Electronic Supplementary Material (ESI) for Medicinal Chemistry Communications

Supplementary data

Design and syntheses of some iminosugar derivatives as potential immunosuppressants

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Electronic Supplementary Material (ESI) for Medicinal Chemistry Communications

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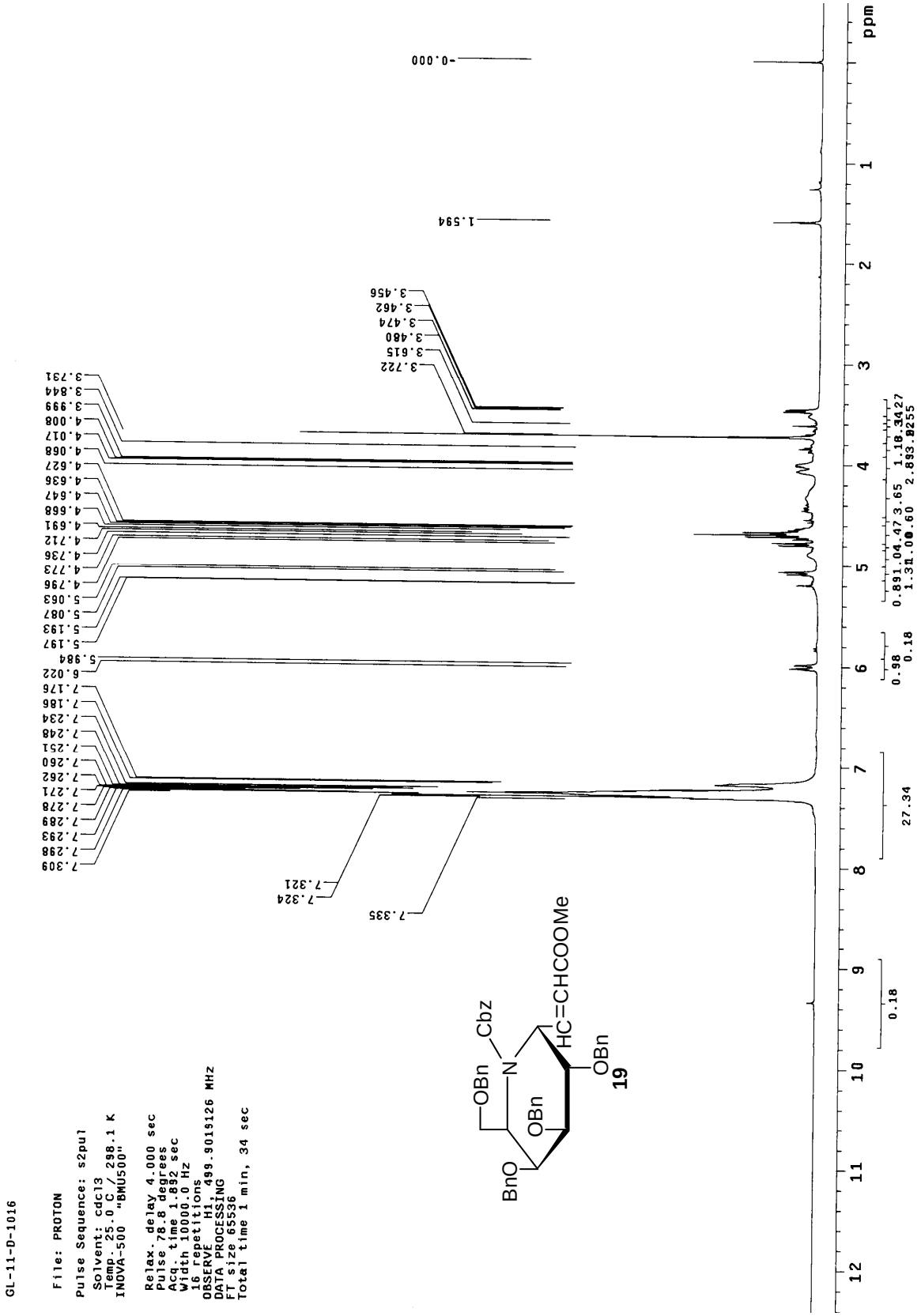
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Pulse 78.8 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
16 repetitions
OBSERVE H1, 499.9019126 MHz
DATA PROCESSING
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Total time 1 min, 34 sec
ppm
19
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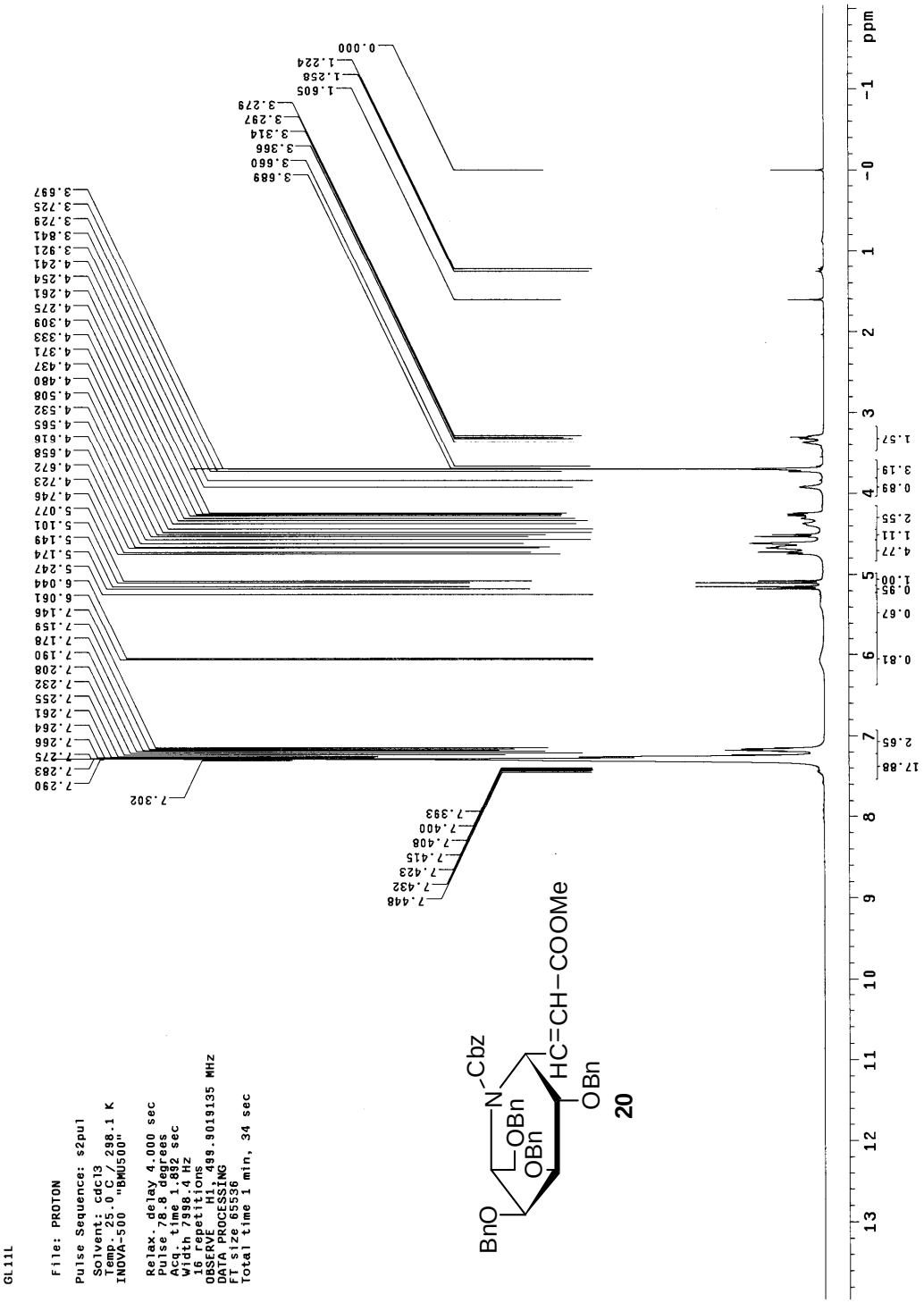
Cbz
N
HC=CHCOOMe
OBn
OBn
BnO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Pulse 78.8 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
16 repetitions
OBSERVE H1, 499.9019135 MHz
DATA PROCESSING
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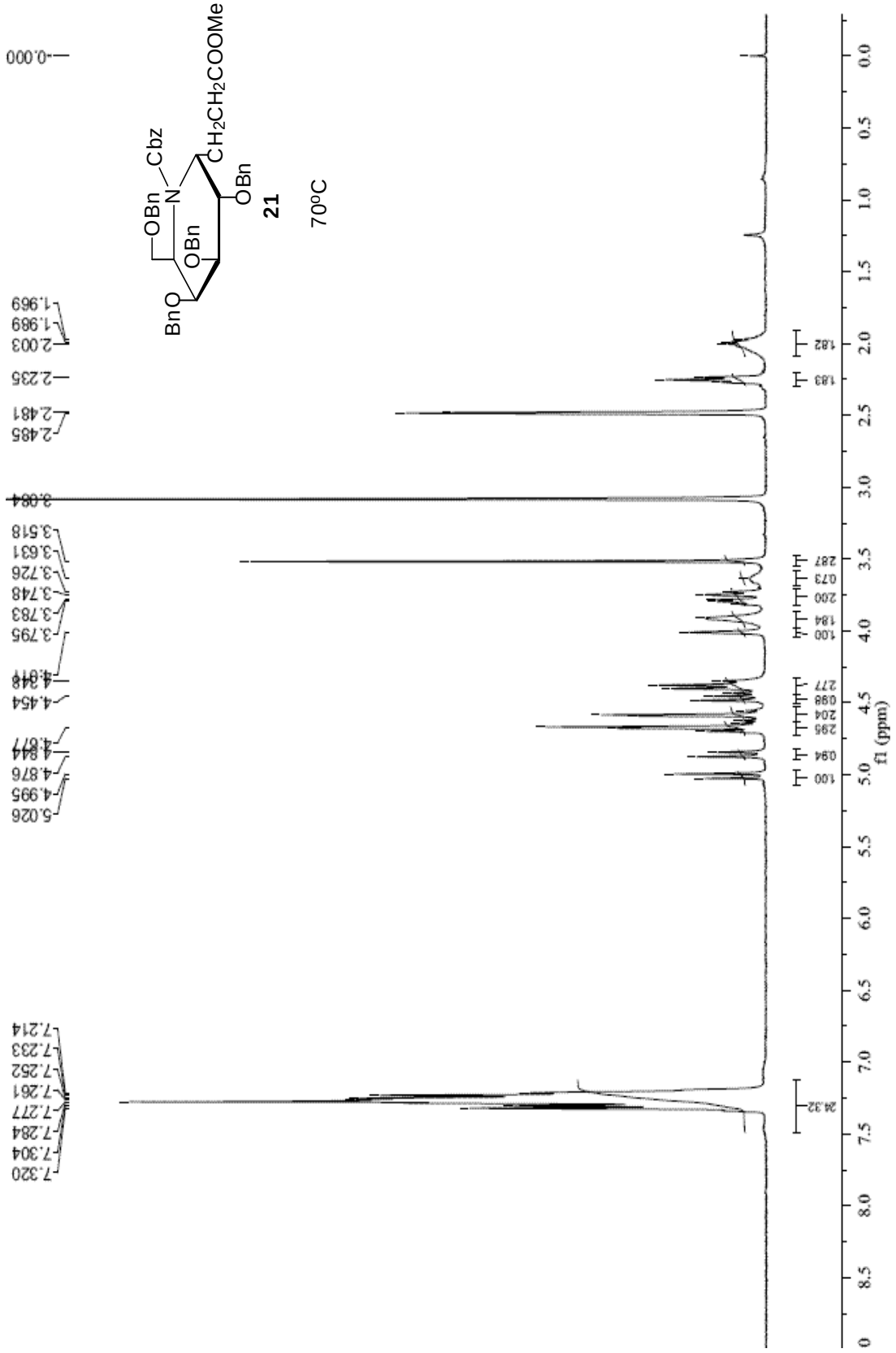
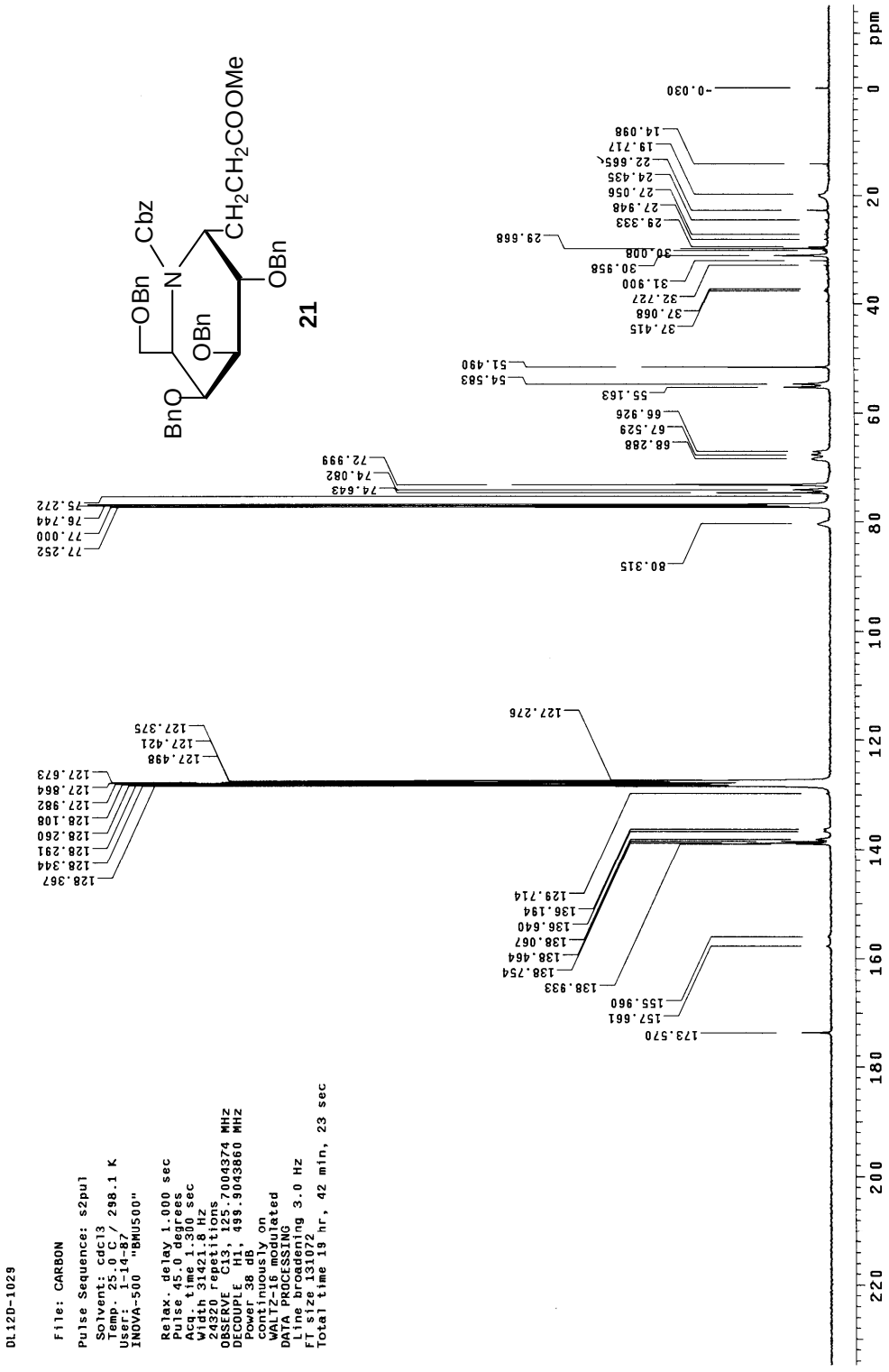
BnO
OBn
OBn
N
Cbz
HC=CH−COOMe
OBn
20
ppm




OBn
Cbz
N
CH2CH2COOMe
OBn
OBn
BnO
21
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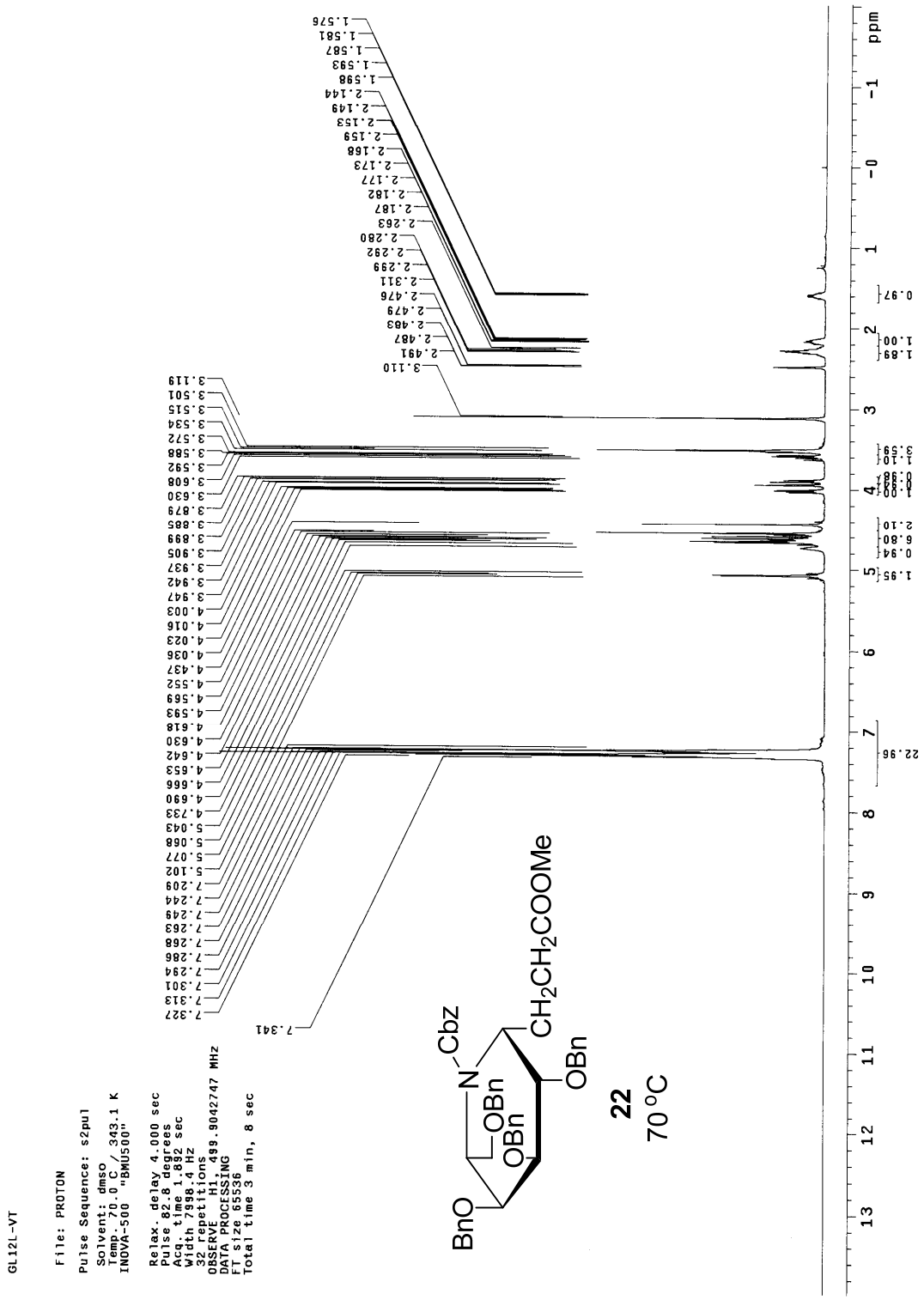
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C/ 298.1 K
User: i-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
24320 repetitions
OBSERVE C13, 125.7004374 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
ppm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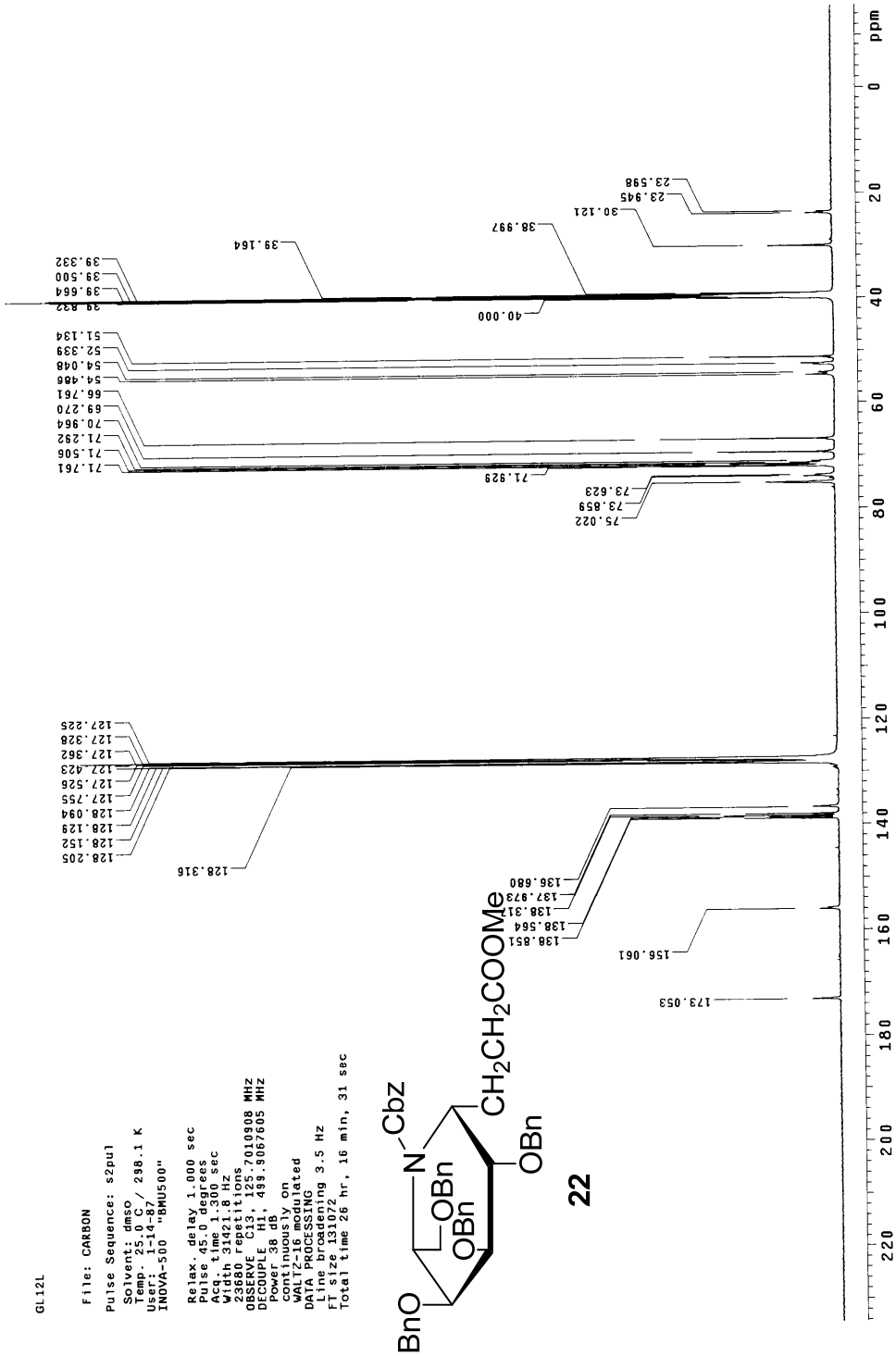
Pulse 82.8 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9042747 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
BnO
OBn
OBn
N
Cbz
CH2CH2COOMe
OBn
22
70 °C
ppm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Pulse Sequence: s2pul
Solvent: dmso
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Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
23680 repetitions
OBSERVE C13, 125.7010908 MHz
DECOUPLE H1, 499.9067605 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
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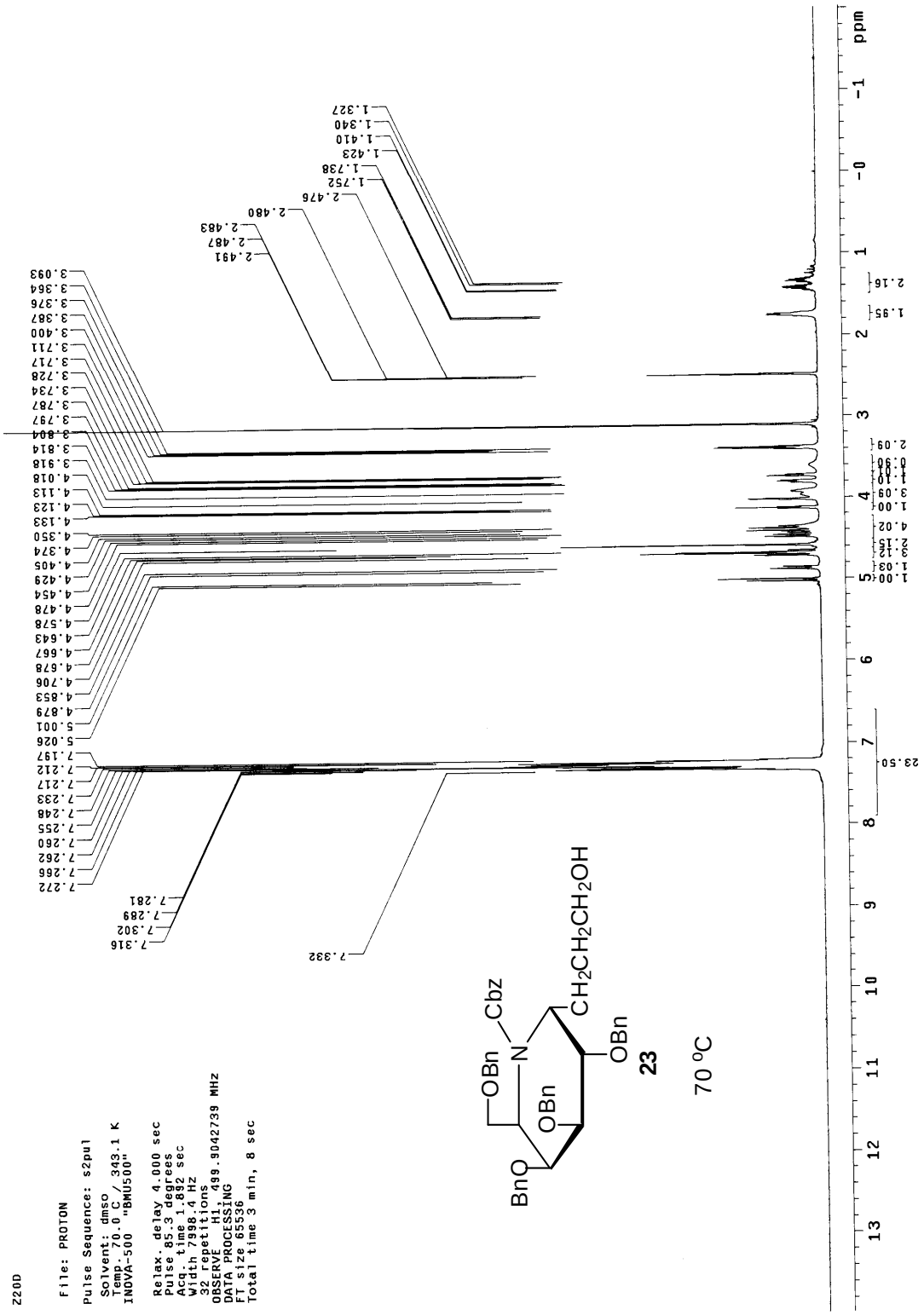
BnO
OBn
OBn
N
Cbz
CH2CH2COOMe
OBn
22
ppm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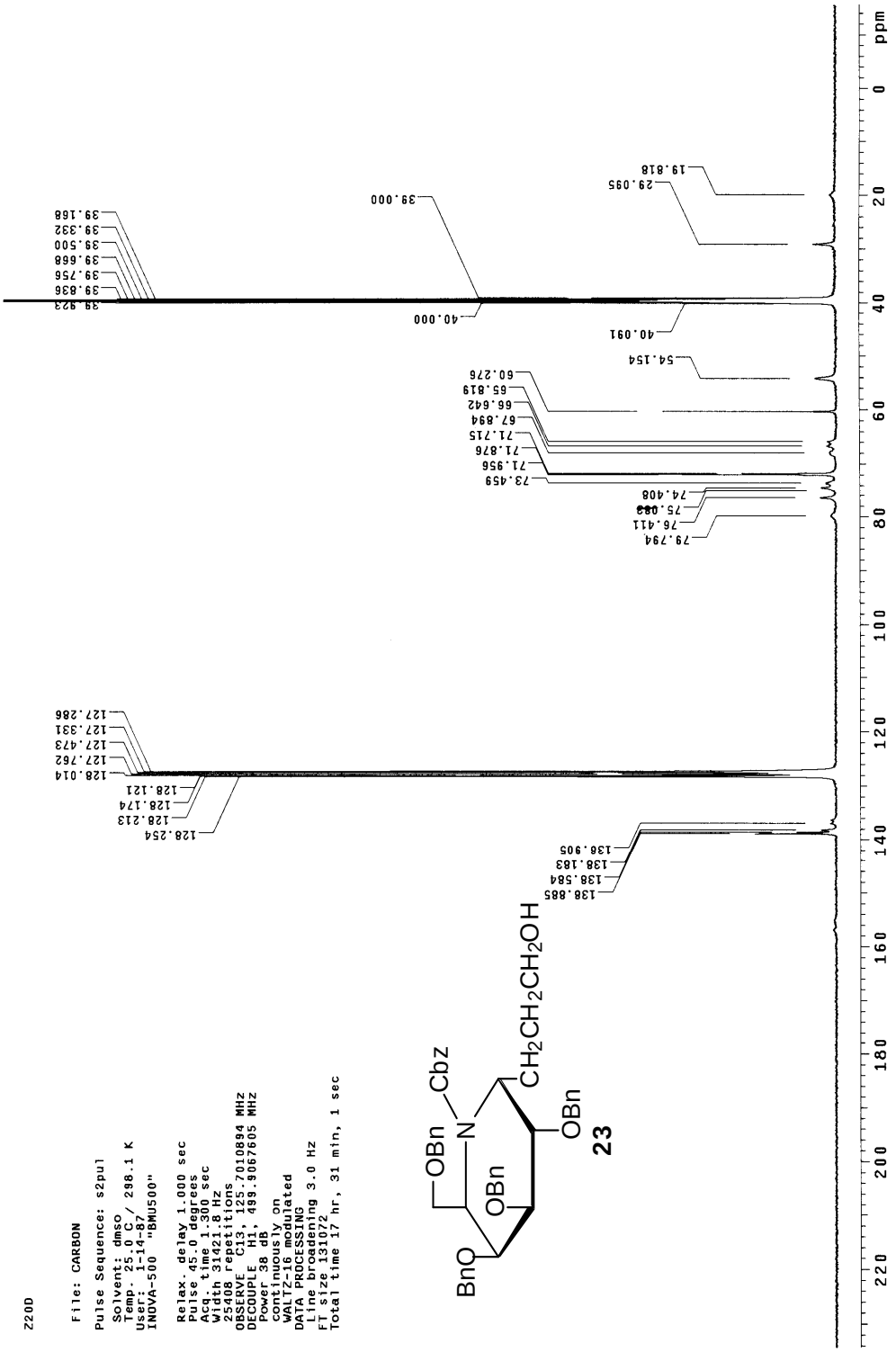
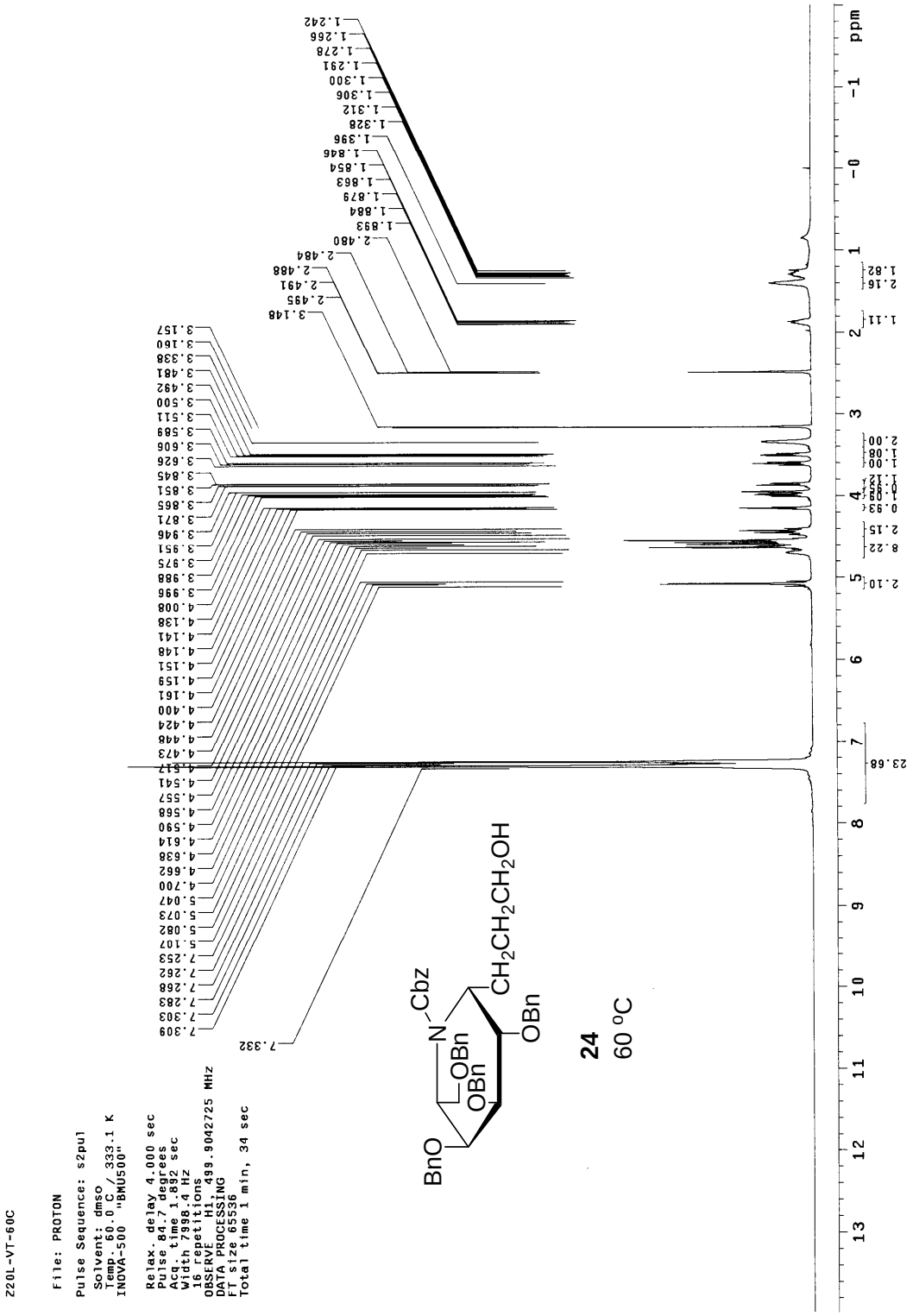
Pulse Sequence: s2pul
Solvent: dmso
Temp. 70.0 C / 343.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9042739 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
Cbz
N
OBn
OBn
BnO
CH2CH2CH2OH
OBn
23
70 °C
ppm


