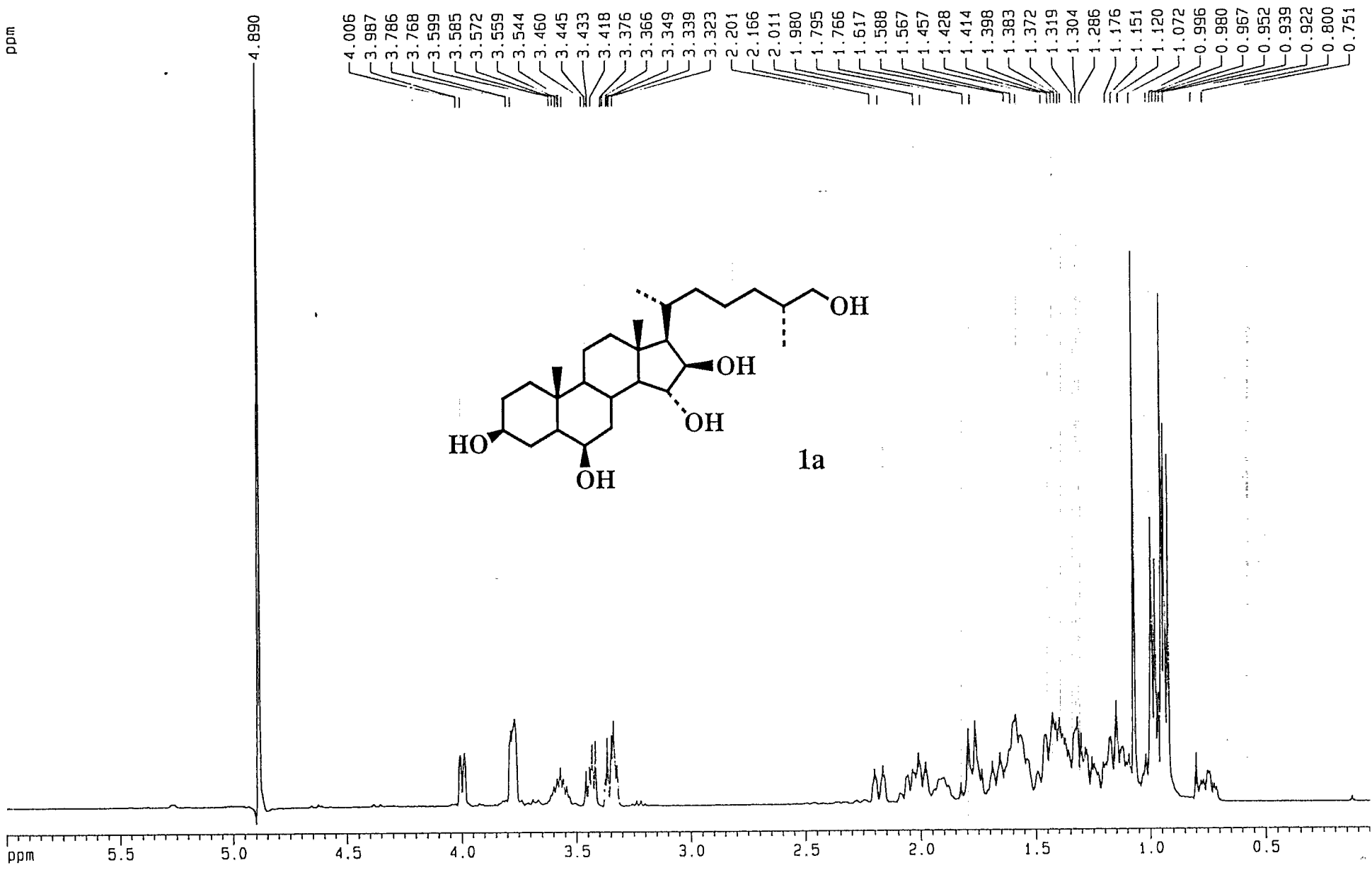
JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(13), 4438-4443, DOI:10.1021/jo980266c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3.987
3.786
3.768
3.599
3.585
3.572
3.559
3.544
3.460
3.445
3.433
3.418
3.376
3.366
3.349
3.339
3.323
2.201
2.166
2.011
1.980
1.795
1.766
1.617
1.588
1.567
1.457
1.428
1.414
1.398
1.383
1.372
1.319
1.304
1.286
1.176
1.151
1.120
1.072
0.996
0.980
0.967
0.952
0.939
0.922
0.800
0.751
OH
OH
OH
HO
OH
1a
ppm
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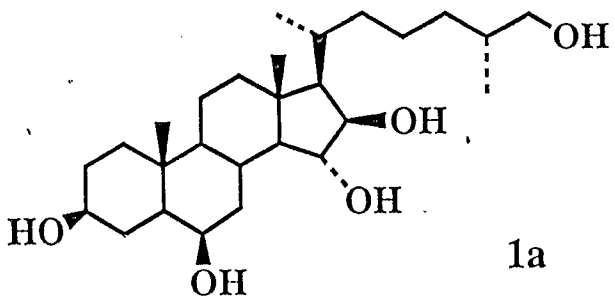
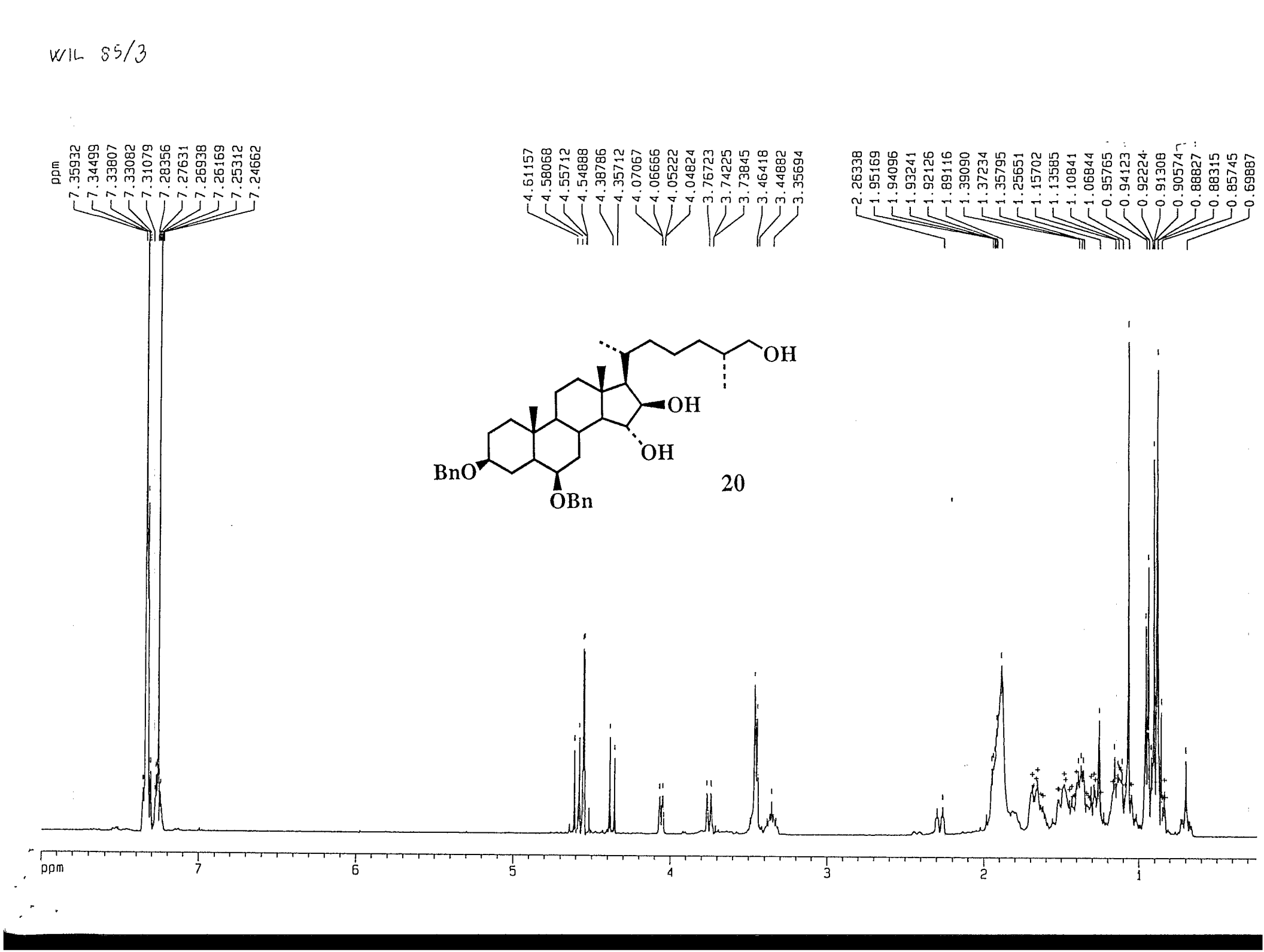
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5



1a

WIL 85/3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7.34499
7.33807
7.33082
7.31079
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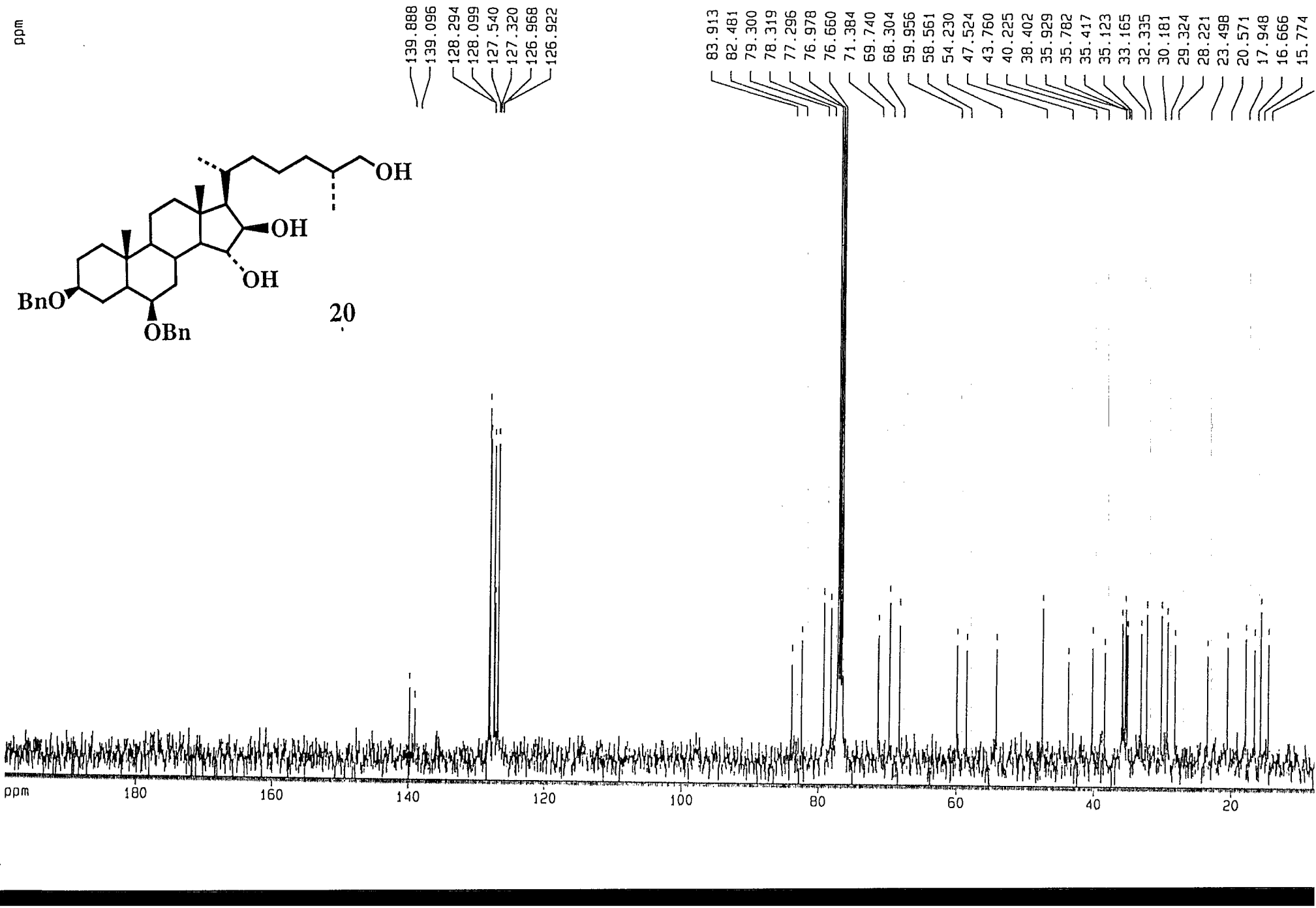
7.27631
7.26938
7.26169
7.25312
7.24662
4.61157
4.58068
4.55712
4.54888
4.38786
4.35712
4.07067
4.06666
4.05222
4.04824
3.76723
3.74225
3.73845
3.46418
3.44882
3.35694
2.26338
1.95169
1.94096
1.93241
1.92126
1.89116
1.39090
1.37234
1.35795
1.25651
1.15702
1.13585
1.10841
1.06844
0.95765
0.94123
0.92224
0.91308
0.90574
0.88827
0.88315
0.85745
0.69887
OH
OH
OH
BnO
OBn
20
ppm
7
6
5
4
3
2
1

