Supporting Information

Total synthesis of neuroprotectin D1 analogs derived from omega-6 docosapentaenoic acid (DPA) and adrenic acid (AdA) from a common pivotal, late-stage intermediate

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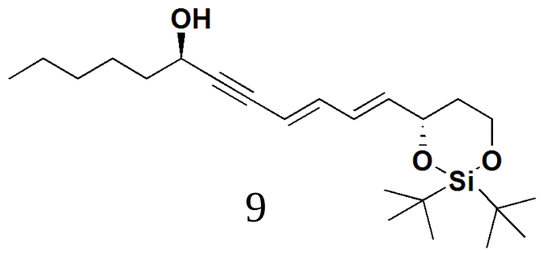
Table of Contents

Effect of the concentration on the multiplicity of proton H10

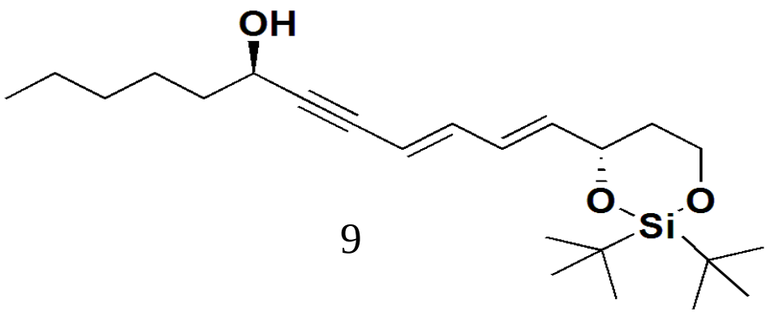
1H NMR spectrum for compound 9
about 5 mg in the NMR tube
sample dg48af2
AMX300, 303K, CDCl3



9

1H NMR spectrum for compound 9
about 15 mg in the NMR tube
sample lodg48/1/1
AMX300, 303K, CDCl3

9



9

141.600
138.858
127.925
110.597
92.779
84.132
77.590
77.166
76.743
73.732
64.165
63.242
38.035
36.836
31.623
27.534
27.303
25.007
22.808
22.705
20.146
14.136

Jmod sur lodg48/7/1
AMX300, 303K, CDCl3

S3

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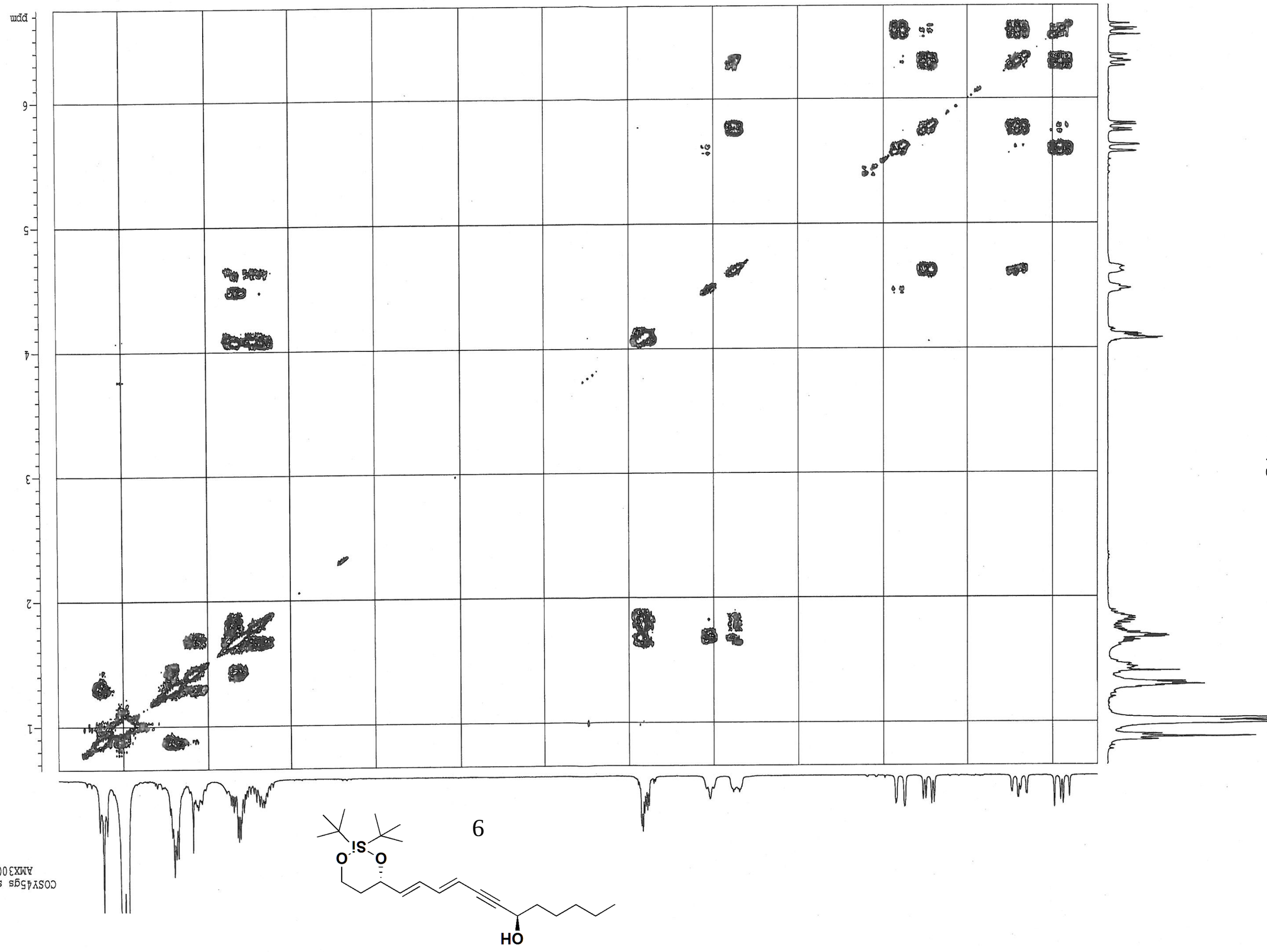
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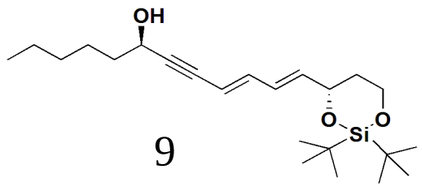
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
6
5
4
3
2
1
COSY45gs sur lodg48/4/1
AMX300, 303K, CDCl3
9
Si
O
O
HO



