Facile Synthesis of Carbohydrate-Integrated Isoxazolines through tandem [4 + 1] cycloaddition and rearrangement of 2 nitroglycals

Shuting Cai, Shaohua Xiang, Jing Zeng, Bala Kishan Gorityala and Xue-Wei Liu*

Division of Chemistry and Biological Chemistry, School of Physical and Mathematical Sciences, Nanyang Technological University, Singapore 637371
E-mail: xuewei@ntu.edu.sg

Supporting Information

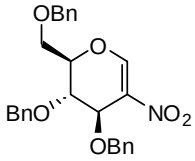
Table of Contents

General experimental details

All reactions were conducted under an atmosphere of nitrogen, unless otherwise indicated. Anhydrous solvents were transferred *via* oven-dried syringe. Flasks were flame-dried and cooled under a stream of nitrogen. All reagents and solvents were obtained from commercial suppliers (Sigma-Aldrich, Fluka and Alfa Aesar) and used without further purification unless otherwise stated. Evaporation of organic solutions was achieved by rotary evaporation with a water bath temperature below 40 °C. Product purification by flash column chromatography was accomplished using silica gel 60 (0.010 - 0.063 mm). Technical grade solvents were used for chromatography and distilled prior to use. Chromatograms were visualized by fluorescence quenching with UV light at 254 nm or by staining using a basic solution of potassium permanganate. Optical rotations were measured in $CHCl_3$ on a Schmidt + Haensdch polarimeter with a 1 cm cell (*c* given in g/100 mL). IR spectra were recorded using FTIR Restige-21 (Shimadzu) and reported in cm^{-1}. High-resolution mass spectra (HRMS) were obtained on a Finnigan/MAT LCQ quadrupole ion trap mass spectrometer, coupled with the TSP4000 HPLC system and the Crystal 310 CE system. Accurate masses are reported for the molecular ion $[M+H]^+$ or a suitable fragment ion. NMR spectra were recorded at room temperature on a 400 MHz Bruker ACF 400 NMR spectrometer. The residual solvent signals were taken as the reference (7.26 ppm for ^1H NMR spectroscopy and 77.0 ppm for ^{13}C NMR spectroscopy). Chemical shifts are reported in delta (δ) units, parts per million (ppm) downfield from triethylsilane. Chemical shift (δ) is referred in terms of ppm, coupling constants (J) are given in Hz. Following abbreviations classify the multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved. X-ray crystallographic data was collected by using a Bruker X8Apex diffractometer with Mo K/α radiation (graphite monochromator). Compound numbers used in the experimental section correspond to those employed in the main paper.

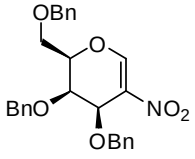
Preparation of starting material 3,4,6-tri-O-benzyl-2-nitroglycals (1)

The starting 3,4,6-tri-*O*-benzyl-2-nitroglycals (**1**) were prepared according to the known procedure.[1] Experimental results obtained were in accordance with the reported reference.



1a

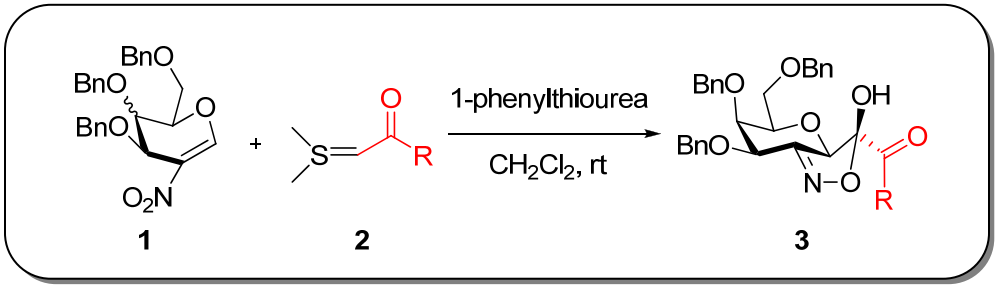
The product was obtained as a pale yellow solid; (1.12 g, 56 % yield); **m.p.** 53-56 °C; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.21 (s, 1H), 7.35-7.22 (m, 15H), 4.68-4.44 (m, 8H), 3.87 (dd, J_1 = 3.2 Hz, J_2 = 2.4 Hz, 1H), 3.72 (dd, J_1 = 10.1 Hz, J_2 = 14.2 Hz, 1H), 3.60 (dd, J_1 = 7.1 Hz, J_2 = 14.2 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)**: δ 154.7, 137.6, 137.0, 128.9, 128.7, 128.5, 128.4, 128.3, 128.1, 128.0, 127.9, 78.6, 73.6, 73.2, 72.0, 71.4, 68.0, 67.7; **IR** (KBr): 3007, 2918, 2870, 1719, 1599, 1557, 1362, 1096 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{27}H_{28}NO_6$ $[M+H]^+$, 462.1917, found: 462.1916.



1b

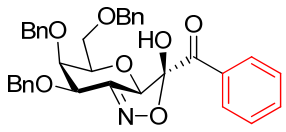
The product was obtained as a yellow solid; (1.10 g, 57 % yield); **^{1}H NMR (400 MHz, $CDCl_3$)**: δ 8.10 (s, 1H), 7.41-7.26 (m, 15H), 4.91-4.46 (m, 8H), 3.97-3.91 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 154.6, 138.1, 137.7, 136.9, 131.6, 128.7, 128.5, 128.4, 128.3, 128.0, 127.9, 127.8, 127.7, 78.1, 77.4, 77.1, 76.8, 75.0, 73.5, 73.1, 72.2, 67.6; **IR** (KBr): 3013, 2922, 2870, 1748, 1636, 1558, 1504, 1344, 1323, 1202, 1094 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{27}H_{28}NO_6$ $[M+H]^+$, 462.1920, found: 462.1917.

General synthetic procedure for carbohydrate-integrated isoxazolines (3)



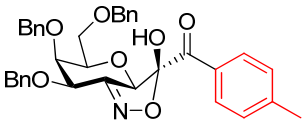
To a solution of 3,4,6-tri-*O*-benzyl-2-nitrogalactal **1a** (50 mg, 0.11 mmol) and 1-phenylthiourea (2 mg, 0.01 mmol) in CH_2Cl_2 (2 mL) was added sulphur ylide **2a** (29 mg, 0.17 mmol). The reaction mixture was stirred at rt for 5.5 h. The resulting mixture was extracted with ether (2 × 50 mL), washed with 10% $NaHCO_3$ (2 × 50 mL) and brine (2 × 50 mL). The organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure to yield the crude residue as a dark yellow oil. The crude residue was then purified by flash column chromatography on silica gel (20% EtOAc in hexanes) to afford compound **3a** (54 mg, 0.09 mmol, 85% yield of diastereomer mixture) as a colourless oil.

Characterization of the carbohydrate-integrated isoxazolines (3)



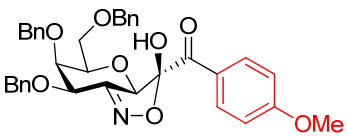
3a

The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (50 mg, 80% yield of diastereomer mixture, 91% *de*); **[α]$^{24}_{D}$** -174.0 (*c* 0.02, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.28 (d, *J* = 7.4 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.41-7.28 (m, 15H), 5.59 (s, 1H), 5.47 (s, 1H), 5.06 (dd, *J* = 11.9, 3.9 Hz, 2H), 4.69 (d, *J* = 11.9 Hz, 1H), 4.61 (d, *J* = 11.3 Hz, 1H), 4.56-4.55 (m, 1H), 4.42 (dd, *J* = 19.5, 11.9 Hz, 2H), 4.19-4.18 (m, 1H), 3.97 (t, *J* = 6.5 Hz, 1H), 3.59 (d, *J* = 6.5 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 189.6, 156.1, 137.8, 137.5, 137.2, 133.8, 132.8, 131.0, 128.6, 128.6, 128.5, 128.4(2), 128.3, 128.1, 128.0, 127.9(2), 127.7, 105.9, 81.1, 77.8, 76.6, 75.1, 75.0, 73.6, 72.4, 67.8; **IR (neat)** 3424, 3030, 2922, 2872, 1688, 1645, 1597 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{35}H_{33}NO_7$ $[M+H]^+$, 580.2257, found 580.2335.



3b

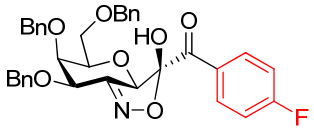
The title compound was prepared according to the general procedure and the compound was obtained as a white solid (50 mg, 78% yield of diastereomer mixture, 92% *de*); **m.p.** 144-145 °C; **[α]$^{24}_{D}$** -164.7 (*c* 0.2, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.18 (d, *J* = 8.2 Hz, 2H), 7.41-7.25 (m, 17H), 5.59 (s, 1H), 5.38 (s, 1H), 5.06 (dd, *J* = 11.9, 4.6 Hz, 2H), 4.69 (d, *J* = 11.9 Hz, 1H), 4.61 (d, *J* = 11.3 Hz, 1H), 4.56-4.55 (m, 1H), 4.41 (dd, *J* = 19.4, 11.9 Hz, 2H), 4.18-4.17 (m, 1H), 3.96 (t, *J* = 6.5 Hz, 1H), 3.59 (d, *J* = 6.5 Hz, 2H), 2.42 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 189.2, 156.2, 144.8, 137.8, 137.5, 137.2, 131.2, 130.2, 129.1, 128.6, 128.5(2), 128.4, 128.1, 128.0(2), 127.9(2), 127.7, 106.0, 81.2, 77.8, 76.6, 75.1, 75.0, 73.6, 72.4, 67.8, 21.8; **IR (neat)** 3584, 3030, 2920, 2870, 1690, 1607 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{36}H_{35}NO_7$ $[M+H]^+$, 594.2414, found 594.2492.



3c

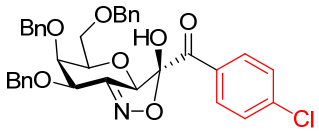
The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (54 mg, 81% yield of diastereomer mixture, 90% *de*); **[α]$^{24}_{D}$** -201.8 (*c* 0.1, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.27 (d, *J* = 9.0 Hz,

2H), 7.41-7.25 (m, 15H), 7.14 (d, *J* = 9.0 Hz, 2H), 5.59 (s, 1H), 5.35 (s, 1H), 5.08-5.04 (m, 2H), 4.69 (d, *J* = 11.9 Hz, 1H), 4.61 (d, *J* = 11.3 Hz, 1H), 4.55-4.54 (m, 1H), 4.41 (dd, *J* = 19.5, 11.9 Hz, 2H), 4.19-4.18 (m, 1H), 3.96 (t, *J* = 6.5 Hz, 1H), 3.88 (s, 3H), 3.59 (d, *J* = 6.5 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** *δ* 188.1 156.1, 137.8, 137.4, 137.2, 133.5, 128.7, 128.6, 128.5(2), 128.4, 128.1, 128.0, 127.8(2), 127.7(2), 81.1, 77.7, 76.5, 75.1, 75.0, 73.6, 72.4, 67.7, 55.5; **IR (neat)** 3418, 3030, 2926, 2874, 1682, 1599, 1090 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{36}H_{33}NO_8$ $[M+H]^+$, 610.2363, found 610.2441.



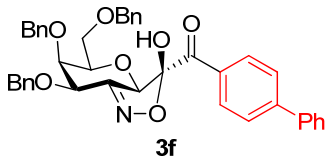
3d

The title compound was prepared according to the general procedure and the compound was obtained as a pale yellow oil (50 mg, 78% yield of diastereomer mixture, 90% *de*); **$[\alpha]^{24}_D$** -202.3 (*c* 0.6, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** *δ* 8.34-8.30 (m, 2H), 7.41-7.25 (m, 15H), 7.14 (t, *J* = 8.6 Hz, 2H), 5.59 (s, 1H), 5.48 (s, 1H), 5.08-5.05 (m, 2H), 4.69 (d, *J* = 11.8 Hz, 1H), 4.61 (d, *J* = 11.2 Hz, 1H), 4.56-4.55 (m, 1H), 4.42 (dd, *J* = 19.5, 11.8 Hz, 2H), 4.19-4.18 (m, 1H), 3.97 (t, *J* = 6.5 Hz, 1H), 3.59 (d, *J* = 6.5 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** *δ* 188.0, 156.3, 137.9, 137.5, 137.2, 134.0, 133.9, 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 127.8, 115.6, 115.4, 105.8, 81.1, 77.9, 77.2, 75.1, 75.0, 73.6, 72.5, 67.8; **IR (neat)** 3582, 3030, 2924, 2855, 1688, 1599, 1364 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{35}H_{32}FNO_7$ $[M+H]^+$, 598.2163, found 598.2241.



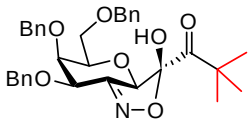
3e

The title compound was prepared according to the general procedure and the compound was obtained as a yellow oil (53 mg, 80% yield of diastereomer mixture, 94% *de*); **$[\alpha]^{24}_D$** -165.6 (*c* 0.1, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** *δ* 8.22 (d, *J* = 8.7 Hz, 2H), 7.45-7.27 (m, 17H), 5.71 (s, 1H), 5.57 (s, 1H), 5.06 (dd, *J* = 11.9, 4.2 Hz, 2H), 4.69 (d, *J* = 11.9 Hz, 1H), 4.60 (d, *J* = 11.1 Hz, 1H), 4.56-4.55 (m, 1H), 4.42 (dd, *J* = 20.1, 11.9 Hz, 2H), 4.20-4.19 (m, 1H), 3.97 (t, *J* = 6.5 Hz, 1H), 3.59 (d, *J* = 6.5 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** *δ* 188.4, 156.2, 140.4, 137.7, 137.4, 137.1, 132.8, 132.4, 131.1, 129.0, 128.6(2), 128.5, 128.4, 128.1, 128.0, 127.9, 127.8, 105.8, 81.0, 77.8, 76.5, 75.1, 73.5, 72.4, 67.7; **IR (neat)** 3422, 3030, 2924, 2868, 1597, 735 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{35}H_{32}ClNO_7$ $[M+H]^+$, 614.1867, found 614.1946.



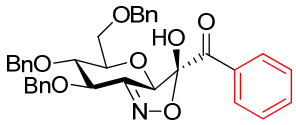
3f

The title compound was prepared according to the general procedure and the compound was obtained as a white solid (59 mg, 83% yield of diastereomer mixture, 90% de); **m.p.** 156-157 °C; **$[\alpha]^{24}_D$** -233.7 (*c* 0.1, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.35 (d, *J* = 8.6 Hz, 2H), 7.69 (d, *J* = 8.6 Hz, 2H), 7.64 (d, *J* = 7.1 Hz, 2H), 7.47 (t, *J* = 7.1 Hz, 2H), 7.42-7.25 (m, 16H), 5.62 (s, 1H), 5.28 (s, 1H), 5.07 (dd, *J* = 11.9, 2.9 Hz, 2H), 4.69 (d, *J* = 11.9 Hz, 1H), 4.62 (d, *J* = 11.4 Hz, 1H), 4.57-4.56 (m, 1H), 4.41 (dd, *J* = 19.2, 11.9 Hz, 2H), 4.19-4.18 (m, 1H), 3.98 (t, *J* = 6.4 Hz, 1H), 3.59 (d, *J* = 6.4 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 189.2, 156.1, 146.4, 140.0, 137.7, 137.4, 137.2, 132.1, 131.6, 131.5, 131.3, 128.9, 128.8, 128.5(2), 128.4, 128.2, 128.1, 128.0, 127.9, 127.8(2), 127.7(2), 127.3, 127.0, 106.0, 81.0, 77.8, 76.5, 75.1(2), 73.6, 72.4, 67.7; **IR (neat)** 3387, 3030, 2924, 2870, 1682, 1603 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{41}H_{37}NO_7$ $[M+H]^+$, 656.2570, found 656.2648.



3g

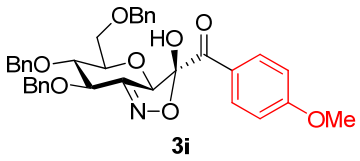
The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (41 mg, 68% yield of diastereomer mixture, 95% *de*); **$[\alpha]^{24}_D$** -224.1 (*c* 0.2, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.38-7.24 (m, 19H), 5.33 (s, 1H), 5.22 (s, 1H), 5.06 (d, *J* = 11.8, 1H), 5.02 (d, *J* = 11.2, 1H), 4.66 (d, *J* = 11.8 Hz, 1H), 4.57 (d, *J* = 11.2 Hz, 1H), 4.52-4.51 (m, 1H), 4.41 (dd, *J* = 16.2, 11.8 Hz, 2H), 4.14-4.13 (m, 1H), 3.90 (t, *J* = 6.5 Hz, 1H), 3.56 (d, *J* = 6.5 Hz, 2H), 1.33 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 205.5, 155.5, 137.9, 137.5, 137.3, 128.5(2), 128.4, 128.3, 128.0(3), 127.9(2), 127.8, 127.3, 127.2, 106.1, 81.8, 77.7, 77.2, 75.1, 73.6, 72.4, 67.8, 53.4, 43.8, 29.7, 27.7, 27.1; **IR (neat)** 3406, 3030, 2959, 2930, 2872, 1713, 1557, 1250 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{33}H_{37}NO_7$ $[M+H]^+$, 560.2570, found 560.2648.



