Supplementary Information

A practical and scalable synthesis of carbohydrate based oxepines

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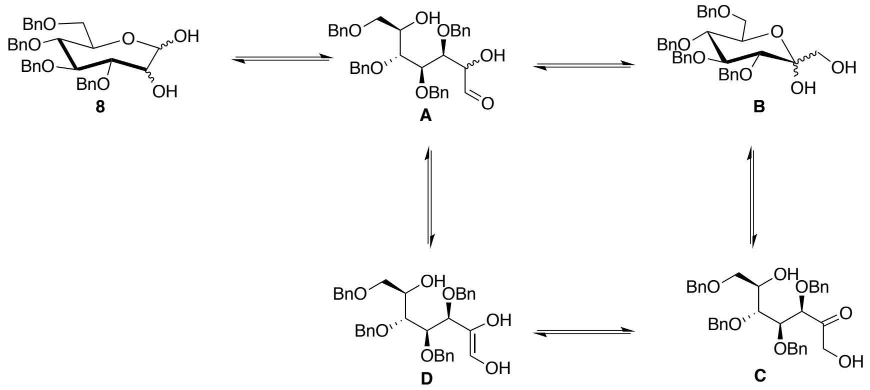
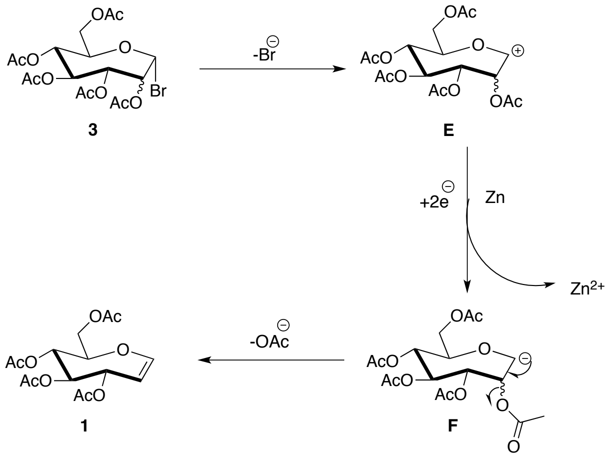


Scheme S1. Possible products during ozonolysis reactions.



Scheme S2. Mechanism for the reductive elimination using anomeric bromide **3** as an example starting material.

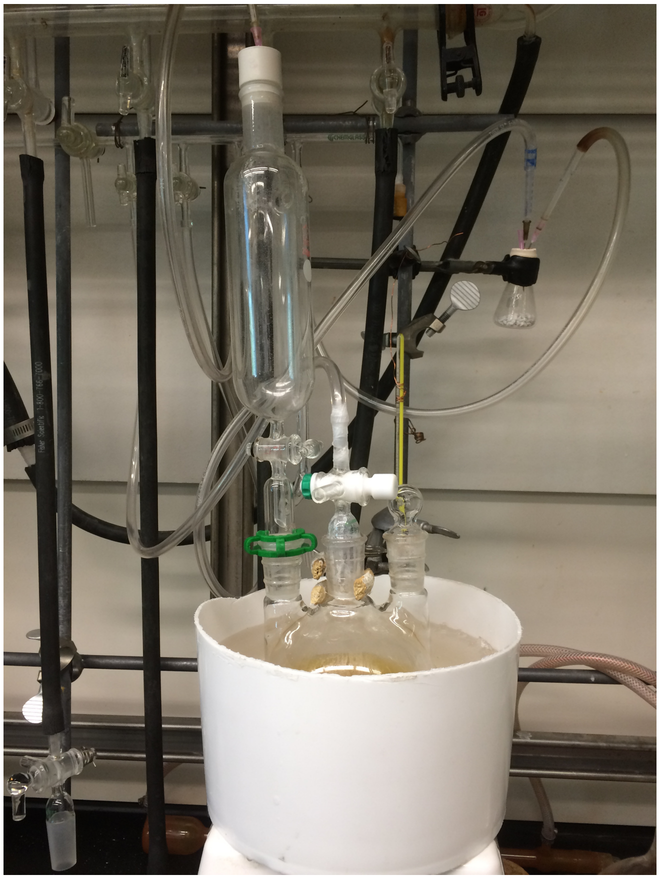


Figure S1. Equipment set up and appearance of vinylation reaction on 20 g scale of 2,3,4,6-tetra-*O*-benzyl D-glucopyranose.

- 35% weight of 10% Pd/C was used if the starting material is >1.00 g scale.

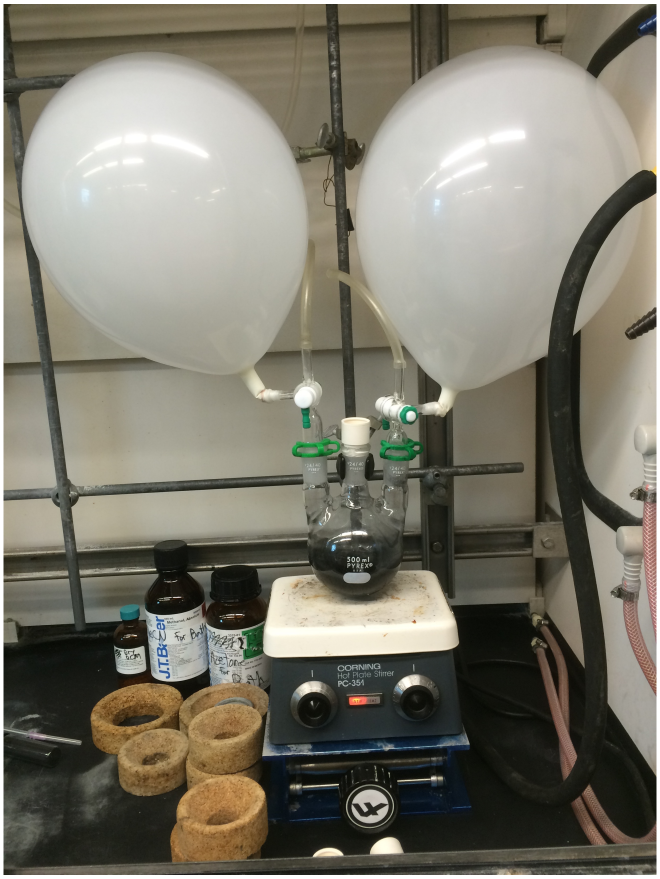
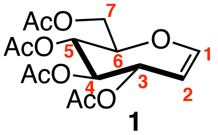
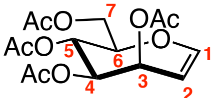
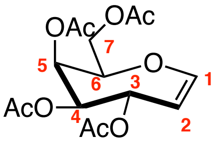
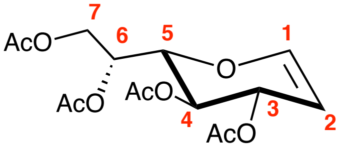


Figure S2. Equipment set up and appearance of debenzylation reaction on 10 g scale of tetra-benzyl-di-*O*-acetyl gluco septanose.

Table S1. Detailed Spectral data of the oxepines **1**, **19**, **25** and glycal **26**.

Compound	**Key Chemical shifts (δ ppm)**		**Key Observations**
	protons	**carbons**	
 **1**	H1-6.49 (d, *J* = 6.9 Hz, 1H)) H2-4.85-4.81 (dd, *J* = 6.9 Hz, 1H)) H3-5.39-5.36 (dd, *J* = 5.9, 5.9 Hz, 1H) H4-5.24-5.18 (m, 2H) H5-5.24-5.18 (m, 2H) H6-4.58-4.55 (m, 1H) H7a-4.34-4.30 (dd, *J* = 12.2, 5.1 Hz, 1H) H7b-4.20-4.17 (dd, *J* = 12.2, 1.8 Hz, 1H)	C1-150.3 C2-103.2 C3-67.5 C4-73.8 C5-71.5 C6-79.0 C7-63.4	(HMBC) H1-C2, C3, C6 H3, H4, H5, H7- C=O
 **19**	H1-6.41-6.38 (dd, *J* = 6.8, 1.5 Hz, 1H) H2-4.67-4.64 (ddd, *J*= 6.8, 3.4, 1.0 Hz, 1H) H3-5.77-5.75 (m, 1H) H4-5.23-5.22 (m, 1H) H5-5.12-5.09 (dd, *J* = 8.9, 4.1 Hz, 1H) H6-4.19-4.10 (m, 2H) H7a-4.19-4.10 (m, 2H) H7b-4.27-4.23 (dd, *J* = 11.7, 5.9 Hz, 1H)	C1-148.4 C2-105.4 C3-68.4 C4-73.3 C5-70.4 C6-80.5 C7-63.2	(HMBC) H1-C2, C3, C6 H3, H4, H5, H7- C=O
 **25**	H1-6.36-6.34 (dd, *J* = 7.6, 2.3 Hz, 1H) H2-4.70-4.68 (dd, *J* = 7.6, 3.0 Hz, 1H) H3-5.73-5.70 (ddd, *J* = 2.7, 2.7, 9.2 Hz, 1H) H4-5.09-5.07 (dd, *J* = 9.3, 2.8 Hz, 1H) H5-5.49 (d, *J* = 2.7, 2.7 Hz, 1H) H6-4.19-4.15 (m, 2H) H7a-4.19- 4.15 (m, 2H) H7b-4.07- 4.03 (m, 1H)	C1-145.9 C2-106.9 C3-68.4 C4-73.1 C5-68.5 C6-76.8 C7-63.1	(HMBC) H1-C2, C3, C6 H3, H4, H5, H7- C=O
 **26**	H1-6.45 (dd, *J* = 6.0, 1.2 Hz, 1H) H2-4.82 (dd, *J* = 6.0, 2.5 Hz, 1H) H3-5.53-5.50 (ddd, *J* = 7.2, 1.8, 1.8, Hz, 1H) H4-5.30-5.26 (dd, *J* = 9.8, 7.2 Hz, 1H) H5-4.25-4.19 (m, 2H) H6-5.41 (m, 1H) H7a-4.25-4.19 (m, 2H), H7b-4.40-4.36 (dd, *J* = 11.8, 4.8 Hz, 1H)	C1-145.8 C2-100.2 C3-69.5 C4-66.3 C5-74.9 C6-66.7 C7-62.5	(HMBC) H1-C2, C3, C5 H3, H4, H6, H7- C=O

General Procedures

All reactions were performed under nitrogen (N_2) atmosphere unless otherwise mentioned. All commercially available reagents and solvents were used as received from the manufacturer. Reactions were monitored by TLC (silica gel HL, w/UV254, 250μm) and visualized either under UV lamp or by charring with 2.5% *p*-anisaldehyde in H_2SO_4, AcOH and EtOH solution. Flash chromatography was performed on silica gel (60 Å, 40-63 μm). Optical rotation was measured at 22±2 °C; ^{1}HNMR spectra were collected at 400 MHz or 500 MHz with chemical shift referenced to $(CH_3)_4Si$ (δ_H 0.00 ppm) or the residual peak in $CDCl_3$ (δ_H 7.24 ppm). All the ^{13}C NMR spectra were collected at 100 MHz and referenced to residual peak in $CDCl_3$ (δ_C 77.23 ppm).

General procedure for alkylation and hydrolysis (*ChemSpider SyntheticPages*, 2015, http://cssp.chemspider.com/784)

Benzylation: To a three-neck RBF equipped with an additional funnel, mechanical stirrer, and a stopper was added methyl D-pyranoside (20 g, 0.103 mol, 1 eq.) and dioxane (5 mL per 1.00 g starting material). The mixture was then heated to 80 °C for 0.5 h with vigorous stirring. Then, finely powdered KOH (99.3 g, 1.17 mol, 17.2 eq.) was added portion-wise to the flask (coffee grinder was used to make KOH pellets into very fine powder). After stirring at 1 h at 80 °C, benzyl chloride (118.6 mL, 1.03 mol, 10.2 eq.) was added to the flask drop-wise by using the additional funnel. Reaction progress was monitored by TLC analysis. Upon disappearance of starting material (R_f 0.0, 7:3 hexane:ethyl acetate) usually (~12 h), water (10 mL per 1.00 g of starting material) was added to the resulting reaction mixture. The combined solutions were extracted with ethyl acetate (3 x 10 mL per 1.00 g of starting material), the combined organic layers were washed with brine (10 mL per 1.00 g of starting material), dried with Na_2SO_4 and solvents were removed under reduced pressure. The resulting residue was purified by column chromatography using hexane: ethyl acetate solvent system, initially with 10% ethyl acetate in hexanes to remove the excess of BnCl, then with 30% of ethyl acetate to liberate the methyl-2,3,4,6-tetra-*O*-benzyl D-pyranoside (75 - 85%).

Hydrolysis: A three-neck round bottom flask equipped with a condenser and two stoppers, was charged with methyl-2,3,4,6-tetra-*O*-benzyl D-pyranoside (31 g, 0.055 mol, 1 eq.) dissolved in AcOH (30 mL per 1.00 g of starting material) and freshly prepared 2N H_2SO_4 (15 mL per 1.00 g of starting material). The reaction flask was heated to 95 °C, and TLC was used to analyze the progress of the reaction. Upon complete disappearance of starting material (~18-24 h, R_f 0.5 in 7:3 hexane:ethyl acetate), cold water (50 mL per 1.00 g of starting material) was added to the reaction mixture.

- In the case of glucose -

The resulting white-solids containing solution was filtered, solids were sequentially washed with cold 80% methanol (10 mL per 1.00 g of starting material) and hexanes (10 mL per 1.00 g of starting material) to get pure 75% yield of 2,3,4,6-tetra-*O*-benzyl D-glucopyranose as a white solid, which could be used for the other reactions without further purification.

- In the case of xylose -

The resulting white-solid solution was filtered. Solids were sequentially washed with hexanes (10 mL per 1.00 g of starting material) and recrystallization from methanol to get pure 69% yield of 2,3,4,6-tetra-*O*-benzyl D-xylopyranose as a white solid, which could be used for the other reactions without further purification.

- In the case of mannose and galactose -

The resulting mixture was extracted with ethyl acetate (3 x 30 mL per 1 g of starting material). The combined organic extracts were sequentially washed with $NaHCO_3$ (2 x 10 mL per 1 g of starting material), water (10 mL per 1 g of starting material) and brine (10 mL per 1 g of starting material) and dried with Na_2SO_4. The solvents were removed under reduced pressure and the resulting crude product was purified by column chromatography using hexane: ethyl acetate as a solvent system to give respective 2,3,4,6-tetra-*O*-benzyl D-pyranoses (75 - 82%) as colorless oils.

Characterization data for new compounds (xylo-series)

Compound 11. Obtained as colorless oil in 71% from **10** using **general procedure-1**. R_f 0.4 (10:3 Hex:EtOAc); ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.39-7.35 (m, 17H), 6.03-5.88 (comp, 1H), 5.48-5.21 (comp, 3H), 4.96-4.43 (comp, 7H), 4.30-4.19 (comp, 1H), 4.05-3.59 (comp, 5.8H), 3.42-3.28 (comp, 0.2H), 2.83 (comp, 2H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 138.6, 138.1, 138.0, 137.8, 128.6, 128.5, 128.4 (2), 128.2 (2), 128.1, 128.0, 127.9 (2), 115.6, 81.5, 79.5, 79.3, 79.1, 78.7, 77.8, 74.8, 74.3, 73.3, 72.9, 72.7, 72.5, 72.0, 61.6, 60.5; TOF HRMS (DART) m/z calcd for $C_{28}H_{33}O_5$ $(M+H)^+$ 449.2328, found 449.2343.

Compound 12. Obtained as colorless oil in 60% yield from **11** after two steps using **general procedure-2**. R_f 0.5 (5:1 Hex:EtOAc); ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.42-7.27 (m, 23H), 6.30 (d, *J* = 2 Hz, 0.1H), 5.99 (d, *J* = 7.7 Hz, 1H), 5.45 (dd, *J* = 7.7, 9.3 Hz, 1H), 5.11 (s, 0.1H), 4.96-4.57 (comp, 9H), 3.83-3.73 (comp, 5H), 2.16-1.94 (comp, 9H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 170.6, 169.9, 169.4, 169.1, 138.5, 138.2, 138.0, 128.4, 128.3 (2), 128.1, 128.0, 127.8 (2), 127.7, 127.6, 127.5, 127.0, 96.1, 96.0, 94.1, 92.0, 86.3, 84.1, 81.7, 79.4, 78.3, 77.9, 76.6, 76.3, 75.8, 74.7, 74.4, 73.6, 73.4, 72.7, 72.0, 71.9, 71.2, 63.4, 63.0, 62.0, 21.0, 20.9, 20.7; TOF HRMS (DART) m/z calcd for $C_{31}H_{35}O_8N$ $(M+NH_4)^+$ 552.2597, found 552.2616.

Compound 13. Obtained as colorless oil in 78% yield from **12** after two steps using **general procedure-3**. R_f 0.4 (1:1 Hex:EtOAc); ^{1}HNMR (400 MHz, $CDCl_3$) δ 5.88 (d, *J* = 7.4 Hz, 1H), 5.38-5.21 (comp, 4H), 5.05-5.00 (m, 1.3H), 3.88-3.79 (m, 2H), 2.09-1.98 (comp, 24H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 170.4, 170.1, 170.0, 169.7, 169.5, 169.3, 169.0, 168.7, 96.6, 95.6, 93.8, 90.0, 79.9, 72.7, 72.0, 70.9, 70.5, 70.1, 69.3, 68.9, 68.2, 66.5, 62.7, 62.0, 53.5, 20.9, 20.7, 20.6, 20.5, 20.4, 20.3; TOF HRMS (DART) m/z calcd for $C_{16}H_{26}O_{11}N$ $(M+NH_4)^+$ 408.1506, found 408.1517.

Characterization data for new compounds (manno-series)

Compound 17. Obtained as colorless oil in 73% yield from **16** after two steps using the **general procedure-2**. R_f 0.5 (5:1 Hex:EtOAc); ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.45-7.20 (m, 85 H), 6.39 (d, *J* = 3.4 Hz, 0.6 H), 6.31 (d, *J* = 7.4 Hz, 1H), 6.03 (d, *J* = 1 Hz, 0.1H), 5.81 (d, *J* = 3.9 Hz, 0.2H), 5.72-5.70 (m, 1.5H), 5.01-4.95 (m, 2.8H), 4.86-4.38 (comp, 31H), 4.18-3.63 (comp, 25H), 2.43 (s, 0.9H), 2.20-1.90 (comp, 26H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 170.3, 170.2, 170.1, 169.9, 169.6, 169.1, 138.6, 138.5, 138.4, 138.3, 138.2, 128.7, 128.6(2), 128.5, 128.4, 128.2, 128.1, 128.0 (3), 127.8, 93.0, 92.9, 89.5, 86.5, 80.2, 78.7, 77.4, 77.3, 76.9, 76.8, 75.7, 75.5, 75.1, 74.9, 74.5, 74.4, 73.9, 73.7, 73.6, 73.5(2), 73.4, 71.3, 71.2, 70.7, 70.5, 70.4, 70.3, 21.7, 21.4, 21.3, 21.2, 21.1, 21.0; TOF HRMS (DART) m/z calcd for $C_{39}H_{46}O_9N$ $(M+NH_4)^+$ 672.3173, found 672.3173.

Compound 18. Obtained in as colorless oil 85% yield from **17** after two steps using the **general procedure-3**. R_f 0.4 (1:1 Hex:EtOAc); ^{1}HNMR (400 MHz, $CDCl_3$) δ 6.18 (d, *J* = 1.5 Hz, 0.5H), 6.03 (d, *J* = 3.5Hz, 0.5H), 5.90 (d, *J* = 7.2 Hz, 1H), 5.50 (d, *J* = 8.0 Hz, 0.5H), 5.39-4.82 (comp, 13H), 4.24-3.82 (comp, 9H), 2.04-1.81 (comp, 55H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 171.1, 170.3, 170.2, 170.1, 169.7, 169.6 (2), 169.5, 169.4, 169.2, 169.1, 169.0, 168.6 (2), 168.2, 95.7, 95.6, 93.9, 92.3, 88.9, 80.8, 78.5, 72.9, 71.4, 70.9, 70.8, 70.7, 70.5, 70.4, 69.7, 69.3, 69.3, 68.7, 68.2, 67.8, 67.7, 67.6, 67.4, 63.2, 61.5, 60.1, 20.8, 20.5, 20.4, 20.3; TOF HRMS (DART) m/z calcd for $C_{19}H_{30}O_8N$ $(M+NH_4)^+$ 480.1717, found 480.1723.

Characterization data for new compounds (galacto-series)

Compound 22. Obtained as colorless oil in 73% yield from **21** over two steps using **general procedure-2**. R_f 0.5 (5:1 Hex:EtOAc); ^{1}HNMR (400 MHz, CDCl$_3$) δ 7.39-7.30(m, 106H), 6.52 (d, *J* = 1.7 Hz, 0.2H), 6.45 (d, *J* = 3.6 Hz, 1 H), 6.35 (d, *J* = 2 Hz, 0.1H), 6.25 (d, *J* = 2.0 Hz, 0.4H), 6.18 (d, *J* = 8.1 Hz, 0.75H), 5.76 (comp, 0.8H), 5.70 (dd, *J* = 2.4 Hz, 0.5H), 5.66-5.64 (m, 1.1H), 5.50-5.46 (m, 0.7H), 5.33 (dd, *J* = 4.6, 1.9 Hz, 0.5H), 5.28 (m, 1.3H), 5.11-4.34 (comp, 46H), 4.32-3.91 (comp, 18H), 3.85-3.54 (comp, 15H), 2.29-1.92 (comp, 28H); ^{13}CNMR (100 MHz, CDCl$_3$) δ 170.8, 170.6, 170.4, 169.8, 169.7, 169.6, 169.4, 169.2, 139.9, 138.8, 138.7, 138.6, 138.5, 138.4, 138.3, 138.1, 128.7, 128.6, 128.5, 128.4, 128.3, 128.1, 128.0, 127.9 (2), 127.7, 95.9, 95.8, 95.4, 94.6, 93.9, 91.1, 90.5, 90.0, 83.7, 83.3, 82.7, 81.5, 80.0, 79.5 (2), 79.1, 78.9, 78.7, 78.5, 78.4, 78.0, 77.6, 76.9, 76.2, 75.7, 75.6, 75.2, 75.1, 75.0, 74.9, 74.8, 74.7, 74.6, 74.5, 74.4, 74.2, 74.1, 74.0, 73.8(2), 73.7, 73.6, 73.4, 73.3, 73.1, 73.0, 72.1, 71.0, 69.1, 68.7, 68.5, 68.3, 21.5, 21.3 (2), 21.1; TOF HRMS (DART) m/z calcd for $C_{39}H_{46}O_9N$ (M+NH$_4$)$^+$ 672.3173, found 672.3172.

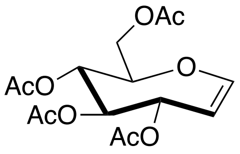
Compounds 23 and **24.** Obtained as colorless oil, 1:1 mixture in 85% from **21** after two steps using the **general procedure-3.** R_f 0.35 (1:1 Hex:EtOAc); ^{1}HNMR (400 MHz, CDCl$_3$) δ 6.33 (d, *J* = 4.1 Hz, 0.1H), 6.28 (comp, 0.1H), 6.23 (d, *J* = 3.6 Hz, 0.2H), 6.20 (dd, *J* = 8.0, 1.3 Hz, 1H), 6.14 (d, *J* = 4.4 Hz, 0.3H), 5.96 (m, 2H), 5.76 (d, *J* = 2.9 Hz, 0.8H), 5.66 (comp, 0.6H), 5.60-4.89 (comp, 28H), 4.29-3.71 (comp, 21H), 2.15-1.88 (comp, 120H) ; ^{13}CNMR (100 MHz, CDCl$_3$) δ 170.9, 170.4, 170.3, 170.2, 170.1, 170.0, 169.9, 169.8, 169.7, 169.6, 169.4, 169.3, 169.2, 169.1, 169.0, 168.9, 168.8, 168.7, 168.1, 93.3, 96.2, 95.4, 94.9, 94.7, 93.6, 91.8, 88.9, 88.7, 80.5, 80.4, 77.7, 75.0, 72.9, 72.3, 71.9, 71.3, 70.5, 69.9, 69.8, 69.7, 69.4, 69.2, 69.1, 69.0, 68.9, 68.4, 68.3, 67.8, 67.7, 67.5, 67.4, 67.4, 67.3, 66.6, 66.3, 62.5, 62.1, 62.0, 61.6, 61.3, 60.2, 20.9, 20.8, 20.7 (2), 20.6, 20.5, 20.4, 20.3 (3); TOF HRMS (DART) m/z calcd for $C_{19}H_{30}O_8N$ (M+NH$_4$)$^+$ 480.1717, found 480.1721.

