Design, Synthesis and Functional Activity of Labeled CD1d Glycolipid Agonists

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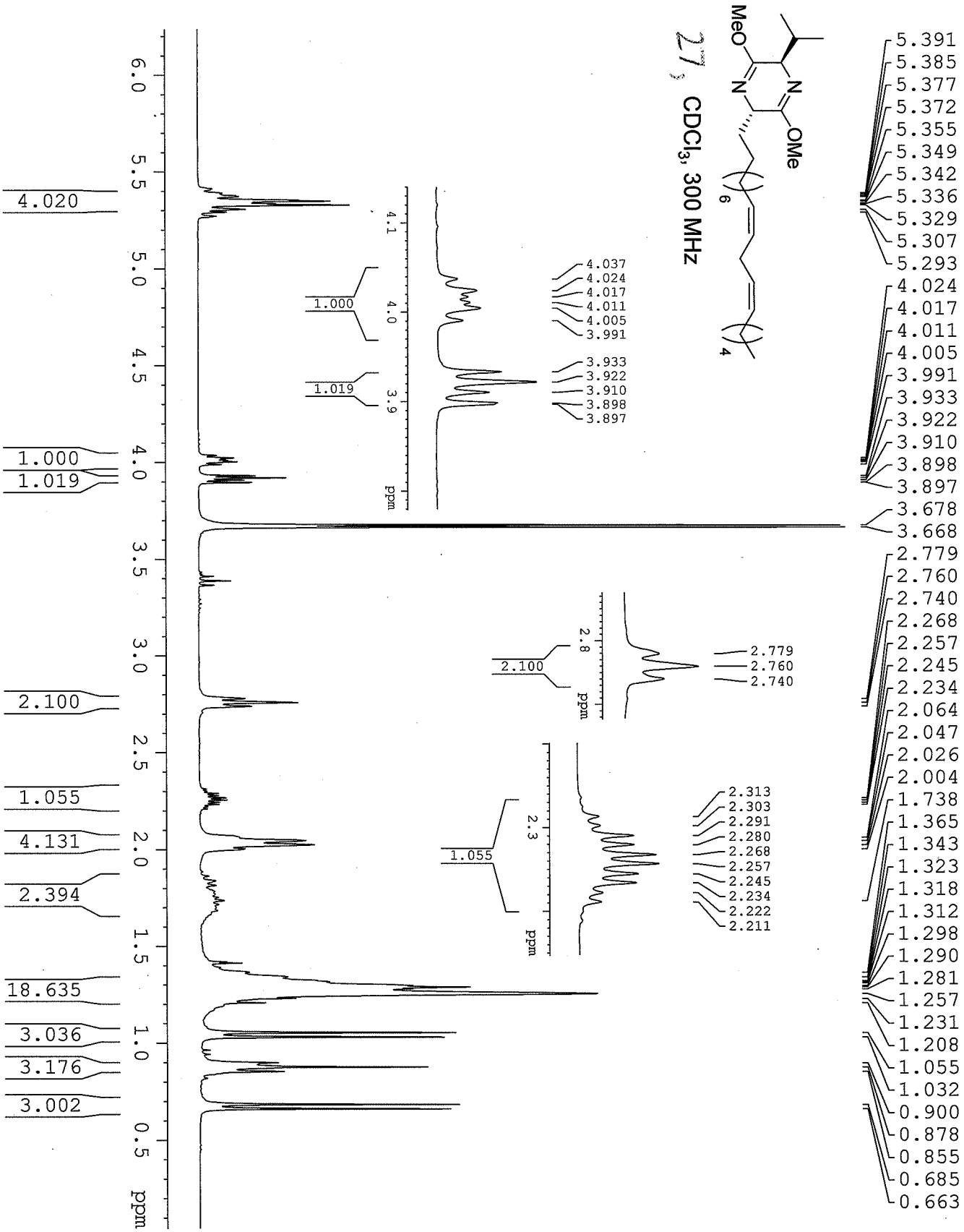
Supporting Information V