19.818
29.095
39.000
39.168
39.332
39.500
39.668
39.756
39.836
39.923
40.000
40.091
54.154
60.276
65.819
66.642
67.894
71.715
71.876
71.956
73.459
74.408
75.083
76.411
79.794
127.286
127.331
127.473
127.762
128.014
128.121
128.174
128.213
128.254
138.885
138.584
138.183
138.905
OBn
Cbz
N
OBn
OBn
BnO
CH2CH2CH2OH
23
ppm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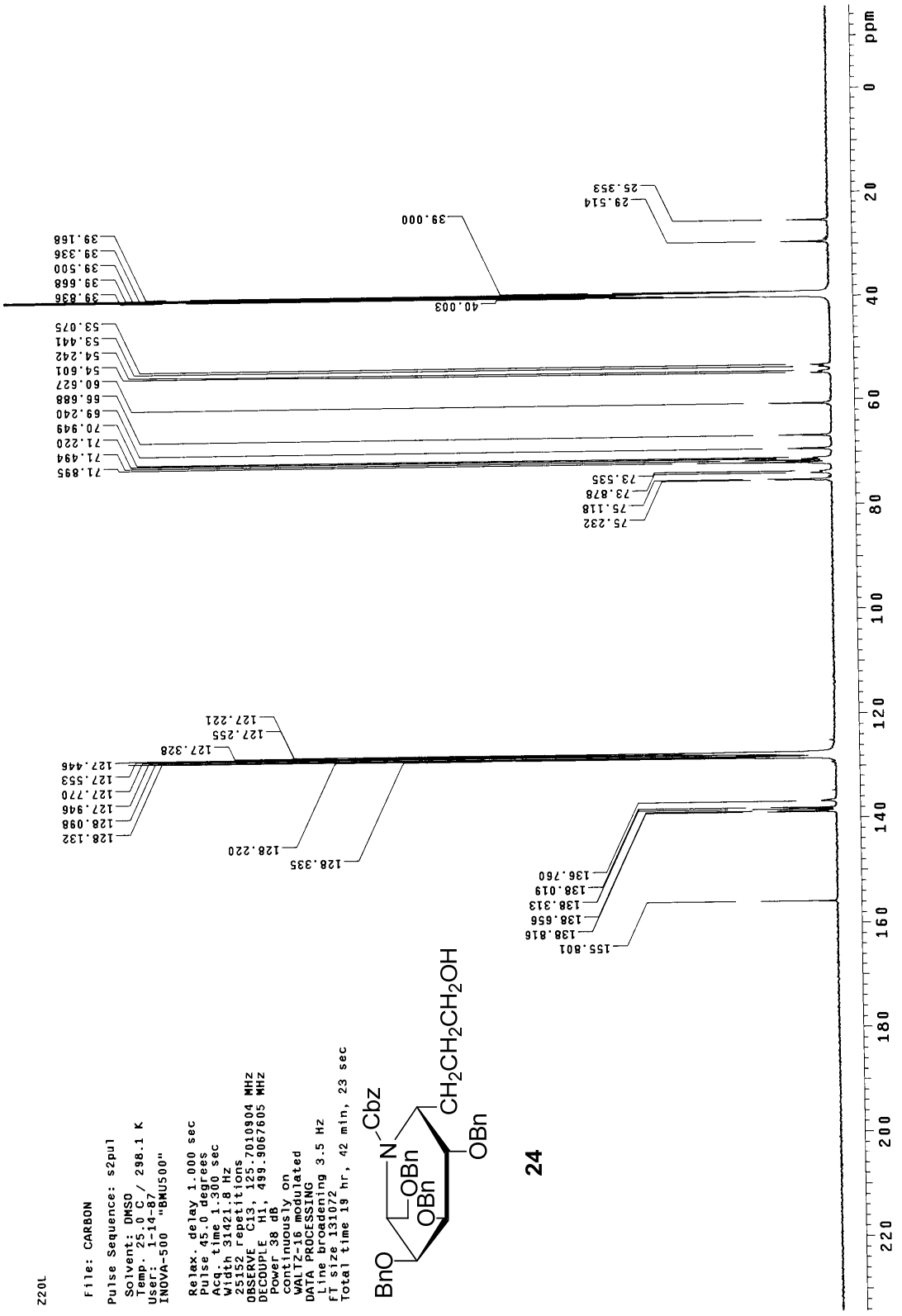
Pulse Sequence: s2pul
Solvent: dmso
Temp. 60.0 C / 333.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 84.7 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
16 repetitions
OBSERVE H1, 499.9042725 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
BnO
OBn
OBn
N
Cbz
CH2CH2CH2OH
OBn
24
60 °C
ppm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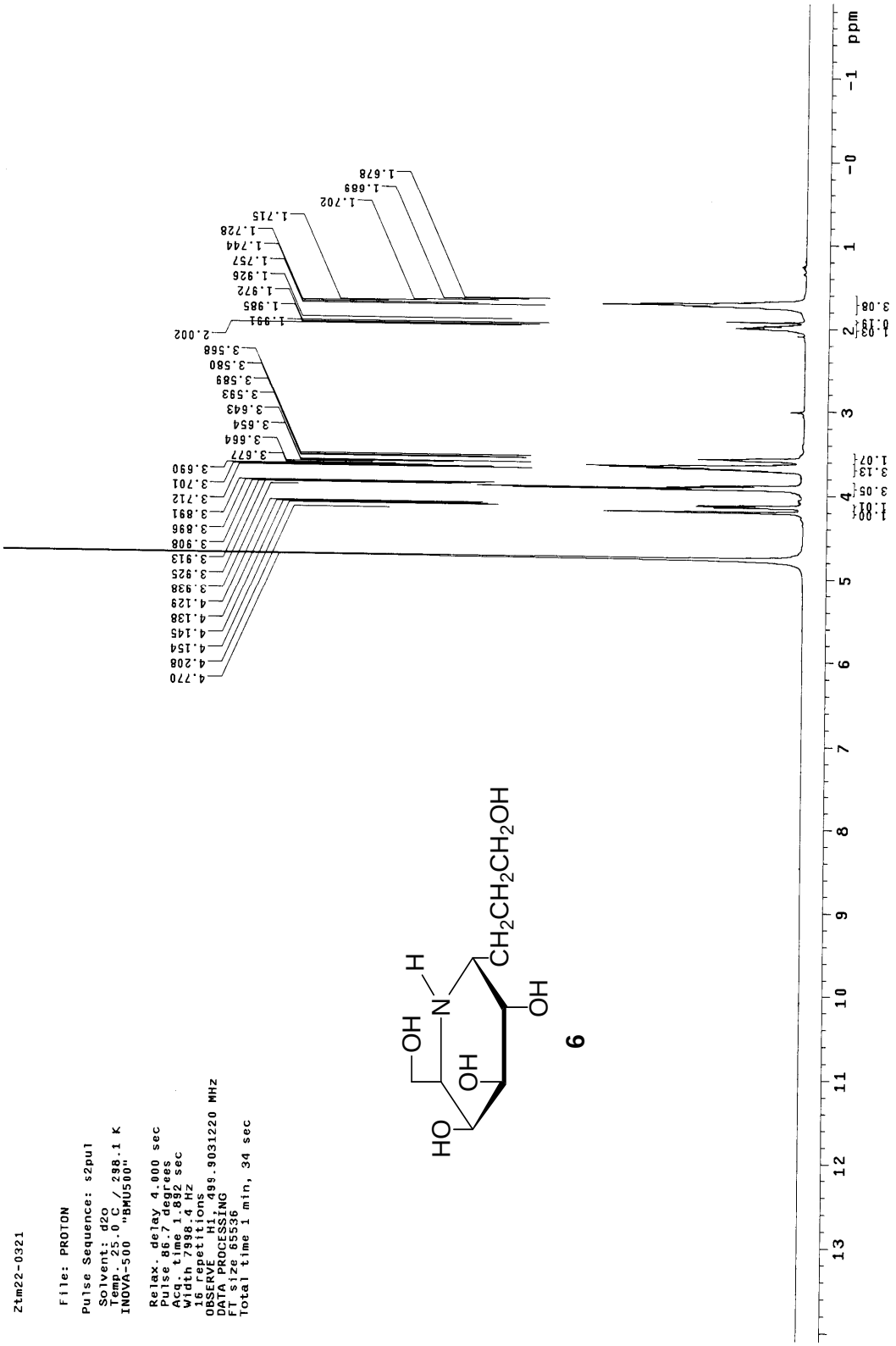
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
25152 repetitions
OBSERVE C13, 125.7010804 MHz
DECOUPLE H1, 499.9067605 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
BnO
OBn
OBn
N
Cbz
CH2CH2CH2OH
OBn
24
ppm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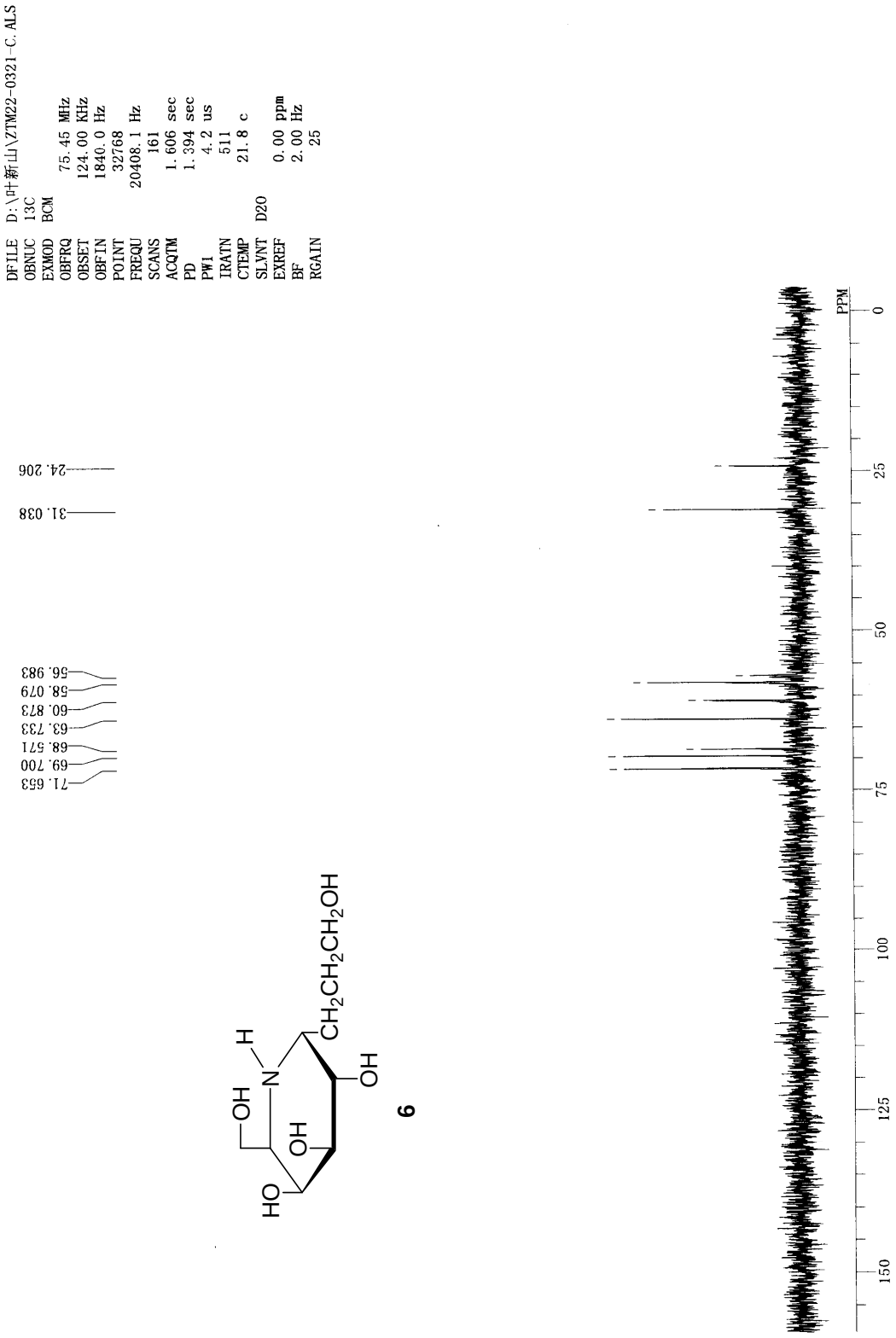
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp: 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 86.7 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
16 repetitions
OBSERVE H1, 499.9031220 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
4.770
4.208
4.154
4.145
4.138
4.129
3.938
3.925
3.913
3.908
3.896
3.891
3.712
3.701
3.690
3.677
3.664
3.654
3.643
3.593
3.589
3.580
3.568
2.002
1.991
1.985
1.972
1.926
1.757
1.744
1.728
1.715
1.702
1.689
1.678
3.08
0.19
1.03
1.00
1.01
3.05
3.13
1.07
ppm
OH
N
H
OH
OH
HO
CH2CH2CH2OH
OH
6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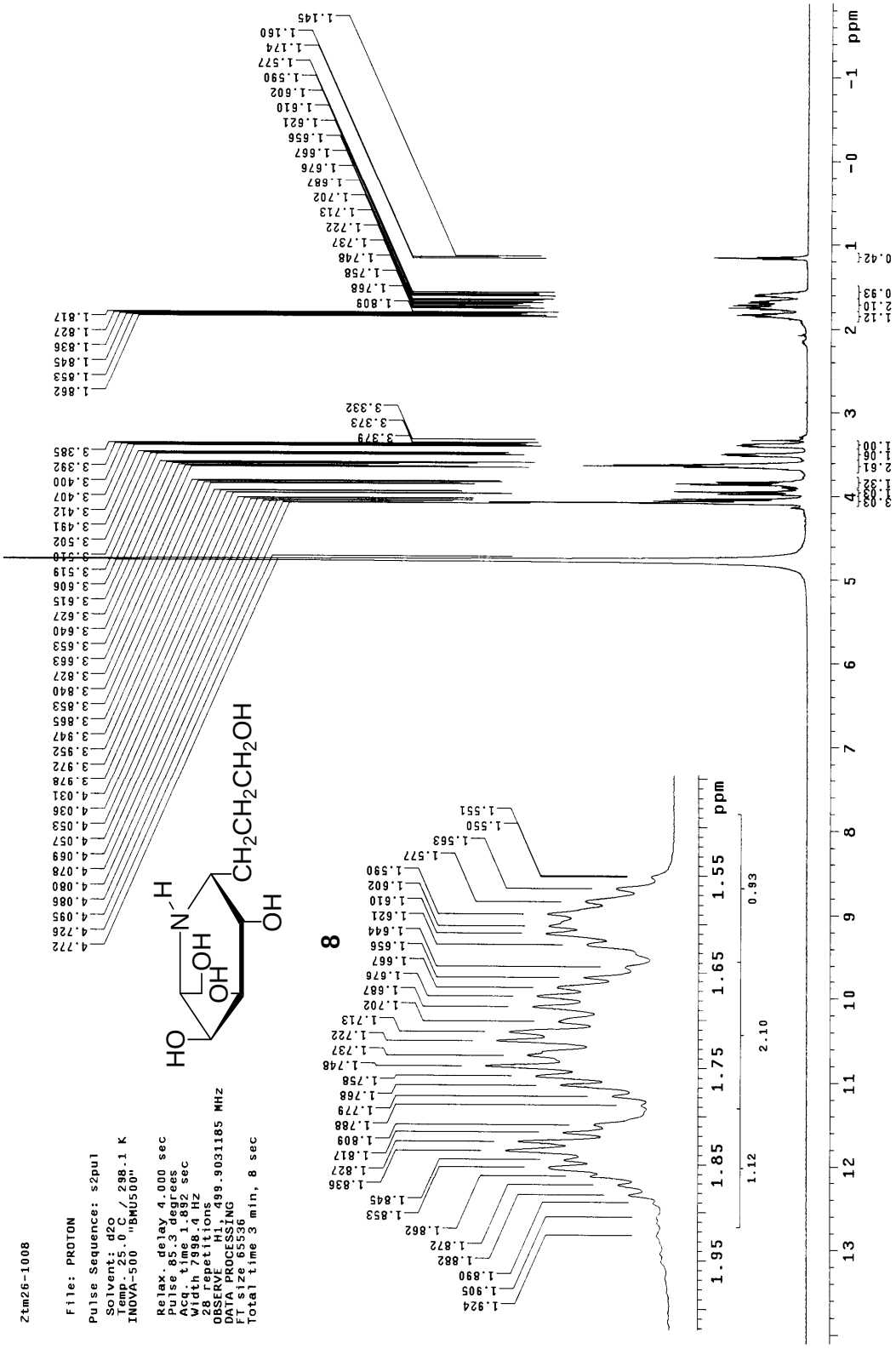
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 161
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 21.8 c
SLVNT D2O
EXREF 0.00 ppm
BF 2.00 Hz
RGAIN 25
24.206
31.038
56.983
58.079
60.873
63.733
68.571
69.700
71.653
PPM
0
25
50
75
100
125
150
OH
HO
OH
N
H
CH2CH2CH2CH2OH
OH
6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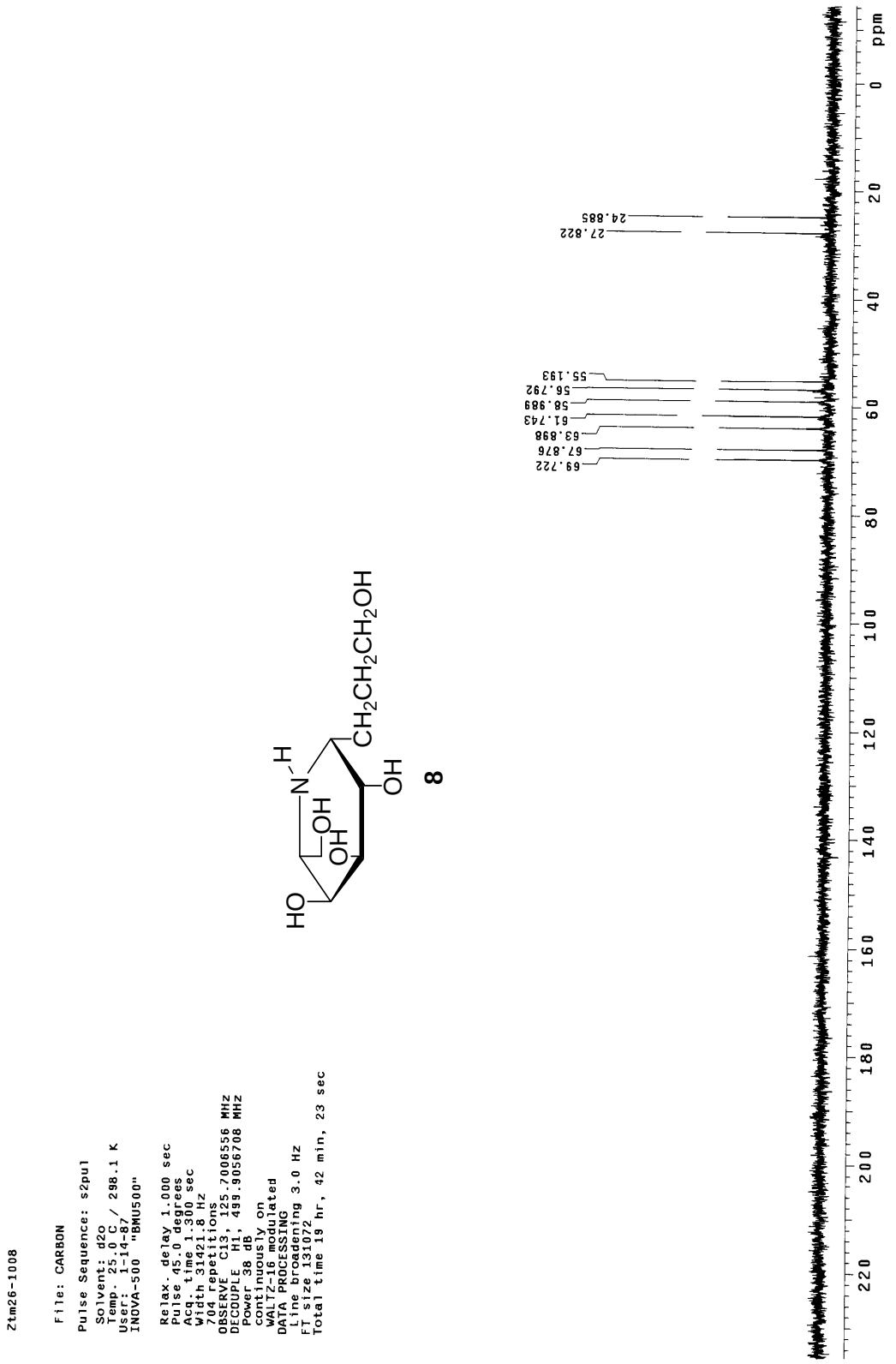
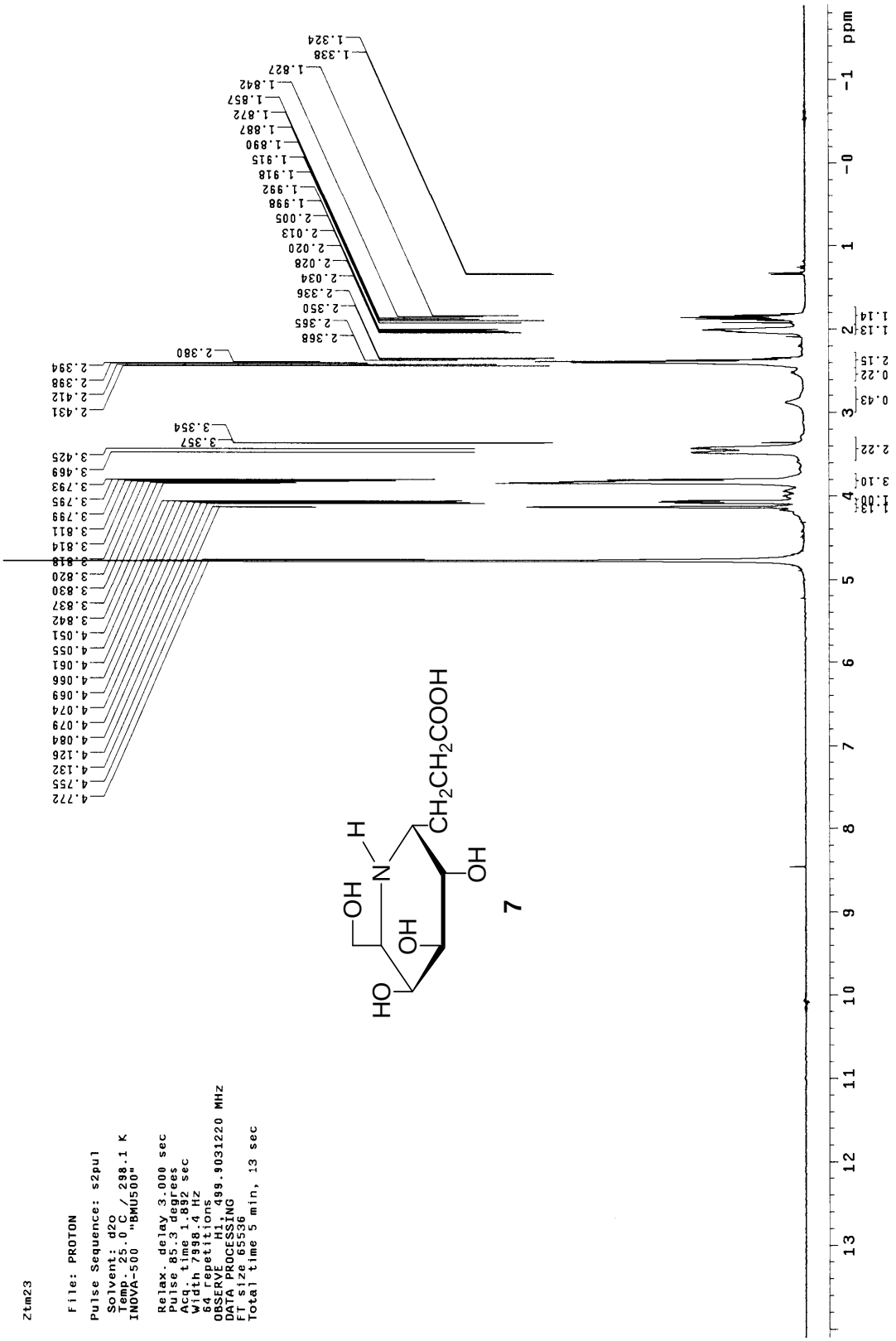
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
28 repetitions
OBSERVE H1, 499.9031185 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
8
ppm
1.12
2.10
0.93


