Electronic Supporting Information

Divergent Pd(II) and Au(III) mediated nitroalkynol cycloisomerizations

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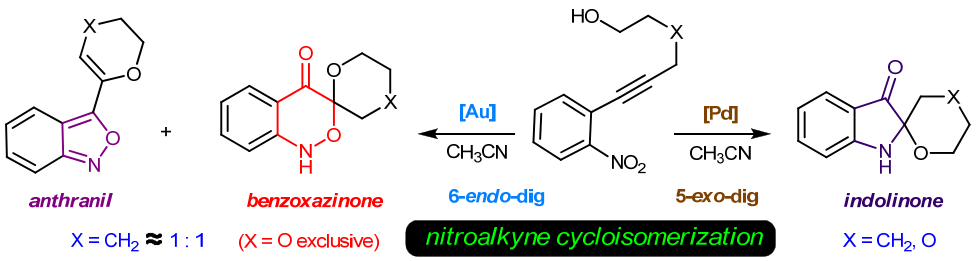

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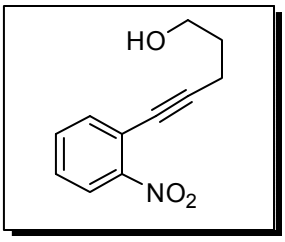
General: Air and/or moisture sensitive reactions were carried out in anhydrous solvents under an atmosphere of argon in oven-dried glassware. All anhydrous solvents were distilled prior to use: Acetonitrile and DMF from CaH_2; Triethylamine from KOH. Commercial reagents were used without purification. Column chromatography was carried out by using Spectrochem silica gel (100–200 mesh). ^1H and ^{13}C NMR spectroscopy measurements were carried out on Bruker AC 200 MHz or Bruker DRX 400 MHz spectrometers, and TMS was used as internal standard. ^1H and ^{13}C NMR chemical shifts are reported in ppm downfield from TMS and coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations are used to designate signal multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. The multiplicity of ^{13}C NMR signals was assigned with the help of DEPT spectra and the abbreviations used: s = singlet d = doublet t = triplet q = quartet, represent C (quaternary), CH, CH_2 and CH_3 respectively. Mass spectroscopy was carried out on a API QStar Pulsar (Hybrid Quadrupole-TOF LC/MS/MS) spectrometer or UPLC coupled Mass Spectrometer (Waters) and HRMS on 4800 plus MALDI TOF/TOF Applied Biosystem spectrometer. Elemental analysis data were obtained on a Thermo Finnigan Flash EA 1112 Series CHNS analyzer.

General Procedure for Sonogashira Coupling:[5] To a solution of alkyne (1 mmol), aryl iodide (1.2 mmol) in Et_3N (16 mL) and DMF (8 mL), TPP (0.1 mmol) and $Pd(PPh_3)_2Cl_2$ (0.1 mmol), were added and degassed with argon for 30 min. CuI (0.1 mmol) was added and degassed with argon for 10 min and stirred at rt for 5 h. The reaction mixture was partitioned between ethyl acetate and water. Organic layer was separated, washed with brine, dried (Na_2SO_4), concentrated and the residue obtained was purified by column chromatography (ethyl acetate in petroleum ether) to afford **1**.

Characterization data

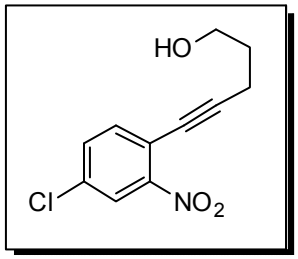


Compound 1aa. Yellow liquid, 83% yield. IR ($CHCl_3$): ν 3351, 2926, 2224, 1678, 1613, 1511, 1466, 1273, 1035 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 1.87 (qui, *J* = 6.4 Hz, 2H), 1.87 (br s, 1H), 2.60 (t, *J* = 6.8 Hz, 2H), 3.83 (t, *J* = 6.1 Hz, 2H), 7.38 (ddd, *J* = 2.1, 6.6, 8.1

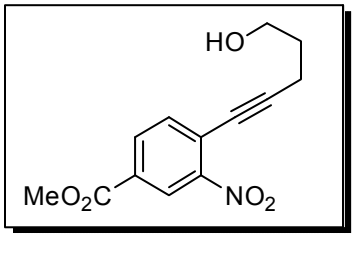
Hz, 1H), 7.51 (dt, *J* = 1.4, 7.8 Hz, 1H), 7.56 (dd, *J* = 2.2, 7.6 Hz, 1H), 7.95 (dd, *J* = 1.3, 7.8 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 16.2 (t), 30.7 (t), 61.2 (t), 76.3 (s), 98.3 (s), 118.9 (s), 124.3 (d), 127.9 (d), 132.6 (d), 134.6 (d), 149.8 (s). ppm. ESI-MS: *m/z* 206.4 (100%, $[M+H]^+$). Anal. Calcd for $C_{11}H_{11}NO_3$: C, 64.38; H, 5.40; N, 6.83. Found: C, 64.47; H, 5.52; N, 6.62.

Compound 1ab. Procuring the clean spectral data of **1ab** was found to be difficult as it was slowly converting to the cycloisomerization products on standing.

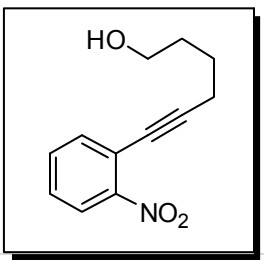
Compound 1ac. Yellow liquid, 81% yield. IR ($CHCl_3$): ν 3368, 2949, 2230, 1605, 1555, 1530, 1478, 1345, 1259, 1110, 1058, cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.76 (br s, 1H), 1.87 (qui, *J* = 6.5 Hz, 2H), 2.60 (t, *J* = 6.9 Hz, 2H), 3.82 (t, *J* = 6.1 Hz, 2H), 7.49 (d, *J* = 1.3 Hz, 2H), 7.97 (t, *J* = 1.3 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 16.3 (t), 30.7 (t), 61.2 (t), 75.5 (s), 99.6 (s), 117.6 (s), 124.6 (d), 132.8 (d), 133.7 (s), 135.6 (d), 150.1 (s) ppm. ESI-MS: *m/z* 240.4 (100%, $[M+H]^+$). Anal. Calcd for $C_{11}H_{10}ClNO_3$: C, 55.13; H, 4.21; Cl, 14.79; N, 5.84. Found: C, 55.22; H, 4.29; Cl, 14.62; N, 5.67.



Compound 1ad. Yellow liquid, 77% yield. IR ($CHCl_3$): ν 3365, 3017, 2954, 2230, 1719, 1619, 1534, 1437, 1289, 1090 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.85 (qui, *J* = 6.4 Hz, 2H), 2.34 (br s, 1H), 2.60 (t, *J* = 6.9 Hz, 2H), 3.79 (t, *J* = 6.1 Hz, 2H), 3.91 (s, 3H), 7.58 (dd, *J* = 8.1 Hz, 1H), 8.10 (dd, *J* = 1.7, 8.1 Hz, 1H), 8.55 (d, *J* = 1.7 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 16.4 (t), 30.6 (t), 52.7 (q), 61.0 (t), 76.1 (s), 102.2 (s), 123.1 (s), 125.4 (d), 129.6 (s), 132.9 (d), 134.9 (d), 149.6 (s), 164.5 (s)ppm. ESI-MS: *m/z* 264.5 (100%, $[M+H]^+$). Anal. Calcd for $C_{13}H_{13}NO_5$: C, 59.31; H, 4.98; N, 5.32. Found: C, 59.32; H, 5.00; N, 5.33.

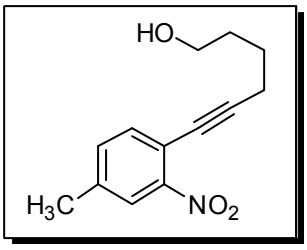


Compound 1ba. Yellow liquid, 71% yield. IR ($CHCl_3$): ν 3351, 2936, 2225, 1682, 1613, 1503, 1466, 1299, 1035 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.66–1.79 (m, 5H), 2.51 (t, *J* = 6.4 Hz, 2H), 3.69 (t, *J* = 6.0

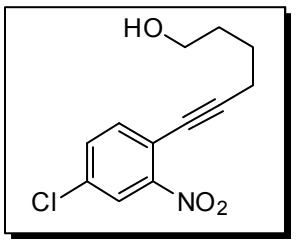


Hz, 2H), 7.37 (ddd, *J* = 2.1, 6.8, 8.2 Hz, 1H), 7.50 (dt, *J* = 1.4, 7.7 Hz, 1H), 7.56 (dd, *J* = 2.1, 7.7 Hz, 1H), 7.94 (dd, *J* = 1.3, 7.8 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 19.4 (t), 24.5 (t), 31.6 (t), 62.1 (t), 76.2 (s), 98.8 (s), 119.0 (s), 124.2 (d), 127.8 (d), 132.5 (d), 134.6 (d), 149.8 (s) ppm. ESI-MS: *m/z* 220.3 (100%, $[M+H]^+$). Anal. Calcd for $C_{12}H_{13}NO_3$: C, 65.74; H, 5.98; N, 6.39. Found: C, 65.59; H, 5.91; N, 6.52.

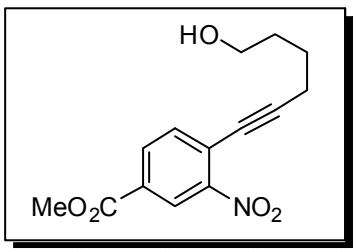
Compound 1bb. Yellow liquid, 70% yield. IR ($CHCl_3$): ν 3368, 3086, 2949, 2230, 1605, 1555, 1530, 1478, 1345, 1152, 1058 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.66–1.76 (m, 4H), 2.01 (br s, 1H), 2.38 (s, 3H), 2.48 (t, *J* = 6.4 Hz, 2H), 3.68 (t, *J* = 5.8 Hz, 2H), 7.29 (dd, *J* = 1.1, 7.9 Hz, 1H), 7.41 (d, *J* = 7.9 Hz, 1H), 7.73 (s, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 19.4 (t), 20.9 (q), 24.5 (t), 31.6 (t), 62.1 (t), 76.1 (s), 97.6 (s), 116.1 (s), 124.5 (d), 133.3 (d), 134.3 (d), 138.7 (s), 149.7 (s)ppm. ESI-MS: *m/z* 234.5 (100%, $[M+H]^+$). Anal. Calcd for $C_{13}H_{15}NO_3$: C, 66.94; H, 6.48; N, 6.00. Found: C, 67.04; H, 6.53; N, 5.89.



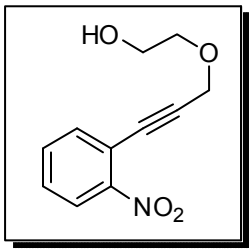
Compound 1bc. Yellow liquid, 73% yield. IR ($CHCl_3$): ν 3387, 3018, 2952, 2230, 1605, 1545, 1528, 1478, 1346, 1258, 1110, 1051 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.62–1.79 (m, 4H), 2.12 (br s, 1H), 2.49 (t, *J* = 6.5 Hz, 2H), 3.68 (t, *J* = 6.0 Hz, 2H), 7.47 (d, *J* = 1.3 Hz, 2H), 7.94 (t, *J* = 1.3 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 19.6 (t), 24.4 (t), 31.6 (t), 62.2 (t), 75.4 (s), 100.1 (s), 117.7 (s), 124.6 (d), 132.8 (d), 133.6 (s), 135.6 (d), 150.1 (s) ppm. ESI-MS: *m/z* 254.7 (100%, $[M+H]^+$). Anal. Calcd for $C_{12}H_{12}ClNO_3$: C, 56.81; H, 4.77;Cl, 13.98; N, 5.52. Found: C, 56.79; H, 4.71;Cl, 13.71; N, 5.39.



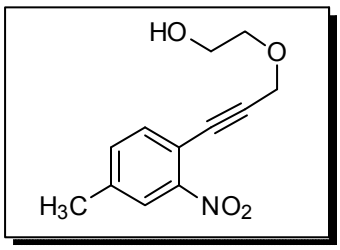
Compound 1bd. Yellow liquid, 83% yield. IR ($CHCl_3$): ν 3377, 3017, 2952, 2228, 1728, 1621, 1533, 1437, 1234, 1098 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.66–1.77 (m, 4H), 2.04 (br s, 1H), 2.52 (t, *J* = 6.5 Hz, 2H), 3.68 (t, *J* = 6.0 Hz, 2H), 3.92 (s, 3H), 7.59 (dd, *J* = 8.1 Hz, 1H), 8.11 (dd, *J* = 1.6, 8.1 Hz, 1H), 8.56 (d, *J* = 1.6 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 19.6 (t), 24.3 (t), 31.5



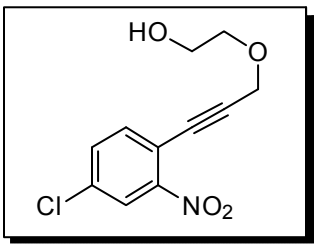
(t), 52.7 (q), 62.0 (t), 76.0 (s), 102.7 (s), 123.2 (s), 125.4 (d), 129.5 (s), 132.8 (d), 134.9 (d), 149.7 (s), 164.5 (s) ppm. ESI-MS: *m/z* 278.4 (100%, $[M+H]^+$). Anal. Calcd for $C_{14}H_{15}NO_5$: C, 60.64; H, 5.45; N, 5.05. Found: C, 60.75; H, 5.39; N, 5.22.



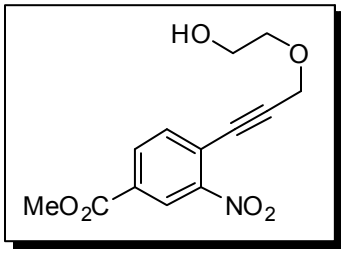
Compound 1ca. Yellow liquid, 70% yield. IR ($CHCl_3$): ν 3020, 2918, 2223, 1608, 1578, 1480, 1345, 1216, 1068 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 2.29 (br s, 1H), 3.71–3.81 (m, 4H), 4.47 (s, 2H), 7.37 (ddd, *J* = 2.1, 6.9, 8.1 Hz, 1H), 7.50 (dt, *J* = 1.3, 7.8 Hz, 1H), 7.56 (dd, *J* = 1.9, 7.7 Hz, 1H), 7.94 (dd, *J* = 1.1, 7.8 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 58.9 (t), 61.5 (t), 71.3 (t), 81.6 (s), 93.0 (s), 117.7 (s), 124.4 (d), 128.8 (d), 132.8 (d), 134.7 (d), 149.5 (s) ppm. ESI-MS: *m/z* 221.4 (100%, $[M]^+$). Anal. Calcd for $C_{11}H_{11}NO_3$: C, 59.73; H, 5.01; N, 6.33. Found: C, 59.59; H, 5.25; N, 6.18.



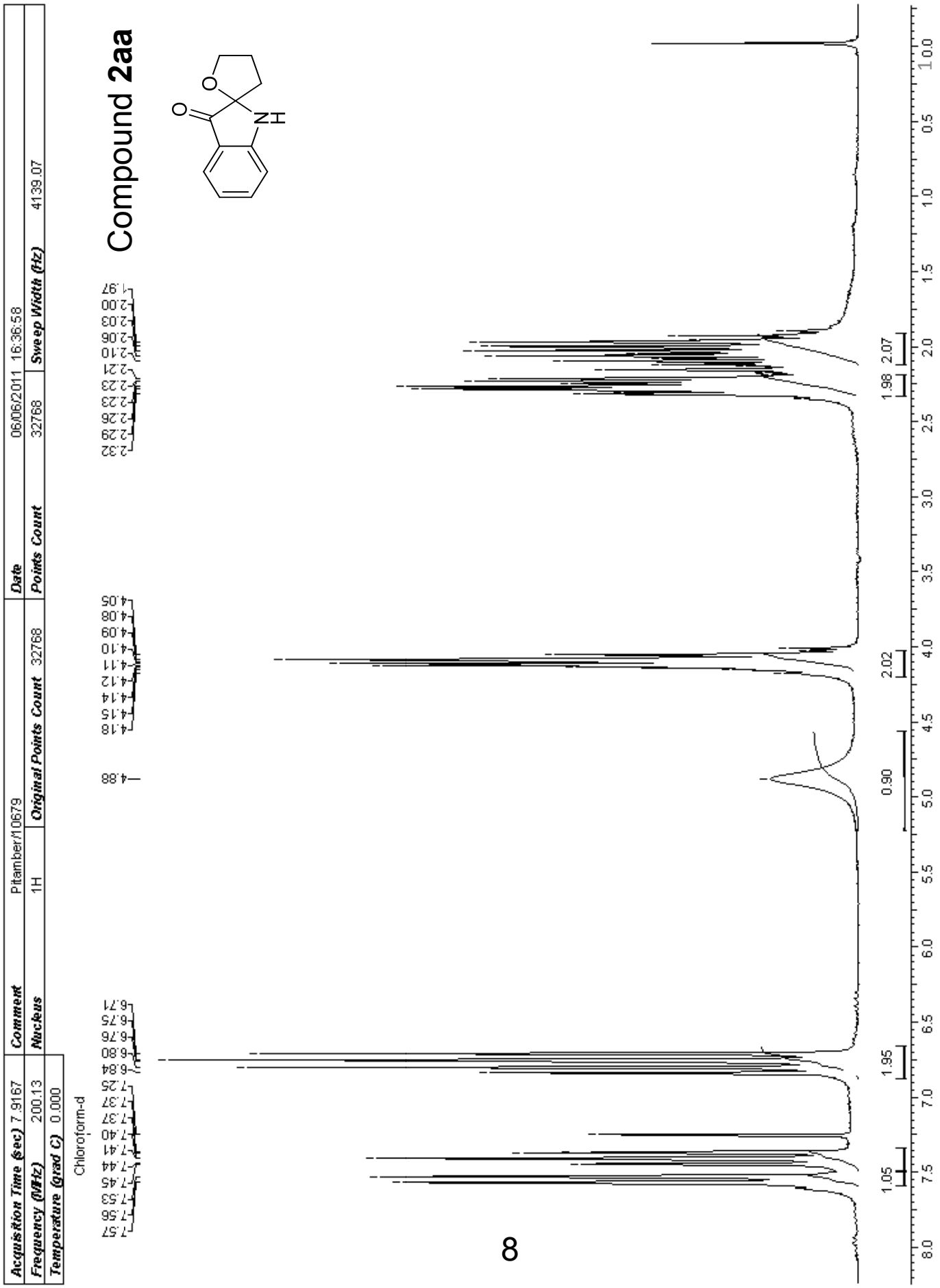
Compound 1cb. Off white solid, Mp. 79 °C, 70% yield. IR ($CHCl_3$): ν 3327, 3021, 2952, 2219, 1609, 1595, 1525, 1434, 1344, 1216, 1084 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 2.23 (br s, 1H), 2.41 (s, 3H), 3.71–3.81 (m, 4H), 4.45 (s, 2H), 7.35 (dd, *J* = 1.0, 7.9 Hz, 1H), 7.48 (d, *J* = 7.9 Hz, 1H), 7.73 (s, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 21.1 (q), 59.1 (t), 61.7 (t), 71.3 (t), 81.7 (s), 92.0 (s), 114.9 (s), 124.9 (d), 133.6 (d), 134.5 (d), 139.9 (s), 149.5 (s)ppm. ESI-MS: *m/z* 236.2 (100%, $[M+H]^+$). Anal. Calcd for $C_{12}H_{13}NO_4$: C, 61.27; H, 5.57; N, 5.95. Found: C, 61.09; H, 5.63; N, 5.81.



Compound 1cc. Yellow liquid, 82% yield. IR ($CHCl_3$): ν 3421, 2974, 2932, 2229, 1605, 1556, 1533, 1478, 1345, 1261, 1110, 882, 833, 759, 656 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 2.15 (br s, 1H), 3.72–3. 83 (m, 4H), 4.47 (s, 2H), 7.54 (d, *J* = 1.3 Hz, 1H), 7.55 (s, 1H), 8.03 (t, *J* = 1.3 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 59.1 (t), 61.7 (t), 71.4 (t), 80.8 (s), 94.2 (s), 116.3 (s), 124.9 (d), 133.1 (d), 134.9 (s), 135.7 (d), 149.9 (s) ppm. ESI-MS: *m/z* 256.6 (100%, $[M+H]^+$). Anal. Calcd for $C_{11}H_{10}ClNO_3$: C, 51.68; H, 3.94; Cl, 13.87; N, 5.48. Found: C, 51.51; H, 3.78; Cl, 13.61; N, 5.39.



Compound 1cd. Yellow liquid, 74% yield. IR ($CHCl_3$): ν 3325, 2927, 2220, 1754, 1608, 1526, 1345, 1250, 1120, 1056 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 2.30 (br s, 1H), 3.71–3.83 (m, 4H), 3.95 (s, 3H), 4.49 (s, 2H), 7.68 (d, *J* = 8.1 Hz, 1H), 8.18 (dd, *J* = 1.5, 8.1 Hz, 1H), 8.65 (d, *J* = 1.5 Hz, 1H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$): δ 52.8 (q), 59.1 (t), 61.4 (t), 71.5 (t), 81.3 (s), 96.6 (s), 121.8 (s), 125.6 (d), 130.6 (s), 133.1 (d), 135.0 (d), 149.5 (s), 164.4 (s)ppm. ESI-MS: *m/z* 280.3 (100%, $[M+H]^+$). Anal. Calcd for $C_{13}H_{13}NO_6$: C, 55.91; H, 4.69; N, 5.02. Found: C, 55.77; H, 4.52; N, 4.95.



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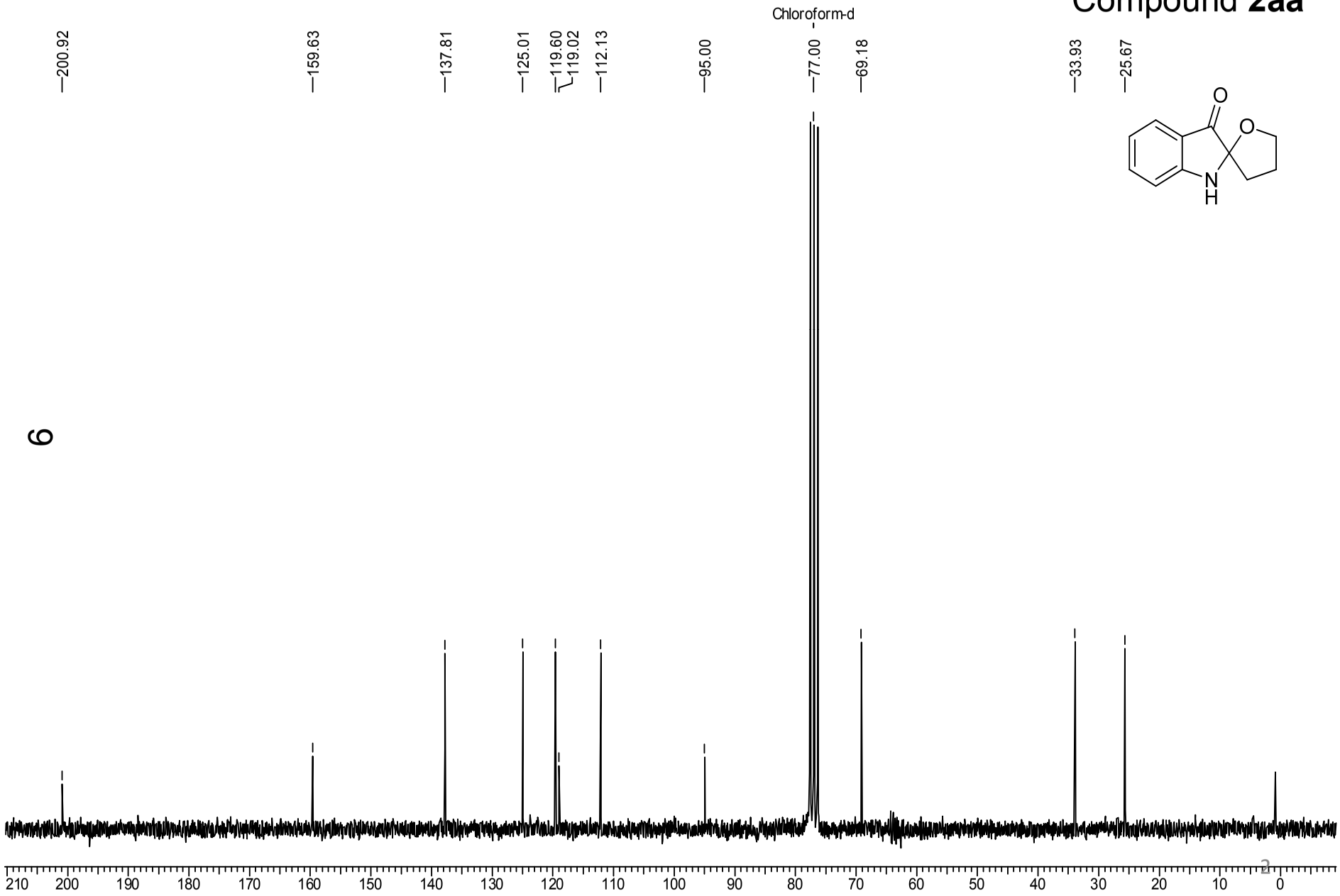

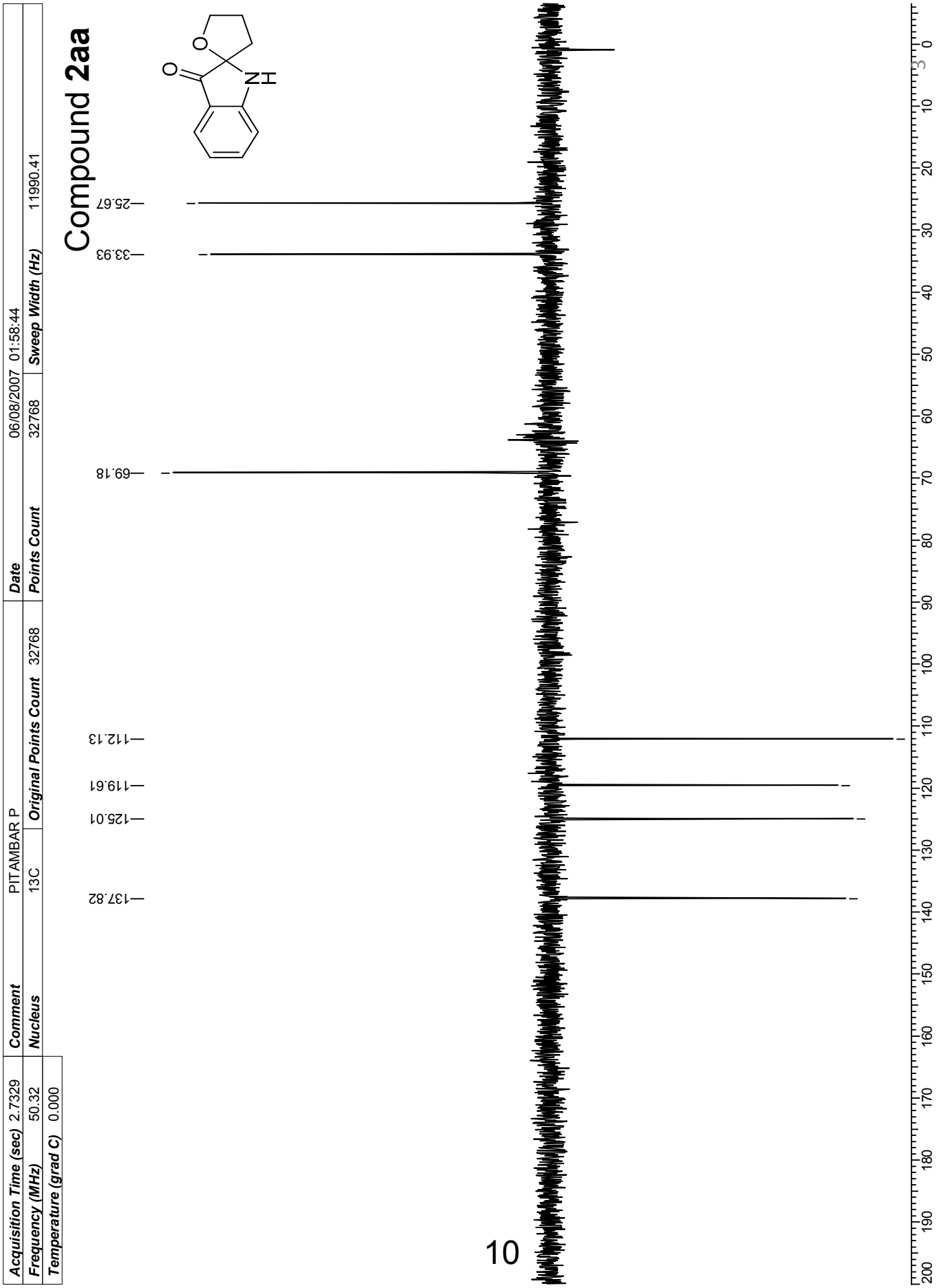

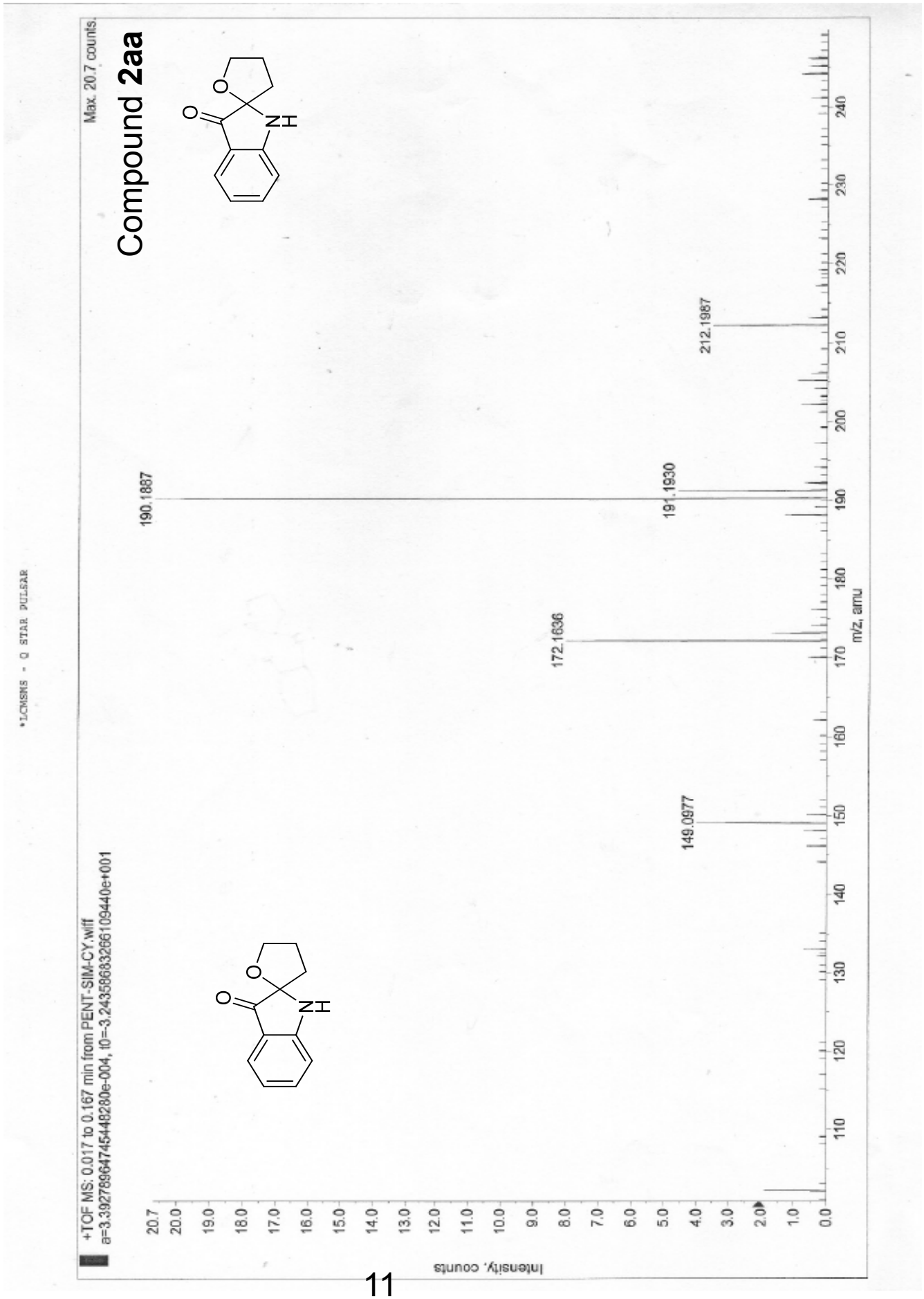
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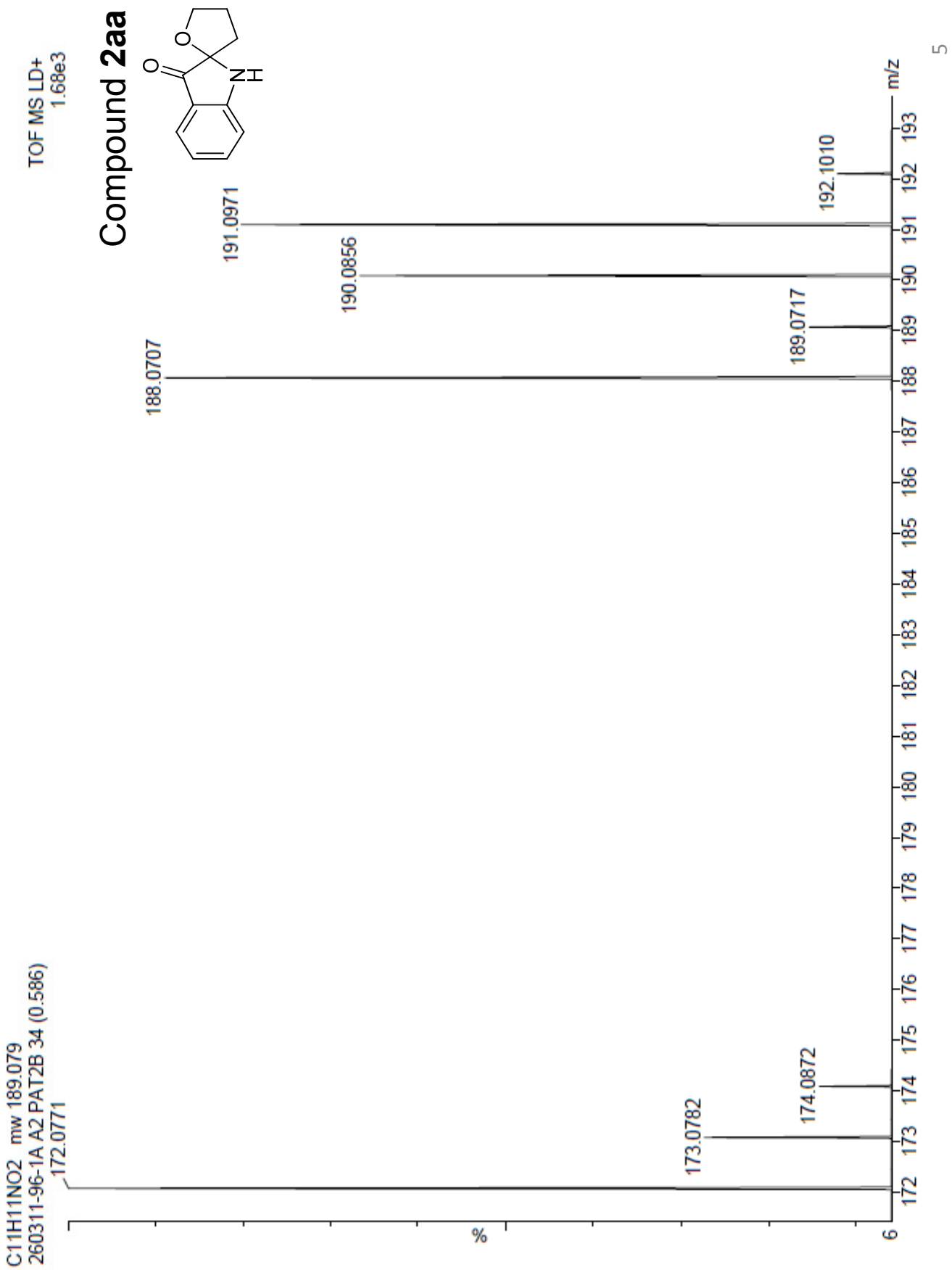

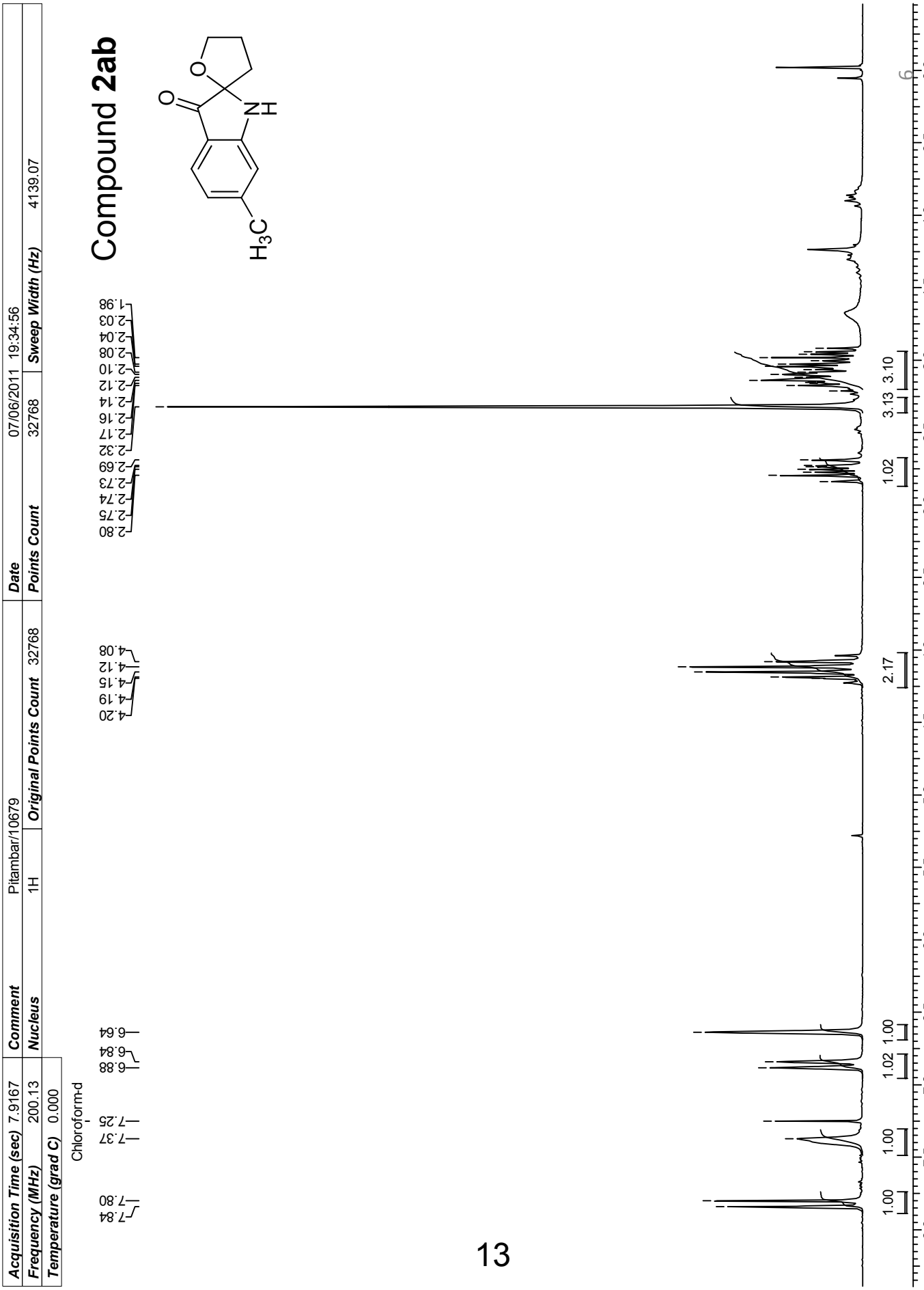
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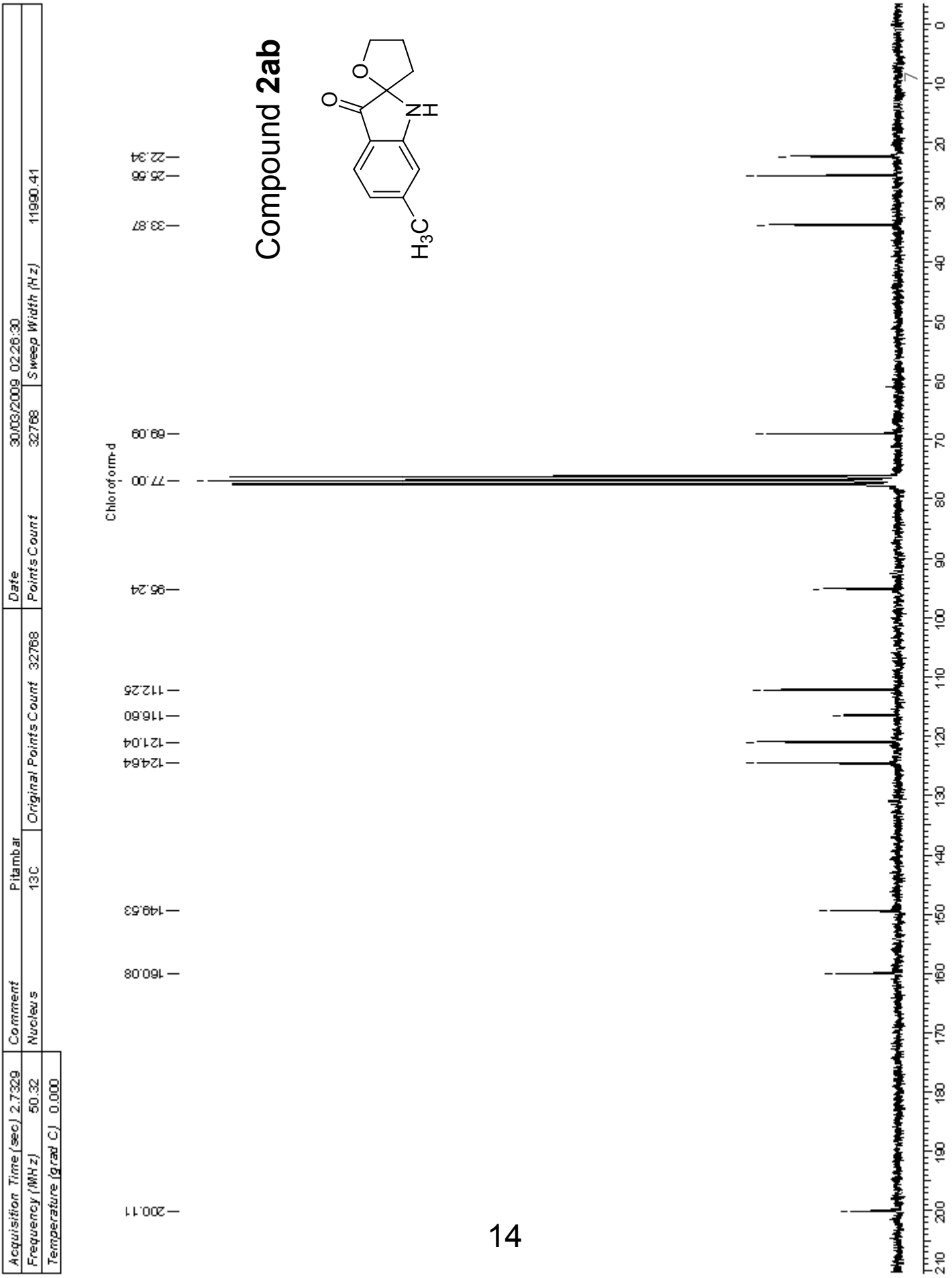
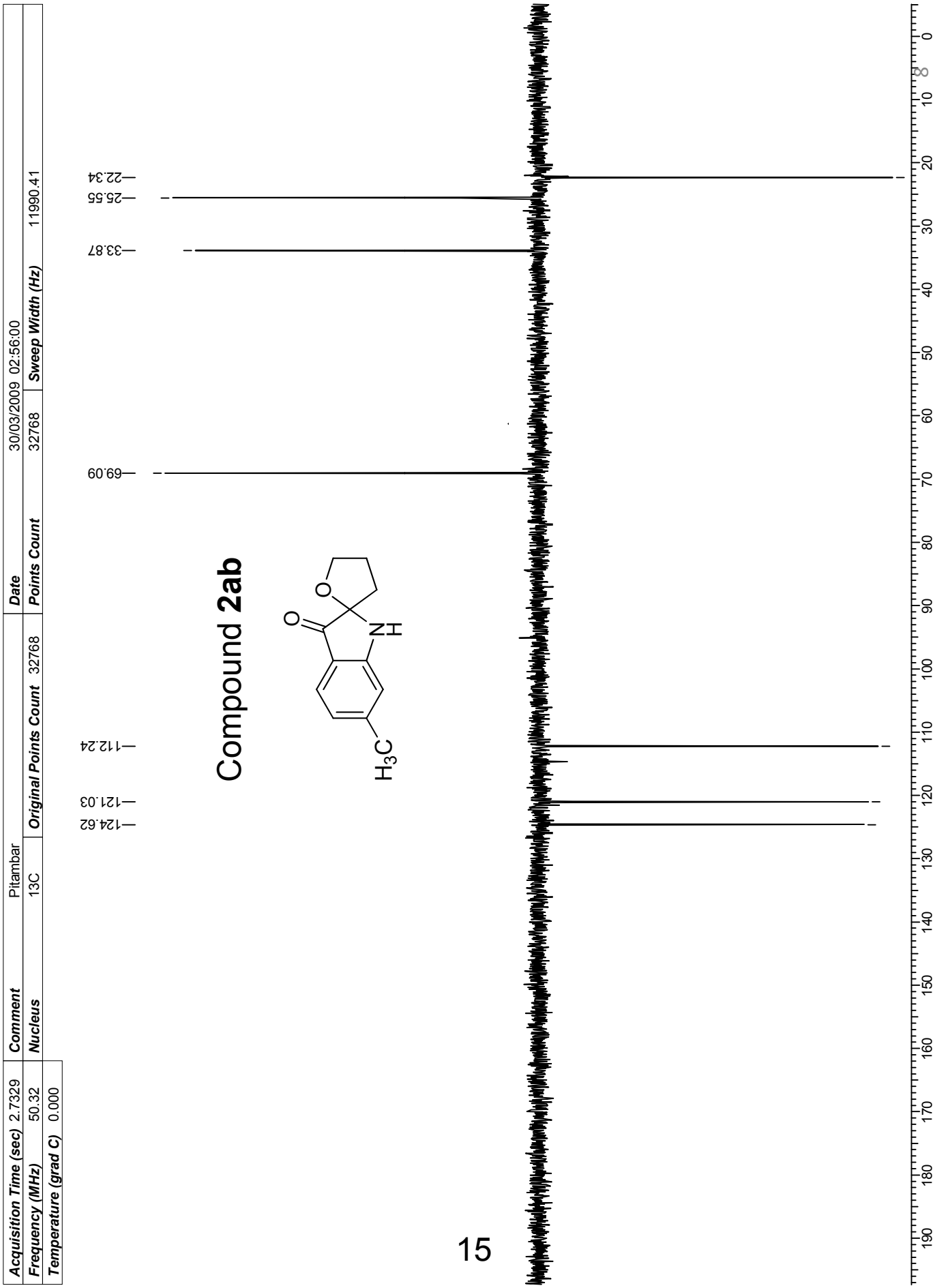
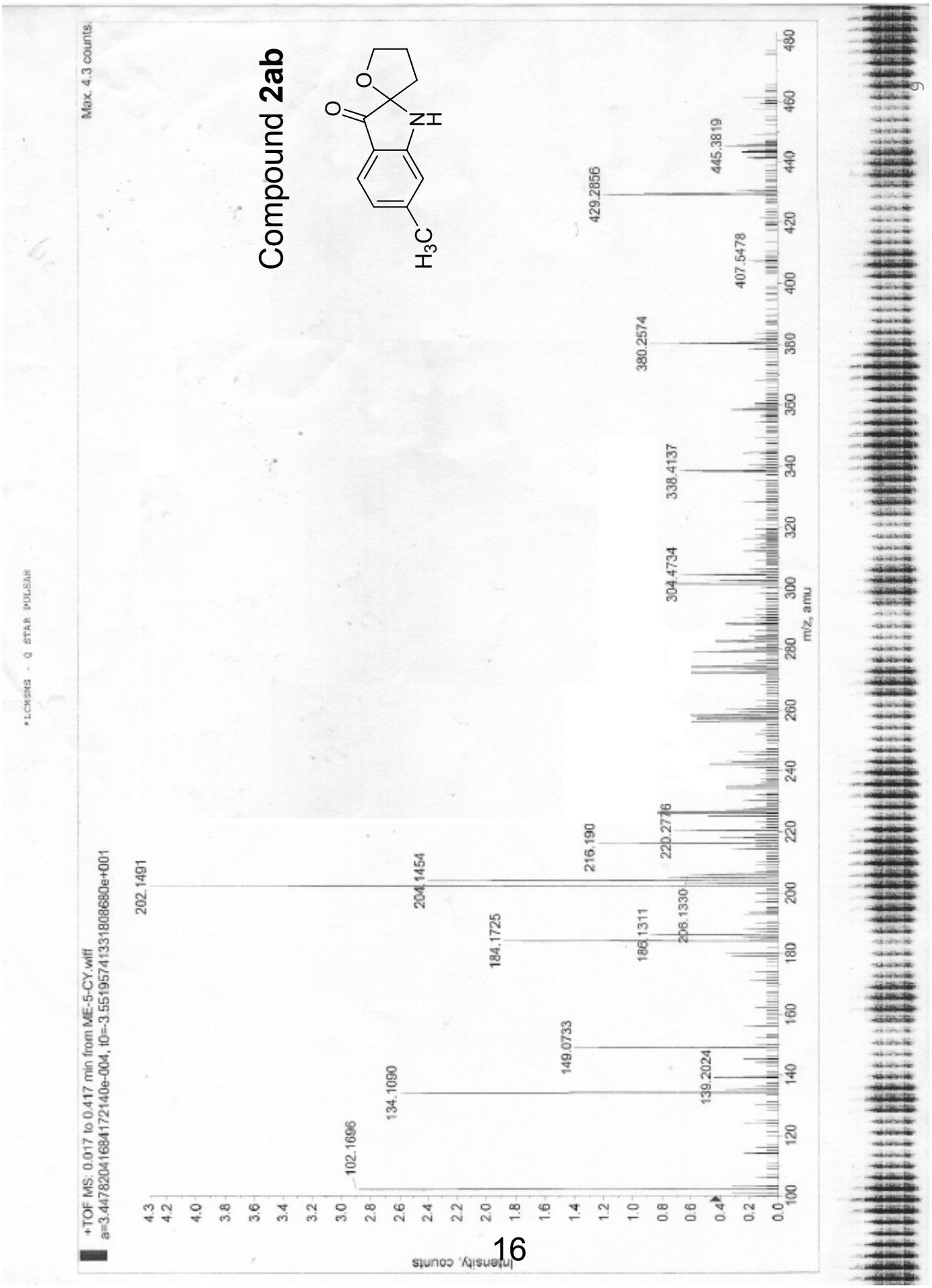
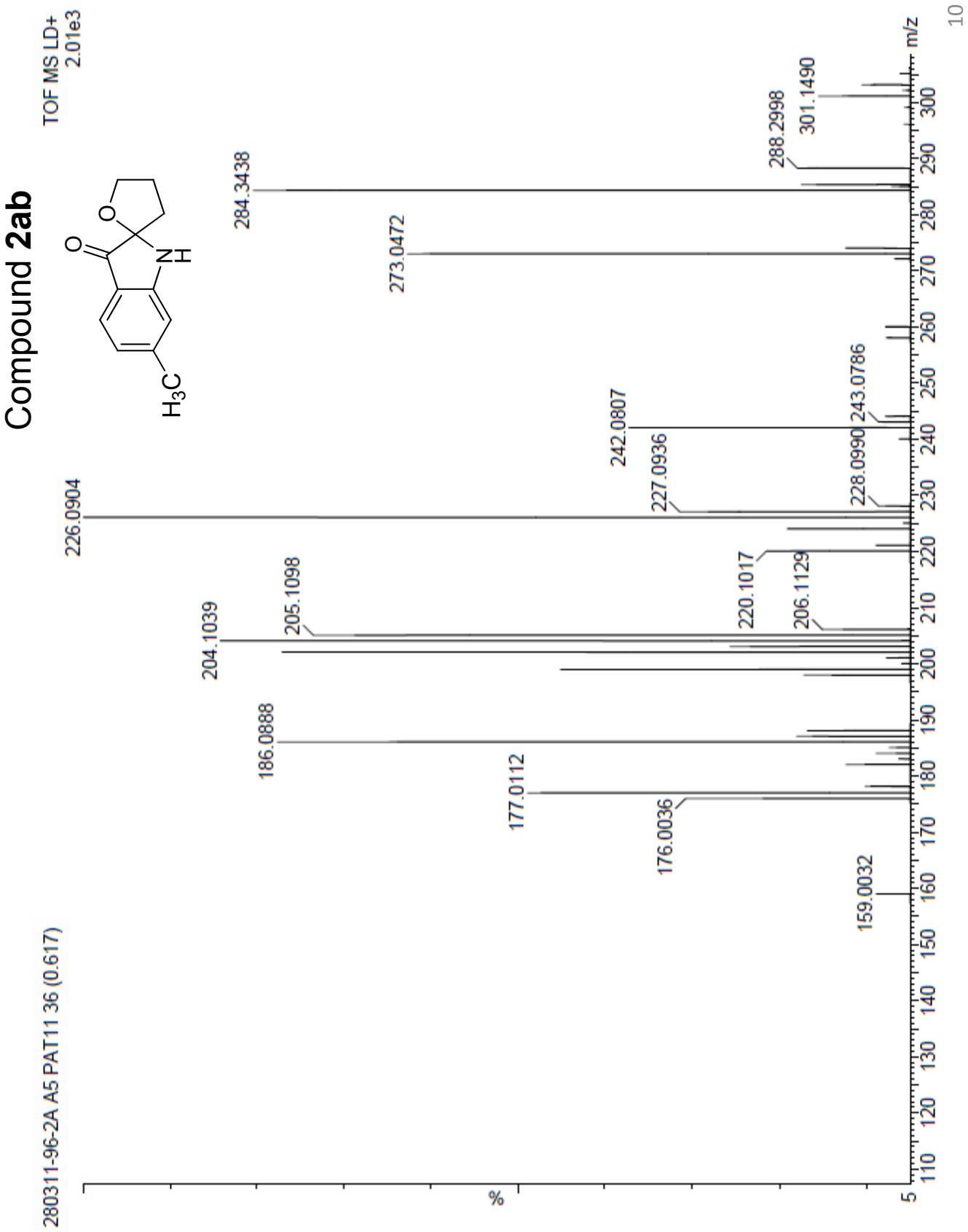
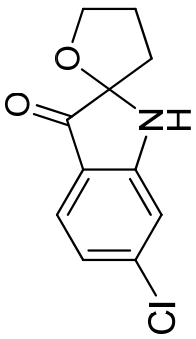








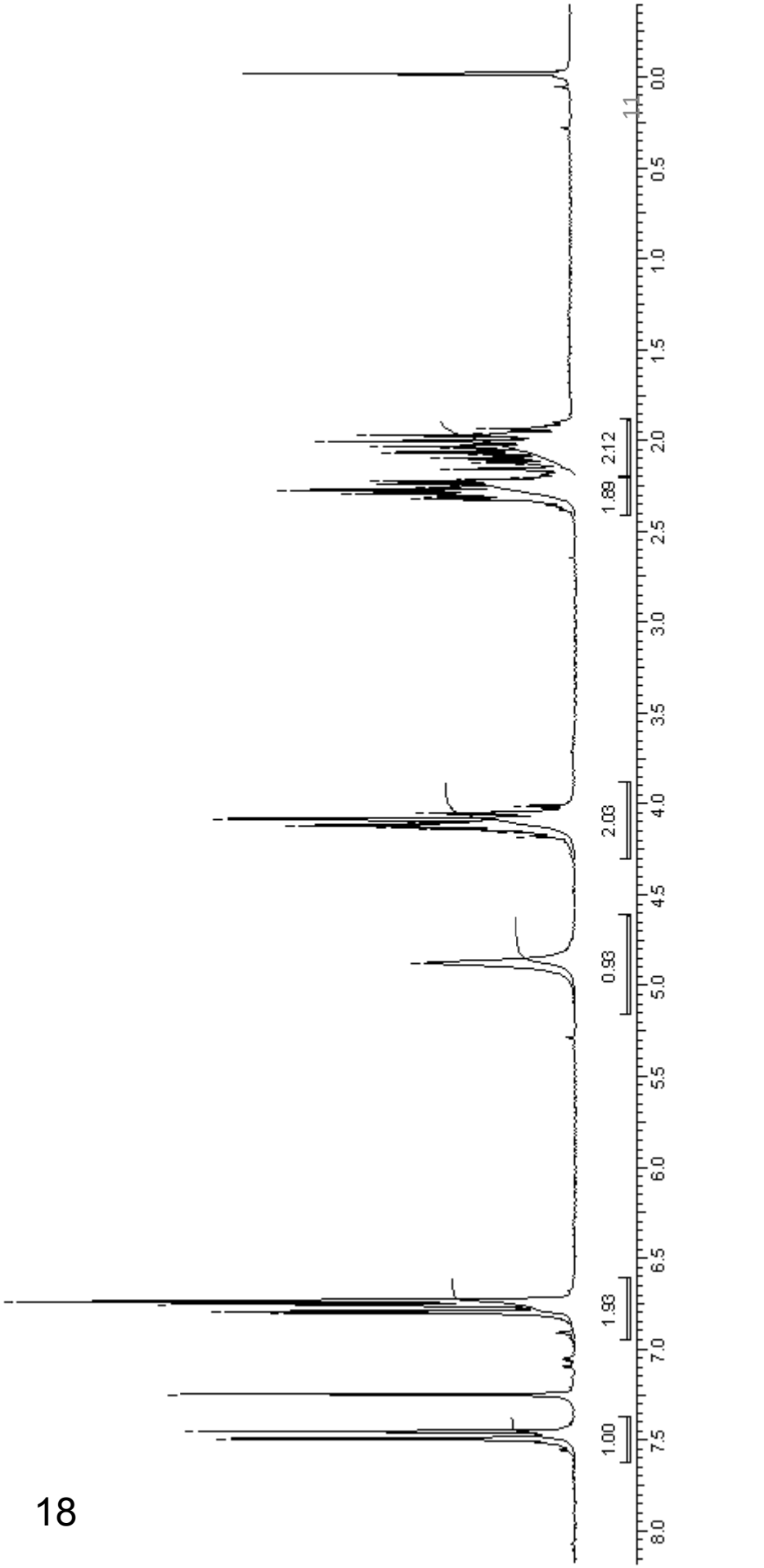
Compound **2ac**



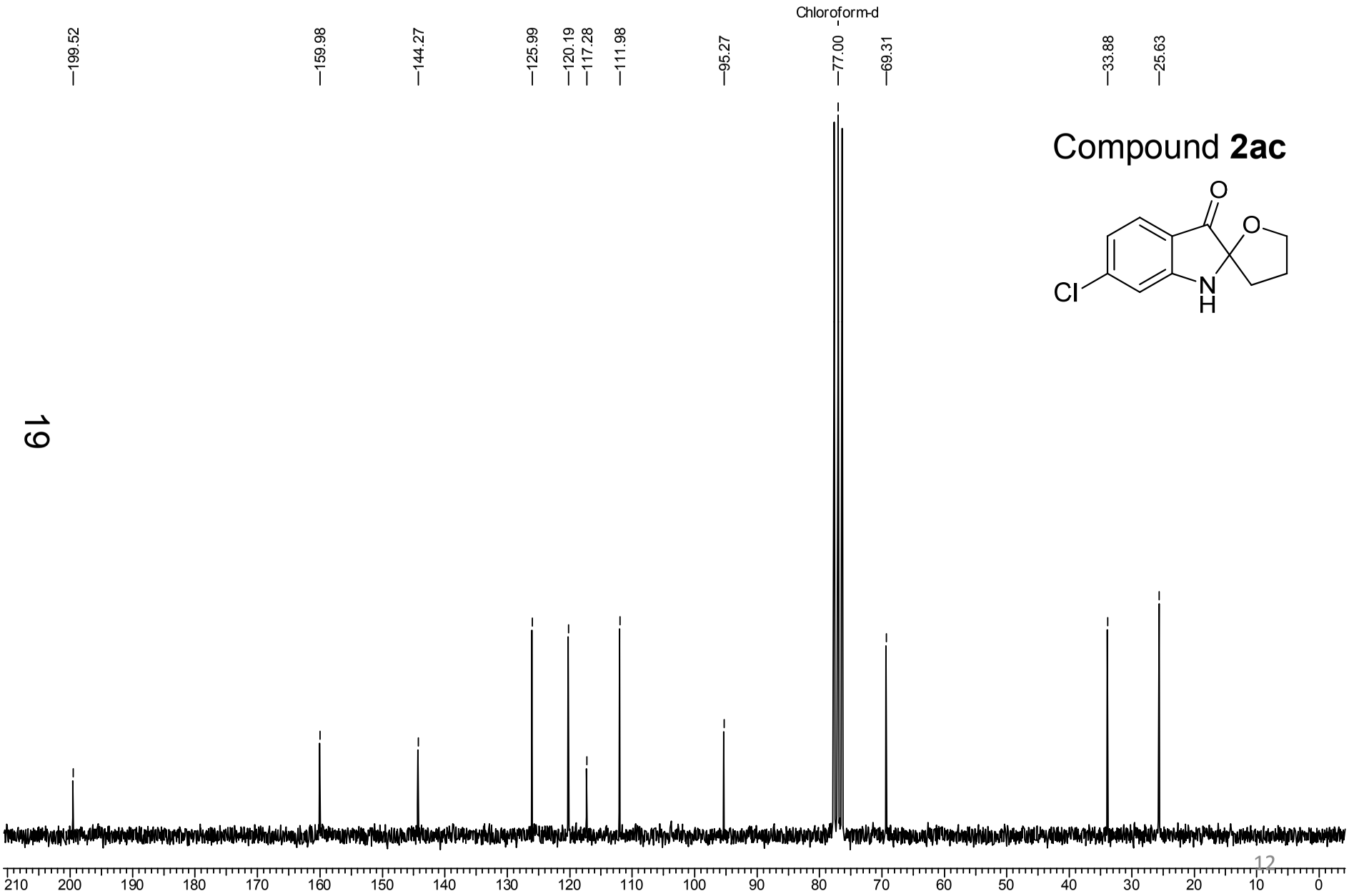
Acquisition Time (sec) 7.9167	*Comment*	Pitambar/10679	*Date*	14/06/2011 19:13:42	
Frequency (MHz) 200.13	*Nucleus* 1H	*Original Points Count* 32768	*Points Count* 32768	*Sweep Width (Hz)* 4139.07	
Temperature (grad C) 0.000					

