

Supporting Information

Synthesis of Oligomeric 4-(Glycosyloxy)benzoate Macrocyclic Glycosides

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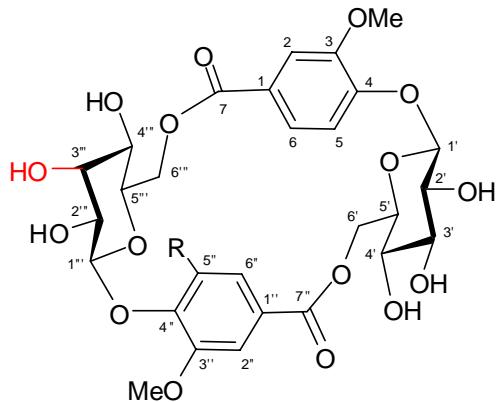
General Remarks for the Synthesis

All solvents were distilled prior to use except where noted. All reactions sensitive to moisture or oxygen were conducted under an atmosphere of nitrogen or argon using flame-dried or oven-dried (170 °C) glassware. Crushed 4Å molecular sieves were activated by thorough flame-drying immediately prior to use.

Flash column chromatography was performed on silica gel H (10-40 µ). Analytical thin layer chromatography (TLC) was performed on glass plates pre-coated with a 0.25 mm thickness of silica gel. The TLC plates were visualized with UV light and/or by staining with acidic methanol or ethanolic phosphomolybdic acid (PMA).

NMR: Chemical shifts are given in ppm relative to TMS.

Comparison of the ^1H and ^{13}C NMR Data of the Synthetic Clemoarmanoside A (7) and Clemahexapetoside A (8) with Those Reported for the Natural products.



Clemoarmanoside A: R = H (7)
Clemahexapetoside A: R = OMe (8)

^1H NMR	Synthetic Compound 7	Authentic Compound ^[5]	Authentic Compound ^[6]
H-2	7.41 (d, J = 3.0 Hz, 1 H)	7.41 (d, J = 2.4 Hz, 1 H)	7.41 (d, J = 1.8 Hz, 1 H)
H-5	7.33 (d, J = 9.5 Hz, 1 H)	7.33 (d, J = 9.0 Hz, 1 H)	7.38 (d, J = 8.7 Hz, 1 H)
H-6	7.77 (dd, J = 1.5, 6.0 Hz, 1 H)	7.75 (dd, J = 2.4, 9.0 Hz, 1 H)	7.79 (dd, J = 1.8, 8.7 Hz, 1 H)
3-OMe	3.79 (s, 3 H)	3.79 (s, 3 H)	3.78 (s, 3 H)
H-1'	5.21 (d, J = 7.0 Hz, 1 H)	5.24 (d, J = 7.2 Hz, 1 H)	5.35 (d, J = 7.5 Hz, 1 H)
H-2'	3.45-3.39 (m, 1 H)	3.39 (m, 1 H)	3.47-3.33 (m, 1 H)
H-3'	3.45-3.39 (m, 1 H)	3.39 (m, 1 H)	3.47-3.33 (m, 1 H)
H-4'	3.18-3.16 (m, 1 H)	3.20 (m, 1 H)	3.21-3.15 (m, 1 H)

H-5'	3.99-3.97 (m, 1 H)	3.97 (m, 1 H)	3.47-3.33 (m, 1 H)
H-6'	4.13-4.07 (m, 1 H) 4.42 (d, <i>J</i> = 11.0 Hz, 1 H)	4.09 (m, 1 H) 4.41 (br d, <i>J</i> = 11.4, 1 H)	3.96-3.92 (m, 1 H) 4.13 (dd, <i>J</i> = 5.0, 11.5 Hz, 1 H)
H-2''	7.42 (d, <i>J</i> = 2.5 Hz, 1 H)	7.42 (d, <i>J</i> = 2.4 Hz, 1 H)	7.40 (d, <i>J</i> = 1.8 Hz, 1 H)
H-5''	7.38 (d, <i>J</i> = 9.0 Hz, 1 H)	7.38 (d, <i>J</i> = 9.0 Hz, 1 H)	7.33 (d, <i>J</i> = 8.7 Hz, 1 H)
H-6''	7.79 (dd, <i>J</i> = 2.0, 7.0 Hz, 1 H)	7.78 (dd, <i>J</i> = 2.4, 9.0 Hz, 1 H)	7.77 (dd, <i>J</i> = 2.0, 8.7 Hz, 1 H)
3''-OMe	3.79 (s, 3 H)	3.79 (s, 3 H)	3.78 (s, 3 H)
H-1'''	5.39 (d, <i>J</i> = 9.0 Hz, 1 H)	5.38 (d, <i>J</i> = 7.8 Hz, 1 H)	5.20 (d, <i>J</i> = 6.5 Hz, 1 H)
H-2'''	3.58 (m, 1 H)	3.58 (td, <i>J</i> = 2.4, 7.8 Hz, 1 H)	3.47-3.33 (m, 1 H)
H-3'''	3.99-3.97 (m, 1 H)	3.98 (m, 1 H)	3.57 (t, <i>J</i> = 2.5 Hz, 1 H)
H-4'''	3.45-3.39 (m, 1 H)	3.45 (m, 1 H)	3.47-3.33 (m, 1 H)
H-5'''	4.28-4.24 (m, 1 H)	4.26 (m, 1 H)	3.96-3.92 (m, 1 H)
H-6'''	4.13-4.07 (m, 1 H) 4.38 (d, <i>J</i> = 11.5 Hz, 1 H)	4.11 (m, 1 H) 4.37 (br d, <i>J</i> = 11.4 Hz, 1 H)	4.21 (br. d, <i>J</i> = 11.5 Hz, 1 H) 4.41 (dd, <i>J</i> = 5.5, 11.5 Hz, 1 H)

¹³ C NMR	Synthetic Compound 7	Authentic Compound ^[6]	Authentic Compound ^[5]
1	123.1	123.1	122.5
2	112.1	112.1	112.1
3	148.5	148.5	148.4
4	150.1	150.1	149.8
5	114.4	114.4	114.4
6	122.5	122.5	123.1
7	165.2	165.3	165.2
3-OMe	55.5	55.5	55.5
1'	98.3	98.2	98.3
2'	72.8	72.8	72.8
3'	76.9	76.9	76.9
4'	70.6	70.6	70.6
5'	73.5	73.5	73.5
6'	65.4	65.5	65.1
1''	123.0	123.1	122.4
2''	112.1	112.1	112.1

3''	148.4	148.4	148.4
4''	149.8	149.8	150.1
5''	114.3	114.3	114.3
6''	122.4	122.4	122.9
7''	165.1	165.2	165.1
3''-OMe	55.5	55.5	55.5
1'''	96.8	96.7	96.7
2'''	71.7	71.7	69.8
3'''	69.8	69.8	71.7
4'''	68.2	68.2	68.2
5'''	71.2	71.5	71.2
6'''	65.1	65.1	65.4

¹ H NMR	Synthetic Compound 8	Authentic Compound ^[6]
H-2	7.36 (d, <i>J</i> = 1.0 Hz, 1 H)	7.34 (d, <i>J</i> = 2.0 Hz, 1 H)
H-5	6.99 (d, <i>J</i> = 8.0 Hz, 1 H)	6.97 (d, <i>J</i> = 8.5 Hz, 1 H)
H-6	6.82 (dd, <i>J</i> = 1.5, 9.0 Hz, 1 H)	6.80 (dd, <i>J</i> = 2.0, 8.5 Hz, 1 H)
3-OMe	3.79 (s, 3 H)	3.77 (s, 3 H)
H-1'	5.61 (d, <i>J</i> = 8.0 Hz, 1H)	5.31 (d, <i>J</i> = 7.5 Hz, 1 H)
H-2'	3.40-3.37 (m, 1 H)	3.49-3.44 (m, 1 H)
H-3'	3.40-3.37 (m, 1 H)	3.40-3.20 (m, 1 H)
H-4'	3.22-3.19 (m, 1 H)	3.14-3.10 (m, 1 H)
H-5'	3.94-3.83 (m, 1 H)	3.40-3.20 (m, 1 H)
H-6'	3.94-3.83 (m, 1 H)	4.39 (dd, <i>J</i> = 5.0, 11.5 Hz, 1 H)
	4.51 (d, <i>J</i> = 10.5 Hz, 1 H)	3.90 (br d, <i>J</i> = 11.5 Hz, 1 H)
H-2''	7.08 (d, <i>J</i> = 1.0 Hz, 1 H)	7.07 (d, <i>J</i> = 2.0 Hz, 1 H)
H-6''	7.50 (d, <i>J</i> = 1.5 Hz, 1 H)	7.49 (d, <i>J</i> = 2.0 Hz, 1 H)
3''-OMe	3.96 (s, 3 H)	3.96 (s, 3 H)
5''-OMe	3.59 (s, 3 H)	3.60 (s, 3 H)
H-1'''	5.05 (d, <i>J</i> = 7.0 Hz, 1 H)	5.13 (d, <i>J</i> = 7.5 Hz, 1 H)
H-2'''	3.49-3.46 (m, 1 H)	3.20-3.40 (m, 1 H)
H-3'''	3.94-3.83 (m, 1 H)	3.55 (t, <i>J</i> = 2.5 Hz, 1 H)
H-4'''	3.40-3.37 (m, 1 H)	3.49-3.44 (m, 1 H)
H-5'''	4.44-4.39 (m, 1 H)	3.98-3.92 (m, 1 H)
H-6'''	3.94-3.83 (m, 1 H)	4.45 (dd, <i>J</i> = 5.5, 11.5 Hz, 1 H)
	4.36 (d, <i>J</i> = 10.0 Hz, 1 H)	4.26 (br d, <i>J</i> = 11.5 Hz, 1 H)

¹³ C NMR	Synthetic Compound 8	Authentic Compound ^[6]
1	122.6	122.4
2	111.9	111.9
3	148.4	148.4
4	149.9	150.1
5	114.4	114.3
6	122.3	122.4
7	165.2	165.1
3'-OMe	55.5	55.5
1'	99.2	100.9
2'	73.0	73.8
3'	77.2	76.7
4'	71.0	71.2
5'	73.9	73.9
6'	65.4	65.0
1''	124.5	124.6
2''	107.0	107.0
3''	152.0	152.0
4''	138.1	137.6
5''	153.2	153.1
6''	106.9	106.7
7''	164.8	164.9
3''-OMe	56.6	56.5
5''-OMe	55.9	55.9
1'''	99.0	97.5
2'''	71.4	71.2
3'''	70.7	69.9
4'''	68.6	68.2
5'''	71.7	71.8
6'''	64.3	64.8

The X-Ray Analysis of Clemahexapetoside A (8)

Methods for X-ray crystal structure determination

A single crystal ($0.17 \times 0.12 \times 0.11$) of 1 was used for X-ray diffraction experiment, which was obtained from methanol: ethyl acetate (30:1). Crystal data were collected using a Bruker APEX-II CCD area detector diffractometer. A full sphere of the reciprocal space was scanned by phi-omega scans. Multi-scan absorption correction

based on redundant reflections was performed by the program SADABS^{S1}. The structure was solved by direct methods using SHELXS-97^{S2} and refined by full matrix least-squares on F^2 for all data using SHELXL-97^{S3}. Hydrogen atoms attached to oxygen were located in the difference Fourier map and constrained to ride on its parent atom with the O-H distance 0.84(0.85 for H atom of water molecules) Å and with $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$. All other hydrogen atoms were added at calculated positions and refined using a riding model. Their isotropic temperature factors were fixed to 1.2 (1.5 for methyl hydrogens) times the equivalent isotropic displacement parameters of its parent atom. Anisotropic temperature factors were used for all non-hydrogen atoms.

Crystal data

Molecular formula, C₂₉H₃₄O_{17·5}(H₂O). M=744.64. Temperature, 173(2)K. Wavelength, 0.71073 Å. Crystal system, monoclinic. Space group, P2₁. Unit cell dimentions: $a=7.7506(10)$ Å, $b=14.8393(19)$ Å, $c=14.9278(19)$ Å; $\alpha=90^\circ$, $\beta=92.947(2)^\circ$, $\gamma=90^\circ$. Volume, 1714.6(4) Å³. Z=2. Density (calculated), 1.442g/cm³. Absorption coefficient, 0.126cm⁻¹. $F(000)$, 788. Crystal size, 0.17×0.12×0.11mm³. Theta range for data collection, 1.37-26.00°. Index ranges, -9 ≤ h ≤ 9, -18 ≤ k ≤ 18, -16 ≤ l ≤ 18. Reflections collected, 9901. Independent reflections, 6237 [$R(\text{int})=0.0263$]. Completeness to theta=26.00°, 98.3%. Absorption correction, multi-scan. Max and min transmission, 0.9863 and 0.9790. Refinement method, full-matrix least-squares on F^2 . Data/restraints/parameters, 6237/1/478. Goodness-of-fit on F^2 , 1.065. Final R indices [$I>2\sigma(I)$]: $R_1=0.0510$, $wR_2=0.1254$; R indices (all data): $R_1=0.0648$, $wR_2=0.1364$. Largest diff. peak and hole, 0.657 and -0.230e/Å³. Full crystallographic details excluding structure features, have been deposited (deposition no. CCDC 814588) with the Cambridge Crystallographic Data Centre. The data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request.cif.

Table 2. Hydrogen bonds for macroglycoside **8** [Å and °]

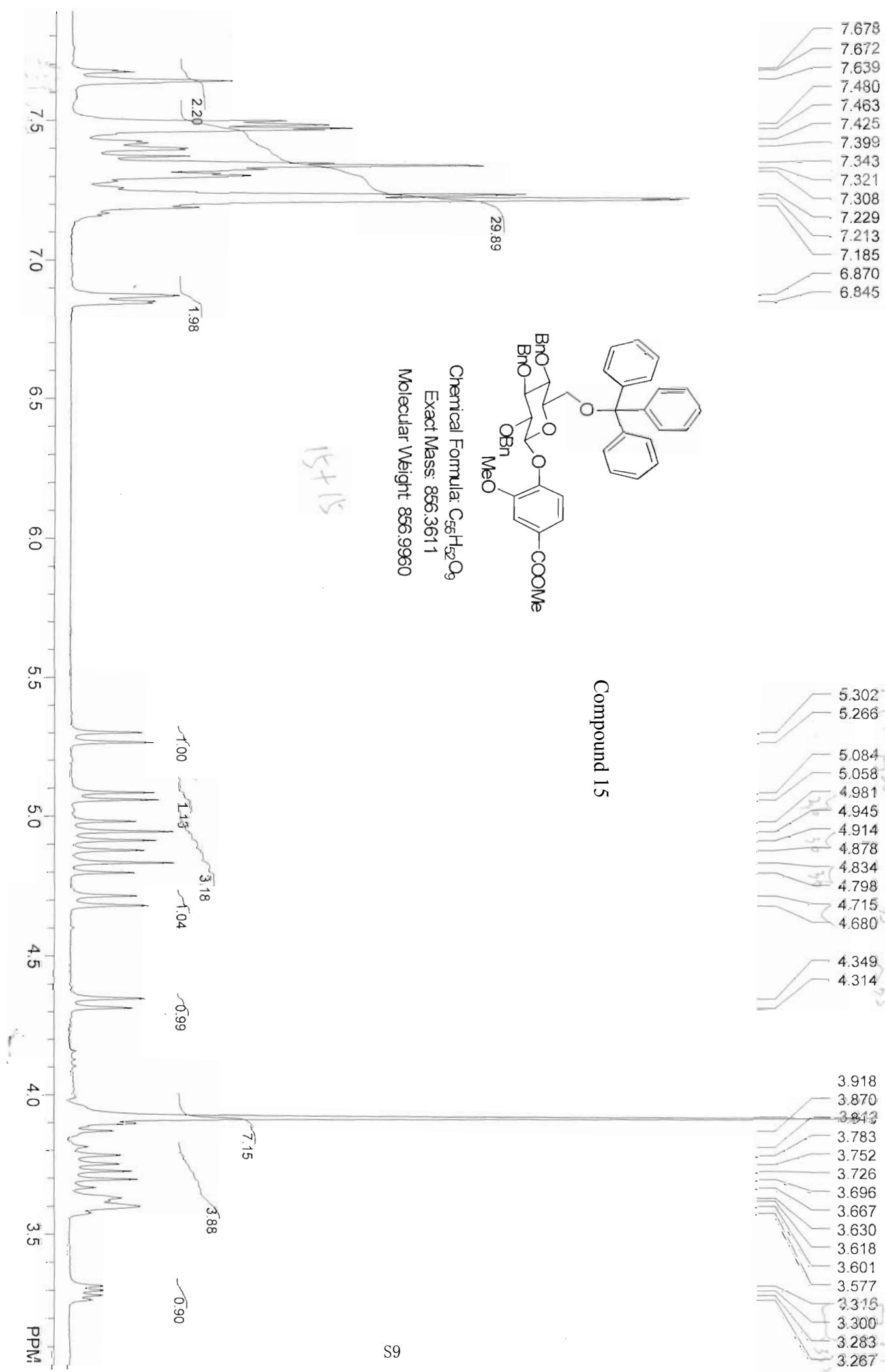
D-H···A	d(D-H)	d(H···A)	d(D···A)	∠(DHA)
O3-H3A···O18 ^a	0.84	1.89	2.726(4)	176.3
O4-H4A···O12 ^b	0.84	1.87	2.708(4)	173.7
O5-H5A···O18 ^b	0.84	1.81	2.620(3)	162.9

O11-H11…O21 ^c	0.84	2.01	2.793(5)	155.9
O12-H12A…O20 ^d	0.84	1.98	2.765(5)	154.9
O13-H13A…O19	0.84	1.83	2.672(4)	177.8
O18-H18A…O21 ^c	0.85	2.23	2.934(4)	140.7
O18-H18B…O12 ^e	0.85	2.26	2.938(5)	136.9
O19-H19A…O3 ^f	0.85	1.90	2.754(4)	179.9
O19-H19B…O20 ^a	0.85	1.85	2.704(5)	179.8
O20-H20C…O5 ^g	0.85	1.81	2.659(4)	179.5
O20-H20D…O17	0.85	2.43	2.847(4)	110.0
O21-H21A…O4 ^g	0.85	2.04	2.766(4)	142.7
O21-H21B…O9 ^a	0.85	2.43	3.026(4)	127.3
O22-H22B…O7 ^h	0.85	1.97	2.822(8)	179.5
O23-H23B…O14	0.85	2.11	2.946(8)	166.6

Symmetry transformations used to generate equivalent atoms: ^a -x+1,y+1/2,-z+1; ^b x-1,y,z-1; ^c -x+1,y-1/2,-z+1; ^d x,y,z+1; ^e -x+1,y-1/2,-z+2; ^f x+1,y,z+1; ^g x+1,y,z; ^h x,y+1,z.

References

- S1. Sheldrick, G. M. SADABS, Bruker AXS Inc., Madison, WI 53711, 2000.
- S2. Sheldrick, G. M. SHELXS-97: Program for Crystal Structure Solution, Göttingen, 1997.
- S3. Sheldrick, G. M. SHELXL-97: Program for Crystal Structure Refinement, Release 2, Göttingen, 1997.



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Archive directory:

Sample directory:

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Pulse Sequence: CARBON (s2px1)
Solvent: cdc13
Data collected on: Apr 28 2010

Temp. 25.0 C / 298.1 K

Operator: bsz

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Pulse 45.0 degrees
Acq. time 1.301 sec

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DECOUPLE H1, 399.6242471 MHz

Power 43 dB

continuously on
WLITZ-16 modulated

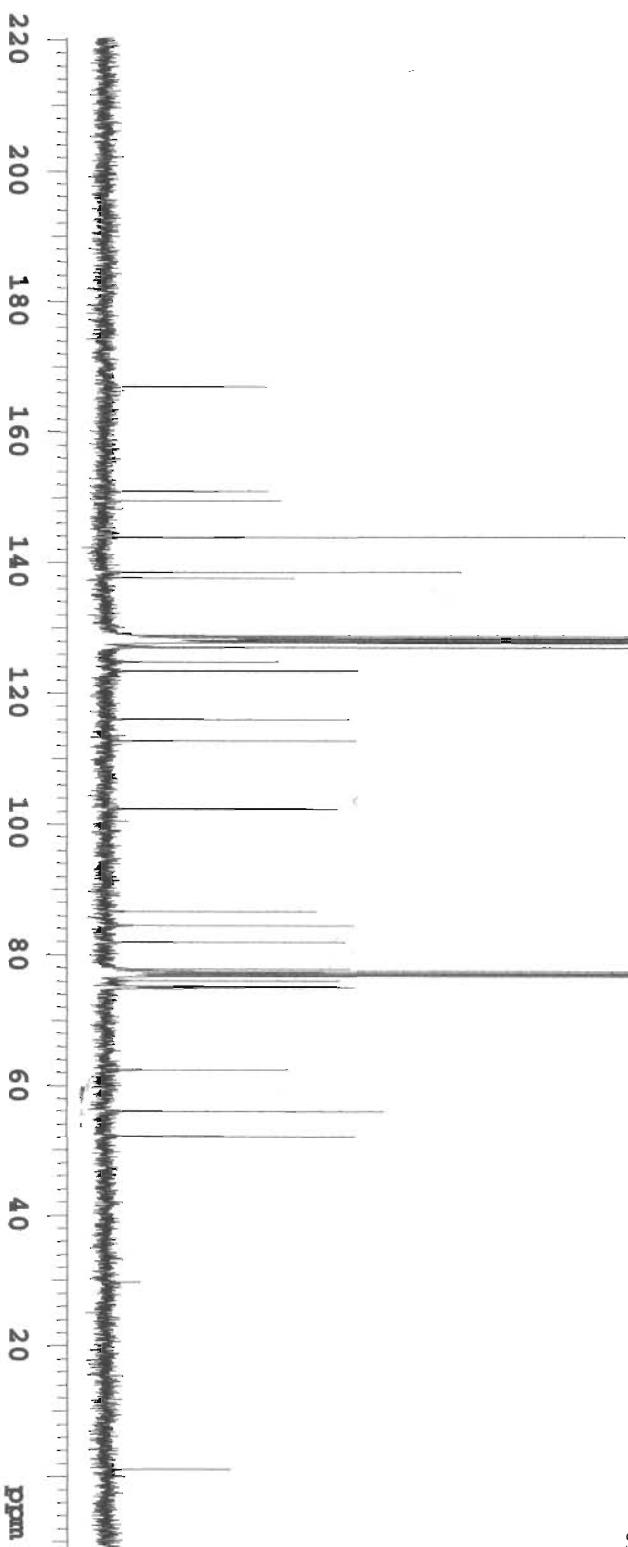
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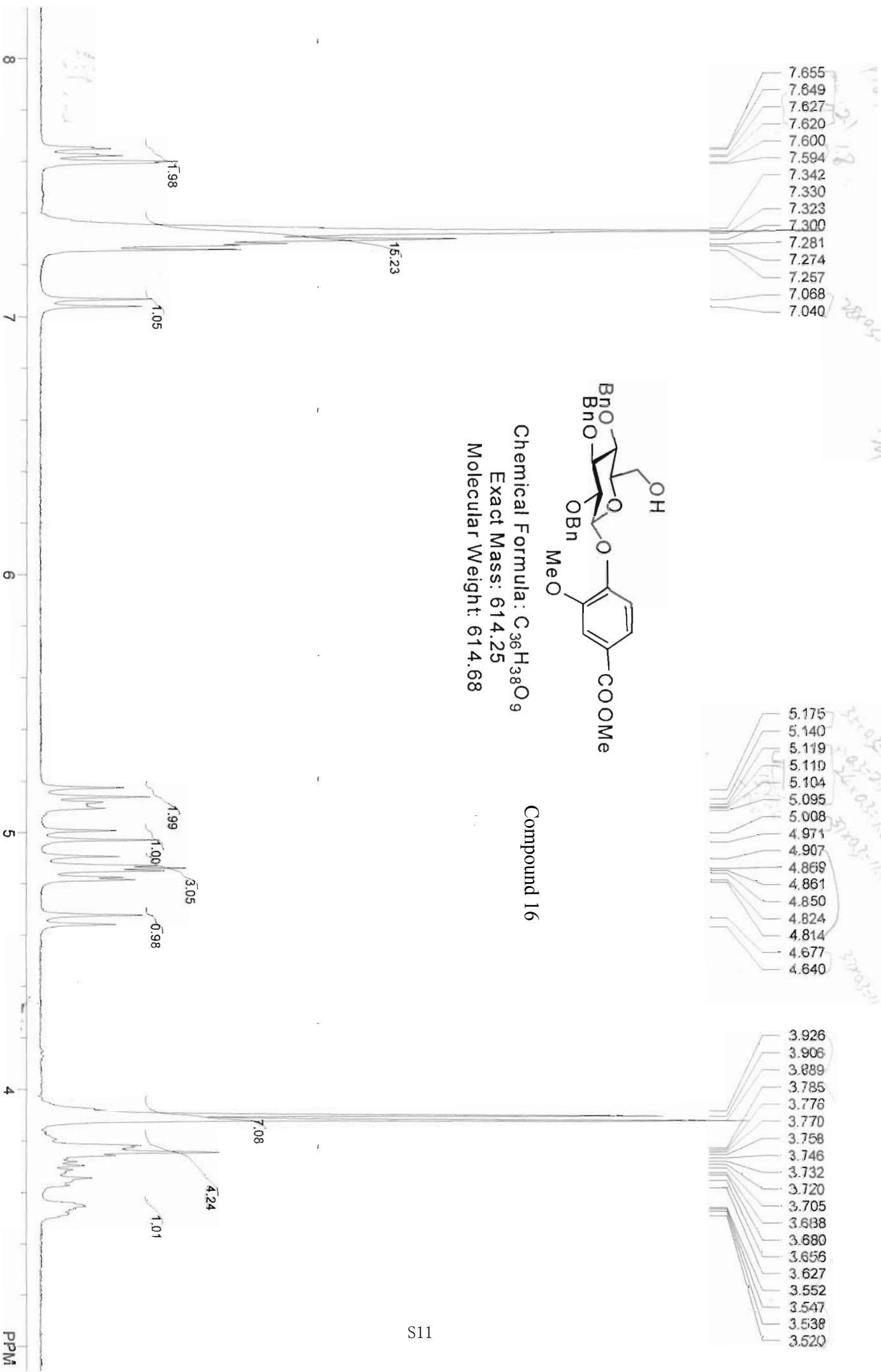
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Compound 15





File: xp

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Solvent: cdc13

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Operator: vnmr1

VNMR.S-400 "400MR"

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Pulse 45.0 degrees

Acq. time 1.300 sec

Width 30487.8 Hz

234 repetitions

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DECOPPLE H1, 399.622471 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

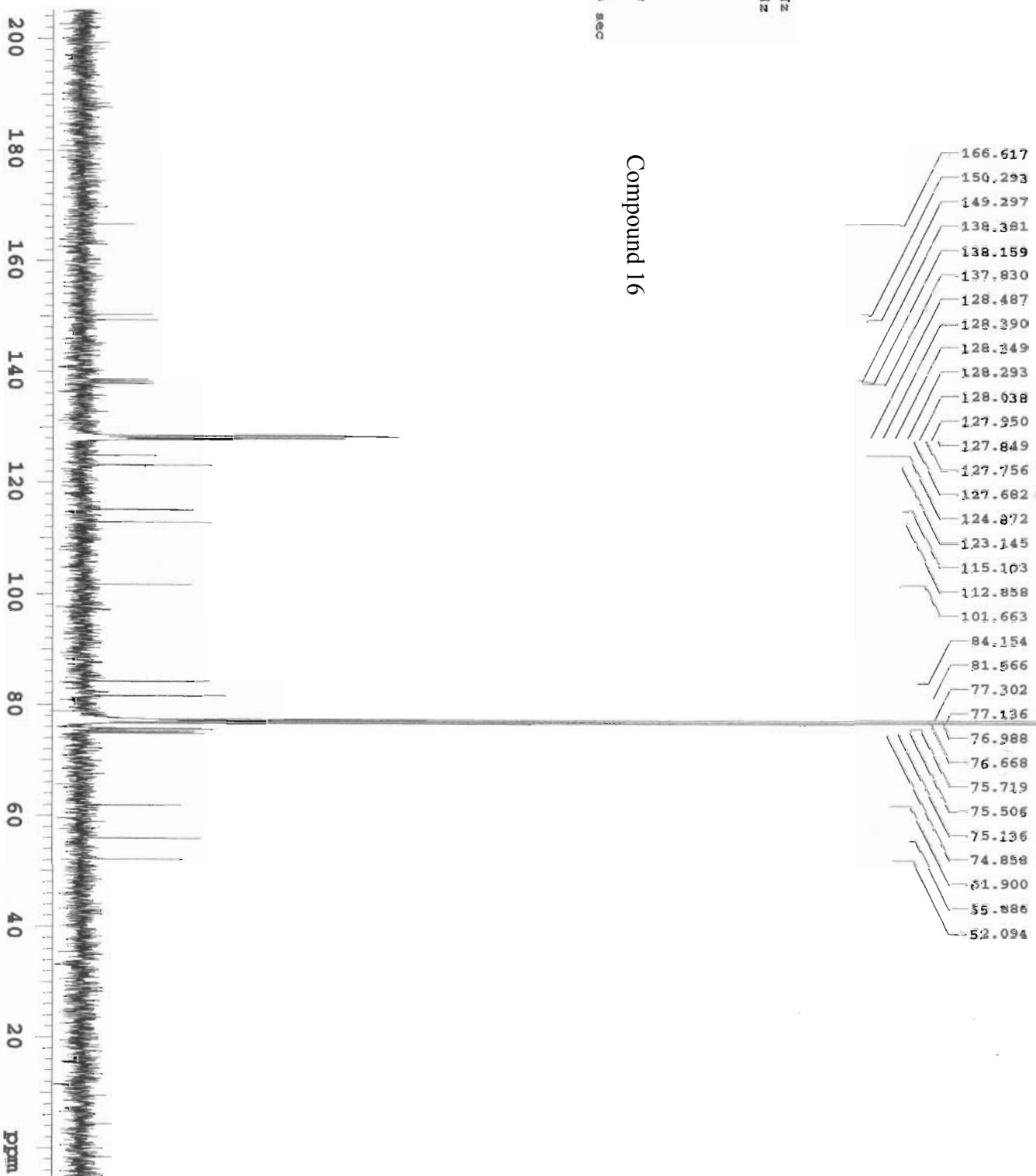
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Gauss apodization 0.390 sec

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Compound 16





File: xP

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: vnmr1

VNMR-S-400 "400MM"