Ztm26-1008
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 31421.8 Hz
704 repetitions
OBSERVE C13, 125.7006556 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
HO
OH
OH
N
H
CH2CH2CH2OH
OH
8
69.722
67.876
63.898
61.743
58.989
56.792
55.193
27.822
24.885
220
200
180
160
140
120
100
80
60
40
20
0
ppm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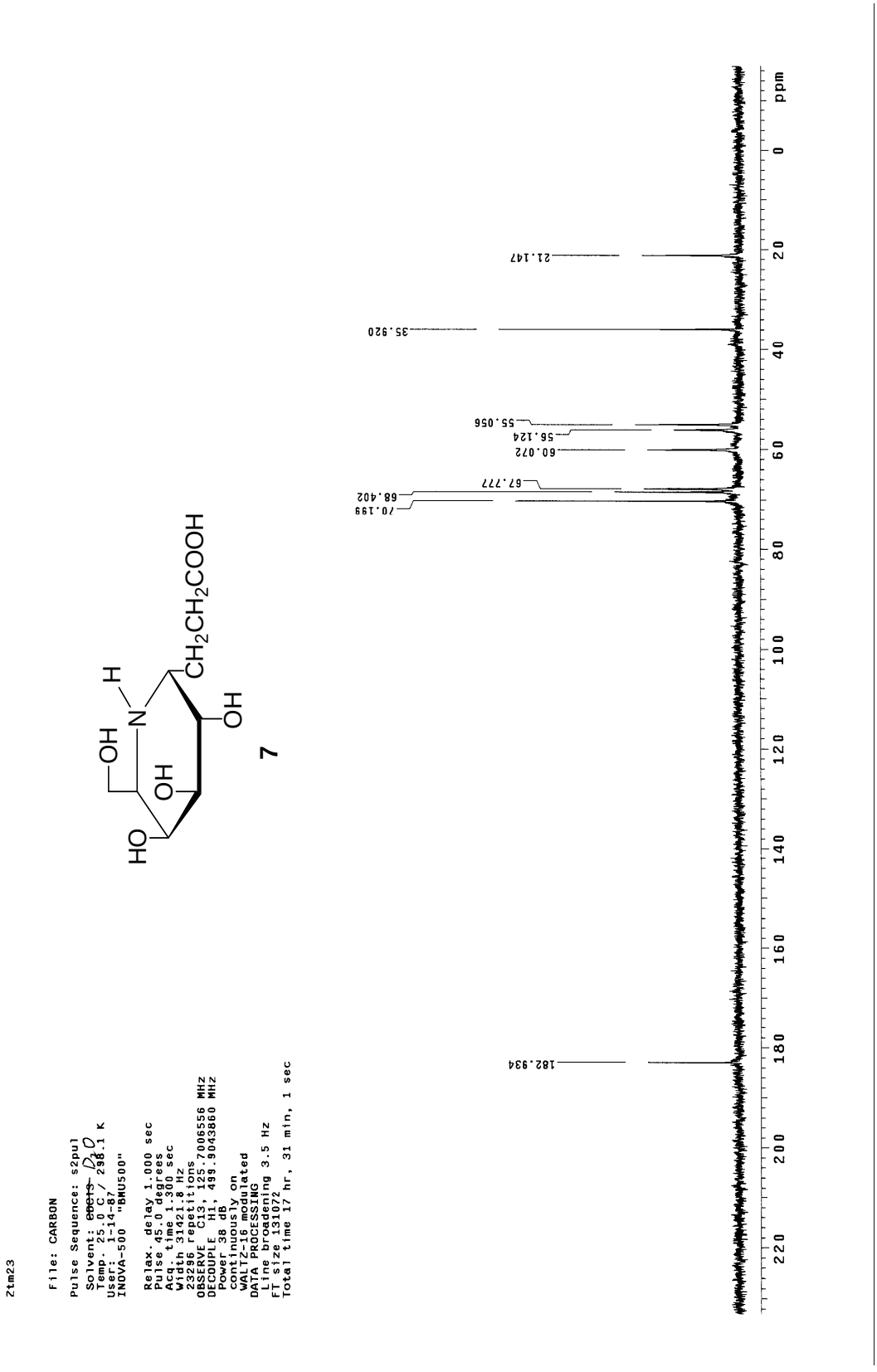
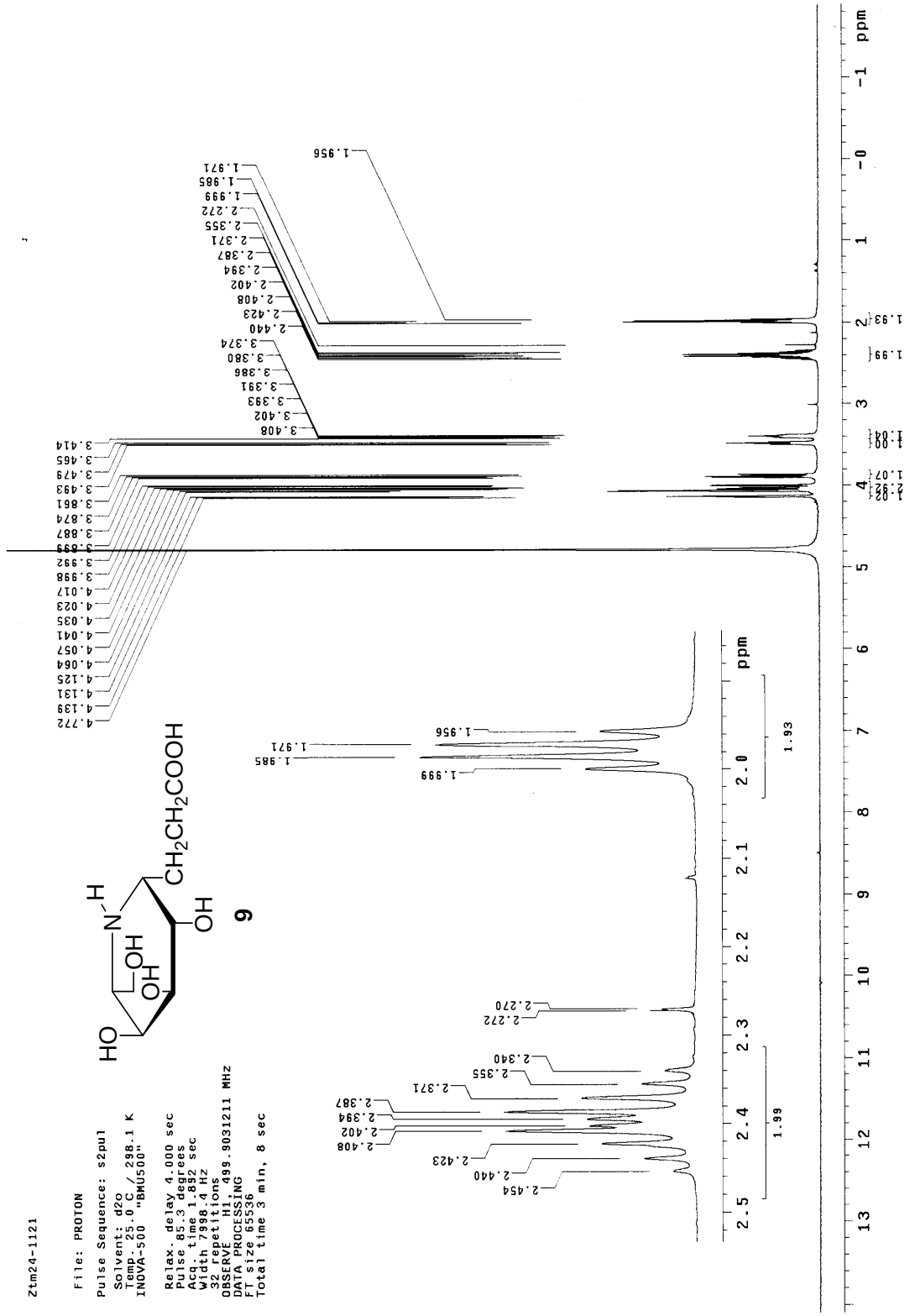
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 3.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 7988.4 HZ
64 repetitions
OBSERVE H1 499.9031220 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 13 sec
ppm
7
OH
HO
OH
N
H
CH2CH2CH2COOH


ztm23
File: CARBON
Pulse Sequence: s2pul
Solvent: ~~pyridine~~ D2O
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
23296 repetitions
OBSERVE C13, 125.7006556 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
Total time 17 hr, 31 min, 1 sec
OH
HO
OH
N
H
CH2CH2COOH
OH
7
182.934
70.199
68.402
67.777
60.072
56.124
55.056
35.920
21.147
220
200
180
160
140
120
100
80
60
40
20
0
ppm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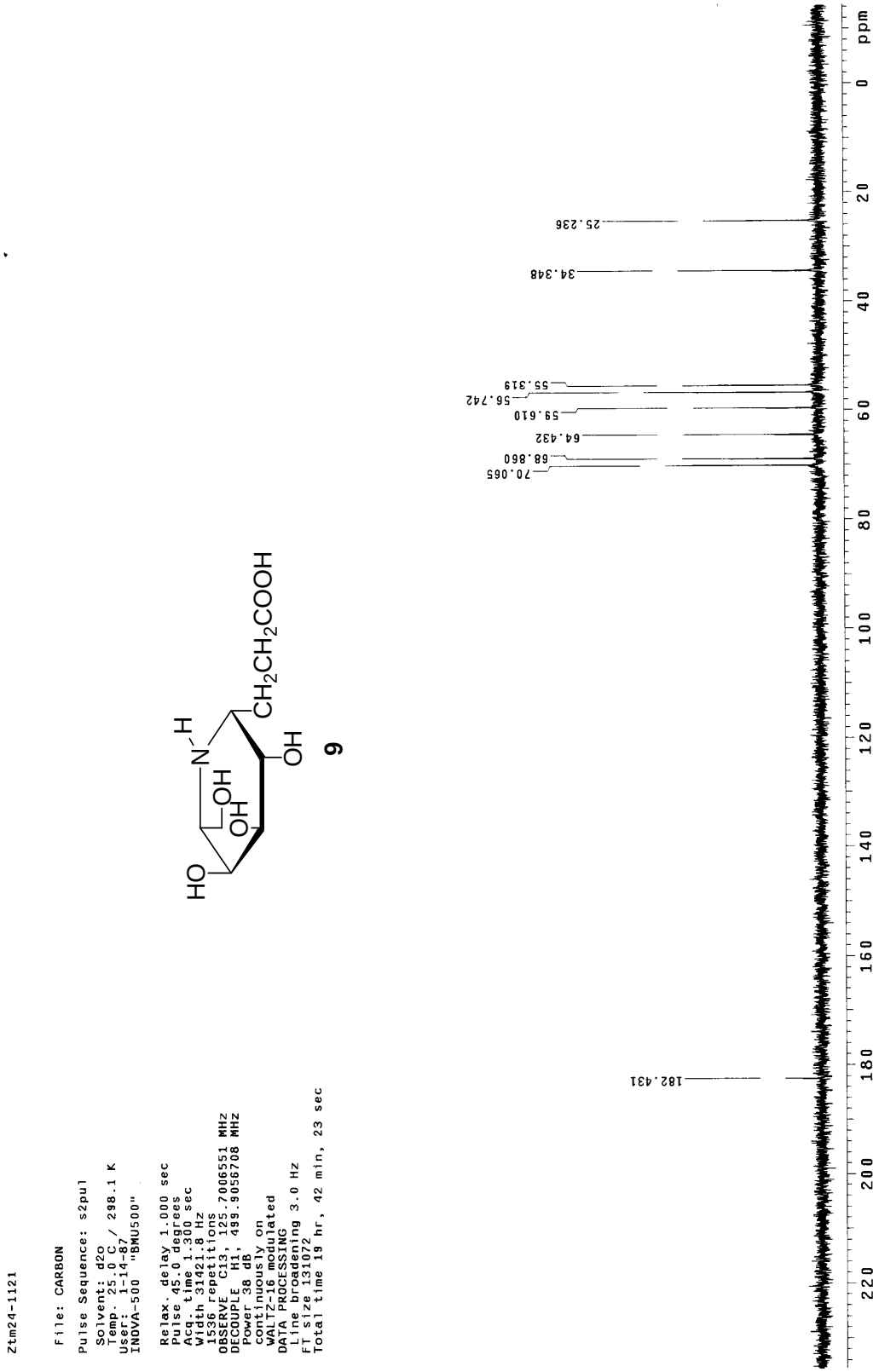
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.882 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9031211 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
9
HO
OH
OH
OH
H
N
CH2CH2COOH
ppm
1.93
1.99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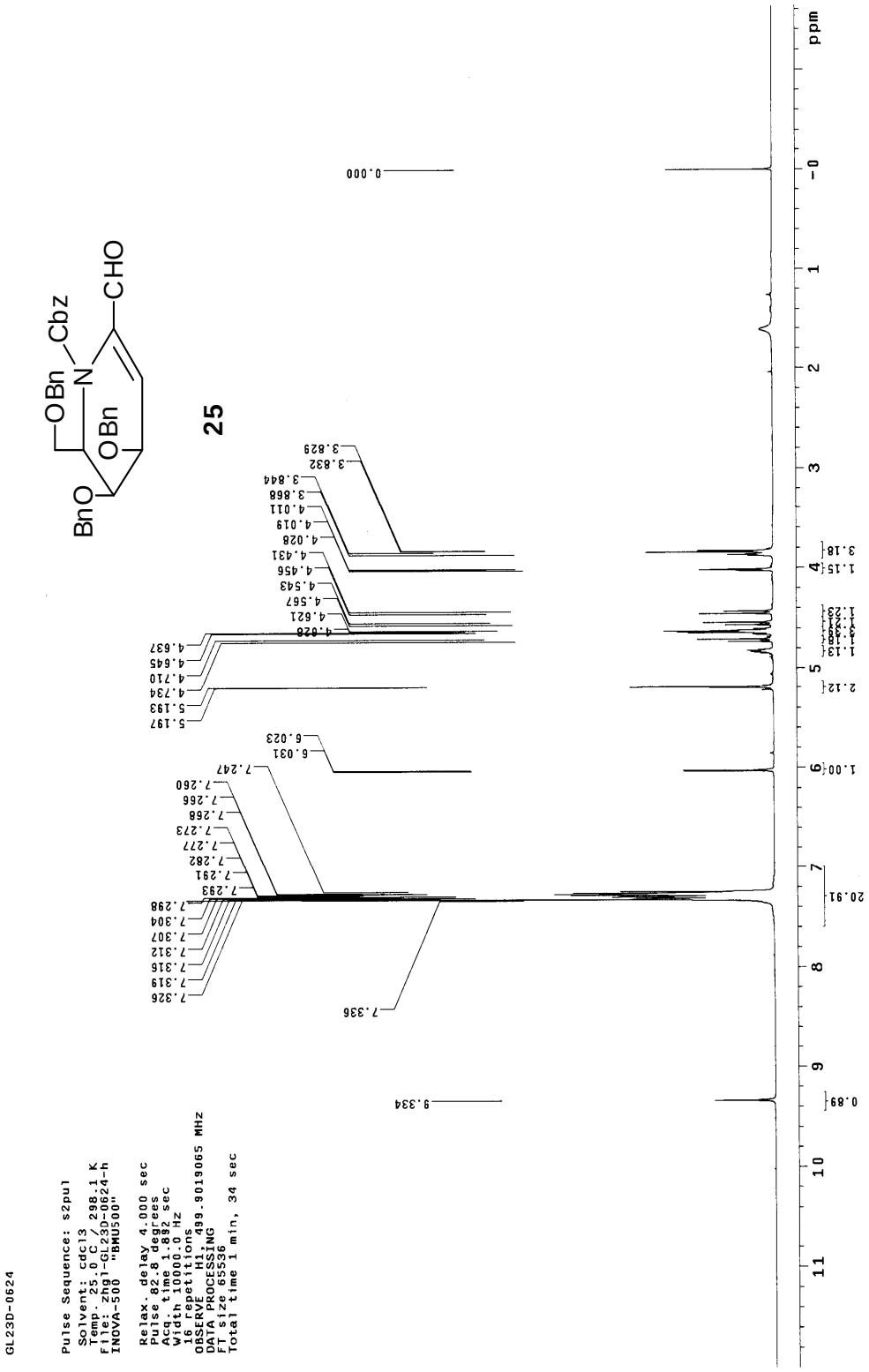
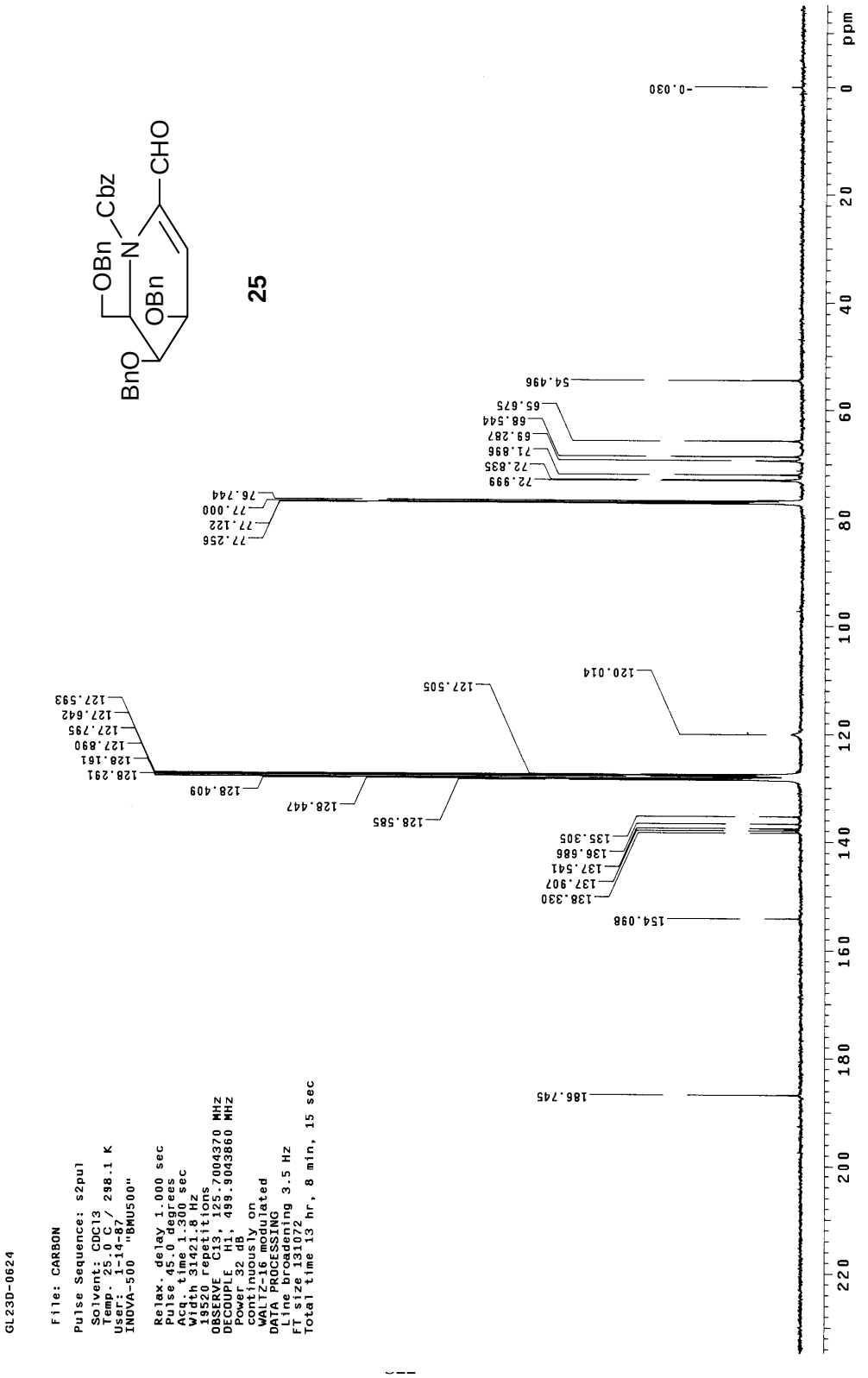
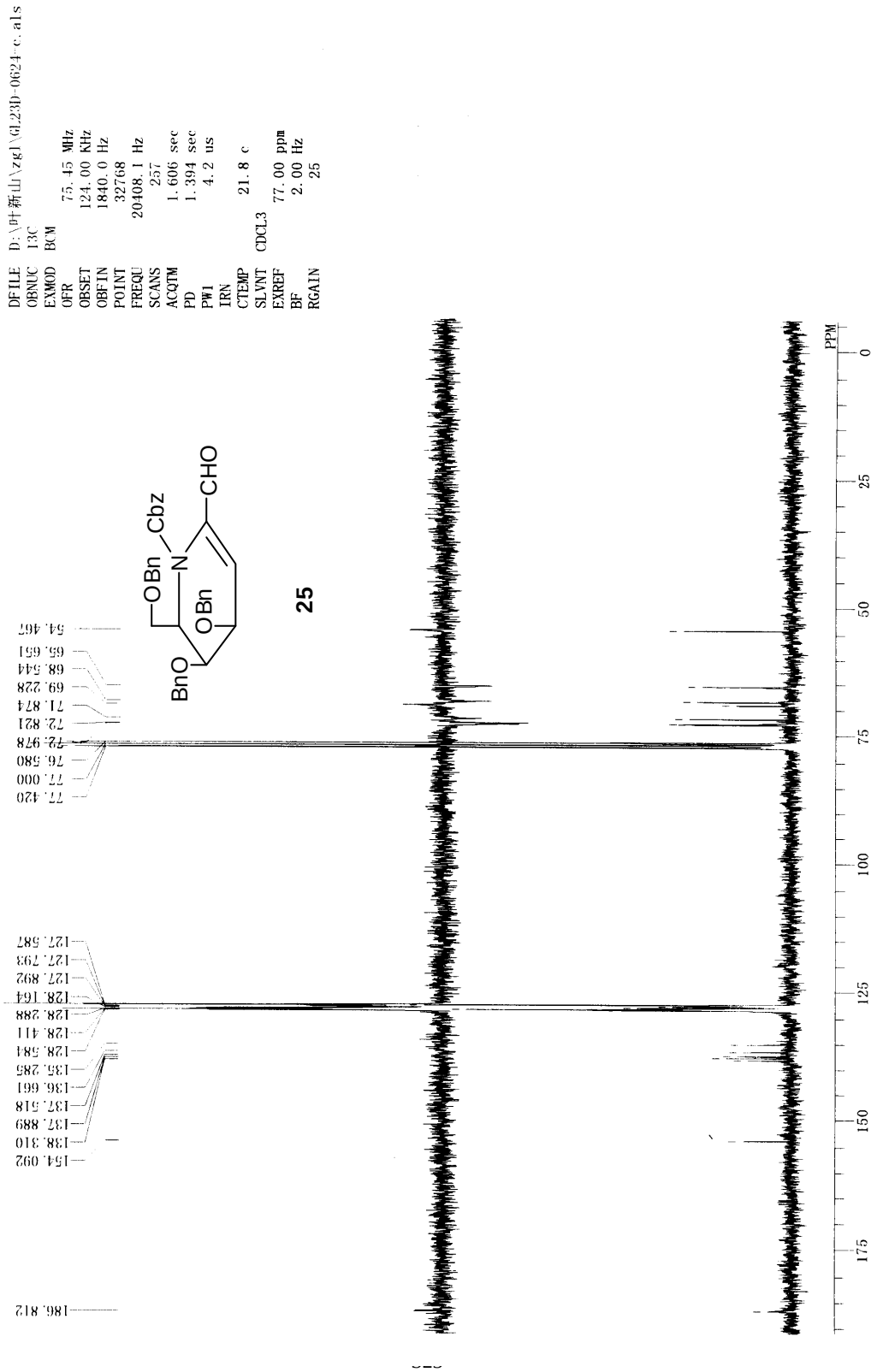
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1536 repetitions
OBSERVE C13, 125.7006551 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
HO
OH
OH
N
H
CH2CH2COOH
OH
6
182.431
70.065
68.860
64.432
59.610
56.742
55.319
34.348
25.236
220
200
180
160
140
120
100
80
60
40
20
0
ppm


25
OBn
Cbz
N
CHO
OBn
BnO
0.000
3.829
3.832
3.844
3.868
4.011
4.019
4.028
4.431
4.456
4.543
4.567
4.621
4.628
4.637
4.645
4.710
4.734
5.193
5.197
6.023
6.031
7.247
7.260
7.266
7.268
7.273
7.277
7.282
7.291
7.293
7.298
7.304
7.307
7.312
7.316
7.319
7.326
7.336
9.334
ppm
3.18
1.15
1.23
1.13
2.12
1.00
20.91
0.89
GL23D-0624
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zhgl-GL23D-0624-h
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 82.8 degrees
Acq. time 1.892 sec
Width 10010.0 Hz
1 repetitions
OBSERVE H1, 499.9019065 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec