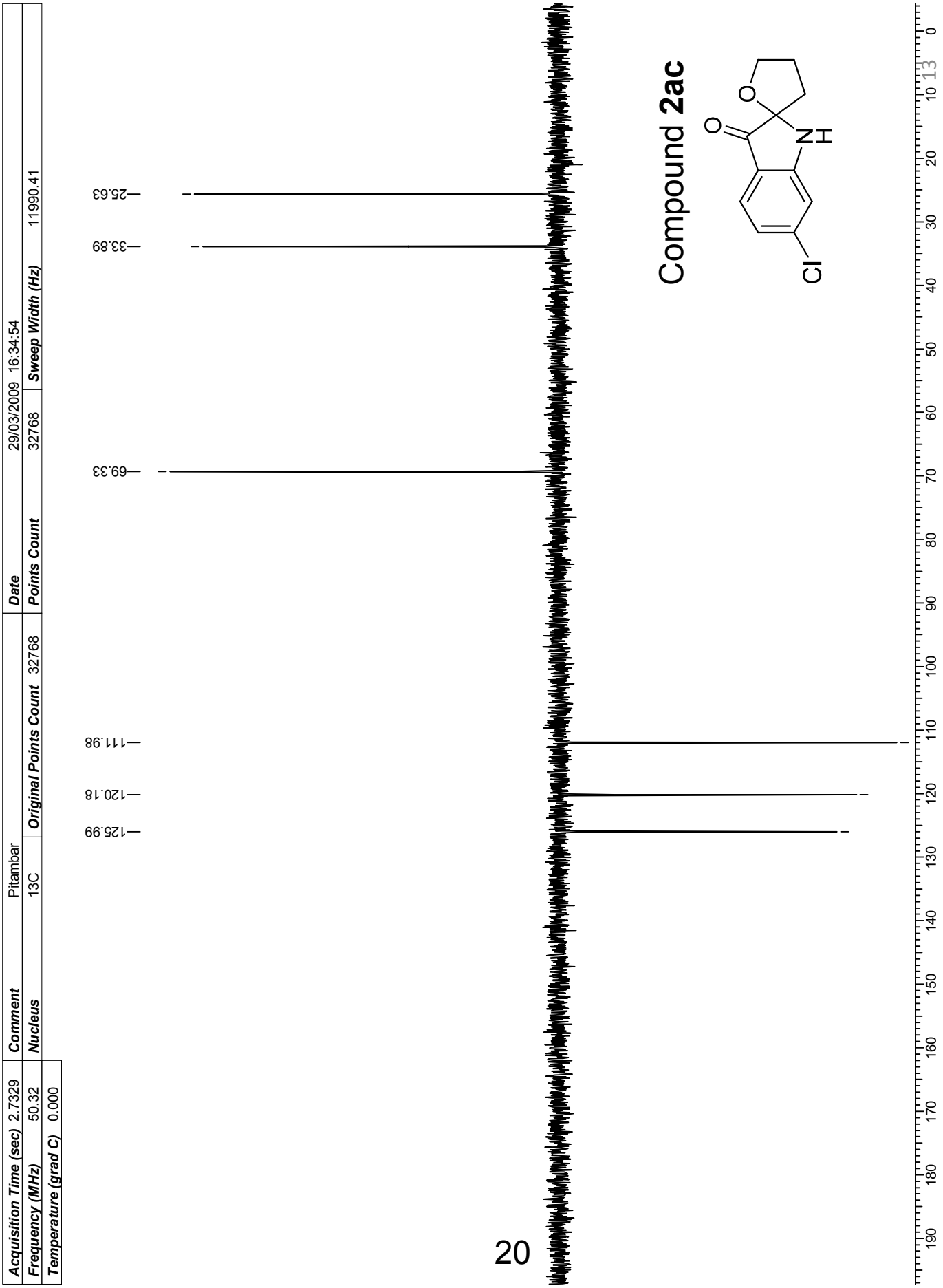
Chloroform-d

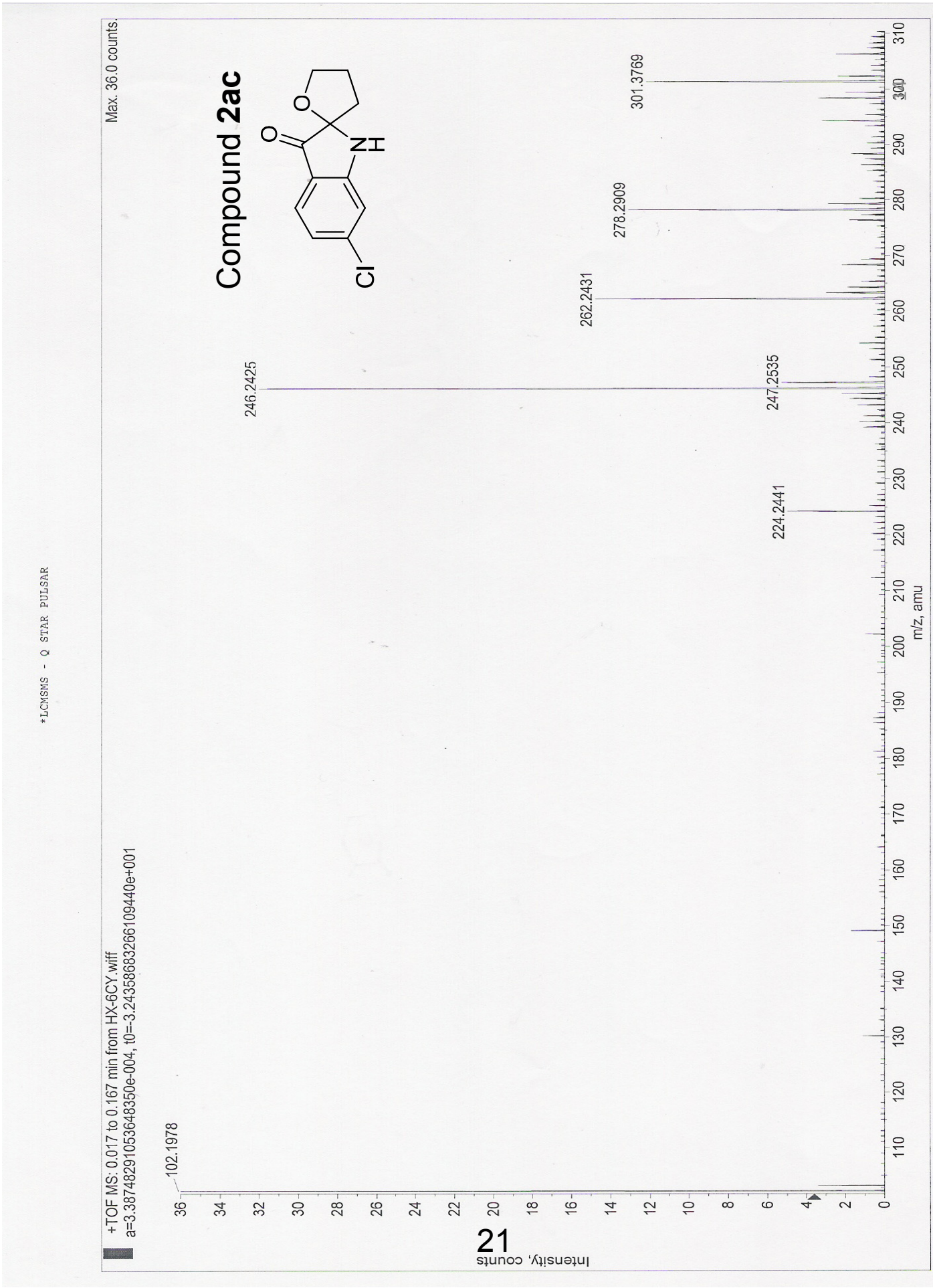
7.49 7.45 7.25 6.80 6.79 6.78 6.76 6.75 6.74 6.73 4.88 4.16 4.14 4.13 4.12 4.10 4.08 4.05 4.04 4.01 2.33 2.29 2.28 2.26 2.24 2.22 2.07 2.03 2.01 1.97



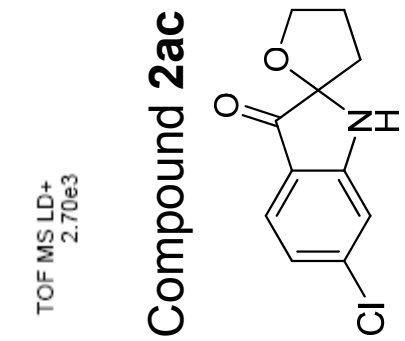
Acquisition Time (sec)	2.7329	***Comment***	Pitambar		***Date***	29/03/2009 16:10:58	
Frequency (MHz)	50.32	***Nucleus***	13C	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 11990.41
Temperature (grad C)	0.000						

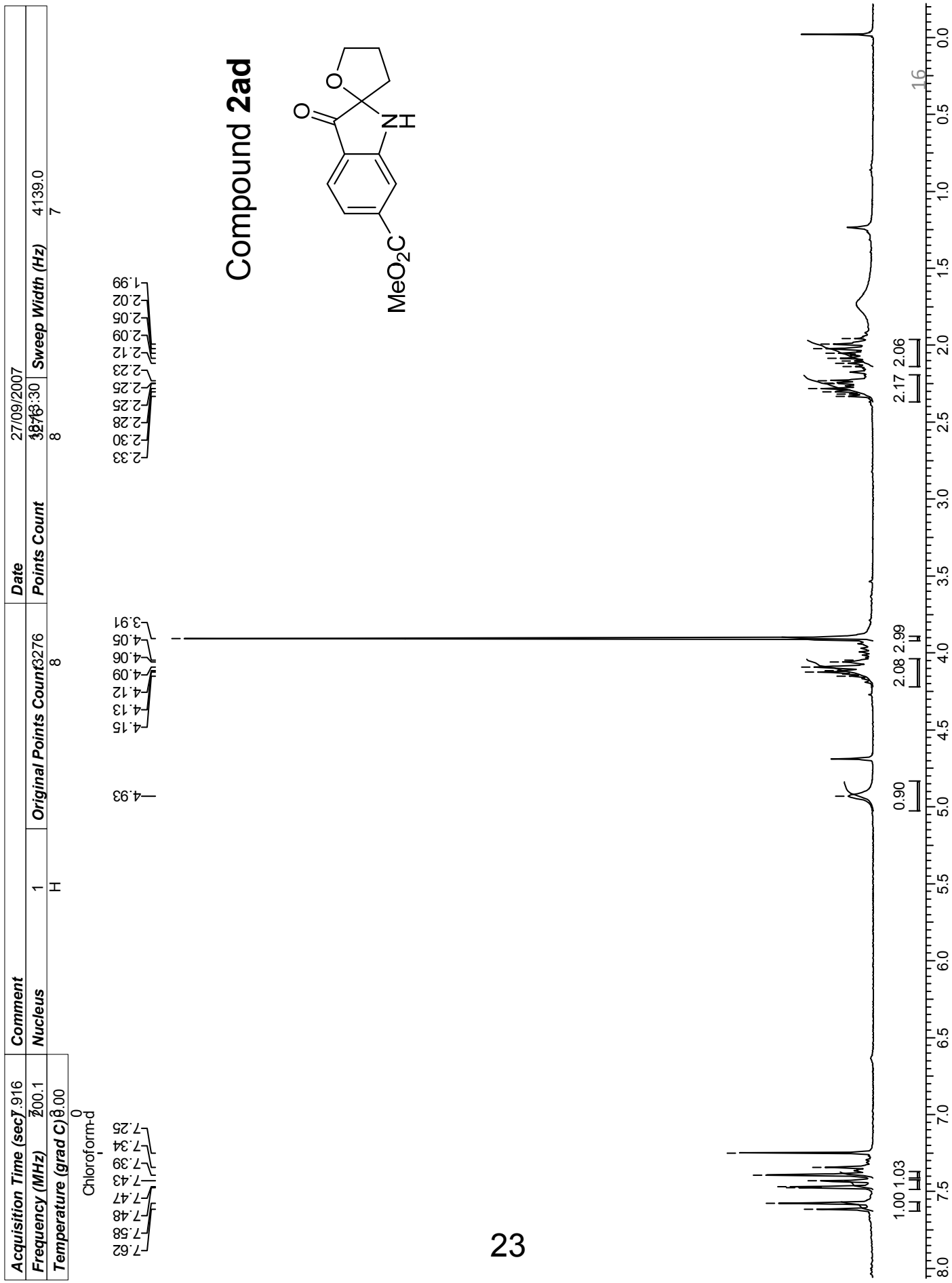






Compound **2ac**



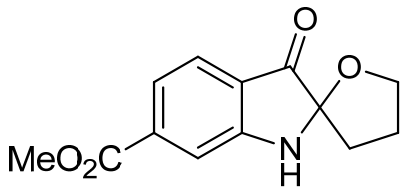

Compound 2ad
MeO2C
O
NH
Acquisition Time (sec) 7.916
Frequency (MHz) 200.1
Temperature (grad C) 8.00
Chloroform-d
Comment
Nucleus 1H
Original Points Count 32768
Points Count 8
Date 27/09/2007
Sweep Width (Hz) 4139.0
7.62
7.58
7.48
7.47
7.43
7.39
7.34
7.25
4.93
4.15
4.13
4.12
4.09
4.06
4.05
3.91
2.33
2.30
2.28
2.25
2.23
2.12
2.09
2.05
2.02
1.99
1.00
1.03
0.90
2.08
2.99
2.17
2.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

Acquisition Time (sec)	1.363	***Date***	09/10/2007		***Frequency (MHz)***	100.61	***Nucleus***	13C
Original Points Count	32768	***Points Count***	32768	***Sweep Width (Hz)***	24038.46		***Temperature (grad C)***	0.00

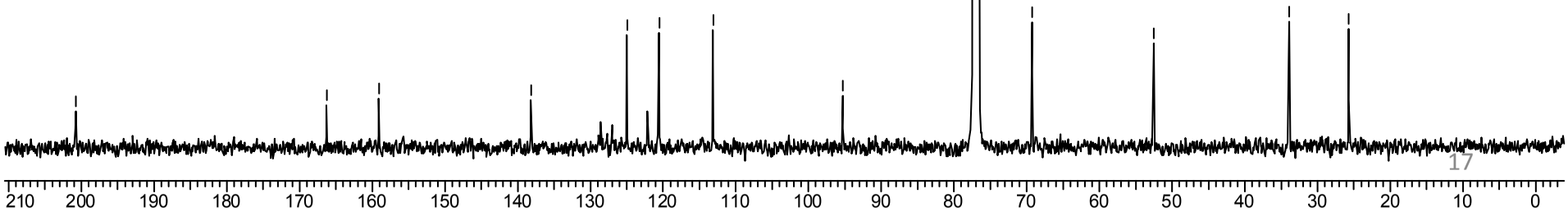
Chloroform-d

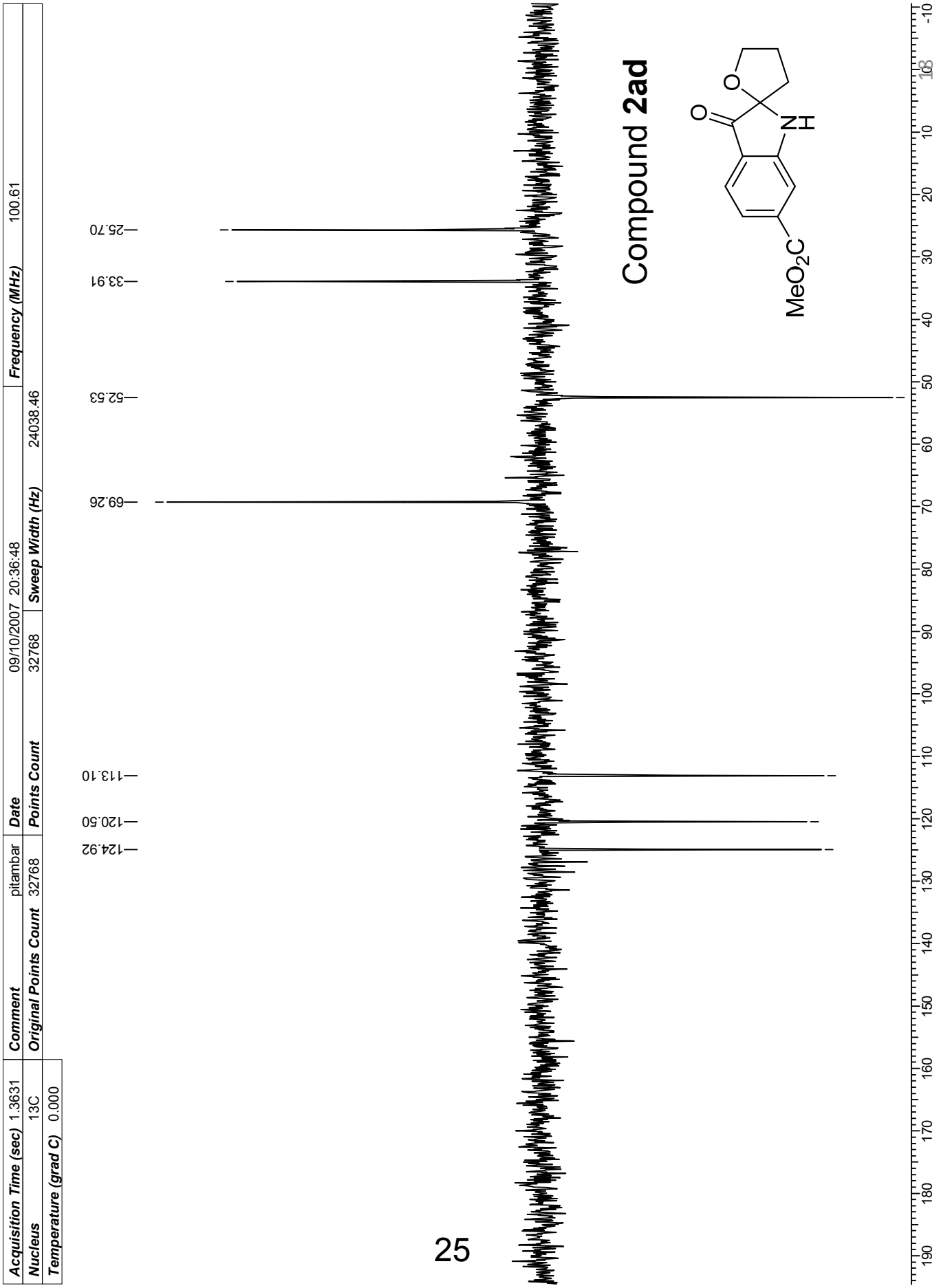
200.74 166.26 159.07 138.12 124.92 120.51 113.11 95.28 77.00 69.26 52.53 33.91 25.70

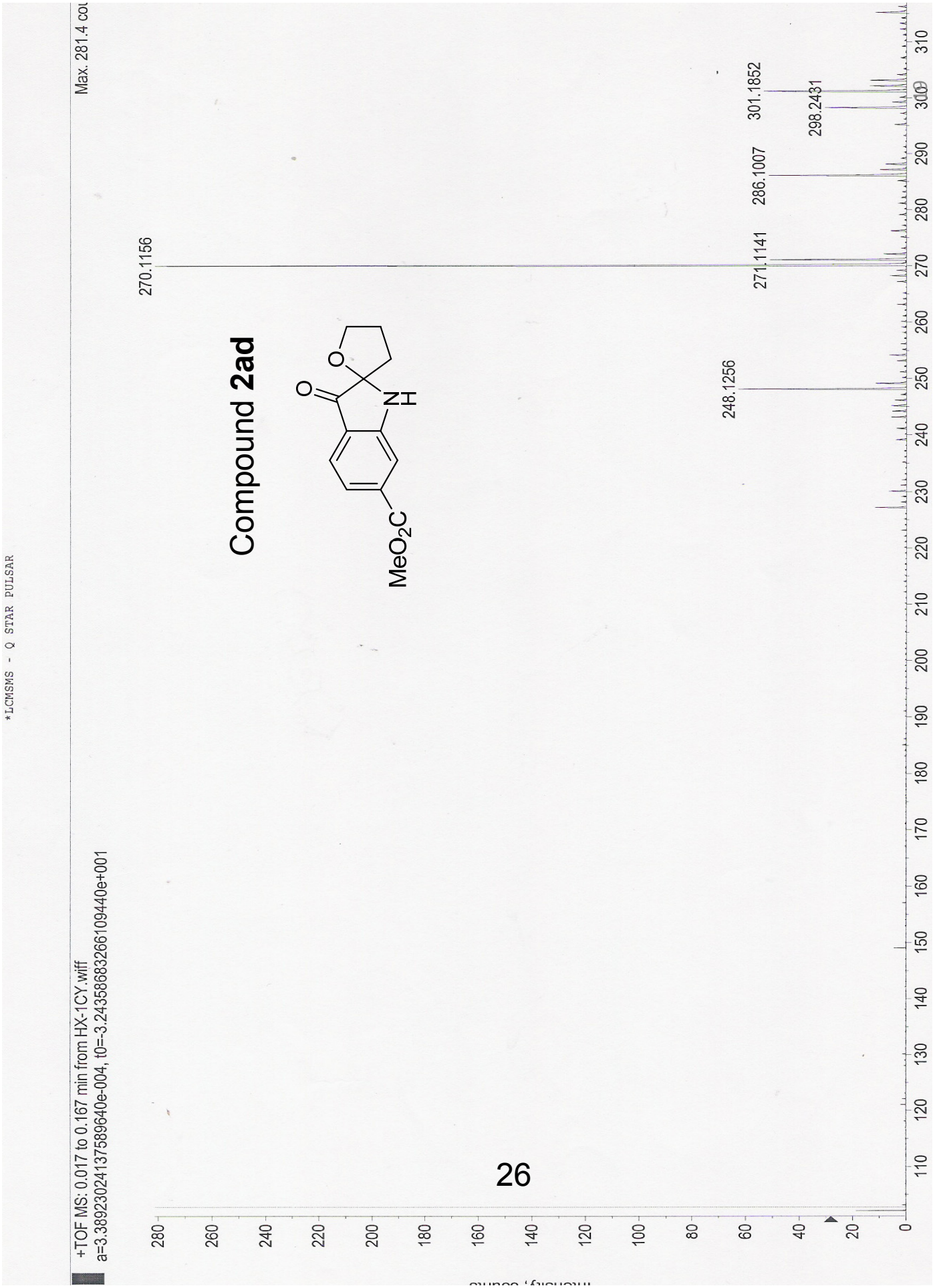
Compound **2ad**

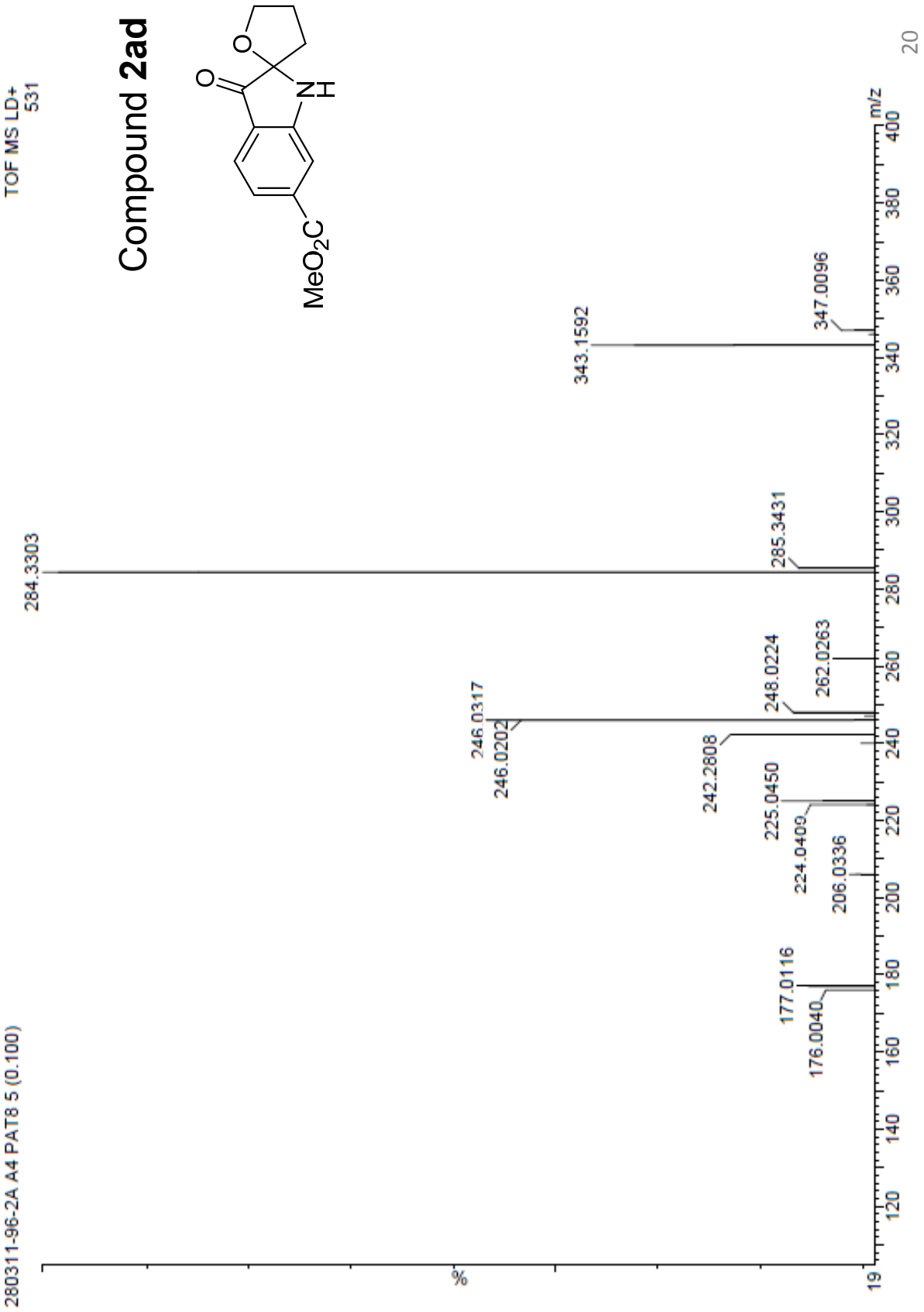


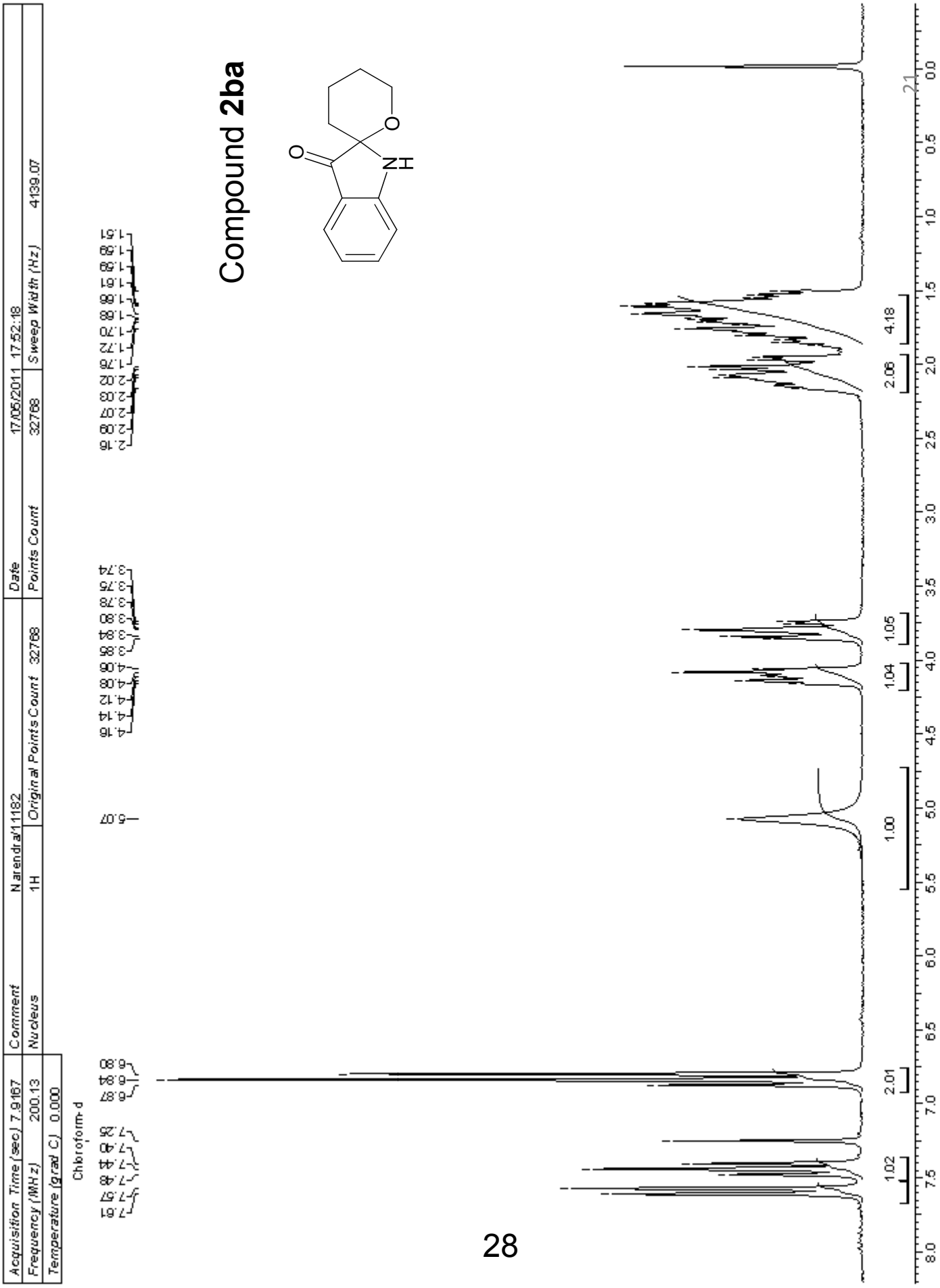
24

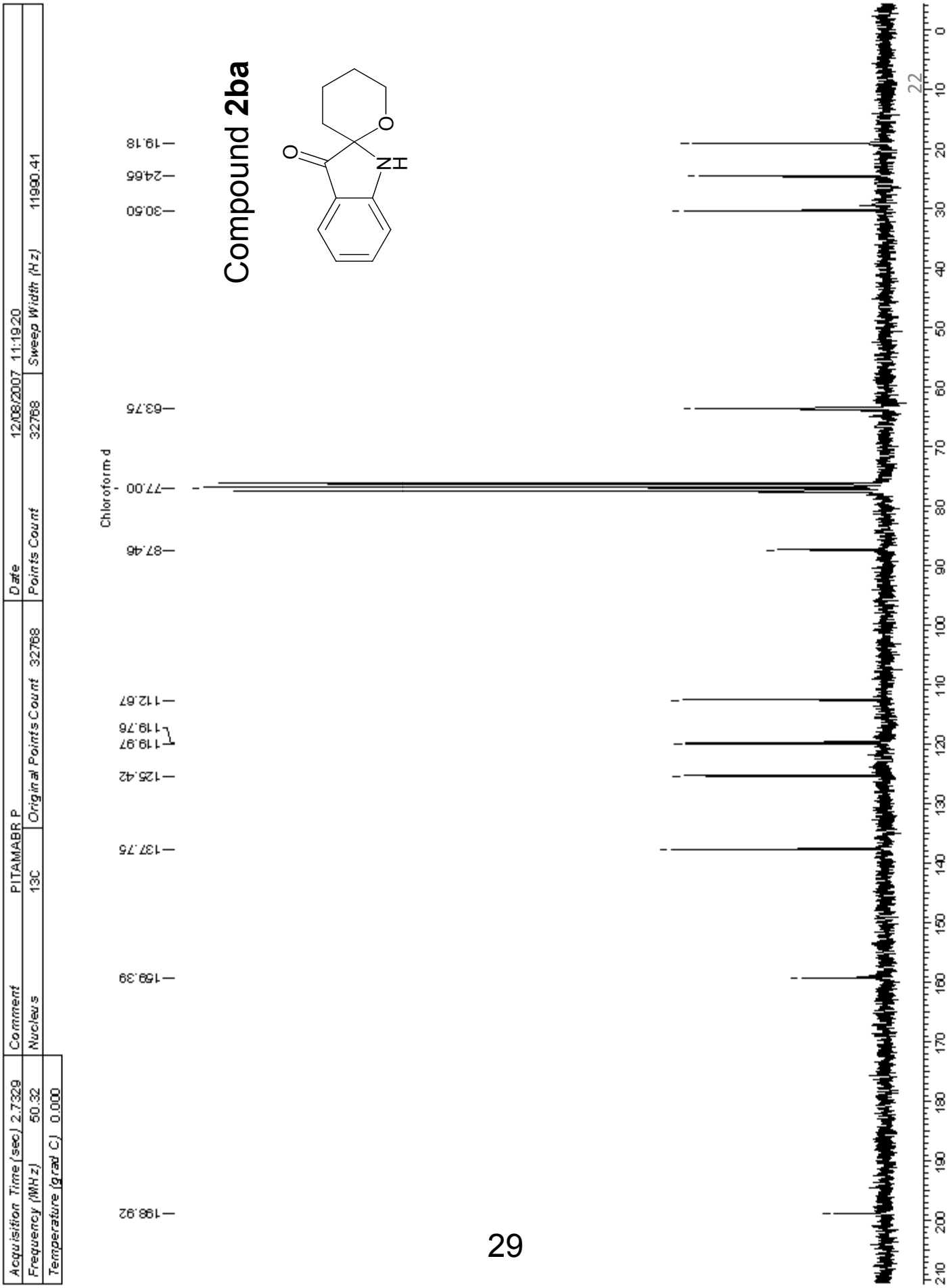


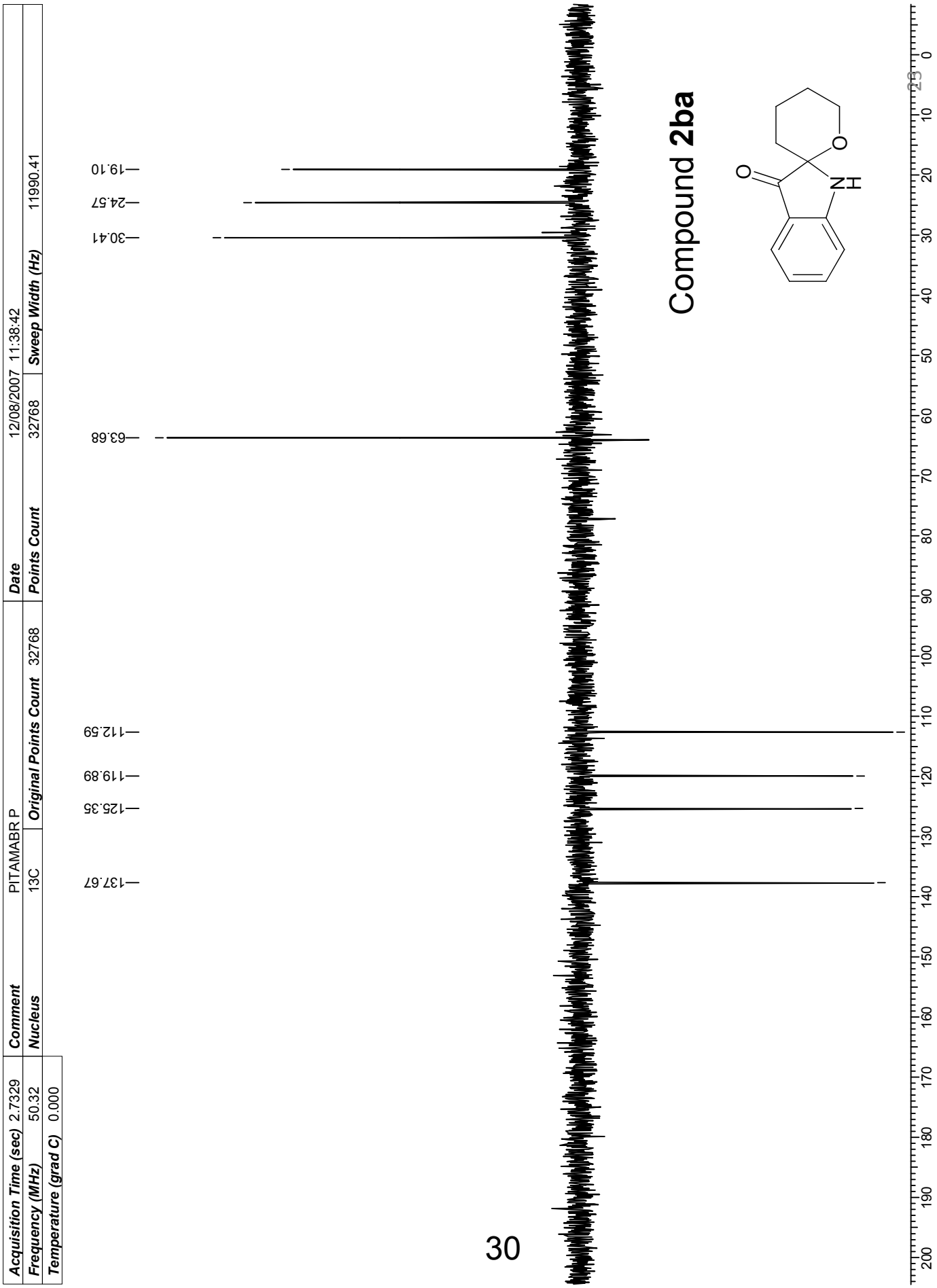


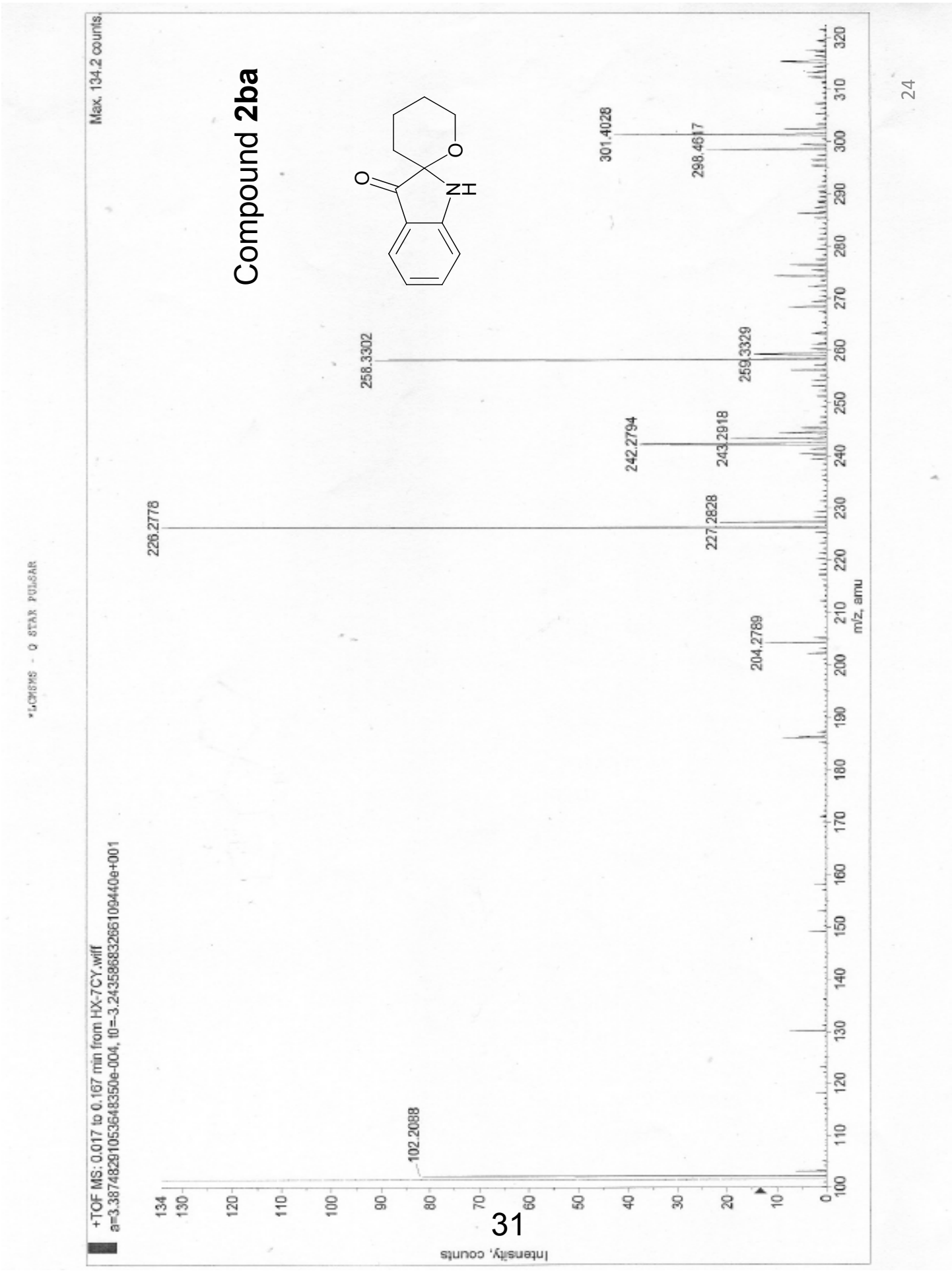


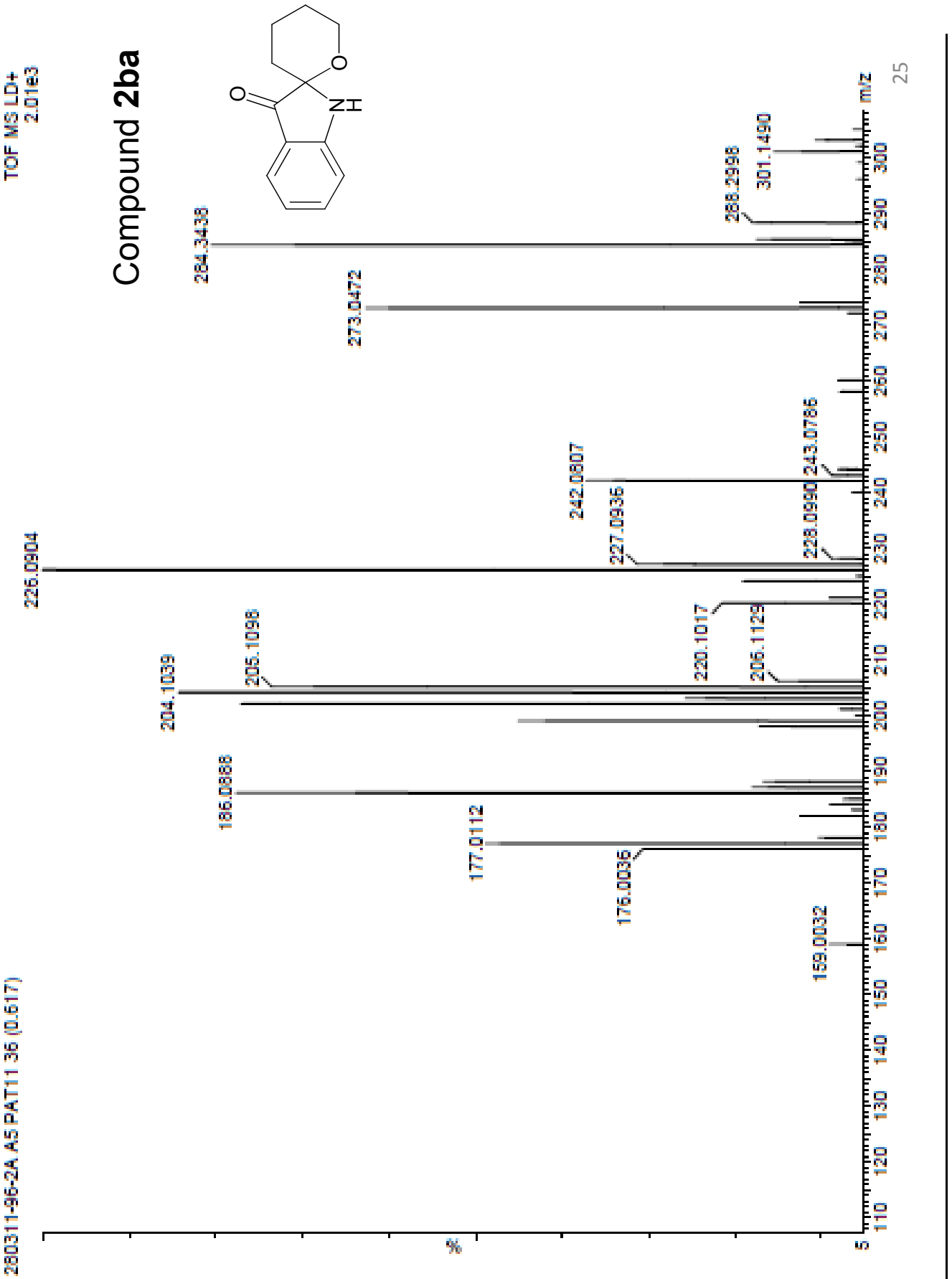


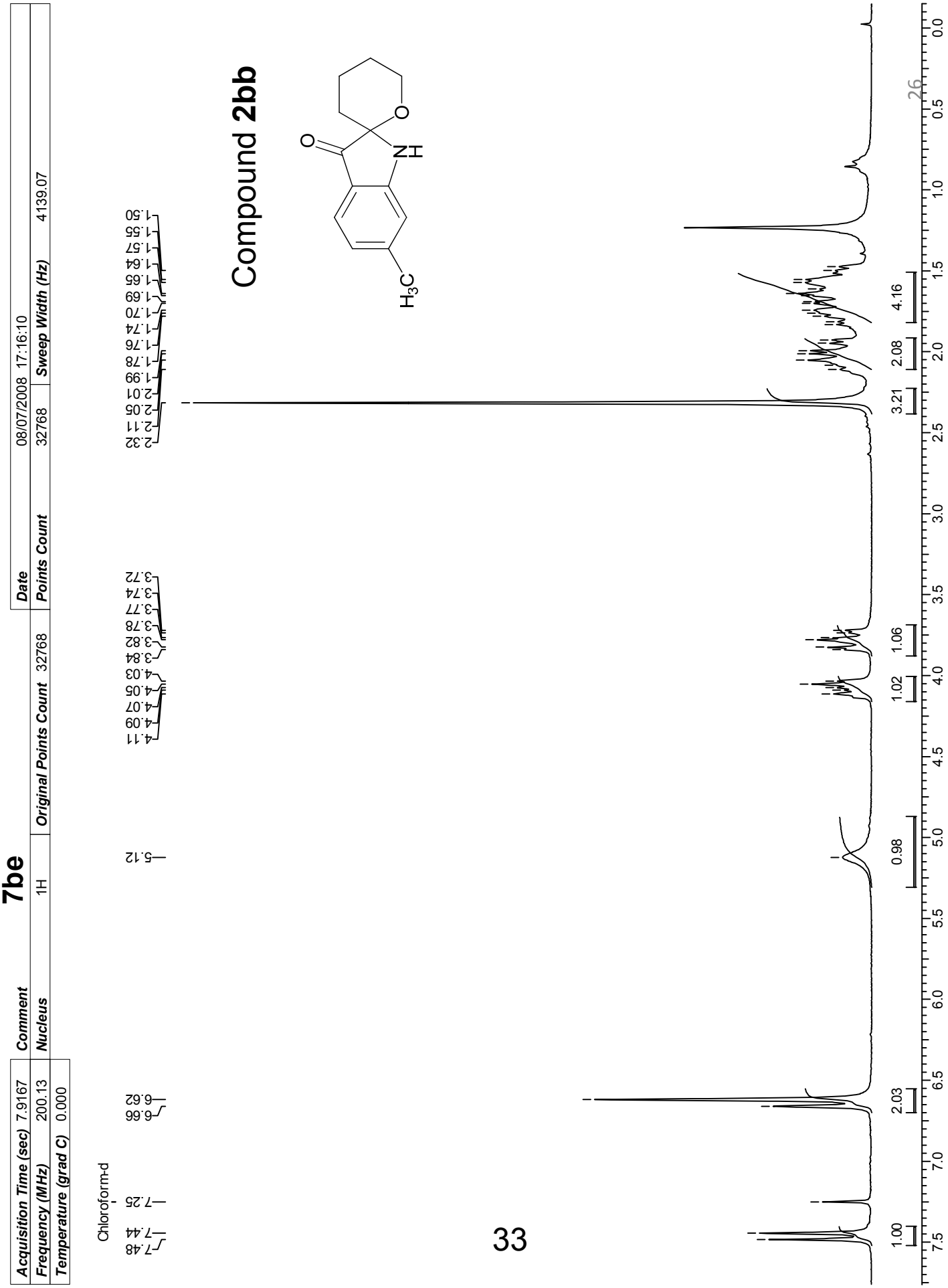


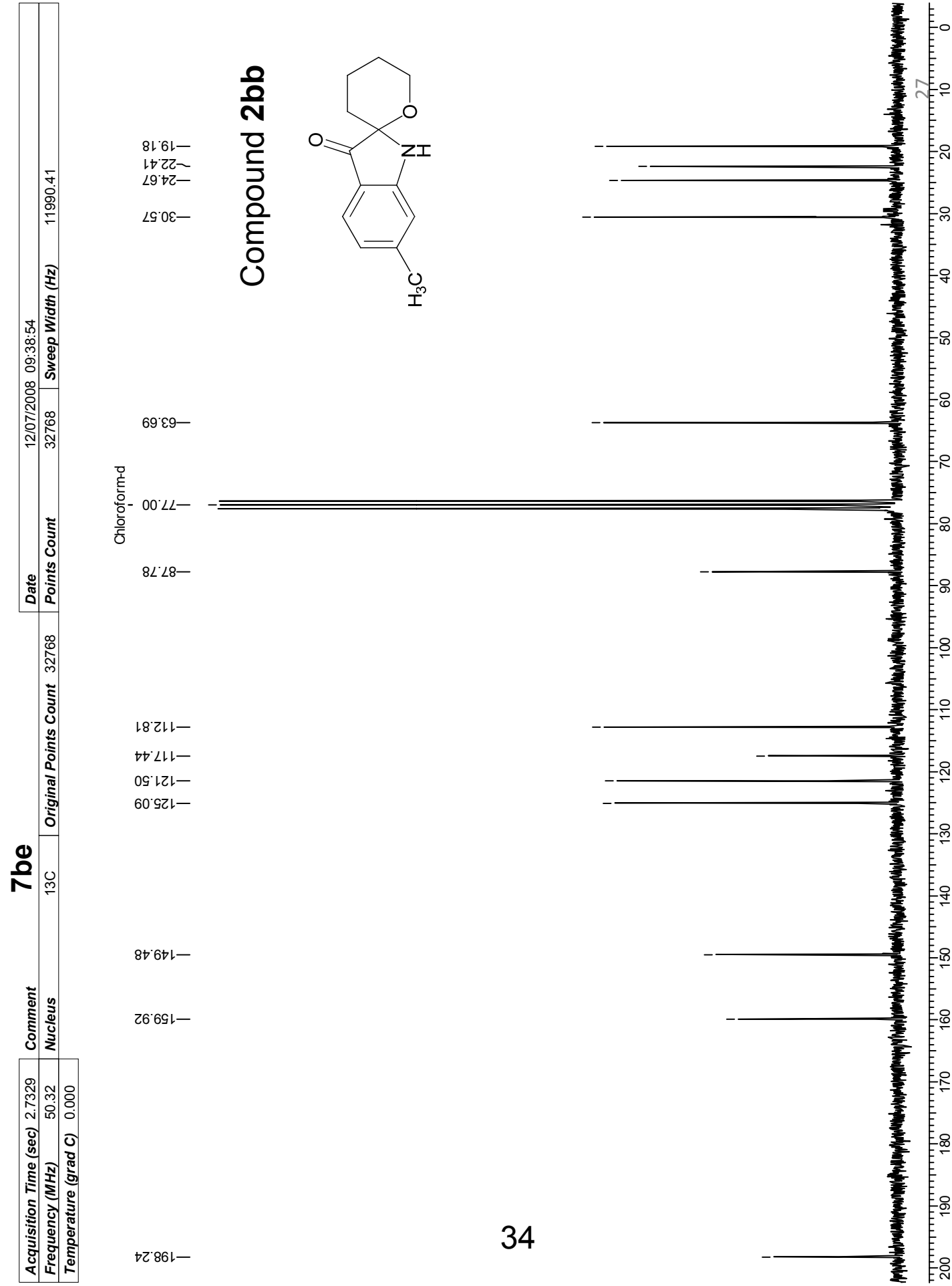


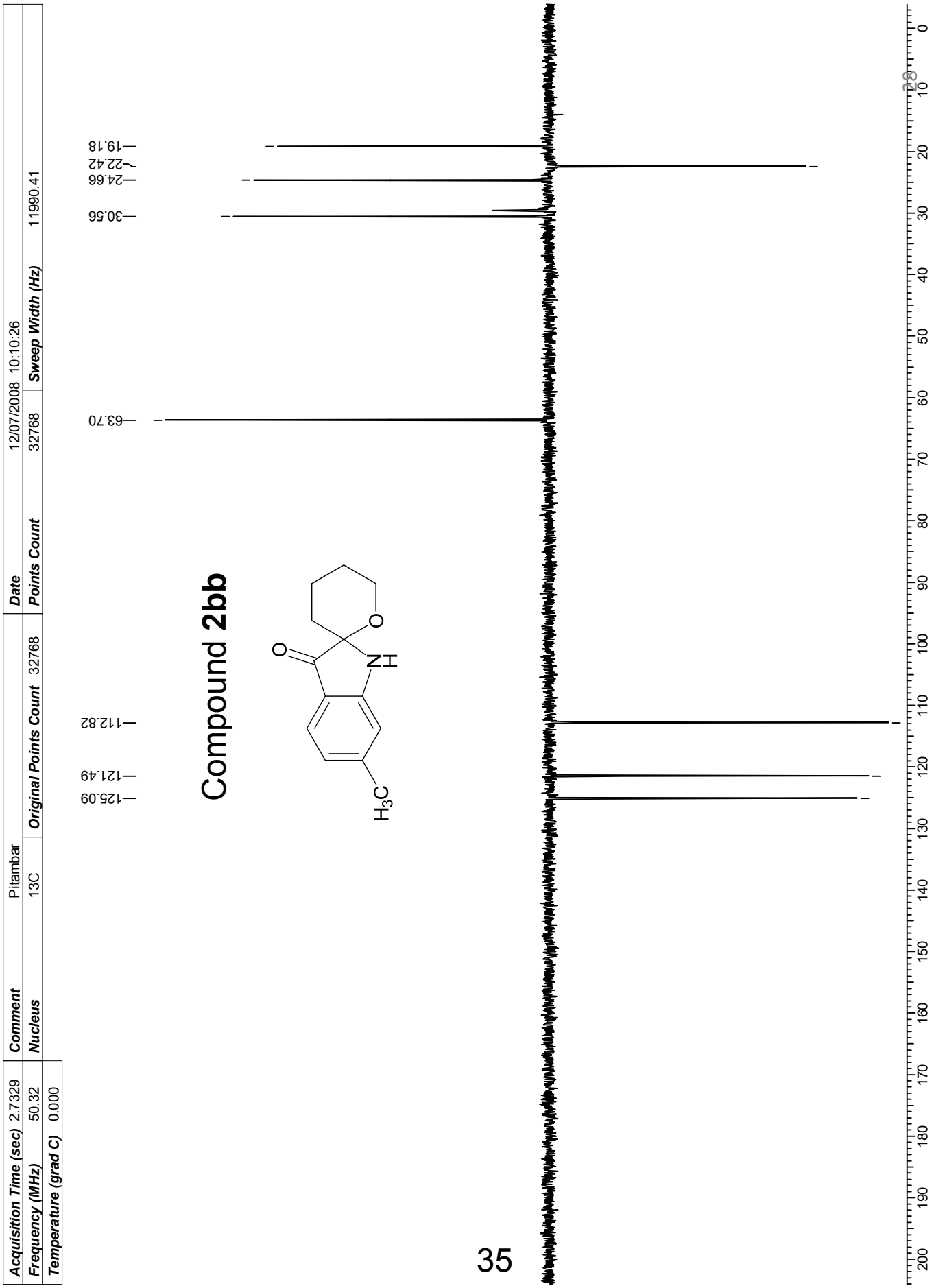


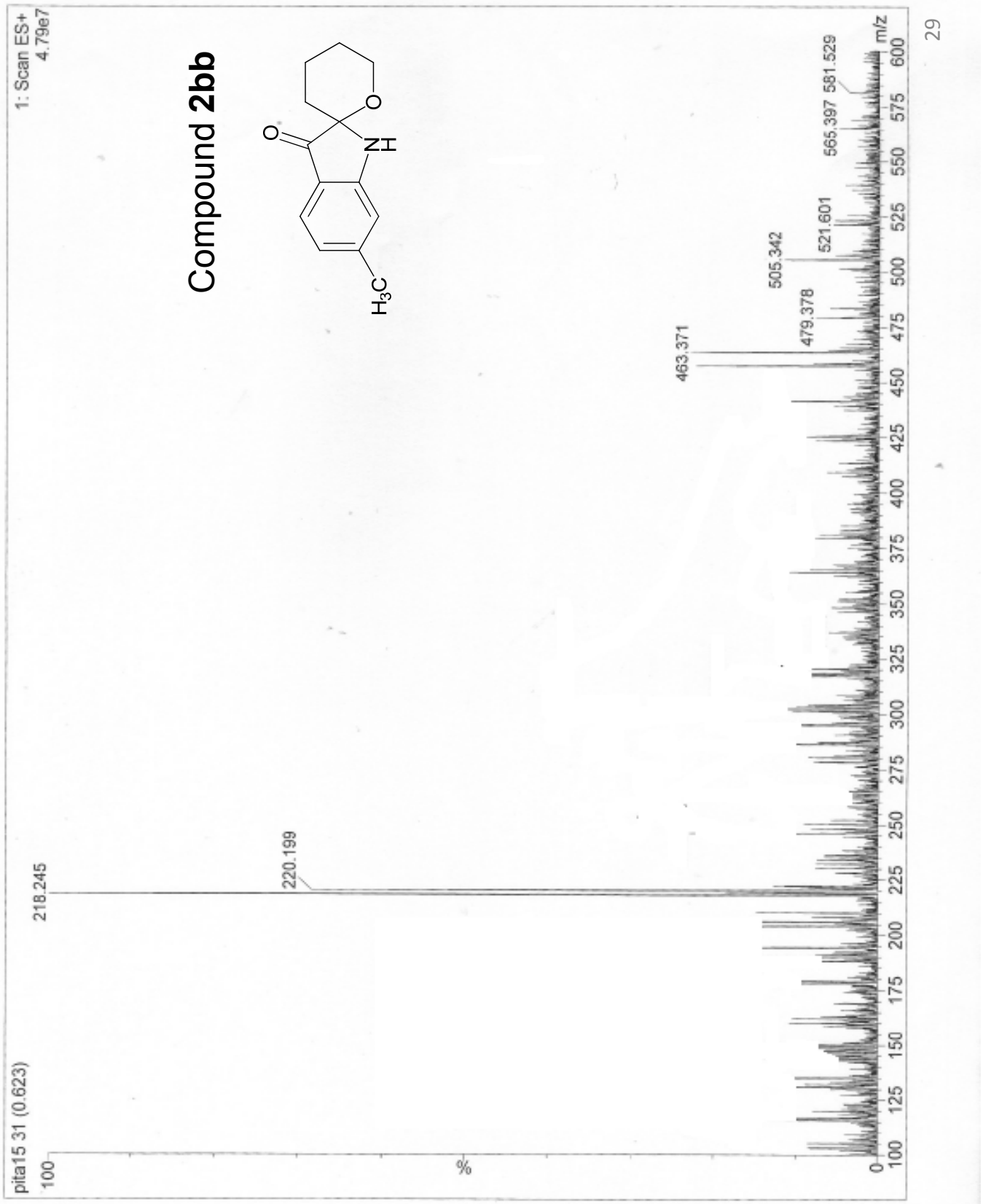

Compound 2ba
+TOF MS: 0.017 to 0.167 min from HX-7CY.wiff
a=3.38748291053648350e-004, t0=-3.24358683266109440e+001
Max. 134.2 counts.
Intensity, counts
m/z, amu
226.2778
258.3302
301.4026
298.4617
259.3329
242.2794
243.2818
227.2828
204.2789
102.2088
O
NH
O