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 30487.8 Hz

704 repetitions

OBSERVE C13, 100.4850962 MHz

DECOUPLE H1, 399.6242471 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

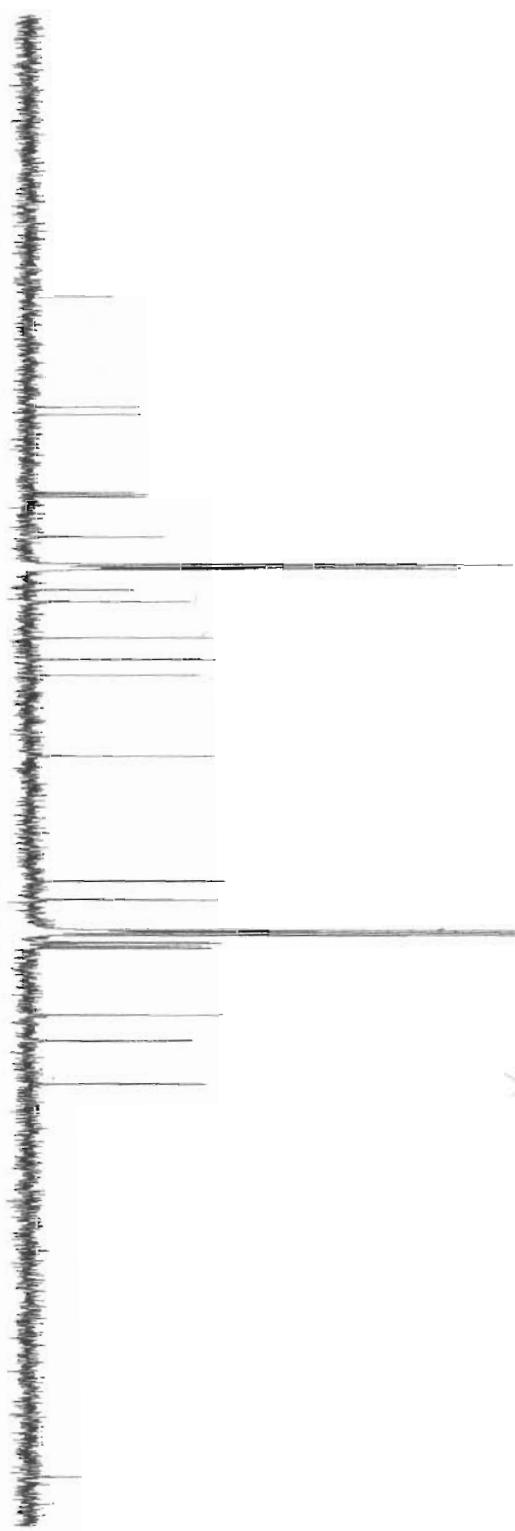
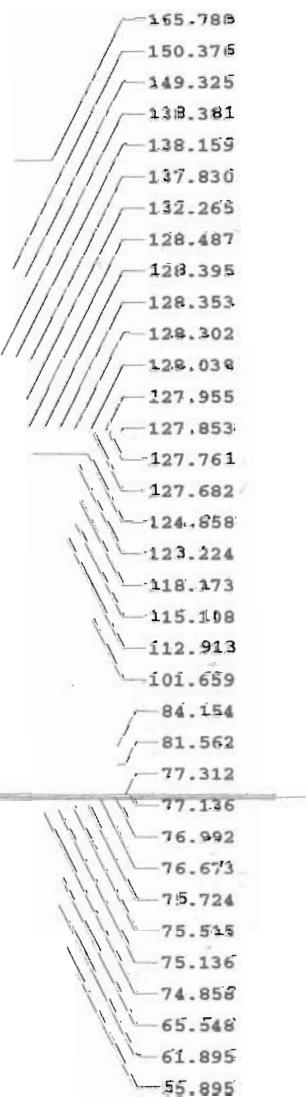
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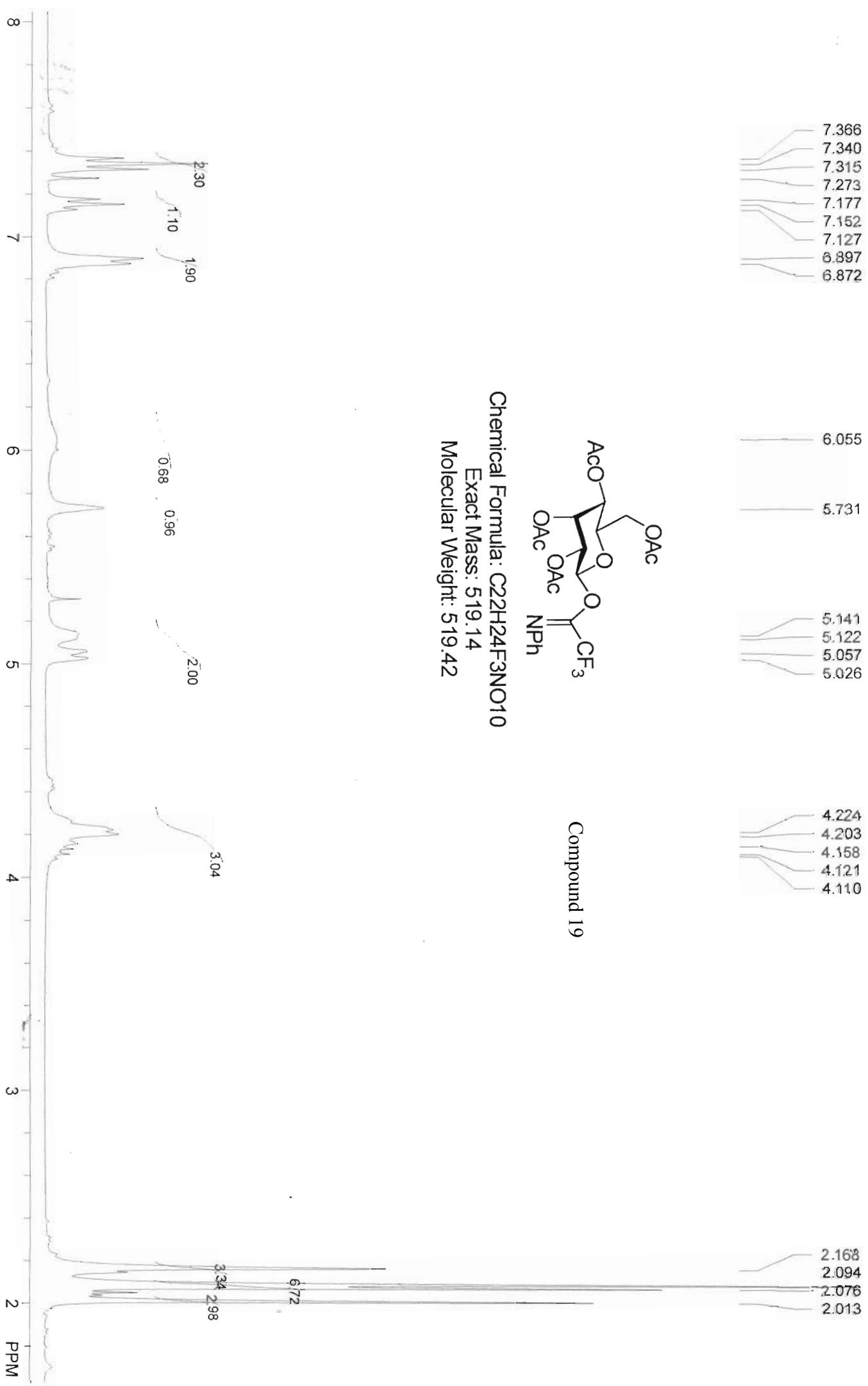
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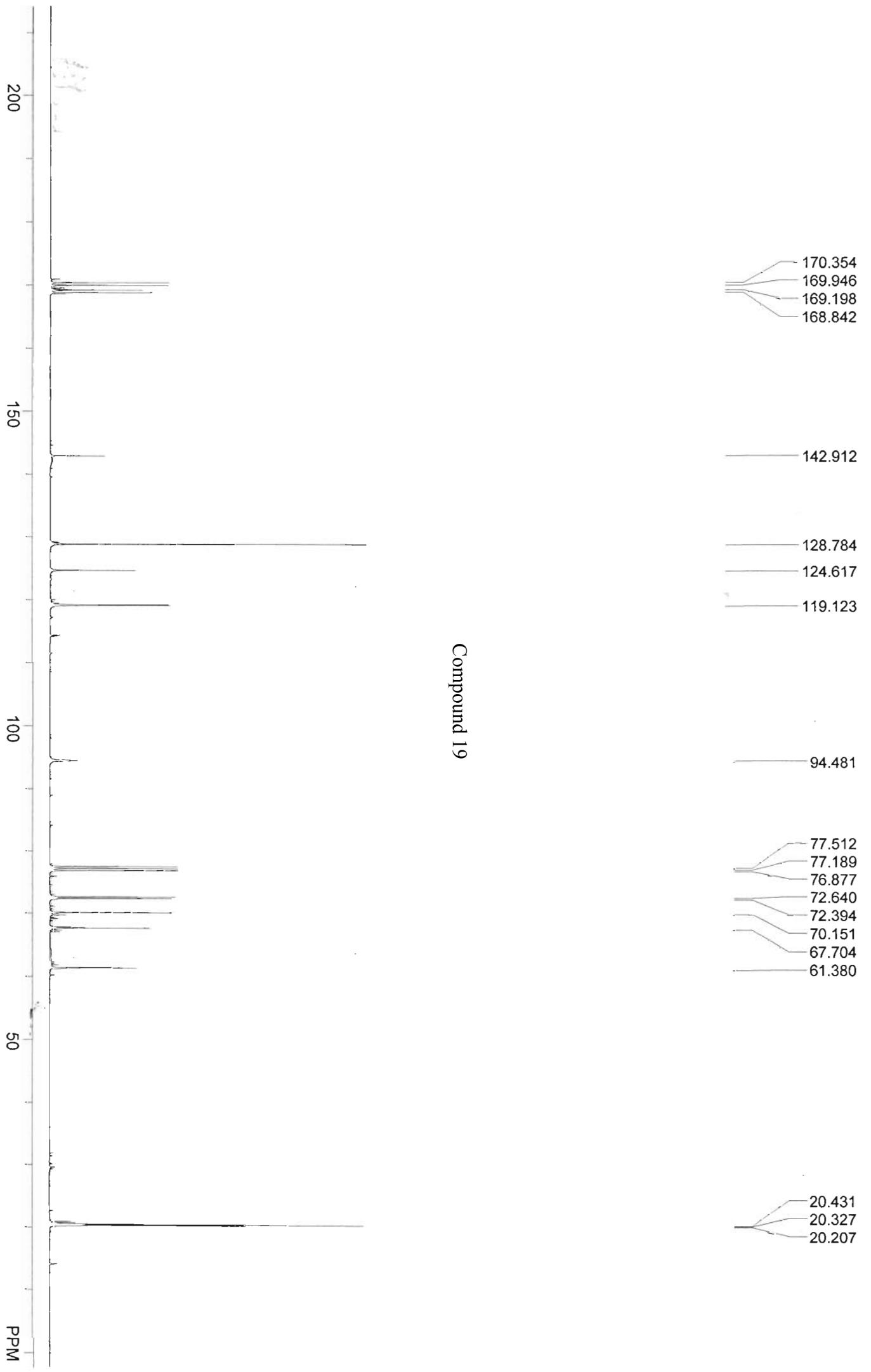
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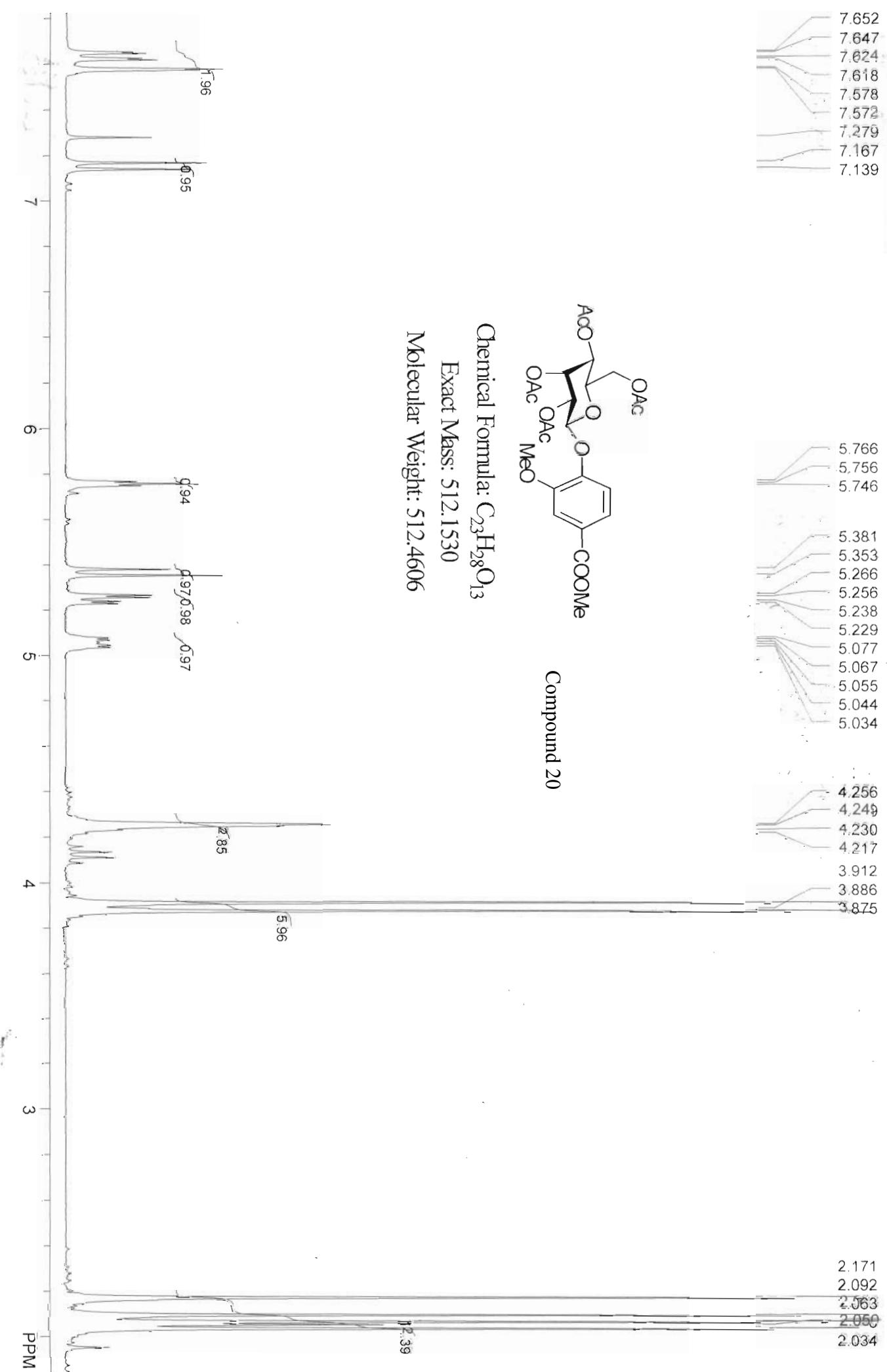
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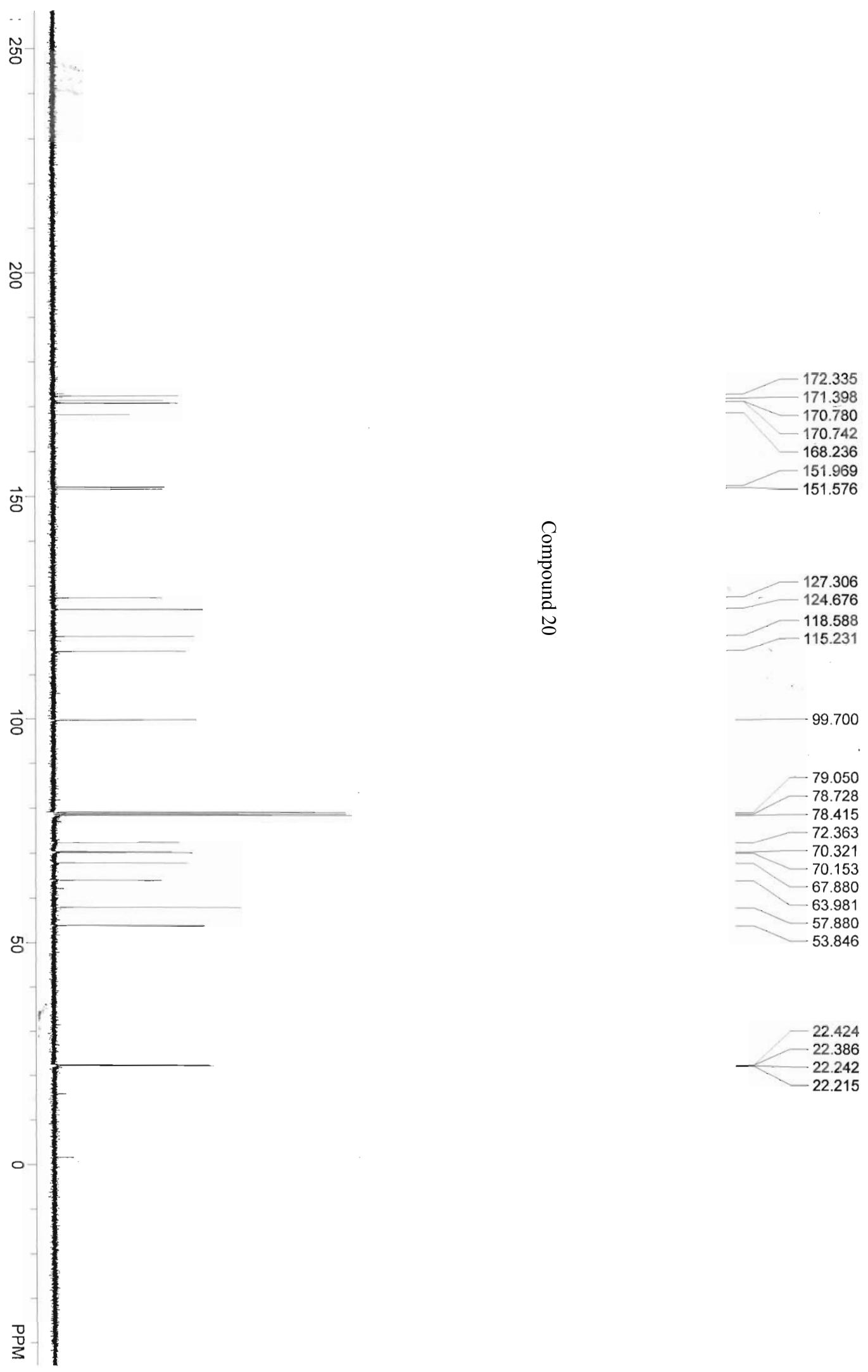


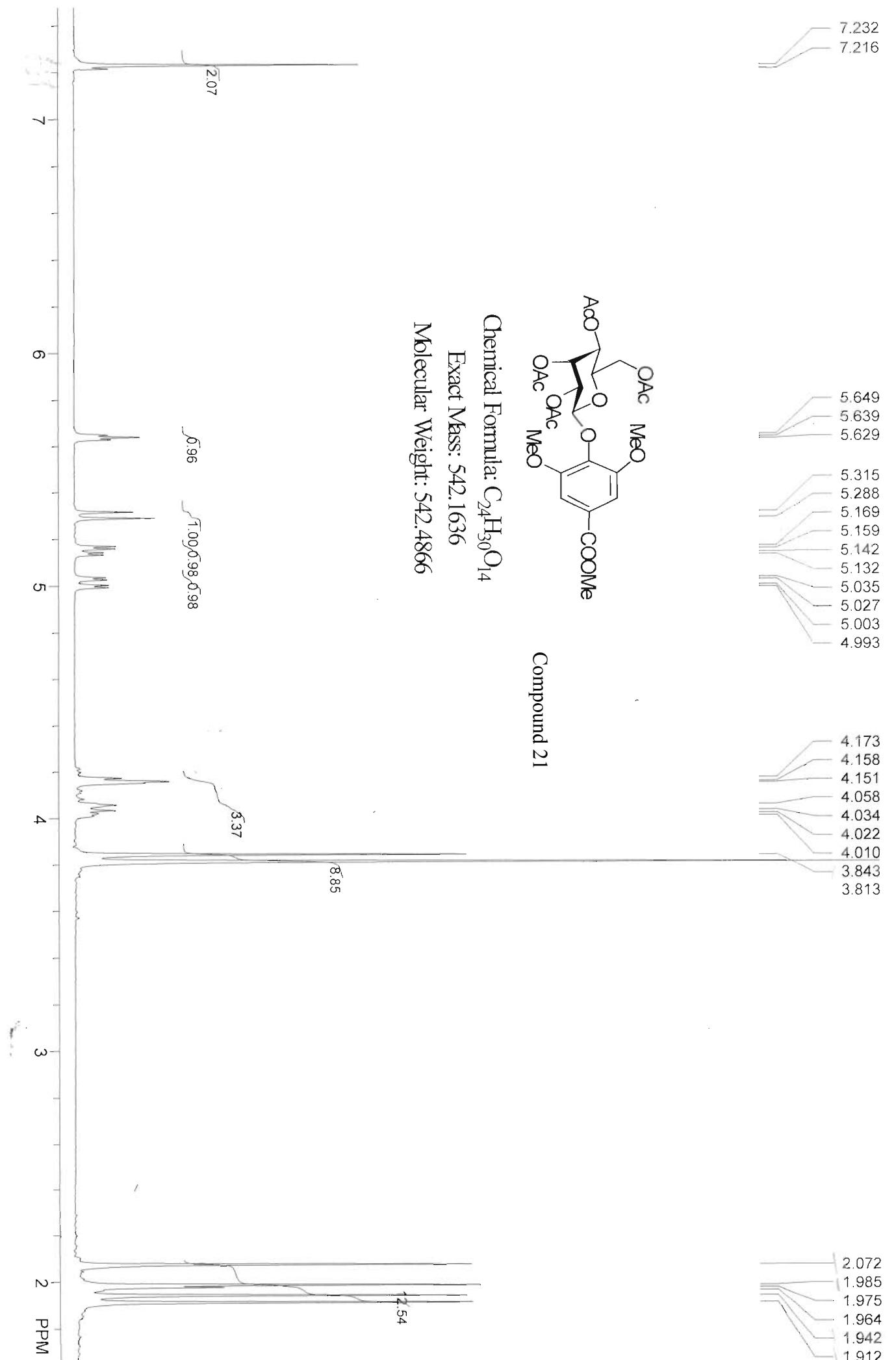
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file: xpi

Pulse Sequence: s2pul

Solvent: cdg13

Temp. 25.0 C / 298.1 K

Operator: vnmrl

VNMRB-400 "400MR"

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 30487.8 Hz

198 repetitions

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DECOPLE H1, 399.6242471 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

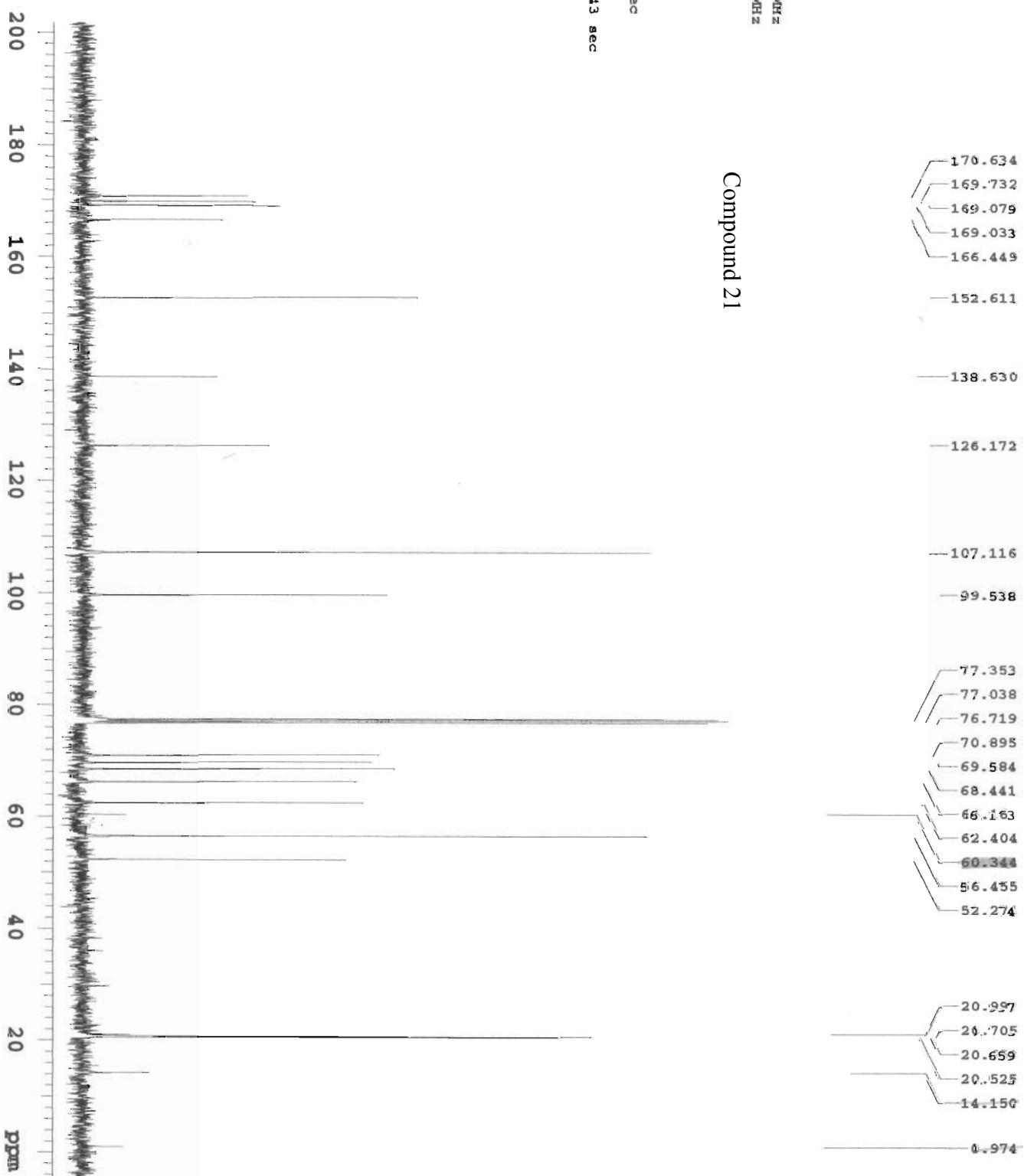
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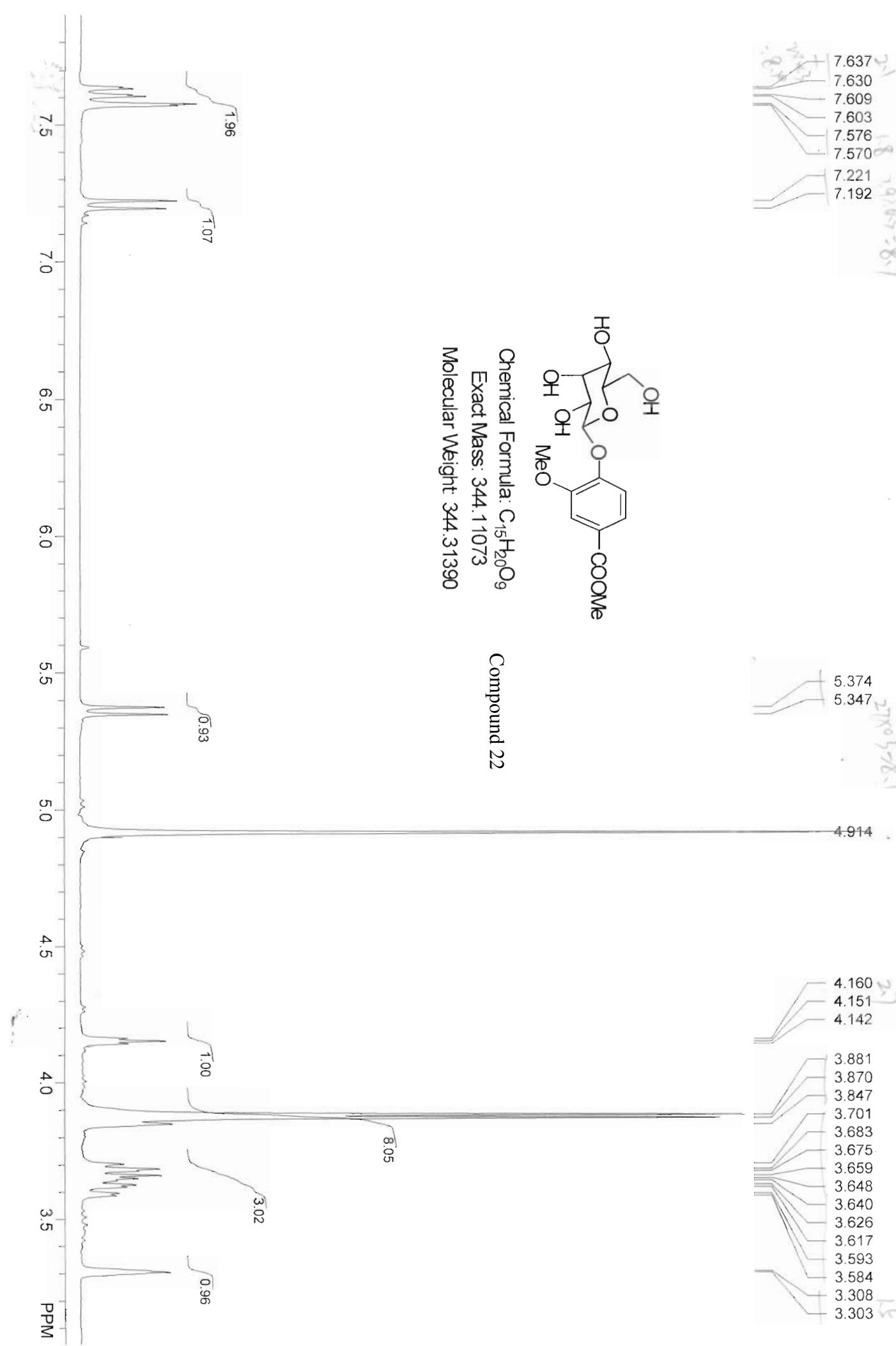
Gauss apodization 0.390 sec

