Supporting Information to Accompany "A Versatile Preparation of α,β–Unsaturated Lactones From Homoallylic Alcohols"

(G. E. Keck*, X.-Y. Li, and C. E. Knutson)

Submitted Electronically For Publication in ***Organic Letters*** May 3, 1999

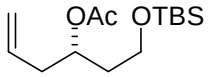
Includes Experimental Details, Spectral and Characterization Data, Copies of NMR Spectra

email: keck@chemistry.chem.utah.edu

General Experimental

All reactions were carried out under an atmosphere of nitrogen. Glassware for all reactions was oven dried at 125 ºC and cooled in a desiccator prior to use. Liquid reagents and solvents were introduced by oven-dried syringes or cannulas through septa sealed flasks under a nitrogen atmosphere. Solvents were purified according to the guidelines in *Purification of Common Laboratory Chemicals* (Perrin, Armarego, and Perrin, Pergamon: Oxford, 1966). Diethyl ether and THF were distilled from sodium metal. Diisopropylamine and dichloromethane were distilled from CaH_2. Yields were calculated for material judged homogenous by thin layer chromatography and NMR. Thin layer chromatography was performed on Merck Kieselgel 60 F_{254} plates eluting with the solvents indicated, visualized by a 254 nm UV lamp, and stained with an ethanolic solution of 12-molybdophosphoric acid. Flash column chromatography was performed with Davisil 62 silica gel, slurry packed with EtOAc in glass columns, and flushed with hexanes prior to use. Radial preparative-layer chromatography (RPLC) was performed on a chromatotron (Harrison Associates, Palo Alto, CA) using glass plates coated with silica gel (P. F. 254 60) of 2- or 4-mm thickness. Nuclear magnetic resonance spectra were acquired on Unity 300 or Unity 500 spectrometer at 300 MHz or 500 MHz for ^{1}H, and 75 MHz or 125.7 MHz for ^{13}C. Chemical shifts for proton nuclear magnetic resonance (^{1}H NMR) spectra are reported in parts per million in reference to the singlet line of $CHCl_3$ at 7.24 ppm. Chemical shifts for carbon nuclear magnetic resonance (^{13}C NMR) spectra are reported in parts per million in reference to the center line of the triplet of $CDCl_3$ at 77.0 ppm. The abbreviations s, d, t, q, br s, br t, and ABq stand for the resonance multiplicities singlet, doublet, triplet, quartet, broad singlet, broad triplet, and AB quartet, respectively. IR spectra were obtained from a Mattson FT-IR GL-3020 spectrometer. Optical rotations were obtained on a Perkin Elmer 241 mc polarimeter (Na D line) using a micro cell with a 1 dm path length. Concentrations are reported in g/100 mL. Analytical C & H analysis were performed by Atlantic Microlab, Inc., Norcross, Georgia.

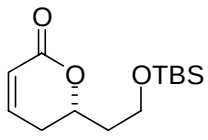
Representative procedure for the preparation of the starting β–acetoxy aldehydes.



1

(1S)-1-[2-(1,1,2,2-tetramethyl-1-silapropoxy)ethyl]but-3-enyl acetate. To a stirring solution of **1** (5.50 g, 20.18 mmol) in a 110 mL of 5:1 mixture of CH_2Cl_2 and MeOH cooled to -78 °C was bubbled in ozone until the solution turned blue; the solution was then purged with nitrogen until the blue color disappeared. Dimethyl sulfide (6 mL, excess) was added and after stirring 10 min, pH 7 buffer (200 mL) was added in portions with stirring while the reaction was allowed to self warm to rt over a period of 8 h. The mixture was separated and the aqueous layer was extracted three times with 80 mL portions of CH_2Cl_2. The combined organic phase was concentrated, redissolved in pentane (150 mL), dried over Na_2SO_4, filtered through a plug of $MgSO_4$, and concentrated in vacuo to give a pale yellow oil (5.67 g). This crude aldehyde having R_f 0.33 (25 % EtOAc/ hexanes) was immediately used for the next reaction without further purification.

Representative procedure for the lactone forming reactions:

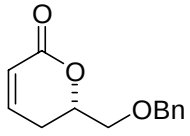


1a

(6S)-6-[2-(1,1,2,2-tetramethyl-1-silapropoxy)ethyl]-5,6-dihydro-2H-pyran-2-one. To a stirring solution of diisopropylamine (2.9 mL, 22.1 mmol) in THF (200 mL) at –78 °C was added n-butyl lithium in hexane (2.1 M, 9.0 mL, 18.5 mmol) and the mixture was stirred for 30 min at 0 °C, then re-cooled to –78 °C. Methyl acetate (1.65 mL, 20.8 mmol) was added dropwise *via* syringe and the mixture was stirred for 30 min. The crude aldehyde (maximum 20.18 mmol) in THF (20 mL) was cooled to –78 °C and added *via* a canula into the enolate solution (and the aldehyde flask was rinsed two times with 5 mL of THF). The mixture was stirred for 40 min at –78 °C, then kept in a –20 °C refrigerator for 10 h. The mixture was quenched with a saturated aqueous $NaHCO_3$ solution (20 mL) and poured into a saturated aqueous solution of $NaHCO_3$ (600 mL), extracted three times with 200 mL of CH_2Cl_2, washed with 300 mL of brine, dried over

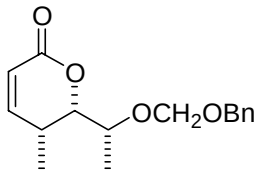
Na_2SO_4 and concentrated. Flash chromatography of the residual oil on a 7x15 cm silica gel column eluting with a 1:5 mixture of ethyl acetate-hexanes gave 4.50 g of **1a** (87% yield) as a colorless oil: R_f 0.20 (25% EtOAc/ hexanes); $[\alpha]_D^{22}$ = -40.5 (*c* 2.55, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.90 (ddd, *J* = 9.9, 4.2, 4.2 Hz. 1H), 6.04 (ddd, *J* = 9.9, 1.8, 1.8 Hz, 1H), 4.63 (dddd, *J* = 7.95, 7.89, 7.88, 4.5 Hz, 2H), 3.88-3.72 (m, 2H), 2.42-2.37 (m, 2H), 2.01 (dddd, *J* = 14.4, 8.4, 4.9, 4.8 Hz, 1H), 1.86 (dddd, *J* = 14.4, 8.7, 5.3, 5.1 Hz, 1H), 0.89 (s, 9H), 0.06 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.4, 145.4, 121.4, 75.1, 58.5, 37.8, 29.6, 25.9, 18.3, –5.4; IR (neat) 1726, 1472 cm^{-1}; Anal Calcd. for $C_{13}H_{24}O_3Si$: C, 60.89; H, 9.43. Found: C, 61.07; H, 9.36.

Analytical data for lactone products:



2a

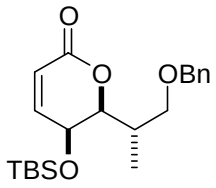
(6S)-6-[(phenylmethoxy)methyl]-5,6-dihydro-2H-pyran-2-one. Olefin **2** (85 mg, 0.36 mmol) provided 55 mg (69 % yield) of **2a** as a colorless oil. The analytical data for this product was in excellent agreement with that reported by Saburi[1]. R_f 0.29 (25% EtOAc/ hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.29 (m, 5H), 6.92 (ddd, *J* = 9.8, 5.9, 2.4 Hz, 1H), 6.05 (ddd, *J* = 9.8, 2.4, 1.0 Hz, 1H) ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 163.7, 144.9, 137.6, 128.5, 127.8, 127.7, 121.2, 76.6, 73.6, 70.7, 26.2.



3a

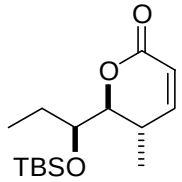
6-{(1R)-1-[(phenylmethoxy)methoxy]ethyl}(6S,5R)-5-methyl-5,6-dihydro-2H-pyran-2-one. Olefin **3** (145 mg, 0.531 mmol) provided 86 mg (61 % yield) of **3a** as a colorless oil: R_f 0.16, mp (45% EtOAc/ hexanes); $[\alpha]_D^{23}$ = +225.5 (*c* 2.80, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.20 (m, 5H), 6.98 (dd, *J* = 9.5, 6.3 Hz, 1H), 6.02 (d, *J* = 9.5 Hz, 1 Hz, 1H), 4.99 (d, *J* = 7.3 Hz, 1H), 4.93 (d, *J* = 7.0 Hz, 1H), 4.75 (ABq, ΔAB = 16.5 Hz, 1H), 4.32 (dd, *J* = 8.8, 3.2 Hz, 1H), 4.04 (dq, *J* = 8.8, 6.1 Hz, 1H), 2.46 (ddq, *J* = 7.1, 6.4, 3.2 Hz, 1H), 1.26 (d, *J* = 6.4 Hz, 3H) 1.07 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 163.9, 156.9, 137.7, 128.3, 128.0, 127.6, 120.0, 94.3, 83.2,

72.1, 69.5, 29.9, 16.4, 11.1; IR (neat) 1875, 1497 cm^{-1}; Anal. Calcd for $C_{16}H_{20}O_4$: C, 69.54; H, 7.30. Found: C, 69.42; H, 7.29.



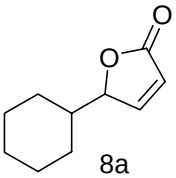
4a

6-[(1S)-1-methyl-2-(phenylmethoxy)ethyl](5S,6S)-5-(1,1,2,2-tetramethyl-1-silapropoxy)-5,6-dihydro-2H-pyran-2-one. Aldehyde **4** (497 mg, 1.26 mmol) yielded 354 mg (75% yield) of **4a** as a colorless solid: mp 84-86 °C; $[\alpha]_D^{22}$ = -70.5 (*c* 1.12, $CHCl_3$); R_f 0.29 (25% EtOAc/ hexanes), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.20 (m, 5 H), 6.84 (dd, *J* = 10.0, 6.0 Hz, 1 H), 6.05 (d, *J* = 10.0 Hz, 1 H), 4.47 (ABq, ΔAB = 14.2 Hz, *J* = 11.5 Hz, 2 H), 4.17-4.13 (m, 2 H), 3.72 (dd, *J* = 9.0, 5.0 Hz, 1 H), 3.56 (dd, *J* = 9.0, 3.0 Hz, 1 H), 2.33-2.25 (m, 1 H), 1.03 (d, *J* = 7.5 Hz, 3 H), 0.81 (s, 9H), 0.06 (s, 3 H), 0.01 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 163.9, 144.4, 138.9, 128.5, 127.73, 127.68, 123.2, 81.9, 73.4, 71.1, 61.2, 34.1, 25.8, 18.2, 13.6, -3.17, -4.48; IR ($CDCl_3$) 1726, 1471 cm^{-1}; Anal. Calcd for $C_{21}H_{32}OSi$: C, 66.98; H, 8.56. Found: C, 66.82; H, 8.49.



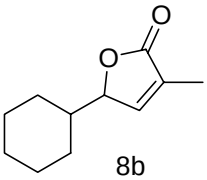
6a

6-[(1S)-1-(1,1,2,2-tetramethyl-1-silapropoxy)propyl](5S,6S)-5-methyl-5,6-dihydro-2H-pyran-2-one. Aldehyde **6** (343 mg, 1.13 mmol) yielded 234 mg (73% yield) of **6a** as a colorless oil: $[\alpha]_D^{22}$ = -11.3 (*c* 1.39, $CHCl_3$); R_f 0.27 (25% EtOAc/ hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.66 (dd, *J* = 9.5, 2.5 Hz, 1 H), 5.94 (dd, *J* = 9.5, 2.5 Hz, 1 H), 4.07 (dd, *J* = 9.5, 2.5 Hz, 1 H), 3.76 (td, *J* = 6.8, 2.5 Hz, 1 H), 2.84-2.76 (m, 1 H), 1.88-1.79 (m, 1 H), 1.57-1.48 (m, 1 H), 1.18 (d, *J* = 7.0 Hz, 3 H), 0.93 (t, *J* = 7.5 Hz, 3 H), 0.89 (s, 9 H), 0.09 (s, 3 H), 0.08 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.2, 152.5, 119.8, 84.4, 73.9, 29.7, 26.0, 25.8, 18.3, 17.0, 10.5, -4.01, -4.21; IR (neat) 1735, 1463 cm^{-1}; Anal. Calcd for $C_{15}H_{28}O_3Si$: C, 63.33; H, 9.92. Found: C, 63.44; H, 9.94.



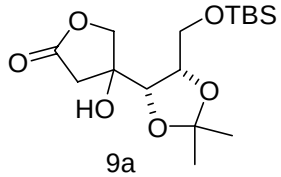
8a

5-cyclohexyl-5-hydrofuran-2-one. Olefin **8** (133 mg, 0.731 mmol) provided 75 mg of **8a** (62 % yield) as a colorless solid: mp 68-72 ^{o}C d. (lit mp 71 ^{o}C [2]); The analytical data for this product was in excellent agreement with that reported by Knochel.[2] R_f 0.34 (35% EtOAc/ hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (dd, *J* = 5.9, 1.5, Hz, 1H), 6.14 (dd, *J* = 5.9, 2.0 Hz, 1H), 4.87 (ddd, *J* = 5.4, 1.7, 1.7 Hz, 1H), 1.83-1.70 (m, 6H), 1.34-1.06 (m,5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.2, 155.0, 121.8, 87.5, 41.2, 28.5, 28.1, 26.0, 25.7, 25.6. (Although the mp of this known compound was somewhat different from that reported, it did return a satisfactory C, H combustion analysis).



8b

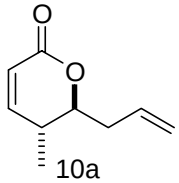
5-cyclohexyl-3-methyl-5-hydrofuran-2-one. Olefin **8** (172 mg, 0.945 mmol) yielded 79 mg (77 % yield) of lactone **8b** as a colorless oil. The analytical data for this product was in excellent agreement with that reported by Marshall.[3] R_f 0.41 (35% EtOAc/ hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.06 (dq, *J* = 1.6, 1.6 Hz, 1H), 4.68 (ddq, *J* = 5.6, 1.6, 1.6 Hz, 1H), 1.92 (t, *J* = 1.6 Hz, 3H), 1.82-1.73 (m, 6H), 1.72-1.59 (m, 6H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 174.4, 147.5, 130.2, 85.2, 41.4, 28.4, 28.2, 26.0, 25.7, 25.6, 10.6.



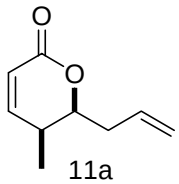
9a

4-{(4S,5S)-2,2-dimethyl-5-[(1,1,2,2-tetramethyl-1-silapropoxy) methyl] (1,3-dioxolan-4-yl)}-4-hydroxy-3,4,5-trihydrofuran-2-one. Ketone **9** (317 mg, 0.869 mmol) yielded 184 mg (64% yield) of **9a** as a colorless oil: R_f 0.15 (25% EtOAc/ hexanes); $[\alpha]_D^{22}$ = -24.6 (*c* 2.02, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.68 (br s, 1 H), 4.59 (d, *J* = 10.0 Hz, 1 H), 4.34 (d, *J* = 6.0 Hz, 1 H), 4.29 (d, *J* = 10.0 Hz, 1 H), 4.27 (ddd, *J* = 10.0, 6.3, 4.0 Hz, 1 H), 3.76 (dd, *J* = 11.3, 4.0 Hz, 1 H), 3.63 (dd, *J* = 11.3, 10.0 Hz, 1

H), 2.84 (d, *J* = 17.5 Hz, 1 H), 2.59 (d, *J* = 17.5 Hz, 1 H), 1.45 (s, 3H), 1.37 (s, 3 H), 0.92 (s, 9H), 0.16 (s, 3 H), 0.15 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 175.1, 109.2, 79.5, 77.0, 76.8, 75.8, 62.0, 40.9, 27.6, 25.9, 25.1, 18.4, -5.31, -5.33; IR (neat) 1787 cm^{-1}; Anal. Calcd for $C_{16}H_{30}O_6Si$: C, 55.46; H, 8.73. Found: C, 55.30; H, 8.74.



10a (±)-5-methyl-6-prop-2-enyl-5,6-dihydro-2H-pyran-2-one. R_f 0.43 (35 % EtOAc/ hexanes); ^{1}H NMR (500 MHz, $CDCl_3$); δ 6.63 (dd, *J* = 9.8, 2.4 Hz, 1H), 5.95 (dd, *J* = 9.8, 2.4 Hz, 1H), 5.94-5.98 (m, 1H), 5.16-5.12 (m, 2H), 4.14 (ddd, *J* = 10.3, 6.4, 3.9 Hz, 1H), 2.58-2.49 (m, 2H), 2.45-2.39 (m, 1H), 1.11 (d, *J* = 7.32 Hz, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.1, 151.6, 132.6, 120.1, 118.6, 82.8, 36.8, 32.2, 16.0.



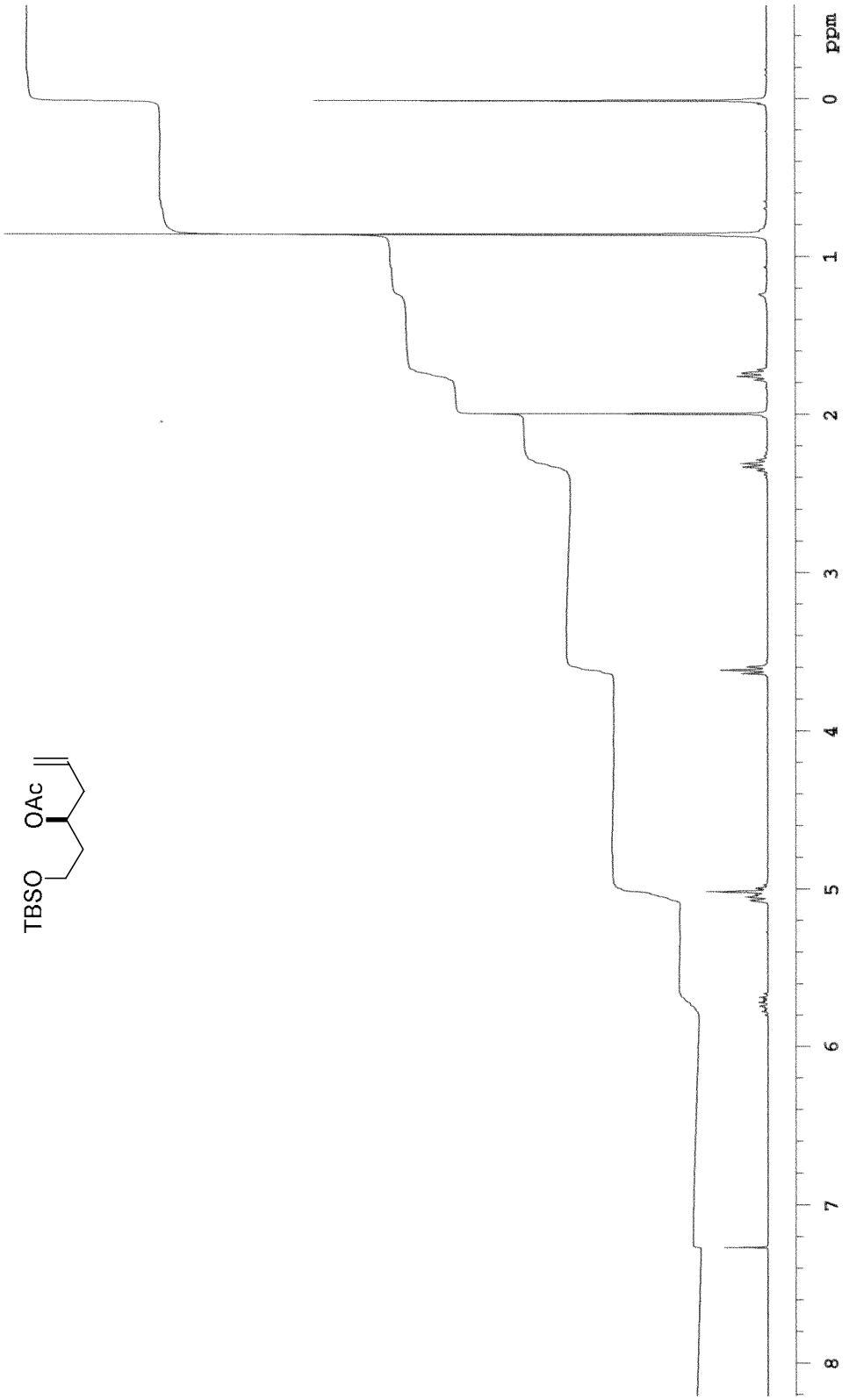
11a (±)-5-methyl-6-prop-2-enyl-5,6-dihydro-2H-pyran-2-one. R_f 0.43 (35 % EtOAc/ hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.97 (dd, *J* = 9.8, 6.4 Hz, 1H), 5.98 (d, *J* = 9.8 Hz, 1H), 5.81 (dddd, *J* = 16.6, 13.7, 7.8, 5.9 Hz, 1H), 5.21 (dd, *J* = 17.1, 1.5 Hz, 1H), 5.46 (d, *J* = 10.3, 1H), 4.47 (ddd, *J* = 7.3, 7.3, 3.4 Hz, 1H), 2.63-2.58 (m, 1H), 2.44-2.33 (m, 2H), 1.06 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.5, 151.6, 132.5, 120.0, 118.6, 79.3, 35.8, 31.4, 11.0.