25
BnO
OBn
OBn
N
Cbz
CHO
GL23D-0624
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
19520 repetitions
OBSERVE C13, 125.7004370 MHz
DECOUPLE H1, 499.9043860 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
Total time 13 hr, 8 min, 15 sec
-0.030
54.496
65.675
68.544
69.287
71.896
72.835
72.999
76.744
77.000
77.122
77.256
120.014
127.505
127.593
127.642
127.795
127.890
128.161
128.291
128.409
128.447
128.585
135.305
136.686
137.541
137.907
138.330
154.098
186.745
ppm
0
20
40
60
80
100
120
140
160
180
200
220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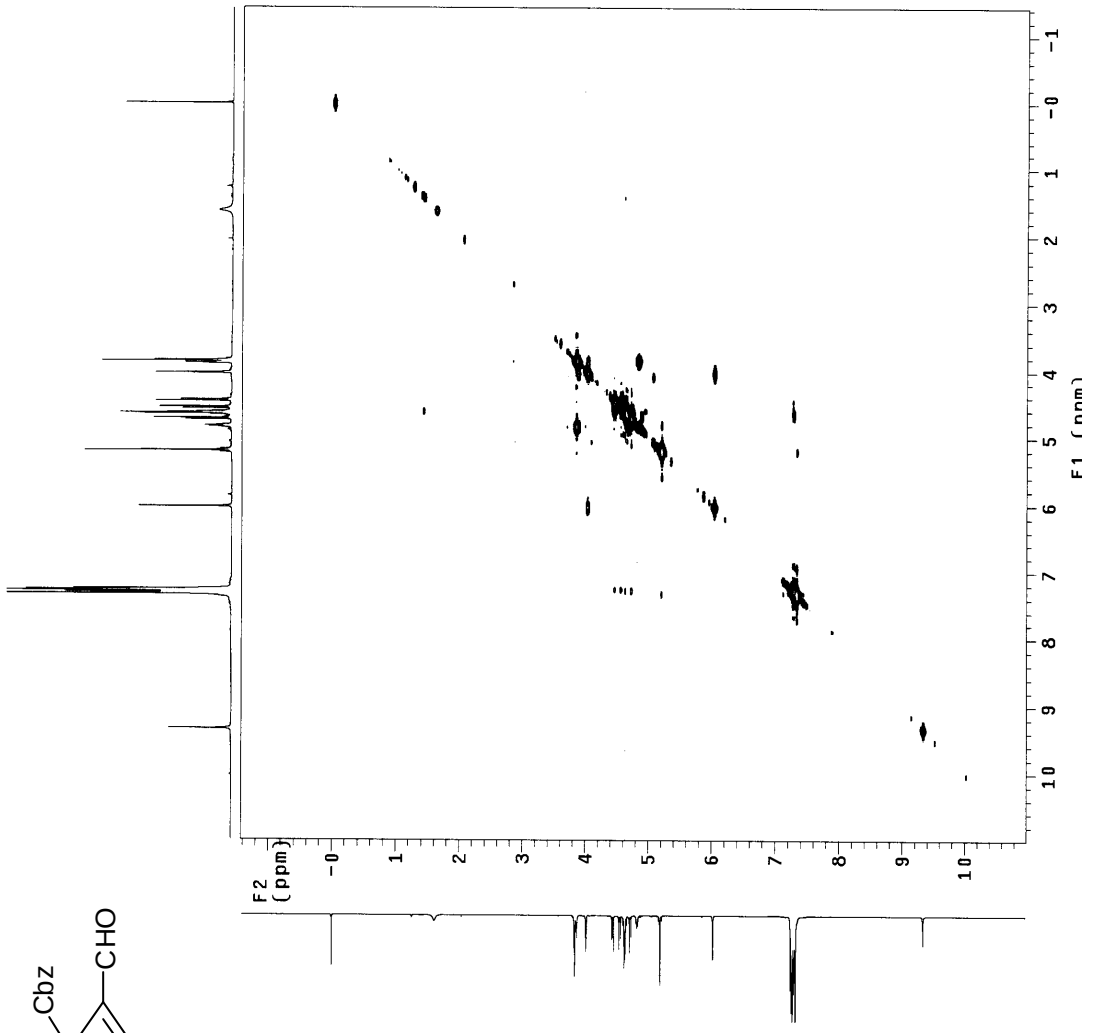
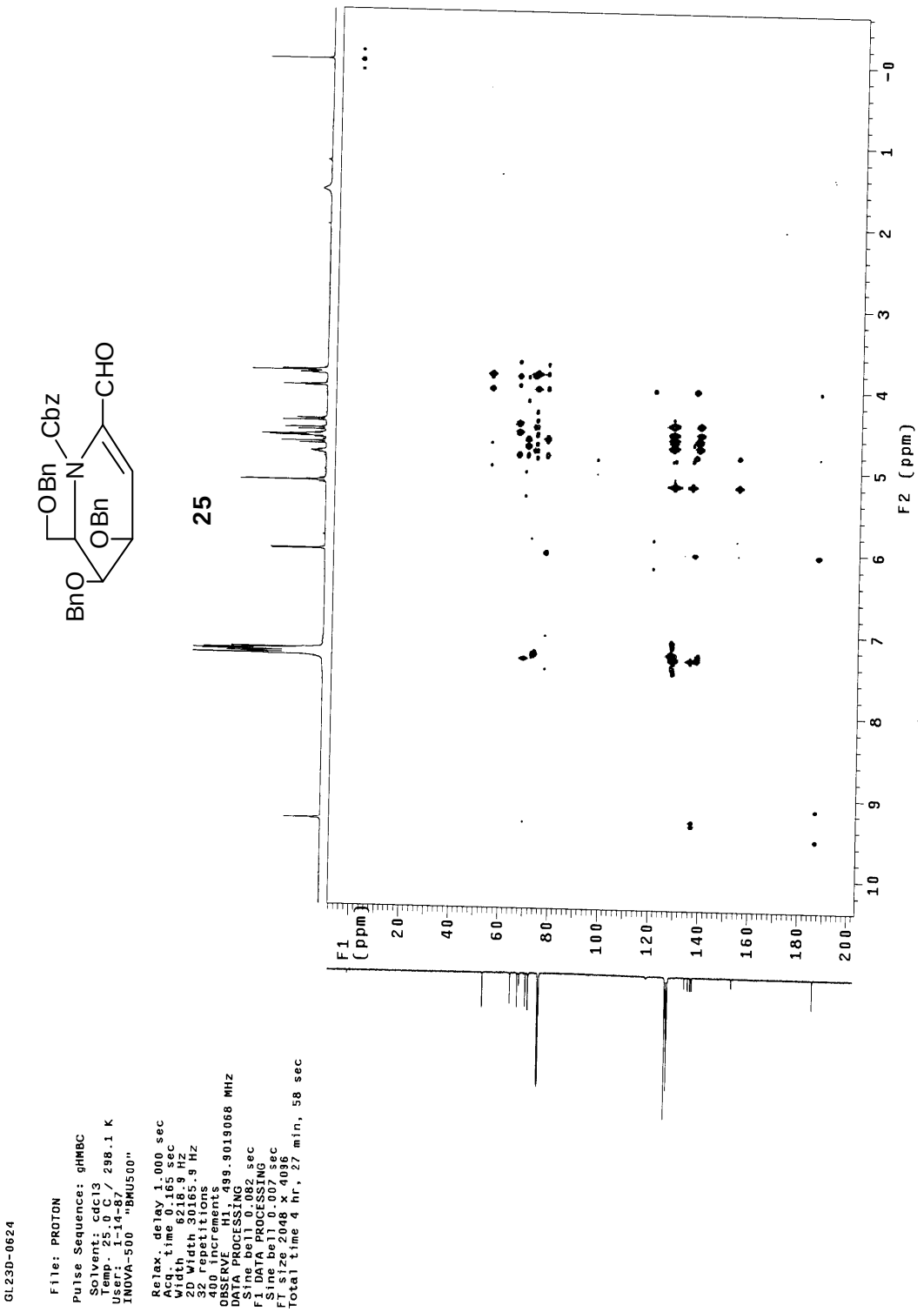
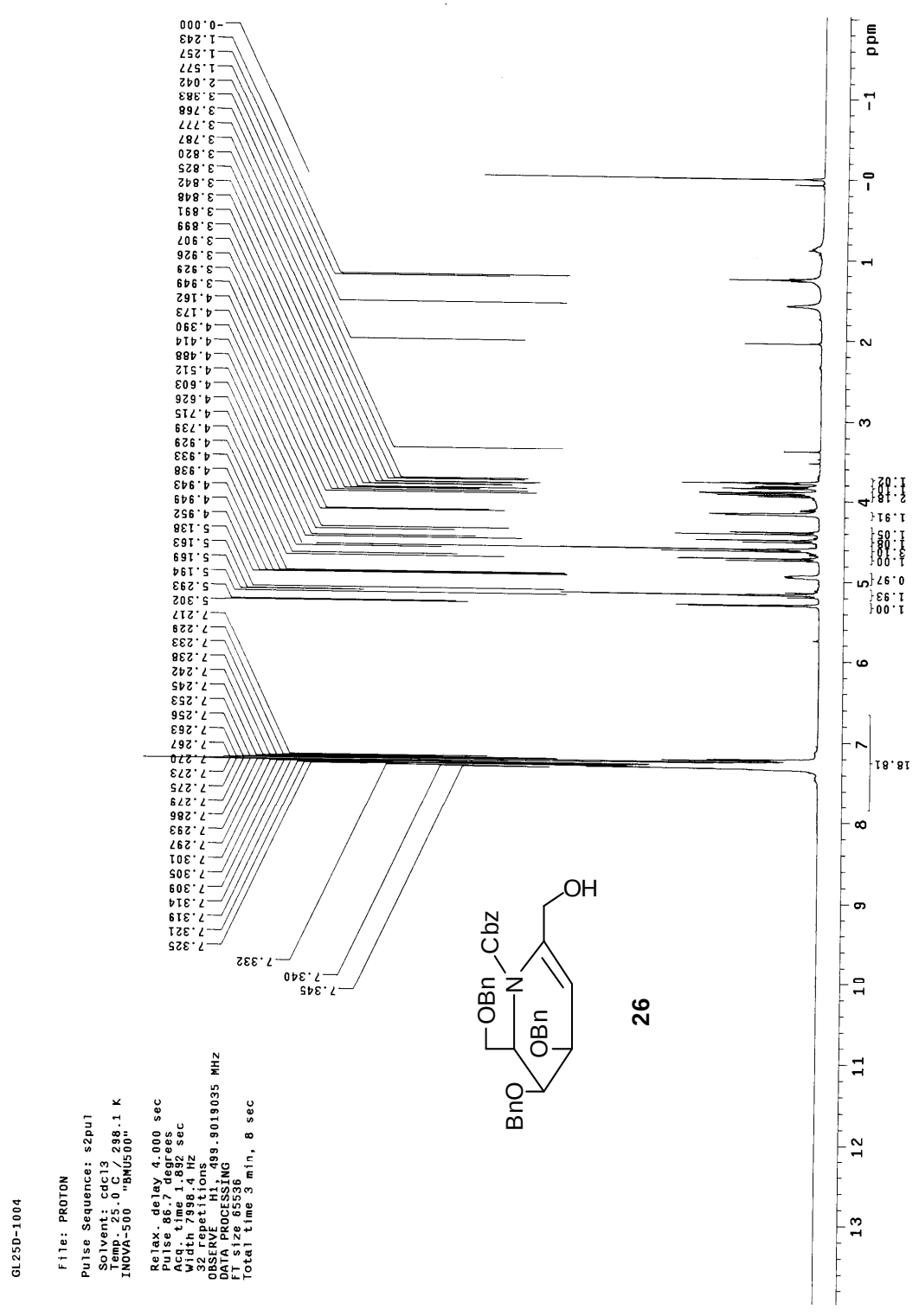
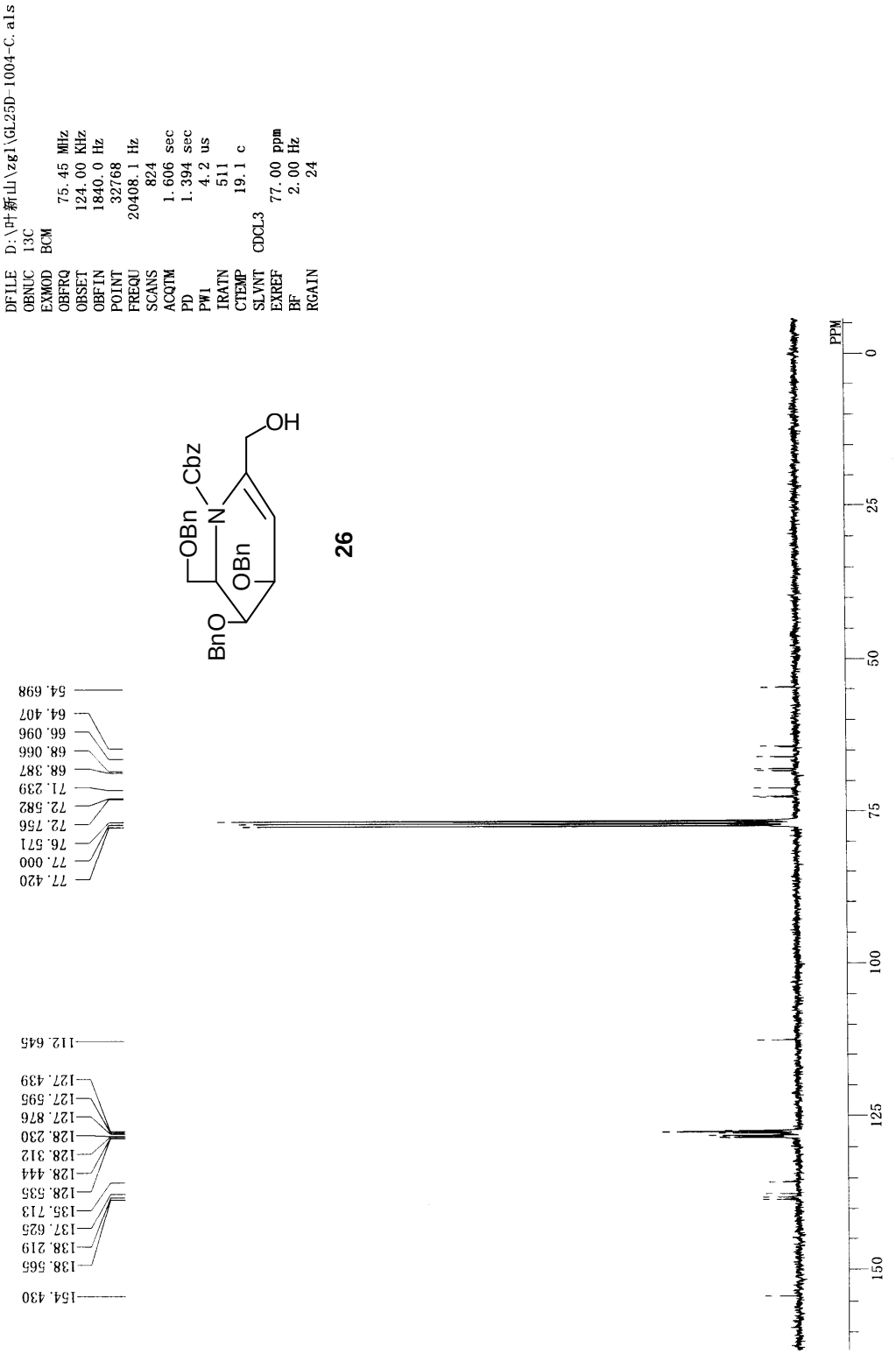
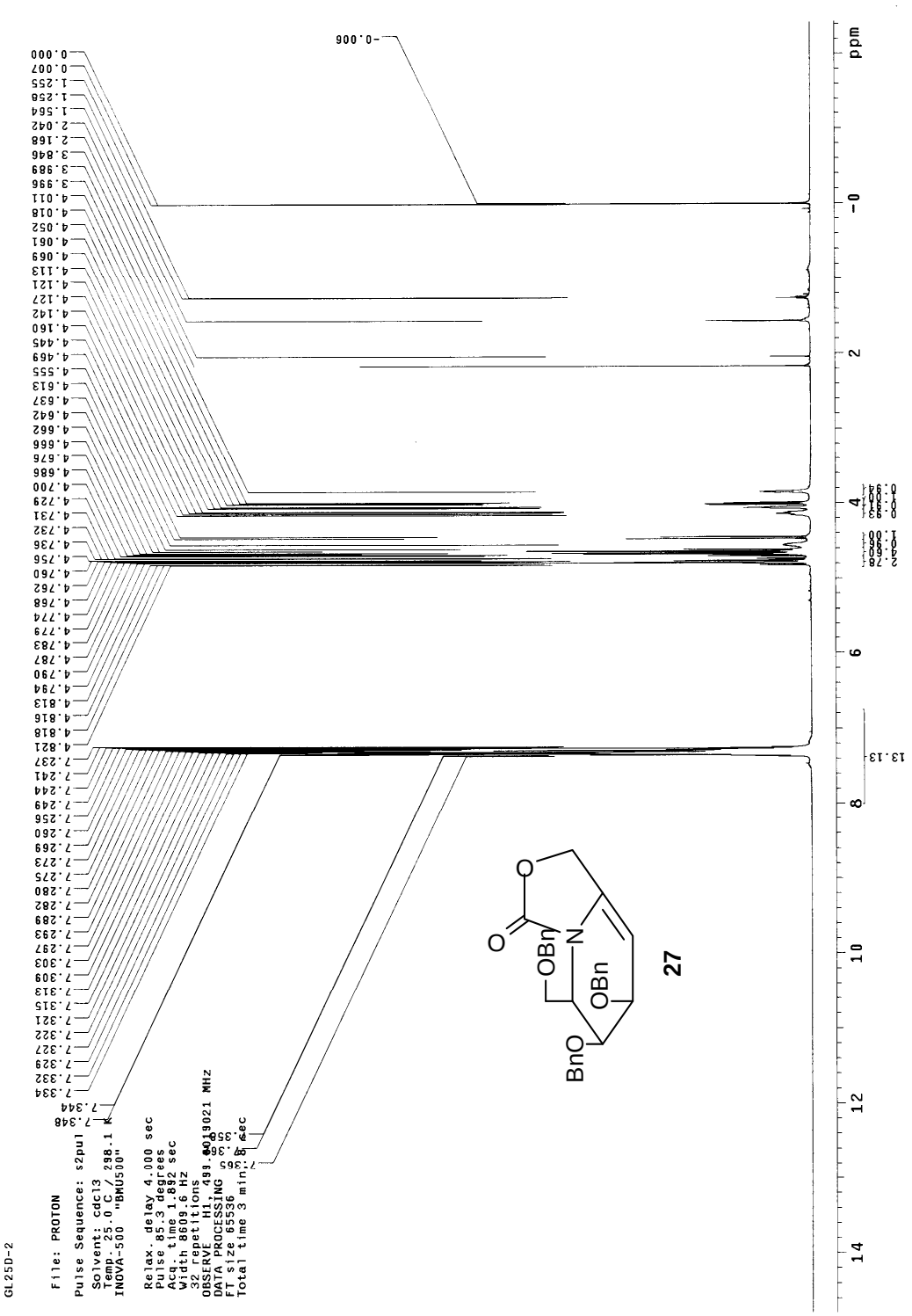
OBNUC 13C
EXMOD BCM
OFR 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 257
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRN
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
OBn
Cbz
N
CHO
BnO
OBn
25
54.467
65.651
68.544
69.228
71.874
72.821
72.978
76.580
77.000
77.420
127.587
127.793
127.892
128.164
128.288
128.411
128.584
135.285
136.661
137.518
137.889
138.310
154.092
186.812
PPM
0
25
50
75
100
125
150
175

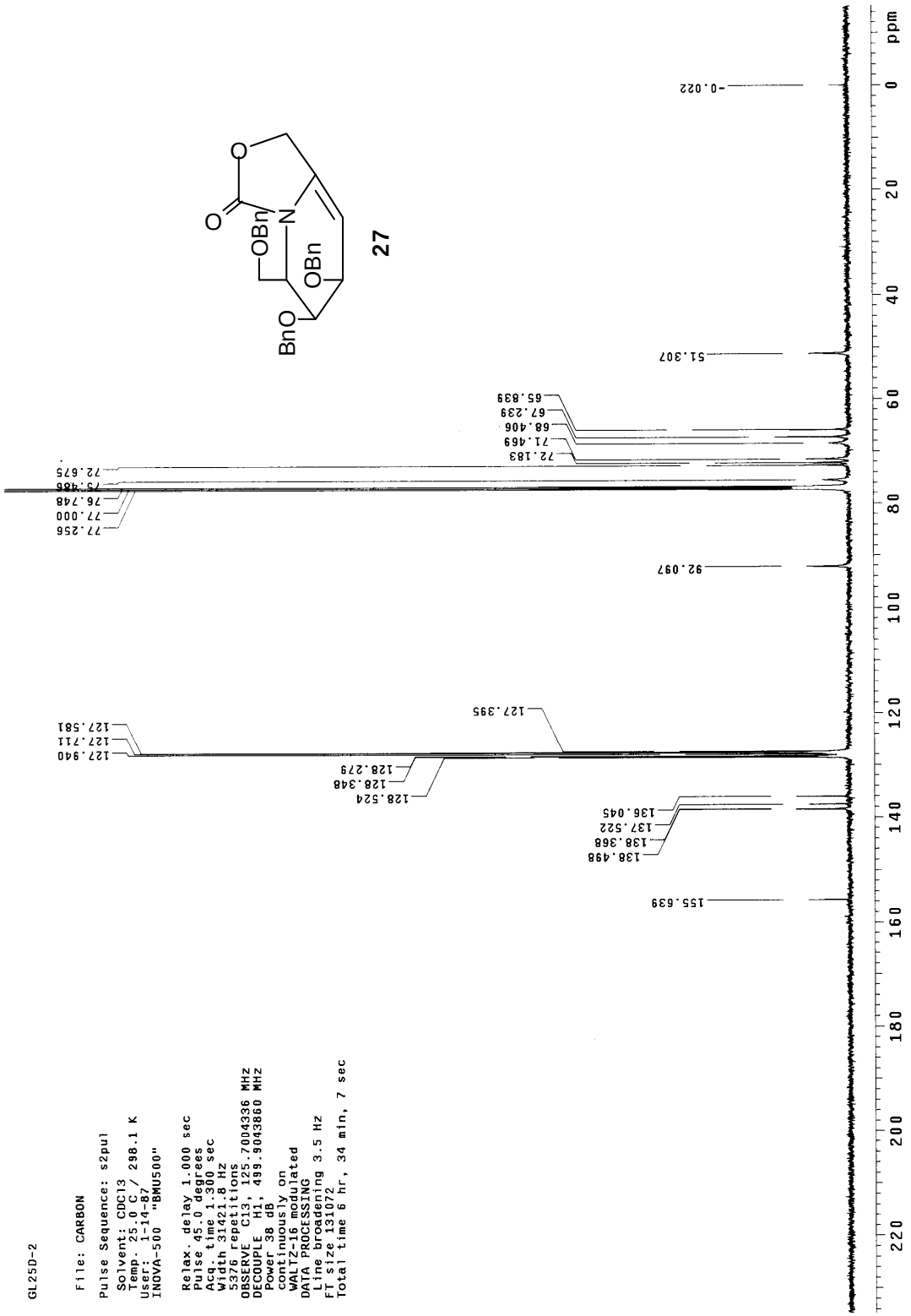

GL23D-0624
Pulse Sequence: gCOSY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zngl-GL23D-0624-cosy
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.165 sec
Width 6205.9 Hz
2D Width 6205.9 Hz
16 repetitions
256 increments
OBSERVE H1, 499.9019065 MHz
DATA PROCESSING
Sq. sine bell 0.083 sec
FT DATA PROCESSING
Sq. sine bell 0.021 sec
FT size 4096 x 4096
Total time 1 hr, 22 min, 8 sec
BnO
OBn
N
Cbz
OBn
CHO
25
F2 (ppm)
F1 (ppm)

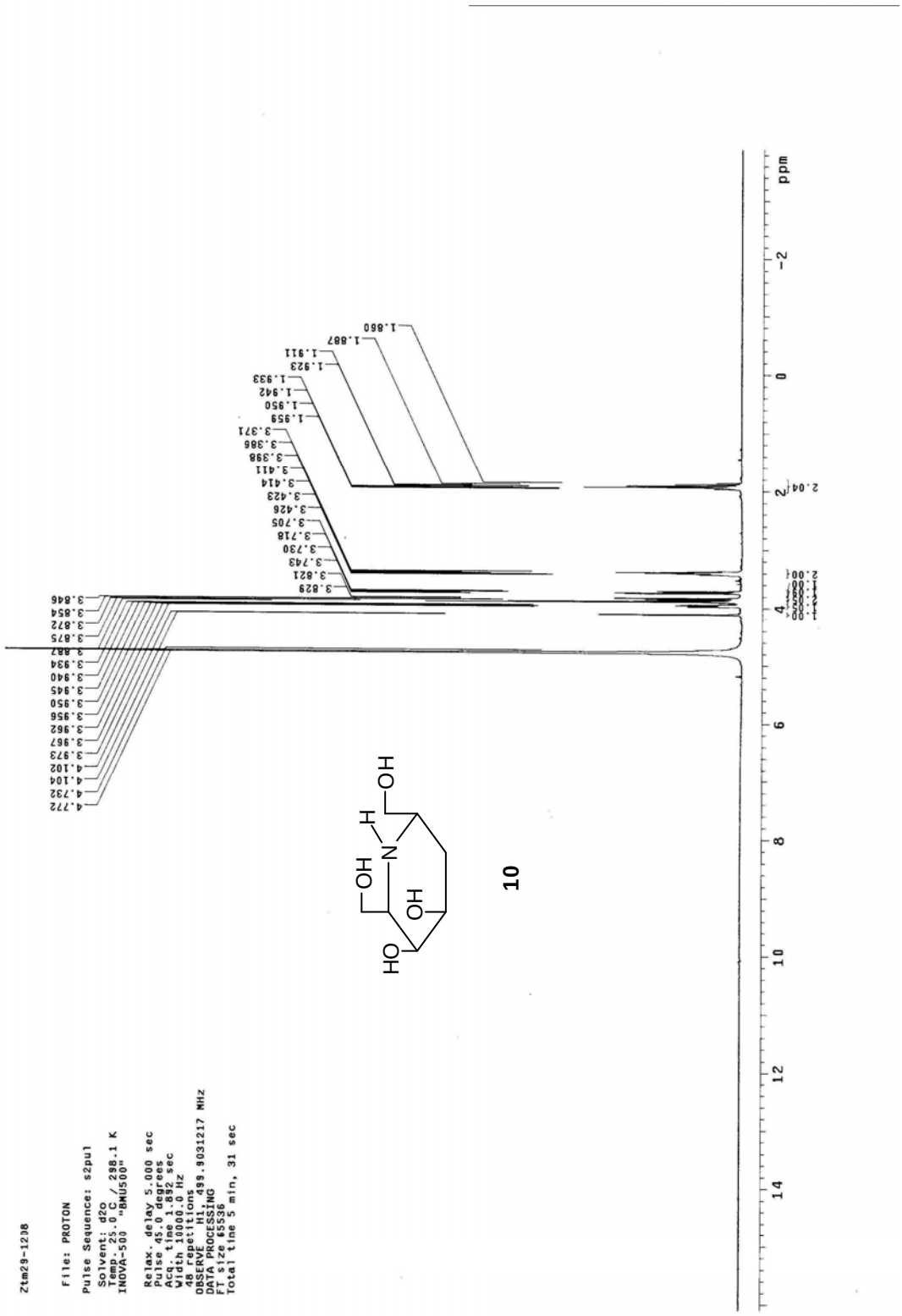

GL23D-0624
File: PROTON
Pulse Sequence: gHMBC
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.165 sec
Width 6218.9 Hz
2D Width 30165.9 Hz
32 repetitions
400 increments
OBSERVE H1, 499.9019068 MHz
DATA PROCESSING
Sine bell 0.082 sec
FT DATA PROCESSING
Sine bell 0.007sec
FT size 2048 x 4096
Total time 4 hr, 27 min, 58 sec
25
BnO
OBn
OBn
N
Cbz
CHO
F1 (ppm)
F2 (ppm)

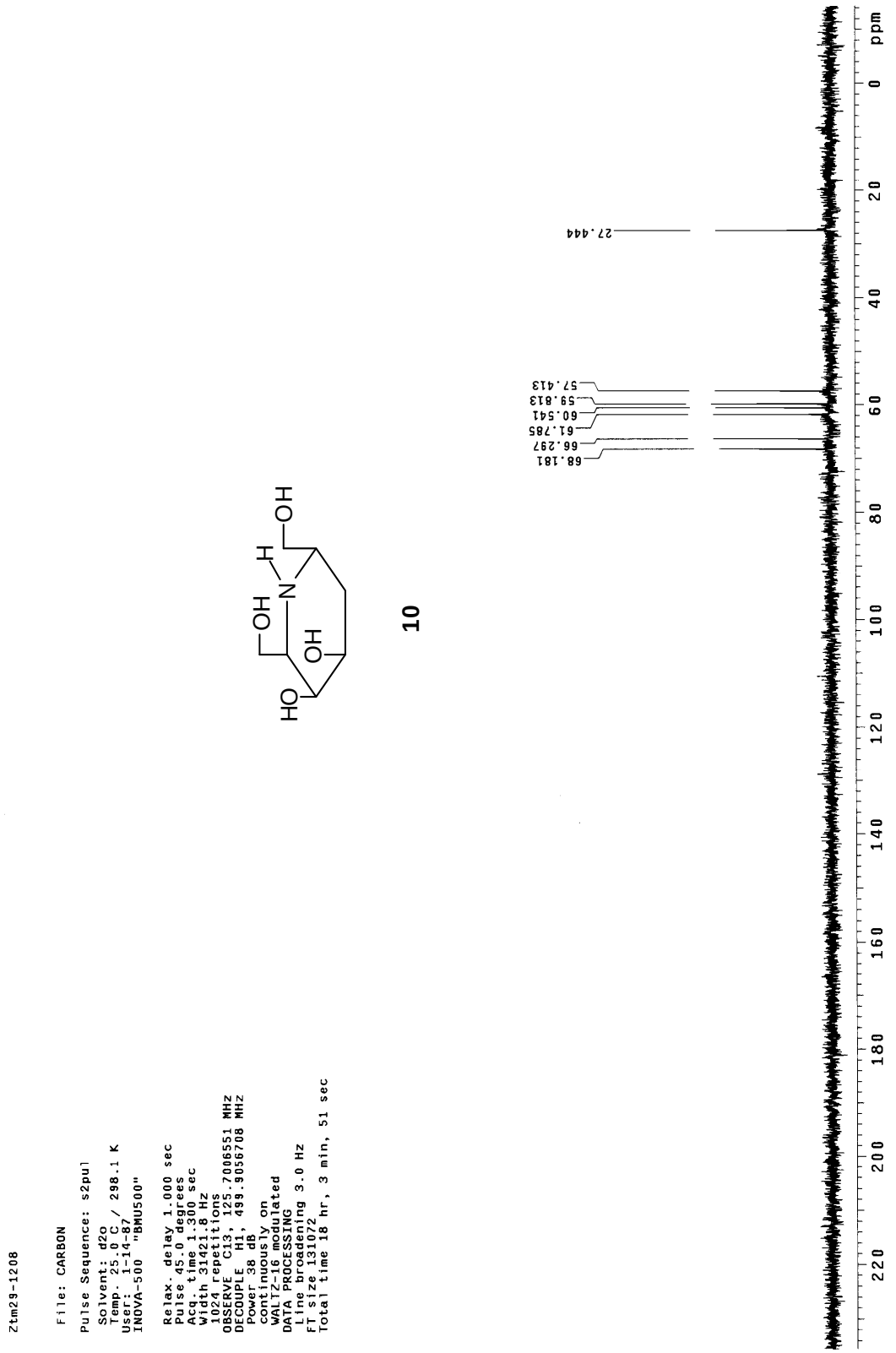

GL25D-1004
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 86.7 degrees
Acq. time 1.682 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9019035 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
7.325
7.321
7.319
7.314
7.309
7.305
7.301
7.297
7.293
7.286
7.279
7.275
7.273
7.270
7.267
7.263
7.256
7.253
7.245
7.242
7.238
7.233
7.229
7.217
5.302
5.293
5.194
5.169
5.163
5.138
4.952
4.949
4.943
4.938
4.933
4.929
4.739
4.715
4.626
4.603
4.512
4.488
4.414
4.390
4.173
4.162
3.949
3.929
3.926
3.907
3.899
3.891
3.848
3.842
3.825
3.820
3.787
3.777
3.766
3.383
2.042
1.577
1.257
1.243
-0.000
7.332
7.340
7.345
OH
Cbz
N
OBn
OBn
BnO
26
ppm
18.81
1.00
0.93
1.00
1.05
1.04
1.00
0.97
1.91
2.18
1.02


DFILE D:\中新山\zg1\GL25D-1004-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 824
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 19.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
OH
Cbz
N
OBn
OBn
BnO
26
54.698
64.407
66.096
68.066
68.387
71.239
72.582
72.756
76.571
77.000
77.420
112.645
127.439
127.596
127.876
128.230
128.312
128.444
128.535
135.713
137.625
138.219
138.565
154.430
PPM
0
25
50
75
100
125
150


GL25D-2
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.882 sec
Width 8609.6 Hz
32 repetitions
OBSERVE H1, 499.8019021 MHz
DATA PROCESSING
FT size 65536
Total time 3 min
-0.006
0.000
0.007
1.255
1.258
1.564
2.042
2.168
3.846
3.989
3.996
4.011
4.018
4.052
4.061
4.069
4.113
4.121
4.127
4.142
4.160
4.445
4.469
4.555
4.613
4.637
4.642
4.662
4.666
4.676
4.686
4.700
4.729
4.731
4.732
4.736
4.756
4.760
4.762
4.768
4.774
4.779
4.783
4.787
4.790
4.794
4.813
4.816
4.818
4.821
7.237
7.241
7.244
7.249
7.256
7.260
7.269
7.273
7.275
7.280
7.282
7.289
7.293
7.297
7.303
7.309
7.313
7.315
7.321
7.322
7.327
7.329
7.332
7.334
7.344
7.348
7.358
7.365
ppm
13.13
2.78
2.60
1.00
0.96
0.93
OBn
N
OBn
BnO
O
O
27


GL25D-2
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5376 repetitions
OBSERVE C13, 125.7004336 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 131072
Total time 6 hr, 34 min, 7 sec
27
O
O
N
OBn
OBn
BnO
-0.022
51.307
65.839
67.239
68.406
71.469
72.183
72.675
76.748
77.000
77.256
92.097
127.395
127.581
127.711
127.940
128.279
128.348
128.524
136.045
137.522
138.368
138.498
155.639
ppm
0
20
40
60
80
100
120
140
160
180
200
220


Ztm29-1298
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp: 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
48 repetitions
OBSERVE H1, 499.9031217 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 31 sec
ppm
10
HO
OH
N
H
OH
OH


Ztm29-1208
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1024 repetitions
OBSERVE C13, 125.7006551 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 18 hr, 3 min, 51 sec
10
OH
OH
N
H
OH
OH
HO
68.181
66.297
61.785
60.541
59.813
57.413
27.444
ppm
0
20
40
60
80
100
120
140
160
180
200
220

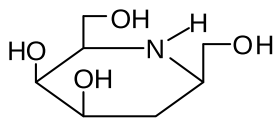
Ztm29-1208

File: PROTON

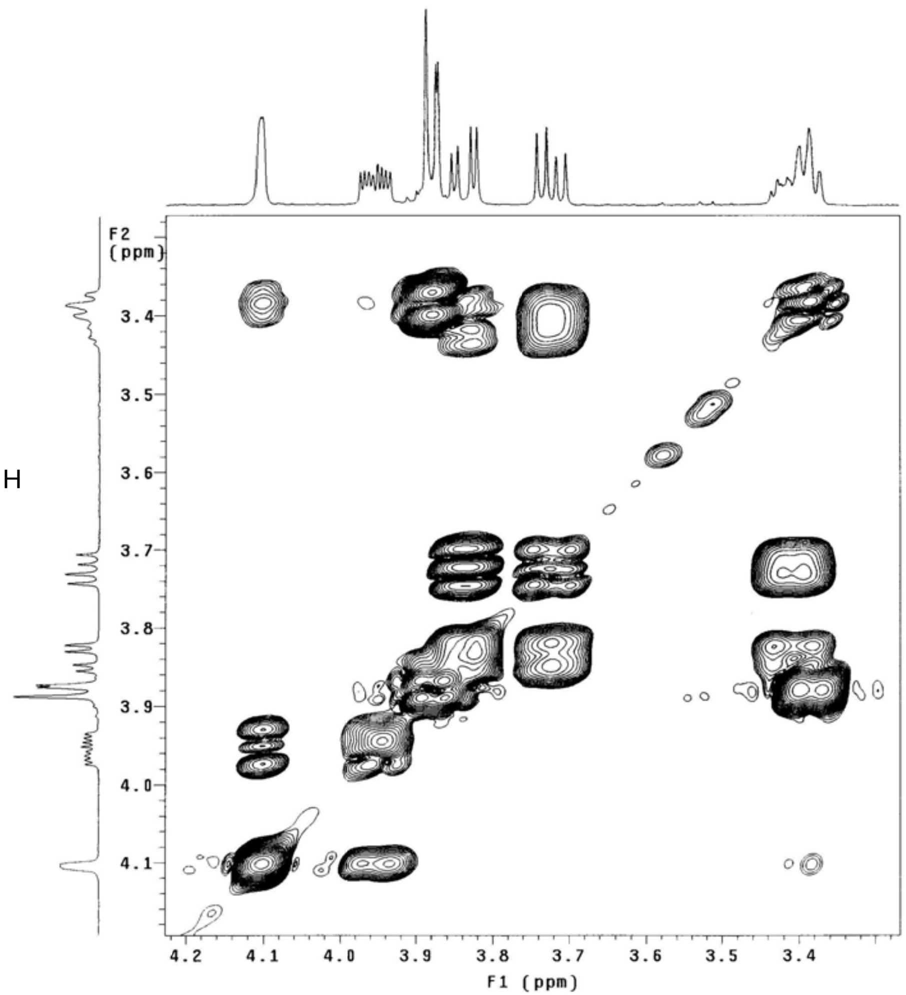
Pulse Sequence: gCOSY

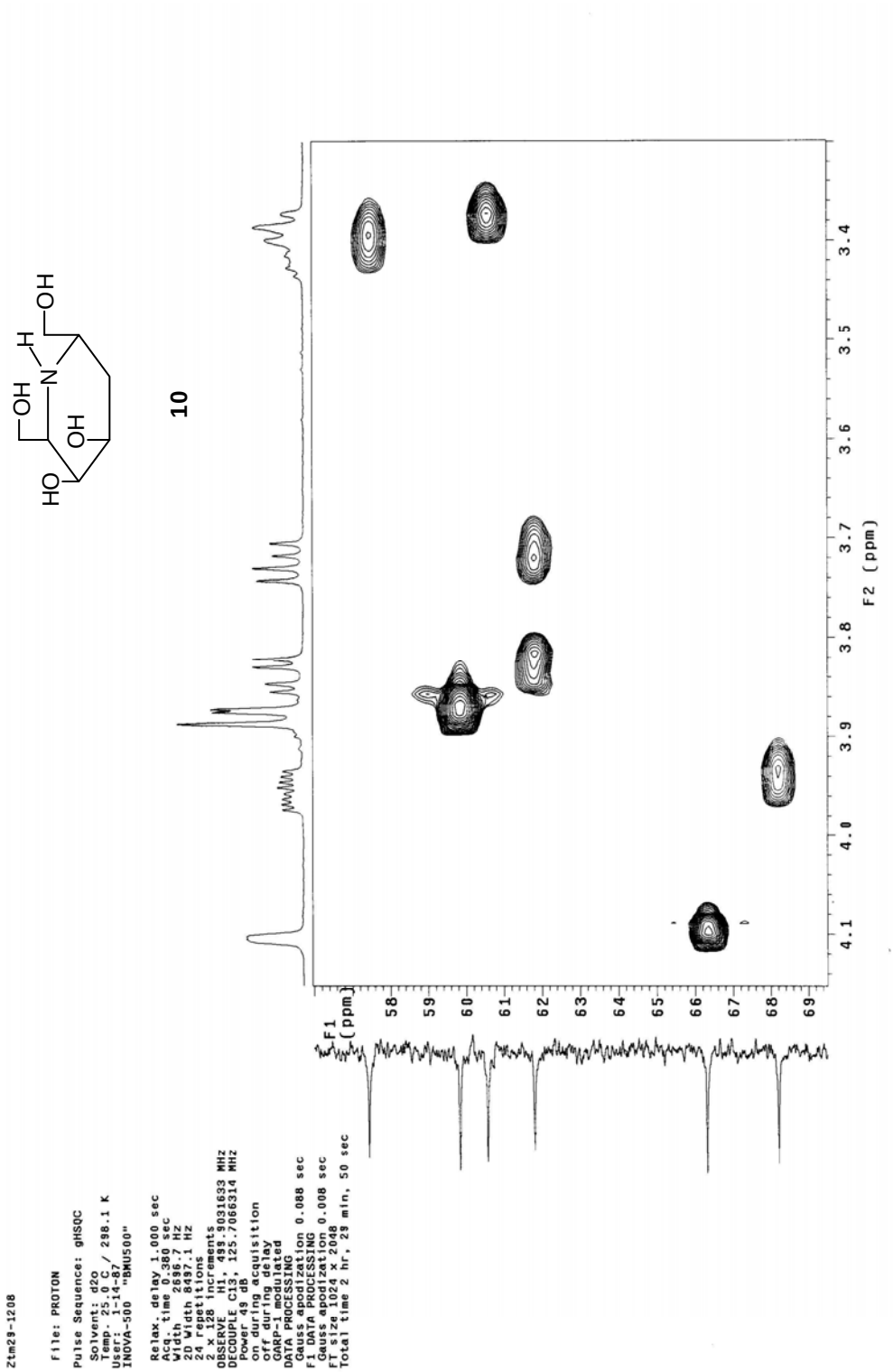
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"