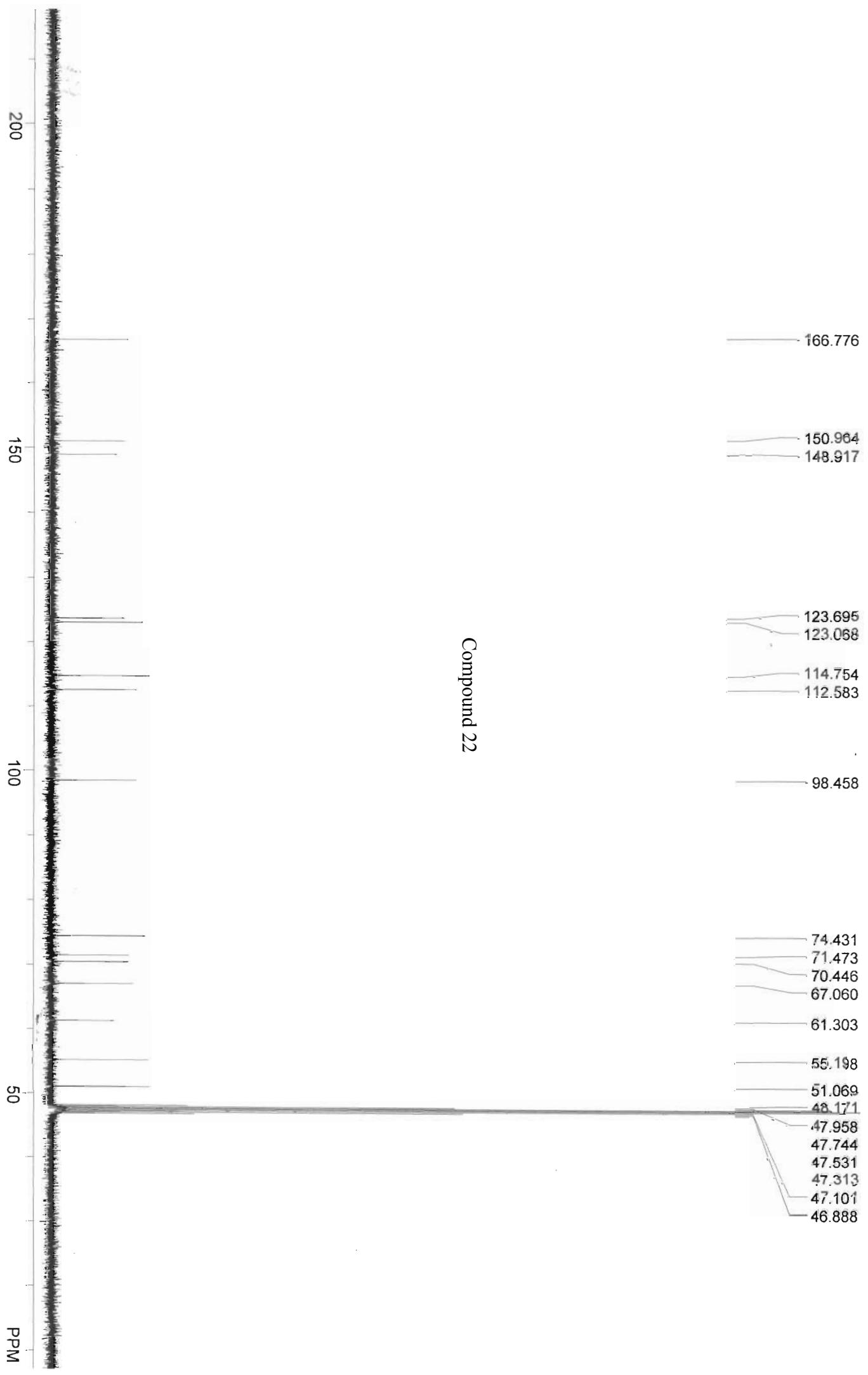
FT size 131072

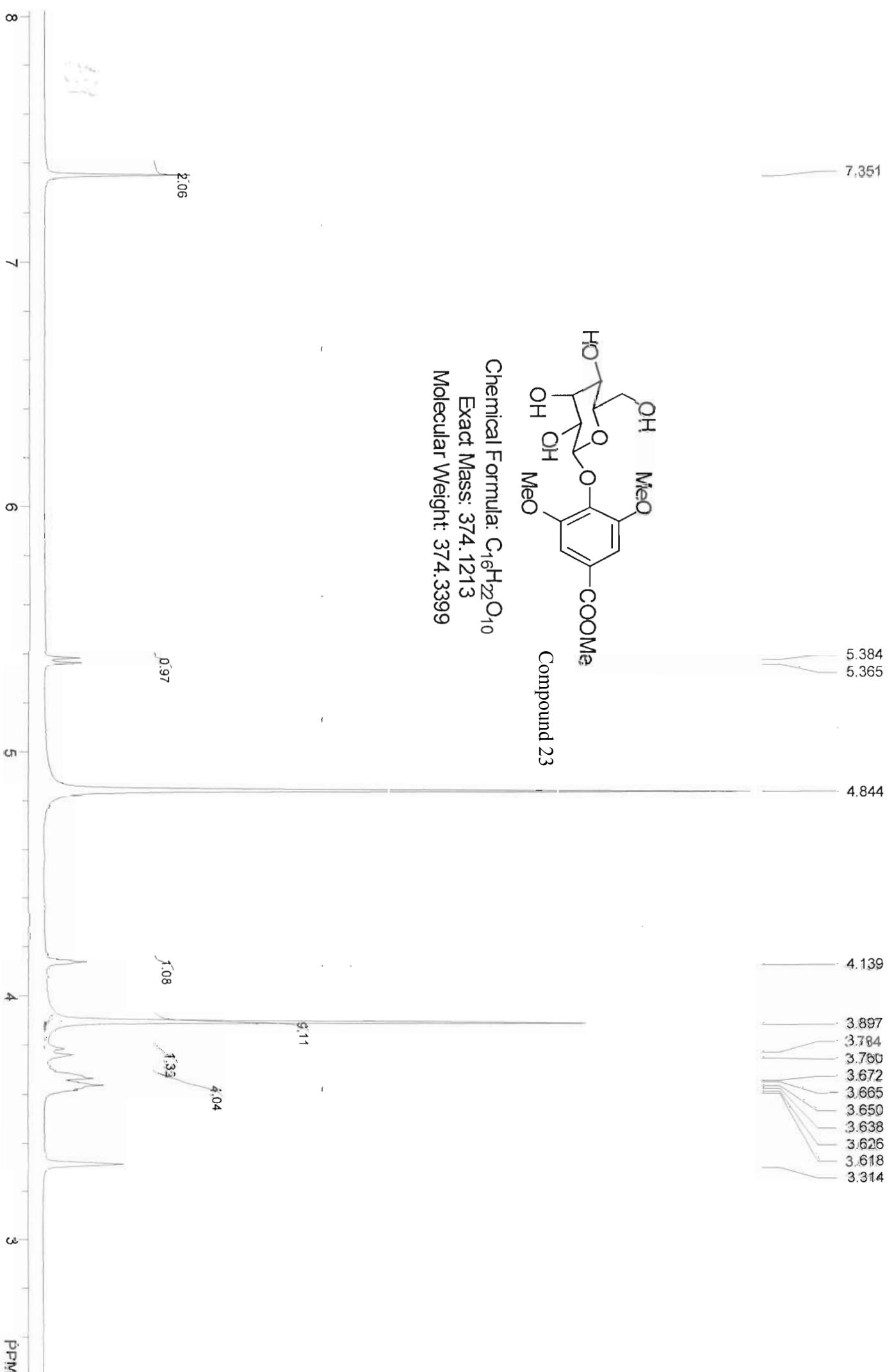
Total time 27 hr, 34 min, 43 sec

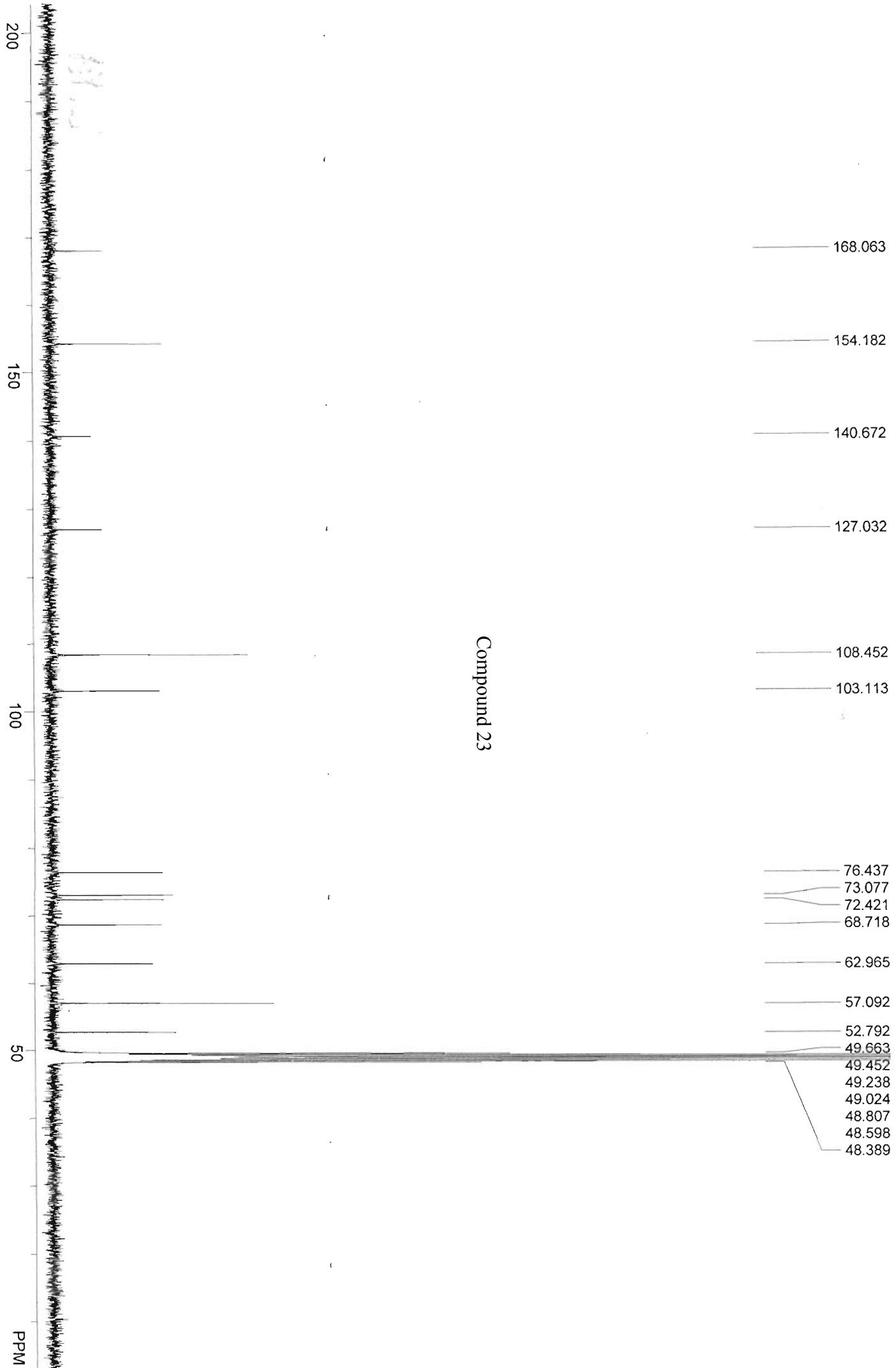
Compound 21

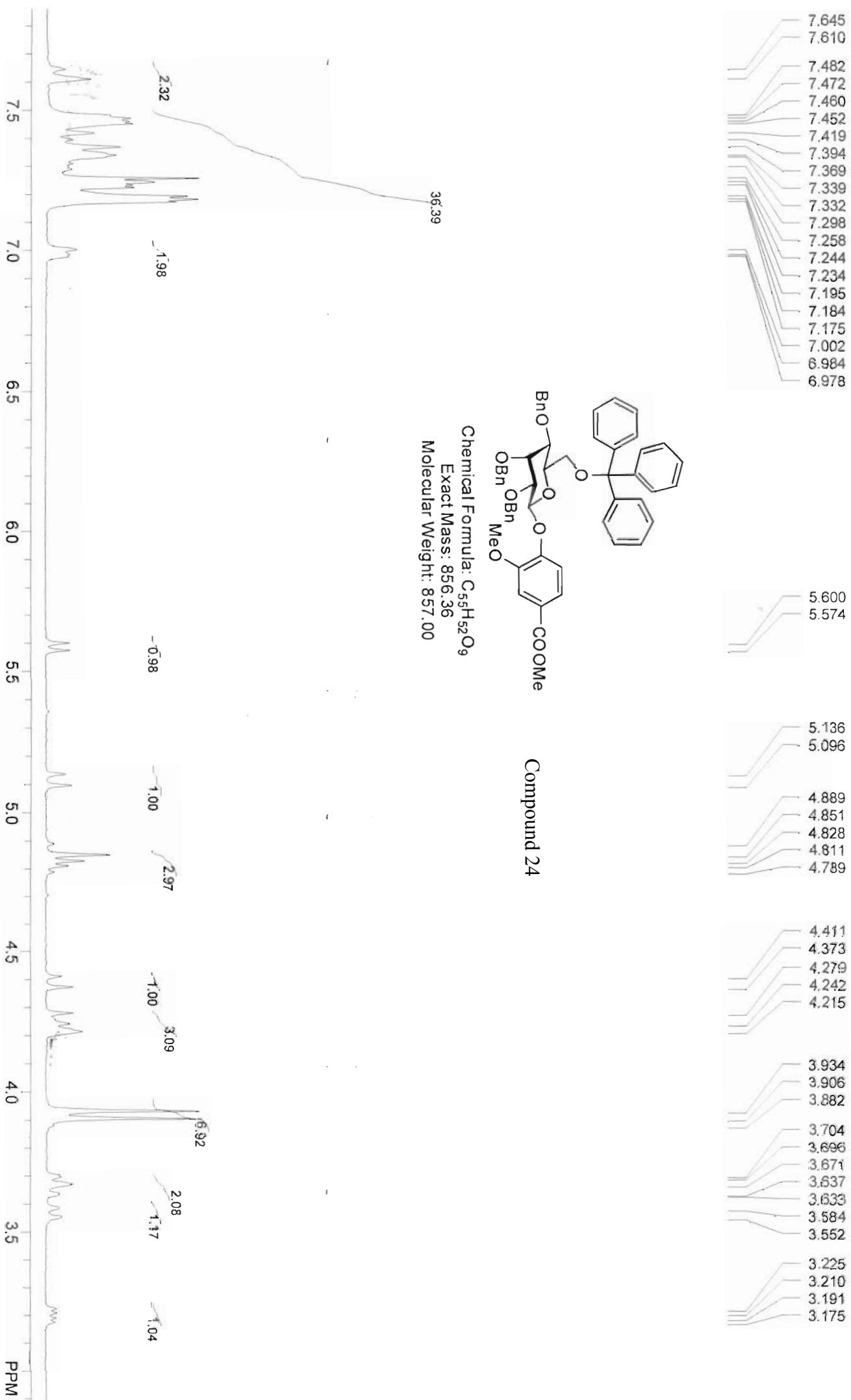


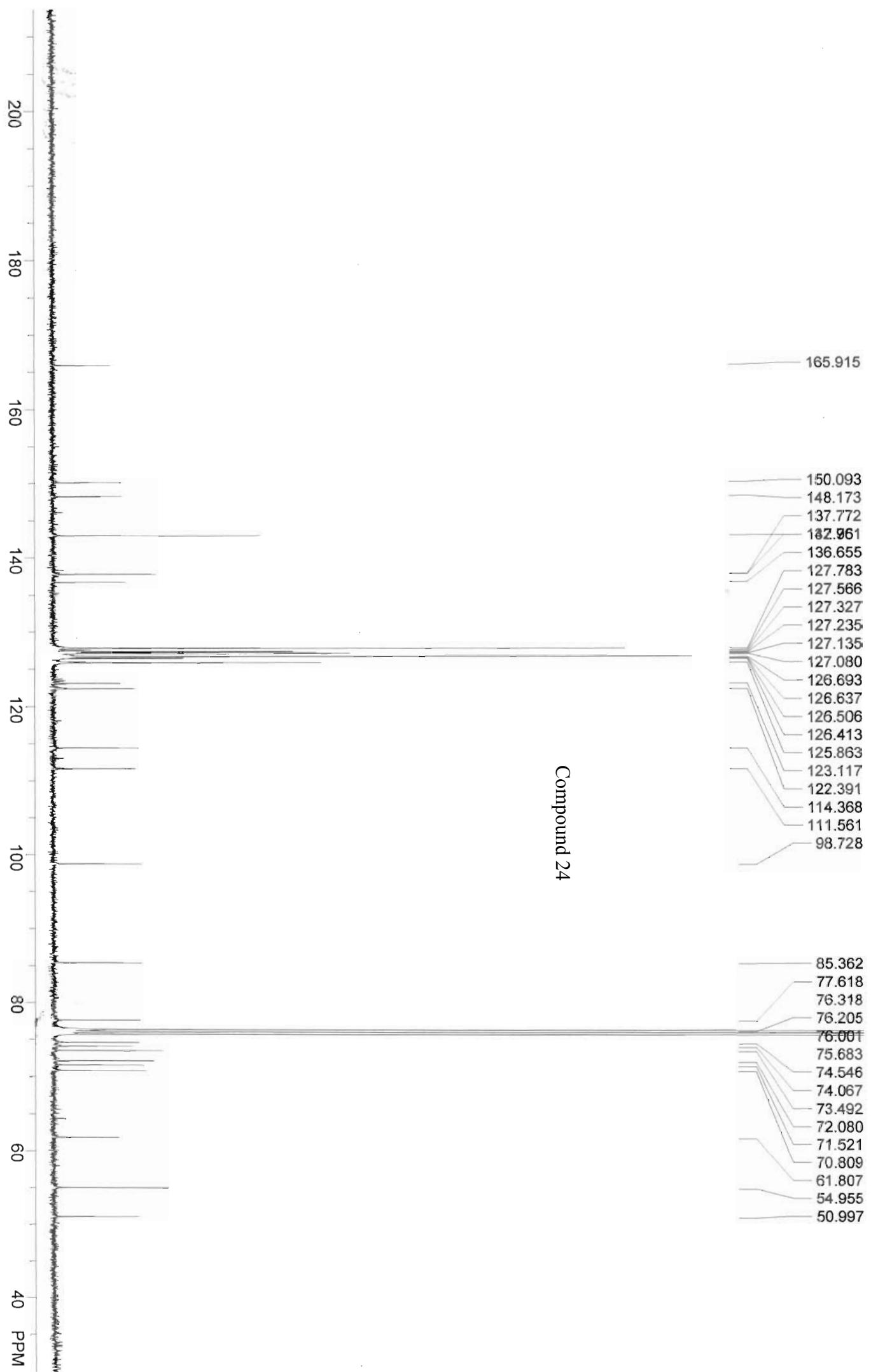


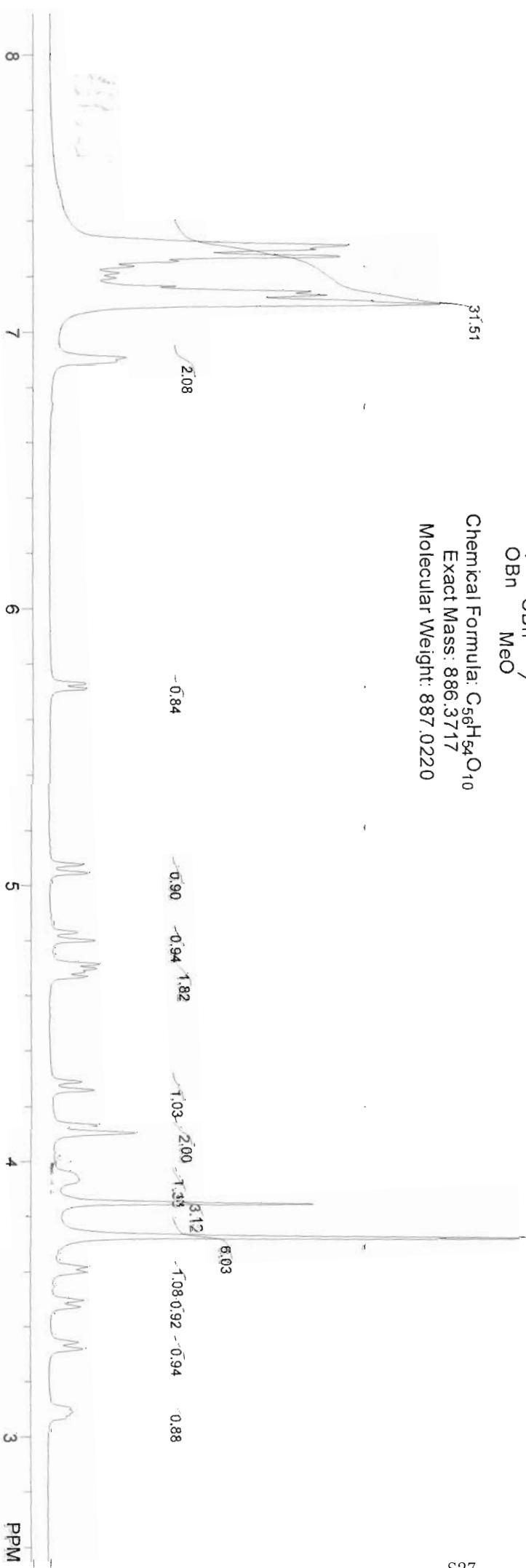




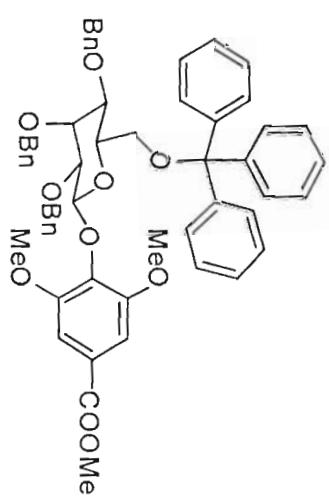




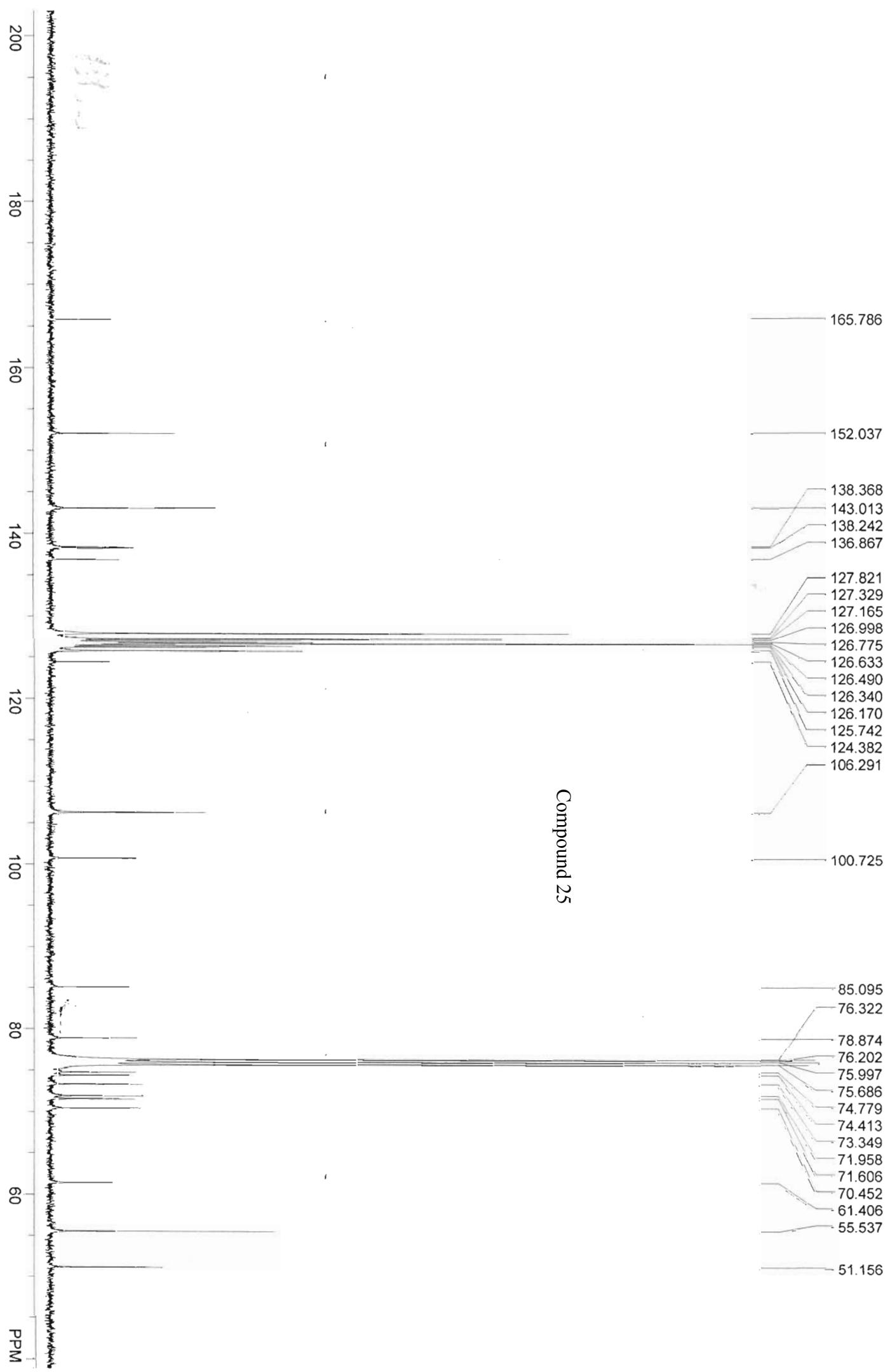


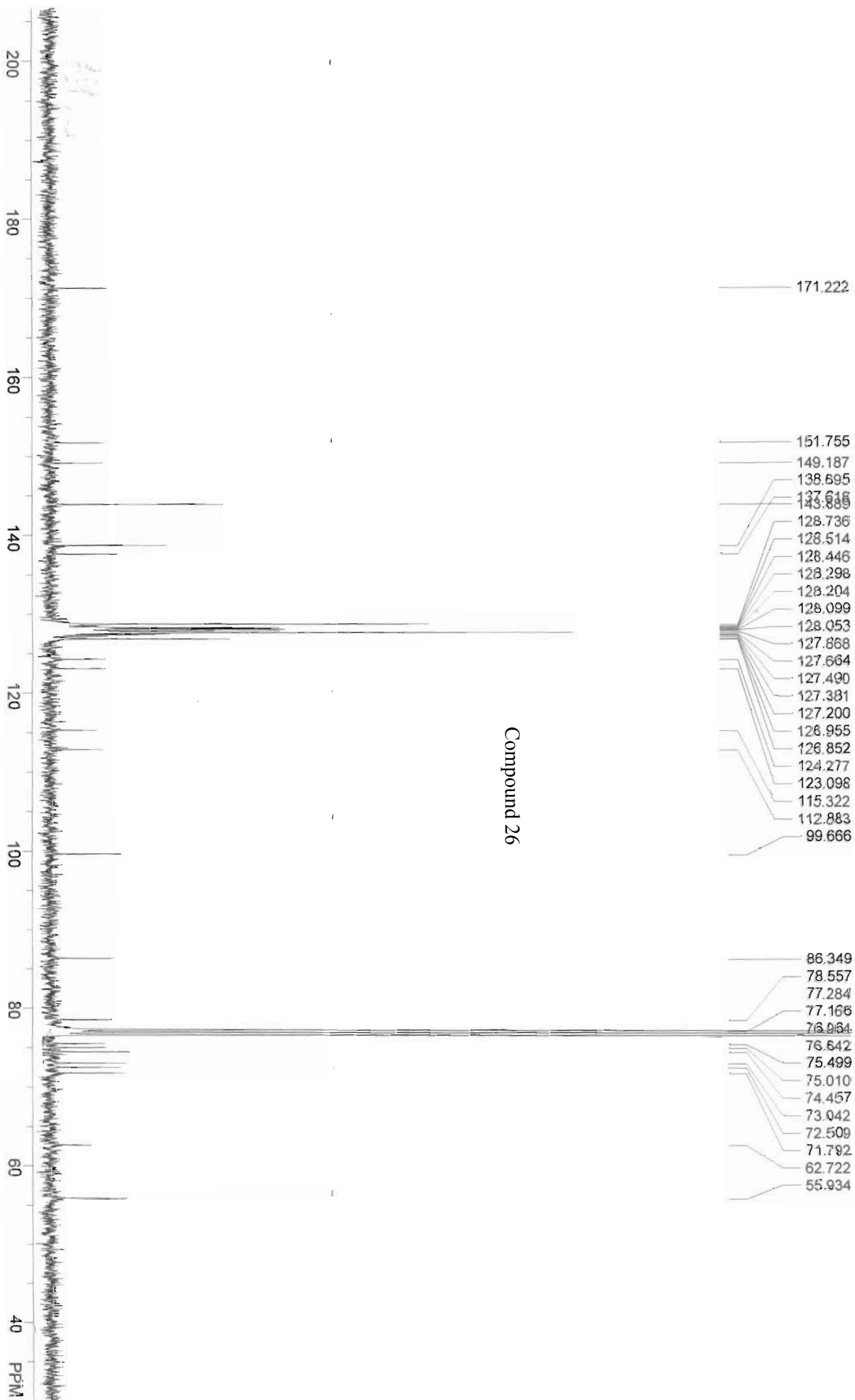


Chemical Formula: C₅₆H₅₄O₁₀
Exact Mass: 886.3717
Molecular Weight: 887.0220