27
CDCl3, 300 MHz
MeO
OMe
N
N
6
4
5.391
5.385
5.377
5.372
5.355
5.349
5.342
5.336
5.329
5.307
5.293
4.024
4.017
4.011
4.005
3.991
3.933
3.922
3.910
3.898
3.897
3.678
3.668
2.779
2.760
2.740
2.268
2.257
2.245
2.234
2.064
2.047
2.026
2.004
1.738
1.365
1.343
1.323
1.318
1.312
1.298
1.290
1.281
1.257
1.231
1.208
1.055
1.032
0.900
0.878
0.855
0.685
0.663
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1.000
1.019
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1.055
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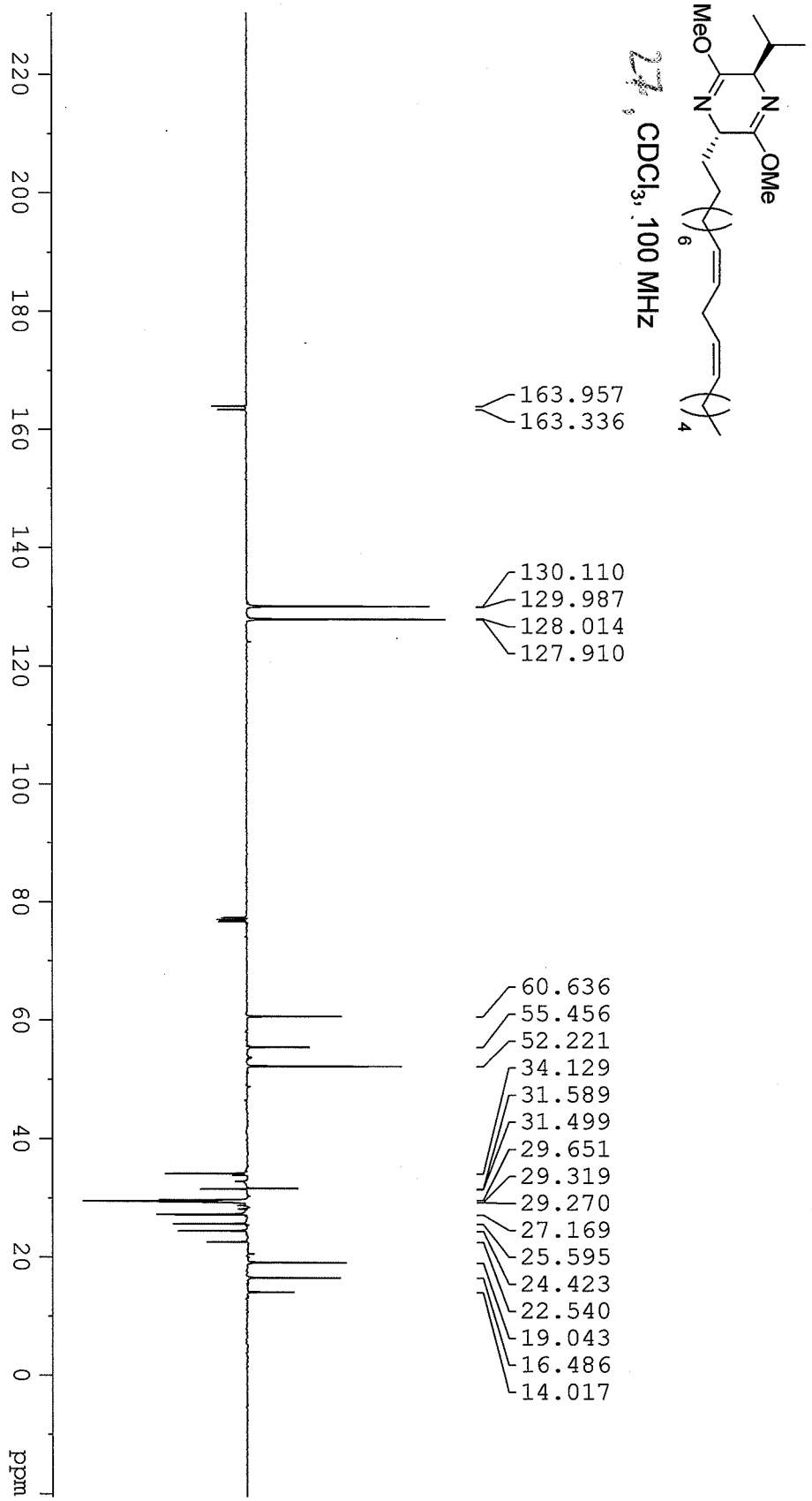
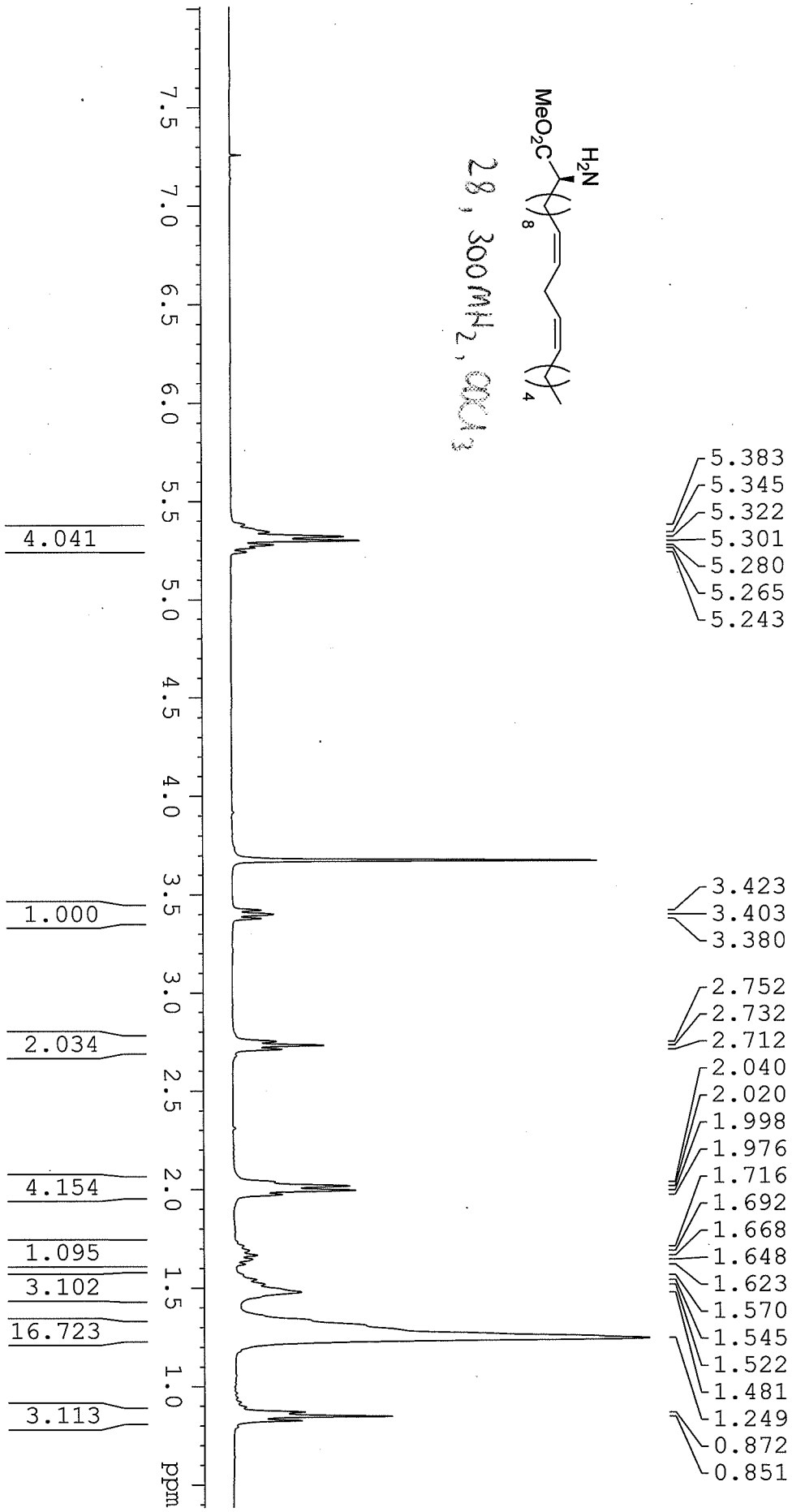
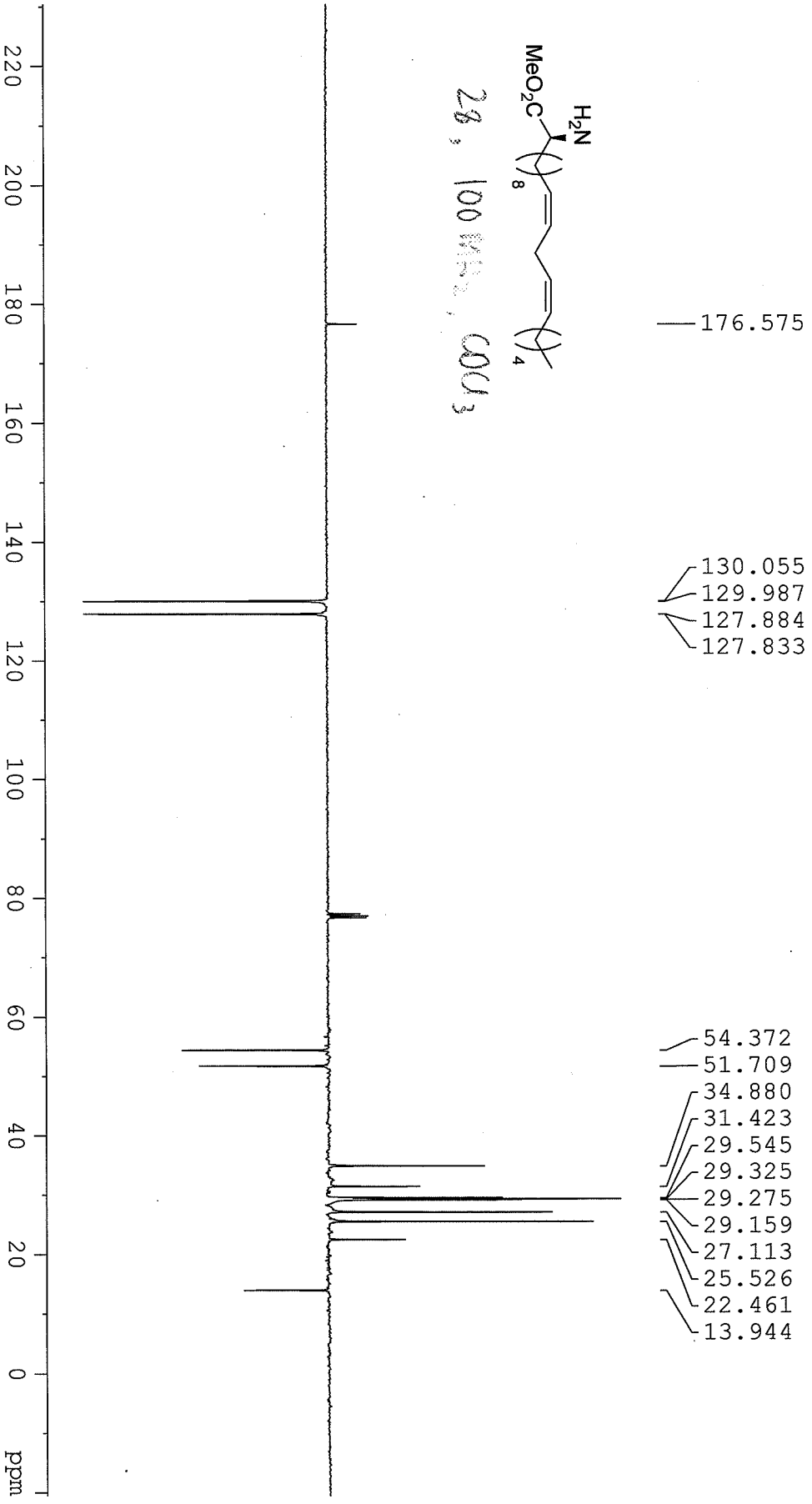
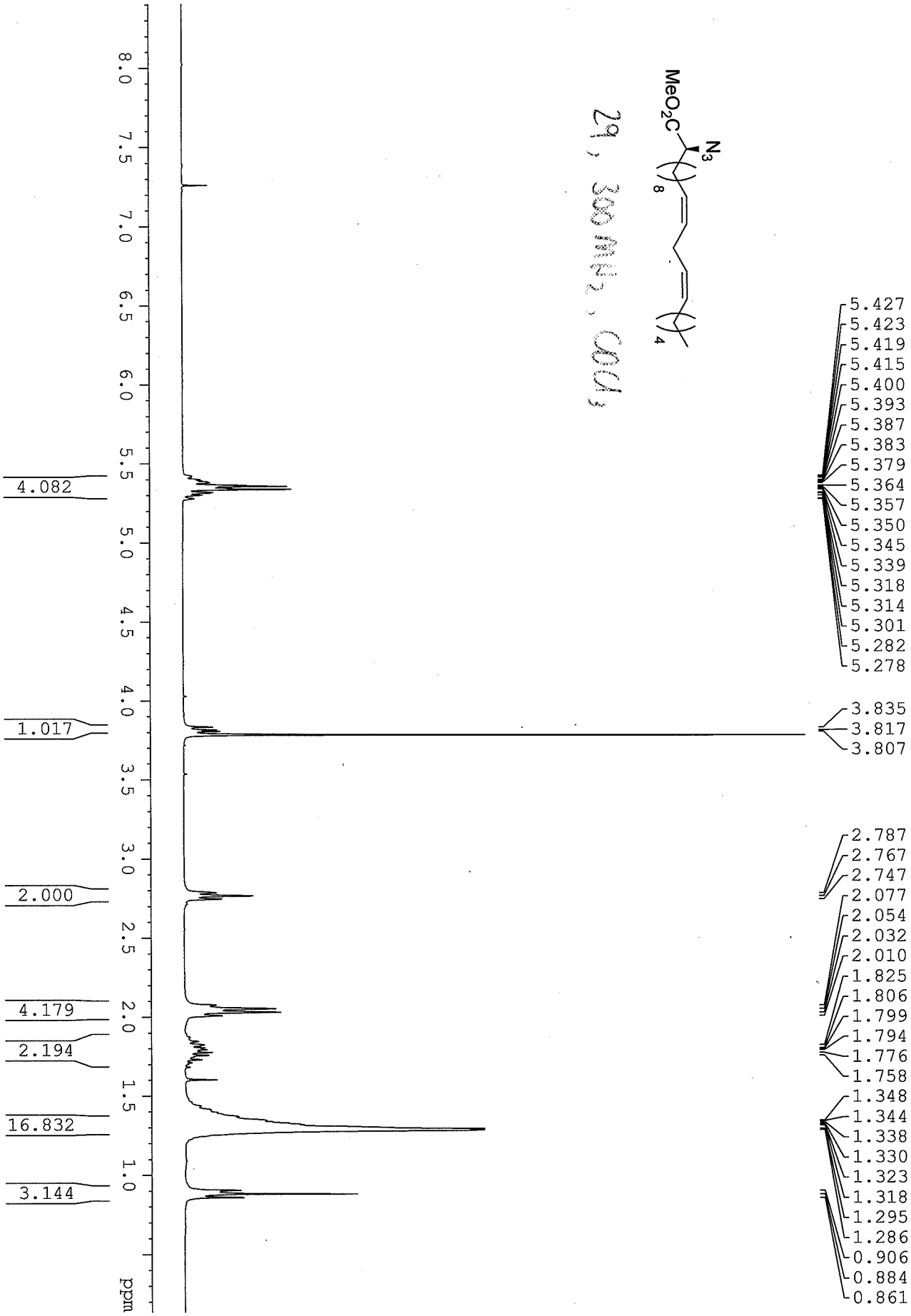
18.635
3.036
3.176
3.002
ppm

