Electronic Supplementary Information

Concise syntheses of selective inhibitors against α-1,3-galactosyltransferase

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COSY spectrum of compound **20**……S25

HSQC spectrum of compound **20**……S26

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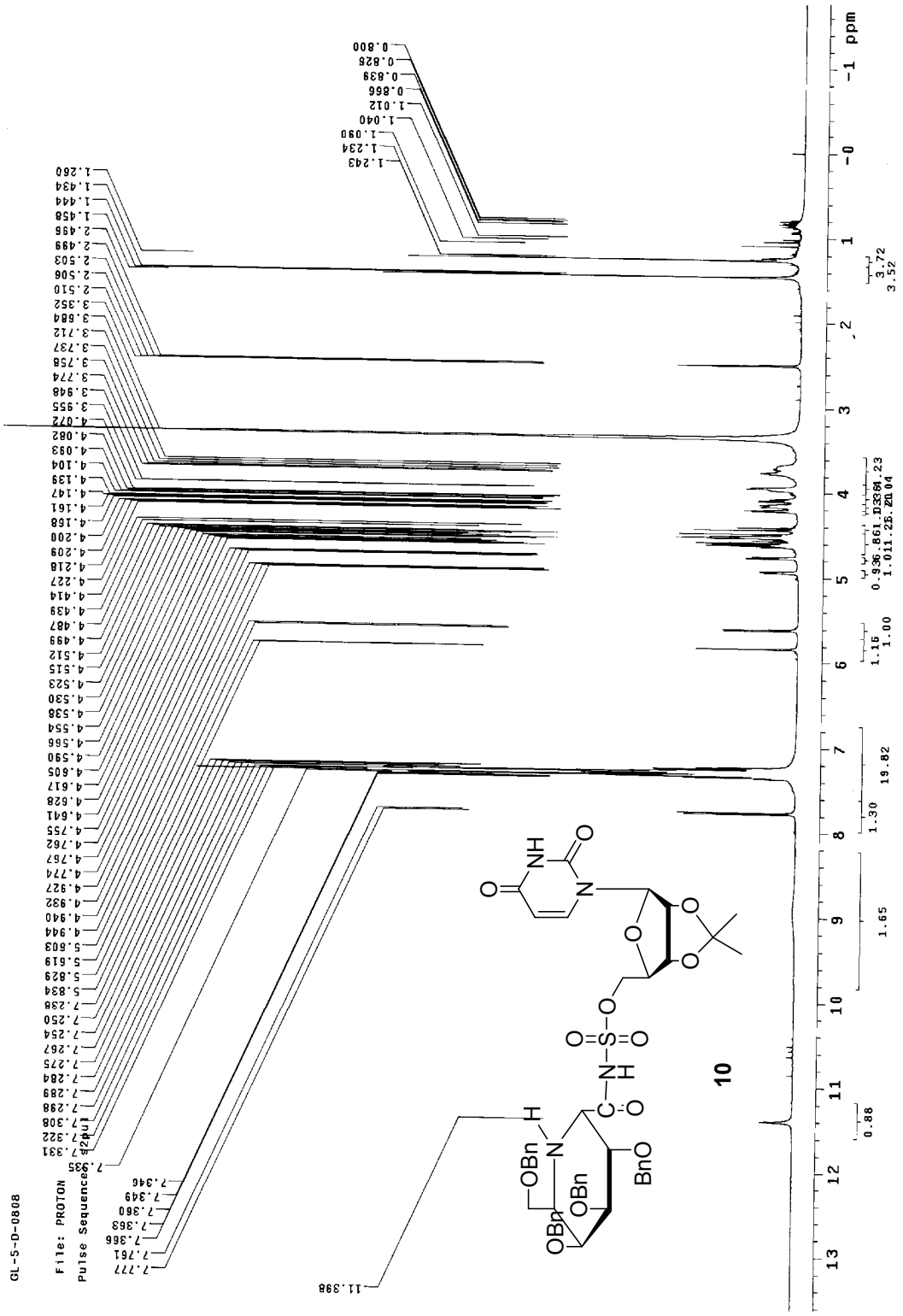
GL-5-D-0808
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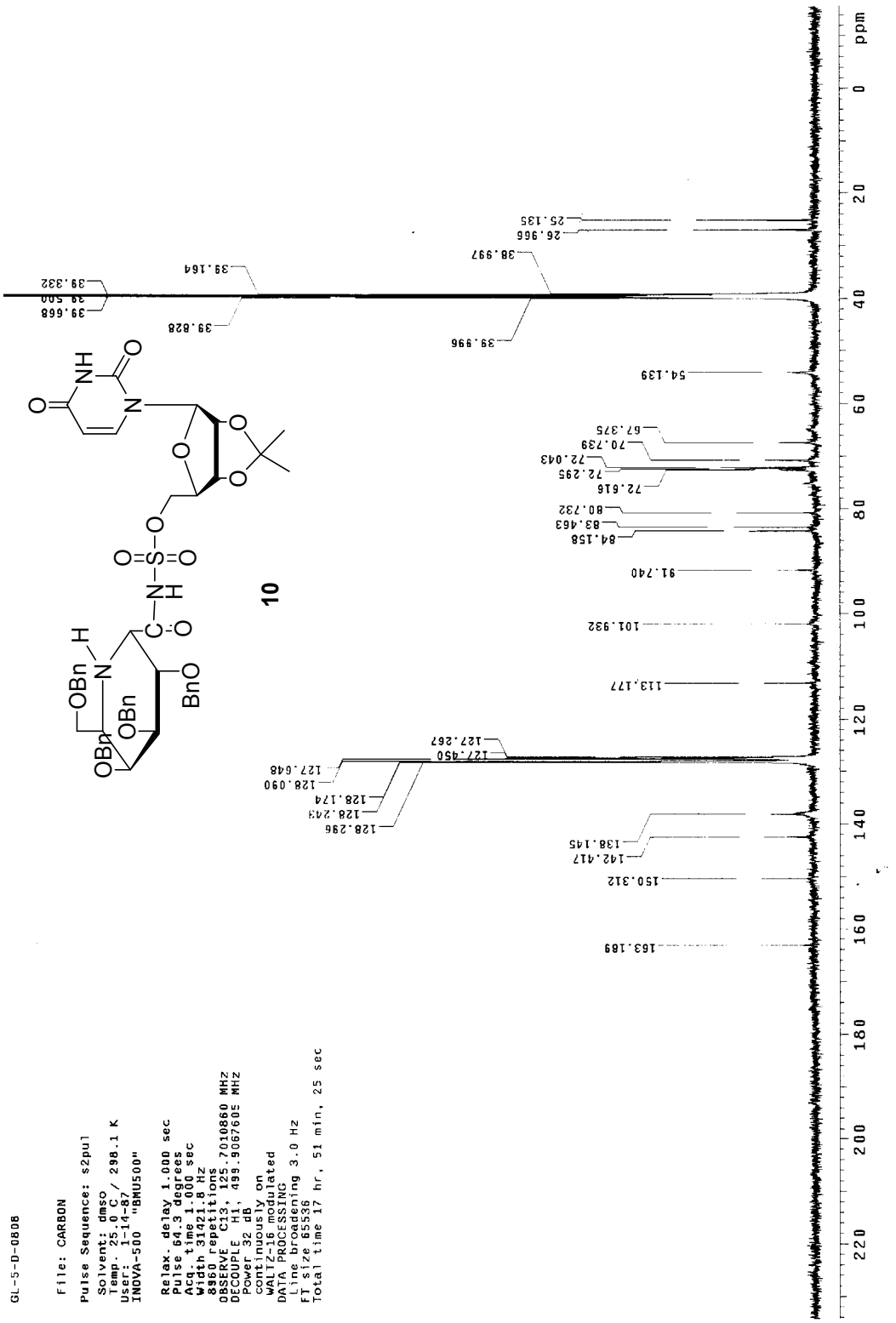
Pulse Sequence: s2pul
10
ppm


