Supporting Information

for

Orthogonal protection of saccharide polyols through solvent-free one-pot sequences based on regioselective silylations

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Experimental and analytical data

Experimental section

Typical procedure for the regioselective mono-*O*-silylation: To a mixture of the polyol substrate (0.5–1 mmol), TBAB and the silylating agent, pyridine was added under air (see Tables 1 and 2 for stoichiometric proportions). The mixture was kept under stirring at room temperature until consumption of the starting material as revealed by TLC analysis (1.5–3 hours). The mixture was concentrated under vacuum and then submitted to silica-gel flash cromatography (eluents: ethyl acetate or ethyl acetate/hexane mixtures) to afford the mono-*O*-silylated products in the yields indicated in Tables 1 and 2.

Regioselective double silylation of monosaccharide polyols or silylation of secondary carbinols: To a mixture of the polyol substrate (0.5–1 mmol), TBAB and the silylating agent, pyridine was added under air (see Table 3 for stoichiometric proportions). The mixture was kept under stirring at 50 °C. Upon completion of the reaction (1–6 hours), the mixture was concentrated under vacuum and then submitted to silica-gel flash chromatography (eluents: hexane/ethyl acetate mixtures) to afford the di-*O*-silylated products in the yields indicated in Table 3. The regiochemistry of the double silylation was determined by acetylation of the isolated products (2:1v/v pyridine/acetic anhydride, overnight, rt) and subsequent NMR analysis.

One-pot synthesis of orthogonally protected building-blocks (silylation–alkylation sequence): Upon completion of the mono-O-silylation step (see above for the procedure), to the mixture were

sequentially added under air DIPEA, benzyl bromide, and Bu_2SnO, and the vessel placed on an oil bath at the suitable temperature (see Table 4 for stoichiometric proportions and the temperature of the second step). The mixture was kept under stirring until TLC analysis indicated optimal conversion. The reaction vessel was cooled to rt and the mixture was diluted with DCM. The organic phase was washed with aqueous NaOH and the aqueous phase re-extracted with DCM. The combined organic phases were dried with anhydrous sodium sulfate and concentrated under vacuum. Flash chromatography (eluents: hexane/ethyl acetate mixtures) provided pure products in the yields indicated in Table 4.

One-pot synthesis of orthogonally protected building-blocks (alkylation-silylation sequence): To a mixture of the substrate (0.5–1 mmol), Bu_2SnO and TBAB, were sequentially added under air DIPEA and benzyl (or allyl) bromide (see Table 4 for stoichiometric proportions). The mixture was kept under stirring at 70 °C (or 90 °C for regioselective allylation) until TLC analysis indicated optimal conversion. The flask was cooled to rt and then pyridine and the silylating agent were sequentially added. On completion of the reaction, the mixture was diluted with DCM. The organic phase was washed with aqueous NaOH and the aqueous phase was re-extracted with DCM. Combined organic phases were dried with anhydrous sodium sulfate and concentrated under vacuum. Flash chromatography (eluents: hexane/ethyl acetate mixtures) provided pure products in the yields indicated in Table 4.

Spectral data

Methyl 6-*O*-*tert*-butyldimethylsilyl-α-D-mannopyranoside (2).[1-3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.67 (s, 1H, H-1), 4.25 (bs, exchangeable, 1H), 4.02 (bs, exchangeable, 1H), 3.90-3.80 (overlapped signals, 3H), 3.76 (bdd, *J* = 2.8 and 9.6 Hz, 1H, H-3), 3.69 (t, *J* = 9.6 Hz, 1H, H-4), 3.53 (m, 1H, H-5), 3.34 (s, 3H, -OCH_3), 0.89 (s, 9H, *t*-butyl protons), 0.09 (s, 6H, -$Si(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 100.7 (C-1), 71.7, 71.4 (C-3, C-5), 70.3 (C-2), 69.5 (C-4), 64.3 (C-6), 54.7 (-OCH_3), 25.8 (-$SiC(CH_3)_3$), 18.2 (-$SiC(CH_3)_3$), - 5.46 (-$Si(CH_3)_2$) ppm . Anal. Calcd. for $C_{13}H_{28}O_6Si$: C, 50.62; H, 9.15. Found: C, 50.75; H, 9.10.MALDI-MS $[M + Na]^+$ calcd. for ($C_{13}H_{28}O_6Si$)331.16, found 331.30.

Methyl 6-*O*-*tert*-butyldimethylsilyl-α-D-glucopyranoside (10). [4-6]

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.32 (bs, exchangeable, 1H), 4.69 (d, *J* = 3.6 Hz, 1H, H-1),4.61 (bs, exchangeable, 1H), 4.55 (bs, exchangeable, 1H), 3.87 (bd, *J* = 10.2 Hz, 1H, H-6a), 3.75 (dd, *J* = 5.6 and 10.2 Hz, 1H, H-6b), 3.71 (t, *J* = 9.6 Hz, 1H, H-3), 3.55-3.45 (overlapped signals, 2H), 3.37 (s,

3H, -OCH_3), 3.49 (t, *J* = 9.6 Hz, 1H, H-4), 0.88 (s, 9H, *t*-butyl protons), 0.06 (s, 6H, -$Si(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 99.3 (C-1), 74.1 (C-3), 71.9, 71.7 (C-5, C-2), 70.8 (C-4), 63.3 (C-6), 54.9 (-OCH_3), 25.9 (-$SiC(CH_3)_3$), 18.3 (-$SiC(CH_3)_3$), -5.3 (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{13}H_{28}O_6Si$: C, 50.62; H, 9.15. Found: C, 50.55; H, 9.15.MALDI-MS $[M + Na]^+$ calcd. for ($C_{13}H_{28}O_6Si$) 331.16, found 331.05.

1,2-*O*-Isopropylidene-6-*O*-*tert*-butyldimethylsilyl-α-D-glucofuranose (11).[7]

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.90 (d, *J* = 3.2 Hz, 1H, H-1), 4.49 (d, *J* = 3.2 Hz, 1H, H-2), 4.30 (d, *J* = 2.0 Hz, 1H, H-3), 4.04 (dd, *J* = 2.0 and 6.8 Hz, 1H, H-4), 3.97 (m, 1H, H-5), 3.82 (dd, *J* = 3.6 and 10.0 Hz, 1H, H-6a), 3.71 (dd, *J* = 4.8 and 10.0 Hz, 1H, H-6b), 3.19 (bs, exchangeable), 1.49 and 1.32 (2 x s, 6H, isopropylidene methyls), 1.09 (s, 9H, t-butyl protons), 0.06 (s, 6 H, -$Si(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.6 (isopropylidene quaternary C), 104.8 (C-1), 85.0 (C-2), 79.5 (C-4), 75.3 (C-3), 69.9 (C-5), 64.0 (C-6), 26.6, 26.1 (isopropylidene CH_3), 25.7 (-$SiC(CH_3)_3$), 18.2 (-$SiC(CH_3)_3$), -5.5 (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{15}H_{30}O_6Si$: C, 53.86; H, 9.04. Found: C, 53.80; H, 9.15. MALDI-MS $[M + Na]^+$ calcd. for ($C_{15}H_{30}O_6Si$)357.40, found 357.30.

Allyl 6-*O*-*tert*-butyldimethylsilyl-β-D-galactopyranoside (12).[8]

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.00-5.90 (m, 1H, -C*H*=CH_2), 5.28 (bd, *J* = 17.2 Hz, 1H, -C*H*=C$H_a$$H_b$), 5.17 (bd, *J* = 10.4 Hz, 1H, -C*H*=$CH_a$$H_b$), 4.34 (dd, *J* = 5.2 and 12.4 Hz, 1H, H-6a), 4.24 (d, *J* = 7.6 Hz, 1H, H-1), 4.11 (dd, *J* = 6.4and 12.4 Hz , 1H, H-6b), 3.94 (bs, 1H, H-4), 3.80-3.60 (m, 2 H, -CH_2CH=CH_2), 3.70 (bt, *J* = 8.8 Hz, 1H, H-2), 3.56 (m, 1H, H-3), 3.43 (m, 1H, H-5), 0.87 (s, 9H, t-butyl protons), 0.06(s, 6 H, -$Si(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.0 (CH_2=*C*H-), 117.9 (*C*H_2=CH-), 101.9 (C-1), 75.0 (C-5), 73.7 (C-3), 71.2 (C-2), 70.0 (CH_2=CH*C*H_2-), 68.7 (C-4), 62.2 (C-6), 25.8 (-$SiC(CH_3)_3$), 18.2 (-$SiC(CH_3)_3$), -5.4 (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{15}H_{30}O_6Si$: C, 53.86; H, 9.04. Found: C, 53.70; H, 9.10. MALDI-MS $[M + Na]^+$ calcd. for ($C_{15}H_{30}O_6Si$) 357.40, found 357.50.

6-*O*-*tert*-Butyldimethylsilyl-1,2,3,4-tetra-*O*-acetyl-α/β-D-mannopyranose (13). (α/β 1:2.1): ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.03 (d, *J* = 1.6 Hz , 1 H, H-1α), 5.80 (bs, 1H, H-1β), 5.40 (d, *J* = 2.4 Hz, 1H, H-2β),5.35 (t, *J* = 9.6 Hz , 1H, H-4α), 5.29 (dd, *J* = 1.6 and 2.4 Hz, 1H, H-2α), 5.28 (t, *J* = 10.0 Hz, 1H, H-4β), 5.18 (dd, *J* = 2.4 and 10.0 Hz, 1H, H-3β), 3.72 (m, 1H, H-5α), 3.80-3-65 (m, 4H), 3.57 (m, 1H, H-5β); 2.13, 2.10, 2.04, 1.99 (x3), 1.96 (x2) (5 x s, 24 H, acetyl methyls), 0.85 (s, 18 H, *t*-butyl protons), 0.01 and – 0.01 (2 x s, 12 H, 2 x -$Si(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.4, 169.8, 169.3, 168.3 (*C*OCH_3), 90.5 (C-1 α), 90.2 (C-1 β), 75.7 (C-5 β), 73.2 (C-5

α), 70.9 (C-3 β), 69.0 (C-2 α), 68.4 (C-2 β), 68.3 (C-4 β), 65.8, 65.7 (C-3 α, C-4 α), 62.1 (C-6 β), 61.8 (C-6 α), 25.6 (-SiC(*C*H_3)$_3$), 20.7-20.4 (CO*C*H_3), 18.1 (-Si*C*(CH_3)$_3$), -5.5 (-Si (CH_3)$_2$) ppm. Anal. Calcd. for $C_{20}H_{34}O_{10}Si$: C, 51.93; H, 7.41. Found: C, 51.80; H, 7.45. MALDI-MS $[M + Na]^+$ calc. for ($C_{20}H_{34}O_{10}Si$) 485.18, found 485.45.

Methyl 6-*O*-*tert*-butyldiphenylsilyl-α-D-mannopyranoside (14).[9-10]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.30 (aromatic H), 4.65 (s, 1H, H-1), 3.93 (dd, *J* = 2.8 and 8.8 Hz, 1H, H-3), 3.90-3.80 (overlapped signals, 2H), 3.80-3.70 (overlapped signals, 2H), 3.61 (m, 1H), 3.29 (s, 3H, -OCH_3), 1.05 (s, 6H, -Si(CH_3)$_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.7, 132.9, 129.9, 127.6 (aromatic signals), 100.6 (C-1), 71.7, 71.1 (C-3 and C-5), 70.3 (C-2), 69.8 (C-4), 65.0 (C-6), 54.7 (-OCH_3), 26.7 (-SiC(*C*H_3)$_3$), 19.1 (-Si*C*(CH_3)$_3$) ppm. Anal. Calcd. for $C_{23}H_{32}O_6Si$: C, 63.86; H, 7.46. Found: C, 63.95; H, 7.45. MALDI-MS $[M + Na]^+$ calcd. for ($C_{23}H_{32}O_6Si$) 455.19, found 455.05.

Methyl 6-*O*-*tert*-butyldiphenylsilyl-α-D-glucopyranoside (15).[6, 11, 12]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.30 (aromatic H), 4.80 (bs, exchangeable, 1H), 4.70 (d, *J* = 3.6 Hz, 1H, H-1), 4.00-3.85 (overlapped signals, 2H), 3.82 (dd, *J* = 5.4 and 10.8 Hz, 1H, H-6b), 3.76 (t, *J* = 9.2 Hz, 1H, H-3), 3.64 (m, 1H, H-5), 3.55-3.43 (overlapped signals, 2H), 3.34 (s, 3H), 1.04 (s, 9H, -Si(CH_3)$_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.7, 133.2, 129.6, 127.7 (aromatic signals), 99.1 (C-1), 74.3 (C-3), 72.0 (C-2), 71.5 (C-5), 71.1 (C-4), 64.0 (C-6), 54.8 (-OCH_3), 26.8 (-SiC(*C*H_3)$_3$), 19.1 (-Si*C*(CH_3)$_3$) ppm. Anal. Calcd. for $C_{23}H_{32}O_6Si$: C, 63.86; H, 7.46. Found: C, 63.75; H, 7.50. MALDI-MS $[M + Na]^+$ calcd. for ($C_{23}H_{32}O_6Si$) 455.19, found 455.40.