Characterization data for new compounds (manno-series with alternative protecting groups)

Compound 32. To the di-*O*-acetyl-tetra-*O*-benzyl septanoses **17** (0.20 g, 0.3 mmol, 1 eq.), dissolved in THF (4 mL), was added 25% wt of 10% Pd/C (0.05 g) and the mixture was stirred for 12 h at rt under positive pressure of hydrogen gas. The resulting solution was filtered through celite and washed with additional MeOH (~20 mL) then concentrated under reduced pressure. The crude material (**31**) was dissolved in DCM (4 mL) and pyridine (3.2 mL), and benzoyl chloride (3 mL) was added at 0 °C. The mixture was stirred it for 12 h at rt then concentrated under reduced pressure and purified by column chromatography using 1:1 hexane:ethyl acetate as solvent system to give tetra-*O*-benzoyl-di-*O*-acetyl septanose **32** as a white solid in 85% yield. R_f 0.3 (10:3 Hex:EtOAc); ^{1}HNMR (400 MHz, CDCl$_3$) δ 8.20-7.80 (comp, 27H), 7.65-7.19 (comp, 50H), 6.43 (d, *J* = 4.2 Hz, 0.5H), 6.32 (d, *J* = 7.2 Hz,1H), 6.12-5.87 (comp, 9.5H), 5.81 (dd, *J* = 7.3, 4.2 Hz, 0.9H), 5.70 (d, *J* = 7.2 Hz, 1.3H), 5.61 (dd, *J* = 7.4, 5.1 Hz, 1.2H), 4.91 (m, 0.7H), 4.77-4.46 (comp, 8.3H), 2.38-1.72 (comp, 24H); ^{13}CNMR (100 MHz, CDCl$_3$) δ 171.2, 169.8, 169.6, 169.5, 169.0, 168.6, 166.3, 166.1, 165.5 (2), 165.4, 165.3, 165.1, 133.8, 133.7, 133.6, 133.5, 133.4, 133.1, 130.3, 130.2 (2), 130.1, 130.0, 129.9 (2), 129.7, 129.1, 129.0, 128.8, 128.7, 128.6 (2), 128.5, 128.4, 128.3, 96.5, 92.9, 89.6, 79.5, 74.1, 72.4, 72.0, 71.5, 71.4 (2), 71.2, 70.7, 70.5 (2), 69.8, 69.0, 68.2, 64.5, 64.3, 64.2, 21.6, 20.8, 20.7, 20.6, 20.5; TOF HRMS (DART) m/z calcd for $C_{39}H_{38}O_{13}N$ (M+NH$_4$)$^+$ 728.2343, found 728.2329.
(for ***benzoylation*** *ref: Carbohydr. Res.,* 2011, **346**, 1250–1256)

Compound 34. To the di-*O*-acetyl-tetra-*O*-benzyl septanoses **17** (0.20 g, 0.3 mmol, 1 eq.) dissolved in THF (4 mL), was added 25% wt of 10% Pd/C (0.05 g) and the mixture was stirred for 12 h at rt under an atmosphere of hydrogen gas. The resulting solution was filtered through celite, and was washed with additional MeOH (~20 mL). The combined filtrates were concentrated under reduced pressure. The crude product (**31**) was dissolved in acetone (12 mL), and dimethoxypropane (12 mL). To this mixture, CSA (0.007 g, 0.003 mmol, 0.01 eq.) was added and it was allowed to stir for 24 h at rt. After, NaHCO$_3$ (1 mL) was added to neutralize the reaction mixture. The mixture was diluted with H$_2$O (5 mL) and the resulting mixture was extracted with EtOAc (3x20 mL); the combined organic extracts were dried with Na$_2$SO$_4$ and concentrated under reduced pressure and purified by column chromatography using 1:1 hexane:ethyl acetate as solvent system to give di-*O*-acetonide-di-*O*-acetyl septanose **34** as colorless oil in 64% yield. R_f 0.5 (1:1 Hex:EtOAc); ^{1}HNMR (400 MHz, CDCl$_3$) δ 6.05 (d, *J* = 4.0 Hz, 1H), 6.01 (d, *J* = 1.6 Hz, 0.5H), 5.75 (d, *J* = 8.7 Hz, 0.4H), 5.55 (d, *J* = 8.1 Hz, 1H), 5.16-5.11 (m, 2.8H), 4.90 (dd, *J* = 8.7, 1.2 Hz, 0.5H), 4.69 (dd, *J* = 7.7, 2.3 Hz, 1H), 4.56 (dd, *J* = 9.8, 7.3 Hz, 0.8H), 4.41 (dd, *J* = 7.7, 7.7

Hz, 1.3H), 4.37-4.27 (comp, 4H), 4.19-4.08 (comp, 2.4H), 4.00-3.47 (comp, 14H), 2.16-2.04 (comp, 23H), 1.52-1.24 (comp, 50H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 169.9, 169.7, 169.3, 168.0, 168.8, 168.2, 110.7, 110.3, 110.2, 109.6, 100.3, 99.6, 99.4, 95.5, 93.2, 91.1, 89.8, 81.0, 79.6, 79.5, 79.1, 78.8, 76.0, 73.4, 73.3, 72.8, 72.7, 72.2, 72.0, 71.9, 71.2, 69.3, 65.9, 65.5, 62.5, 62.2, 62.1, 61.1, 29.8, 27.6, 27.5, 27.1, 27.0, 26.8, 26.7, 26.4, 25.2, 25.2, 24.7, 24.4, 21.3, 21.2, 21.0, 20.9, 20.7, 20.0; TOF HRMS (DART) m/z calcd for $C_{17}H_{27}O_9$ $(M+H)^+$ 375.1655, found. 375.1677 (*for* ***acetonide protection*** *ref* : *J. Am. Chem. Soc.,* **2006**, *128*, 11764–11765)



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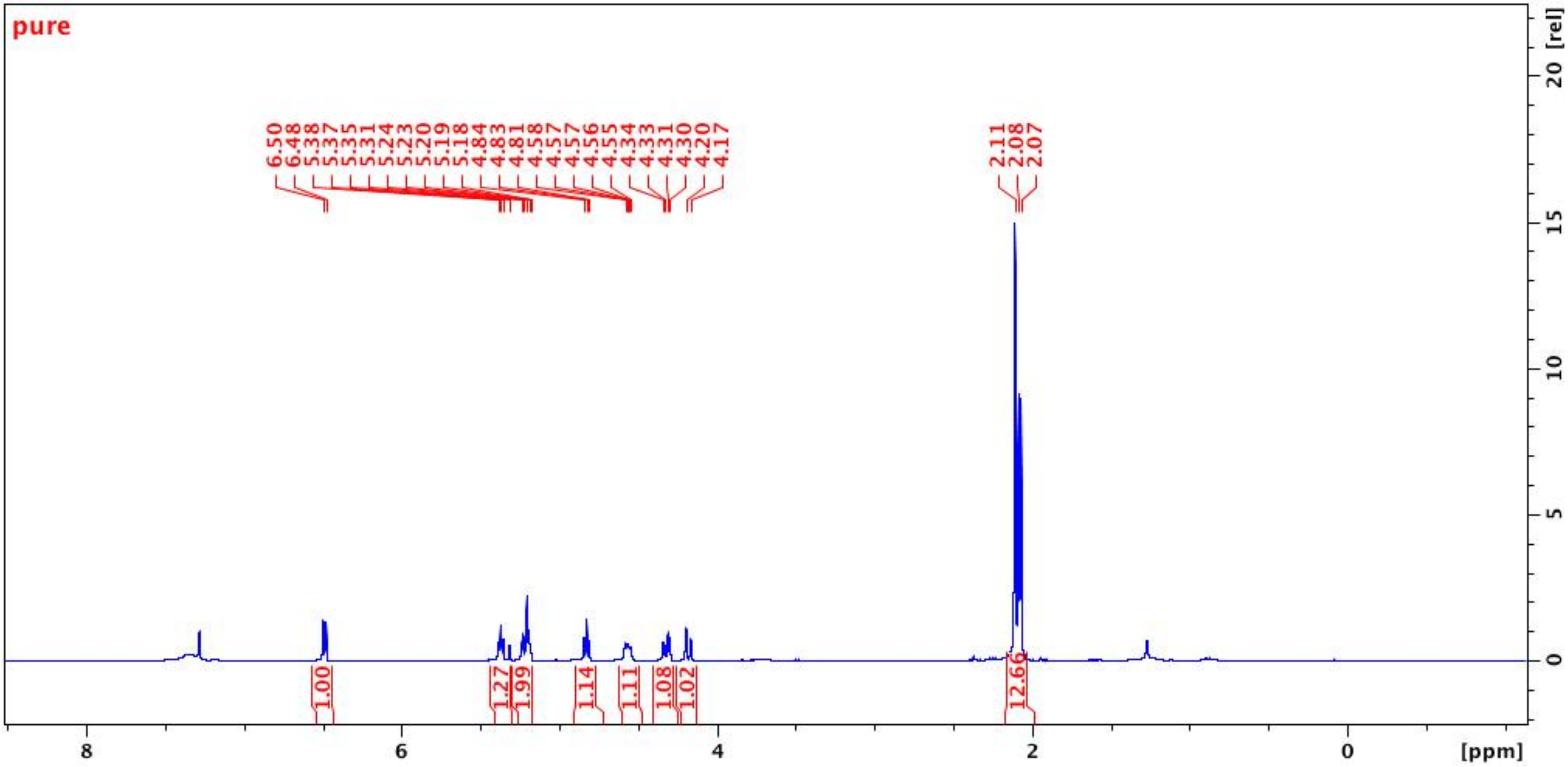

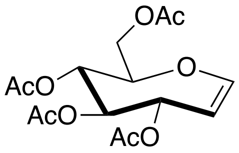

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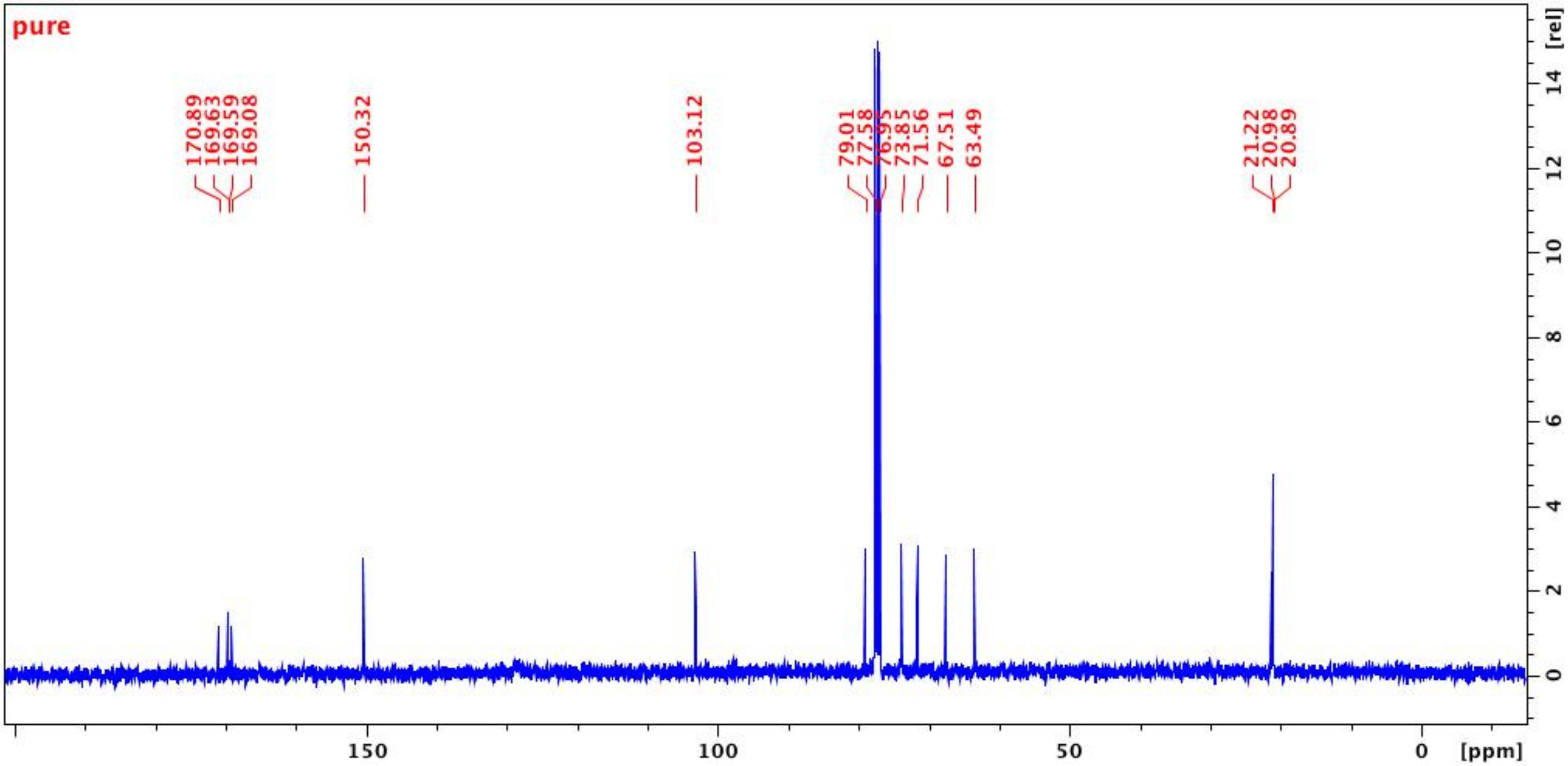

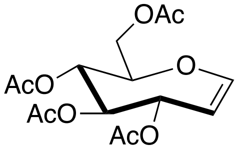

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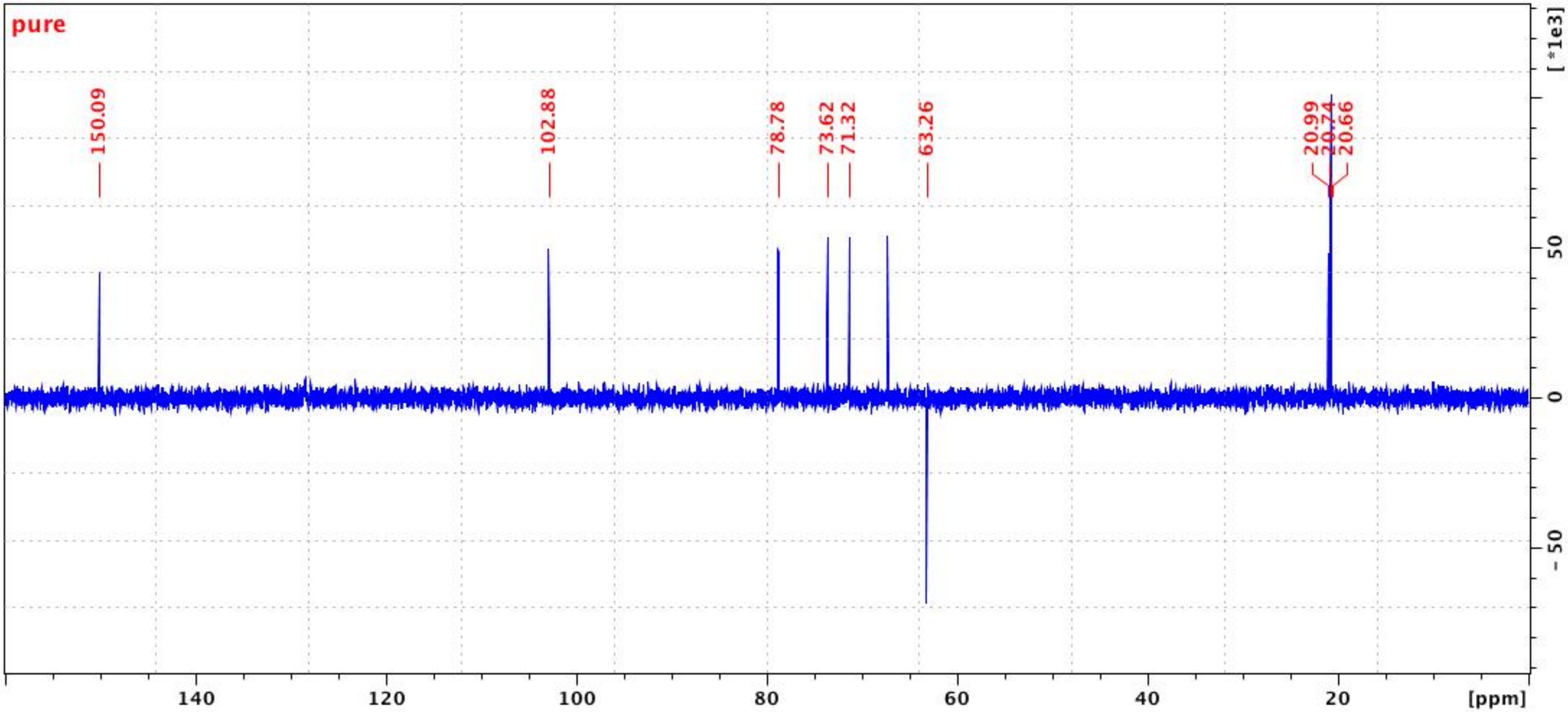

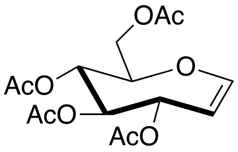

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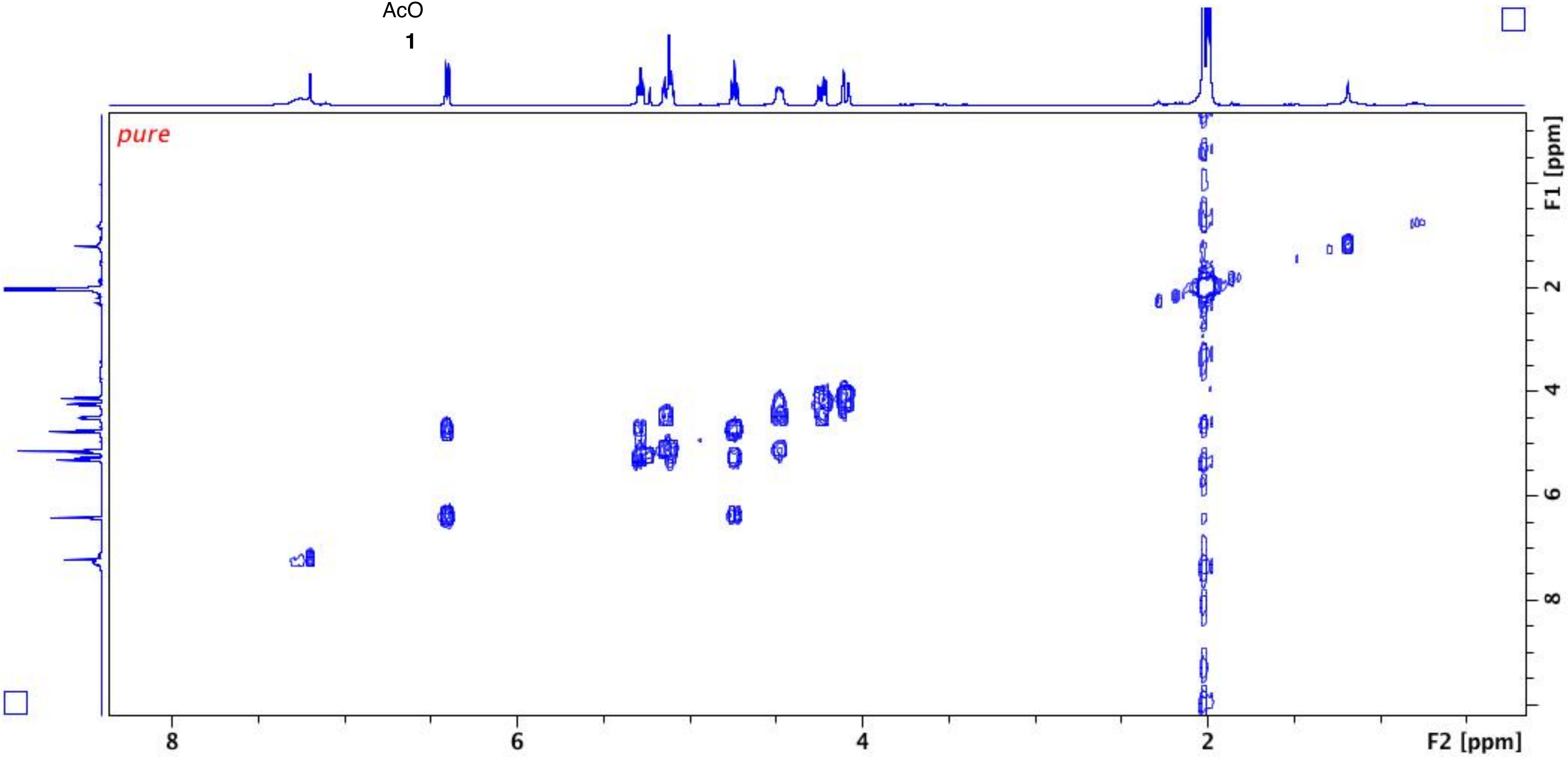

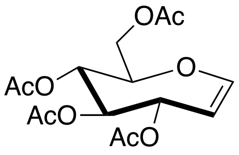

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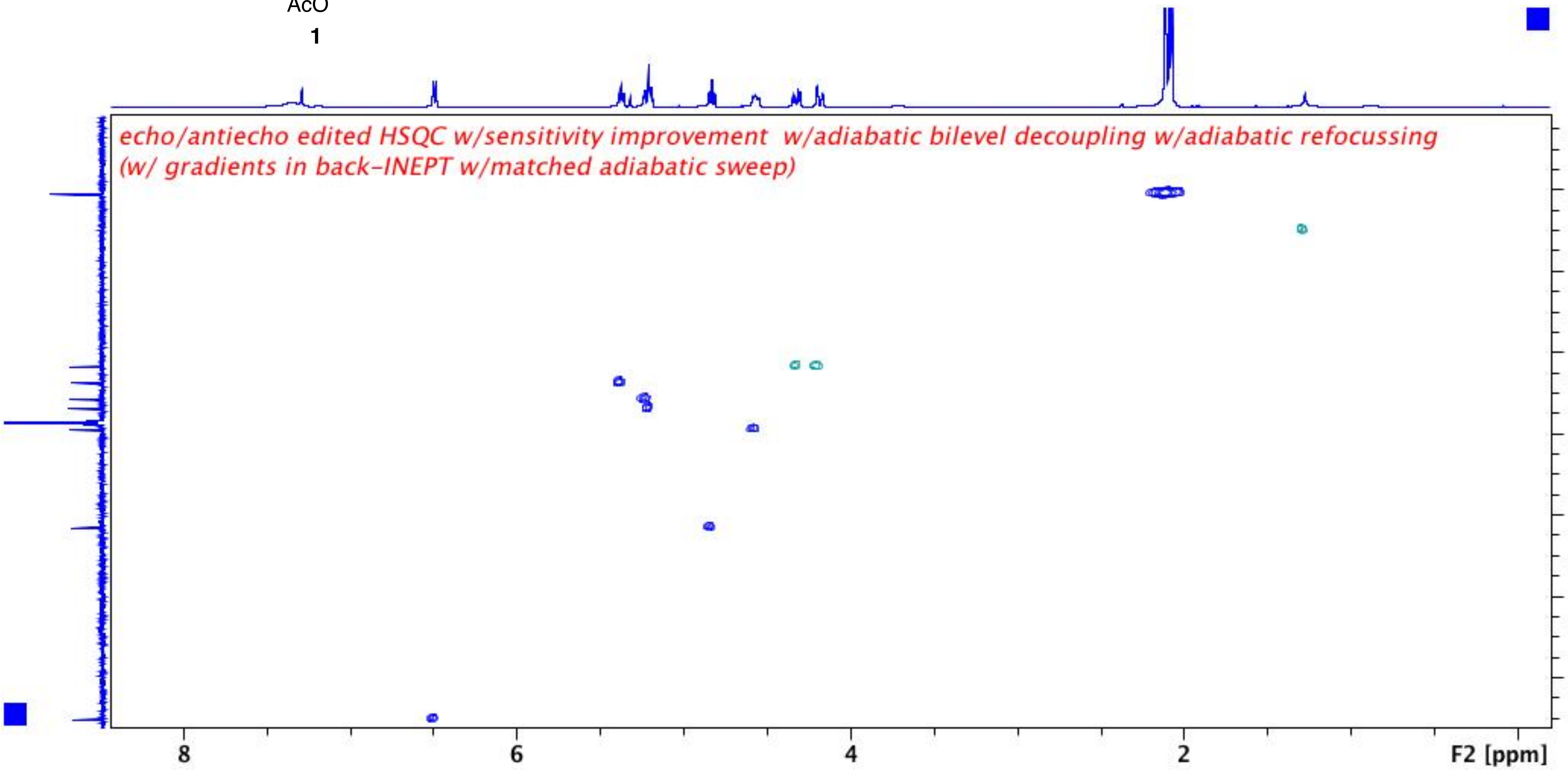

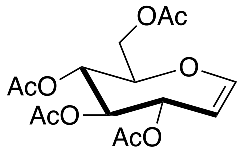

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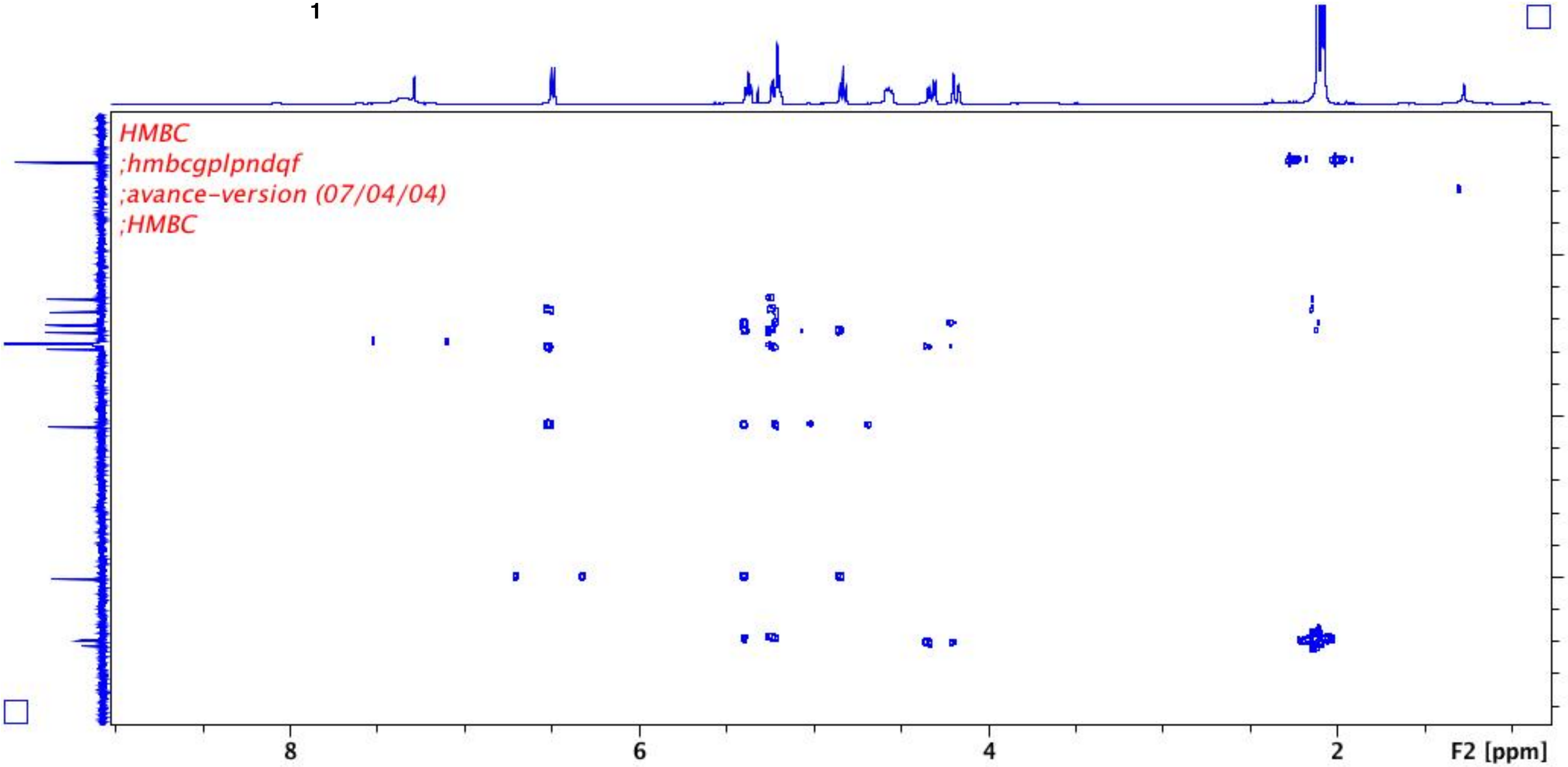

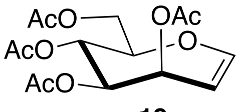

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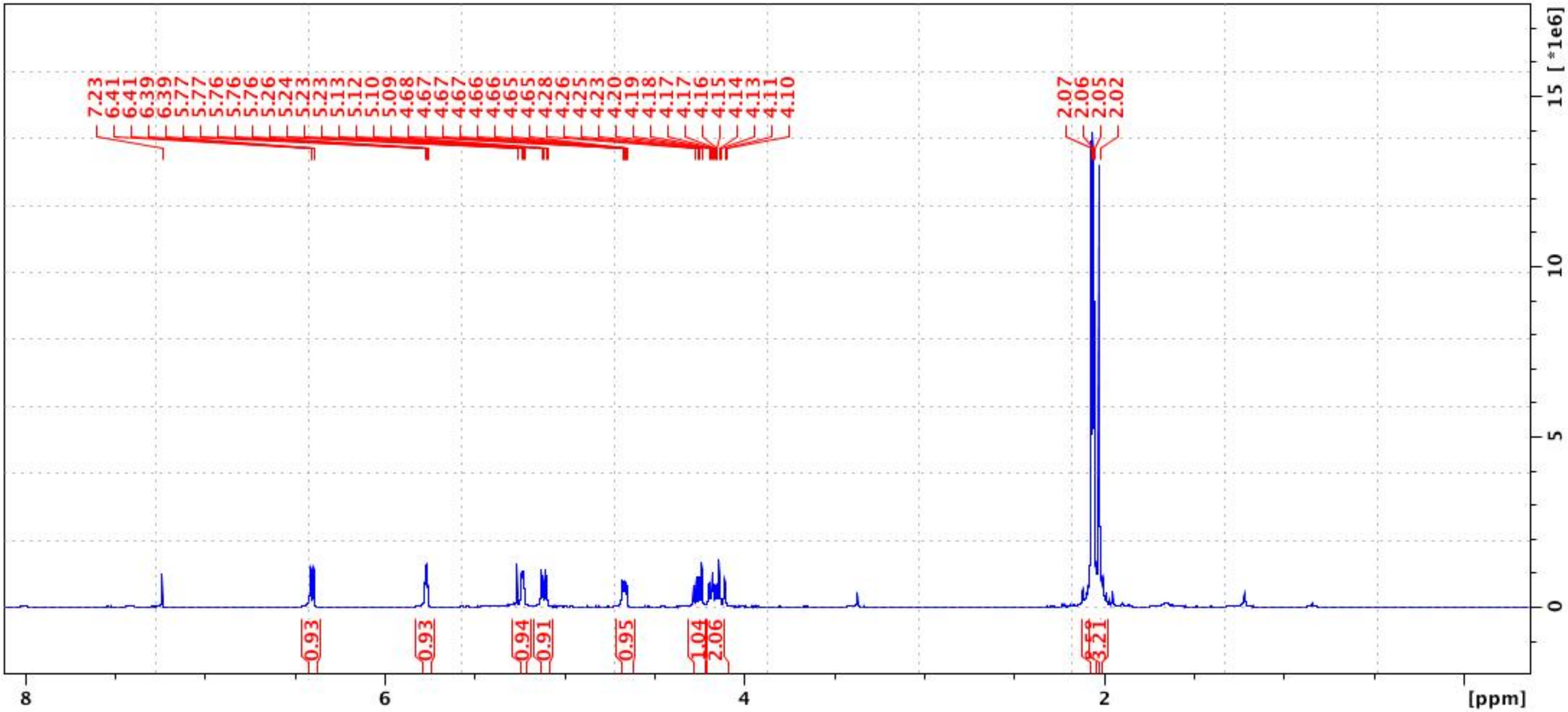