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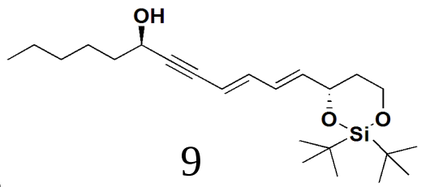
HMQC sur lodg48/5/1
AMX300, 303K, CDCl3



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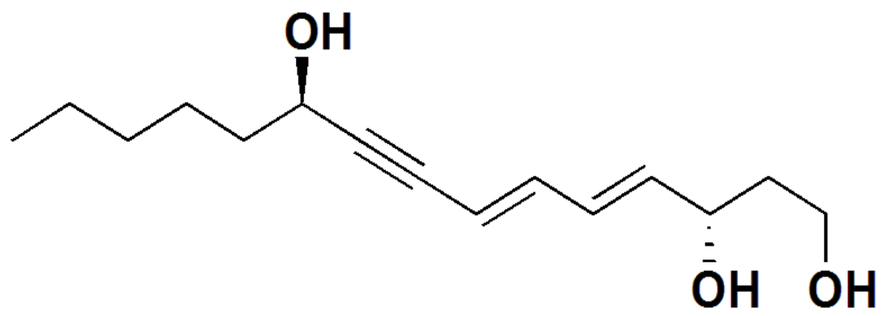
HMBC sur lodg48/6/1
AMX300, 303K, CDCl3

S6

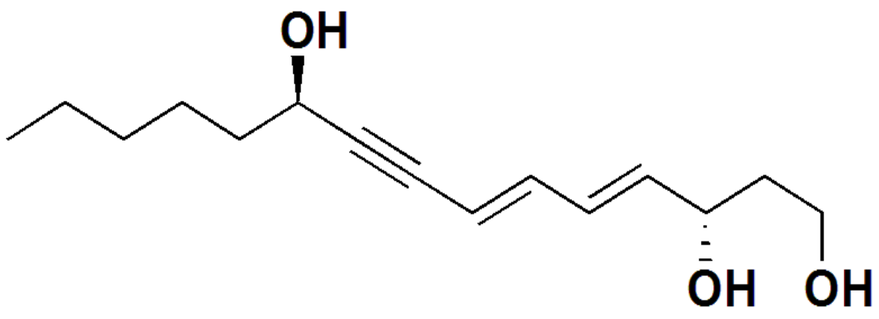
ppm
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

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



1H sur lodg49/1/1
compund dg49aF2
AMX300, 303K, CDCl3

S7



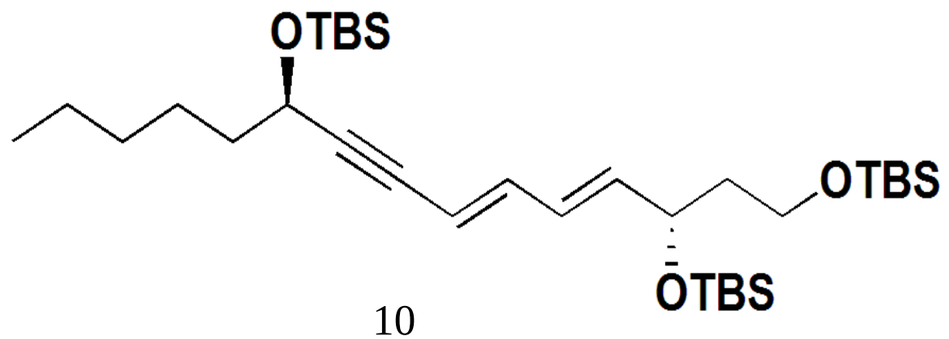
141.240
138.290
129.272
111.242
93.278
83.901
77.589
77.165
76.742
71.732
63.118
60.997
38.486
37.964
31.599
25.017
22.685
14.124

Jmod sur lodg49/7/1
compund dg49aF2
AMX300, 303K, CDCl3

S8

ppm 200 180 160 140 120 100 80 60 40 20

dg50ff1 14.11.2013 1H sur
AMX300, 303K, CDCl3



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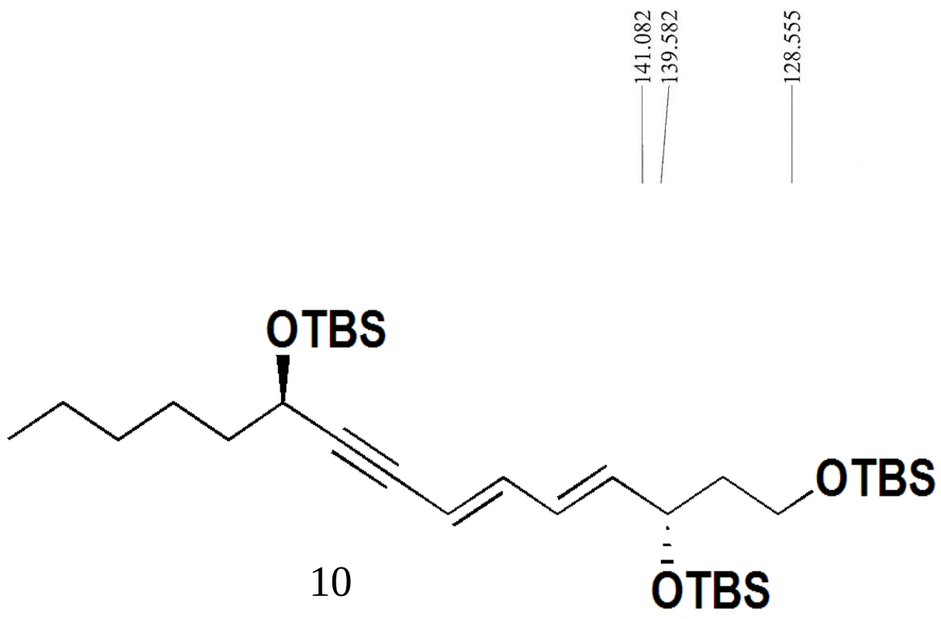

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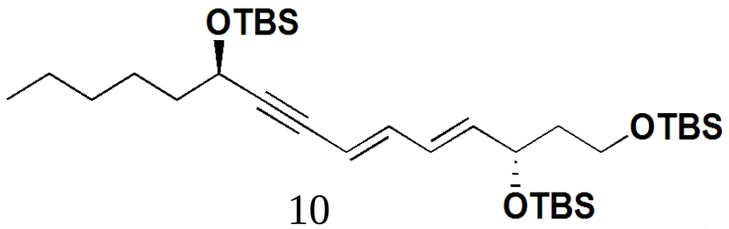
Jmod sur lodg50/7/1
sample dg50aF1 - 45mg