GL-5-D-0808
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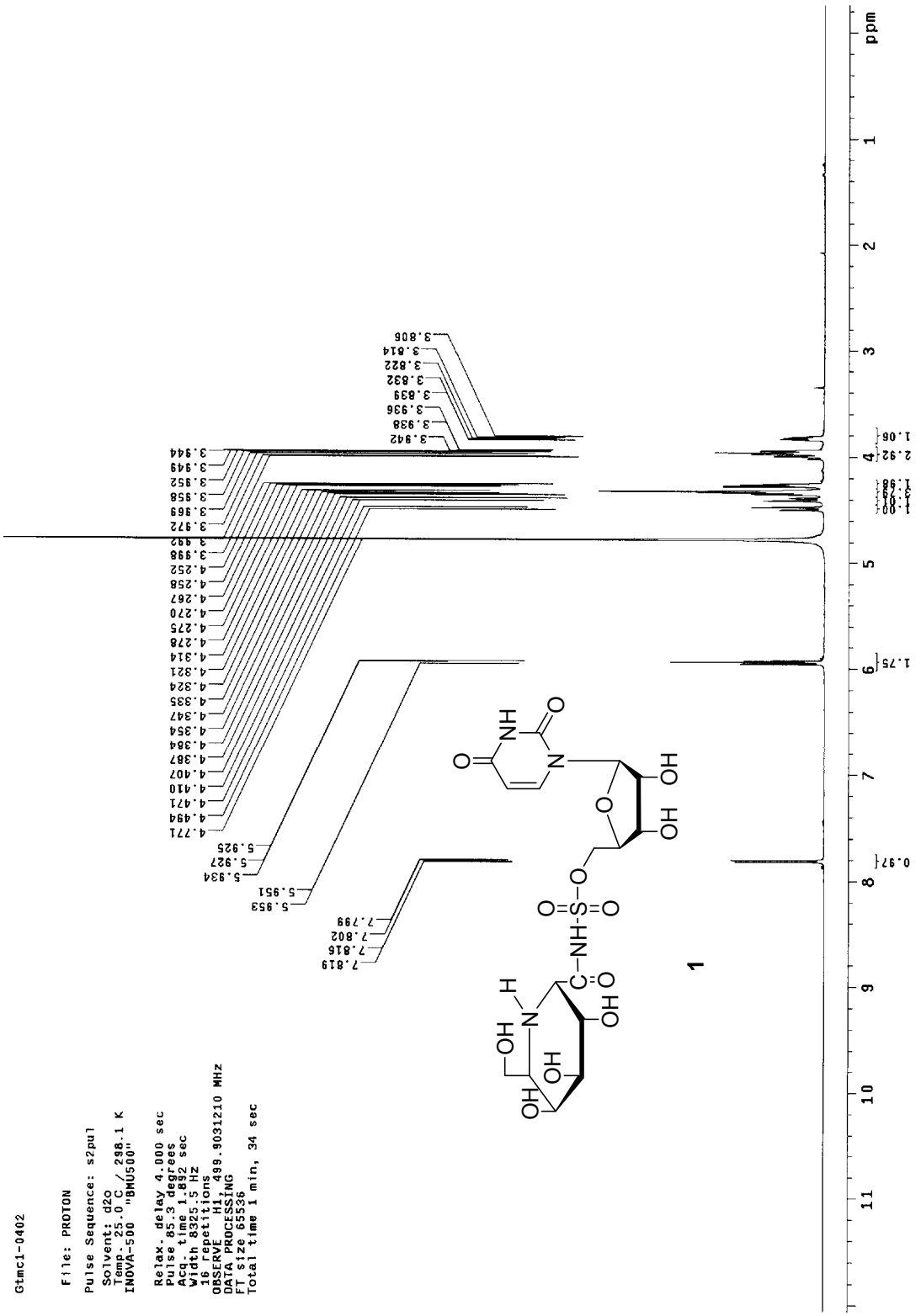
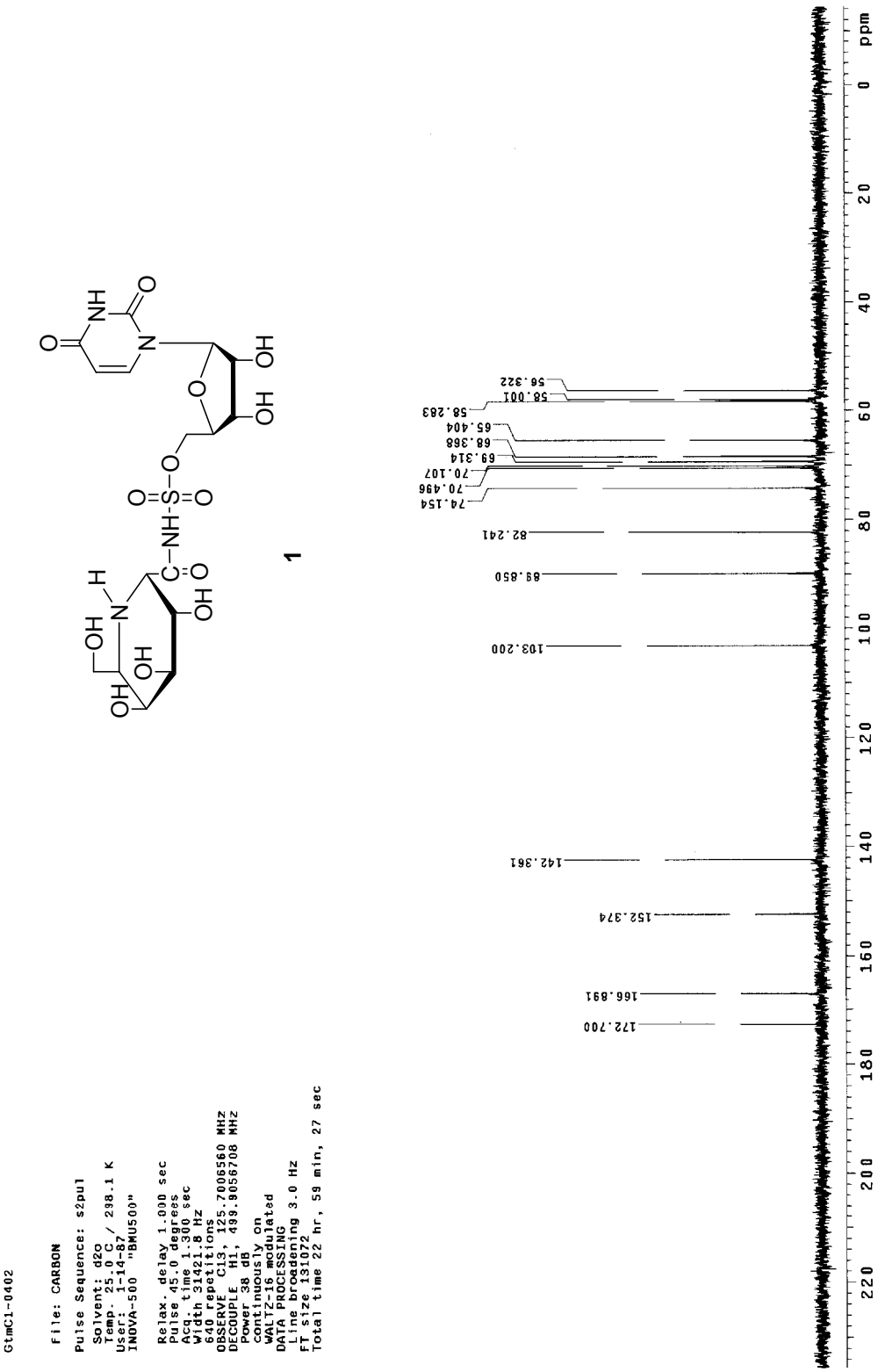
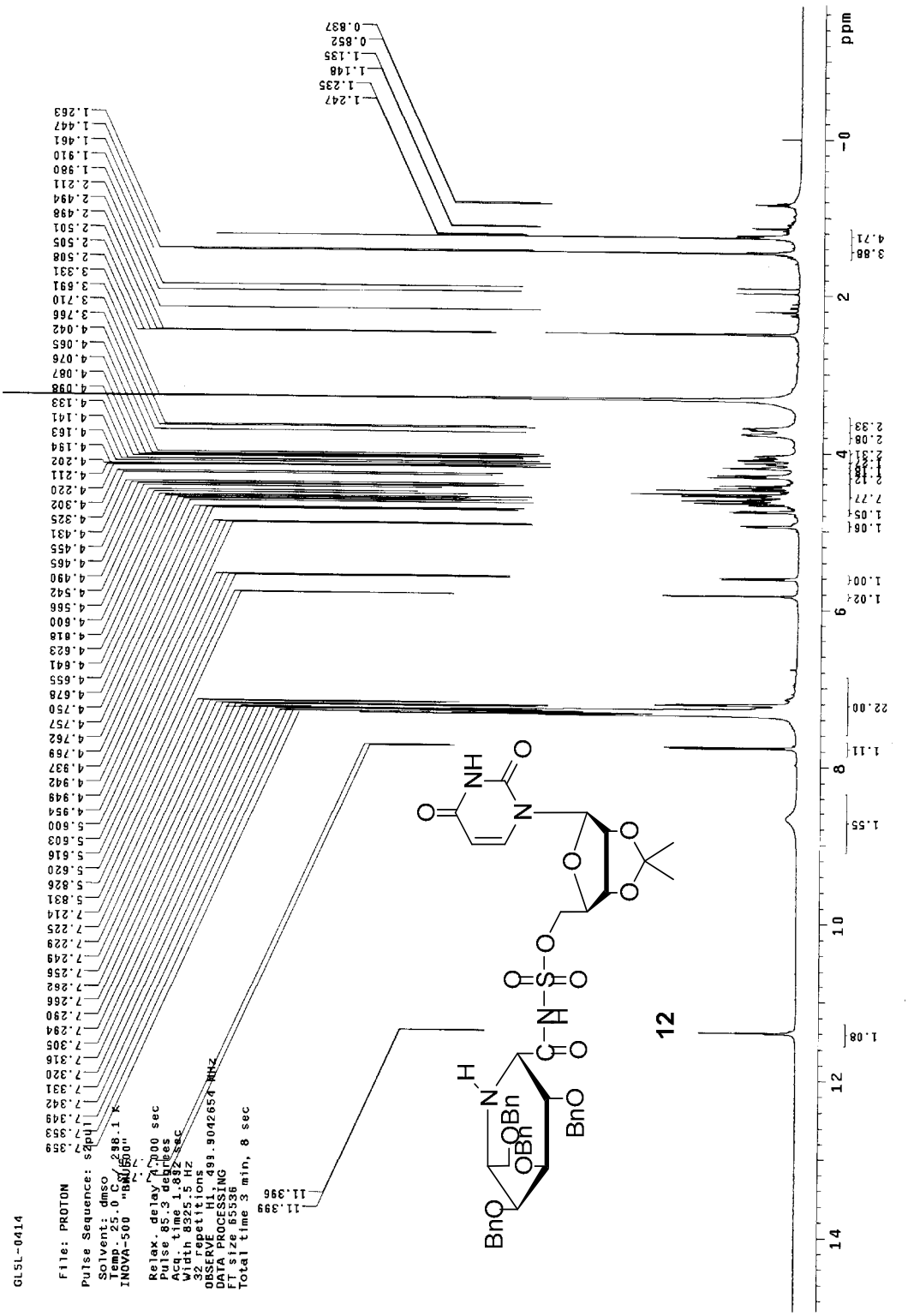
Pulse Sequence: s2pul
Solvent: dmso
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
8960 repetitions
OBSERVE C13, 125.7010860 MHz
DECOUPLE H1, 499.9067605 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 17 hr, 51 min, 25 sec
10
ppm


Gtmc1-0402
File: PROTON
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 8325.5 Hz
16 repetitions
OBSERVE H1, 499.9031210 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
ppm
1
1.06
2.92
1.98
1.75
0.97


GtmC1-0402
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
640 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 22 hr, 59 min, 27 sec
1
74.154
70.496
70.107
69.314
68.388
65.404
58.283
58.001
56.322
82.241
89.850
103.200
142.361
152.374
166.891
172.700
ppm


GLSL-0414
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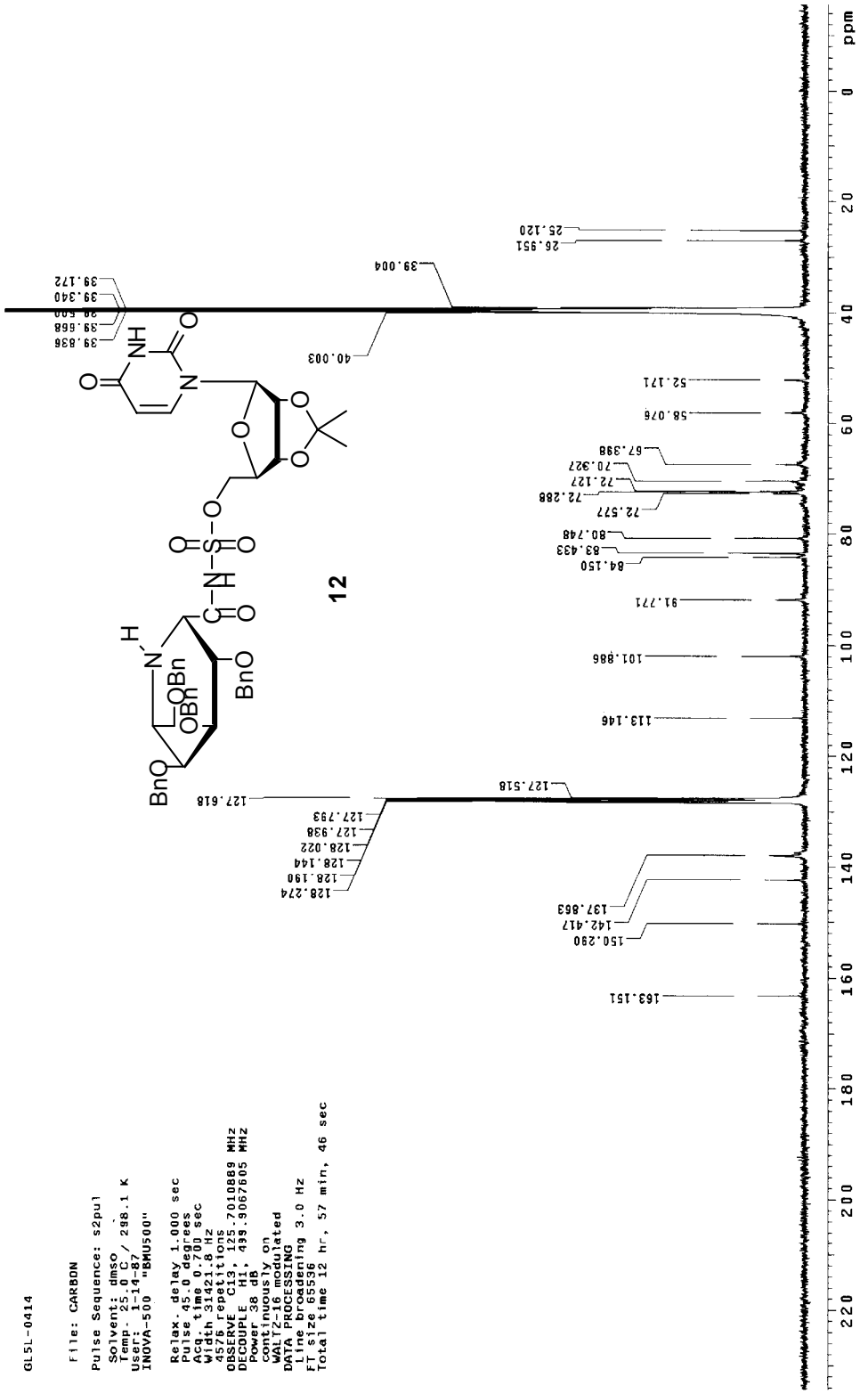
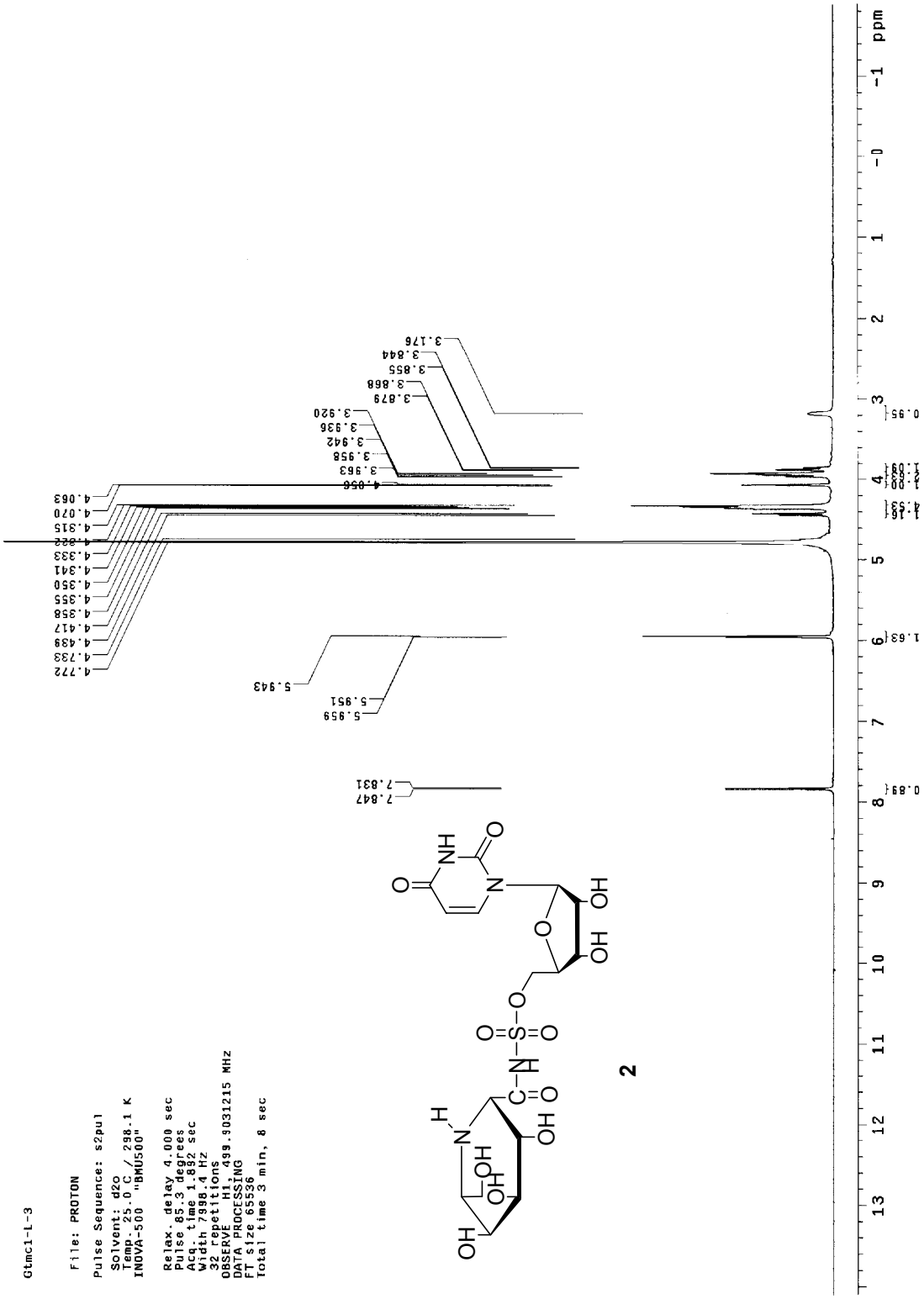
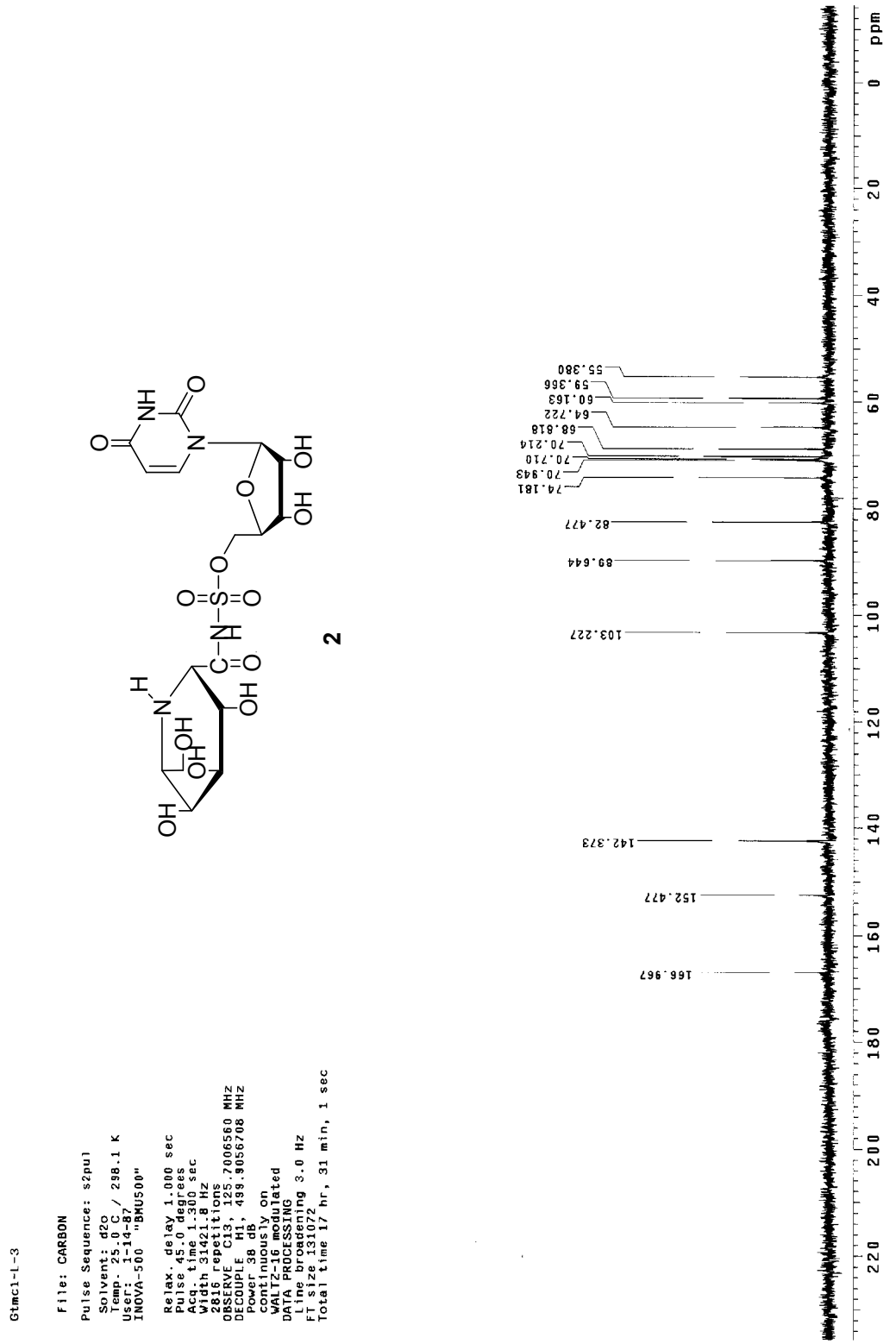
Pulse Sequence: s2pul
Solvent: dmso
Temp. 25.0 C / 298.1 K
INOVA-500 "BMS500"
Relax. delay 7.000 sec
Pulse 85.3 degrees
Acq. time 1.882 sec
Width 8325.5 Hz
32 repetitions
OBSERVE H1, 499.9042654 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
12


