SUPPLEMENTARY INFORMATION (Part 2)

Racemic Marinopyrrole B by Total Synthesis

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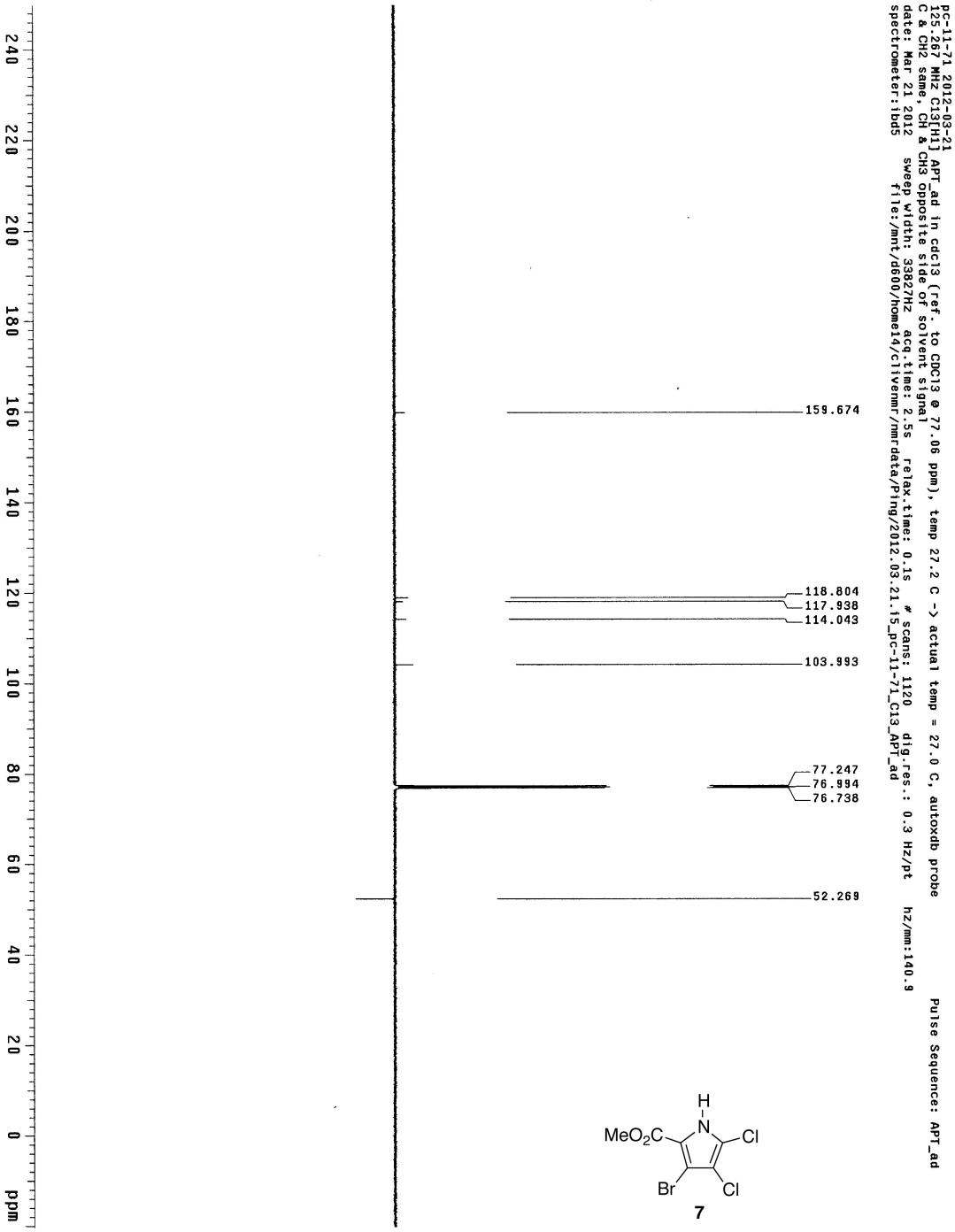
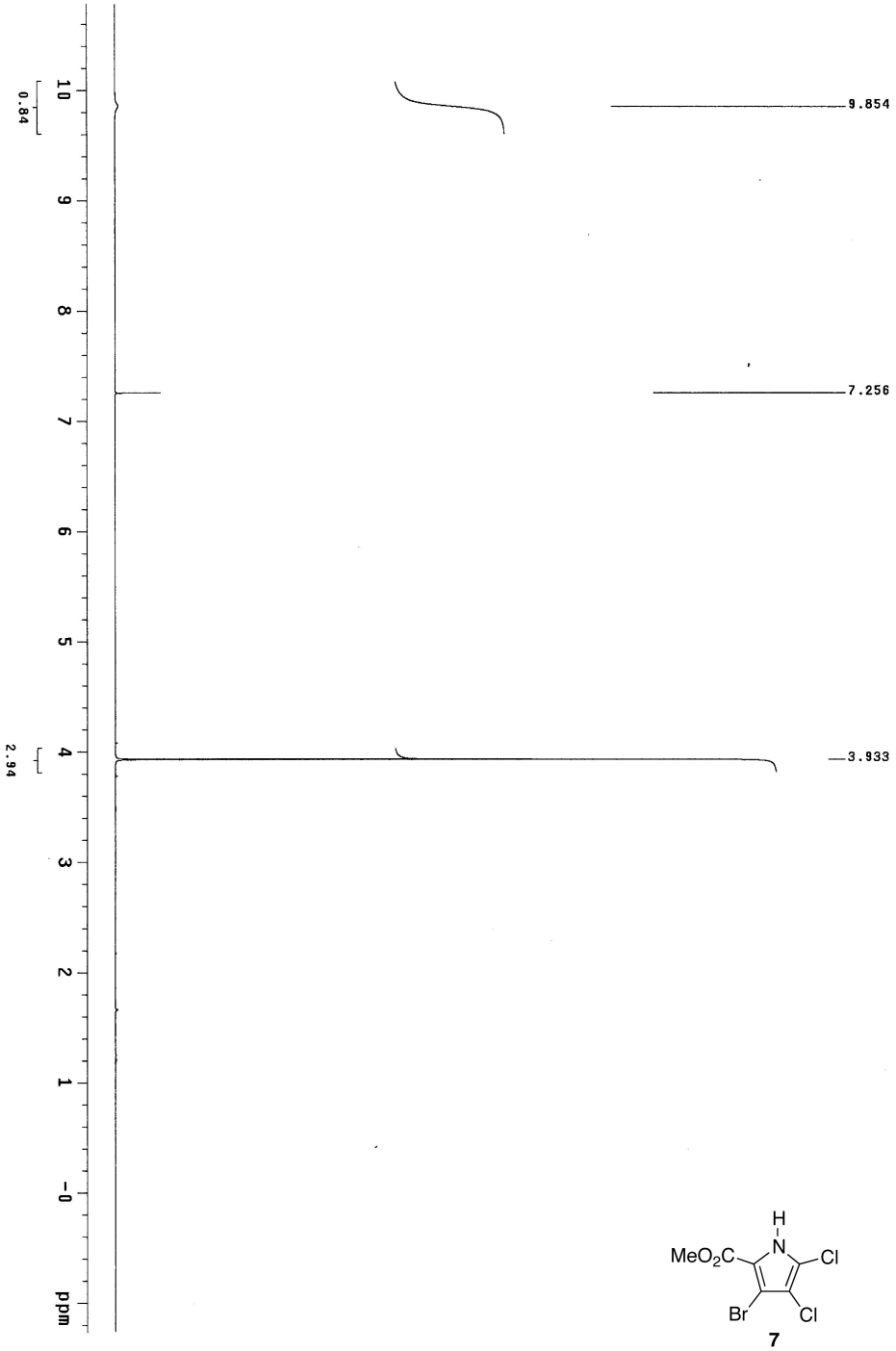
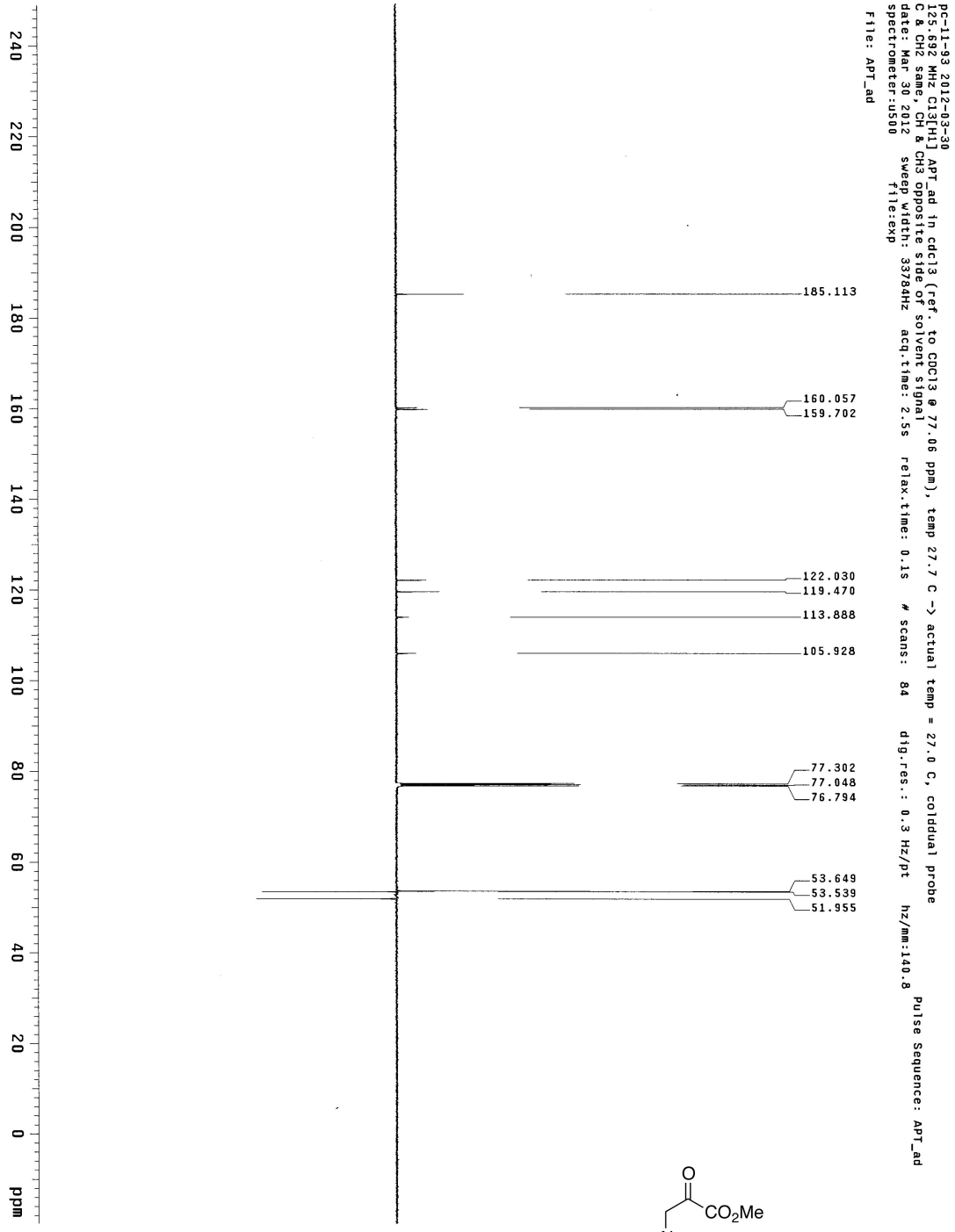
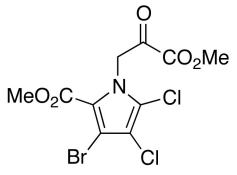
SPECTRA