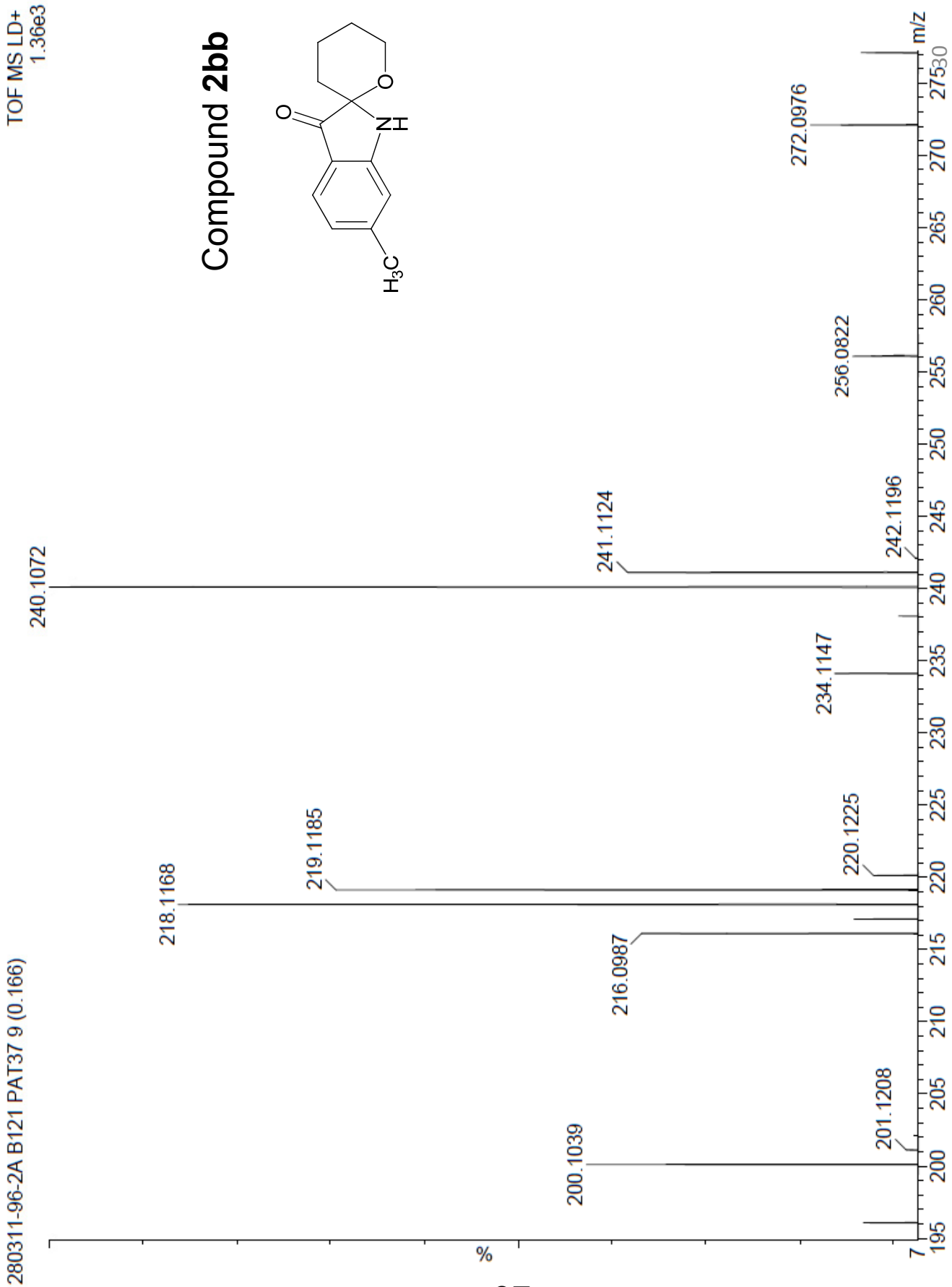




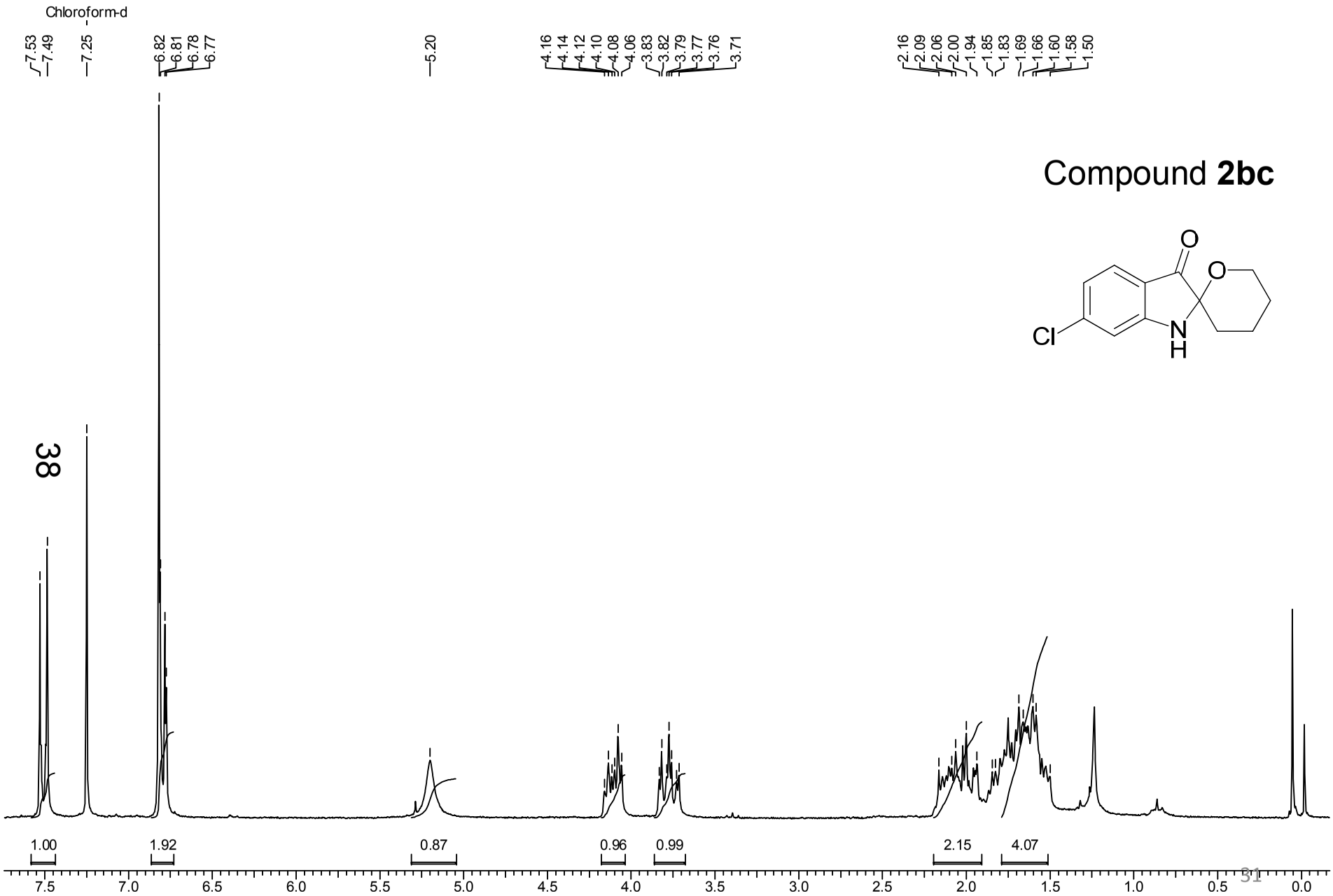




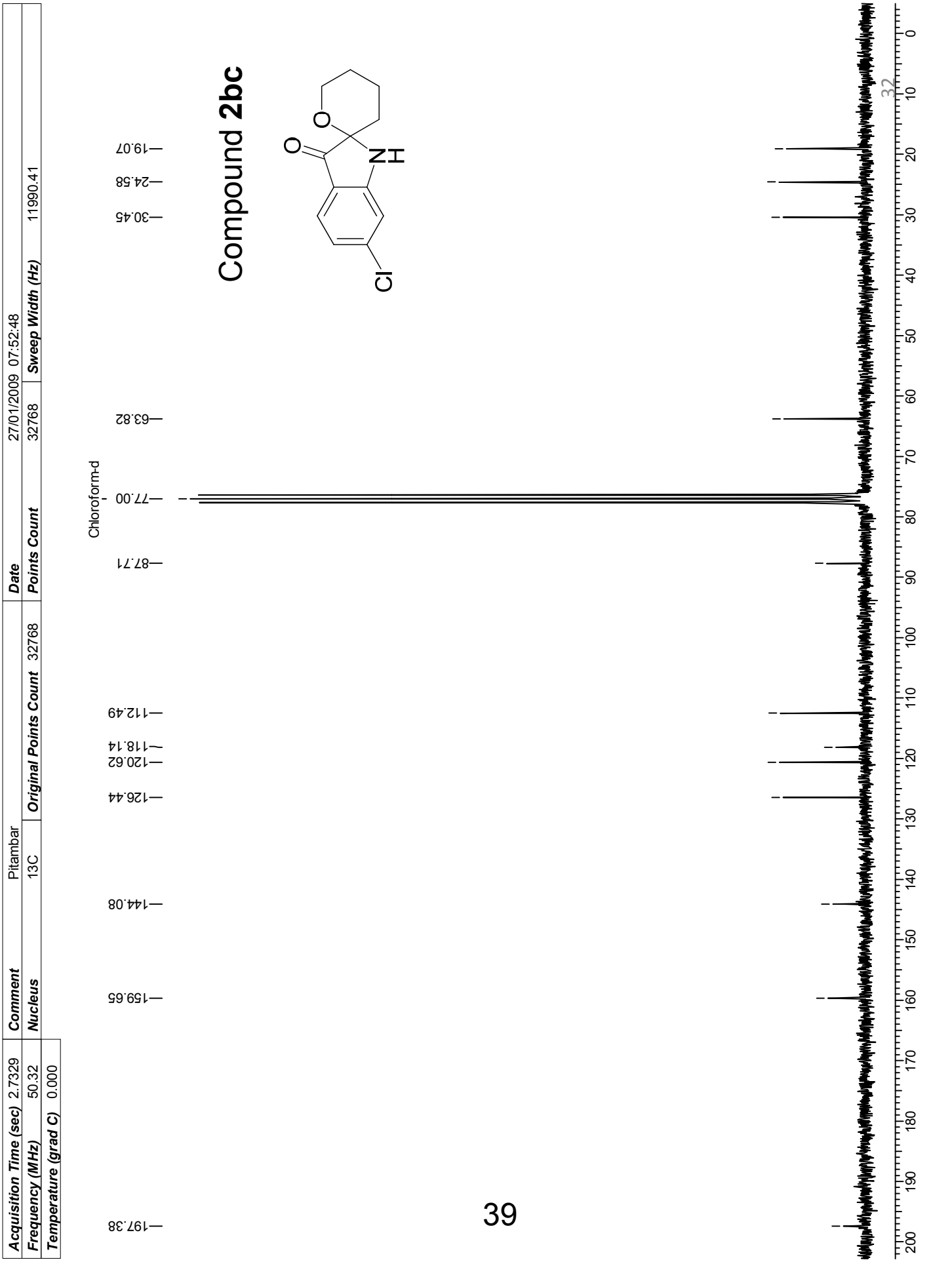


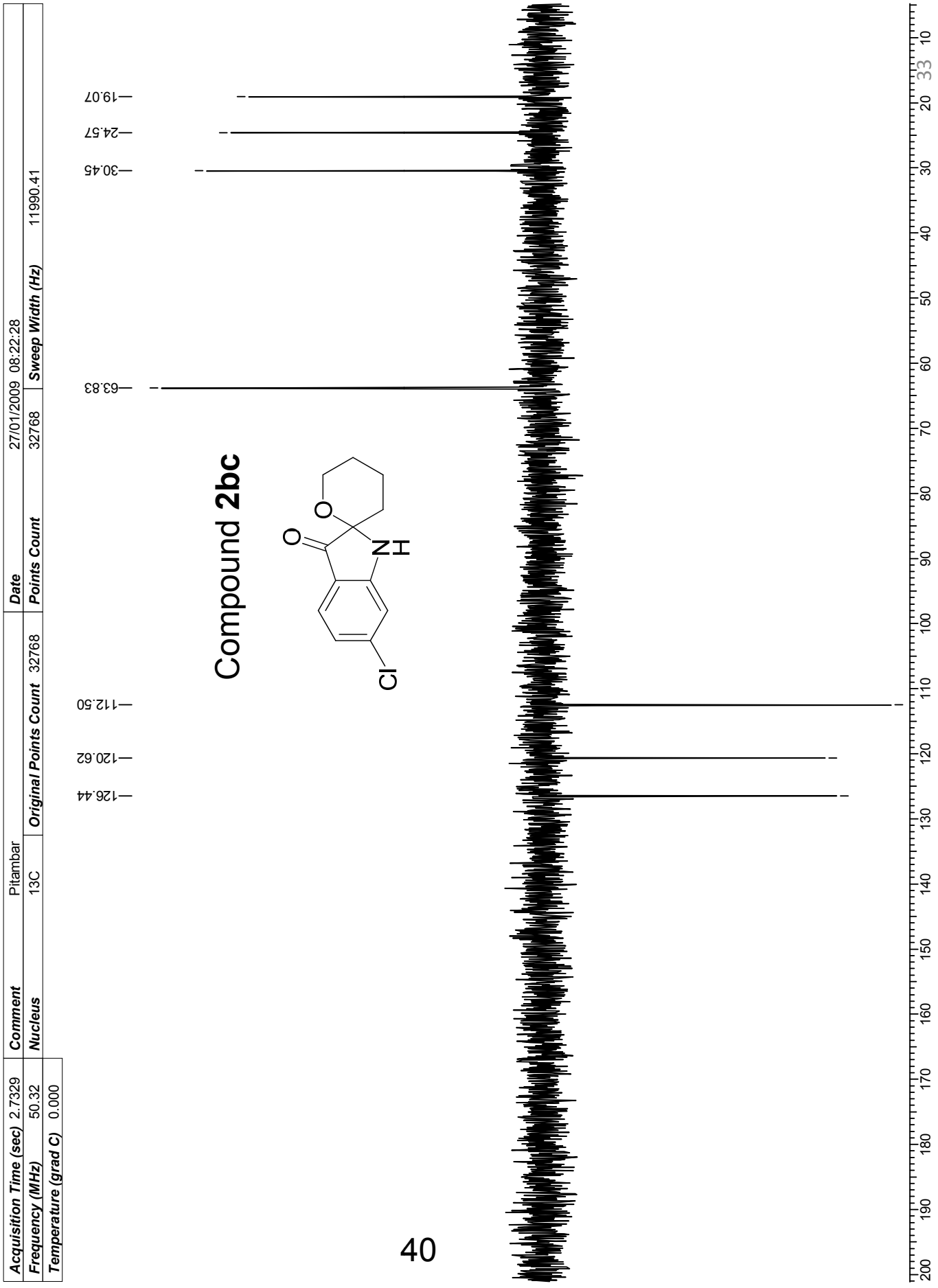


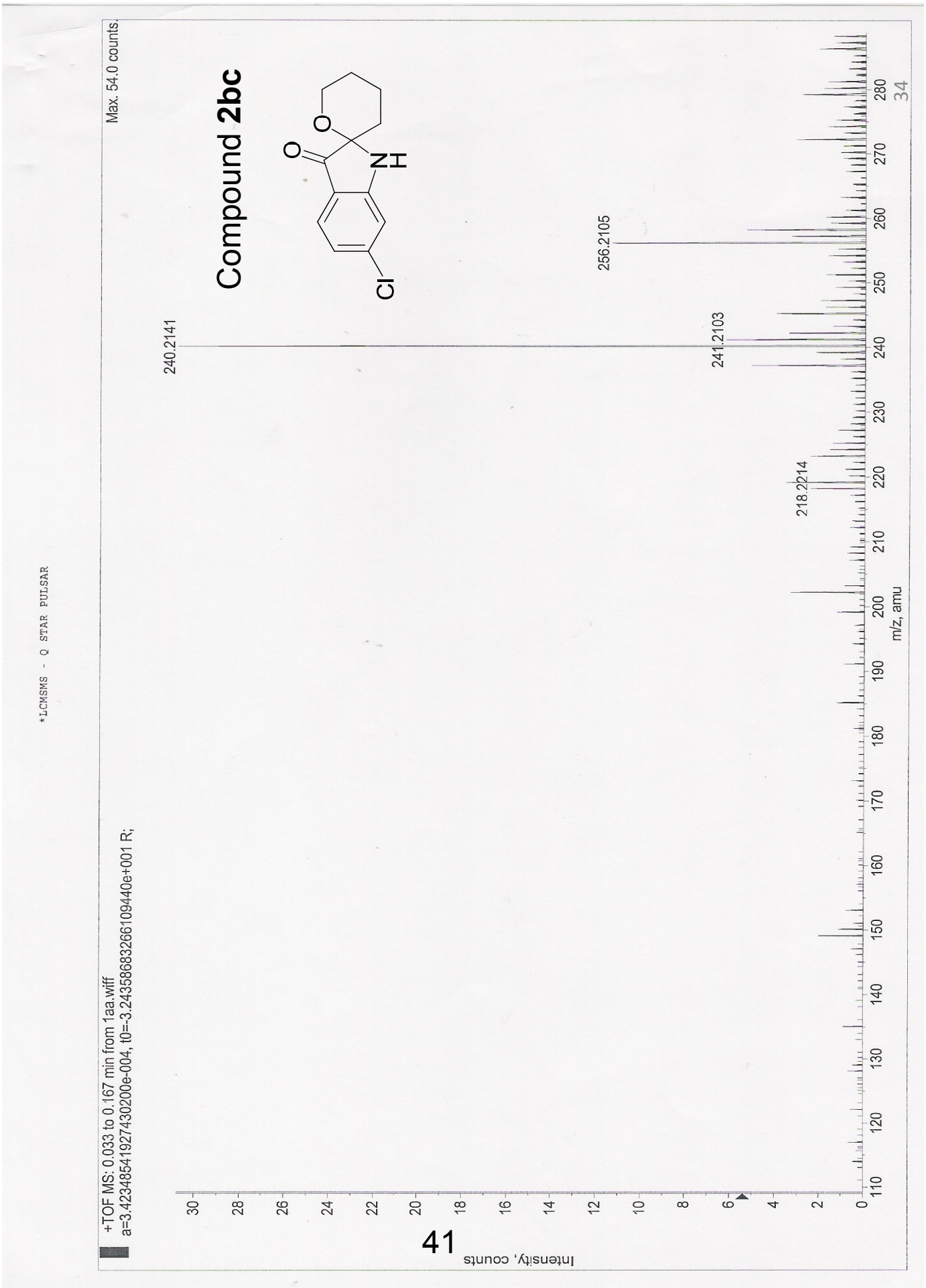
Acquisition Time (sec)	7.9167	***Comment***	Pitambar		***Date***	28/01/2009 17:56:18	
Frequency (MHz)	200.13	***Nucleus***	1H	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 4139.07
Temperature (grad C)	0.000						