WIL 85/3


ppm
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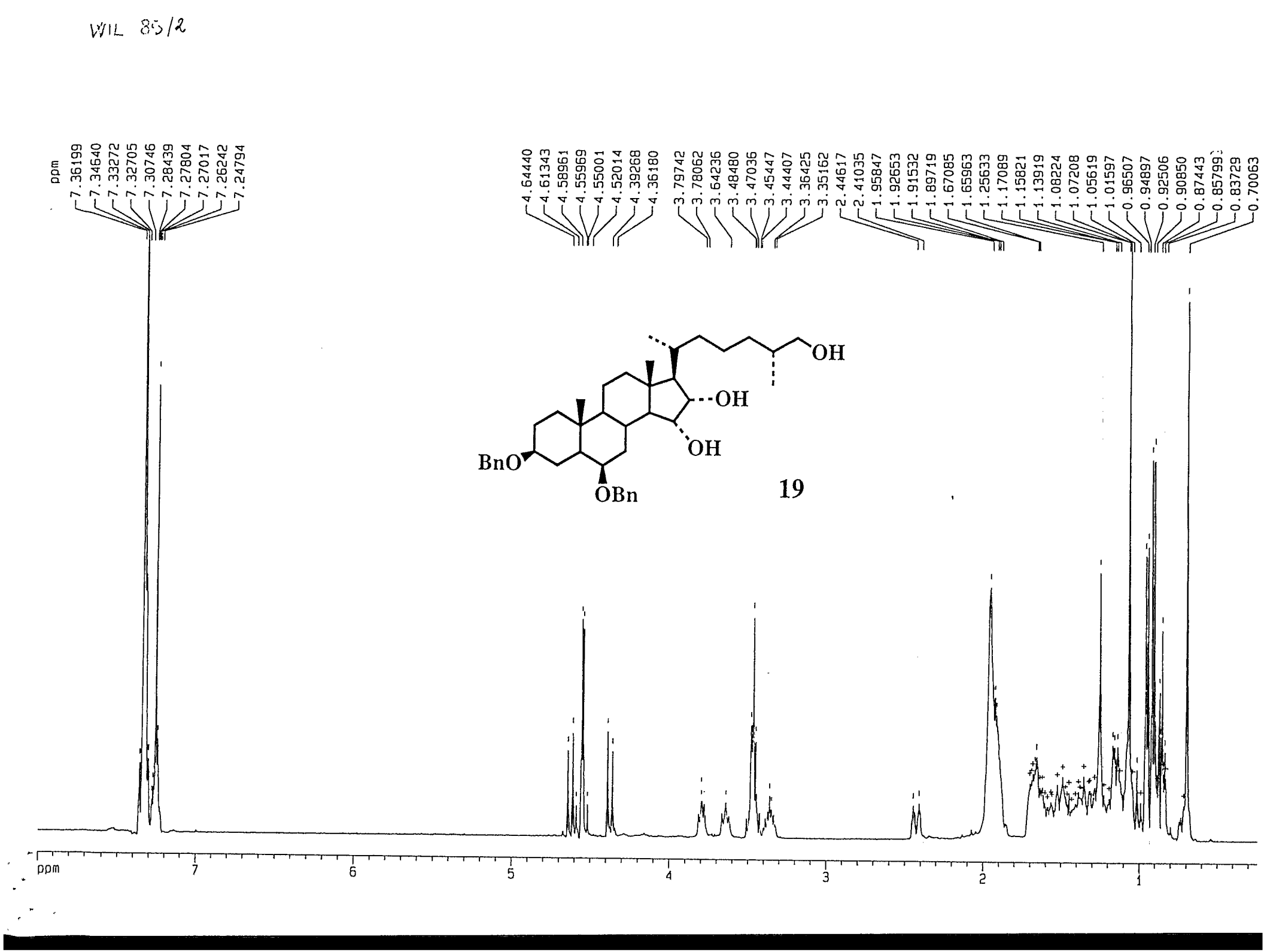
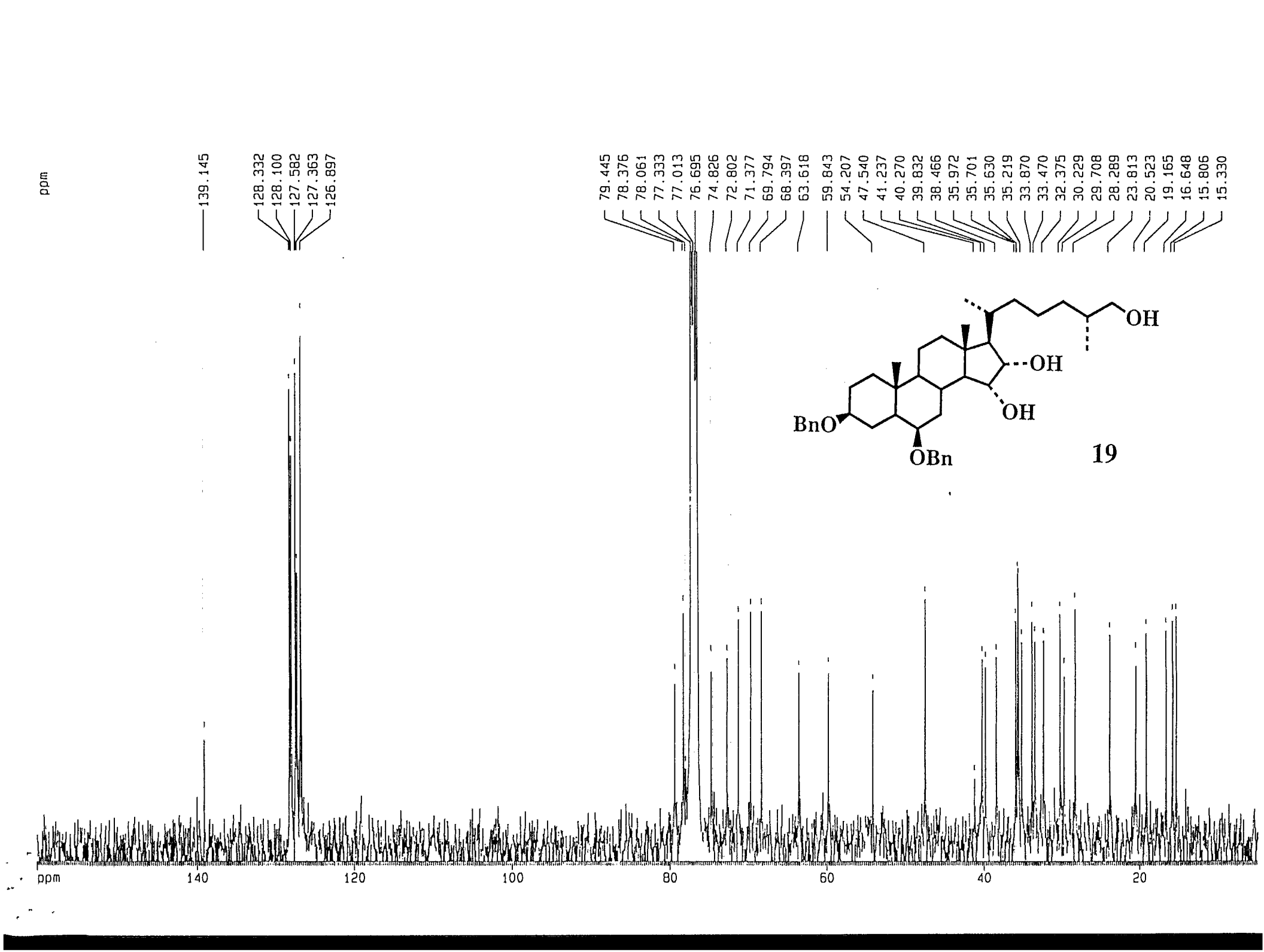
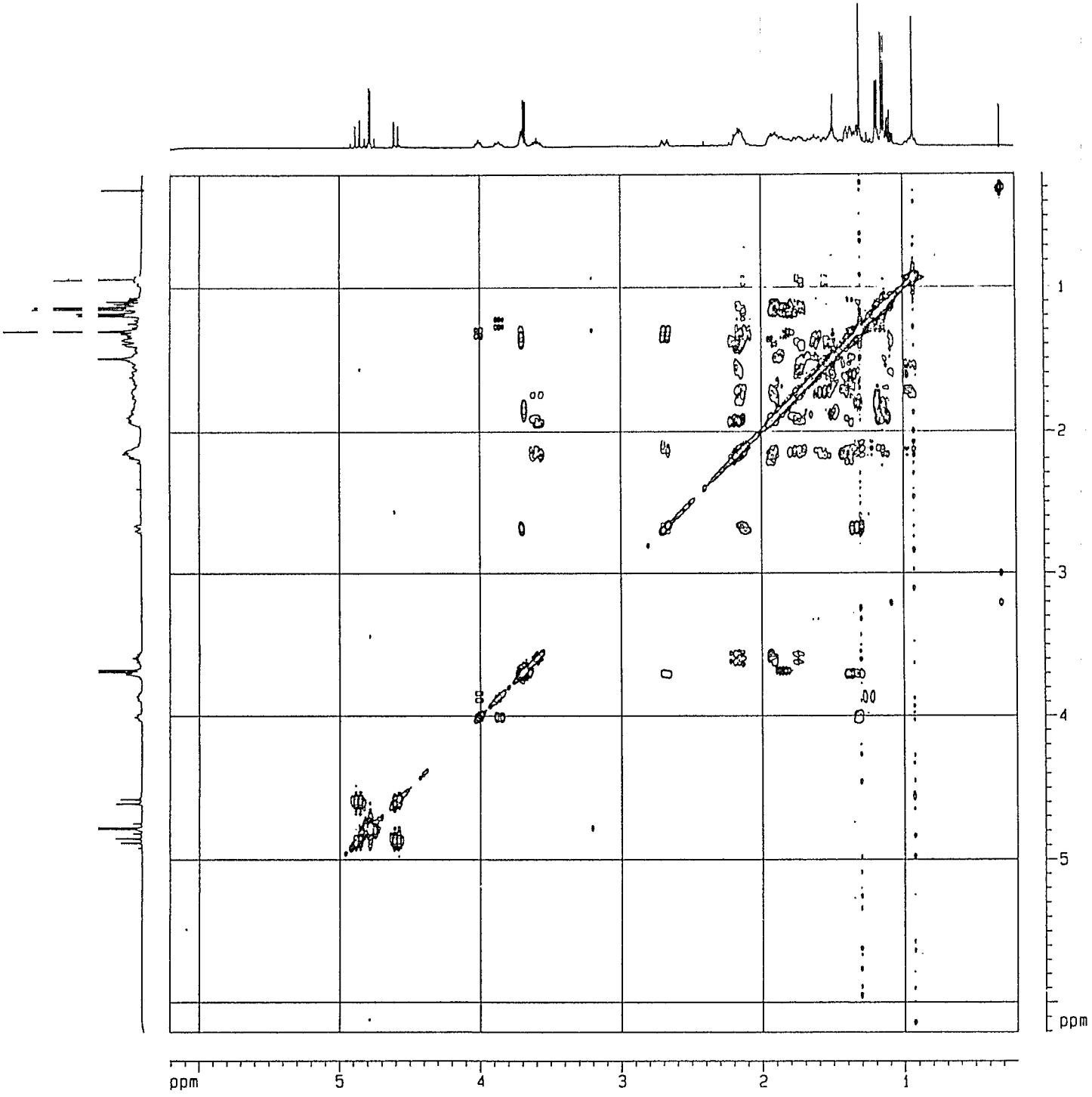
139.096
128.294
128.099
127.540
127.320
126.968
126.922
83.913
82.481
79.300
78.319
77.296
76.978
76.660
71.384
69.740
68.304
59.956
58.561
54.230
47.524
43.760
40.225
38.402
35.929
35.782
35.417
35.123
33.165
32.335
30.181
29.324
28.221
23.498
20.571
17.948
16.666
15.774
OH
OH
OH
BnO
OBn
20
ppm
180
160
140
120
100
80
60
40
20

WIL 85/2


ppm
7.36199
7.34640
7.33272
7.32705
7.30746
7.28439
7.27804
7.27017
7.26242
7.24794
4.64440
4.61343
4.58961
4.55969
4.55001
4.52014
4.39268
4.36180
3.79742
3.78062
3.64236
3.48480
3.47036
3.45447
3.44407
3.36425
3.35162
2.44617
2.41035
1.95847
1.92653
1.91532
1.89719
1.67085
1.65963
1.25633
1.17089
1.15821
1.13919
1.08224
1.07208
1.05619
1.01597
0.96507
0.94897
0.92506
0.90850
0.87443
0.85799
0.83729
0.70063
OH
OH
OH
BnO
OBn
19
ppm
7
6
5
4
3
2
1


ppm
139.145
128.332
128.100
127.582
127.363
126.897
79.445
78.376
78.061
77.333
77.013
76.695
74.826
72.802
71.377
69.794
68.397
63.618
59.843
54.207
47.540
41.237
40.270
39.832
38.466
35.972
35.701
35.630
35.219
33.870
33.470
32.375
30.229
29.708
28.289
23.813
20.523
19.165
16.648
15.806
15.330
OH
OH
OH
BnO
OBn
19
ppm
140
120
100
80
60
40
20

COSY (400 MHz)



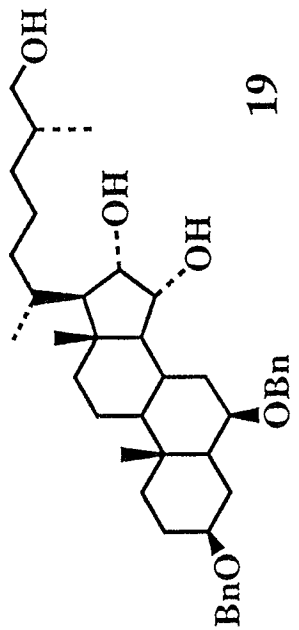
Current Data Parameters

NAME	st-dupat-cosy
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

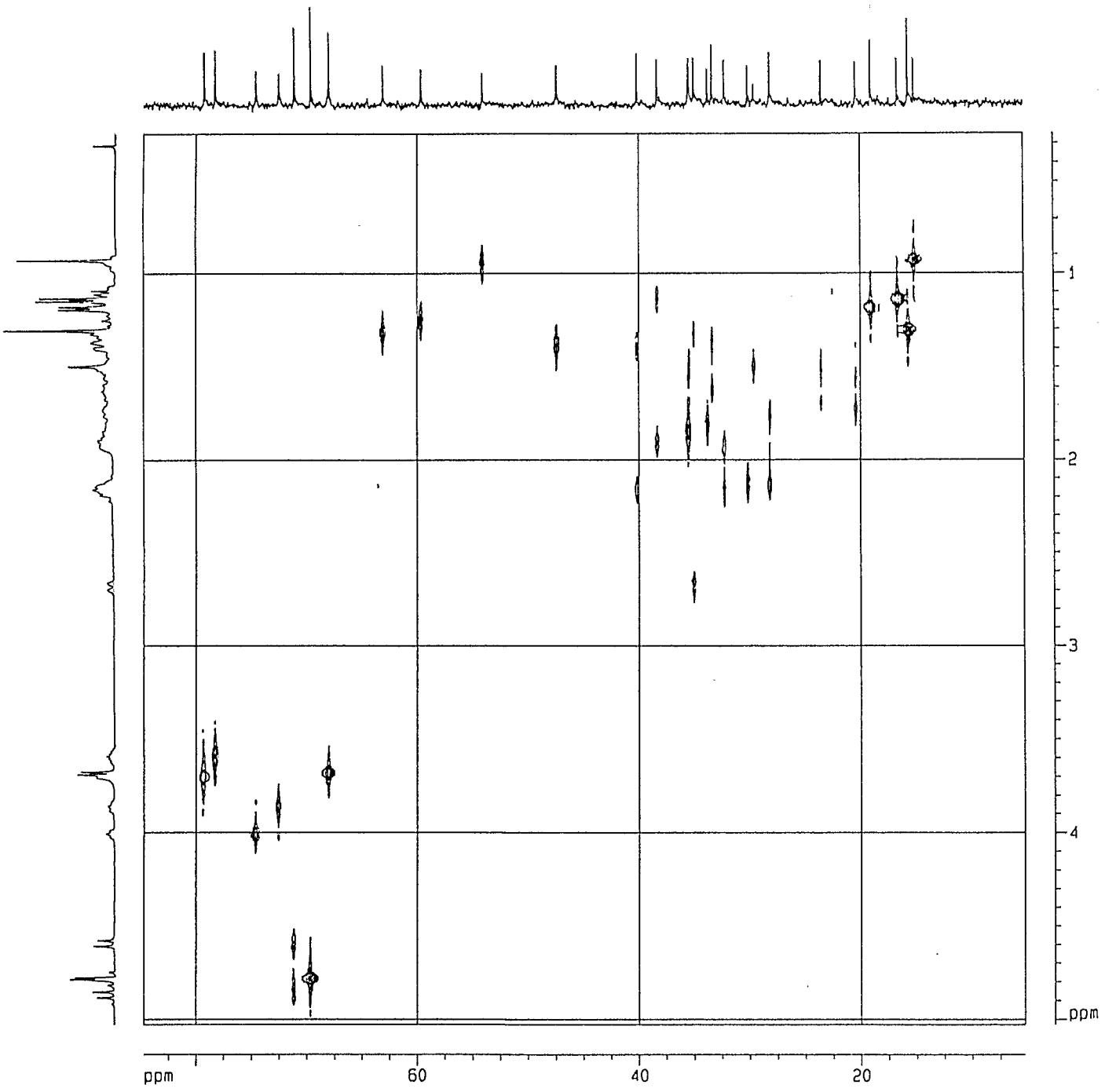
Date_	971126
Time	14.05
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	[illegible]
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	2403.846 Hz
FIDRES	2.347506 Hz
AQ	0.2130420 sec
RG	35.9
DW	200.000 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	3.00000000 sec
P1	10.40 usec
SFO1	400.1312004 MHz
NUC1	1H
PL1	7.50 dB
IN0	0.00041653 sec

F1 - Acquisition para



19

HETCOR (400 MHz)



