SUPPORTING INFORMATION

An Oxidative Entry into the Amido Trioxadecalin Ring System

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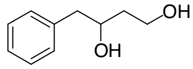
Experimental

General Experimental:

Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were recorded on Bruker Avance 300 spectrometers at 300 MHz and 75 MHz, respectively. The chemical shifts are given in parts per million (ppm) on the delta (δ) scale. The solvent peak or the internal standard tetramethylsilane were used as reference values. For ^{1}H NMR: $CDCl_3$ = 7.27 ppm, TMS = 0.00. For ^{13}C NMR: $CDCl_3$ = 77.23, TMS = 0.00. For proton data: s = singlet; d = doublet; t = triplet; q = quartet; p = pentet; dd = doublet of doublets, dt = doublet of triplets, ddt = doublet of doublets of triplets; br = broad; m = multiplet; app t = apparent triplet; app q = apparent quartet; app p = apparent pentet.

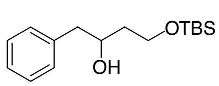
High resolution and low resolution mass spectra were recorded on a VG 7070 spectrometer. Infrared (IR) spectra were collected on a Mattson Gygnus 100 spectrometer.

Analytical thin layer chromatography (TLC) was performed on E. Merck pre-coated (25 nm) silica gel 60F-254 plates. Visualization was done under UV (245 nm). Flash column chromatography was preformed using ICN SiliTech 32-63 60μ silica gel. Reagent grade ethyl acetate and hexanes (commercial mixture) were purchased from EM Science and used as is for chromatography. Reagent grade methylene chloride (CH_2Cl_2), dichloroethane ($C_2H_4Cl_2$), acetonitrile (CH_3CN), benzene and toluene were distilled from CaH_2. Diethyl ether (Et_2O) and tetrahydrofuran (THF) were distilled from sodium benzophenone ketal prior to use. Anhydrous *N,N*-dimethylformamide (DMF), methanol (MeOH), dimethylformamide (DMF), methanol (MeOH), dimethyl sulfoxide (DMSO) were purchased from Aldrich and used as is. All reactions were conducted under nitrogen atmosphere, unless otherwise specified.



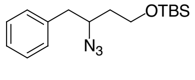
4-Phenylbutane-1,3-diol

Ozone was bubbled through a solution of 1-phenylpent-4-en-2-ol[1] (7.00 g, 43.1 mmol) in CH_2Cl_2 (50 mL) at -78 °C was bubbled O_3 until the reaction mixture sustained a dark blue color. Methanol (50 mL) was added to the solution at -78 °C followed by the slow addition of $NaBH_4$ (6.53 g, 172 mmol). The reaction was stirred at -78 °C for 1 hour then it was slowly warmed to room temperature and stirred for 4 hours. The reaction was concentrated under reduced pressure and extracted with EtOAc. The organic layers were combined, washed with water and brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (40% EtOAc in hexanes) to provide the desired product (5.21 g, 78%): ^1H NMR (300 MHz, $CDCl_3$) δ 7.30-7.22 (m, 5 H), 4.12-4.08 (m, 1 H), 3.89-3.82 (m, 2 H), 2.83-2.77 (m, 2 H), 2.28 (bs, 2 H), 1.80-1.75 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.9, 129.4, 128.6, 126.6, 73.1, 61.7, 44.3, 37.7; IR (neat) 3349, 3026, 2938, 1052 cm^{-1}; HRMS (EI) calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0993, found 166.0996.



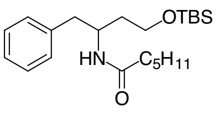
4-(*tert*-Butyldimethylsilanyloxy)-1-phenylbutan-2-ol

To a solution of 3-phenylpropane-1,2-diol (1.72 g, 10.2 mmol) in CH_2Cl_2 (50 mL) at room temperature was added imidazole (1.51 g, 22.4 mmol). The solution was stirred for 10 minutes then cooled to -42 °C. TBSCl (1.64 g, 11.2 mmol) was added to the reaction mixture which was stirred for two hours at -42 °C. Saturated aqueous NH_4Cl was added and the reaction mixture to warmed to room temperature. The reaction mixture was extracted with CH_2Cl_2. The organic layers were combined, washed with brine, dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (30% EtOAc in hexanes) to provided the desired product (2.63 g, 93%): ^1H NMR (300 MHz, $CDCl_3$) δ 7.32-7.18 (m, 5 H), 4.11-4.03 (m, 1 H), 3.92-3.85 (m, 1 H), 3.82-3.74 (m, 1 H), 3.32 (bs, 1 H), 2.85 (dd, J = 13.5, 7.0 Hz, 1 H), 2.73 (dd, J = 13.5, 6.2 Hz, 1 H), 1.72-1.60 (m, 2 H), 0.90 (s, 9 H), 0.07 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.8, 129.5, 128.4, 126.3, 72.8, 62.5, 44.0, 36.9, 25.8, 18.1, -5.5; IR (neat) 3450, 2953, 2857 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{28}O_2Si$ (M^+) 280.1858, found 280.1815.

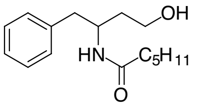


3-Azido-4-phenylbutoxy-*tert*-butyldimethylsilane

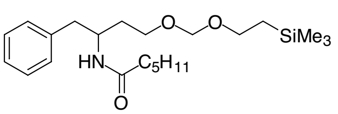
Warning: To avoid a possible explosion, extreme care should be taken to remove all CH_2Cl_2 after mesylate formation before NaN_3 is added. To a solution of 4-(*tert*-butyldimethylsilanyloxy)-1-phenylbutan-2-ol (9.36 g, 33.2 mmol) in CH_2Cl_2 (100 mL) was added triethylamine (13.4 g, 0.132 mol) and the solution was cooled to 0 °C. Methanesulfonyl chloride (5.70 g, 49.8 mmol) was added dropwise and the solution was stirred for 3.5 hours at 0 °C. Brine was added and the reaction was allowed to warm to room. The reaction mixture was extracted with hexanes. The organic layers were combined, washed with H_2O, dried with $MgSO_4$, filtered and concentrated under reduced pressure. The resulting oil was taken up in DMF (60 mL) and NaN_3 (10.3g, 0.158 mol) and tetrabutylammonium iodide (0.031 g, 0.080 mmol) were added. The solution was stirred at 55 °C for 14 hours. The reaction was allowed to cool to room temperature, H_2O was added and the reaction mixture was extracted with hexanes. The organic layers were combined, dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (5% EtOAc in hexanes) to provide the desired product (6.93 g, 68%): ^1H NMR (300 MHz, $CDCl_3$) δ 7.34-7.21 (m, 5 H), 3.77-3.71 (m, 1 H), 3.74-3.71 (m, 2 H), 2.85 (dd, J = 13.8, 6.0 Hz, 1 H), 2.84 (dd, J = 13.8, 7.8 Hz, 1 H), 1.78-1.76 (m, 1 H), 1.67-1.62 (m, 1 H), 0.89 (s, 9 H), 0.05 (s, 3 H), 0.04 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.5, 129.3, 128.5, 126.7, 60.8, 59.5, 41.1, 37.0, 25.9, 18.1, -5.4; IR (neat) 2955, 2930, 2857, 2100, 1812, 1742 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{27}N_3OSi$ ($M-C_4H_9$) 248.1201, found 248.1207.



Hexanoic acid 1-benzyl-3-*tert*-butyldimethylsilyloxypropylamide
To a solution of 3-azido-4-phenylbutoxy-*tert*-butyldimethylsilane (1.20 g, 3.93 mmol) in THF (20 mL) was added triphenylphosphine (1.24 g, 4.72 mmol) and the reaction mixture was stirred for 6 hours at room temperature. To the reaction mixture was added H_2O (4 mL). The reaction mixture was stirred for 8 hours at room temperature. To the reaction mixture was added triethylamine (1.19 g, 11.8 mmol) followed by hexanoyl chloride (1.05 g, 7.86 mmol). The reaction mixture was stirred for 6 hours at room temperature. The reaction mixture was extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (1.31 g, 88%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.19 (m, 5 H), 6.17 (d, *J* = 7.5 Hz, 1 H), 4.34-4.23 (m, 1 H), 3.83 (ddd, *J* = 10.5, 8.7, 4.5 Hz, 1 H), 3.72-3.64 (m, 1 H), 3.00 (dd, *J* = 13.5, 5.5 Hz, 1 H), 2.74 (dd, *J* = 13.5, 8.2 Hz, 1 H), 2.11 (t, *J* = 7.2 Hz, 2 H), 1.81-1.70 (m, 1 H), 1.67-1.51 (m, 3 H), 1.35-1.24 (m, 4 H), 0.92 (s, 9 H), 0.89 (t, *J* = 7.1 Hz, 3 H), 0.08 (s, 3 H), 0.07 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 138.4, 129.4, 128.3, 126.3, 60.6, 49.3, 40.0, 37.1, 34.8, 33.9, 31.2, 25.9, 24.4, 22.4, 13.9, -5.4; IR (neat) 3287, 2930, 2858, 1711, 1643, 1097 cm^{-1}; HRMS (EI) calcd. for $C_{21}H_{36}NO_2Si$ (M-CH_3) 362.2515, found 362.2521.

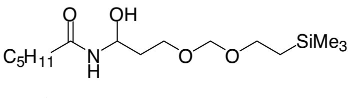


Hexanoic acid 1-benzyl-3-hydroxypropylamide (5)
To a solution of hexanoic acid 1-benzyl-3-*tert*-butyl-dimethyl-silanyloxy-propyl-amide (549 mg, 1.45 mmol) in THF (13 mL) at 0 °C was added tetrabutylammonium fluoride (TBAF) (1.14 g, 4.35 mmol). The reaction mixture was warmed to room temperature and stirred for 2 hours. H_2O was added the reaction mixture was extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50%EtOAc in hexanes) to provide the desired product (3.43 mg, 89%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.13 (m, 5 H), 5.52 (d, *J* = 8.4 Hz, 1 H), 4.43-4.34 (m, 1 H), 3.71 (bs, 1 H), 3.61-3.64 (m, 2 H), 2.87 (dd, *J* = 14.0, 6.0 Hz, 1 H), 2.73 (dd, *J* = 14.0, 7.8 Hz, 1 H), 2.13-2.06 (m, 2 H), 1.92-1.84 (m, 1 H), 1.48 (p, *J* = 7.2, 2 H), 1.36-1.27 (m, 1 H), 1.24-1.07 (m, 4 H), 0.82 (t, *J* = 6.9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.5, 137.5, 129.1, 128.6, 126.7, 58.4, 46.8, 41.2, 37.8, 36.6, 31.2, 25.4, 22.3, 13.9; IR (neat) 3280, 2929, 1647, 1621, 1544, 1054 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{25}NO_2$ (M^+) 263.1885, found 263.1886.



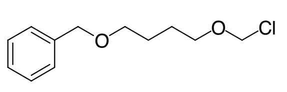
Hexanoic acid 1-benzyl-3-(2-trimethylsilylethoxymethoxy)propylamide (6)
To a solution of **5** (0.10 g, 0.38 mmol) in CH_2Cl_2 (2 mL) at room temperature was added *N,N*-diisopropylethylamine (DIPEA) (2 mL) followed by 2-(trimethylsilyl)ethoxymethyl chloride (0.13 g, 0.76 mmol). The reaction mixture was heated to 45 °C for 8 hours. The reaction mixture was cooled to room temperature and H_2O was added. The reaction mixture was extracted with CH_2Cl_2. The organic layers were combined, washed with 10% aqueous acid and brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting

oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (0.14 g, 92%): ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.29-7.16 (m, 5 H), 5.79 (d, *J* = 8.2 Hz, 1 H), 4.63 (s, 2 H), 4.35-4.23 (m, 1 H), 3.71-3.55 (m, 4 H), 2.89 (dd, *J* = 13.6, 6.0 Hz, 1 H), 2.79 (dd, *J* = 13.6, 8.1 Hz, 1 H), 2.10 (t, *J* = 8.1 Hz, 2 H), 1.88-1.77 (m, 1 H), 1.65-1.50 (m, 3 H), 1.33-1.17 (m, 4 H), 0.92 (t, *J* = 8.5 Hz, 2 H), 0.87 (t, *J* = 6.8 Hz, 3 H), 0.01 (s, 9 H); ^{13}C NMR (300 MHz, CDCl$_3$) δ 172.4, 137.9, 129.3, 128.3, 126.3, 94.9, 65.2, 48.4, 40.3, 36.9, 32.9, 31.3, 25.3, 22.3, 21.0, 18.0, 13.8, -1.5; IR (neat) 3282, 2954, 1639, 1549, 1060 cm^{-1}; HRMS (EI) calcd. for $C_{22}H_{39}NO_3Si$ (M$^+$) 393.2699, found 393.2706.



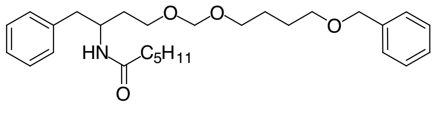
Hexanoic acid 1-hydroxy-3-(2-trimethylsilylethoxymethoxy)propylamide (7)

To a solution of **6** (0.11 g, 0.28 mmol) in dichloroethane (6 mL) at room temperature was added *N*-methylquinolinium hexafluorophosphate (NMQ) (4.0 mg, 0.01 mmol), NaOAc (0.22 g, 200 wt.%), $Na_2S_2O_3$ (0.22 g, 200 wt.%) and toluene (1 mL). Air was gently bubbled through the suspension and the solution was irradiated for 4 hours. The reaction mixture was filtered through a small plug of silica, and concentrated under reduced pressure. The resulting oil was purified by column flash chromatography (80% EtOAc in hexanes) to provide **7** (60 mg, 67%): ^{1}H NMR (300 MHz, CD$_2$Cl$_2$) δ 6.85 (br, 1H), 5.44-5.38 (m, 1 H), 4.65 (s, 2 H), 3.94 (d, *J* = 3.2 Hz, 1 H), 3.82-3.75 (m, 1 H), 3.67-3.54 (m, 3 H), 2.14 (t, *J* = 7.3 Hz, 2 H), 1.92-1.84 (m, 2 H), 1.63-1.53 (m, 2 H), 1.36-1.24 (m, 4 H), 0.96-0.87 (m, 5 H), 0.02 (s, 9 H); ^{13}C NMR (75 MHz, CD$_2$Cl$_2$) δ 175.1, 95.3, 73.8, 65.6, 63.9, 37.1, 34.7, 31.7, 25.4, 22.7, 18.4, 14.0, -1.4; IR (neat) 3299, 2955, 1654, 1538, 1249, 1060 cm^{-1}; LRMS (EI): *m/z* 320, 302, 262, 246, 232, 202, 188, 172.



4-Chloromethoxybutoxymethylbenzene

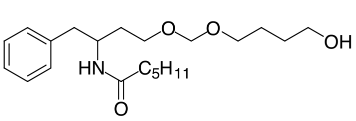
To a solution of 4-benzyloxybutanol (1.22 g, 6.78 mmol) in CH$_2$Cl$_2$ was added paraformaldehyde (0.200 g, 6.78 mmol) and the suspension was cooled to 0 °C. HCl (g) was bubbled through the reaction mixture until the paraformaldehyde was consumed (approximately 1 hour). The reaction mixture was place under HCl atmosphere and stirred for an additional 4 hours at 0 °C. Nitrogen was bubbled through the solution to remove the HCl, the solution was dried with NaSO$_4$, filtered, concentrated under reduced pressure providing the desired product in quantitative yield. The product was used without purification: crude ^{1}H NMR (300 MHz, C$_6$D$_6$) δ 7.28-7.15 (m, 5 H), 5.07 (s, 2 H), 4.35 (s, 2 H), 3.43-3.38 (m, 2 H), 3.32-3.25 (m, 2 H), 1.63-1.55 (m, 4 H).



Hexanoic acid 1-benzyl-3-(4-benzyloxybutoxymethoxy)propylamide

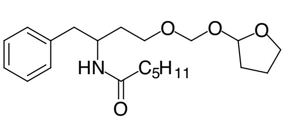
To a solution of **5** (0.20 g, 0.77 mmol) in CH$_2$Cl$_2$ (2 mL) at room temperature was added *i*-Pr$_2$NEt (2 mL) followed by 4-chloromethoxybutoxymethyl-benzene (0.30 g, 1.2 mmol). The reaction mixture was heated to 45 °C for 8 hours. The reaction mixture was cooled to room temperature and H$_2$O was added. The reaction mixture was extracted with CH$_2$Cl$_2$. The organic layers were combined, washed with 10% aqueous acid and brine, dried with

$NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (0.32 g, 93%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.17 (m, 10 H), 5.65 (d, *J* = 8.2 Hz, 1 H), 4.64 (s, 2 H), 4.50 (s, 2 H), 4.29 (br, 1 H), 3.69-3.47 (m, 6 H), 2.90 (dd, *J* = 13.6, 5.9 Hz, 1 H), 2.77 (dd, *J* = 13.6, 7.3 Hz, 1 H), 2.15-2.10 (m, 2 H), 1.89-1.79 (m, 1 H), 1.72-1.52 (m, 7 H), 1.32-1.18 (m, 4 H), 0.89 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.8, 138.9, 138.3, 129.8, 128.7, 128.6, 127.9, 127.8, 126.7, 95.8, 73.2, 70.4, 68.1, 65.4, 48.8, 40.8, 37.3, 33.4, 31.7, 26.8, 26.8, 25.7, 22.7, 17.2; IR (neat) 3290, 2928, 1641, 1547, 1113 cm^{-1}; HRMS (EI) calcd. for $C_{21}H_{34}NO_4$ (M-C_7H_7) 364.2488, found 364.2495.



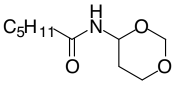
Hexanoic acid 1-benzyl-3-(4-hydroxybutoxymethoxy)propylamide

To a solution of hexanoic acid 1-benzyl-3-(4-benzyloxybutoxymethoxy)propylamide (0.22 g, 0.49 mmol) in ethanol at room temperature was added Pd/C (44 mg, 20 wt.%). The reaction mixture was placed under hydrogen atmosphere and stirred at room temperature for 8 hours. The reaction mixture was run through a pad of celite, concentrated under reduced pressure and purified by flash chromatography (80% EtOAc in hexanes) to provide the desired product (0.17 g, 98% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.17 (m, 5 H), 5.67 (d, *J* = 8.0 Hz, 1 H), 4.66 (s, 2 H), 4.38-4.26 (m, 1 H), 3.69-3.51 (m, 6 H), 2.91 (dd, *J* = 13.7, 6.2 Hz, 1 H), 2.79 (dd, *J* = 13.7, 7.1 Hz, 1 H), 2.26 (bs, 1 H), 2.11 (t, *J* = 6.7 Hz, 2 H), 1.91-1.82 (m, 1 H), 1.71-1.49 (m, 7 H), 1.34-1.17 (m, 4 H), 0.86 (t, *J* = 6.7 Hz, 3 H); ^{13}C NMR (300 MHz, $CDCl_3$) δ 172.7, 137.9, 129.5, 138.4, 126.4, 95.5, 68.0, 65.2, 62.5, 48.3, 40.7, 36.9, 33.4, 31.4, 29.8, 26.4, 25.4, 33.4, 13.9; IR (neat) 3289, 2930, 1643, 1549, 1046 cm^{-1}; HRMS (EI) calcd. for $C_{21}H_{36}NO_4$ (M+H) 366.2644, found 366.2632.