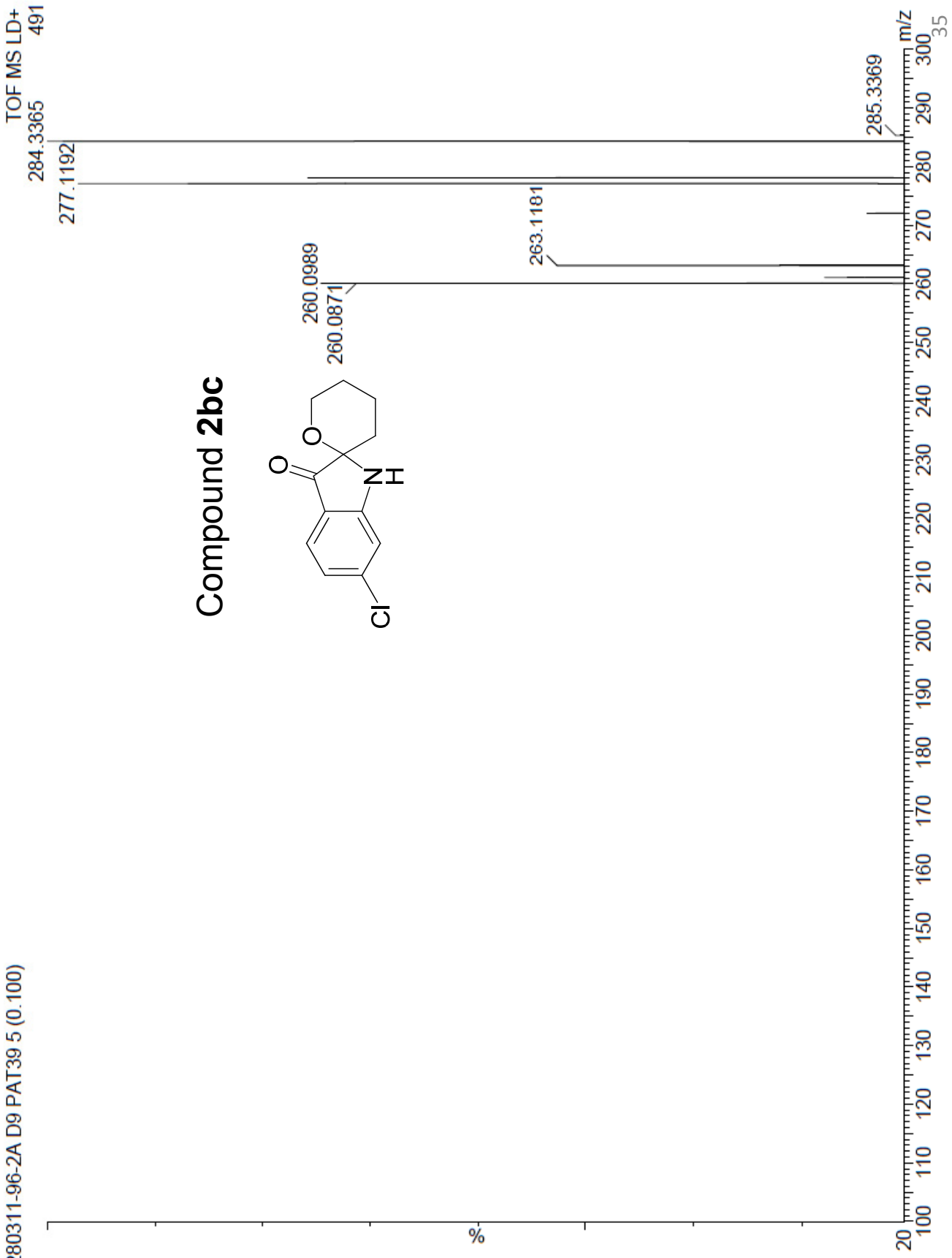


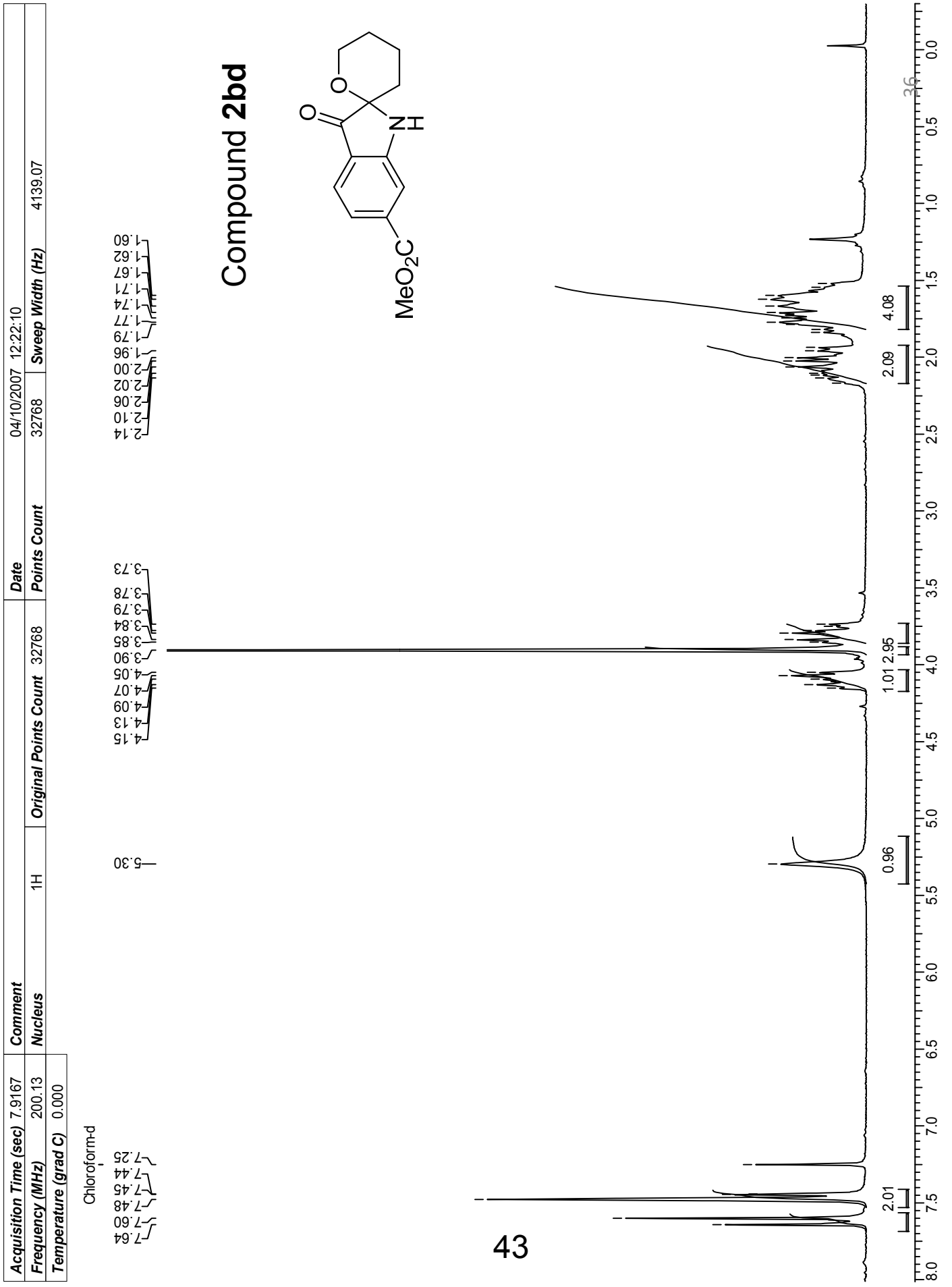
Compound **2bc**

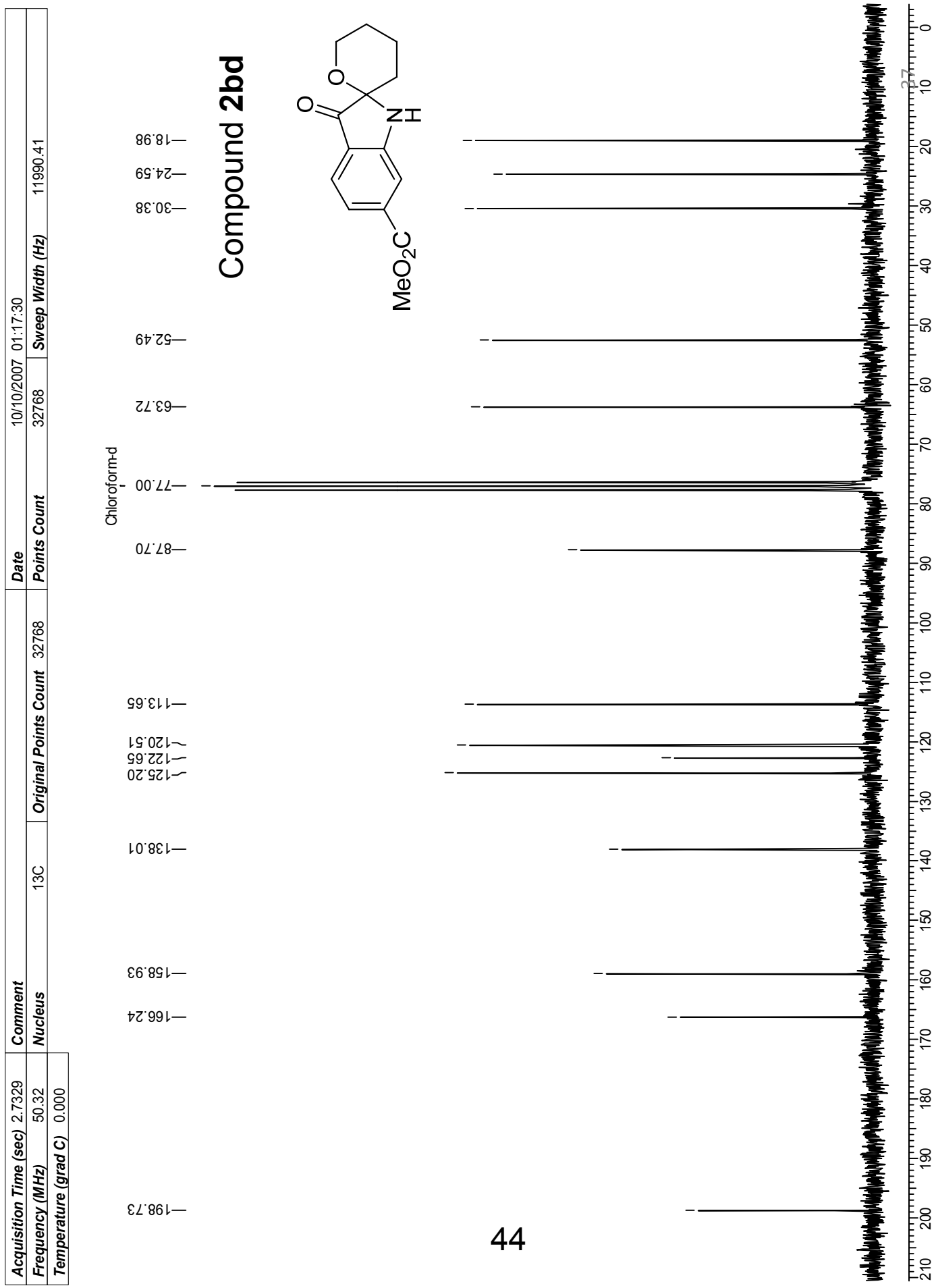


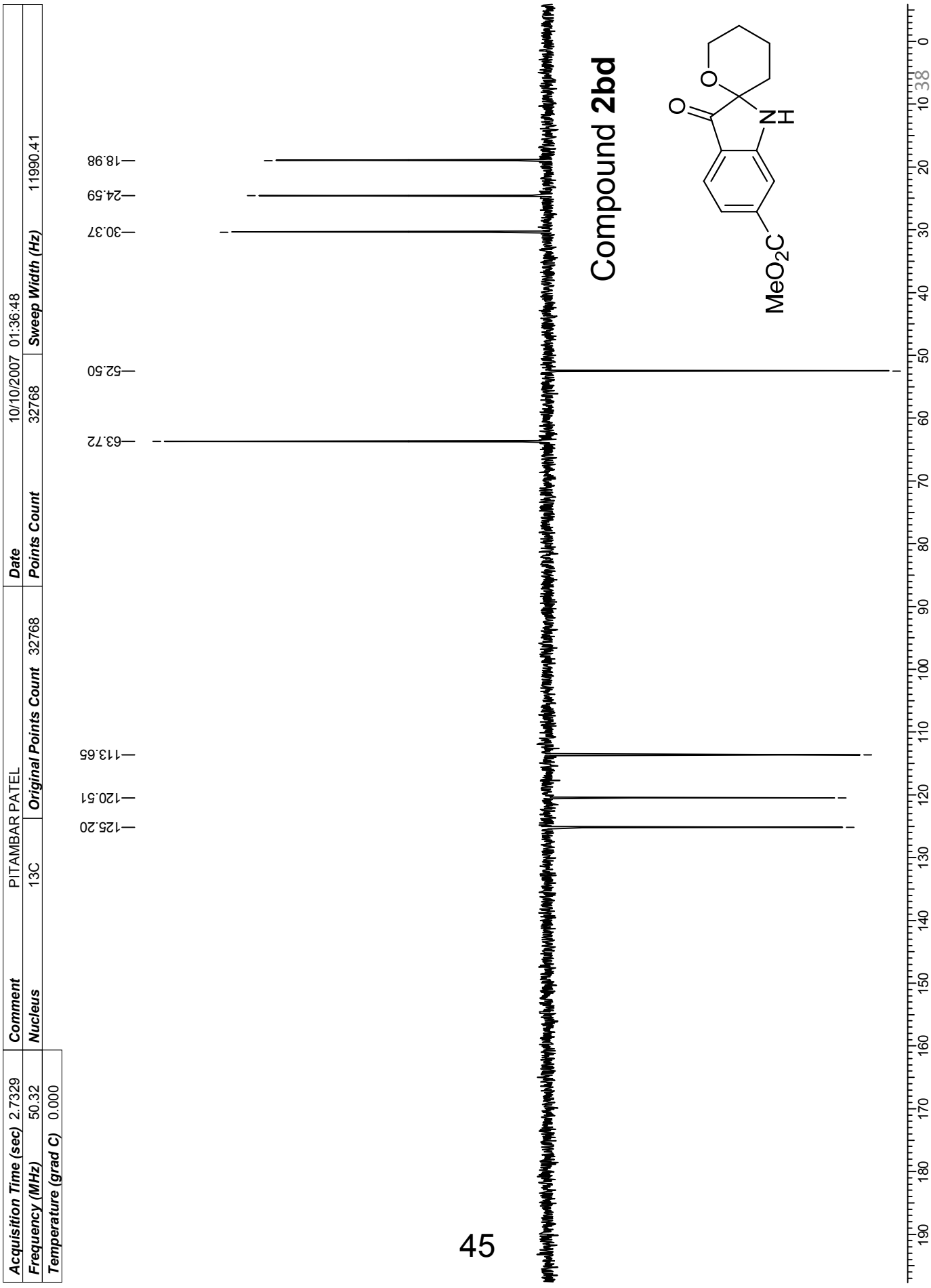


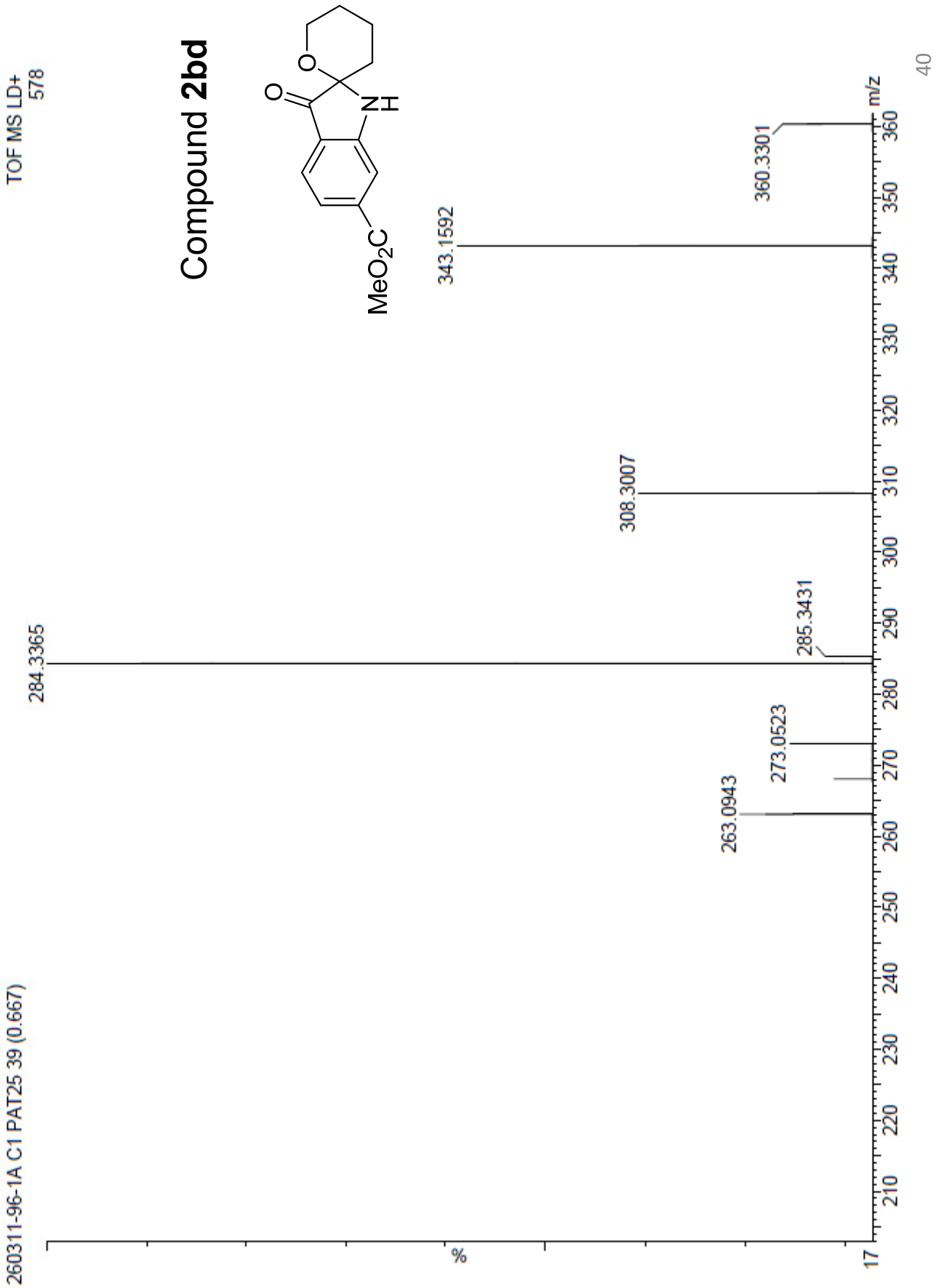


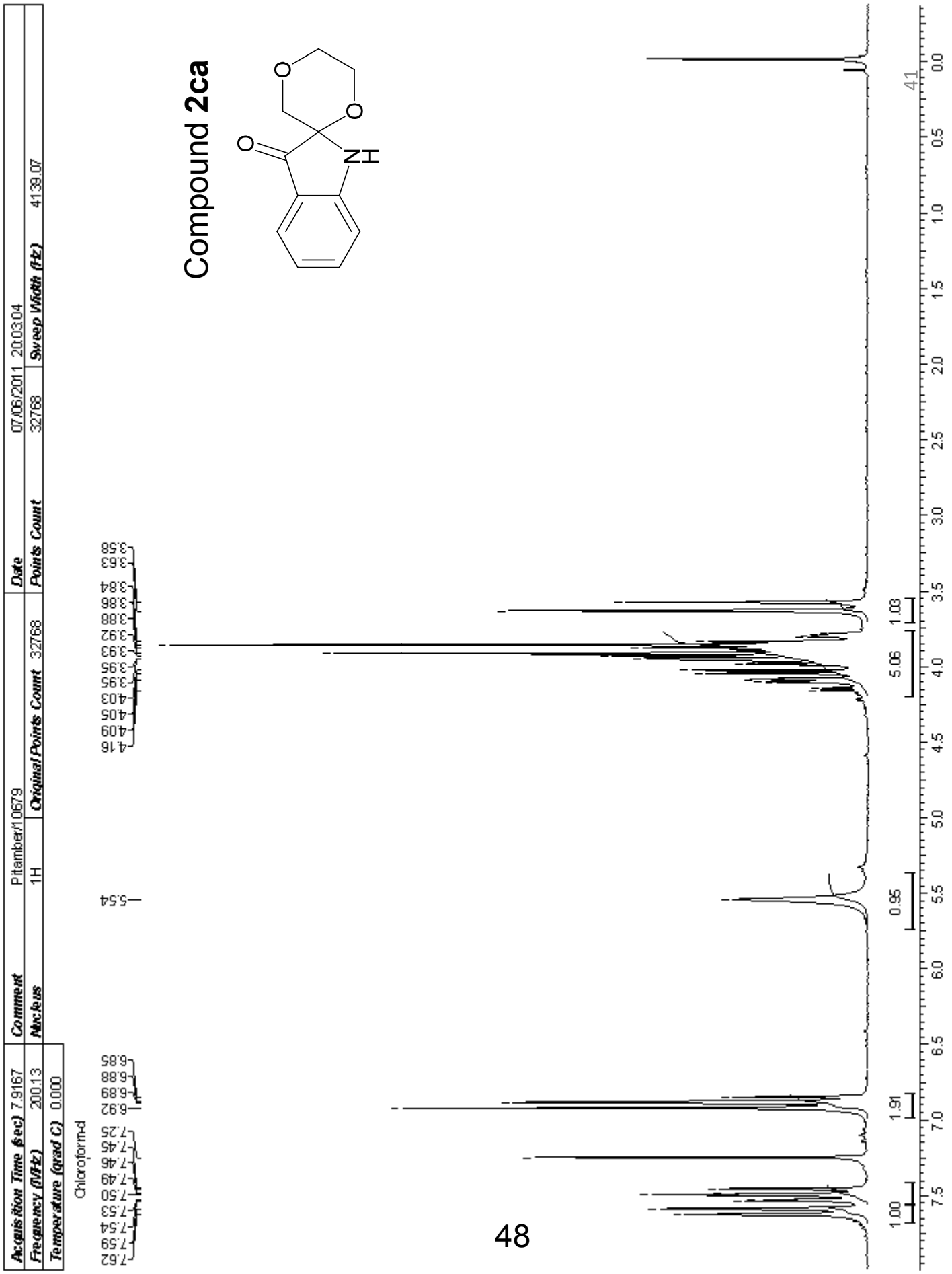


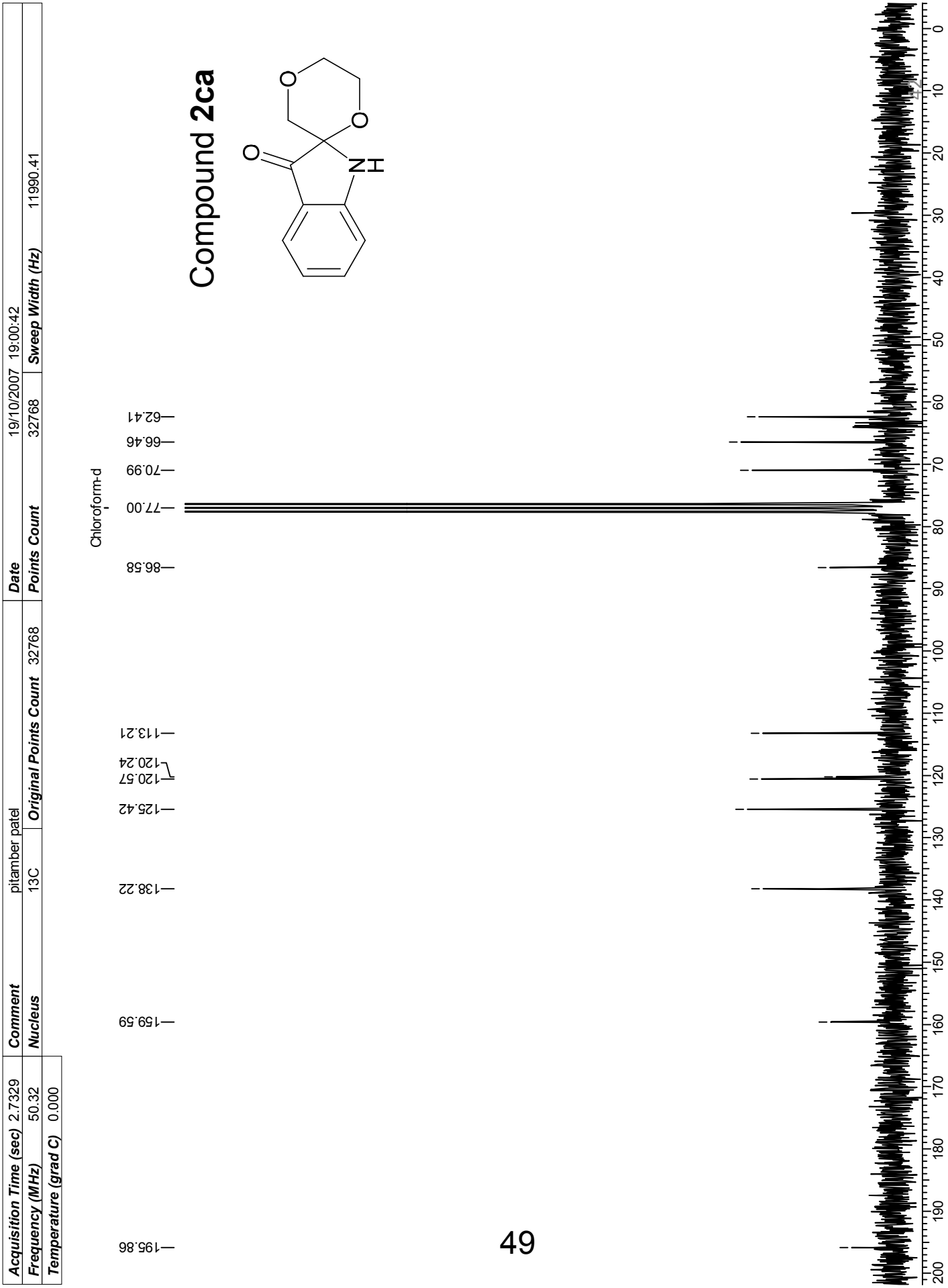


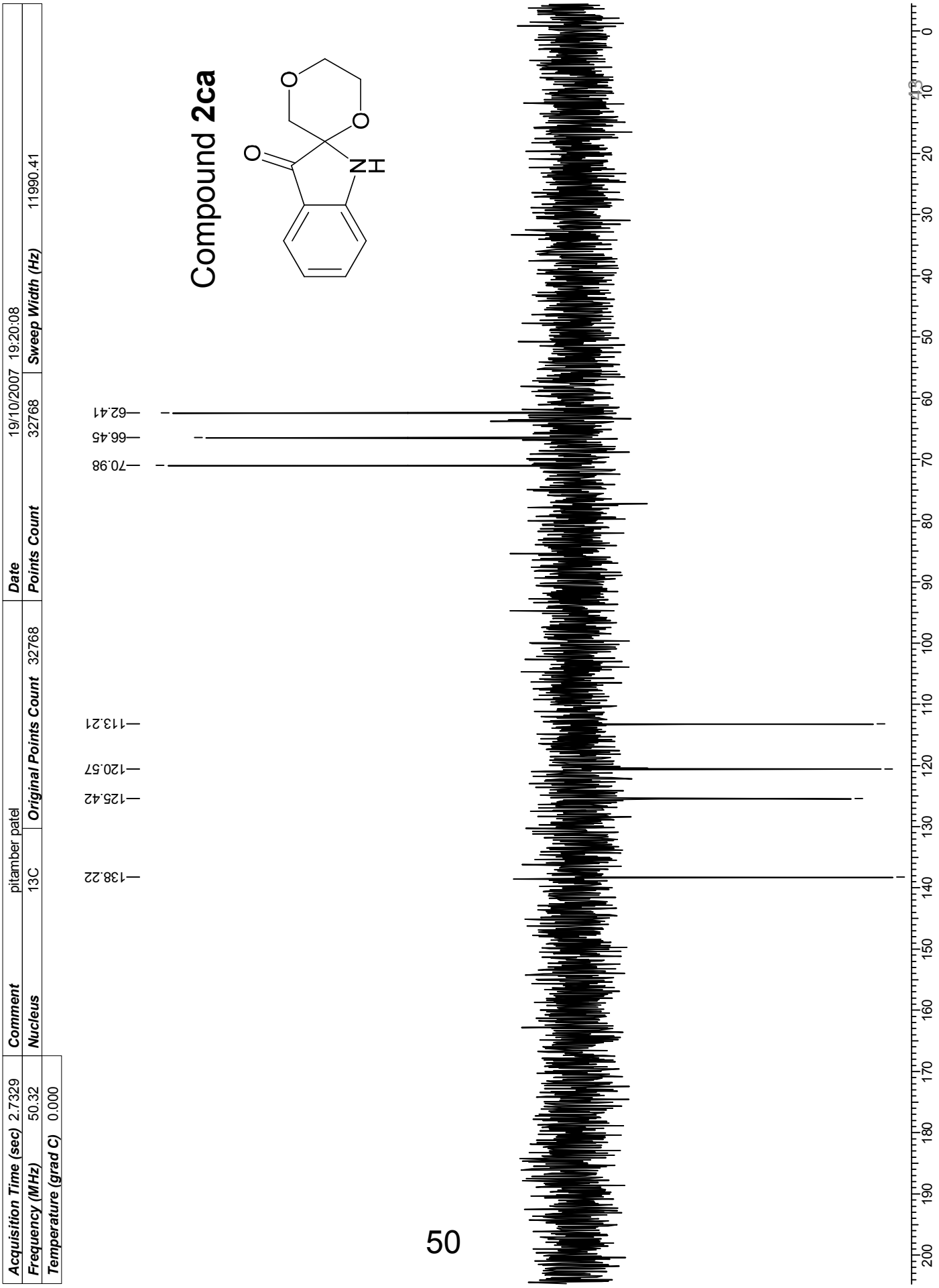




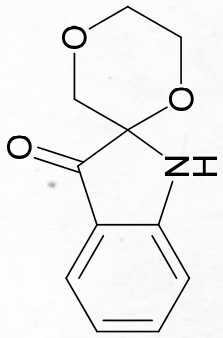


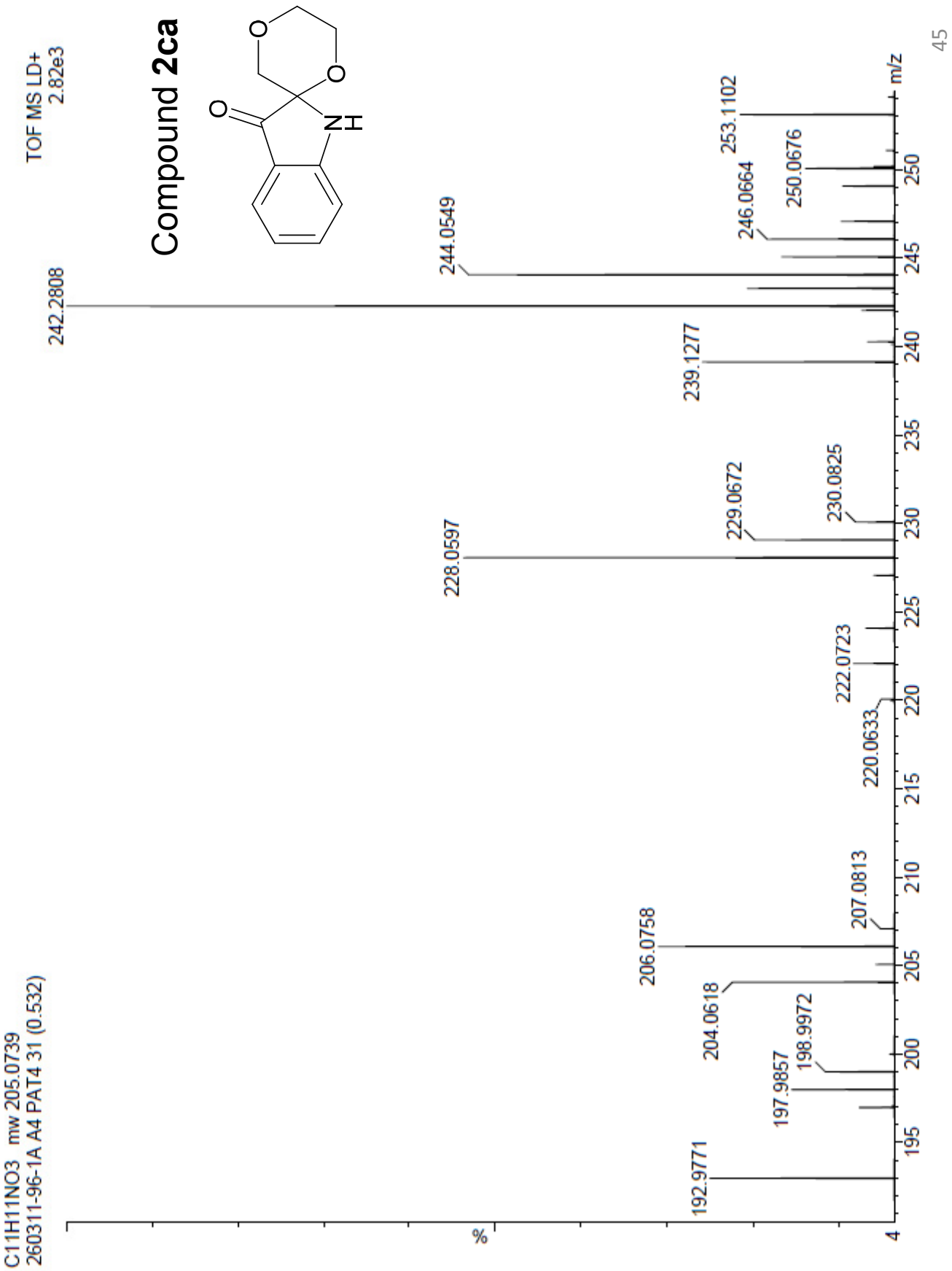


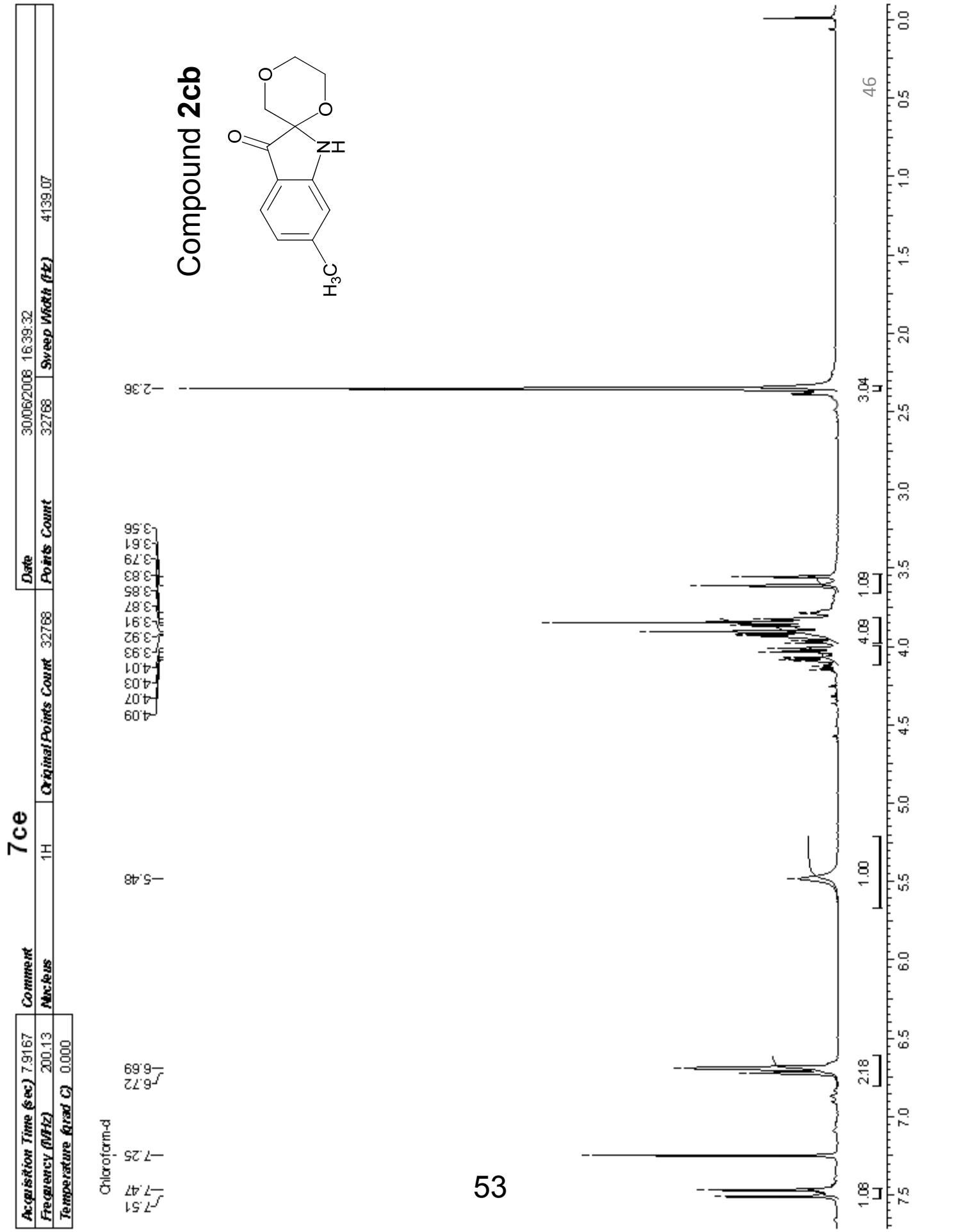


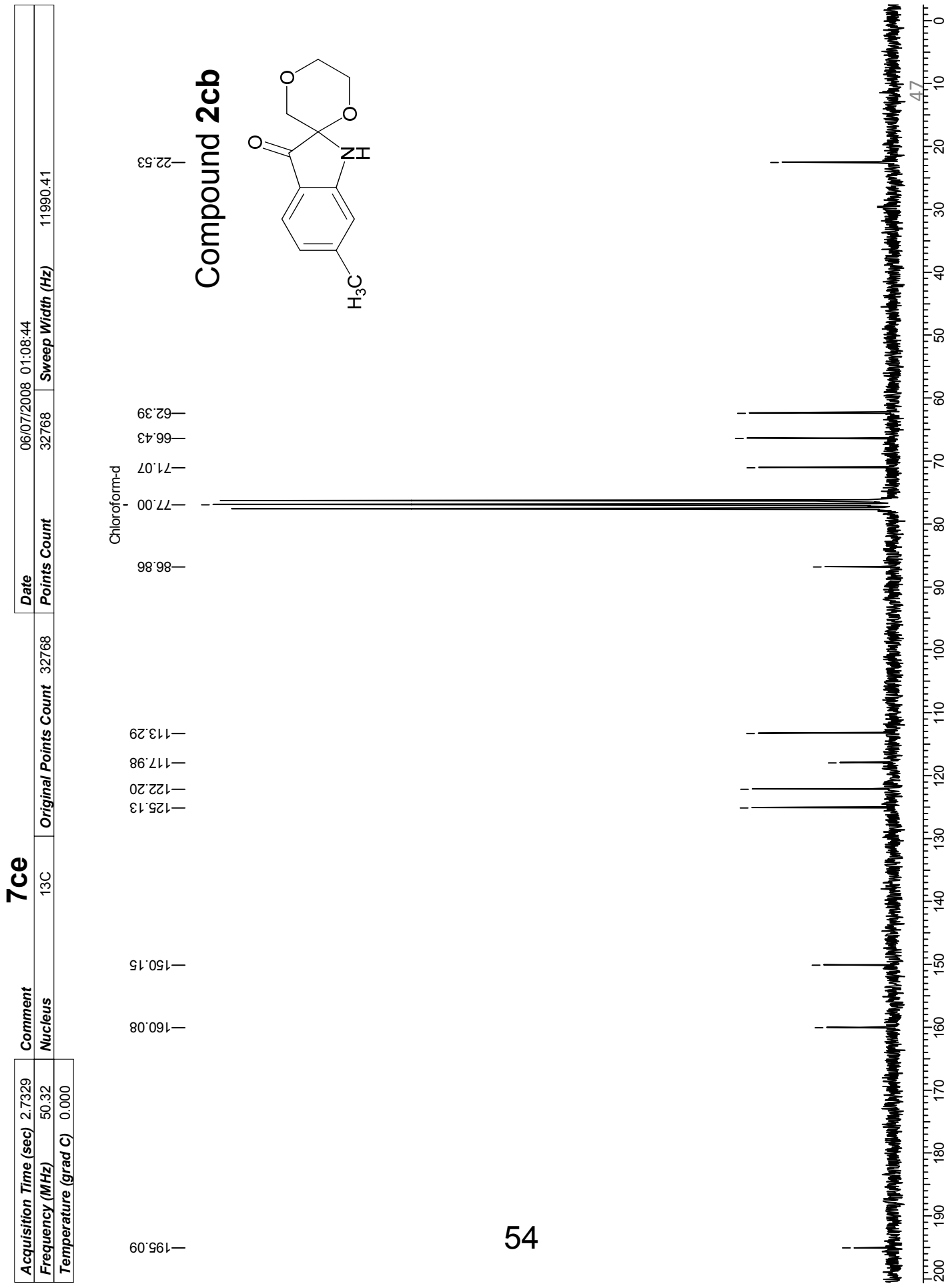


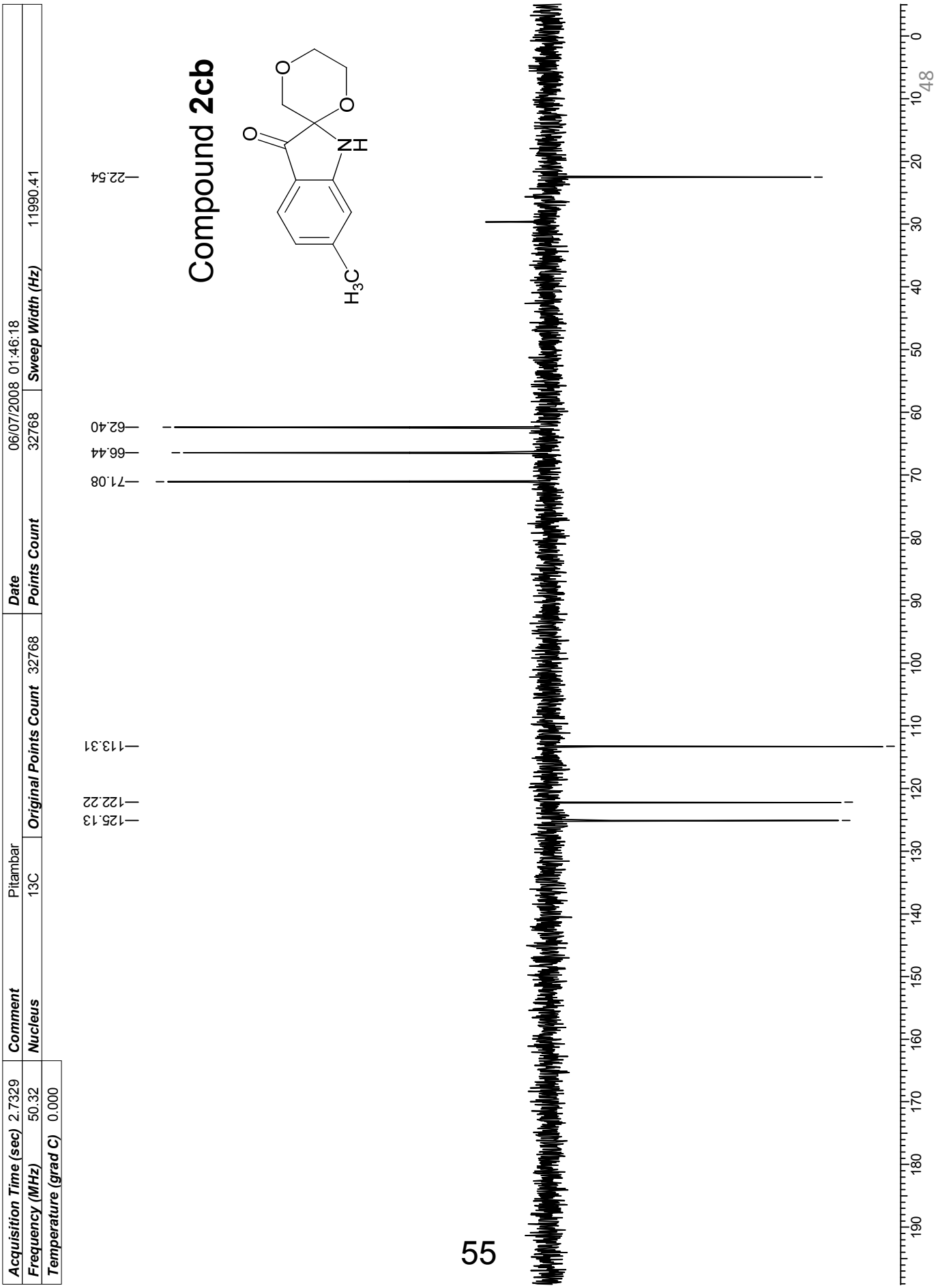
Compound **2ca**

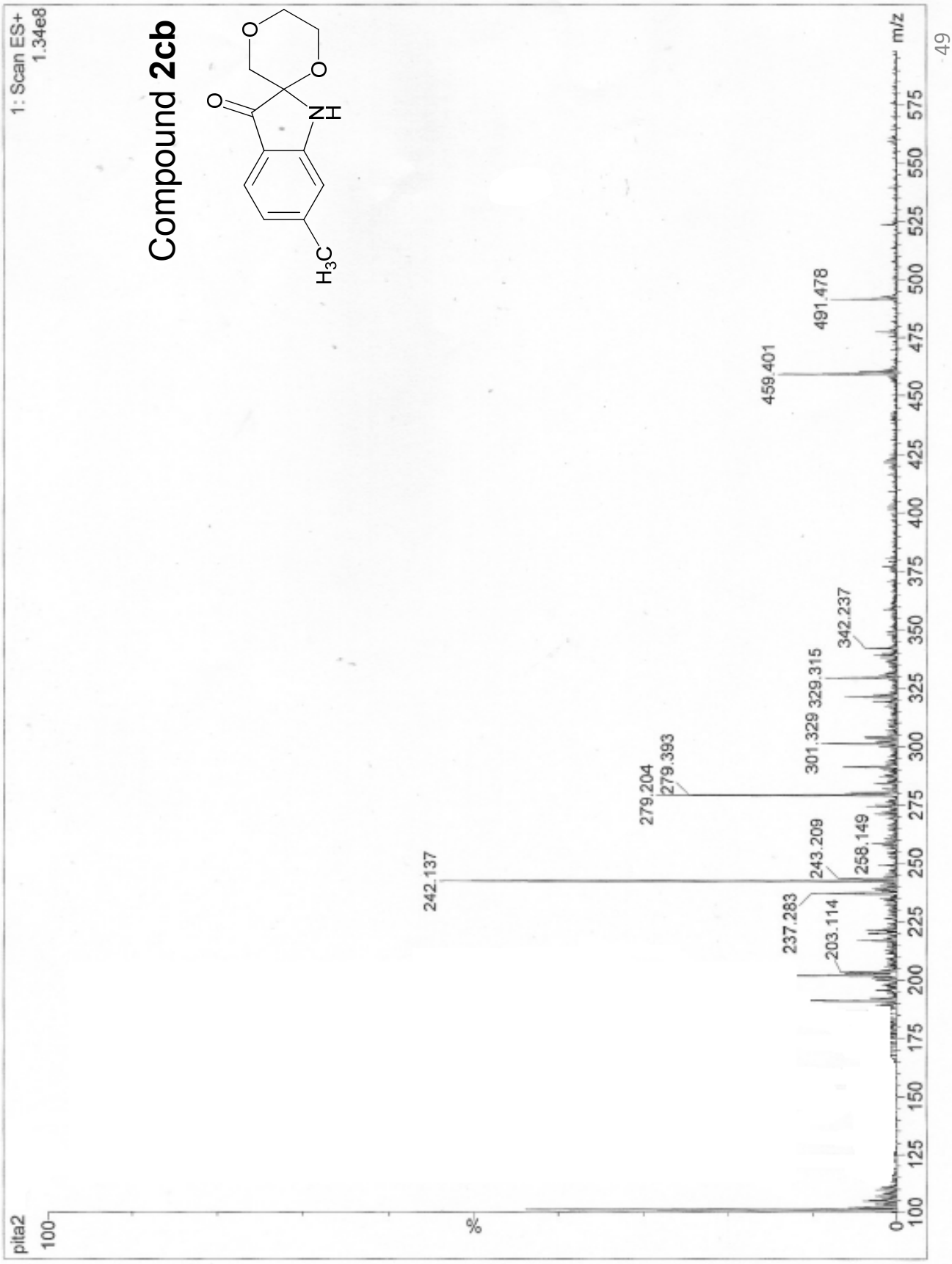


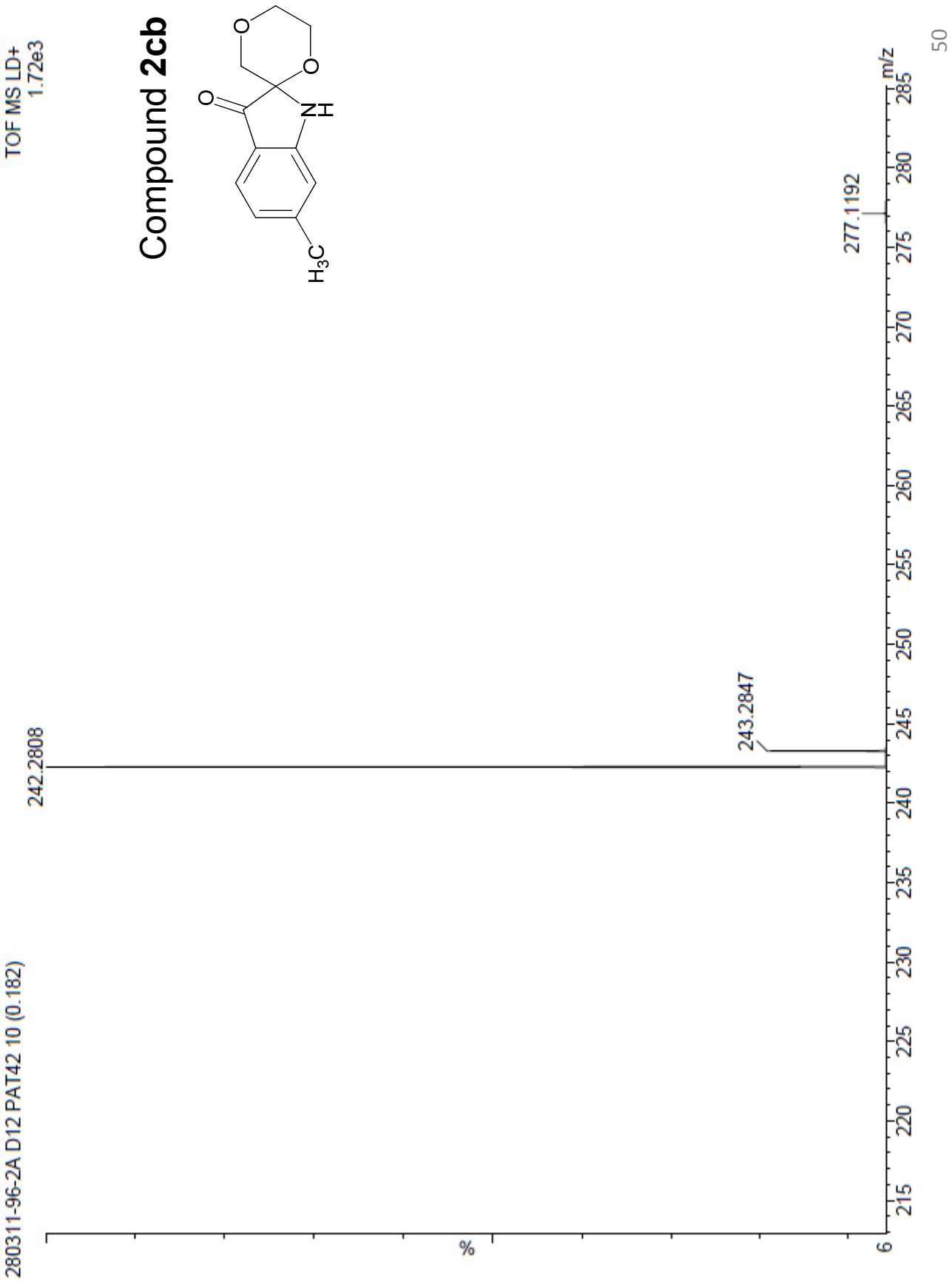


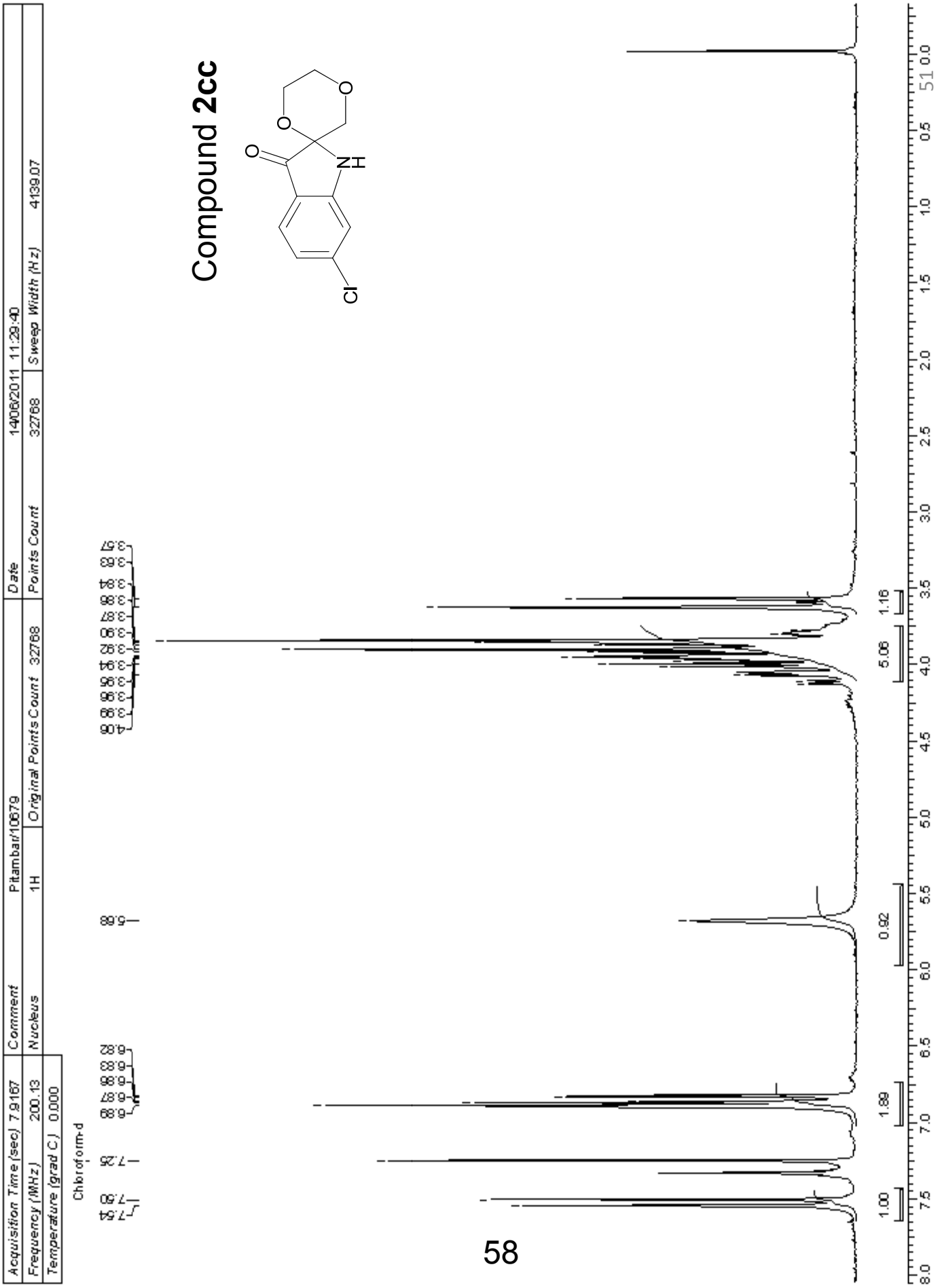


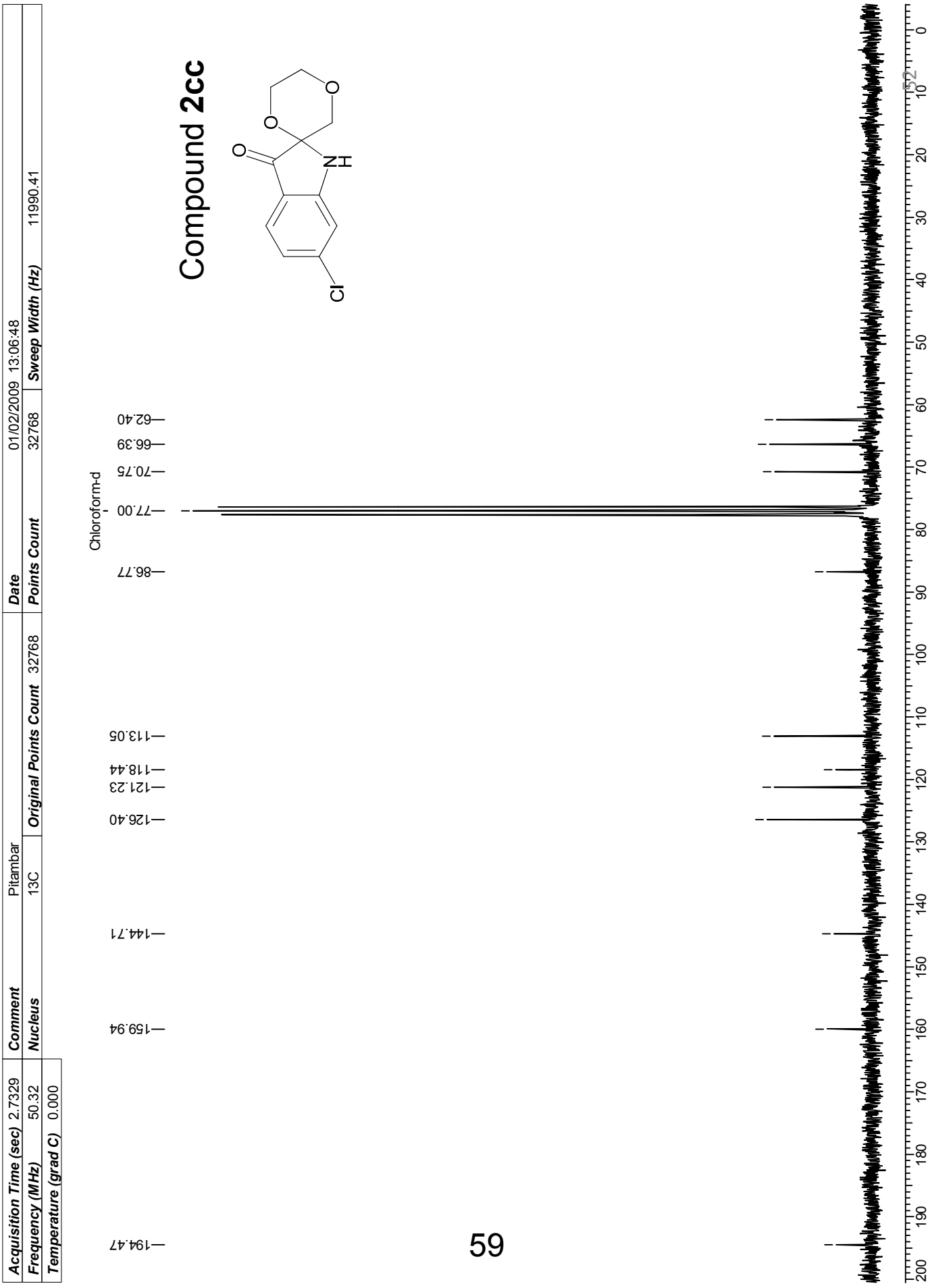


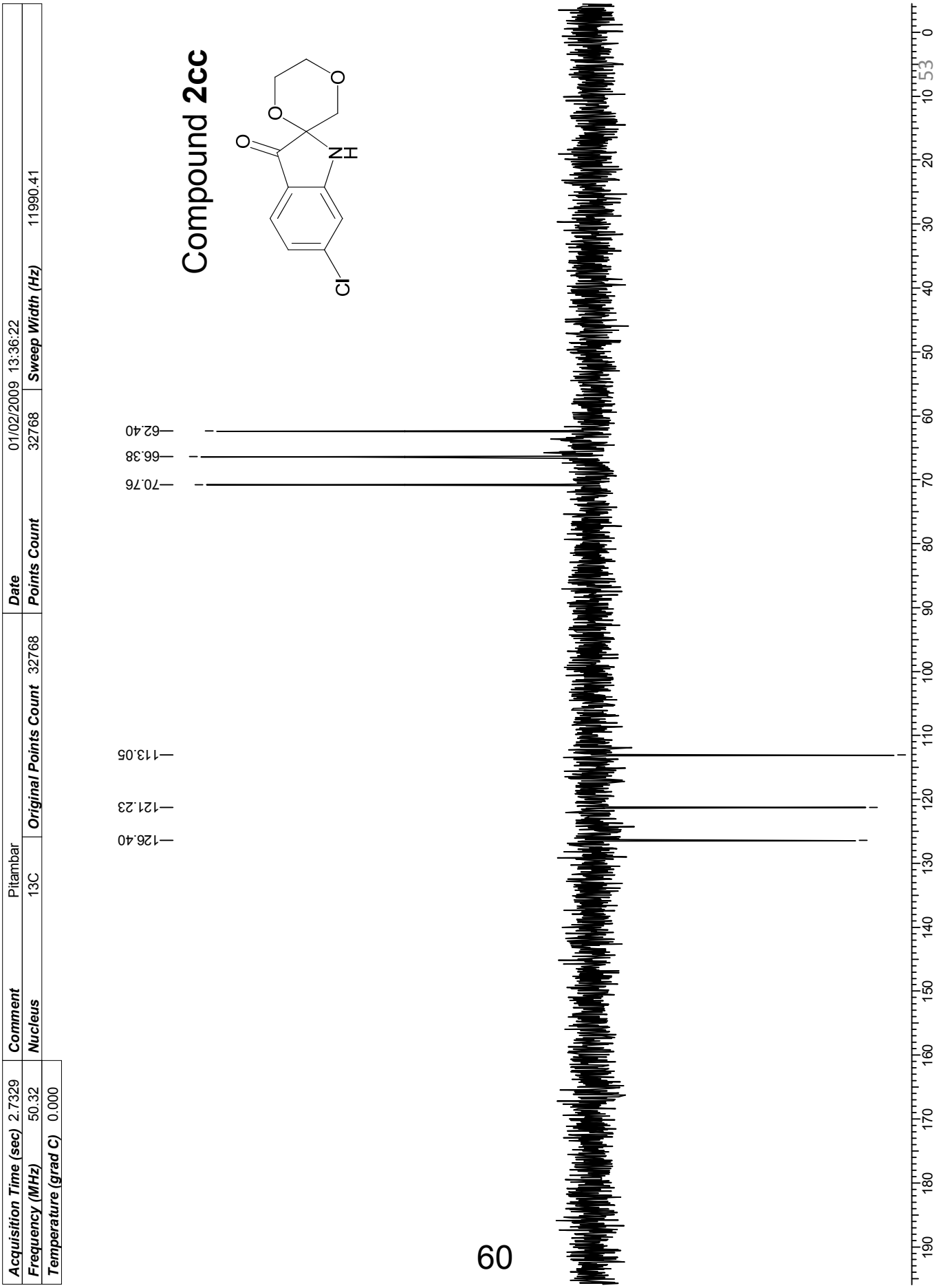


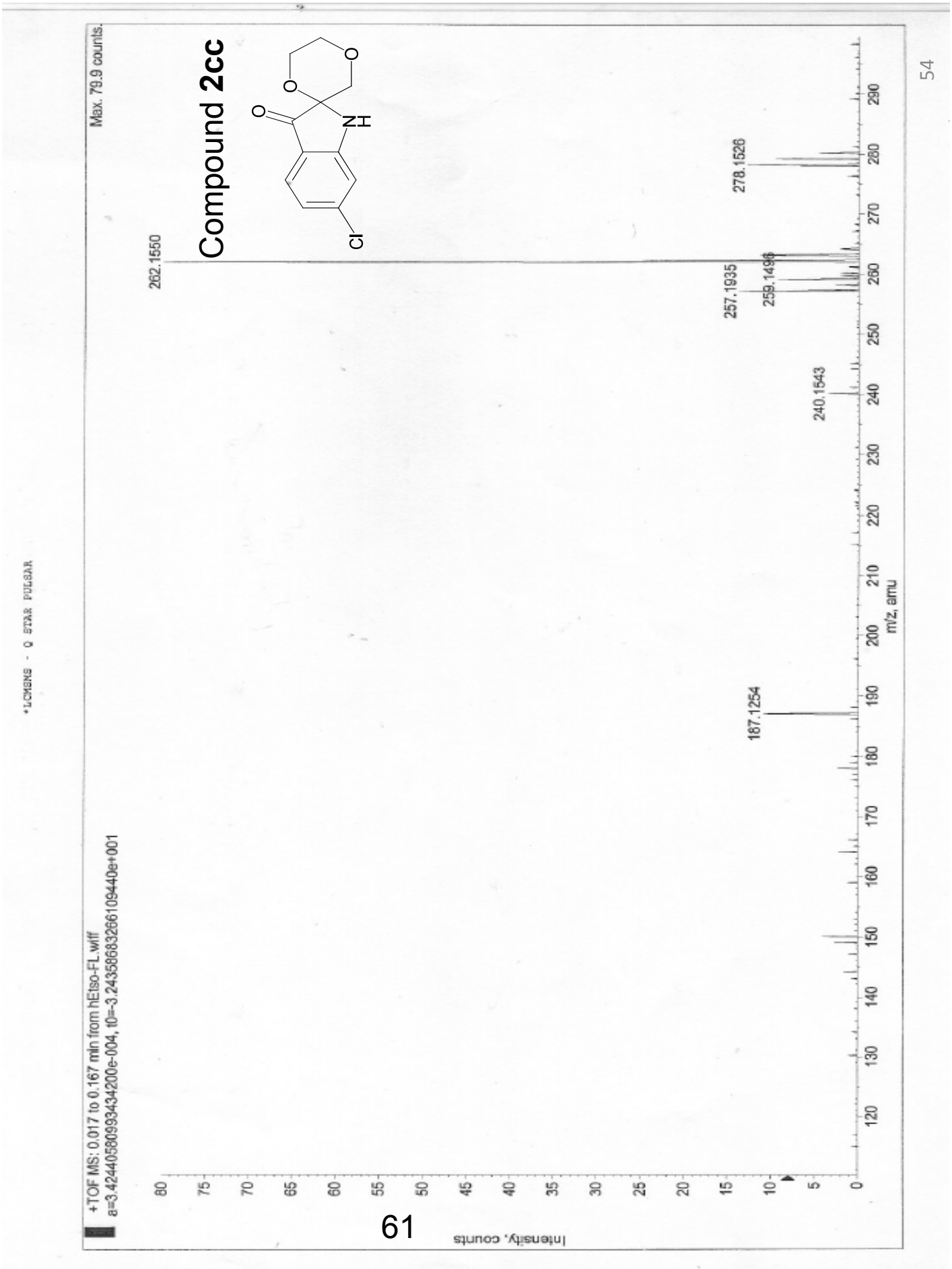




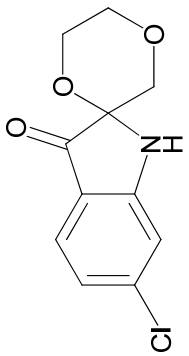


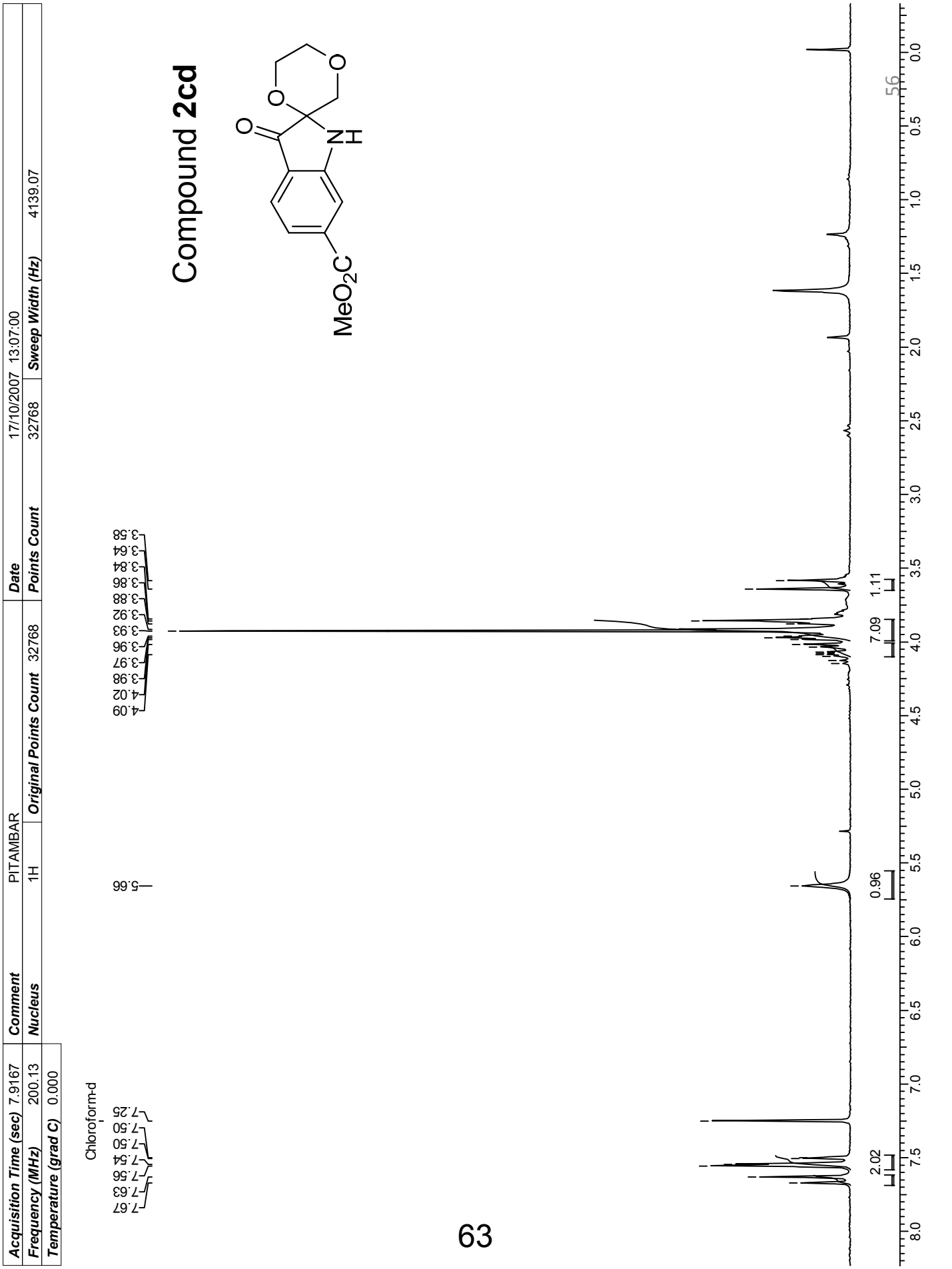


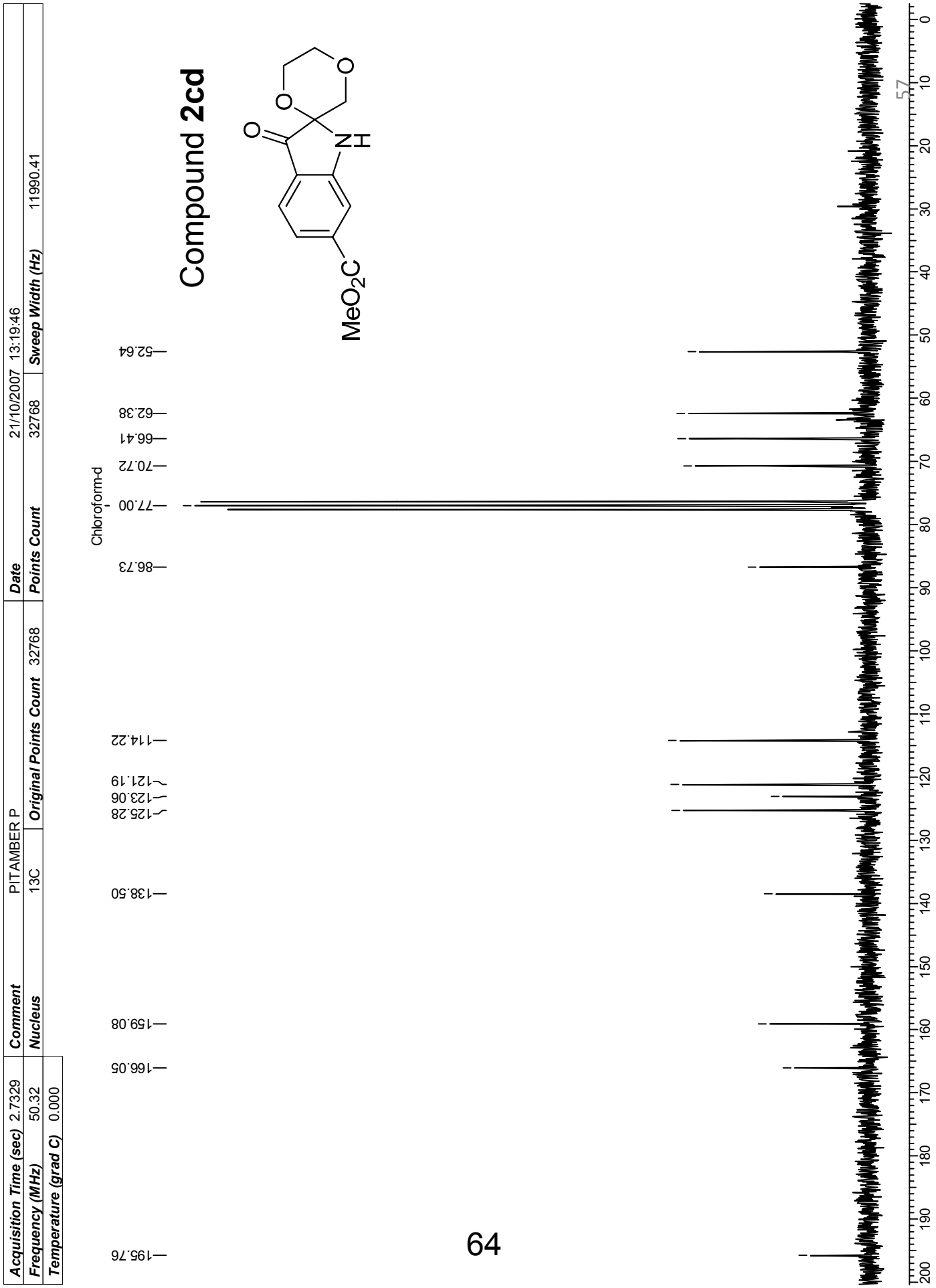


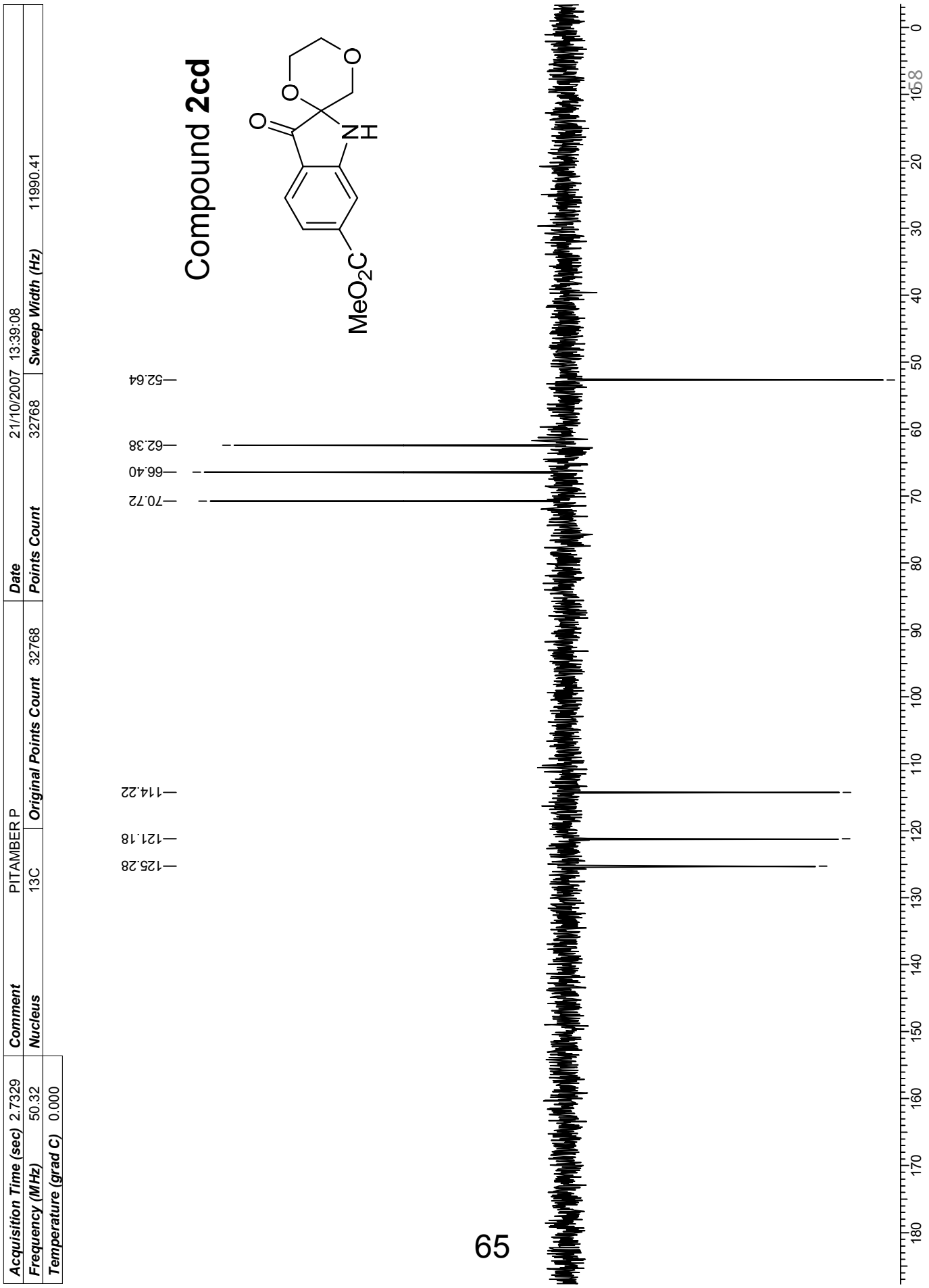

Compound 2cc
+TOF MS: 0.017 to 0.167 min from hEtso-FL.wiff
a=3.42440580993434200e-004, t0=-3.24358683266109440e+001
Max. 79.9 counts.
262.1550
257.1935
259.1496
278.1526
240.1543
187.1254
Intensity, counts
m/z, amu
61
54

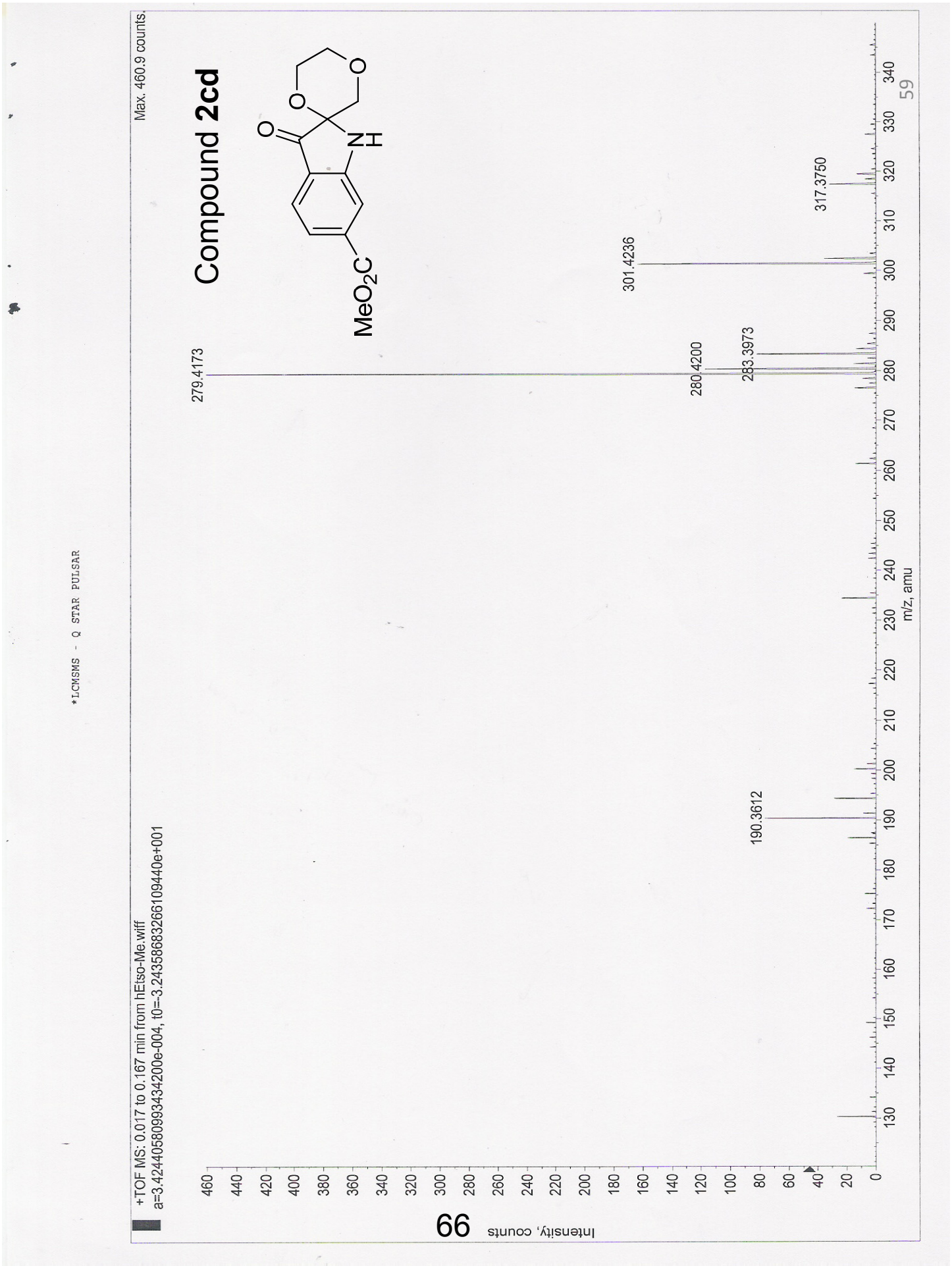
Compound **2cc**

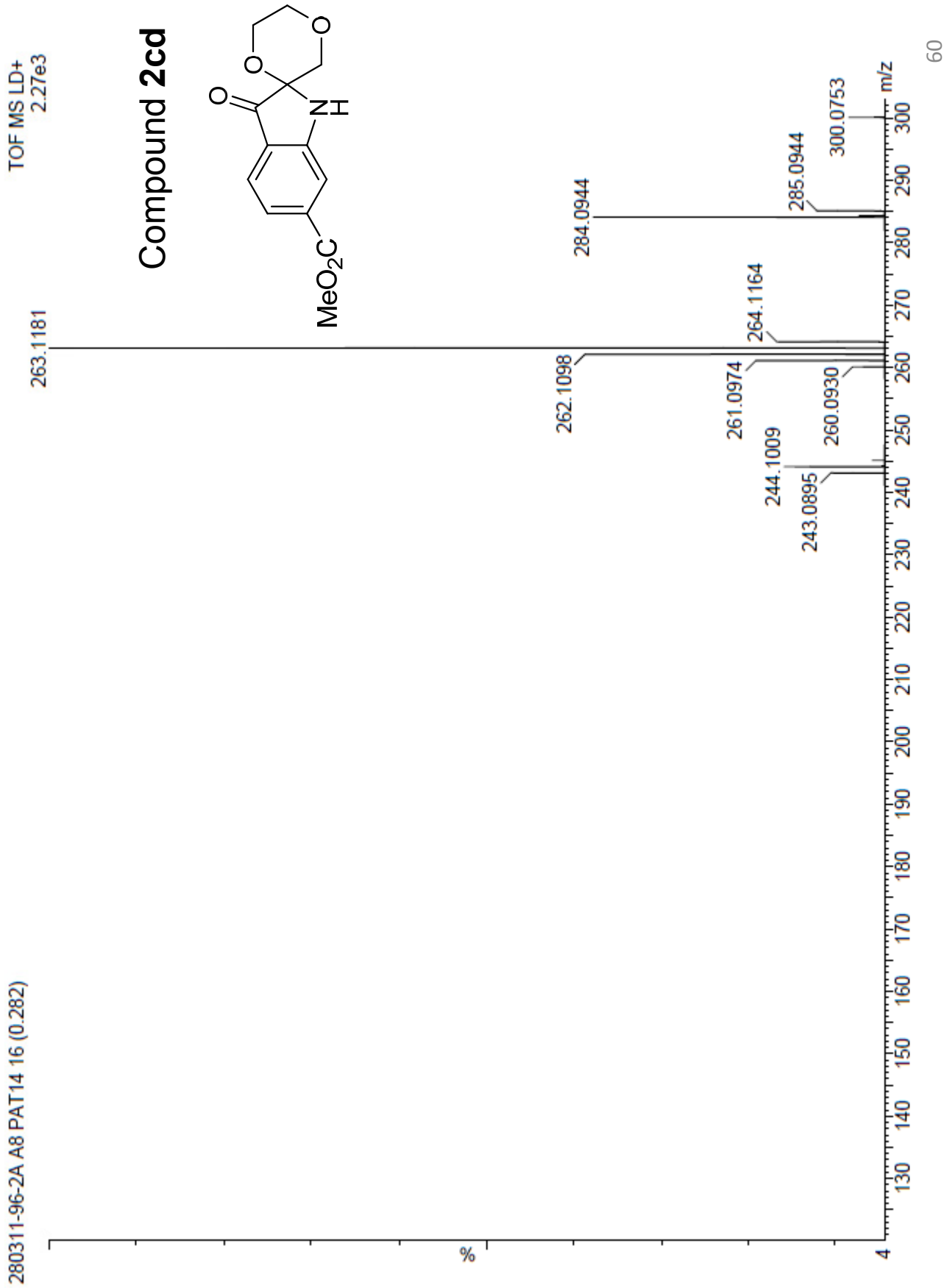




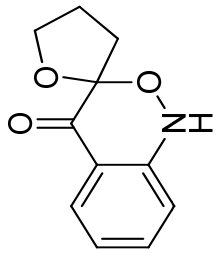


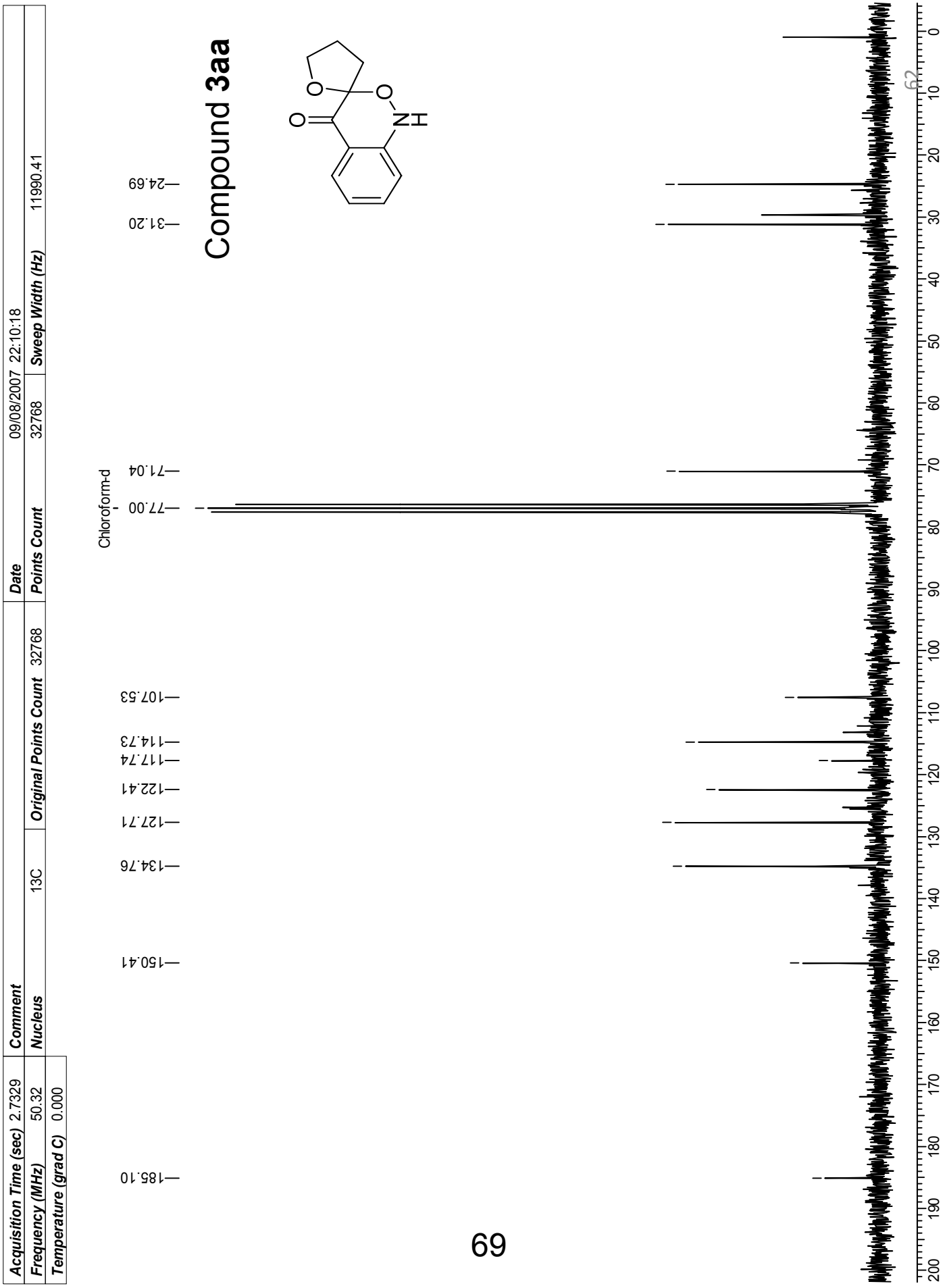


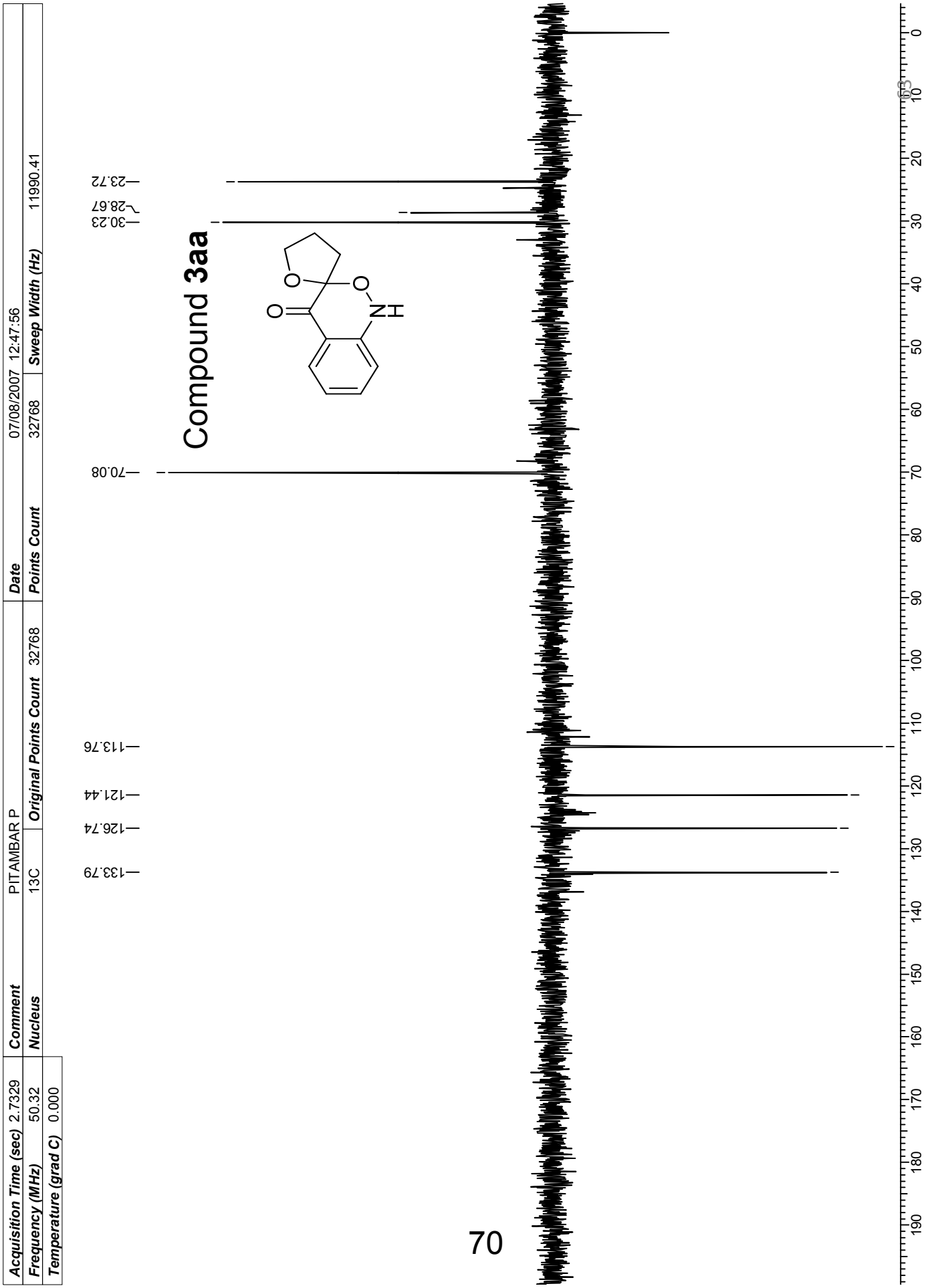


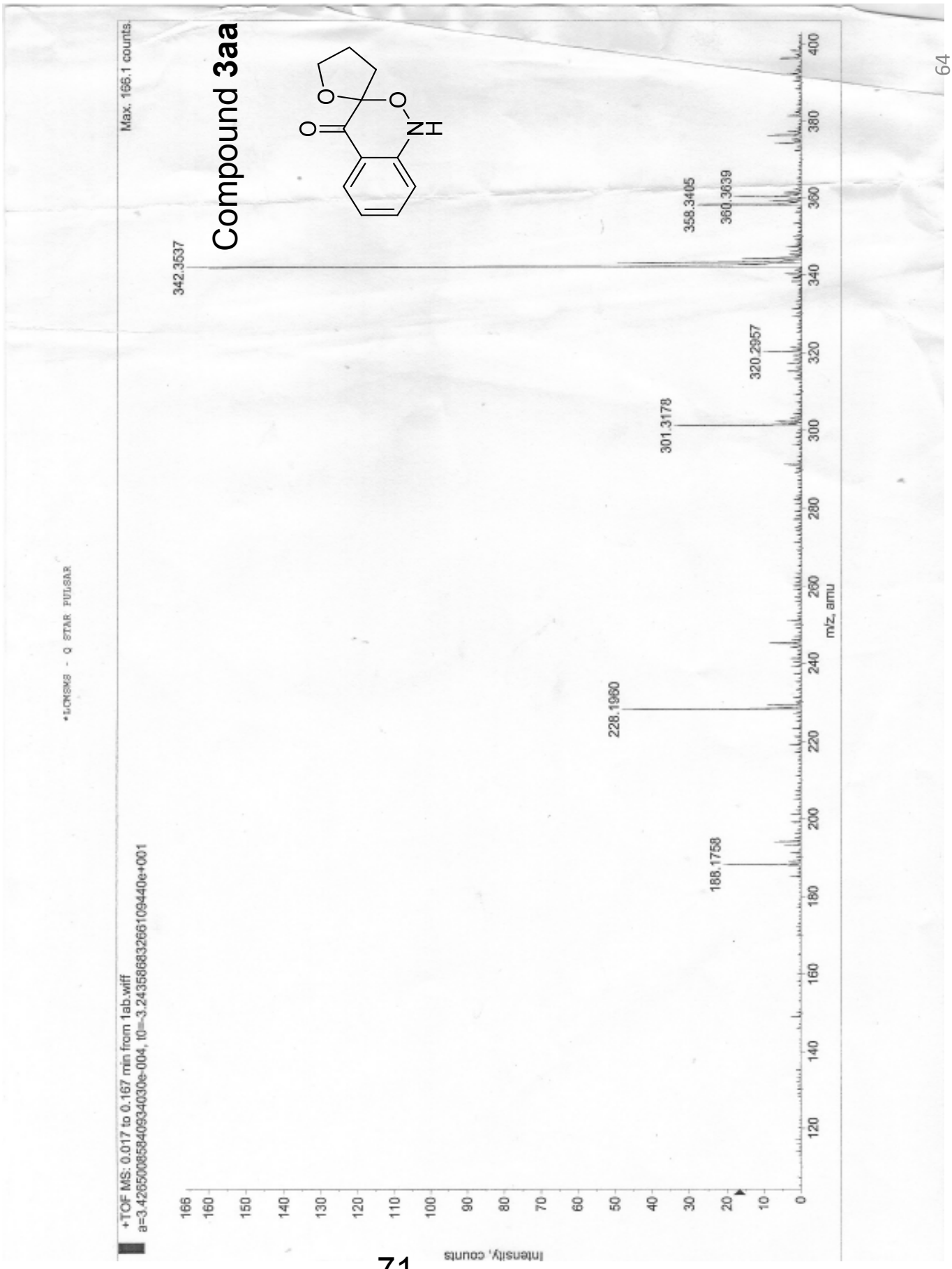

Compound 2cd
MeO2C
TOF MS LD+
2.27e3
263.1181
262.1098
261.0974
260.0930
244.1009
243.0895
284.0944
285.0944
264.1164
300.0753
280311-96-2A A8 PAT14 16 (0.282)
%
m/z
4
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300

Compound **3aa**

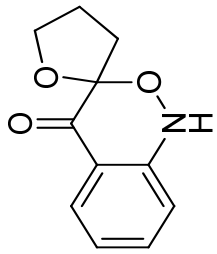


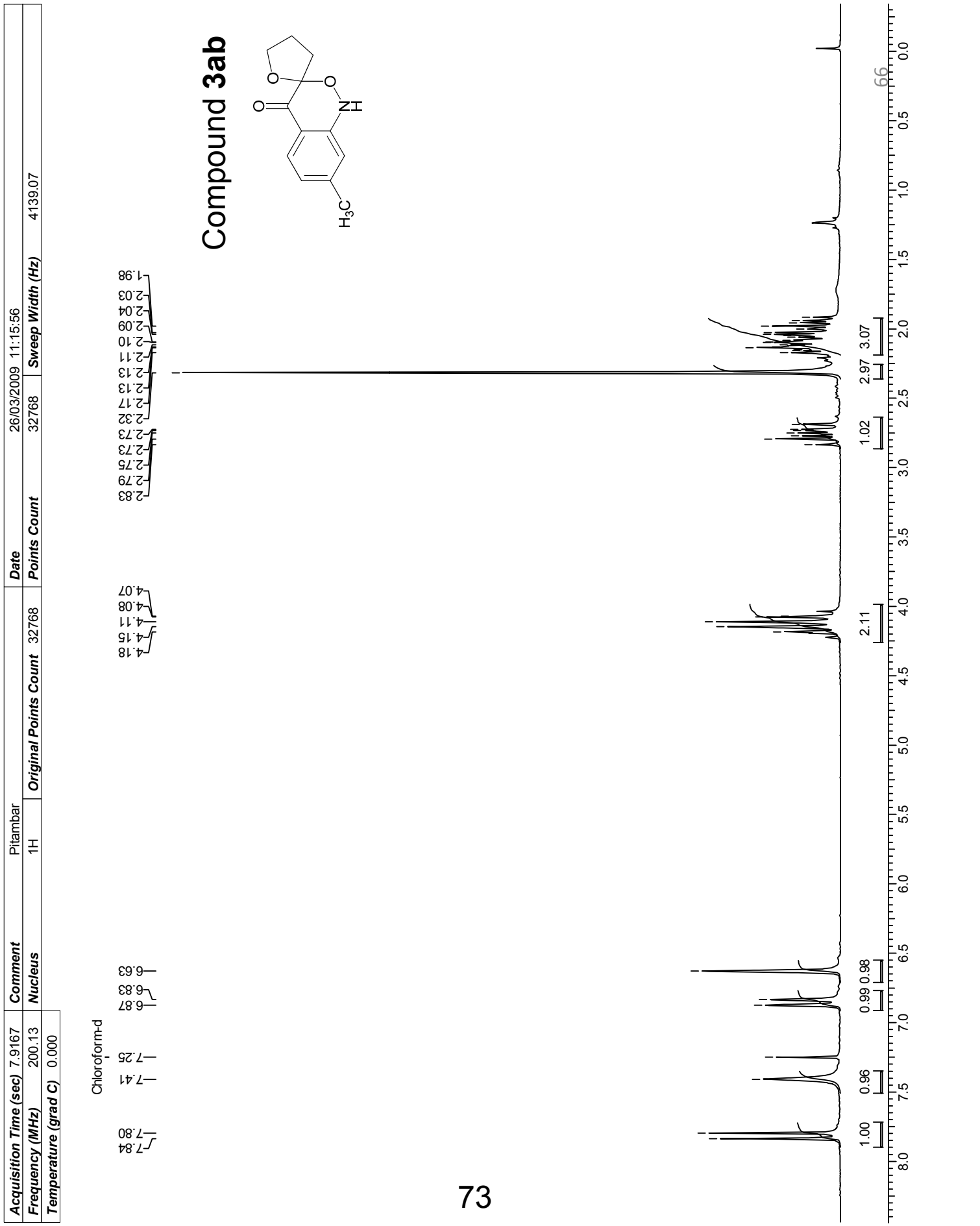


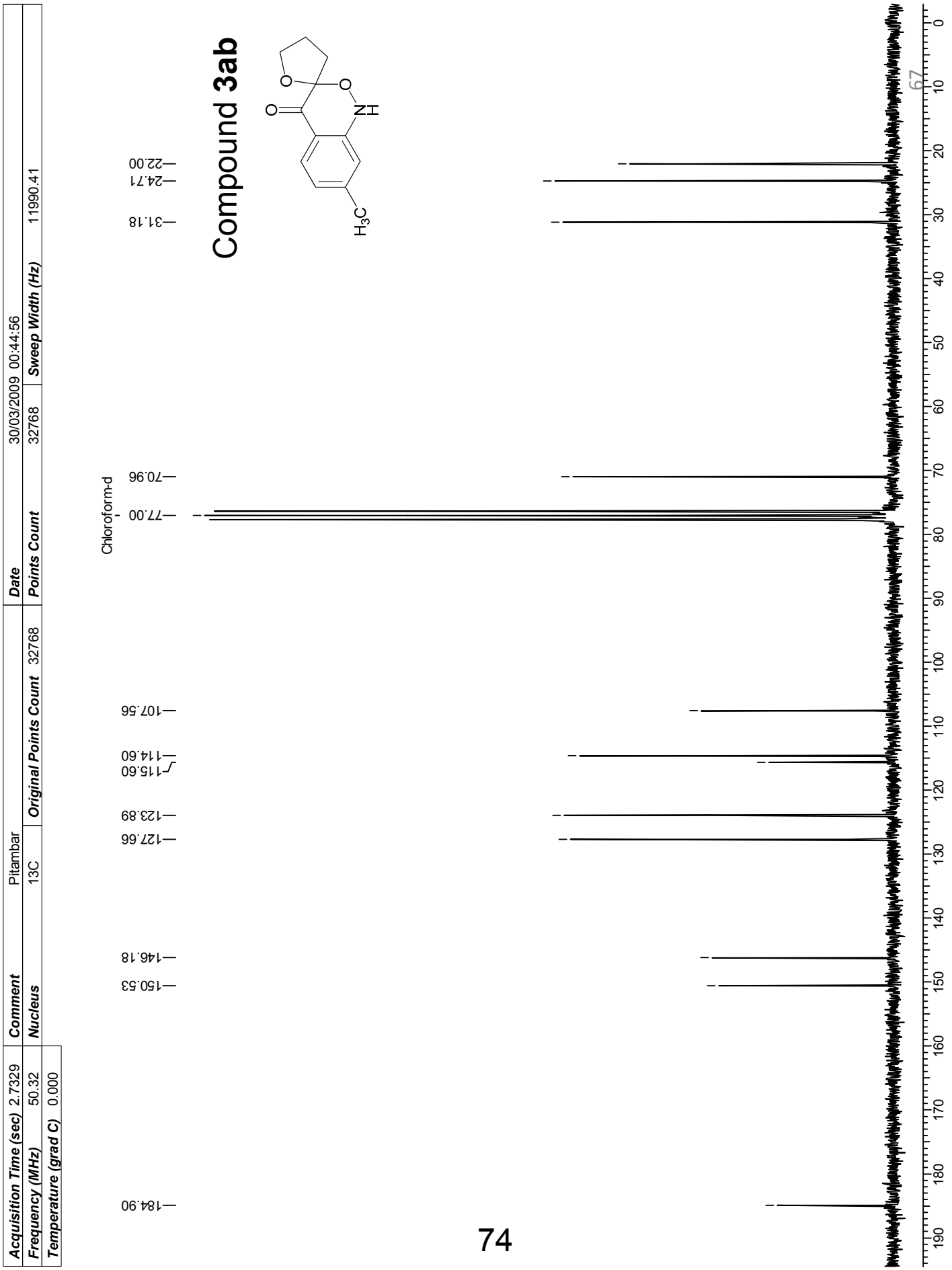


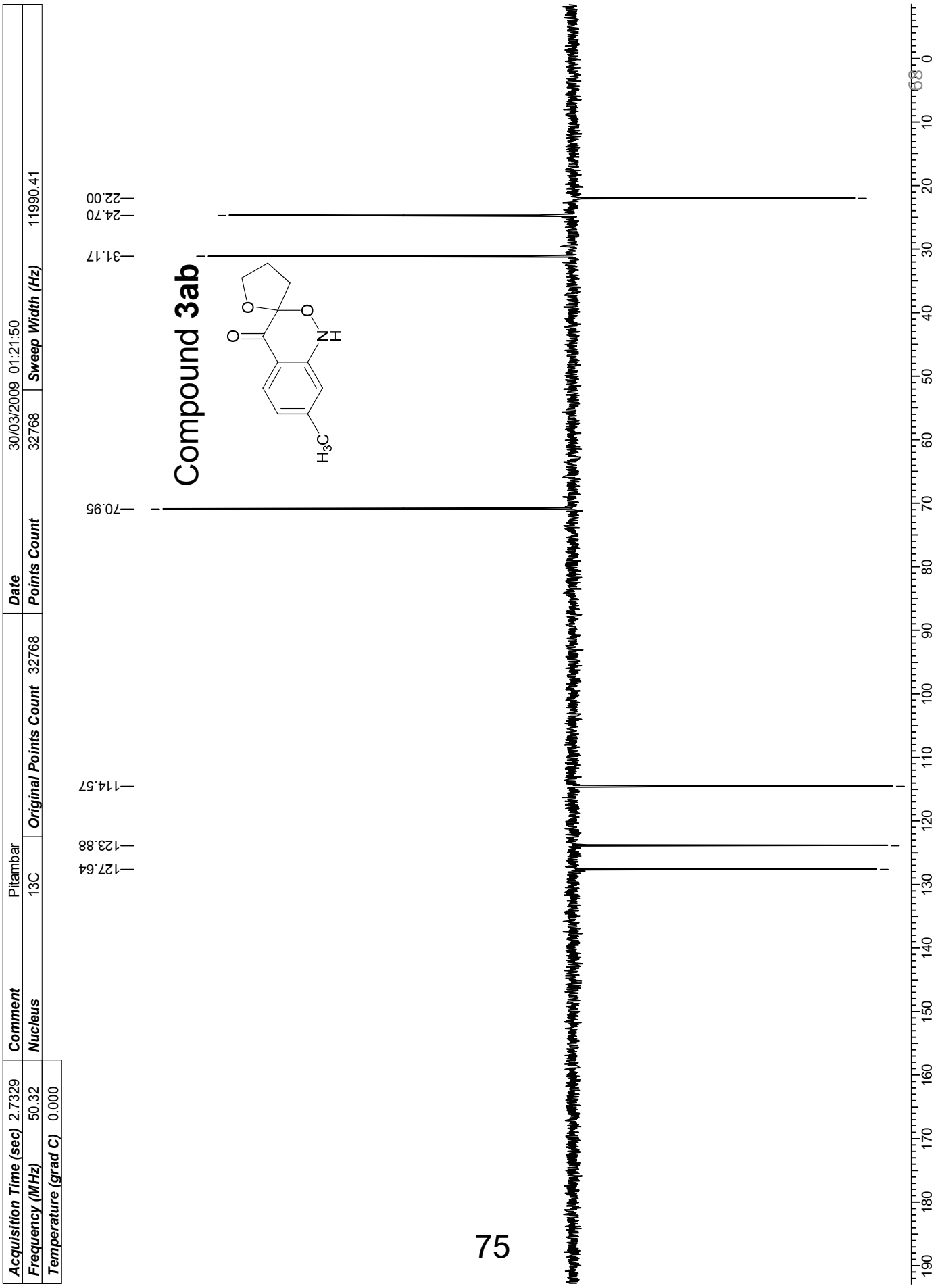

Compound 3aa
+TOF MS: 0.017 to 0.167 min from 1ab.wiff
a=3.42650085840934030e-004, t0=-3.24358683266109440e+001
*LCMSMS - Q STAR PULSAR
Max. 166.1 counts.
342.3537
358.3405
360.3839
320.2957
301.3178
228.1960
188.1758
Intensity, counts
m/z, amu

