Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Visible-Light-Induced Photoacid Catalysis: Application in Glycosylation with *O*-Glycosyl Trichloroacetimidates

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1. General Information

All commercially available chemicals were used without further purification unless otherwise noted. Reactions were monitored by thin layer chromatography using TLC silica gel 60-F_{254} plates. TLC plates were visualized by UV fluorescence (254 nm) or stained by Cerium Molybdate followed by heating. Purification of the reaction products was carried out by column chromatography using Siliaflash-P60 (40-63 μm) silica gel available from Silicycle. ^{1}H-NMR spectra were recorded on a BRUKER AV-400 (400 MHz) and ^{13}C-NMR spectra were recorded on a BRUKER AV-400 or AV-600 (100 MHz or 150 MHz). Data for ^{1}H-NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet), coupling constant(s) in Hz and integration. Data for ^{13}C-NMR are reported in terms of chemical shift (δ, ppm). IR spectra were recorded on a PerkinElmer Spectrum Two IR spectrometer and only major peaks were reported in cm^{-1}. High-resolution mass spectral analysis (HRMS) data were obtained using Agilent Technologies 6530 Accurate Mass Q-TOF LC/MS. Irradiation of photochemical reactions was carried out using two 12W PAR38 Blue LED flood lamps from ABi LED lighting. Yields refer to chromatographically and spectroscopically purified compounds.

2. Experimental Procedures and Characterization Date

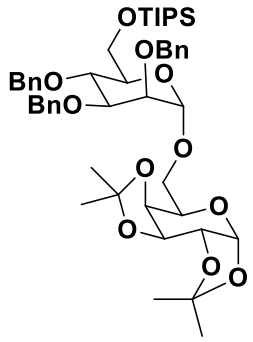
Preparation of Substrates

All glycosyl donors and acceptors were prepared according to the literature procedure.[1-10]

General Glycosylation Procedure

To an oven-dried 1.5 dram vial equipped with a stirbar were added 0.2 mmol glycosyl acceptor, 0.24 mmol glycosyl dornor, 0.004 mmol PhSSPh, 0.002 mmol Eosin Y free acid and 0.5 mL dry dichloromethane. The vial was sealed with a Teflon cap and stirred at room temperature under irradiation with blue LEDs. Upon completion of the reaction (6-8 hours), the solution was concentrated in vacuo, and the residue was purified by flash column chromatograph to afford glycosides.

Compound 17



17

Following the General Glycosylation Procedure: After purification, **17** was obtained as a colourless oil (148 mg, 87%, α/β = 11:1).

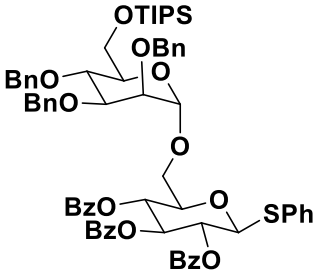
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.31 (m, 15H), 5.56 (d, *J* = 4.8 Hz, 1H), 5.02 (s, 1H), 4.94 (d, *J* = 10.8 Hz, 1H), 4.78 – 4.58 (m, 6H), 4.37 – 4.32 (m, 1H), 4.19 (d, *J* = 7.9 Hz, 1H), 4.10 – 3.92 (m, 5H), 3.86 (s, 1H), 3.77 (dd, *J* = 10.4, 7.3 Hz, 1H), 3.72 – 3.65 (m, 2H), 1.53 (s, 3H), 1.47 (s, 3H), 1.36 (s, 6H), 1.10 – 1.09 (m, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 138.7, 138.5, 128.3, 128.2, 128.1, 128.0, 127.6, 127.6, 127.5, 127.4, 127.3, 109.3, 108.5, 96.5, 96.3, 80.1, 75.1, 74.9, 74.7, 73.6, 72.2, 72.0, 70.9, 70.7, 70.6, 65.0, 64.7, 62.9, 26.1, 25.9, 24.9, 24.5, 18.0, 18.0, 12.0;

IR (CH_2Cl_2, cm^{-1}) 2939, 2665, 1725, 1496, 1454, 1381, 1255, 1210, 1096, 1066, 1027, 882, 735.;

ESI HRMS calcd for $C_{48}H_{68}NaO_{11}Si$ $[M + Na]^+$ 871.4423, found 871.4427.

Compound 18



18

Following the General Glycosylation Procedure: After purification, **18** was obtained as a white solid (211 mg, 90%, α/β = 21:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.5 Hz, 2H), 7.97 (d, *J* = 7.6 Hz, 2H), 7.89 (d, *J* = 7.5 Hz, 2H), 7.57 (dd, *J* = 9.9, 4.3 Hz, 3H), 7.50 – 7.28 (m, 26H), 5.97 (t, *J* = 9.6 Hz, 1H), 5.64 (t, *J* = 9.6 Hz, 1H), 5.57 (t, *J* = 9.7 Hz, 1H), 5.09 (d, *J* = 10.0 Hz, 1H), 5.01 (d, *J* = 11.0 Hz, 1H), 4.96 (s, 1H), 4.81 – 4.69 (m, 3H), 4.55 (q, *J* = 11.6 Hz, 2H), 4.05 (t, *J* = 9.6 Hz, 3H), 3.94 (dd, *J* = 13.8, 2.6 Hz, 3H), 3.75 (d, *J* = 9.8 Hz, 2H), 3.64 (d, *J* = 9.7 Hz, 1H), 1.14 (s, 21H).

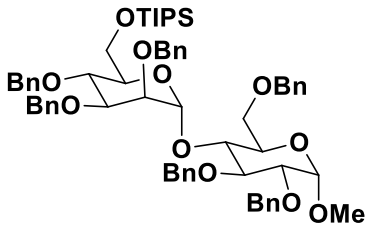
^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 165.0, 165.0, 138.8, 138.6, 138.5, 133.2, 133.1, 131.7, 129.7, 129.6, 129.1, 128.9, 128.7, 128.3, 128.2, 128.1, 128.1, 127.9, 127.8, 127.6, 127.5, 127.4,

127.3, 97.9, 86.3, 80.0, 76.9, 74.9, 74.4, 74.2, 73.5, 72.5, 72.1, 70.7, 69.9, 66.4, 62.9, 18.1, 18.0, 12.0.

IR (CH_2Cl_2, cm^{-1}) 2941, 2865, 1733, 1601, 1584, 1495, 1452, 1315, 1276, 1263, 1089, 1067, 1026, 882, 733, 706.;

ESI HRMS calcd for $C_{69}H_{76}NaO_{13}SSi$ $[M + Na]^+$ 1195.4668, found 1195.4669.

Compound 19



19

Following the General Glycosylation Procedure: After purification, 1**9** was obtained as a white solid (132 mg, 63%, α/β = 18:1).

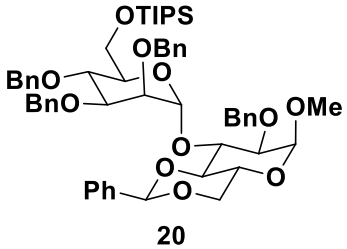
^1H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.17 (m, 30H), 5.28 (s, 1H), 5.08 (d, *J* = 11.4 Hz, 1H), 4.91 (d, *J* = 10.8 Hz, 1H), 4.72 -4.58 (m, 7H), 4.50 (t, *J* = 12.3 Hz, 2H), 4.37 (d, *J* = 12.2 Hz, 1H), 4.26 (d, *J* = 12.2 Hz, 1H), 4.06 (d, *J* = 9.5 Hz, 1H), 3.93 – 3.73 (m, 9H), 3.66 (d, *J* = 8.7 Hz, 1H), 3.55 (dd, *J* = 9.5, 2.6 Hz, 1H), 3.41 (s, 3H), 1.04 (s, 21H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 138.8, 138.7, 138.7, 138.3, 137.9, 128.4, 128.3, 128.3, 128.2, 128.1, 128.0, 127.9, 127.9, 127.6, 127.5, 127.4, 127.4, 127.3, 127.1, 127.0, 126.7, 100.1, 97.7, 81.5, 80.0, 79.9, 76.2, 75.0, 74.4, 74.3, 73.3, 73.2, 72.1, 72.0, 69.9, 69.4, 62.8, 55.2, 18.0, 17.9, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2922, 2864, 1732, 1496, 1453, 1360, 1098, 1045, 1027, 882, 733, 696.;

ESI HRMS calcd for $C_{64}H_{80}NaO_{11}Si$ $[M + Na]^+$ 1075.5362, found 1075.5367.

Compound 20



20

Following the General Glycosylation Procedure: After purification, **20** was obtained as a colourless oil (131 mg, 86%, α/β = 18:1).

^1H NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, *J* = 7.1, 2.3 Hz, 2H), 7.49 – 7.15 (m, 23H), 5.52 (s, 1H), 5.43 (s, 1H), 4.95 (d, *J* = 11.0 Hz, 1H), 4.79 -4.3 (m, 3H), 4.68 (d, *J* = 4.2 Hz, 2H), 4.50 (d, *J* = 11.8 Hz, 1H), 4.47 – 4.35 (m, 2H), 4.24 (ddd, *J* = 14.8, 9.9, 5.2 Hz, 3H), 4.05 – 3.75 (m,

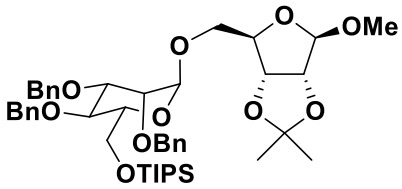
6H), 3.69 (d, *J* = 10.2 Hz, 1H), 3.51 (t, *J* = 9.4 Hz, 1H), 3.44 (d, *J* = 3.6 Hz, 1H), 3.41 (s, 3H), 1.08 (s, 21H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.5, 138.9, 138.6, 137.8, 137.2, 129.2, 128.4, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.7, 127.6, 127.4, 127.3, 127.2, 127.0, 126.1, 101.6, 99.0, 97.8, 82.8, 79.5, 78.1, 75.0, 74.8, 74.3, 73.7, 73.2, 71.8, 71.5, 69.1, 62.7, 61.8, 18.1, 18.0, 12.1.;

IR (CH_2Cl_2, cm^{-1}) 2936, 1703, 1496, 1453, 1365, 1280, 1209, 1087, 1045, 1026, 992, 912, 881, 733, 695.;

ESI HRMS calcd for $C_{57}H_{72}NaO_{11}Si$ $[M + Na]^+$ 983.4736, found 983.4746.

Compound 21



21

Following the General Glycosylation Procedure: After purification, **21** was obtained as a colourless oil (130 mg, 82%, α/β = 13:1).

Data for α:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.30 (m, 15H), 4.96 (d, *J* = 10.9 Hz, 2H), 4.86 (s, 1H), 4.79 (d, *J* = 12.4 Hz, 1H), 4.74 – 4.66 (m, 4H), 4.64 (d, *J* = 5.9 Hz, 1H), 4.57 (d, *J* = 5.9 Hz, 1H), 4.31 (dd, *J* = 8.2, 5.5 Hz, 1H), 3.97 (q, *J* = 10.5 Hz, 4H), 3.81 (s, 1H), 3.76 (dd, *J* = 10.1, 5.3 Hz, 1H), 3.69 – 3.64 (m, 1H), 3.37 (t, *J* = 9.5 Hz, 1H), 3.29 (s, 3H), 1.52 (s, 3H), 1.36 (s, 3H), 1.10 (s, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 138.6, 138.5, 128.4, 128.3, 128.3, 128.1, 127.7, 127.6, 127.5, 127.5, 112.3, 109.4, 98.2, 85.3, 84.8, 82.2, 80.2, 75.2, 75.1, 74.8, 73.8, 72.6, 72.3, 68.1, 54.8, 26.5, 25.1, 18.0, 18.0, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2939, 2864, 1730, 1496, 1454, 1381, 1370, 1272, 1209, 1093, 1047, 1027, 1013, 870, 734, 696.;

ESI HRMS calcd for $C_{45}H_{64}NaO_{10}Si$ $[M + Na]^+$ 815.4166, found 815.4171.

Data for β:

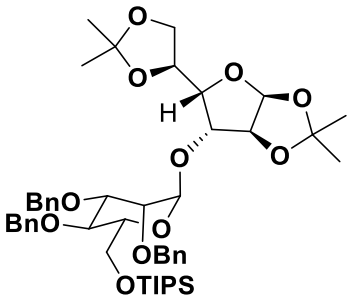
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.1 Hz, 2H), 7.32 – 7.26 (m, 13H), 4.96 (dd, *J* = 11.4, 4.4 Hz, 3H), 4.86 (d, *J* = 12.3 Hz, 1H), 4.73 (d, *J* = 5.9 Hz, 1H), 4.66 (d, *J* = 10.8 Hz, 1H), 4.62 – 4.51 (m, 3H), 4.41 (s, 1H), 4.34 (dd, *J* = 9.1, 6.1 Hz, 1H), 4.04 – 3.85 (m, 5H), 3.55 – 3.45 (m, 2H), 3.28 (s, 4H), 1.51 (s, 3H), 1.31 (s, 3H), 1.08 (s, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.9, 138.5 138.2, 128.4, 128.1, 128.0, 127.6, 127.6, 127.2, 112.2, 109.4, 101.2, 85.1, 84.9, 82.2, 82.1, 75.2, 74.6, 74.0, 73.8, 71.5, 69.1, 62.9, 54.7, 26.4, 24.8,, 17.9, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2939, 2864, 1454, 1369, 1107, 1063, 871, 736, 696.;

ESI HRMS calcd for $C_{45}H_{64}NaO_{10}Si$ $[M + Na]^+$ 815.4166, found 815.4171.

Compound 22



22

Following the General Glycosylation Procedure: After purification, **22** was obtained as a colourless oil (137 mg, 81%, α/β = 12:1).

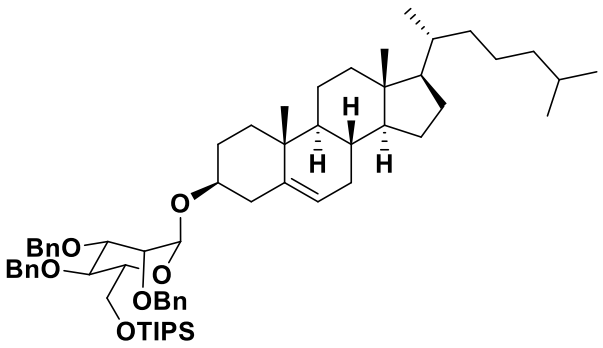
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 15H), 5.80 (d, *J* = 3.0 Hz, 1H), 5.18 (s, 1H), 4.94 (d, *J* = 10.8 Hz, 1H), 4.68 – 4.57 (m, 5H), 4.27 (s, 1H), 4.11 – 3.93 (m, 8H), 3.85 (d, *J* = 9.5 Hz, 1H), 3.76 (s, 1H), 3.68 (dd, *J* = 9.1, 5.2 Hz, 1H), 1.49 (s, 3H), 1.41 (s, 3H), 1.33 (s, 3H), 1.28 (s, 3H), 1.09 (s, 21H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.5, 138.4, 138.3, 128.4, 128.3, 128.3, 128.2, 127.8, 127.7, 127.6, 127.5, 111.9, 109.2, 105.3, 98.7, 83.9, 81.5, 80.3, 79.6, 75.3, 74.7, 74.7, 74.5, 72.5, 72.2, 72.1, 67.7, 63.3, 26.9, 26.8, 26.2, 25.5, 18.0, 18.0, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2939, 2865, 1496, 1454, 1381, 1371, 1250, 1214, 1164, 1027, 1015, 916, 881, 844, 734, 696.;

ESI HRMS calcd for $C_{48}H_{68}NaO_{11}Si$ $[M + Na]^+$ 871.4423, found 871.4432.

Compound 23



23

Following the General Glycosylation Procedure: After purification, **23** was obtained as a colourless oil (154 mg, 79%, α/β = 4:1).

Data for α:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 15H), 5.30 (d, *J* = 4.3 Hz, 1H), 5.01 (s, 1H), 4.93 (d, *J* = 10.8 Hz, 1H), 4.77 (d, *J* = 12.4 Hz, 1H), 4.66 (dd, *J* = 19.9, 12.3 Hz, 4H), 3.97 (dd, *J* =

9.5, 3.2 Hz, 2H), 3.93 – 3.86 (m, 2H), 3.76 (s, 1H), 3.74 – 3.68 (m, 1H), 3.57 – 3.47 (m, 1H), 2.26 (d, *J* = 11.4 Hz, 2H), 2.00 (t, *J* = 17.6 Hz, 2H), 1.83 (d, *J* = 10.4 Hz, 3H), 1.57 – 1.25 (m, 11H), 1.22 – 0.97 (m, 34H), 0.93 (d, *J* = 6.5 Hz, 4H), 0.88 (dd, *J* = 6.6, 1.6 Hz, 7H), 0.69 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 138.7, 138.6, 138.6, 128.3, 128.3, 128.2, 128.1, 127.7, 127.6, 127.4, 121.7, 94.6, 80.5, 75.5, 75.2, 75.1, 73.5, 72.5, 72.1, 63.4, 56.8, 56.1, 50.1, 42.3, 39.7, 39.5, 36.9, 36.7, 36.2, 35.8, 31.9, 31.8, 28.2, 28.0, 27.1, 24.3, 23.8, 22.8, 22.5, 21.0, 19.3, 18.7, 18.1, 18.0, 12.0, 11.8.;

IR (CH_2Cl_2, cm^{-1}) 2936, 2864, 1705, 1496, 1454, 1364, 1255, 1205, 1096, 1049, 1027, 882, 733, 695.;

ESI HRMS calcd for $C_{63}H_{94}NaO_6Si$ $[M + Na]^+$ 997.6717, found 997.6698.

Data for β:

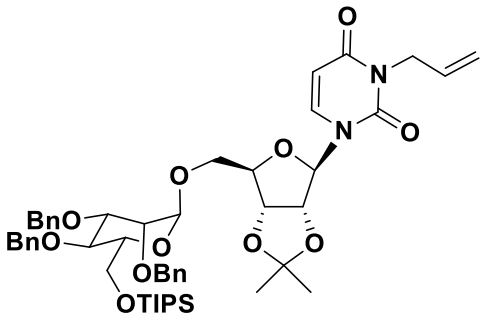
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 6.8 Hz, 2H), 7.31 – 7.27 (m, 13H), 5.34 (s, 1H), 4.93 (dt, *J* = 21.2, 12.5 Hz, 3H), 4.72 – 4.45 (m, 4H), 3.99 (d, *J* = 10.8 Hz, 1H), 3.92 – 3.79 (m, 3H), 3.66 – 3.48 (m, 2H), 3.33 – 3.22 (m, 1H), 2.26 (dt, *J* = 24.2, 10.8 Hz, 2H), 2.00 (t, *J* = 15.9 Hz, 3H), 1.83 (d, *J* = 13.2 Hz, 2H), 1.64 – 1.25 (m, 12H), 1.22 – 0.99 (m, 33H), 0.93 (d, *J* = 6.5 Hz, 4H), 0.88 (dd, *J* = 6.6, 1.5 Hz, 7H), 0.69 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 139.2, 138.7, 138.4, 128.3, 128.1, 127.9, 127.6, 127.5, 127.2, 121.7, 99.5, 82.4, 78.1, 75.1, 74.9, 74.3, 73.6, 71.3, 63.2, 56.7, 56.1, 50.2, 42.3, 39.8, 39.5, 38.8, 37.3, 36.8, 36.2, 35.8, 32.0, 31.9, 29.7, 28.2, 28.0, 24.3, 23.8, 22.8, 22.5, 21.0, 19.4, 18.7, 18.0, 18.0, 12.0, 11.8.;

IR (CH_2Cl_2, cm^{-1}) 2937, 2864, 1729, 1496, 1454, 1364, 1105, 1065, 1027, 882, 733, 695.;

ESI HRMS calcd for $C_{63}H_{94}NaO_6Si$ $[M + Na]^+$ 997.6717, found 997.6698.

Compound 24



24

Following the General Glycosylation Procedure: After purification, **24** was obtained as a colourless oil (158 mg, 87%, α/β = 27:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 15H), 5.89 (dq, *J* = 10.6, 5.9 Hz, 1H), 5.82 (s, 1H), 5.50 (d, *J* = 8.1 Hz, 1H), 5.29 (d, *J* = 17.1 Hz, 1H), 5.18 (d, *J* = 10.2 Hz, 1H), 4.95 (d, *J* = 10.8 Hz, 1H), 4.86 (s, 1H), 4.79 (d, *J* = 12.4 Hz, 1H), 4.73 – 4.59 (m, 7H), 4.59 – 4.52 (m,

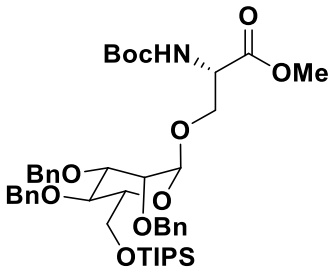
2H), 4.41 (s, 1H), 4.06 – 3.85 (m, 4H), 3.76 (d, *J* = 9.4 Hz, 1H), 3.67 (s, 1H), 3.60 (d, *J* = 10.5 Hz, 1H), 3.54 (d, *J* = 5.4 Hz, 1H), 1.60 (s, 3H), 1.37 (s, 3H), 1.09 (s, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 150.4, 138.3, 138.2, 138.0, 131.2, 128.3, 128.1, 127.7, 127.5, 118.3, 114.0, 101.2, 97.9, 93.9, 85.3, 85.1, 80.7, 79.5, 75.2, 74.6, 74.4, 74.2, 72.6, 72.0, 66.9, 62.8, 42.9, 28.0, 25.3, 18.0, 17.9, 11.9;

IR (CH_2Cl_2, cm^{-1}) 2941, 2865, 1710, 1665, 1496, 1453, 1383, 1336, 1266, 1210, 1070, 1027, 881, 805, 733, 697.;

ESI HRMS calcd for $C_{51}H_{68}N_2NaO_{11}Si$ $[M + Na]^+$ 935.4485, found 935.4487.

Compound 25



25

Following the General Glycosylation Procedure: After purification, **25** was obtained as a colourless oil (140 mg, 87%, α only).

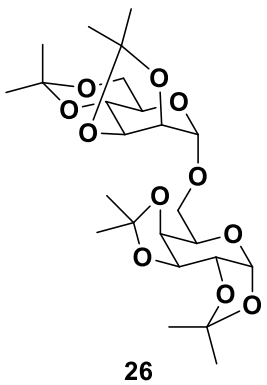
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 15H), 5.25 (d, *J* = 7.9 Hz, 1H), 4.91 (d, *J* = 10.9 Hz, 1H), 4.76 – 4.62 (m, 6H), 4.46 (d, *J* = 8.1 Hz, 1H), 4.04 – 3.83 (m, 6H), 3.68 – 3.66 (m, 4H), 3.57 (d, *J* = 9.4 Hz, 1H), 1.47 (s, 9H), 1.09 (s, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 155.4, 138.6, 138.5, 138.4, 128.3, 128.2, 128.0, 127.8, 127.6, 127.5, 98.5, 80.1, 79.7, 75.0, 74.5, 73.9, 72.6, 72.4, 68.0, 62.9, 53.8, 52.4, 28.3, 18.0, 17.9, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2941, 2865, 1749, 1715, 1496, 1454, 1208, 1161, 1103, 1055, 1027, 881, 733, 696.;

ESI HRMS calcd for $C_{45}H_{65}NNaO_{10}Si$ $[M + Na]^+$ 830.4270, found 830.4280.

Compound 26



26

Following the General Glycosylation Procedure: After purification, **26** was obtained as a colourless oil (90 mg, 90%, α/β > 30:1).

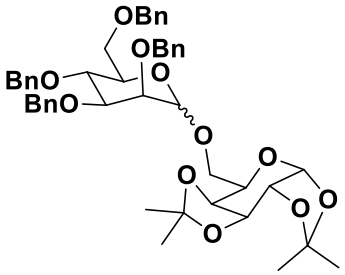
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.55 (d, *J* = 5.0 Hz, 1H), 5.04 (s, 1H), 4.80 (dd, *J* = 5.9, 3.6 Hz, 1H), 4.65 – 4.61 (m, 2H), 4.40 (ddd, *J* = 8.0, 6.2, 4.3 Hz, 1H), 4.33 (dd, *J* = 5.0, 2.5 Hz, 1H), 4.23 (dd, *J* = 7.9, 1.9 Hz, 1H), 4.11 (dd, *J* = 8.7, 6.2 Hz, 1H), 4.06 (dd, *J* = 8.7, 4.3 Hz, 1H), 3.98 (ddd, *J* = 8.5, 7.4, 2.7 Hz, 2H), 3.76 (dd, *J* = 10.5, 6.7 Hz, 1H), 3.66 (dd, *J* = 10.5, 6.8 Hz, 1H), 1.55 (s, 3H), 1.48 (s, 3H), 1.46 (d, *J* = 1.3 Hz, 6H), 1.39 (s, 3H), 1.36 (s, 3H), 1.34 (s, 3H), 1.34 (s, 3H).

δ^{13}C NMR (125 MHz, $CDCl_3$) δ 112.5, 109.3, 109.1, 108.5, 106.6, 96.2, 85.0, 80.3, 79.4, 73.0, 70.9, 70.5, 70.4, 66.8, 66.0, 26.8, 26.0, 25.9, 25.8, 25.2, 24.8, 24.5, 24.4.;

IR (CH_2Cl_2, cm^{-1}) 3366, 3241, 2987, 1733, 1690, 1615, 1456, 1381, 1353, 1210, 1165, 1107, 1068, 1001, 830, 733, 617.;

ESI HRMS calcd for $C_{24}H_{38}NaO_{11}$ $[M + Na]^+$ 525.2306, found 525.2306.

Compound 27



27

Following the General Glycosylation Procedure: After purification, **27** was obtained as a colourless oil (128 mg, 82%, α/β = 3:1).

Data for α:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 18H), 7.20 – 7.18 (m, 2H), 5.55 (d, *J* = 5.0 Hz, 1H), 5.05 (s, 1H), 4.90 (d, *J* = 10.8 Hz, 1H), 4.77 (s, 2H), 4.71 (d, *J* = 12.2 Hz, 1H), 4.64 – 4.60 (m, 3H), 4.55 (dd, *J* = 11.3, 8.0 Hz, 2H), 4.34 (dd, *J* = 4.9, 2.2 Hz, 1H), 4.19 (d, *J* = 7.9 Hz, 1H), 4.03 (dt, *J* = 12.8, 7.4 Hz, 2H), 3.94 (dd, *J* = 9.4, 2.9 Hz, 1H), 3.83 (dd, *J* = 18.0, 9.1 Hz, 4H), 3.71 (dt, *J* = 17.3, 5.5 Hz, 2H), 1.54 (s, 3H), 1.46 (s, 3H), 1.36 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 138.6, 138.5, 138.4, 128.3, 128.0, 127.8, 127.7, 127.6, 127.5, 127.5, 109.4, 108.6, 97.3, 96.4, 80.1, 75.1, 74.9, 74.7, 73.4, 72.4, 72.0, 70.9, 70.6, 70.6, 69.1, 65.5, 65.4, 26.2, 26.0, 25.0, 24.6.;

IR (CH_2Cl_2, cm^{-1}) 2916, 1730, 1496, 1453, 1370, 1381, 1254, 1167, 1209, 1096, 1065, 1027, 899, 917, 733, 696.;

ESI HRMS calcd for $C_{46}H_{54}NaO_{11}$ $[M + Na]^+$ 805.3558, found 805.3564.

Data for β:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.5 Hz, 2H), 7.37 – 7.27 (m, 16H), 7.18 (d, *J* = 7.1 Hz, 2H), 5.61 (d, *J* = 4.9 Hz, 1H), 5.03 (d, *J* = 12.4 Hz, 1H), 4.93 (dd, *J* = 11.7, 5.2 Hz, 2H),

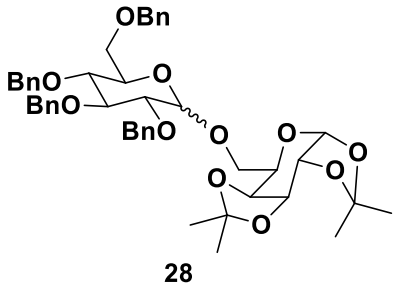
4.70 – 4.42 (m, 6H), 4.39 – 4.32 (m, 2H), 4.24 (dd, *J* = 7.4, 5.1 Hz, 2H), 4.13 (d, *J* = 7.9 Hz, 1H), 4.02 (s, 1H), 3.91 (t, *J* = 9.5 Hz, 1H), 3.85 – 3.70 (m, 2H), 3.70 – 3.61 (m, 1H), 3.47 (dd, *J* = 14.6, 8.5 Hz, 2H), 1.50 (s, 3H), 1.46 (s, 3H), 1.35 (s, 3H), 1.34 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 138.4, 138.1, 128.7, 128.3, 128.2, 128.0, 127.9, 127.6, 127.5, 127.5, 127.4, 109.5, 108.8, 102.4, 96.4, 81.9, 75.8, 75.1, 74.8, 73.6, 73.5, 72.7, 71.7, 71.1, 70.8, 70.5, 69.9, 69.5, 68.1, 26.1, 26.0, 25.1, 24.4.

IR (CH_2Cl_2, cm^{-1}) 2903, 1726, 1496, 1453, 1371, 1254, 1209, 1098, 1064, 1026, 1003, 899, 735, 696.;

ESI HRMS calcd for $C_{46}H_{54}NaO_{11}$ $[M + Na]^+$ 805.3558, found 805.3564.

Compound 28



28

Following the General Glycosylation Procedure: After purification, **28** was obtained as a colourless oil (148 mg, 92%, α/β = 1.3:1).

Gram scale experiment: To an oven-dried 25ml flask equipped with a stirbar were added 2 mmol glycosyl acceptor, 2.4 mmol glycosyl dornor, 0.04 mmol PhSSPh, 0.02 mmol Eosin Y free acid and 10 mL dry dichloromethane. The flask was sealed with a rubber stopper and stirred at room temperature under irradiation with blue LEDs. Upon completion of the reaction (16 hours), the solution was concentrated in vacuo, and the residue was purified by flash column chromatograph to afford glycosides with 1.28 g yield (82%, α/β = 1.2:1).

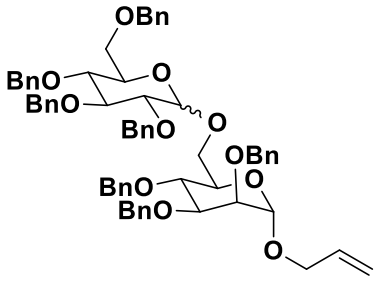
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 5.9 Hz, 2H), 7.22 – 7.18 (m, 36H), 7.06 (d, *J* = 4.3 Hz, 4H), 5.50 (d, *J* = 4.9 Hz, 1H), 5.46 (d, *J* = 5.0 Hz, 1H), 4.96 (d, *J* = 11.1 Hz, 1H), 4.89 (dd, *J* = 13.1, 7.4 Hz, 3H), 4.73 (dt, *J* = 18.5, 9.2 Hz, 8H), 4.65 (d, *J* = 7.7 Hz, 3H), 4.54 (d, *J* = 12.1 Hz, 4H), 4.47 – 4.36 (m, 5H), 4.25 (t, *J* = 5.9 Hz, 3H), 4.17 (d, *J* = 7.8 Hz, 1H), 4.08 (dd, *J* = 10.6, 3.3 Hz, 1H), 4.02 (d, *J* = 7.2 Hz, 1H), 3.99 – 3.88 (m, 2H), 3.78 – 3.47 (m, 13H), 3.39 (t, *J* = 8.1 Hz, 2H), 1.46 (s, 3H), 1.43 (s, 3H), 1.38 (s, 6H), 1.24 (d, *J* = 2.0 Hz, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 138.6, 138.5, 138.3, 138.2, 138.1, 138.0, 137.9, 128.7, 128.4, 128.4, 128.3, 128.0, 128.0, 128.0, 128.0, 127.9, 127.9, 127.8, 127.8, 127.7, 127.7, 127.6, 109.4, 109.3, 108.7, 104.3, 96.9, 96.3, 96.3, 84.5, 81.9, 81.6, 79.8, 77.6, 77.6, 77.5, 77.2, 76.8, 75.7, 75.0, 74.6, 74.5, 73.5, 73.5, 72.5, 71.4, 70.9, 70.7, 70.6, 70.6, 70.5, 70.2, 69.7, 68.6, 68.3, 67.4, 66.2, 65.8, 26.2, 26.1, 26.1, 26.0, 25.0, 24.9, 24.6, 24.4.

IR (CH_2Cl_2, cm^{-1}) 2905, 1727, 1597, 1496, 1453, 1371, 1255, 1209, 1164, 1065, 1027, 1000, 918, 898, 823, 734, 696.;

ESI HRMS calcd for $C_{46}H_{54}NaO_{11}$ $[M + Na]^+$ 805.3558, found 805.3563.

Compound 29



29

Following the General Glycosylation Procedure: After purification, **29** was obtained as a colourless oil (176 mg, 87%, α/β = 1.5:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.22 (m, 33H), 7.17 – 7.14 (m, 2H), 5.84 – 5.81 (m, 1H), 5.23 (dq, *J* = 17.2, 1.6 Hz, 2H), 5.14 (dt, *J* = 11.2, 2.4 Hz, 2H), 4.97 – 4.92 (m, 2H), 4.87 (dd, *J* = 9.3, 6.3 Hz, 2H), 4.78 (d, *J* = 10.9 Hz, 1H), 4.72 (d, *J* = 12.0 Hz, 2H), 4.69 (d, *J* = 4.2 Hz, 2H), 4.64 (dt, *J* = 11.8, 4.0 Hz, 4H), 4.49 (d, *J* = 3.9 Hz, 1H), 4.47 (d, *J* = 5.1 Hz, 1H), 4.20 – 4.15 (m, 1H), 4.06 – 4.01 (m, 2H), 3.97 (ddd, *J* = 5.9, 5.2, 3.1 Hz, 1H), 3.94 – 3.83 (m, 5H), 3.77 – 3.64 (m, 3H), 3.58 (dd, *J* = 9.6, 3.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 138.7, 138.6, 138.6, 138.5, 138.2, 138.1, 133.8, 128.3, 128.3, 128.3, 128.3, 128.3, 128.3, 128.2, 128.2, 128.1, 128.0, 127.9, 127.9, 127.9, 127.7, 127.7, 127.6, 127.6, 127.6, 127.5, 127.5, 127.4, 117.4, 96.9, 96.5, 81.7, 80.4, 80.1, 77.7, 75.5, 75.2, 75.1, 74.9, 73.4, 72.8, 72.5, 72.2, 72.0, 70.1, 68.6, 67.7, 66.0.;

IR (CH_2Cl_2, cm^{-1}) 3029, 2918, 1496, 1453, 1360, 1208, 1090, 1072, 1027, 914, 735, 696.;

ESI HRMS calcd for $C_{64}H_{68}NaO_{11}$ $[M + Na]^+$ 1035.4654, found 1035.4653.

Data for β:

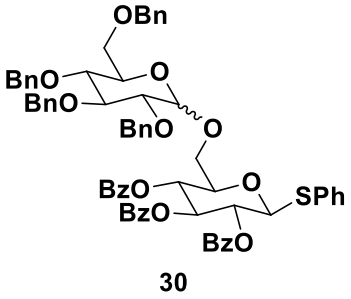
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.18 (m, 35H), 5.79 (dddd, *J* = 17.2, 10.5, 6.1, 4.9 Hz, 1H), 5.18 (ddd, *J* = 17.2, 3.3, 1.7 Hz, 1H), 5.13 (ddd, *J* = 10.4, 2.9, 1.3 Hz, 1H), 5.08 (d, *J* = 11.0 Hz, 1H), 4.97 (dd, *J* = 7.4, 6.5 Hz, 2H), 4.87 – 4.78 (m, 3H), 4.72 (d, *J* = 11.6 Hz, 3H), 4.67 – 4.62 (m, 3H), 4.59 – 4.55 (m, 2H), 4.53 (d, *J* = 11.1 Hz, 1H), 4.43 (d, *J* = 7.8 Hz, 1H), 4.29 (dd, *J* = 10.7, 1.9 Hz, 1H), 4.16 – 4.10 (m, 1H), 4.01 (d, *J* = 9.4 Hz, 1H), 3.98 (dd, *J* = 9.3, 3.0 Hz, 1H), 3.93 – 3.83 (m, 3H), 3.81 – 3.70 (m, 3H), 3.69 – 3.59 (m, 2H), 3.53 (dd, *J* = 8.9, 7.9 Hz, 1H), 3.49 (ddd, *J* = 9.5, 4.7, 2.0 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.7, 138.6, 138.6, 138.3, 138.3, 138.2, 133.7, 128.4, 128.3, 128.3, 128.3, 128.2, 127.9, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 117.2, 104.1, 97.0, 84.7, 82.1, 80.3, 78.0, 75.7, 75.0, 75.0, 74.8, 74.7, 73.5, 72.8, 72.1, 71.6, 69.1, 69.0, 67.7.;

IR (CH_2Cl_2, cm^{-1}) 3029, 2918, 1496, 1453, 1360, 1208, 1069, 1028, 735, 697.;

ESI HRMS calcd for $C_{64}H_{68}NaO_{11}$ $[M + Na]^+$ 1035.4654, found 1035.4666.

Compound 30



30

Following the General Glycosylation Procedure: After purification, **30** was obtained as a colourless oil (179 mg, 81%, α/β = 1.3:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.95 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.81 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.60 – 7.11 (m, 34H), 5.88 (t, *J* = 9.5 Hz, 1H), 5.46 (dt, *J* = 12.7, 9.9 Hz, 2H), 5.01 (d, *J* = 10.1 Hz, 1H), 4.95 (d, *J* = 10.9 Hz, 1H), 4.86 – 4.77 (m, 3H), 4.69 (d, *J* = 3.6 Hz, 1H), 4.63 (d, *J* = 2.2 Hz, 1H), 4.60 (d, *J* = 2.2 Hz, 1H), 4.51 (d, *J* = 11.0 Hz, 1H), 4.44 (d, *J* = 12.1 Hz, 1H), 4.15 (ddd, *J* = 9.7, 6.1, 1.9 Hz, 1H), 3.94 (ddd, *J* = 18.4, 14.8, 8.4 Hz, 3H), 3.73 – 3.61 (m, 3H), 3.59 – 3.53 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 165.7, 165.3, 165.0, 138.6, 138.5, 138.2, 138.0, 133.5, 133.3, 133.2, 132.8, 132.4, 129.9, 129.7, 129.3, 129.0, 128.8, 128.8, 128.5, 128.4, 128.4, 127.9, 127.9, 127.8, 127.7, 127.6, 103.9, 85.8, 84.6, 82.3, 78.3, 77.7, 75.7, 74.9, 74.9, 74.8, 74.3, 73.5, 73.3, 70.6, 70.1, 69.6, 68.3, 67.3.;

IR (CH_2Cl_2, cm^{-1}) 2920, 1733, 1601, 1452, 1278, 1259, 1089, 1069, 1027, 738, 706.;

ESI HRMS calcd for $C_{67}H_{62}NaO_{13}S$ $[M + Na]^+$ 1129.3803, found 1129.3814.

Data for β:

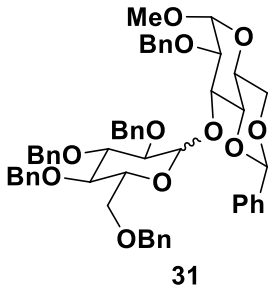
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.93 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.80 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.64 – 7.21 (m, 32H), 7.16 (dd, *J* = 7.2, 2.2 Hz, 2H), 5.90 (t, *J* = 9.5 Hz, 1H), 5.49 (t, *J* = 9.7 Hz, 1H), 5.42 (t, *J* = 9.8 Hz, 1H), 5.07 (d, *J* = 10.0 Hz, 1H), 4.96 (dd, *J* = 12.4, 11.1 Hz, 2H), 4.83 (d, *J* = 10.8 Hz, 1H), 4.79 (d, *J* = 10.9 Hz, 1H), 4.66 (d, *J* = 11.0 Hz, 1H), 4.55 (d, *J* = 5.5 Hz, 1H), 4.53 (d, *J* = 4.1 Hz, 1H), 4.50 (d, *J* = 7.7 Hz, 1H), 4.45 (d, *J* = 12.2 Hz, 1H), 4.18 (ddd, *J* = 9.9, 7.6, 2.1 Hz, 1H), 4.08 (dd, *J* = 11.4, 2.1 Hz, 1H), 3.93 (dd, *J* = 11.3, 7.7 Hz, 1H), 3.70 – 3.58 (m, 4H), 3.57 – 3.53 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 165.7, 165.4, 165.0, 138.6, 138.1, 133.4, 133.3, 133.2, 132.4, 132.1, 129.9, 129.7, 129.2, 129.1, 128.9, 128.9, 128.8, 128.5, 128.4, 128.3, 128.3, 128.2, 128.2, 128.1, 128.0, 127.9, 127.8, 127.8, 127.7, 127.7, 127.6, 127.5, 127.4, 97.3, 85.8, 84.6, 82.3, 78.3, 77.7, 75.7, 74.9, 74.8, 74.3, 73.5, 70.6, 69.9, 69.0, 68.7.;

IR (CH_2Cl_2, cm^{-1}) 3031, 2869, 1729, 1601, 1496, 1452, 1361, 1315, 1278, 1260, 1087, 1068, 1027, 825, 708.;

ESI HRMS calcd for $C_{67}H_{62}NaO_{13}S$ $[M + Na]^+$ 1129.3803, found 1129.3809.

Compound 31



31

Following the General Glycosylation Procedure: After purification, **31** was obtained as a colourless oil (130 mg, 73%, α/β = 1.5:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.18 (m, 24H), 7.17 – 7.16 (m, 4H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.61 (d, *J* = 3.6 Hz, 1H), 5.49 (s, 1H), 5.02 (d, *J* = 10.8 Hz, 1H), 4.83 (d, *J* = 4.6 Hz, 1H), 4.81 (d, *J* = 4.9 Hz, 1H), 4.73 (d, *J* = 3.7 Hz, 1H), 4.68 (d, *J* = 11.2 Hz, 1H), 4.62 (d, *J* = 6.2 Hz, 1H), 4.60 (d, *J* = 1.8 Hz, 1H), 4.58 (d, *J* = 8.9 Hz, 1H), 4.37 (ddd, *J* = 27.4, 18.5, 11.7 Hz, 3H), 4.27 (dd, *J* = 10.2, 4.8 Hz, 1H), 4.22 (d, *J* = 10.1 Hz, 1H), 3.99 (t, *J* = 9.3 Hz, 1H), 3.90 (td, *J* = 9.9, 4.7 Hz, 1H), 3.81 (t, *J* = 9.4 Hz, 1H), 3.75 (d, *J* = 10.3 Hz, 1H), 3.72 – 3.65 (m, 2H), 3.56 – 3.48 (m, 2H), 3.26 (d, *J* = 0.9 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.0, 138.7, 138.5, 138.2, 138.1, 137.4, 134.5, 129.7, 129.0, 128.8, 128.4, 128.4, 128.3, 128.2, 128.2, 128.1, 128.0, 127.9, 127.9, 127.8, 127.8, 127.7, 127.6, 127.5, 126.2, 102.5, 101.4, 98.5, 84.9, 82.9, 80.5, 80.4, 78.0, 75.8, 75.6, 75.0, 74.8, 74.7, 73.8, 73.5, 69.1, 68.7, 62.2, 55.3.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2919, 1730, 1496, 1453, 1362, 1210, 1156, 1087, 1074, 1056, 1028, 998, 735, 697.;

ESI HRMS calcd for $C_{55}H_{58}NaO_{11}$ $[M + Na]^+$ 917.3871, found 917.3874.

Data for β:

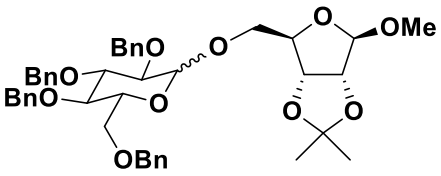
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.37 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.36 – 7.21 (m, 24H), 7.17 (dd, *J* = 7.3, 2.2 Hz, 2H), 5.49 (s, 1H), 5.09 (d, *J* = 11.2 Hz, 1H), 4.94 (d, *J* = 10.9 Hz, 1H), 4.91 (d, *J* = 7.8 Hz, 1H), 4.83 – 4.72 (m, 4H), 4.55 (d, *J* = 10.7 Hz, 1H), 4.51 – 4.47 (m, 3H), 4.38 (t, *J* = 9.1 Hz, 1H), 4.23 (dd, *J* = 10.1, 4.7 Hz, 1H), 3.84 (td, *J* = 10.0, 4.7 Hz, 1H), 3.73 – 3.60 (m, 5H), 3.59 (d, *J* = 3.1 Hz, 1H), 3.52 (t, *J* = 8.3 Hz, 1H), 3.50 (s, 3H), 3.48 (dd, *J* = 6.1, 3.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.0, 138.9, 138.1, 137.9, 137.4, 137.1, 129.4, 128.8, 128.5, 128.5, 128.4, 128.3, 128.2, 128.1, 128.1, 128.1, 127.9, 127.9, 127.9, 127.7, 127.6, 127.5, 127.4, 127.4, 127.3, 126.4, 102.1, 98.5, 96.1, 84.9, 82.9, 80.5, 81.7, 78.0, 77.6, 77.4, 77.1, 76.9, 76.6, 75.5, 74.8, 73.4, 73.3, 72.7, 71.2, 69.8, 69.2, 68.2, 61.8, 55.3.;

IR (CH_2Cl_2, cm^{-1}) 3374, 2866, 1729, 1693, 1617, 1496, 1453, 1359, 1265, 1088, 1027, 833, 734, 697.;

ESI HRMS calcd for $C_{55}H_{58}NaO_{11}$ $[M + Na]^+$ 917.3871, found 917.3881.

Compound 32



32

Following the General Glycosylation Procedure: After purification, **32** was obtained as a colourless oil (130 mg, 90%, α/β = 2:1).

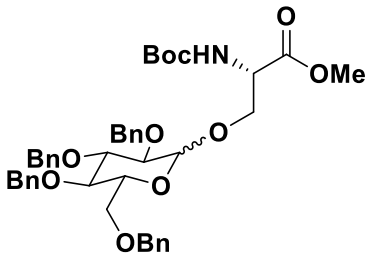
^1H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.18 (m, 18H), 7.17 – 7.16 (m, 2H), 5.04 – 4.92 (m, 3H), 4.91 – 4.79 (m, 3H), 4.76 (dd, *J* = 7.7, 4.6 Hz, 1H), 4.69 – 4.55 (m, 4H), 4.51 (t, *J* = 10.3 Hz, 1H), 4.44 (ddd, *J* = 17.8, 8.6, 3.7 Hz, 2H), 4.05 – 4.01 (m, 1H), 3.96 (ddd, *J* = 9.6, 8.3, 1.5 Hz, 1H), 3.85 (d, *J* = 10.1 Hz, 1H), 3.79 – 3.59 (m, 5H), 3.53 – 3.46 (m, 2H), 3.34 – 3.31 (m, 3H), 1.53 (s, 2H), 1.51 (s, 1H), 1.35 – 1.34 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 138.6, 138.4, 138.1, 138.0, 128.3, 128.3, 127.9, 127.8, 127.7, 127.6, 127.6, 112.3, 109.3, 109.2, 103.7, 98.1, 85.2, 85.1, 84.8, 84.6, 82.2, 82.1, 82.0, 81.8, 80.2, 77.8, 75.6, 75.0, 74.9, 73.5, 73.1, 70.5, 69.7, 68.9, 68.4, 54.8, 26.5, 25.1, 25.0.;

IR (CH_2Cl_2, cm^{-1}) 2918, 1496, 1453, 1360, 1309, 1273, 1239, 1208, 1157, 1087, 1063, 1027, 1013, 961, 869, 734, 695.;

ESI HRMS calcd for $C_{43}H_{50}NaO_{20}$ $[M + Na]^+$ 749.3296, found 749.3291.

Compound 33



33

Following the General Glycosylation Procedure: After purification, **33** was obtained as a colourless oil (93 mg, 63%, α/β = 1.1:1).

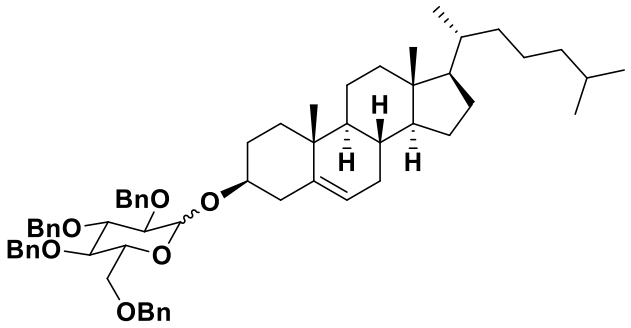
^1H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.14 (m, 20H), 5.68 (d, *J* = 12.6 Hz, 1H), 5.48 (d, *J* = 7.9 Hz, 1H), 4.98 – 4.01 (m, 8H), 4.37 (d, *J* = 12.5 Hz, 2H), 4.07 (d, *J* = 7.8 Hz, 1H), 3.95 – 3.66 (m, 7H), 3.56 (dd, *J* = 9.5, 3.1 Hz, 1H), 3.45 (m, 2H), 1.47 (s, 21H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 170.8, 170.7, 155.5, 155.4, 138.8, 138.5, 138.4, 138.2, 138.2, 138.1, 137.8, 128.5, 128.4, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.9, 127.8, 127.7, 127.7, 127.7, 127.6, 127.6, 127.5, 103.8, 98.4, 84.5, 81.9, 81.7, 80.0, 79.9, 75.6, 75.2, 75.0, 74.9, 73.5, 73.5, 72.9, 70.7, 69.9, 69.8, 68.6, 68.3, 54.1, 54.0, 52.5, 52.4, 28.3.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2918, 1748, 1714, 1496, 1453, 1364, 1248, 1208, 1159, 1065, 1027, 734, 696.;

ESI HRMS calcd for $C_{43}H_{51}NNaO_{10}$ $[M + Na]^+$ 764.3405, found 764.3406.

Compound 34



34

Following the General Glycosylation Procedure: After purification, **34** was obtained as a white solid (158 mg, 87%, α/β = 1.2:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.26 (m, 18H), 7.19 – 7.14 (m, 2H), 5.36 (s, 1H), 5.03 (d, *J* = 10.8 Hz, 1H), 4.97 (d, *J* = 3.7 Hz, 1H), 4.85 (dd, *J* = 10.7, 7.7 Hz, 2H), 4.79 (d, *J* = 12.0 Hz, 1H), 4.68 (d, *J* = 12.0 Hz, 1H), 4.64 (d, *J* = 12.1 Hz, 1H), 4.49 (dd, *J* = 11.4, 7.5 Hz, 2H), 4.03 (t, *J* = 9.3 Hz, 1H), 3.91 (ddd, *J* = 10.1, 3.5, 2.1 Hz, 1H), 3.77 (dd, *J* = 10.6, 3.7 Hz, 1H), 3.66 (ddd, *J* = 9.1, 5.2, 3.2 Hz, 2H), 3.58 (dd, *J* = 9.7, 3.7 Hz, 1H), 3.50 (tt, *J* = 11.3, 4.3 Hz, 1H), 2.49 – 2.41 (m, 1H), 2.33 – 2.25 (m, 1H), 2.04 (dd, *J* = 9.2, 3.3 Hz, 1H), 2.01 – 1.95 (m, 1H), 1.92 – 1.82 (m, 3H), 1.64 – 1.46 (m, 7H), 1.36 (ddd, *J* = 38.5, 23.7, 7.4 Hz, 5H), 1.22 – 0.97 (m, 12H), 0.95 (d, *J* = 6.5 Hz, 3H), 0.90 (dd, *J* = 6.6, 2.2 Hz, 6H), 0.71 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 140.6, 138.7, 138.6, 138.3, 138.2, 128.4, 128.4, 128.4, 128.3, 128.1, 128.0, 127.9, 127.8, 127.7, 127.7, 127.6, 127.5, 121.9, 102.3, 84.8, 82.4, 79.7, 78.0, 76.6, 75.6, 75.0, 74.9, 74.8, 73.4, 70.1, 69.2, 56.8, 56.2, 50.1, 42.3, 39.8, 39.5, 39.1, 37.3, 36.8, 36.2, 35.8, 31.9, 31.9, 30.0, 28.2, 28.0, 24.3, 23.8, 22.8, 22.5, 21.1, 19.4, 18.7, 11.9.;

IR (CH_2Cl_2, cm^{-1}) 2931, 2865, 1496, 1453, 1361, 1207, 1156, 1069, 1027, 732, 695.;

ESI HRMS calcd for $C_{61}H_{80}NaO_6$ $[M + Na]^+$ 931.5847, found 931.5840.