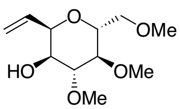
Hexanoic acid 1-benzyl-3-(tetrahydrofuran-2-yloxymethoxy)propylamide (8)

To a solution of hexanoic acid 1-benzyl-3-(4-hydroxybutoxymethoxy)propylamide (0.17 g, 0.48 mmol) in cyclohexane was added iodobenzene diacetate (0.34 g, 1.1 mmol) and iodine (96 mg, 0.76 mmol). The reaction mixture was irradiated for 2 hours. The reaction mixture was extracted with EtOAc, washed with saturated aqueous $Na_2S_2O_3$ and brine, dried with $NaSO_4$, filtered, concentrated under reduced pressure and purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product as a mixture of diastereomers (0.10 g, 59%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.17 (m, 5 H), 5.80 (d, *J* = 7.9 Hz, 1 H), 5.36-5.32 (m, 1 H), 4.90-4.85 (m, 1 H), 4.64-4.58 (m, 1 H), 4.32-4.24 (m, 1 H), 3.96-3.81 (m, 1 H), 3.79-3.71 (m, 1 H), 3.70-3.62 (m, 1 H), 3.56-3.48 (m, 1 H), 2.96-2.86 (m, 1 H), 2.81-2.74 (m, 1 H), 2.13-2.00 (m, 2 H), 1.99-1.75 (m, 5 H), 1.67-1.52 (m, 3 H), 1.34-1.15 (m, 4 H), 0.88 (t, *J* = 6.7 Hz, 3 H); ^{13}C NMR (300 MHz, $CDCl_3$) δ 172.6, 138.1, 129.4, 129.1, 126.3, 101.5, 92.7, 67.3, 65.7, 48.8, 48.4, 40.4, 36.9, 32.7, 31.4, 25.4, 23.5, 23.2, 13.9; IR (neat) 3289, 2929, 1614, 1544 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{28}NO_4$ (M-C_2H_5) 334.2018, found 334.2036.



Hexanoic acid 1,3-dioxan-4-ylamide (9)

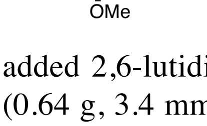
To a solution of **8** (90 mg, 0.25 mmol) in dichloroethane (6 mL) at room temperature were added NMQPF$_6$ (3.5 mg, 0.01 mmol), NaOAc (0.18 g, 200 wt.%), $Na_2S_2O_3$ (0.18 g, 200 wt.%), 4 Å molecular sieves (0.18 g, 200 wt.%) and toluene (1 mL). Air was gently bubbled through the suspension while it was irradiated for 4 hours. The reaction mixture was filtered through a small plug of silica, and concentrated under reduced pressure. The resulting oil was purified by column flash chromatography (50% EtOAc in hexanes) to provide the desired product (39 mg, 79%): ^{1}H NMR (300 MHz, CD_2Cl_2) δ 5.92 (br, 1 H), 5.43 (td, J = 10.0, 2.8 Hz, 1 H), 5.05 (d, J = 6.5 Hz, 1 H), 4.83 (d, J = 6.5 Hz, 1 H), 4.15 (app dd, J = 11.6, 4.9 Hz, 1 H), 3.79 (td, J = 11.7, 2.8 Hz, 1 H), 2.22 (t, J = 7.3 Hz, 2 H), 1.91-1.78 (m, 1 H), 1.75-1.60 (m, 3 H), 1.35-1.26 (m, 4 H), 0.90 (t, J = 6,9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 92.8, 77.2, 65.9, 36.7, 32.1, 31.3, 25.0, 22.4, 13.9; IR (neat) 3325, 2927, 1661, 1540, 1015 cm^{-1}; HRMS (EI) calcd. for $C_{10}H_{19}NO_3$ (M^+) 201.1365, found 201.1361.



4,5-Dimethoxy-6-methoxymethyl-2-vinyltetrahydropyran-3-ol

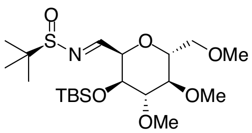
Preparation of 0.010 M trivinylalane:[2] To a suspension of $AlCl_3$ (4.44 g, 33.3 mmol) in CH_2Cl_2 (233 mL) at room temperature was added vinylmagnesium bromide (1 M in THF, 100 mL, 100 mmol) drop wise, over a period of 1 hour. The solution was cooled when necessary to prevent reflux and stirred at room temperature for 4 hours.

Synthesis of 4,5-dimethoxy-6-methoxymethyl-2-vinyltetrahydropyran-3-ol: To a solution of **10** (3.00 g, 16.0 mmol) in CH_2Cl_2 (10 mL) cooled to 0 °C was added dimethyldioxirane[3-5] (0.2 M, 76.8 mL, 17.2 mmol) and the solution was stirred for 30 minutes. The solution was concentrated under reduced pressure, taken up in CH_2Cl_2 (60 mL), and added drop wise over a period of 1 hour to a solution of trivinylalane (0.010 M, 333 mL) cooled to -60 °C. The reaction mixture was allowed to slowly warm to room temperature and stir for 8 hours. The reaction was carefully quenched with H_2O, 10% aqueous HCl was added until the aluminum salts disappeared, and the solution was extracted with ether. The organic layers were combined, washed with 10% HCl and brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (60% EtOAc) to provide the desired product (2.52 g, 62 %): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.05 (ddd, J = 16.2, 10.8, 5.4 Hz, 1 H), 5.42 (ddd, J = 17.5, 1.7, 1.7 Hz, 1 H), 5.37 (ddd, J = 10.7, 1.7, 1.7 Hz, 1 H), 4.46 (ddd, J = 5.1, 1.6, 1.6 Hz, 1 H), 3.83 (app dt, J = 7.8, 4.1 Hz, 1 H), 3.71 (dd, J = 7.6, 5.0 Hz, 1 H), 3.60 (dd, J = 10.0, 4.7 Hz, 1 H), 3.59 (dd, J = 10.4, 3.7 Hz, 1 H), 3.59 (s, 3 H), 3.50 (s, 3 H), 3.44 (s, 3 H), 3.34 (app t, J = 7.2 Hz, 1 H), 3.28 (app t, J = 7.2 Hz, 1 H), 2.28 (bs, 1 H); ^{13}C (125 MHz, $CDCl_3$) δ 132.7, 119.2, 82.0, 78.8, 73.9, 72.8, 71.0, 70.5, 59.9, 59.4, 59.2; IR (neat) 3450, 2930, 1081 cm^{-1}; $[\alpha]^{25}_{D}$ +98.87 (c 3.20, $CHCl_3$); HRMS (EI) calcd. for $C_{11}H_{20}O_5$ (M^+) 232.1311, found 232.1303.



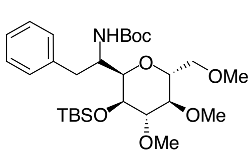
tert-Butyl-(4,5-dimethoxy-6-methoxymethyl-2-vinyltetrahydropyran-3-yloxy)dimethylsilane (11)

To a solution of 4,5-dimethoxy-6-methoxymethyl-2-vinyltetrahydropyran-3-ol (0.40 g, 1.6 mmol) in CH_2Cl_2 (1.6 mL) was added 2,6-lutidine (0.35 g, 3.2 mmol). The solution was cooled to 0 °C and TBSOTf (0.64 g, 3.4 mmol) was added dropwise. The reaction mixture was allowed to warm to room temperate and was stirred for 30 minutes. The reaction was quenched with saturated aqueous NH_4Cl and extracted with CH_2Cl_2. The organic layers were combined, washed with brine, dried $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (5% EtOAc in hexanes) to provide the desired product (0.43 g, 77%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.08 (ddd, *J* = 17.6, 11.0, 3.4 Hz, 1 H), 5.45-5.36 (m, 2 H), 4.39-4.36 (m, 1 H), 3.77-3.72 (m, 1 H), 3.63-3.53 (m, 3 H), 3.58 (s, 3 H), 3.53 (s, 3 H), 3.40 (s, 3 H), 3.17 (app t, *J* = 9.0 Hz, 1 H), 3.10 (app t, *J* = 9.0 Hz, 1 H), 0.89 (s, 9 H), 0.11 (s, 3 H), 0.05 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 131.9, 119.1, 85.2, 80.3, 76.3, 72.7, 71.6, 71.5, 61.2, 60.4, 59.2, 25.7, 17.9, -4.7, -4.9; IR (neat) 2931, 2894, 1114, 1093 cm^{-1}; $[\alpha_D]^{25}{}_D$ +81.5 (*c* 3.2, $CHCl_3$); HRMS (ES) calcd. for (M+K) 385.1813, found 385.1800.



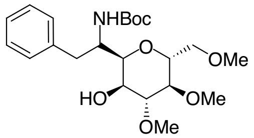
2-Methyl-propane-2-sulfinic acid 3-(tert-butyldimethylsilyloxy)-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-ylmethyleneamide (12)

A solution of **11** (0.387 g, 1.12 mmol) in CH_2Cl_2 was cooled to -78 °C and ozone was gently bubbled through the solution until it sustained a deep purple. Triphenylphosphine (0.587 g, 2.24 mmol) was added at -78 °C and the reaction mixture was warmed to room temperature and stirred for 2 hours. The reaction mixture was concentrated under reduced pressure, taken up in THF (4 mL) and at room temperature Ti(O*i*Pr)$_4$ (1.59 g, 5.60 mmol) was added followed by the addition of (R$_s$)-*tert*-butanesulfinamide (0.245 g, 2.00 mmol). The reaction mixture was stirred for 4 hours at room temperature. The reaction mixture was poured into an equal volume of brine, filtered through a pad of Celite and extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, concentrated under reduced pressure and purified by flash chromatography (20% EtOAc in hexanes) to provide the desired product (0.396 g, 71 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.34 (d, *J* = 2.3 Hz, 1 H), 4.60 (dd, *J* = 6.7, 2.3 Hz, 1 H), 3.95 (m, 2 H), 3.62-3.55 (m, 2 H), 3.57 (s, 3 H), 3.53 (s, 3 H), 3.41 (s, 3 H), 3.42 (app t, *J* = 9.2 Hz, 1 H), 3.09 (app t, *J* = 9.4 Hz, 1 H), 1.12 (s, 9 H), 0.91 (s, 9 H), 0.14 (s, 3H), 0.12 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 166.3, 85.6, 80.0, 76.6, 73.8, 72.8, 71.1, 61.2, 60.4, 59.1, 57.1, 25.8, 22.4, 18.0, -4.7, -4.9; IR (neat) 2929, 1712, 1095 cm^{-1}; $[\alpha]^{25}{}_D$ + 28.27 (*c* 0.53, $CHCl_3$); HRMS (EI) calcd. for ($M-CH_3$) 436.2189 found 436.2170.



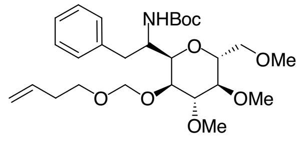
1,3-(*tert*-Butyldimethylsilyloxy)-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl-2-phenylethylcarbamic acid *tert*-butyl ester (14)

To a solution of **12** (0.380 g, 0.811 mmol) in CH_2Cl_2 cooled to -78 °C was treated with benzylmagnesium chloride (1 M in diethylether, 1.63 mL, 1.63 mmol) and the reaction mixture was stirred at -78 °C for 1 hour. The reaction was quenched with saturated aqueous NH_4Cl, warmed to room temperature and extracted with CH_2Cl_2. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered and concentrated under reduced pressure. The resulting oil was taken up in methanol (5 mL) and aqueous HCl (4 M, 2.00 mL, 8.16 mmol) was added. The reaction mixture was stirred for 1 hour at room temperature. The reaction was quenched with saturated aqueous $NaHCO_3$ and extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was taken up in CH_2Cl_2 (3 mL), triethylamine (2 mL) and di-*tert*-butyl dicarbonate (0.712 g, 3.26 mmol) were added and the solution was heated to 43°C for 8 hours. The reaction was cooled to room temperature, extracted with CH_2Cl_2, washed with 5 % aqueous HCl and brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (0.270 g, 68 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.17 (m, 5 H), 4.66 (br, 1 H), 4.17 (br, 1 H), 3.83 (dd, *J* = 7.6, 5.1 Hz, 1 H), 3.74 (app t, *J* = 5.5 Hz, 1 H), 3.75-3.63 (m, 1H), 3.54 (s, 3 H), 3.54-3.48 (m, 2 H), 3.48 (s, 3 H), 3.40 (s, 3H), 3.30 (d, *J* = 5.5 Hz, 1 H), 3.25 (d, *J* = 5.5, 1 H), 3.23-3.17 (m, 1 H), 2.52 (br, 1 H), 1.32 (s, 9 H), 0.93 (s, 9 H), 0.14 (s, 3 H), 0.09 (s, 3 H); ^{13}C (75 MHz, $CDCl_3$) δ 155.1, 138.4, 129.6, 128.2, 126.1, 84.0, 79.7, 77.6, 76.5, 73.2, 72.4, 71.5, 60.2, 59.4, 59.1, 51.8, 34.1, 28.3, 26.0, 18.0, -4.4, -4.6; IR (neat) 2930, 1710, 1109, 1030 cm^{-1}; $[\alpha]^{25}_D$ +36.49 (*c* 0.57, $CHCl_3$); HRMS (EI) calcd. for $C_{24}H_{40}N0_6Si$ (M-C_4H_9O) 466.2625, found 466.2261.



1-(3-Hydroxy-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl)-2-phenylethylcarbamic acid *tert*-butyl ester

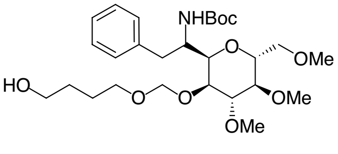
To a solution of **14** (230 mg, 0.431 mmol) in THF (5 mL) was added TBAF (340 mg, 1.29 mmol). The reaction mixture was stirred for 1 hour at room temperature, quenched with H_2O and extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (80% EtOAc in hexanes) to provide the desired product (140 mg, 76 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.18 (m, 5 H), 4.82 (br, 1 H), 4.12 (br, 1 H), 4.03-3.94 (m, 1 H), 3.84 (br, 1 H), 3.72 (m, 1 H), 3.66 (dd, *J* = 10.3, 6.9 Hz, 1 H), 3.51-3.47 (m, 2 H), 3.47 (s, 3 H), 3.41 (s, 3 H), 3.40 (s, 3 H), 3.28 (app t, *J* = 3.4 Hz, 1 H), 2.98 (d, *J* = 6.6, 2 H), 1.61 (bs, 1 H), 1.40 (s, 9 H); ^{13}C (75 MHz, $CDCl_3$) δ 155.8, 138.3, 129.3, 128.1, 126.1, 79.0, 78.5, 76.6, 74.0, 70.2, 69.2, 68.5, 58.8, 58.2, 58.0, 53.8, 37.3, 28.2; IR (neat) 3360, 2930, 1712, 1114 cm^{-1}; $[\alpha]^{25}_D$ +7.41 (*c* 0.27, $CHCl_3$); HRMS (EI) calcd. for $C_{18}H_{26}NO_6$ (M-C_4H_9O) 352.1760 found 352.1768.



1-(3-But-3-enyloxymethoxy-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl)-2-phenylethylcarbamic acid *tert*-butyl ester

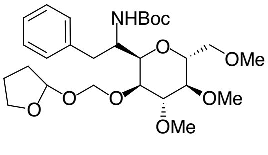
To a solution of 1-(3-hydroxy-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl)-2-phenylethylcarbamic acid *tert*-butyl ester (428 mg, 1.01 mmol) in CH_2Cl_2 (5 mL) and DIPEA (3 mL) was added 4-chloromethoxy-but-1-ene[6] (608 mg, 5.05 mmol) and the reaction mixture was heated to 40 °C and stirred for 8 hours. The solution was cooled to room temperature, saturated aqueous NH_4Cl was added and the reaction mixture was extracted with CH_2Cl_2. The organic layers were combined, washed with 5% aqueous HCl, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (497 mg, 97%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.18 (m, 5 H), 5.87-5.74 (m, 1 H), 5.12-5.02 (m, 2

H), 4.93 (d, J = 7.3 Hz, 1 H), 4.79 (d, J = 6.8 Hz, 1 H), 4.69 (d, J = 6.9 Hz, 1 H), 4.07 (br, 1 H), 3.89-3.79 (m, 2 H), 3.73 (br, 1 H), 3.70-3.55 (m, 3 H), 3.52-3.45 (m, 2 H), 3.46 (s, 3 H), 3.45 (s, 3 H), 3.39 (s, 3 H), 3.15 (app t, 1 H), 3.04 (dd, J = 13.6, 6.4 Hz, 1 H), 2.86 (br, 1 H), 2.32 (m, 2 H), 1.34 (s, 9 H); ^{13}C (75 MHz, $CDCl_3$) δ 155.3, 138.3, 135.1, 129.6, 128.3, 126.2, 116.6, 95.9, 78.1, 75.9, 73.5, 71.4, 70.7, 67.7, 65.6, 60.0, 59.1, 58.8, 58.7, 52.0, 34.1, 32.8, 28.3; IR (neat) 2930, 1720, 1172 cm^{-1}; $[\alpha]^{25}_D$ +18.7 (*c* 3.2, $CHCl_3$); HRMS (EI) calcd. for $C_{27}H_{44}NO_8$ (M+H) 510.3067, found 510.3079.



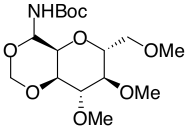
1,3-(4-Hydroxybutoxymethoxy)-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl-2-phenylethylcarbamic acid *tert*-butyl ester

To a solution of 1-(3-but-3-enyloxymethoxy-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl)-2-phenylethylcarbamic acid *tert*-butyl ester (481 mg, 0.944 mmol) in THF (30 mL) cooled to -10° C was added BH_3•THF (4.72 mL, 1 M, 4.72 mmol) and the reaction mixture was stirred for 4 hours at -10 °C. The excess borane was quenched with H_2O followed by the addition of NaOH/HOOH solution (20% NaOH/30% HOOH, 1:1 by volume) (5 mL) and the reaction mixture was stirred for 2 hours at room temperature. Saturated aqueous NaS_2O_3 was added and the reaction mixture was stirred for 1 hour at room temperature. The reaction mixture was extracted with EtOAc. The organic layers were combined, washed with brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (461 mg, 93%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27-7.17 (m, 5 H), 4.95 (br, 1 H), 4.80 (d, J = 6.9 Hz, 1 H), 4.69 (d, J = 6.9 Hz, 1 H), 4.05 (br, 1 H), 3.84 (br, 1 H), 3.81 (dd, J = 5.6, 3.6 Hz, 1 H), 3.73 (br, 1 H), 3.66-3.37 (m, 7 H), 3.46 (s, 3 H), 3.45 (s, 3 H), 3.39 (s, 3 H), 3.19 (br, 1 H), 3.04 (dd, J = 13.6, 6.5 Hz, 1 H), 2.85 (br, 1 H), 1.96 (br, 1 H), 1.69-1.63 (m, 4 H), 1.36 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.4, 138.3, 129.6, 128.2, 126.2, 95.7, 78.0, 77.4, 75.8, 73.5, 73.5, 71.3, 70.5, 68.4, 62.5, 59.1, 58.9, 58.7, 52.0, 37.3, 29.6, 28.3, 26.2; IR (neat) 3452, 2932, 1707, 1107 cm^{-1}; $[\alpha]^{25}_D$ +14.2 (*c* 2.4, $CHCl_3$); HRMS (EI) calcd. for $C_{20}H_{38}NO_9$ (M-C_7H_7) 436.2547, found 436.2553.