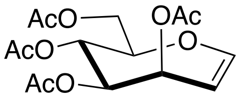

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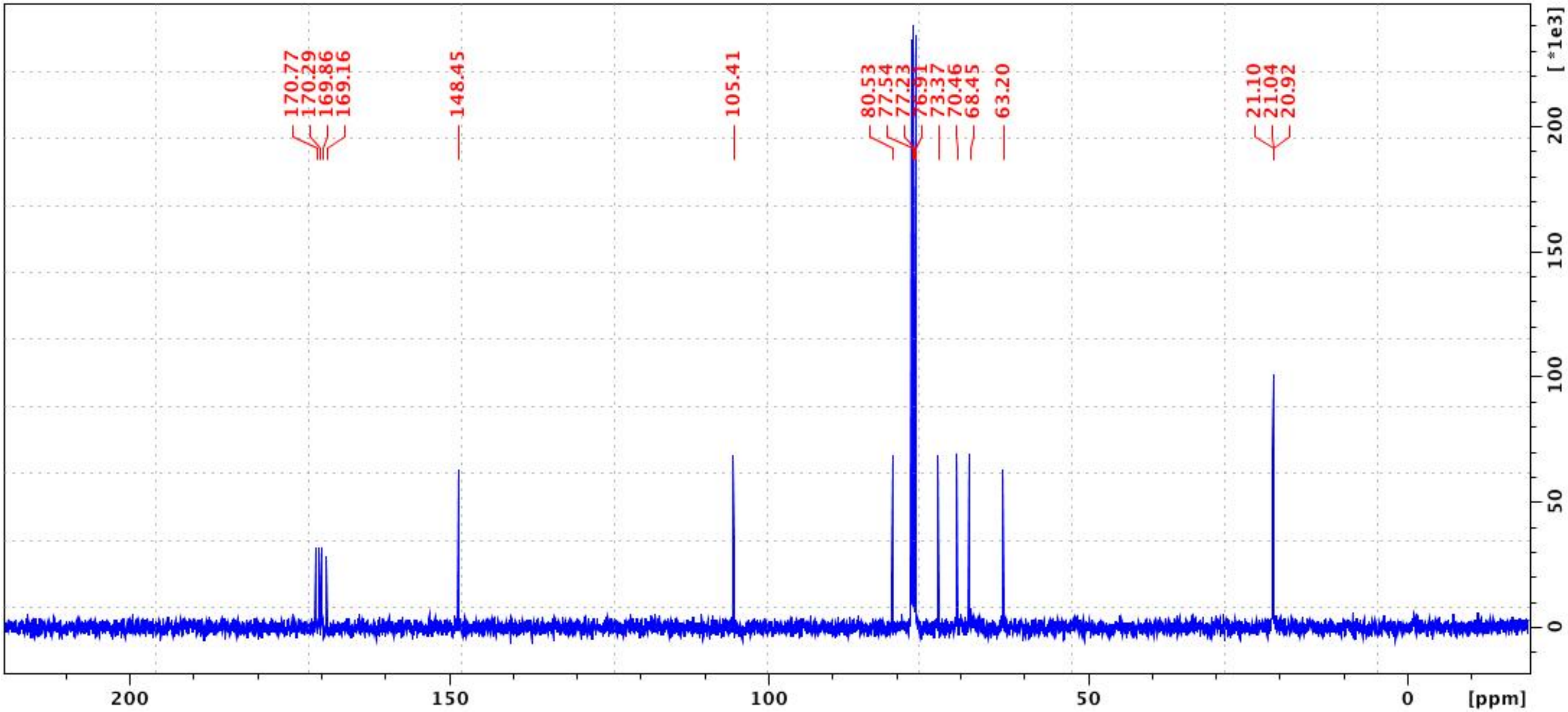

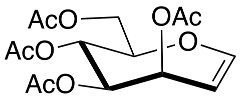

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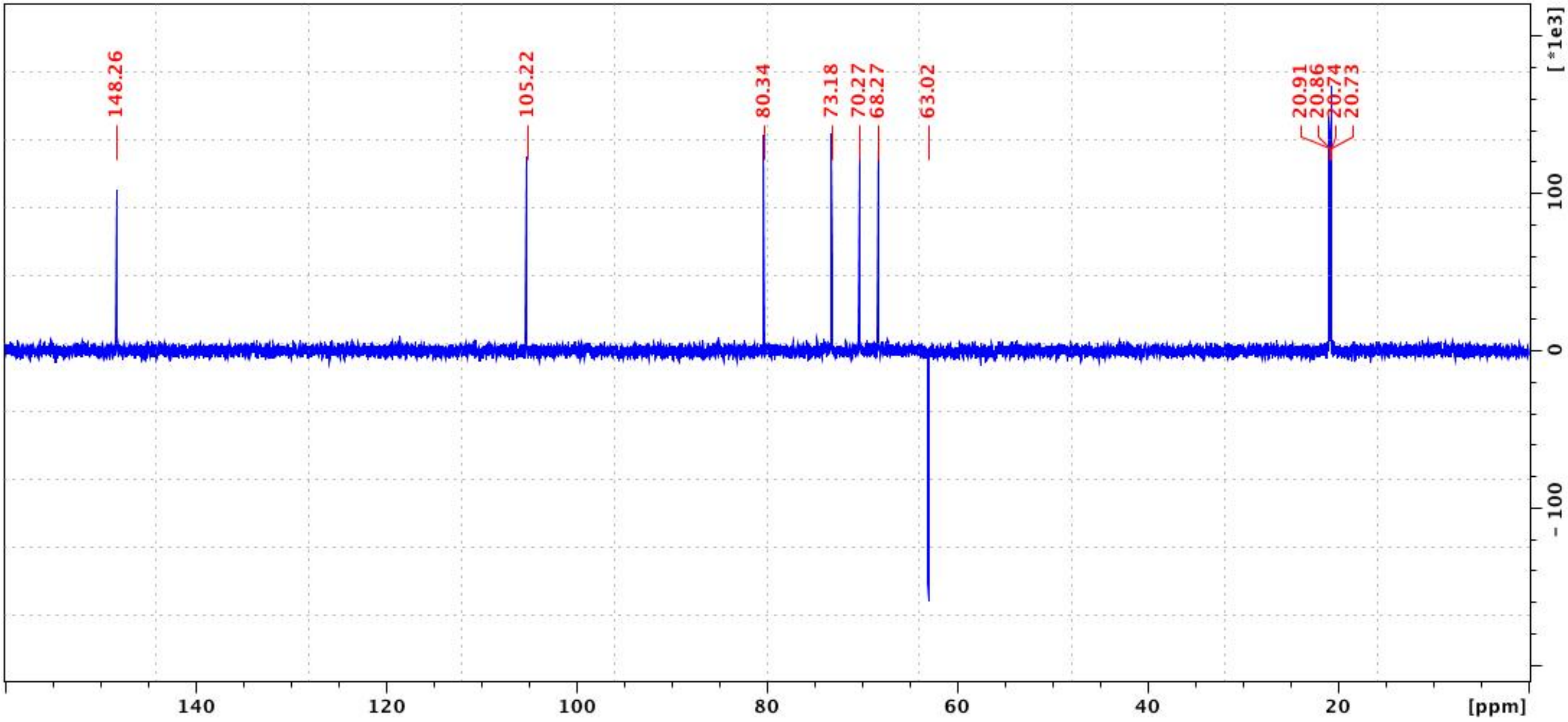

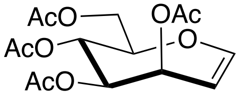

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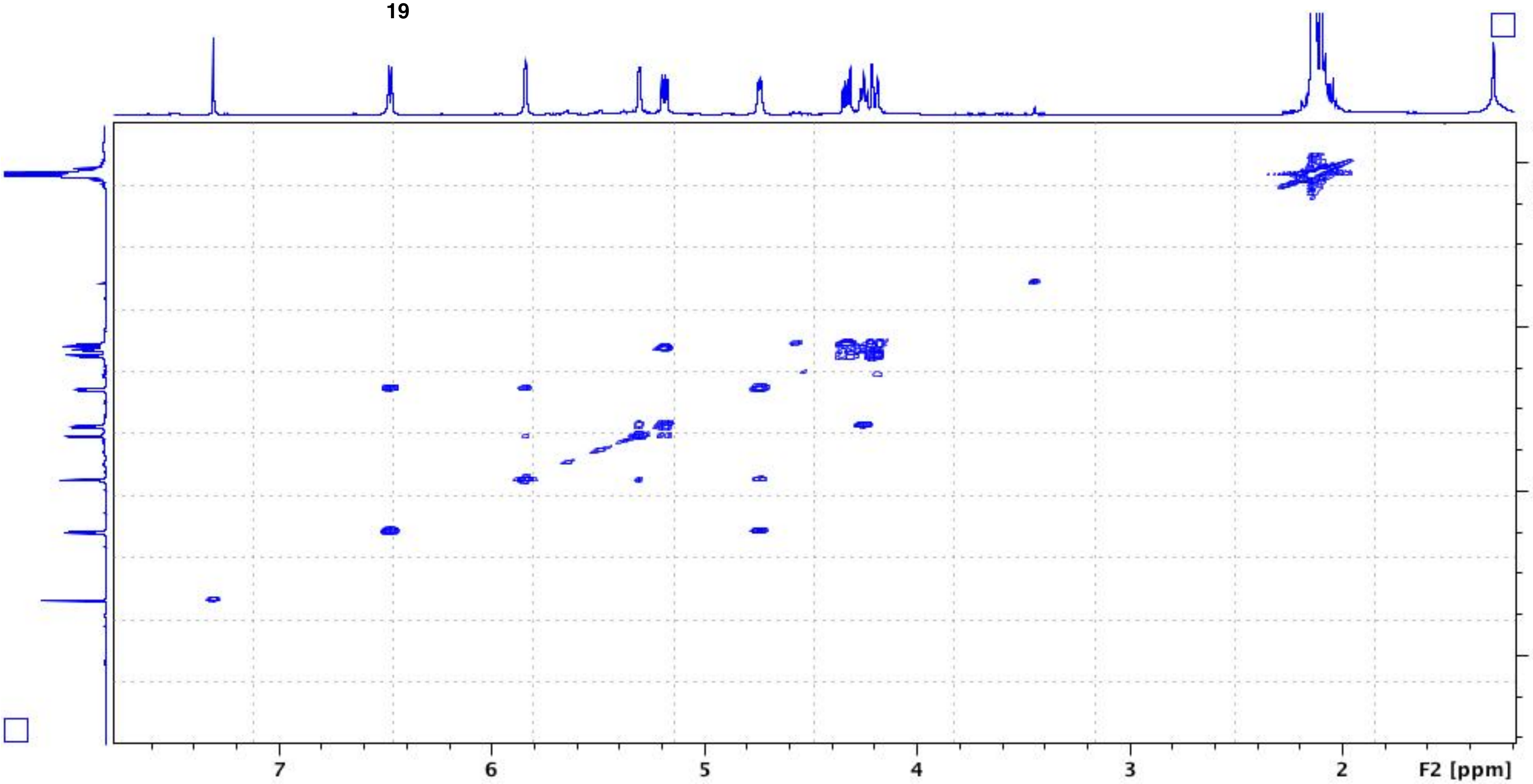

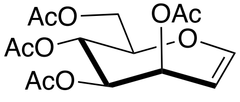

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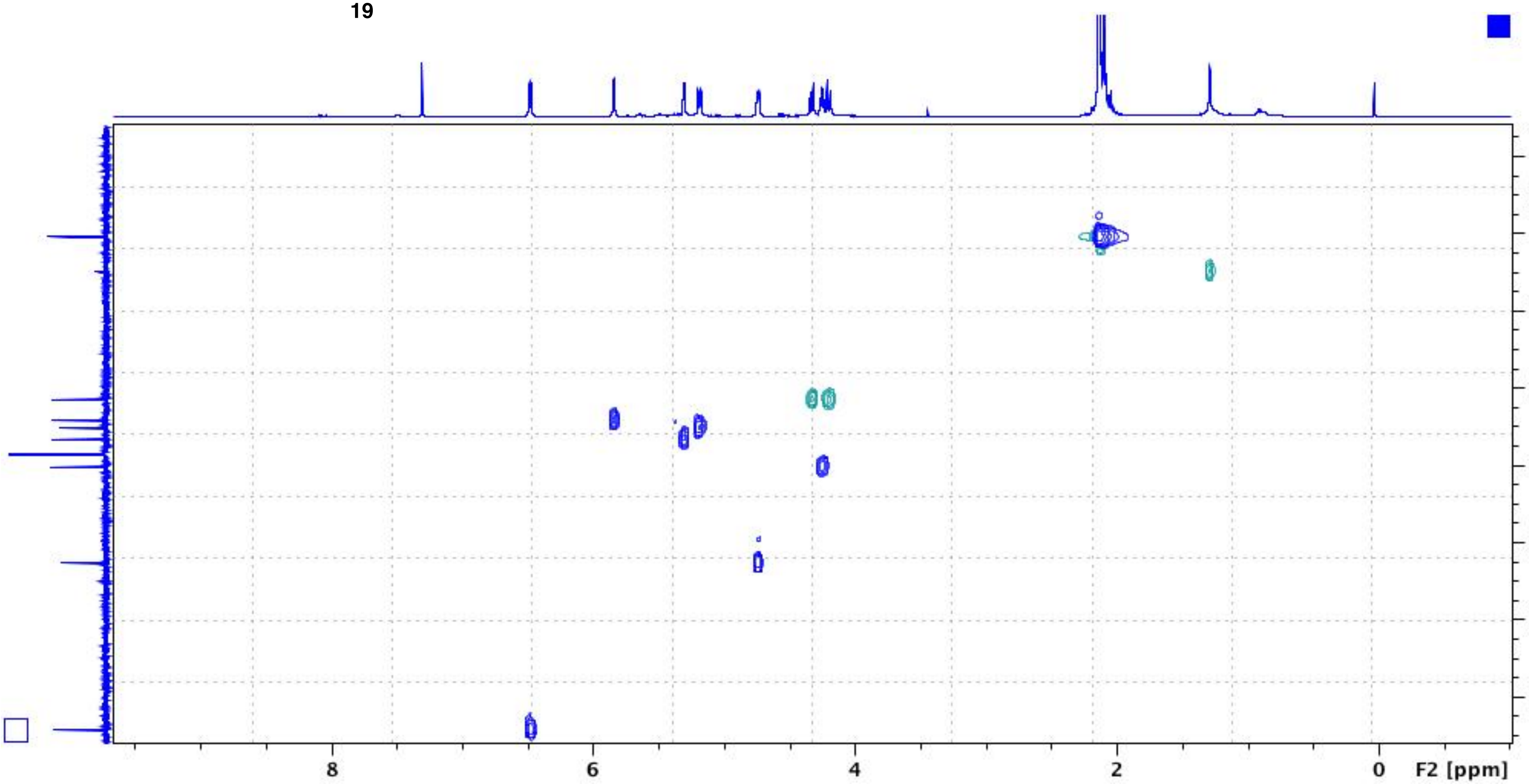

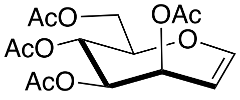

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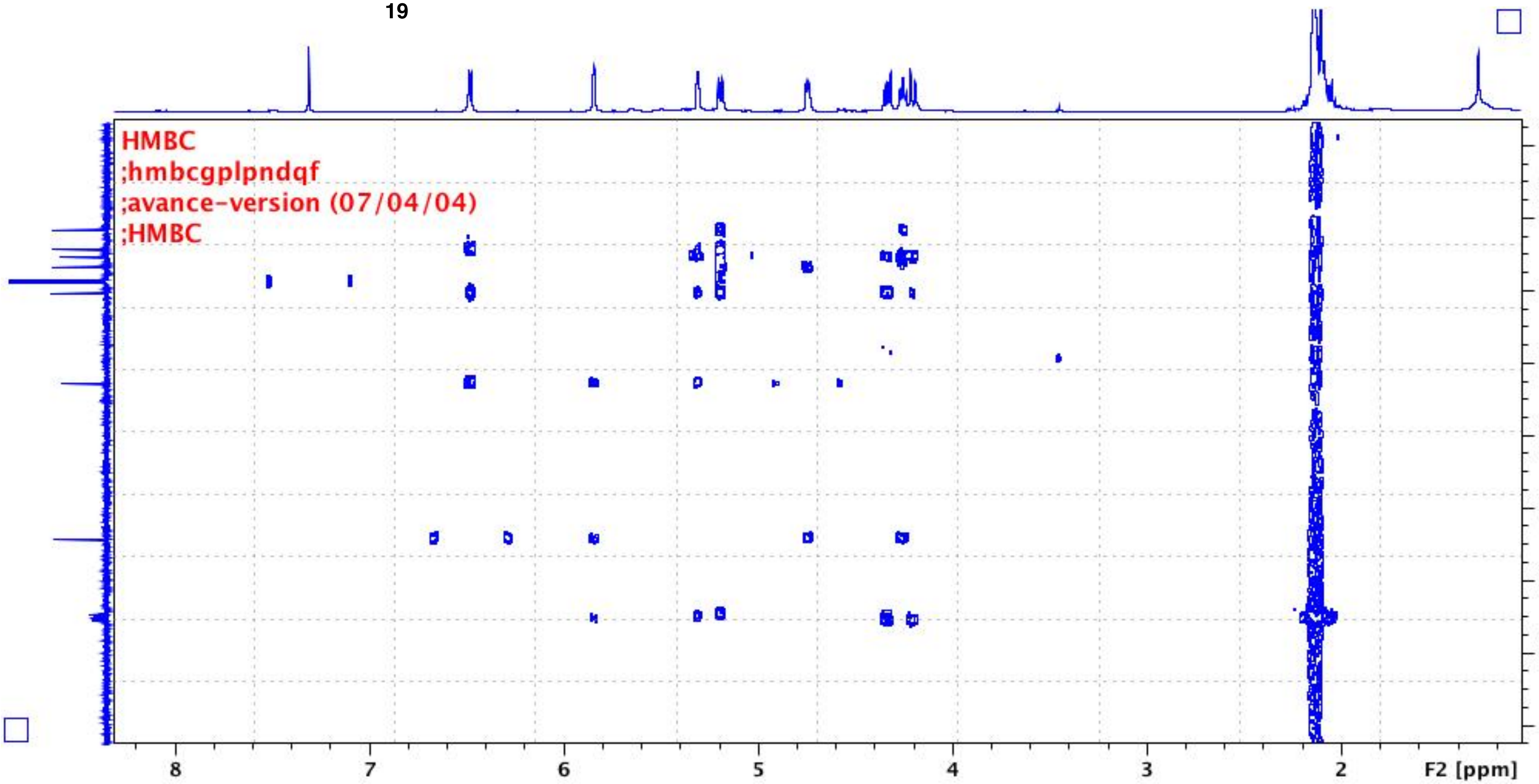

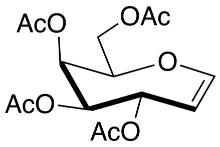

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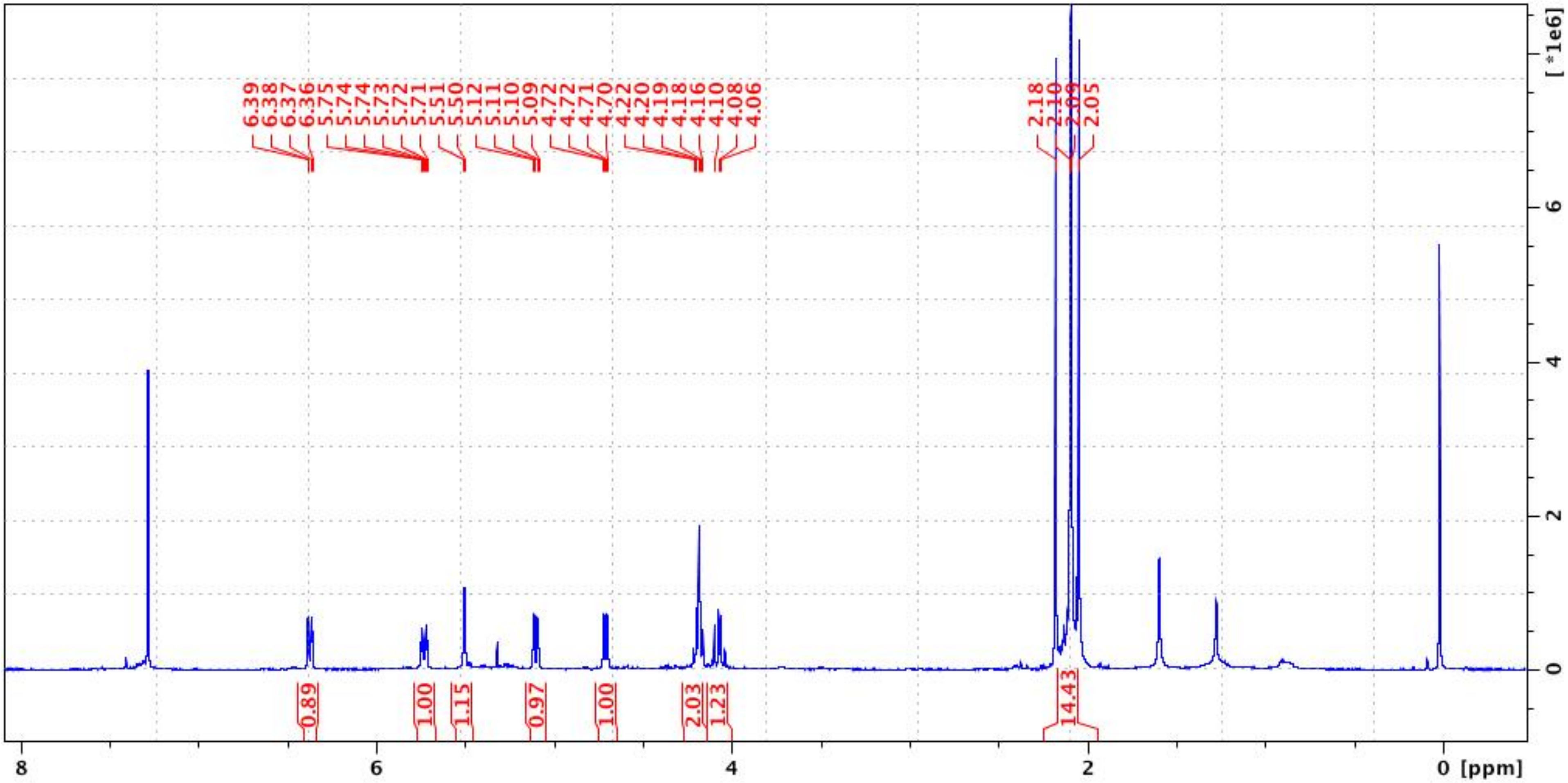

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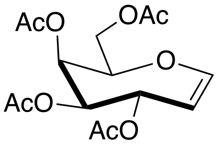

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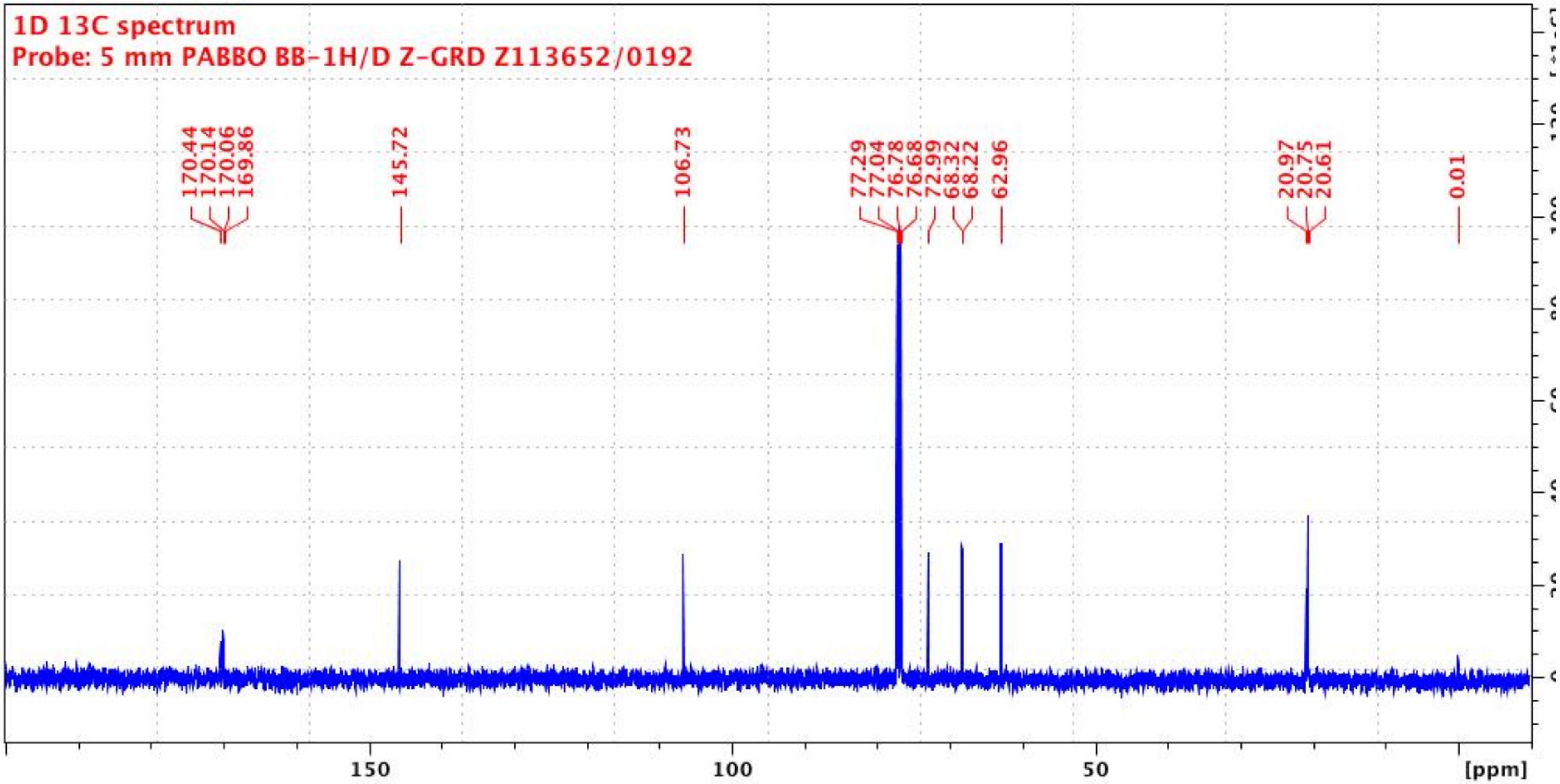

