Structural Interrogation of Benzosuberene-Based Inhibitors of Tubulin Polymerization

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[b] Department of Radiology, The University of Texas Southwestern Medical Center, 5323 Harry Hines Boulevard, Dallas, Texas 75390-9058, United States

[c] Screening Technologies Branch, Developmental Therapeutics Program, Division of Cancer Treatment and Diagnosis, National Cancer Institute, Frederick National Laboratory for Cancer Research, National Institutes of Health, Frederick, MD 21702, United States

[d] OXiGENE Inc., 701 Gateway Boulevard, Suite 210, South San Francisco, California 94080, United States

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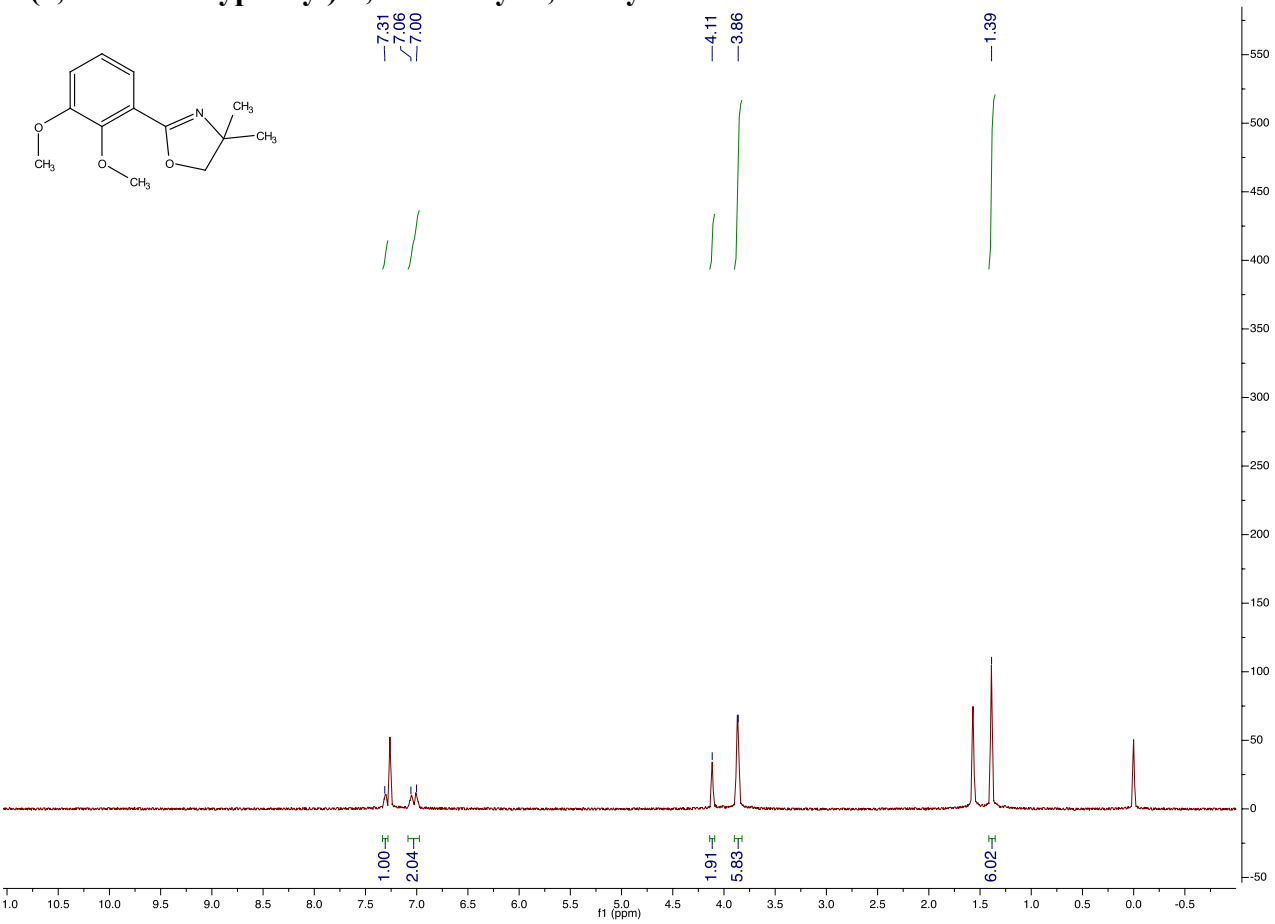
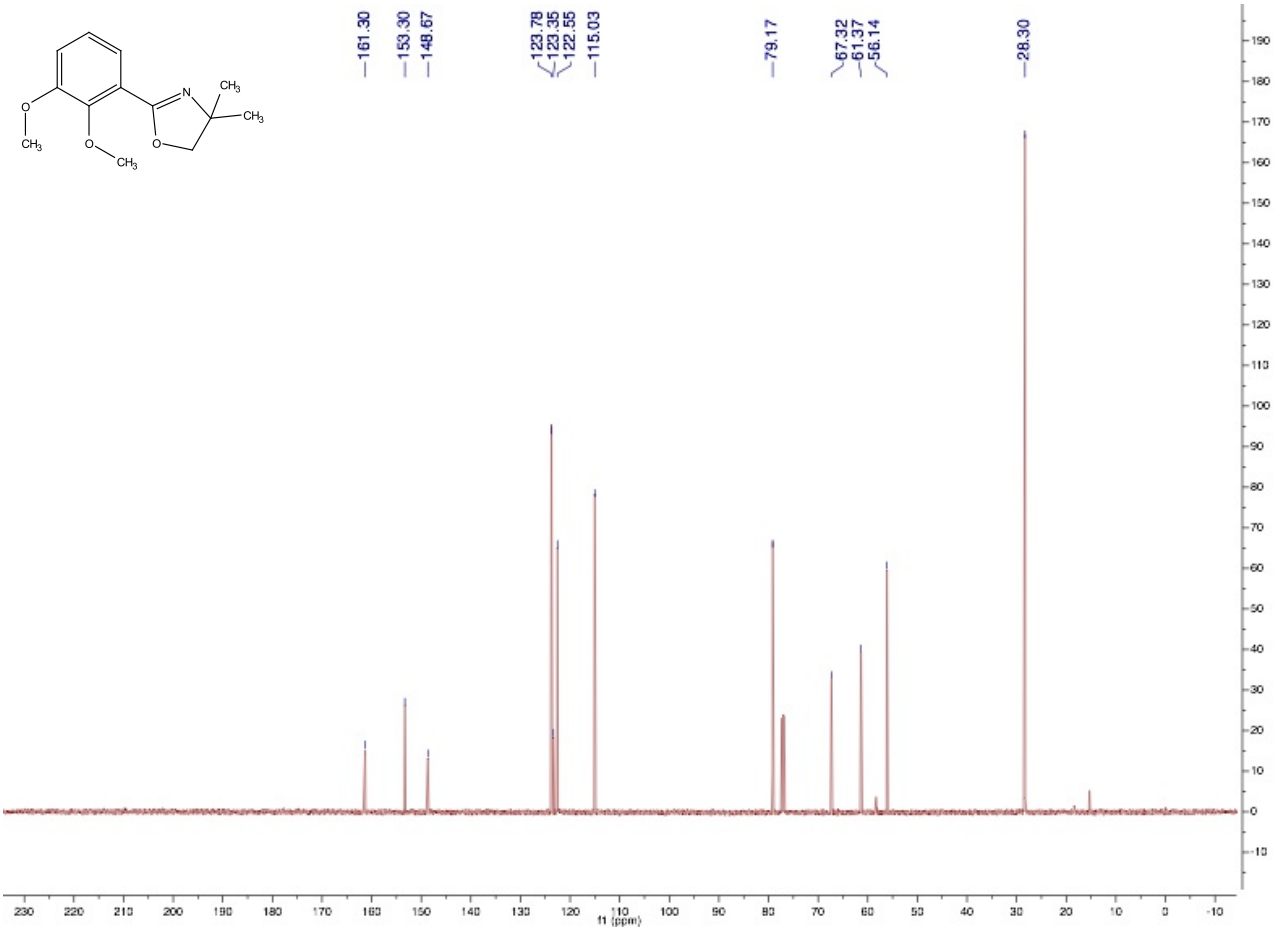
E-mail address: Kevin_Pinney@baylor.edu (K.G. Pinney).

Supplementary Data Table of Contents

2-(2,3-dimethoxyphenyl)-4,4-dimethyl-4,5-dihydrooxazole





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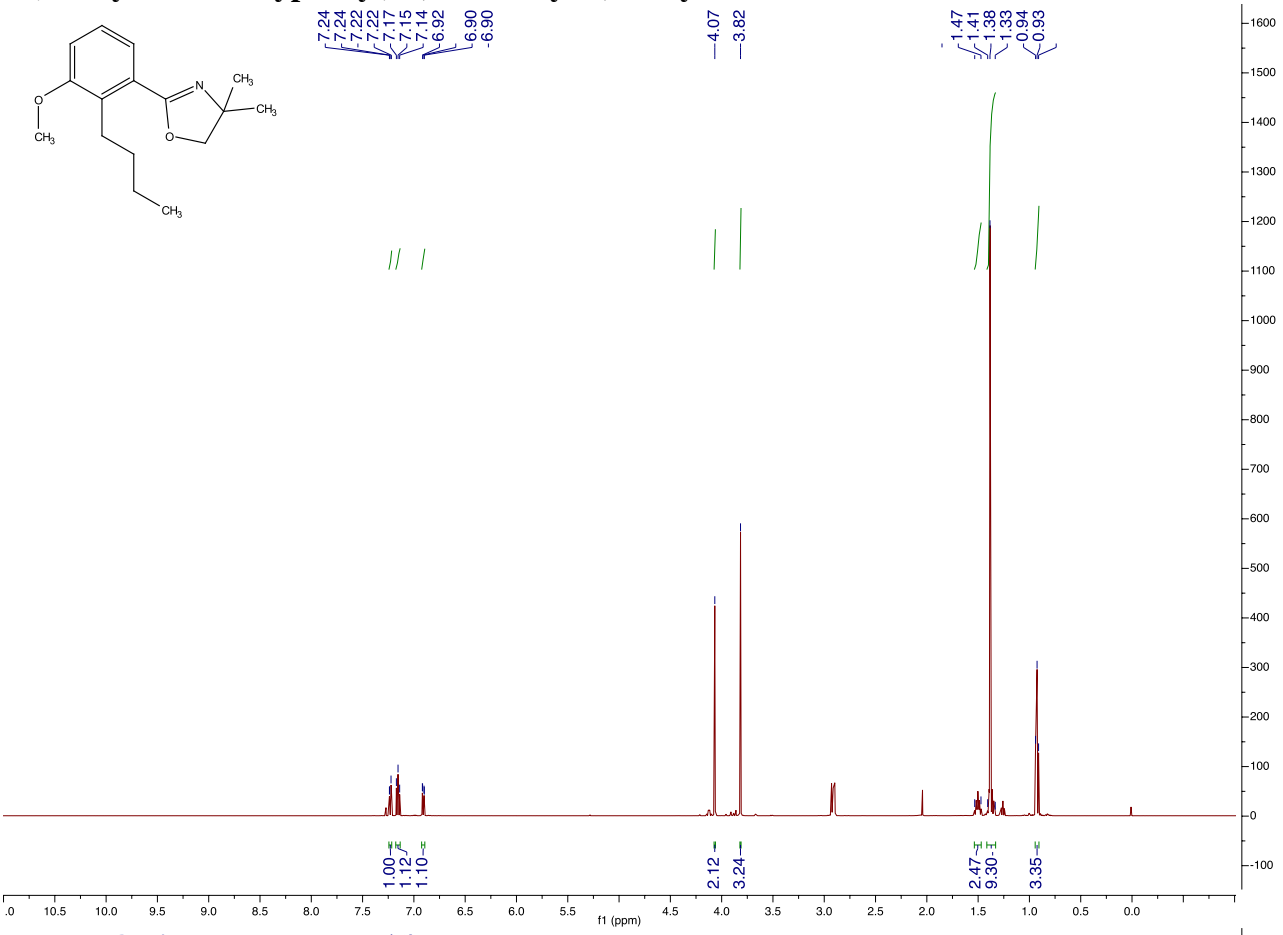

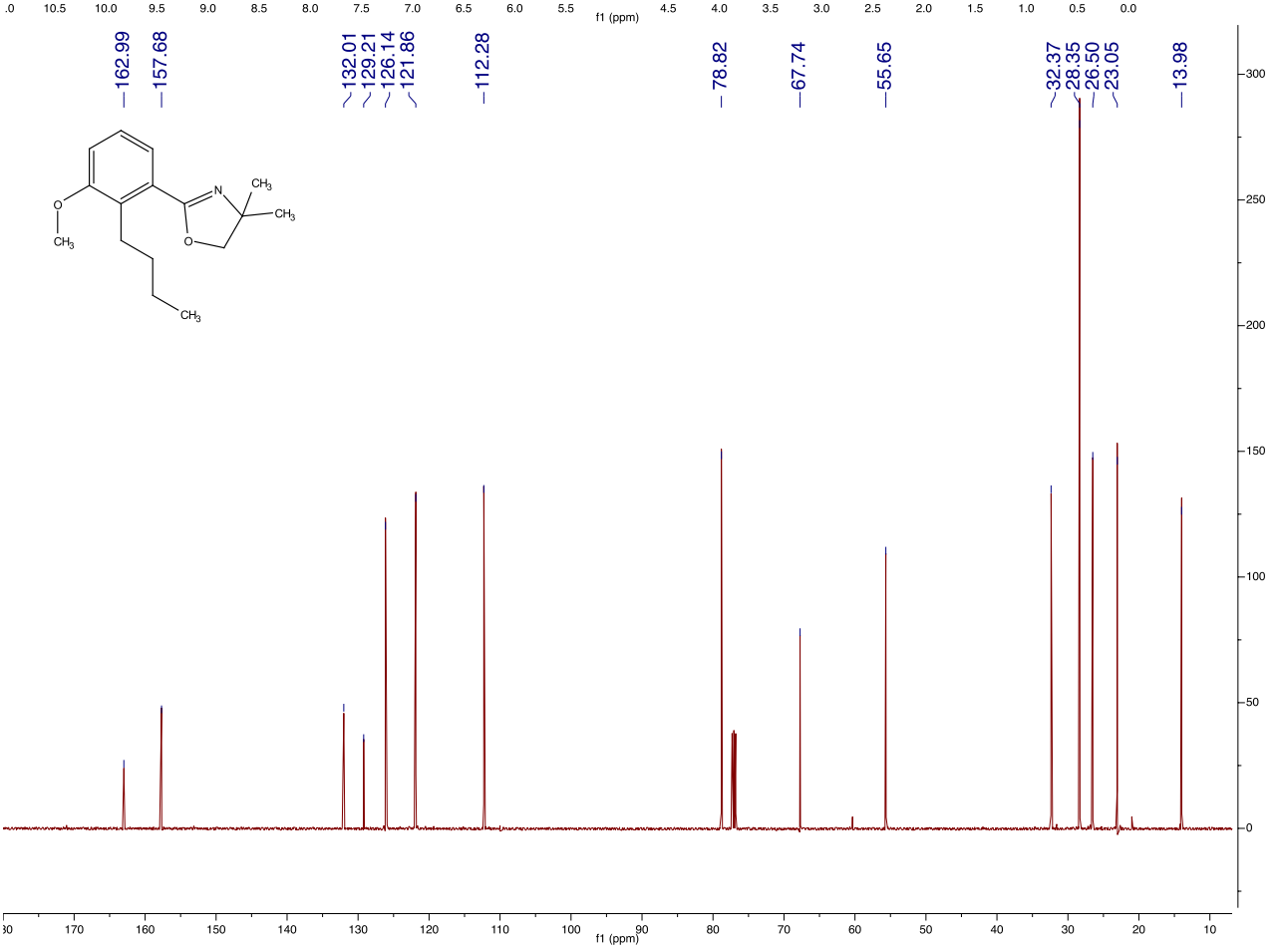

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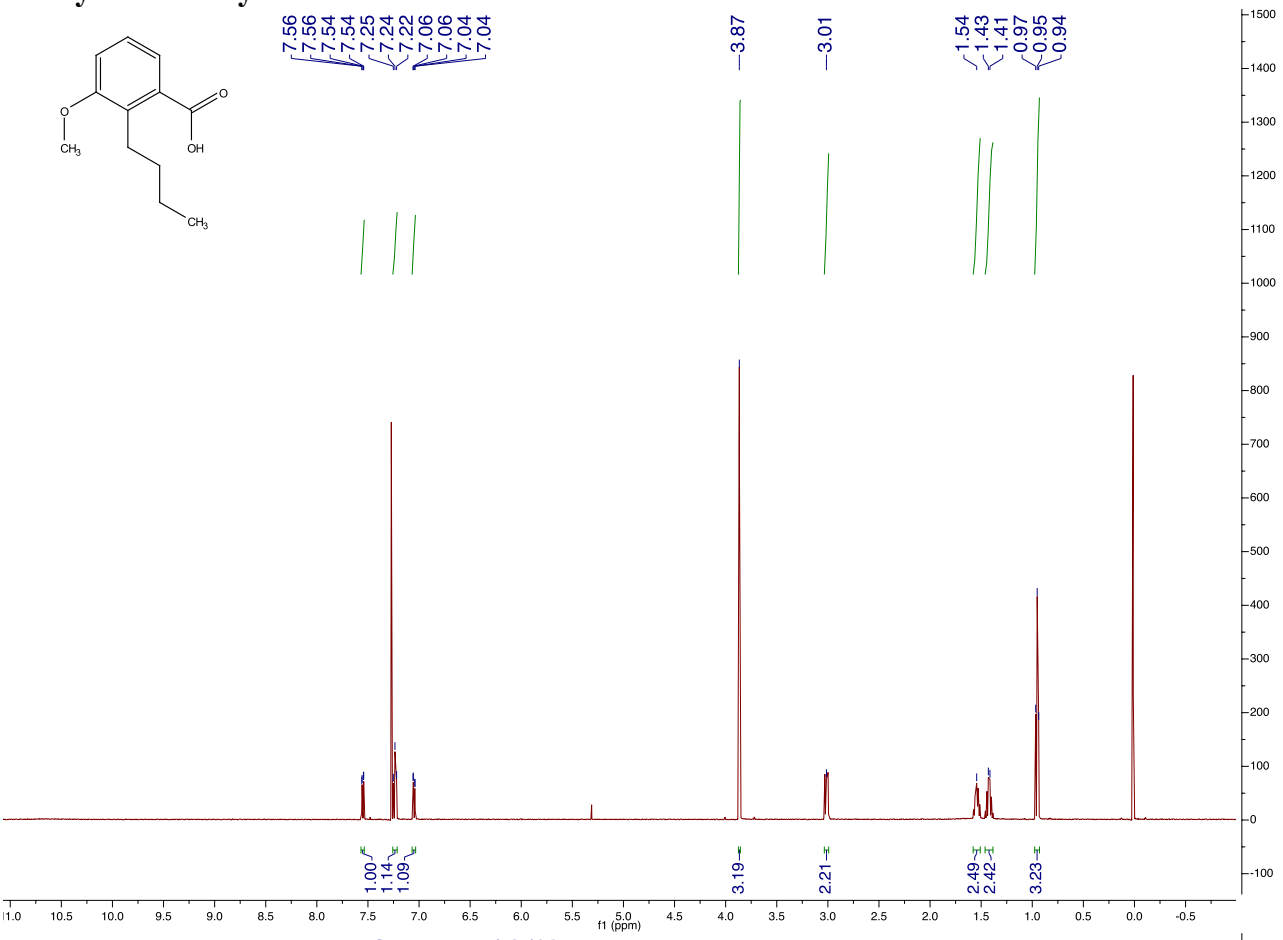

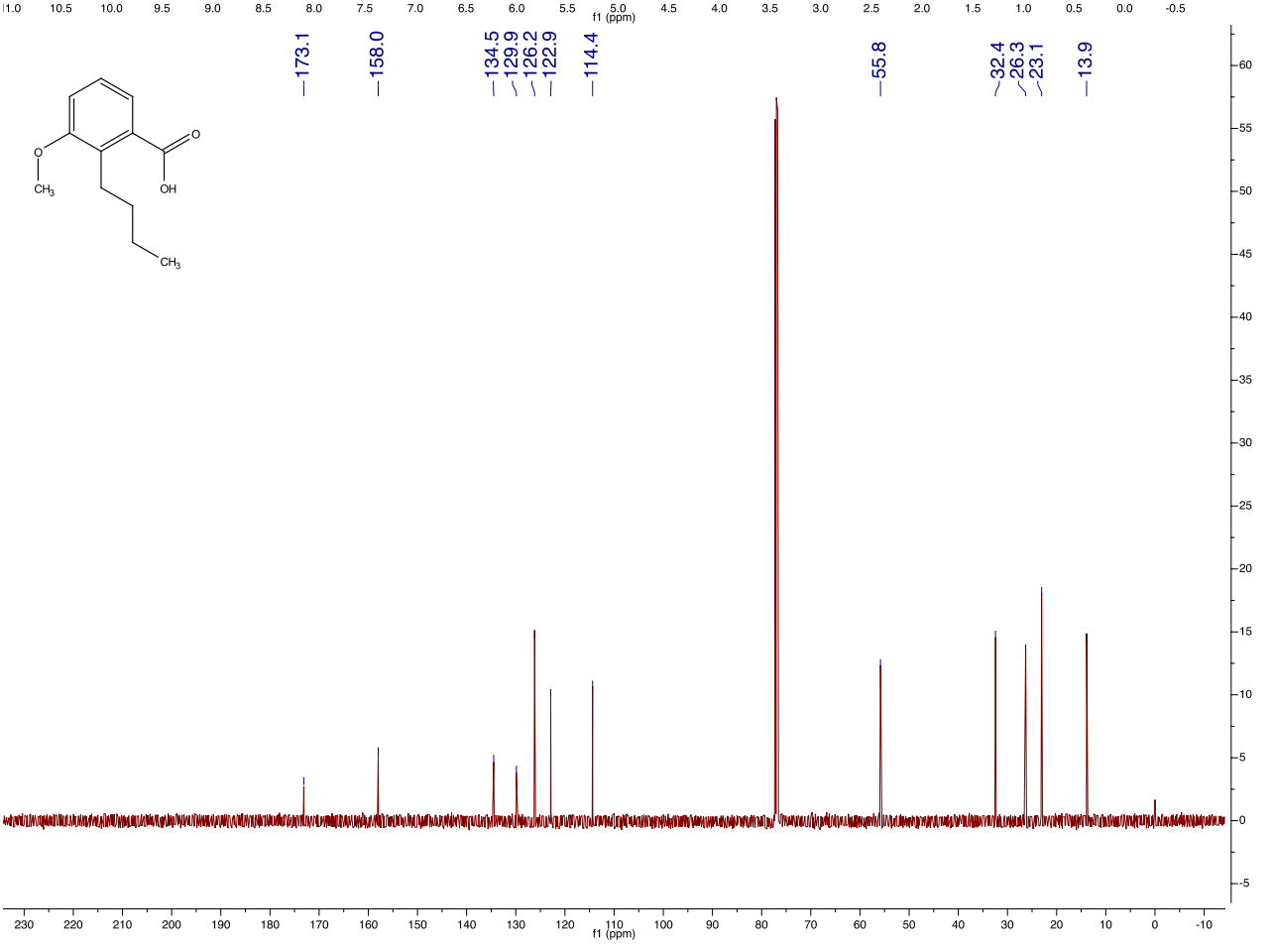

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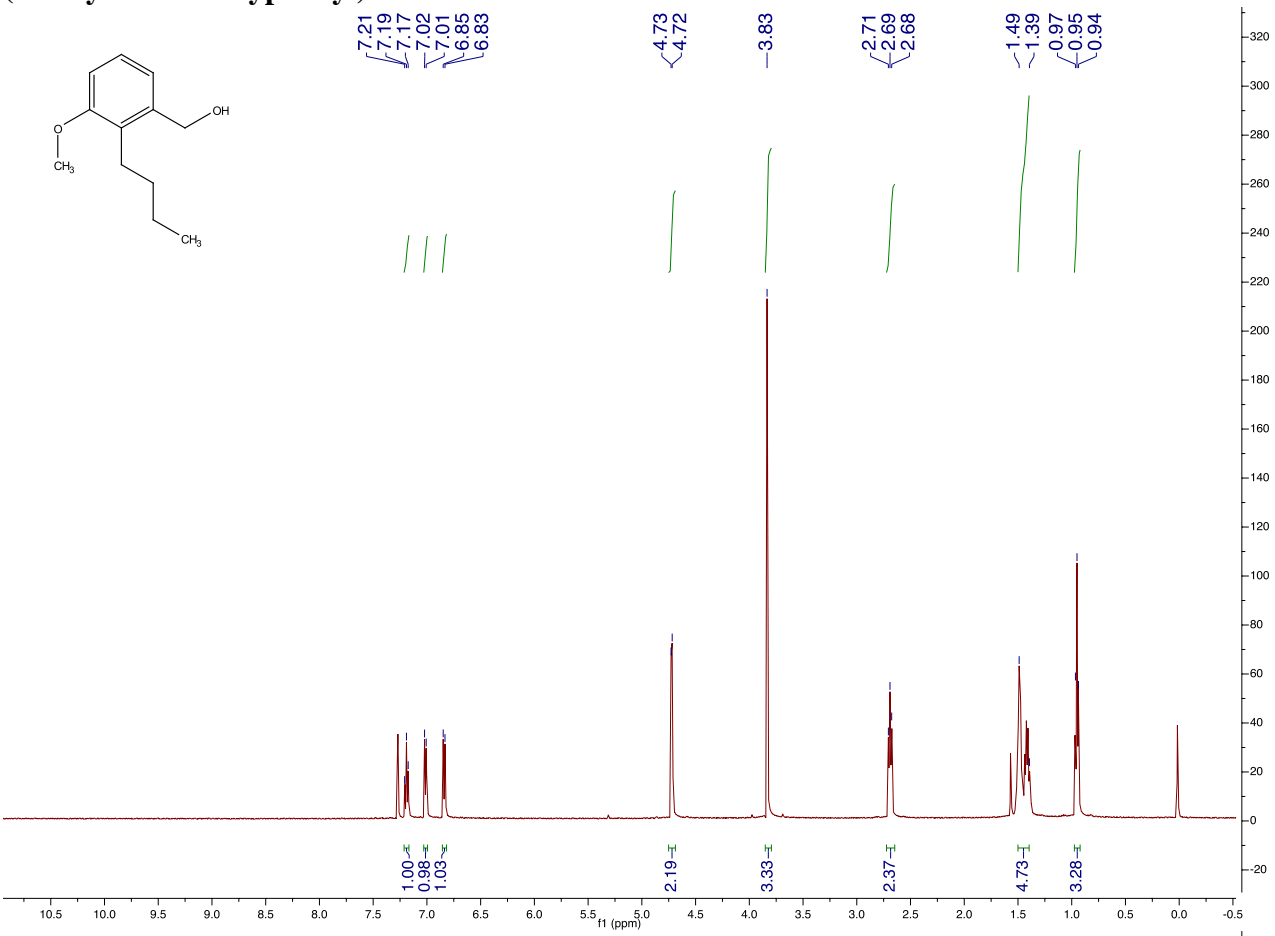

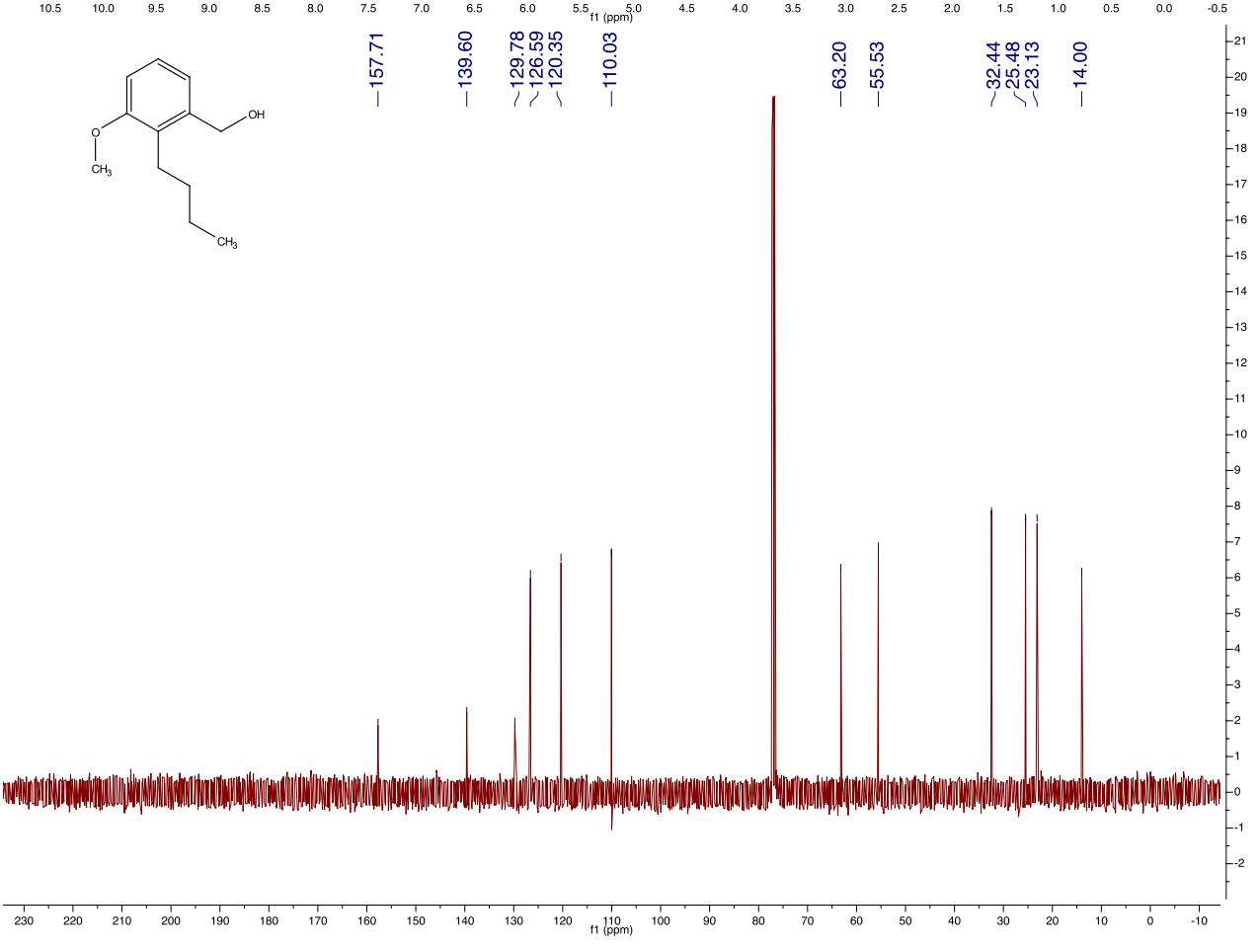

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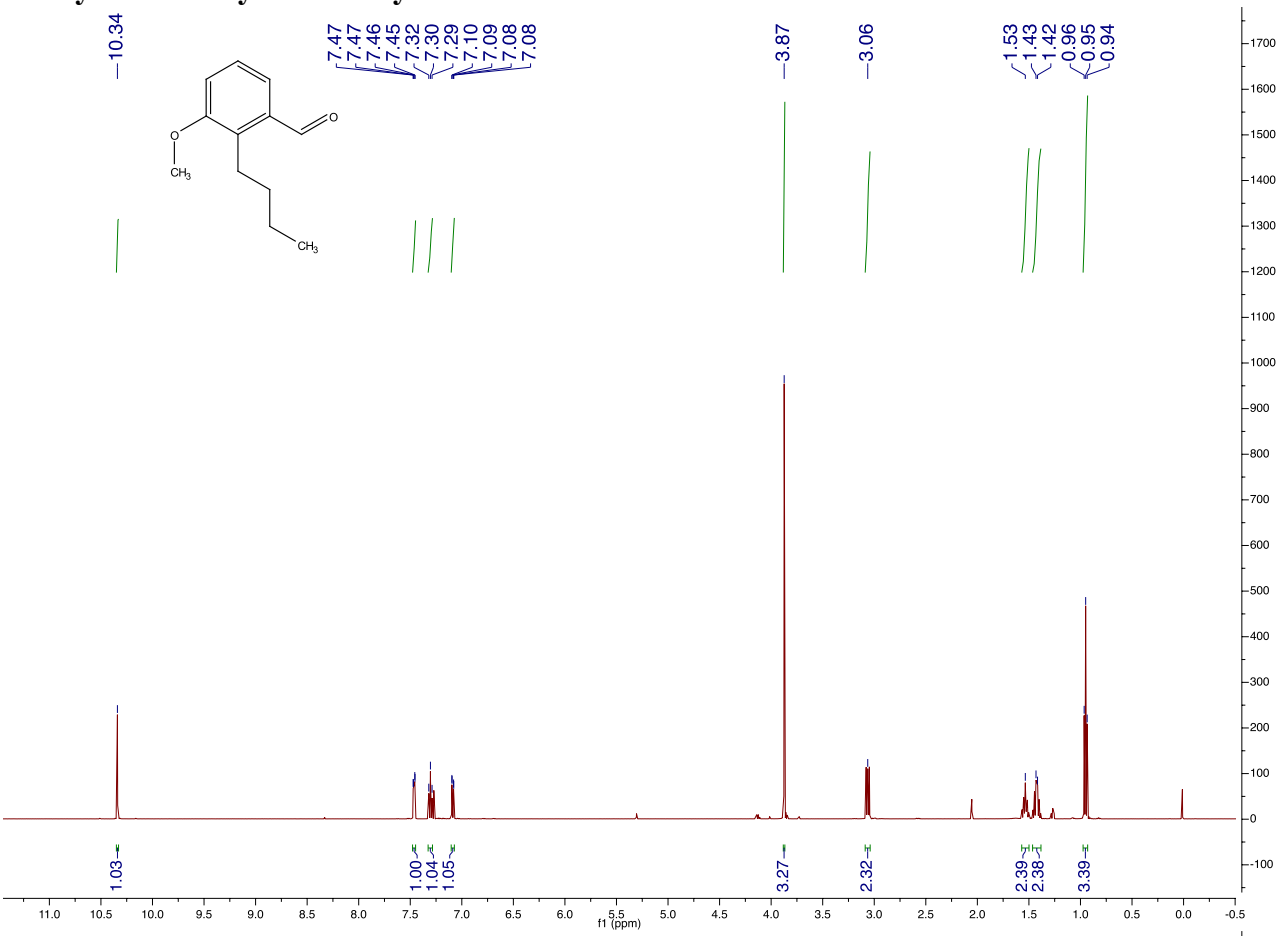
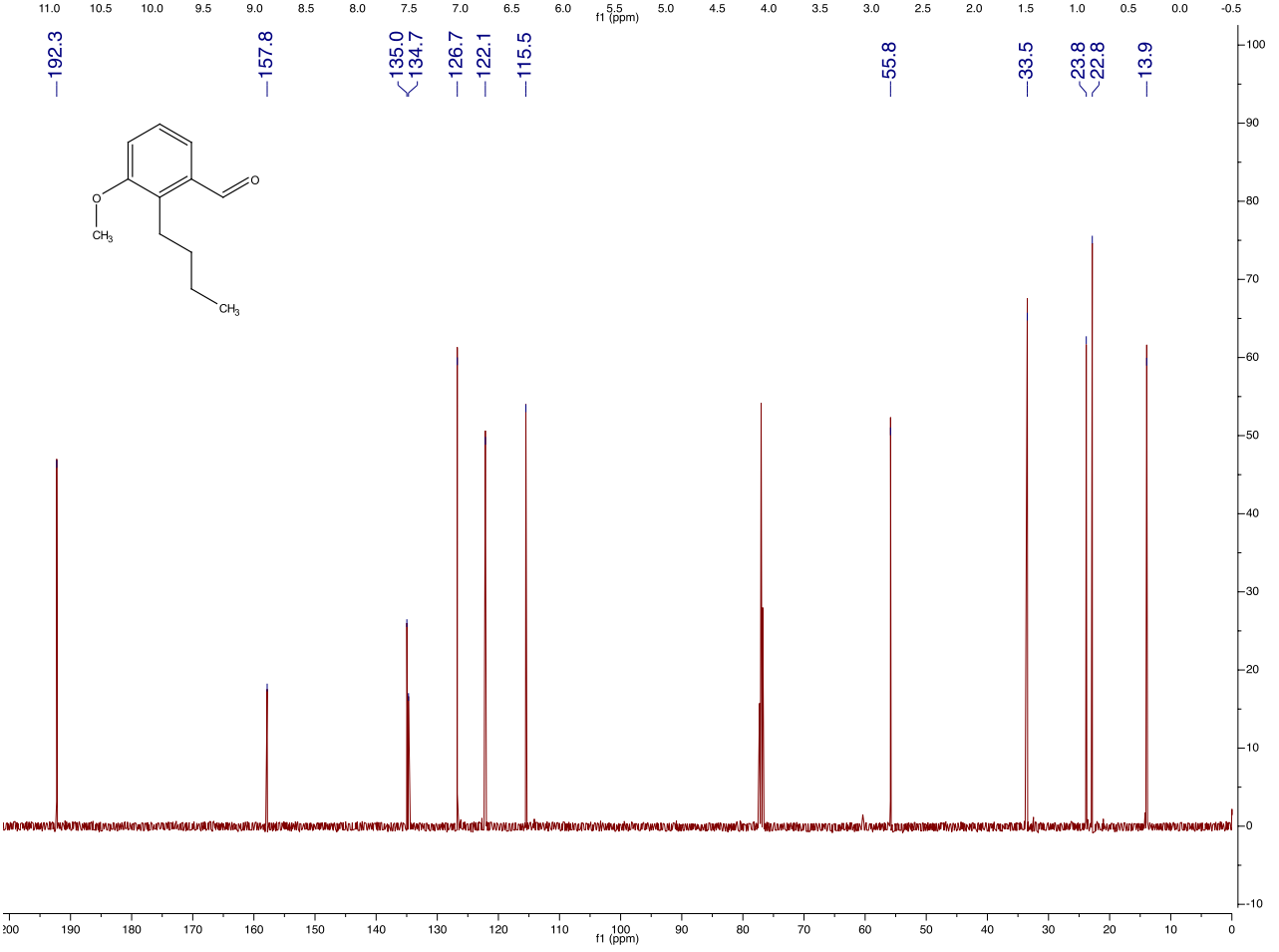
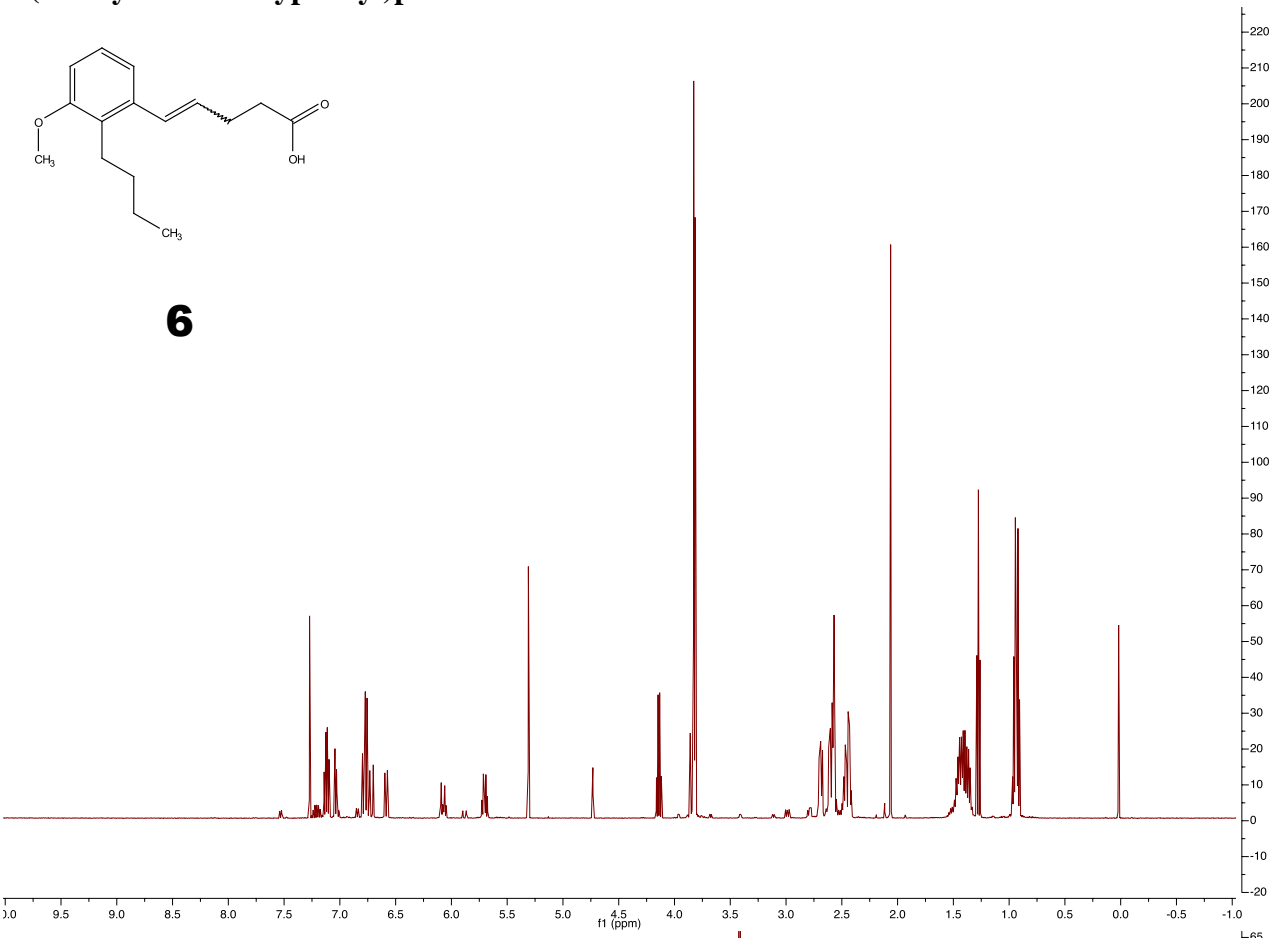
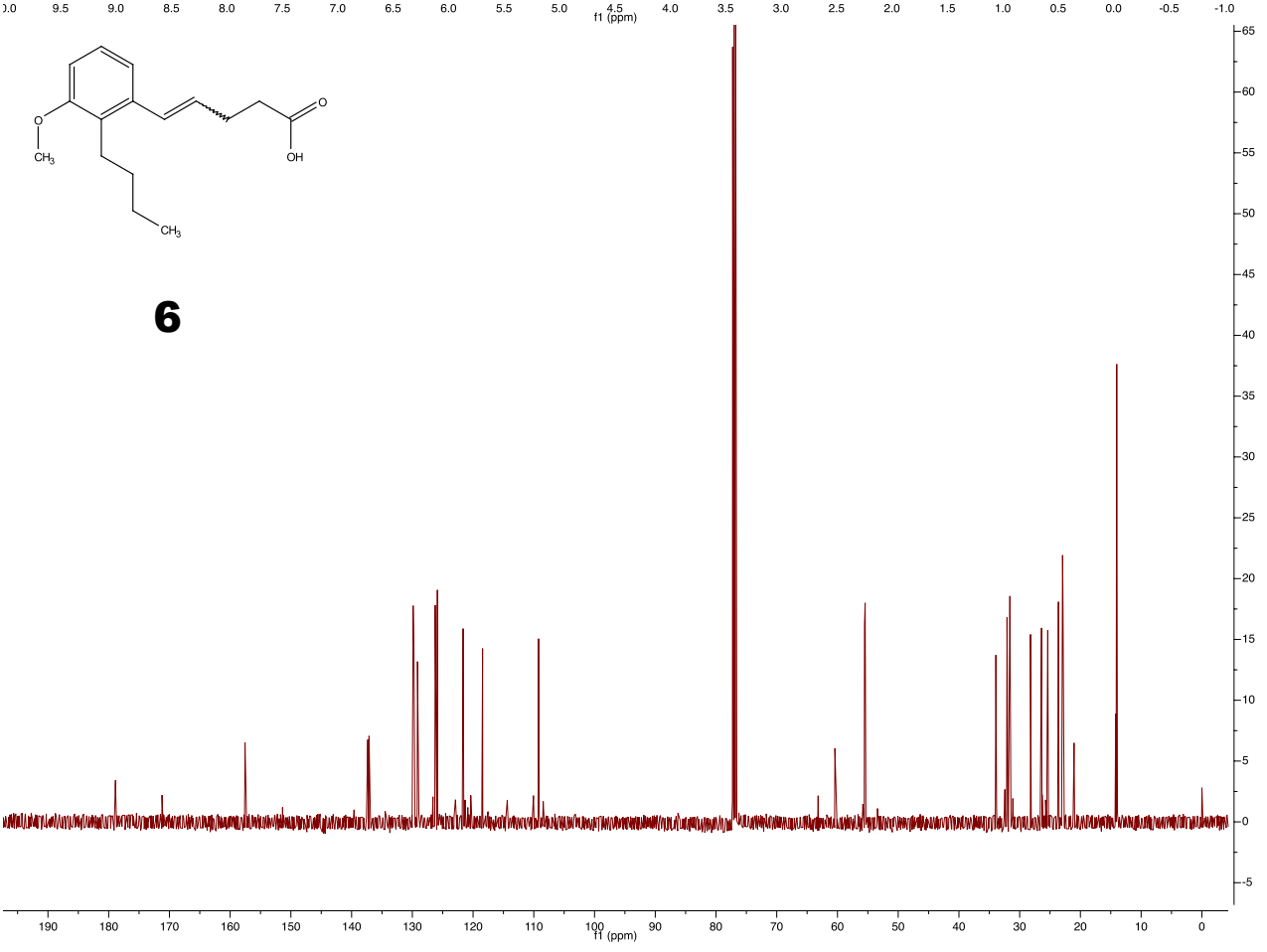
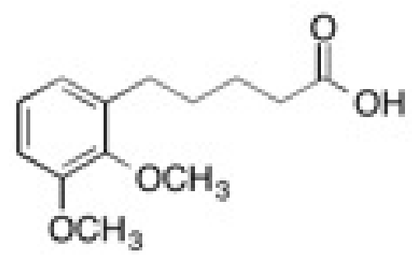




5-(2-butyl-3-methoxyphenyl)pent-4-enoic acid







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121.875
110.249
60.571
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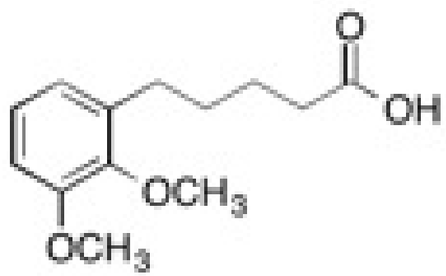
29.439
24.501



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S8

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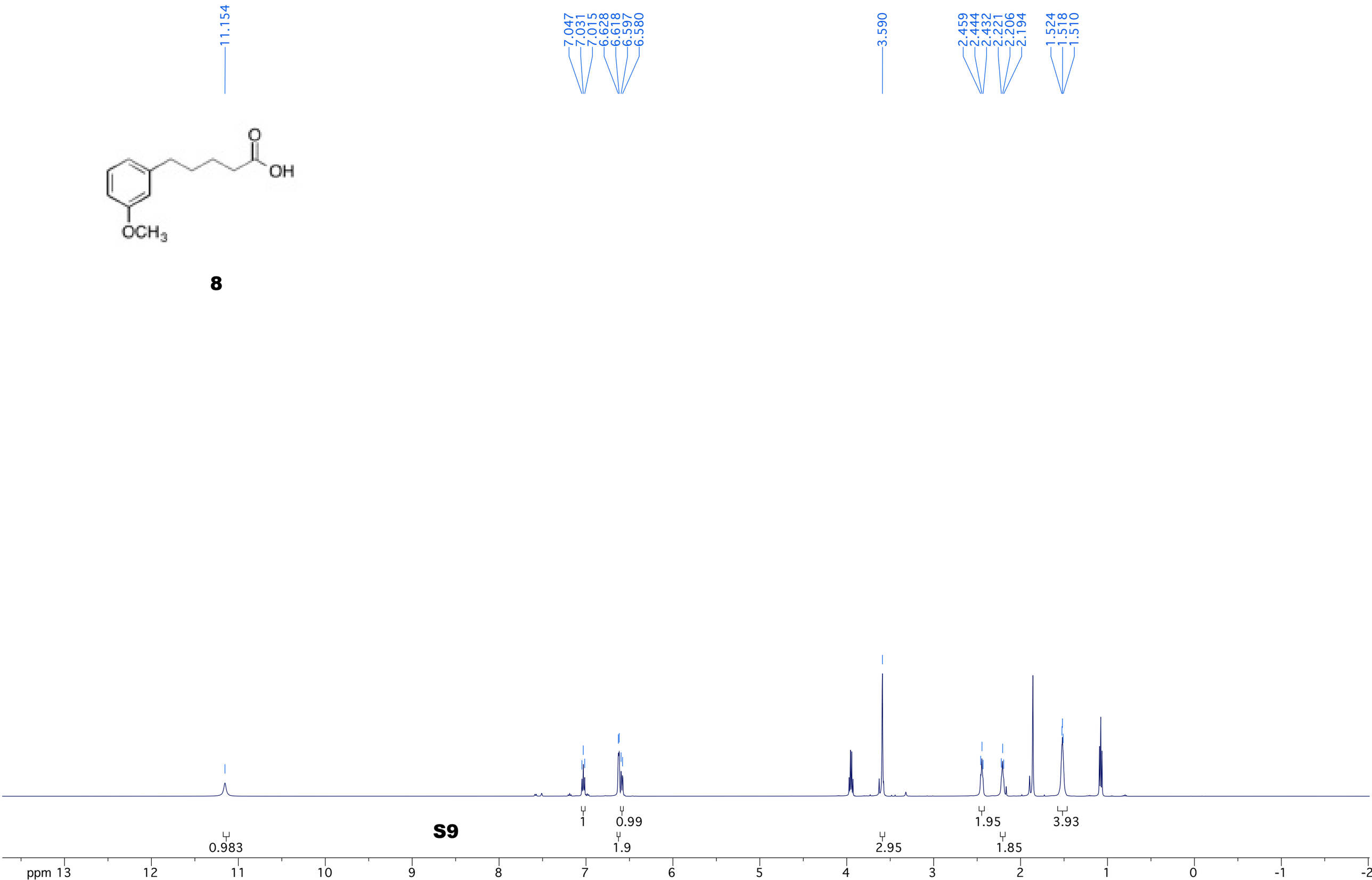
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2.206
2.194
1.524
1.518
1.510
O
OH
OCH3
0.983
1
0.99
1.9
2.95
1.95
1.85
3.93
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12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2

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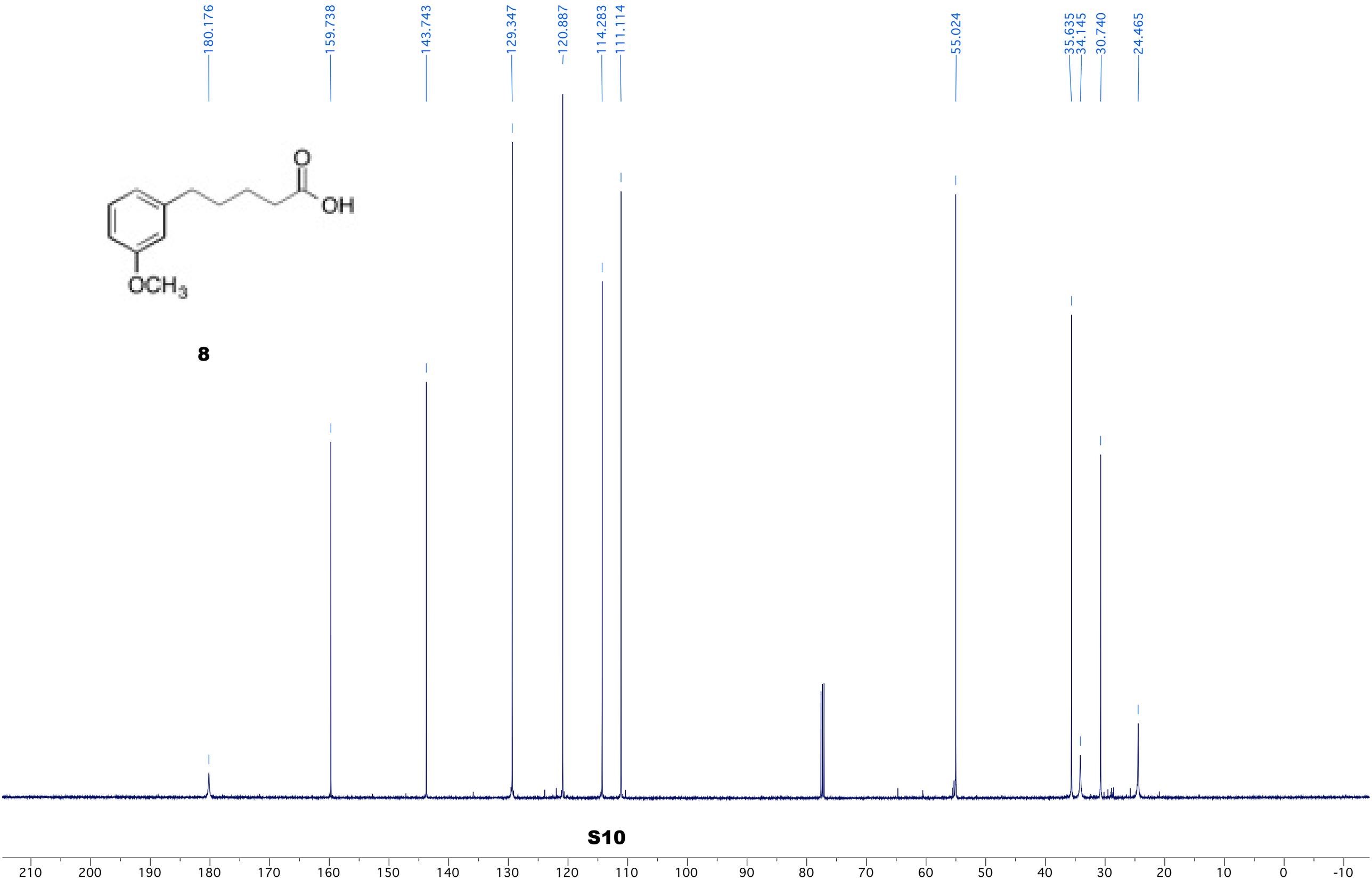
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24.465
O
OH
OCH3
8
S10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

9.969

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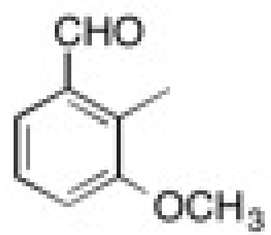
6.966
6.747
6.731

3.572
3.537

2.232



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1.11
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13
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9
8
7
6
5
4
3
2
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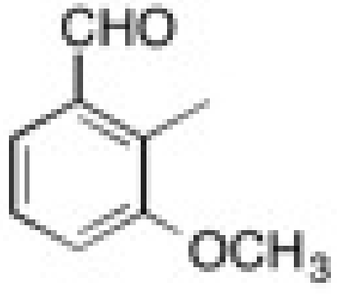

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7.212
6.921
6.906
6.852
6.835

3.926

2.790
2.774
2.759
2.525
2.511
2.496
2.344
2.184
1.887
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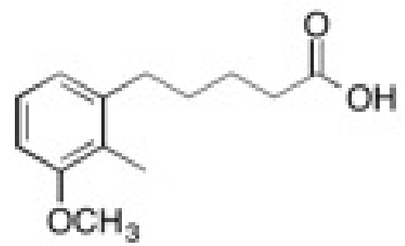

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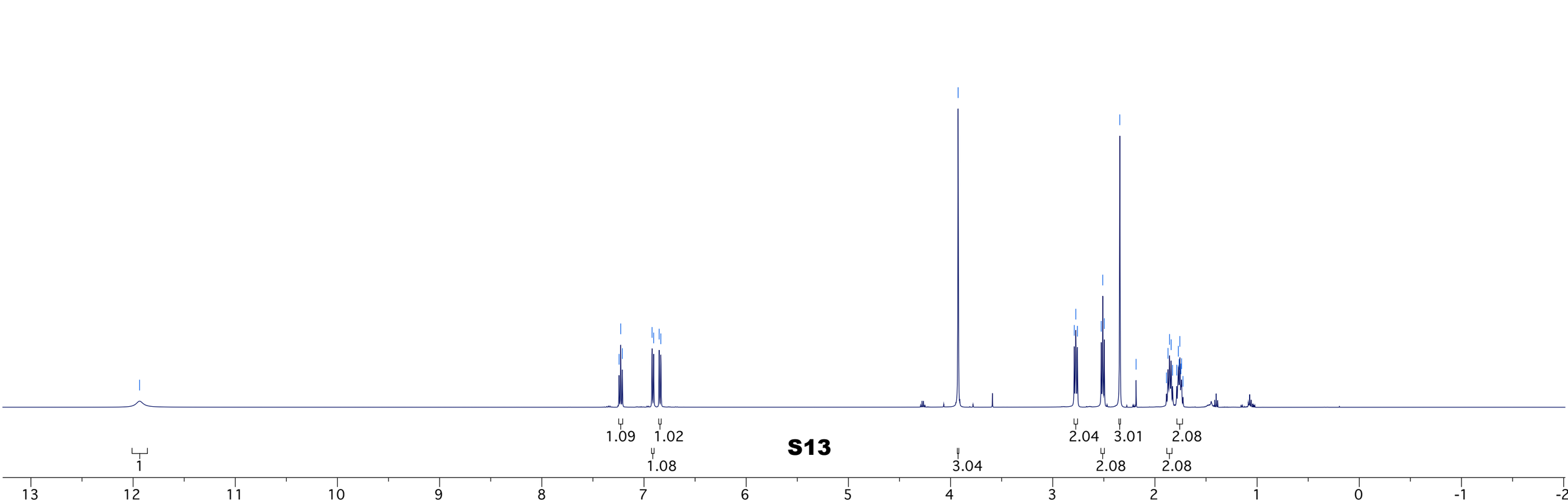

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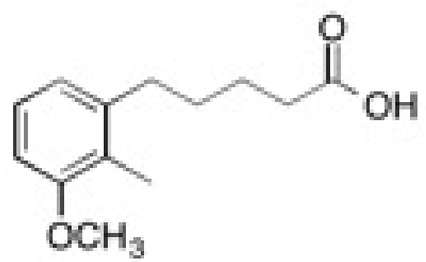

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7.228
7.212
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1.137
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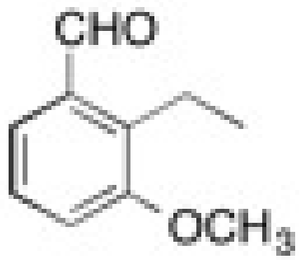

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12
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4
3
2
1
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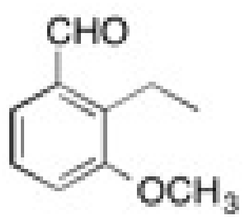

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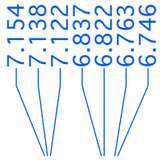
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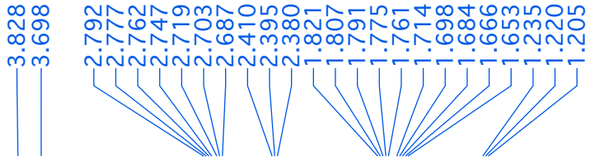
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2.747
2.719
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2.687
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2.380
1.821
1.807
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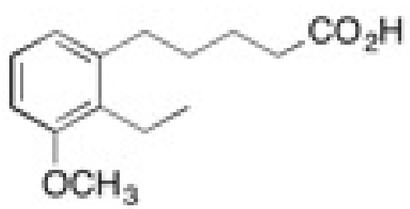
1.235
1.220
1.205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OCH3