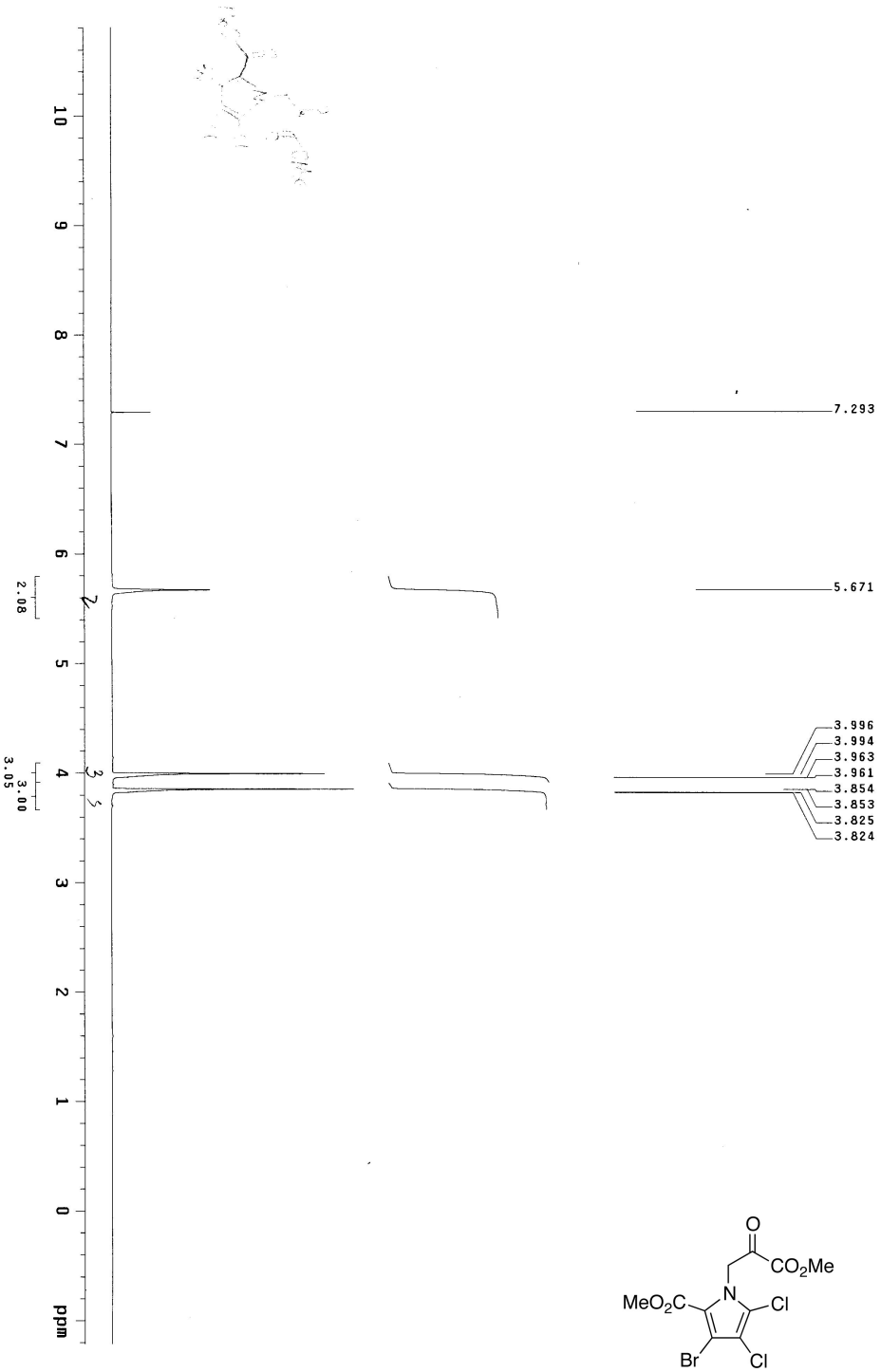


7

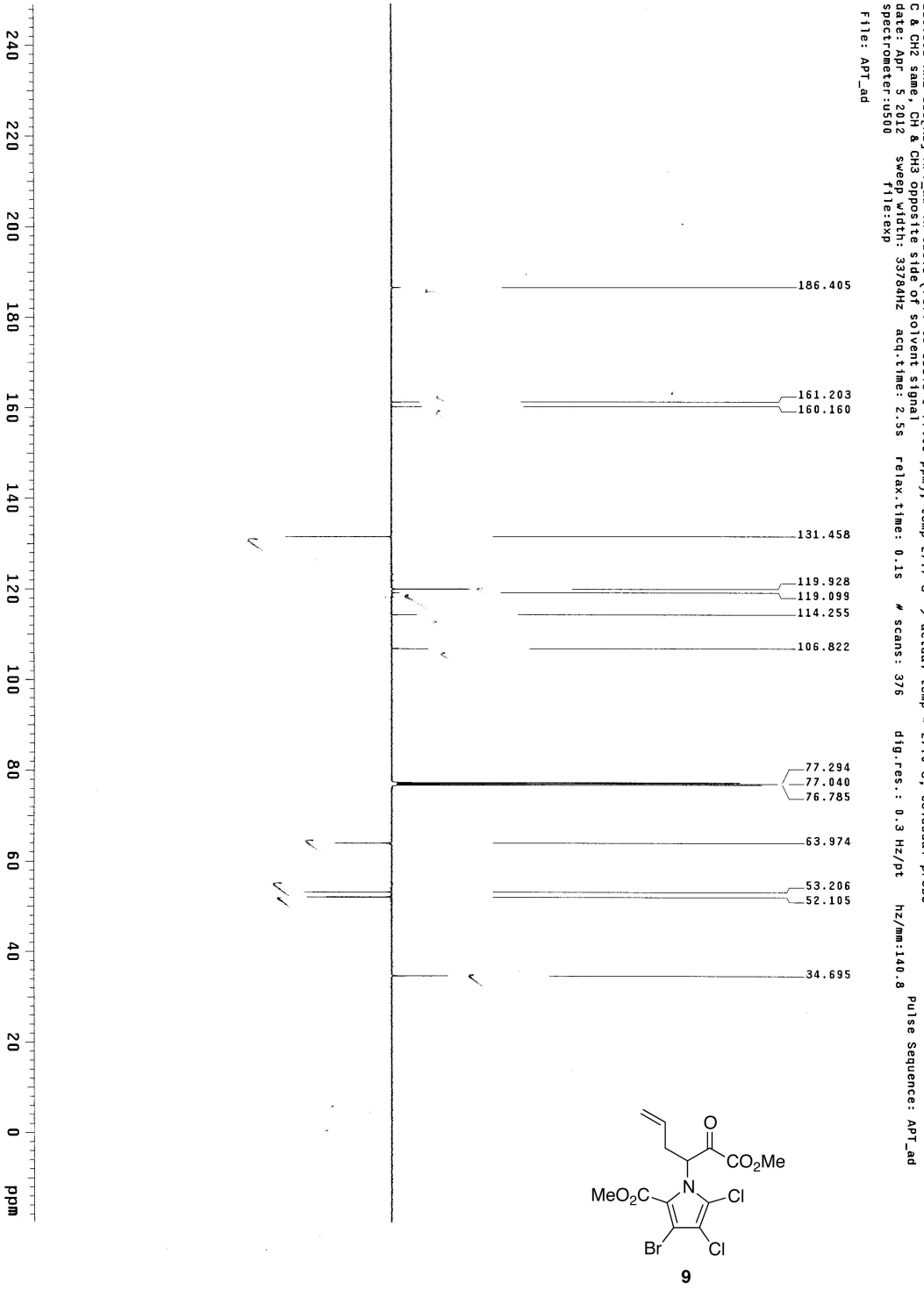

pc-11-93 2012-03-30
125.692 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Mar 30 2012 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 84 dig.res.: 0.3 Hz/pt hz/mm:140.8 Pulse Sequence: APT_ad
spectrometer:u500 file:exp
File: APT_ad
185.113
160.057
159.702
122.030
119.470
113.888
105.928
77.302
77.048
76.794
53.649
53.539
51.955
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm


O
CO2Me
MeO2C
N
Cl
Br
Cl

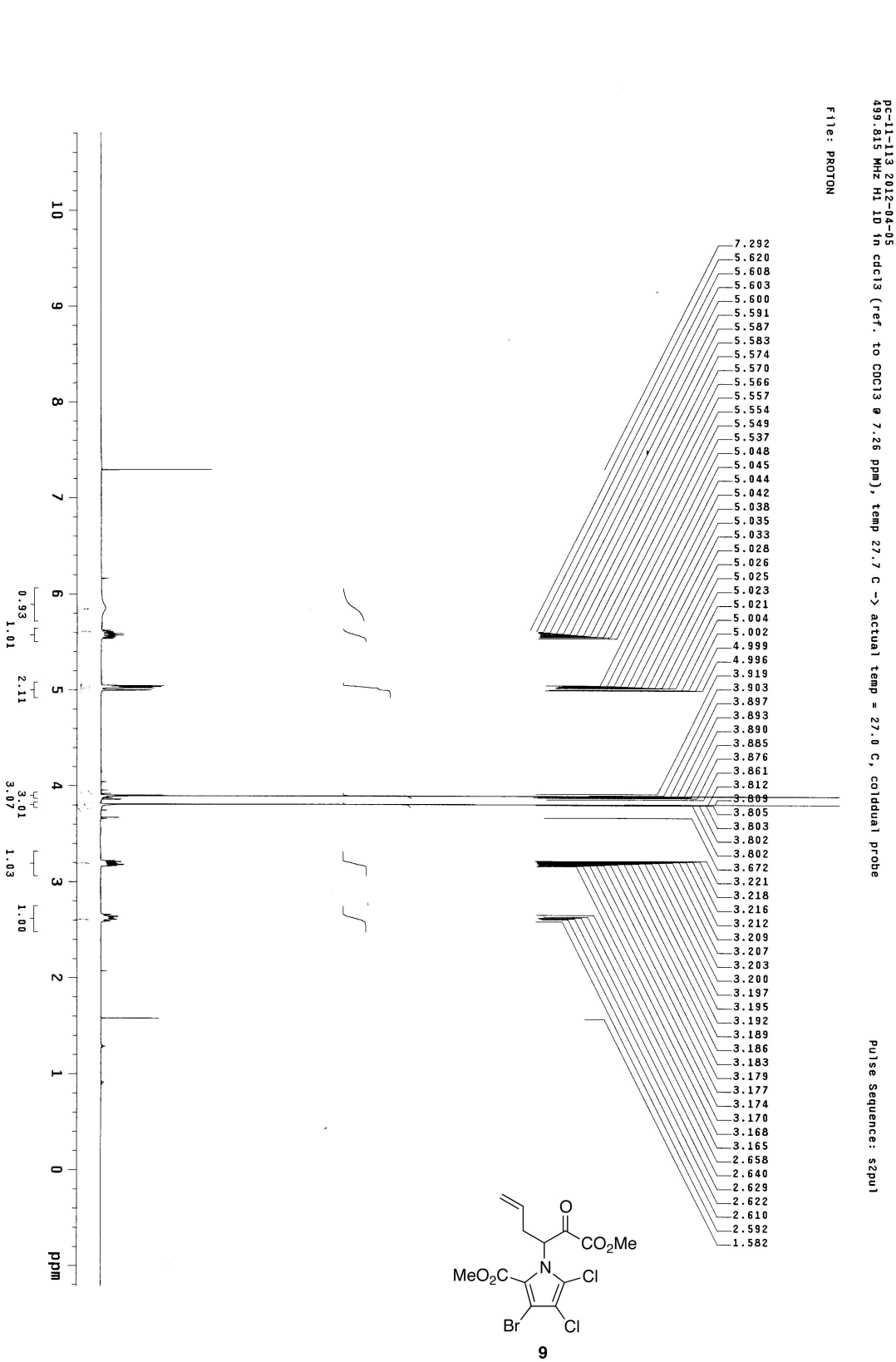
8



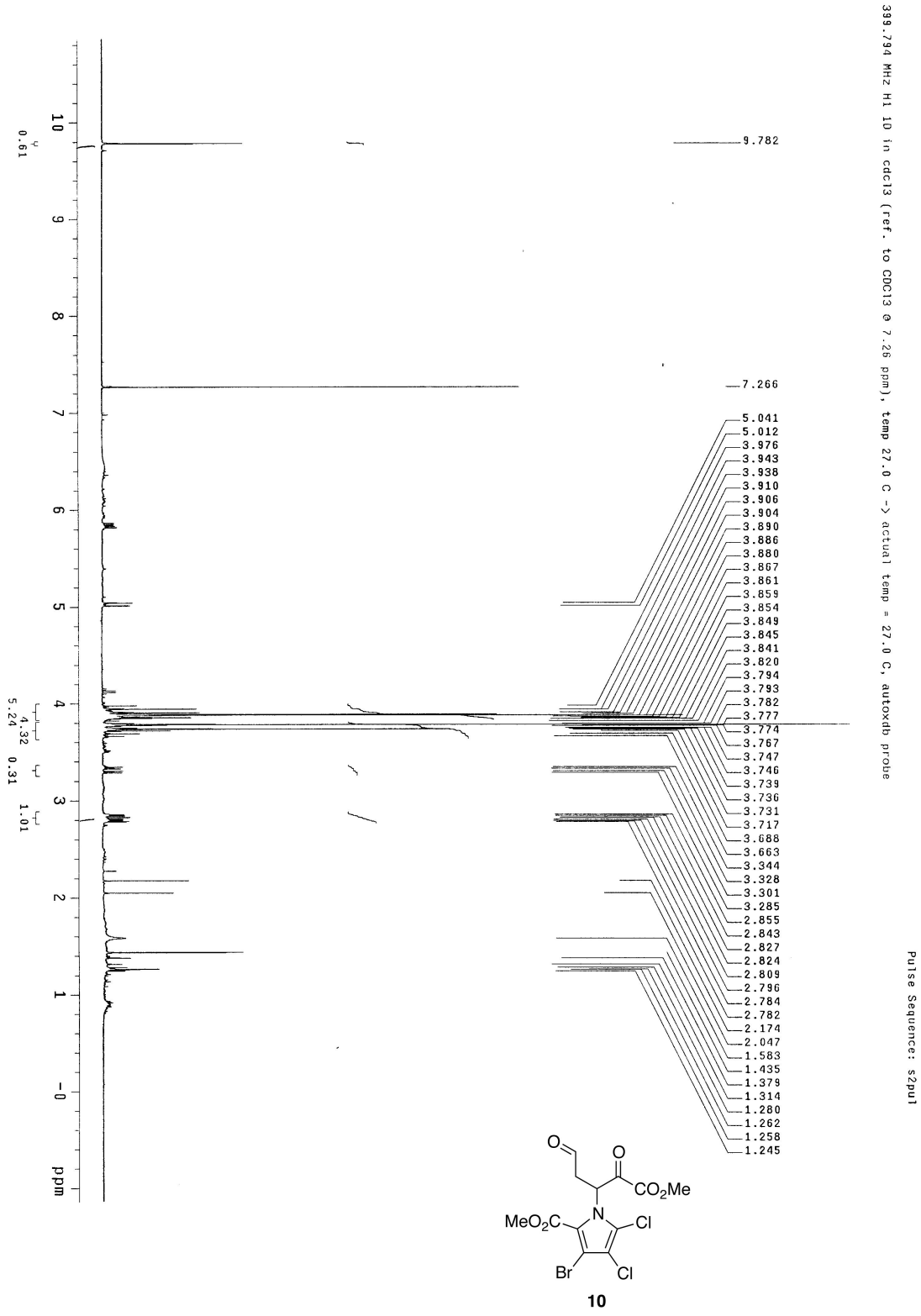
8

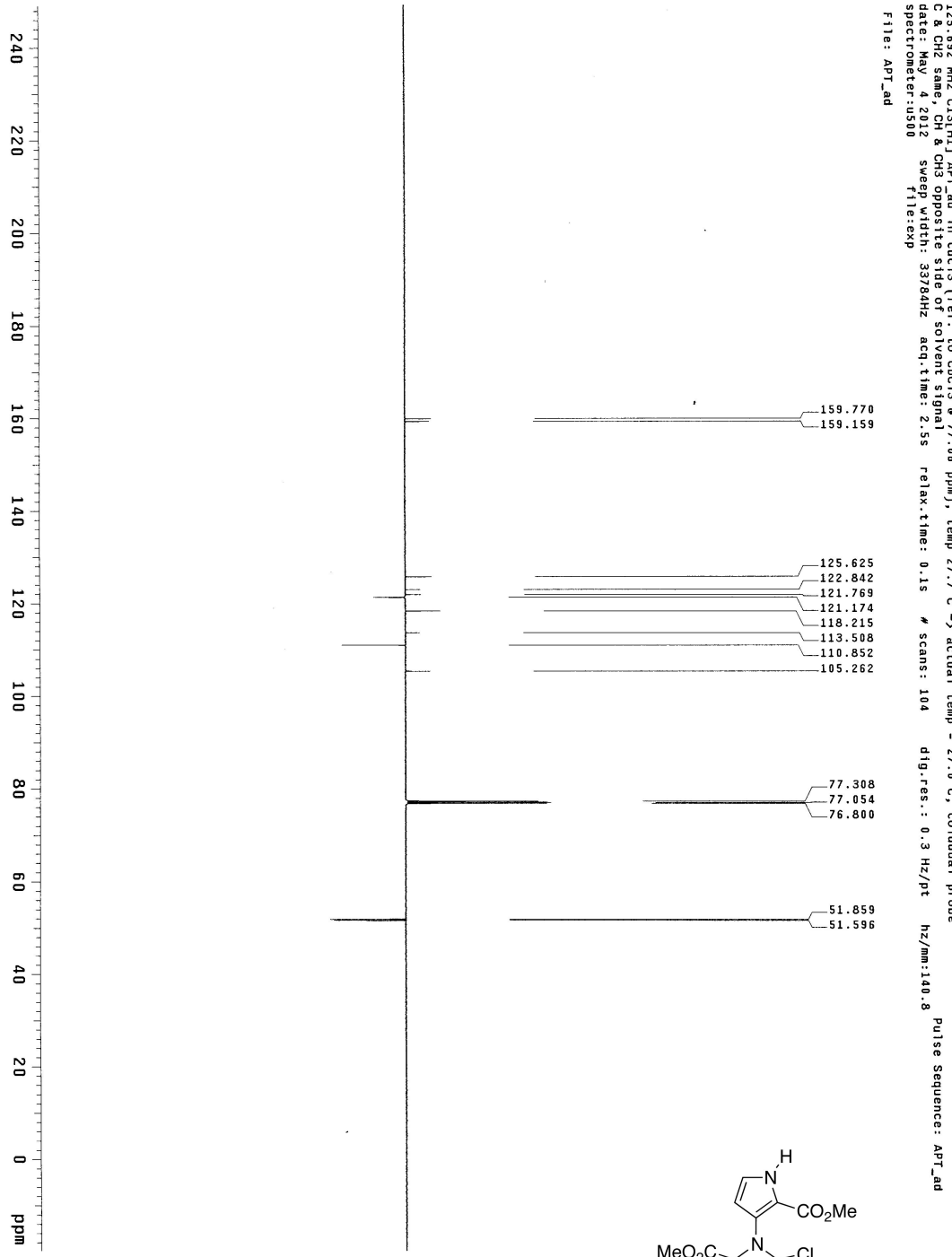

pc-11-113 2012-04-05
125.692 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Apr 5 2012 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 376 dig.res.: 0.3 Hz/pt hz/mm:140.8 Pulse Sequence: APT_ad
spectrometer:u500 file:exp
File: APT_ad
186.405
161.203
160.160
131.458
119.928
119.099
114.255
106.822
77.294
77.040
76.785
63.974
53.206
52.105
34.695
240 220 200 180 160 140 120 100 80 60 40 20 0 ppm
O
CO2Me
MeO2C
N
Cl
Br
Cl