3h

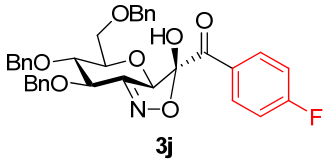
The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (52 mg, 83% yield of diastereomer mixture, 90% *de*); **$[\alpha]^{24}_D$** -234.2 (*c* 0.2, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.26 (d, *J* = 7.4 Hz, 2H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.41-7.19 (m, 15H), 5.60 (s, 1H), 5.57 (s, 1H), 5.05 (d, *J* = 11.3 Hz, 1H), 4.65 (d, *J* = 10.8 Hz, 1H), 4.69 (d, *J* = 11.3 Hz, 1H), 4.62-4.59 (m, 2H), 4.49 (dd, *J* = 19.7, 11.9 Hz, 2H), 3.88-3.68 (m,

4H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 189.5, 156.3, 137.6, 137.4, 137.4, 137.2, 133.9, 132.5, 131.0, 128.6, 128.5(2), 128.4, 128.3, 128.1(2), 128.0(2), 127.8, 127.7, 106.1, 80.8, 78.6, 78.3, 75.5, 73.5, 73.1, 68.2; **IR (neat)** 3443, 3030, 2918, 2872, 1694, 1682, 1599 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{35}H_{33}NO_7$ $[M+H]^+$, 580.2257, found 580.2335.



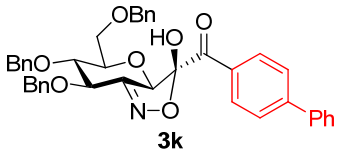
3i

The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (54 mg, 81% yield of diastereomer mixture, 93% *de*); **$[\alpha]^{24}_D$** -243.4 (*c* 0.5, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.25 (d, *J* = 9.0 Hz, 2H), 7.41-7.19 (m, 15H), 6.95 (d, *J* = 9.0 Hz, 2H), 5.61 (s, 1H), 5.50 (s, 1H), 5.05 (d, *J* = 11.4 Hz, 1H), 4.94 (d, *J* = 10.8 Hz, 1H), 4.68 (d, *J* = 11.4 Hz, 1H), 4.61-4.57 (m, 2H), 4.51 (dd, *J* = 21.0, 12.0 Hz, 2H), 4.03-3.66 (m, 4H), 3.87 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 188.1, 164.2, 156.3, 137.7, 137.4, 137.3, 133.5, 128.5(3), 128.3, 128.1(2), 128.0, 127.9, 125.4, 113.7, 106.4, 80.9, 78.6, 78.5, 78.3, 76.7, 75.5, 73.5, 73.1, 68.2, 55.5; **IR (neat)** 3418, 3030, 2934, 2872, 1682, 1599, 1098 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{36}H_{33}NO_8$ $[M+H]^+$, 610.2363, found 610.2441.



3j

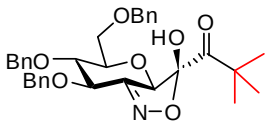
The title compound was prepared according to the general procedure and the compound was obtained as a pale yellow oil (52 mg, 81% yield of diastereomer mixture, 93% *de*); **$[\alpha]^{24}_D$** -155.4 (*c* 0.5, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.33-8.29 (m, 2H), 7.40-7.13 (m, 17H), 5.60 (s, 1H), 5.51 (s, 1H), 5.04 (d, *J* = 11.4 Hz, 1H), 4.94 (d, *J* = 10.8 Hz, 1H), 4.68 (d, *J* = 11.4 Hz, 1H), 4.61-4.58 (m, 2H), 4.48 (dd, *J* = 19.5, 11.90 Hz, 2H), 3.95-3.72 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 188.0, 165.0, 156.4, 137.6, 137.3, 137.2, 134.0, 133.9, 129.0, 128.6, 128.5(2), 128.3, 128.1(2), 128.0, 127.9, 115.7, 115.5, 106.3, 80.6, 78.6, 78.5, 78.2, 75.6, 73.5, 73.1, 68.2; **IR (neat)** 3383, 3030, 2957, 2922, 2870, 1599, 1099 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{35}H_{32}FNO_7$ $[M+H]^+$, 598.2163, found 598.2241.



3k

The title compound was prepared according to the general procedure and the compound was obtained as a colourless oil (56 mg, 79% yield of diastereomer mixture, 92% *de*); **$[\alpha]^{24}_D$** -173.0 (*c* 0.1, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.34 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.62 (d, *J* =

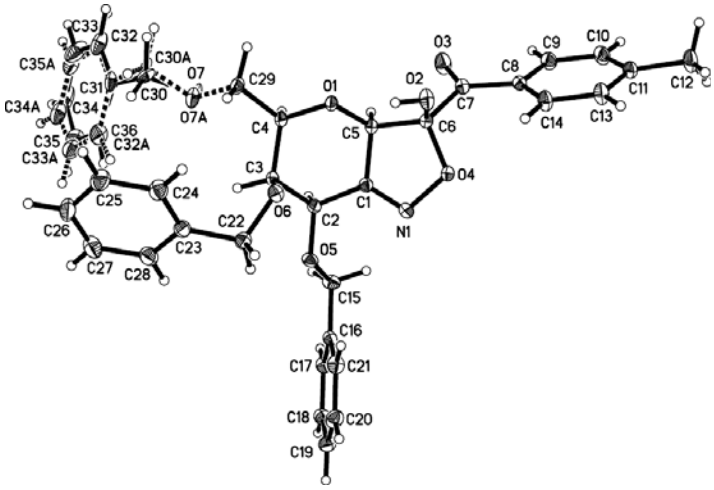
7.3 Hz, 2H), 7.46 (t, *J* = 7.3 Hz, 2H), 7.41-7.19 (m, 16H), 5.63 (s, 1H), 5.52 (s, 1H), 5.05 (d, *J* = 11.4 Hz, 1H), 4.94 (d, *J* = 10.8 Hz, 1H), 4.68 (d, *J* = 11.4 Hz, 1H), 4.63-4.57 (m, 2H), 4.48 (dd, *J* = 20.6, 11.9 Hz, 2H), 3.95-3.75 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 189.2, 156.4, 146.5, 139.9, 137.7, 137.5, 137.3, 131.8, 131.6, 131.3, 129.0, 128.6, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1(2), 128.0, 127.9, 127.4, 127.2, 127.1, 126.7, 106.3, 80.9, 78.7, 78.6, 78.3, 75.6, 73.5, 73.1, 68.2; **IR (neat)** 3420, 3030, 2922, 2870, 1684, 1603 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{41}H_{37}NO_7$ $[M+H]^+$, 656.2570, found 656.2648.



3l

The title compound was prepared according to the general procedure and the compound was obtained as a pale yellow oil (42 mg, 70% yield of diastereomer mixture, 92% *de*); **$[\alpha]^{24}_D$** -176.3 (*c* 0.1, $CHCl_3$); **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.38-7.25 (m, 19H), 5.31 (s, 1H), 5.06-5.03 (m, 1H), 4.92 (d, *J* = 10.8 Hz, 1H), 4.67-4.47 (m, 7H), 3.84-3.73 (m, 3H), 1.34 (s, 6H), 1.20 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 205.3, 155.6, 137.6, 137.4, 137.2, 128.5, 128.4(2), 128.2(2), 128.1, 128.0, 127.9, 127.8(2), 127.7(2), 106.3, 81.3, 78.6, 78.5, 78.4, 75.5, 73.4, 73.3, 72.9, 72.2, 72.1, 68.2, 43.7, 27.7, 27.1, 25.9, 25.6; **IR (neat)** 3391, 3030, 2959, 2930, 2872, 1713, 1557, 1059 cm^{-1}; **HRMS (ESI):** *m/z* calcd for $C_{33}H_{37}NO_7$ $[M+H]^+$, 560.2570, found 560.2648.

X-ray spectra of carbohydrate-integrated isoxazolines (3b)



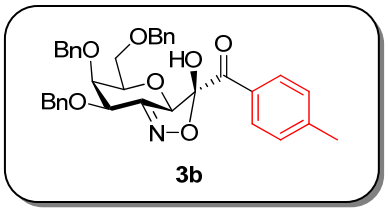
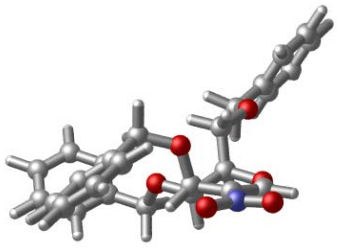


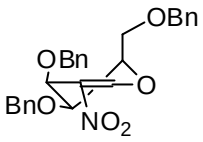
Table 1. Crystal data and structure refinement.

Identification code	Compound 3b	
Empirical formula	C36 H35 N O7	
Formula weight	593.65	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 10.4039(4) Å	α= 90°.
	b = 11.4624(4) Å	β= 91.861(2)°.
	c = 12.6741(4) Å	γ = 90°.
Volume	1510.64(9) $Å^3$	
Z	2	
Density (calculated)	1.305 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	628	
Crystal size	0.40 x 0.38 x 0.26 mm^3	
Theta range for data collection	1.61 to 33.16°.	
Index ranges	-15<=h<=15, -16<=k<=17, -19<=l<=19	
Reflections collected	37285	

Independent reflections	5923 [R(int) = 0.0371]
Completeness to theta = 33.16°	98.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9769 and 0.9647
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5923 / 264 / 472
Goodness-of-fit on F^2	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0405, wR2 = 0.1015
R indices (all data)	R1 = 0.0507, wR2 = 0.1139
Largest diff. peak and hole	0.535 and -0.412 e.$Å^{-3}$

Gaussian Calculations[2]

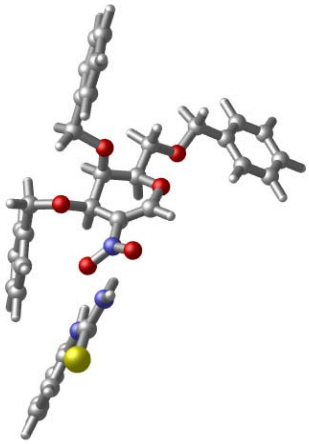


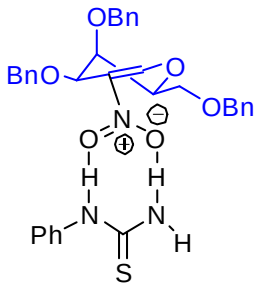


0 1

C	-1.04307900	-2.79705000	-0.93814800
C	0.18429600	-3.12224700	-1.40884300
H	0.44501700	-4.12765800	-1.70959100
O	1.19881100	-2.23878300	-1.58880100
C	-1.41655300	-1.40669000	-0.51830100
H	-2.44857000	-1.18000100	-0.80296500
C	-0.45595800	-0.44164000	-1.23978700
H	-0.69645700	-0.48665800	-2.31363100
N	-2.01917700	-3.84774100	-0.81477900
O	-3.20154000	-3.52068900	-0.49218900
O	-1.66302000	-5.04628700	-1.04905200
O	-1.28936900	-1.35873300	0.93595200
O	-0.67107000	0.90741600	-0.75004900
C	1.02167900	-0.84859000	-1.06575900
H	1.65517100	-0.26362500	-1.73639300
C	-2.02427100	-0.29130500	1.65782300
H	-1.80052300	-0.53640300	2.70011400
H	-1.58491400	0.67671600	1.41050100
C	-0.44428700	1.97753700	-1.74190300
H	0.60214000	1.95548100	-2.07297100
H	-1.08763300	1.79070200	-2.61167600
C	-3.51351800	-0.27667600	1.41107600
C	-4.29713000	-1.41700900	1.66008400
C	-4.13937500	0.89386700	0.95149500
C	-5.67957300	-1.38238000	1.46095800
H	-3.81722800	-2.33375400	1.98411800
C	-5.52603300	0.93243600	0.76180000
H	-3.53644100	1.77382000	0.74507600
C	-6.29829800	-0.20604200	1.01679700
H	-6.27295900	-2.27142200	1.64829500
H	-5.99981500	1.84503600	0.41276200
H	-7.37323200	-0.18026700	0.86680700
C	-0.77309900	3.29214600	-1.09248500
C	-2.07387000	3.81660700	-1.16607700
C	0.20536700	3.99453400	-0.37125600
C	-2.38976800	5.02362200	-0.53347900
H	-2.83698700	3.27820300	-1.72086200
C	-0.10748400	5.20098700	0.26281000
H	1.21335800	3.59419200	-0.30712400
C	-1.40643700	5.71744700	0.18217200
H	-3.39684600	5.42236300	-0.60048500
H	0.65758800	5.73646200	0.81562600
H	-1.65007200	6.65502300	0.67139600
C	1.56286700	-0.76844600	0.36294900
H	1.15603900	-1.57298200	0.97388600
H	1.25583100	0.18955000	0.80229000
O	3.00609500	-0.91552300	0.39540600

C	3.76008800	0.30273200	0.14470800
H	3.37790800	1.09875100	0.80748400
H	3.62175700	0.64698400	-0.89222400
C	5.22522500	0.04651500	0.40837100
C	6.19949000	0.80530600	-0.25878700
C	5.63013600	-0.91599900	1.34530000
C	7.55844100	0.61541200	0.01352100
H	5.89621000	1.54338700	-0.99748400
C	6.98954100	-1.11158700	1.61098600
H	4.87590000	-1.51490600	1.84105100
C	7.95714800	-0.34502400	0.95041300
H	8.30217500	1.20788300	-0.51036300
H	7.29296800	-1.86405000	2.33234700
H	9.01134100	-0.49862400	1.15871200

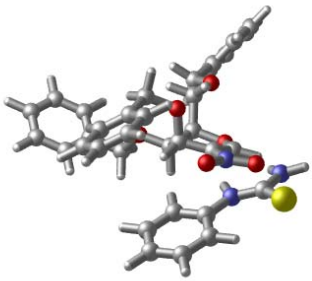


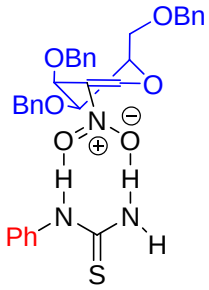


0 1

C	0.07360600	0.07684600	-0.68136200
C	-0.91937100	-0.30167200	-1.53061800
H	-0.75073200	-0.46808100	-2.58613700
O	-2.18205300	-0.56040300	-1.14929000
C	-0.17391300	0.38447100	0.77292200
H	0.12769500	-0.45419800	1.41639400
C	-1.69571900	0.64684200	0.95177800
H	-1.92539200	0.68809800	2.02716900
C	-2.51261100	-0.47352800	0.31045600
H	-2.25150300	-1.44955100	0.73468300
O	0.59921100	1.55925300	1.12878400
C	0.90022200	1.74288900	2.56674700
H	1.54090400	2.62754200	2.55049500
H	-0.02282600	1.99781400	3.10446400
C	1.58560600	0.57767500	3.23716900
C	2.86082000	0.15800300	2.81755800
C	0.96827800	-0.08510300	4.31129400
C	3.50489500	-0.89968600	3.46517400
H	3.33744500	0.65310700	1.97905500
C	1.61374400	-1.14249900	4.96338300
H	-0.01417400	0.23760200	4.64791400
C	2.88316400	-1.55026800	4.54012800
H	4.48790100	-1.21319200	3.13029700
H	1.12906000	-1.64228400	5.79609600

H	3.38755200	-2.36803300	5.04504900
O	-2.14864200	1.84937700	0.28867500
C	-1.92770300	3.14756700	0.95983500
H	-0.88709100	3.44261700	0.81813700
C	-4.01230700	-0.27258100	0.38674700
H	-4.28783700	0.06810000	1.39728100
H	-4.31369000	0.49748100	-0.33181200
O	-4.61964300	-1.55075100	0.09515400
C	-6.05368100	-1.48981000	-0.16486600
H	-6.59136100	-1.18655900	0.74595500
H	-6.23842900	-0.72003400	-0.93219400
C	-6.52299900	-2.84363600	-0.63489900
C	-5.74156400	-3.59598300	-1.52669100
C	-7.76254700	-3.34899300	-0.21596000
C	-6.19772200	-4.83266700	-1.99217700
H	-4.77492000	-3.21200900	-1.83266000
C	-8.22356800	-4.58286200	-0.68942300
H	-8.36763100	-2.77895400	0.48421500
C	-7.44128700	-5.32781200	-1.57844100
H	-5.58464500	-5.40992300	-2.67731000
H	-9.18472100	-4.96347200	-0.35829800
H	-7.79451200	-6.28738600	-1.94264500
H	-2.12058400	3.02267000	2.03531400
C	-2.87922600	4.14125300	0.35393300
C	-4.18610600	4.27712700	0.84998800
C	-2.48279700	4.92381600	-0.74210100
C	-5.07918800	5.18069300	0.26639000
H	-4.50116100	3.67673000	1.69946000
C	-3.37412200	5.82744000	-1.32991600
H	-1.47504100	4.82163700	-1.13400300
C	-4.67375800	5.95746000	-0.82620800
H	-6.08470100	5.28206000	0.66246500
H	-3.05585500	6.42833500	-2.17572100
H	-5.36483600	6.66119500	-1.27919000
N	1.38994500	0.13561600	-1.21601400
O	2.37790100	0.03066100	-0.42076300
O	1.54542200	0.25745700	-2.48135500
C	5.50479800	0.47538100	-2.70595600
N	4.39719100	1.01993700	-3.28303200
H	3.44775300	0.78712100	-3.00594000
S	7.06395000	0.77664000	-3.41830800
N	5.22284000	-0.26579600	-1.58661900
H	4.25158100	-0.22676900	-1.28029400
C	6.02323700	-1.09733800	-0.76373400
C	5.32143700	-1.89788900	0.16371800
C	7.42662300	-1.16969900	-0.80248400
C	6.00636300	-2.75411600	1.02649100
H	4.23675100	-1.84924900	0.19679300
C	8.09846200	-2.03516300	0.06833600
H	7.97319100	-0.56022800	-1.50645900
C	7.40425400	-2.83101000	0.98454600
H	5.44455600	-3.37004400	1.72272100
H	9.18233000	-2.08034300	0.02400700
H	7.93904100	-3.49985300	1.65112900
H	4.54335200	1.59790700	-4.09401100