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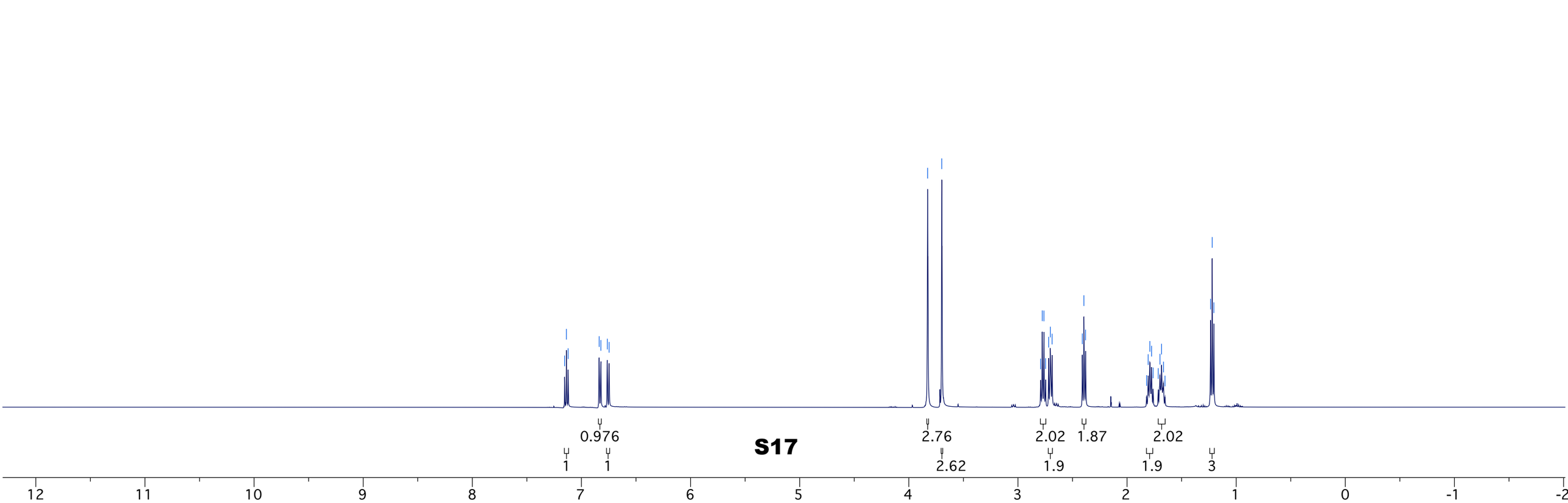
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3
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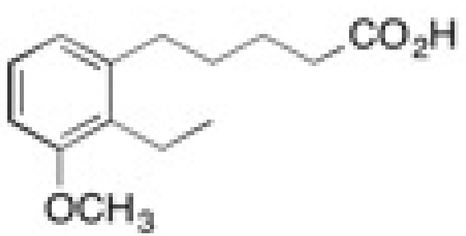
0
-1
-2

S17



10

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6.942

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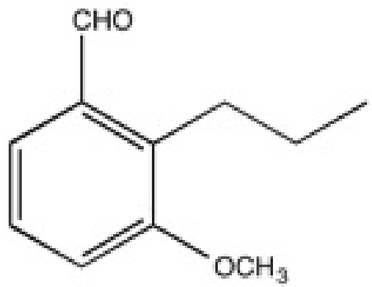

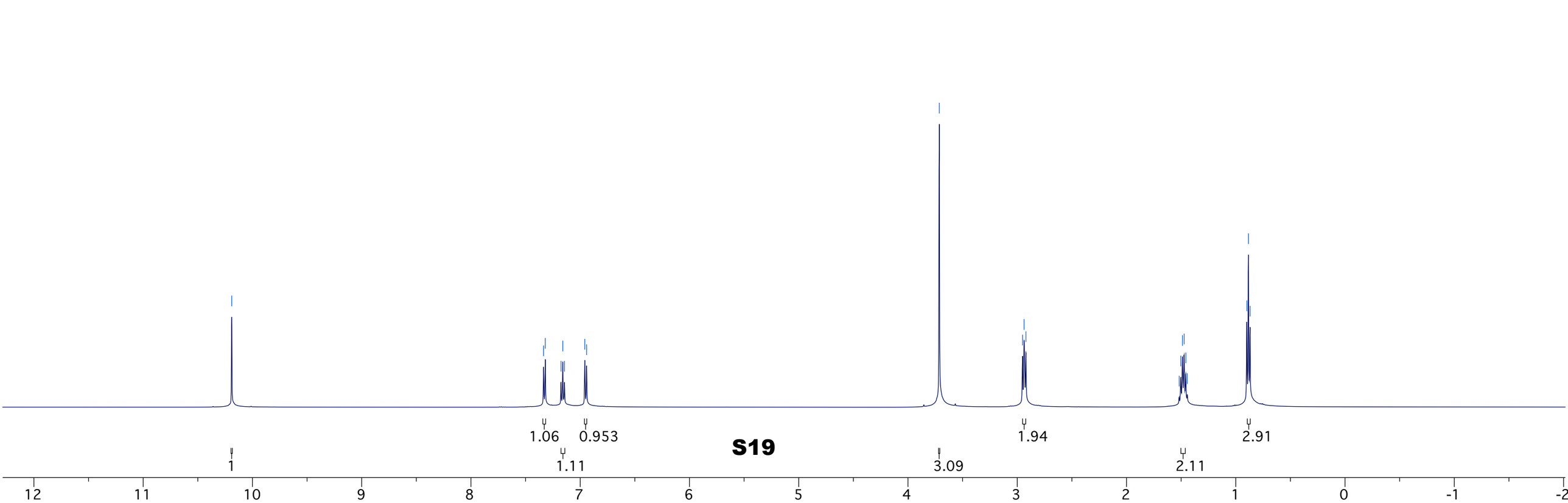

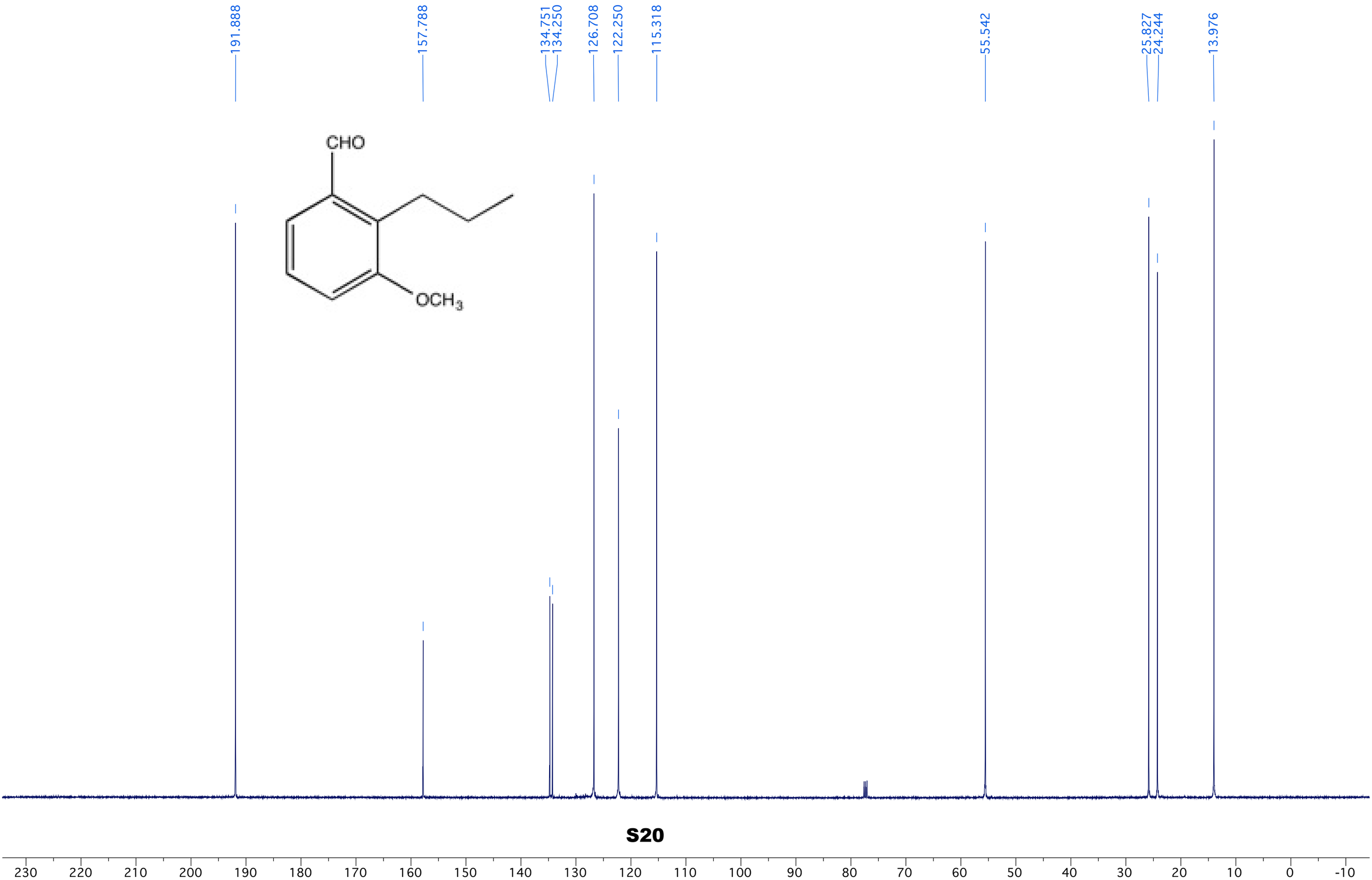
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24.244
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OCH3
230
220
210
200
190
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140
130
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110
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60
50
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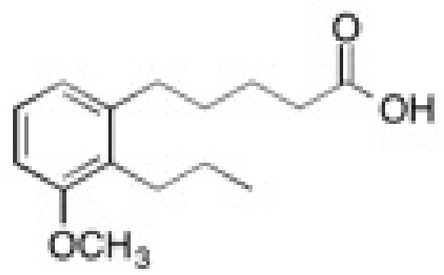
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OH
OCH3

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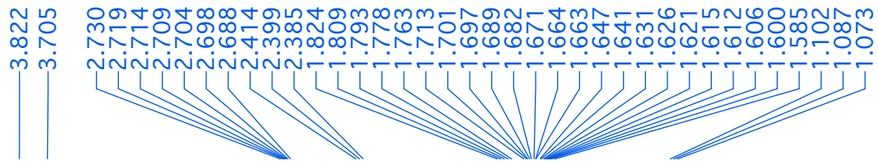
6.762
6.746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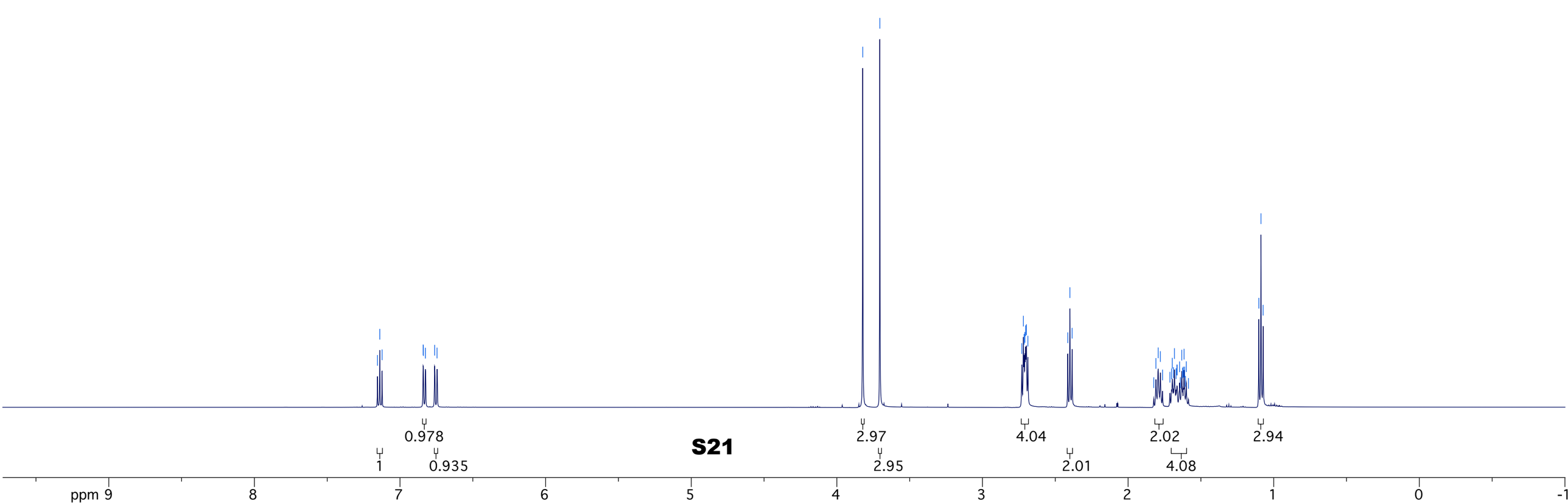
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5
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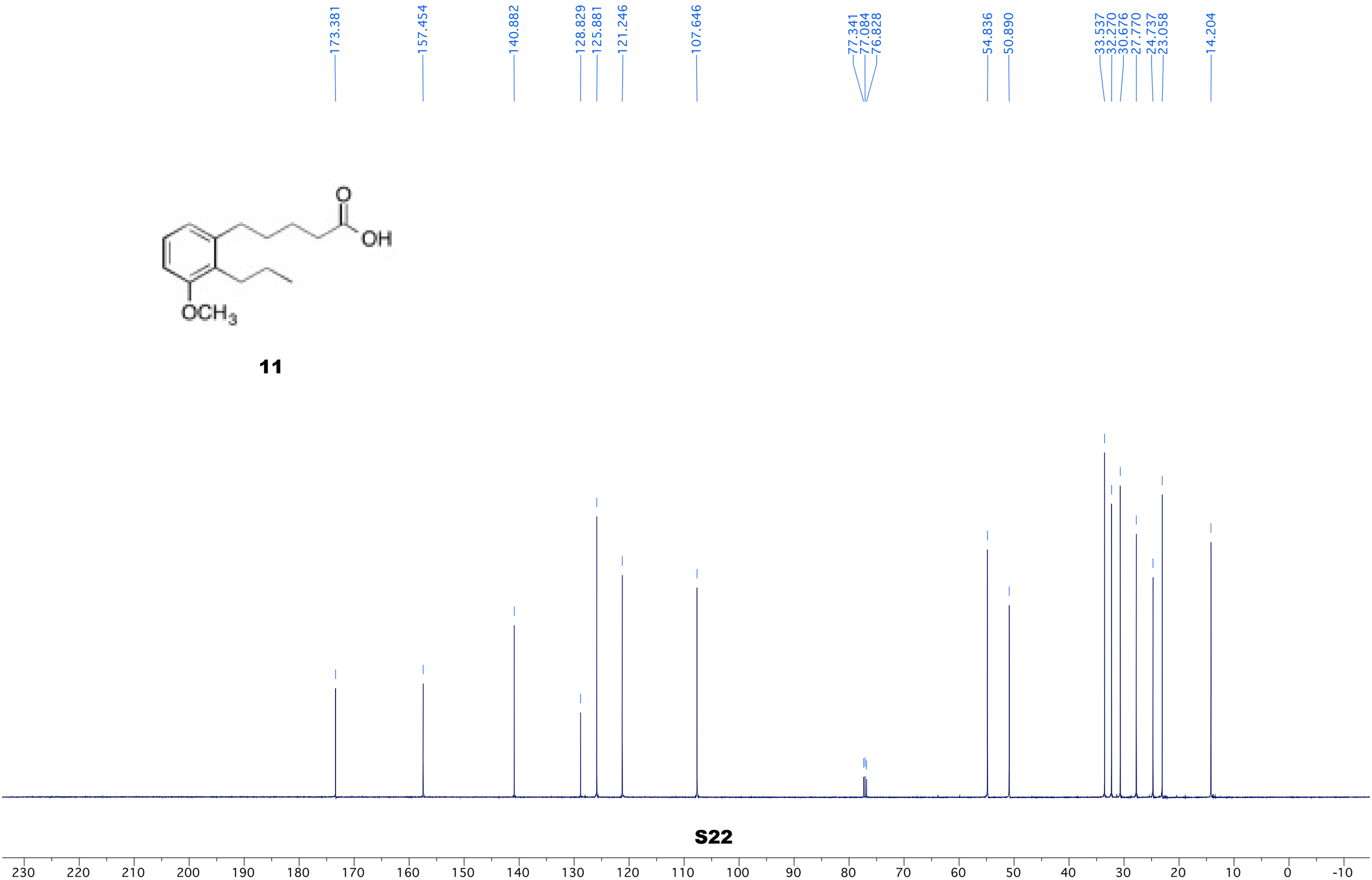
2
1
0
-1

S21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50.890
33.537
32.270
30.676
27.770
24.737
23.058
14.204
O
OH
OCH3
230
220
210
200
190
180
170
160
150
140
130
120
110
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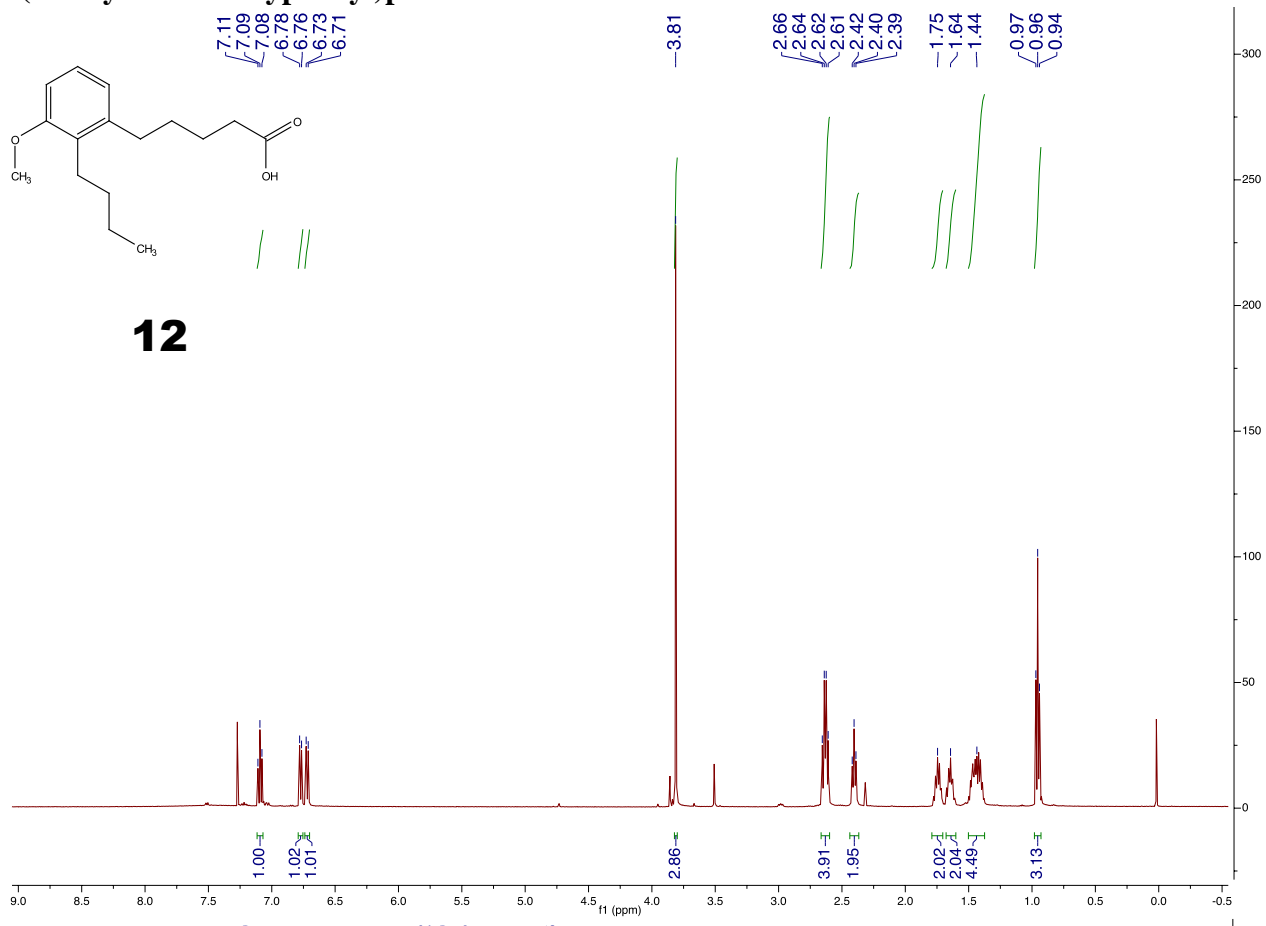
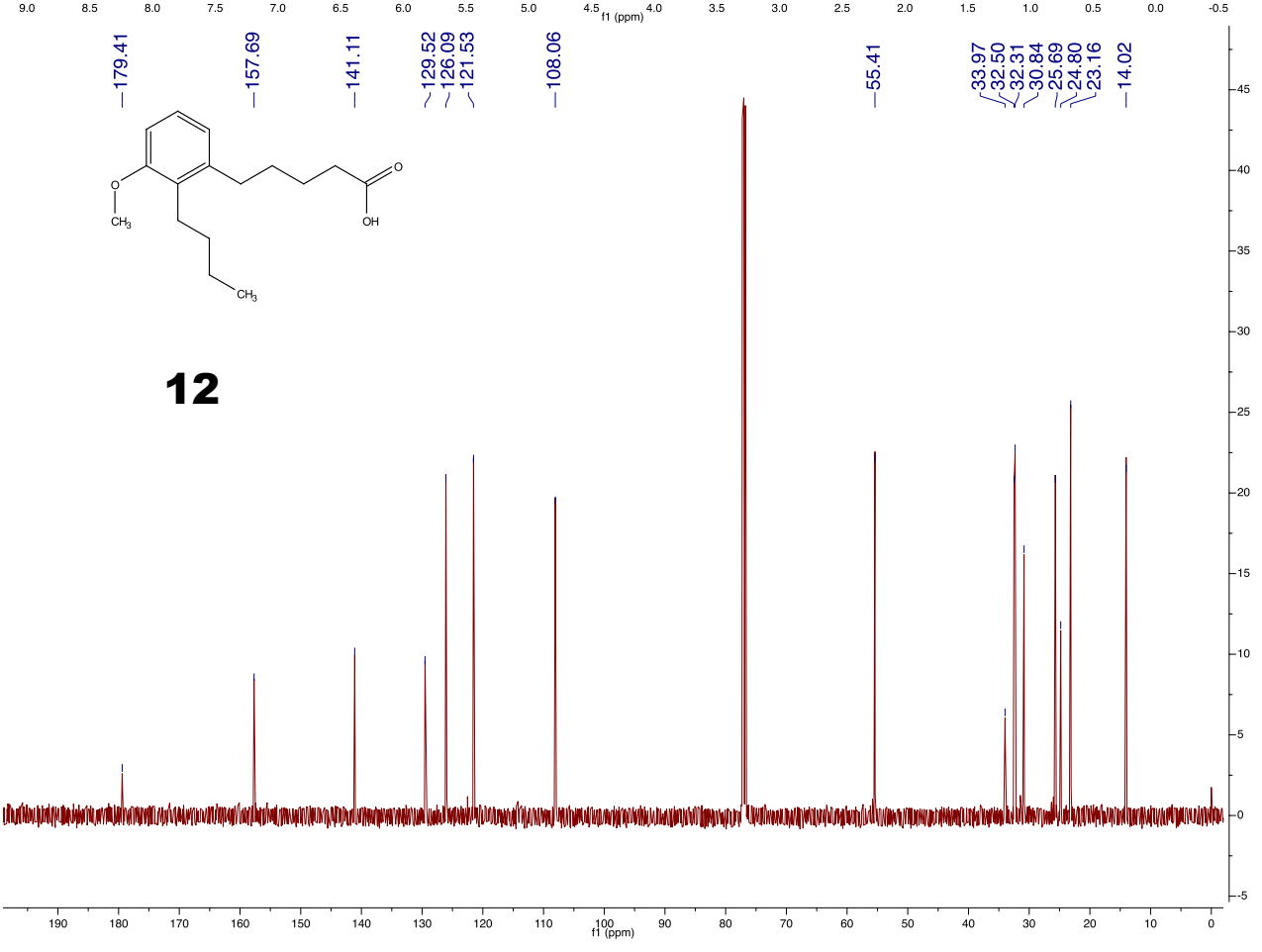
-10

11

S22

5-(2-butyl-3-methoxyphenyl)pentanoic acid





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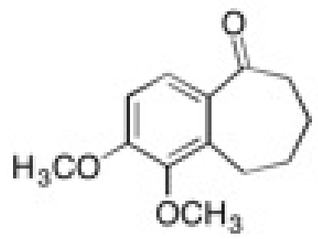

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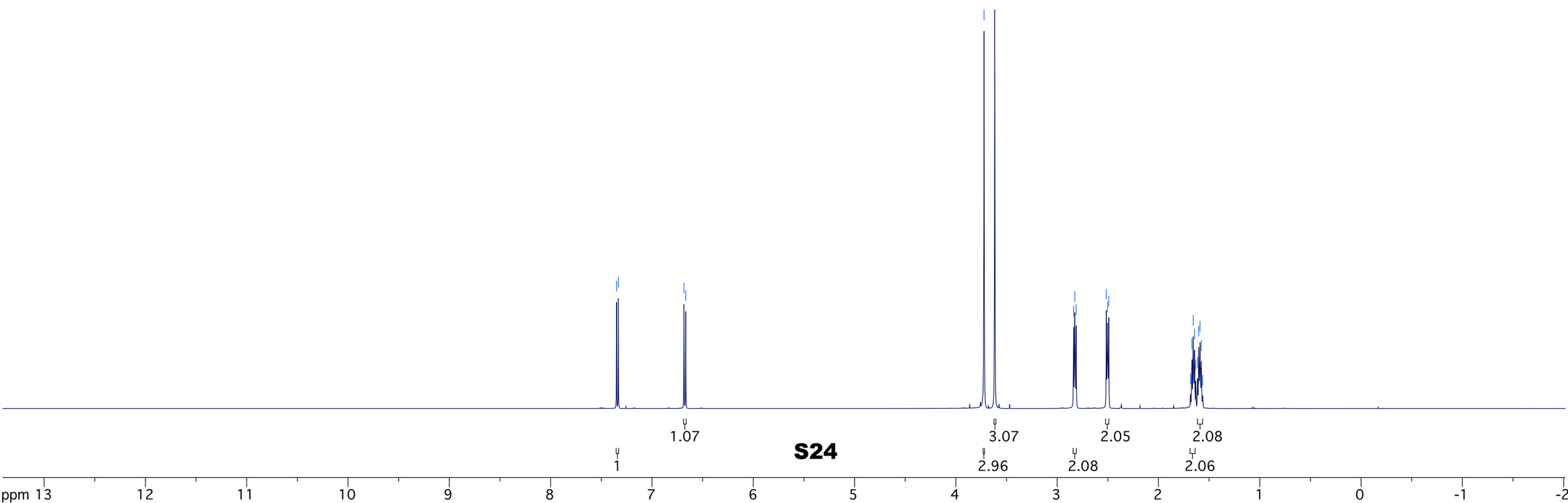

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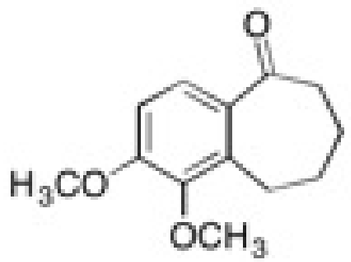
23.043
20.712



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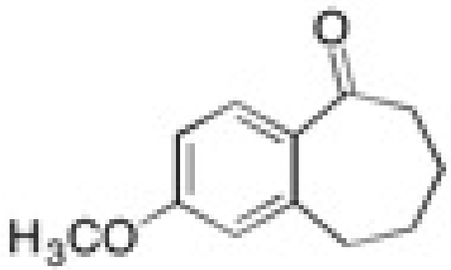
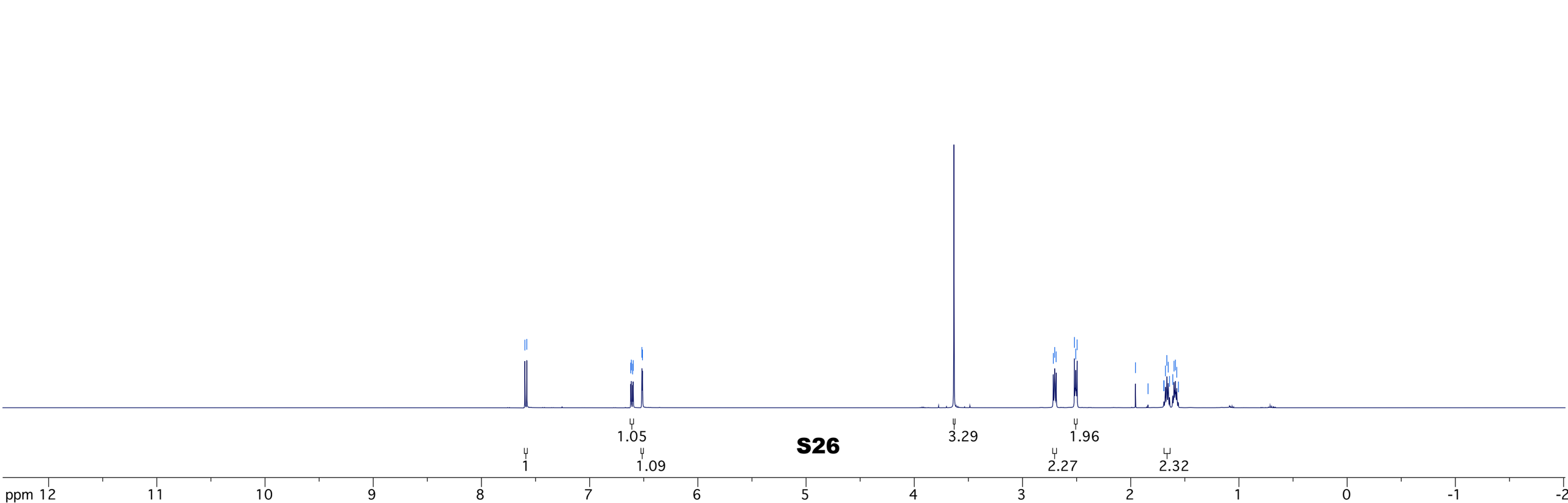
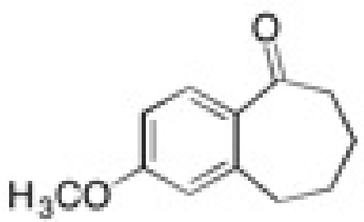


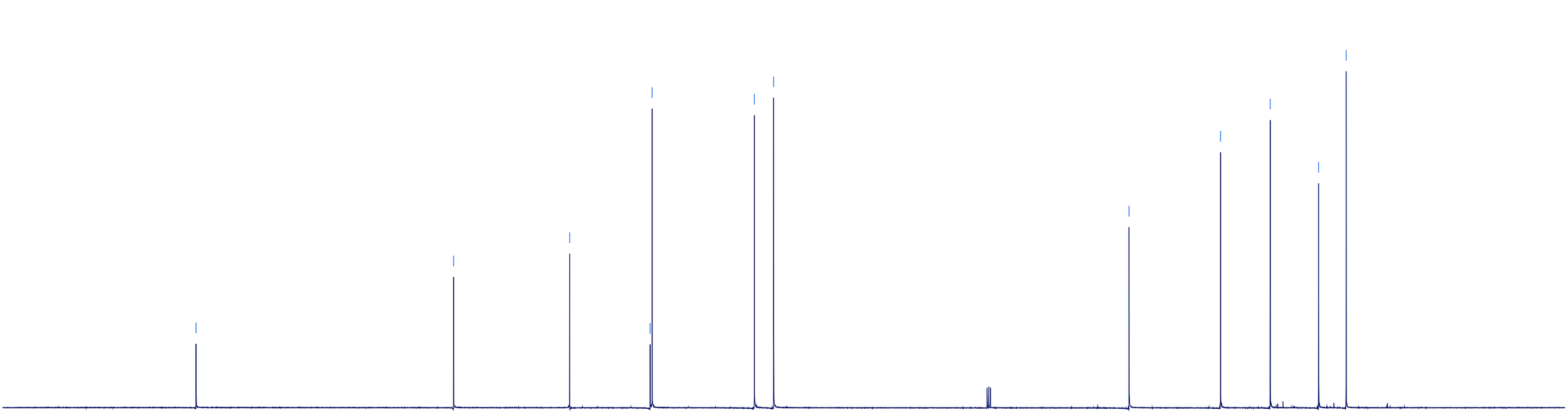
14



203.532
162.545
144.084
131.288
130.972
114.703
111.638
55.087
40.523
32.627
24.922
20.539



14

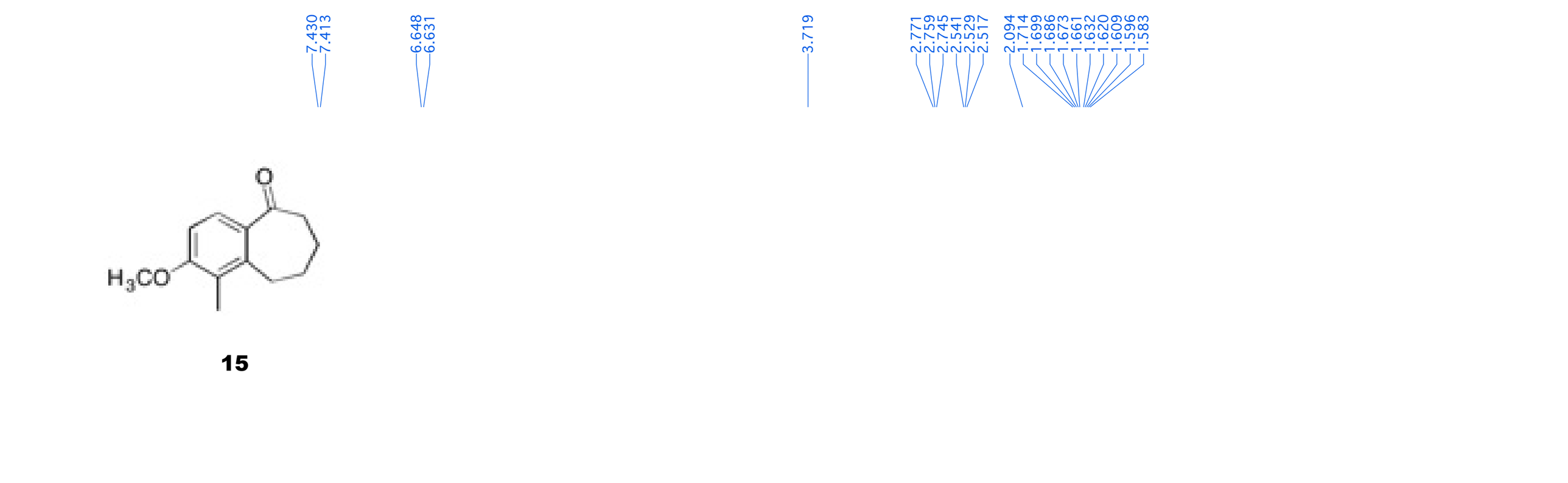


S27

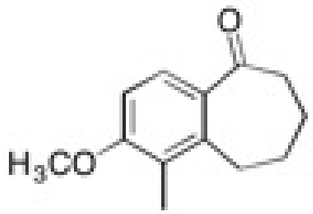
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

O
H3CO

15


7.430
7.413
6.648
6.631
3.719
2.771
2.759
2.745
2.541
2.529
2.517
2.094
1.714
1.699
1.686
1.673
1.661
1.632
1.620
1.609
1.596
1.583
1.04
2.05
3.07
1.97
1
3.03
2.02
2.04
ppm 9
8
7
6
5
4
3
2
1
0
-1
-2

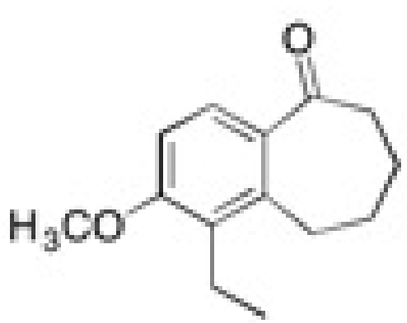
205.877
160.390
140.351
132.595
127.390
123.671
107.647
55.376
40.411
26.952
24.120
20.555
11.038



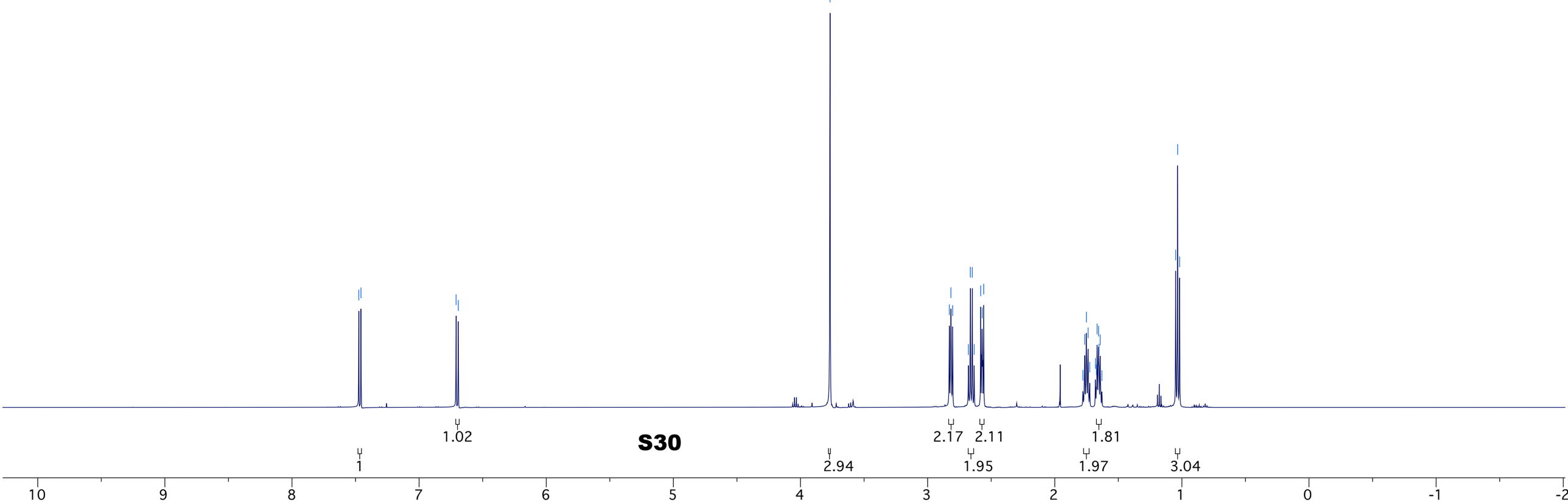
15

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

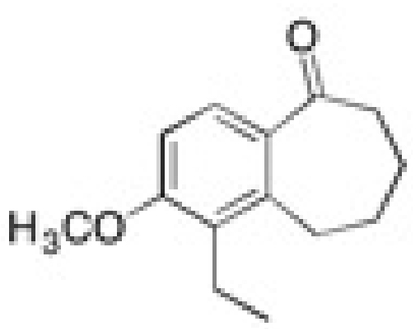
7.474
7.457
6.709
6.692
3.768
2.829
2.817
2.803
2.679
2.664
2.649
2.634
2.583
2.571
2.559
1.778
1.764
1.751
1.738
1.725
1.680
1.672
1.667
1.655
1.652
1.643
1.629
1.048
1.033
1.018



16

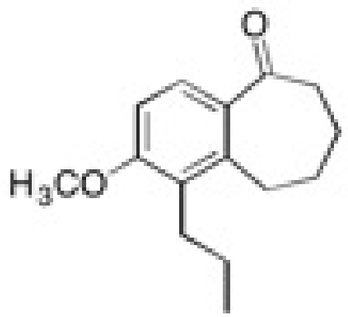


206.340
160.314
139.515
132.921
130.001
127.694
107.986
55.405
40.427
26.251
24.932
20.378
18.879
14.491



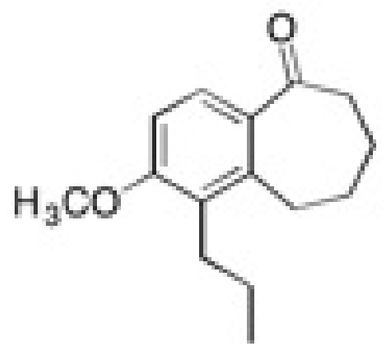
16

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10



17

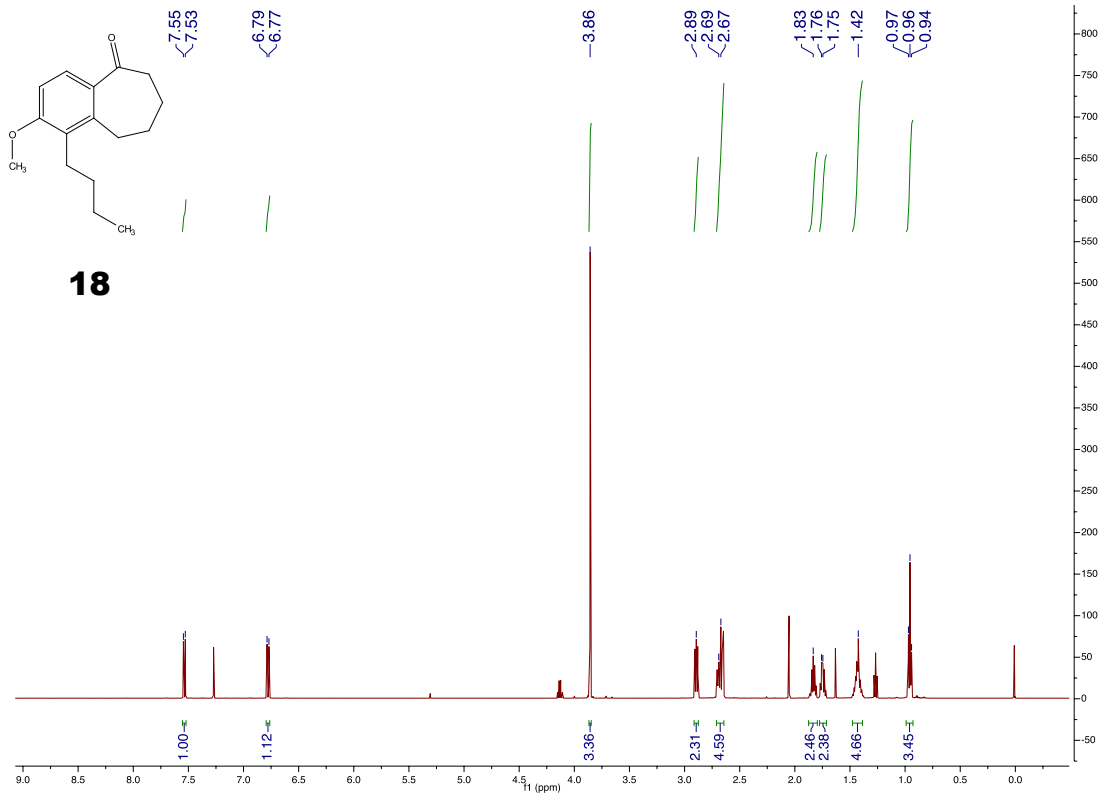
206.240
160.438
139.656
132.850
128.494
127.647
107.832
55.248
40.325
27.560
26.315
24.845
23.284
20.298
14.161



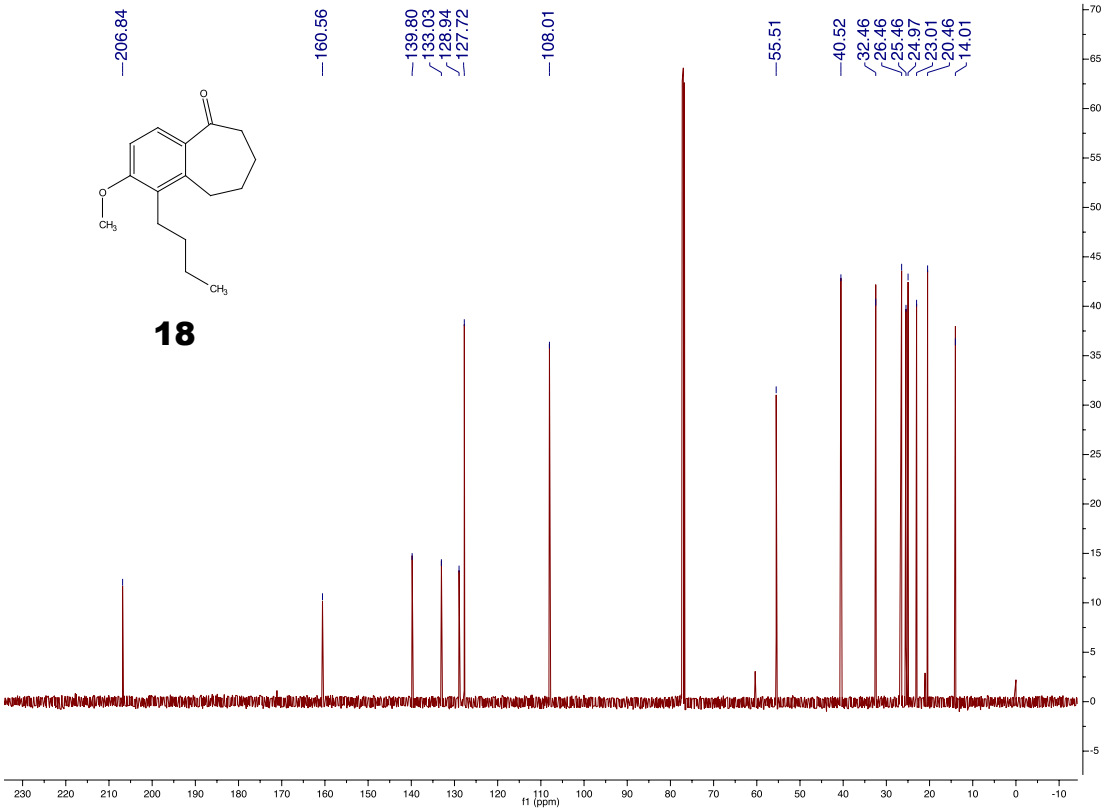
17

S33

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

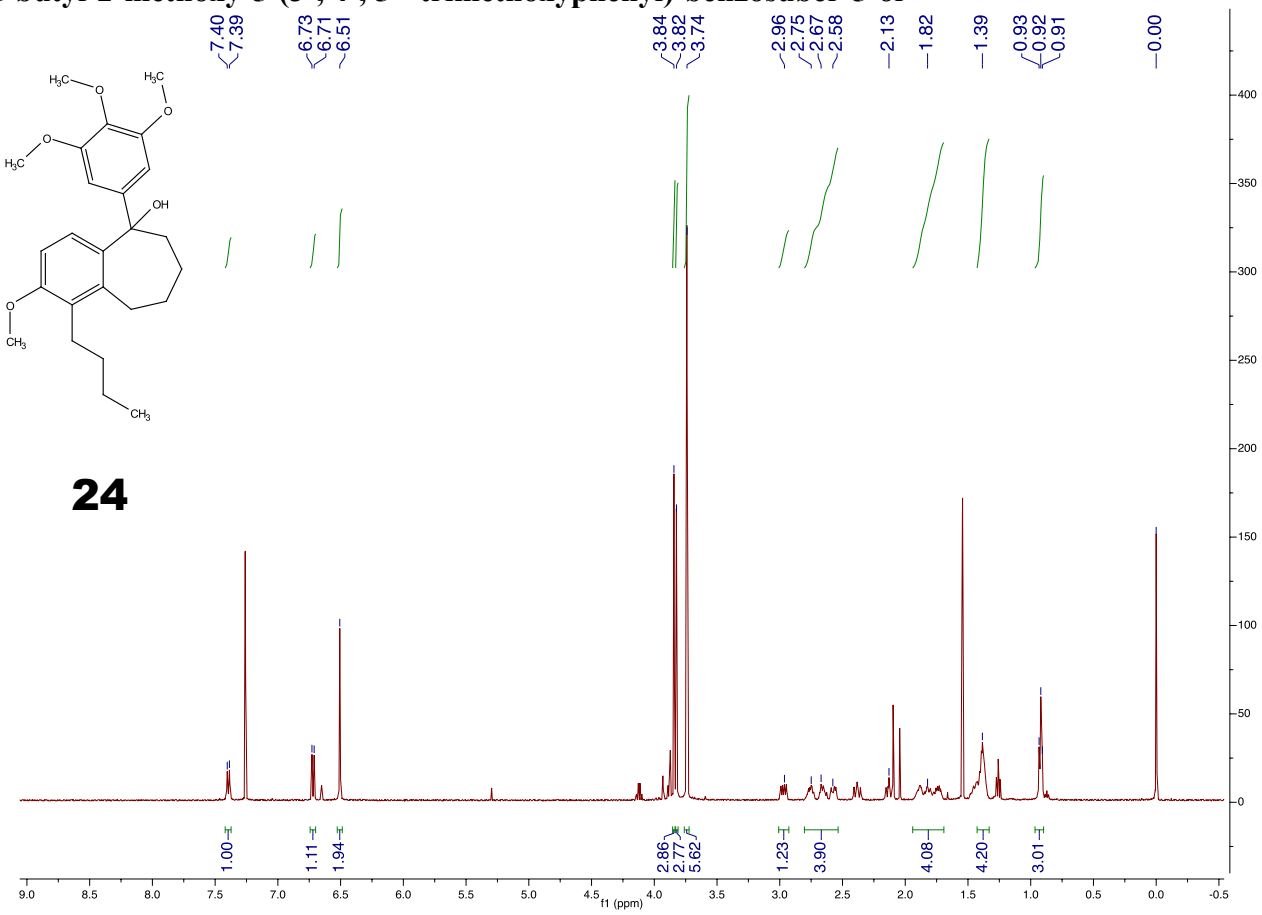

7.55
7.53
6.79
6.77
3.86
2.89
2.69
2.67
1.83
1.76
1.75
1.42
0.97
0.96
0.94
1.00
1.12
3.36
2.31
4.59
2.46
2.38
4.66
3.45
f1 (ppm)

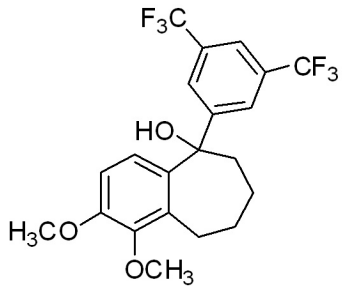
18


206.84
160.56
139.80
133.03
128.94
127.72
108.01
55.51
40.52
32.46
26.46
25.46
24.97
23.01
20.46
14.01
f1 (ppm)

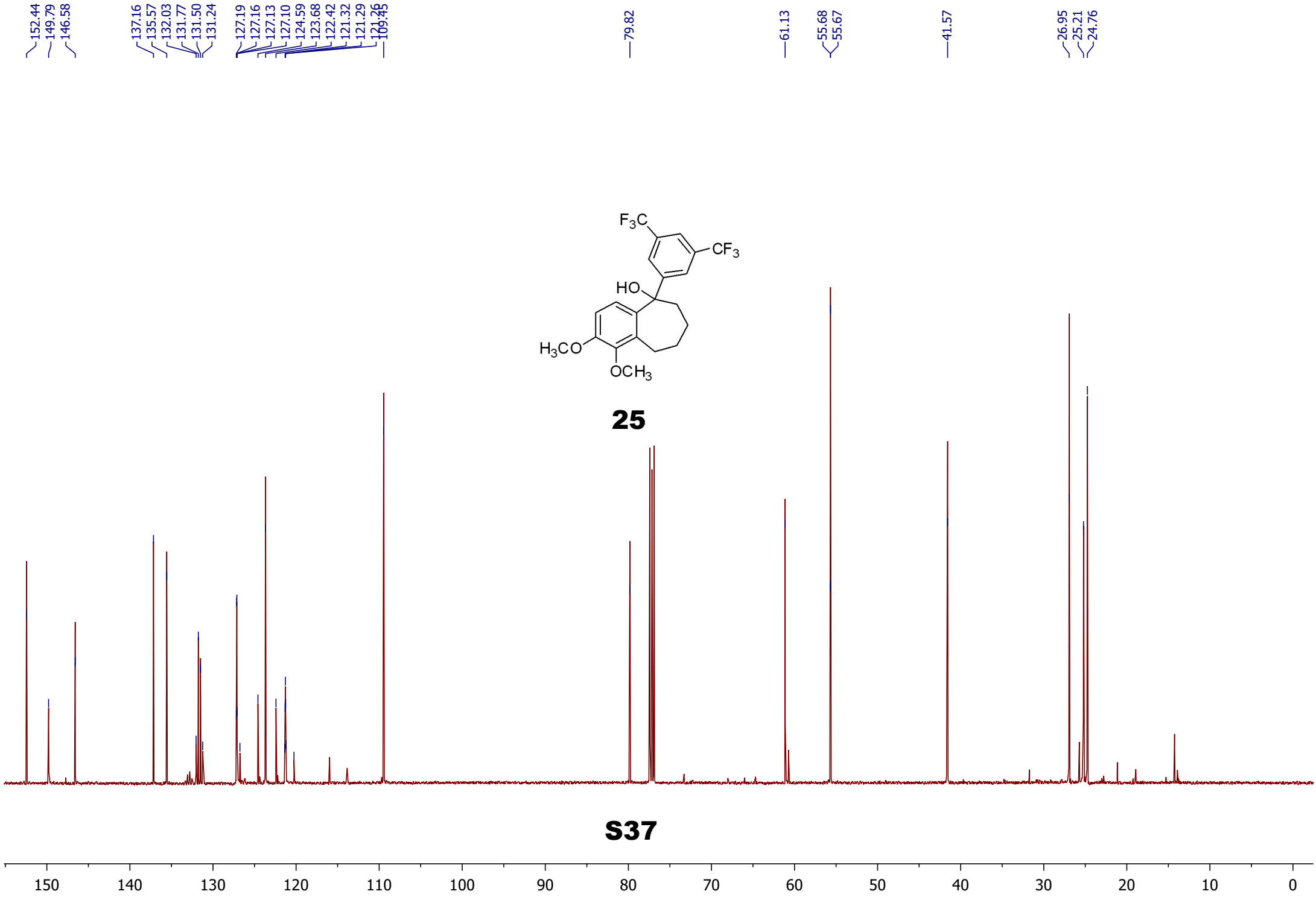
18

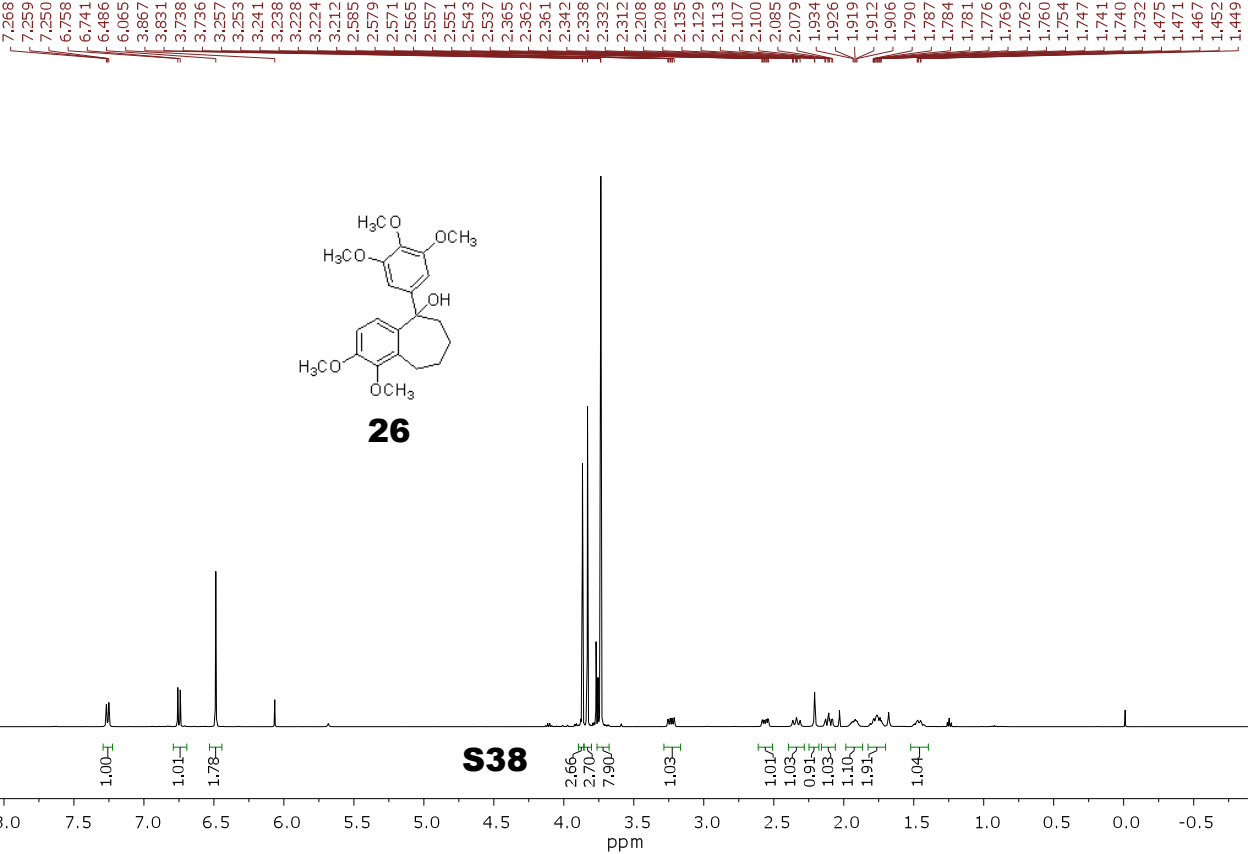
1-butyl-2-methoxy-5-(3', 4', 5'- trimethoxyphenyl)-benzosuber-5-ol