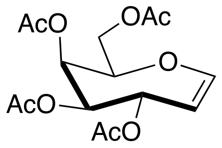

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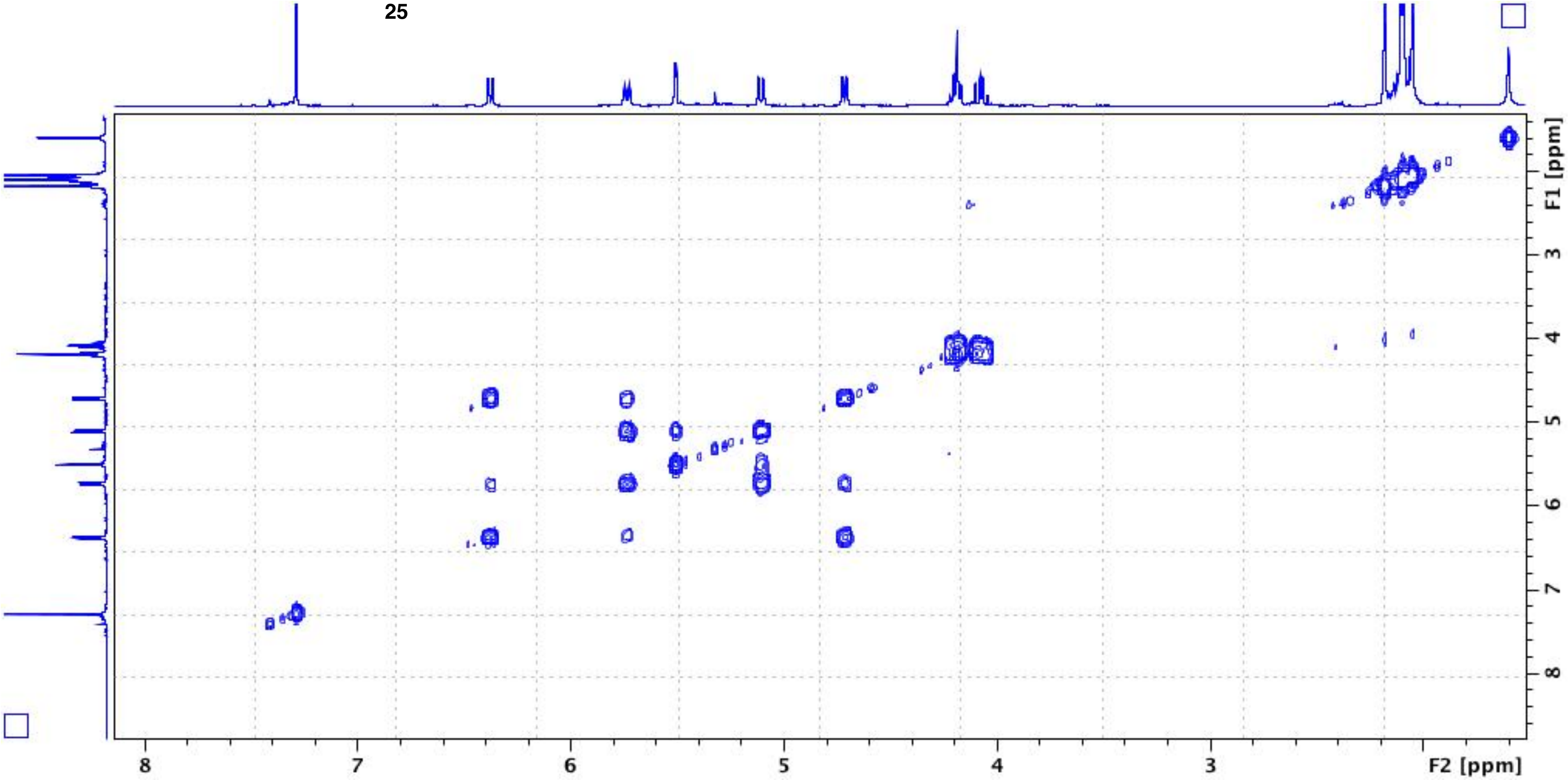

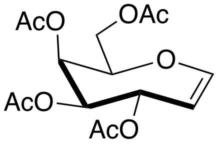

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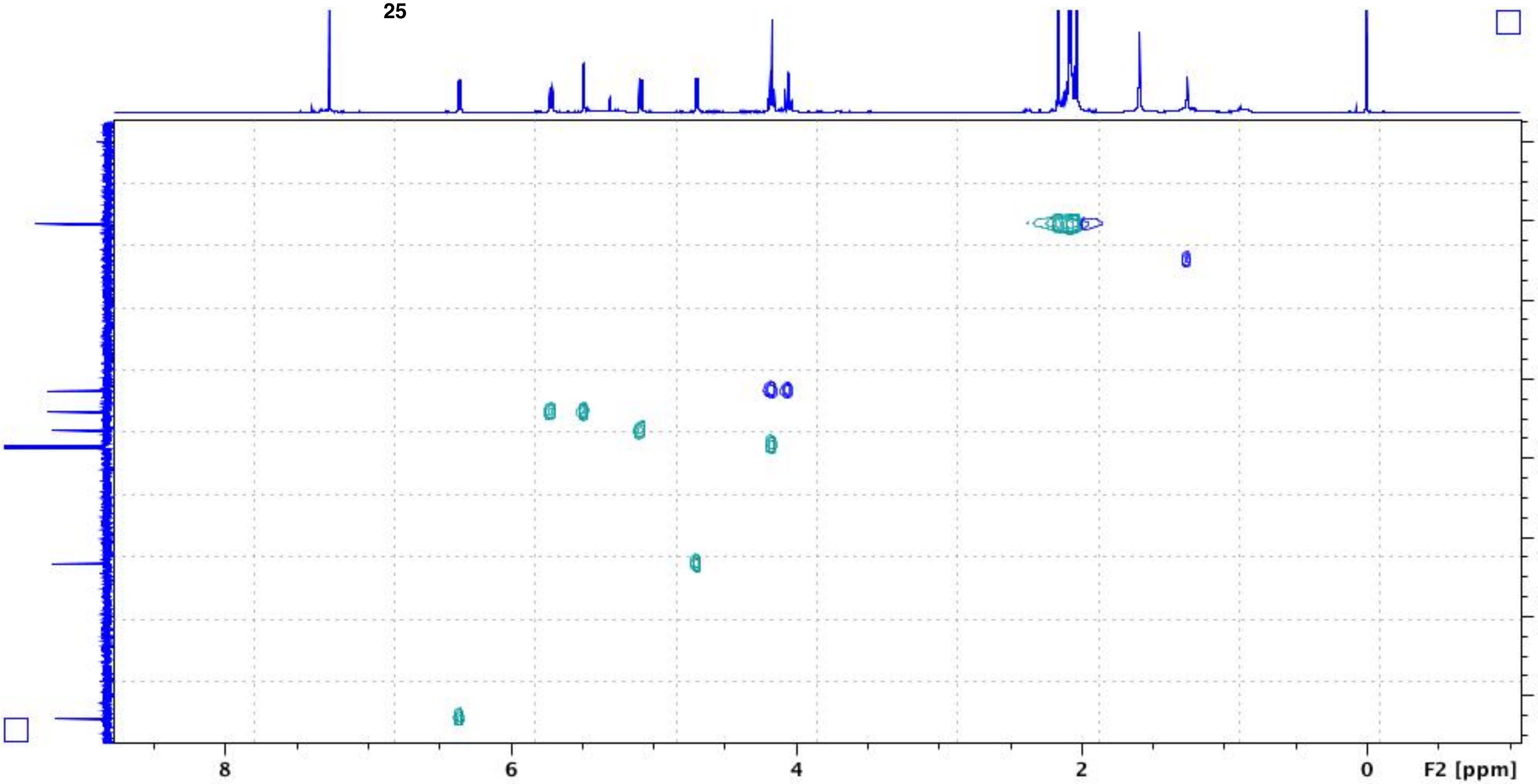

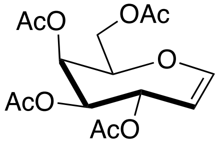

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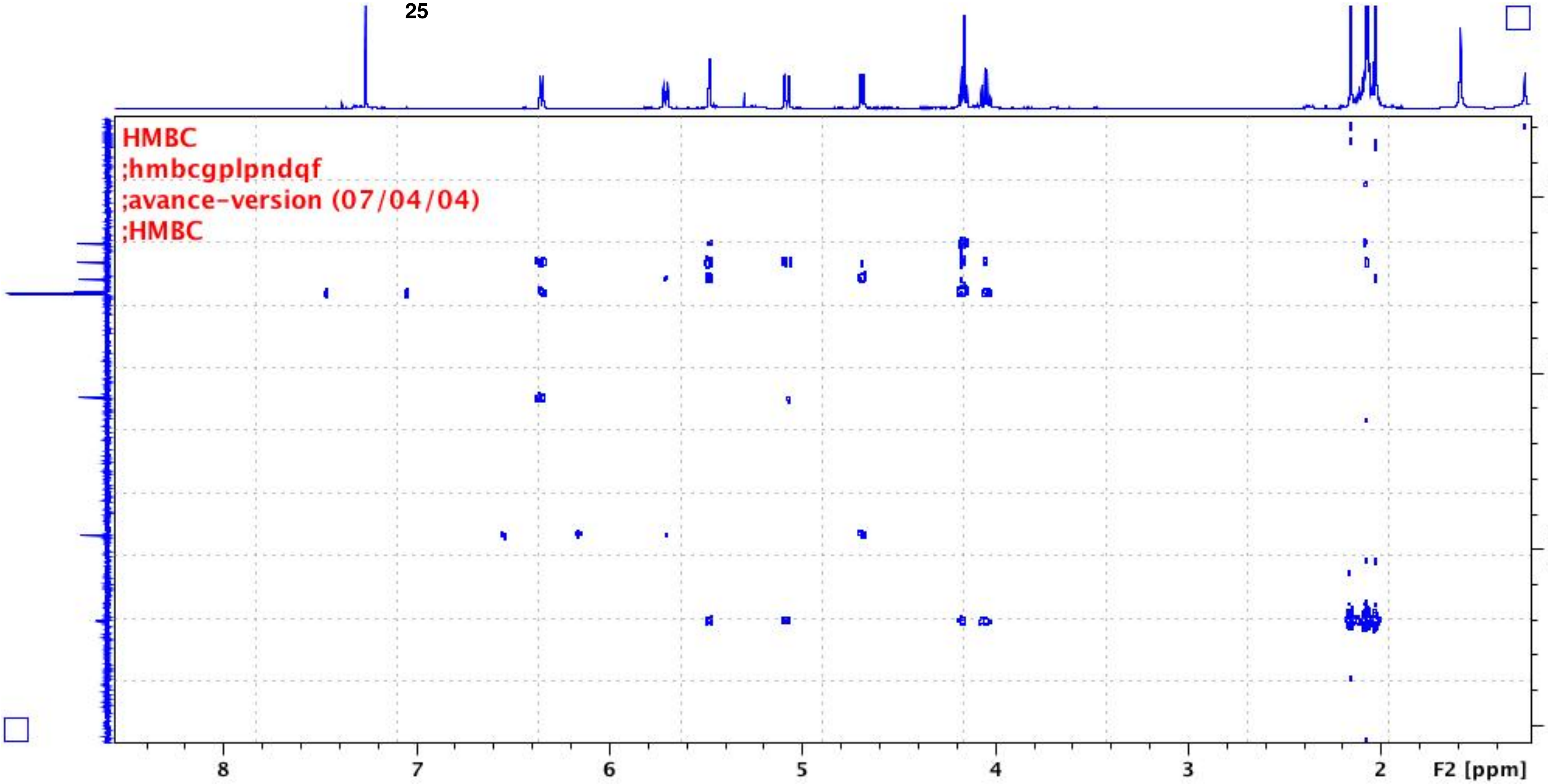

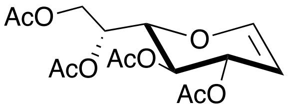

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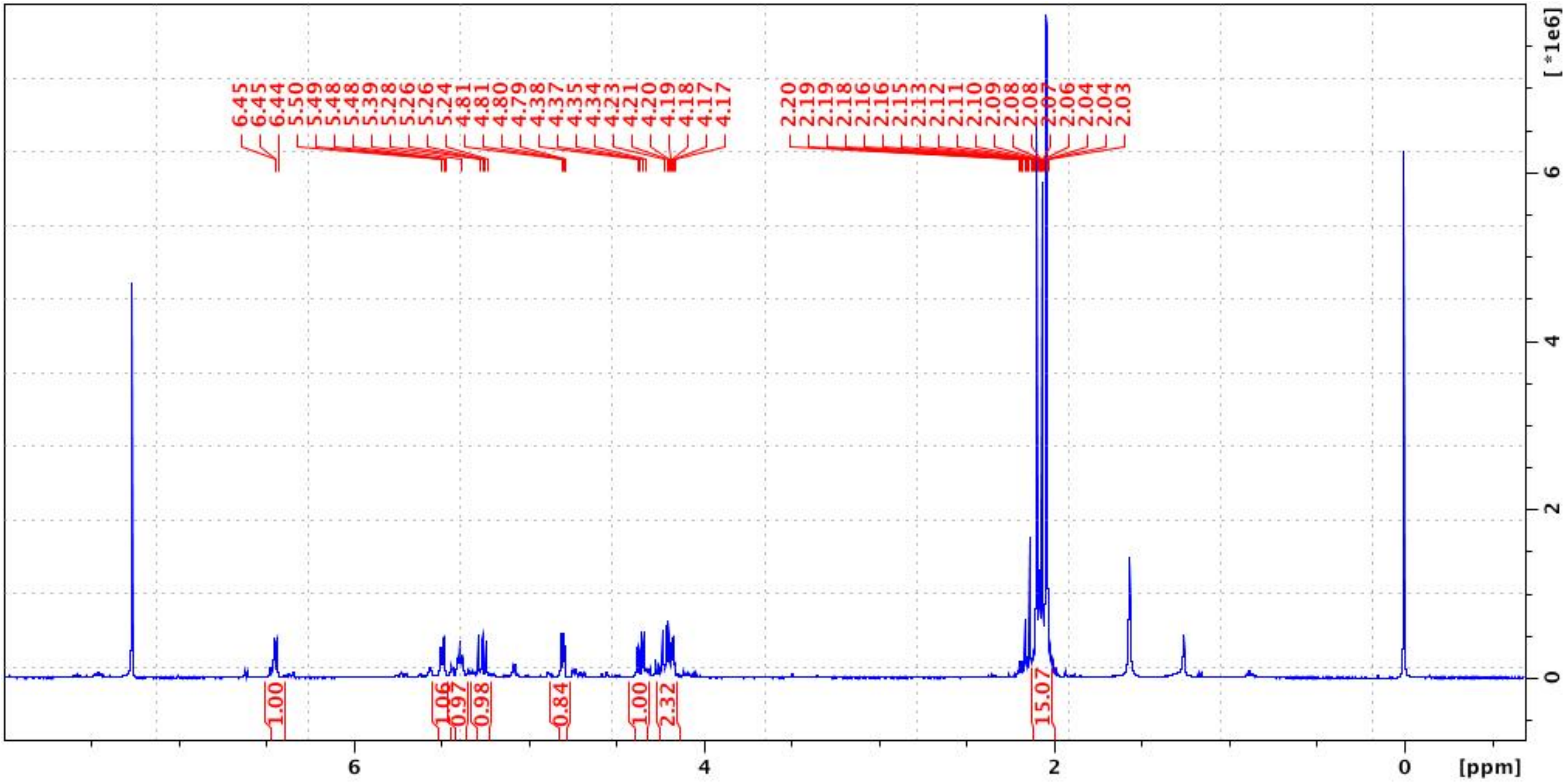

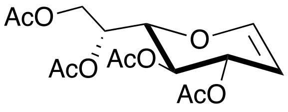

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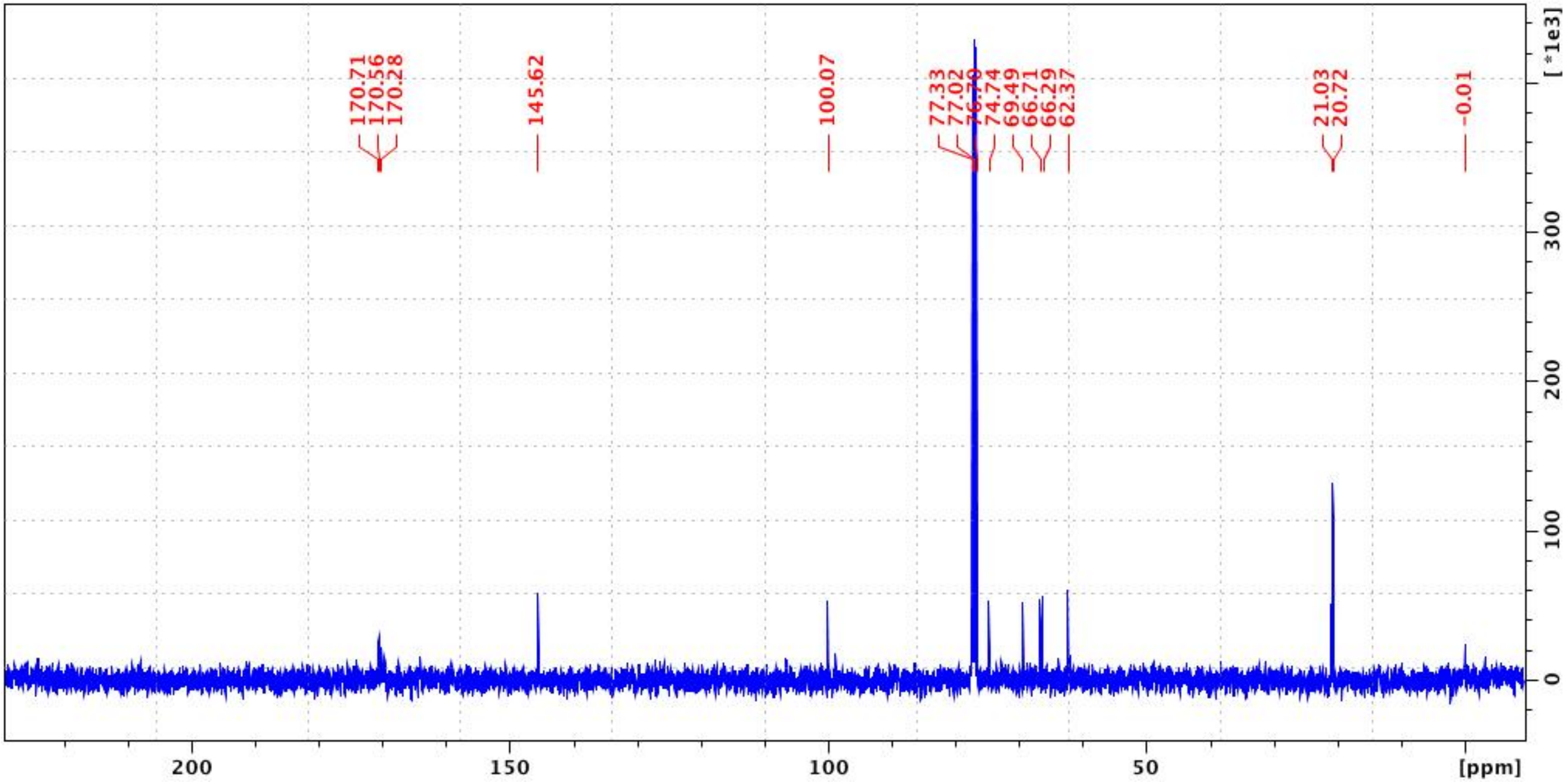

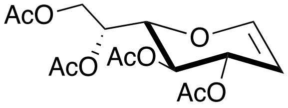

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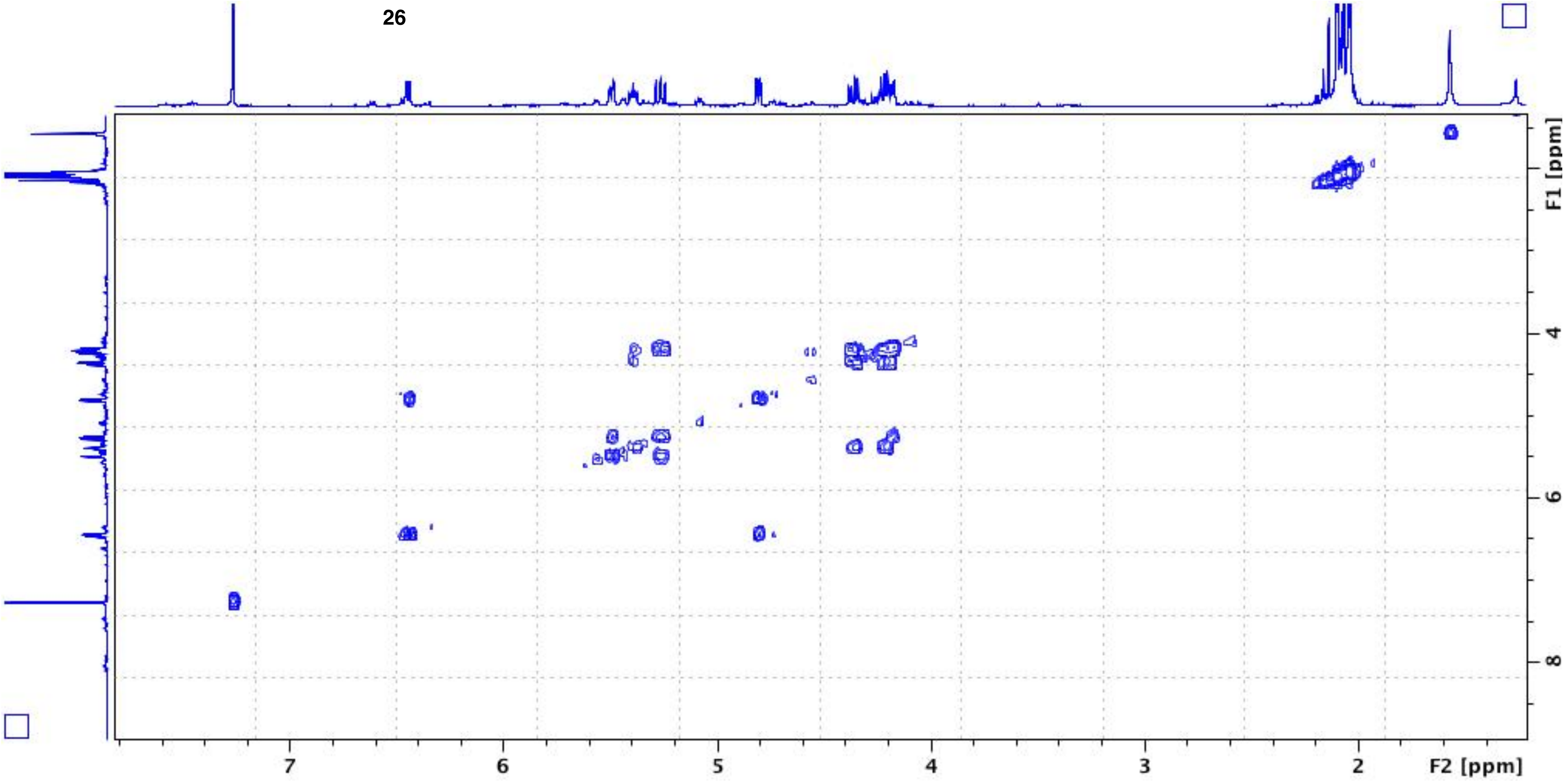

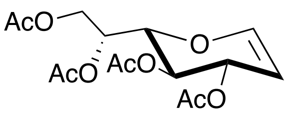

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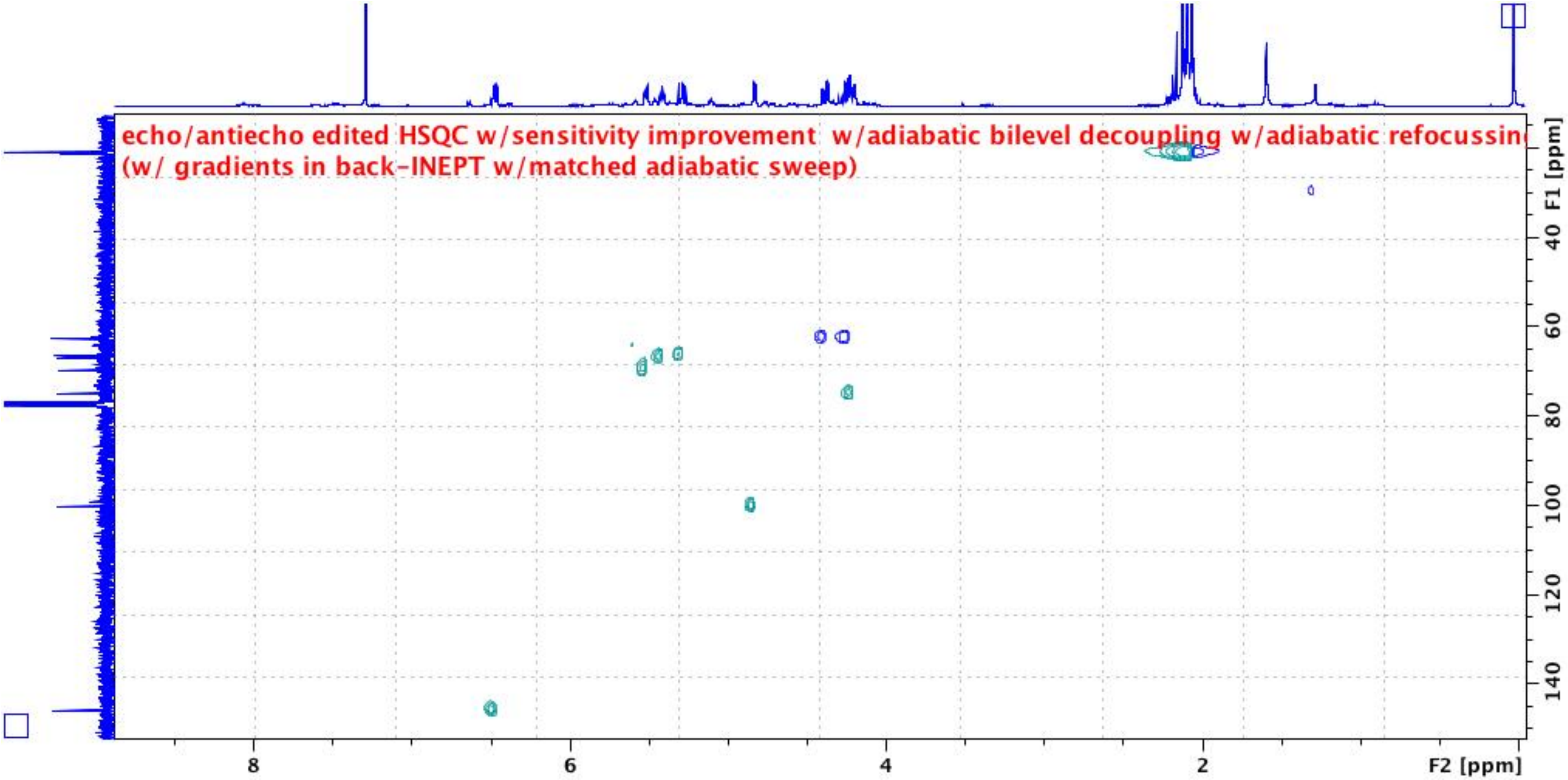
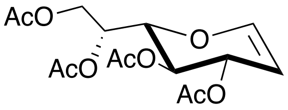
HSQC $CDCl_3$ 500 MHZ





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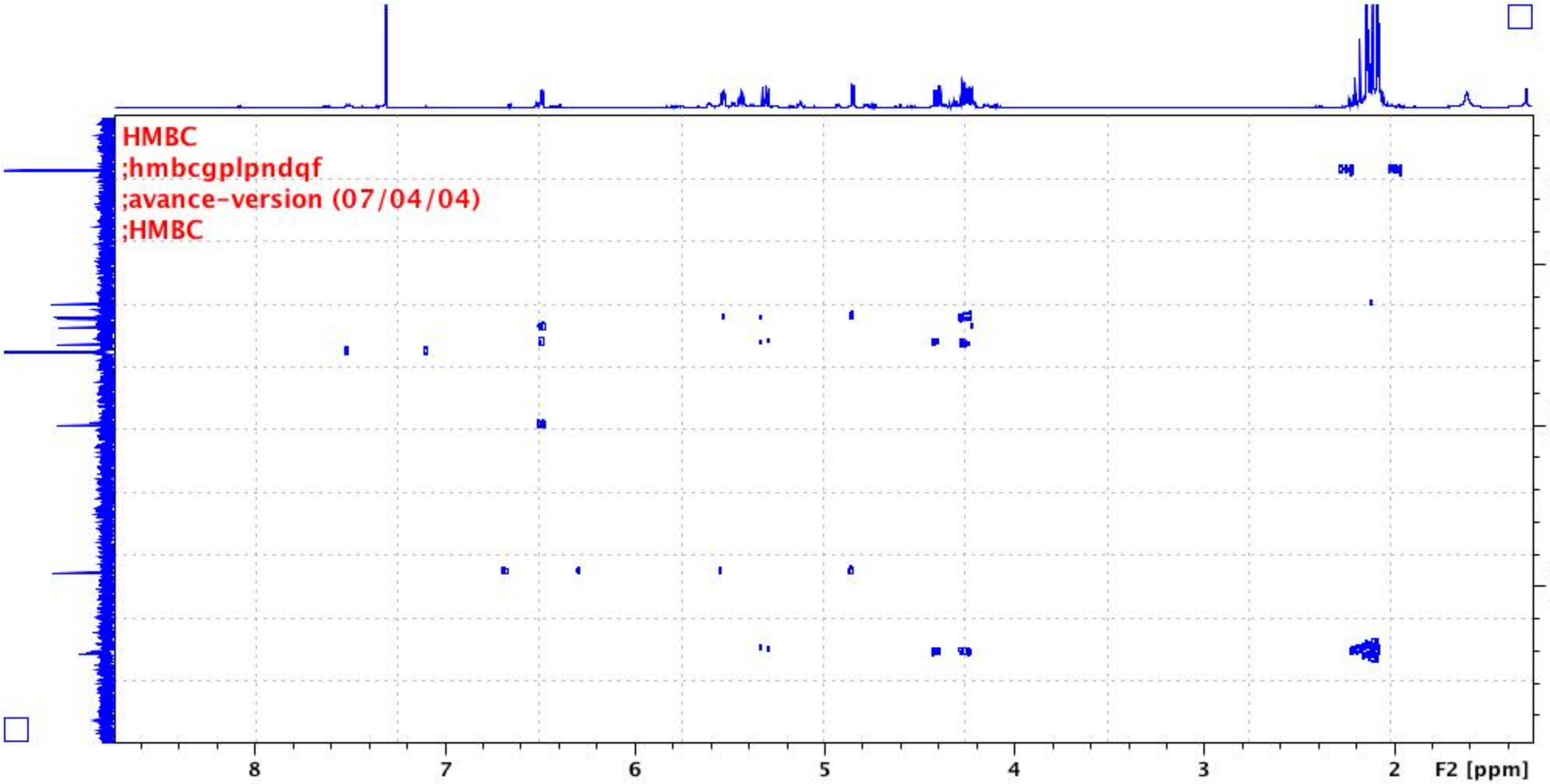