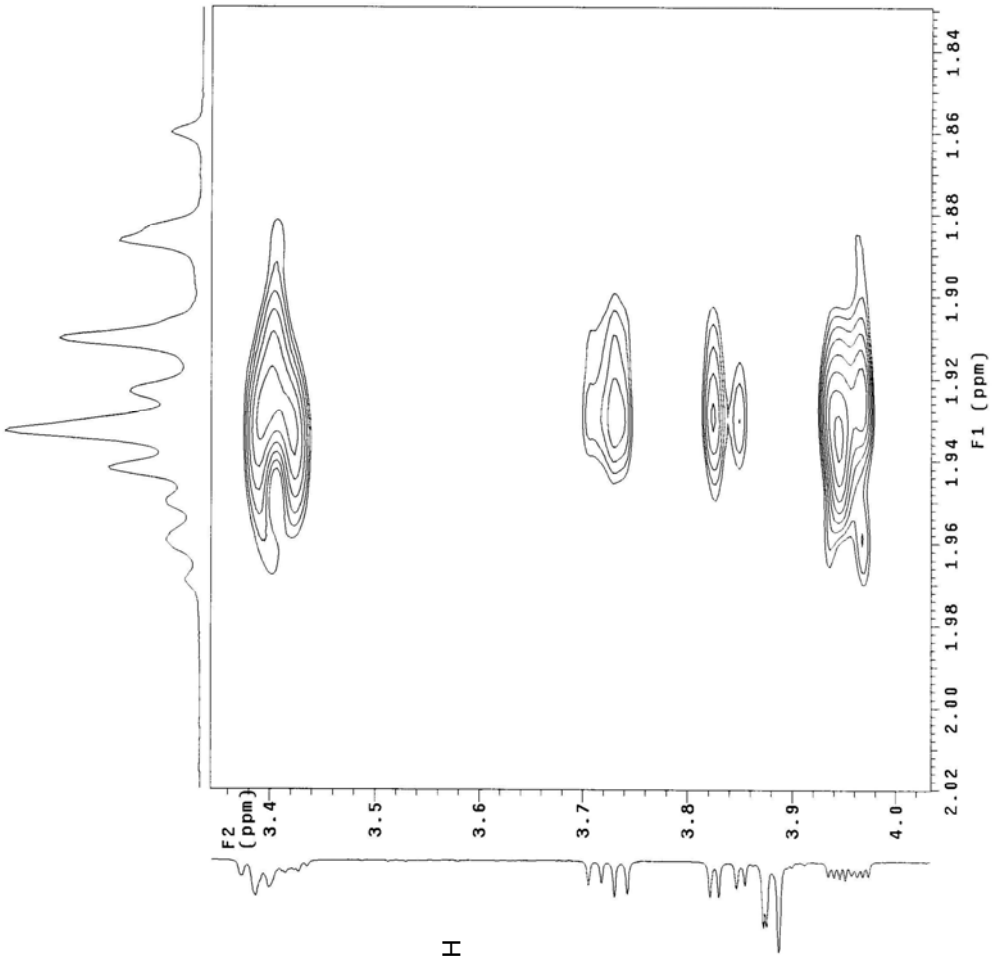
Relax. delay 1.000 sec
Acq. time 0.393 sec
Width 2604.7 Hz
2D Width 2604.7 Hz
16 repetitions
300 increments
OBSERVE H1, 499.9031225 MHz
DATA PROCESSING
Sq. sine bell 0.098 sec
F1 DATA PROCESSING
Sq. sine bell 0.059 sec
FT size 4096 x 4096
Total time 1 hr, 57 min, 27 sec

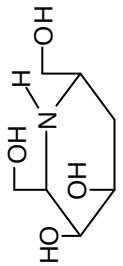


10



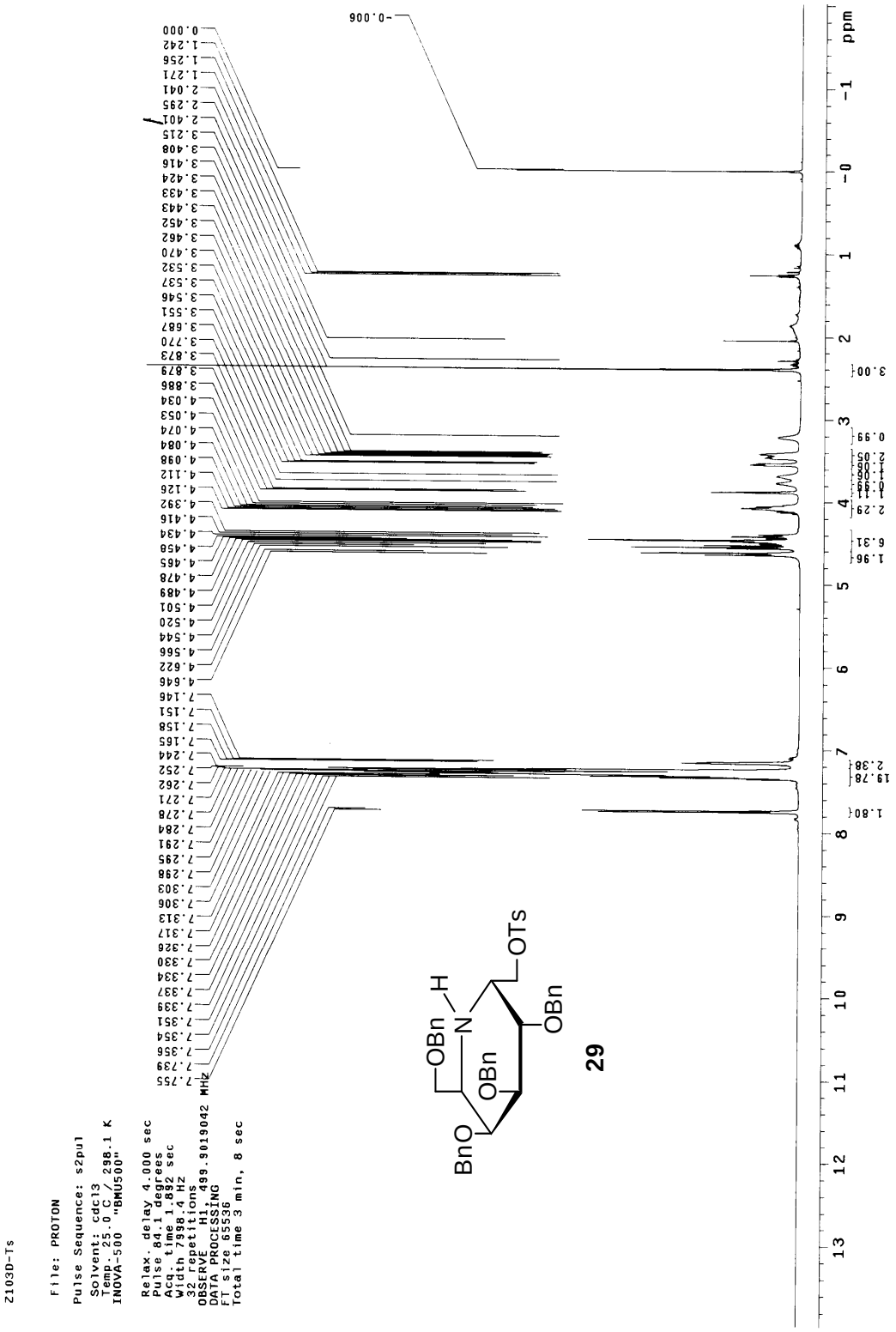

10
F1 (ppm)
F2 (ppm)
Ztm29-1208
File: PROTON
Pulse Sequence: gHSQC
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14--82
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.380 sec
Width 2564.7 HZ
2D Width 8497.1 HZ
24 repetitions
2 x 128 increments
OBSERVE H1, 499.9031633 MHz
DECOUPLE C13, 125.7066314 MHz
Power 49 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.088 sec
F1 DATA PROCESSING
Gauss apodization 0.008 sec
FT size 1024 x 2048
Total time 2 hr, 29 min, 50 sec

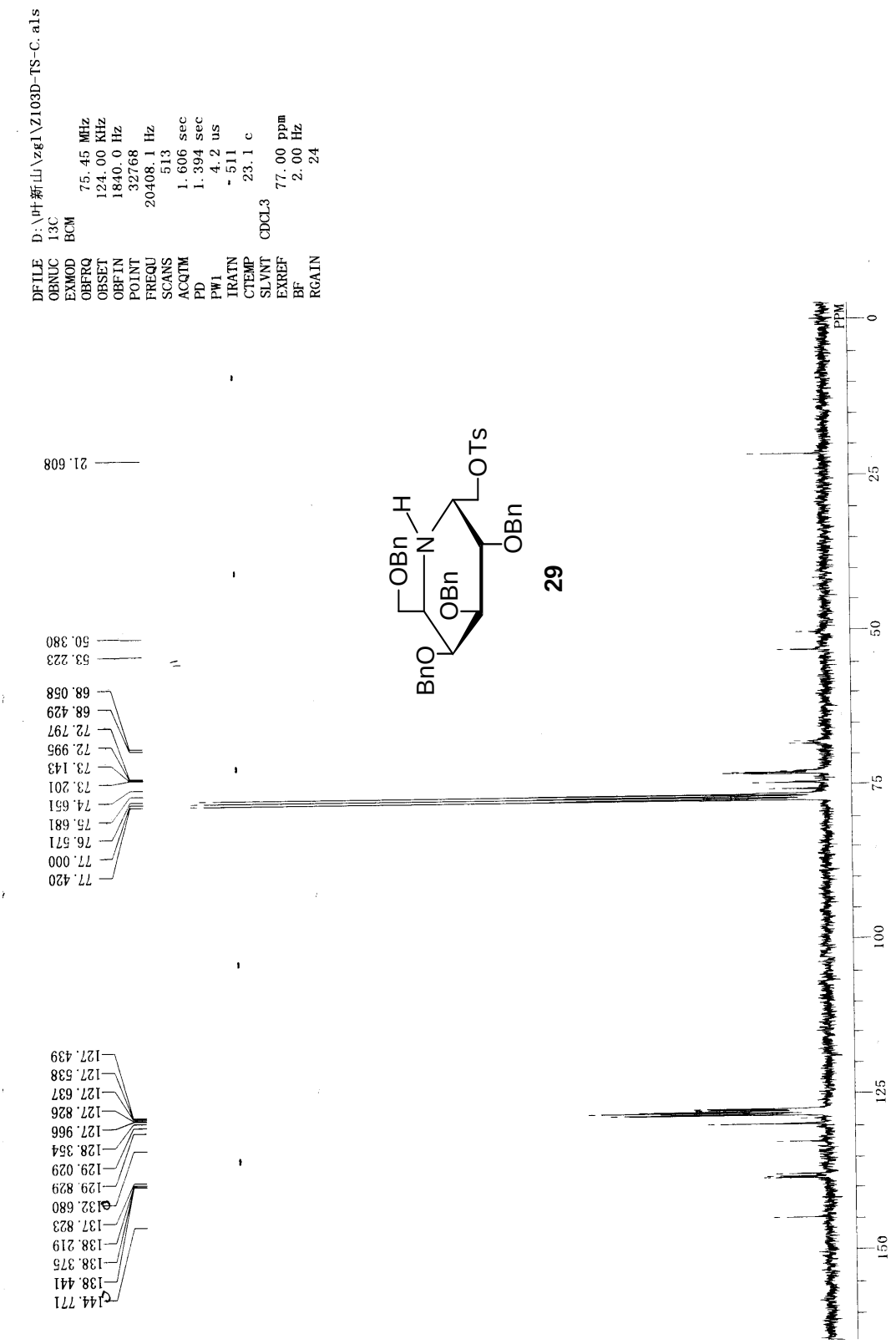


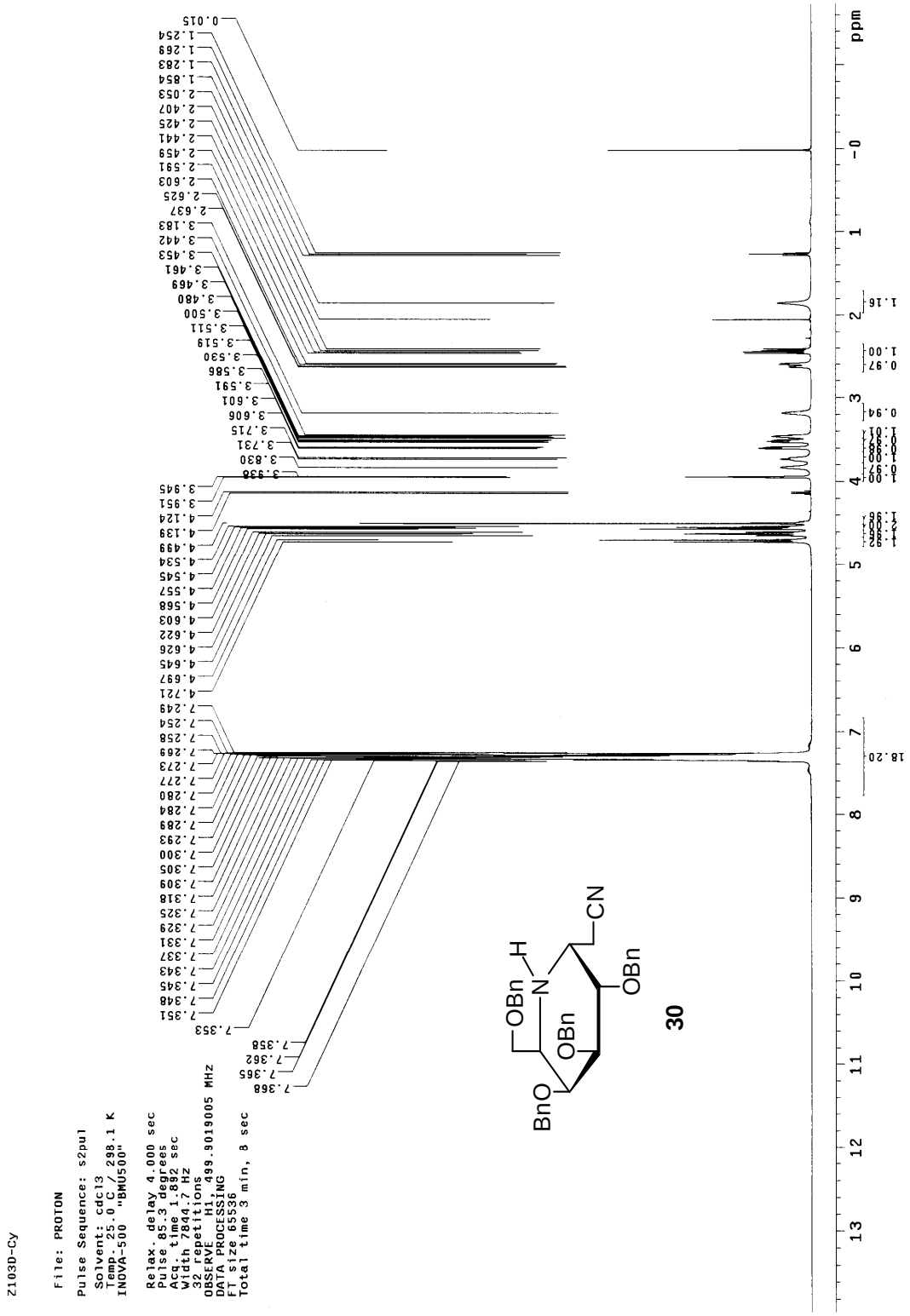


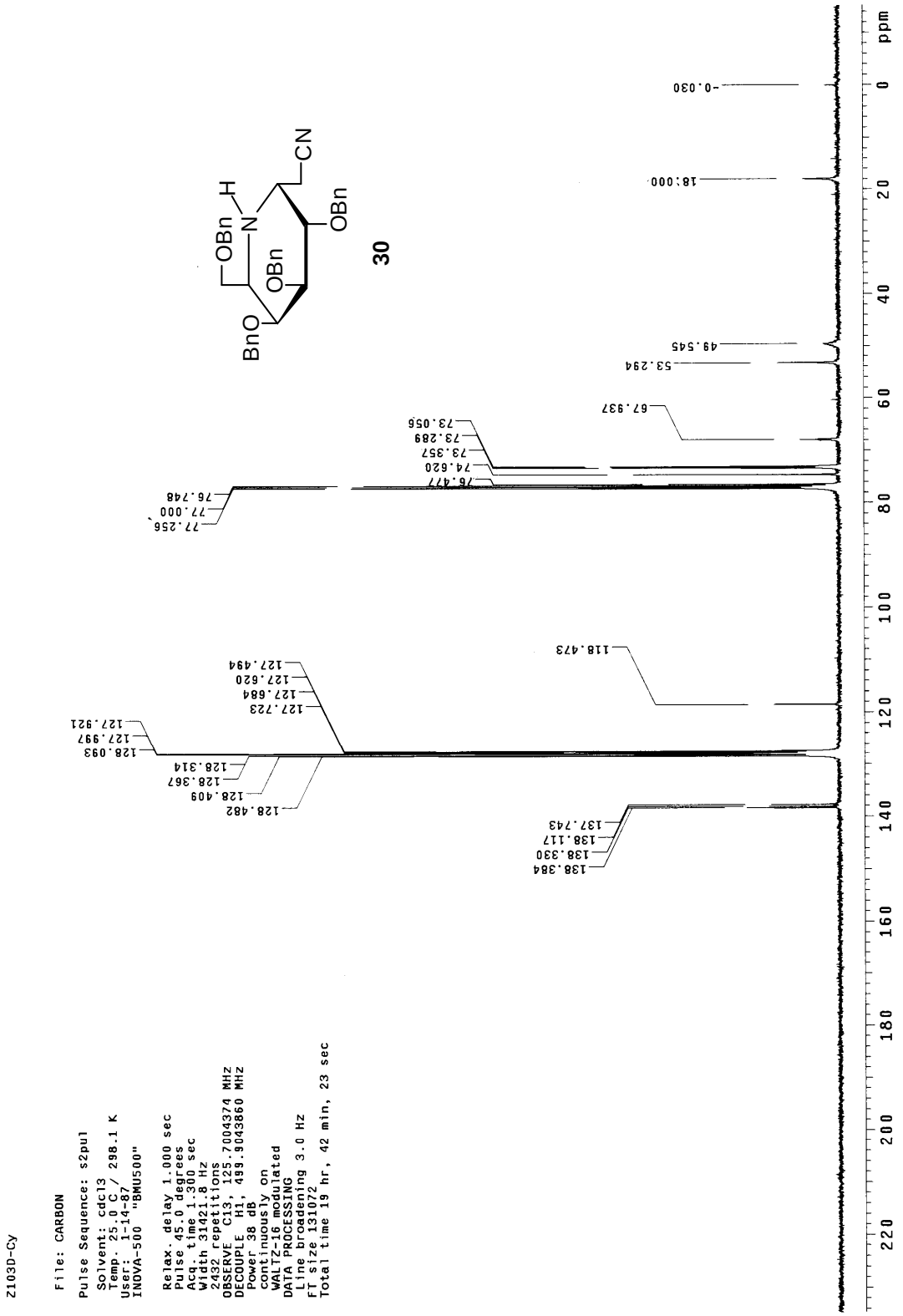
10

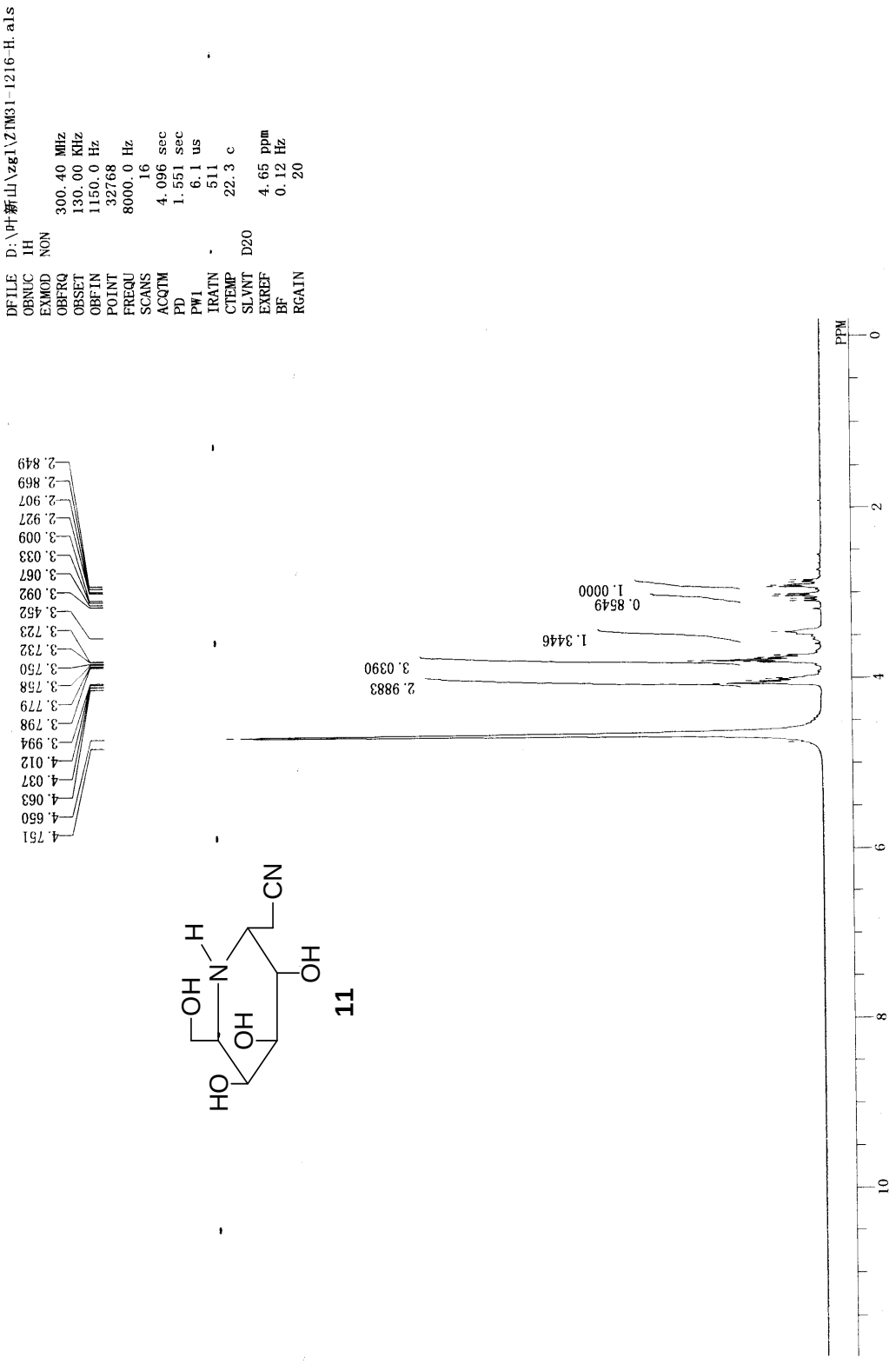
```
Ztm29-1208

   File: PROTON

Pulse Sequence: NOESY
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"

Relax. delay 1.000 sec
Mixing 0.800 sec
Acq. time 0.357 sec
Width  2871.6 Hz
2D Width 2871.6 Hz
32 repetitions
2 x 300 increments
OBSERVE   H1, 499.9031215 MHz
DATA PROCESSING
 Gauss apodization 0.082 sec
F1 DATA PROCESSING
 Gauss apodization 0.053 sec
FT size 4096 x 4096
Total time 11 hr, 52 min, 19 sec
```

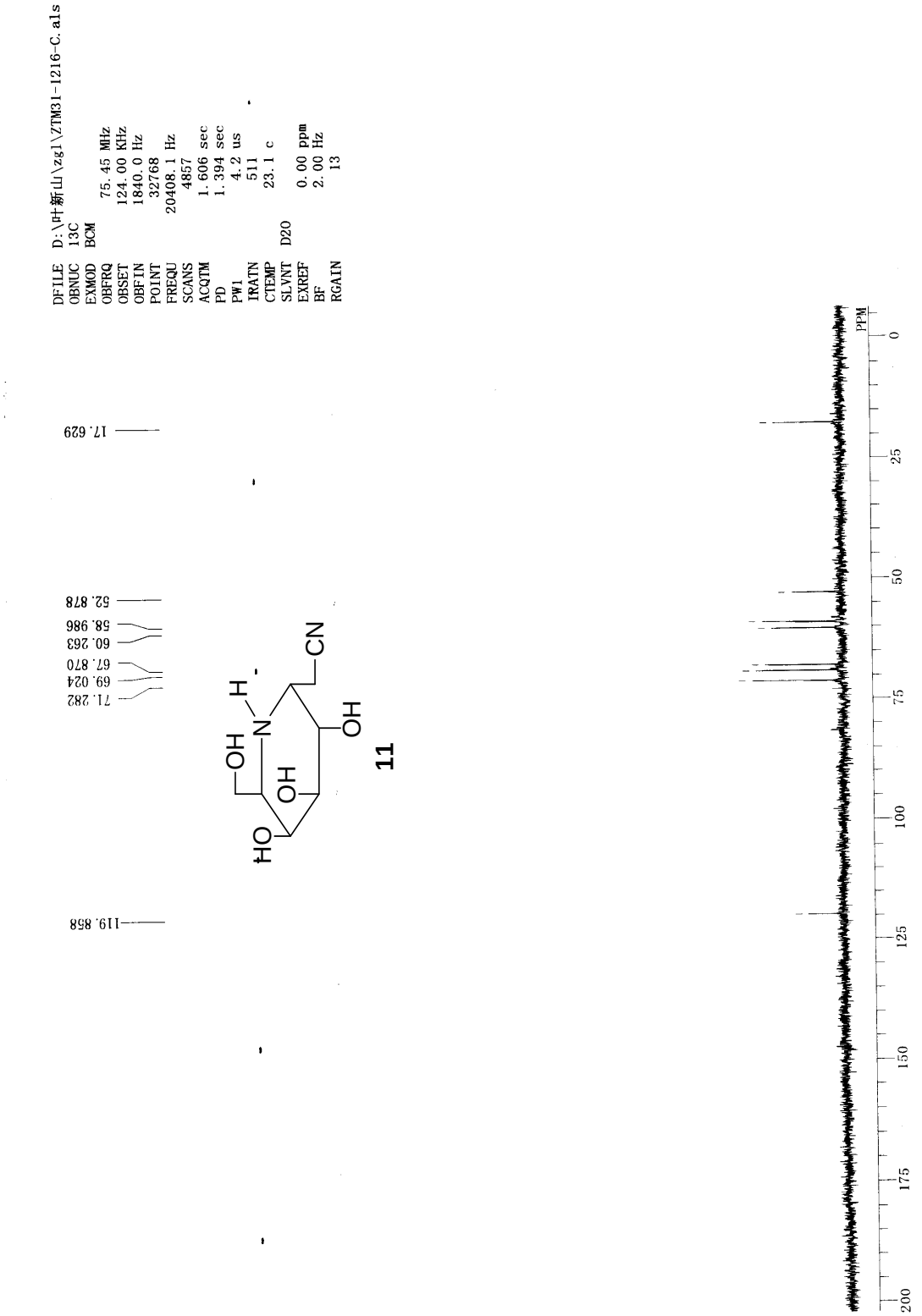

Z103D-Ts
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 84.1 degrees
Acq. time 1.892 sec
Width 7088.4 Hz
32 repetitions
OBSERVE H1, 499.9019042 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
OBn
H
N
OTs
OBn
OBn
BnO
29