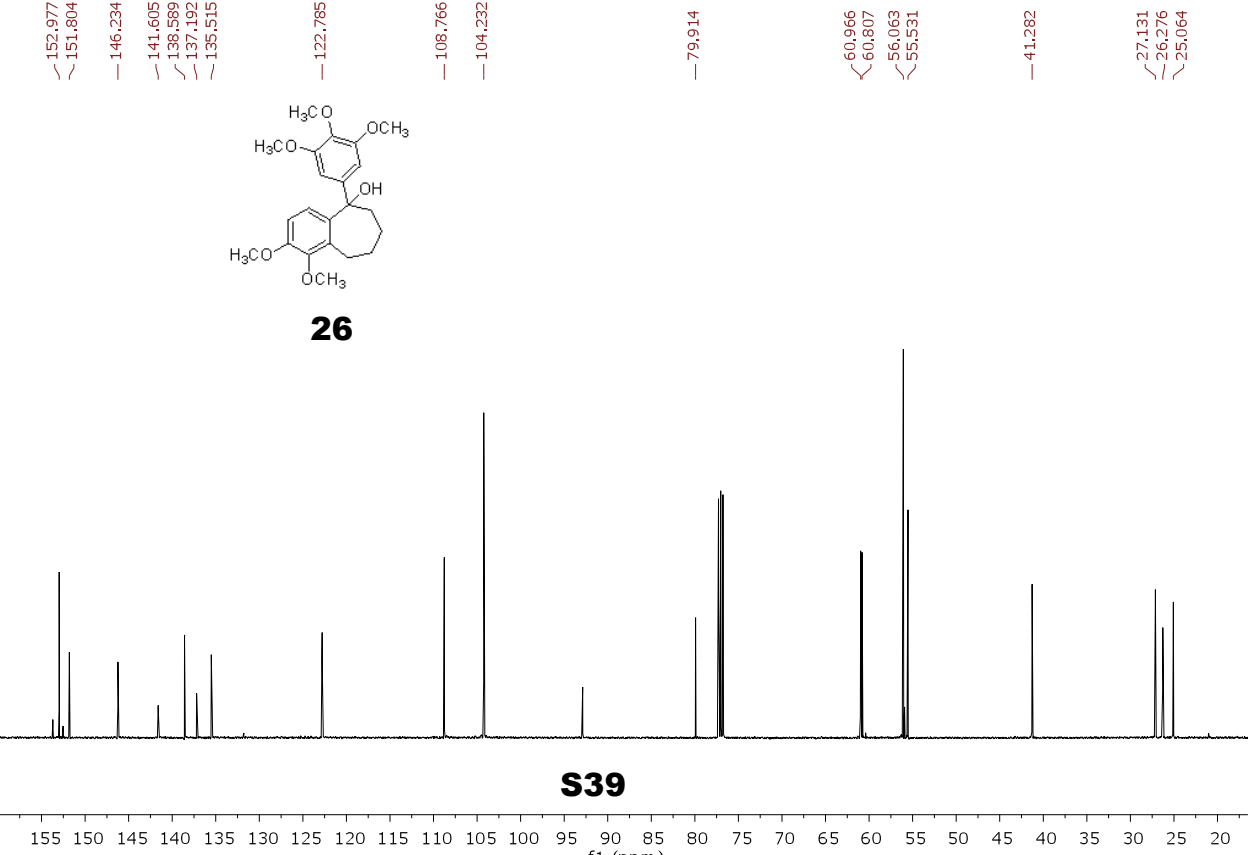




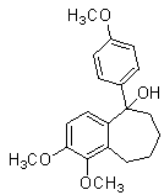
25


152.44
149.79
146.58
137.16
135.57
132.03
131.77
131.50
131.24
127.19
127.16
127.13
127.10
124.59
123.68
122.42
121.32
121.29
79.82
61.13
55.68
55.67
41.57
26.95
25.21
24.76
F3C
CF3
HO
H3CO
OCH3
25
S37
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

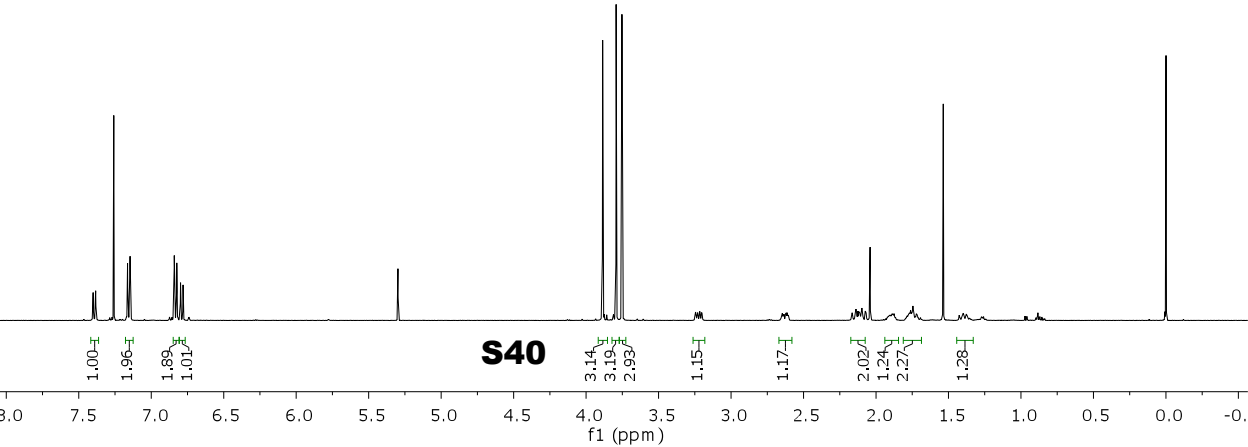

7.268 7.259 7.250 6.758 6.741 6.486 6.065 3.867 3.831 3.738 3.736 3.257 3.253 3.241 3.238 3.228 3.224 3.212 2.585 2.579 2.571 2.565 2.557 2.551 2.543 2.537 2.365 2.362 2.361 2.342 2.338 2.332 2.312 2.208 2.208 2.135 2.129 2.113 2.107 2.100 2.085 2.079 1.934 1.926 1.919 1.912 1.906 1.790 1.787 1.784 1.781 1.776 1.769 1.762 1.760 1.754 1.747 1.741 1.740 1.732 1.475 1.471 1.467 1.452 1.449
H3CO
OCH3
H3CO
OH
H3CO
OCH3
26
S38
1.00
1.01
1.78
2.66
2.70
7.90
1.03
1.01
1.03
0.91
1.03
1.10
1.91
1.04
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 -0.5
ppm


152.977
151.804
146.234
141.605
138.589
137.192
135.515
122.785
108.766
104.232
79.914
60.966
60.807
56.063
55.531
41.282
27.131
26.276
25.064
H3CO
OCH3
H3CO
OH
H3CO
OCH3
26
S39
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20
f1 (ppm)

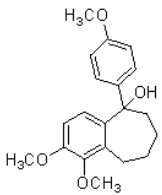
7.401 7.384 7.259 7.164 7.146 6.841 6.824 6.798 6.780 3.886 3.793 3.753 3.245 3.242 3.234 3.230 3.219 3.216 3.214 3.204 3.201 3.199 2.652 2.646 2.640 2.635 2.633 2.625 2.624 2.618 2.612 2.606 2.604 2.166 2.163 2.143 2.138 2.134 2.126 2.120 2.112 2.103 2.098 2.096 2.092 2.074 2.068 1.902 1.893 1.888 1.882 1.875 1.775 1.771 1.767 1.761 1.753 1.745 1.739 1.722 1.399 1.376



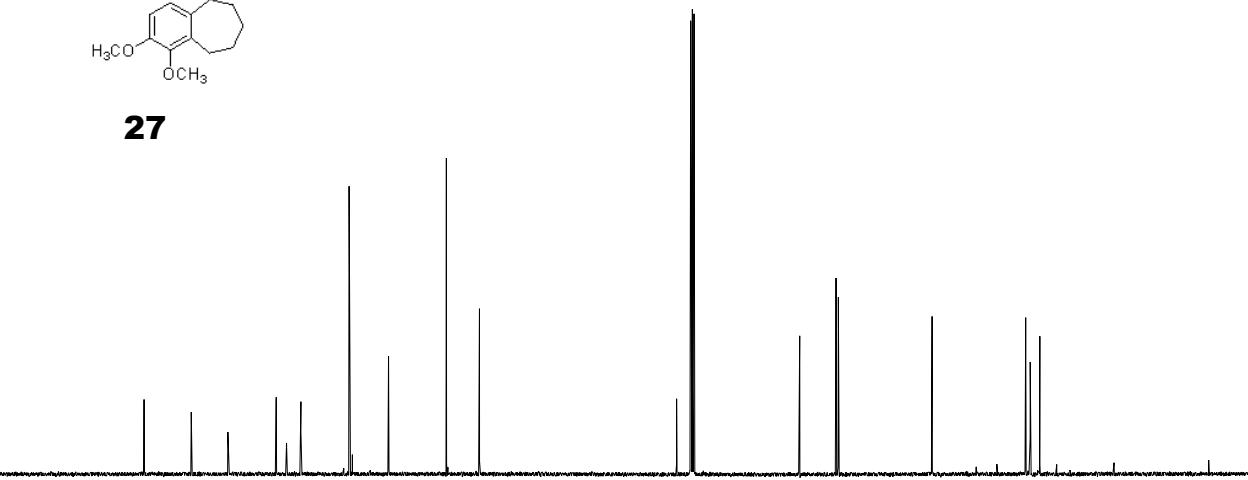
27



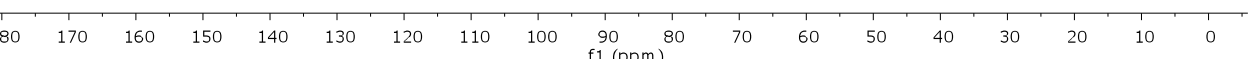
158.783
151.739
146.279
139.078
137.530
135.388
128.204
122.314
113.693
108.751
79.349
77.249 cdcl3
76.994 cdcl3
76.740 cdcl3
60.977
55.561
55.218
41.234
27.273
26.598
25.163



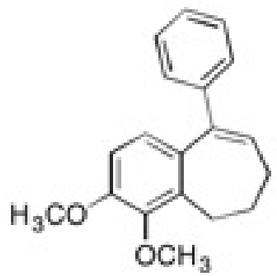
27



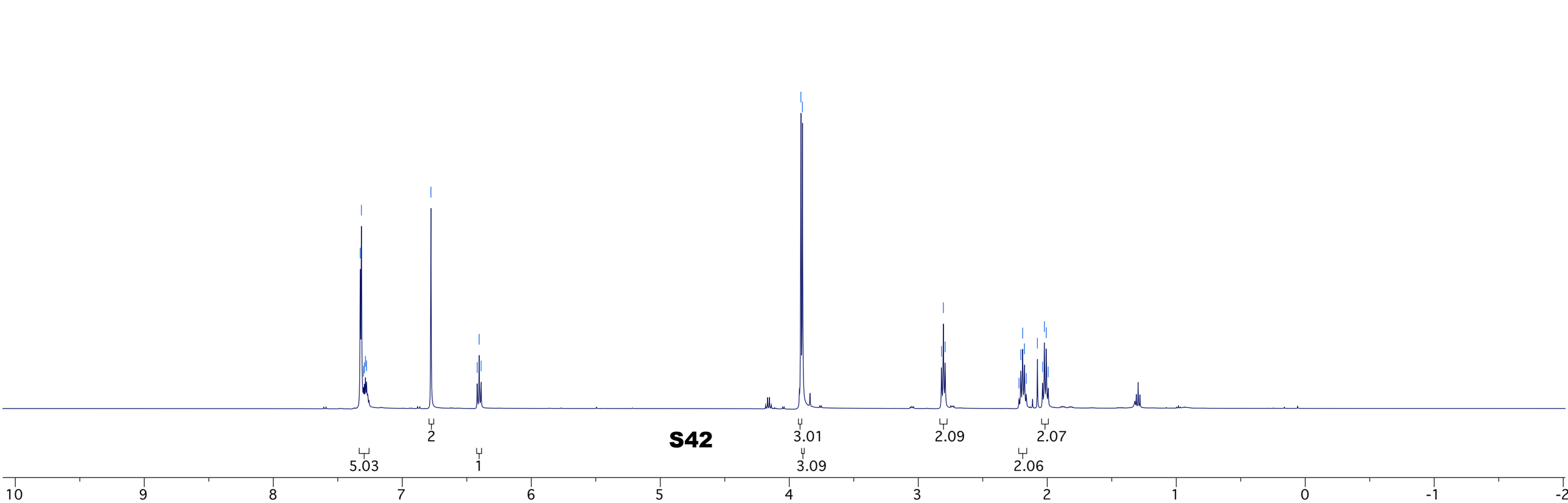
S41




7.326
7.317
7.301
7.293
7.286
7.277
6.778
6.419
6.404
6.389
3.910
3.898
2.820
2.806
2.792
2.219
2.205
2.191
2.177
2.163
2.076
2.036
2.022
2.008
1.993

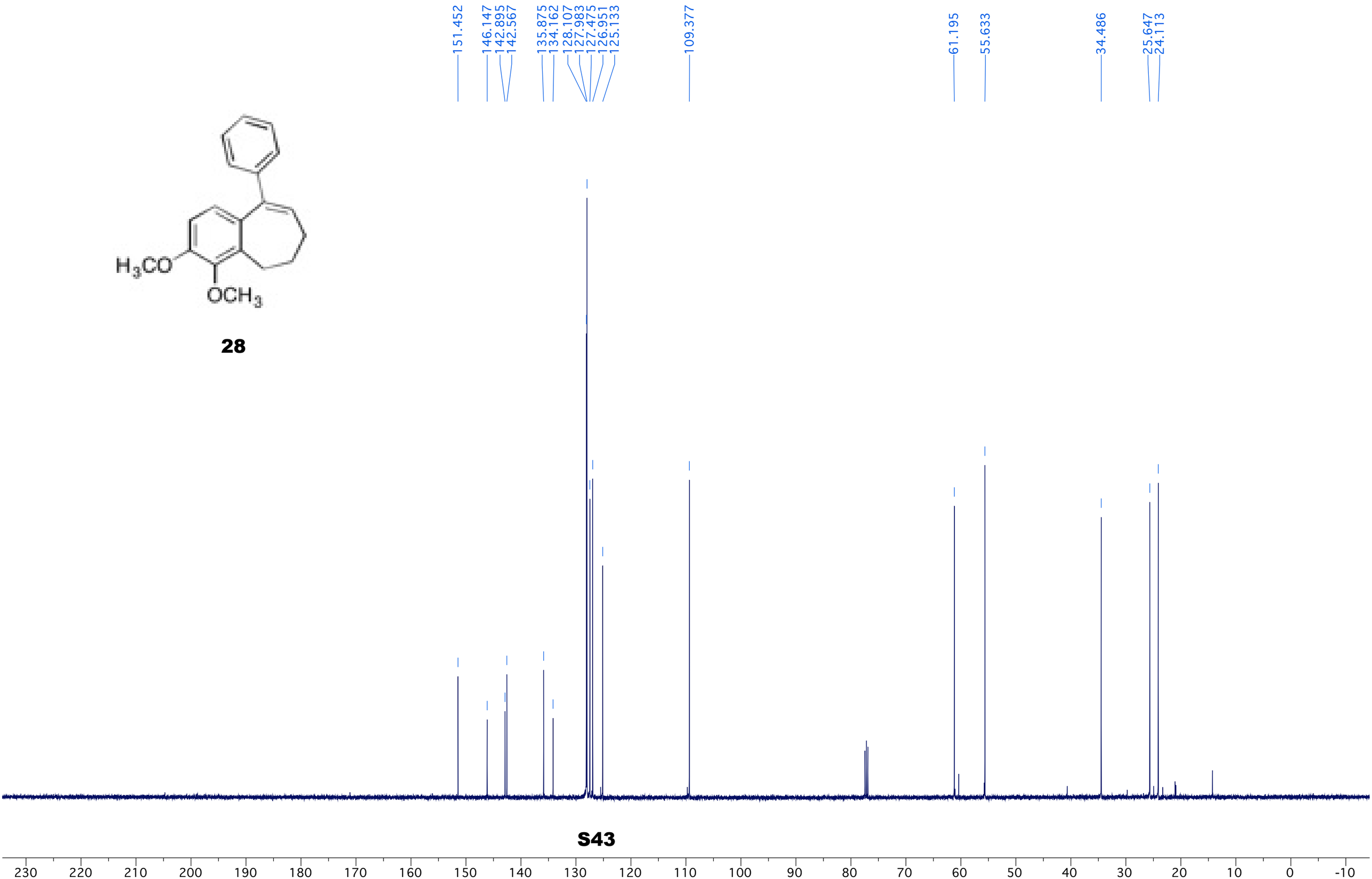

H3CO
OCH3

28


2
3.01
2.09
2.07
5.03
1
3.09
2.06
10
9
8
7
6
5
4
3
2
1
0
-1
-2

H3CO
OCH3

28


151.452
146.147
142.895
142.567
135.875
134.162
128.107
127.983
127.475
126.951
125.133
109.377
61.195
55.633
34.486
25.647
24.113
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_134000010.D
Sample Name: CAH_3_134

HPLC for Compound 28

```
==========================================================================
Acq. Operator   : Christine
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/9/2014 3:11:56 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 1/9/2014 3:02:55 PM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_3_134000010.D\DA.M (MASTERMETHOD.M)
Last changed    : 2/25/2014 11:06:43 AM by Christine
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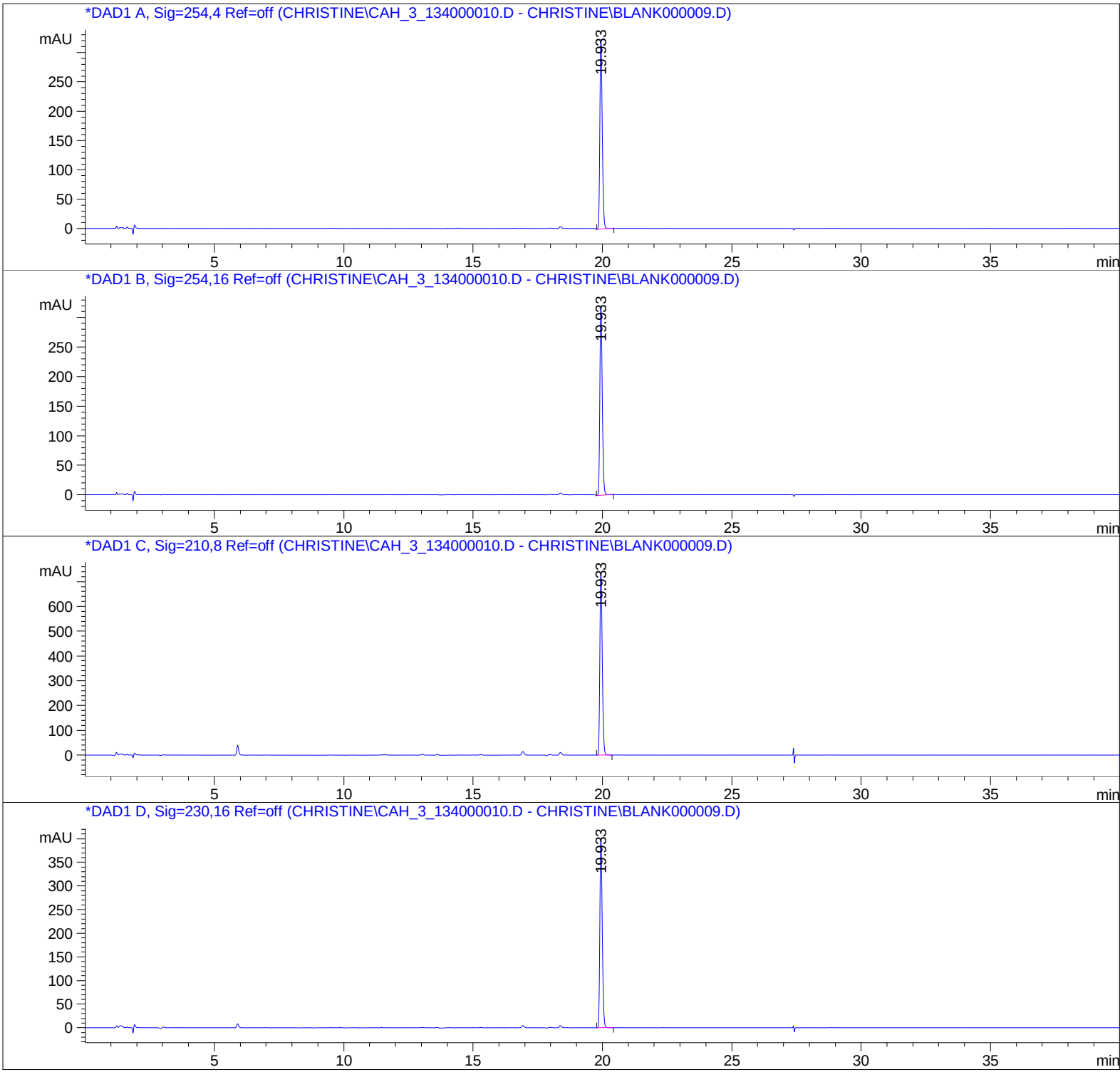
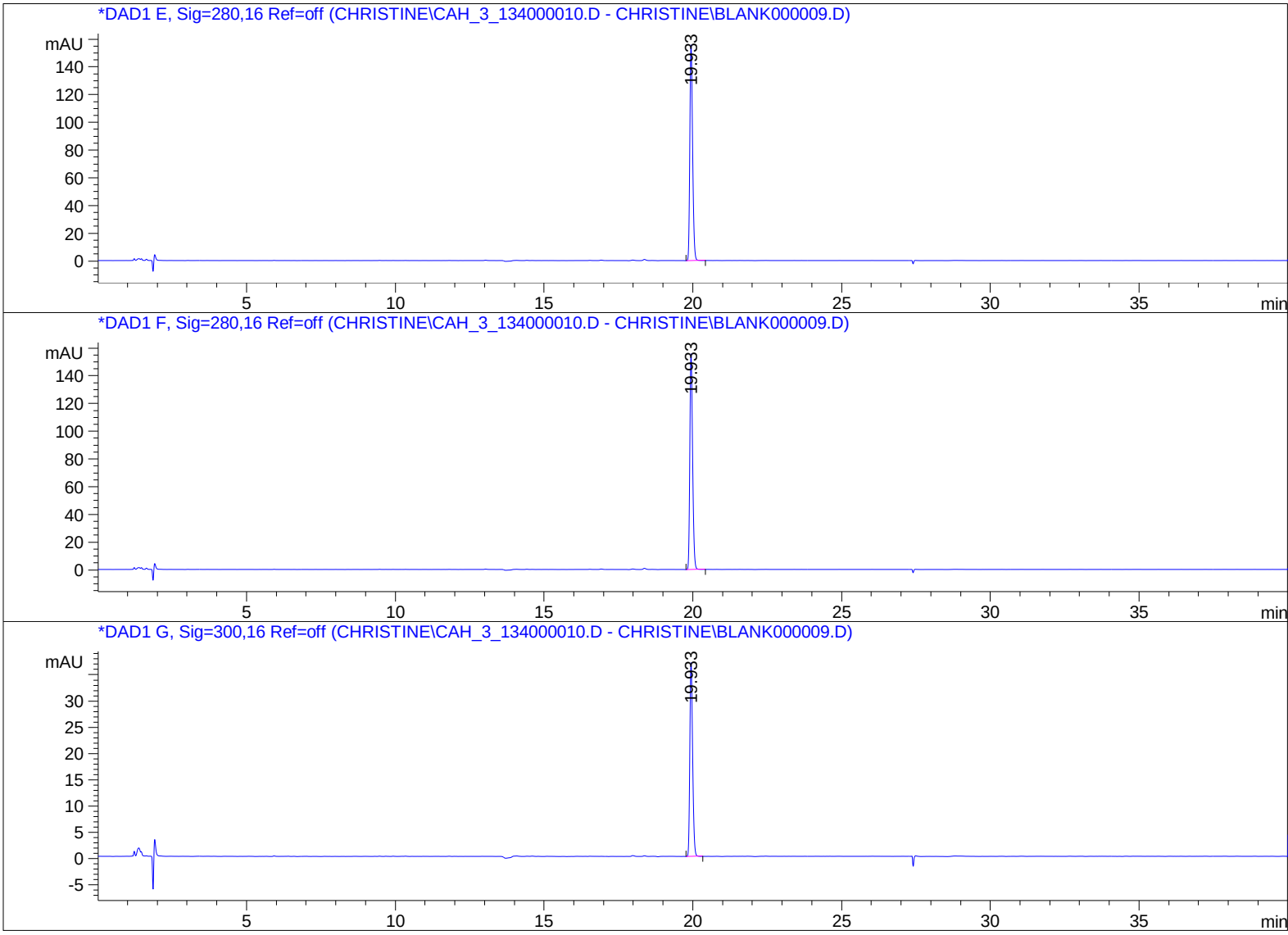


Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_134000010.D
Sample Name: CAH_3_134



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.1000	2109.92017	323.73047	100.0000
Totals :				2109.92017	323.73047	

S45

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_134000010.D
Sample Name: CAH_3_134

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.1000	2092.94263	321.40930	100.0000
Totals :				2092.94263	321.40930	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.0995	4793.37109	740.26355	100.0000
Totals :				4793.37109	740.26355	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.0994	2596.50854	401.65051	100.0000
Totals :				2596.50854	401.65051	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.0995	1007.30444	155.69983	100.0000
Totals :				1007.30444	155.69983	

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_134000010.D
Sample Name: CAH_3_134

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.0995	1007.30444	155.69983	100.0000
Totals :				1007.30444	155.69983	

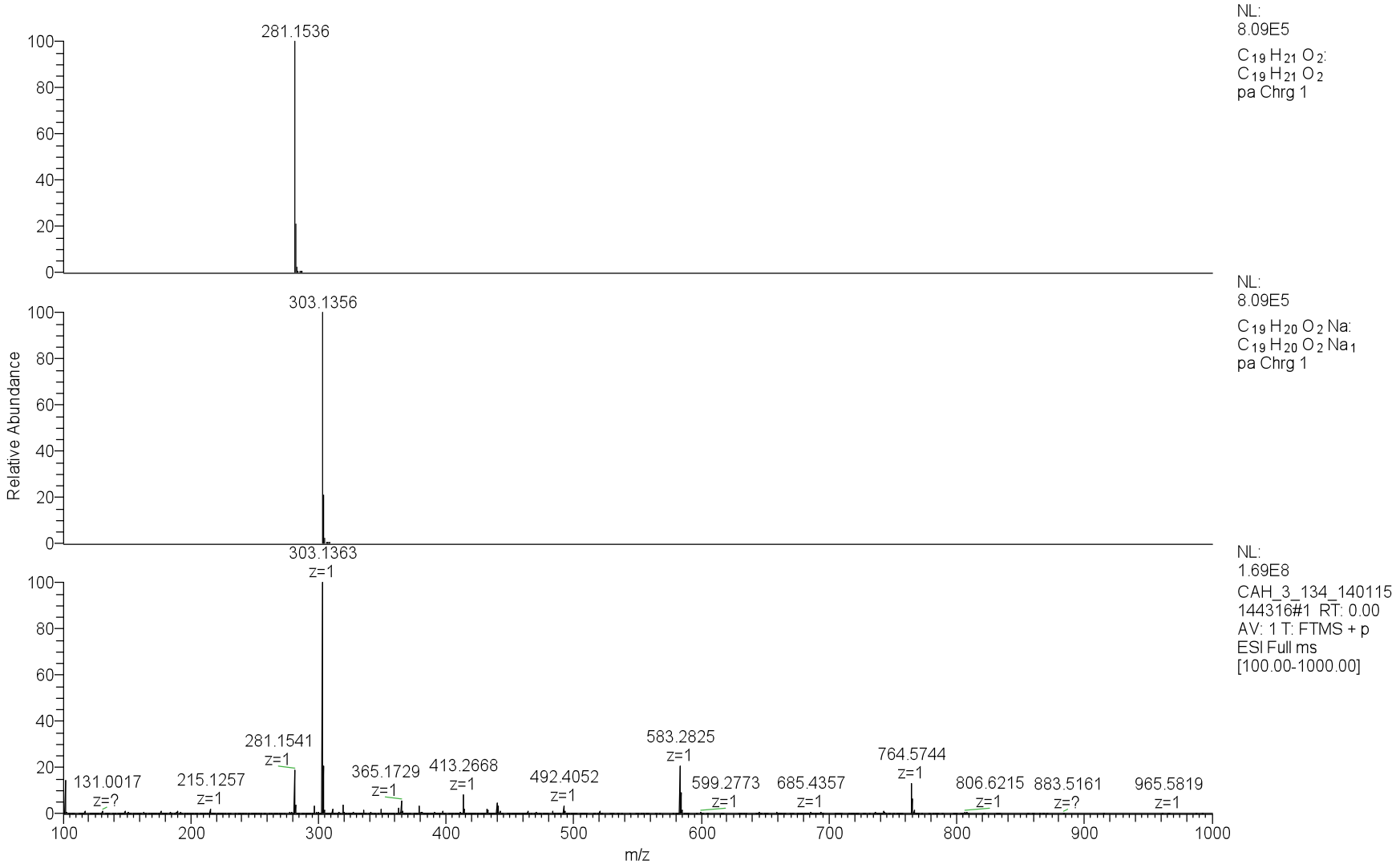
Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.933	BB	0.1001	240.84787	36.89976	100.0000
Totals :				240.84787	36.89976	

==========

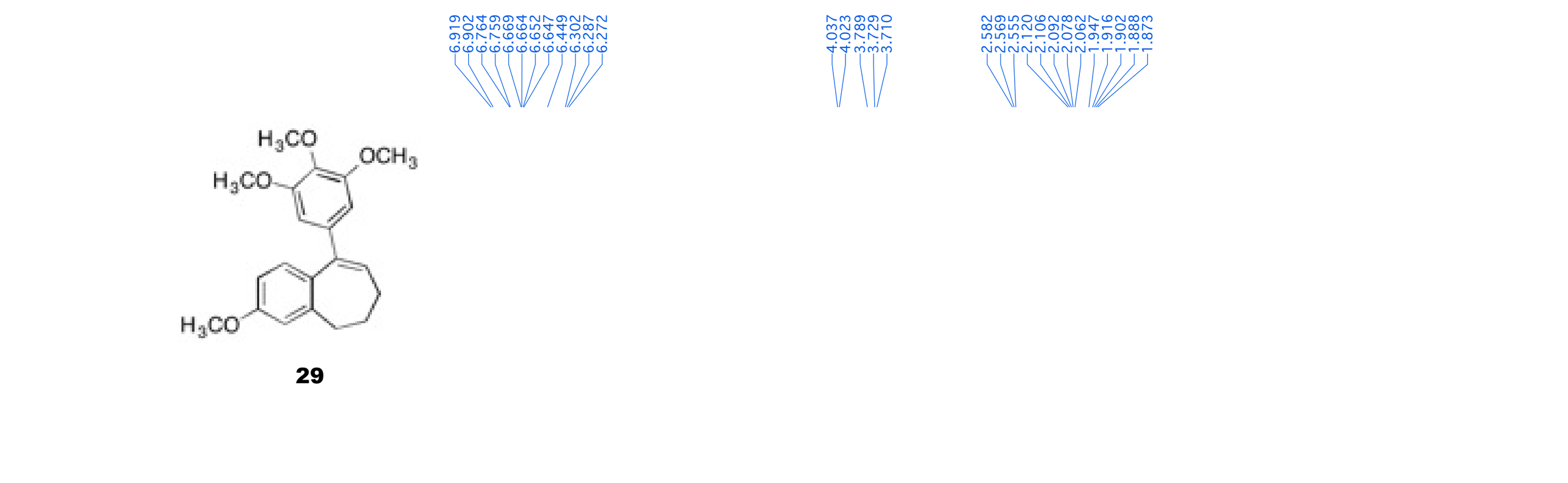
*** End of Report ***

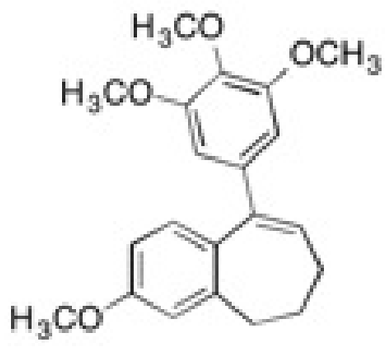
HRMS for Compound 28



H3CO
OCH3
H3CO
H3CO

29


6.919
6.902
6.764
6.759
6.669
6.664
6.652
6.647
6.449
6.302
6.287
6.272
4.037
4.023
3.789
3.729
3.710
2.582
2.569
2.555
2.120
2.106
2.092
2.078
2.062
1.947
1.916
1.902
1.888
1.873
0.929
1.97
2.88
5.89
2.17
1
0.97
1.05
3.12
1.97
1.87
ppm 10
9
8
7
6
5
4
3
2
1
0
-1
-2



29

158.453
152.832
143.666
142.719
138.355
137.315
132.301
130.471
126.838
113.832
111.125
105.138
60.664
55.905
54.919
35.030
32.732
25.418

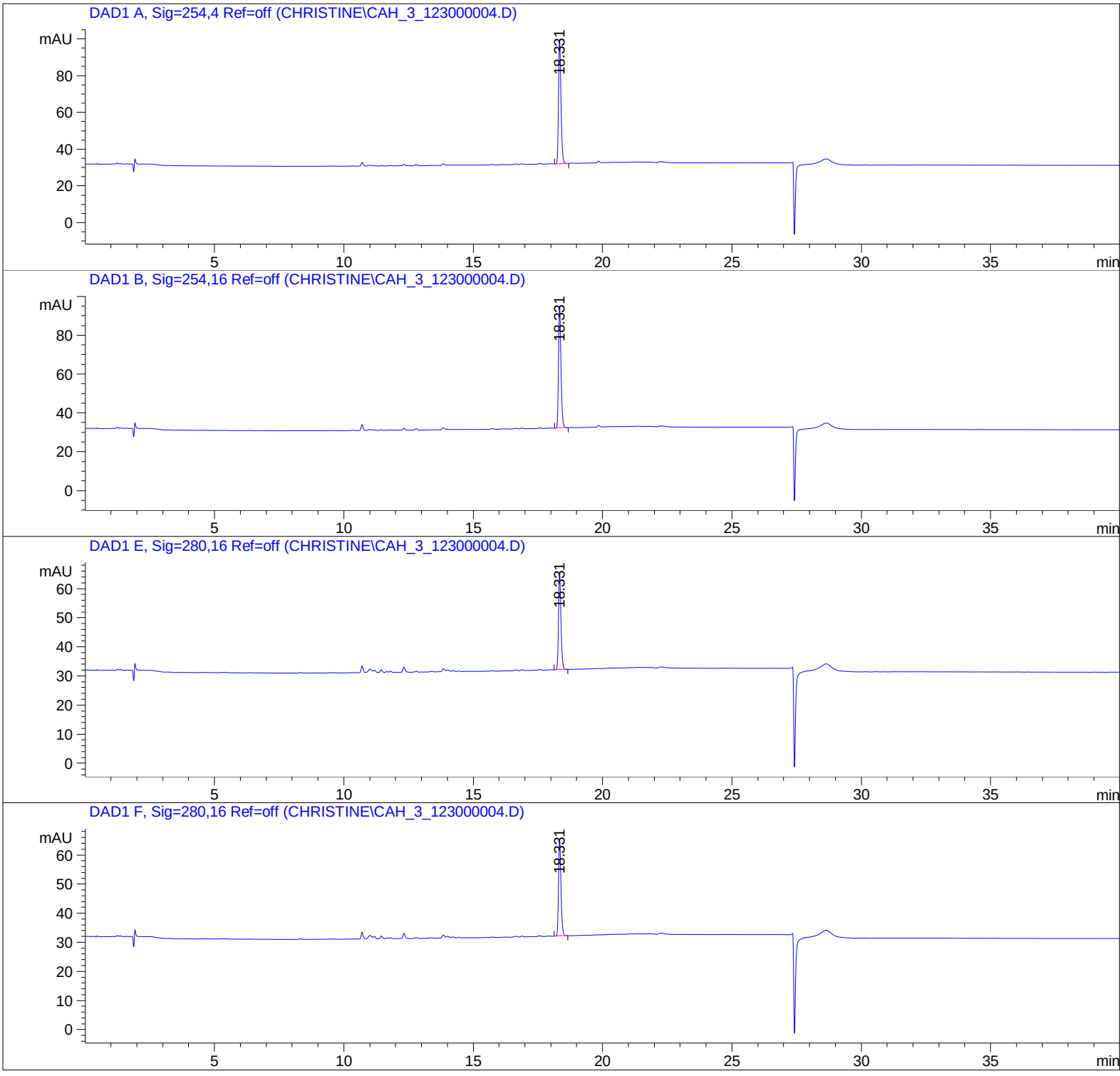
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_123000004.D
Sample Name: CAH_3_123

HPLC for compound 29

==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 10/9/2013 11:38:16 AM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 10/9/2013 11:36:20 AM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_3_123000004.D\DA.M (MASTERMETHOD.M)
Last changed : 10/9/2013 3:39:32 PM by Christine



Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_123000004.D
Sample Name: CAH_3_123

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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.331	BB	0.1006	444.65436	67.72849	100.0000
Totals :				444.65436	67.72849	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.331	BB	0.1008	414.83444	63.04776	100.0000
Totals :				414.83444	63.04776	

Signal 3: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.331	BB	0.1004	220.03041	33.57787	100.0000
Totals :				220.03041	33.57787	

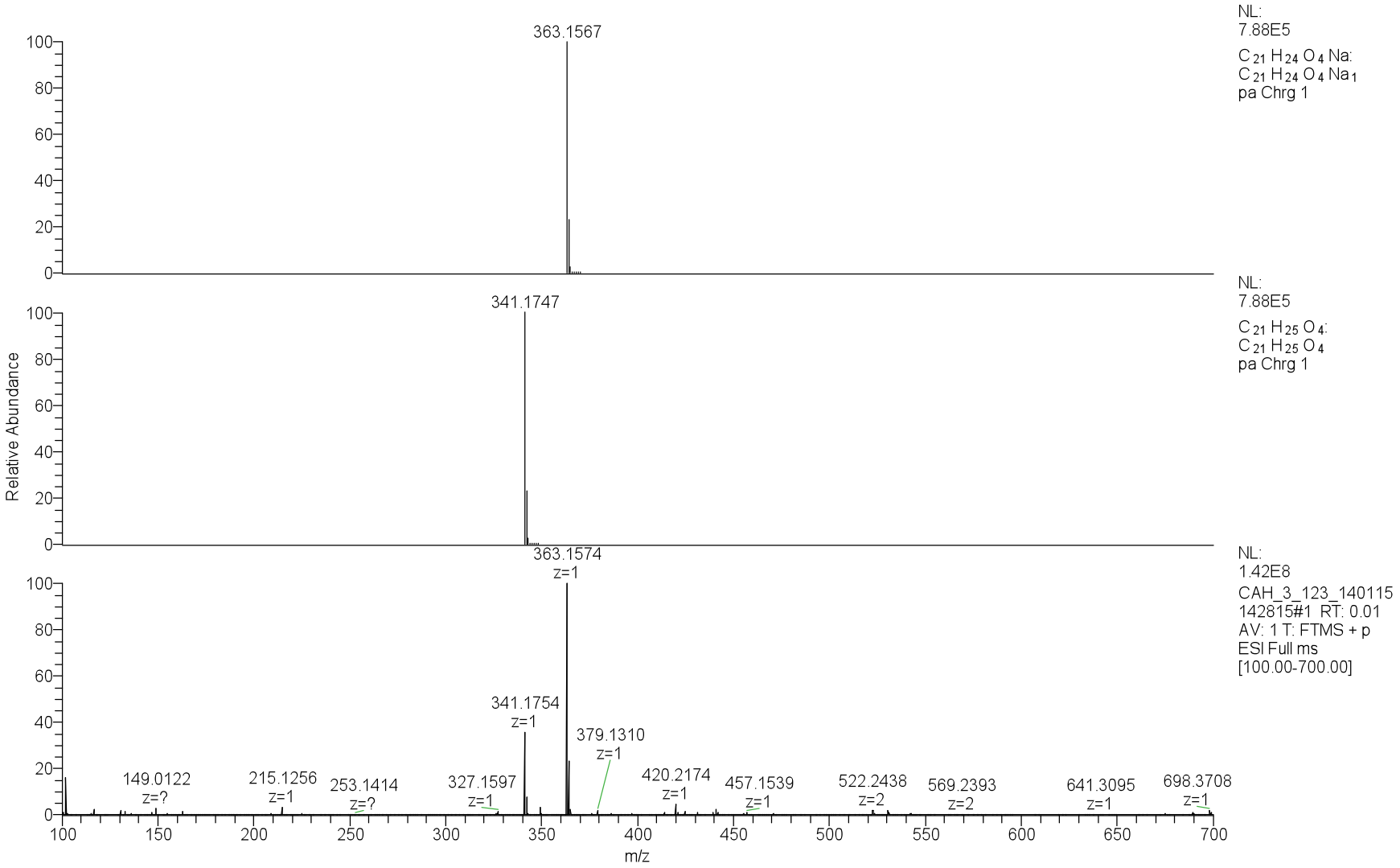
Signal 4: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.331	BB	0.1004	220.03041	33.57787	100.0000
Totals :				220.03041	33.57787	

==========

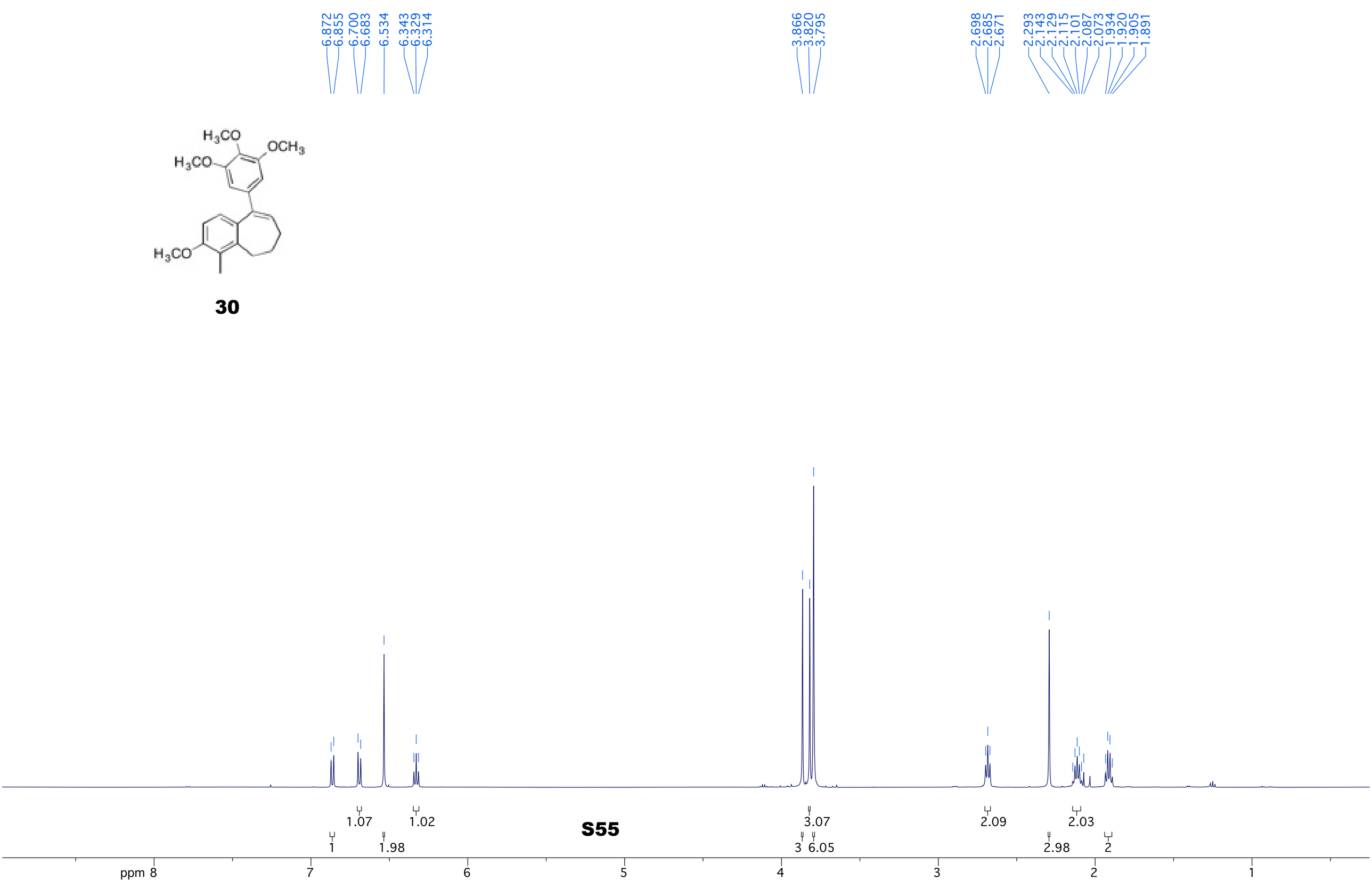
*** End of Report ***

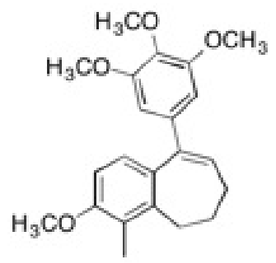
HRMS for compound 29



H3CO
OCH3
H3CO
H3CO

30


6.872
6.855
6.700
6.683
6.534
6.343
6.329
6.314
3.866
3.820
3.795
2.698
2.685
2.671
2.293
2.143
2.129
2.115
2.101
2.087
2.073
1.934
1.920
1.905
1.891
1.07
1.02
3.07
2.09
2.03
1
1.98
3
6.05
2.98
2
ppm 8
7
6
5
4
3
2
1



30

156.461
152.840
143.477
141.610
138.583
137.309
132.938
127.374
126.449
123.129
107.422
105.285
60.837
56.083
55.382
34.038
27.741
25.489
11.774

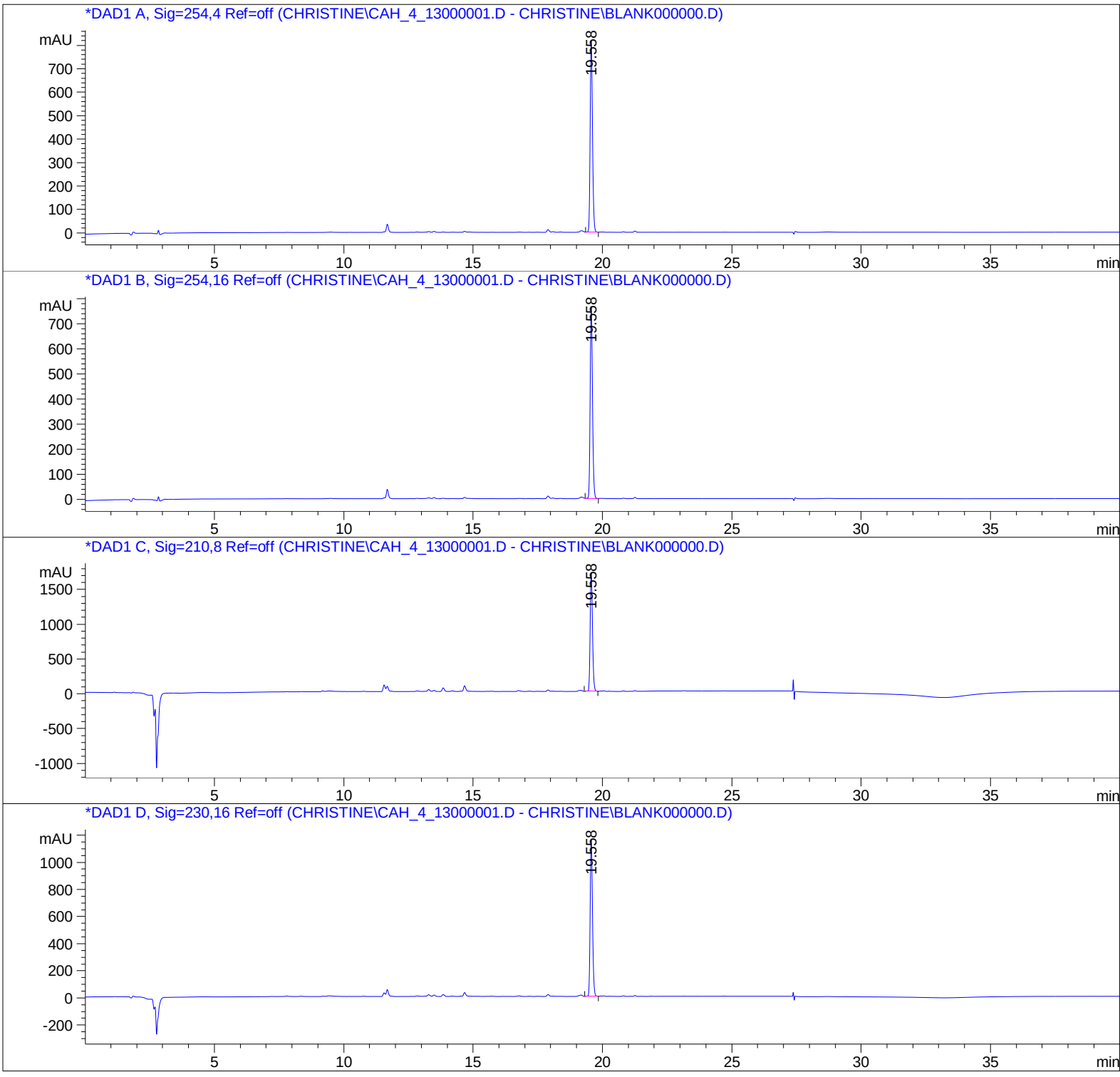
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_13000001.D
Sample Name: CAH_4_13

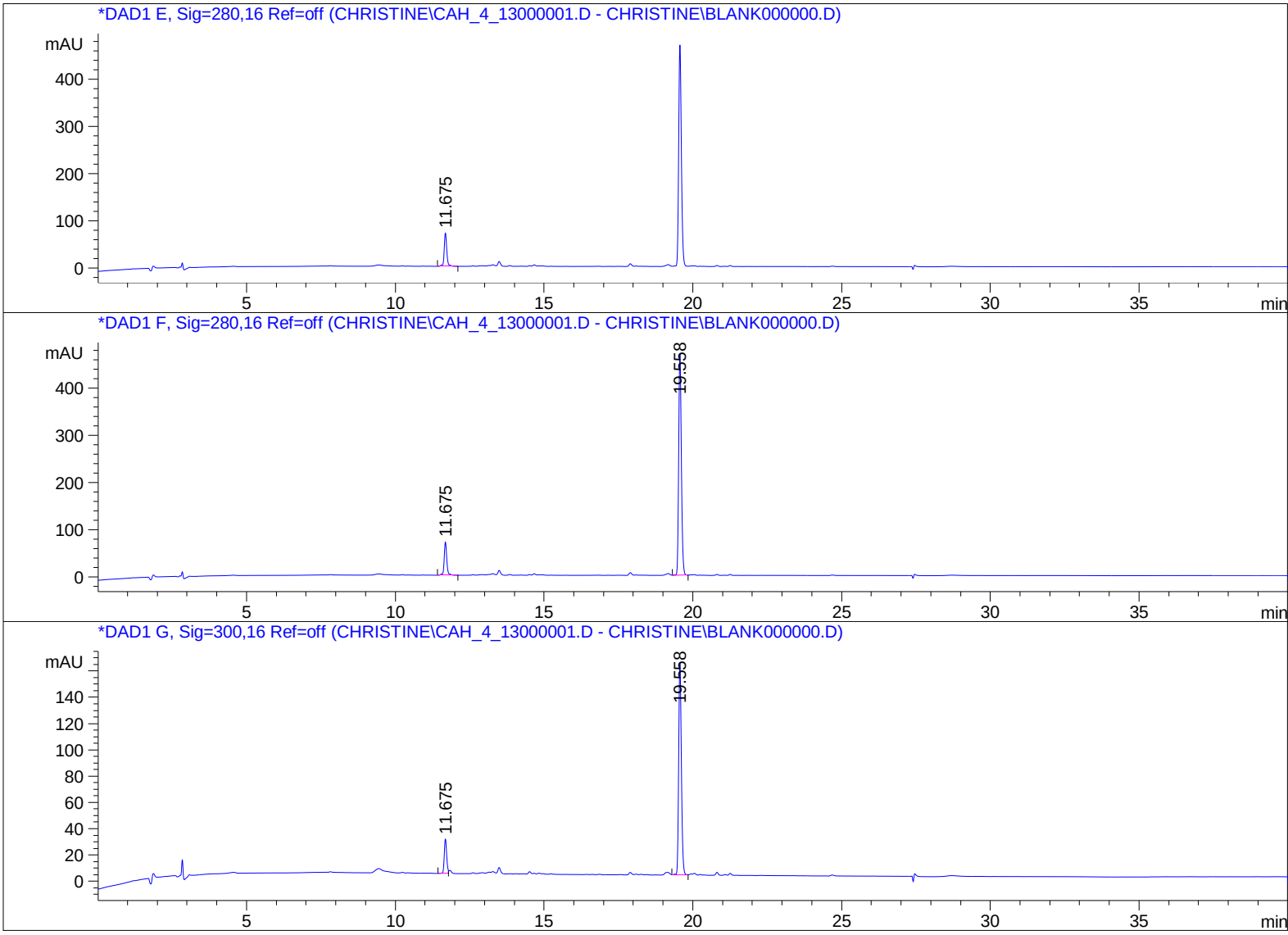
HPLC for compound 30

==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 2/25/2014 9:28:56 AM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 8/6/2013 10:18:10 PM by Blake
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_4_13000001.D\DA.M (MASTERMETHOD.M)
Last changed : 2/25/2014 10:59:23 AM by Christine



Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_13000001.D
Sample Name: CAH_4_13



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.558	VV	0.0950	5127.37158	819.62360	100.0000
Totals :				5127.37158	819.62360	

S58

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_13000001.D
Sample Name: CAH_4_13

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.558	VV	0.0950	4792.61084	765.89771	100.0000
Totals :				4792.61084	765.89771	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.558	VV	0.0978	1.07562e4	1701.31848	100.0000
Totals :				1.07562e4	1701.31848	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.558	VV	0.0952	7260.10791	1157.33716	100.0000
Totals :				7260.10791	1157.33716	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.675	BB	0.0895	420.21866	70.51888	100.0000
Totals :				420.21866	70.51888	

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_13000001.D
Sample Name: CAH_4_13

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.675	BB	0.0895	420.21866	70.51888	12.5043
2	19.558	VV	0.0950	2940.38770	469.76230	87.4957
Totals :				3360.60635	540.28118	

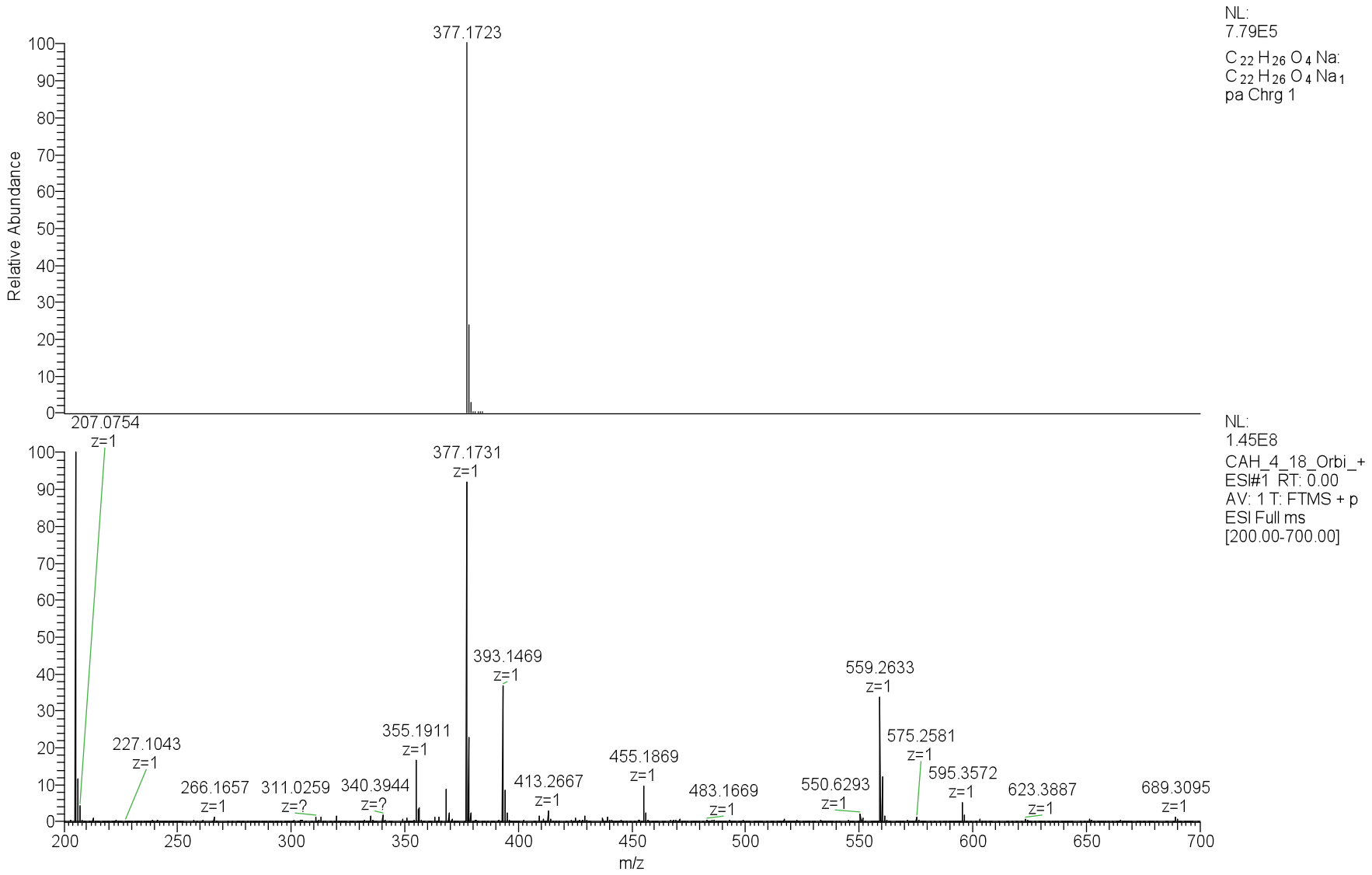
Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

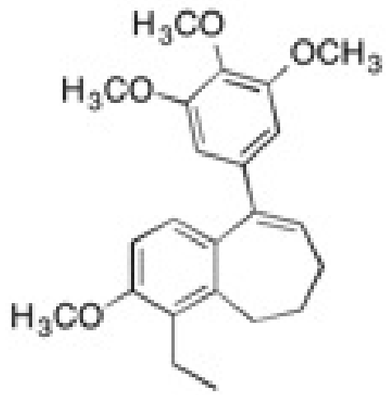
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.675	BV	0.0861	149.43964	26.37713	12.7927
2	19.558	VV	0.0955	1018.72174	161.60686	87.2073
Totals :				1168.16138	187.98399	

*** End of Report ***

S60

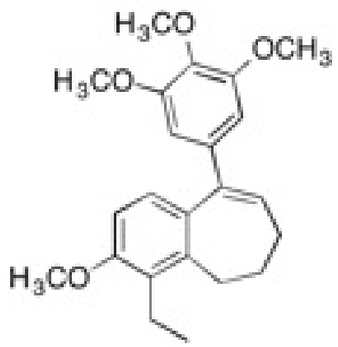
HRMS for compound 30




H3CO
OCH3
H3CO
H3CO

31

6.860
6.843
6.705
6.688
6.522
6.336
6.321
6.307
3.863
3.831
3.805
2.808
2.793
2.778
2.763
2.704
2.691
2.677
2.164
2.150
2.136
2.122
2.108
2.092
1.942
1.928
1.914
1.899
1.198
1.183
1.168
1.05
1.01
3
2.01
2.04
3.08
1
2.09
3.03
6.02
2
2.02
ppm 9
8
7
6
5
4
3
2
1
0
-1



31

156.508
153.108
143.837
141.290
138.974
137.592
133.415
129.894
127.857
126.720
107.825
105.646
77.595
77.342
77.087
61.162
56.447
55.658
35.261
27.490
25.733
19.996
15.200

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_21000000.D
Sample Name: CAH_4_21

HPLC for compound 31