Data for β:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.27 (m, 18H), 7.17 – 7.15 (m, 2H), 5.32 (s, 1H), 4.98 (d, *J* = 10.9 Hz, 1H), 4.93 (d, *J* = 10.9 Hz, 1H), 4.83 – 4.80 (m, 2H), 4.75 (d, *J* = 13.6 Hz, 1H), 4.71 – 4.50 (m, 4H), 3.74 (d, *J* = 10.5 Hz, 1H), 3.67 – 3.43 (m, 6H), 2.38 (dd, *J* = 14.6, 3.9 Hz,

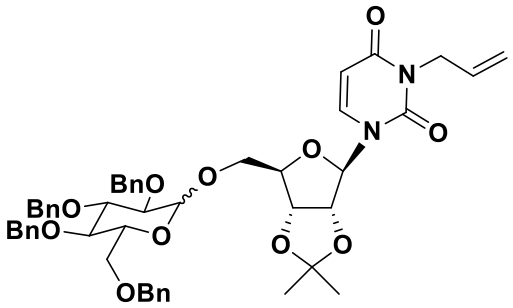
2H), 2.04 – 1.82 (m, 5H), 1.57 – 0.99 (m, 24H), 0.93 (d, *J* = 6.3 Hz, 3H), 0.89 (d, *J* = 6.6 Hz, 6H),0.88 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 139.0, 138.3, 138.0, 128.4, 128.4, 128.3, 128.2, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 121.7, 94.7, 82.1, 80.0, 77.9, 75.7, 75.6, 73.4, 73.0, 70.1, 68.7, 56.8, 56.2, 50.2, 42.4, 39.9, 39.8, 39.5, 37.3, 36.8, 36.2, 35.8, 32.0, 31.9, 28.2, 28.0, 27.5, 24.3, 23.8, 22.8, 22.6, 21.1, 19.4, 18.7, 11.9.;

IR (CH_2Cl_2, cm^{-1}) 2933, 2866, 1496, 1453, 1362, 1274, 1208, 1069, 1028, 733, 696.;

ESI HRMS calcd for $C_{61}H_{80}NaO_6$ $[M + Na]^+$ 931.5847, found 931.5840.

Compound 35



35

Following the General Glycosylation Procedure: After purification, **35** was obtained as a colourless oil (115 mg, 68%, α/β = 1.3:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.1 Hz, 1H), 7.41 – 7.26 (m, 20H), 7.17 – 7.10 (m, 2H), 6.10 (d, *J* = 2.8 Hz, 1H), 5.90 (ddt, *J* = 17.1, 10.2, 5.8 Hz, 1H), 5.60 (d, *J* = 8.0 Hz, 1H), 5.28 (dq, *J* = 17.1, 1.4 Hz, 1H), 5.20 (dq, *J* = 10.2, 1.1 Hz, 1H), 4.97 (d, *J* = 10.9 Hz, 1H), 4.85 (d, *J* = 2.3 Hz, 1H), 4.82 (t, *J* = 6.9 Hz, 2H), 4.77 (dd, *J* = 6.2, 3.9 Hz, 1H), 4.70 – 4.65 (m, 2H), 4.62 (dd, *J* = 12.2, 6.3 Hz, 2H), 4.57 – 4.51 (m, 1H), 4.50 (t, *J* = 2.4 Hz, 1H), 4.48 (d, *J* = 3.7 Hz, 1H), 4.38 (dd, *J* = 6.6, 3.0 Hz, 1H), 3.90 (dd, *J* = 10.9, 3.2 Hz, 1H), 3.86 (dd, *J* = 9.5, 8.5 Hz, 1H), 3.75 – 3.58 (m, 5H), 1.62 (s, 3H), 1.38 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 162.2, 150.7, 139.0, 138.5, 137.9, 137.9, 137.8, 131.5, 128.6, 128.5, 128.4, 128.4, 128.2, 128.0, 127.9, 127.9, 127.8, 127.8, 127.8, 118.1, 114.6, 102.4, 97.7, 91.6, 85.0, 84.4, 82.0, 80.0, 79.9, 77.5, 75.7, 75.3, 73.8, 73.6, 70.9, 68.3, 67.6, 43.0, 27.5, 25.6.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2918, 1710, 1666, 1496, 1451, 1362, 1356, 1270, 1209, 1155, 1068, 1027, 858, 734, 696.;

ESI HRMS calcd for $C_{49}H_{54}N_2NaO_{11}$ $[M + Na]^+$ 869.3620, found 869.3629.

Data for β:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.1 Hz, 1H), 7.39 – 7.27 (m, 20H), 7.20 – 7.15 (m, 2H), 5.91 (ddt, *J* = 17.1, 10.2, 5.8 Hz, 1H), 5.84 (d, *J* = 2.5 Hz, 1H), 5.76 (d, *J* = 8.1 Hz, 1H), 5.28 (dq, *J* = 17.1, 1.4 Hz, 1H), 5.19 (dq, *J* = 10.2, 1.2 Hz, 1H), 4.91 (d, *J* = 11.0 Hz, 1H), 4.85 (d, *J* = 1.8 Hz, 1H), 4.83 (d, *J* = 2.2 Hz, 1H), 4.79 – 4.74 (m, 3H), 4.68 (dd, *J* = 6.3, 2.5 Hz,

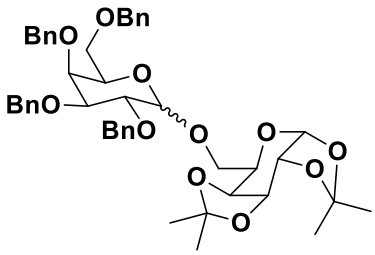
1H), 4.63 (s, 1H), 4.59 – 4.53 (m, 3H), 4.53 – 4.49 (m, 1H), 4.43 (d, *J* = 7.8 Hz, 1H), 4.27 (dd, *J* = 10.8, 2.8 Hz, 1H), 3.76 – 3.60 (m, 5H), 3.47 (ddd, *J* = 9.4, 4.5, 1.9 Hz, 1H), 3.39 – 3.33 (m, 1H), 1.60 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 162.3, 150.6, 139.1, 138.4, 138.1, 138.0, 137.9, 131.5, 128.4, 128.4, 128.0, 127.8, 127.7, 127.7, 127.6, 127.6, 127.6, 118.1, 114.1, 103.2, 101.4, 94.5, 85.8, 85.7, 84.7, 82.1, 80.9, 77.7, 75.6, 75.1, 75.0, 74.9, 73.5, 69.5, 68.7, 42.9, 27.2, 25.2.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2917, 1710, 1665, 1496, 1452, 1414, 1382, 1360, 1270, 1209, 1155, 1064, 1027, 1000, 806, 733, 696.;

ESI HRMS calcd for $C_{49}H_{54}N_2NaO_{11}$ $[M + Na]^+$ 869.3620, found 869.3628.

Compound 36



36

Following the General Glycosylation Procedure: After purification, **36** was obtained as a colourless oil (134 mg, 86%, α/β = 1.5:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.25 (m, 20H), 5.54 (d, *J* = 5.1 Hz, 1H), 5.04 (d, *J* = 3.7 Hz, 1H), 4.96 (d, *J* = 11.5 Hz, 1H), 4.86 (d, *J* = 11.7 Hz, 1H), 4.81 – 4.74 (m, 3H), 4.64 – 4.57 (m, 2H), 4.51 (d, *J* = 11.8 Hz, 1H), 4.44 (d, *J* = 11.8 Hz, 1H), 4.37 – 4.31 (m, 2H), 4.11 – 4.02 (m, 4H), 3.99 (dd, *J* = 10.1, 2.8 Hz, 1H), 3.79 (ddd, *J* = 25.8, 10.5, 6.8 Hz, 2H), 3.60 (dd, *J* = 9.2, 7.6 Hz, 1H), 3.55 (dd, *J* = 9.2, 5.7 Hz, 1H), 1.55 (s, 3H), 1.46 (s, 3H), 1.35 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 138.8, 138.1, 128.4, 128.3, 128.2, 128.2, 127.8, 127.7, 127.6, 127.4, 127.4, 127.3, 109.2, 108.5, 97.6, 96.3, 79.0, 76.4, 74.7, 73.4, 73.0, 72.6, 70.9, 70.7, 69.2, 68.7, 66.3, 65.8, 26.1, 26.0, 24.9, 24.6.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2985, 2914, 1724, 1495, 1453, 1331, 1254, 1210, 1165, 1095, 1067, 1028, 1000, 889, 736, 696.;

ESI HRMS calcd for $C_{46}H_{54}NaO_{11}$ $[M + Na]^+$ 805.3558, found 805.3562.

Data for β:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.45 (m, 2H), 7.41 – 7.26 (m, 18H), 5.59 (d, *J* = 5.0 Hz, 1H), 5.08 (d, *J* = 11.0 Hz, 1H), 4.96 (d, *J* = 11.6 Hz, 1H), 4.82 (d, *J* = 11.9 Hz, 1H), 4.75 (t, *J* = 12.0 Hz, 2H), 4.64 (d, *J* = 11.6 Hz, 1H), 4.60 (dd, *J* = 7.9, 2.4 Hz, 1H), 4.49 – 4.41 (m, 3H), 4.33 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.24 (dd, *J* = 7.9, 1.9 Hz, 1H), 4.15 (dd, *J* = 10.6, 3.6 Hz, 1H), 4.10 (ddd, *J* = 7.3, 3.4, 1.8 Hz, 1H), 3.91 (d, *J* = 2.5 Hz, 1H), 3.85 (dd, *J* = 9.8, 7.7 Hz, 1H),

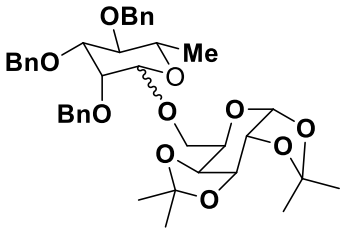
3.72 (dd, *J* = 10.6, 7.5 Hz, 1H), 3.57 (dddd, *J* = 12.7, 9.8, 7.3, 5.3 Hz, 4H), 1.52 (s, 3H), 1.46 (s, 3H), 1.34 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.1, 138.7, 138.7, 137.9, 128.6, 128.4, 128.4, 128.3 128.1, 128.1, 127.9, 127.5, 127.5, 109.3, 108.6, 104.7, 96.4, 82.0, 79.1, 74.7, 74.5, 73.7, 73.5, 73.3, 73.1, 71.5, 70.8, 70.5, 69.6, 68.7, 67.4, 26.0, 26.0, 25.0, 24.4.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2932, 2865, 1496, 1453, 1362, 1255, 1208, 1070, 1028, 733, 696.;

ESI HRMS calcd for $C_{46}H_{54}NaO_{11}$ $[M + Na]^+$ 805.3558, found 805.3562.

Compound 37



37

Following the General Glycosylation Procedure: After purification, **37** was obtained as a colourless oil (120 mg, 89%, α/β = 1.6:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.42 (m, 2H), 7.40 – 7.30 (m, 13H), 5.57 (d, *J* = 5.0 Hz, 1H), 4.99 (d, *J* = 11.0 Hz, 1H), 4.94 (d, *J* = 1.5 Hz, 1H), 4.78 (s, 2H), 4.69 (d, *J* = 11.0 Hz, 1H), 4.64 (d, *J* = 1.9 Hz, 2H), 4.63 (dd, *J* = 7.9, 2.3 Hz, 1H), 4.35 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.20 (dd, *J* = 7.9, 1.9 Hz, 1H), 3.95 (td, *J* = 5.2, 2.6 Hz, 1H), 3.90 – 3.84 (m, 3H), 3.80 (ddd, *J* = 12.4, 7.9, 4.7 Hz, 1H), 3.67 (t, *J* = 9.2 Hz, 1H), 3.61 (dd, *J* = 10.5, 7.0 Hz, 1H), 1.56 (s, 3H), 1.49 (s, 3H), 1.39 – 1.37 (m, 6H), 1.37 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.8, 138.6, 138.5, 128.2, 127.8, 127.8, 127.6, 127.5, 127.5, 127.4, 109.2, 108.5, 98.0, 96.2, 80.4, 79.9, 75.2, 74.7, 72.6, 71.9, 71.1, 70.6, 70.5, 68.0, 67.2, 65.9, 26.1, 25.9, 24.9, 24.4, 17.9.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2910, 1496, 1453, 1381, 1371, 1254, 1209, 1167, 1113, 1064, 1027, 1000, 916, 732, 696.;

ESI HRMS calcd for $C_{39}H_{48}NaO_{10}$ $[M + Na]^+$ 699.3140, found 699.3148.

Data for β:

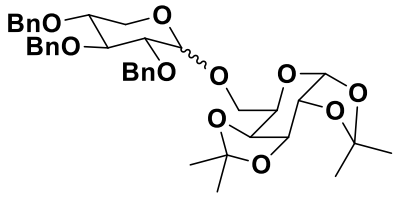
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.46 (m, 2H), 7.37 – 7.26 (m, 13H), 5.55 (d, *J* = 5.0 Hz, 1H), 4.98 (d, *J* = 11.0 Hz, 2H), 4.91 (d, *J* = 12.5 Hz, 1H), 4.67 (d, *J* = 10.8 Hz, 1H), 4.62 (dd, *J* = 8.0, 2.3 Hz, 1H), 4.54 (d, *J* = 11.9 Hz, 1H), 4.47 (d, *J* = 11.8 Hz, 1H), 4.43 (s, 1H), 4.34 (dd, *J* = 5.0, 2.3 Hz, 1H), 4.26 (dd, *J* = 8.0, 1.7 Hz, 1H), 4.12 – 4.07 (m, 1H), 4.00 – 3.92 (m, 2H), 3.74 (dd, *J* = 9.4, 8.5 Hz, 1H), 3.63 (t, *J* = 9.3 Hz, 1H), 3.48 (dd, *J* = 9.4, 3.0 Hz, 1H), 3.33 (dq, *J* = 9.2, 6.1 Hz, 1H), 1.57 (s, 3H), 1.48 (s, 3H), 1.39 (d, *J* = 6.2 Hz, 3H), 1.36 (s, 3H), 1.35 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.8, 138.5, 138.2, 128.5, 128.4, 128.3, 128.1, 128.1, 128.1, 127.7, 127.6, 127.5, 127.4, 109.0, 108.5, 101.7, 96.3, 82.1, 80.1, 75.4, 73.9, 73.7, 72.0, 71.5, 70.8, 70.6, 70.5, 67.7, 65.8, 26.2, 26.0, 24.9, 24.4, 17.9.;

IR (CH_2Cl_2, cm^{-1}) 3293, 3030, 2933, 1730, 1601, 1496, 1453, 1371, 1254, 1209, 1168, 1103, 1067, 998, 823, 735, 696.;

ESI HRMS calcd for $C_{39}H_{48}NaO_{10}$ $[M + Na]^+$ 699.3140, found 699.3151.

Compound 38



38

Following the General Glycosylation Procedure: After purification, **38** was obtained as a colourless oil (107 mg, 81%, α/β = 1.1:1).

Data for α:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 15H), 5.57 (d, *J* = 5.0 Hz, 1H), 4.96 (d, *J* = 10.9 Hz, 1H), 4.92 (d, *J* = 3.6 Hz, 1H), 4.89 (d, *J* = 10.9 Hz, 1H), 4.79 – 4.72 (m, 3H), 4.66 (d, *J* = 11.6 Hz, 1H), 4.65 – 4.62 (m, 1H), 4.40 (dd, *J* = 8.0, 1.9 Hz, 1H), 4.35 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.10 – 4.04 (m, 1H), 3.96 – 3.89 (m, 1H), 3.81 (dd, *J* = 10.3, 6.0 Hz, 1H), 3.75 (dd, *J* = 10.3, 7.7 Hz, 1H), 3.69 – 3.55 (m, 3H), 3.50 (dd, *J* = 9.5, 3.6 Hz, 1H), 1.58 (s, 3H), 1.49 (s, 3H), 1.37 (s, 3H), 1.35 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.0, 138.4, 138.3, 128.4, 128.3, 128.3, 128.0, 127.8, 127.7, 127.7, 127.6, 127.5, 109.1, 108.6, 97.2, 96.3, 81.2, 79.6, 78.0, 75.6, 73.4, 72.5, 70.8, 70.7, 70.6, 66.3, 65.8, 60.0, 26.1, 26.0, 24.9, 24.6.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2985, 2933, 1729, 1496, 1454, 1380, 1371, 1254, 1209, 1166, 1066, 998, 736, 697.;

ESI HRMS calcd for $C_{38}H_{46}NaO_{10}$ $[M + Na]^+$ 685.2983, found 685.2991.

Data for β:

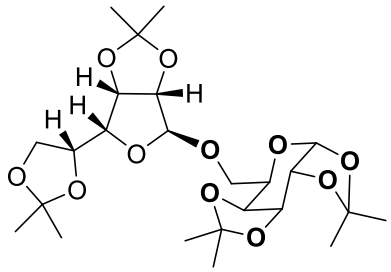
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.37 – 7.29 (m, 13H), 5.58 (d, *J* = 5.0 Hz, 1H), 5.02 (d, *J* = 11.1 Hz, 1H), 4.91 (d, *J* = 11.0 Hz, 1H), 4.85 (d, *J* = 11.0 Hz, 1H), 4.74 (t, *J* = 11.6 Hz, 2H), 4.63 (d, *J* = 11.6 Hz, 1H), 4.61 (dd, *J* = 7.9, 2.4 Hz, 1H), 4.43 (d, *J* = 7.6 Hz, 1H), 4.34 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.25 (dd, *J* = 7.9, 1.6 Hz, 1H), 4.13 – 4.04 (m, 2H), 3.93 (dd, *J* = 11.5, 5.0 Hz, 1H), 3.81 – 3.72 (m, 1H), 3.61 (ddd, *J* = 24.5, 12.9, 7.0 Hz, 2H), 3.45 – 3.37 (m, 1H), 3.22 (dd, *J* = 11.6, 9.7 Hz, 1H), 1.52 (s, 3H), 1.48 (s, 3H), 1.34 (s, 3H), 1.34 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.8, 138.7, 138.2, 128.4, 128.3, 128.2, 127.9, 127.8, 127.8, 127.5, 127.5, 109.4, 108.5, 104.9, 96.4, 83.7, 81.4, 77.8, 75.6, 74.5, 73.4, 71.3, 70.7, 70.5, 69.4, 67.1, 63.9, 26.0, 26.0, 25.0, 24.4.;

IR (CH_2Cl_2, cm^{-1}) 3030, 2982, 2902, 1731, 1496, 1454, 1372, 1254, 1209, 1167, 1067, 1002, 898, 735, 697.;

ESI HRMS calcd for $C_{38}H_{46}NaO_{10}$ $[M + Na]^+$ 685.2983, found 685.2992.

Compound 39



39

Following the General Glycosylation Procedure: After purification, **39** was obtained as a colourless oil (81 mg, 81%, α only).

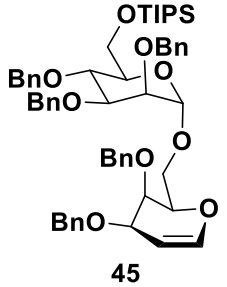
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.53 (d, *J* = 5.1 Hz, 1H), 5.03 (s, 1H), 4.79 (dd, *J* = 5.9, 3.6 Hz, 1H), 4.64 (d, *J* = 5.9 Hz, 1H), 4.61 (dd, *J* = 7.9, 2.4 Hz, 1H), 4.39 (ddd, *J* = 8.0, 6.2, 4.4 Hz, 1H), 4.32 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.22 (dd, *J* = 7.9, 1.9 Hz, 1H), 4.10 (dd, *J* = 8.7, 6.2 Hz, 1H), 4.05 (dd, *J* = 8.7, 4.4 Hz, 1H), 3.97 (ddd, *J* = 8.5, 7.3, 2.7 Hz, 2H), 3.75 (dd, *J* = 10.5, 6.7 Hz, 1H), 3.65 (dd, *J* = 10.5, 6.7 Hz, 1H), 1.54 (s, 3H), 1.47 (s, 3H), 1.45 (s, 3H), 1.45 (s, 3H), 1.38 (s, 3H), 1.35 (s, 3H), 1.33 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 112.5, 109.3, 109.2, 108.5, 106.7, 96.3, 85.0, 80.3, 79.5, 73.0, 70.9, 70.6, 70.5, 66.9, 66.0, 66.0, 26.9, 26.0, 25.9, 25.9, 25.2, 24.9, 24.6, 24.5.;

IR (CH_2Cl_2, cm^{-1}) 3294, 2986, 2936, 1729, 1061, 1455, 1371, 1255, 1209, 1164, 1065, 999, 846, 824.;

ESI HRMS calcd for $C_{24}H_{38}NaO_{11}$ $[M + Na]^+$ 525.2306, found 525.2310.

Compound 45



45

Following the General Glycosylation Procedure: After purification, **45** was obtained as a colourless oil (73 mg, 40%, α/β > 20:1).

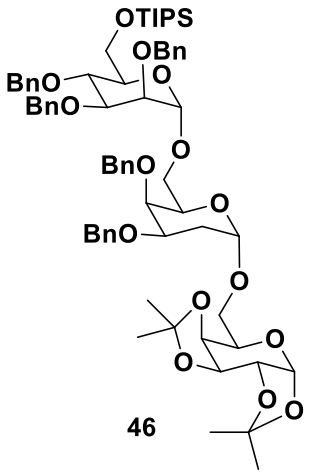
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 25H), 6.34 (d, *J* = 6.2 Hz, 1H), 4.92 (dd, *J* = 15.9, 11.4 Hz, 3H), 4.70 (ddd, *J* = 27.5, 22.8, 13.1 Hz, 9H), 4.22 (s, 1H), 4.06 (d, *J* = 9.2 Hz, 1H), 4.03 – 3.85 (m, 6H), 3.69 (s, 1H), 3.60 (dd, *J* = 10.1, 5.4 Hz, 2H), 1.10 (s, 21H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 138.7, 138.6, 138.5, 138.4, 128.4, 128.3, 128.3, 128.3, 128.2, 128.1, 127.7, 127.6, 127.5, 127.5, 127.5, 127.4, 99.8, 97.5, 80.1, 75.3, 75.2, 74.9, 74.7, 73.5, 73.3, 72.5, 72.2, 70.9, 70.8, 65.0, 62.9, 18.0, 18.0, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2940, 2864, 1496, 1453, 1362, 1098, 1056, 1027, 882, 734, 698.;

ESI HRMS calcd for $C_{56}H_{70}NaO_9Si$ $[M + Na]^+$ 937.4681, found 937.4685.

Compound 46



46

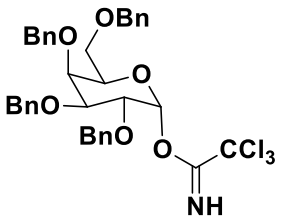
To an oven-dried 1.5 dram vial equipped with a stirbar were added 0.05 mmol glycosyl acceptor **45**, 0.06 mmol glycosyl dornor **43**, 0.001 mmol PhSSPh, 0.0005 mmol Eosin Y free acid and 0.25 mL dry dichloromethane. The vial was sealed with a Teflon cap and stirred at room temperature under irradiation with blue LEDs. Upon completion of the reaction 6 hours, the solution was concentrated in vacuo, and the residue was purified by flash column chromatograph to afford glycosides. After purification, **46** was obtained as a colourless oil (46 mg, 78%, α/β > 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 25H), 5.51 (d, *J* = 5.0 Hz, 1H), 5.03 (d, *J* = 2.7 Hz, 1H), 4.90 (dd, *J* = 11.1, 8.7 Hz, 2H), 4.76 – 4.55 (m, 8H), 4.53 (d, *J* = 4.1 Hz, 1H), 4.50 (s, 1H), 4.25 (dd, *J* = 5.0, 2.4 Hz, 1H), 4.19 (dd, *J* = 7.9, 1.7 Hz, 1H), 4.08 (t, *J* = 9.6 Hz, 1H), 3.98 (dt, *J* = 22.2, 5.8 Hz, 4H), 3.88 – 3.82 (m, 2H), 3.79 – 3.63 (m, 4H), 3.59 (d, *J* = 11.8 Hz, 2H), 3.46 – 3.39 (m, 1H), 2.21 (td, *J* = 12.5, 3.4 Hz, 1H), 2.04 (dd, *J* = 12.6, 4.2 Hz, 1H), 1.58 (s, 3H), 1.50 (s, 3H), 1.41 (s, 3H), 1.29 (s, 3H), 1.09 (s, 21H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 138.9, 138.8, 138.6, 138.6, 128.4, 128.3, 128.3, 128.2, 128.2, 128.1, 128.0, 127.7, 127.5, 127.5, 127.5, 127.4, 127.3, 127.3, 109.3, 108.4, 97.3, 96.8, 96.4, 80.1, 75.6, 75.1, 74.8, 74.6, 74.2, 73.3, 72.9, 72.4, 71.1, 70.7, 70.5, 70.5, 69.3, 65.6, 65.5, 65.1, 62.8, 30.9, 26.1, 26.0, 24.9, 24.5, 18.0, 18.0, 12.0.;

IR (CH_2Cl_2, cm^{-1}) 2938, 2865, 1454, 1381, 1254, 1210, 1069, 1003, 883, 735, 697.;
ESI HRMS calcd for $C_{68}H_{90}NaO_{15}Si$ $[M + Na]^+$ 1197.5941, found 1197.5938.

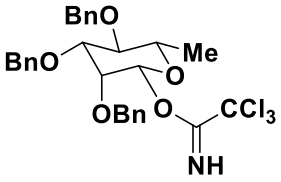
Galactose donor



S-4

^1H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.39 – 7,27 (m, 20H), 6.55 (d, *J* = 2.3 Hz, 1H), 5.00 (d, *J* = 11.3 Hz, 1H), 4.87 – 4.78 (m, 4H), 4.63 (d, *J* = 11.2 Hz, 1H), 4.51 – 4.42 (m, 2H), 4.28 (d, *J* = 2.6 Hz, 1H), 4.26 (d, *J* = 2.5 Hz, 1H), 4.21 – 4.04 (m, 2H), 3.67 – 3.57 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.3, 138.5, 138.5, 138.4, 137.8, 128.4, 128.3, 128.2, 128.2, 128.1, 127.8, 127.7, 127.6, 127.6, 127.5, 127.4, 95.2, 77.9, 75.9, 74.9, 74.7, 73.4, 73.0, 72.9, 72.2, 68.3.;

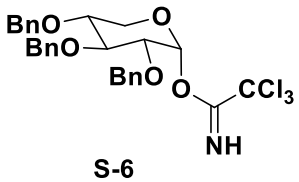
Rhamnose donor



S-5

^1H NMR (500 MHz, $CDCl_3$) δ 8.54 (s, 1H), 7.47 – 7.42 (m, 2H), 7.41 – 7.30 (m, 13H), 6.28 (d, *J* = 1.9 Hz, 1H), 5.00 (d, *J* = 10.7 Hz, 1H), 4.80 (s, 2H), 4.72 – 4.59 (m, 3H), 3.98 – 3.86 (m, 3H), 3.74 (t, *J* = 9.4 Hz, 1H), 1.39 (d, *J* = 6.2 Hz, 4H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 160.5, 138.3, 138.1, 137.9, 128.4, 128.4, 128.4, 128.4, 128.4, 128.3, 128.2, 127.9, 127.9, 127.8, 127.8, 127.7, 96.0, 79.8, 78.9, 75.6, 73.9, 72.8, 72.3, 71.1, 18.0.;

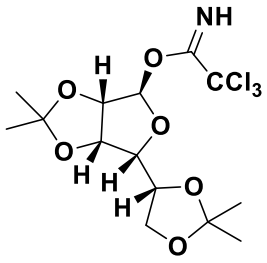
Xylose donor



S-6

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.74 (s, 1H), 7.42 – 7.26 (m, 15H), 5.86 (d, *J* = 6.8 Hz, 1H), 5.03 – 4.87 (m, 2H), 4.85 – 4.76 (m, 3H), 4.73 – 4.60 (m, 1H), 4.07 – 3.93 (m, 1H), 3.85 – 3.66 (m, 3H), 3.50 (dd, *J* = 11.9, 8.8 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.2, 138.4, 137.9, 137.9, 128.5, 128.3, 128.3, 128.3, 128.0, 127.9, 127.9, 127.8, 127.8, 127.5, 98.8, 94.3, 82.9, 80.8, 80.0, 79.0, 77.2, 77.1, 75.7, 75.3, 74.8, 73.7, 73.1, 73.0, 64.5, 62.4.;

Mannofuranose derived donor



S-8

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (s, 1H), 6.28 (s, 1H), 4.94 (dd, *J* = 5.9, 3.4 Hz, 1H), 4.88 (d, *J* = 5.9 Hz, 1H), 4.45 (ddd, *J* = 8.2, 6.2, 4.2 Hz, 1H), 4.19 – 4.11 (m, 2H), 4.06 (dd, *J* = 8.9, 4.2 Hz, 1H), 1.53 (s, 3H), 1.47 (s, 3H), 1.40 (d, *J* = 0.4 Hz, 3H), 1.39 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 160.7, 113.4, 109.4, 104.8, 84.8, 82.9, 79.2, 72.7, 67.1, 27.0, 26.0, 25.1, 24.7;

3. References

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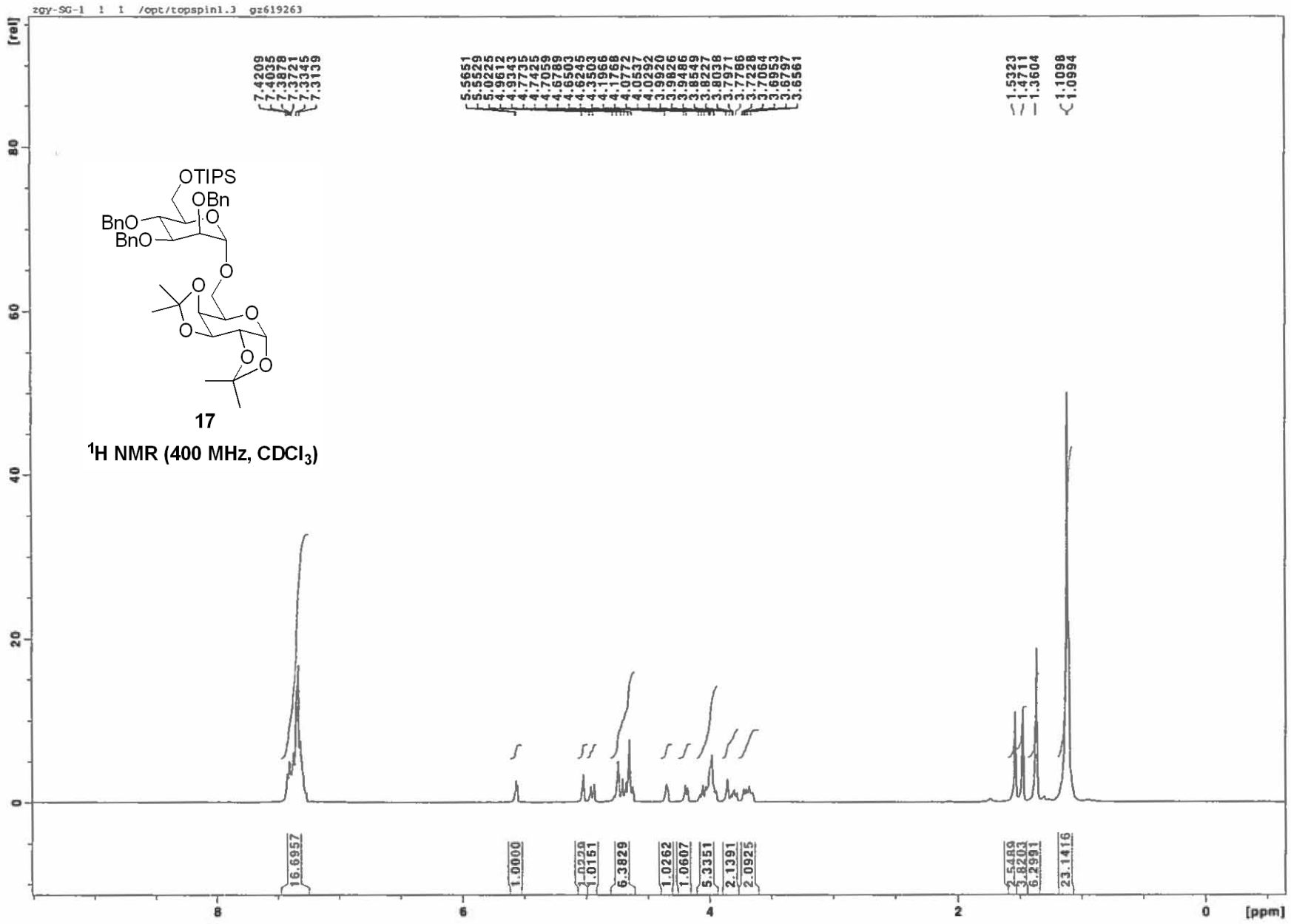
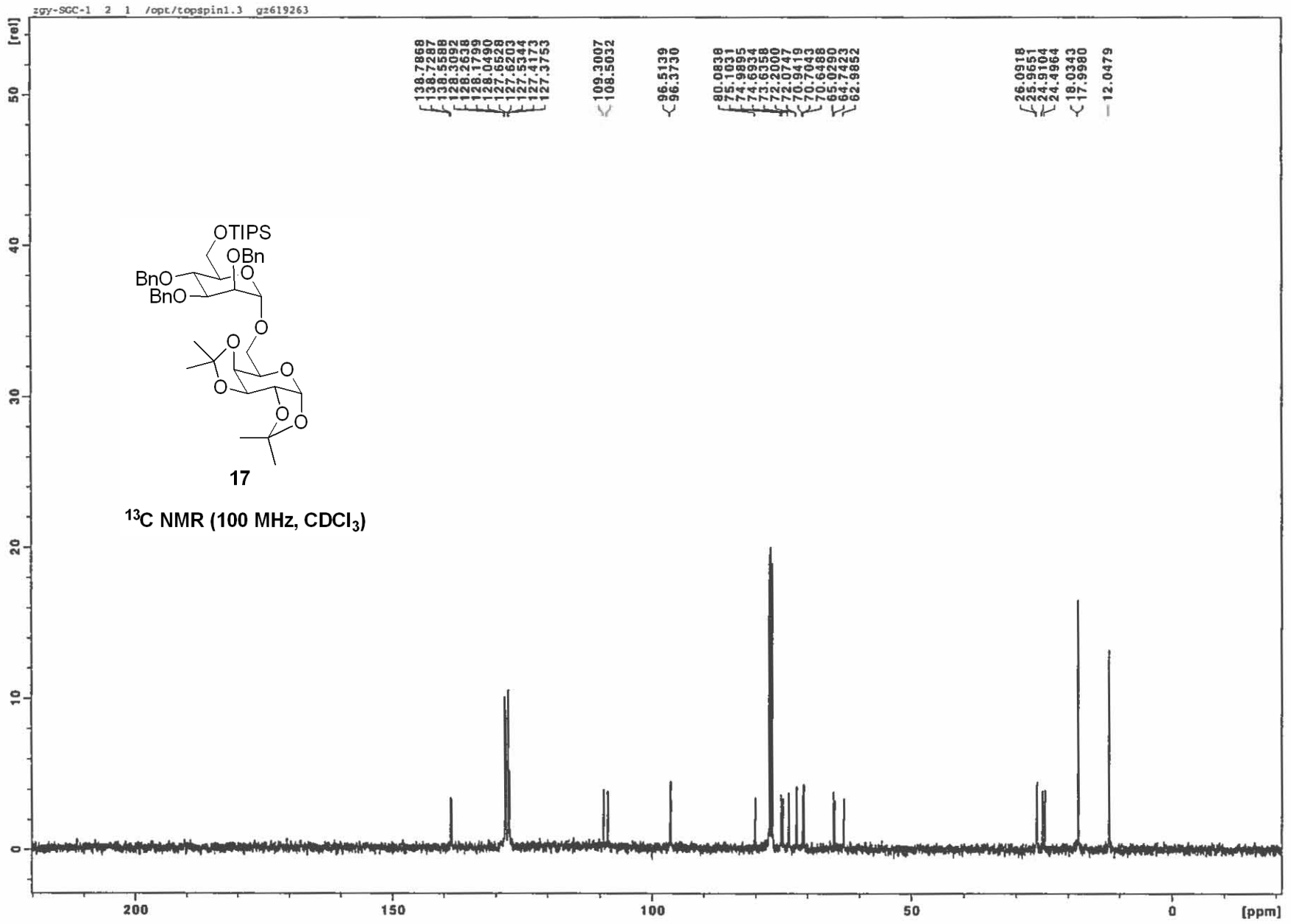
4. Copies of the Related NMR Spectra



17

^{1}H NMR (400 MHz, $CDCl_3$)



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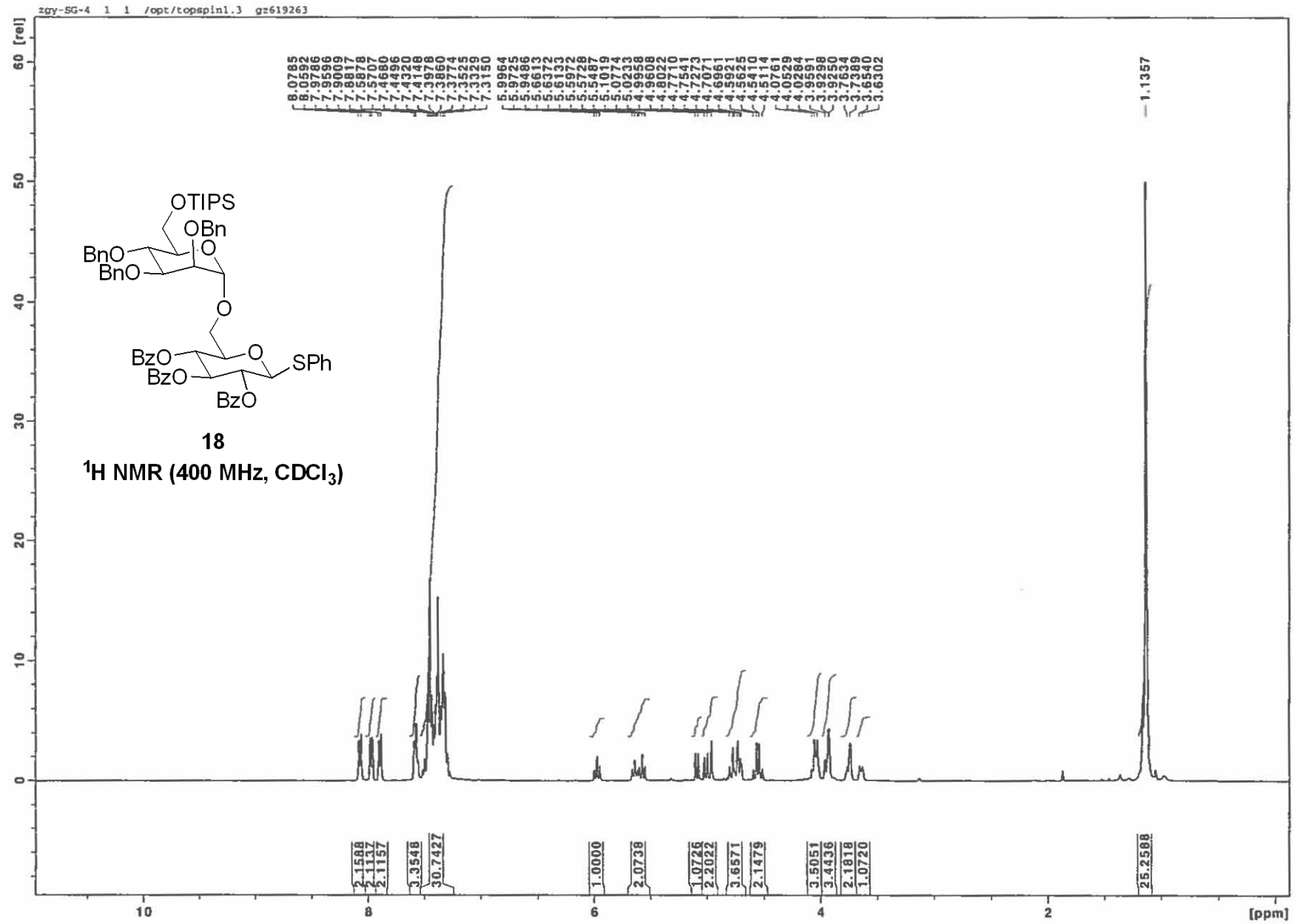
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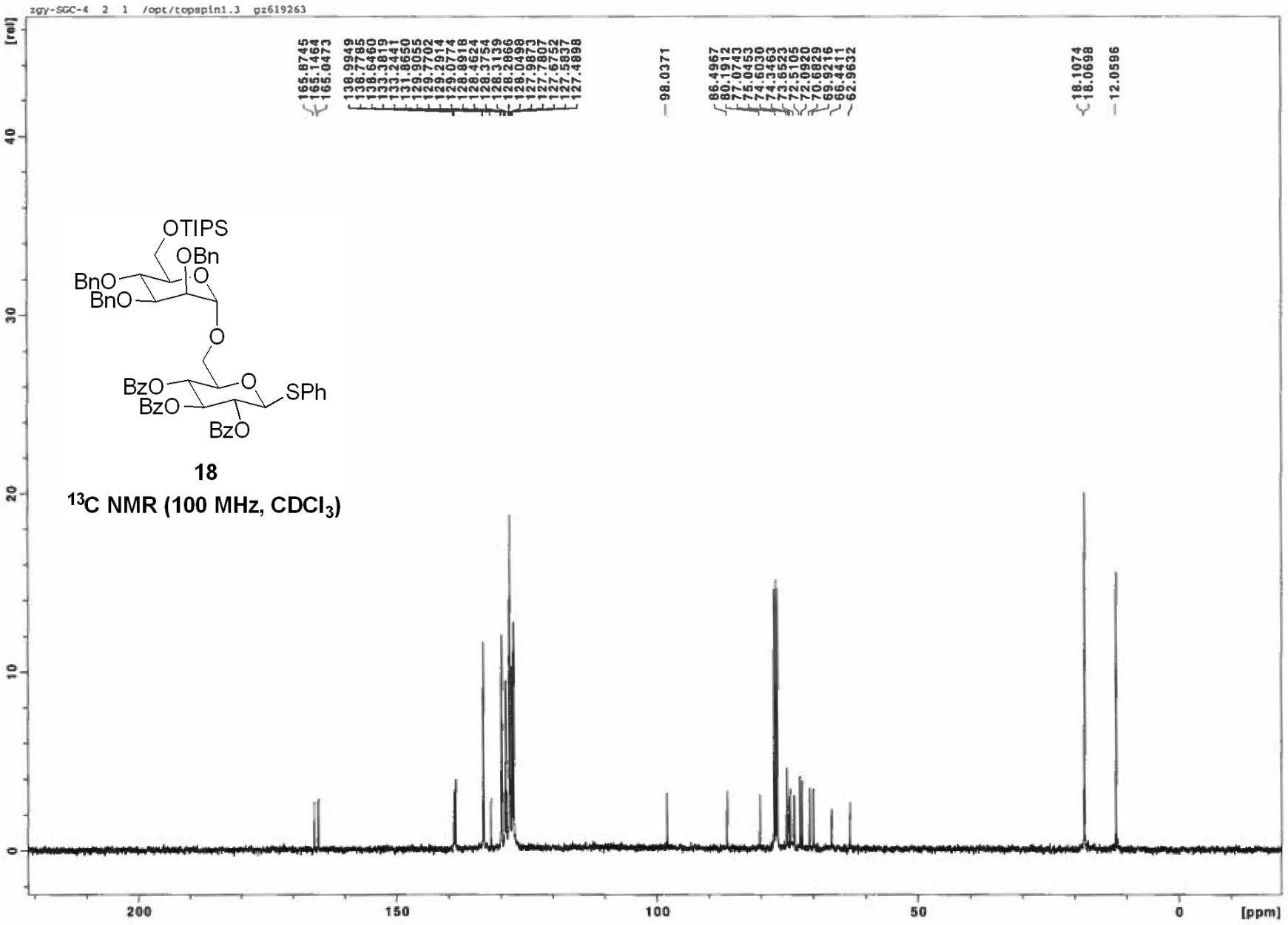
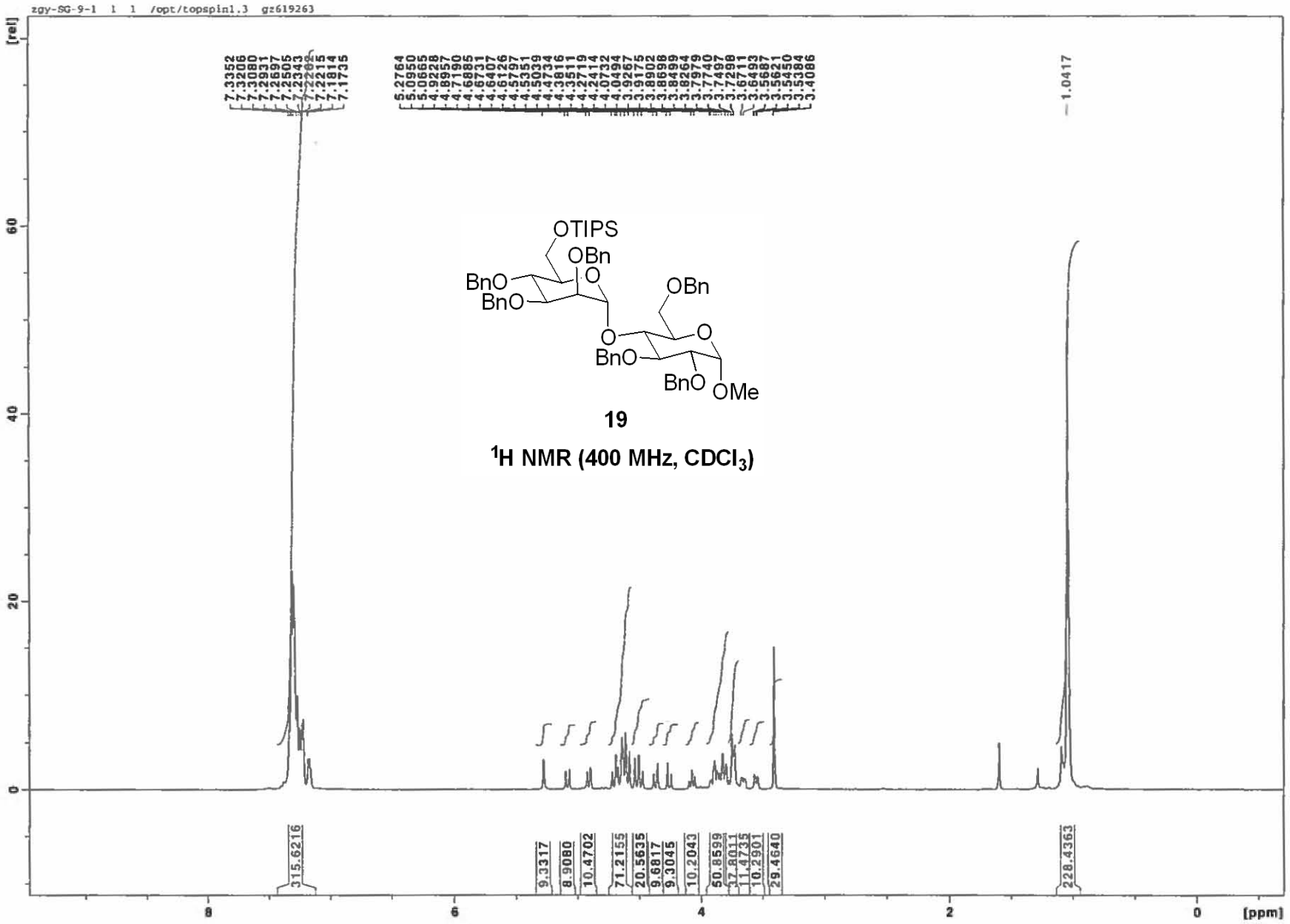


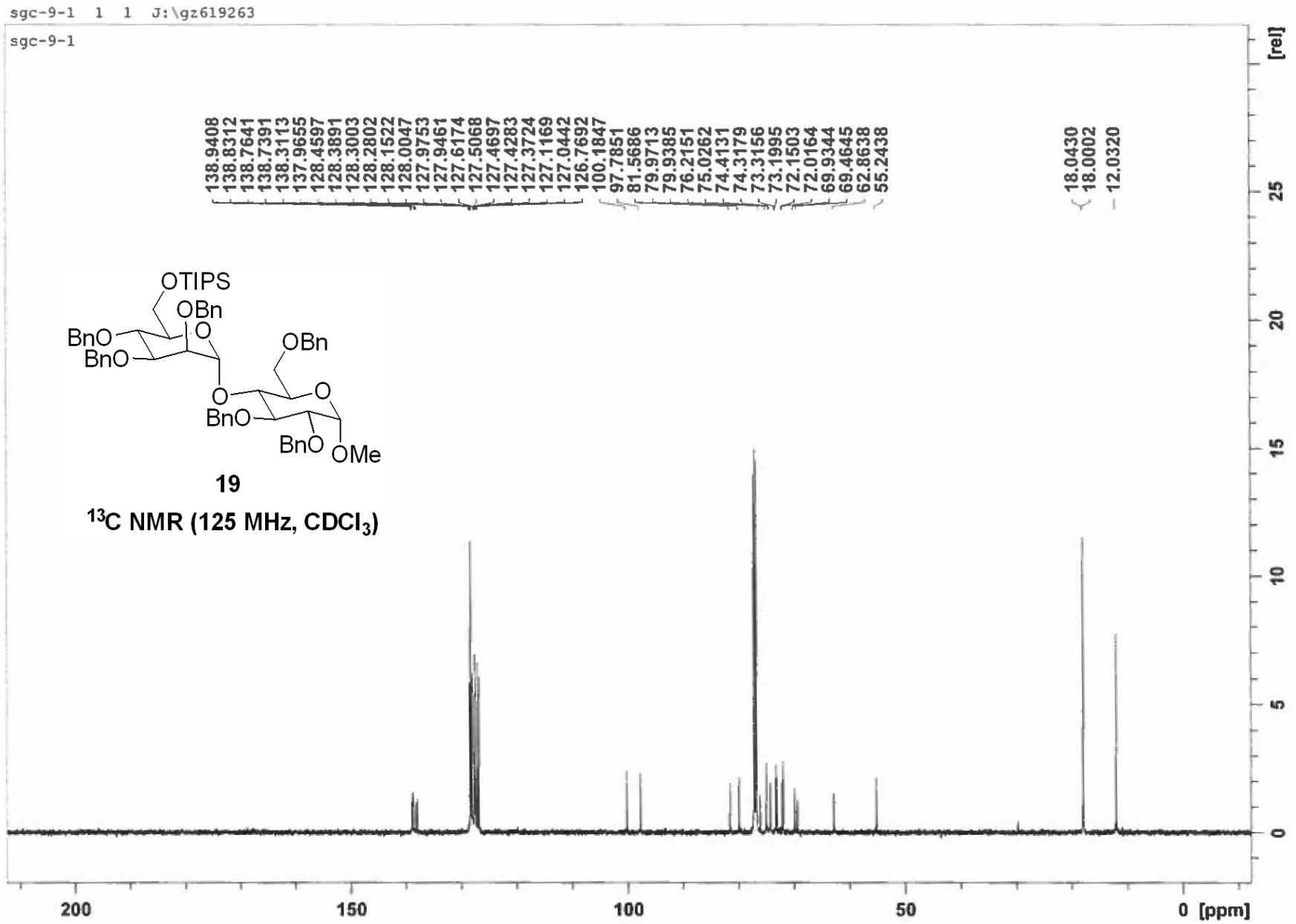
18

^{13}C NMR (100 MHz, $CDCl_3$)