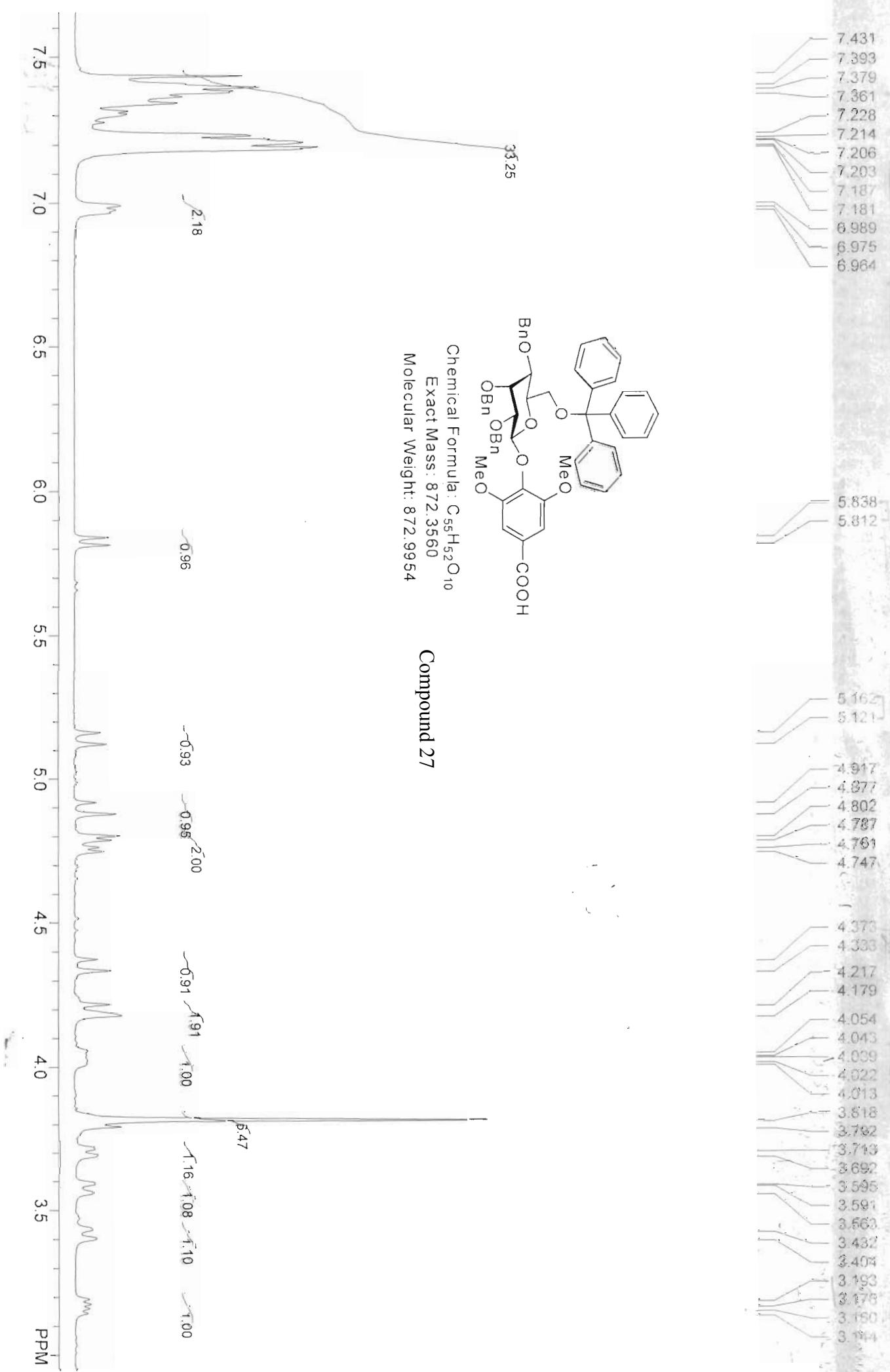


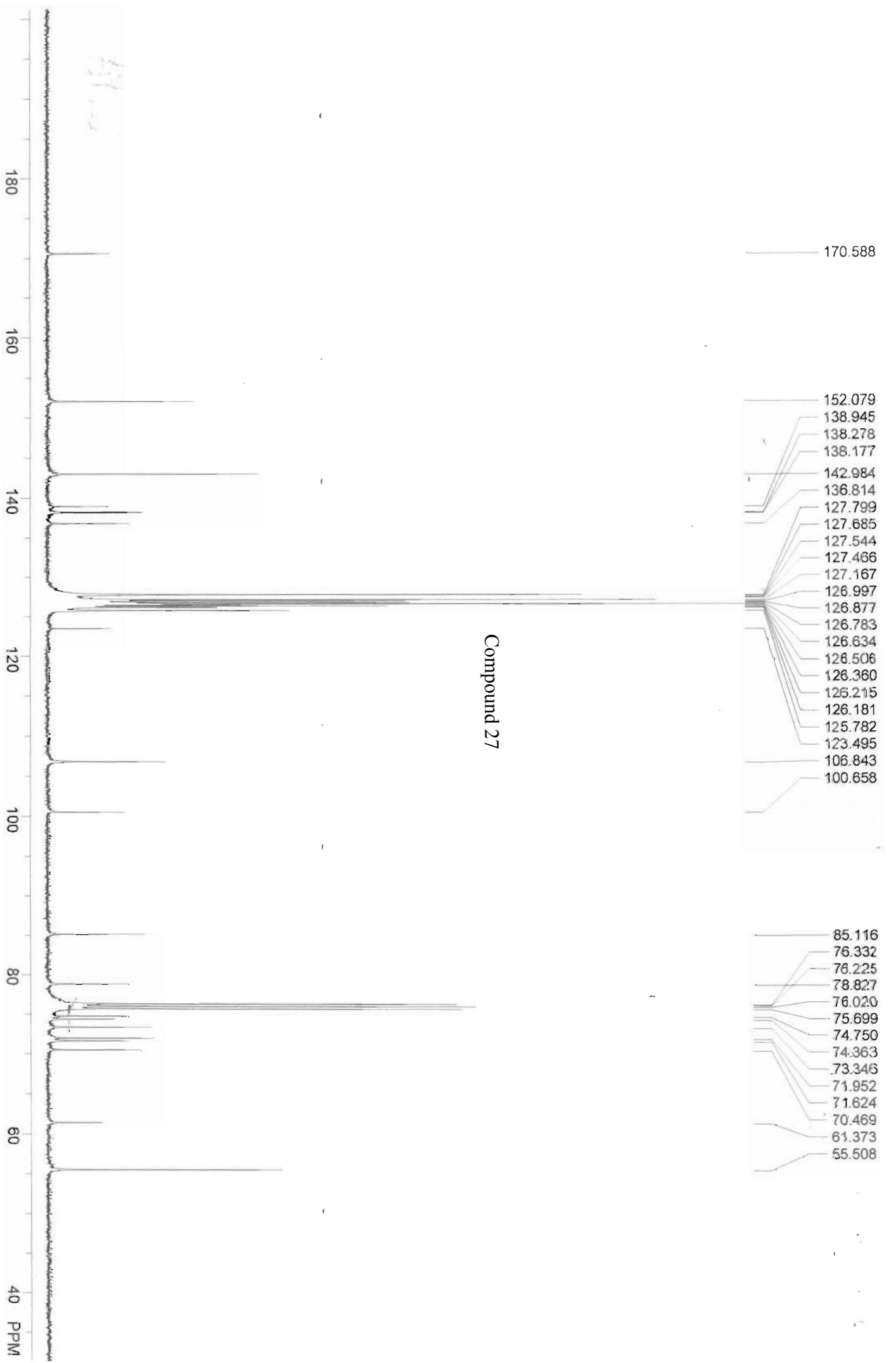
Compound 25

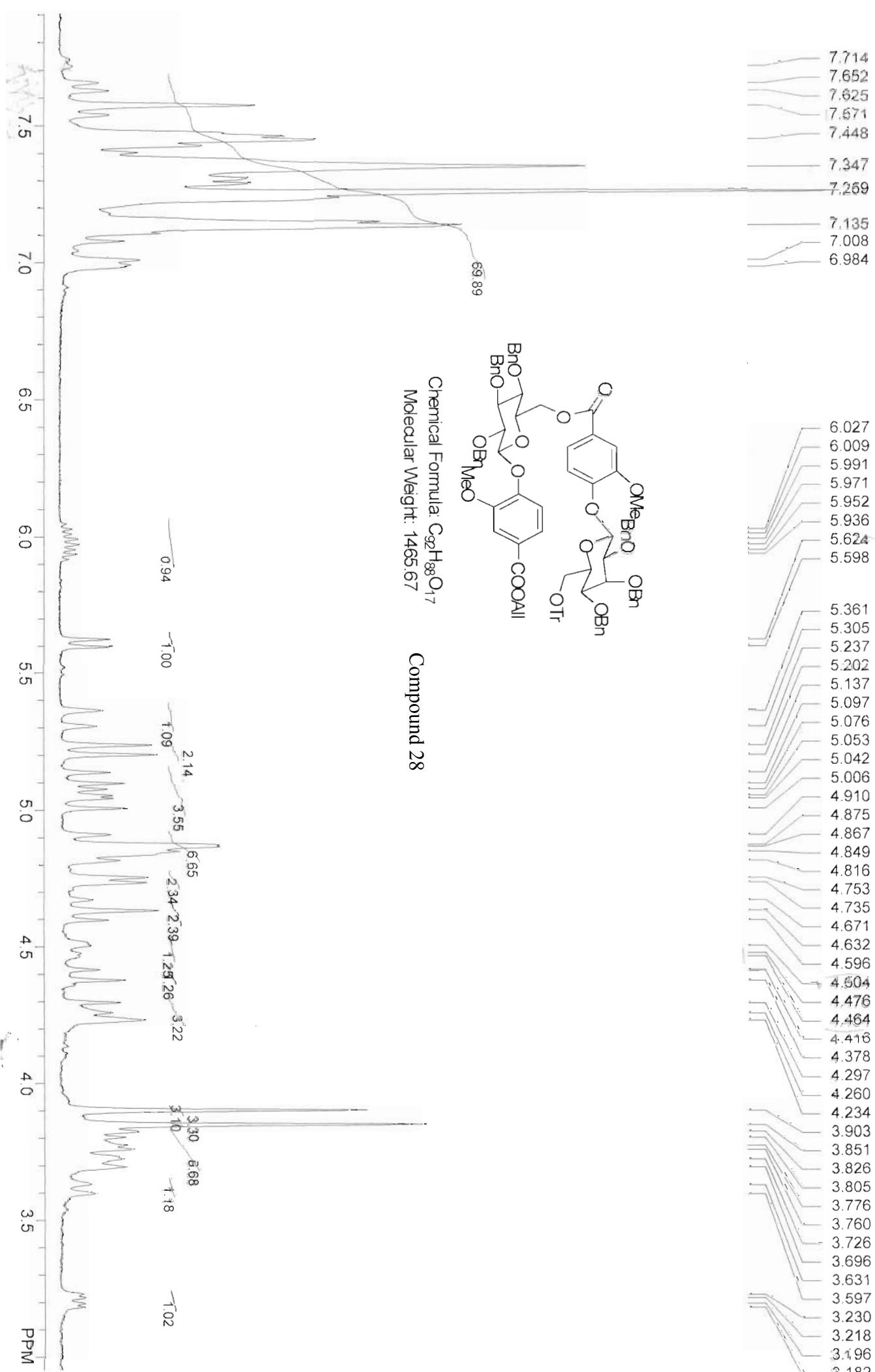


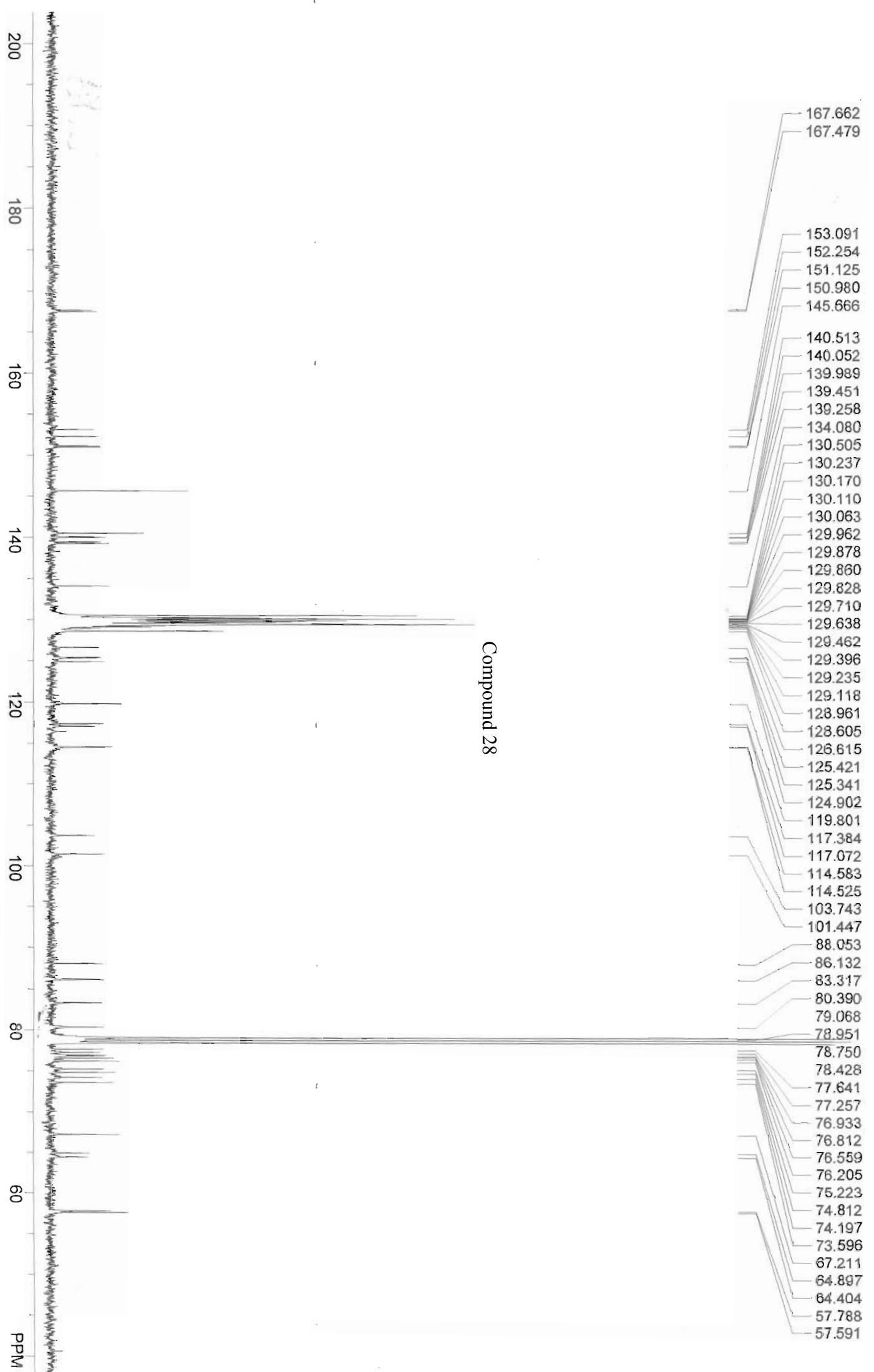


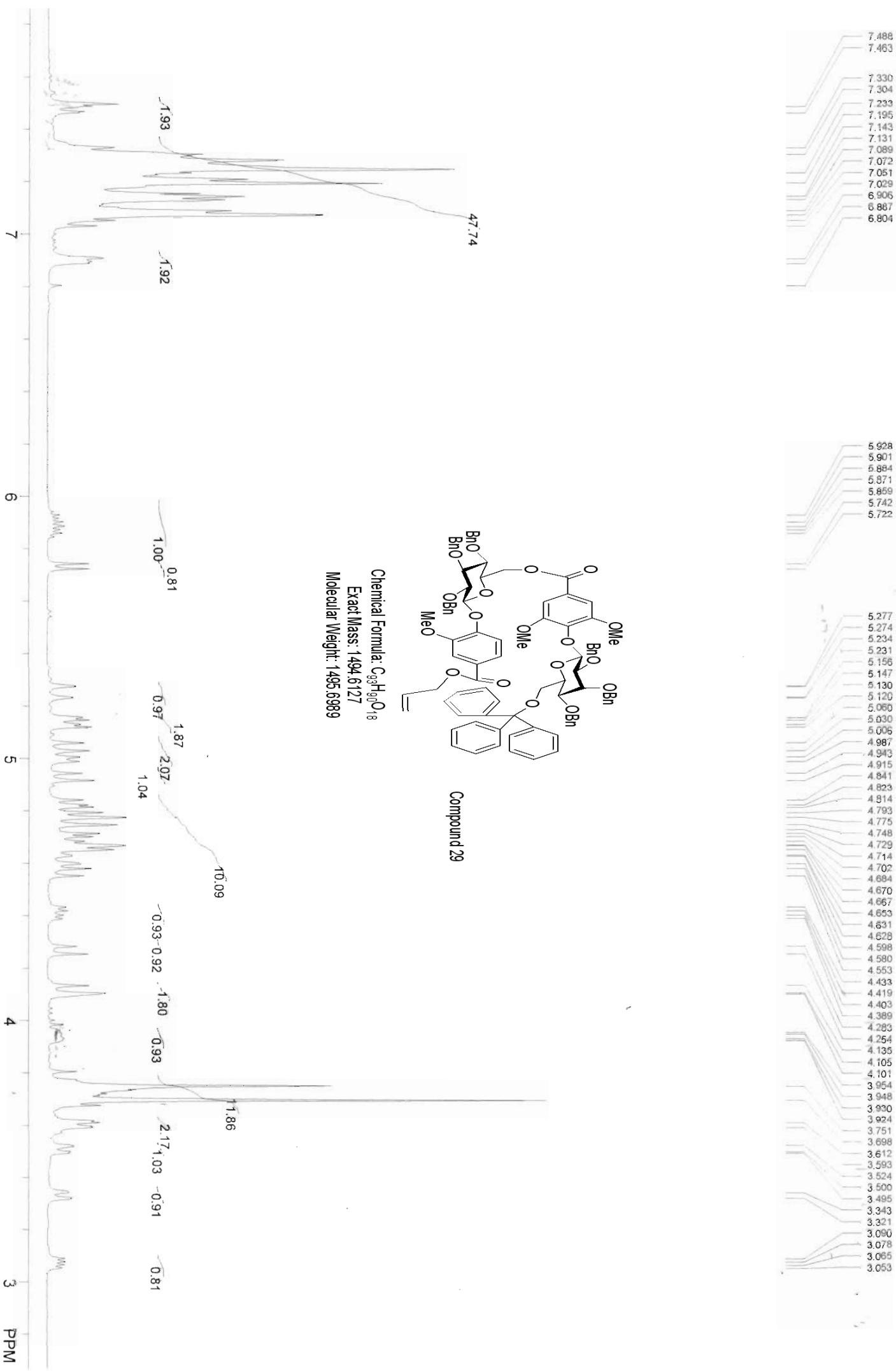
Compound 26

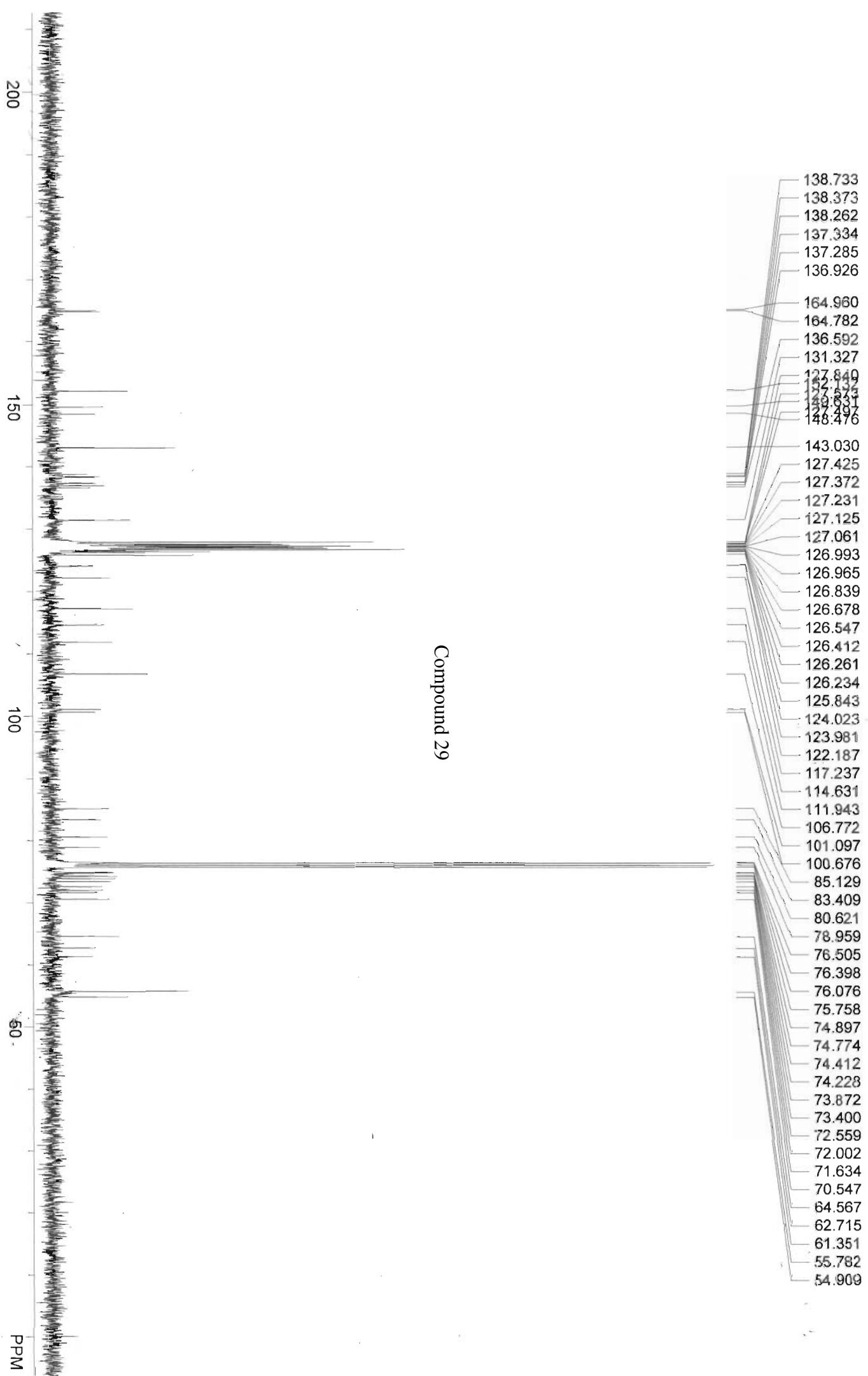


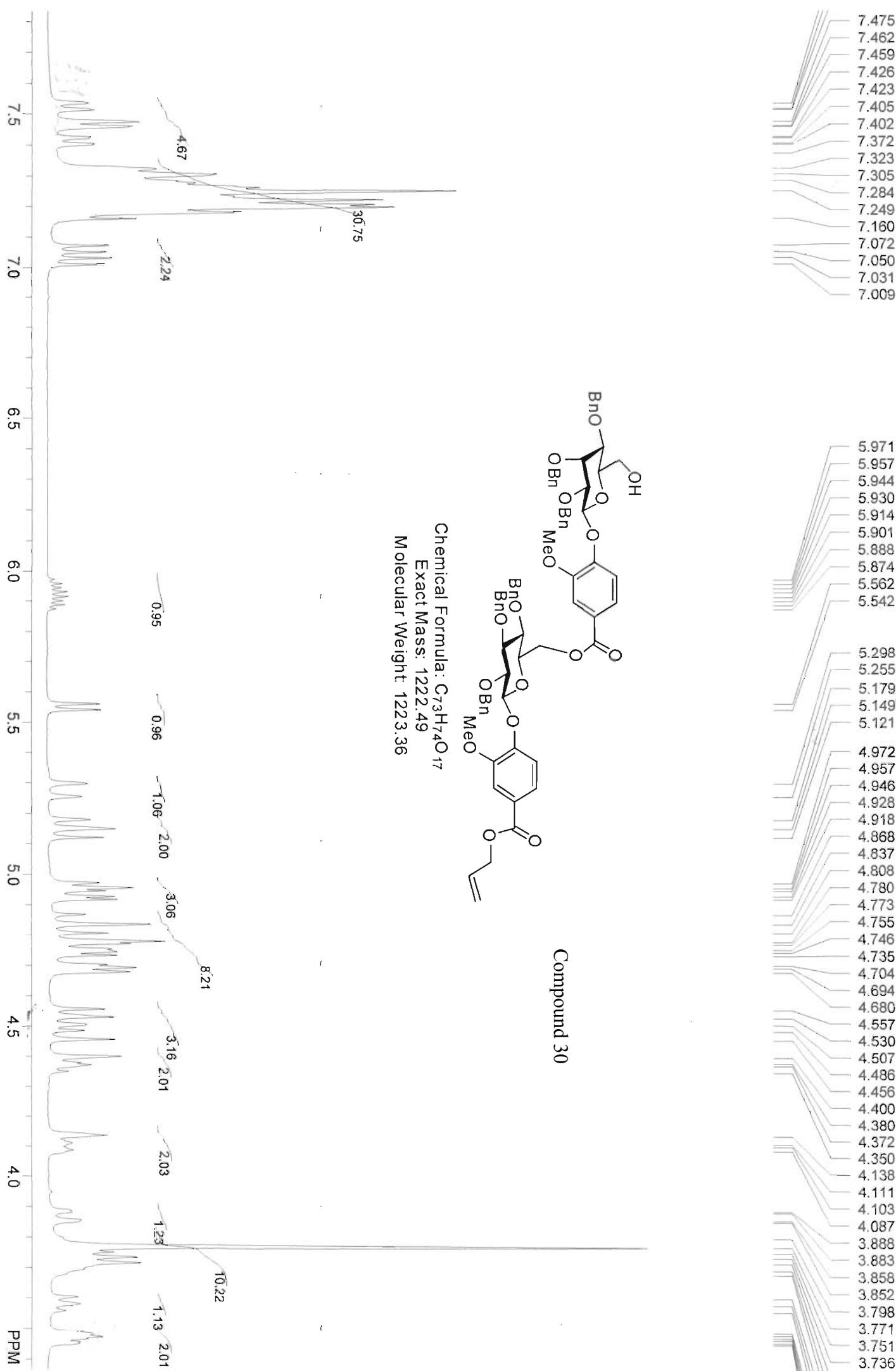


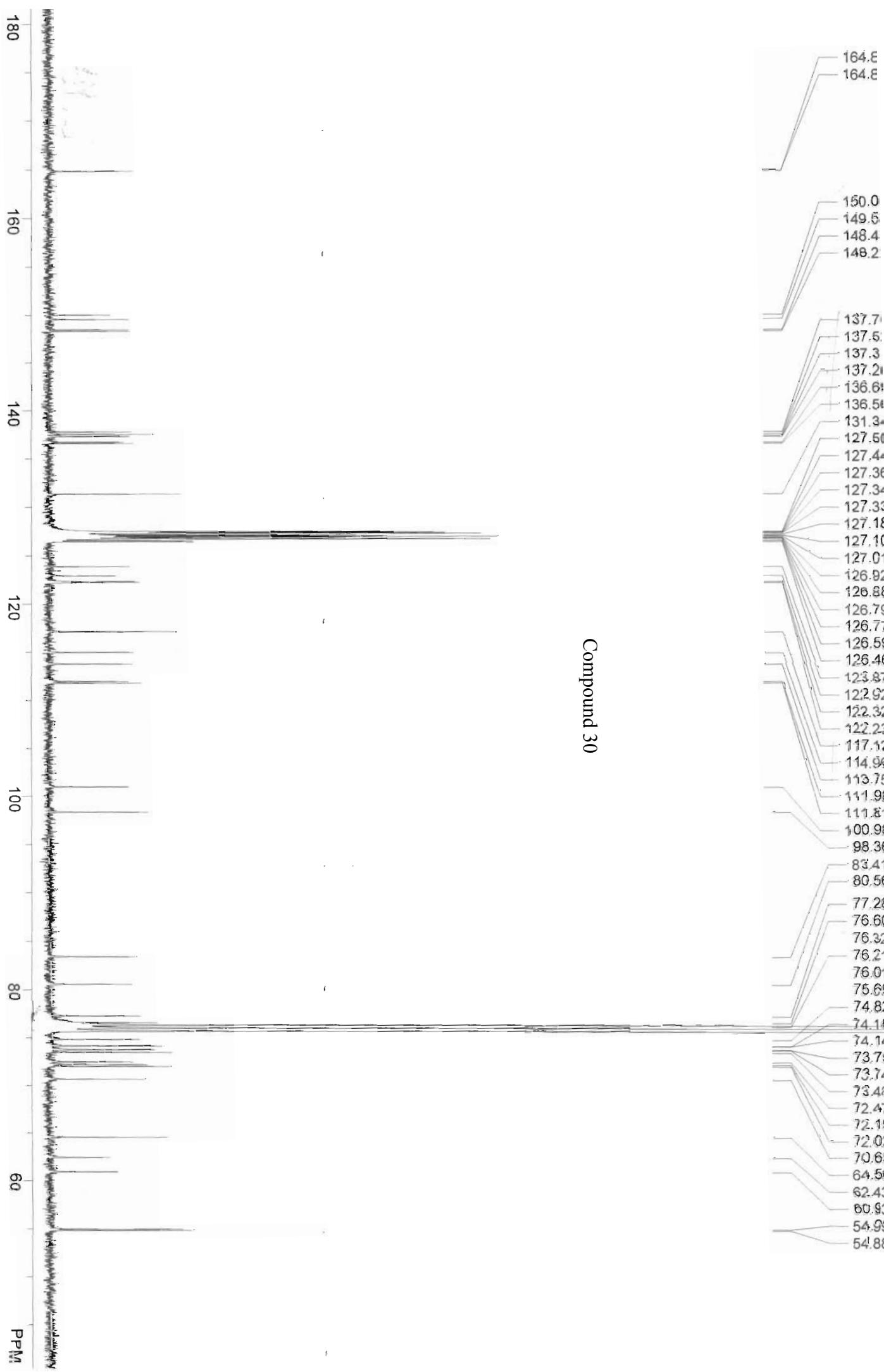


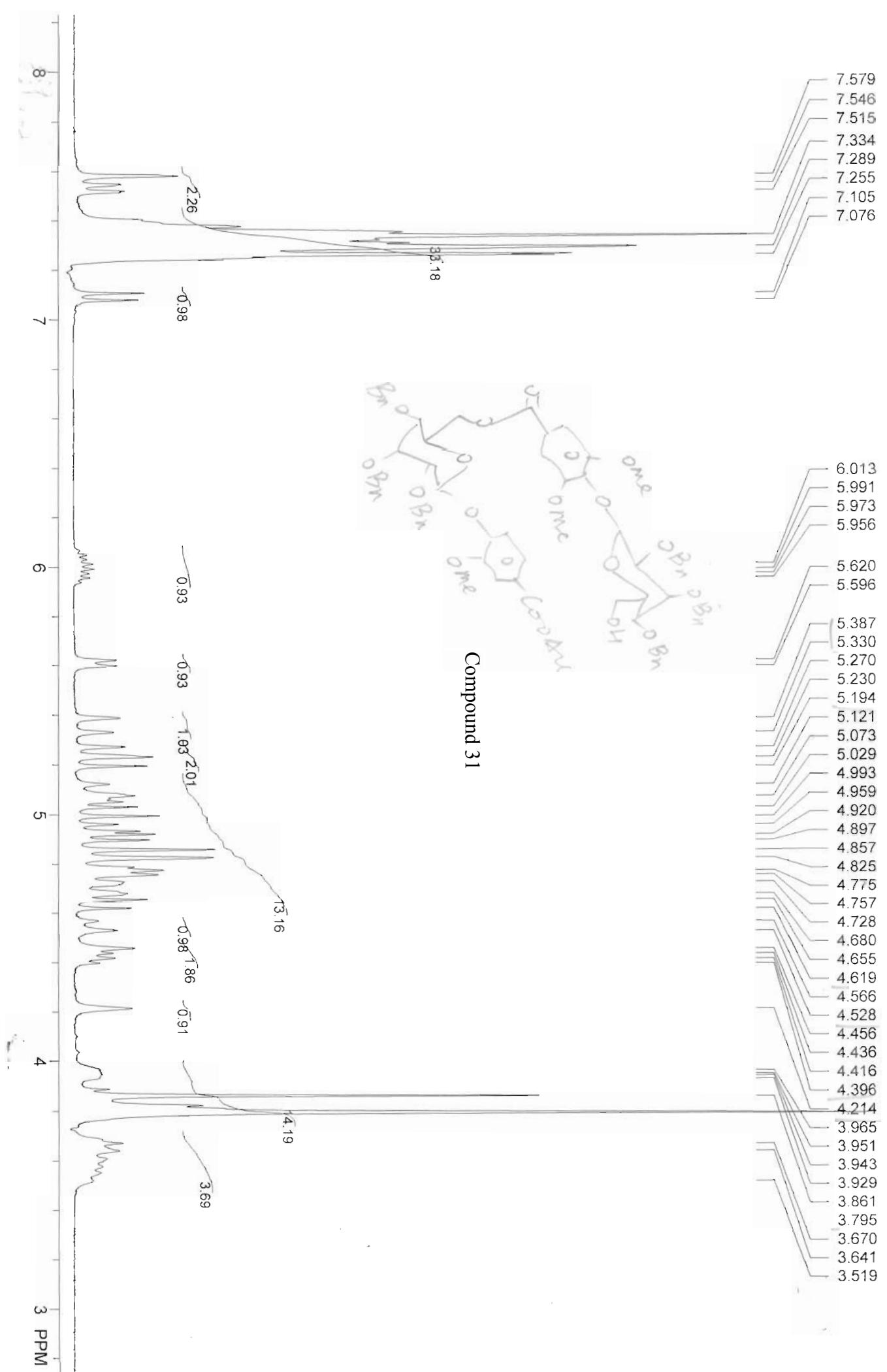


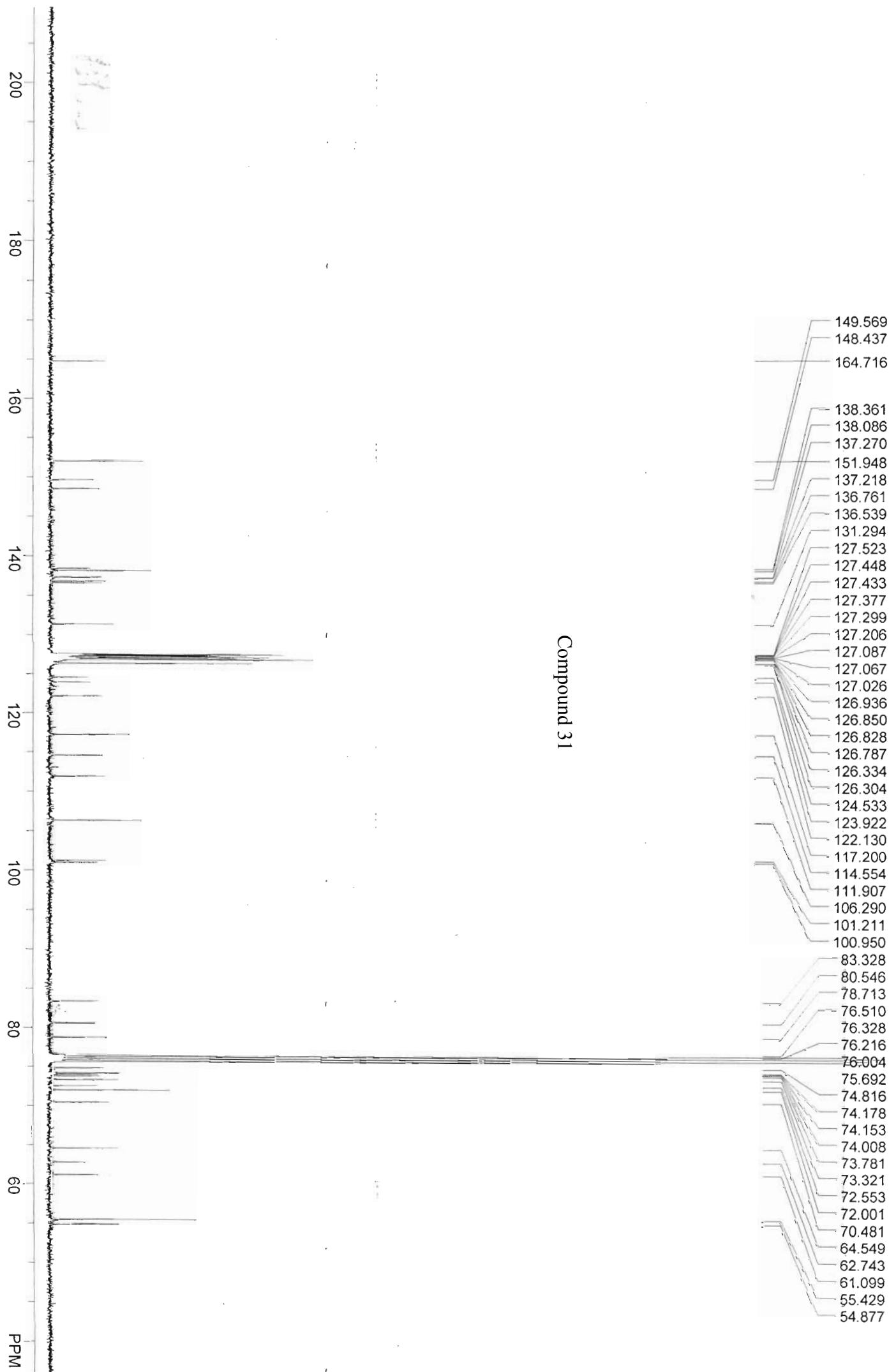


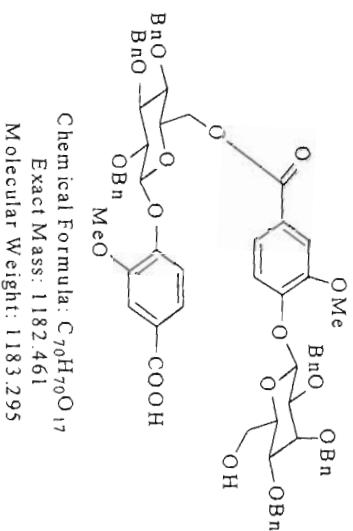
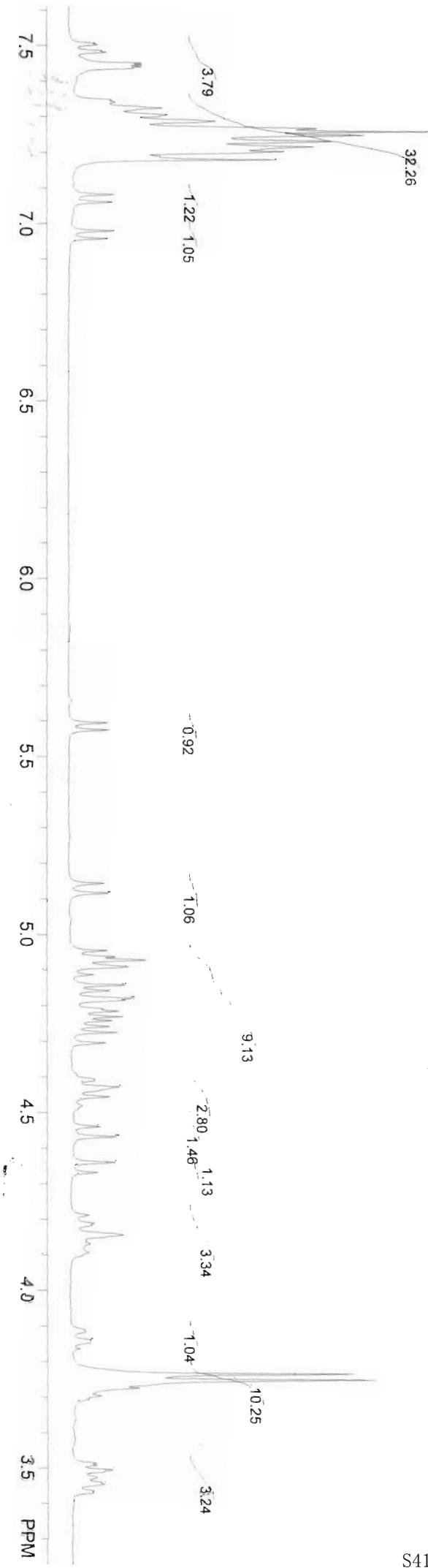