1,2-*O*-Isopropylidene-6-*O*-*tert*-butyldiphenylsilyl-α-D-glucofuranose (16).[13]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.30 (aromatic H), 5.96 (d, *J* = 3.2 Hz, 1H, H-1), 4.54 (d, *J* = 3.2 Hz, 1H, H-2),4.40 (d, *J* = 1.6Hz, 1H, H-3),4.21 (dd, *J* = 1.6 and 6.4 Hz, 1H, H-4), 4.12 (m, 1H, H-5), 3.91 (dd, *J* = 4.0 and 10.4 Hz, 1H, H-6a), 3.85 (dd, *J* = 5.2 and 10.0 Hz, 1H, H-6b), 1.44 and 1.28 (2 x s, 6H), 0.89 (s, 9H), 0.06 (s, 6H, -Si(CH_3)$_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.5, 132.2, 129.8, 127.8 (aromatic signals), 111.4 (isopropylidene quaternary C), 104.8 (C-1), 85.0 (C-2), 79.2 (C-4), 75.4 (C-3), 70.2 (C-5), 64.8 (C-6), 26.7 (-SiC(*C*H_3)$_3$), 26.7, 26.1 (isopropylidene CH_3), 19.1 (-Si*C*(CH_3)$_3$) ppm. Anal. Calcd. for $C_{25}H_{34}O_6Si$: C, 65.47; H, 7.47. Found: C, 65.67; H, 7.35. MALDI-MS $[M + Na]^+$ calcd. for ($C_{25}H_{34}O_6Si$) 481.20, found 481.30.

Methyl 3,6-di-*O*-*tert*-butyldimethylsilyl-α-D-mannopyranoside (17).[14, 15]

^{1}H NMR (400 MHz, $CDCl_3$):δ 4.71 (s, 1H, H-1), 3.86 (d, *J* = 5.6 Hz, 2H, H_2-6), 3.83 (dd, *J* = 3.6 and 9.2 Hz, 1H, H-3), 3.74 (bd, *J* = 2.0 Hz, 1 H, H-2), 3.70 (t, *J* = 9.2 Hz, 1H, H-4), 3.55 (m, 1H, H-5), 3.36 (s, 3H, 1-OCH_3), 2.70 (bs, exchangeable, 1H), 2.57 (bs,exchangeable, 1H), 0.91 and 0.90 (2 x s, 18 H, *t*-butyl protons); 0.15, 0.13, 0.09 (x2) (3 x s, 18H, 2 x -Si$(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 100.0 (C-1), 72.9 (C-3), 71.0, 70.4 (x2) (C-2, C-4, C-5), 64.9 (C-6), 54.8 (-OCH_3), 25.8, 25.7 (2 x -SiC(CH_3)$_3$), 18.1 (2 x -Si*C*$(CH_3)_3$) -4.5, -4.9, -5.5 (x2) (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{19}H_{42}O_6Si_2$: C, 53.99; H, 10.01. Found: C, 53.80; H, 9.95. MALDI-MS $[M + Na]^+$ calcd. for ($C_{19}H_{42}O_6Si_2$) 445.24, found 445.45.

Methyl 2,6-di-*O*-*tert*-butyldimethylsilyl-α-D-glucopyranoside (18).[16]

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.60 (d, *J* = 3.2 Hz, 1H, H-1), 3.85-3.82 (overlapped signals, 2H), 3.80 (t, *J* = 9.2 Hz, 1H, H-3), 3.60 (m, 1H, H-5), 3.54 (dd, *J* = 3.2 and 9.2 Hz, 1H, H-2), 3.51 (t, *J* = 9.2 Hz, 1H, H-4), 3.38 (s, 3H), 0.90 (s, 18H), 0.14, 0.11, 0.10 (x2) (3 x s, 12 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 99.8 (C-1), 73.9 (C-3), 73.4 (C-2), 71.8 (C-4), 70.5 (C-5), 64.0 (C-6), 55.2 (-OCH_3), 25.9, 25.8 (2 x -SiC(CH_3)$_3$), 18.3, 18.1 (2 x -Si*C*$(CH_3)_3$), -4.6, -5.4 (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{19}H_{42}O_6Si_2$: C, 53.99; H, 10.01. Found: C, 53.95; H, 10.05. MALDI-MS $[M + Na]^+$ calc. for ($C_{19}H_{42}O_6Si_2$) 445.24, found 445.10.

3,6-Di-*O*-*tert*-butyldimethylsilyl-D-galactal (19).[17]

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.45 (d, *J* = 6.0 Hz, 1H, H-1), 4.63 (d, *J* = 6.0 Hz, 1H, H-2), 4.57 (d, *J* = 2.4 Hz, 1H), 4.20-3.95 (overlapped signals, 4H), 2.82 (s, exchangeable 1H), 1.03 and 1.02 (2 x s, 18H, *t*-butyl protons), 0.24 and 0.21 (2 x s, 12H, 2 x -Si$(CH_3)_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.2 (C-1), 102.2 (C-2), 76.8 (C-5), 65.0, 64.8 (C-3, C-4), 61.9 (C-6), 25.9, 25.8 (2 x -SiC(CH_3)$_3$), 18.3, 18.0 (2 x -Si*C*$(CH_3)_3$), -4.6, -4.9, -5.3, -5.4 (-Si $(CH_3)_2$) ppm. Anal. Calcd. for $C_{18}H_{38}O_4Si_2$: C, 57.70; H, 10.22. Found: C, 57.75; H, 10.35. MALDI-MS $[M + Na]^+$ calc. for ($C_{18}H_{38}O_4Si_2$) 397.22, found 397.45.

Allyl 6′-*O*-*tert*-butyldimethylsilyl-β-D-galacatopyranosyl-(1→4)-6-*O*-*tert*-butyldimethylsilyl-β-D-glucopyranoside (21). $[\alpha]_D^{25}$+41 (*c* 1.0, $CHCl_3$);^{1}H NMR (400 MHz, $CDCl_3$): δ 6.00-5.90 (m, 1H, -C*H*=CH_2), 5.41 (bd, *J* = 17.6 Hz, 1H, -CH=CH_aH$_b$), 5.41 (bd, *J* = 10.4 Hz, 1H,-CH=C$H_a$$H_b$), 4.98 (d, *J* = 3.2 Hz, 1H, H-1), 4.47 (d, *J* = 7.6 Hz, 1H, H-1′), 4.26 (dd, *J* = 6.0 and 12.8 Hz, 1H, –C*Ha*HbCH=CH_2), 4.13 (dd, *J* = 6.0 and 12.8 Hz, 1H, –CHa*Hb*CH=CH_2), 4.09 (bs, 1H, H-4′), 4.00-3.70 (overlapped signals), 1.00 (s, 18H, *t*-butyl protons), 0.18 (s, 9H, -Si$(CH_3)_2$) ppm. ^{13}C NMR

(100 MHz, $CDCl_3$): δ 133.7 (CH_2=*C*H), 117.8 (*C*H_2=CH), 103.1 (C-1′), 98.9 (C-1), 79.0 (C-4), 75.1 (C-5′), 73.8 (C-3′), 72.3, 72.2, 71.3, 70.7 (C-2, C-3, C-5, C-2′), 68.4, 68.3 (C-4′ and CH_2=CH*C*H_2-), 62.4, 61.7 (C-6, C-6′), 25.8 (-SiC(*C*H_3)$_3$), 18.3 (-Si*C*(CH_3)$_3$), -5.28 (-Si (CH_3)$_2$) ppm. Anal. Calcd. for $C_{27}H_{54}O_{11}Si_2$: C, 53.09; H, 8.91. Found: C, 53.15; H, 8.80. MALDI-MS [M + Na]$^+$ calcd. for ($C_{27}H_{54}O_{11}Si_2$) 633.31, found 633.15.

Ethyl 3-*O*-*tert*-butyldimethylsilyl-α/β-L-1-thio-rhamnopyranoside (23).

(α/β ca 4:1).^{1}H NMR (400 MHz, $CDCl_3$): signals of prevalent α-anomer at δ 5.30 (s, 1H, H-1), 4.02 (m, 1H, H-5), 3.89 (bs, 1H, H-2), 3.79 (dd, *J* = 3.2 and 8.8 Hz, 1H, H-3), 3.53 (t, *J* = 8.8 Hz, 1H, H-4), 2.75-2.55 (m, 2H, -C*H*$_2$CH$_3$), 1.31 (t, *J* = 5.6 Hz, 3 H, -CH$_2$C*H*$_3$), 0.92-0.89 (overlapped signals), 0.15 and 0.13 (2 x s, 6H, -Si(CH_3)$_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 83.0 (C-1), 74.0, 73.6, 73.2 (C-2, C-3, C-4), 68.0 (C-5), 25.7 (-SiC(*C*H_3)$_3$), 24.9 (-S*C*H_2CH$_3$), 18.1 (-Si*C*(CH_3)$_3$), 17.5 (C-6), 14.8 (-SCH$_2$*C*H$_3$), -4.6, -4.7 (-Si(CH_3)$_2$) ppm. Anal. Calcd. for $C_{14}H_{28}O_4SSi$: C, 52.13; H, 9.38. Found: C, 52.30; H, 9.30. MALDI-MS [M + Na]$^+$ calcd. for ($C_{14}H_{28}O_4SSi$) 345.15, found 345.35.

Ethyl 4-*O*-benzoyl-3-*O*-*tert*-butyldimethylsilyl-α/β-L-1-thio-rhamnopyranoside (25).

(α:β 4:1).^{1}H NMR (400 MHz, $CDCl_3$): signals of prevalent α-anomer at δ = 8.00-7.30 (aromatic Hs, H), 5.38 (s, 1H, H-1), 5.28 (t, *J* = 8.8 Hz, 1H, H-4), 4.25 (m, 1H, H-5), 4.08 (dd, *J* = 2.4 and 9.2 Hz, 1H, H-3), 3.97 (bs, 1H, H-2), 2.75-2.50 (m, 2H, -C*H*$_2$CH$_3$), 1.31 (t, *J* = 7.2 Hz, 3H, -CH$_2$C*H*$_3$), 1.22 (d, *J* = 6.4 Hz, 3H, H$_3$-6), 0.77 (s, 9H, t-butyl protons), 0.04, -0.1 (2 x s, 6H, -Si(CH_3)$_2$) ppm.^{13}C NMR (100 MHz, $CDCl_3$):δ = 135.5, 133.1, 129.7, 128.4 (aromatic signals), 82.9 (C-1), 74.5, 73.1, 71.2 (C-2, C-3, C-4), 66.6 (C-5), 25.4 (-SiC(*C*H_3)$_3$), 24.9 (-S*C*H_2CH$_3$), 18.1 (-Si*C*(CH_3)$_3$), 17.3 (C-6), 14.8 (-SCH$_2$*C*H$_3$), -4.6, -4.7 (-Si(CH_3)$_2$) ppm. Anal. Calcd. for $C_{21}H_{34}O_5SSi$: C, 59.12; H, 8.05. Found: C, 59.00; H, 8.15. MALDI-MS [M + Na]$^+$ cald. for ($C_{21}H_{34}O_5SSi$) 449.18, found 449.05.

Methyl 2,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranoside (26).[18,19]

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.68 (d, *J* = 3.6 Hz, 1H, H-1), 3.85-3.80 (overlapped signals, 2 H), 3.76 (dd, *J* = 4.8 and 11.6 Hz, 1H, H-6a), 3.60-3.50 (overlapped signals, 3H), 3.41 (s, 3H, -OCH$_3$); 0.24, 0.23 (x2), 0.20 (3 x s, 12H, 4 x -Si(CH_3)$_3$). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 99.6 (C-1), 75.2 (C-3), 73.9 (C-2), 72.1 (C-4), 71.9 (C-5), 62.1 (C-6), 54.4 (-OCH$_3$), 1.2, 0.8, 0.4 -0.3 (4 x -Si(CH_3)$_3$). Anal. Calcd. for $C_{19}H_{46}O_6Si_4$: C, 47.26; H, 9.60. Found: C, 47.05; H, 9.70.

2-Amino-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (27).[20-24]

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.05 (d, *J* = 3.2 Hz, 1H, H-1), 3.70-3.55 (overlapped signals, 3H), 3.47 (t, *J* = 8.8 Hz, 1H, H-3), 3.43 (t, *J* = 8.8 Hz, 1H, H-4), 2.47 (dd, *J* = 3.2 and 8.8 Hz, 1H, H-2), 0.14, 0.11, 0.09, 0.03 (4 x s, 12H, 4 x -Si$(CH_3)_3$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 94.5 (C-1), 77.5 (C-3), 72.7 (C-5), 71.9 (C-4), 61.9 (C-6), 57.3 (C-2), 1.2, 0.7, -0.2, -0.4 (4 x -Si$(CH_3)_3$) ppm. Anal. Calcd. for $C_{18}H_{45}NO_5Si_4$: C, 46.22; H, 9.69. Found: C, 46.10; H, 9.80.