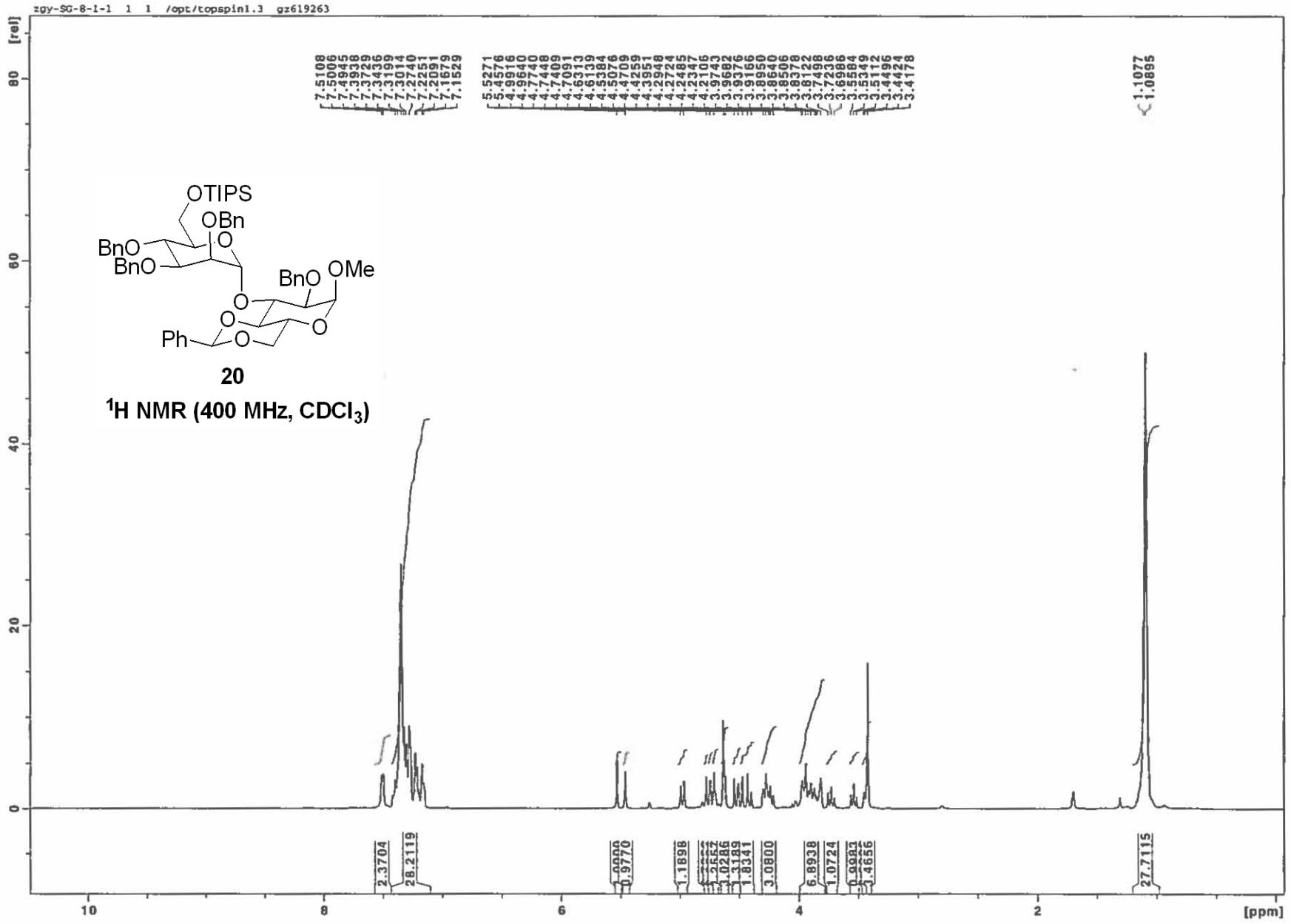
19

^{1}H NMR (400 MHz, $CDCl_3$)



19

^{13}C NMR (125 MHz, $CDCl_3$)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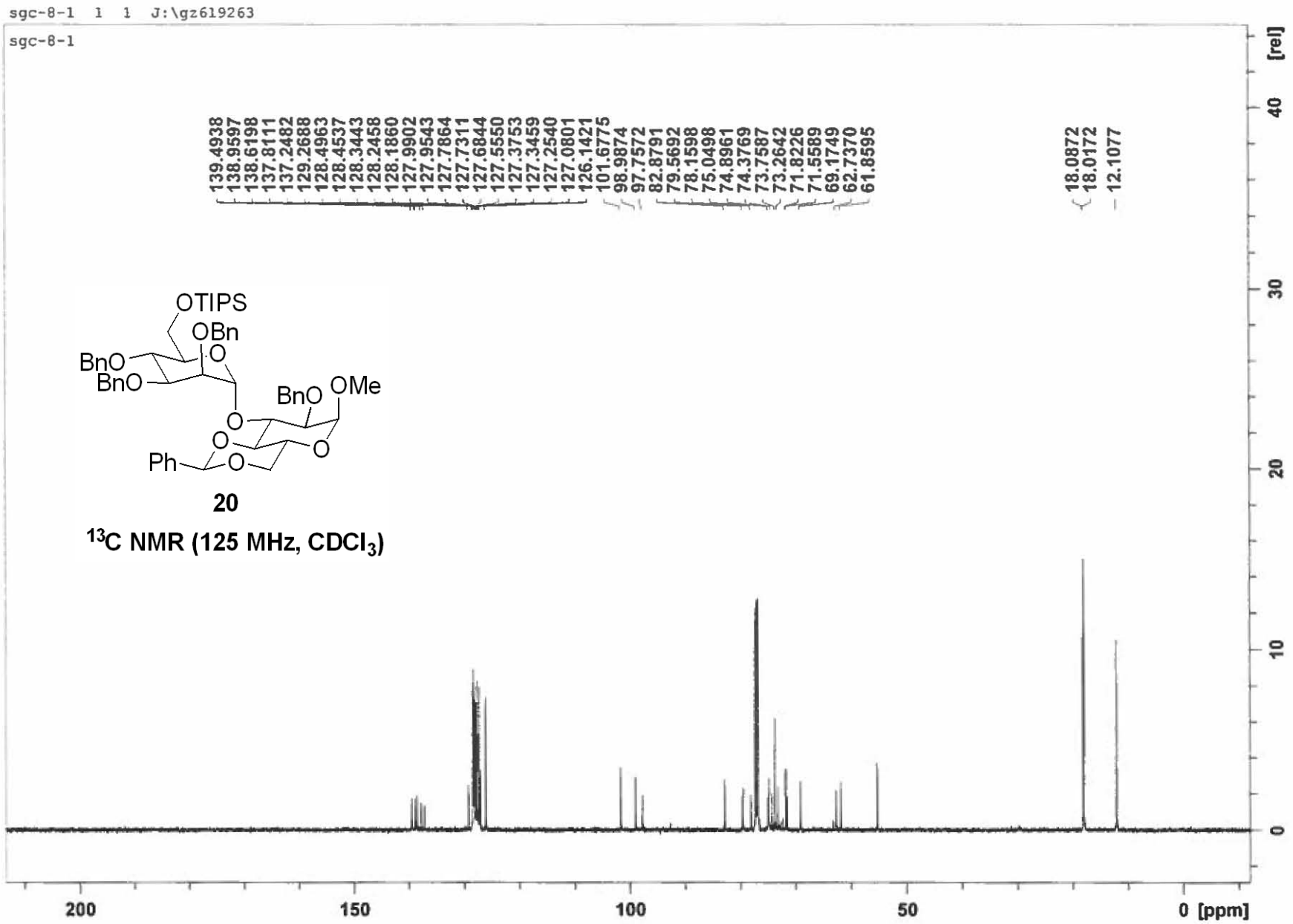
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150
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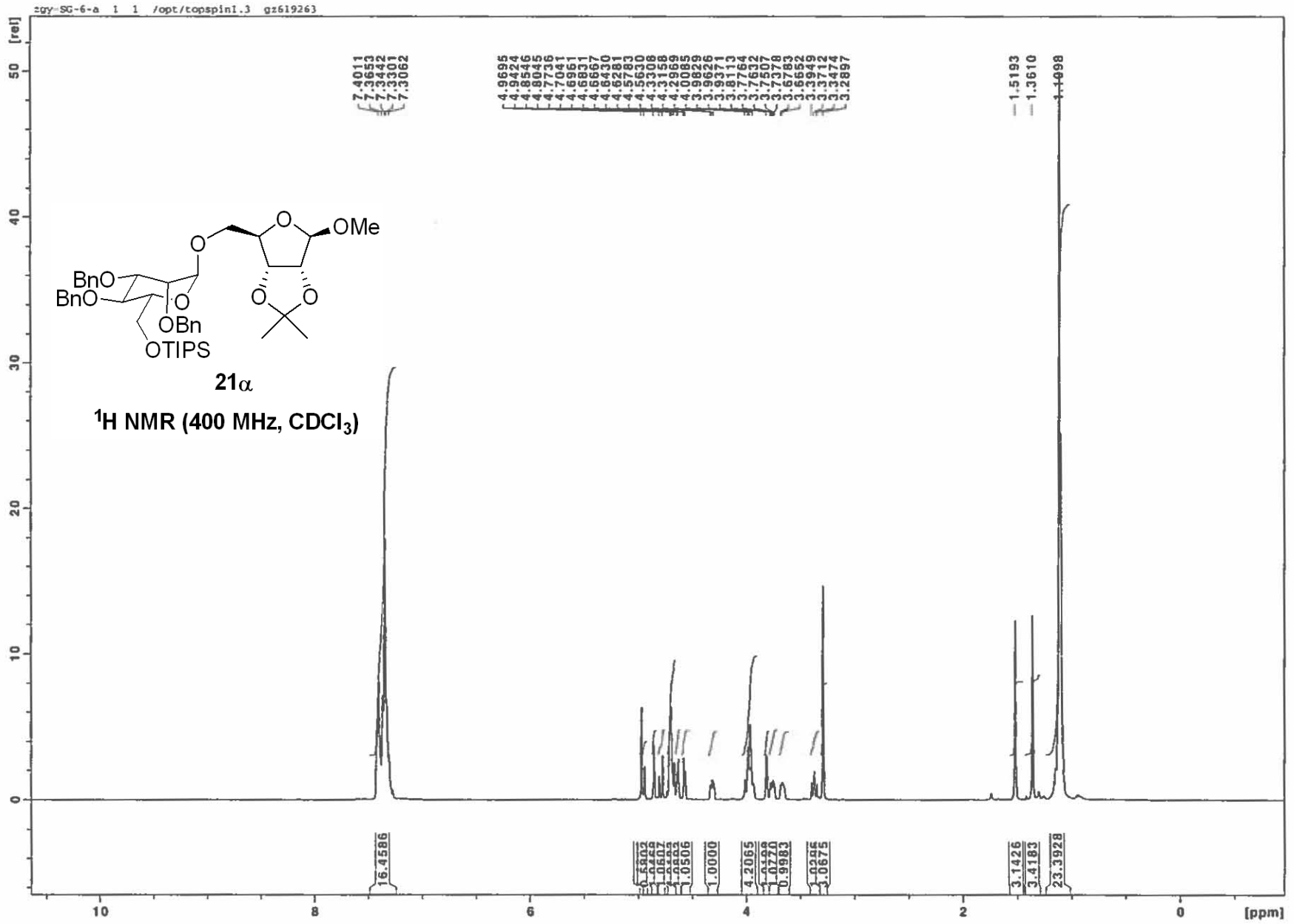
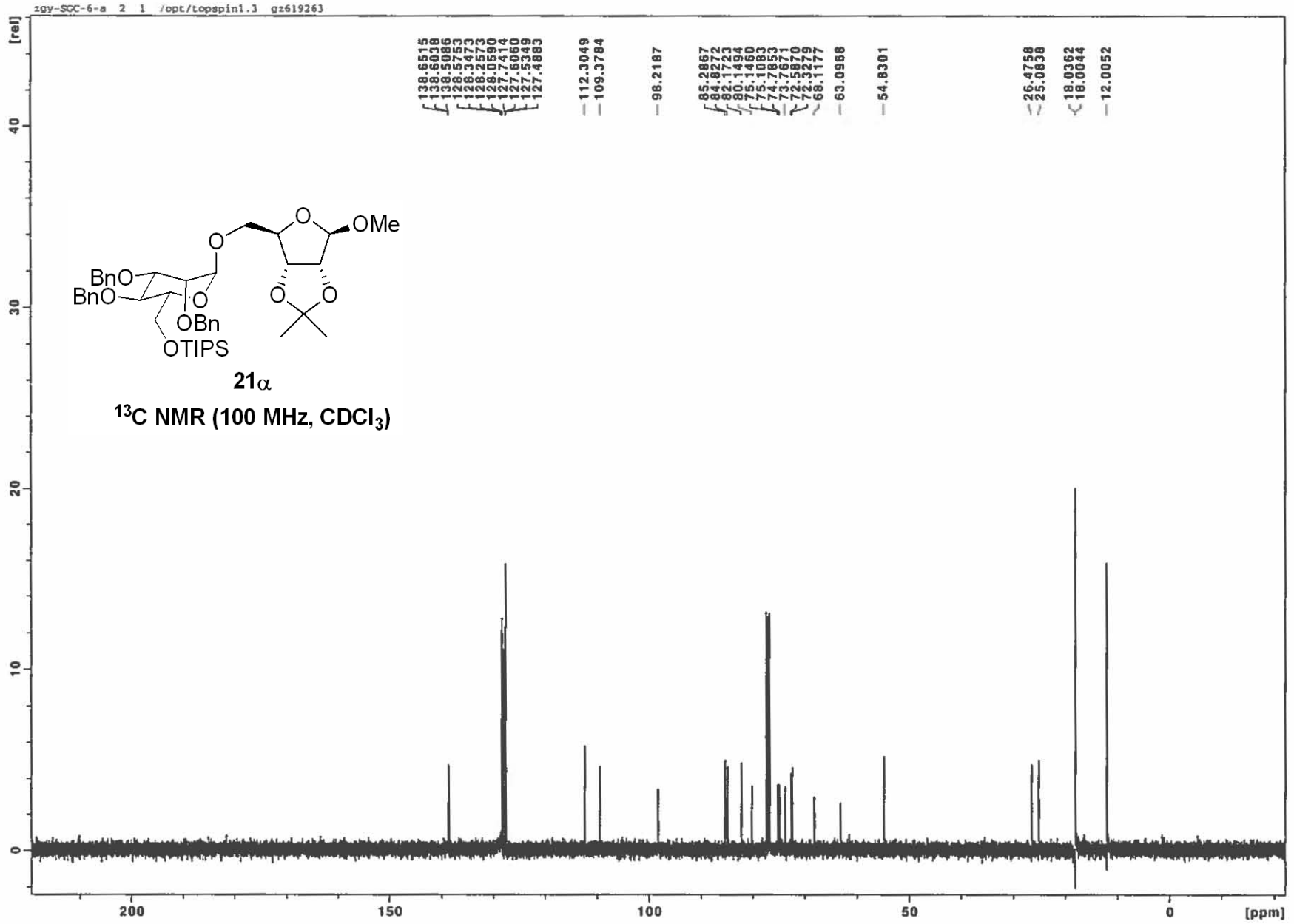


21α

^{1}H NMR (400 MHz, $CDCl_3$)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21α
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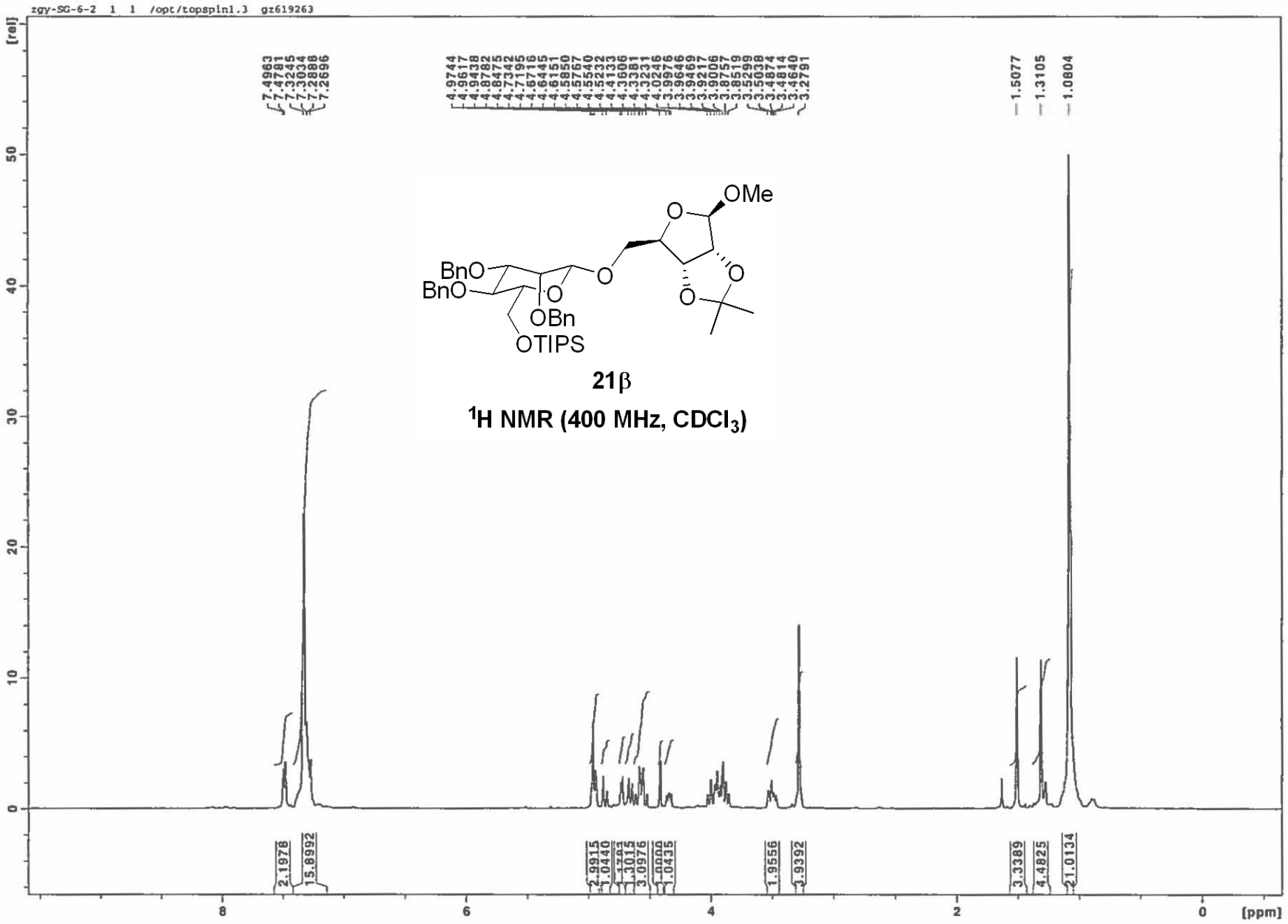
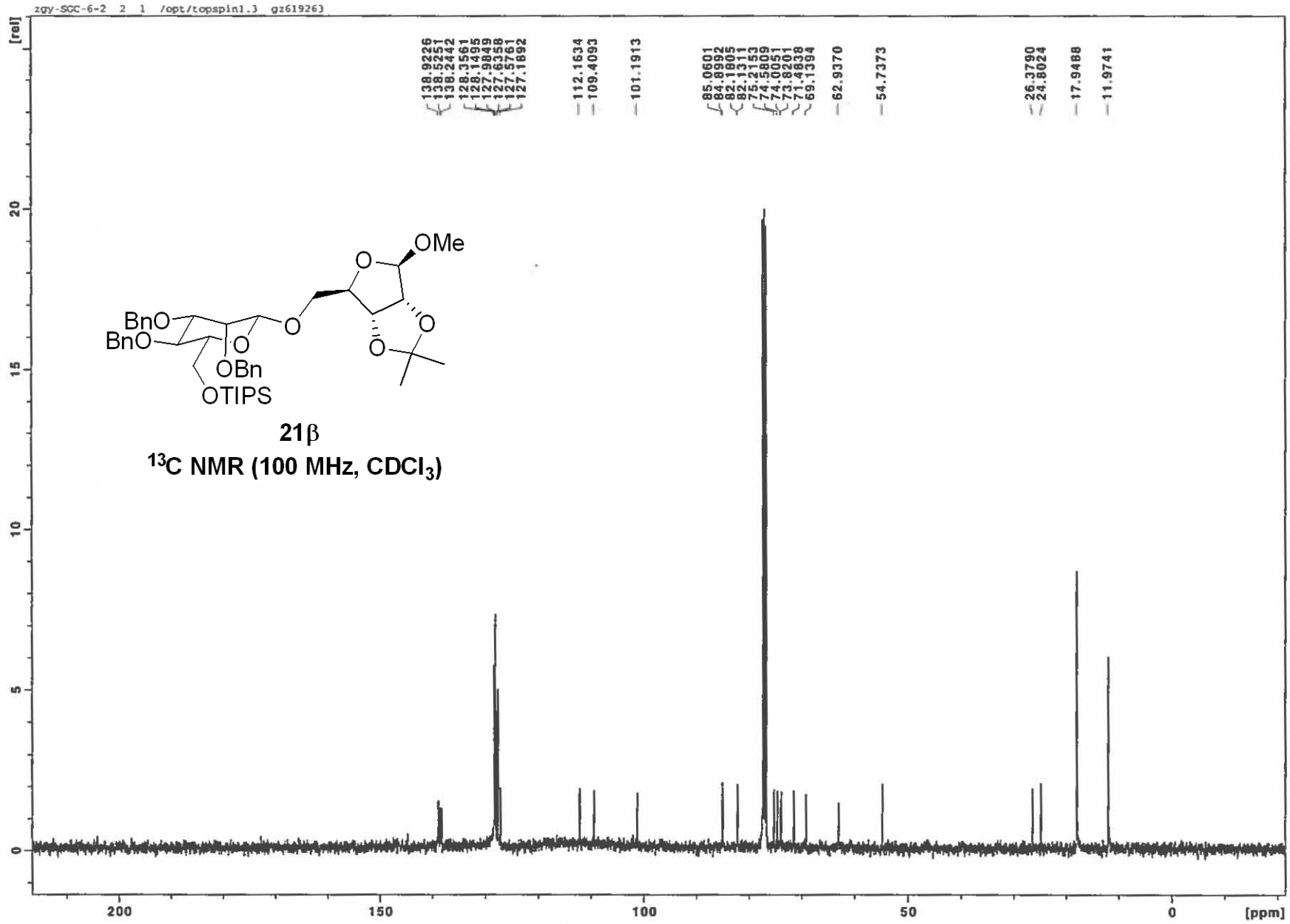


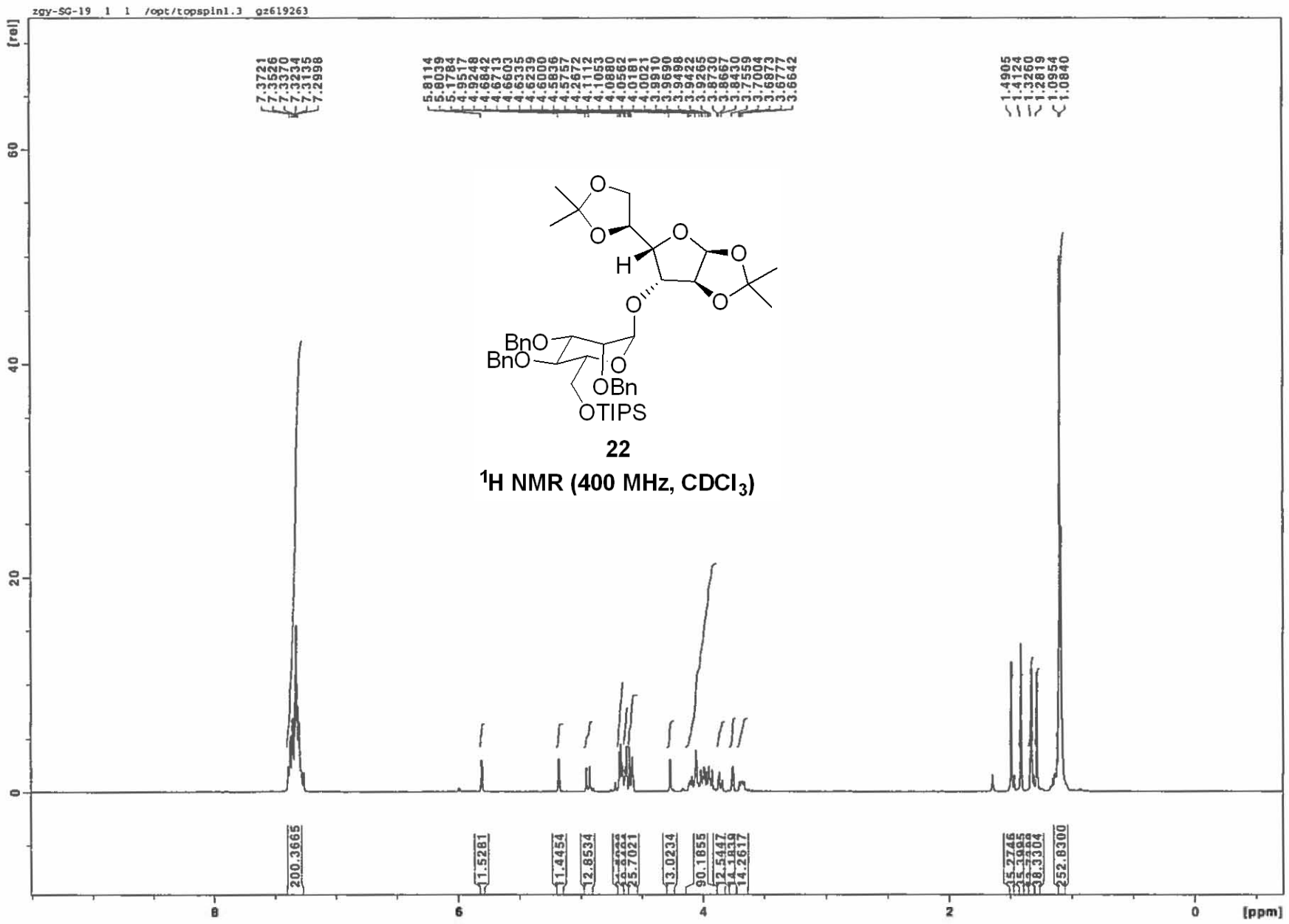
21β

^{1}H NMR (400 MHz, $CDCl_3$)



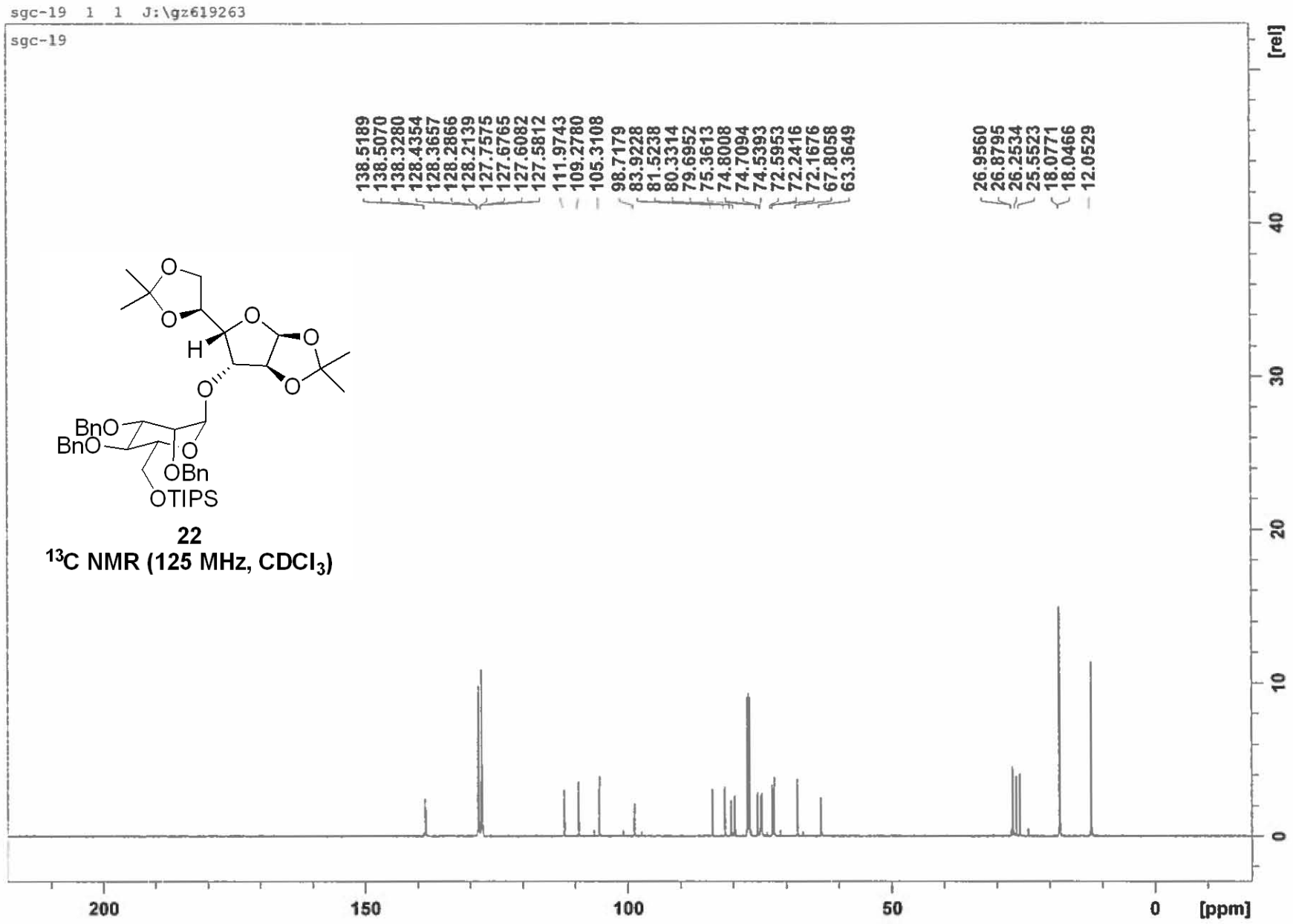
21β

^{13}C NMR (100 MHz, $CDCl_3$)



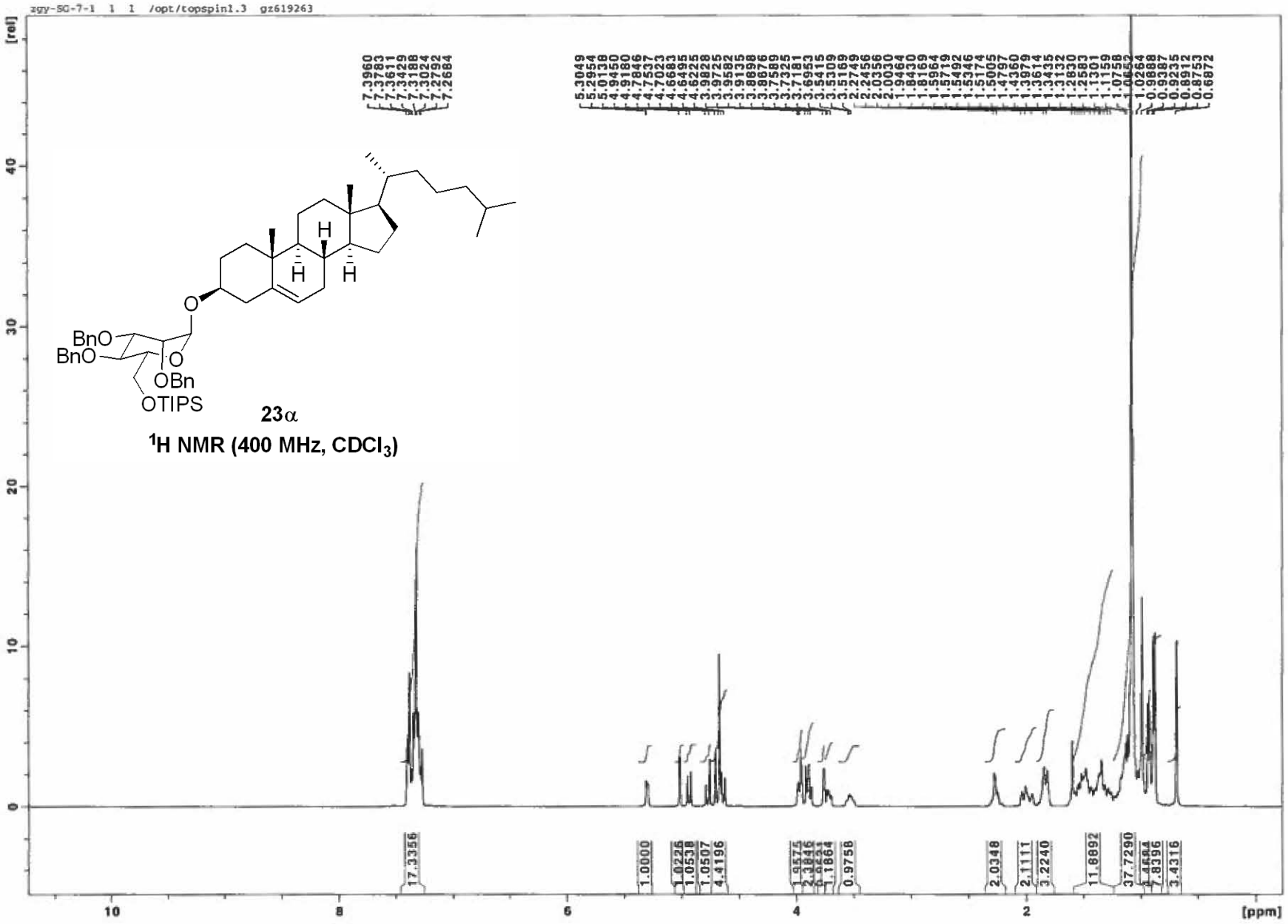
22

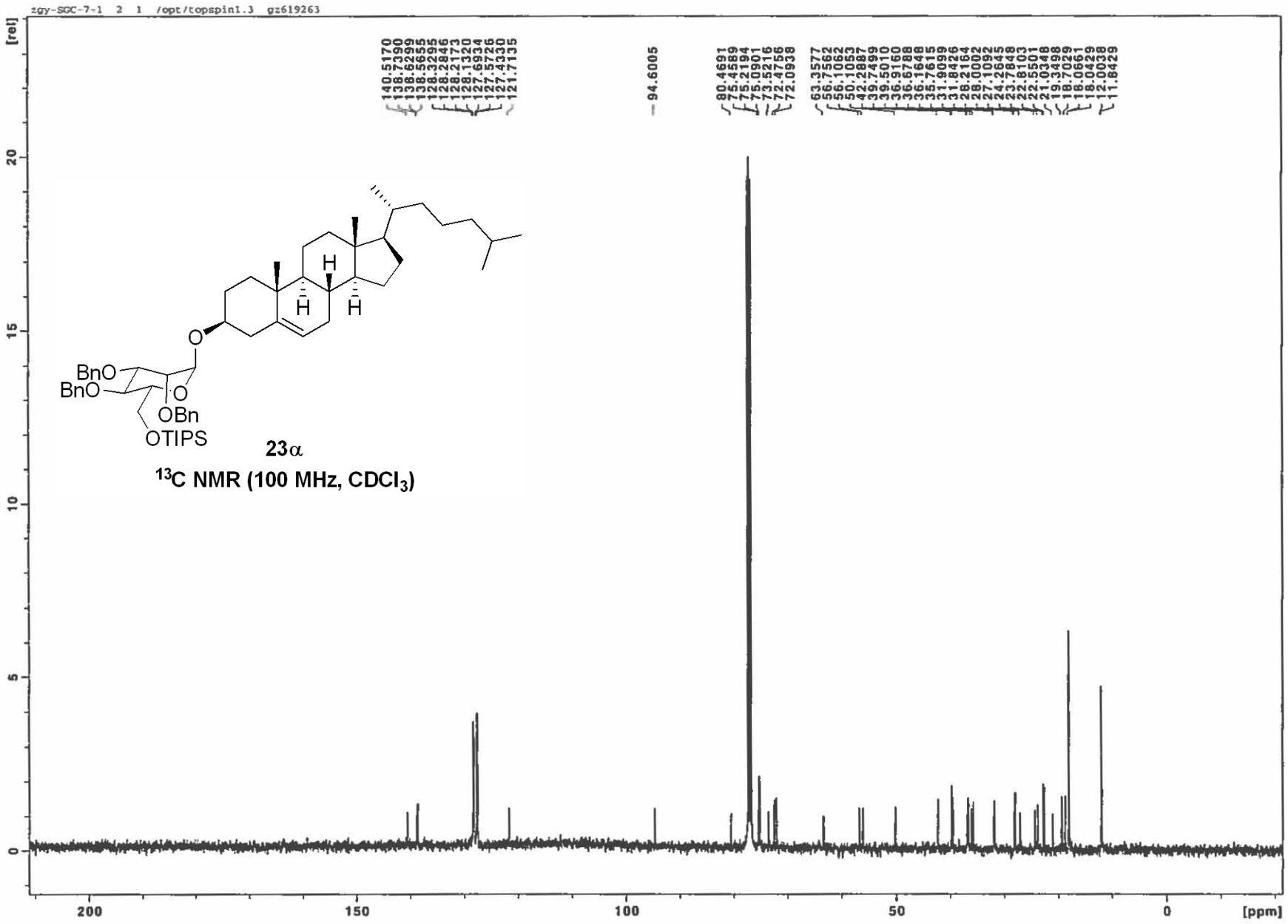
^{1}H NMR (400 MHz, $CDCl_3$)



22

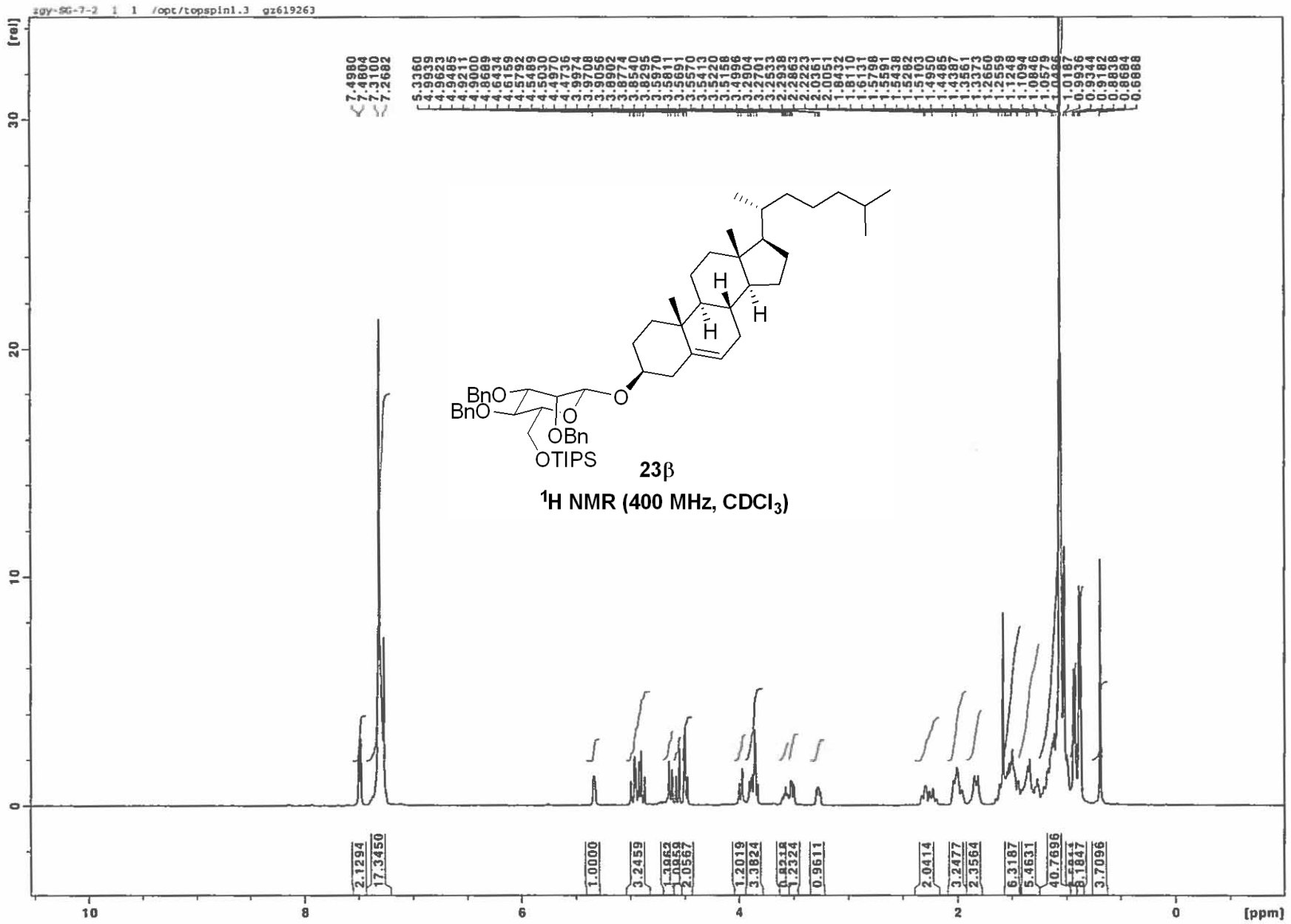
^{13}C NMR (125 MHz, $CDCl_3$)


zgy-SG-7-1 1 1 /opt/topspin1.3 gz619263
[rel]
BnO
BnO
O
OBn
OTIPS
H
H
H
O
23α
1H NMR (400 MHz, CDCl3)
[ppm]



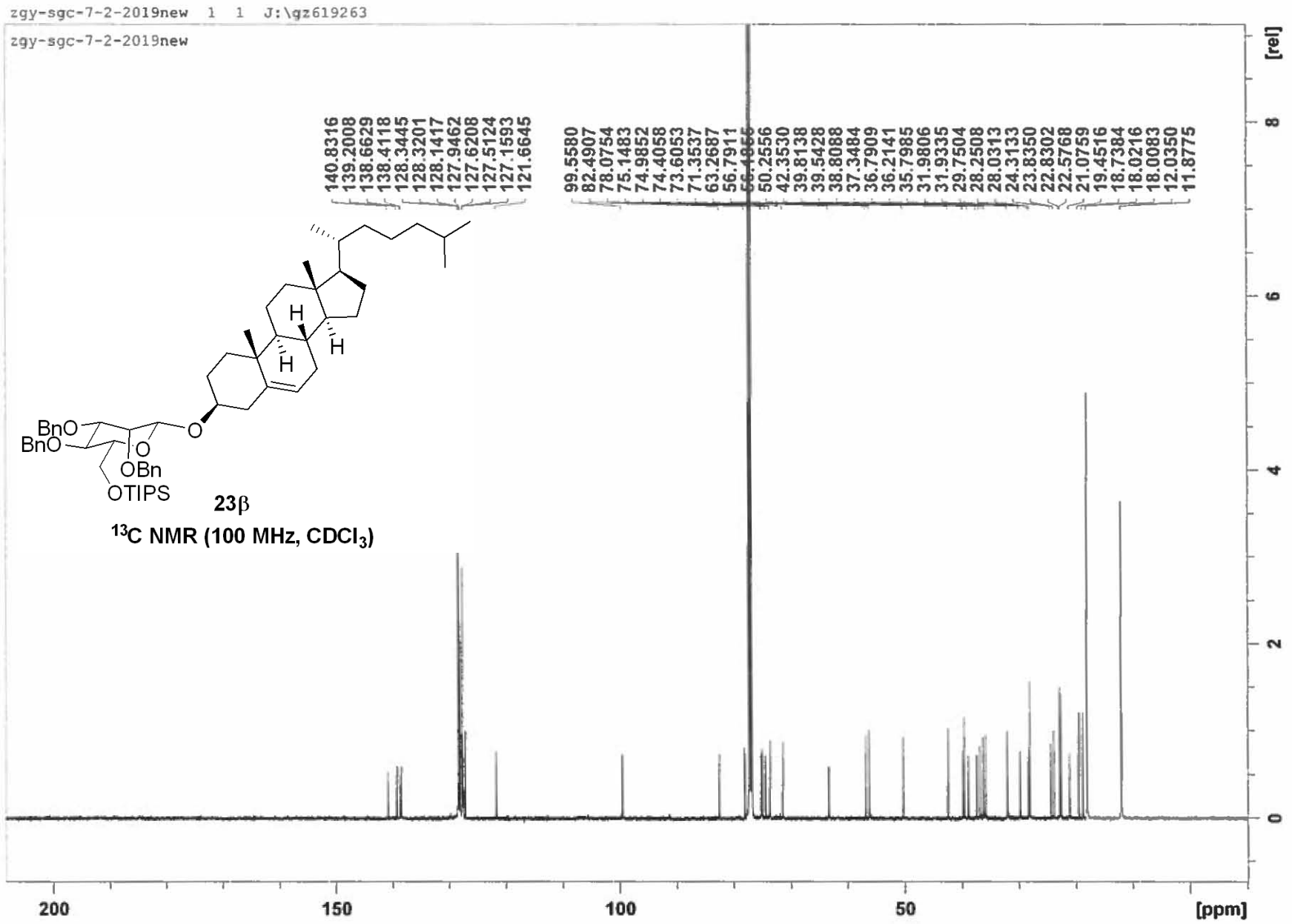
23α

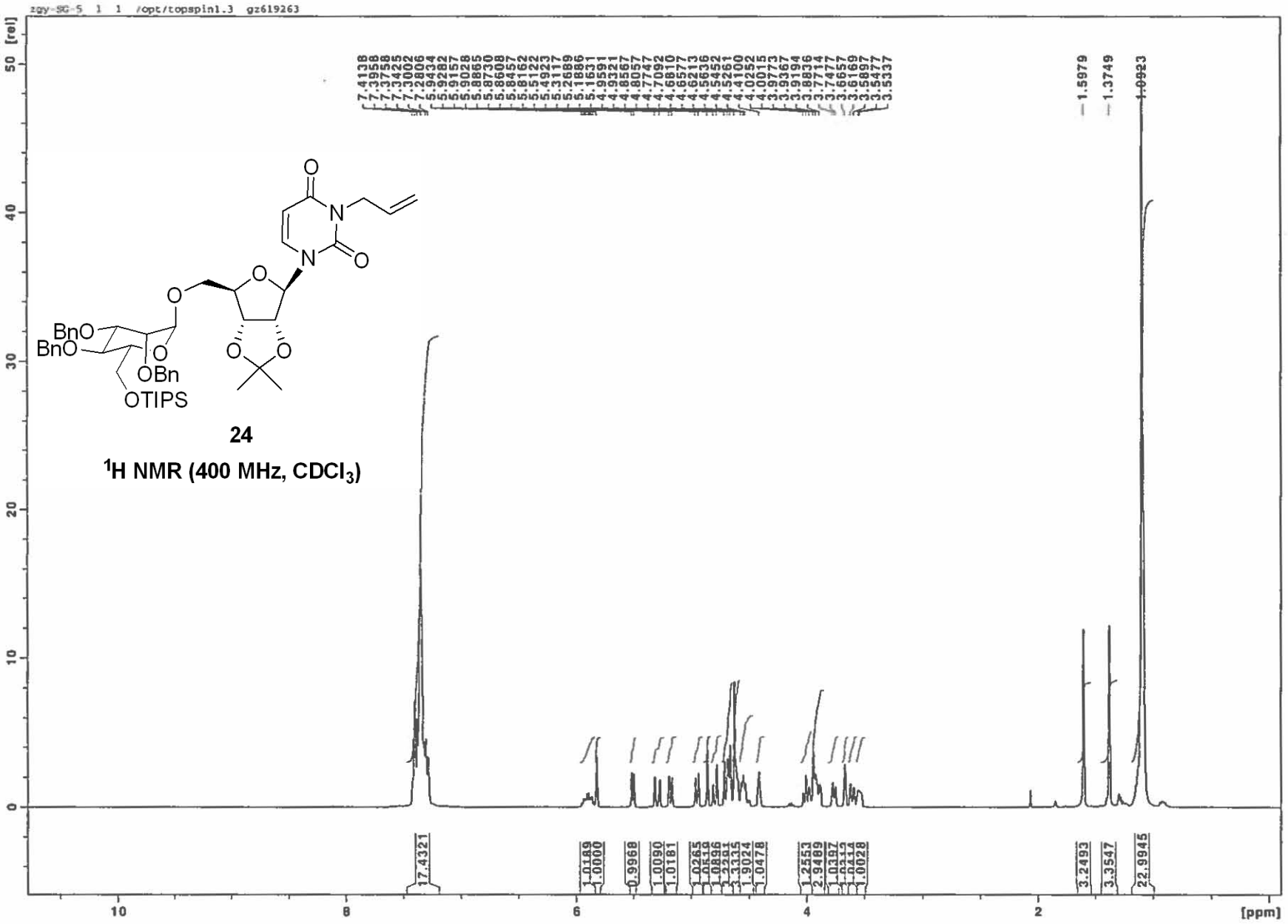
^{13}C NMR (100 MHz, $CDCl_3$)