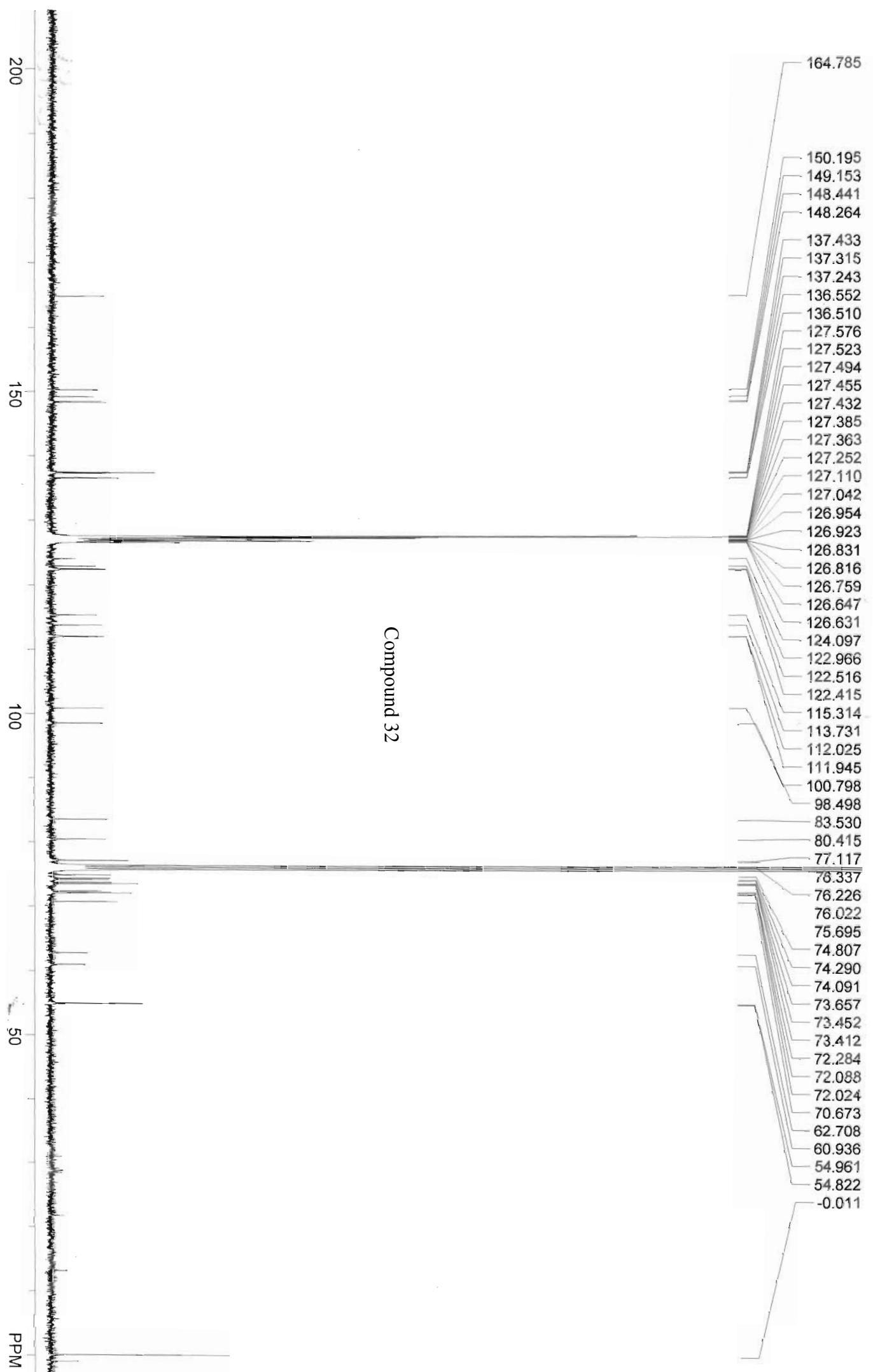


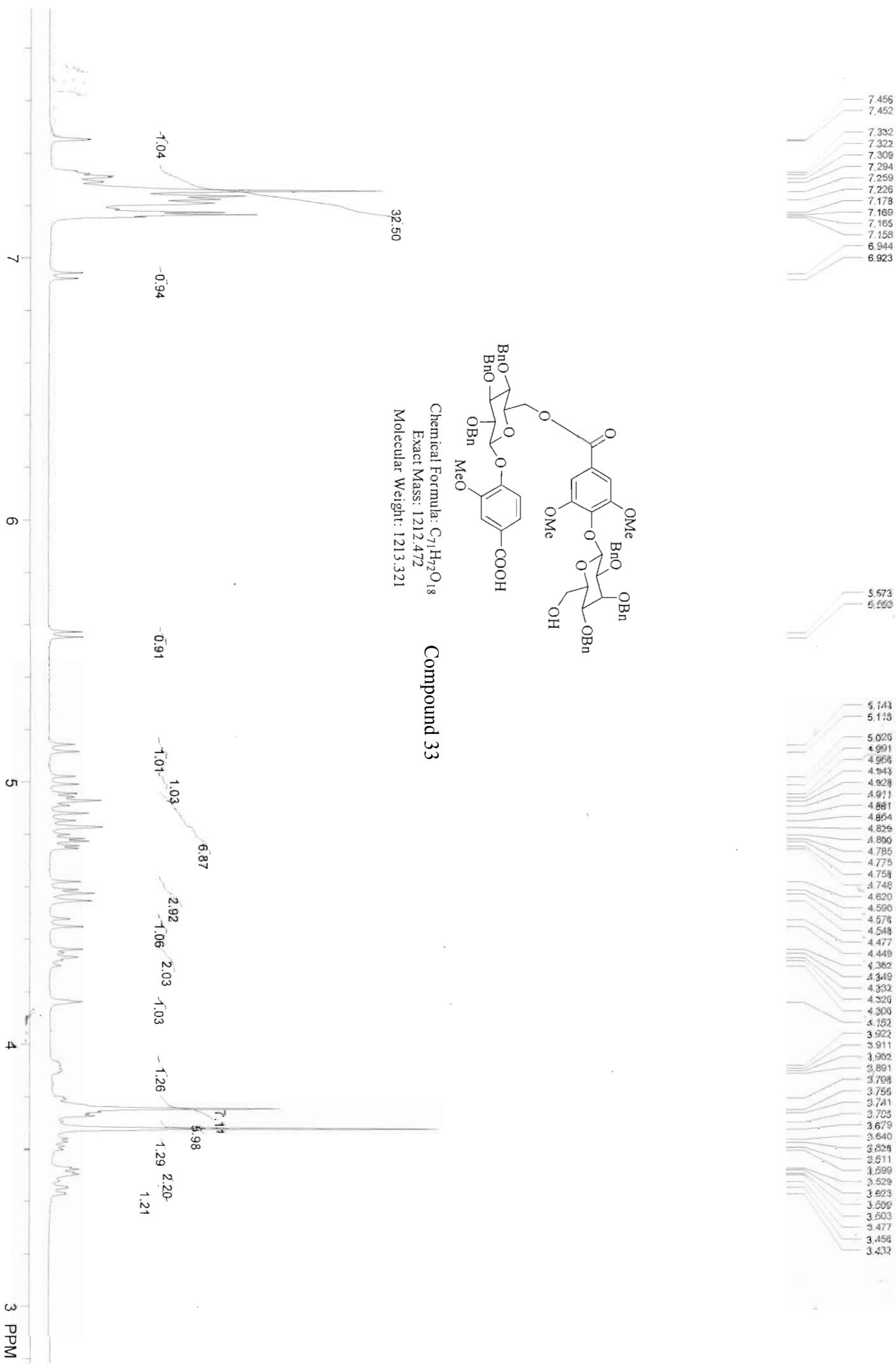


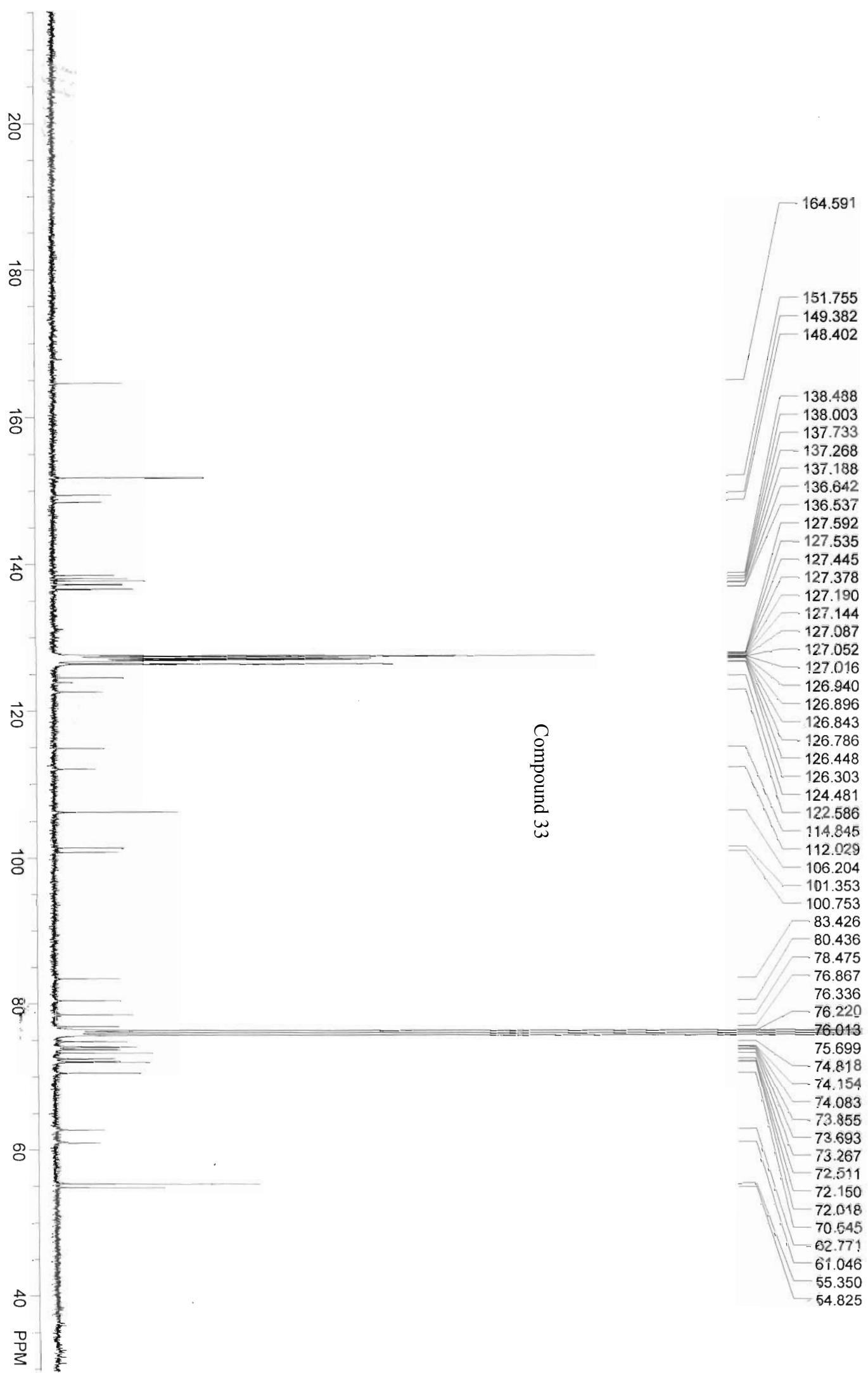


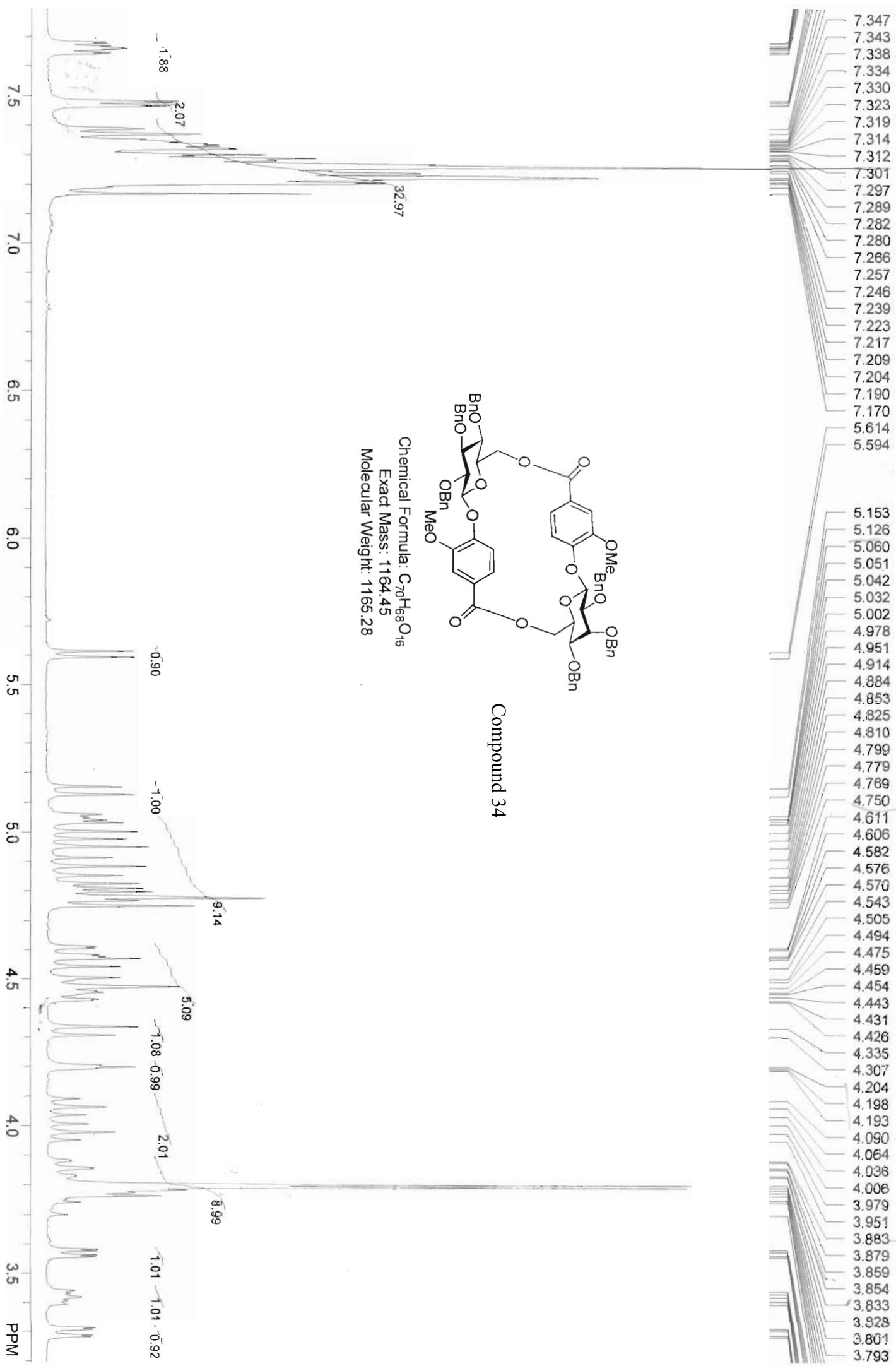


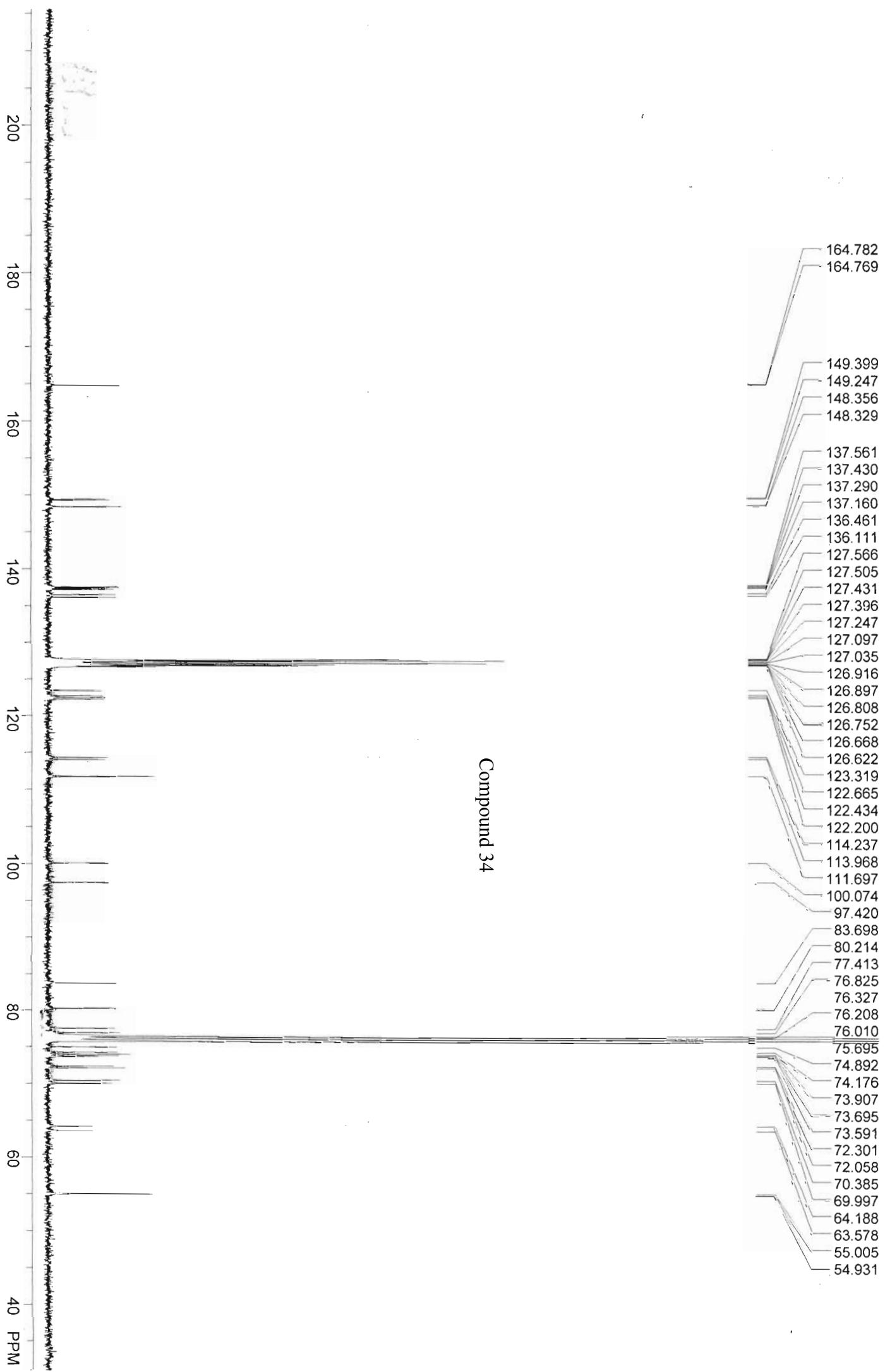
Chemical Formula: $C_{70}H_{70}O_{17}$
 Exact Mass: 1182.461
 Molecular Weight: 1183.295

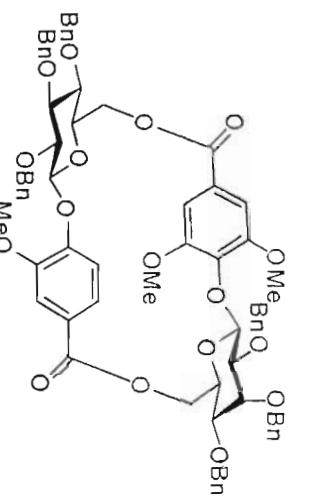
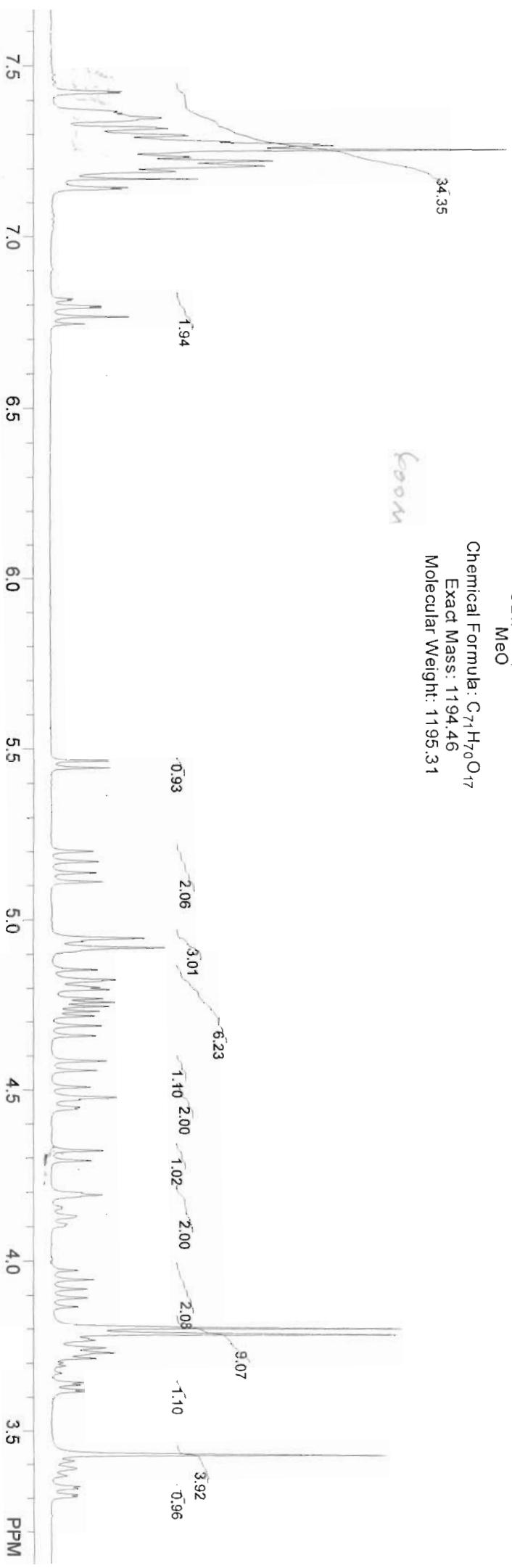






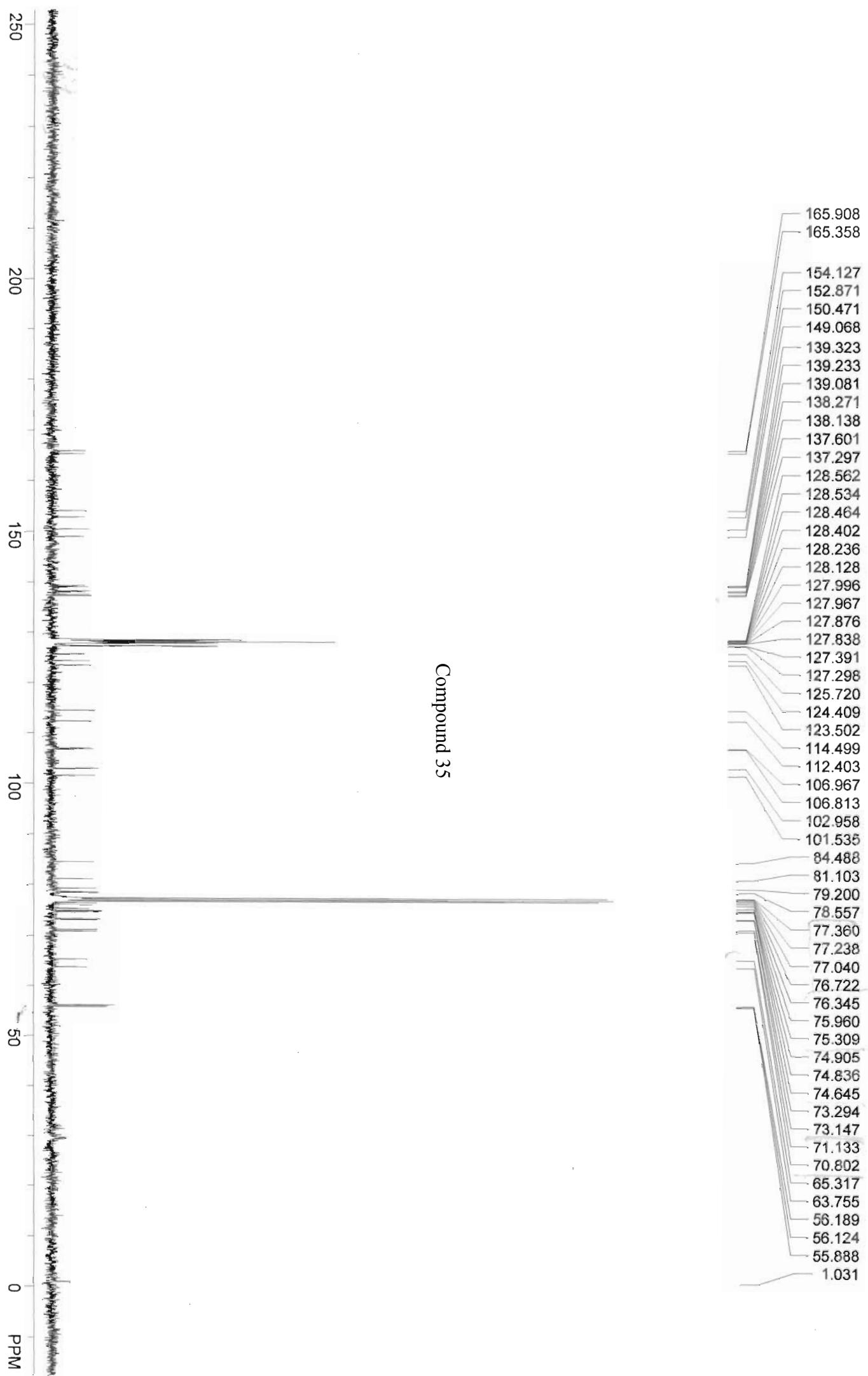


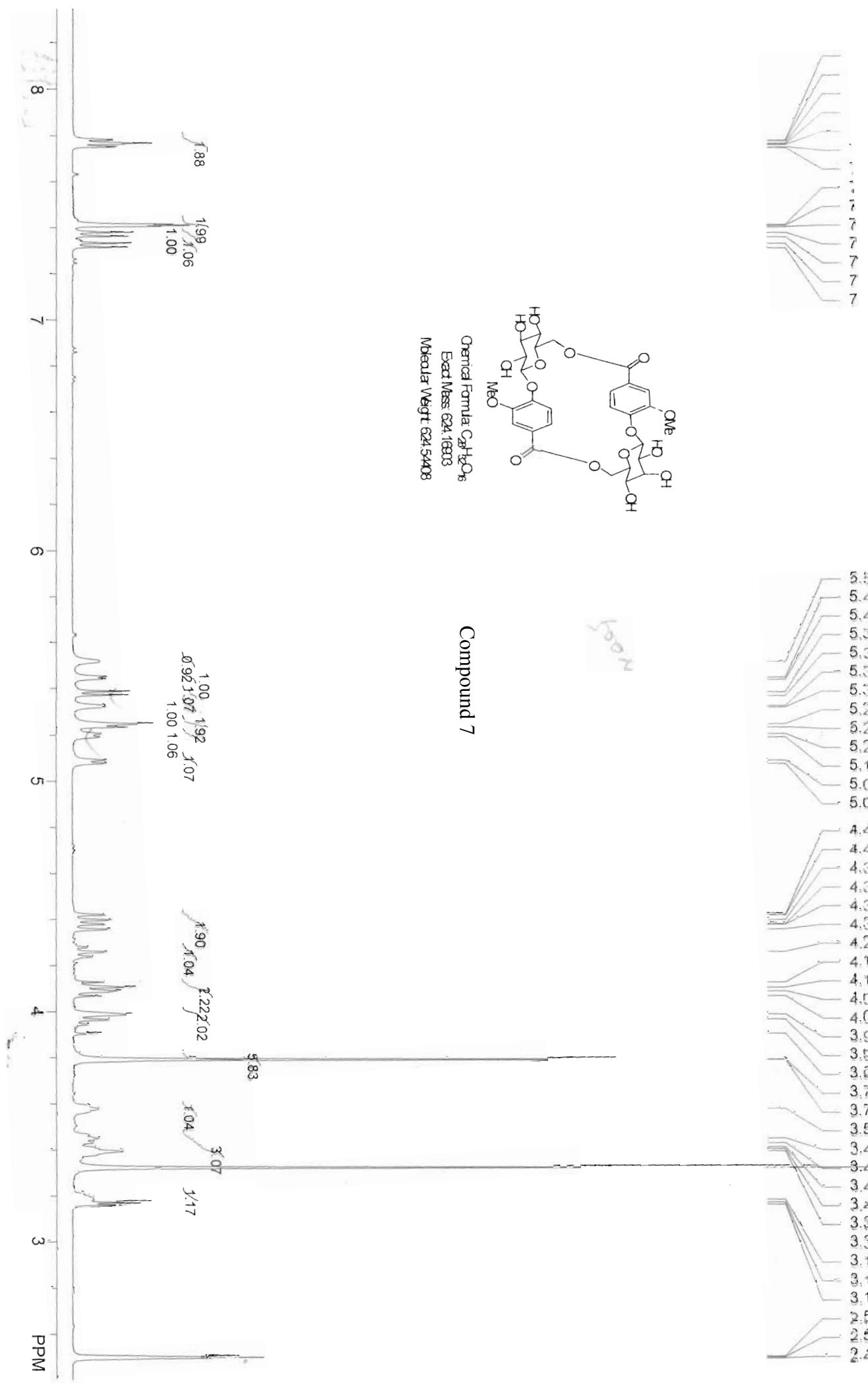


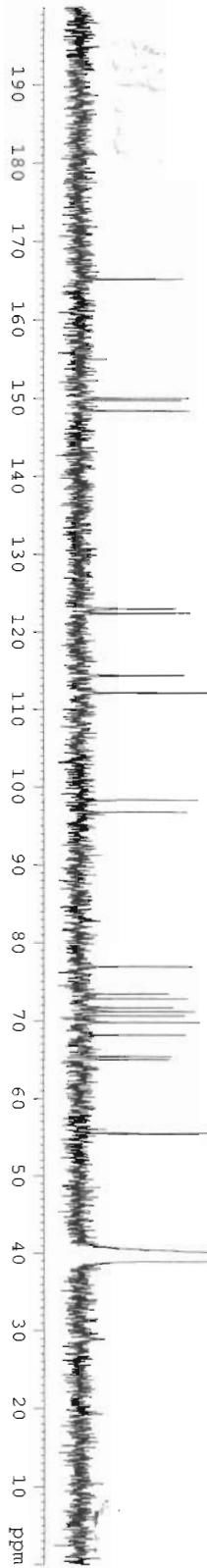


Compound 35

Chemical Formula: C₇₁H₇₀O₁₇
Exact Mass: 1194.46
Molecular Weight: 1195.31







Compound 7

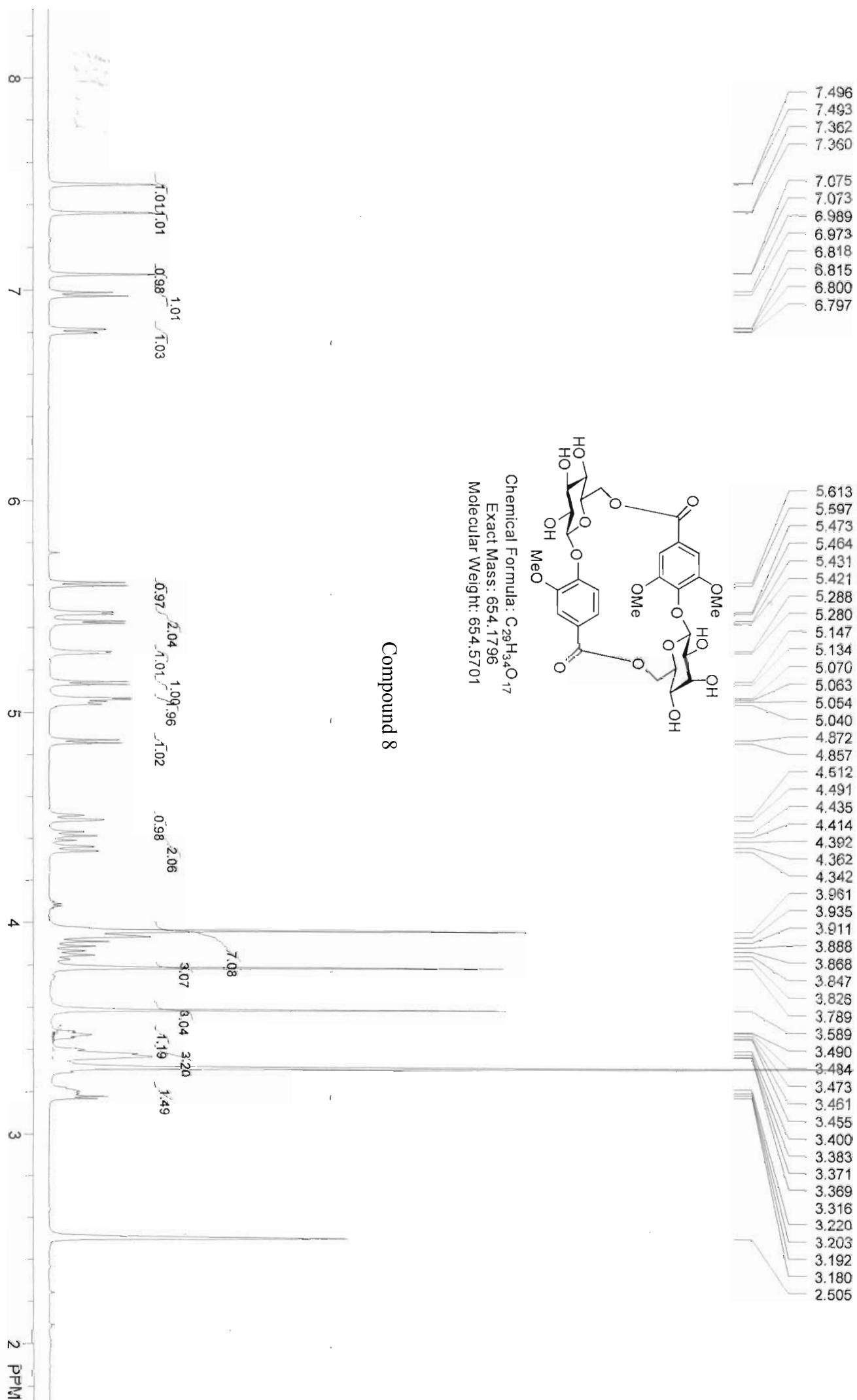
Current Data Parameters
 NAME 2009574cc-ly1-tm
 EXPNO 1
 PROCN 1

F2 - Acquisition Parameters
 Date 20100401
 Time 8.39
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1200
 DS 4
 SWH 43859.648 Hz
 FIDRES 0.669245 Hz
 AQ 0.7471718 sec
 RG 18390.4
 DW 11.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TDO 1

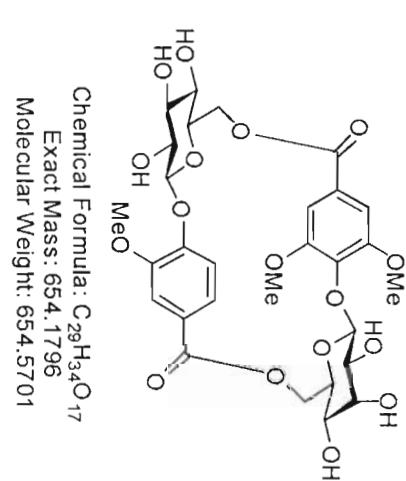
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PLL -4.00 dB
 SFO1 125.7703249 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL12 15.00 dB
 PL13 13.36 dB
 PL2 -4.00 dB
 SFO2 500.1320005 MHz

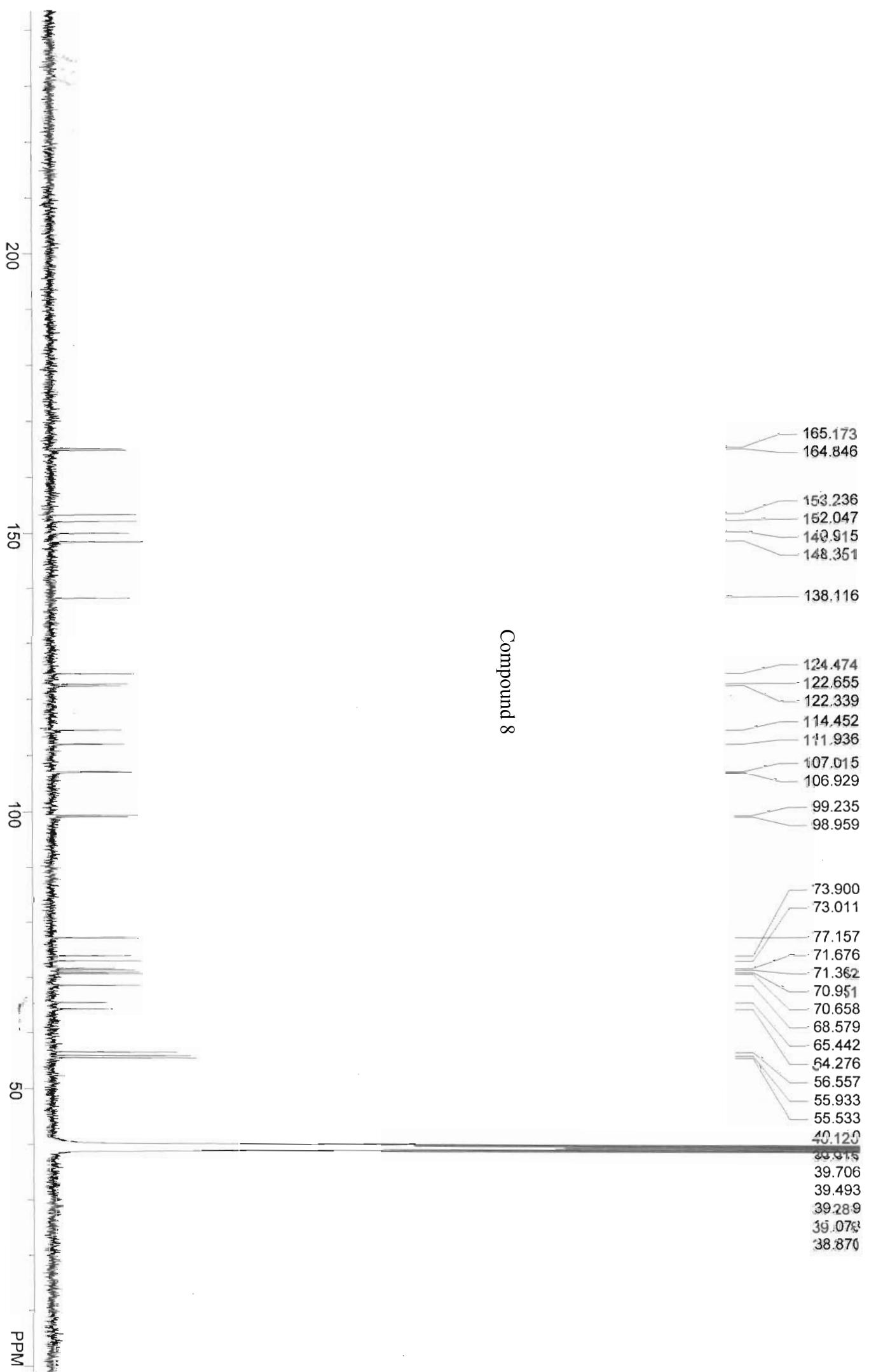
F2 - Processing parameters
 SI 32768
 SF 125.757835 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

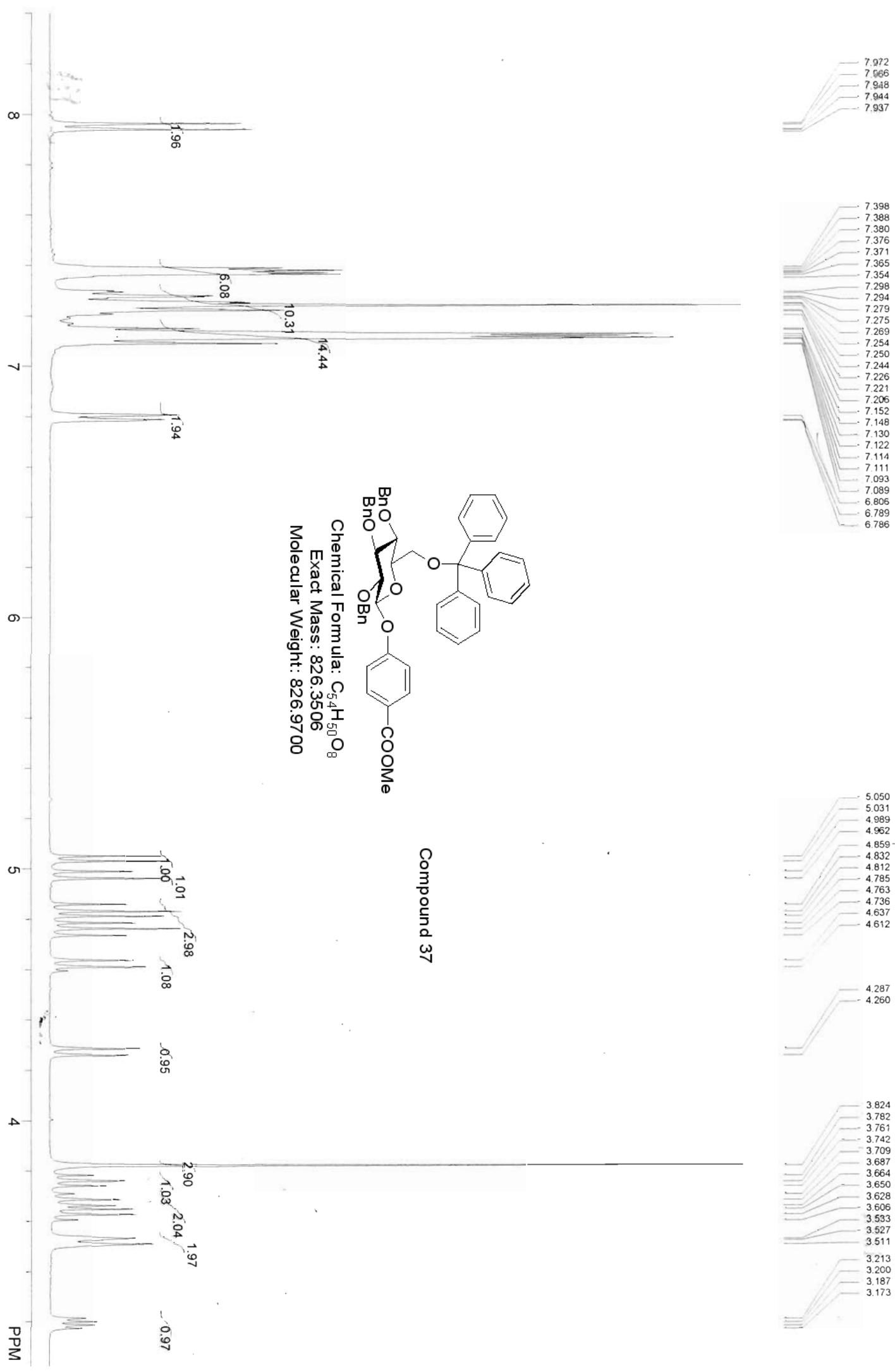


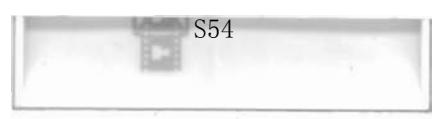
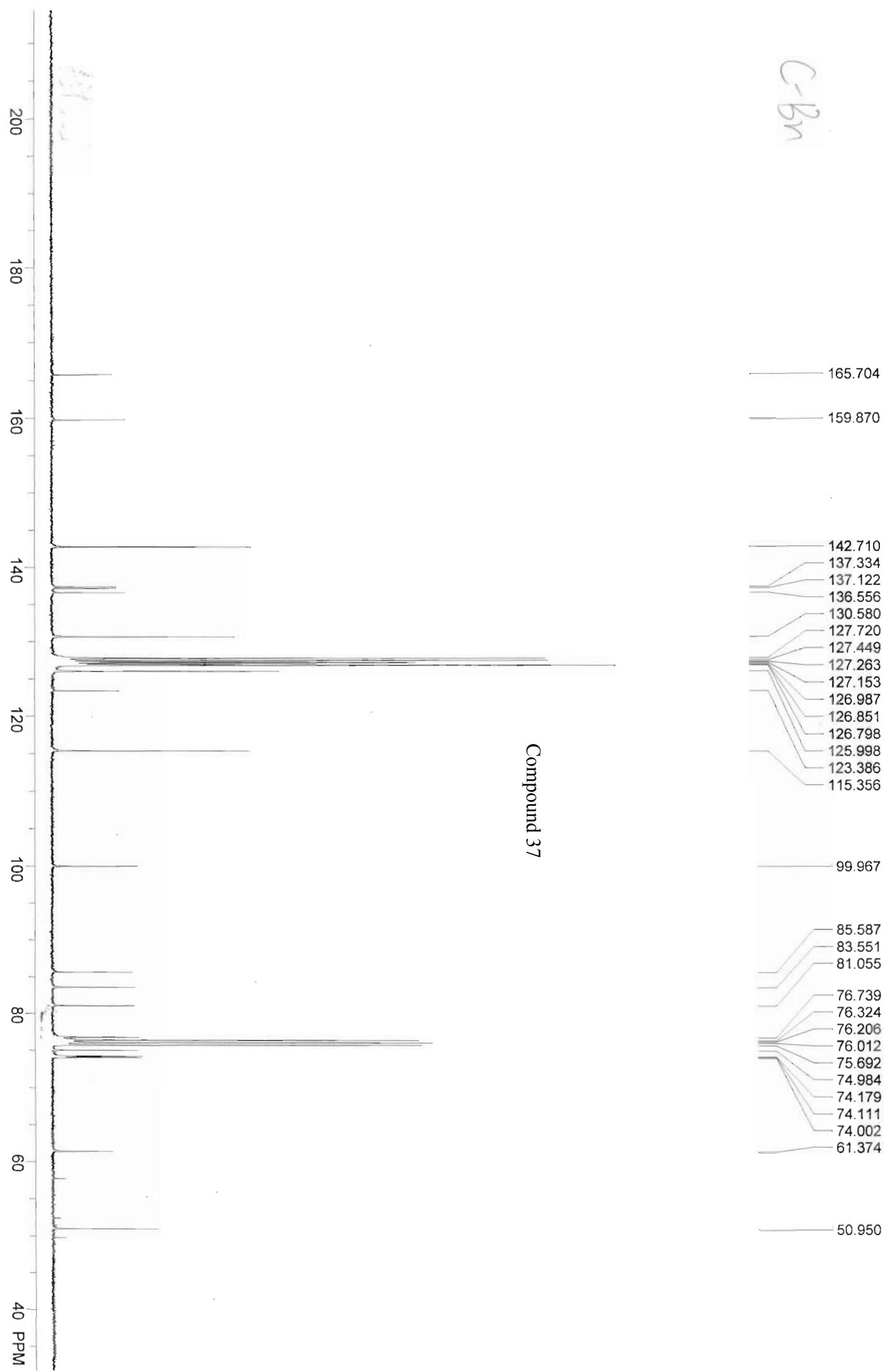
Compound 8