1-(4,5-Dimethoxy-6-methoxymethyl)-3-(tetrahydrofuran-2-yloxymethoxy)-tetrahydropyran-2-yl-2-phenylethylcarbamic acid *tert*-butyl ester (15)

To a solution of 1,3-(4-hydroxybutoxymethoxy)-4,5-dimethoxy-6-methoxymethyltetrahydropyran-2-yl-2-phenylethylcarbamic acid *tert*-butyl ester (47 mg, 0.09 mmol) in cyclohexane was added iodobenzene diacetate (63 mg, 0.20 mmol) and iodine (18 mg, 0.14 mmol) at room temperature. The reaction mixture was irradiated for 2 hours. The reaction mixture was extracted with EtOAc, washed with saturated aqueous $NaSO_4$ and brine, dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (30 mg, 64%) as a mixture of diastereomers: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.15 (m, 5 H), 5.41 (m, 0.6 H), 5.37 (m, 0.4 H), 4.98 (d, J = 6.8 Hz, 0.4 H), 4.97 (d, J = 6.8 Hz, 0.6 H), 4.91 (br, 1 H), 4.77 (d, *J* = 6.8 Hz, 0.4 H), 4.65 (d, *J* = 6.8, 0.6 H), 4.10-4.02 (m, 1 H), 3.87-3.70 (m, 5 H), 3.55-3.40 (m, 4 H), 3.44 (s, 3 H), 3.43 (s, 3 H), 3.37 (s, 3 H), 3.20 (br, 1 H), 3.06 (d, *J* = 10.7, 5.0 Hz, 1 H), 2.83 (br, 1 H), 2.02-1.70 (m, 4 H), 1.34 (s, 9 H); ^{13}C (75 MHz, CDCl$_3$) δ 155.30, 155.23, 138.4, 138.3, 129.6, 128.2, 126.0, 100.9, 92.5, 91.3, 81.4 81.1, 79.0, 78.6, 75.1, 73.3, 73.3, 71.3, 71.2, 70.5, 67.1, 59.0, 58.9, 58.7, 51.7, 38.5, 38.2, 32.2, 32.1, 28.2, 23.2, 23.1; IR (neat) 2930, 1709, 1668, 1105 cm^{-1}; $[\alpha]^{25}_D$ + 28.0 (*c* 0.99, CHCl$_3$); HRMS (ES) calcd. for $C_{27}H_{42}NO_9Na$ (M + Na) 548.2789, found 548.2836.

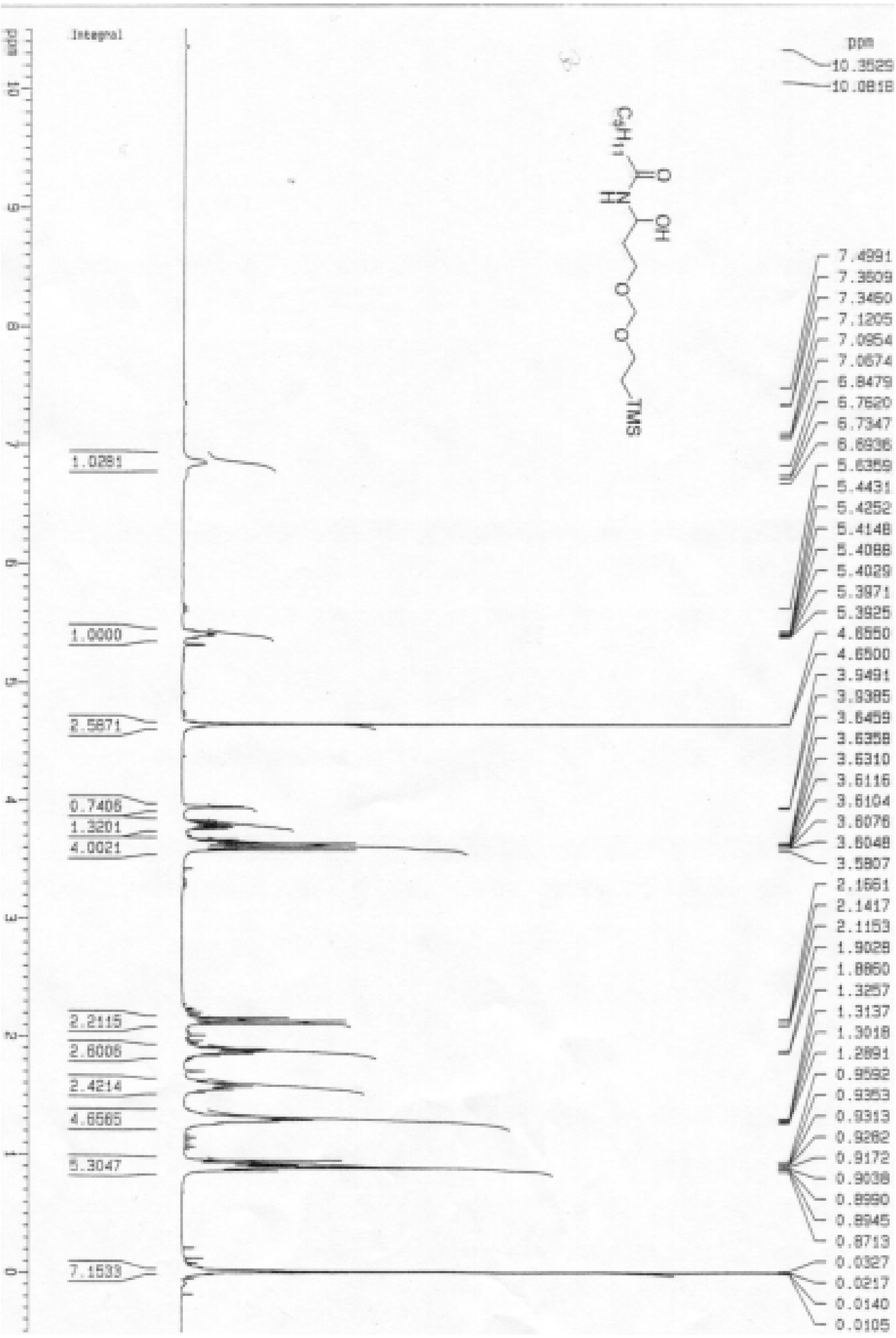


7,8-Dimethoxy-6-methoxymethylhexahydropyrano[3,2-d][1,3]dioxin-4-yl-carbamic acid tert-butyl ester

To a solution of **15** (29 mg, 0.05 mmol) in DCE (6 mL) was added NMQPF$_6$ (0.8 mg, .003 mmol), NaOAc (58 mg, 200 wt.%), Na$_2$S$_2$O$_3$ (58 mg, 200 wt.%), molecular sieves (58 mg, 200 wt.%) and toluene (1 mL). The suspension was stirred at room temperature for 30 minutes. Air was gently bubbled through the suspension while it was irradiated for 2 hours. The reaction mixture was filtered through a small plug of silica and concentrated under reduced pressure. The resulting oil was purified by flash chromatography (50% EtOAc in hexanes) to provide the desired product (18 mg, 94%) a 10:1 mixture of diastereomers:

major diastereomer (16): ^{1}H NMR (300 MHz, CDCl$_3$) δ 5.97 (d, *J* = 9.7 Hz, 1 H), 5.12 (d, *J* = 6.7 Hz, 1 H), 5.08 (dd, *J* = 9.7, 1.4 Hz, 1 H), 4.84 (d, *J* = 6.7 Hz, 1 H), 4.17 (dt, *J* = 7.8, 4.0 Hz, 1 H), 3.92 (dd, *J* = 1.8, 1.8, 1 H), 3.72 (dd, *J* = 1.8, 1.8 Hz, 1 H), 3.62-3.60 (m, 2 H), 3.50 (dd, *J* = 3.8, 2.5 Hz, 1 H), 3.47 (s, 3 H), 3.47 (s, 3 H), 3.42 (s, 3 H), 3.31-3.29 (m, 1 H) 1.47 (s, 9 H); ^{13}C (125 MHz, CDCl$_3$) δ 154.9, 91.6, 80.7, 80.5, 79.6, 76.8, 74.5, 73.5, 71.5, 65.5, 59.3, 58.4, 57.7, 28.3; IR (neat) 2922, 1724, 1500 cm^{-1}; $[\alpha]^{25}_D$ +17.87 (*c* 0.62 CHCl$_3$); HRMS (ES) calcd. for $C_{16}H_{29}NO_8Na$ (M + Na) 386.1791, found 386.1828.

minor diastereomer (17): ^{1}HNMR (300 MHz, CDCl$_3$) δ 5.44 (dd, *J* = 9.3, 8.6 Hz, 1 H), 5.17 (d, *J* = 9.3 Hz, 1 H), 5.10 (d, *J* = 7.0 Hz, 1 H), 4.88 (d, *J* = 7.0 Hz, 1 H), 4.14 (dd, *J* = 9.6, 6.3 Hz, 1 H), 3.87 (app t, *J* = 9.0 Hz, 1 H), 3.76 (dd, *J* = 9.6, 6.3 Hz, 1 H), 3.70-3.64 (m, 1 H), 3.64 (s, 3 H), 3.75-3.50 (m, 2 H), 3.54 (s, 3 H), 3.38 (s, 3 H), 3.27-3.20 (m, 1 H), 1.47 (s, 9 H); ^{13}C (75 MHz, CDCl$_3$) δ 155. 86.9, 81.1, 79.8, 79.0, 78.7, 77.9, 73.5, 71.2, 70.9, 60.8, 60.7, 59.3, 30.0, 28.5; $[\alpha]^{25}_D$ +47.53 (*c* 0.65 CHCl$_3$).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5.3971
5.3925
4.6550
4.6500
3.9491
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3.6459
3.6358
3.6310
3.6116
3.6104
3.6076
3.6048
3.5807
2.1661
2.1417
2.1153
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1.3257
1.3137
1.3018
1.2891
0.9592
0.9353
0.9313
0.9282
0.9172
0.9038
0.8990
0.8945
0.8713
0.0327
0.0217
0.0140
0.0105
Integral
1.0281
1.0000
2.5671
0.7406
1.3201
4.0021
2.2115
2.6006
2.4214
4.6565
5.3047
7.1533
ppm
10
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5
4
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C5H11
O
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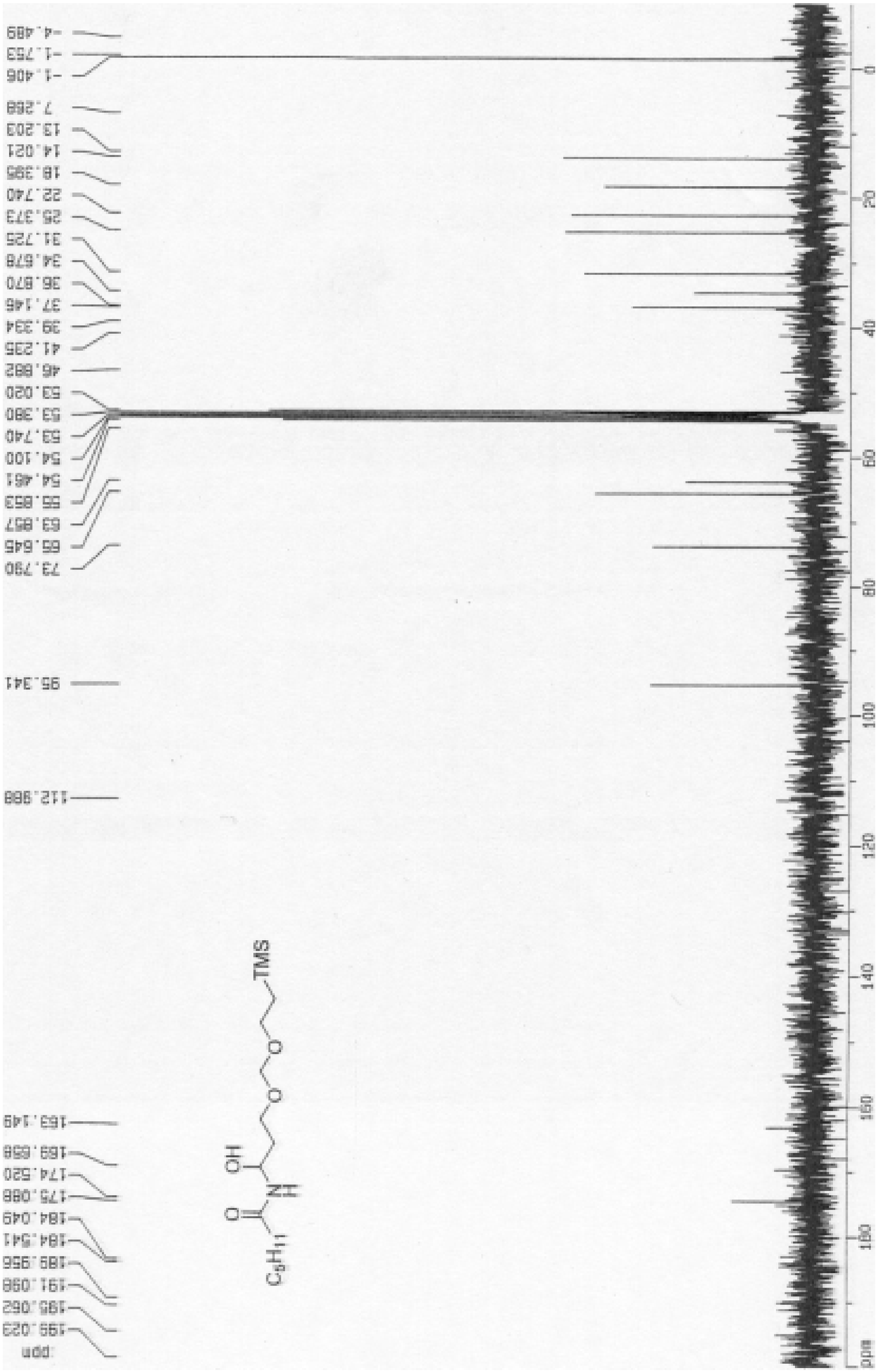
H
OH
O
O
TMS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25.373
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36.870
37.146
39.334
41.235
46.882
53.020
53.380
53.740
54.100
54.461
55.853
63.857
65.645
73.790
95.341
112.988
163.149
169.698
174.520
175.088
184.049
184.541
189.956
191.098
195.062
199.029
ppm
0
20
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60
80
100
120
140
160
180
ppm
C_5H_{11}
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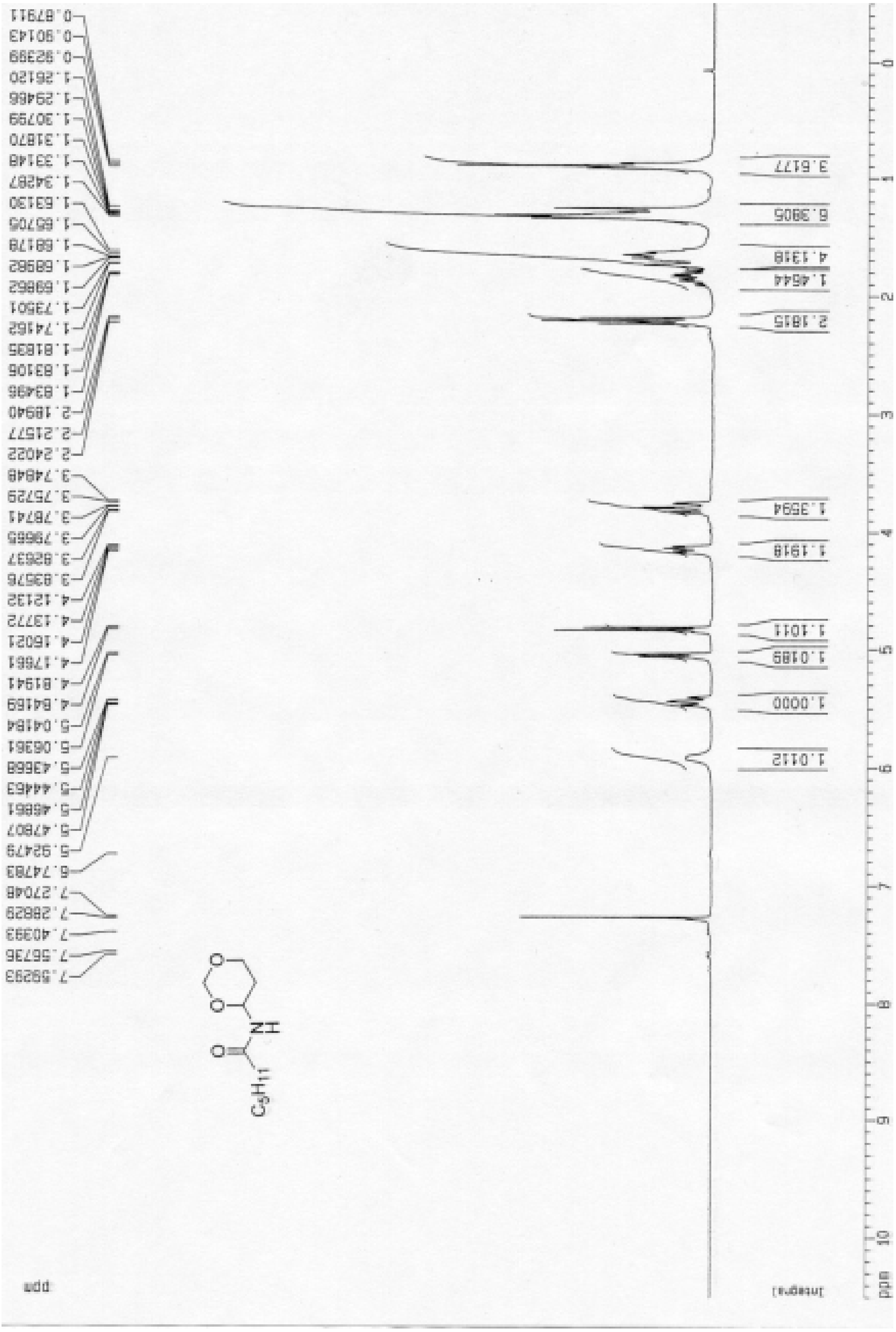
N
H
OH
O
O
TMS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5.43668
5.06361
5.04184
4.84169
4.81941
4.17661
4.16021
4.13772
4.12132
3.83676
3.82637
3.79665
3.78741
3.75729
3.74848
2.24022
2.21577
2.18940
1.83496
1.83106
1.81835
1.74162
1.73501
1.69862
1.68982
1.68178
1.65705
1.63130
1.34287
1.33148
1.31870
1.30799
1.29466
1.26120
0.92399
0.90143
0.87911
ppm
1.0112
1.0000
1.0189
1.1011
1.1918
1.3594
2.1815
1.4544
4.1318
6.3905
3.6177
Integral
10
9
8
7
6
5
4
3
2
1
0
ppm
C5H11
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N
H
O
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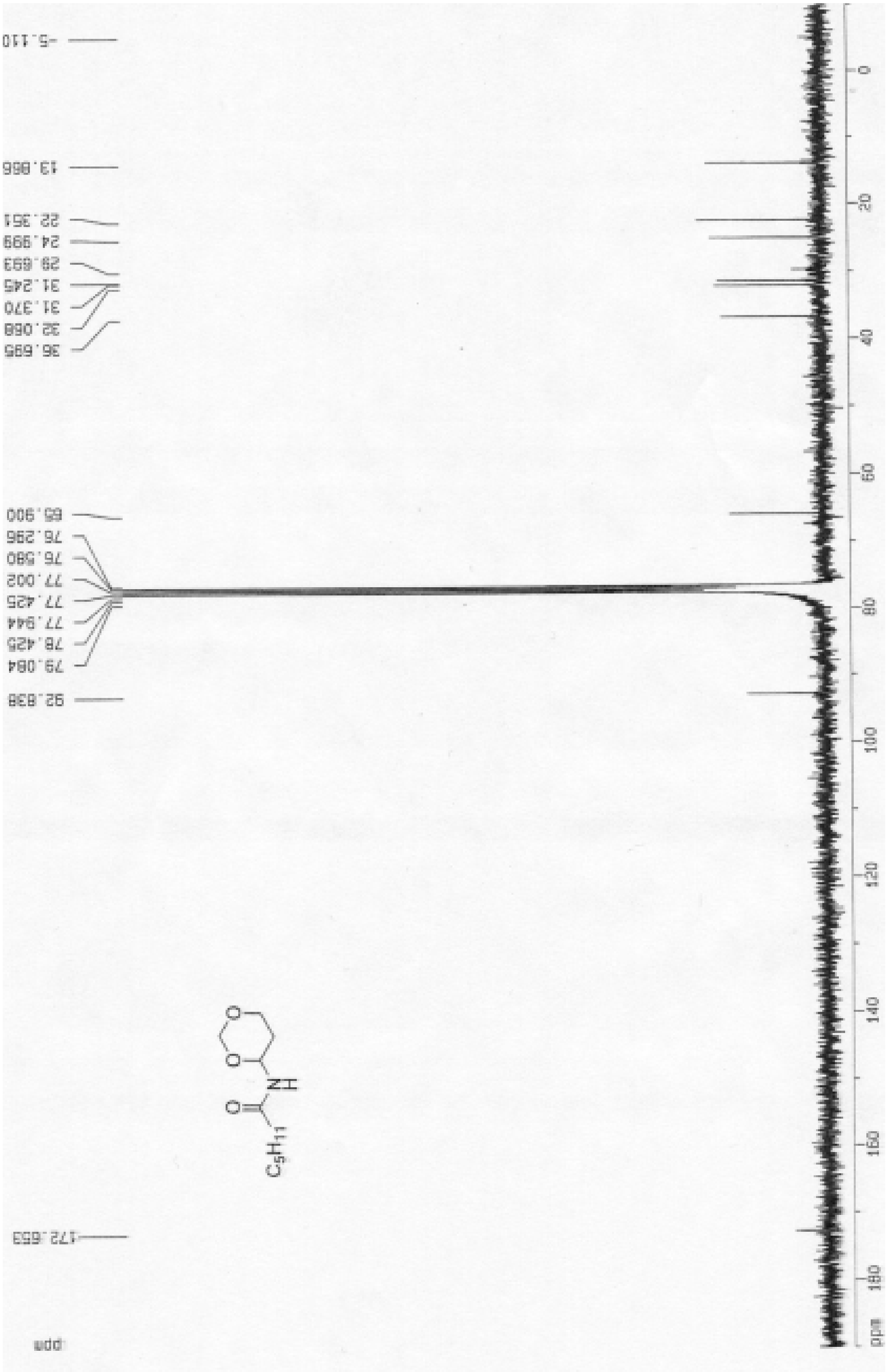
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24.999
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31.245
31.370
32.068
36.695
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76.580
77.002
77.425
77.944
78.425
79.084
92.838
172.659
ppm
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60
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100
120
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160
180
ppm
C_5H_{11}
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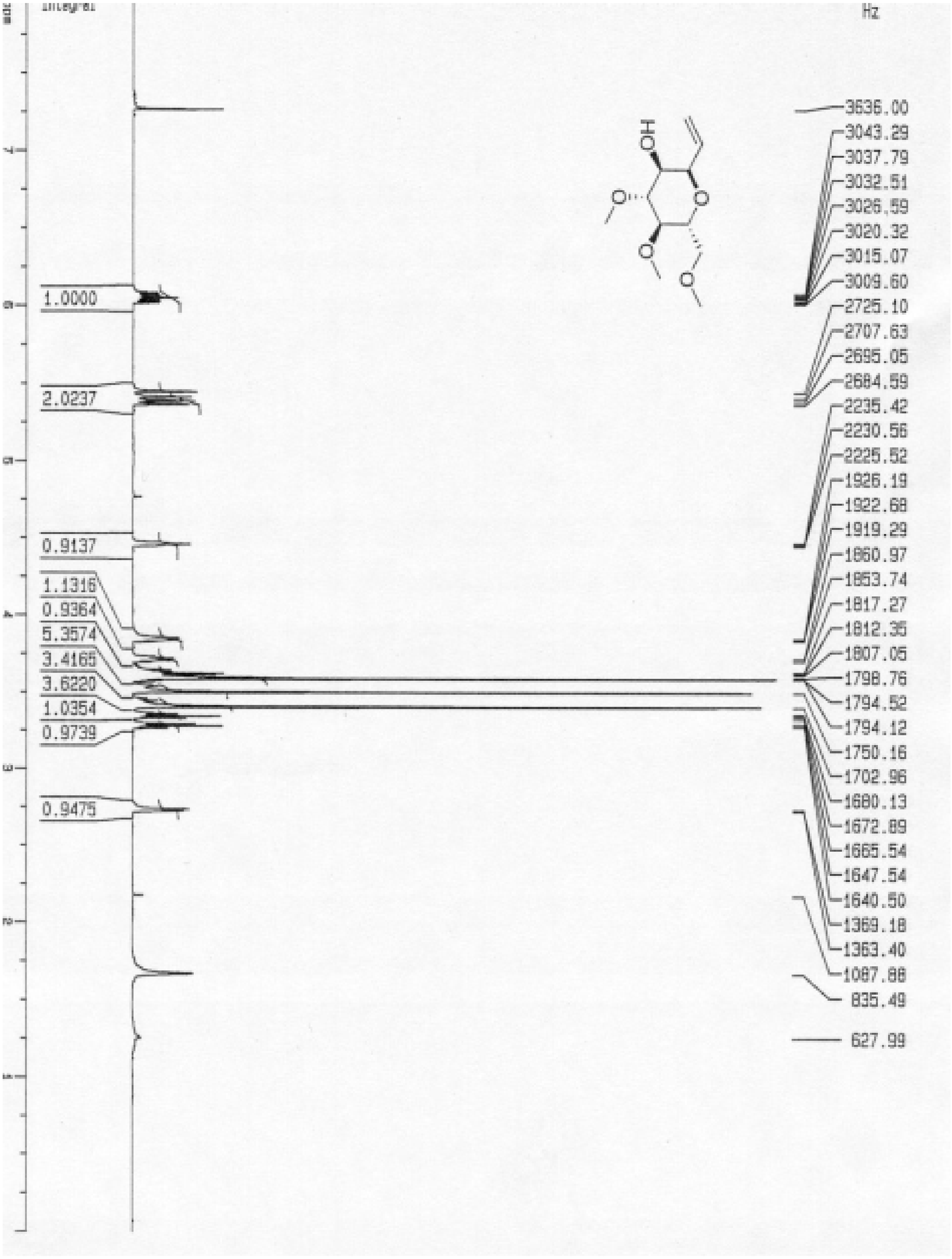
N
H
O
O