23β

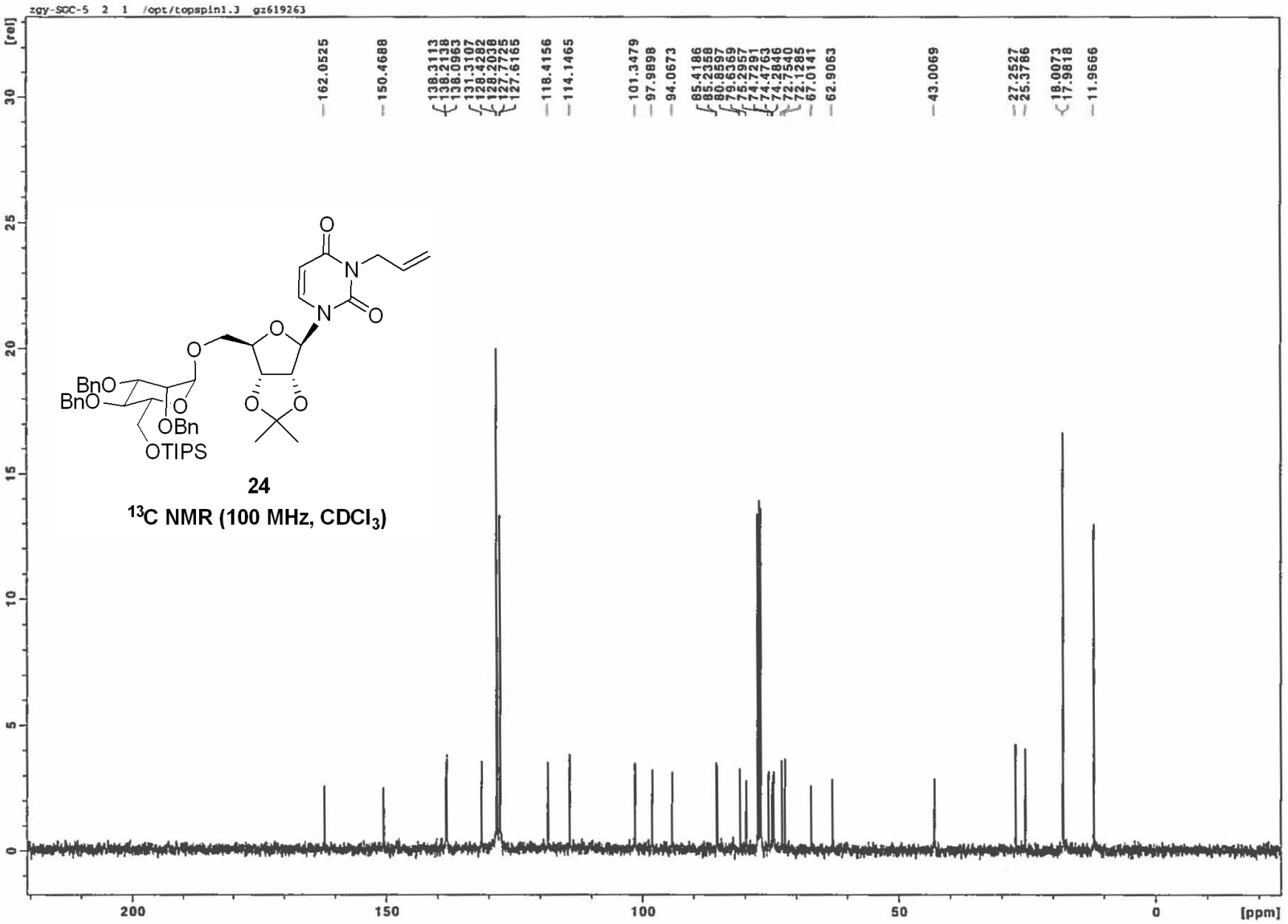
^{1}H NMR (400 MHz, $CDCl_3$)


zgy-sgc-7-2-2019new 1 1 J:\gz619263
zgy-sgc-7-2-2019new
140.8316
139.2008
138.6629
138.4118
128.3445
128.3201
128.1417
127.9462
127.6208
127.5124
127.1593
121.6645
99.5580
82.4907
78.0754
75.1483
74.9852
74.4058
73.6053
71.3537
63.2687
56.7911
50.2556
42.3530
39.8138
39.5428
38.8088
37.3484
36.7909
36.2141
35.7985
31.9806
31.9335
29.7504
28.2508
28.0313
24.3133
23.8350
22.8302
22.5768
21.0759
19.4516
18.7384
18.0216
18.0083
12.0350
11.8775
BnO
BnO
O
OBn
OTIPS
H
H
H
O
23β
13C NMR (100 MHz, CDCl3)
[rel]
8
6
4
2
0
200
150
100
50
[ppm]



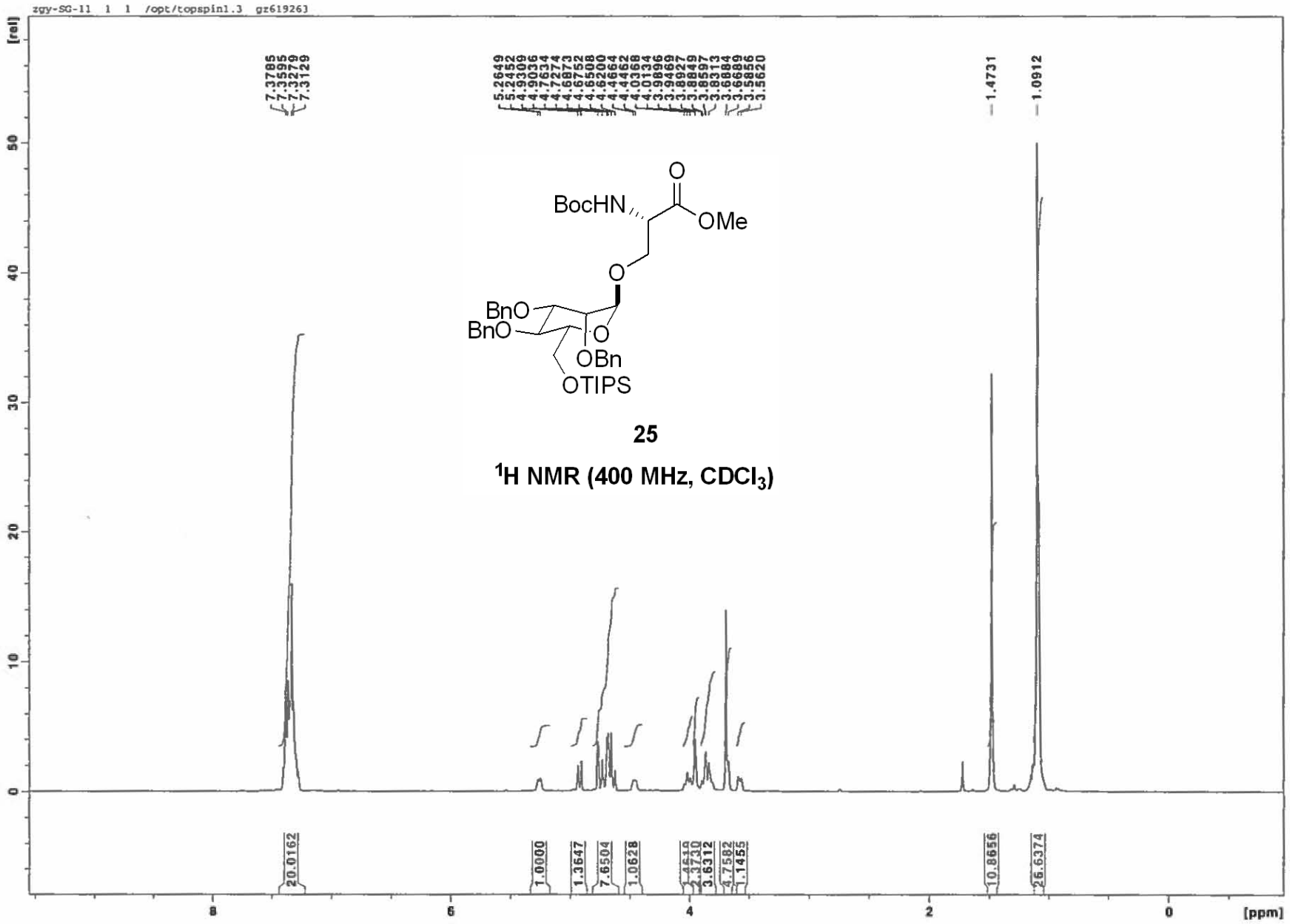
24

^{1}H NMR (400 MHz, $CDCl_3$)



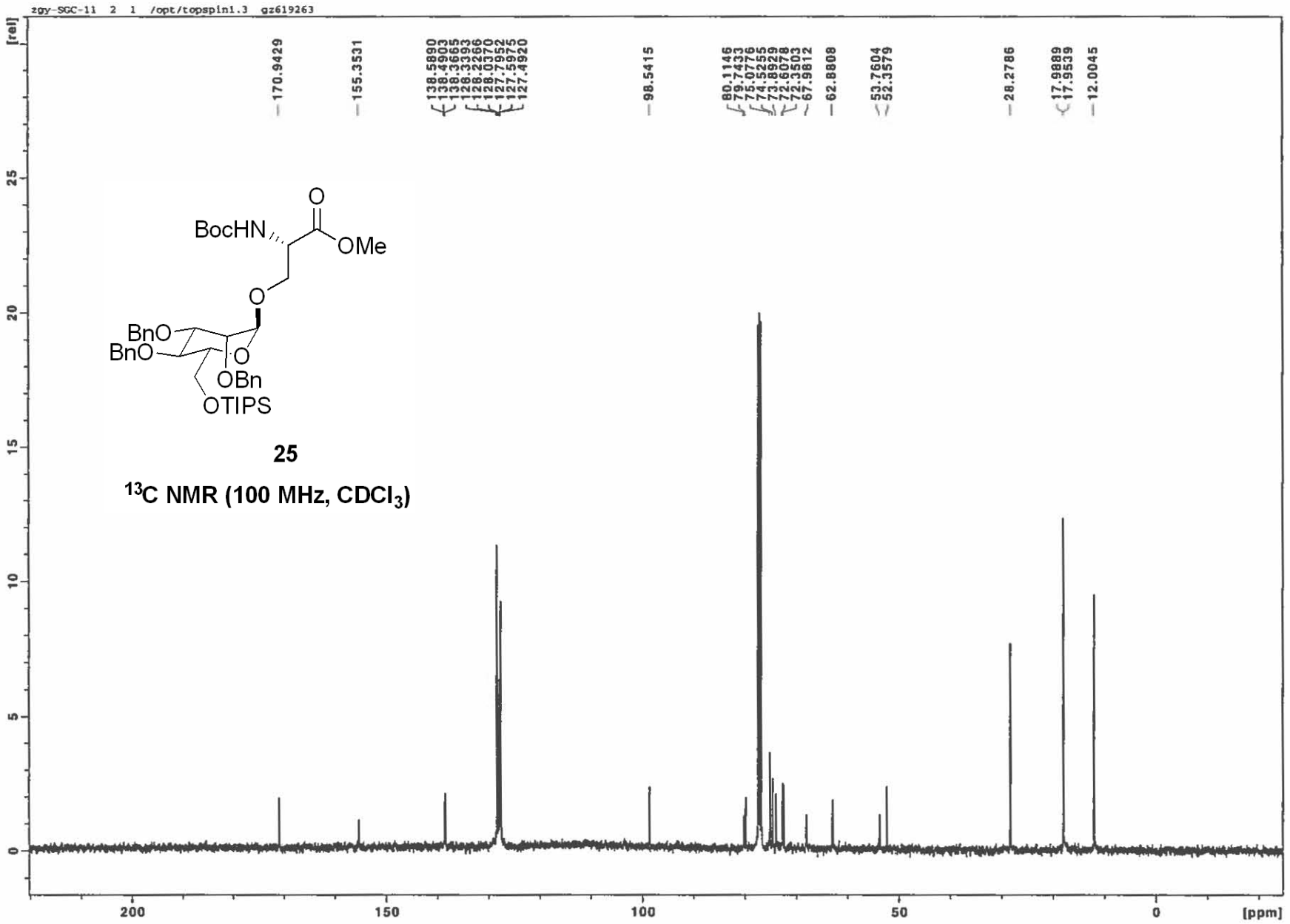
24

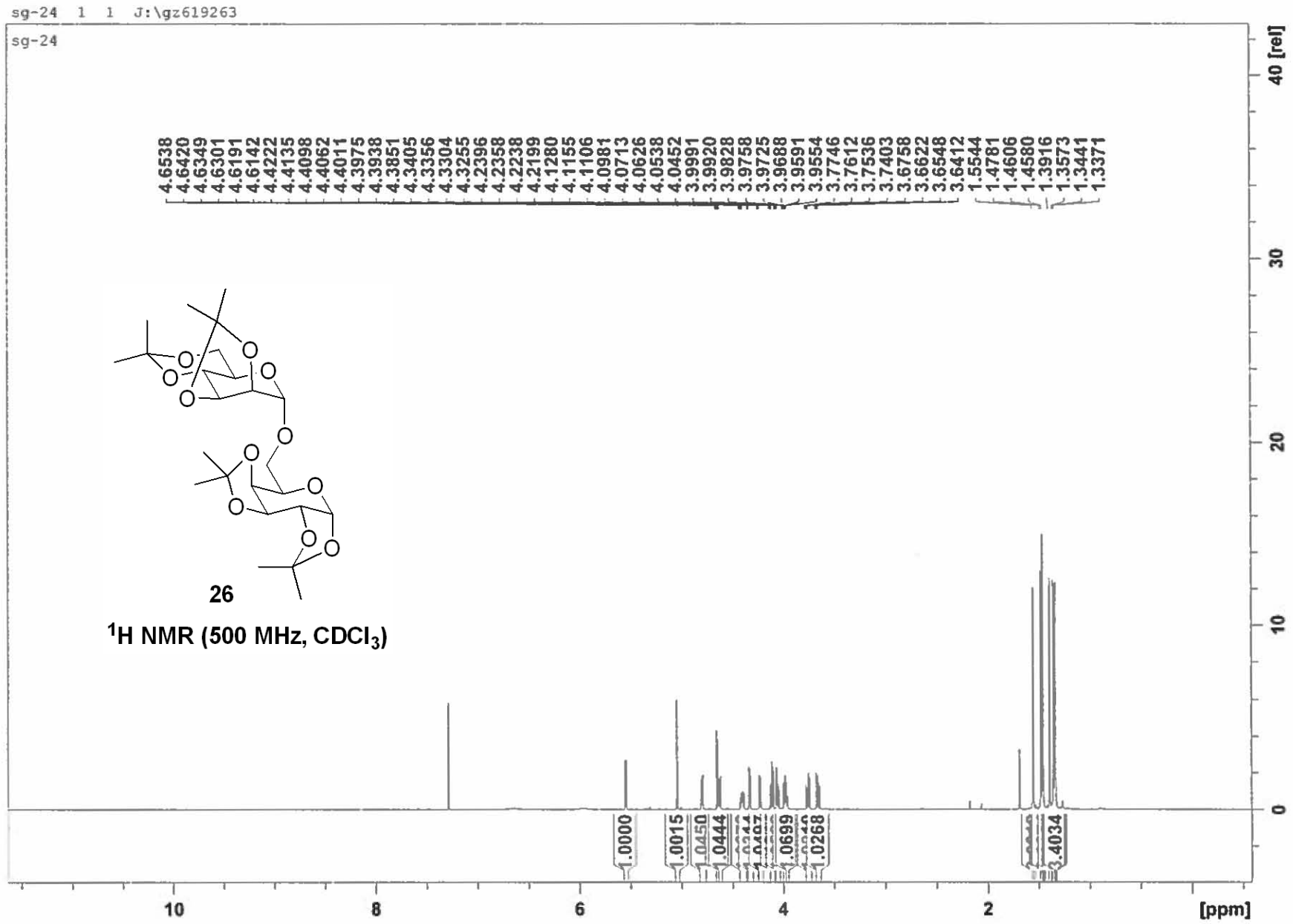
^{13}C NMR (100 MHz, $CDCl_3$)



25

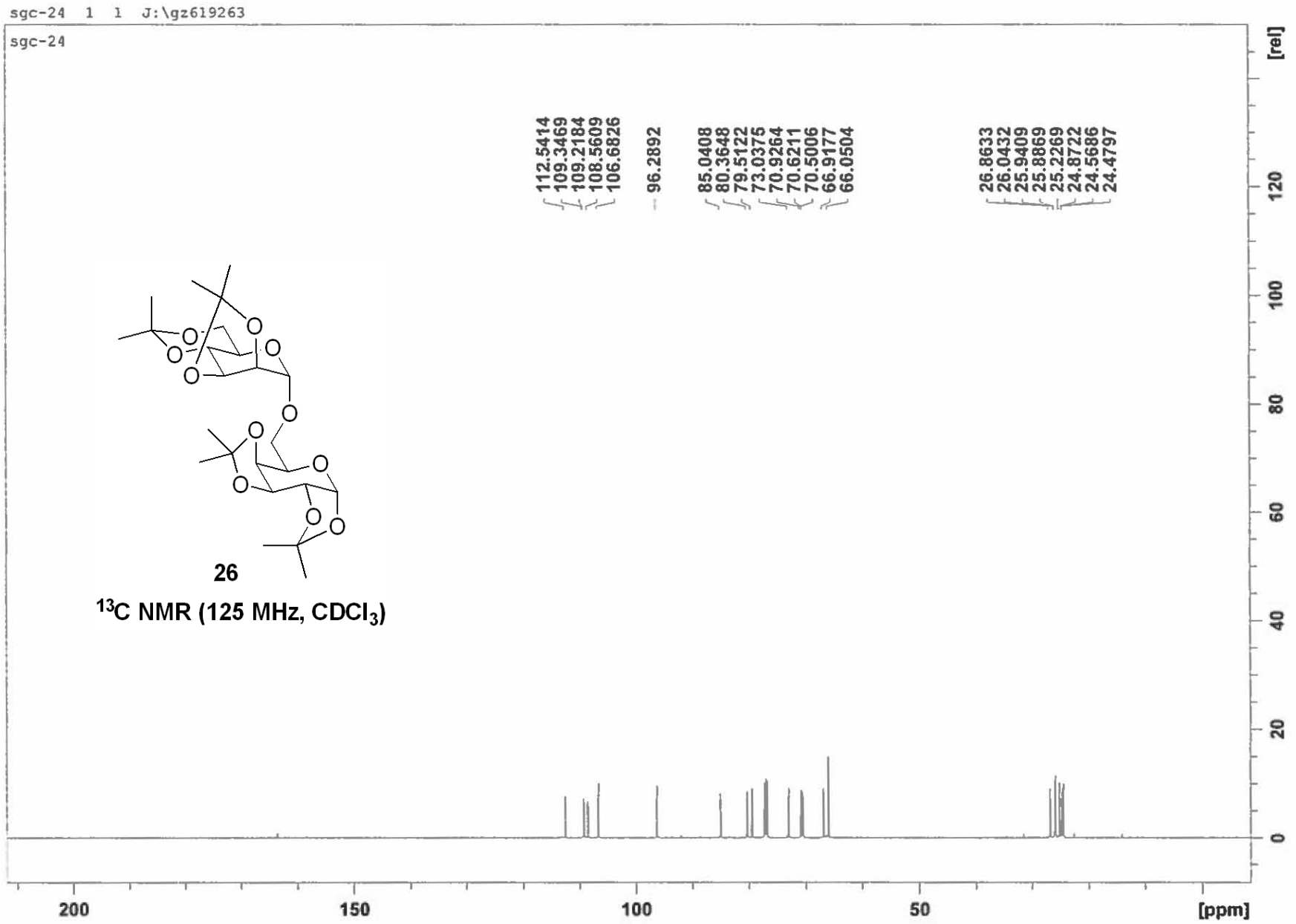
^{1}H NMR (400 MHz, $CDCl_3$)


zgy-SGC-11 2 1 /opt/topspin1.3 gz619263
[rel]
170.9429
155.3531
138.5890
138.4903
138.3665
138.3393
128.2266
128.0370
127.7952
127.5975
127.4920
98.5415
80.1146
79.7433
75.0776
74.5255
73.8829
72.6078
72.3503
67.9812
62.8808
53.7604
52.3579
28.2786
17.9889
17.9539
12.0045
BocHN
O
OMe
O
BnO
BnO
O
OBn
OTIPS
25
13C NMR (100 MHz, CDCl3)
200
150
100
50
0
[ppm]



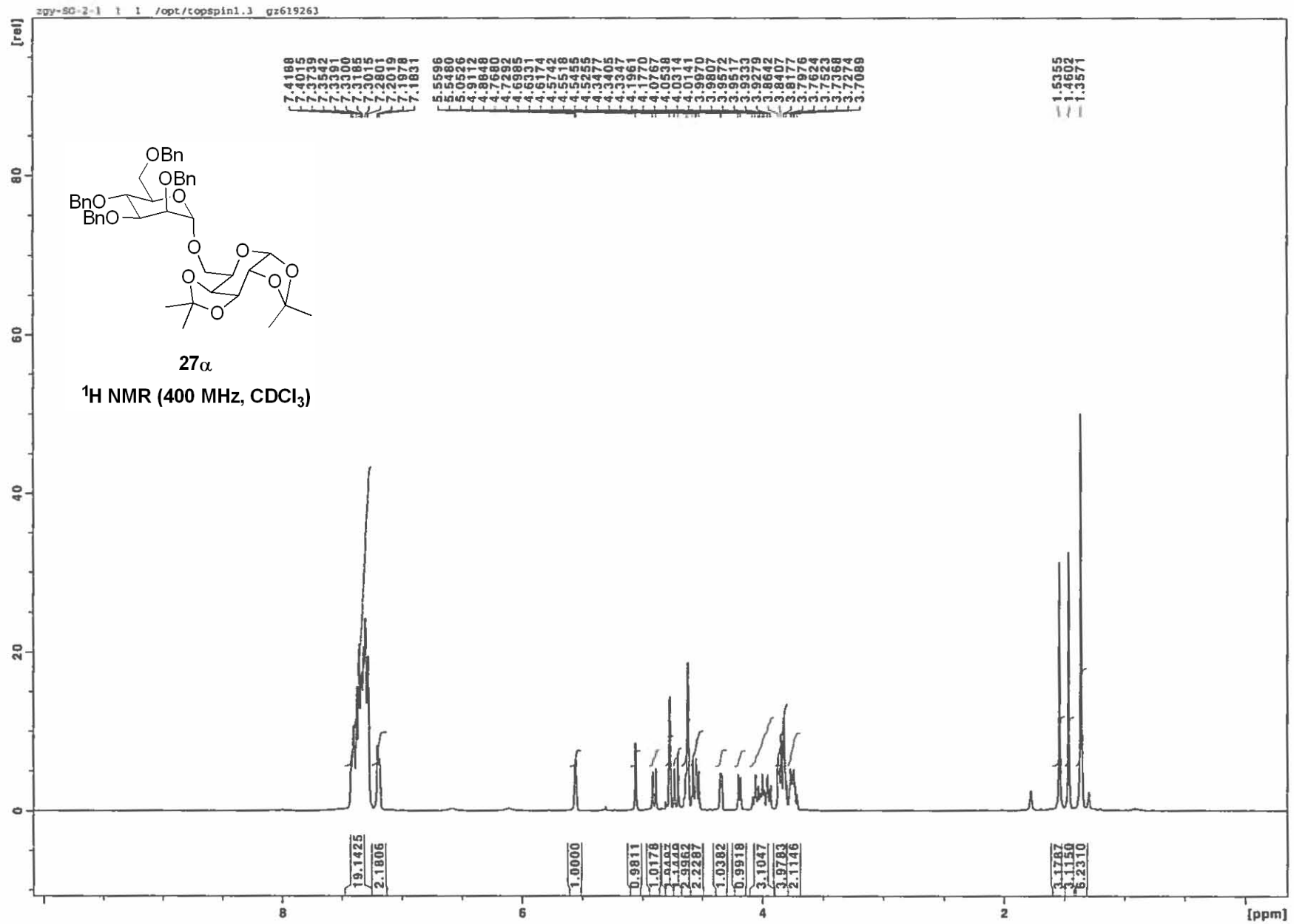
26

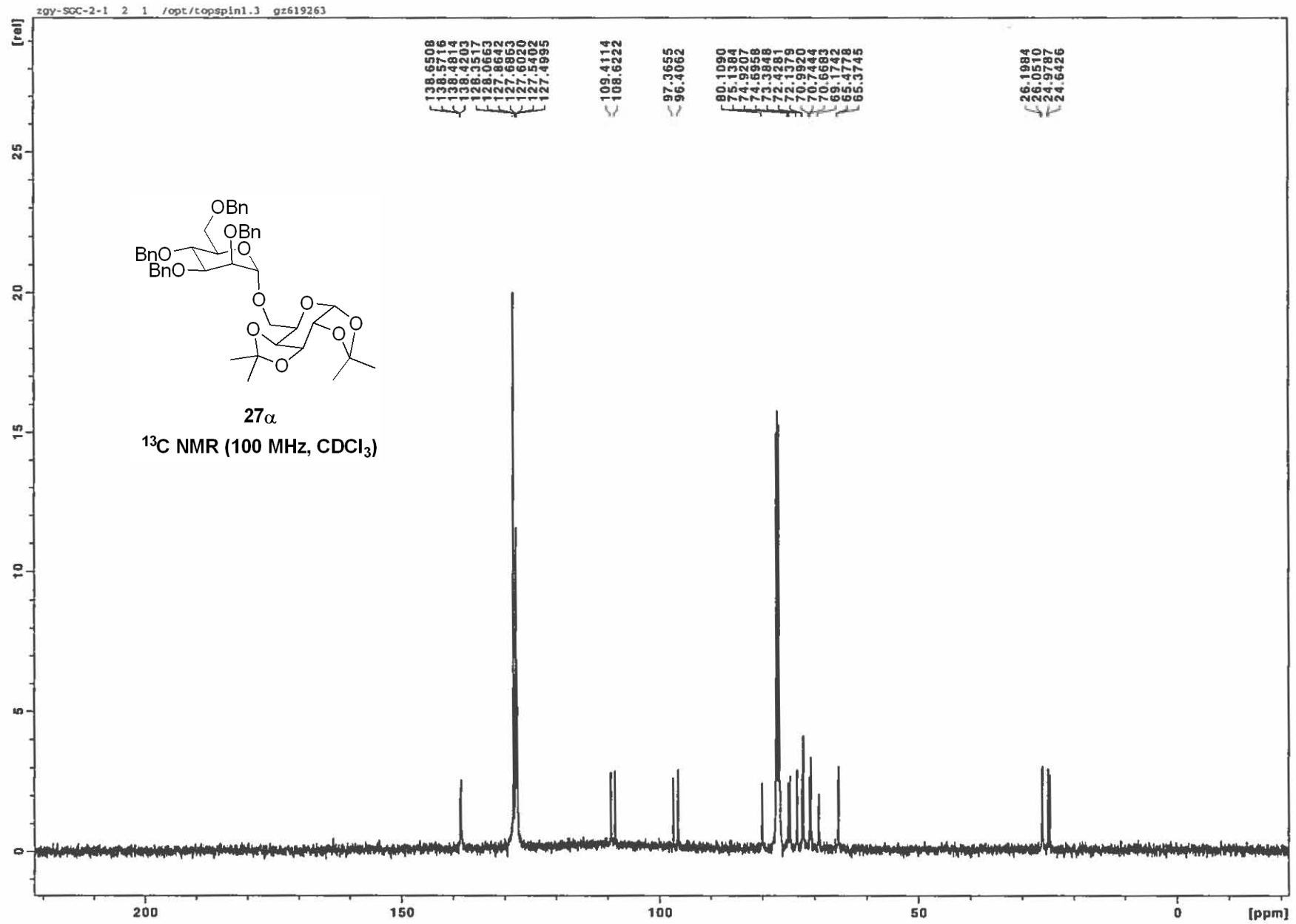
^{1}H NMR (500 MHz, $CDCl_3$)



26

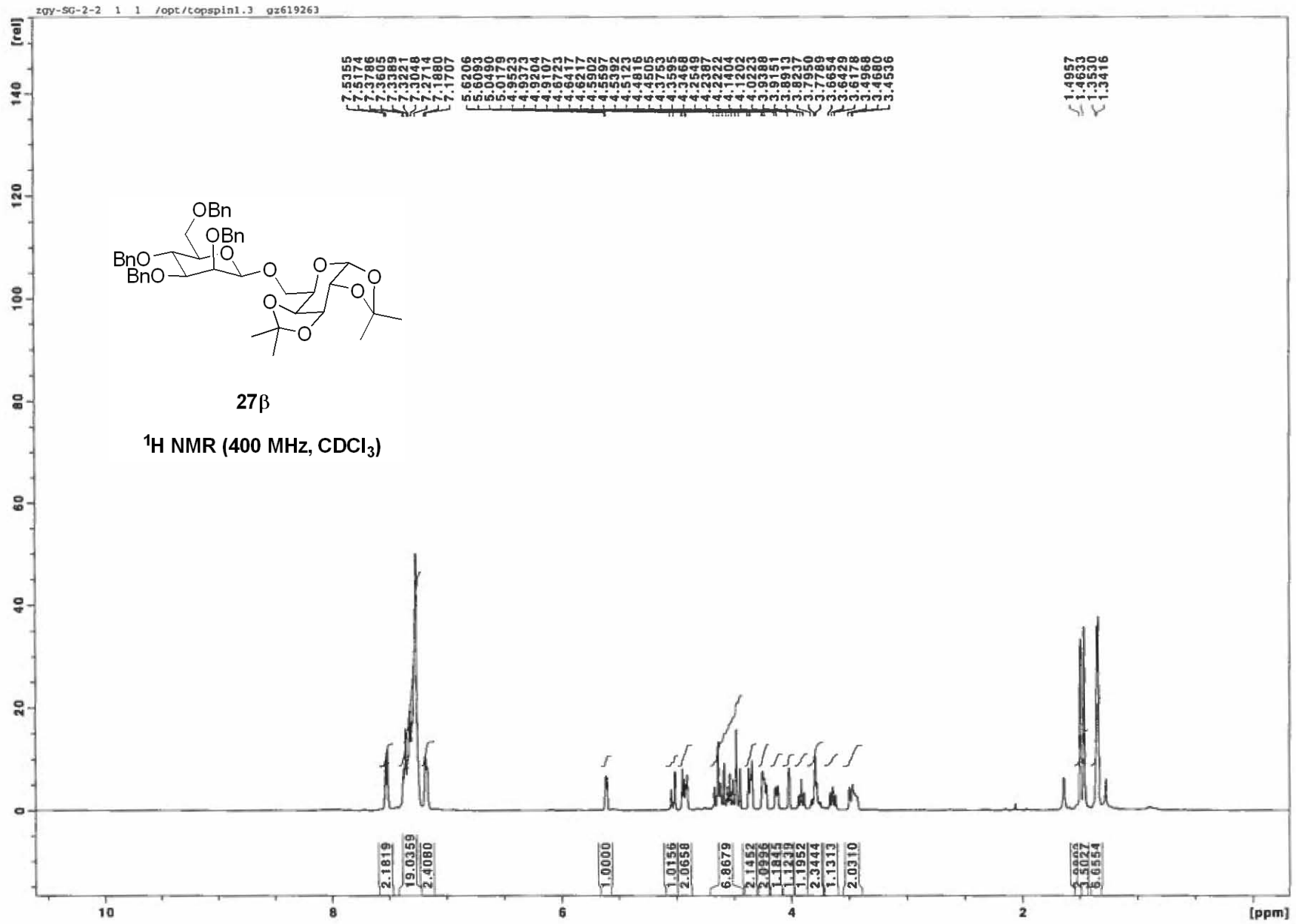
^{13}C NMR (125 MHz, $CDCl_3$)


zgy-SC-2-1 1 1 /opt/topspin1.3 gz619263
[rel]
[ppm]
OBn
OBn
BnO
BnO
O
27α
1H NMR (400 MHz, CDCl3)



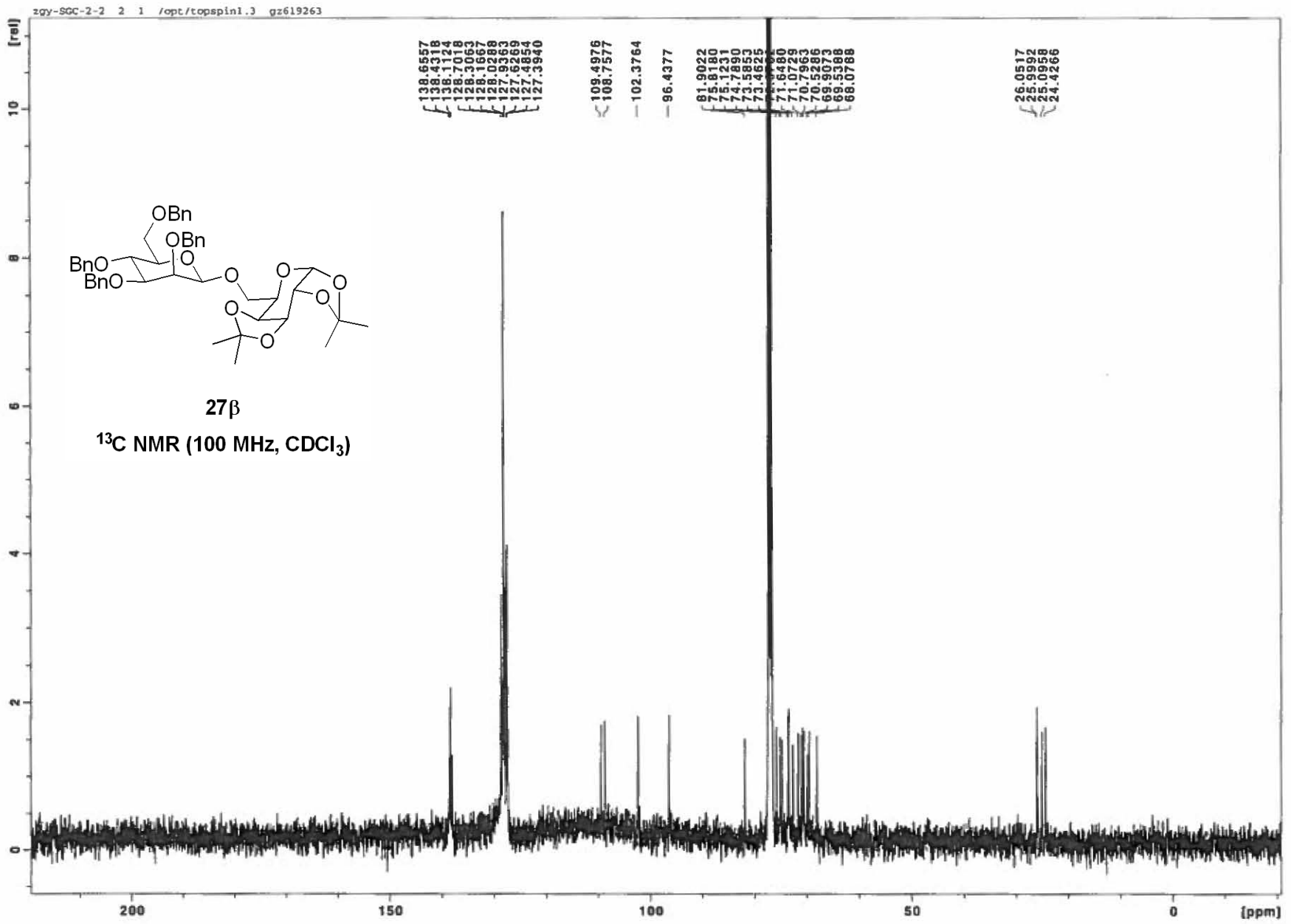
27α

^{13}C NMR (100 MHz, $CDCl_3$)



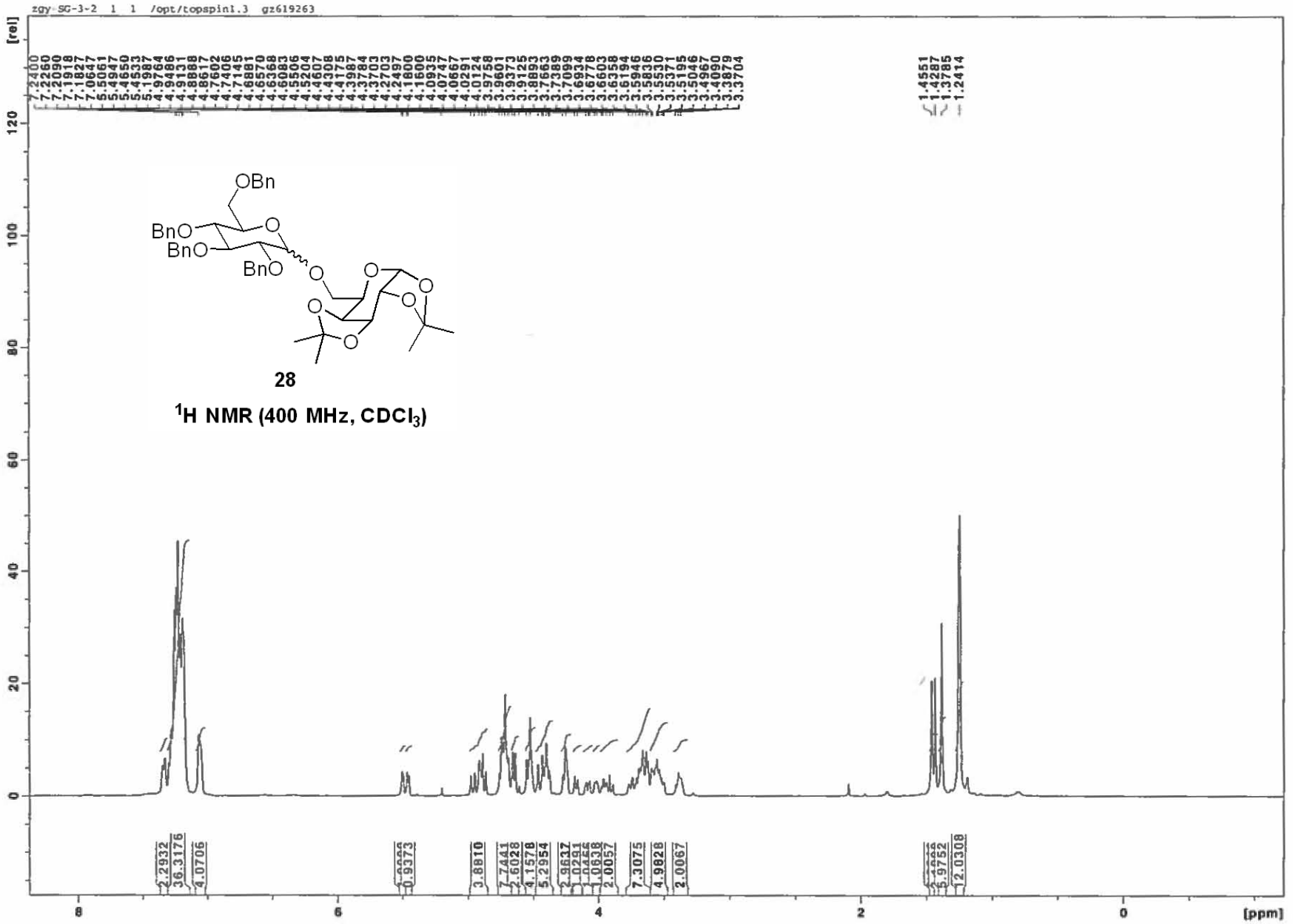
27β

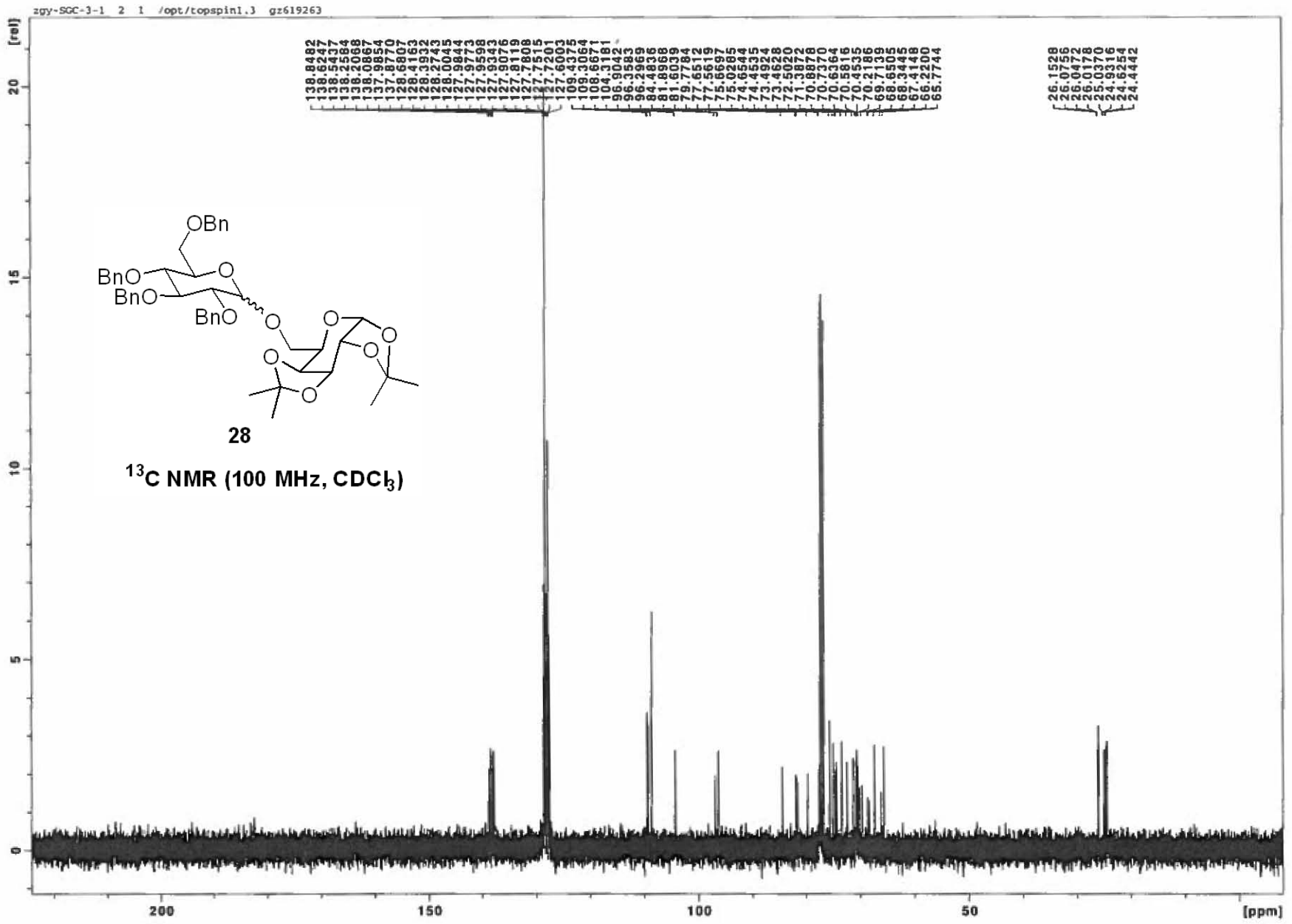
^{1}H NMR (400 MHz, $CDCl_3$)

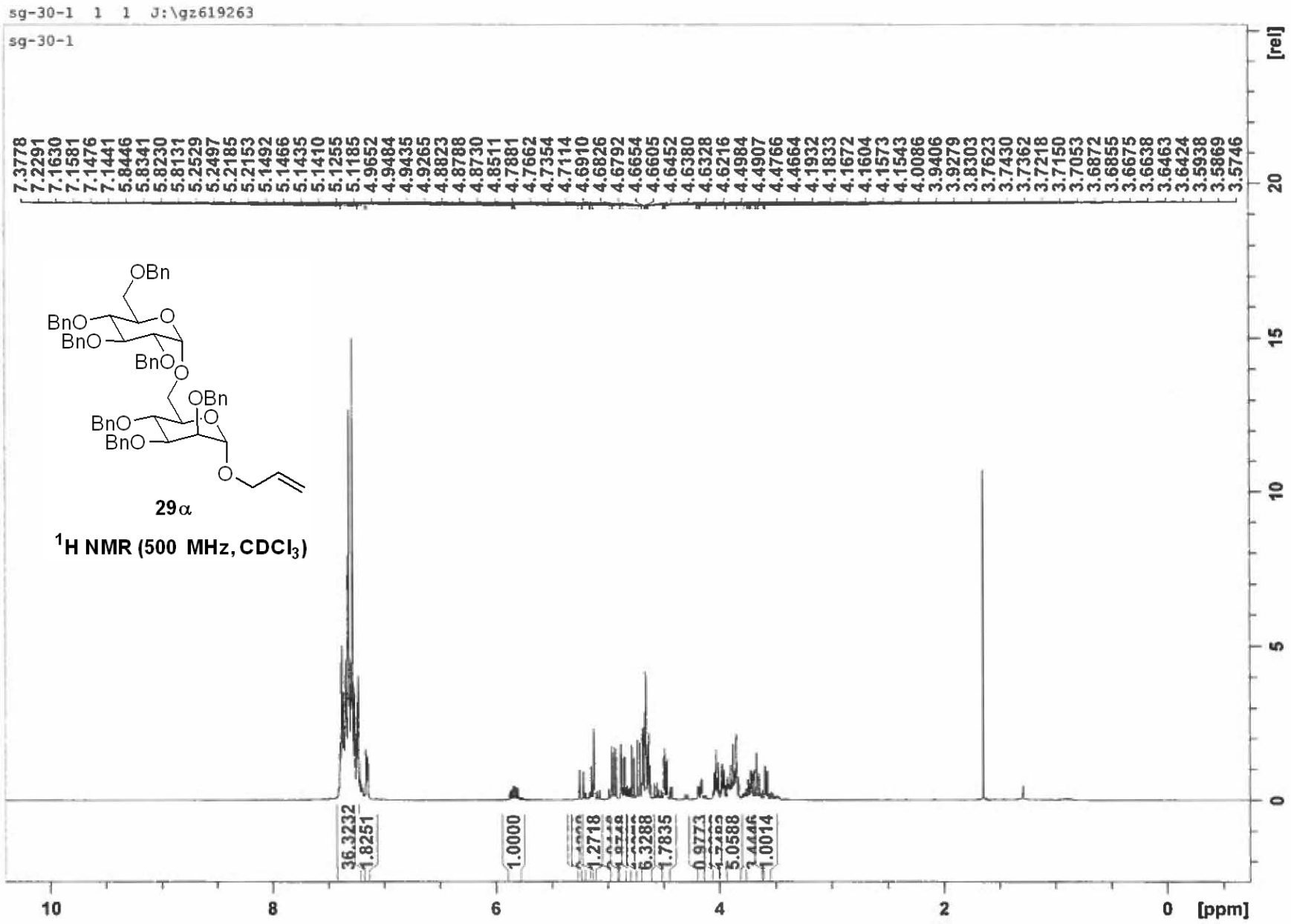


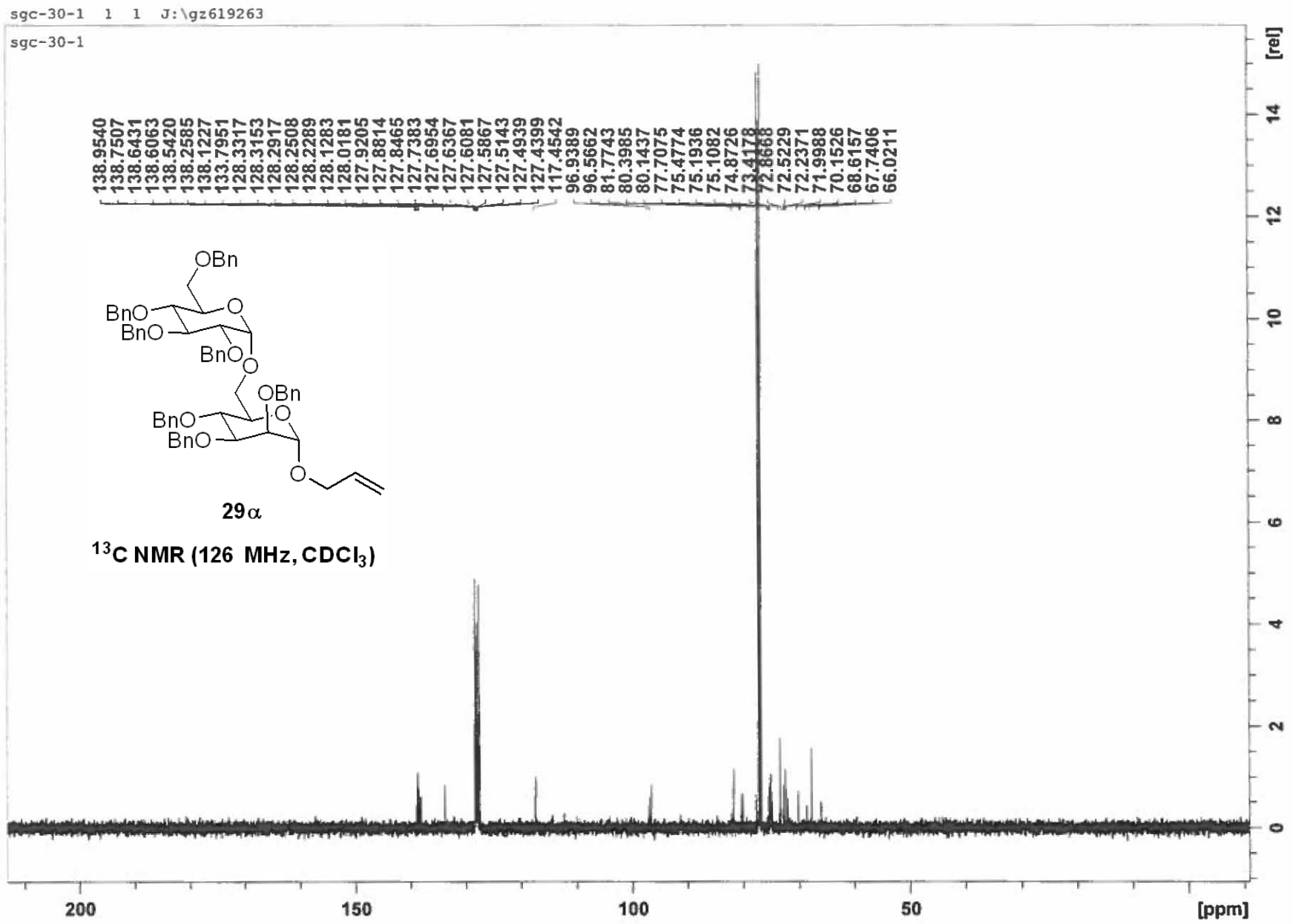
27β

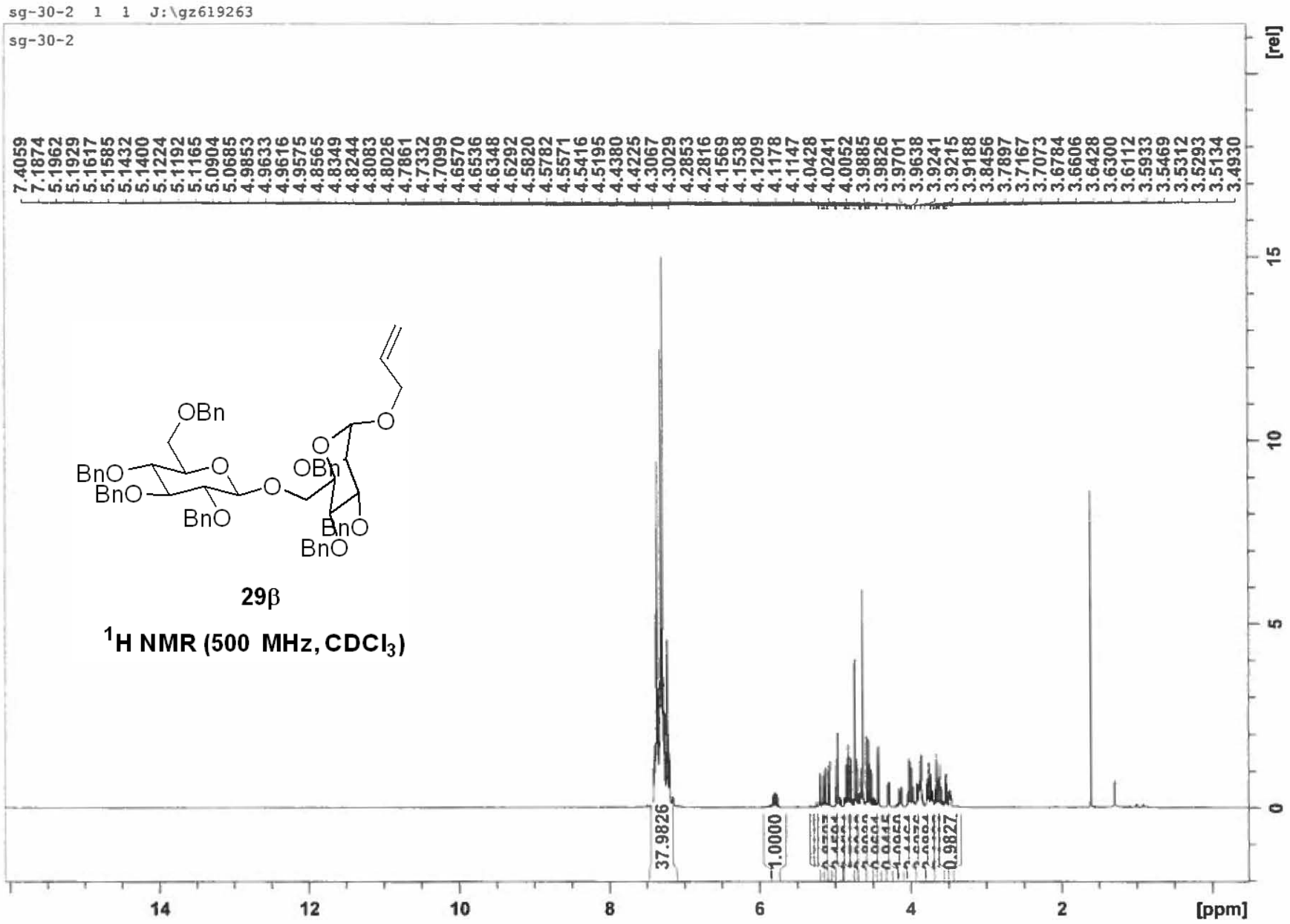
^{13}C NMR (100 MHz, $CDCl_3$)