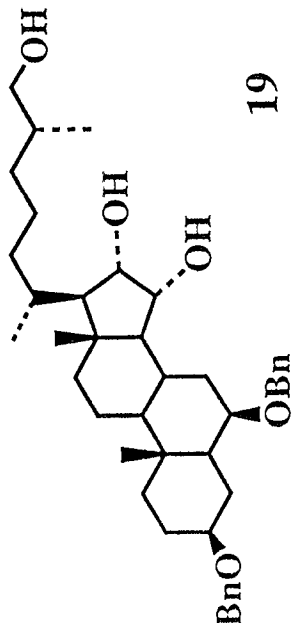
Current Data Parameters

NAME	si-oupat-hscor
EXPNO	3
PROCNO	1

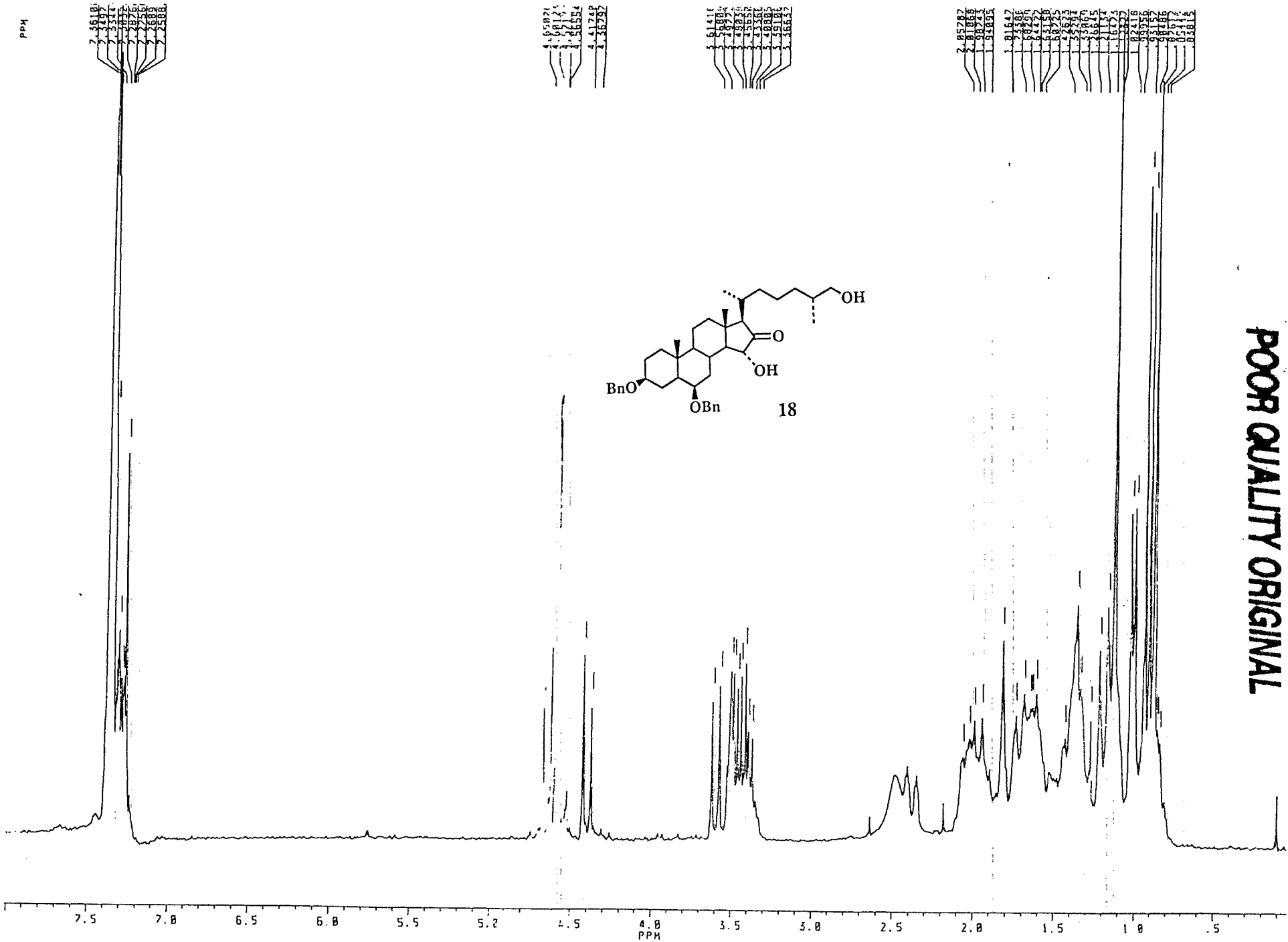
F2 - Acquisition Parameters

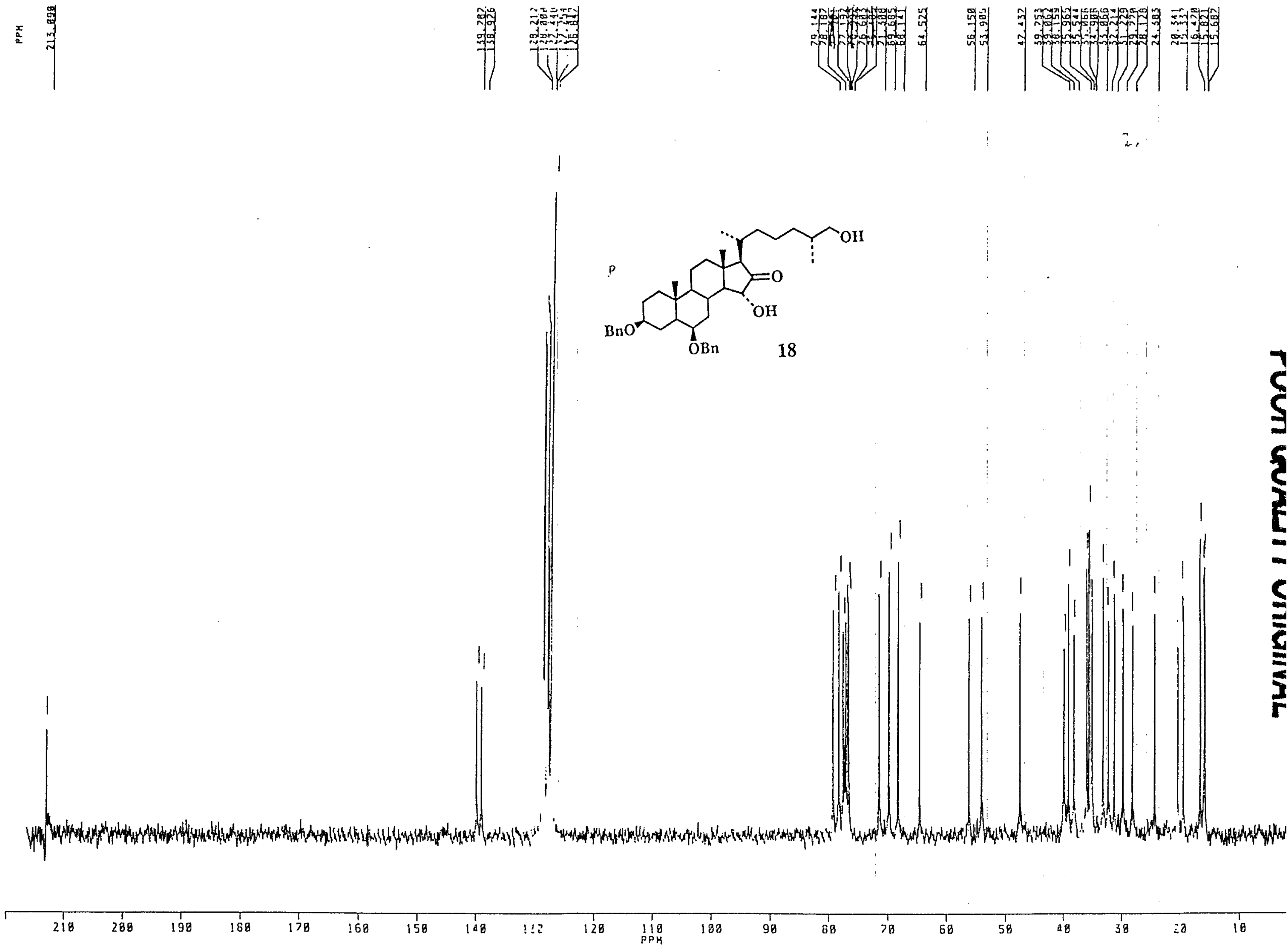
Date_	971125
Time	20 00
INSTRUM	spect
PROBHD	5 mm Dual 13
PULPROG	hscp
TD	2048
SFO1	[illegible]
NS	80
DS	16
SWH	16025.641 Hz
FIDRES	7.825020 Hz
AQ	0.0639476 sec
RG	1625.5
DW	31.200 usec
DE	6.00 usec
TE	300.0 K
P1	10.30 usec
P2	20.60 usec
d0	0.00000300 sec
CNS12	150.0000000
d2	0.00333333 sec
CNS111	1.0000000
d3	0.00222222 sec
d11	0.03000000 sec
d12	0.00002000 sec
D1	2.00000000 sec
PL2	7.50 dB
P3	10.40 usec
SFO2	400.1316005 MHz
NUC2	1H
SFO1	100.6207780 MHz
NUC1	13C
PL1	8.00 dB
PL12	27.50 dB
CPDPRG2	waltz16
PCPD2	100.00 usec
IN0	0.00015620 sec

[illegible] parameters

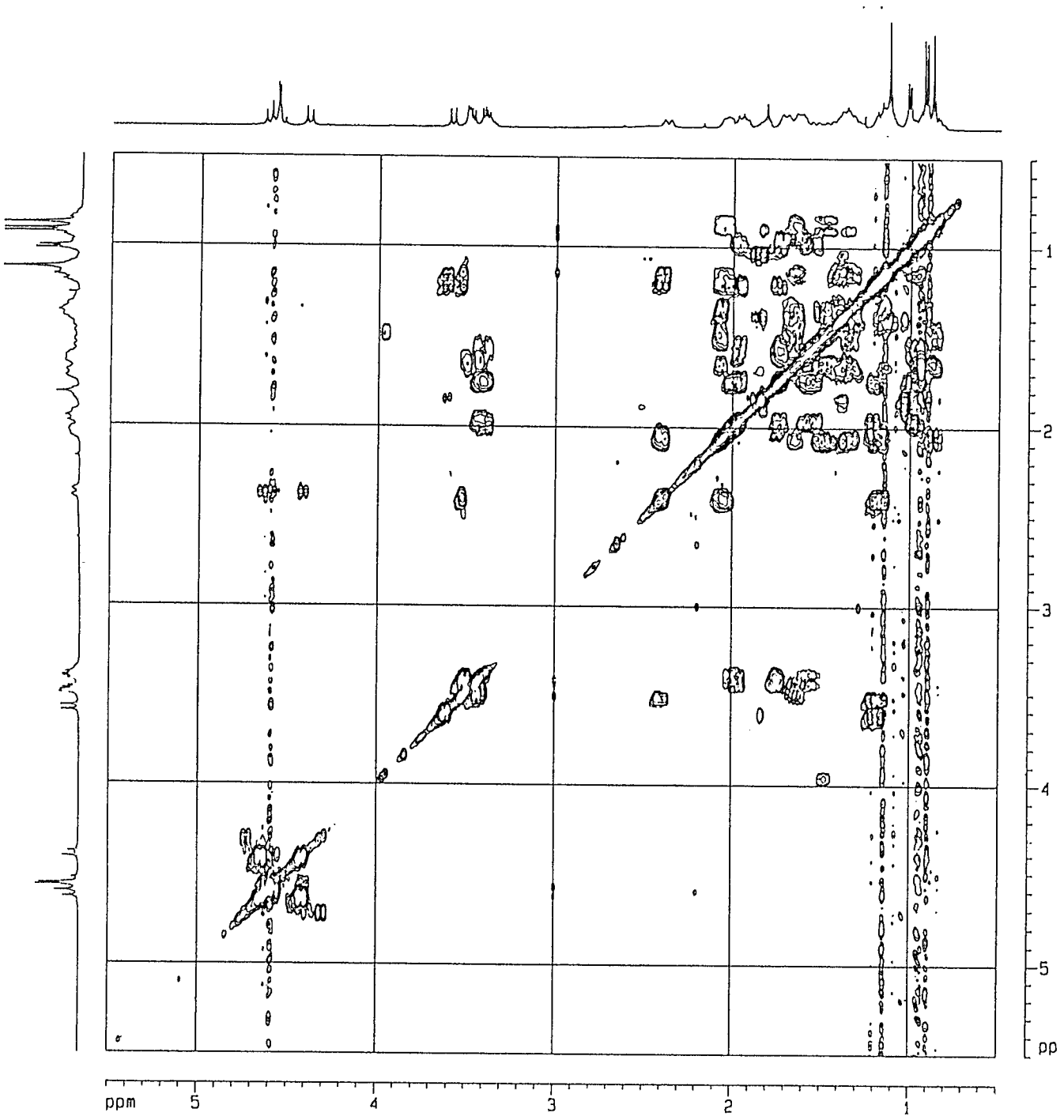


19




PPM
213.098
139.787
138.926
79.144
78.187
71.308
69.685
68.141
64.525
56.150
53.985
47.432
39.753
24.583
20.341
16.470
15.821
15.687
OH
O
OH
BnO
OBn
210
200
190
180
170
160
150
140
120
110
100
90
80
70
60
50
40
30
20
10
PPM
18

COSY (400 MHz)



Current Data Parameters

NAME	xill
EXPNO	4
PROCNO	1

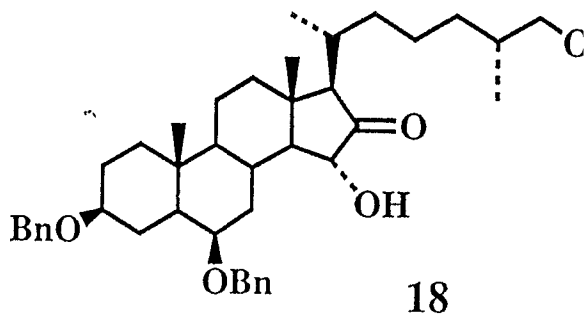
F2 - Acquisition Parameters

Date_	970918
Time	21.29
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	cosy45
TD	1024
SOLVENT	CDCl3
NS	4
DS	16
SWH	2003.205 Hz
FIDRES	1.956255 Hz
AQ	0.2556404 sec
RG	4
DW	249.600 usec
DE	6.00 usec
TE	300.0 K
D1	5.00000000 sec
P1	11.30 usec
SFO1	400.1312004 MHz
NUC1	1H
PL1	10.00 dB
D0	0.00000300 sec
IN0	0.00049984 sec

F1 - Acquisition parameters

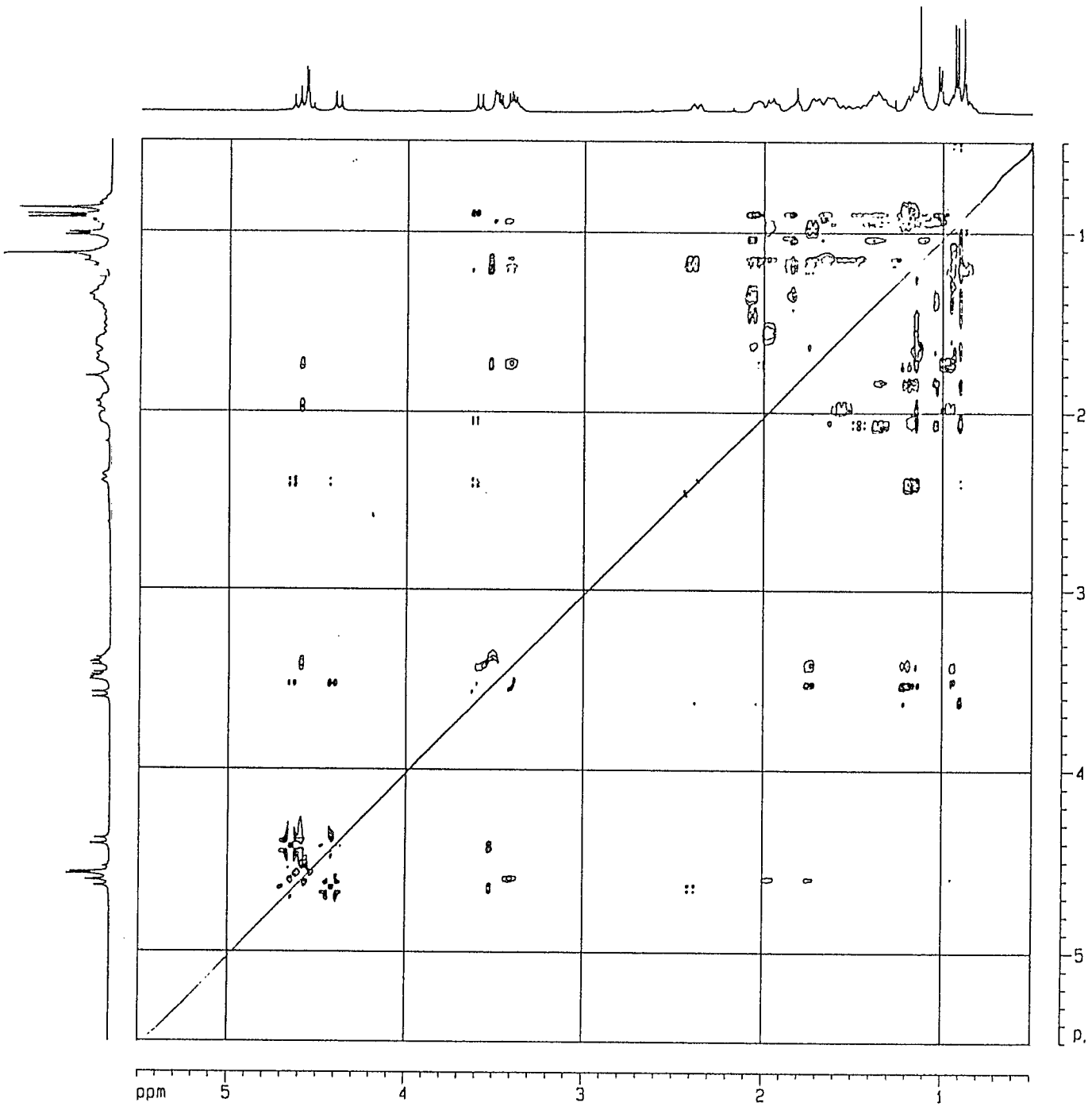
MDD	1
TD	512

$



ROESY (400 MHz)