2-Deoxy-2-trichloroethoxycarbonylamino-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (28).[21]

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.08 (d, *J* = 2.4 Hz, 1H, H-1), 4.87 (d, *J* = 9.2 Hz, 1H, -NHTroc), 4.81 and 4.59 (2 x d, AB, *J* = 12.0 Hz, 2 H, -C*H*$_2$CCl_3), 3.75-3.50 (overlapped signals, 6H), 0.16, 0.14, 0.13, 0.08 (4 x s, 12H, 4 x -Si$(CH_3)_3$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.1 (carbamate CO), 92.5 (C-1), 74.7 (-OCH_2CCl_3), 73.8, 72.4, 71.9 (C-3, C-4, C-5), 61.6 (C-6), 56.8 (C-2), 0.9, 0.7, -0.2, -0.3 (4 x -Si$(CH_3)_3$) ppm. Anal. Calcd. for $C_{21}H_{46}Cl_3NO_7Si_4$: C, 39.21; H, 7.21. Found: C, 39.20; H, 7.10.

Methyl 3-O-benzyl-6-*O*-*tert*-butyldimethylsilyl-α-D-mannopyranoside (29).[25-27]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.20 (aromatic H), 4.71 (s, 1H, H-1), 4.67 (s, 2H, -C*H*$_2$Ph), 3.94 (bs, 1H, 2-H), 3.90-3.84 (overlapped signals, 3H), 3.66 (dd, *J* = 2.8 and 8.8 Hz, H-3), 3.58 (m, 1H, H-5), 3.34 (s, 3H, -OCH_3), 0.90 (s, 9H, *t*-butyl protons), 0.08 (s, 6H, -Si$(CH_3)_2$). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 128.5, 127.9, 127.8 (aromatic signals), 100.3 (C-1), 79.4 (C-3), 72.0 (-CH_2Ph), 71.0 (C-5), 68.6 (C-4), 67.7 (C-2), 64.4 (C-6), 54.7 (-OCH_3), 25.8 (-SiC(*C*$H_3)_3$), 18.2 (-Si*C*$(CH_3)_3$), -5.5 (-Si$(CH_3)_2$) ppm. Anal. Calcd. for $C_{20}H_{34}O_6Si$: C, 60.27; H, 8.60. Found: C, 60.45; H, 8.50. MALDI-MS $[M + Na]^+$ calcd. for ($C_{20}H_{34}O_6Si$) 421.20, found 421.55.

Methyl 3-*O*-allyl-6-*O*-*tert*-butyldiphenylsilyl-α-D-mannopyranoside (30).[28]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.30 (aromatic H), 6.00-5.90 (m, 1H, -C*H*=CH_2), 5.33 (bd, *J* = 17.2 Hz, 1H, -CH=C*H*$_a$H$_b$), 5.22 (bd, *J* = 10.4 Hz, 1H, -CH=CH$_a$*H*$_b$), 4.75 (s, 1H, H-1), 4.20-4.10 (m, 2H, -C*H*$_2$CH=CH_2), 4.00 (bs, 1H, H-2), 3.99-3.85 (overlapped signals, 3H), 3.67 (m, 1H, H-5), 3.62 (dd, *J* = 2.8 and 8.8 Hz, 1H, H-3), 3.34 (s, 3H, -OCH_3), 1.07 (s, 9H, *t*-butyl protons) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.5, 134.4, 129.6, 127.6 (aromatic), 132.0 (CH_2=*C*H-), 117.6 (*C*H_2=CH-), 100.3 (C-1), 79.0 (C-3), 71.2 (C-5), 70.7 (-*C*H_2CH=CH_2), 68.3 (C-4), 67.6 (C-2), 64.9 (C-6), 54.6 (-OCH_3), 26.7 (-SiC(*C*$H_3)_3$), 19.1 (-Si*C*$(CH_3)_3$) ppm. Anal. Calcd. for $C_{26}H_{36}O_6Si$: C,

66.07; H, 7.68. Found: C, 66.25; H, 7.55. MALDI-MS $[M + Na]^+$ calcd. for ($C_{26}H_{36}O_6Si$) 495.22, found 495.30.

Methyl 3-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-α-D-mannopyranoside (31).[29]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.25(aromatic H), 4.88 (s, 1H, H-1), 4.84 (s, 2H, -C*H*$_2$Ph), 4.13 (bs, 1H, H-2), 4.10-4.03 (overlapped signals, 3H), 3.90-3.75 (overlapped signals, 2H), 3.48 (s, 3H, -OCH_3), 2.93 (bs, exchangeable, 1H), 2.61 (bs, exchangeable, 1H), 1.22 (s, 9 H, *t*-butyl protons) ppm. ^{13}C NMR (100 MHz, $CDCl_3$):δ = 137.9, 135.6, 133.0, 128.7, 128.5, 127.9, 127.7 (aromatic signals), 100.2 (C-1), 79.5 (C-3), 72.0 (-CH_2Ph), 71.1 (C-5), 68.5 (C-4), 67.7 (C-2), 64.9 (C-6), 54.7 (-OCH_3), 26.7 (-SiC(*C*H_3)$_3$), 19.1 (-Si*C*(CH_3)$_3$) ppm. Anal. Calcd. for $C_{30}H_{38}O_6Si$: C, 68.93; H, 7.33. Found: C, 68.75; H, 7.40. MALDI-MS $[M + Na]^+$ calcd. for ($C_{30}H_{38}O_6Si$) 545.23, found 545.40.

Methyl 2-*O*-benzyl-6-*O*-*tert*-butyldiphenylsilyl-α-D-glucopyranoside (32).[27]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.25 (aromatic H), 4.85 and 4.82 (2 x d, AB, *J* = 12.0 Hz, 2H, -CH_2Ph), 4.76 (d, *J* = 3.2 Hz, 1H, H-1), 4.07 (t, *J* = 9.2 Hz, 1H, H-3), 4.04 (dd, *J* = 3.2 and 10.4 Hz, 1H, H-6a), 3.97 (dd, *J* = 5.2and 10.4 Hz, 1H, H-6b), 3.78 (m, 1H, H-5), 3.66 (t, *J* = 9.2 Hz, 1H, H-4), 3.48 (dd, *J* = 3.2 and 9.2 Hz, 1H, H-2), 3.48 (s, 3H, OCH_3), 3.20 (bs, exchangeable, 1H), 1.20 (s, 9H, *t*-butyl protons) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 135.5, 133.1, 129.6, 128.4, 128.0, 127.9, 127.6 (aromatic signals), 97.4 (C-1), 79.2 (C-2), 73.0 (-CH_2Ph), 72.9 (C-3), 71.6 (C-4), 70.6 (C-5), 64.3 (C-6), 54.9(-OCH_3), 26.7 (-SiC(*C*H_3)$_3$), 19.1 (-Si*C*(CH_3)$_3$) ppm. Anal. Calcd. for $C_{30}H_{38}O_6Si$: C, 68.93; H, 7.33. Found: C, 68.80; H, 7.35. MALDI-MS $[M + Na]^+$ calcd. for ($C_{30}H_{38}O_6Si$) 545.23, found 545.55.

3-*O*-Benzyl-6-*O*-*tert*-butyldimethylsilyl-D-galactal (33).[30]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.30 (aromatic H), 6.40 (d, *J* = 6.0 Hz, 1H, H-1),4.70 (d, *J* = 6.0 Hz, 1H, H-2), 4.69-4.60 (AB, *J* = 12.0 Hz, 2H, -C*H*$_2$Ph), 4.21 (bs, 1H), 4.14 (bs, 1H), 3.97 (m, 1H), 3.90-3.80 (m, 2H), 2.63 (bs, 1H), 0.92 (s,9H, *t*-butyl protons), 0.11 (s, 6H, -Si(CH_3)$_2$) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.9 (C-1), 137.7, 128.5, 127.9, 127.7 (aromatic signals), 99.5 (C-2), 76.6 (C-5), 70.9 (-CH_2Ph), 70.4 (C-3), 62.4, 62.1 (C-4, C-6), 26.0 (-SiC(*C*H_3)$_3$), 19.2 (-Si*C*(CH_3)$_3$), -5.4 (-Si (CH_3)$_2$) ppm. Anal. Calcd. for $C_{19}H_{30}O_4Si$: C, 65.10; H, 8.63. Found: C, 65.25; H, 8.55. MALDI-MS $[M + Na]^+$ calcd. for ($C_{19}H_{30}O_4Si$) 350.42, found 350.55

3-O-Benzyl-6-*O*-*tert*-butyldiphenylsilyl-D-galactal (34).

$[\alpha]_D^{25}$ - 3 (*c* 1.9, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.30 (aromatic H), 6.50 (d, *J* = 6.0 Hz, 1H, H-1), 4.84 (d, *J* = 6.0 Hz, 1H, H-2), 4.82-4.75 (AB, *J* = 12.0 Hz, 2H, -C*H*$_2$Ph), 4.35 (s, 2H), 4.23 (m, 1H), 4.15-4.00 (overlapped signals, 2H), 2.73 (s, 1 H), 1.22 (s, 9 H, *t*-butyl protons) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.9 (C-1), 137.7, 135.5, 133.1, 129.7-127.7 (aromatic signals), 99.5 (C-2), 76.5 (C-5), 70.9 (-CH_2Ph), 70.4 (C-3), 62.6, 62.5 (C-4, C-6), 26.7 (-SiC(*C*H_3)$_3$), 19.1 (-Si*C*(CH$_3$)$_3$) ppm. Anal. Calcd. for $C_{29}H_{34}O_4Si$: C, 73.38; H, 7.22. Found: C, 73.20; H, 7.40. MALDI-MS $[M + Na]^+$ calc. for ($C_{29}H_{34}O_4Si$) 497.21, found 497.45.

Allyl 3-*O*-benzyl-6-*O*-*tert*-butyldimethylsilyl-α-D-galactopyranoside (35).

$[\alpha]_D^{25}$+81 (*c* 1.6, $CHCl_3$);^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.20 (aromatic H), 6.00-5.90 (m, 1H, -C*H*=CH$_2$), 5.28 (bd, *J* = 1.6 and 17.2 Hz, 1H, -CH=C*H*$_a$H$_b$), 5.20 (bd, *J* = 1.6 and 10.4 Hz, 1H, -CH=CH$_a$*H*$_b$), 4.95 (d, *J* = 4.0 Hz, 1H, H-1), 4.76 and 4.71 (2 x d, AB, *J* = 11.6 Hz, 2 H, -C*H*$_2$Ph), 4.19 (bdd, *J* = 5.2 and 12.8 Hz, 1H, -C*Ha*HbCH=CH$_2$), 4.05 (d, *J* = 3.2 Hz, 1H, H-4), 4.02 (dd, *J* = 6.6 and 12.8 Hz, 1H, -CHa*Hb*CH=CH$_2$), 3.84 (dd, *J* = 5.6 and 9.6 Hz, 1H, H-6a), 3.80-3.70 (overlapped signals, 2H), 3.63 (dd, *J* = 2.8 and 9.6 Hz, 1H, H-3), 2.63 (bs, exchangeable, 1H), 2.23 (bs, exchangeable, 1H), 0.89 (s, 9H, *t*-butyl protons), 0.07 (s, 6H, -Si(CH$_3$)$_2$). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 128.5, 127.9, 127.8 (aromatic signals), 133.6 (CH$_2$=*C*H-), 117.8 (*C*H$_2$=CH-), 97.6 (C-1), 78.7 (C-3), 72.0 (-CH_2Ph), 70.2 (C-5), 68.6 (C-2), 68.4 (CH$_2$=CH*C*H$_2$-), 66.9 (C-4), 62.5 (C-6), 25.8 (-SiC(*C*H$_3$)$_3$), 18.2 (-Si*C*(CH$_3$)$_3$), -5.4 (-Si (CH$_3$)$_2$). Anal. Calcd. for $C_{22}H_{36}O_6Si$: C, 62.23; H, 8.55. Found: C, 62.10; H, 8.60. MALDI-MS $[M + Na]^+$ calcd. for ($C_{22}H_{36}O_6Si$) 447.22, found 447.40.