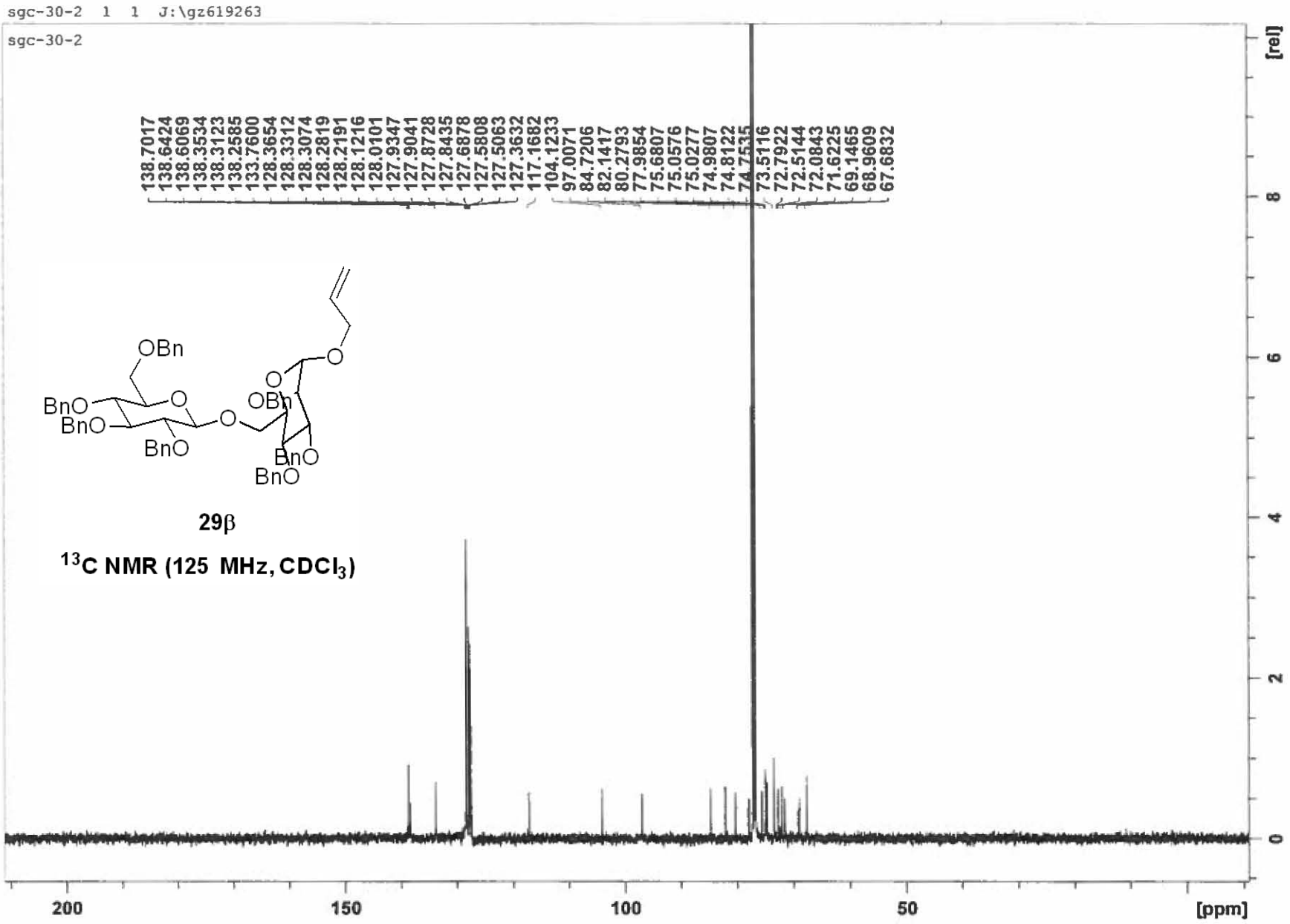

zgy SG-3-2 1 1 /opt/topspin1.3 gz619263
[rel]
OBn
BnO
BnO
BnO
28
^{1}H NMR (400 MHz, CDCl$_3$)
[ppm]


zgy-SGC-3-1 2 1 /opt/topspin1.3 gz619263
[rel]
20
15
10
5
0
200
150
100
50
[ppm]
OBn
BnO
BnO
BnO
O
28
^{13}C NMR (100 MHz, CDCl$_3$)


sg-30-1 1 1 J:\gz619263
sg-30-1
7.3778
7.2291
7.1630
7.1581
7.1476
7.1441
5.8446
5.8341
5.8230
5.8131
5.2529
5.2497
5.2185
5.2153
5.1492
5.1466
5.1435
5.1410
5.1255
5.1185
4.9652
4.9484
4.9435
4.9265
4.8823
4.8788
4.8730
4.8511
4.7881
4.7662
4.7354
4.7114
4.6910
4.6826
4.6792
4.6654
4.6605
4.6452
4.6380
4.6328
4.6216
4.4984
4.4907
4.4766
4.4664
4.1932
4.1833
4.1672
4.1604
4.1573
4.1543
4.0086
3.9406
3.9279
3.8303
3.7623
3.7430
3.7362
3.7218
3.7150
3.7053
3.6872
3.6855
3.6675
3.6638
3.6463
3.6424
3.5938
3.5869
3.5746
OBn
BnO
BnO
BnO
O
O
OBn
BnO
BnO
O
O
29α
1H NMR (500 MHz, CDCl3)
36.3232
1.8251
1.0000
1.2718
6.3288
1.7835
0.9773
5.0588
1.0014
10
8
6
4
2
0
[ppm]
[rel]
20
15
10
5
0


sgc-30-1 1 1 J:\gz619263
sgc-30-1
138.9540
138.7507
138.6431
138.6063
138.5420
138.2585
138.1227
133.7951
128.3317
128.3153
128.2917
128.2508
128.2289
128.1283
128.0181
127.9205
127.8814
127.8465
127.7383
127.6954
127.6367
127.6081
127.5867
127.5143
127.4939
127.4399
117.4542
96.9389
96.5662
81.7743
80.3985
80.1437
77.7075
75.4774
75.1936
75.1082
74.8726
73.4178
72.8668
72.5229
72.2371
71.9988
70.1526
68.6157
67.7406
66.0211
OBn
BnO
BnO
BnO
O
OBn
BnO
BnO
O
O
29α
13C NMR (126 MHz, CDCl3)
[rel]
0
2
4
6
8
10
12
14
200
150
100
50
[ppm]


sg-30-2 1 1 J:\gz619263
sg-30-2
7.4059
7.1874
5.1962
5.1929
5.1617
5.1585
5.1432
5.1400
5.1224
5.1192
5.1165
5.0904
5.0685
4.9853
4.9633
4.9616
4.9575
4.8565
4.8349
4.8244
4.8083
4.8026
4.7861
4.7332
4.7099
4.6570
4.6536
4.6348
4.6292
4.5820
4.5782
4.5571
4.5416
4.5195
4.4380
4.4225
4.3067
4.3029
4.2853
4.2816
4.1569
4.1538
4.1209
4.1178
4.1147
4.0428
4.0241
4.0052
3.9885
3.9826
3.9701
3.9638
3.9241
3.9215
3.9188
3.8456
3.7897
3.7167
3.7073
3.6784
3.6606
3.6428
3.6300
3.6112
3.5933
3.5469
3.5312
3.5293
3.5134
3.4930
OBn
BnO
BnO
BnO
O
O
OBn
BnO
BnO
O
O
29β
1H NMR (500 MHz, CDCl3)
37.9826
1.0000
0.9827
[rel]
15
10
5
0
14
12
10
8
6
4
2
[ppm]


sgc-30-2 1 1 J:\gz619263
sgc-30-2
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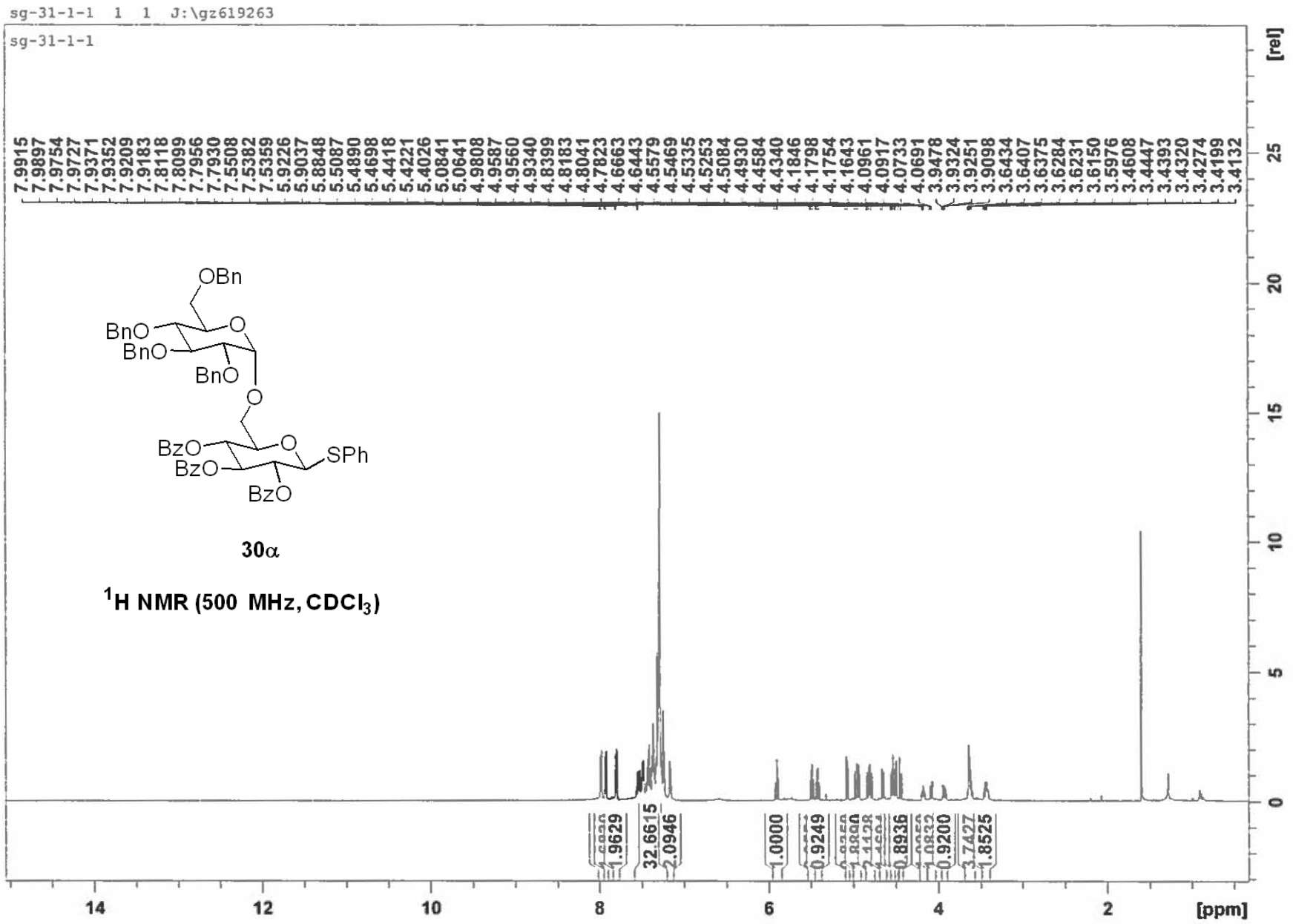
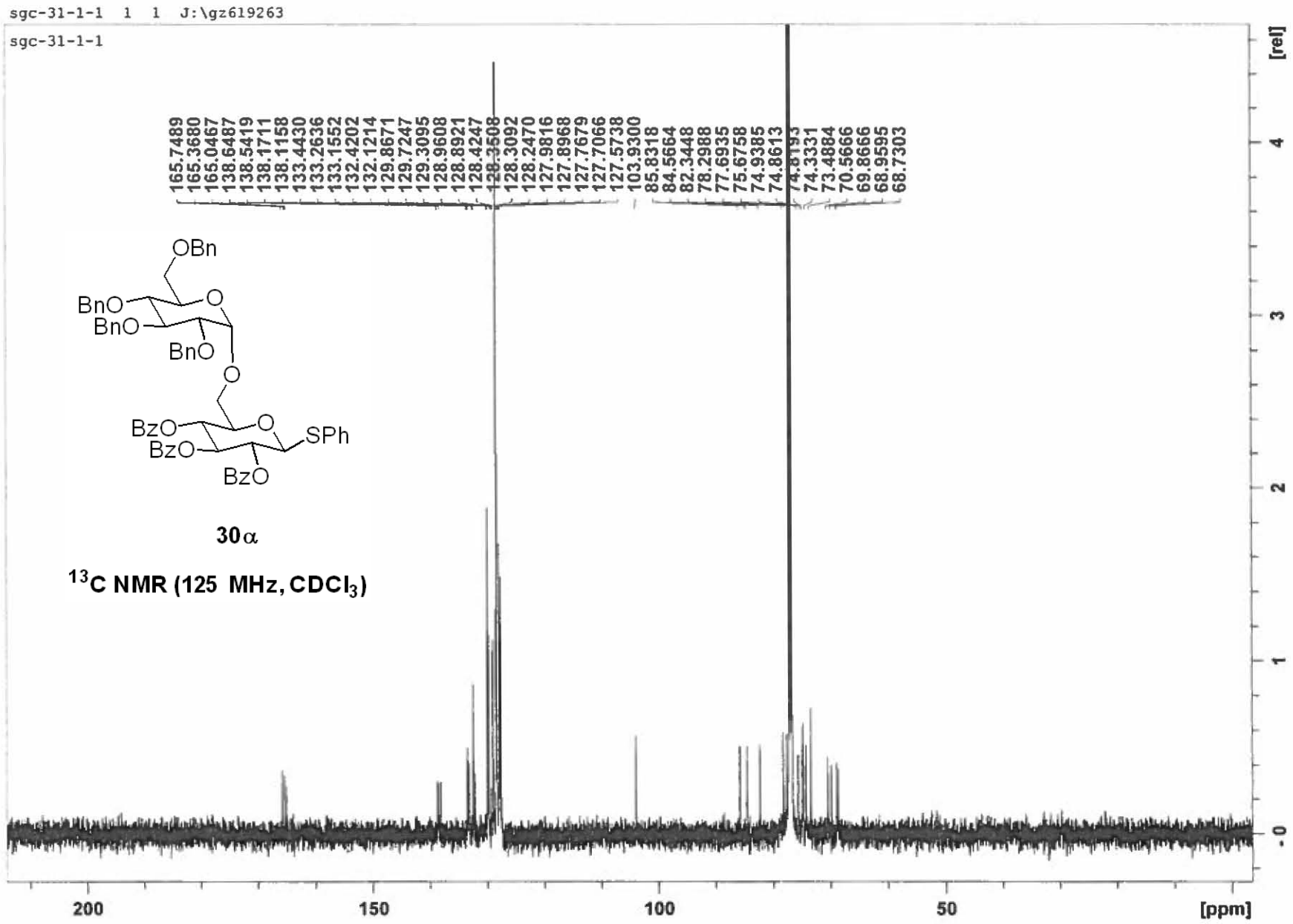
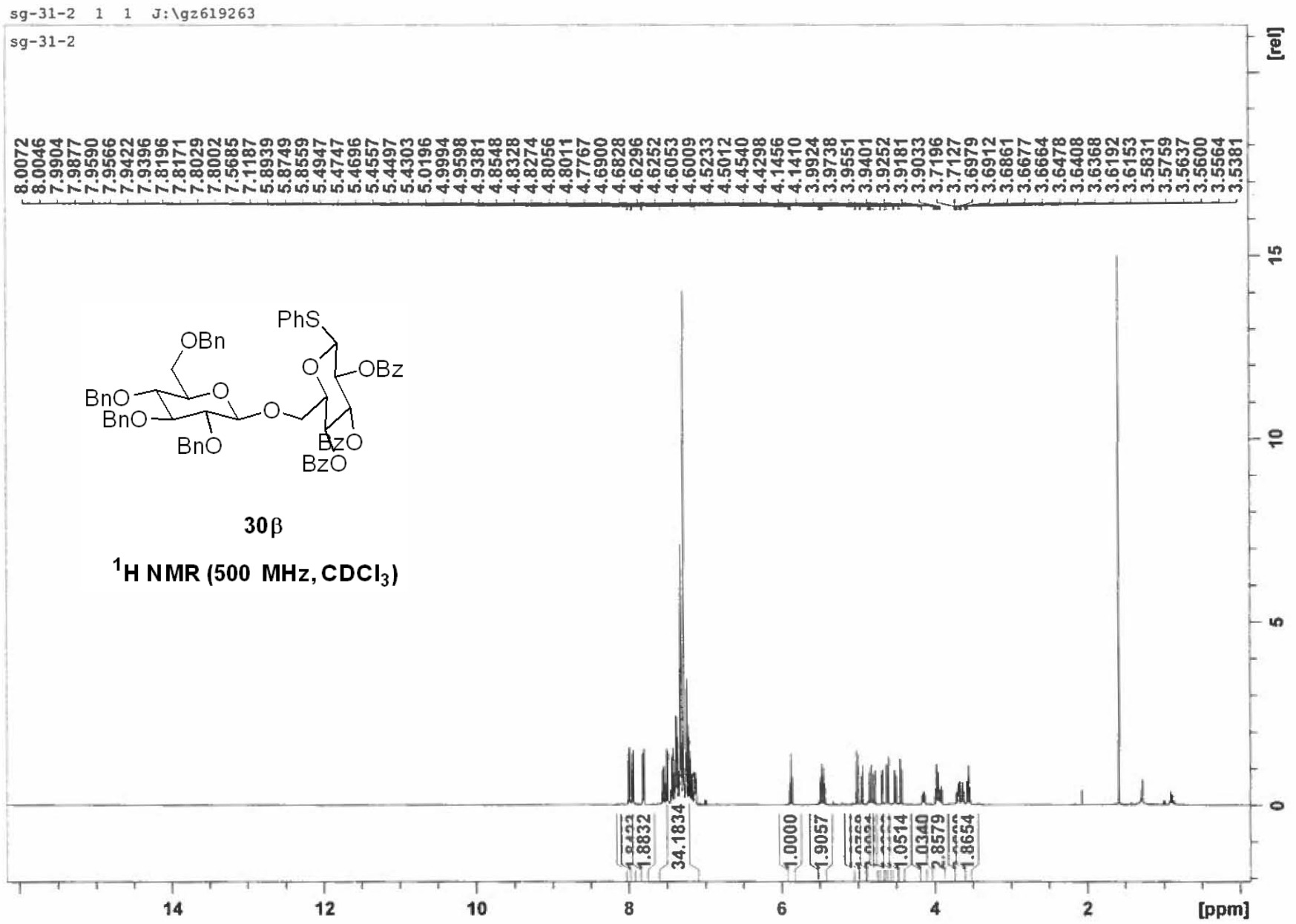
138.6424
138.6069
138.3534
138.3123
138.2585
133.7600
128.3654
128.3312
128.3074
128.2819
128.2191
128.1216
128.0101
127.9347
127.9041
127.8728
127.8435
127.6878
127.5808
127.5063
127.3632
117.1682
104.1233
97.0071
84.7206
82.1417
80.2793
77.9854
75.6807
75.0576
75.0277
74.9807
74.8122
74.7535
73.5116
72.7922
72.5144
72.0843
71.6225
69.1465
68.9609
67.6832
OBn
BnO
BnO
BnO
O
O
OBn
O
BnO
BnO
29β
13C NMR (125 MHz, CDCl3)
[rel]
0
2
4
6
8
200
150
100
50
[ppm]



30α

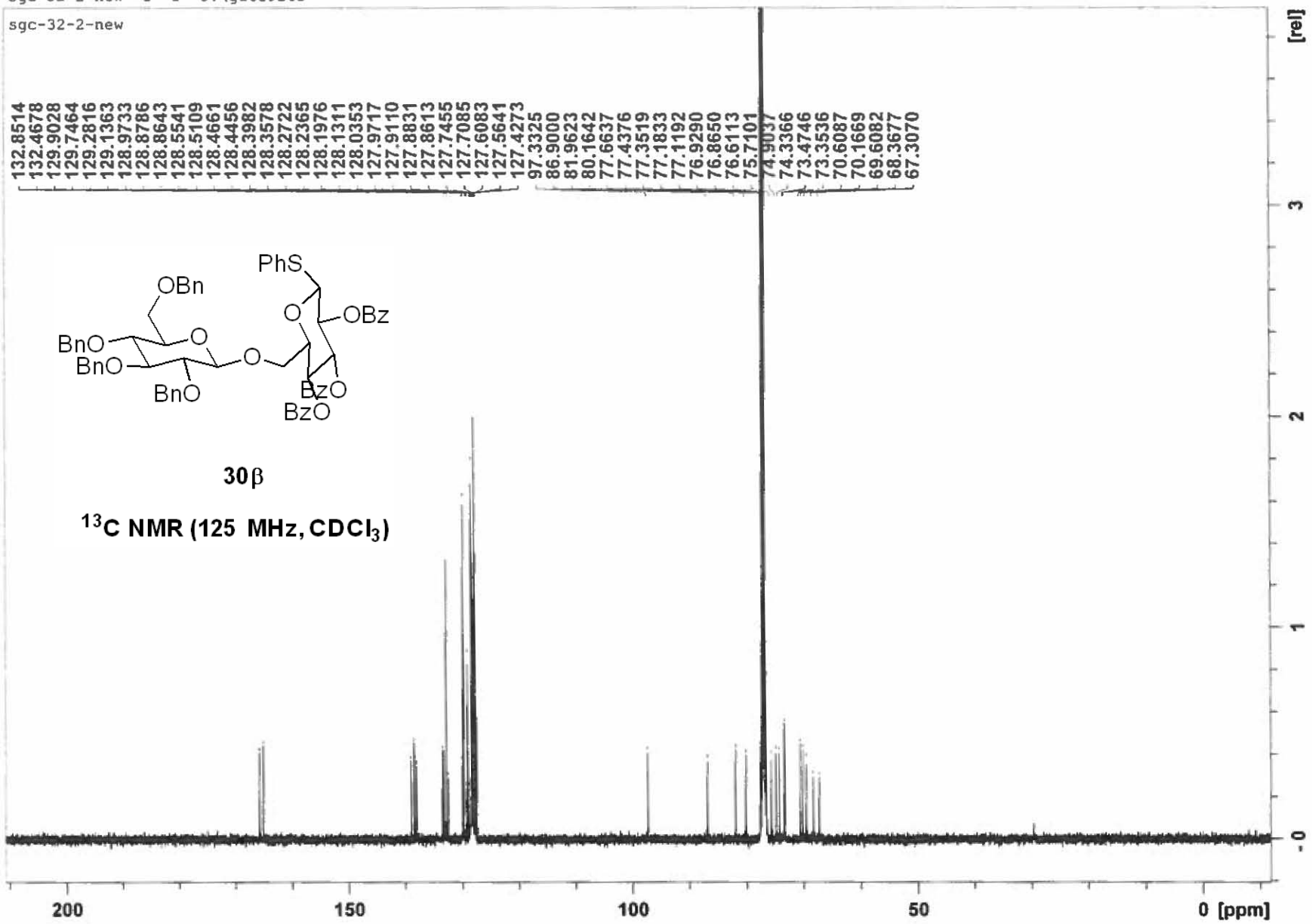
^{1}H NMR (500 MHz, $CDCl_3$)

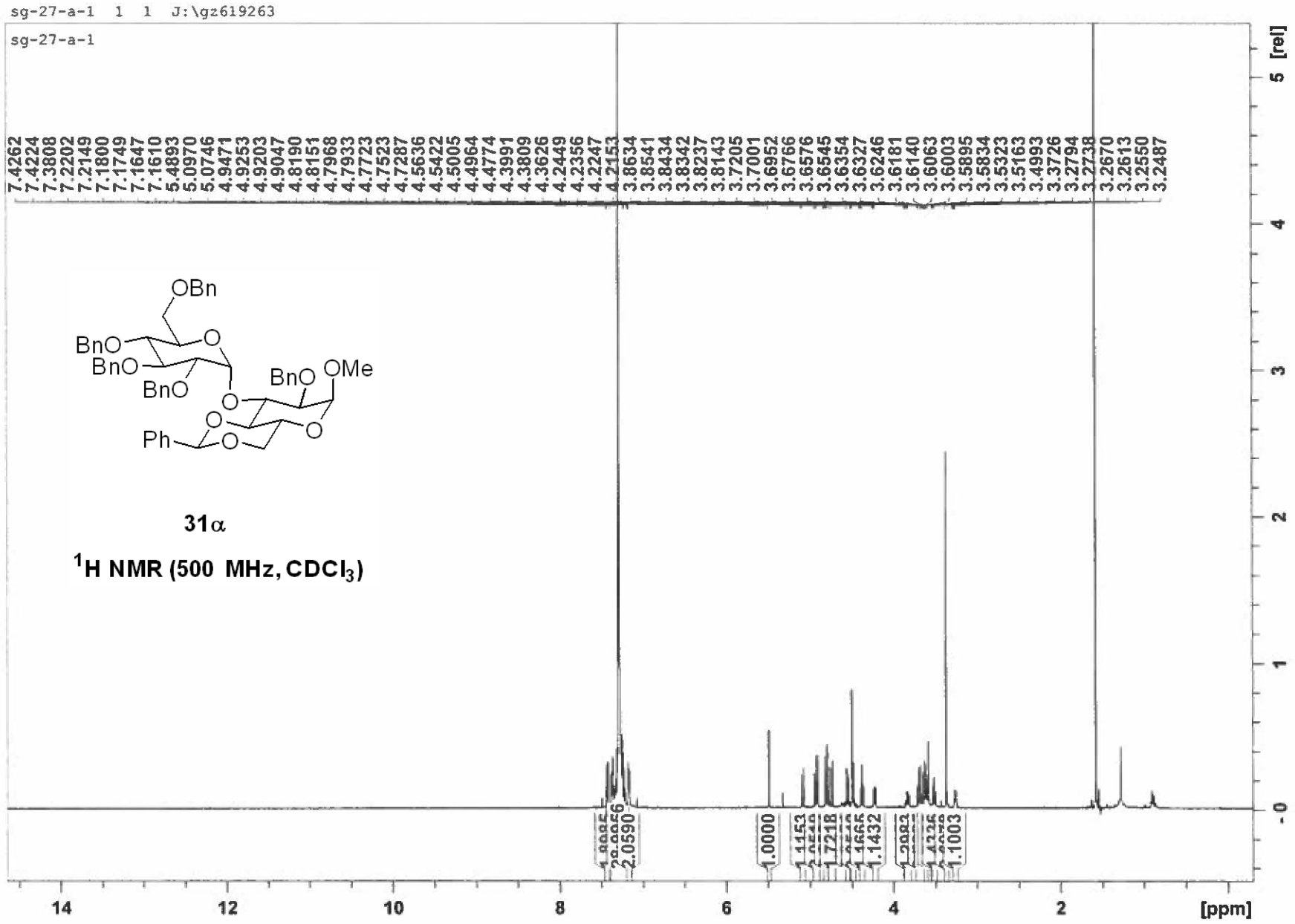

sgc-31-1-1 1 1 J:\gz619263
sgc-31-1-1
165.7489
165.3680
165.0467
138.6487
138.5419
138.1711
138.1158
133.4430
133.2636
133.1552
132.4202
132.1214
129.8671
129.7247
129.3095
128.9608
128.8921
128.4247
128.3508
128.3092
128.2470
127.9816
127.8968
127.7679
127.7066
127.5738
103.9300
85.8318
84.5664
82.3448
78.2988
77.6935
75.6758
74.9385
74.8613
74.8193
74.3331
73.4884
70.5666
69.8666
68.9595
68.7303
OBn
BnO
BnO
BnO
O
O
BzO
BzO
BzO
O
SPh
30α
13C NMR (125 MHz, CDCl3)
[rel]
4
3
2
1
0
200
150
100
50
[ppm]


sg-31-2 1 1 J:\gz619263
sg-31-2
8.0072
8.0046
7.9904
7.9877
7.9590
7.9566
7.9422
7.9396
7.8196
7.8171
7.8029
7.8002
7.5685
7.1187
5.8939
5.8749
5.8559
5.4947
5.4747
5.4696
5.4557
5.4497
5.4303
5.0196
4.9994
4.9598
4.9381
4.8548
4.8328
4.8274
4.8056
4.8011
4.7767
4.6900
4.6828
4.6296
4.6252
4.6053
4.6009
4.5233
4.5012
4.4540
4.4298
4.1456
4.1410
3.9924
3.9738
3.9551
3.9401
3.9252
3.9181
3.9033
3.7196
3.7127
3.6979
3.6912
3.6861
3.6677
3.6664
3.6478
3.6408
3.6368
3.6192
3.6153
3.5831
3.5759
3.5637
3.5600
3.5564
3.5381
[rel]
15
10
5
0
PhS
OBn
O
OBz
BnO
O
BnO
O
BnO
BzO
BzO
30β
1H NMR (500 MHz, CDCl3)
1.8832
34.1834
1.0000
1.9057
1.0514
1.0340
2.8579
1.8654
14
12
10
8
6
4
2
[ppm]

sgc-31-2

sgc-32-2-new 1 1 J:\gz619263




sg-27-a-1 1 1 J:\gz619263
sg-27-a-1
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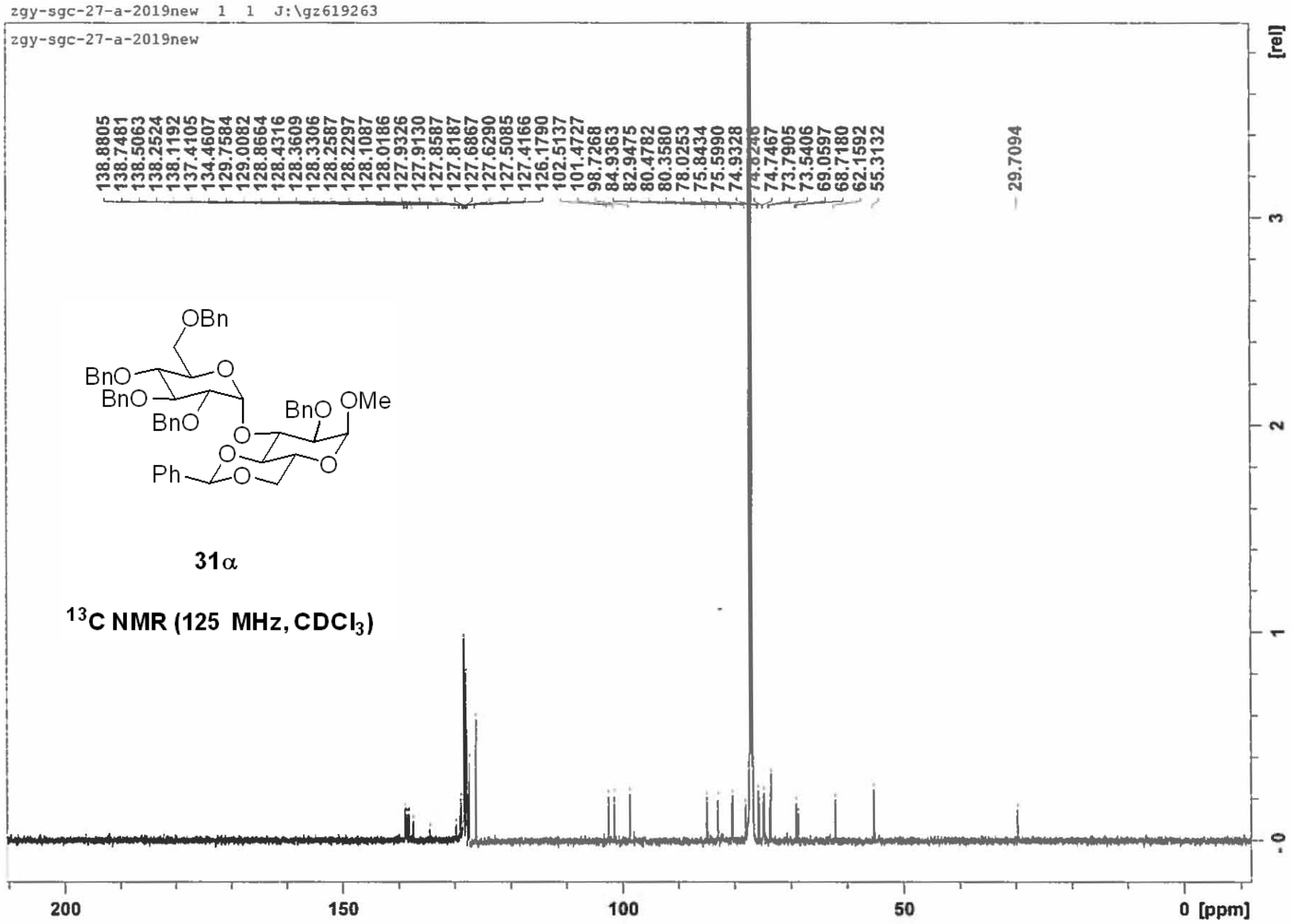
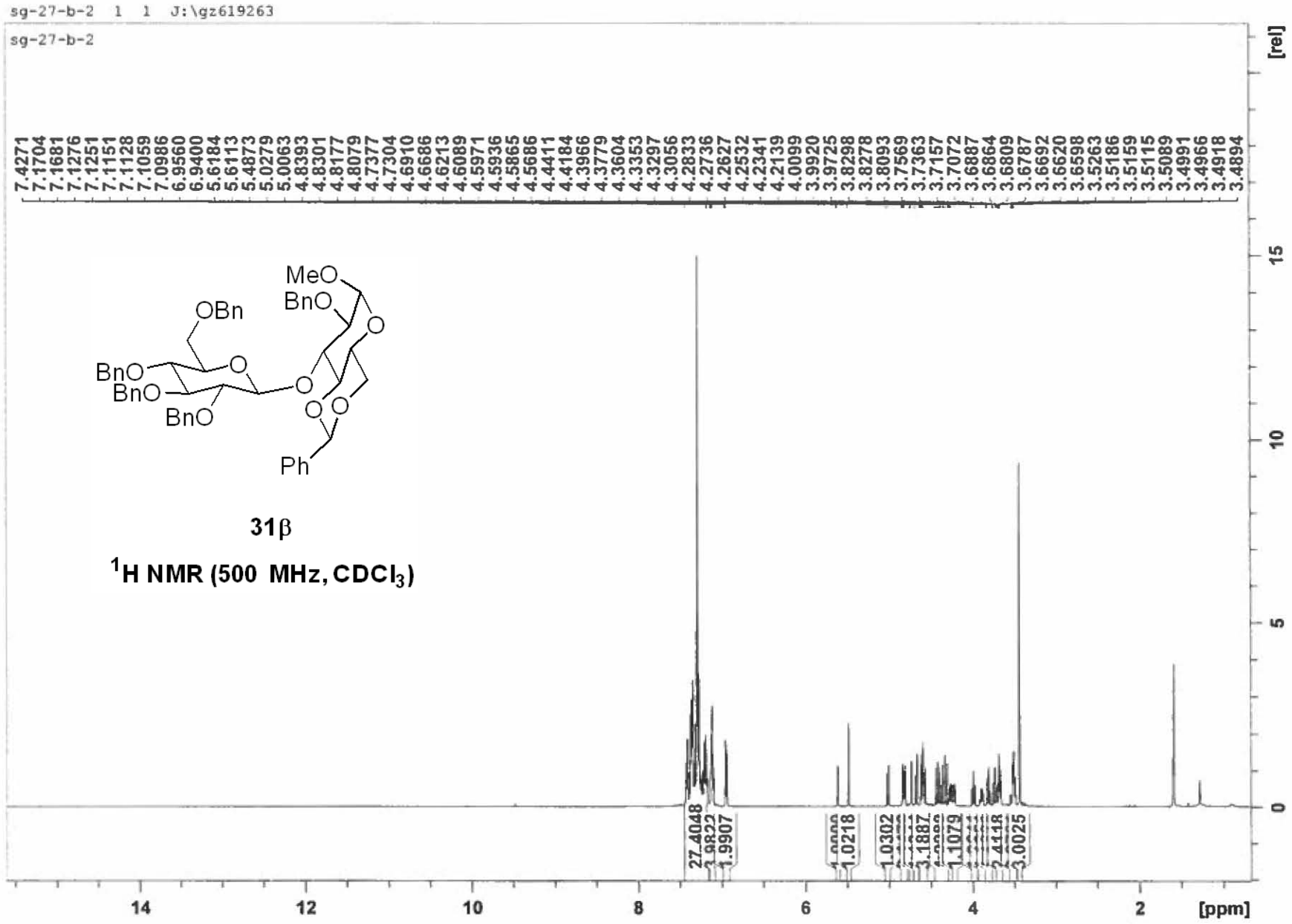
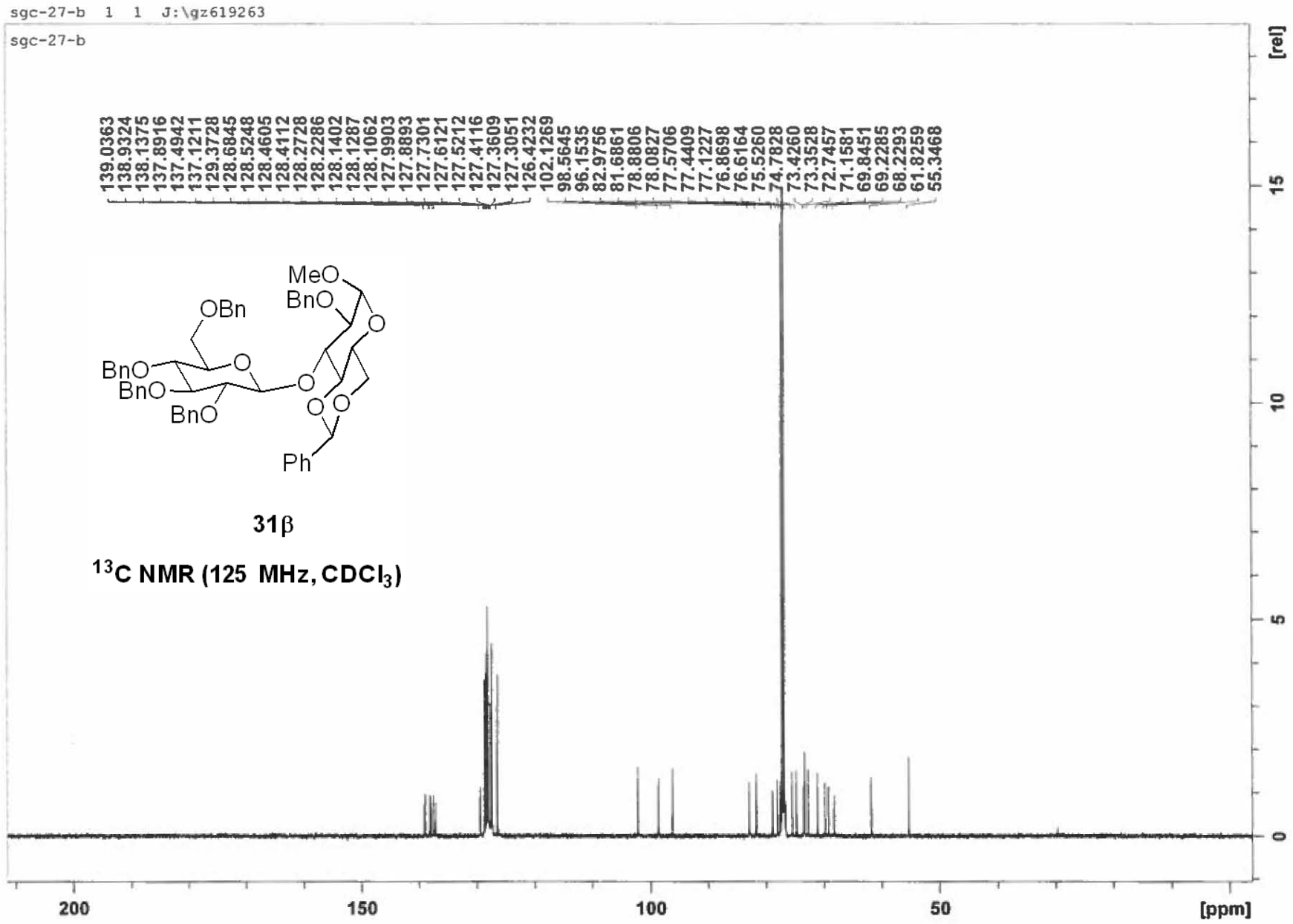
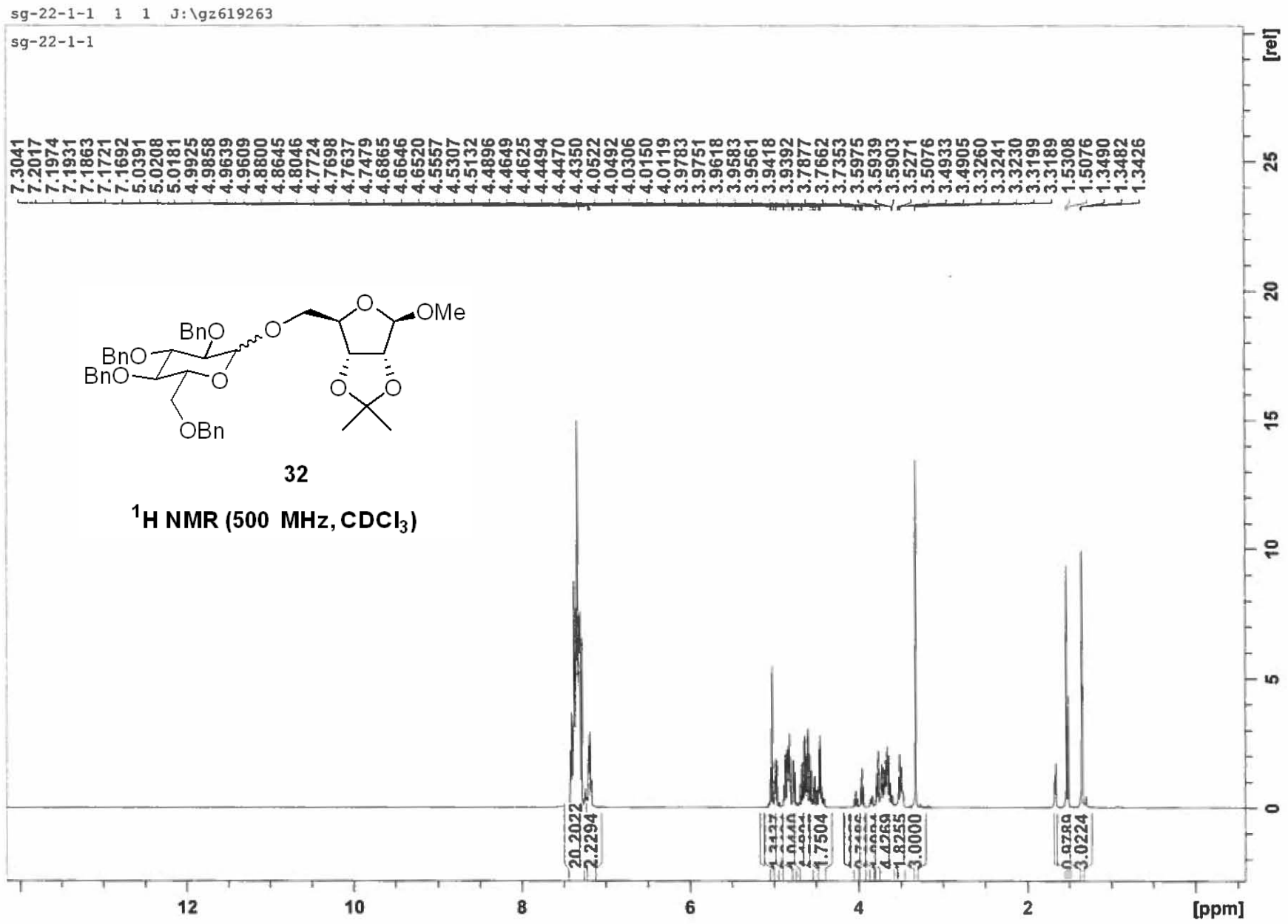
7.4224
7.3808
7.2202
7.2149
7.1800
7.1749
7.1647
7.1610
5.4893
5.0970
5.0746
4.9471
4.9253
4.9203
4.9047
4.8190
4.8151
4.7968
4.7933
4.7723
4.7523
4.7287
4.5636
4.5422
4.5005
4.4964
4.4774
4.3991
4.3809
4.3626
4.2449
4.2356
4.2247
4.2153
3.8634
3.8541
3.8434
3.8342
3.8237
3.8143
3.7205
3.7001
3.6952
3.6766
3.6576
3.6545
3.6354
3.6327
3.6246
3.6181
3.6140
3.6063
3.6003
3.5895
3.5834
3.5323
3.5163
3.4993
3.3726
3.2794
3.2738
3.2670
3.2613
3.2550
3.2487
OBn
BnO
BnO
BnO
O
BnO
OMe
O
O
O
Ph
O
31α
1H NMR (500 MHz, CDCl3)
2.0590
1.0000
1.1153
1.7218
1.1432
1.1003
14
12
10
8
6
4
2
[ppm]
5 [rel]
4
3
2
1
-0


zgy-sgc-27-a-2019new 1 1 J:\gz619263
zgy-sgc-27-a-2019new
138.8805
138.7481
138.5063
138.2524
138.1192
137.4105
134.4607
129.7584
129.0082
128.8664
128.4316
128.3609
128.3306
128.2587
128.2297
128.1087
128.0186
127.9326
127.9130
127.8587
127.8187
127.6867
127.6290
127.5085
127.4166
126.1790
102.5137
101.4727
98.7268
84.9363
82.9475
80.4782
80.3580
78.0253
75.8434
75.5990
74.9328
74.8248
74.7467
73.7905
73.5406
69.0597
68.7180
62.1592
55.3132
29.7094
OBn
BnO
BnO
BnO
O
BnO
OMe
O
O
O
Ph
O
31α
13C NMR (125 MHz, CDCl3)
[rel]
3
2
1
0
200
150
100
50
0 [ppm]

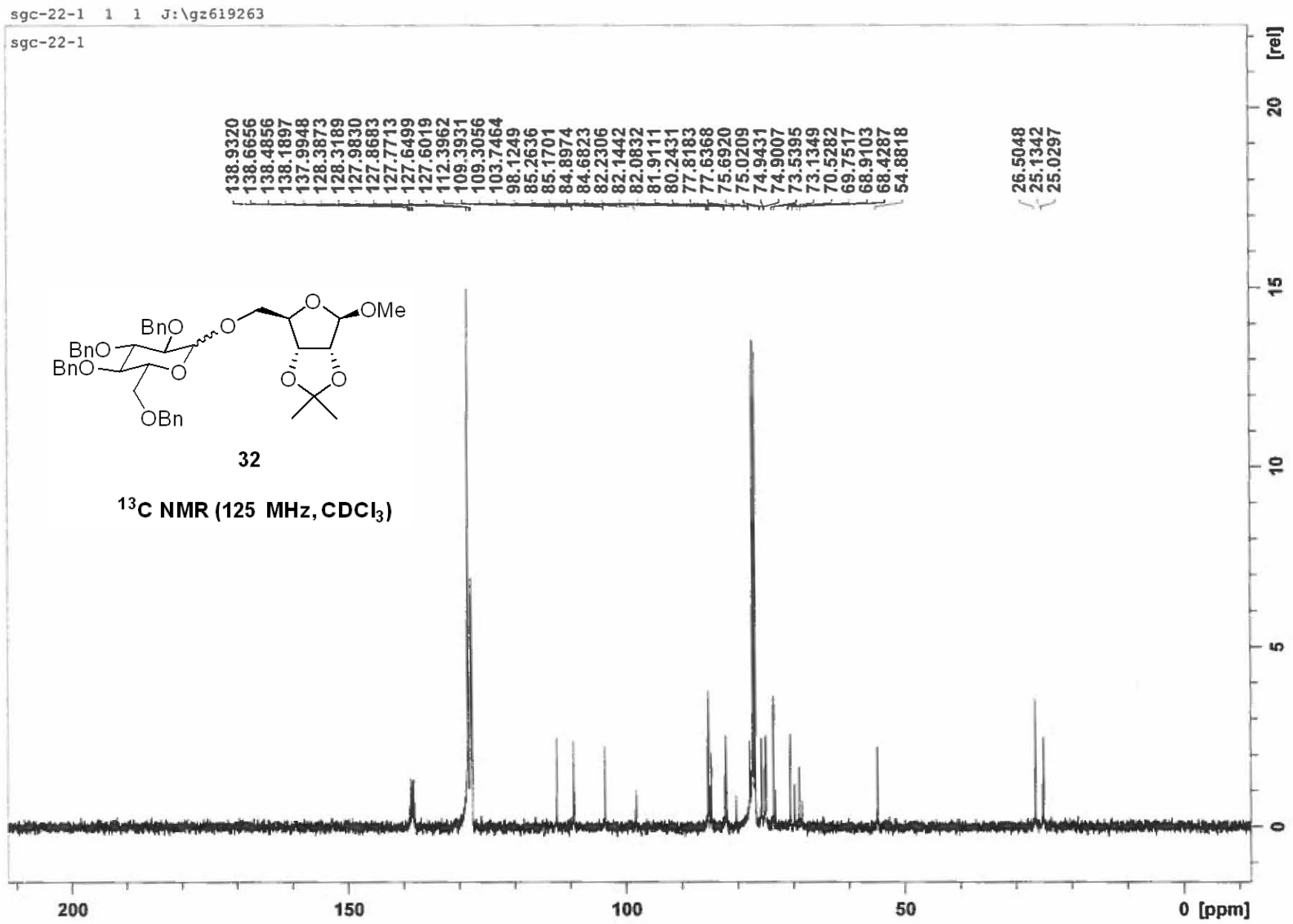

sg-27-b-2 1 1 J:\gz619263
sg-27-b-2
[rel]
MeO
BnO
OBn
BnO
BnO
BnO
O
O
O
O
O
Ph
31β
1H NMR (500 MHz, CDCl3)
[ppm]


sgc-27-b 1 1 J:\gz619263
sgc-27-b
139.0363
138.9324
138.1375
137.8916
137.4942
137.1211
129.3728
128.6845
128.5248
128.4605
128.4112
128.2728
128.2286
128.1402
128.1287
128.1062
127.9903
127.8893
127.7301
127.6121
127.5212
127.4116
127.3609
127.3051
126.4232
102.1269
98.5645
96.1535
82.9756
81.6861
78.8806
78.0827
77.5706
77.4409
77.1227
76.8698
76.6164
75.5260
74.7828
73.4260
73.3528
72.7457
71.1581
69.8451
69.2285
68.2293
61.8259
55.3468
MeO
BnO
OBn
BnO
BnO
BnO
Ph
31β
13C NMR (125 MHz, CDCl3)
[rel]
15
10
5
0
200
150
100
50
[ppm]



