Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.


# Characterisation data of compounds for

# Total Synthesis of Lycorine-type Alkaloids by Cycloproply Ring-Opening Rearrangement

Dandan Liu [a, b], Long Ai [a], Fan Li [b], Annan Zhao [a], Jingbo Chen [a], Hongbin Zhang [a] and Jianping Liu*[a]

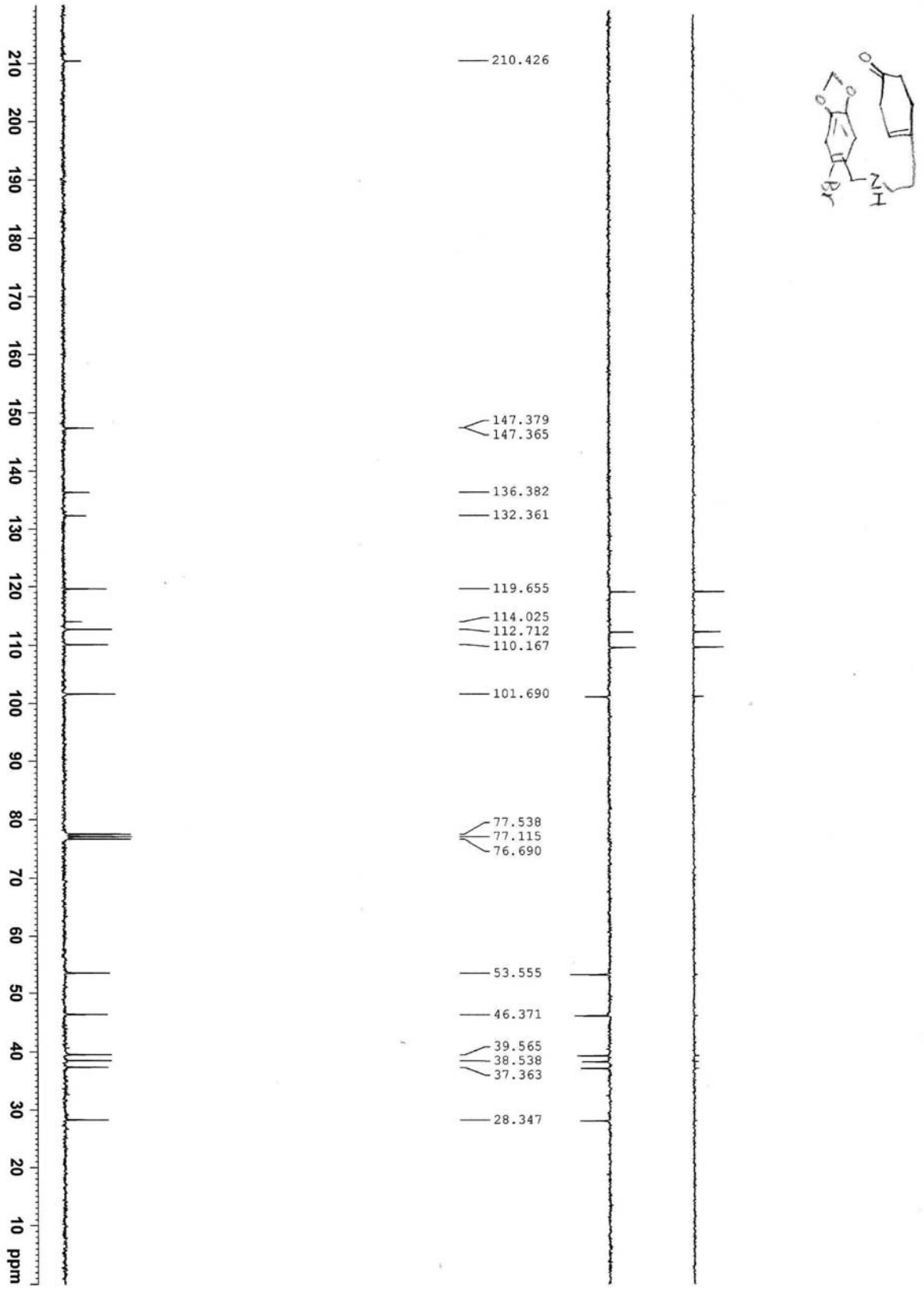
[a] Key Laboratory of Medicinal Chemistry for Natural Resource (Yunnan University), Ministry of Education, School of Chemical Science and Technology, Yunnan University, Kunming, Yunnan 650091, P. R. China

[b] School of Pharmaceutical Sciences and Yunnan Key Laboratory of Pharmacology for Natural Products, Kunming Medical University, 1168 Western Chunrong Road, Yuhua Street, Chenggong New City, Kunming, Yunnan 650500, P. R. China

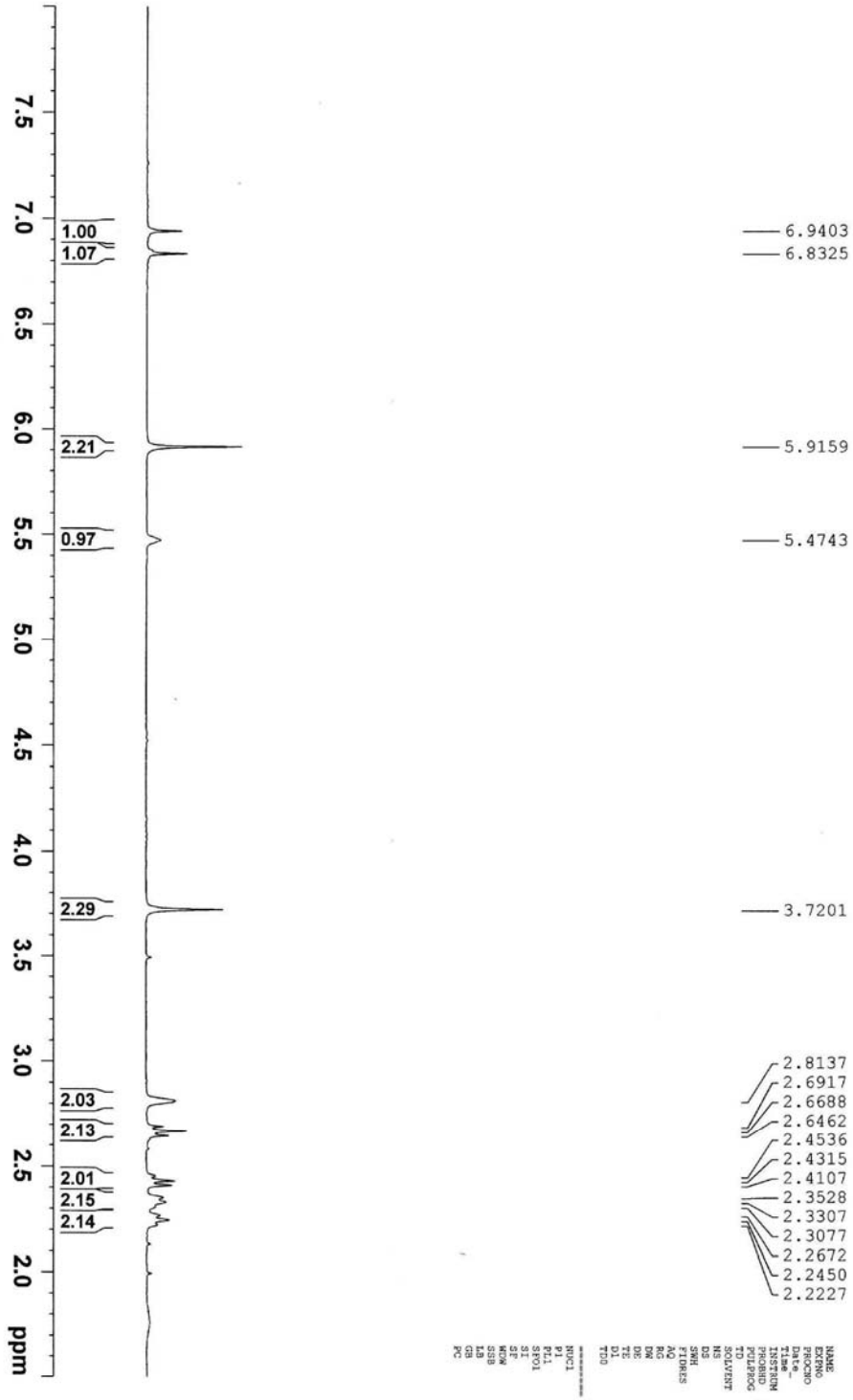
Fax: 86-871-65035538

E-mail: jpliu@ynu.edu.cn

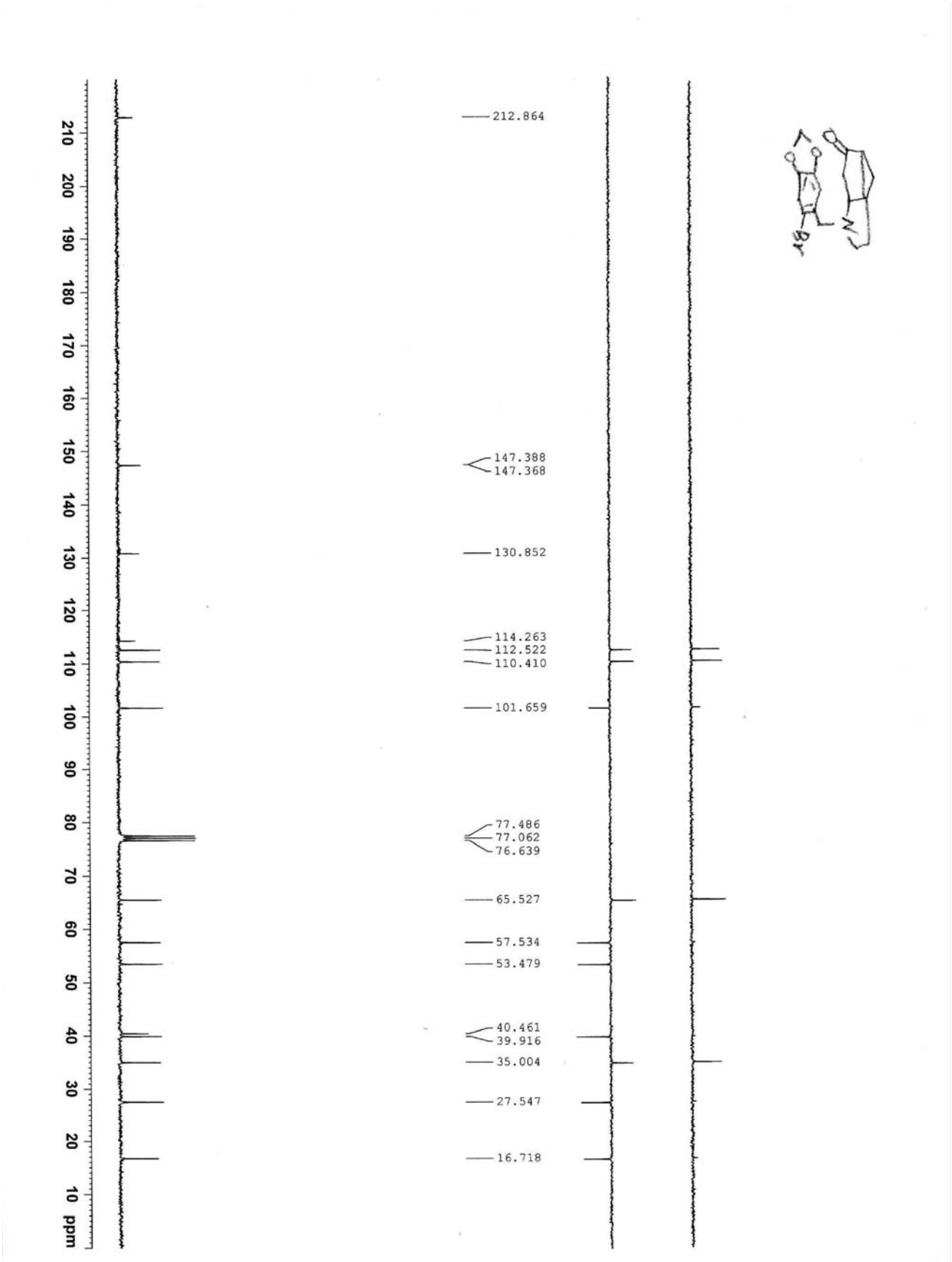
Compound 6


210.426
147.379
147.365
136.382
132.361
119.655
114.025
112.712
110.167
101.690
77.538
77.115
76.690
53.555
46.371
39.565
38.538
37.363
28.347
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
O
O
O
Br
ZnI

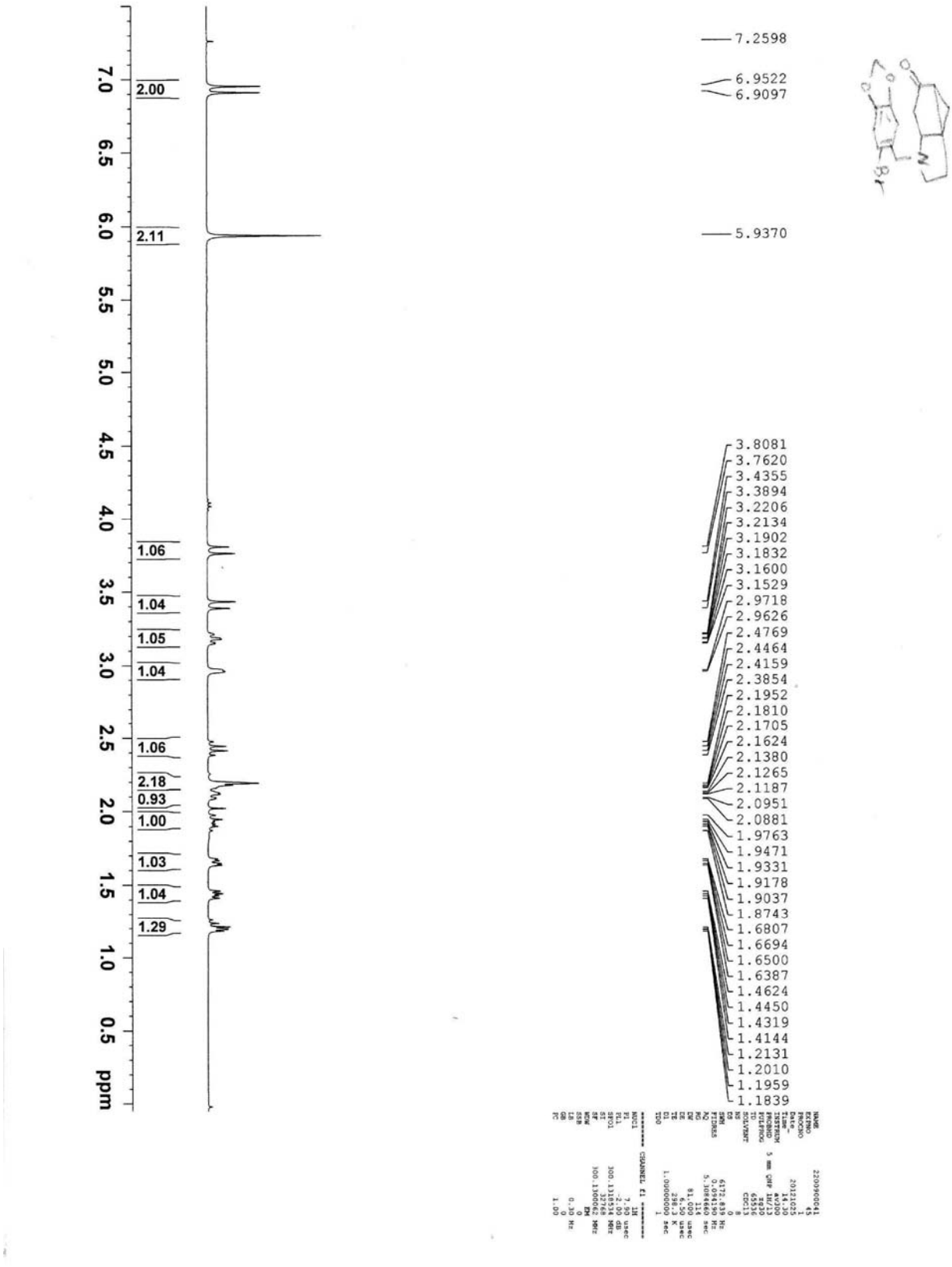
Compound 6


7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
1.00
1.07
2.21
0.97
2.29
2.03
2.13
2.01
2.15
2.14
6.9403
6.8325
5.9159
5.4743
3.7201
2.8137
2.6917
2.6688
2.6462
2.4536
2.4315
2.4107
2.3528
2.3307
2.3077
2.2672
2.2450
2.2227
Br
NH

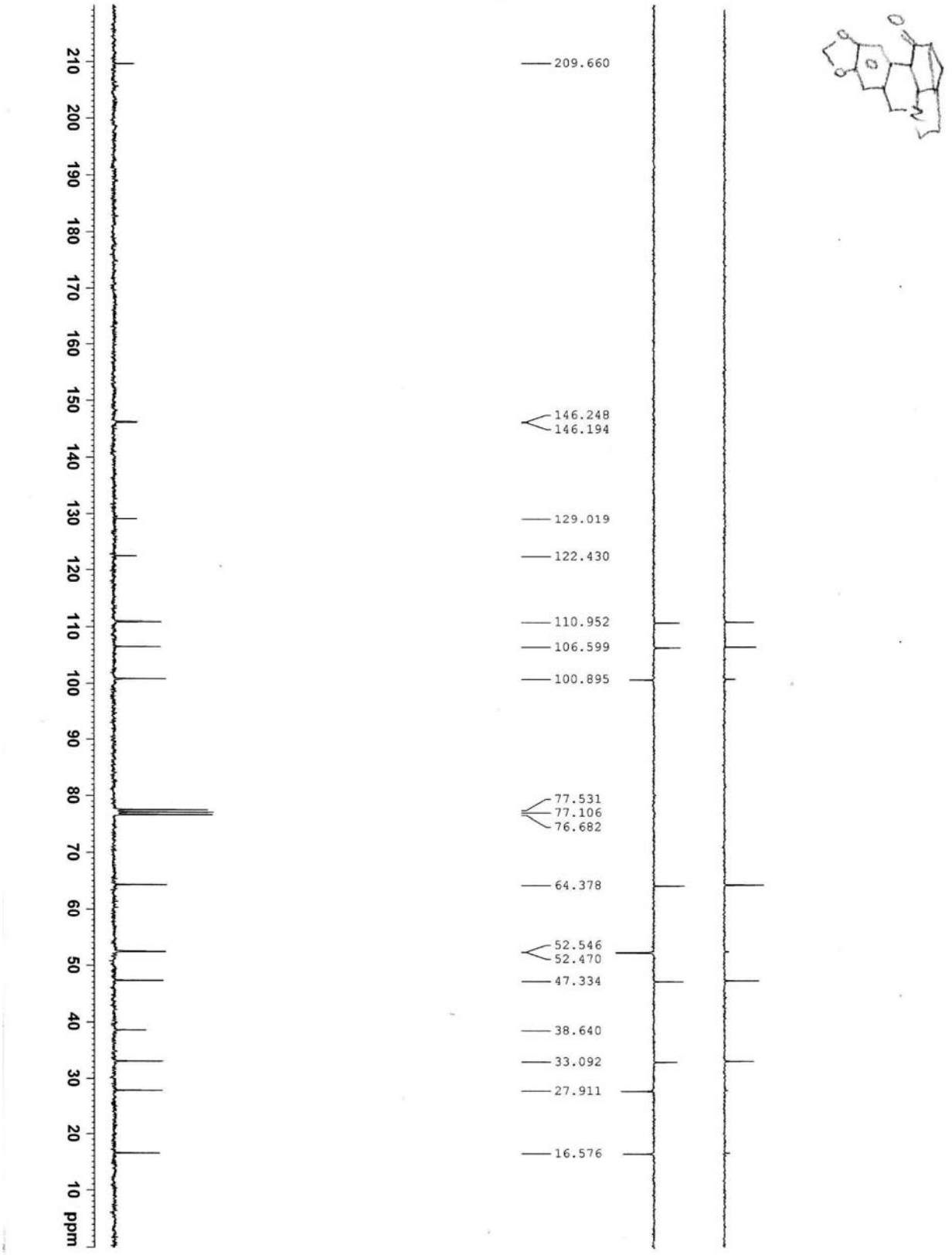
Compound 9


212.864
147.388
147.368
130.852
114.263
112.522
110.410
101.659
77.486
77.062
76.639
65.527
57.534
53.479
40.461
39.916
35.004
27.547
16.718
Br
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

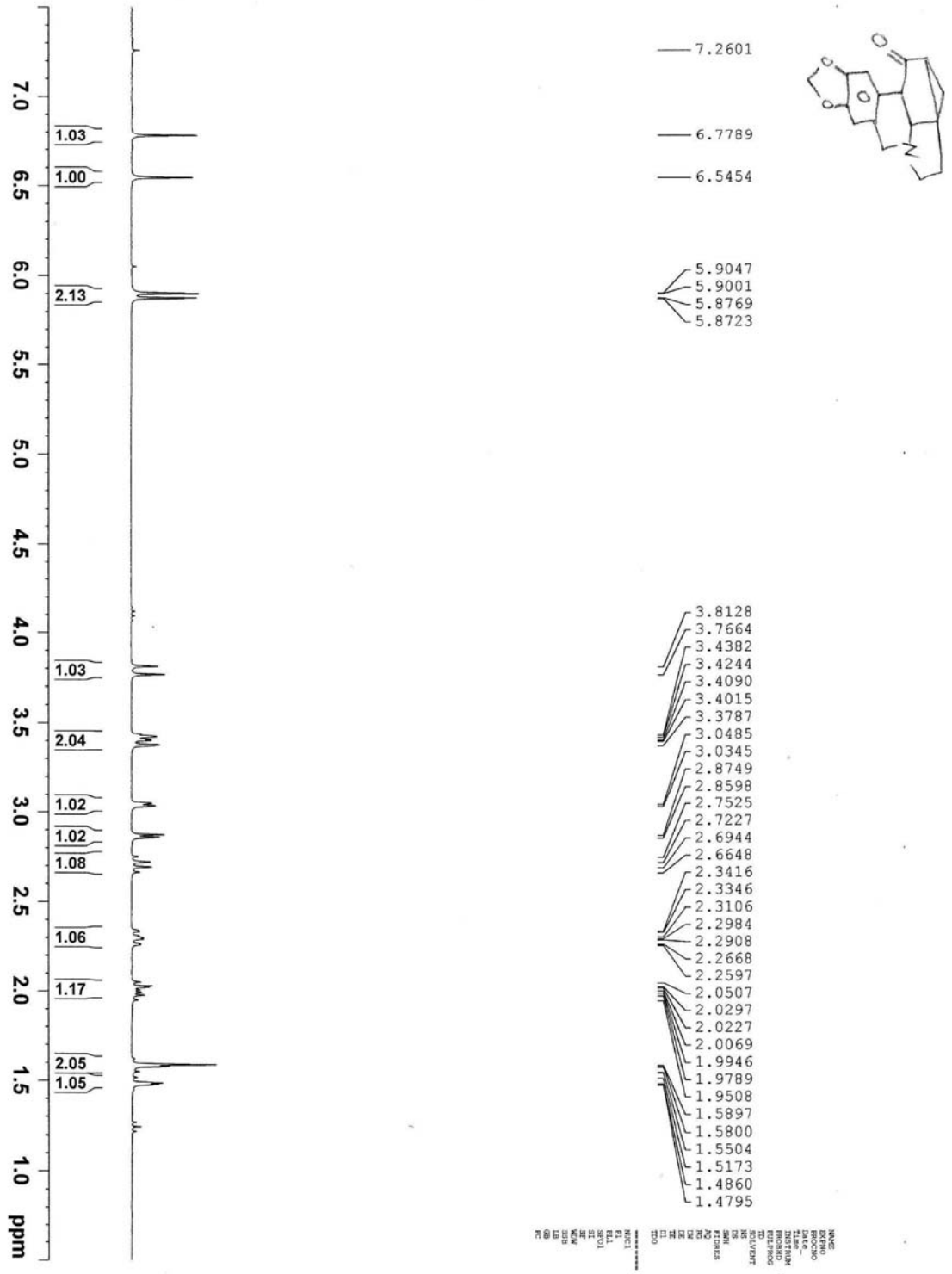
Compound 9


7.2598
6.9522
6.9097
5.9370
3.8081
3.7620
3.4355
3.3894
3.2206
3.2134
3.1902
3.1832
3.1600
3.1529
2.9718
2.9626
2.4769
2.4464
2.4159
2.3854
2.1952
2.1810
2.1705
2.1624
2.1380
2.1265
2.1187
2.0951
2.0881
1.9763
1.9471
1.9331
1.9178
1.9037
1.8743
1.6807
1.6694
1.6500
1.6387
1.4624
1.4450
1.4319
1.4144
1.2131
1.2010
1.1959
1.1839
2.00
2.11
1.06
1.04
1.05
1.04
1.06
2.18
0.93
1.00
1.03
1.04
1.29
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
ppm

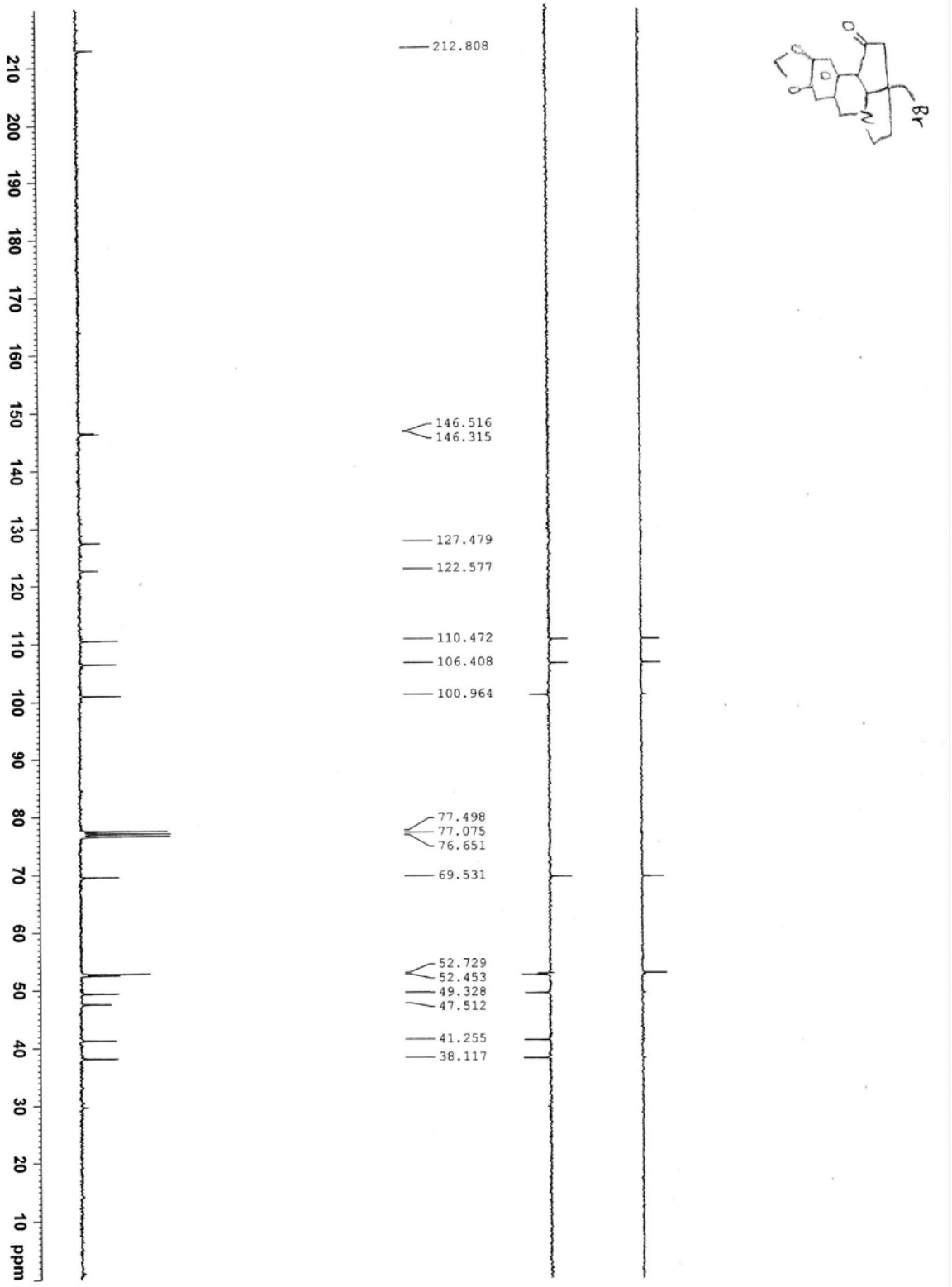
Compound 12


209.660
146.248
146.194
129.019
122.430
110.952
106.599
100.895
77.531
77.106
76.682
64.378
52.546
52.470
47.334
38.640
33.092
27.911
16.576
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

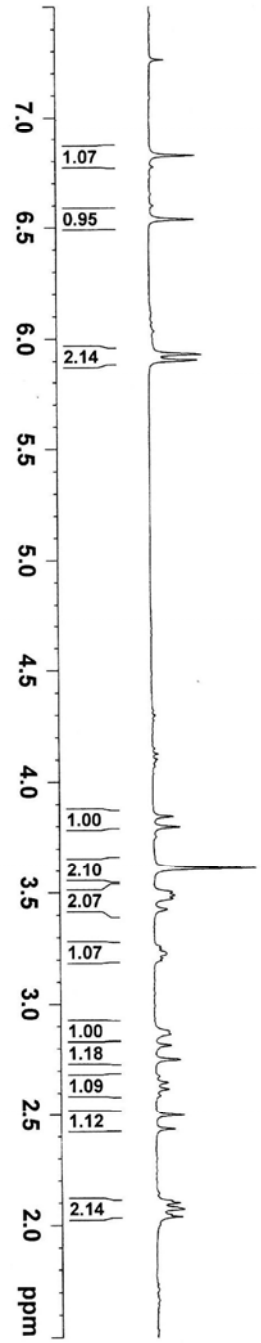
Compound 12

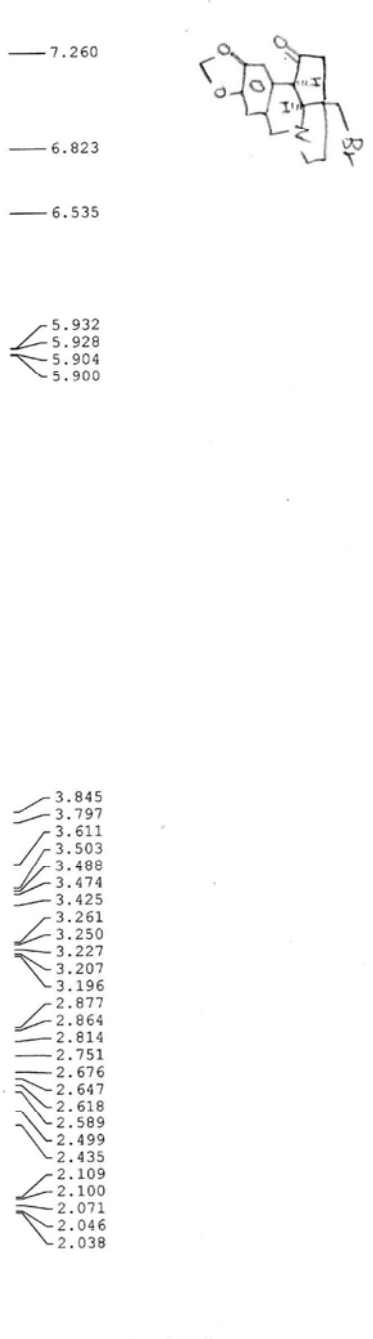

7.2601
6.7789
6.5454
5.9047
5.9001
5.8769
5.8723
3.8128
3.7664
3.4382
3.4244
3.4090
3.4015
3.3787
3.0485
3.0345
2.8749
2.8598
2.7525
2.7227
2.6944
2.6648
2.3416
2.3346
2.3106
2.2984
2.2908
2.2668
2.2597
2.0507
2.0297
2.0227
2.0069
1.9946
1.9789
1.9508
1.5897
1.5800
1.5504
1.5173
1.4860
1.4795
1.03
1.00
2.13
1.03
2.04
1.02
1.02
1.08
1.06
1.17
2.05
1.05
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
ppm