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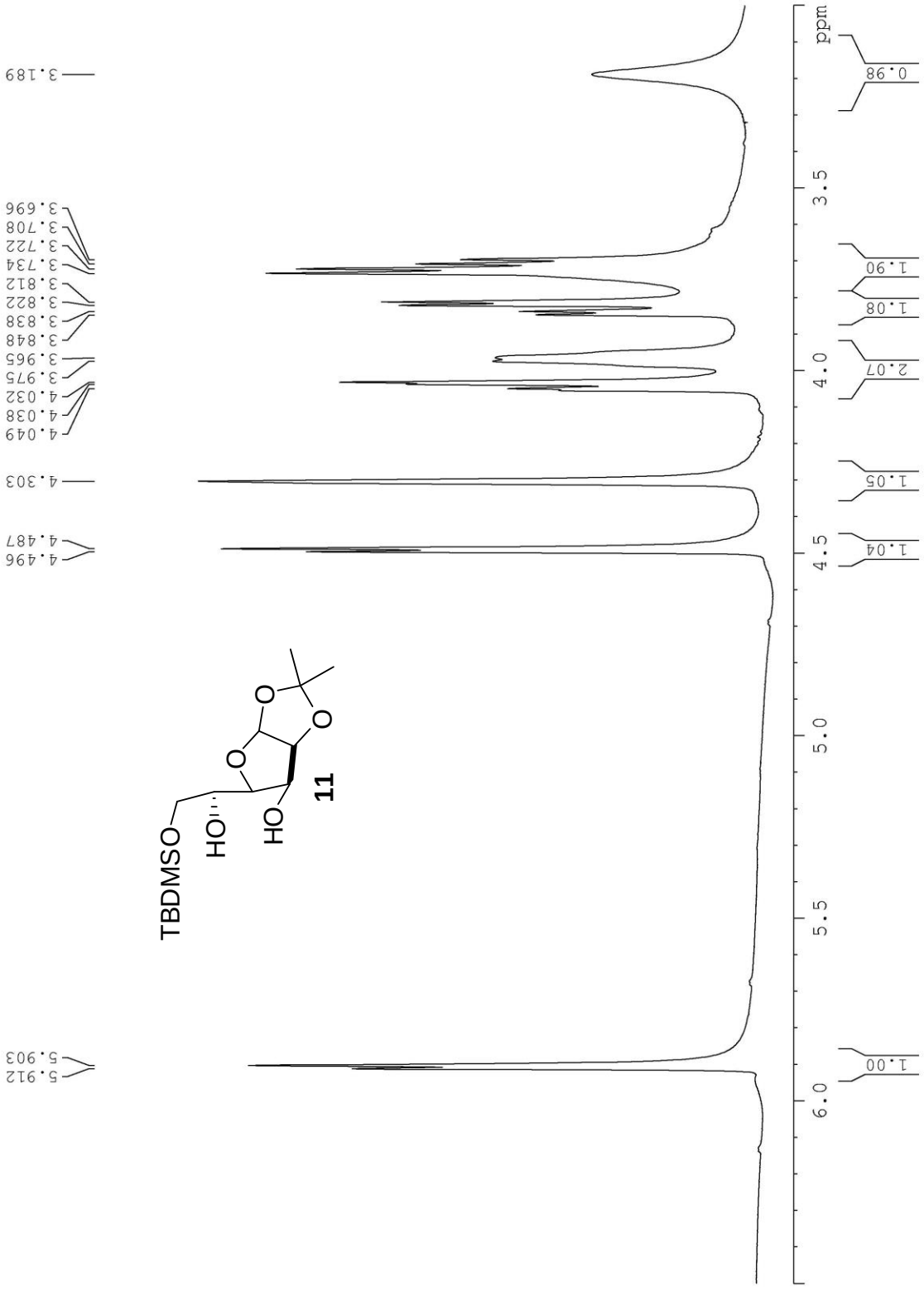
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HO
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1.05
2.07
1.08
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5.903
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4.303
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4.038
4.032
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3.965
3.848
3.838
3.822
3.812
3.734
3.722
3.708
3.696
3.189

-5.55
18.17
25.75
26.08
26.65
64.06
69.94
75.30
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76.99
77.31
79.47
85.00
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111.48
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O
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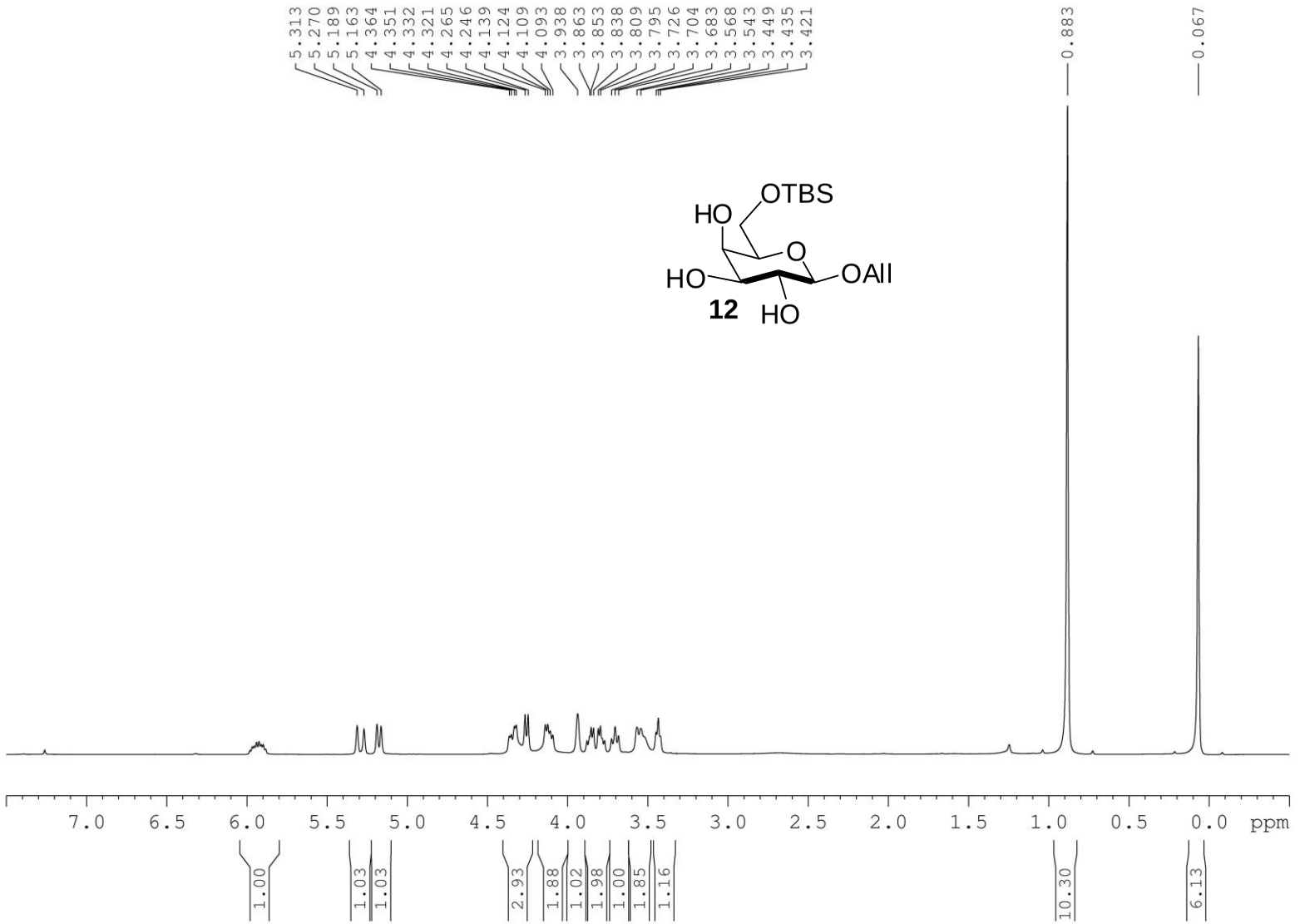
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4.093
3.938
3.863
3.853
3.838
3.809
3.795
3.726
3.704
3.683
3.568
3.543
3.449
3.435
3.421
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0.067
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HO
O
HO
OAll
12
HO
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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1.03
1.03
2.93
1.88
1.02
1.98
1.00
1.85
1.16
10.30
6.13

-5.45
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71.40
73.73
74.92
76.65
76.97
77.29
101.96
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133.99
OTBS
HO
O
OAll
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HO
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ppm
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60
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120
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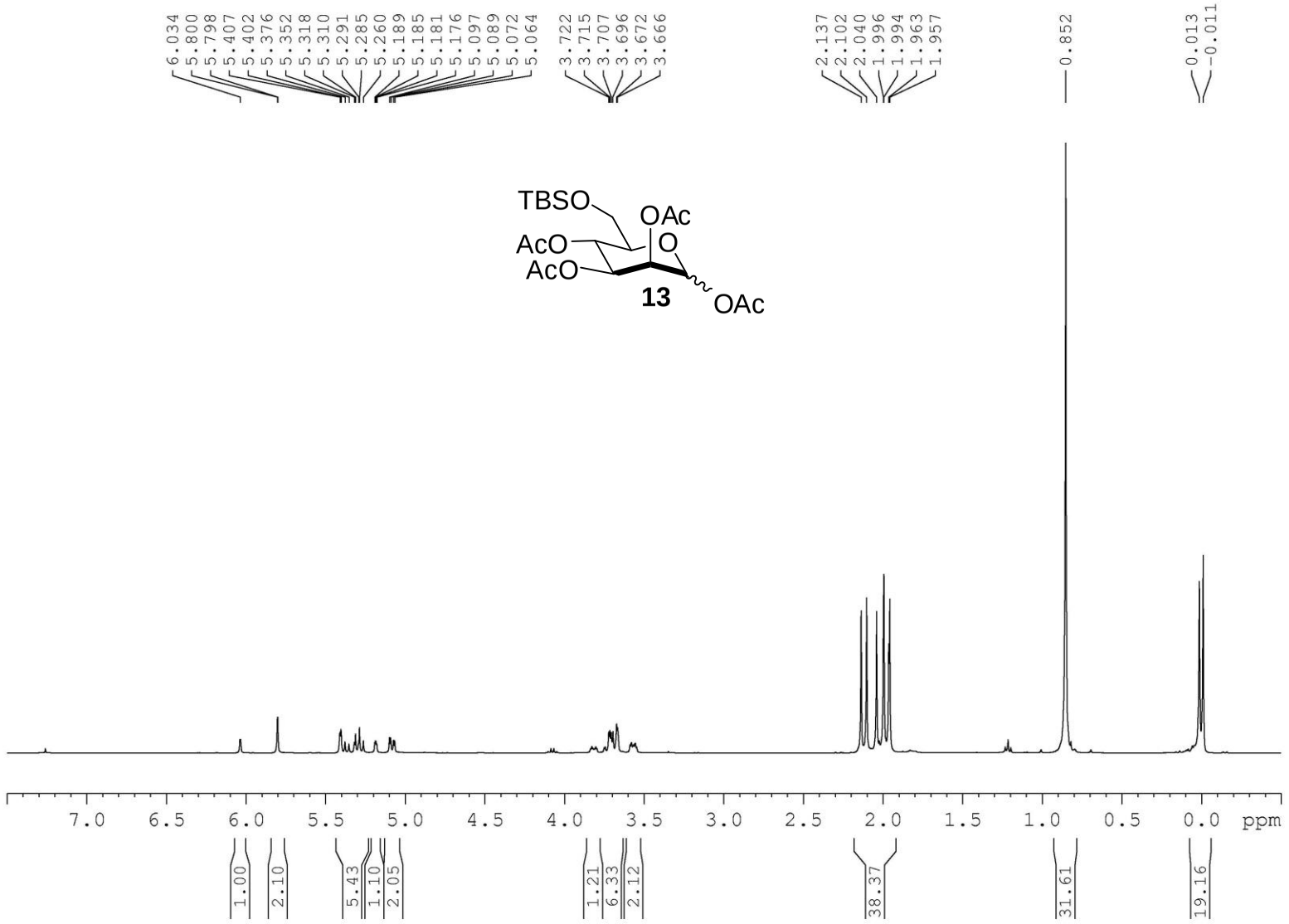
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5.181
5.176
5.097
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5.064
3.722
3.715
3.707
3.696
3.672
3.666
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2.102
2.040
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1.994
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0.013
-0.011
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OAc
AcO
O
AcO
13
OAc
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6.0
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5.0
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4.0
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2.0
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1.0
0.5
0.0
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1.10
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1.21
6.33
2.12
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31.61
19.16