```
==========================================================================
Acq. Operator   : Christine
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/25/2014 1:47:25 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 2/25/2014 1:25:25 PM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_4_21000000.D\DA.M (MASTERMETHOD.M)
Last changed    : 2/25/2014 2:37:40 PM by Christine
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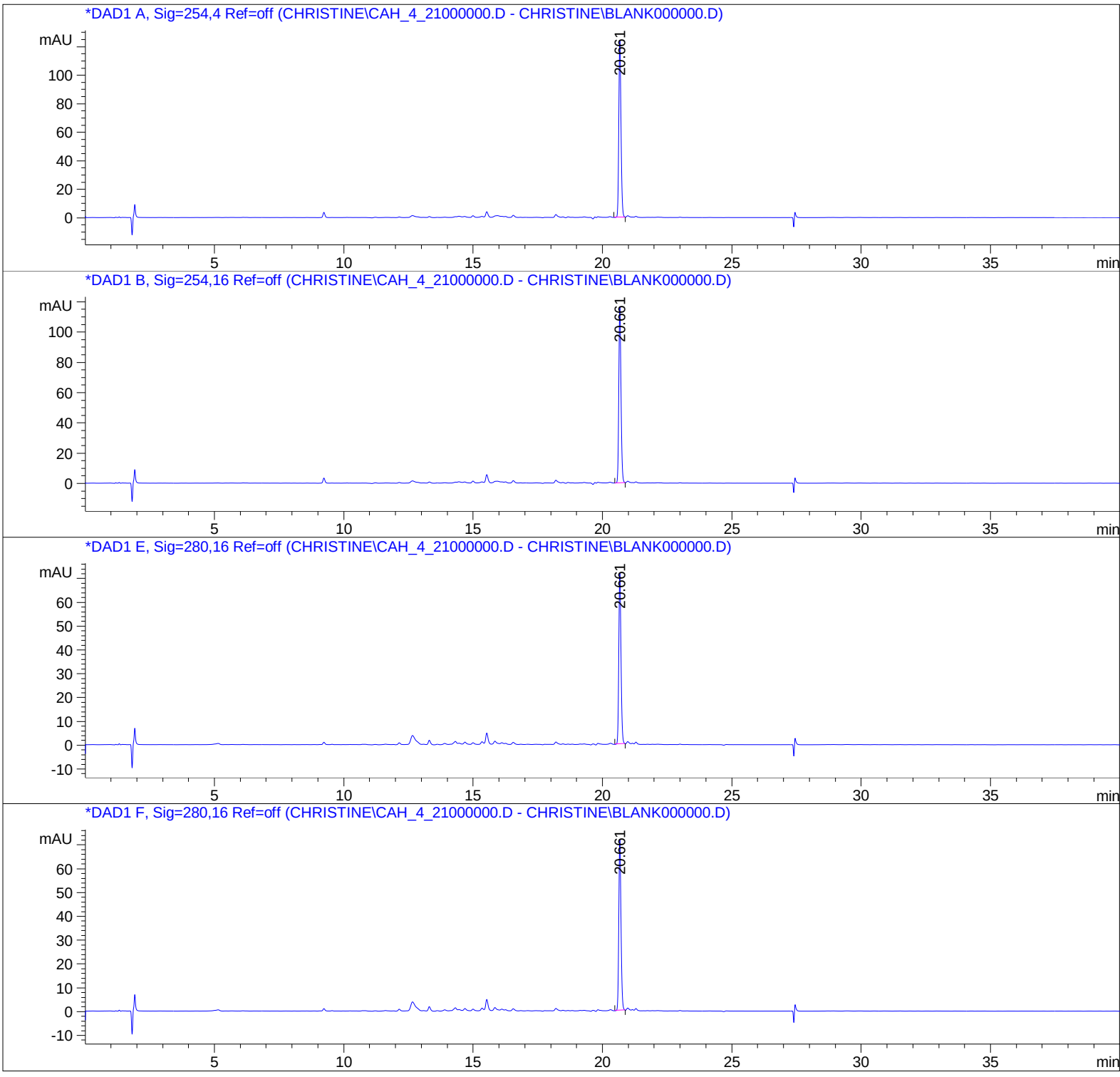
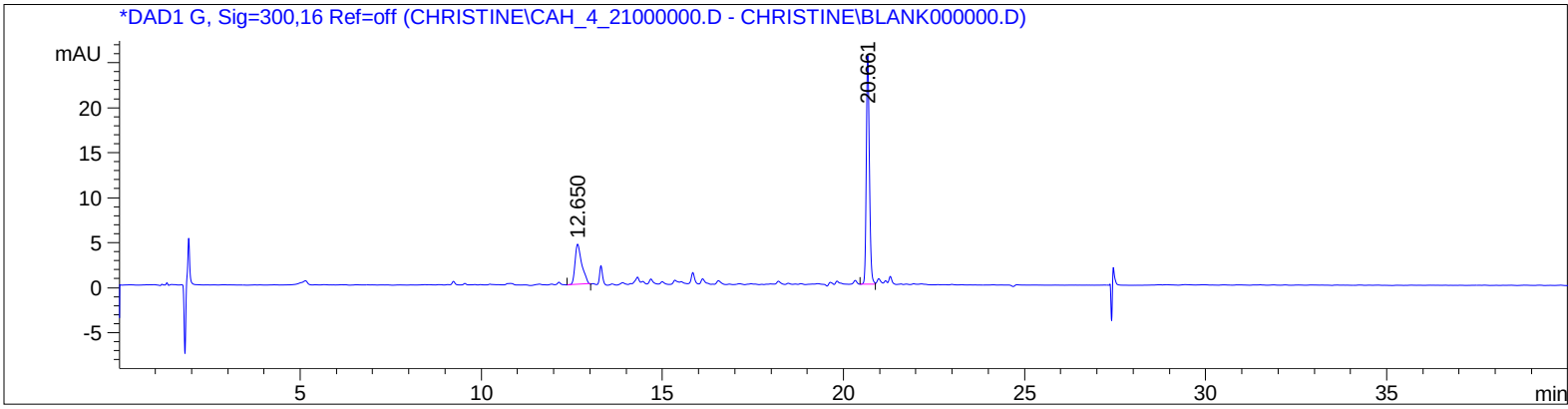


Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_21000000.D
Sample Name: CAH_4_21



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.661	BV	0.0908	733.27759	124.40224	100.0000
Totals :				733.27759	124.40224	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.661	BV	0.0906	685.97784	116.60737	100.0000
Totals :				685.97784	116.60737	

Signal 3: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_21000000.D
Sample Name: CAH_4_21

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.661	BV	0.0908	424.10693	71.94486	100.0000
Totals :				424.10693	71.94486	

Signal 4: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.661	BV	0.0908	424.10693	71.94486	100.0000
Totals :				424.10693	71.94486	

Signal 5: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.650	BB	0.1914	58.18542	4.48376	27.7229
2	20.661	BV	0.0916	151.69656	25.41301	72.2771
Totals :				209.88199	29.89677	

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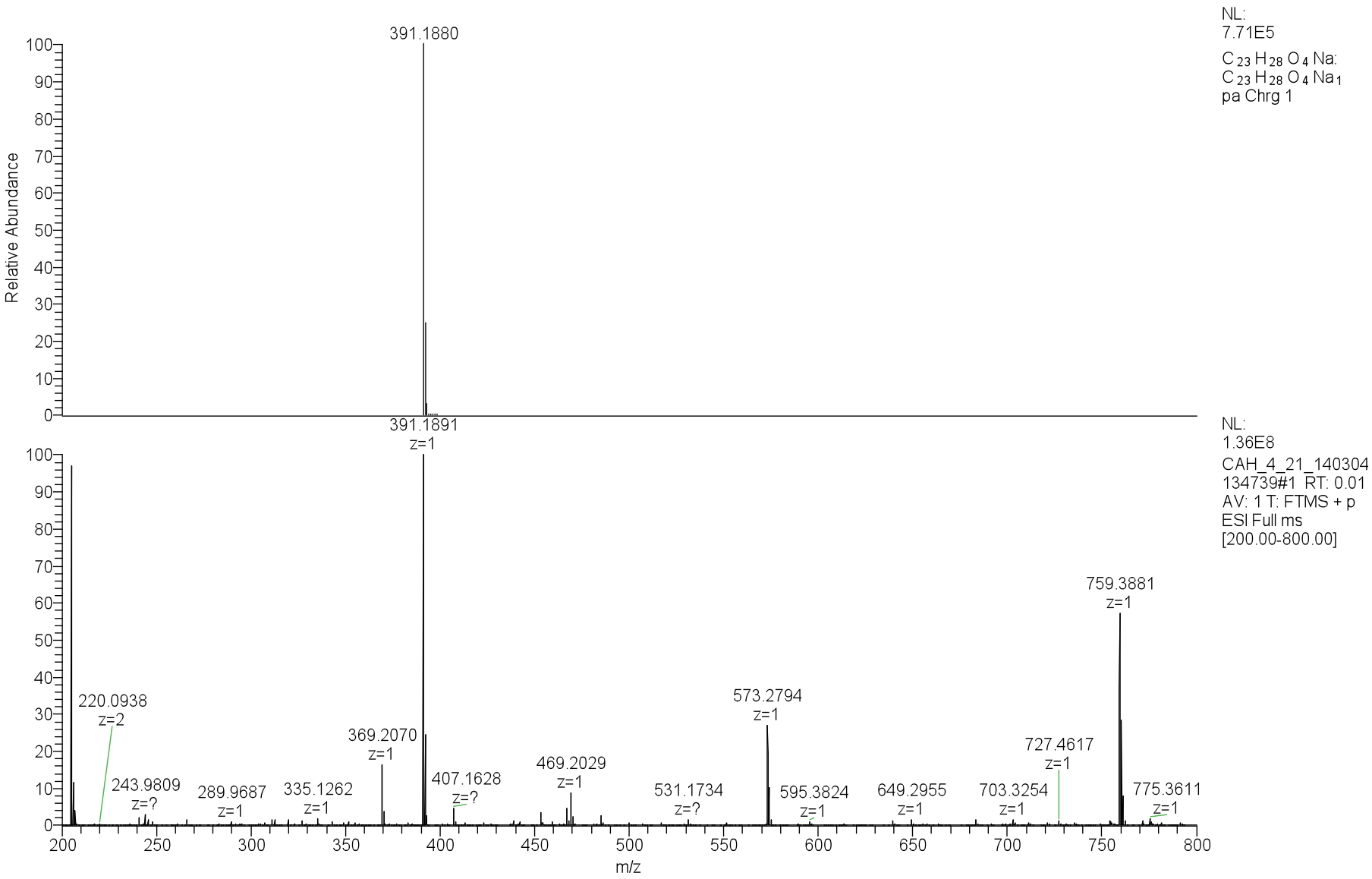
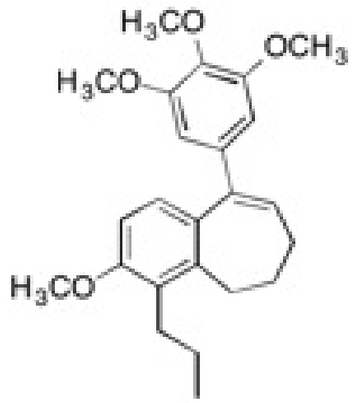
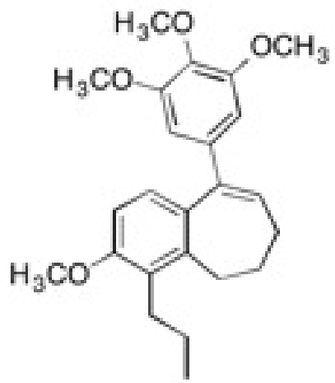
*** End of Report ***

HRMS for compound 31





32



32

156.444
152.831
143.537
141.269
138.697
137.309
133.069
128.256
127.596
126.430
107.481
105.325
77.280
77.025
76.771
60.899
56.172
55.367
34.951
28.608
27.330
25.455
23.772
14.545

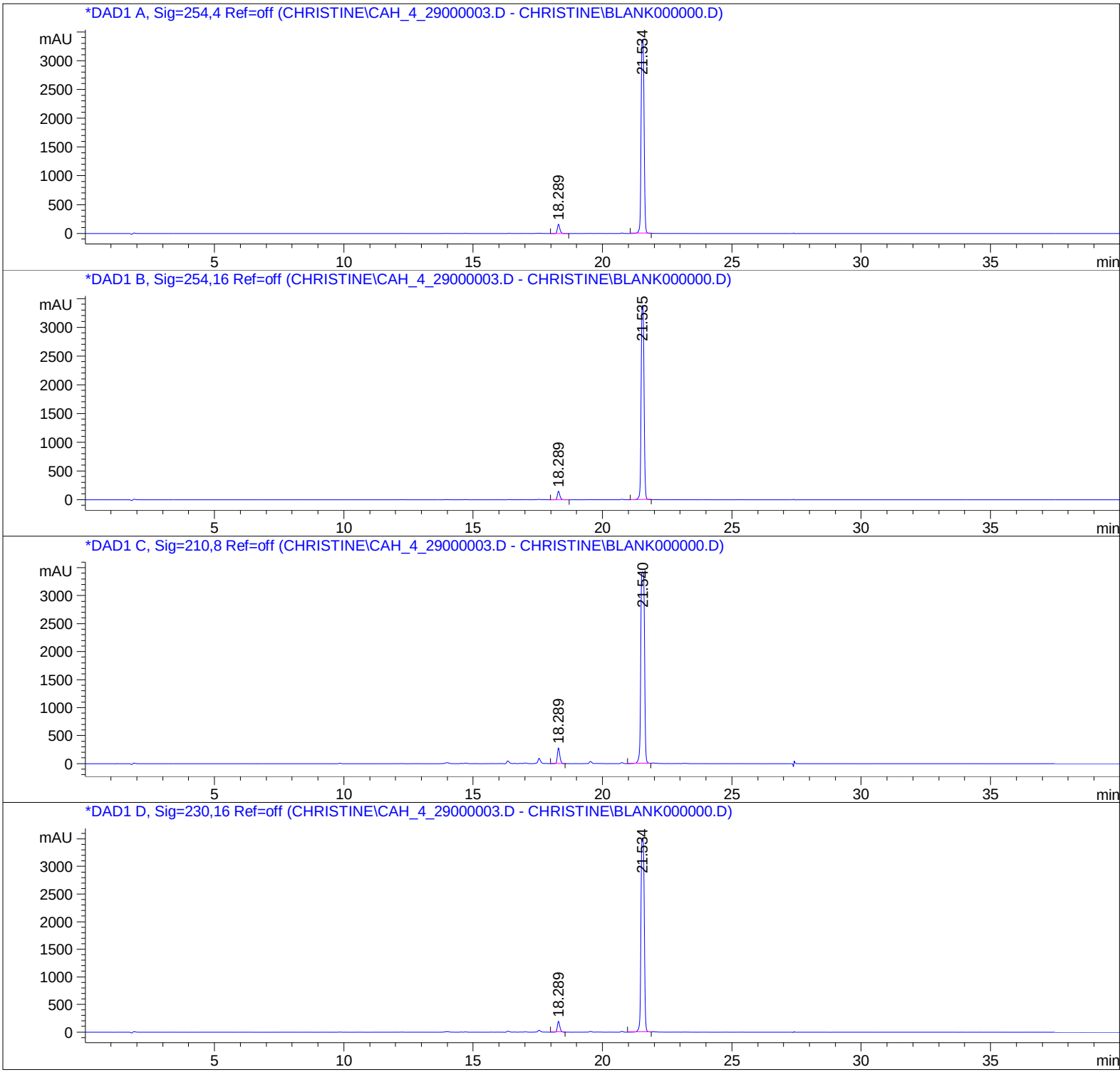
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_29000003.D
Sample Name: CAH_4_29

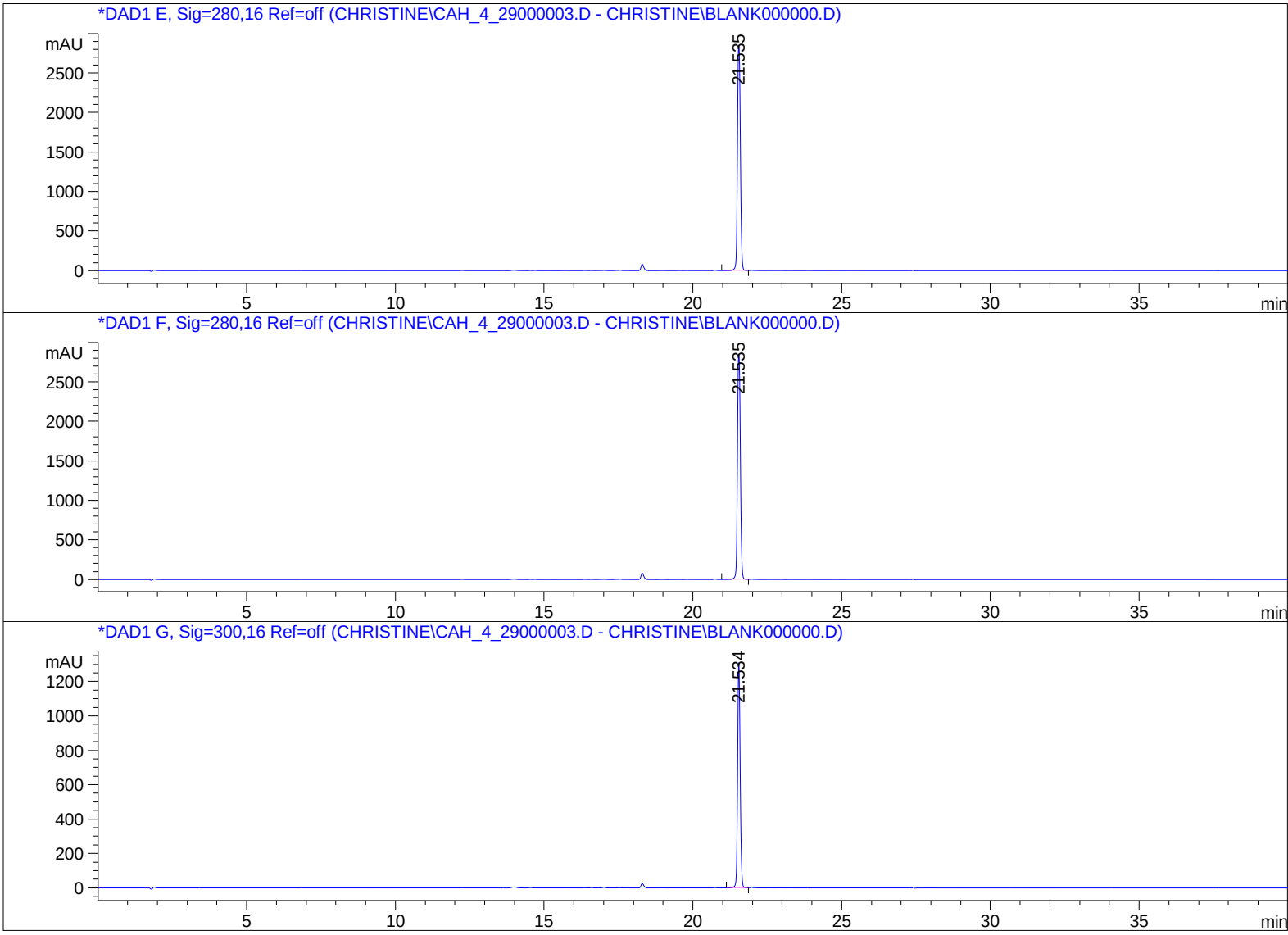
HPLC for compound 32

==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/19/2014 3:07:57 PM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 5/19/2014 3:05:49 PM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_4_29000003.D\DA.M (MASTERMETHOD.M)
Last changed : 5/19/2014 4:47:24 PM by Christine



Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_29000003.D
Sample Name: CAH_4_29



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.289	BB	0.0993	1064.28625	160.63548	3.8307
2	21.534	BB	0.1285	2.67186e4	3368.47388	96.1693
Totals :				2.77828e4	3529.10936	

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_29000003.D
Sample Name: CAH_4_29

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.289	BB	0.1013	982.76642	148.33012	3.6253
2	21.535	BB	0.1243	2.61255e4	3375.99023	96.3747
Totals :				2.71083e4	3524.32036	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.289	BV	0.1015	1865.43298	280.66962	5.1837
2	21.540	BV	0.1622	3.41209e4	3424.46191	94.8163
Totals :				3.59864e4	3705.13153	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.289	BV	0.1014	1306.26282	196.81532	4.0403
2	21.534	BV	0.1430	3.10247e4	3515.33618	95.9597
Totals :				3.23309e4	3712.15150	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.535	BV	0.1074	1.94384e4	2855.78076	100.0000
Totals :				1.94384e4	2855.78076	

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_29000003.D
Sample Name: CAH_4_29

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

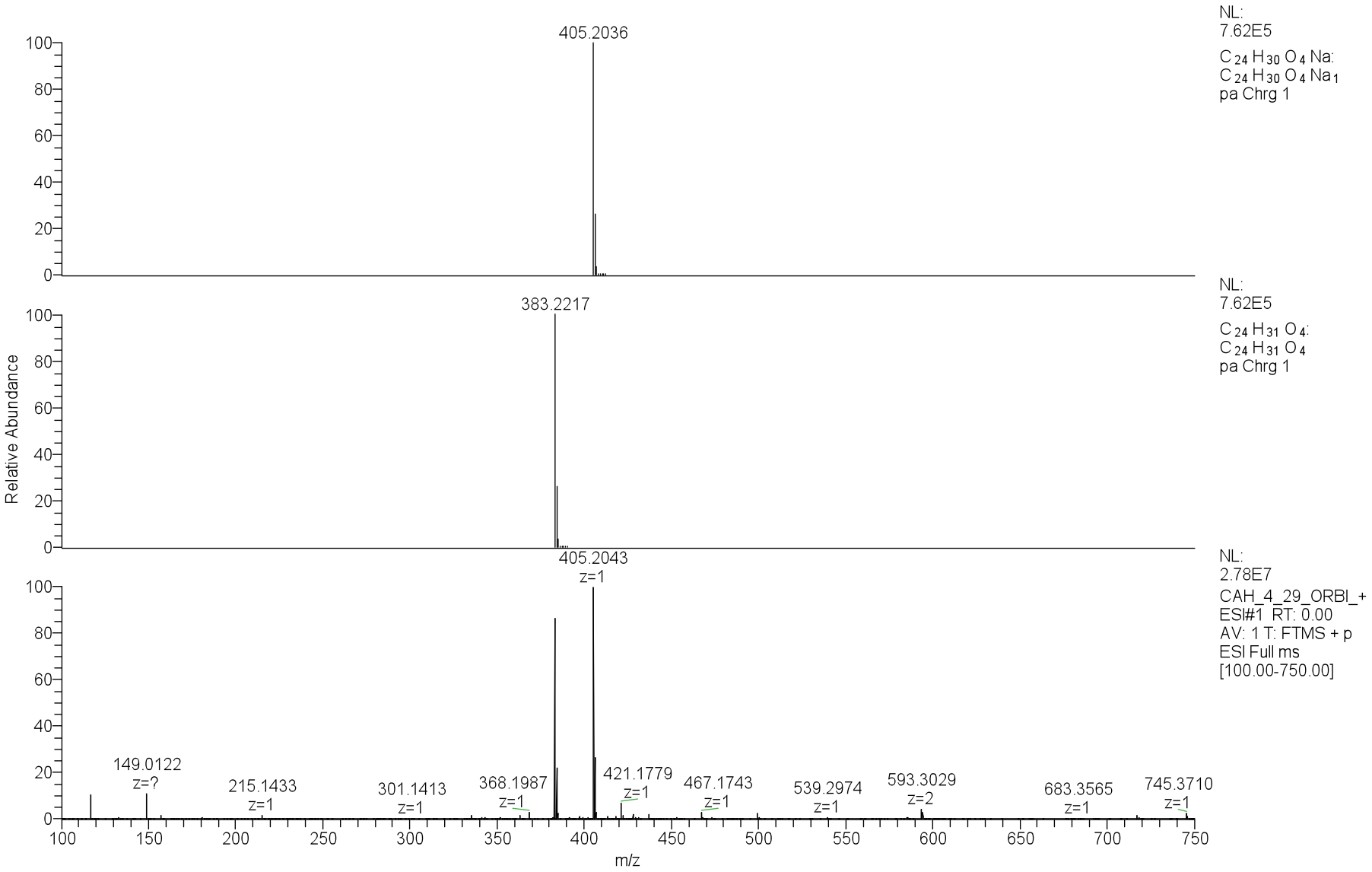
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.535	BV	0.1074	1.94384e4	2855.78076	100.0000
Totals :				1.94384e4	2855.78076	

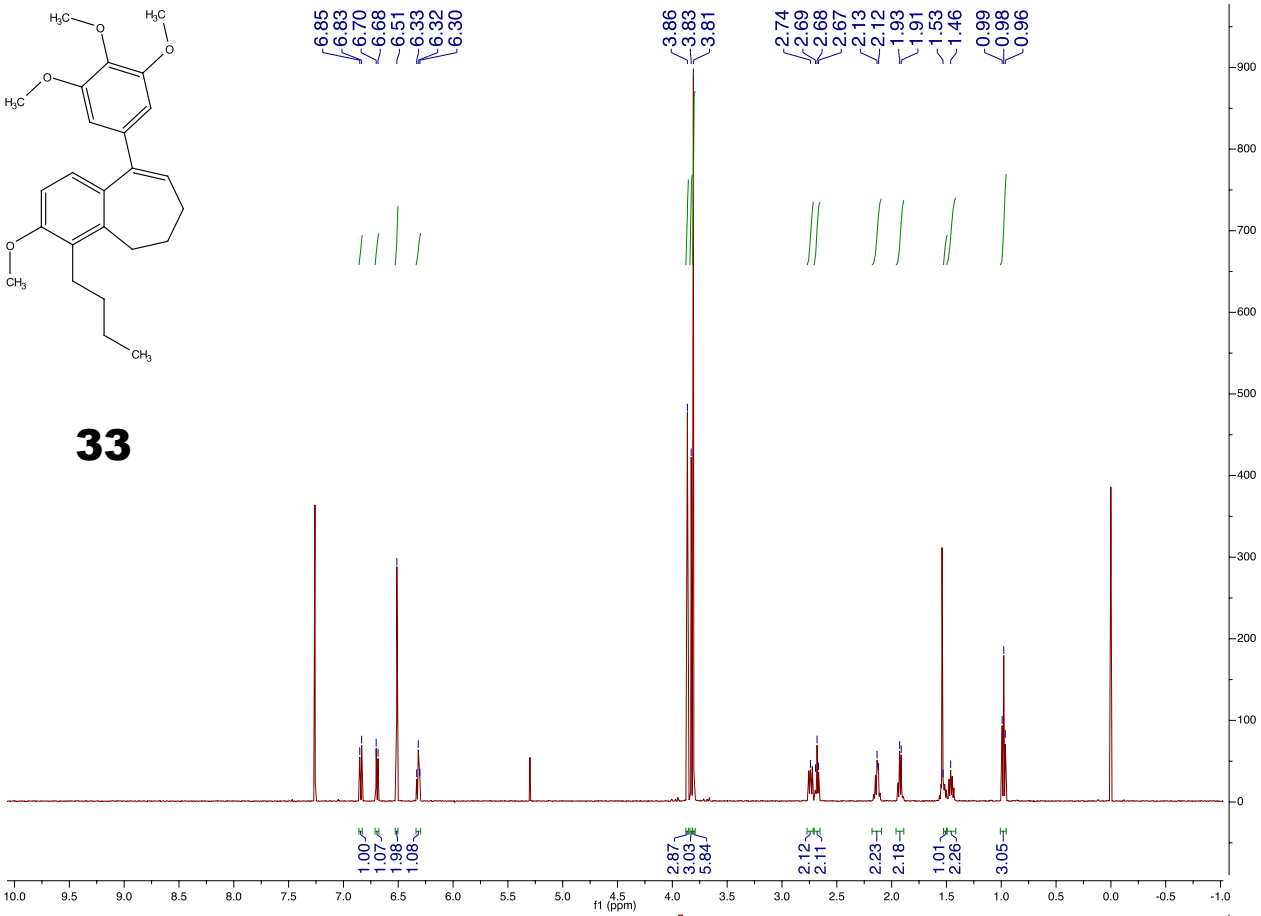
Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.534	BV	0.0938	7832.92432	1309.62927	100.0000
Totals :				7832.92432	1309.62927	

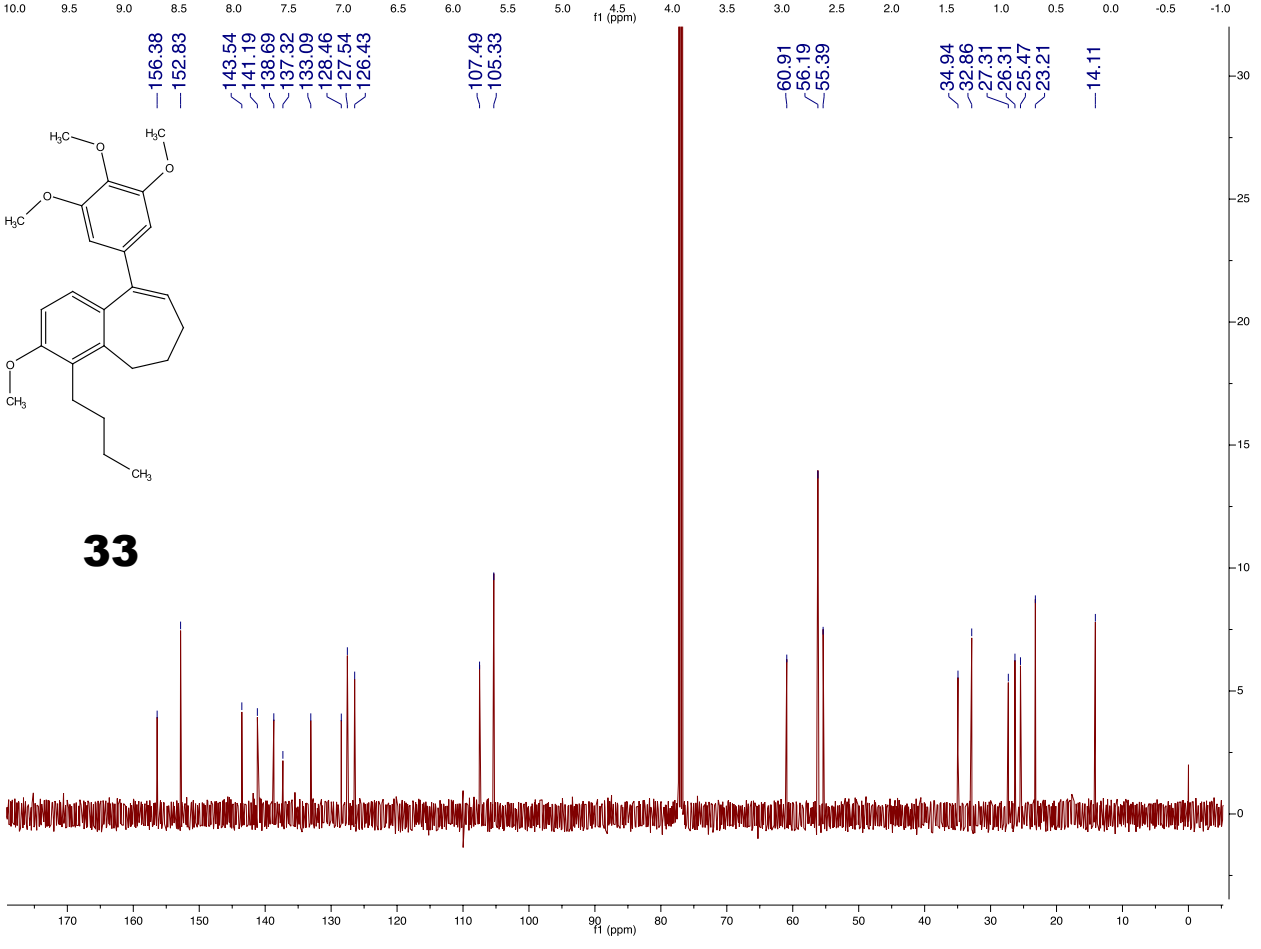
*** End of Report ***

HRMS for compound 32




6.85
6.83
6.70
6.68
6.51
6.33
6.32
6.30
3.86
3.83
3.81
2.74
2.69
2.68
2.67
2.13
2.12
1.93
1.91
1.53
1.46
0.99
0.98
0.96
1.00
1.07
1.98
1.08
2.87
3.03
5.84
2.12
2.11
2.23
2.18
1.01
2.26
3.05
f1 (ppm)

33


156.38
152.83
143.54
141.19
138.69
137.32
133.09
128.46
127.54
126.43
107.49
105.33
60.91
56.19
55.39
34.94
32.86
27.31
26.31
25.47
23.21
14.11
f1 (ppm)

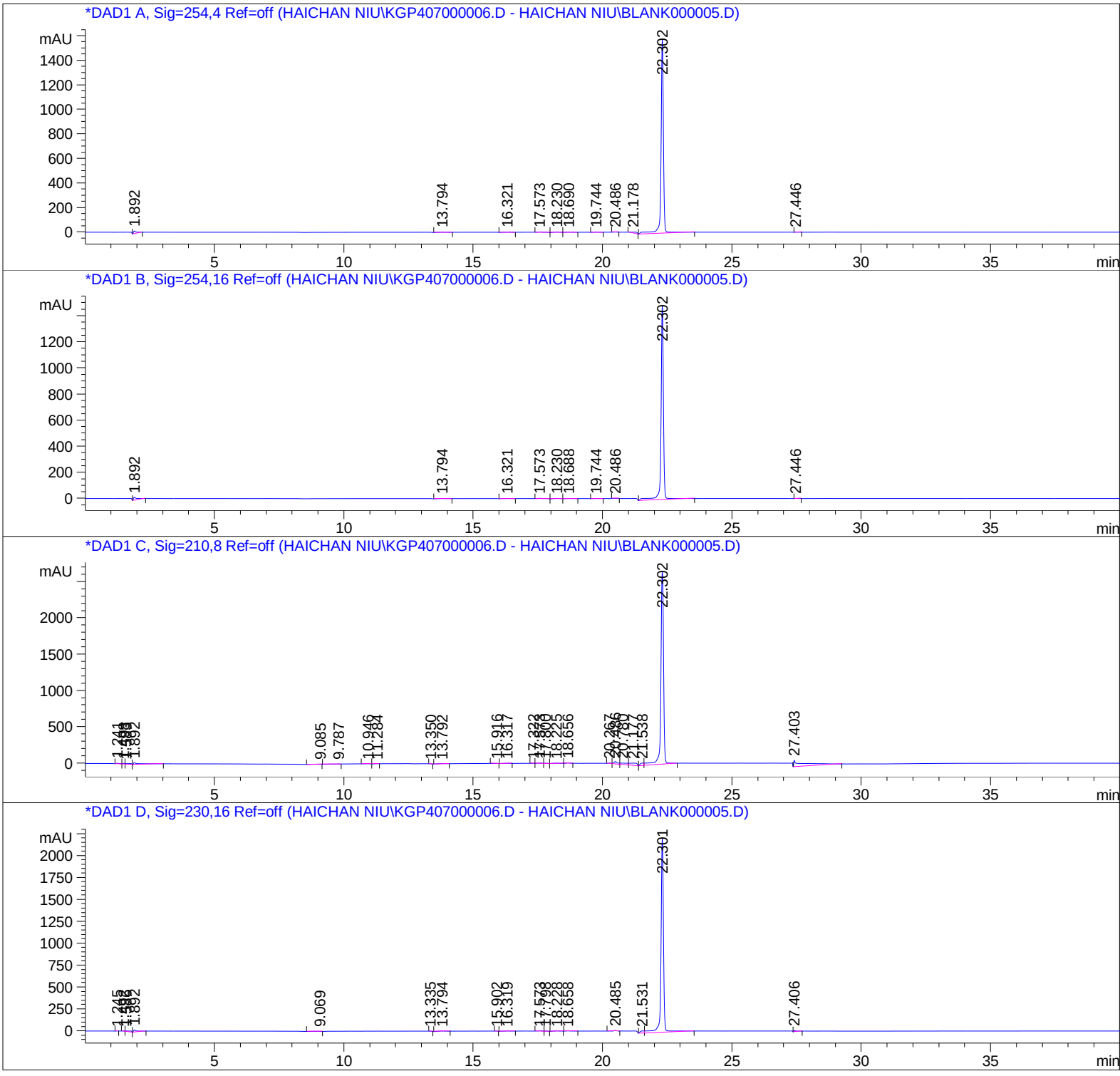
33

Data File C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D
Sample Name: KGP407

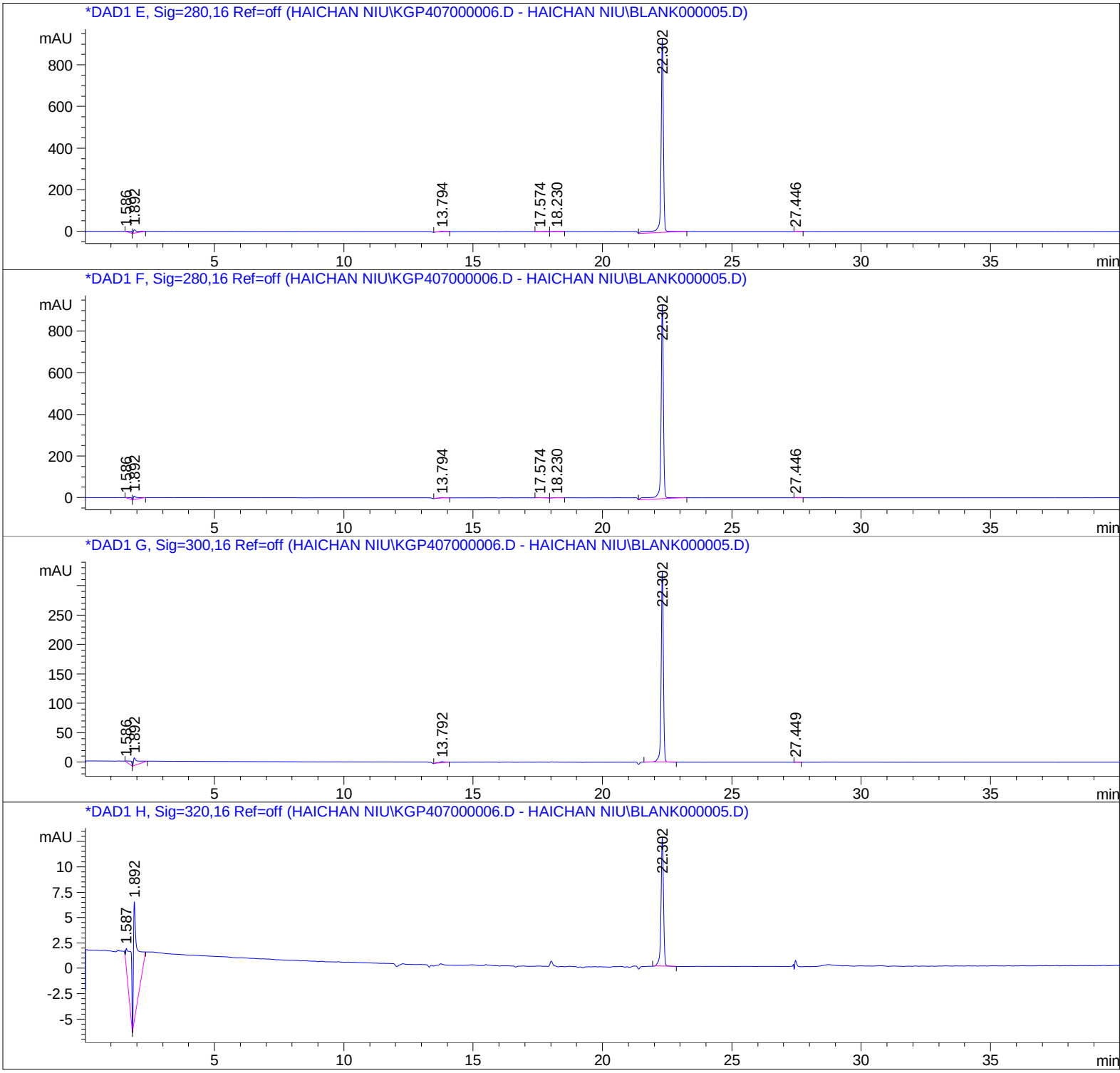
HPLC for compound 33

==========

Acq. Operator : HAICHAN NIU
Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/22/2014 5:52:09 PM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 5/22/2014 5:43:44 PM by HAICHAN NIU
Analysis Method : C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D\DA.M (MASTERMETHOD.M)
Last changed : 5/22/2014 7:08:38 PM by HAICHAN NIU



Data File C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D
Sample Name: KGP407



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

S77

Data File C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D
Sample Name: KGP407

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.892	BB	0.1220	205.61472	23.05705	1.8377
2	13.794	BB	0.2701	37.55608	1.82508	0.3357
3	16.321	BB	0.2518	29.26817	1.48607	0.2616
4	17.573	BB	0.2588	59.74598	2.94312	0.5340
5	18.230	BV	0.2229	63.97398	3.75268	0.5718
6	18.690	VB	0.2388	31.14396	1.72357	0.2784
7	19.744	BB	0.1574	17.63064	1.54186	0.1576
8	20.486	BB	0.0941	8.51472	1.41805	0.0761
9	21.178	BB	0.2529	172.74390	8.96248	1.5439
10	22.302	BB	0.0998	1.05476e4	1581.28418	94.2714
11	27.446	BB	0.0992	14.75580	2.12002	0.1319
Totals :				1.11886e4	1630.11415	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.892	BB	0.1472	257.07364	23.21323	2.4674
2	13.794	BB	0.2658	42.60092	2.10777	0.4089
3	16.321	BB	0.2519	29.39858	1.49186	0.2822
4	17.573	BB	0.2544	55.46112	2.78468	0.5323
5	18.230	BV	0.2228	58.92264	3.45830	0.5655
6	18.688	VB	0.2361	29.00475	1.62596	0.2784
7	19.744	BB	0.1552	15.84321	1.40894	0.1521
8	20.486	BB	0.0947	10.03956	1.65767	0.0964
9	22.302	BB	0.0997	9906.66016	1486.84314	95.0838
10	27.446	BB	0.0952	13.86449	2.04266	0.1331
Totals :				1.04189e4	1526.63420	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.241	BV	0.1166	112.43739	12.53983	0.4377
2	1.498	VV	0.0978	86.76828	12.38144	0.3378
3	1.583	VB	0.2030	212.25829	12.96423	0.8263

Data File C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D
Sample Name: KGP407

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
4	1.892	BB	0.2722	610.39325	28.00976	2.3763
5	9.085	BB	0.3602	37.33203	1.28795	0.1453
6	9.787	BB	0.2183	29.35772	1.72761	0.1143
7	10.946	BB	0.2118	16.74347	1.06277	0.0652
8	11.284	BB	0.0998	9.72657	1.45764	0.0379
9	13.350	BB	0.0951	9.75790	1.69781	0.0380
10	13.792	BB	0.2570	68.78865	3.59853	0.2678
11	15.916	BB	0.1142	30.48277	3.85870	0.1187
12	16.317	BB	0.1950	49.96380	3.36880	0.1945
13	17.322	BV	0.1185	11.81782	1.52267	0.0460
14	17.573	VV	0.1797	82.90123	6.05778	0.3227
15	17.800	VB	0.1581	45.04564	4.68686	0.1754
16	18.225	BV	0.2371	97.94218	5.36237	0.3813
17	18.656	VB	0.1299	51.54269	5.90078	0.2007
18	20.267	BV	0.1402	54.50468	5.57007	0.2122
19	20.486	VV	0.1512	369.62842	33.90767	1.4390
20	20.780	VV	0.2662	439.56110	21.00230	1.7112
21	21.177	VB	0.2569	660.56427	32.80515	2.5716
22	21.538	BV	0.1592	361.84375	36.00524	1.4087
23	22.302	VB	0.1133	1.93617e4	2647.61377	75.3753
24	27.403	BB	0.3710	2875.97852	92.43199	11.1962
Totals :				2.56870e4	2976.82171	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.245	BB	0.0525	6.08144	1.75071	0.0372
2	1.492	BV	0.0864	23.75330	3.93887	0.1453
3	1.586	VB	0.2551	169.37418	8.12729	1.0359
4	1.892	BB	0.1505	300.81332	26.48990	1.8398
5	9.069	BB	0.3211	40.21803	1.57932	0.2460
6	13.335	BB	0.1097	11.15828	1.59429	0.0682
7	13.794	BB	0.2532	86.28983	4.55197	0.5277
8	15.902	BB	0.0896	8.10509	1.39794	0.0496
9	16.319	BB	0.2450	47.99840	2.51118	0.2936
10	17.573	BV	0.1604	43.86290	3.69786	0.2683
11	17.798	VB	0.1637	28.22668	2.79859	0.1726
12	18.228	BB	0.2038	54.62132	3.54339	0.3341
13	18.658	BB	0.1438	20.38937	2.05419	0.1247
14	20.485	BB	0.1017	46.16708	6.93190	0.2824
15	21.531	BV	0.1647	265.59775	25.25007	1.6244
16	22.301	VB	0.1035	1.50731e4	2211.43604	92.1866
17	27.406	BB	0.0975	124.87911	16.64479	0.7638

| Totals : | | | 1.63506e4 | 2324.29829 | |

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.586	BB	0.3822	88.06649	2.77503	1.3521
2	1.892	BB	0.1496	198.73843	17.89013	3.0512
3	13.794	BB	0.2504	67.00623	3.57945	1.0287
4	17.574	BB	0.2352	23.93932	1.31028	0.3675
5	18.230	BB	0.2013	23.77900	1.54734	0.3651
6	22.302	BB	0.0984	6101.15137	931.20624	93.6697
7	27.446	BB	0.1011	10.79483	1.48062	0.1657
Totals :				6513.47568	959.78909	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.586	BB	0.3822	88.06649	2.77503	1.3521
2	1.892	BB	0.1496	198.73843	17.89013	3.0512
3	13.794	BB	0.2504	67.00623	3.57945	1.0287
4	17.574	BB	0.2352	23.93932	1.31028	0.3675
5	18.230	BB	0.2013	23.77900	1.54734	0.3651
6	22.302	BB	0.0984	6101.15137	931.20624	93.6697
7	27.446	BB	0.1011	10.79483	1.48062	0.1657
Totals :				6513.47568	959.78909	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.586	BB	0.3828	66.66472	2.09758	2.9911
2	1.892	BB	0.1638	167.54794	13.59008	7.5176
3	13.792	BB	0.2464	39.83583	2.16750	1.7874
4	22.302	BB	0.0922	1948.63611	324.06201	87.4318
5	27.449	BB	0.0828	6.06465	1.02903	0.2721
Totals :				2228.74926	342.94620	

Data File C:\CHEM32\1\DATA\HAICHAN NIU\KGP407000006.D
Sample Name: KGP407

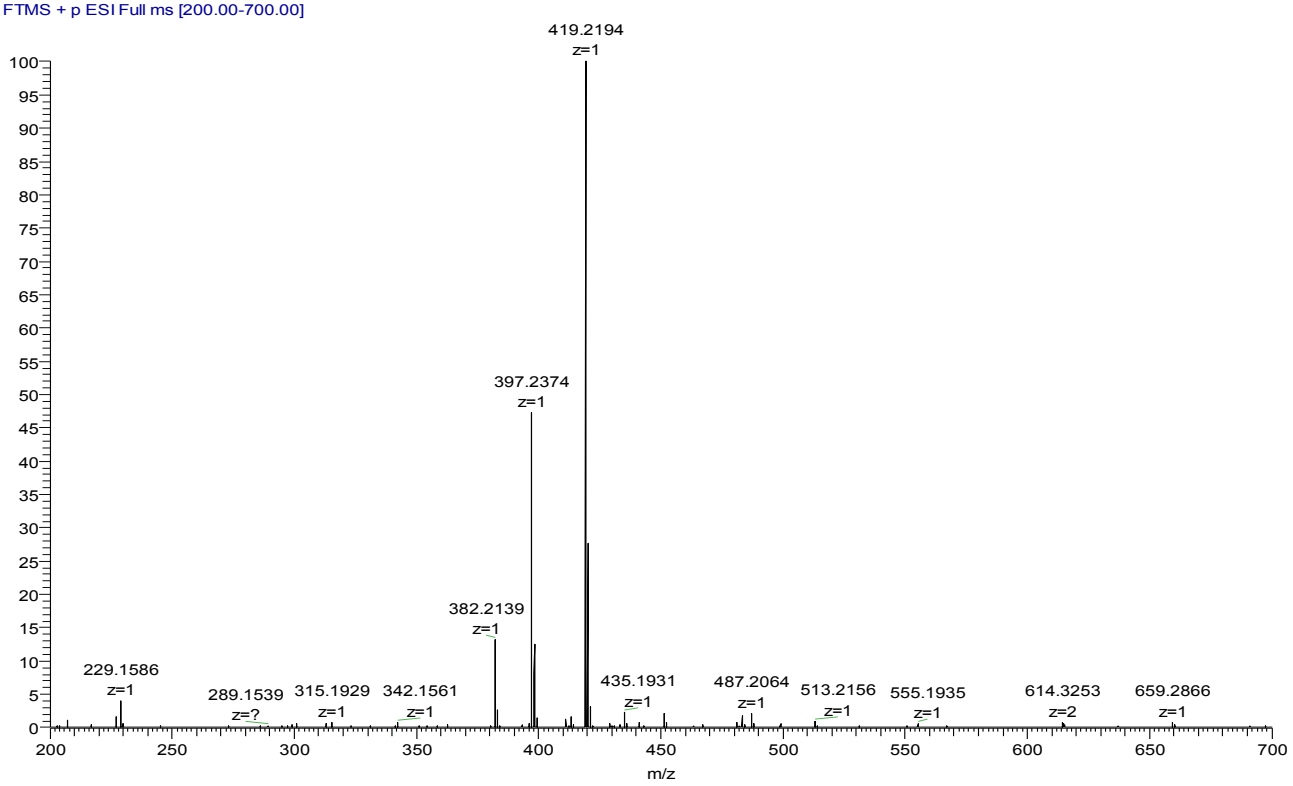
Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

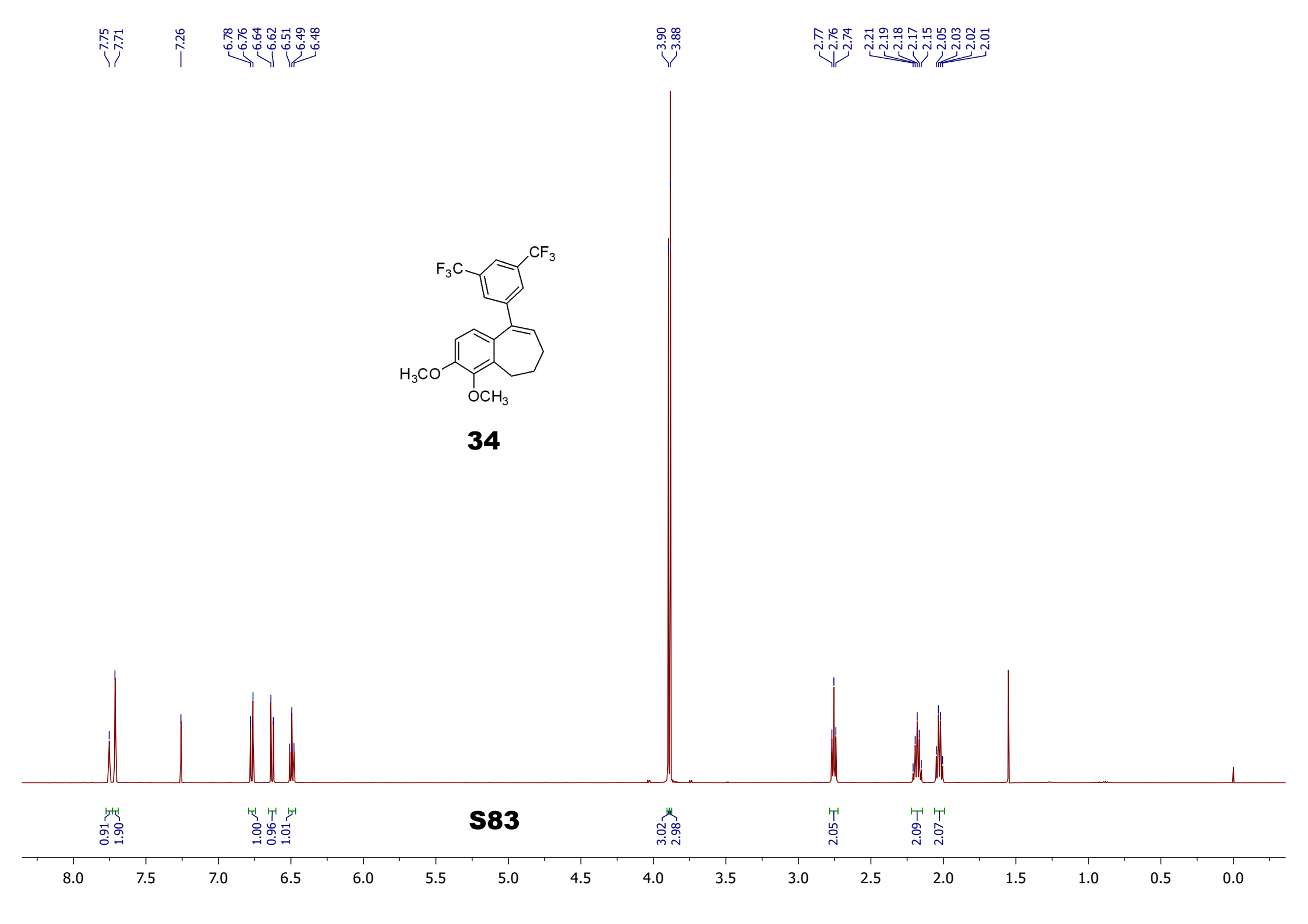
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.587	BB	0.3806	59.15536	1.87249	21.4918
2	1.892	BB	0.1479	130.22919	11.87656	47.3137
3	22.302	BB	0.1031	85.86178	12.66659	31.1945
Totals :				275.24632	26.41563	

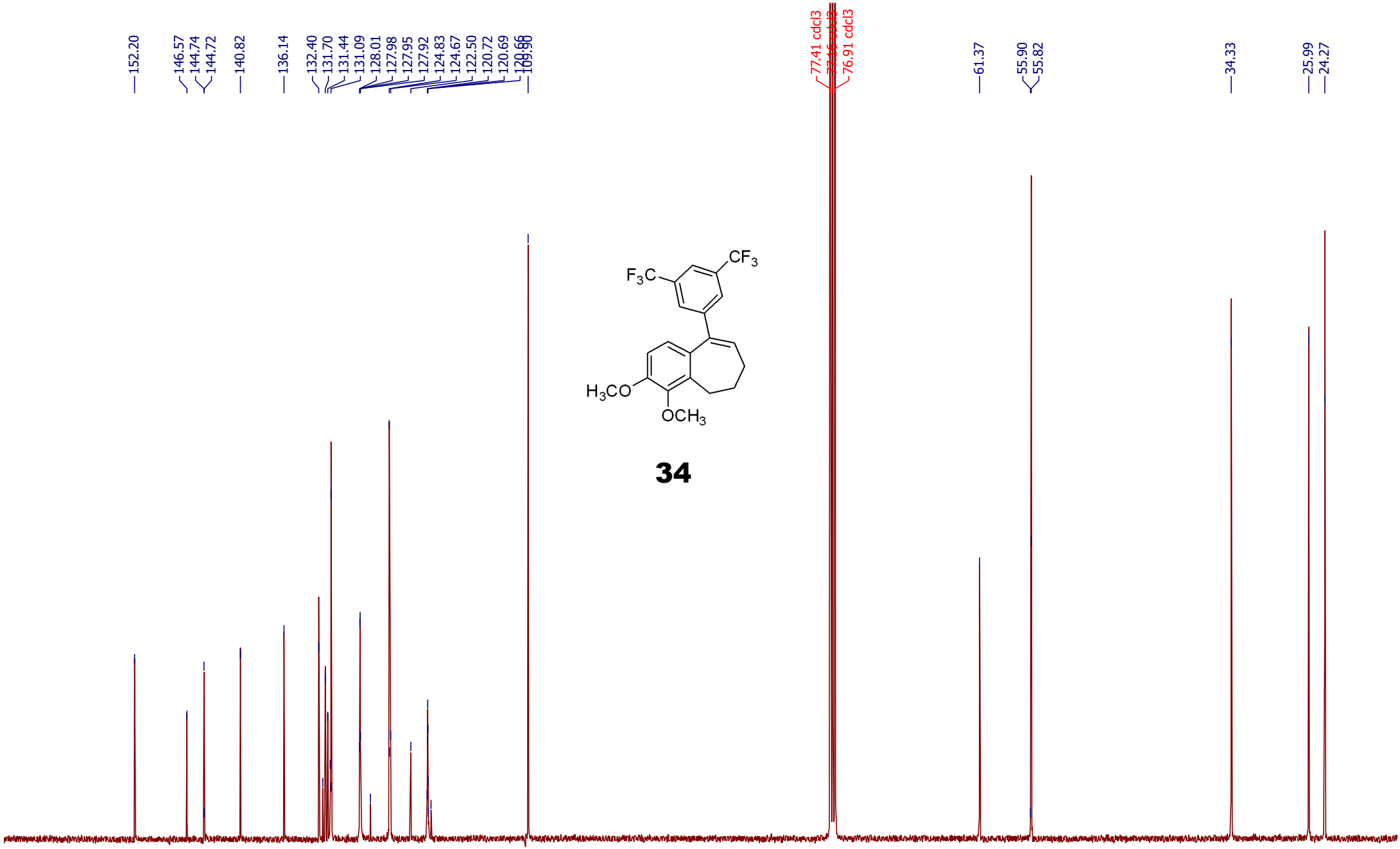
*** End of Report ***

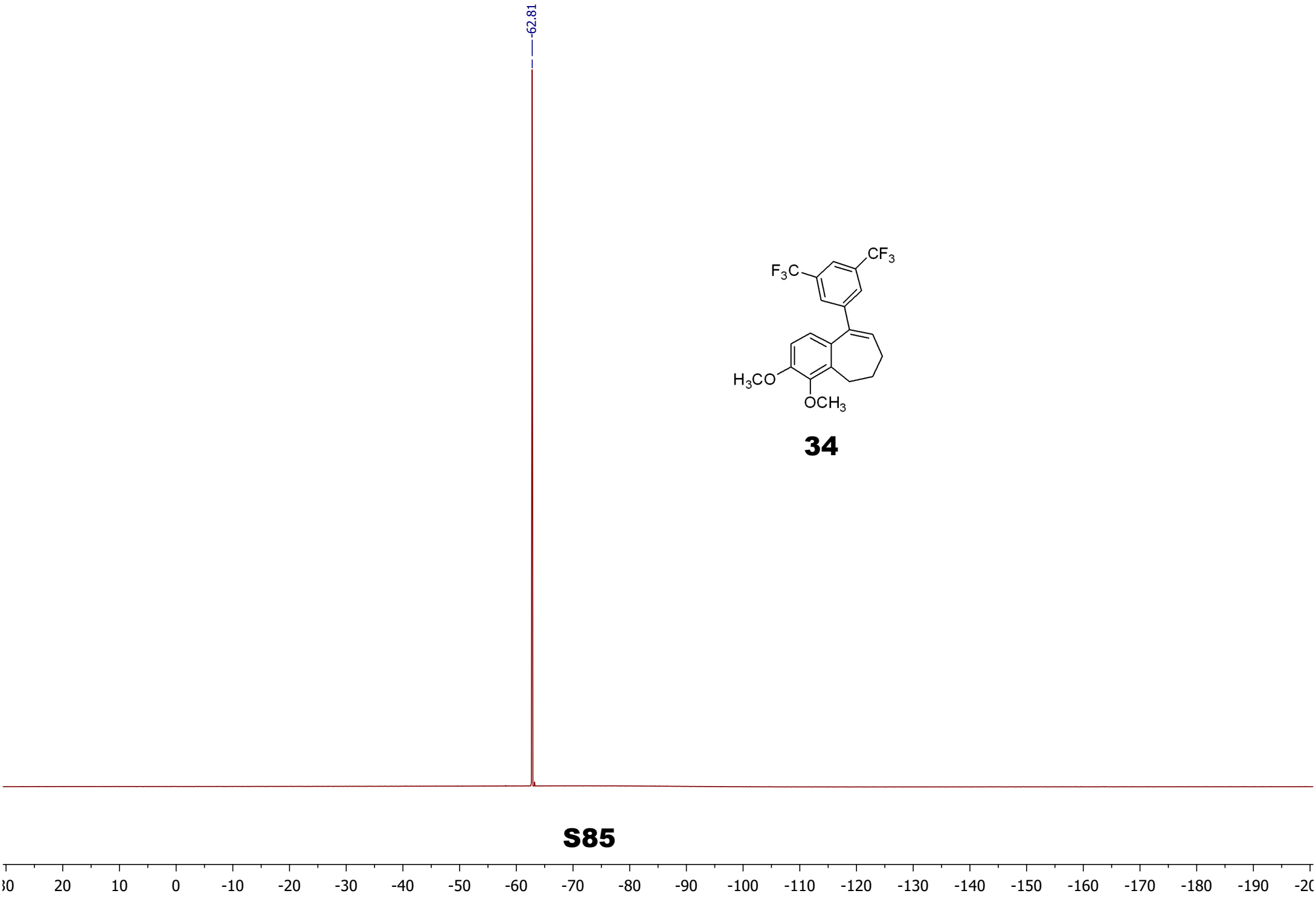
HRMS for compound 33

1_125_KGP407_1_125_KGP407 #500 RT: 3.99 AV: 1 N E8
T: FTMS + p ESI Full ms [200.00-700.00]