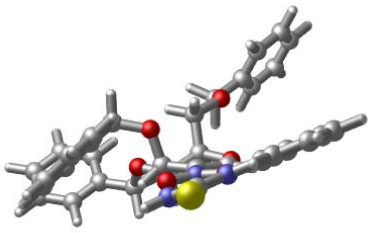


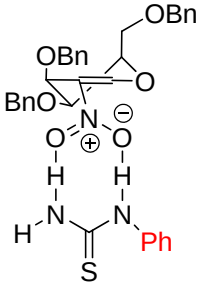


0 1

C	-0.22724000	-1.47029900	-0.67944100
C	0.64286600	-2.38048800	-1.19654800
H	0.34385400	-3.38318500	-1.47154700
O	1.94020600	-2.13018800	-1.45555200
C	0.18573300	-0.08192500	-0.28479600
H	-0.60220700	0.64291700	-0.51240900
C	1.44839800	0.26518800	-1.09804200
H	1.14397600	0.34373300	-2.15378000
N	-1.56983300	-1.88396900	-0.46293600
O	0.42054700	-0.12211200	1.15305400
O	1.96902200	1.53892300	-0.64499100
C	2.53151600	-0.82765800	-0.99426500
H	3.31606300	-0.65839700	-1.73467300
C	0.38314200	1.16528700	1.89894500
H	0.53025300	0.82448400	2.92713800
H	1.23217700	1.77806100	1.59180100
C	2.63781900	2.35178800	-1.68571800
H	3.51789000	1.81256100	-2.05868400
H	1.93761100	2.49500300	-2.51872200
C	-0.90921400	1.92857700	1.75511600
C	-2.12559300	1.36905400	2.18516600
C	-0.90890600	3.22153000	1.20593900
C	-3.31592200	2.09317100	2.07752100
H	-2.13573200	0.36296300	2.59159100
C	-2.09976500	3.95107300	1.10412800
H	0.02712100	3.65435100	0.86451600
C	-3.30415300	3.38821600	1.54006300
H	-4.25050100	1.65168200	2.40666800
H	-2.08687100	4.95304500	0.68618800
H	-4.22951100	3.95015700	1.46282700
C	3.03206700	3.66414400	-1.07114000
C	2.16776900	4.76933200	-1.13626200
C	4.25496100	3.79307800	-0.39322700
C	2.52084800	5.98291900	-0.53677000
H	1.22067400	4.67848200	-1.66069700
C	4.60993800	5.00459200	0.20769700
H	4.92920700	2.94294600	-0.33753000
C	3.74265200	6.10162800	0.13655700
H	1.84828400	6.83227700	-0.59744900
H	5.55869000	5.09406000	0.72667600
H	4.01859500	7.04338300	0.59965900
O	-1.88808500	-3.10812500	-0.65493100
O	-2.42195200	-1.01085900	-0.09625800
N	-5.45972700	-1.74386400	-0.17093800
N	-4.70721300	-3.86018100	0.24576400
H	-4.88297100	-4.81274700	0.51920900
H	-3.76771000	-3.58999300	-0.03021200
H	-4.46147100	-1.54044800	-0.17638100
C	-5.78690200	-3.03363700	0.16257100

S	-7.36997200	-3.67166800	0.50338600
C	-6.25347500	-0.60411500	-0.45695000
C	-5.55887200	0.62061200	-0.55027700
C	-7.64090000	-0.62159000	-0.68253600
C	-6.23661200	1.80088500	-0.85555500
H	-4.48707700	0.64276900	-0.37772600
C	-8.30625300	0.57134000	-0.98592800
H	-8.18449400	-1.55139600	-0.60921000
C	-7.61946800	1.78636000	-1.07465900
H	-5.67981400	2.73106600	-0.91939100
H	-9.37831500	0.54139700	-1.15526300
H	-8.14976300	2.70356400	-1.31008100
C	3.15300200	-1.02335300	0.39006900
H	2.44294300	-1.49677900	1.06700000
H	3.41390800	-0.03774100	0.79741700
O	4.31327500	-1.88913700	0.33059300
C	5.56716300	-1.22431100	-0.00212500
H	5.69618600	-0.36114100	0.67326400
H	5.54539400	-0.83498200	-1.03183600
C	6.70601100	-2.20287800	0.15329300
C	7.84546600	-2.08720500	-0.65760600
C	6.66077700	-3.20697900	1.13260300
C	8.93104300	-2.95290700	-0.48586900
H	7.88319700	-1.32154400	-1.42859700
C	7.74233400	-4.07811700	1.29775900
H	5.77051600	-3.30834900	1.74171900
C	8.88162100	-3.95145300	0.49338300
H	9.80644300	-2.85387300	-1.12007000
H	7.69523000	-4.85643000	2.05303700
H	9.71999500	-4.62829700	0.62425500

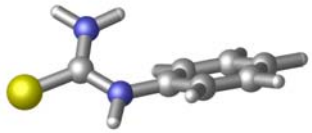


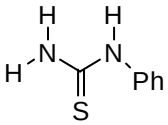


0 1

C	0.58973800	0.24243600	-0.90457900
C	0.20609200	1.46704200	-1.35675100
H	0.91369500	2.23036600	-1.65205800
O	-1.07528600	1.84701300	-1.52162000
C	-0.38938700	-0.81341200	-0.48308000
H	-0.03492200	-1.80944900	-0.76584300
C	-1.71958800	-0.51889600	-1.20233300
H	-1.55675400	-0.69259300	-2.27836400
N	1.98027000	-0.02319700	-0.78373900
O	-0.49637300	-0.72099700	0.96834000
O	-2.72379500	-1.43718500	-0.70939400
C	-2.16965000	0.94580300	-1.02021200
H	-2.98852300	1.18293700	-1.70243500
C	-0.94087900	-1.93979500	1.69872200
H	-0.93225200	-1.58995500	2.73417500
H	-1.96357700	-2.17748100	1.40162500
C	-3.82384800	-1.74491200	-1.64949200
H	-4.45649500	-0.85777500	-1.77670100
H	-3.38517700	-2.00427100	-2.62235800

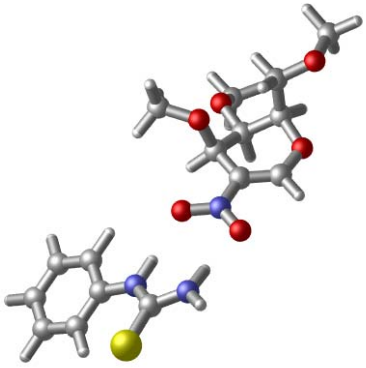
C	-0.03161600	-3.12766800	1.51253900
C	1.30485900	-3.08066300	1.94720800
C	-0.51136700	-4.30147500	0.90839000
C	2.14118100	-4.18825900	1.78900000
H	1.68667900	-2.17098900	2.39949900
C	0.32437000	-5.41444200	0.75393100
H	-1.54192600	-4.34109200	0.56582600
C	1.65106600	-5.35925400	1.19435800
H	3.17072500	-4.14054900	2.12911800
H	-0.05831400	-6.31917900	0.29173800
H	2.30028200	-6.22112700	1.07626900
C	-4.61309100	-2.89066000	-1.08307400
C	-4.20218000	-4.21489000	-1.31081600
C	-5.74790000	-2.65469100	-0.29168400
C	-4.91480400	-5.28373500	-0.75890600
H	-3.32591200	-4.40561800	-1.92404500
C	-6.46289500	-3.72219700	0.26143000
H	-6.07178000	-1.63372000	-0.10972800
C	-6.04690000	-5.03819600	0.02852500
H	-4.59242000	-6.30301600	-0.94499500
H	-7.34044200	-3.52849800	0.86957400
H	-6.60230000	-5.86727500	0.45478600
O	2.82388400	0.91704700	-0.98867200
O	2.34130900	-1.20694200	-0.48613900
N	5.34034200	-1.71040400	-0.33643000
N	5.83580300	0.52038400	-0.29021400
H	4.84402800	0.59879500	-0.51073500
H	4.34962800	-1.50397300	-0.41946100
C	6.31015300	-0.76189500	-0.20044500
S	7.95557700	-1.27756000	0.04662500
H	5.63808200	-2.67114300	-0.30371500
C	6.46186500	1.78729100	-0.16163300
C	5.62680400	2.90494300	-0.37699500
C	7.81235900	1.99710700	0.16651500
C	6.13053200	4.20051500	-0.26961400
H	4.58149500	2.74939300	-0.62757700
C	8.30216500	3.30398200	0.27021200
H	8.46120100	1.15020200	0.33105500
C	7.47629100	4.41126100	0.05511700
H	5.46986100	5.04525100	-0.44009100
H	9.34797100	3.44923600	0.52351500
H	7.87111200	5.41868800	0.13851100
C	-2.54027700	1.35590900	0.40600500
H	-1.64983800	1.42041400	1.03014100
H	-3.20075700	0.58802400	0.82976100
O	-3.16660200	2.66186200	0.43396200
C	-4.60706800	2.66550000	0.21285900
H	-5.07139200	1.94951000	0.91250900
H	-4.84947900	2.33049600	-0.80784100
C	-5.14193100	4.05938700	0.43731200
C	-6.28689600	4.48901300	-0.25117200
C	-4.53292000	4.92289800	1.36050500
C	-6.82501300	5.75821800	-0.01314200
H	-6.75715600	3.83228200	-0.97903500
C	-5.06588700	6.19531400	1.59124700
H	-3.63626600	4.59651800	1.87343800
C	-6.21465100	6.61532000	0.90966000
H	-7.71058200	6.07924800	-0.55266500
H	-4.58346700	6.85911800	2.30192300
H	-6.62663400	7.60292000	1.09140000

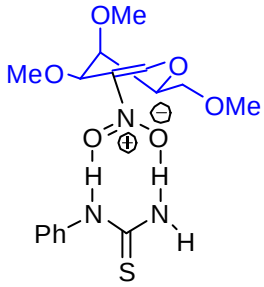




01

C	-1.81590200	0.15950300	0.13130000
S	-3.42908900	-0.37027000	-0.21971400
N	-1.57188400	1.30267600	0.82027000
H	-2.36672500	1.83908700	1.12633500
H	-0.64650800	1.57659800	1.11637700
N	-0.73758600	-0.58990300	-0.26713600
H	-0.99685000	-1.47832400	-0.67404800
C	0.65340900	-0.29684400	-0.15297500
C	1.52556400	-1.31977500	0.25691000
C	1.17432500	0.96556000	-0.49030700
C	2.89948100	-1.08037200	0.33635400
H	1.12009500	-2.29185900	0.51959300
C	2.54931700	1.20161800	-0.38619400
H	0.51474000	1.74033500	-0.86517000
C	3.41660200	0.18353900	0.02535000
H	3.56419400	-1.87809200	0.65139300
H	2.94311000	2.17804000	-0.64944700
H	4.48287000	0.36975400	0.09582700

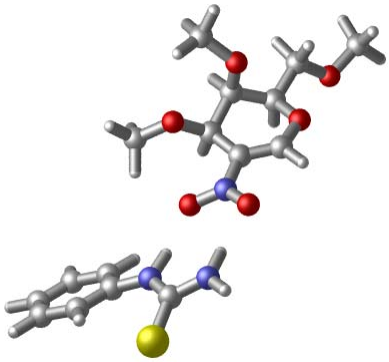


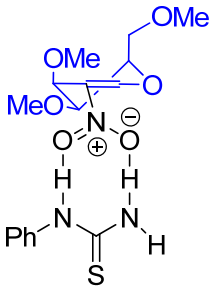


0 1

C	1.53751500	-0.76372900	-0.47114200
C	2.38368300	-1.70448700	-0.97175100
H	2.05399600	-2.69055200	-1.27154800
O	3.69952200	-1.50316300	-1.17375100
C	1.99021900	0.60317000	-0.05670100
H	1.24462300	1.35412500	-0.33846900
C	3.31154200	0.90608100	-0.78488600
H	3.08243600	1.02681700	-1.85558700
N	0.17195200	-1.10517000	-0.28445100
O	2.15720400	0.59261700	1.38802500
O	3.84895900	2.13936700	-0.25553400
C	4.32818000	-0.24399600	-0.63876700
H	5.16794600	-0.10207400	-1.32165200
O	-0.22633600	-2.28916700	-0.55040800
O	-0.61917300	-0.19759500	0.13783600
N	-3.68134600	-0.58798400	0.03625500

N	-3.19431100	-2.82006700	0.09358800
H	-3.49016100	-3.77712100	0.19135700
H	-2.21481800	-2.62222200	-0.08611800
H	-2.66660800	-0.50643200	0.07064900
C	-4.16697700	-1.86670800	0.13488400
S	-5.82553300	-2.35607600	0.32873500
C	-4.33141200	0.67020500	-0.03773000
C	-3.51455300	1.80081000	0.17379900
C	-5.69058500	0.85630900	-0.34318300
C	-4.04557500	3.08773100	0.08828100
H	-2.46309000	1.66120200	0.40795700
C	-6.20949400	2.15319400	-0.42107900
H	-6.32597900	-0.00173300	-0.50299900
C	-5.40126200	3.27440000	-0.20763900
H	-3.39985200	3.94433200	0.25762400
H	-7.26207800	2.28080000	-0.65467300
H	-5.81820600	4.27439500	-0.27024100
C	4.84189800	-0.50164600	0.77944900
H	4.06106900	-0.93912700	1.40050200
H	5.13067100	0.45956600	1.22385800
O	5.94541800	-1.43982700	0.77831200
C	7.24932200	-0.85243300	0.53221700
H	7.32965000	-0.41509700	-0.47379400
H	7.48331100	-0.07873500	1.27679300
H	7.96586700	-1.66966600	0.61836500
C	1.76258900	1.82303600	2.07452800
H	2.38877300	2.66134200	1.76007400
H	0.70480400	2.04055000	1.88528000
H	1.90824100	1.61762300	3.13538900
C	4.65909500	2.91857400	-1.18602000
H	4.08813300	3.16643000	-2.08971400
H	4.91773900	3.83339100	-0.65335200
H	5.57814600	2.39103400	-1.46862300





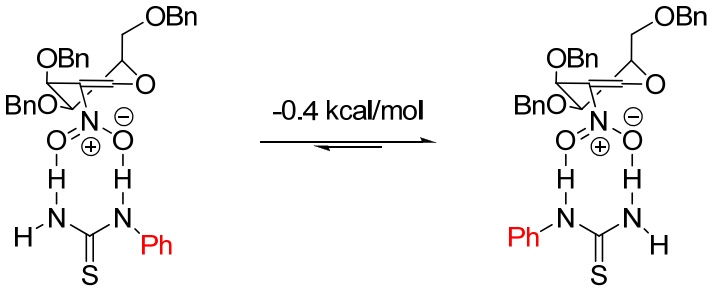
0 1

C	1.64003800	-0.59332900	-0.05263600
C	2.57390000	-1.57078300	-0.21496000
H	2.32464500	-2.62359500	-0.20013300
O	3.87335000	-1.33677700	-0.45412300
C	1.99483200	0.87219100	0.02100400
H	1.76779500	1.37873600	-0.92848800
C	3.51678400	0.98947500	0.30560900
H	3.81730000	2.03588000	0.14812800
C	4.31596500	0.08639700	-0.63154500
H	4.11912500	0.32910100	-1.68195800
O	1.28878500	1.53072000	1.10229600
O	3.87502300	0.55380100	1.63335300

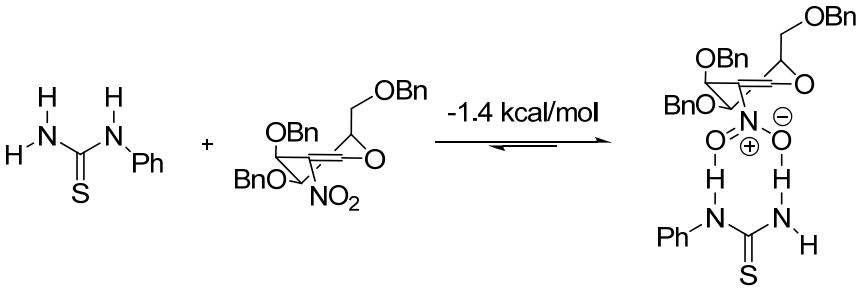
C	5.81027700	0.09450700	-0.38612500
H	6.14834500	1.12475000	-0.19095800
H	6.03830300	-0.51589300	0.49488400
O	6.43350400	-0.42875600	-1.58019300
N	0.28210000	-0.99385500	0.01763800
O	-0.61431100	-0.10472900	-0.16681200
O	-0.01070400	-2.21961300	0.22981300
C	-4.03541700	-1.95707600	0.24567400
N	-3.02354400	-2.84102600	0.46887400
H	-2.03798900	-2.60427200	0.40404600
S	-5.68375000	-2.50909900	0.32852900
N	-3.60130200	-0.69105700	-0.04986900
H	-2.59076300	-0.57350200	-0.10620500
C	-4.31442300	0.49848900	-0.34910800
C	-3.61229100	1.47949800	-1.07905100
C	-5.62740500	0.76731000	0.07290500
C	-4.21110100	2.70274900	-1.38340000
H	-2.60195000	1.26746500	-1.41702000
C	-6.21812700	1.99448000	-0.24690900
H	-6.17410800	0.01998800	0.62854000
C	-5.52248800	2.96811900	-0.97148500
H	-3.65629000	3.44329900	-1.95190800
H	-7.23472700	2.18699800	0.08186800
H	-5.99338200	3.91553700	-1.21309800
H	-3.28525700	-3.78866100	0.68419000
C	0.40797700	2.63258400	0.71806700
H	-0.40833300	2.27383700	0.08791200
H	0.00755600	3.01790200	1.65584300
H	0.96883500	3.42561600	0.20657000
C	3.70647000	1.49831300	2.73583300
H	2.65775500	1.58468900	3.01677300
H	4.29729900	1.08595800	3.55501900
H	4.09857100	2.48729800	2.46103600
C	7.85354300	-0.68505100	-1.43602200
H	8.18590900	-1.08550300	-2.39391800
H	8.40789600	0.23769000	-1.21267800
H	8.04632300	-1.42056800	-0.64319100