References:

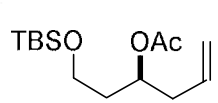
(1) Shoa, L.; Hiroyuki, K.; Saburi, M.; Uchida, Y. *Tetrahedron*, **1993**, *49*, 1997.

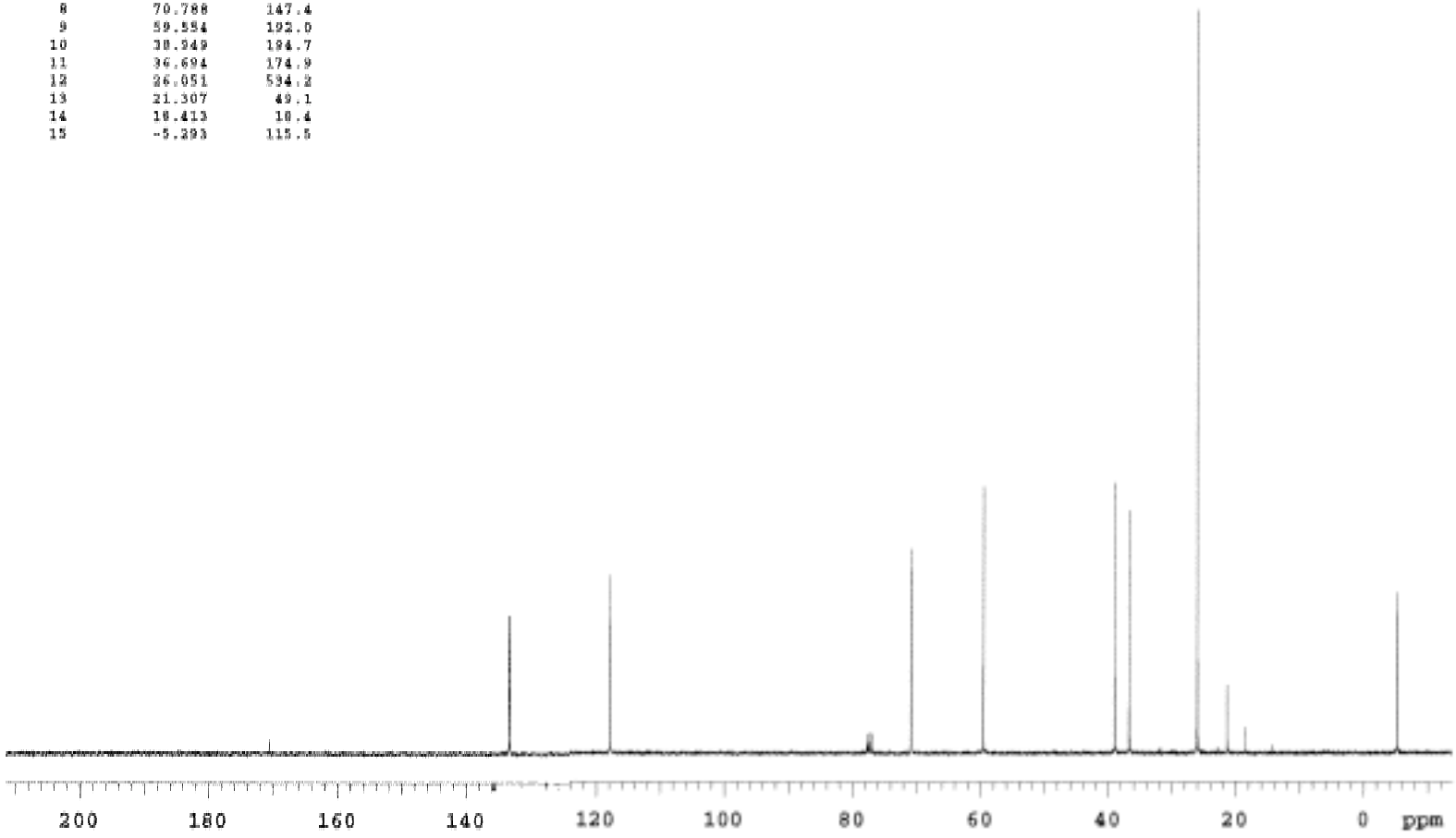
(2) Knochel, P.; Rao, C. J. *Tetrahedron*, **1993**, *49*, 29.

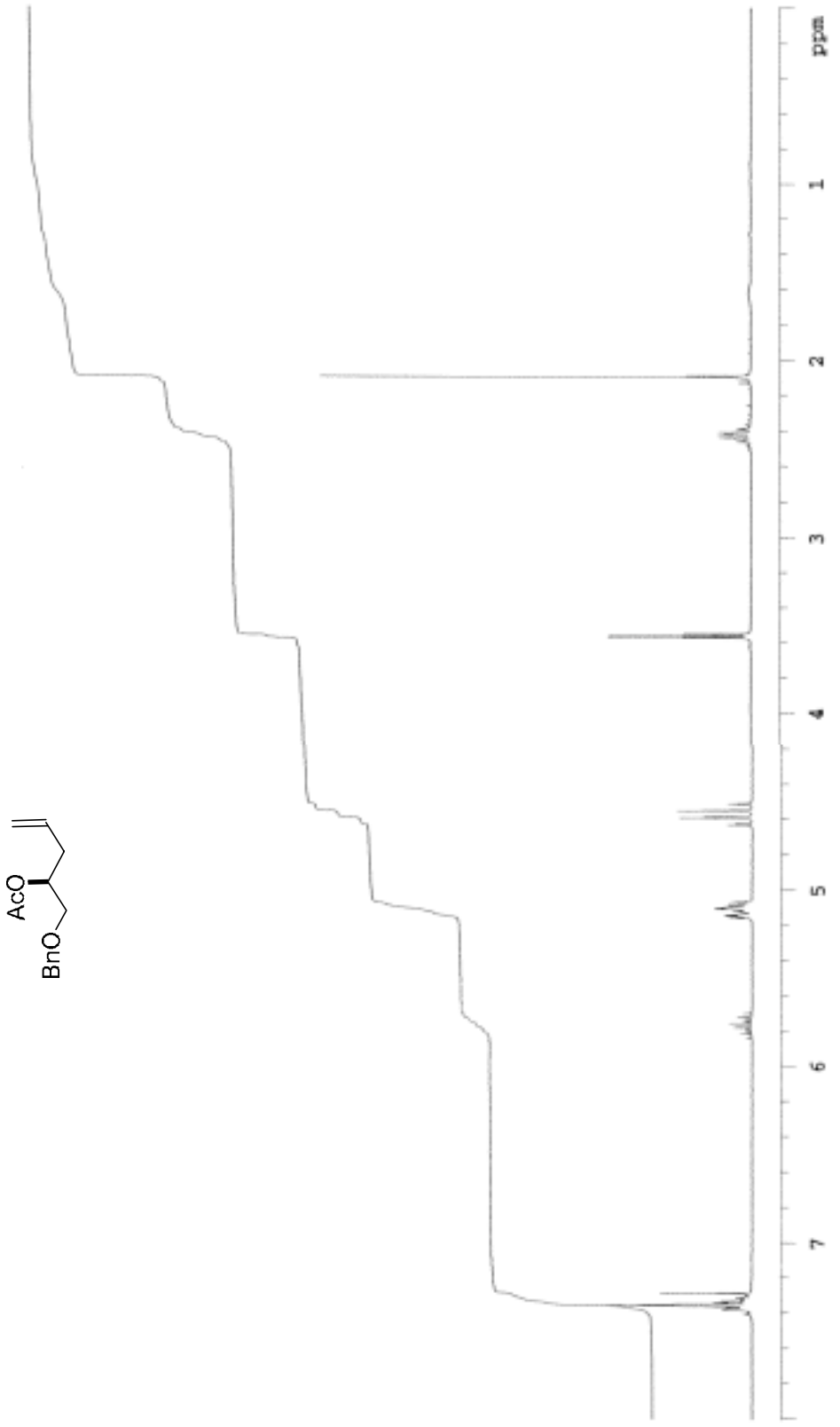
(3) Marshall, J. A.; Wolf, M. A.; Wallace, E. M. *J.Org.Chem.*, **1997,** *62*, 367.

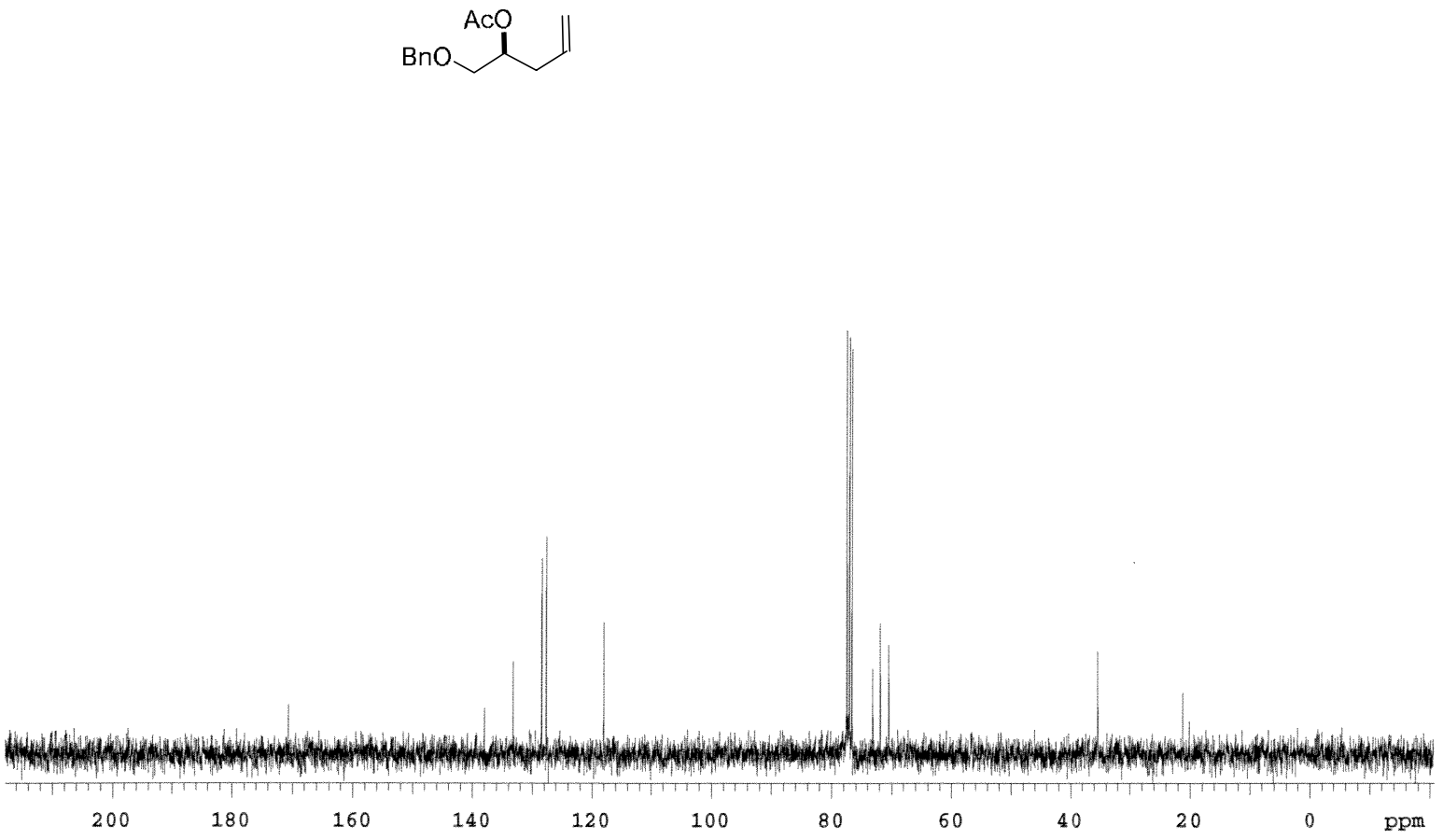

TBSO
OAc
0
1
2
3
4
5
6
7
8
ppm

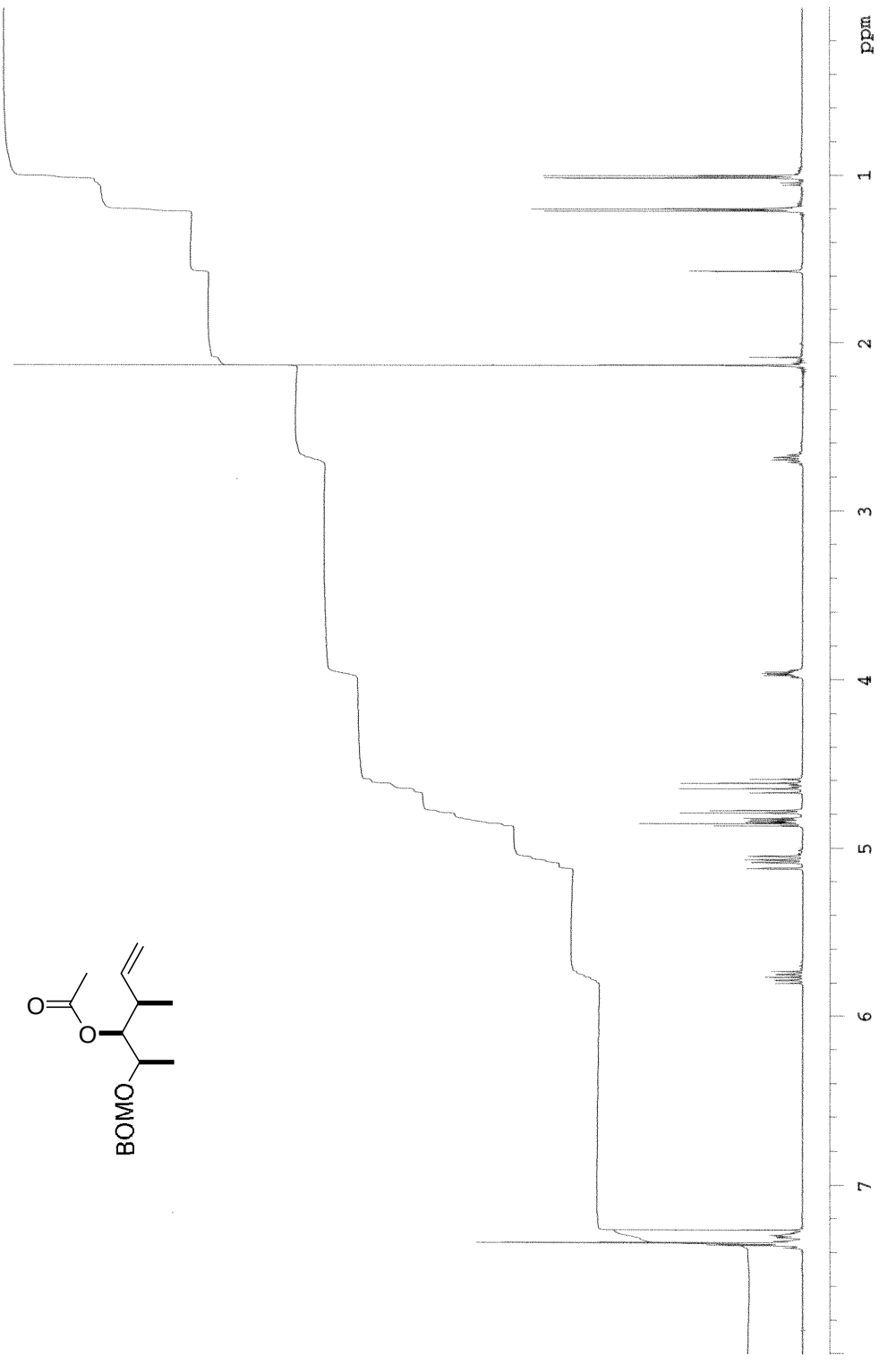
INDEX	FREQUENCY (PPM)	HEIGHT
1	170.574	9.2
2	133.778	98.4
3	117.842	128.0
4	77.656	13.5
5	77.429	9.0
6	77.230	14.6
7	76.804	13.9
8	70.788	147.4
9	59.554	192.0
10	38.949	194.7
11	36.694	174.9
12	26.051	534.2
13	21.307	49.1
14	18.413	18.4
15	-5.293	115.5