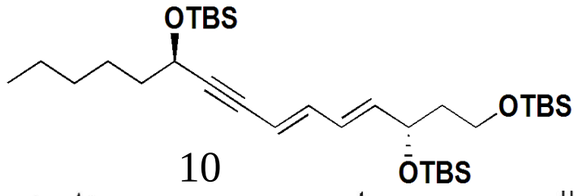
141.082
139.582
128.555
110.702
93.838
83.375
77.581
77.151
76.734
69.977
63.718
59.444
41.468
38.865
31.639
26.082
26.037
25.865
25.108
22.742
18.422
18.376
18.279
14.150
-2.786
-4.216
-4.254
-4.741
-4.808

200
175
150
125
100
75
50
25
0


OTBS
OTBS
OTBS

10

COSY45gs sur lodg50/4/1
sample dg50aF1 - 45mg
AMX300, 303K, CDCl3
0
1
2
3
4
5
6
ppm
ppm
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



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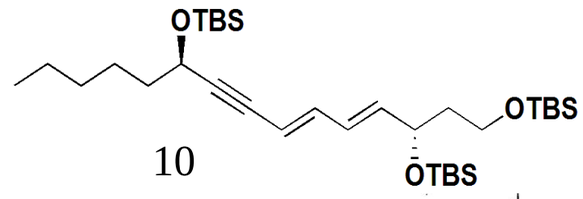
HMQC sur lodg50/5/1
sample dg50aF1 - 45mg
AMX300, 303K, CDCl3



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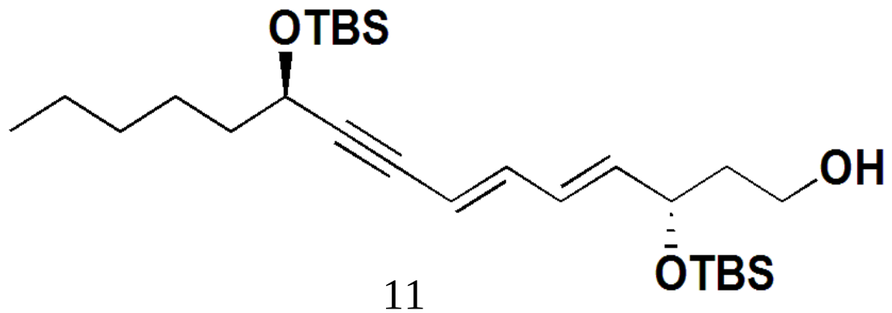
HMBC sur lodg50/6/1
sample dg50aF1
AMX300, 303K, CDCl3

dg51af1 06.11.12 1H sur
AMX300, 303K, CDCl3



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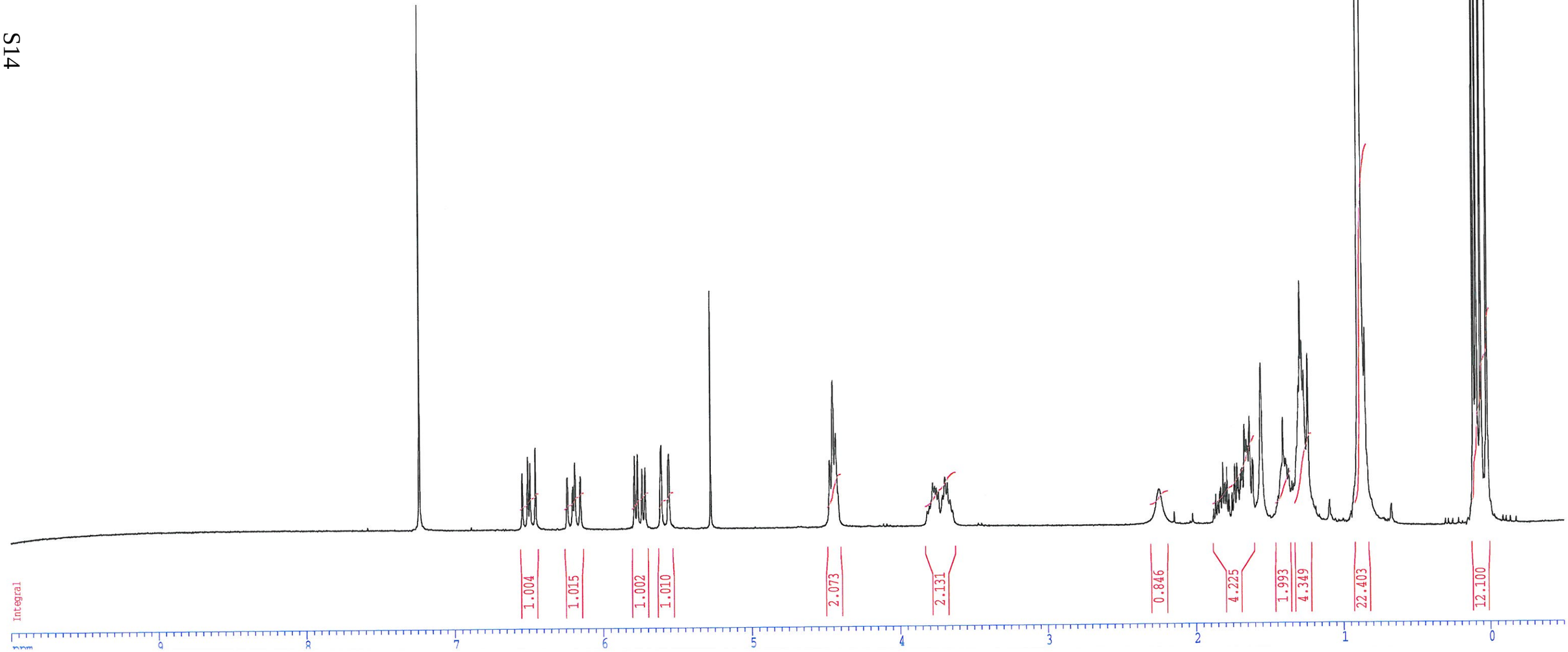
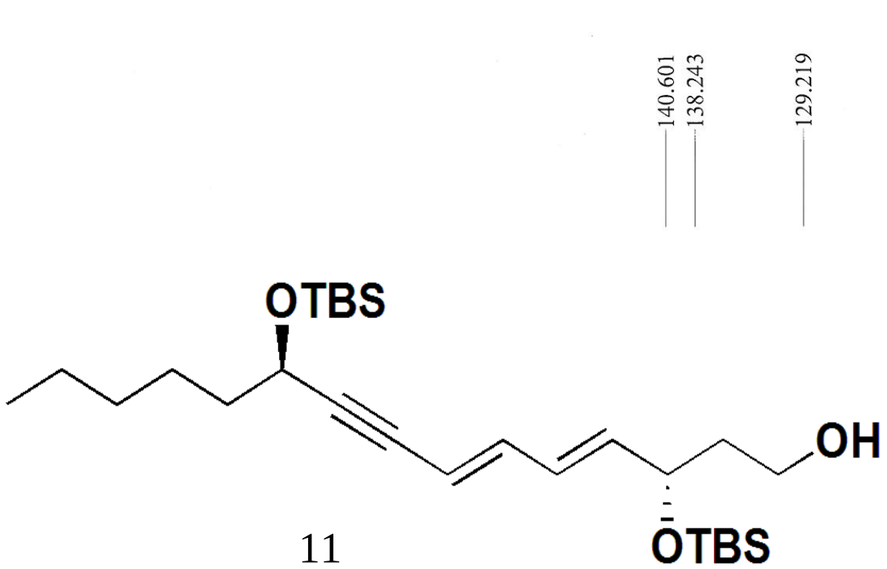
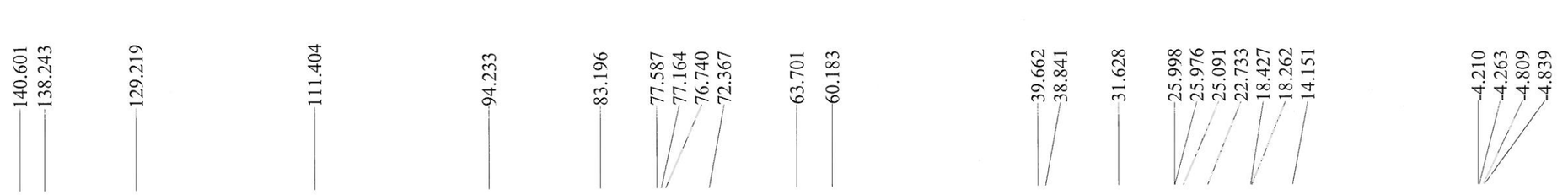