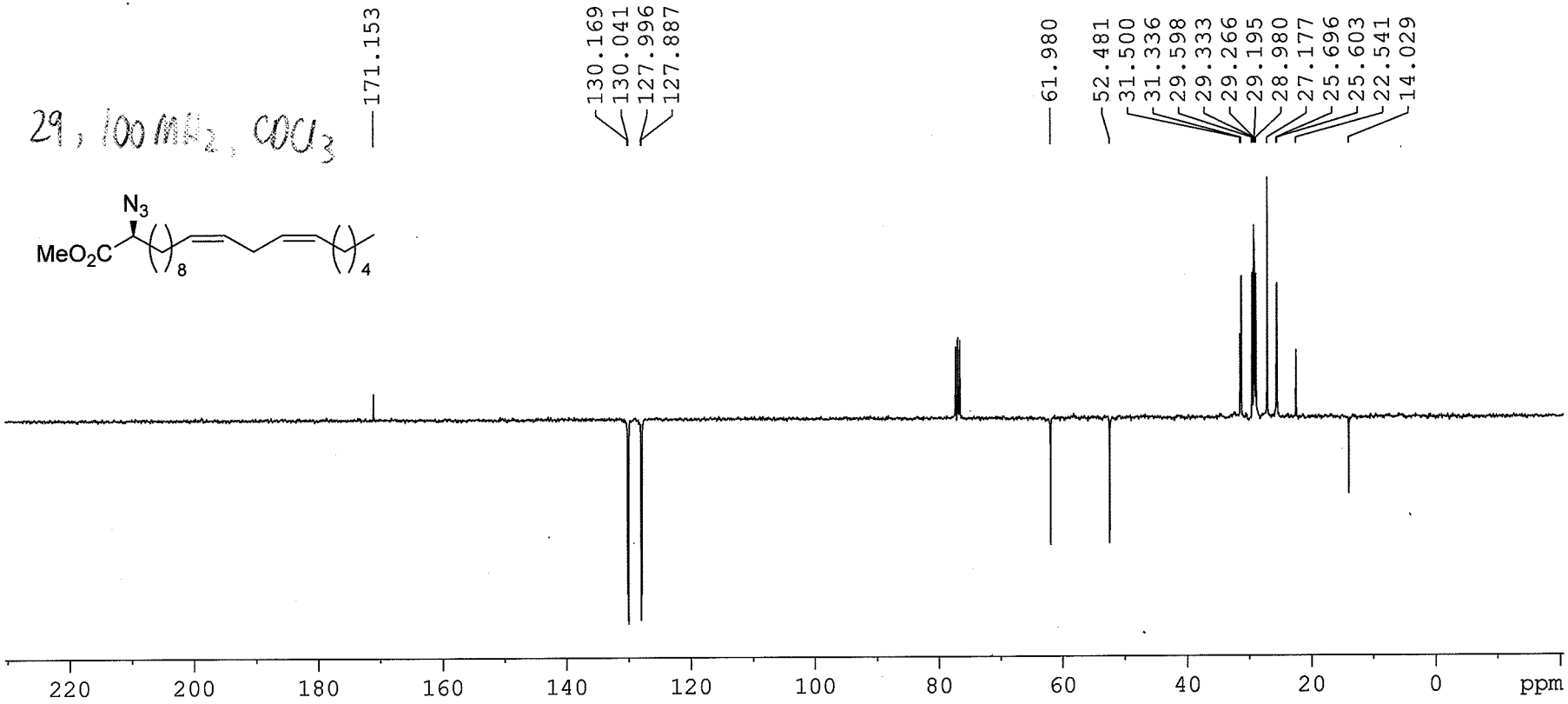

MeO
N
N
OMe
6
4
CDCl3, 100 MHz
163.957
163.336
130.110
129.987
128.014
127.910
60.636
55.456
52.221
34.129
31.589
31.499
29.651
29.319
29.270
27.169
25.595
24.423
22.540
19.043
16.486
14.017
220
200
180
160
140
120
100
80
60
40
20
0
ppm

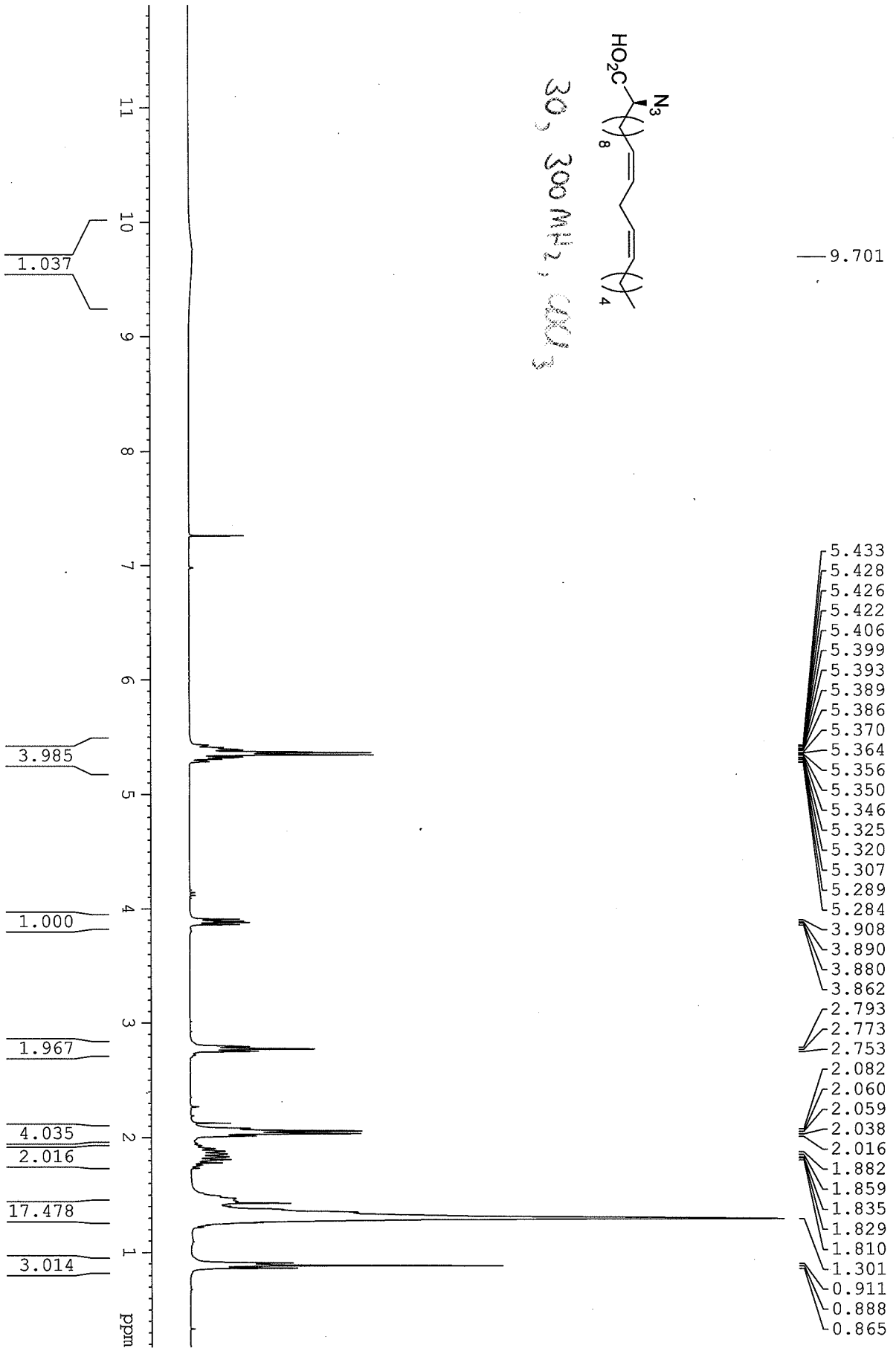

MeO2C
H2N
8
4
28, 300MHz, CDCl3
5.383
5.345
5.322
5.301
5.280
5.265
5.243
3.423
3.403
3.380
2.752
2.732
2.712
2.040
2.020
1.998
1.976
1.716
1.692
1.668
1.648
1.623
1.570
1.545
1.522
1.481
1.249
0.872
0.851
4.041
1.000
2.034
4.154
1.095
3.102
16.723
3.113
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