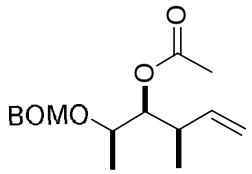


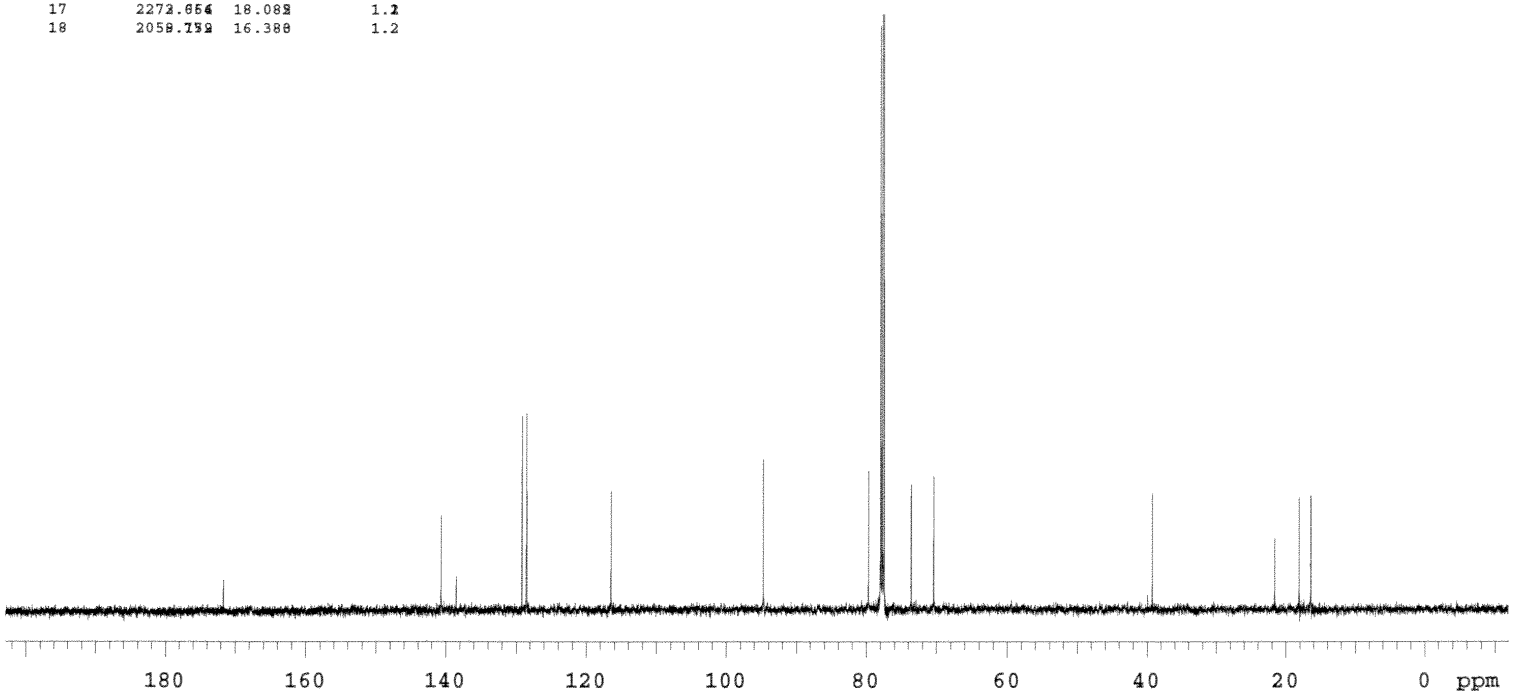

AcO
BnO
ppm
1
2
3
4
5
6
7

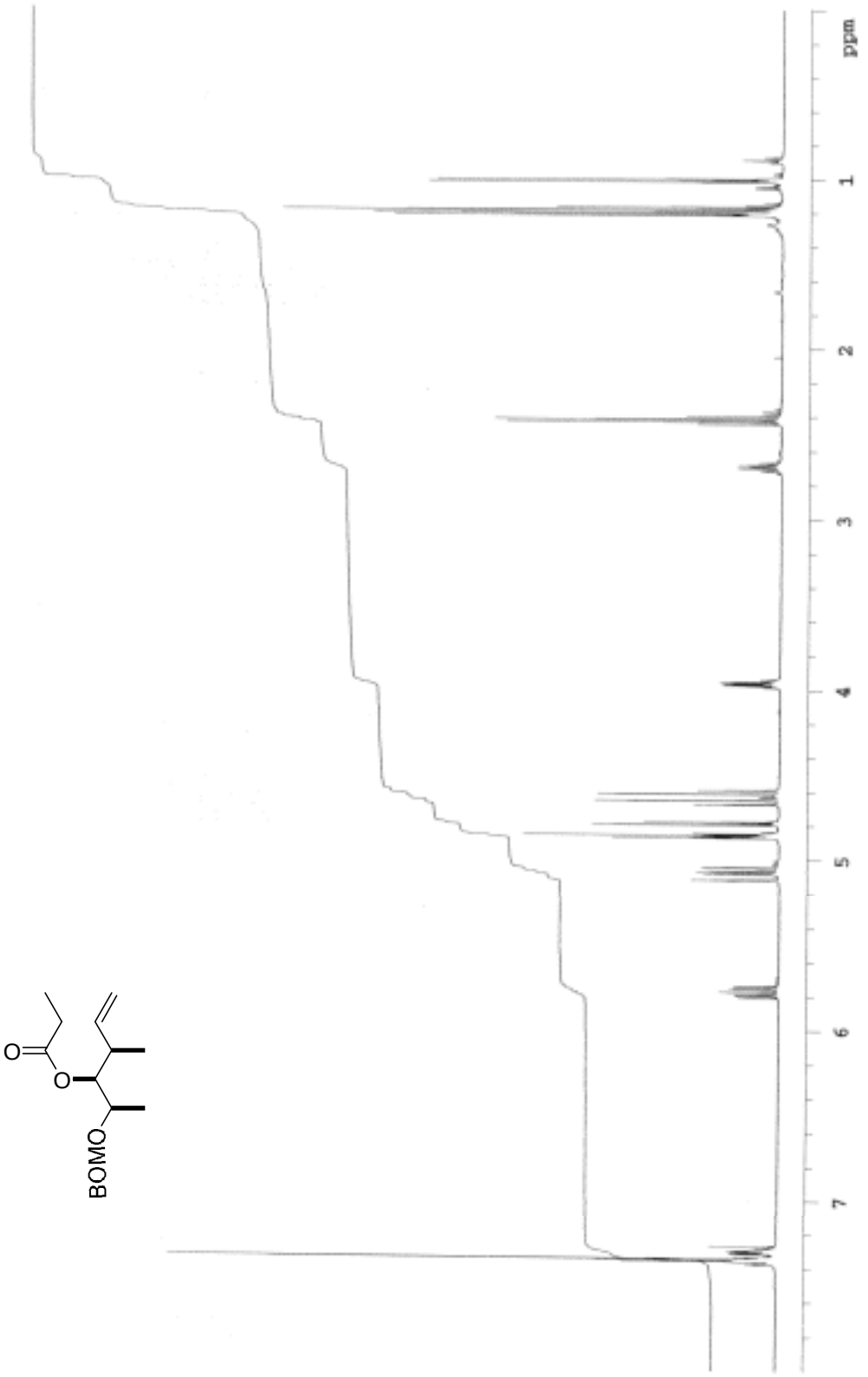

AcO
BnO
200
180
160
140
120
100
80
60
40
20
0
ppm


O
O
BOMO
ppm
1
2
3
4
5
6
7

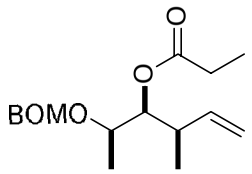
INDEX	FREQUENCY	PPM	HEIGHT
1	21575.657	171.658	0.3
2	17680.451	140.676	1.0
3	17408.625	138.509	0.4
4	16235.946	129.177	2.0
5	16151.872	128.508	2.0
6	16144.866	128.452	1.8
7	14641.611	116.484	1.2
8	11908.607	94.747	1.6
9	10014.881	79.681	1.5
10	9798.927	77.962	6.2
11	9767.193	77.710	6.3
12	9735.047	77.454	6.8
13	9247.912	73.578	1.3
14	8844.027	70.365	1.4
15	4930.464	39.228	1.2
16	2714.454	21.597	0.8
17	2272.654	18.082	1.2
18	2059.752	16.388	1.2

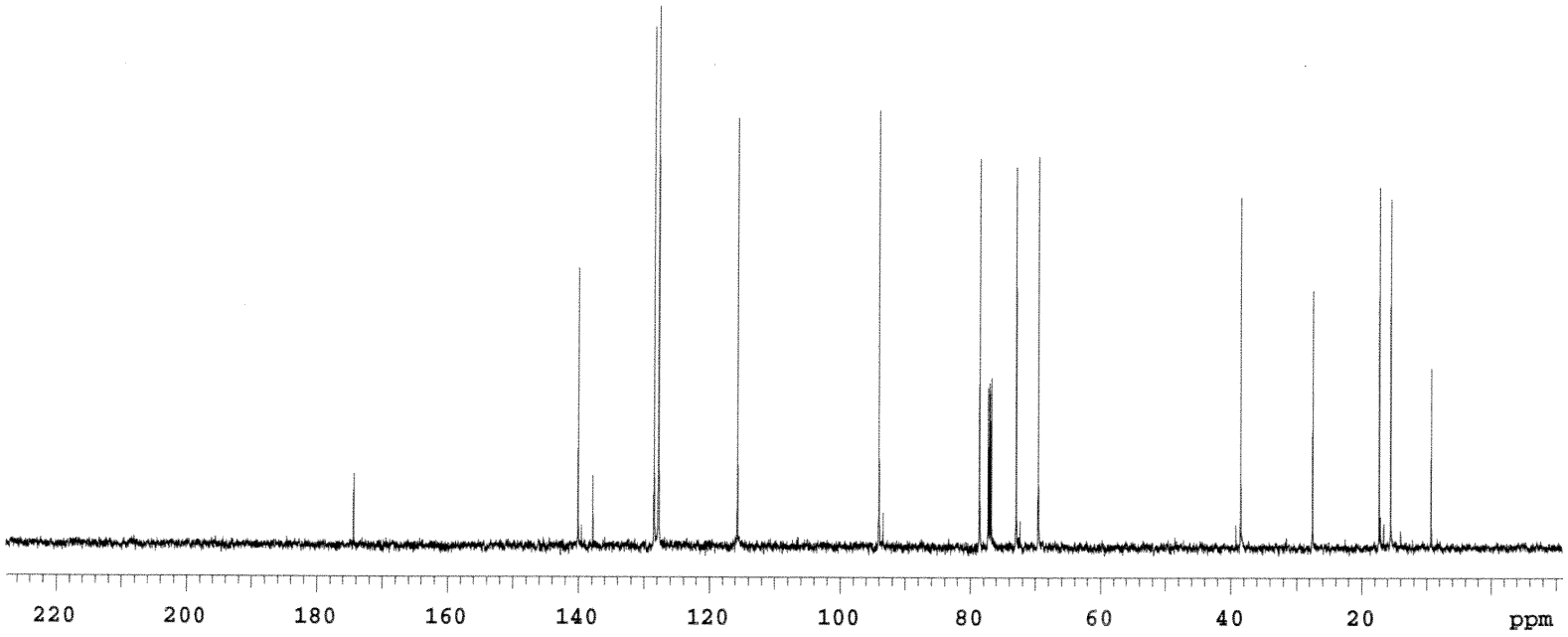


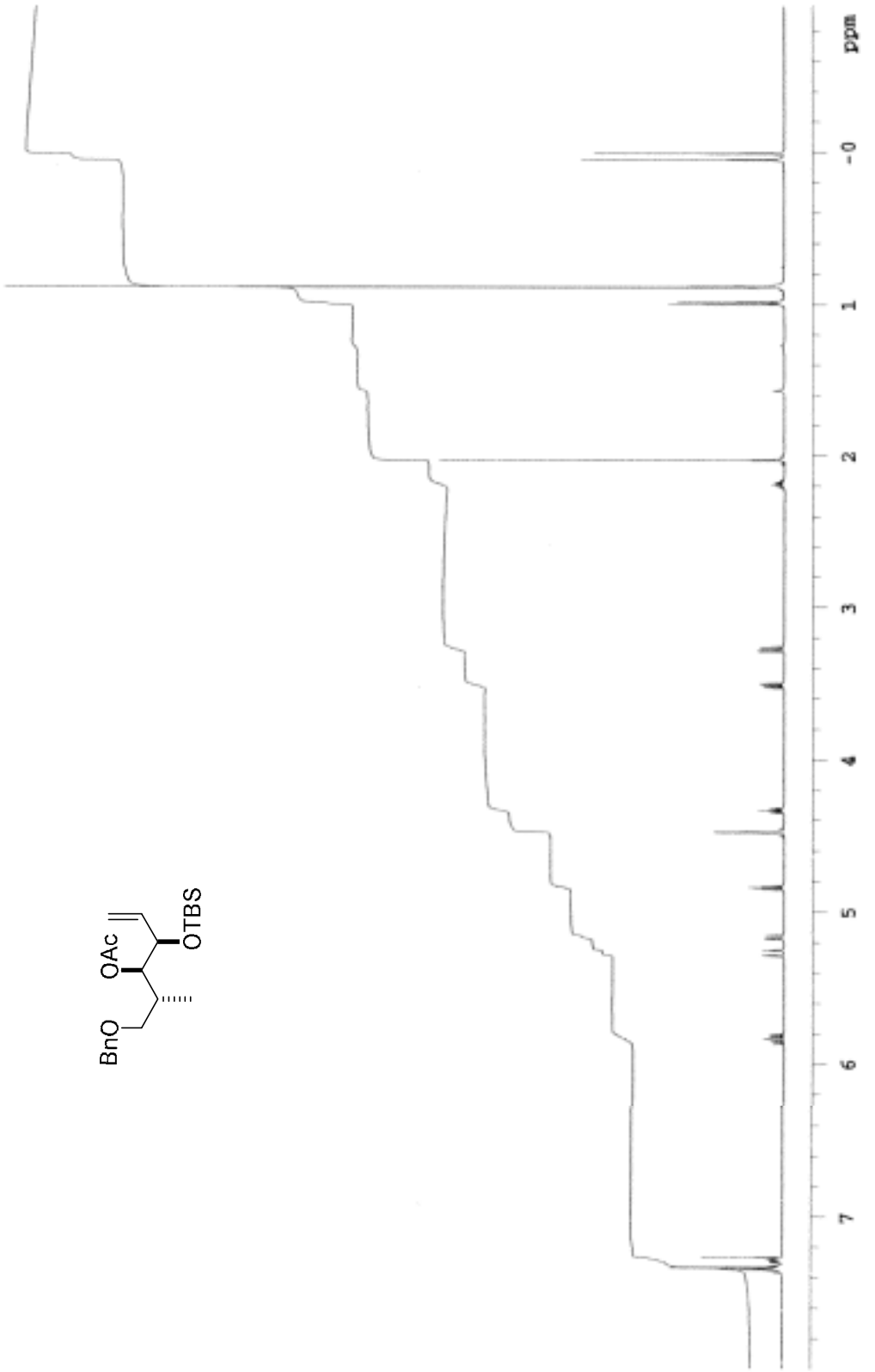



O
O
BOMO
ppm
1
2
3
4
5
6
7

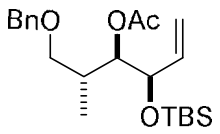
INDEX	FREQUENCY	PPM	HEIGHT
1	21917.869	174.397	11.3
2	17601.386	140.051	44.1
3	17321.160	137.821	11.1
4	16143.479	128.451	82.4
5	16061.060	127.795	85.8
6	16051.902	127.722	61.6
7	14535.387	115.656	67.9
8	11817.839	94.032	69.2
9	9878.695	78.603	61.5
10	9709.278	77.255	25.1
11	9677.226	77.000	25.9
12	9645.632	76.749	26.6
13	9169.431	72.960	60.2
14	8745.887	69.590	61.8
15	4837.380	38.490	55.5
16	3457.772	27.513	40.5
17	2176.609	17.319	57.1
18	1952.704	15.537	55.2
19	1168.346	9.296	28.3