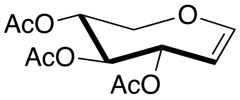

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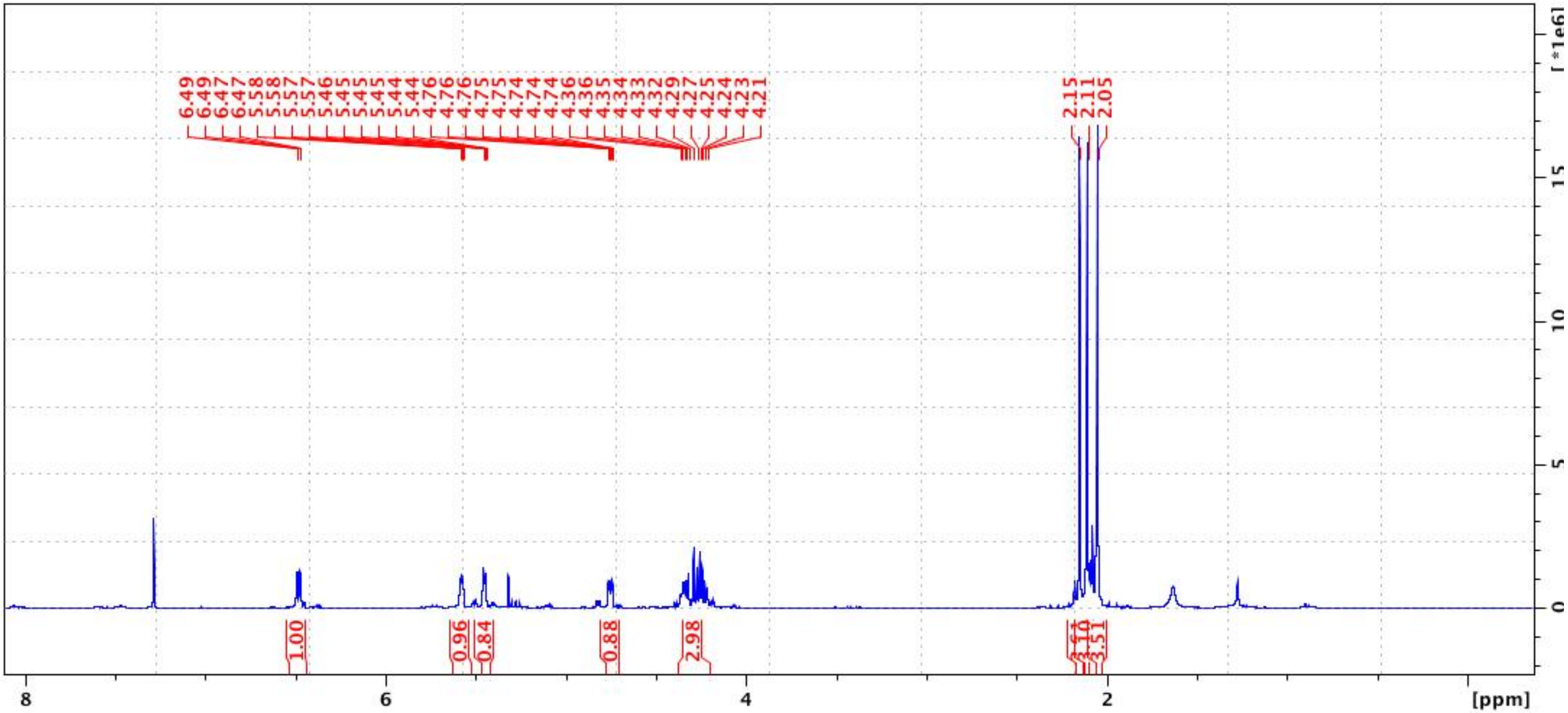

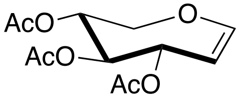

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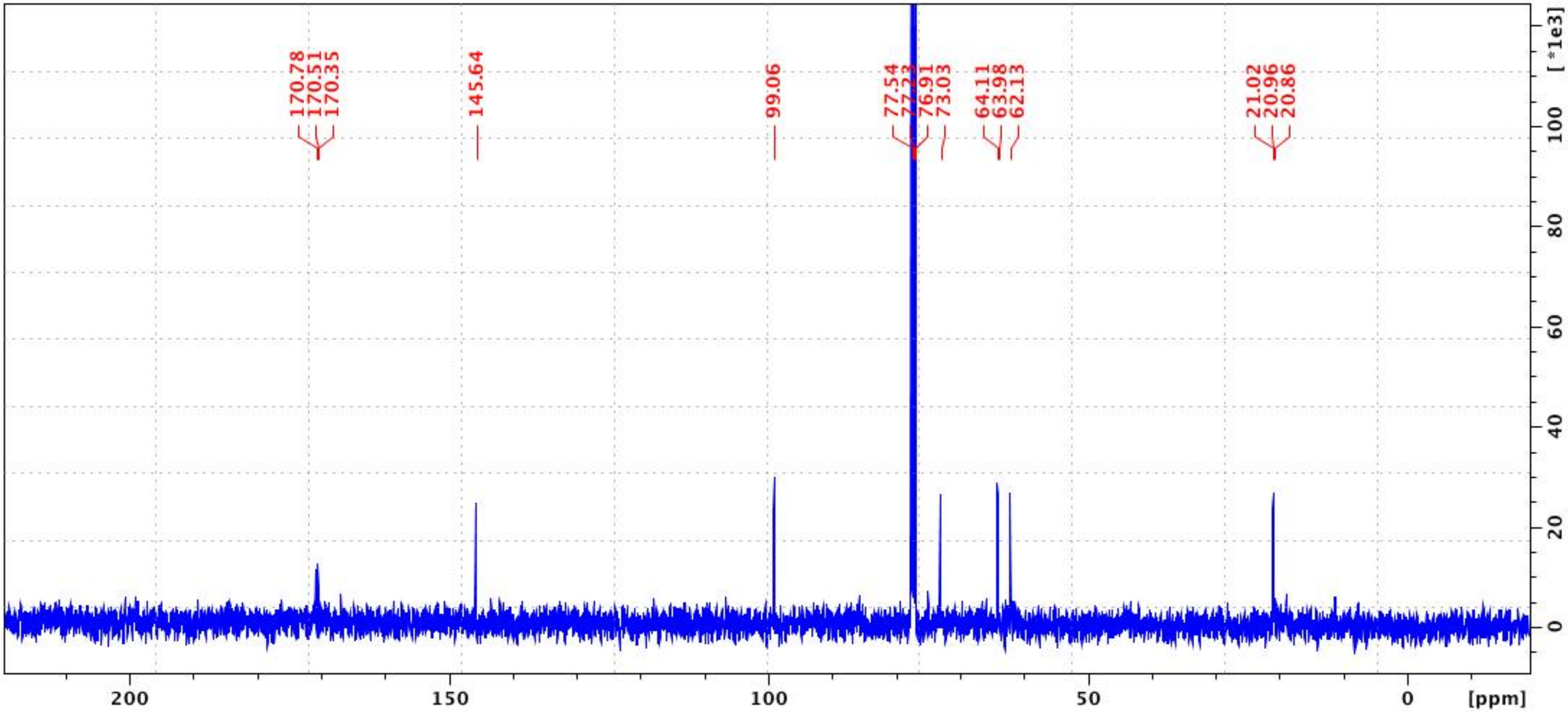

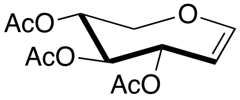

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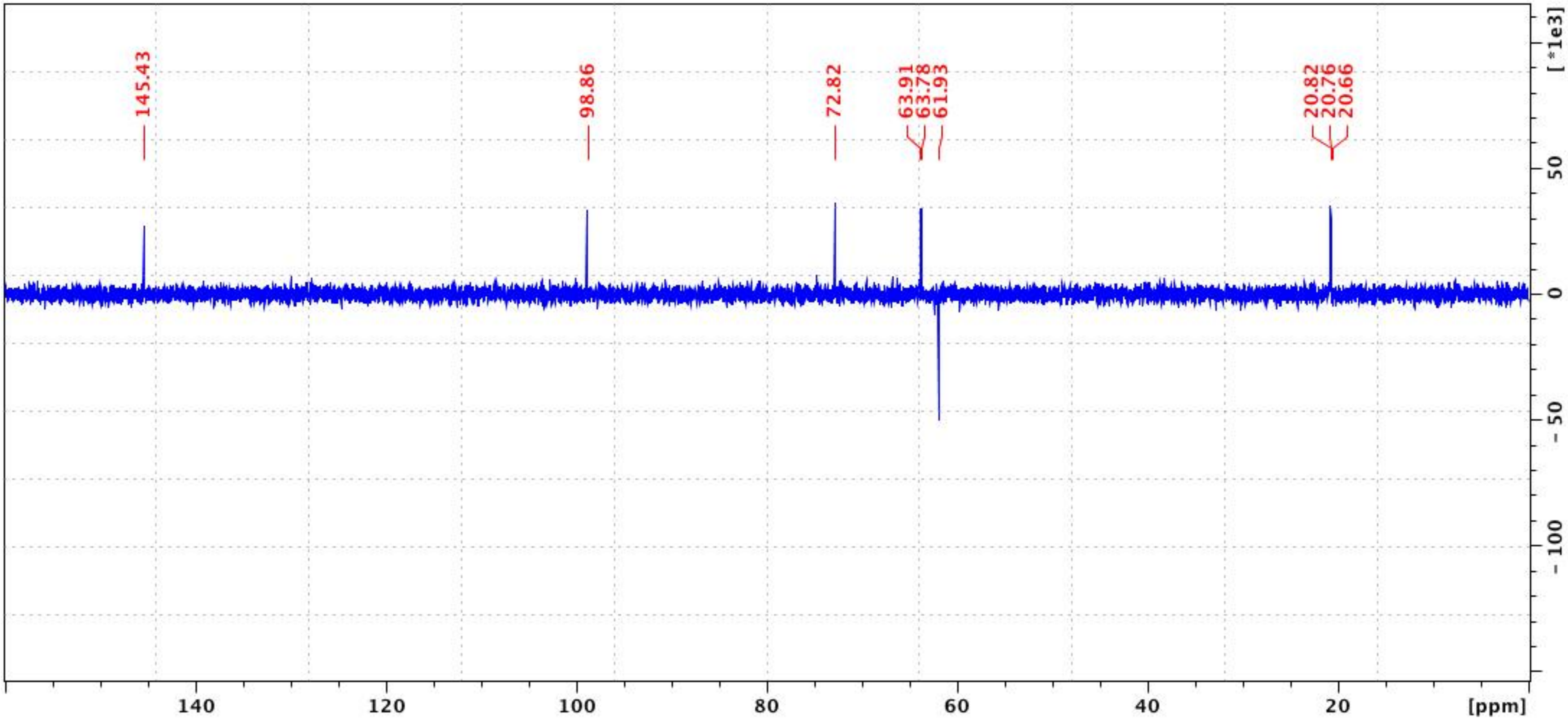

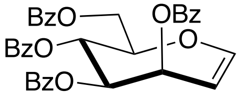

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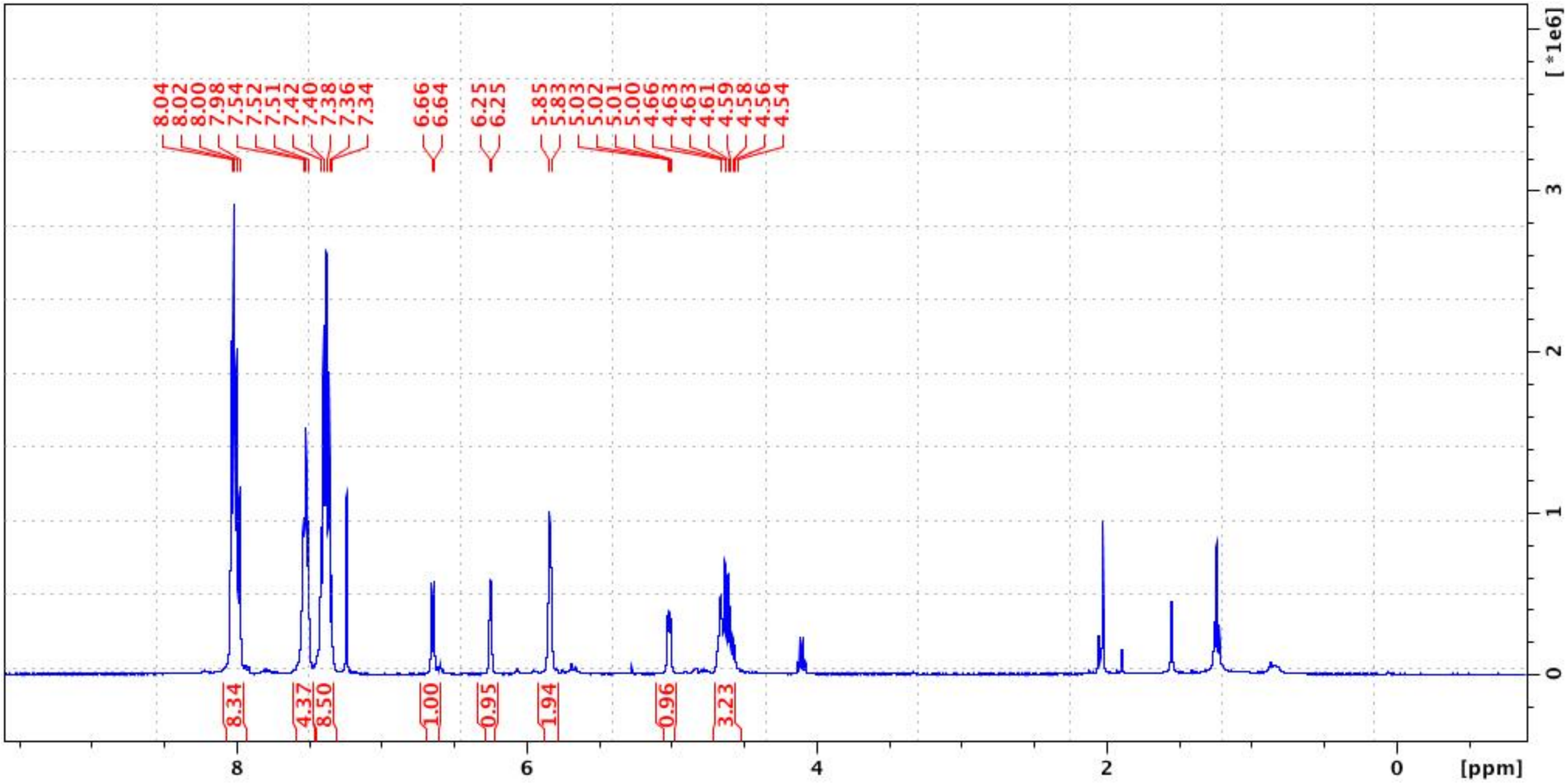

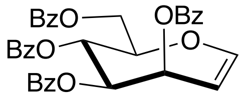

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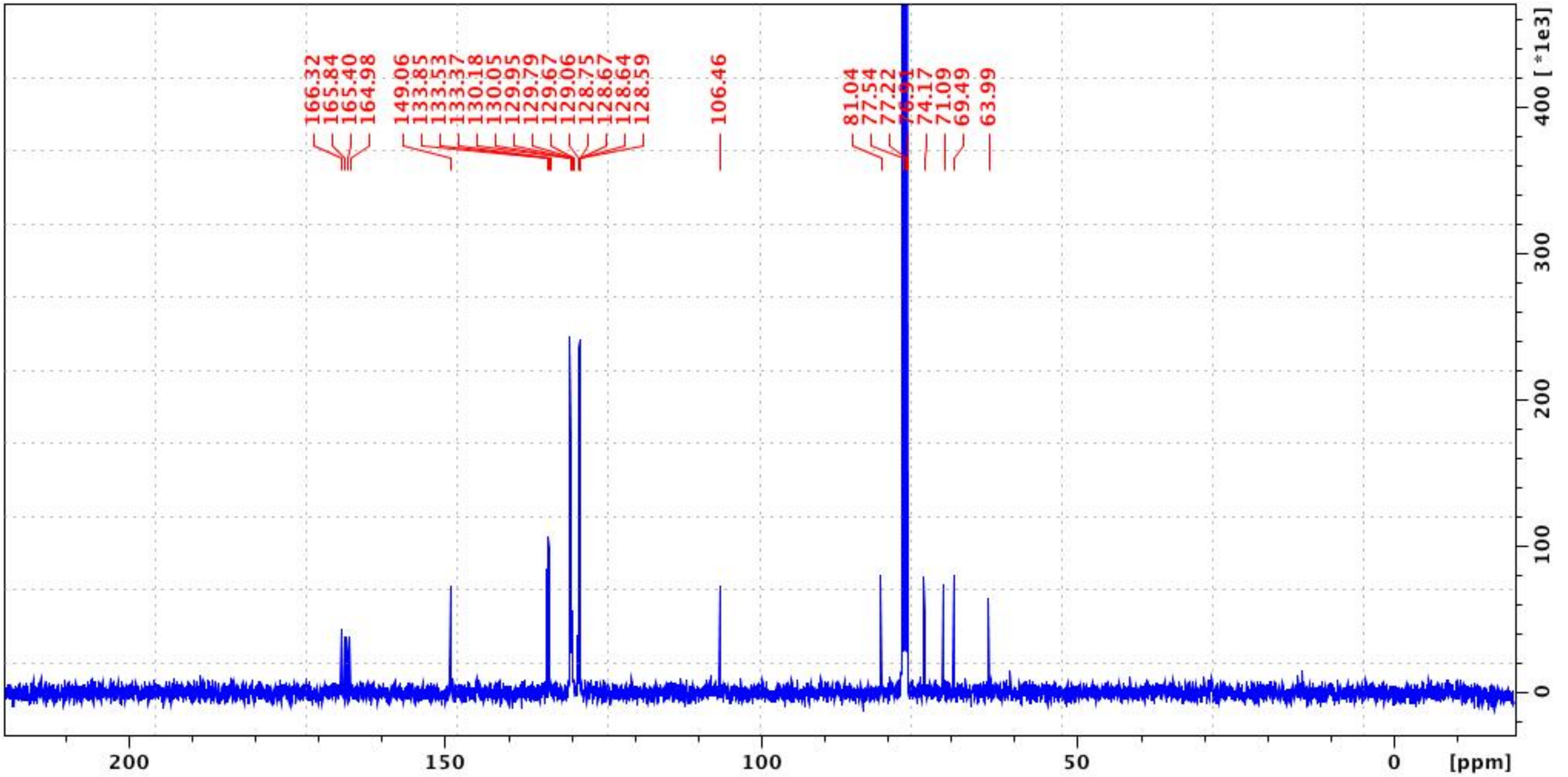

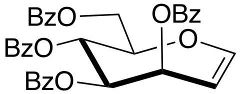

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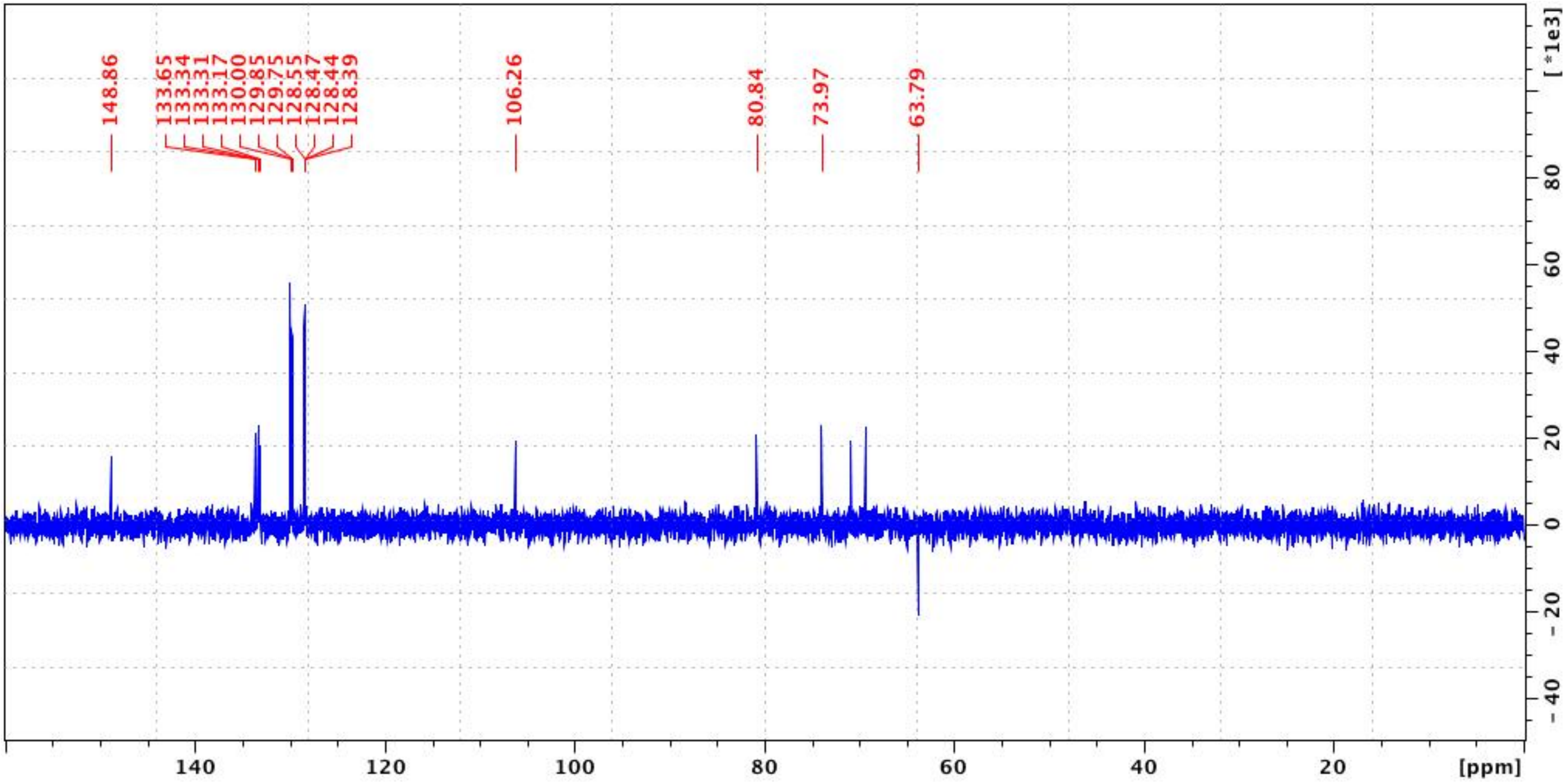

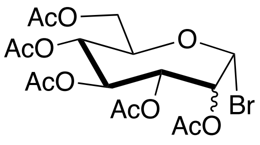

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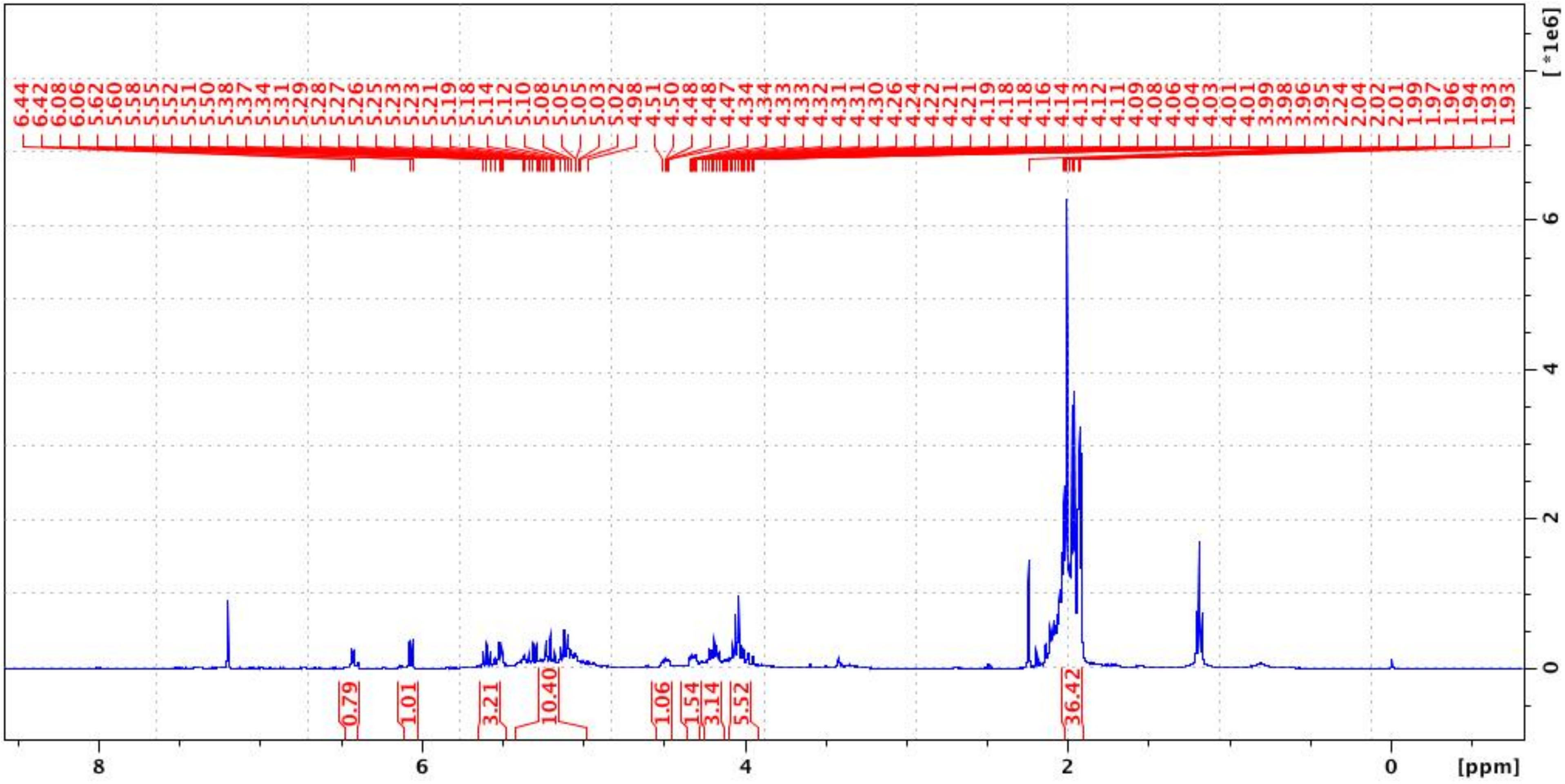