12
GL5L-0414
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 0.700 sec
Width 31421.8 Hz
4576 repetitions
OBSERVE C13, 125.7010889 MHz
DECOUPLE H1, 499.9067605 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 12 hr, 57 min, 46 sec
ppm


Gtmc1-L-3
File: PROTON
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, 499.9031215 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
2


Gtmc1-1-3
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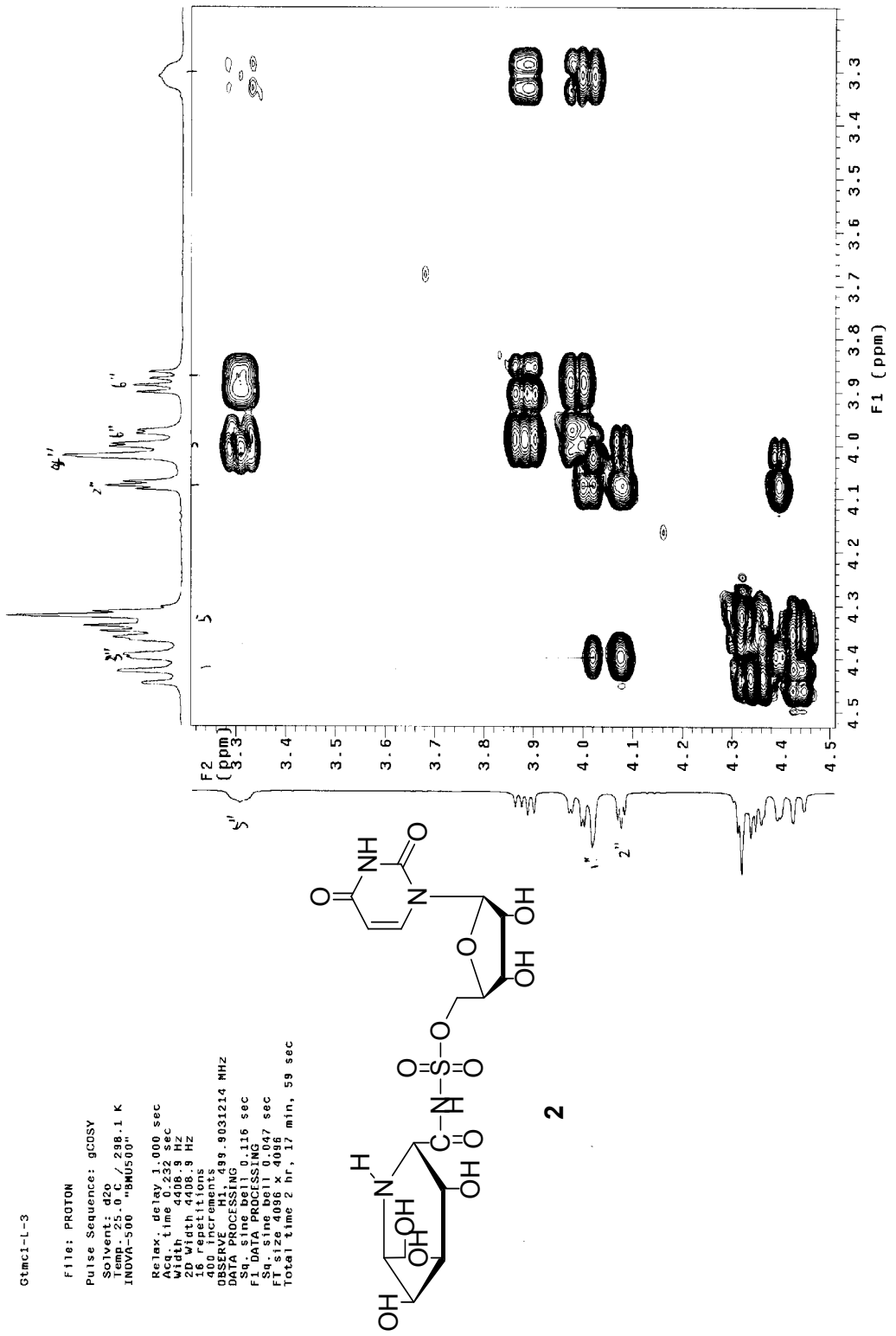
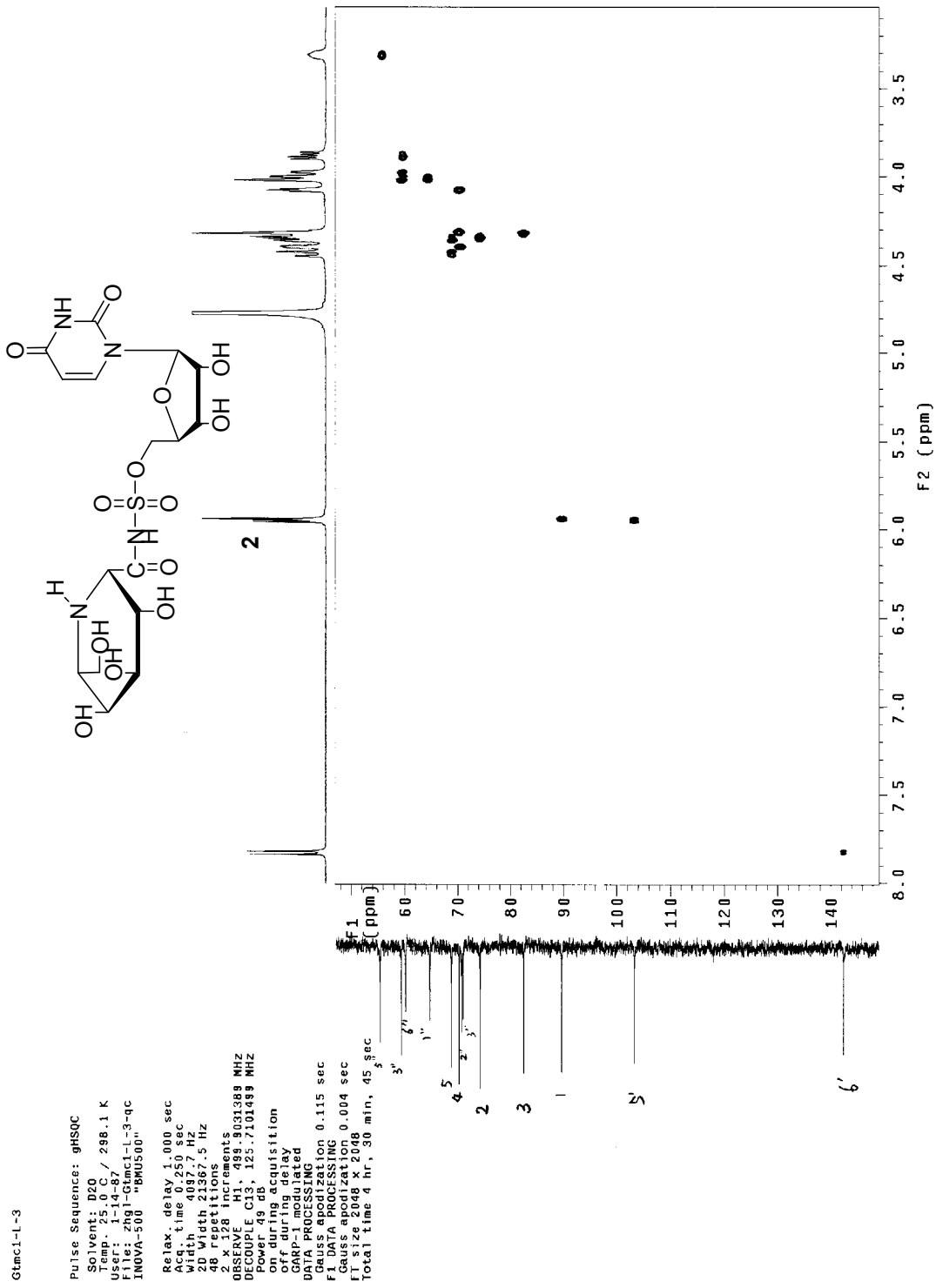
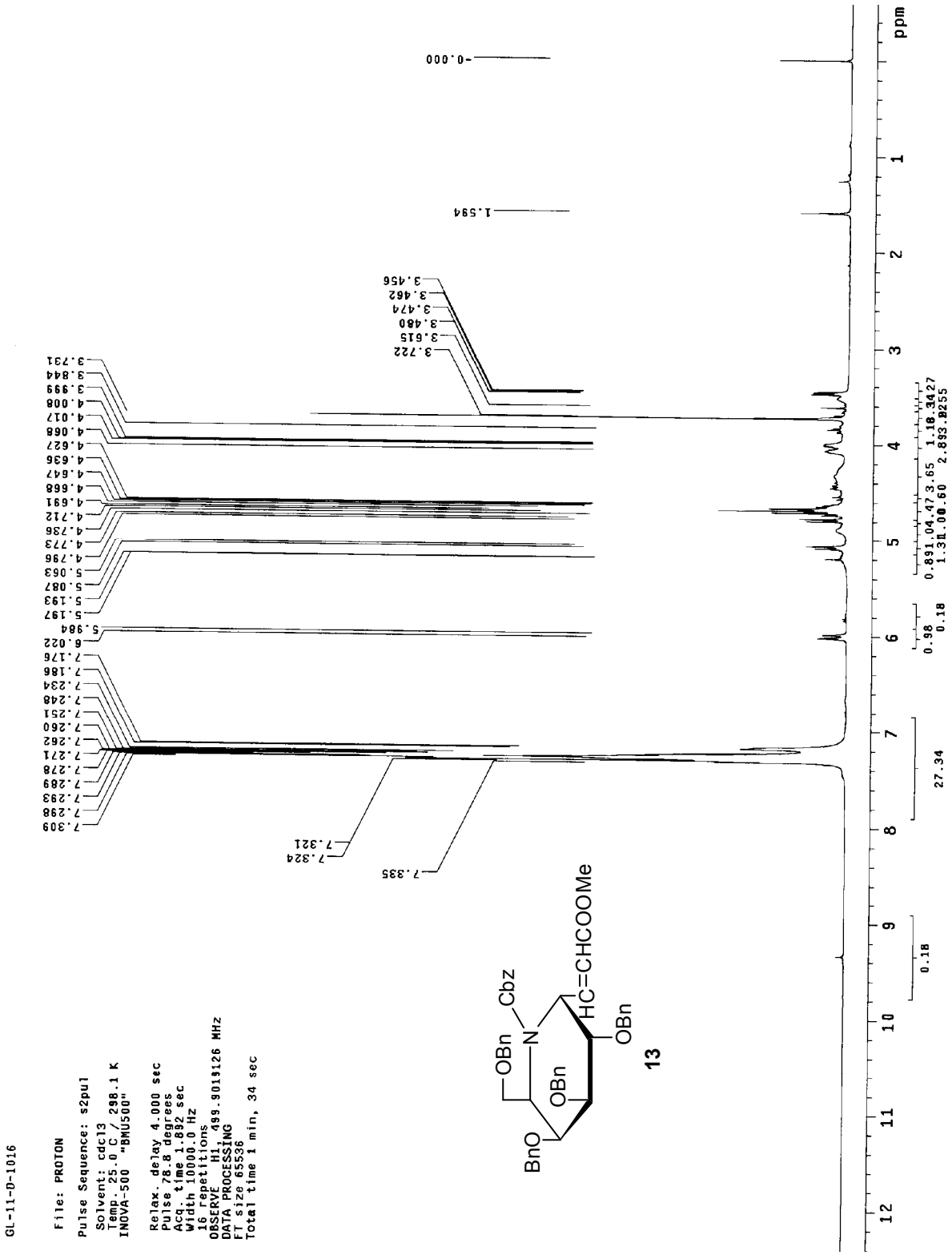
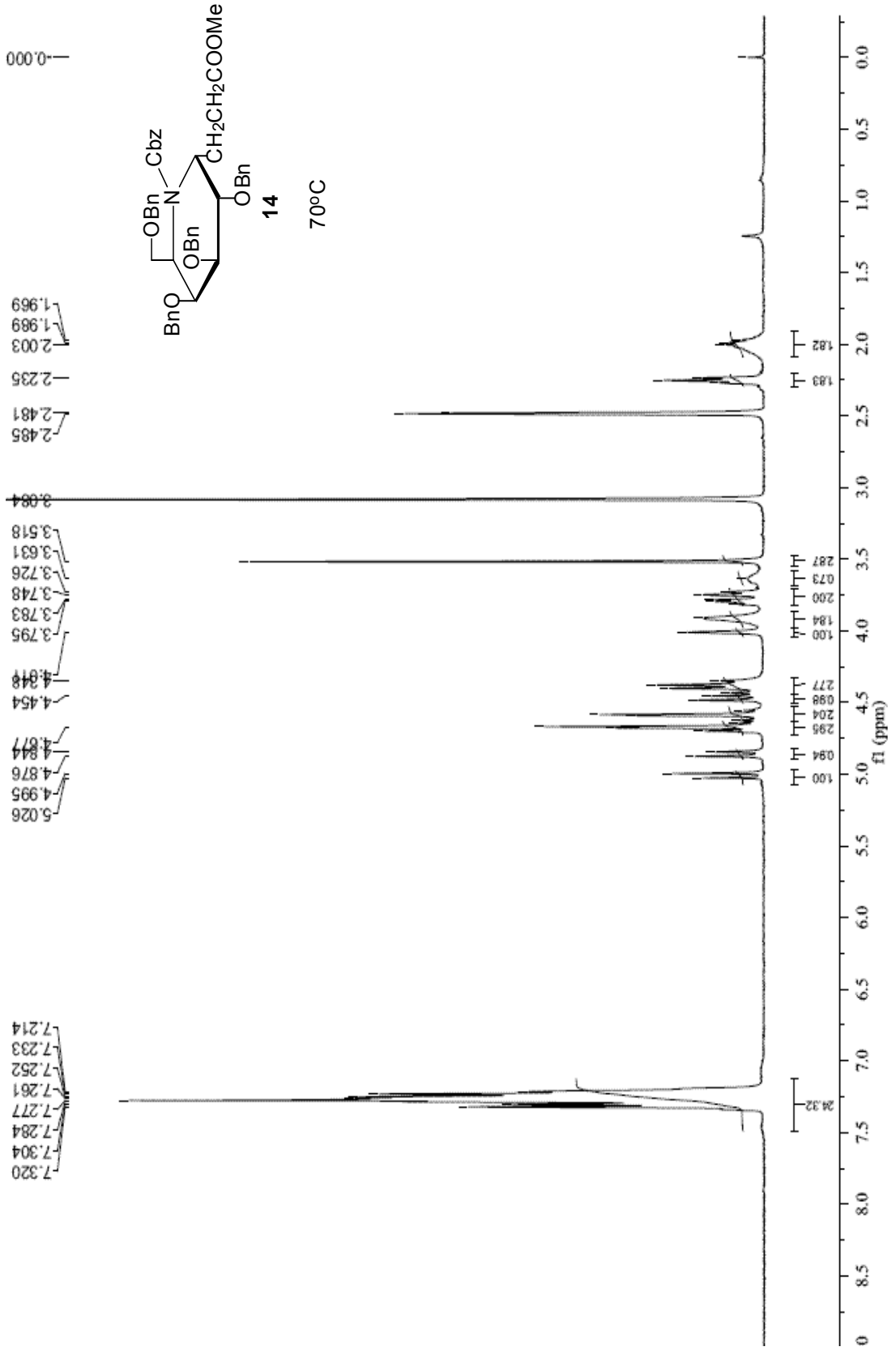
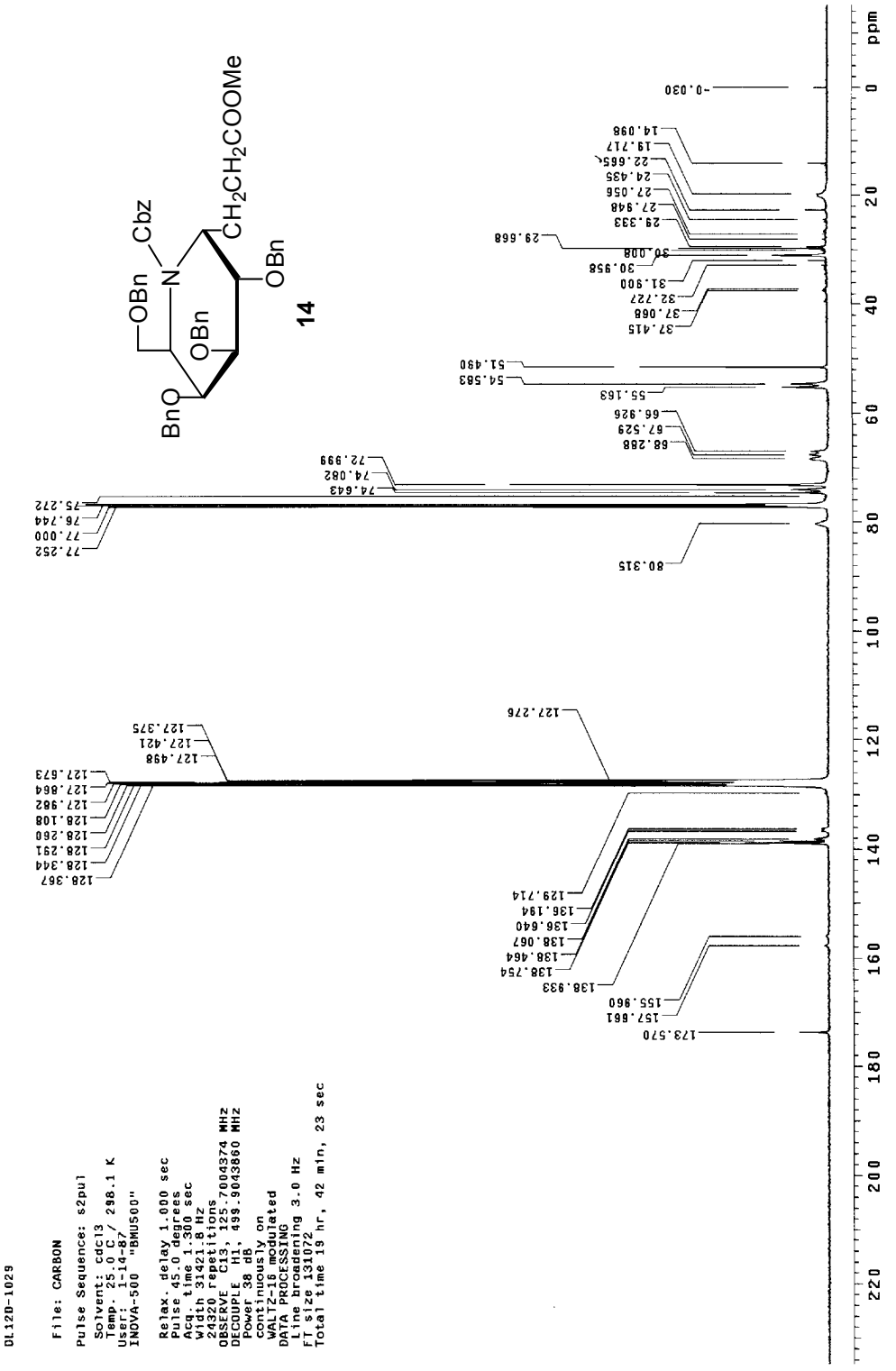
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