MIXING TIME = 400ms



Current Data Parameters

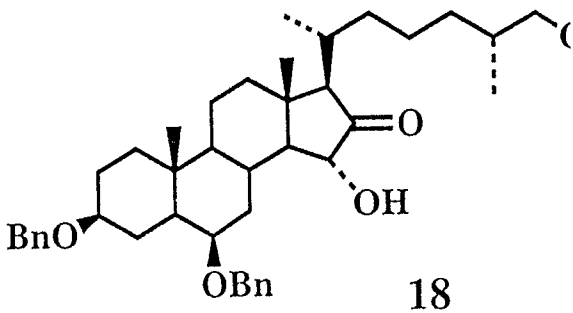
NAME	mill
EXPNO	3
PROCNO	1

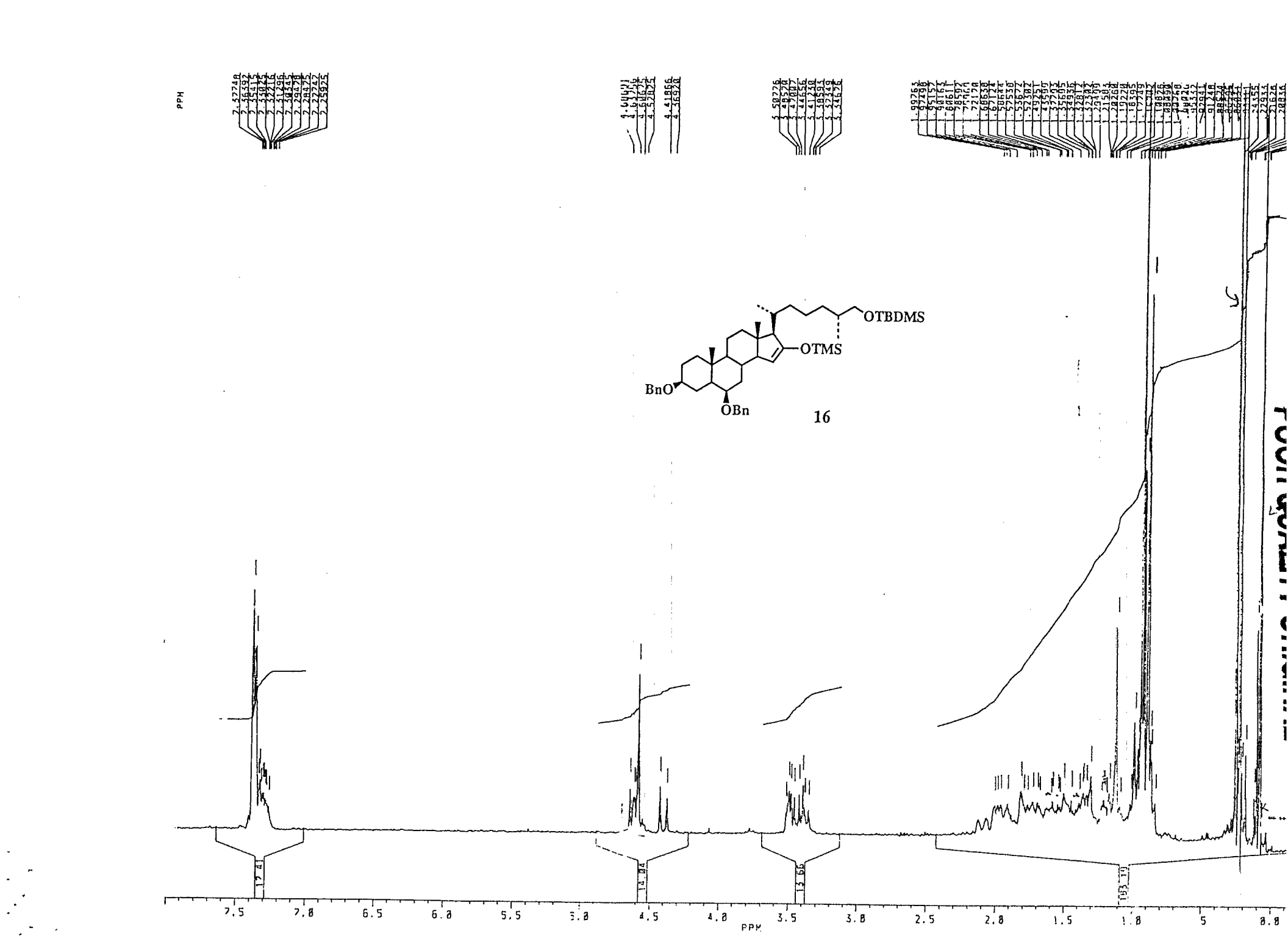
F2 - Acquisition Parameters

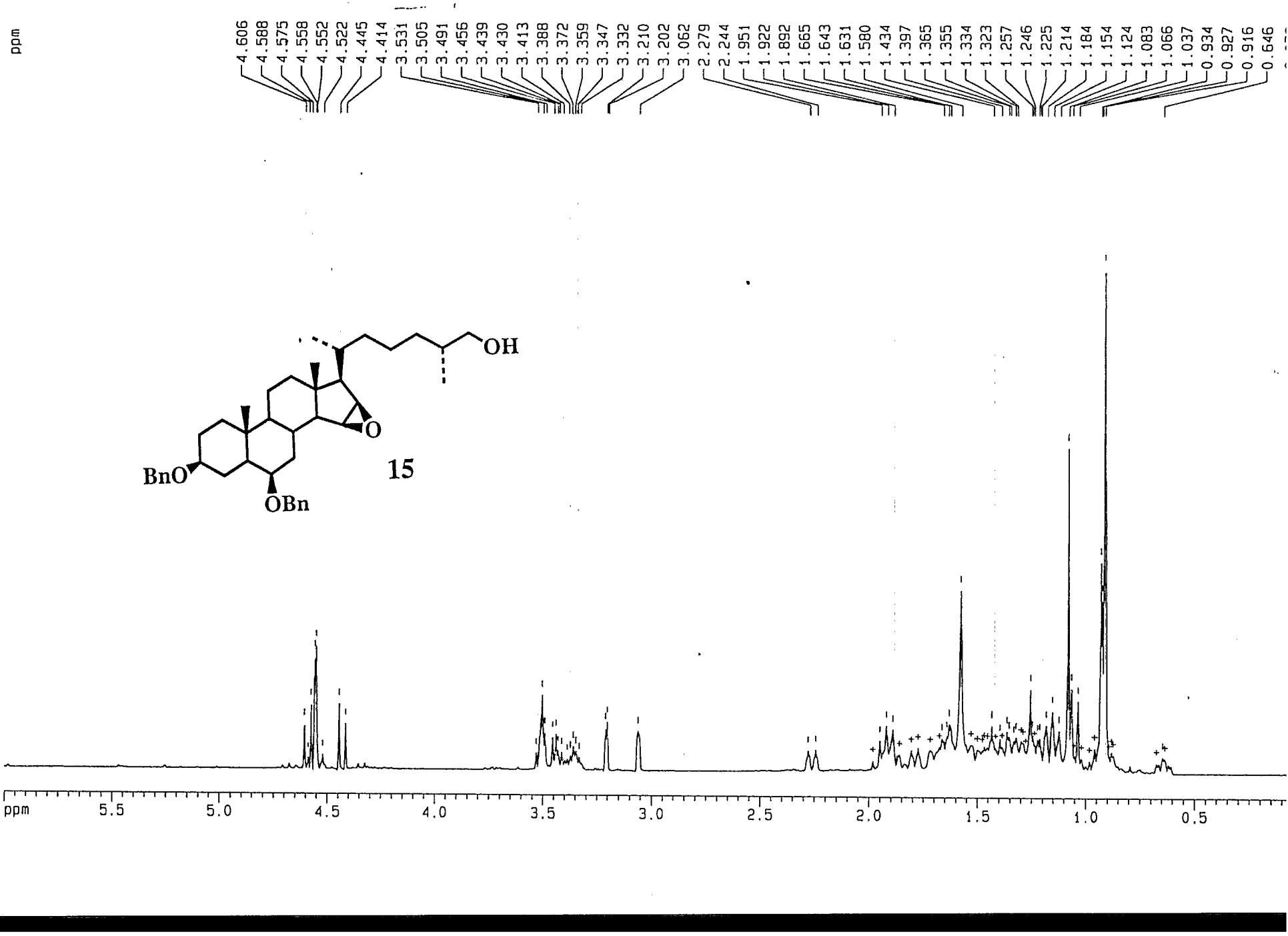
Date_	970918
Time	18 28
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	roesyld
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	2003.205 Hz
FIDRES	1.956255 Hz
AQ	0.2556404 sec
RG	4
DW	249.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D12	0.00002000 sec
PL1	10.00 dB
P1	11.30 usec
SFO1	400.1312004 MHz
NUC1	1H
D0	0.00000300 sec
PL11	31.00 dB
P15	300000.00 usec
TM0	0.00024992 sec

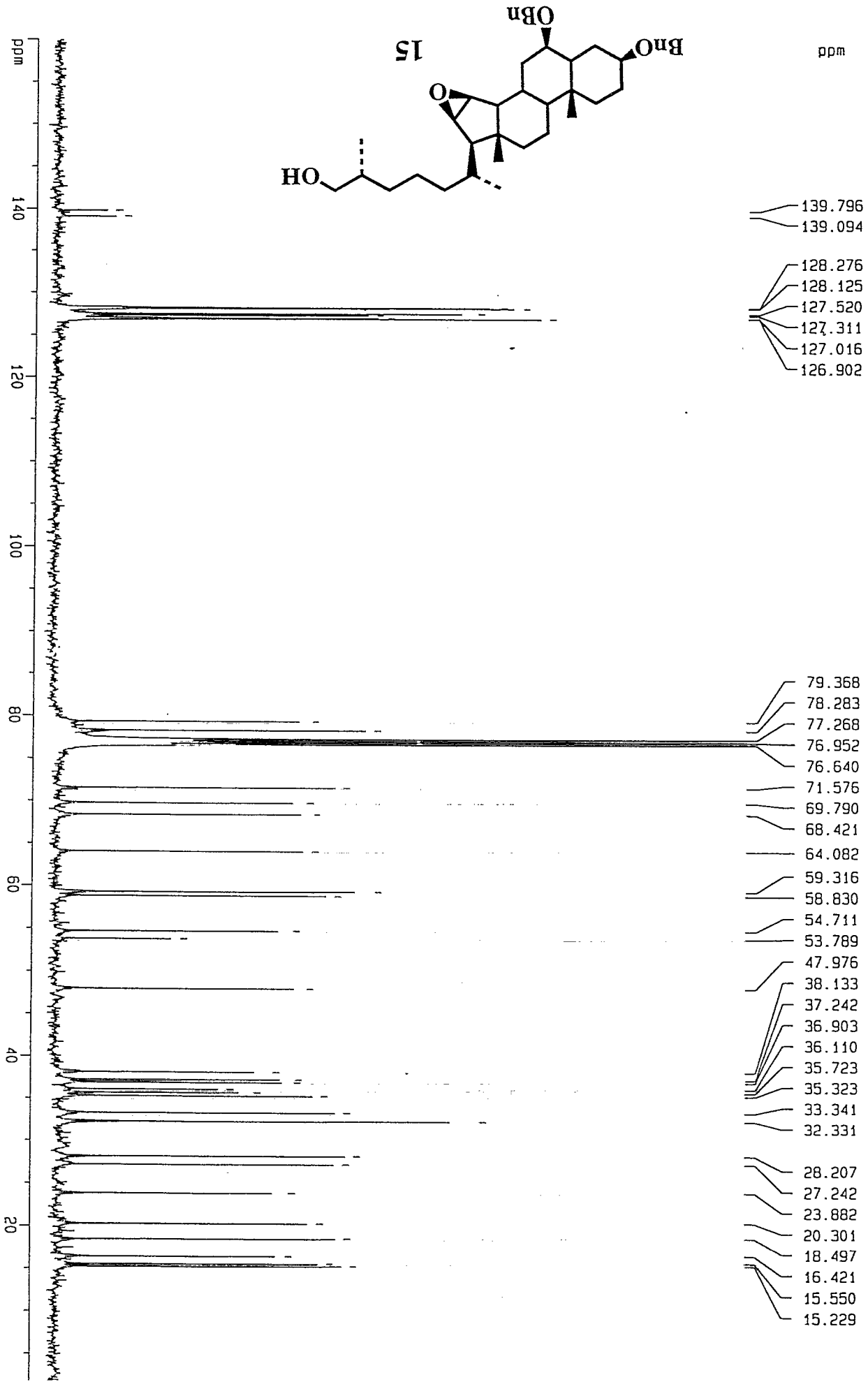
F1 - Acquisition parameters

NDO	2
TD	512
SFO1	400.1312 MHz
FIDRES	3.907531 Hz

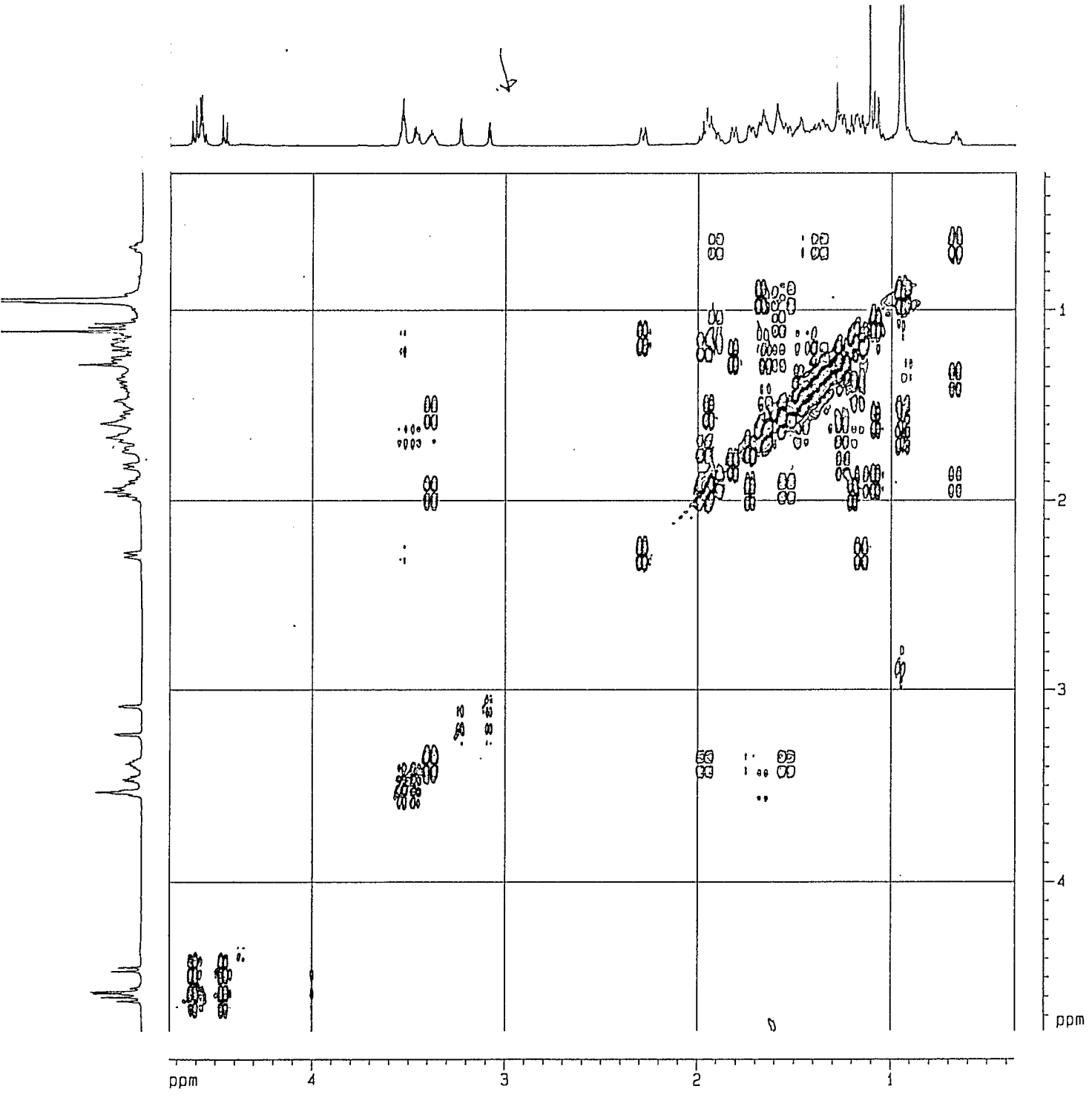


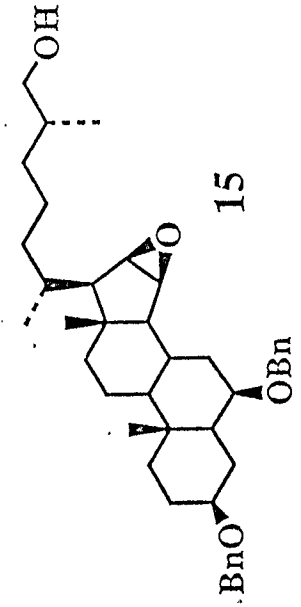

OTBDMS
OTMS
BnO
OBn
16
PPM


ppm
4.606
4.588
4.575
4.558
4.552
4.522
4.445
4.414
3.531
3.505
3.491
3.456
3.439
3.430
3.413
3.388
3.372
3.359
3.347
3.332
3.210
3.202
3.062
2.279
2.244
1.951
1.922
1.892
1.665
1.643
1.631
1.580
1.434
1.397
1.365
1.355
1.334
1.323
1.257
1.246
1.225
1.214
1.184
1.154
1.124
1.083
1.066
1.037
0.934
0.927
0.916
0.646
OH
O
BnO
OBn
15
ppm
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5


ppm
140
120
100
80
60
40
20
15
OBn
BnO
O
HO
ppm
139.796
139.094
128.276
128.125
127.520
127.311
127.016
126.902
79.368
78.283
77.268
76.952
76.640
71.576
69.790
68.421
64.082
59.316
58.830
54.711
53.789
47.976
38.133
37.242
36.903
36.110
35.723
35.323
33.341
32.331
28.207
27.242
23.882
20.301
18.497
16.421
15.550
15.229

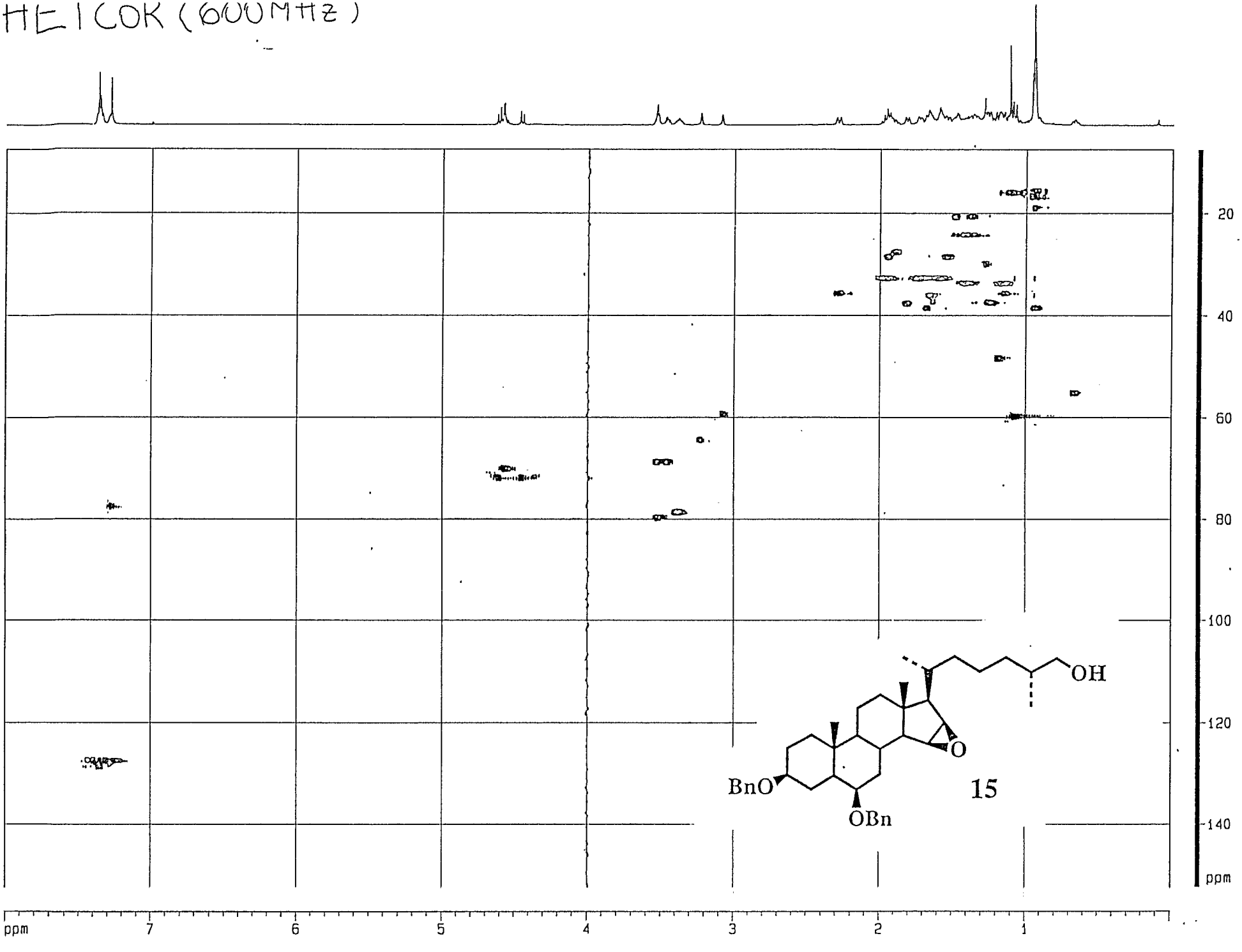
COSY (600MHz)