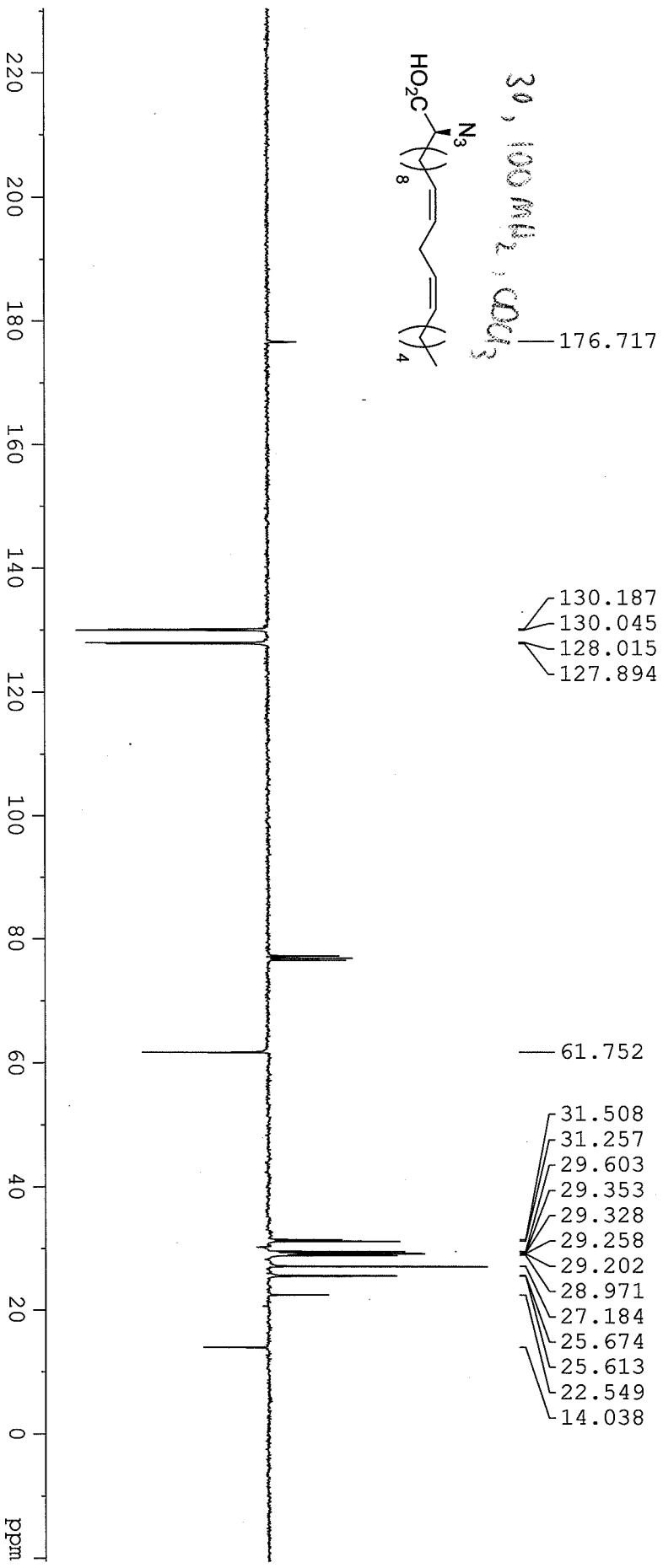

MeO2C
H2N
8
4
28, 100 MHz, CDCl3
176.575
130.055
129.987
127.884
127.833
54.372
51.709
34.880
31.423
29.545
29.325
29.275
29.159
27.113
25.526
22.461
13.944
220
200
180
160
140
120
100
80
60
40
20
0
ppm

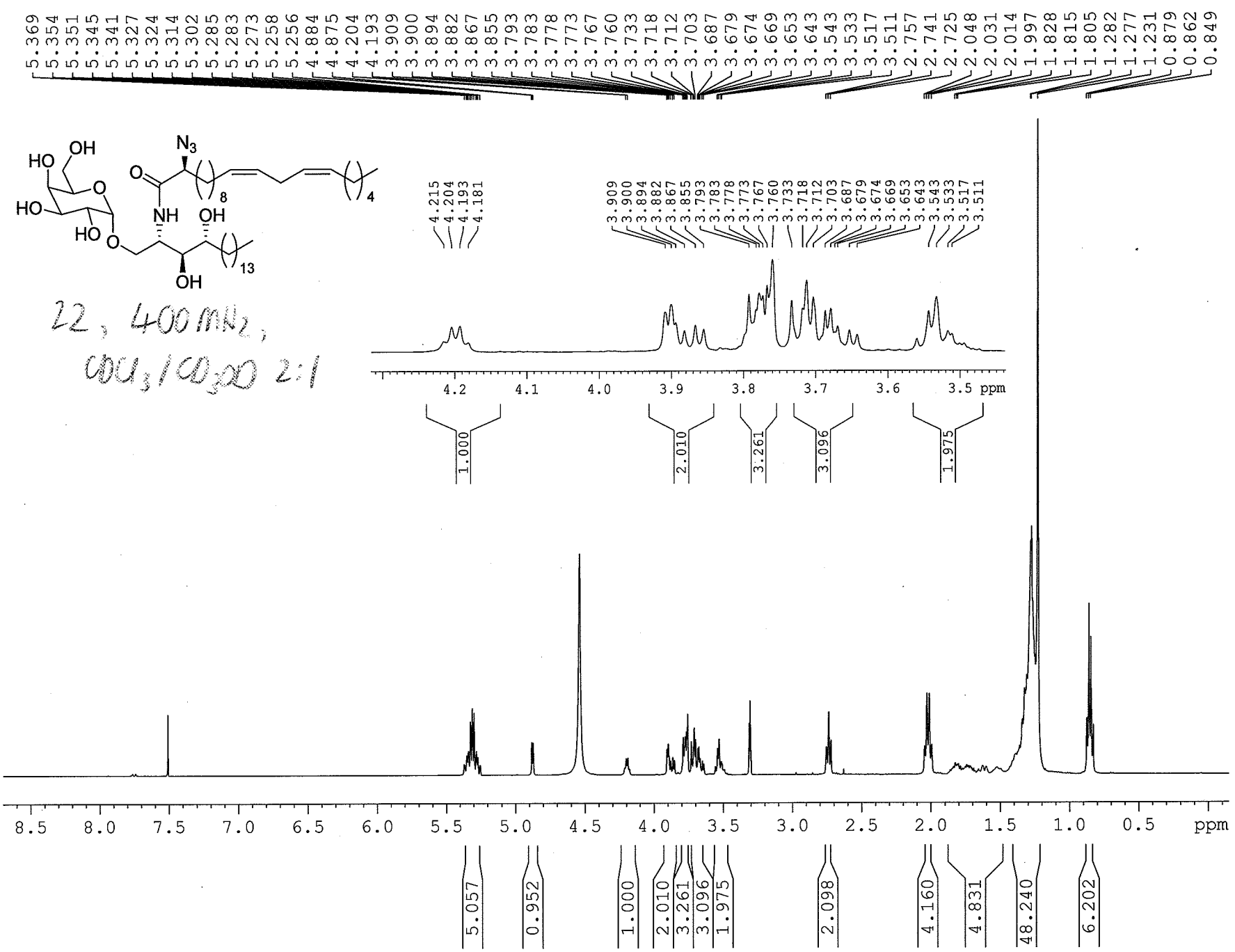

MeO2C
N3
8
4
29, 300 MHz, CDCl3
4.082
1.017
2.000
4.179
2.194
16.832
3.144
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
5.427
5.423
5.419
5.415
5.400
5.393
5.387
5.383
5.379
5.364
5.357
5.350
5.345
5.339
5.318
5.314
5.301
5.282
5.278
3.835
3.817
3.807
2.787
2.767
2.747
2.077
2.054
2.032
2.010
1.825
1.806
1.799
1.794
1.776
1.758
1.348
1.344
1.338
1.330
1.323
1.318
1.295
1.286
0.906
0.884
0.861

29, 100 MHz, $CDCl_3$




HO2C
N3
8
4
30, 300 MHz, CDCl3
9.701
5.433
5.428
5.426
5.422
5.406
5.399
5.393
5.389
5.386
5.370
5.364
5.356
5.350
5.346
5.325
5.320
5.307
5.289
5.284
3.908
3.890
3.880
3.862
2.793
2.773
2.753
2.082
2.060
2.059
2.038
2.016
1.882
1.859
1.835
1.829
1.810
1.301
0.911
0.888
0.865
1.037
3.985
1.000
1.967
4.035
2.016
17.478
3.014
ppm


30, 100 MHz, CDCl3
HO2C
N3
8
4
176.717
130.187
130.045
128.015
127.894
61.752
31.508
31.257
29.603
29.353
29.328
29.258
29.202
28.971
27.184
25.674
25.613
22.549
14.038
220
200
180
160
140
120
100
80
60
40
20
0
ppm