9

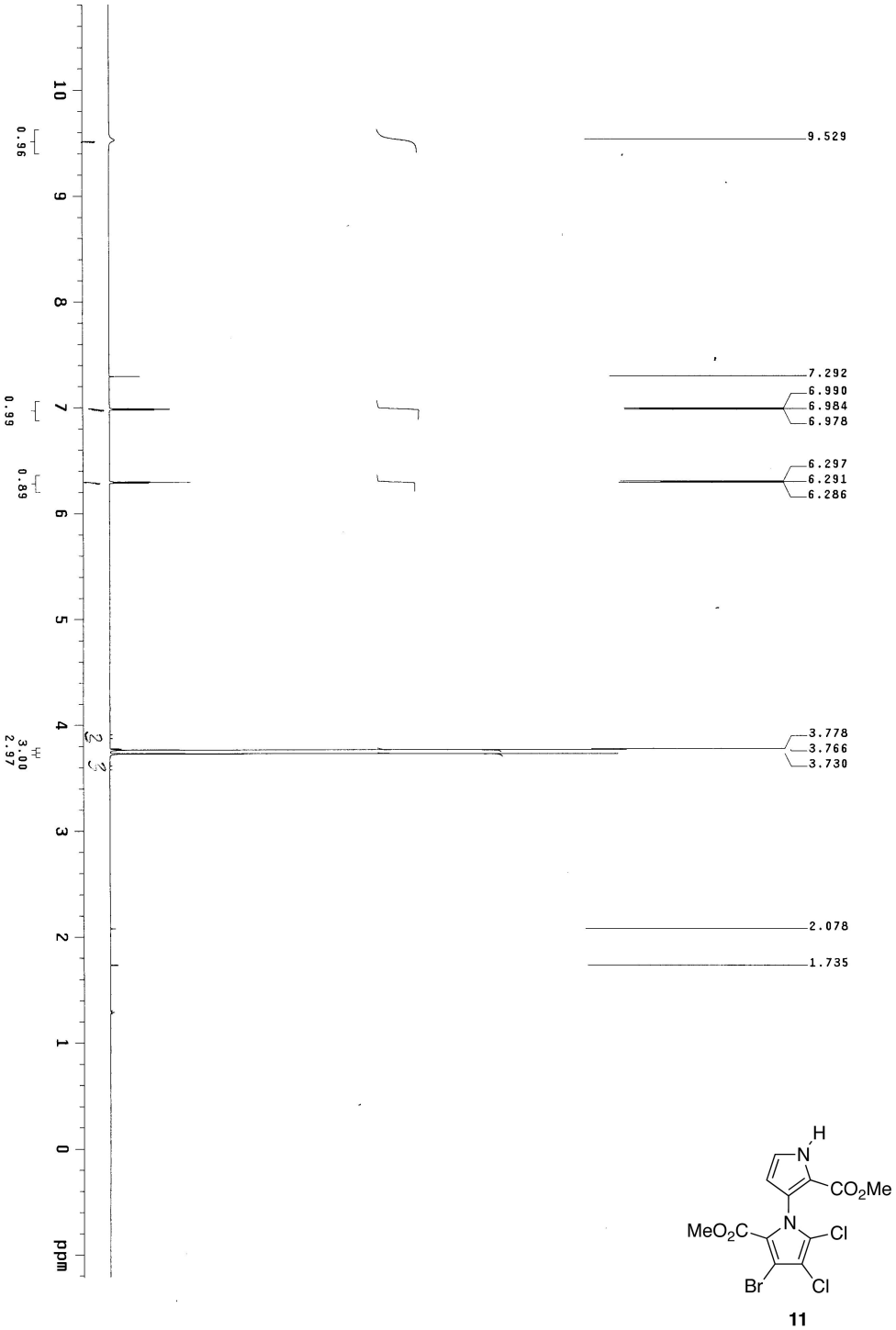


9

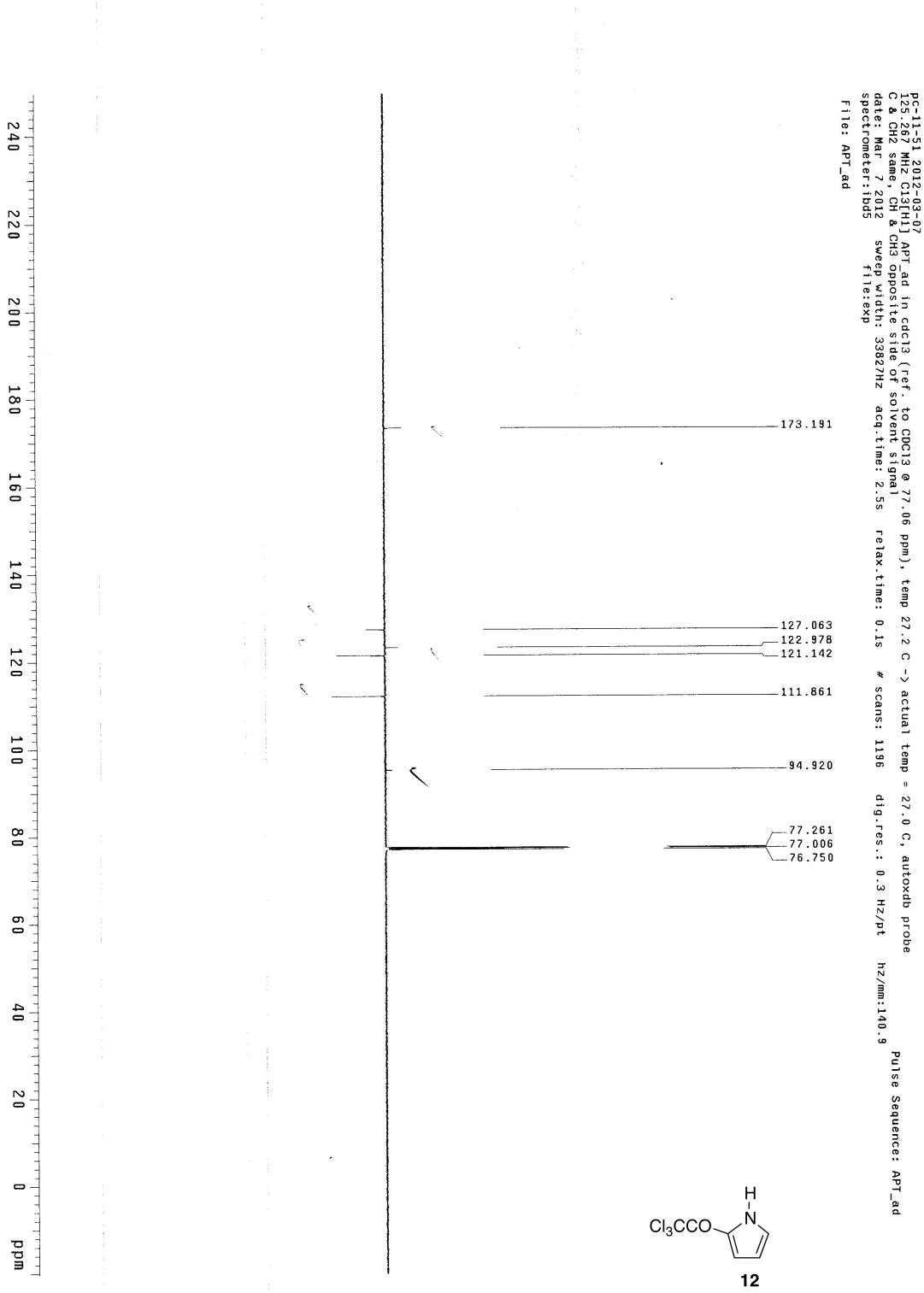



pc-11-153 2012-05-04
125.692 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: May 4 2012 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 104 dig.res.: 0.3 Hz/pt hz/mm:140.8 Pulse Sequence: APT_ad
spectrometer:u500 file:exp
File: APT_ad
159.770
159.159
125.625
122.842
121.769
121.174
118.215
113.508
110.852
105.262
77.308
77.054
76.800
51.859
51.596
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm
H
N
CO2Me
N
MeO2C
Cl
Br
Cl

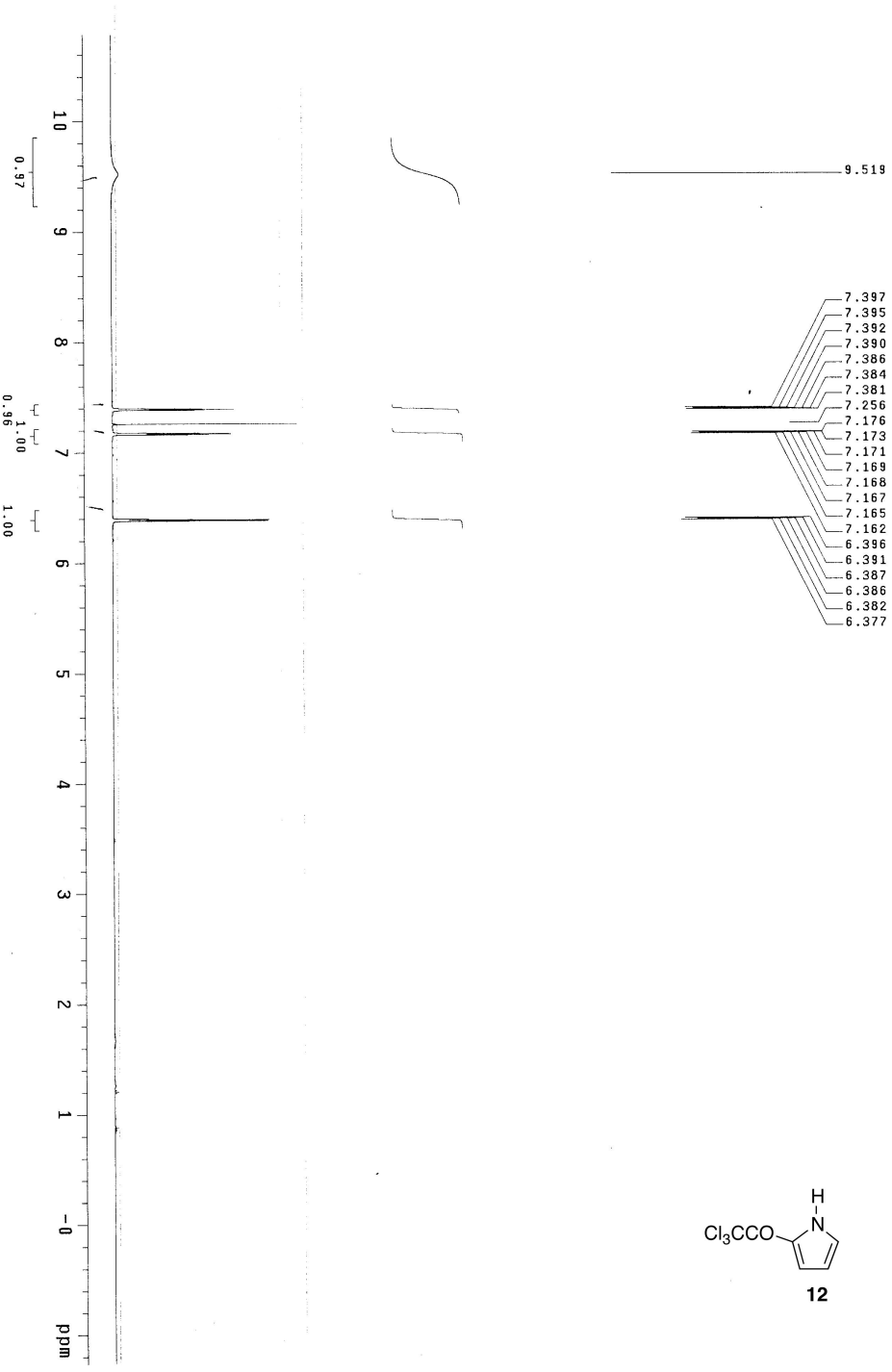
11

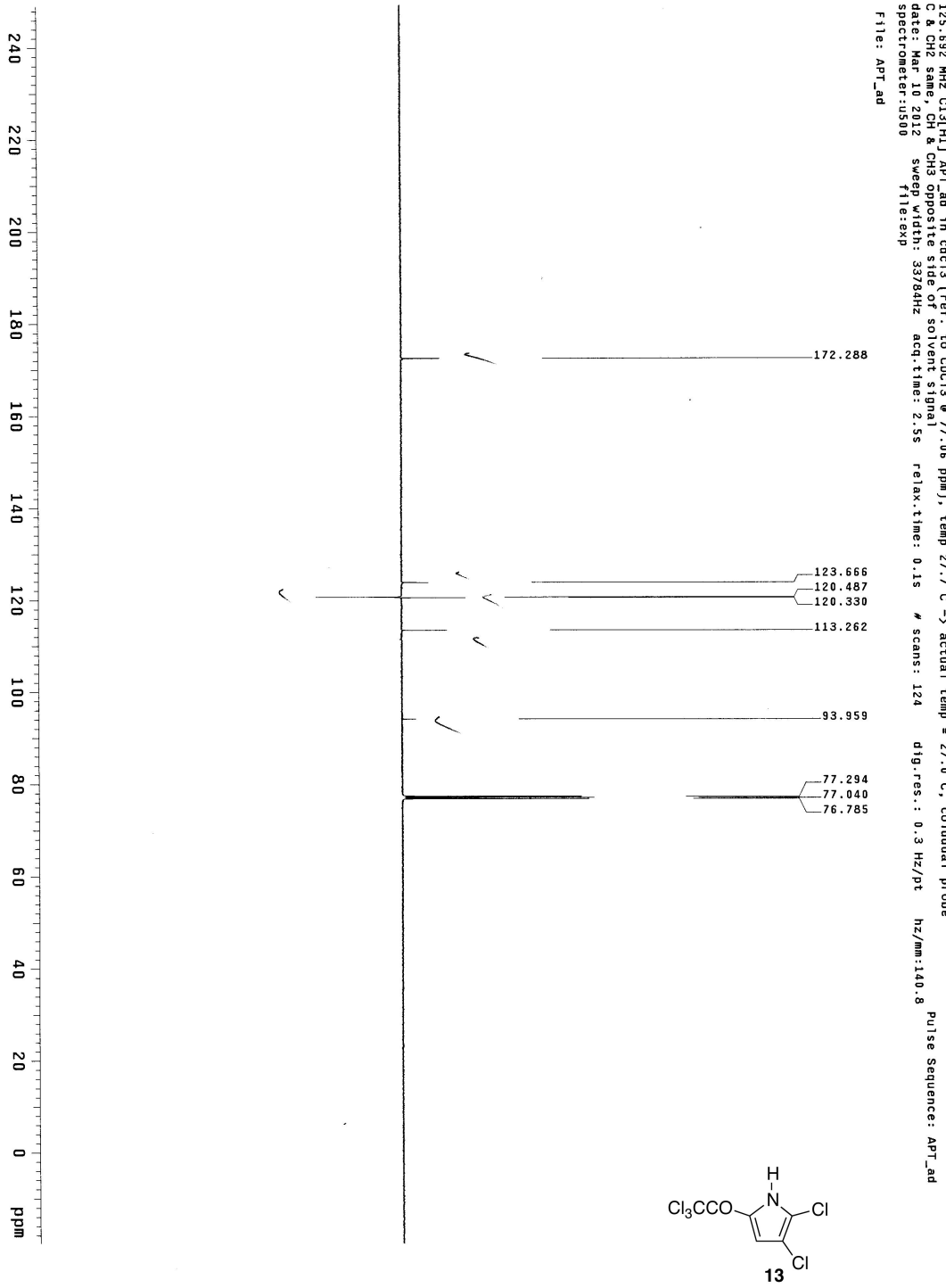


11

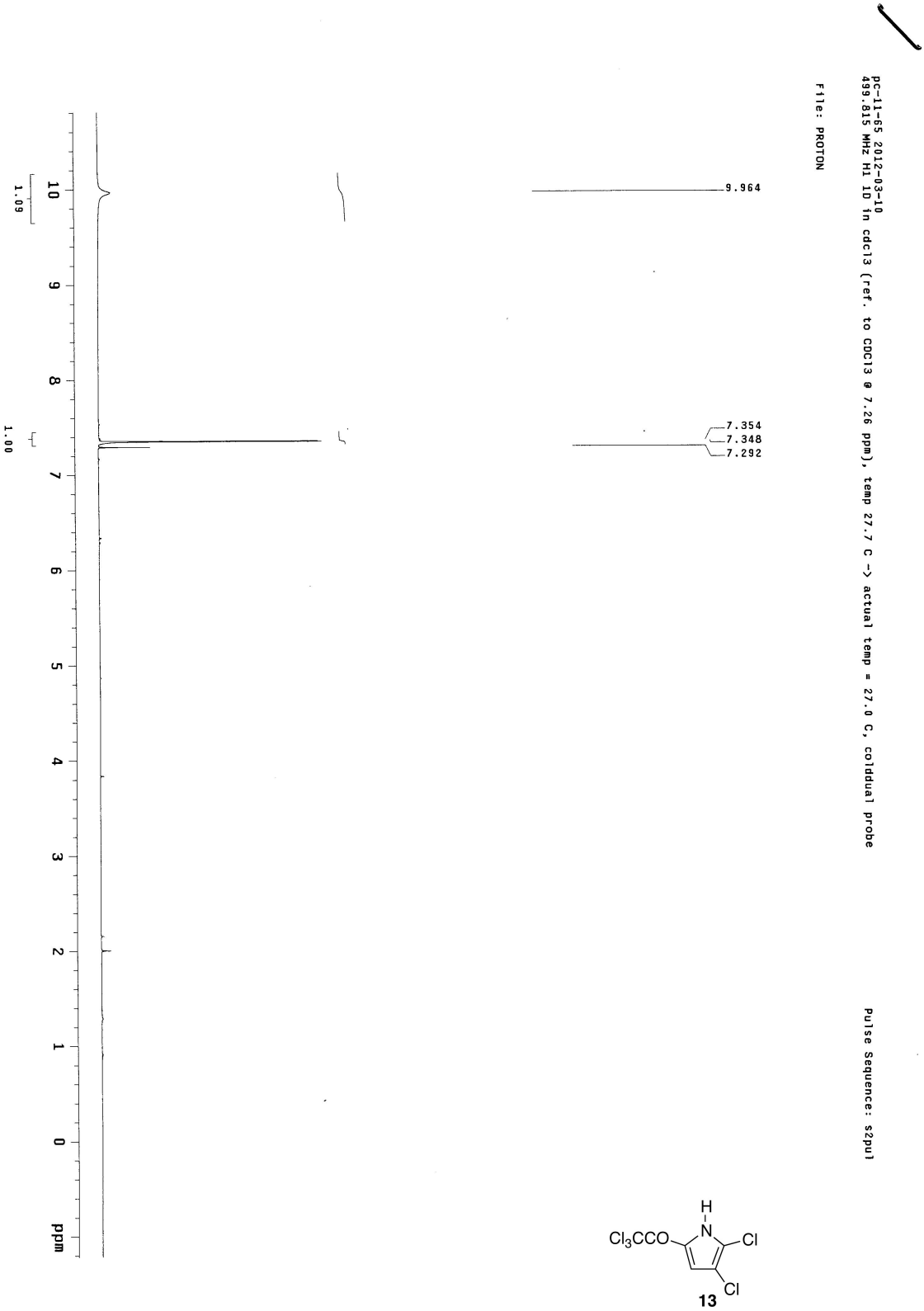

pc-11-51 2012-03-07
125.267 MHz C13{H1} APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Mar 7, 2012 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 1196 dig.res.: 0.3 Hz/pt hz/mm:140.9
spectrometer:bd3 file:exp Pulse Sequence: APT_ad
file: APT_ad
173.191
127.063
122.978
121.142
111.861
94.920
77.261
77.006
76.750
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Cl3CCO
H
N