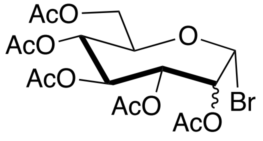

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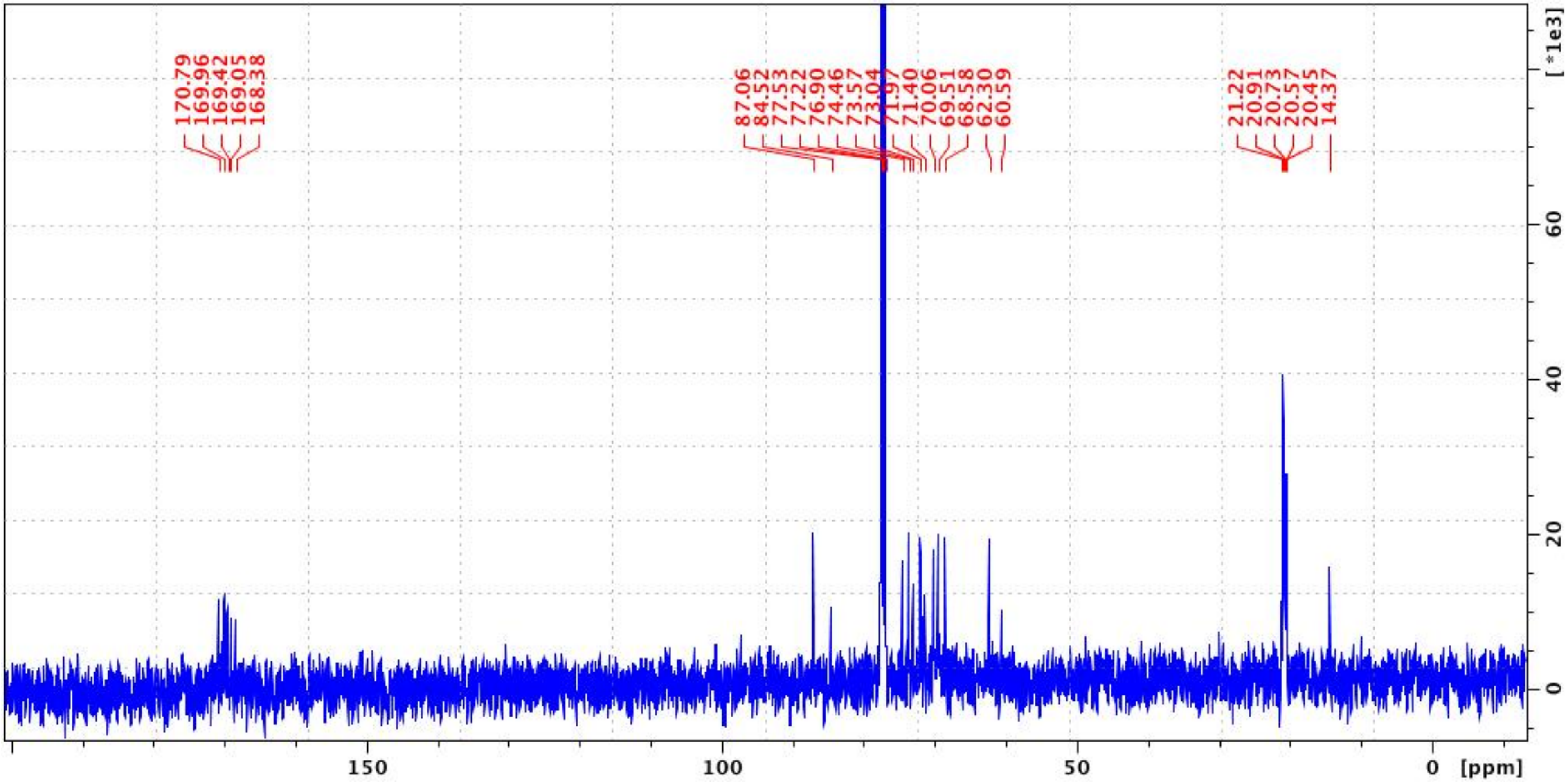

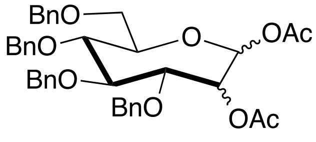

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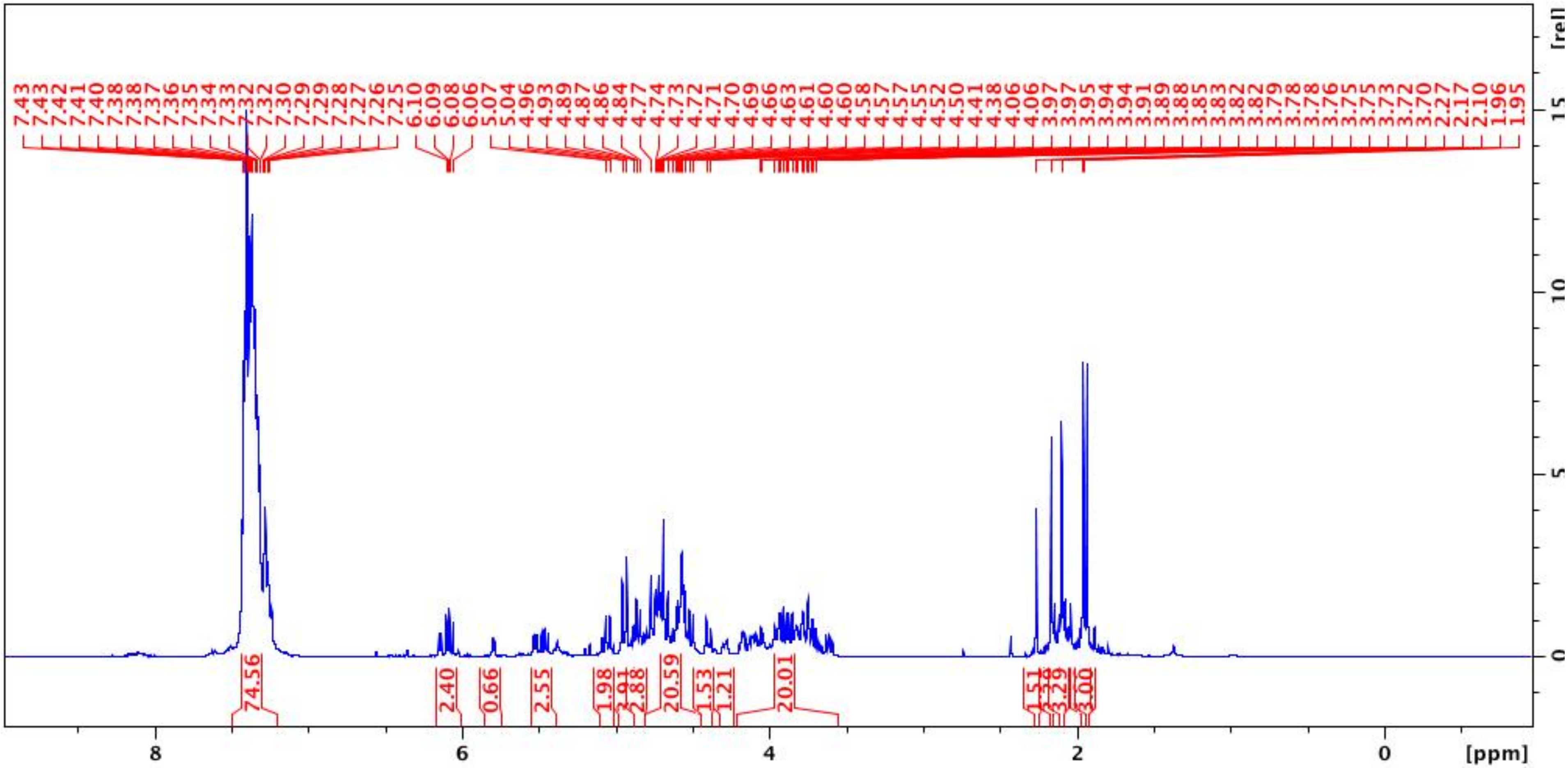
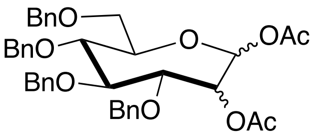
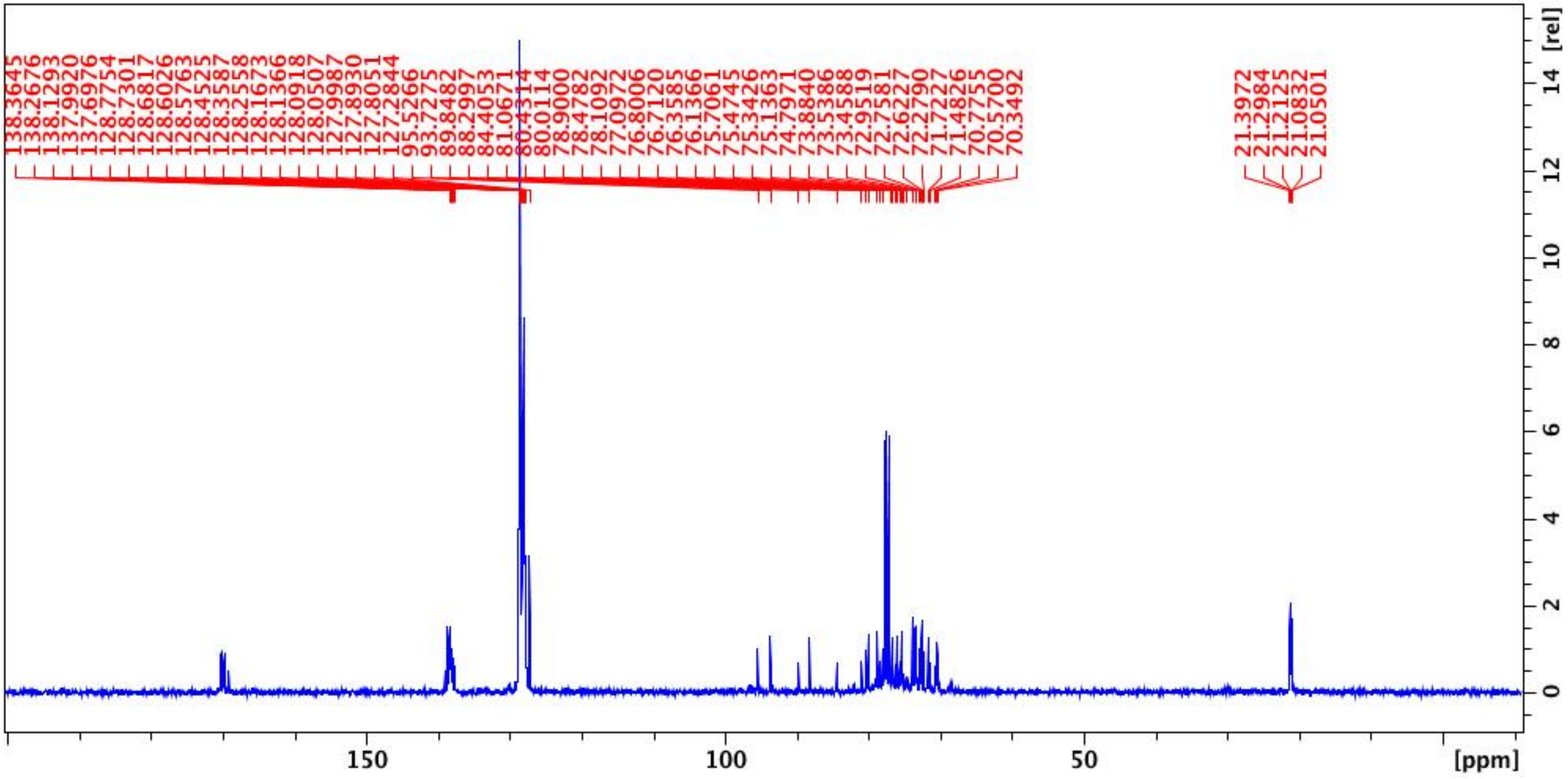
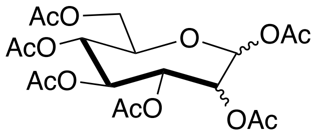




9

^{13}C CDCl$_3$ 400 MHZ





4

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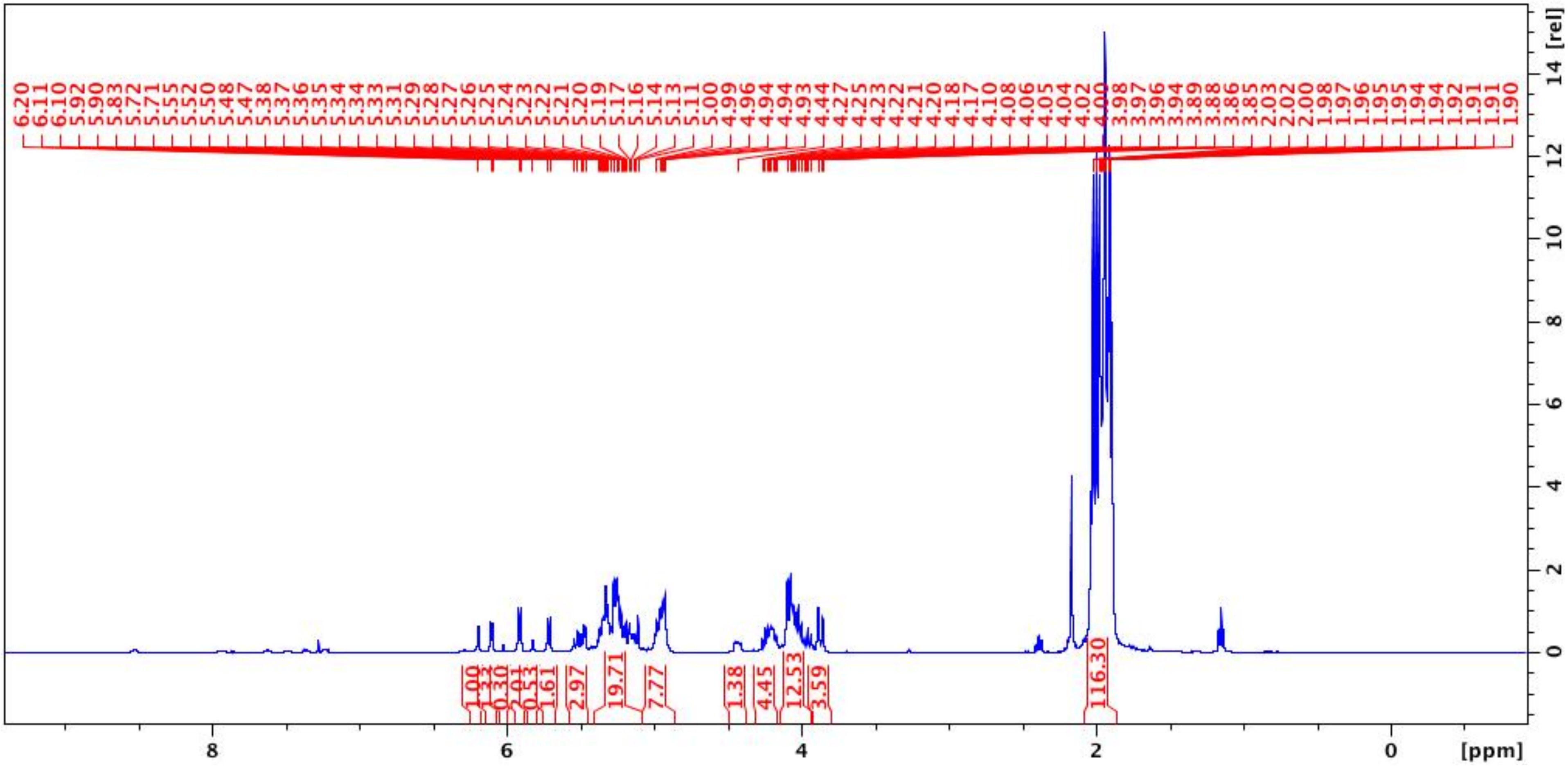

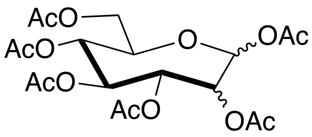

4

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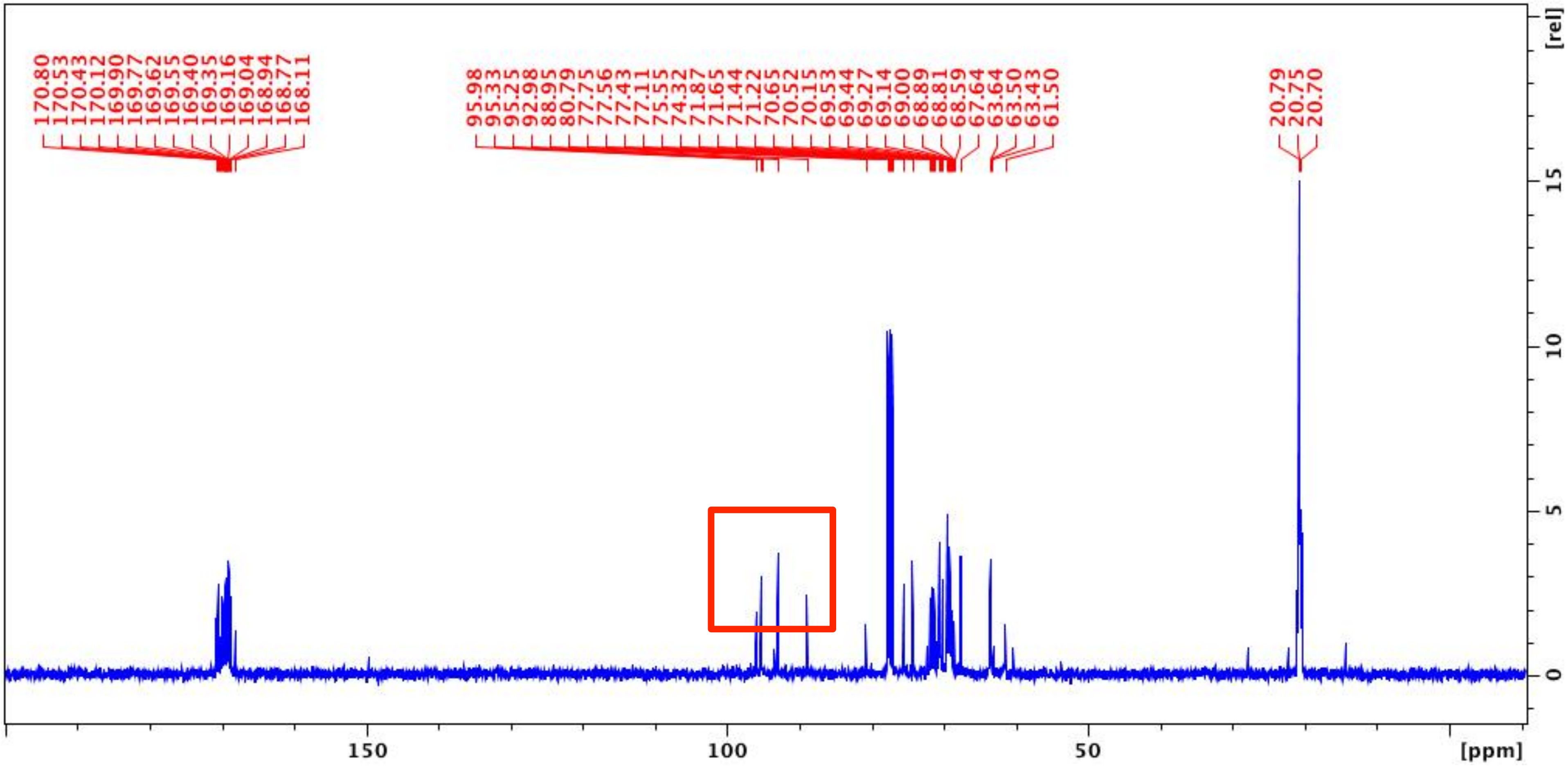

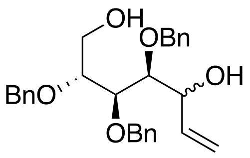

11

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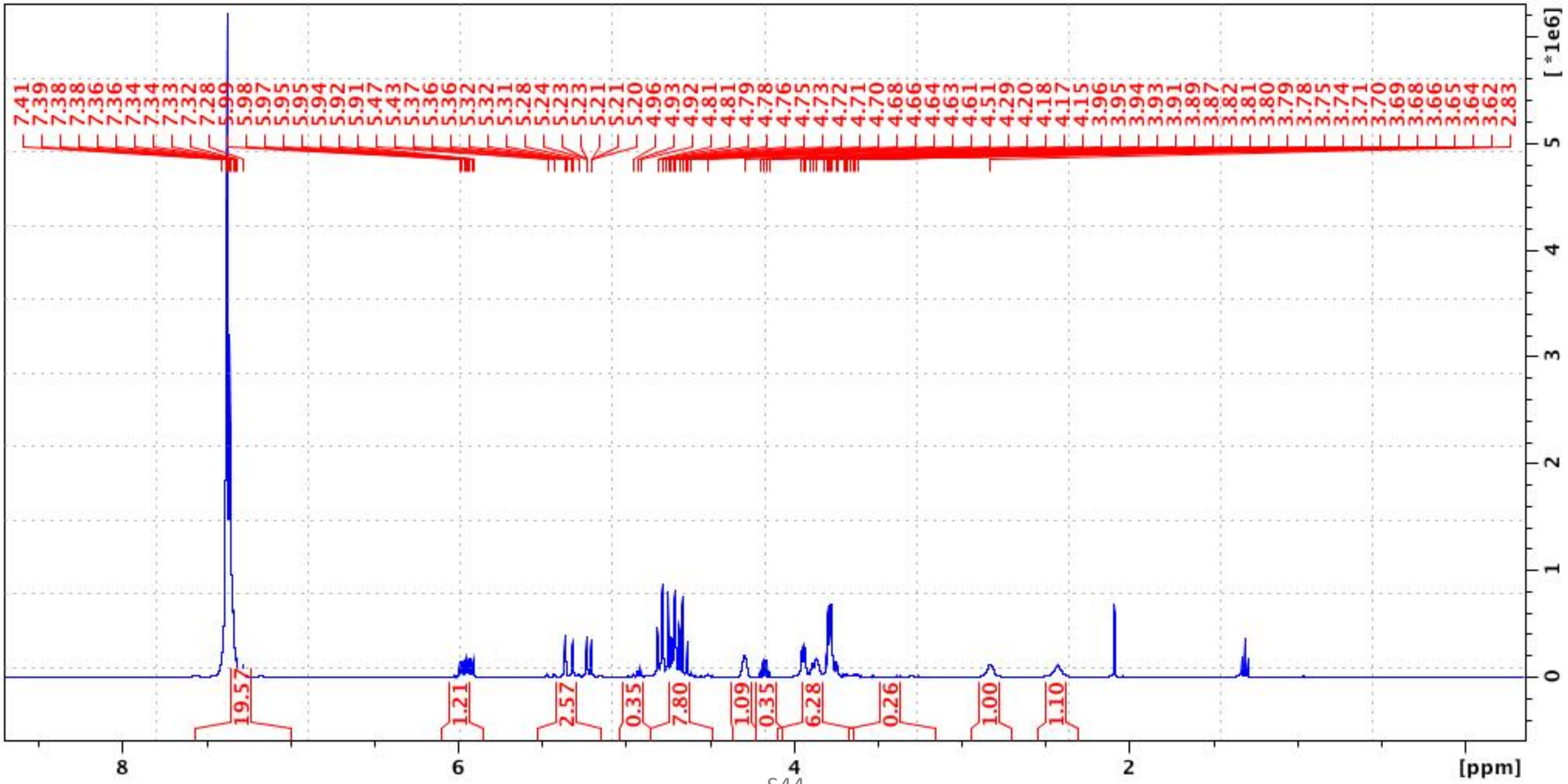

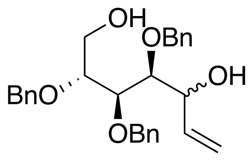

11

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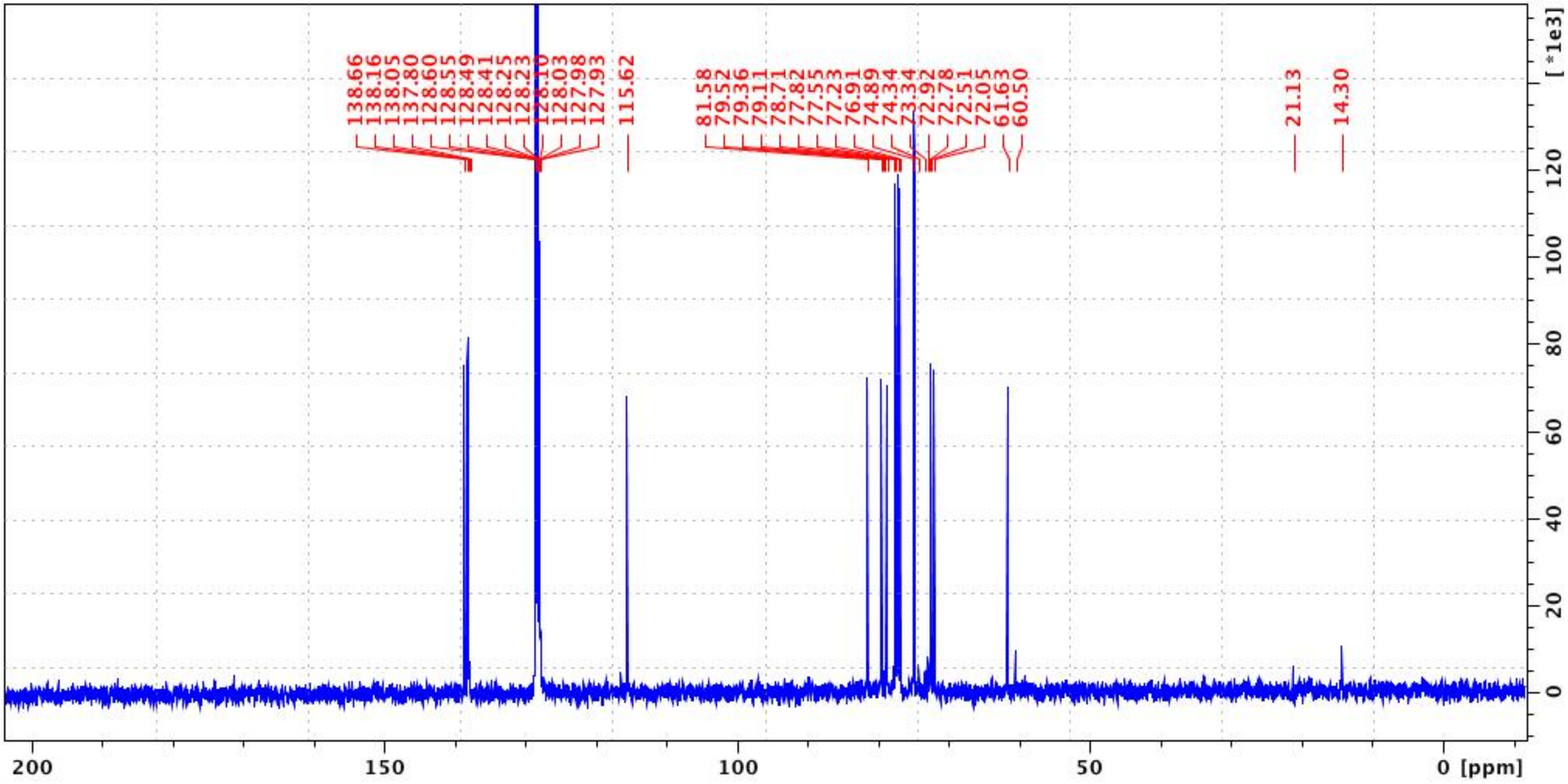