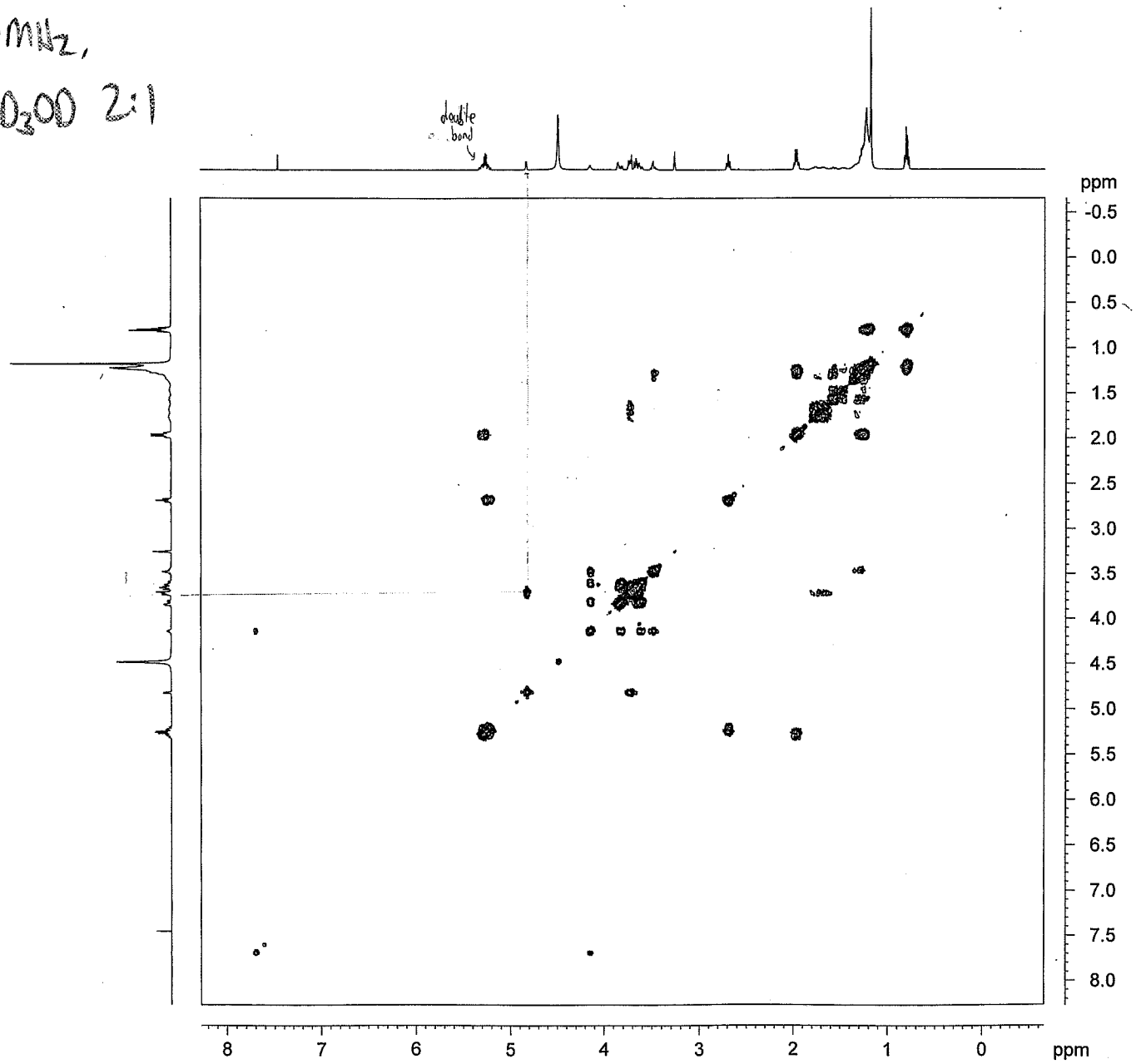

5.369
5.354
5.351
5.345
5.341
5.327
5.324
5.314
5.302
5.285
5.283
5.273
5.258
5.256
4.884
4.875
4.204
4.193
3.909
3.900
3.894
3.882
3.867
3.855
3.793
3.783
3.778
3.773
3.767
3.760
3.733
3.718
3.712
3.703
3.687
3.679
3.674
3.669
3.653
3.643
3.543
3.533
3.517
3.511
2.757
2.741
2.725
2.048
2.031
2.014
1.997
1.828
1.815
1.805
1.282
1.277
1.231
0.879
0.862
0.849
HO
OH
HO
O
HO
O
N3
O
8
NH
OH
4
13
OH
22, 400 MHz,
CDCl3/CD3OD 2:1
4.215
4.204
4.193
4.181
3.909
3.900
3.894
3.882
3.867
3.855
3.793
3.783
3.778
3.773
3.767
3.760
3.733
3.718
3.712
3.703
3.687
3.679
3.674
3.669
3.653
3.643
3.543
3.533
3.517
3.511
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5 ppm
1.000
2.010
3.261
3.096
1.975
8.5
8.0
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
5.057
0.952
1.000
2.010
3.261
3.096
1.975
2.098
4.160
4.831
48.240
6.202

22, 100MHz, CDCl3/CD3OD 2:1
171.011
130.533
130.441
128.450
128.379
100.282
74.880
72.344
71.376
70.784
70.302
69.389
67.561
64.006
62.293
51.034
32.877
32.374
32.302
32.042
31.972
30.154
29.943
29.795
29.709
29.571
27.617
26.288
26.183
26.034
23.095
22.985
14.252
220
200
180
160
140
120
100
80
60
40
20
0
ppm