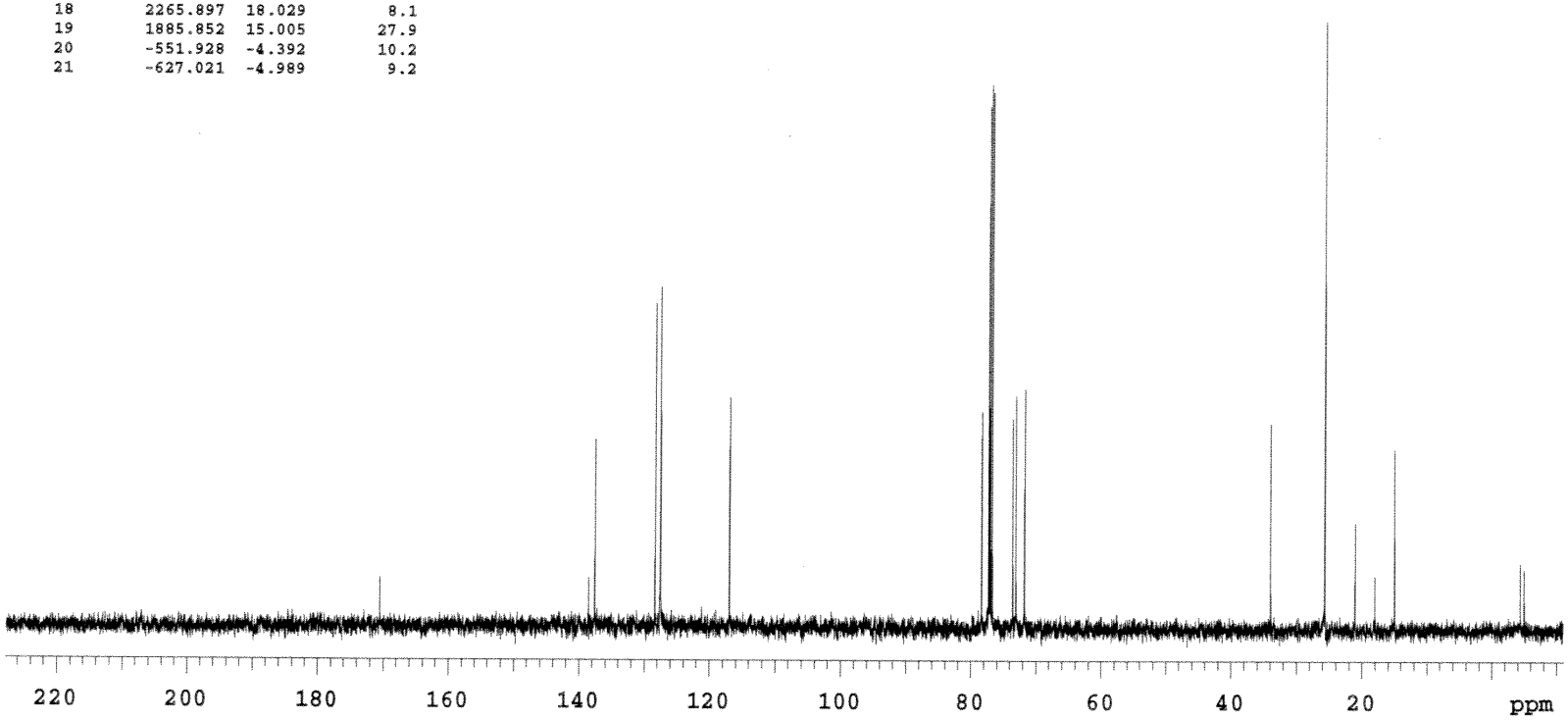


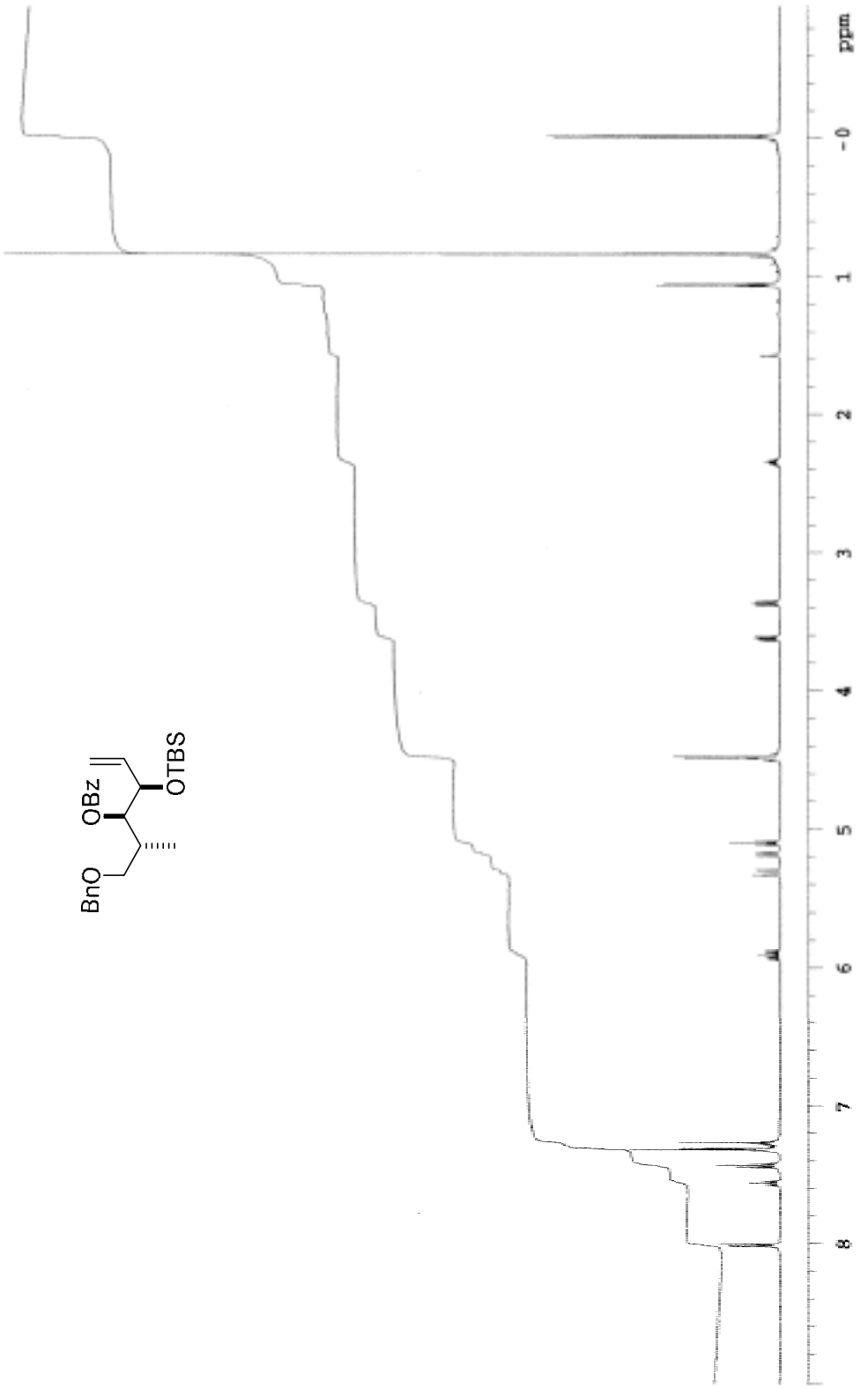


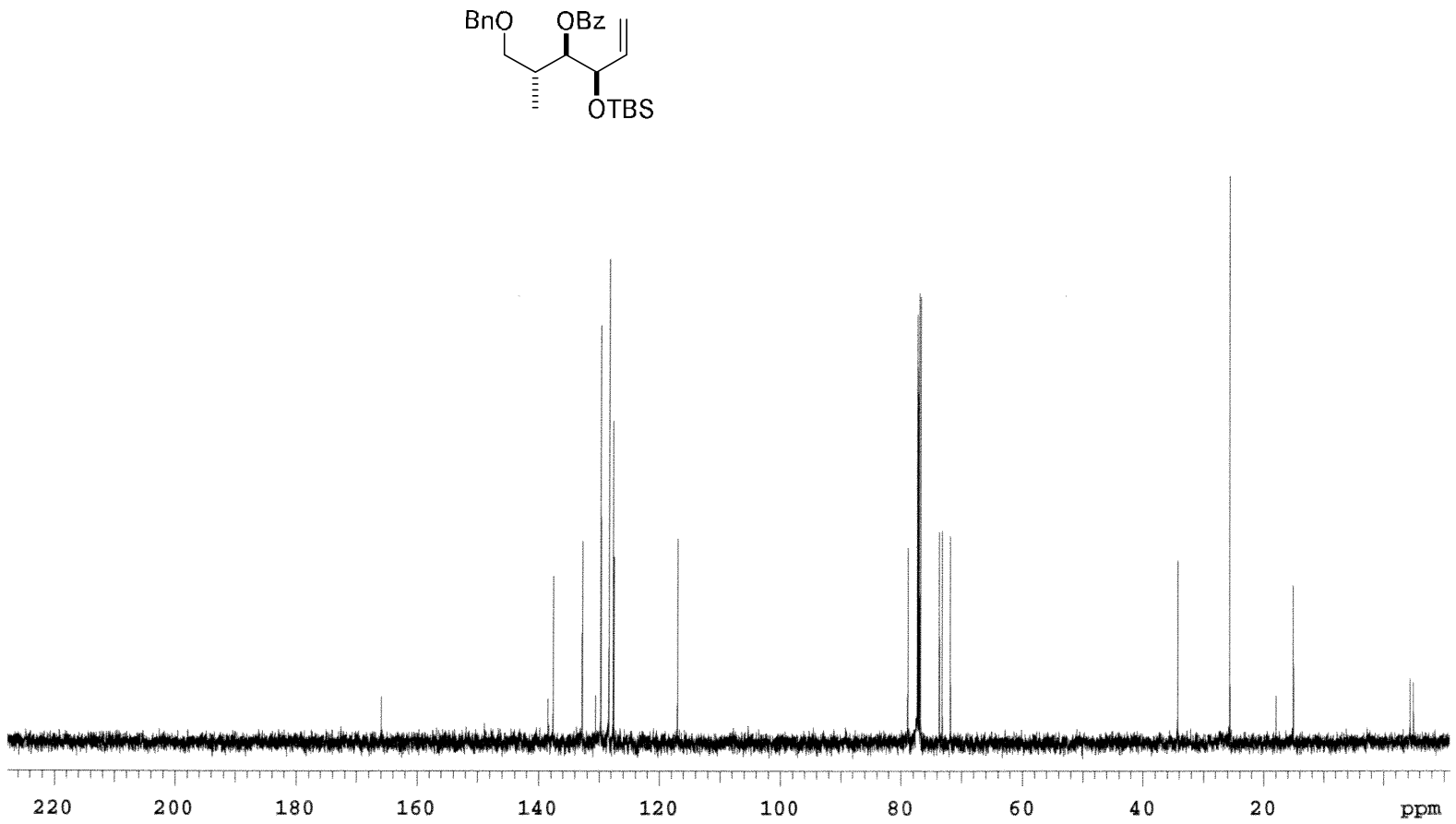

BnO
OAc
OTBS
ppm
-0
1
2
3
4
5
6
7

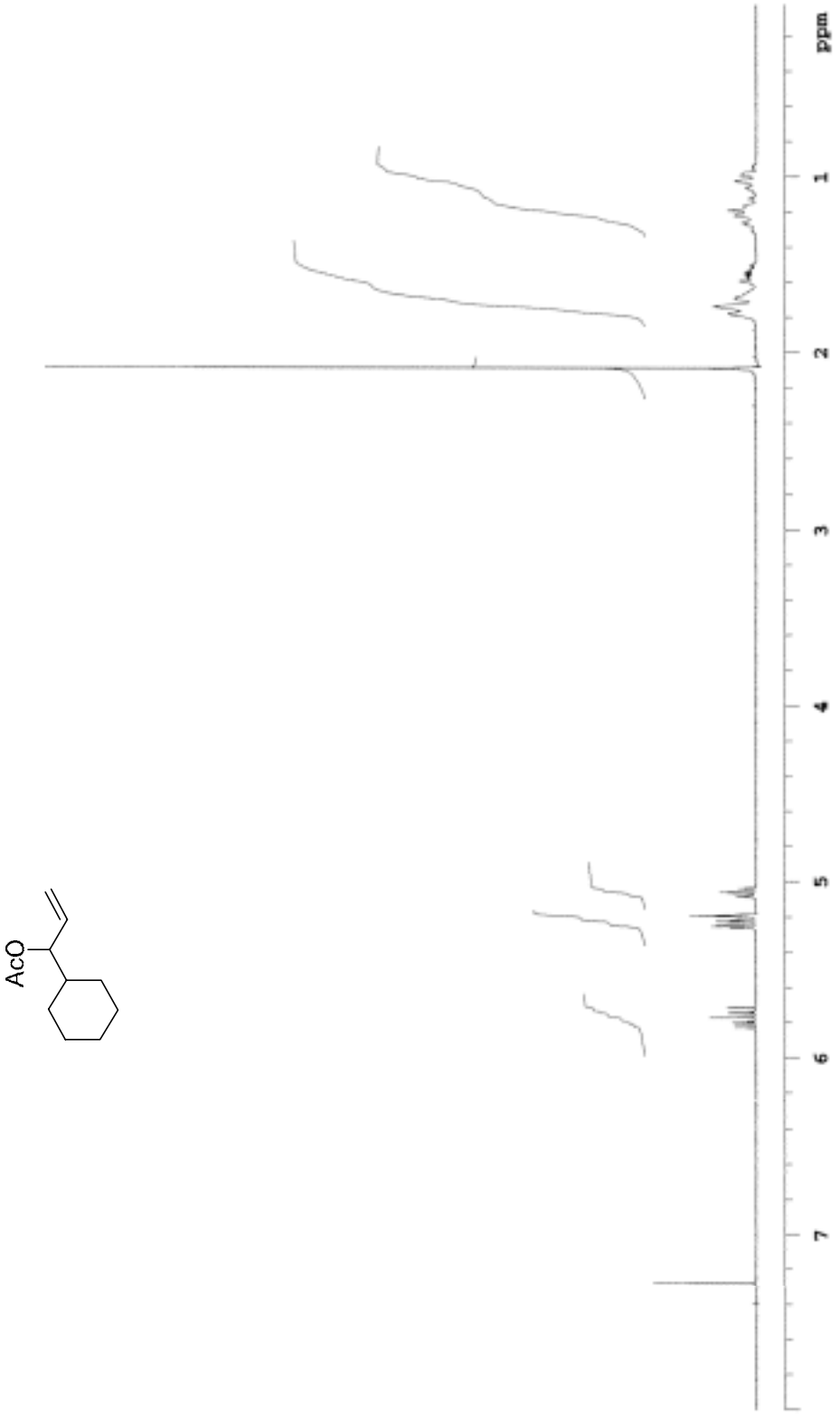
INDEX	FREQUENCY	PPM	HEIGHT
1	21427.474	170.495	7.7
2	17411.363	138.539	7.7
3	17289.108	137.566	29.2
4	16128.369	128.331	50.4
5	16035.418	127.591	52.9
6	16023.513	127.496	33.4
7	14695.189	116.927	35.8
8	9842.064	78.312	33.7
9	9709.277	77.255	81.1
10	9677.225	77.000	84.5
11	9645.174	76.745	83.2
12	9244.066	73.553	32.5
13	9184.541	73.080	36.0
14	9016.955	71.746	37.1
15	4269.602	33.972	31.8
16	3229.287	25.695	94.3
17	2646.857	21.061	16.5
18	2265.897	18.029	8.1
19	1885.852	15.005	27.9
20	-551.928	-4.392	10.2
21	-627.021	-4.989	9.2



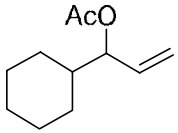


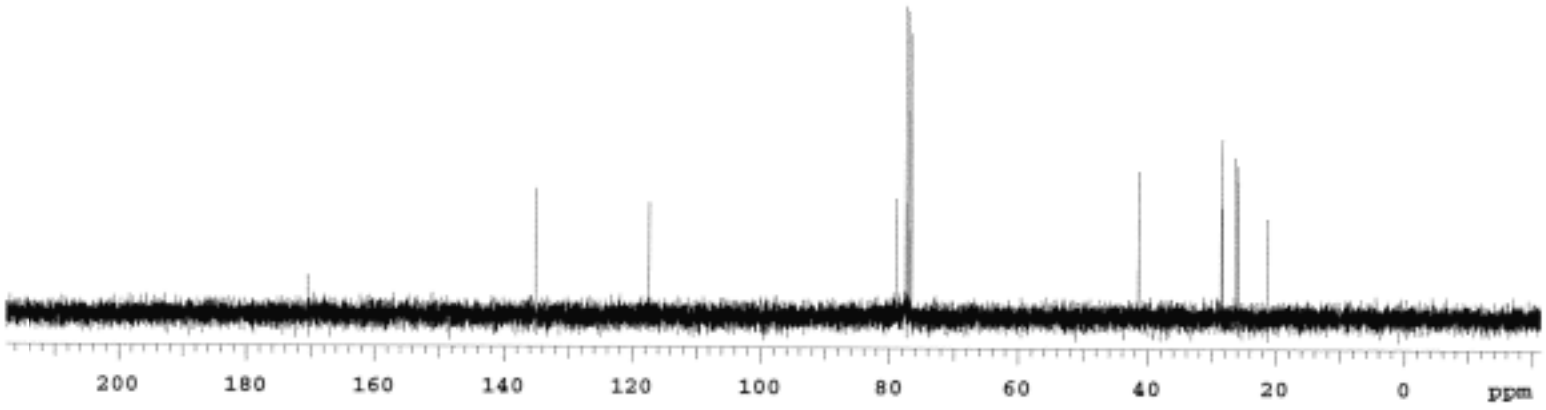

BnO
OBz
OTBS
ppm
0
1
2
3
4
5
6
7
8

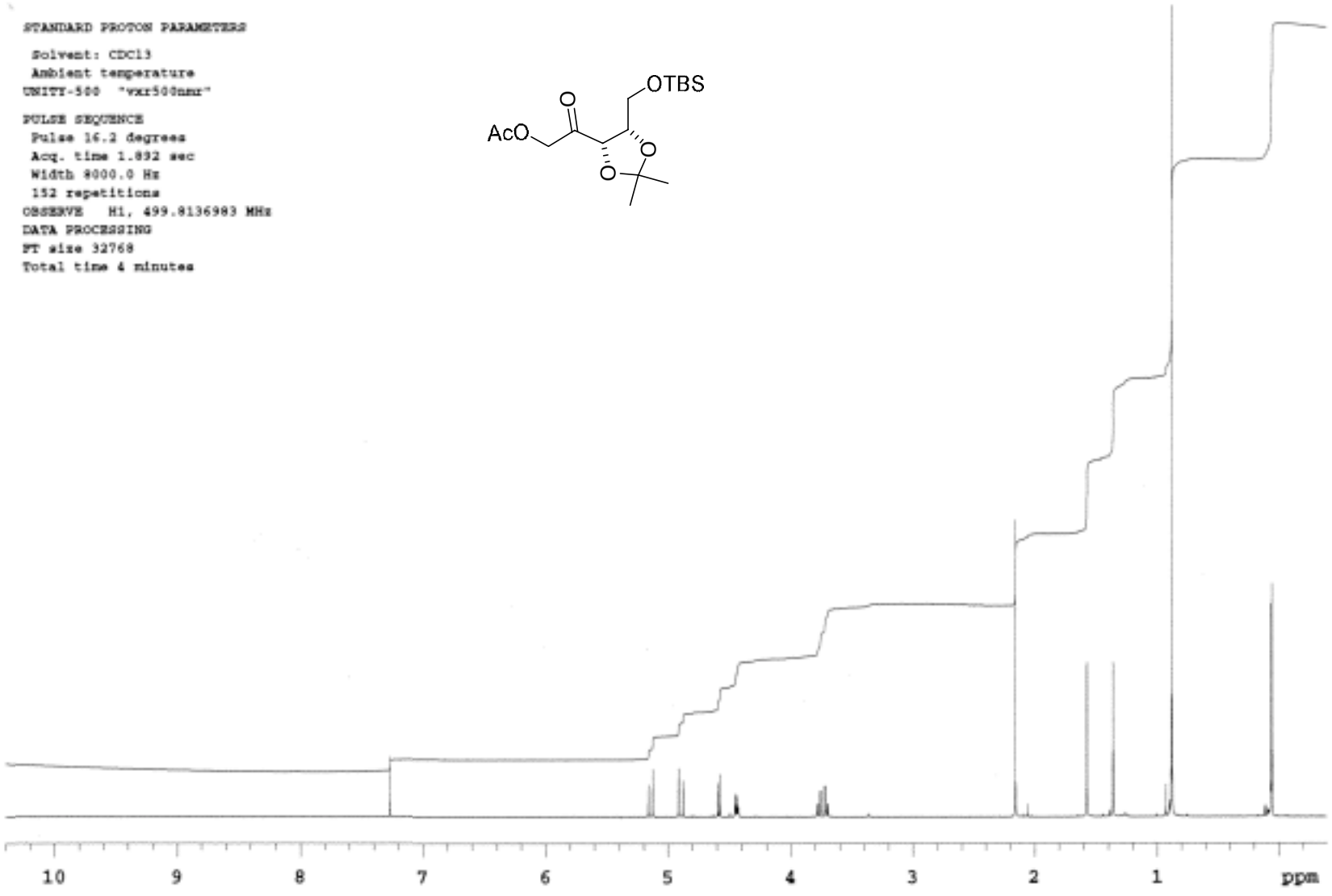

BnO
OBz
OTBS
220
200
180
160
140
120
100
80
60
40
20
ppm


AcO
ppm
1
2
3
4
5
6
7

INDEX	FREQUENCY (PPM)	HEIGHT
1	170.417	0.0
2	135.106	0.1
3	117.392	0.1
4	78.695	0.1
5	77.423	0.3
6	77.000	0.3
7	76.577	0.3
8	41.441	0.2
9	28.531	0.1
10	28.392	0.2
11	26.322	0.2
12	25.921	0.2
13	25.892	0.1
14	21.205	0.1





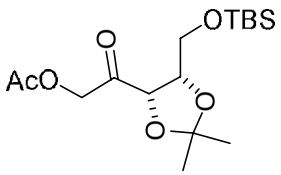

STANDARD PROTON PARAMETERS
Solvent: CDCl3
Ambient temperature
UNITY-500 "vxr500nmr"
PULSE SEQUENCE
Pulse 16.2 degrees
Acq. time 1.832 sec
Width 8000.0 Hz
152 repetitions
OBSERVE H1, 499.8136983 MHz
DATA PROCESSING
FT size 32768
Total time 4 minutes
OTBS
AcO
O
O
O
10
9
8
7
6
5
4
3
2
1
ppm

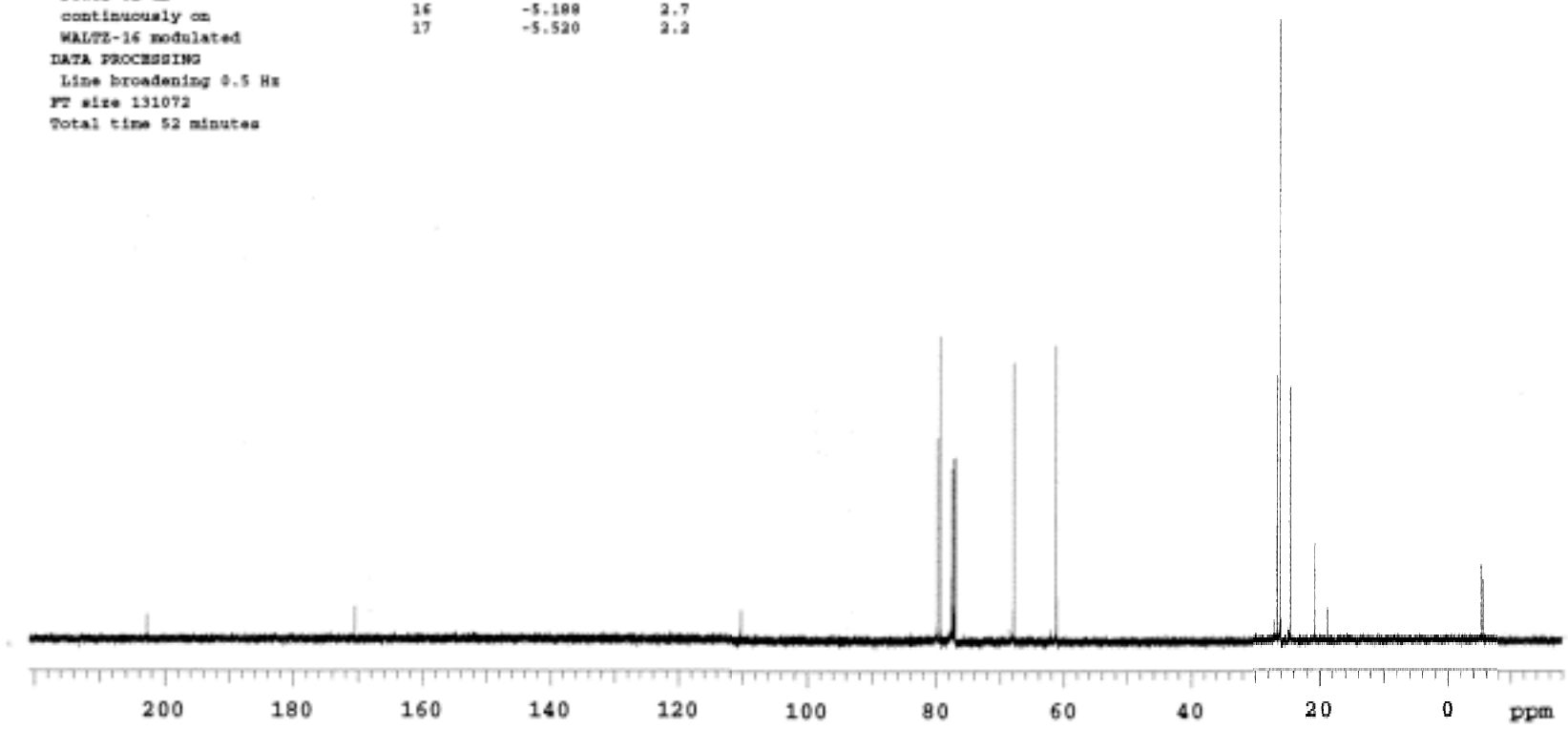
STANDARD CARBON PARAMETERS

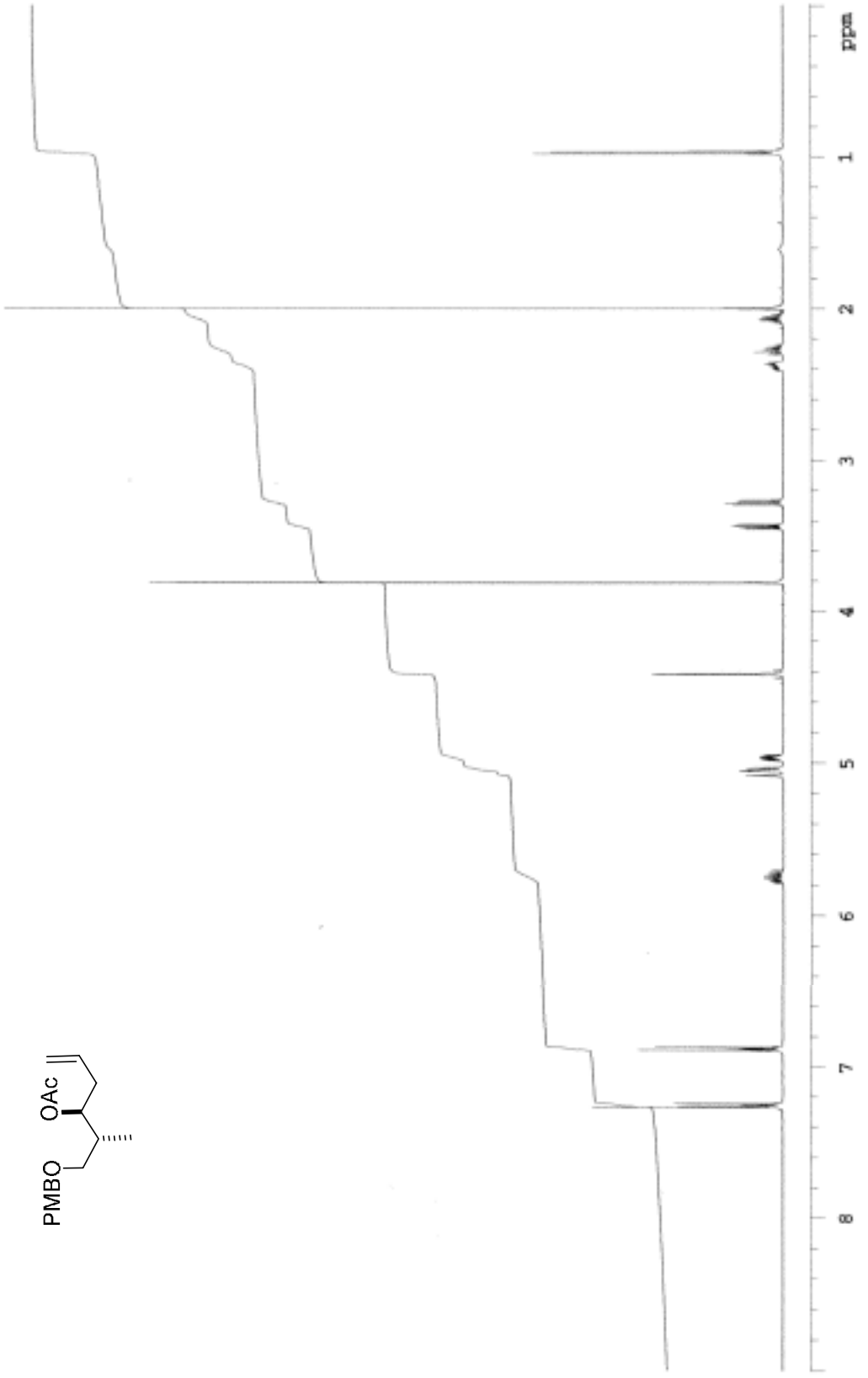
Solvent: CDCl3
Ambient temperature
User: 1-14-87
UNITY-500 "vxr500nmr"