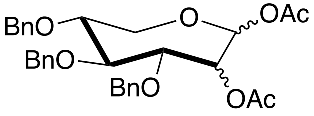

12

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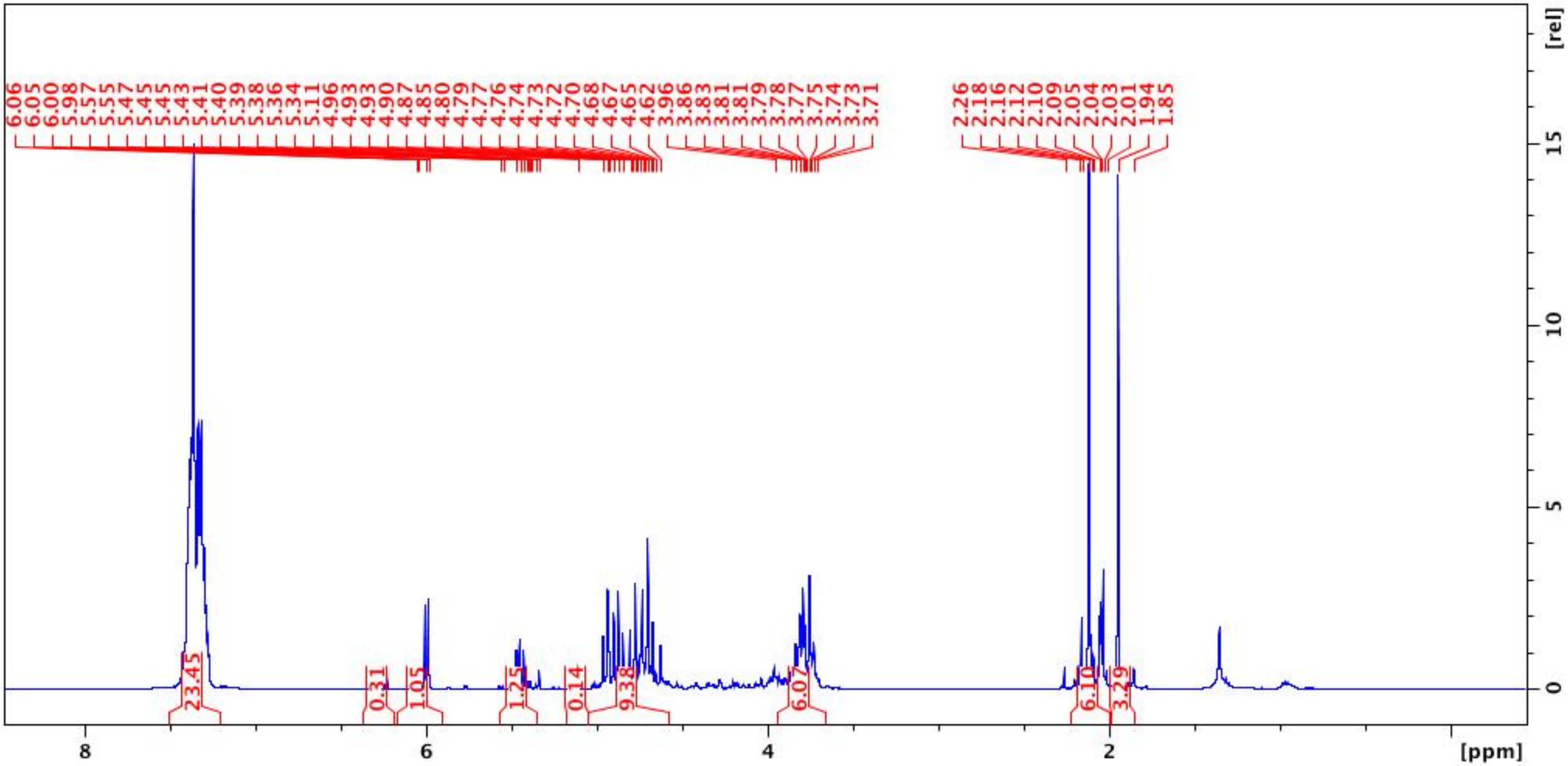

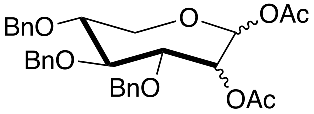

12

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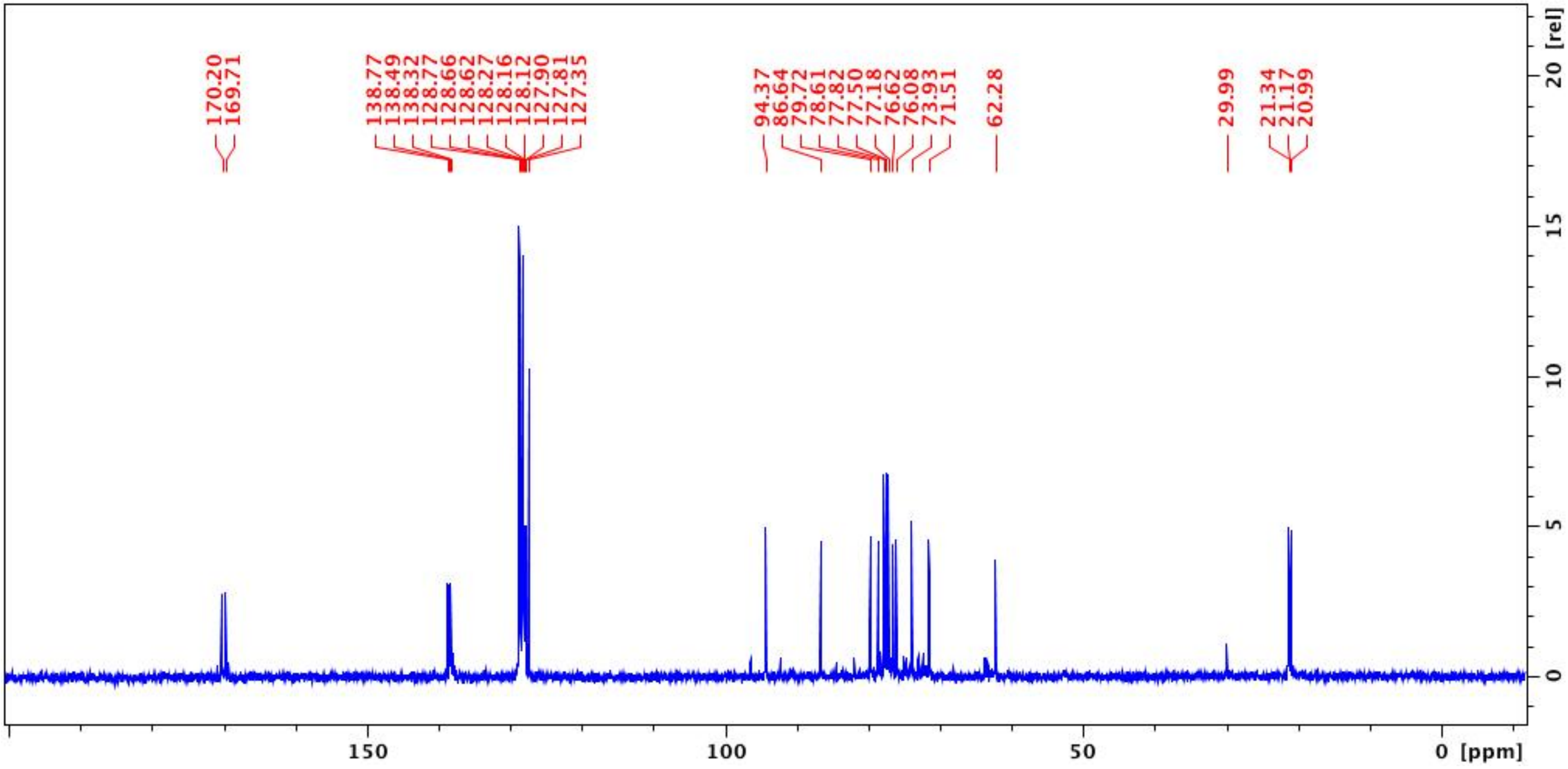

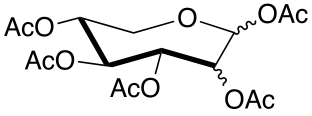

13

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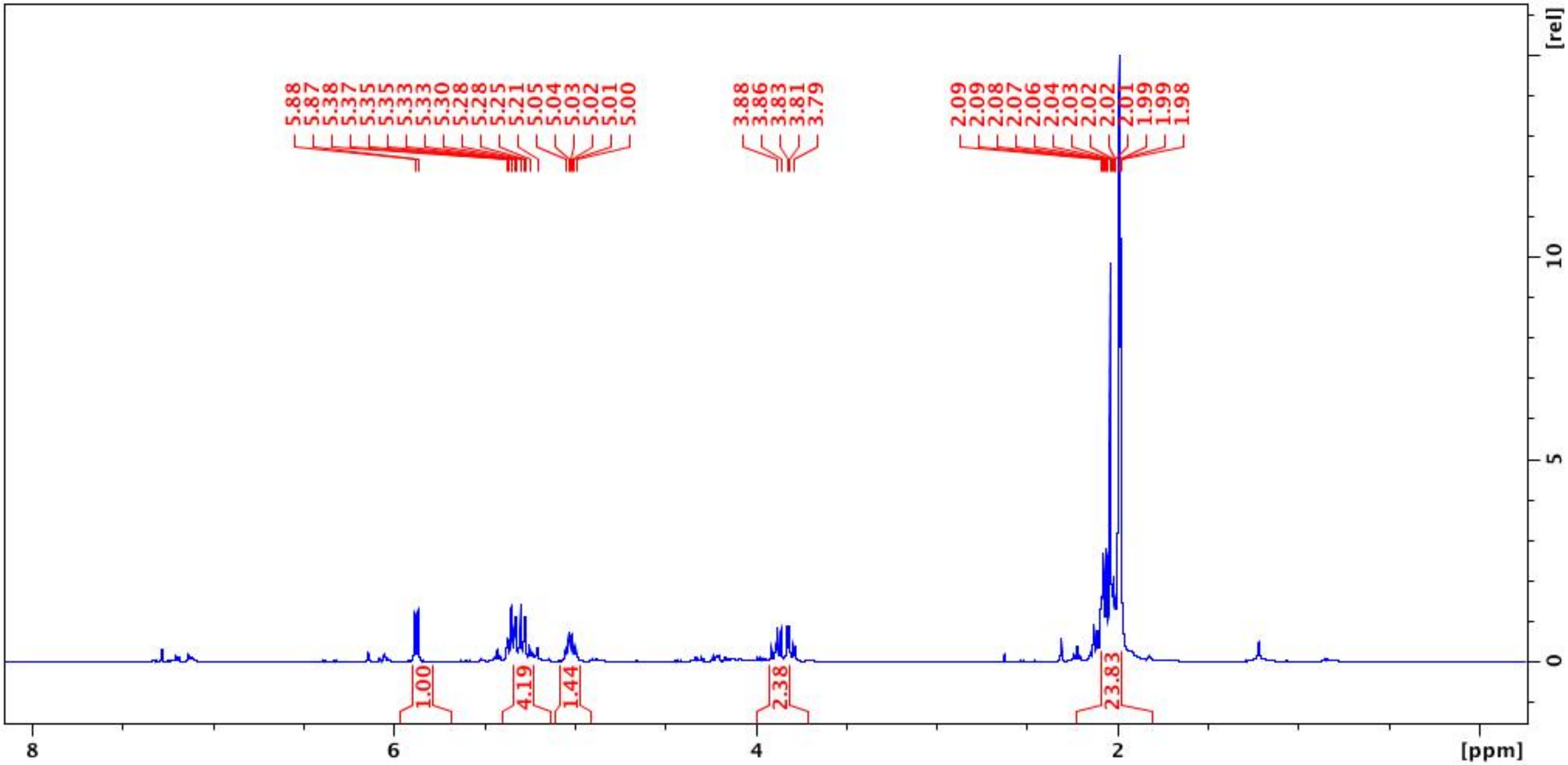

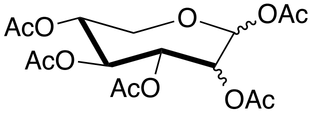

13

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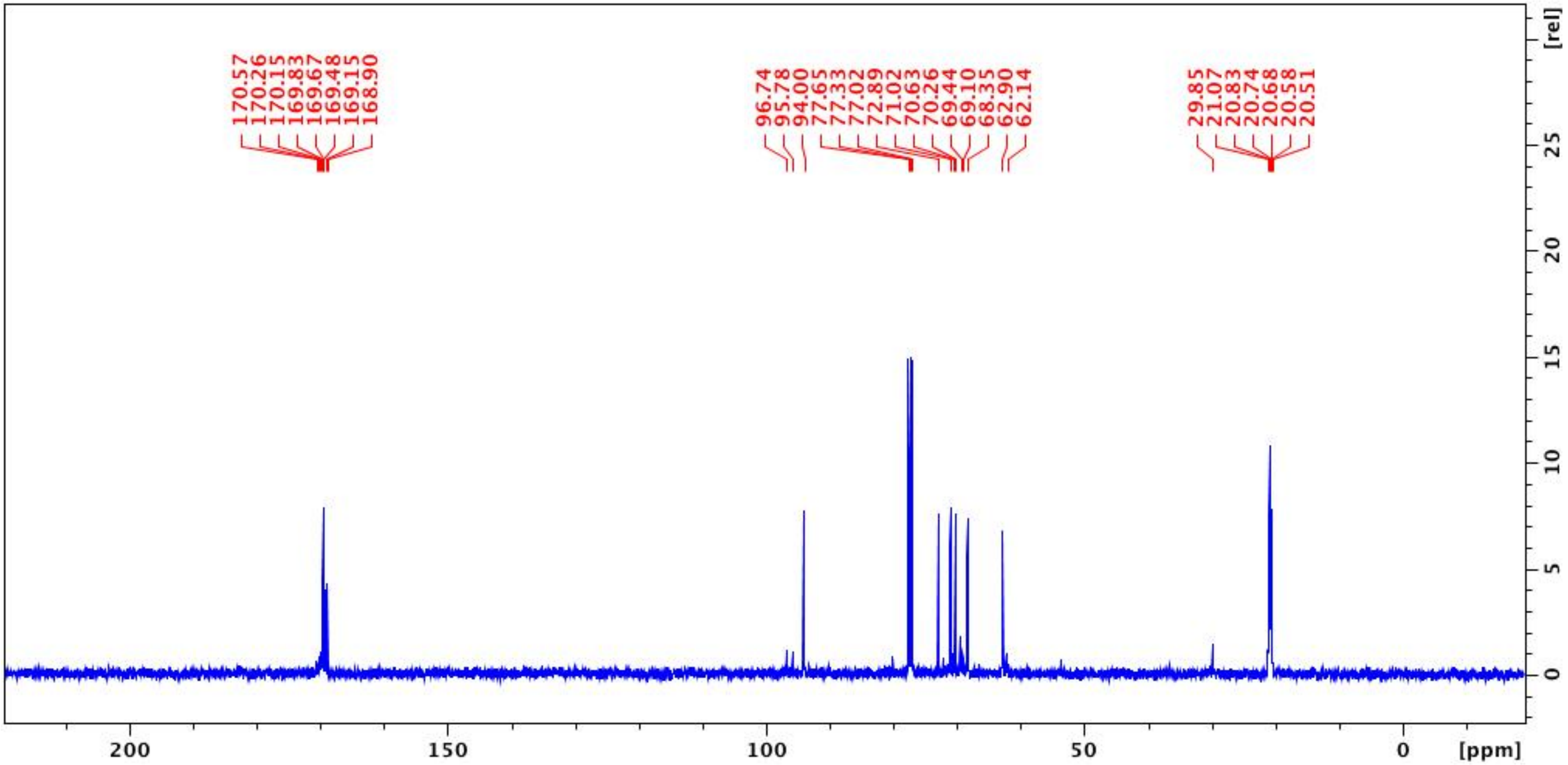

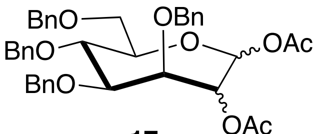

17

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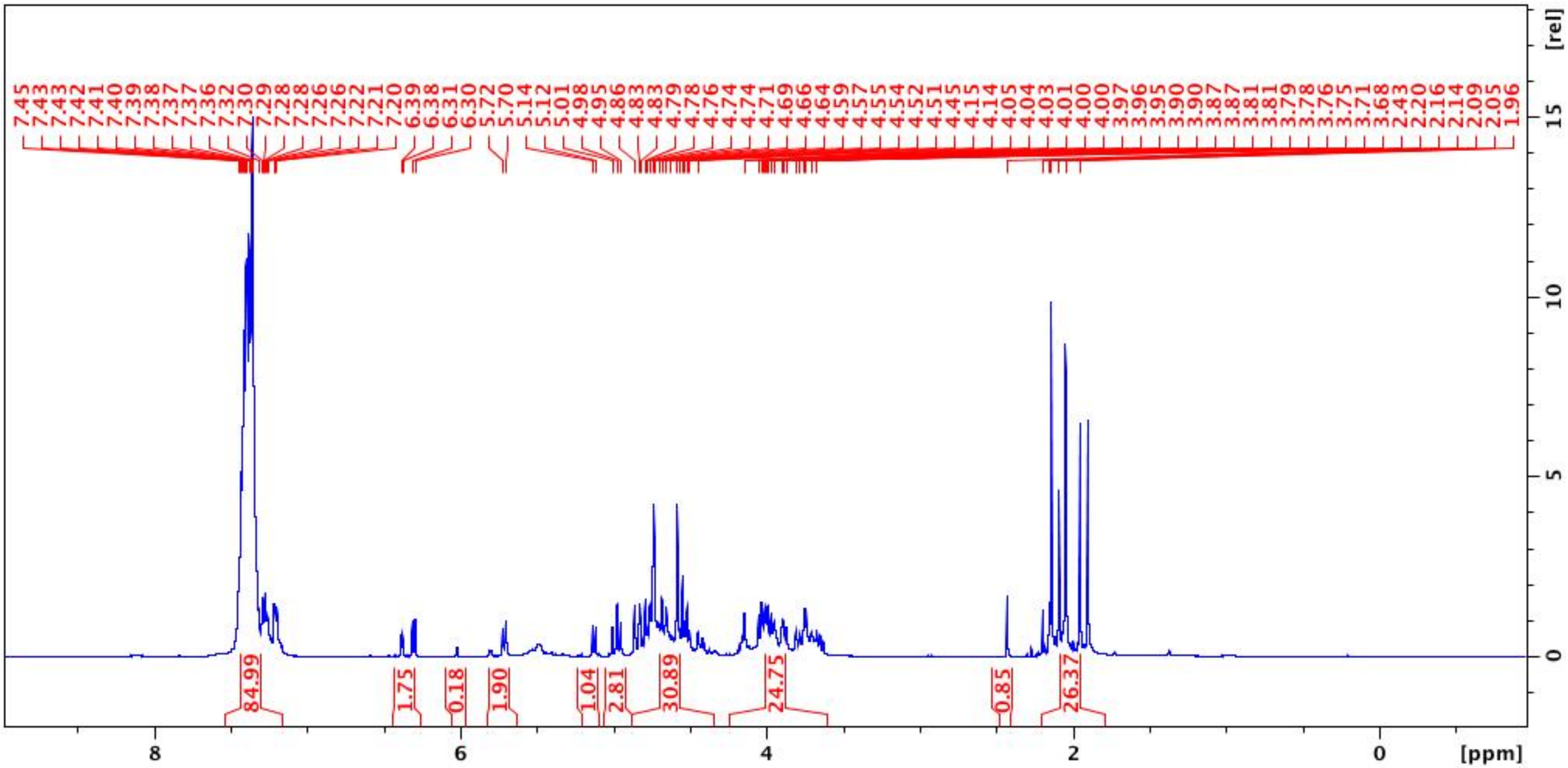

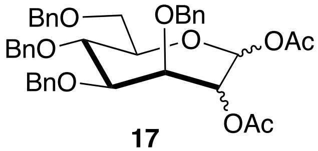

17

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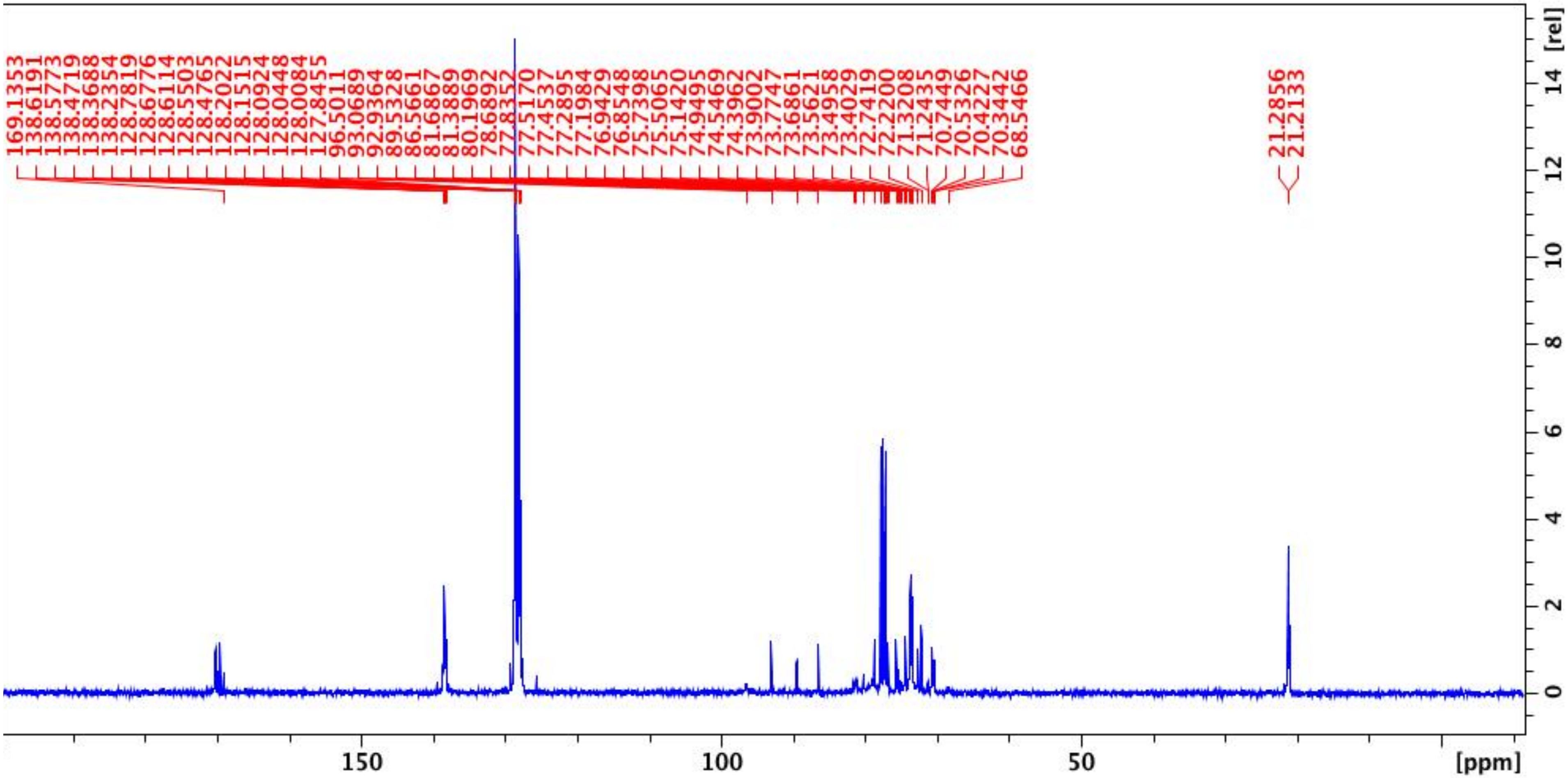

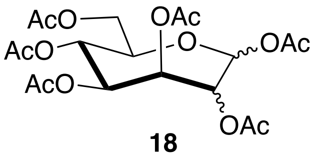

18

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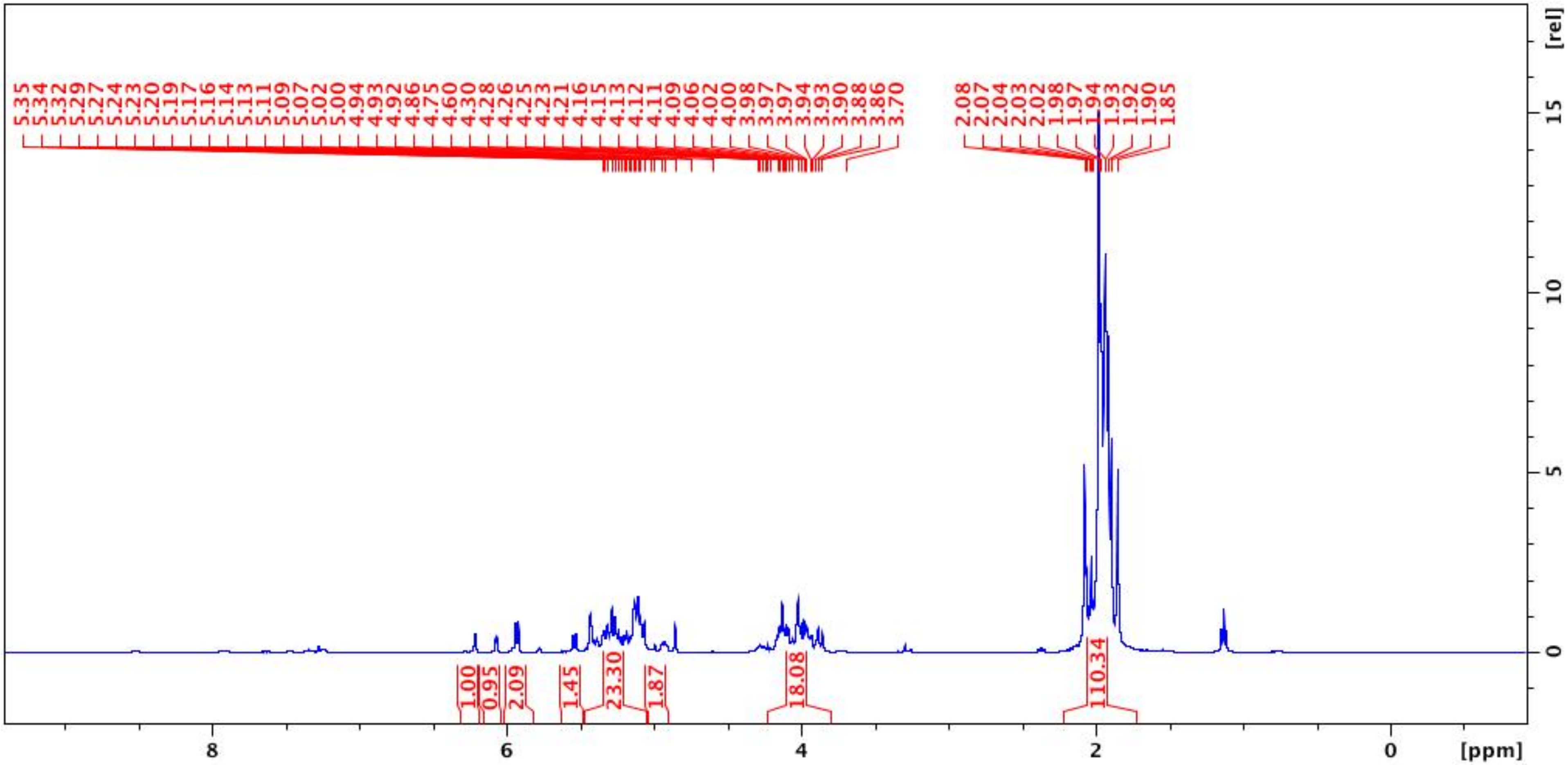

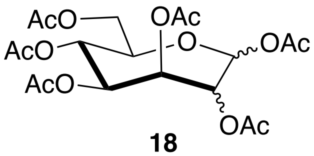

18

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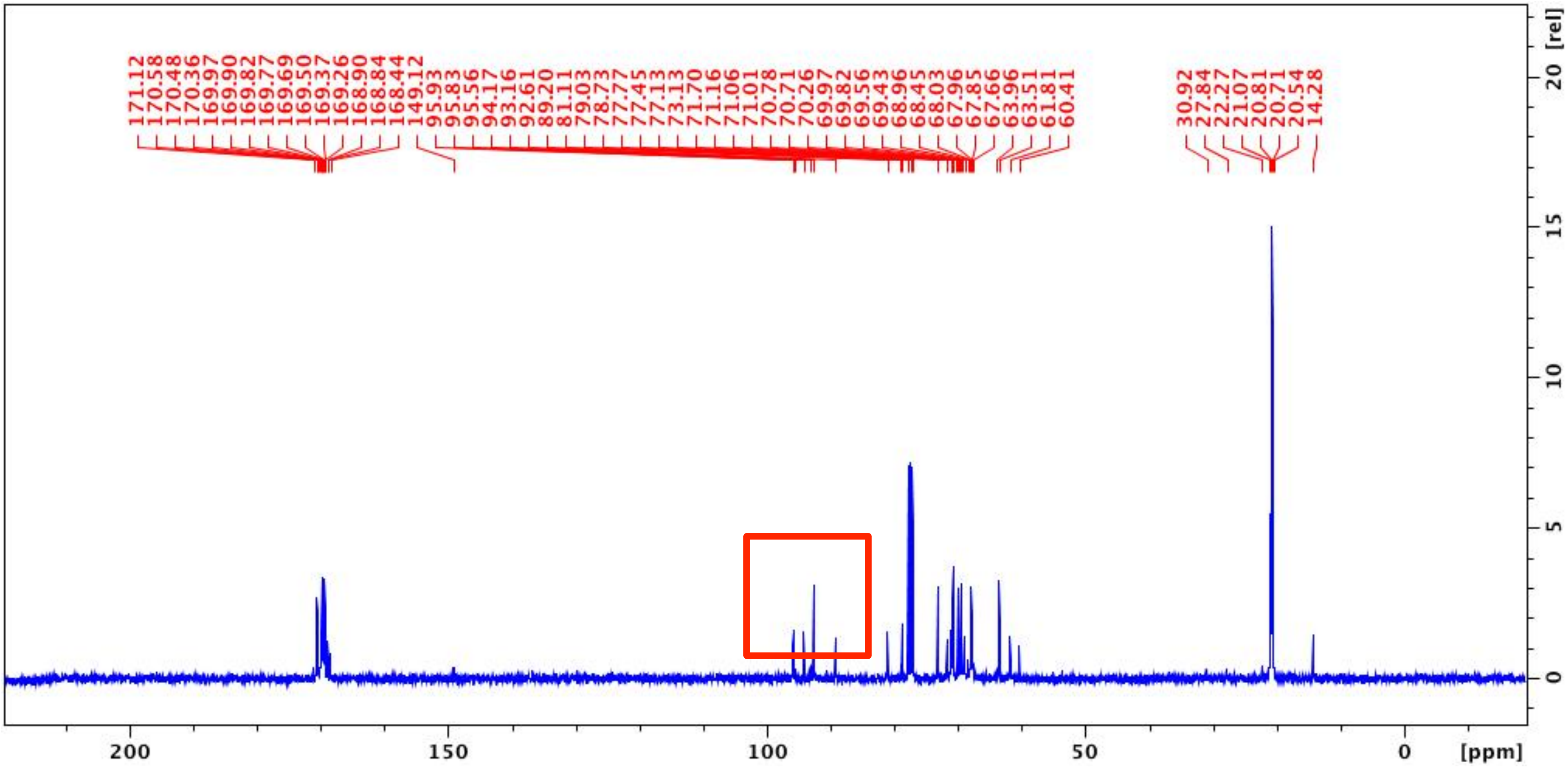