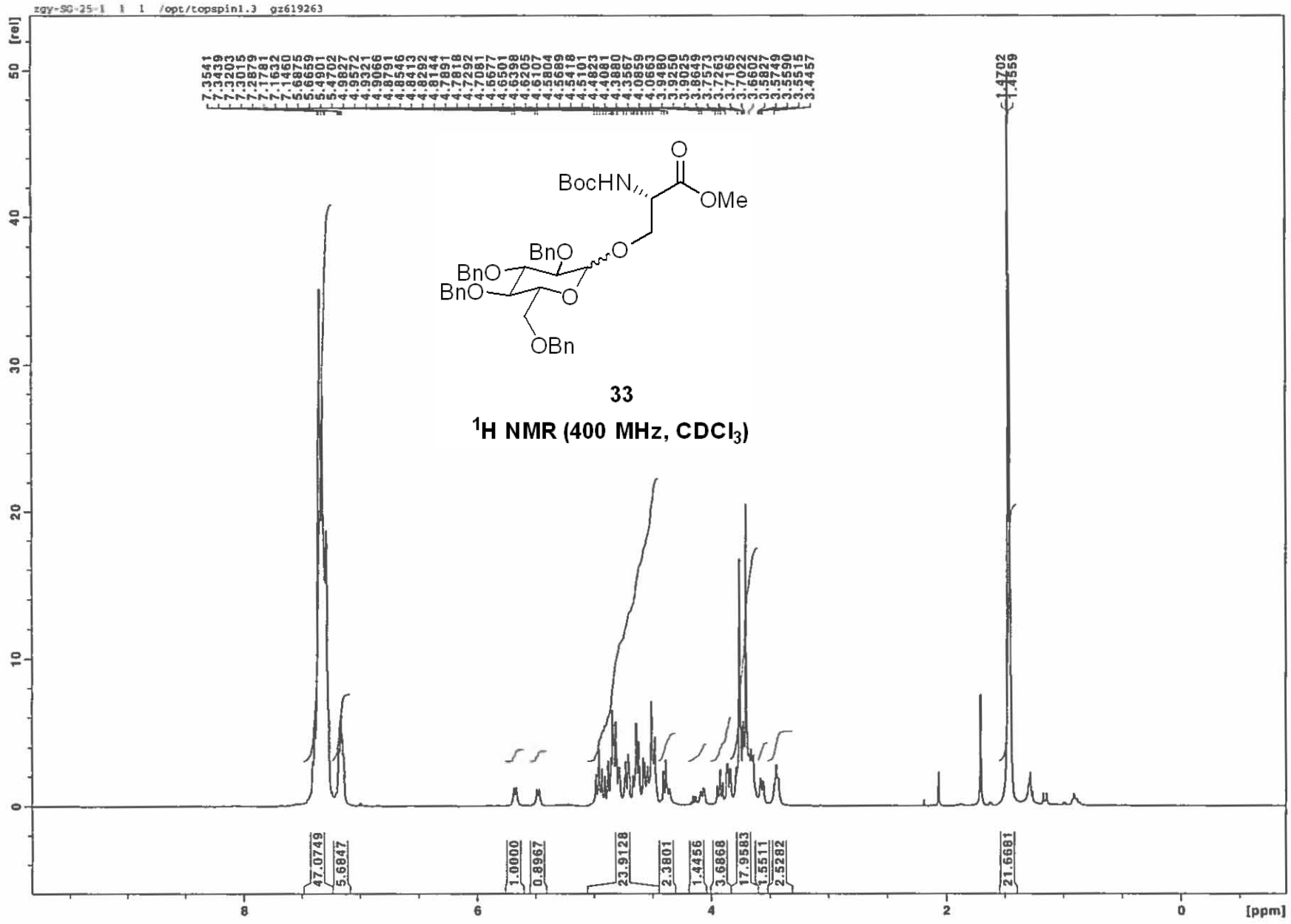
32

^{1}H NMR (500 MHz, $CDCl_3$)



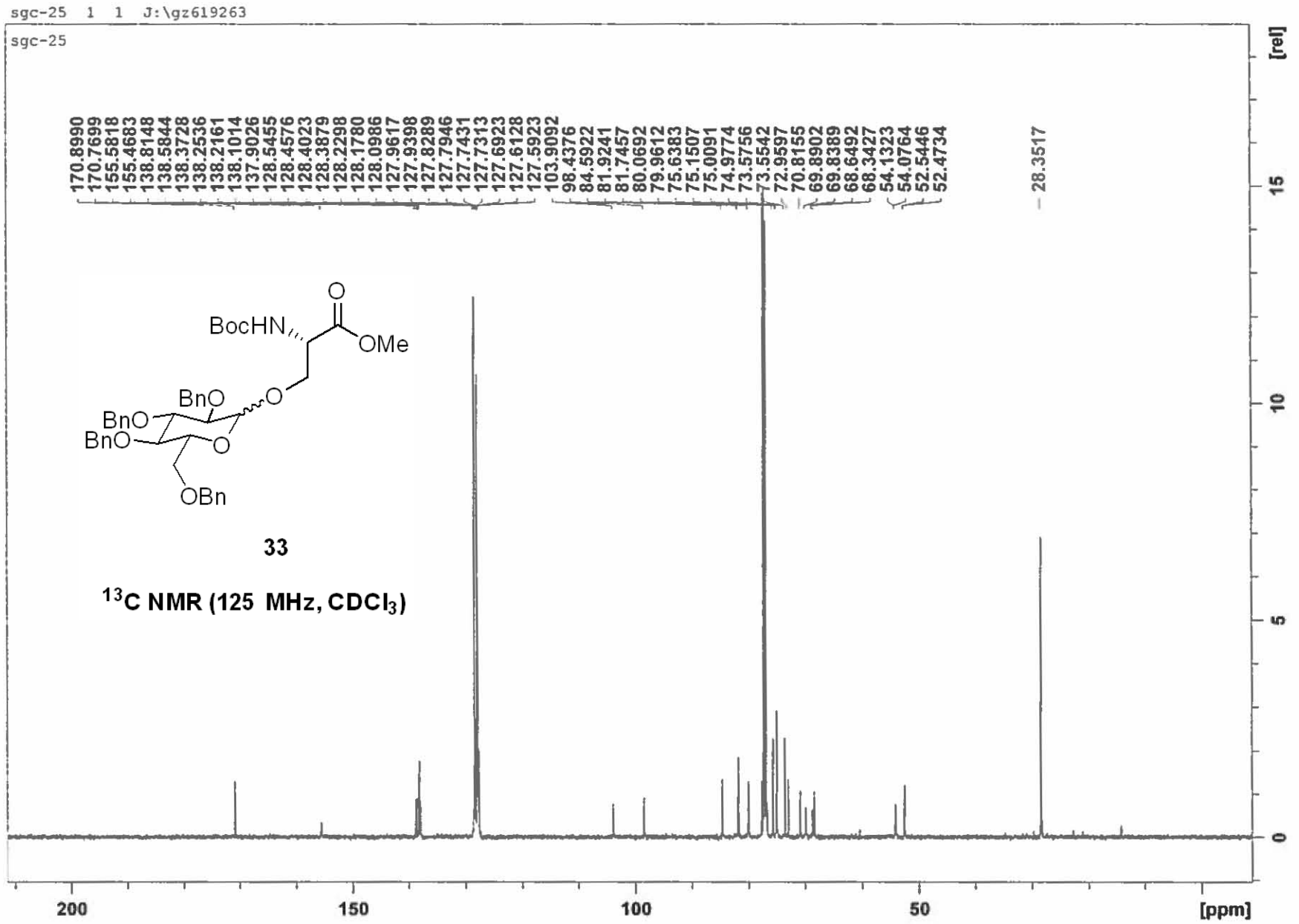
32

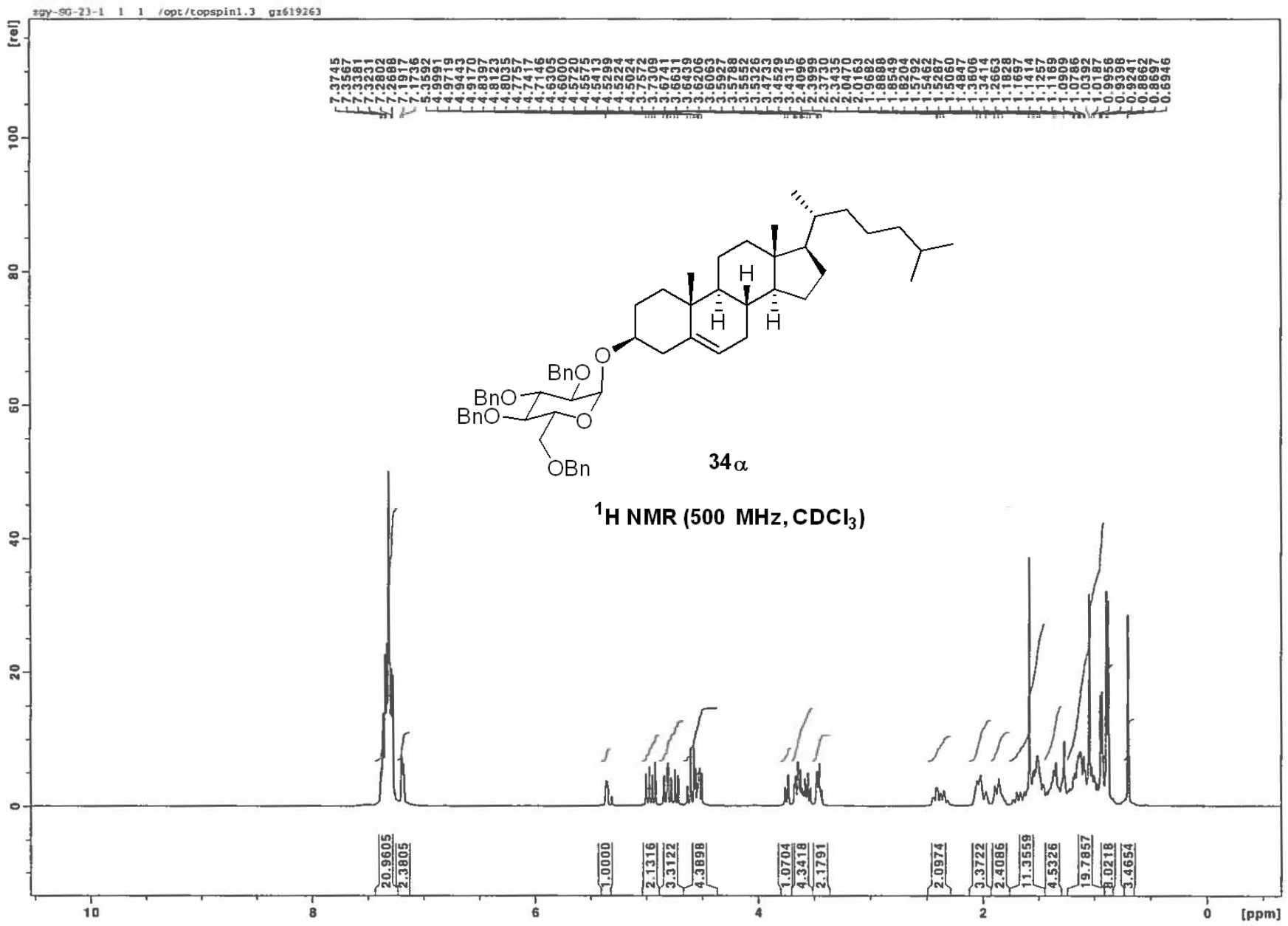
^{13}C NMR (125 MHz, $CDCl_3$)

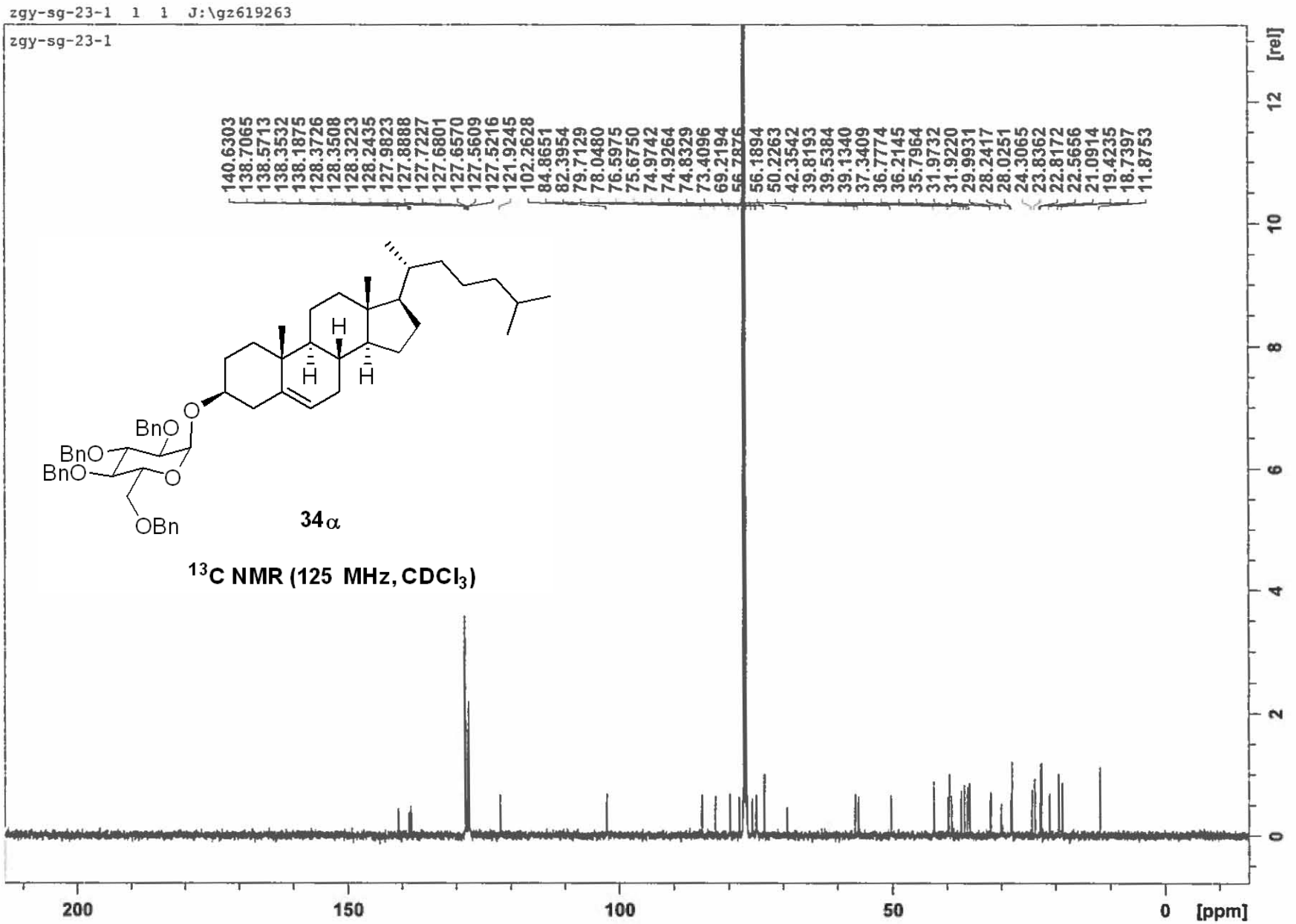


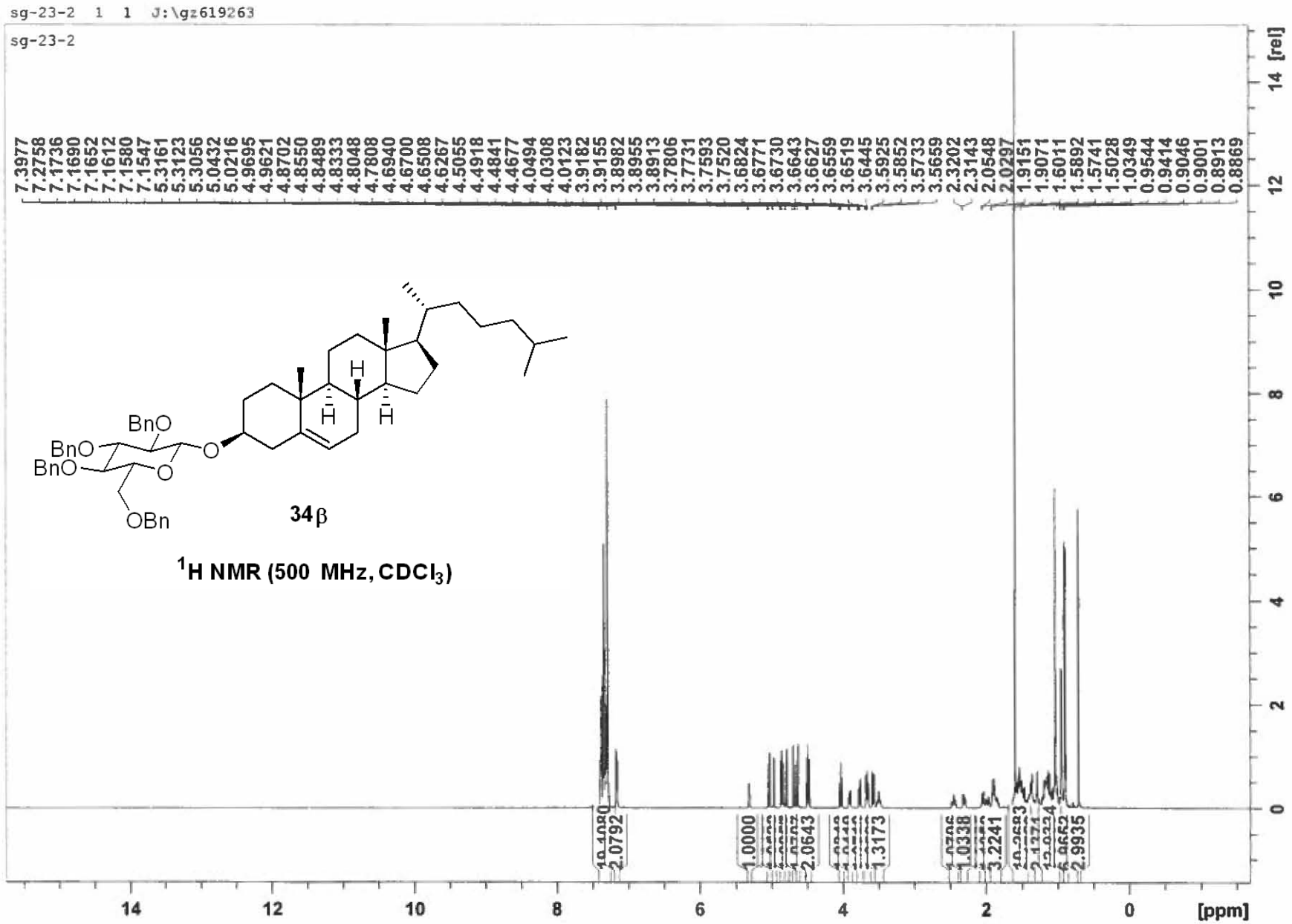
33

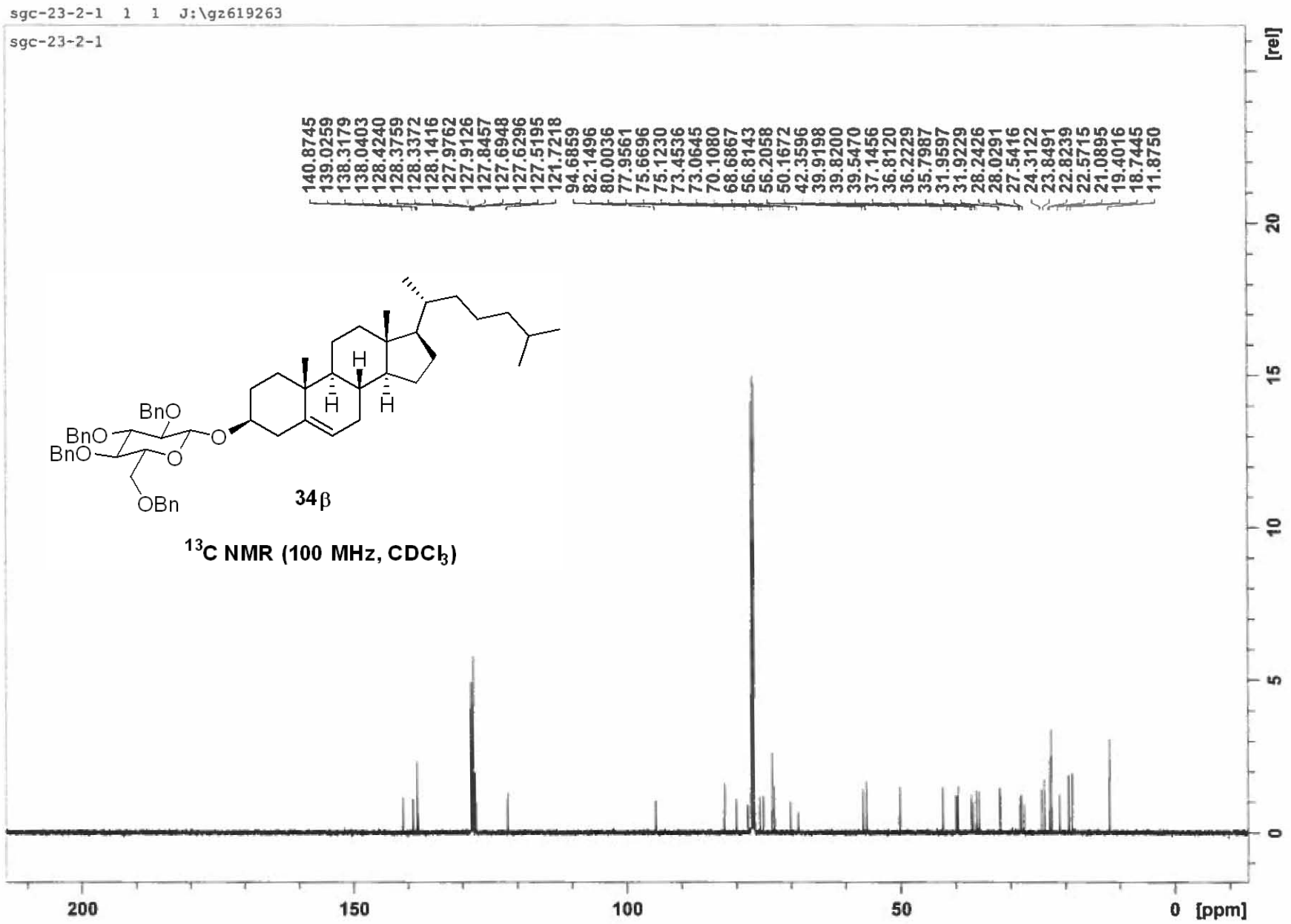
^{1}H NMR (400 MHz, $CDCl_3$)

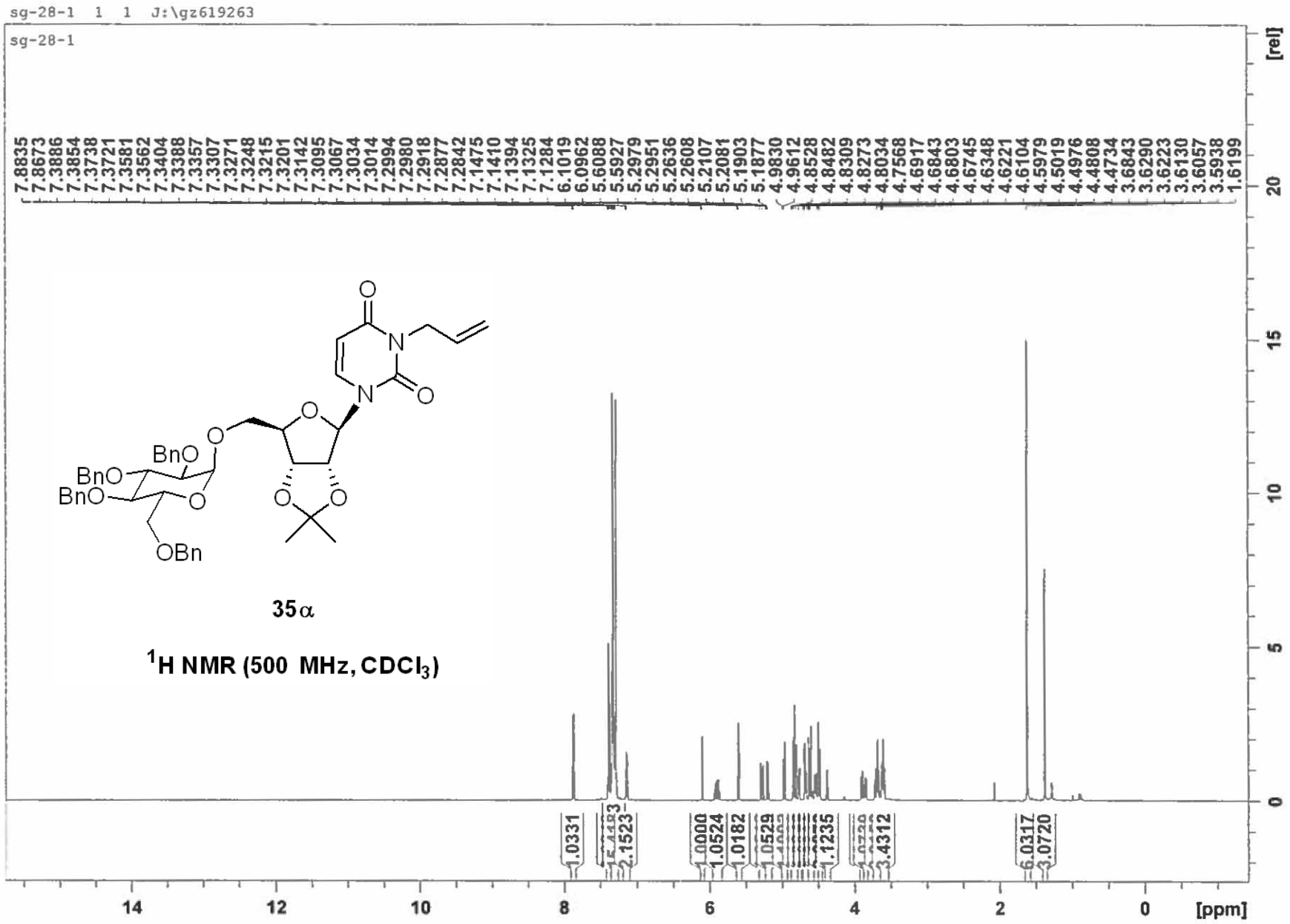

sgc-25 1 1 J:\gz619263
sgc-25
170.8990
170.7699
155.5818
155.4683
138.8148
138.5844
138.3728
138.2536
138.2161
138.1014
137.9026
128.5455
128.4576
128.4023
128.3879
128.2298
128.1780
128.0986
127.9617
127.9398
127.8289
127.7946
127.7431
127.7313
127.6923
127.6128
127.5923
103.9092
98.4376
84.5922
81.9241
81.7457
80.0692
79.9612
75.6383
75.1507
75.0091
74.9774
73.5756
73.5542
72.9597
70.8155
69.8902
69.8389
68.6492
68.3427
54.1323
54.0764
52.5446
52.4734
28.3517
BocHN
OMe
O
BnO
BnO
BnO
O
O
OBn
33
13C NMR (125 MHz, CDCl3)
[rel]
0
5
10
15
200
150
100
50
[ppm]

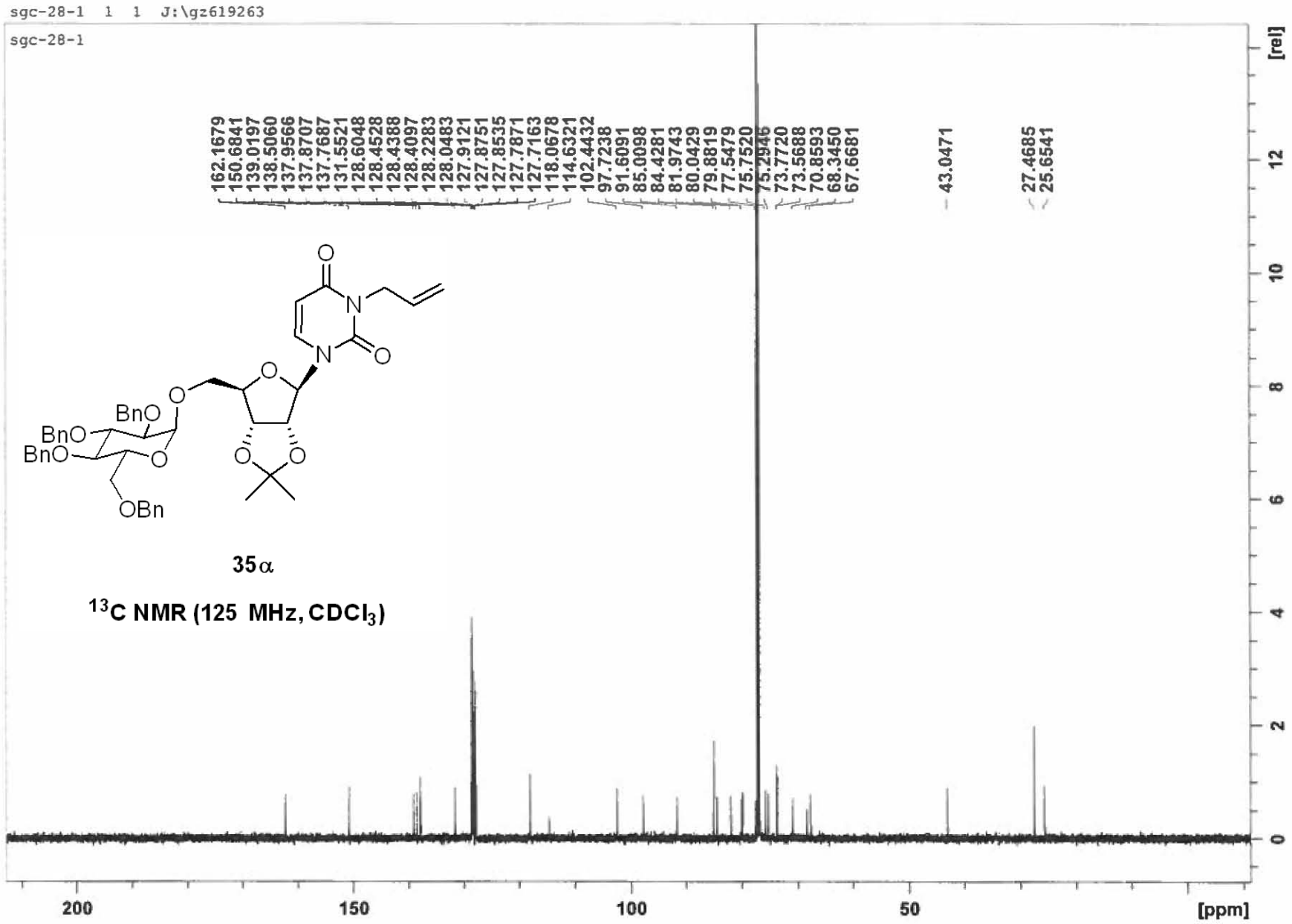

zgy-90-23-1 1 1 /opt/topspin1.3 gz619263
[rel]
BnO
BnO
BnO
OBn
H
H
H
O
O
34α
^{1}H NMR (500 MHz, CDCl$_3$)
[ppm]

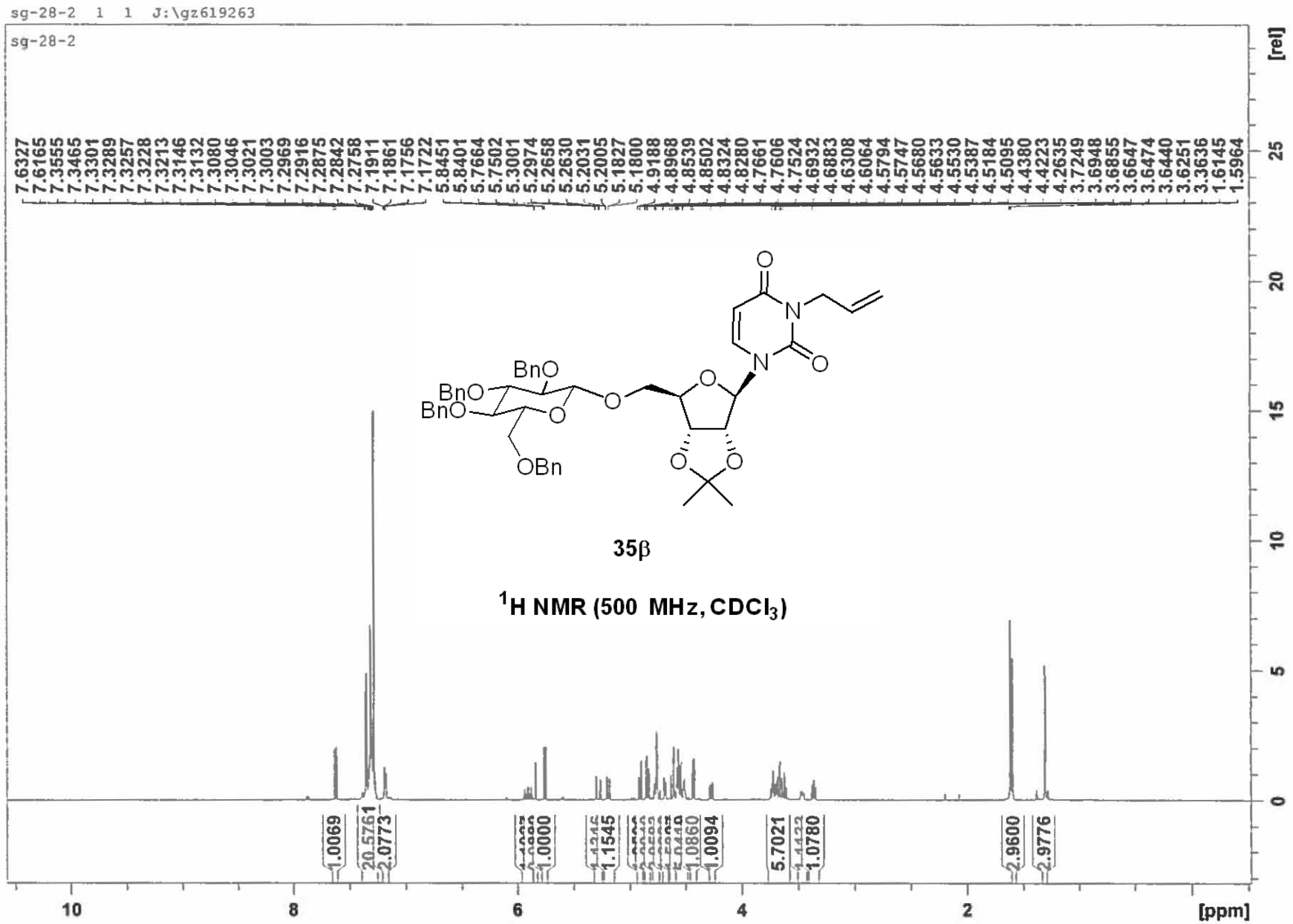

zgy-sg-23-1 1 1 J:\gz619263
zgy-sg-23-1
140.6303
138.7065
138.5713
138.3532
138.1875
128.3726
128.3508
128.3223
128.2435
127.9823
127.8888
127.7227
127.6801
127.6570
127.5609
127.5216
121.9245
102.2628
84.8651
82.3954
79.7129
78.0480
76.5975
75.6750
74.9742
74.9264
74.8329
73.4096
69.2194
56.7875
56.1894
50.2263
42.3542
39.8193
39.5384
39.1340
37.3409
36.7774
36.2145
35.7964
31.9732
31.9220
29.9931
28.2417
28.0251
24.3065
23.8362
22.8172
22.5656
21.0914
19.4235
18.7397
11.8753
BnO
BnO
BnO
OBn
O
H
H
H
34α
13C NMR (125 MHz, CDCl3)
[rel]
0
2
4
6
8
10
12
200
150
100
50
0
[ppm]


sg-23-2 1 1 J:\gz619263
sg-23-2
7.3977
7.2758
7.1736
7.1690
7.1652
7.1612
7.1580
7.1547
5.3161
5.3123
5.3056
5.0432
5.0216
4.9695
4.9621
4.8702
4.8550
4.8489
4.8333
4.8048
4.7808
4.6940
4.6700
4.6508
4.6267
4.5055
4.4918
4.4841
4.4677
4.0494
4.0308
4.0123
3.9182
3.9155
3.8982
3.8955
3.8913
3.7806
3.7731
3.7593
3.7520
3.6824
3.6771
3.6730
3.6643
3.6627
3.6559
3.6519
3.6445
3.5925
3.5852
3.5733
3.5659
2.3202
2.3143
2.0548
2.0297
1.9151
1.9071
1.6011
1.5892
1.5741
1.5028
1.0349
0.9544
0.9414
0.9046
0.9001
0.8913
0.8869
BnO
BnO
BnO
O
O
OBn
H
H
H
34β
1H NMR (500 MHz, CDCl3)
2.0792
1.0000
2.0643
1.3173
1.0338
3.2241
2.9935
14 [rel]
12
10
8
6
4
2
0
[ppm]


sgc-23-2-1 1 1 J:\gz619263
sgc-23-2-1
140.8745
139.0259
138.3179
138.0403
128.4240
128.3759
128.3372
128.1416
127.9762
127.9126
127.8457
127.6948
127.6296
127.5195
121.7218
94.6859
82.1496
80.0036
77.9561
75.6696
75.1230
73.4536
73.0645
70.1080
68.6867
56.8143
56.2058
50.1672
42.3596
39.9198
39.8200
39.5470
37.1456
36.8120
36.2229
35.7987
31.9597
31.9229
28.2426
28.0291
27.5416
24.3122
23.8491
22.8239
22.5715
21.0895
19.4016
18.7445
11.8750
[rel]
BnO
BnO
BnO
OBn
O
O
H
H
H
34β
^{13}C NMR (100 MHz, CDCl$_3$)
200
150
100
50
0 [ppm]

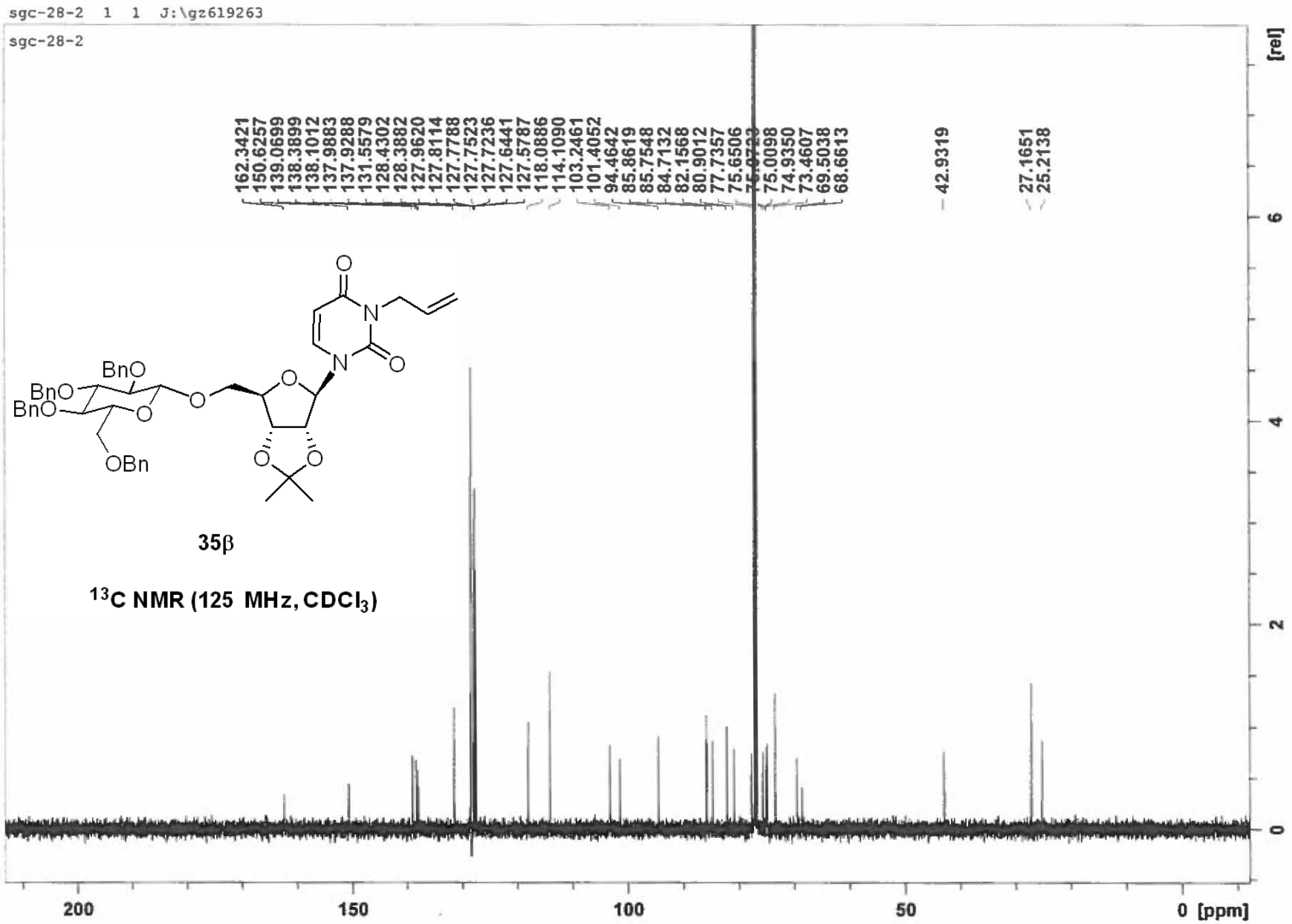

sg-28-1 1 1 J:\gz619263
sg-28-1
7.8835
7.8673
7.3886
7.3854
7.3738
7.3721
7.3581
7.3562
7.3404
7.3388
7.3357
7.3307
7.3271
7.3248
7.3215
7.3201
7.3142
7.3095
7.3067
7.3034
7.3014
7.2994
7.2980
7.2918
7.2877
7.2842
7.1475
7.1410
7.1394
7.1325
7.1284
6.1019
6.0962
5.6088
5.5927
5.2979
5.2951
5.2636
5.2608
5.2107
5.2081
5.1903
5.1877
4.9830
4.9612
4.8528
4.8482
4.8309
4.8273
4.8034
4.7568
4.6917
4.6843
4.6803
4.6745
4.6348
4.6221
4.6104
4.5979
4.5019
4.4976
4.4808
4.4734
3.6843
3.6290
3.6223
3.6130
3.6057
3.5938
1.6199
BnO
BnO
BnO
OBn
35α
1H NMR (500 MHz, CDCl3)
1.0331
2.1523
1.0000
1.0524
1.0182
1.0529
1.1235
3.4312
6.0317
3.0720
[rel]
[ppm]


sgc-28-1 1 1 J:\gz619263
sgc-28-1
162.1679
150.6841
139.0197
138.5060
137.9566
137.8707
137.7687
131.5521
128.6048
128.4528
128.4388
128.4097
128.2283
128.0483
127.9121
127.8751
127.8535
127.7871
127.7163
118.0678
114.6321
102.4432
97.7238
91.6091
85.0098
84.4281
81.9743
80.0429
79.8819
77.5479
75.7520
75.2946
73.7720
73.5688
70.8593
68.3450
67.6681
43.0471
27.4685
25.6541
BnO
BnO
BnO
OBn
35α
13C NMR (125 MHz, CDCl3)
[rel]
0
2
4
6
8
10
12
200
150
100
50
[ppm]



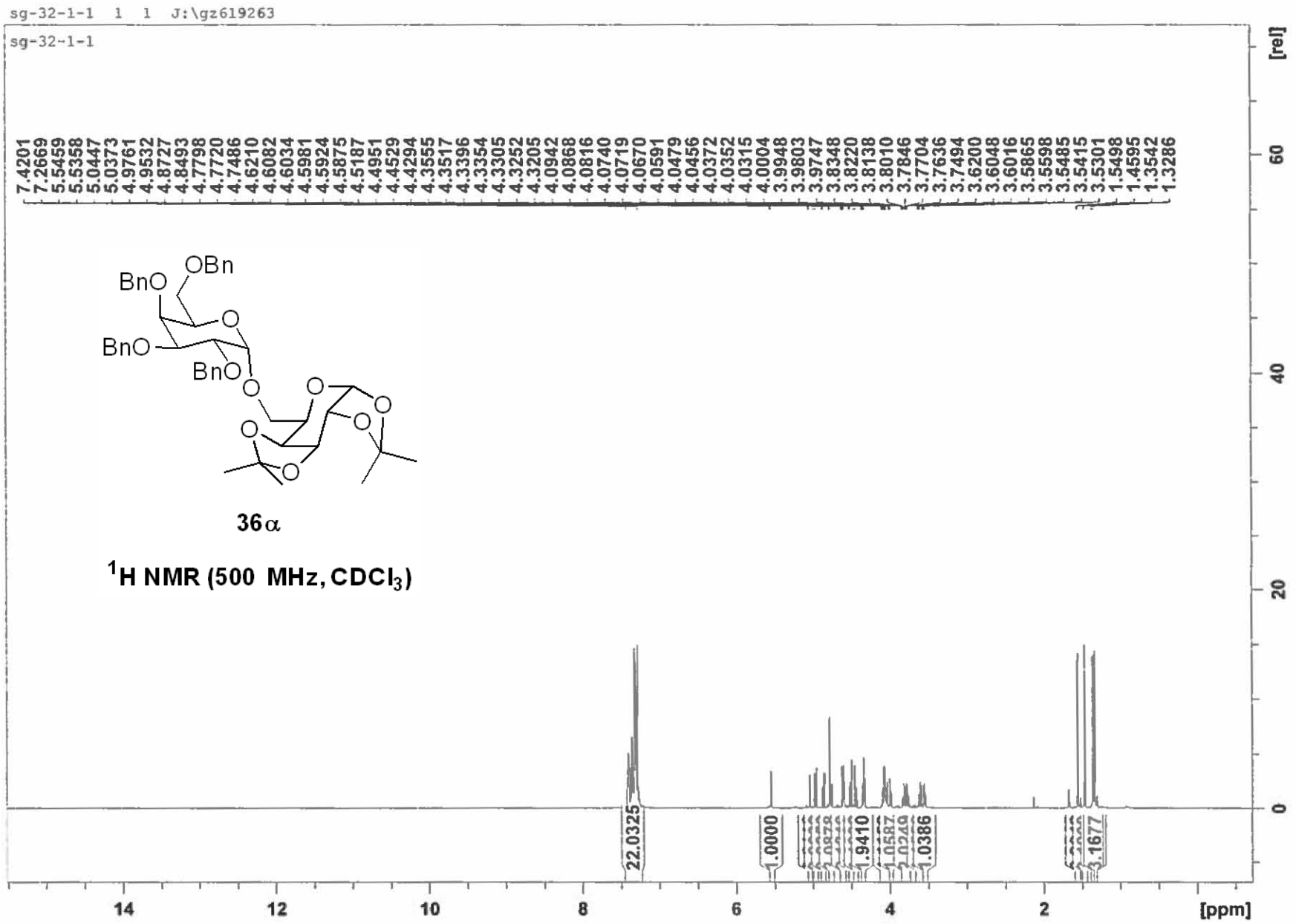
35β

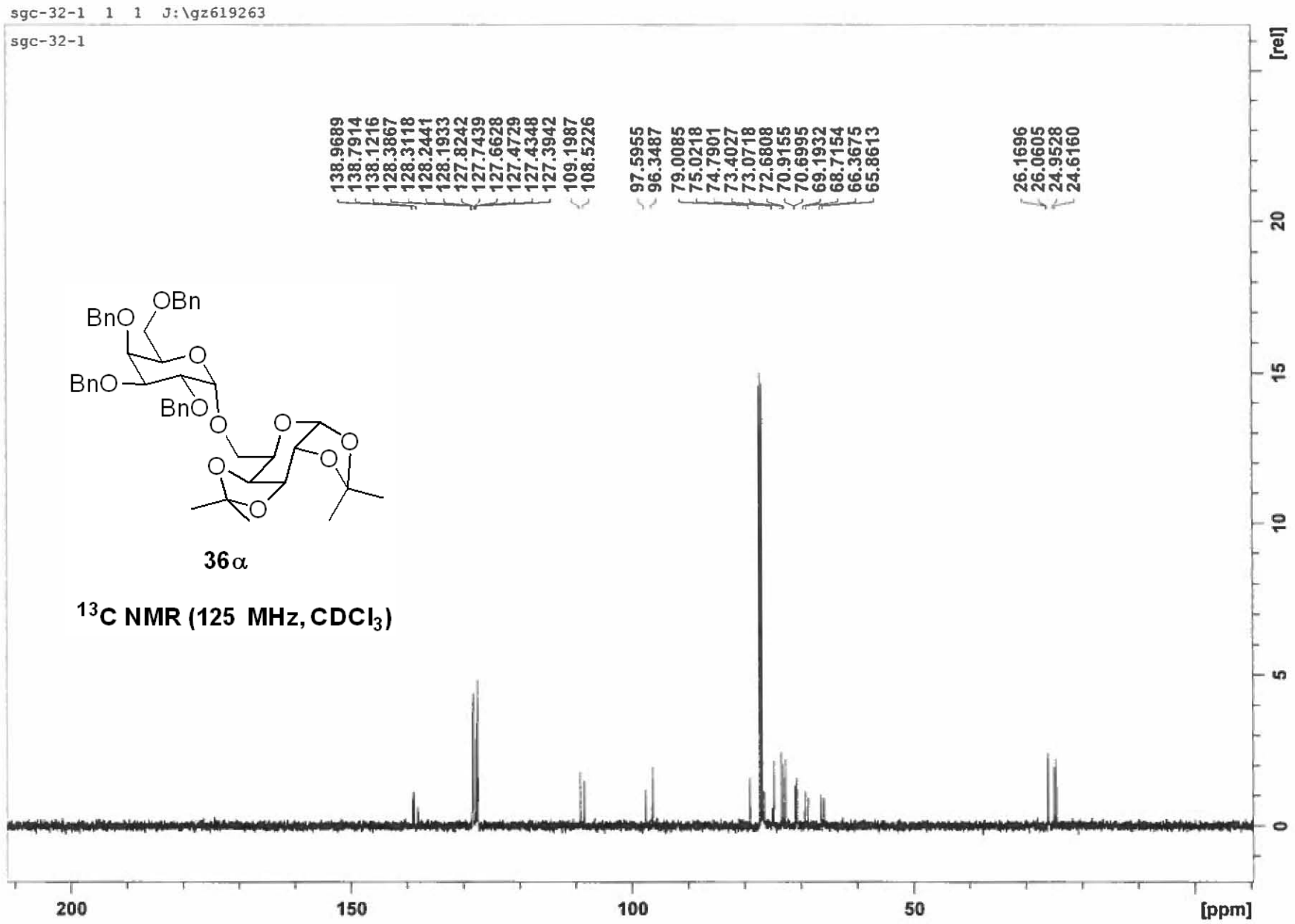
^{1}H NMR (500 MHz, $CDCl_3$)