PULSE SEQUENCE
Pulse 99.5 degrees
Acq. time 1.301 sec
Width 30030.0 Hz
2416 repetitions
OBSERVE C13, 125.6782258 MHz
DECOUPLE H1, 499.8151988 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 52 minutes

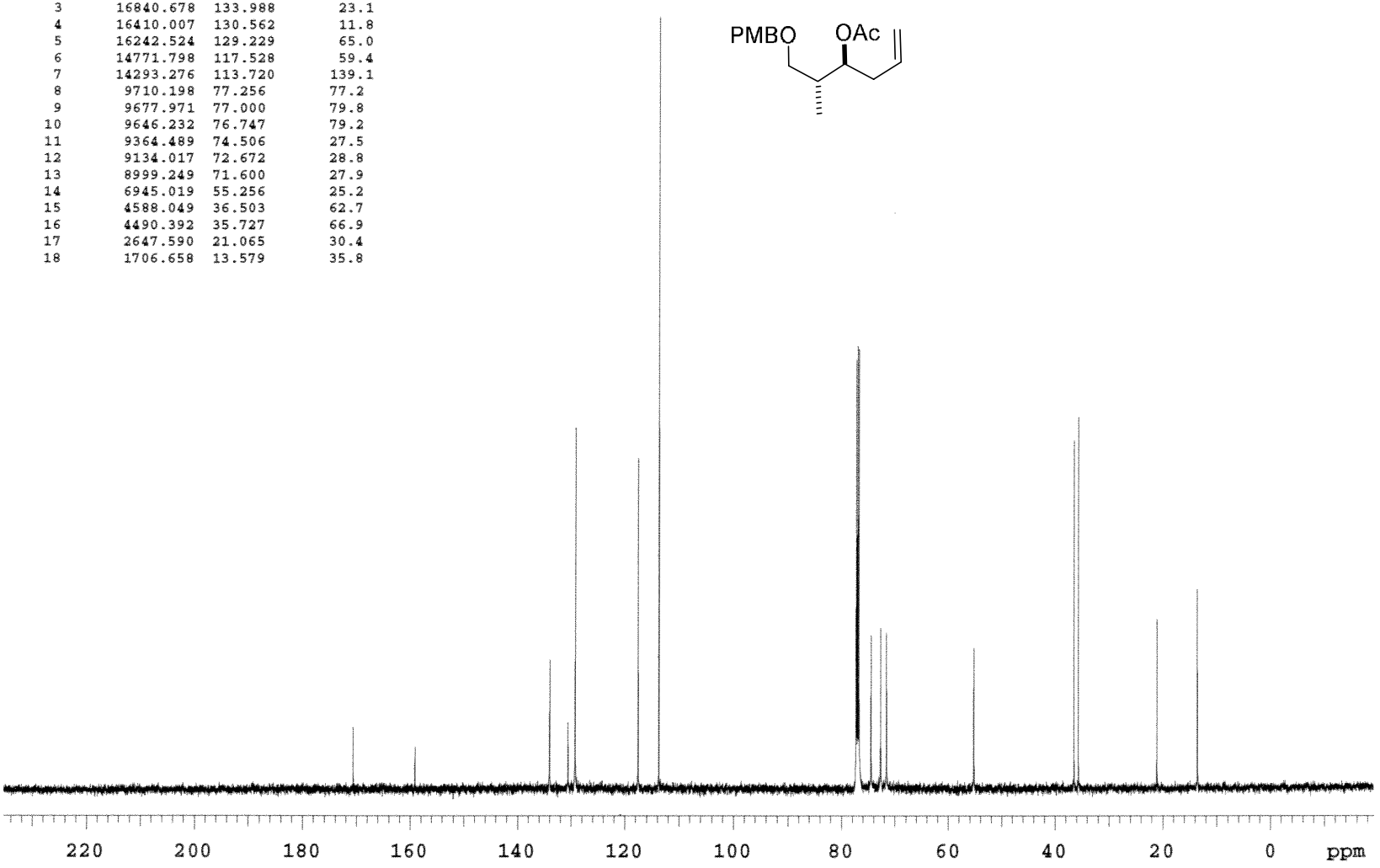
INDEX	FREQUENCY (PPM)	HEIGHT
1	202.820	0.9
2	170.450	1.2
3	110.223	1.1
4	79.822	7.4
5	79.374	11.1
6	77.485	6.3
7	77.230	6.7
8	76.975	6.7
9	67.839	10.2
10	61.222	10.8
11	26.617	9.7
12	26.074	22.7
13	24.554	9.2
14	20.780	3.5
15	18.779	1.1
16	-5.188	2.7
17	-5.520	2.2

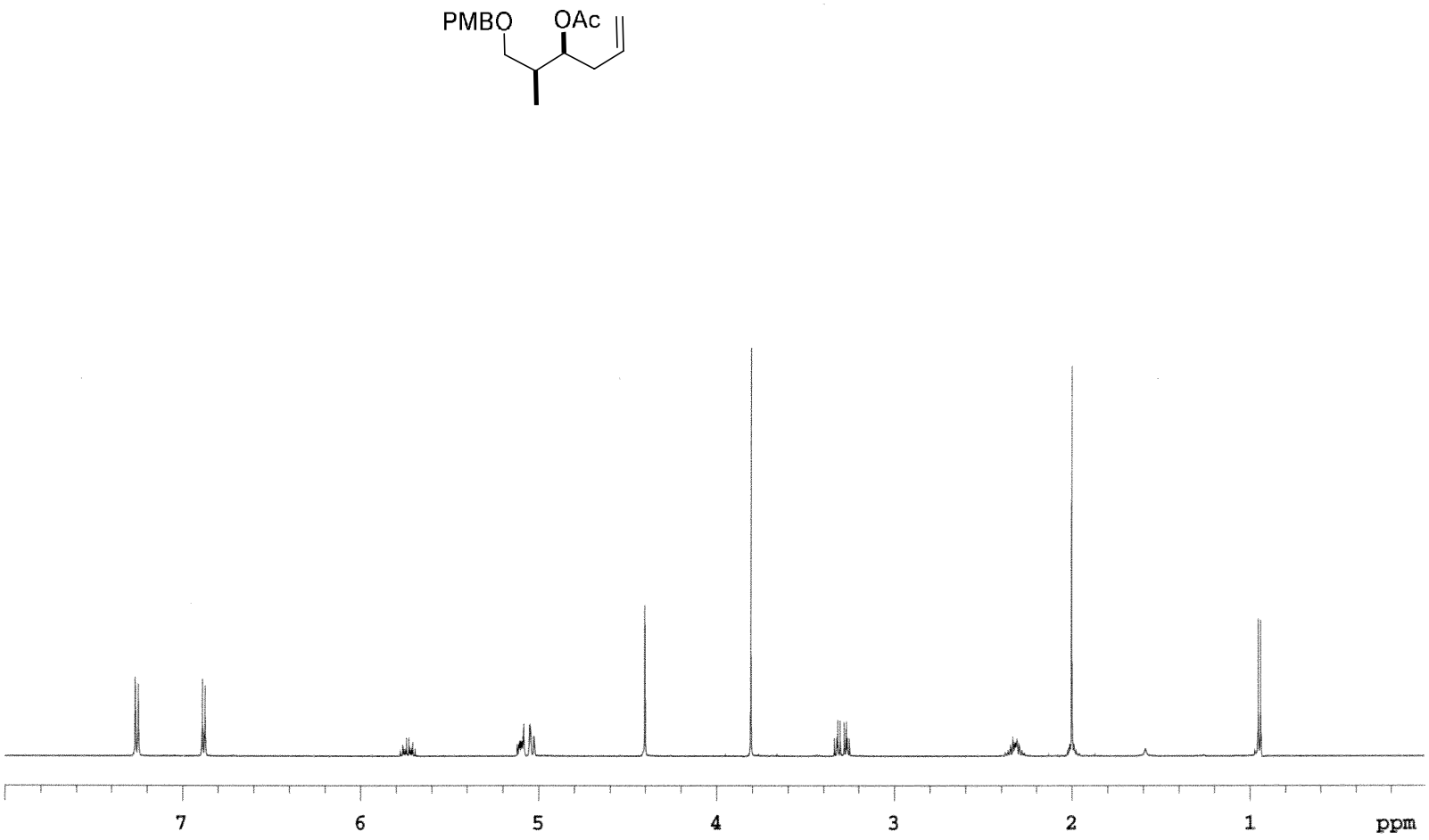




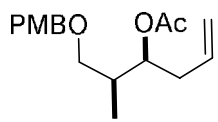

PMBO
OAc
ppm
1
2
3
4
5
6
7
8

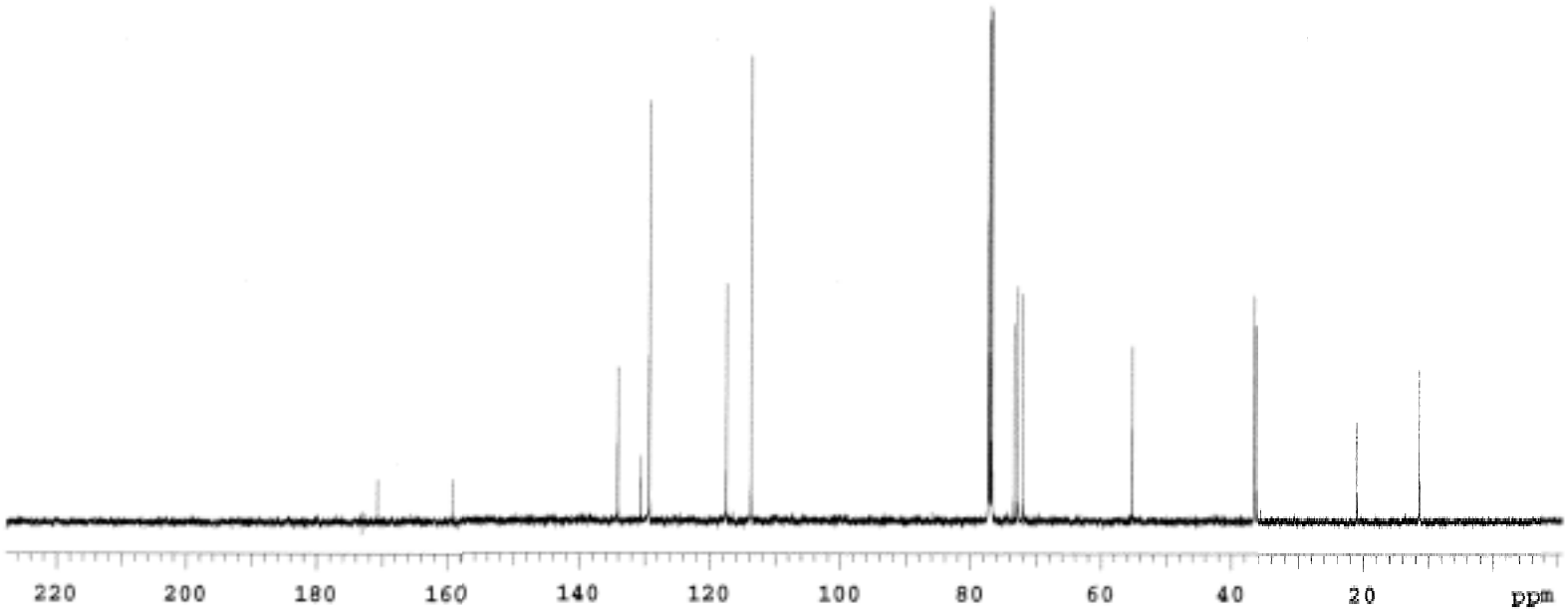
INDEX	FREQUENCY	PPM	HEIGHT
1	21446.217	170.631	10.9
2	19998.441	159.112	7.4
3	16840.678	133.988	23.1
4	16410.007	130.562	11.8
5	16242.524	129.229	65.0
6	14771.798	117.528	59.4
7	14293.276	113.720	139.1
8	9710.198	77.256	77.2
9	9677.971	77.000	79.8
10	9646.232	76.747	79.2
11	9364.489	74.506	27.5
12	9134.017	72.672	28.8
13	8999.249	71.600	27.9
14	6945.019	55.256	25.2
15	4588.049	36.503	62.7
16	4490.392	35.727	66.9
17	2647.590	21.065	30.4
18	1706.658	13.579	35.8



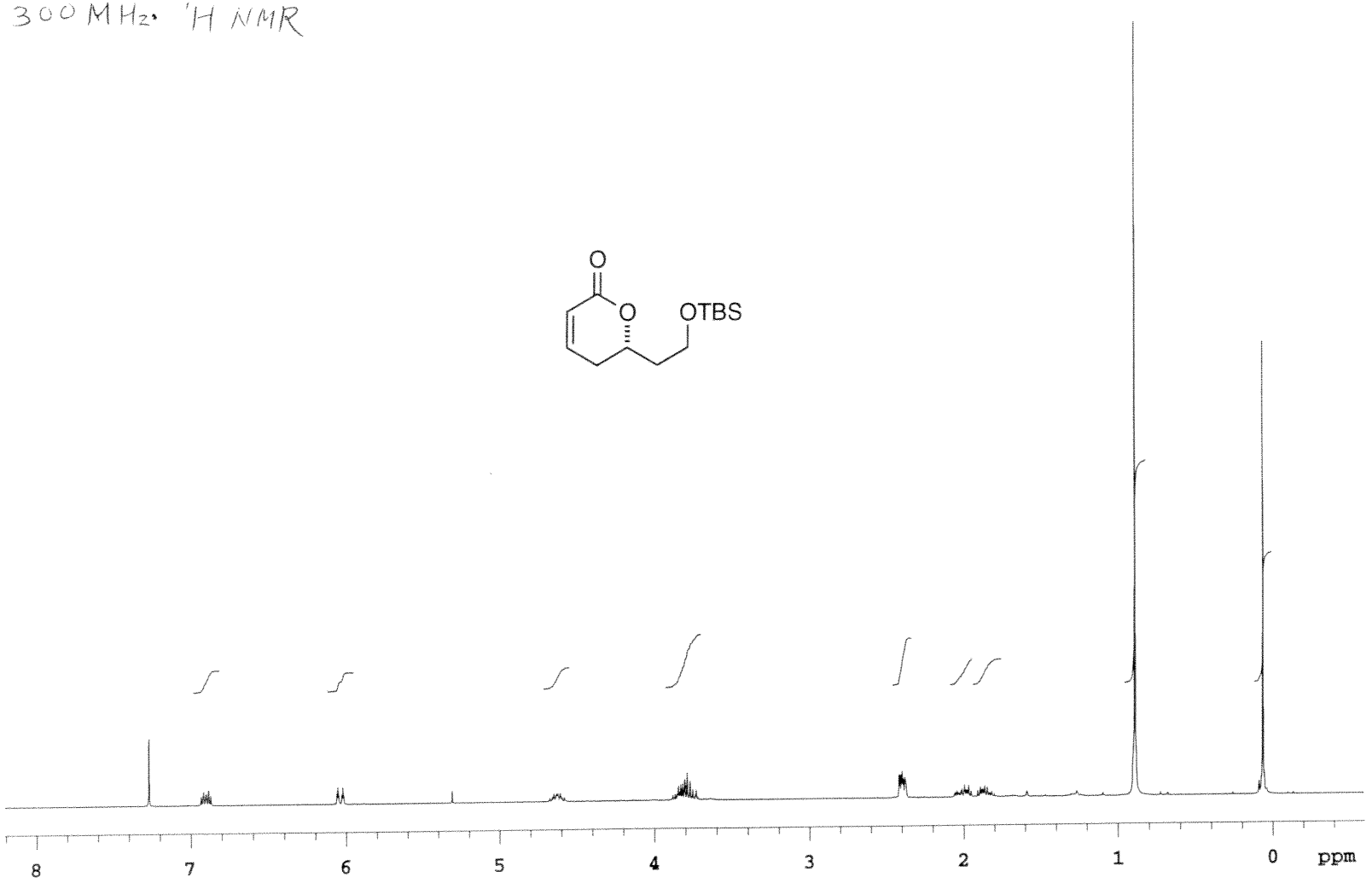

PMBO
OAc
7
6
5
4
3
2
1
ppm

INDEX	FREQUENCY	PPM	HEIGHT
1	21456.778	170.729	6.7
2	20006.198	159.186	6.8
3	16853.659	134.102	24.5
4	16406.305	130.542	10.5
5	16252.456	129.318	66.7
6	14765.703	117.488	37.7
7	14296.371	113.754	73.8
8	9709.735	77.259	79.5
9	9677.683	77.004	81.8
10	9646.089	76.752	81.1
11	9218.425	73.349	31.4
12	9146.537	72.777	37.3
13	9054.502	72.045	36.1
14	6942.278	55.238	27.9
15	4584.169	36.475	35.9
16	4565.854	36.330	31.1
17	2641.821	21.021	15.8
18	1441.704	11.471	24.1