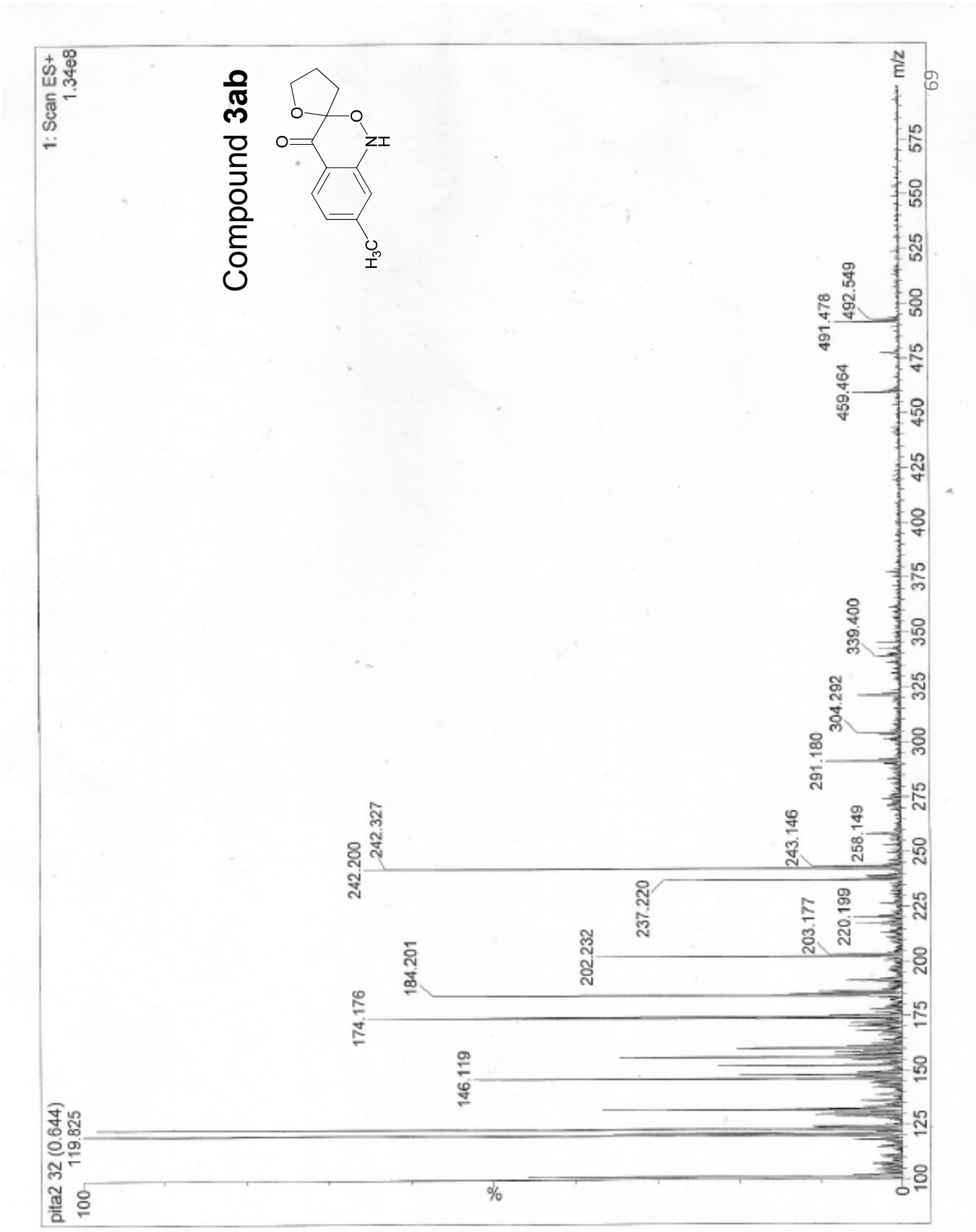
Compound **3aa**

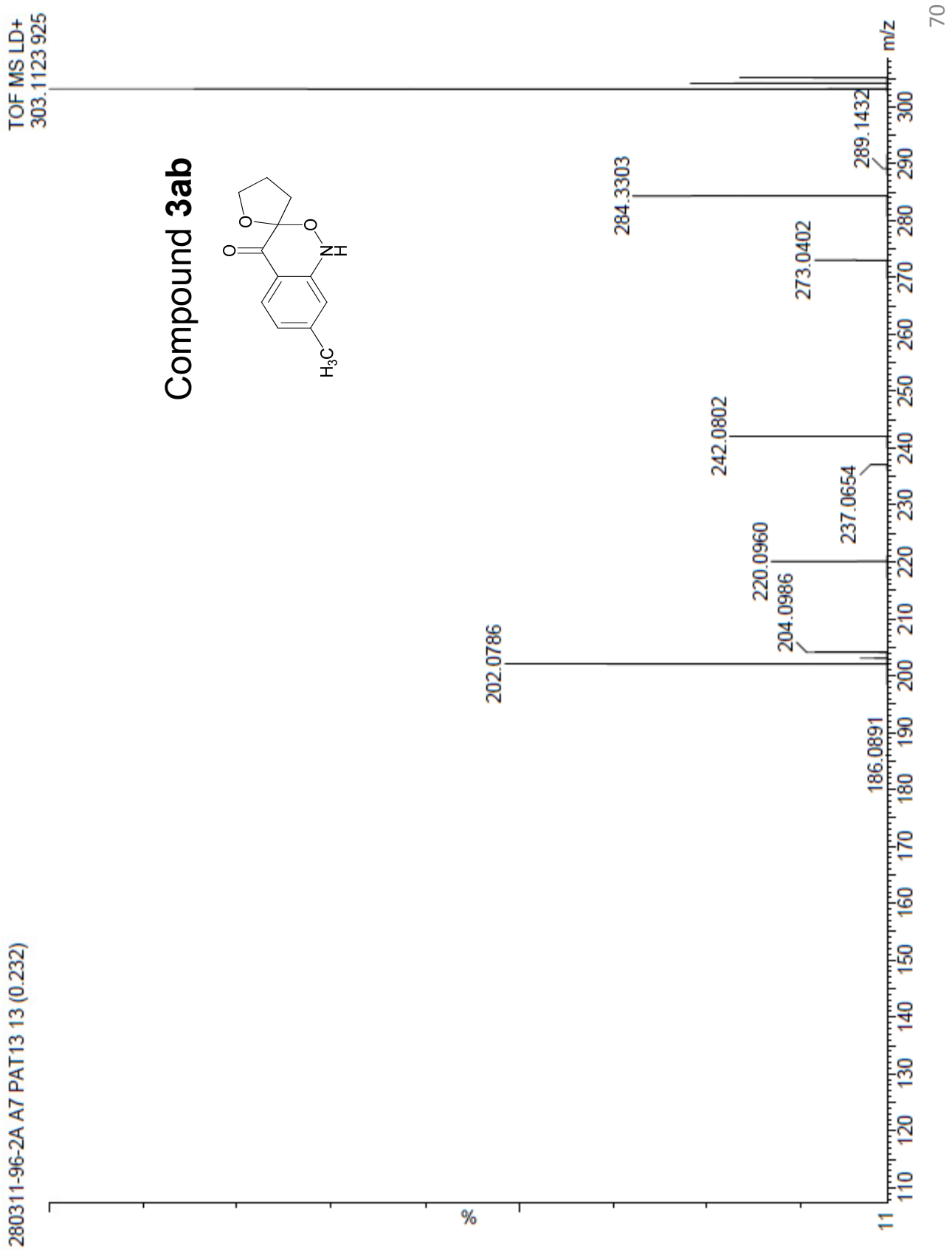


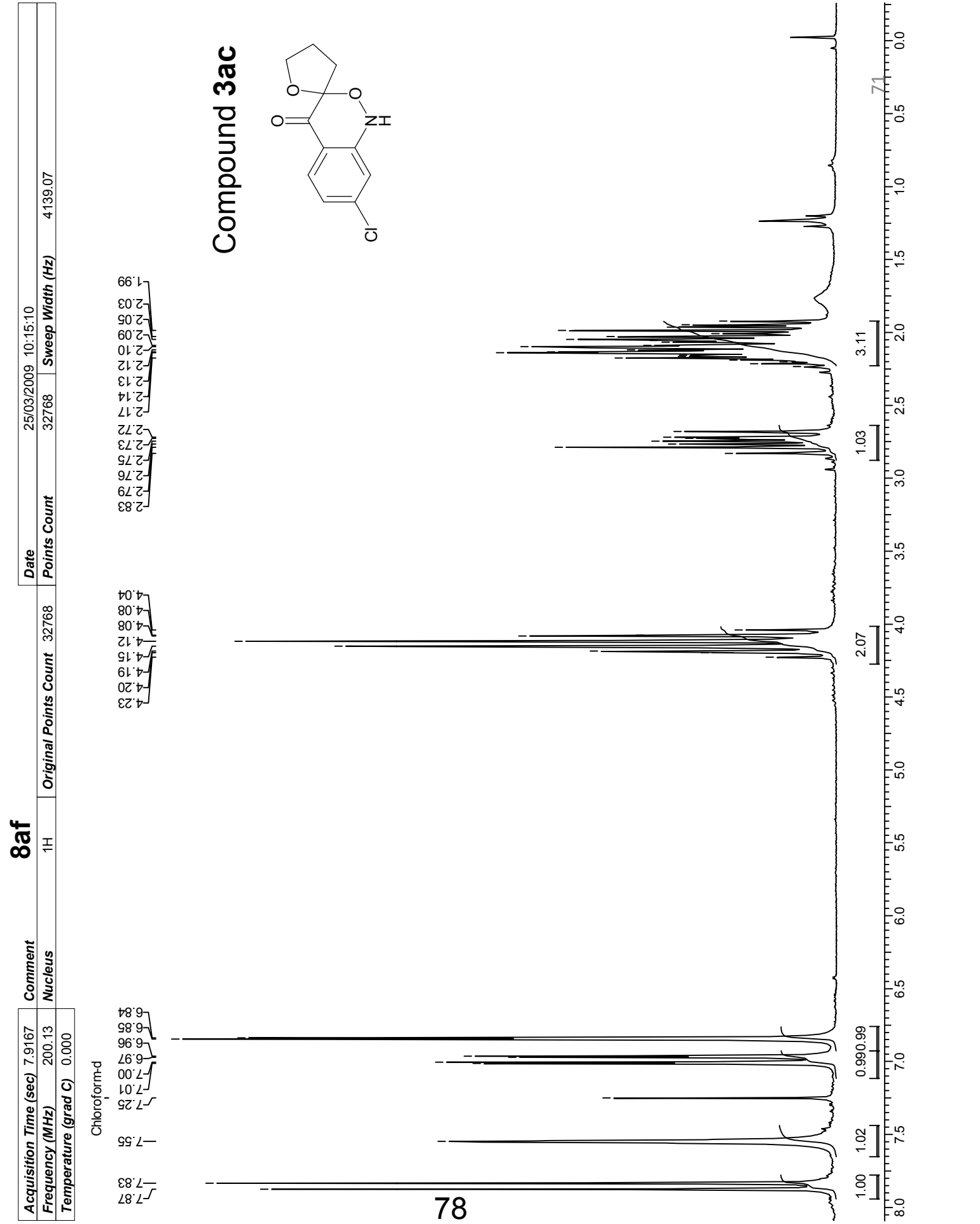


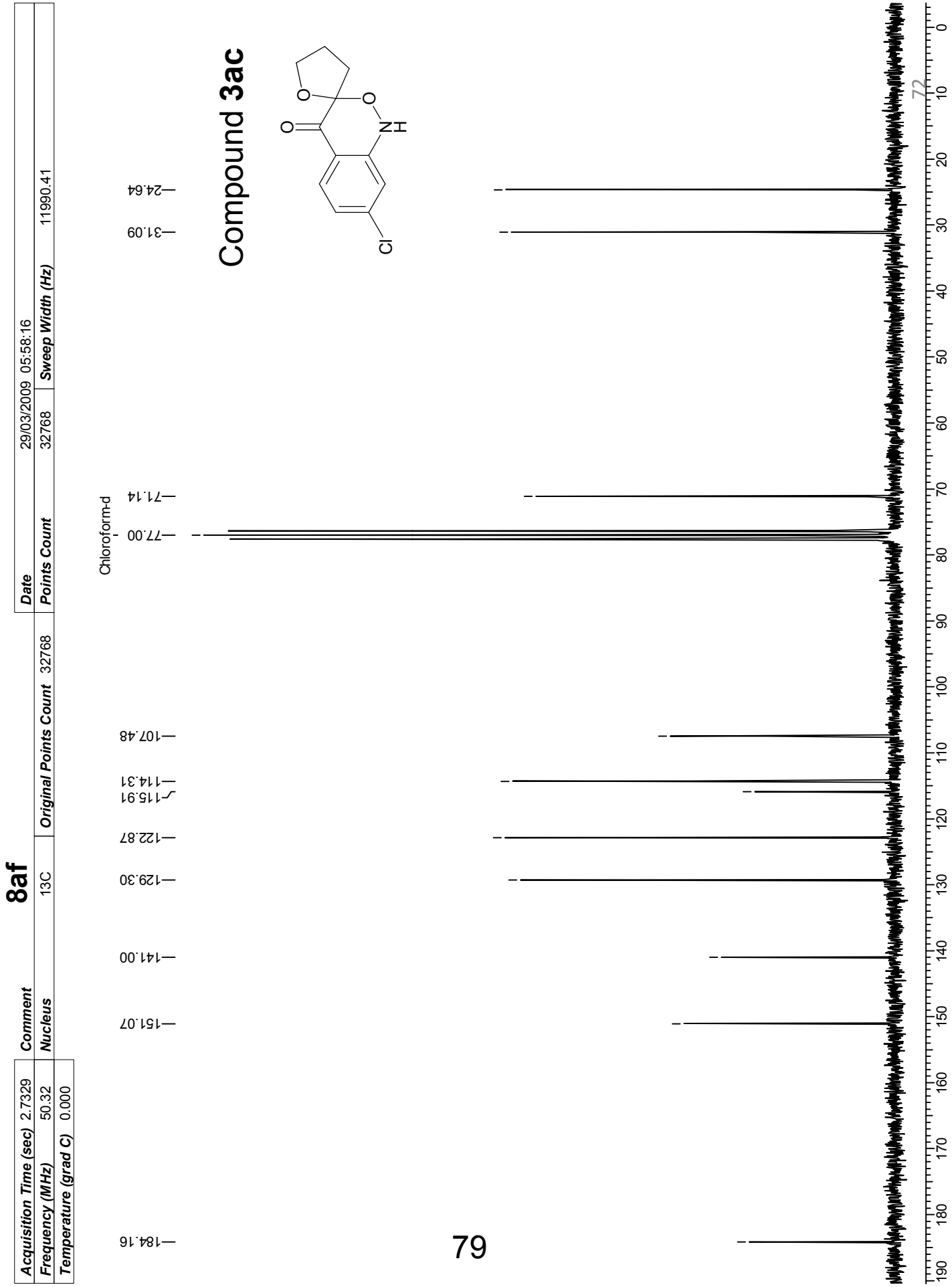


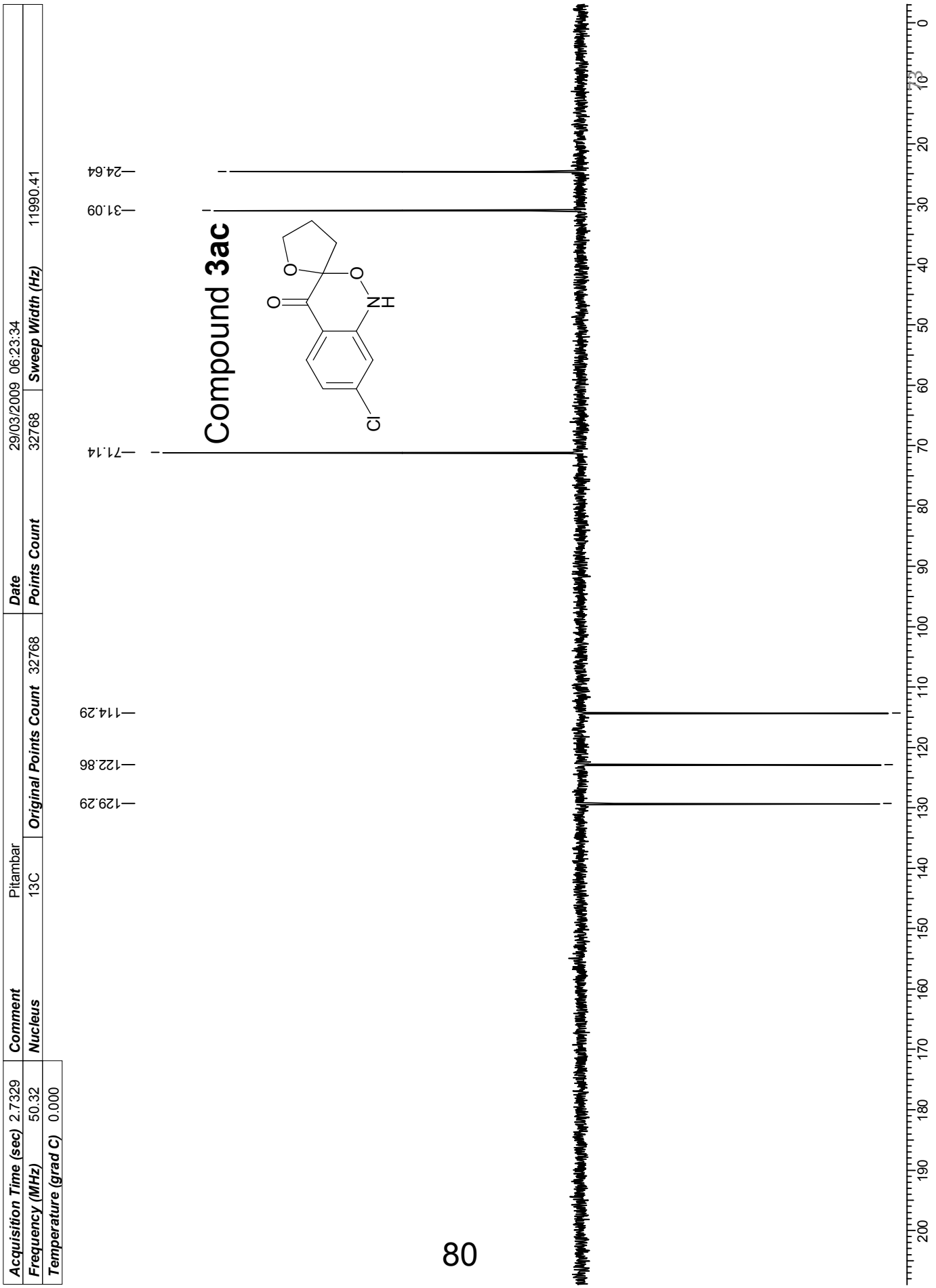


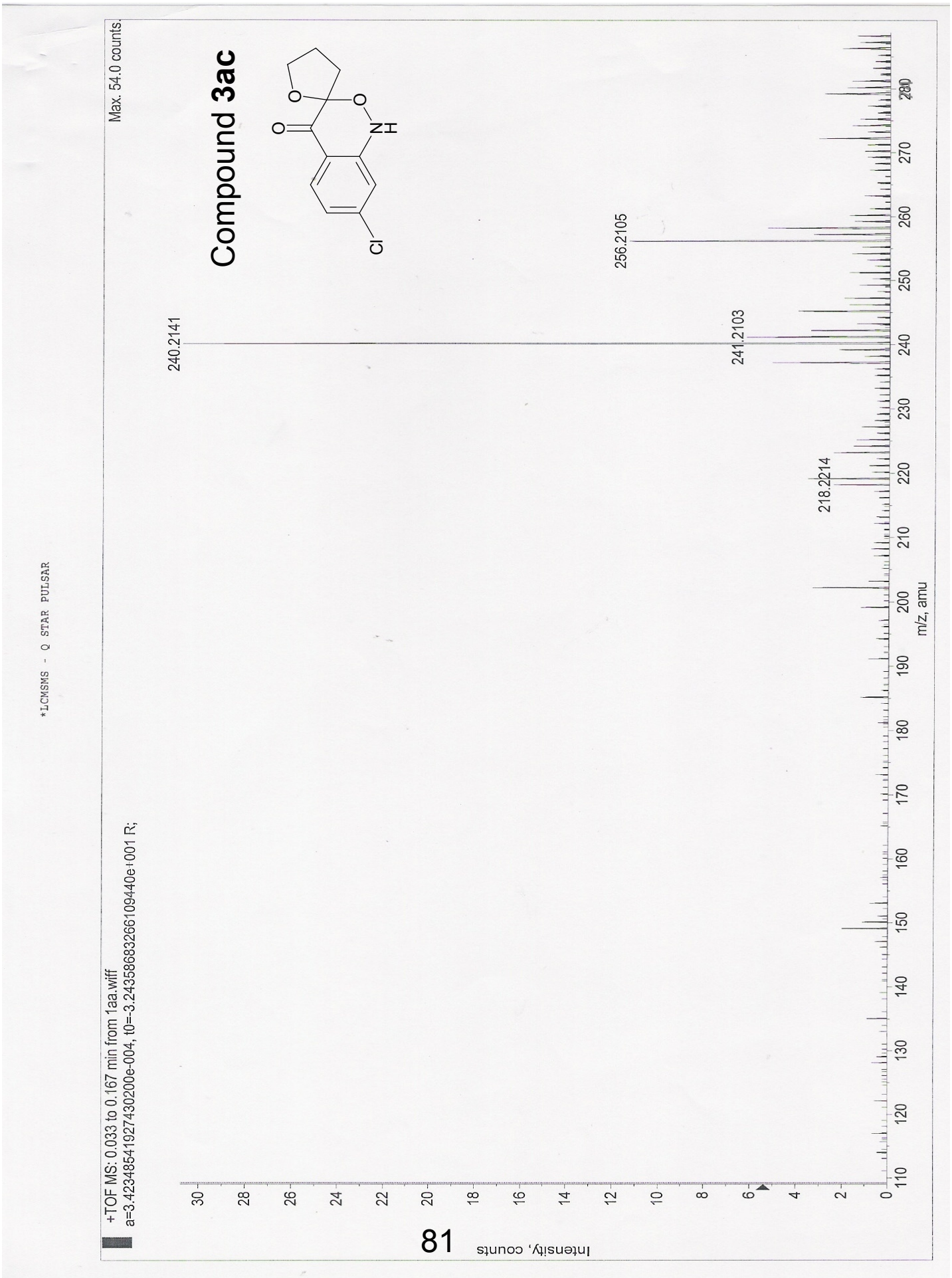


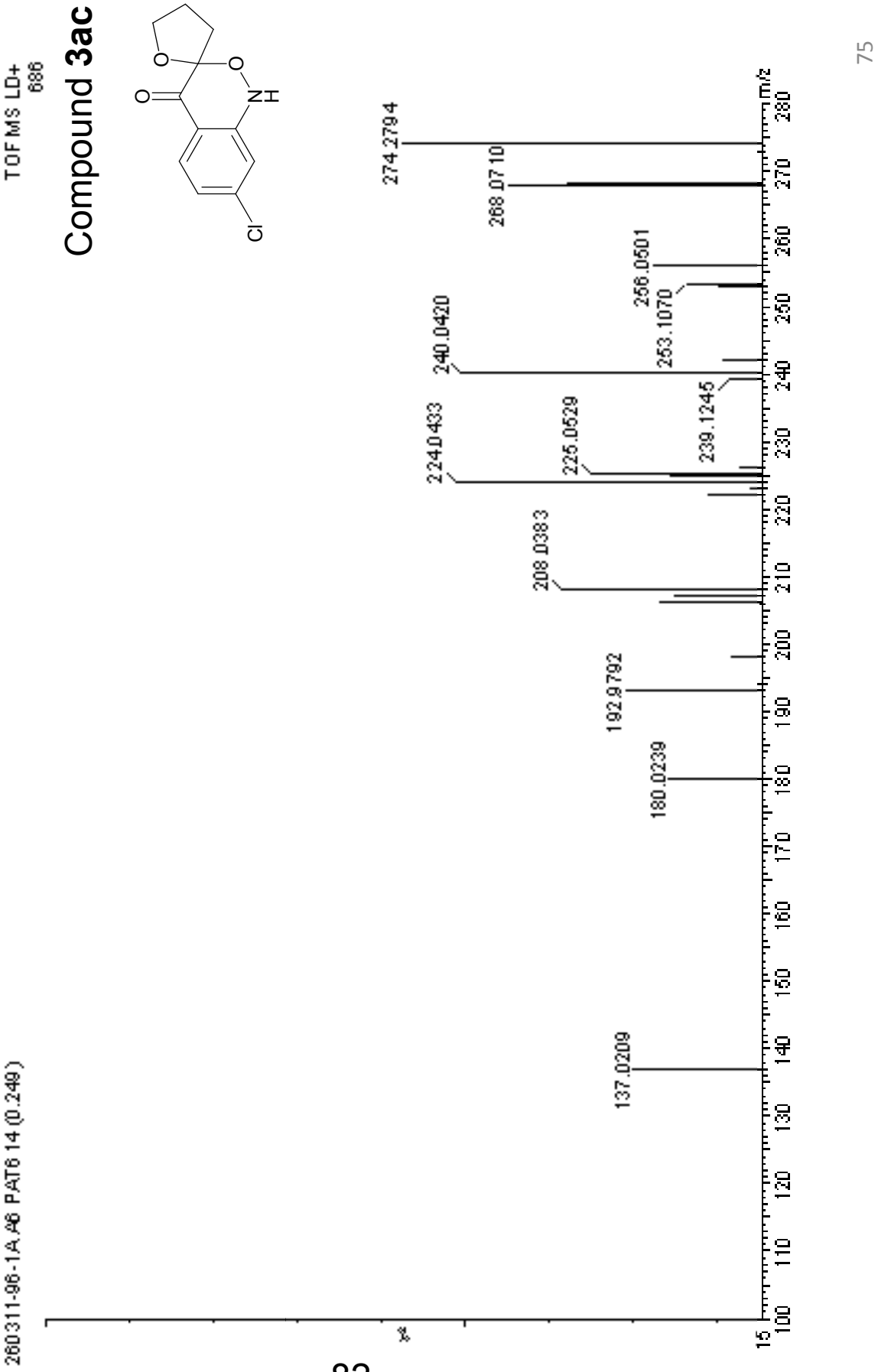


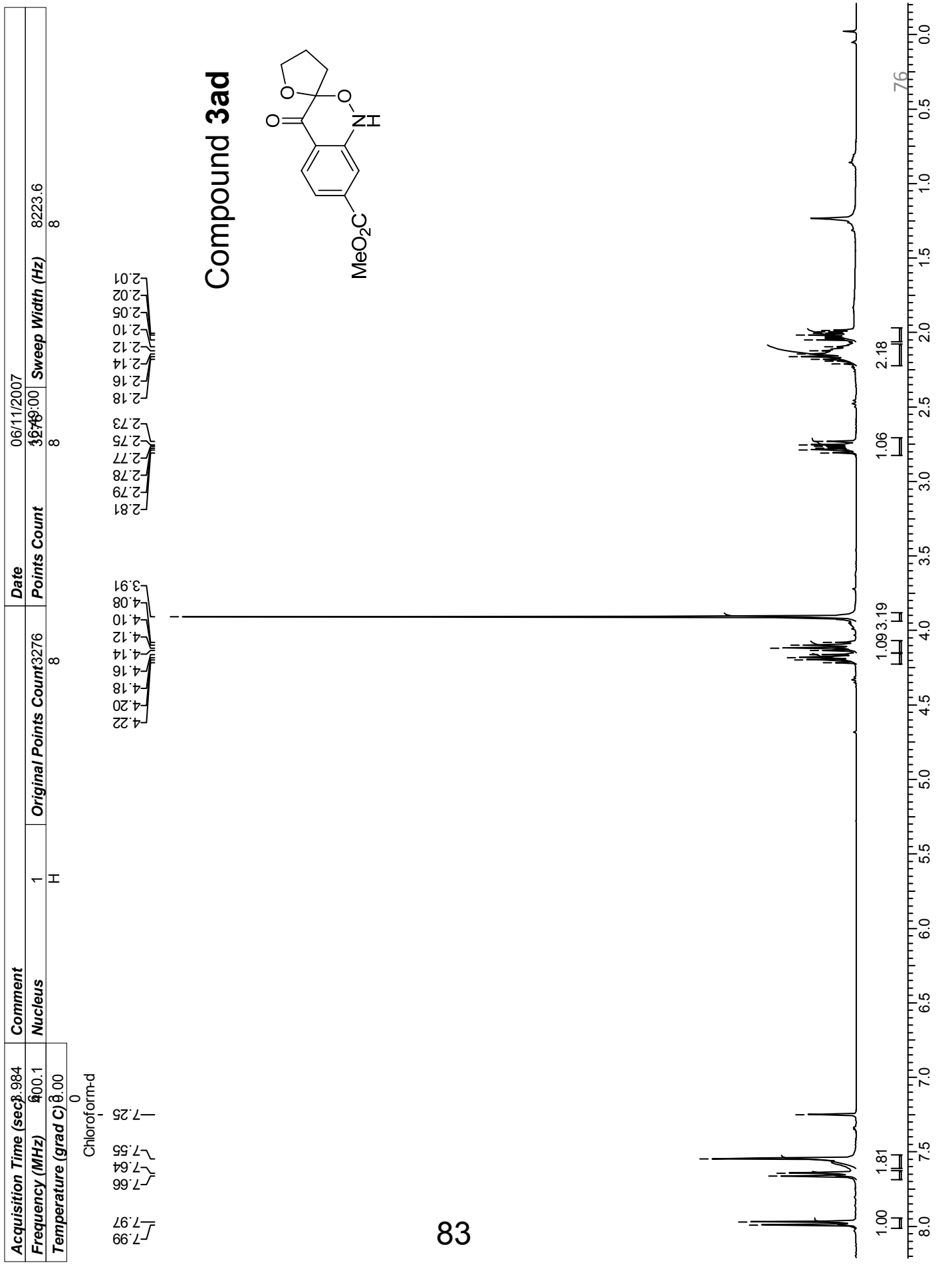


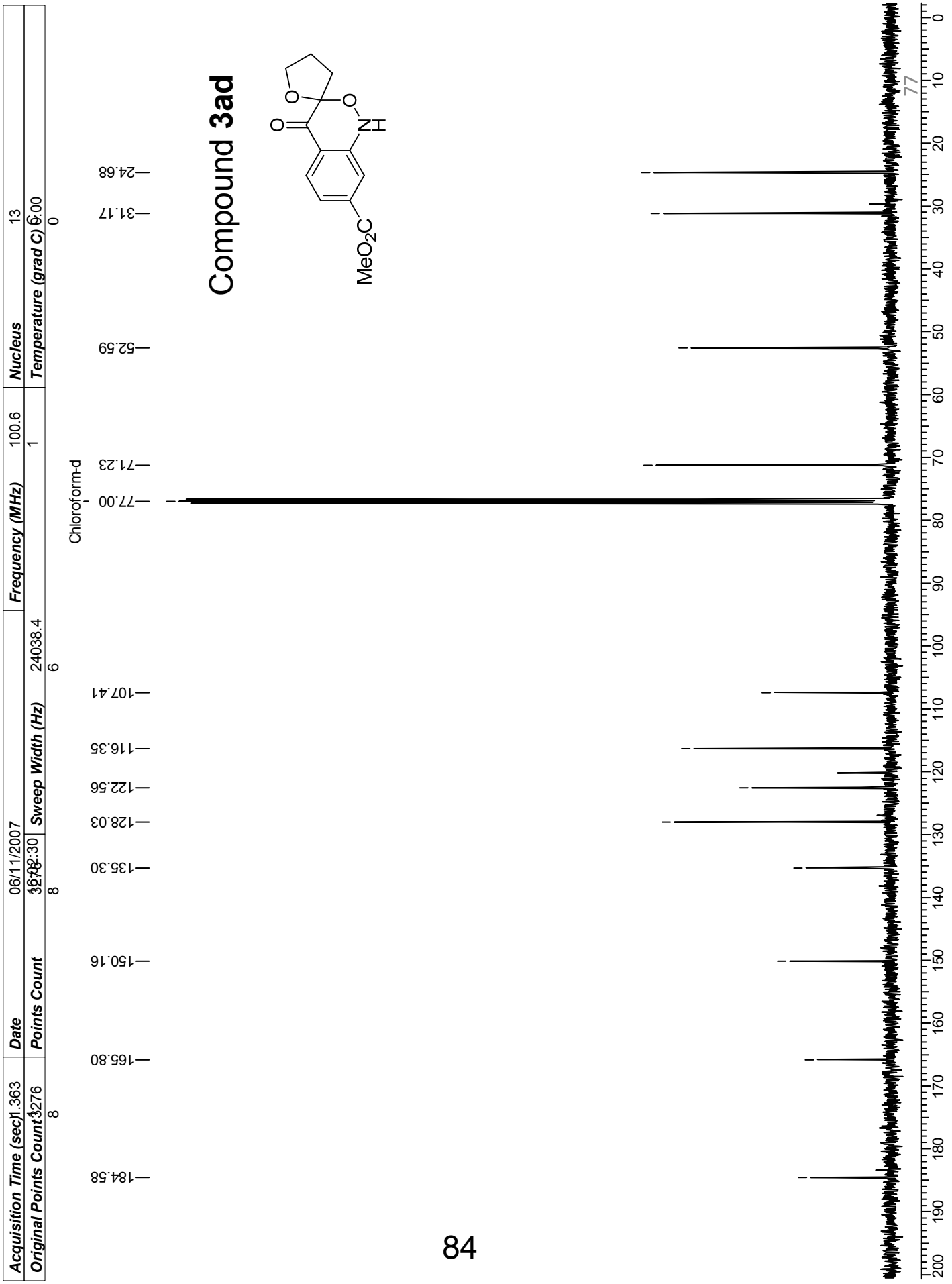


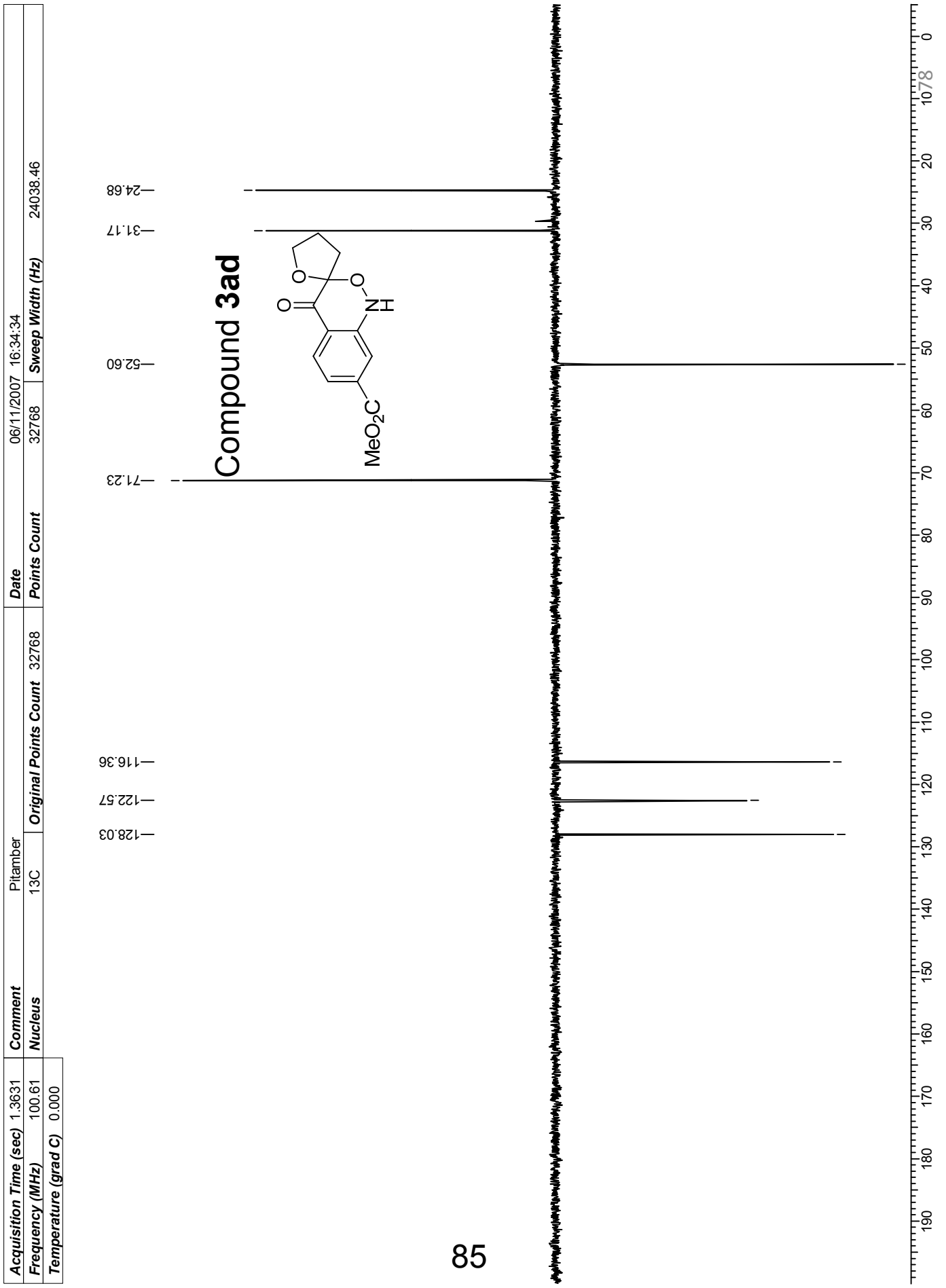

Acquisition Time (sec) 2.7329
Comment
Pitambar
Date 29/03/2009 06:23:34
Frequency (MHz) 50.32
Nucleus 13C
Original Points Count 32768
Points Count 32768
Sweep Width (Hz) 11990.41
Temperature (grad C) 0.000
Compound 3ac
24.64
31.09
71.14
114.29
122.86
129.29
O
O
O
NH
Cl

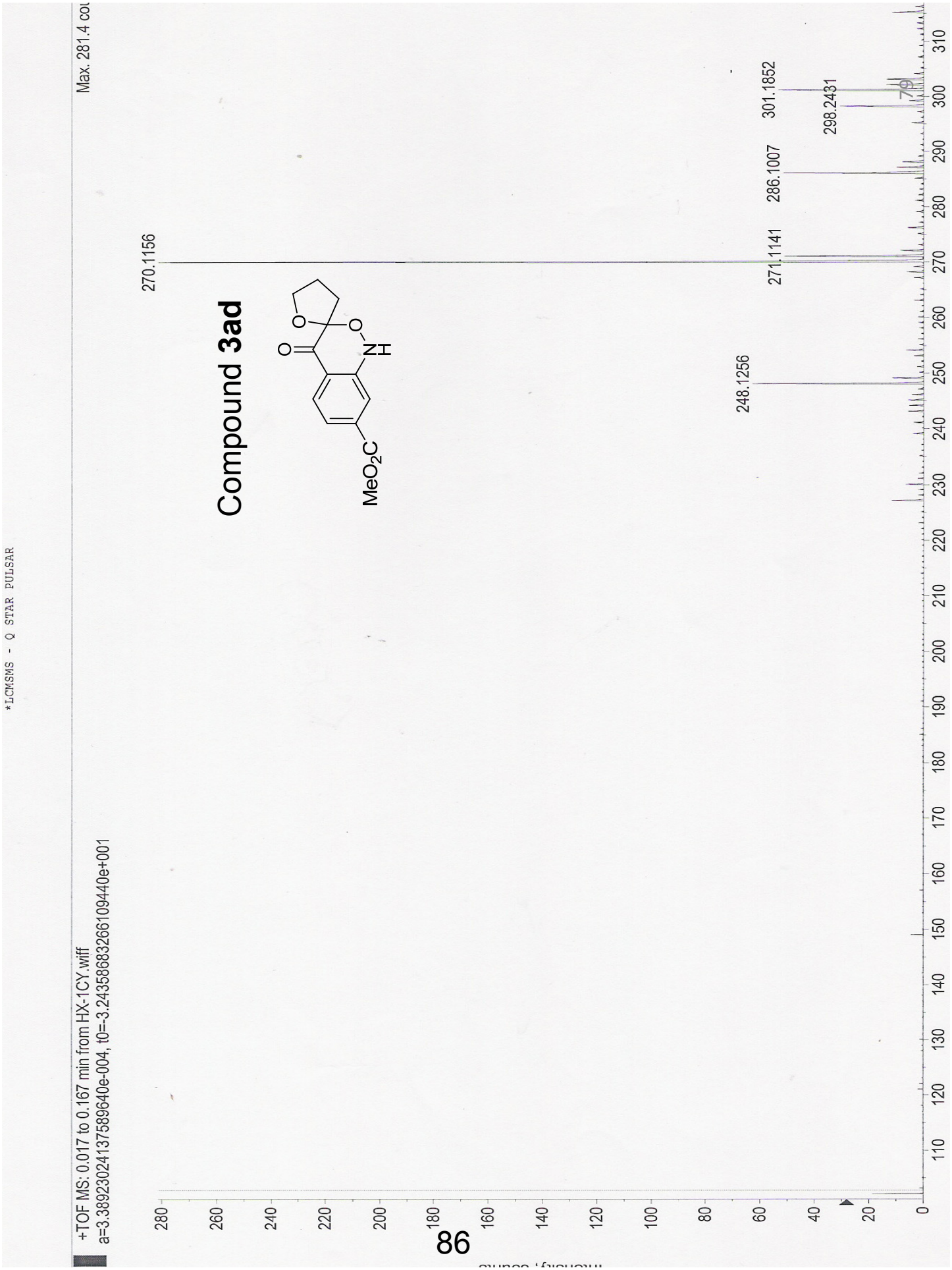


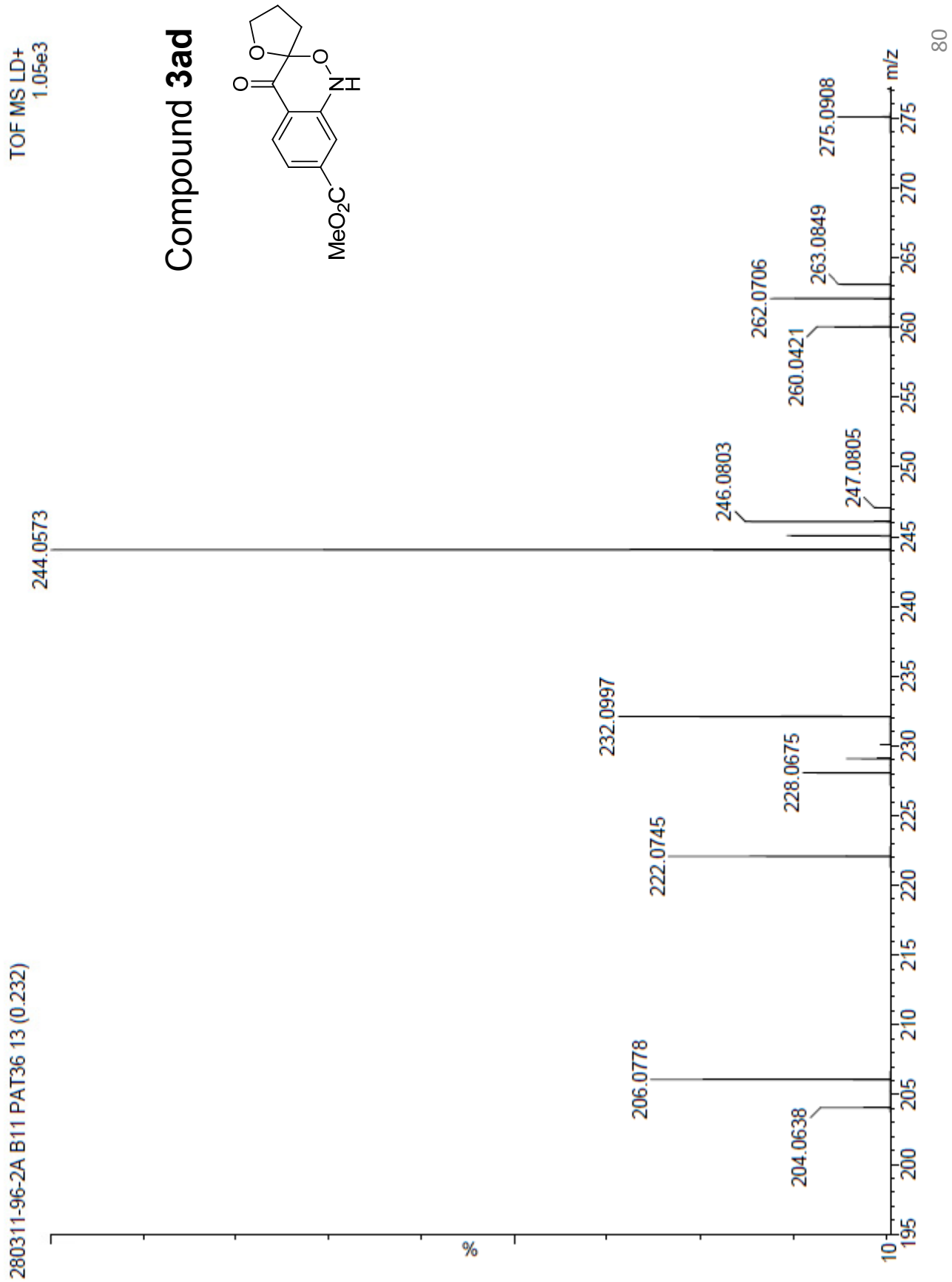

TOF MS LD+
686
Compound 3ac
Cl
260311-96-1A A6 PAT6 14 (0.249)
%
m/z
274.2794
268.0710
256.0501
253.1070
240.0420
239.1245
225.0529
224.0433
208.0383
192.9792
180.0239
137.0209


Compound 3ad
MeO2C
Acquisition Time (sec) 3.984
Frequency (MHz) 400.1
Temperature (grad C) 300.0
Comment
Nucleus 1H
Original Points Count 32768
Points Count 8
Date 06/11/2007
Sweep Width (Hz) 8223.6
Chloroform-d
7.99
7.97
7.66
7.64
7.55
7.25
4.22
4.20
4.18
4.16
4.14
4.12
4.10
4.08
3.91
2.81
2.79
2.78
2.77
2.75
2.73
2.18
2.16
2.14
2.12
2.10
2.05
2.02
2.01
1.00
1.81
1.09
3.19
1.06
2.18

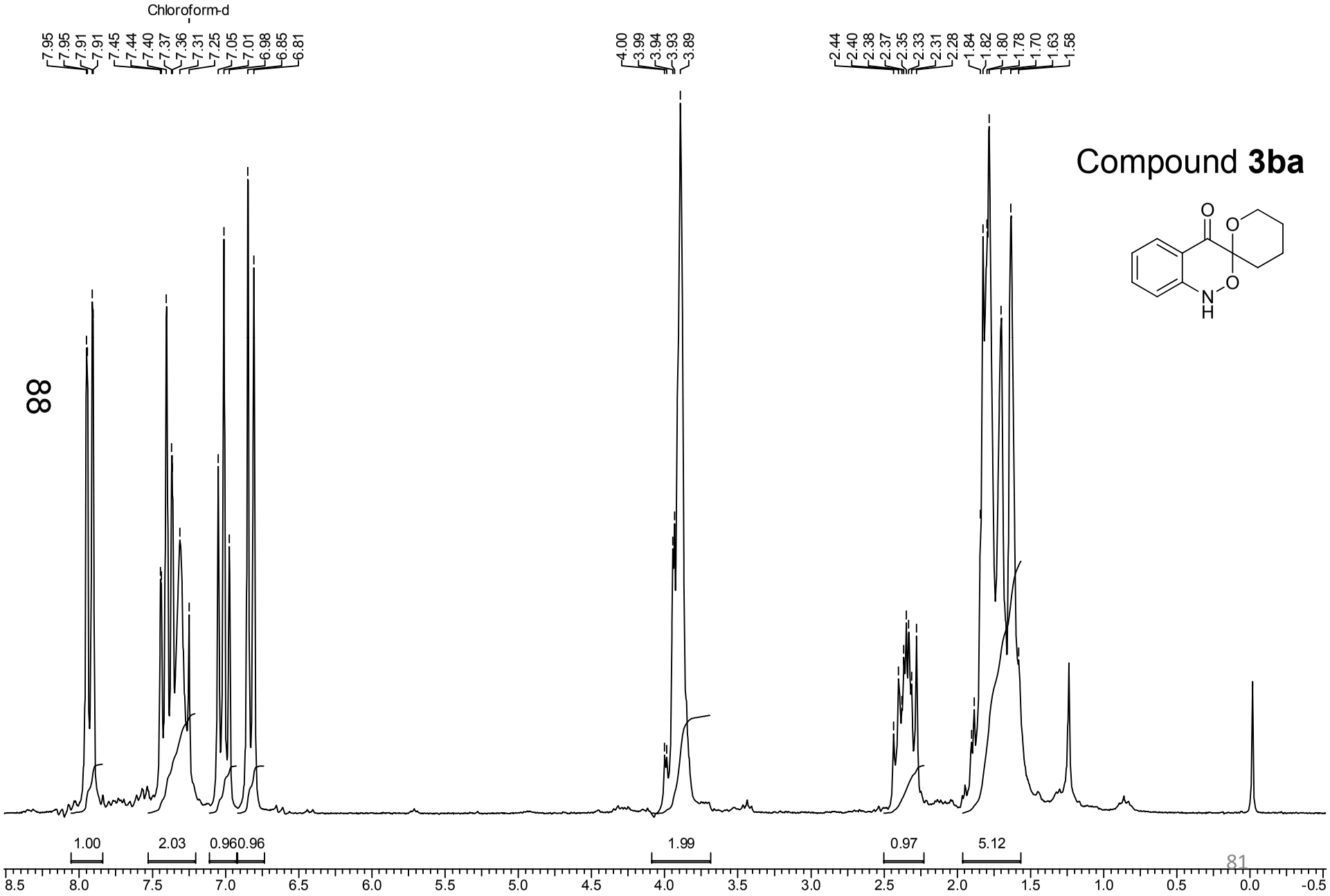


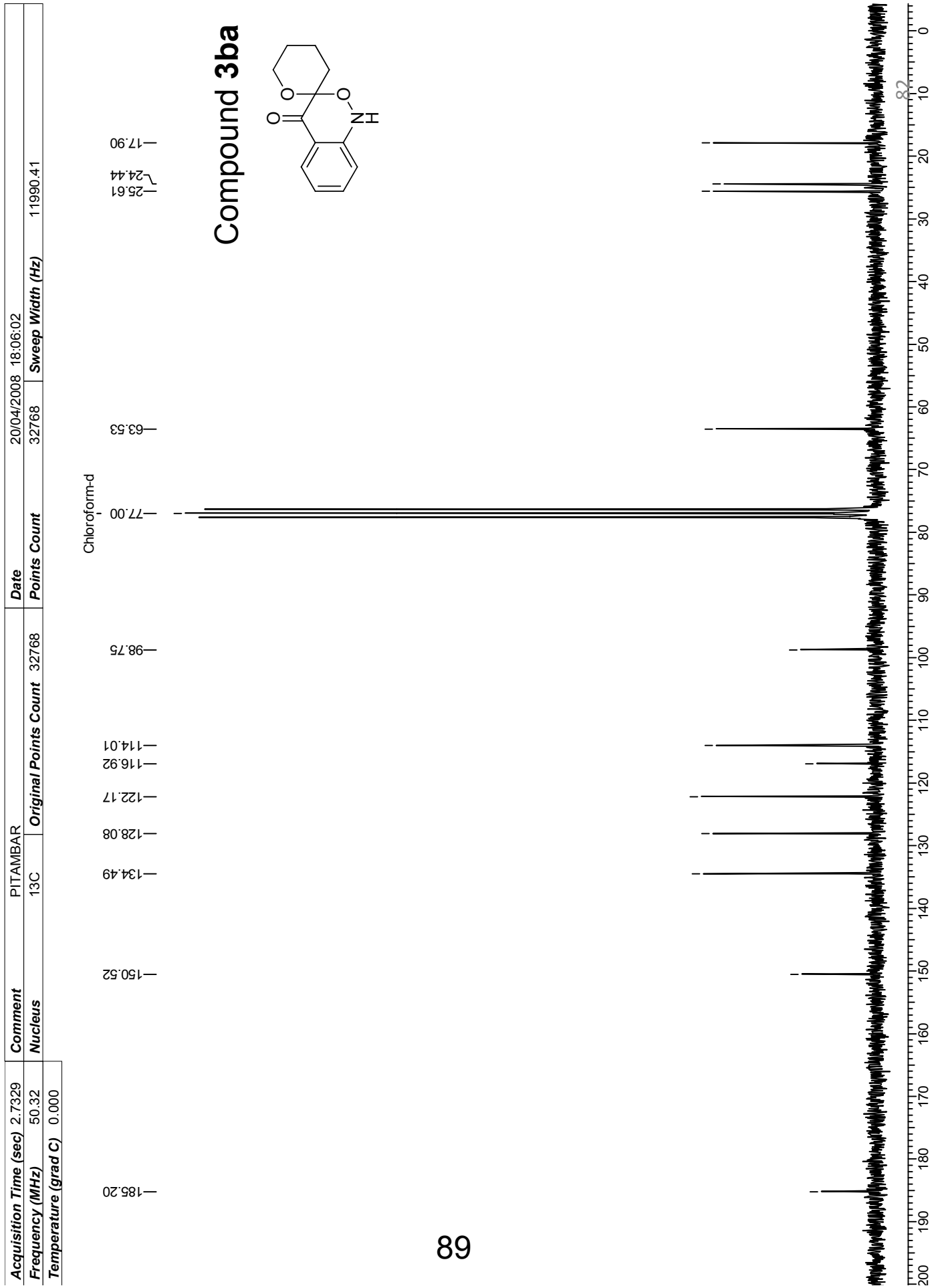


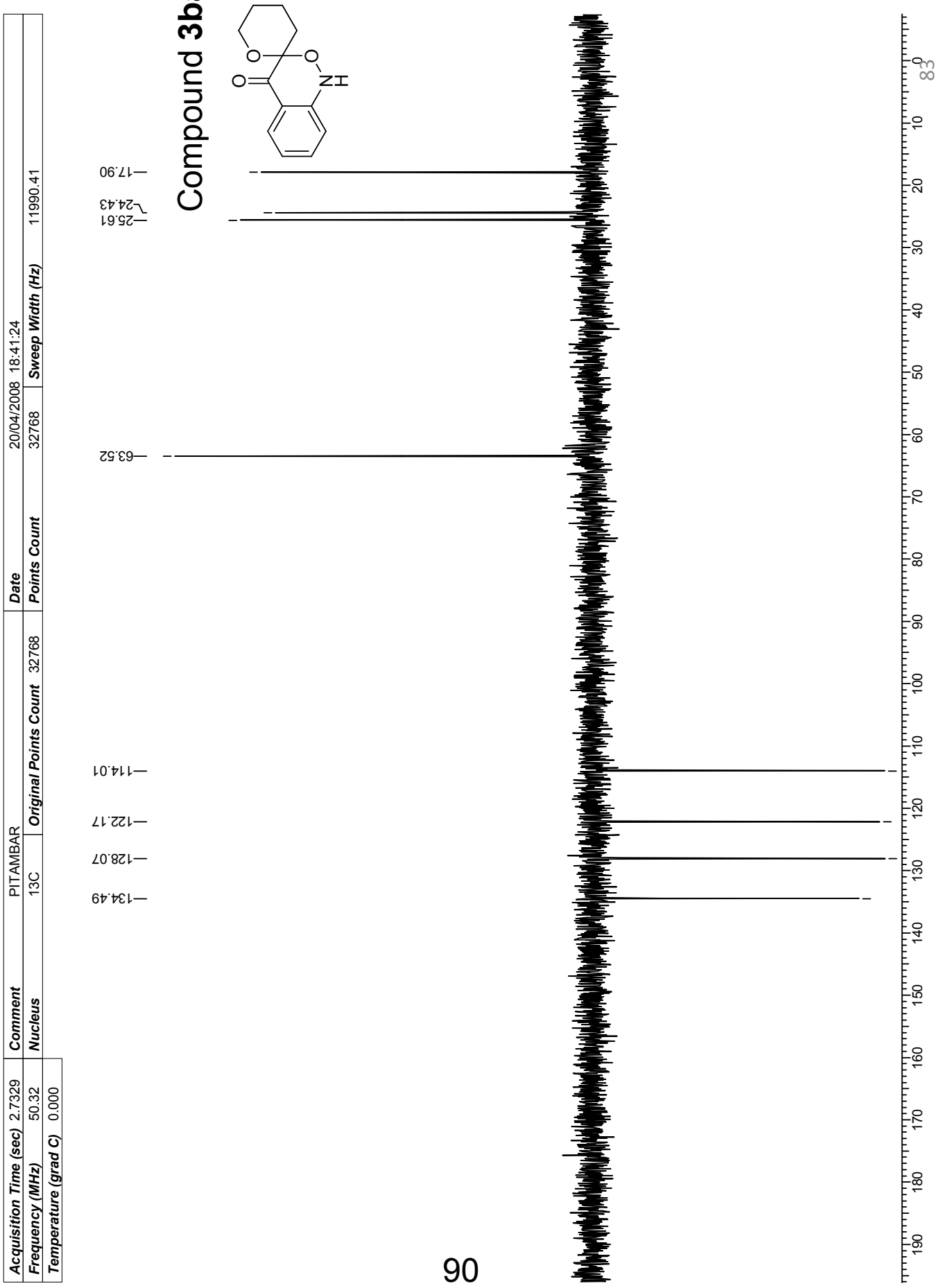


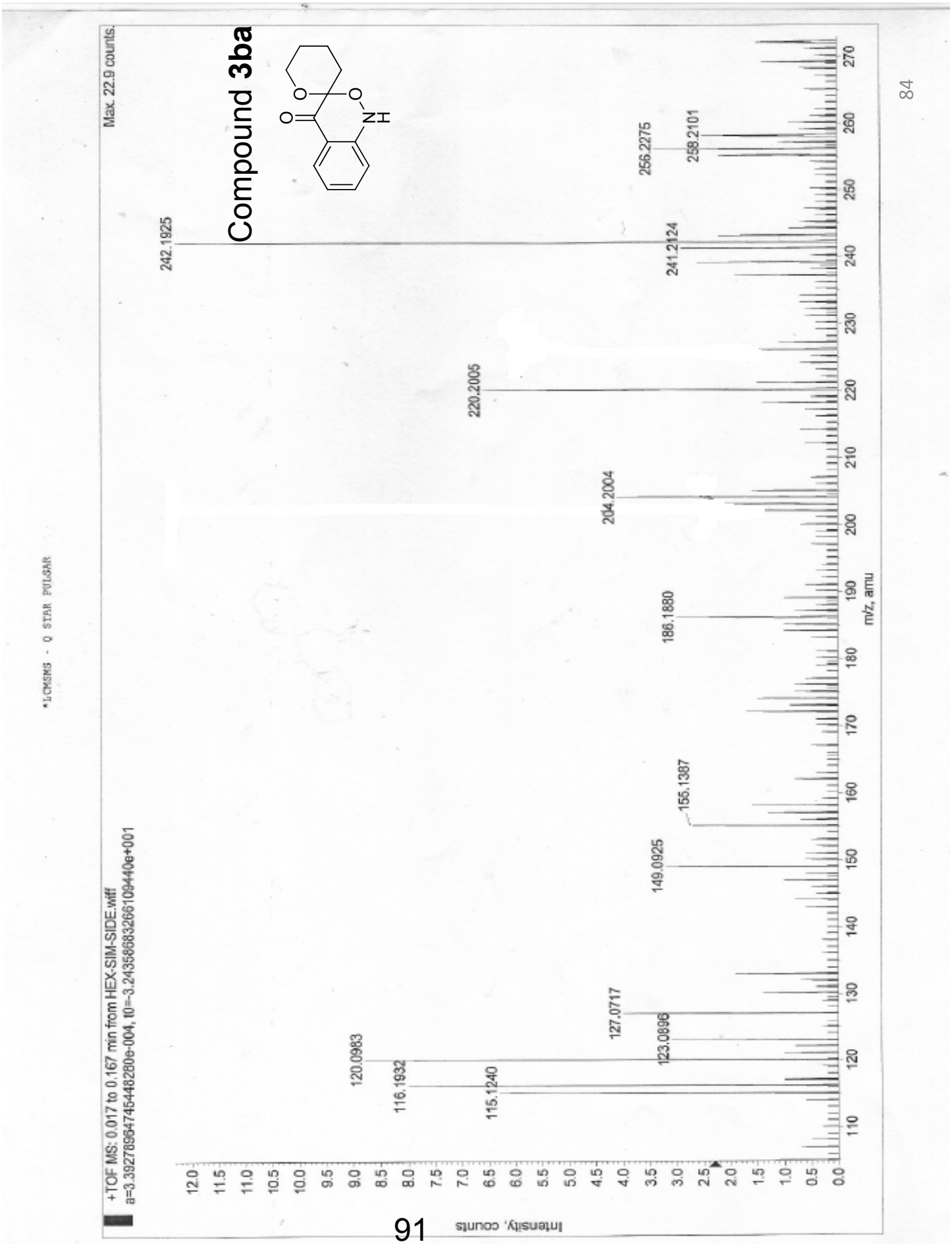


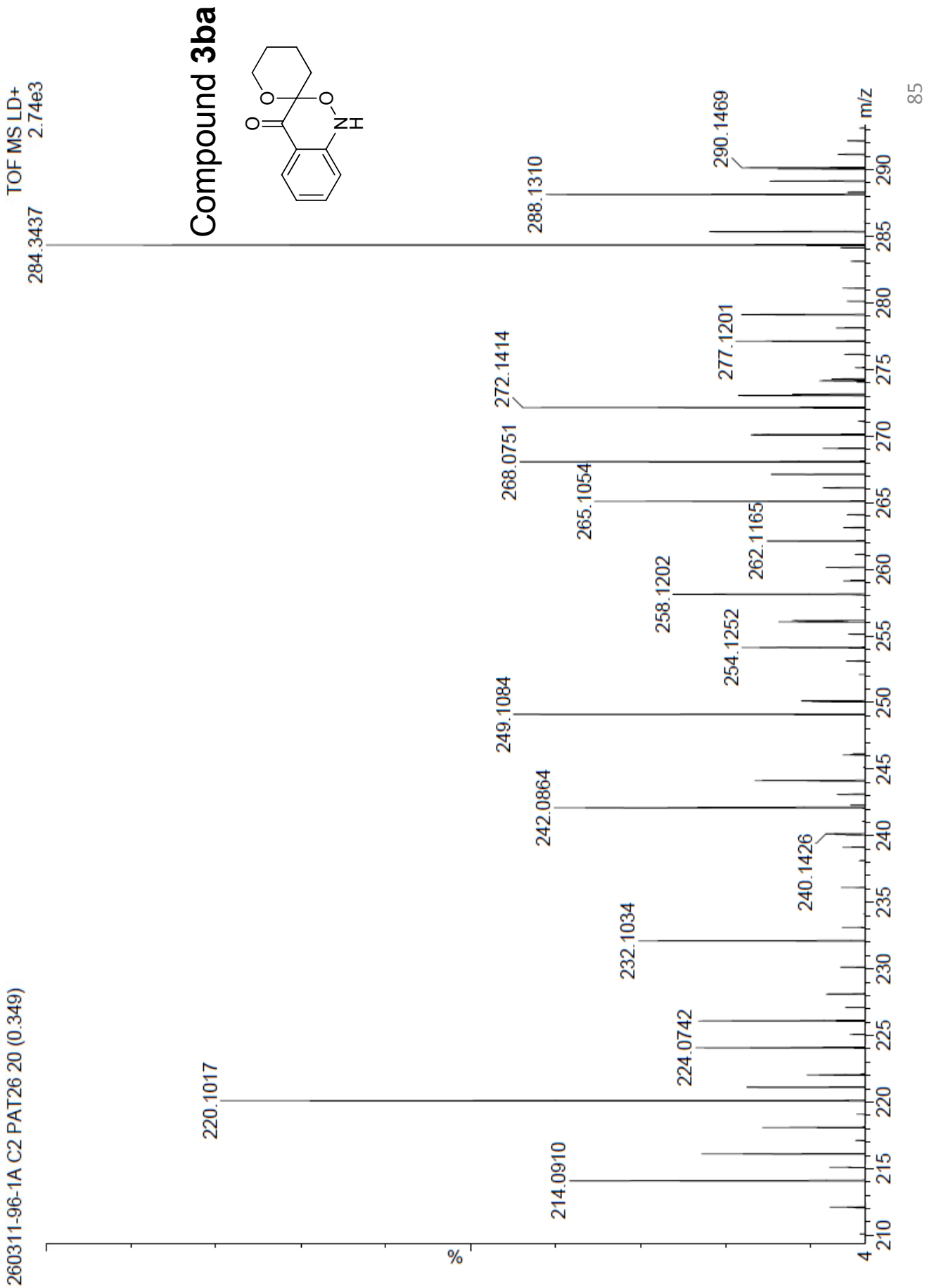
Acquisition Time (sec)	7.9167	***Comment***	Narendra/11182		***Date***	17/05/2011 14:44:50	
Frequency (MHz)	200.13	***Nucleus***	1H	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 4139.07
Temperature (grad C)	0.000						