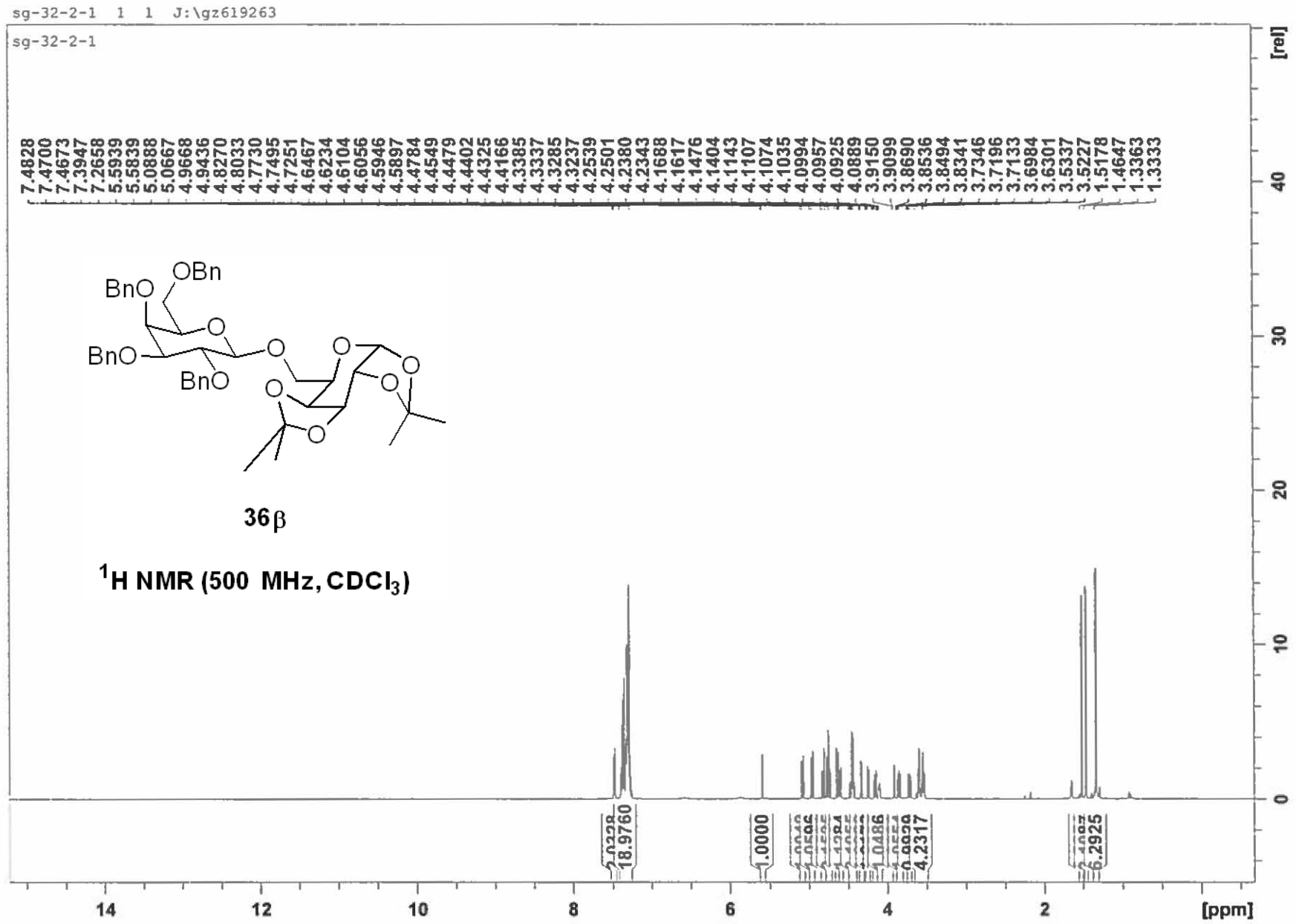


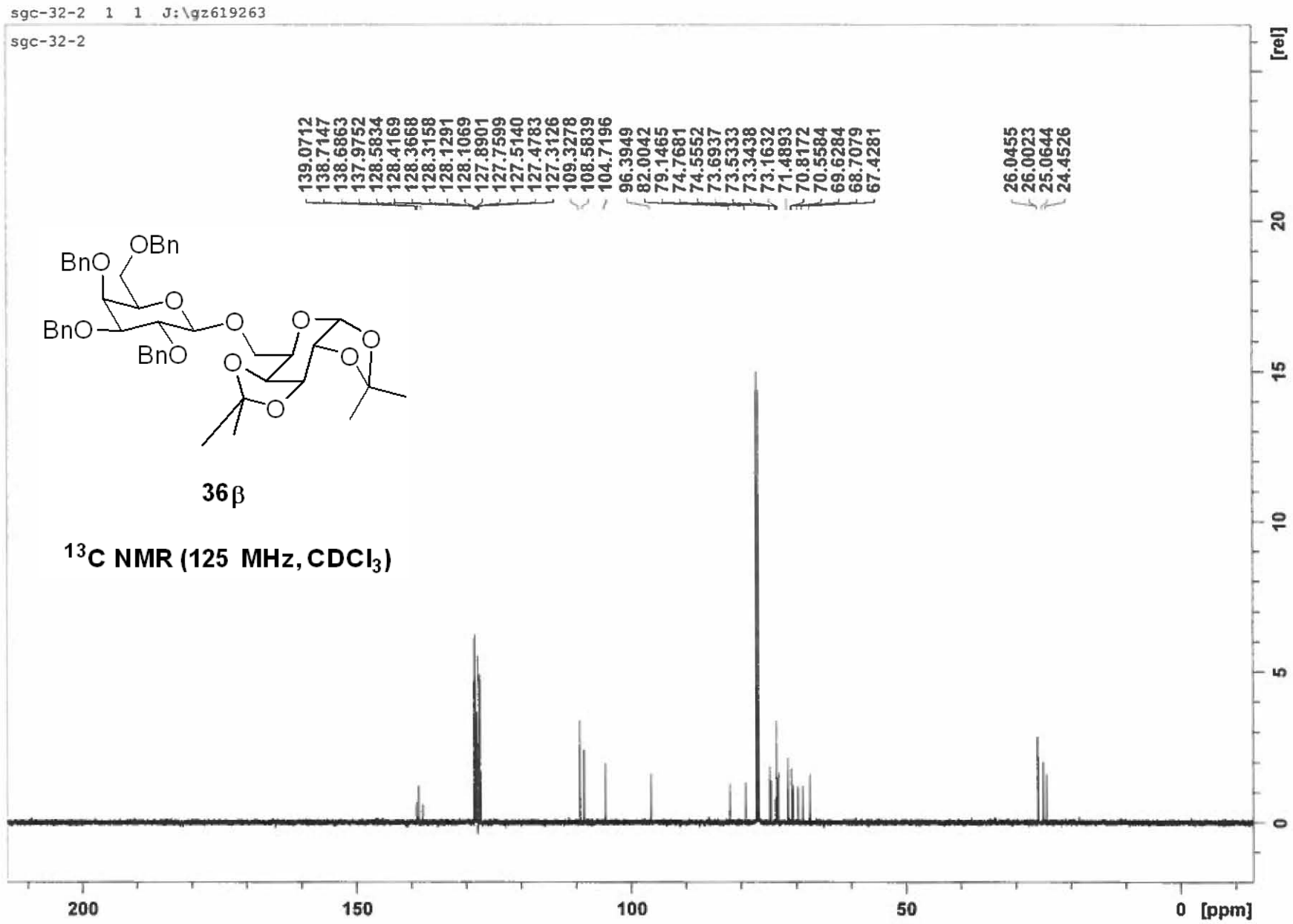
35β

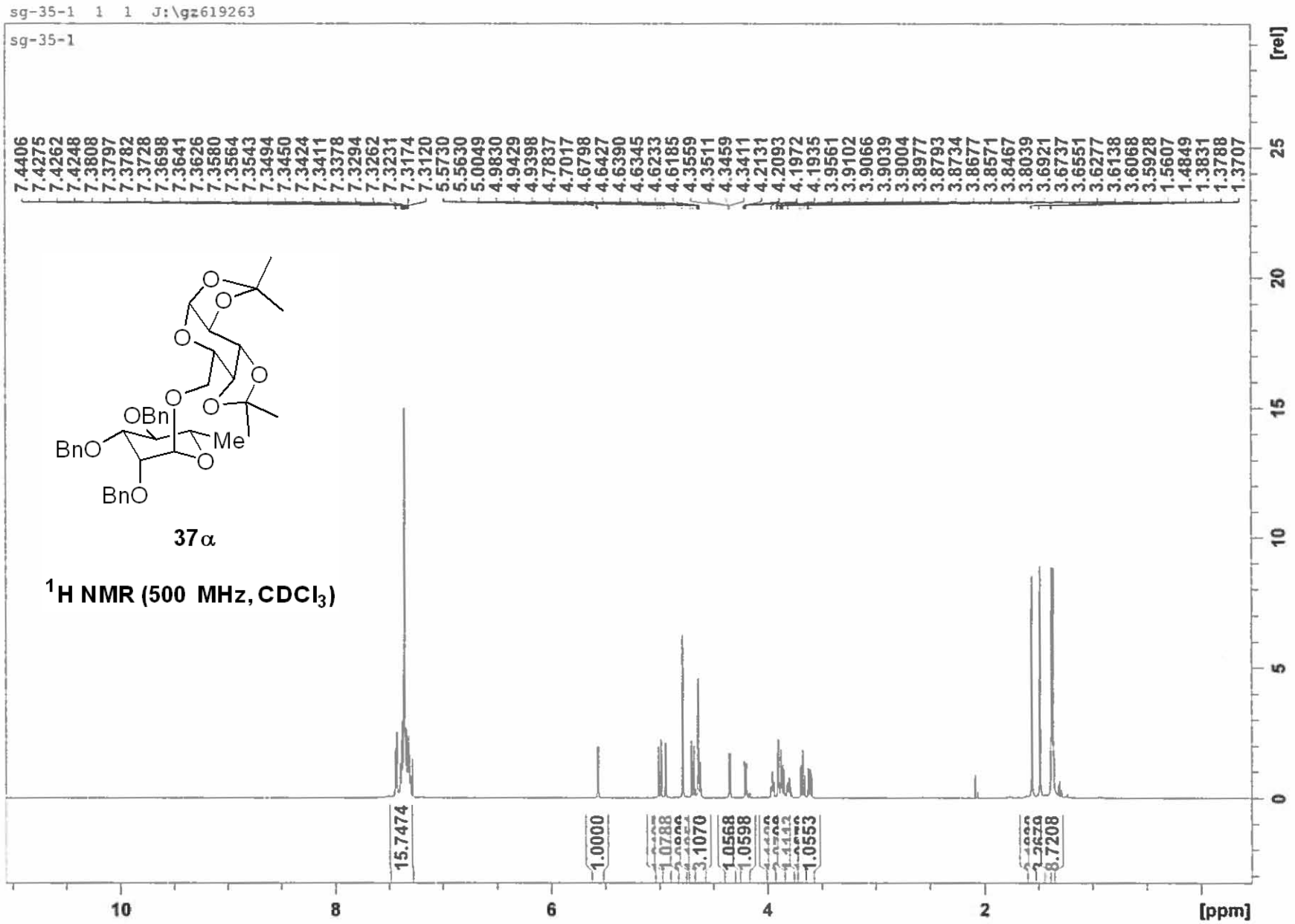
^{13}C NMR (125 MHz, $CDCl_3$)

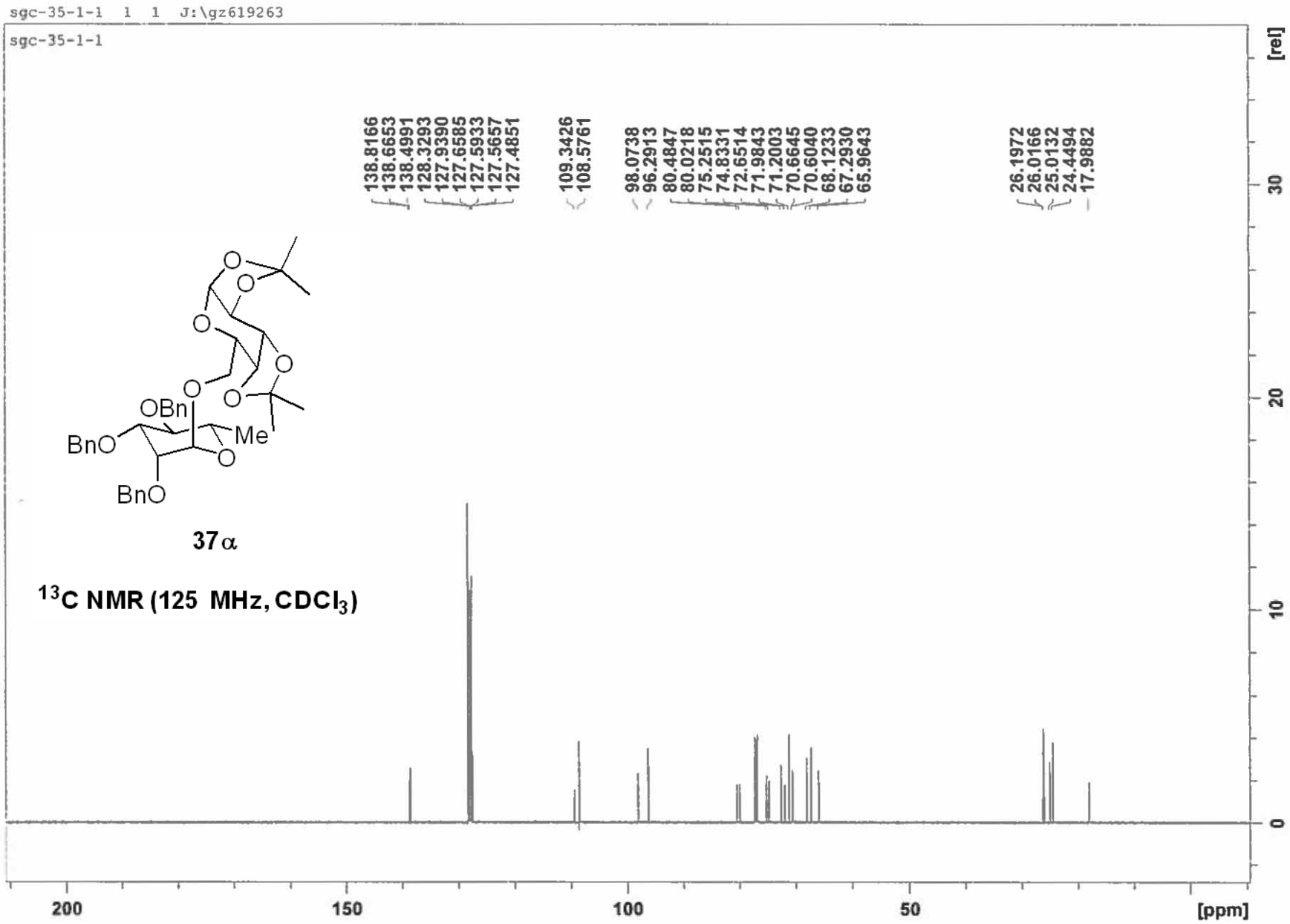

sg-32-1-1 1 1 J:\gz619263
sg-32-1-1
7.4201
7.2669
5.5459
5.5358
5.0447
5.0373
4.9761
4.9532
4.8727
4.8493
4.7798
4.7720
4.7486
4.6210
4.6082
4.6034
4.5981
4.5924
4.5875
4.5187
4.4951
4.4529
4.4294
4.3555
4.3517
4.3396
4.3354
4.3305
4.3252
4.3205
4.0942
4.0868
4.0816
4.0740
4.0719
4.0670
4.0591
4.0479
4.0456
4.0372
4.0352
4.0315
4.0004
3.9948
3.9803
3.9747
3.8348
3.8220
3.8138
3.8010
3.7846
3.7704
3.7636
3.7494
3.6200
3.6048
3.6016
3.5865
3.5598
3.5485
3.5415
3.5301
1.5498
1.4595
1.3542
1.3286
BnO
OBn
BnO
BnO
O
O
O
O
O
O
O
O
O
36α
^{1}H NMR (500 MHz, CDCl$_3$)
22.0325
1.0000
1.9410
1.0587
1.0386
3.1677
14
12
10
8
6
4
2
[ppm]
[rel]
60
40
20
0

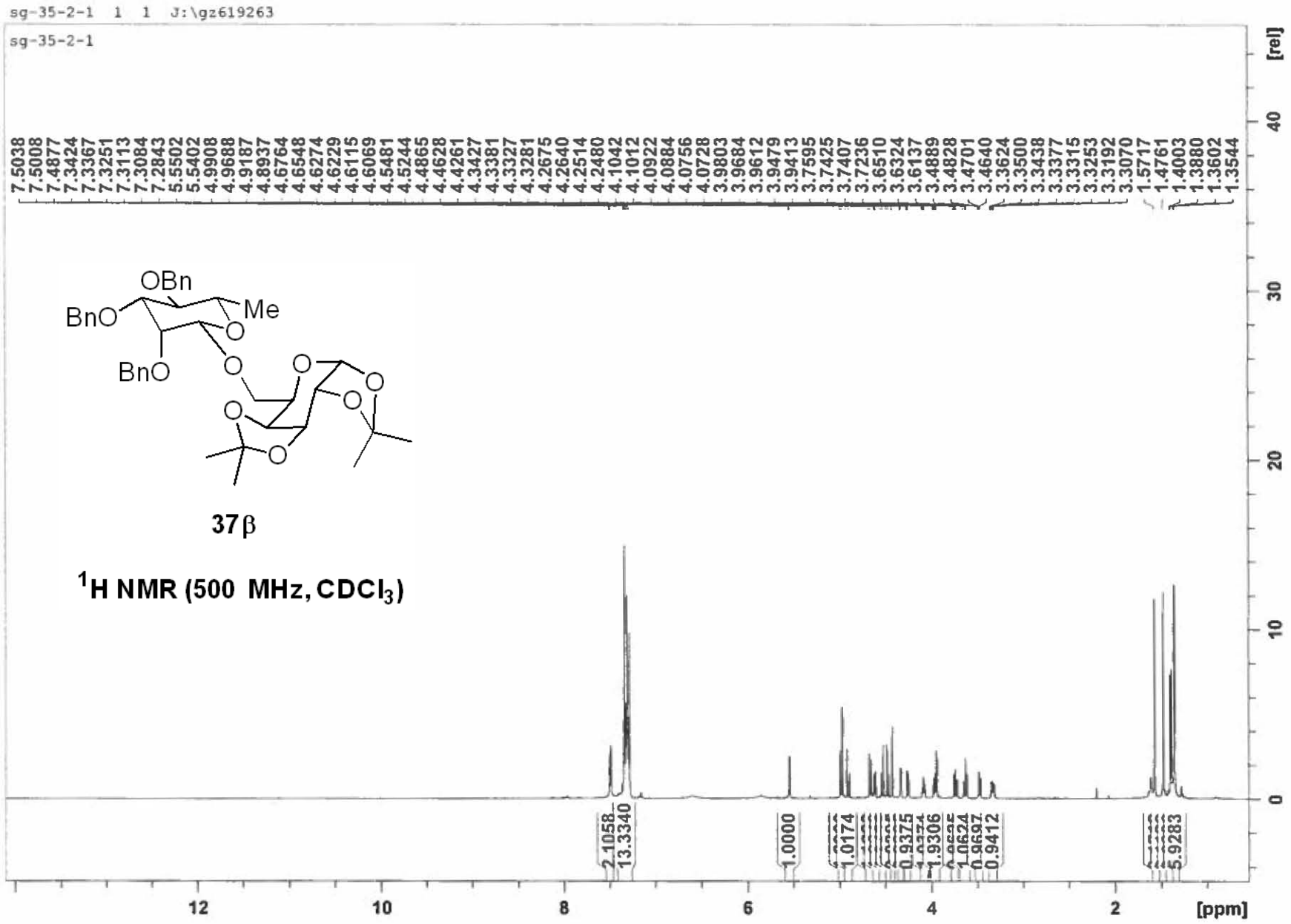

sgc-32-1 1 1 J:\gz619263
sgc-32-1
138.9689
138.7914
138.1216
128.3867
128.3118
128.2441
128.1933
127.8242
127.7439
127.6628
127.4729
127.4348
127.3942
109.1987
108.5226
97.5955
96.3487
79.0085
75.0218
74.7901
73.4027
73.0718
72.6808
70.9155
70.6995
69.1932
68.7154
66.3675
65.8613
26.1696
26.0605
24.9528
24.6160
BnO
OBn
BnO
BnO
O
O
O
O
O
O
O
O
36α
13C NMR (125 MHz, CDCl3)
[rel]
20
15
10
5
0
200
150
100
50
[ppm]

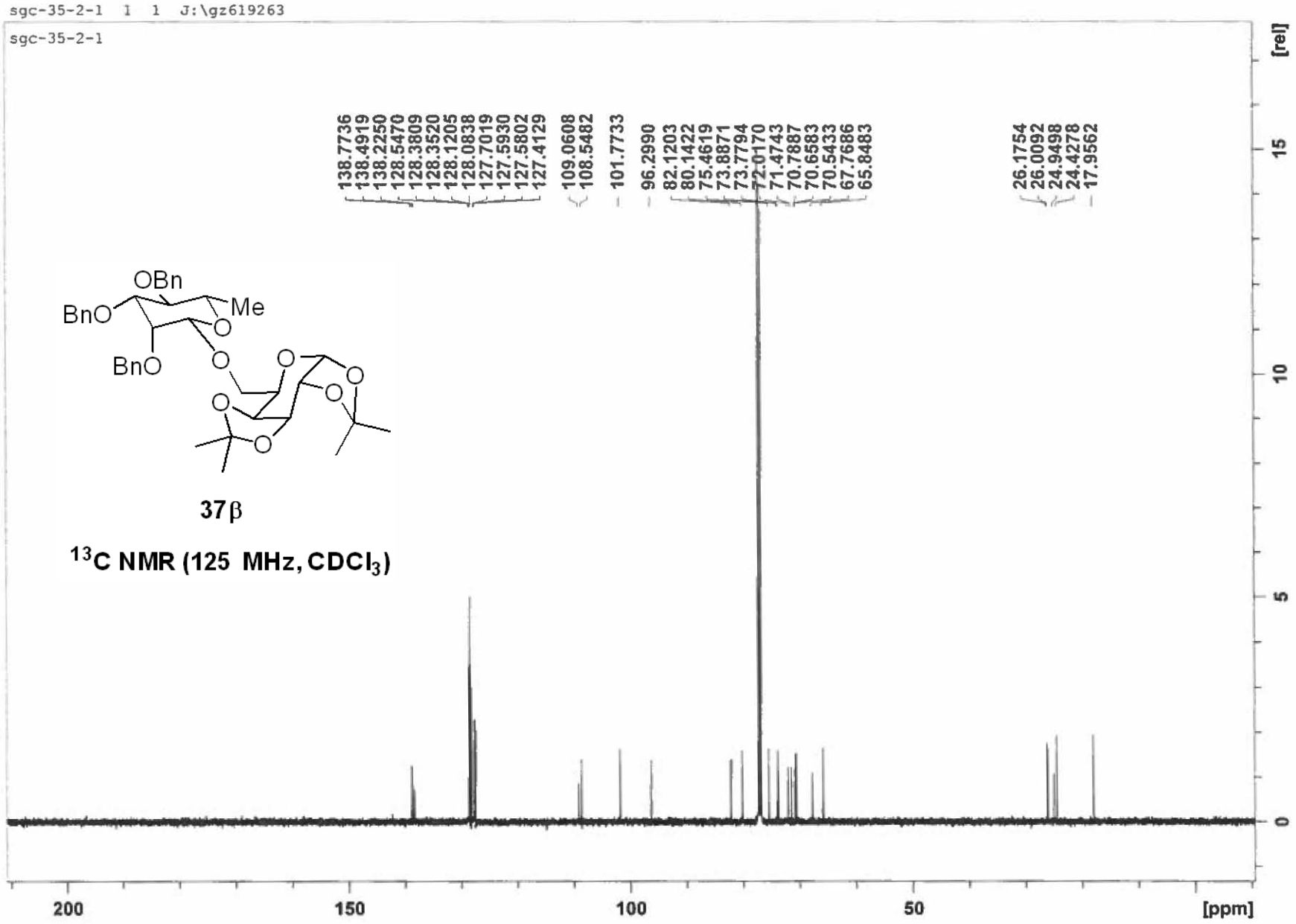

sg-32-2-1 1 1 J:\gz619263
sg-32-2-1
7.4828
7.4700
7.4673
7.3947
7.2658
5.5939
5.5839
5.0888
5.0667
4.9668
4.9436
4.8270
4.8033
4.7730
4.7495
4.7251
4.6467
4.6234
4.6104
4.6056
4.5946
4.5897
4.4784
4.4549
4.4479
4.4402
4.4325
4.4166
4.3385
4.3337
4.3285
4.3237
4.2539
4.2501
4.2380
4.2343
4.1688
4.1617
4.1476
4.1404
4.1143
4.1107
4.1074
4.1035
4.0994
4.0957
4.0925
4.0889
3.9150
3.9099
3.8690
3.8536
3.8494
3.8341
3.7346
3.7196
3.7133
3.6984
3.6301
3.5337
3.5227
1.5178
1.4647
1.3363
1.3333
BnO
OBn
BnO
BnO
36β
1H NMR (500 MHz, CDCl3)
[rel]
40
30
20
10
0
2.0328
18.9760
1.0000
1.0486
4.2317
6.2925
14
12
10
8
6
4
2
[ppm]

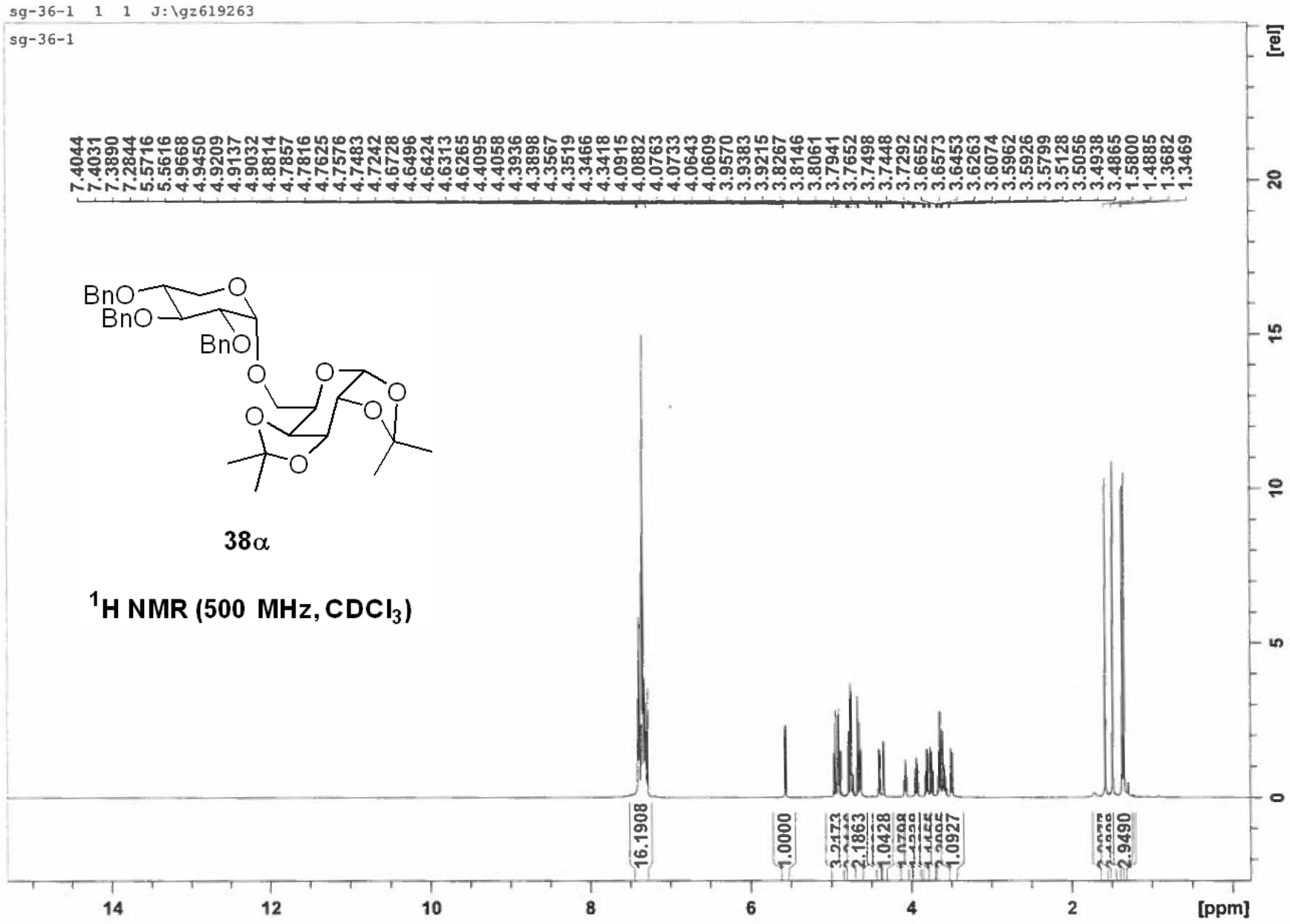

sgc-32-2 1 1 J:\gz619263
sgc-32-2
139.0712
138.7147
138.6863
137.9752
128.5834
128.4169
128.3668
128.3158
128.1291
128.1069
127.8901
127.7599
127.5140
127.4783
127.3126
109.3278
108.5839
104.7196
96.3949
82.0042
79.1465
74.7681
74.5552
73.6937
73.5333
73.3438
73.1632
71.4893
70.8172
70.5584
69.6284
68.7079
67.4281
26.0455
26.0023
25.0644
24.4526
BnO
OBn
BnO
BnO
O
O
O
O
O
O
O
O
O
36β
13C NMR (125 MHz, CDCl3)
[rel]
20
15
10
5
0
200
150
100
50
0 [ppm]


sg-35-1 1 1 J:\gz619263
sg-35-1
7.4406
7.4275
7.4262
7.4248
7.3808
7.3797
7.3782
7.3728
7.3698
7.3641
7.3626
7.3580
7.3564
7.3543
7.3494
7.3450
7.3424
7.3411
7.3378
7.3294
7.3262
7.3231
7.3174
7.3120
5.5730
5.5630
5.0049
4.9830
4.9429
4.9398
4.7837
4.7017
4.6798
4.6427
4.6390
4.6345
4.6233
4.6185
4.3559
4.3511
4.3459
4.3411
4.2131
4.2093
4.1972
4.1935
3.9561
3.9102
3.9066
3.9039
3.9004
3.8977
3.8793
3.8734
3.8677
3.8571
3.8467
3.8039
3.6921
3.6737
3.6551
3.6277
3.6138
3.6068
3.5928
1.5607
1.4849
1.3831
1.3788
1.3707
OBn
BnO
Me
O
BnO
37α
1H NMR (500 MHz, CDCl3)
15.7474
1.0000
1.0788
3.1070
1.0568
1.0598
1.0553
8.7208
10
8
6
4
2
[ppm]
[rel]
25
20
15
10
5
0


sgc-35-1-1 1 1 J:\gz619263
sgc-35-1-1
138.8166
138.6653
138.4991
128.3293
127.9390
127.6585
127.5933
127.5657
127.4851
109.3426
108.5761
98.0738
96.2913
80.4847
80.0218
75.2515
74.8331
72.6514
71.9843
71.2003
70.6645
70.6040
68.1233
67.2930
65.9643
26.1972
26.0166
25.0132
24.4494
17.9882
OBn
BnO
Me
BnO
37α
13C NMR (125 MHz, CDCl3)
[rel]
30
20
10
0
200
150
100
50
[ppm]

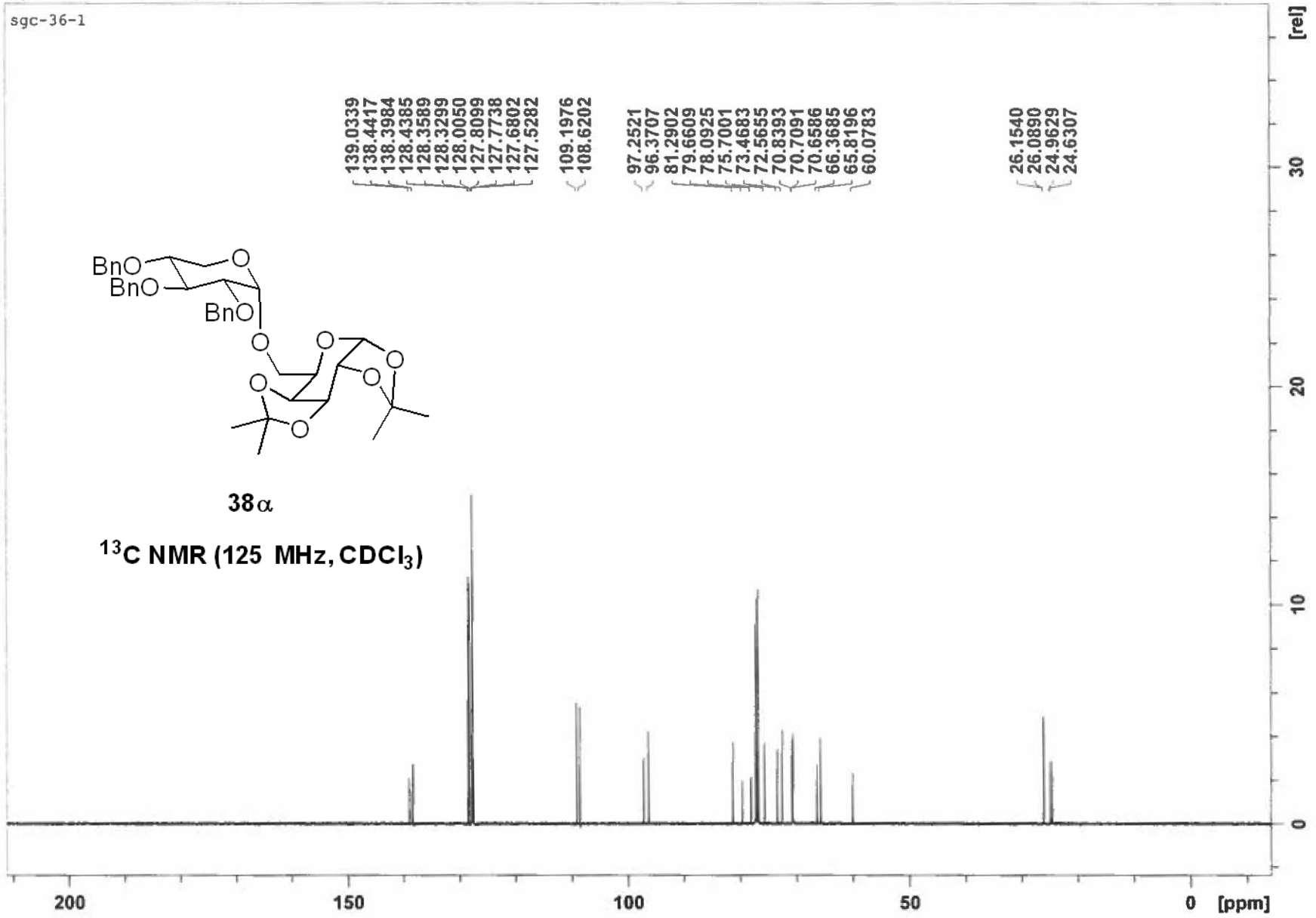

sg-35-2-1 1 1 J:\gz619263
sg-35-2-1
7.5038
7.5008
7.4877
7.3424
7.3367
7.3251
7.3113
7.3084
7.2843
5.5502
5.5402
4.9908
4.9688
4.9187
4.8937
4.6764
4.6548
4.6274
4.6229
4.6115
4.6069
4.5481
4.5244
4.4865
4.4628
4.4261
4.3427
4.3381
4.3327
4.3281
4.2675
4.2640
4.2514
4.2480
4.1042
4.1012
4.0922
4.0884
4.0756
4.0728
3.9803
3.9684
3.9612
3.9479
3.9413
3.7595
3.7425
3.7407
3.7236
3.6510
3.6324
3.6137
3.4889
3.4828
3.4701
3.4640
3.3624
3.3500
3.3438
3.3377
3.3315
3.3253
3.3192
3.3070
1.5717
1.4761
1.4003
1.3880
1.3602
1.3544
[rel]
40
30
20
10
0
OBn
BnO
Me
BnO
37β
1H NMR (500 MHz, CDCl3)
2.1058
13.3340
1.0000
1.0174
0.9375
1.9306
1.0624
0.9697
0.9412
5.9283
12
10
8
6
4
2
[ppm]


sgc-35-2-1 1 1 J:\gz619263
sgc-35-2-1
138.7736
138.4919
138.2250
128.5470
128.3809
128.3520
128.1205
128.0838
127.7019
127.5930
127.5802
127.4129
109.0608
108.5482
101.7733
96.2990
82.1203
80.1422
75.4619
73.8871
73.7794
72.0170
71.4743
70.7887
70.6583
70.5433
67.7686
65.8483
26.1754
26.0092
24.9498
24.4278
17.9562
OBn
BnO
Me
BnO
37β
^{13}C NMR (125 MHz, CDCl$_3$)
[rel]
15
10
5
0
200
150
100
50
[ppm]


sg-36-1 1 1 J:\gz619263
sg-36-1
7.4044
7.4031
7.3890
7.2844
5.5716
5.5616
4.9668
4.9450
4.9209
4.9137
4.9032
4.8814
4.7857
4.7816
4.7625
4.7576
4.7483
4.7242
4.6728
4.6496
4.6424
4.6313
4.6265
4.4095
4.4058
4.3936
4.3898
4.3567
4.3519
4.3466
4.3418
4.0915
4.0882
4.0763
4.0733
4.0643
4.0609
3.9570
3.9383
3.9215
3.8267
3.8146
3.8061
3.7941
3.7652
3.7498
3.7448
3.7292
3.6652
3.6573
3.6453
3.6263
3.6074
3.5962
3.5926
3.5799
3.5128
3.5056
3.4938
3.4865
1.5800
1.4885
1.3682
1.3469
BnO
BnO
BnO
38α
1H NMR (500 MHz, CDCl3)
16.1908
1.0000
3.2473
2.1863
1.0428
1.0927
2.9490
[rel]
[ppm]

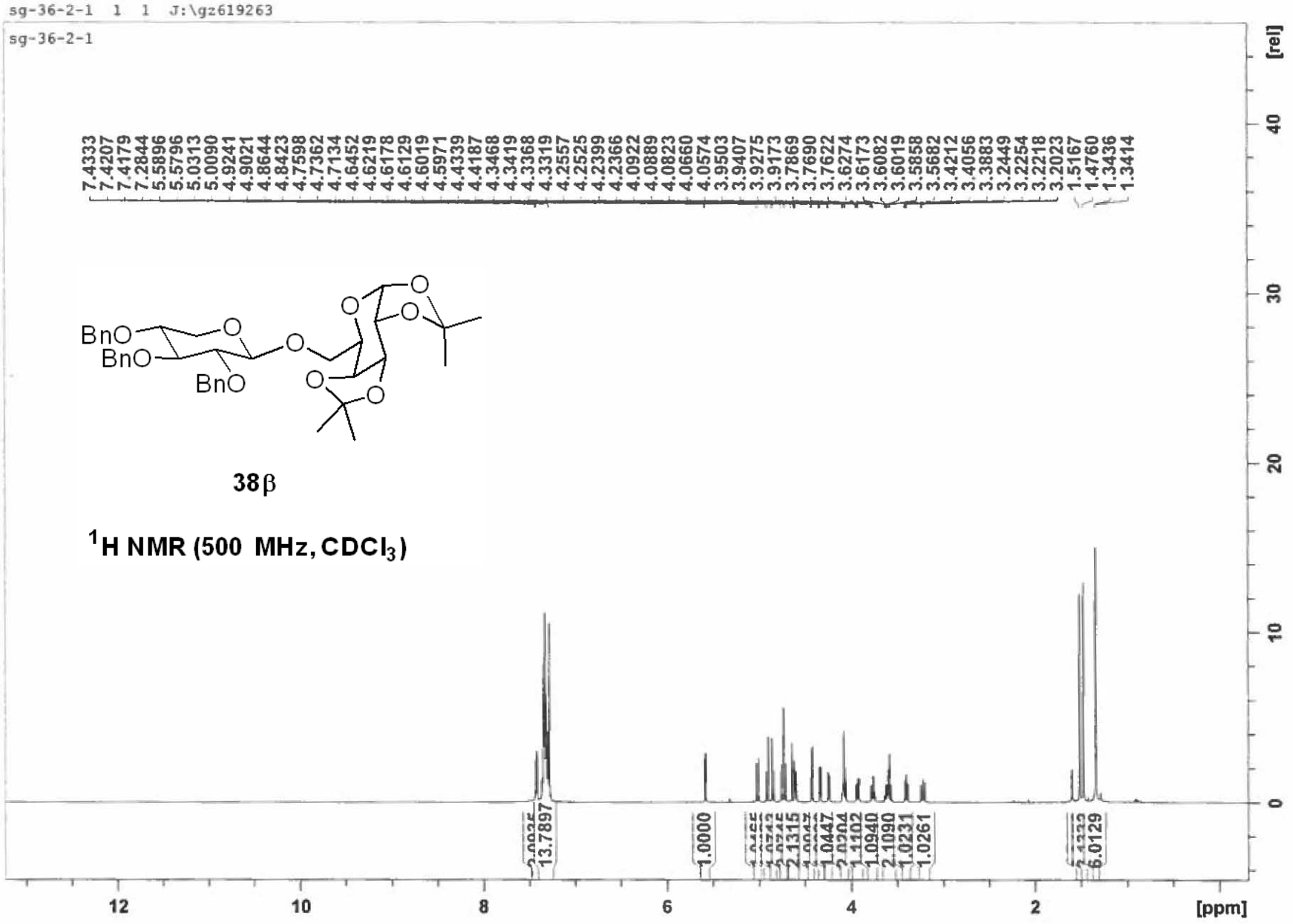
sgc-36-1 1 1 J:\gz619263

sgc-36-1



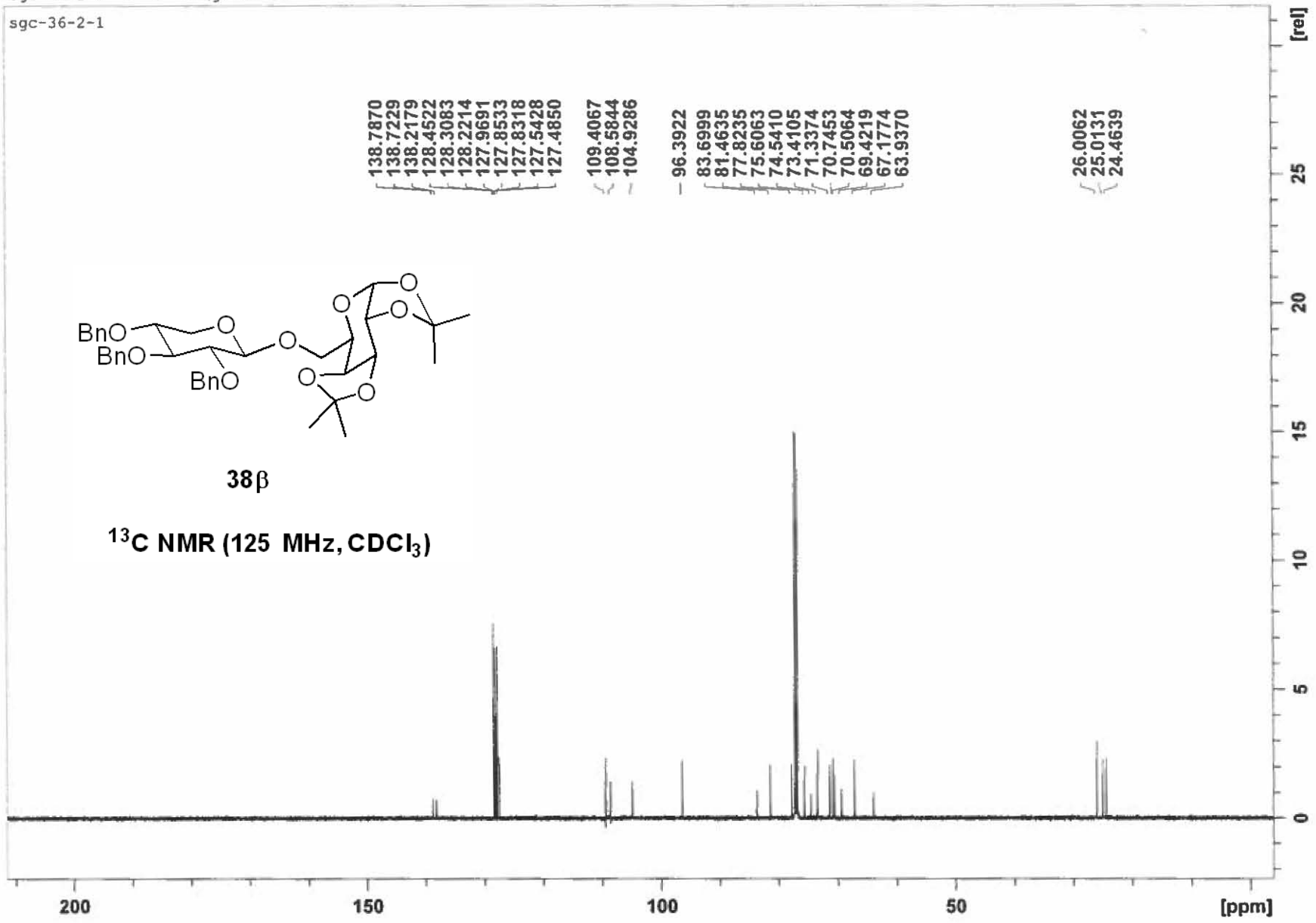
38α

^{13}C NMR (125 MHz, $CDCl_3$)


sg-36-2-1 1 1 J:\gz619263
sg-36-2-1
7.4333
7.4207
7.4179
7.2844
5.5896
5.5796
5.0313
5.0090
4.9241
4.9021
4.8644
4.8423
4.7598
4.7362
4.7134
4.6452
4.6219
4.6178
4.6129
4.6019
4.5971
4.4339
4.4187
4.3468
4.3419
4.3368
4.3319
4.2557
4.2525
4.2399
4.2366
4.0922
4.0889
4.0823
4.0660
4.0574
3.9503
3.9407
3.9275
3.9173
3.7869
3.7690
3.7622
3.6274
3.6173
3.6082
3.6019
3.5858
3.5682
3.4212
3.4056
3.3883
3.2449
3.2254
3.2218
3.2023
1.5167
1.4760
1.3436
1.3414
BnO
BnO
BnO
O
O
O
O
O
O
O
O
38β
1H NMR (500 MHz, CDCl3)
[rel]
40
30
20
10
0
13.7897
1.0000
2.1315
1.0447
2.0904
1.1102
1.0940
2.1090
1.0231
1.0261
6.0129
12
10
8
6
4
2
[ppm]

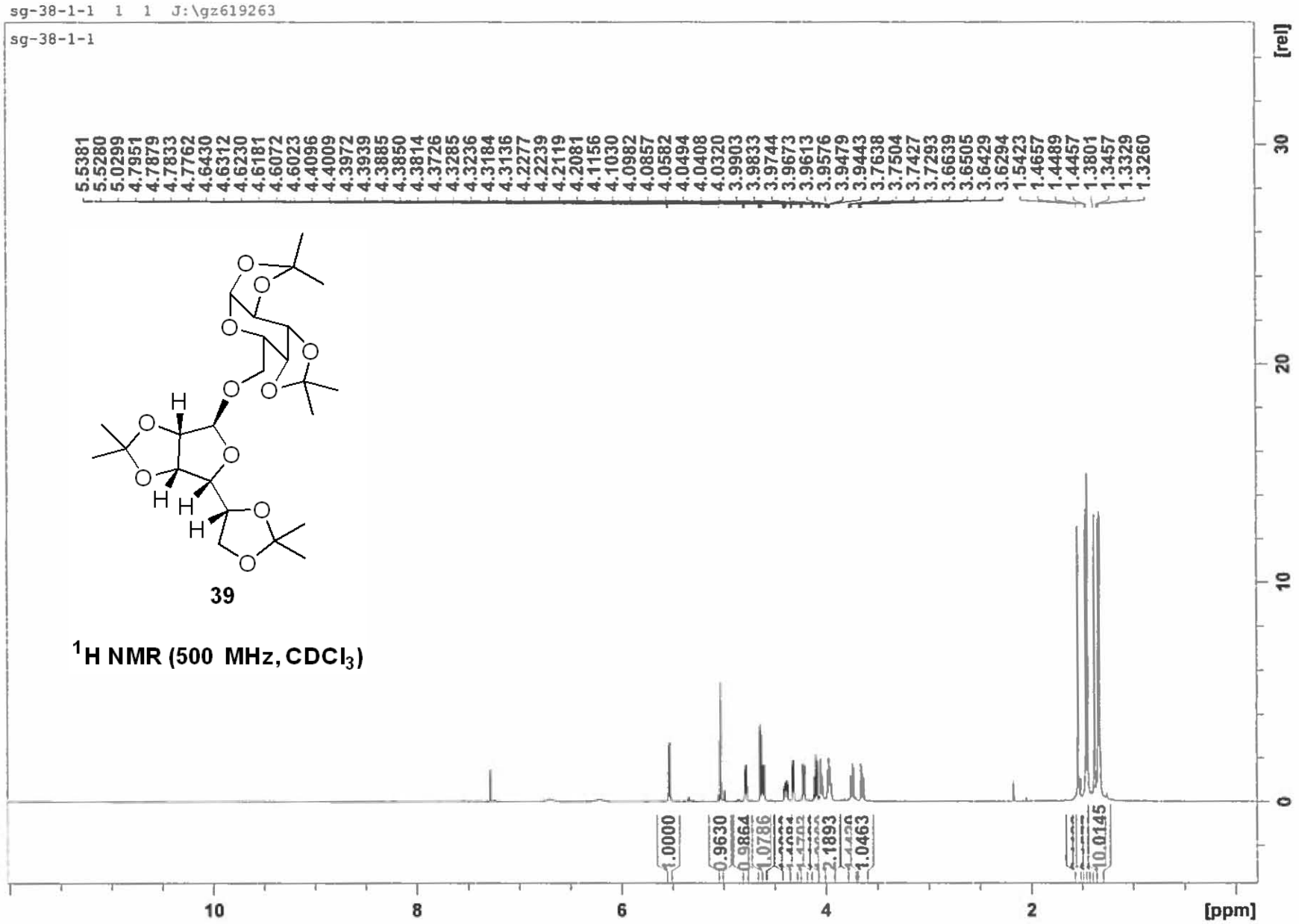
sgc-36-2-1 1 1 J:\gz619263

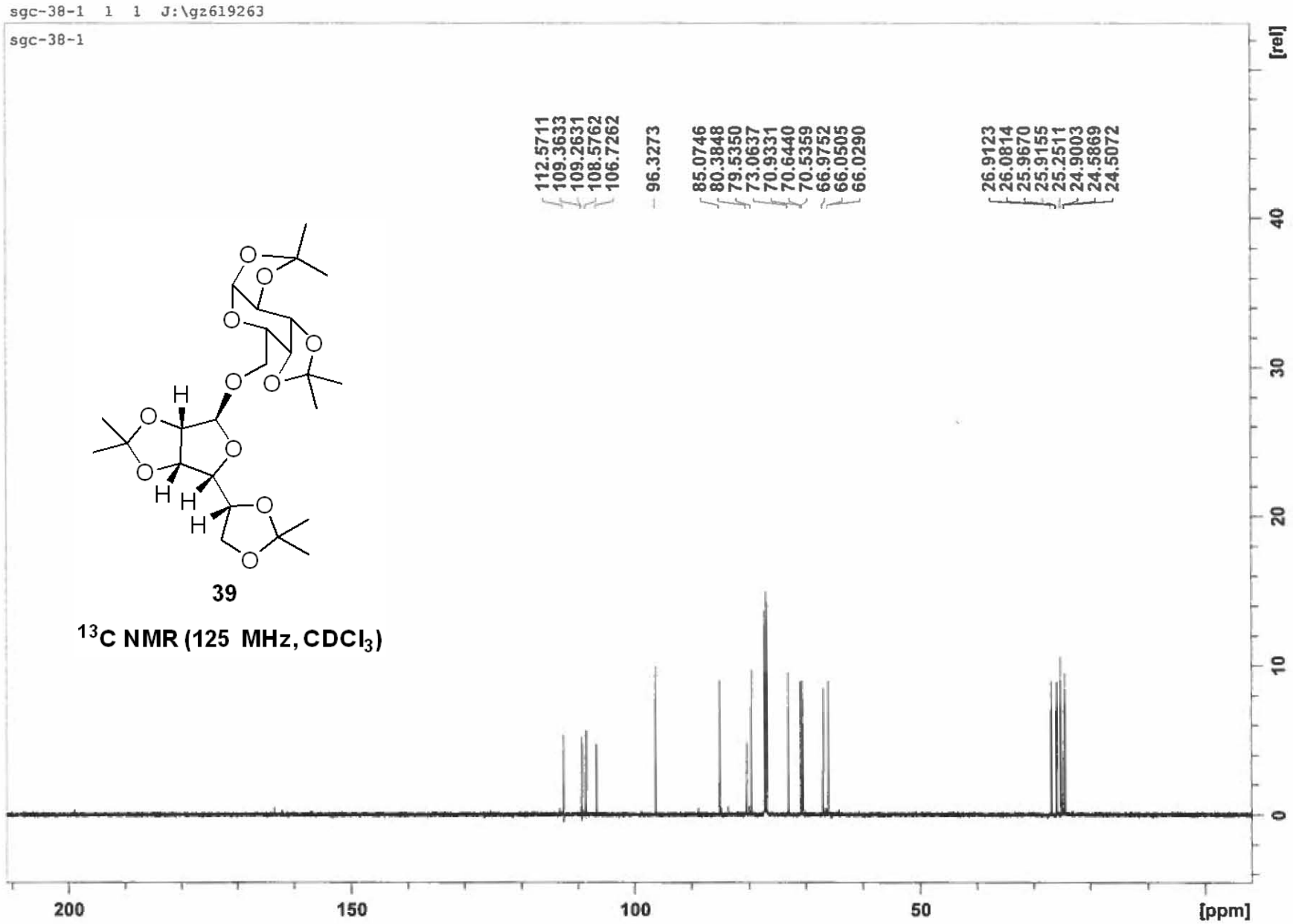
sgc-36-2-1



38β

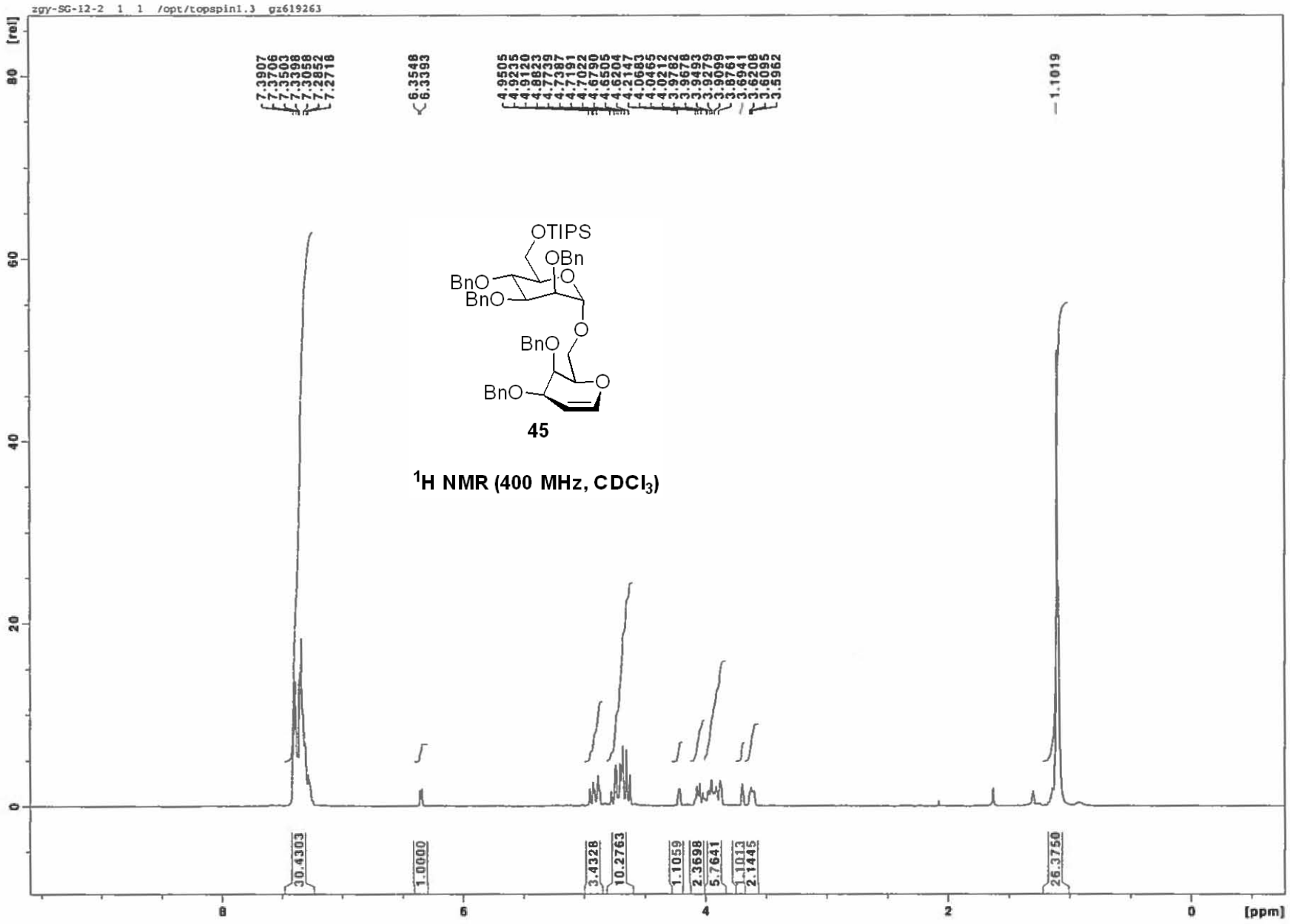
^{13}C NMR (125 MHz, $CDCl_3$)