Compound 13

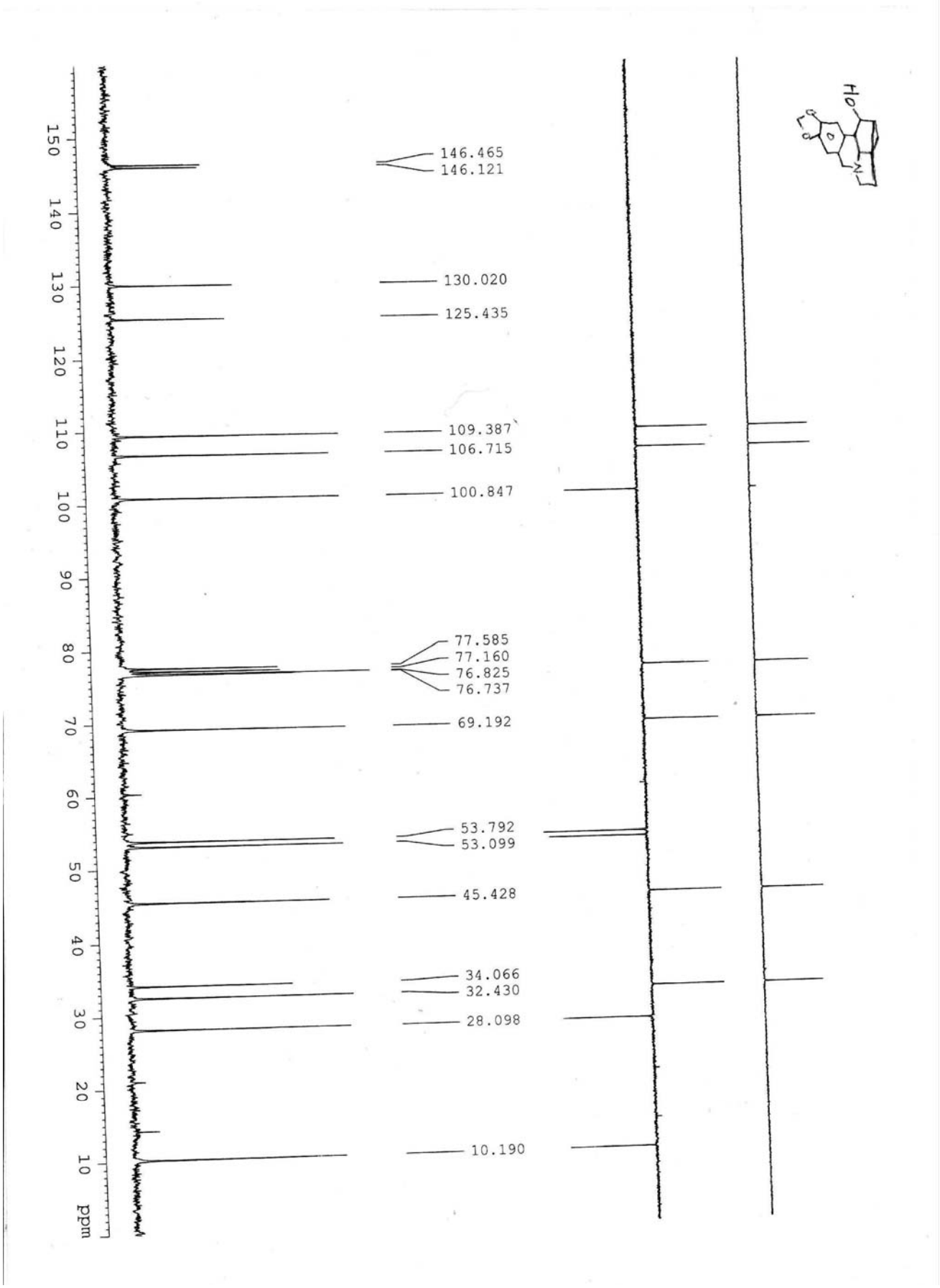

212.808
146.516
146.315
127.479
122.577
110.472
106.408
100.964
77.498
77.075
76.651
69.531
52.729
52.453
49.328
47.512
41.255
38.117
O
O
O
N
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound 13

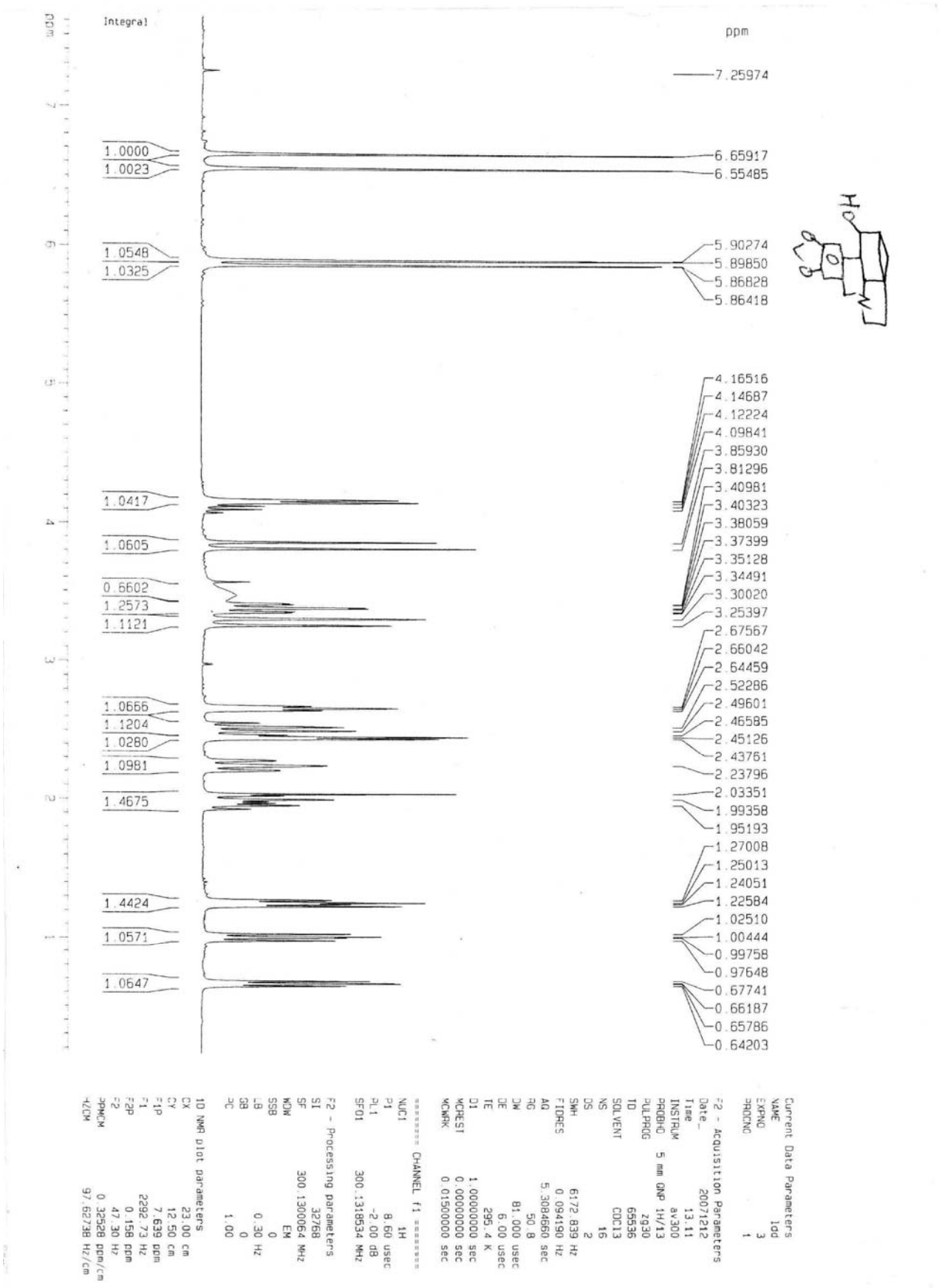

7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
1.07
0.95
2.14
1.00
2.10
2.07
1.07
1.00
1.18
1.09
1.12
2.14


7.260
6.823
6.535
5.932
5.928
5.904
5.900
3.845
3.797
3.611
3.503
3.488
3.474
3.425
3.261
3.250
3.227
3.207
3.196
2.877
2.864
2.814
2.751
2.676
2.647
2.618
2.589
2.499
2.435
2.109
2.100
2.071
2.046
2.038
Br

Compound 14


146.465
146.121
130.020
125.435
109.387
106.715
100.847
77.585
77.160
76.825
76.737
69.192
53.792
53.099
45.428
34.066
32.430
28.098
10.190
ppm

Compound 14





Current Data Parameters
NAME 1od
EXPNO 3
PROCNO 1

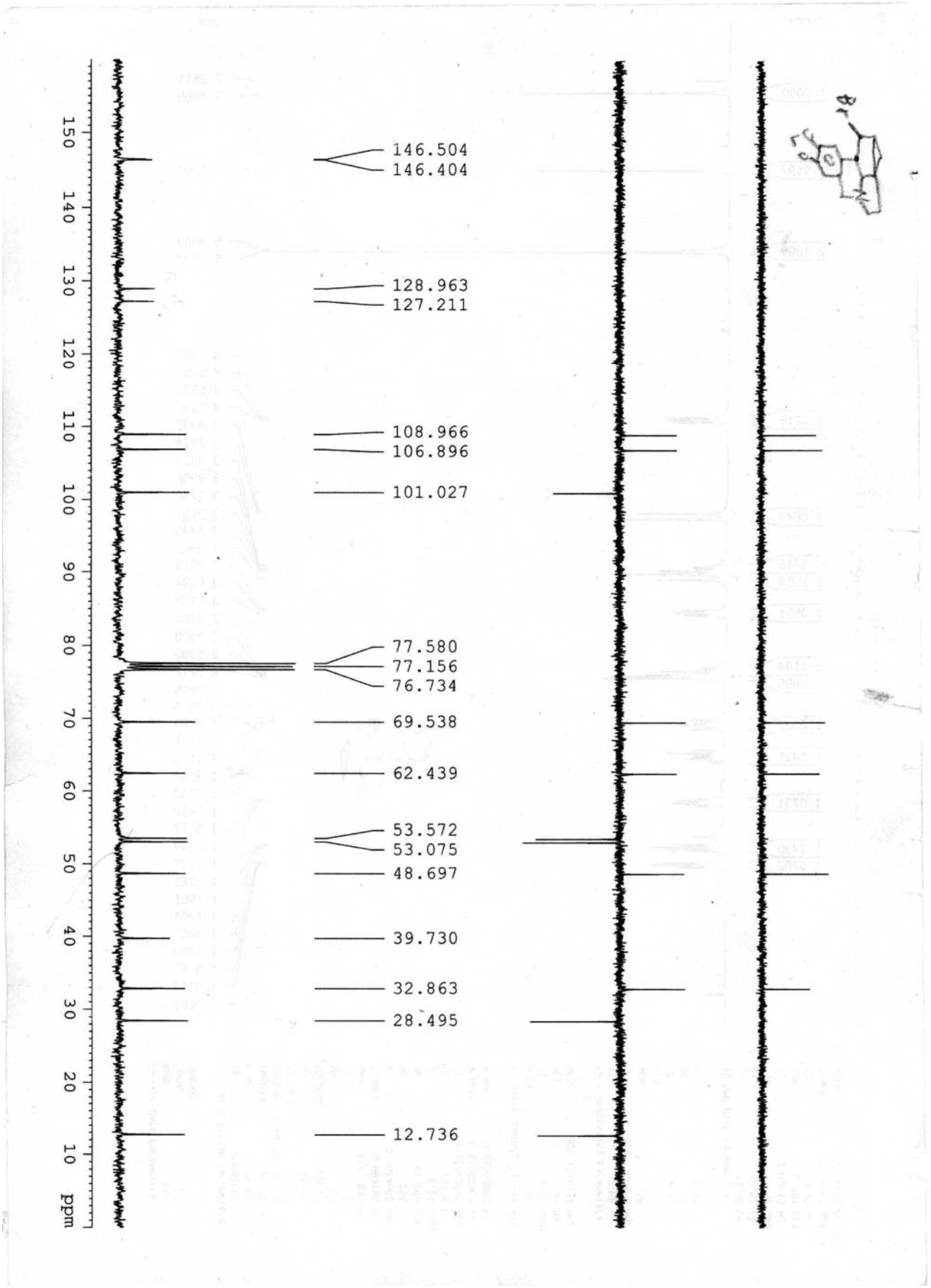
F2 - Acquisition Parameters
Date_ 20071212
Time 13.11
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 50.8
DW 81.000 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz

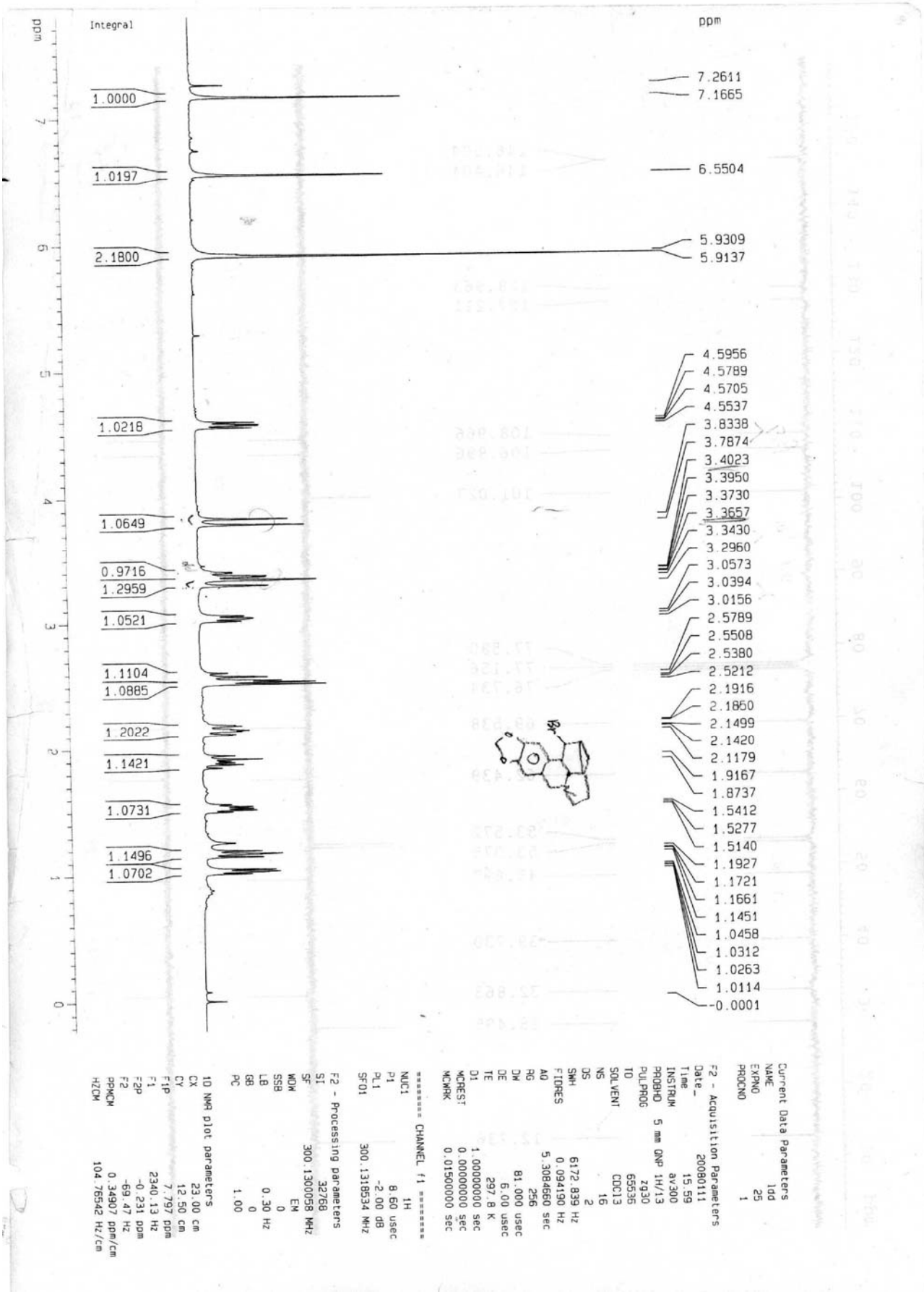
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 7.639 ppm
F1 2292.73 Hz
F2P 0.158 ppm
F2 47.30 Hz
PPMCM 0.32528 ppm/cm
HZCM 97.62738 Hz/cm

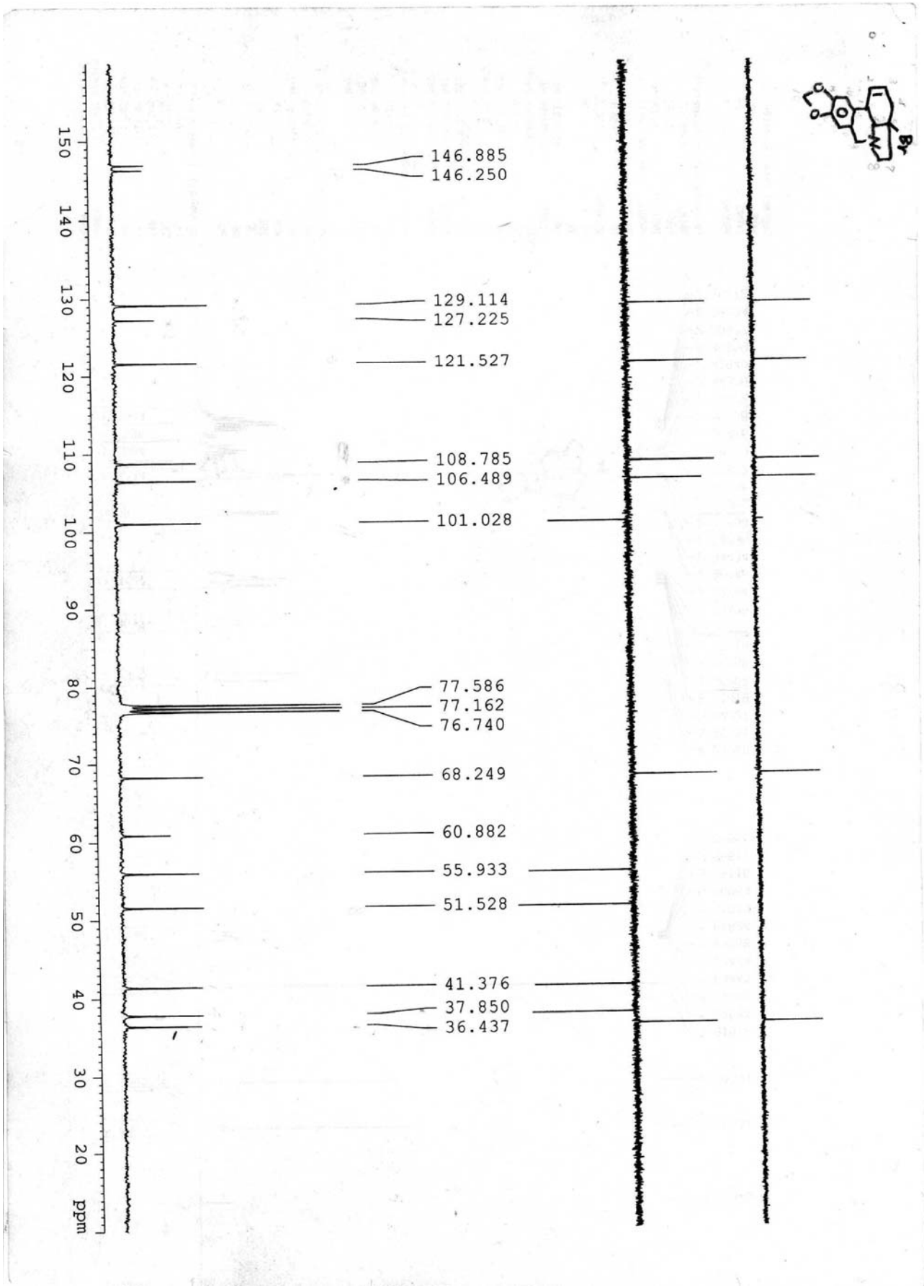
Compound 15


146.504
146.404
128.963
127.211
108.966
106.896
101.027
77.580
77.156
76.734
69.538
62.439
53.572
53.075
48.697
39.730
32.863
28.495
12.736
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

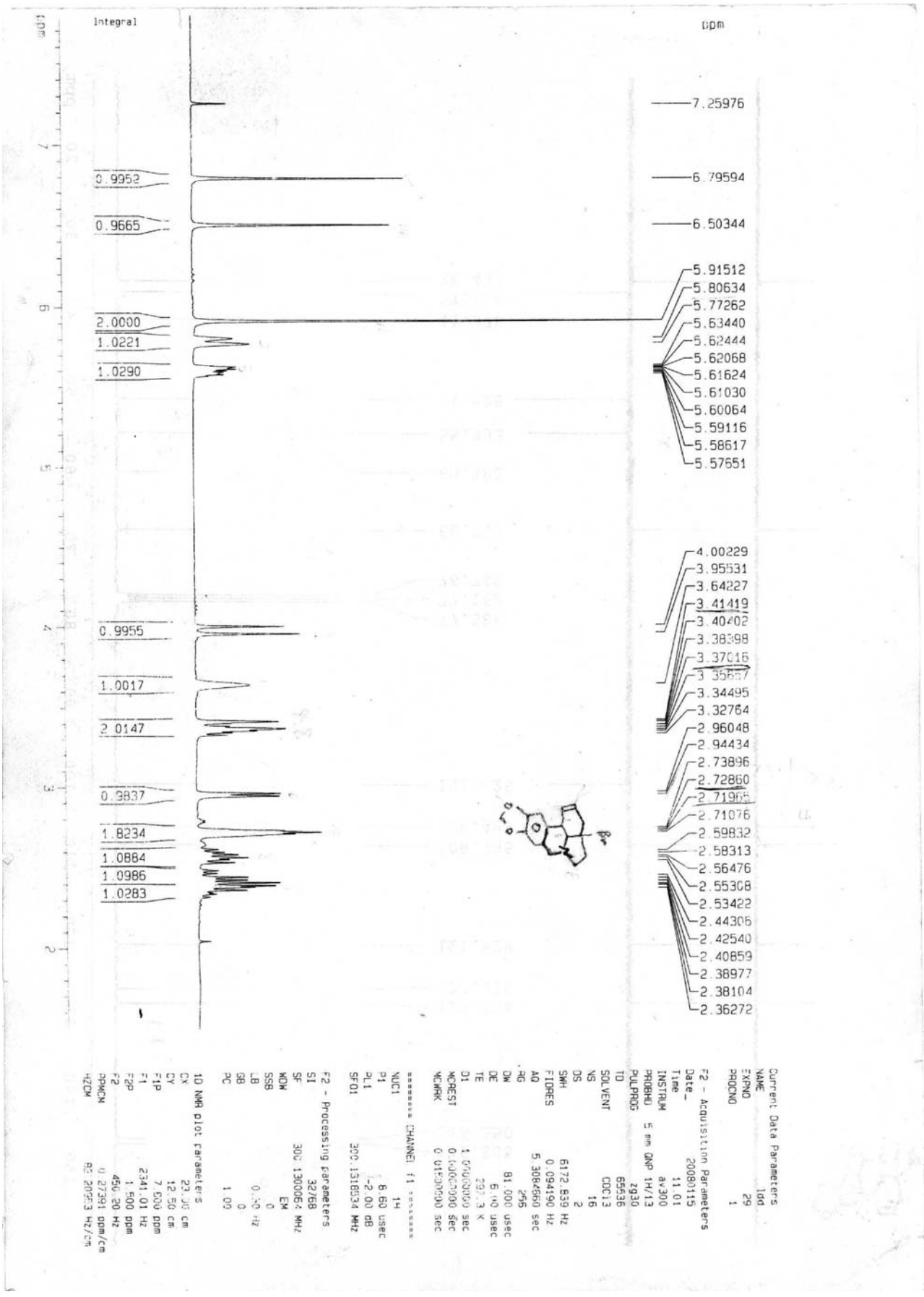
Compound 15


Integral
ppm
1.0000
1.0197
2.1800
1.0218
1.0649
0.9716
1.2959
1.0521
1.1104
1.0885
1.2022
1.1421
1.0731
1.1496
1.0702
7.2611
7.1665
6.5504
5.9309
5.9137
4.5956
4.5789
4.5705
4.5537
3.8338
3.7874
3.4023
3.3950
3.3730
3.3657
3.3430
3.2960
3.0573
3.0394
3.0156
2.5789
2.5508
2.5380
2.5212
2.1916
2.1850
2.1499
2.1420
2.1179
1.9167
1.8737
1.5412
1.5277
1.5140
1.1927
1.1721
1.1661
1.1451
1.0458
1.0312
1.0263
1.0114
-0.0001
Br
Current Data Parameters
NAME ldd
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080111
Time 15.59
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 297.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 7.797 ppm
F1 2340.13 Hz
F2P -0.231 ppm
F2 -69.47 Hz
PPMCM 0.34907 ppm/cm
HZCM 104.76542 Hz/cm

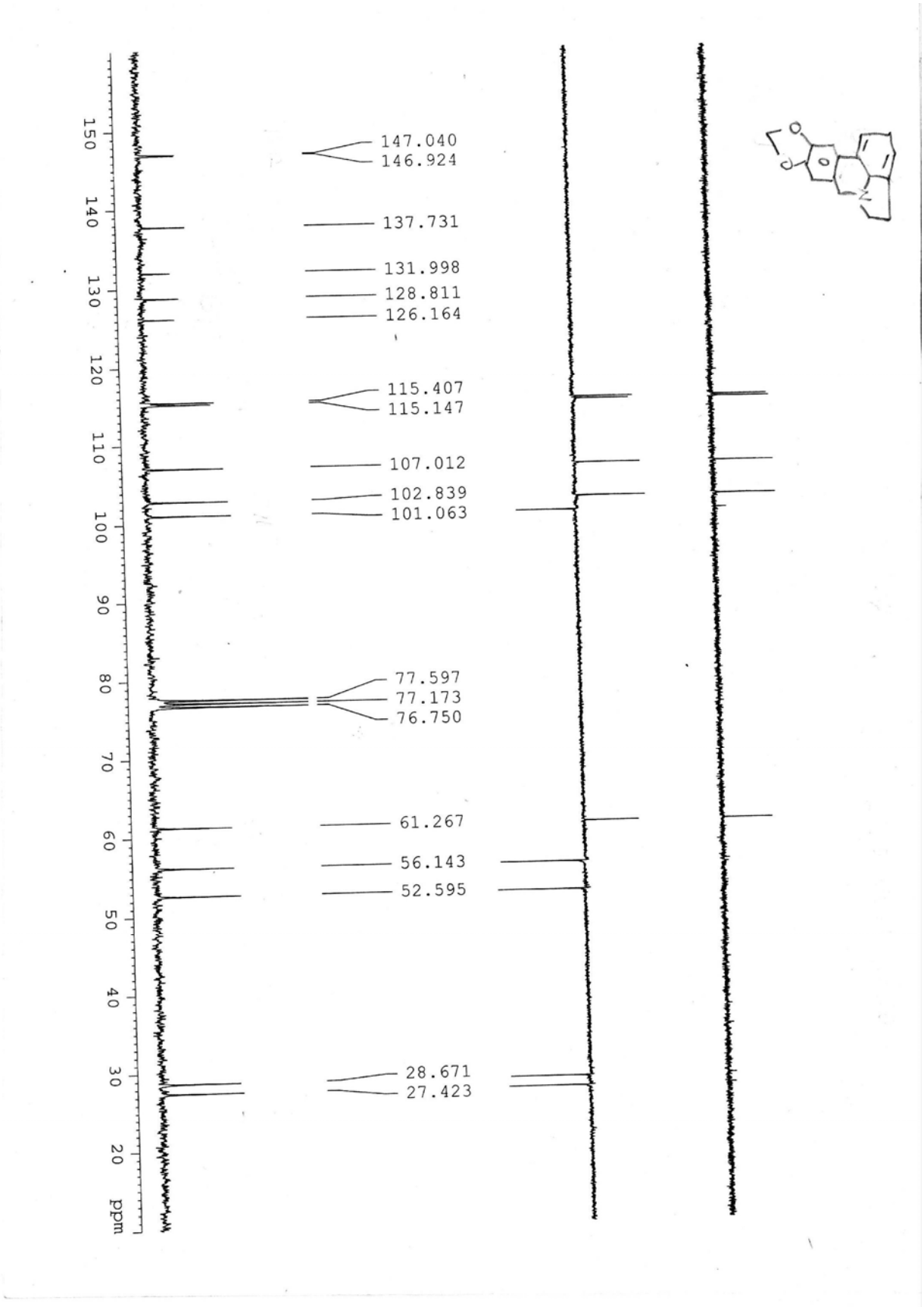
Compound 16


146.885
146.250
129.114
127.225
121.527
108.785
106.489
101.028
77.586
77.162
76.740
68.249
60.882
55.933
51.528
41.376
37.850
36.437
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
Br