12

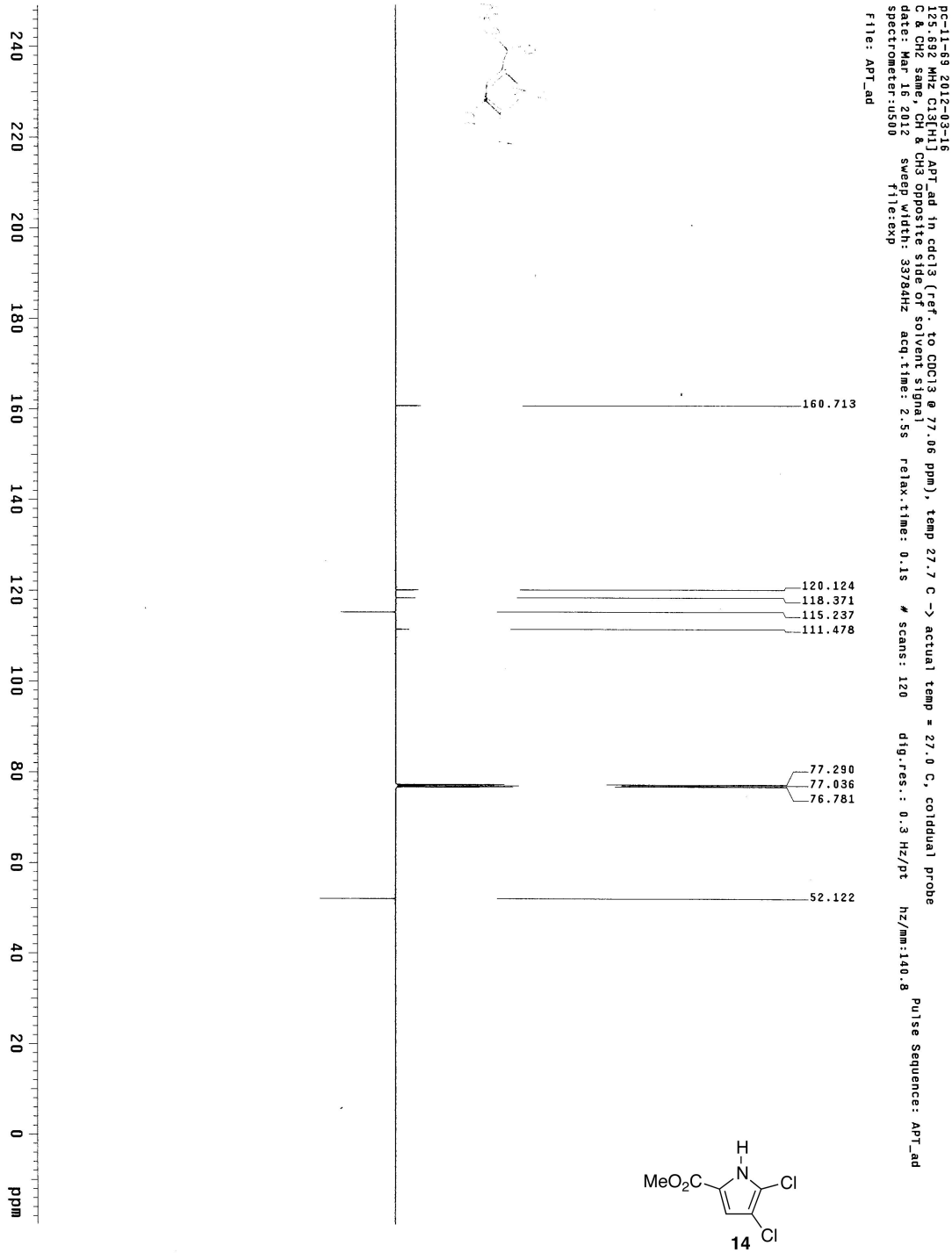


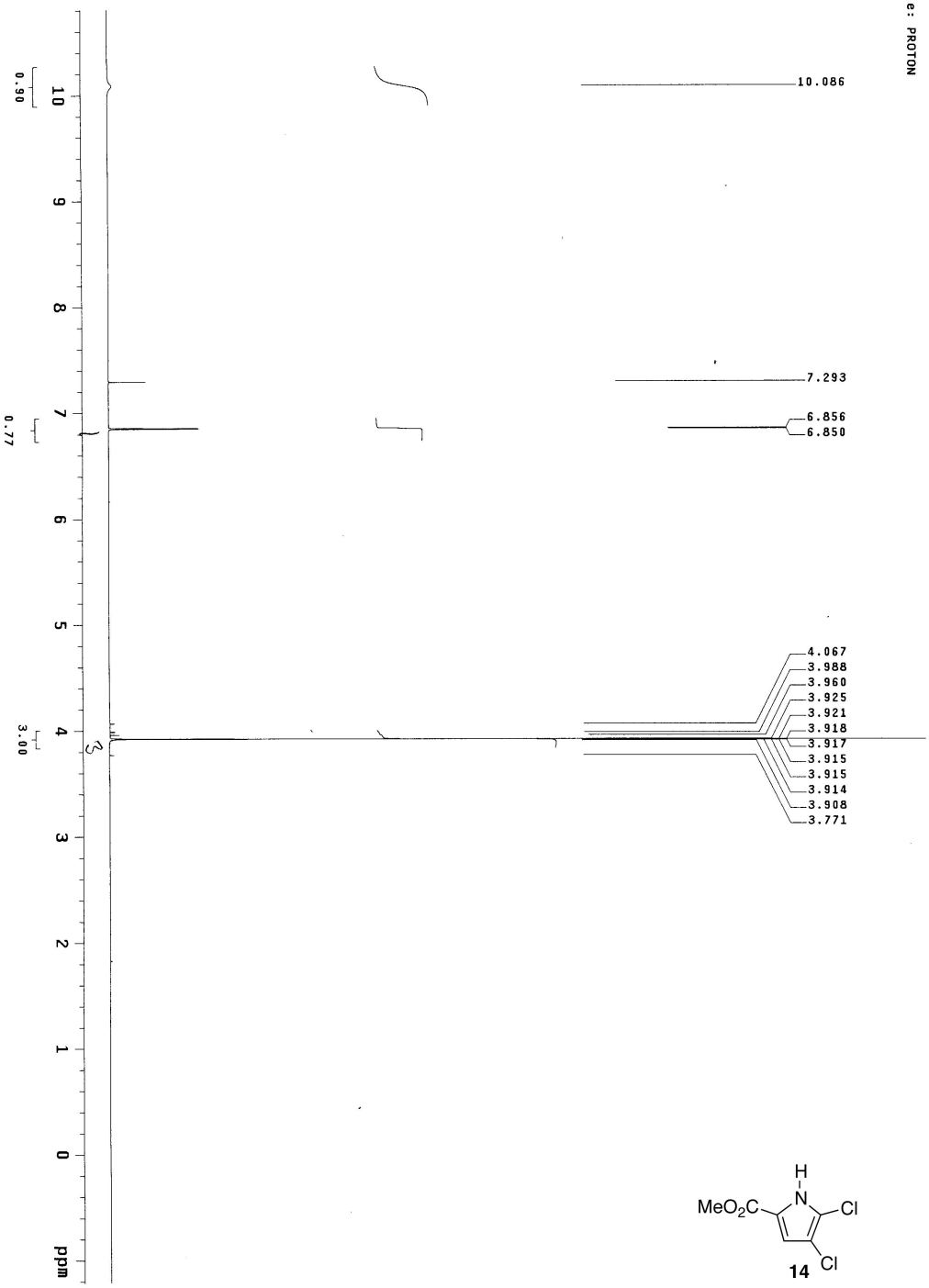

pc-11-65 2012-03-10
125.692 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Mar 10 2012 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 124 dig.res.: 0.3 Hz/pt hz/mm:140.8 Pulse Sequence: APT_ad
spectrometer:U500 file:exp
File: APT_ad
172.288
123.666
120.487
120.330
113.262
93.959
77.294
77.040
76.785
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm
H
N
Cl3CCO
Cl
Cl