6.034
5.800
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TBSO
AcO
AcO
OAc
O
OAc
13
ppm
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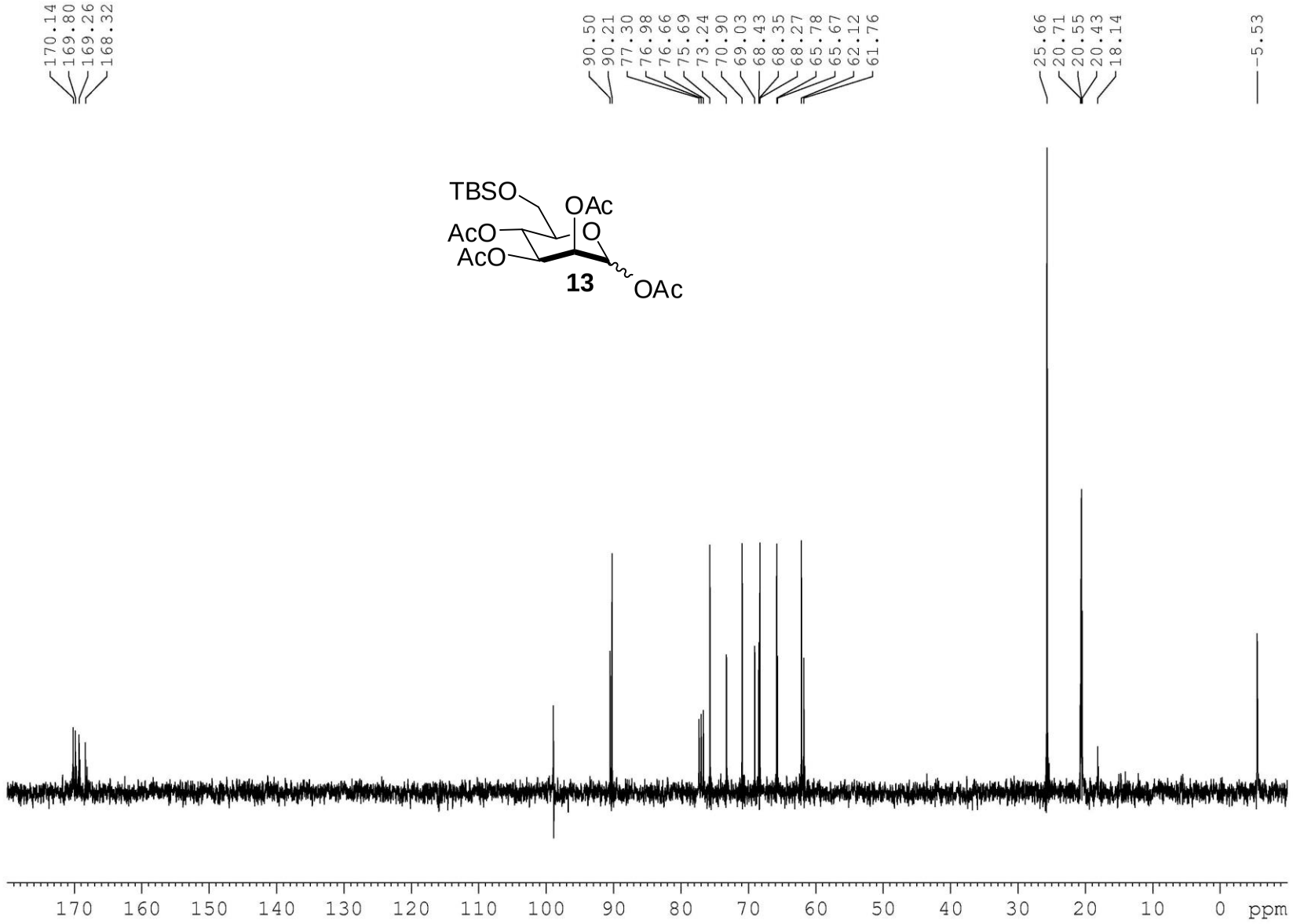
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68.35
68.27
65.78
65.67
62.12
61.76
25.66
20.71
20.55
20.43
18.14
-5.53
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OAc
AcO
O
AcO
13
OAc
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160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

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HO
HO
OH
O
14
OCH3
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3.5
4.0
4.5
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6.0
6.5
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6.10
3.94

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19.13
TBDPSO
HO
HO
OH
O
14
OCH3
ppm

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3.623
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HO
HO
O
HO
OCH3
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1.01
1.90
2.78
9.55

4.702
4.693
3.931
3.912
3.905
3.844
3.830
3.817
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3.623
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3.476
3.453
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3.338
TBDPSO
HO
HO
O
HO
OCH3
15
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4.5
4.4
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3.3
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40
50
60
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80
90
100
110
120
130
140
TBDPSO
HO
HO
O
HO
OCH3
15

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1.03
1.00
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4.34

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HO
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O
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4.2
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1.05
1.04
2.11

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85.04
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50
60
70
80
90
100
110
120
130
TBDPSO
HO
HO
O
O
O
16

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0.910
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3.556
3.569
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3.745
3.751
3.818
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3.856
3.869
4.717
TBSO
HO
TBSO
OH
O
17
OCH3
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1.5
2.0
2.5
3.0
3.5
4.0
4.5
12.01
20.48
2.11
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2.07
2.17
3.30
1.00

4.717
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3.840
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3.818
3.751
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3.569
3.556
3.545
3.358
2.705
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HO
TBSO
OH
O
17
OCH3
4.8
4.6
4.4
4.2
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3.8
3.6
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2.6
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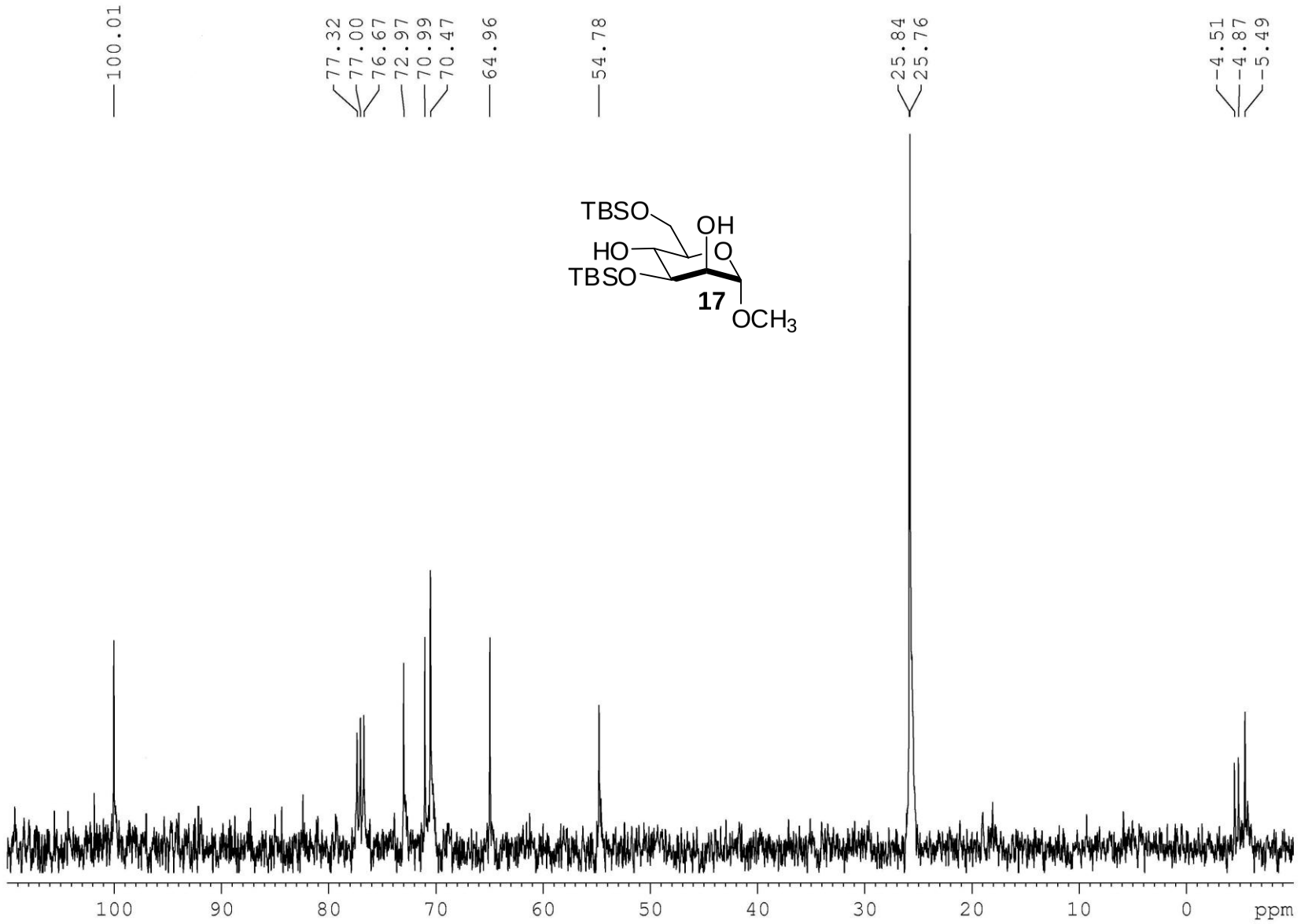
2.11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-5.49
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OH
HO
O
TBSO
17
OCH3
100
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80
70
60
50
40
30
20
10
0
ppm

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3.537
3.545
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3.828
3.840
3.853
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4.614
TBSO
HO
HO
O
TBSO
OCH3
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1.5
2.0
2.5
3.0
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4.0
4.5
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1.00
3.18
3.02
3.02
1.00

4.614
4.605
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3.403
3.385
TBSO
HO
HO
O
TBSO
OCH3
18
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
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2.6
2.4
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HO
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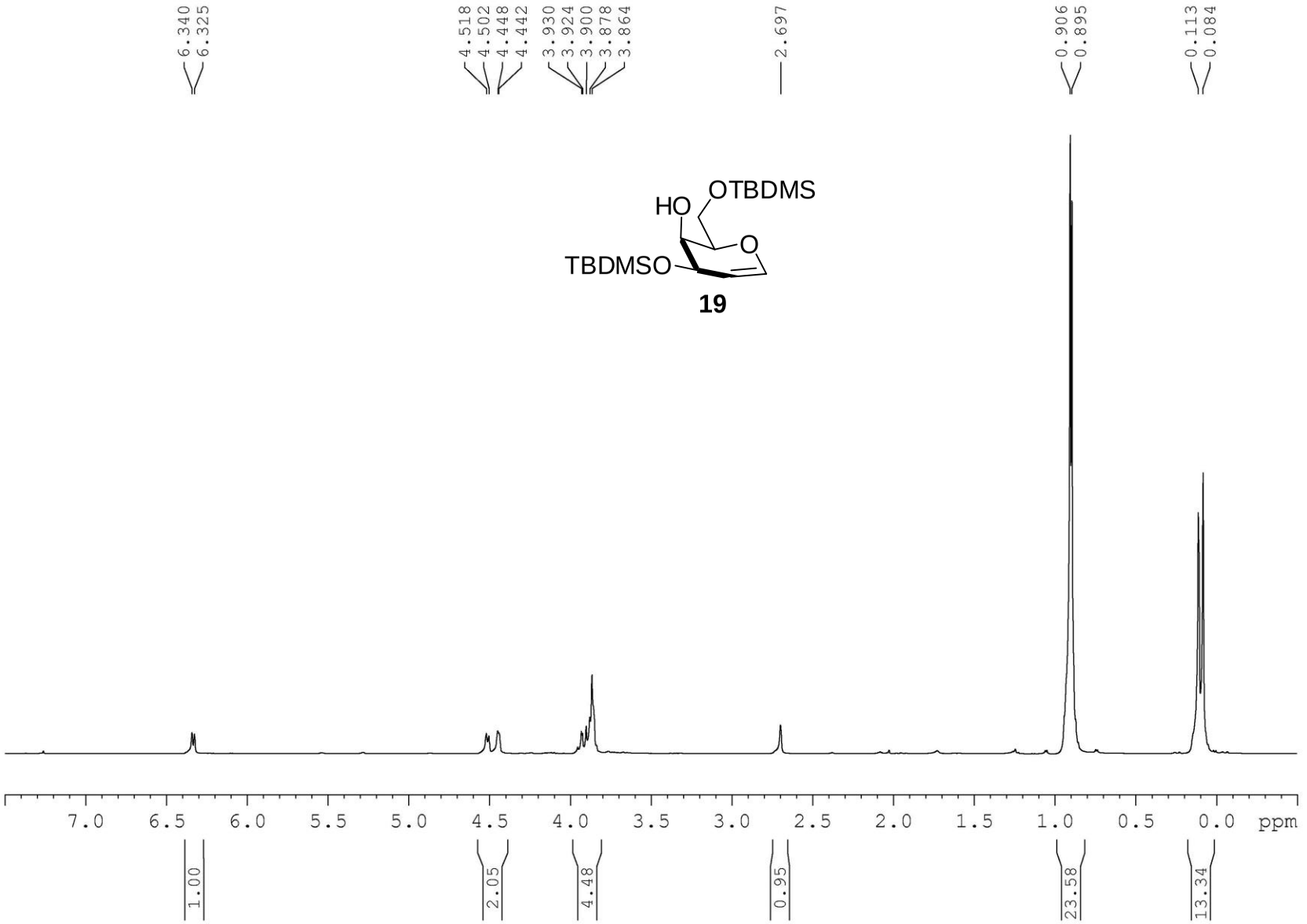
TBSO
OCH3
18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O
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2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.05
4.48
0.95
23.58
13.34