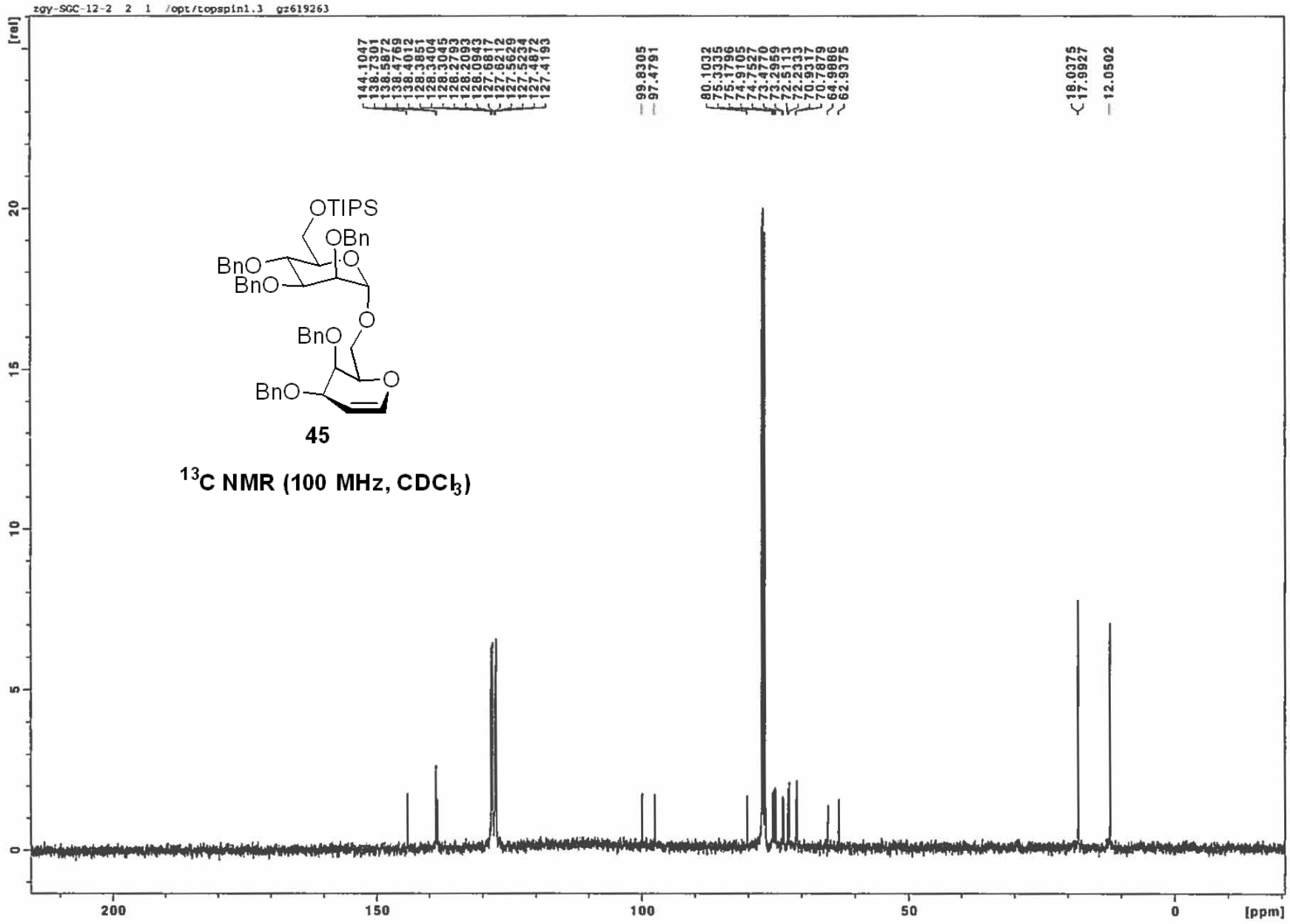

sg-38-1-1 1 1 J:\gz619263
sg-38-1-1
5.5381
5.5280
5.0299
4.7951
4.7879
4.7833
4.7762
4.6430
4.6312
4.6230
4.6181
4.6072
4.6023
4.4096
4.4009
4.3972
4.3939
4.3885
4.3850
4.3814
4.3726
4.3285
4.3236
4.3184
4.3136
4.2277
4.2239
4.2119
4.2081
4.1156
4.1030
4.0982
4.0857
4.0582
4.0494
4.0408
4.0320
3.9903
3.9833
3.9744
3.9673
3.9613
3.9576
3.9479
3.9443
3.7638
3.7504
3.7427
3.7293
3.6639
3.6505
3.6429
3.6294
1.5423
1.4657
1.4489
1.4457
1.3801
1.3457
1.3329
1.3260
39
1H NMR (500 MHz, CDCl3)
1.0000
0.9630
0.9864
1.0786
2.1893
1.0463
10.0145
10
8
6
4
2
[ppm]
[rel]
30
20
10
0

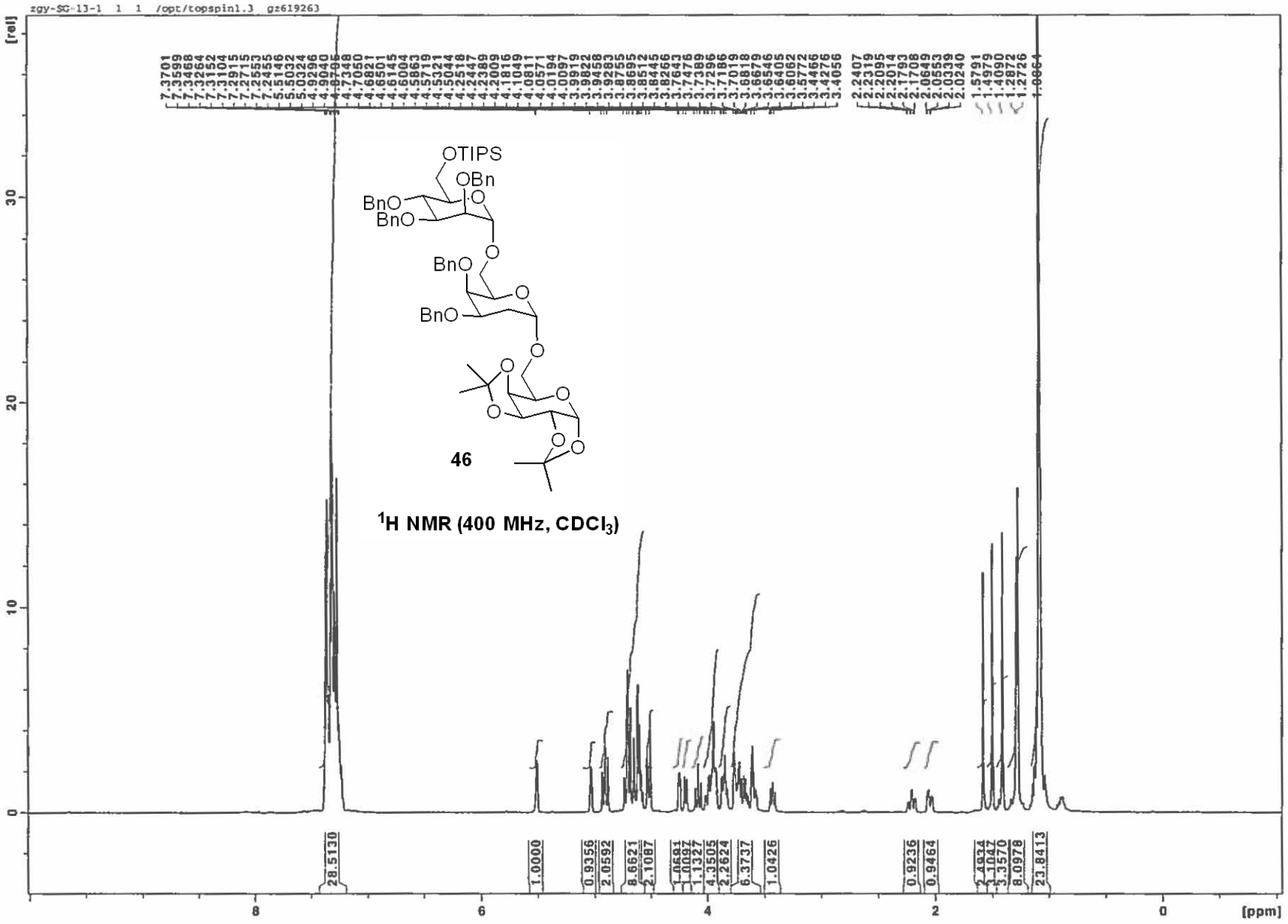


39

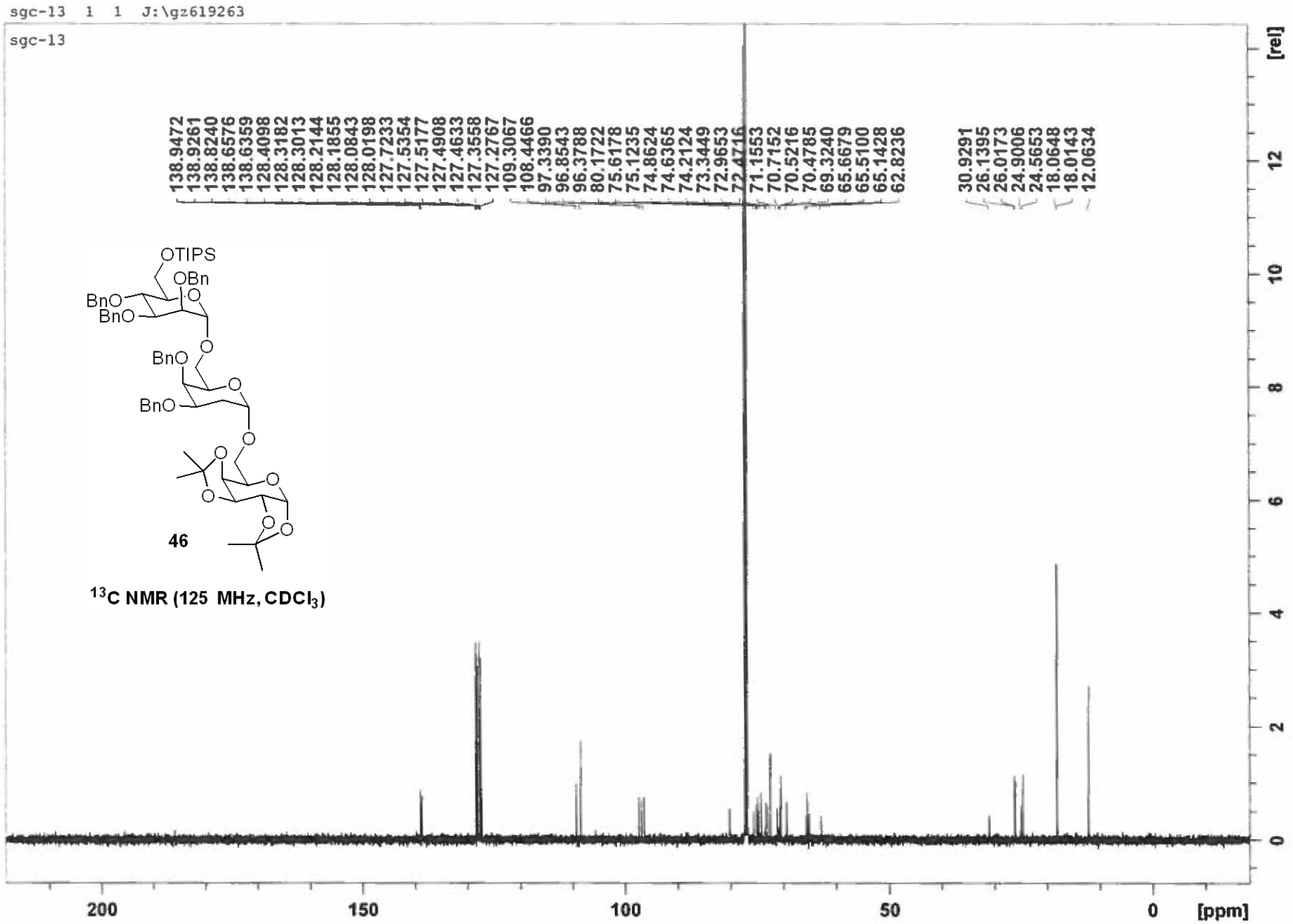
^{13}C NMR (125 MHz, $CDCl_3$)


zgy-SG-12-2 1 1 /opt/topspin1.3 gz619263
[rel]
7.3907
7.3706
7.3503
7.3398
7.3058
7.2852
7.2718
6.3548
6.3393
4.9505
4.9235
4.9120
4.8823
4.7739
4.7387
4.7191
4.7022
4.6790
4.6505
4.6204
4.2147
4.0683
4.0465
4.0212
3.9782
3.9678
3.9493
3.9279
3.9099
3.8761
3.6941
3.6208
3.6095
3.5962
1.1019
OTIPS
OBn
BnO
BnO
O
BnO
O
BnO
O
45
1H NMR (400 MHz, CDCl3)
30.4303
1.0000
3.4328
10.2763
1.1059
2.3698
5.7641
1.1013
2.1445
26.3750
[ppm]

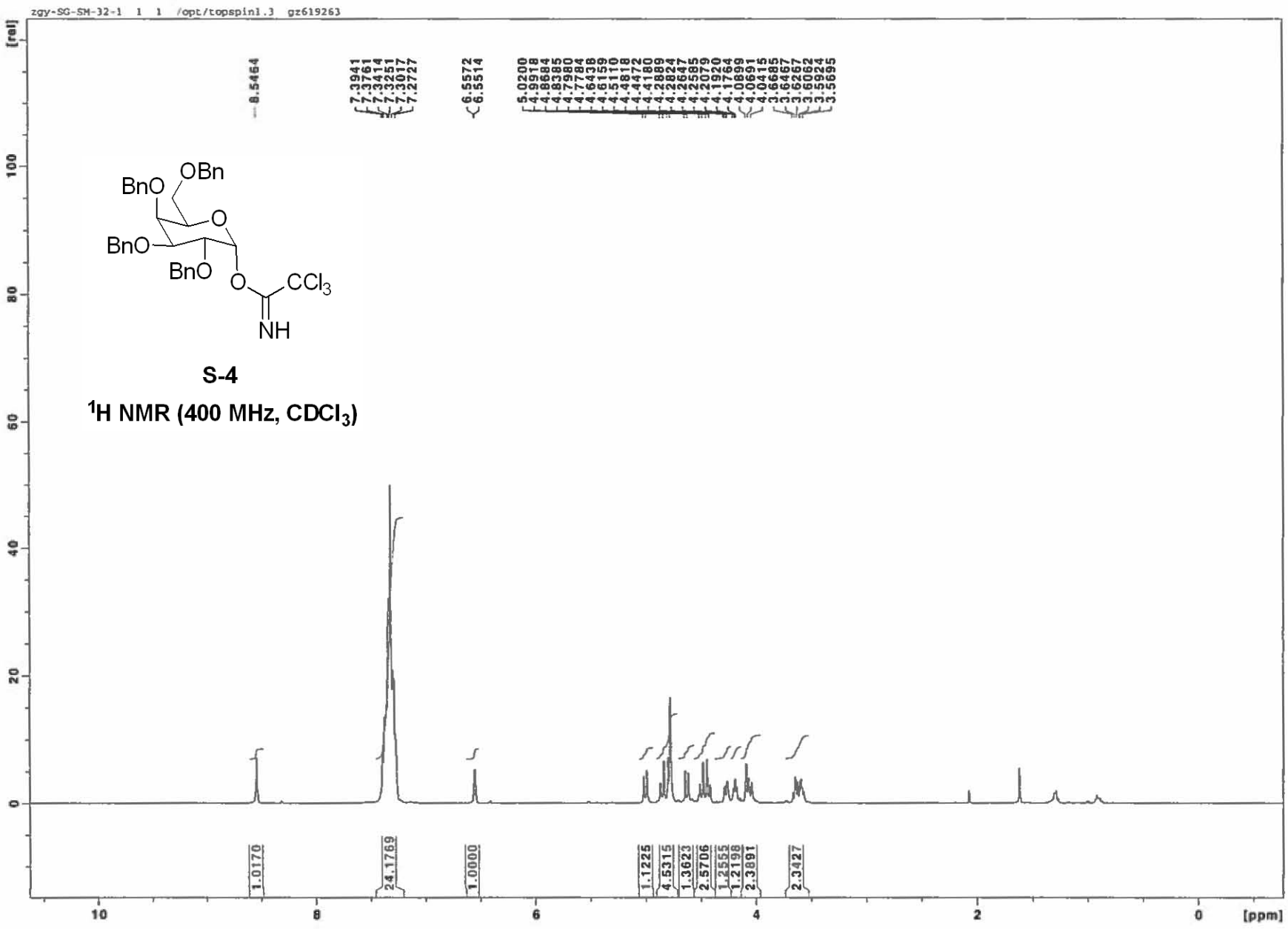

zgy-SGC-12-2 2 1 /opt/topspin1.3 gz619263
[rel]
144.1047
138.7301
138.5872
138.4769
138.4012
128.3851
128.3004
128.3045
128.2793
128.2093
128.0943
127.6817
127.6212
127.5629
127.5234
127.4872
127.4193
99.8305
97.4791
80.1032
75.3335
75.1796
74.9105
74.7527
74.4720
73.2959
72.5113
72.2333
70.9317
70.7879
64.9886
62.9375
18.0375
17.9927
12.0502
OTIPS
OBn
BnO
BnO
O
BnO
O
BnO
45
^{13}C NMR (100 MHz, CDCl$_3$)
20
15
10
5
0
200
150
100
50
0
[ppm]

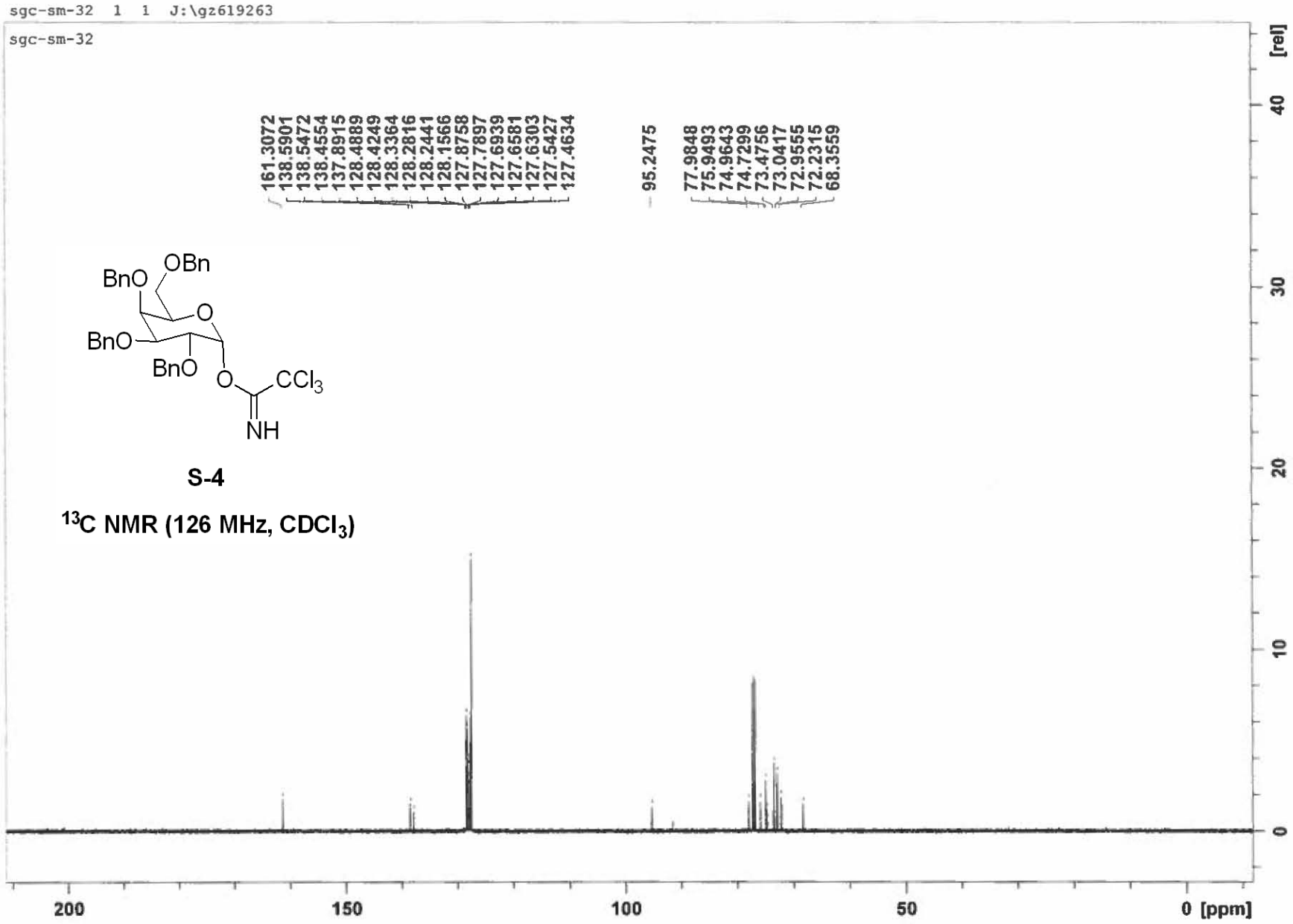


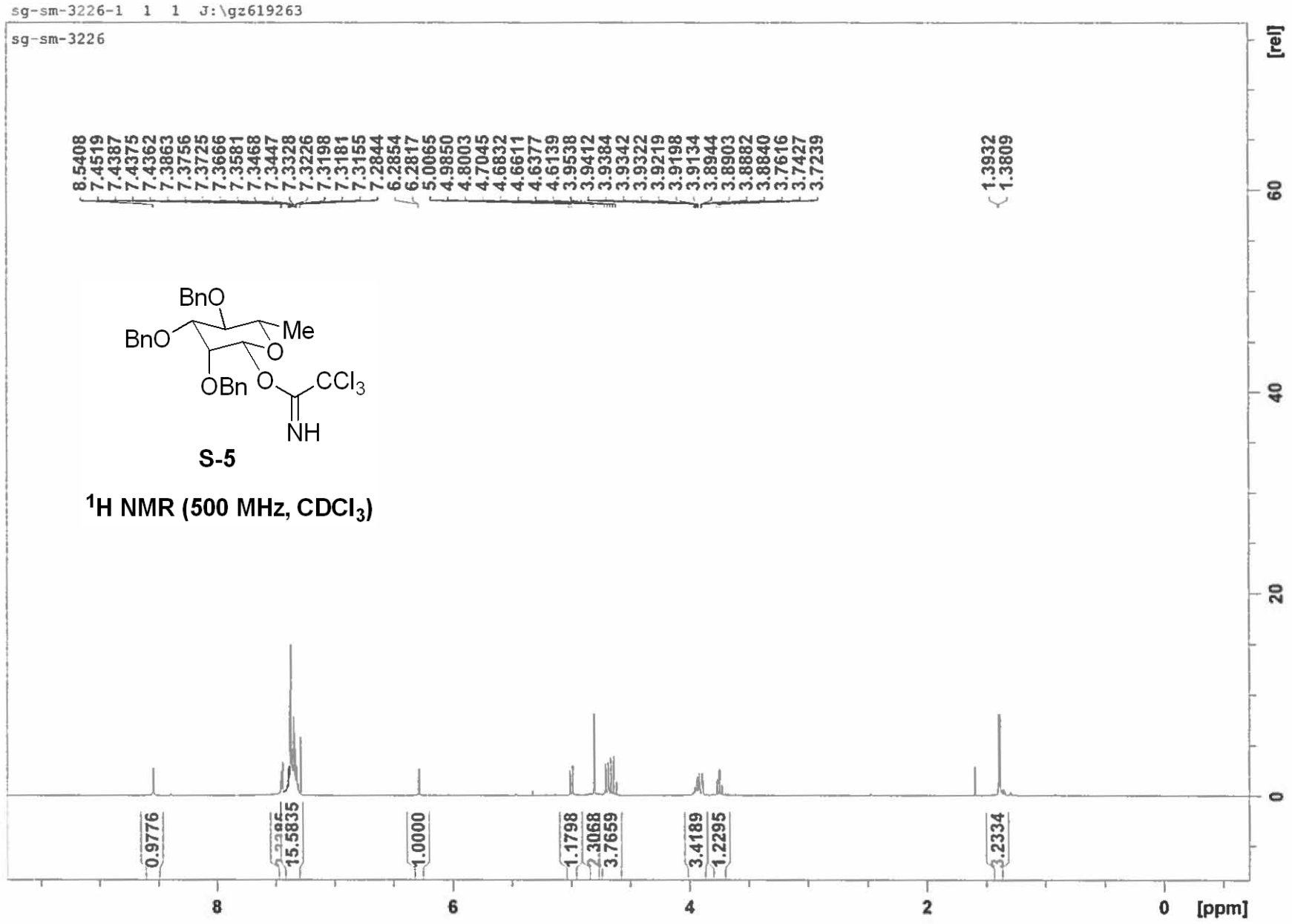
^{1}H NMR (400 MHz, $CDCl_3$)



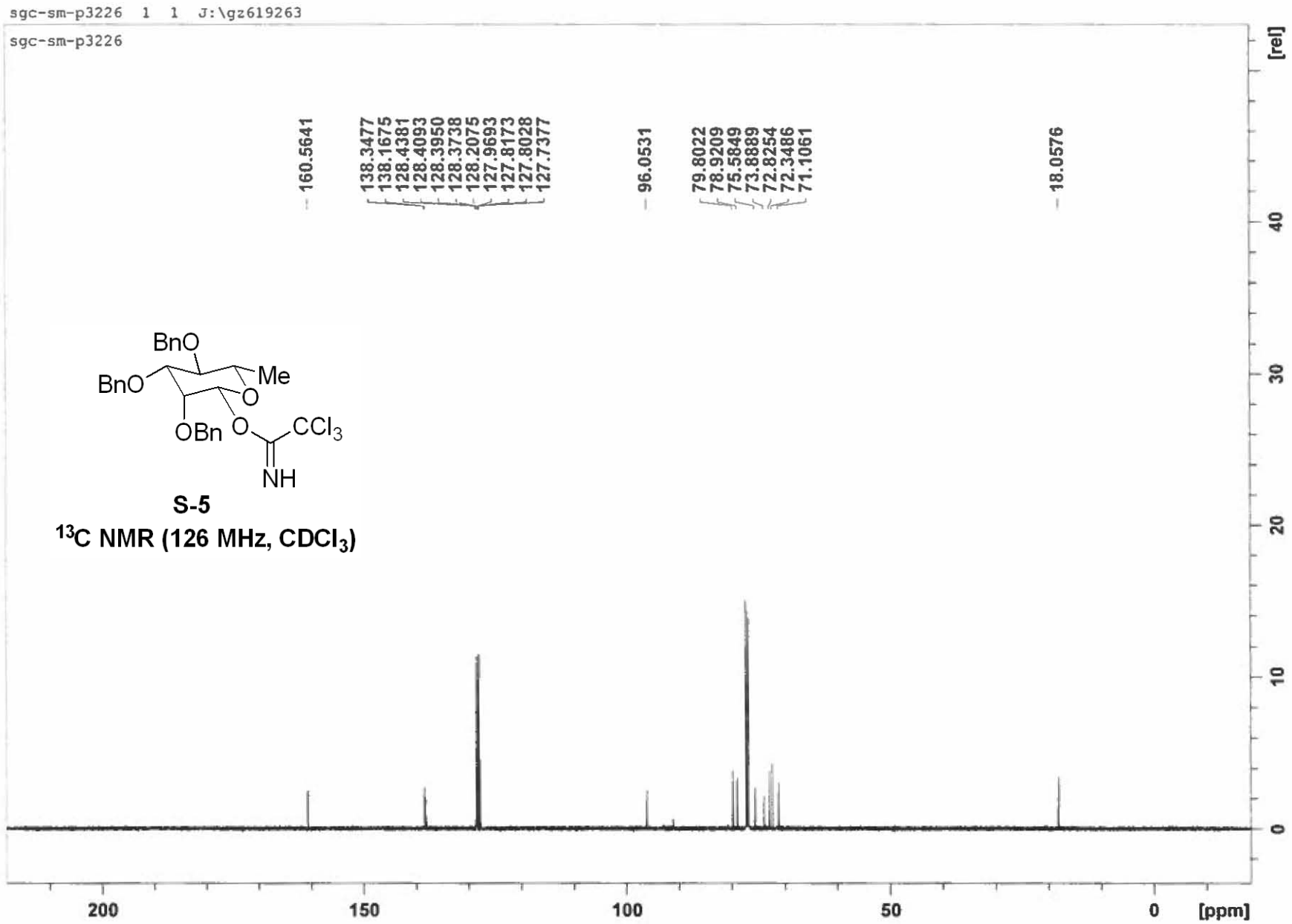
^{13}C NMR (125 MHz, $CDCl_3$)


zgy-SG-SM-32-1 1 1 /opt/topspin1.3 gz619263
[rel]
8.5464
7.3941
7.3761
7.3414
7.3251
7.3017
7.2727
6.5572
6.5514
5.0200
4.9918
4.8684
4.8385
4.7980
4.7784
4.6438
4.6159
4.5110
4.4818
4.4472
4.4180
4.2889
4.2824
4.2647
4.2585
4.2079
4.1920
4.1764
4.0899
4.0691
4.0415
3.6685
3.6467
3.6267
3.6062
3.5924
3.5695
BnO
OBn
O
BnO
BnO
O
CCl3
NH
S-4
1H NMR (400 MHz, CDCl3)
1.0170
24.1769
1.0000
1.1225
4.5315
1.3623
2.5706
1.2555
1.2198
2.3891
2.3427
[ppm]


sgc-sm-32 1 1 J:\gz619263
sgc-sm-32
161.3072
138.5901
138.5472
138.4554
137.8915
128.4889
128.4249
128.3364
128.2816
128.2441
128.1566
127.8758
127.7897
127.6939
127.6581
127.6303
127.5427
127.4634
95.2475
77.9848
75.9493
74.9643
74.7299
73.4756
73.0417
72.9555
72.2315
68.3559
BnO
OBn
O
BnO
BnO
O
CCl3
NH
S-4
13C NMR (126 MHz, CDCl3)
[rel]
40
30
20
10
0
200
150
100
50
0 [ppm]

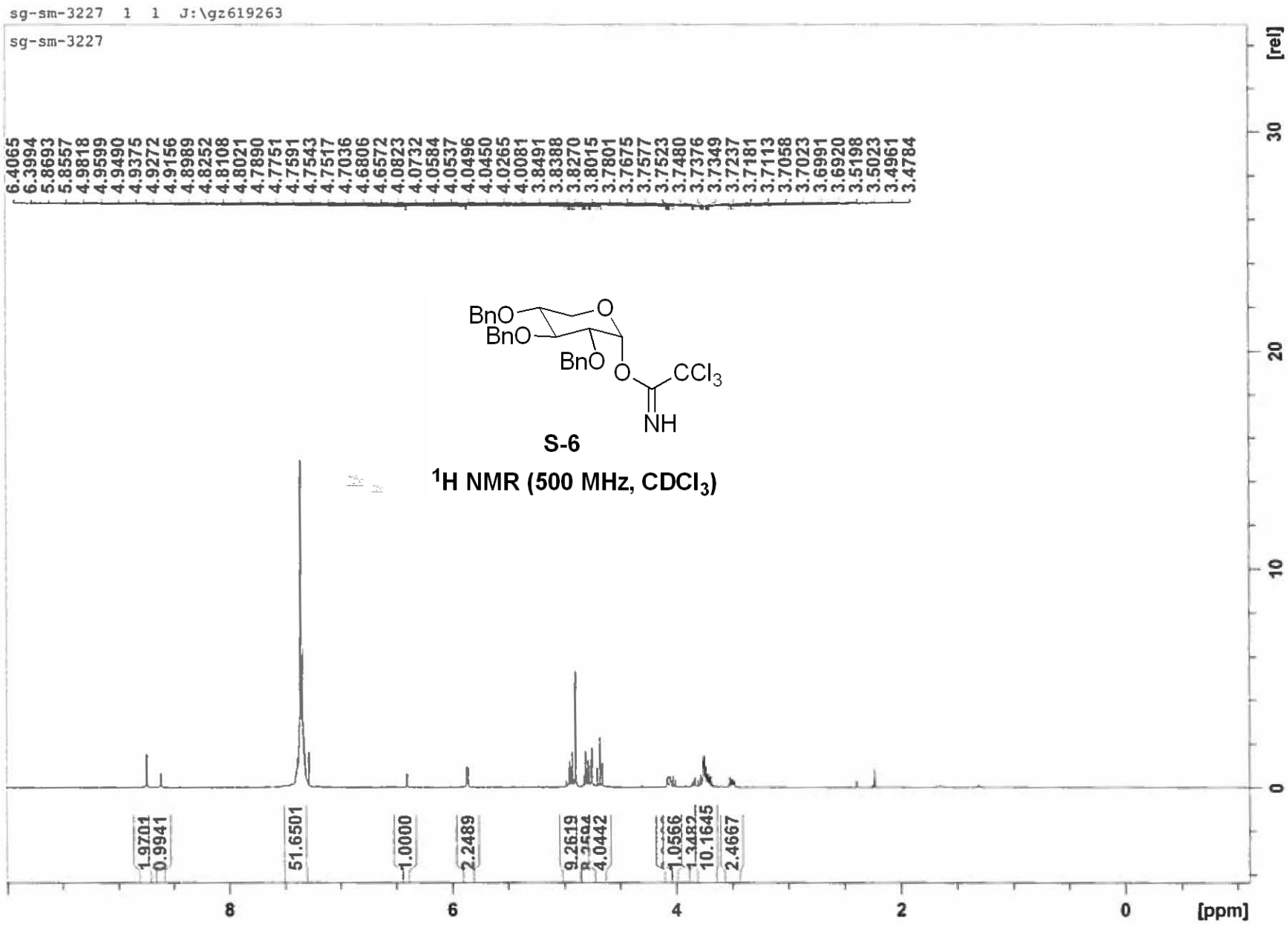


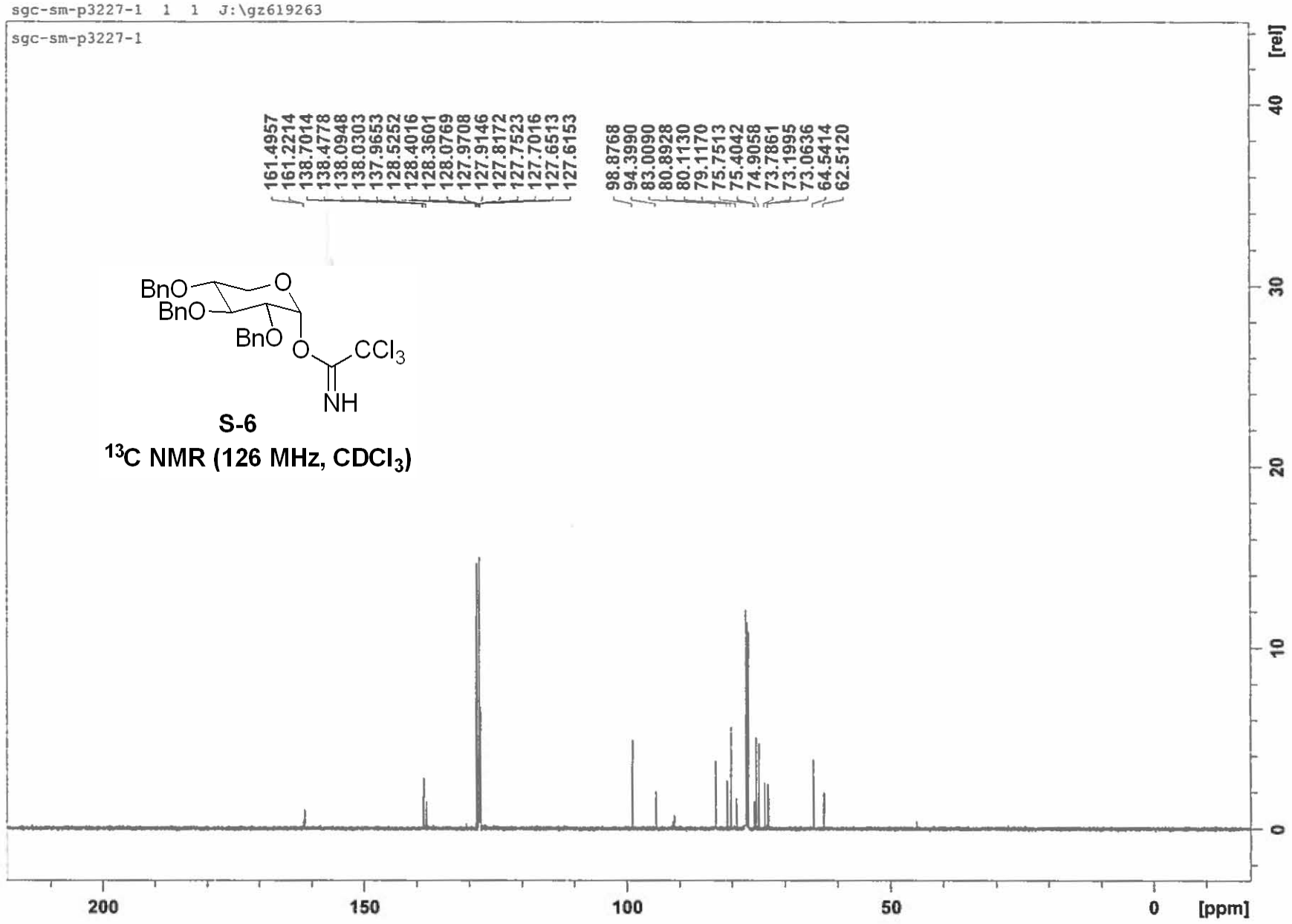
^{1}H NMR (500 MHz, $CDCl_3$)



S-5

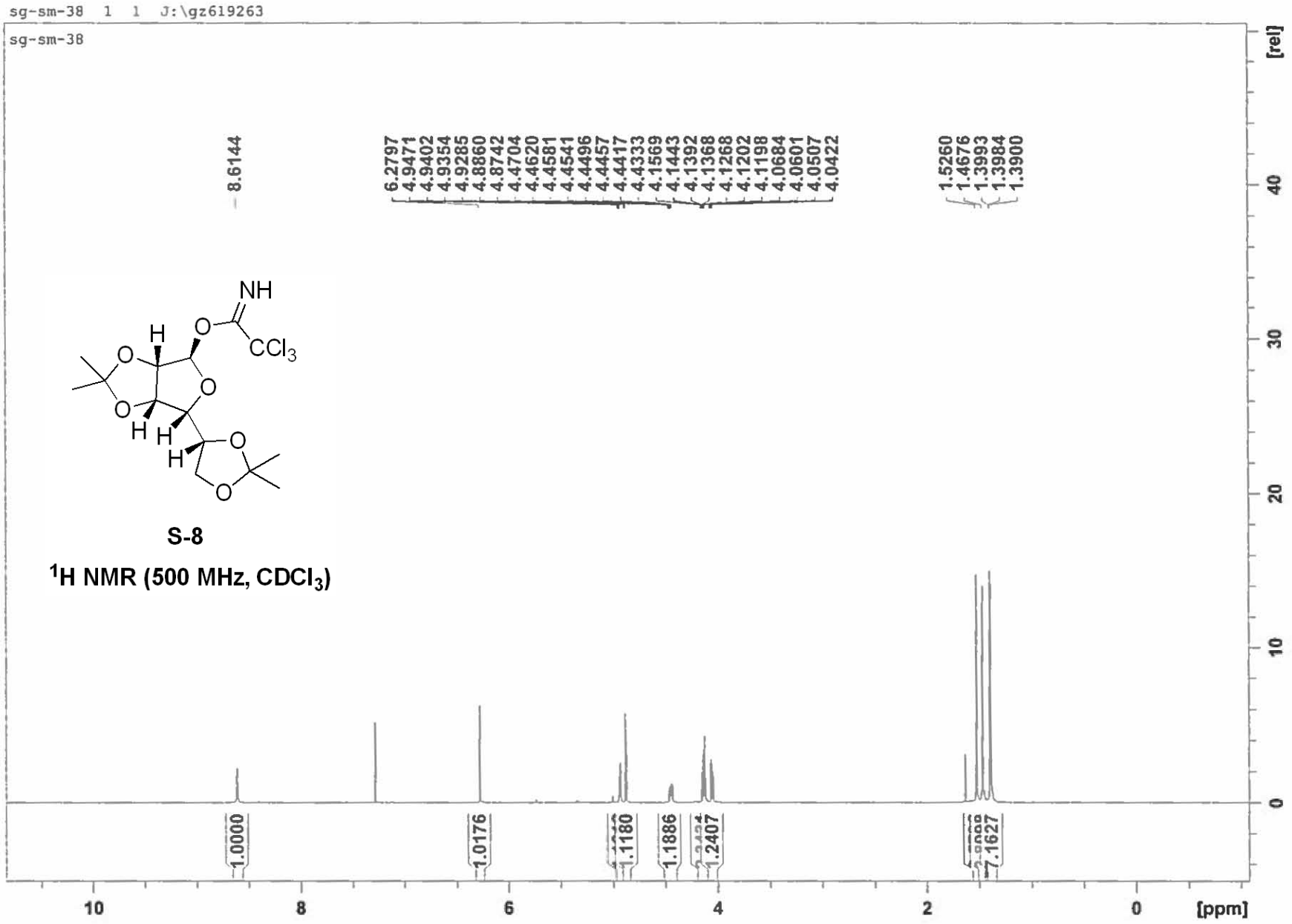
^{13}C NMR (126 MHz, $CDCl_3$)


sg-sm-3227 1 1 J:\gz619263
sg-sm-3227
6.4065
6.3994
5.8693
5.8557
4.9818
4.9599
4.9490
4.9375
4.9272
4.9156
4.8989
4.8252
4.8108
4.8021
4.7890
4.7751
4.7591
4.7543
4.7517
4.7036
4.6806
4.6572
4.0823
4.0732
4.0584
4.0537
4.0496
4.0450
4.0265
4.0081
3.8491
3.8388
3.8270
3.8015
3.7801
3.7675
3.7577
3.7523
3.7480
3.7376
3.7349
3.7237
3.7181
3.7113
3.7058
3.7023
3.6991
3.6920
3.5198
3.5023
3.4961
3.4784
BnO
BnO
BnO
O
O
CCl3
NH
S-6
1H NMR (500 MHz, CDCl3)
1.9701
0.9941
51.6501
1.0000
2.2489
9.2619
4.0442
1.0566
1.3482
10.1645
2.4667
8
6
4
2
0
[ppm]
[rel]
30
20
10
0



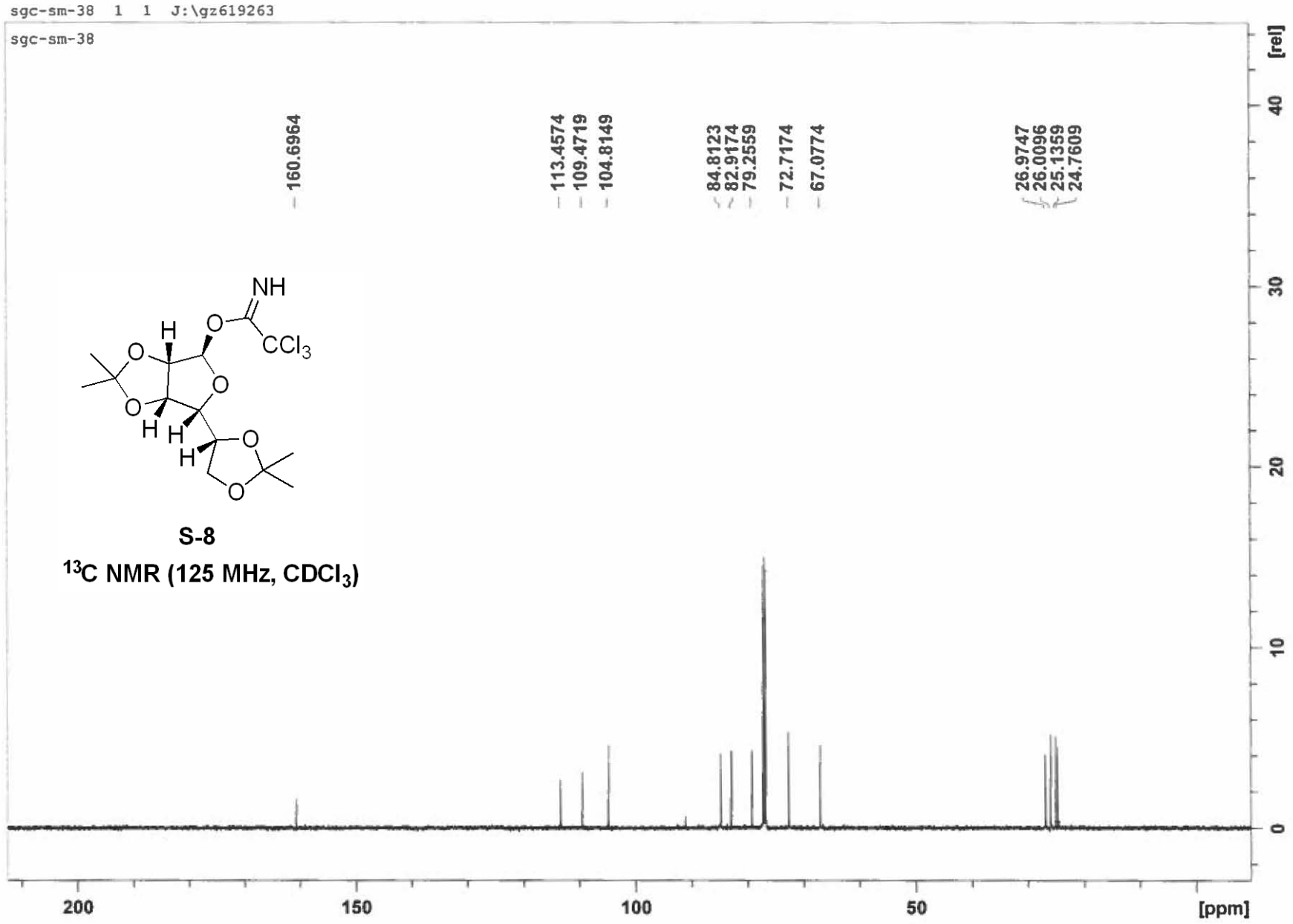
S-6

^{13}C NMR (126 MHz, $CDCl_3$)



S-8

^{1}H NMR (500 MHz, $CDCl_3$)



S-8

^{13}C NMR (125 MHz, $CDCl_3$)