OTBS
OH
OTBS

11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129.219
111.404
94.233
83.196
77.587
77.164
76.740
72.367
63.701
60.183
39.662
38.841
31.628
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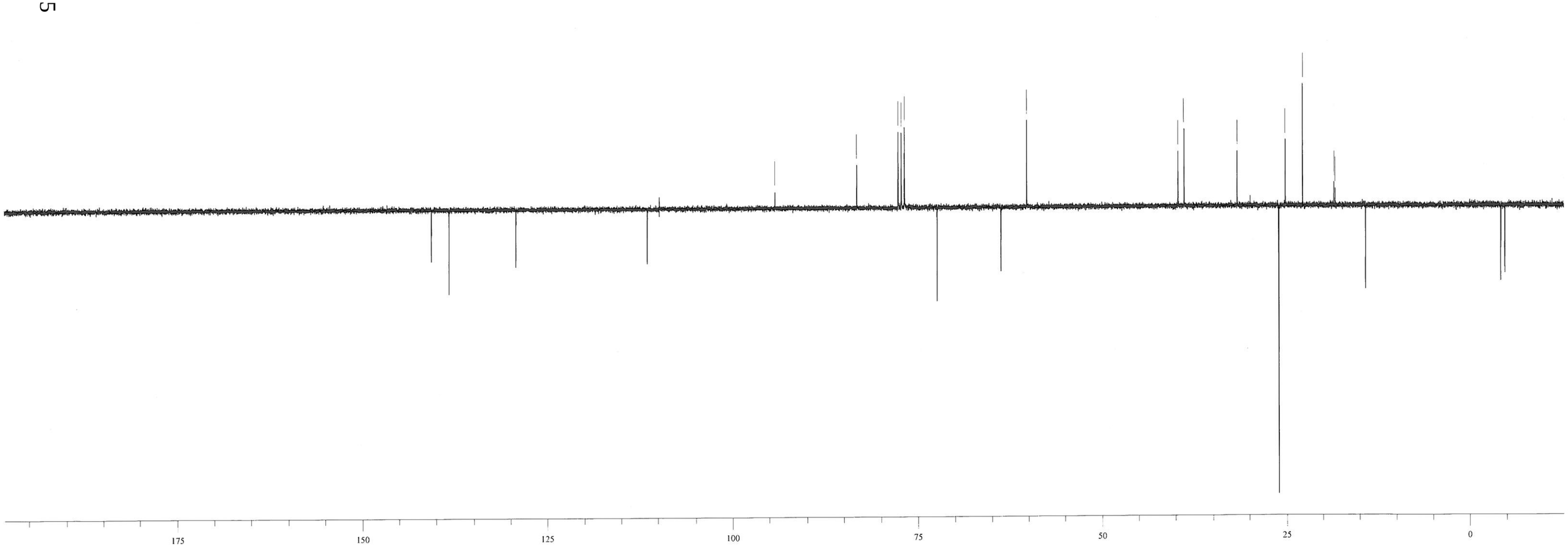
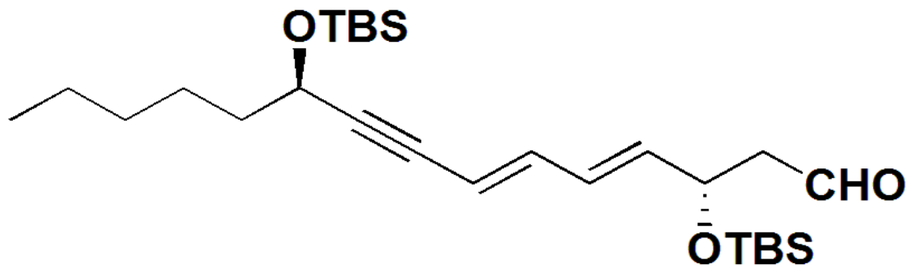
25.976
25.091
22.733
18.427
18.262
14.151
4.210
4.263
4.809
4.839


175
150
125
100
75
50
25
0

dg52gf1 19.11.2013 1H sur
AMX300, 303K, CDCl3



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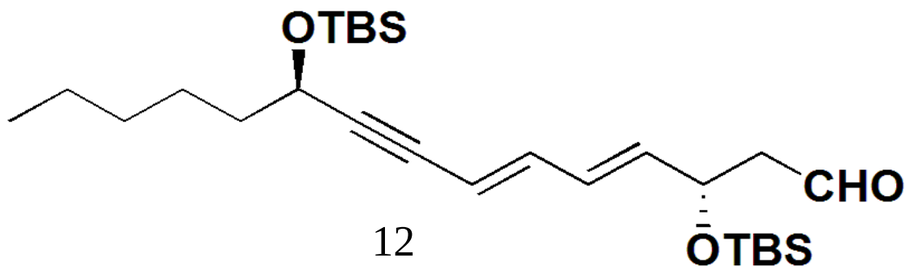