Calculation 1:

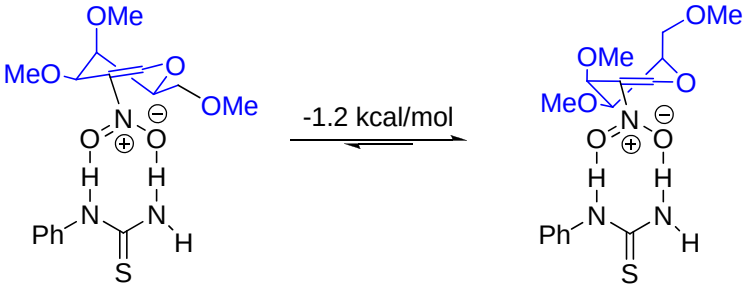
Calculation 2:



Calculation 3:



Calculation 4:

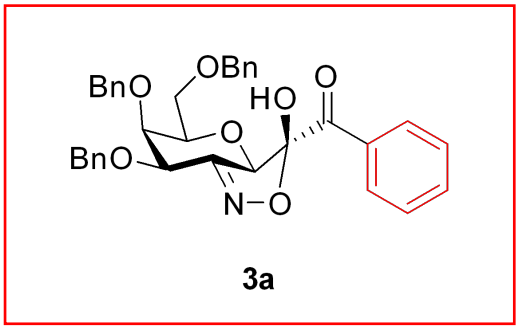


References

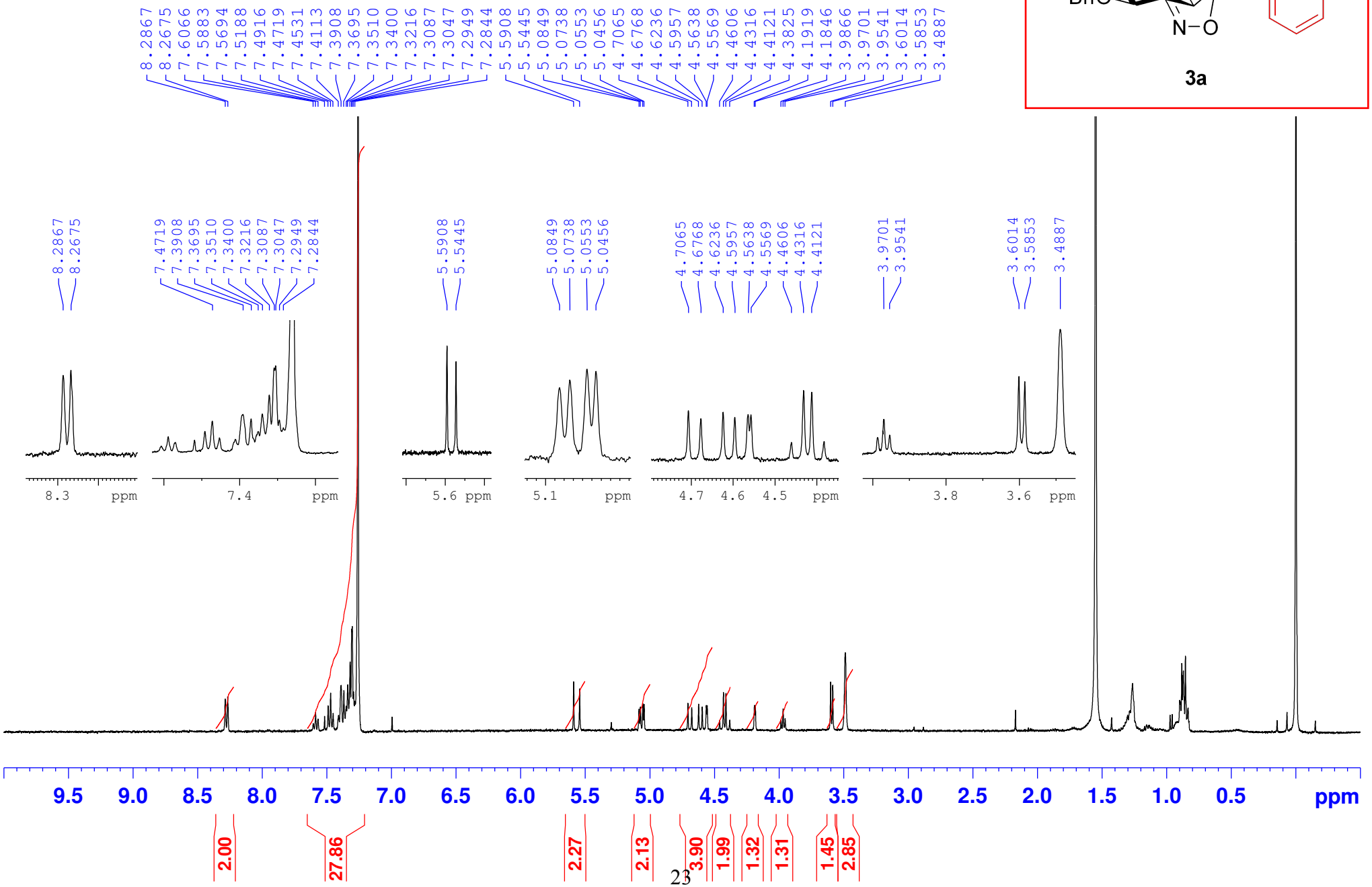
1. J. Das and R. R. Schmidt, *Eur. J. Org. Chem.* 1998, **2**, 1609.