7.75
7.71
7.26
6.78
6.76
6.64
6.62
6.51
6.49
6.48
3.90
3.88
2.77
2.76
2.74
2.21
2.19
2.18
2.17
2.15
2.05
2.03
2.02
2.01
F3C
CF3
H3CO
OCH3
34
S83
0.91
1.90
1.00
0.96
1.01
3.02
2.98
2.05
2.09
2.07
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


152.20
146.57
144.74
144.72
140.82
136.14
132.40
131.70
131.44
131.09
128.01
127.98
127.95
127.92
124.83
124.67
122.50
120.72
120.69
77.41 cdcl3
76.91 cdcl3
61.37
55.90
55.82
34.33
25.99
24.27
CF3
F3C
H3CO
OCH3
34


-62.81
F3C
CF3
H3CO
OCH3
34
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -20

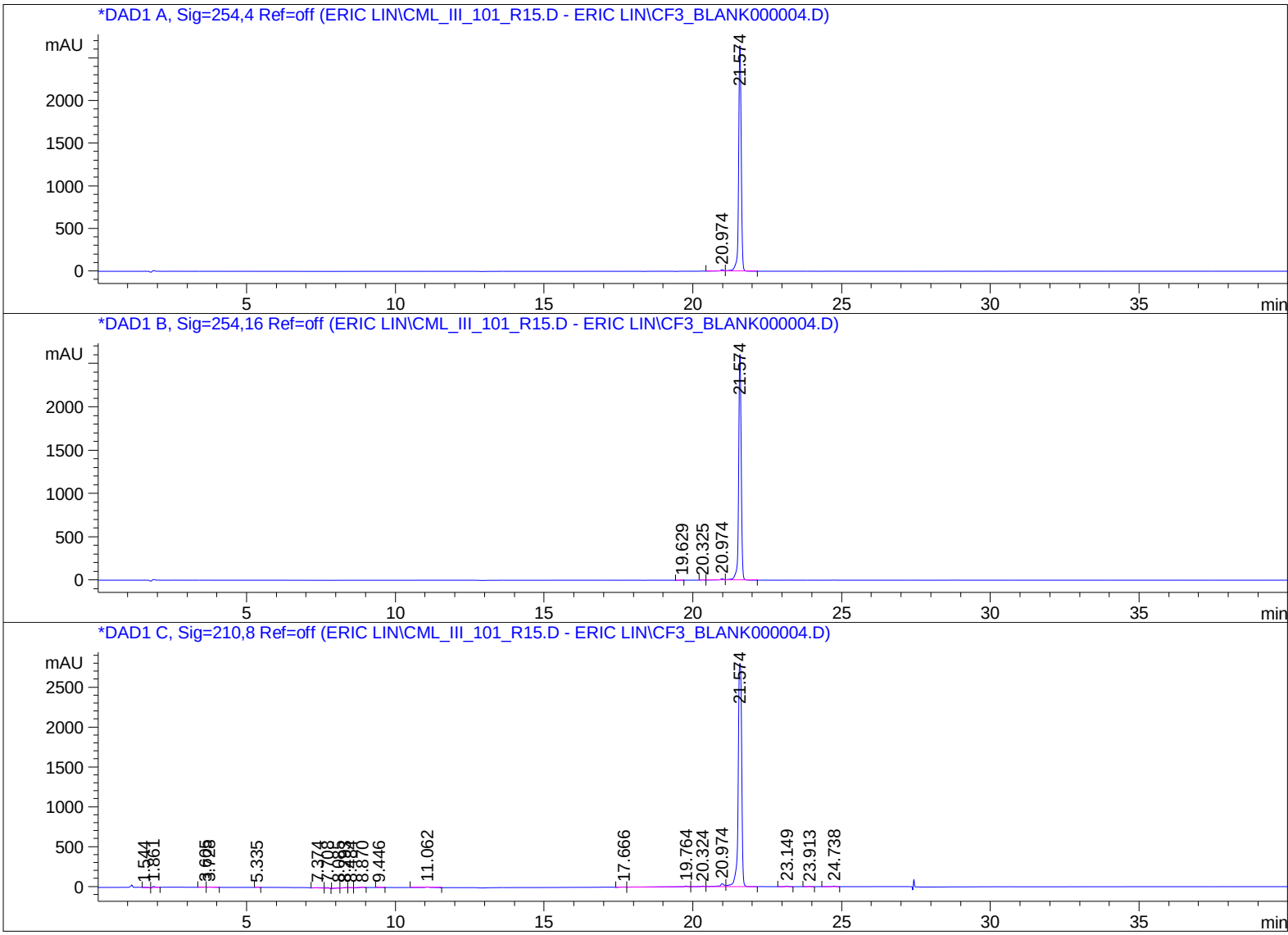
Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_101_R15.D
Sample Name: CML_III_101_r1

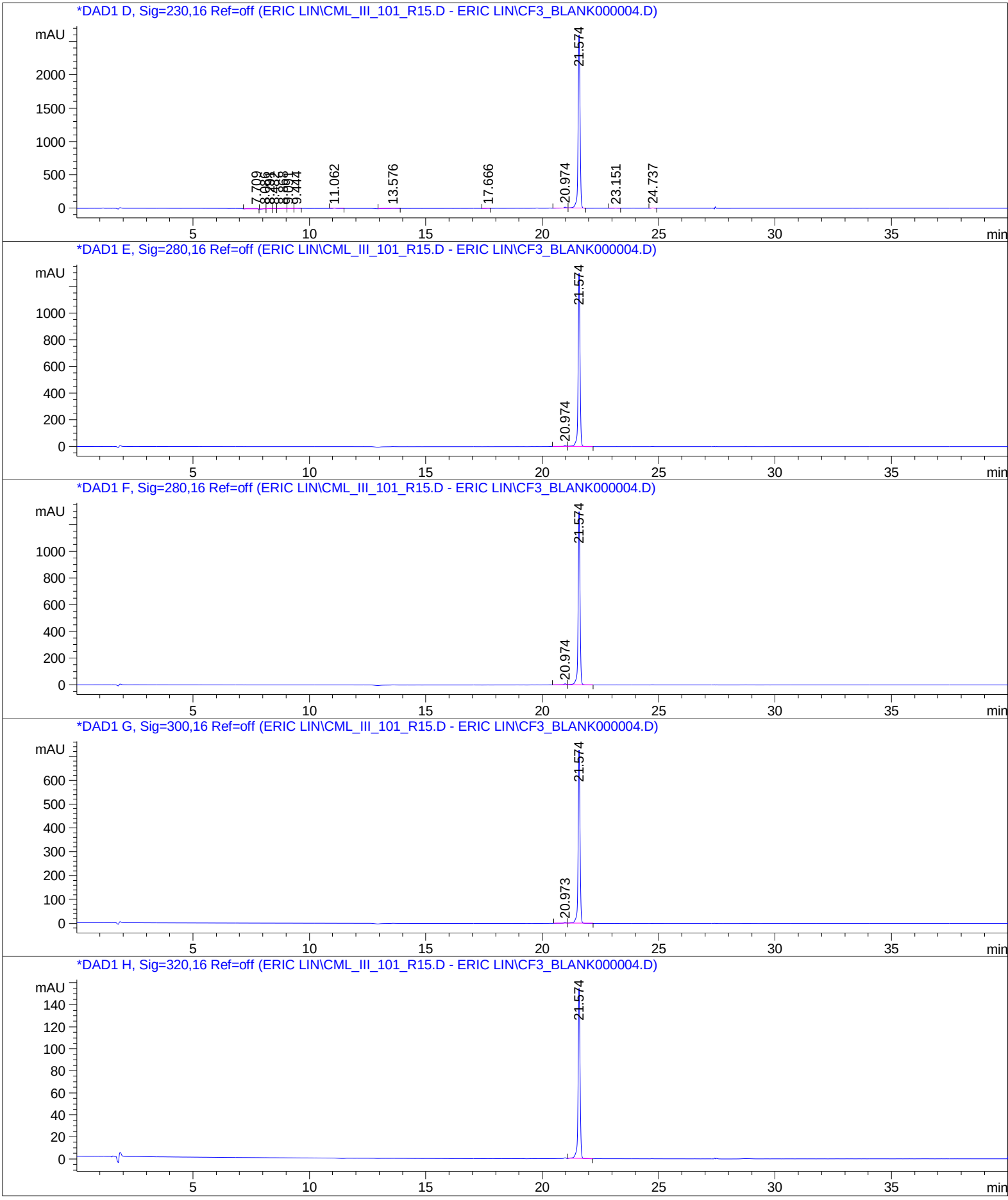
HPLC for compound 34

```
==========================================================================
Acq. Operator   : Eric Lin
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 3/11/2014 1:58:36 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 3/11/2014 1:55:06 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\CML_III_101_R15.D\DA.M (MASTERMETHOD.M)
Last changed    : 3/11/2014 2:57:57 PM by Eric Lin
Sample Info     : wash

                  Method:

                  0-25 min. (50:50 to 100:0) ACN:Water
                  25-30 min. (100:0) ACN:Water
                  30-35 min. (100:0 to 50:50) ACN:Water
                  35-40 min. (50:50) ACN:Water
```




*DAD1 D, Sig=230,16 Ref=off (ERIC LIN\CML_III_101_R15.D - ERIC LIN\CF3_BLANK000004.D)
mAU
7.709
11.062
13.576
17.666
20.974
21.574
23.151
24.737
min
*DAD1 E, Sig=280,16 Ref=off (ERIC LIN\CML_III_101_R15.D - ERIC LIN\CF3_BLANK000004.D)
mAU
20.974
21.574
min
*DAD1 F, Sig=280,16 Ref=off (ERIC LIN\CML_III_101_R15.D - ERIC LIN\CF3_BLANK000004.D)
mAU
20.974
21.574
min
*DAD1 G, Sig=300,16 Ref=off (ERIC LIN\CML_III_101_R15.D - ERIC LIN\CF3_BLANK000004.D)
mAU
20.973
21.574
min
*DAD1 H, Sig=320,16 Ref=off (ERIC LIN\CML_III_101_R15.D - ERIC LIN\CF3_BLANK000004.D)
mAU
21.574
min

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_101_R15.D
Sample Name: CML_III_101_r1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.974	BV	0.1266	162.18532	17.74556	0.9957
2	21.574	VB	0.0950	1.61269e4	2649.16064	99.0043
Totals :				1.62891e4	2666.90621	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.629	BV	0.1434	15.67968	1.50705	0.0984
2	20.325	BB	0.0837	7.84197	1.48258	0.0492
3	20.974	BV	0.1269	156.03926	17.03149	0.9797
4	21.574	VB	0.0944	1.57484e4	2610.57349	98.8727
Totals :				1.59279e4	2630.59461	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.544	VB	0.1616	91.00436	7.09602	0.3682
2	1.861	BB	0.1195	129.13742	16.46471	0.5225
3	3.605	BV	0.1238	19.76291	2.22098	0.0800
4	3.728	VB	0.1876	36.14043	2.74495	0.1462
5	5.335	BV	0.0699	7.31299	1.58168	0.0296
6	7.374	BV	0.3155	131.54974	5.77983	0.5323
7	7.708	VB	0.1477	82.41358	8.61145	0.3335
8	8.085	BV	0.1492	48.02391	5.04047	0.1943
9	8.293	VV	0.1788	82.41489	6.46100	0.3335

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_101_R15.D
Sample Name: CML_III_101_r1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10	8.484	VB	0.1196	28.29939	3.60454	0.1145
11	8.870	BB	0.1467	40.12056	4.08138	0.1623
12	9.446	BB	0.1206	8.52363	1.05068	0.0345
13	11.062	BB	0.1804	111.10935	8.28815	0.4496
14	17.666	BV	0.1830	76.52775	5.76194	0.3097
15	19.764	VB	0.3768	263.92871	8.67955	1.0680
16	20.324	BV	0.1196	57.96089	6.93019	0.2345
17	20.974	VV	0.1513	427.89676	38.00670	1.7314
18	21.574	VB	0.1296	2.29555e4	2800.36768	92.8875
19	23.149	BB	0.0962	46.80672	7.35477	0.1894
20	23.913	BB	0.1068	15.62614	2.20080	0.0632
21	24.738	BB	0.1166	53.17654	7.00522	0.2152
Totals :				2.47132e4	2949.33269	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.709	BB	0.3239	249.13312	10.09571	1.5249
2	8.086	BV	0.1551	57.38152	5.81611	0.3512
3	8.291	VV	0.1863	98.30637	7.43379	0.6017
4	8.482	VB	0.1140	29.62998	4.01957	0.1814
5	8.868	BV	0.1762	75.49585	6.10649	0.4621
6	9.091	VV	0.1764	31.43843	2.34578	0.1924
7	9.444	VB	0.1488	19.71043	1.84204	0.1206
8	11.062	BB	0.1049	24.78027	3.57223	0.1517
9	13.576	BB	0.5029	106.49190	2.63814	0.6518
10	17.666	BB	0.1714	20.12987	1.66064	0.1232
11	20.974	BV	0.1380	173.49957	17.14832	1.0620
12	21.574	VB	0.0909	1.54119e4	2608.66772	94.3329
13	23.151	BB	0.1007	10.27145	1.52356	0.0629
14	24.737	BB	0.1105	29.60491	4.18738	0.1812
Totals :				1.63378e4	2677.05750	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.974	BV	0.1231	72.74569	8.22962	0.9803
2	21.574	VB	0.0858	7347.78955	1302.42627	99.0197

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_101_R15.D
Sample Name: CML_III_101_r1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				7420.53524	1310.65589	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.974	BV	0.1231	72.74569	8.22962	0.9803
2	21.574	VB	0.0858	7347.78955	1302.42627	99.0197
Totals :				7420.53524	1310.65589	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.973	BV	0.1175	35.49311	4.24565	0.8536
2	21.574	VB	0.0860	4122.53955	729.12970	99.1464
Totals :				4158.03266	733.37535	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.574	BB	0.0878	901.81482	155.12813	100.0000
Totals :				901.81482	155.12813	

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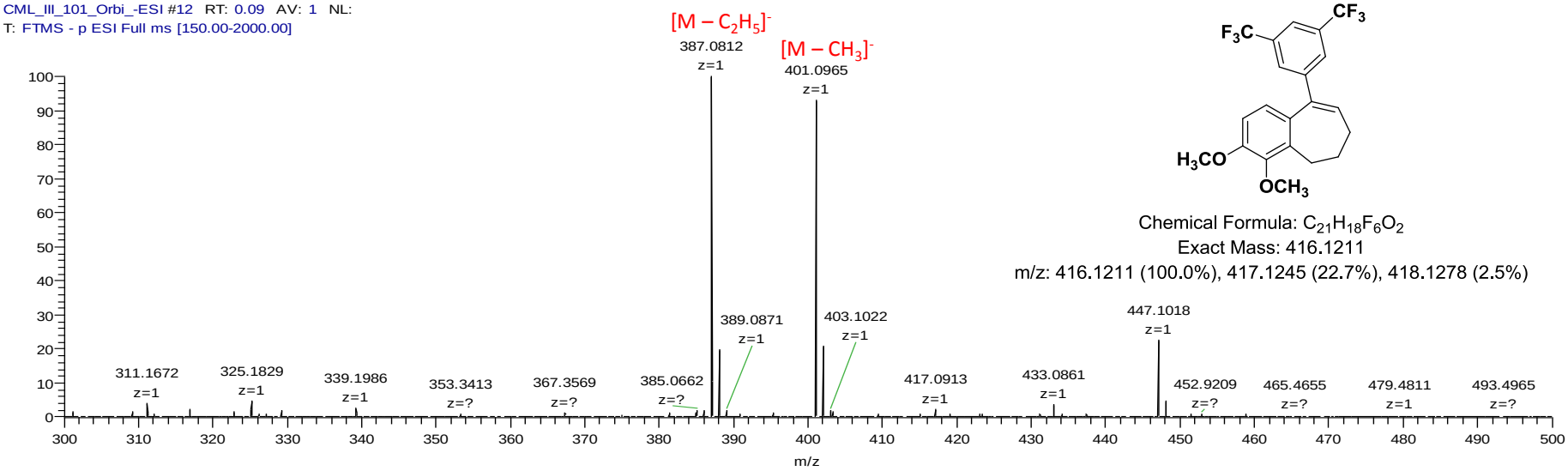
*** End of Report ***

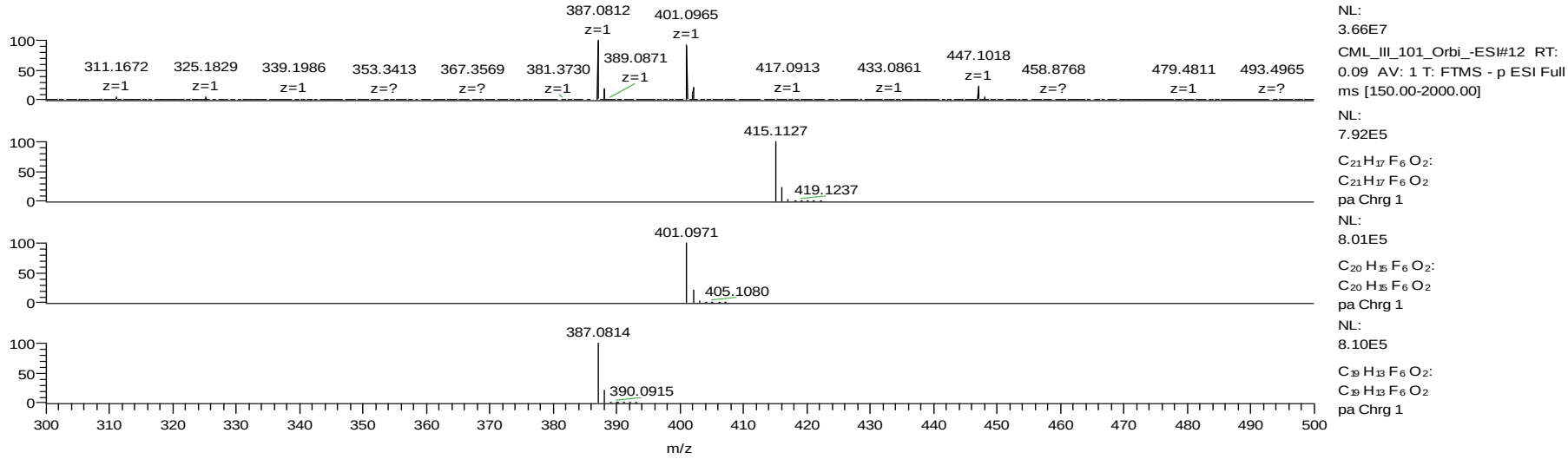
HRMS for compound 34

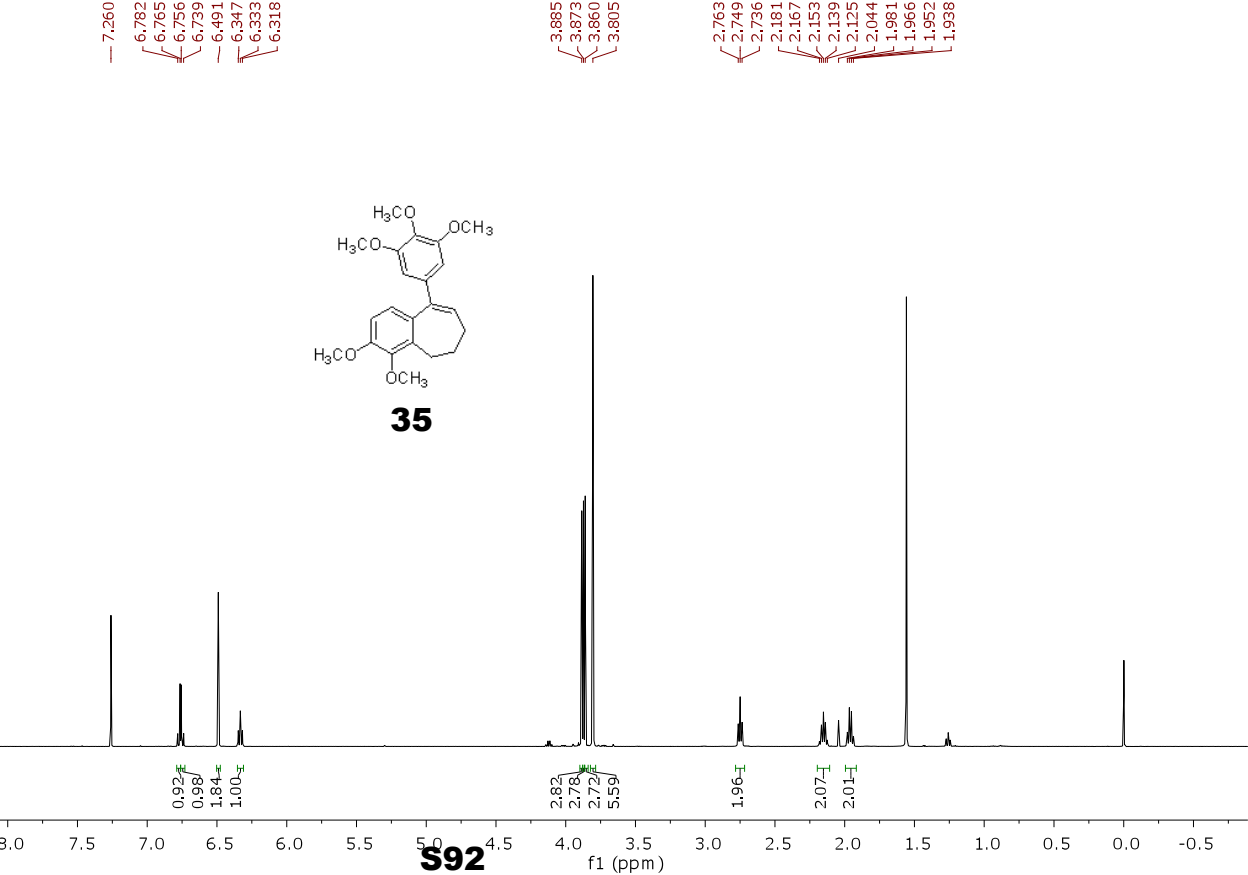
C:\Xcalibur\...\CML_III_101_Orbi_-ESI 3/17/2014 3:21:06 PM CML_III_101

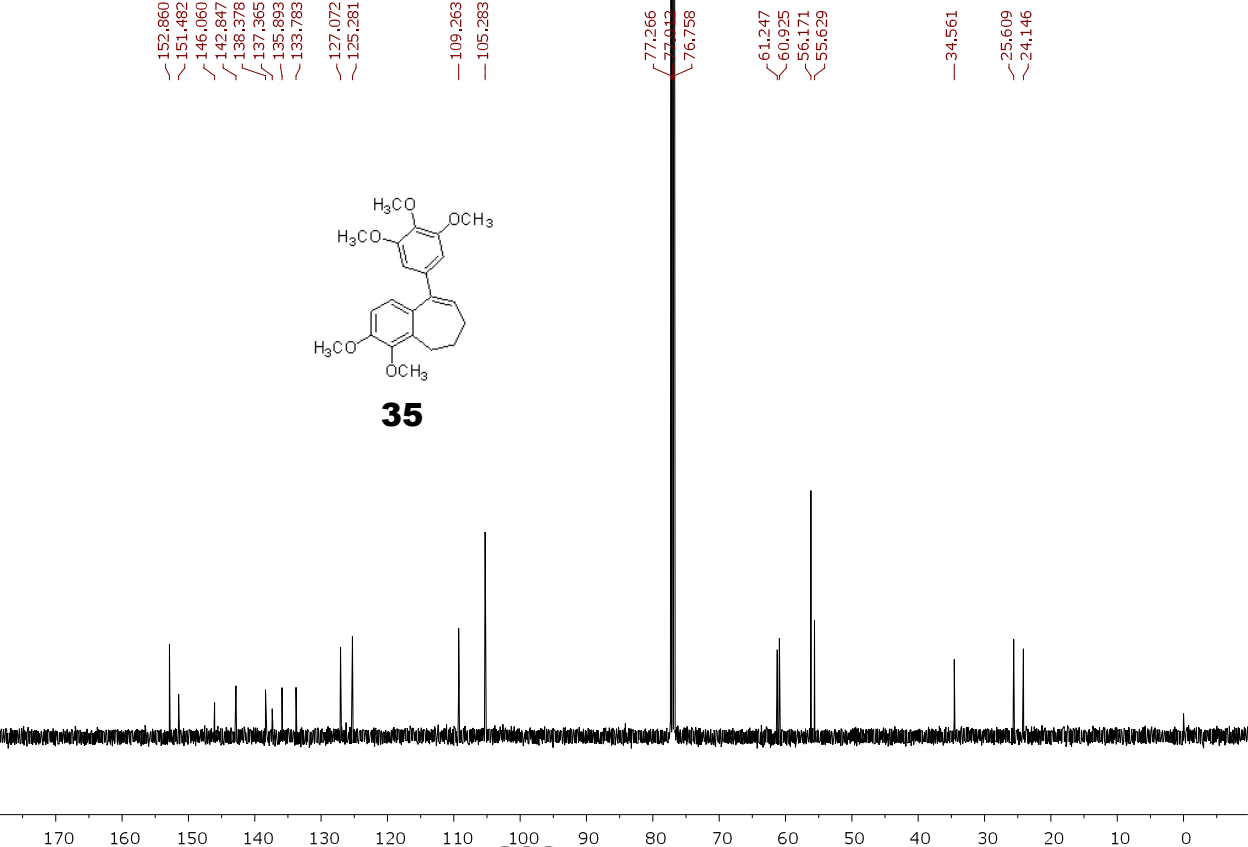
CML_III_101_Orbi_-ESI #12 RT: 0.09 AV: 1 NL:
T: FTMS - p ESI Full ms [150.00-2000.00]



Chemical Formula: $C_{21}H_{18}F_6O_2$
Exact Mass: 416.1211
m/z: 416.1211 (100.0%), 417.1245 (22.7%), 418.1278 (2.5%)



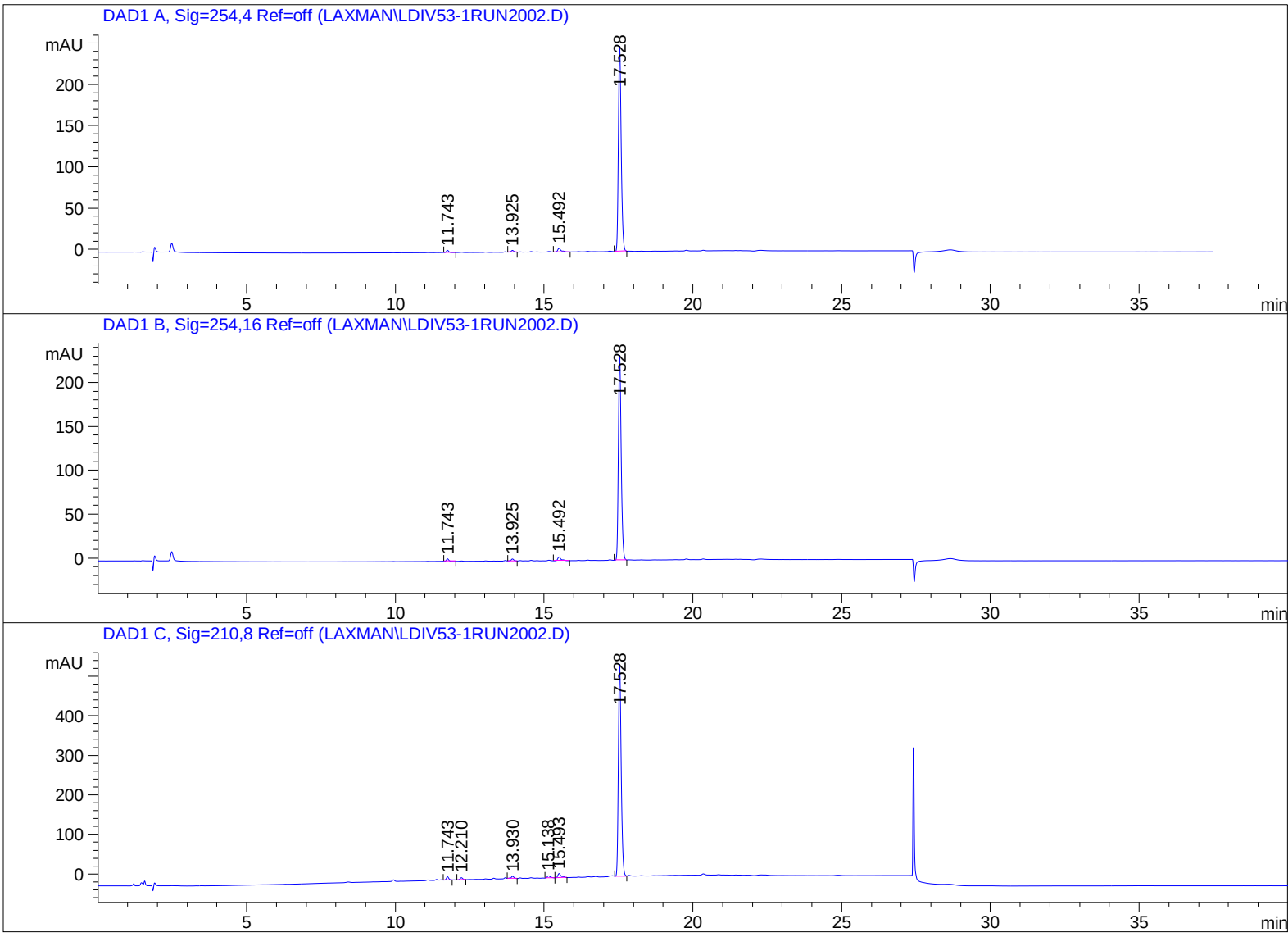

7.260
6.782
6.765
6.756
6.739
6.491
6.347
6.333
6.318
3.885
3.873
3.860
3.805
2.763
2.749
2.736
2.181
2.167
2.153
2.139
2.125
2.044
1.981
1.966
1.952
1.938
H3CO
OCH3
H3CO
H3CO
OCH3
35
0.92
0.98
1.84
1.00
2.82
2.78
2.72
5.59
1.96
2.07
2.01
f1 (ppm)


152.860
151.482
146.060
142.847
138.378
137.365
135.893
133.783
127.072
125.281
109.263
105.283
77.266
77.012
76.758
61.247
60.925
56.171
55.629
34.561
25.609
24.146
H3CO
OCH3
H3CO
H3CO
OCH3
35
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

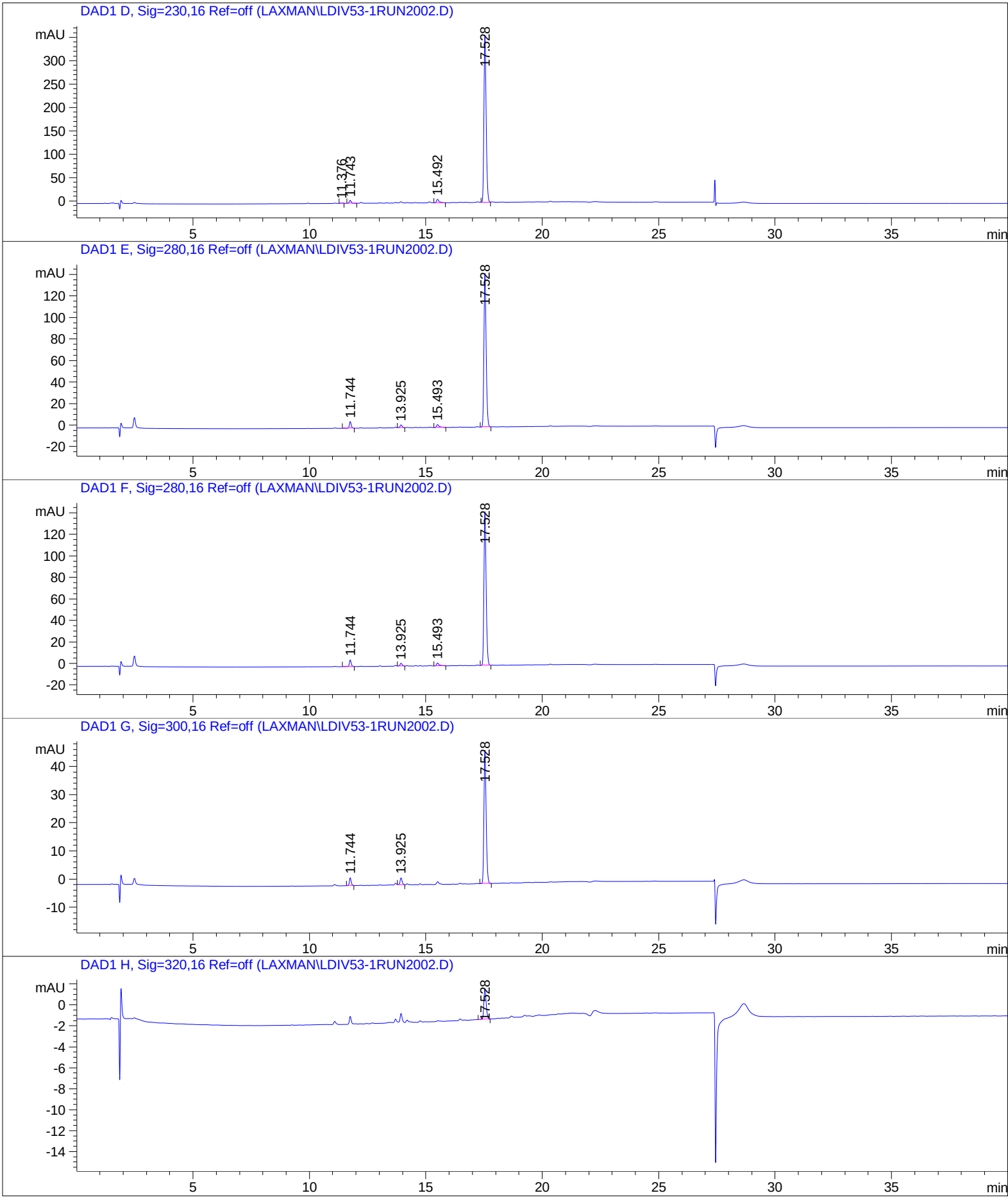
Data File C:\CHEM32\1\DATA\LAXMAN\LDIV53-1RUN2002.D
Sample Name: LD-IV-53-1A-run2

HPLC for compound 35

```
==========================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/28/2014 4:36:52 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 1/28/2014 4:31:37 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LDIV53-1RUN2002.D\DA.M (MASTERMETHOD.M)
Last changed    : 1/28/2014 5:32:55 PM by Laxman
Sample Info     : run2
```



Data File C:\CHEM32\1\DATA\LAXMAN\LDIV53-1RUN2002.D
Sample Name: LD-IV-53-1A-run2



Data File C:\CHEM32\1\DATA\LAXMAN\LDIV53-1RUN2002.D
Sample Name: LD-IV-53-1A-run2

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.743	BB	0.0826	14.26201	2.65778	0.8361
2	13.925	BB	0.0929	13.40179	2.14591	0.7857
3	15.492	BB	0.1162	36.18954	4.57916	2.1215
4	17.528	BB	0.1009	1641.95728	249.16393	96.2567
Totals :				1705.81062	258.54678	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.743	BB	0.0825	15.31448	2.85651	0.9551
2	13.925	BB	0.0930	13.93967	2.22854	0.8693
3	15.492	BB	0.1162	34.20804	4.32975	2.1333
4	17.528	BB	0.1009	1540.03906	233.65747	96.0423
Totals :				1603.50126	243.07228	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.743	VB	0.0846	46.10949	8.32722	1.2122
2	12.210	BB	0.0942	31.44279	5.22819	0.8266
3	13.930	VB	0.1058	44.68492	6.07767	1.1747
4	15.138	BB	0.1074	40.66746	5.55600	1.0691
5	15.493	BB	0.1166	84.69407	10.67318	2.2265
6	17.528	VV	0.1012	3556.32837	537.31000	93.4910
Totals :				3803.92710	573.17226	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.376	BB	0.0778	5.07398	1.02429	0.2062
2	11.743	BB	0.0837	34.19037	6.25711	1.3895
3	15.492	VB	0.1148	57.49661	7.38936	2.3366
4	17.528	VV	0.1010	2363.93970	358.03088	96.0677
Totals :				2460.70065	372.70165	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.744	BB	0.0836	33.69853	6.18095	3.3237
2	13.925	BB	0.0929	16.22744	2.59767	1.6005
3	15.493	BB	0.1161	21.33865	2.70186	2.1046
4	17.528	BB	0.1010	942.63556	142.84668	92.9712
Totals :				1013.90018	154.32717	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.744	BB	0.0836	33.69853	6.18095	3.3237
2	13.925	BB	0.0929	16.22744	2.59767	1.6005
3	15.493	BB	0.1161	21.33865	2.70186	2.1046
4	17.528	BB	0.1010	942.63556	142.84668	92.9712
Totals :				1013.90018	154.32717	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.744	BB	0.0831	15.00270	2.77378	4.3629
2	13.925	BB	0.0900	14.52386	2.41986	4.2237
3	17.528	BB	0.1013	314.33951	47.44872	91.4134
Totals :				343.86607	52.64236	

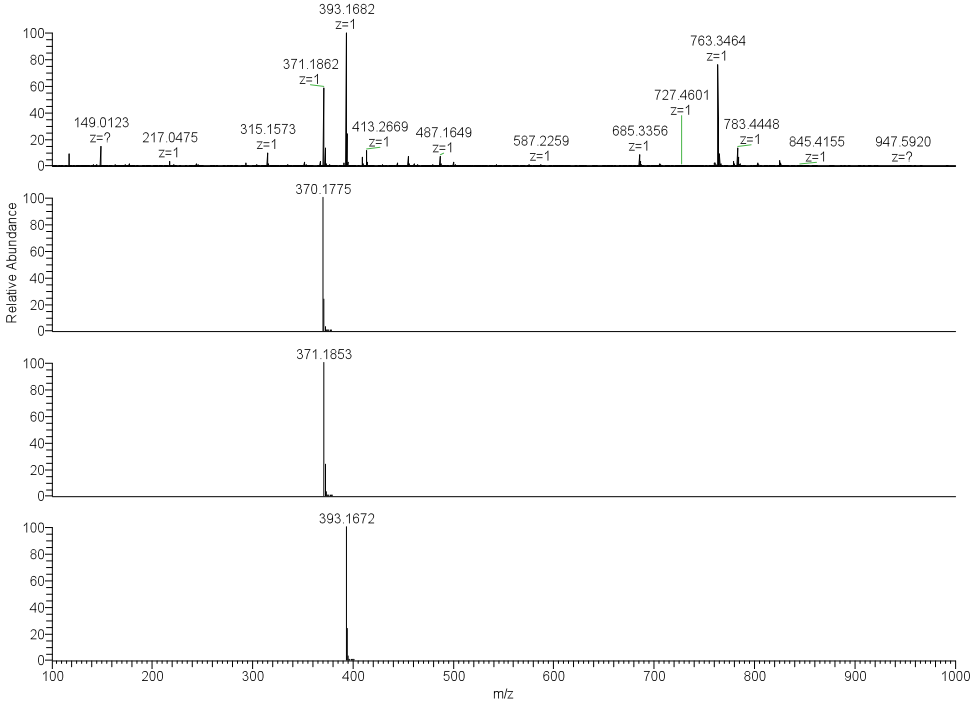
Data File C:\CHEM32\1\DATA\LAXMAN\LDIV53-1RUN2002.D
Sample Name: LD-IV-53-1A-run2

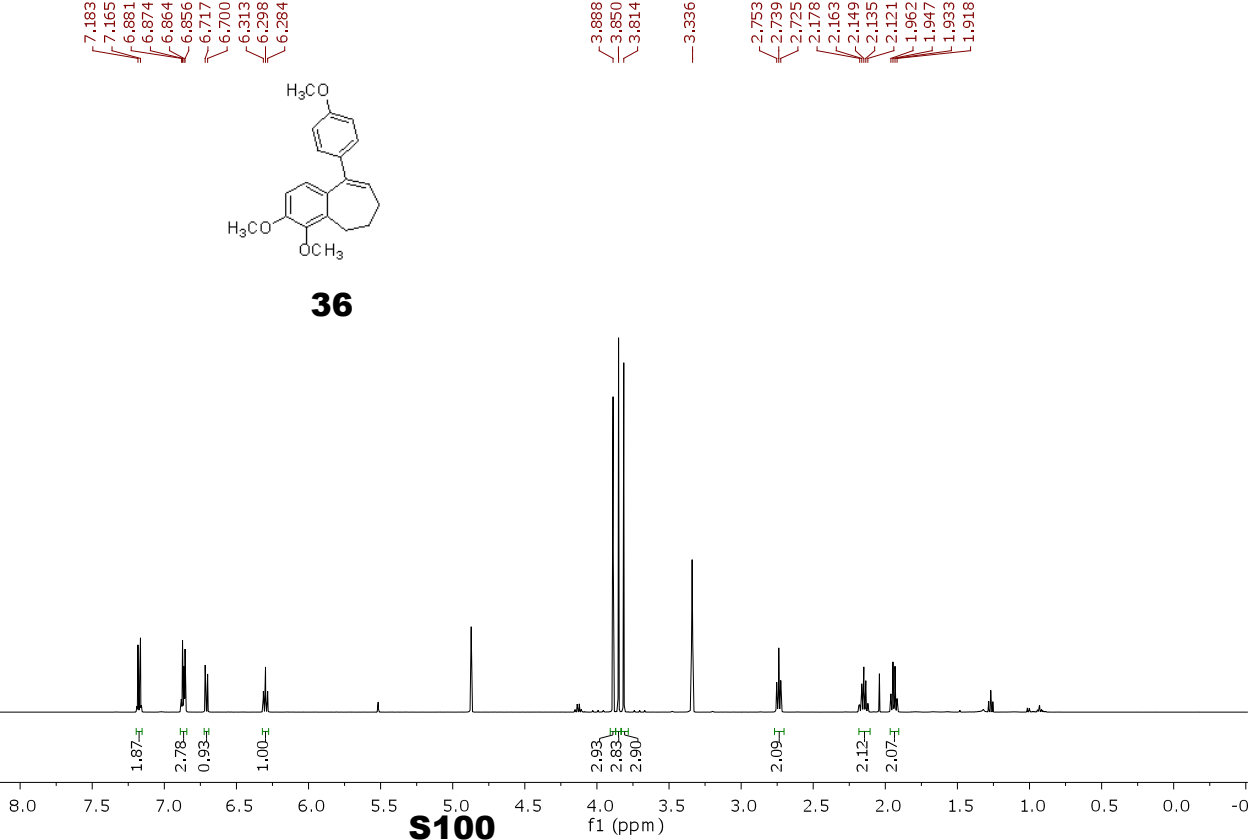
Signal 8: DAD1 H, Sig=320,16 Ref=off

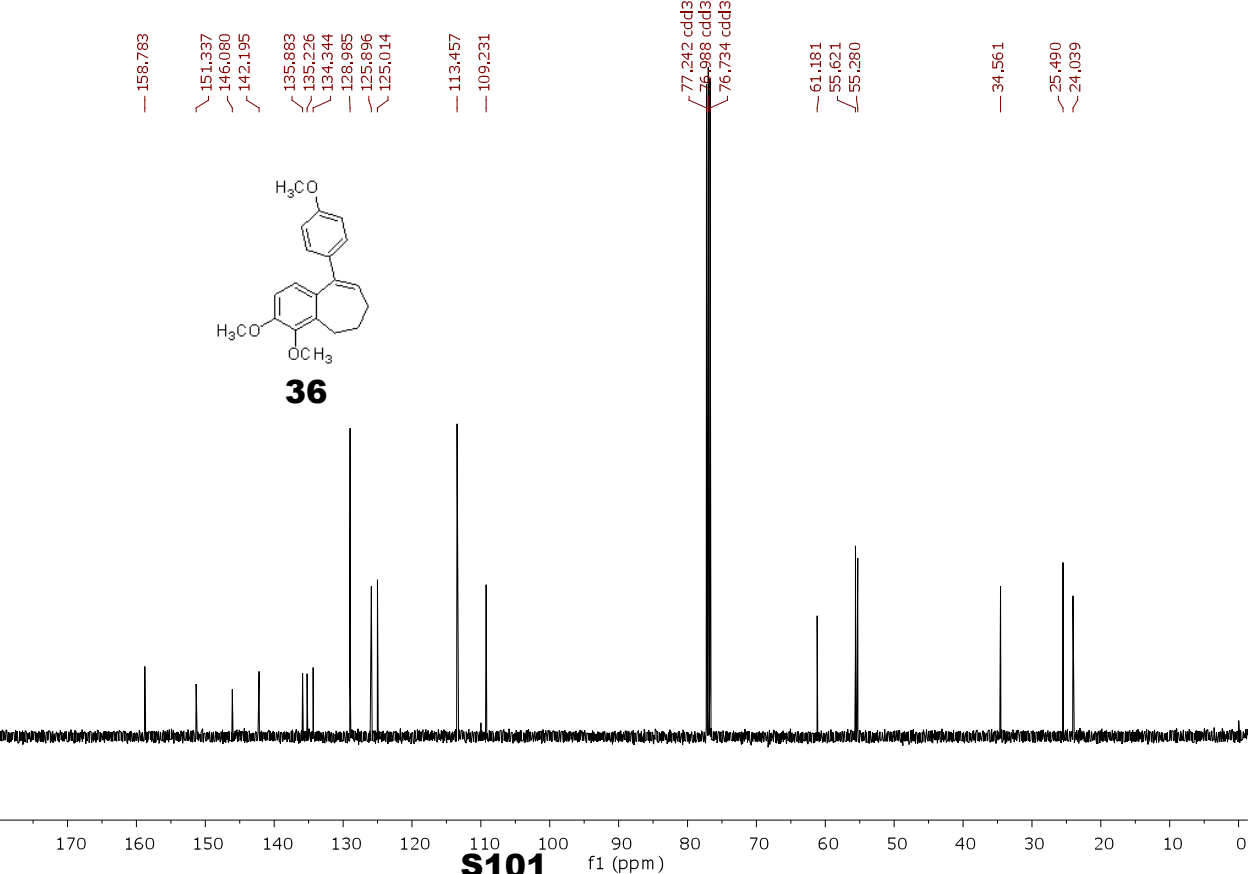
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.528	BB	0.1072	20.50176	2.94557	100.0000
Totals :				20.50176	2.94557	

*** End of Report ***

HRMS for compound 35



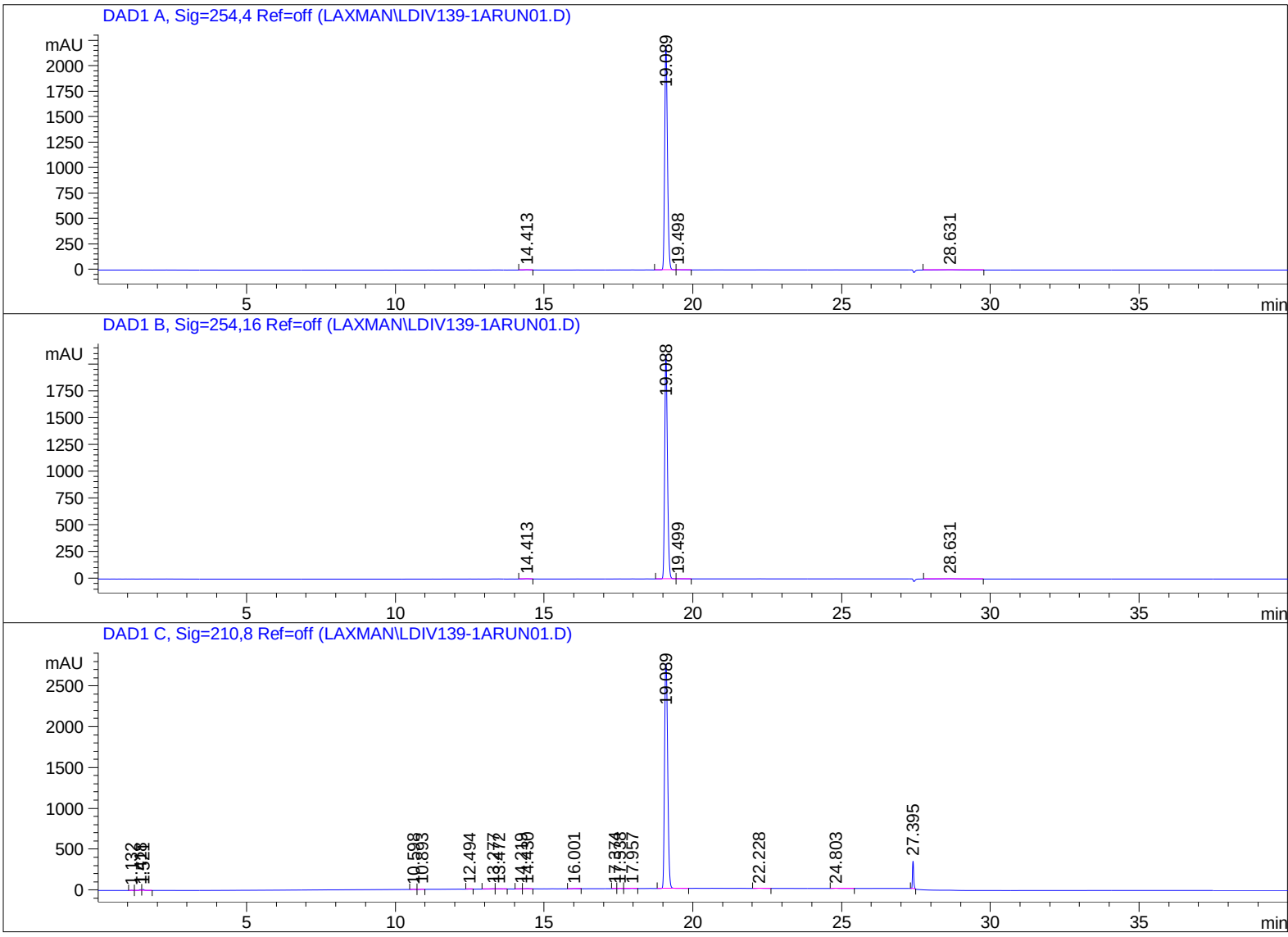

7.183
7.165
6.881
6.874
6.864
6.856
6.717
6.700
6.313
6.298
6.284
3.888
3.850
3.814
3.336
2.753
2.739
2.725
2.178
2.163
2.149
2.135
2.121
1.962
1.947
1.933
1.918
H3CO
H3CO
OCH3
36
1.87
2.78
0.93
1.00
2.93
2.83
2.90
2.09
2.12
2.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


158.783
151.337
146.080
142.195
135.883
135.226
134.344
128.985
125.896
125.014
113.457
109.231
77.242 cdd3
76.988 cdd3
76.734 cdd3
61.181
55.621
55.280
34.561
25.490
24.039
H3CO
H3CO
OCH3
36
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Data File C:\CHEM32\1\DATA\LAXMAN\LDIV139-1ARUN01.D
Sample Name: LD-IV-139-1A-1

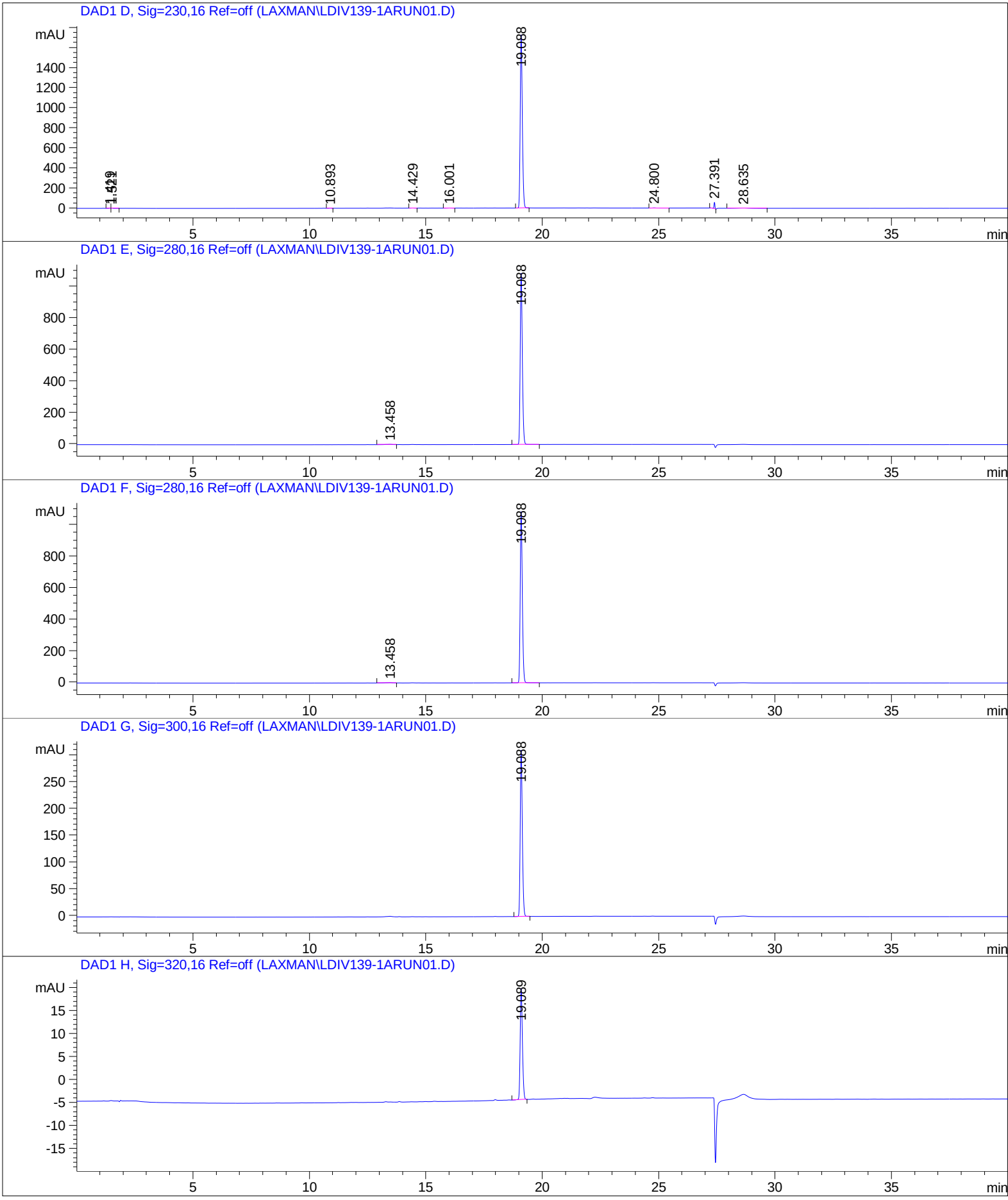
HPLC for compound 36

```
==========================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/30/2014 10:16:04 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 1/30/2014 9:43:27 AM by Laxman
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 1/30/2014 11:06:05 AM by Laxman
Sample Info     : Run1
```



S102

Data File C:\CHEM32\1\DATA\LAXMAN\LDIV139-1ARUN01.D
Sample Name: LD-IV-139-1A-1



Data File C:\CHEM32\1\DATA\LAXMAN\LDIV139-1ARUN01.D
Sample Name: LD-IV-139-1A-1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.413	BB	0.1604	21.42000	2.07520	0.1499
2	19.089	BV	0.0989	1.41765e4	2207.01196	99.1916
3	19.498	VB	0.1340	18.92470	1.93655	0.1324
4	28.631	BB	0.4860	75.18730	2.19272	0.5261
Totals :				1.42921e4	2213.21642	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.413	BB	0.1601	19.95540	1.93899	0.1477
2	19.088	BV	0.0986	1.34034e4	2097.21265	99.1985
3	19.499	VB	0.1348	17.67035	1.79536	0.1308
4	28.631	BB	0.4580	70.67089	2.11384	0.5230
Totals :				1.35117e4	2103.06084	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.132	BB	0.0614	14.57730	3.58883	0.0679
2	1.418	BV	0.0759	48.01664	10.02032	0.2238
3	1.521	VB	0.0610	54.63629	13.01121	0.2547
4	10.598	BB	0.0938	8.46140	1.41429	0.0394
5	10.893	BB	0.0874	14.87960	2.57484	0.0694
6	12.494	BB	0.1000	8.76446	1.38110	0.0409
7	13.277	BV	0.1336	24.42828	2.55433	0.1139
8	13.472	VV	0.2102	32.02443	2.02785	0.1493
9	14.219	BV	0.0878	7.31985	1.29744	0.0341
10	14.430	VB	0.1658	35.88203	3.38270	0.1673

Data File C:\CHEM32\1\DATA\LAXMAN\LDIV139-1ARUN01.D
Sample Name: LD-IV-139-1A-1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
11	16.001	BB	0.1367	17.37272	1.73647	0.0810
12	17.374	BV	0.0930	7.81489	1.32111	0.0364
13	17.538	VB	0.0939	12.18892	1.97756	0.0568
14	17.957	BB	0.1325	13.73361	1.53351	0.0640
15	19.089	BB	0.1154	2.01518e4	2751.83130	93.9339
16	22.228	BB	0.1848	28.84549	2.29175	0.1345
17	24.803	BB	0.1325	21.64288	2.41514	0.1009
18	27.395	BB	0.0445	950.77936	341.76141	4.4319
Totals :				2.14531e4	3146.12117	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.419	BV	0.0785	8.55047	1.64809	0.0759
2	1.521	VB	0.0891	11.86712	1.79461	0.1053
3	10.893	BB	0.0867	7.44412	1.30266	0.0661
4	14.429	VB	0.1630	15.97235	1.53950	0.1418
5	16.001	BB	0.1503	12.82694	1.16594	0.1138
6	19.088	BB	0.0976	1.09185e4	1729.82043	96.9030
7	24.800	BB	0.1450	15.16185	1.53794	0.1346
8	27.391	BB	0.0488	207.03316	65.66882	1.8374
9	28.635	BB	0.4285	70.09544	2.34659	0.6221
Totals :				1.12674e4	1806.82458	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.458	BB	0.2502	33.25743	1.77782	0.4806
2	19.088	BB	0.0978	6887.35742	1088.69373	99.5194
Totals :				6920.61486	1090.47154	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Data File C:\CHEM32\1\DATA\LAXMAN\LDIV139-1ARUN01.D
Sample Name: LD-IV-139-1A-1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.458	BB	0.2502	33.25743	1.77782	0.4806
2	19.088	BB	0.0978	6887.35742	1088.69373	99.5194
Totals :				6920.61486	1090.47154	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.088	BB	0.0978	1975.91748	312.50003	100.0000
Totals :				1975.91748	312.50003	