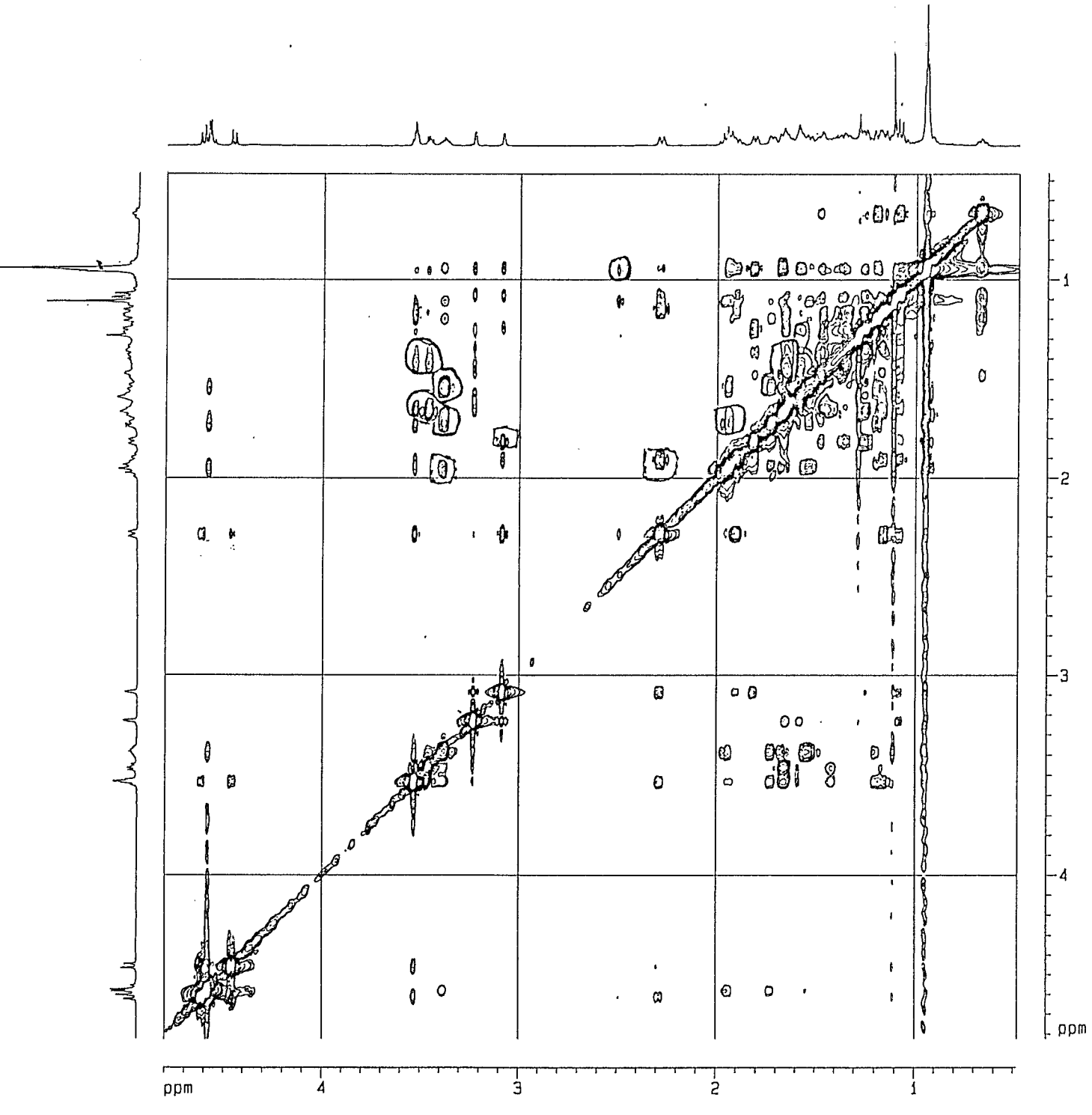
15

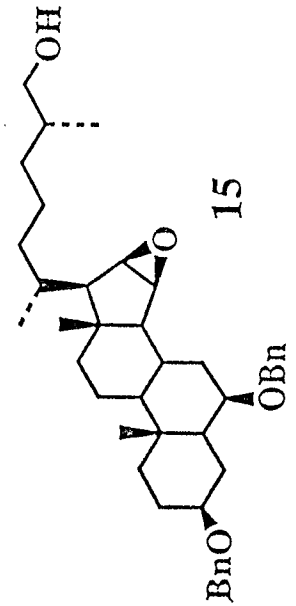
HETCOR (600MHz)



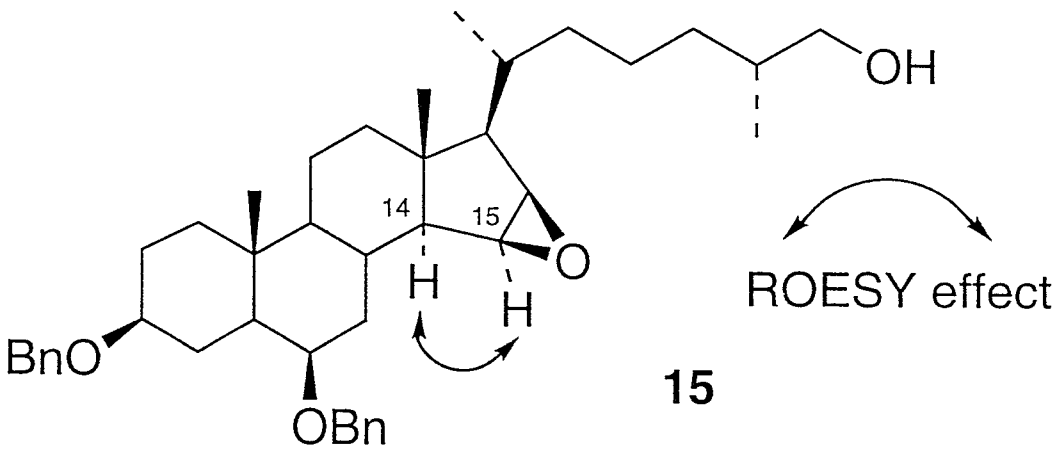
ROESY (600 MHz)