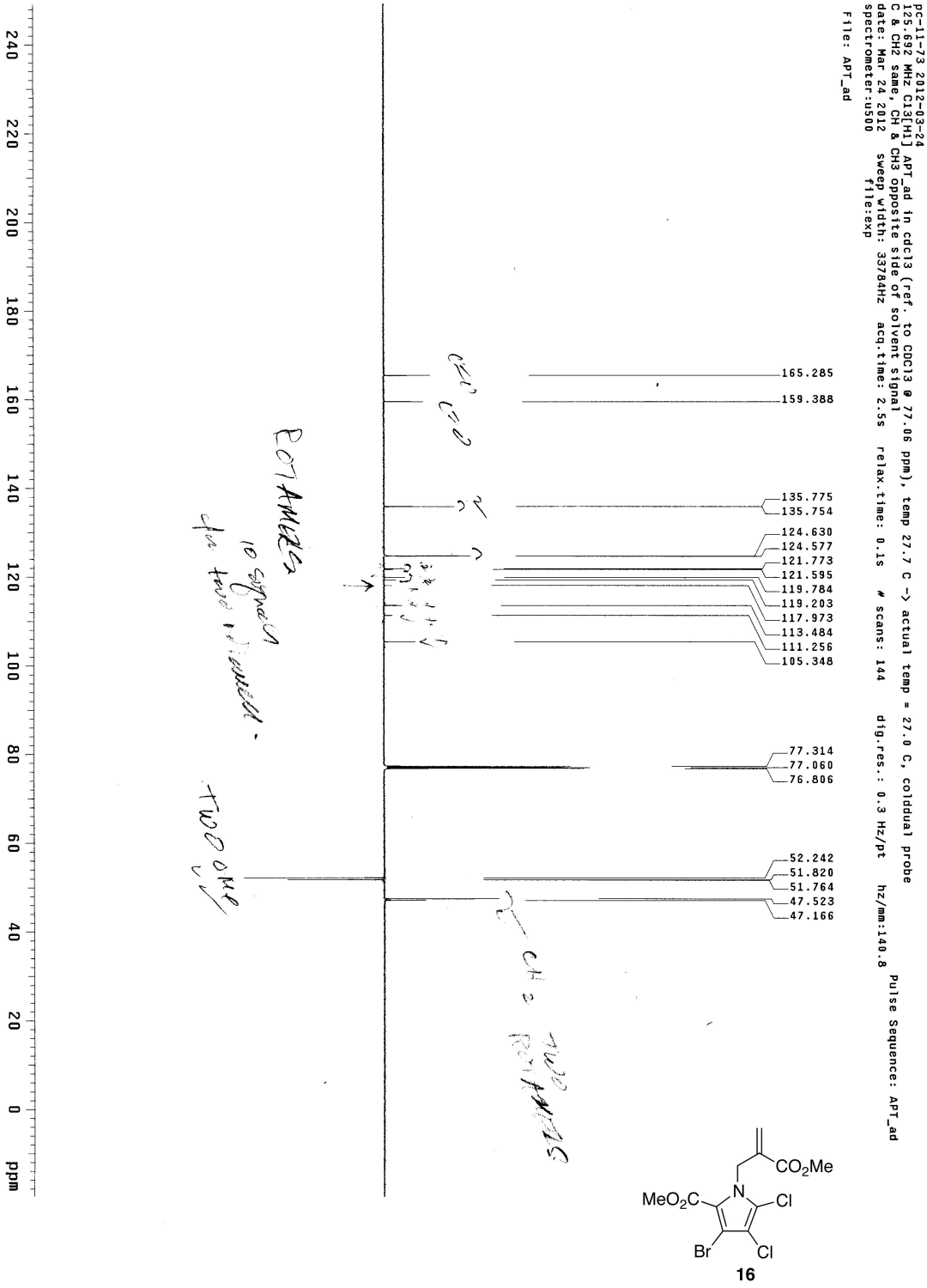
13

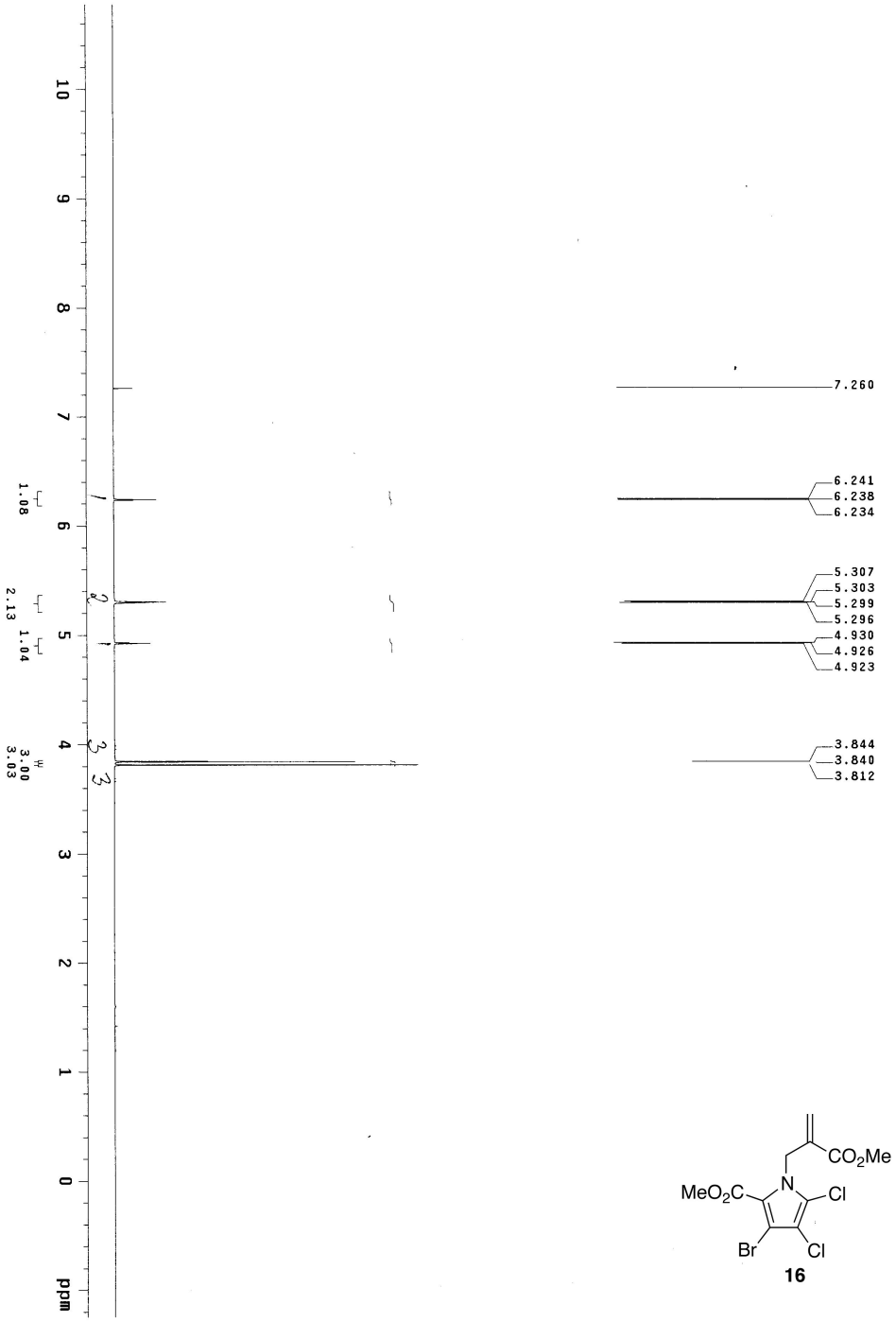


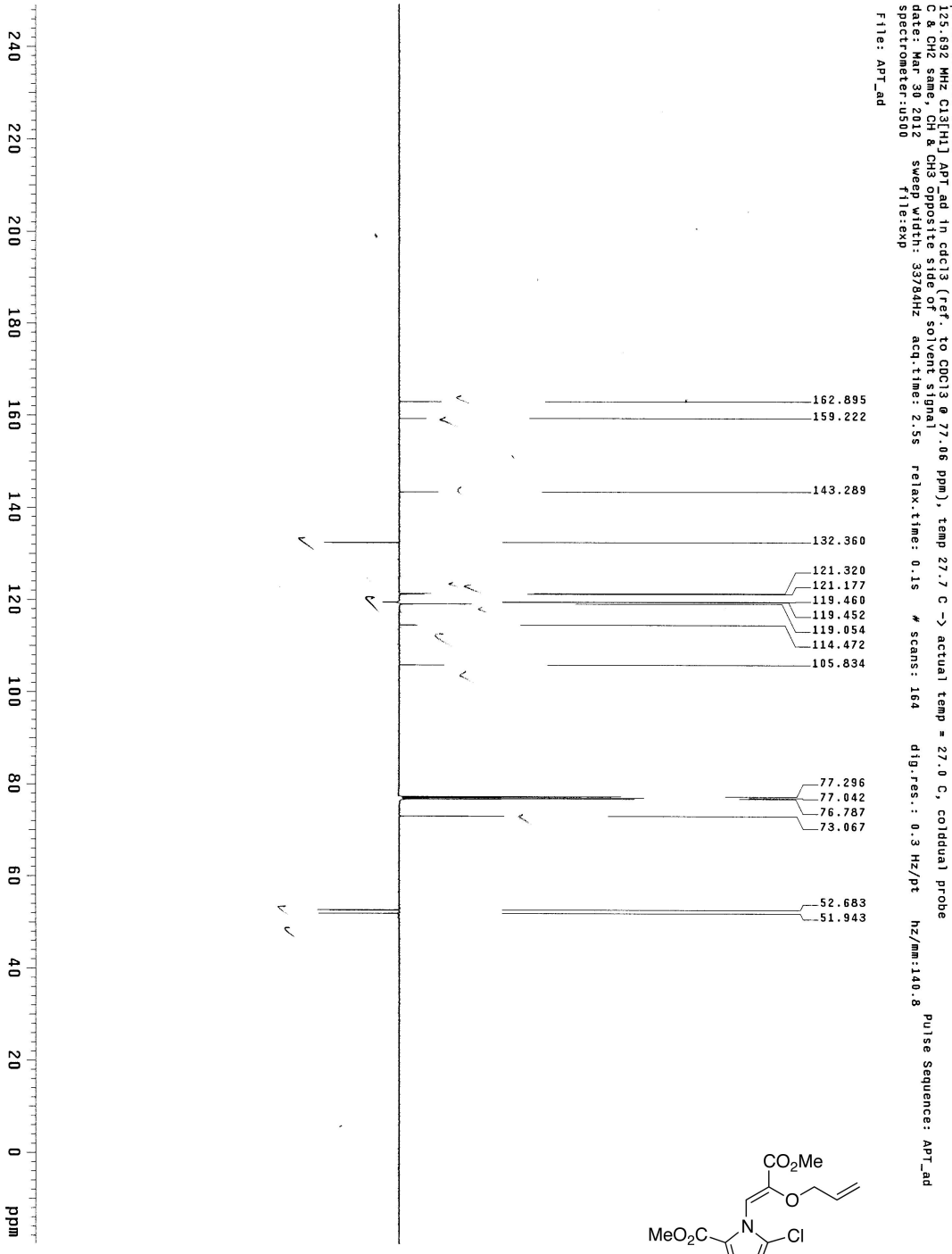
13



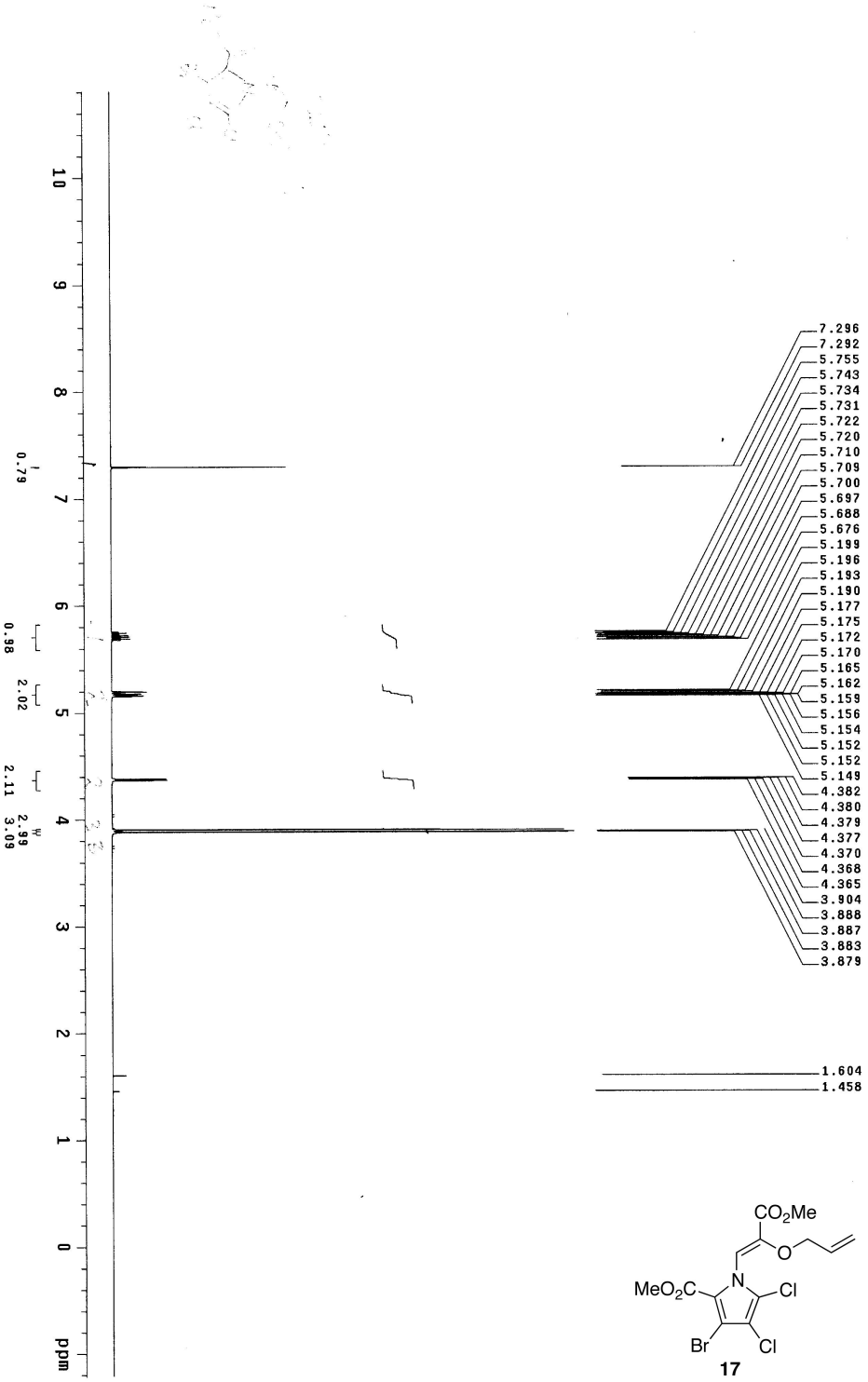


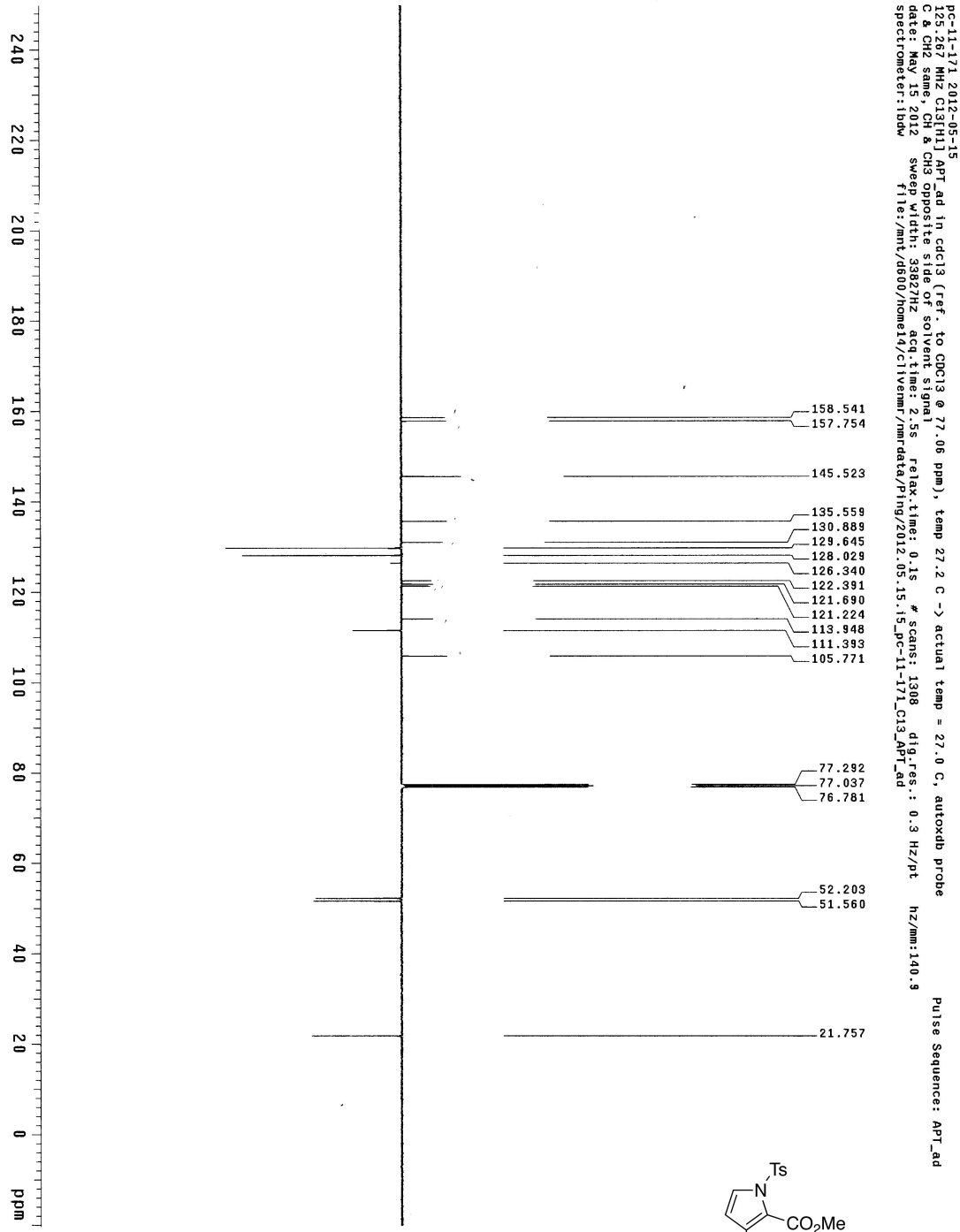


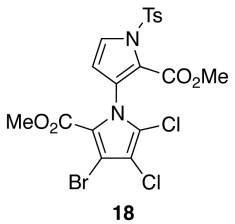



pc-11-97 2012-03-30
125.692 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Mar 30 2012 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 164 dig.res.: 0.3 Hz/pt hz/mm:140.8 Pulse Sequence: APT_ad
spectrometer:u500 file:exp
File: APT_ad
162.895
159.222
143.289
132.360
121.320
121.177
119.460
119.452
119.054
114.472
105.834
77.296
77.042
76.787
73.067
52.683
51.943
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm
CO2Me
O
N
MeO2C
Cl
Br
Cl