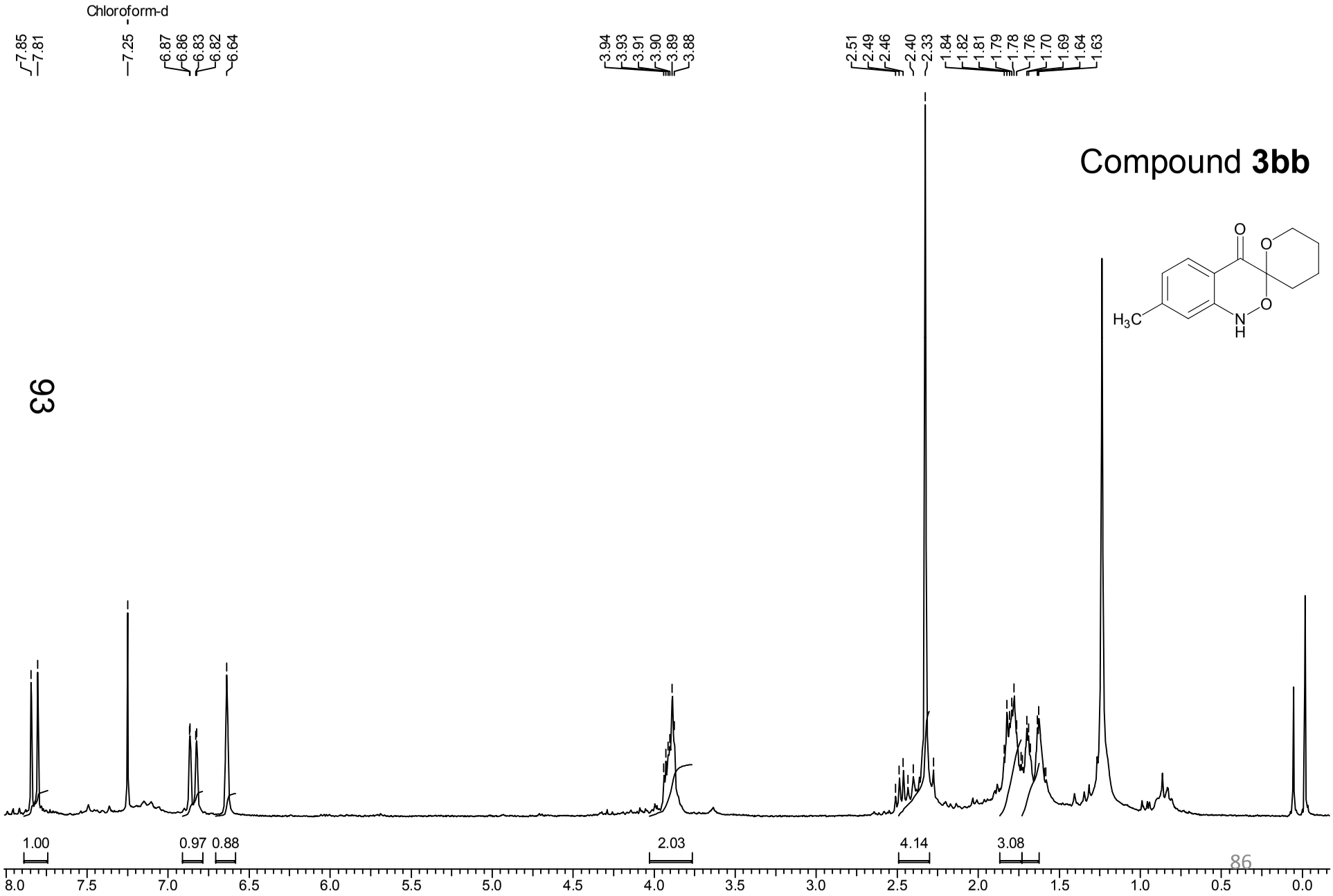

Compound 3ba
Acquisition Time (sec) 2.7329
Frequency (MHz) 50.32
Temperature (grad C) 0.000
Comment
Nucleus
PITAMBAR
13C
Original Points Count 32768
Date
Points Count
20/04/2008 18:06:02
32768
Sweep Width (Hz) 11990.41
Chloroform-d
17.90
24.44
25.61
63.53
77.00
98.75
114.01
116.92
122.17
128.08
134.49
150.52
185.20
82



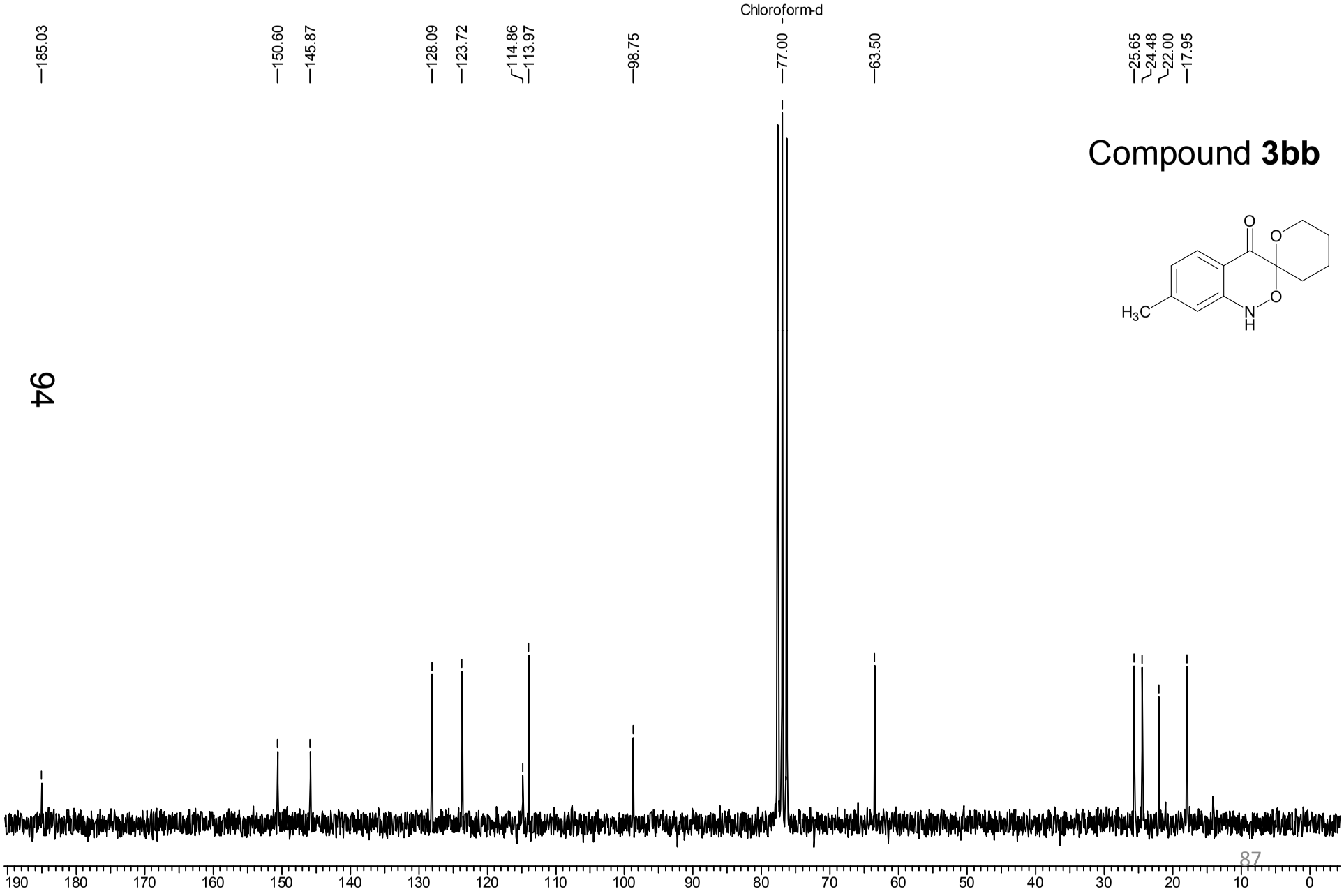

Compound 3ba
+TOF MS: 0.017 to 0.167 min from HEX-SIM-SIDE.wiff
a=3.39278964745448280e-004, t0=-3.24356683266109440e+001
Max. 22.9 counts.
*LCMSMS - Q STAR PULSAR
242.1925
241.2124
256.2275
258.2101
220.2005
204.2004
186.1880
155.1387
149.0925
127.0717
123.0896
120.0983
116.1932
115.1240
Intensity, counts
m/z, amu

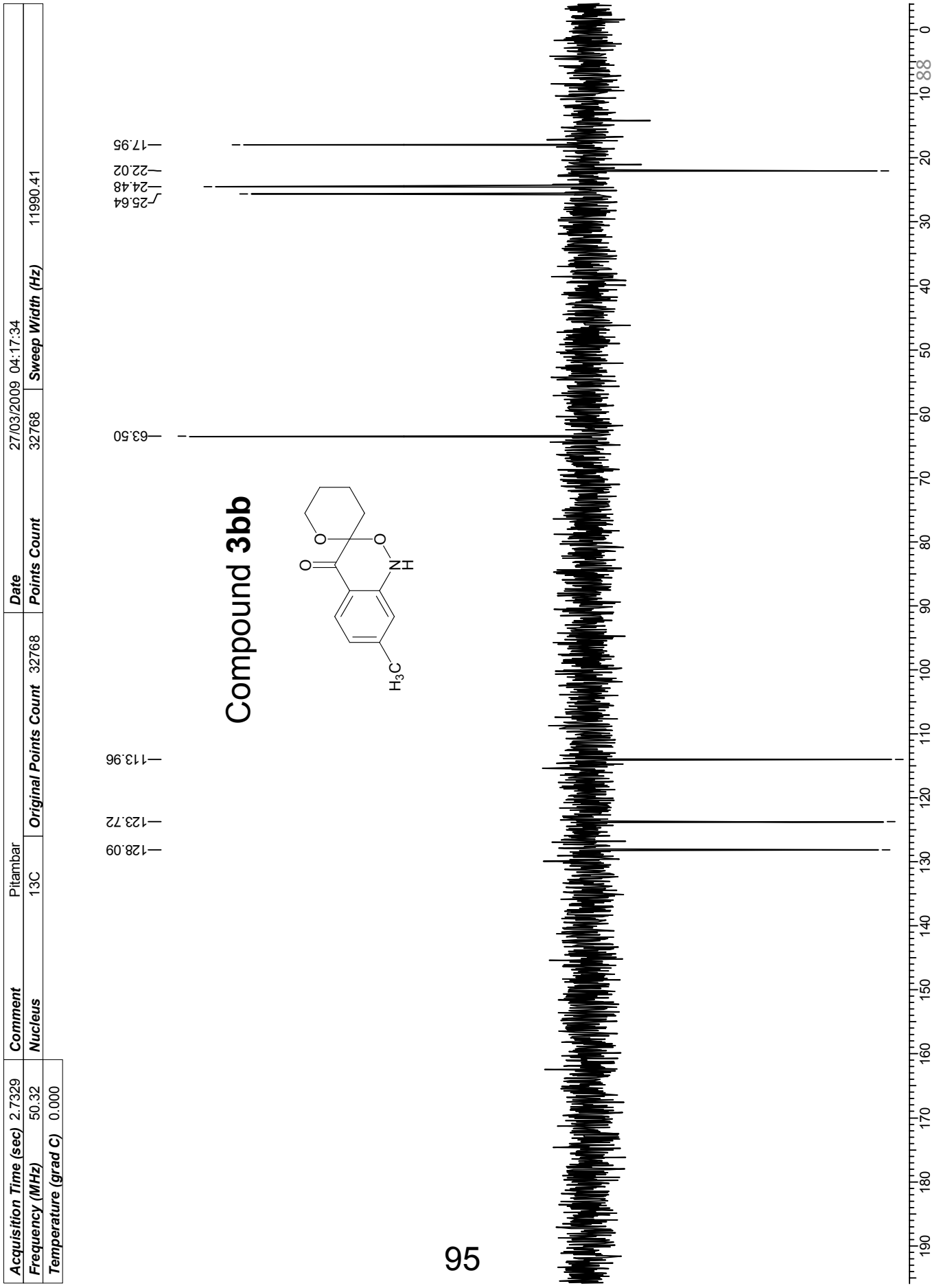


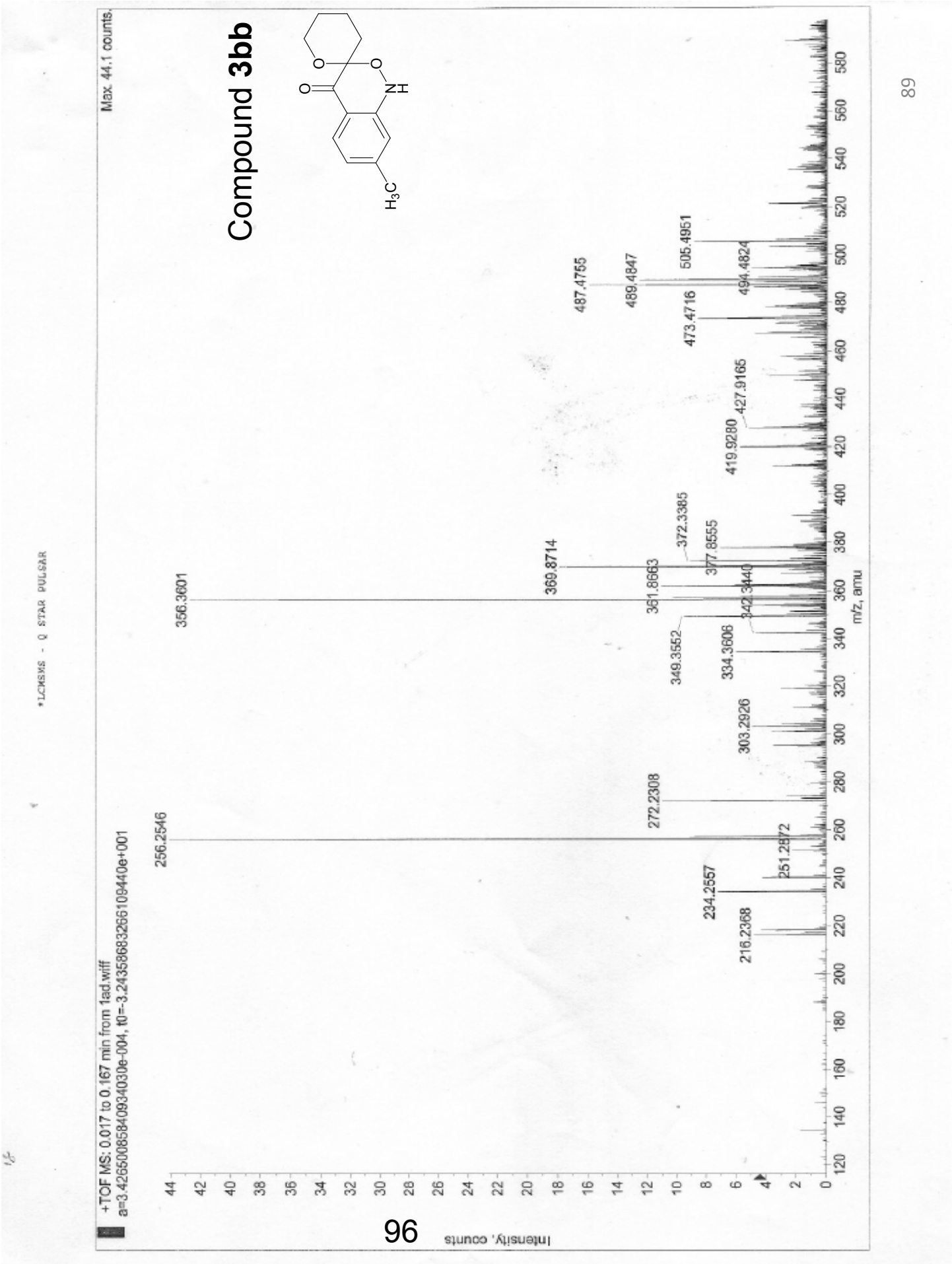
Acquisition Time (sec)	7.9167	***Comment***	**8be**		***Date***	19/06/2009 15:23:04	
Frequency (MHz)	200.13	***Nucleus***	1H	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 4139.07
Temperature (grad C)	0.000						



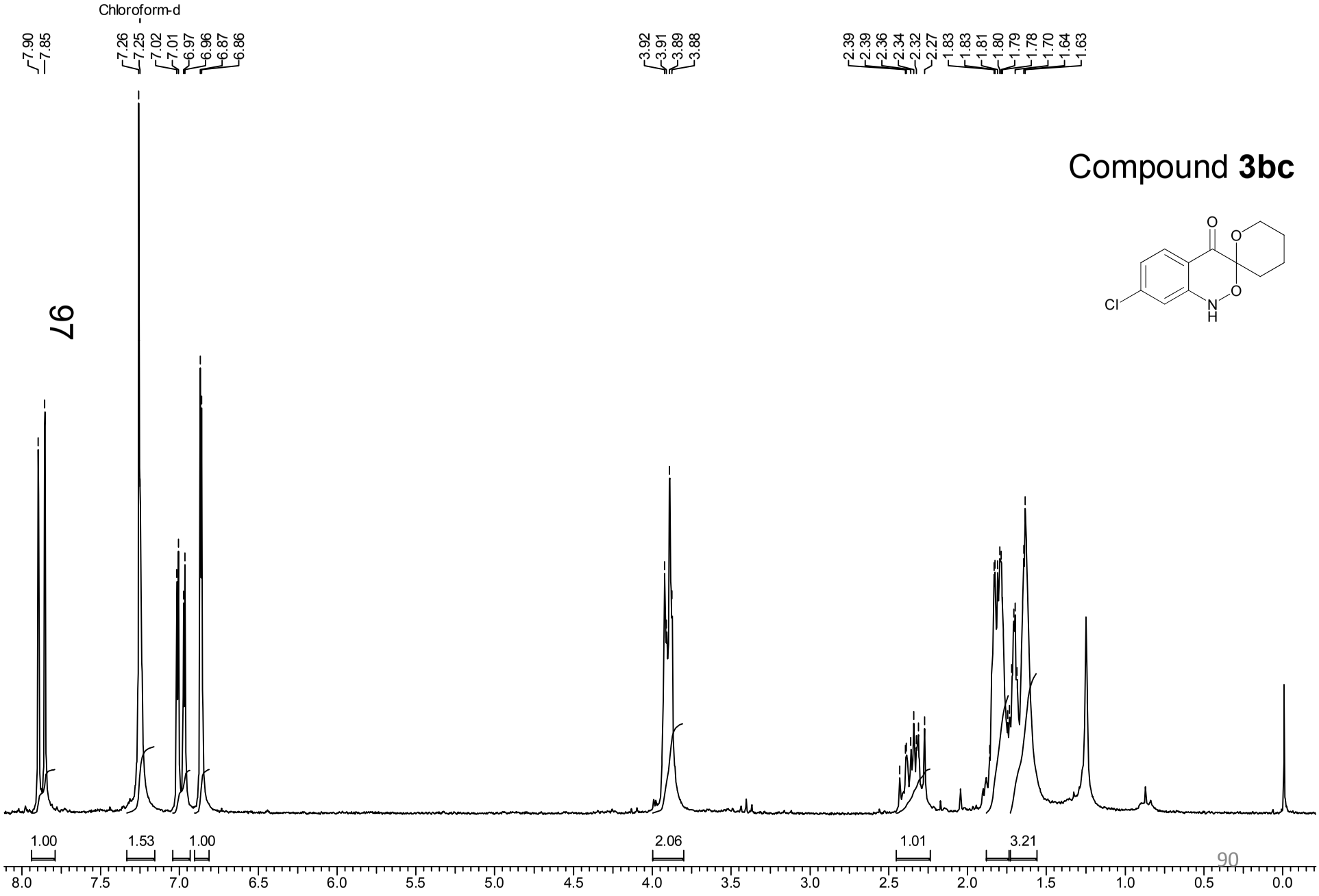
Acquisition Time (sec)	2.7329	***Comment***	**8be**		***Date***	27/03/2009 03:53:44	
Frequency (MHz)	50.32	***Nucleus***	13C	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 11990.41
Temperature (grad C)	0.000						





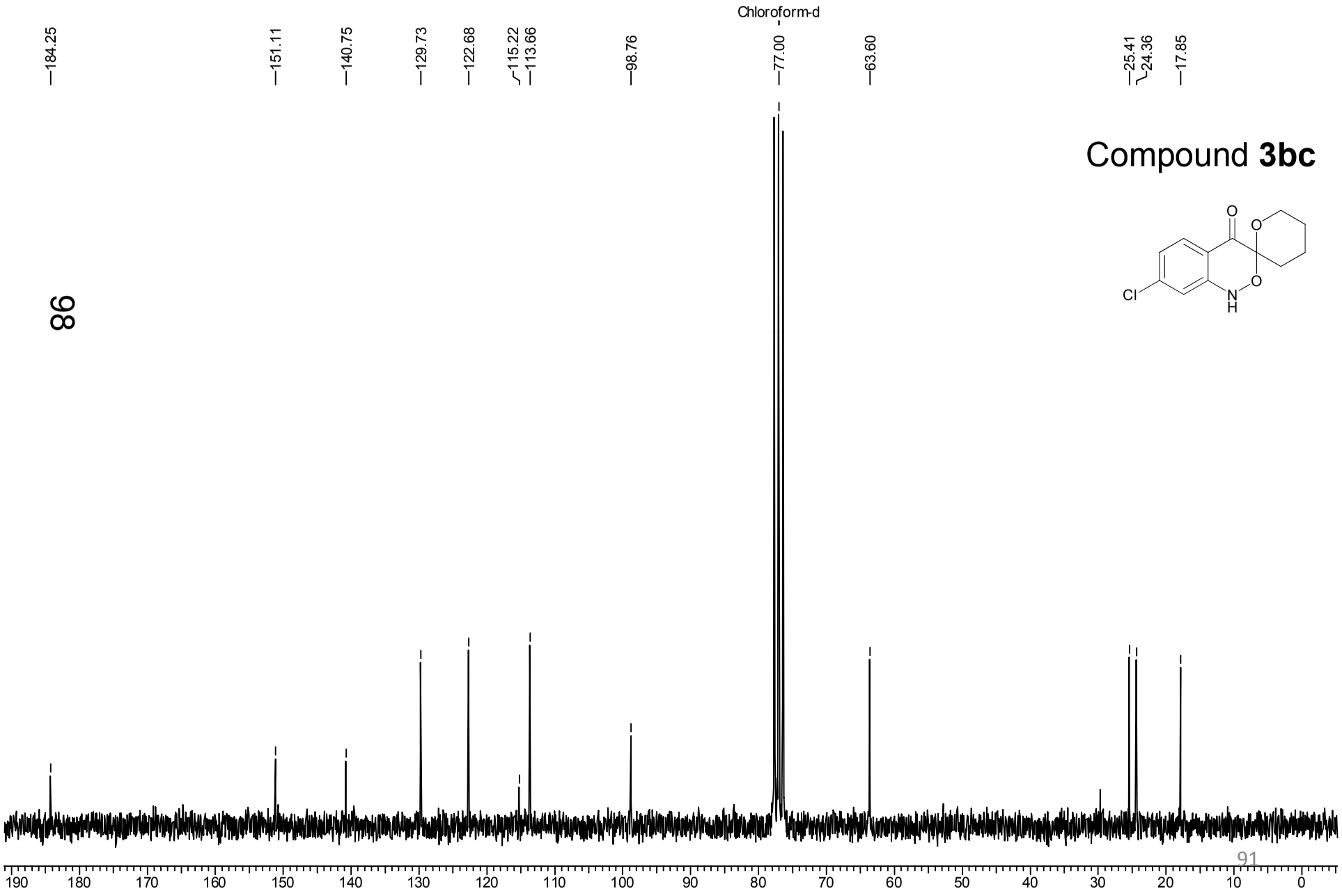

Compound 3bb
+TOF MS: 0.017 to 0.167 min from 1ad.wiff
a=3.42650085840934030e-004, t0=-3.24358683266109440e+001
Max. 44.1 counts.
Intensity, counts
m/z, amu
256.2546
356.3601
369.8714
487.4755
489.4847
505.4951
473.4716
494.4824
419.9280
427.9165
372.3385
377.8555
361.8663
349.3552
342.3440
334.3606
303.2926
272.2308
251.2572
234.2557
216.2368

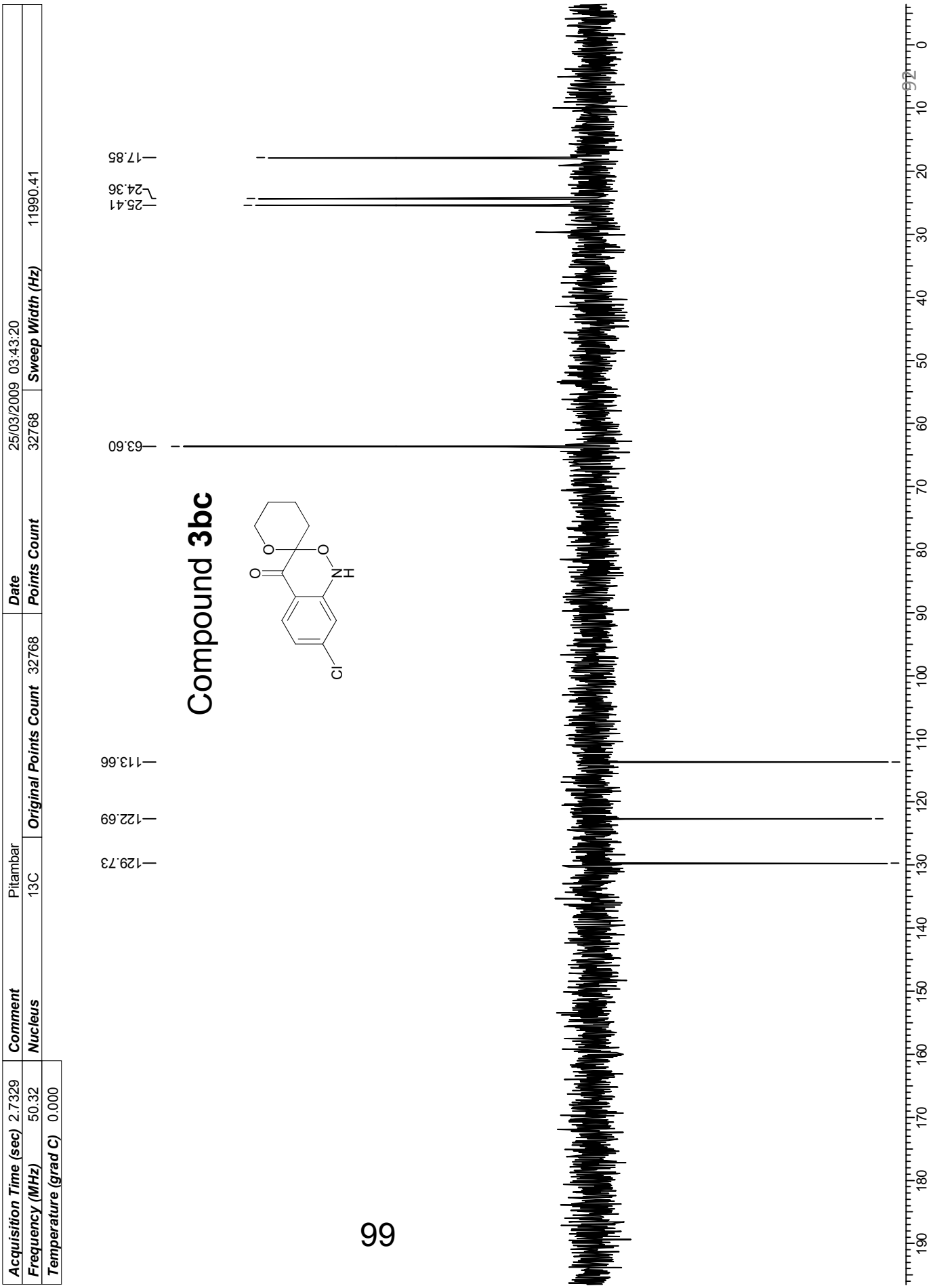
Acquisition Time (sec)	7.9167	***Comment***	**8bf**		***Date***	28/01/2009 17:46:42	
Frequency (MHz)	200.13	***Nucleus***	1H	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 4139.07
Temperature (grad C)	0.000						

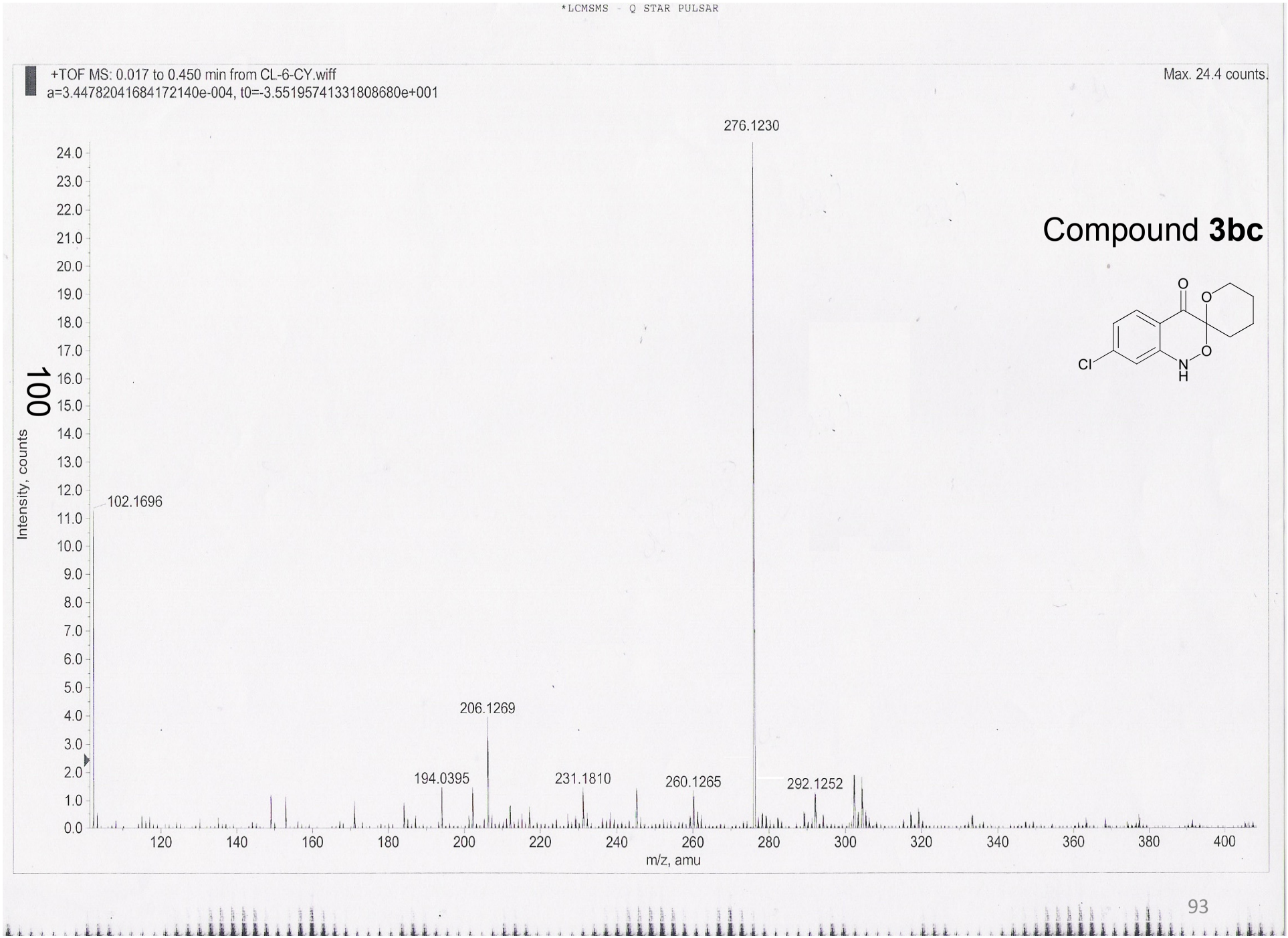


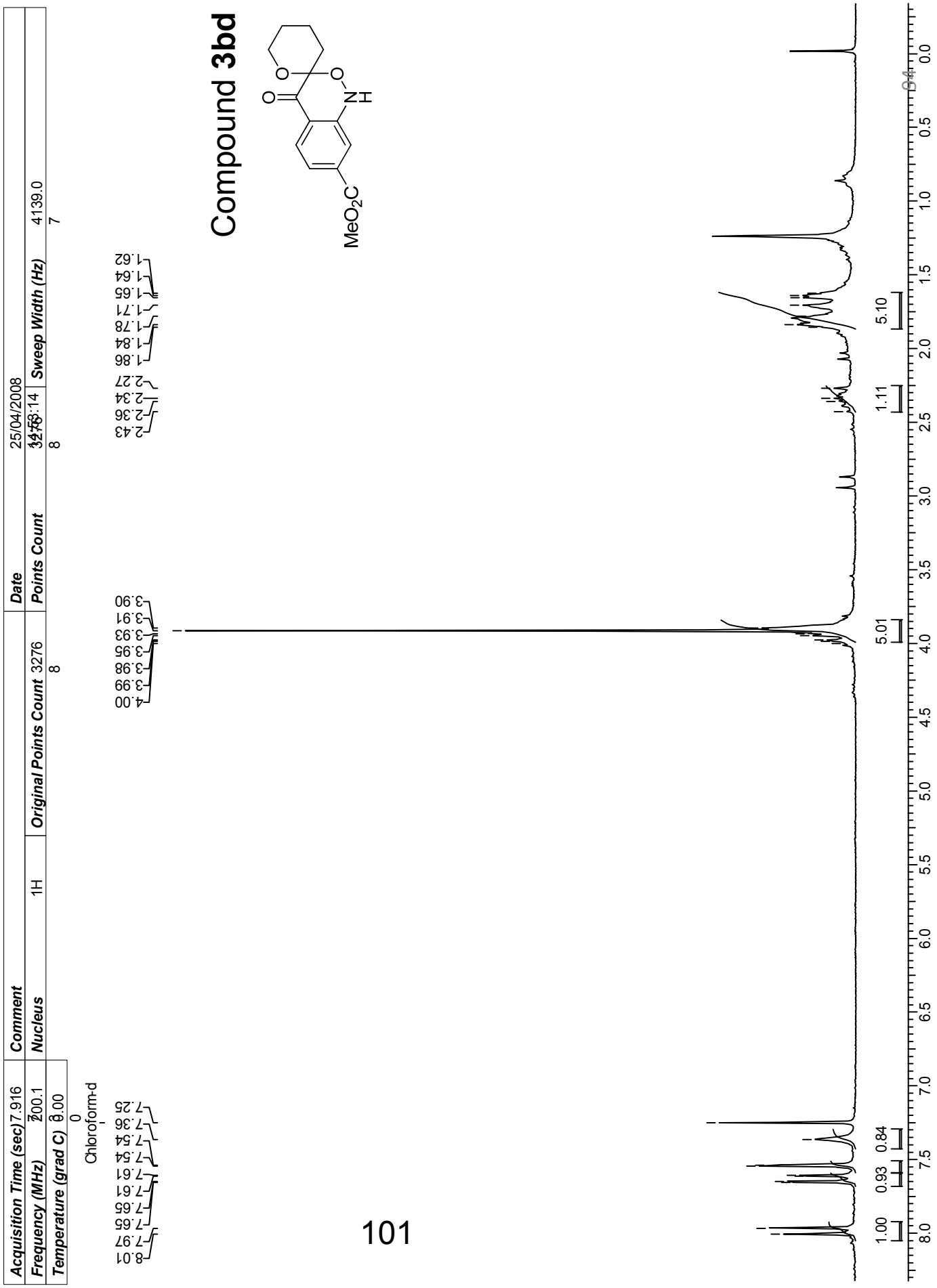
Compound **3bc**

Acquisition Time (sec)	2.7329	***Comment***	**8bf**			***Date***	25/03/2009 03:19:32
Frequency (MHz)	50.32	***Nucleus***	13C	***Original Points Count***	32768	***Points Count***	32768
Sweep Width (Hz)	11990.41						
Temperature (grad C)	0.000						

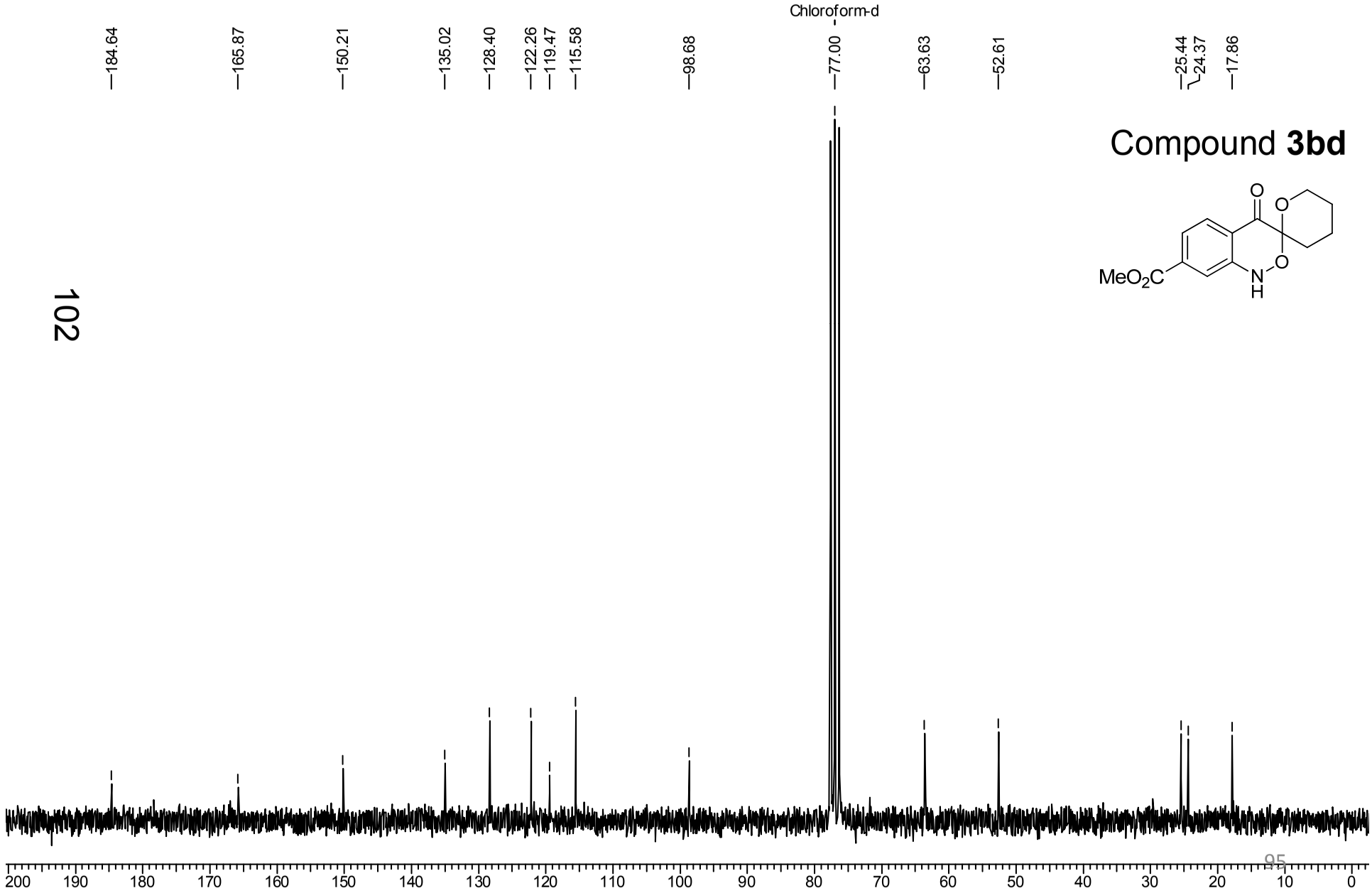


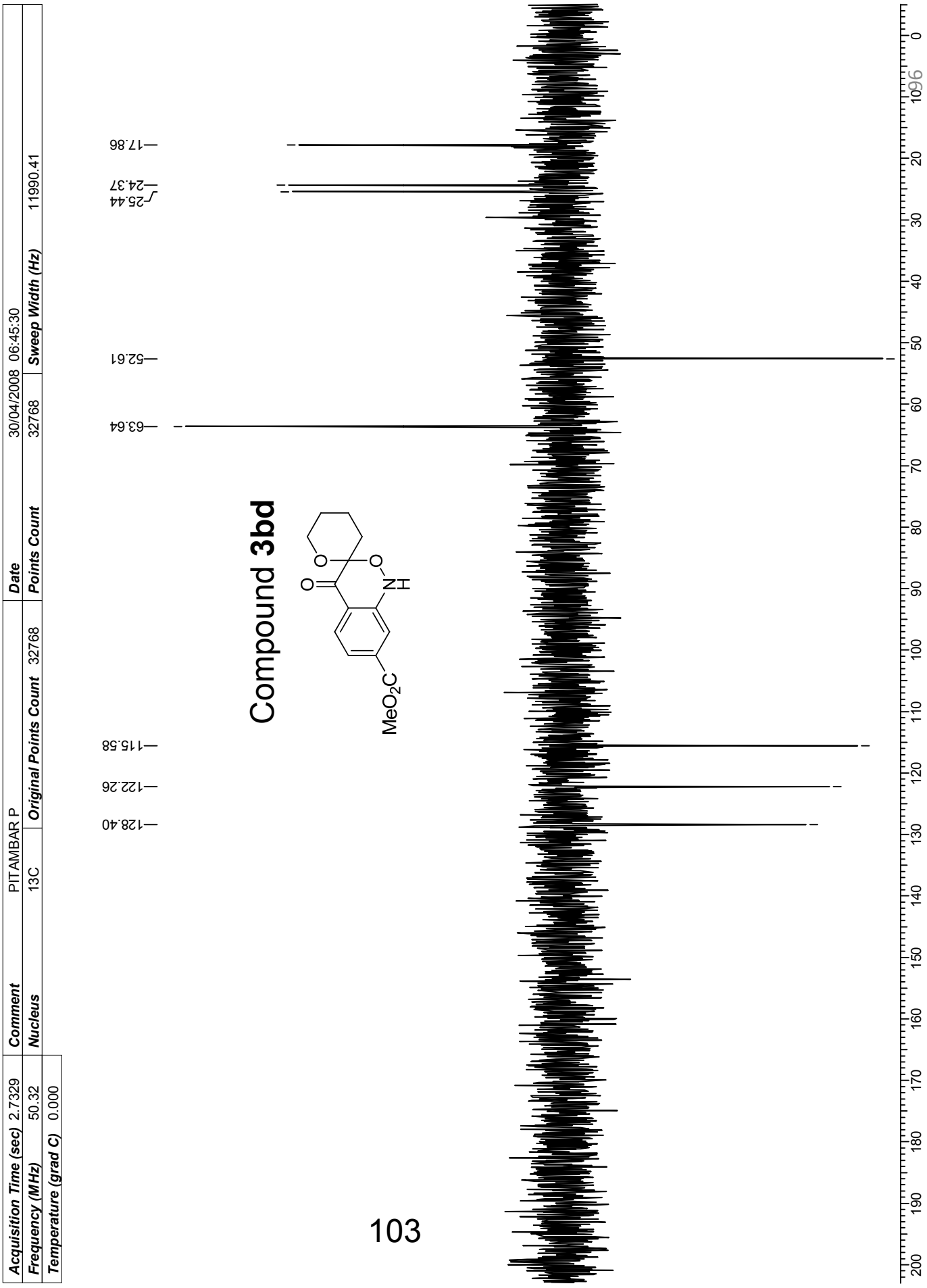


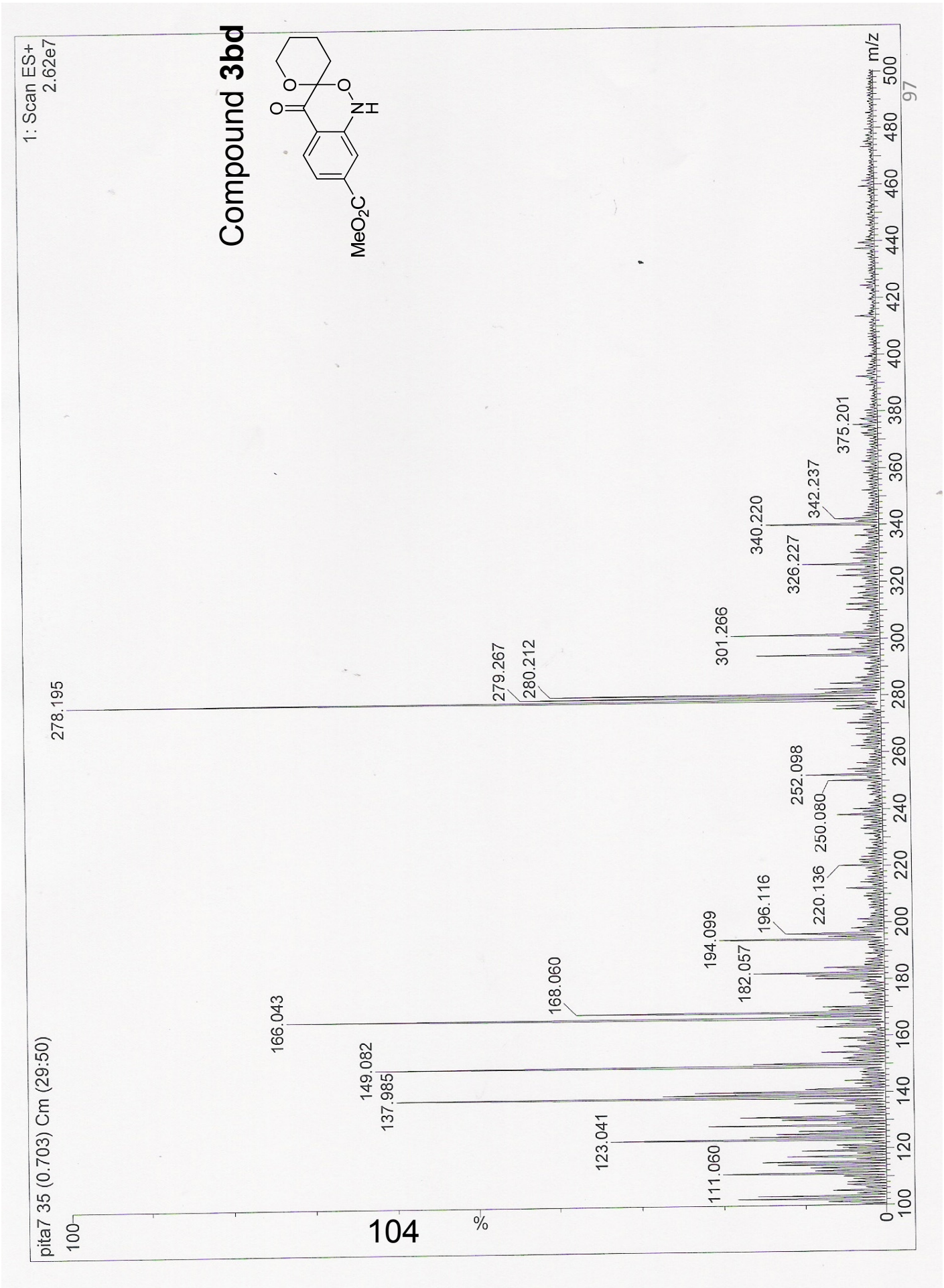


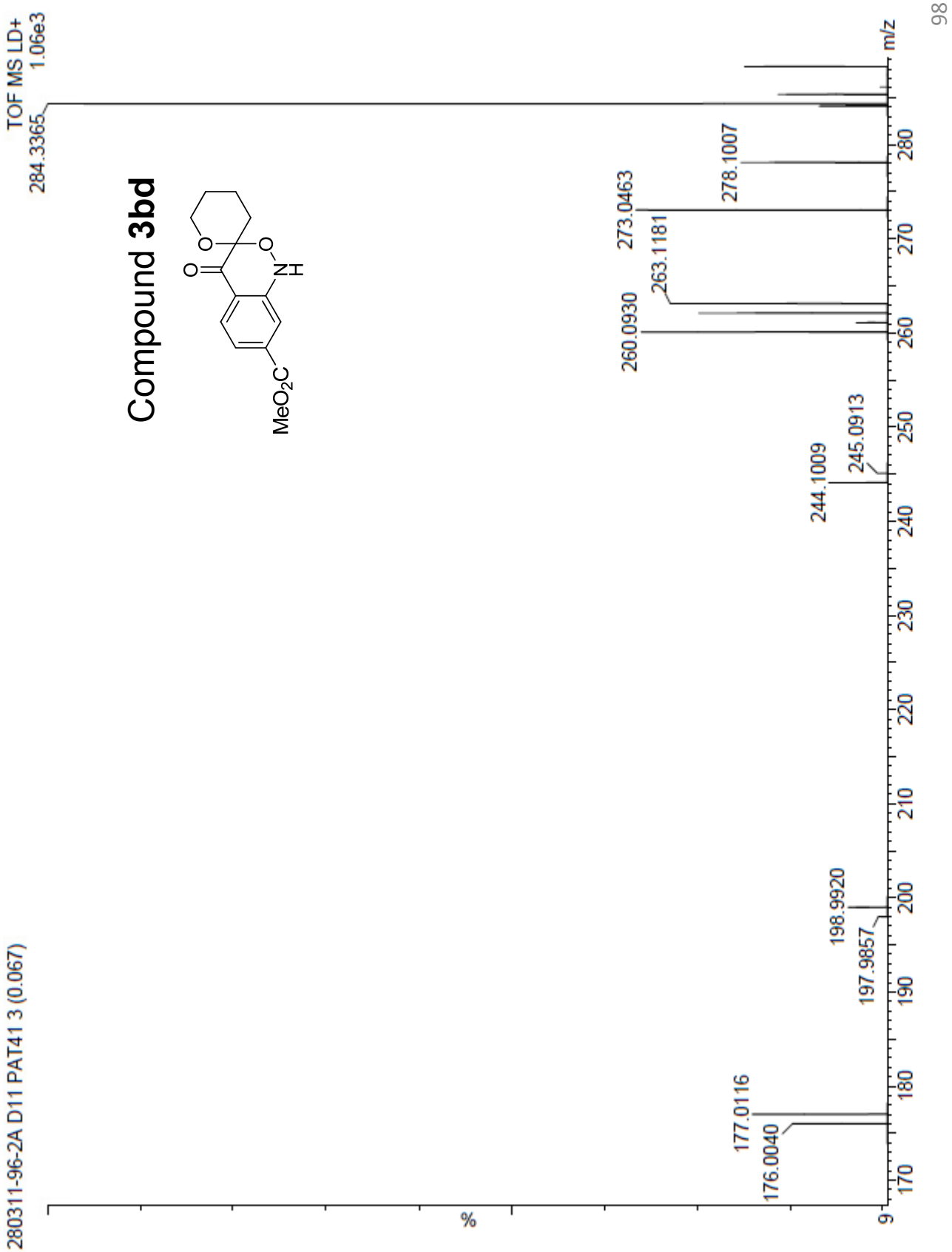


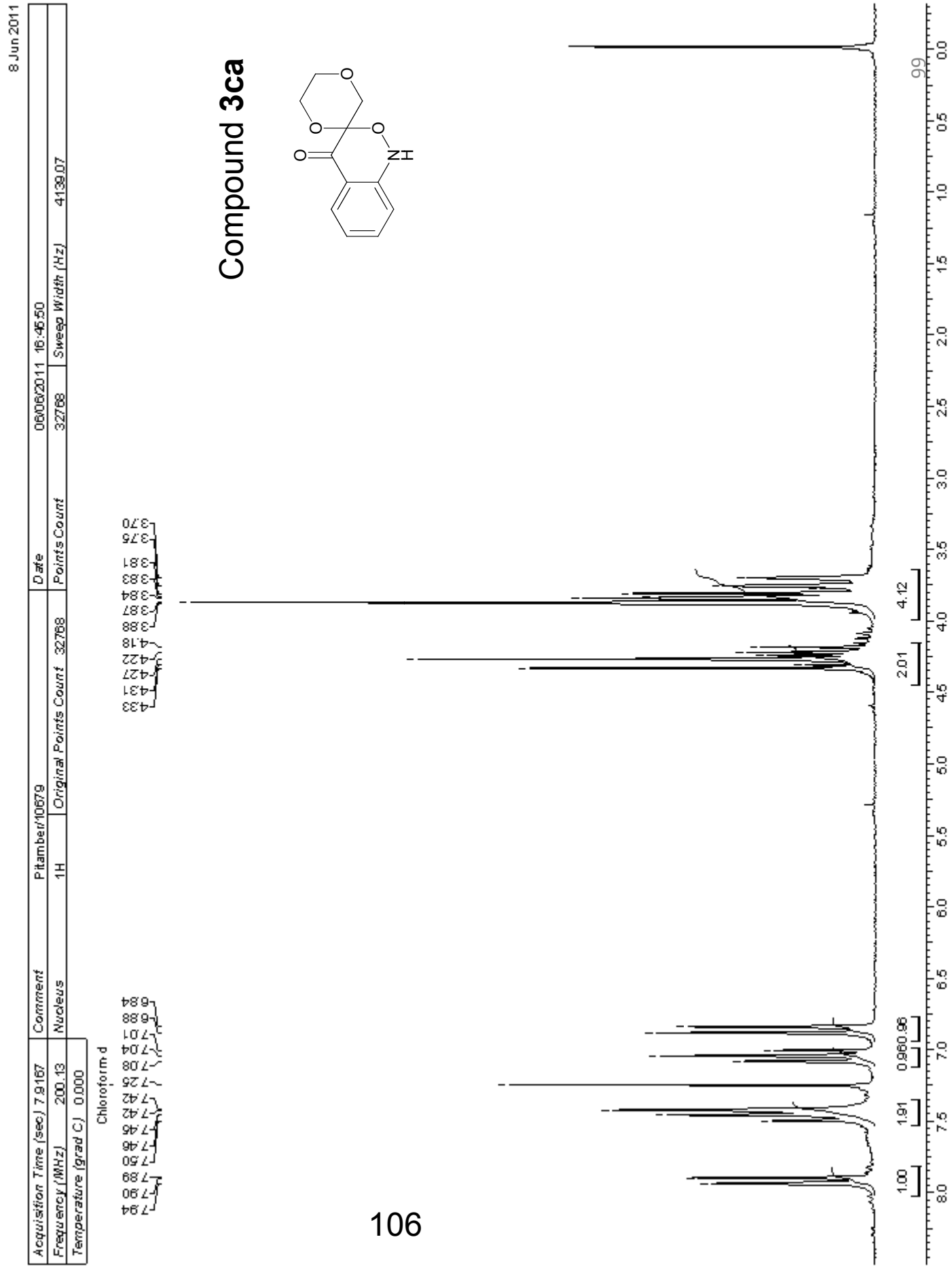
Acquisition Time (sec)	2.732	***Comment***			***Date***	30/04/2008	
Frequency (MHz)	90.3	***Nucleus***	13C	***Original Points Count***	32768	***Points Count***	32768
Temperature (grad C)	0.00					***Sweep Width (Hz)***	11990.4