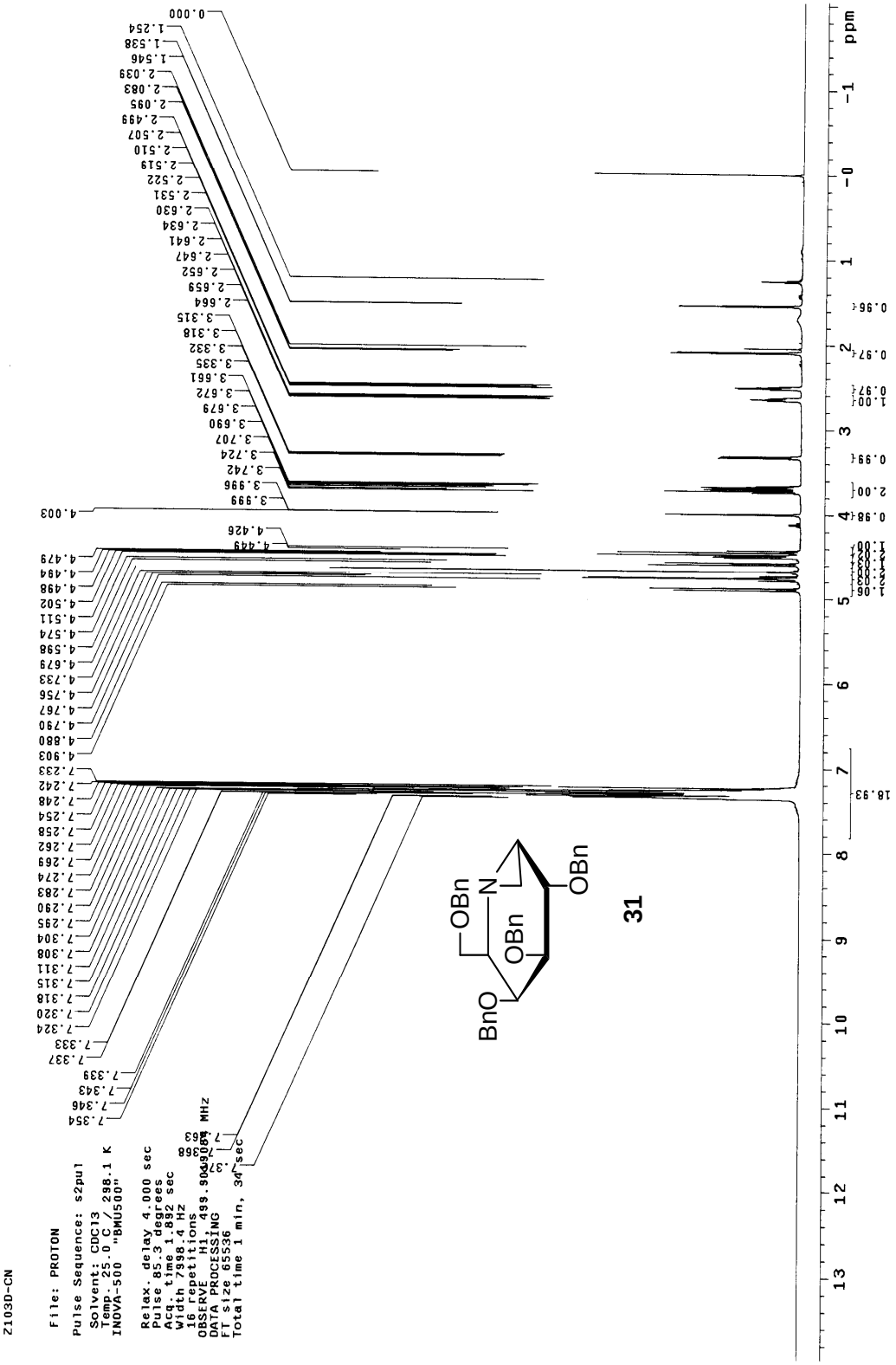

DFILE D:\VH新山\zg1\Z103D-TS-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 513
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN -511
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
21.608
50.380
53.223
68.058
68.429
72.797
72.995
73.143
73.201
74.651
75.681
76.571
77.000
77.420
127.439
127.538
127.637
127.826
127.966
128.354
129.029
129.829
132.680
137.823
138.219
138.375
138.441
144.771
OBn
H
N
OBn
OBn
BnO
OTs
29
PPM
0
25
50
75
100
125
150

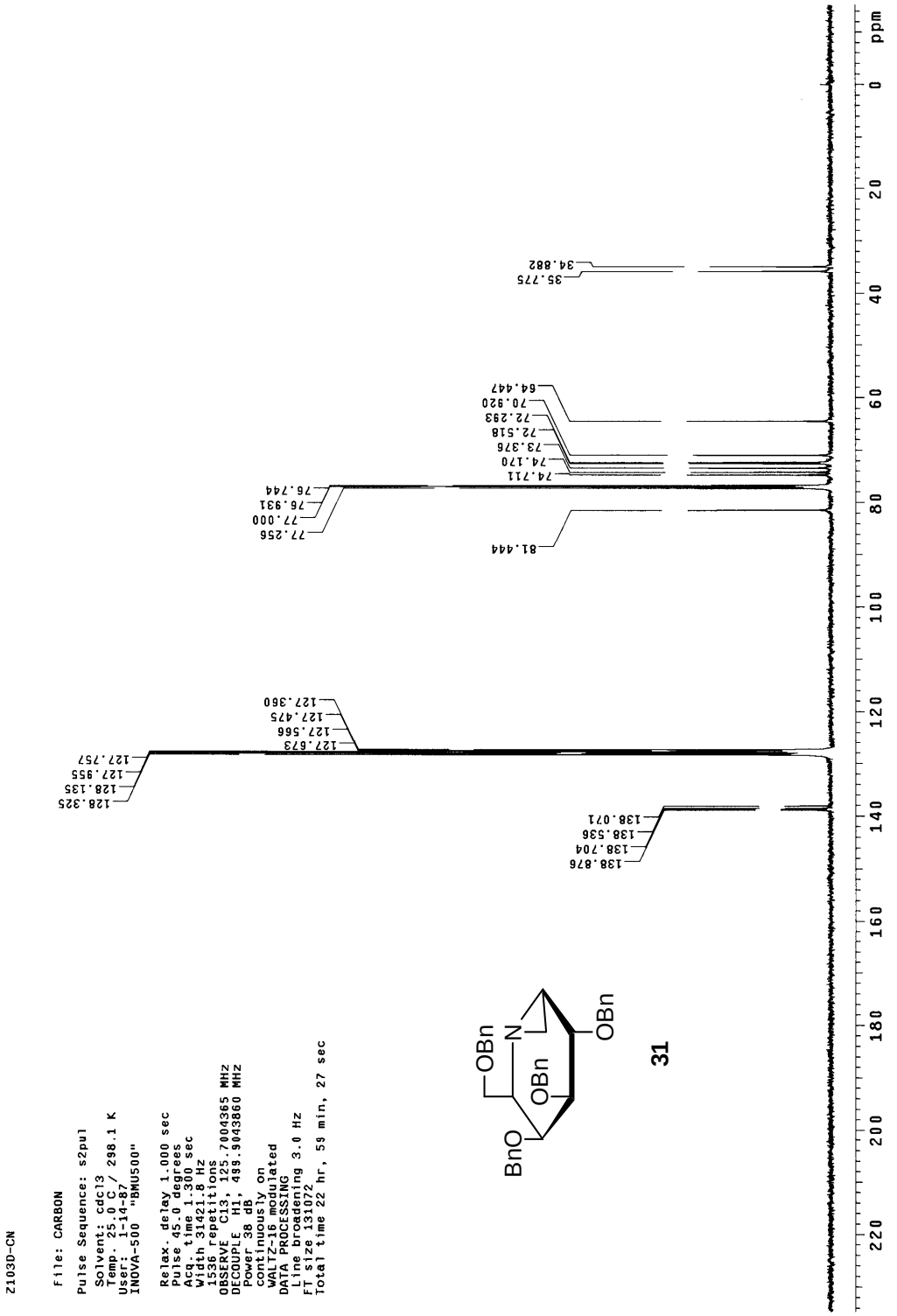

Z103D-Cy
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.882 sec
Width 7844.7 Hz
32 repetitions
OBSERVE H1, 499.9019005 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
OBn
N
H
CN
OBn
OBn
BnO
30

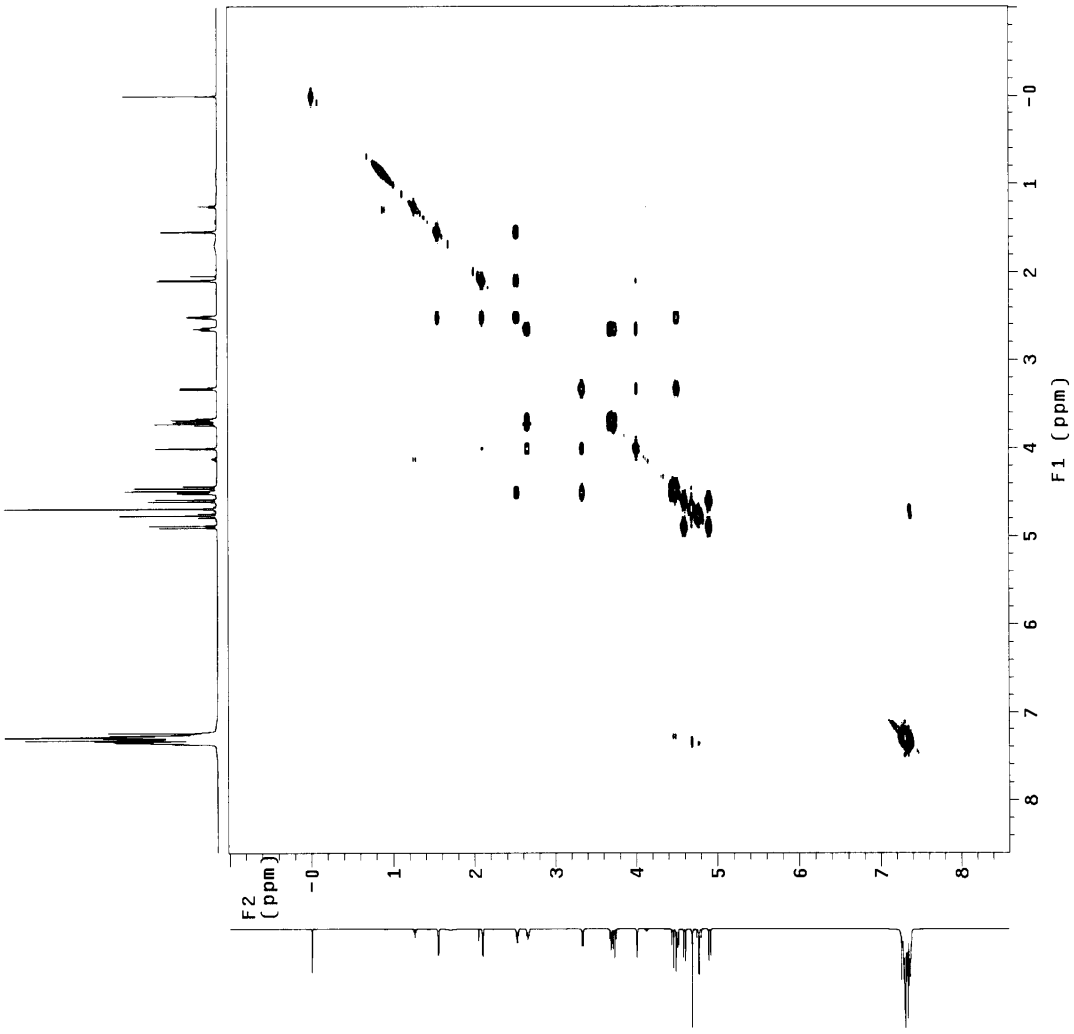

Z103D-Cy
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
2432 repetitions
OBSERVE C13, 125.7004374 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
BnO
OBn
OBn
N
H
OBn
CN
30
-0.030
18.000
49.545
53.294
67.937
73.056
73.289
73.357
74.620
76.477
76.748
77.000
77.256
118.473
127.494
127.620
127.684
127.723
127.921
127.997
128.093
128.314
128.367
128.409
128.482
137.743
138.117
138.330
138.384
ppm


DFILE D:\中新山\zg1\ZTM31-1216-H.als
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 16
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 511
CTEMP 22.3 c
SLVNT D20
EXREF 4.65 ppm
BF 0.12 Hz
RGAIN 20
2.849
2.869
2.907
2.927
3.009
3.033
3.067
3.092
3.452
3.723
3.732
3.750
3.758
3.779
3.798
3.994
4.012
4.037
4.063
4.650
4.751
2.9883
3.0390
1.3446
0.8549
1.0000
PPM
0
2
4
6
8
10
HO
OH
OH
N
H
CN
OH
11


DFILE D:\卢新山\zg1\ZTM31-1216-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 4857
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.1 c
SLVNT D2O
EXREF 0.00 ppm
BF 2.00 Hz
RGAIN 13
17.629
52.878
58.986
60.263
67.870
69.024
71.282
119.858
OH
H
N
CN
OH
OH
HO
11
PPM
0
25
50
75
100
125
150
175
200


Z103D-CN
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
16 repetitions
OBSERVE H1, 499.9000 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
OBn
N
OBn
OBn
BnO
31
ppm

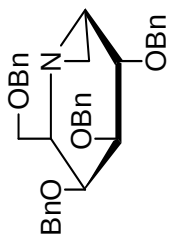

Z103D-CN
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1536 repetitions
OBSERVE C13, 125.7004365 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 22 hr, 59 min, 27 sec
128.325
128.135
127.955
127.757
127.673
127.566
127.475
127.360
138.876
138.704
138.536
138.071
81.444
77.256
77.000
76.931
76.744
74.711
74.170
73.376
72.518
72.293
70.920
64.447
35.775
34.882
ppm
OBn
N
OBn
OBn
BnO
31



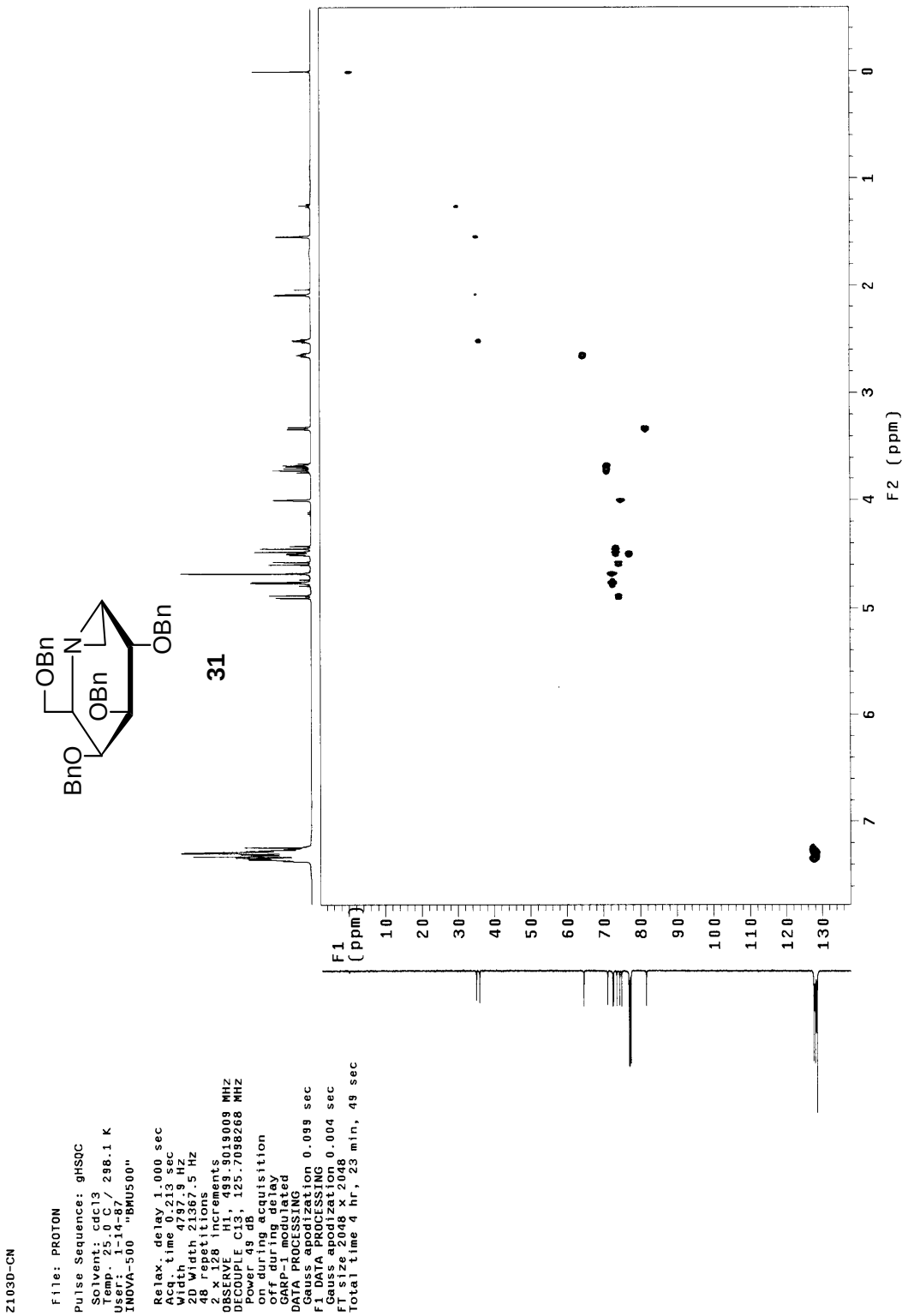
Z103D-CN

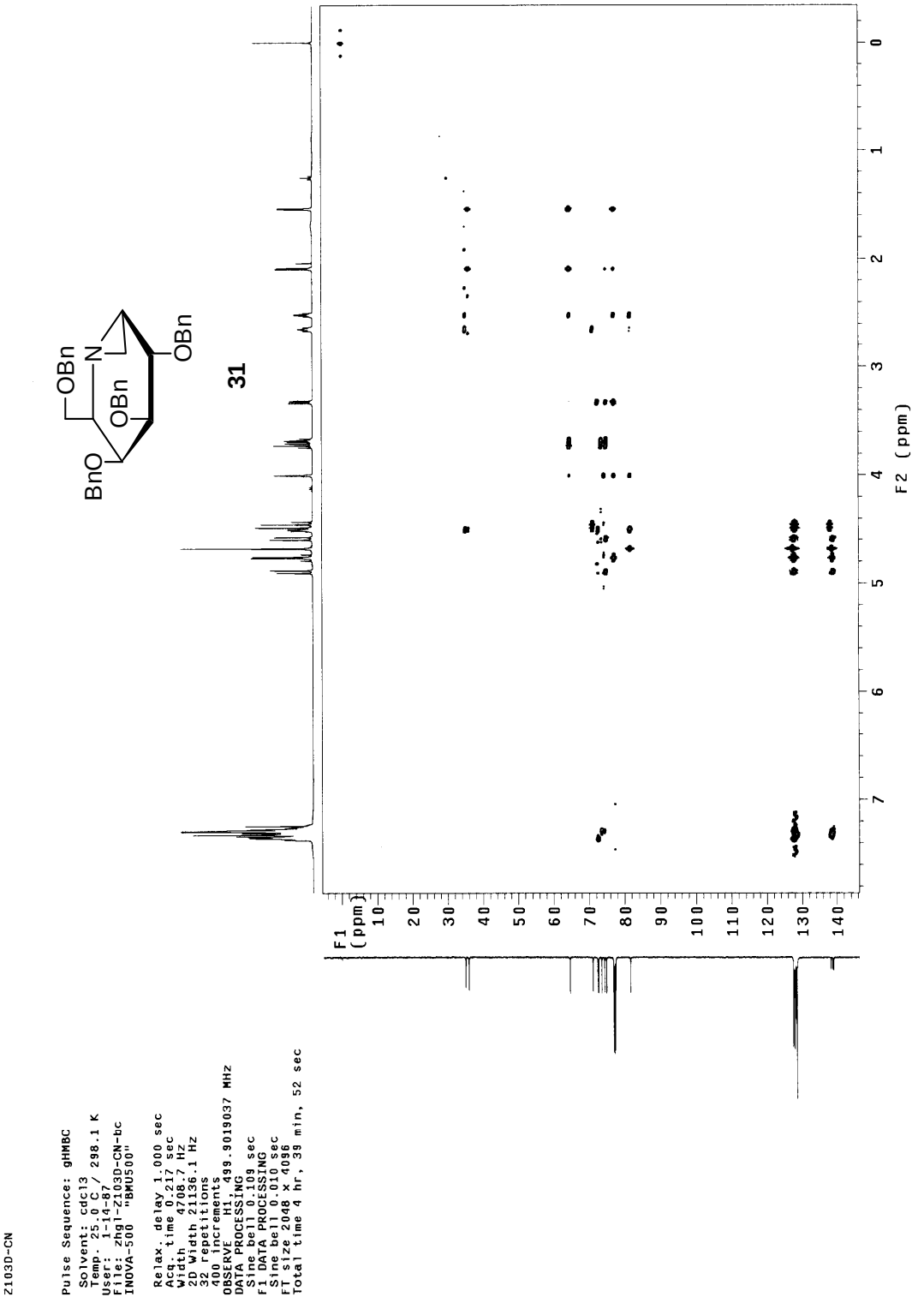
Pulse Sequence: gCOSY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: zhg1-Z103D-CN-cosy
INOVA-500 "BMUS00"

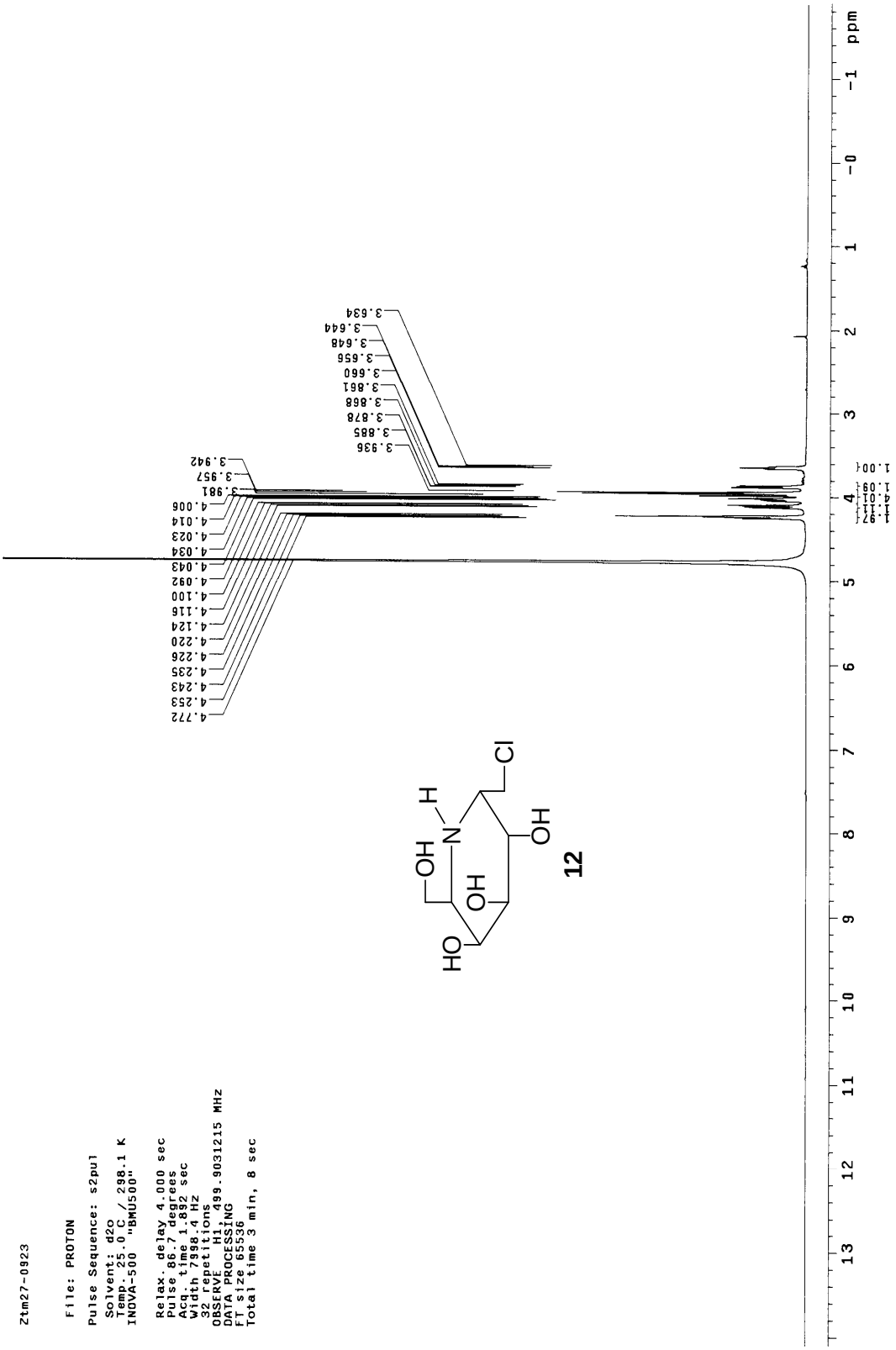
Relax. delay 1.000 sec
Acq. time 0.213 sec
Width 4804.5 Hz
2D Width 4804.5 Hz
16 repetitions
256 increments
OBSERVE H1, 499.9019037 MHz
DATA PROCESSING
Sq. sine bell 0.107 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
FT size 4096 x 4096
Total time 1 hr, 25 min, 50 sec

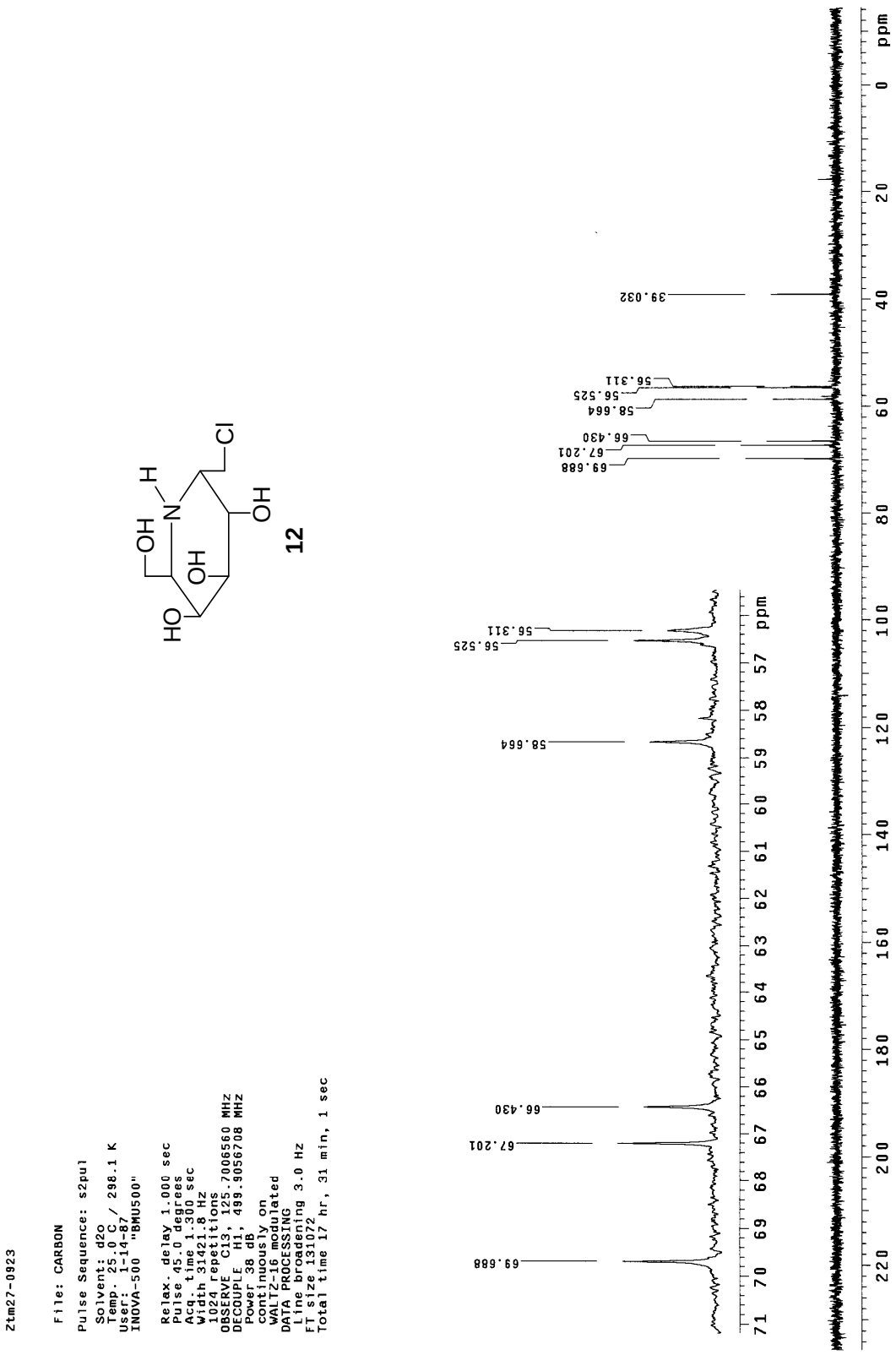


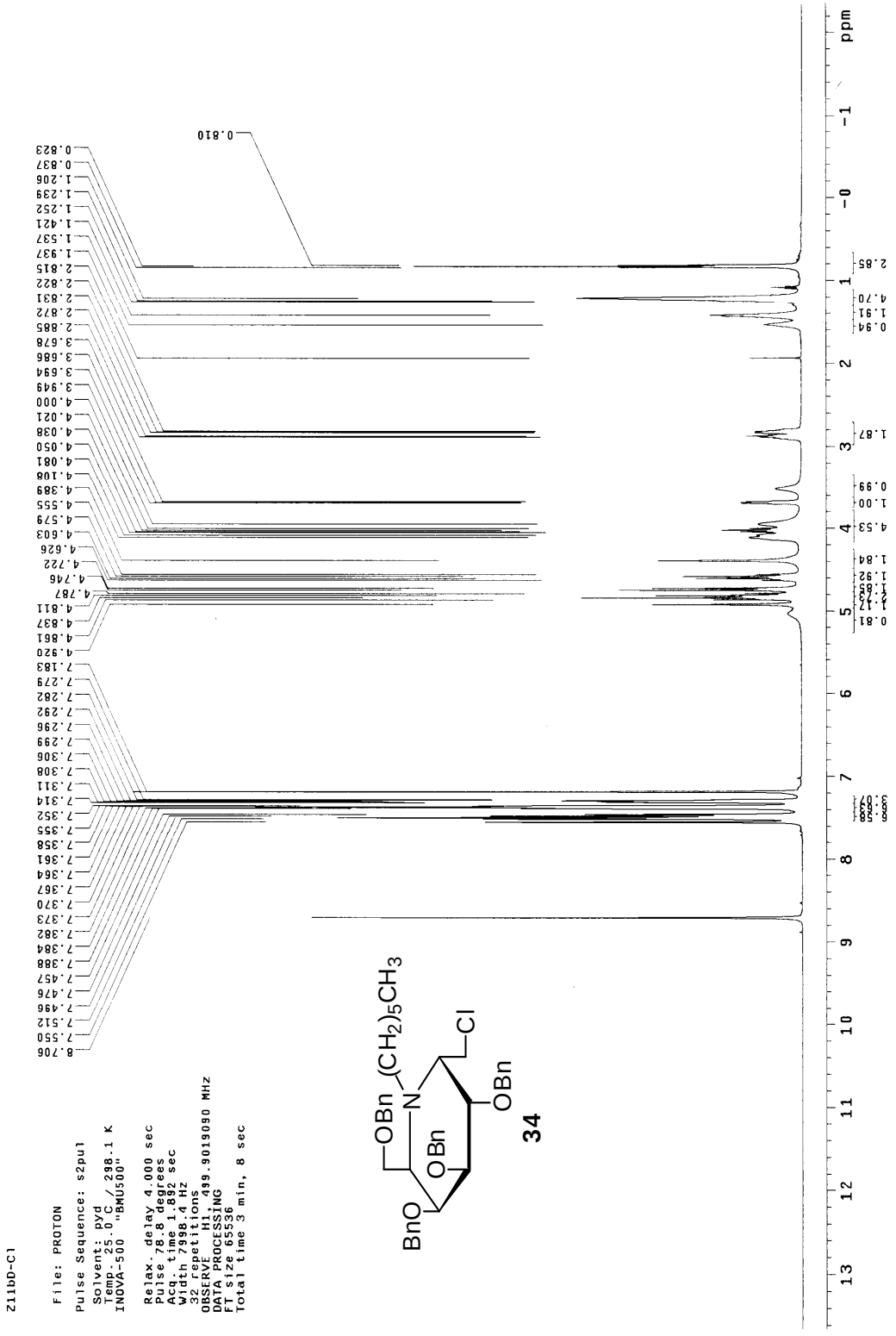
31

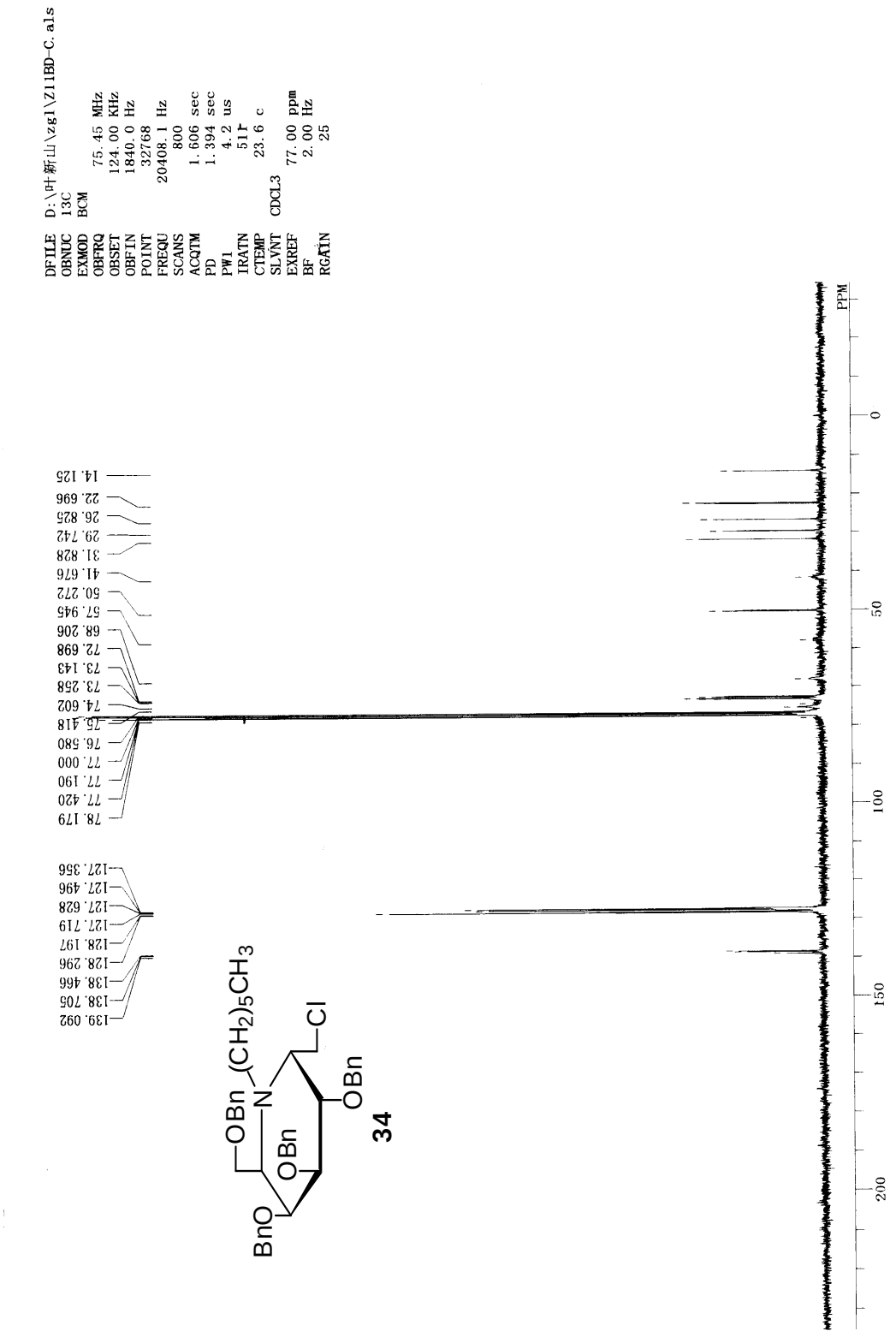

Z103D-CN
File: PROTON
Pulse Sequence: gHSQC
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.213 sec
Width 47379.9 Hz
20 Width 21367.5 Hz
48 repetitions
256 increments
OBSERVE H1, 499.9019009 MHz
DECOUPLE C13, 125.7098268 MHz
Power 49 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.099 sec
F1 DATA PROCESSING
Gauss apodization 0.004 sec
FT size 2048 x 2048
Total time 4 hr, 23 min, 49 sec
OBn
N
OBn
OBn
BnO
31
F1 (ppm)
F2 (ppm)