2816 repetitions
OBSERVE C13, 125.7006560 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 17 hr, 31 min, 1 sec
2
55.380
59.366
60.163
64.722
68.818
70.214
70.710
70.943
74.181
82.477
89.644
103.227
142.373
152.477
166.967
220
200
180
160
140
120
100
80
60
40
20
0
ppm

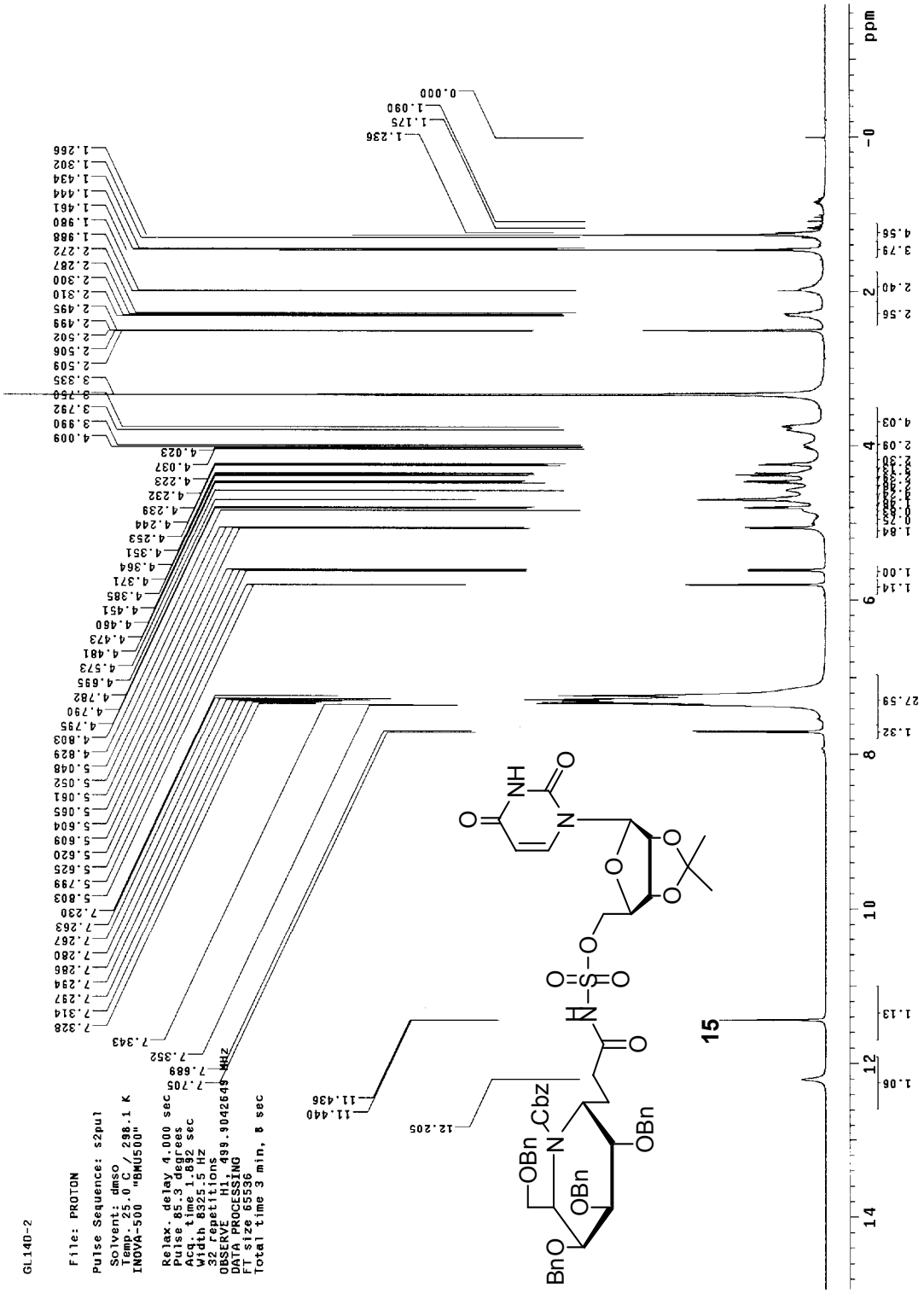

Gtmc1-L-3
File: PROTON
Pulse Sequence: gCOSY
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.232 sec
Width 4406.3 Hz
2D Width 4408.9 Hz
16 repetitions
400 increments
OBSERVE H1, 499.9031214 MHz
DATA PROCESSING
Sq. sine bell 0.116 sec
F1 DATA PROCESSING
Sq. sine bell 0.047 sec
FT size 4096 x 4096
Total time 2 hr, 17 min, 59 sec
F2 (ppm)
F1 (ppm)
2