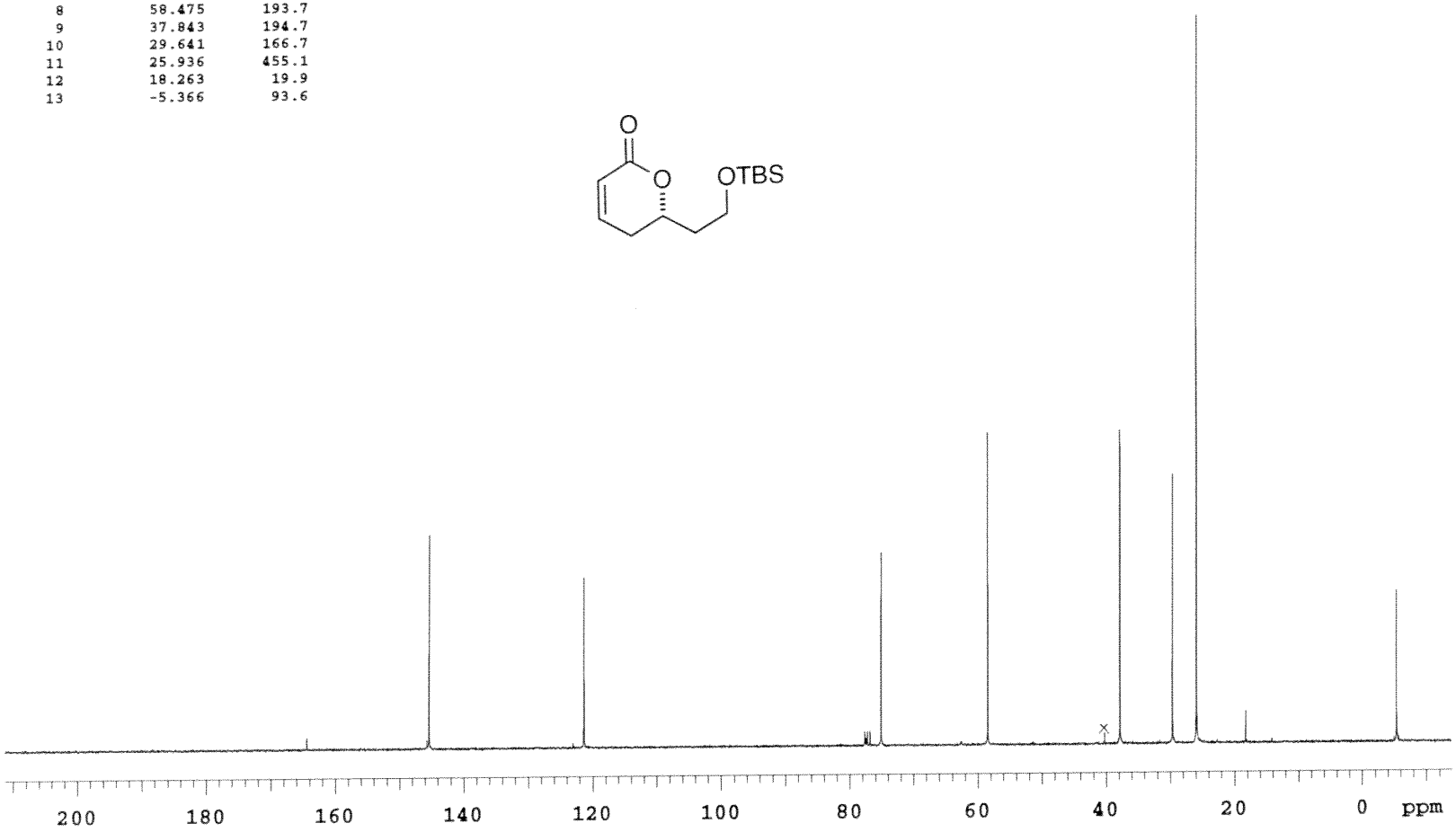


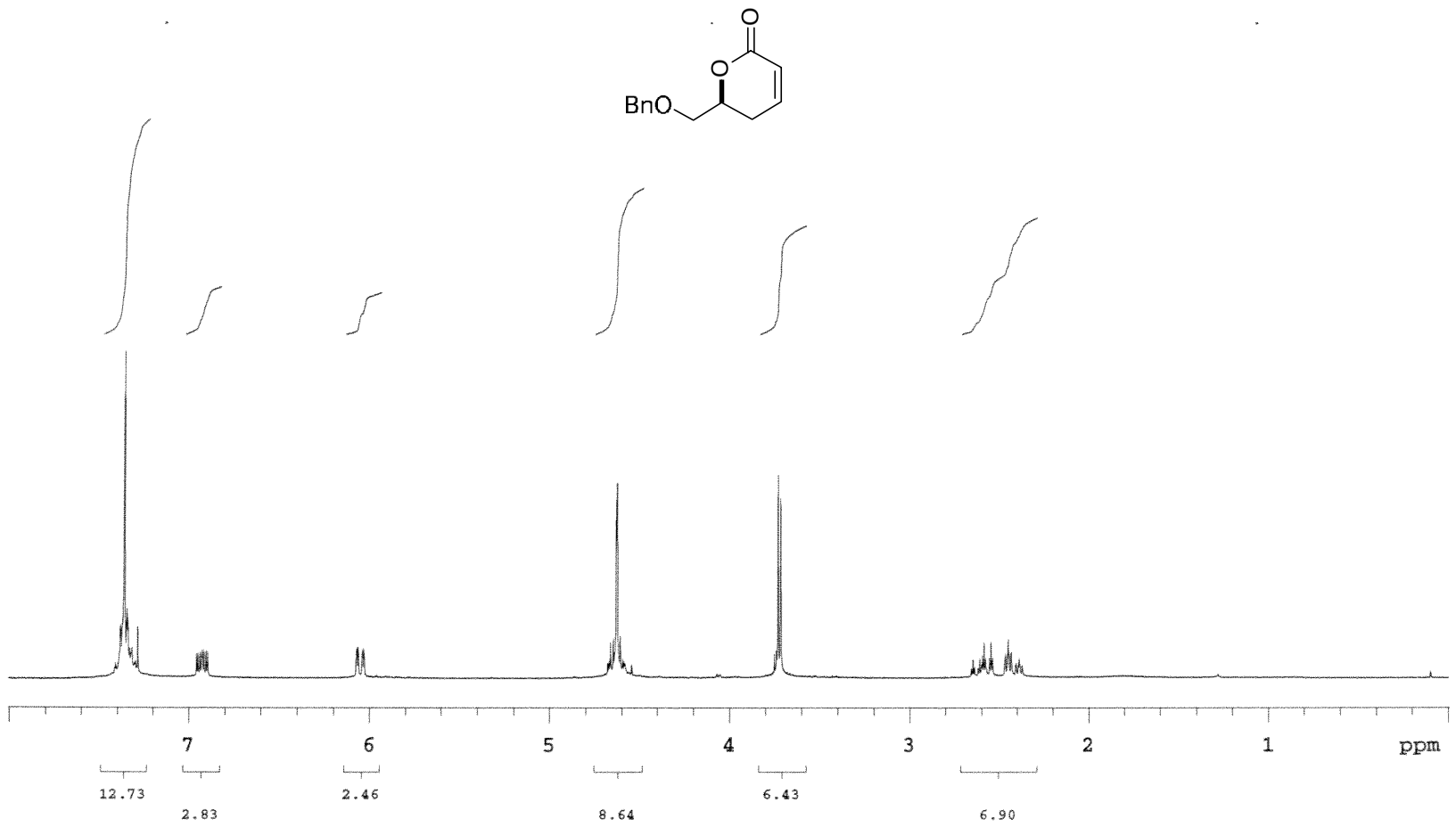
300 MHz ^{1}H NMR



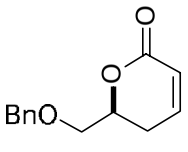
75 MHz ^{13}C NMR

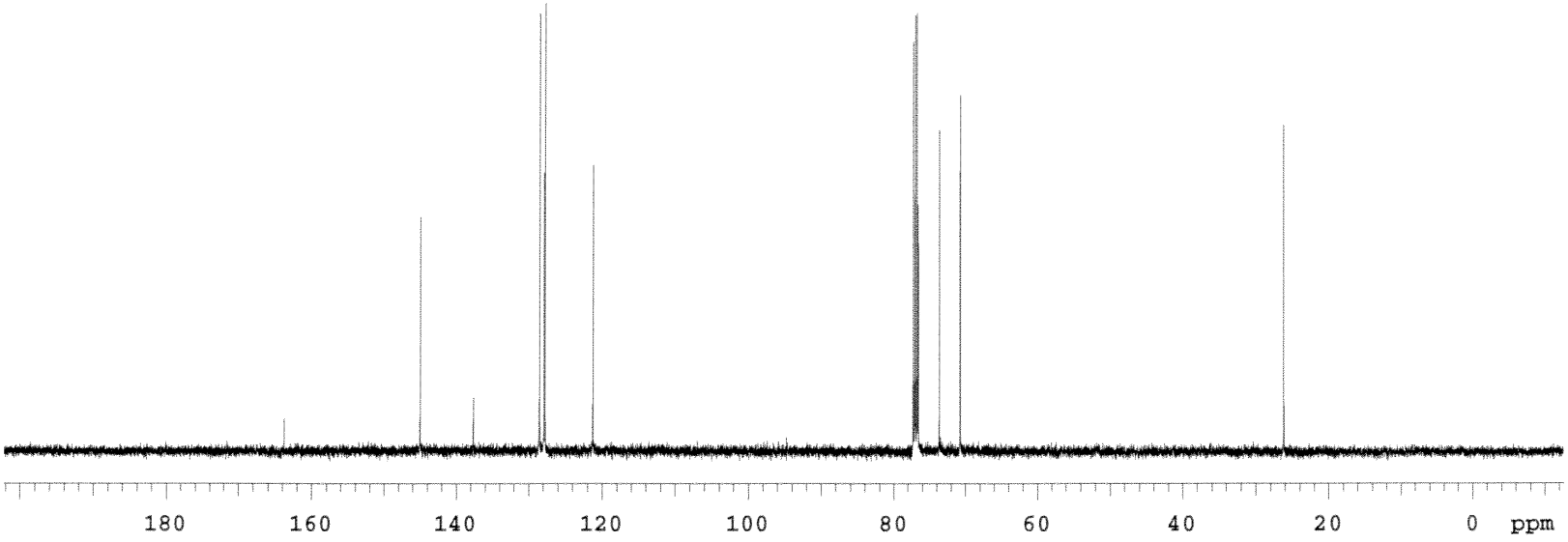
INDEX	FREQUENCY (PPM)	HEIGHT
1	164.412	7.1
2	145.382	132.1
3	121.354	105.5
4	77.670	8.6
5	77.230	8.5
6	76.817	8.8
7	75.112	119.4
8	58.475	193.7
9	37.843	194.7
10	29.641	166.7
11	25.936	455.1
12	18.263	19.9
13	-5.366	93.6

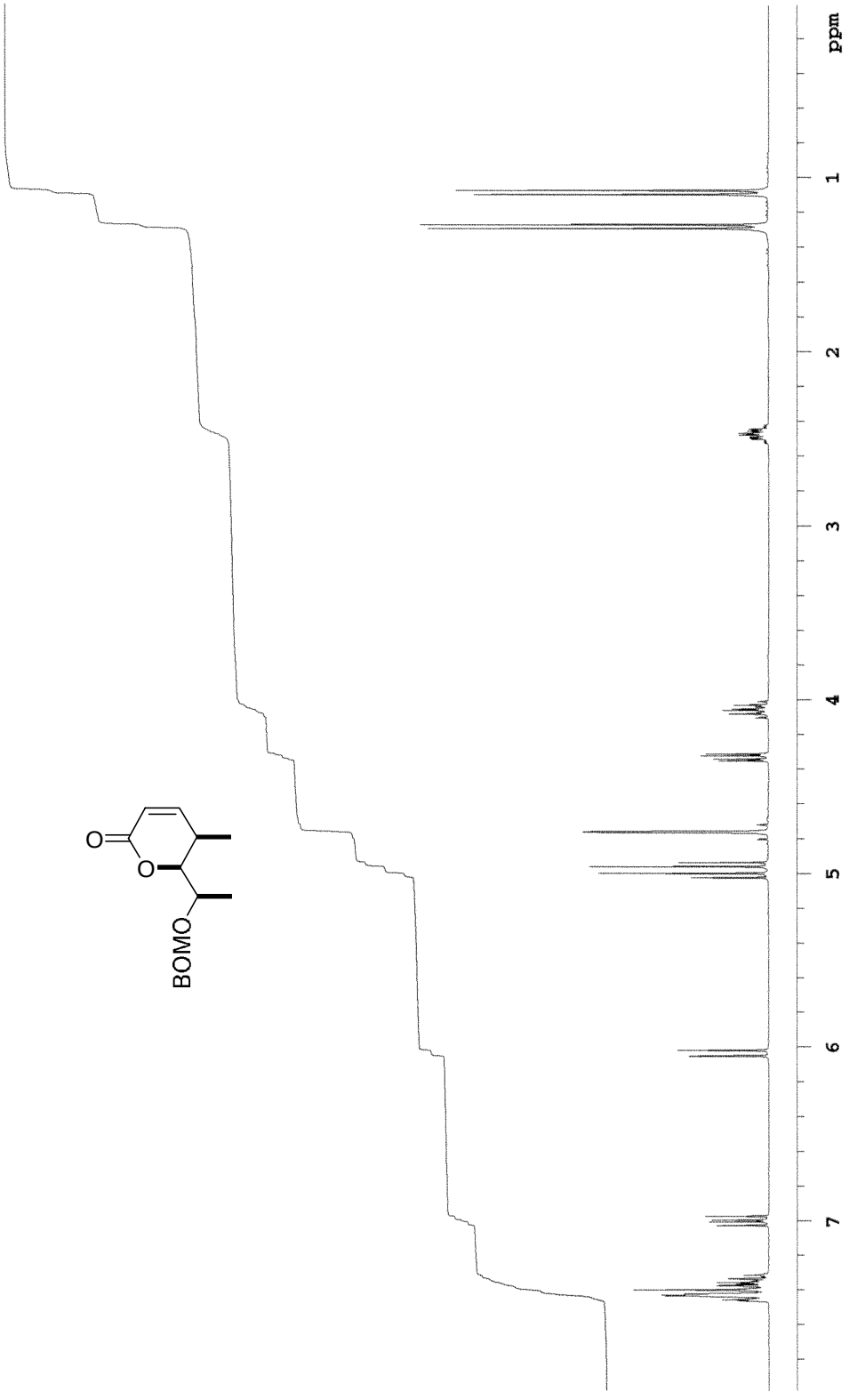


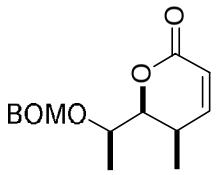

BnO
O
O
7
6
5
4
3
2
1
ppm
12.73
2.83
2.46
8.64
6.43
6.90

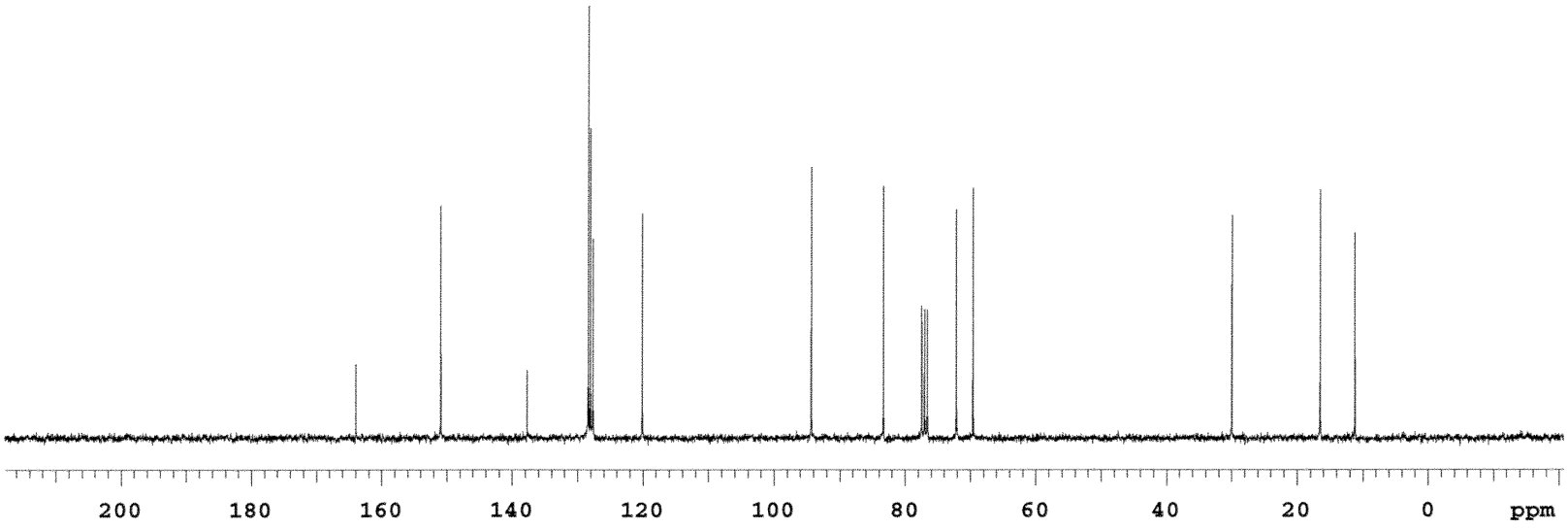
INDEX	FREQUENCY	PPM	HEIGHT
1	20583.524	163.767	0.2
2	18220.797	144.969	1.7
3	17299.280	137.637	0.4
4	16149.856	128.492	3.2
5	16074.025	127.888	2.0
6	16056.715	127.751	3.3
7	15236.170	121.222	2.1
8	9710.364	77.258	3.0
9	9678.630	77.005	3.2
10	9646.484	76.749	3.2
11	9625.053	76.579	1.8
12	9254.551	73.631	2.3
13	8891.878	70.746	2.6
14	3288.592	26.165	2.4






O
O
BOMO
ppm
1
2
3
4
5
6
7

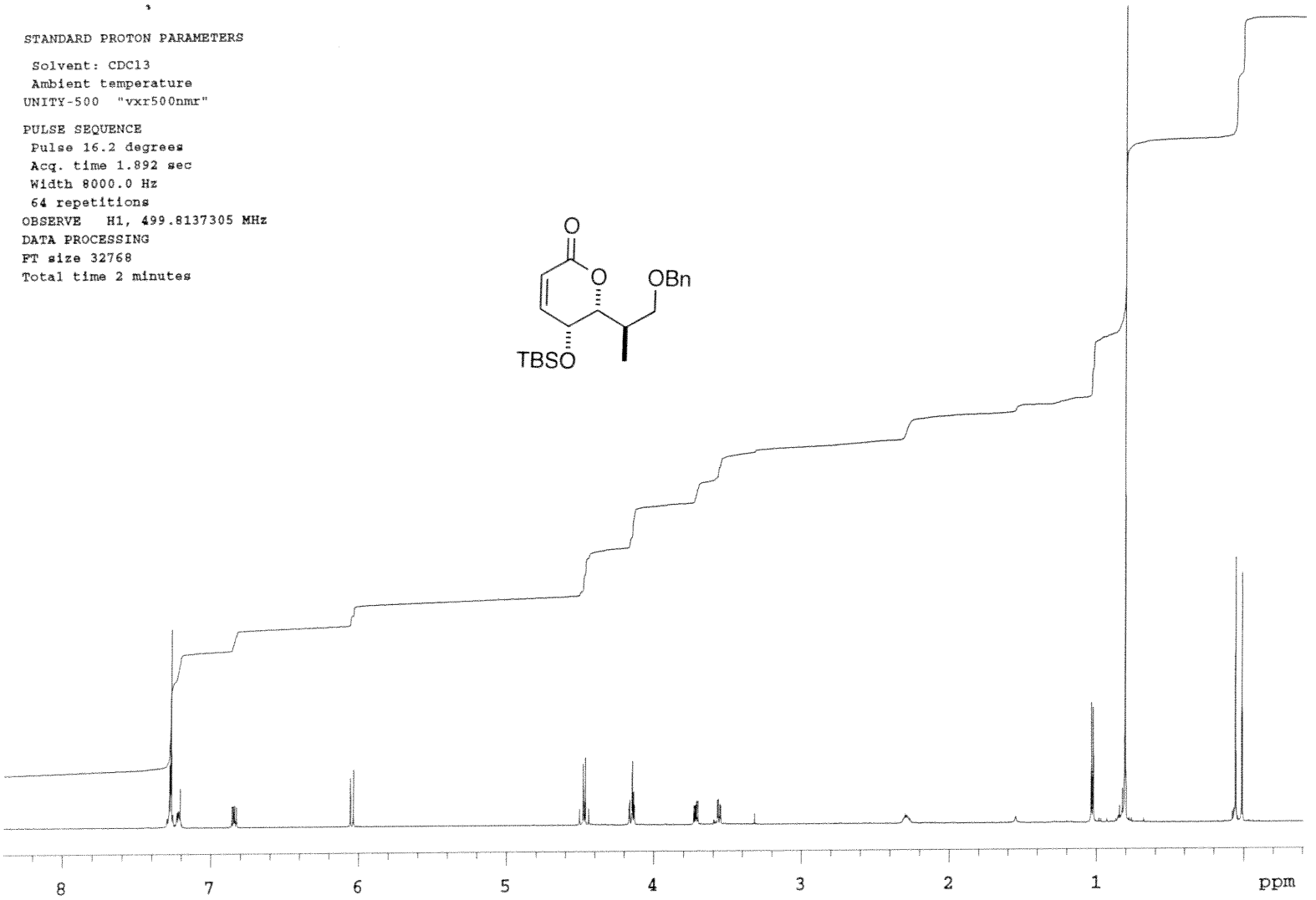

O
O
BOMO


200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD PROTON PARAMETERS

Solvent: CDCl3
Ambient temperature
UNITY-500 "vxr500nmr"

PULSE SEQUENCE
Pulse 16.2 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.8137305 MHz
DATA PROCESSING
FT size 32768
Total time 2 minutes