Hz
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3043.29
3037.79
3032.51
3026.59
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3015.07
3009.60
2725.10
2707.63
2695.05
2684.59
2235.42
2230.56
2225.52
1926.19
1922.68
1919.29
1860.97
1853.74
1817.27
1812.35
1807.05
1798.76
1794.52
1794.12
1750.16
1702.96
1680.13
1672.89
1665.54
1647.54
1640.50
1369.18
1363.40
1087.88
835.49
627.99
integral
1.0000
2.0237
0.9137
1.1316
0.9364
5.3574
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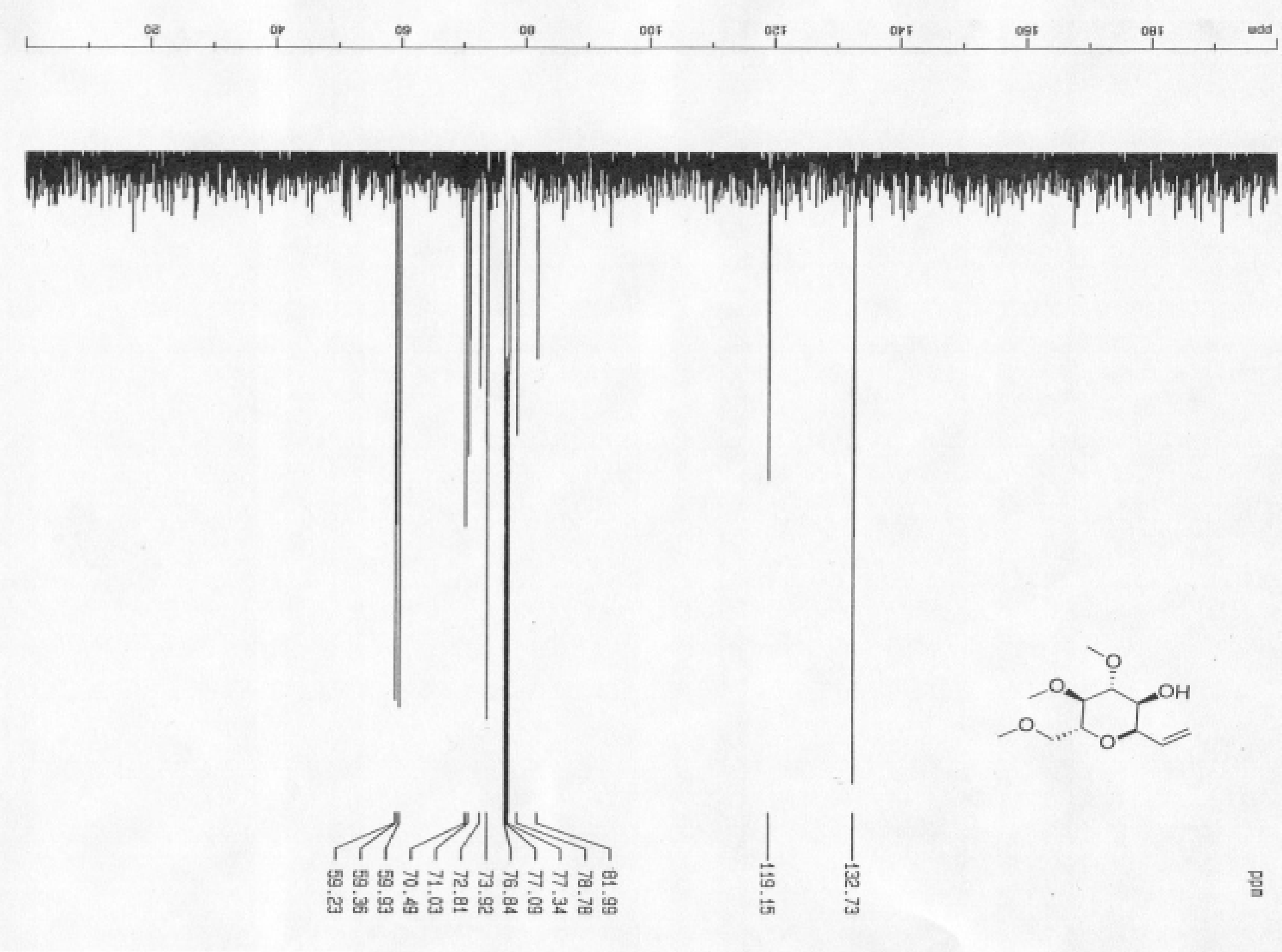
3.6220
1.0354
0.9739
0.9475
7
6
5
4
3
2
1
OH
O


20
40
60
80
100
120
140
160
180
ppm
59.23
59.36
59.93
70.49
71.03
72.81
73.92
76.84
77.09
77.34
78.78
81.99
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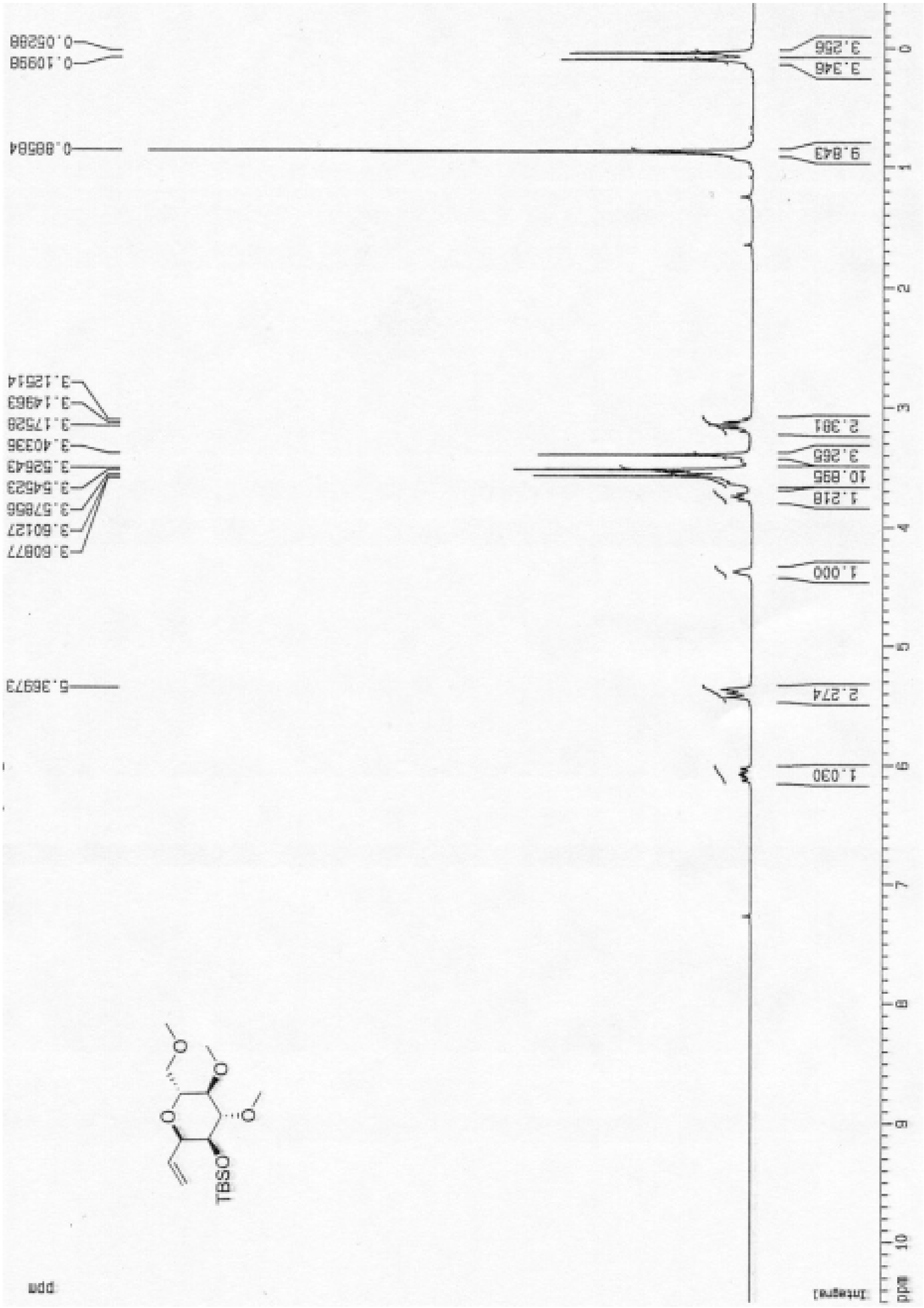
132.73
ppm
OH
O
O
O
O



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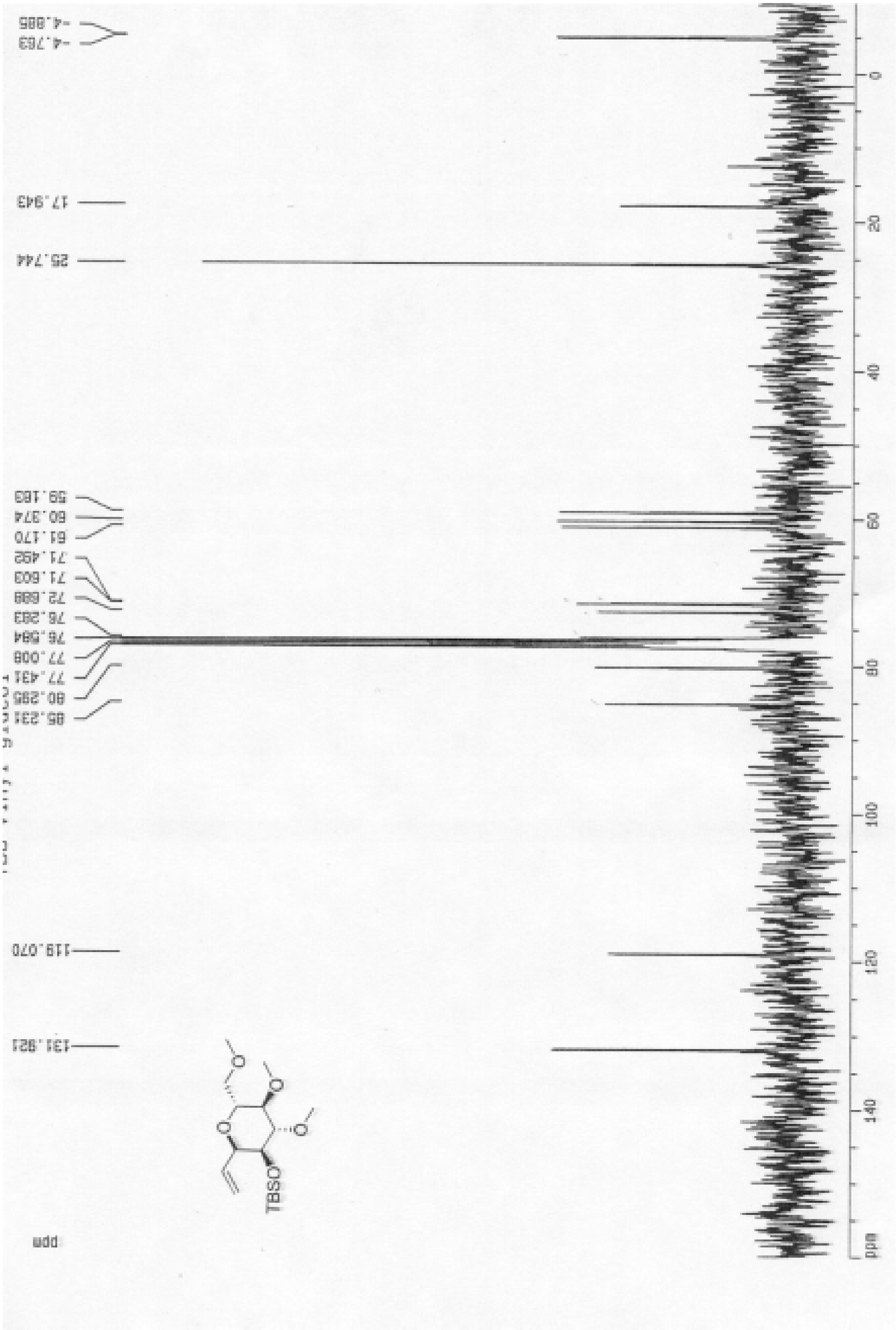
0.88584
3.12514
3.14963
3.17528
3.40336
3.52643
3.54523
3.57856
3.60127
3.60877
5.36973
3.256
3.346
9.843
2.381
3.265
10.895
1.218
1.000
2.274
1.030
TBSO
Integral
ppm
0
1
2
3
4
5
6
7
8
9
10