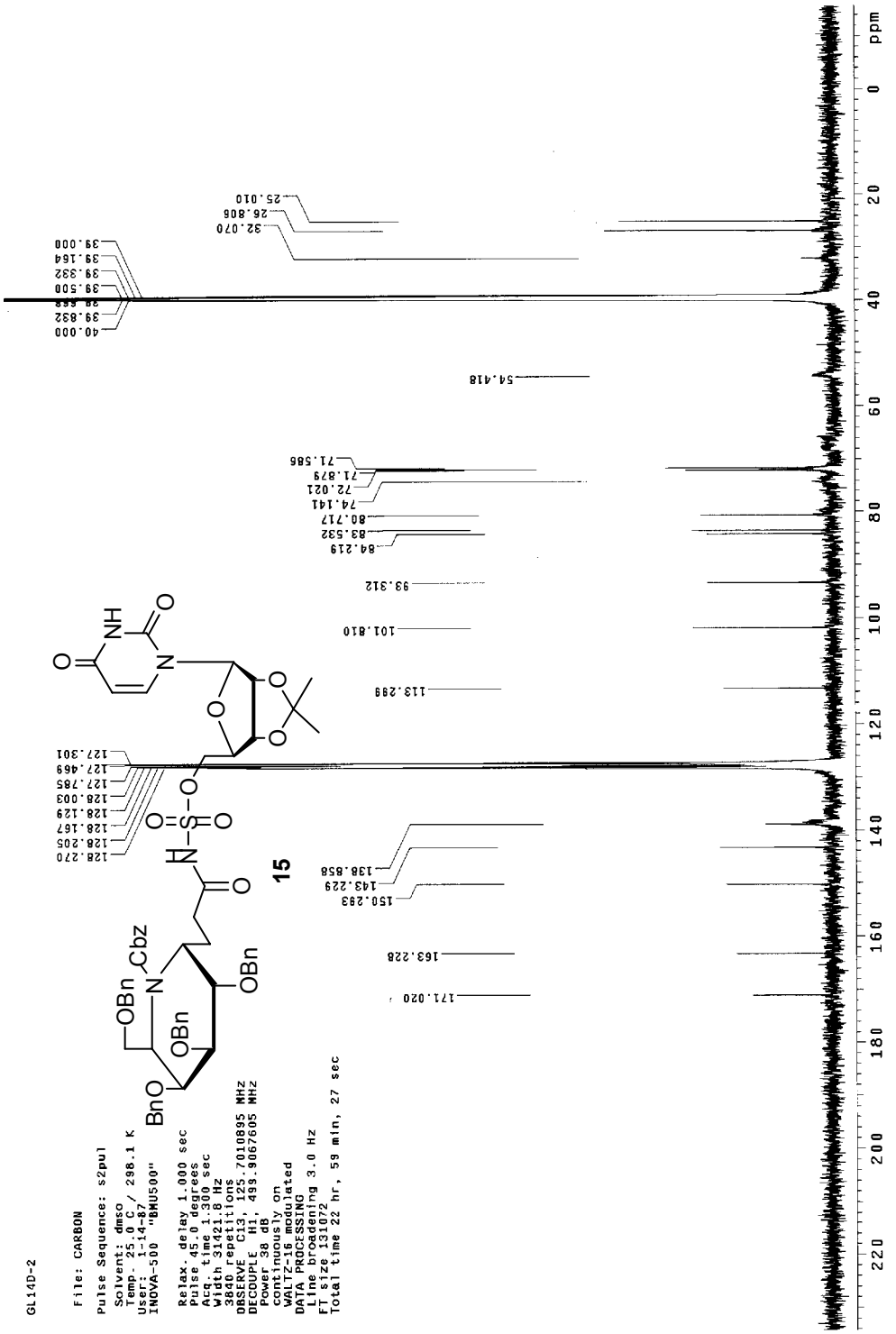

Gtmc1-L-3
Pulse Sequence: gHSQC
Solvent: D2O
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: zhgl-Gtmc1-L-3-qc
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.250 sec
Width 4097.7 Hz
2D Width 21367.5 Hz
48 repetitions
2 x 128 increments
OBSERVE H1, 499.9031389 MHz
DECOUPLE C13, 125.7101499 MHz
Power 49 dB
On during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.115 sec
F1 DATA PROCESSING
Gauss apodization 0.004 sec
FT size 2048 x 2048
Total time 4 hr, 30 min, 45 sec
F2 (ppm)
F1 (ppm)
2


GL-11-D-1016
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 78.8 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
16 repetitions
OBSERVE H1, 499.9019126 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 34 sec
ppm
-0.000
1.594
3.456
3.462
3.474
3.480
3.615
3.722
3.731
3.844
3.999
4.008
4.017
4.068
4.627
4.636
4.647
4.668
4.691
4.712
4.736
4.773
4.796
5.063
5.087
5.193
5.197
5.984
6.022
7.176
7.186
7.234
7.248
7.251
7.260
7.262
7.271
7.278
7.289
7.293
7.298
7.309
7.321
7.324
7.335
OBn
Cbz
N
OBn
BnO
HC=CHCOOMe
OBn
13
0.891 1.04 1.47 3.65 1.18 3.427
1.31 1.00 0.60 2.89 3.255
0.98
0.18
27.34
0.18


Cbz
N
OBn
OBn
BnO
CH2CH2COOMe
OBn
14
70°C
f1 (ppm)


OBn
Cbz
N
CH2CH2COOMe
OBn
OBn
BnO
14
-0.030
14.098
19.717
22.665
24.435
27.056
27.948
29.333
29.668
30.008
30.958
31.900
32.727
37.068
37.415
51.490
54.583
55.163
66.926
67.529
68.288
72.999
74.082
74.643
75.272
76.744
77.000
77.252
80.315
127.276
127.375
127.421
127.498
127.673
127.864
127.982
128.108
128.260
128.291
128.344
128.367
129.714
136.194
136.640
138.067
138.464
138.754
138.983
155.960
157.861
173.570
ppm
0
20
40
60
80
100
120
140
160
180
200
220
DL12D-1029
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
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


GL140-2
File: PROTON
Pulse Sequence: s2pul
Solvent: dmso
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 8325.5 Hz
32 repetitions
OBSERVE H1, 499.9042649 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
15
Cbz
OBn
BnO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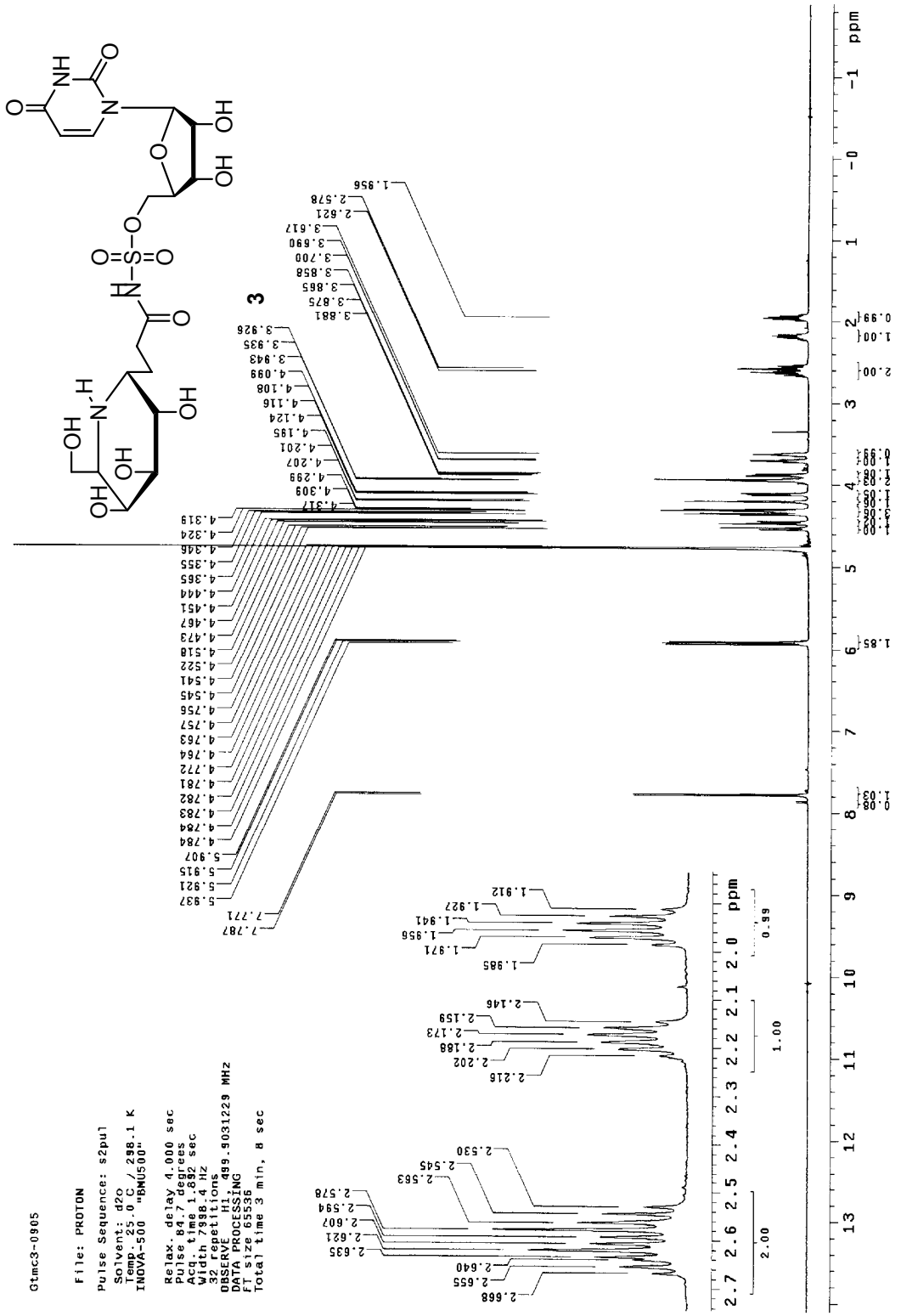
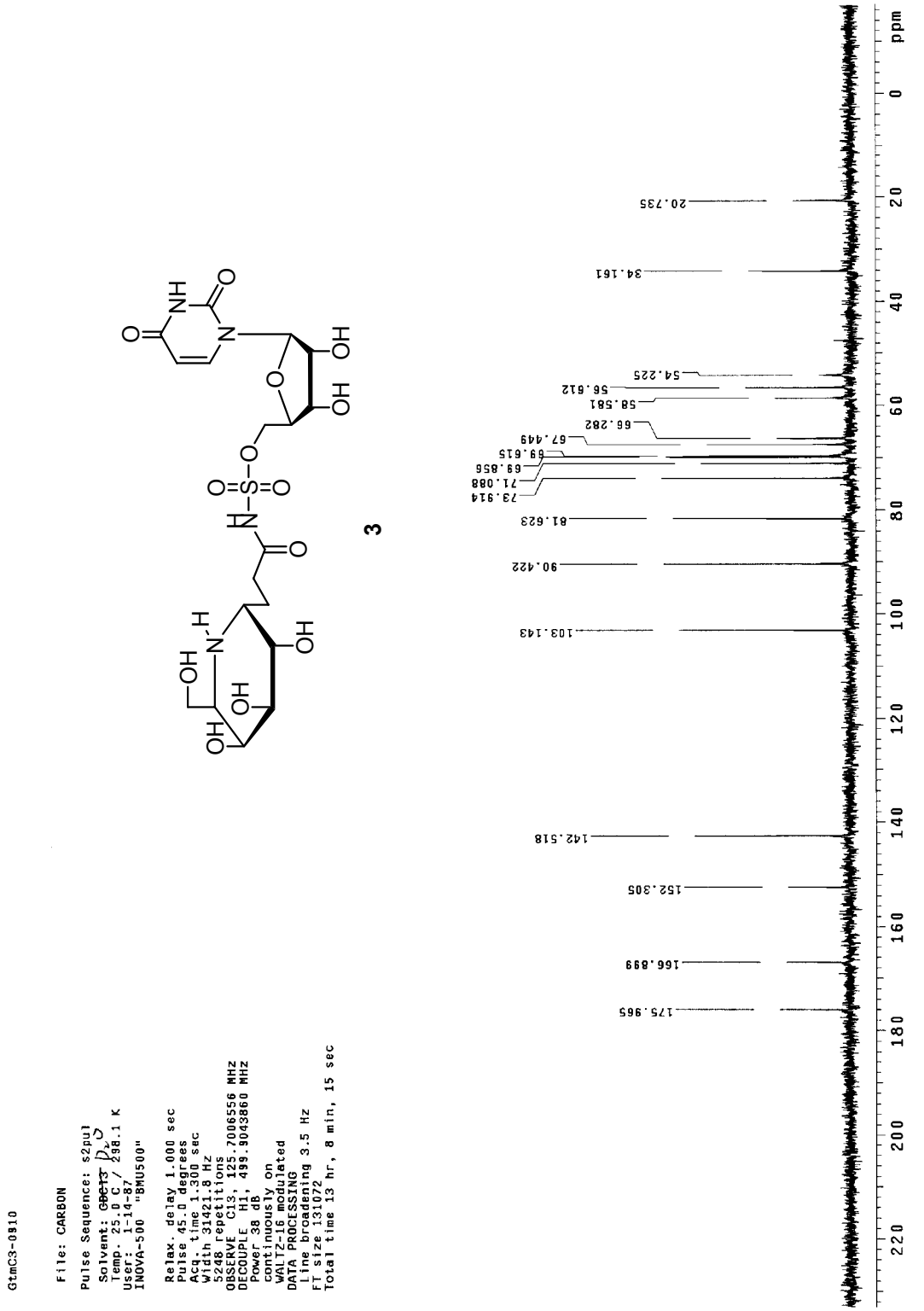
Pulse Sequence: s2pul
Solvent: dmso
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3840 repetitions
OBSERVE C13, 125.7010895 MHz
DECOUPLE H1, 499.9067605 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 22 hr, 59 min, 27 sec
15
ppm