2. Gaussian 09, Revision A.02,
M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

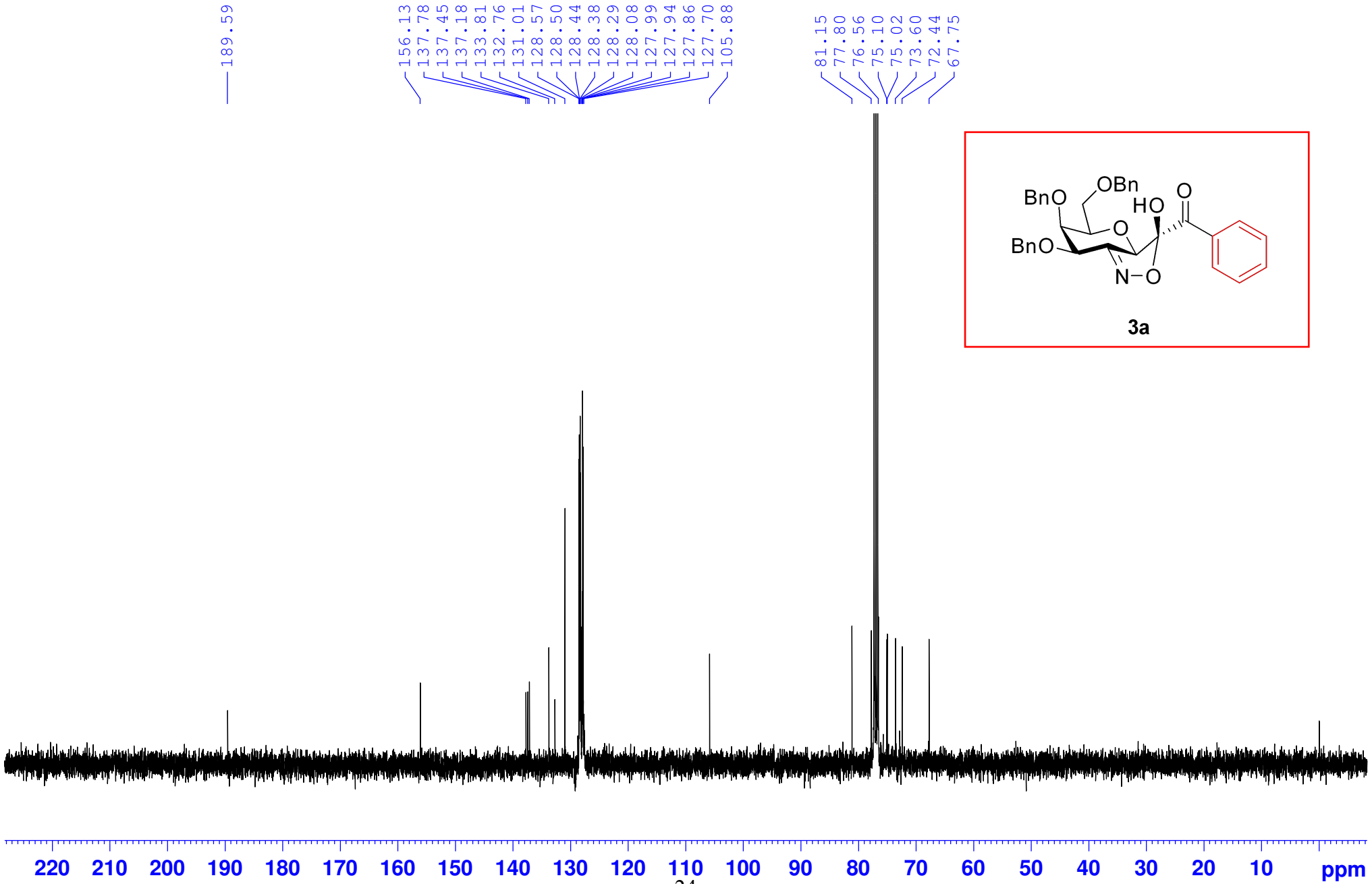
Compound 3a, 400 MHz,CDCl3,1H NMR



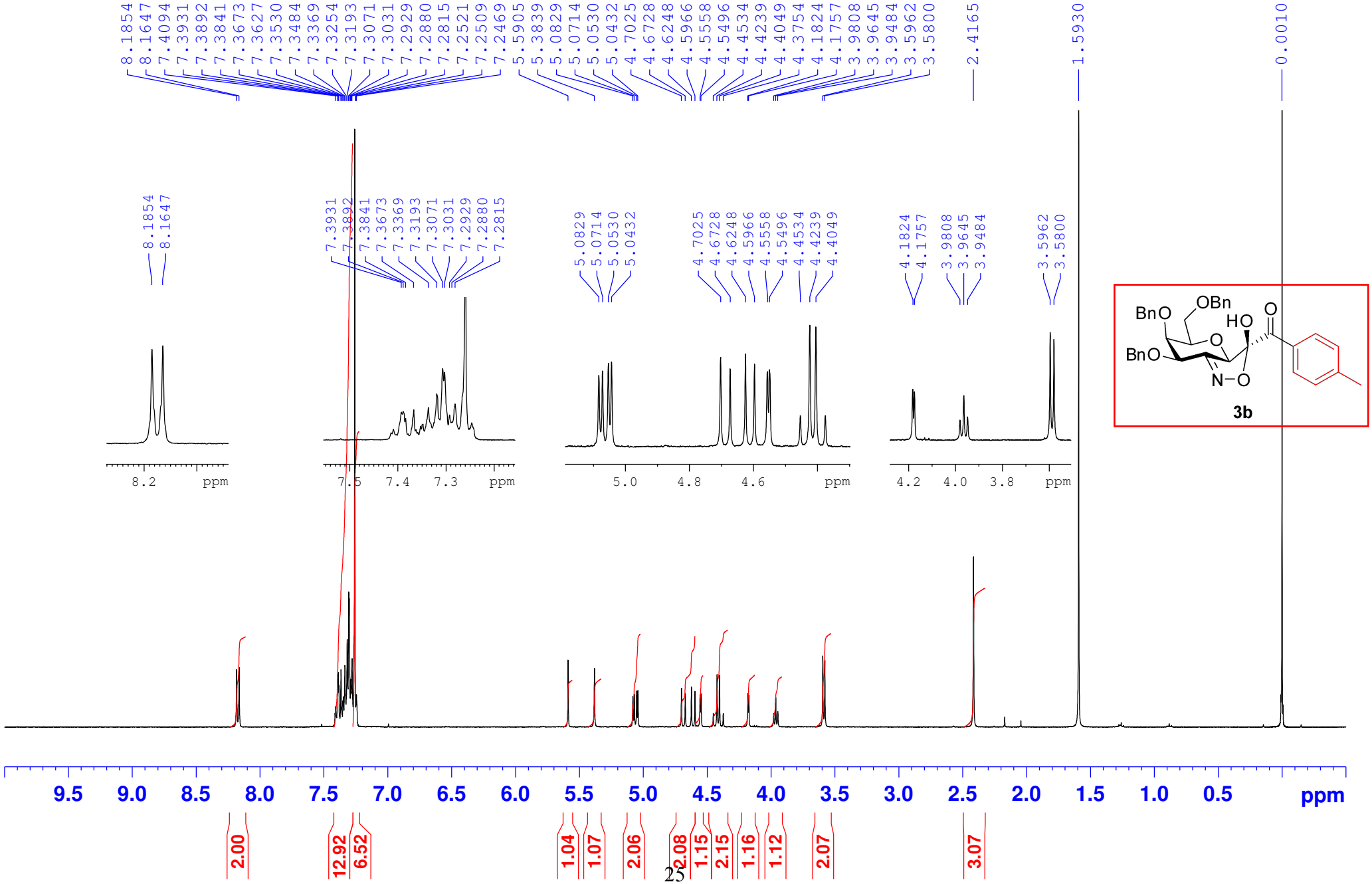
3a



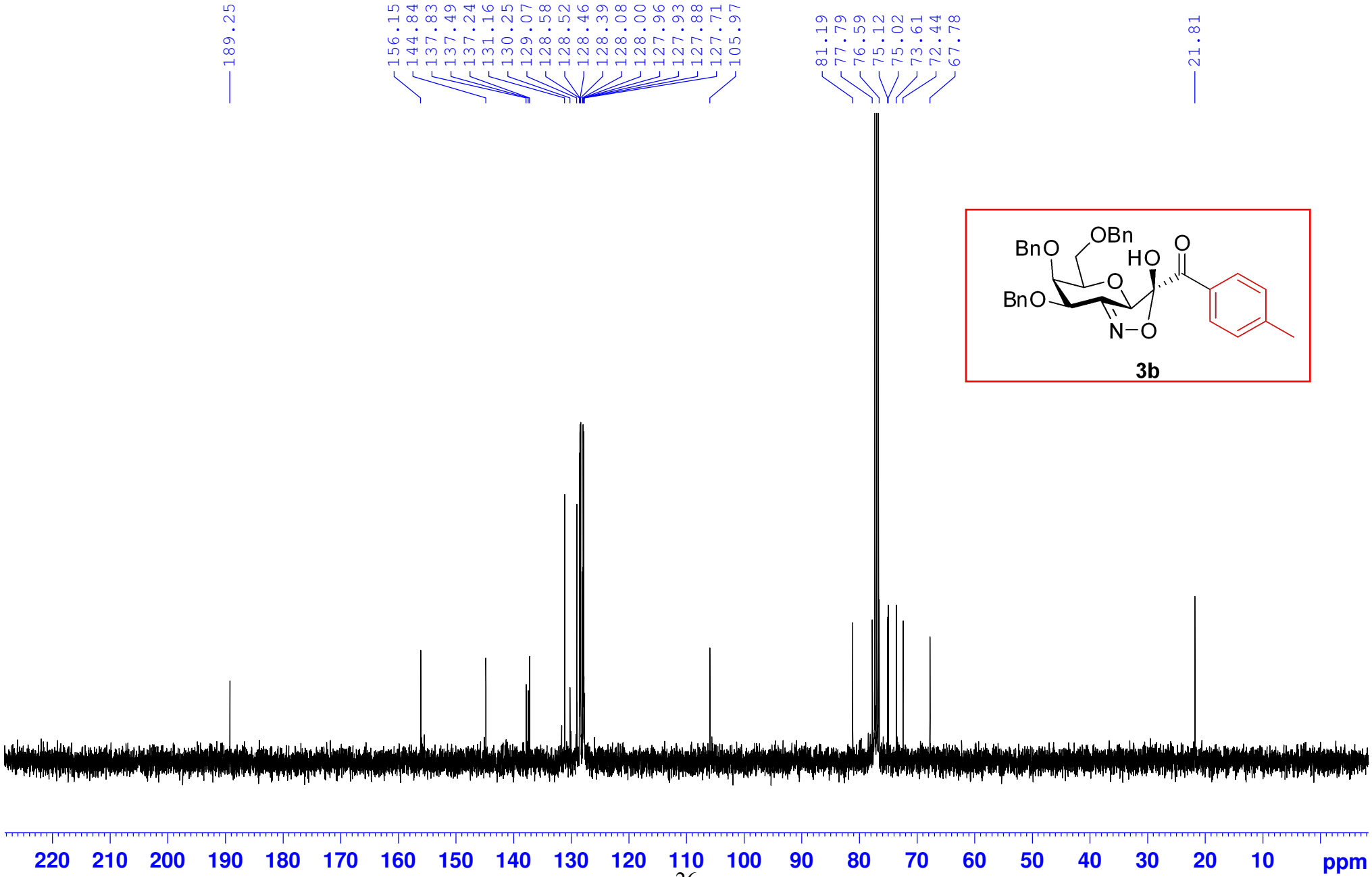
Compound 3a, 13C NMR, 400MHz, CDCl3



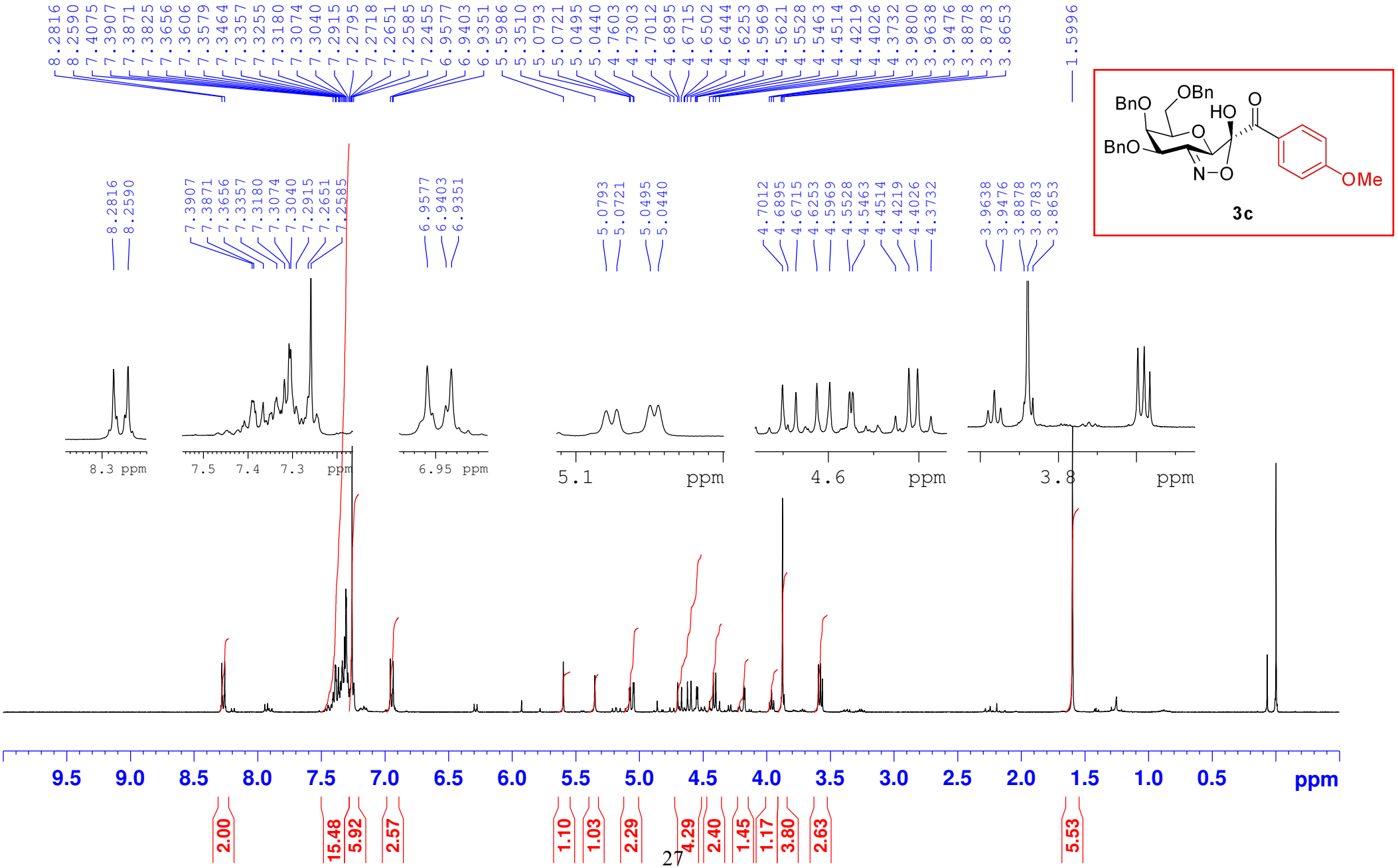
Compound 3b, 1H NMR, 400MHz, CDCl3



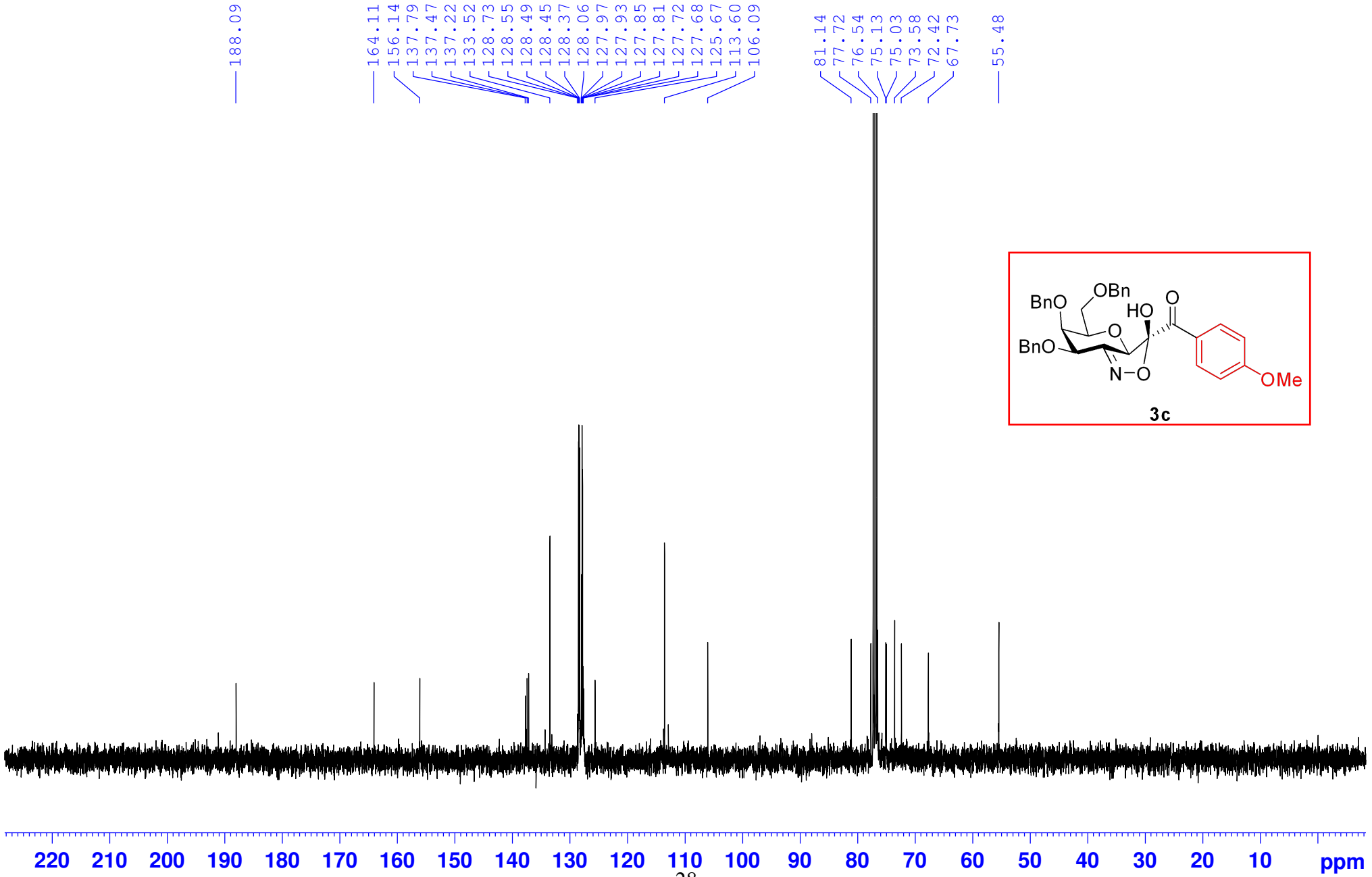
Compound 3b, 13C NMR, 400MHz, CDCl3



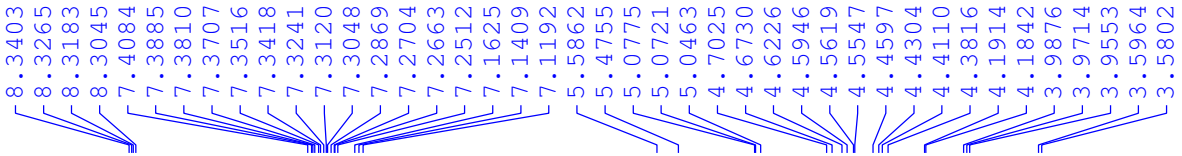
Compound 3c, 1H NMR, 400MHz, CDCl3