MIXING TIME : 400 ms



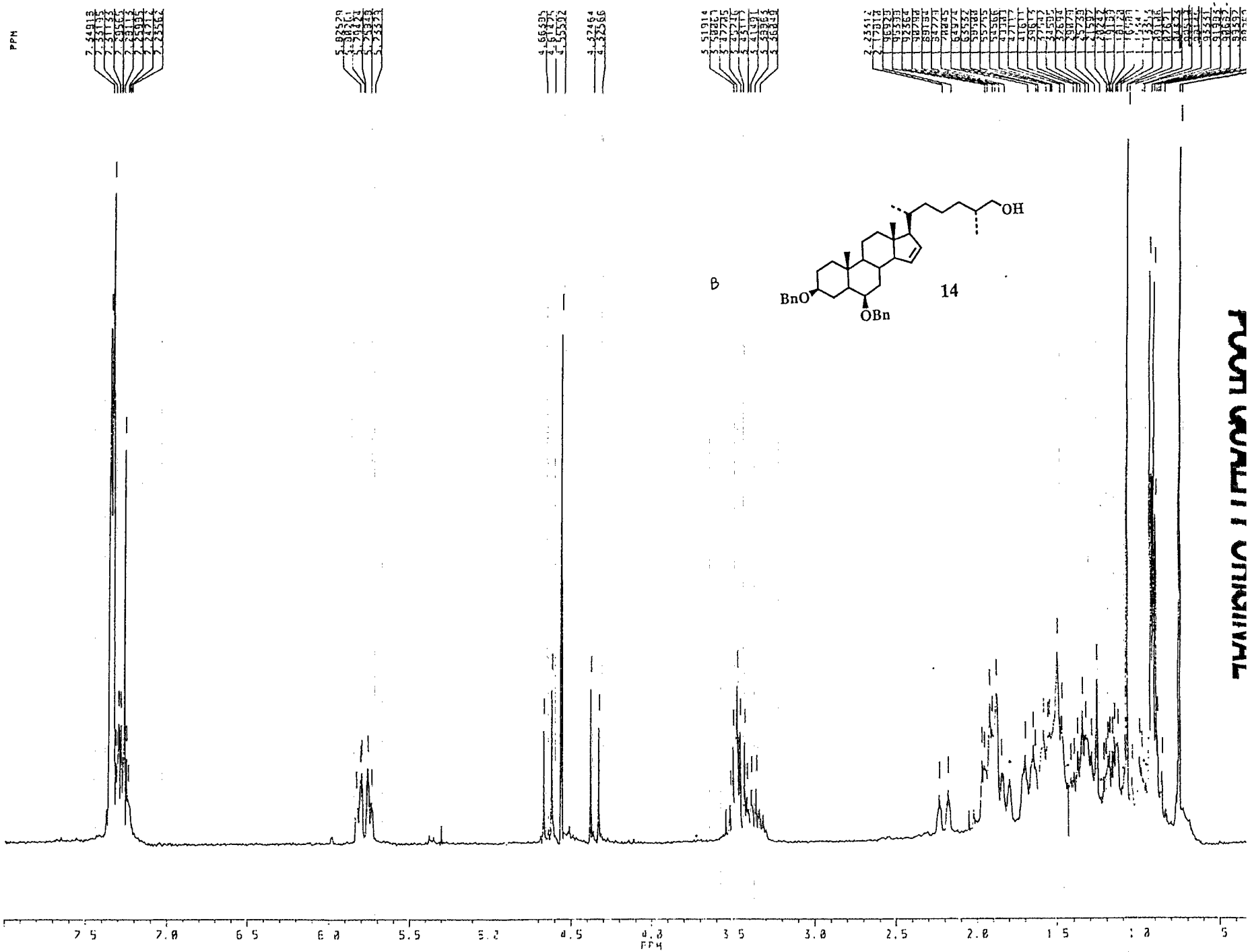


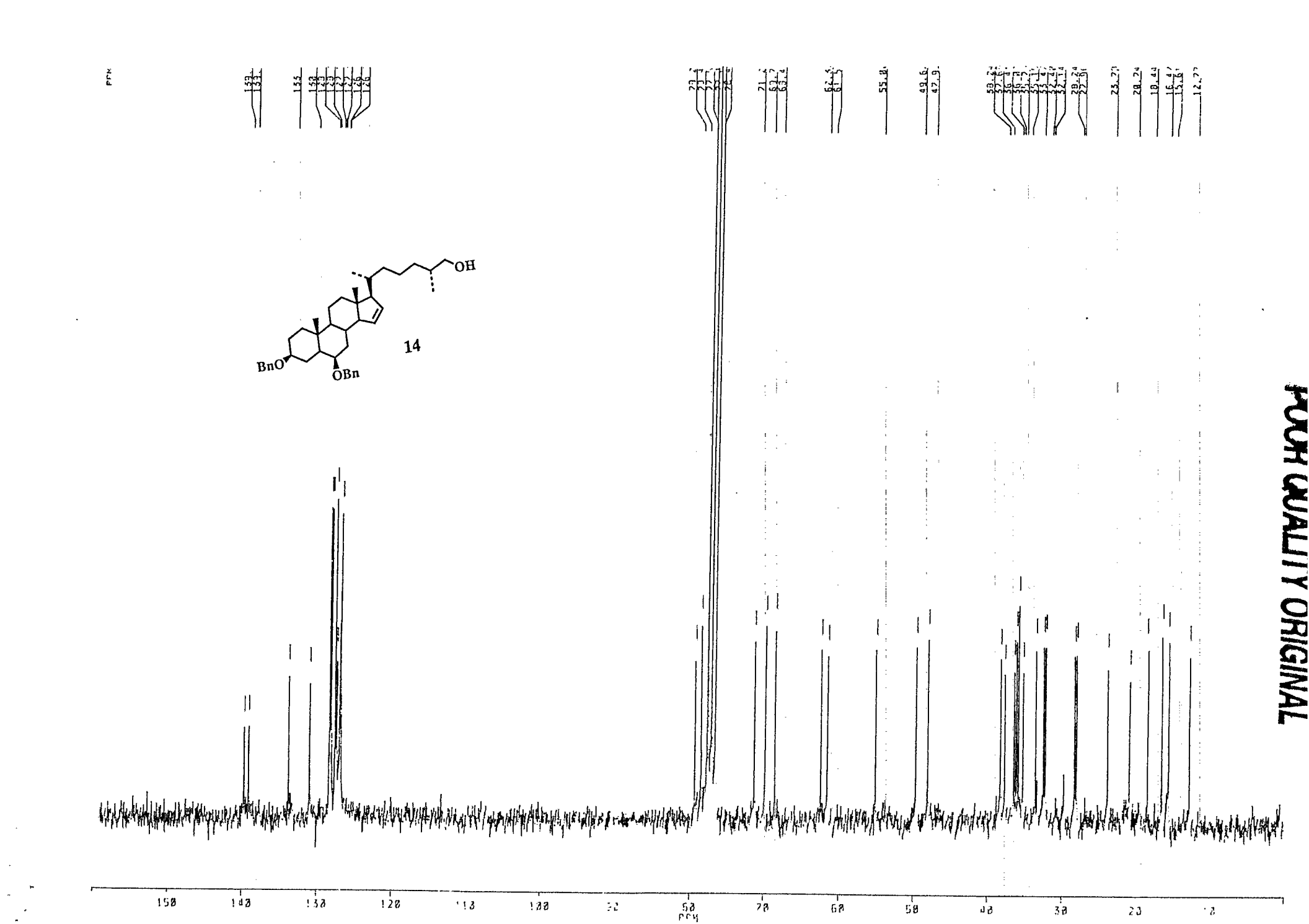
15

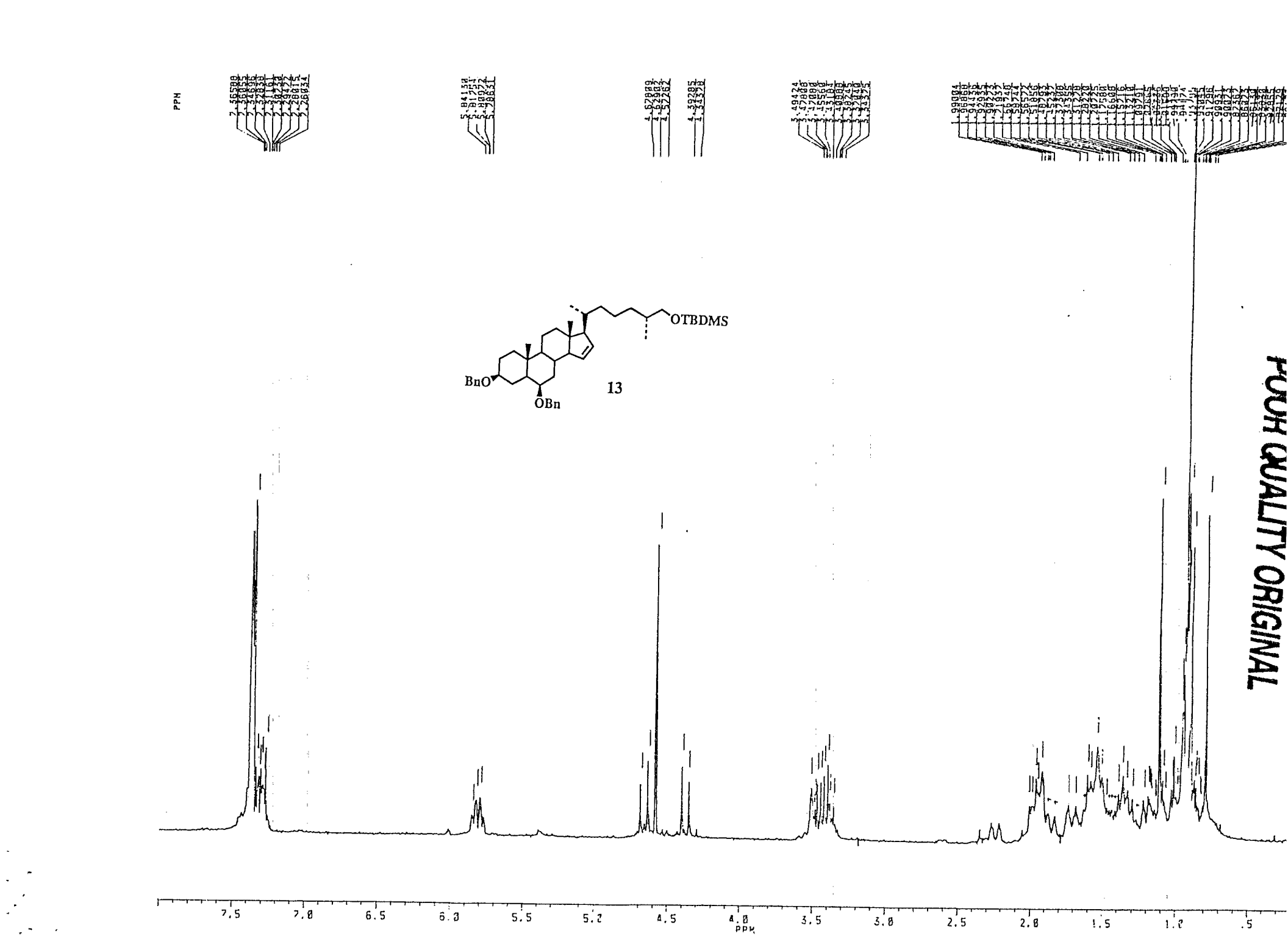


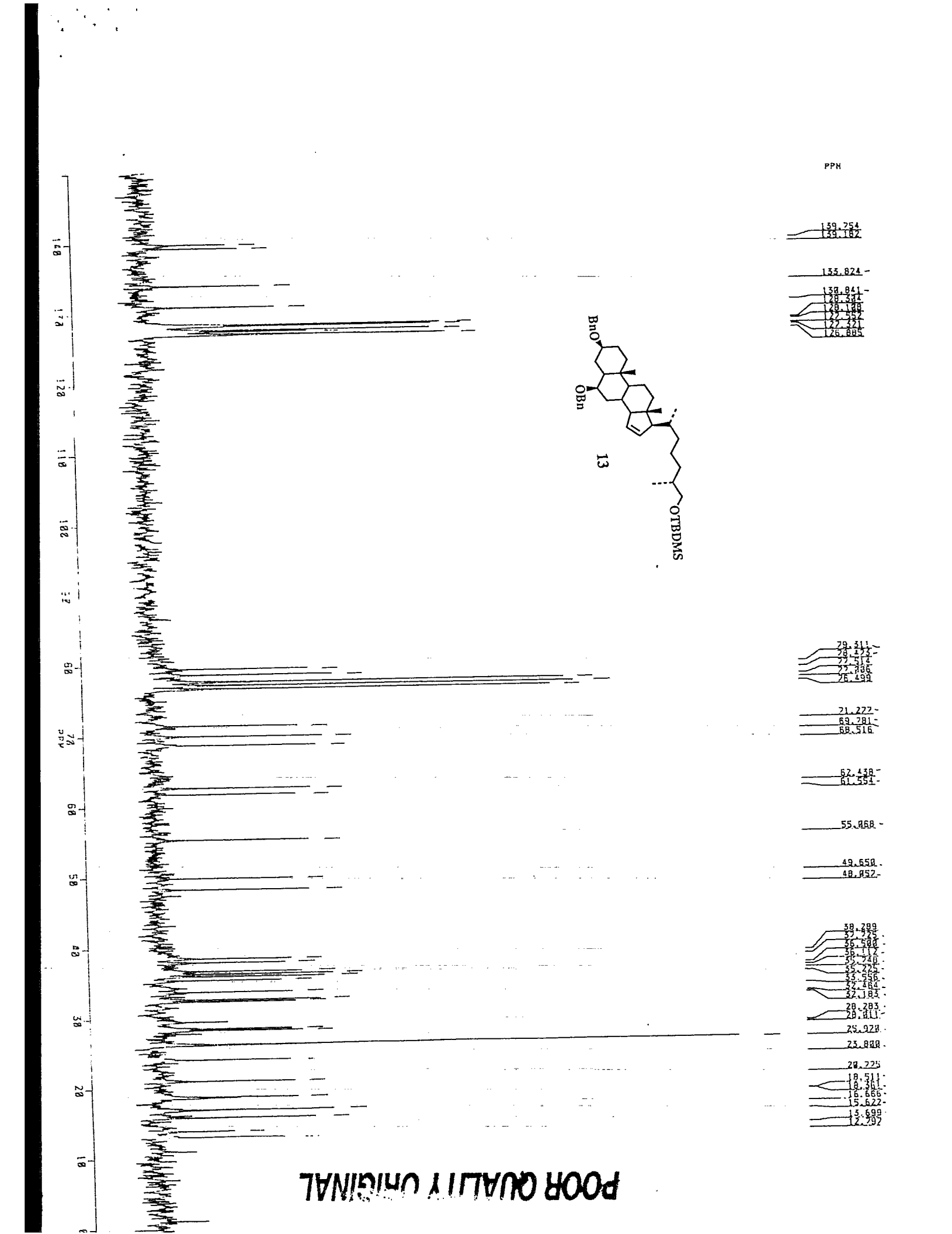
15

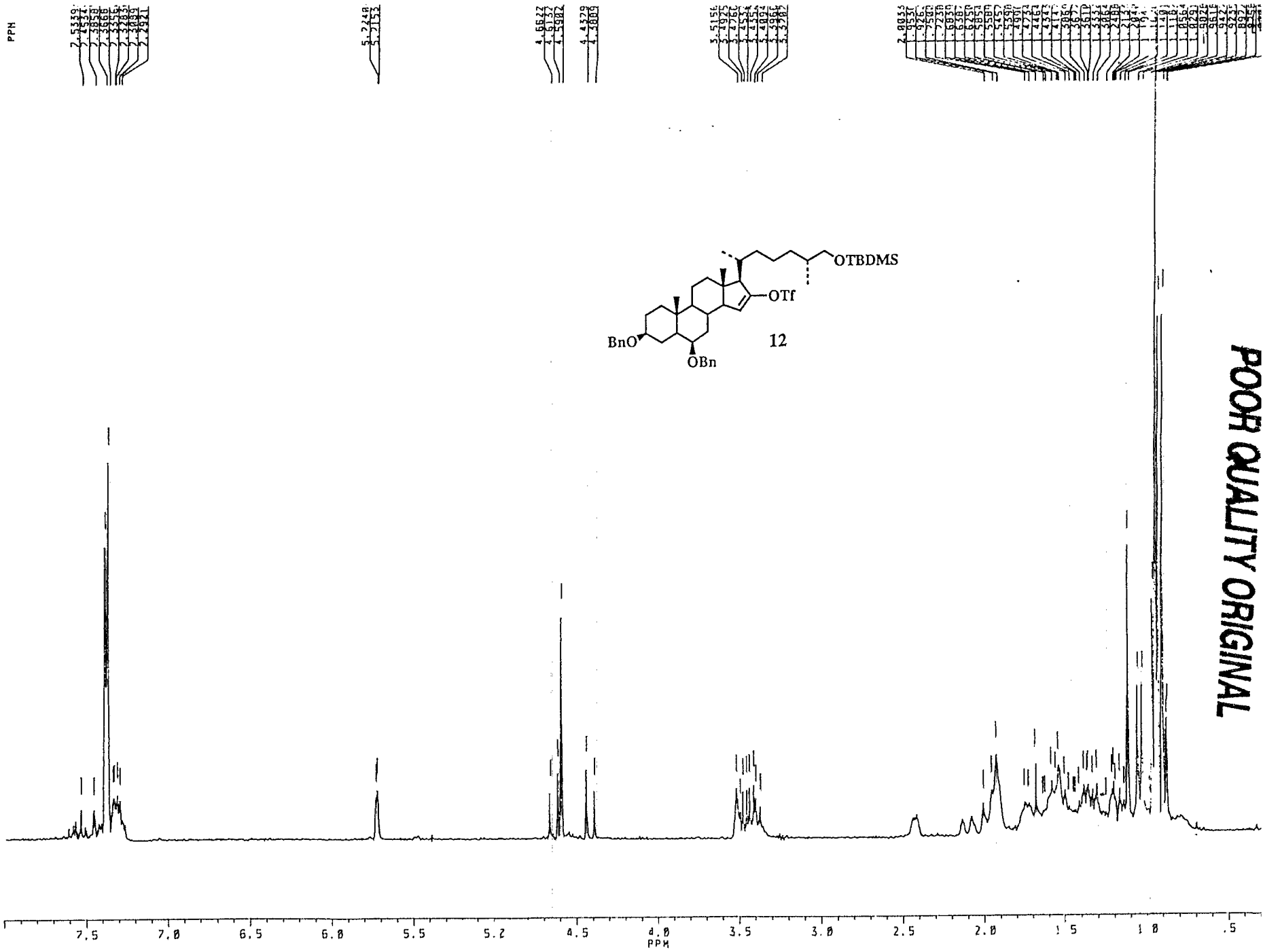
$\delta_{H\text{-}14}$ = 1.05 ppm, $\delta_{C\text{-}14}$ = 59.3 ppm
$\delta_{H\text{-}15}$ = 3.06 ppm