3
Gtmc3-0905
File: PROTON
Pulse Sequence: s2pul
Solvent: d2o
Temp: 25.0 / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 84.7 degrees
Acq. time 1.882 sec
Width 7936.4 Hz
32 repetitions
OBSERVE H1, 499.9031229 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm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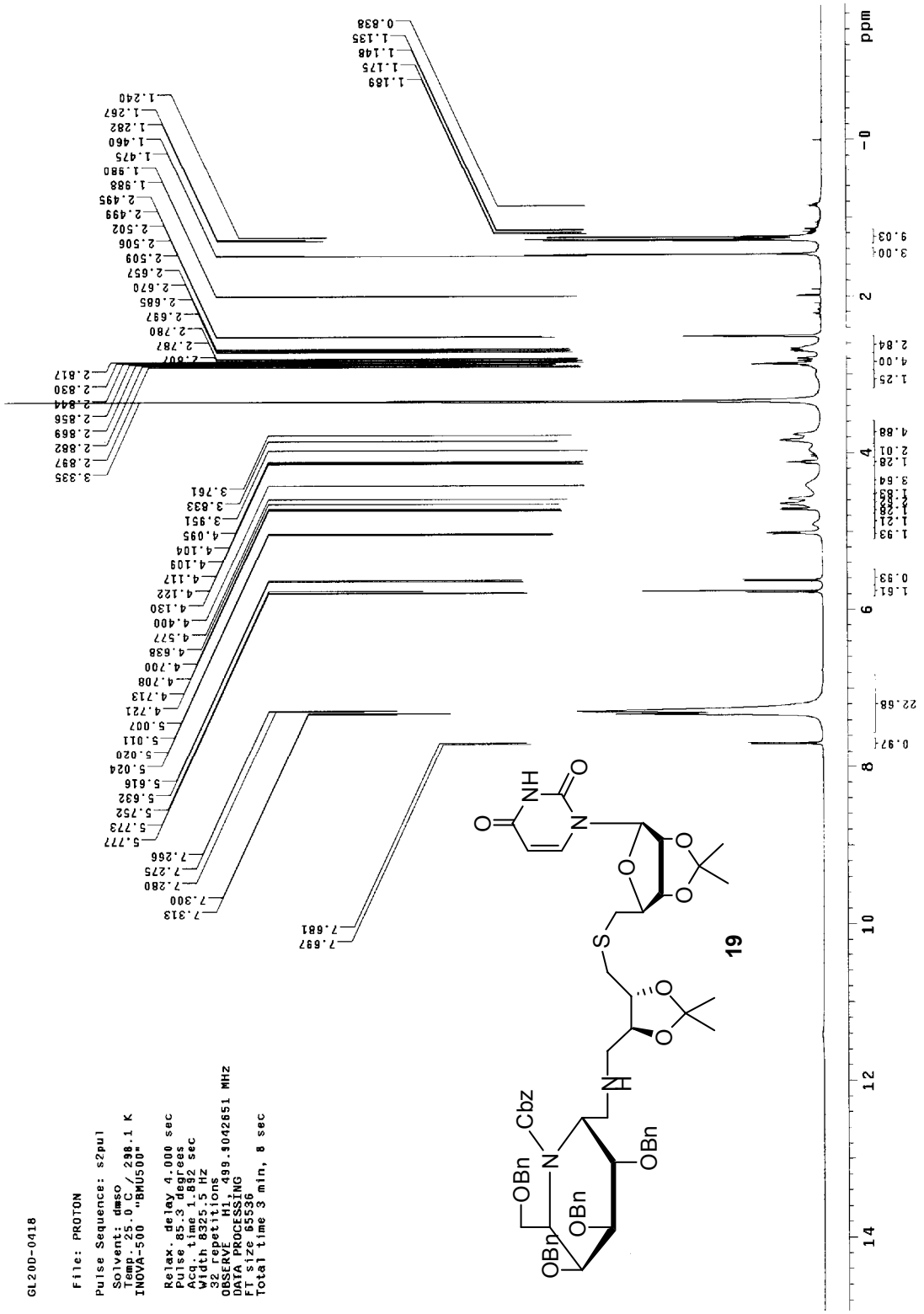
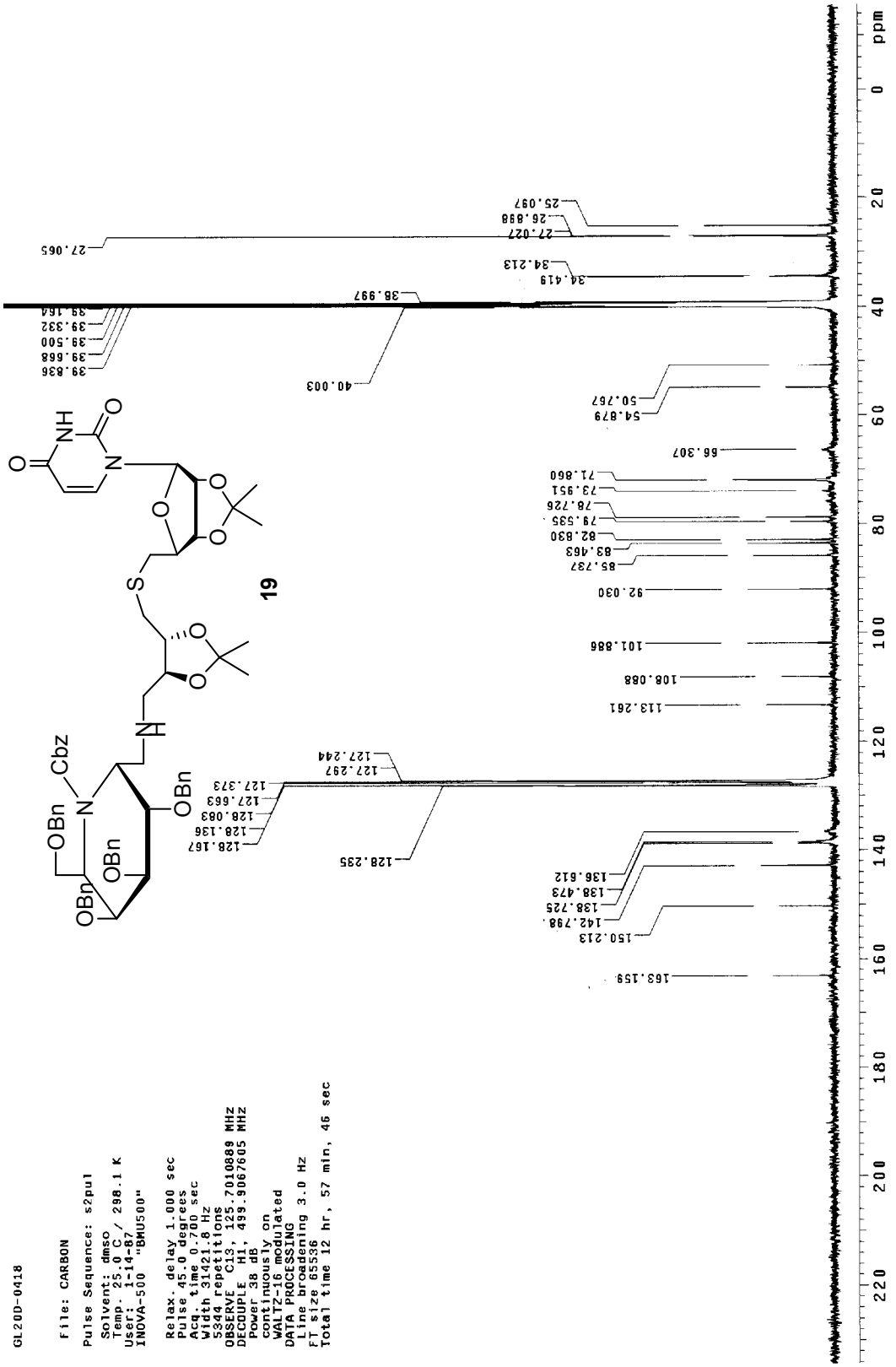
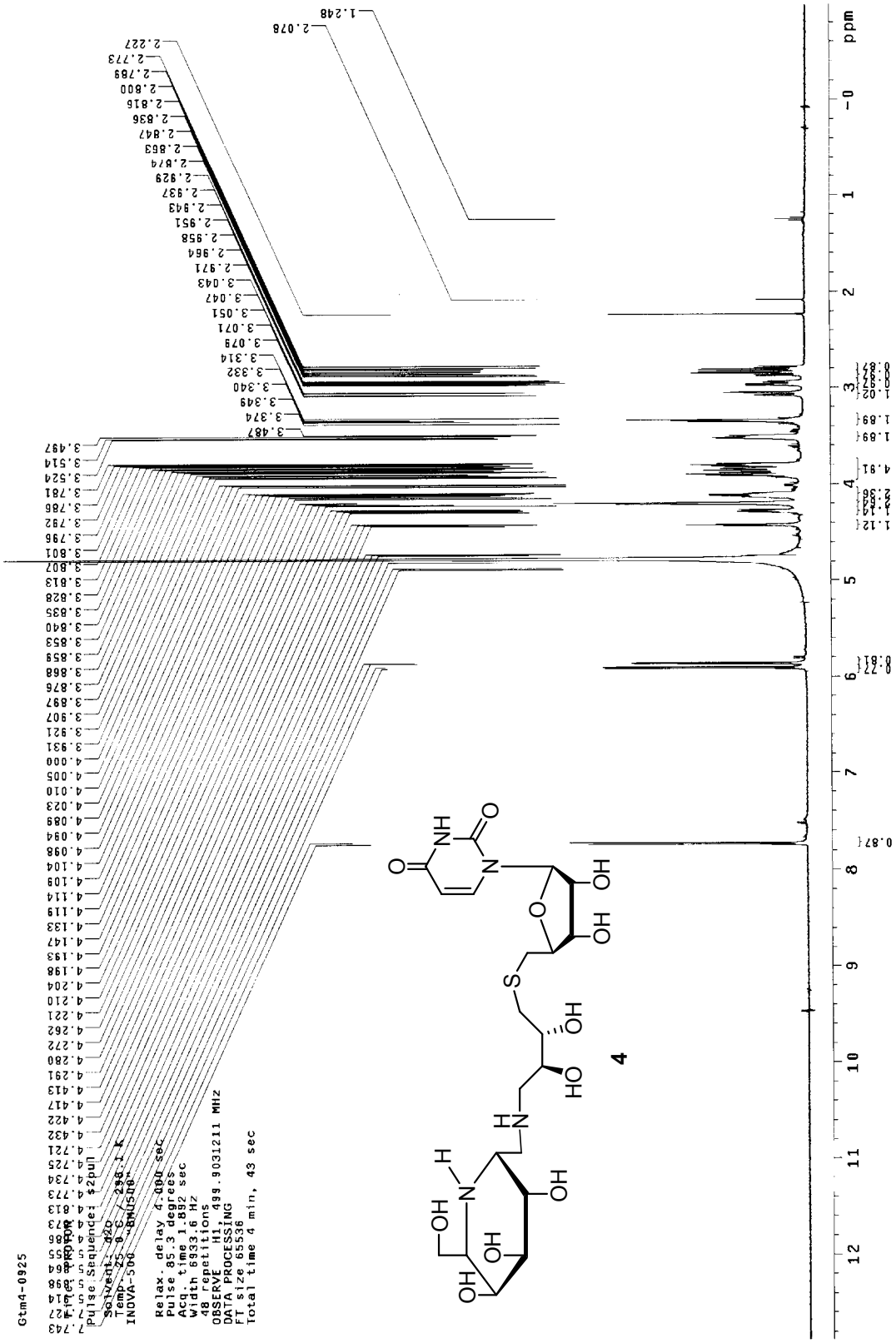
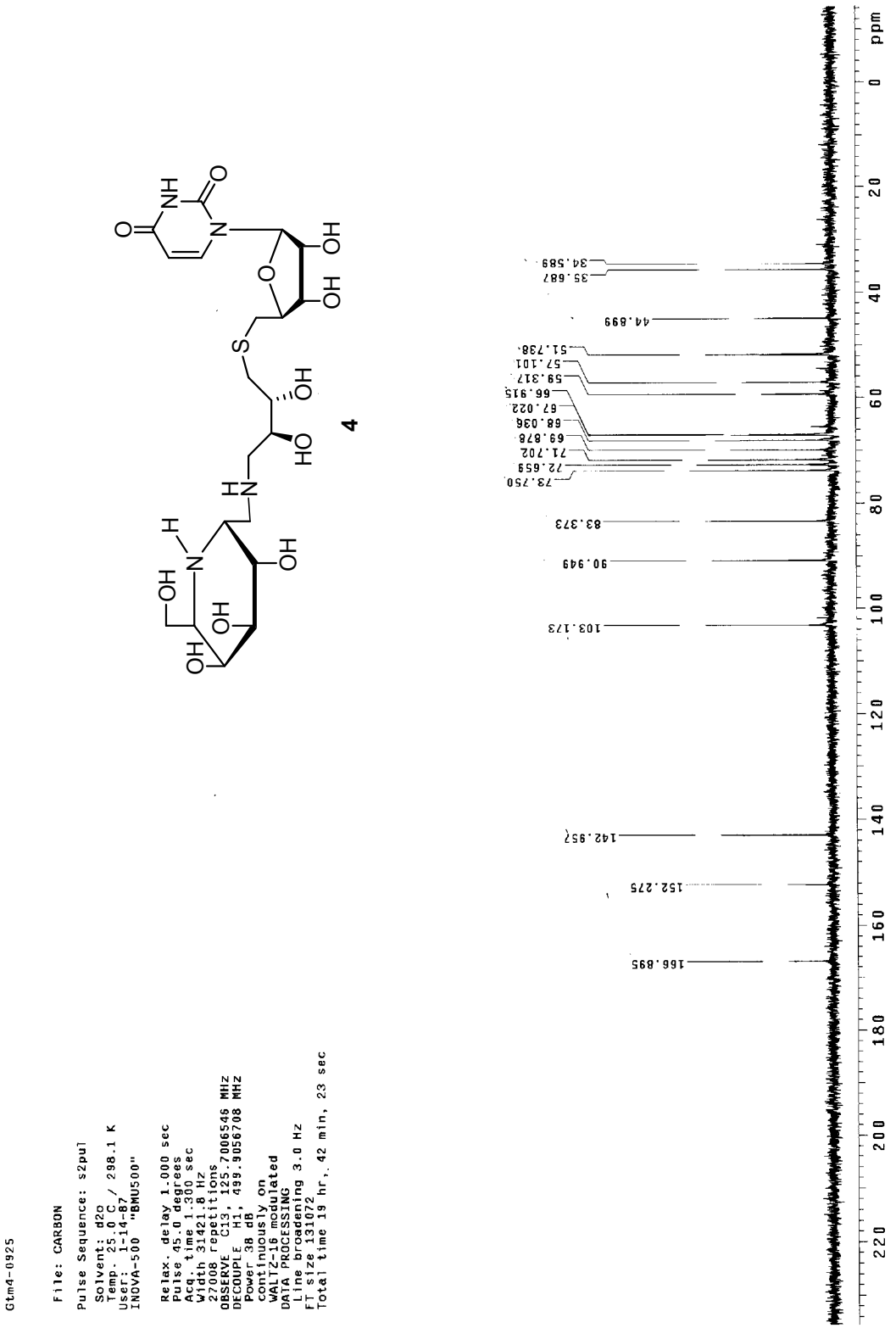
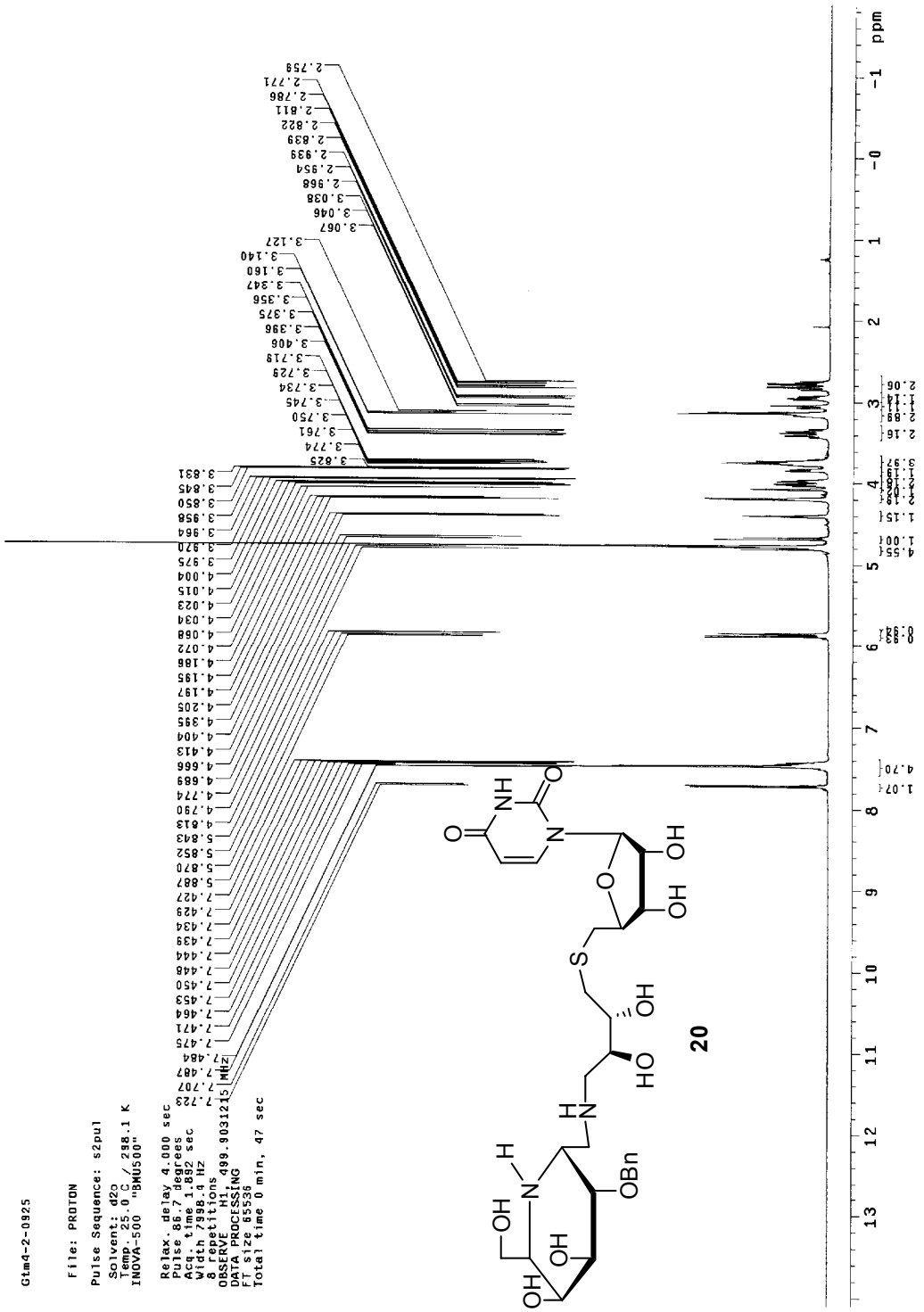
File: CARBON
Pulse Sequence: s2pul
Solvent: deut
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5248 repetitions
OBSERVE C13, 125.7006556 MHz
DECOUPLE H1, 499.9043860 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 3.5 Hz
FT size 131072
Total time 13 hr, 8 min, 15 sec
3
20.735
34.161
54.225
56.612
58.581
66.282
67.449
69.615
69.856
71.088
73.914
81.623
90.422
103.143
142.518
152.305
166.899
175.965
ppm