P Jervis, Gal N3 C20:2

22, 400MHz,

$CDCl_3/CD_3OD$ 2:1



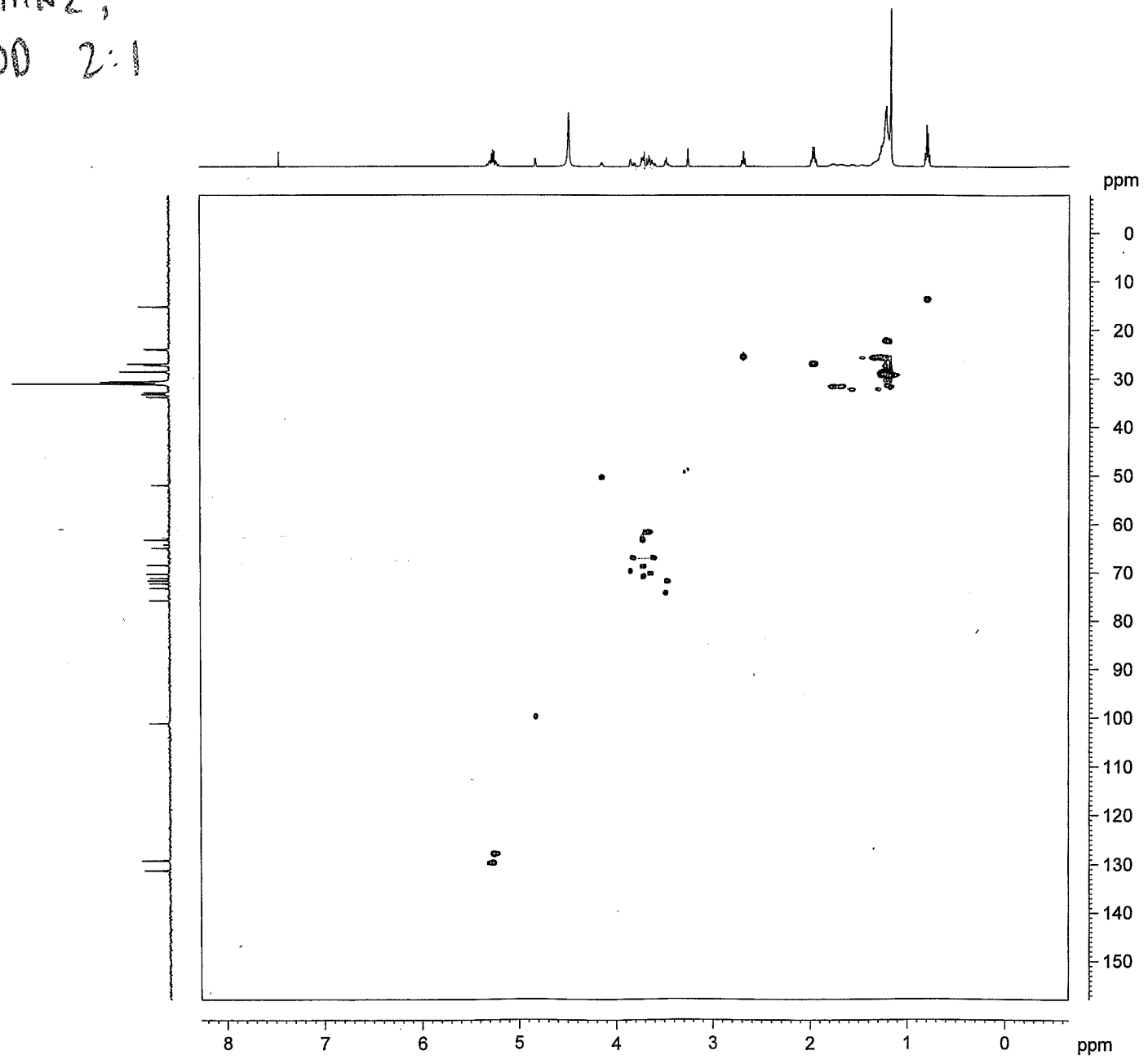

```
NAME      03-15-Besra-10
EXPNO             11
PROCNO             1
Date_         20120315
Time             20.32
INSTRUM           spect
PROBHD  5 mm PADUL 13C
PULPROG       cosygpmfqf
TD                1024
SOLVENT           MeOD
NS                   4
DS                  16
SWH          3571.428 Hz
FIDRES        3.487723 Hz
AQ           0.1434100 sec
RG                2050
DW             140.000 usec
DE               6.50 usec
TE              296.5 K
D0           0.00000300 sec
D1           1.43937802 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00028000 sec

======== CHANNEL f1 ========
NUC1               1H
P1               9.50 usec
PL1              -4.00 dB
PL1W       24.29185867 W
SFO1       400.1315530 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPNAM3         SINE.100
GPZ1            16.00 %
GPZ2            12.00 %
GPZ3            40.00 %
P16           1000.00 usec
ND0                  1
TD                 256
SFO1       400.1316 MHz
FIDRES      13.950892 Hz
SW           8.926 ppm
FnMODE             QF
SI                1024
SF         400.1300296 MHz
WDW              SINE
SSB                  0
LB            0.00 Hz
GB                   0
PC                1.40
SI                 512
MC2                QF
SF         400.1300296 MHz
WDW              SINE
SSB                  0
LB            0.00 Hz
GB                   0
```

P Jervis, Gal N3 C20:2

22 , 400 MHz ,
$CDCl_3/CD_3OD$ 2:1



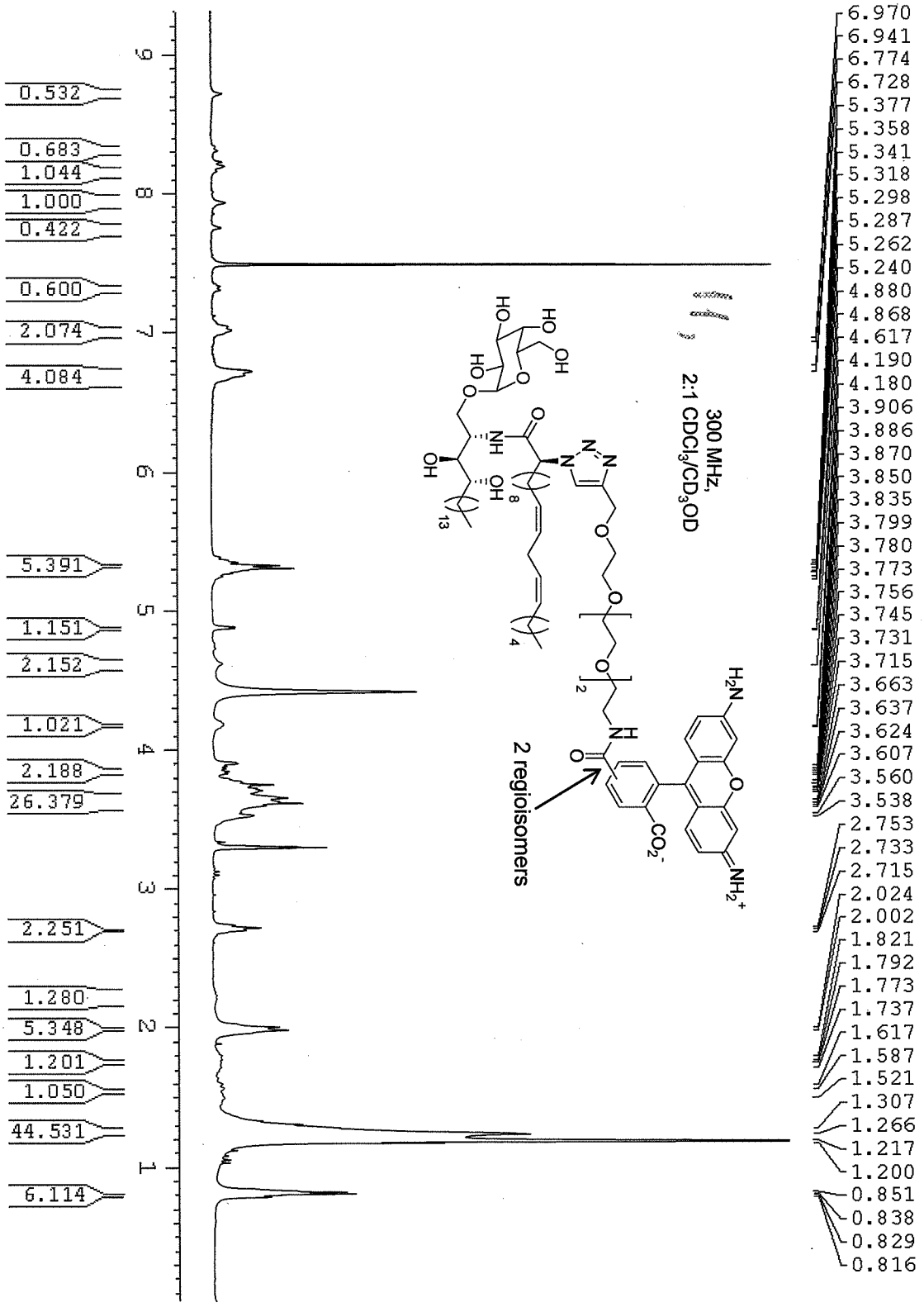
NAME 03-15-Besra-10
EXPNO 13
PROCNO 1
Date_ 20120315
Time 21.27
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG hsqcetgp
TD 1024
SOLVENT MeOD
NS 4
DS 16
SWH 3571.428 Hz
FIDRES 3.487723 Hz
AQ 0.1434100 sec
RG 2050
DW 140.000 usec
DE 6.50 usec
TE 297.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.45269096 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00003000 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
P2 19.00 usec
P28 0.00 usec
PL1 -4.00 dB
PL1W 24.29185867 W
SFO1 400.1315530 MHz