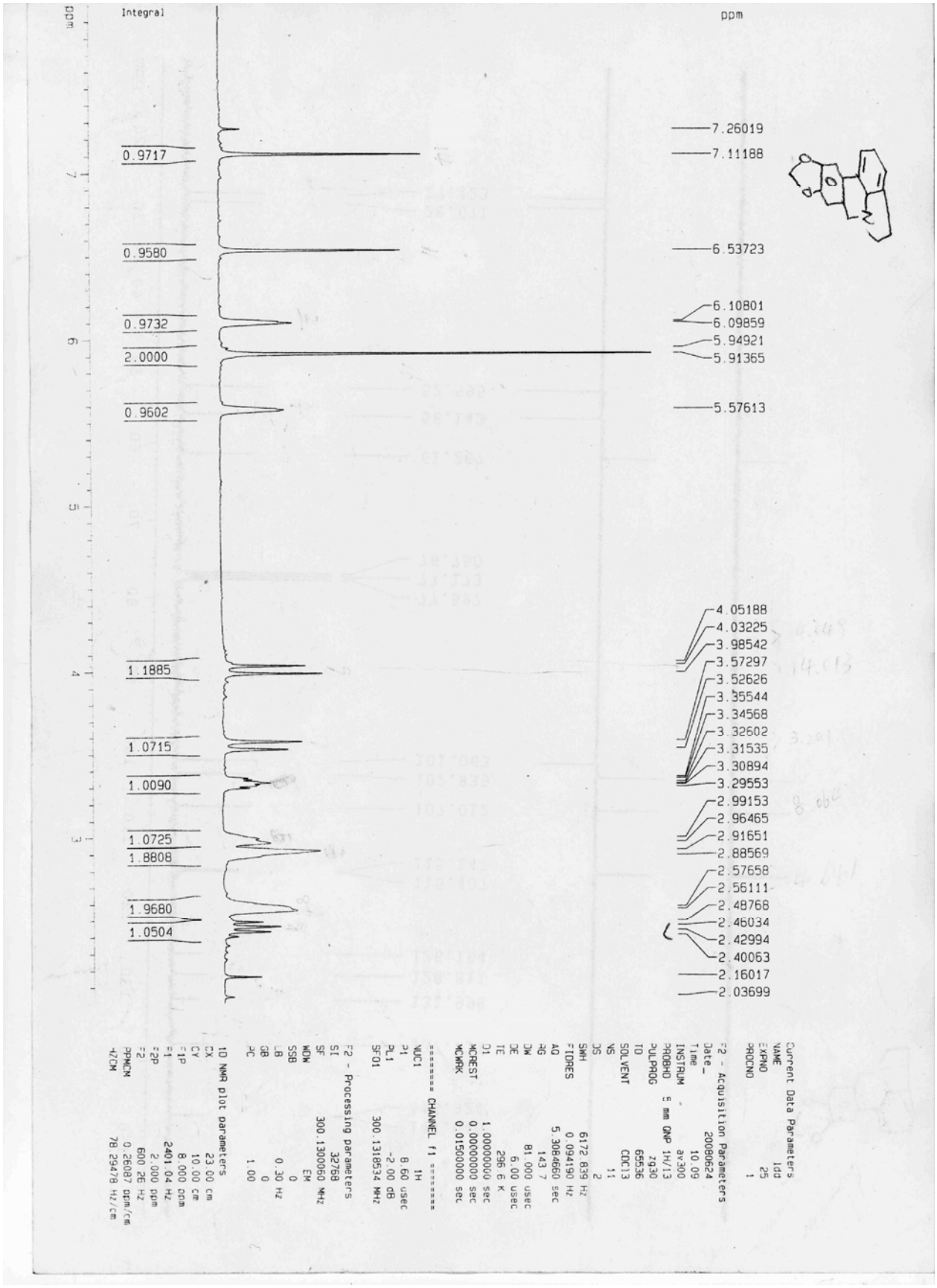
Compound 16


Integral
ppm
7.25976
6.79594
6.50344
5.91512
5.80634
5.77262
5.63440
5.62444
5.62068
5.61624
5.61030
5.60064
5.59116
5.58617
5.57651
4.00229
3.95531
3.64227
3.41419
3.40402
3.38398
3.37016
3.35657
3.34495
3.32764
2.96048
2.94434
2.73896
2.72860
2.71965
2.71076
2.59832
2.58313
2.56476
2.55308
2.53422
2.44306
2.42540
2.40859
2.38977
2.38104
2.36272
0.9952
0.9665
2.0000
1.0221
1.0290
0.9955
1.0017
2.0147
0.9837
1.8234
1.0884
1.0986
1.0283
Current Data Parameters
NAME
EXPNO 29
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080115
Time 11.01
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084960 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 297.3 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1316934 MHz
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.96 cm
CY 12.50 cm
F1P 7.500 ppm
F1 2341.01 Hz
F2P 1.500 ppm
F2 450.20 Hz
PPMCM 0.27391 ppm/cm
HZCM 82.20823 Hz/cm

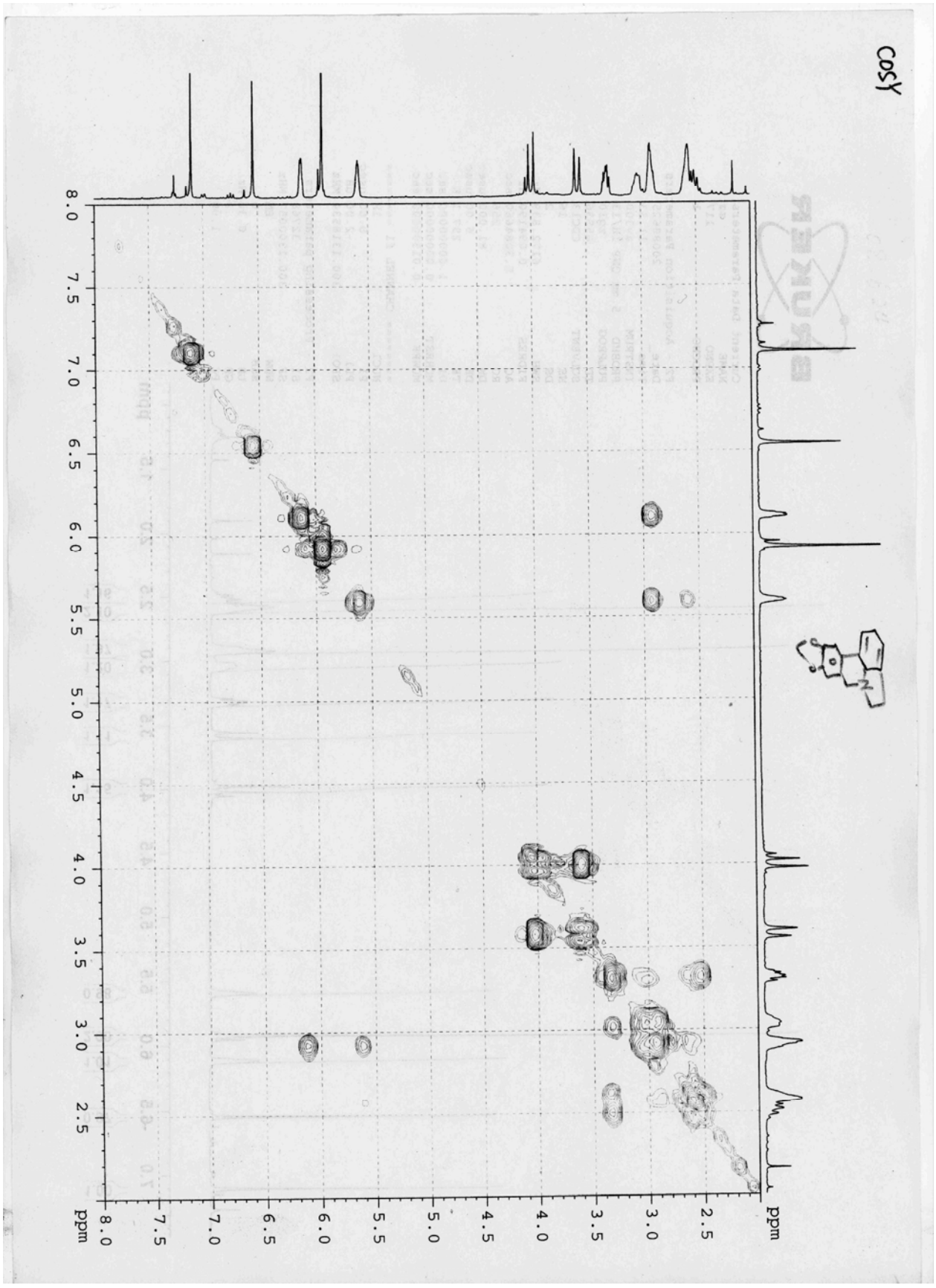
Compound 1


147.040
146.924
137.731
131.998
128.811
126.164
115.407
115.147
107.012
102.839
101.063
77.597
77.173
76.750
61.267
56.143
52.595
28.671
27.423
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

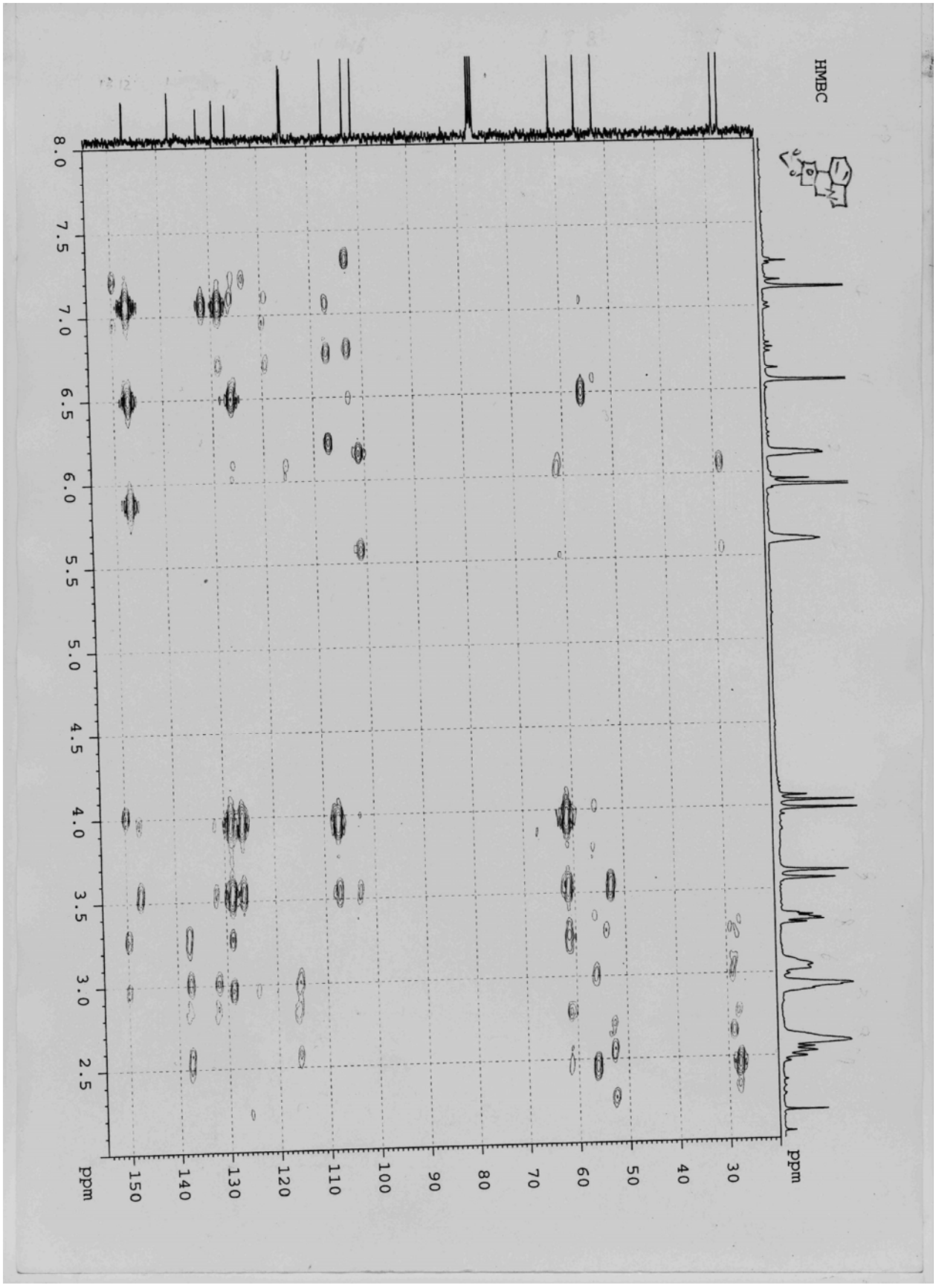
Compound 1


Integral
ppm
0.9717
0.9580
0.9732
2.0000
0.9602
1.1885
1.0715
1.0090
1.0725
1.8808
1.9680
1.0504
7.26019
7.11188
6.53723
6.10801
6.09859
5.94921
5.91365
5.57613
4.05188
4.03225
3.98542
3.57297
3.52626
3.35544
3.34568
3.32602
3.31535
3.30894
3.29553
2.99153
2.96465
2.91651
2.88569
2.57658
2.56111
2.48768
2.46034
2.42994
2.40063
2.16017
2.03699
Current Data Parameters
NAME
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080624
Time 10.09
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 10.00 cm
F1P 8.000 ppm
F1 2401.04 Hz
F2P 2.000 ppm
F2 600.26 Hz
PPMCM 0.26087 ppm/cm
HZCM 78.29478 Hz/cm

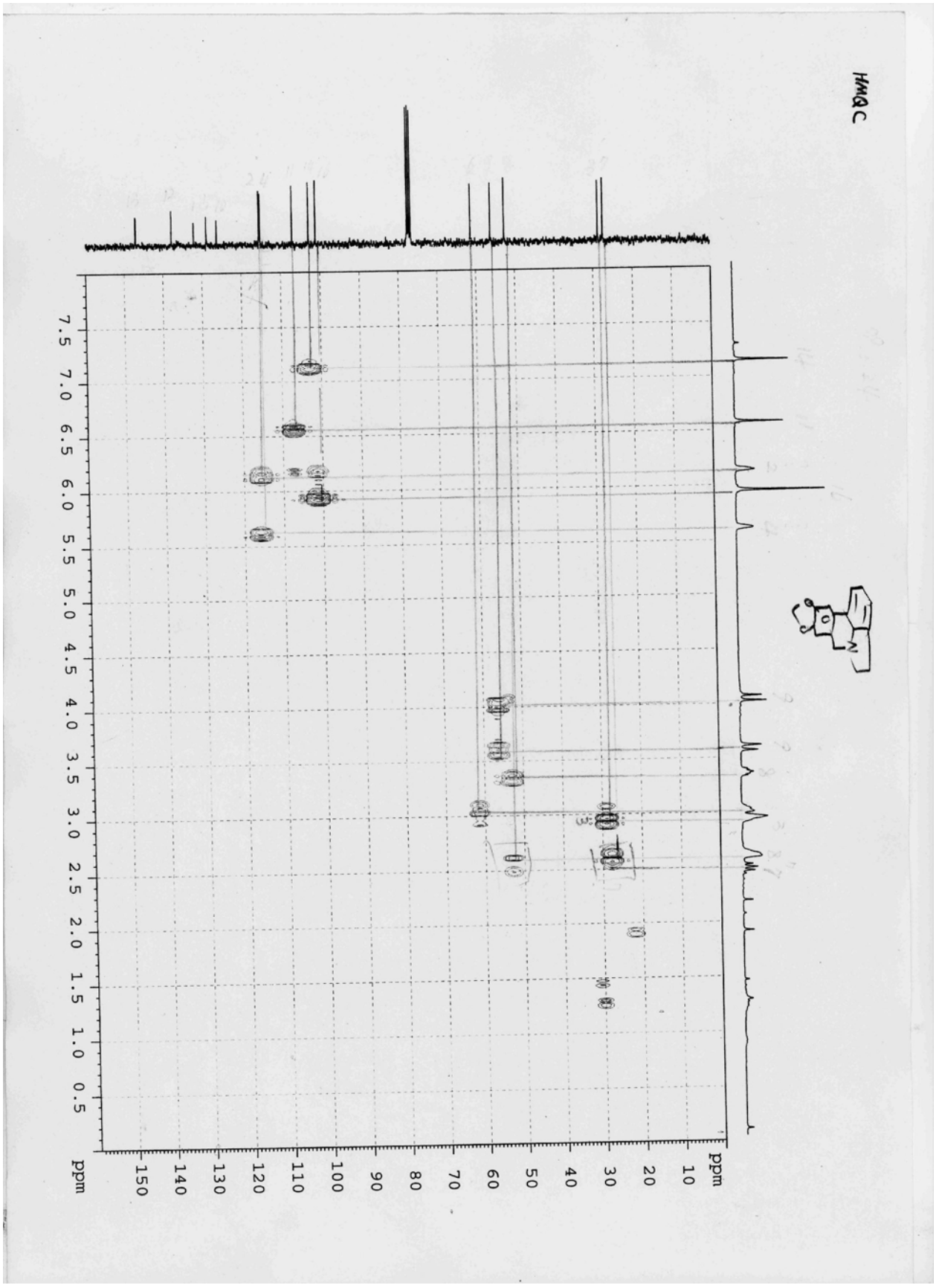
Compound 1



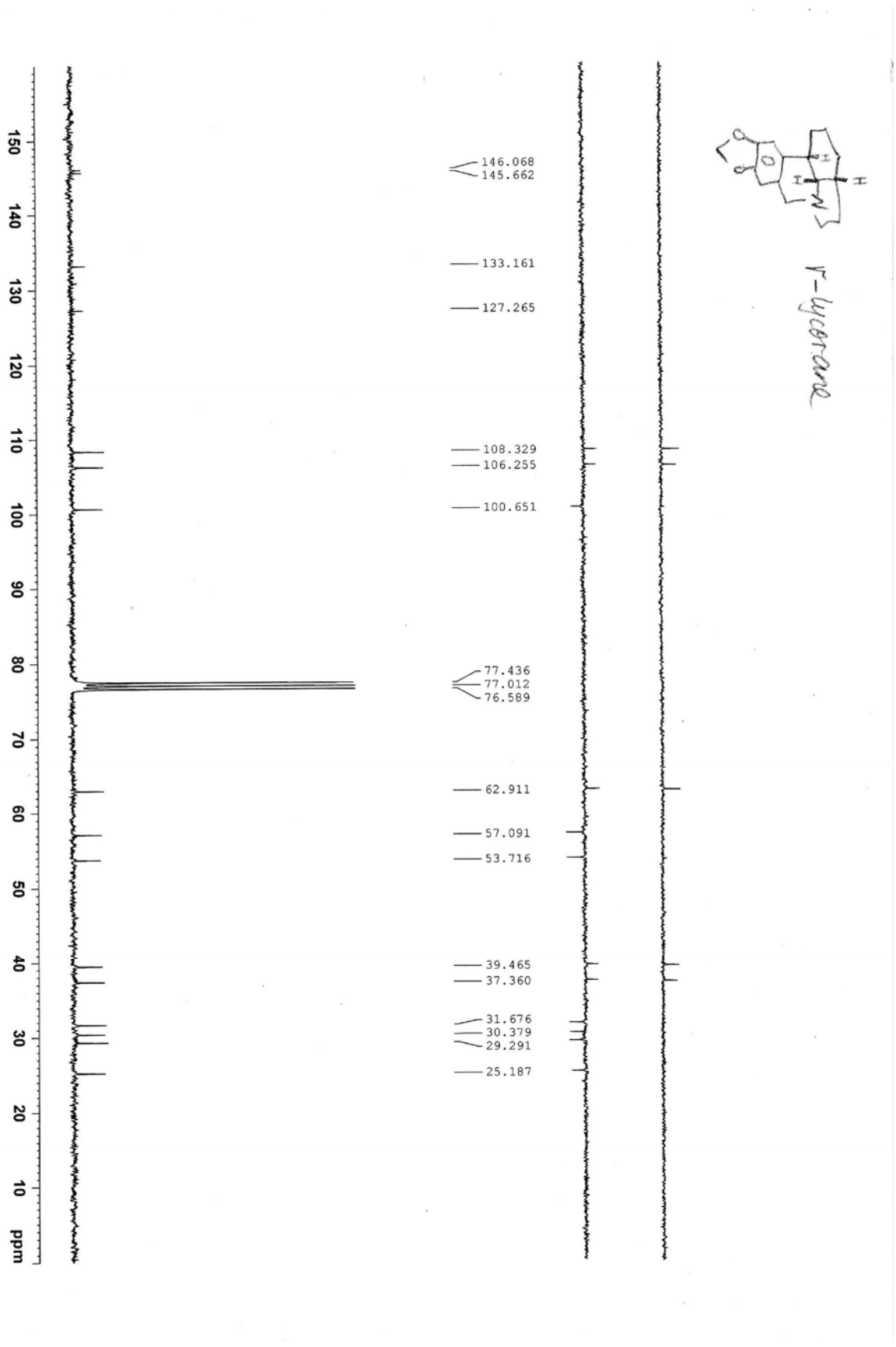
Compound 1


HMBC
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

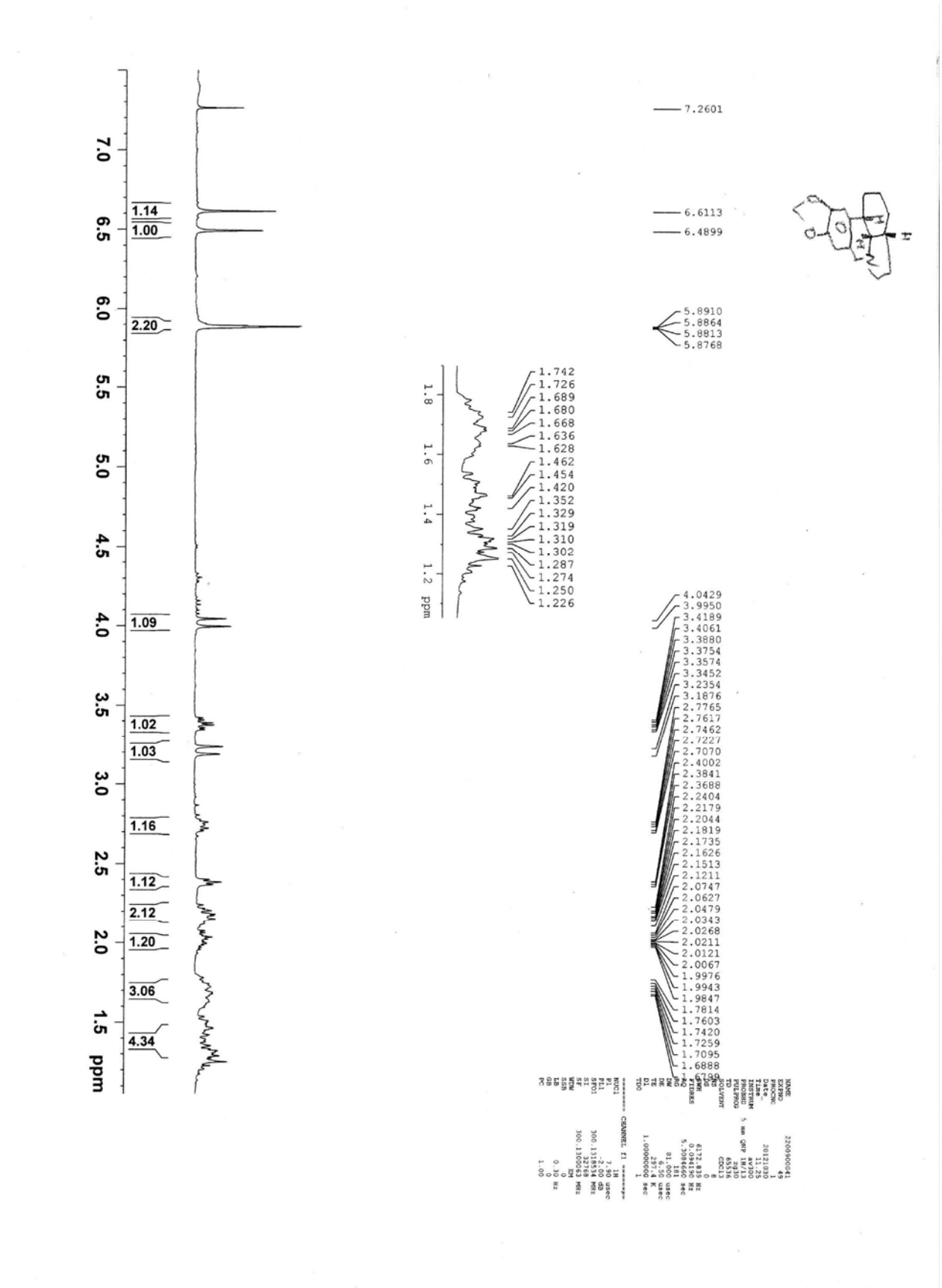
Compound 1



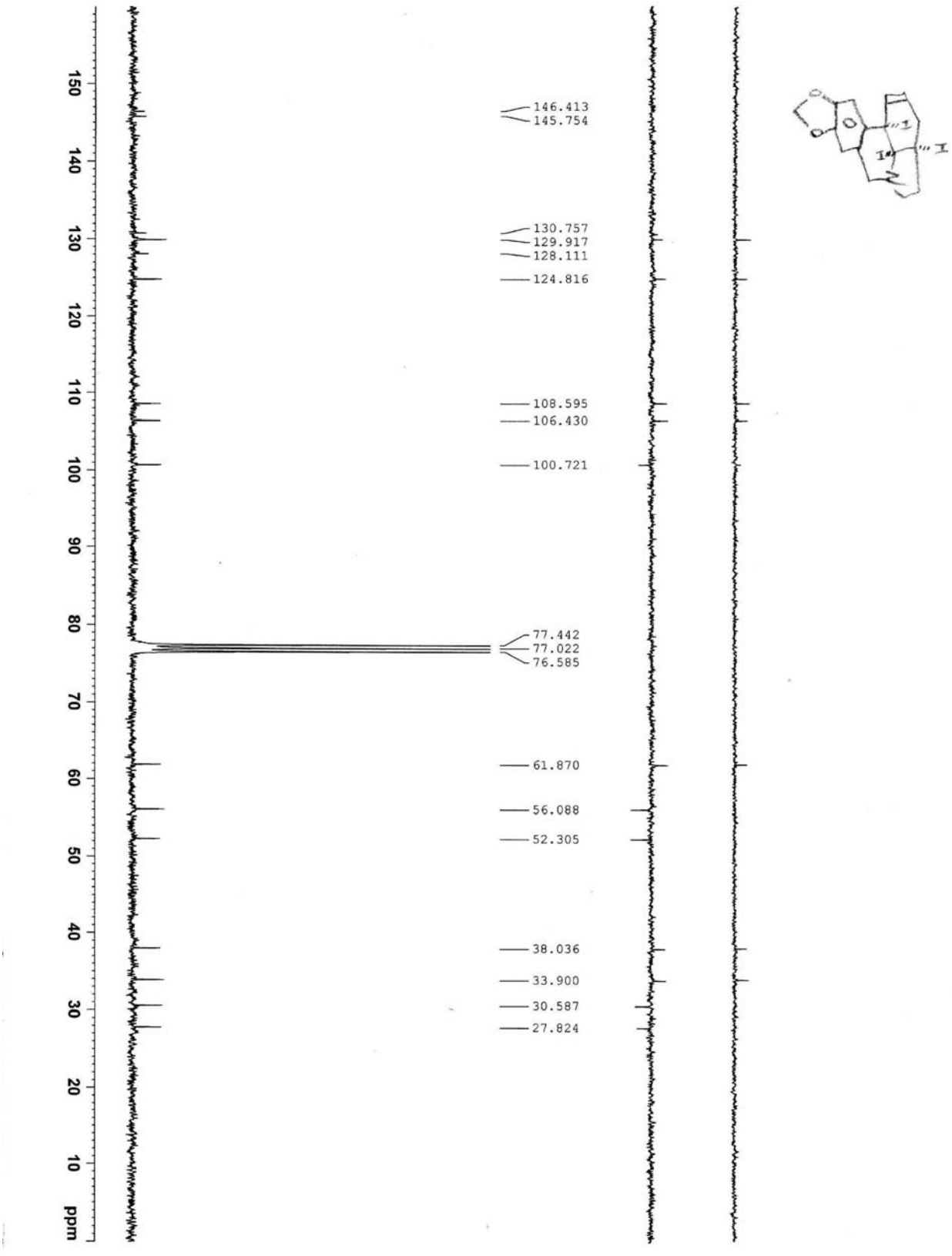
Compound 2


146.068
145.662
133.161
127.265
108.329
106.255
100.651
77.436
77.012
76.589
62.911
57.091
53.716
39.465
37.360
31.676
30.379
29.291
25.187
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

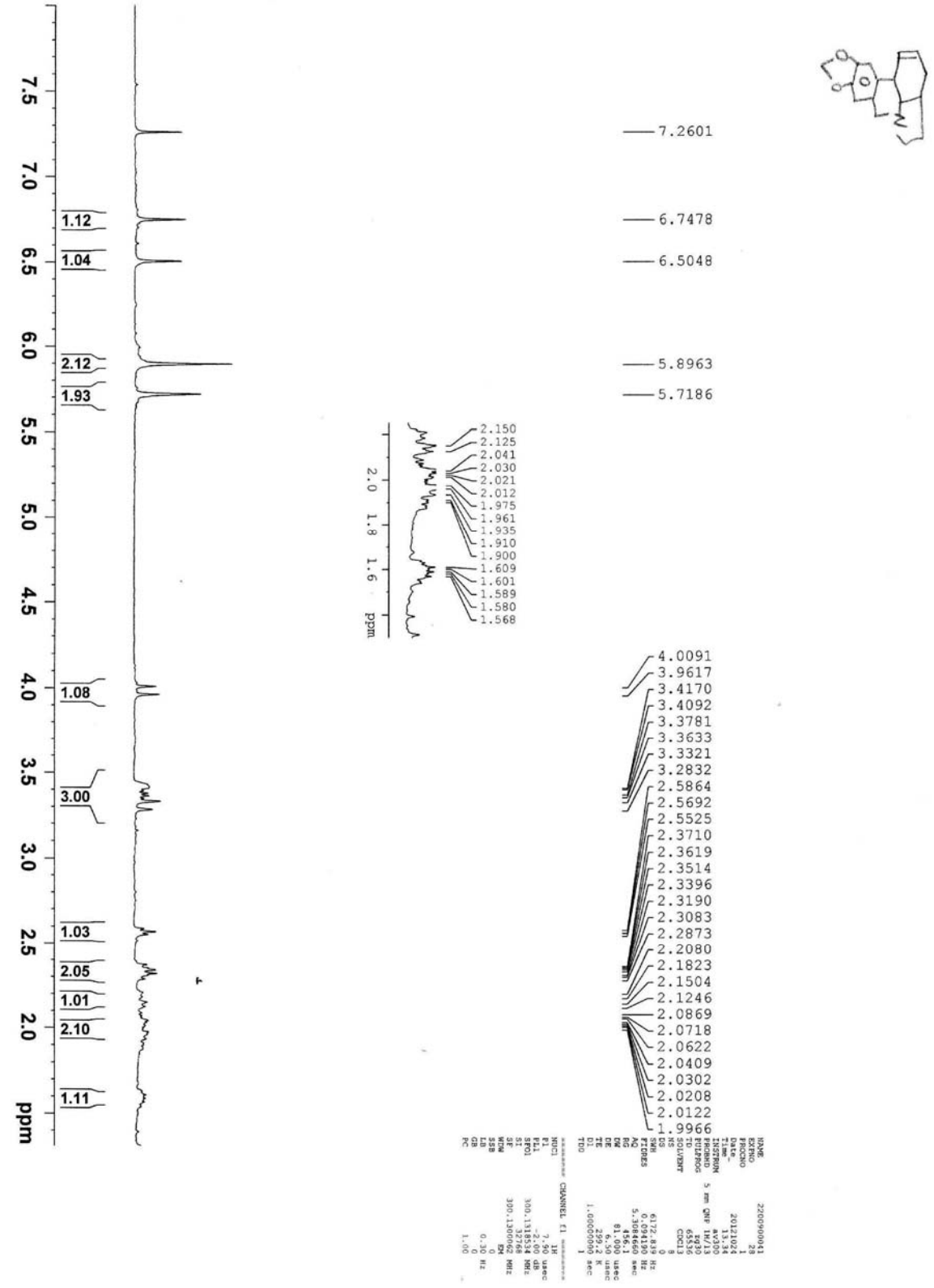
Compound 2


7.2601
6.6113
6.4899
5.8910
5.8864
5.8813
5.8768
1.742
1.726
1.689
1.680
1.668
1.636
1.628
1.462
1.454
1.420
1.352
1.329
1.319
1.310
1.302
1.287
1.274
1.250
1.226
4.0429
3.9950
3.4189
3.4061
3.3880
3.3754
3.3574
3.3452
3.2354
3.1876
2.7765
2.7617
2.7462
2.7227
2.7070
2.4002
2.3841
2.3688
2.2404
2.2179
2.2044
2.1819
2.1735
2.1626
2.1513
2.1211
2.0747
2.0627
2.0479
2.0343
2.0268
2.0211
2.0121
2.0067
1.9976
1.9943
1.9847
1.7814
1.7603
1.7420
1.7259
1.7095
1.6888
1.14
1.00
2.20
1.09
1.02
1.03
1.16
1.12
2.12
1.20
3.06
4.34
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.8
1.6
1.4
1.2
ppm