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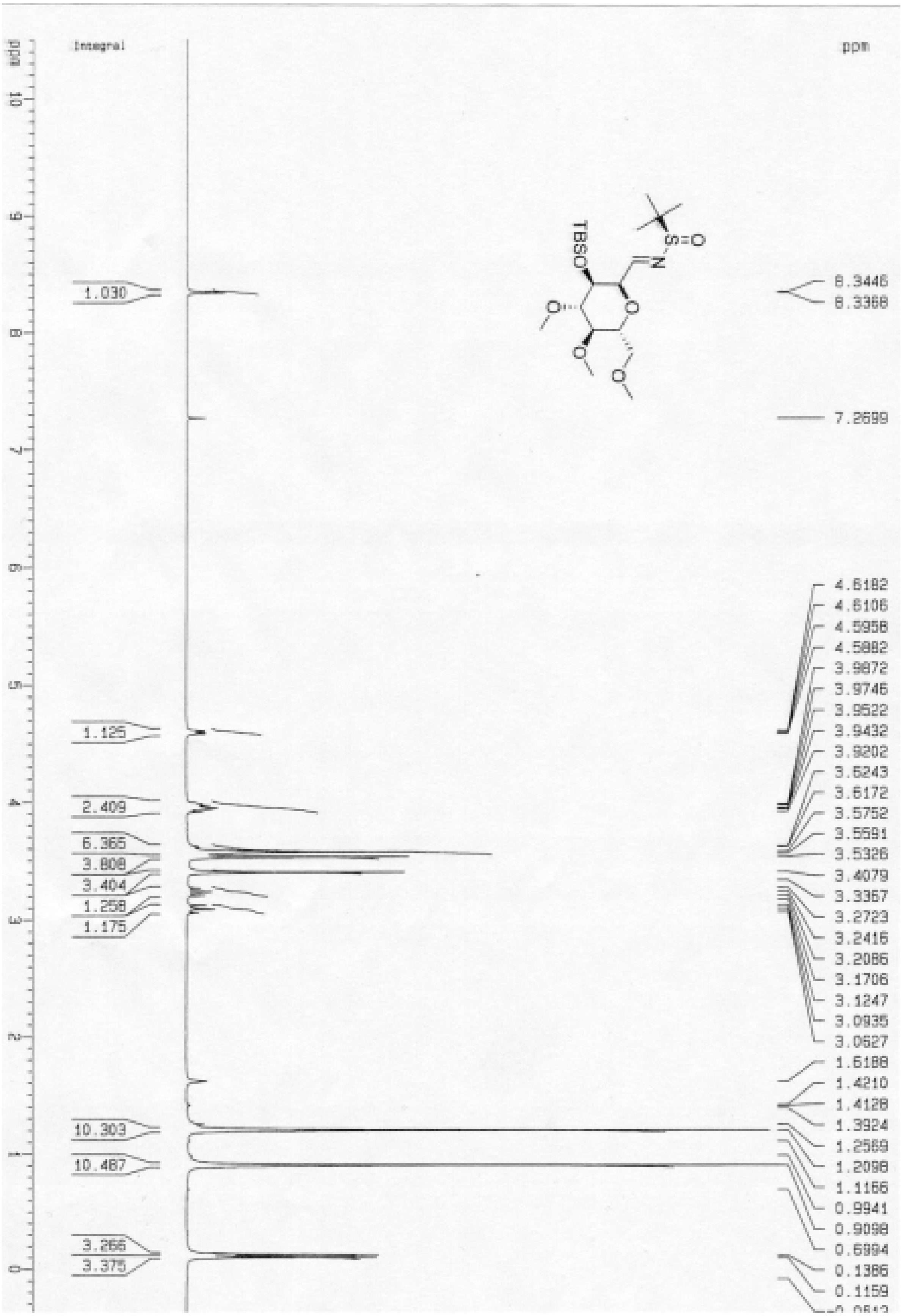
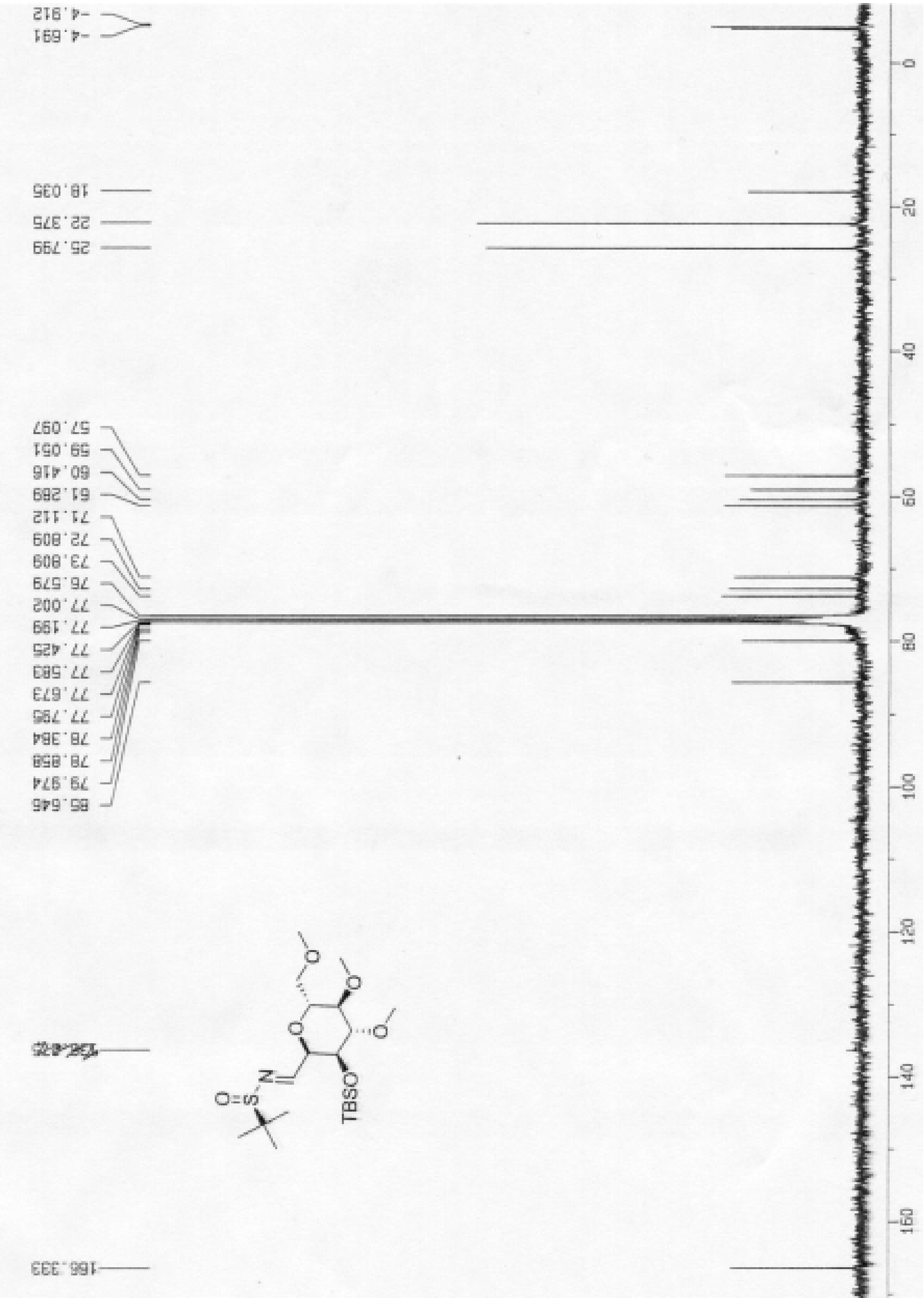
25.744
59.183
60.374
61.170
71.492
71.603
72.688
76.293
76.584
77.008
77.431
80.295
85.231
119.070
131.821
ppm
TBSO
0
20
40
60
80
100
120
140
ppm


ppm
Integral
ppm
10
9
8
7
6
5
4
3
2
1
0
1.030
1.125
2.409
6.365
3.808
3.404
1.258
1.175
10.303
10.487
3.266
3.375
8.3446
8.3368
7.2699
4.6182
4.6106
4.5958
4.5882
3.9872
3.9746
3.9522
3.9432
3.9202
3.6243
3.6172
3.5752
3.5591
3.5326
3.4079
3.3367
3.2723
3.2416
3.2086
3.1706
3.1247
3.0935
3.0627
1.6188
1.4210
1.4128
1.3924
1.2569
1.2098
1.1166
0.9941
0.9098
0.6994
0.1386
0.1159
TBSO
O
N
S
O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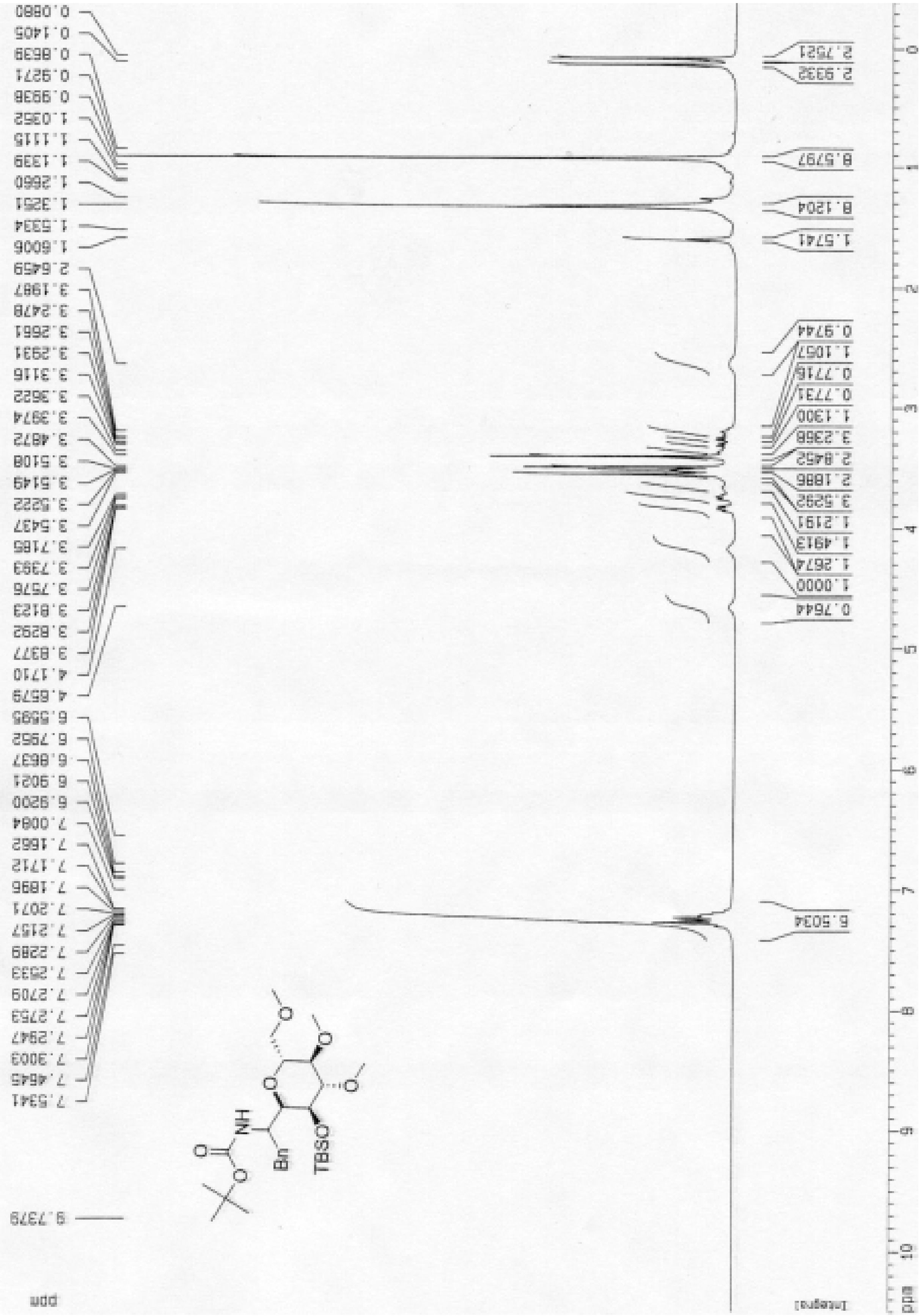
25.799
57.097
59.051
60.416
61.289
71.112
72.809
73.809
76.579
77.002
77.199
77.425
77.583
77.673
77.795
78.384
78.858
79.974
85.646
166.333
ppm
0
20
40
60
80
100
120
140
160
ppm
TBSO
N
S
O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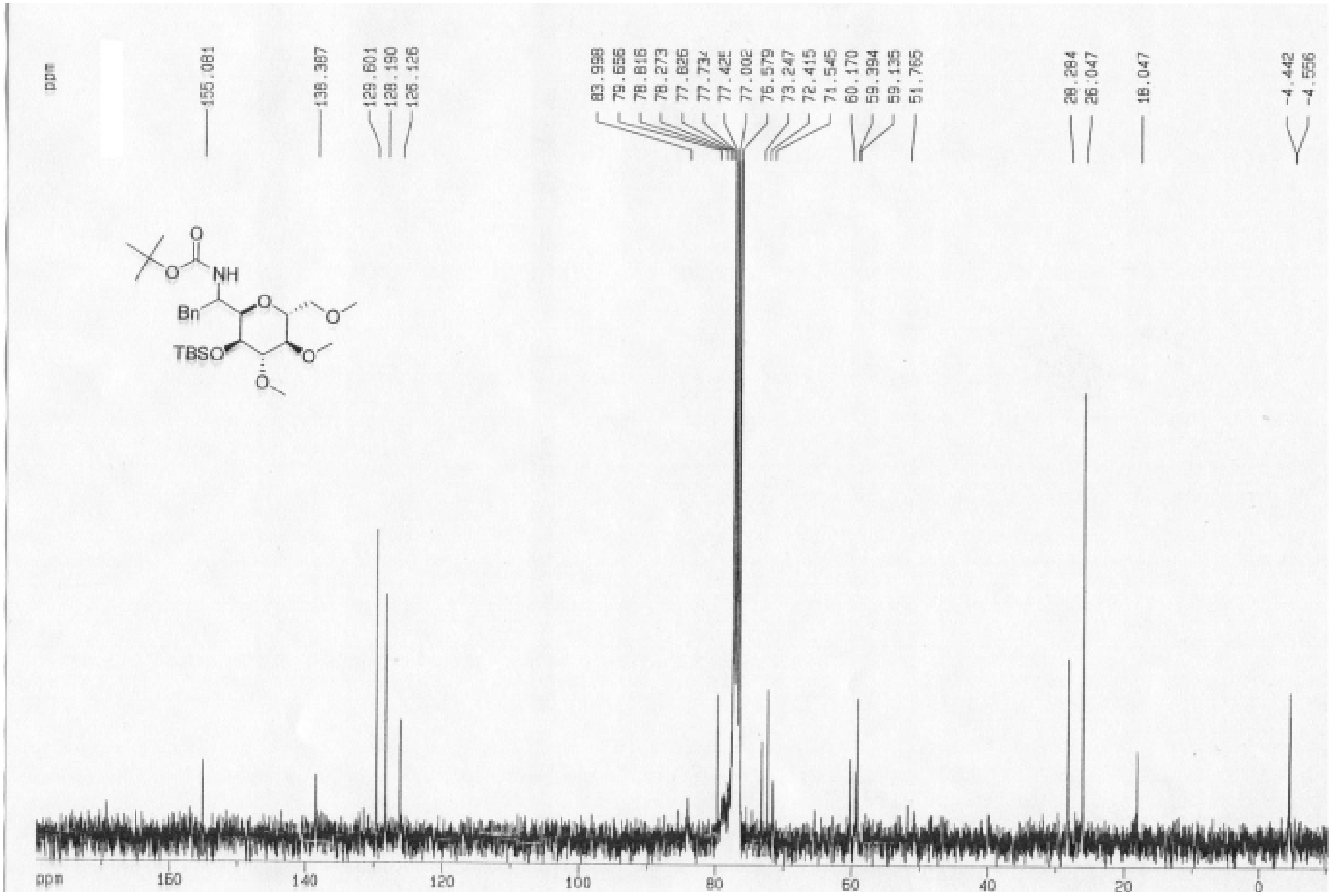
7.5341
7.4545
7.3003
7.2947
7.2753
7.2709
7.2533
7.2289
7.2157
7.2071
7.1896
7.1712
7.1662
7.0084
6.9200
6.9021
6.8637
6.7962
6.5595
4.6579
4.1710
3.8377
3.8292
3.8123
3.7576
3.7393
3.7185
3.5437
3.5222
3.5149
3.5108
3.4872
3.3974
3.3622
3.3116
3.2931
3.2661
3.2478
3.1987
2.6459
1.6006
1.5334
1.3251
1.2660
1.1339
1.1115
1.0352
0.9938
0.9271
0.8639
0.1405
0.0880
Integral
6.5034
0.7644
1.0000
1.2674
1.4913
1.2191
3.5292
2.1886
2.8452
3.2368
1.1300
0.7731
0.7716
1.1057
0.9744
1.5741
8.1204
8.5797
2.9332
2.7521
ppm
10
9
8
7
6
5
4
3
2
1
0
O
O
NH
Bn
TBSO
O
O
O
O