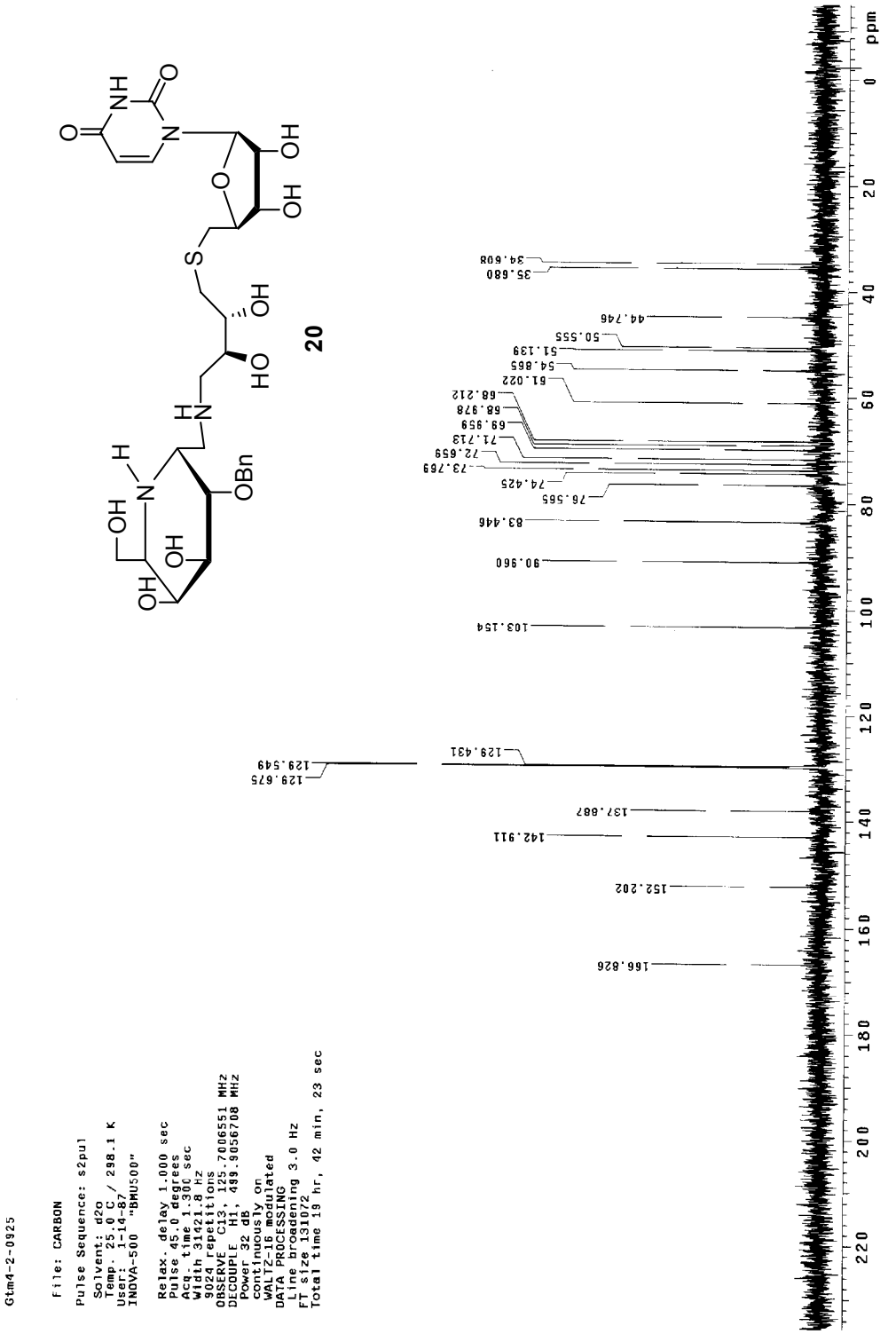

GL20D-0418
File: PROTON
Pulse Sequence: s2pul
Solvent: dmso
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 8325.5 Hz
32 repetitions
OBSERVE H1, 499.9042651 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 8 sec
ppm
19