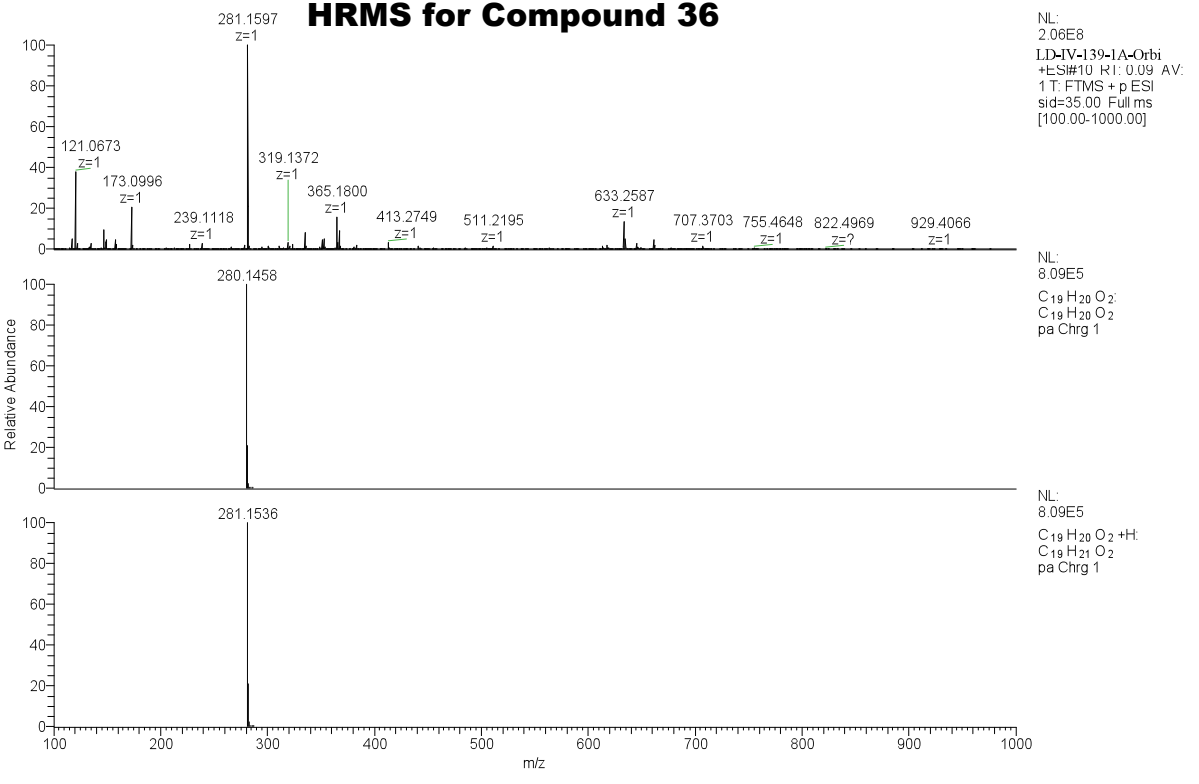
Signal 8: DAD1 H, Sig=320,16 Ref=off

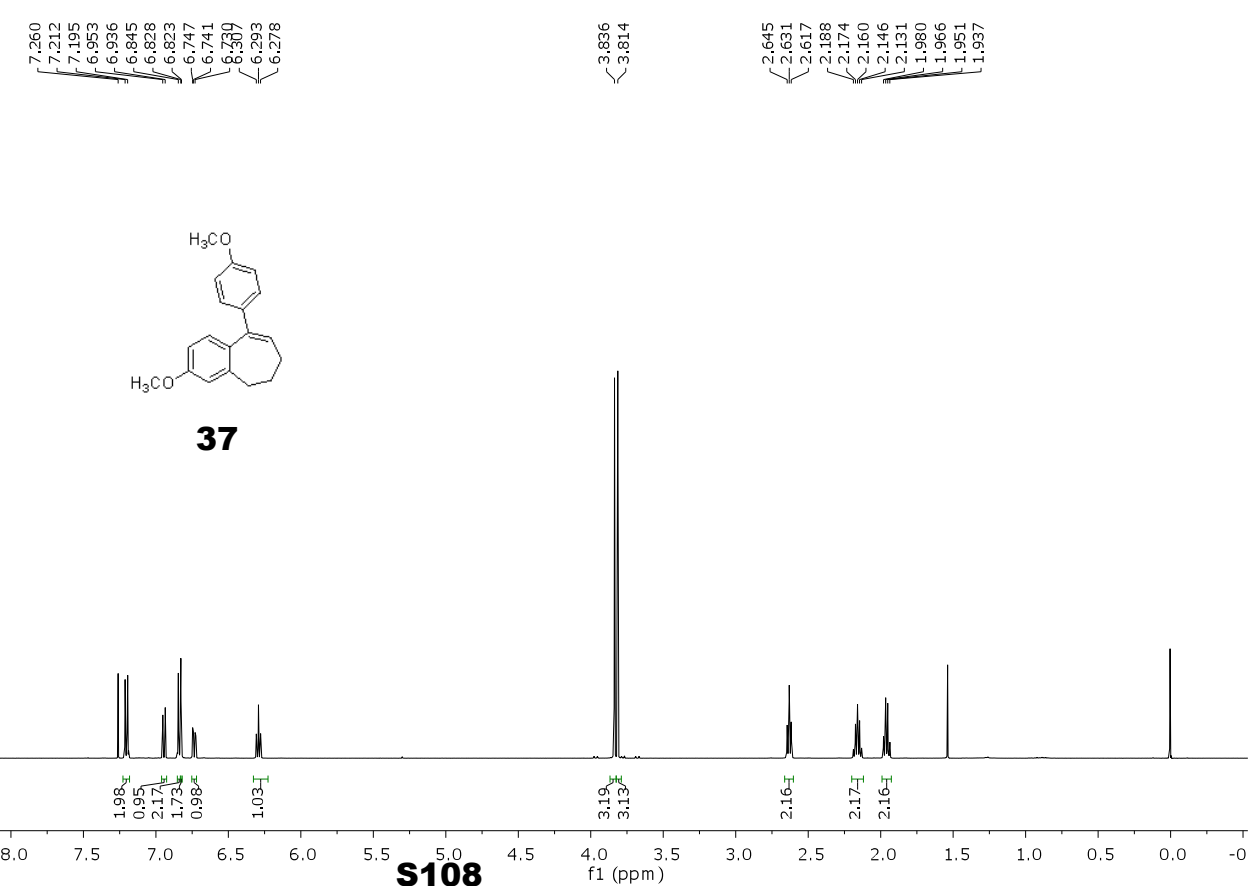
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.089	BB	0.1021	162.21338	24.24150	100.0000
Totals :				162.21338	24.24150	

==========

*** End of Report ***

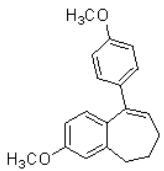
HRMS for Compound 36



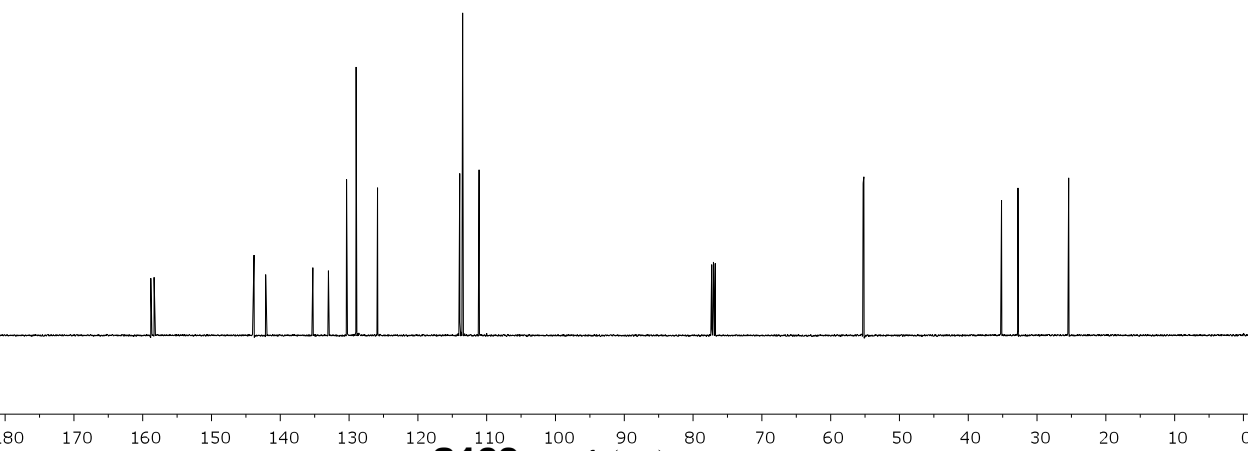

7.260
7.212
7.195
6.953
6.936
6.845
6.828
6.823
6.747
6.741
6.307
6.293
6.278
3.836
3.814
2.645
2.631
2.617
2.188
2.174
2.160
2.146
2.131
1.980
1.966
1.951
1.937
H3CO
H3CO
1.98
0.95
2.17
1.73
0.98
1.03
3.19
3.13
2.16
2.17
2.16
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

37

158.822
158.344
143.823
142.128
135.274
133.001
130.366
125.870
113.912
113.503
113.498
111.100
55.285
55.168
35.160
32.770
25.400



37

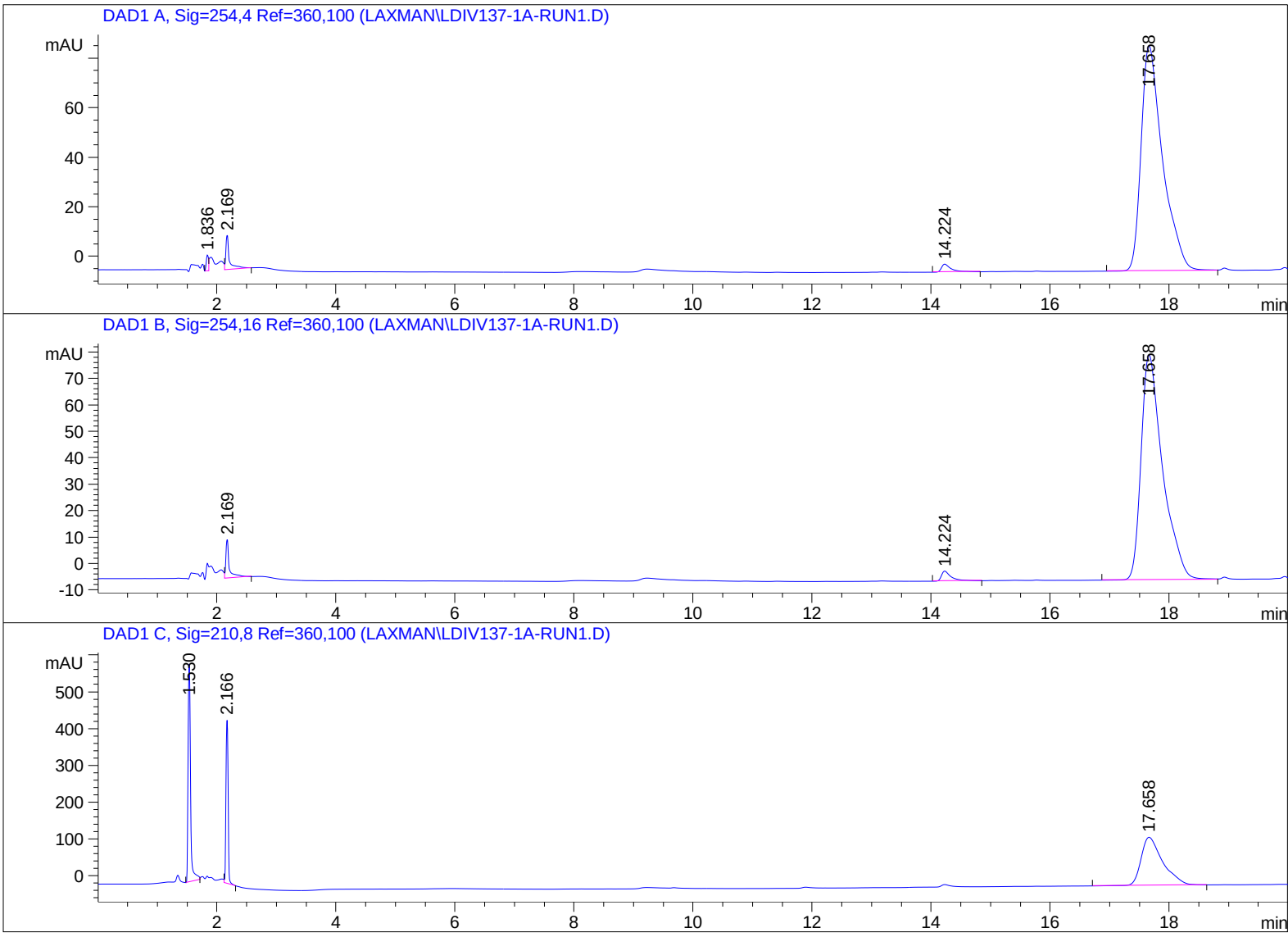


Data File C:\CHEM32\1\DATA\LAXMAN\LDIV137-1A-RUN1.D
Sample Name: LD-IV-137-1A-Run1

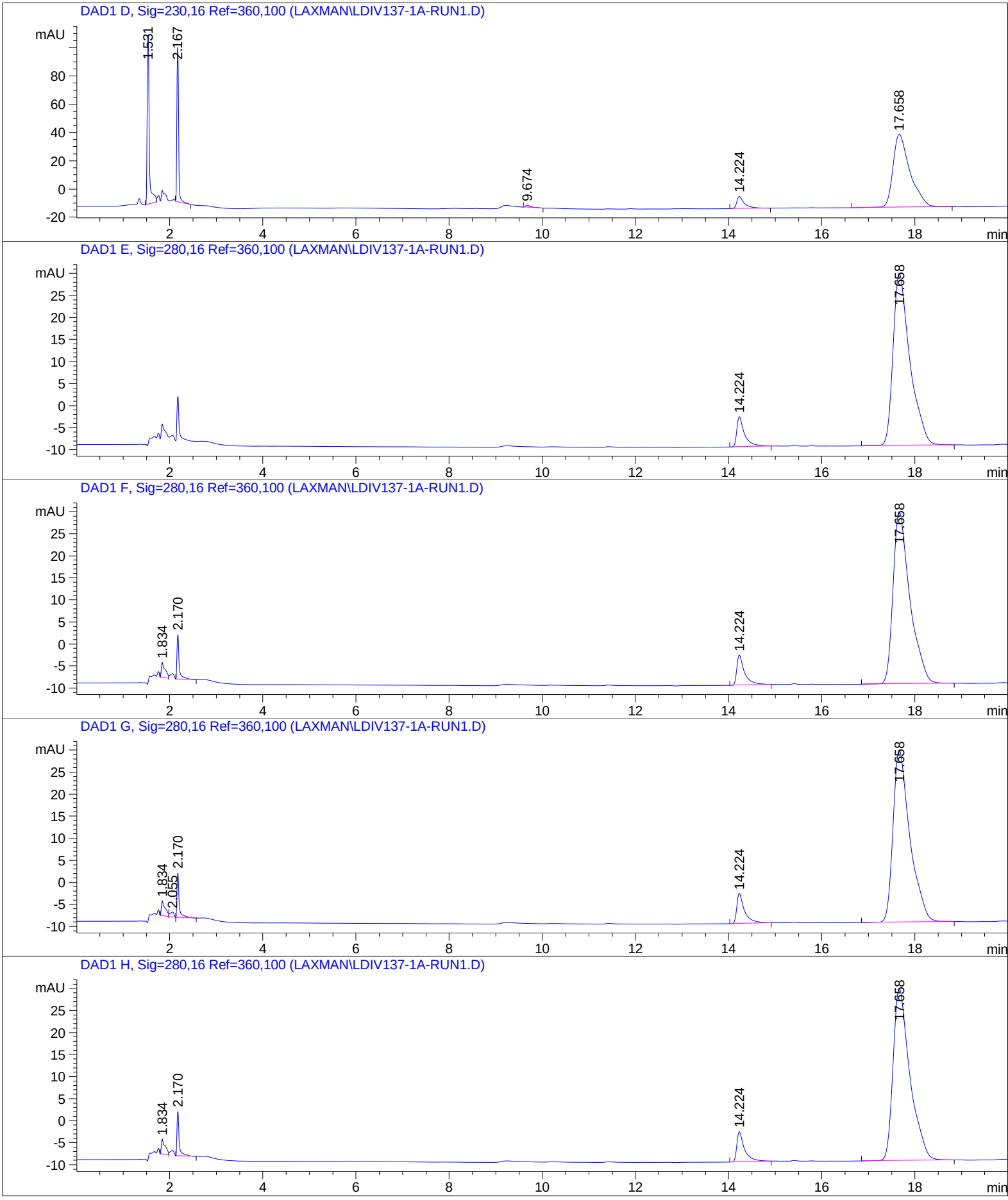
HPLC for compound 37

==========

Acq. Operator : Laxman
Acq. Instrument : Instrument 1 Location : -
Injection Date : 1/24/2014 4:55:16 PM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 1/24/2014 4:30:40 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LDIV137-1A-RUN1.D\DA.M (MASTERMETHOD.M)
Last changed : 1/24/2014 6:21:25 PM by Laxman
Sample Info : Run1-Mastermethod



Data File C:\CHEM32\1\DATA\LAXMAN\LDIV137-1A-RUN1.D
Sample Name: LD-IV-137-1A-Run1



Data File C:\CHEM32\1\DATA\LAXMAN\LDIV137-1A-RUN1.D
Sample Name: LD-IV-137-1A-Run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.836	BV	0.0409	18.19539	6.45327	0.7590
2	2.169	VB	0.0613	58.36508	13.82253	2.4345
3	14.224	BB	0.1651	36.58915	3.20765	1.5262
4	17.658	BV	0.3693	2284.27368	90.85317	95.2804
Totals :				2397.42330	114.33662	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.169	VB	0.0590	58.52003	14.53531	2.6126
2	14.224	BB	0.1630	43.06221	3.77694	1.9225
3	17.658	BV	0.3693	2138.35132	85.04366	95.4649
Totals :				2239.93356	103.35591	

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.530	BV	0.0460	1748.86523	600.82697	28.3435
2	2.166	VB	0.0416	1125.40662	444.25931	18.2392
3	17.658	BB	0.3717	3295.98486	130.90834	53.4173
Totals :				6170.25671	1175.99461	

Data File C:\CHEM32\1\DATA\LAXMAN\LDIV137-1A-RUN1.D
Sample Name: LD-IV-137-1A-Run1

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.531	BV	0.0484	378.01849	121.24623	18.1187
2	2.167	VB	0.0430	291.49344	109.53181	13.9715
3	9.674	BB	0.1070	11.05938	1.48353	0.5301
4	14.224	BB	0.1651	97.43915	8.54247	4.6703
5	17.658	BB	0.3736	1308.33740	51.63680	62.7095
Totals :				2086.34786	292.44084	

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.224	BB	0.1659	78.54280	6.84231	7.4125
2	17.658	BB	0.3691	981.05591	39.04969	92.5875
Totals :				1059.59871	45.89200	

Signal 6: DAD1 F, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.834	BV	0.0789	19.77675	3.44678	1.7756
2	2.170	BB	0.0512	34.41515	10.23419	3.0899
3	14.224	BB	0.1659	78.54280	6.84231	7.0518
4	17.658	BB	0.3691	981.05591	39.04969	88.0826
Totals :				1113.79061	59.57297	

Signal 7: DAD1 G, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.834	BV	0.0789	19.77675	3.44678	1.7652
2	2.055	VB	0.0840	6.56384	1.16043	0.5859
3	2.170	BB	0.0512	34.41515	10.23419	3.0718
4	14.224	BB	0.1659	78.54280	6.84231	7.0105
5	17.658	BB	0.3691	981.05591	39.04969	87.5666

Totals :	1120.35444	60.73339

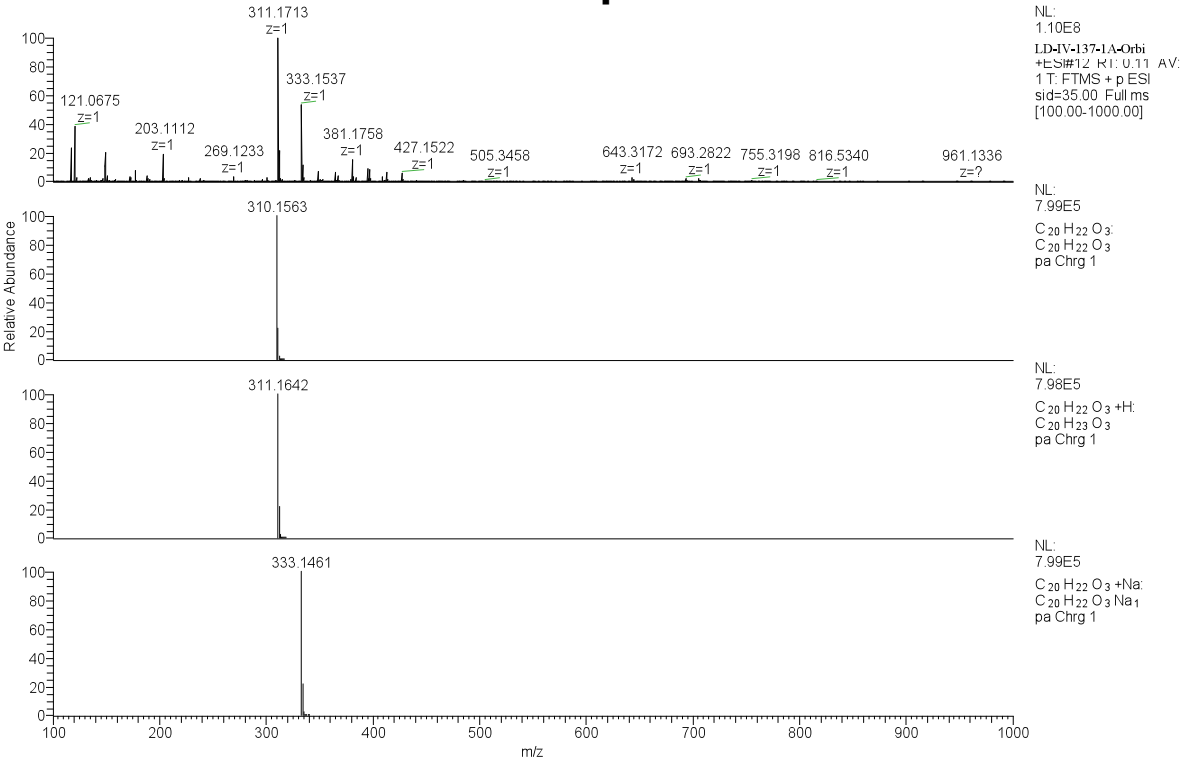
Signal 8: DAD1 H, Sig=280,16 Ref=360,100

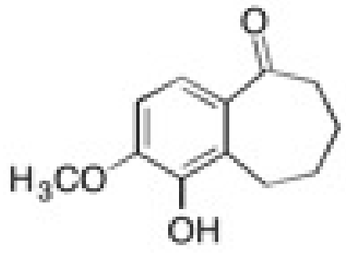
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.834	BV	0.0789	19.77675	3.44678	1.7756
2	2.170	BB	0.0512	34.41515	10.23419	3.0899
3	14.224	BB	0.1659	78.54280	6.84231	7.0518
4	17.658	BB	0.3691	981.05591	39.04969	88.0826
Totals :				1113.79061	59.57297	

==========

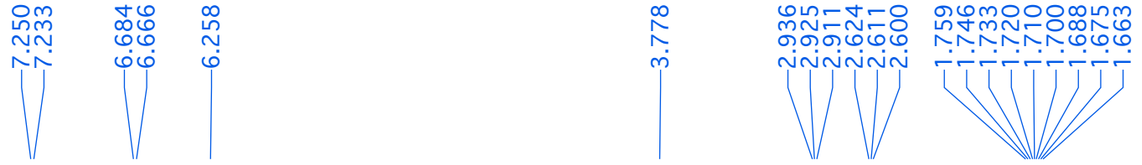
*** End of Report ***

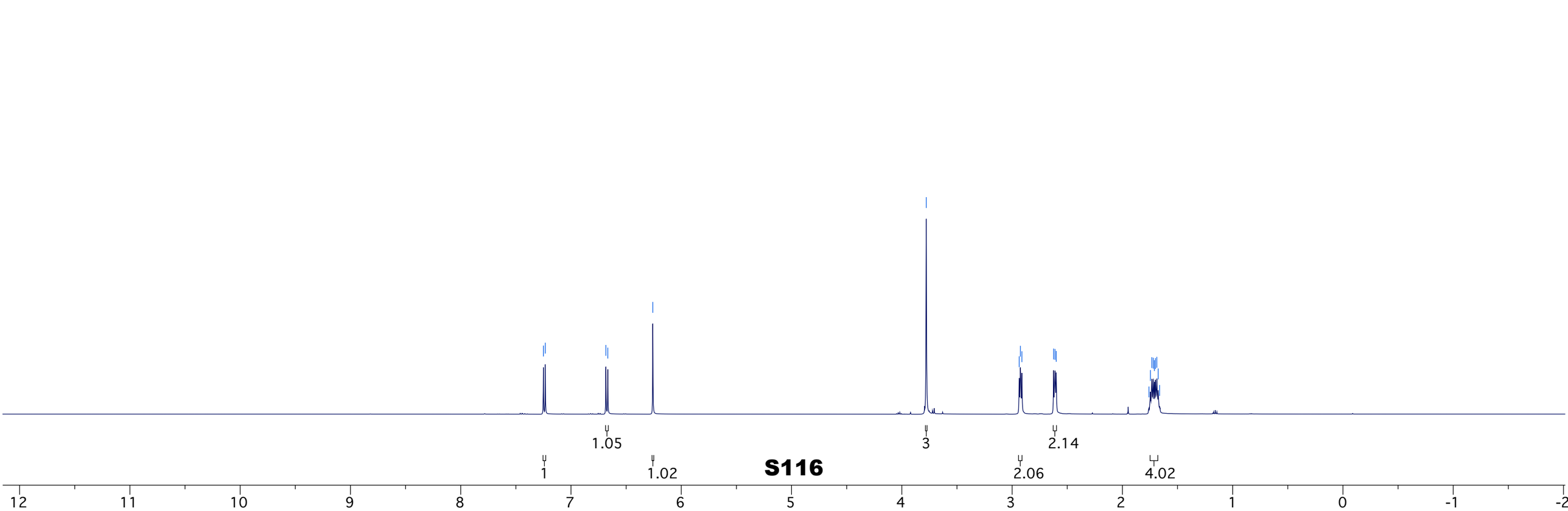
HRMS for compound 37



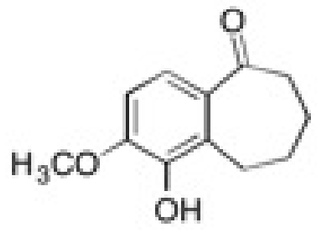

O
H3CO
OH

38


7.250
7.233
6.684
6.666
6.258
3.778
2.936
2.925
2.911
2.624
2.611
2.600
1.759
1.746
1.733
1.720
1.710
1.700
1.688
1.675
1.663


1.05
3
2.14
1
1.02
2.06
4.02
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2

205.037
149.464
142.505
133.005
127.801
120.706
107.929
55.923
40.617
24.439
22.966
21.244



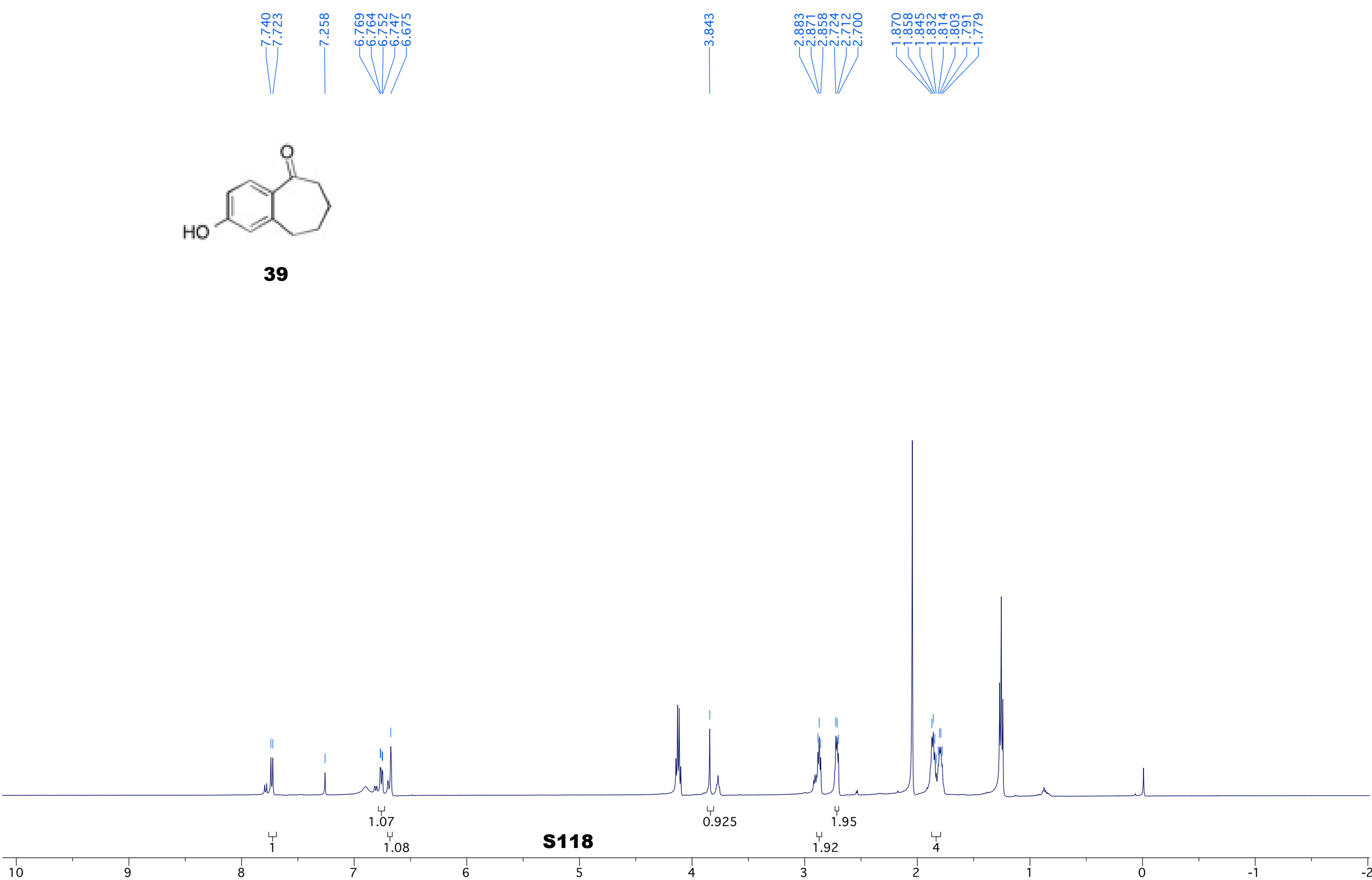
38

S117

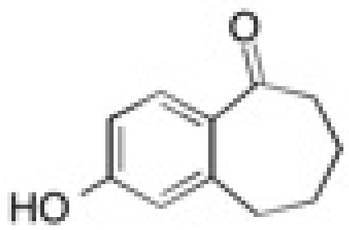
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

O
HO

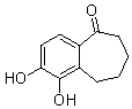
39


7.740
7.723
7.258
6.769
6.764
6.752
6.747
6.675
3.843
2.883
2.871
2.858
2.724
2.712
2.700
1.870
1.858
1.845
1.832
1.814
1.803
1.791
1.779
1.07
0.925
1.95
1
1.08
1.92
4
10
9
8
7
6
5
4
3
2
1
0
-1
-2

204.856
159.897
144.760
131.556
131.306
116.305
113.680
40.693
32.671
24.956
20.653



39

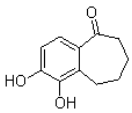

O
HO
OH

40

S120

7.14
7.13
6.79
6.78
3.04
3.03
3.03
3.02
2.64
2.63
2.63
2.62
2.62
1.83
1.83
1.82
1.82
1.81
1.80
1.79
1.78
1.77
1.76
1.76
1.75
1.74
1.74
1.73
1.73
1.72
1.72
0.90
1.00
2.20
2.15
2.18
2.19
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
ppm

205.37
148.11
142.01
132.32
128.93
120.55
112.34
40.25
24.40
22.76
21.02

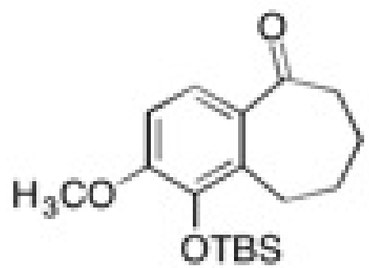


40

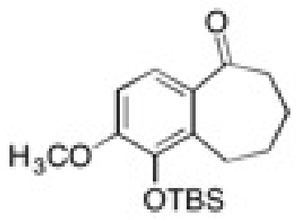
S121

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

ppm



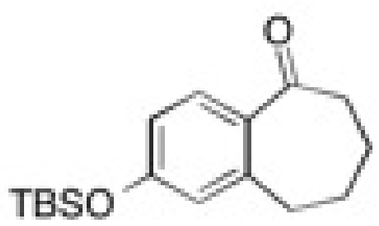
41



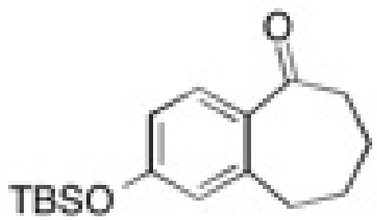
41

204.332
152.941
141.551
132.938
132.732
122.230
108.647
54.603
40.502
25.972
24.582
23.764
21.064
18.785
4.000

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10



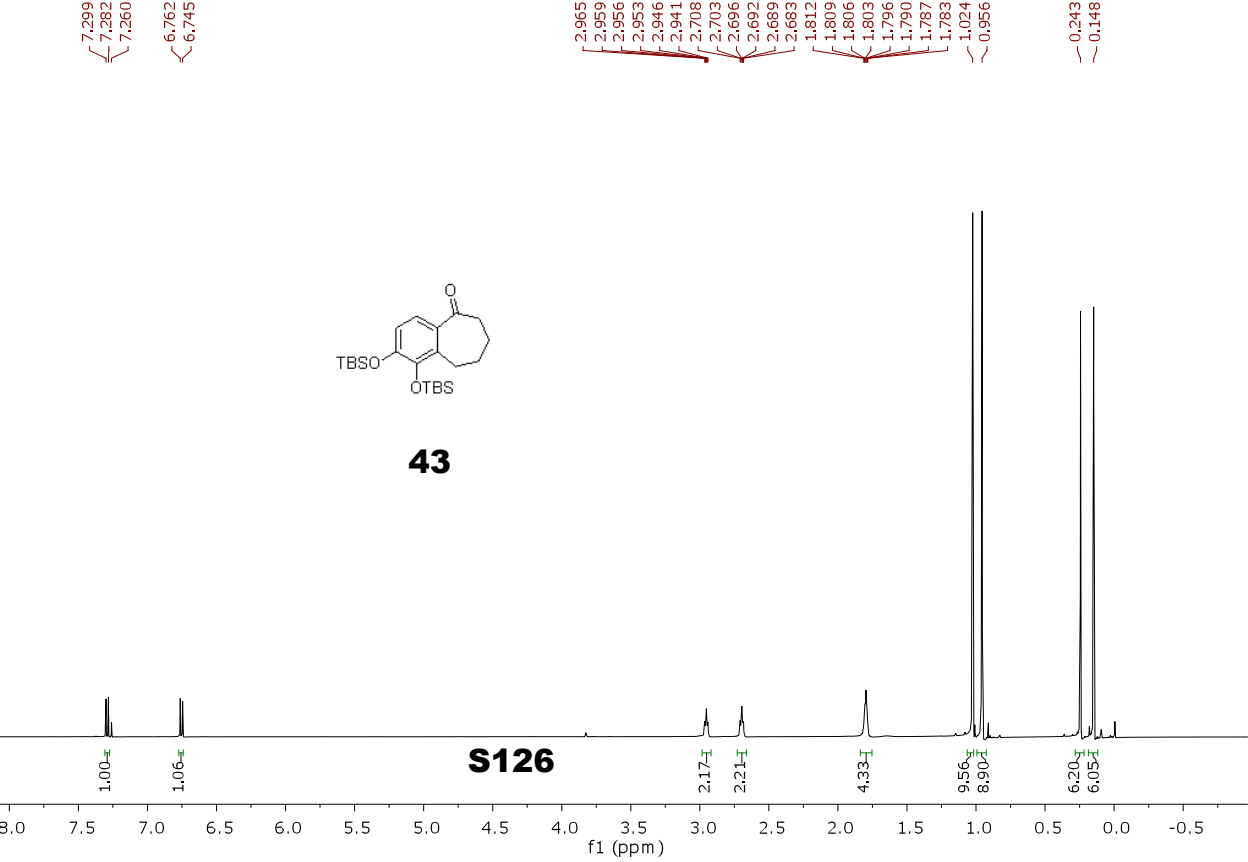
42



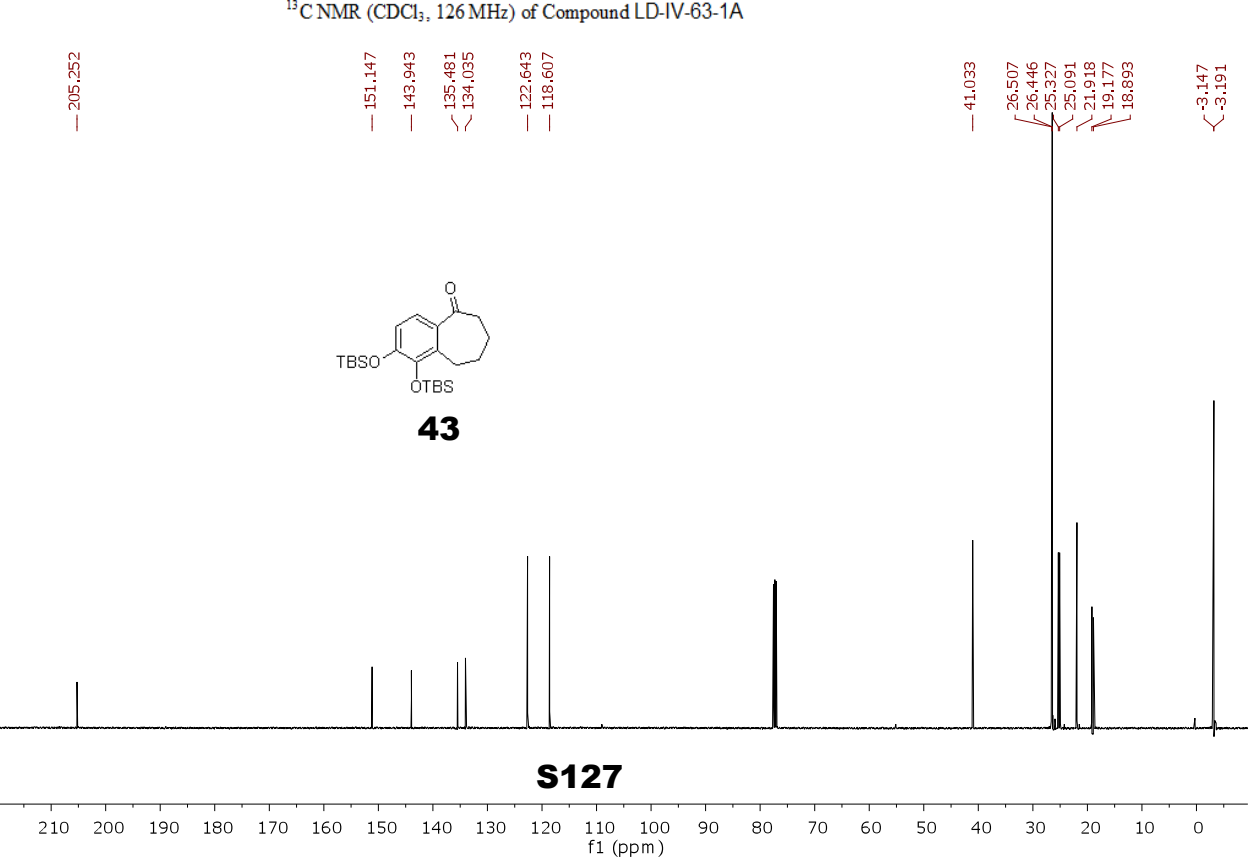
42

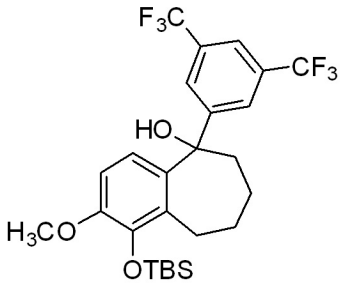
203.662
159.039
143.893
132.003
130.872
120.750
117.783
77.416
77.160
76.905
40.533
32.488
25.455
24.944
20.571
18.013
-4.510

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

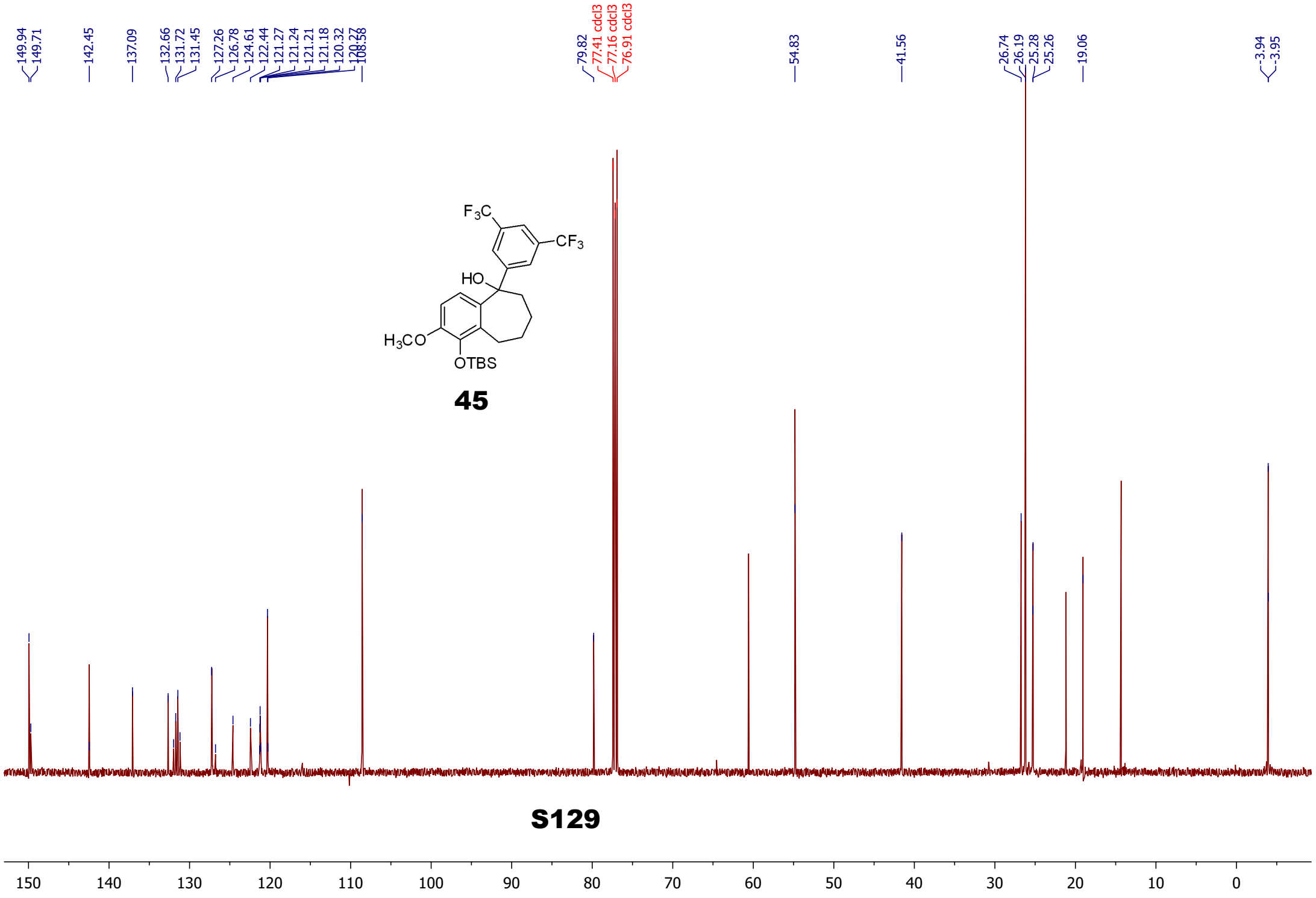

7.299
7.282
7.260
6.762
6.745
2.965
2.959
2.956
2.953
2.946
2.941
2.708
2.703
2.696
2.692
2.689
2.683
1.812
1.809
1.806
1.803
1.796
1.790
1.787
1.783
1.024
0.956
0.243
0.148
O
TBSO
OTBS
43
S126
1.00
1.06
2.17
2.21
4.33
9.56
8.90
6.20
6.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

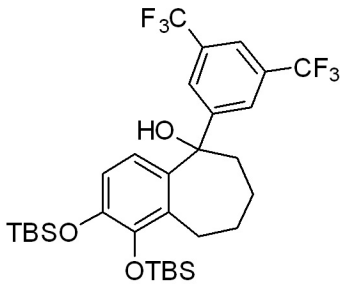
^{13}C NMR ($CDCl_3$, 126 MHz) of Compound LD-IV-63-1A





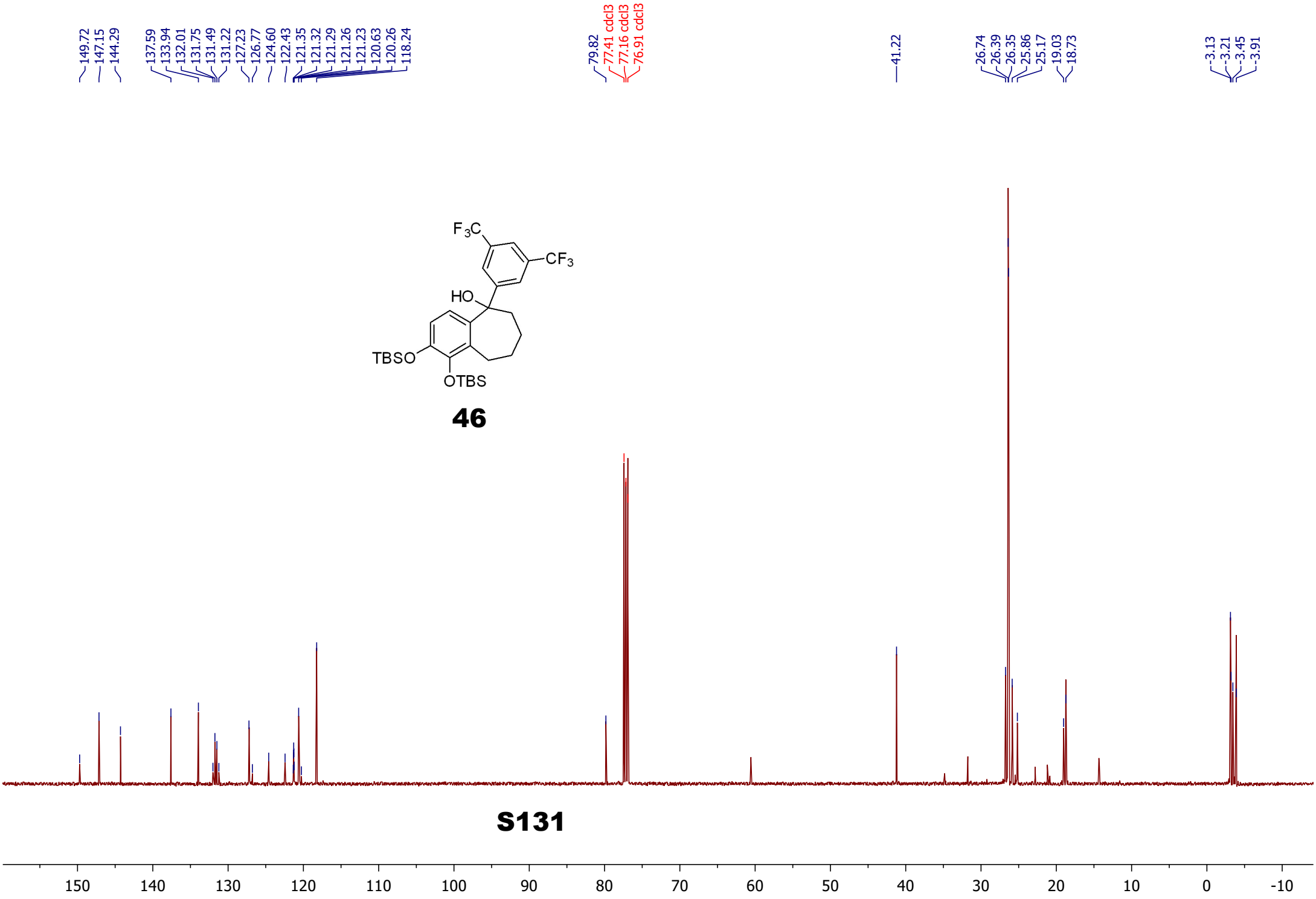
45

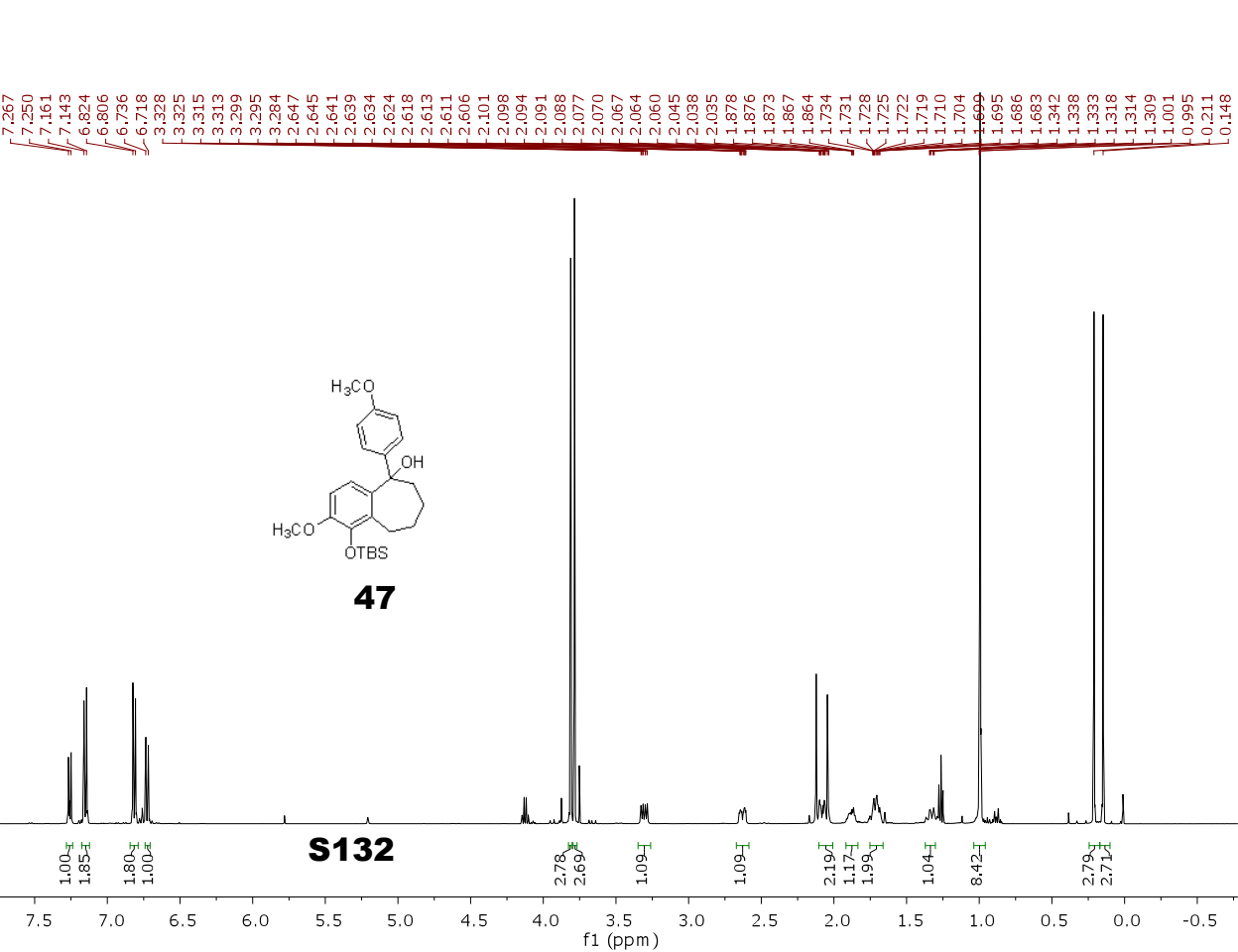

149.94
149.71
142.45
137.09
132.66
131.72
131.45
127.26
126.78
124.61
122.44
121.27
121.24
121.21
121.18
120.32
79.82
77.41 cdcl3
77.16 cdcl3
76.91 cdcl3
54.83
41.56
26.74
26.19
25.28
25.26
19.06
-3.94
-3.95
F3C
CF3
HO
H3CO
OTBS
45
S129
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



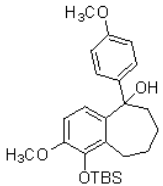
46

S130


149.72
147.15
144.29
137.59
133.94
132.01
131.75
131.49
131.22
127.23
126.77
124.60
122.43
121.35
121.32
121.29
121.26
121.23
120.63
120.26
118.24
79.82
77.41 cdcl3
77.16 cdcl3
76.91 cdcl3
41.22
26.74
26.39
26.35
25.86
25.17
19.03
18.73
-3.13
-3.21
-3.45
-3.91
F3C
CF3
HO
TBSO
OTBS
46
S131
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


7.267
7.250
7.161
7.143
6.824
6.806
6.736
6.718
3.328
3.325
3.315
3.313
3.299
3.295
3.284
2.647
2.645
2.641
2.639
2.634
2.624
2.618
2.613
2.611
2.606
2.101
2.098
2.094
2.091
2.088
2.077
2.070
2.067
2.064
2.060
2.045
2.038
2.035
1.878
1.876
1.873
1.867
1.864
1.734
1.731
1.728
1.725
1.722
1.719
1.710
1.704
1.699
1.695
1.686
1.683
1.342
1.338
1.333
1.318
1.314
1.309
1.001
0.995
0.211
0.148
H3CO
OH
H3CO
OTBS
47
S132
1.00
1.85
1.80
1.00
2.78
2.69
1.09
1.09
2.19
1.17
1.99
1.04
8.42
2.79
2.71
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
-0.5
f1 (ppm)

158.725
149.163
141.838
139.029
137.634
132.676
128.291
119.128
113.611
107.826
79.360
55.207
54.635
41.147
27.127
26.677
26.063
25.458
-3.856
-4.215

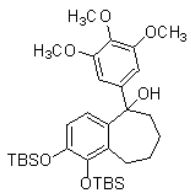


47

S133

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

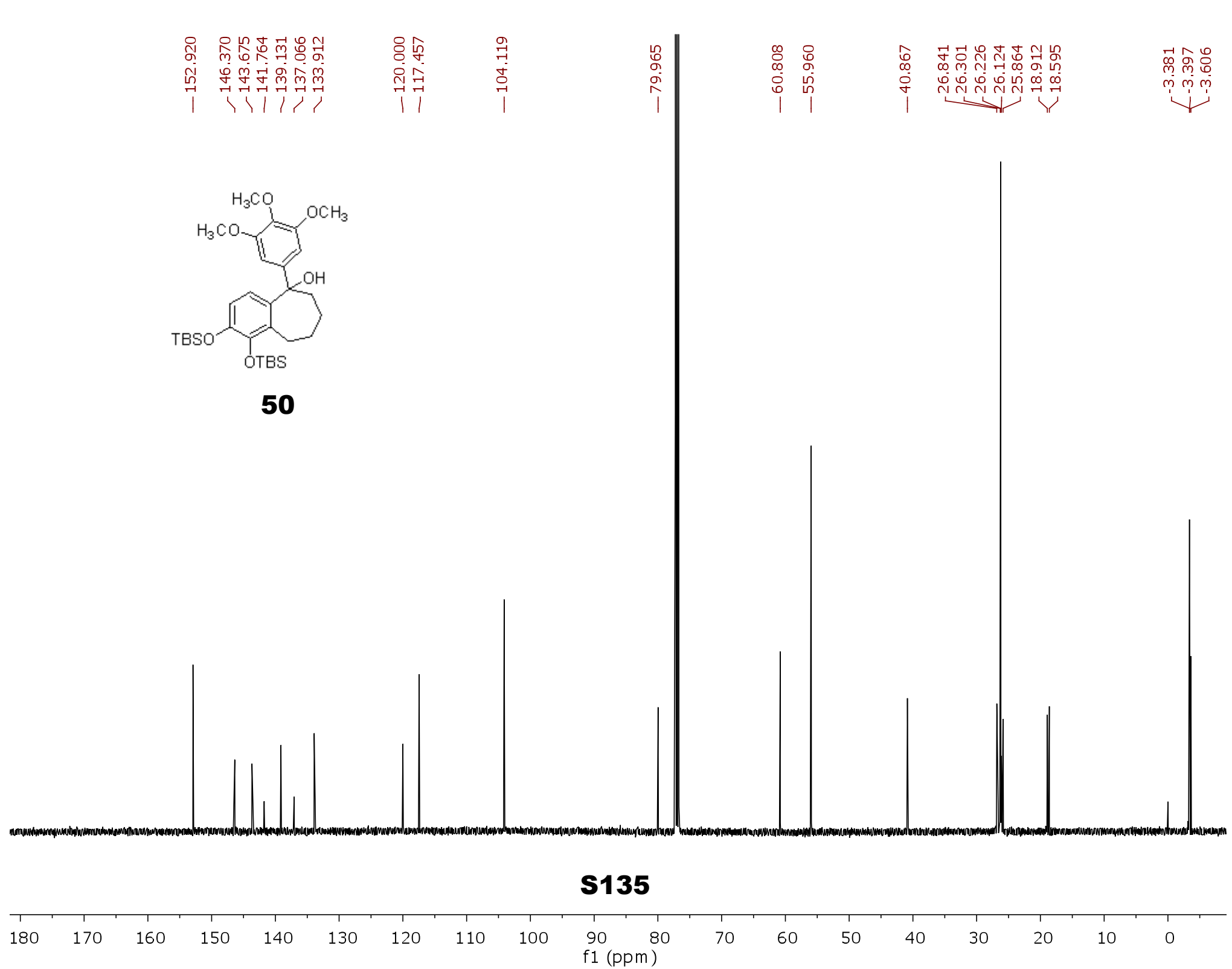
f1 (ppm)