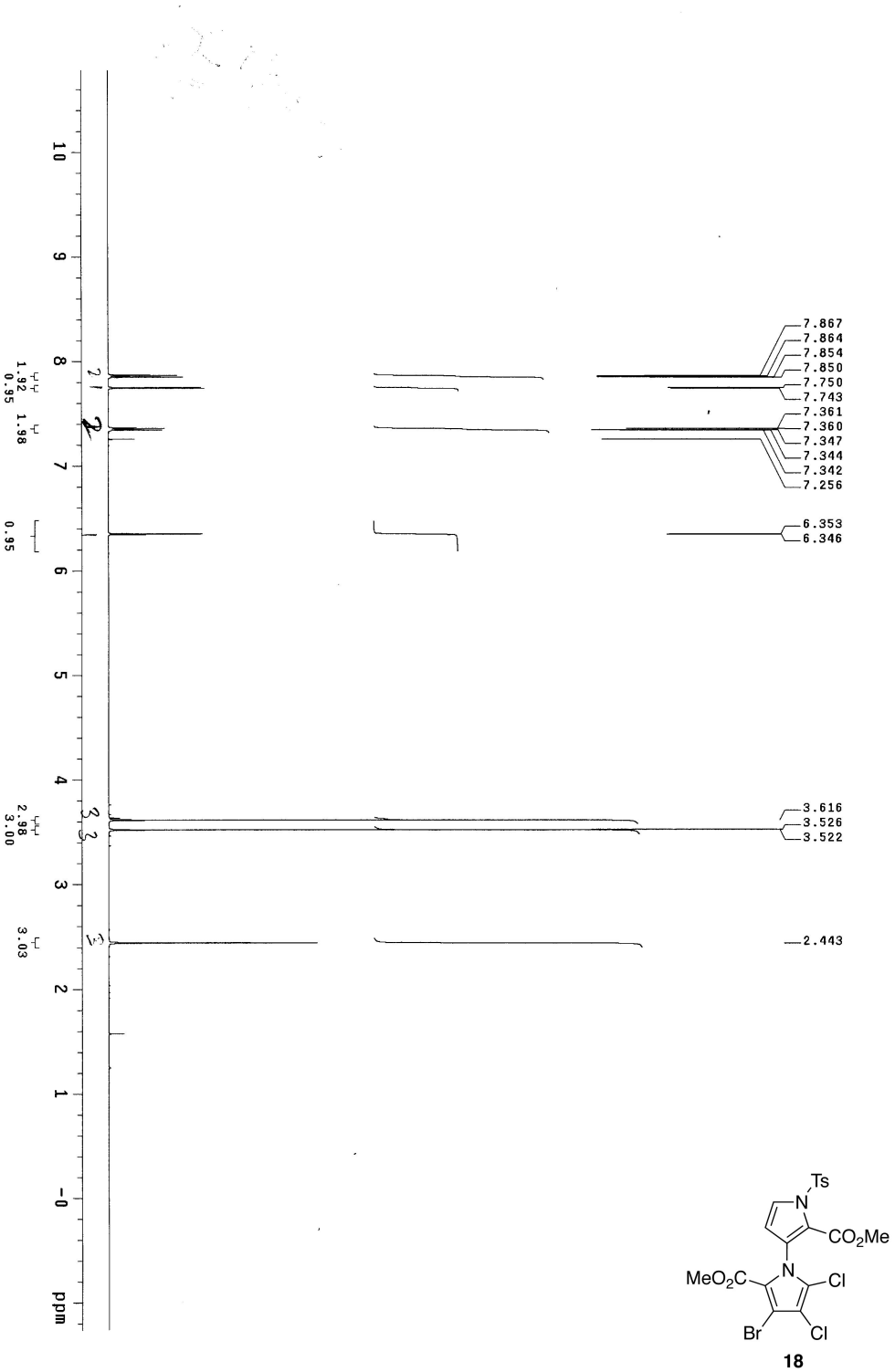
17

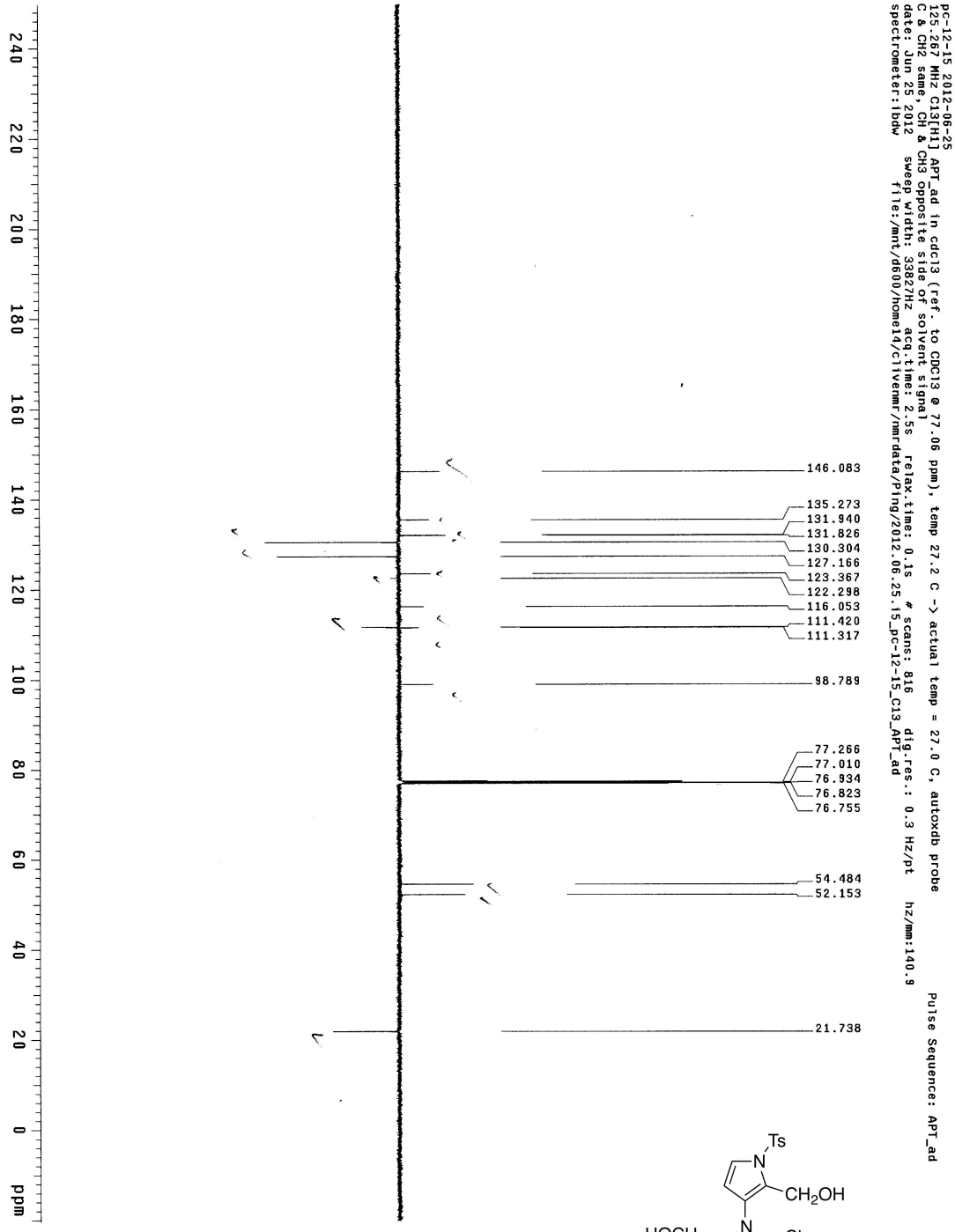




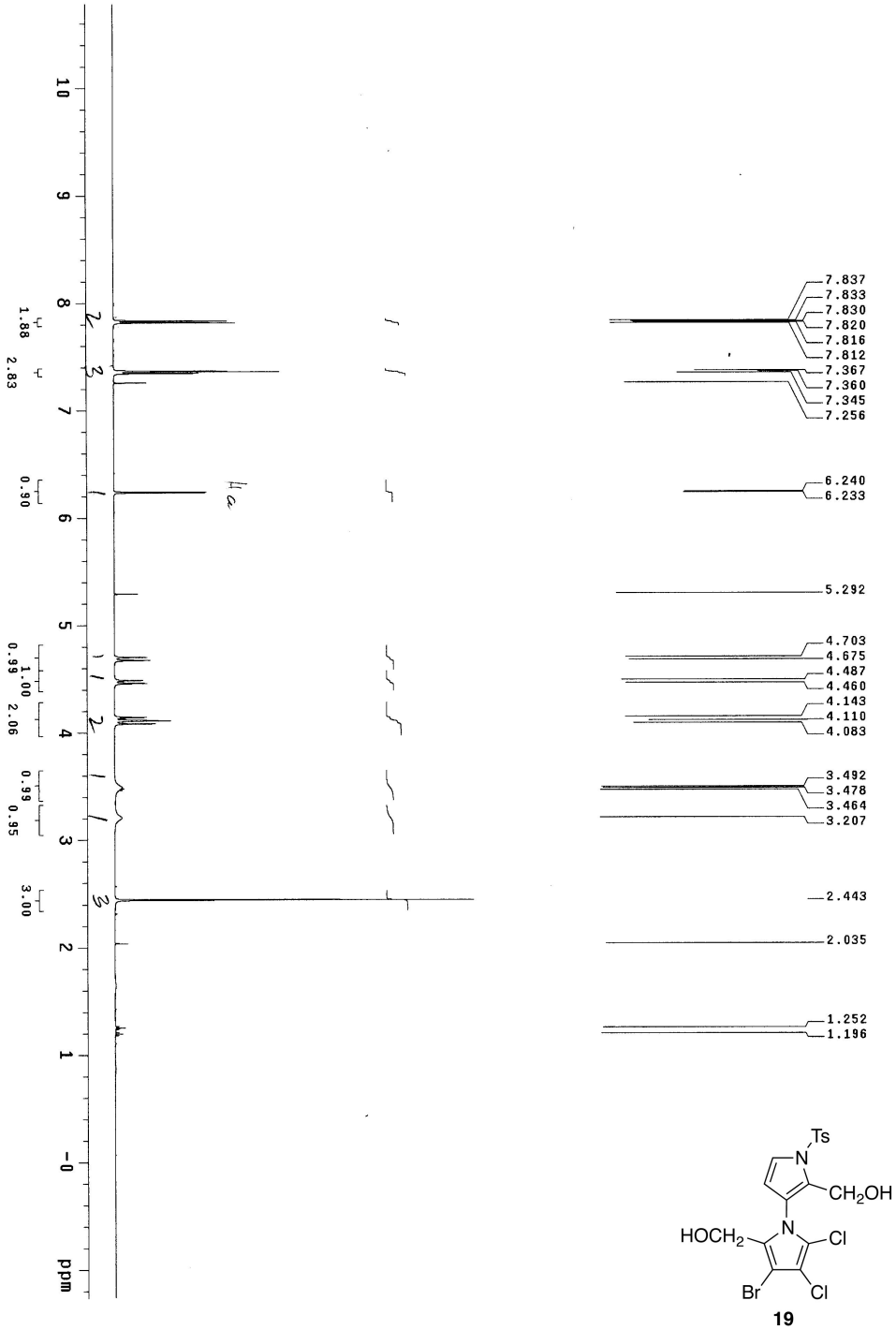


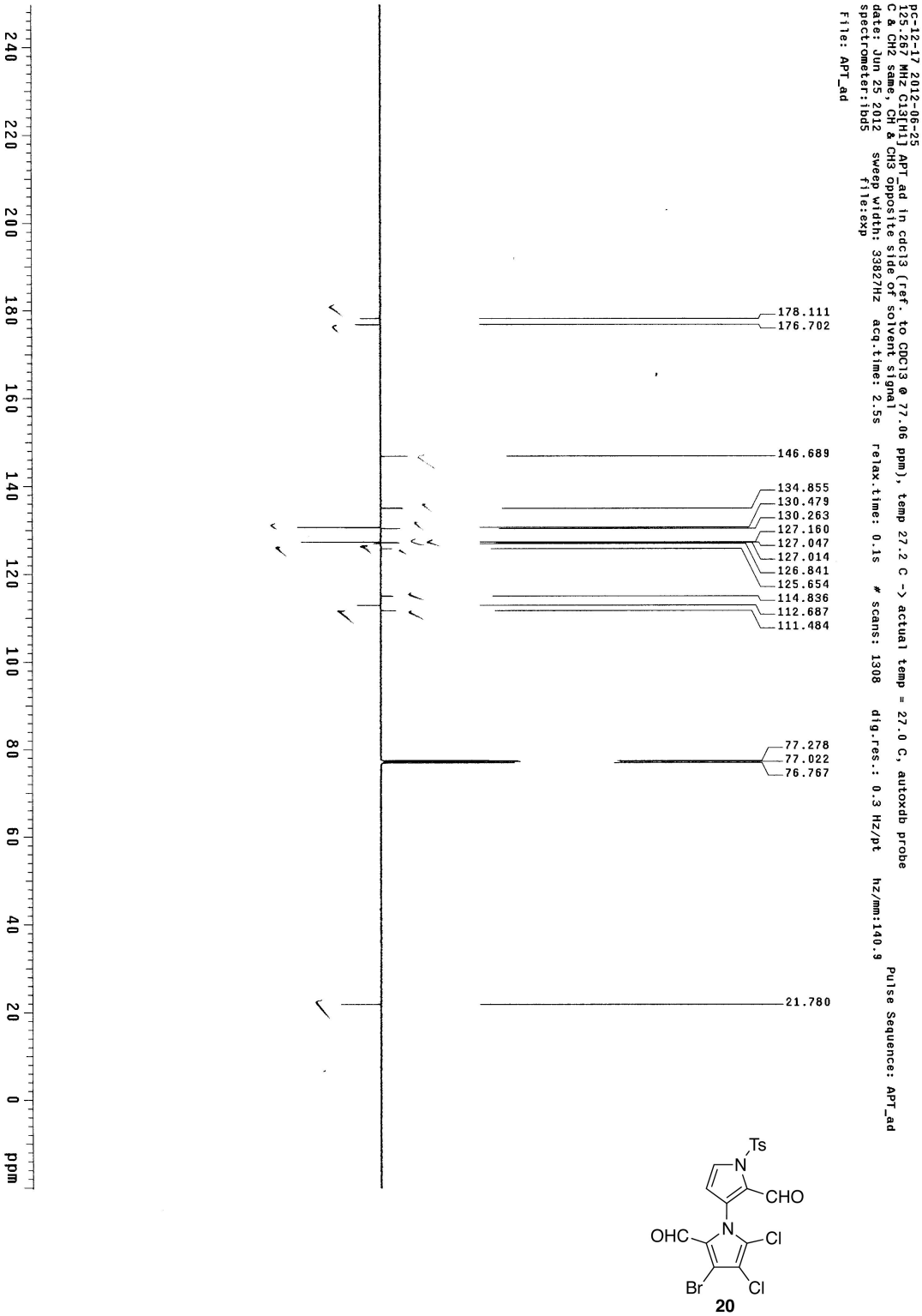
18



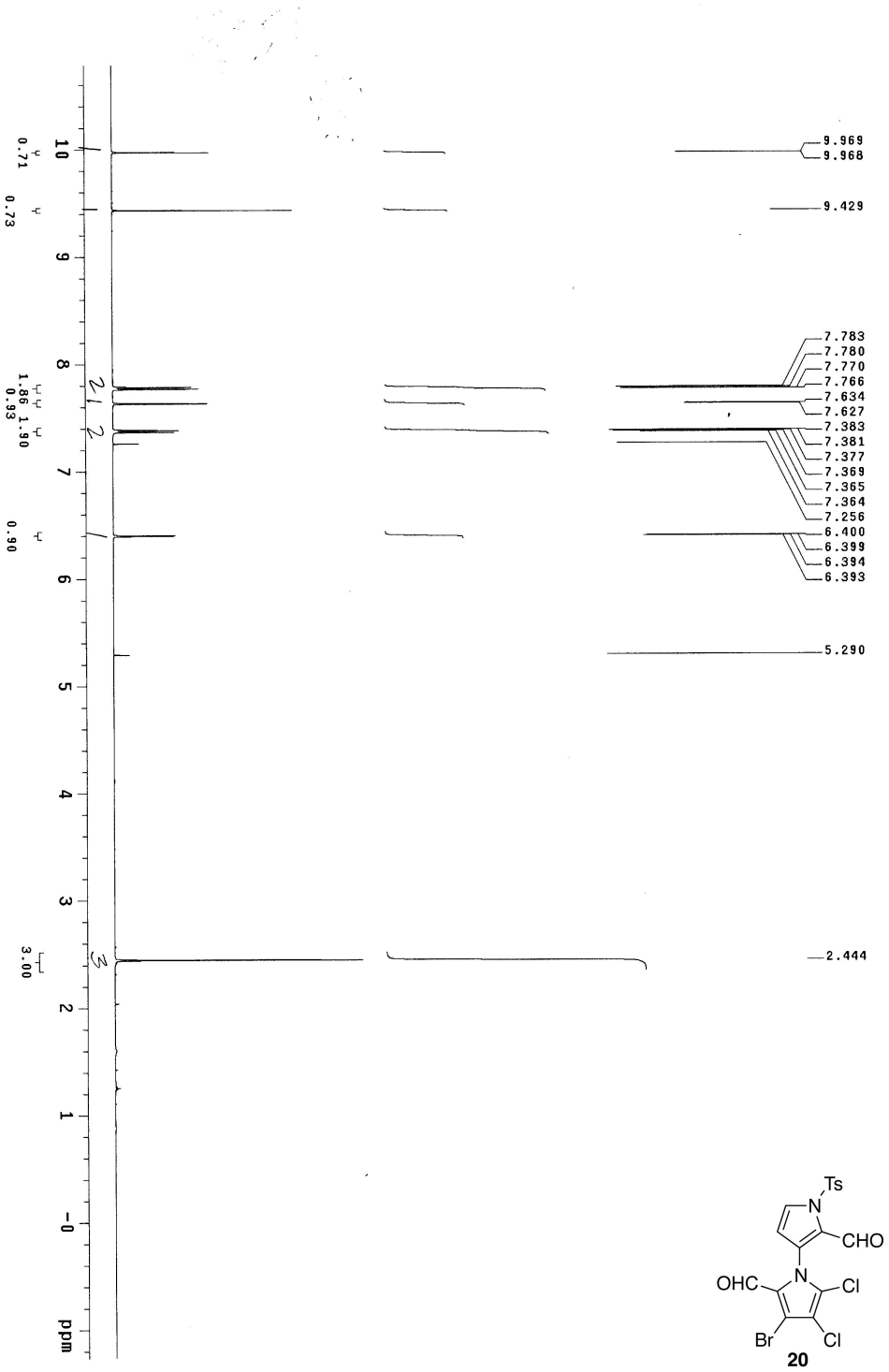


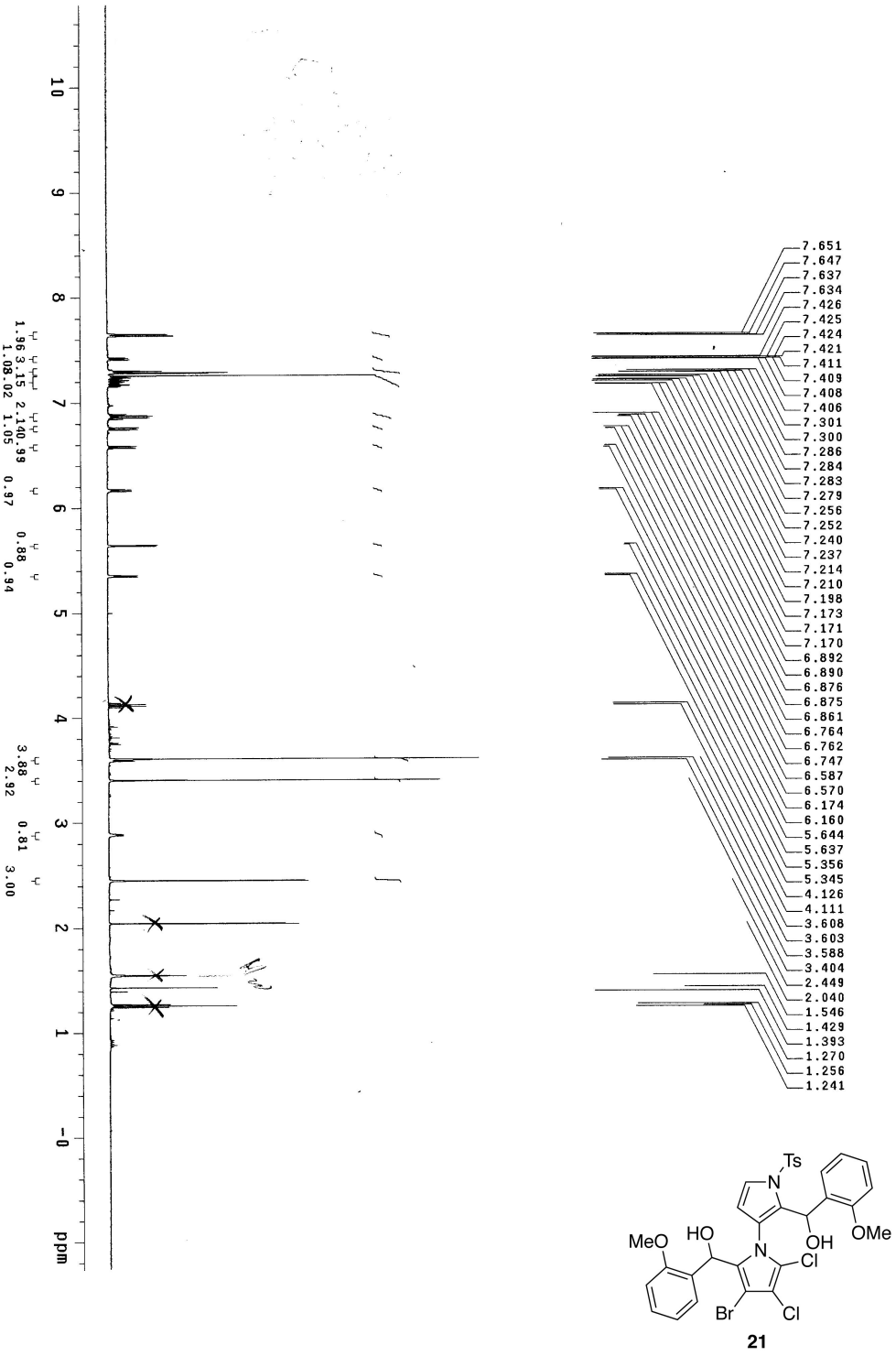
19

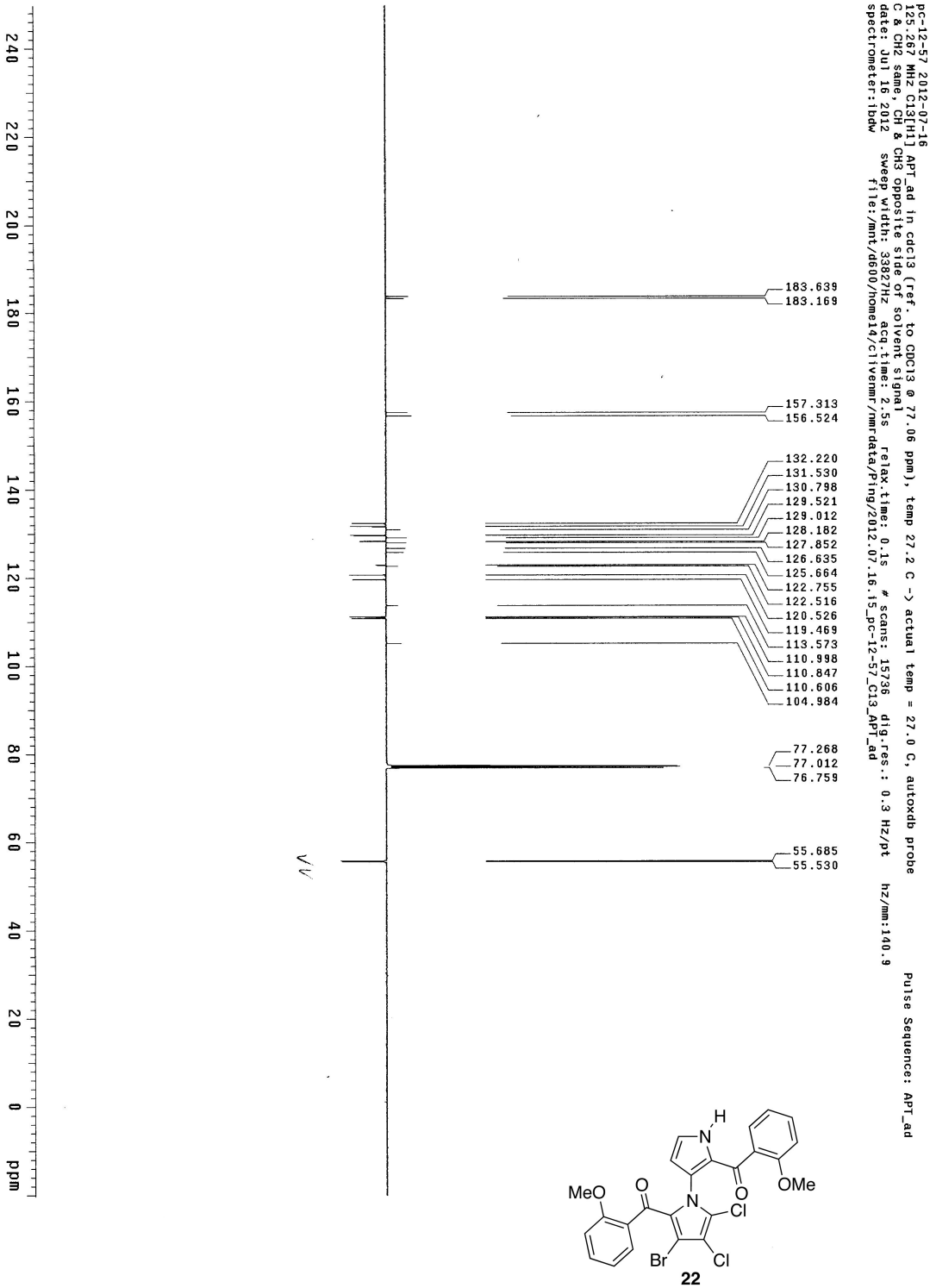




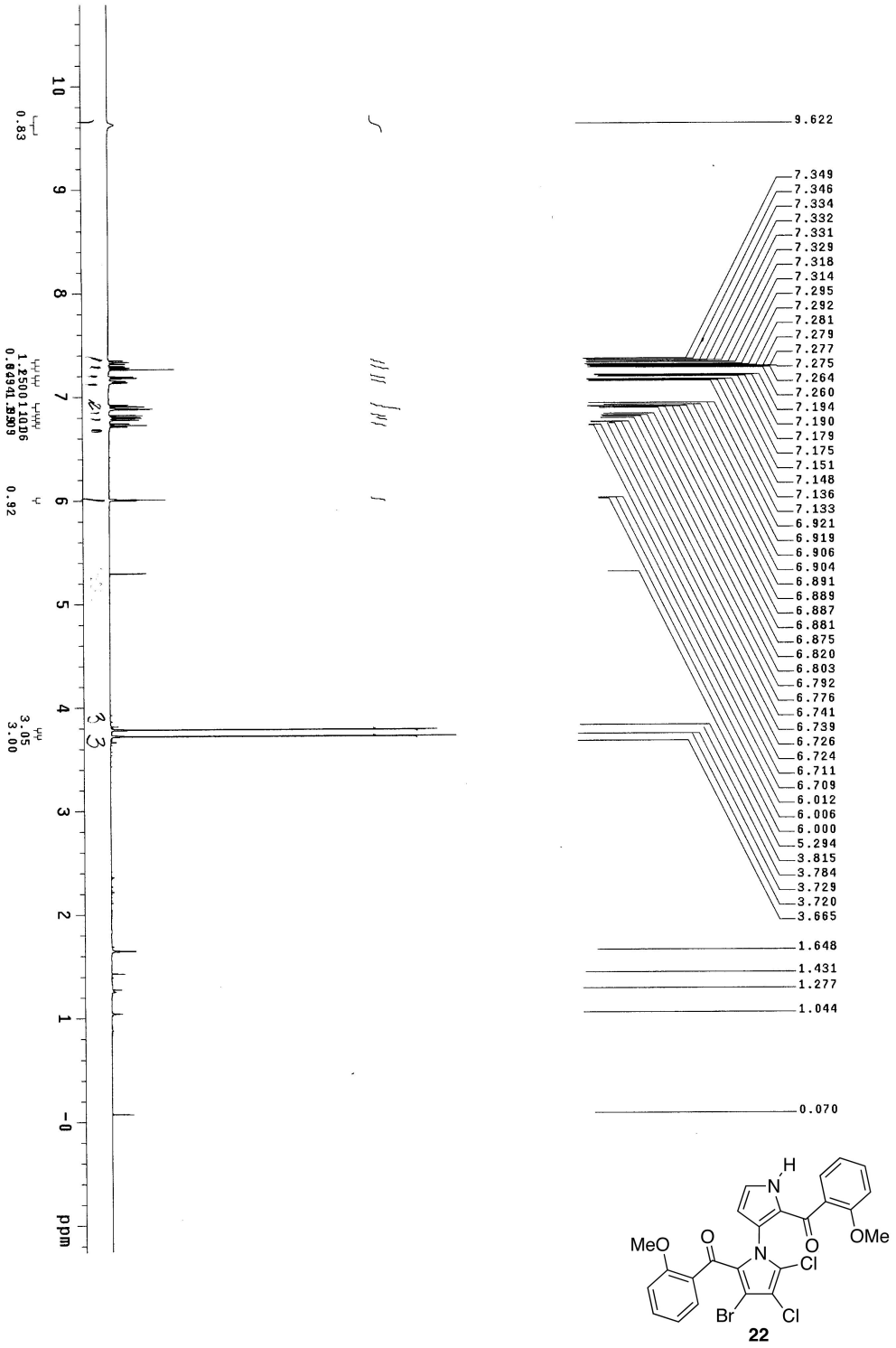
20

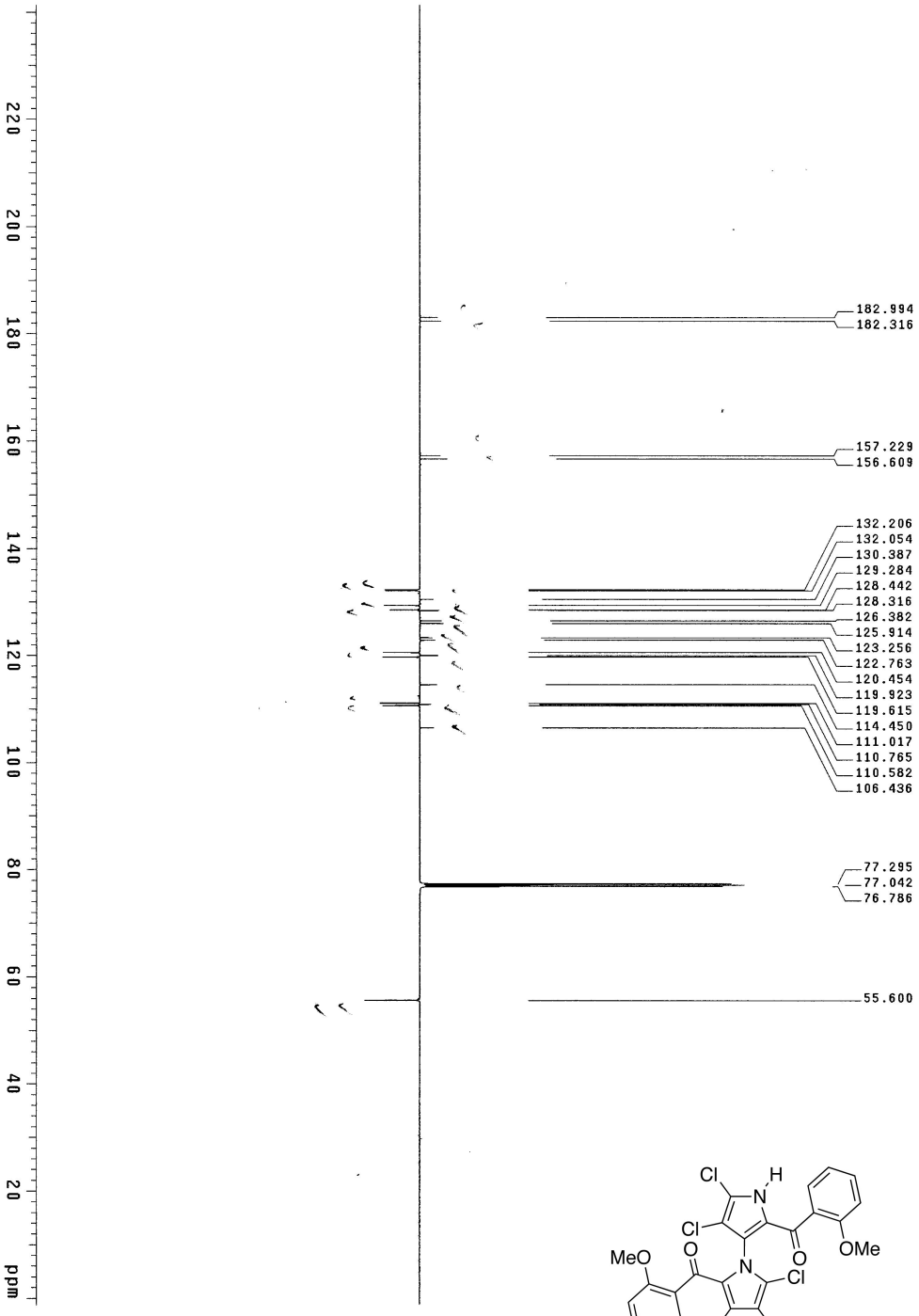


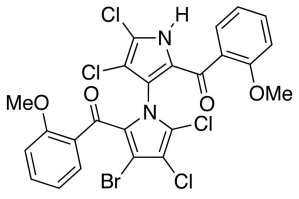

498.122 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
Pulse Sequence: s2pul
7.651
7.647
7.637
7.634
7.426
7.425
7.424
7.421
7.411
7.409
7.408
7.406
7.301
7.300
7.286
7.284
7.283
7.279
7.256
7.252
7.240
7.237
7.214
7.210
7.198
7.173
7.171
7.170
6.892
6.890
6.876
6.875
6.861
6.764
6.762
6.747
6.587
6.570
6.174
6.160