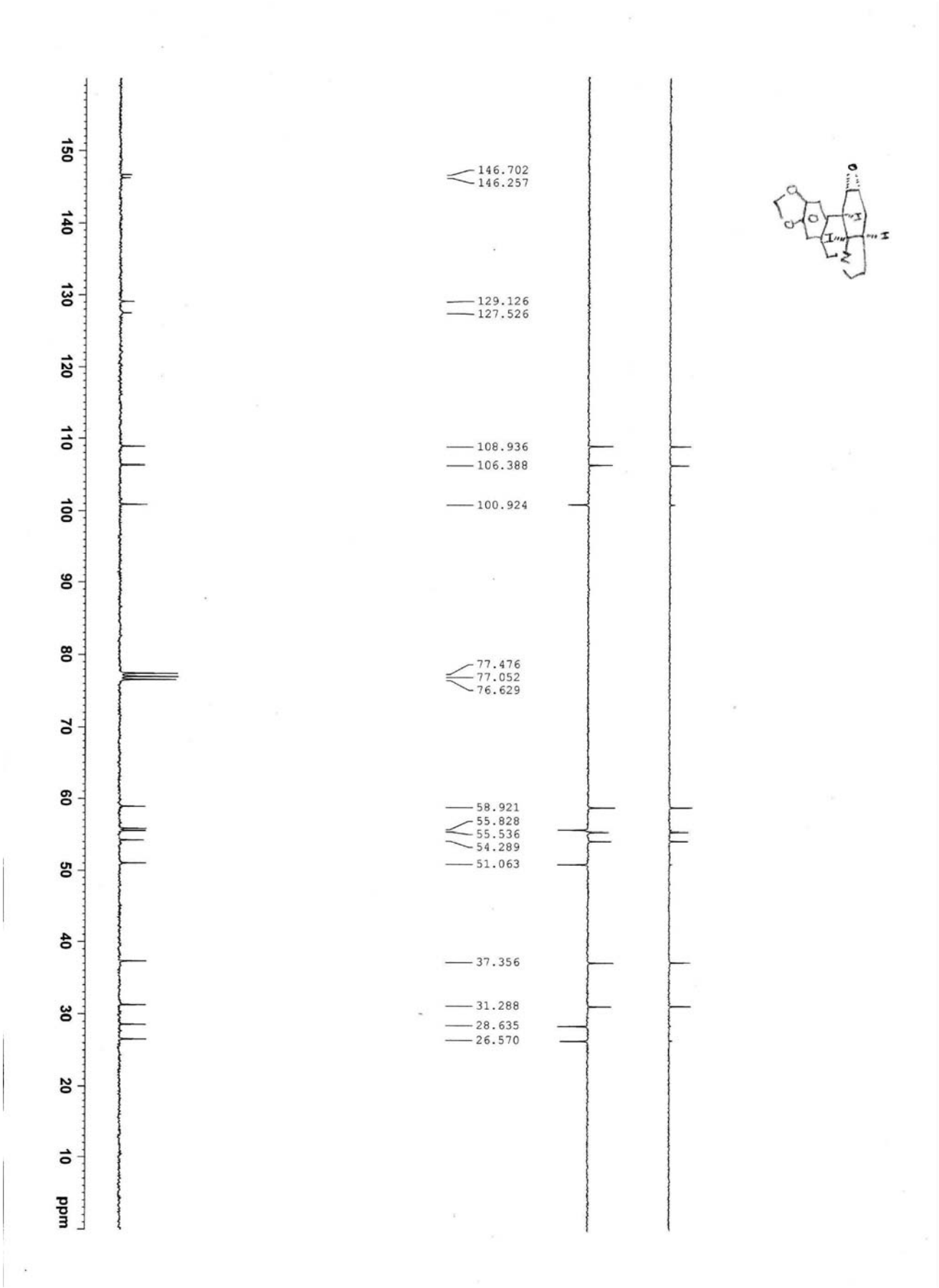
Compound 18


146.413
145.754
130.757
129.917
128.111
124.816
108.595
106.430
100.721
77.442
77.022
76.585
61.870
56.088
52.305
38.036
33.900
30.587
27.824
ppm

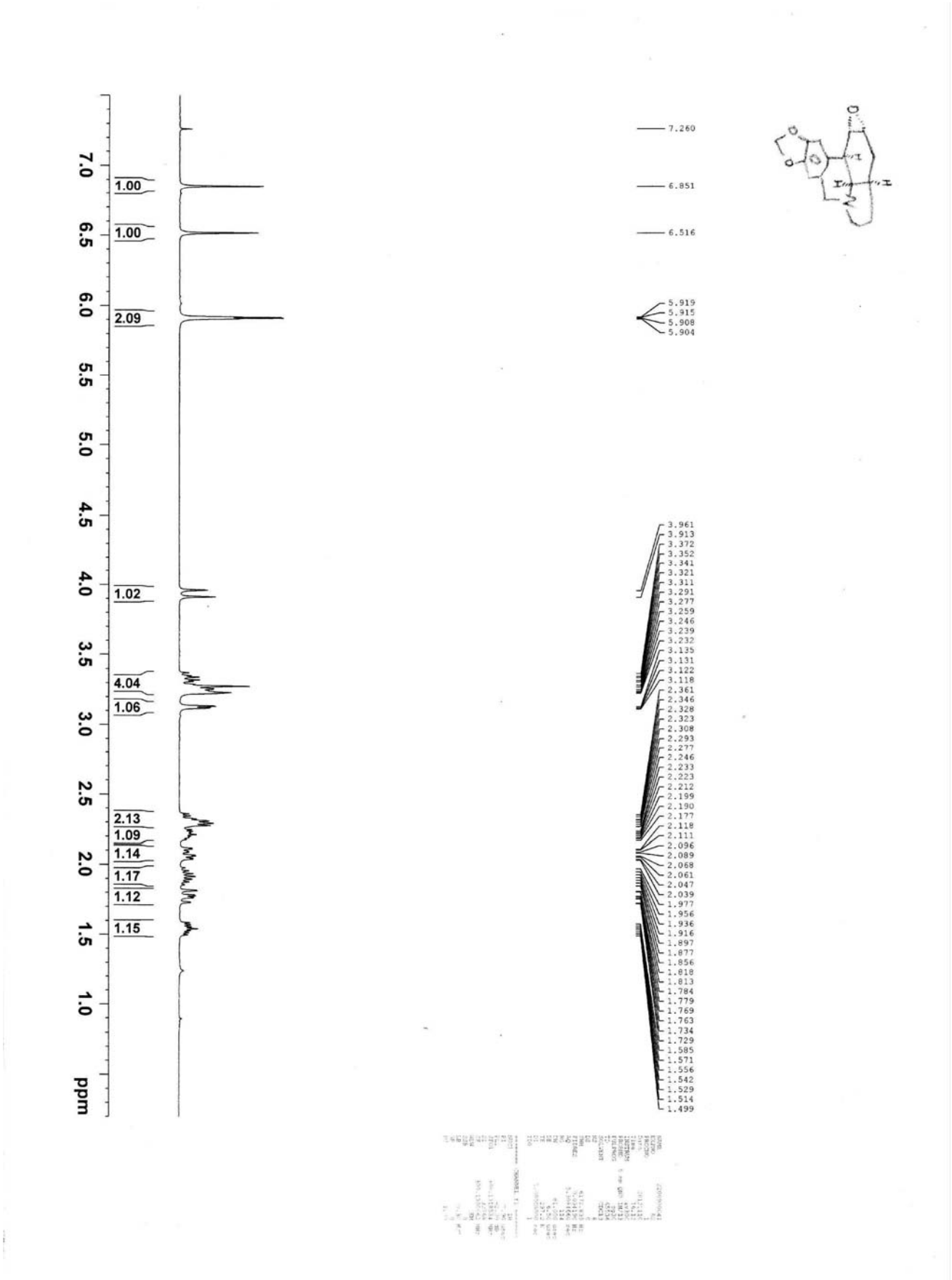
Compound 18


7.2601
6.7478
6.5048
5.8963
5.7186
4.0091
3.9617
3.4170
3.4092
3.3781
3.3633
3.3321
3.2832
2.5864
2.5692
2.5525
2.3710
2.3619
2.3514
2.3396
2.3190
2.3083
2.2873
2.2080
2.1823
2.1504
2.1246
2.0869
2.0718
2.0622
2.0409
2.0302
2.0208
2.0122
1.9966
2.150
2.125
2.041
2.030
2.021
2.012
1.975
1.961
1.935
1.910
1.900
1.609
1.601
1.589
1.580
1.568
2.0 1.8 1.6 ppm
1.12
1.04
2.12
1.93
1.08
3.00
1.03
2.05
1.01
2.10
1.11
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm

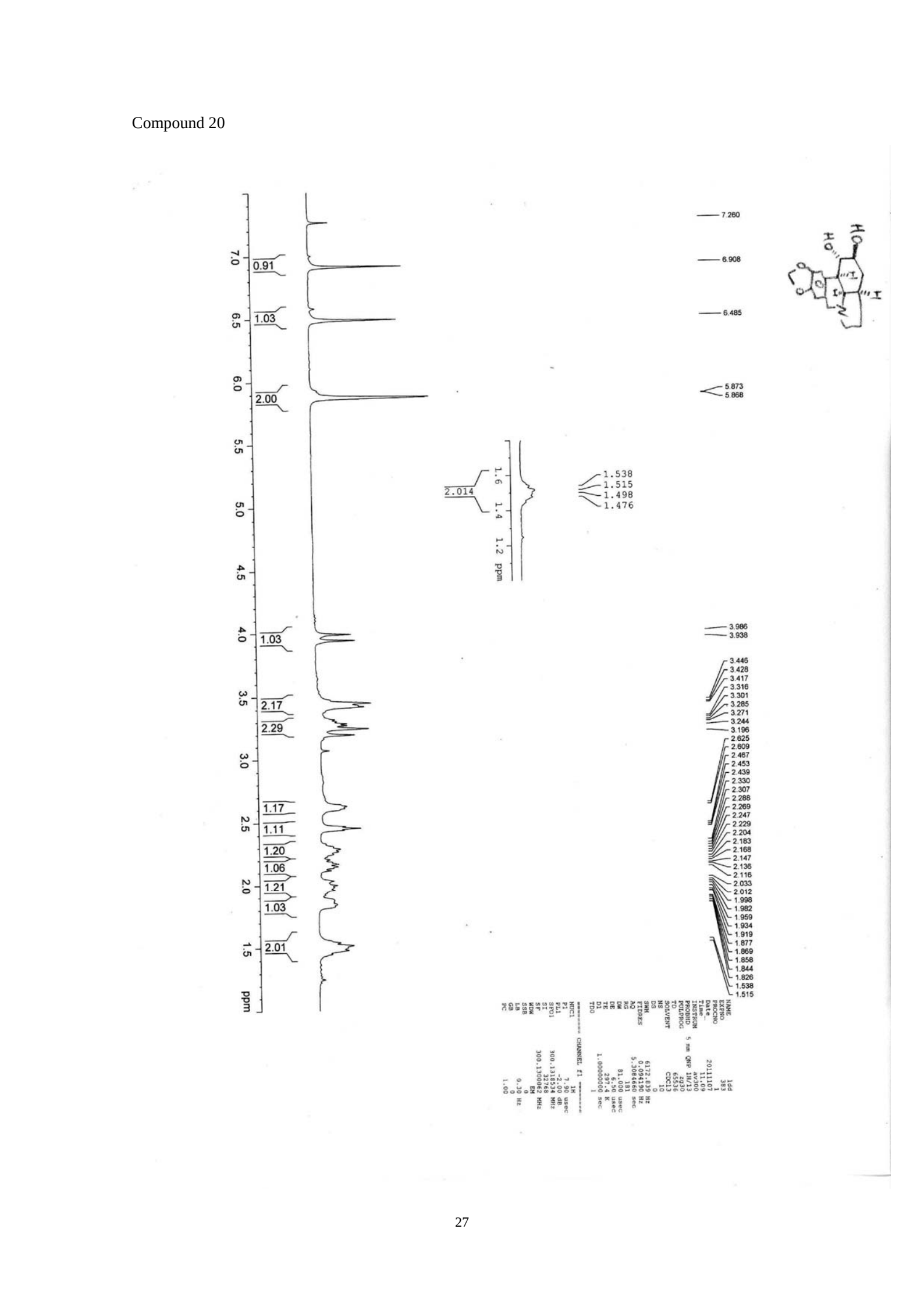
Compound 19


146.702
146.257
129.126
127.526
108.936
106.388
100.924
77.476
77.052
76.629
58.921
55.828
55.536
54.289
51.063
37.356
31.288
28.635
26.570
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

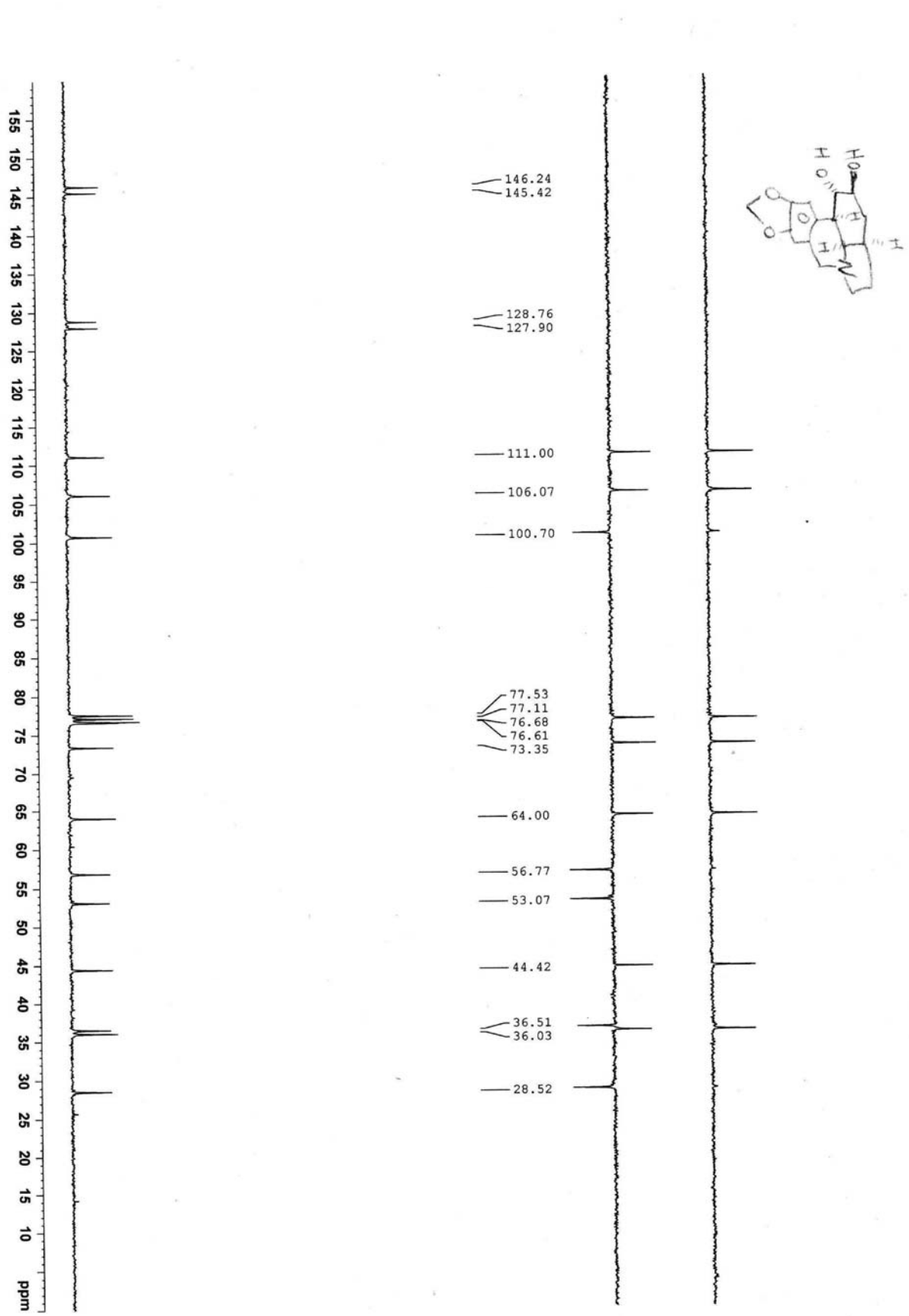
Compound 19


7.260
6.851
6.516
5.919
5.915
5.908
5.904
3.961
3.913
3.372
3.352
3.341
3.321
3.311
3.291
3.277
3.259
3.246
3.239
3.232
3.135
3.131
3.122
3.118
2.361
2.346
2.328
2.323
2.308
2.293
2.277
2.246
2.233
2.223
2.212
2.199
2.190
2.177
2.118
2.111
2.096
2.089
2.068
2.061
2.047
2.039
1.977
1.956
1.936
1.916
1.897
1.877
1.856
1.818
1.813
1.784
1.779
1.769
1.763
1.734
1.729
1.585
1.571
1.556
1.542
1.529
1.514
1.499
1.00
1.00
2.09
1.02
4.04
1.06
2.13
1.09
1.14
1.17
1.12
1.15
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
ppm

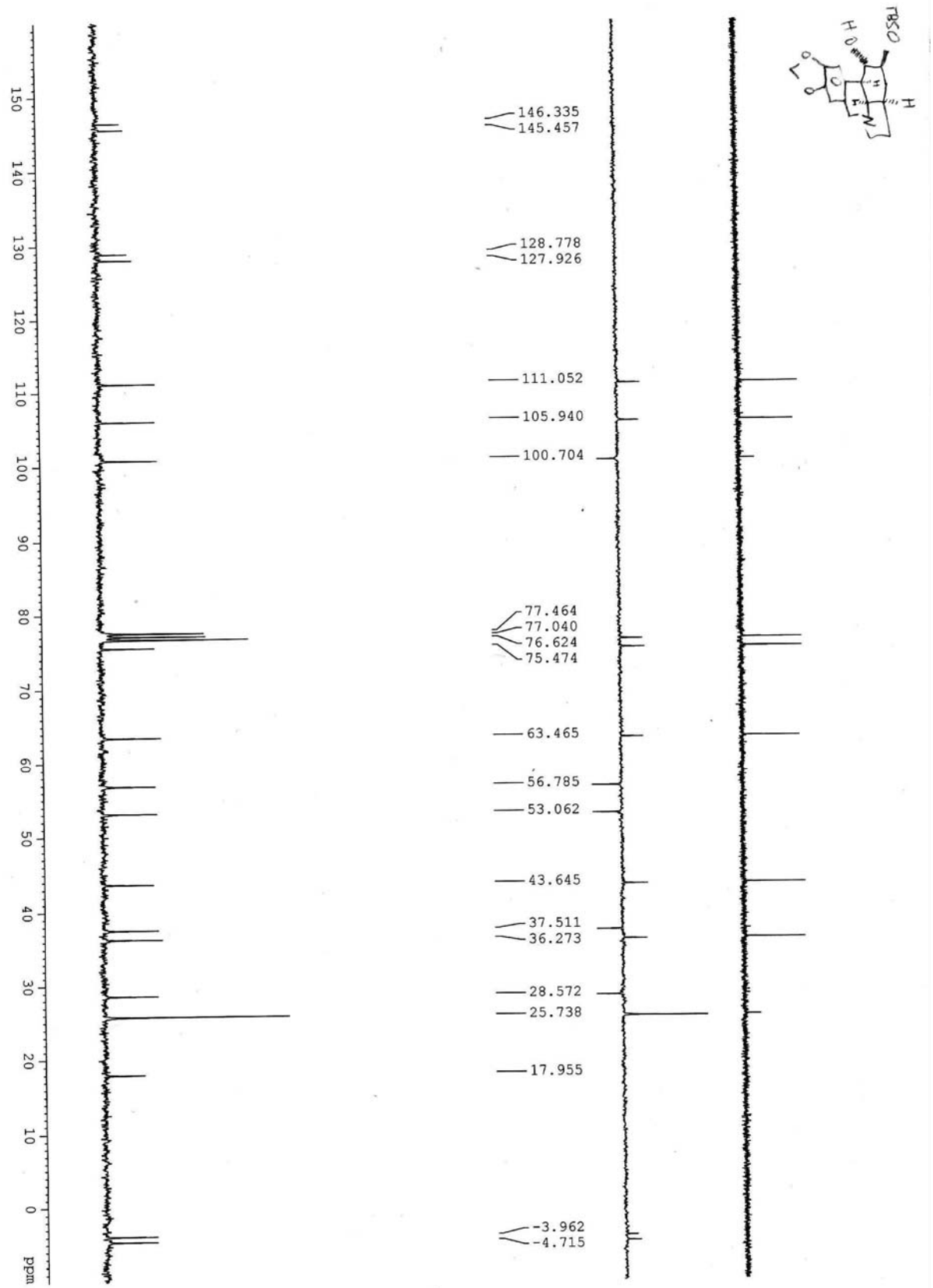
Compound 20


7.260
6.908
6.485
5.873
5.868
1.538
1.515
1.498
1.476
2.014
3.986
3.938
3.446
3.428
3.417
3.316
3.301
3.285
3.271
3.244
3.196
2.625
2.609
2.467
2.453
2.439
2.330
2.307
2.288
2.269
2.247
2.229
2.204
2.183
2.168
2.147
2.136
2.116
2.033
2.012
1.998
1.982
1.959
1.934
1.919
1.877
1.869
1.858
1.844
1.826
1.538
1.515
0.91
1.03
2.00
1.03
2.17
2.29
1.17
1.11
1.20
1.06
1.21
1.03
2.01
ppm

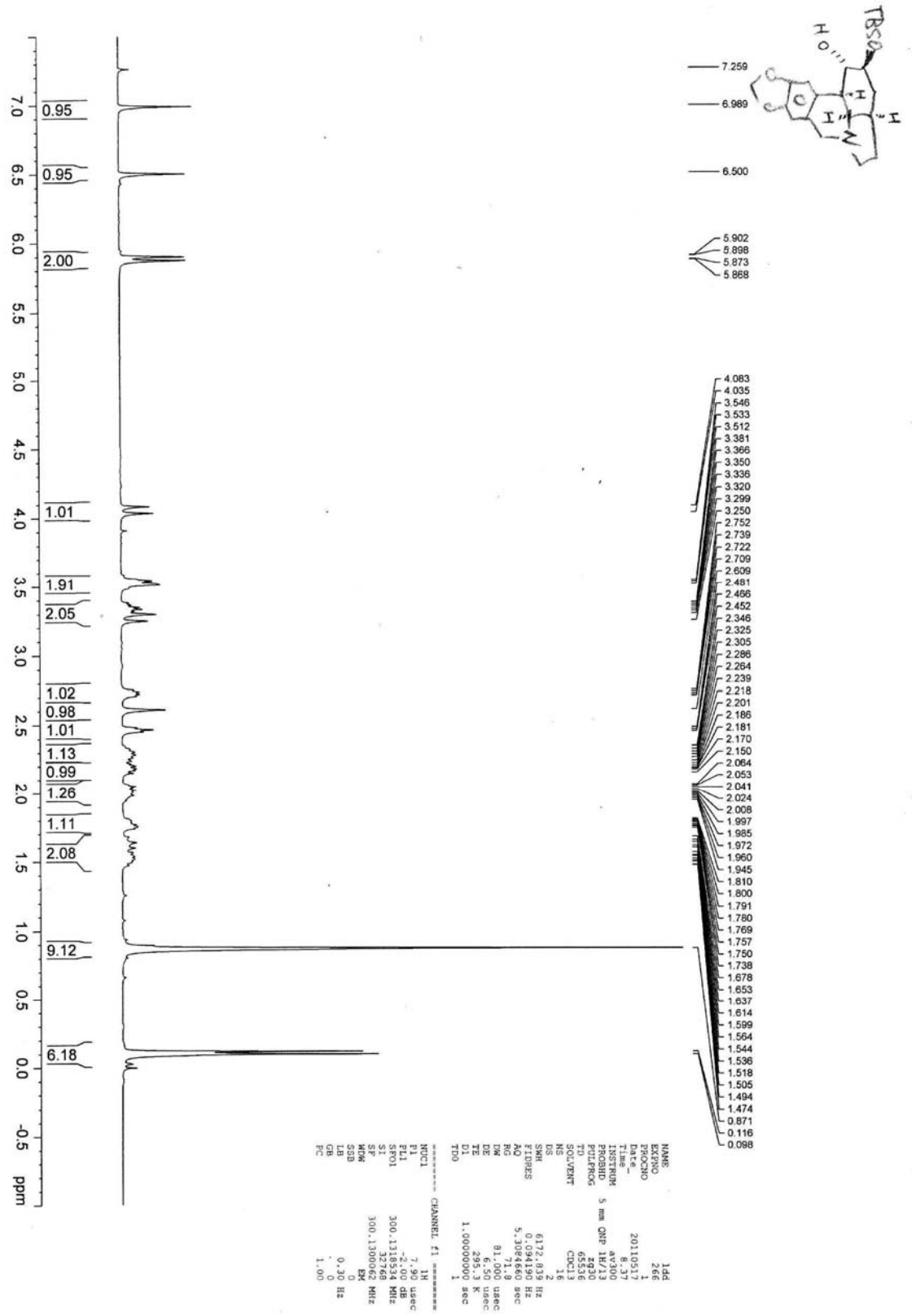
Compound 20


146.24
145.42
128.76
127.90
111.00
106.07
100.70
77.53
77.11
76.68
76.61
73.35
64.00
56.77
53.07
44.42
36.51
36.03
28.52
ppm

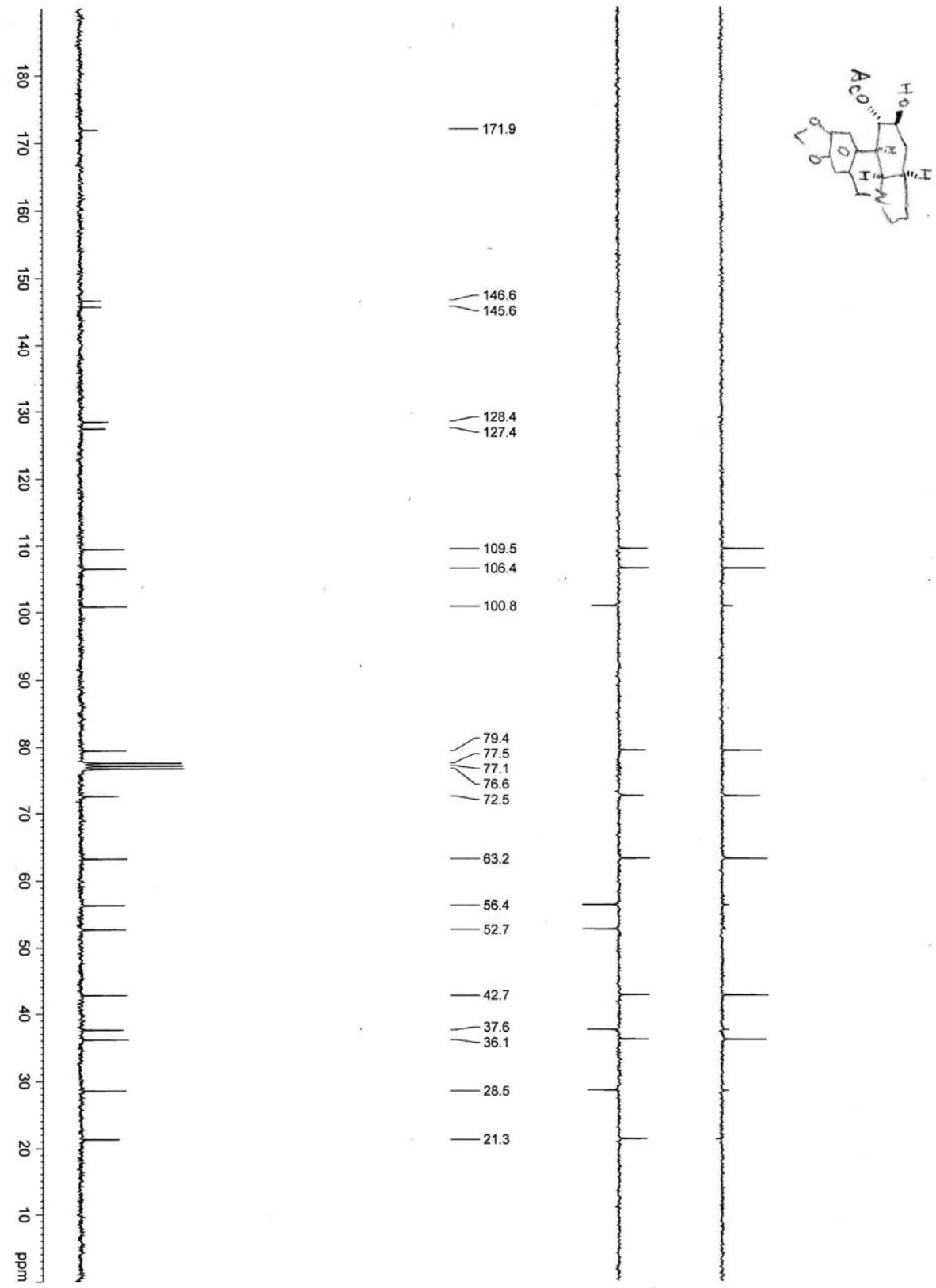
Compound 21


TBSO
H
146.335
145.457
128.778
127.926
111.052
105.940
100.704
77.464
77.040
76.624
75.474
63.465
56.785
53.062
43.645
37.511
36.273
28.572
25.738
17.955
-3.962
-4.715
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