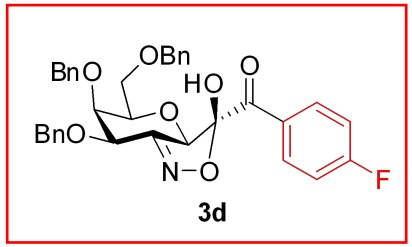


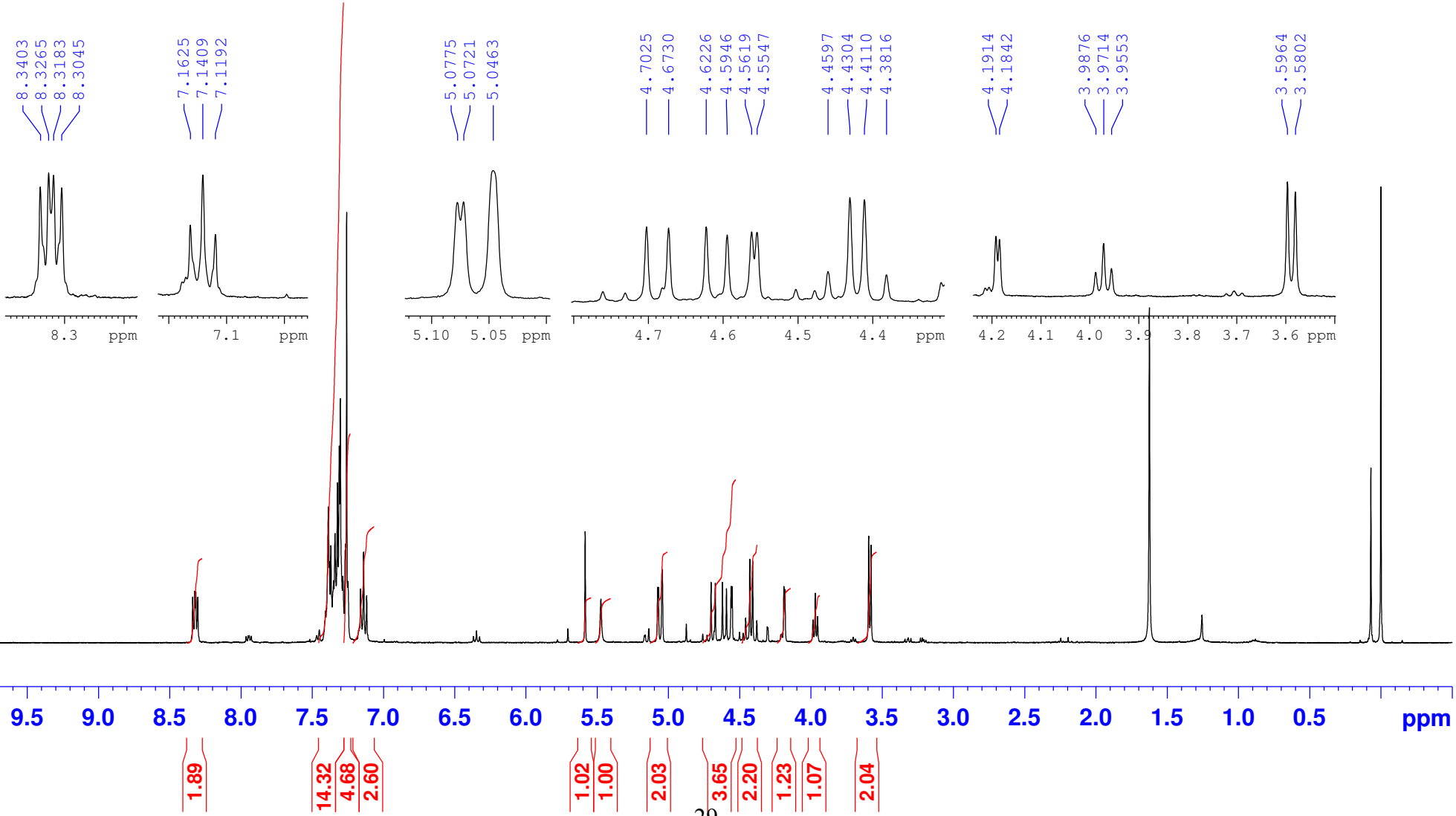
Compound 3c, 13C NMR, 400MHz, CDCl3



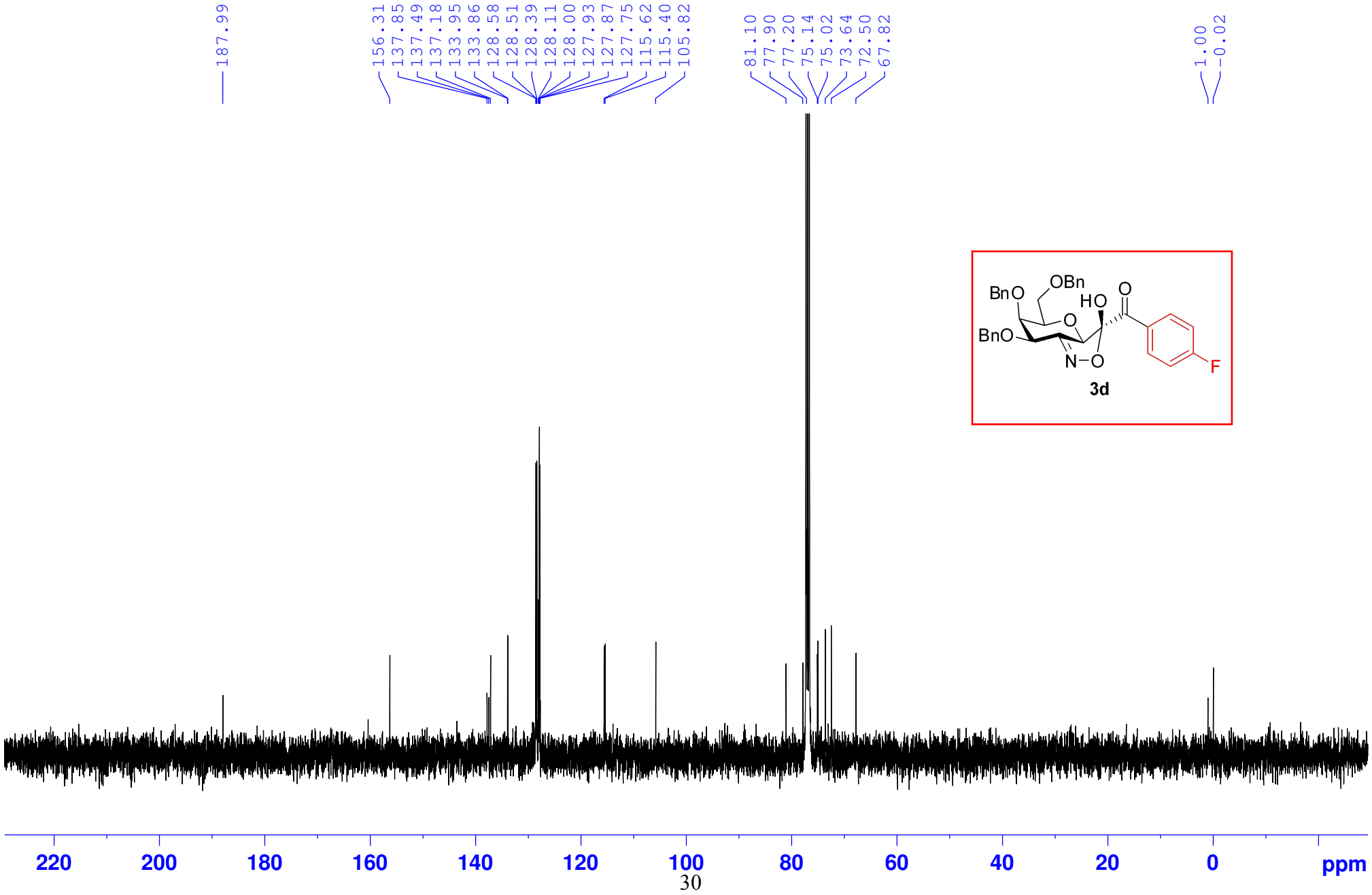
Compound 3d, 1H NMR, 400MHz, CDCl3



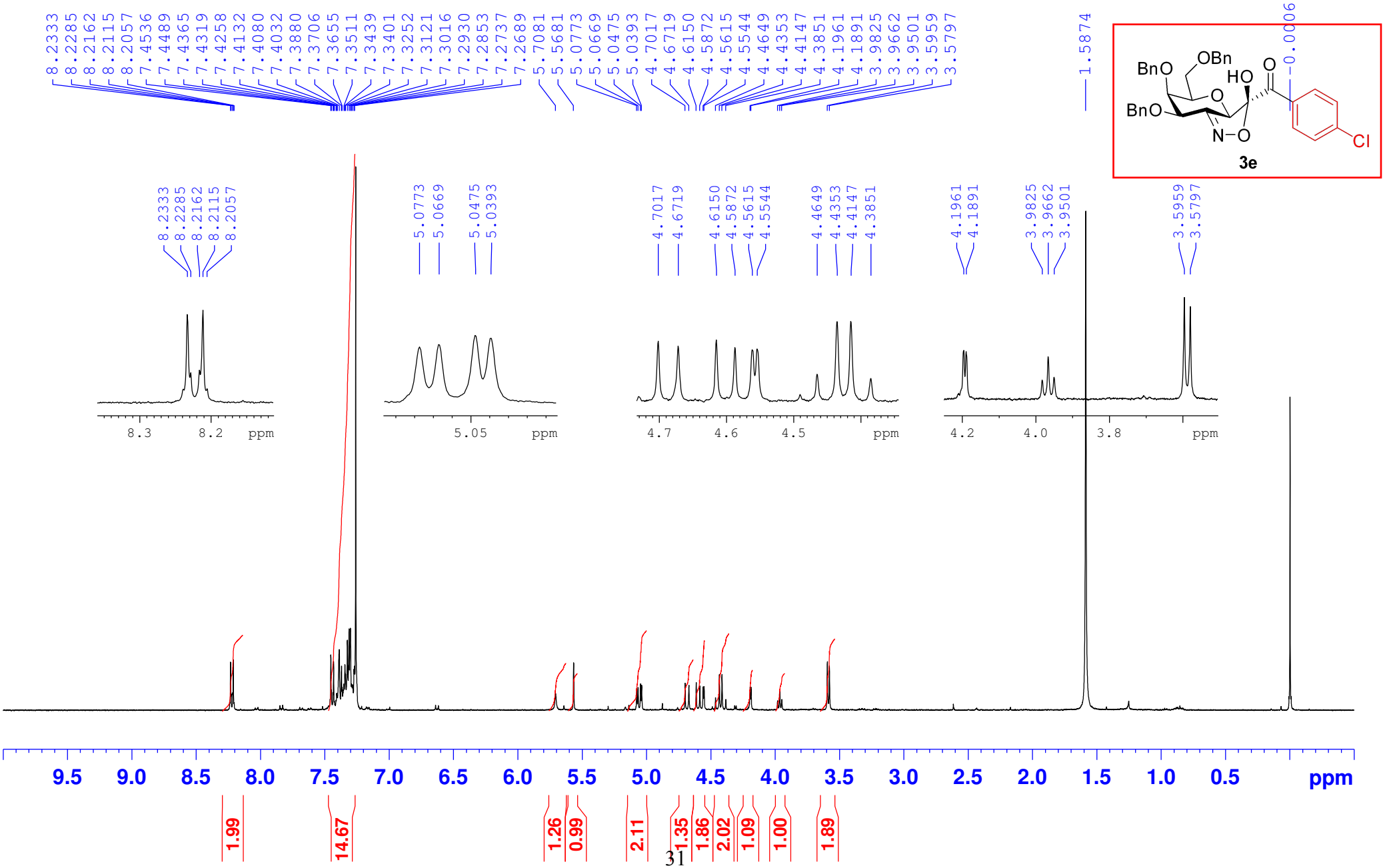




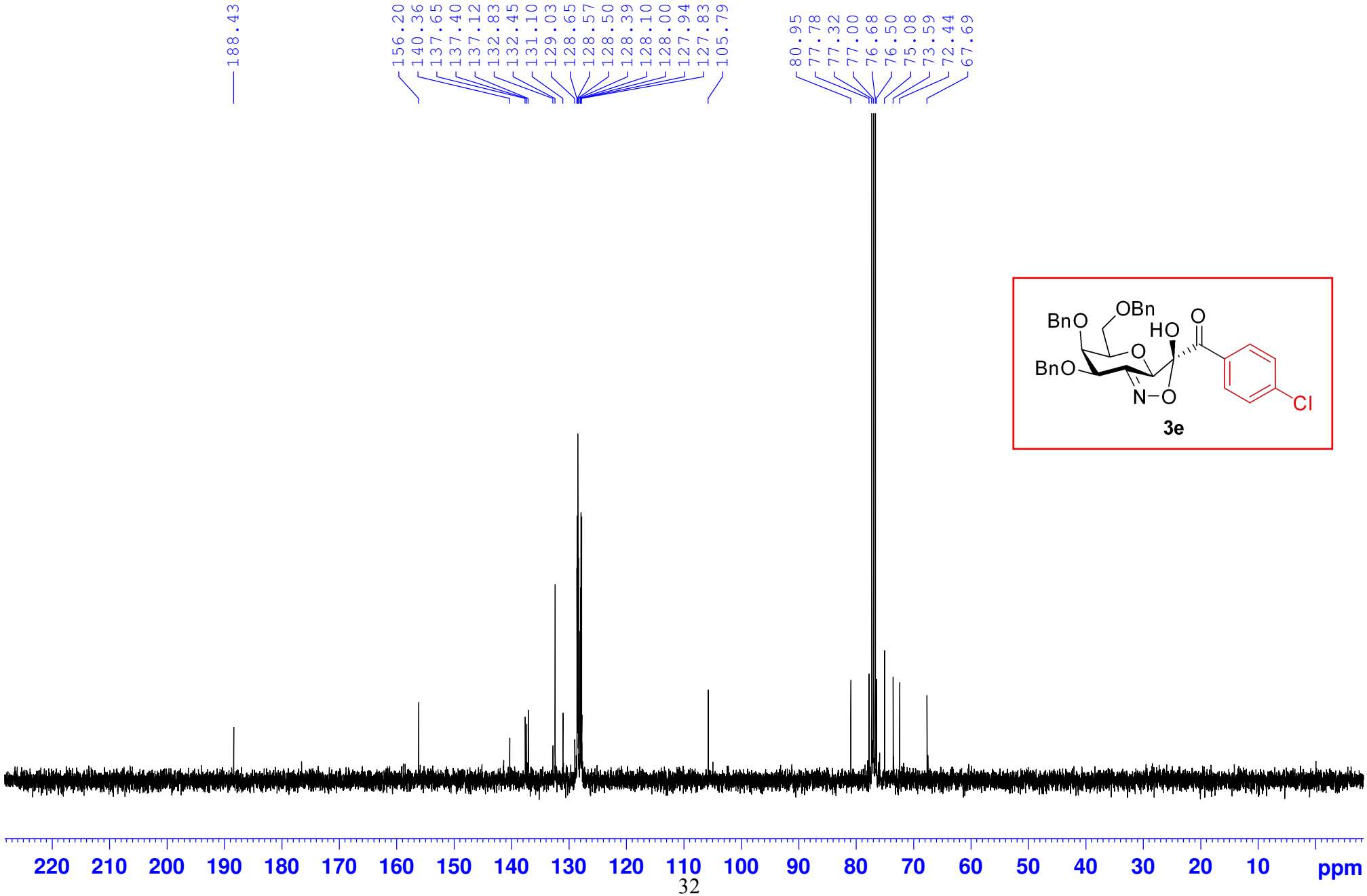
Compound 3d, 13C NMR, 400 MHz, CDCl3



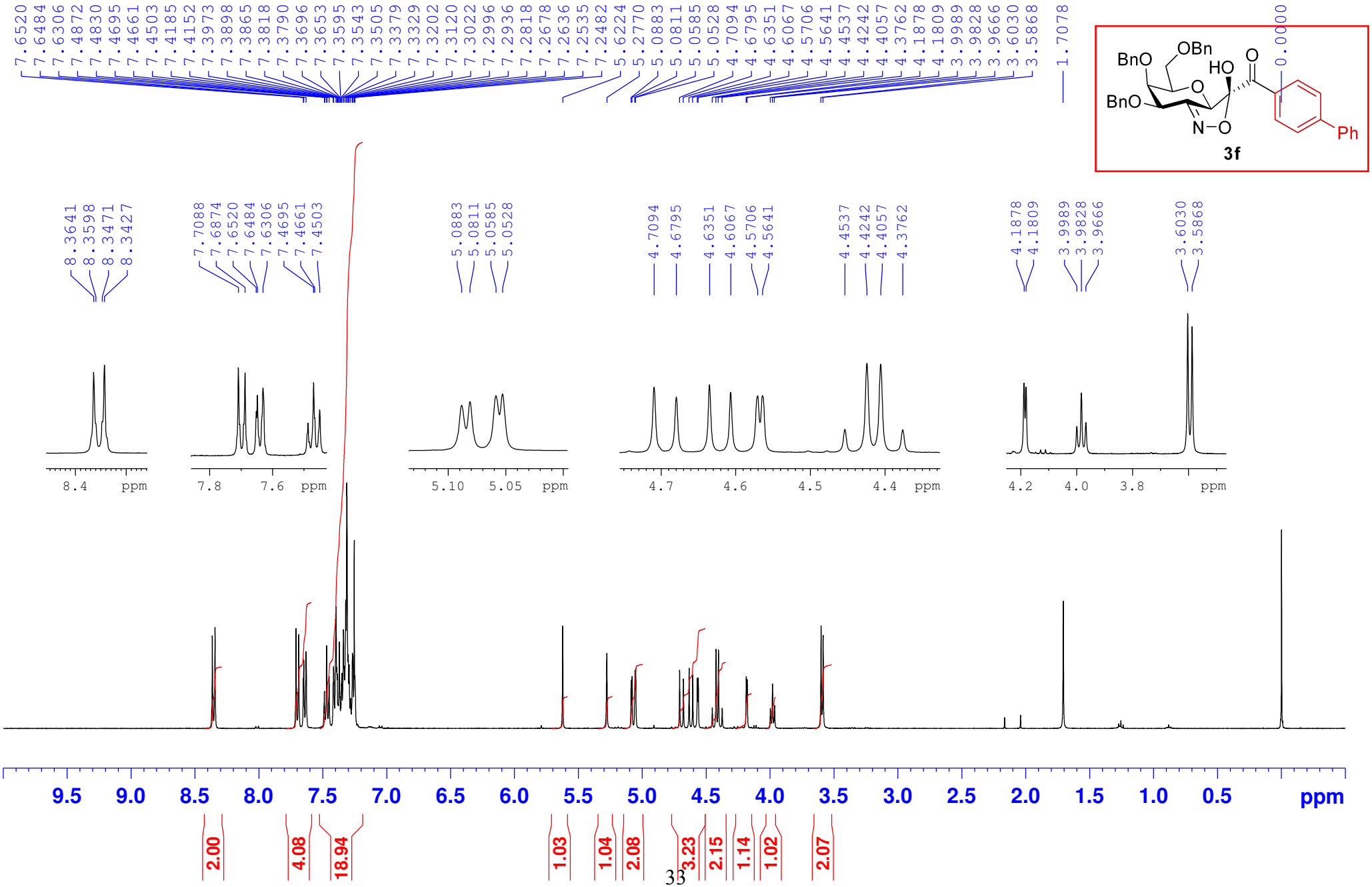
Compound 3e, 1H NMR, 400MHz, CDCl3



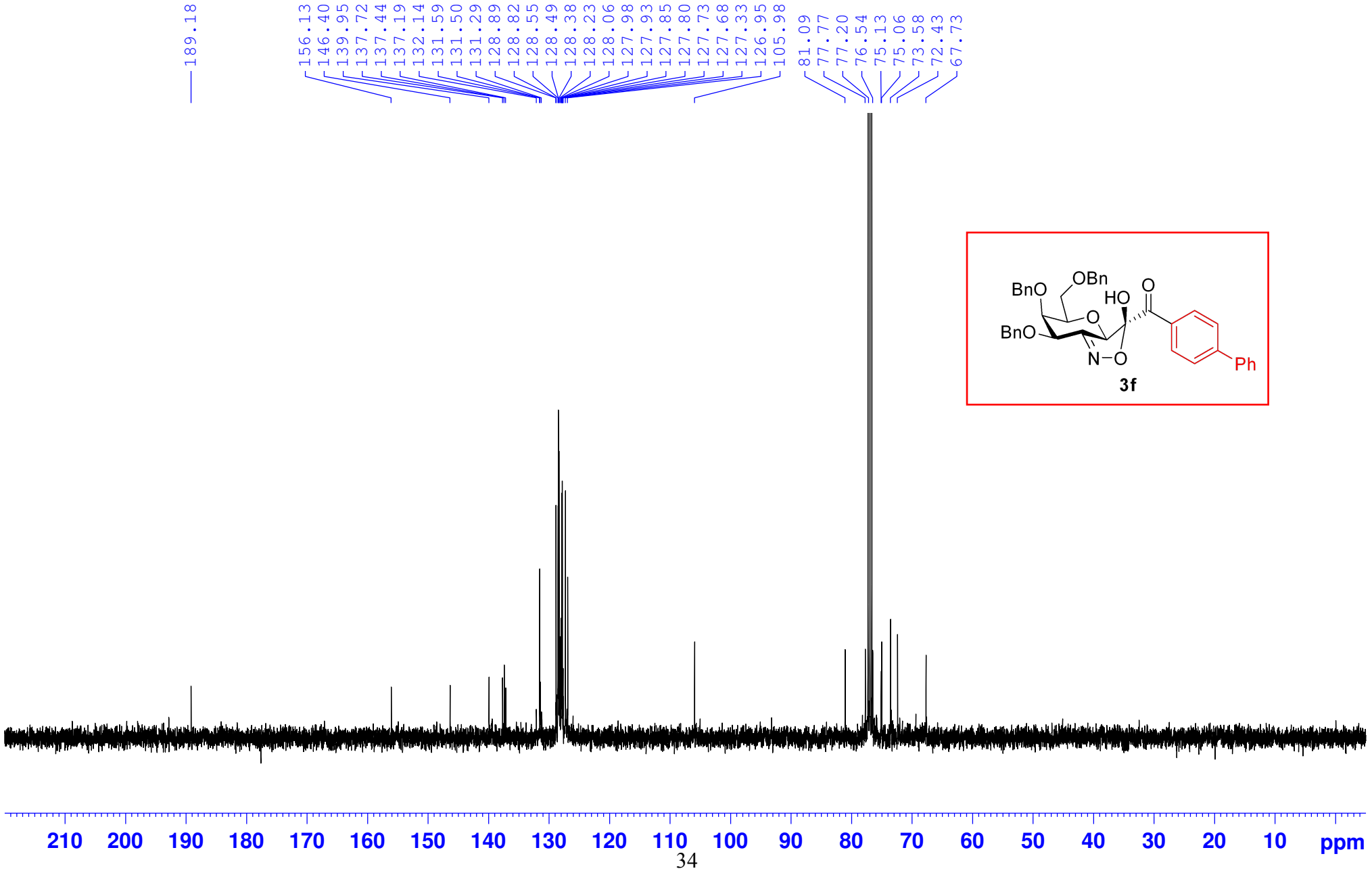
Compound 3e, 13C NMR, 400 MHz, CDCl3