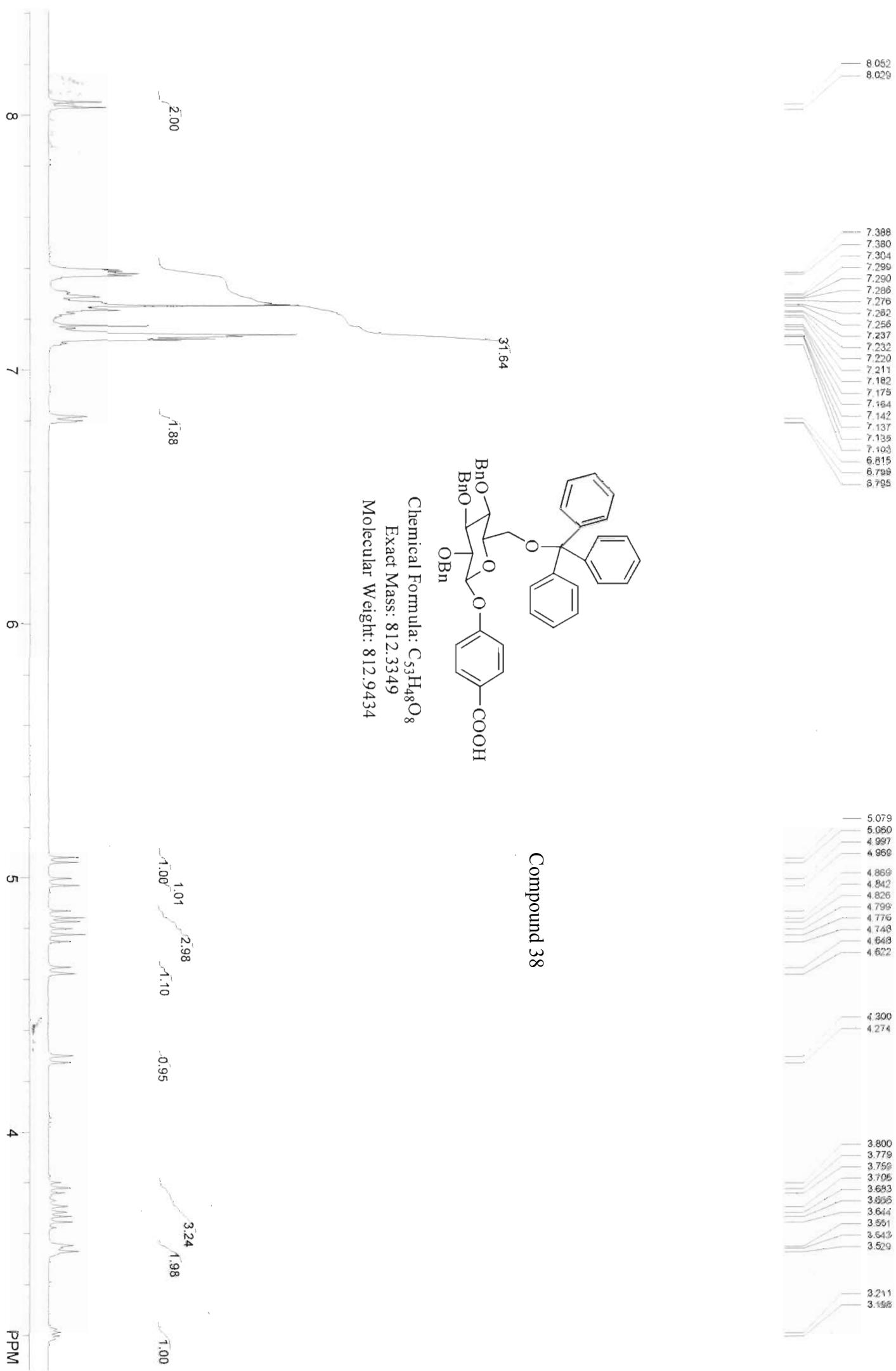


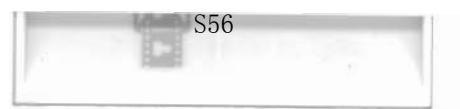
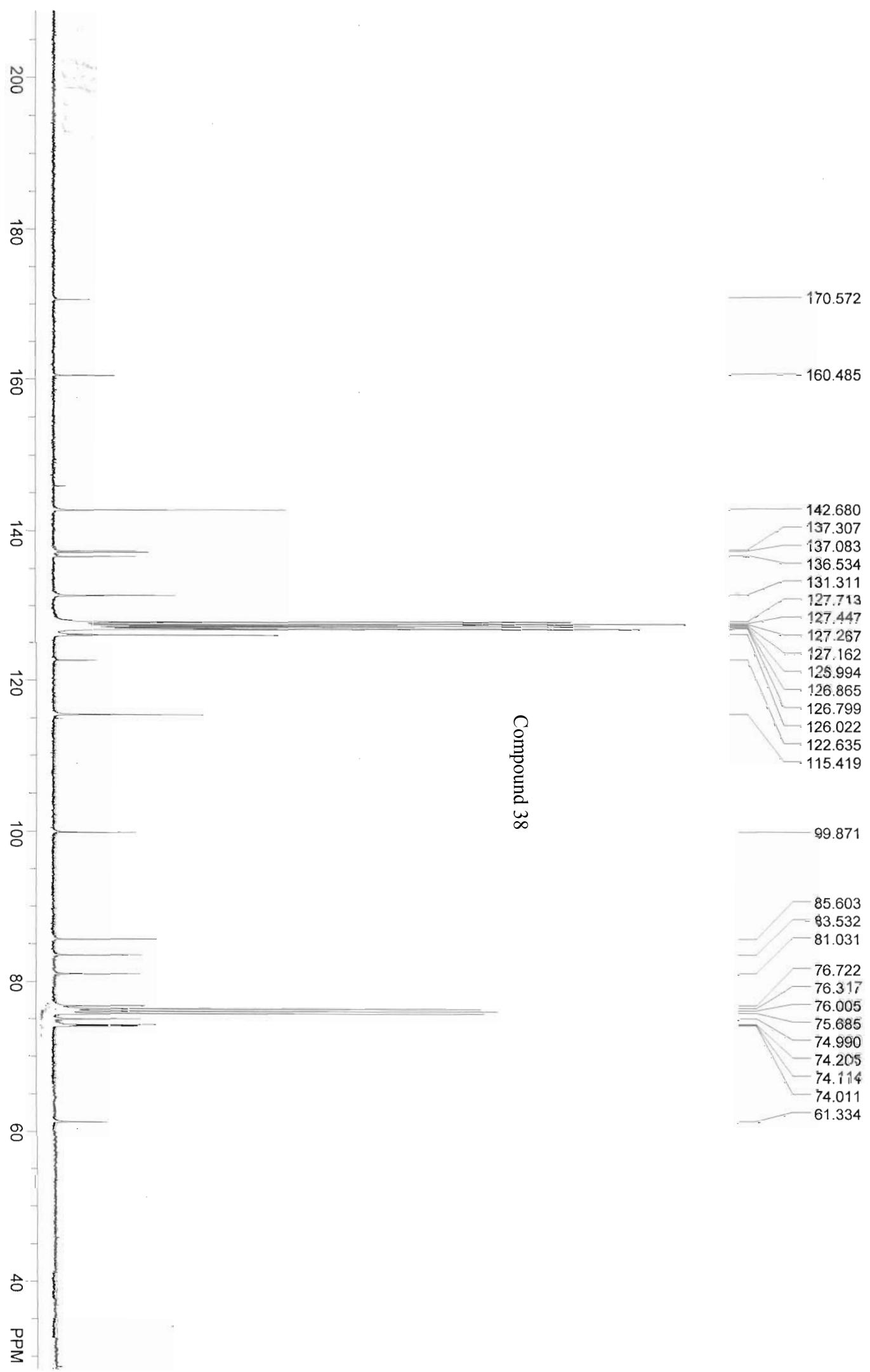
Chemical Formula: C₂₉H₃₄O₁₇
Exact Mass: 654.1796
Molecular Weight: 654.5701

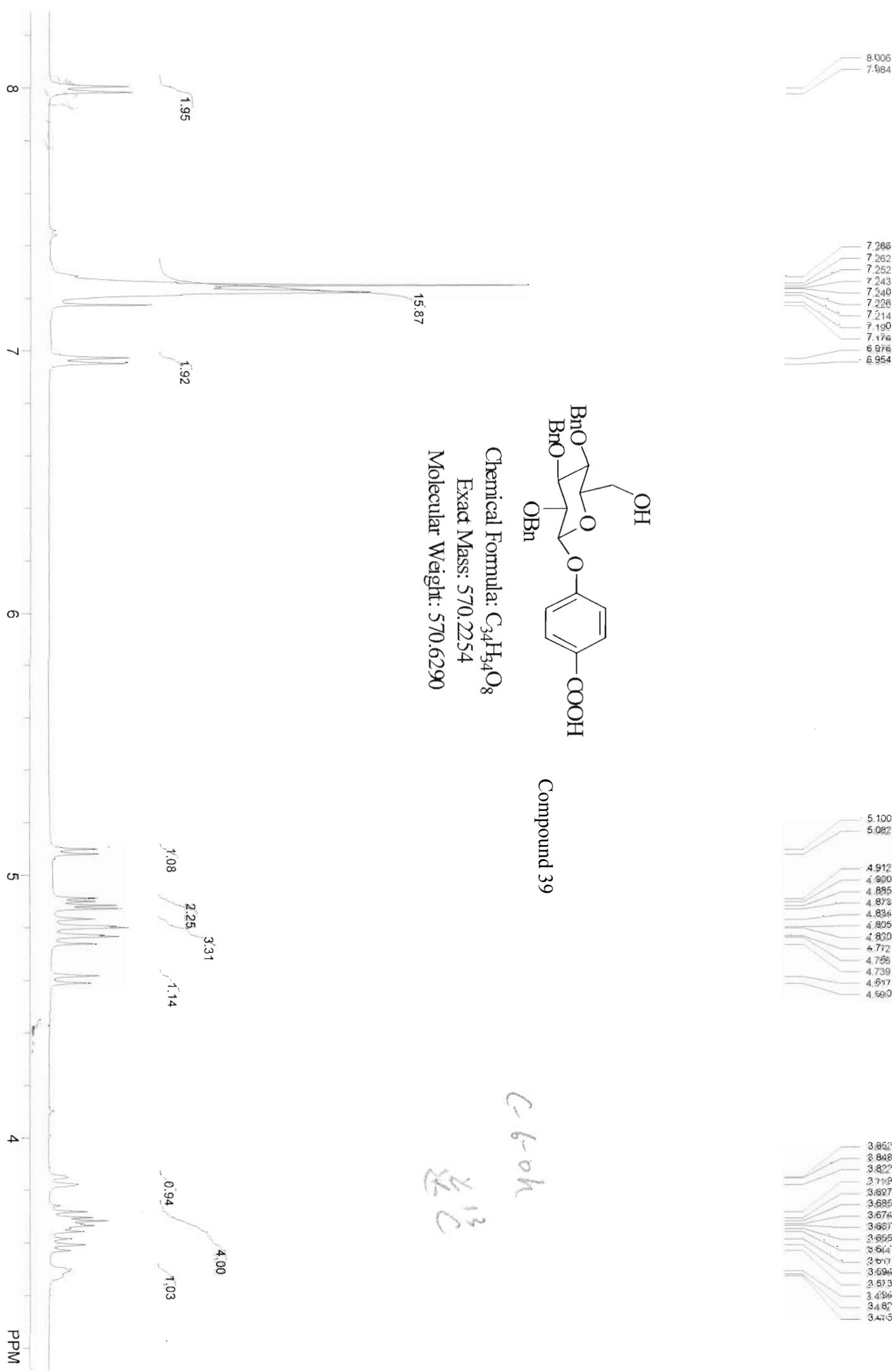


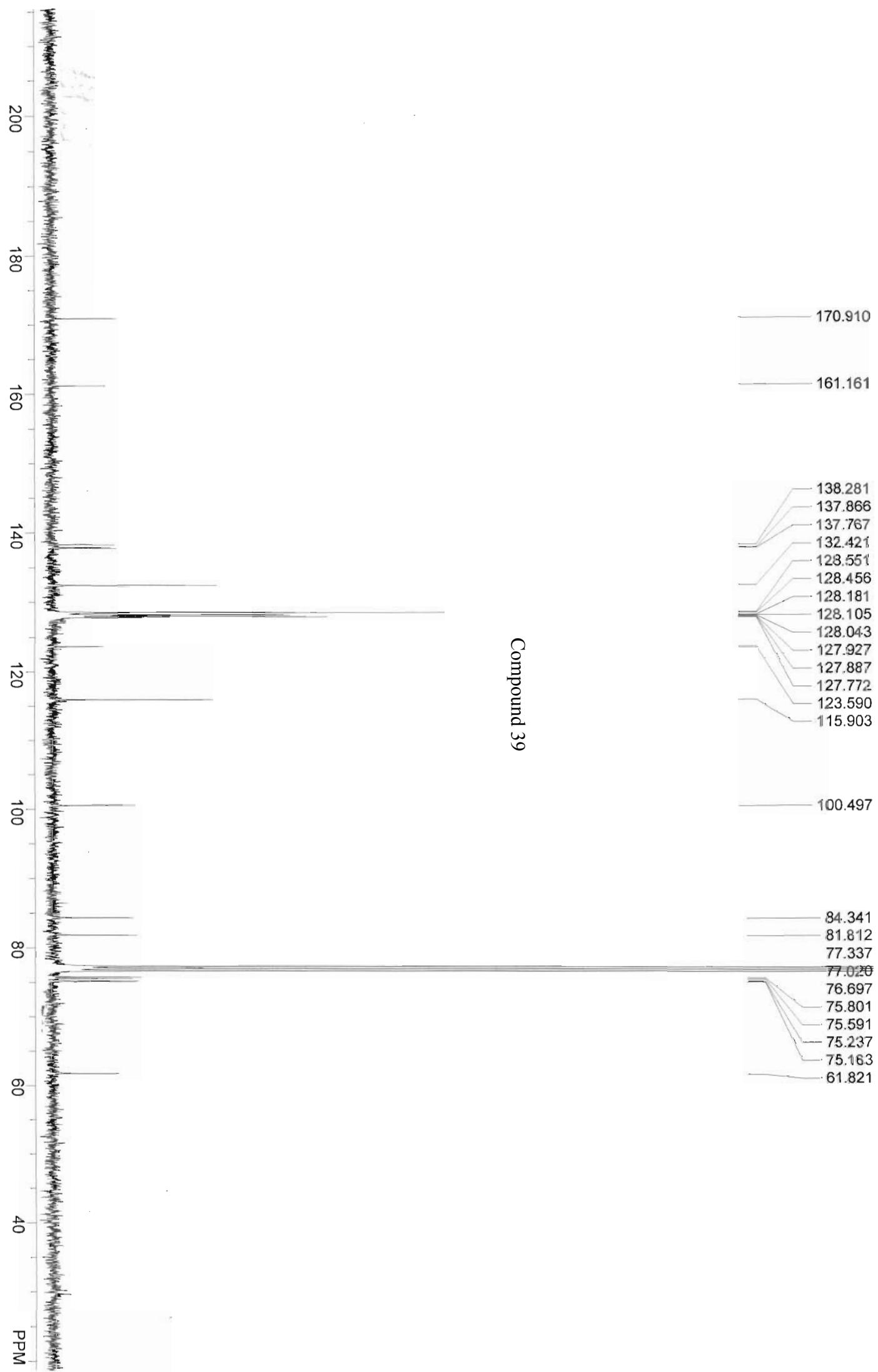


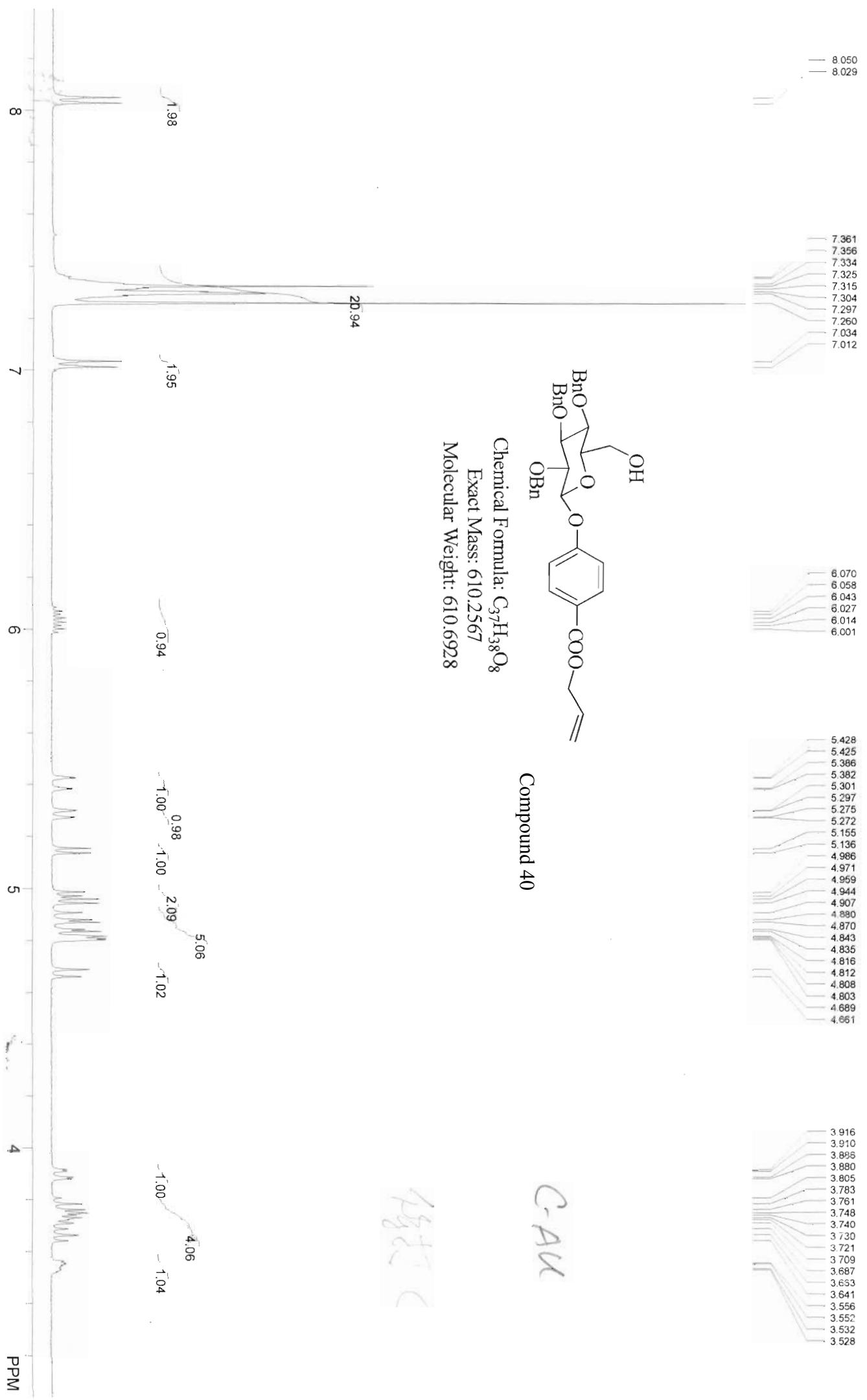












File: >sp

Pulse Sequence: s2pul

Solvent: cdc13

Temp. 25.0 C / 298.1 K

Operator: vmarl

VNMR-S-400 "400MR"