12

Jmod sur dg52c 23.11.12 /7/1
AMX300, 303K, CDCl3

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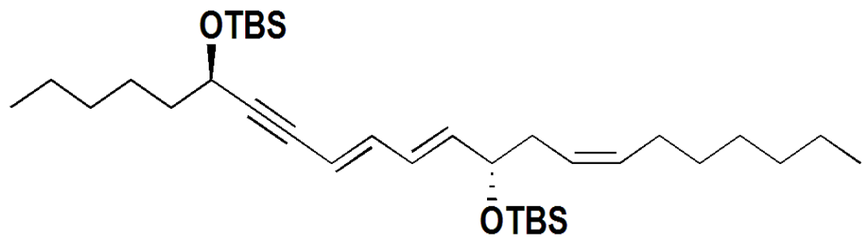
137.107
129.558
112.050
94.505
83.055
77.154
77.579
76.724
68.737
63.659
51.565
38.789
31.593
25.970
25.858
25.056
22.702
18.391
18.211
14.123
-4.239
-4.293
-4.838
-4.868

225 200 175 150 125 100 75 50 25 0

dg53af1 19.11.12 1H sur
AMX300, 303K, CDCl3



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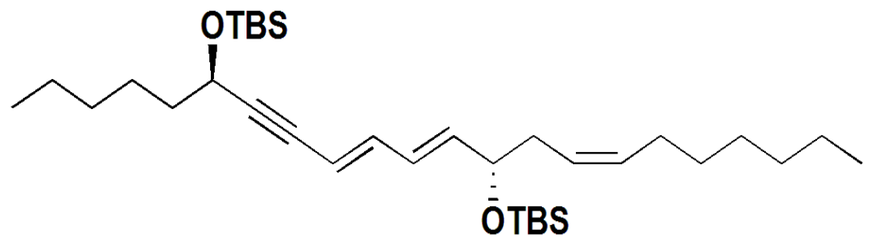
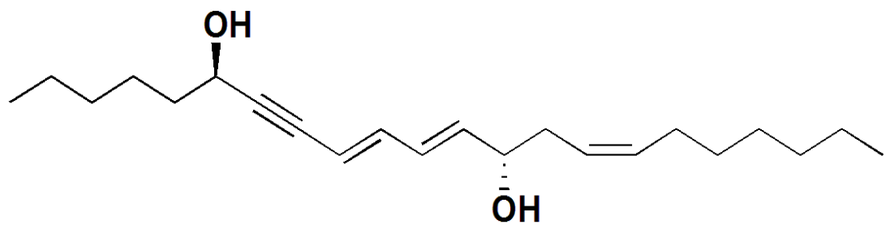


14

Jmod sur lodg53/7/1
sample dg53aF1

141.061
139.276
132.313
128.607
125.005
110.730
93.830
83.384
77.153
72.983
77.571
76.725
63.709
38.864
36.436
31.928
31.628
29.729
29.152
27.640
27.378
26.022
25.100
22.786
22.726
18.414
14.228
14.146
4.261
4.344
4.613
4.823

dg54af1 21.11.12 1H sur
AMX300, 303K, CDCl3



15

141.368
138.309
134.230
129.386
123.995
110.993
93.075
83.991
77.580
77.153
76.732
71.739
63.195
37.989
35.428
31.871
31.601
29.689
29.106
27.602
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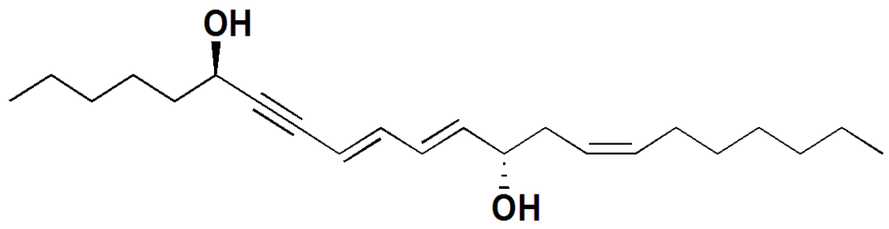
22.750
22.683
14.198
14.116



15

Jmod sur lodg54/7/1
sample dg54aF1 - 25mg

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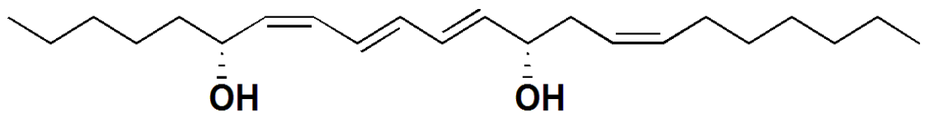
150
125
100
75
50
25
0

dg61af4 08.02.2013 1H sur
AMX300, 303K, CDCl3



3

136.754
134.470
134.095
133.909
130.390
130.060
127.864
124.204

77.578
77.154
76.724
72.053
68.257

37.644
35.530
31.901
29.849
29.729
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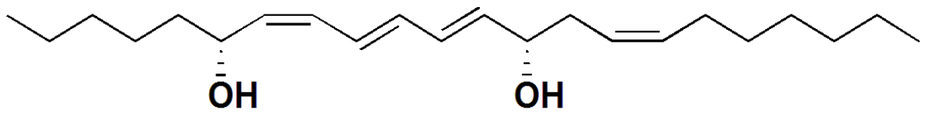
14.153

dg61af4 08.02.2013 Jmod sur
AMX300, 303K, CDCl3



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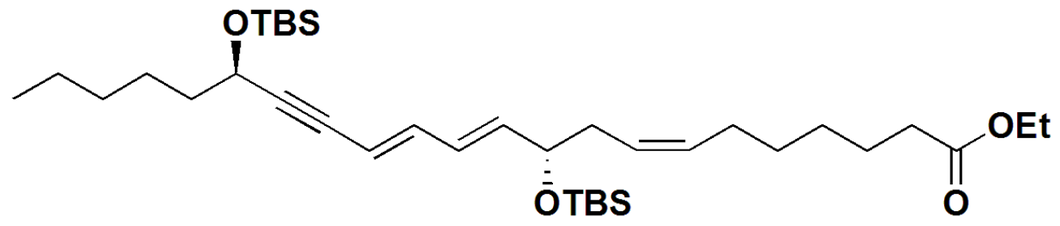
175
150
125
100
75
50
25
0