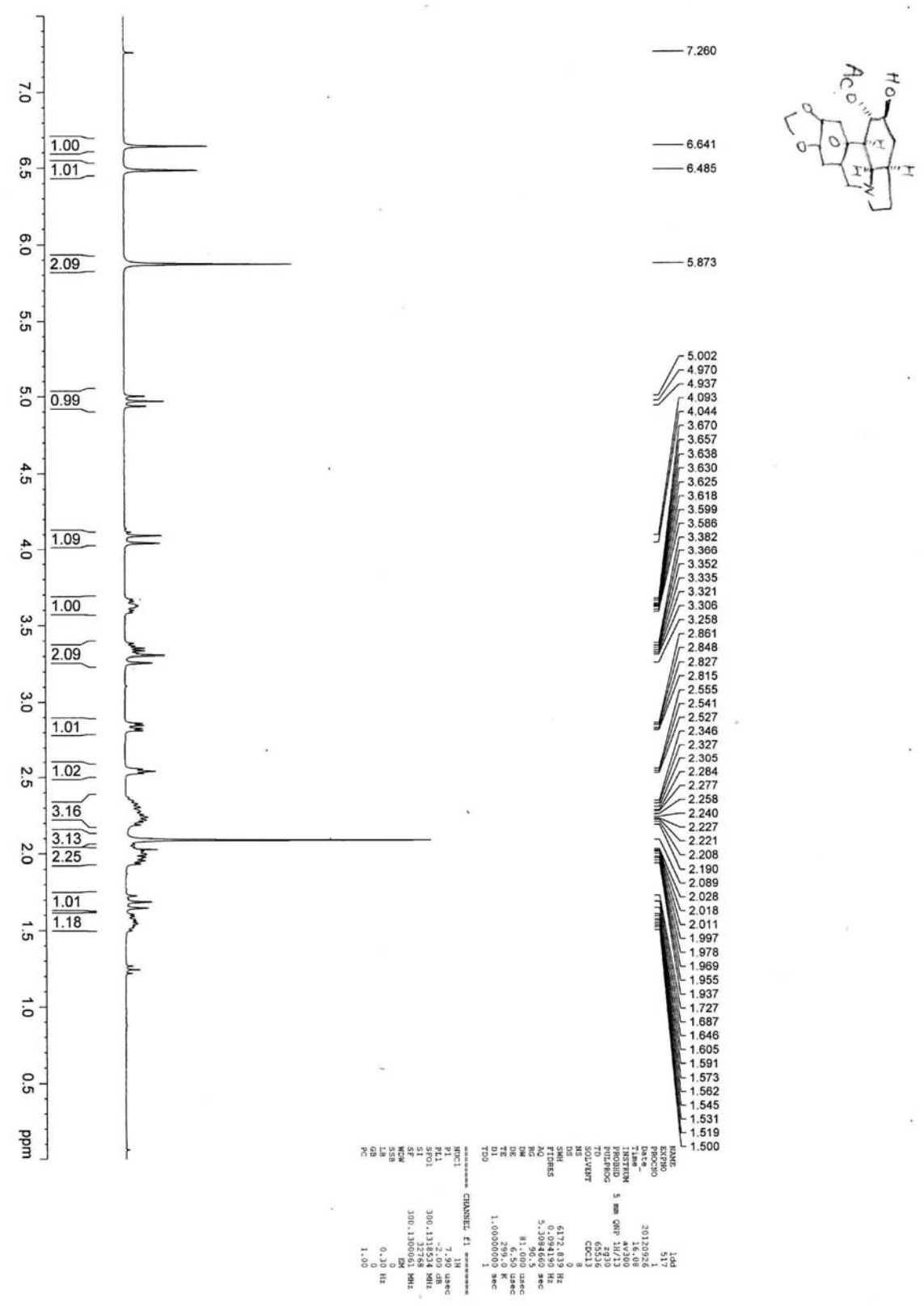
Compound 21


TBSO
HO
H
N
7.259
6.989
6.500
5.902
5.898
5.873
5.868
4.083
4.035
3.546
3.533
3.512
3.381
3.366
3.350
3.336
3.320
3.299
3.250
2.752
2.739
2.722
2.709
2.609
2.481
2.466
2.452
2.346
2.325
2.305
2.286
2.264
2.239
2.218
2.201
2.186
2.181
2.170
2.150
2.064
2.053
2.041
2.024
2.008
1.997
1.985
1.972
1.960
1.945
1.810
1.800
1.791
1.780
1.769
1.757
1.750
1.738
1.678
1.653
1.637
1.614
1.599
1.564
1.544
1.536
1.518
1.505
1.494
1.474
0.871
0.116
0.098
0.95
0.95
2.00
1.01
1.91
2.05
1.02
0.98
1.01
1.13
0.99
1.26
1.11
2.08
9.12
6.18
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
-0.5
ppm
NAME 1dd
EXPNO 26
PROCNO 1
Date_ 20110517
Time_ 8.37
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084719 sec
RG 81.000
DW 81.000 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1316154 MHz
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

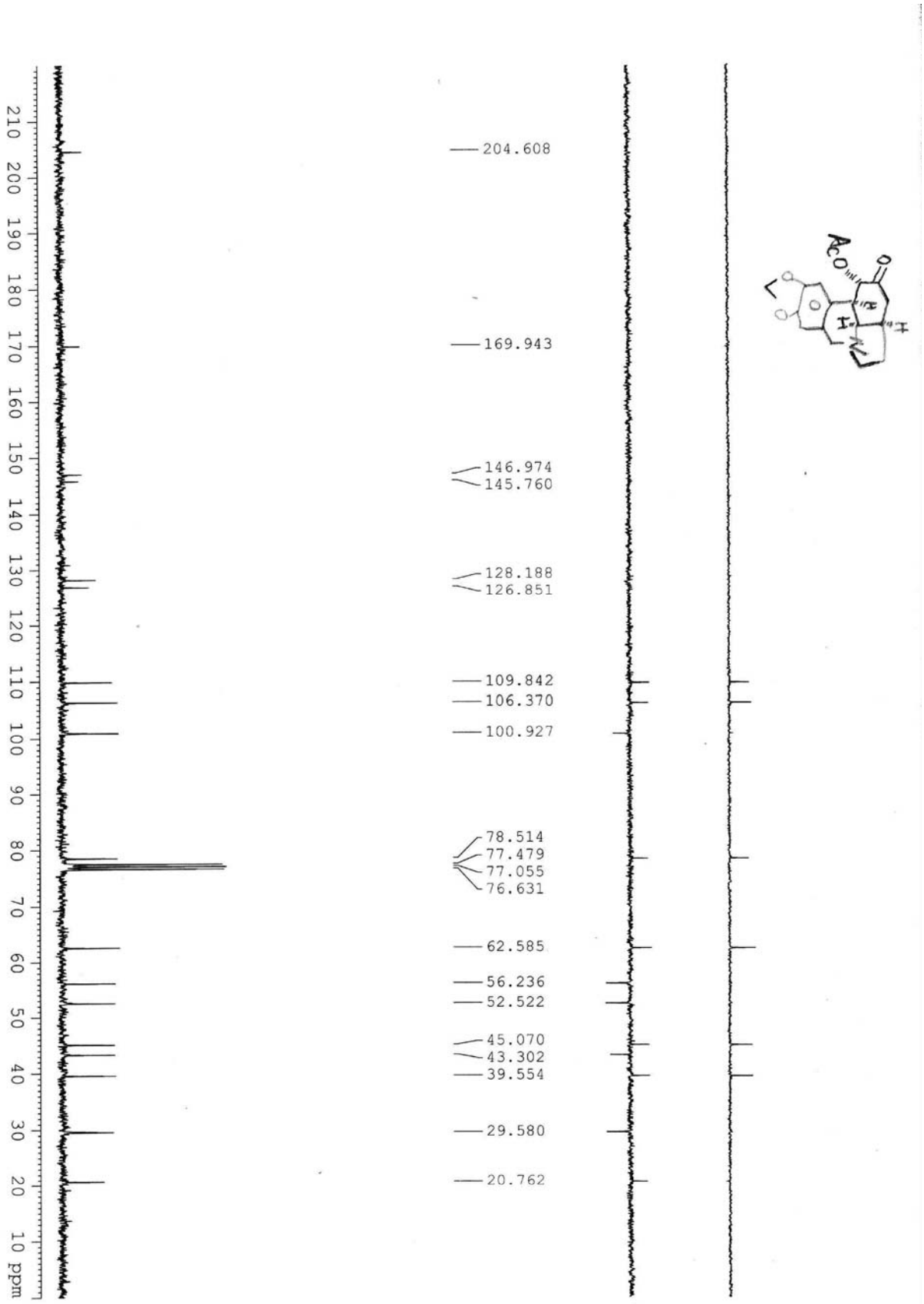
Compound 22


171.9
146.6
145.6
128.4
127.4
109.5
106.4
100.8
79.4
77.5
77.1
76.6
72.5
63.2
56.4
52.7
42.7
37.6
36.1
28.5
21.3
ppm

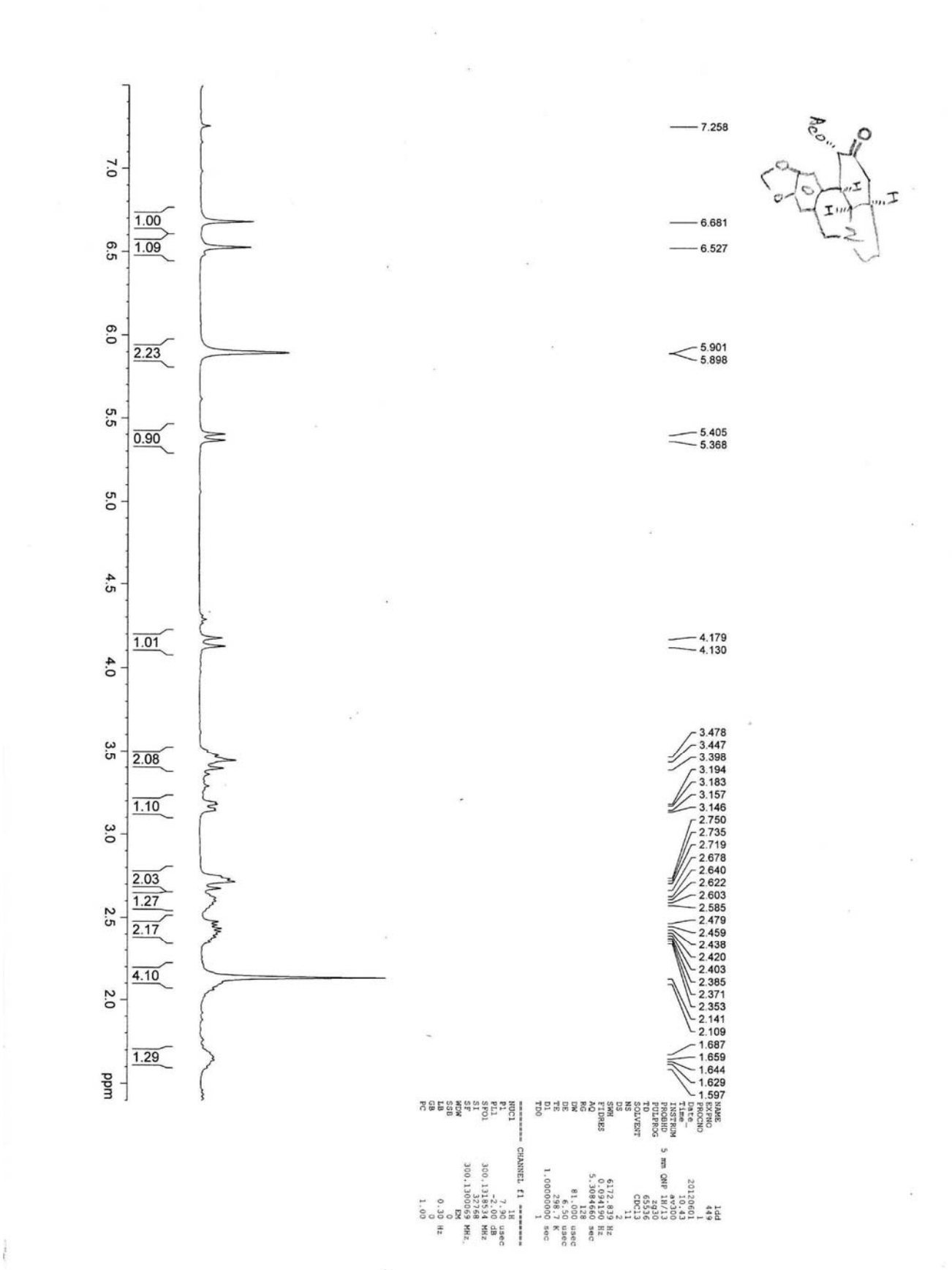
Compound 22


7.260
6.641
6.485
5.873
5.002
4.970
4.937
4.093
4.044
3.670
3.657
3.638
3.630
3.625
3.618
3.599
3.586
3.382
3.366
3.352
3.335
3.321
3.306
3.258
2.861
2.848
2.827
2.815
2.555
2.541
2.527
2.346
2.327
2.305
2.284
2.277
2.258
2.240
2.227
2.221
2.208
2.190
2.089
2.028
2.018
2.011
1.997
1.978
1.969
1.955
1.937
1.727
1.687
1.646
1.605
1.591
1.573
1.562
1.545
1.531
1.519
1.500
1.00
1.01
2.09
0.99
1.09
1.00
2.09
1.01
1.02
3.16
3.13
2.25
1.01
1.18
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
ppm

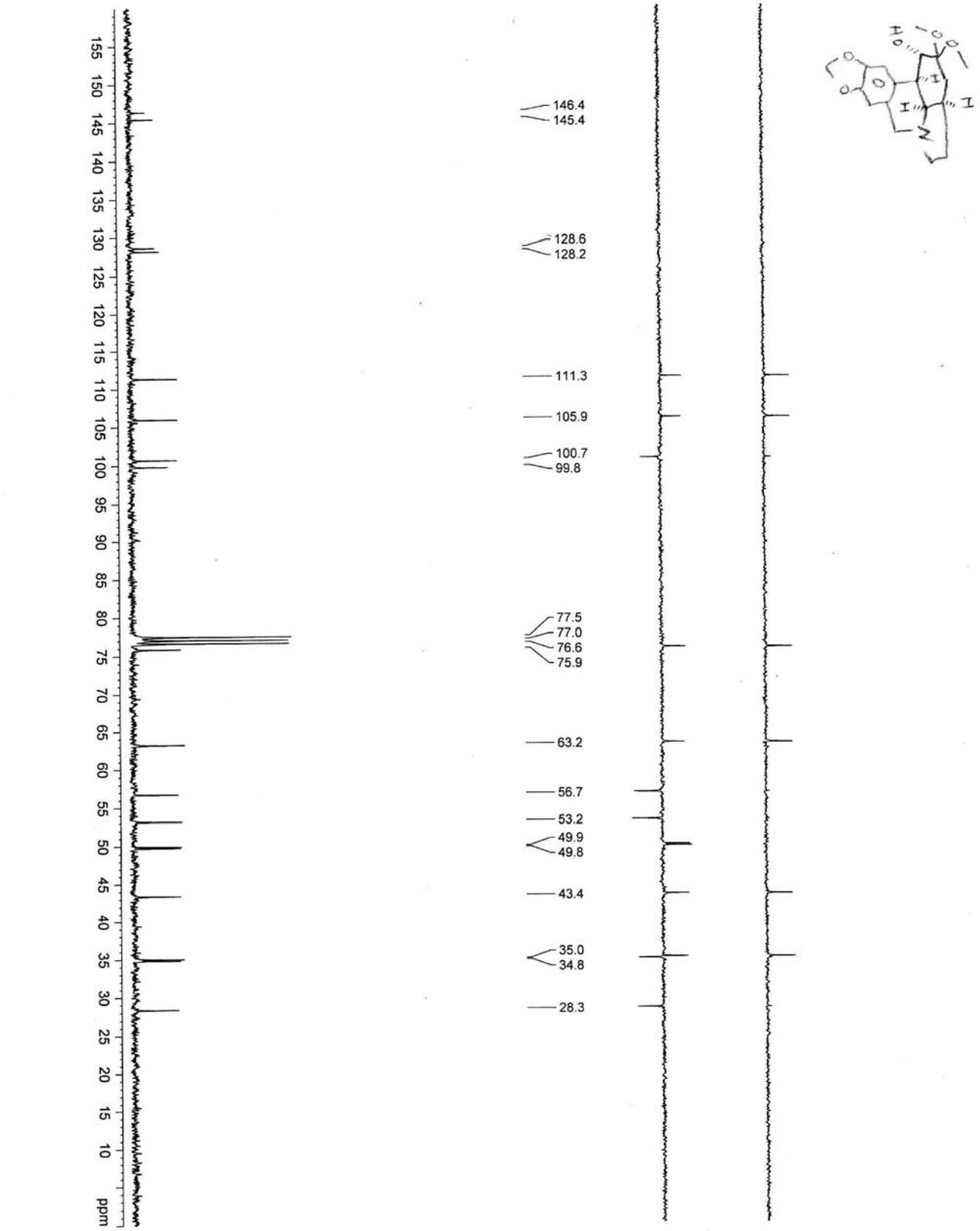
Compound 23


204.608
169.943
146.974
145.760
128.188
126.851
109.842
106.370
100.927
78.514
77.479
77.055
76.631
62.585
56.236
52.522
45.070
43.302
39.554
29.580
20.762
AcO
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

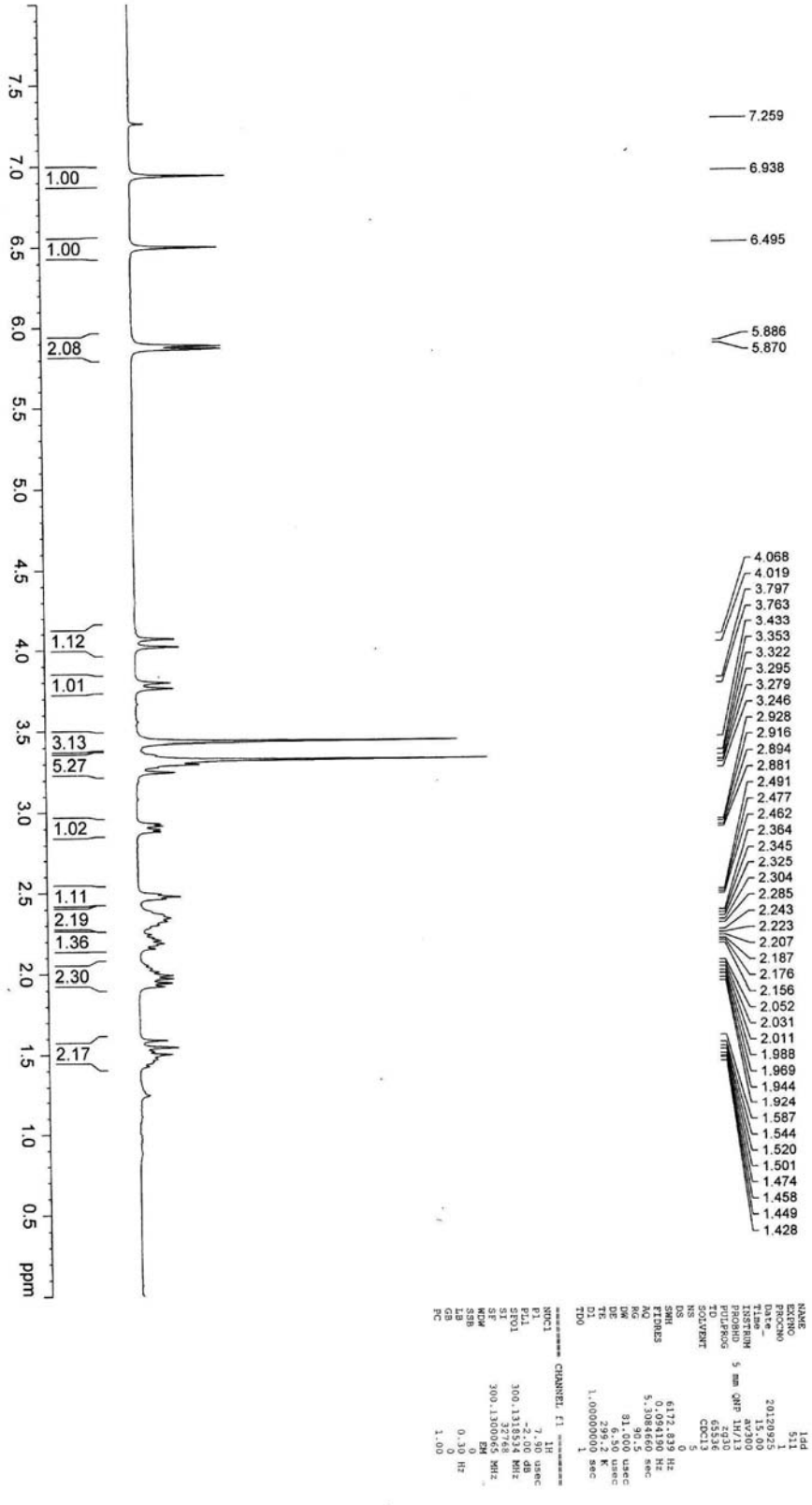
Compound 23


7.258
6.681
6.527
5.901
5.898
5.405
5.368
4.179
4.130
3.478
3.447
3.398
3.194
3.183
3.157
3.146
2.750
2.735
2.719
2.678
2.640
2.622
2.603
2.585
2.479
2.459
2.438
2.420
2.403
2.385
2.371
2.353
2.141
2.109
1.687
1.659
1.644
1.629
1.597
1.00
1.09
2.23
0.90
1.01
2.08
1.10
2.03
1.27
2.17
4.10
1.29
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm

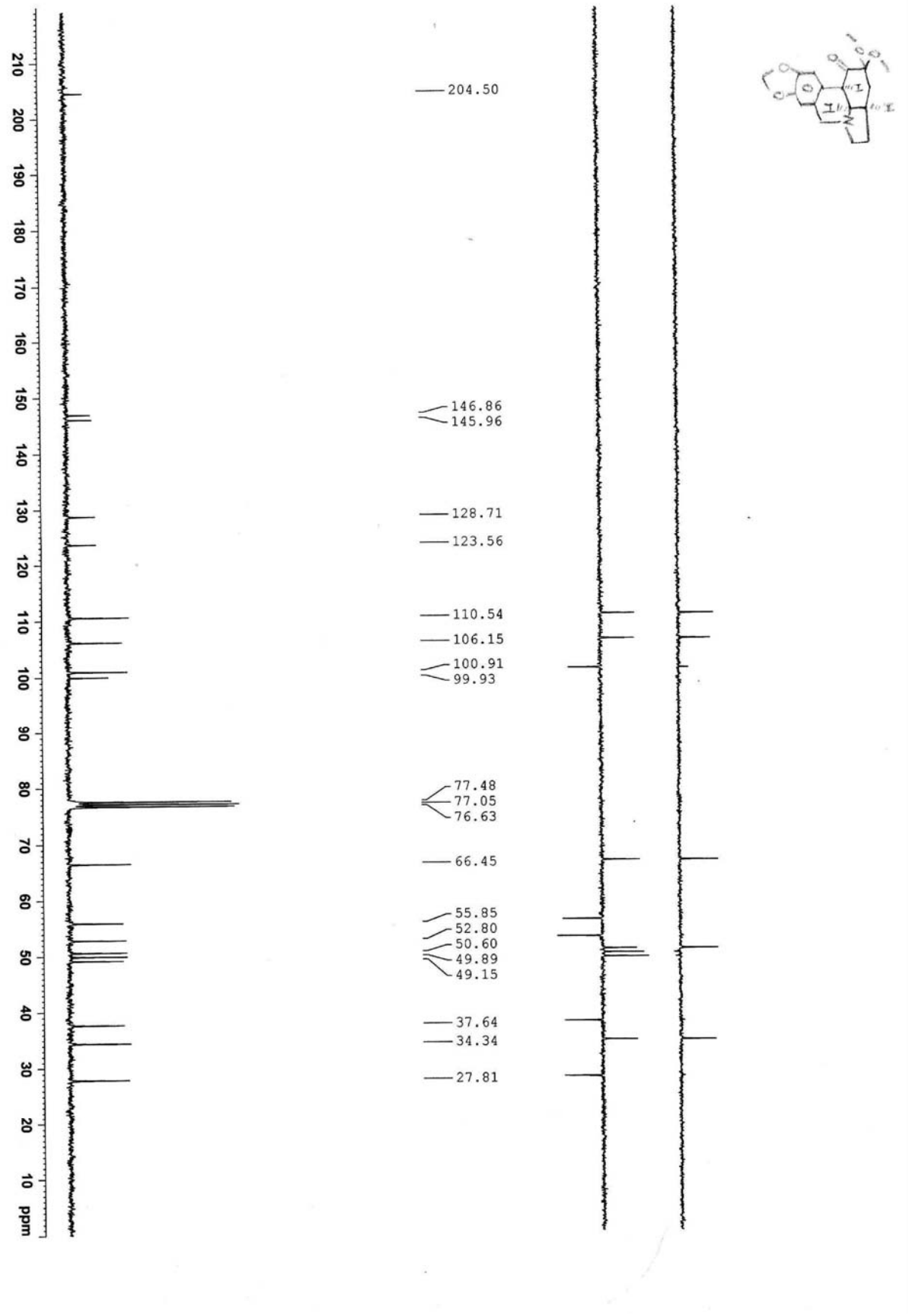
Compound 24


155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm
146.4
145.4
128.6
128.2
111.3
105.9
100.7
99.8
77.5
77.0
76.6
75.9
63.2
56.7
53.2
49.9
49.8
43.4
35.0
34.8
28.3

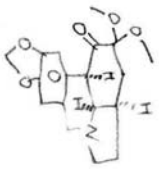
Compound 24

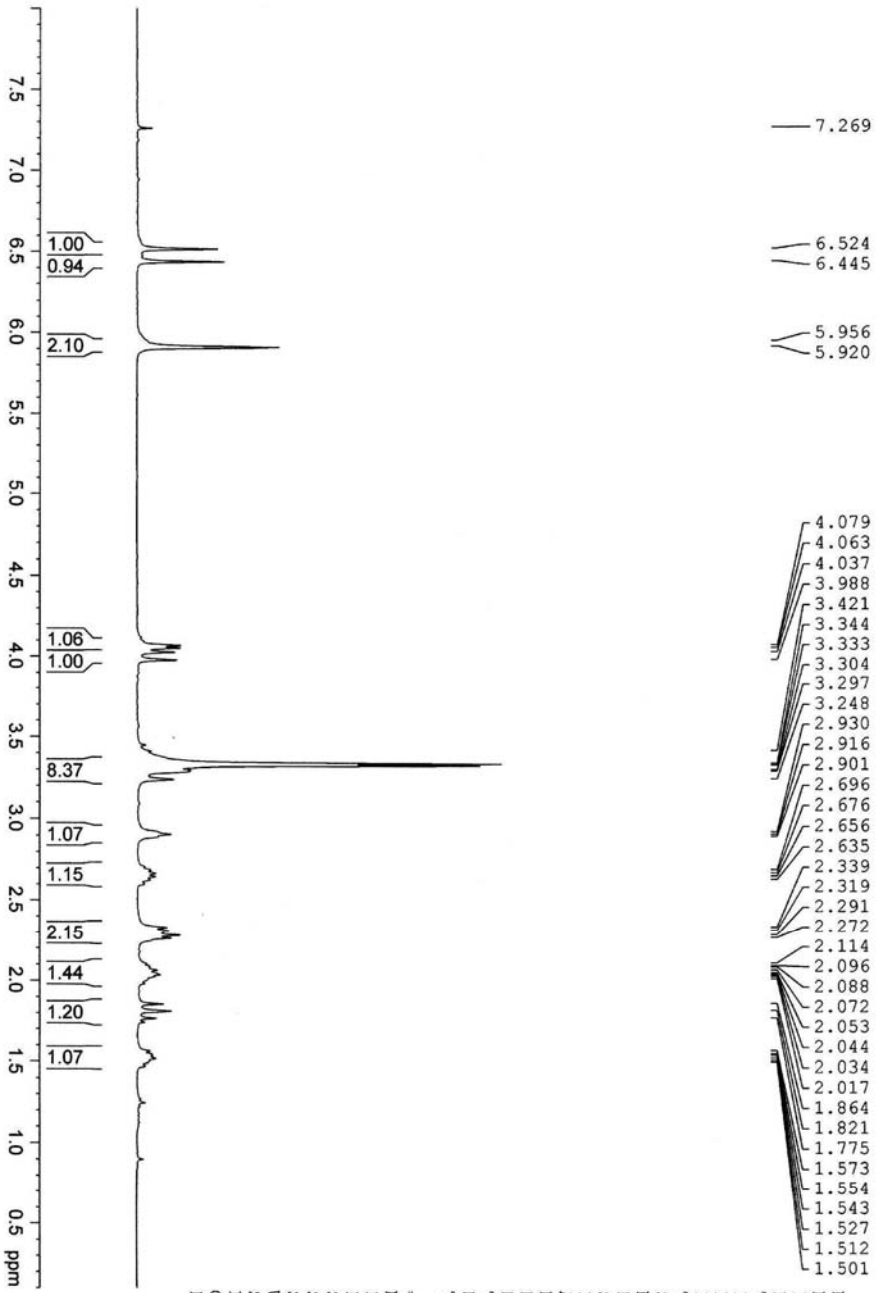

7.259
6.938
6.495
5.886
5.870
4.068
4.019
3.797
3.763
3.433
3.353
3.322
3.295
3.279
3.246
2.928
2.916
2.894
2.881
2.491
2.477
2.462
2.364
2.345
2.325
2.304
2.285
2.243
2.223
2.207
2.187
2.176
2.156
2.052
2.031
2.011
1.988
1.969
1.944
1.924
1.587
1.544
1.520
1.501
1.474
1.458
1.449
1.428
1.00
1.00
2.08
1.12
1.01
3.13
5.27
1.02
1.11
2.19
1.36
2.30
2.17
ppm

Compound 25


204.50
146.86
145.96
128.71
123.56
110.54
106.15
100.91
99.93
77.48
77.05
76.63
66.45
55.85
52.80
50.60
49.89
49.15
37.64
34.34
27.81
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