ppm
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129.601
128.190
126.126
83.998
79.656
78.816
78.273
77.826
77.734
77.425
77.002
76.579
73.247
72.415
71.545
60.170
59.394
59.135
51.765
28.284
26.047
18.047
-4.442
-4.556
O
NH
O
Bn
O
O
TBSO
O
O
ppm
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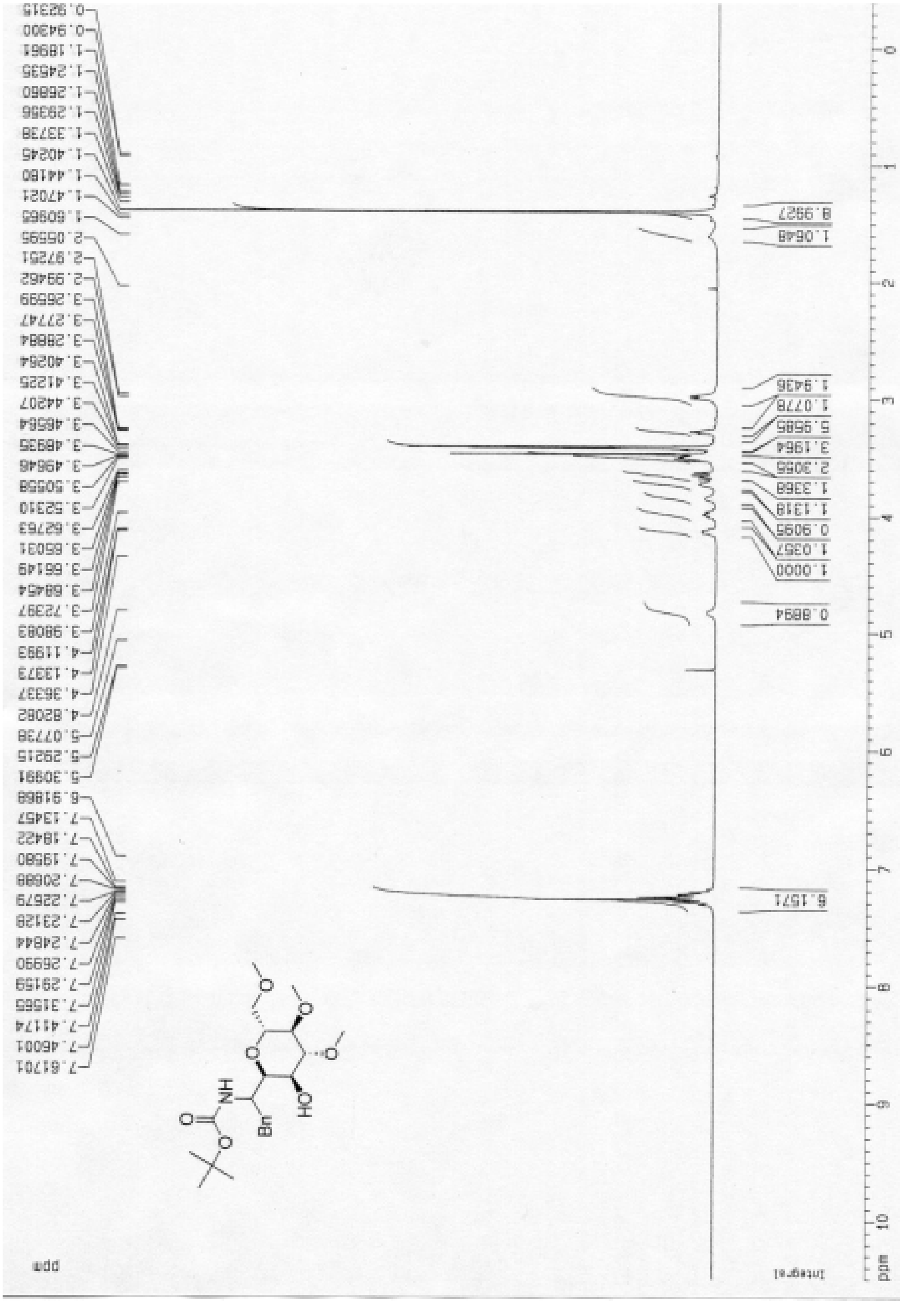
140
120
100
80
60
40
20
0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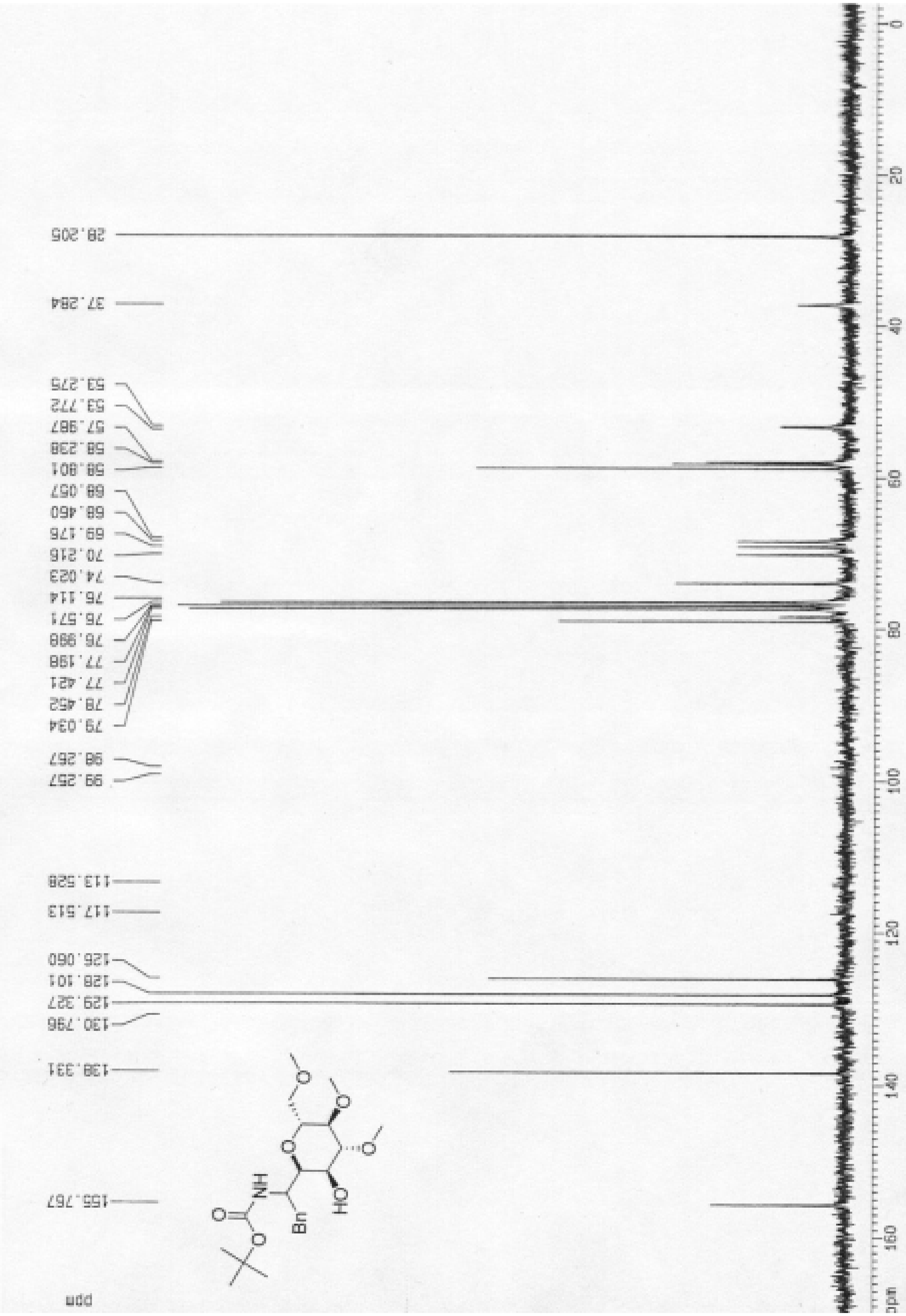
1.33738
1.40245
1.44180
1.47021
1.60965
2.05595
2.97251
2.99462
3.26599
3.27747
3.28884
3.40264
3.41225
3.44207
3.46564
3.48935
3.49646
3.50558
3.52310
3.62763
3.65031
3.66149
3.68454
3.72397
3.98083
4.11993
4.13373
4.36337
4.82082
5.07738
5.29215
5.30991
6.91868
7.13457
7.18422
7.19580
7.20688
7.22679
7.23128
7.24844
7.26990
7.29159
7.31565
7.41174
7.46001
7.61701
ppm
8.9927
1.0648
1.9436
1.0778
5.9585
3.1964
2.3055
1.3368
1.1318
0.9095
1.0357
1.0000
0.8894
6.1571
Integral
0
1
2
3
4
5
6
7
8
9
10
ppm
O
NH
O
Bn
O
OH
O
O
O



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37.284
53.275
53.772
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58.238
58.801
68.057
68.460
69.176
70.216
74.023
76.114
76.571
76.988
77.198
77.421
78.452
79.034
98.267
99.257
113.528
117.513
126.060
128.101
129.327
130.796
138.331
155.767
ppm
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20
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60
80
100
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140
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ppm
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O
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Bn
O
HO
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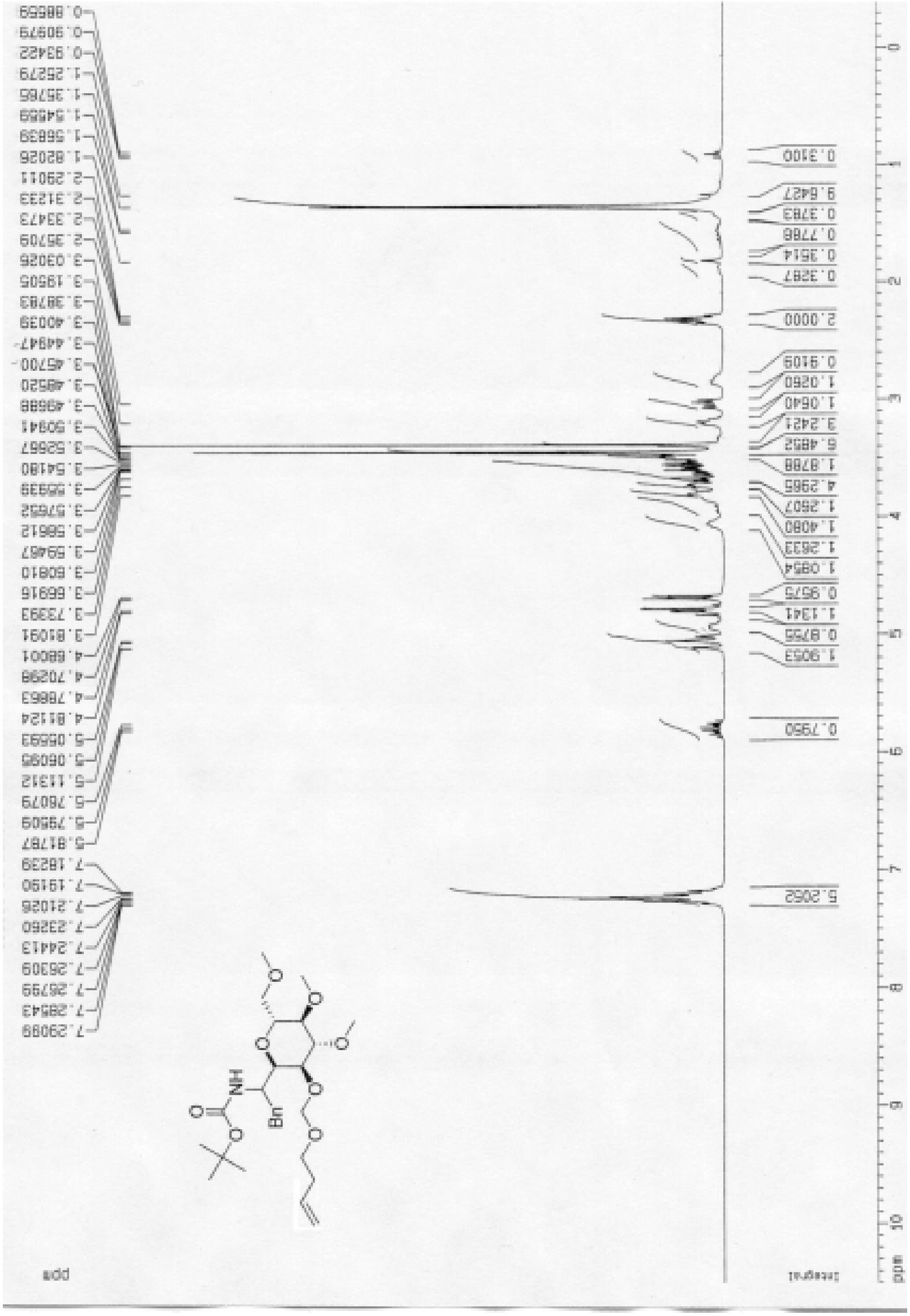
O
O