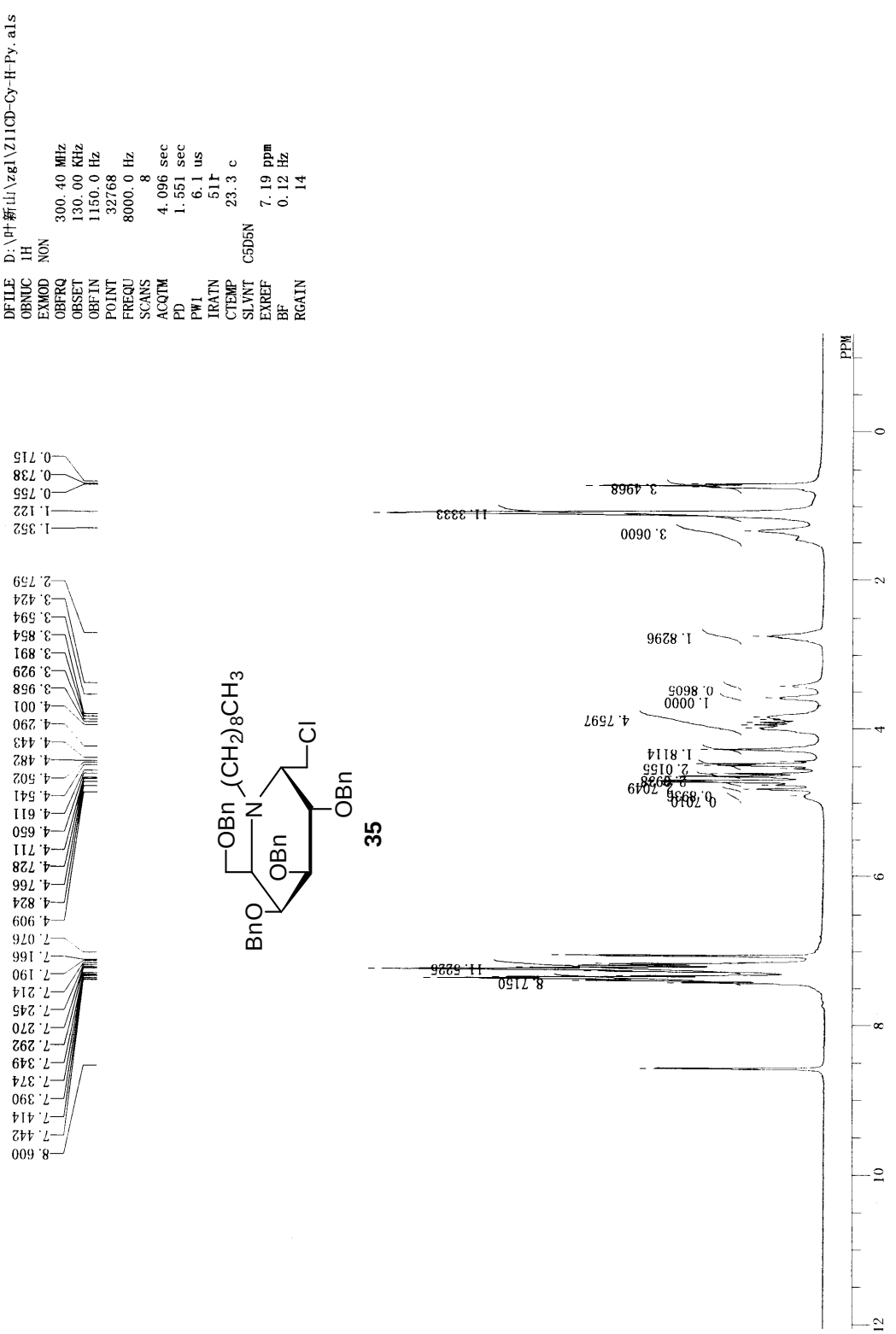

OBn
N
OBn
OBn
BnO
31
F1 (ppm)
F2 (ppm)
Z103D-CN
Pulse Sequence: gHMBC
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: zhgl-Z103D-CN-bc
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.217 sec
Width 4708.7 Hz
2D Width 21136.1 Hz
32 repetitions
400 increments
OBSERVE H1 499.9019037 MHz
DATA PROCESSING
Sine bell 0.109 sec
F1 DATA PROCESSING
Sine bell 0.010 sec
FT size 2048 x 4096
Total time 4 hr, 39 min, 52 sec

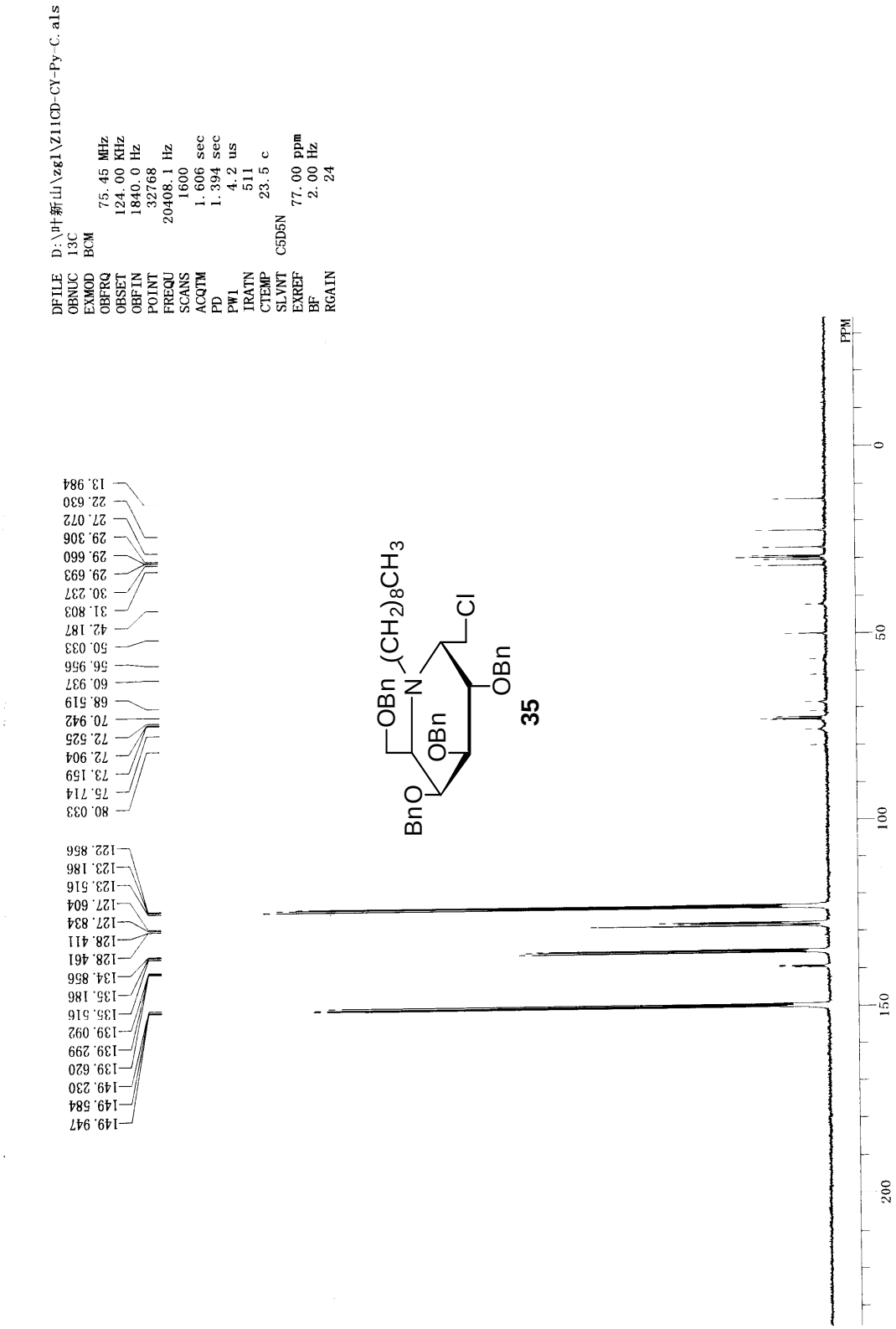

Ztm27-0923
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 86.7 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9031215 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
4.772
4.253
4.243
4.235
4.226
4.220
4.124
4.116
4.100
4.092
4.043
4.034
4.023
4.014
4.006
3.981
3.957
3.942
3.936
3.885
3.878
3.868
3.861
3.660
3.656
3.648
3.644
3.634
1.00
1.09
4.01
11.11
1.97
ppm
12

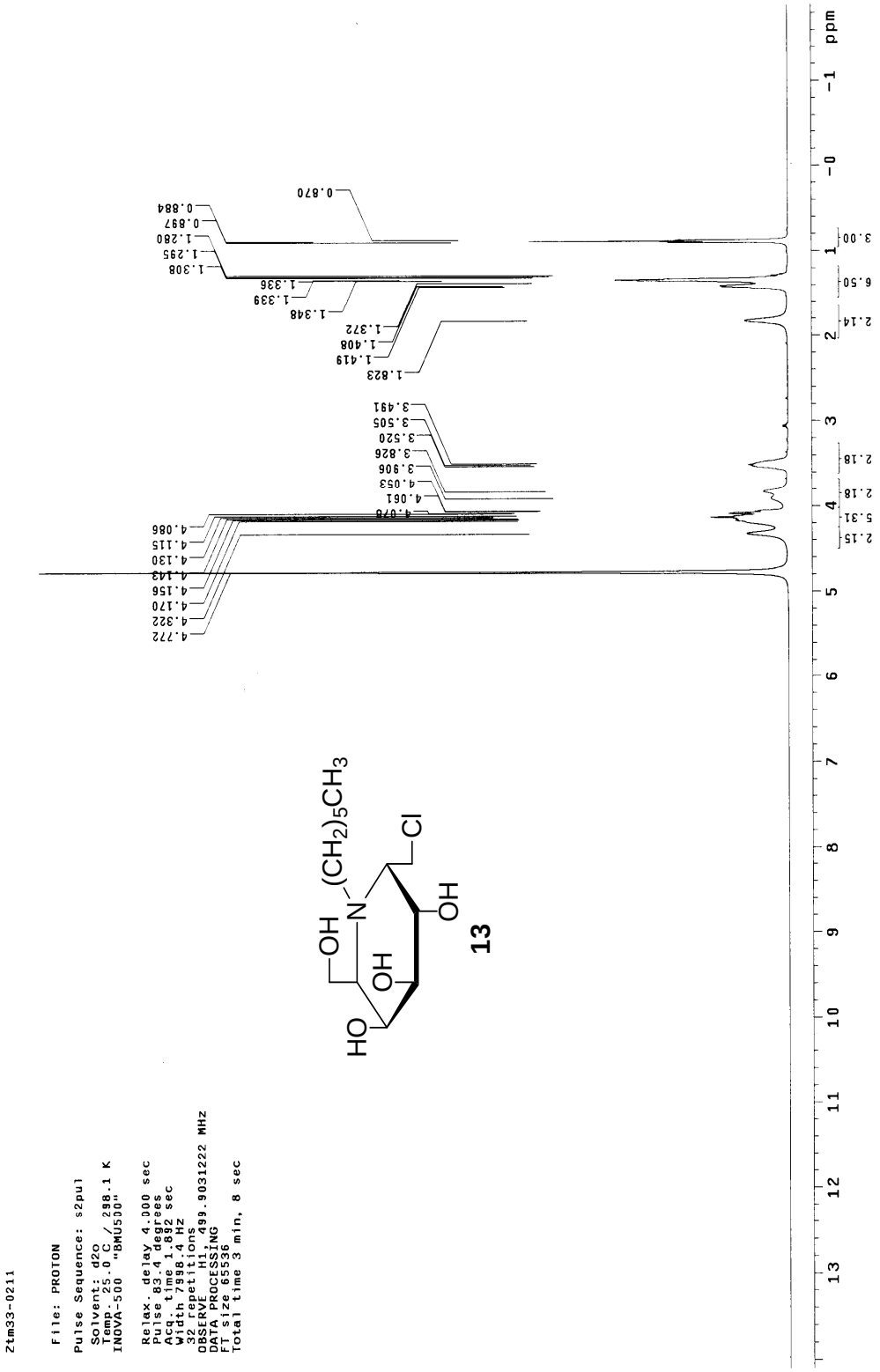

Ztm27-0923
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1024 repetitions
OBSERVE C13, 125.7006560 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 17 hr, 31 min, 1 sec
HO
OH
OH
N
H
Cl
OH
12
69.688
67.201
66.430
58.664
56.525
56.311
39.032
ppm

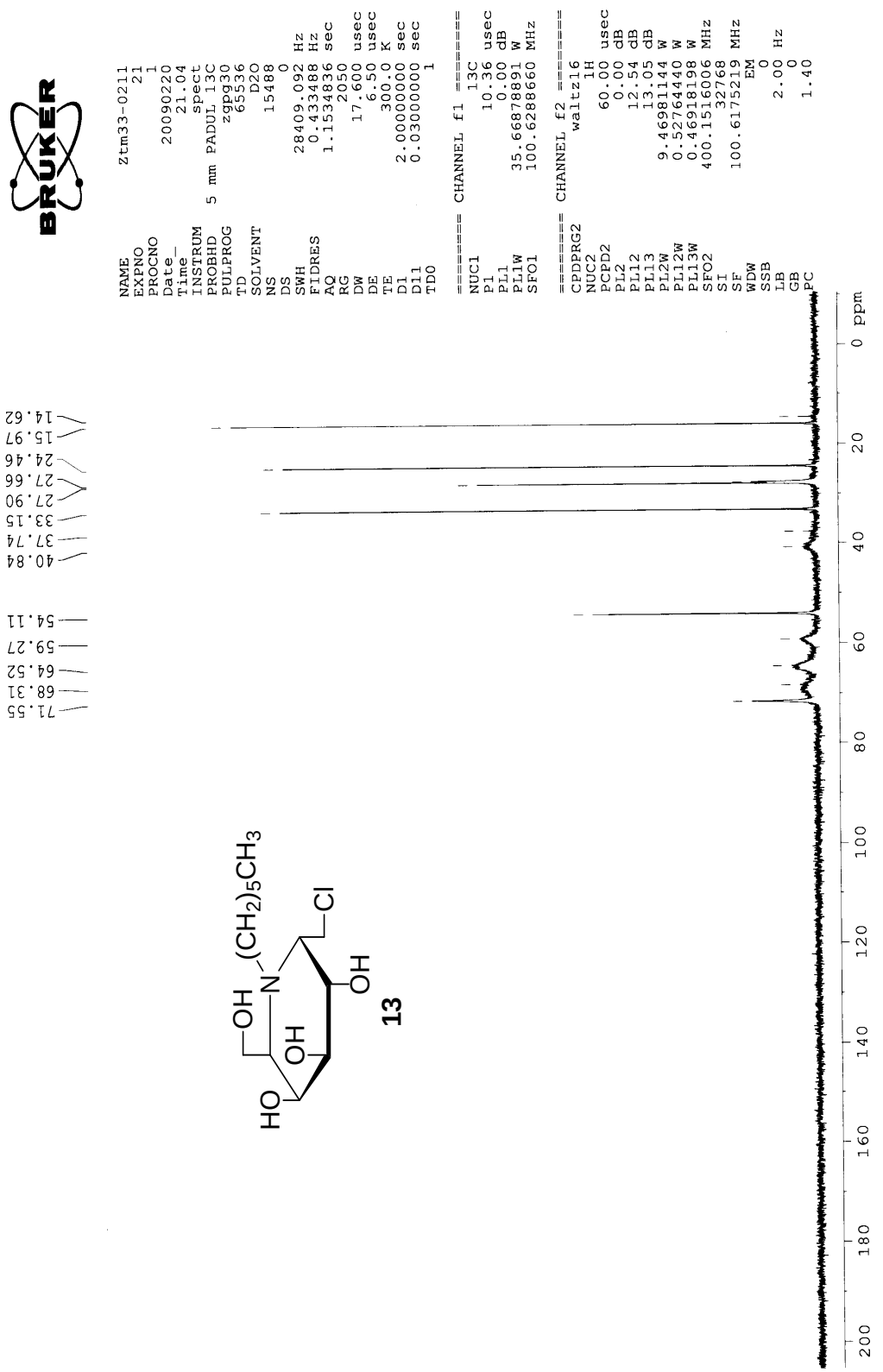

Z11bD-Cl
File: PROTON
Pulse Sequence: s2pul
Solvent: pyd
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 78.8 degrees
Acq. time 1.882 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9019090 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
BnO
OBn
N
(CH2)5CH3
OBn
Cl
OBn
34

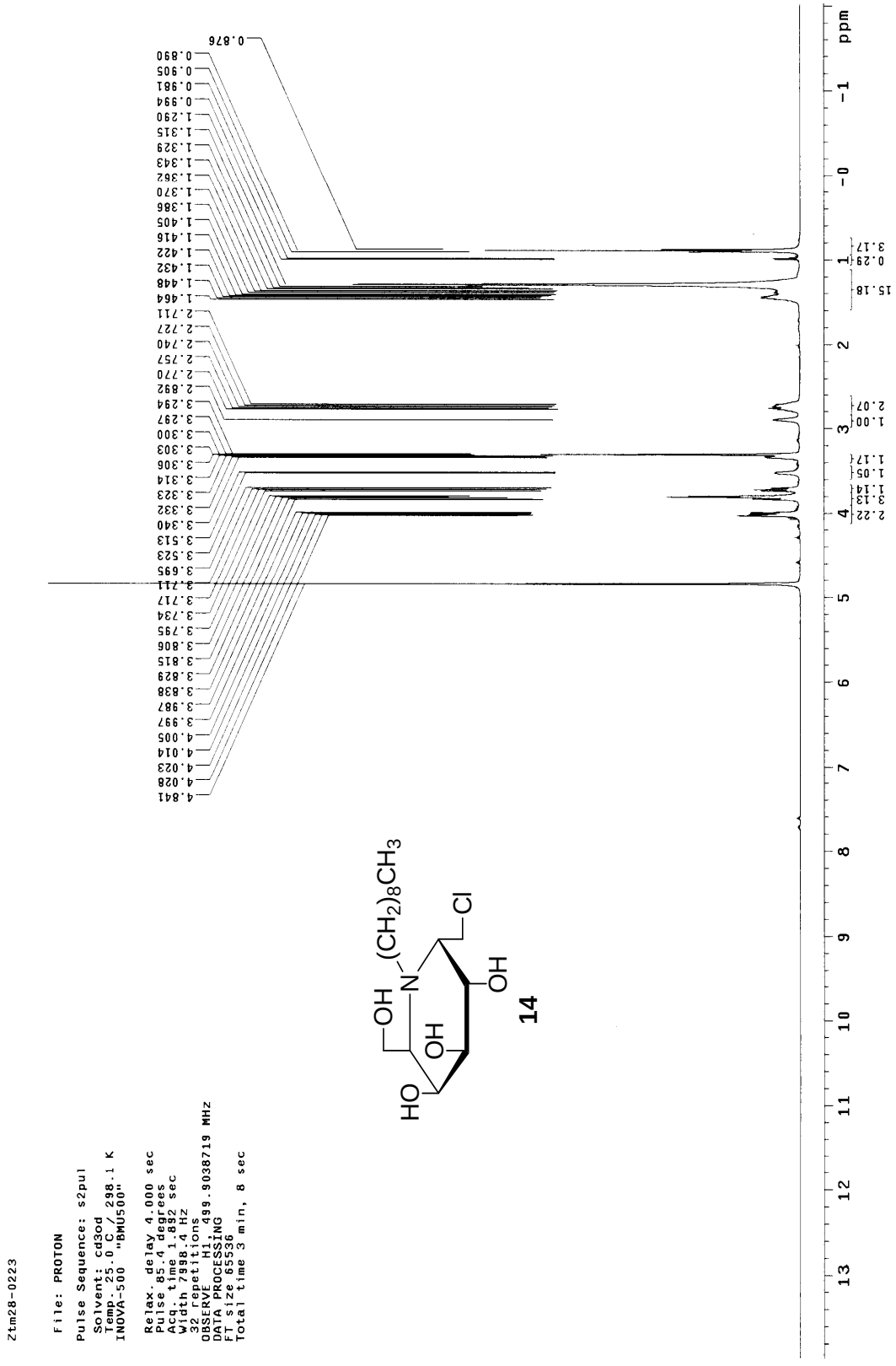

DFILE D:\pH新山\zg1\Z11BD-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 800
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 51
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
14.125
22.696
26.825
29.742
31.828
41.676
50.272
57.945
68.206
72.698
73.143
73.258
74.602
76.418
76.580
77.000
77.190
77.420
78.179
127.356
127.496
127.628
127.719
128.197
128.296
138.466
138.705
139.092
PPM
0
50
100
150
200
BnO
OBn
N
OBn
(CH2)5CH3
Cl
OBn
34

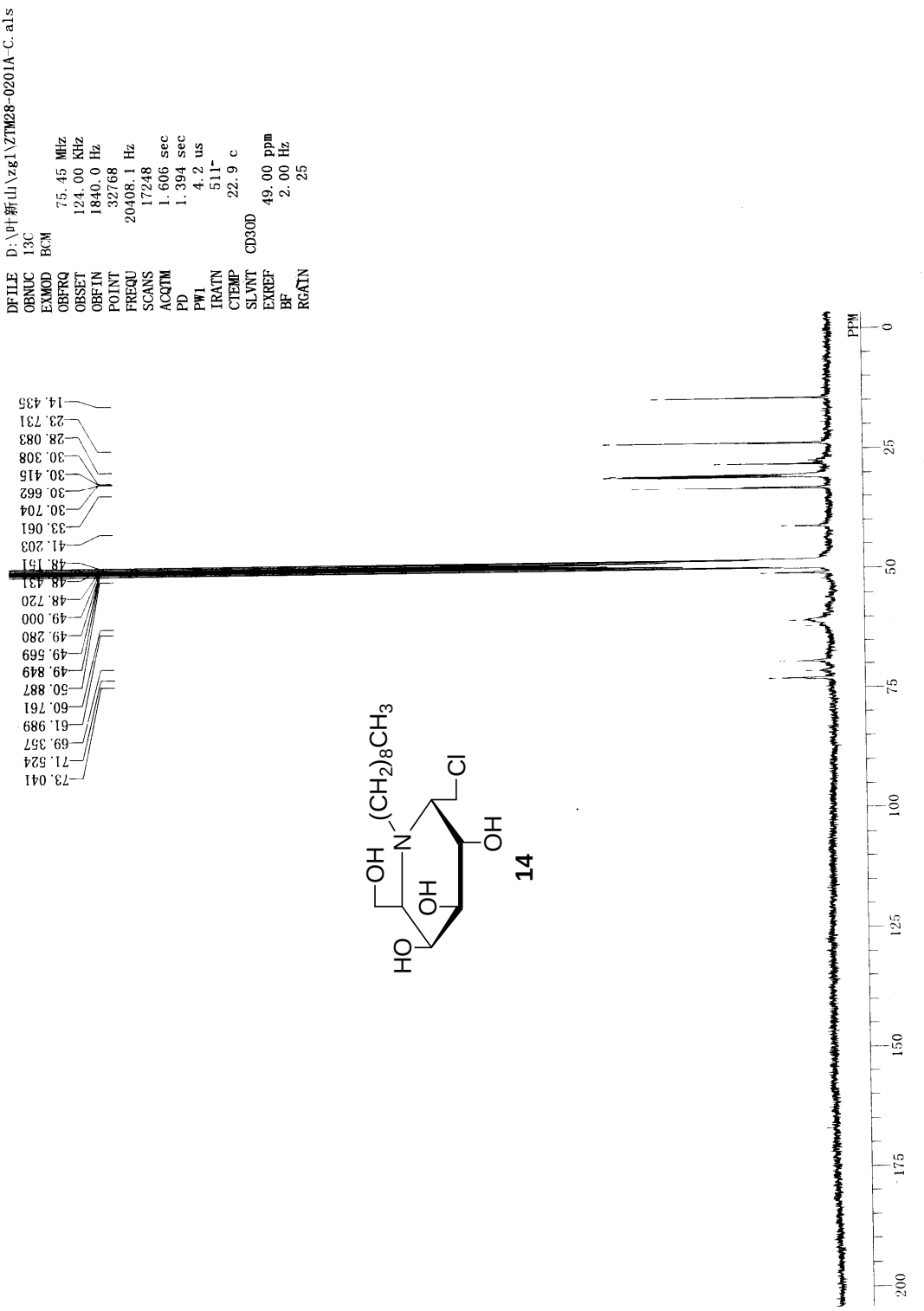

DFILE D:\叶新山\zg1\ZJ1CD-Cy-H-Py.als
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRATN 51
CTEMP 23.3 c
SLVNT C5D5N
EXREF 7.19 ppm
BF 0.12 Hz
RGAIN 14
OBn
(CH2)8CH3
N
Cl
OBn
OBn
BnO
35
PPM