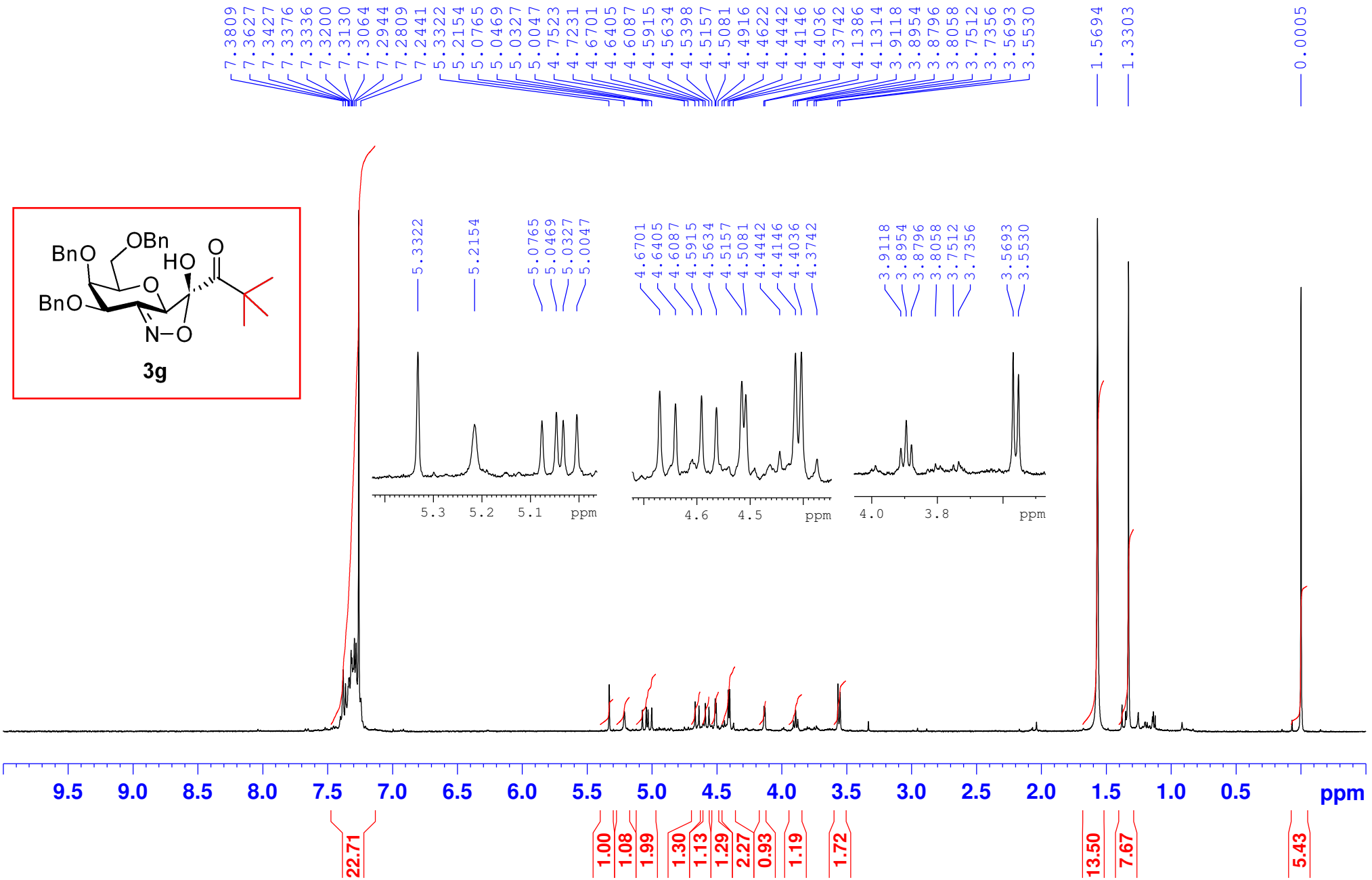
Compound 3f, 1H NMR, 400MHz, CDCl3



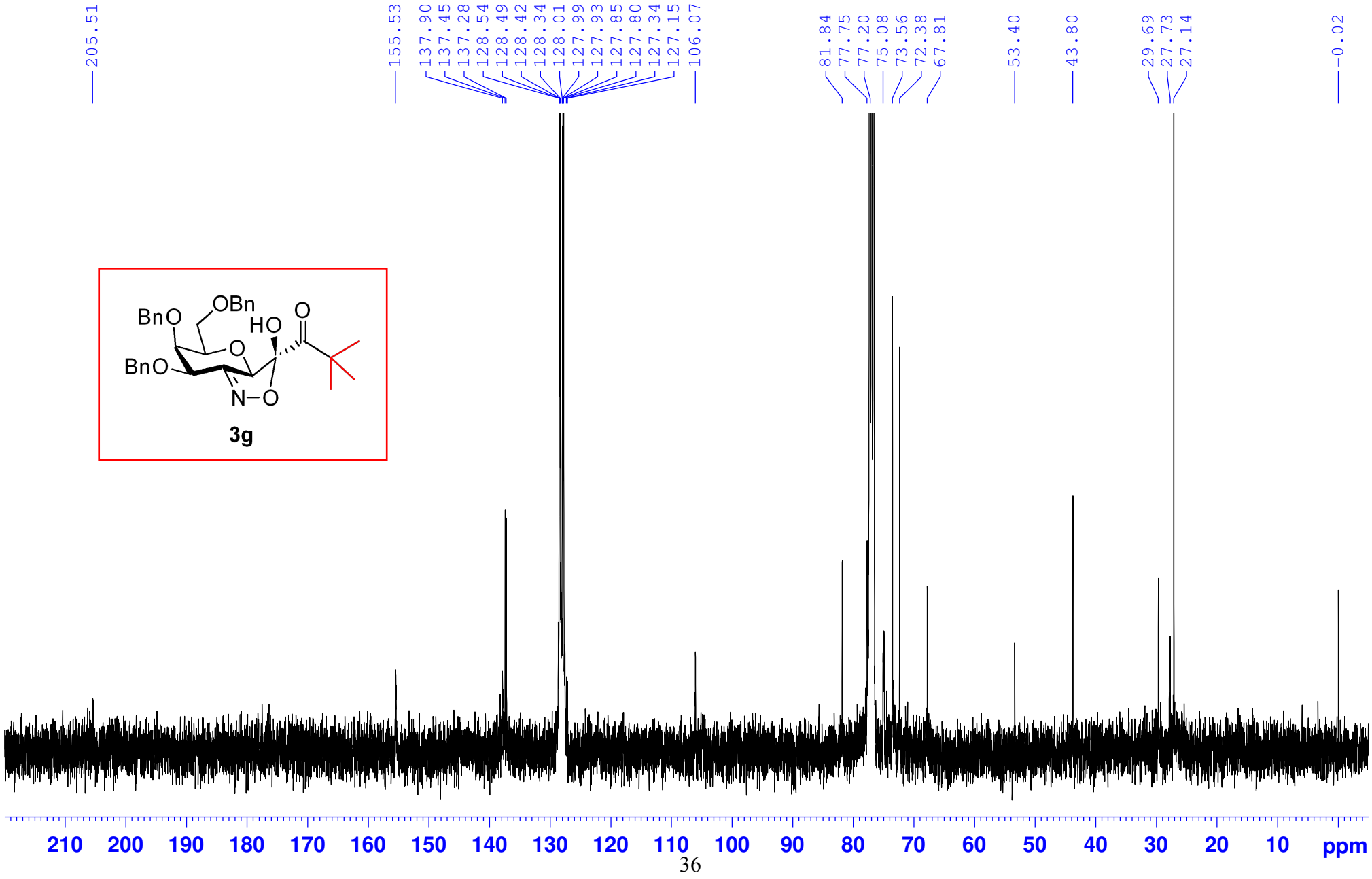
Compound 3f, 13C NMR, 400MHz, CDCl3



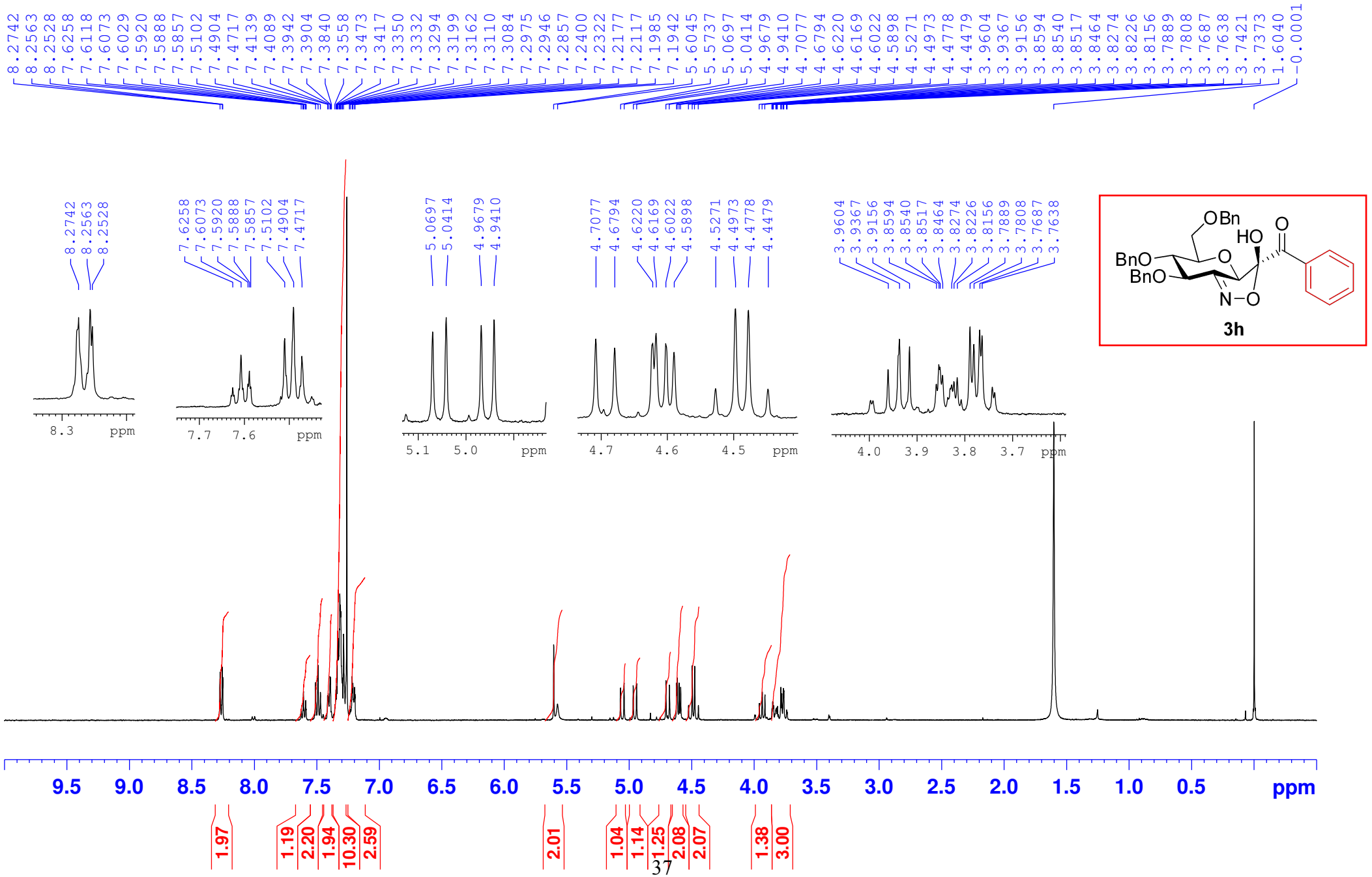
Compound 3g, 400 MHz, CDCl3,1H NMR



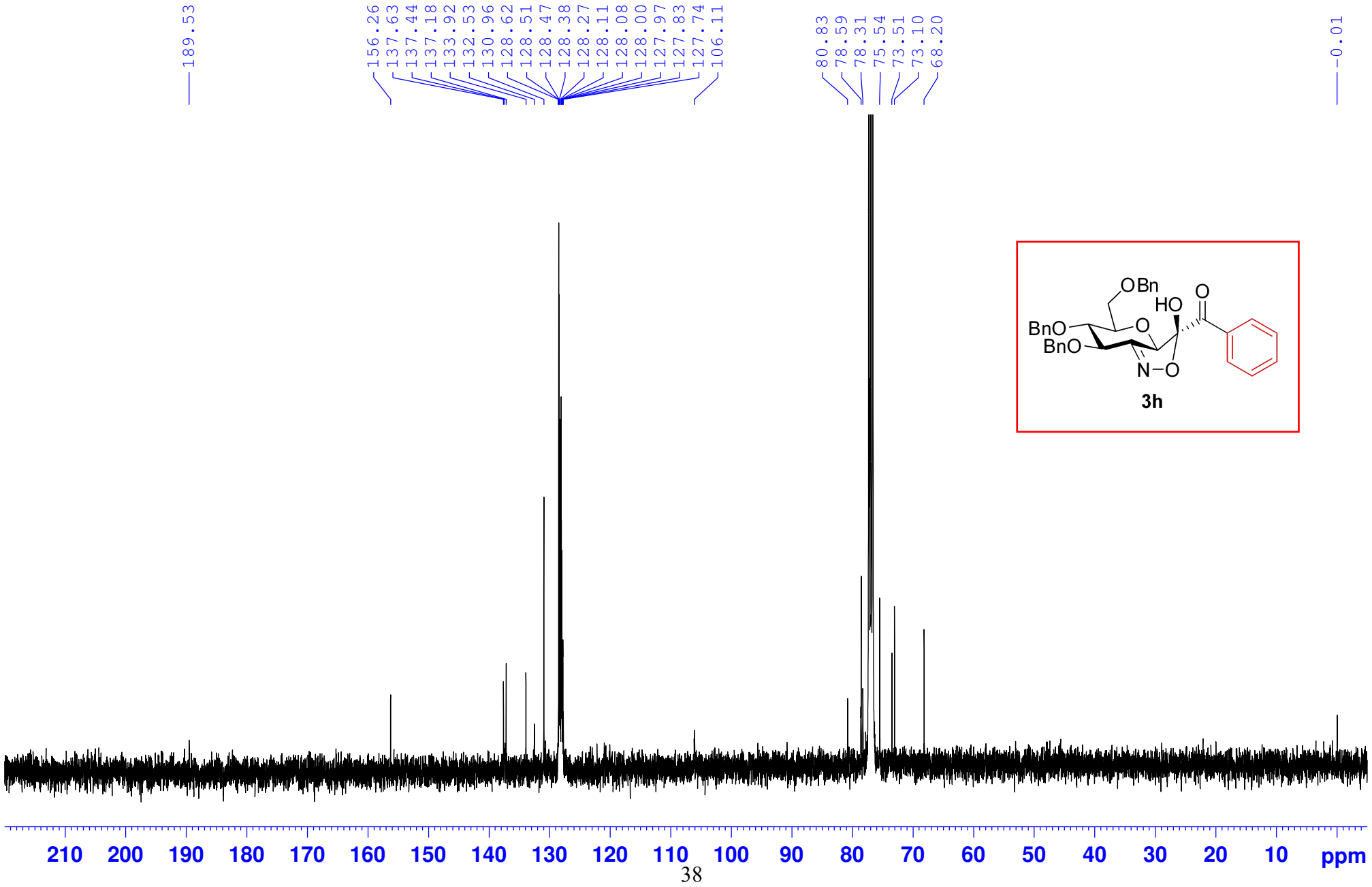
Compound 3g, 13C NMR, CDCl3, 400 MHz

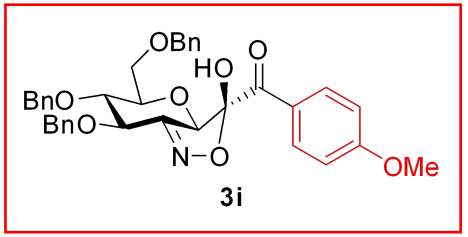


Compound 3h, 1H NMR, 400 MHz, CDCl3

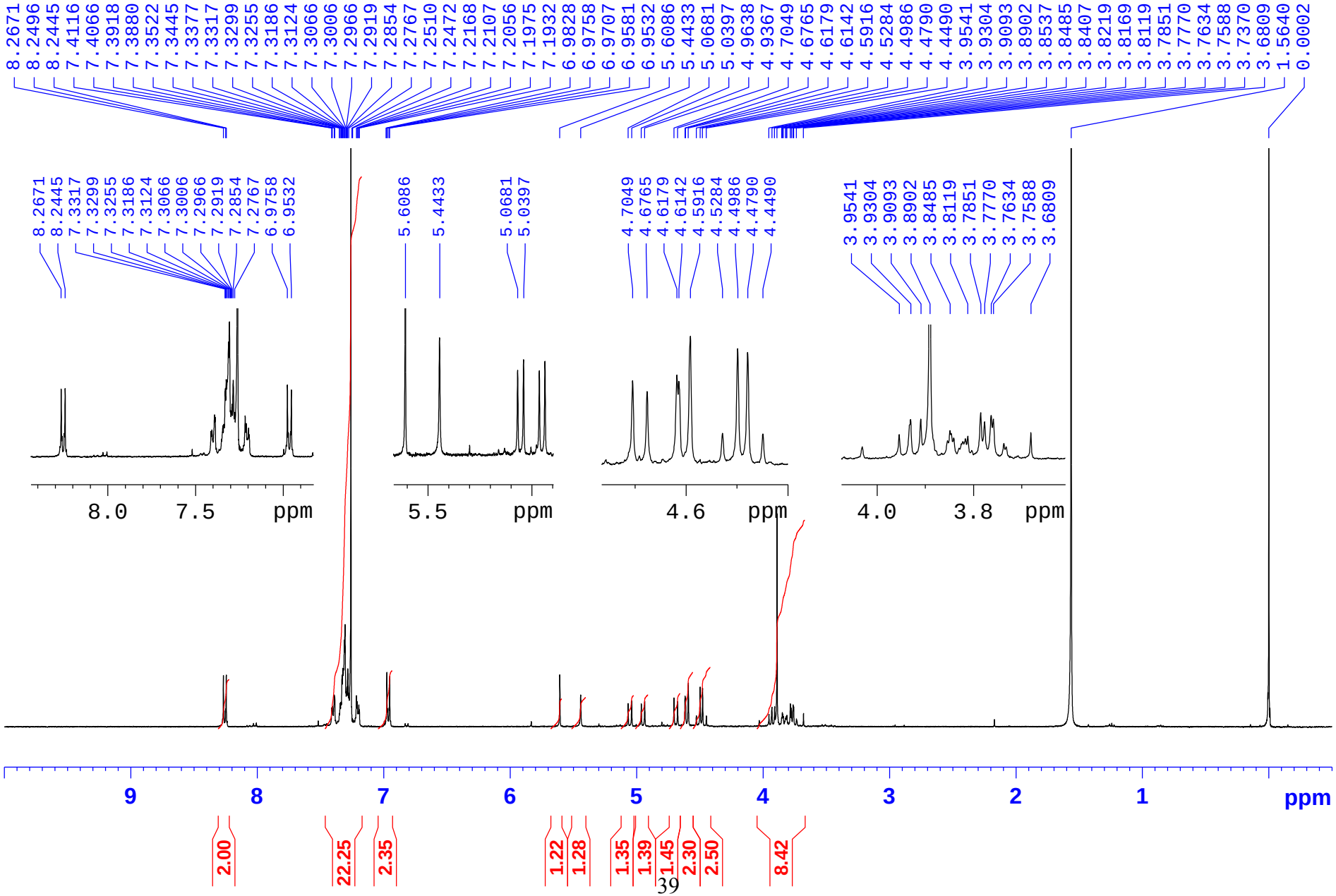


Compound 3h, 13C NMR, 400 MHz, CDCl3

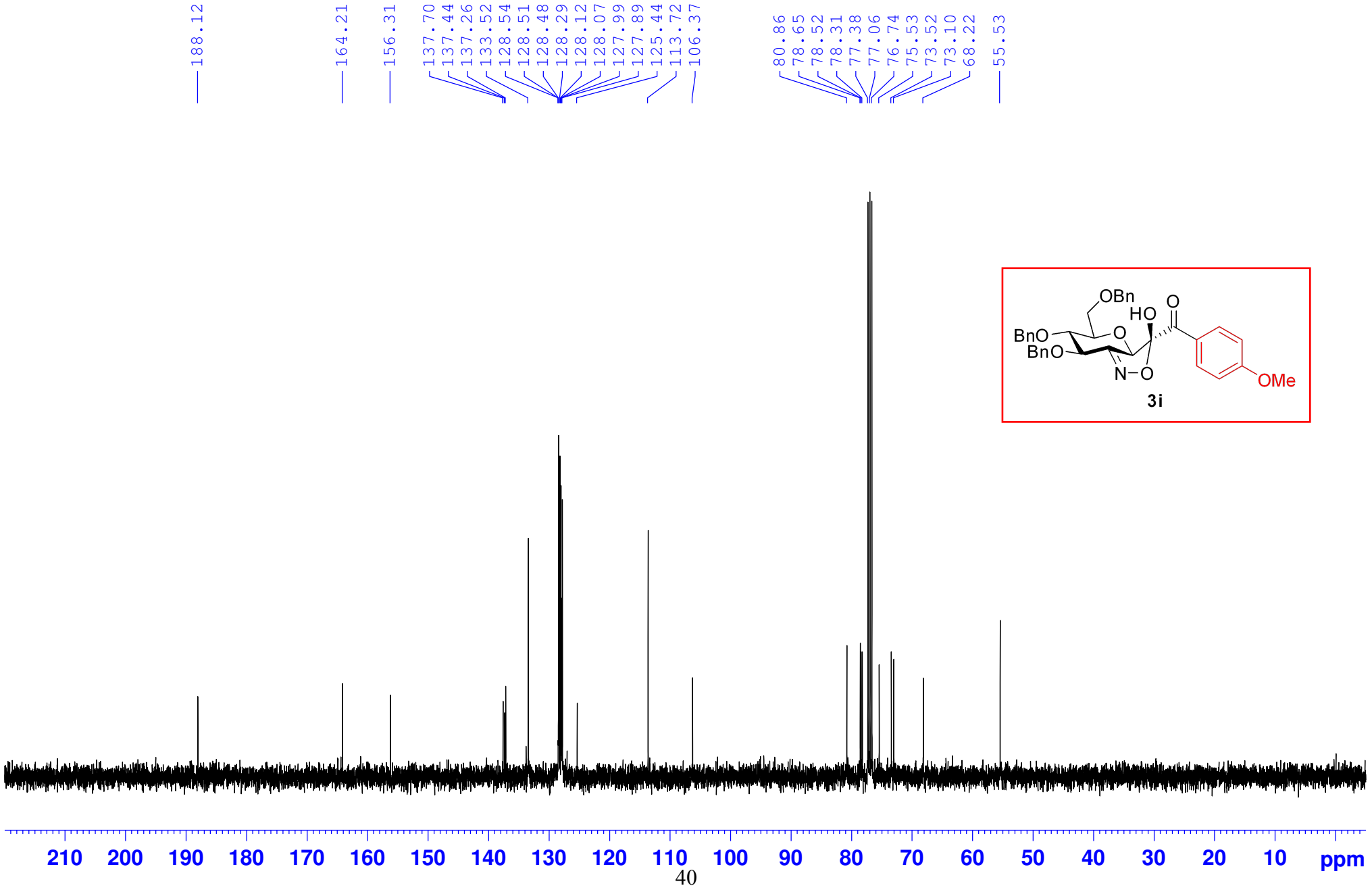