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2.29011
2.31233
2.33473
2.35709
3.03026
3.19505
3.38783
3.40039
3.44947
3.45700
3.48520
3.49688
3.50941
3.52667
3.54180
3.55939
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3.60810
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5.81787
7.18239
7.19190
7.21026
7.23260
7.24413
7.26309
7.26799
7.28543
7.29089
ppm
NH
O
O
Bn
O
0.3100
9.6427
0.3783
0.7786
0.3514
0.3287
2.0000
0.9109
1.0260
1.0640
3.2421
6.4852
1.8788
4.2965
1.2607
1.4080
1.2633
1.0854
0.9575
1.1341
0.8755
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5.2052
Integral
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2
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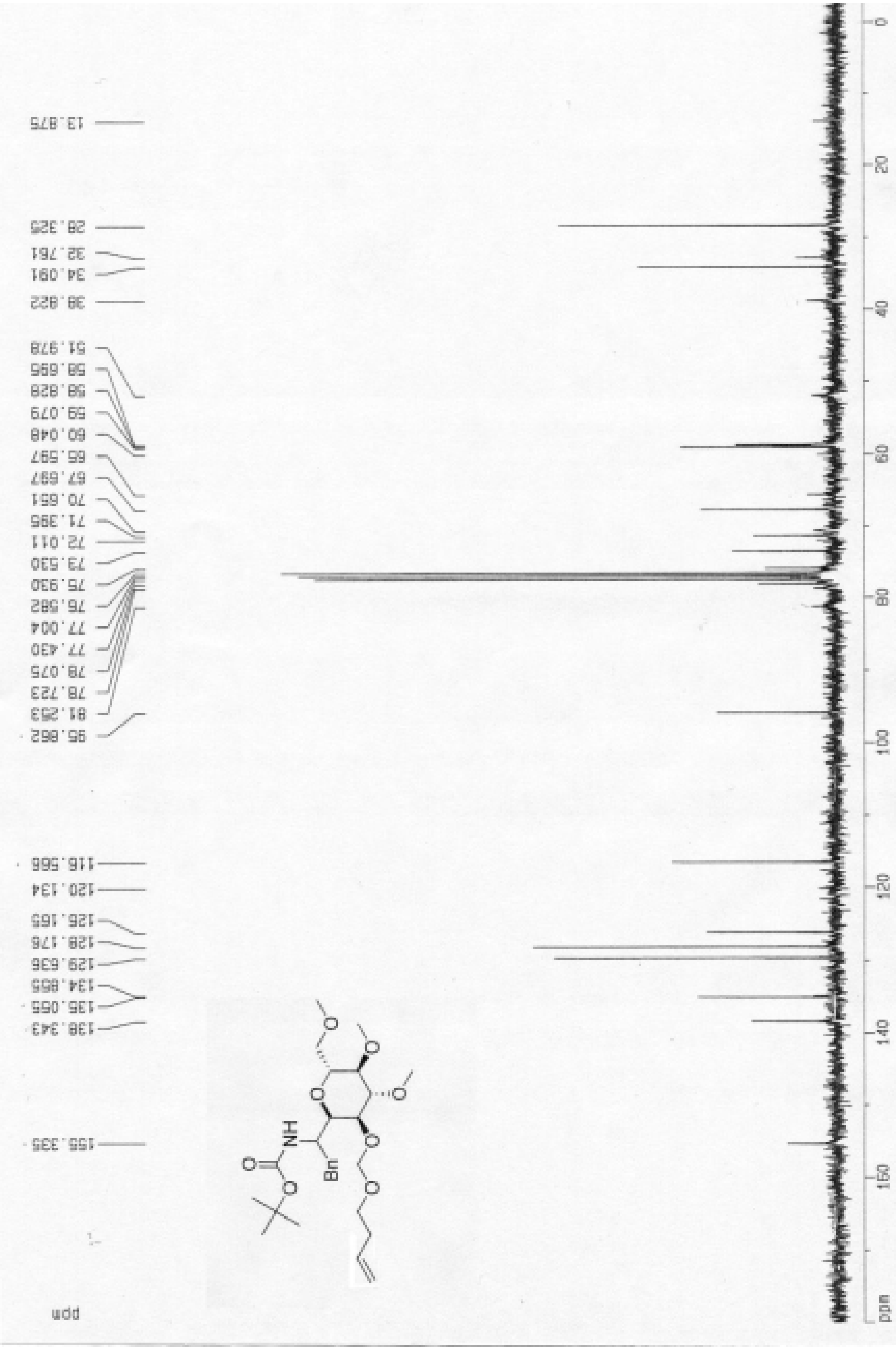
4
5
6
7
8
9
10
ppm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58.695
58.828
59.079
60.048
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67.697
70.651
71.395
72.011
73.530
75.930
76.582
77.004
77.430
78.075
78.723
81.253
95.862
116.566
120.134
126.165
128.176
129.636
134.865
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ppm
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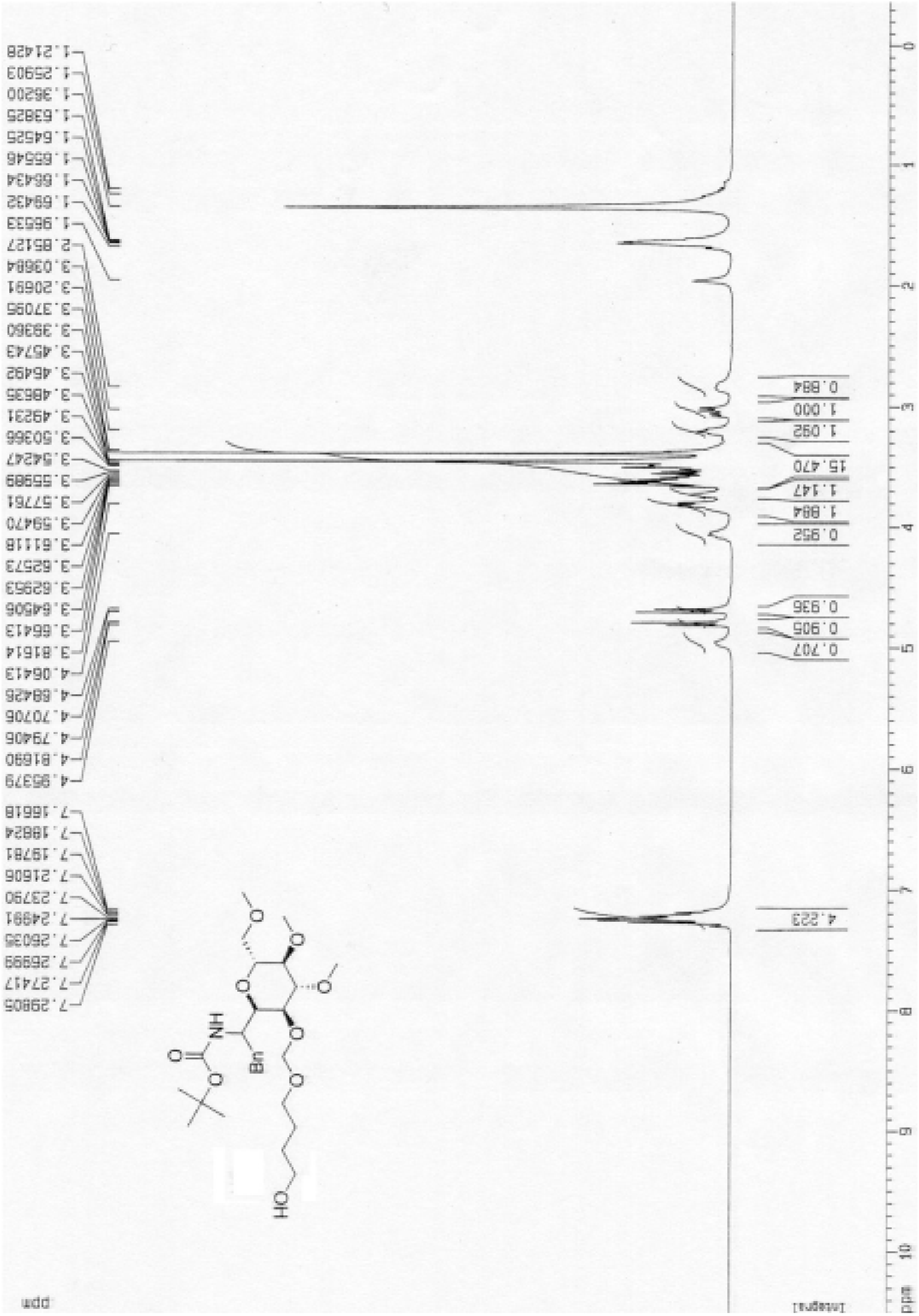
40
60
80
100
120
140
160
ppm
NH
O
Bn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7.19781
7.18824
7.16618
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4.81690
4.79406
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4.06413
3.81614
3.66413
3.64506
3.62953
3.62573
3.61118
3.59470
3.57761
3.55989
3.54247
3.50366
3.49231
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3.46492
3.45743
3.39360
3.37095
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3.03684
2.85127
1.96533
1.69432
1.66434
1.65546
1.64525
1.63825
1.36200
1.25903
1.21428
ppm
HO
O
O
Bn
NH
O
O
O
O
O
Integral
4.223
0.707
0.905
0.936
0.952
1.884
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1.000
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ppm
10
9
8
7
6
5
4
3
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1
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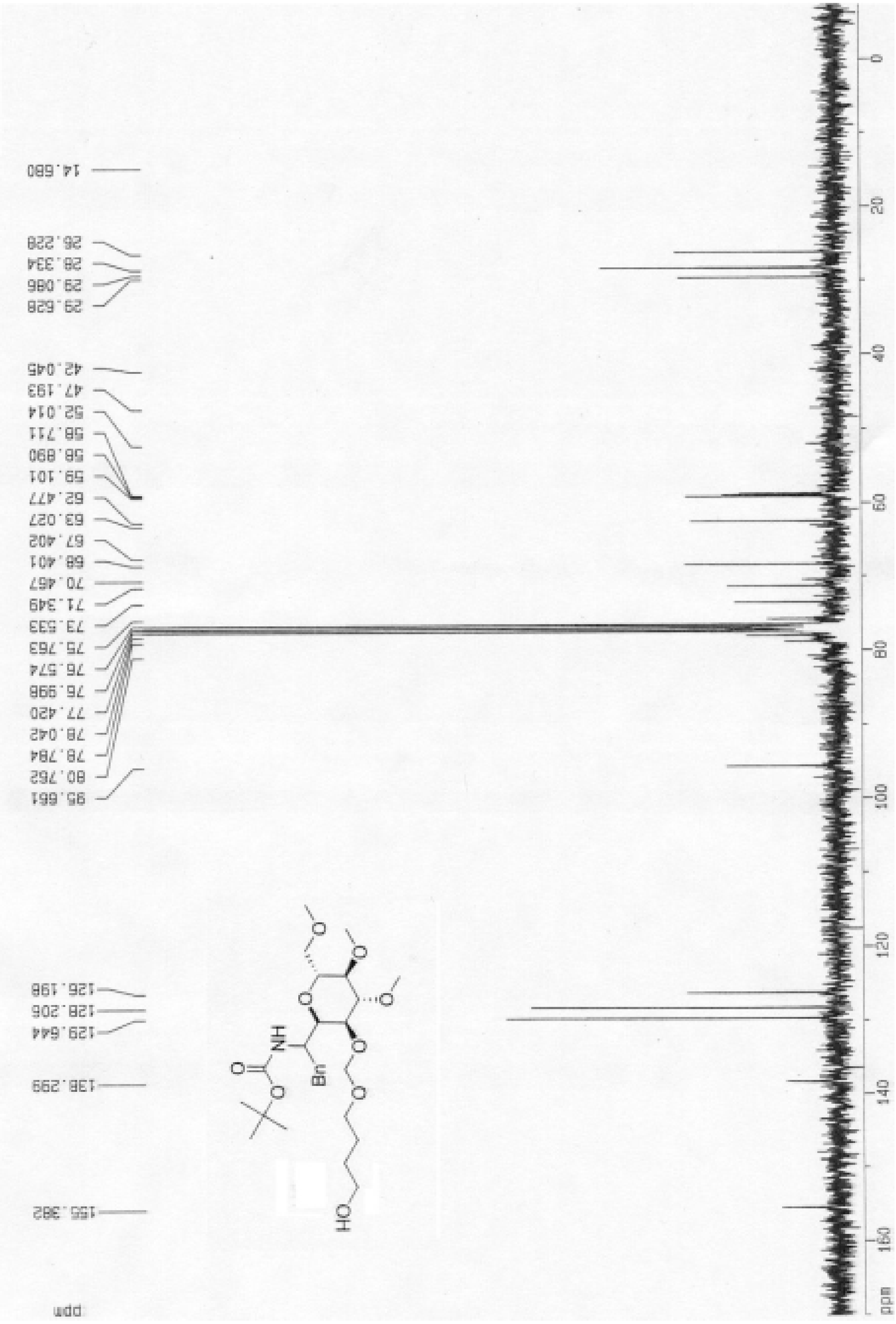
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29.086
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42.045
47.193
52.014
58.711
58.890
59.101
62.477
63.027
67.402
68.401
70.467
71.349
73.533
75.763
76.574
76.998
77.420
78.042
78.784
80.762
95.661
126.198
128.206
129.644
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ppm
NH
O
Bn
HO
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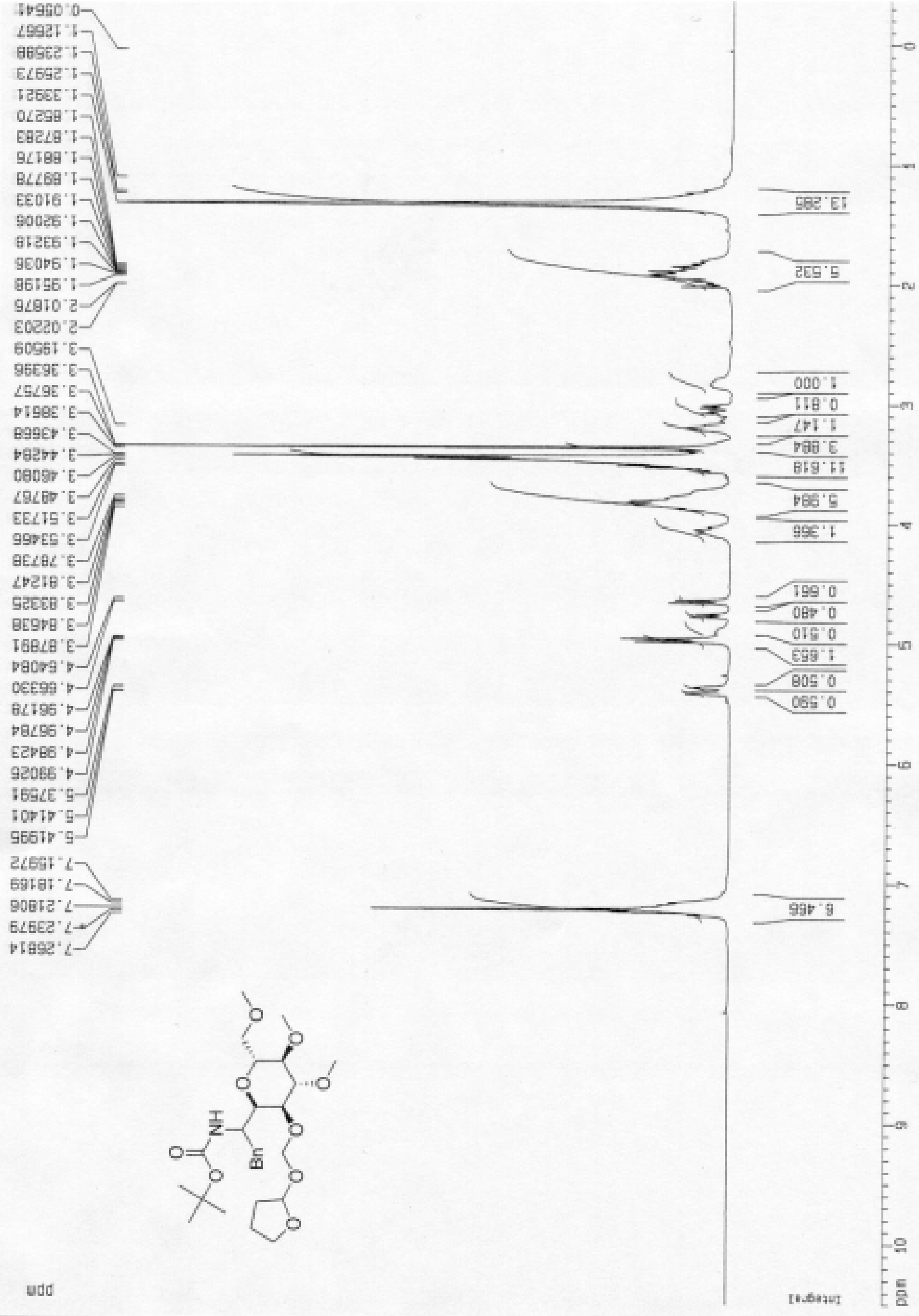
20
40
60
80
100
120
140
160
ppm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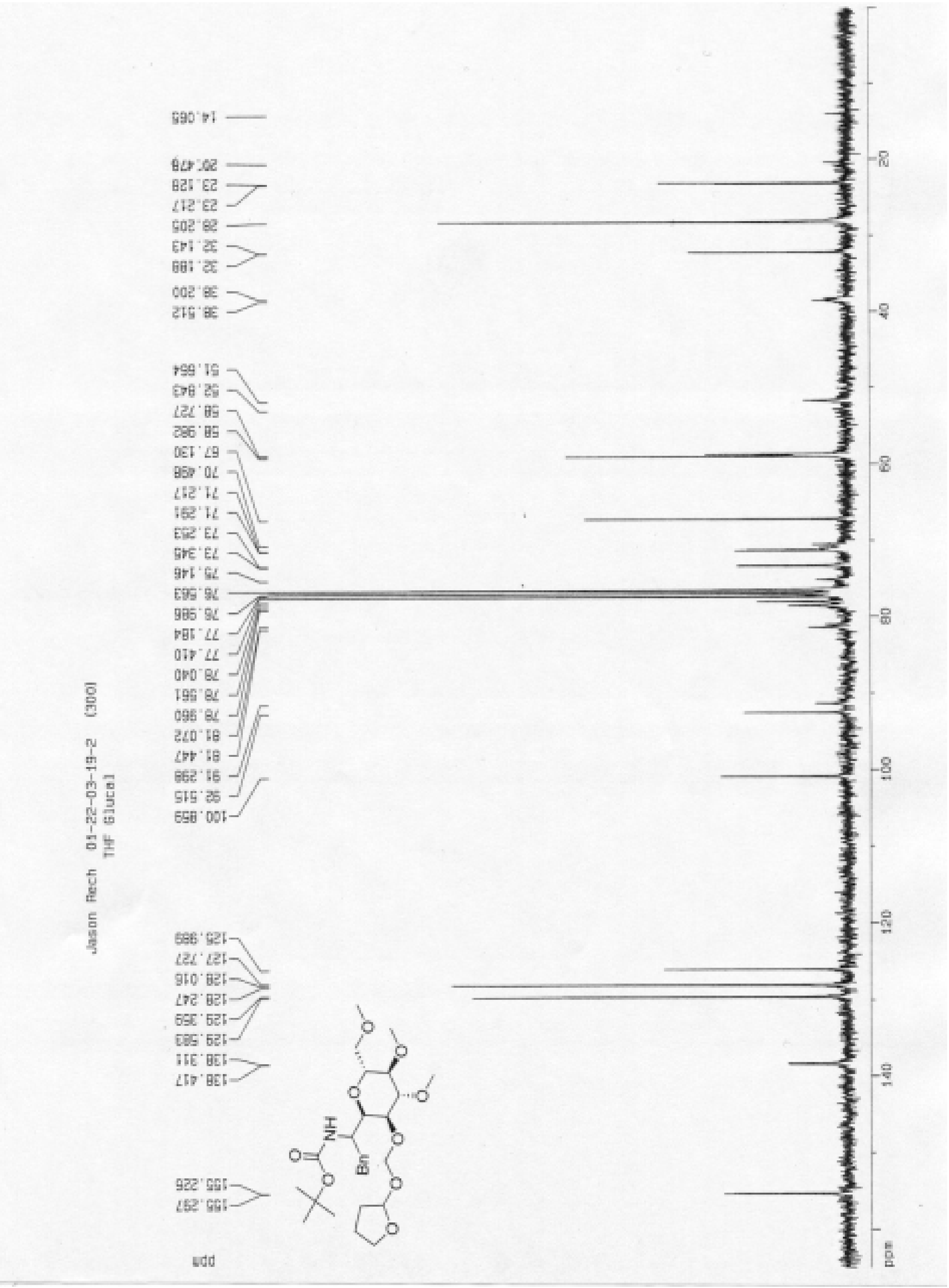
5.41995
5.41401
5.37591
4.99026
4.98423
4.96784
4.96178
4.66330
4.64084
3.87891
3.84638
3.83325
3.81247
3.78738
3.53466
3.51733
3.48767
3.46080
3.44284
3.43668
3.38614
3.36757
3.36396
3.19509
2.02203
2.01876
1.95198
1.94036
1.93218
1.92006
1.91033
1.89778
1.88176
1.87283
1.85270
1.33921
1.25973
1.23588
1.12667
0.05641
6.466
0.590
0.508
1.653
0.510
0.480
0.661
1.366
5.984
11.618
3.884
1.147
0.811
1.000
5.532
13.285
10
9
8
7
6
5
4
3
2
1
0
ppm
O
NH
Bn
O
O
O
O
O
O
O


Jason Rech 03-22-03-19-2 (300)
THF Glucal
ppm
14.085
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23.128
23.217
28.205
32.143
32.189
38.200
38.512
51.664
52.843
58.727
58.982
67.130
70.498
71.217
71.291
73.253
73.345
75.146
76.563
76.986
77.184
77.410
78.040
78.561
78.960
81.072
81.447
91.298
92.515
100.859
125.989
127.727
128.016
128.247
129.359
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155.226
155.257
NH
O
Bn
20
40
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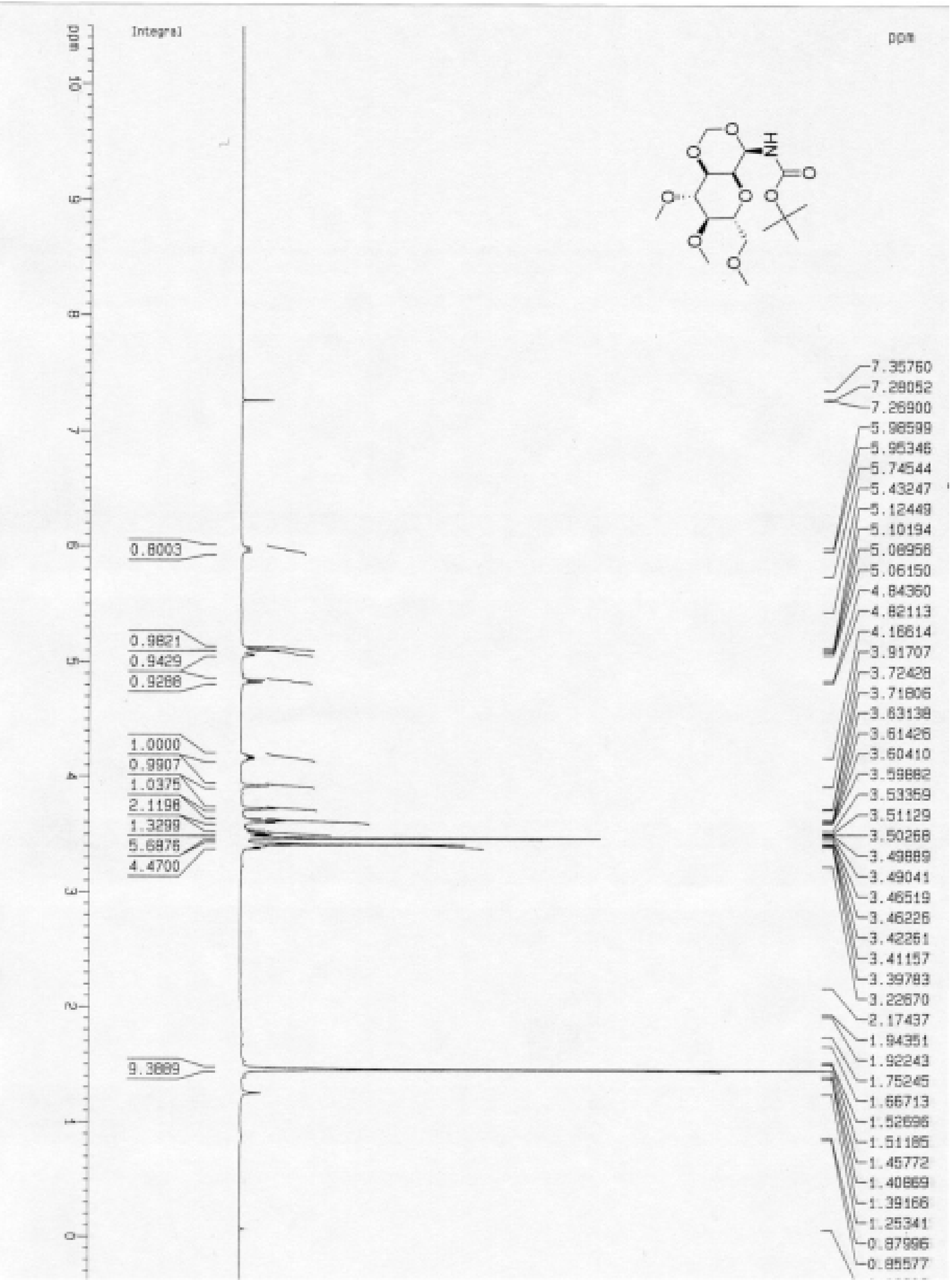
80
100
120
140
ppm