6.340
6.325
4.518
4.502
4.448
4.442
3.930
3.924
3.900
3.878
3.864
2.697
OTBDMS
HO
TBDMSO
19
ppm
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5.0
4.5
4.0
3.5
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1.00
2.05
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140
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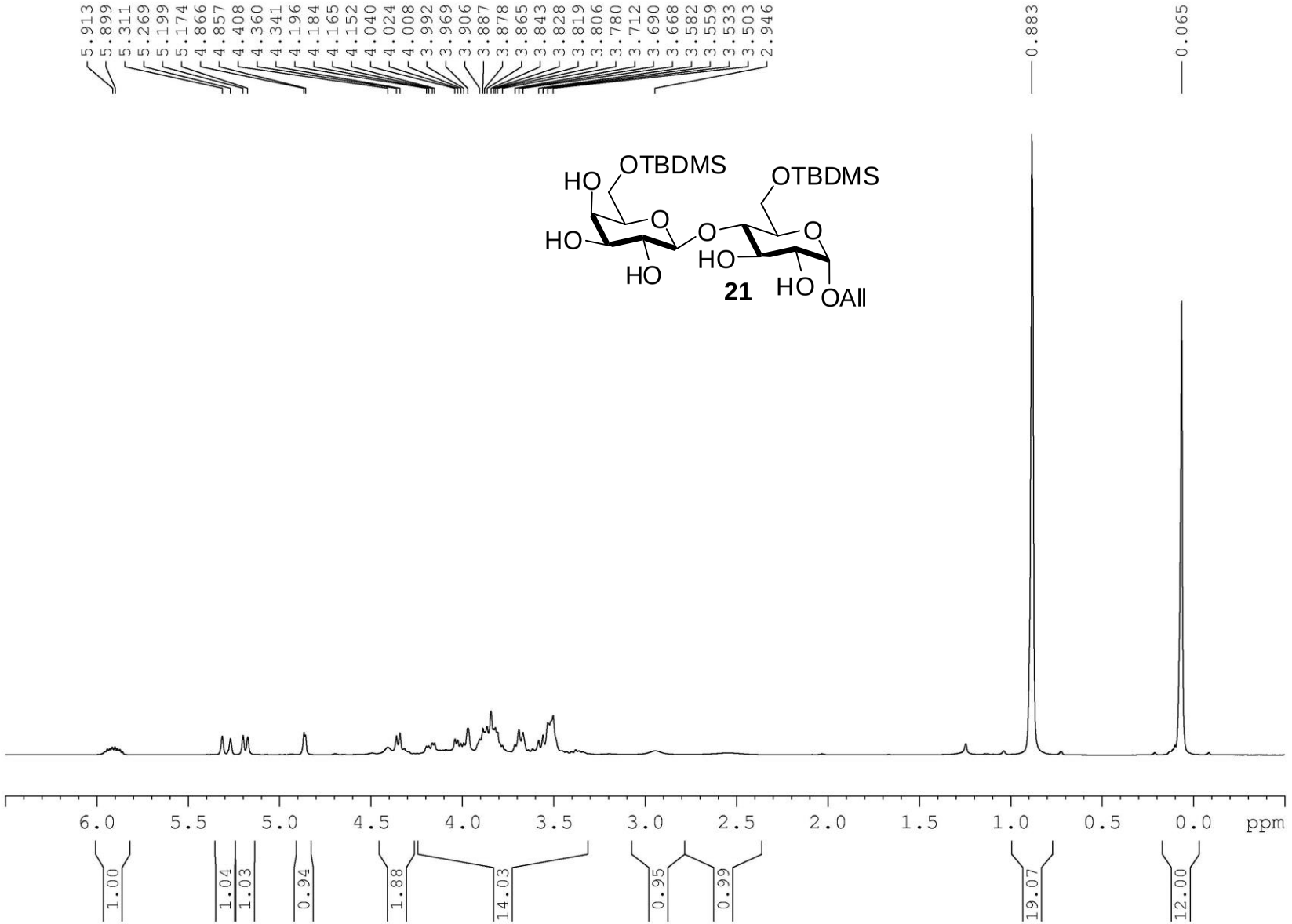
OTBDMS
O
TBDMSO
19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4.184
4.165
4.152
4.040
4.024
4.008
3.992
3.969
3.906
3.887
3.878
3.865
3.843
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HO
OTBDMS
O
HO
HO
O
HO
OTBDMS
O
HO
OAll
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1.03
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1.88
14.03
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19.07
12.00

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5.899
5.311
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2.946
OTBDMS
HO
HO
O
O
HO
HO
OTBDMS
O
HO
HO
O
HO
OAll
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5.0
4.5
4.0
3.5
3.0
2.5
ppm
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1.04
1.03
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1.88
14.03
0.95
0.99

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77.31
79.04
97.11
103.09
117.78
133.72
OTBDMS
OTBDMS
HO
HO
HO
HO
HO
HO
OAll
21
ppm

SEt
O
OH
TBSO
CH3
HO
23
ppm
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4.032
5.303

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-4.59
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17.49
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25.62
25.78
25.87
68.06
73.05
73.39
73.71
83.01
SEt
O
CH3
HO
TBSO
OH
23
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80
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50
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40
35
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25
20
15
10
5
0
-5
ppm

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O
TBSO
OH
CH3
BzO
25
ppm
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6
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1.33
1.02
1.02
0.24
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1.32
2.54

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SEt
O
CH3
BzO
25
TBSO
OH
ppm
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1.32
0.24
1.02
1.02
1.33
0.27
0.29
2.67
1.18