H3CO
OCH3
H3CO
OH
TBSO
OTBS

50

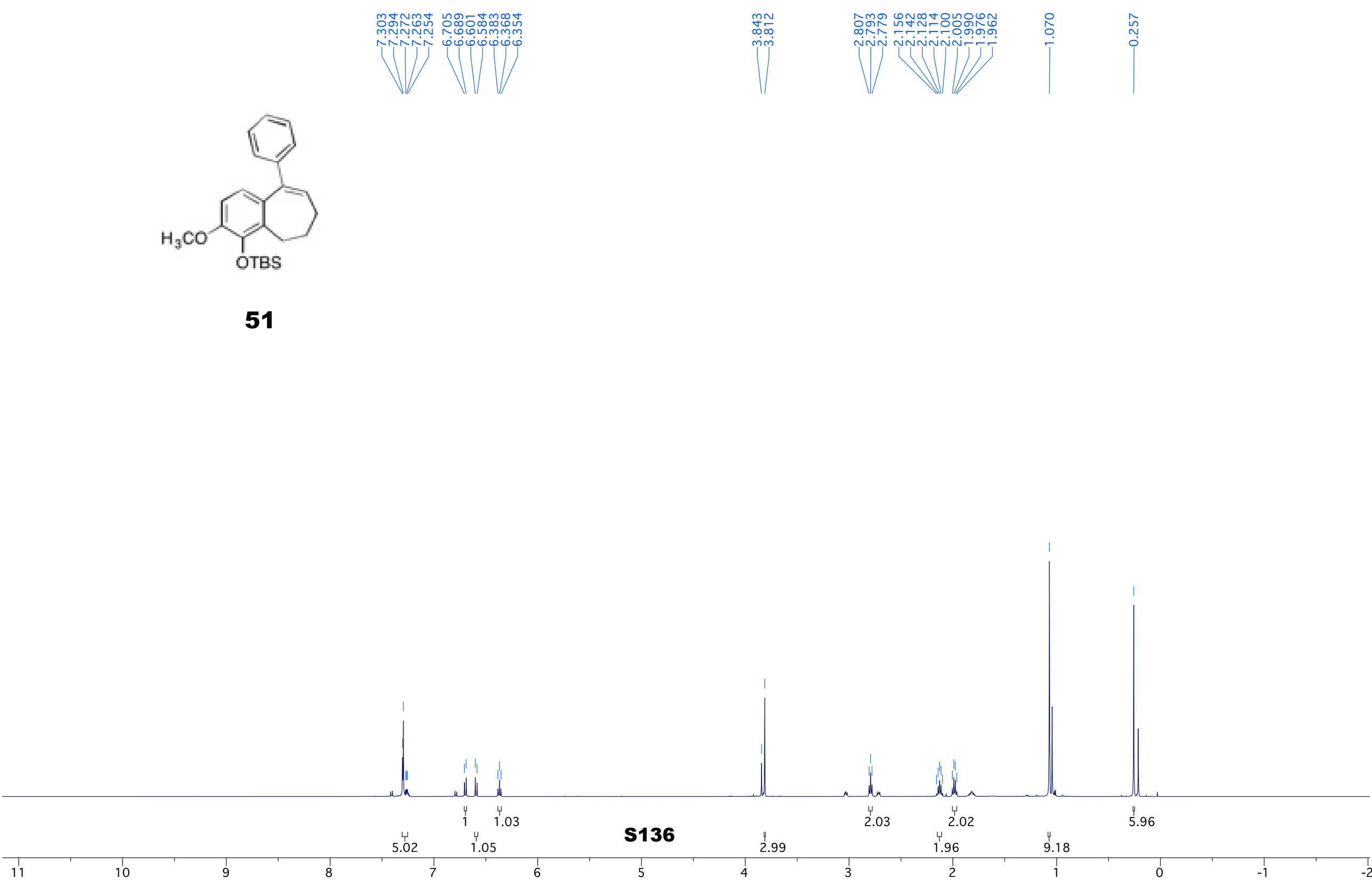
S134

7.259
7.113
7.096
6.733
6.716
6.463
3.831
3.735
3.219
3.216
3.203
3.191
3.187
3.174
2.593
2.591
2.585
2.579
2.573
2.564
2.557
2.551
2.545
2.205
2.181
2.174
2.154
2.148
2.141
2.125
2.119
2.113
2.104
2.097
2.090
1.914
1.907
1.904
1.895
1.884
1.875
1.865
1.785
1.777
1.769
1.762
1.755
1.748
1.746
1.741
1.727
1.718
1.705
1.692
1.679
1.406
1.398
1.385
1.376
0.998
0.947
0.240
0.232
0.153
0.095
1.00
1.03
1.93
2.88
5.58
1.13
1.12
3.06
1.17
2.30
1.27
8.37
8.46
2.79
2.62
2.64
2.64
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
-0.5
f1 (ppm)

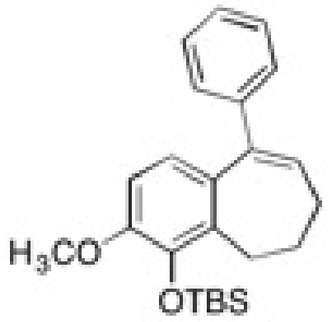

152.920
146.370
143.675
141.764
139.131
137.066
133.912
120.000
117.457
104.119
79.965
60.808
55.960
40.867
26.841
26.301
26.226
26.124
25.864
18.912
18.595
-3.381
-3.397
-3.606
H3CO
OCH3
H3CO
OH
TBSO
OTBS
50
S135
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

H_3CO
OTBS

51


7.303
7.294
7.272
7.263
7.254
6.705
6.689
6.601
6.584
6.383
6.368
6.354
3.843
3.812
2.807
2.793
2.779
2.156
2.142
2.128
2.114
2.100
2.005
1.990
1.976
1.962
1.070
0.257
1
1.03
2.03
2.02
5.96
5.02
1.05
2.99
1.96
9.18
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2

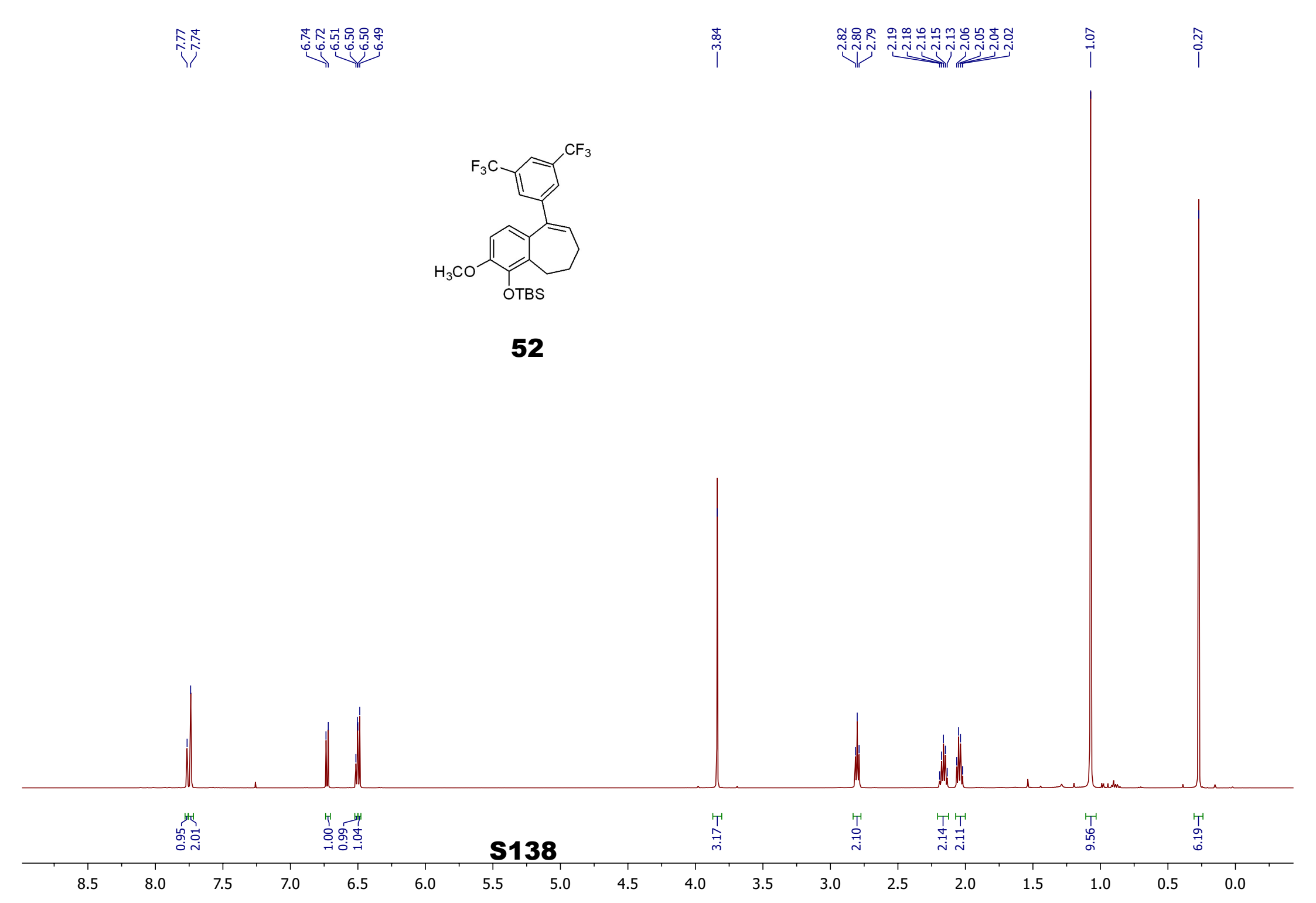
148.616
143.043
142.796
141.572
134.121
133.271
127.968
127.285
126.792
122.102
108.770
108.383
54.661
33.838
26.201
25.645
24.227
19.029
-3.803

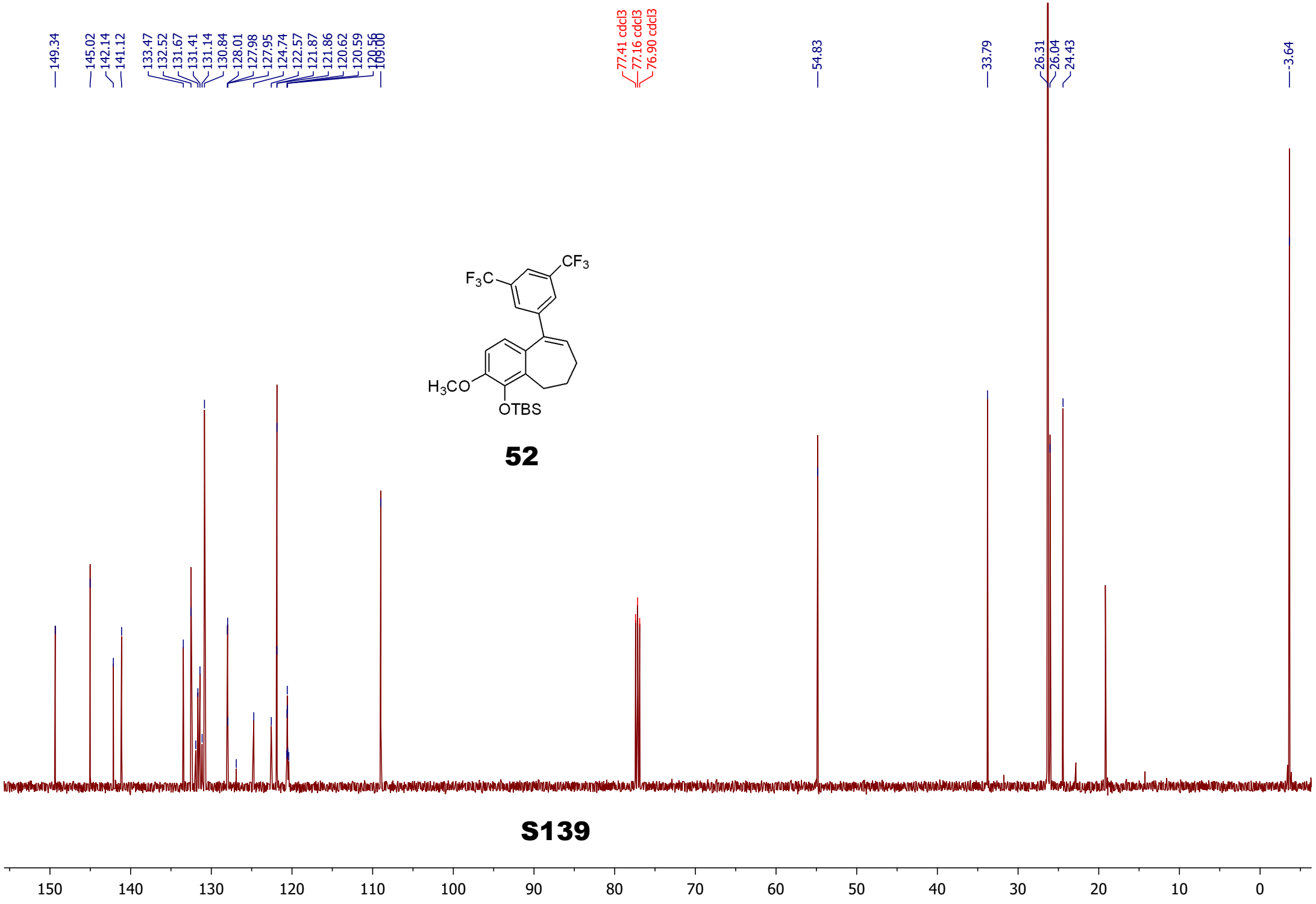


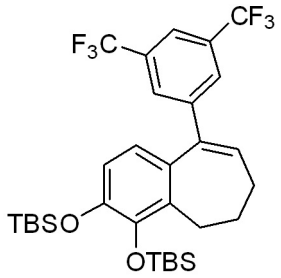
51

S137

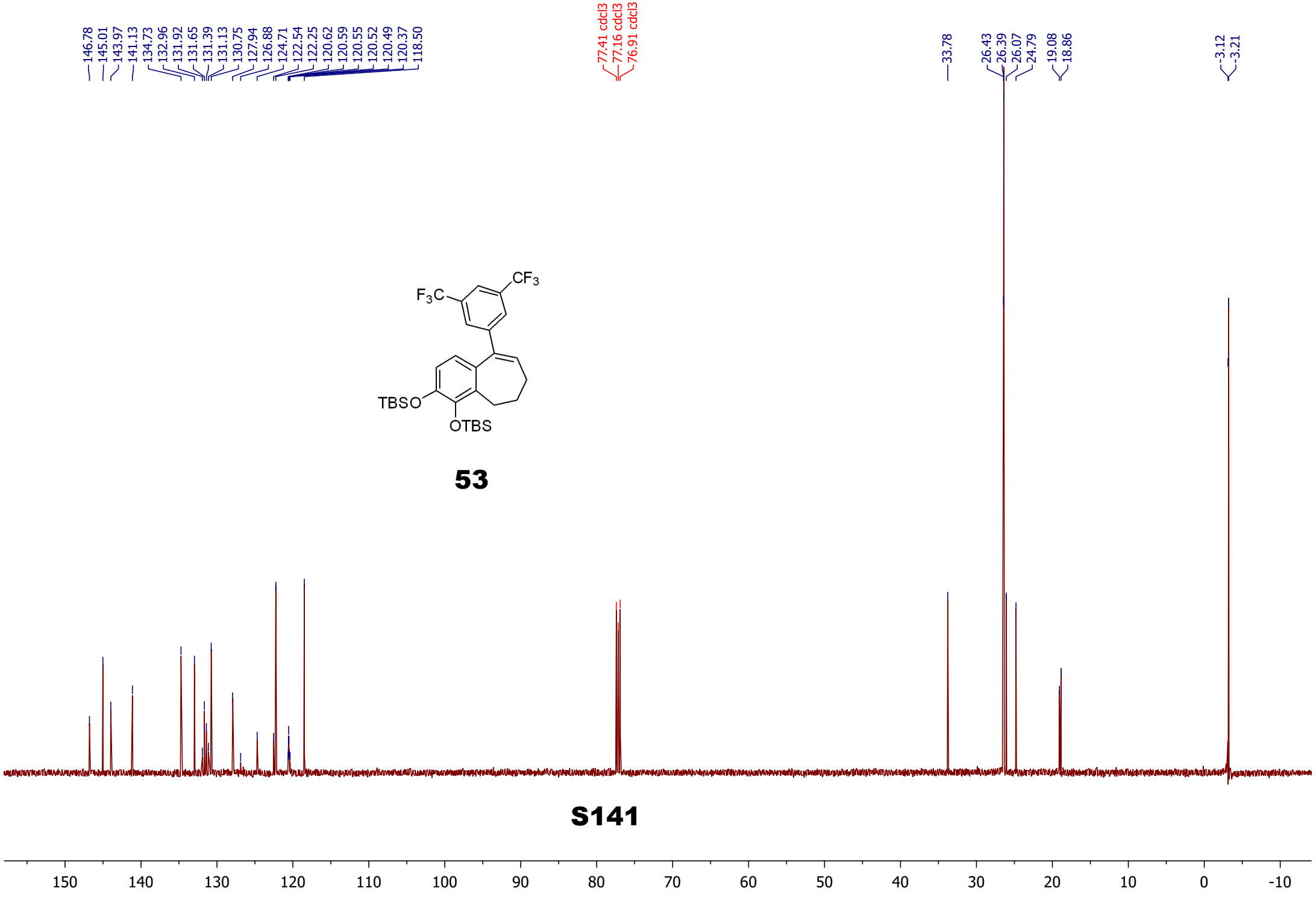
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


7.77
7.74
6.74
6.72
6.51
6.50
6.50
6.49
3.84
2.82
2.80
2.79
2.19
2.18
2.16
2.15
2.13
2.06
2.05
2.04
2.02
1.07
0.27
F3C
CF3
H3CO
OTBS
52
0.95
2.01
1.00
0.99
1.04
3.17
2.10
2.14
2.11
9.56
6.19
S138
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

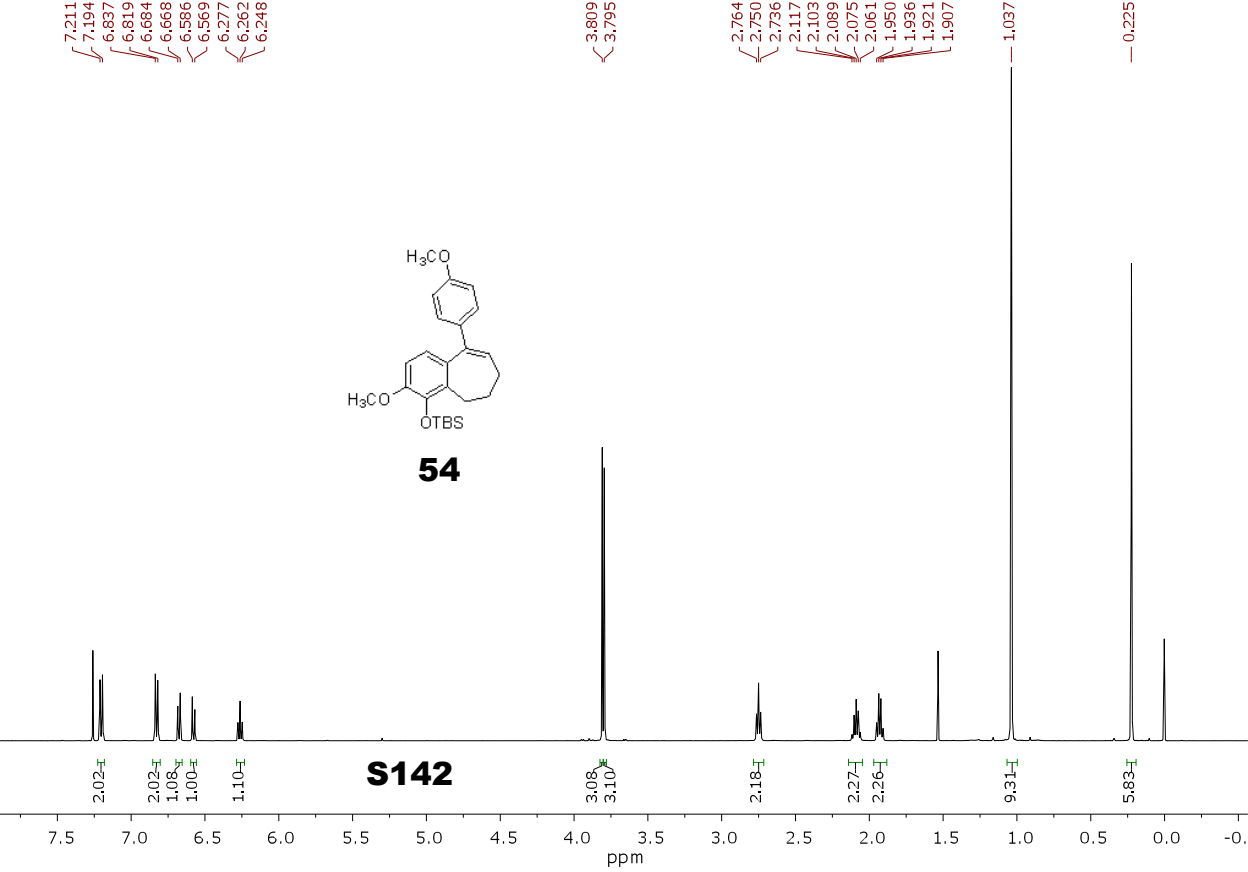

149.34
145.02
142.14
141.12
133.47
132.52
131.67
131.41
131.14
130.84
128.01
127.98
127.95
124.74
122.57
121.87
121.86
120.62
120.59
77.41 cdcl3
77.16 cdcl3
76.90 cdcl3
54.83
33.79
26.31
26.04
24.43
-3.64
F3C
CF3
H3CO
OTBS
52
S139
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



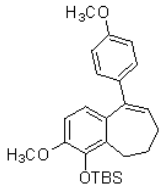
53


146.78
145.01
143.97
141.13
134.73
132.96
131.92
131.65
131.39
131.13
130.75
127.94
126.88
124.71
122.54
122.25
120.62
120.59
120.55
120.52
120.49
120.37
118.50
77.41 cdcl3
77.16 cdcl3
76.91 cdcl3
33.78
26.43
26.39
26.07
24.79
19.08
18.86
-3.12
-3.21
F3C
CF3
TBSO
OTBS
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

53


7.211
7.194
6.837
6.819
6.684
6.668
6.586
6.569
6.277
6.262
6.248
3.809
3.795
2.764
2.750
2.736
2.117
2.103
2.089
2.075
2.061
1.950
1.936
1.921
1.907
1.037
0.225
H3CO
H3CO
OTBS
54
S142
2.02
2.02
1.08
1.00
1.10
3.08
3.10
2.18
2.27
2.26
9.31
5.83
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
-0.
ppm

158.695
148.545
142.389
141.530
135.476
134.306
133.281
128.976
125.689
122.020
113.399
108.311
55.272
54.648
33.940
26.167
25.513
24.187
19.000
-3.847



54

S143

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

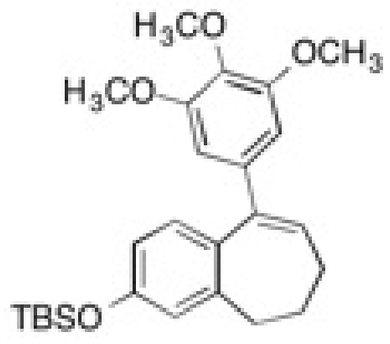
6.914
6.898
6.771
6.766
6.687
6.682
6.670
6.665
6.486
6.354
6.339
6.325

3.859
3.793

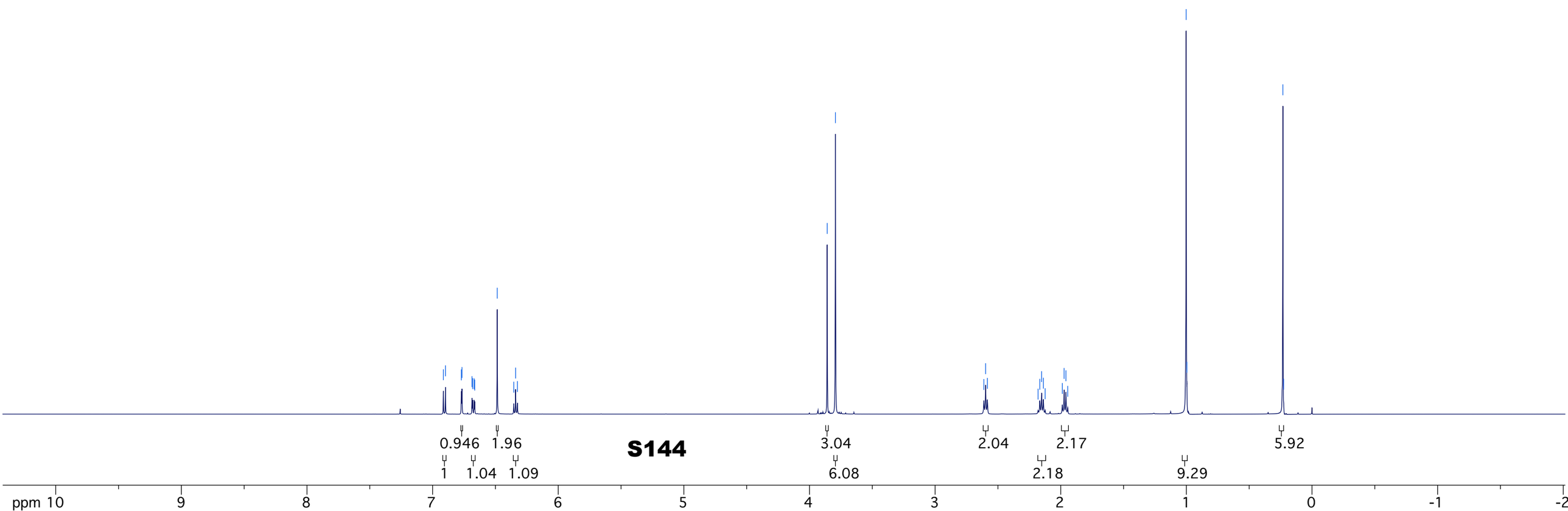
2.612
2.598
2.584
2.181
2.166
2.152
2.138
2.124
1.987
1.973
1.959
1.944

1.002
0.996

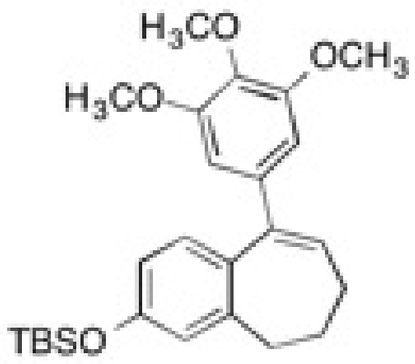
0.232
0.226



55



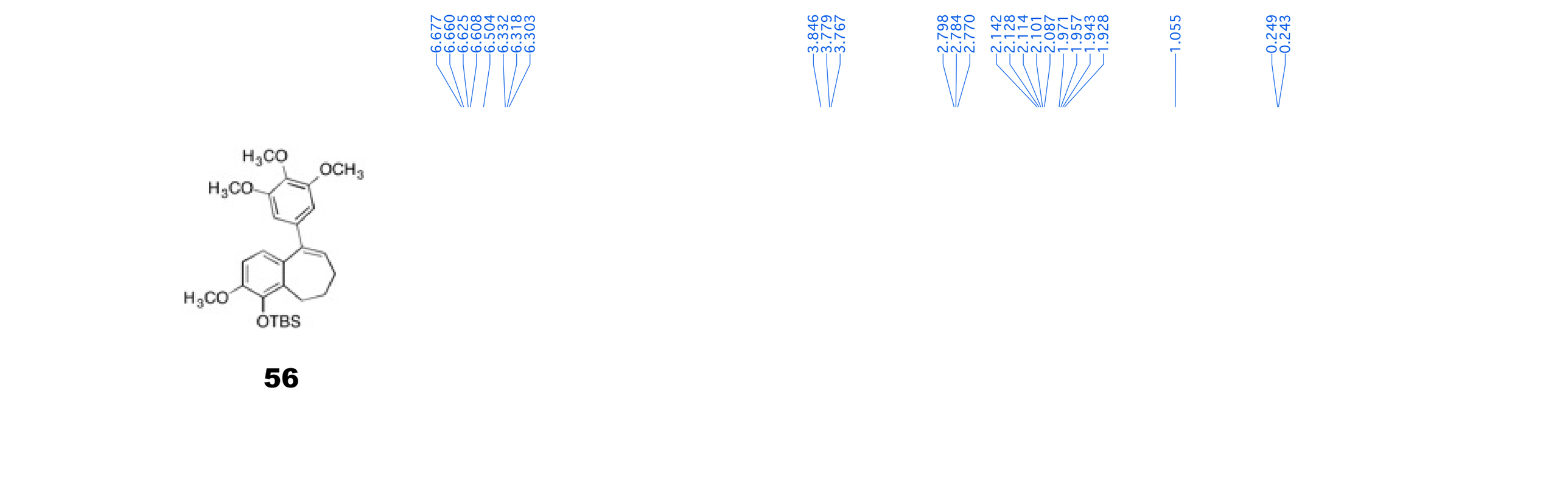
154.441
152.828
143.740
142.756
138.402
137.311
133.028
130.450
127.004
119.970
117.276
105.198
60.876
56.065
35.026
32.583
25.677
25.470
18.200
-4.327



55

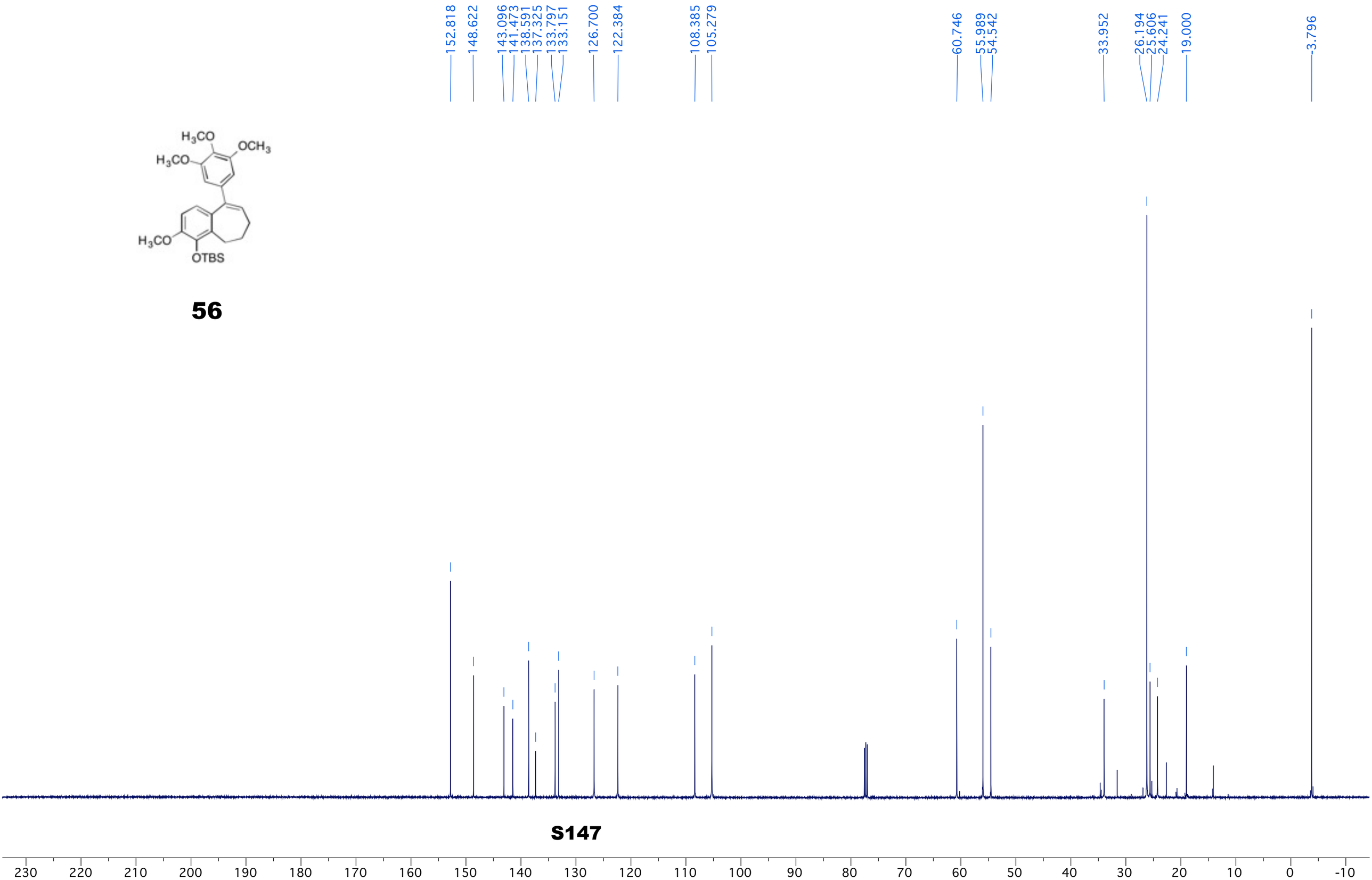
H3CO
OCH3
H3CO
H3CO
OTBS

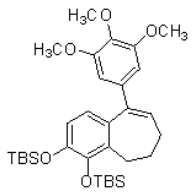
56


6.677
6.660
6.625
6.608
6.504
6.332
6.318
6.303
3.846
3.779
3.767
2.798
2.784
2.770
2.142
2.128
2.114
2.101
2.087
1.971
1.957
1.943
1.928
1.055
0.249
0.243
0.972
1.07
3.05
1.99
2.04
6.02
1
2.01
2.97
6.03
2.02
8.92
10
9
8
7
6
5
4
3
2
1
0
-1
-2

H3CO
OCH3
H3CO
H3CO
OTBS

56

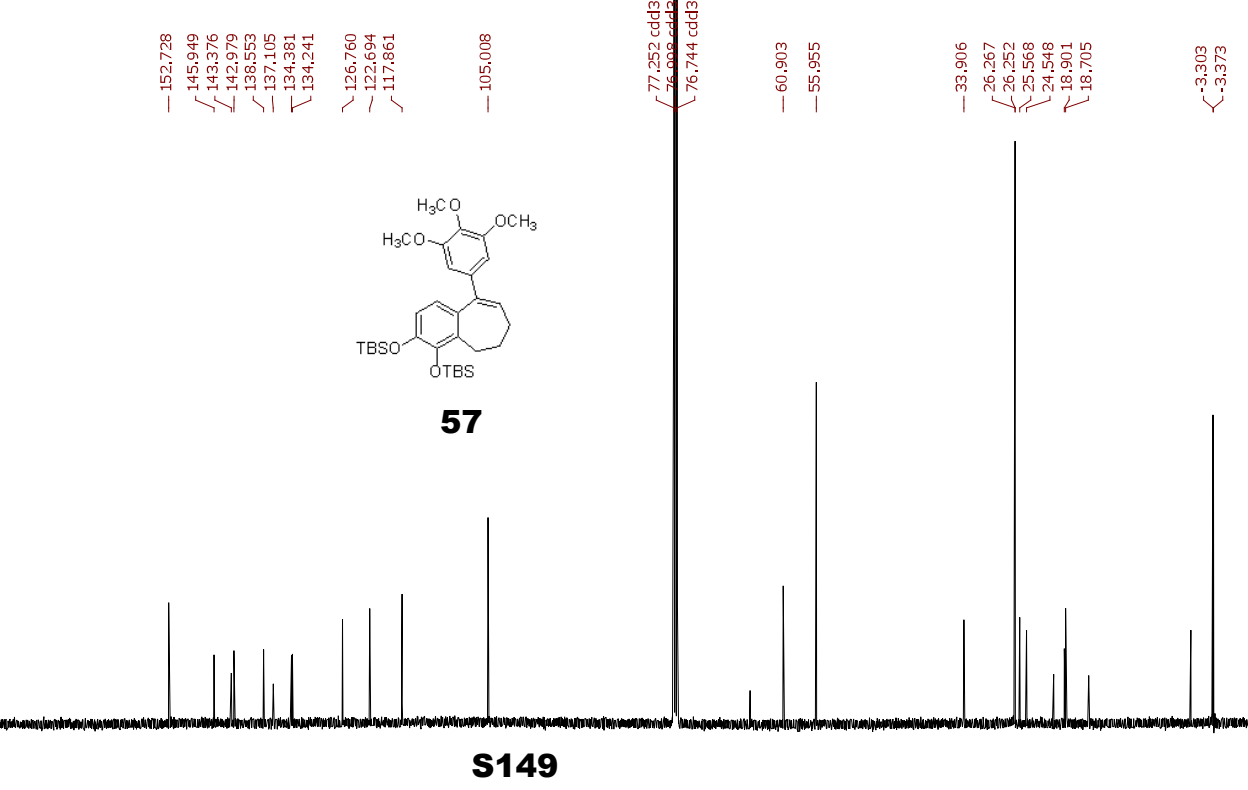

152.818
148.622
143.096
141.473
138.591
137.325
133.797
133.151
126.700
122.384
108.385
105.279
60.746
55.989
54.542
33.952
26.194
25.606
24.241
19.000
3.796
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

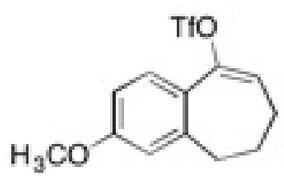

H3CO
OCH3
H3CO
TBSO
OTBS

57

S148

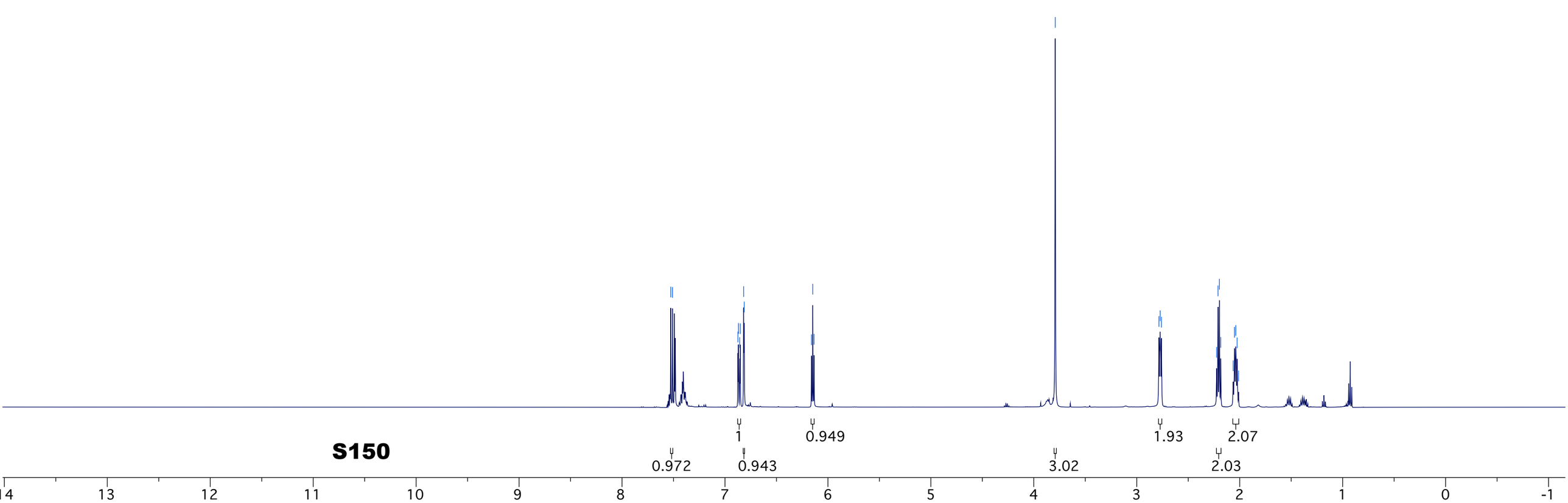
7.260
6.696
6.679
6.532
6.515
6.452
6.343
6.329
6.314
3.850
3.775
2.717
2.703
2.689
2.130
2.116
2.088
2.074
1.971
1.968
1.954
1.940
1.925
1.039
0.953
0.235
0.200
-0.002
1.00
1.02
1.83
1.15
2.67
5.22
2.07
4.86
2.03
8.04
8.09
5.07
5.02
f1 (ppm)


152.728
145.949
143.376
142.979
138.553
137.105
134.381
134.241
126.760
122.694
117.861
105.008
77.252 cdd3
76.744 cdd3
60.903
55.955
33.906
26.267
26.252
25.568
24.548
18.901
18.705
-3.303
-3.373
H3CO
OCH3
H3CO
TBSO
OTBS
57
S149
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

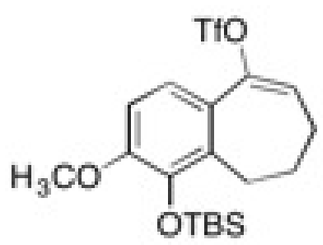

TfO
H3CO

59

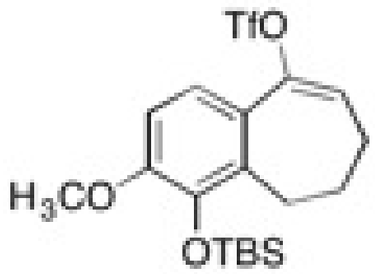

7.527
7.509
6.874
6.869
6.857
6.852
6.819
6.813
6.159
6.147
6.135
3.792
2.785
2.772
2.760
2.224
2.210
2.197
2.184
2.064
2.050
2.038
2.025
2.011


1
0.949
1.93
2.07
0.972
0.943
3.02
2.03
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1

S150



60

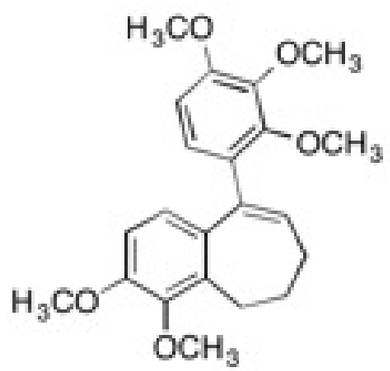


60

150.784
146.512
132.937
130.931
129.924
125.955
120.932
119.727
108.816
54.703
30.472
25.994
24.985
24.507
18.869
-4.022

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

6.921
6.904
6.660
6.643
6.625
6.590
6.573
6.119
6.105
6.090
3.856
3.836
3.820
3.813
3.375
2.869
2.855
2.841
2.175
2.163
2.149
2.134
2.121
1.977
1.963
1.949
1.935

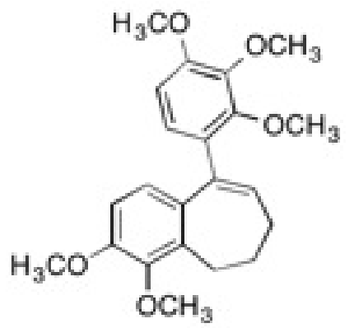


61

2.07
1.01
3.05
3.03
2.03
1.98
1
0.914
2.99
3.02
2.9
2.05

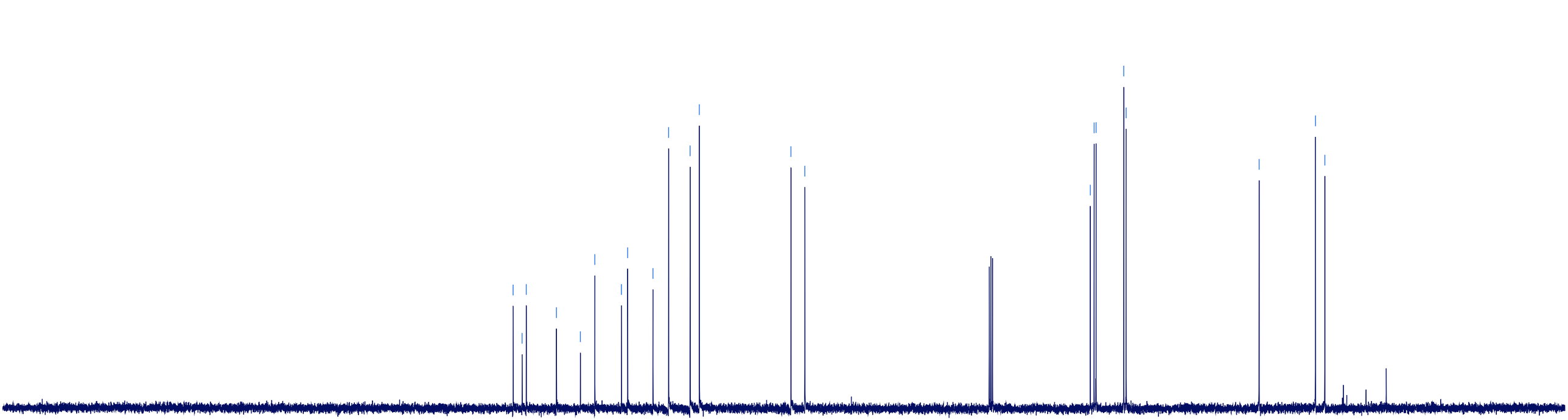
S153

ppm 9
8
7
6
5
4
3
2
1
0
-1
-2



61

153.089
151.660
151.003
146.194
142.389
140.092
135.857
134.864
130.835
128.354
124.926
123.467
108.885
106.679
61.246
60.648
60.329
55.929
55.550
34.392
25.419
23.932



S154

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_51_CLEAN2.D
Sample Name: CAH_3_51_Clean

HPLC for compound 61

```
==========================================================================
Acq. Operator   : Christine
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/18/2013 10:11:00 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 7/18/2013 10:07:07 AM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_3_51_CLEAN2.D\DA.M (MASTERMETHOD.M)
Last changed    : 7/18/2013 11:01:15 AM by Christine
Sample Info     :
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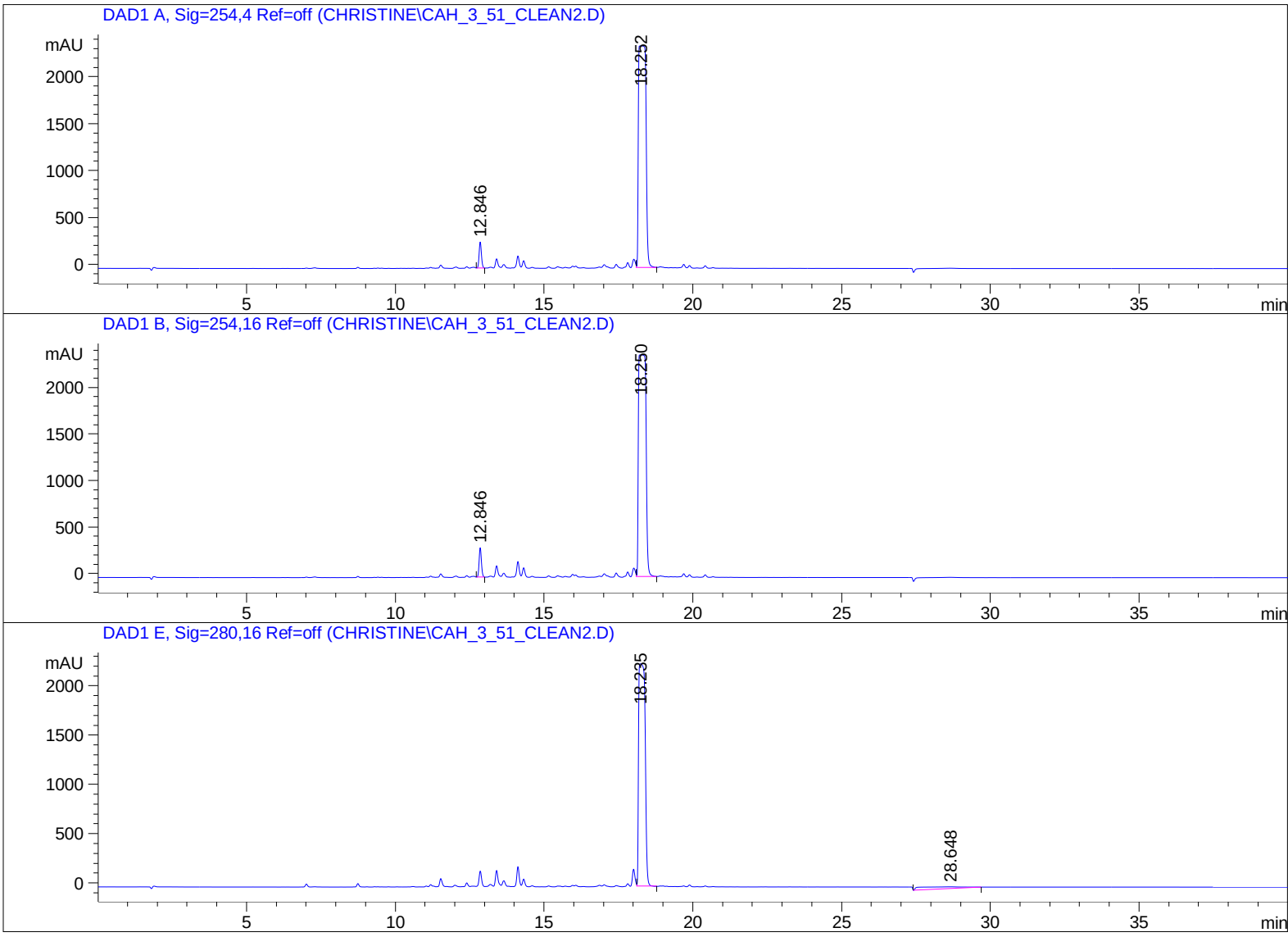
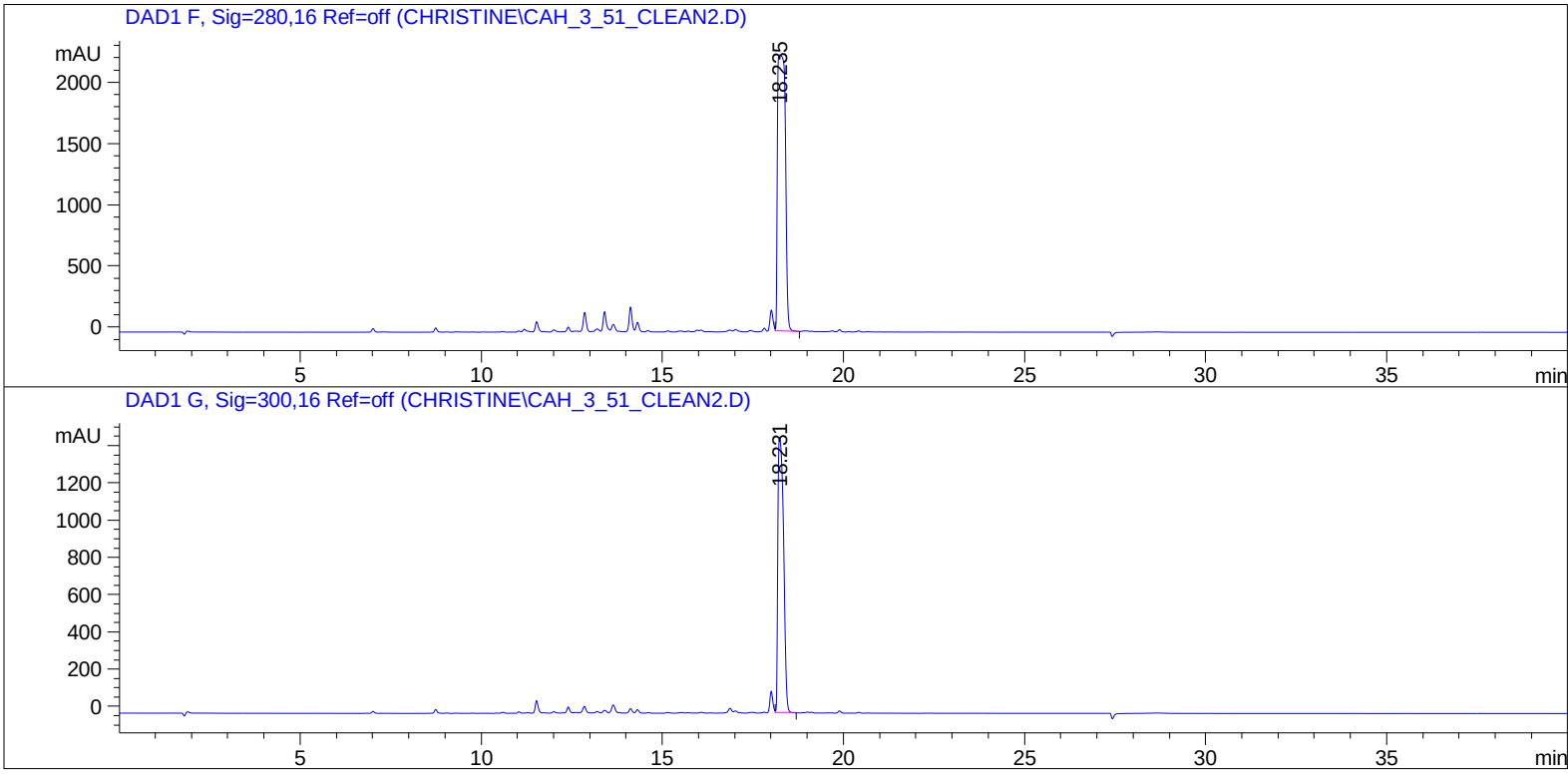


Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_3_51_CLEAN2.D
Sample Name: CAH_3_51_Clean



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.846	VB	0.0826	1498.54224	279.23978	3.5576
2	18.252	VV	0.2144	4.06234e4	2365.51514	96.4424

Totals : 4.21219e4 2644.75491

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.846	VB	0.0826	1700.93689	317.25293	3.9841
2	18.250	VV	0.2824	4.09918e4	2386.45435	96.0159

Totals :			4.26927e4	2703.70728	

Signal 3: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.235	VB	0.2472	3.38849e4	2256.16064	93.6335
2	28.648	BB	1.5395	2303.96753	18.24143	6.3665
Totals :				3.61888e4	2274.40207	

Signal 4: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.235	VB	0.2472	3.38849e4	2256.16064	100.0000
Totals :				3.38849e4	2256.16064	

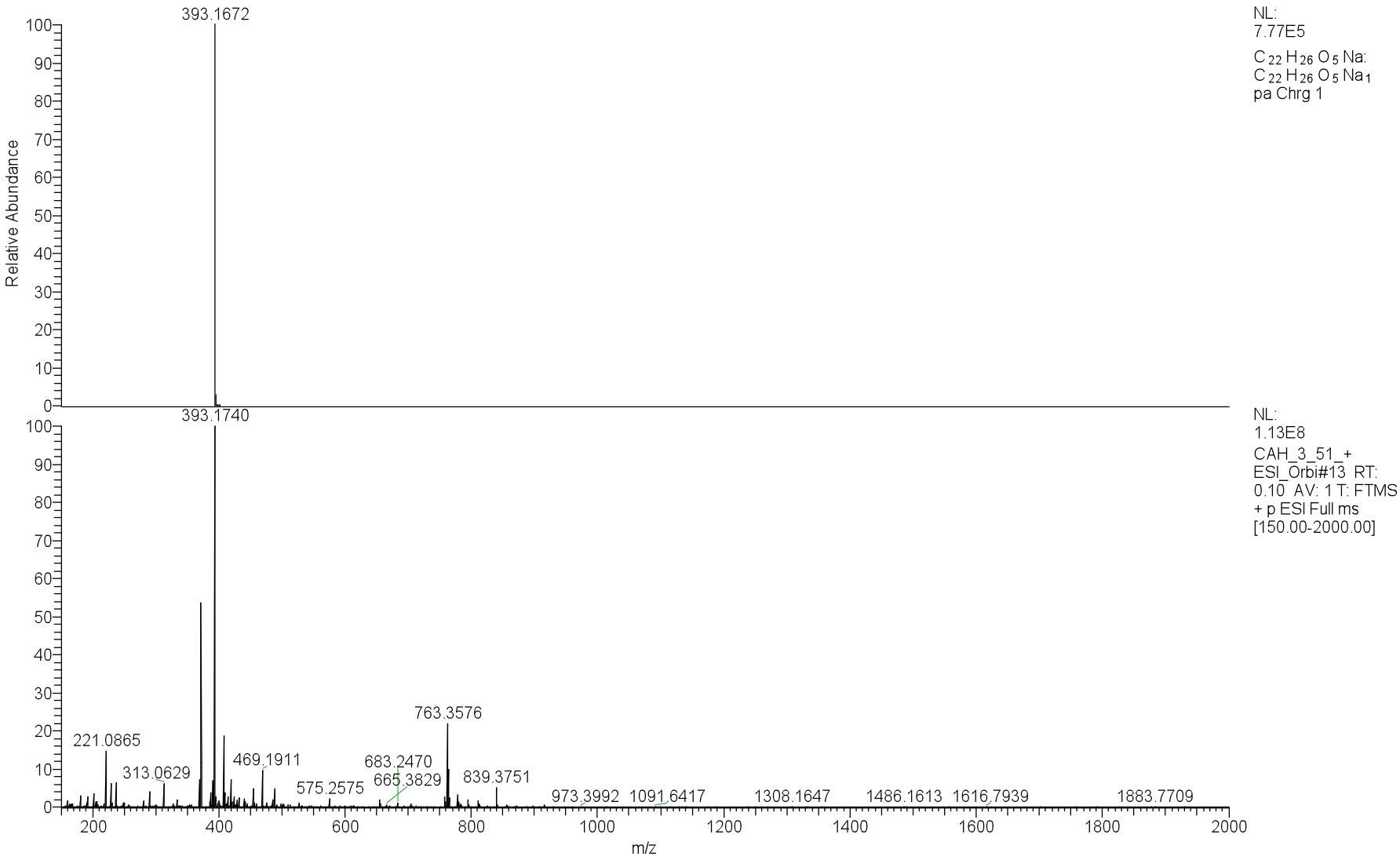
Signal 5: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.231	VB	0.1840	1.62398e4	1479.67639	100.0000
Totals :				1.62398e4	1479.67639	

==========

*** End of Report ***

HRMS for compound 61



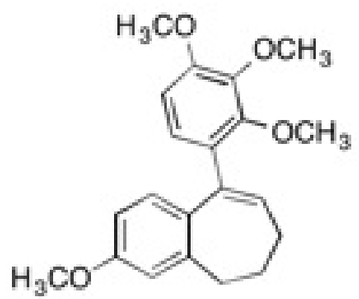
7.039
7.022
6.882
6.877
6.780
6.775
6.763
6.758
6.580
6.421
6.406
6.392

4.144
4.130

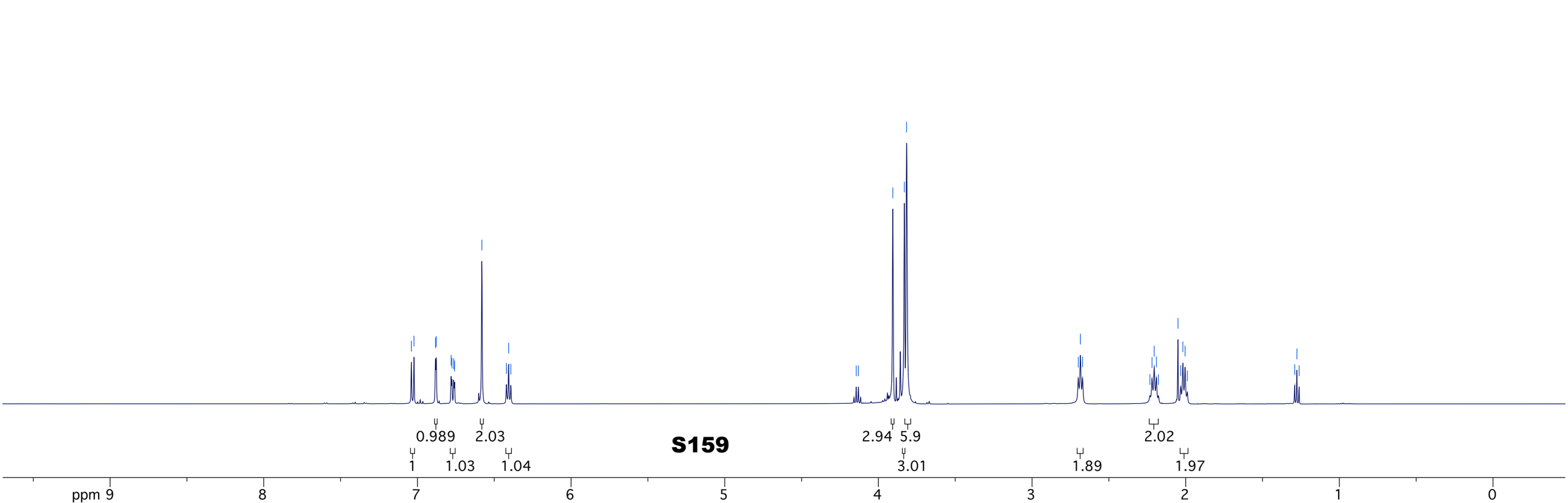
3.906
3.831
3.816

2.699
2.685
2.671
2.233
2.219
2.205
2.191
2.177
2.050
2.032
2.018
2.004
1.989

1.290
1.276
1.262

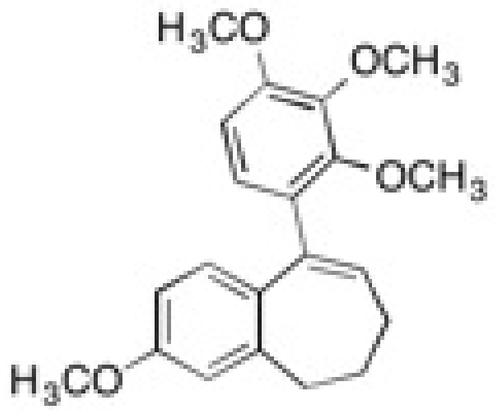


62



S159

158.481
153.433
152.847
143.615
142.725
138.316
137.409
132.270
130.435
126.740
123.534
113.837
111.119
105.215
60.557
60.094
55.846
54.835
34.999
32.702
25.403



62

S160

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

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_18000001.D
Sample Name: CAH_4_18

HPLC for compound 62