Compound 25



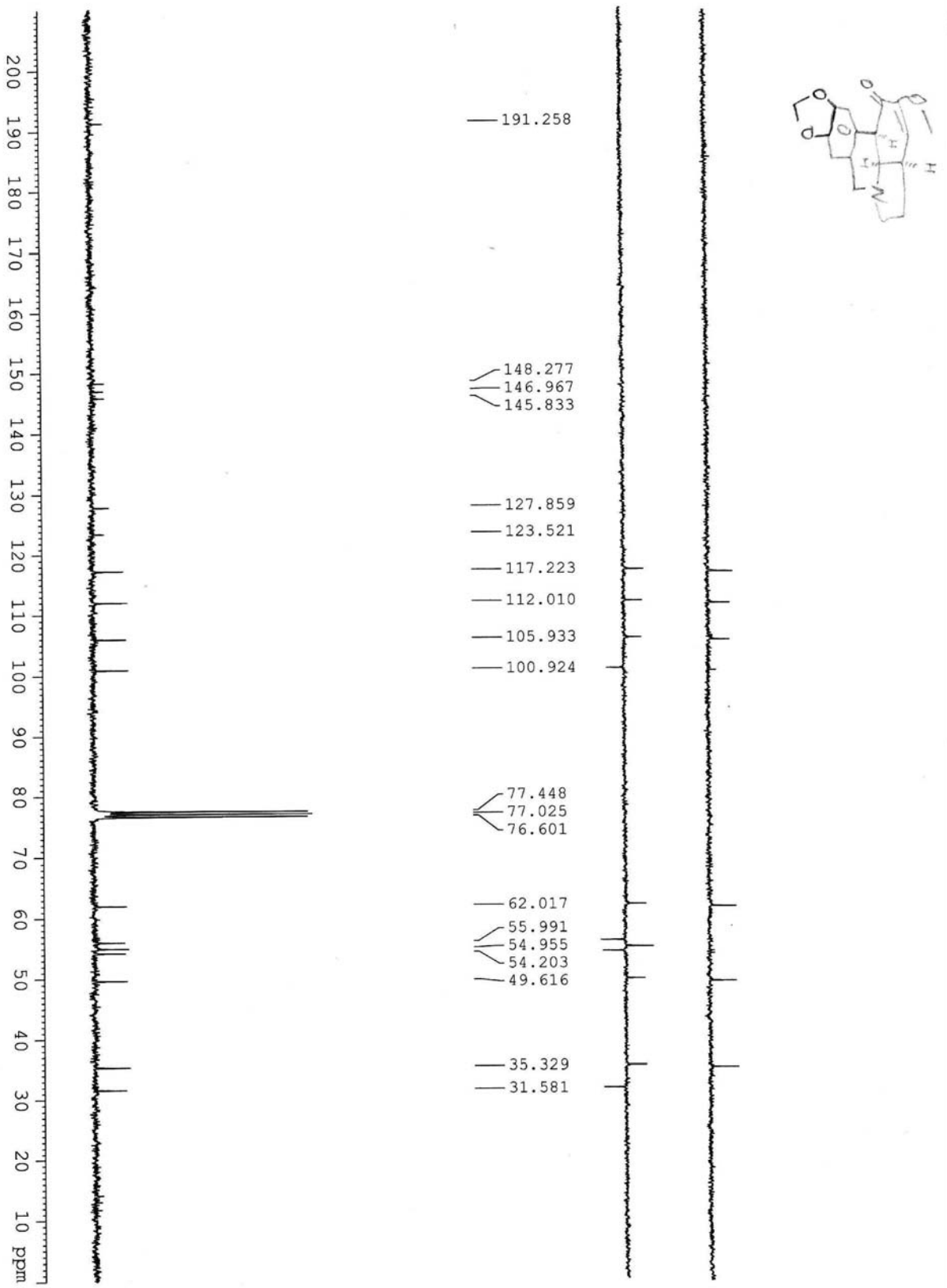




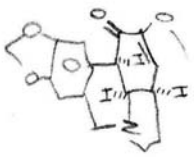
NAME 1dd
EXPNO 417
PROCNO 1
Date_ 20111222
Time_ 17.27
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

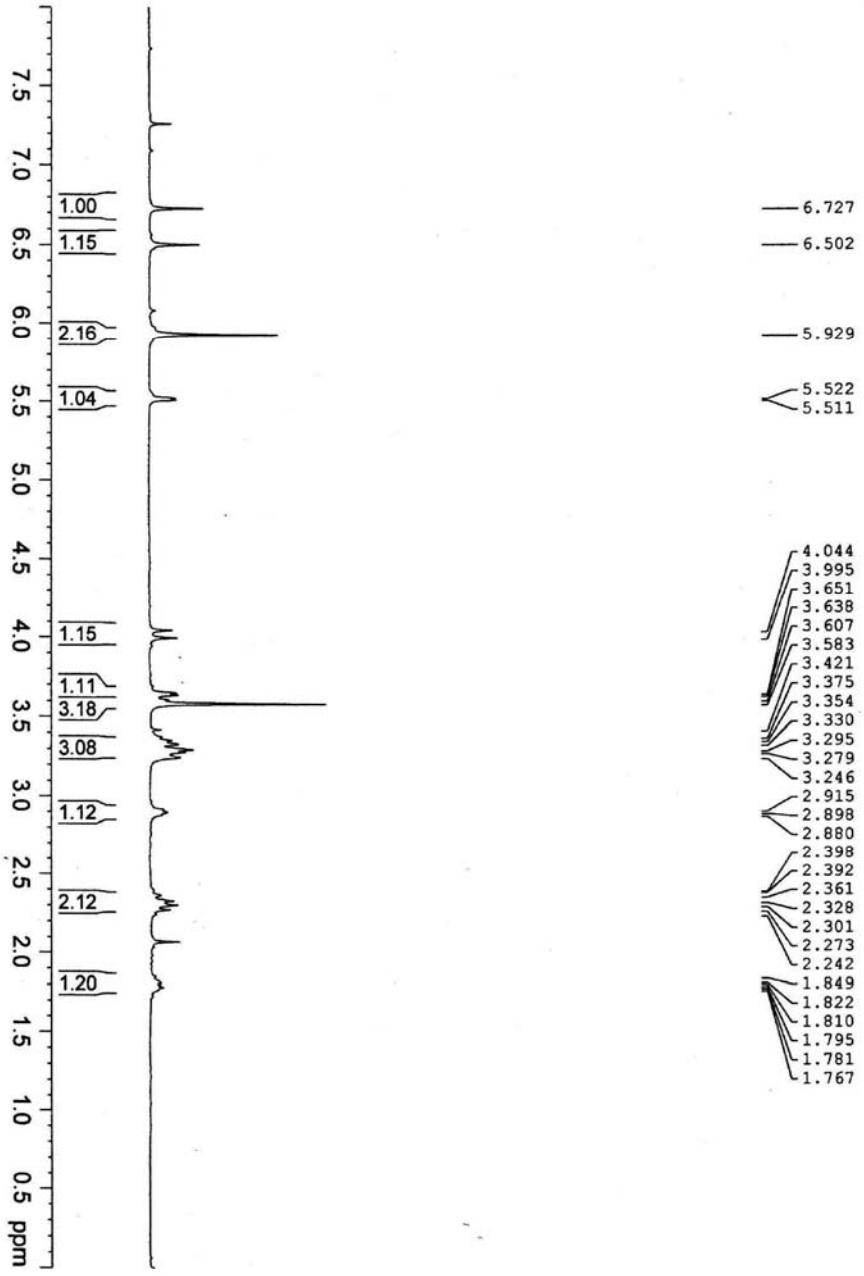
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Compound 26

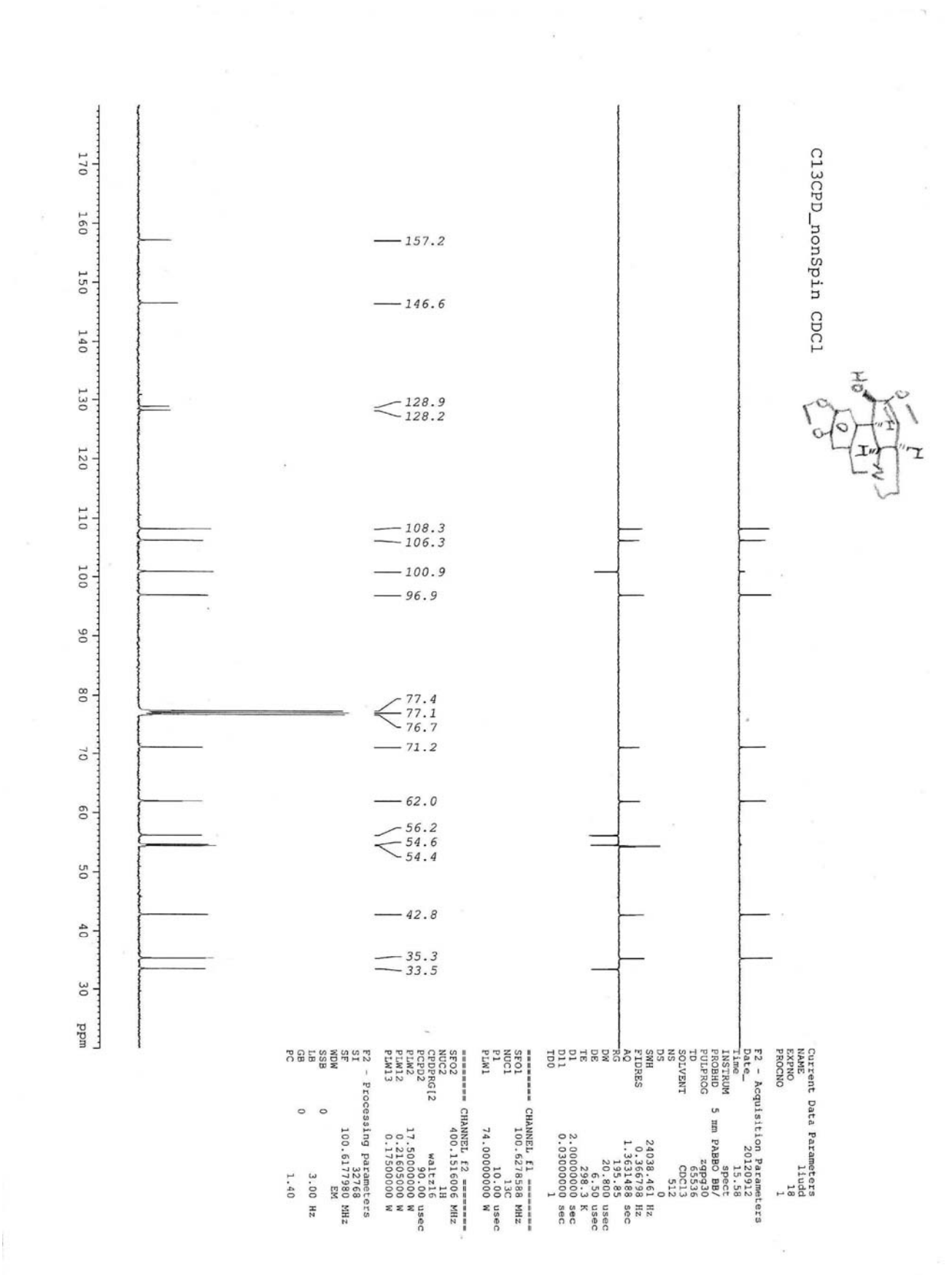

191.258
148.277
146.967
145.833
127.859
123.521
117.223
112.010
105.933
100.924
77.448
77.025
76.601
62.017
55.991
54.955
54.203
49.616
35.329
31.581
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Compound 26

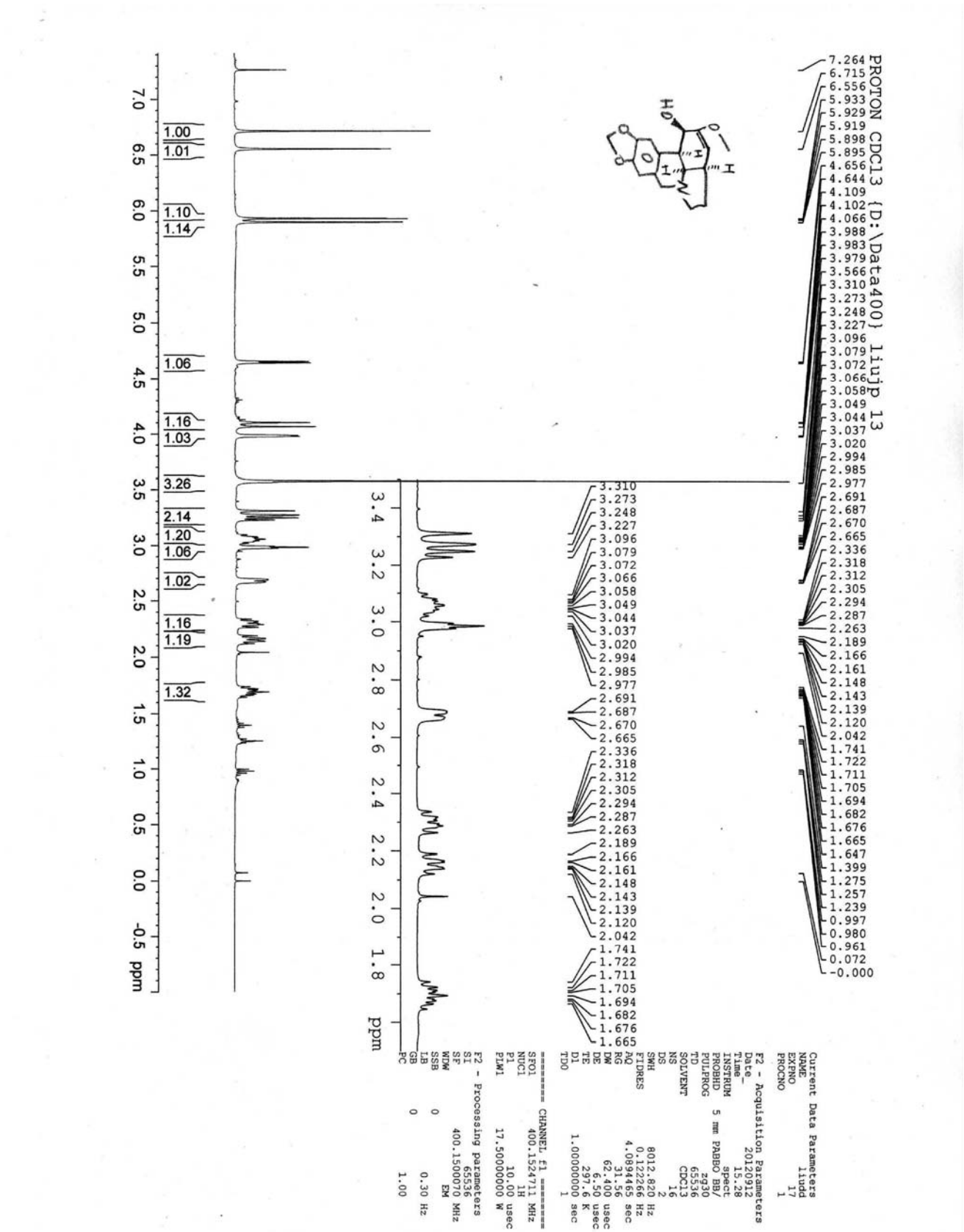



7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.15
2.16
1.04
1.15
1.11
3.18
3.08
1.12
2.12
1.20
6.727
6.502
5.929
5.522
5.511
4.044
3.995
3.651
3.638
3.607
3.583
3.421
3.375
3.354
3.330
3.295
3.279
3.246
2.915
2.898
2.880
2.398
2.392
2.361
2.328
2.301
2.273
2.242
1.849
1.822
1.810
1.795
1.781
1.767
NAME 1dd
EXPNO 470
PROCNO 1
Date_ 20120629
Time 16.27
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

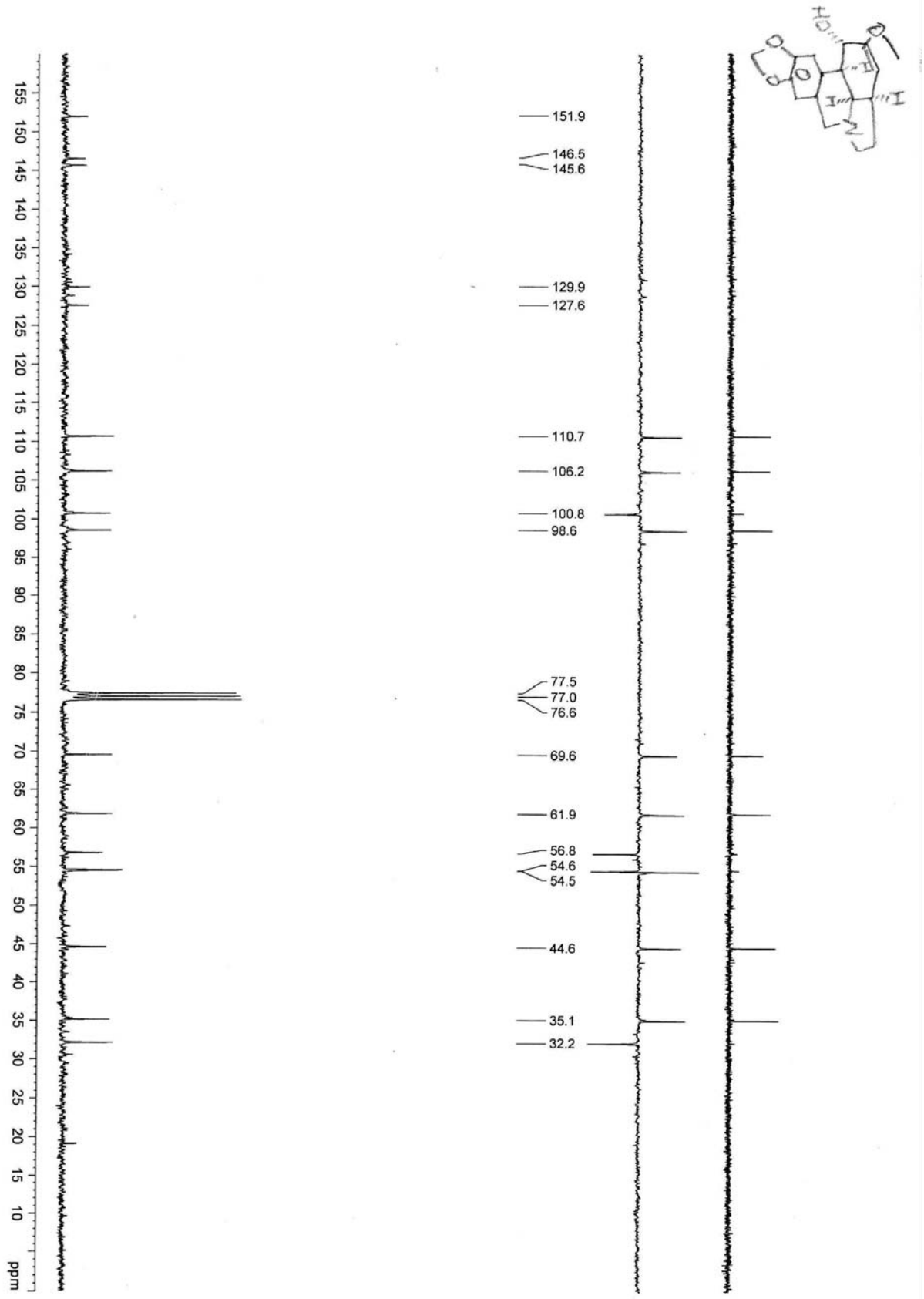
Compound 3


C13CPD_nonSpin CDCl
157.2
146.6
128.9
128.2
108.3
106.3
100.9
96.9
77.4
77.1
76.7
71.2
62.0
56.2
54.6
54.4
42.8
35.3
33.5
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
Current Data Parameters
NAME liudd
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120912
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.85
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 10.00 usec
PLW1 74.00000000 W
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 17.50000000 W
PLW12 0.21605000 W
PLW13 0.11500000 W
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

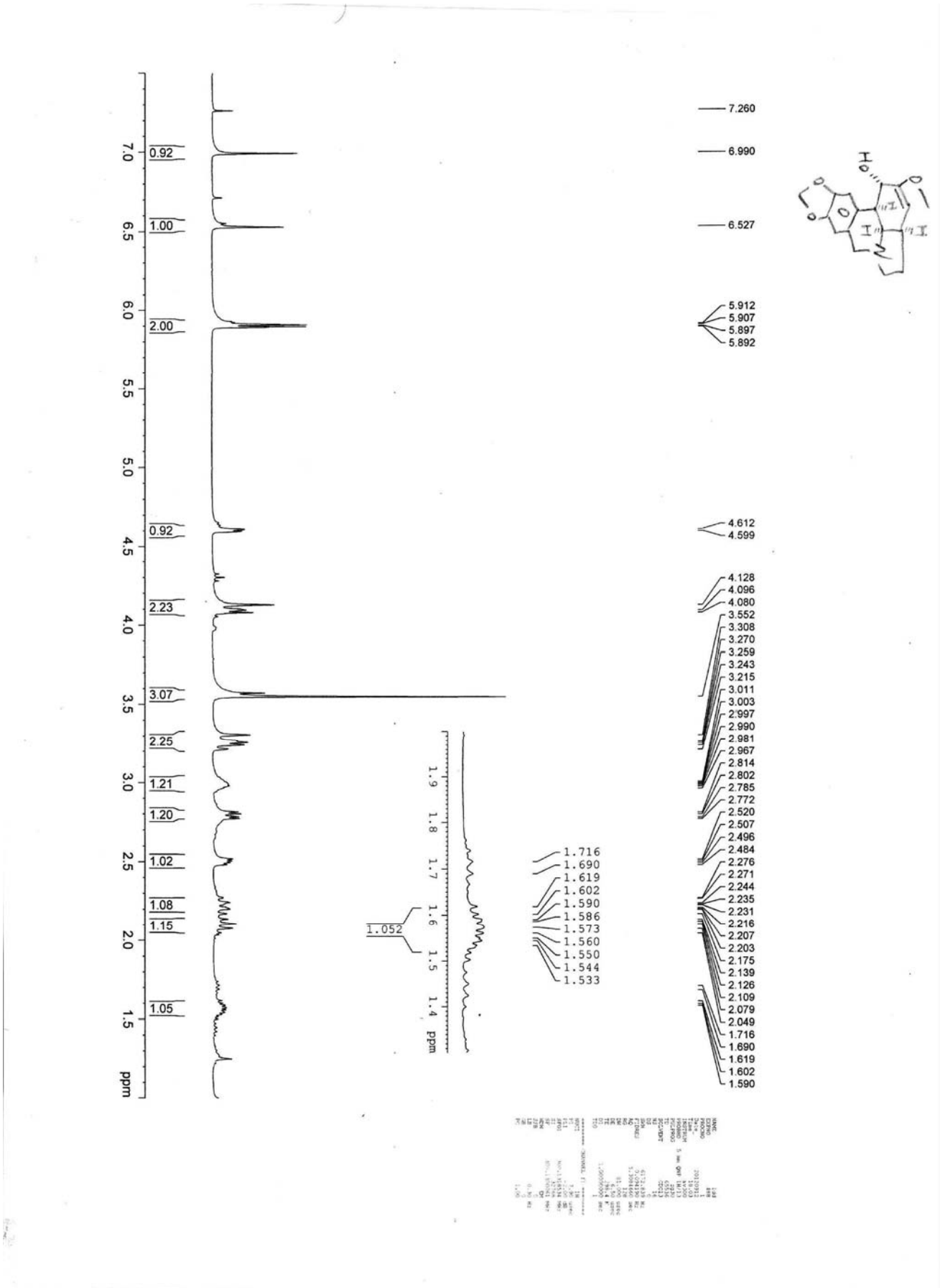
Compound 3


PROTON CDCl3 {D:\Data400} liujp 13
Current Data Parameters
NAME liudd
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120912
Time_ 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.56
DW 62.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 400.1524711 MHz
NUC1 1H
P1 10.00 usec
PLW1 17.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1500070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
1.00
1.01
1.10
1.14
1.06
1.16
1.03
3.26
2.14
1.20
1.06
1.02
1.16
1.19
1.32

Compound 27


151.9
146.5
145.6
129.9
127.6
110.7
106.2
100.8
98.6
77.5
77.0
76.6
69.6
61.9
56.8
54.6
54.5
44.6
35.1
32.2
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
ppm

Compound 27


7.260
6.990
6.527
5.912
5.907
5.897
5.892
4.612
4.599
4.128
4.096
4.080
3.552
3.308
3.270
3.259
3.243
3.215
3.011
3.003
2.997
2.990
2.981
2.967
2.814
2.802
2.785
2.772
2.520
2.507
2.496
2.484
2.276
2.271
2.244
2.235
2.231
2.216
2.207
2.203
2.175
2.139
2.126
2.109
2.079
2.049
1.716
1.690
1.619
1.602
1.590
1.716
1.690
1.619
1.602
1.590
1.586
1.573
1.560
1.550
1.544
1.533
1.052
0.92
1.00
2.00
0.92
2.23
3.07
2.25
1.21
1.20
1.02
1.08
1.15
1.05
ppm

# X-ray crystallographic data for compound 12

All data were collected on Bruker apes Ⅱ.

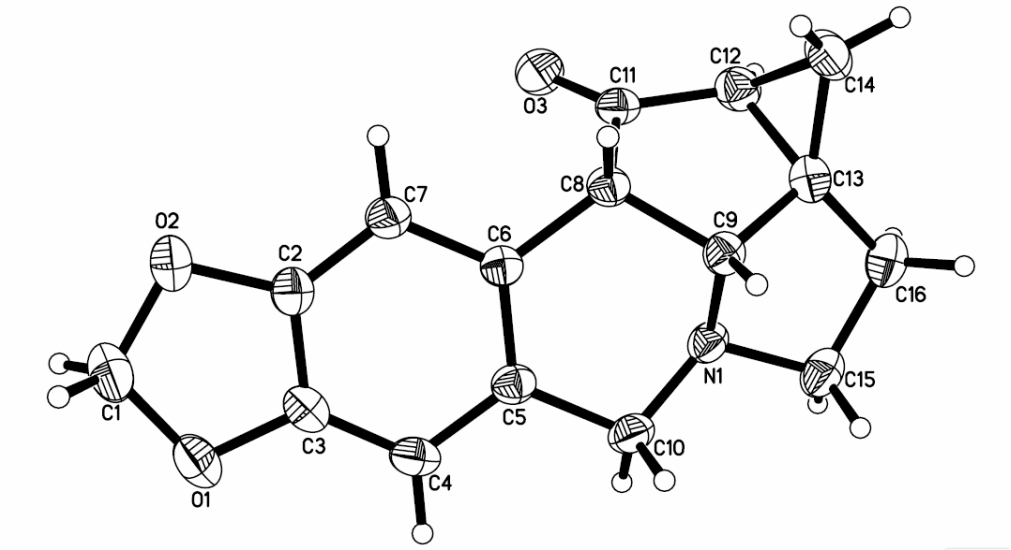



**Table 1**. Crystal data and structure refinement for compound **12**.

| | |
|---|---|
| Empirical formula | C16 H15 N O3 |
| Formula weight | 269.29 |
| Temperature | 273(2) K |
| Wavelength | 0.71073 |
| Crystal system, space group | Monoclinic, P2(1)/c |
| Unit cell dimensions | a = 10.3180(11) A α= 90 deg.<br>b = 12.8037(14) A β= 112.7060(10) deg.<br>c = 10.6038(11) A γ= 90 deg. |
| Volume | 1292.3(2) A^3 |
| Z, Calculated density | 4, 1.384 Mg/m^3 |
| Absorption coefficient | 0.096 mm^-1 |
| F(000) | 568 |
| Crystal size | 0.35 x 0.28 x 0.18 mm |
| Theta range for data collection | 2.14 to 28.43 deg. |
| Limiting indices | -13<=h<=13, -17<=k<=16, -13<=l<=14 |
| Reflections collected / unique | 10853 / 3037 [R(int) = 0.0201] |
| Completeness to theta = 28.32 | 93.5 % |
| Absorption correction | Mult-scan |
| Max. and min. transmission | 1.000000 and 0.815364 |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 3037 / 0 / 182 |
| Goodness-of-fit on F^2 | 1.044 |
| Final R indices [I>2sigma(I)] | R1 = 0.0405, wR2 = 0.1024 |
| R indices (all data) | R1 = 0.0553, wR2 = 0.1111 |
| Extinction coefficient | 0.0170(19) |
| Largest diff. peak and hole | 0.206 and -0.163 e.A^-3 |

**Table 2**. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **12**. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| C(1) | 3776(2) | 1515(1) | 4115(2) | 66(1) |
| C(2) | 6090(1) | 1273(1) | 5027(1) | 47(1) |
| C(3) | 5678(1) | 1608(1) | 6053(2) | 48(1) |
| C(4) | 6629(2) | 1754(1) | 7361(2) | 50(1) |
| C(5) | 8053(1) | 1544(1) | 7642(1) | 41(1) |
| C(6) | 8465(1) | 1216(1) | 6594(1) | 38(1) |
| C(7) | 7463(1) | 1081(1) | 5258(1) | 45(1) |
| C(8) | 9989(1) | 1016(1) | 6867(1) | 38(1) |
| C(9) | 10974(1) | 1391(1) | 8262(1) | 40(1) |
| C(10) | 9128(1) | 1682(1) | 9086(1) | 48(1) |
| C(11) | 10403(1) | -139(1) | 6885(1) | 43(1) |
| C(12) | 11868(1) | -272(1) | 7867(2) | 50(1) |
| C(13) | 12269(1) | 706(1) | 8757(1) | 47(1) |
| C(14) | 12982(2) | 495(1) | 7814(2) | 60(1) |
| C(15) | 11568(2) | 1162(1) | 10588(1) | 56(1) |
| C(16) | 12820(2) | 719(1) | 10309(2) | 57(1) |
| N(1) | 10389(1) | 1103(1) | 9261(1) | 42(1) |
| O(1) | 4241(1) | 1727(1) | 5544(1) | 69(1) |
| O(2) | 4928(1) | 1159(1) | 3824(1) | 71(1) |
| O(3) | 9619(1) | -827(1) | 6236(1) | 61(1) |