dg59df2 05.02.2013 1H sur
AMX300, 303K, CDCl3



17

S24

lodg59_500MHz 7 1 C:\Bruker\TOPSPIN topspin

jmod sur lodg59/7/1
d1=1.5s
AV500, CDCl3, 295.6K

173.9613
141.0141
139.0720
131.8583
128.6676
125.4075
110.8414
93.7993
83.3772
77.4138
77.1604
76.9050
72.9109
63.6369
60.2365
38.8634
36.3945
34.4742
31.6394
29.3838
28.9571
27.4025
26.0309
25.0479
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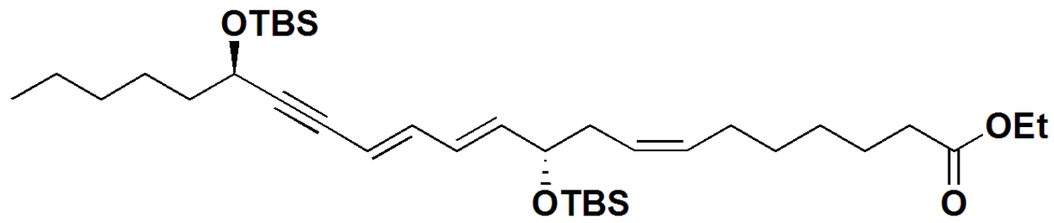
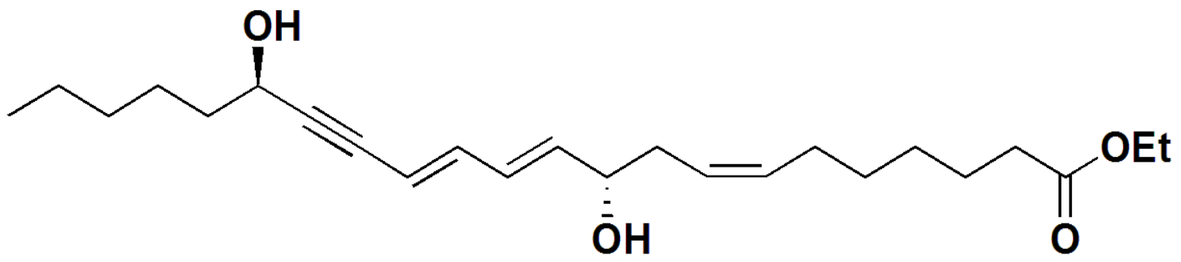
18.4106
14.4176
14.1737
-4.2780
-4.3575
-4.6303
-4.8394



17

200
150
100
50
0
[ppm]

dg60bf1 06.02.2013 1H sur
AMX300, 303K, CDCl3



18

S26

lodg60aF1_500MHz 7 1 C:\Bruker\TOPSPIN topspin

jmod sur lodg60aF1/7/1
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AV500, CDCl3, 294.8K

174.0737
141.3825
138.2567
133.7791
129.3437
124.3169
110.9686
93.1023
83.9494
77.4142
77.1611
76.9043
71.6916
63.1838
60.4016
38.0232
35.4426
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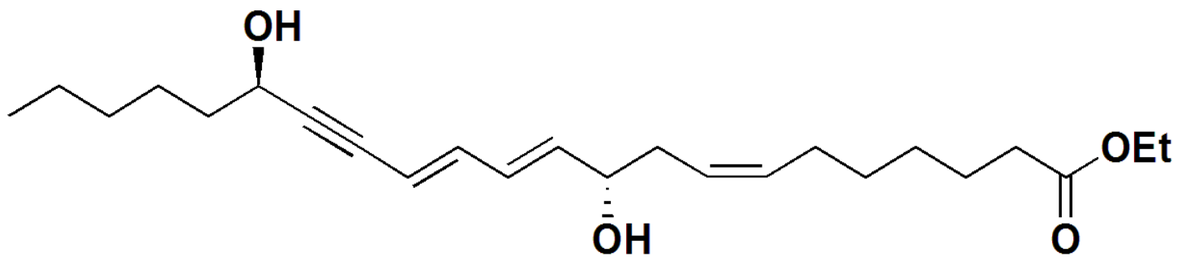
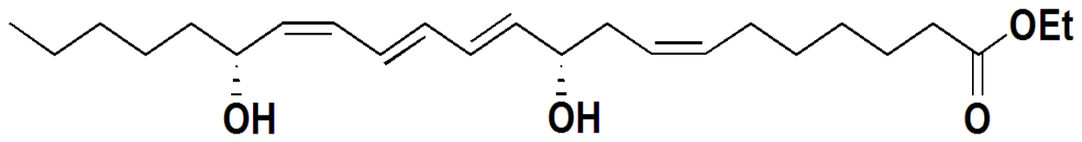
31.6524
29.3944
28.9105
27.4186
24.9993
22.7413
14.4351
14.1932



18

200 150 100 50 0 [ppm]

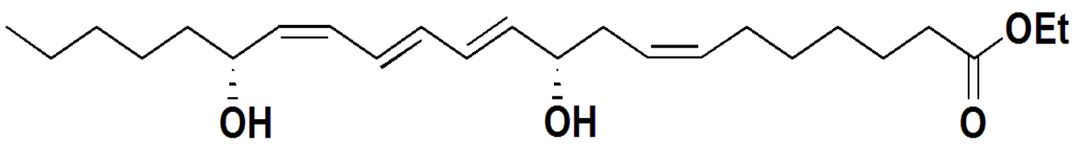
dg62af3 13.02.2013 1H sur
AMX300, 303K, CDCl3



2

173.949
136.709
134.507
133.872
133.565
130.389
130.030
127.896
124.560
77.570
77.154
76.724
71.994
68.235
60.341
37.636
35.533
34.452
31.922
29.340
28.897
27.375
25.139
24.981
22.734
14.400
14.153

dg62af3 13.02.2013 Jmod sur
AMX300, 303K, CDCl3



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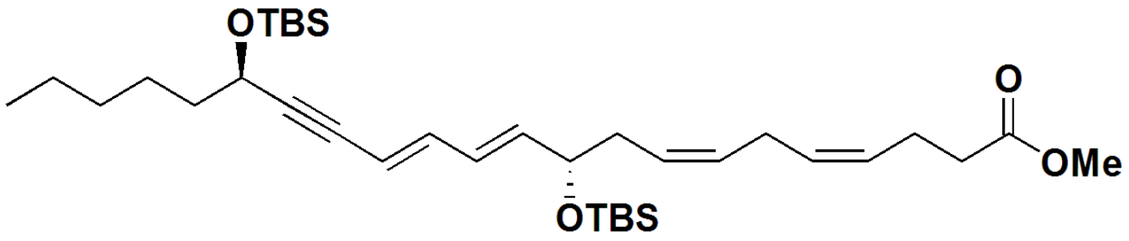