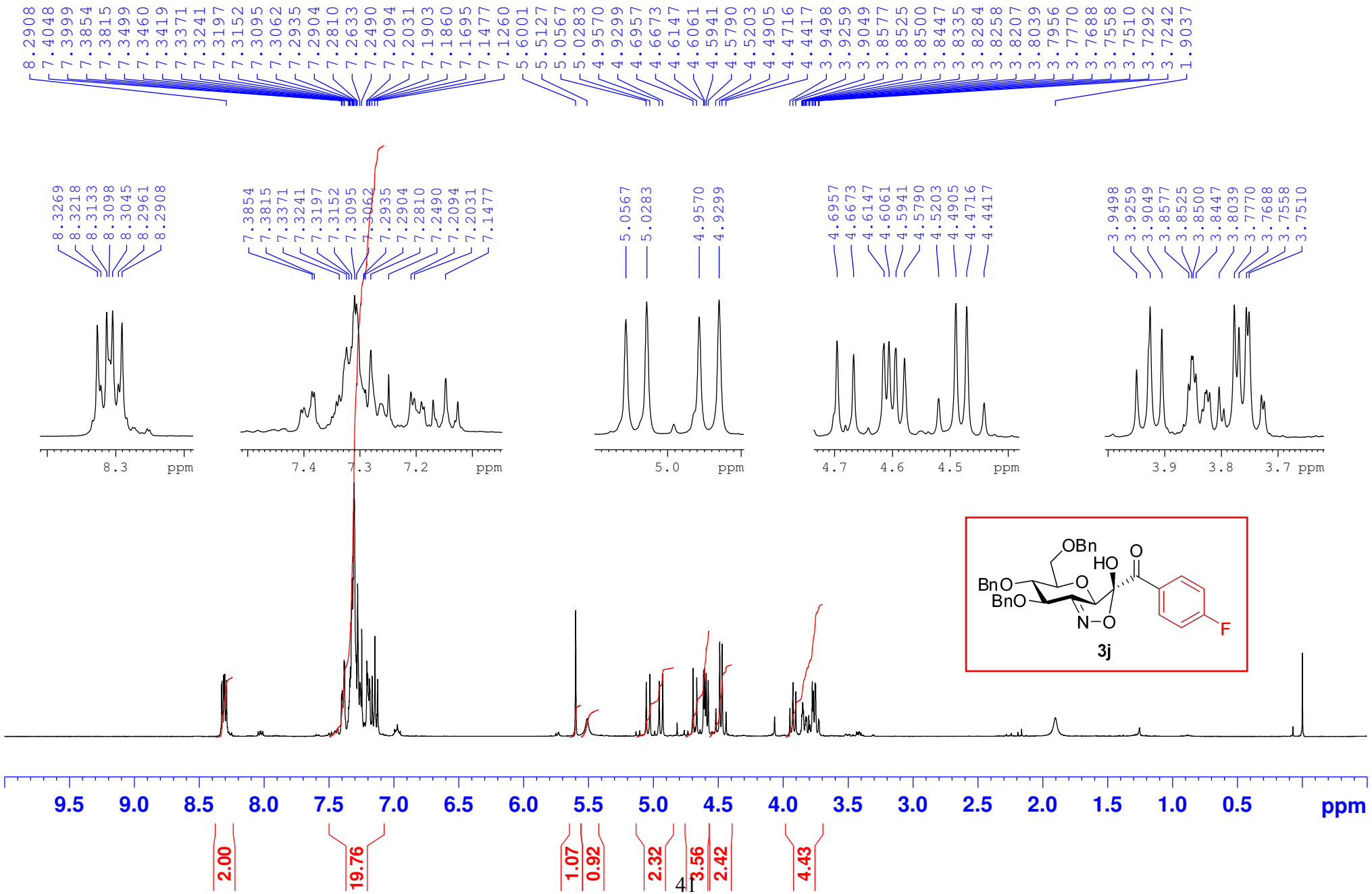
Compound 3i, 400 MHz, CDCl3,1H NMR



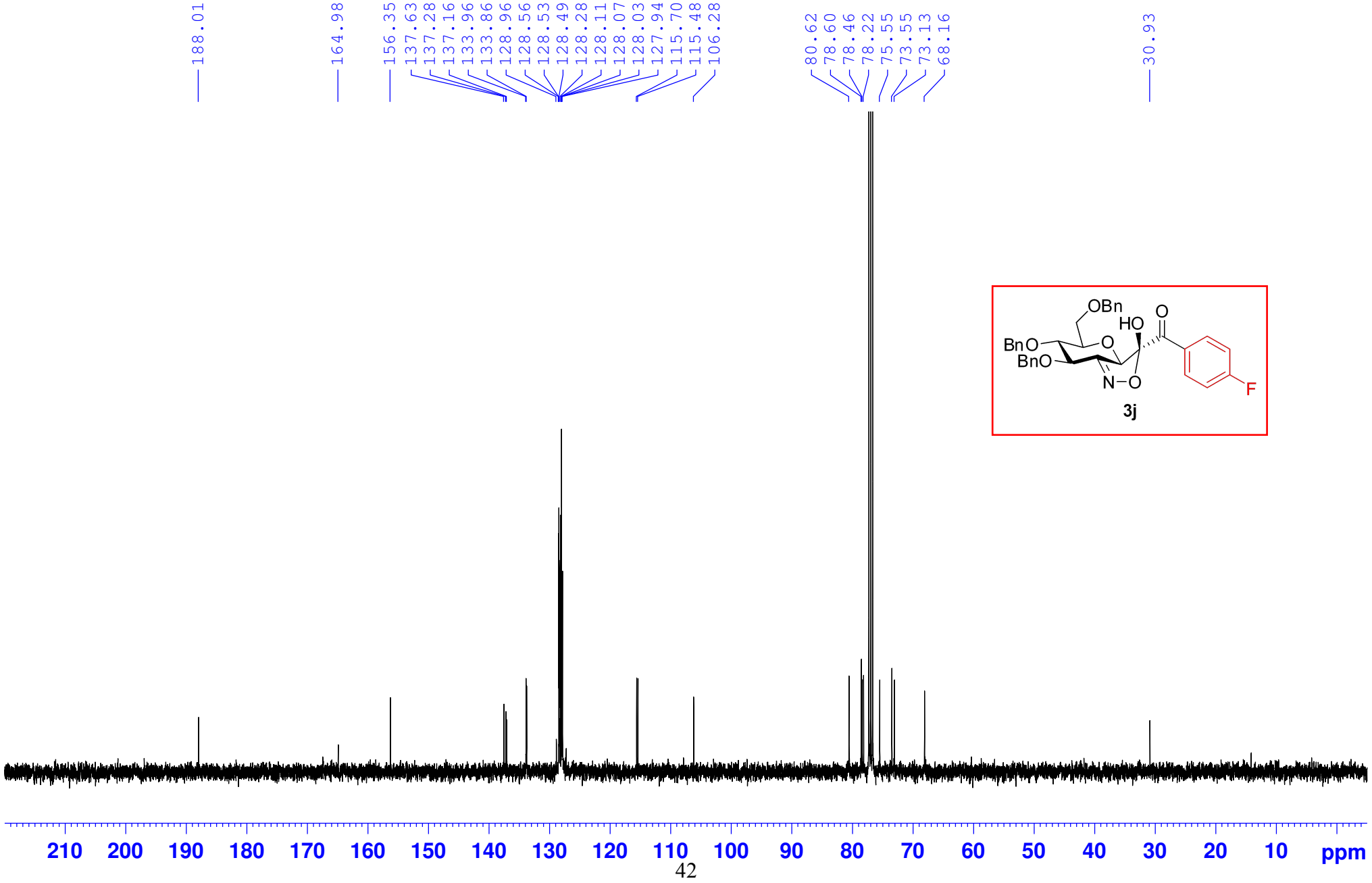
Compound 3i, 13C NMR, 400MHz, CDCl3



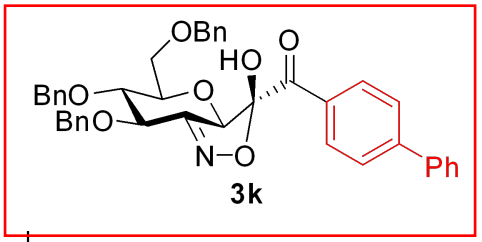
Compound 3j, 1H NMR, 400MHz, CDCl3



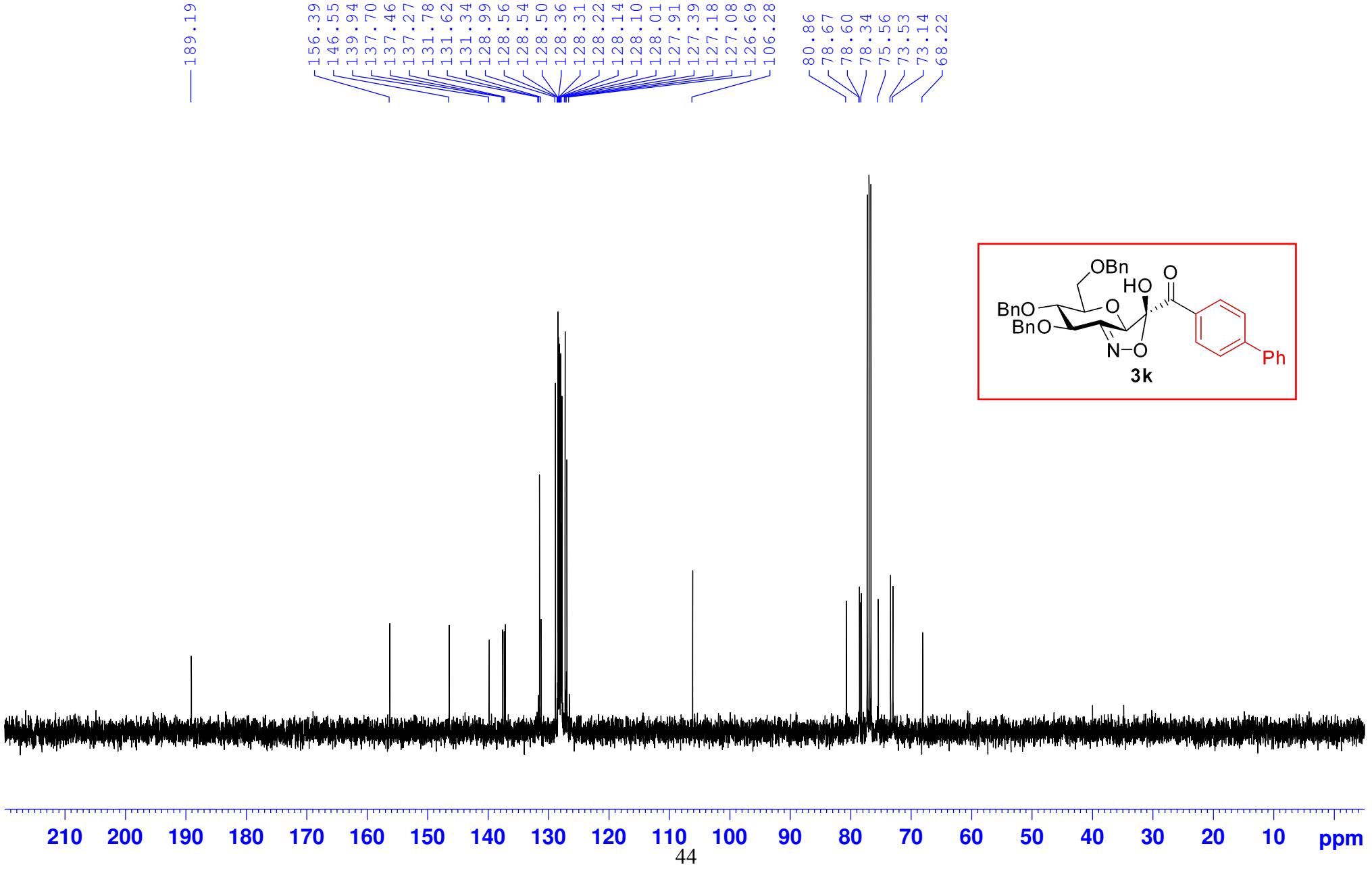
Compound 3j, 13C NMR, 400MHz, CDCl3



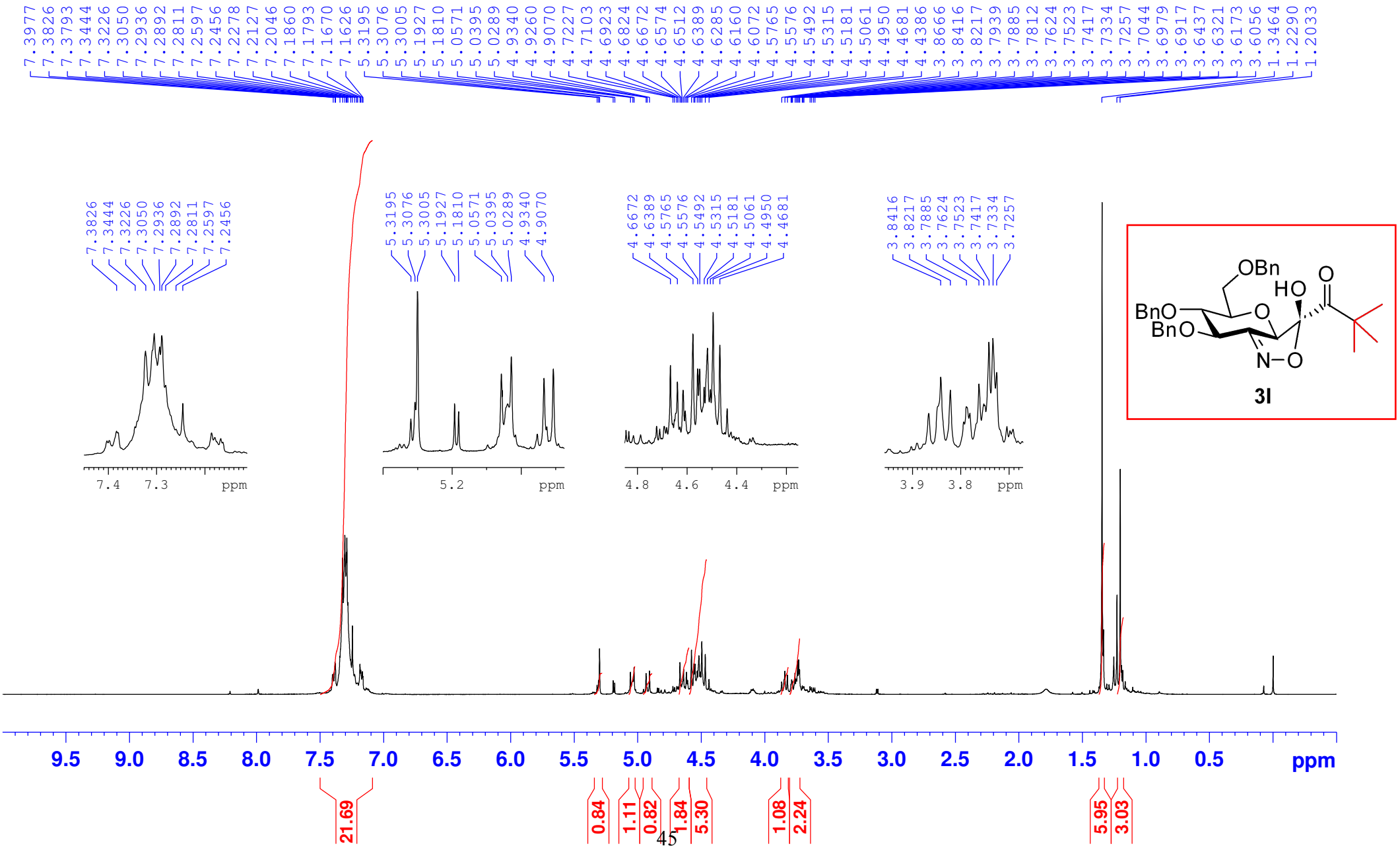
Compound 3k, 400 MHz, CDCl3,1H NMR



Compound 3k, 13C NMR, 400MHz, CDCl3



Compound 3l, 1H NMR, 400MHz, CDCl3



Compound 3l, 13C NMR, 400MHz, CDCl3