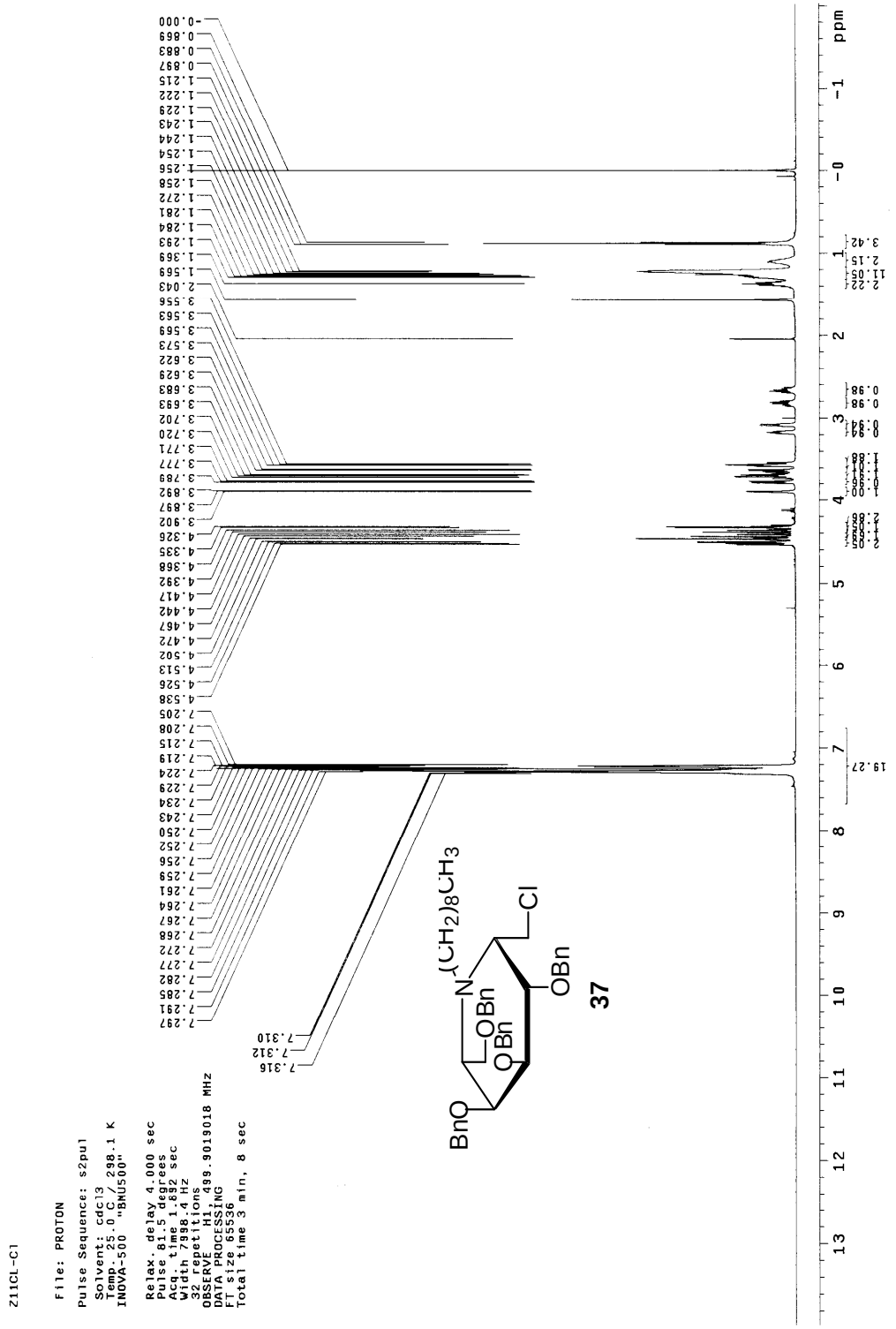

DFILE D:\叶新山\zg1\ZI1CD-CY-Py-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
PO1NT 32768
FREQU 20408.1 Hz
SCANS 1600
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 23.5 c
SLVNT C5D5N
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
13.984
22.630
27.072
29.306
29.660
29.693
30.237
31.803
42.187
50.033
56.956
60.937
68.519
70.942
72.525
72.904
73.159
75.714
80.033
122.856
123.186
123.516
127.604
127.834
128.411
128.461
134.856
135.186
135.516
139.092
139.299
139.620
149.230
149.584
149.947
BnO
OBn
OBn
N
(CH2)8CH3
Cl
OBn
35
PPM
0
50
100
150
200

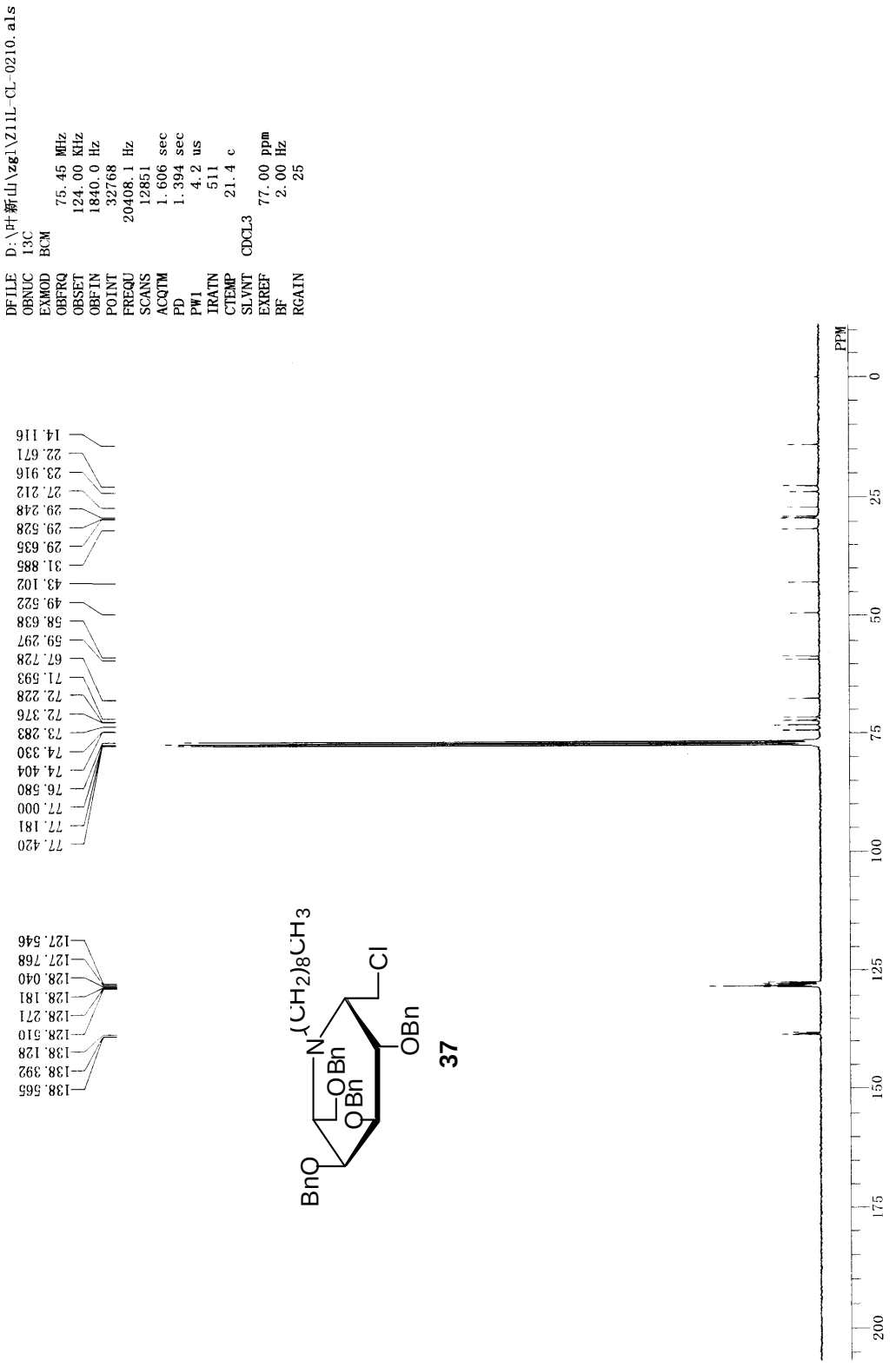

Ztm33-0211
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 83.4 degrees
Acq. time 1.882 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9031222 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
0.870
0.884
0.897
1.280
1.295
1.308
1.336
1.339
1.348
1.372
1.408
1.419
1.823
3.491
3.505
3.520
3.826
3.906
4.053
4.061
4.078
4.086
4.115
4.130
4.143
4.156
4.170
4.322
4.772
3.00
6.50
2.14
2.18
2.18
5.31
2.15
ppm
OH
N
(CH2)5CH3
Cl
OH
OH
HO
13

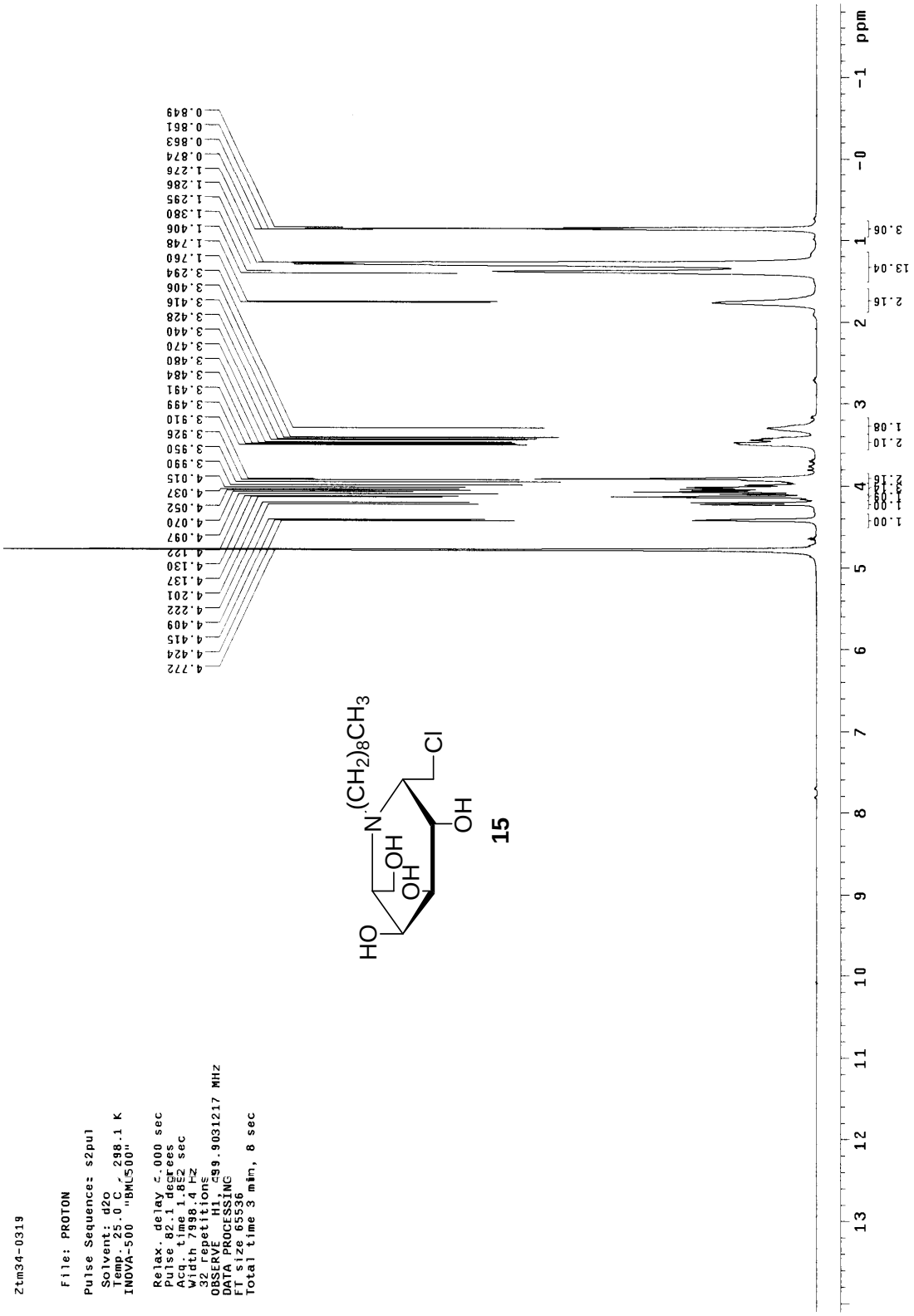

BRUKER
NAME Ztm33-0211
EXPNO 21
PROCNO 1
Date_ 20090220
Time_ 21.04
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 15488
DS 0
SWH 28409.092 Hz
FIDRES 0.433488 Hz
AQ 1.1534836 sec
RG 2050
DW 17.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.36 usec
PL1 0.00 dB
PL1W 35.66878891 W
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 12.54 dB
PL13 13.05 dB
PL2W 9.46981144 W
PL12W 0.52764440 W
PL13W 0.46918198 W
SFO2 400.1516006 MHz
SI 32768
SF 100.6175219 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
14.62
15.97
24.46
27.66
27.90
33.15
37.74
40.84
54.11
59.27
64.52
68.31
71.55
0 ppm
20
40
60
80
100
120
140
160
180
200
OH
(CH2)5CH3
N
Cl
OH
OH
HO
13

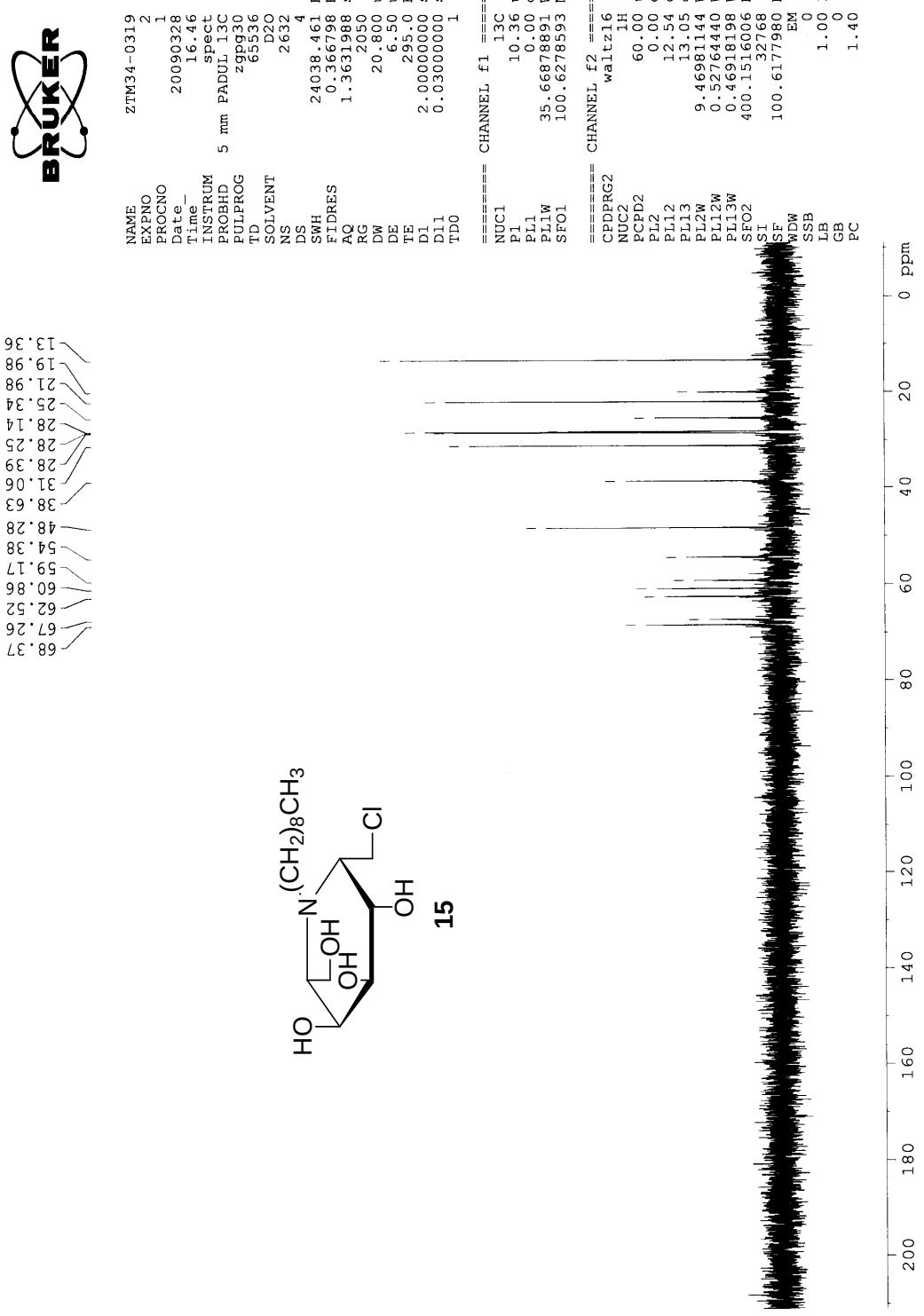

Ztm28-0223
File: PROTON
Pulse Sequence: s2pul
Solvent: cd3od
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.4 degrees
Acq. time 1.882 sec
Width 7988.4 HZ
32 repetitions
OBSERVE H1 499.9038719 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
0.876
0.890
0.905
0.981
0.994
1.290
1.315
1.329
1.343
1.362
1.370
1.386
1.405
1.416
1.422
1.432
1.448
1.464
2.711
2.727
2.740
2.757
2.770
2.892
3.294
3.297
3.300
3.303
3.306
3.314
3.323
3.332
3.340
3.513
3.523
3.695
3.711
3.717
3.734
3.795
3.806
3.815
3.829
3.838
3.987
3.997
4.005
4.014
4.023
4.028
4.841
15.18
0.29
3.17
2.07
1.00
1.17
1.05
1.14
3.13
2.22
ppm
OH
N
(CH2)8CH3
Cl
OH
OH
HO
14

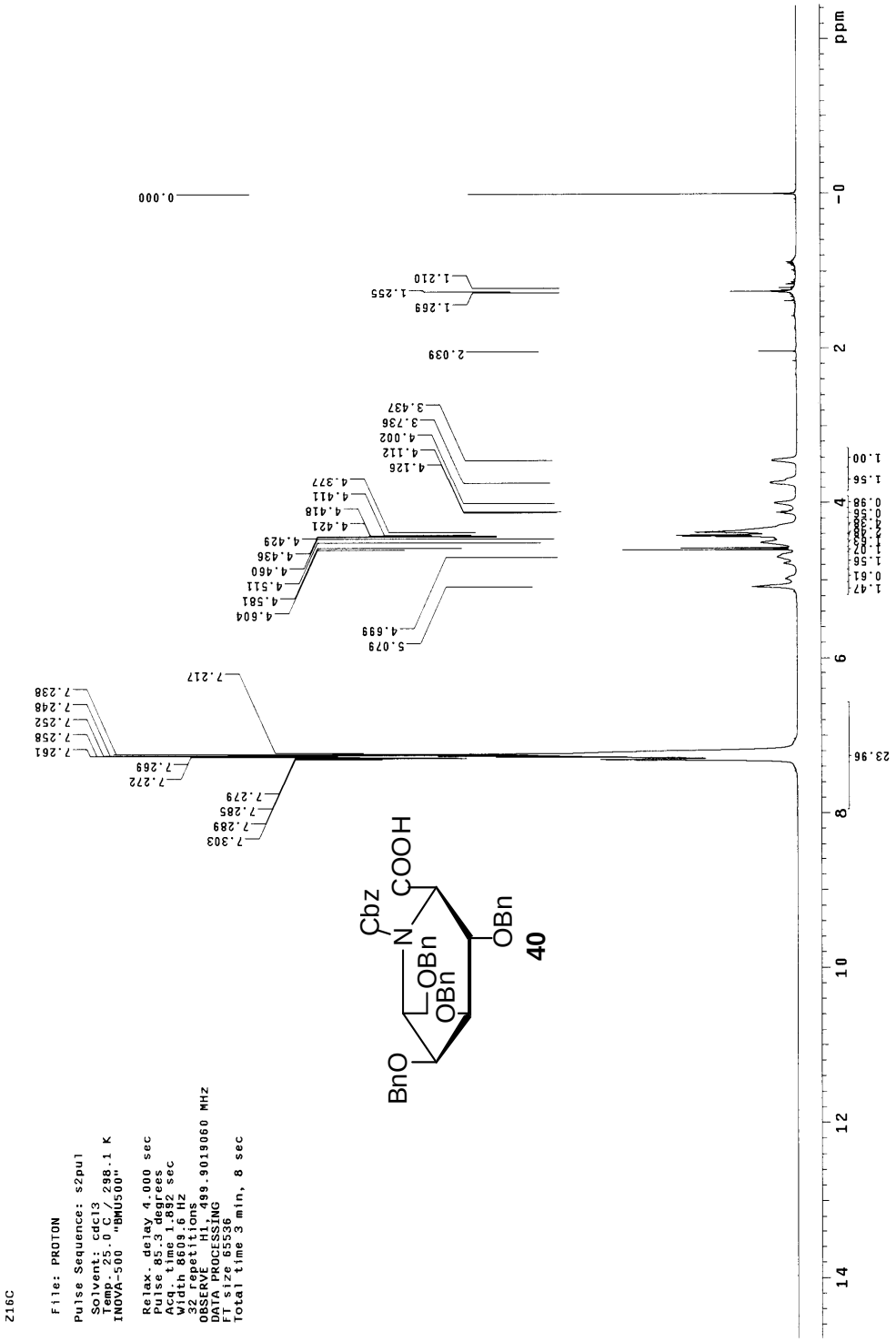

DFILE D:\叶新山\zg1\ZTM28-0201A-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 17248
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 22.9 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 2.00 Hz
RGAIN 25
14.435
23.731
28.083
30.308
30.415
30.662
30.704
33.061
41.203
48.151
48.431
48.720
49.000
49.280
49.569
49.849
50.887
60.761
61.989
69.357
71.524
73.041
PPM
0
25
50
75
100
125
150
175
200
OH
(CH2)8CH3
N
Cl
OH
OH
HO
14

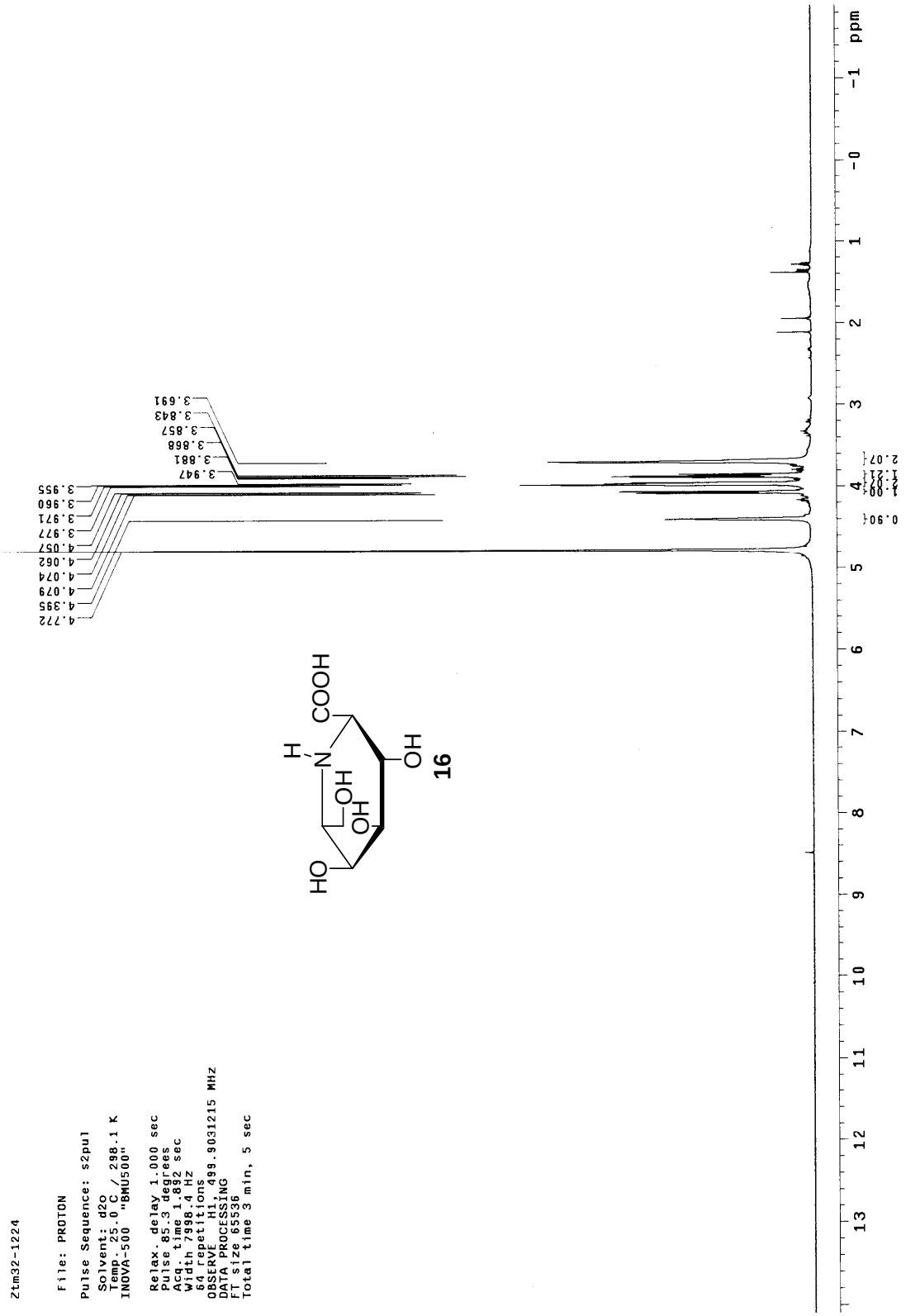

Z11CL-Cl
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 / 298.1 K
INOVA-500 "BNU500"
Relax. delay 4.000 sec
Pulse 81.5 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9019018 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
7.316
7.312
7.310
BnO
OBn
OBn
N
(CH2)8CH3
Cl
OBn
37
ppm

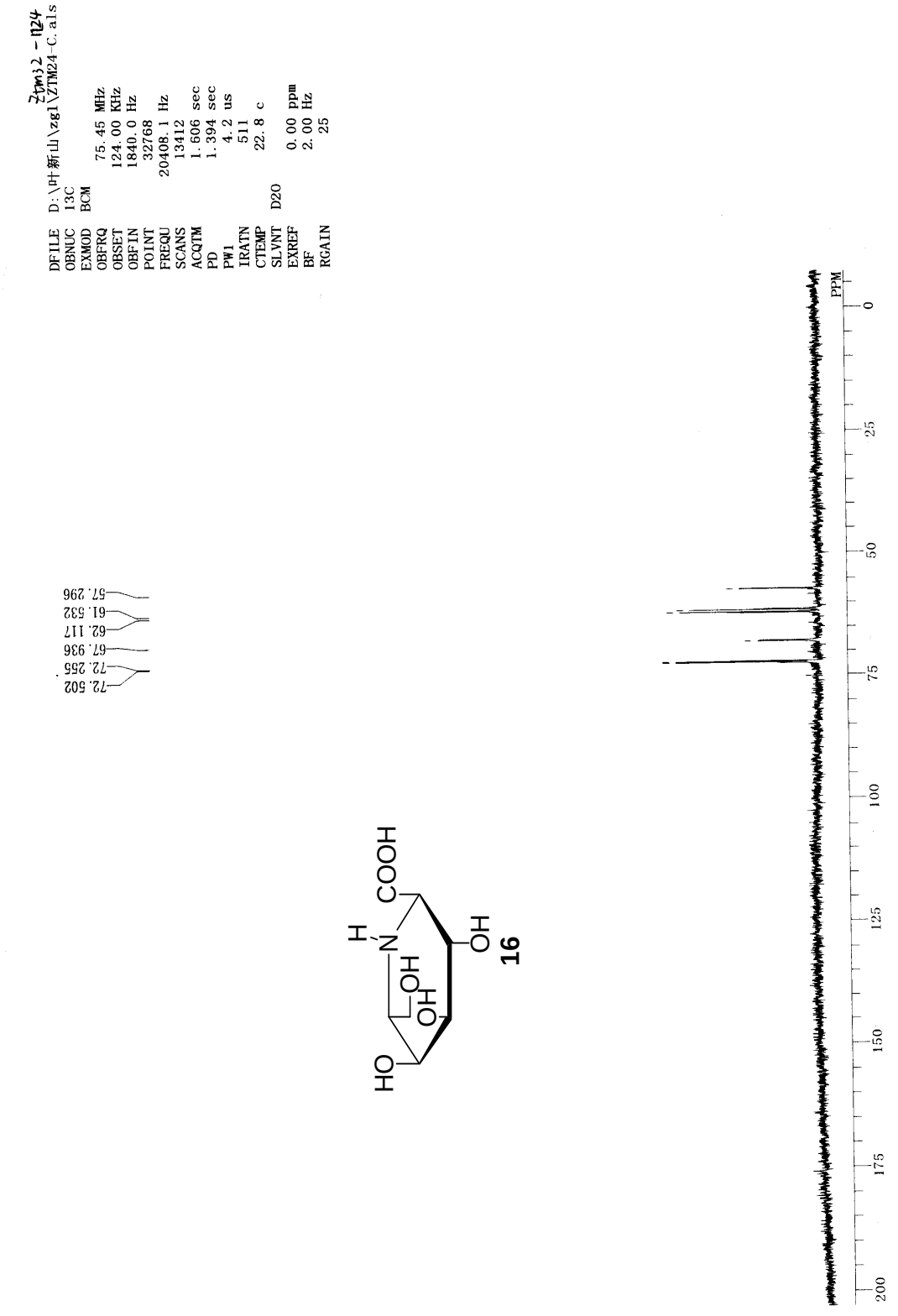

DFILE D:\叶新山\zg1\ZI1L-CL-0210.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 12851
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
14.116
22.671
23.916
27.212
29.248
29.528
29.635
31.885
43.102
49.522
58.638
59.297
67.728
71.593
72.228
72.376
73.283
74.330
74.404
76.580
77.000
77.181
77.420
127.546
127.768
128.040
128.181
128.271
128.510
138.128
138.392
138.565
PPM
0
25
50
75
100
125
150
175
200
BnO
OBn
OBn
N
(CH2)8CH3
Cl
OBn
37


2tm34-0319
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp 25.0 C / 298.1 K
INOVA-500 "BMC500"
Relax. delay 5.000 sec
Pulse 82.1 degrees
Acq. time 1.852 sec
Width 7998.4 Hz
32 repetitions
OBSERVE H1, 499.9031217 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
4.772
4.424
4.415
4.409
4.222
4.201
4.137
4.130
4.122
4.097
4.070
4.052
4.037
4.015
3.990
3.950
3.926
3.910
3.499
3.491
3.484
3.480
3.470
3.440
3.428
3.416
3.406
3.294
1.760
1.748
1.406
1.380
1.295
1.286
1.276
0.874
0.863
0.861
0.849
3.06
13.04
2.16
1.08
2.10
2.16
1.00
ppm
HO
OH
OH
N
(CH2)8CH3
Cl
OH
15


BRUKER
NAME ZTM34-0319
EXPNO 2
PROCNO 1
Date_ 20090328
Time 16.46
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 2632
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.36 usec
PL1 0.00 dB
PL1W 35.66878891 W
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 12.54 dB
PL13 13.05 dB
PL2W 9.46981144 W
PL12W 0.52764440 W
PL13W 0.46918198 W
SFO2 400.1516006 MHz
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.36
19.98
21.98
25.34
28.14
28.25
28.39
31.06
38.63
48.28
54.38
59.17
60.86
62.52
67.26
68.37
200 180 160 140 120 100 80 60 40 20 0 ppm
HO
OH
OH
N
(CH2)8CH3
Cl
OH
15


Z16C
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 8609.6 Hz
32 repetitions
OBSERVE H1, 499.9019060 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
0.000
1.210
1.255
1.269
2.039
3.437
3.736
4.002
4.112
4.126
4.377
4.411
4.418
4.421
4.429
4.436
4.460
4.511
4.581
4.604
5.079
4.699
7.217
7.238
7.248
7.252
7.258
7.261
7.269
7.272
7.279
7.285
7.289
7.303
Cbz
N
COOH
BnO
OBn
OBn
OBn
40
ppm


Ztm32-1224
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 7998.4 Hz
64 repetitions
OBSERVE H1, 499.9031215 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 5 sec
4.772
4.395
4.079
4.074
4.062
4.057
3.977
3.971
3.960
3.955
3.947
3.881
3.868
3.857
3.843
3.691
2.07
1.21
1.00
0.90
ppm
16


Ztm32 -124
DATA D:\中新山\zg1\ZTM24-C.als
DFILE D:\中新山\zg1\ZTM24-C.als
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 13412
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRATN 511
CTEMP 22.8 c
SLVNT D2O
EXREF 0.00 ppm
BF 2.00 Hz
RGAIN 25
72.502
72.255
67.936
62.117
61.532
57.296
HO
OH
OH
OH
H
N
COOH
16
PPM
0
25
50
75
100
125
150
175
200