OTBS
O
OMe
OTBS

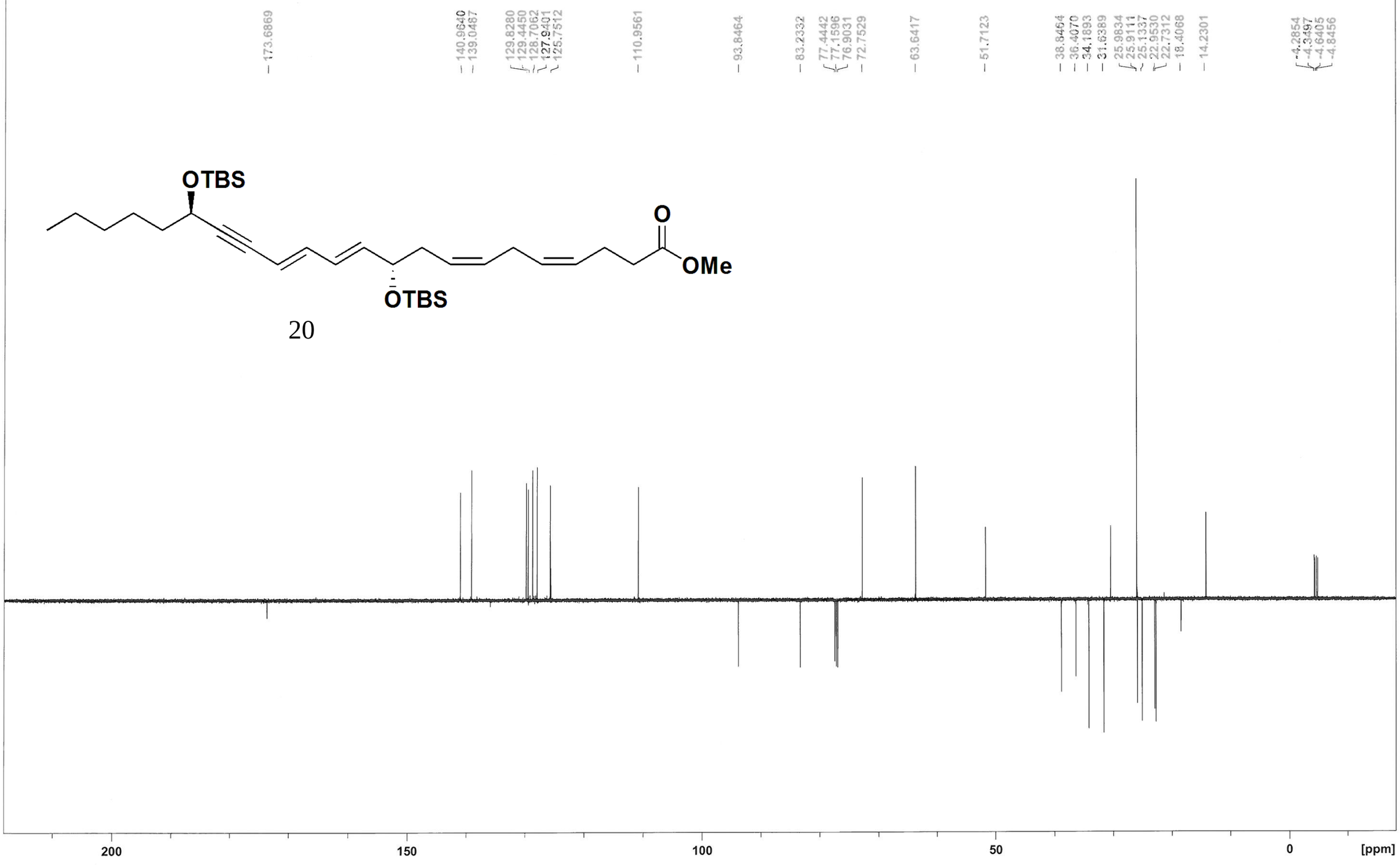
20

1H sur lodg113/1/1
sample lodg113aF1
AMX300, 303K, CDCl3
Integral
1.000
1.038
0.998
1.002
4.045
1.067
1.008
3.011
2.004
6.020
2.081
6.748
22.072
12.012
ppm
9
8
7
6
5
4
3
2
1
0

S30

"dg113 500 MHz" 1 1 C:\Bruker\TOPSPIN topspin

dg113af1, CDCl3,
JMOD, 27 nov 2013



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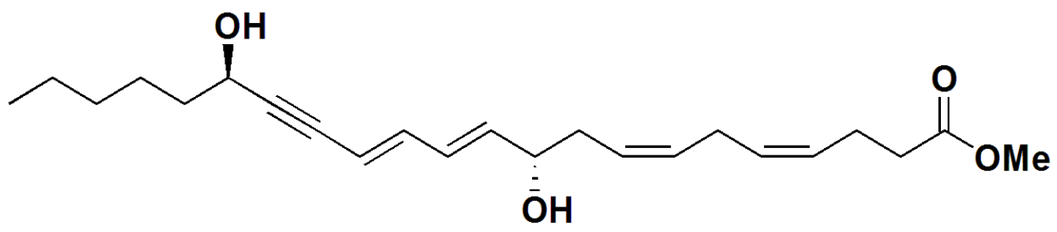

OH
O
OMe
OH

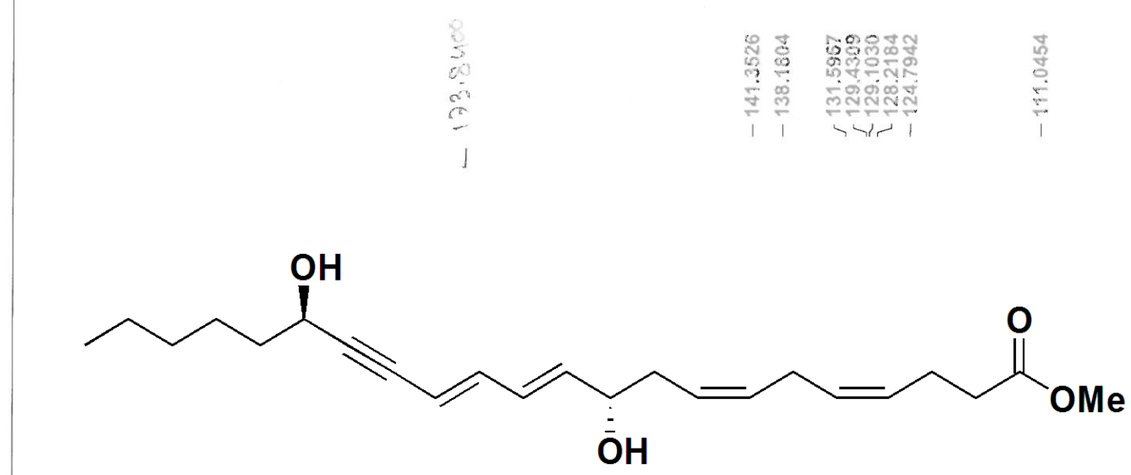
21

1H sur lodg114/1/1
AV500MHZ CDCl3 294K
2 dec2013
9.5
9.0
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
0.0
ppm
0.98
1.00
0.99
0.99
1.03
3.07
1.01
0.98
3.09
2.01
6.02
1.74
3.01
2.01
4.01
0.18
3.01