Integral
ppm
HN
O
O
O
O
O
O
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
0.8003
0.9821
0.9429
0.9288
1.0000
0.9907
1.0375
2.1198
1.3298
5.6876
4.4700
9.3889
7.35760
7.28052
7.26900
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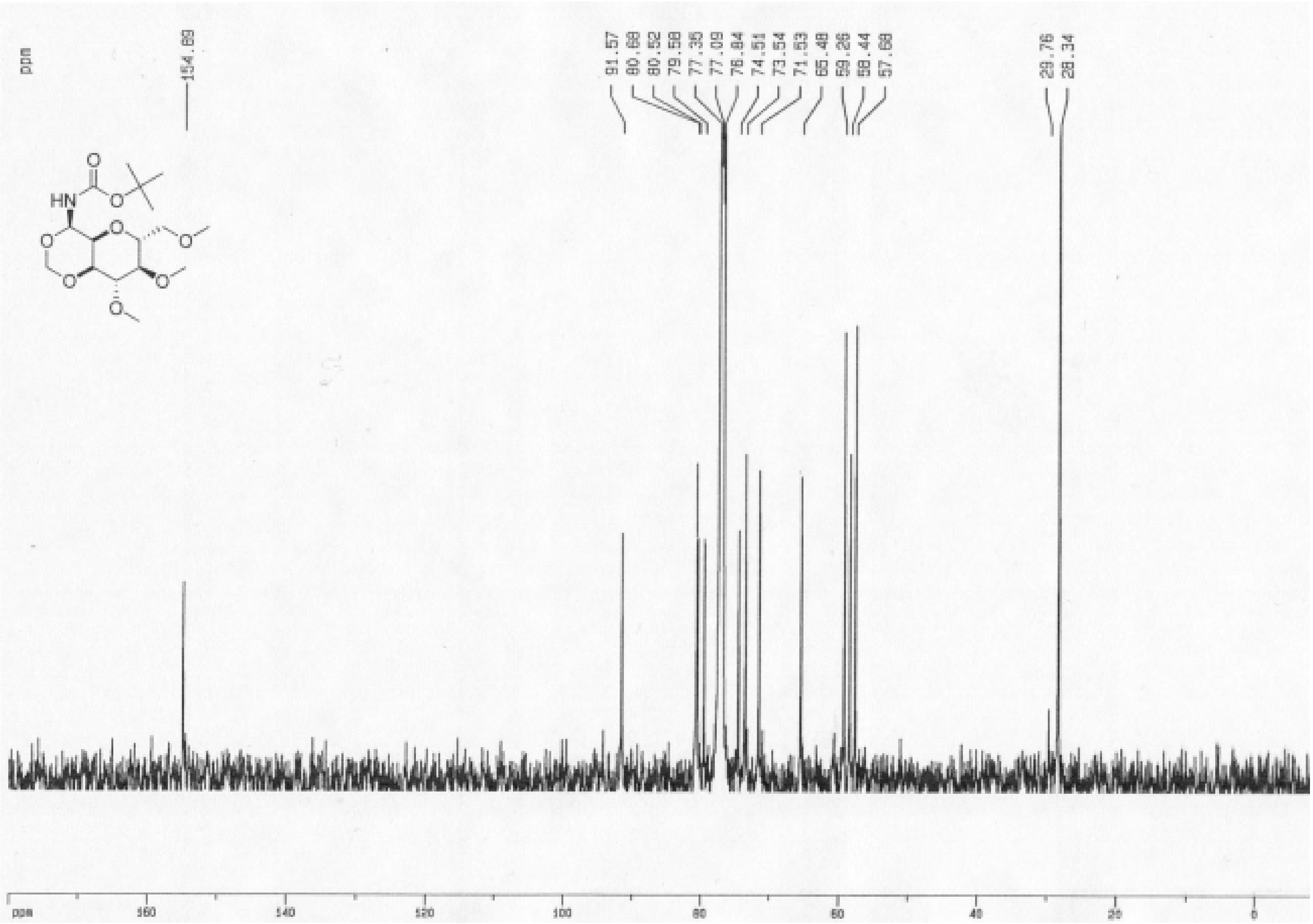
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80.52
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65.48
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29.76
28.34
HN
O
ppm
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140
120
100
80
60
40
20
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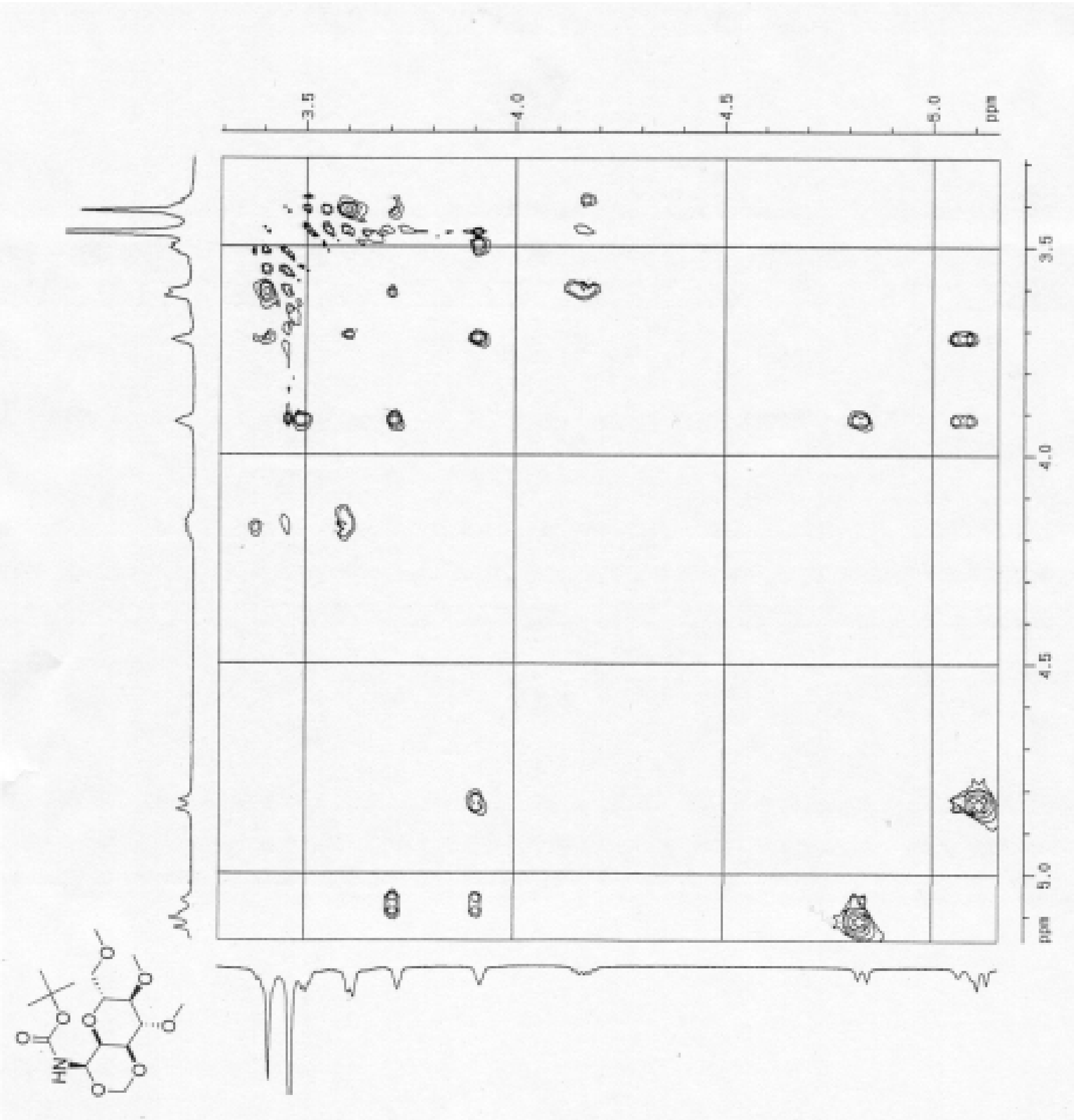

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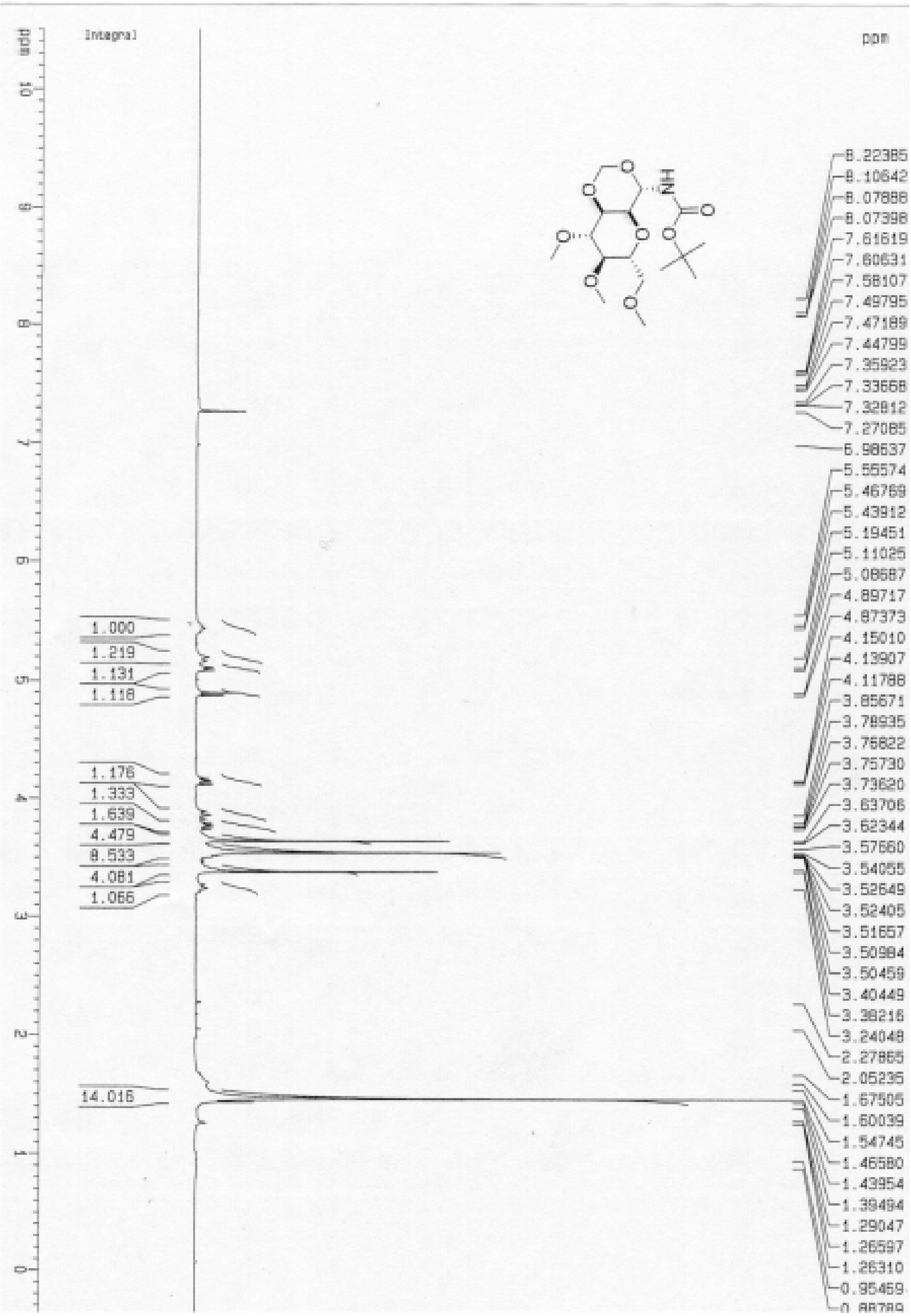

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7.44799
7.35923
7.33668
7.32812
7.27085
6.98637
5.55574
5.46769
5.43912
5.19451
5.11025
5.08687
4.89717
4.87373
4.15010
4.13907
4.11788
3.85671
3.78935
3.76822
3.75730
3.73620
3.63706
3.62344
3.57660
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3.52649
3.52405
3.51657
3.50984
3.50459
3.40449
3.38216
3.24048
2.27865
2.05235
1.67505
1.60039
1.54745
1.46580
1.43954
1.39494
1.29047
1.26597
1.26310
0.95459
0.88789
1.000
1.219
1.131
1.118
1.176
1.333
1.639
4.479
8.533
4.081
1.066
14.016
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4
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O
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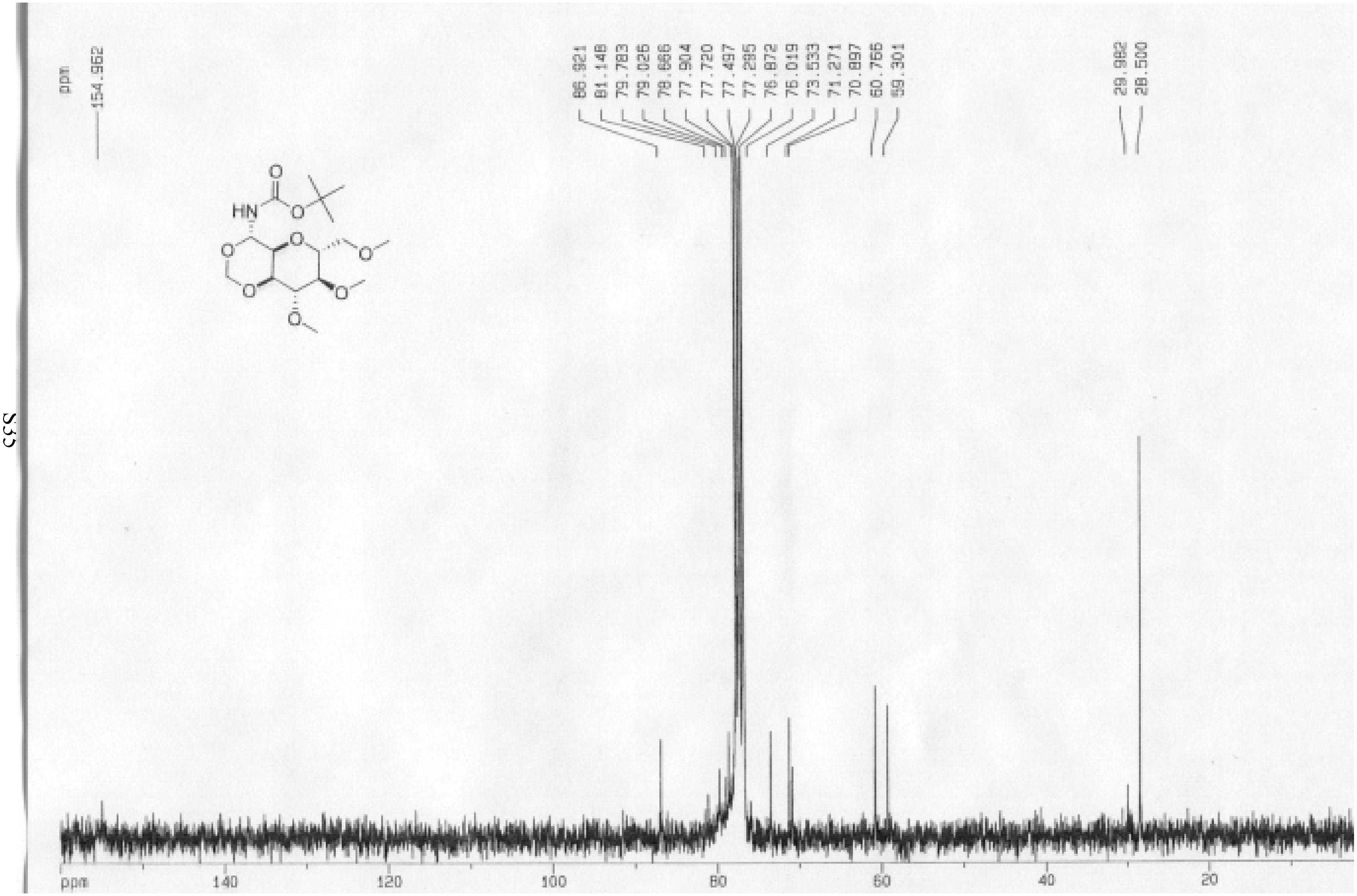
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71.271
70.897
60.766
59.301
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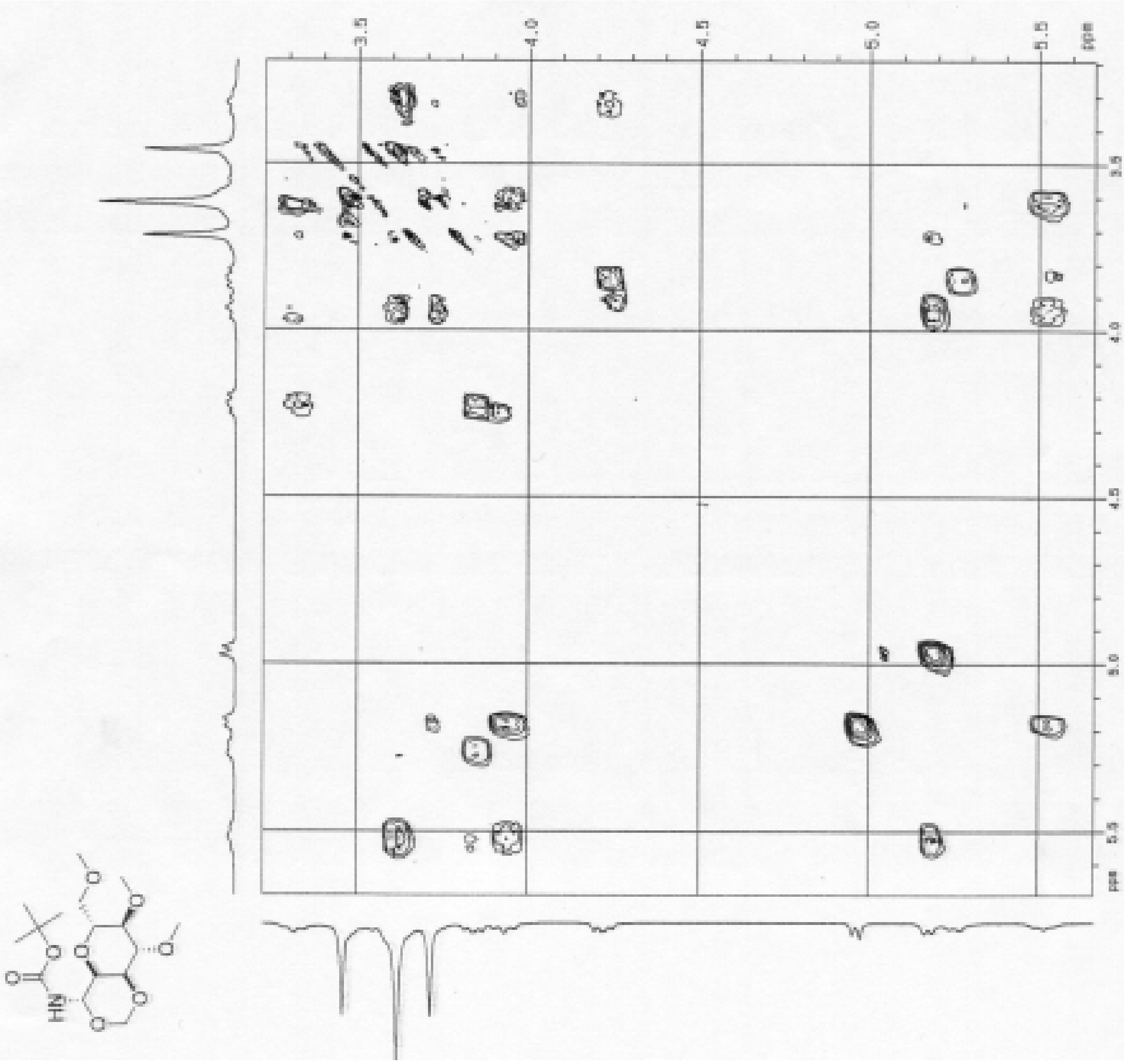

NOESY

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