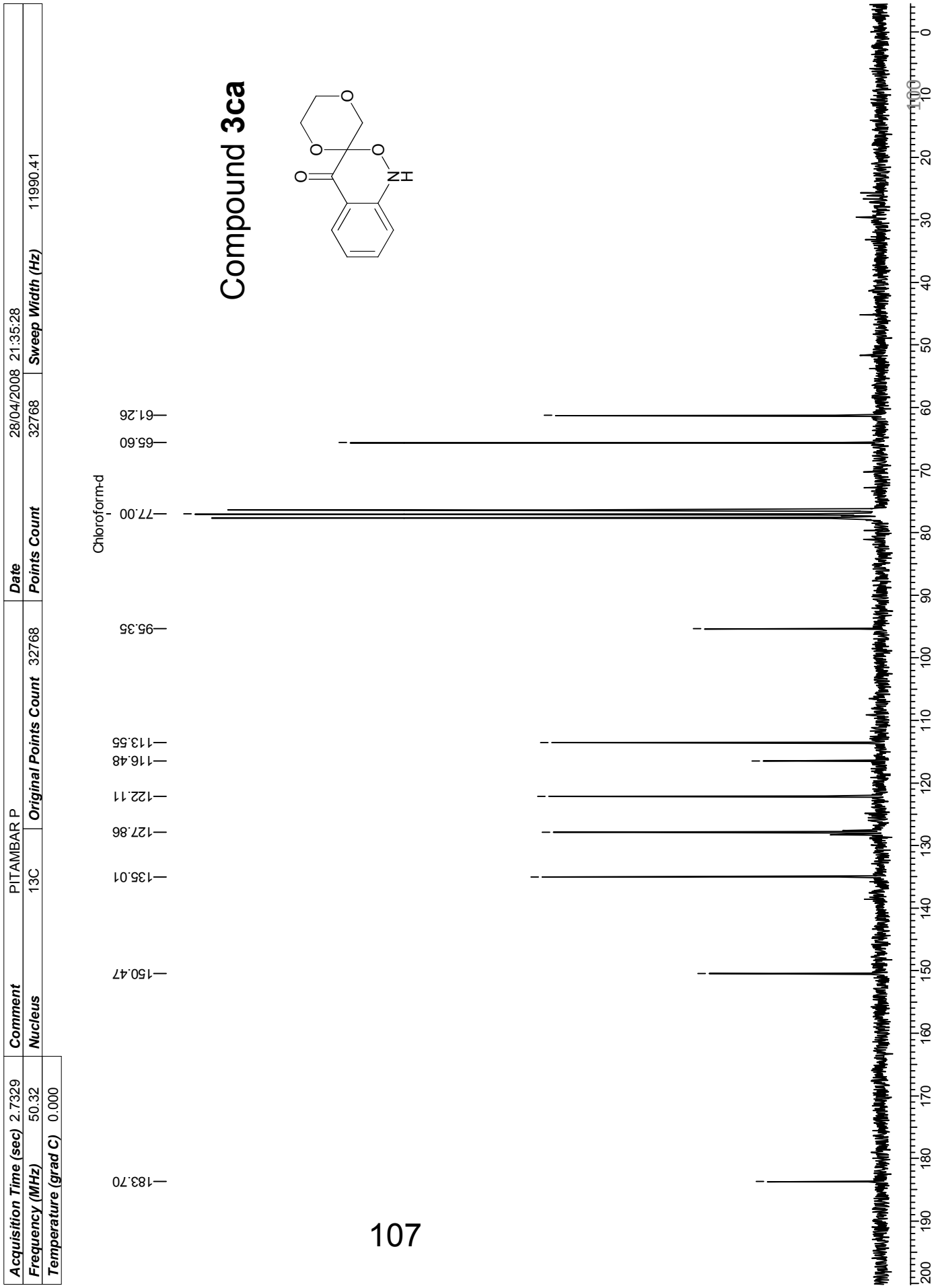


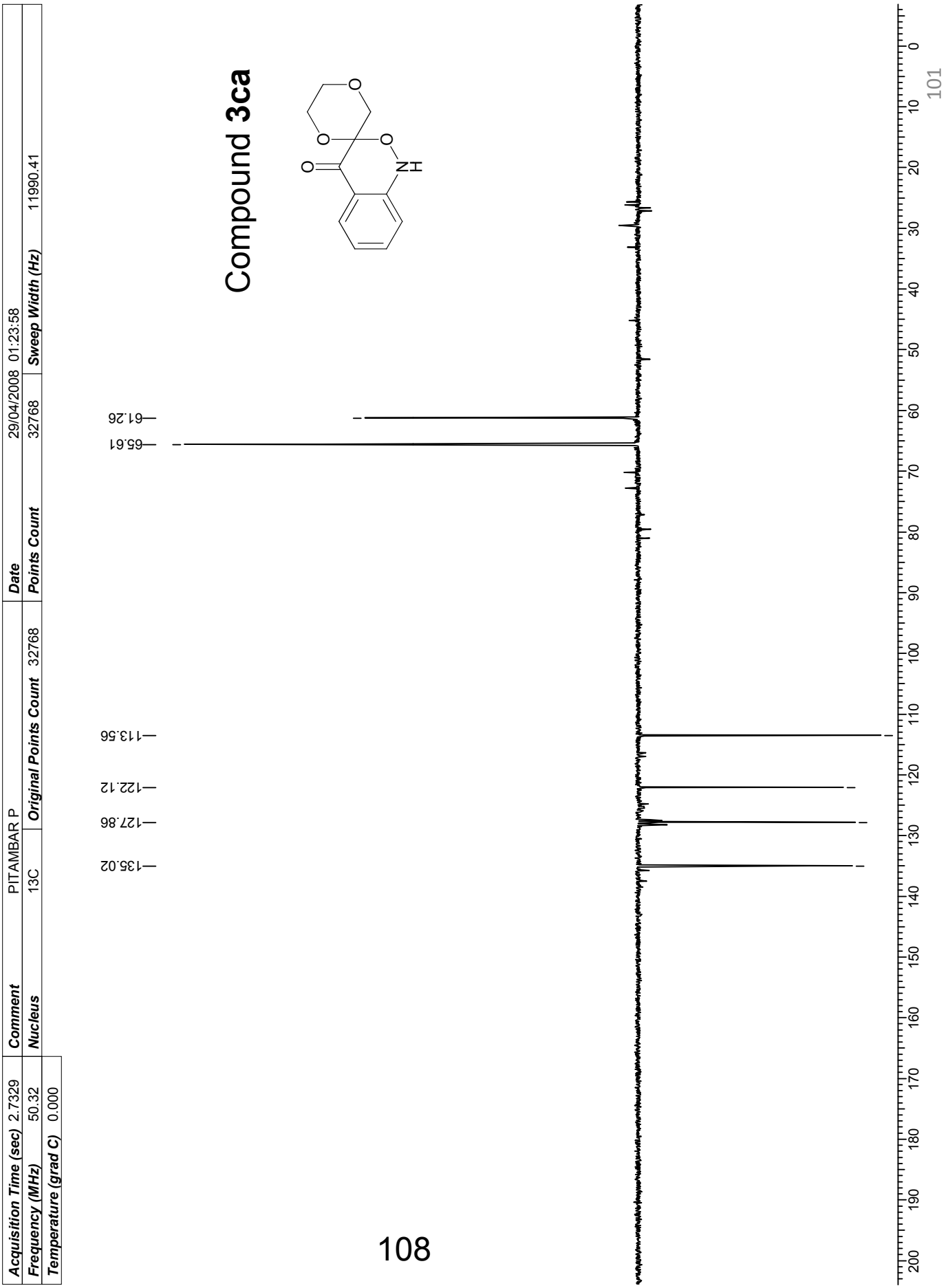




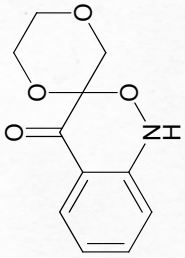

Compound 3bd
TOF MS LD+
1.06e3
284.3365
280311-96-2A D11 PAT41 3 (0.067)
%
m/z
278.1007
273.0463
263.1181
260.0930
245.0913
244.1009
198.9920
197.9857
177.0116
176.0040
MeO2C

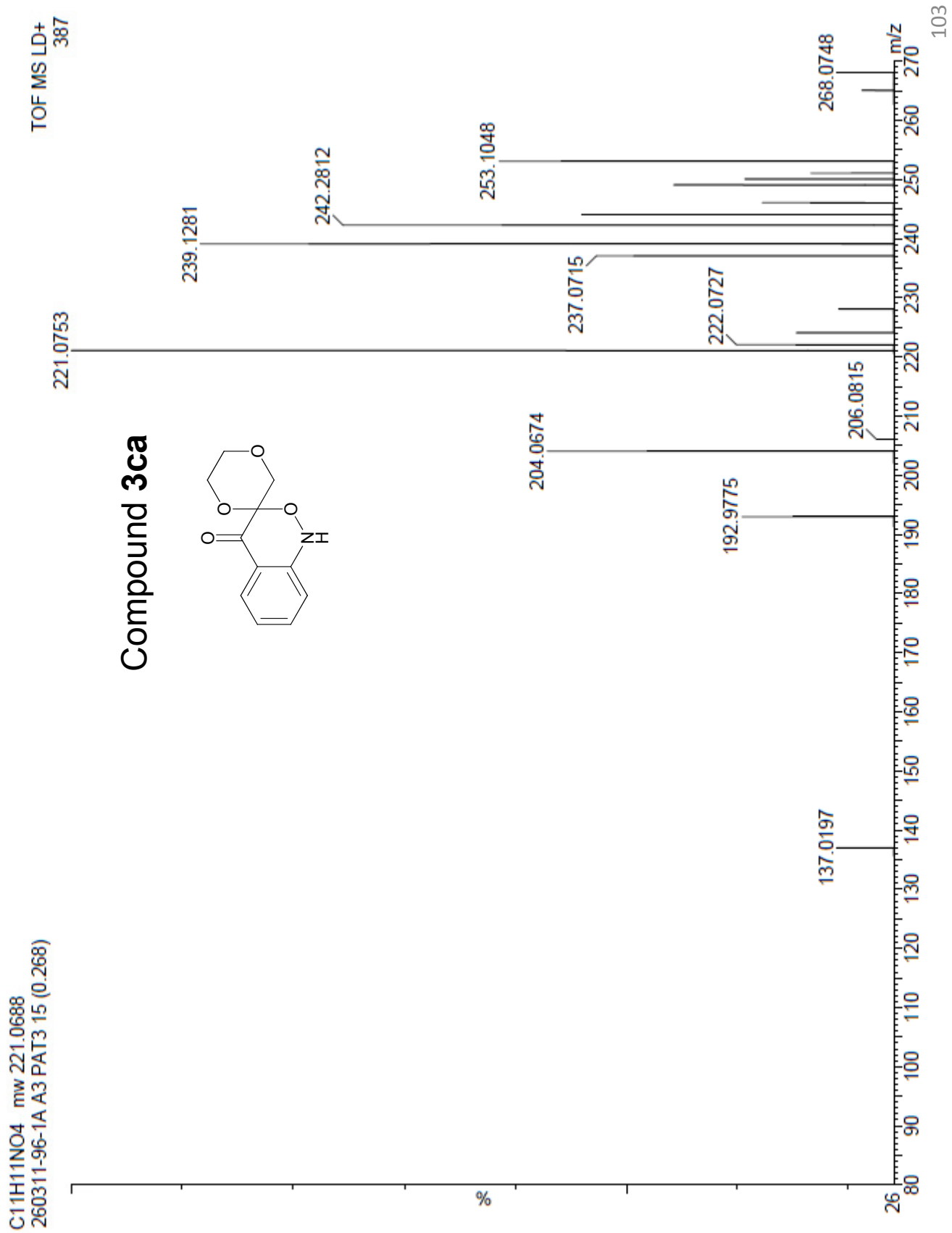


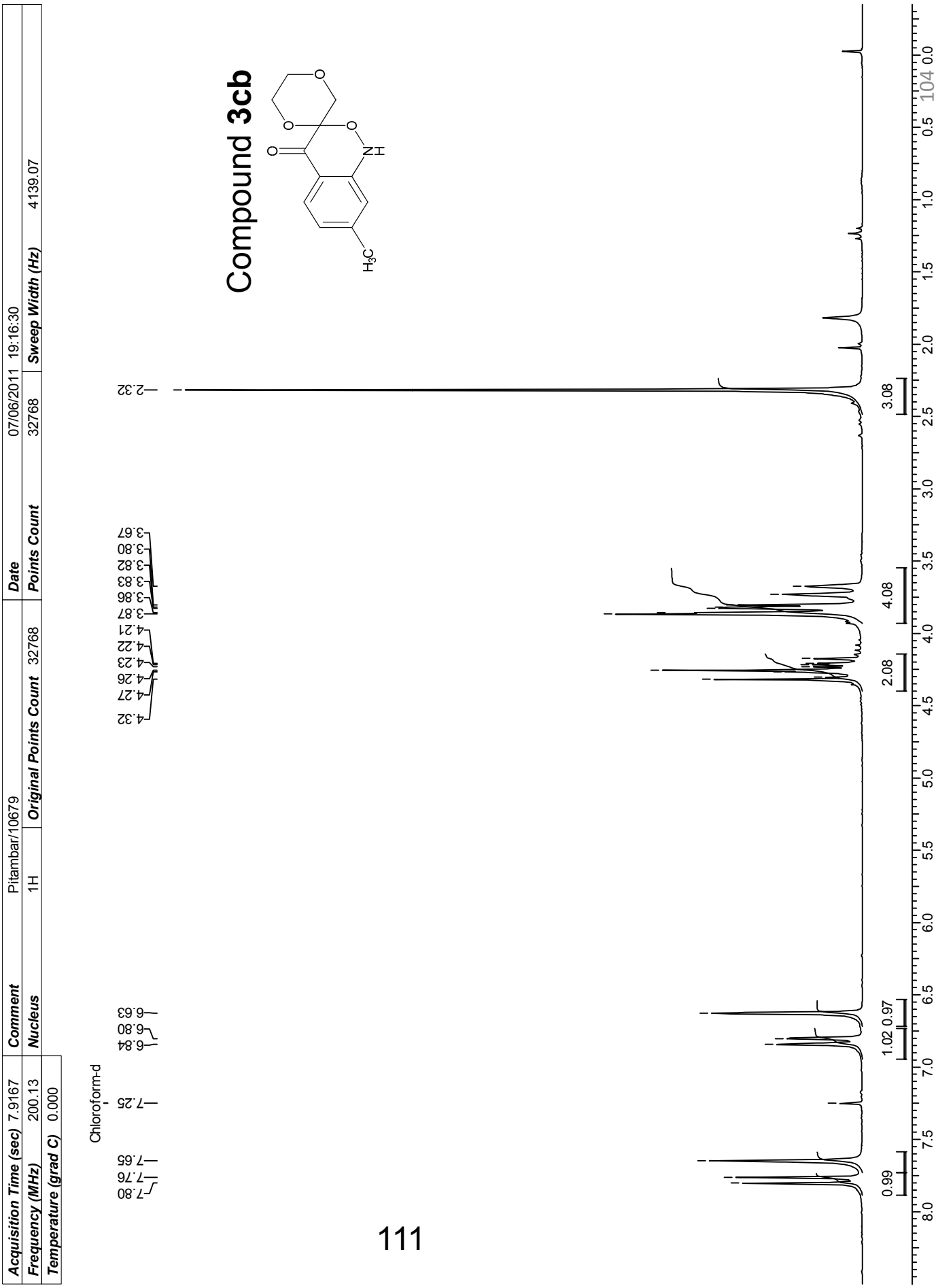


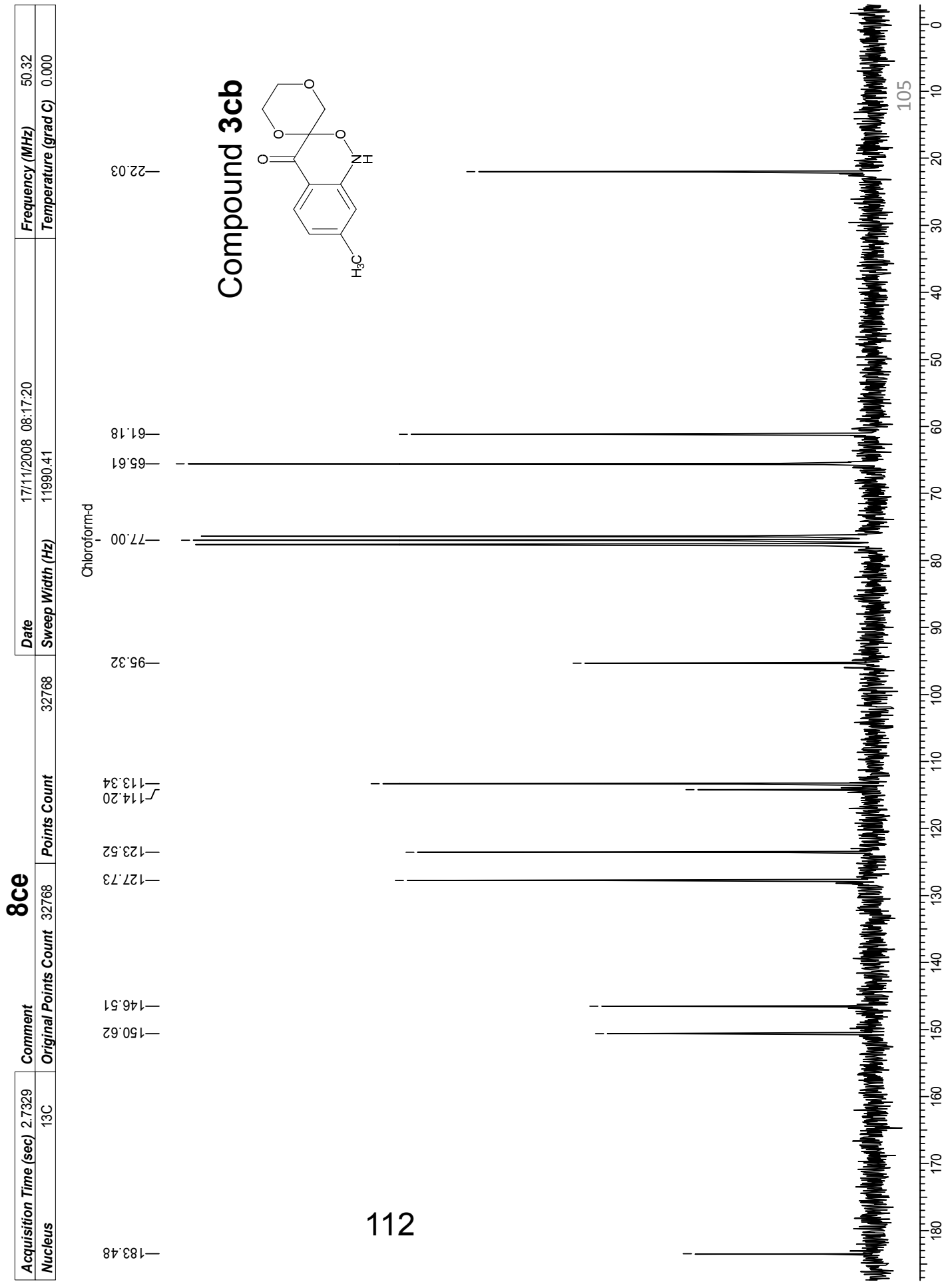


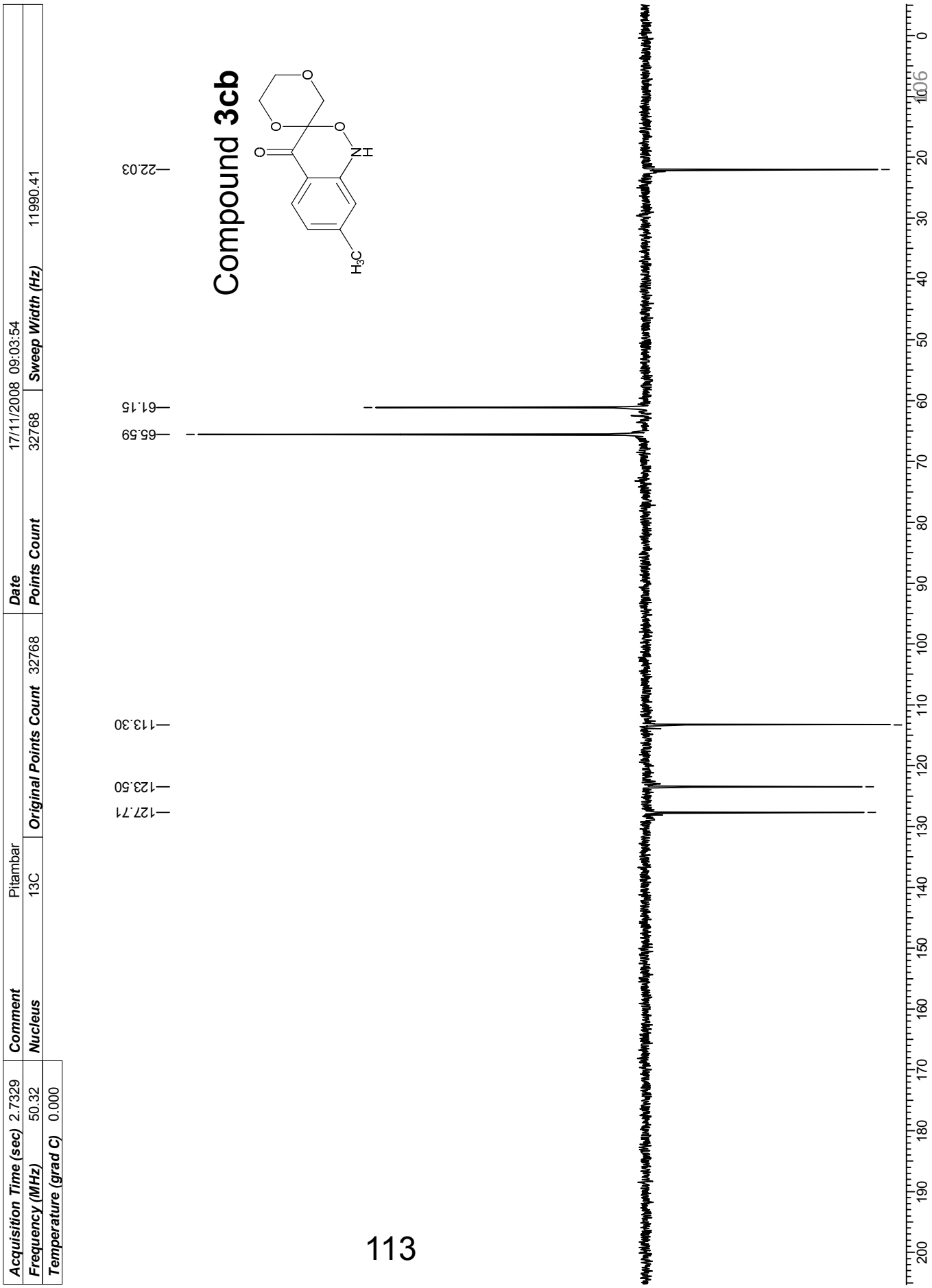
Compound **3ca**

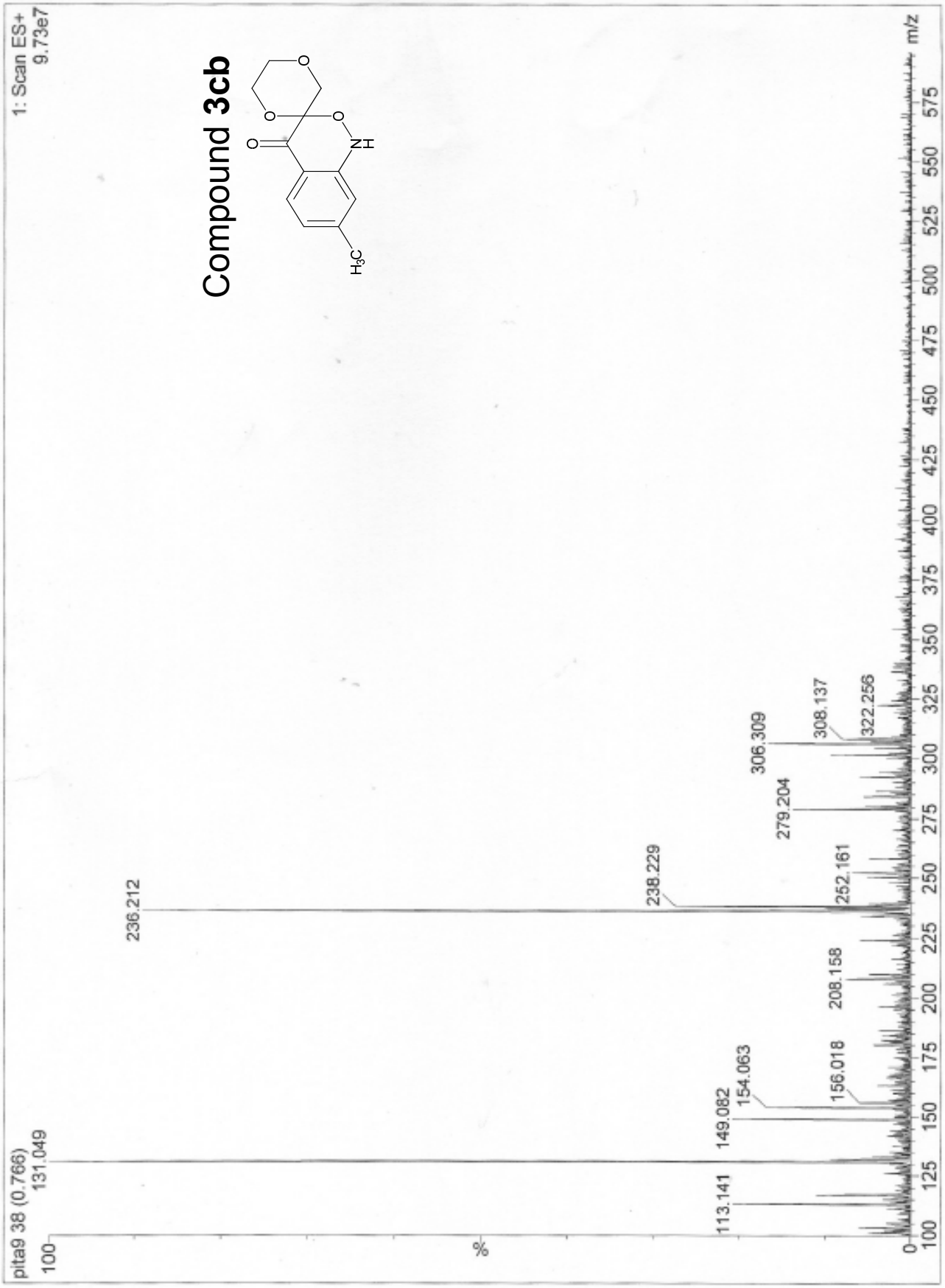


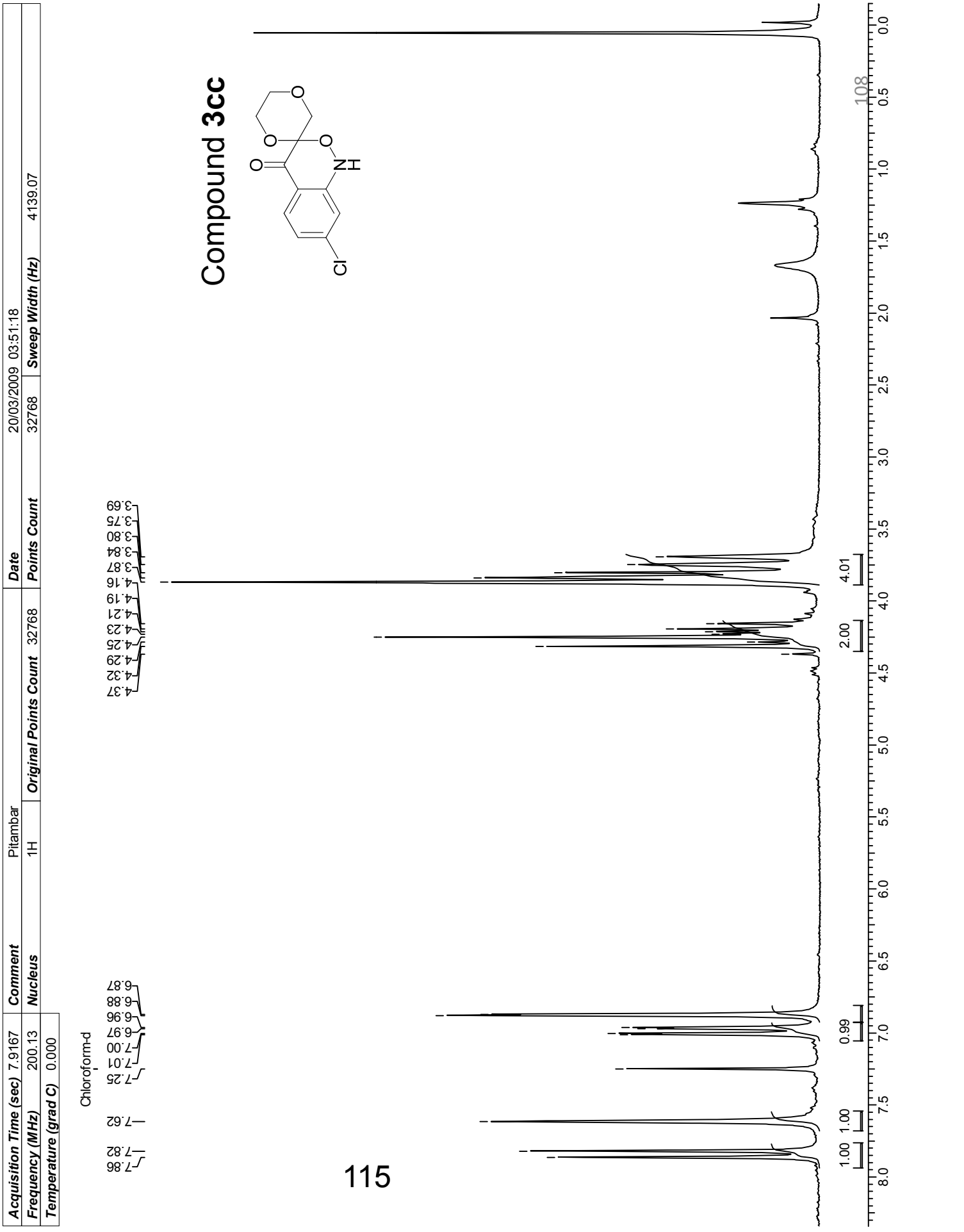


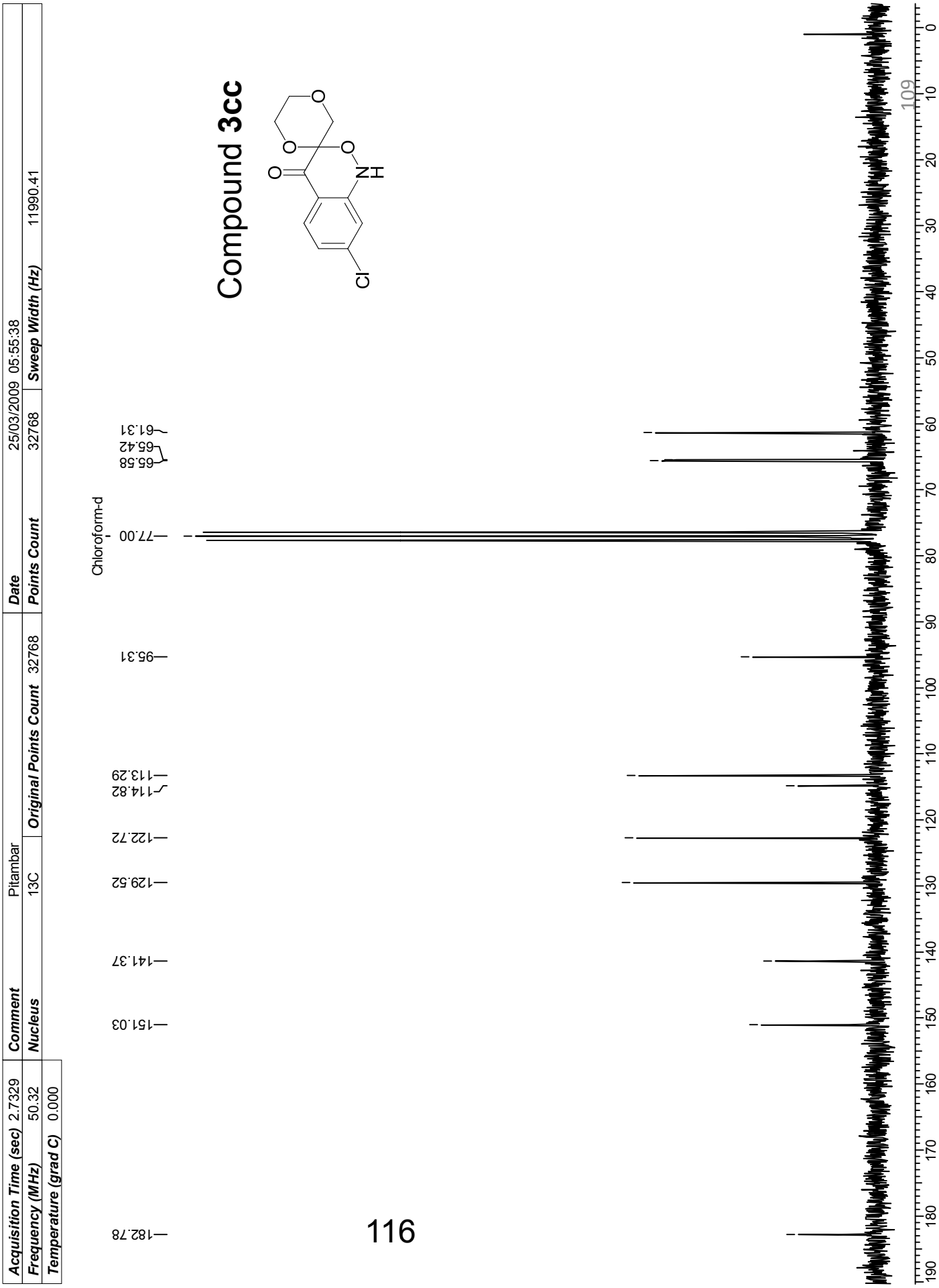


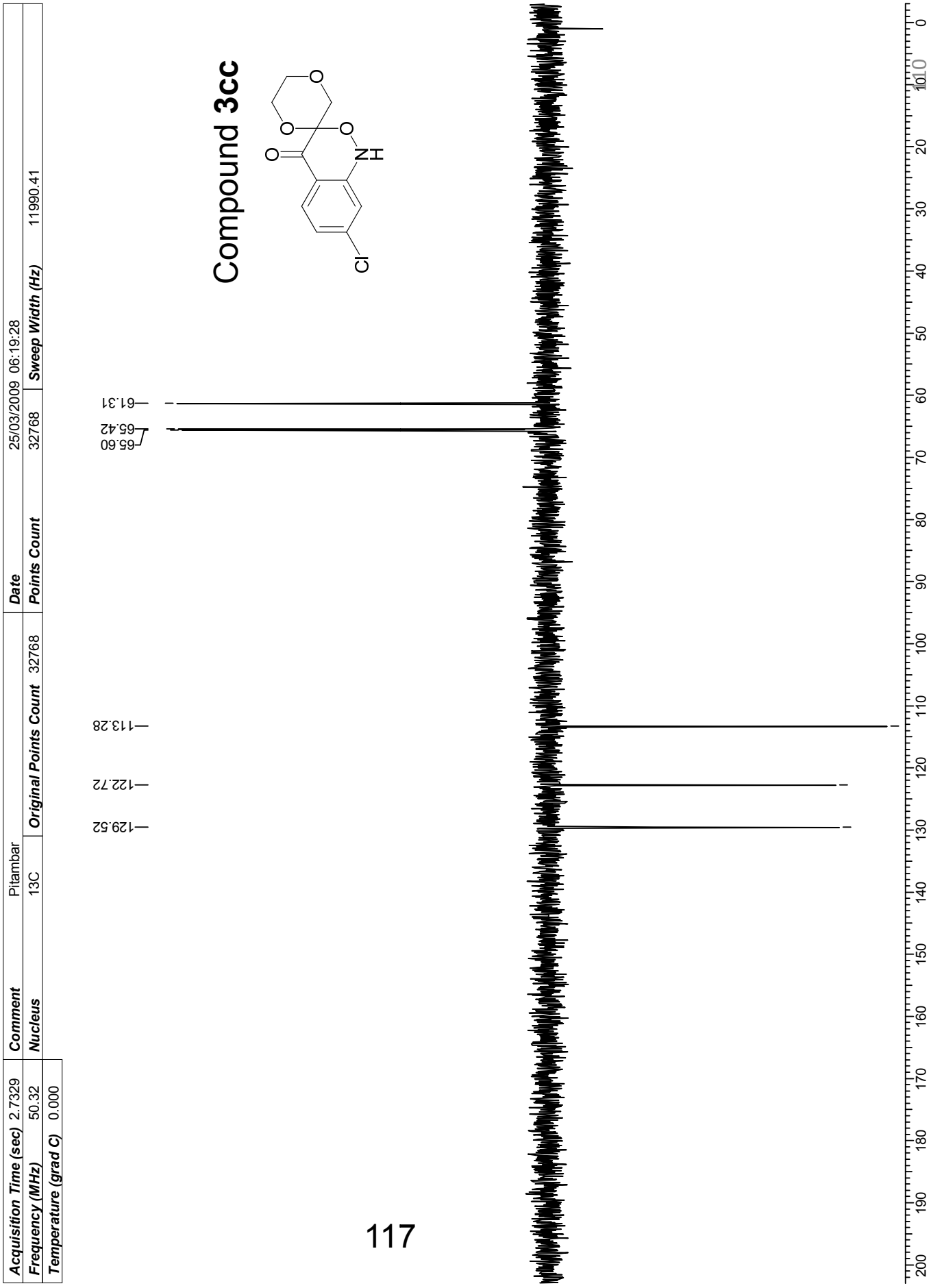


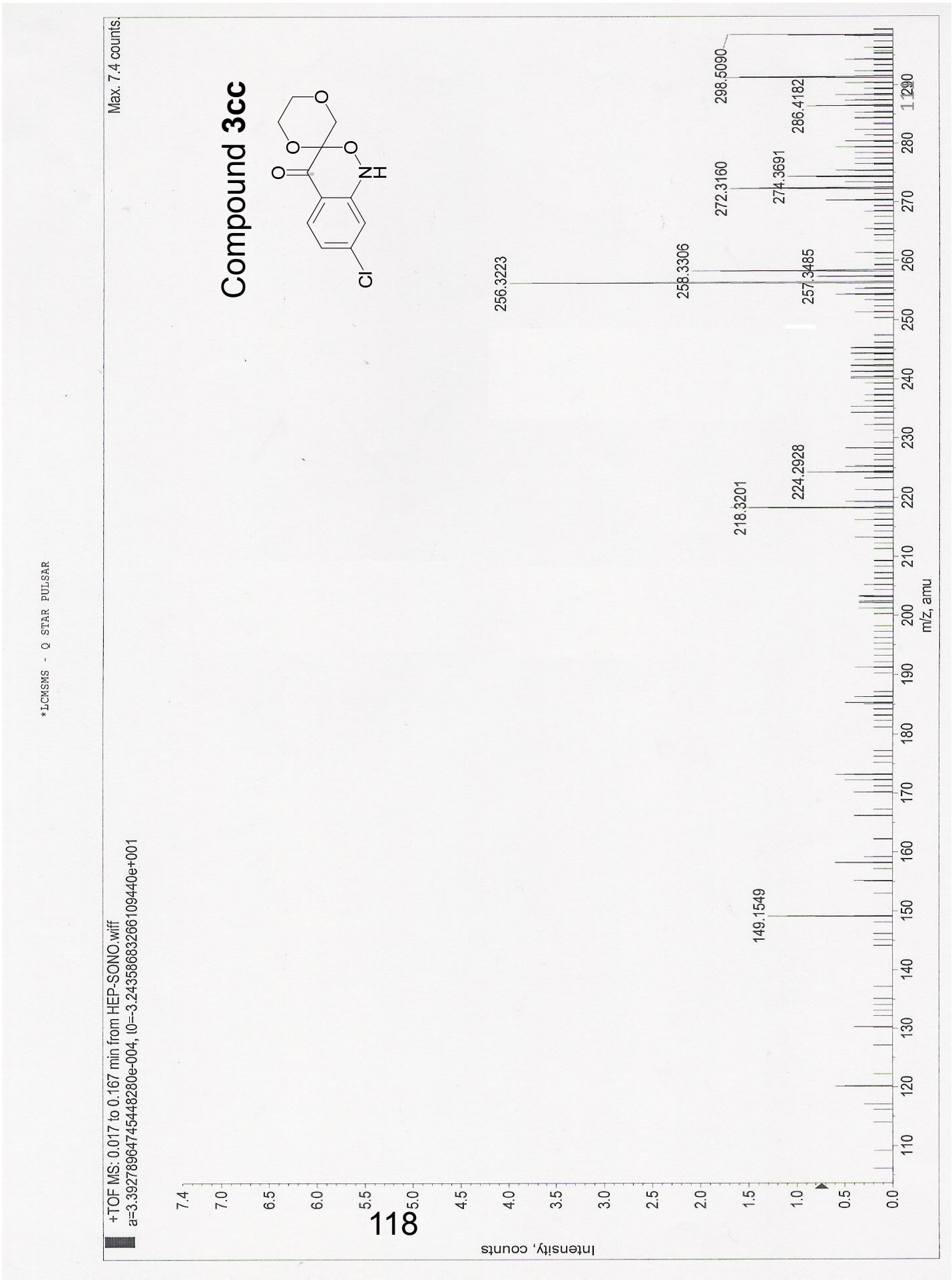


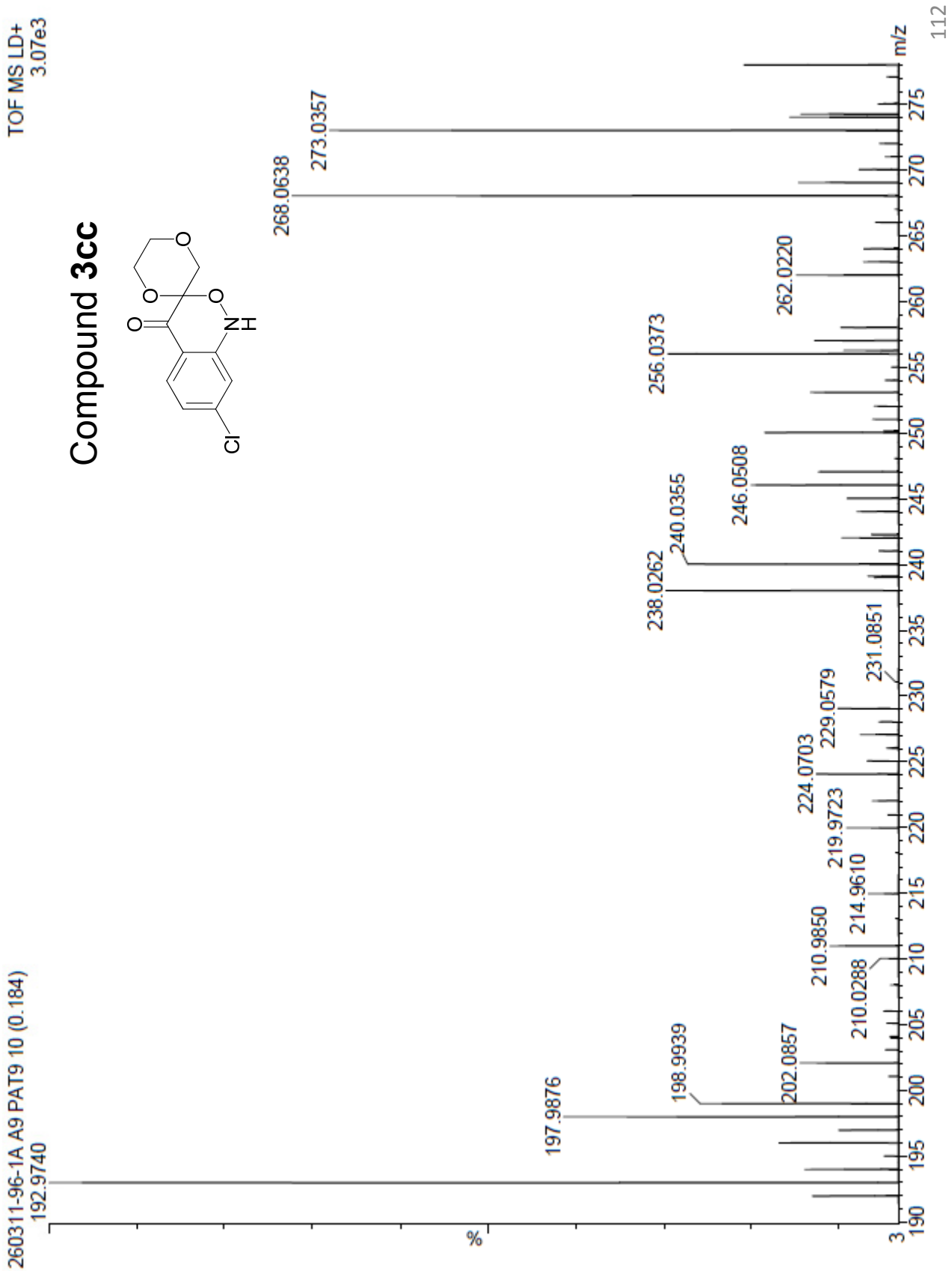

Compound 3cb
1: Scan ES+
9.73e7
pita9 38 (0.766)
100
%
0
m/z
131.049
236.212
238.229
279.204
306.309
308.137
322.256
252.161
208.158
156.018
154.063
149.082
113.141
100
125
150
175
200
225
250
275
300
325
350
375
400
425
450
475
500
525
550
575
O
O
O
O
NH
H3C

