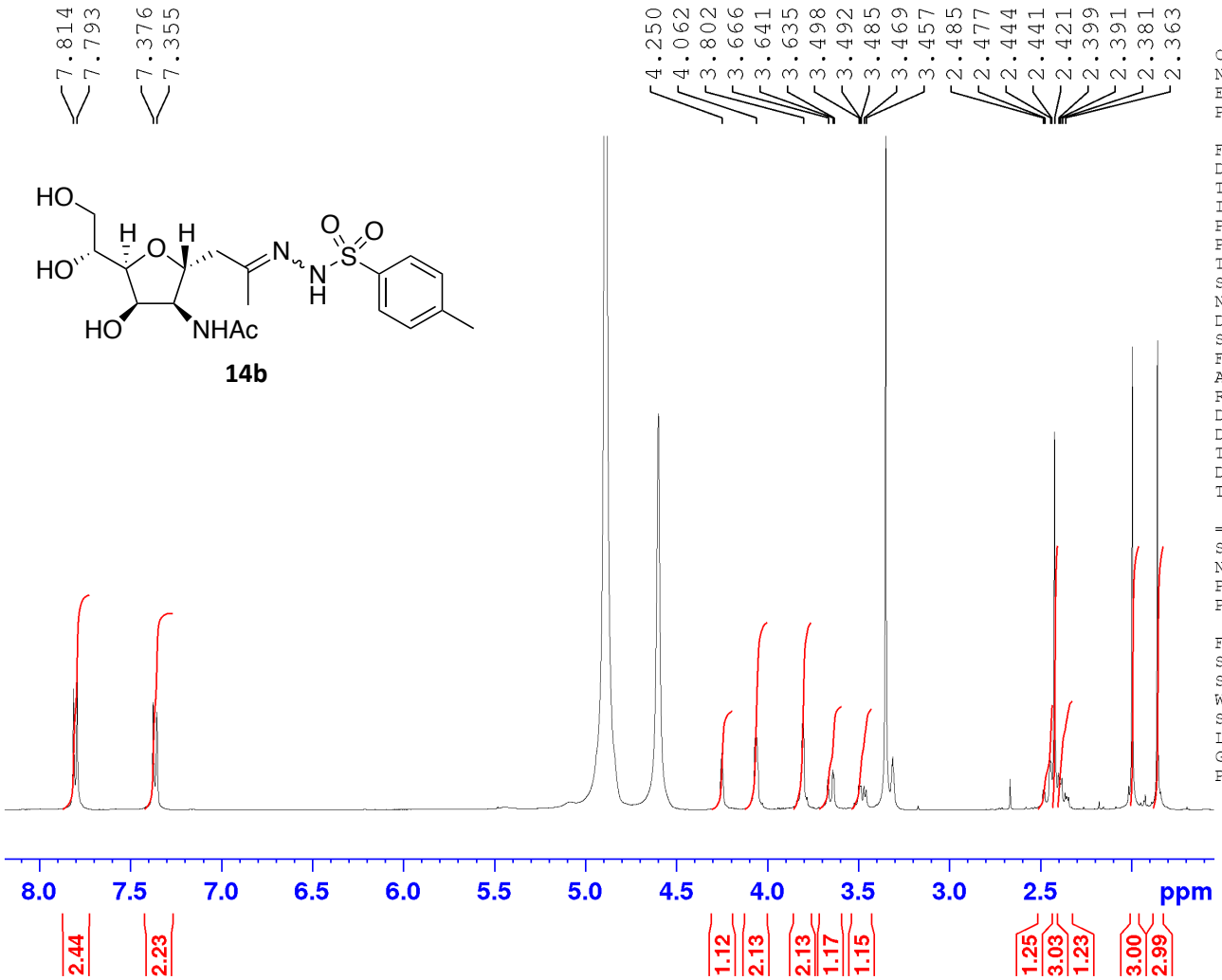



```
Current Data Parameters
NAME          SLJXIII1021
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150430
Time               14.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 17.32
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300075 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

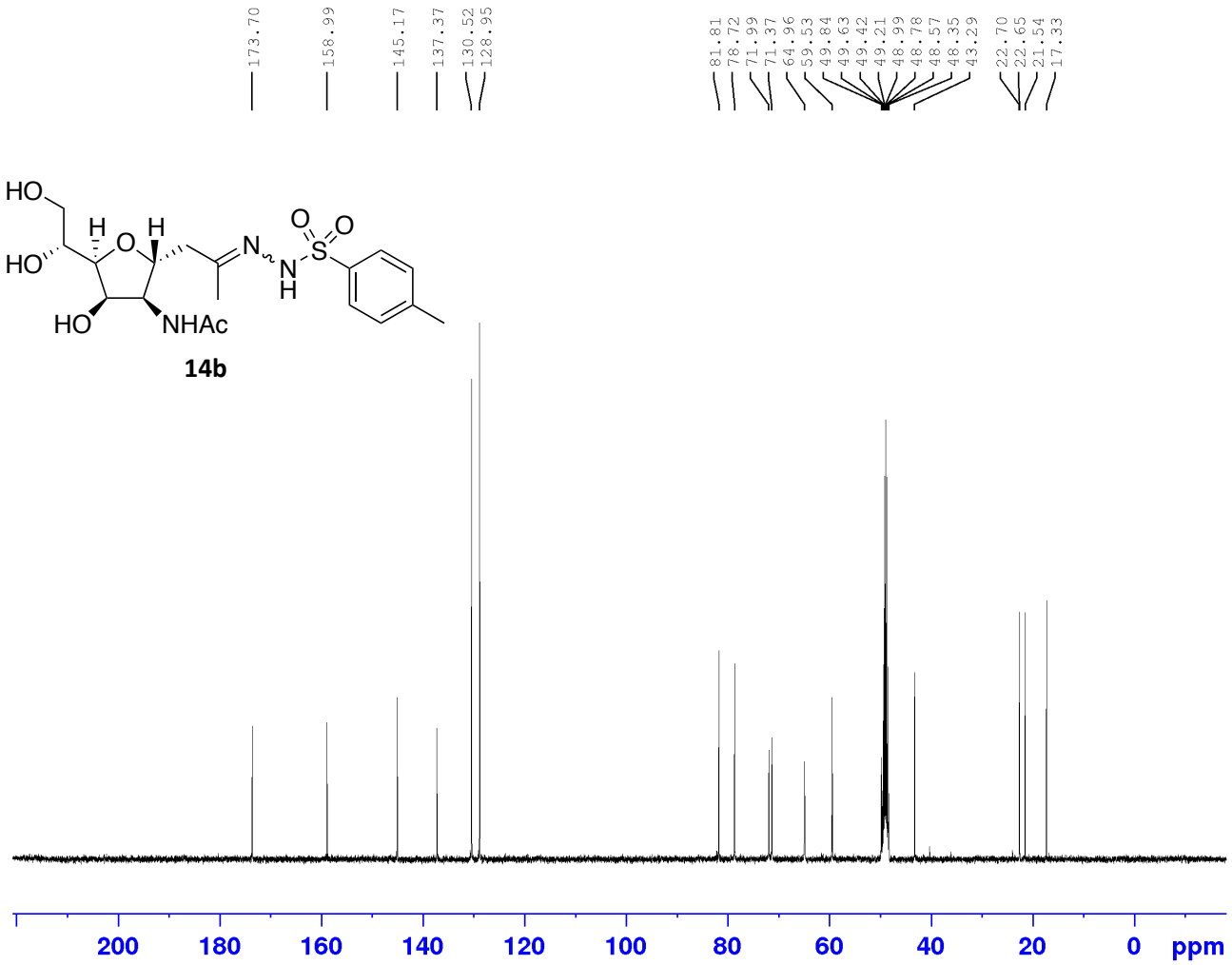



```
Current Data Parameters
NAME          SLJXIII1021
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150430
Time               14.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 17.32
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                15.00 usec
PLW1           8.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300075 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



14b

```
Current Data Parameters
NAME         SLJXIII1021
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150430
Time              18.23
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT            MeOD
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               195.88
DW               20.800 usec
DE                6.50 usec
TE                299.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1               10.00 usec
PLW1        70.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2         8.00000000 W
PLW12        0.28125000 W
PLW13        0.28125000 W

F2 - Processing parameters
SI                32768
SF          100.6126343 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```