**Table 3.** Bond lengths [A] and angles [deg] for **12**

| | | | |
|---|---|---|---|
| C(1)-O(2) | 1.4139(19) | C(1)-O(1) | 1.428(2) |
| C(1)-H(1A) | 0.9700 | C(1)-H(1B) | 0.9700 |
| C(2)-C(7) | 1.3638(19) | C(2)-C(3) | 1.379(2) |
| C(2)-O(2) | 1.3802(16) | C(3)-C(4) | 1.3686(19) |
| C(3)-O(1) | 1.3766(16) | C(4)-C(5) | 1.4077(18) |
| C(4)-H(4) | 0.9300 | C(5)-C(6) | 1.3984(17) |
| C(5)-C(10) | 1.5150(18) | C(6)-C(7) | 1.4057(17) |
| C(6)-C(8) | 1.5064(17) | C(7)-H(7) | 0.9300 |
| C(8)-C(9) | 1.5135(17) | C(8)-C(11) | 1.5370(18) |
| C(8)-H(8) | 0.9800 | C(9)-N(1) | 1.4546(16) |
| C(9)-C(13) | 1.5129(18) | C(9)-H(9) | 0.9800 |
| C(10)-N(1) | 1.4447(17) | C(10)-H(10A) | 0.9700 |
| C(10)-H(10B) | 0.9700 | C(11)-O(3) | 1.2138(16) |
| C(11)-C(12) | 1.4776(19) | C(12)-C(13) | 1.526(2) |
| C(12)-C(14) | 1.529(2) | C(12)-H(12) | 0.9800 |
| C(13)-C(14) | 1.477(2) | C(13)-C(16) | 1.519(2) |
| C(14)-H(14A) | 0.9700 | C(14)-H(14B) | 0.9700 |
| C(15)-N(1) | 1.4644(16) | C(15)-C(16) | 1.541(2) |
| C(15)-H(15A) | 0.9700 | C(15)-H(15B) | 0.9700 |
| C(16)-H(16A) | 0.9700 | C(16)-H(16B) | 0.9700 |
| O(2)-C(1)-O(1) | 109.05(12) | O(2)-C(1)-H(1A) | 109.9 |
| O(1)-C(1)-H(1A) | 109.9 | O(2)-C(1)-H(1B) | 109.9 |
| O(1)-C(1)-H(1B) | 109.9 | H(1A)-C(1)-H(1B) | 108.3 |
| C(7)-C(2)-C(3) | 121.95(13) | C(7)-C(2)-O(2) | 128.17(13) |
| C(3)-C(2)-O(2) | 109.88(12) | C(4)-C(3)-O(1) | 128.63(13) |
| C(4)-C(3)-C(2) | 121.56(13) | O(1)-C(3)-C(2) | 109.78(13) |
| C(3)-C(4)-C(5) | 117.99(12) | C(3)-C(4)-H(4) | 121.0 |
| C(5)-C(4)-H(4) | 121.0 | C(6)-C(5)-C(4) | 120.15(12) |
| C(6)-C(5)-C(10) | 120.59(12) | C(4)-C(5)-C(10) | 119.25(12) |
| C(5)-C(6)-C(7) | 120.42(12) | C(5)-C(6)-C(8) | 120.89(11) |
| C(7)-C(6)-C(8) | 118.69(11) | C(2)-C(7)-C(6) | 117.91(12) |

| | | | |
|---|---|---|---|
| C(2)-C(7)-H(7) | 121.0 | C(6)-C(7)-H(7) | 121.0 |
| C(6)-C(8)-C(9) | 113.01(10) | C(6)-C(8)-C(11) | 115.52(10) |
| C(9)-C(8)-C(11) | 102.41(10) | C(6)-C(8)-H(8) | 108.5 |
| C(9)-C(8)-H(8) | 108.5 | C(11)-C(8)-H(8) | 108.5 |
| N(1)-C(9)-C(13) | 99.17(10) | N(1)-C(9)-C(8) | 108.68(10) |
| C(13)-C(9)-C(8) | 108.95(11) | N(1)-C(9)-H(9) | 113.0 |
| C(13)-C(9)-H(9) | 113.0 | C(8)-C(9)-H(9) | 113.0 |
| N(1)-C(10)-C(5) | 110.00(10) | N(1)-C(10)-H(10A) | 109.7 |
| C(5)-C(10)-H(10A) | 109.7 | N(1)-C(10)-H(10B) | 109.7 |
| C(5)-C(10)-H(10B) | 109.7 | H(10A)-C(10)-H(10B) | 108.2 |
| O(3)-C(11)-C(12) | 126.36(13) | O(3)-C(11)-C(8) | 124.71(12) |
| C(12)-C(11)-C(8) | 108.86(11) | C(11)-C(12)-C(13) | 107.50(11) |
| C(11)-C(12)-C(14) | 118.66(12) | C(13)-C(12)-C(14) | 57.84(9) |
| C(11)-C(12)-H(12) | 118.9 | C(13)-C(12)-H(12) | 118.9 |
| C(14)-C(12)-H(12) | 118.9 | C(14)-C(13)-C(9) | 118.35(12) |
| C(14)-C(13)-C(16) | 131.21(13) | C(9)-C(13)-C(16) | 106.30(12) |
| C(14)-C(13)-C(12) | 61.20(10) | C(9)-C(13)-C(12) | 105.61(11) |
| C(16)-C(13)-C(12) | 125.31(12) | C(13)-C(14)-C(12) | 60.96(9) |
| C(13)-C(14)-H(14A) | 117.7 | C(12)-C(14)-H(14A) | 117.7 |
| C(13)-C(14)-H(14B) | 117.7 | C(12)-C(14)-H(14B) | 117.7 |
| H(14A)-C(14)-H(14B) | 114.8 | N(1)-C(15)-C(16) | 104.05(11) |
| N(1)-C(15)-H(15A) | 110.9 | C(16)-C(15)-H(15A) | 110.9 |
| N(1)-C(15)-H(15B) | 110.9 | C(16)-C(15)-H(15B) | 110.9 |
| H(15A)-C(15)-H(15B) | 109.0 | C(13)-C(16)-C(15) | 102.79(11) |
| C(13)-C(16)-H(16A) | 111.2 | C(15)-C(16)-H(16A) | 111.2 |
| C(13)-C(16)-H(16B) | 111.2 | C(15)-C(16)-H(16B) | 111.2 |
| H(16A)-C(16)-H(16B) | 109.1 | C(10)-N(1)-C(9) | 112.49(10) |
| C(10)-N(1)-C(15) | 118.01(11) | C(9)-N(1)-C(15) | 105.23(10) |
| C(3)-O(1)-C(1) | 105.33(11) | C(2)-O(2)-C(1) | 105.51(12) |

**Table 4**. Anisotropic displacement parameters (A^2 x 10^3) for **12**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a*b*U12]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|

| | | | | | | |
|---|---|---|---|---|---|---|
| C(1) | 44(1) | 77(1) | 68(1) | 11(1) | 14(1) | 9(1) |
| C(2) | 44(1) | 50(1) | 45(1) | 3(1) | 13(1) | 3(1) |
| C(3) | 41(1) | 47(1) | 60(1) | 4(1) | 22(1) | 5(1) |
| C(4) | 53(1) | 49(1) | 56(1) | -6(1) | 31(1) | 3(1) |
| C(5) | 46(1) | 36(1) | 43(1) | -3(1) | 21(1) | -3(1) |
| C(6) | 41(1) | 36(1) | 38(1) | 1(1) | 18(1) | -1(1) |
| C(7) | 46(1) | 53(1) | 39(1) | 1(1) | 18(1) | 4(1) |
| C(8) | 40(1) | 42(1) | 36(1) | 3(1) | 19(1) | -1(1) |
| C(9) | 41(1) | 39(1) | 42(1) | 0(1) | 17(1) | -6(1) |
| C(10) | 53(1) | 53(1) | 43(1) | -13(1) | 24(1) | -9(1) |
| C(11) | 47(1) | 46(1) | 42(1) | -3(1) | 24(1) | 1(1) |
| C(12) | 50(1) | 49(1) | 53(1) | 2(1) | 20(1) | 7(1) |
| C(13) | 41(1) | 49(1) | 48(1) | 2(1) | 14(1) | -2(1) |
| C(14) | 42(1) | 76(1) | 64(1) | 4(1) | 23(1) | 4(1) |
| C(15) | 59(1) | 63(1) | 39(1) | -5(1) | 13(1) | -10(1) |
| C(16) | 52(1) | 61(1) | 47(1) | 2(1) | 7(1) | -6(1) |
| N(1) | 45(1) | 48(1) | 34(1) | -4(1) | 16(1) | -7(1) |
| O(1) | 43(1) | 88(1) | 76(1) | 2(1) | 22(1) | 13(1) |
| O(2) | 44(1) | 103(1) | 54(1) | -4(1) | 5(1) | 11(1) |
| O(3) | 60(1) | 51(1) | 70(1) | -18(1) | 24(1) | -5(1) |

**Table 5**. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **12**.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H(1A) | 3046 | 987 | 3854 | 79 |
| H(1B) | 3391 | 2145 | 3597 | 79 |
| H(4) | 6344 | 1986 | 8045 | 60 |
| H(7) | 7727 | 866 | 4554 | 55 |
| H(8) | 10224 | 1379 | 6170 | 46 |
| H(9) | 11194 | 2137 | 8279 | 48 |
| H(10A) | 9353 | 2416 | 9264 | 57 |
| H(10B) | 8743 | 1434 | 9733 | 57 |
| H(12) | 12168 | -956 | 8296 | 61 |

| | | | | |
|---|---|---|---|---|
| H(14A) | 13956 | 274 | 8210 | 72 |
| H(14B) | 12743 | 935 | 7011 | 72 |
| H(15A) | 11385 | 746 | 11267 | 67 |
| H(15B) | 11744 | 1879 | 10907 | 67 |
| H(16A) | 13639 | 1165 | 10692 | 68 |
| H(16B) | 13059 | 21 | 10682 | 68 |

# X-ray crystallographic data for compound 16

All data were collected on Bruker apes Ⅱ.

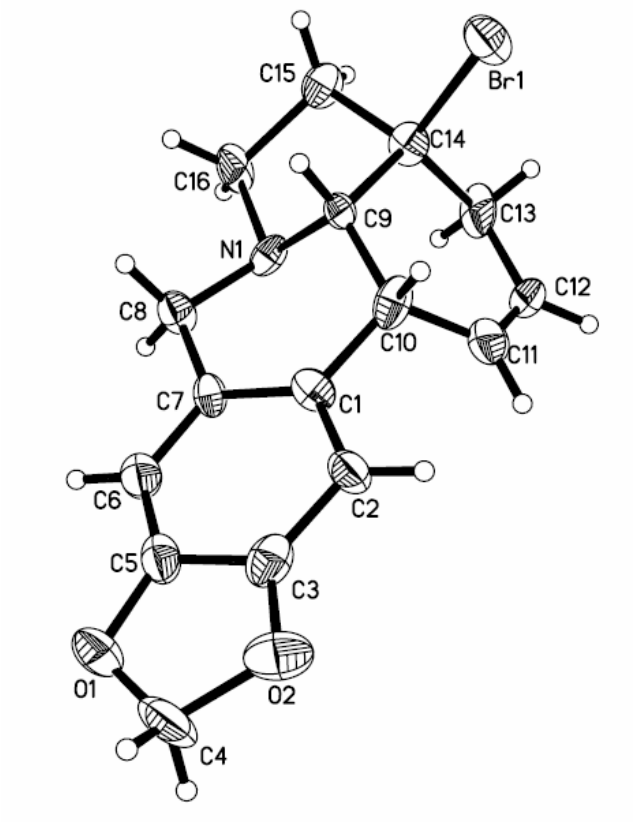



**Table 1**. Crystal data and structure refinement for compound **16**.

| | |
|---|---|
| Empirical formula | C16 H16 Br N O2 |
| Formula weight | 334.21 |
| Temperature | 298(2) K |
| Wavelength | 0.71073 |
| Crystal system, space group | Triclinic, P1 |
| Unit cell dimensions | a = 7.7674 (11) A  α = 103.452(2) deg.<br>b = 9.4798 (13) A  β = 95.382(2) deg.<br>c = 9.7608 (14) A  γ = 93.343(2) deg. |
| Volume | 693.53(17) A^3 |
| Z, Calculated density | 2,  1.600 Mg/m^3 |
| Absorption coefficient | 2.964 mm^-1 |
| F(000) | 340 |
| Crystal size | 0.21 x 0.14 x 0.10 mm |
| Theta range for data collection | 2.16 to 28.23 deg. |
| Limiting indices | -10<=h<=9, -12<=k<=12, -12<=l<=12 |

| | |
|---|---|
| Reflections collected / unique | 6027 / 5439 [R(int) = 0.0147] |
| Completeness to theta = 28.32 | 91.8 % |
| Absorption correction | Mulit-scan |
| Max. and min. transmission | 0.7559 and 0.5749 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 5439 / 3 / 362 |
| Goodness-of-fit on F^2 | 1.007 |
| Final R indices [I>2sigma(I)] | R1 = 0.0432, wR2 = 0.0776 |
| R indices (all data) | R1 = 0.0756, wR2 = 0.0903 |
| Absolute structure parameter | 0.00 |
| Extinction coefficien | 0.0003(10) |
| Largest diff. peak and hole | 0.381 and -0.367 e.A^-3 |

**Table 2**. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **16**. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| N(1) | 4393(13) | 2027(10) | 708(10) | 39(2) |
| N(2) | 6064(13) | 7267(11) | 4511(12) | 48(3) |
| O(1) | 7318(15) | 7393(12) | -1112(12) | 73(4) |
| O(2) | 4453(17) | 7915(11) | -1419(12) | 75(3) |
| O(3) | 6086(14) | 1378(12) | 6695(13) | 71(3) |
| O(4) | 3215(17) | 1943(13) | 6360(13) | 81(4) |
| Br(1) | 3(1) | 2061(1) | 2597(1) | 67(1) |
| Br(2) | 10560(1) | 7193(1) | 2627(1) | 66(1) |
| C(1) | 3670(20) | 4585(14) | -29(15) | 44(3) |
| C(2) | 3184(17) | 5763(14) | -583(14) | 53(3) |
| C(3) | 4530(20) | 6691(15) | -923(16) | 58(4) |
| C(4) | 6270(20) | 8222(17) | -1702(16) | 81(5) |
| C(5) | 6291(19) | 6331(16) | -738(16) | 52(4) |
| C(6) | 6630(20) | 5087(16) | -269(17) | 53(3) |
| C(7) | 5420(16) | 4228(13) | 100(15) | 39(3) |
| C(8) | 5933(18) | 2982(15) | 717(16) | 47(3) |
| C(9) | 2901(16) | 2748(13) | 1301(13) | 41(3) |

| | | | | |
|---|---|---|---|---|
| C(10) | 2246(17) | 3724(13) | 382(14) | 50(3) |
| C(11) | 986(18) | 2938(18) | -806(16) | 59(4) |
| C(12) | 339(18) | 1584(17) | -1010(13) | 61(4) |
| C(13) | 940(18) | 557(14) | -124(15) | 62(4) |
| C(14) | 1736(18) | 1367(12) | 1339(14) | 43(3) |
| C(15) | 3084(18) | 464(15) | 2091(16) | 56(3) |
| C(16) | 4820(15) | 973(14) | 1551(16) | 58(4) |
| C(17) | 7514(15) | 6525(10) | 3965(12) | 37(3) |
| C(18) | 5240(20) | 4894(14) | 5123(14) | 46(3) |
| C(19) | 8667(17) | 7788(14) | 3824(15) | 48(3) |
| C(20) | 6896(19) | 4597(14) | 5282(14) | 41(3) |
| C(21) | 4350(20) | 2931(16) | 6004(16) | 55(4) |
| C(22) | 8363(15) | 5556(13) | 4840(12) | 38(3) |
| C(23) | 4672(19) | 6161(15) | 4512(15) | 49(3) |
| C(24) | 5970(20) | 2632(13) | 6169(15) | 48(4) |
| C(25) | 9607(17) | 6410(16) | 6101(14) | 49(3) |
| C(26) | 7263(17) | 3352(13) | 5797(13) | 48(3) |
| C(27) | 5699(18) | 8280(13) | 3588(14) | 57(4) |
| C(28) | 3790(20) | 4019(16) | 5475(16) | 50(3) |
| C(29) | 10158(19) | 7798(15) | 6252(16) | 64(4) |
| C(30) | 7490(20) | 8671(15) | 3232(18) | 62(4) |
| C(31) | 4430(20) | 1026(14) | 6969(12) | 67(5) |
| C(32) | 9567(18) | 8664(13) | 5246(15) | 60(4) |

**Table 3.** Bond lengths [A] and angles [deg] for **16**.

| | | | |
|---|---|---|---|
| N(1)-C(8) | 1.456(16) | N(1)-C(9) | 1.476(15) |
| N(1)-C(16) | 1.468(16) | N(2)-C(17) | 1.442(15) |
| N(2)-C(23) | 1.462(17) | N(2)-C(27) | 1.483(16) |
| O(1)-C(4) | 1.344(17) | O(1)-C(5) | 1.384(19) |
| O(2)-C(3) | 1.360(16) | O(2)-C(4) | 1.489(18) |
| O(3)-C(31) | 1.378(19) | O(3)-C(24) | 1.405(14) |
| O(4)-C(21) | 1.37(2) | O(4)-C(31) | 1.492(16) |

| | | | |
|---|---|---|---|
| Br(1)-C(14) | 1.946(14) | Br(2)-C(19) | 1.990(13) |
| C(1)-C(2) | 1.406(19) | C(1)-C(7) | 1.419(19) |
| C(1)-C(10) | 1.48(2) | C(2)-C(3) | 1.44(2) |
| C(2)-H(2) | 0.9300 | C(3)-C(5) | 1.43(2) |
| C(4)-H(4A) | 0.9700 | C(4)-H(4B) | 0.9700 |
| C(5)-C(6) | 1.39(2) | C(6)-C(7) | 1.34(2) |
| C(6)-H(6) | 0.9300 | C(7)-C(8) | 1.503(18) |
| C(8)-H(8A) | 0.9700 | C(8)-H(8B) | 0.9700 |
| C(9)-C(10) | 1.508(17) | C(9)-C(14) | 1.558(17) |
| C(9)-H(9) | 0.9800 | C(10)-C(11) | 1.47(2) |
| C(10)-H(10) | 0.9800 | C(11)-C(12) | 1.31(2) |
| C(11)-H(11) | 0.9300 | C(12)-C(13) | 1.51(2) |
| C(12)-H(12) | 0.9300 | C(13)-C(14) | 1.510(18) |
| C(13)-H(13A) | 0.9700 | C(13)-H(13B) | 0.9700 |
| C(14)-C(15) | 1.619(16) | C(15)-C(16) | 1.582(19) |
| C(15)-H(15A) | 0.9700 | C(15)-H(15B) | 0.9700 |
| C(16)-H(16A) | 0.9700 | C(16)-H(16B) | 0.9700 |
| C(17)-C(19) | 1.493(17) | C(17)-C(22) | 1.529(16) |
| C(17)-H(17) | 0.9800 | C(18)-C(20) | 1.34(2) |
| C(18)-C(28) | 1.47(2) | C(18)-C(23) | 1.529(18) |
| C(19)-C(30) | 1.444(18) | C(19)-C(32) | 1.522(19) |
| C(20)-C(26) | 1.419(17) | C(20)-C(22) | 1.570(18) |
| C(21)-C(24) | 1.31(2) | C(21)-C(28) | 1.33(2) |
| C(22)-C(25) | 1.526(18) | C(22)-H(22) | 0.9800 |
| C(23)-H(23A) | 0.9700 | C(23)-H(23B) | 0.9700 |
| C(24)-C(26) | 1.31(2) | C(25)-C(29) | 1.33(2) |
| C(25)-H(25) | 0.9300 | C(26)-H(26) | 0.9300 |
| C(27)-C(30) | 1.51(2) | C(27)-H(27A) | 0.9700 |
| C(27)-H(27B) | 0.9700 | C(28)-H(28) | 0.9300 |
| C(29)-C(32) | 1.48(2) | C(29)-H(29) | 0.9300 |
| C(30)-H(30A) | 0.9700 | C(30)-H(30B) | 0.9700 |
| C(31)-H(31A) | 0.9700 | C(31)-H(31B) | 0.9700 |

| | | | |
|---|---|---|---|
| C(32)-H(32A) | 0.9700 | C(32)-H(32B) | 0.9700 |
| C(8)-N(1)-C(9) | 115.8(9) | C(8)-N(1)-C(16) | 109.5(9) |
| C(9)-N(1)-C(16) | 106.0(10) | C(17)-N(2)-C(23) | 107.7(9) |
| C(17)-N(2)-C(27) | 104.5(10) | C(23)-N(2)-C(27) | 116.6(10) |
| C(4)-O(1)-C(5) | 108.2(12) | C(3)-O(2)-C(4) | 103.1(13) |
| C(31)-O(3)-C(24) | 105.0(11) | C(21)-O(4)-C(31) | 101.7(12) |
| C(2)-C(1)-C(7) | 122.0(13) | C(2)-C(1)-C(10) | 115.8(13) |
| C(7)-C(1)-C(10) | 122.2(11) | C(1)-C(2)-C(3) | 118.1(13) |
| C(1)-C(2)-H(2) | 120.9 | C(3)-C(2)-H(2) | 121.0 |
| O(2)-C(3)-C(5) | 110.6(15) | O(2)-C(3)-C(2) | 130.9(15) |
| C(5)-C(3)-C(2) | 118.5(13) | O(1)-C(4)-O(2) | 109.5(10) |
| O(1)-C(4)-H(4A) | 109.8 | O(2)-C(4)-H(4A) | 109.8 |
| O(1)-C(4)-H(4B) | 109.8 | O(2)-C(4)-H(4B) | 109.8 |
| H(4A)-C(4)-H(4B) | 108.2 | C(6)-C(5)-O(1) | 133.9(15) |
| C(6)-C(5)-C(3) | 119.1(15) | O(1)-C(5)-C(3) | 107.0(12) |
| C(7)-C(6)-C(5) | 124.0(15) | C(7)-C(6)-H(6) | 118.0 |
| C(5)-C(6)-H(6) | 118.0 | C(6)-C(7)-C(1) | 118.1(12) |
| C(6)-C(7)-C(8) | 120.2(13) | C(1)-C(7)-C(8) | 121.6(12) |
| N(1)-C(8)-C(7) | 109.2(11) | N(1)-C(8)-H(8A) | 109.9 |
| C(7)-C(8)-H(8A) | 109.8 | N(1)-C(8)-H(8B) | 109.8 |
| C(7)-C(8)-H(8B) | 109.9 | H(8A)-C(8)-H(8B) | 108.3 |
| N(1)-C(9)-C(10) | 109.3(10) | N(1)-C(9)-C(14) | 98.7(9) |
| C(10)-C(9)-C(14) | 118.7(11) | N(1)-C(9)-H(9) | 109.8 |
| C(10)-C(9)-H(9) | 109.9 | C(14)-C(9)-H(9) | 109.8 |
| C(11)-C(10)-C(1) | 114.5(12) | C(11)-C(10)-C(9) | 112.2(12) |
| C(1)-C(10)-C(9) | 112.3(11) | C(11)-C(10)-H(10) | 105.6 |
| C(1)-C(10)-H(10) | 105.7 | C(9)-C(10)-H(10) | 105.7 |
| C(12)-C(11)-C(10) | 125.9(14) | C(12)-C(11)-H(11) | 117.2 |
| C(10)-C(11)-H(11) | 117.0 | C(11)-C(12)-C(13) | 124.0(12) |
| C(11)-C(12)-H(12) | 118.0 | C(13)-C(12)-H(12) | 118.0 |
| C(14)-C(13)-C(12) | 111.8(10) | C(14)-C(13)-H(13A) | 109.2 |
| C(12)-C(13)-H(13A) | 109.2 | C(14)-C(13)-H(13B) | 109.3 |

| | | | |
|---|---|---|---|
| C(12)-C(13)-H(13B) | 109.4 | H(13A)-C(13)-H(13B) | 107.9 |
| C(13)-C(14)-C(9) | 111.9(11) | C(13)-C(14)-C(15) | 114.0(10) |
| C(9)-C(14)-C(15) | 102.0(10) | C(13)-C(14)-Br(1) | 112.7(10) |
| C(9)-C(14)-Br(1) | 105.5(7) | C(15)-C(14)-Br(1) | 110.0(9) |
| C(16)-C(15)-C(14) | 99.5(10) | C(16)-C(15)-H(15A) | 111.8 |
| C(14)-C(15)-H(15A) | 111.7 | C(16)-C(15)-H(15B) | 112.1 |
| C(14)-C(15)-H(15B) | 111.9 | H(15A)-C(15)-H(15B) | 109.6 |
| N(1)-C(16)-C(15) | 107.6(9) | N(1)-C(16)-H(16A) | 110.2 |
| C(15)-C(16)-H(16A) | 110.3 | N(1)-C(16)-H(16B) | 110.2 |
| C(15)-C(16)-H(16B) | 110.0 | H(16A)-C(16)-H(16B) | 108.5 |
| N(2)-C(17)-C(19) | 100.3(8) | N(2)-C(17)-C(22) | 116.7(10) |
| C(19)-C(17)-C(22) | 114.7(10) | N(2)-C(17)-H(17) | 108.2 |
| C(19)-C(17)-H(17) | 108.2 | C(22)-C(17)-H(17) | 108.2 |
| C(20)-C(18)-C(28) | 123.6(12) | C(20)-C(18)-C(23) | 122.6(14) |
| C(28)-C(18)-C(23) | 113.8(13) | C(30)-C(19)-C(17) | 103.6(11) |
| C(30)-C(19)-C(32) | 109.6(11) | C(17)-C(19)-C(32) | 112.5(11) |
| C(30)-C(19)-Br(2) | 112.8(10) | C(17)-C(19)-Br(2) | 112.8(8) |
| C(32)-C(19)-Br(2) | 105.7(9) | C(18)-C(20)-C(26) | 117.7(14) |
| C(18)-C(20)-C(22) | 120.6(11) | C(26)-C(20)-C(22) | 121.6(12) |
| C(24)-C(21)-C(28) | 125.4(14) | C(24)-C(21)-O(4) | 113.0(12) |
| C(28)-C(21)-O(4) | 121.5(16) | C(25)-C(22)-C(17) | 113.1(10) |
| C(25)-C(22)-C(20) | 112.8(10) | C(17)-C(22)-C(20) | 108.6(10) |
| C(25)-C(22)-H(22) | 107.4 | C(17)-C(22)-H(22) | 107.3 |
| C(20)-C(22)-H(22) | 107.3 | N(2)-C(23)-C(18) | 114.9(11) |
| N(2)-C(23)-H(23A) | 108.5 | C(18)-C(23)-H(23A) | 108.4 |
| N(2)-C(23)-H(23B) | 108.6 | C(18)-C(23)-H(23B) | 108.6 |
| H(23A)-C(23)-H(23B) | 107.5 | C(21)-C(24)-C(26) | 124.0(11) |
| C(21)-C(24)-O(3) | 110.5(12) | C(26)-C(24)-O(3) | 125.3(14) |
| C(29)-C(25)-C(22) | 122.8(12) | C(29)-C(25)-H(25) | 118.5 |
| C(22)-C(25)-H(25) | 118.7 | C(24)-C(26)-C(20) | 117.5(12) |
| C(24)-C(26)-H(26) | 121.2 | C(20)-C(26)-H(26) | 121.2 |
| N(2)-C(27)-C(30) | 102.0(10) | N(2)-C(27)-H(27A) | 111.4 |

| | | | |
|---|---|---|---|
| C(30)-C(27)-H(27A) | 111.6 | N(2)-C(27)-H(27B) | 111.3 |
| C(30)-C(27)-H(27B) | 111.2 | H(27A)-C(27)-H(27B) | 109.2 |
| C(21)-C(28)-C(18) | 111.5(14) | C(21)-C(28)-H(28) | 124.3 |
| C(18)-C(28)-H(28) | 124.2 | C(25)-C(29)-C(32) | 123.0(12) |
| C(25)-C(29)-H(29) | 118.5 | C(32)-C(29)-H(29) | 118.5 |
| C(19)-C(30)-C(27) | 107.2(11) | C(19)-C(30)-H(30A) | 110.5 |
| C(27)-C(30)-H(30A) | 110.3 | C(19)-C(30)-H(30B) | 110.3 |
| C(27)-C(30)-H(30B) | 110.0 | H(30A)-C(30)-H(30B) | 108.5 |
| O(3)-C(31)-O(4) | 108.8(11) | O(3)-C(31)-H(31A) | 110.0 |
| O(4)-C(31)-H(31A) | 109.9 | O(3)-C(31)-H(31B) | 109.9 |
| O(4)-C(31)-H(31B) | 109.9 | H(31A)-C(31)-H(31B) | 108.3 |
| C(29)-C(32)-C(19) | 115.4(10) | C(29)-C(32)-H(32A) | 108.5 |
| C(19)-C(32)-H(32A) | 108.5 | C(29)-C(32)-H(32B) | 108.3 |
| C(19)-C(32)-H(32B) | 108.4 | H(32A)-C(32)-H(32B) | 107.5 |

**Table 4**. Anisotropic displacement parameters (A^2 x 10^3) for **16**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a*b*U12]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| N(1) | 48(5) | 28(5) | 39(5) | 7(4) | -10(4) | 9(4) |
| N(2) | 24(4) | 56(6) | 66(7) | 13(5) | 17(4) | 10(4) |
| O(1) | 68(8) | 69(6) | 83(8) | 32(6) | 6(6) | -29(6) |
| O(2) | 111(10) | 52(5) | 71(7) | 27(5) | 15(6) | 11(6) |
| O(3) | 58(7) | 72(6) | 95(8) | 49(6) | 2(6) | -4(5) |
| O(4) | 73(9) | 103(8) | 81(8) | 50(7) | 8(7) | 4(7) |
| Br(1) | 52(1) | 72(1) | 82(1) | 26(1) | 22(1) | 7(1) |
| Br(2) | 55(1) | 69(1) | 79(1) | 23(1) | 20(1) | 8(1) |
| C(1) | 46(8) | 33(6) | 51(7) | 7(5) | 13(6) | 1(5) |
| C(2) | 44(6) | 63(7) | 50(7) | 13(5) | 8(5) | -14(5) |
| C(3) | 59(9) | 64(8) | 43(8) | 1(6) | -4(7) | 11(7) |
| C(4) | 68(9) | 83(9) | 104(10) | 44(8) | 31(7) | -18(7) |
| C(5) | 37(7) | 64(8) | 50(7) | 3(6) | 6(6) | 1(6) |
| C(6) | 43(7) | 57(7) | 61(7) | 20(6) | 3(5) | 2(5) |
| C(7) | 25(5) | 41(6) | 50(7) | 7(5) | 6(4) | 9(4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(8) | 38(6) | 45(6) | 62(8) | 17(6) | 10(6) | 4(5) |
| C(9) | 23(4) | 67(7) | 37(6) | 14(5) | 11(4) | 14(4) |
| C(10) | 46(7) | 41(6) | 53(7) | -2(6) | -10(6) | 17(6) |
| C(11) | 41(7) | 75(9) | 68(9) | 25(7) | 15(6) | 14(6) |
| C(12) | 42(7) | 104(10) | 39(6) | 24(6) | -6(5) | 0(7) |
| C(13) | 47(7) | 58(7) | 59(8) | -15(6) | -16(6) | -20(6) |
| C(14) | 52(7) | 20(4) | 53(7) | 4(4) | -3(5) | -8(4) |
| C(15) | 50(7) | 60(7) | 68(7) | 30(6) | 1(6) | 23(6) |
| C(16) | 29(5) | 61(7) | 94(9) | 33(7) | 14(5) | 21(5) |
| C(17) | 44(6) | 17(4) | 43(6) | 0(4) | -7(4) | -1(4) |
| C(18) | 63(8) | 40(6) | 32(6) | 7(4) | 2(5) | 3(5) |
| C(19) | 26(5) | 61(7) | 59(7) | 18(6) | 10(5) | 11(5) |
| C(20) | 40(7) | 49(7) | 34(6) | 12(5) | -4(5) | 11(6) |
| C(21) | 74(10) | 54(8) | 41(7) | 22(6) | 8(7) | -15 (7) |
| C(22) | 29(6) | 48(7) | 40(6) | 17(5) | 10(5) | -2(5) |
| C(23) | 41(7) | 50(7) | 50(8) | 6(6) | -7(6) | 12(5) |
| C(24) | 70(10) | 31(6) | 48(7) | 19(5) | 11(7) | -4(6) |
| C(25) | 38(7) | 65(8) | 40(7) | 16(6) | -17(5) | 0(6) |
| C(26) | 53(7) | 40(6) | 53(7) | 13(5) | -6(6) | 25(5) |
| C(27) | 85(9) | 37(6) | 49(6) | 16(5) | -14(6) | 9(6) |
| C(28) | 40(6) | 60(7) | 47(7) | 5(5) | 6(5) | -2(5) |
| C(29) | 54(8) | 47(6) | 71(8) | -16(5) | -13(6) | -3(5) |
| C(30) | 63(9) | 41(6) | 90(9) | 34(6) | 15(7) | -5(5) |
| C(31) | 114(12) | 52(7) | 30(5) | 5(5) | -5(6) | 10(7) |
| C(32) | 58(8) | 40(6) | 89(10) | 22(6) | 18(7) | 12(5) |