======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 8.90 usec
P4 17.80 usec
PCPD2 78.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL2W 58.63890457 W
PL12W 0.73822010 W
SFO2 100.6203124 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec
ND0 2
TD 256
SFO1 100.6203 MHz
FIDRES 65.108421 Hz
SW 165.650 ppm
FnMODE Echo-Antiecho
SI 1024
SF 400.1300296 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 512
MC2 echo-antiecho
SF 100.6127690 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

S106

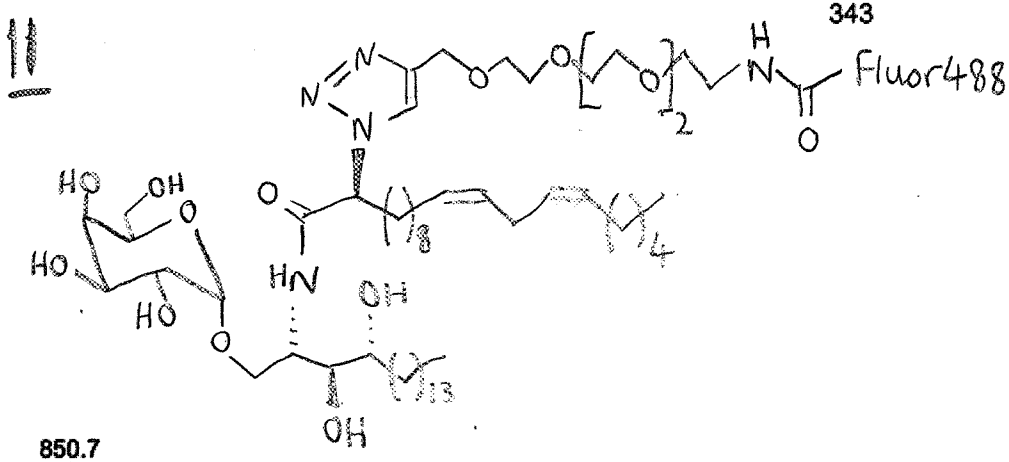

300 MHz,
2:1 CDCl3/CD3OD
2 regioisomers
0.532
0.683
1.044
1.000
0.422
0.600
2.074
4.084
5.391
1.151
2.152
1.021
2.188
26.379
2.251
1.280
5.348
1.201
1.050
44.531
6.114
6.970
6.941
6.774
6.728
5.377
5.358
5.341
5.318
5.298
5.287
5.262
5.240
4.880
4.868
4.617
4.190
4.180
3.906
3.886
3.870
3.850
3.835
3.799
3.780
3.773
3.756
3.745
3.731
3.715
3.663
3.637
3.624
3.607
3.560
3.538
2.753
2.733
2.715
2.024
2.002
1.821
1.792
1.773
1.737
1.617
1.587
1.521
1.307
1.266
1.217
1.200
0.851
0.838
0.829
0.816

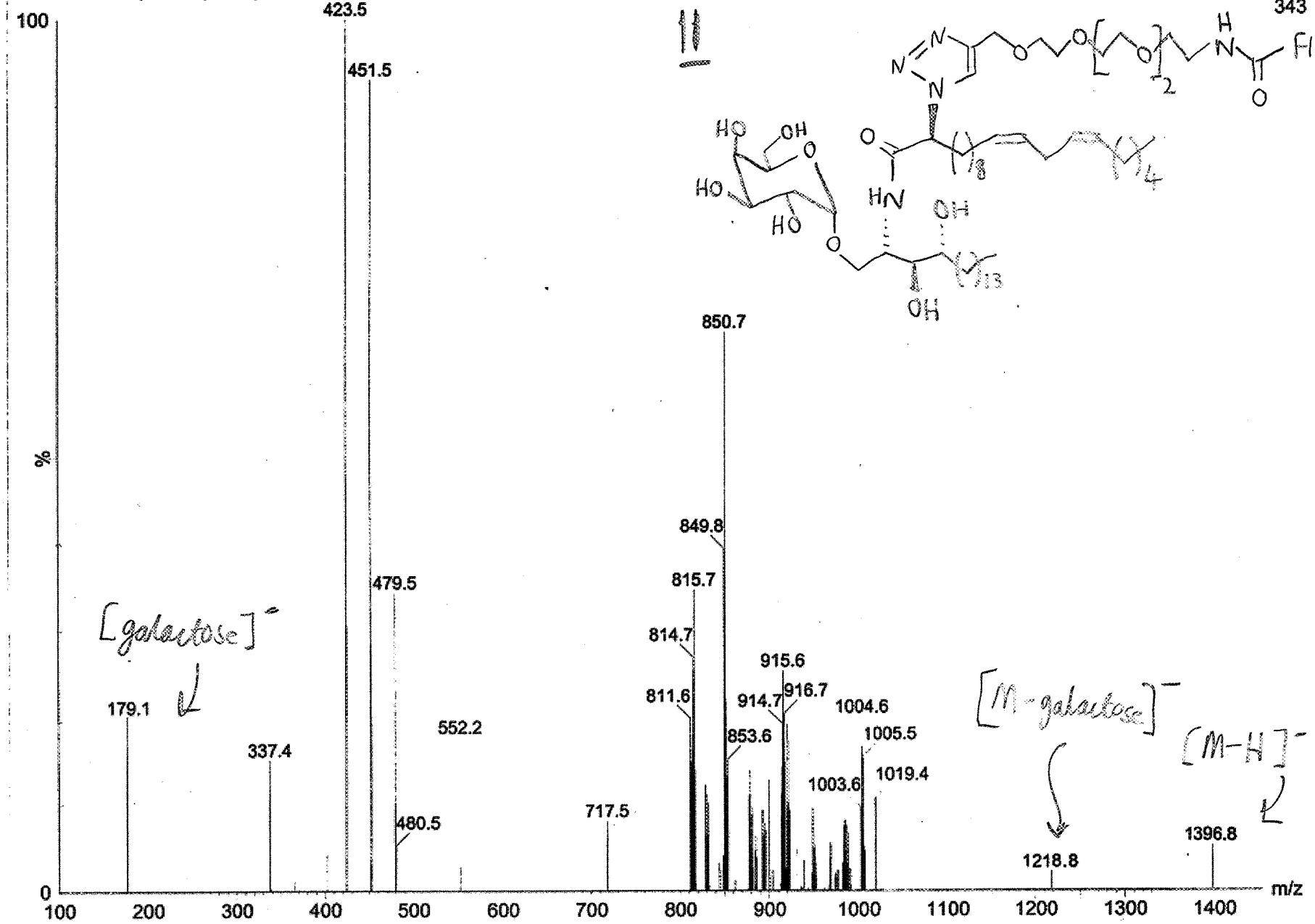
Peter Jervis PJJ488

ES29485 294 (10.295) Cm (290:300-1:116)

TOF MS ES-

343





301S