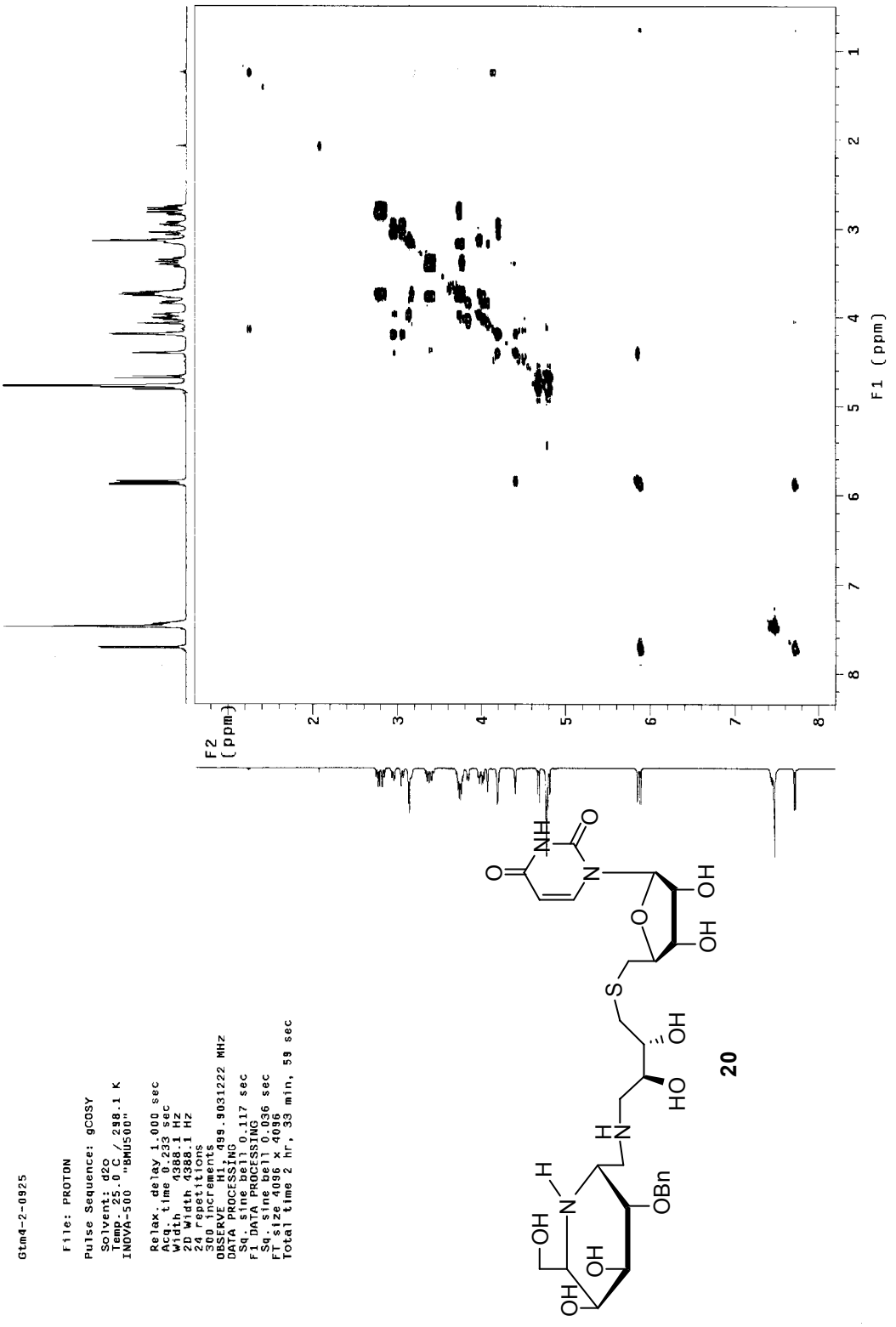

19
GL20D-0418
File: CARBON
Pulse Sequence: s2pul
Solvent: dmso
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.700 sec
Width 31421.8 Hz
5344 repetitions
OBSERVE C13, 125.7010889 MHz
DECOUPLE H1, 499.9067605 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 12 hr, 57 min, 46 sec
ppm

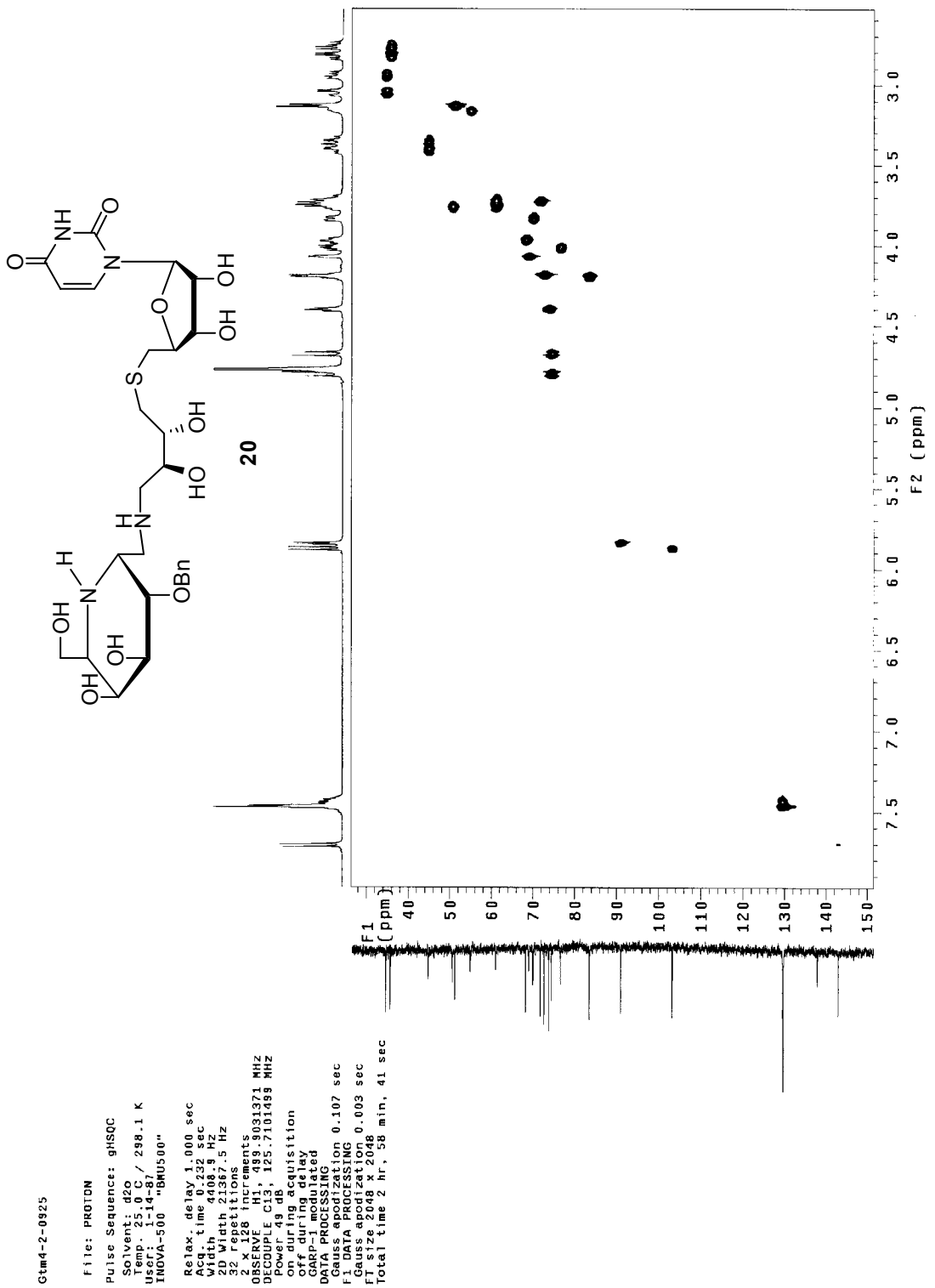

Gtm4-0925
Relax. delay 4.000 sec
Pulse 85.3 degrees
Acq. time 1.892 sec
Width 6933.6 Hz
48 repetitions
OBSERVE H1, 499.9031211 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 43 sec
Pulse Sequence: s2pul
Solvent: D2O
Temp: 25.0 C / 298.1 K
INOVA-500 "BM500"
ppm
4

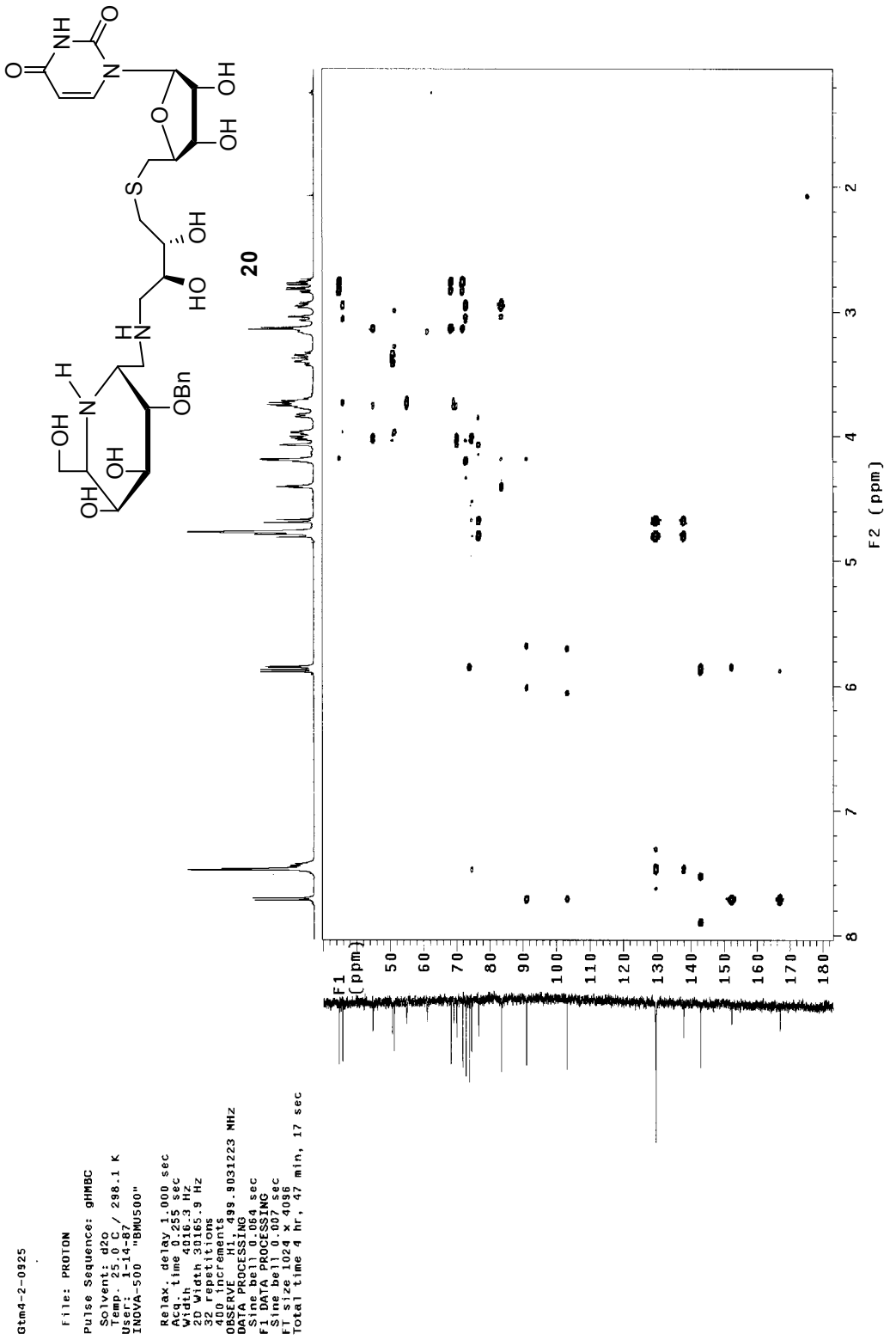

Gtm4-0925
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
27008 repetitions
OBSERVE C13, 125.7006546 MHz
DECOUPLE H1, 499.9056708 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
4
34.589
35.687
44.699
51.738
57.101
59.317
66.913
67.022
68.036
69.878
71.702
72.659
73.750
83.373
90.949
103.173
142.952
152.275
166.895
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Gtm4-2-0925
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 1.882 sec
Width 7998.4 Hz
8 repetitions
OBSERVE H1, 499.9031215 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 47 sec
20
ppm


20
Gtm4-2-0925
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 11-14-87
INOVA-500 "BMUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
9024 repetitions
OBSERVE C13, 125.7006551 MHz
DECOUPLE H1, 499.9056708 MHz
Power 32 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 19 hr, 42 min, 23 sec
34.608
35.680
44.746
50.555
51.139
54.865
61.022
68.212
68.978
69.959
71.713
72.659
73.769
74.425
76.565
83.446
90.960
103.154
129.431
129.549
129.675
137.887
142.911
152.202
166.826
ppm


Gtm4-2-0925
File: PROTON
Pulse Sequence: gCOSY
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.233 sec
Width 4388.1 Hz
2D Width 4388.1 Hz
24 repetitions
300 increments
OBSERVE H1, 499.9031222 MHz
DATA PROCESSING
Sq. sine bell 0.117 sec
FT DATA PROCESSING
F1 sine-bell 0.036 sec
FT size 4096 x 4096
Total time 2 hr, 33 min, 58 sec
F2 (ppm)
F1 (ppm)
20


Gtm4-2-0925
File: PROTON
Pulse Sequence: gHSQC
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.232 sec
Width 4408.9 Hz
2D Width 21367.5 Hz
32 repetitions
2 x 128 increments
OBSERVE H1, 499.9031371 MHz
DECOUPLE C13, 125.7101499 MHz
Power 49 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.107 sec
F1 DATA PROCESSING
Gauss apodization 0.003 sec
FT size 2048 x 2048
Total time 2 hr, 58 min, 41 sec
20
F1 (ppm)
F2 (ppm)


20
F1 (ppm)
F2 (ppm)
Gtm4-2-0925
File: PROTON
Pulse Sequence: gHMBC
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMUS00"
Relax. delay 1.000 sec
Acq. time 0.255 sec
Width 4016.3 Hz
2D Width 30165.9 Hz
32 repetitions
400 increments
OBSERVE H1, 499.9031223 MHz
DATA PROCESSING
Sine bell 0.104 sec
FT DATA PROCESSING
Sine bell 0.007 sec
FT size 1024 x 4096
Total time 4 hr, 47 min, 17 sec