**Table 5**. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **16**.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H(2) | 2024 | 5939 | -726 | 64 |
| H(4A) | 6336 | 8011 | -2717 | 97 |
| H(4B) | 6641 | 9243 | -1308 | 97 |
| H(6) | 7773 | 4837 | -208 | 64 |

| | | | | |
|---|---|---|---|---|
| H(8A) | 6760 | 2444 | 162 | 57 |
| H(8B) | 6479 | 3349 | 1680 | 57 |
| H(9) | 3256 | 3325 | 2269 | 49 |
| H(10) | 1598 | 4433 | 983 | 60 |
| H(11) | 612 | 3445 | -1473 | 71 |
| H(12) | -543 | 1236 | -1746 | 73 |
| H(13A) | -40 | -89 | -41 | 75 |
| H(13B) | 1789 | -35 | -595 | 75 |
| H(15A) | 3135 | 747 | 3116 | 68 |
| H(15B) | 2806 | -578 | 1764 | 68 |
| H(16A) | 5670 | 1420 | 2350 | 69 |
| H(16B) | 5301 | 144 | 974 | 69 |
| H(17) | 7126 | 5925 | 3012 | 44 |
| H(22) | 9043 | 4896 | 4218 | 46 |
| H(23A) | 4147 | 5773 | 3545 | 58 |
| H(23B) | 3787 | 6624 | 5053 | 58 |
| H(25) | 9997 | 5940 | 6794 | 59 |
| H(26) | 8383 | 3059 | 5868 | 58 |
| H(27A) | 5187 | 9133 | 4089 | 69 |
| H(27B) | 4933 | 7806 | 2743 | 69 |
| H(28) | 2631 | 4203 | 5345 | 60 |
| H(29) | 10953 | 8245 | 7027 | 77 |
| H(30A) | 7503 | 8477 | 2213 | 74 |
| H(30B) | 7830 | 9696 | 3635 | 74 |
| H(31A) | 4380 | 1199 | 7983 | 80 |
| H(31B) | 4092 | 4 | 6546 | 80 |
| H(32A) | 10563 | 9237 | 5073 | 72 |
| H(32B) | 8774 | 9338 | 5689 | 72 |

**Table 6**. Torsion angles [deg] for **16**.

| | | | |
|---|---|---|---|
| C(7)-C(1)-C(2)-C(3) | -5(2) | C(10)-C(1)-C(2)-C(3) | 176.4(12) |
| C(4)-O(2)-C(3)-C(5) | 6.4(16) | C(4)-O(2)-C(3)-C(2) | -173.4(15) |

| | | | |
|---|---|---|---|
| C(1)-C(2)-C(3)-O(2) | -177.5(14) | C(1)-C(2)-C(3)-C(5) | 3(2) |
| C(5)-O(1)-C(4)-O(2) | 13.3(16) | C(3)-O(2)-C(4)-O(1) | -12.2(16) |
| C(4)-O(1)-C(5)-C(6) | 170.3(16) | C(4)-O(1)-C(5)-C(3) | -9.1(16) |
| O(2)-C(3)-C(5)-C(6) | -178.4(13) | C(2)-C(3)-C(5)-C(6) | 1(2) |
| O(2)-C(3)-C(5)-O(1) | 1.1(17) | C(2)-C(3)-C(5)-O(1) | -179.1(12) |
| O(1)-C(5)-C(6)-C(7) | 176.9(16) | C(3)-C(5)-C(6)-C(7) | -4(2) |
| C(5)-C(6)-C(7)-C(1) | 2(2) | C(5)-C(6)-C(7)-C(8) | -174.5(14) |
| C(2)-C(1)-C(7)-C(6) | 3(2) | C(10)-C(1)-C(7)-C(6) | -178.6(13) |
| C(2)-C(1)-C(7)-C(8) | 178.9(13) | C(10)-C(1)-C(7)-C(8) | -2(2) |
| C(9)-N(1)-C(8)-C(7) | -49.4(15) | C(16)-N(1)-C(8)-C(7) | -169.2(10) |
| C(6)-C(7)-C(8)-N(1) | -166.5(13) | C(1)-C(7)-C(8)-N(1) | 17.4(18) |
| C(8)-N(1)-C(9)-C(10) | 65.1(13) | C(16)-N(1)-C(9)-C(10) | -173.3(10) |
| C(8)-N(1)-C(9)-C(14) | -170.3(10) | C(16)-N(1)-C(9)-C(14) | -48.7(11) |
| C(2)-C(1)-C(10)-C(11) | 65.5(16) | C(7)-C(1)-C(10)-C(11) | -113.2(15) |
| C(2)-C(1)-C(10)-C(9) | -165.1(11) | C(7)-C(1)-C(10)-C(9) | 16.2(18) |
| N(1)-C(9)-C(10)-C(11) | 86.5(13) | C(14)-C(9)-C(10)-C(11) | -25.4(17) |
| N(1)-C(9)-C(10)-C(1) | -44.1(14) | C(14)-C(9)-C(10)-C(1) | -156.0(11) |
| C(1)-C(10)-C(11)-C(12) | 135.7(15) | C(9)-C(10)-C(11)-C(12) | 6(2) |
| C(10)-C(11)-C(12)-C(13) | -7(2) | C(11)-C(12)-C(13)-C(14) | 25(2) |
| C(12)-C(13)-C(14)-C(9) | -40.7(16) | C(12)-C(13)-C(14)-C(15) | -155.7(12) |
| C(12)-C(13)-C(14)-Br(1) | 78.0(13) | N(1)-C(9)-C(14)-C(13) | -73.6(12) |
| C(10)-C(9)-C(14)-C(13) | 44.1(16) | N(1)-C(9)-C(14)-C(15) | 48.6(11) |
| C(10)-C(9)-C(14)-C(15) | 166.3(11) | N(1)-C(9)-C(14)-Br(1) | 163.5(7) |
| C(10)-C(9)-C(14)-Br(1) | -78.8(12) | C(13)-C(14)-C(15)-C(16) | 89.9(14) |
| C(9)-C(14)-C(15)-C(16) | -30.9(13) | Br(1)-C(14)-C(15)-C(16) | -142.5(9) |
| C(8)-N(1)-C(16)-C(15) | 155.0(11) | C(9)-N(1)-C(16)-C(15) | 29.4(14) |
| C(14)-C(15)-C(16)-N(1) | 2.3(14) | C(23)-N(2)-C(17)-C(19) | 171.5(10) |
| C(27)-N(2)-C(17)-C(19) | 46.9(11) | C(23)-N(2)-C(17)-C(22) | -64.0(13) |
| C(27)-N(2)-C(17)-C(22) | 171.4(10) | N(2)-C(17)-C(19)-C(30) | -42.6(13) |
| C(22)-C(17)-C(19)-C(30) | -168.5(11) | N(2)-C(17)-C(19)-C(32) | 75.7(12) |
| C(22)-C(17)-C(19)-C(32) | -50.2(14) | N(2)-C(17)-C(19)-Br(2) | -164.9(8) |
| C(22)-C(17)-C(19)-Br(2) | 69.3(12) | C(28)-C(18)-C(20)-C(26) | 1(2) |

| | | | |
|---|---|---|---|
| C(23)-C(18)-C(20)-C(26) | -177.0(12) | C(28)-C(18)-C(20)-C(22) | 178.2(12) |
| C(23)-C(18)-C(20)-C(22) | 0(2) | C(31)-O(4)-C(21)-C(24) | 6.8(17) |
| C(31)-O(4)-C(21)-C(28) | -176.7(13) | N(2)-C(17)-C(22)-C(25) | -79.9(13) |
| C(19)-C(17)-C(22)-C(25) | 36.9(15) | N(2)-C(17)-C(22)-C(20) | 46.1(13) |
| C(19)-C(17)-C(22)-C(20) | 162.9(10) | C(18)-C(20)-C(22)-C(25) | 113.6(14) |
| C(26)-C(20)-C(22)-C(25) | -69.4(15) | C(18)-C(20)-C(22)-C(17) | -12.5(16) |
| C(26)-C(20)-C(22)-C(17) | 164.5(11) | C(17)-N(2)-C(23)-C(18) | 46.6(15) |
| C(27)-N(2)-C(23)-C(18) | 163.6(11) | C(20)-C(18)-C(23)-N(2) | -17.6(19) |
| C(28)-C(18)-C(23)-N(2) | 164.2(12) | C(28)-C(21)-C(24)-C(26) | -2(3) |
| O(4)-C(21)-C(24)-C(26) | 174.6(14) | C(28)-C(21)-C(24)-O(3) | -177.4(14) |
| O(4)-C(21)-C(24)-O(3) | -1.0(19) | C(31)-O(3)-C(24)-C(21) | -5.8(16) |
| C(31)-O(3)-C(24)-C(26) | 178.7(14) | C(17)-C(22)-C(25)-C(29) | -12.9(19) |
| C(20)-C(22)-C(25)-C(29) | -136.5(15) | C(21)-C(24)-C(26)-C(20) | 5(2) |
| O(3)-C(24)-C(26)-C(20) | 179.8(12) | C(18)-C(20)-C(26)-C(24) | -4.3(19) |
| C(22)-C(20)-C(26)-C(24) | 178.6(12) | C(17)-N(2)-C(27)-C(30) | -32.8(13) |
| C(23)-N(2)-C(27)-C(30) | -151.5(11) | C(24)-C(21)-C(28)-C(18) | -2(2) |
| O(4)-C(21)-C(28)-C(18) | -177.7(13) | C(20)-C(18)-C(28)-C(21) | 2(2) |
| C(23)-C(18)-C(28)-C(21) | -180.0(12) | C(22)-C(25)-C(29)-C(32) | 3(2) |
| C(17)-C(19)-C(30)-C(27) | 22.6(15) | C(32)-C(19)-C(30)-C(27) | -97.7(14) |
| Br(2)-C(19)-C(30)-C(27) | 144.9(11) | N(2)-C(27)-C(30)-C(19) | 5.5(15) |
| C(24)-O(3)-C(31)-O(4) | 9.9(14) | C(21)-O(4)-C(31)-O(3) | -10.3(14) |
| C(25)-C(29)-C(32)-C(19) | -16(2) | C(30)-C(19)-C(32)-C(29) | 153.6(12) |
| C(17)-C(19)-C(32)-C(29) | 38.9(16) | Br(2)-C(19)-C(32)-C(29) | -84.7(13) |

# X-ray crystallographic data for amarbellisine (3)

All data were collected on Bruker apes Ⅱ.

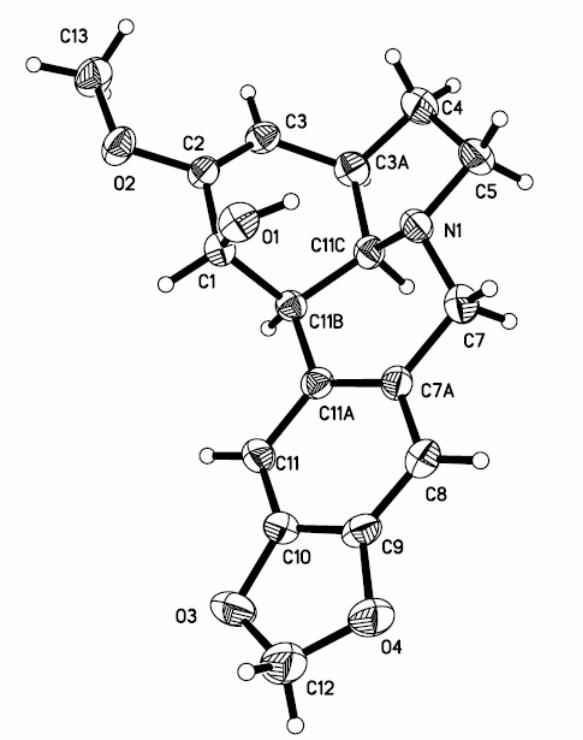



**Table 1**. Crystal data and structure refinement for **amarbellisine (3)**.

| | |
|---|---|
| Empirical formula | C17 H19 N O4 |
| Formula weight | 301.33 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 |
| Crystal system, space group | Orthorhombic, P2(1)2(1)2(1) |
| Unit cell dimensions | a = 9.1314 (13) A α= 90 deg.<br>b = 12.4725(18) A β= 90 deg.<br>c = 12.8711(18) A γ= 90 deg. |
| Volume | 1465.9(4) A^3 |
| Z, Calculated density | 4, 1.365 Mg/m^3 |
| Absorption coefficient | 0.097 mm^-1 |
| F(000) | 640 |
| Crystal size | 0.27 x 0.21 x 0.14 mm |
| Theta range for data collection | 2.27 to 28.28 deg. |

| | |
|---|---|
| Limiting indices | -12<=h<=11, -16<=k<=16, -16<=l<=11 |
| Reflections collected / unique | 10110 / 3441 [R(int) = 0.0485] |
| Completeness to theta = 28.28 | 96.9 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 1.0000 and 0.7651 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 3441 / 0 / 202 |
| Goodness-of-fit on F^2 | 0.975 |
| Final R indices [I>2sigma(I)] | R1 = 0.0462, wR2 = 0.0796 |
| R indices (all data) | R1 = 0.0948, wR2 = 0.0969 |
| Absolute structure parameter | 1.3(14) |
| Extinction coefficien | 0.0144(14) |
| Largest diff. peak and hole | 0.135 and -0.152 e.A^-3 |

**Table 2**. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **amarbellisine (3)**. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| N(1) | 2608(2) | 8627(1) | 8222(1) | 46(1) |
| O(1) | 2053(2) | 8918(1) | 10396(1) | 53(1) |
| O(2) | 3218(2) | 7014(1) | 11599(1) | 57(1) |
| O(3) | 5692(2) | 12584(1) | 10474(2) | 75(1) |
| O(4) | 4325(2) | 13294(1) | 9119(1) | 71(1) |
| C(1) | 3497(2) | 8482(2) | 10468(2) | 42(1) |
| C(2) | 3443(2) | 7288(2) | 10576(2) | 43(1) |
| C(3) | 3565(2) | 6625(2) | 9779(2) | 47(1) |
| C(4) | 2469(3) | 6774(2) | 7958(2) | 68(1) |
| C(5) | 2093(3) | 7840(2) | 7457(2) | 59(1) |
| C(7) | 2790(3) | 9710(2) | 7808(2) | 57(1) |
| C(8) | 3512(2) | 11551(2) | 8415(2) | 51(1) |
| C(9) | 4249(3) | 12184(2) | 9106(2) | 51(1) |
| C(10) | 5061(2) | 11766(2) | 9900(2) | 51(1) |
| C(11) | 5181(2) | 10688(2) | 10058(2) | 49(1) |

| | | | | |
|---|---|---|---|---|
| C(12) | 5114(4) | 13541(2) | 10041(2) | 83(1) |
| C(13) | 3160(3) | 5902(2) | 11853(2) | 64(1) |
| C(3A) | 3790(3) | 6996(2) | 8676(2) | 51(1) |
| C(7A) | 3609(2) | 10430(2) | 8562(2) | 42(1) |
| C(11A) | 4429(2) | 10004(2) | 9364(2) | 42(1) |
| C(11B) | 4476(2) | 8805(2) | 9552(2) | 40(1) |
| C(11C) | 4036(2) | 8206(2) | 8571(2) | 44(1) |

**Table 3**. Bond lengths [A] and angles [deg] for **amarbellisine (3)**

| | | | |
|---|---|---|---|
| N(1)-C(7) | 1.461(3) | N(1)-C(5) | 1.468(3) |
| N(1)-C(11C) | 1.475(3) | O(1)-C(1) | 1.430(2) |
| O(1)-H(1) | 0.8200 | O(2)-C(2) | 1.376(2) |
| O(2)-C(13) | 1.425(2) | O(3)-C(10) | 1.385(3) |
| O(3)-C(12) | 1.419(3) | O(4)-C(9) | 1.387(2) |
| O(4)-C(12) | 1.422(3) | C(1)-C(2) | 1.498(3) |
| C(1)-C(11B) | 1.533(3) | C(1)-H(1A) | 0.9800 |
| C(2)-C(3) | 1.322(3) | C(3)-C(3A) | 1.508(3) |
| C(3)-H(3) | 0.9300 | C(4)-C(5) | 1.518(3) |
| C(4)-C(3A) | 1.544(3) | C(4)-H(4A) | 0.9700 |
| C(4)-H(4B) | 0.9700 | C(5)-H(5A) | 0.9700 |
| C(5)-H(5B) | 0.9700 | C(7)-C(7A) | 1.519(3) |
| C(7)-H(7A) | 0.9700 | C(7)-H(7B) | 0.9700 |
| C(8)-C(9) | 1.366(3) | C(8)-C(7A) | 1.414(3) |
| C(8)-H(8) | 0.9300 | C(9)-C(10) | 1.365(3) |
| C(10)-C(11) | 1.364(3) | C(11)-C(11A) | 1.413(3) |
| C(11)-H(11) | 0.9300 | C(12)-H(12A) | 0.9700 |
| C(12)-H(12B) | 0.9700 | C(13)-H(13A) | 0.9600 |
| C(13)-H(13B) | 0.9600 | C(13)-H(13C) | 0.9600 |
| C(3A)-C(11C) | 1.532(3) | C(3A)-H(3A) | 0.9800 |
| C(7A)-C(11A) | 1.381(3) | C(11A)-C(11B) | 1.516(3) |
| C(11B)-C(11C) | 1.521(3) | C(11B)-H(11B) | 0.9800 |
| C(11C)-H(11C) | 0.9800 | C(7)-N(1)-C(5) | 114.22(18) |

| | | | |
|---|---|---|---|
| C(7)-N(1)-C(11C) | 109.88(16) | C(5)-N(1)-C(11C) | 104.45(17) |
| C(1)-O(1)-H(1) | 109.5 | C(2)-O(2)-C(13) | 117.81(17) |
| C(10)-O(3)-C(12) | 104.8(2) | C(9)-O(4)-C(12) | 104.58(19) |
| O(1)-C(1)-C(2) | 110.73(17) | O(1)-C(1)-C(11B) | 112.82(17) |
| C(2)-C(1)-C(11B) | 110.58(17) | O(1)-C(1)-H(1A) | 107.5 |
| C(2)-C(1)-H(1A) | 107.5 | C(11B)-C(1)-H(1A) | 107.5 |
| C(3)-C(2)-O(2) | 126.86(19) | C(3)-C(2)-C(1) | 123.2(2) |
| O(2)-C(2)-C(1) | 109.91(17) | C(2)-C(3)-C(3A) | 123.4(2) |
| C(2)-C(3)-H(3) | 118.3 | C(3A)-C(3)-H(3) | 118.3 |
| C(5)-C(4)-C(3A) | 105.86(18) | C(5)-C(4)-H(4A) | 110.6 |
| C(3A)-C(4)-H(4A) | 110.6 | C(5)-C(4)-H(4B) | 110.6 |
| C(3A)-C(4)-H(4B) | 110.6 | H(4A)-C(4)-H(4B) | 108.7 |
| N(1)-C(5)-C(4) | 103.22(18) | N(1)-C(5)-H(5A) | 111.1 |
| C(4)-C(5)-H(5A) | 111.1 | N(1)-C(5)-H(5B) | 111.1 |
| C(4)-C(5)-H(5B) | 111.1 | H(5A)-C(5)-H(5B) | 109.1 |
| N(1)-C(7)-C(7A) | 111.7(2) | N(1)-C(7)-H(7A) | 109.3 |
| C(7A)-C(7)-H(7A) | 109.3 | N(1)-C(7)-H(7B) | 109.3 |
| C(7A)-C(7)-H(7B) | 109.3 | H(7A)-C(7)-H(7B) | 107.9 |
| C(9)-C(8)-C(7A) | 116.9(2) | C(9)-C(8)-H(8) | 121.5 |
| C(7A)-C(8)-H(8) | 121.5 | C(8)-C(9)-C(10) | 122.3(2) |
| C(8)-C(9)-O(4) | 127.5(2) | C(10)-C(9)-O(4) | 110.2(2) |
| C(11)-C(10)-C(9) | 122.1(2) | C(11)-C(10)-O(3) | 127.8(2) |
| C(9)-C(10)-O(3) | 110.1(2) | C(10)-C(11)-C(11A) | 117.4(2) |
| C(10)-C(11)-H(11) | 121.3 | C(11A)-C(11)-H(11) | 121.3 |
| O(3)-C(12)-O(4) | 109.5(2) | O(3)-C(12)-H(12A) | 109.8 |
| O(4)-C(12)-H(12A) | 109.8 | O(3)-C(12)-H(12B) | 109.8 |
| O(4)-C(12)-H(12B) | 109.8 | H(12A)-C(12)-H(12B) | 108.2 |
| O(2)-C(13)-H(13A) | 109.5 | O(2)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 | O(2)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 | H(13B)-C(13)-H(13C) | 109.5 |
| C(3)-C(3A)-C(11C) | 113.89(19) | C(3)-C(3A)-C(4) | 113.67(19) |
| C(11C)-C(3A)-C(4) | 103.82(19) | C(3)-C(3A)-H(3A) | 108.4 |

| | | | |
|---|---|---|---|
| C(11C)-C(3A)-H(3A) | 108.4 | C(4)-C(3A)-H(3A) | 108.4 |
| C(11A)-C(7A)-C(8) | 121.0(2) | C(11A)-C(7A)-C(7) | 121.12(19) |
| C(8)-C(7A)-C(7) | 117.9(2) | C(7A)-C(11A)-C(11) | 120.3(2) |
| C(7A)-C(11A)-C(11B) | 120.93(19) | C(11)-C(11A)-C(11B) | 118.7(2) |
| C(11A)-C(11B)-C(11C) | 110.17(18) | C(11A)-C(11B)-C(1) | 111.44(16) |
| C(11C)-C(11B)-C(1) | 110.82(16) | C(11A)-C(11B)-H(11B) | 108.1 |
| C(11C)-C(11B)-H(11B) | 108.1 | C(1)-C(11B)-H(11B) | 108.1 |
| N(1)-C(11C)-C(11B) | 108.16(17) | N(1)-C(11C)-C(3A) | 104.38(18) |
| C(11B)-C(11C)-C(3A) | 116.73(19) | N(1)-C(11C)-H(11C) | 109.1 |
| C(11B)-C(11C)-H(11C) | 109.1 | C(3A)-C(11C)-H(11C) | 109.1 |

**Table 4**. Anisotropic displacement parameters (A^2 x 10^3) for **amarbellisine (3)**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a*b*U12]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| N(1) | 49(1) | 46(1) | 43(1) | -4(1) | -6(1) | -3(1) |
| O(1) | 47(1) | 53(1) | 59(1) | -2(1) | 10(1) | 9(1) |
| O(2) | 81(1) | 43(1) | 47(1) | 8(1) | 9(1) | 4(1) |
| O(3) | 96(1) | 50(1) | 81(1) | -8(1) | -17(1) | -19(1) |
| O(4) | 95(1) | 43(1) | 75(1) | 4(1) | 1(1) | -5(1) |
| C(1) | 43(1) | 44(1) | 39(1) | -2(1) | -1(1) | 1(1) |
| C(2) | 47(1) | 41(1) | 41(1) | 3(1) | 5(1) | -1(1) |
| C(3) | 51(1) | 38(1) | 52(2) | 0(1) | 7(1) | 1(1) |
| C(4) | 86(2) | 58(2) | 59(2) | -10(1) | -9(2) | -14(1) |
| C(5) | 63(2) | 62(2) | 50(2) | -12(1) | -7(1) | -10(1) |
| C(7) | 62(2) | 58(2) | 49(2) | 3(1) | -9(1) | -2(1) |
| C(8) | 56(1) | 50(1) | 47(2) | 9(1) | 2(1) | 3(1) |
| C(9) | 61(2) | 36(1) | 55(2) | 3(1) | 12(1) | -3(1) |
| C(10) | 58(1) | 47(2) | 48(2) | -4(1) | -1(1) | -12(1) |
| C(11) | 52(1) | 48(1) | 47(2) | 1(1) | -10(1) | -4(1) |
| C(12) | 125(3) | 52(2) | 73(2) | -3(2) | 8(2) | -9(2) |
| C(13) | 86(2) | 44(1) | 63(2) | 16(1) | -3(2) | 4(1) |
| C(3A) | 59(2) | 42(1) | 52(2) | -11(1) | 7(1) | 5(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(7A) | 43(1) | 46(1) | 37(1) | 2(1) | 1(1) | 0(1) |
| C(11A) | 42(1) | 42(1) | 41(1) | 1(1) | 5(1) | -3(1) |
| C(11B) | 35(1) | 43(1) | 41(1) | -1(1) | -1(1) | 0(1) |
| C(11C) | 44(1) | 50(1) | 38(1) | -3(1) | 6(1) | 0(1) |

**Table 5**. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **amrbellisine (3)**.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H(1) | 1719 | 8799 | 9816 | 79 |
| H(1A) | 3945 | 8770 | 11102 | 51 |
| H(3) | 3508 | 5892 | 9906 | 57 |
| H(4A) | 2722 | 6247 | 7434 | 81 |
| H(4B) | 1645 | 6506 | 8357 | 81 |
| H(5A) | 2597 | 7926 | 6799 | 70 |
| H(5B) | 1047 | 7906 | 7343 | 70 |
| H(7A) | 1834 | 10017 | 7667 | 68 |
| H(7B) | 3324 | 9676 | 7158 | 68 |
| H(8) | 2971 | 11845 | 7873 | 61 |
| H(11) | 5737 | 10414 | 10603 | 59 |
| H(12A) | 5905 | 14033 | 9883 | 100 |
| H(12B) | 4468 | 13884 | 10539 | 100 |
| H(13A) | 2379 | 5568 | 11474 | 97 |
| H(13B) | 2991 | 5821 | 12585 | 97 |
| H(13C) | 4073 | 5569 | 11672 | 97 |
| H(3A) | 4647 | 6625 | 8391 | 61 |
| H(11B) | 5486 | 8607 | 9722 | 48 |
| H(11C) | 4769 | 8338 | 8030 | 53 |

**Table 6**. Torsion angles [deg] for **amarbellisine (3)**.

| | | | |
|---|---|---|---|
| C(13)-O(2)-C(2)-C(3) | 2.6(3) | C(13)-O(2)-C(2)-C(1) | -178.98(19) |
| O(1)-C(1)-C(2)-C(3) | 95.1(2) | C(11B)-C(1)-C(2)-C(3) | -30.7(3) |
| O(1)-C(1)-C(2)-O(2) | -83.4(2) | C(11B)-C(1)-C(2)-O(2) | 150.83(17) |
| O(2)-C(2)-C(3)-C(3A) | 179.1(2) | C(1)-C(2)-C(3)-C(3A) | 0.8(3) |

| | | | |
|---|---|---|---|
| C(7)-N(1)-C(5)-C(4) | 162.3(2) | C(11C)-N(1)-C(5)-C(4) | 42.2(2) |
| C(3A)-C(4)-C(5)-N(1) | -27.1(2) | C(5)-N(1)-C(7)-C(7A) | -168.39(19) |
| C(11C)-N(1)-C(7)-C(7A) | -51.4(2) | C(7A)-C(8)-C(9)-C(10) | 0.7(3) |
| C(7A)-C(8)-C(9)-O(4) | 179.6(2) | C(12)-O(4)-C(9)-C(8) | 175.0(2) |
| C(12)-O(4)-C(9)-C(10) | -6.0(3) | C(8)-C(9)-C(10)-C(11) | -0.4(4) |
| O(4)-C(9)-C(10)-C(11) | -179.5(2) | C(8)-C(9)-C(10)-O(3) | 179.7(2) |
| O(4)-C(9)-C(10)-O(3) | 0.6(3) | C(12)-O(3)-C(10)-C(11) | -174.9(2) |
| C(12)-O(3)-C(10)-C(9) | 5.0(2) | C(9)-C(10)-C(11)-C(11A) | 0.0(3) |
| O(3)-C(10)-C(11)-C(11A) | 180.0(2) | C(10)-O(3)-C(12)-O(4) | -8.8(3) |
| C(9)-O(4)-C(12)-O(3) | 9.1(3) | C(2)-C(3)-C(3A)-C(11C) | 6.4(3) |
| C(2)-C(3)-C(3A)-C(4) | -112.3(2) | C(5)-C(4)-C(3A)-C(3) | 126.9(2) |
| C(5)-C(4)-C(3A)-C(11C) | 2.6(3) | C(9)-C(8)-C(7A)-C(11A) | -0.7(3) |
| C(9)-C(8)-C(7A)-C(7) | 179.6(2) | N(1)-C(7)-C(7A)-C(11A) | 17.8(3) |
| N(1)-C(7)-C(7A)-C(8) | -162.44(19) | C(8)-C(7A)-C(11A)-C(11) | 0.4(3) |
| C(7)-C(7A)-C(11A)-C(11) | -179.9(2) | C(8)-C(7A)-C(11A)-C(11B) | 177.10(19) |
| C(7)-C(7A)-C(11A)-C(11B) | -3.2(3) | C(10)-C(11)-C(11A)-C(7A) | -0.1(3) |
| C(10)-C(11)-C(11A)-C(11B) | -176.83(19) | C(7A)-C(11A)-C(11B)-C(11C) | 21.0(3) |
| C(11)-C(11A)-C(11B)-C(11C) | -162.24(19) | C(7A)-C(11A)-C(11B)-C(1) | -102.4(2) |
| C(11)-C(11A)-C(11B)-C(1) | 74.3(2) | O(1)-C(1)-C(11B)-C(11A) | 49.9(2) |
| C(2)-C(1)-C(11B)-C(11A) | 174.50(17) | O(1)-C(1)-C(11B)-C(11C) | -73.2(2) |
| C(2)-C(1)-C(11B)-C(11C) | 51.4(2) | C(7)-N(1)-C(11C)-C(11B) | 71.2(2) |
| C(5)-N(1)-C(11C)-C(11B) | -165.84(17) | C(7)-N(1)-C(11C)-C(3A) | -163.83(17) |
| C(5)-N(1)-C(11C)-C(3A) | -40.9(2) | C(11A)-C(11B)-C(11C)-N(1) | -53.4(2) |
| C(1)-C(11B)-C(11C)-N(1) | 70.4(2) | C(11A)-C(11B)-C(11C)-C(3A) | -170.61(18) |
| C(1)-C(11B)-C(11C)-C(3A) | -46.8(3) | C(3)-C(3A)-C(11C)-N(1) | -101.4(2) |
| C(4)-C(3A)-C(11C)-N(1) | 22.7(2) | C(3)-C(3A)-C(11C)-C(11B) | 17.9(3) |
| C(4)-C(3A)-C(11C)-C(11B) | 142.0(2) | | |

**Table 7**. Hydrogen bonds for **amarbellisine (3)** [A and deg.].

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| O(1)-H(1)...N(1) | 0.82 | 2.22 | 2.867(3) | 136.4 |