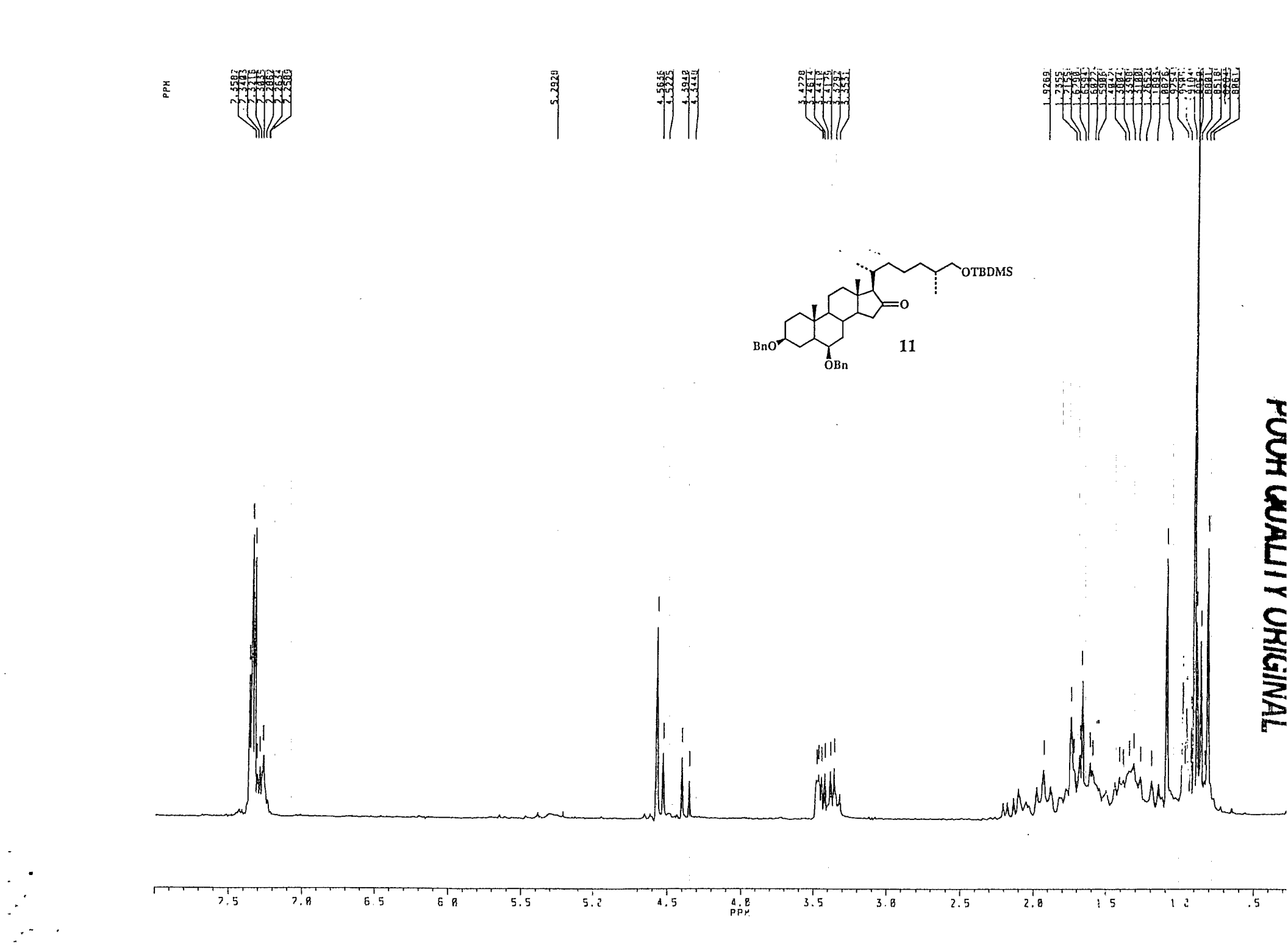

PPM
B
BnO
OBn
OH
14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
PPM

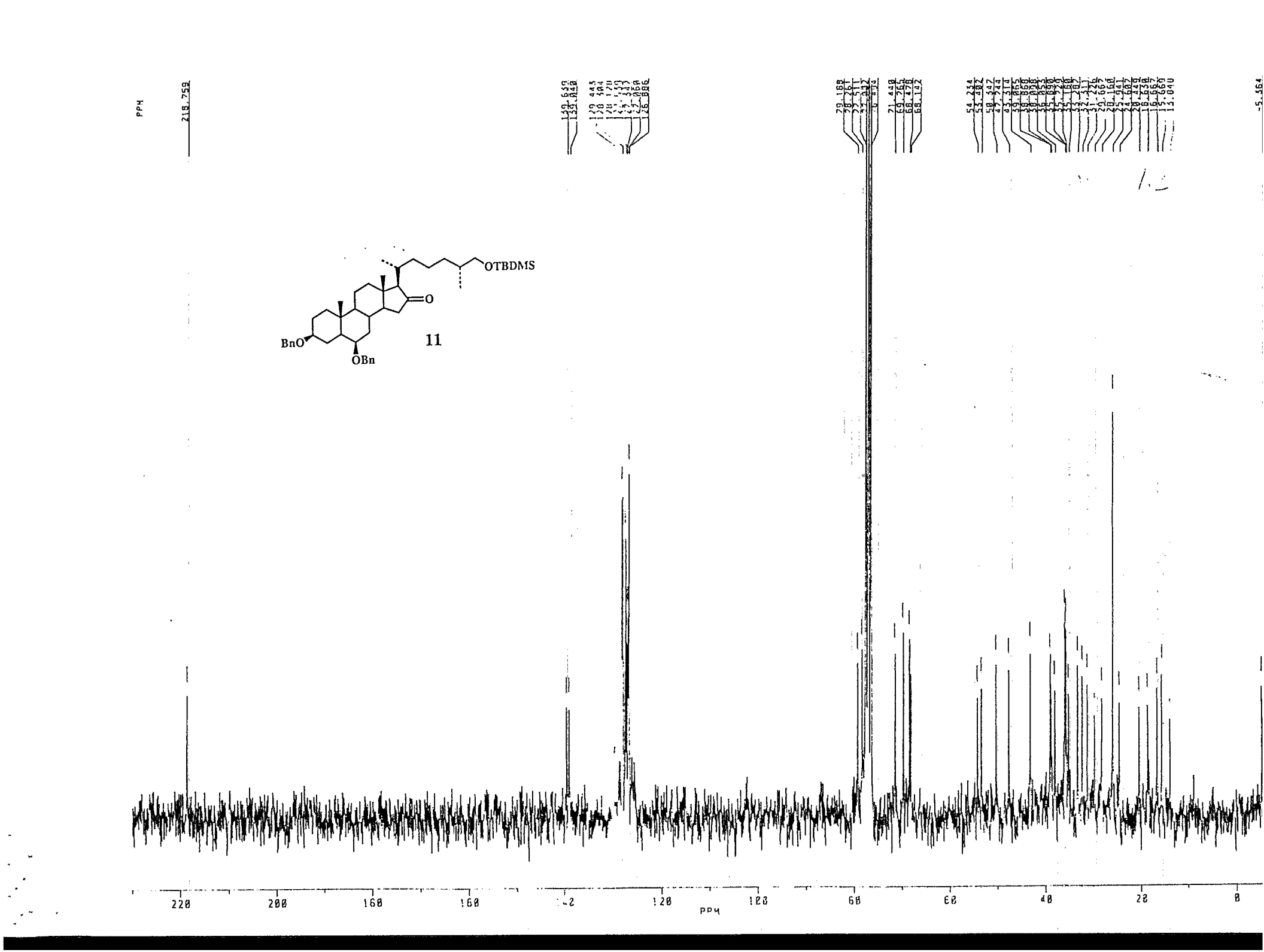


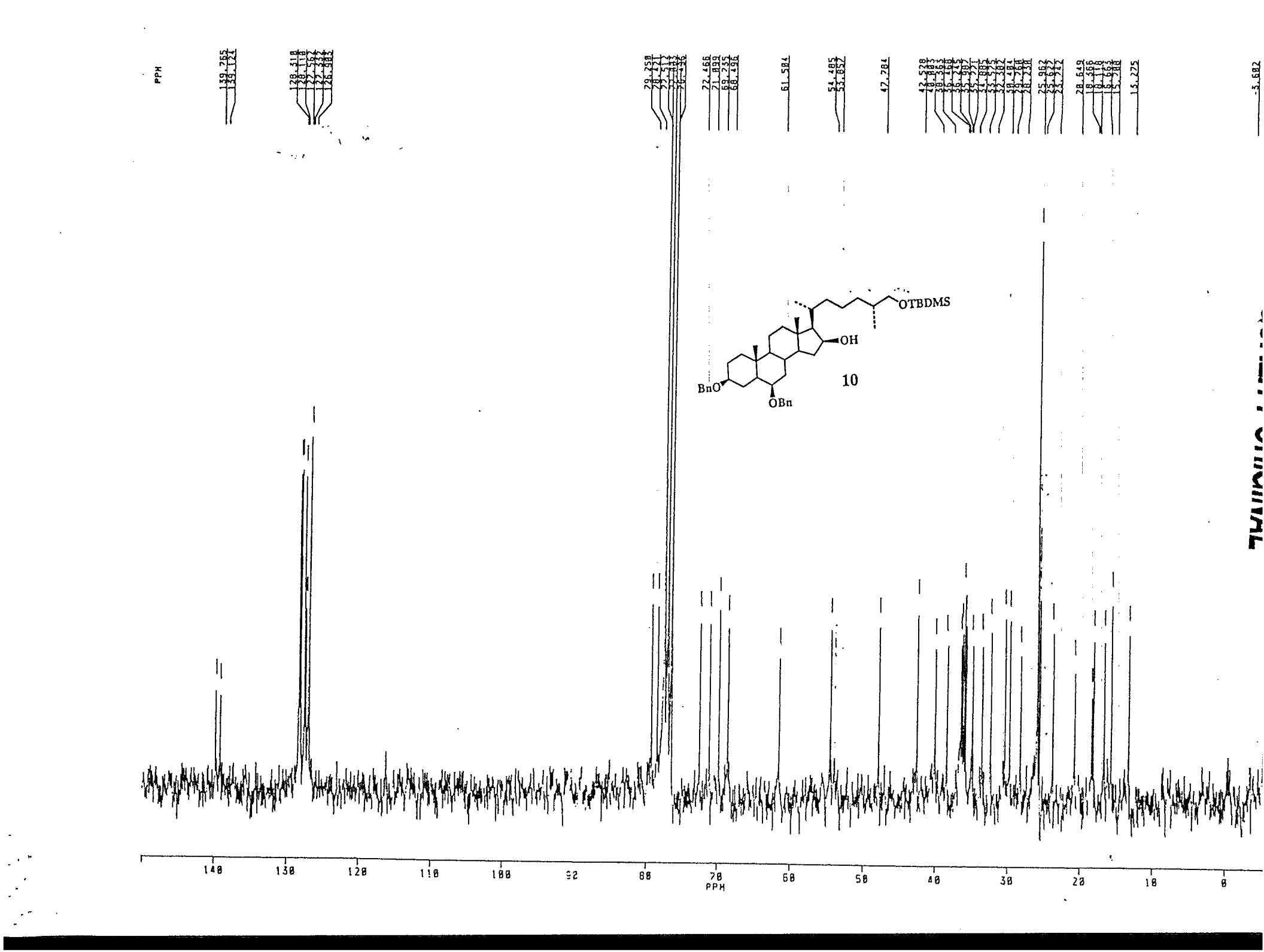


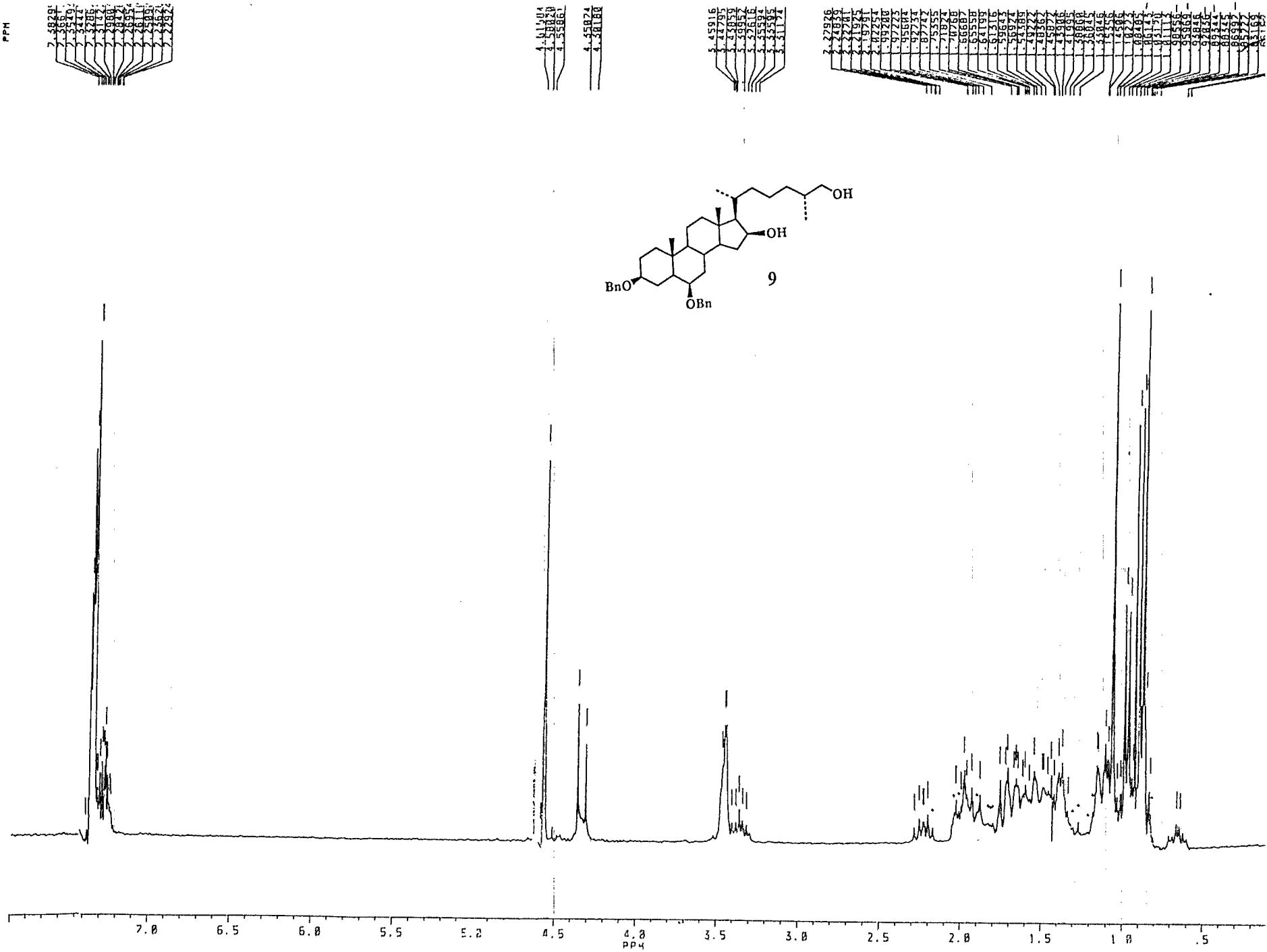


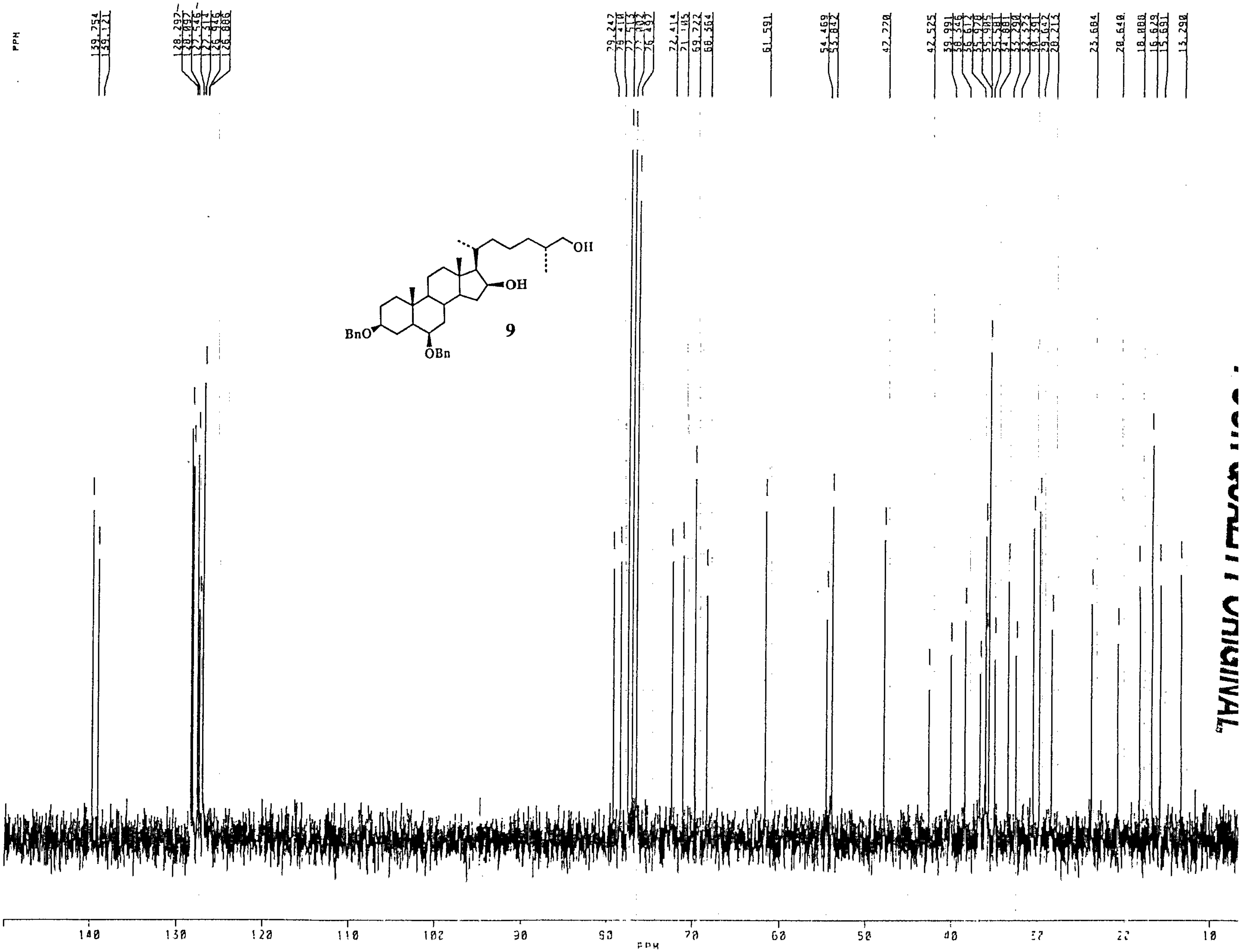




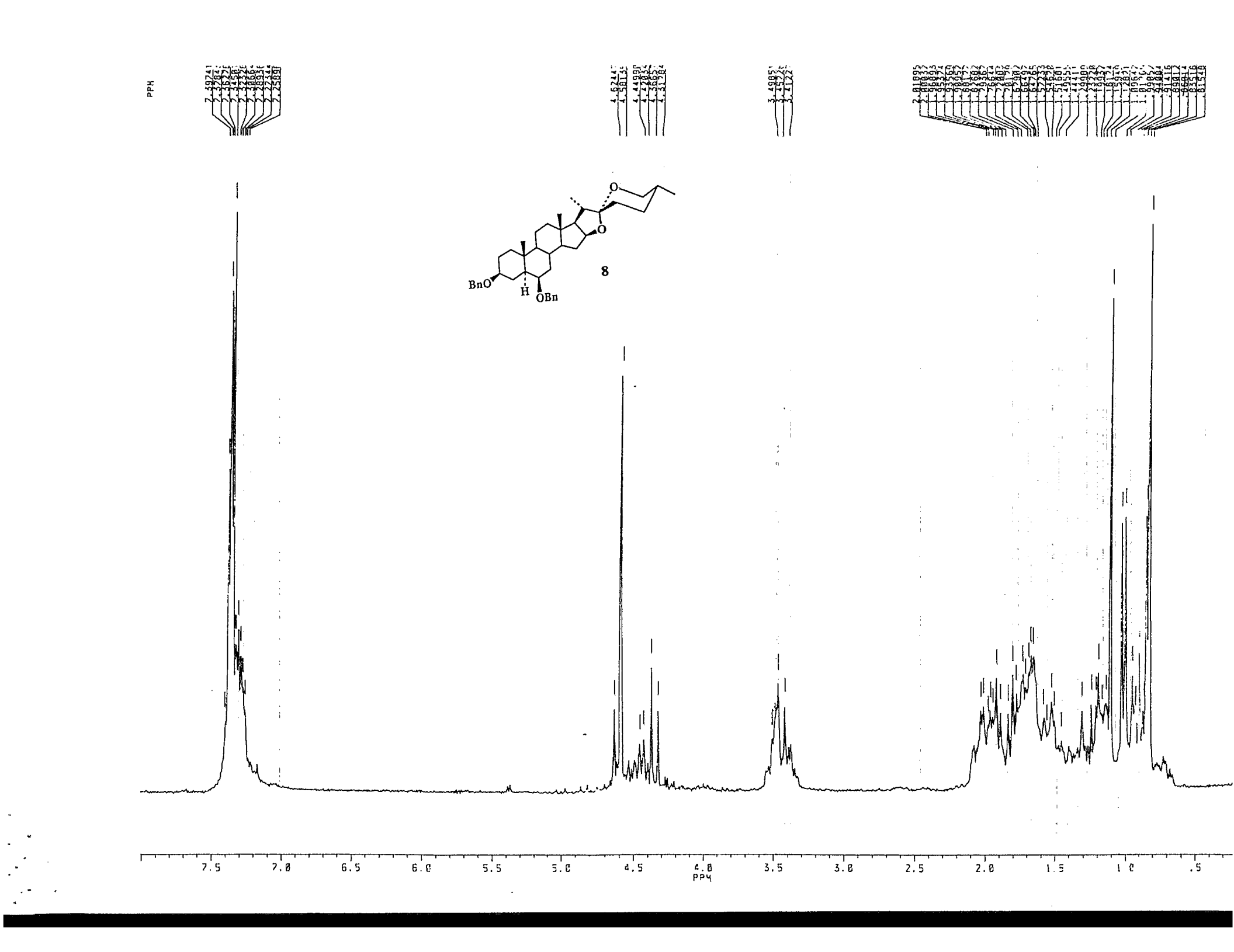

OTBDMS
O
BnO
OBn
11
PPM
220
200
180
160
140
120
100
80
60
40
20
0

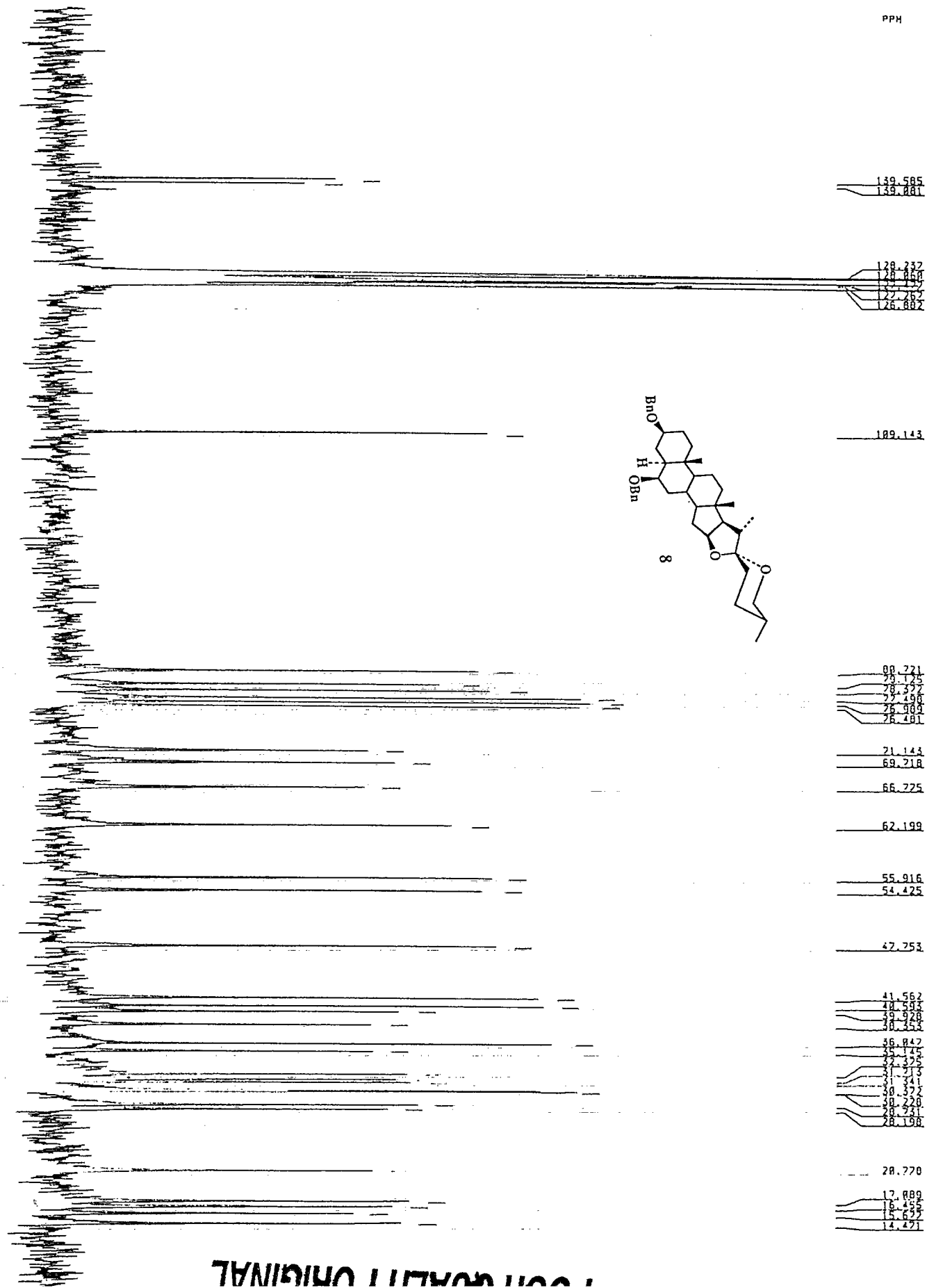

OTBDMS
OH
BnO
OBn
10
PPM


PPM
OH
OH
BnO
OBn
9
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
.5
PPM

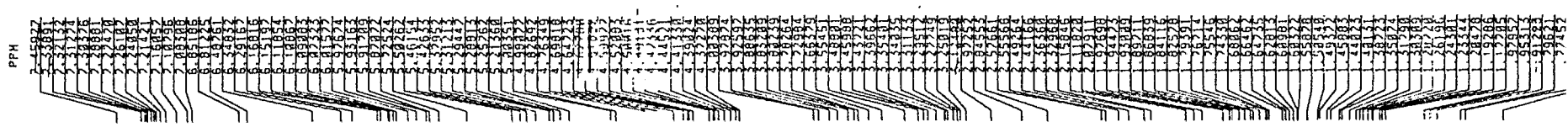


PPM
139.254
139.121
128.292
72.414
71.105
69.722
68.564
61.591
54.469
53.842
47.220
42.525
39.991
23.684
20.640
18.880
16.629
15.691
13.290
OH
OH
BnO
OBn
9
140
130
120
110
100
90
80
70
60
50
40
30
20
10
PPM