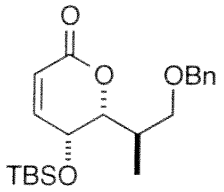


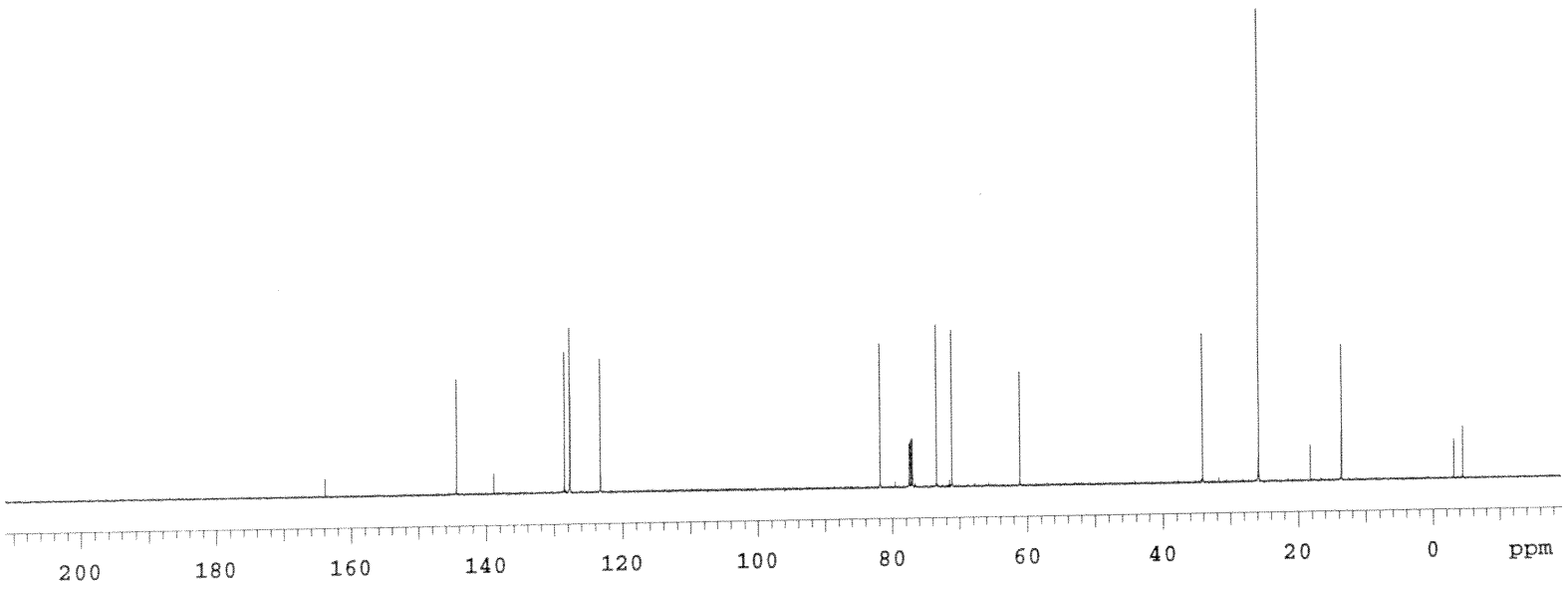
STANDARD CARBON PARAMETERS

Solvent: CDCl3
Ambient temperature
User: 1-14-87
UNITY-500 "vxr500nmr"

PULSE SEQUENCE
Pulse 99.5 degrees
Acq. time 1.300 sec
Width 28964.5 Hz
3424 repetitions
OBSERVE C13, 125.6782284 MHz
DECOUPLE H1, 499.8151988 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 74 minutes

INDEX	FREQUENCY (PPM)	HEIGHT
1	163.866	2.7
2	144.371	16.7
3	138.861	3.0
4	128.516	20.4
5	127.728	23.8
6	127.679	15.8
7	123.231	19.3
8	81.854	20.8
9	77.483	6.4
10	77.230	6.9
11	76.973	7.0
12	73.408	23.4
13	71.129	22.5
14	61.153	16.4
15	34.113	21.4
16	25.853	3.7
17	25.821	67.9
18	18.233	5.1
19	13.581	19.5
20	-3.168	5.6
21	-4.476	7.5

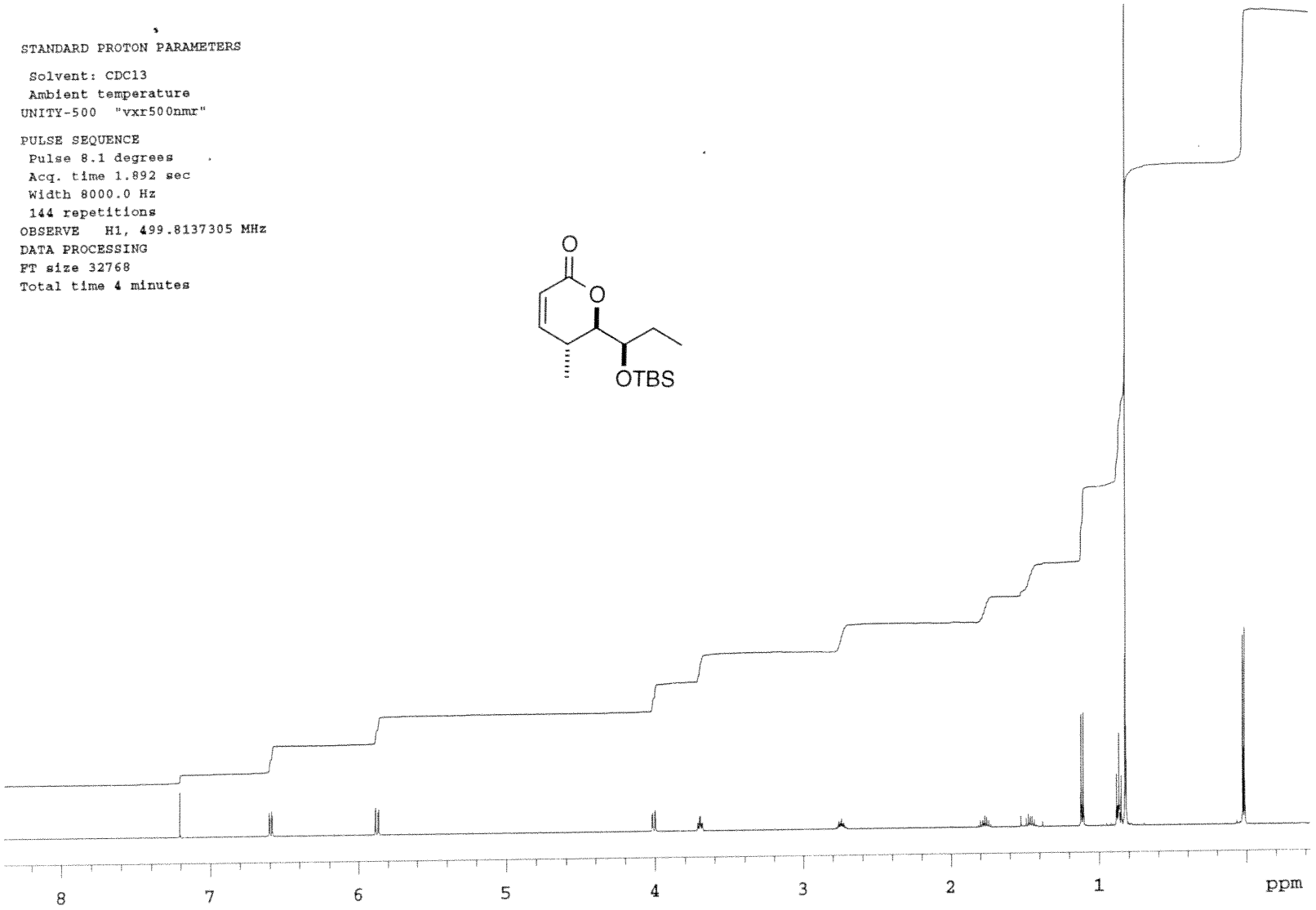




STANDARD PROTON PARAMETERS

Solvent: CDCl3
Ambient temperature
UNITY-500 "vxr500nmr"

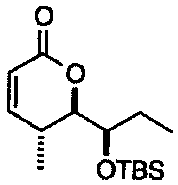
PULSE SEQUENCE
Pulse 8.1 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
144 repetitions
OBSERVE H1, 499.8137305 MHz
DATA PROCESSING
FT size 32768
Total time 4 minutes

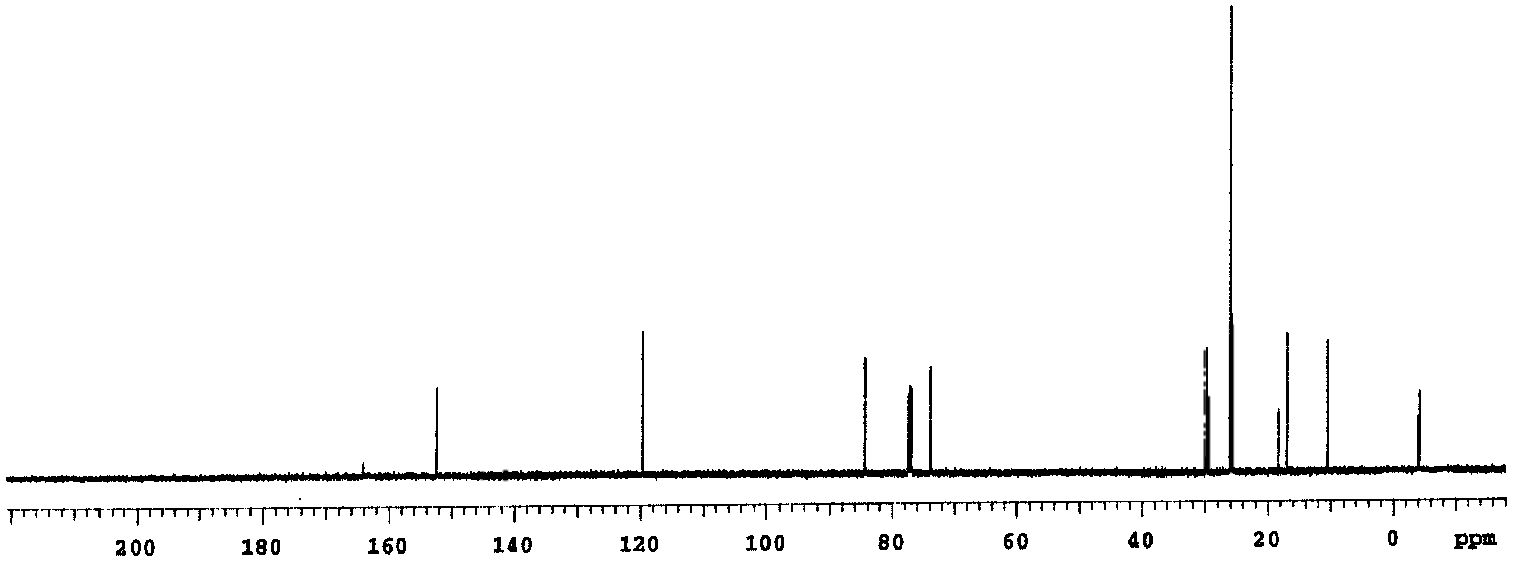


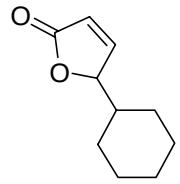
STANDARD CARBON PARAMETERS
Solvent: CDCl3
Ambient temperature
User: 1-14-07
UNITY-500 "vxr500nmr"

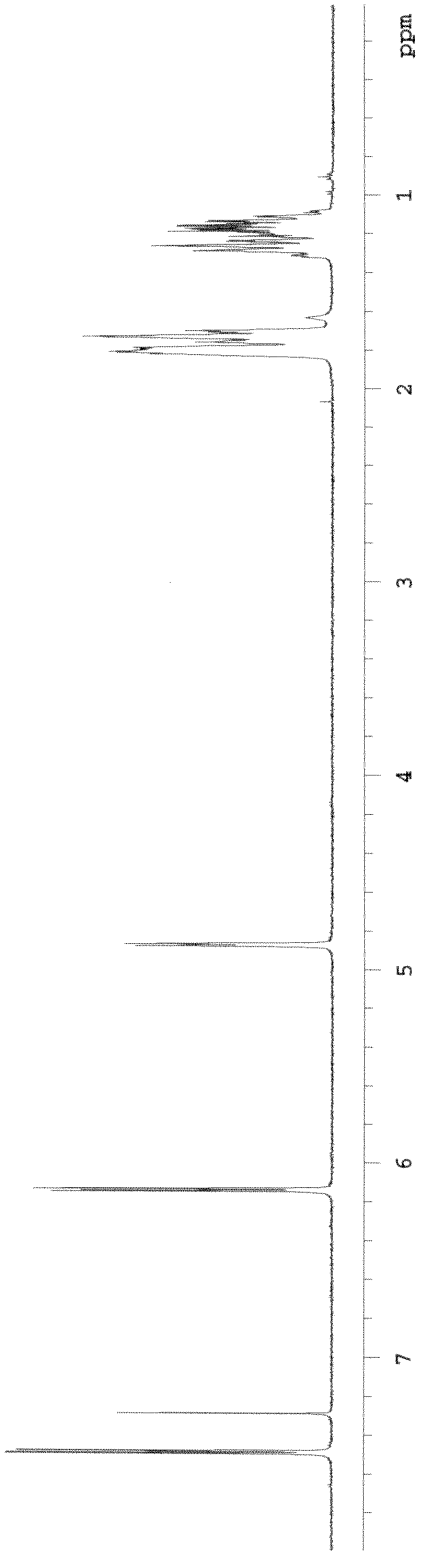
PULSE SEQUENCE
Pulse 99.5 degrees
Acq. time 1.301 sec
Width 30030.0 Hz
1216 repetitions
OBSERVE C13, 125.6782263 MHz
DECOUPLE H1, 499.8151988 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 26 minutes

INDEX	FREQUENCY (PPM)	HEIGHT
1	154.230	1.5
2	150.506	9.3
3	119.797	14.9
4	84.418	12.0
5	77.402	8.2
6	77.230	9.0
7	76.975	8.6
8	73.830	11.1
9	69.684	12.7
10	38.037	47.9
11	28.786	16.3
12	18.289	6.3
13	18.067	14.3
14	14.836	13.6
15	-4.007	5.6
16	-4.214	8.2

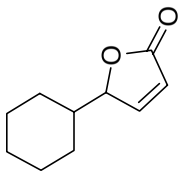


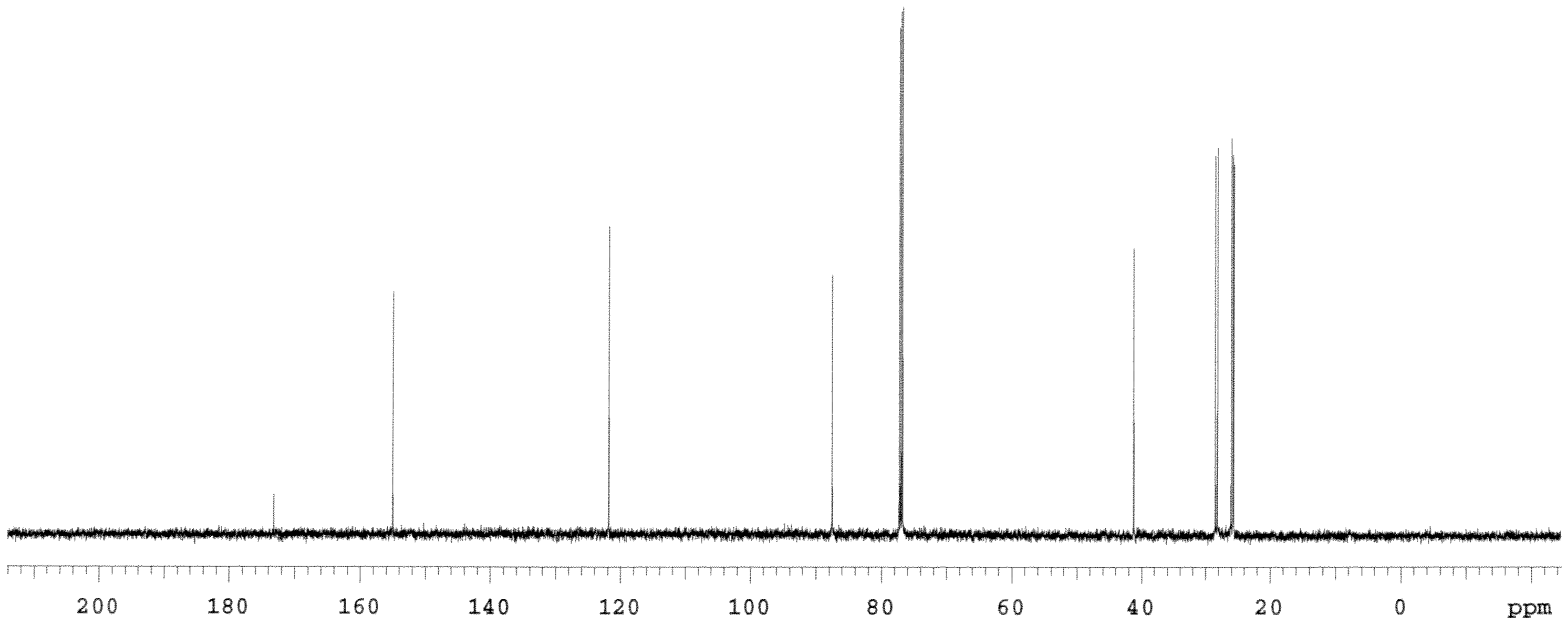



O
O


ppm
1
2
3
4
5
6
7

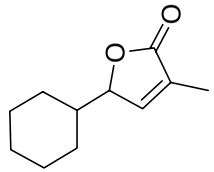
INDEX	FREQUENCY (PPM)	HEIGHT
1	173.156	0.2
2	155.032	1.0
3	121.827	1.2
4	87.506	1.0
5	77.248	2.0
6	76.996	2.1
7	76.741	2.1
8	41.193	1.1
9	28.490	1.5
10	28.141	1.5
11	26.024	1.6
12	25.733	1.5
13	25.634	1.5

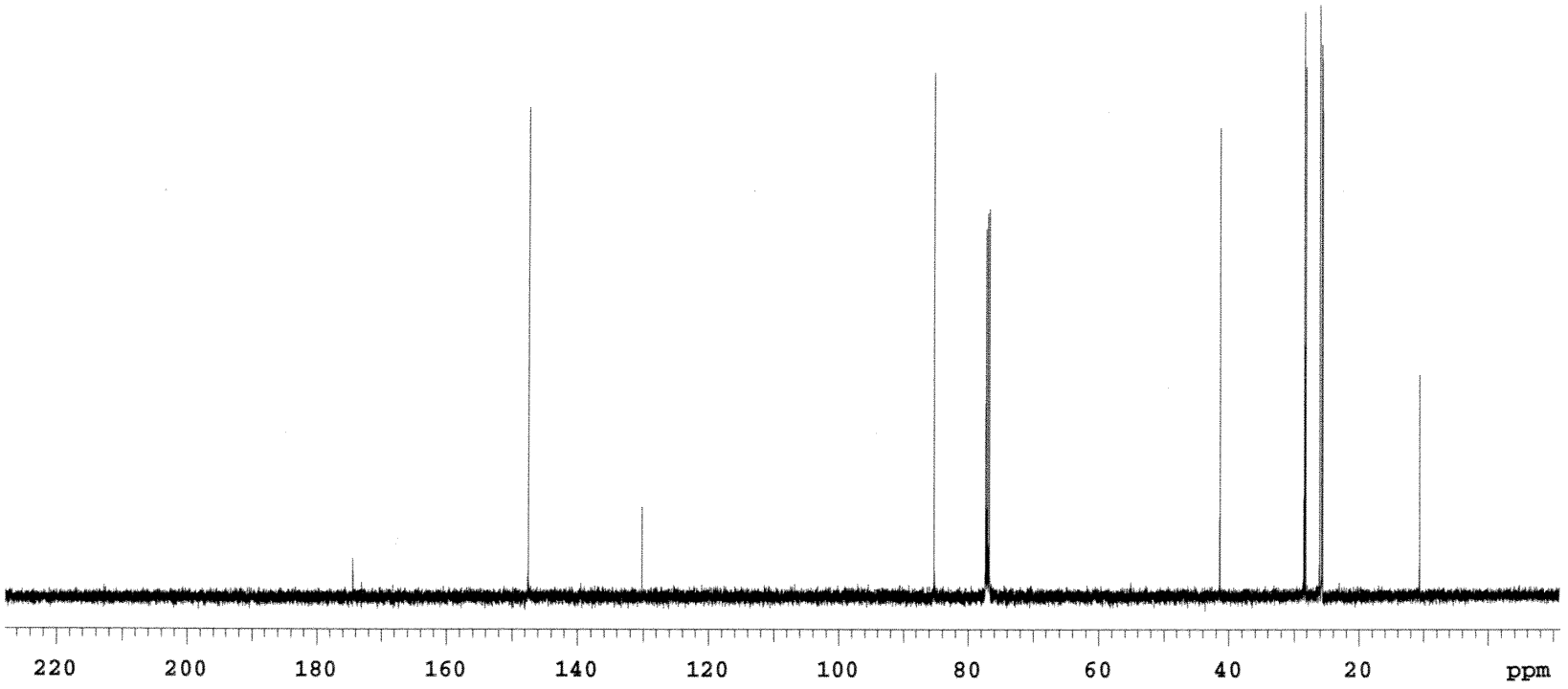





ppm
1
2
3
4
5
6
7
O
O

INDEX	FREQUENCY	PPM	HEIGHT
1	21921.990	174.429	6.1
2	18539.592	147.516	77.6
3	16361.890	130.189	14.3
4	10710.672	85.223	83.1
5	9709.277	77.255	58.4
6	9677.225	77.000	60.9
7	9645.174	76.745	61.6
8	5200.940	41.383	74.4
9	3572.243	28.424	92.8
10	3543.396	28.194	84.1
11	3273.702	26.048	93.9
12	3231.119	25.709	87.7
13	3218.756	25.611	85.1
14	1332.269	10.601	35.4