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.301 sec

Width 27173.9 Hz

2252 repetitions

OBSERVE C13, 100.4851907 MHz

DECOPPLER H1, 399.6242471 MHz

Power 43 dB

continuously on

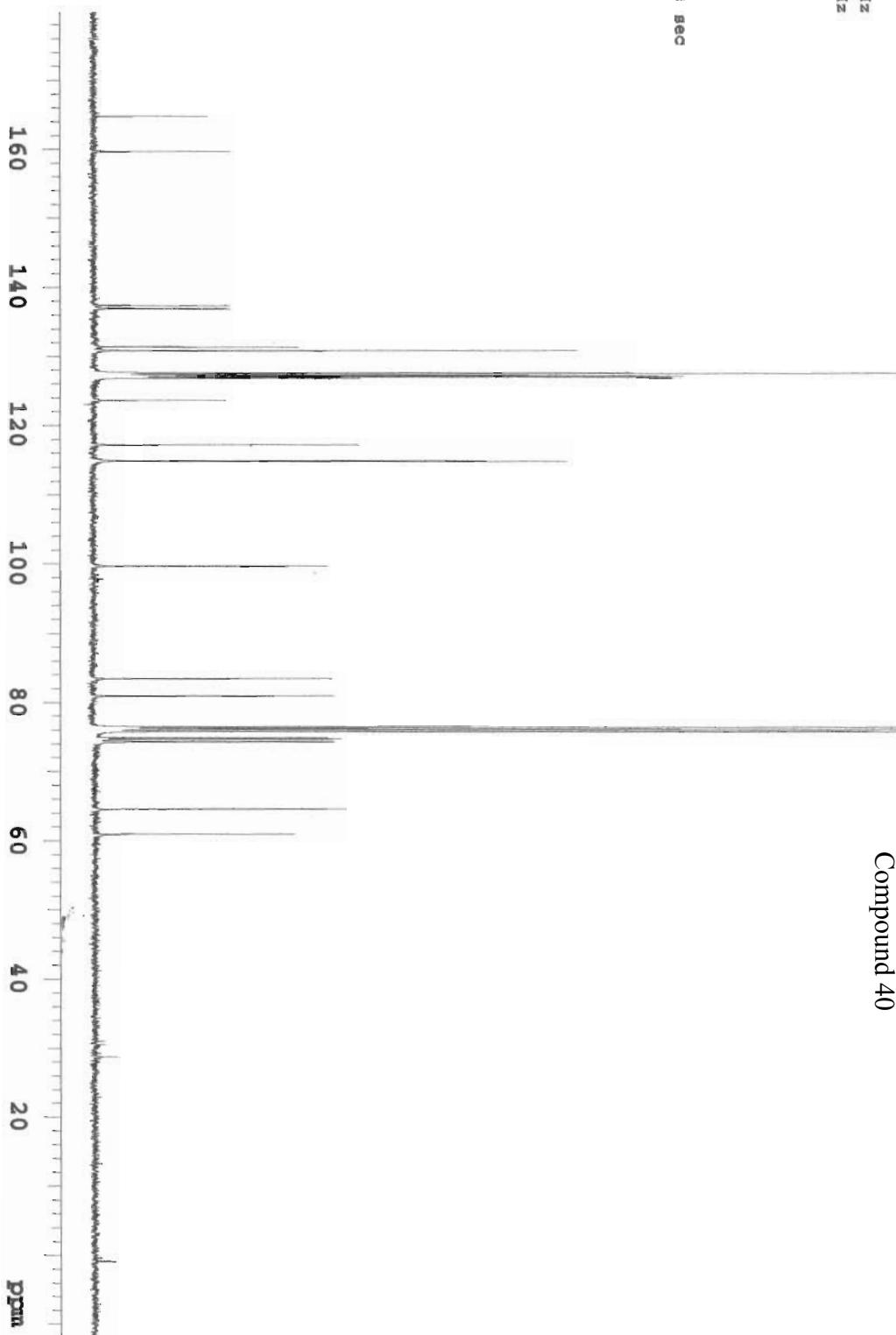
WALTZ-16 modulated

DATA PROCESSING

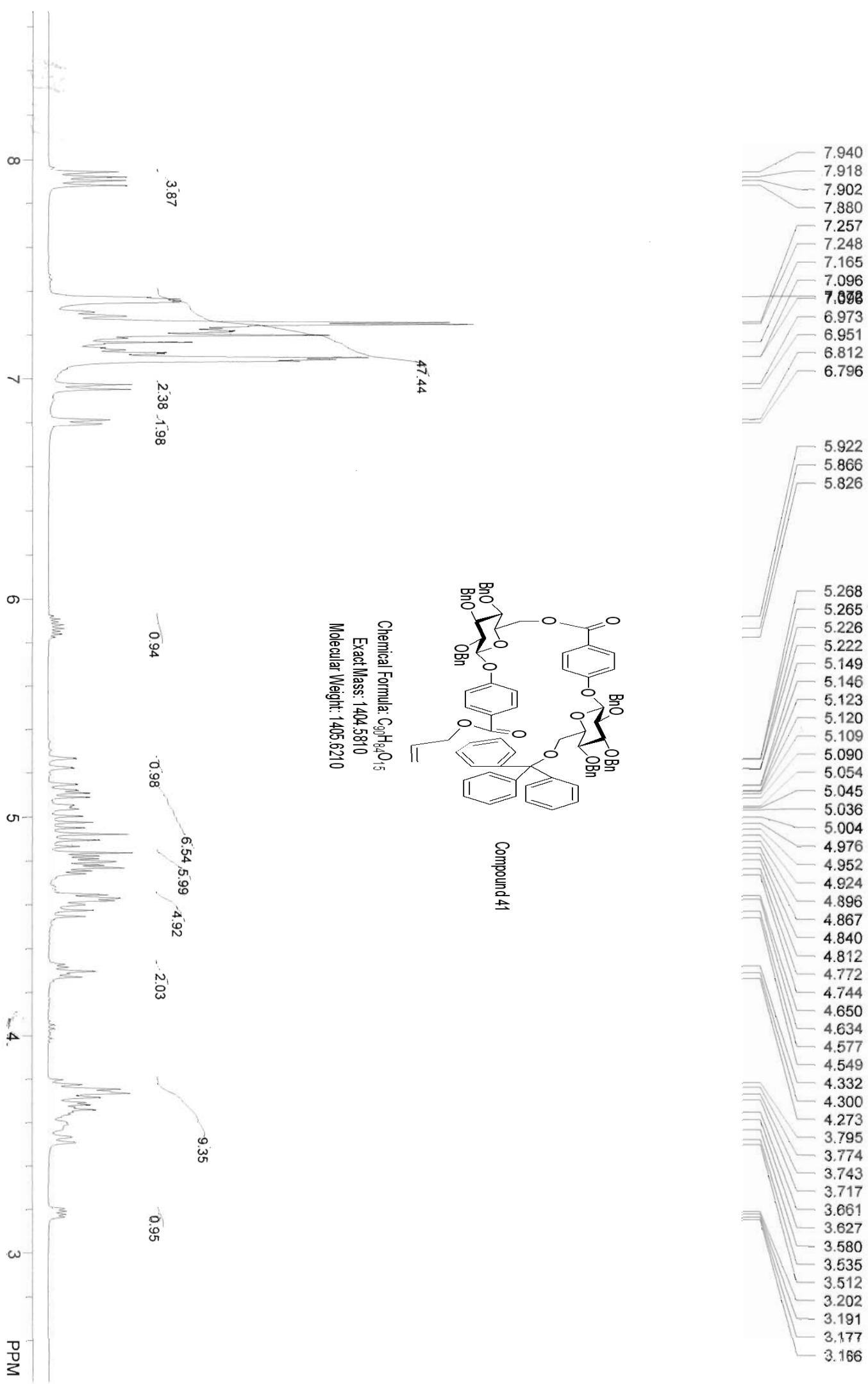
Line broadening 2.0 Hz

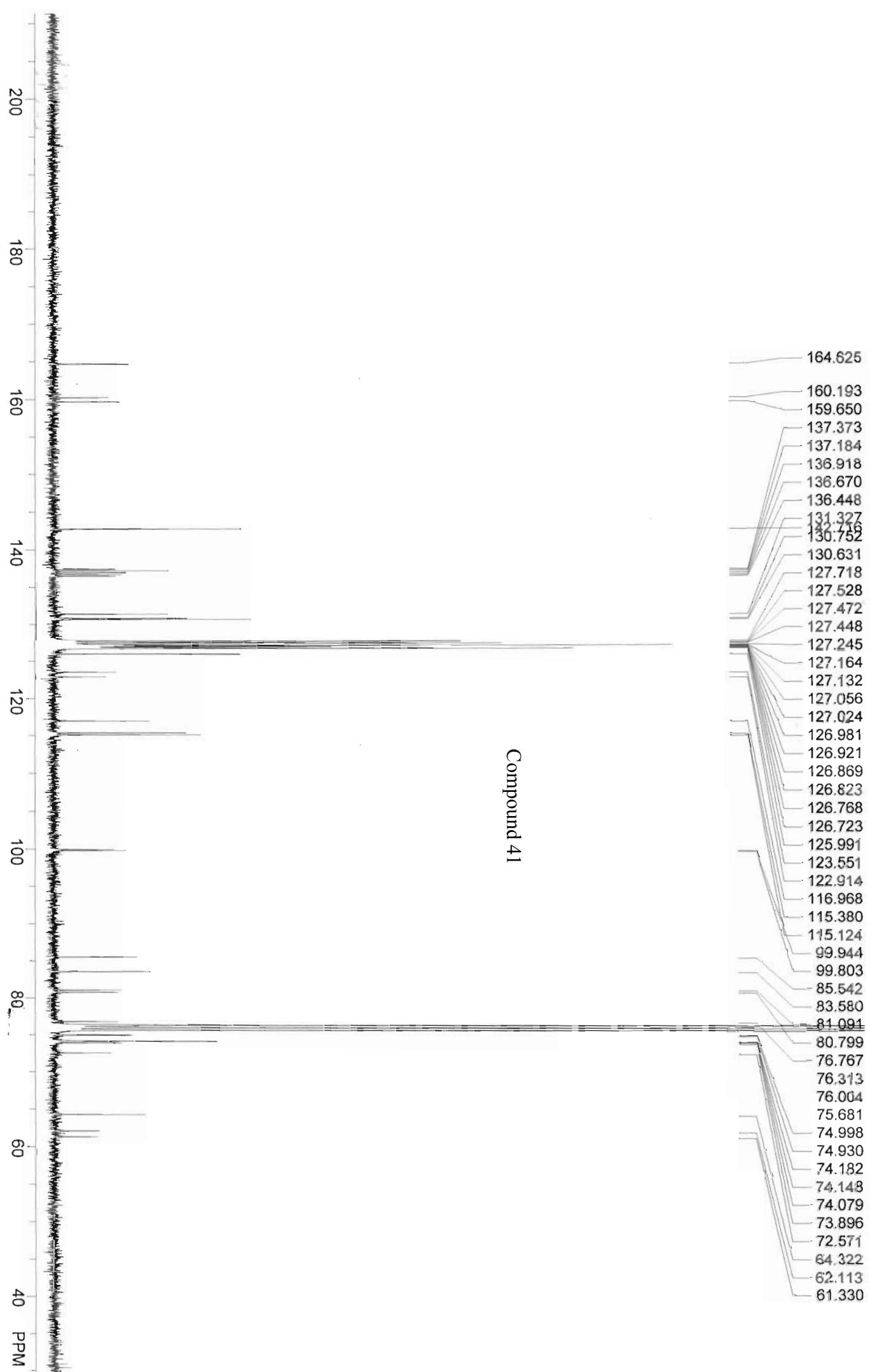
FT size 131072

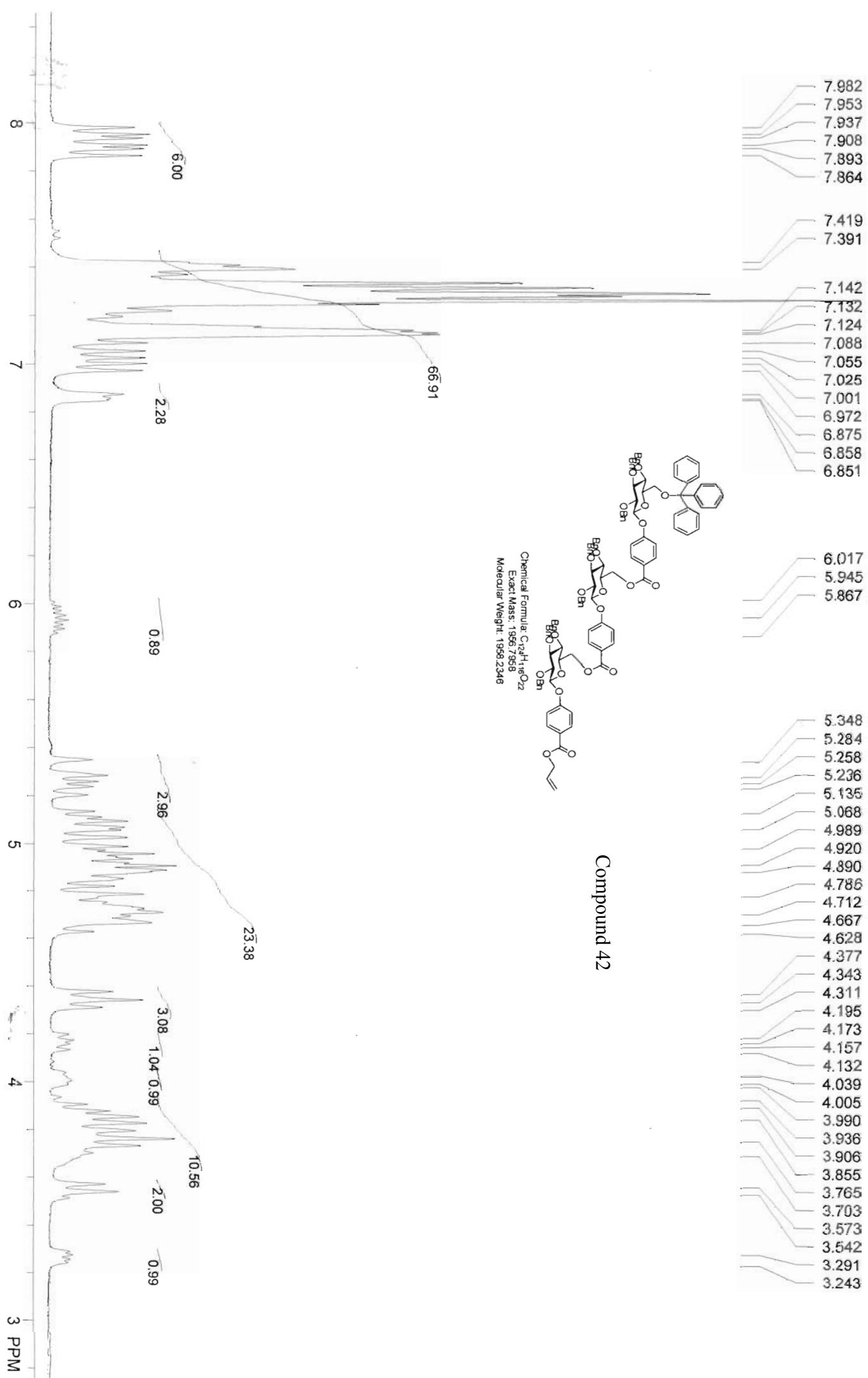
Total time 27 hr, 34 min, 42 sec

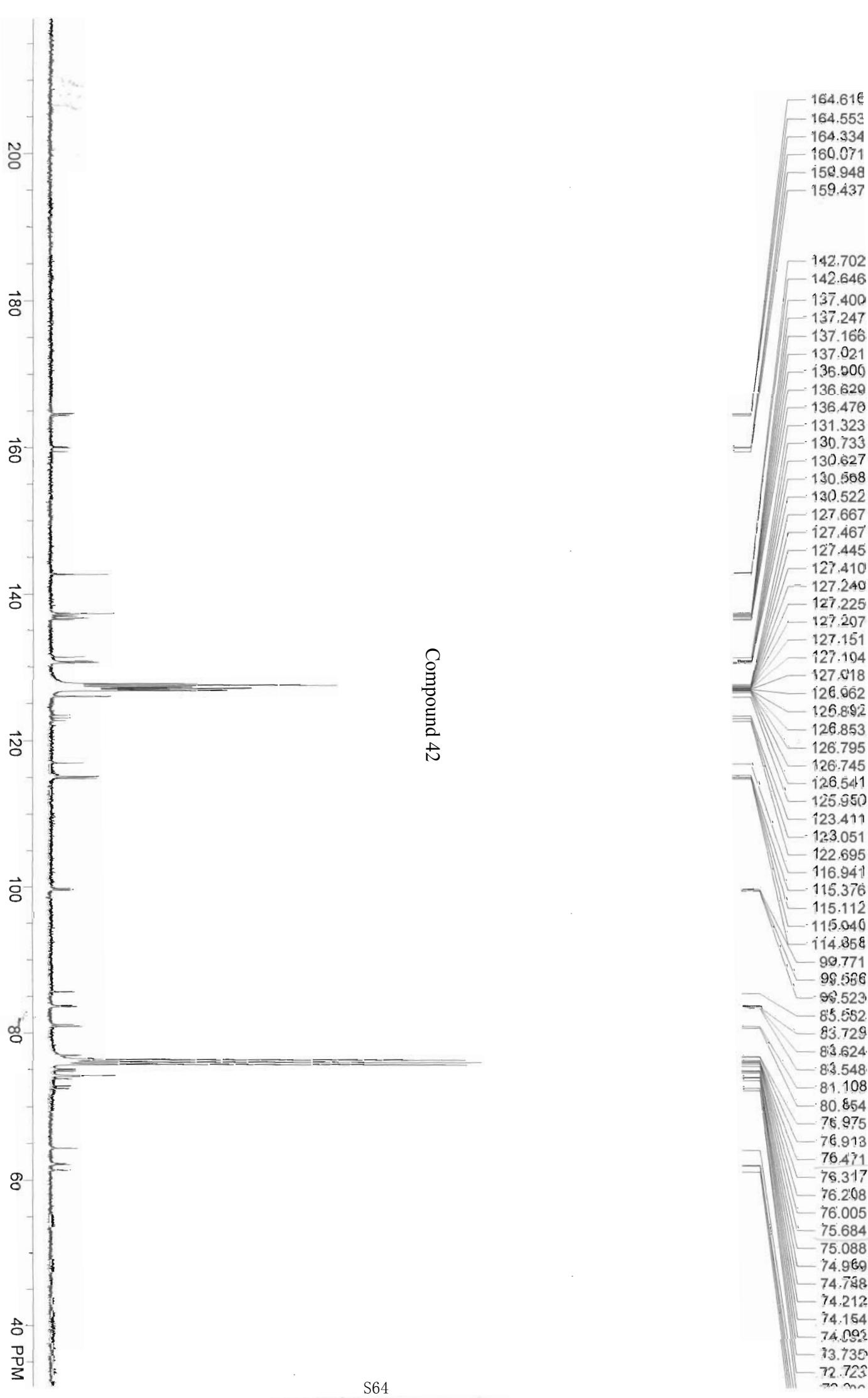


Compound 40

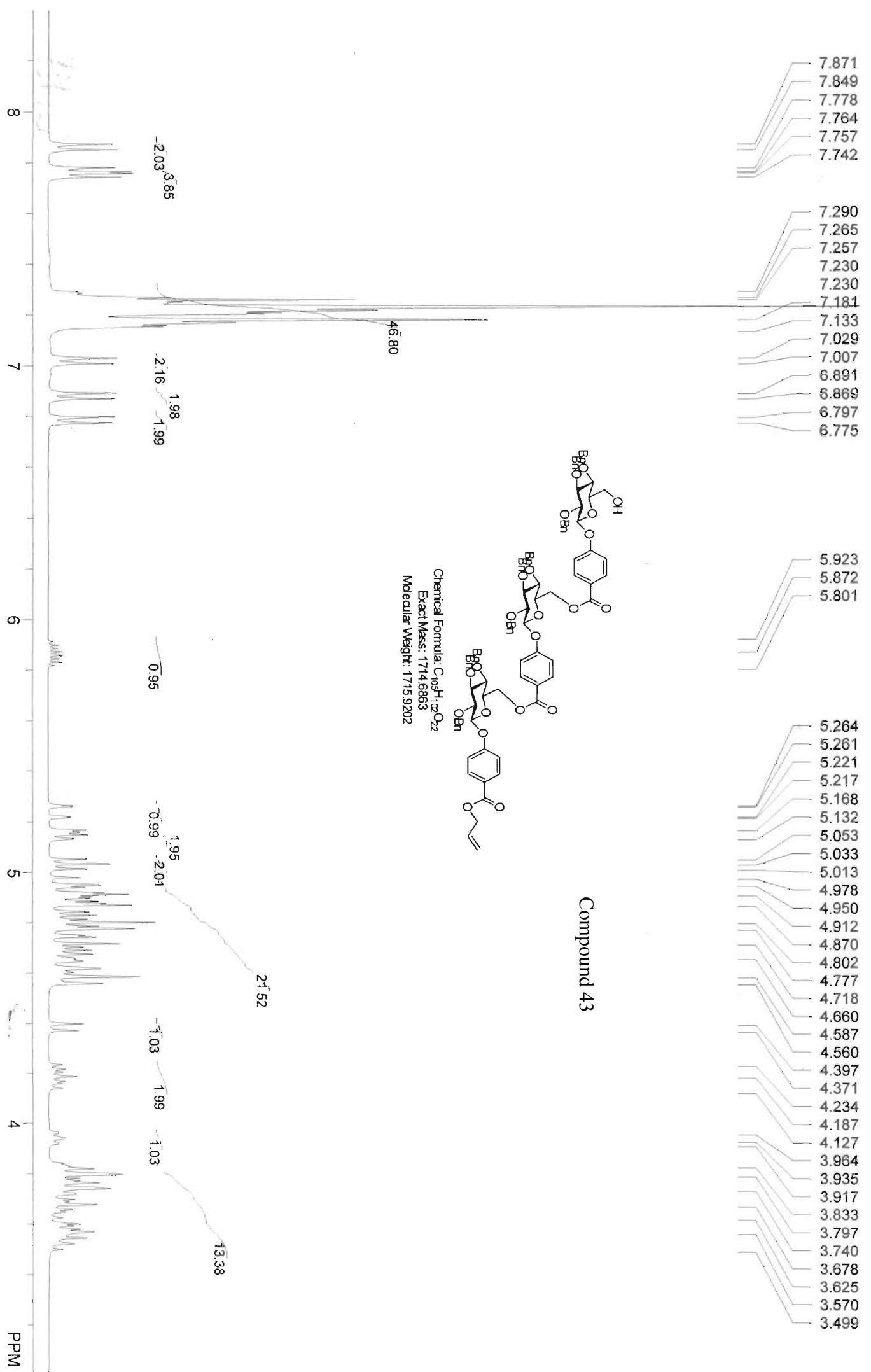




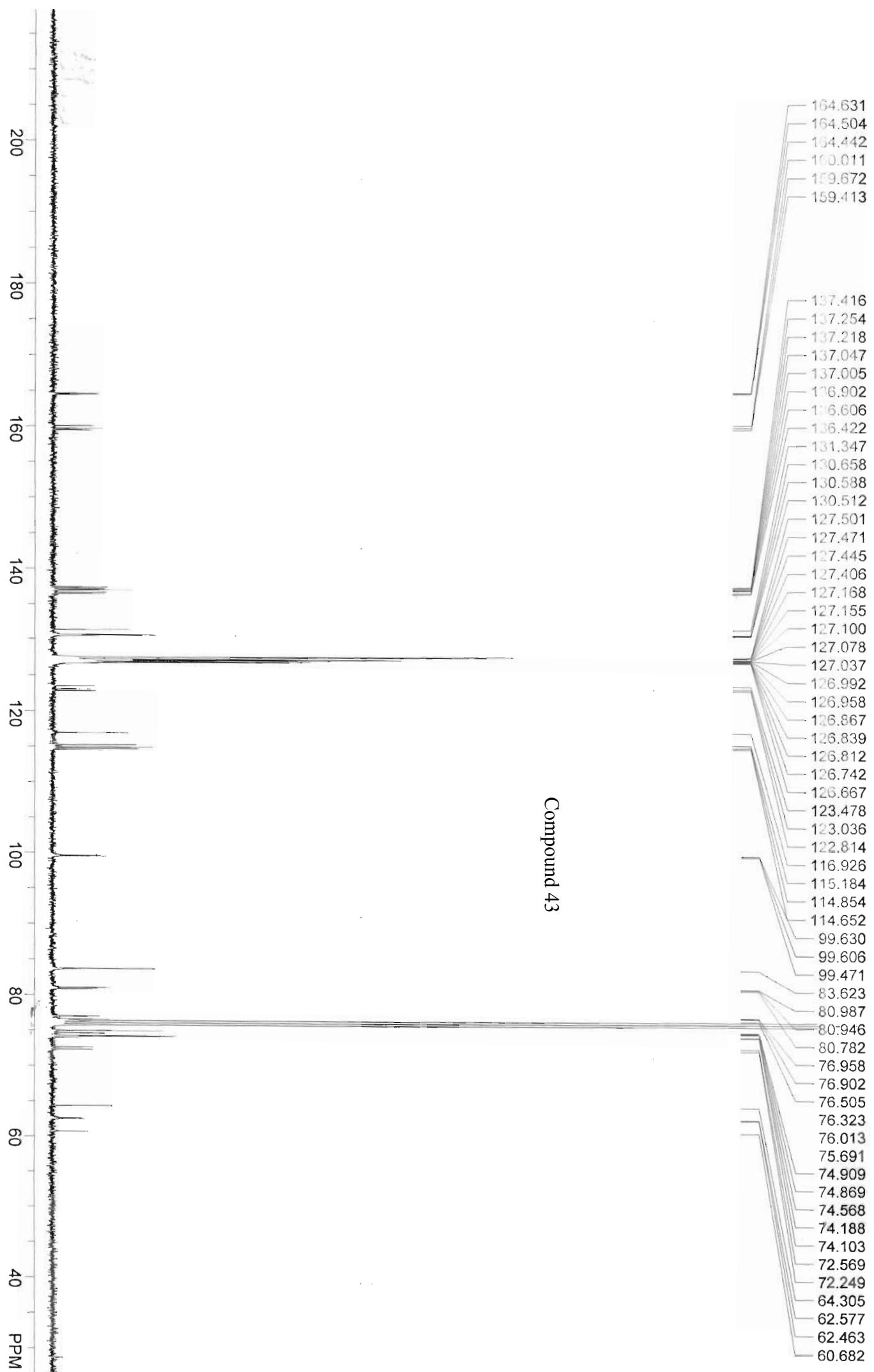




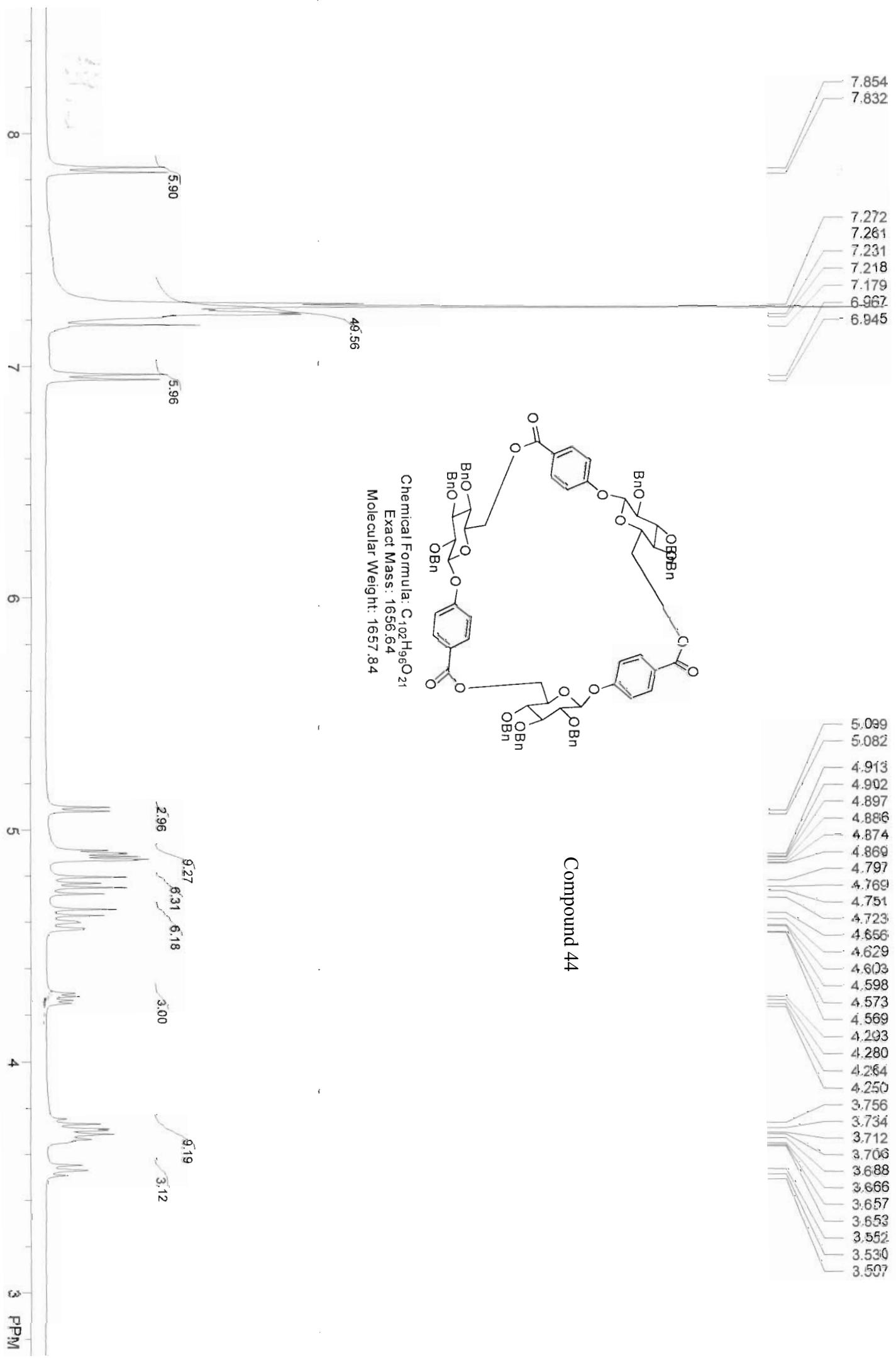
Compound 42

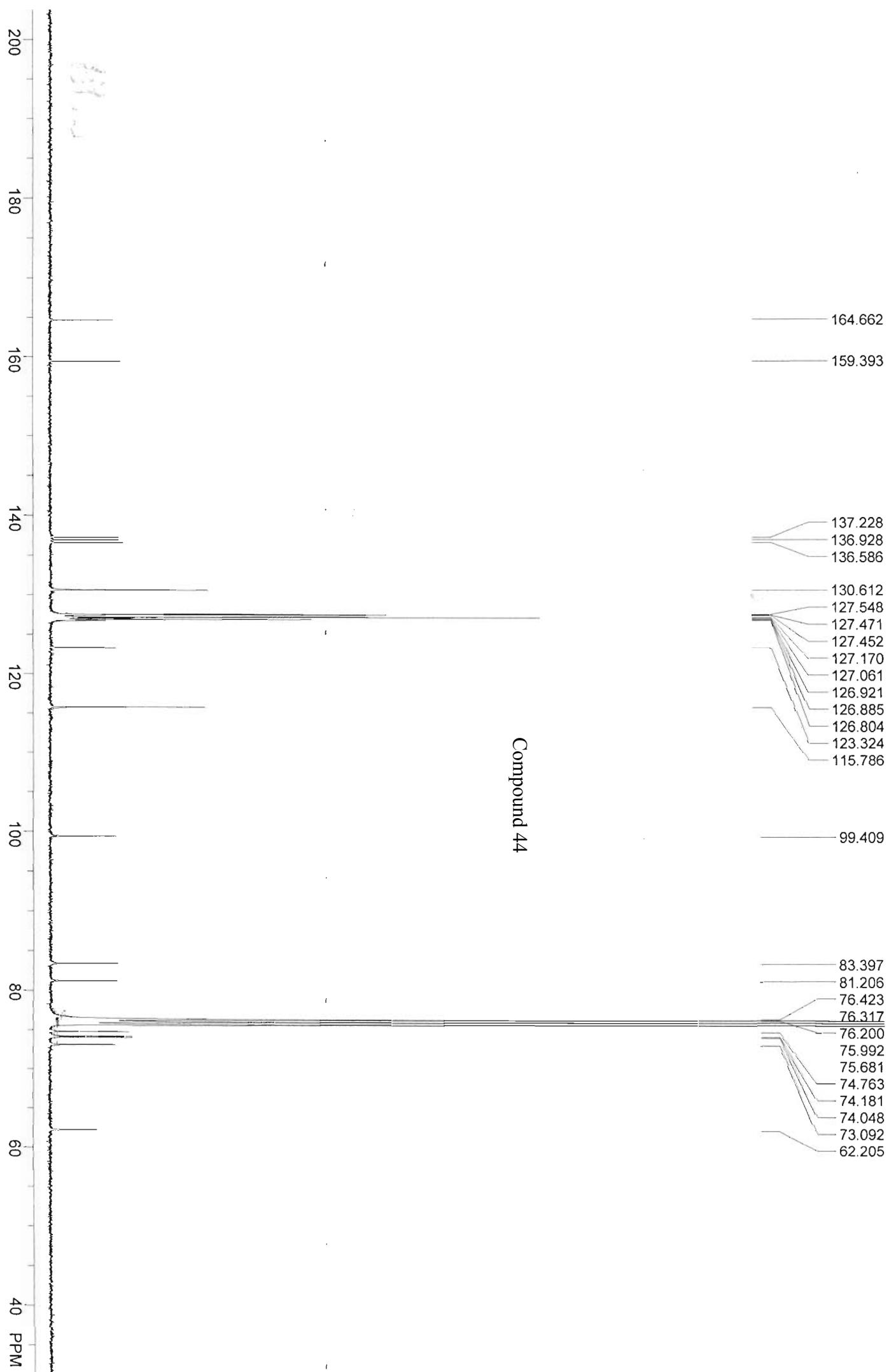