5.644
5.637
5.356
5.345
4.126
4.111
3.608
3.603
3.588
3.404
2.449
2.040
1.546
1.429
1.393
1.270
1.256
1.241
1.96
3.15
2.14
0.99
1.08
0.02
1.05
0.97
0.88
0.94
3.88
2.92
0.81
3.00
ppm
Ts
N
MeO
HO
OH
OMe
N
Cl
Br
Cl
21



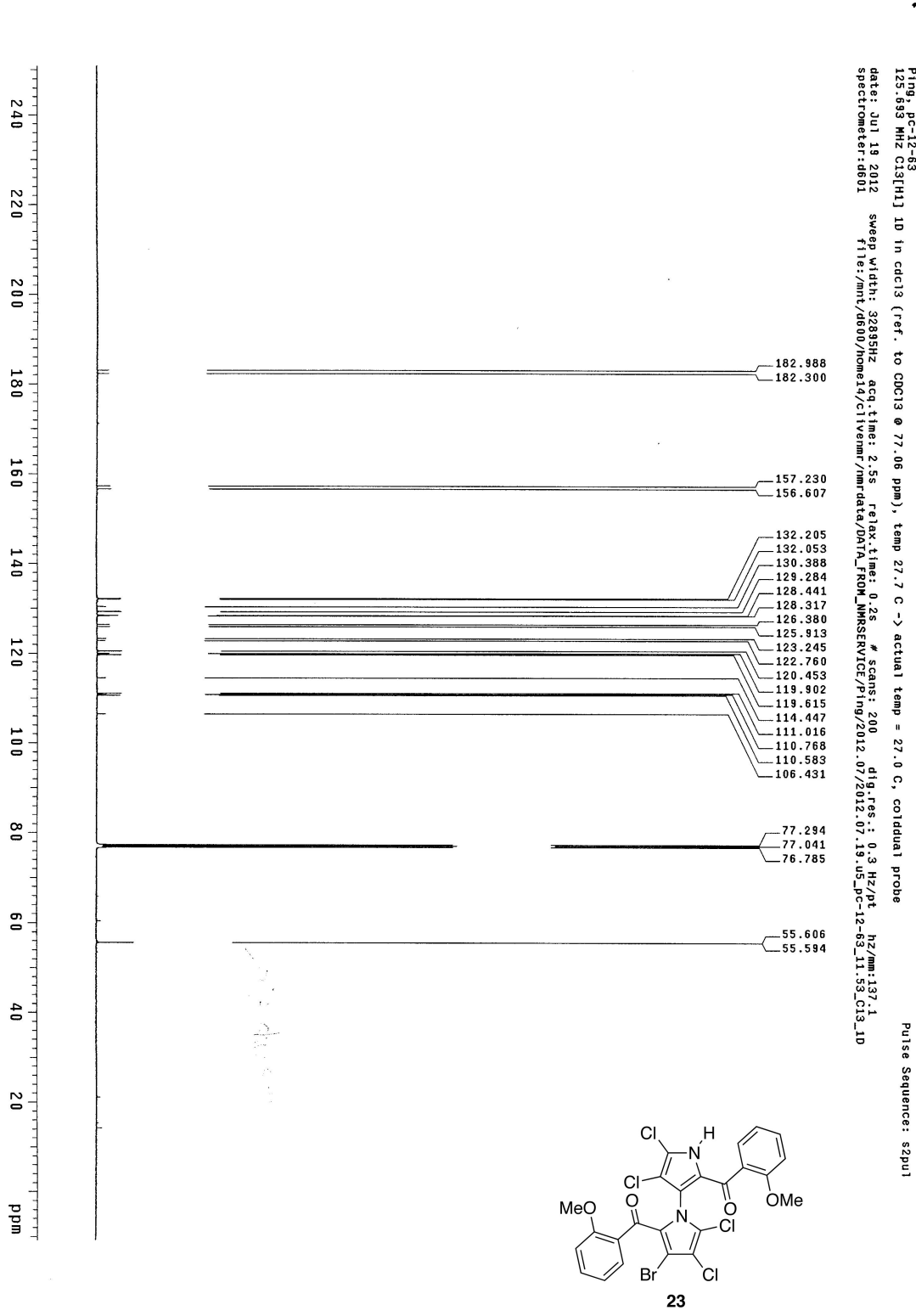
22


pc-12-57 2012-07-16
498.122 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
Pulse Sequence: s2pul
9.622
7.349
7.346
7.334
7.332
7.331
7.329
7.318
7.314
7.295
7.292
7.281
7.279
7.277
7.275
7.264
7.260
7.194
7.190
7.179
7.175
7.151
7.148
7.136
7.133
6.921
6.919
6.906
6.904
6.891
6.889
6.887
6.881
6.875
6.820
6.803
6.792
6.776
6.741
6.739
6.726
6.724
6.711
6.709
6.012
6.006
6.000
5.294
3.815
3.784
3.729
3.720
3.665
1.648
1.431
1.277
1.044
0.070
0.83
0.92
3.05
3.00
ppm
MeO
OMe
Cl
Br
Cl
H
N
O
22