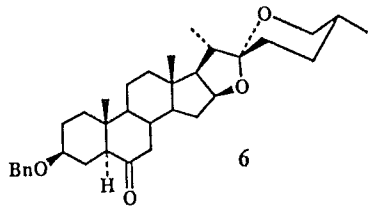

PPM
BnO
H
OBn
O
O
8
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
PPM


PPM
139.585
139.081
128.232
126.802
109.143
80.221
76.125
78.372
77.466
76.909
76.481
71.143
69.218
66.725
62.199
55.916
54.425
42.753
41.562
40.593
39.928
38.353
36.847
35.145
33.325
32.452
31.213
31.341
30.372
30.220
29.931
28.198
20.770
17.889
16.455
15.657
14.471
BnO
H
OBn
O
O
8

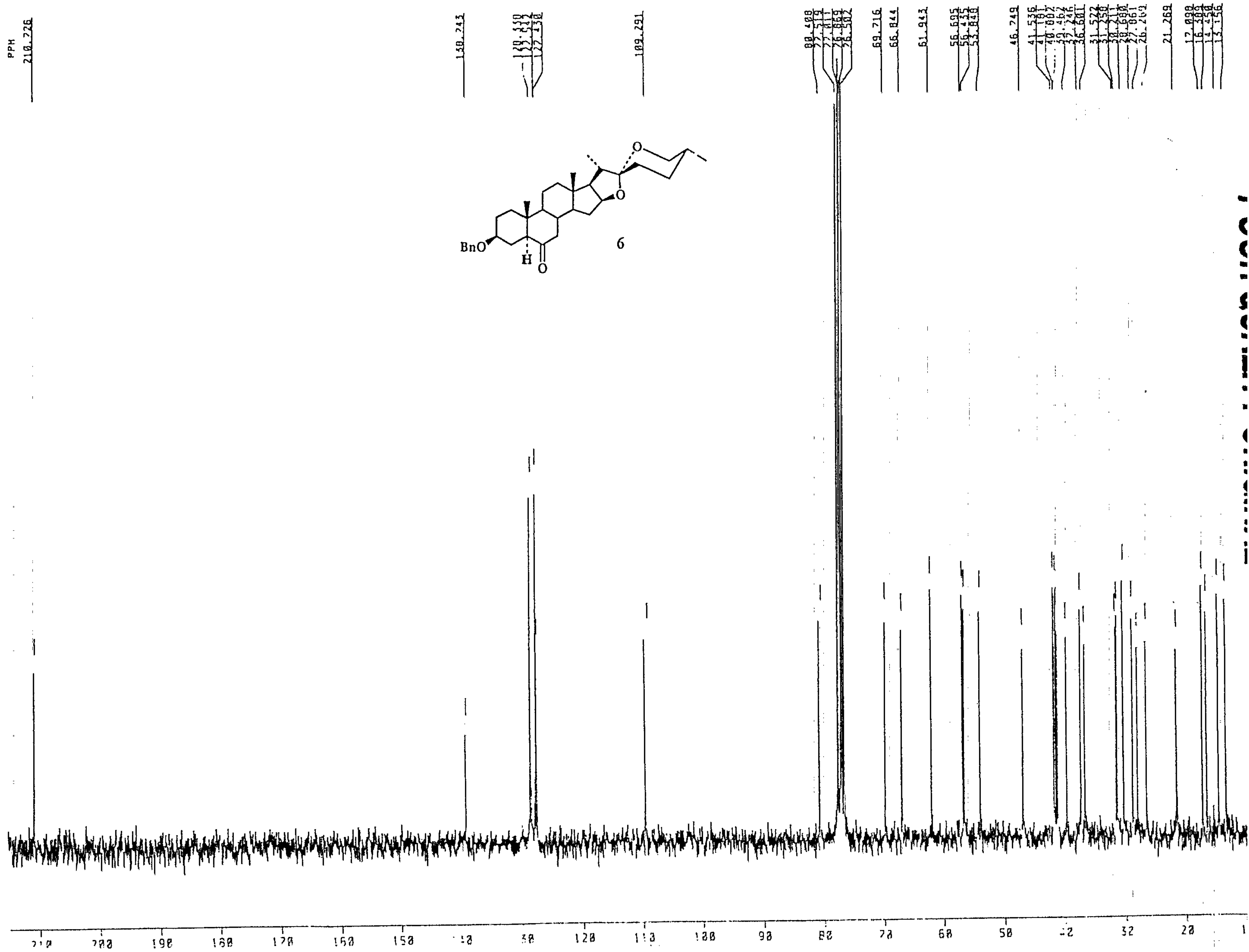
PPM
138.841
128.215
127.416
127.282
109.185
84.654
78.117
77.586
76.939
76.408
71.523
62.664
56.283
51.965
55.989
54.151
42.181
41.459
40.454
39.867
39.602
38.291
35.658
31.835
31.646
31.165
30.148
29.810
28.603
28.126
28.214
17.892
16.428
15.622
14.482
BnO
H
OH
O
O
7
140
130
120
110
100
90
80
70
60
50
40
30
20
10
F1 PPM



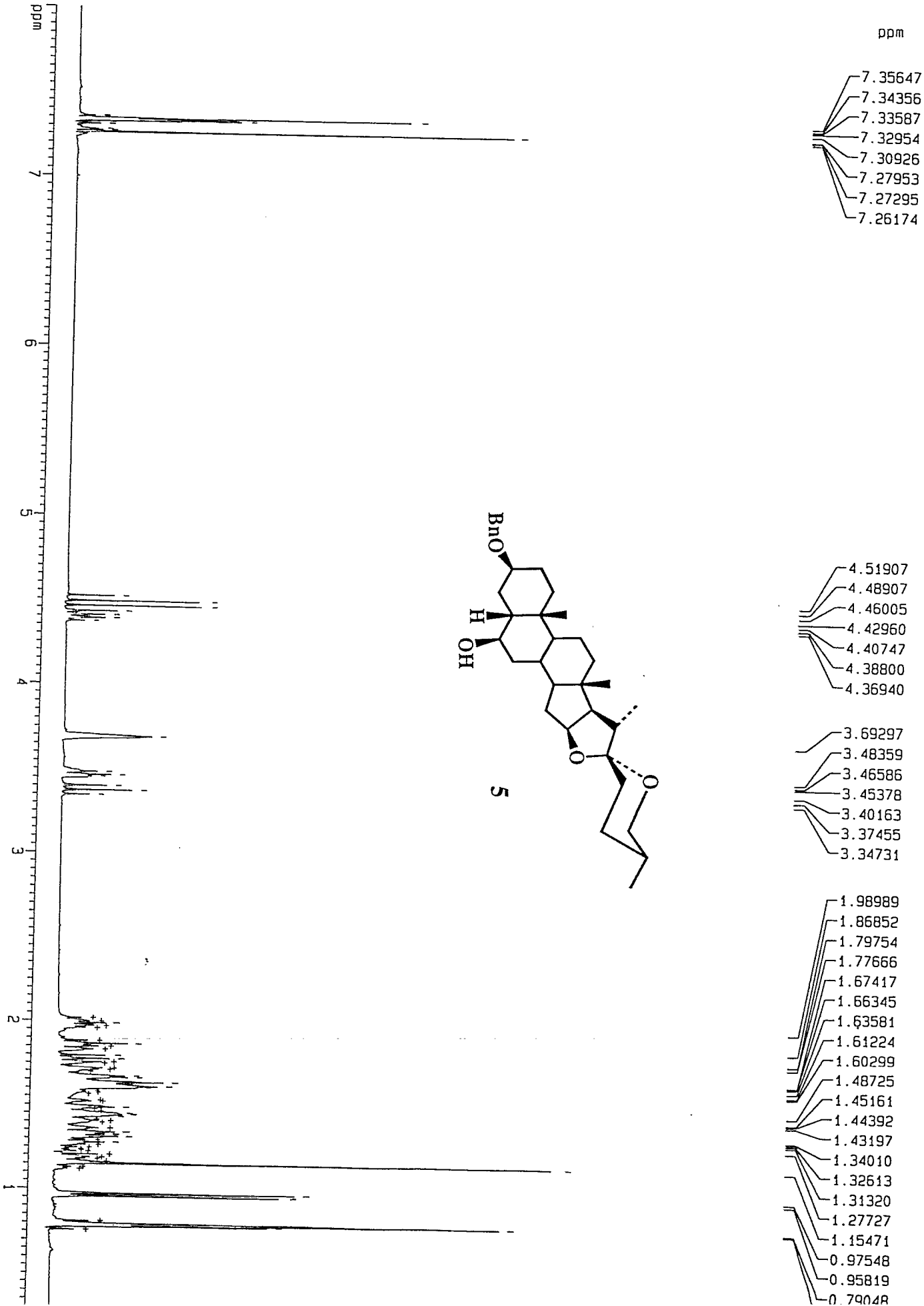
WIL 30

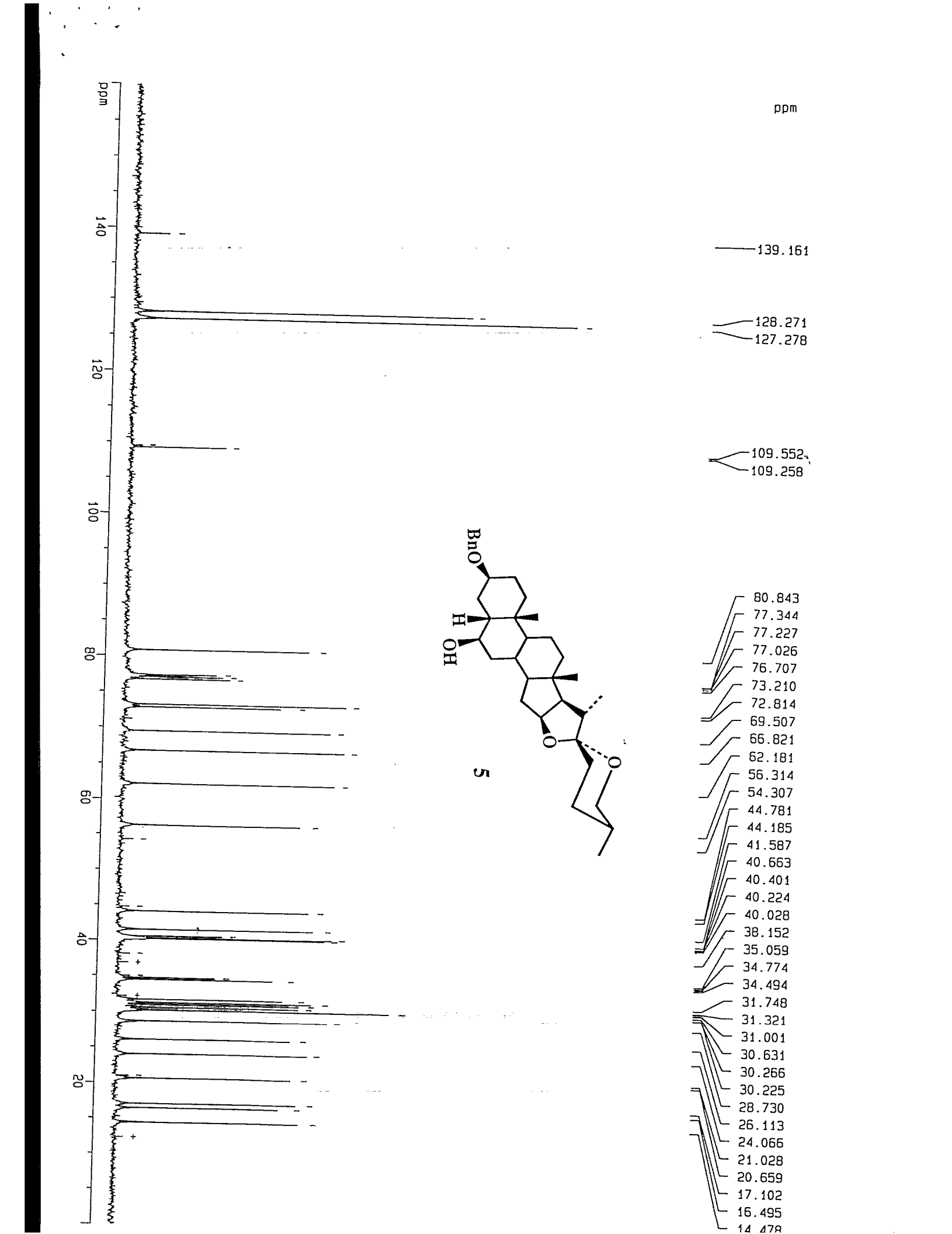


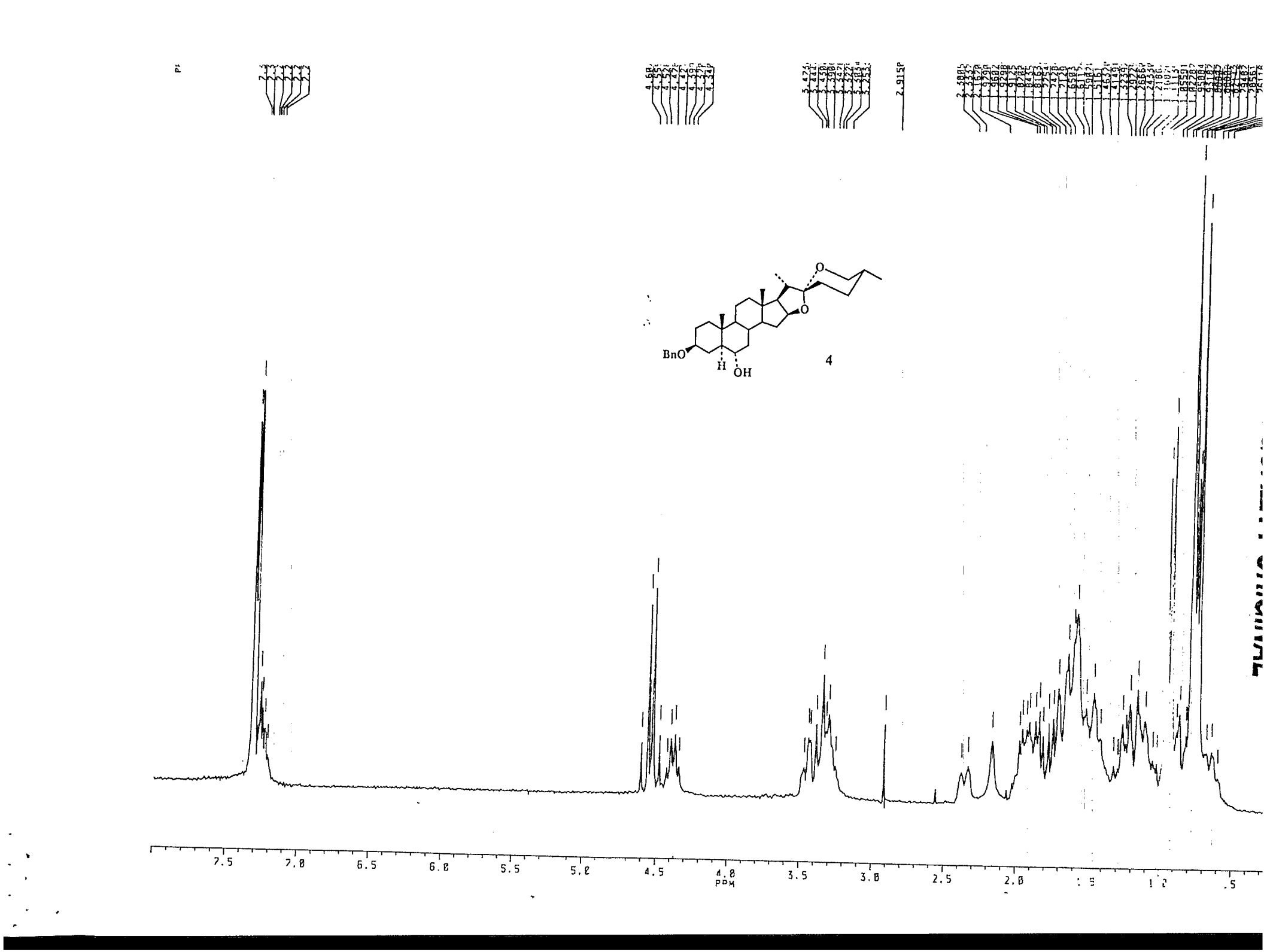
6



PPM
210.226
138.243
109.291
80.488
77.519
76.502
69.716
66.844
61.943
56.695
56.435
46.249
41.536
41.181
40.082
39.462
37.246
36.601
31.522
21.269
17.098
16.309
14.450
13.156
BnO
H
O
6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20


ppm
7.35647
7.34356
7.33587
7.32954
7.30926
7.27953
7.27295
7.26174
BnO
H
HO
O
O
5
4.51907
4.48907
4.46005
4.42960
4.40747
4.38800
4.36940
3.69297
3.48359
3.46586
3.45378
3.40163
3.37455
3.34731
1.98989
1.86852
1.79754
1.77666
1.67417
1.66345
1.63581
1.61224
1.60299
1.48725
1.45161
1.44392
1.43197
1.34010
1.32613
1.31320
1.27727
1.15471
0.97548
0.95819
0.79048


ppm
140
120
100
80
60
40
20
ppm
139.161
128.271
127.278
109.552
109.258
80.843
77.344
77.227
77.026
76.707
73.210
72.814
69.507
66.821
62.181
56.314
54.307
44.781
44.185
41.587
40.663
40.401
40.224
40.028
38.152
35.059
34.774
34.494
31.748
31.321
31.001
30.631
30.266
30.225
28.730
26.113
24.066
21.028
20.659
17.102
16.495
14.478
BnO
H
OH
O
O
5


BnO
H
OH
O
O
4
2.915P
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
3.5
3.0
2.5
2.0
1.5
1.0
.5