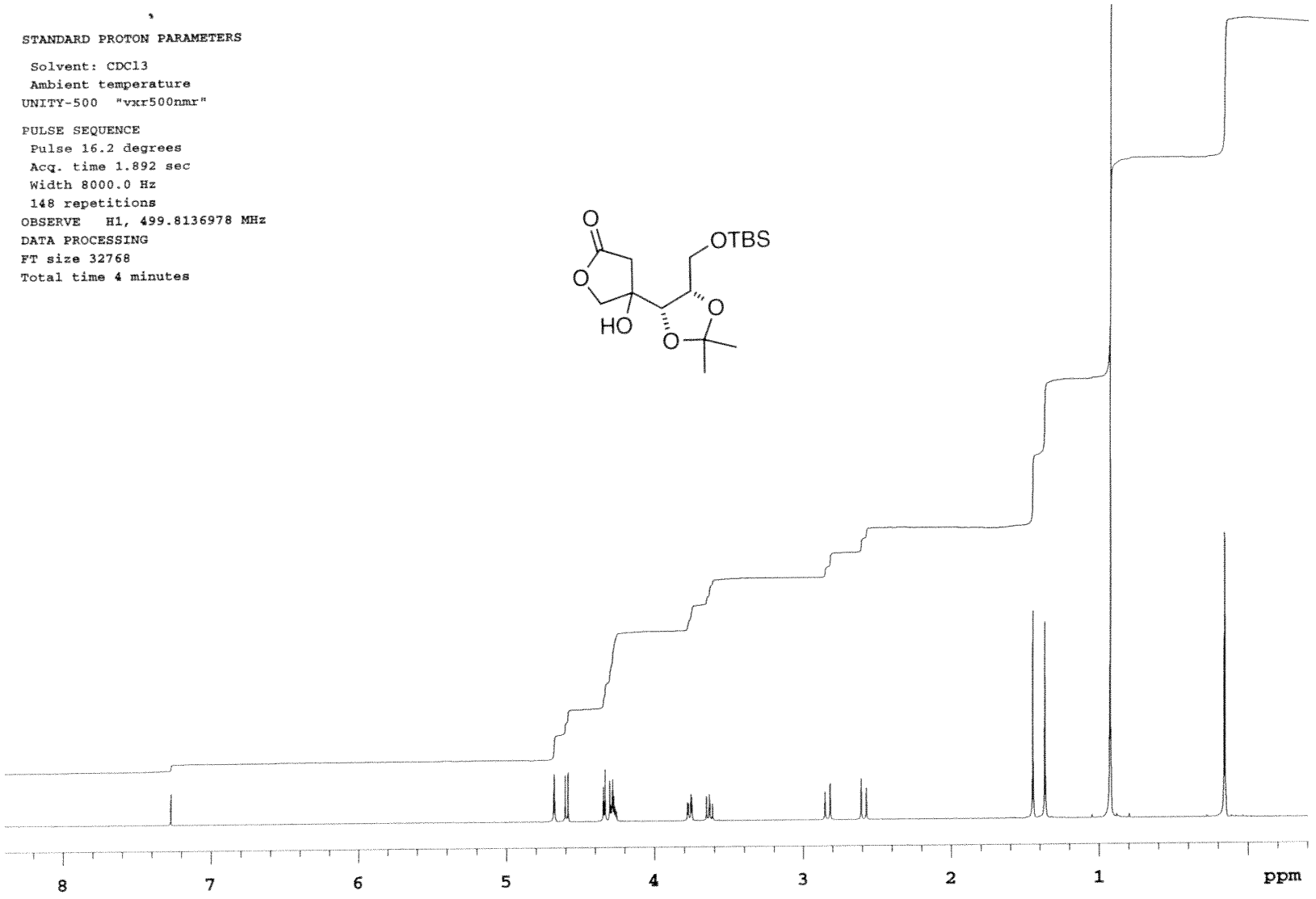




STANDARD PROTON PARAMETERS

Solvent: CDCl3
Ambient temperature
UNITY-500 "vxr500nmr"

PULSE SEQUENCE
Pulse 16.2 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
148 repetitions
OBSERVE H1, 499.8136978 MHz
DATA PROCESSING
FT size 32768
Total time 4 minutes

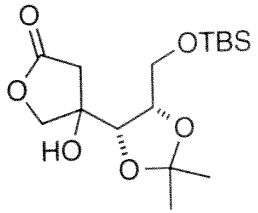


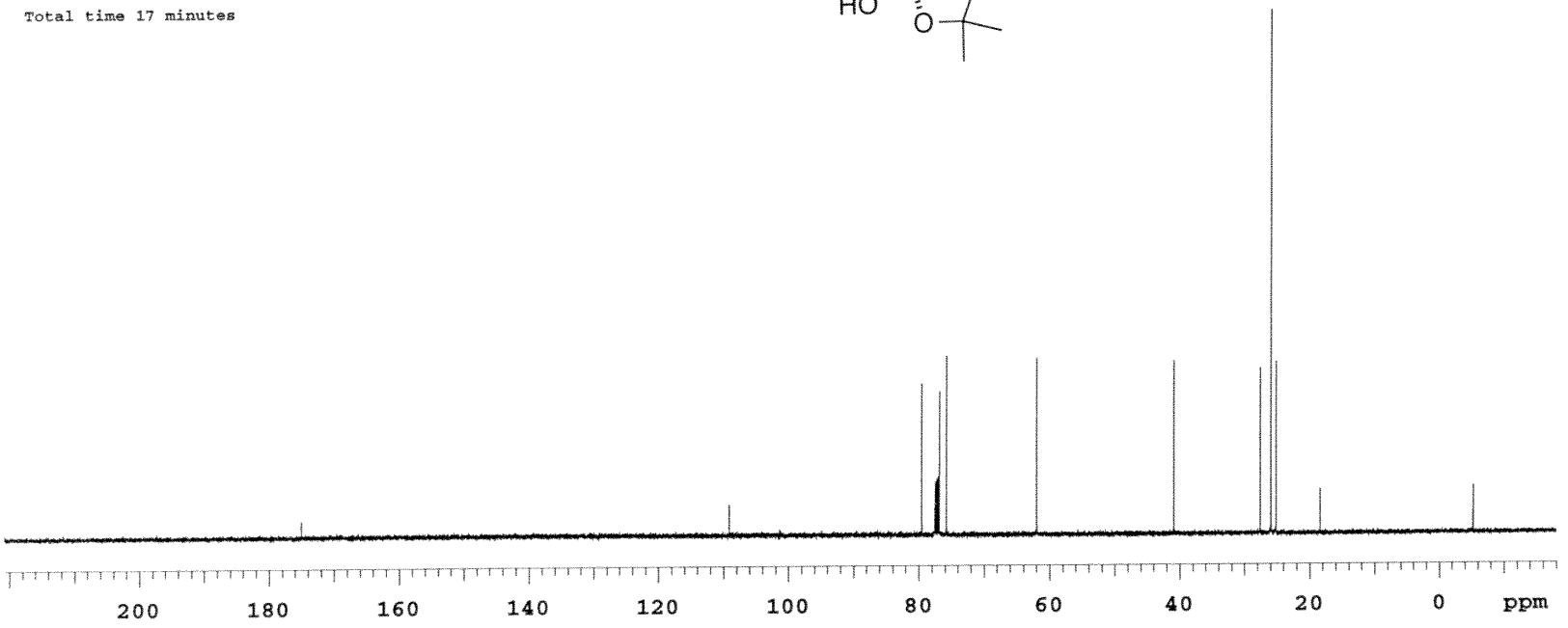
STANDARD CARBON PARAMETERS

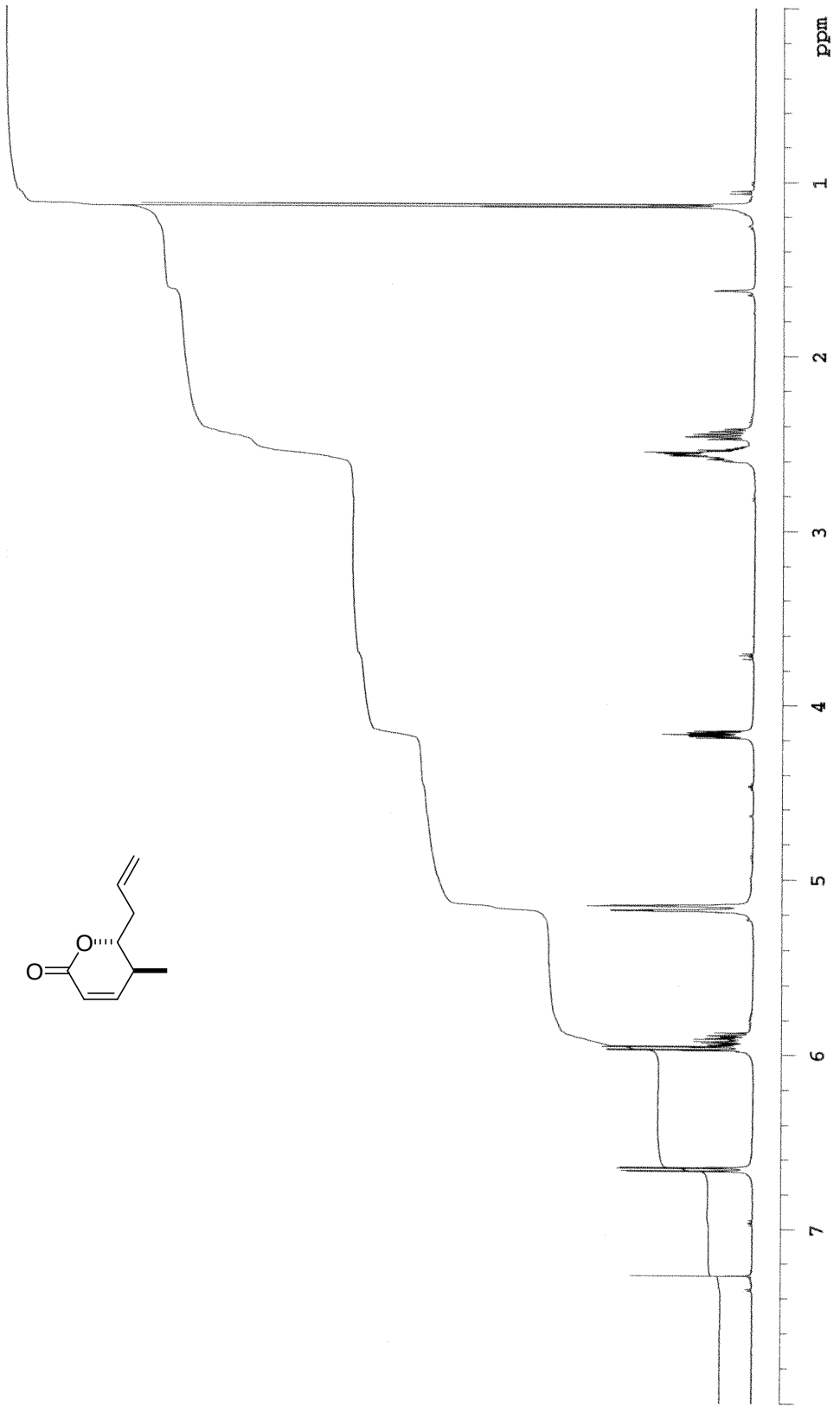
Solvent: CDCl3
Ambient temperature
User: 1-14-87
UNITY-500 "vxr500nmr"

PULSE SEQUENCE
Pulse 99.5 degrees
Acq. time 1.301 sec
Width 30030.0 Hz
800 repetitions
OBSERVE C13, 125.6782258 MHz
DECOUPLE H1, 499.8151988 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 17 minutes

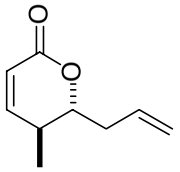
INDEX	FREQUENCY (PPM)	HEIGHT
1	175.120	0.4
2	109.163	0.7
3	79.538	3.2
4	77.489	1.1
5	77.234	1.2
6	77.040	1.1
7	76.982	1.2
8	76.840	3.0
9	75.750	3.8
10	61.962	3.7
11	40.926	3.7
12	27.558	3.5
13	25.935	11.2
14	25.130	3.6
15	18.403	0.9
16	-5.305	0.9
17	-5.330	1.0

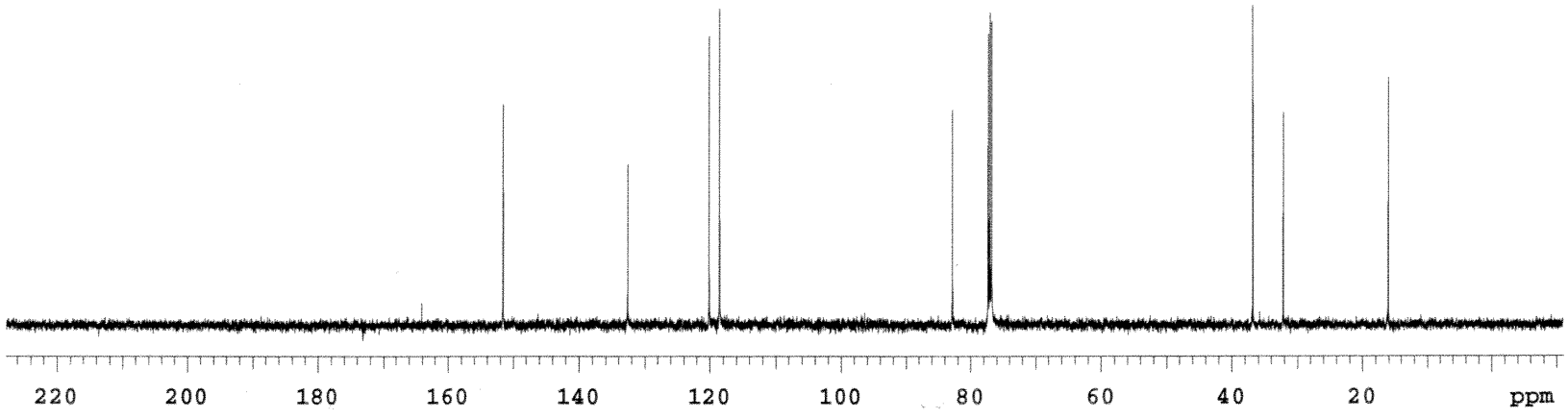


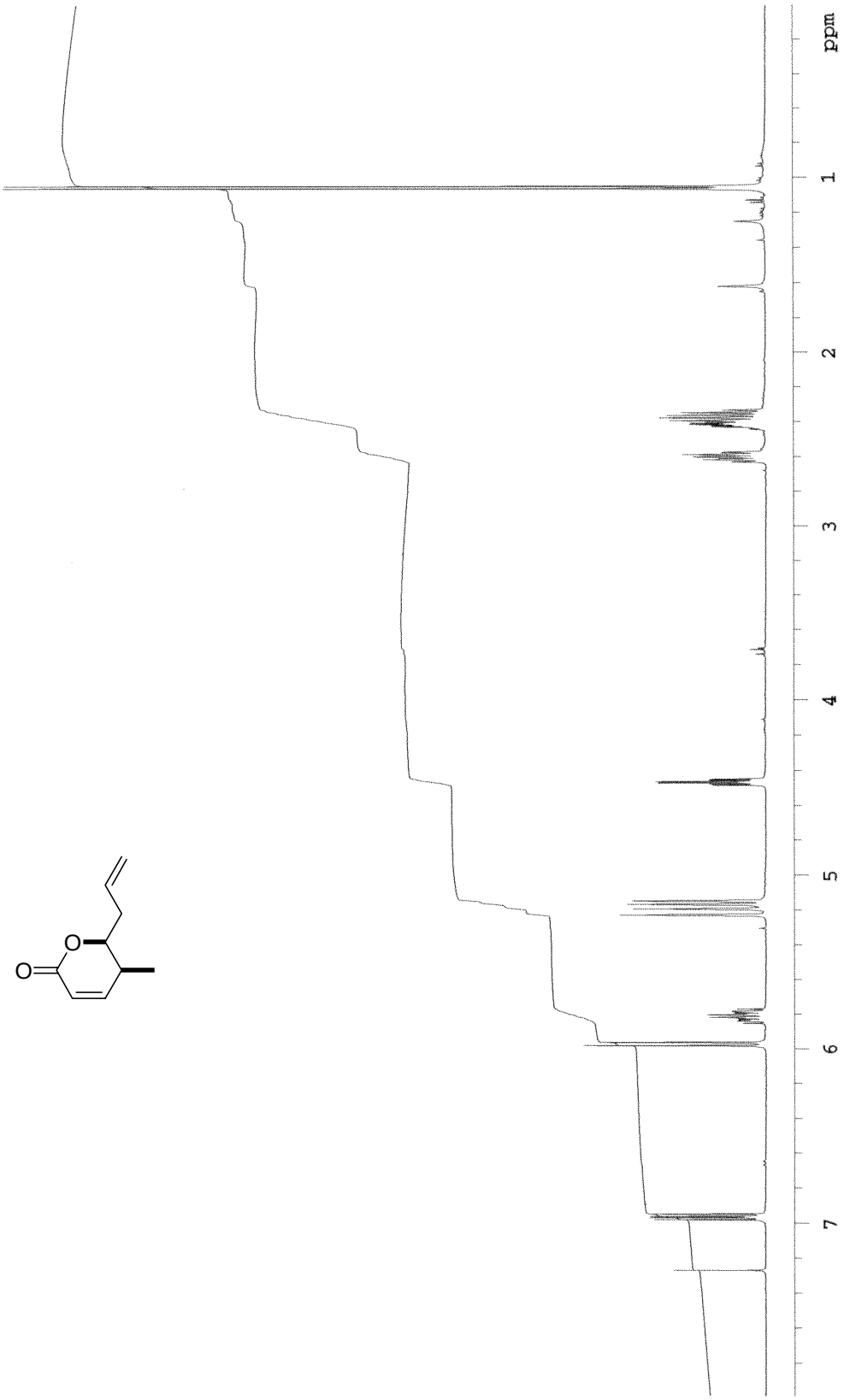



O
O
ppm
1
2
3
4
5
6
7

INDEX	FREQUENCY	PPM	HEIGHT
1	21758.525	173.129	-2.5
2	20627.090	164.126	3.4
3	19052.881	151.600	34.8
4	16667.758	132.622	25.2
5	15095.838	120.115	45.6
6	14901.695	118.570	49.9
7	10405.263	82.793	33.8
8	9709.277	77.255	45.9
9	9677.225	77.000	49.3
10	9645.174	76.745	48.0
11	4623.089	36.785	50.4
12	4041.575	32.158	33.6
13	2015.891	16.040	39.0






O
O
ppm
1
2
3
4
5
6
7

INDEX	FREQUENCY	PPM	HEIGHT
1	20680.540	164.539	0.5
2	19055.521	151.610	4.4
3	16648.696	132.461	2.8
4	15081.787	119.994	5.4
5	14905.396	118.591	6.2
6	9966.460	79.295	4.8
7	9710.117	77.256	4.9
8	9677.971	77.000	5.1
9	9646.237	76.748	5.2
10	4502.473	35.823	6.7
11	3951.871	31.442	5.8
12	1384.729	11.017	6.6