-4.88
-4.95
14.81
17.29
24.94
25.40
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77.30
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129.66
133.09
SEt
O
TBSO
OH
CH3
BzO
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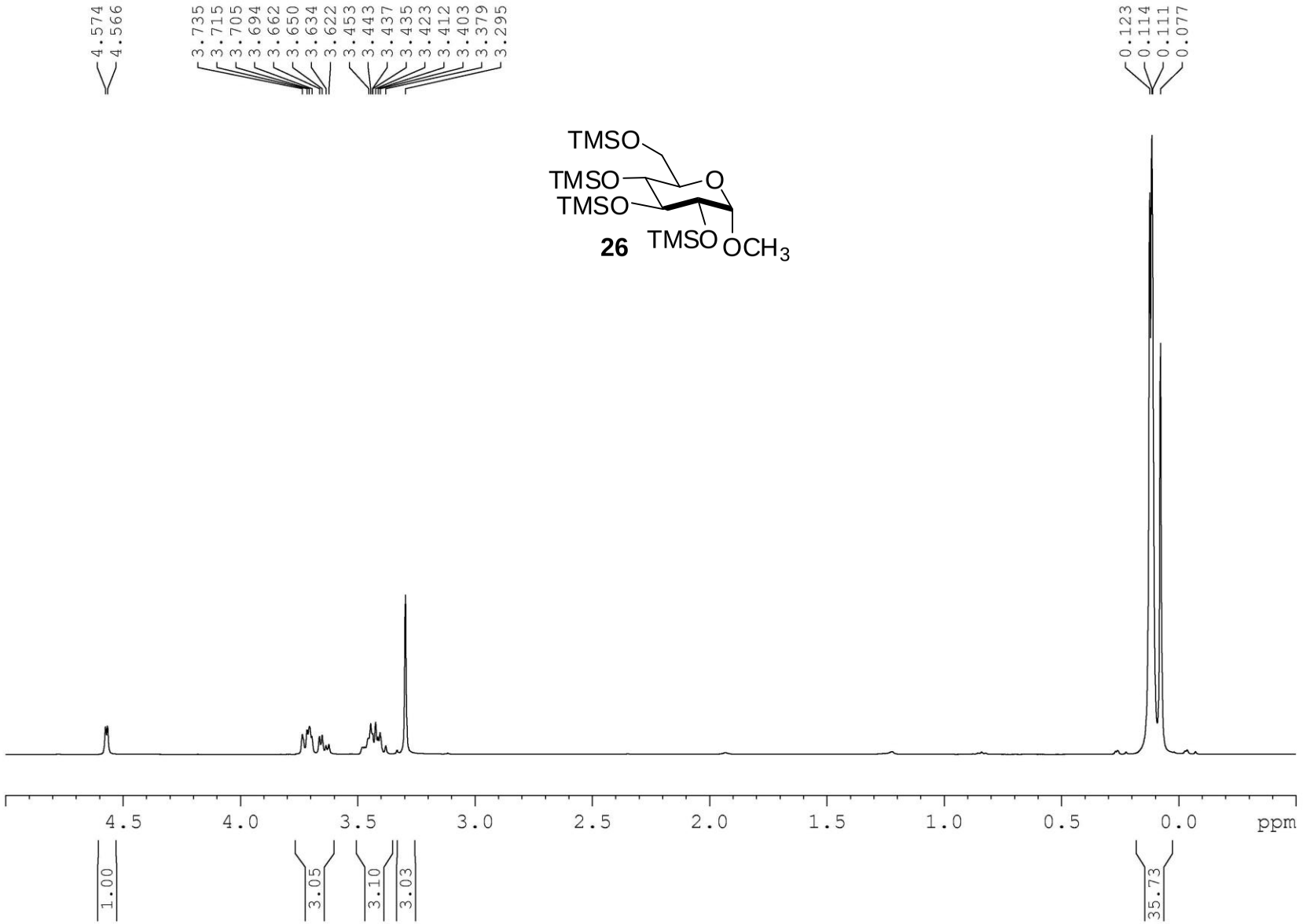
130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3.379
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0.111
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TMSO
TMSO
TMSO
TMSO
O
OCH3
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2.0
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ppm
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3.10
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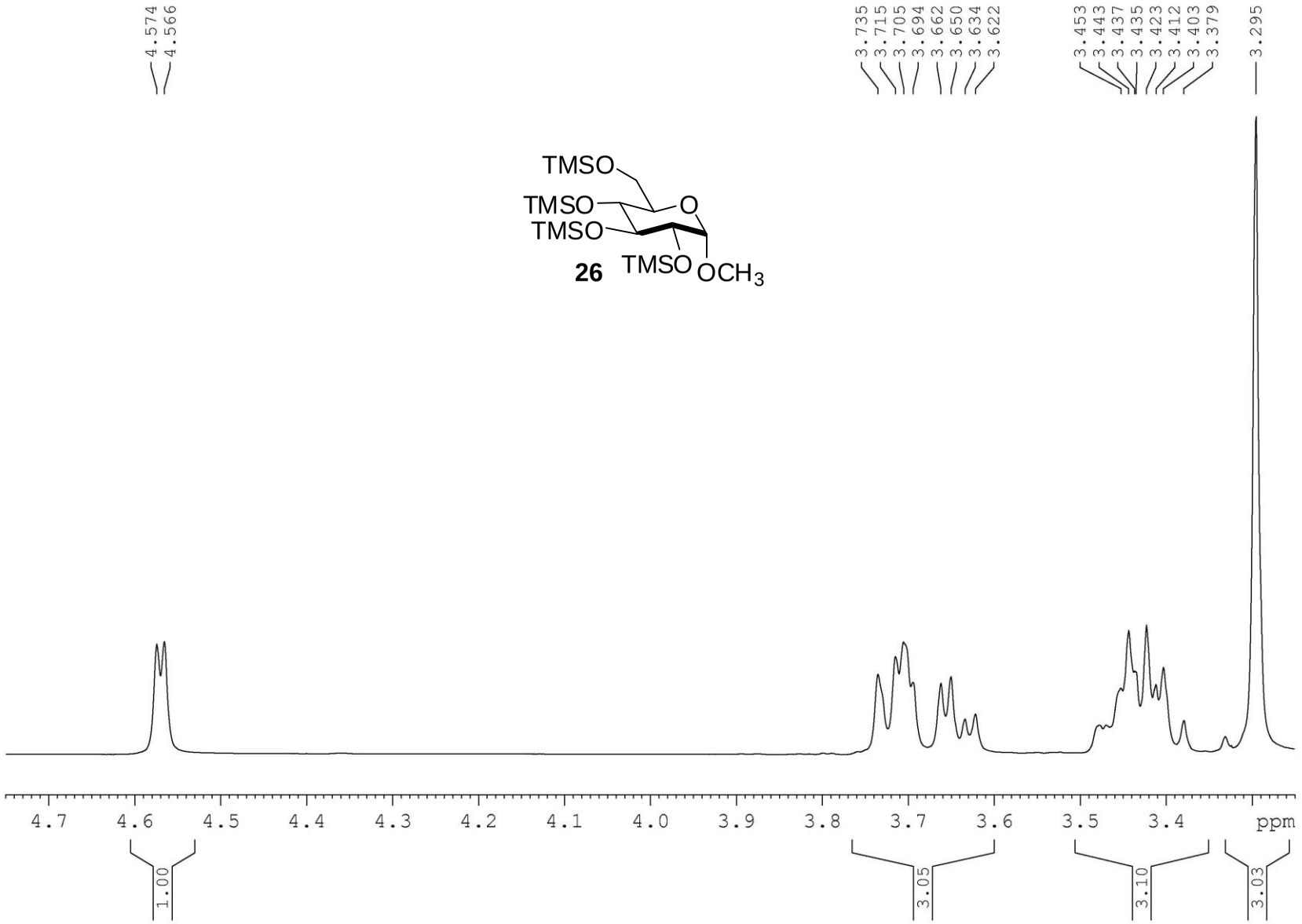
35.73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3.437
3.435
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3.412
3.403
3.379
3.295
TMSO
TMSO
TMSO
TMSO
O
OCH3
26
4.7
4.6
4.5
4.4
4.3
4.2
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ppm
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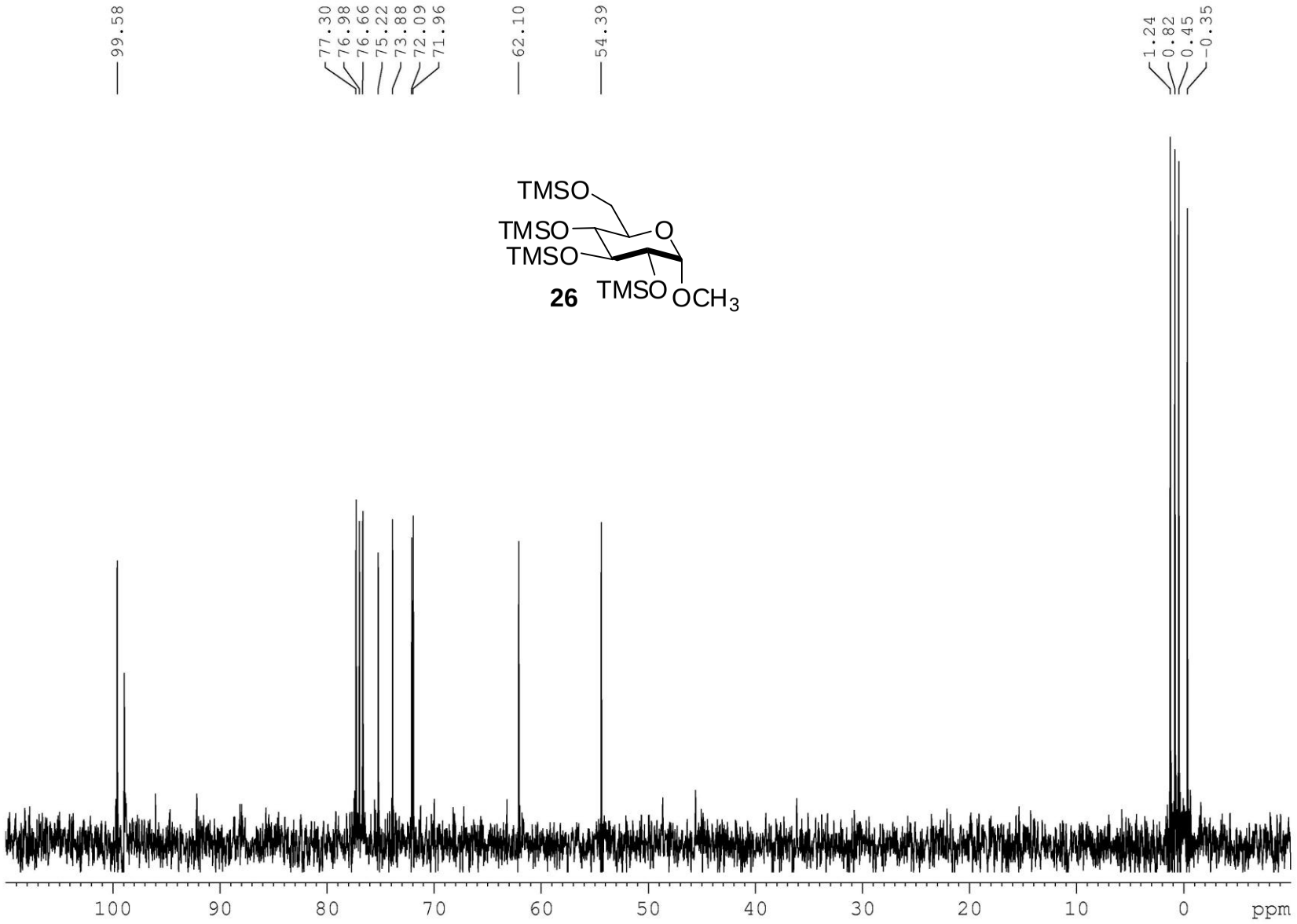
3.03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-0.35
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TMSO
TMSO
TMSO
O
OCH3
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60
50
40
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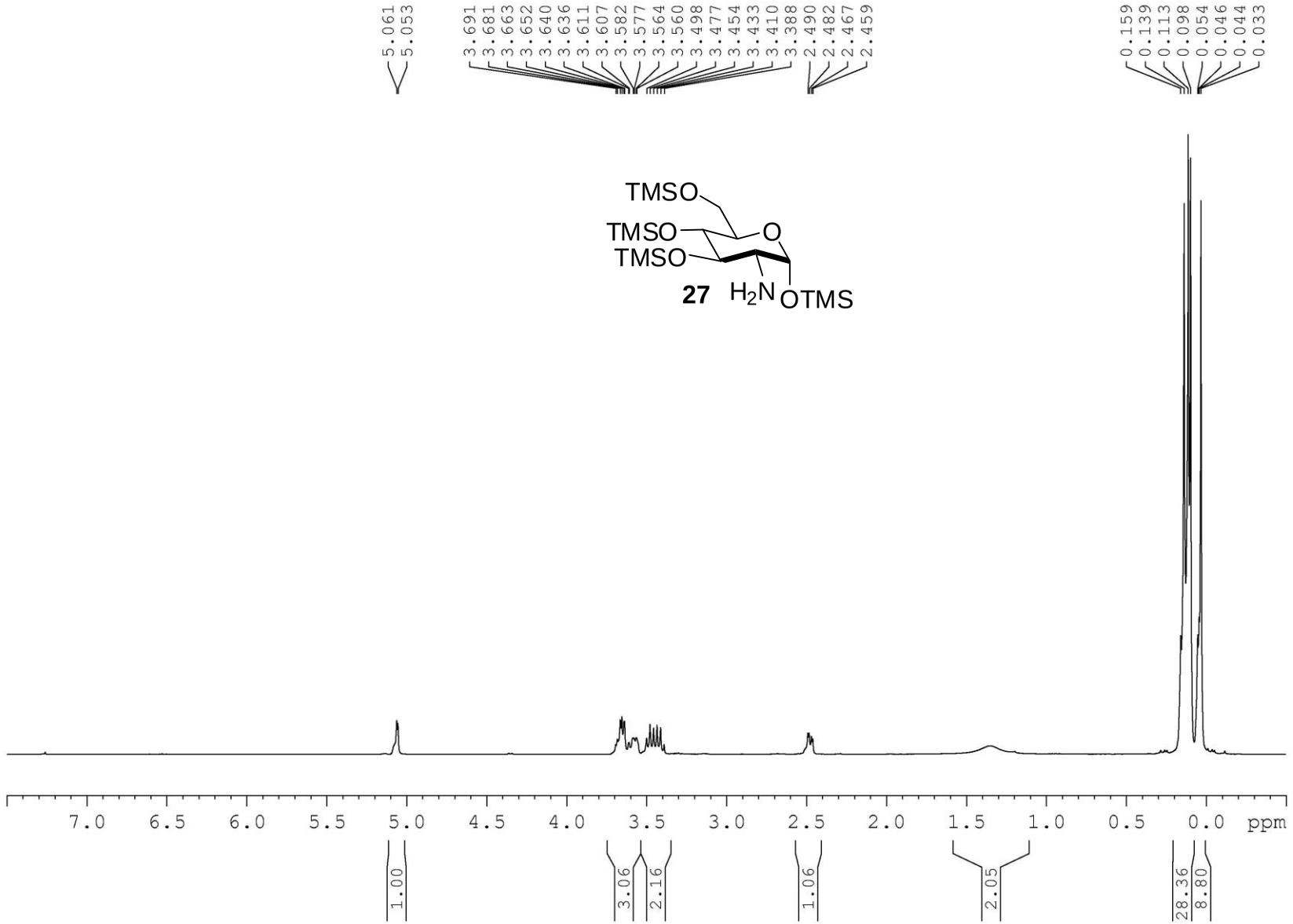
20
10
0
ppm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3.636
3.611
3.607
3.582
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3.560
3.498
3.477
3.454
3.433
3.410
3.388
2.490
2.482
2.467
2.459
0.159
0.139
0.113
0.098
0.054
0.046
0.044
0.033
TMSO
TMSO
TMSO
O
27
H2N
OTMS
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
3.06
2.16
1.06
2.05
28.36
8.80