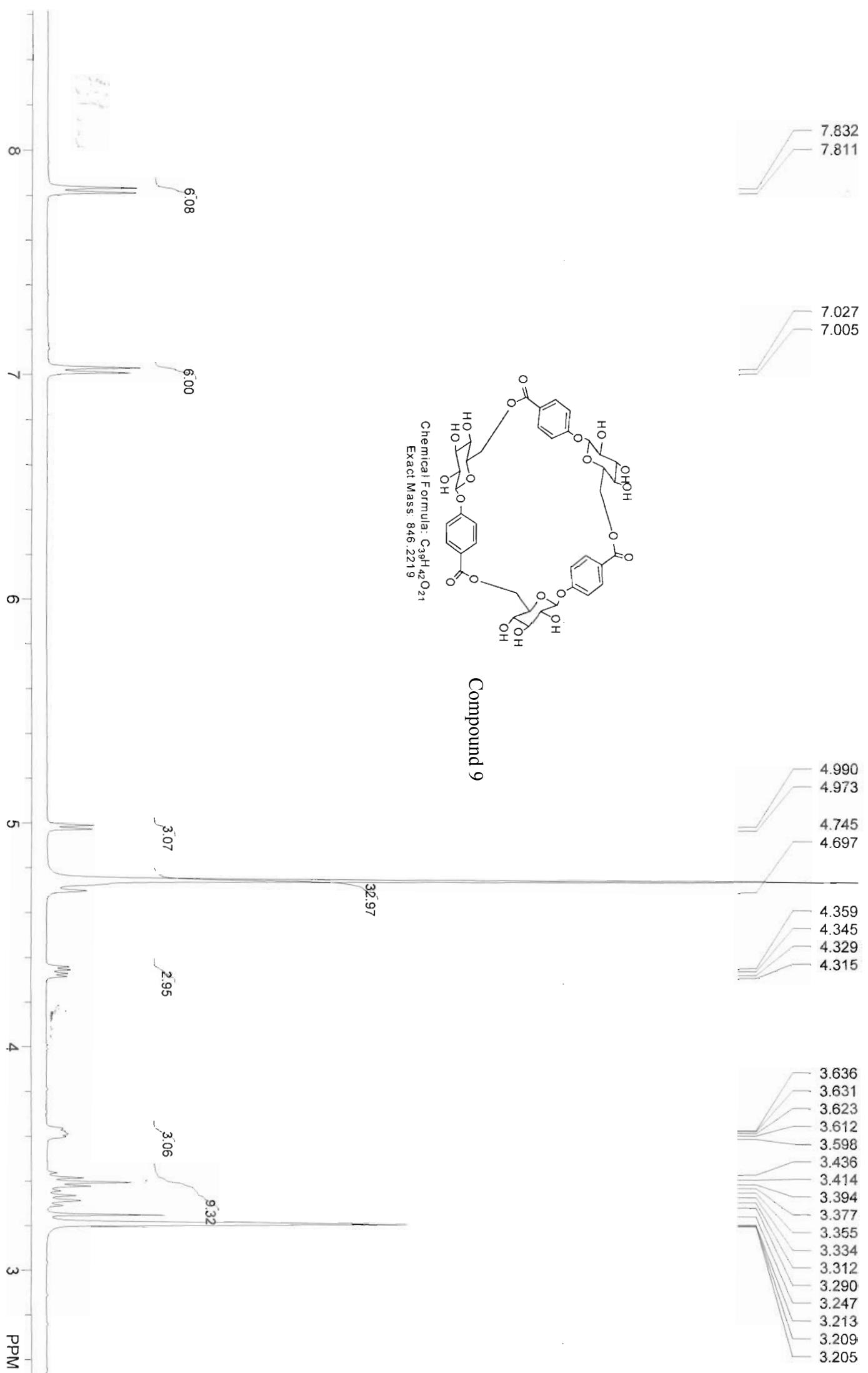
Compound 43

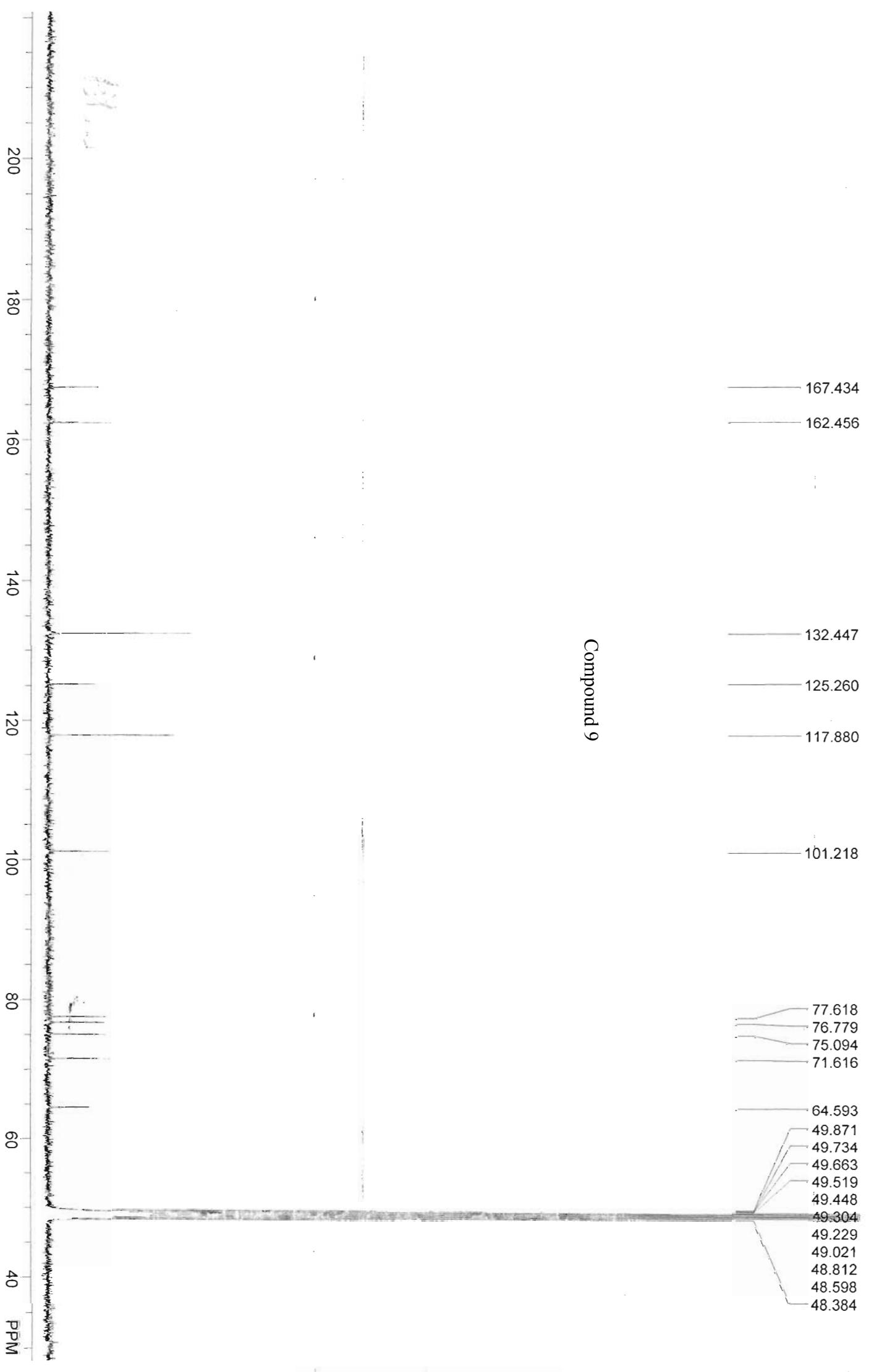


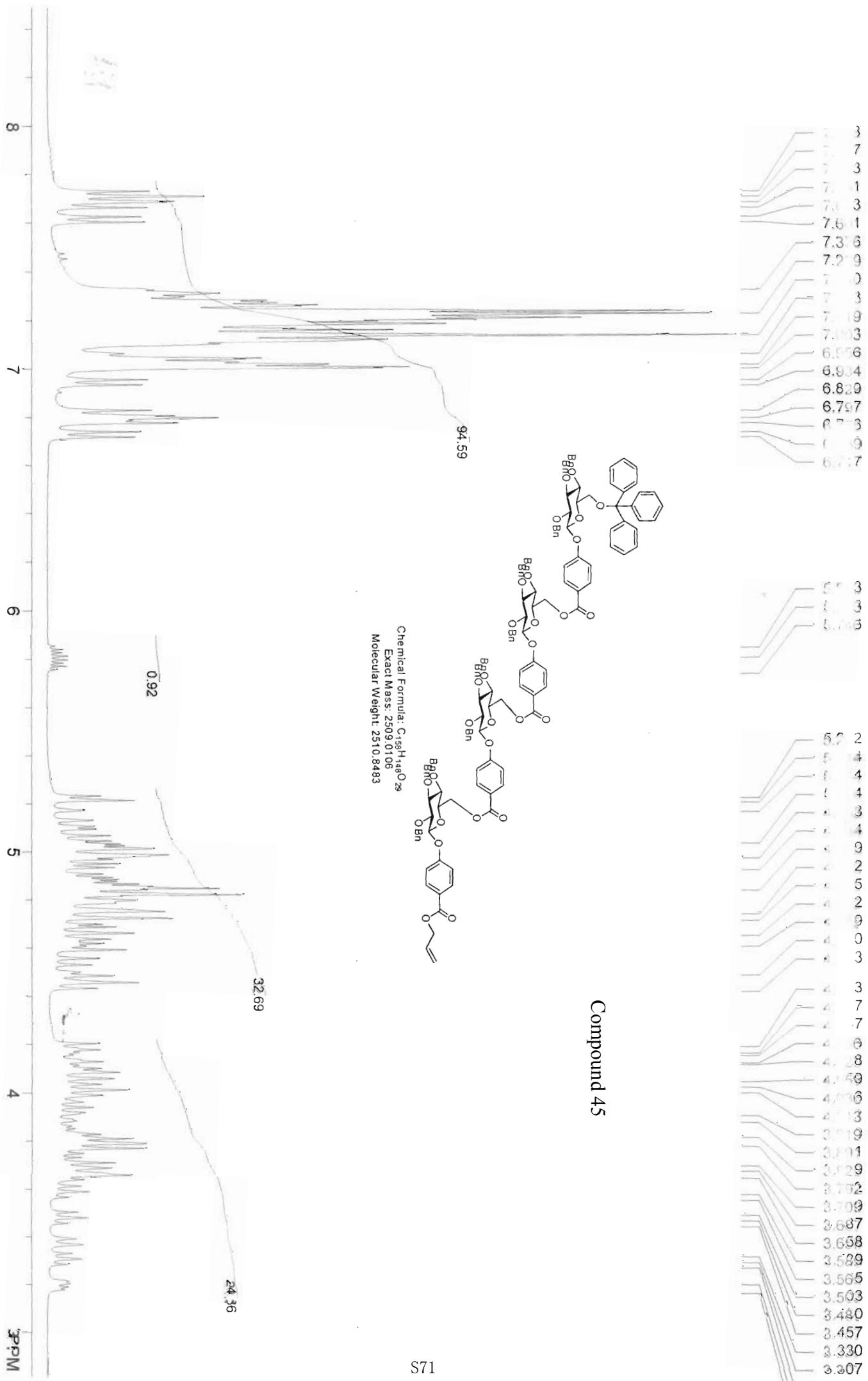
Compound 43

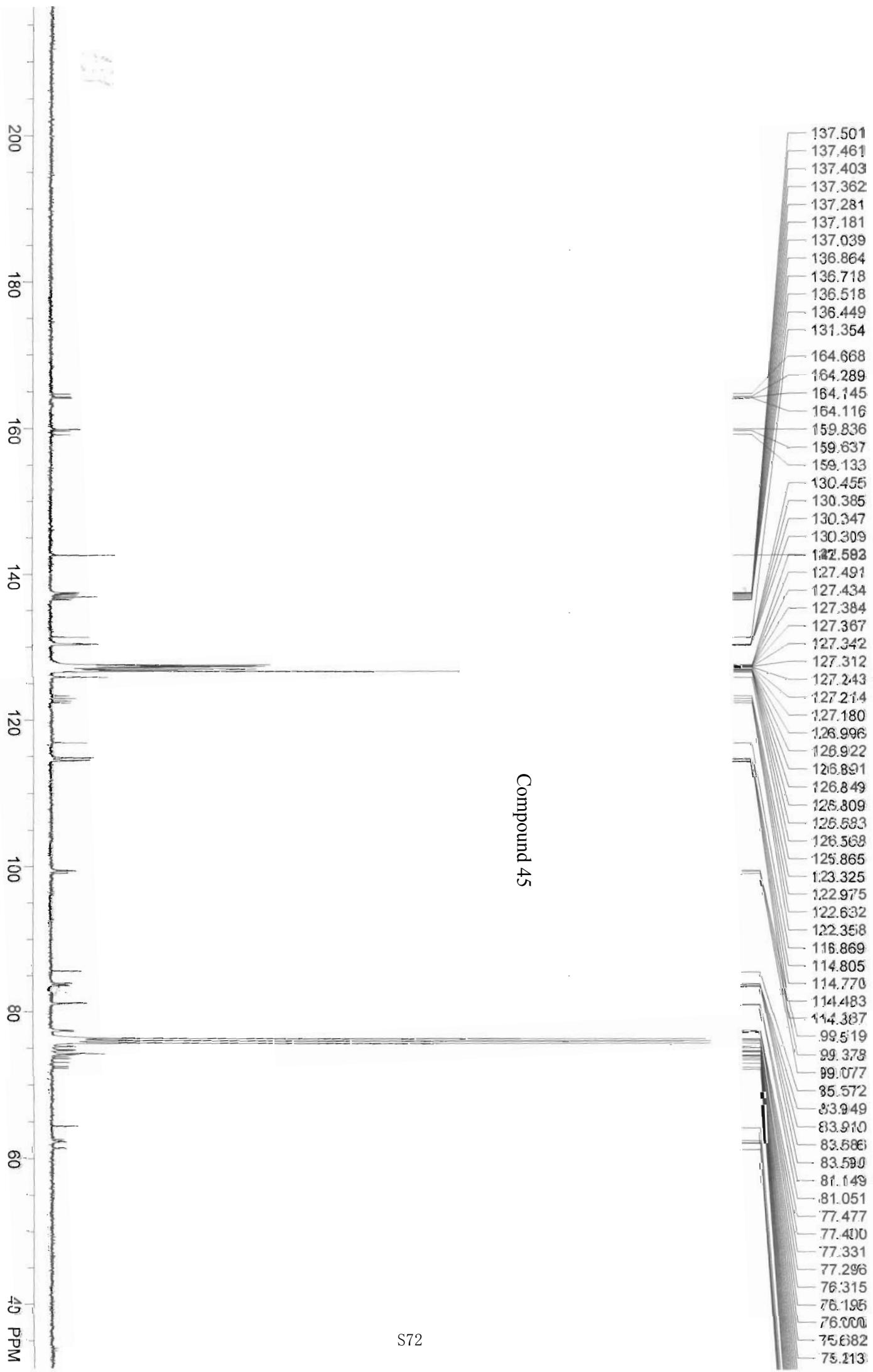




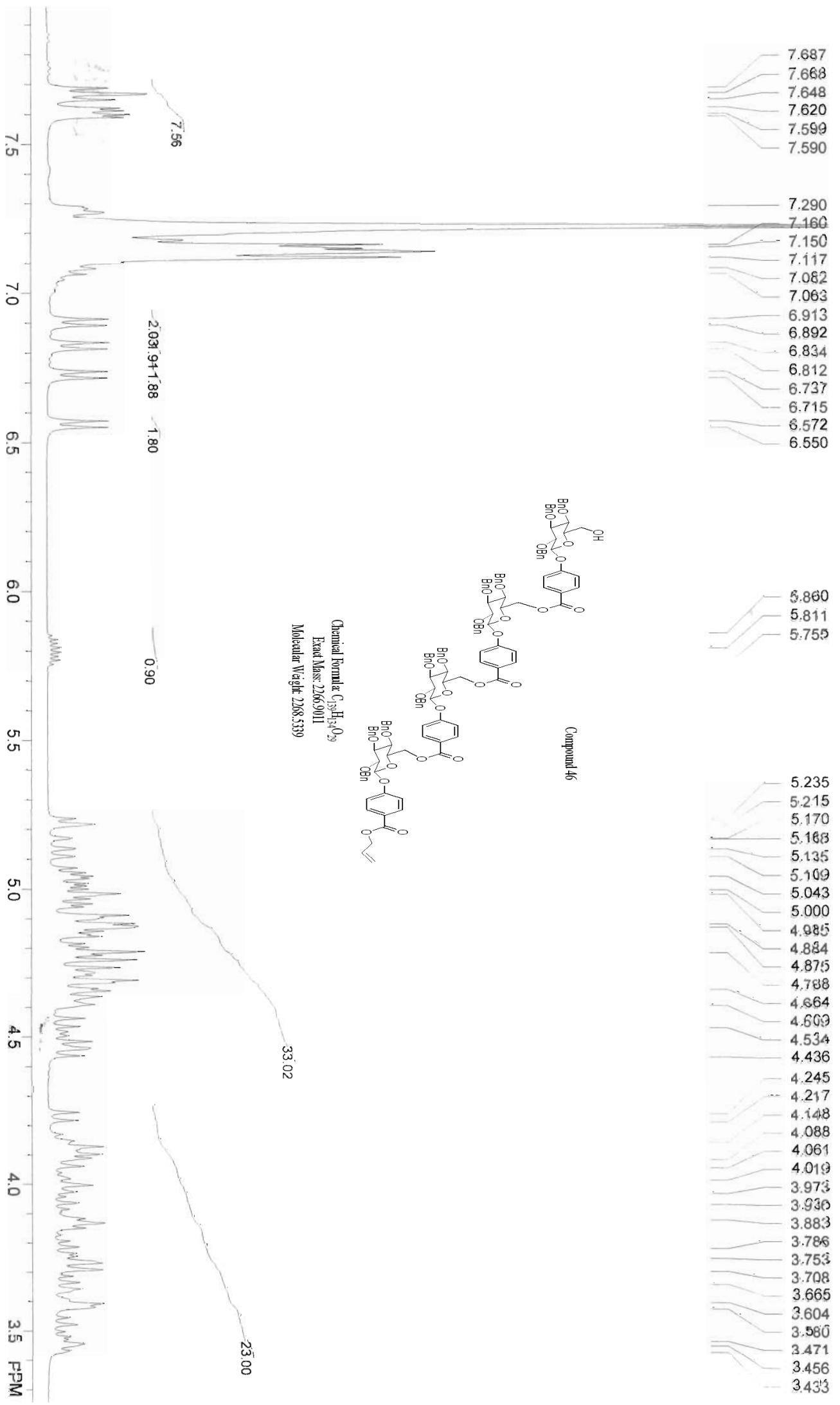


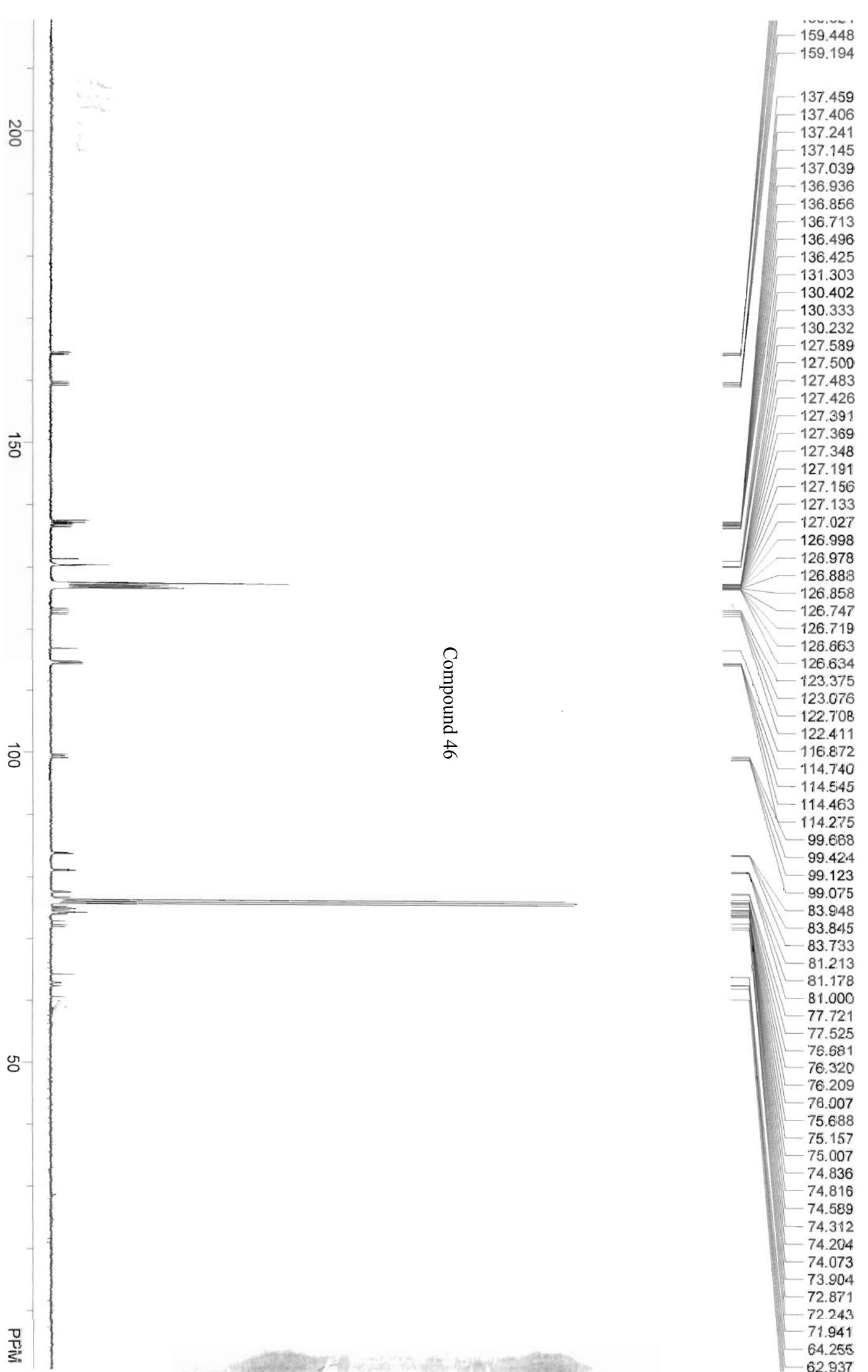


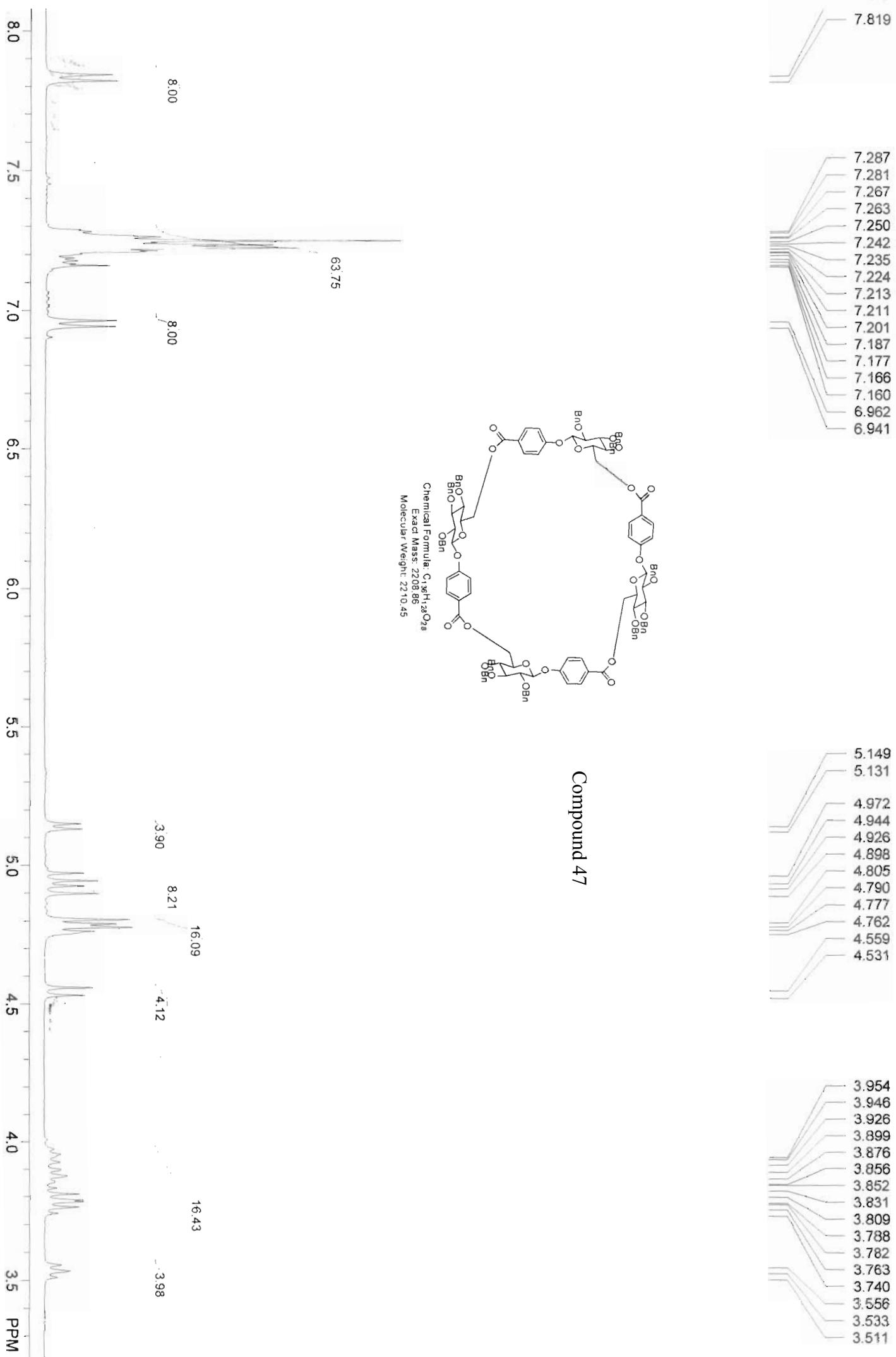




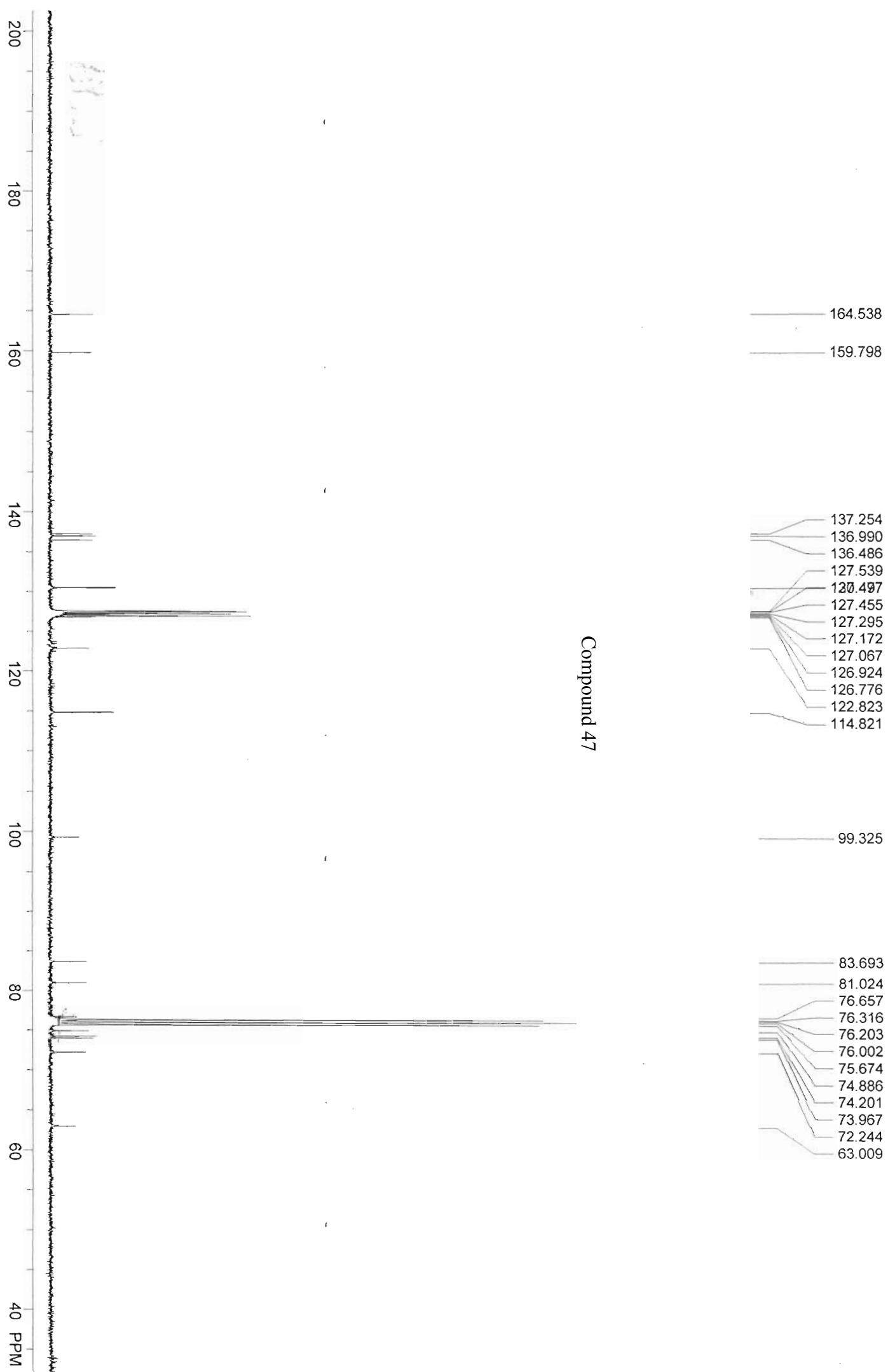
Compound 45

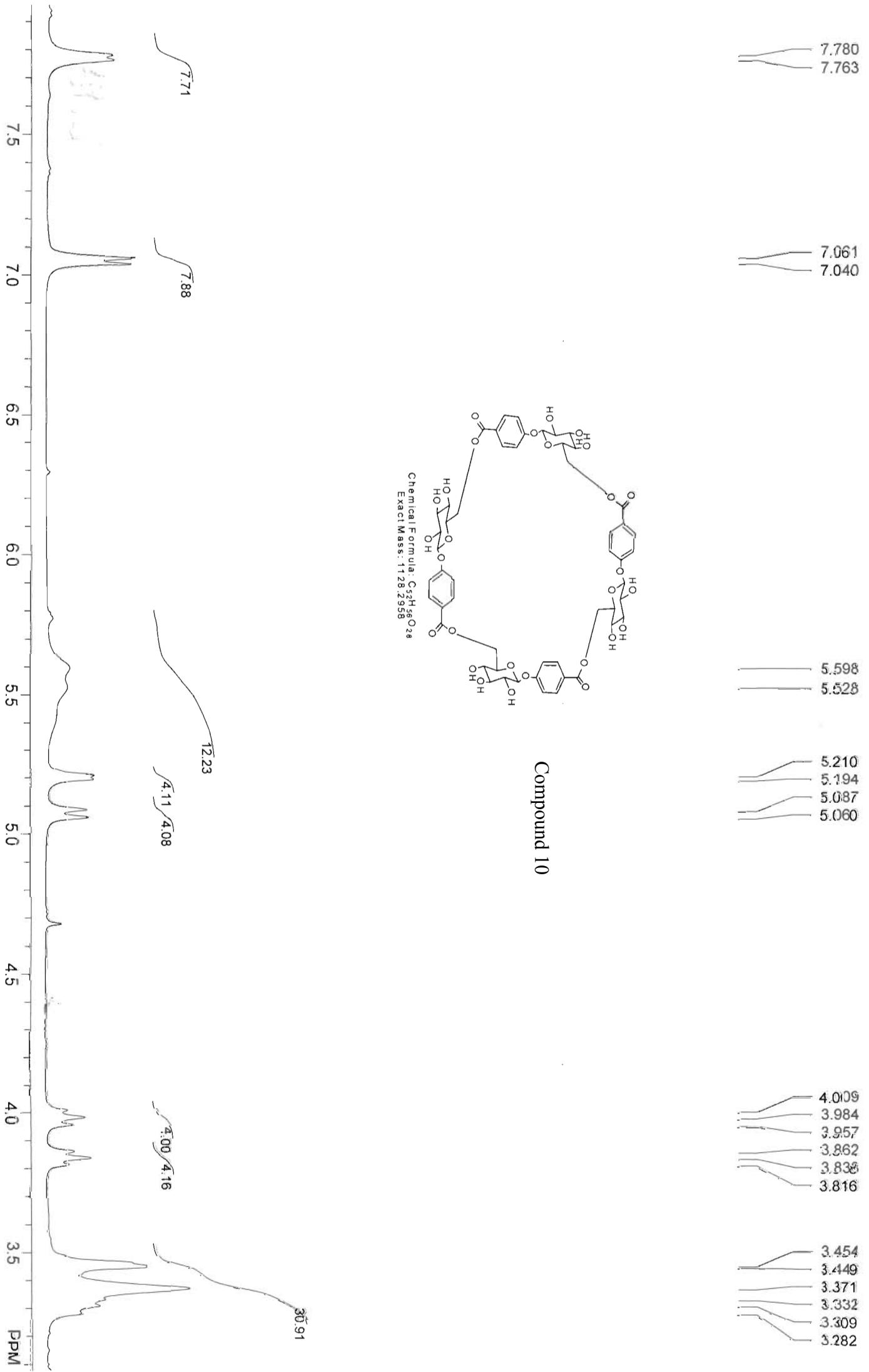


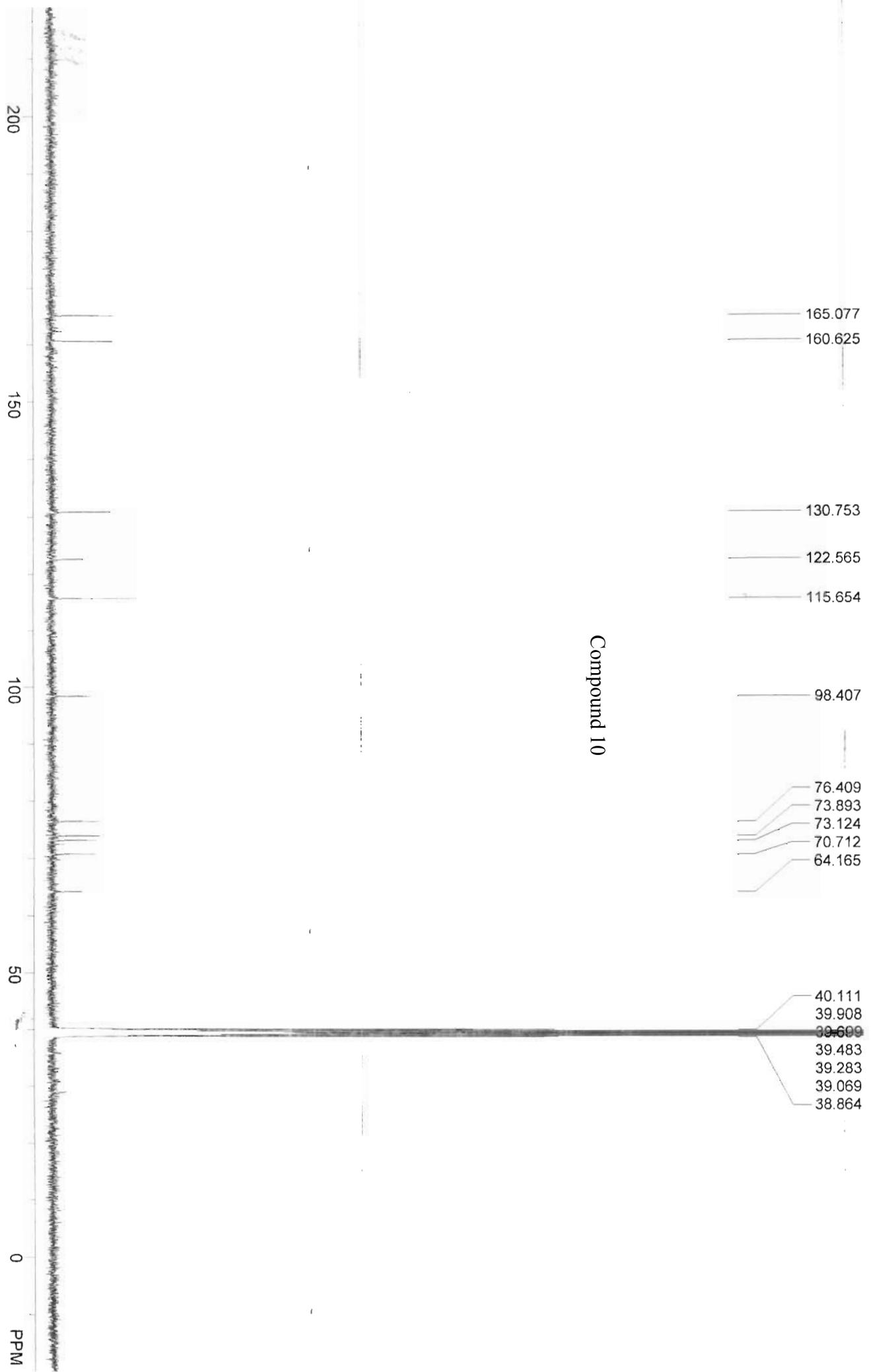


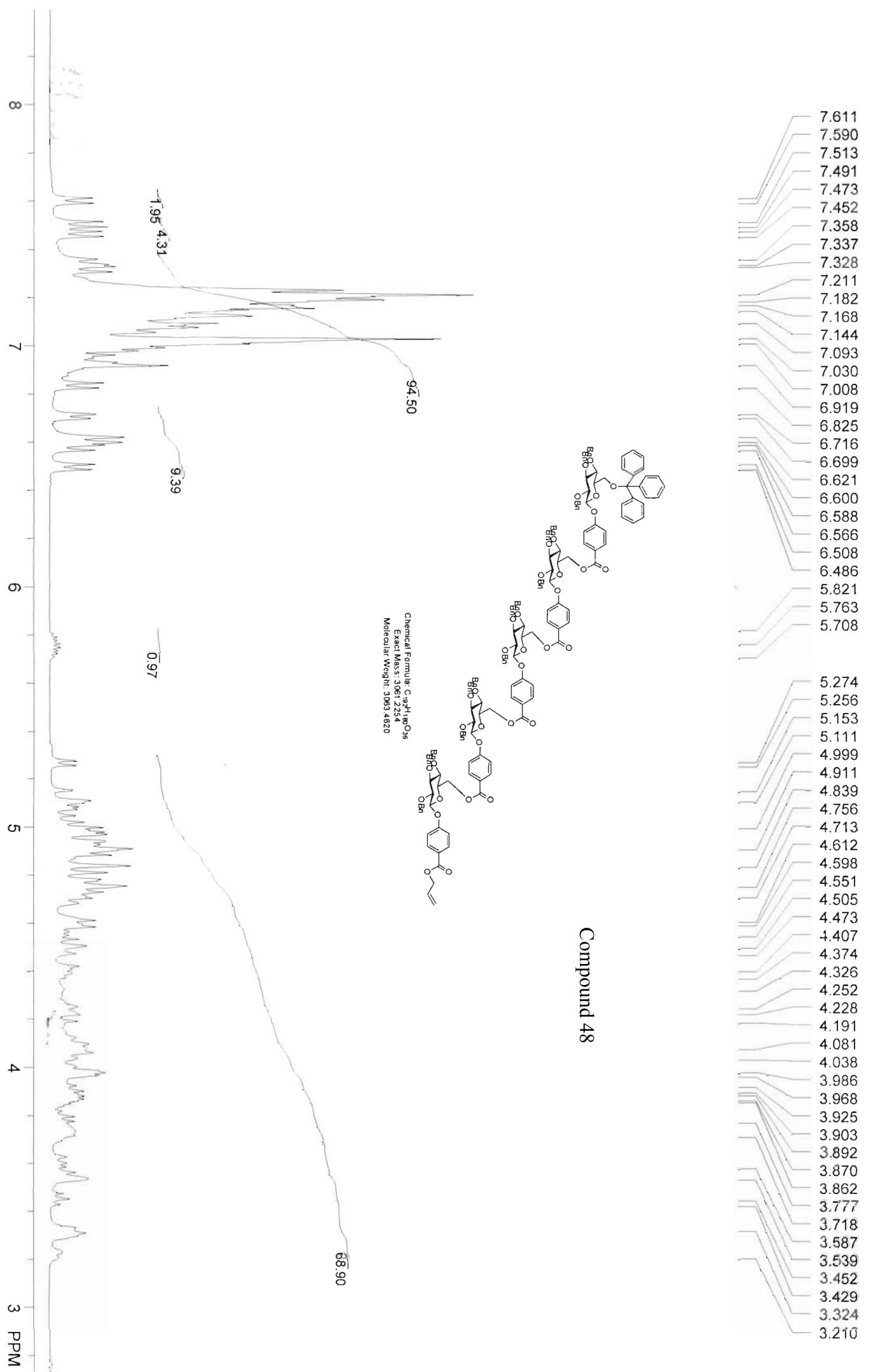


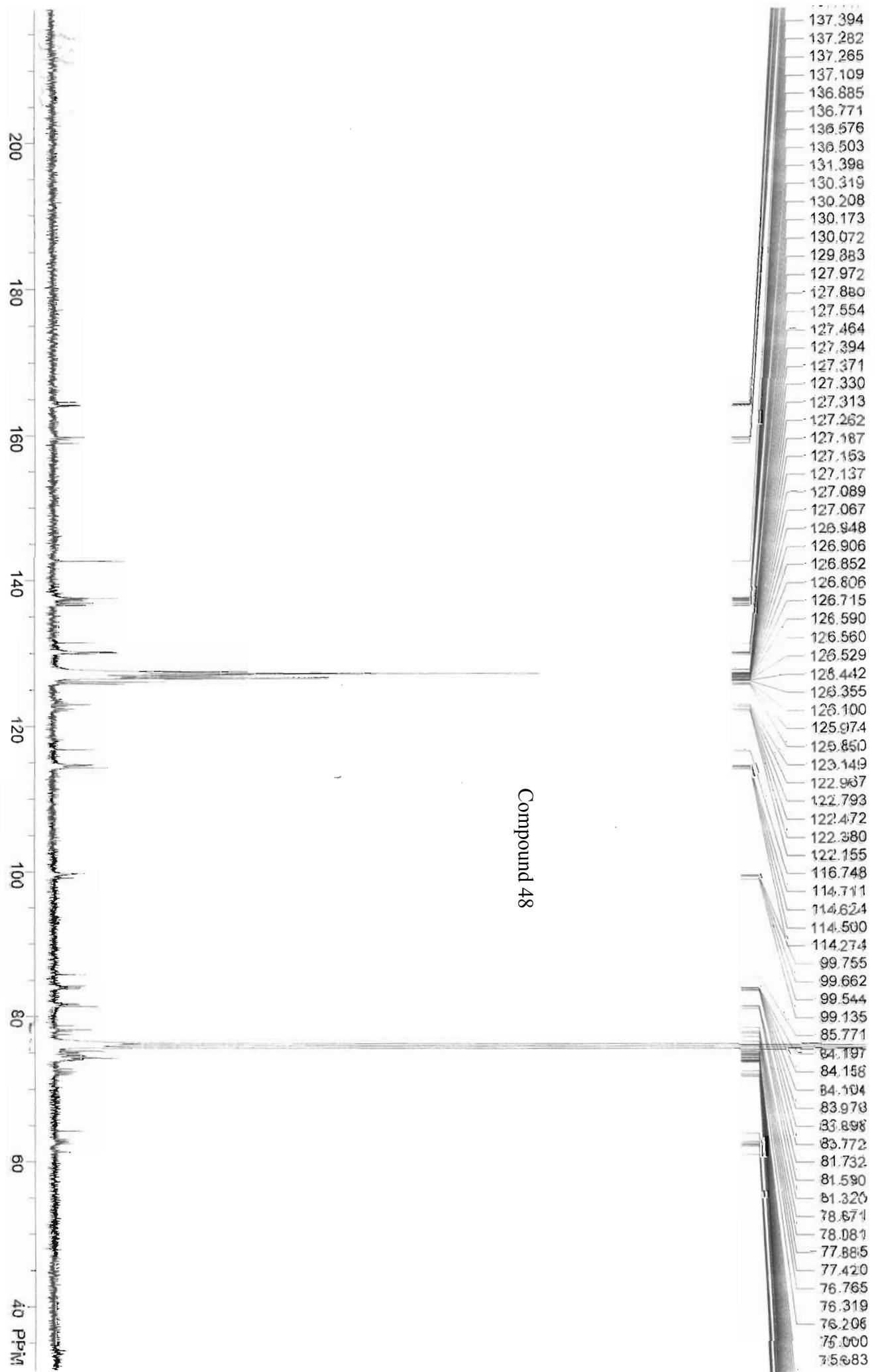
Compound 47

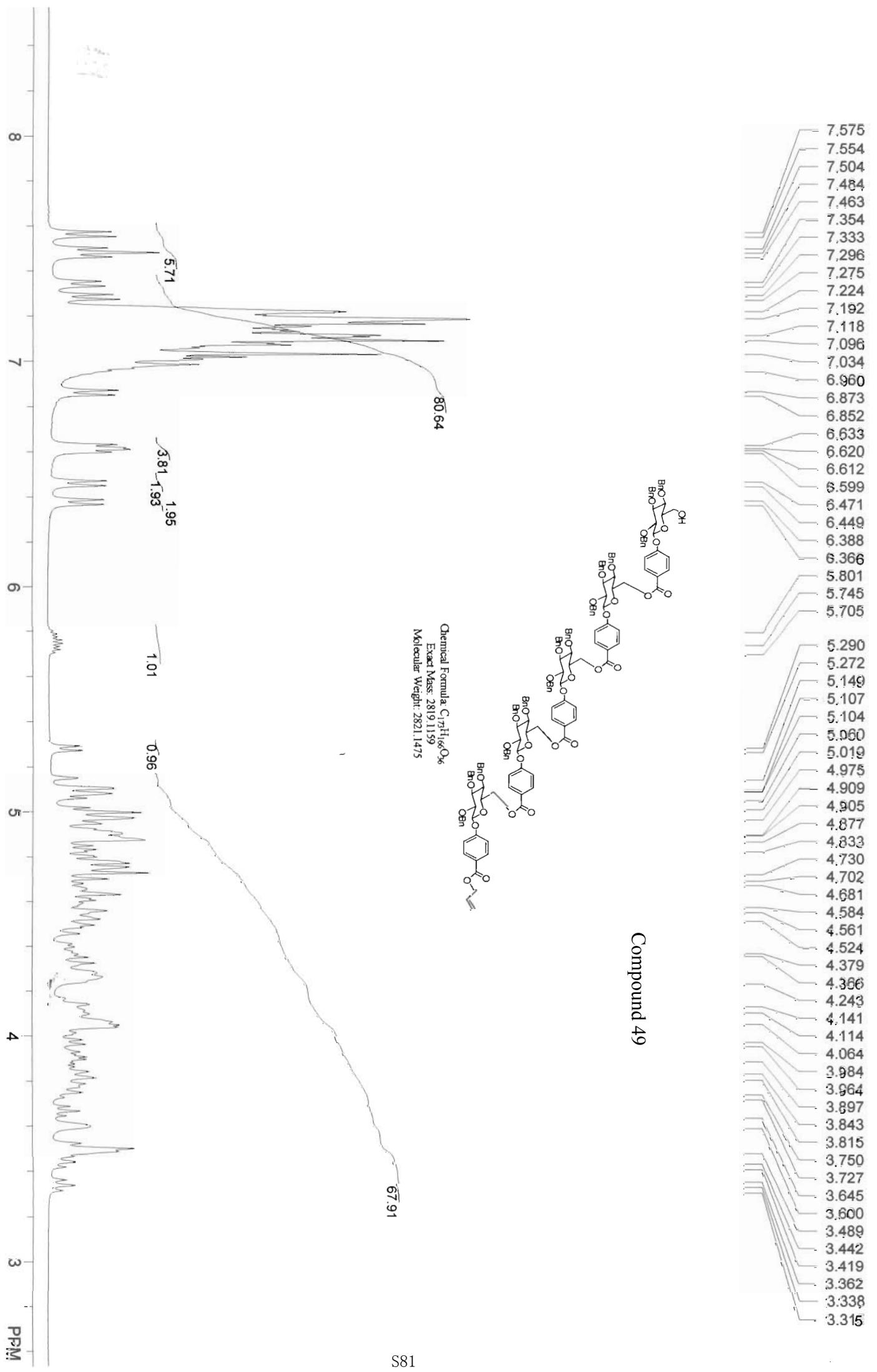


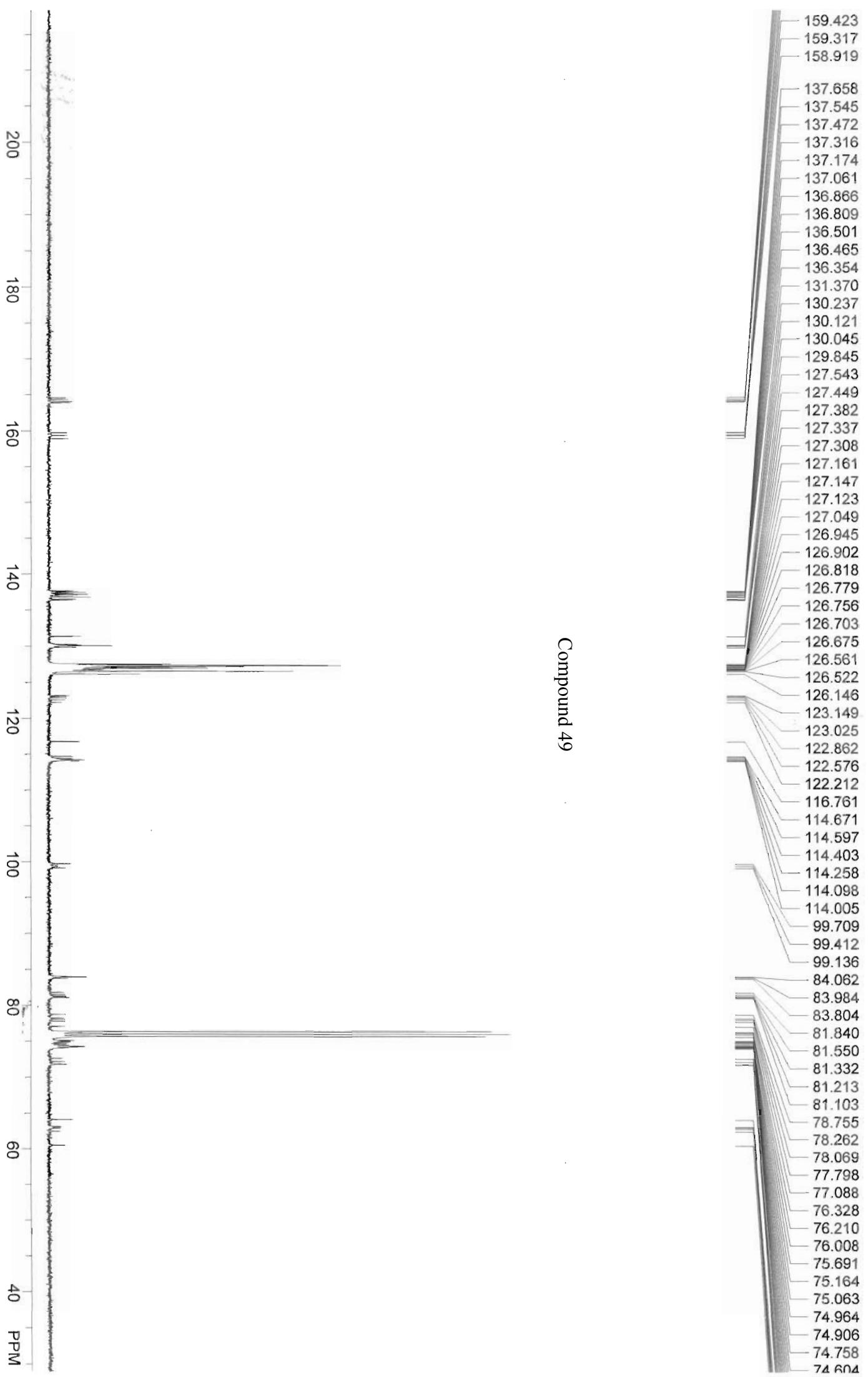












Compound 49