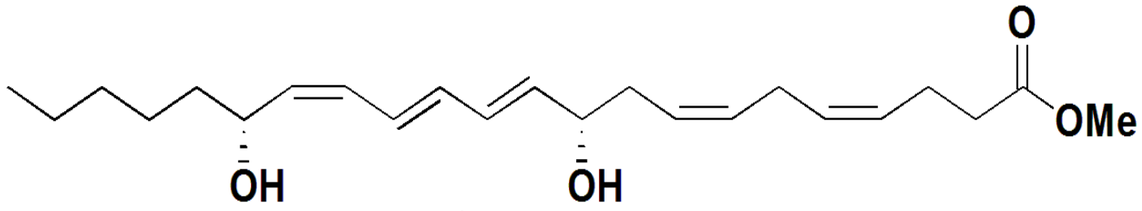
S32

"dg114af1 500 MHz" 2 1 C:\Bruker\TOPSPIN topspin

dg114af1, 500 Mhz, CDCl3



21



1

1H sur dg115af2-2
file3
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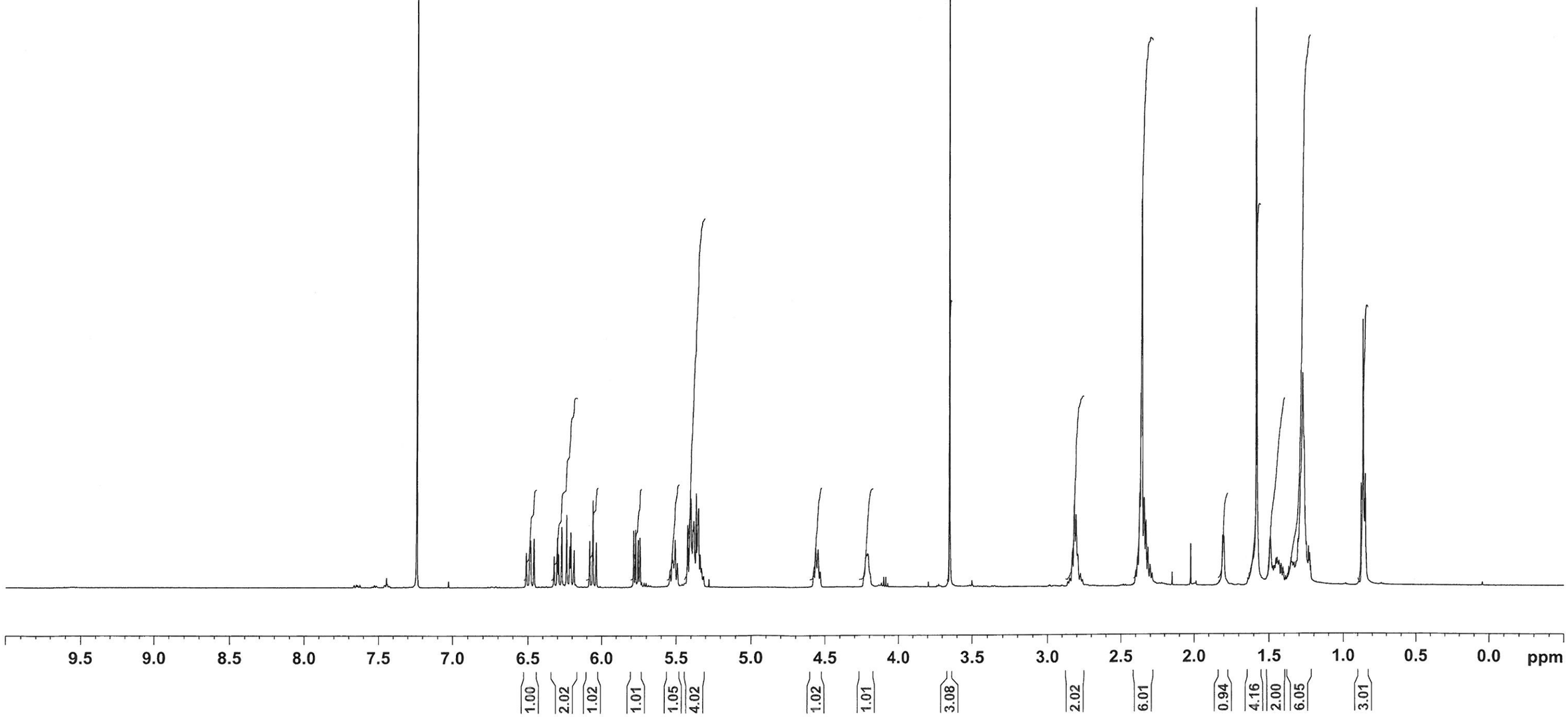

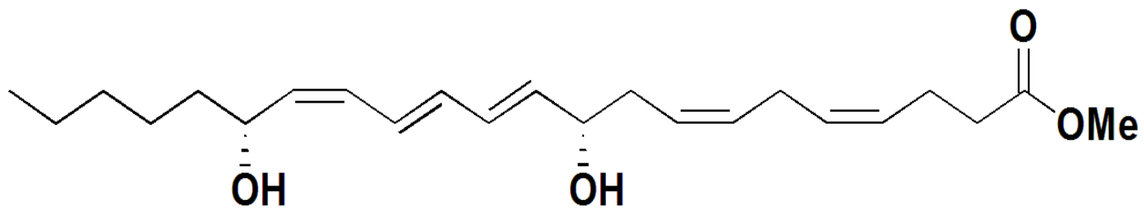

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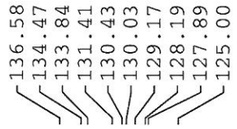

Jmod sur dg114af1
AV500, CDCl3 alumine, 294K