5.061
5.053
3.691
3.681
3.663
3.652
3.640
3.636
3.611
3.607
3.582
3.577
3.564
3.560
3.498
3.477
3.454
3.433
3.410
3.388
2.490
2.482
2.467
2.459
TMSO
TMSO
TMSO
O
H_2N
OTMS
27
ppm
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
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2.8
2.6
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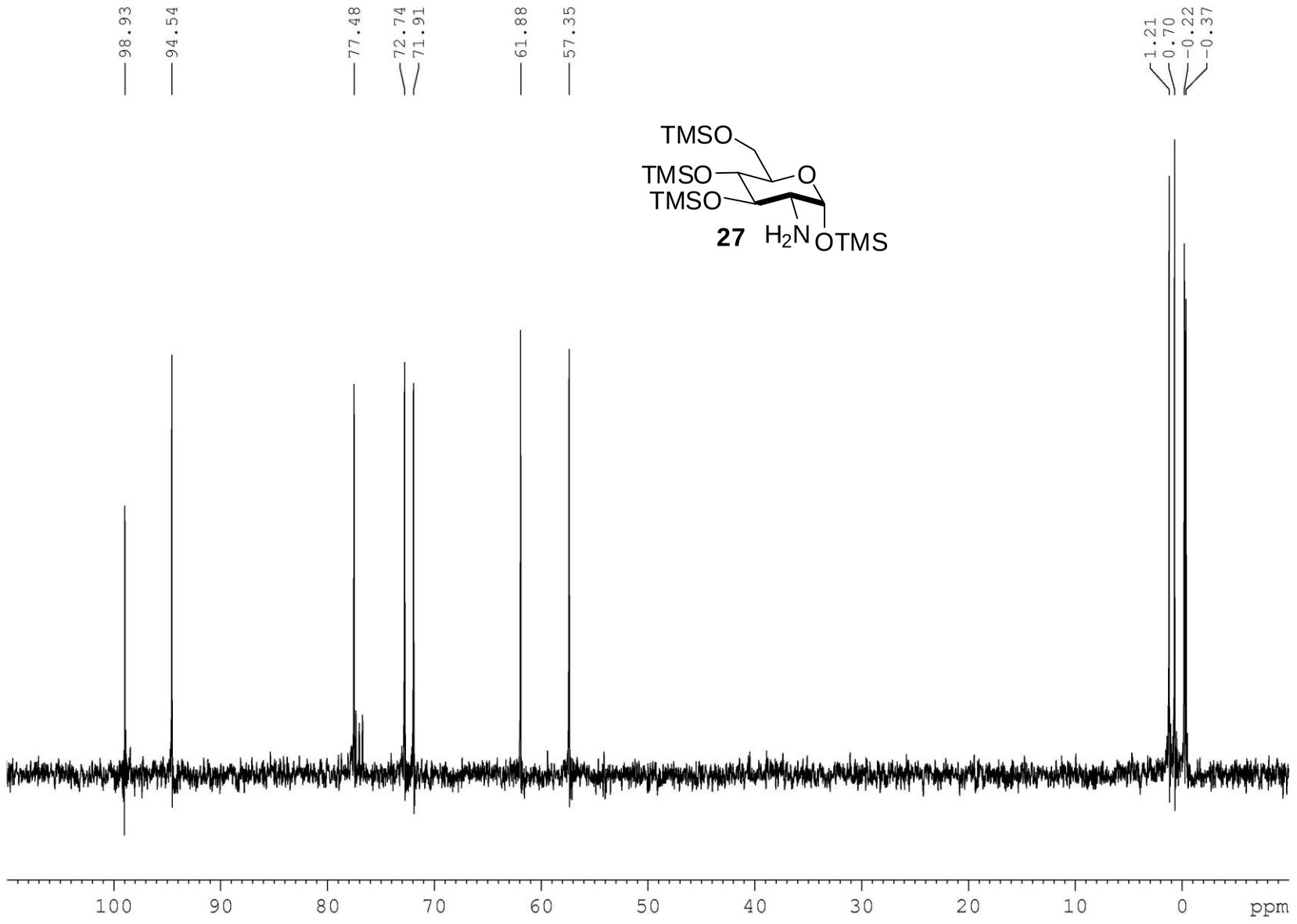
3.06
2.16
1.06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71.91
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-0.22
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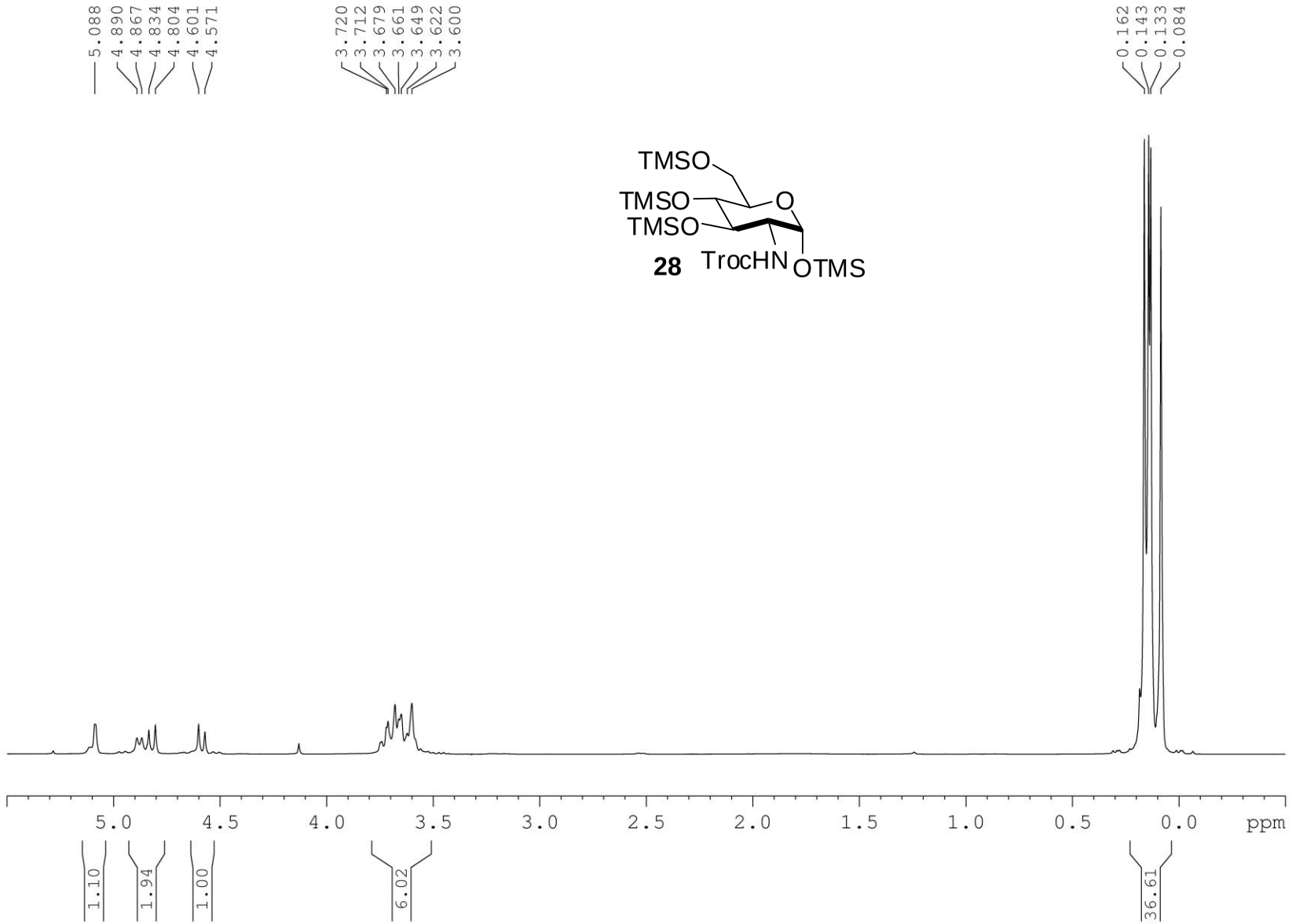
TMSO
TMSO
O
27
H2N
OTMS
100
90
80
70
60
50
40
30
20
10
0
ppm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3.622
3.600
0.162
0.143
0.133
0.084
TMSO
TMSO
TMSO
O
TrocHN
OTMS
28
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.10
1.94
1.00
6.02
36.61

0.91
0.72
-0.24
-0.34
56.81
61.59
71.93
72.41
73.82
74.69
76.66
76.98
77.30
92.51
154.13
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
TMSO
TMSO
TMSO
O
TrocHN
OTMS
28

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7.360
7.343
7.324
7.305
7.289
4.710
4.675
3.945
3.942
3.887
3.874
3.862
3.841
3.679
3.671
3.656
3.648
3.590
3.578
3.566
3.341
0.900
0.089
TBSO
HO
BnO
OH
O
29
OCH3
ppm
0
1
2
3
4
5
6
7
5.92
9.51
0.91
0.87
3.09
2.10
4.12
3.00
4.64

4.710
4.675
3.945
3.942
3.887
3.874
3.862
3.841
3.679
3.671
3.656
3.648
3.590
3.578
3.566
3.341
TBSO
HO
BnO
OH
O
29
OCH3
ppm
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
3.00
4.12
2.10
3.09
0.87
0.91

-5.54
18.18
25.79
54.69
64.40
67.70
68.64
70.99
71.97
76.68
77.00
77.32
79.39
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127.91
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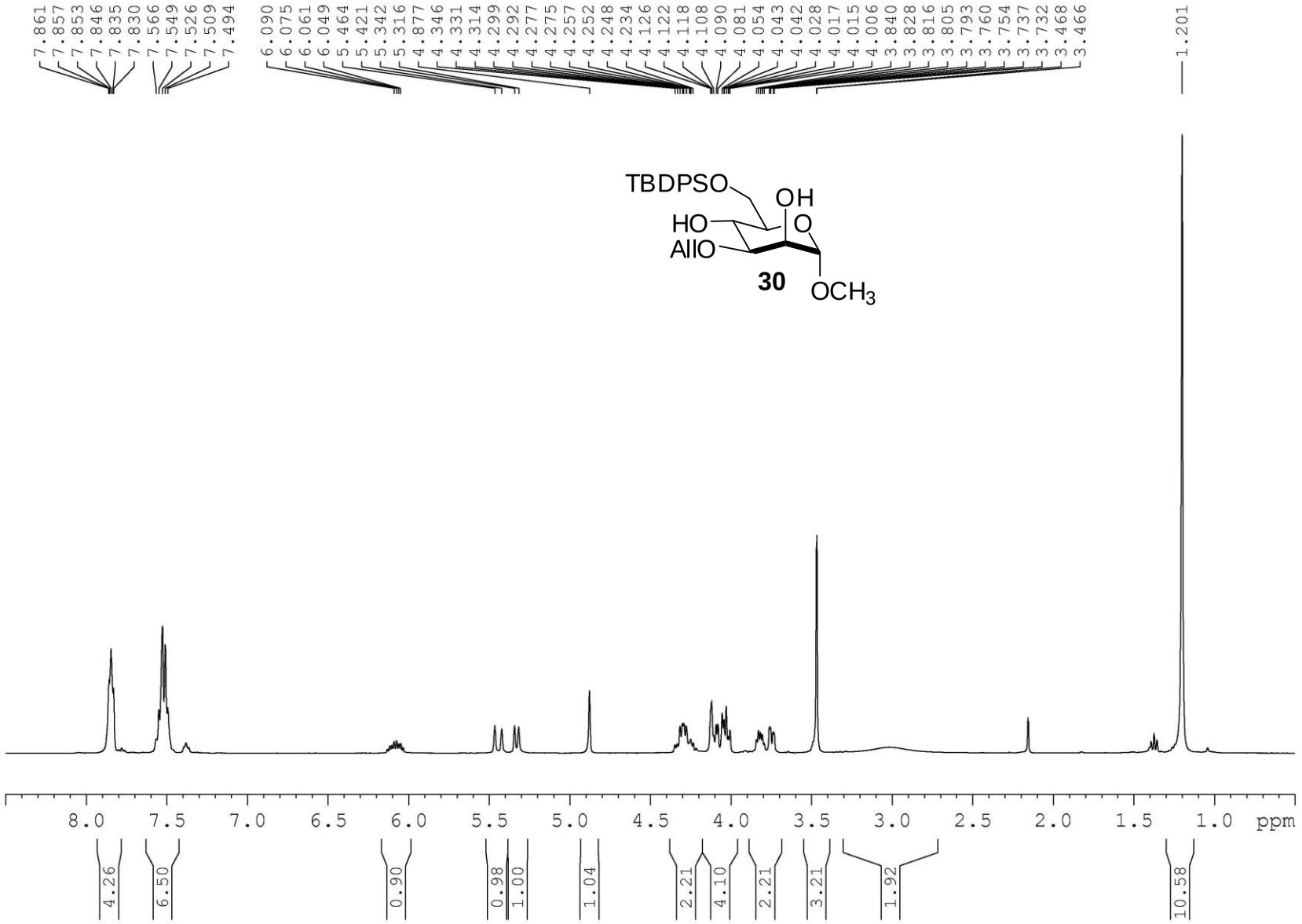
HO
BnO
OH
O
29
OCH3
ppm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7.509
7.494
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6.075
6.061
6.049
5.464
5.421
5.342
5.316
4.877
4.346
4.331
4.314
4.299
4.292
4.277
4.275
4.257
4.252
4.248
4.234
4.126
4.122
4.118
4.108
4.090
4.081
4.054
4.043
4.042
4.028
4.017
4.015
4.006
3.840
3.828
3.816
3.805
3.793
3.760
3.754
3.737
3.732
3.468
3.466
1.201
TBDPSO
OH
HO
O
AllO
30
OCH3
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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0.90
0.98
1.00
1.04
2.21
4.10
2.21
3.21
1.92
10.58

6.090
6.075
6.061
6.049
5.464
5.421
5.342
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4.877
4.346
4.331
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4.108
4.090
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4.028
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3.816
3.805
3.793
3.760
3.754
3.737
3.732
3.468
3.466
TBDPSO
HO
AllO
OH
O
OCH3
30
ppm
6.0
5.5
5.0
4.5
4.0
3.5
3.0
0.90
0.98
1.00
1.04
2.21
4.10
2.21
2.21
3.21
1.92

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134.41
133.00
132.92
129.64
127.60
117.65
100.26
78.98
77.31
76.99
76.67
71.20
70.69
68.25
67.60
64.90
54.62
26.68
19.08
ppm
TBDPSO
HO
AllO
OH
O
OCH3
30

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7.531
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7.506
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4.095
4.080
4.070
4.056
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TBDPSO
HO
BnO
OH
O
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ppm
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1.0
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0.92
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3.807
TBDPSO
HO
BnO
OH
O
31
OCH3
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4.6
4.4
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3.6
3.4
3.2
3.0
2.8
2.91
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2.04
3.00
0.90
0.92

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68.50
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128.53
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135.64
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TBDPSO
HO
BnO
OH
O
31
OCH3
ppm

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7.516
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4.050
4.033
4.024
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3.794
3.782
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3.688
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3.457
1.205
TBDPSO
HO
HO
O
BnO
OCH3
32
ppm
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TBDPSO
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O
BnO
OCH3
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3.08

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ppm
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30
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50
60
70
80
90
100
110
120
130
140
TBDPSO
HO
HO
O
BnO
OCH3
32

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HO
BnO
O
33
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2
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4
5
6
7
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0.96
0.99
3.04
1.00
5.53

6.410
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4.635
4.606
4.209
4.142
3.975
3.967
3.881
3.867
3.848
OTBDMS
HO
BnO
O
33
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5.0
4.5
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3.0
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1.00
1.98
0.96

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HO
BnO
O
33
ppm
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10
20
30
40
50
60
70
80
90
100
110
120
130
140
150

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4.179
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4.052
4.034
2.711
1.222
OTBDPS
HO
BnO
O
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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3.13
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1.10
2.03
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11.23

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4.179
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4.034
2.711
OTBDPS
HO
BnO
O
34
ppm
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
1.00
3.13
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1.10
2.03
0.95

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76.49
76.67
76.99
77.31
99.48
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127.77
127.90
128.49
129.71
134.75
135.52
135.57
144.92
OTBDPS
HO
BnO
O
34
ppm

OTBS
HO
BnO
O
HO
OAll
35
ppm
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1
2
3
4
5
6
7
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9.29
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0.88
1.10
3.07
2.98
0.98
2.06
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5.084
4.906
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3.882
3.776
3.768
3.752
3.744
OTBS
HO
O
HO
OAll
BnO
35
ppm
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6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
1.00
2.01
0.98
2.06
0.98
2.98
3.07
1.10

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70.25
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77.30
78.67
97.55
117.82
127.75
127.88
128.49
133.66
137.97
ppm
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20
30
40
50
60
70
80
90
100
110
120
130
140
OTBS
HO
BnO
O
HO
OAll
35