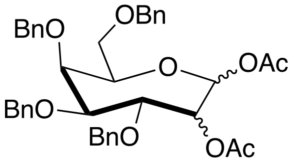

22

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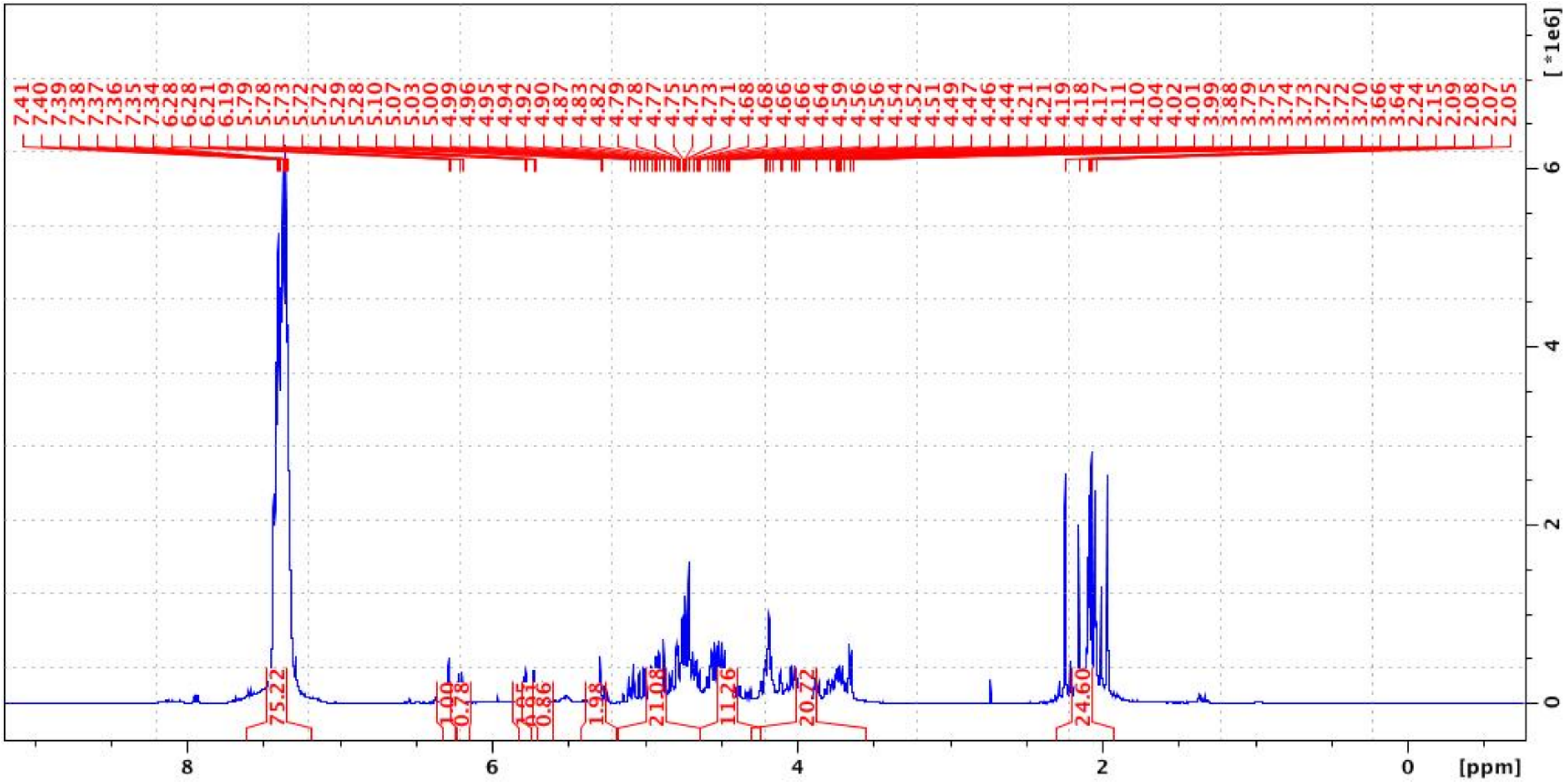

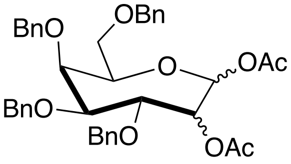

22

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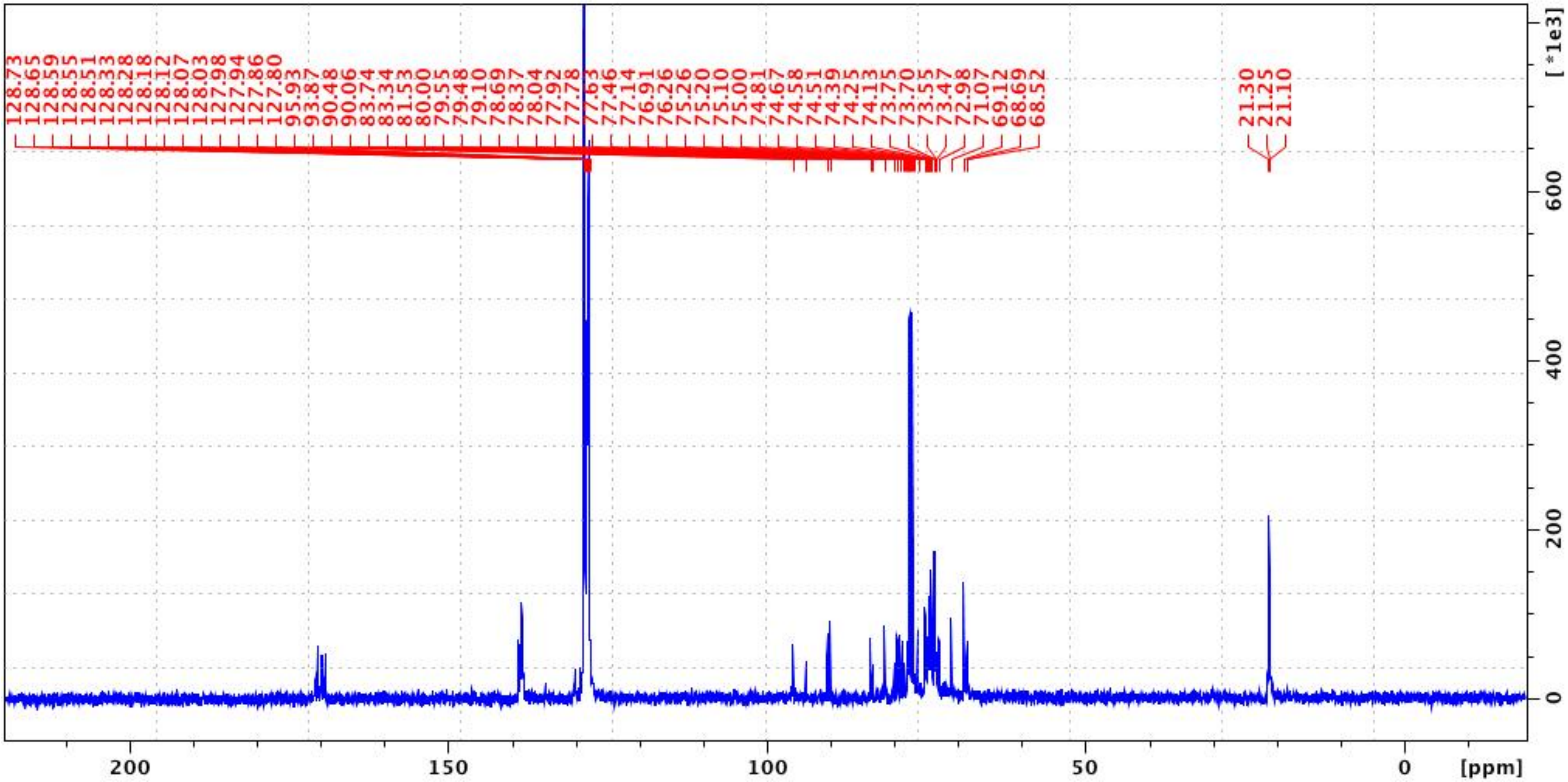

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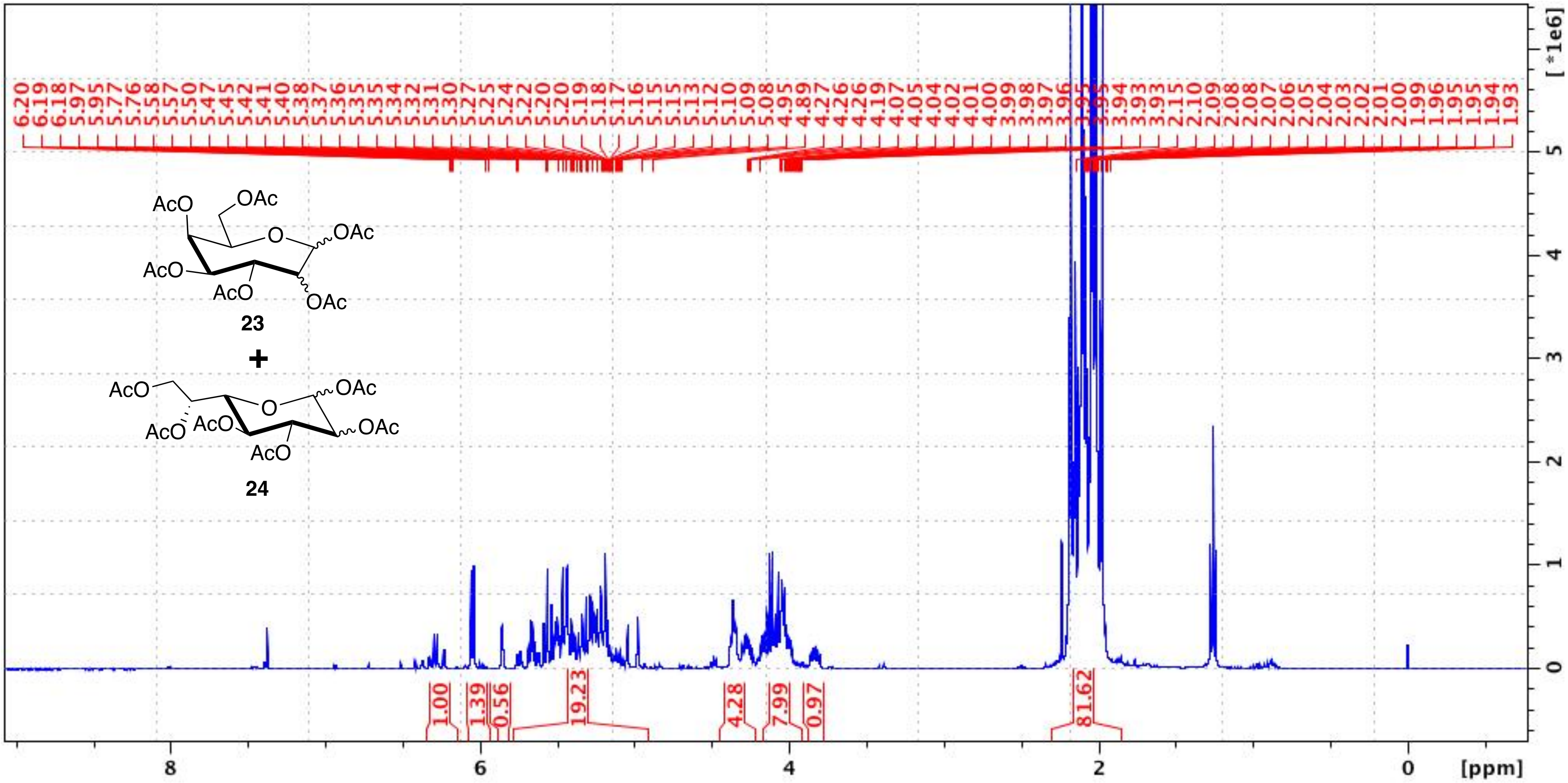

23

+

24

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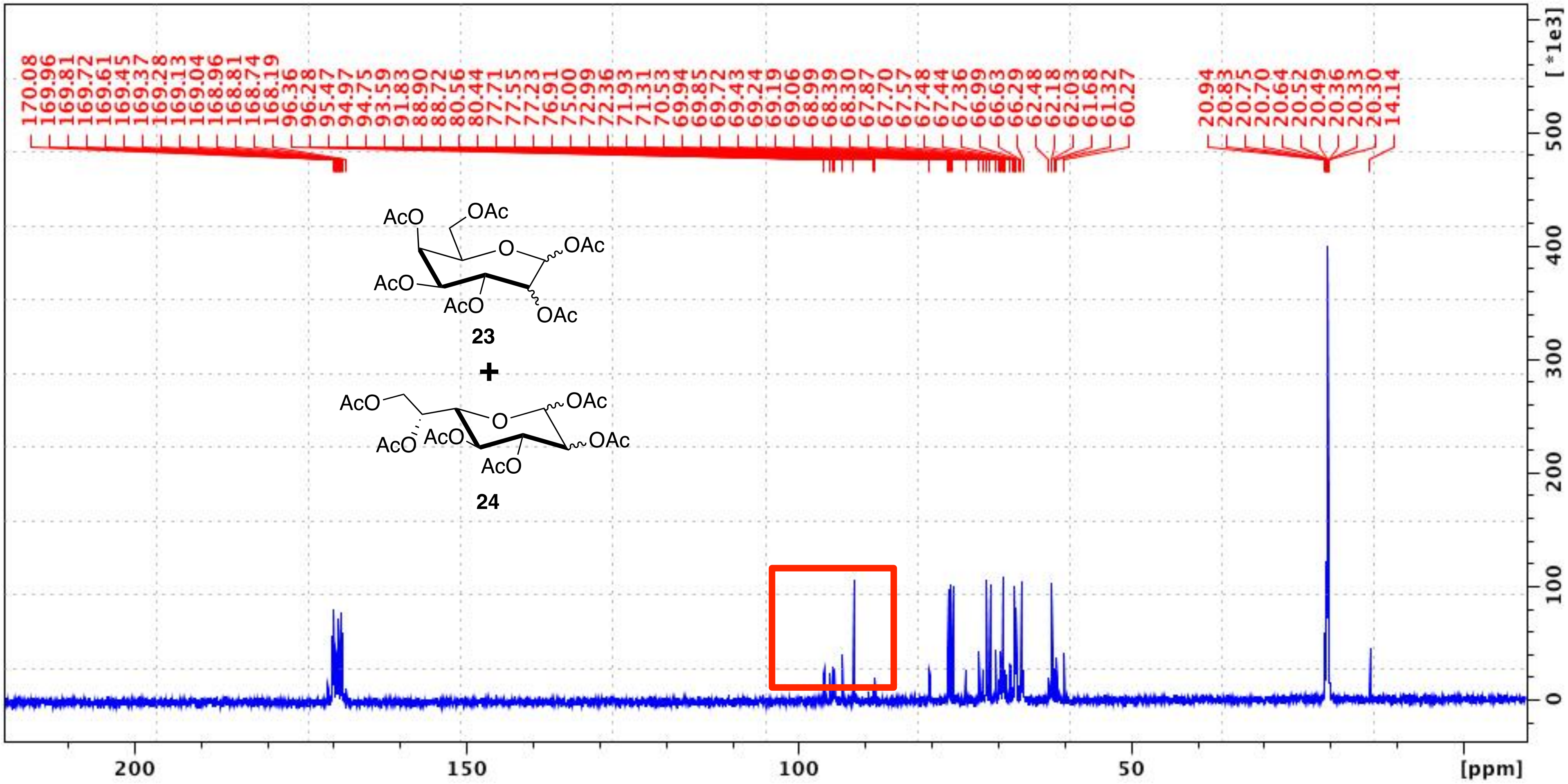

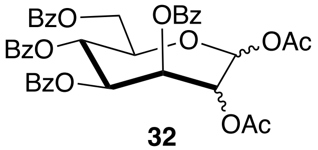

32

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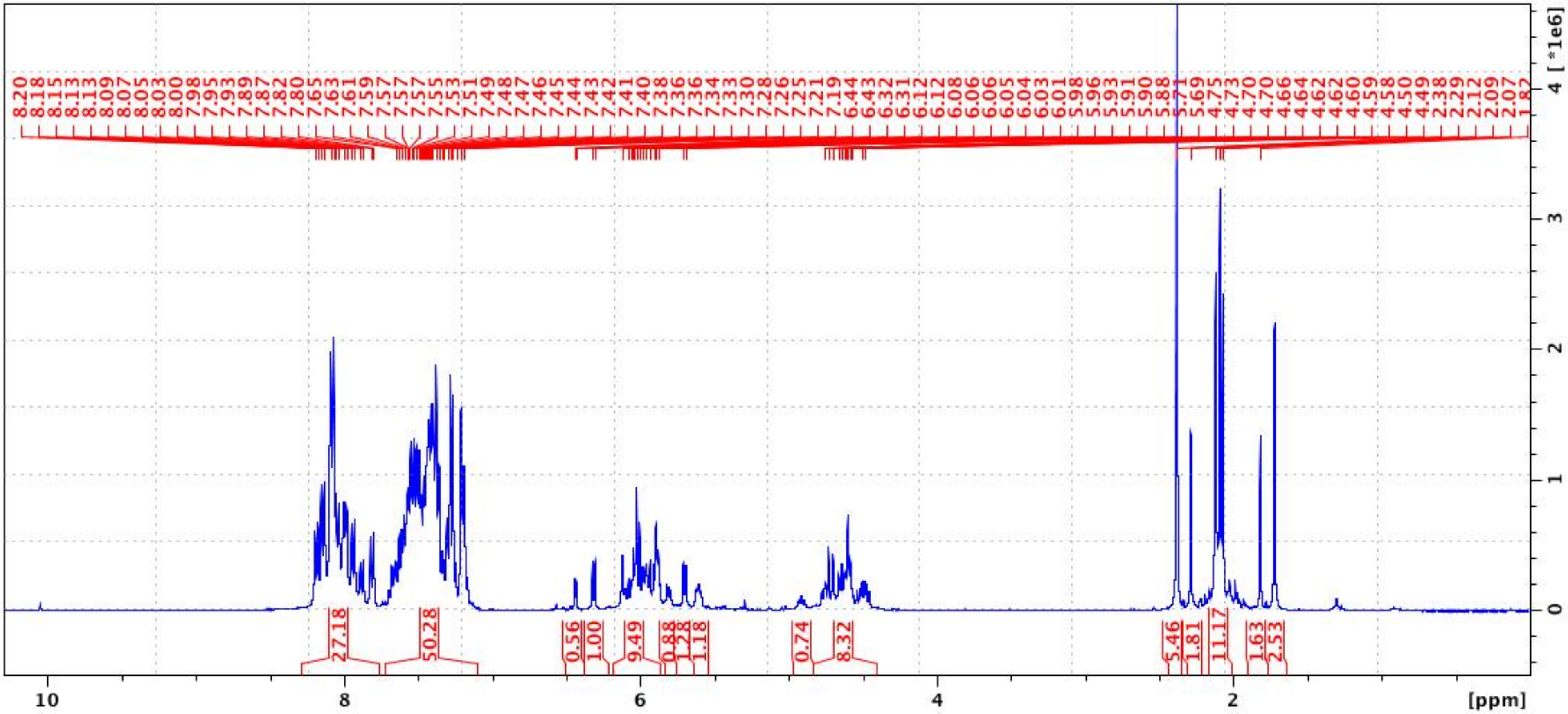

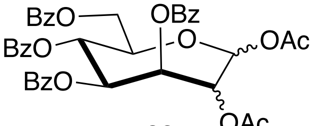

32

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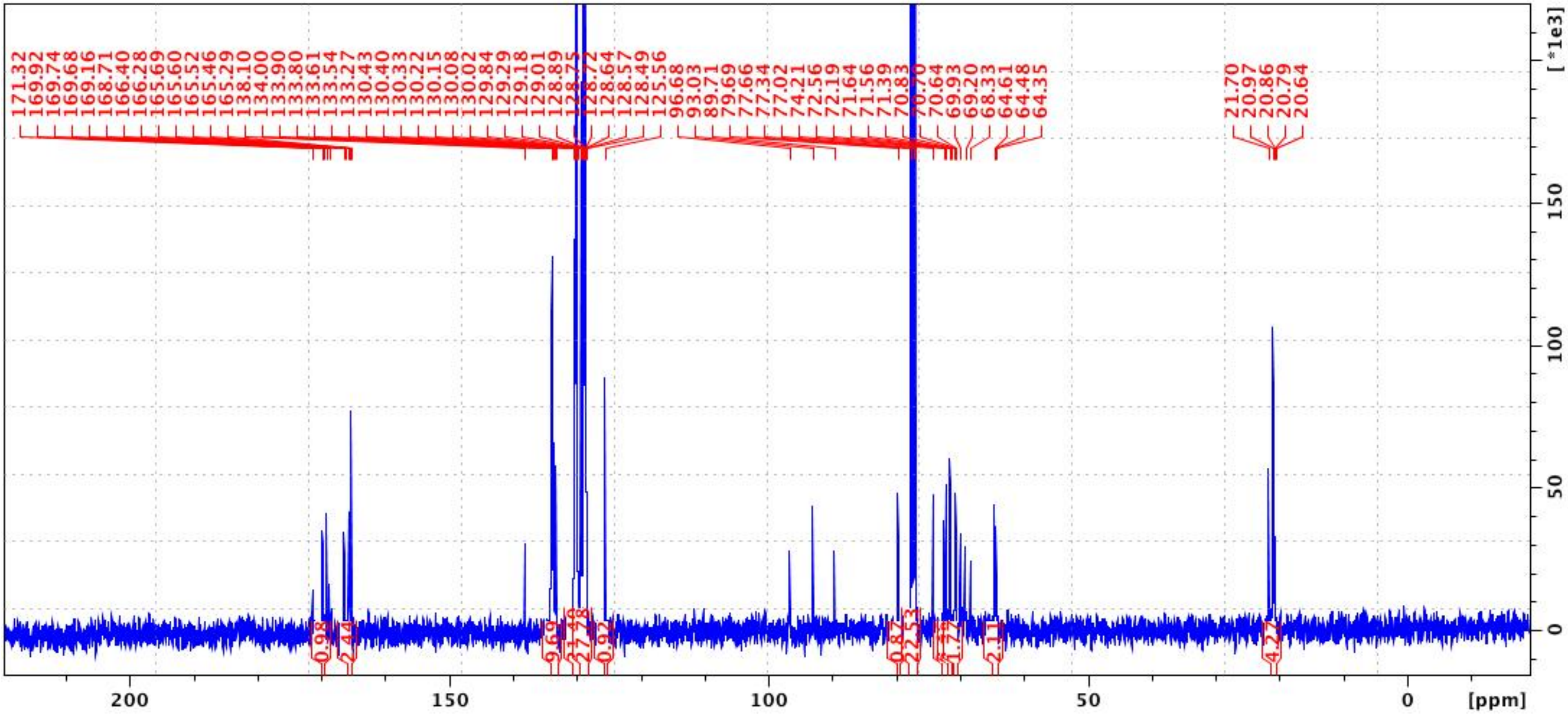

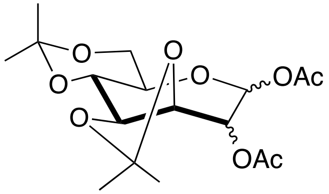

34

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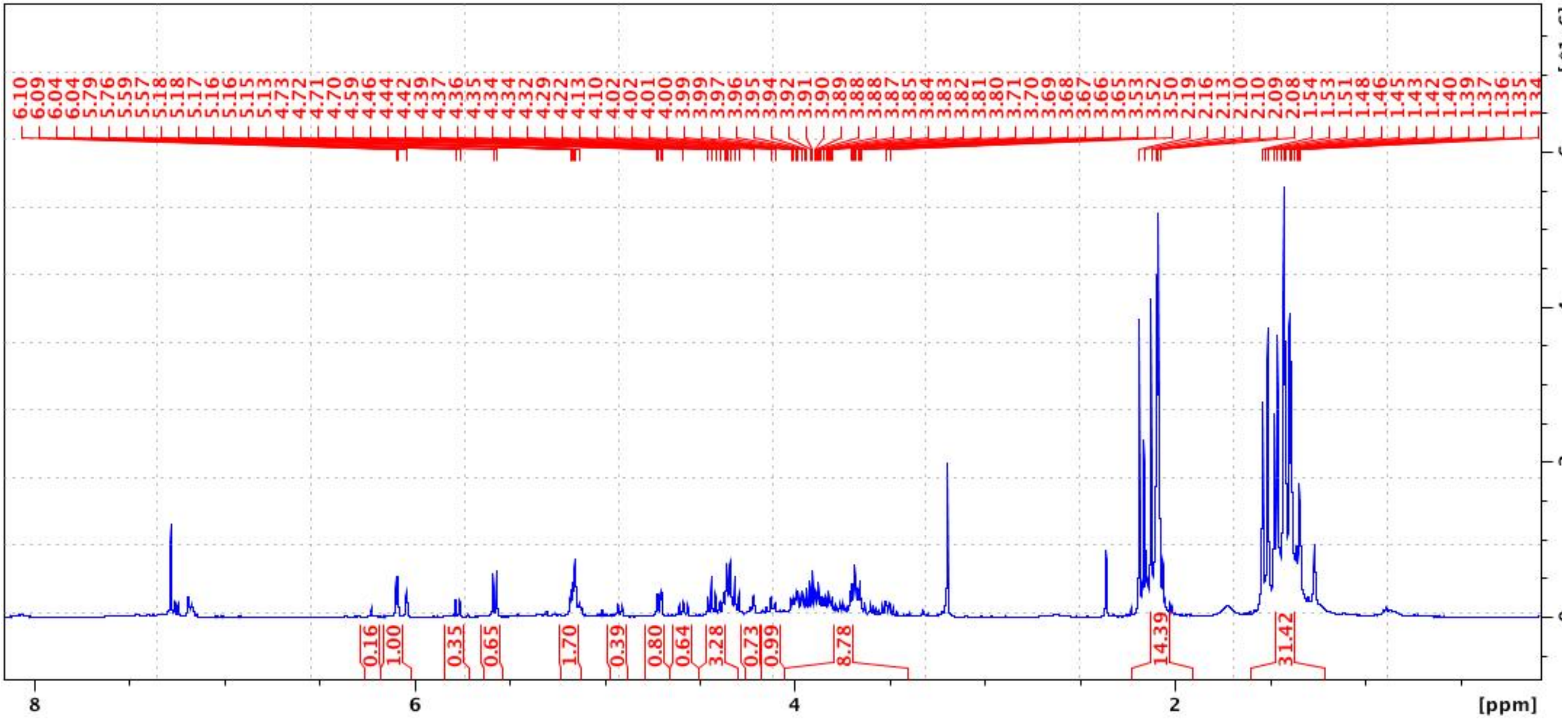

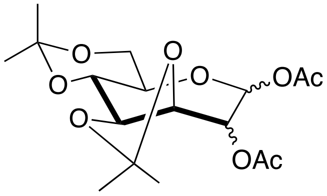

34

^{13}C $CDCl_3$ 400 MHZ