Ping, pc-12-63
125.693 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Jul 21 2012 sweep width: 30488HZ acq.time: 2.5s relax.time: 0.1s # scans: 1000 dig.res.: 0.2 Hz/pt hz/mm:127.0
spectrometer:ibdw file:/mnt/d600/home14/civnmr/nmrdata/DATA_FROM_NMRSERVICE/Ping/2012.07/2012.07.21.u5_pc-12-63_15.27_C13_APT_ad
Pulse Sequence: APT_ad
182.994
182.316
157.229
156.609
132.206
132.054
130.387
129.284
128.442
128.316
126.382
125.914
123.256
122.763
120.454
119.923
119.615
114.450
111.017
110.765
110.582
106.436
77.295
77.042
76.786
55.600
220
200
180
160
140
120
100
80
60
40
20
ppm


Cl
H
N
Cl
O
O
OMe
MeO
N
Cl
Br
Cl

23


Ping, pc-12-63
125.693 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
date: Jul 19 2012 sweep width: 32895Hz acq.time: 2.5s relax.time: 0.2s # scans: 200 dig.res.: 0.3 Hz/pt hz/mm:137.1
spectrometer:d601 file:/mnt/d600/home14/clivenmr/nmrdata/DATA_FROM_NMRSERVICE/Ping/2012.07/2012.07.19.u5_pc-12-63_11.53_C13_1D
Pulse Sequence: s2pul
182.988
182.300
157.230
156.607
132.205
132.053
130.388
129.284
128.441
128.317
126.380
125.913
123.245
122.760
120.453
119.902
119.615
114.447
111.016
110.768
110.583
106.431
77.294
77.041
76.785
55.606
55.594
240
220
200
180
160
140
120
100
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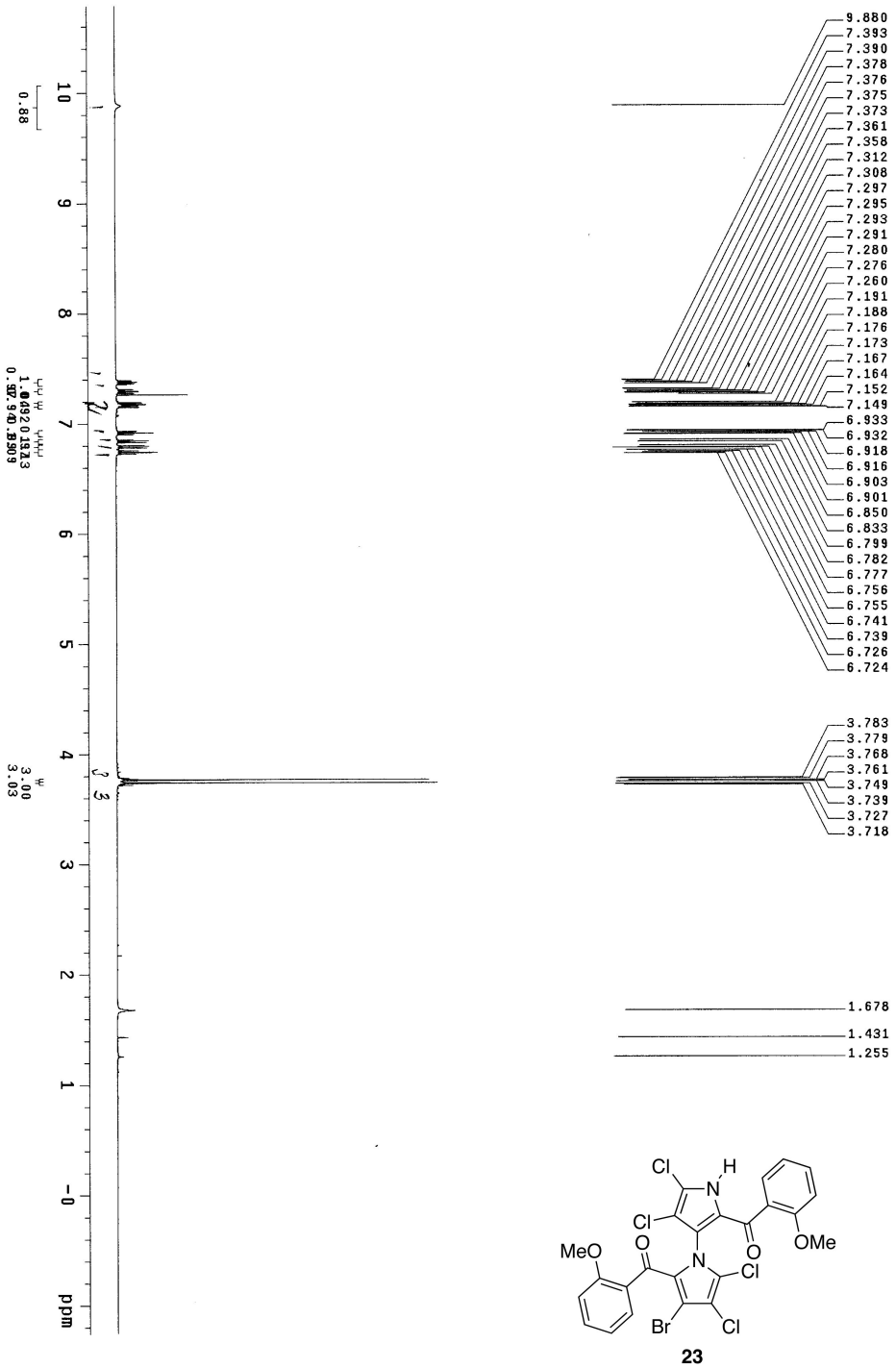
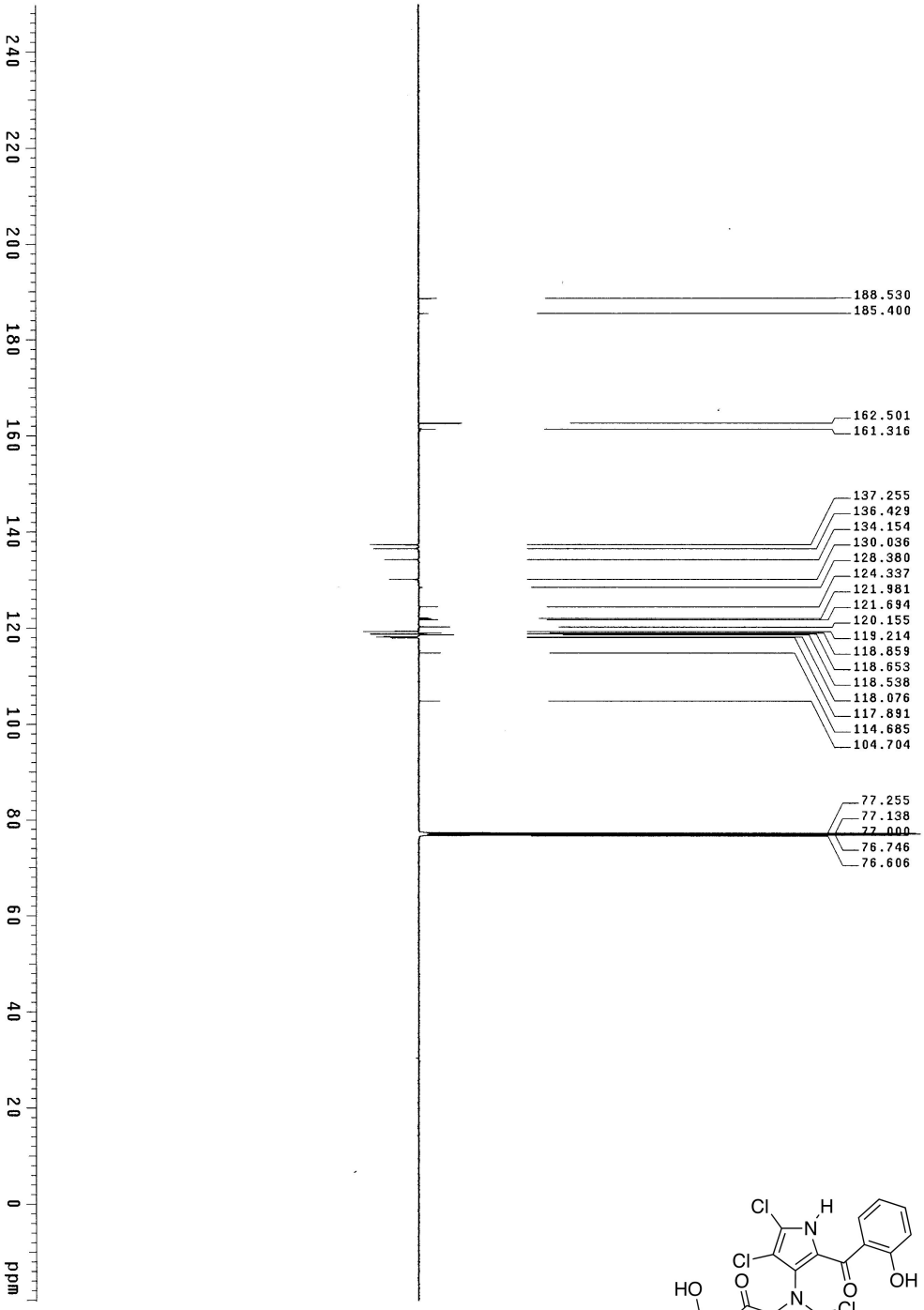
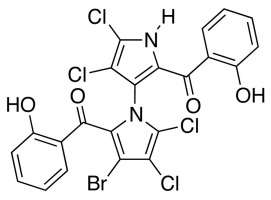
O
OMe
MeO
O
N
Cl
Br
Cl
23

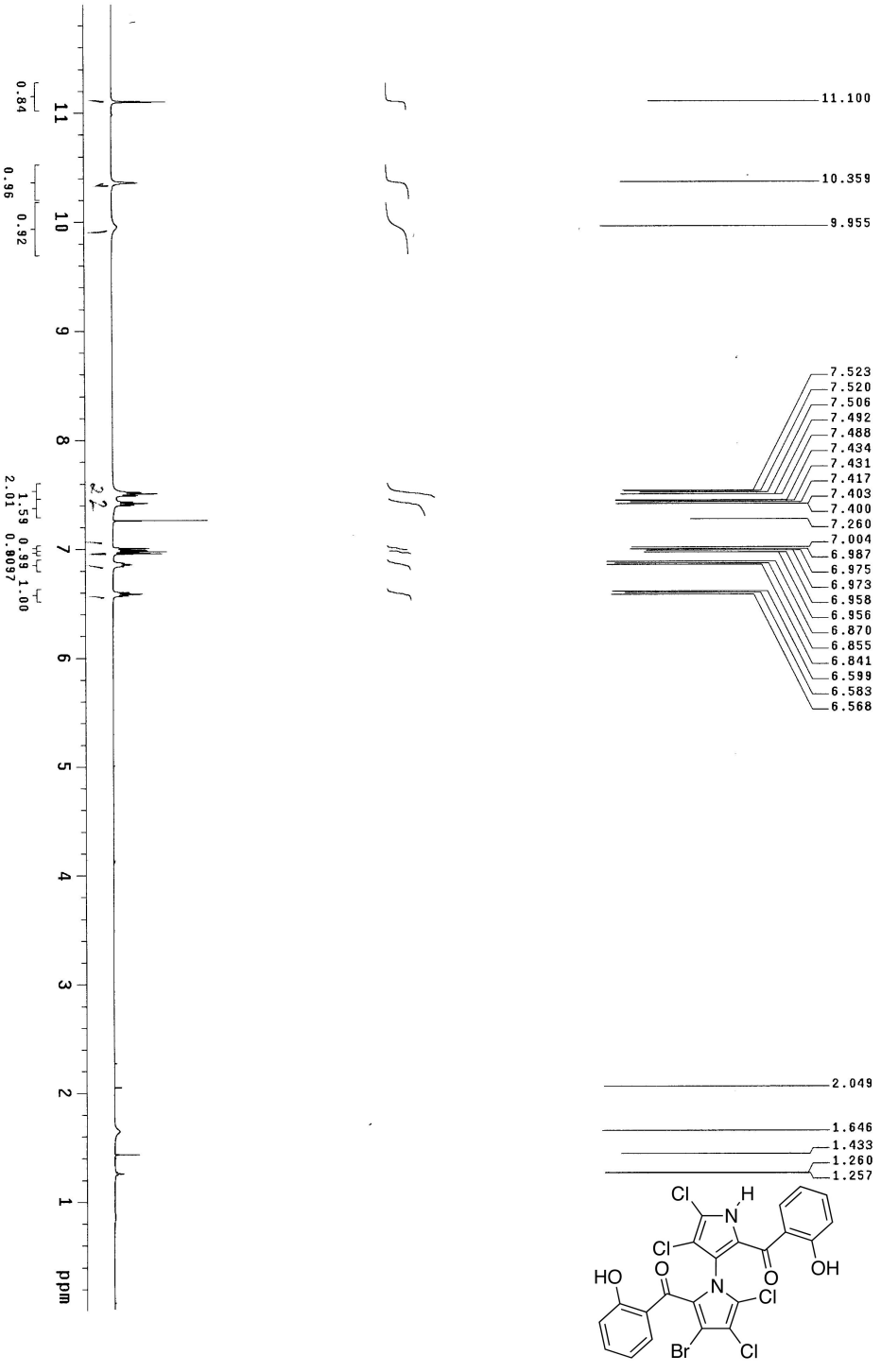


23

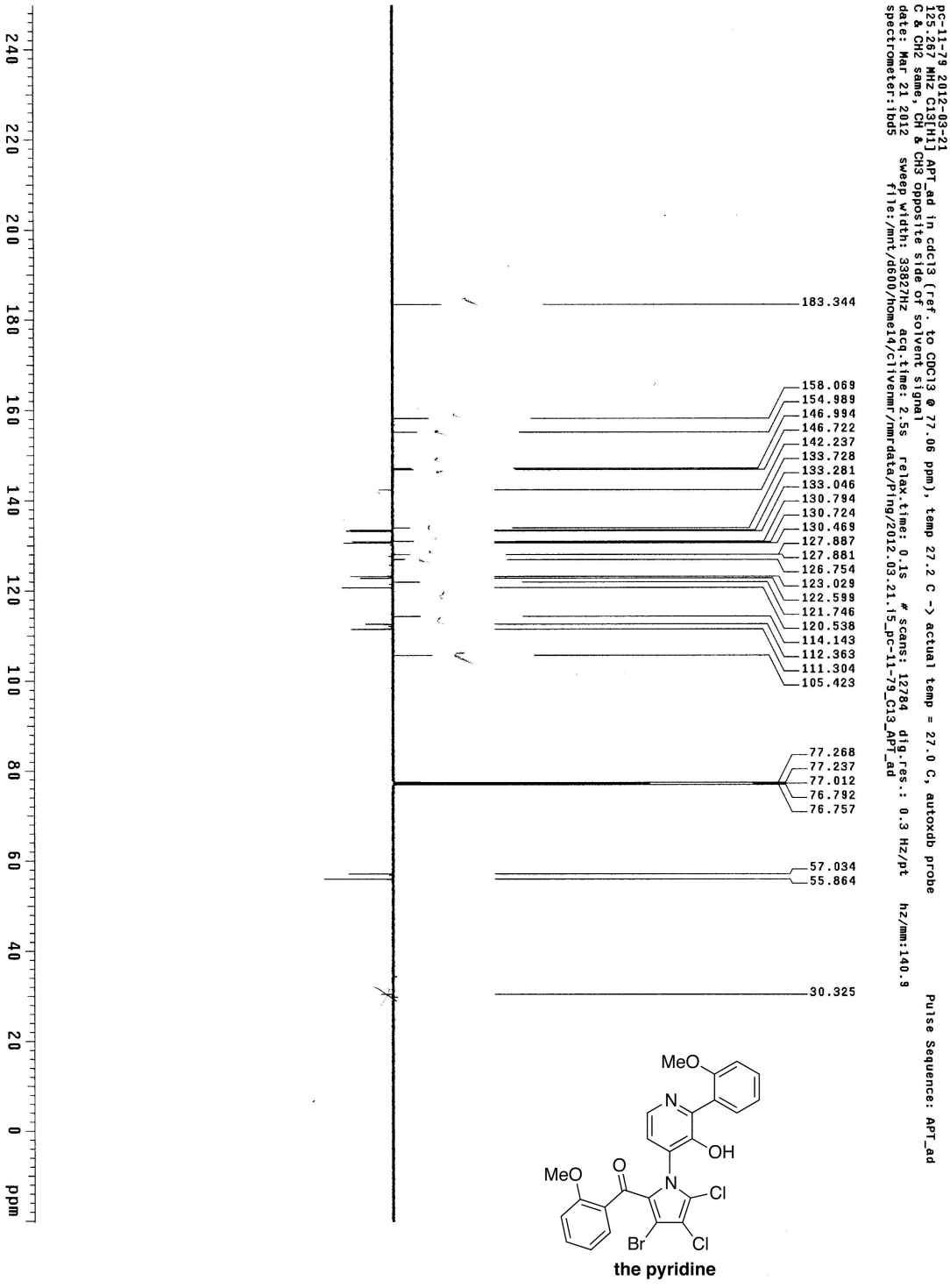




(±)-marinopyrrole B



(±)-marinopyrrole B



the pyridine