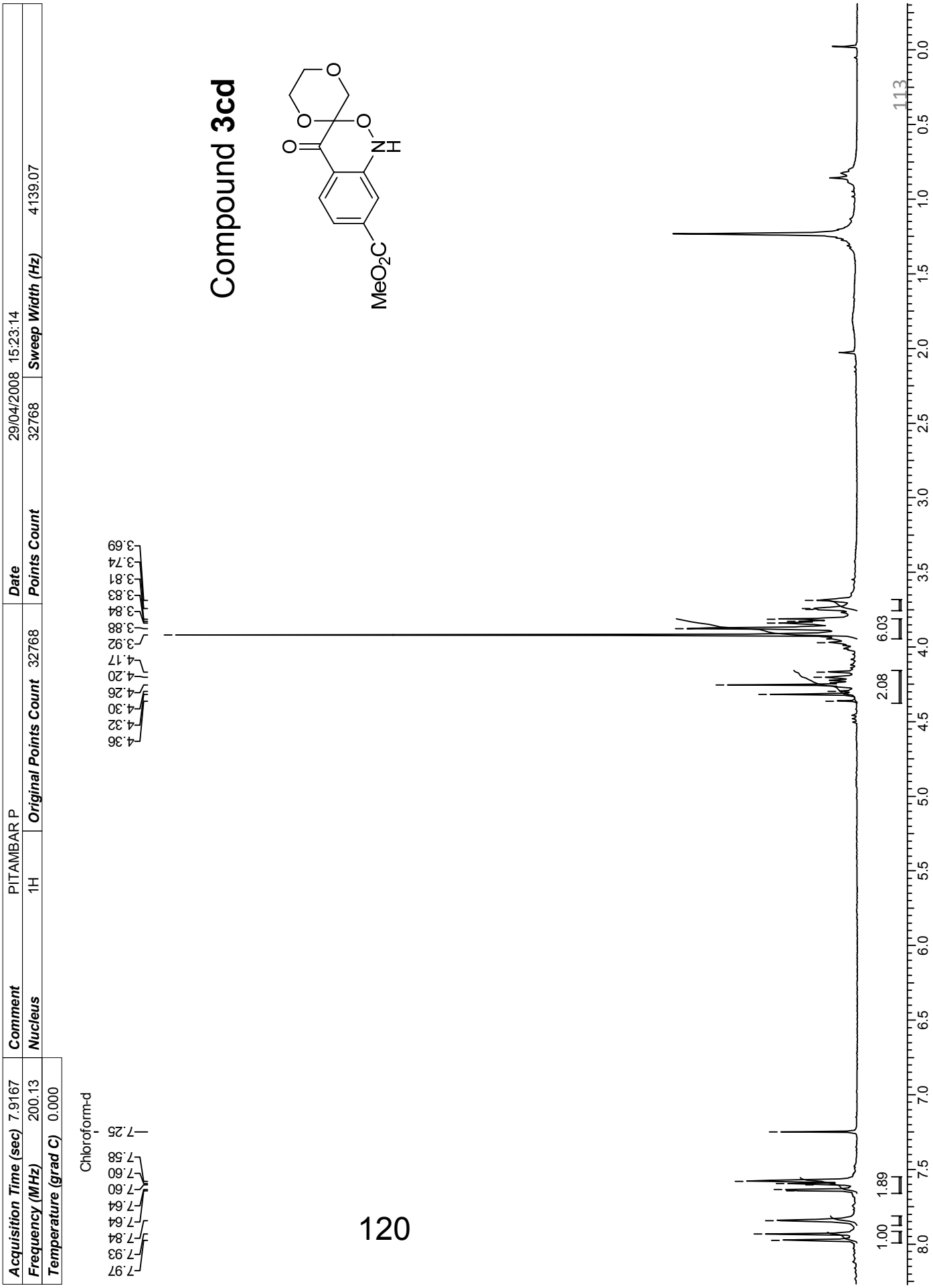


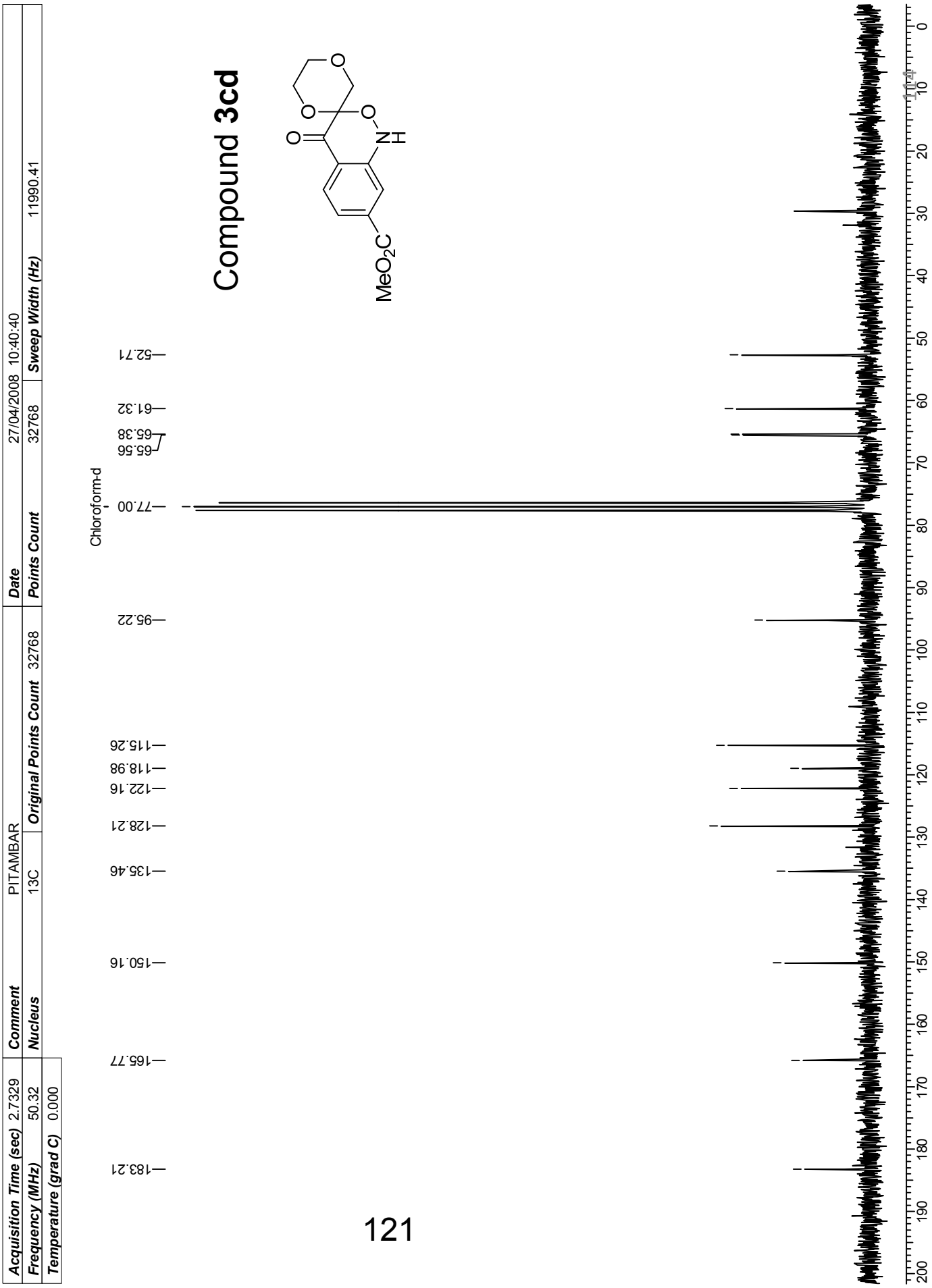


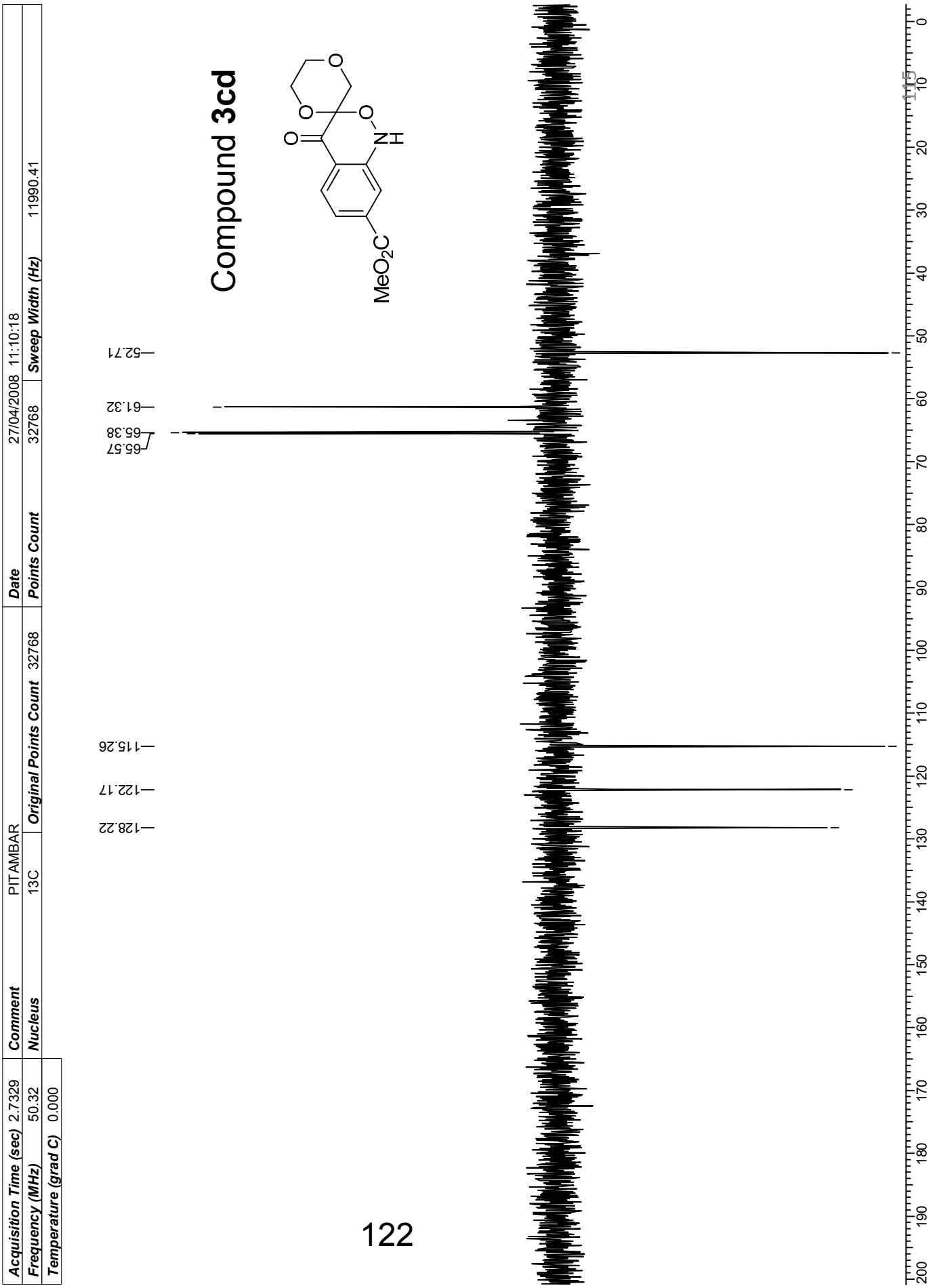


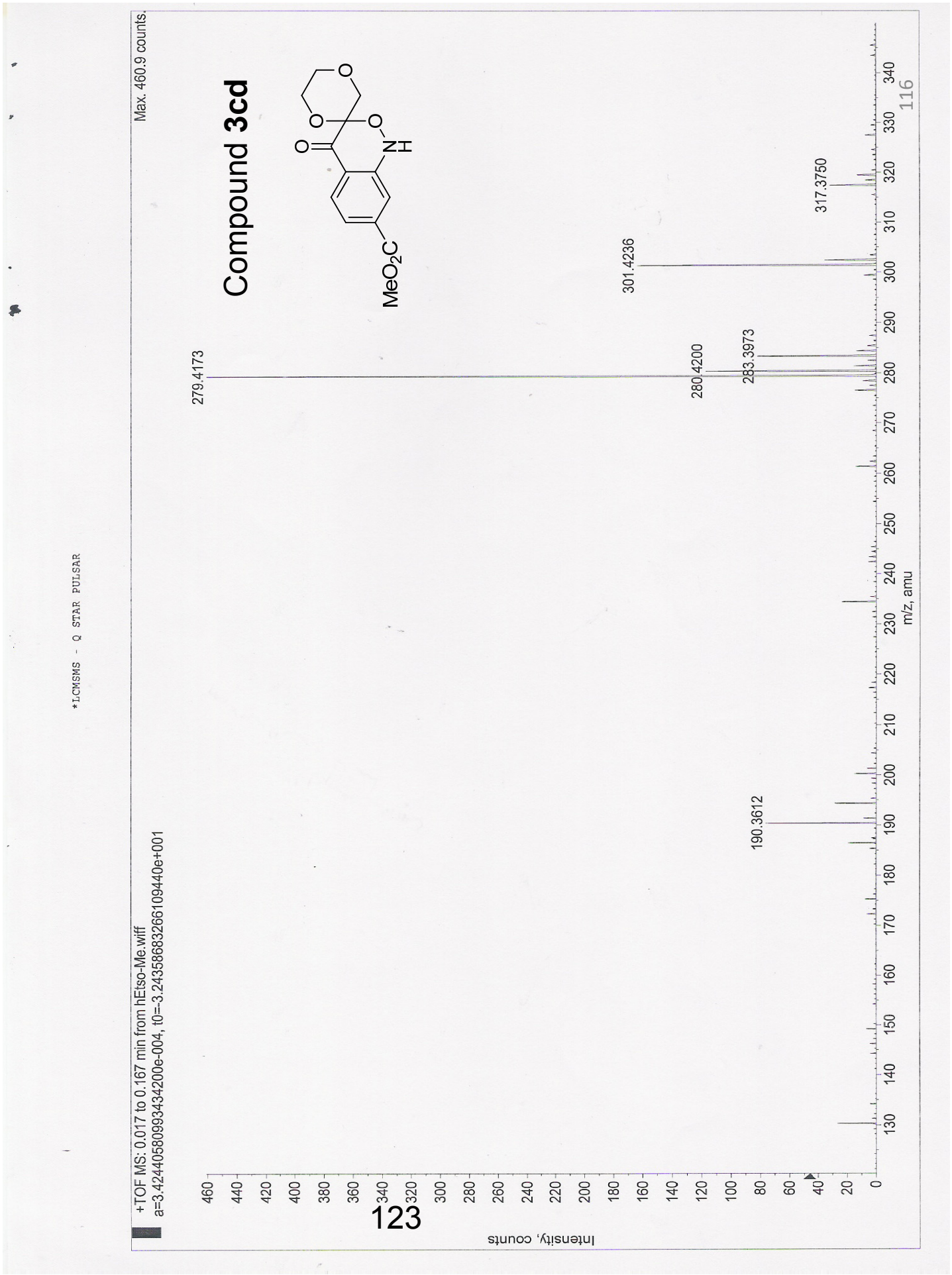


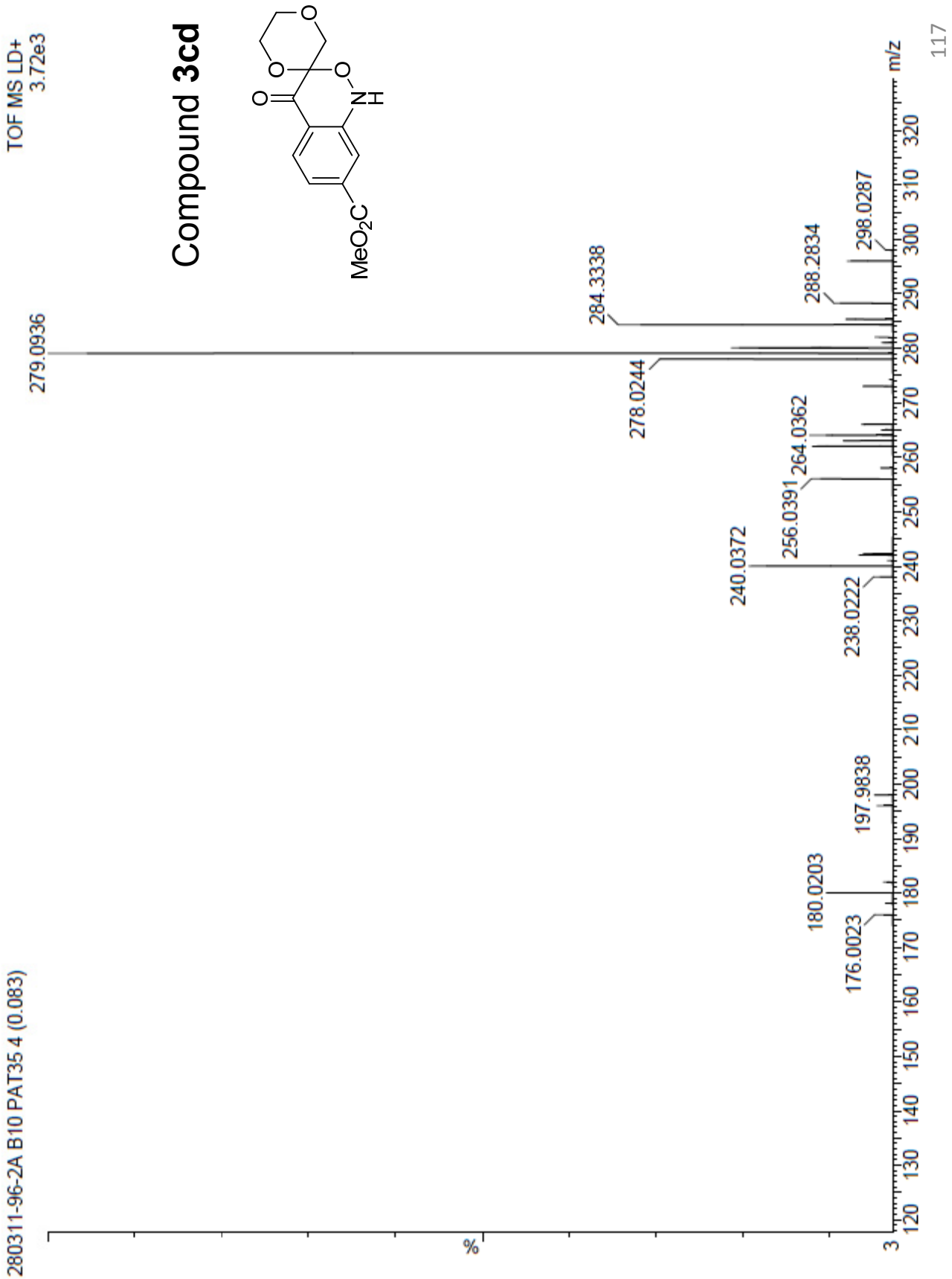


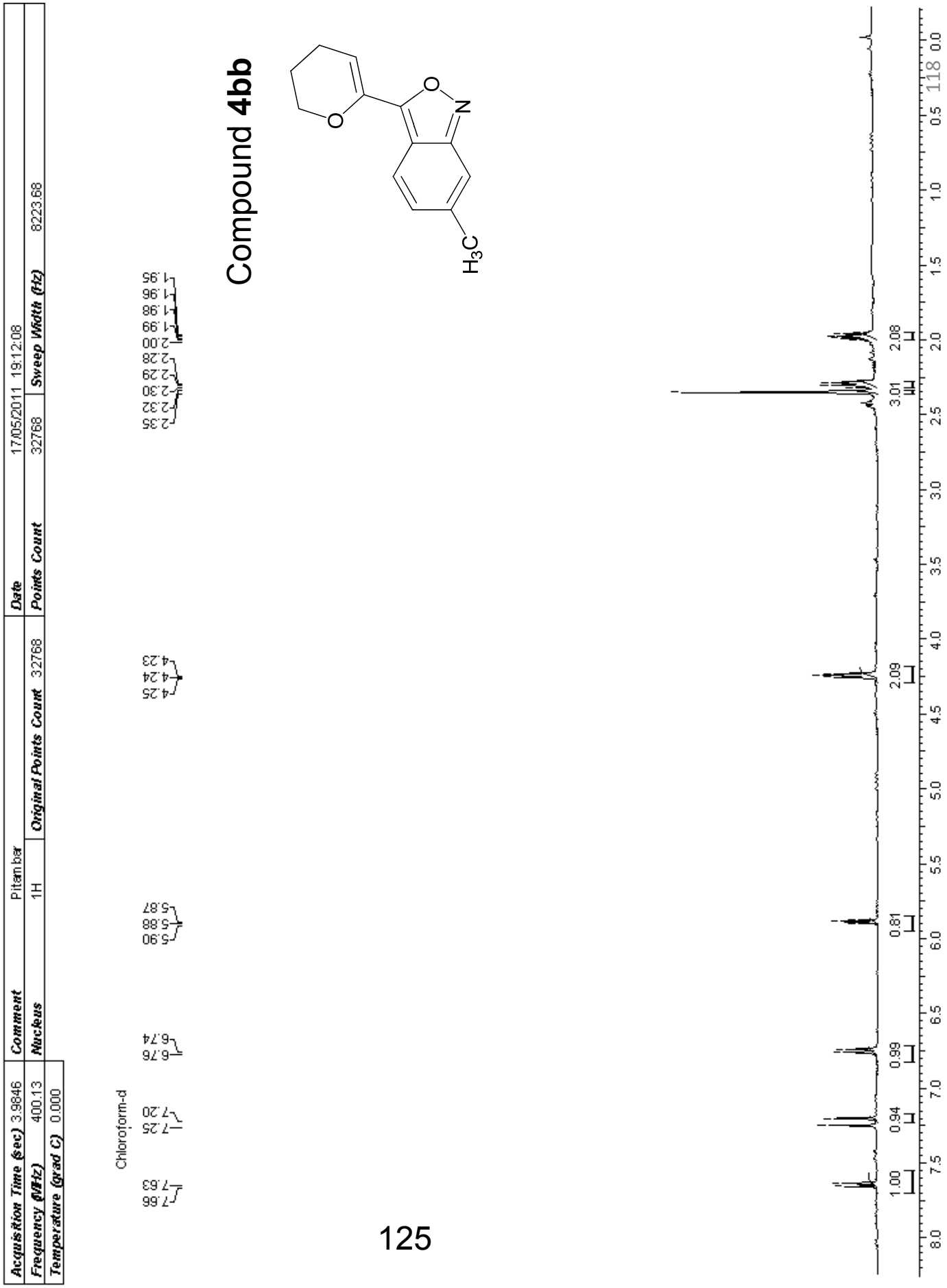


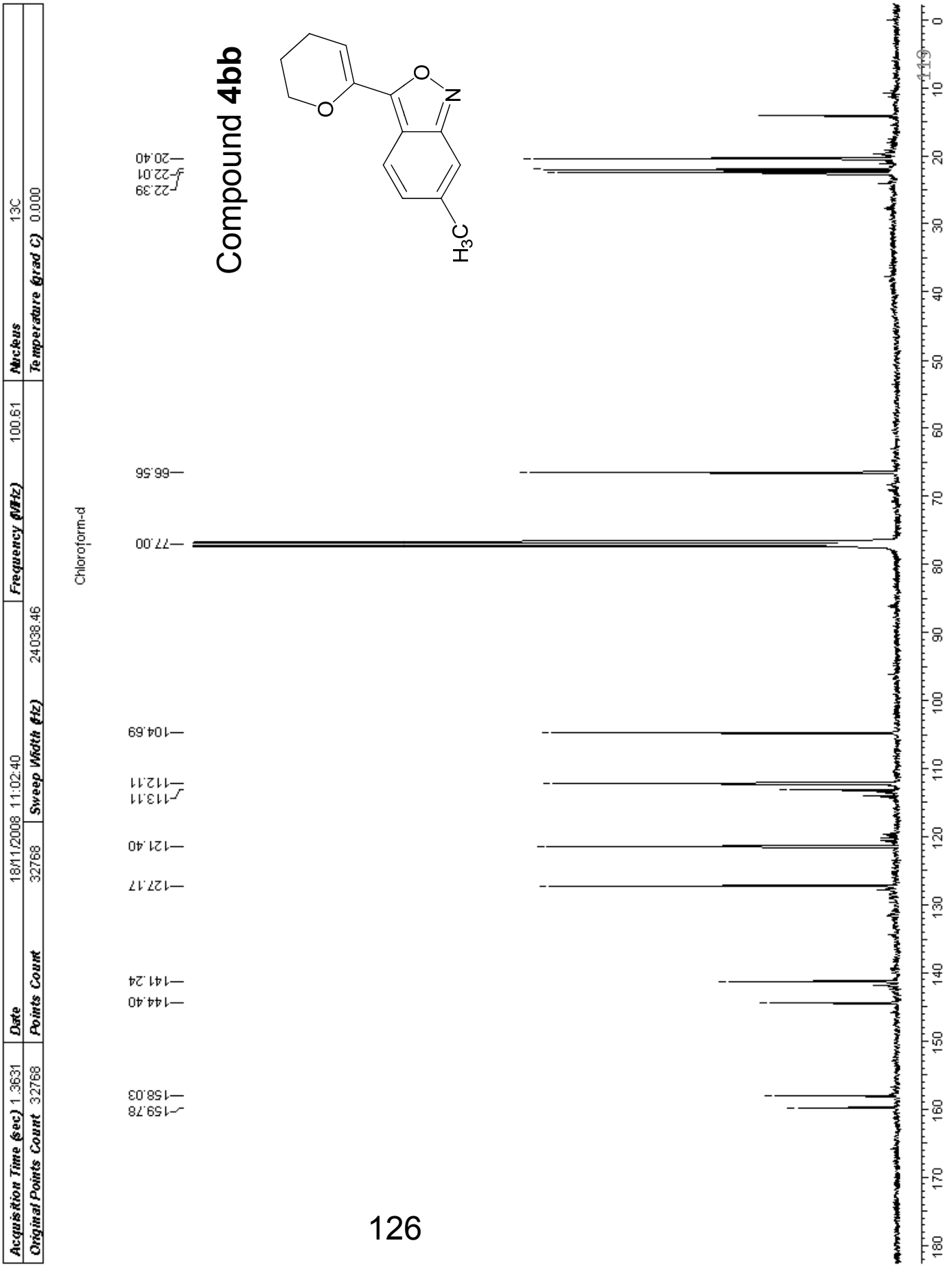


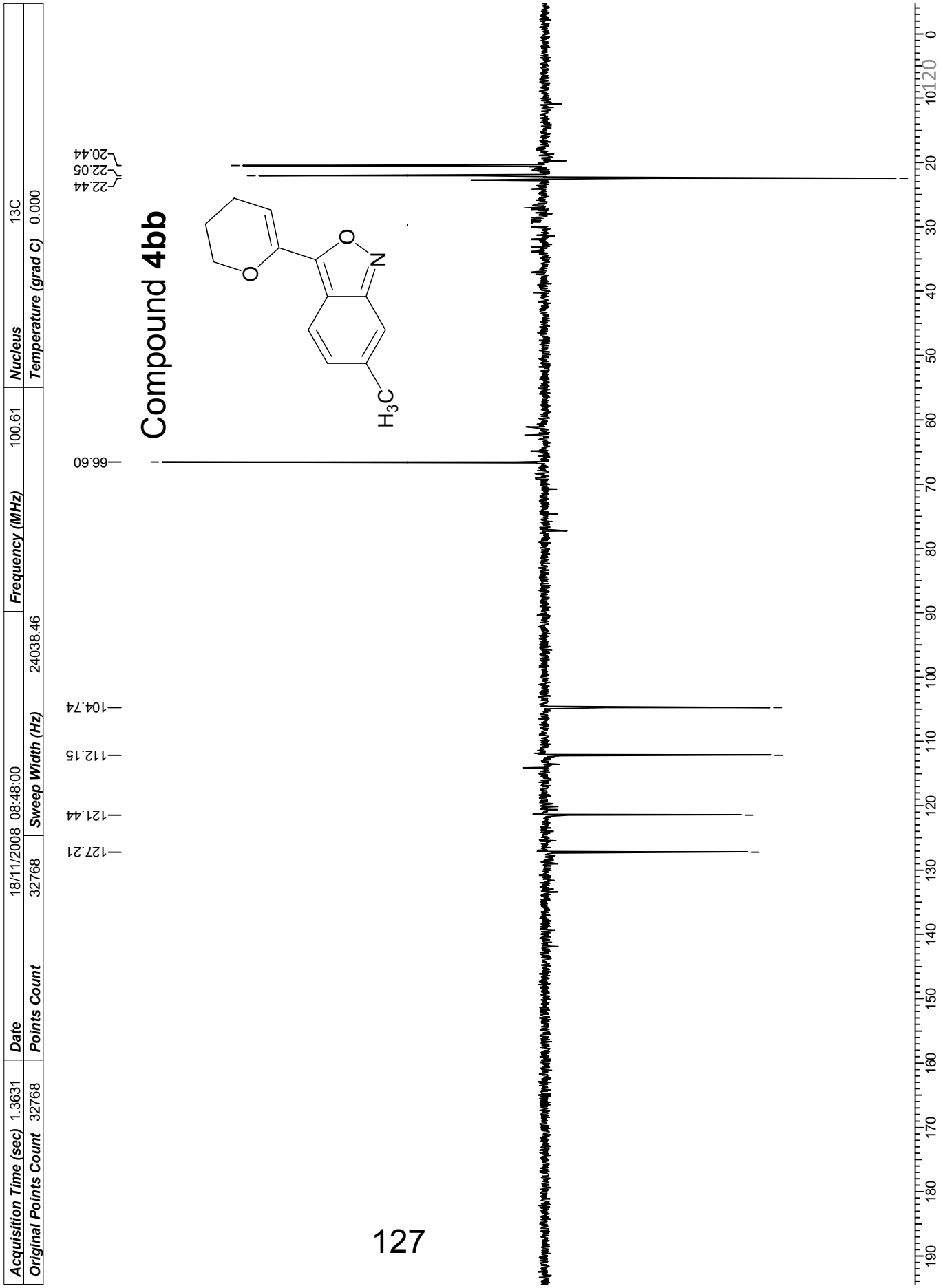


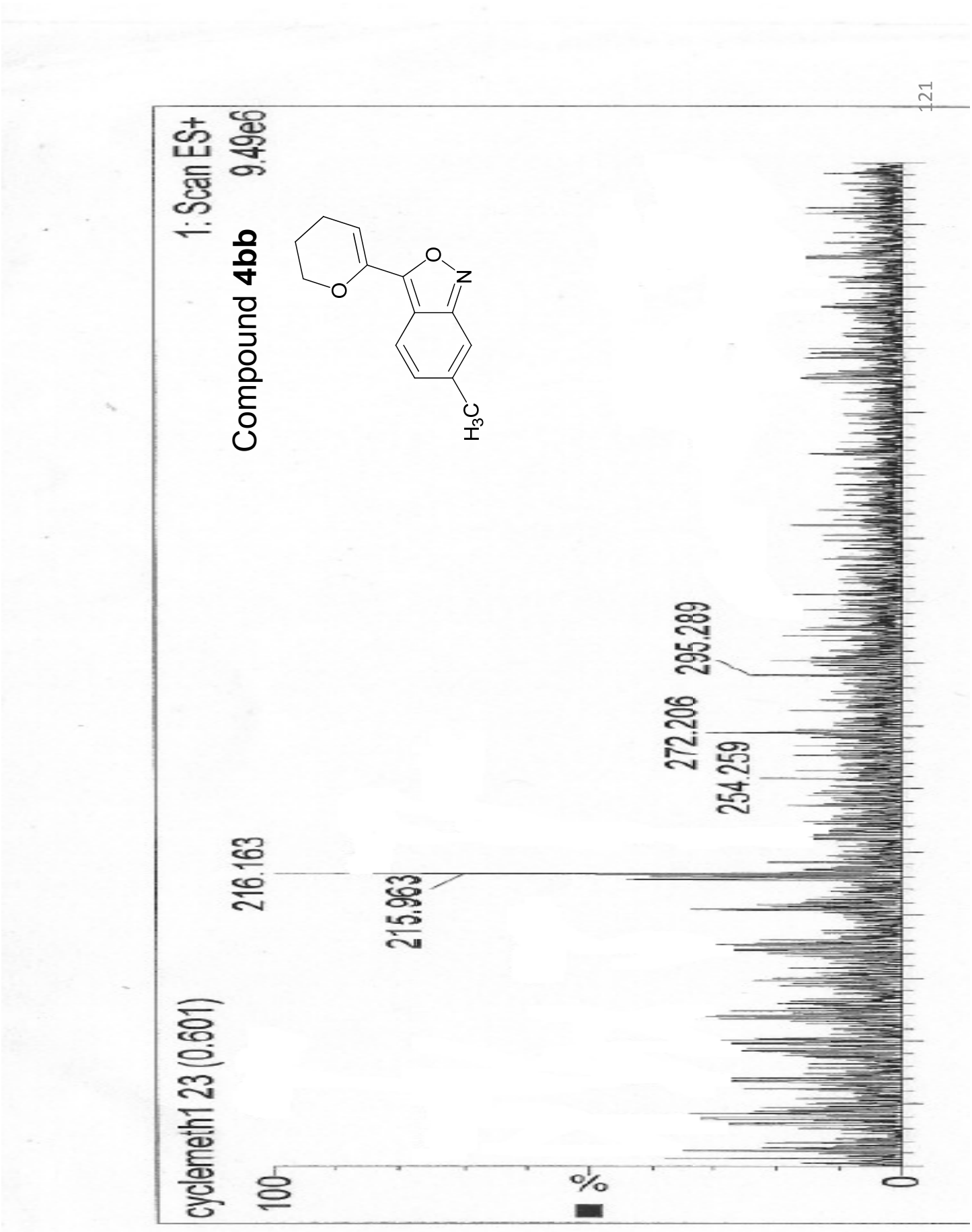


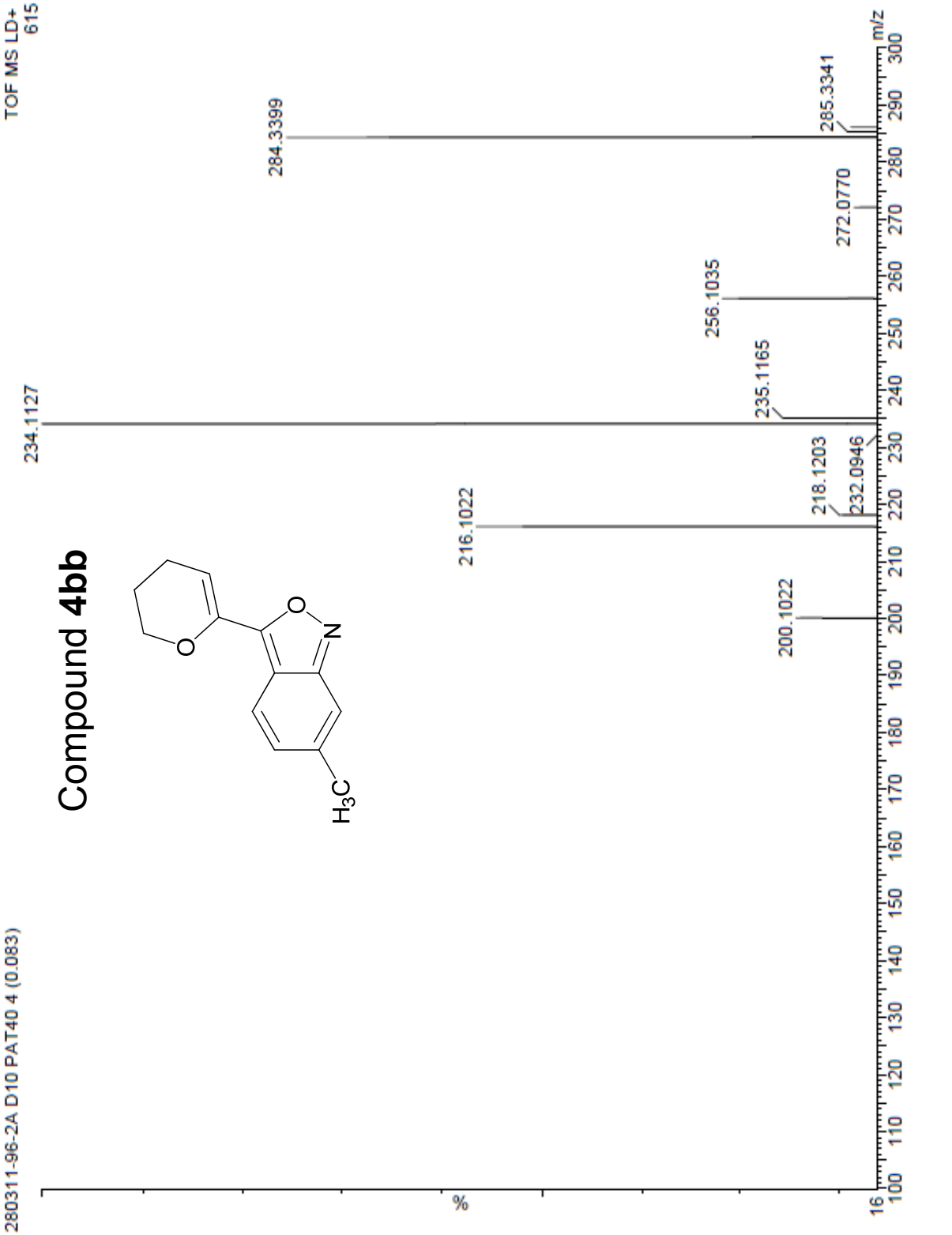


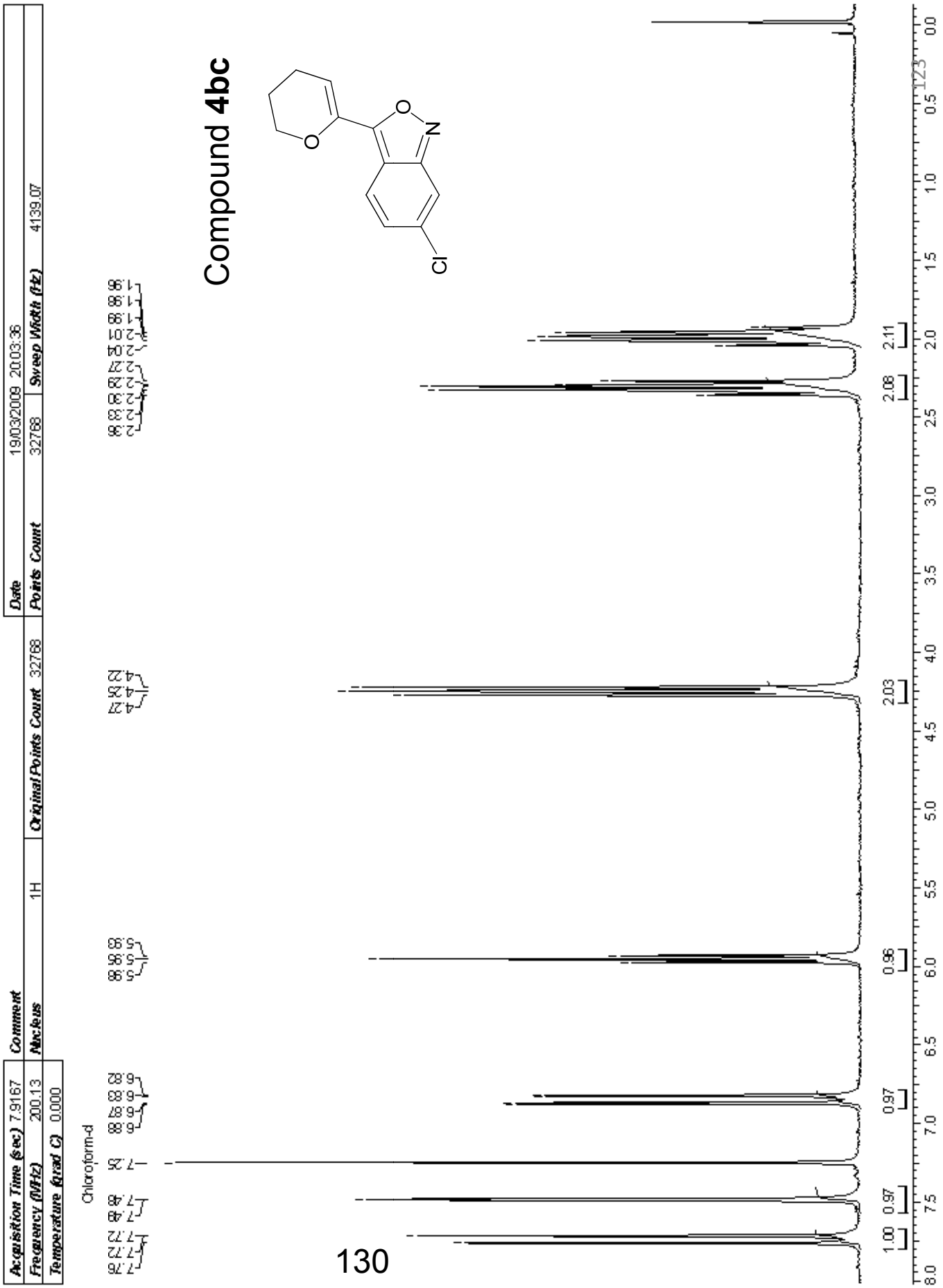


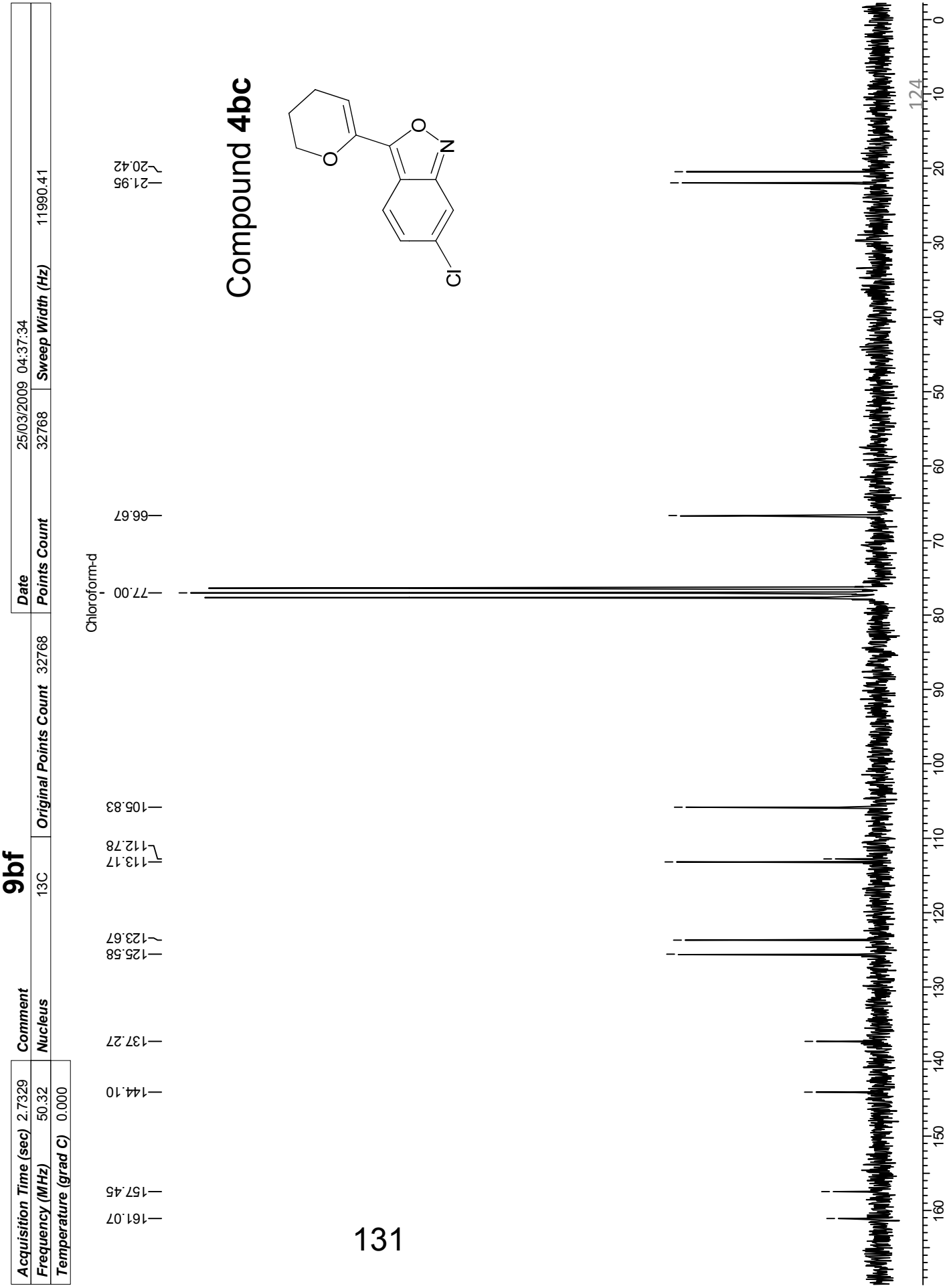


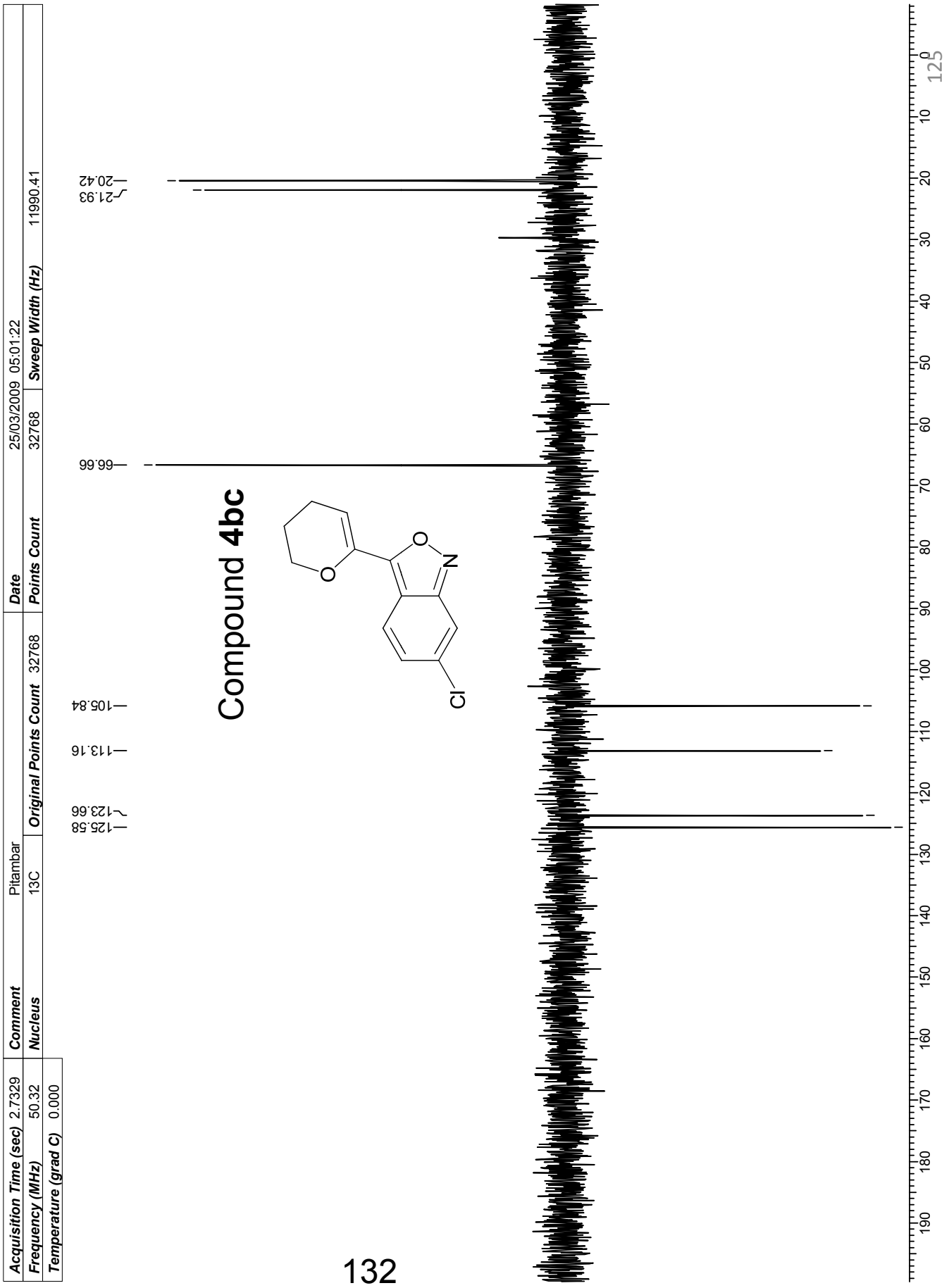

Compound 4bb
O
O
N
H3C
20.44
22.05
22.44
66.60
104.74
112.15
121.44
127.21
Acquisition Time (sec) 1.3631
Original Points Count 32768
Date 18/11/2008 08:48:00
Points Count 32768
Sweep Width (Hz) 24038.46
Frequency (MHz) 100.61
Nucleus 13C
Temperature (grad C) 0.000

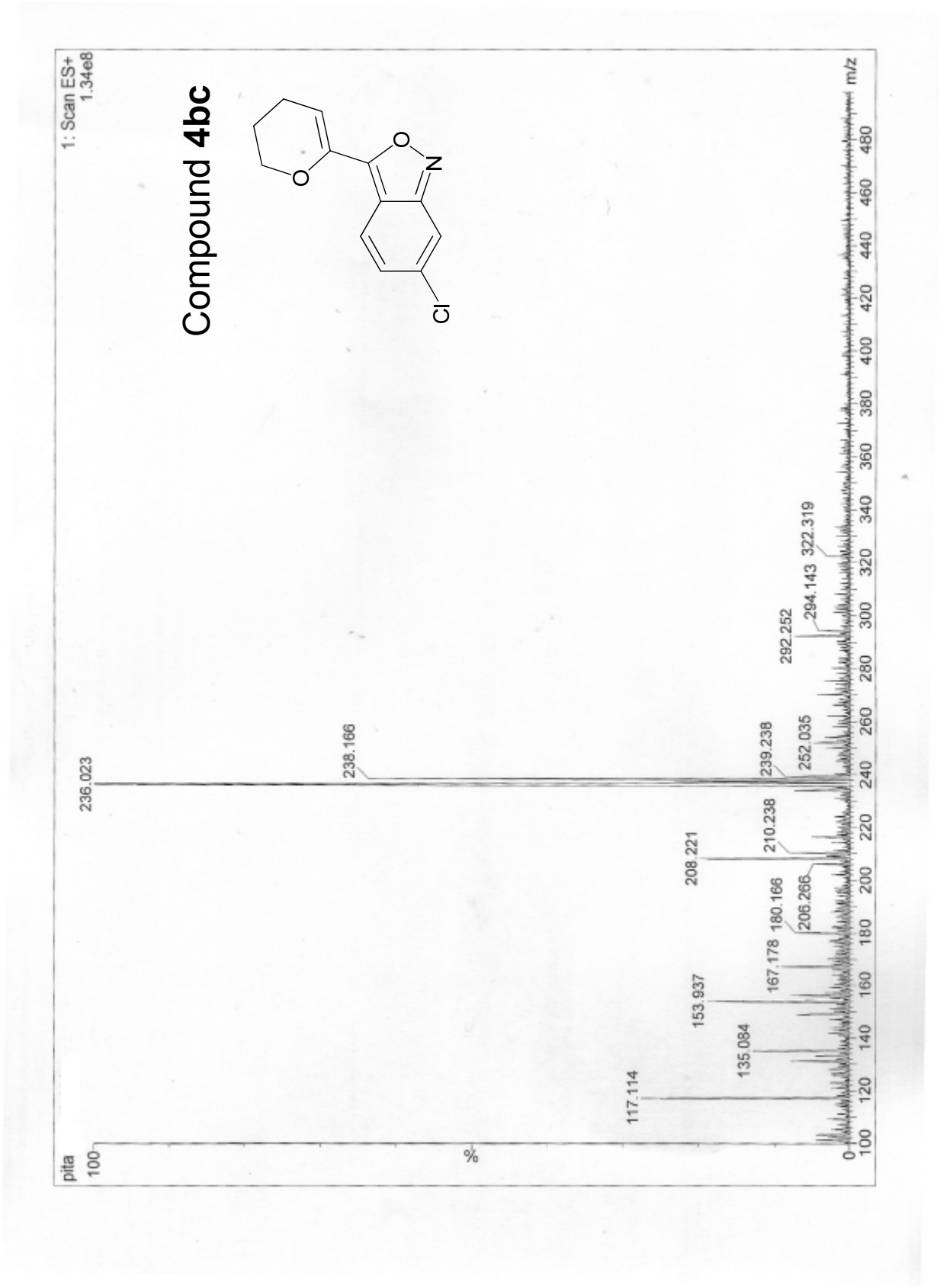


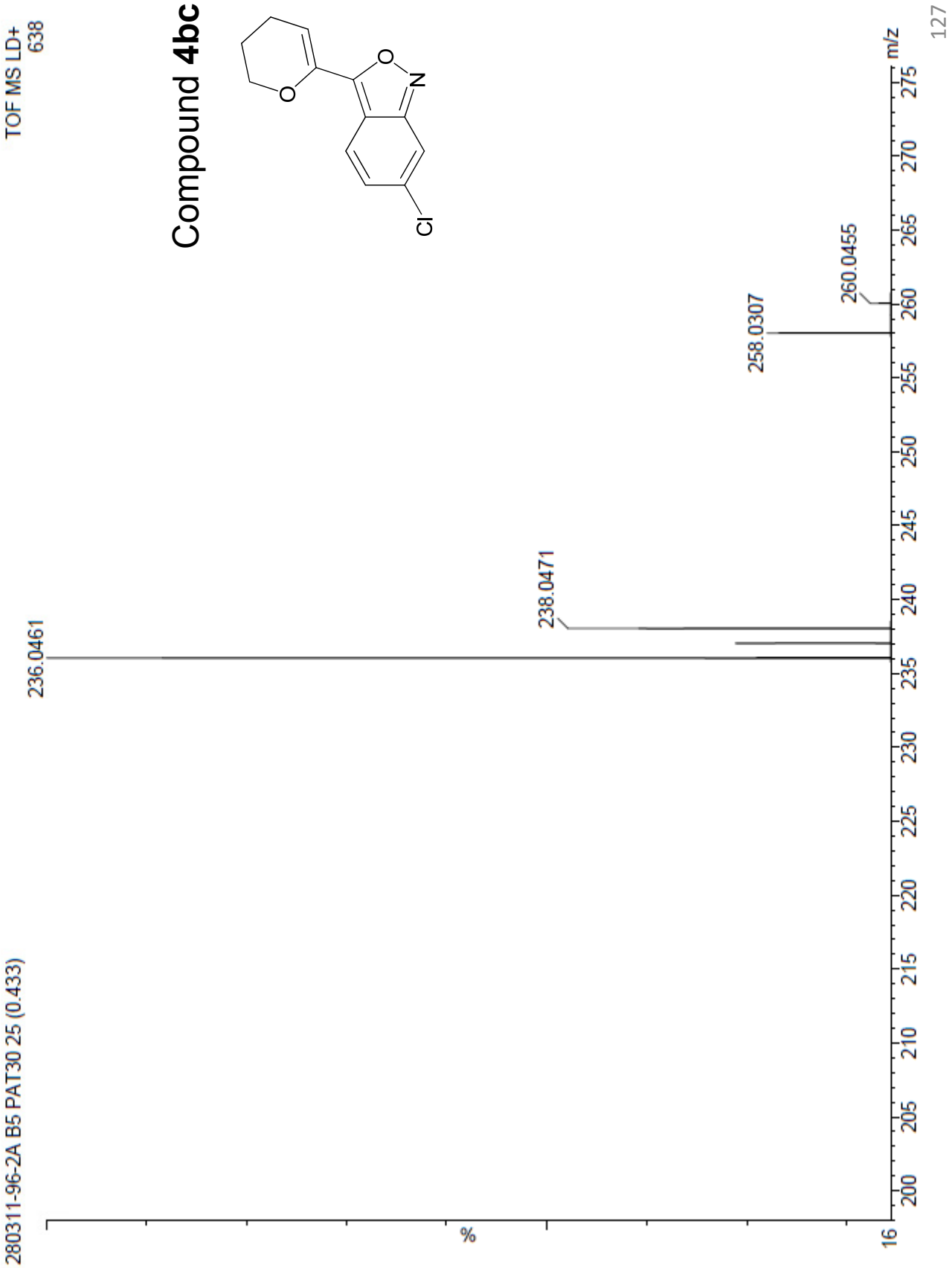

TOF MS LD+
615
284.3399
285.3341
272.0770
256.1035
235.1165
234.1127
232.0946
218.1203
216.1022
200.1022
m/z
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300
%
16
280311-96-2A D10 PAT40 4 (0.083)
Compound 4bb
O
O
N
H3C

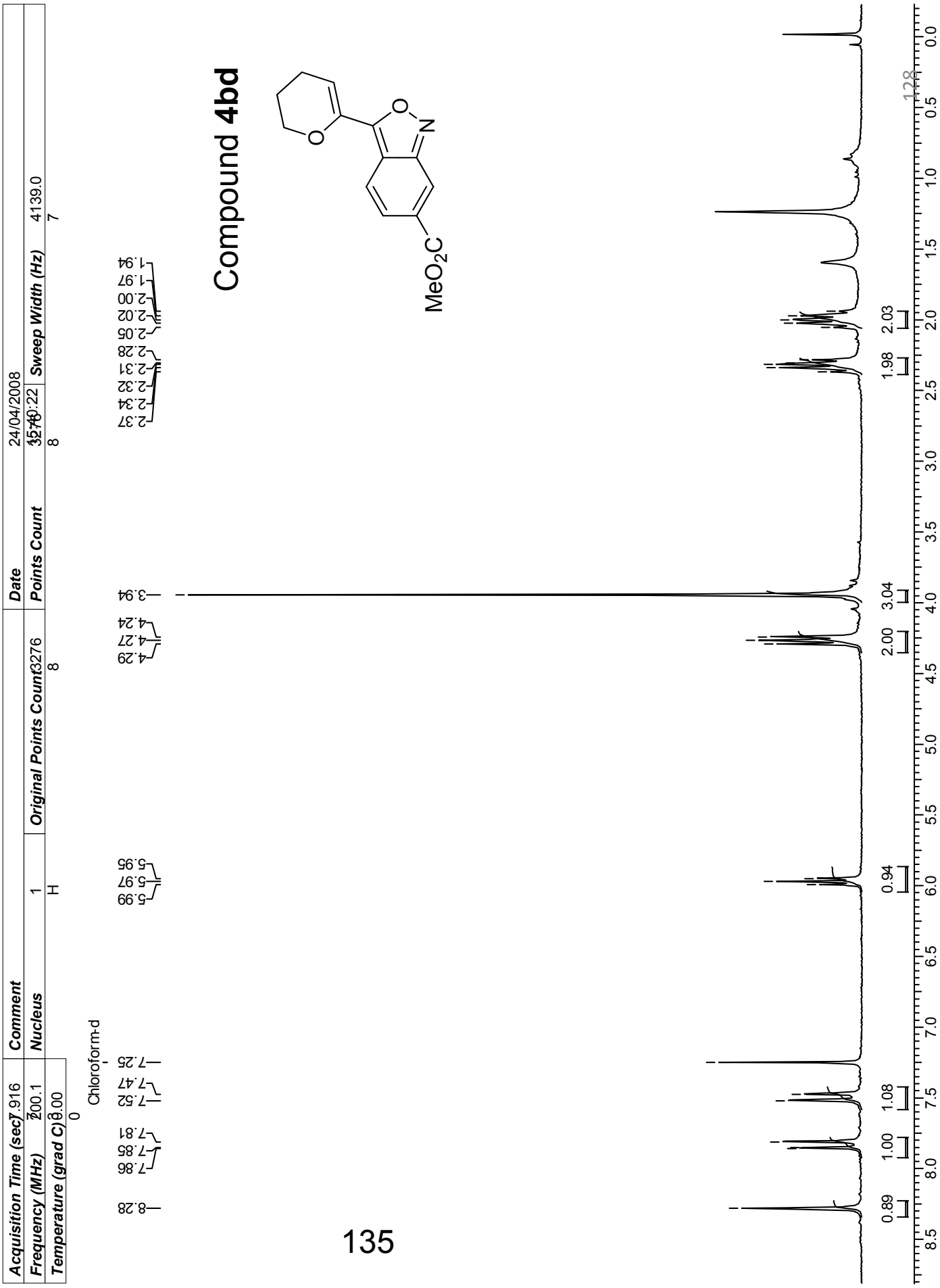


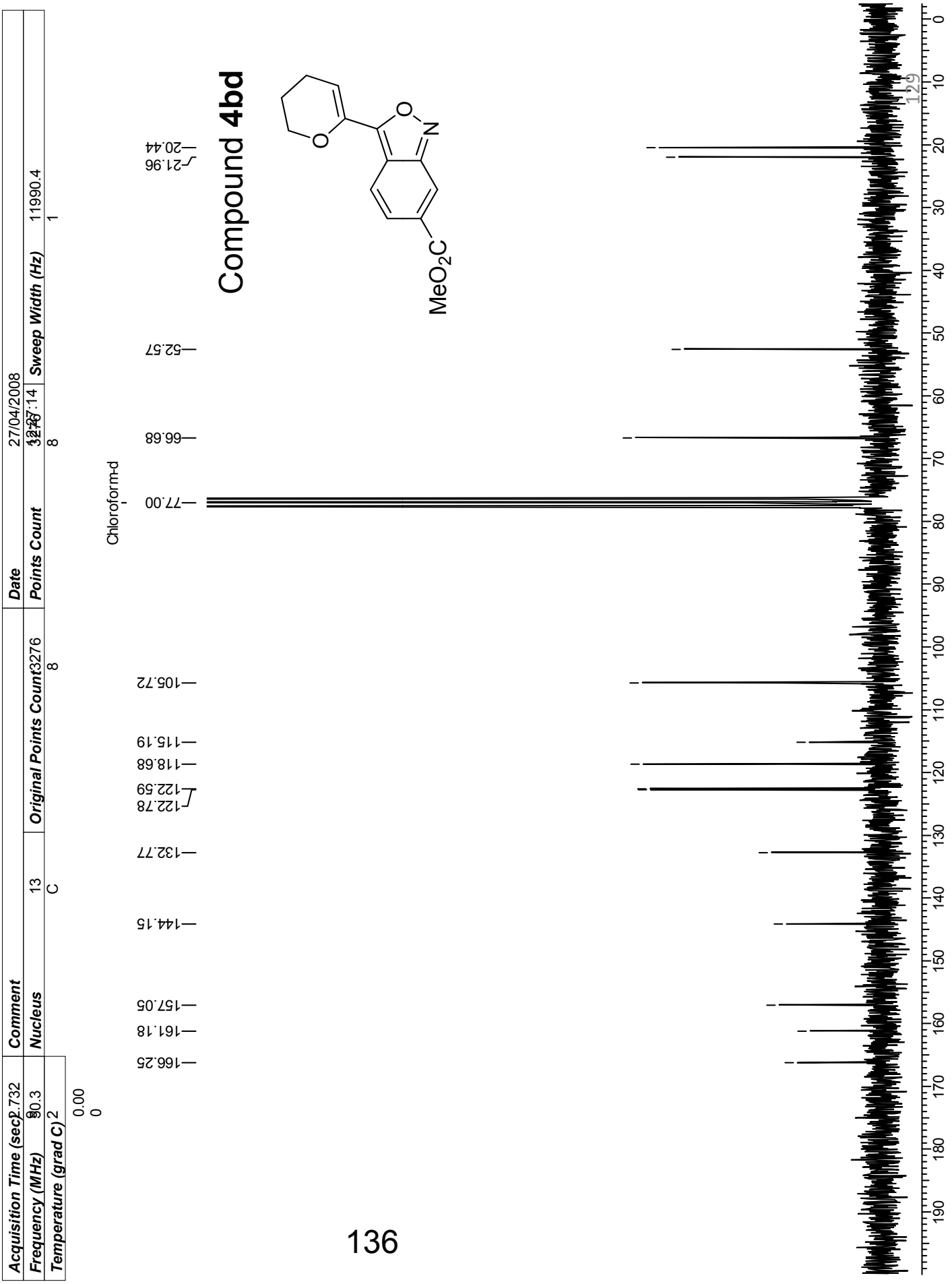


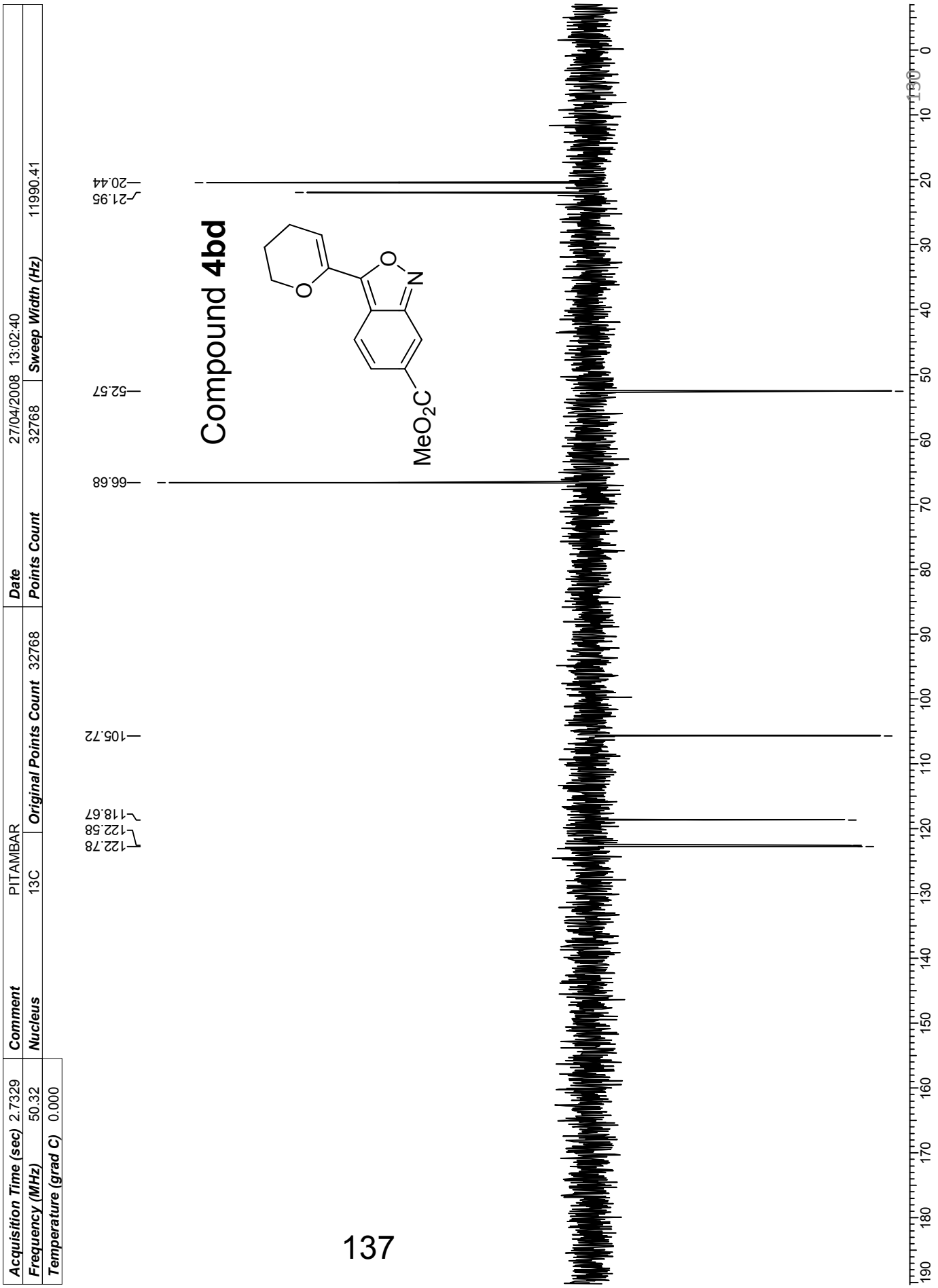


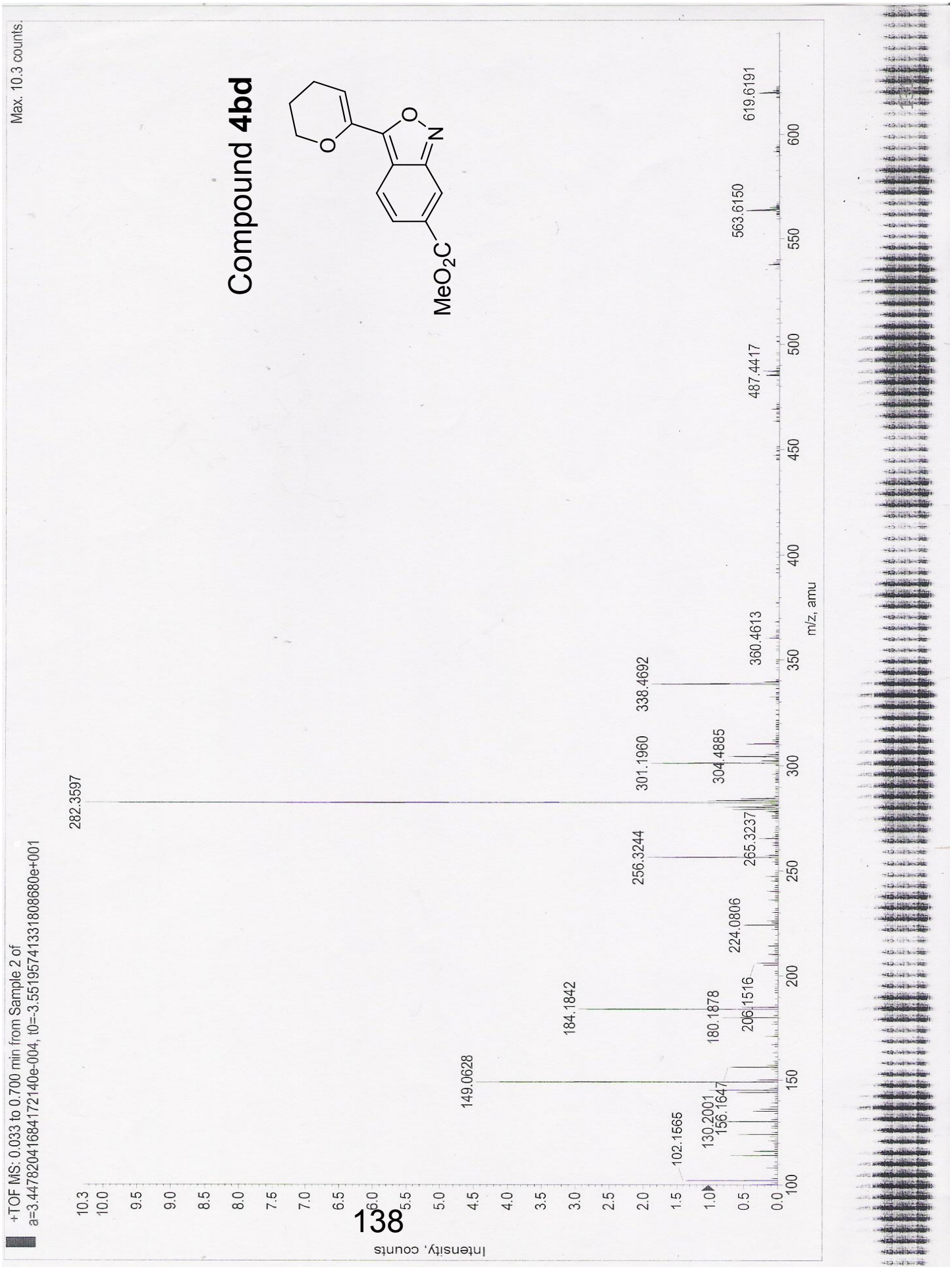

Compound 4bc
1: Scan ES+
1.34e8
pita
100
%
0
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
m/z
236.023
238.166
239.238
252.035
292.252
294.143
322.319
208.221
210.238
206.266
180.166
167.178
153.937
135.084
117.114
O
O
N
Cl

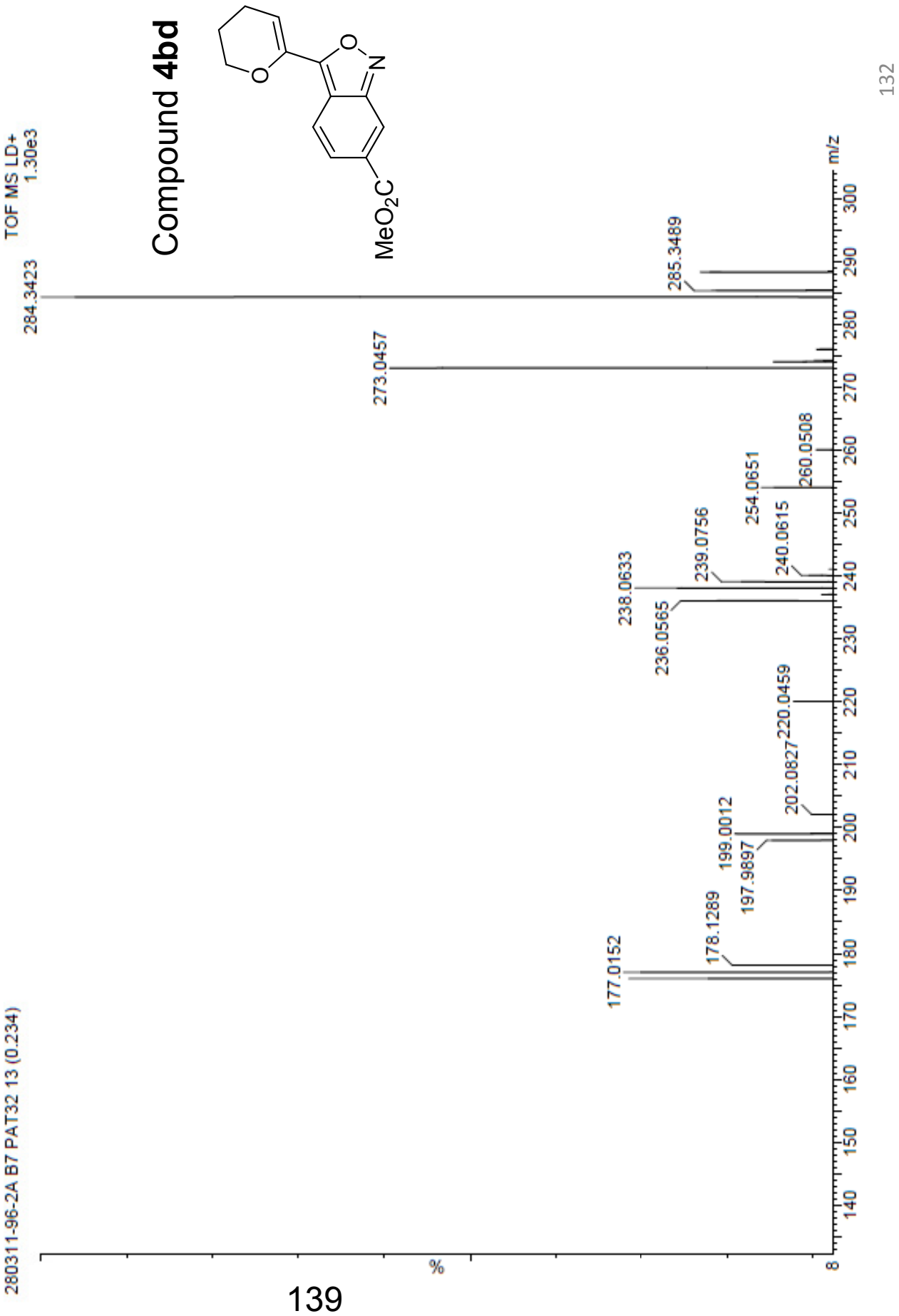











Compound 4bd
MeO2C
TOF MS LD+
1.30e3
280311-96-2A B7 PAT32 13 (0.234)
284.3423
285.3489
273.0457
260.0508
254.0651
240.0615
239.0756
238.0633
236.0565
220.0459
202.0827
199.0012
197.9897
178.1289
177.0152
m/z
%