```
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Acq. Operator   : Christine
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 5/19/2014 1:03:12 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 5/19/2014 1:01:32 PM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\CAH_4_18000001.D\DA.M (MASTERMETHOD.M)
Last changed    : 5/19/2014 2:14:01 PM by Christine
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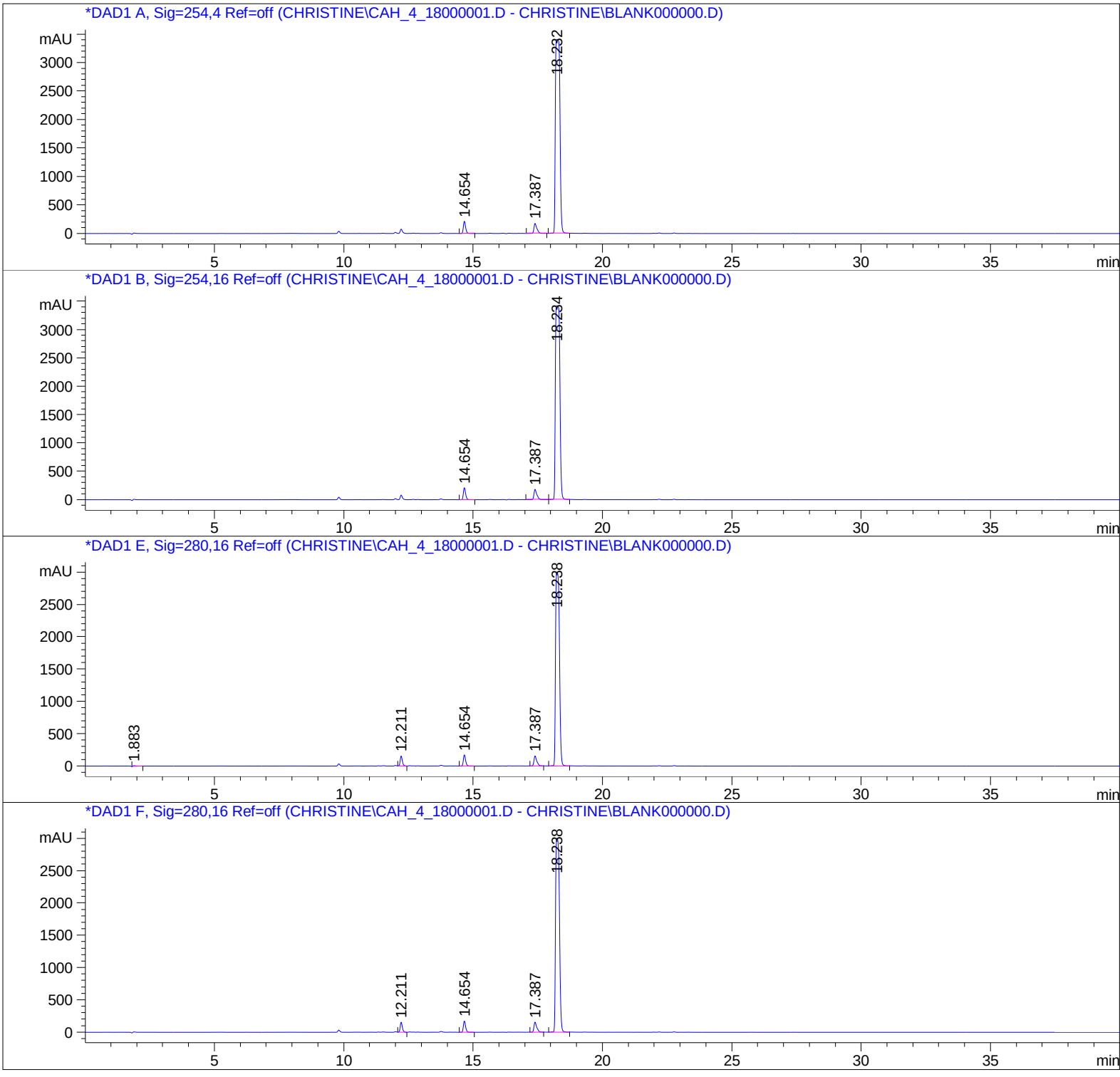
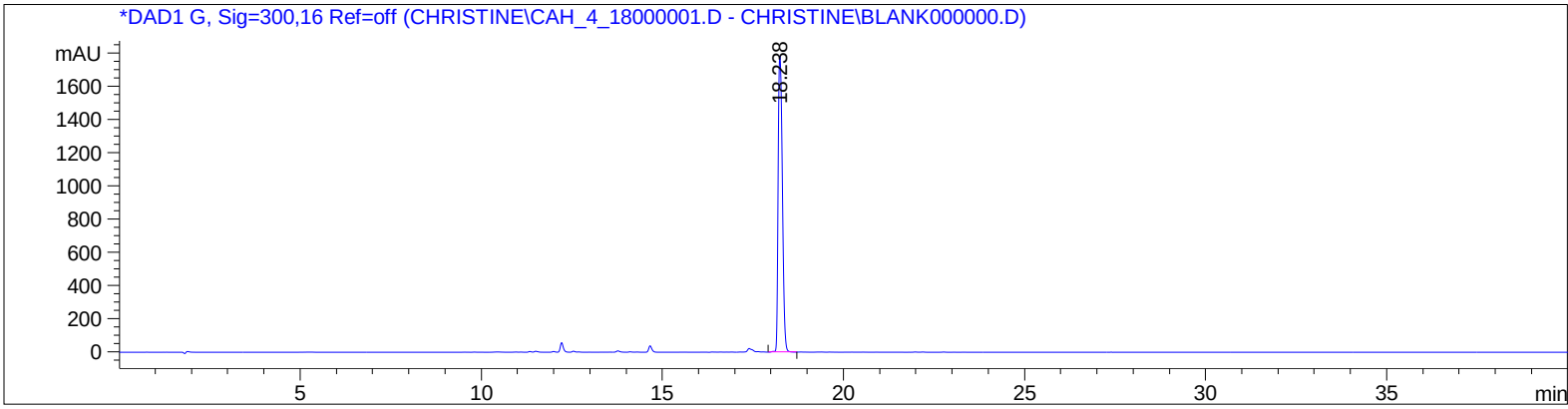


Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_18000001.D
Sample Name: CAH_4_18



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.654	BB	0.0924	1296.31226	214.94673	2.9570
2	17.387	BB	0.1137	1383.36279	176.06273	3.1556
3	18.232	BV	0.1989	4.11593e4	3408.02661	93.8875
Totals :				4.38390e4	3799.03607	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.654	BB	0.0924	1277.60864	211.86137	2.9561
2	17.387	BB	0.1133	1417.87061	181.21844	3.2806
3	18.234	BV	0.1921	4.05239e4	3422.38574	93.7633
Totals :				4.32194e4	3815.46556	

S162

Data File C:\CHEM32\1\DATA\CHRISTINE\CAH_4_18000001.D
Sample Name: CAH_4_18

Signal 3: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.883	BB	0.1486	147.29576	14.24430	0.4289
2	12.211	VB	0.0867	883.39655	154.41168	2.5722
3	14.654	BB	0.0921	1038.45081	172.92358	3.0237
4	17.387	VV	0.1117	1195.93066	155.58759	3.4822
5	18.238	BV	0.1687	3.10785e4	3004.24097	90.4929
Totals :				3.43436e4	3501.40812	

Signal 4: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.211	VB	0.0867	883.39655	154.41168	2.5833
2	14.654	BB	0.0921	1038.45081	172.92358	3.0367
3	17.387	VV	0.1117	1195.93066	155.58759	3.4972
4	18.238	BV	0.1687	3.10785e4	3004.24097	90.8827
Totals :				3.41963e4	3487.16382	

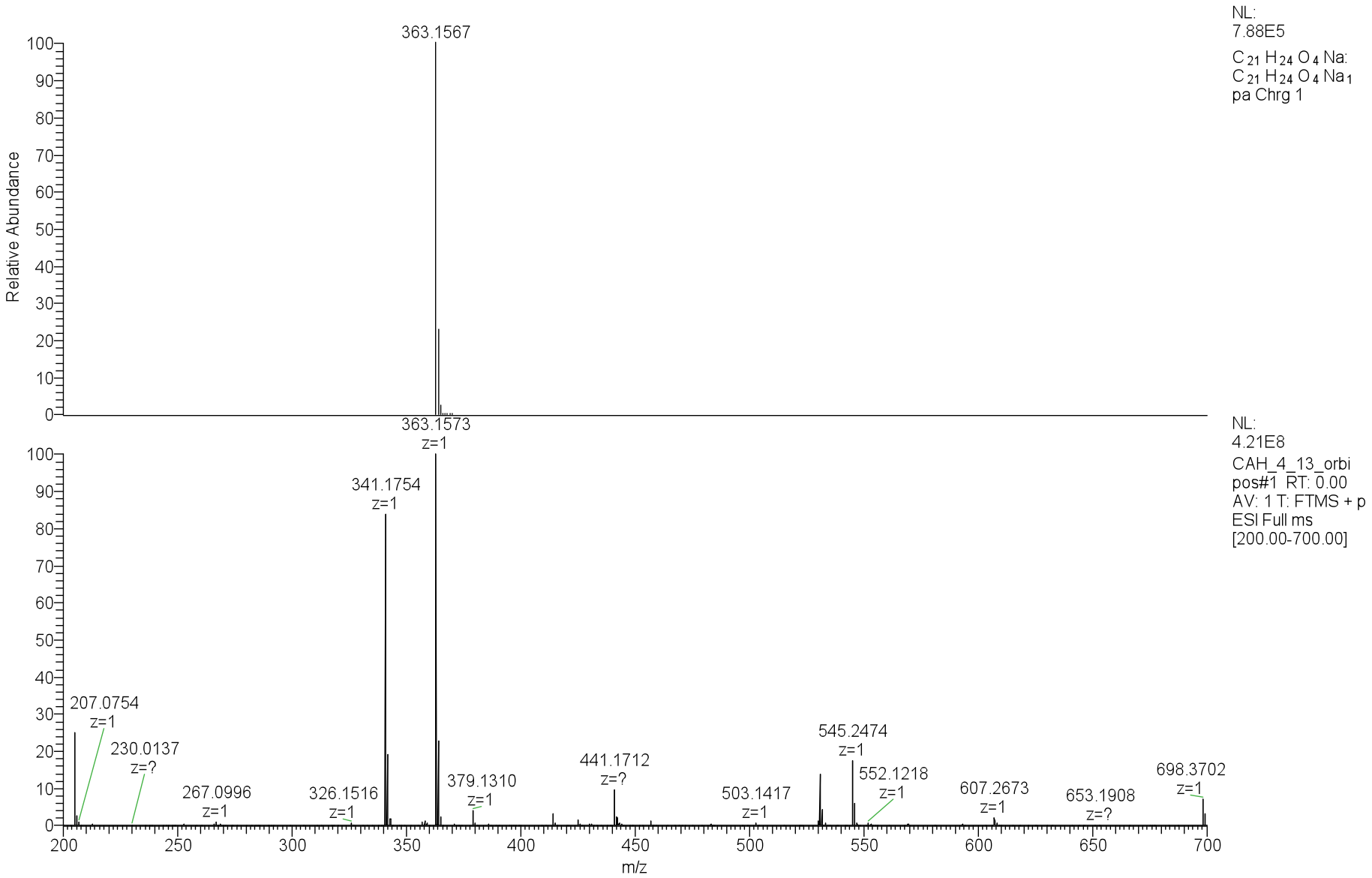
Signal 5: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

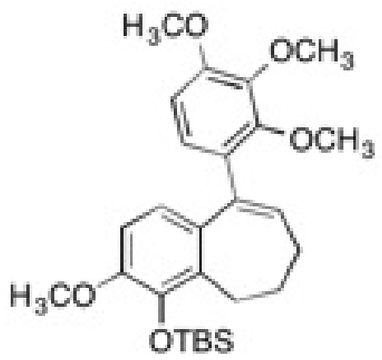
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.238	BV	0.1329	1.45295e4	1785.51025	100.0000
Totals :				1.45295e4	1785.51025	

*** End of Report ***

HRMS for compound 62

CAH-4-18





63

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151.717
148.475
142.391
140.294
135.813
132.450
131.140
128.164
124.942
120.653
107.933
106.614
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60.337
55.936
54.624
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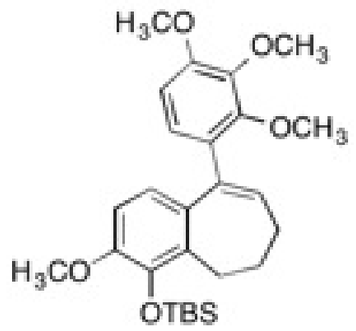
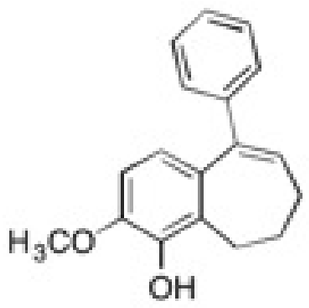
26.206
25.515
24.163
19.005
-3.907

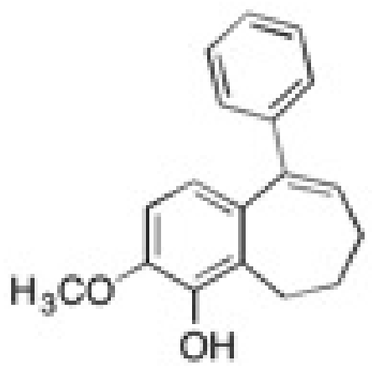


63

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



64



64

144.995
142.764
142.663
142.362
134.584
128.022
127.998
127.778
127.649
126.851
120.614
107.663
55.944
33.547
25.729
23.497

S168

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_77000003.D
Sample Name: TG-1-77

HPLC for compound 64

```
==========================================================================
Acq. Operator   : Christine
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 12/30/2013 10:46:04 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 12/30/2013 10:42:27 AM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\TG_1_77000003.D\DA.M (MASTERMETHOD.M)
Last changed    : 5/19/2014 12:27:06 PM by Christine
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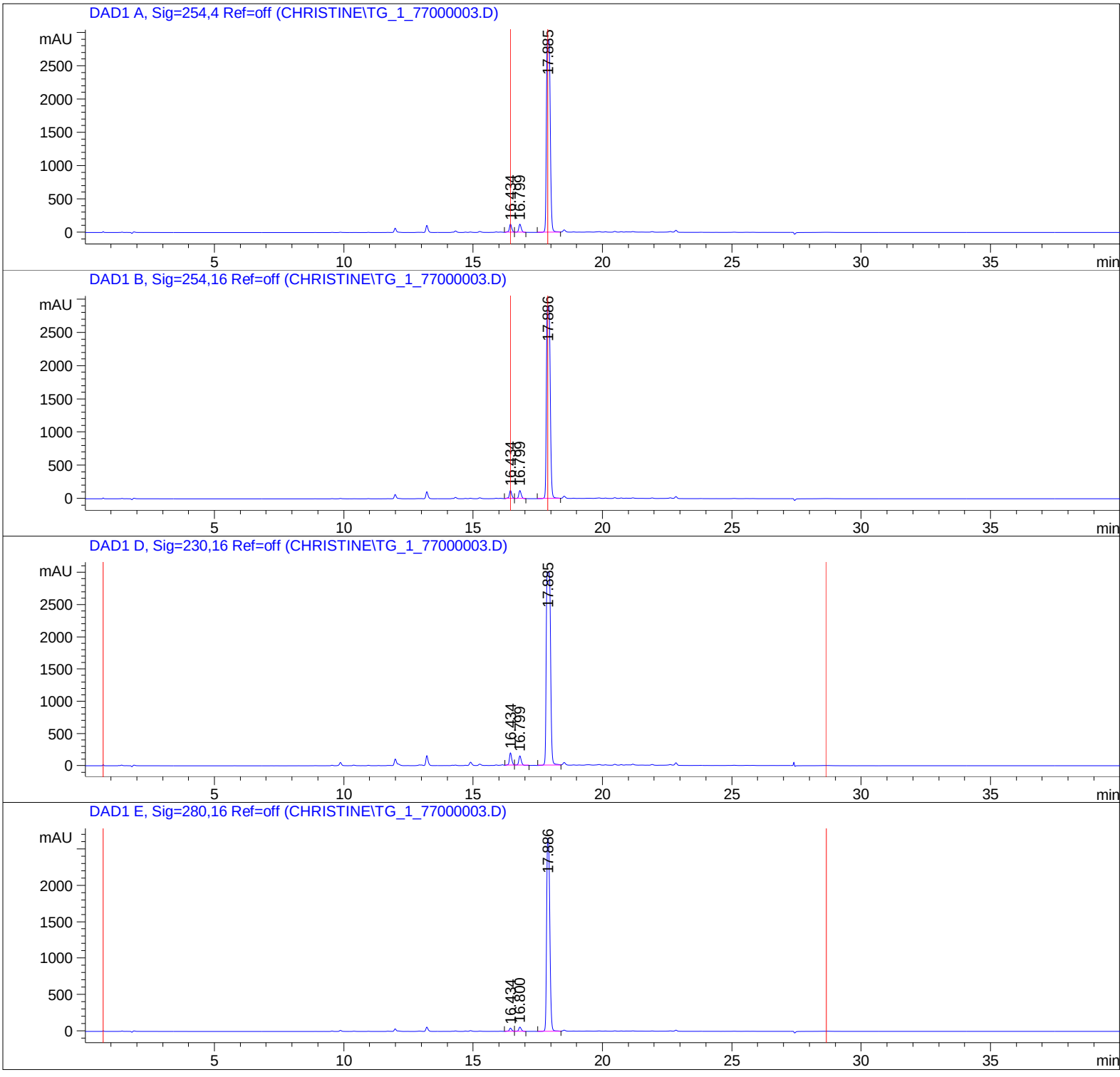
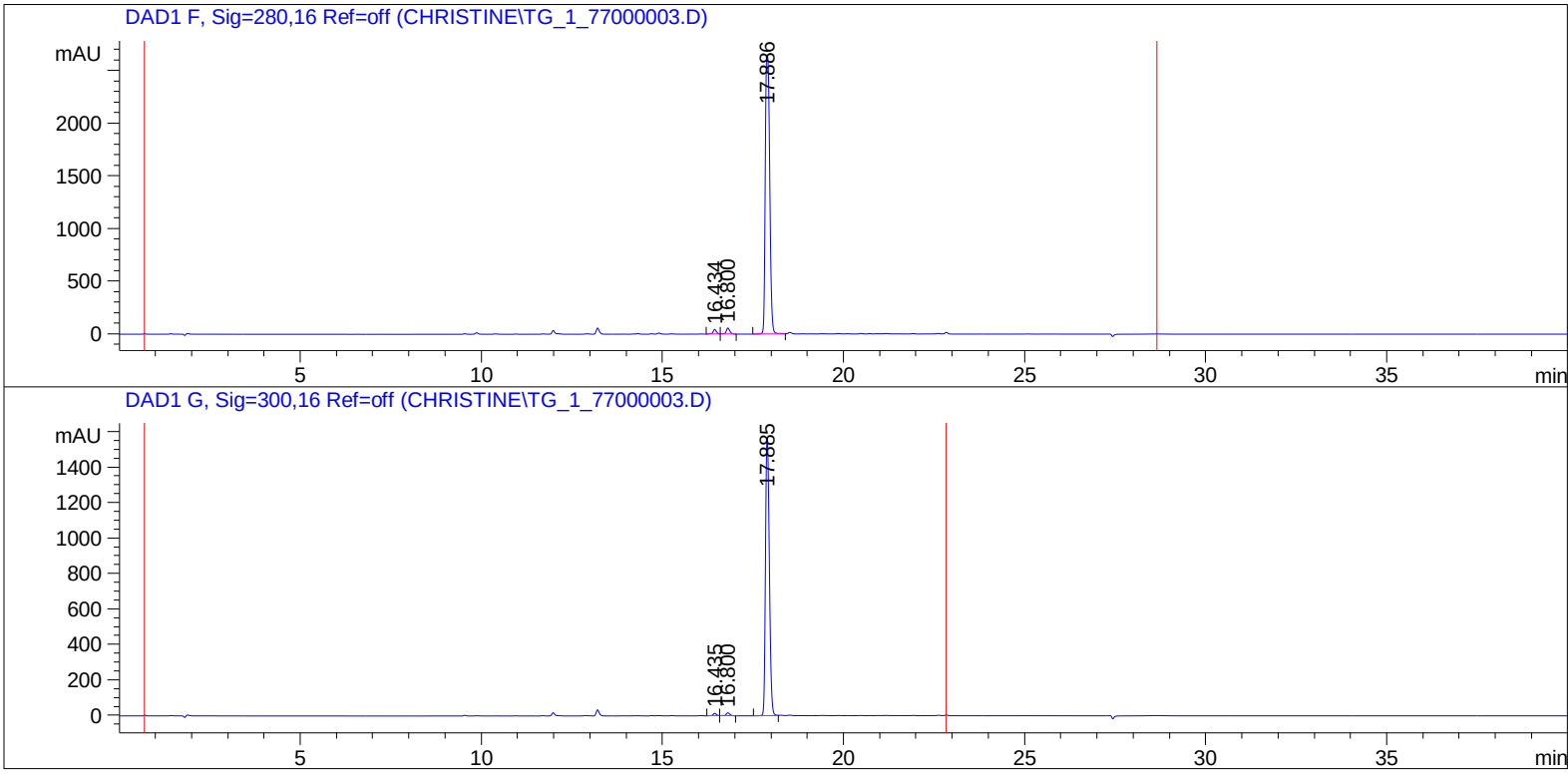


S169

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_77000003.D
Sample Name: TG-1-77



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.434	BV	0.1007	842.14648	124.77140	2.7087
2	16.799	VB	0.1029	830.52429	122.81729	2.6713
3	17.885	BV	0.1644	2.94180e4	2897.86890	94.6200
Totals :				3.10907e4	3145.45759	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.434	BV	0.1008	833.83966	123.42804	2.6899
2	16.799	VB	0.1030	826.67096	122.01691	2.6668

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_77000003.D
Sample Name: TG-1-77

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
3	17.886	BV	0.1638	2.93381e4	2905.61011	94.6433
Totals :				3.09986e4	3151.05506	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.434	BV	0.0987	1284.22107	195.25409	3.5227
2	16.799	VB	0.1065	1051.25366	148.74681	2.8837
3	17.885	BV	0.1846	3.41199e4	3002.46069	93.5936
Totals :				3.64554e4	3346.46159	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.434	BV	0.1028	309.91739	44.74255	1.3397
2	16.800	VB	0.1064	410.00235	58.08295	1.7723
3	17.886	BV	0.1366	2.24138e4	2651.25903	96.8880
Totals :				2.31337e4	2754.08453	

Signal 5: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.434	BV	0.1028	309.91739	44.74255	1.3397
2	16.800	VB	0.1064	410.00235	58.08295	1.7723
3	17.886	BV	0.1366	2.24138e4	2651.25903	96.8880
Totals :				2.31337e4	2754.08453	

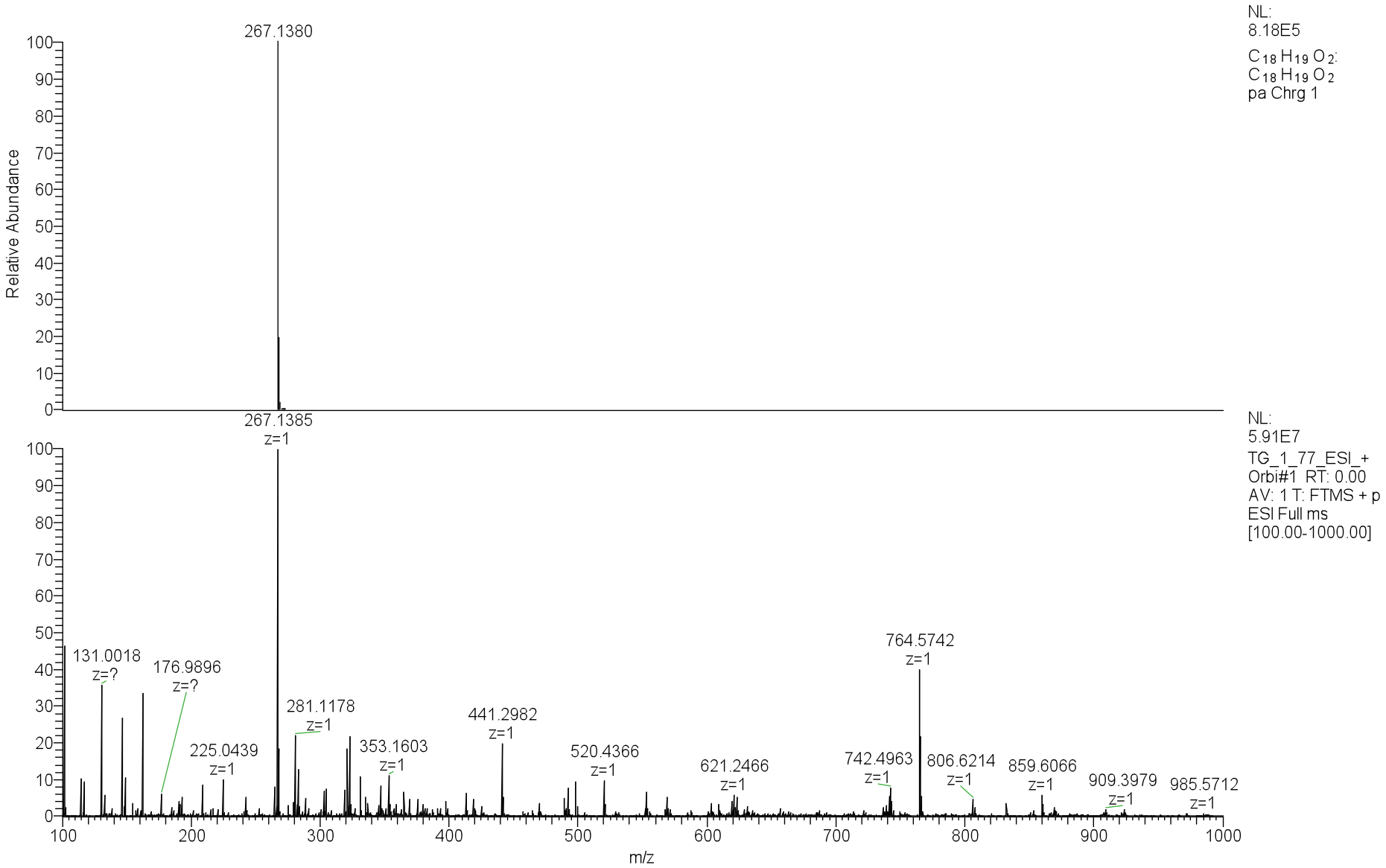
Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_77000003.D
Sample Name: TG-1-77

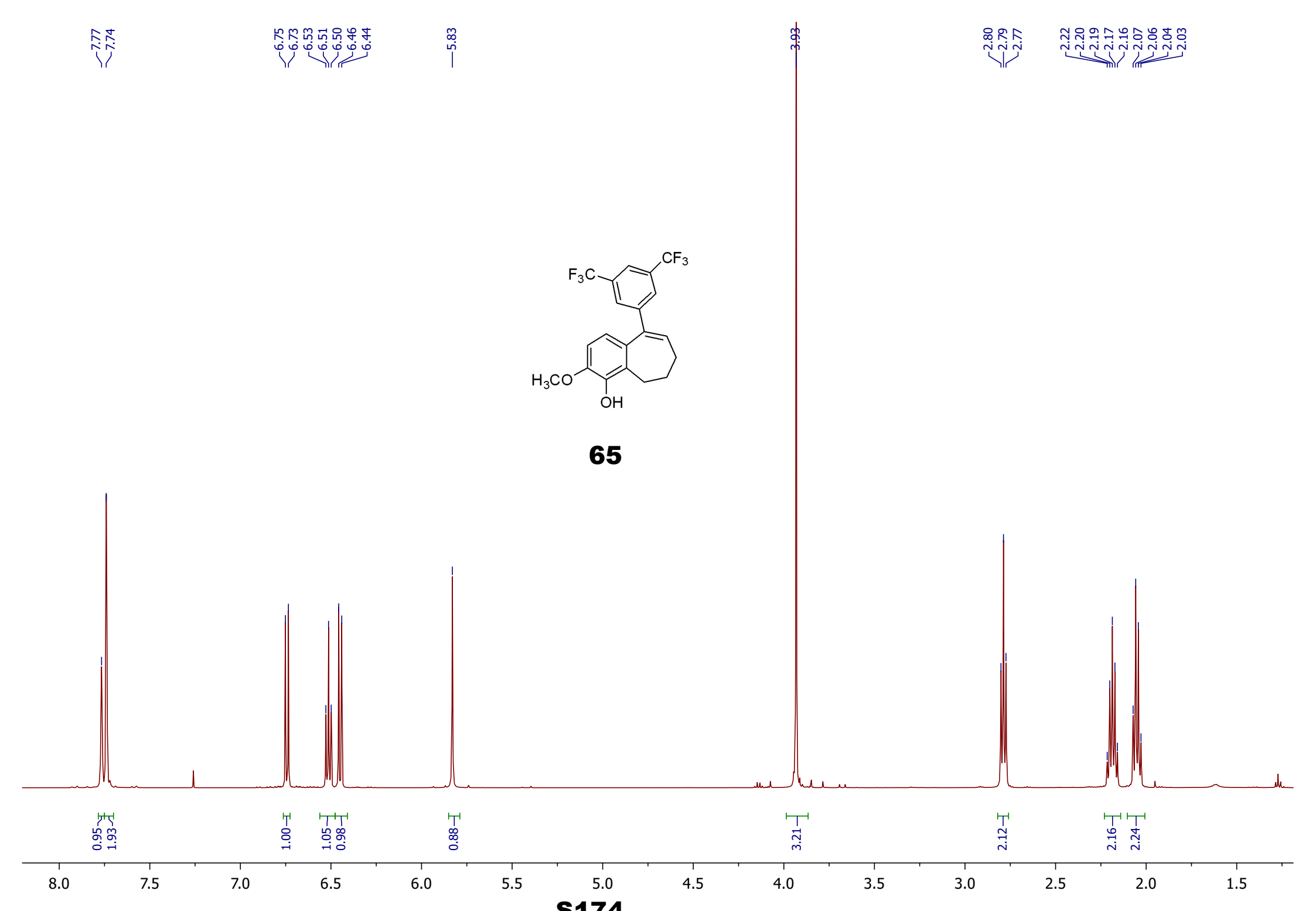
Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.435	VV	0.1028	98.29136	14.19804	0.8305
2	16.800	VB	0.1115	128.30803	17.11167	1.0841
3	17.885	BV	0.1162	1.16091e4	1572.00085	98.0855
Totals :				1.18357e4	1603.31057	

*** End of Report ***

HRMS for compound 64

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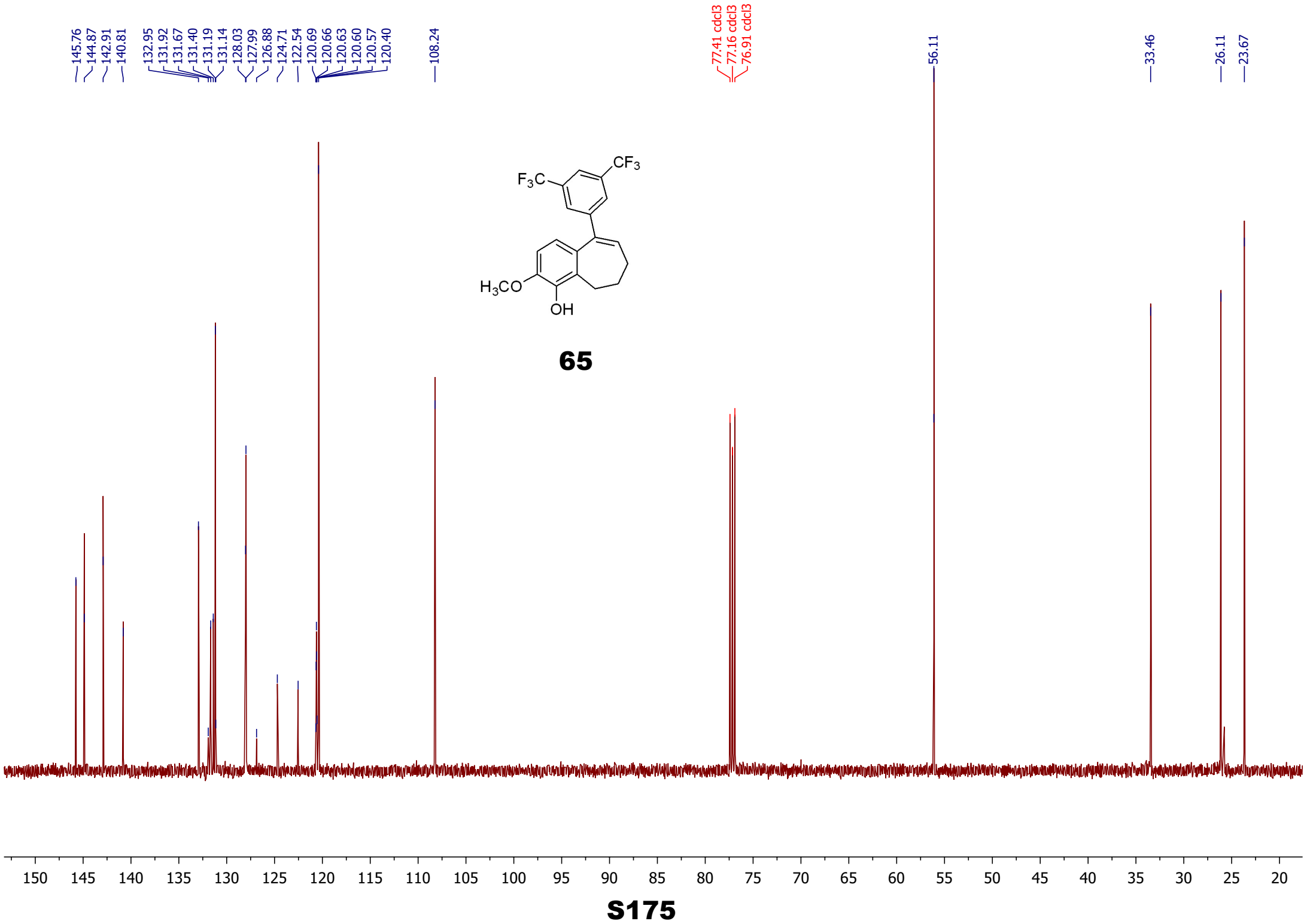
6.75
6.73
6.53
6.51
6.50
6.46
6.44
5.83
3.93
2.80
2.79
2.77
2.22
2.20
2.19
2.17
2.16
2.07
2.06
2.04
2.03
F3C
CF3
H3CO
OH
0.95
1.93
1.00
1.05
0.98
0.88
3.21
2.12
2.16
2.24
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5

65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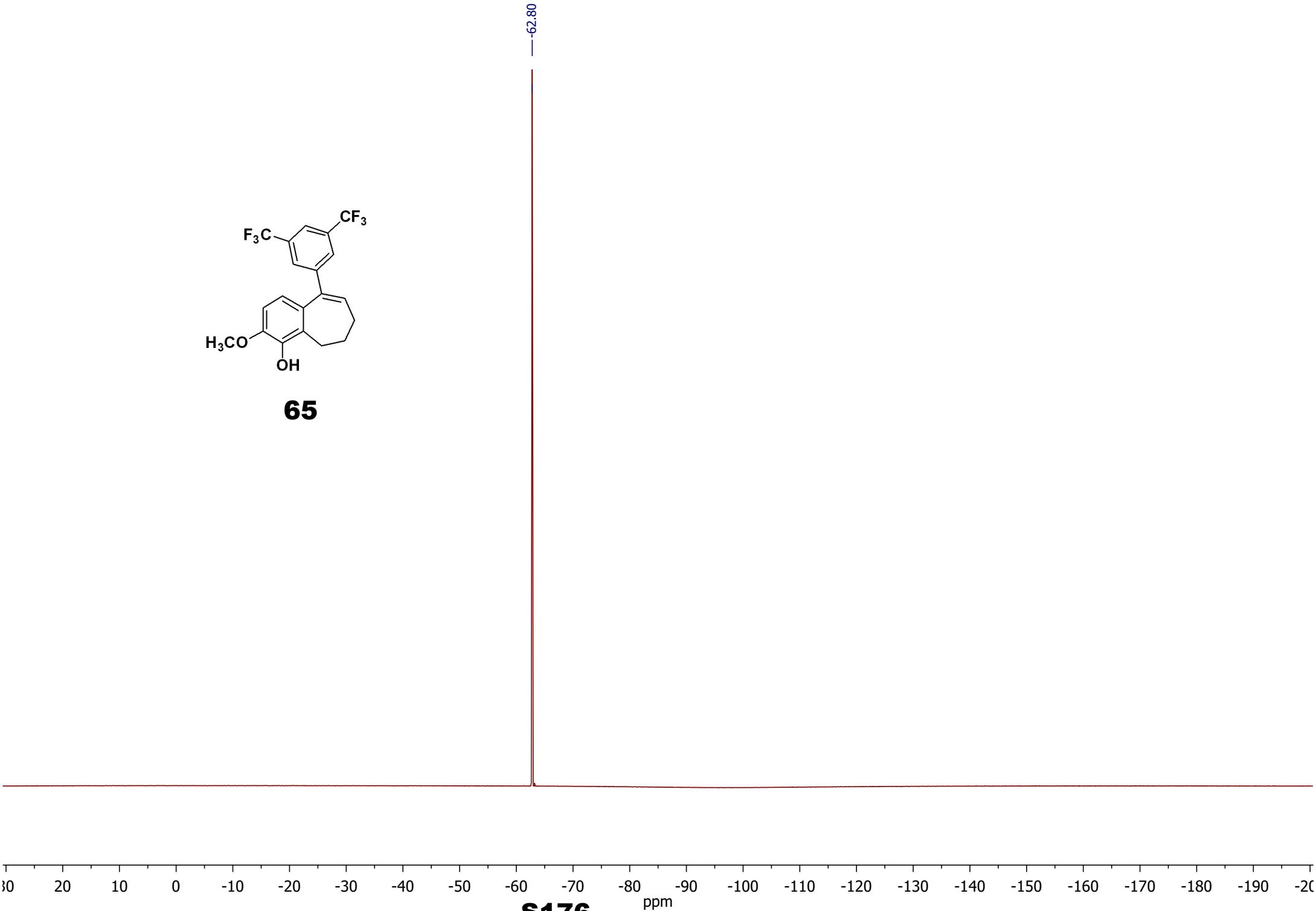
144.87
142.91
140.81
132.95
131.92
131.67
131.40
131.19
131.14
128.03
127.99
126.88
124.71
122.54
120.69
120.66
120.63
120.60
120.57
120.40
108.24
77.41 cdcl3
77.16 cdcl3
76.91 cdcl3
56.11
33.46
26.11
23.67
F3C
CF3
H3CO
OH
65
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20

F3C
CF3
H3CO
OH

65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30
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10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-20
ppm

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1

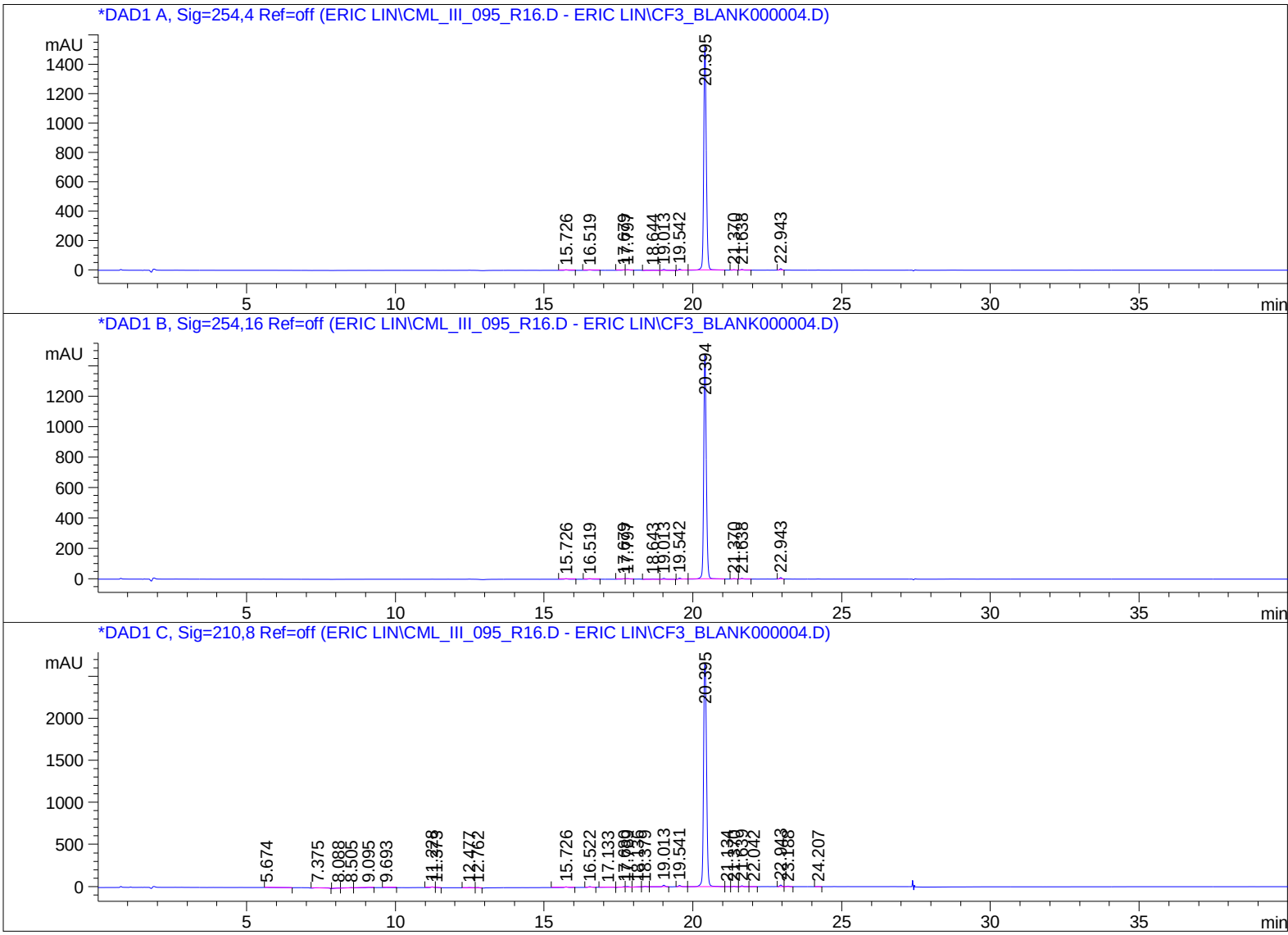
HPLC for compound 65

==========

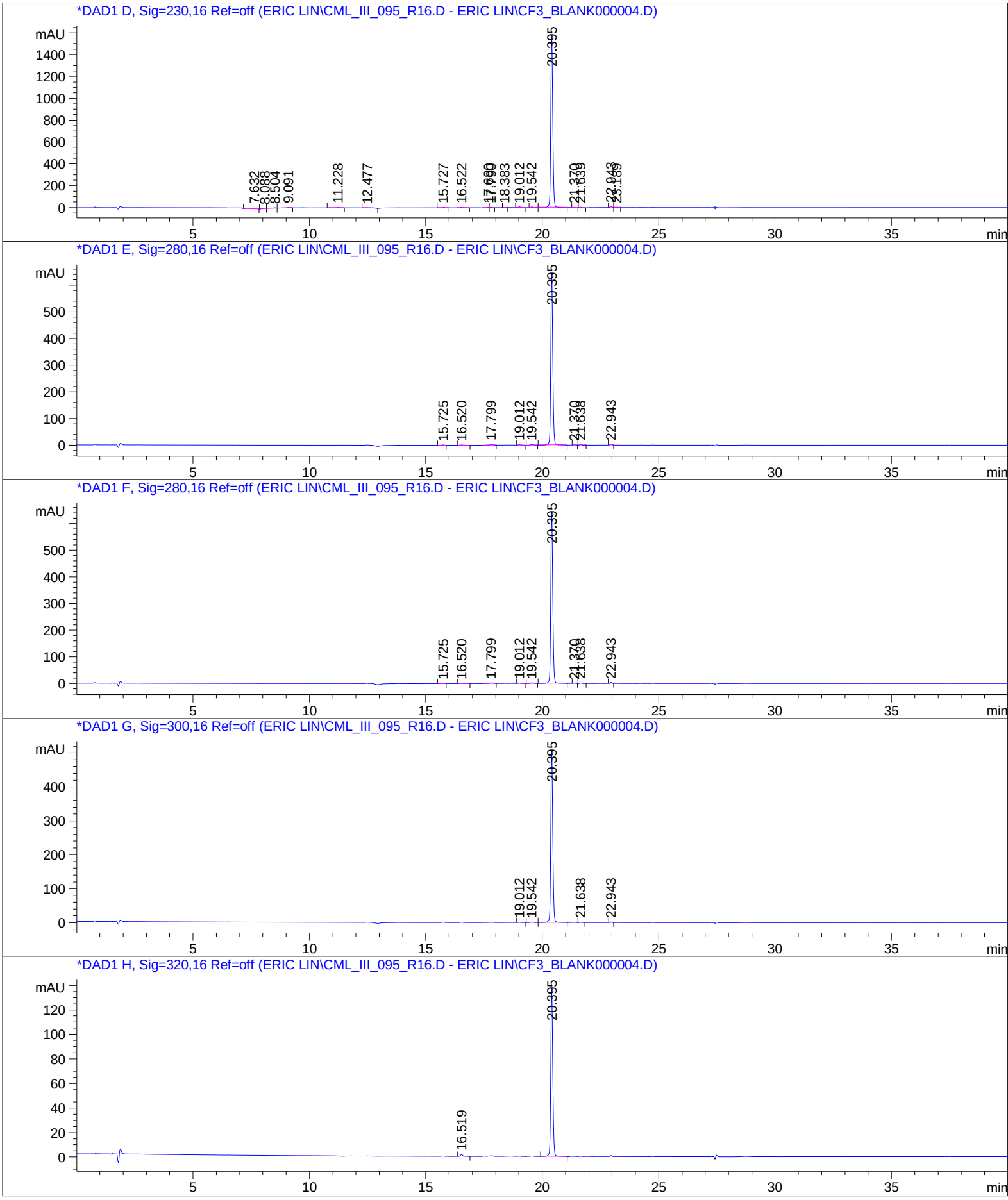
Acq. Operator : Eric Lin
Acq. Instrument : Instrument 1 Location : -
Injection Date : 3/11/2014 2:51:58 PM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 3/11/2014 2:48:52 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D\DA.M (MASTERMETHOD.M)
Last changed : 3/11/2014 3:54:40 PM by Eric Lin
Sample Info : wash

Method:

0-25 min. (50:50 to 100:0) ACN:Water
25-30 min. (100:0) ACN:Water
30-35 min. (100:0 to 50:50) ACN:Water
35-40 min. (50:50) ACN:Water



Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1



Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1

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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.726	BB	0.0982	20.13693	3.08261	0.2106
2	16.519	BB	0.1184	21.30018	2.68834	0.2227
3	17.679	BV	0.1106	17.70028	2.27860	0.1851
4	17.797	VB	0.1132	25.49945	3.33327	0.2667
5	18.644	BV	0.3221	61.30715	2.46542	0.6411
6	19.013	VB	0.1513	70.46623	6.35964	0.7369
7	19.542	BB	0.1120	57.89999	7.50579	0.6055
8	20.395	BB	0.0922	9198.03418	1528.99988	96.1876
9	21.370	BB	0.0791	13.52444	2.66984	0.1414
10	21.638	BB	0.0835	26.06196	4.78922	0.2725
11	22.943	VV	0.0783	50.66928	10.13774	0.5299

Totals : 9562.60008 1574.31034

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.726	BB	0.0983	19.38648	2.96598	0.2103
2	16.519	BB	0.1190	20.82996	2.61210	0.2260
3	17.679	BV	0.1112	17.47881	2.23609	0.1896
4	17.797	VB	0.1132	25.27899	3.30642	0.2742
5	18.643	BV	0.3208	56.03249	2.24808	0.6078
6	19.013	VB	0.1471	65.22282	6.08367	0.7075
7	19.542	BB	0.1113	54.82693	7.16507	0.5947
8	20.394	BB	0.0921	8872.07324	1475.70349	96.2390
9	21.370	BB	0.0791	12.99658	2.56620	0.1410
10	21.638	BB	0.0836	25.24567	4.63317	0.2739
11	22.943	VV	0.0783	49.41745	9.88459	0.5361

Totals : 9218.78941 1519.40486

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.674	VB	0.3926	44.06831	1.38739	0.2177
2	7.375	BB	0.4786	214.54793	5.86683	1.0599
3	8.088	BV	0.1815	70.84110	6.00896	0.3500
4	8.505	VV	0.2886	131.47398	5.84208	0.6495
5	9.095	VB	0.3278	81.57391	3.28358	0.4030
6	9.693	BB	0.2099	17.39349	1.11562	0.0859
7	11.228	BV	0.1006	57.96552	8.60018	0.2864
8	11.373	VB	0.0894	14.11614	2.37216	0.0697
9	12.477	BV	0.1629	46.31915	3.78189	0.2288
10	12.762	VB	0.1005	34.67432	5.15360	0.1713
11	15.726	BB	0.1044	51.22066	7.24781	0.2530
12	16.522	BB	0.1089	55.50475	7.80679	0.2742
13	17.133	BB	0.2957	60.25353	2.56871	0.2977
14	17.680	BV	0.1381	80.84849	7.85592	0.3994
15	17.789	VV	0.1288	75.16172	8.37082	0.3713
16	18.136	VB	0.2330	26.68663	1.61339	0.1318
17	18.379	BV	0.1083	47.17024	6.52840	0.2330
18	19.013	VB	0.1081	139.61841	18.91397	0.6897
19	19.541	VB	0.0978	95.36326	14.67350	0.4711
20	20.395	BV	0.1118	1.86236e4	2657.28906	92.0020
21	21.134	VV	0.1510	20.76570	2.03656	0.1026
22	21.370	VB	0.0898	37.02381	6.19101	0.1829
23	21.639	BB	0.0880	63.57571	10.91086	0.3141
24	22.042	BB	0.1008	7.01408	1.01333	0.0347
25	22.943	VV	0.0789	94.95434	18.80930	0.4691
26	23.188	VB	0.1056	43.21199	6.03252	0.2135
27	24.207	BB	0.1019	7.66240	1.17747	0.0379
Totals :				2.02426e4	2822.45171	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.632	BB	0.3457	244.82117	8.66334	2.3247
2	8.088	BV	0.1865	83.24166	6.91353	0.7904
3	8.504	VV	0.2903	155.94417	6.88412	1.4808
4	9.091	VB	0.3319	95.37034	3.78608	0.9056
5	11.228	BB	0.0986	26.37656	4.01844	0.2505
6	12.477	BB	0.3337	77.63901	2.88735	0.7372
7	15.727	BB	0.1022	20.69559	3.00905	0.1965
8	16.522	BB	0.1230	29.73312	3.57454	0.2823

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
9	17.680	BV	0.1323	34.43248	3.57857	0.3270
10	17.790	VB	0.1142	25.67534	3.39784	0.2438
11	18.383	BB	0.1008	12.53002	1.95390	0.1190
12	19.012	BB	0.0930	42.09318	6.91282	0.3997
13	19.542	VB	0.1016	41.41371	6.06741	0.3932
14	20.395	BB	0.0922	9513.58496	1581.17224	90.3374
15	21.370	BB	0.0906	17.37347	2.87103	0.1650
16	21.639	BB	0.1049	40.14706	5.51546	0.3812
17	22.943	VV	0.0787	58.11080	11.55836	0.5518
18	23.189	VB	0.1223	11.98790	1.39506	0.1138
Totals :				1.05312e4	1664.15915	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.725	BB	0.0922	7.46464	1.24071	0.1873
2	16.520	BB	0.1243	9.99923	1.18650	0.2509
3	17.799	BB	0.1403	28.14871	2.87234	0.7064
4	19.012	BB	0.1053	13.62076	1.90712	0.3418
5	19.542	BB	0.1191	24.31960	2.92321	0.6103
6	20.395	BB	0.0920	3864.91748	643.91962	96.9896
7	21.370	BB	0.0796	5.79338	1.13348	0.1454
8	21.638	BB	0.0838	11.43022	2.08863	0.2868
9	22.943	VB	0.0768	19.18251	3.93981	0.4814
Totals :				3984.87653	661.21142	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.725	BB	0.0922	7.46464	1.24071	0.1873
2	16.520	BB	0.1243	9.99923	1.18650	0.2509
3	17.799	BB	0.1403	28.14871	2.87234	0.7064
4	19.012	BB	0.1053	13.62076	1.90712	0.3418
5	19.542	BB	0.1191	24.31960	2.92321	0.6103
6	20.395	BB	0.0920	3864.91748	643.91962	96.9896
7	21.370	BB	0.0796	5.79338	1.13348	0.1454
8	21.638	BB	0.0838	11.43022	2.08863	0.2868
9	22.943	VB	0.0768	19.18251	3.93981	0.4814

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_095_R16.D
Sample Name: CML_III_095_r1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				3984.87653	661.21142	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.012	BB	0.1139	8.24947	1.04762	0.2664
2	19.542	BB	0.1270	16.53314	1.83632	0.5339
3	20.395	BB	0.0921	3057.46191	508.62131	98.7265
4	21.638	BB	0.0804	6.04191	1.16629	0.1951
5	22.943	BB	0.0767	8.61605	1.77379	0.2782
Totals :				3096.90248	514.44532	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.519	BB	0.1121	10.13603	1.37369	1.2035
2	20.395	BB	0.0928	832.06708	137.10928	98.7965
Totals :				842.20311	138.48297	

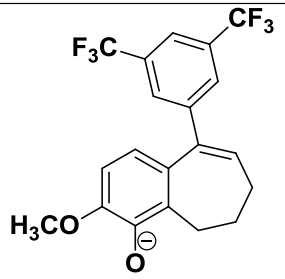
==========

*** End of Report ***

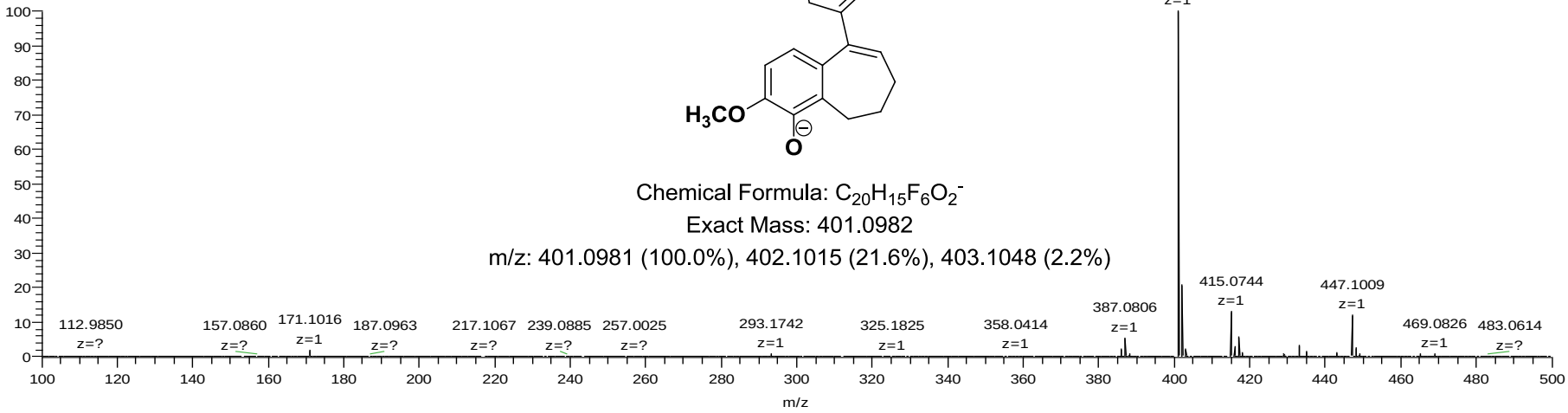
HRMS for compound 65

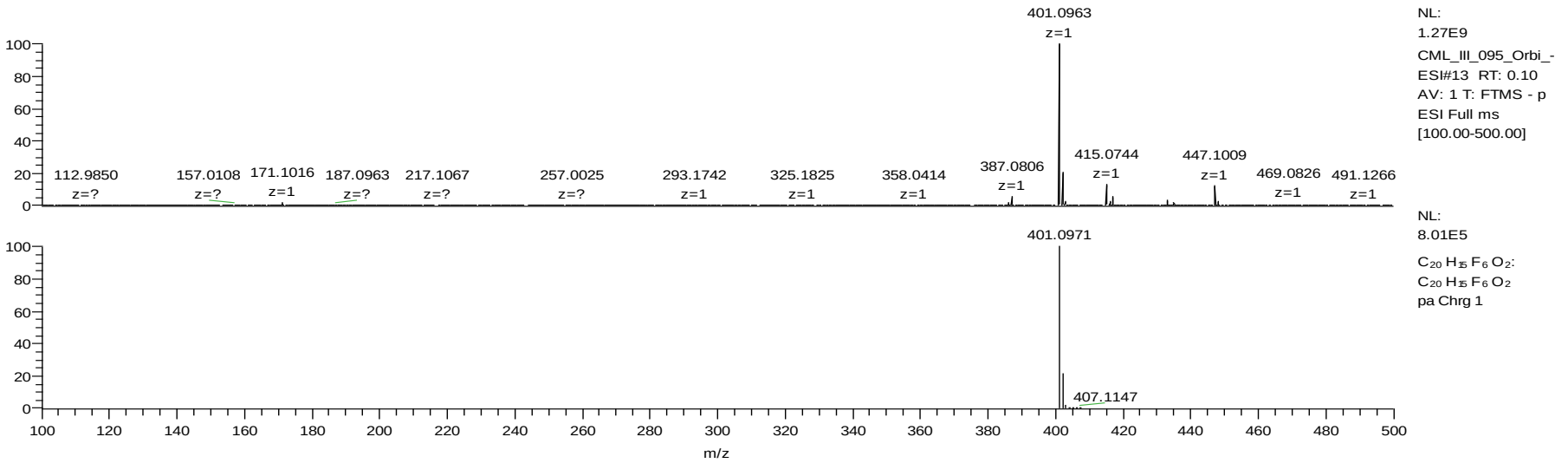
C:\Xcalibur\...\CML_III_095_Orbi_-ESI 3/17/2014 2:52:06 PM CML_III_095

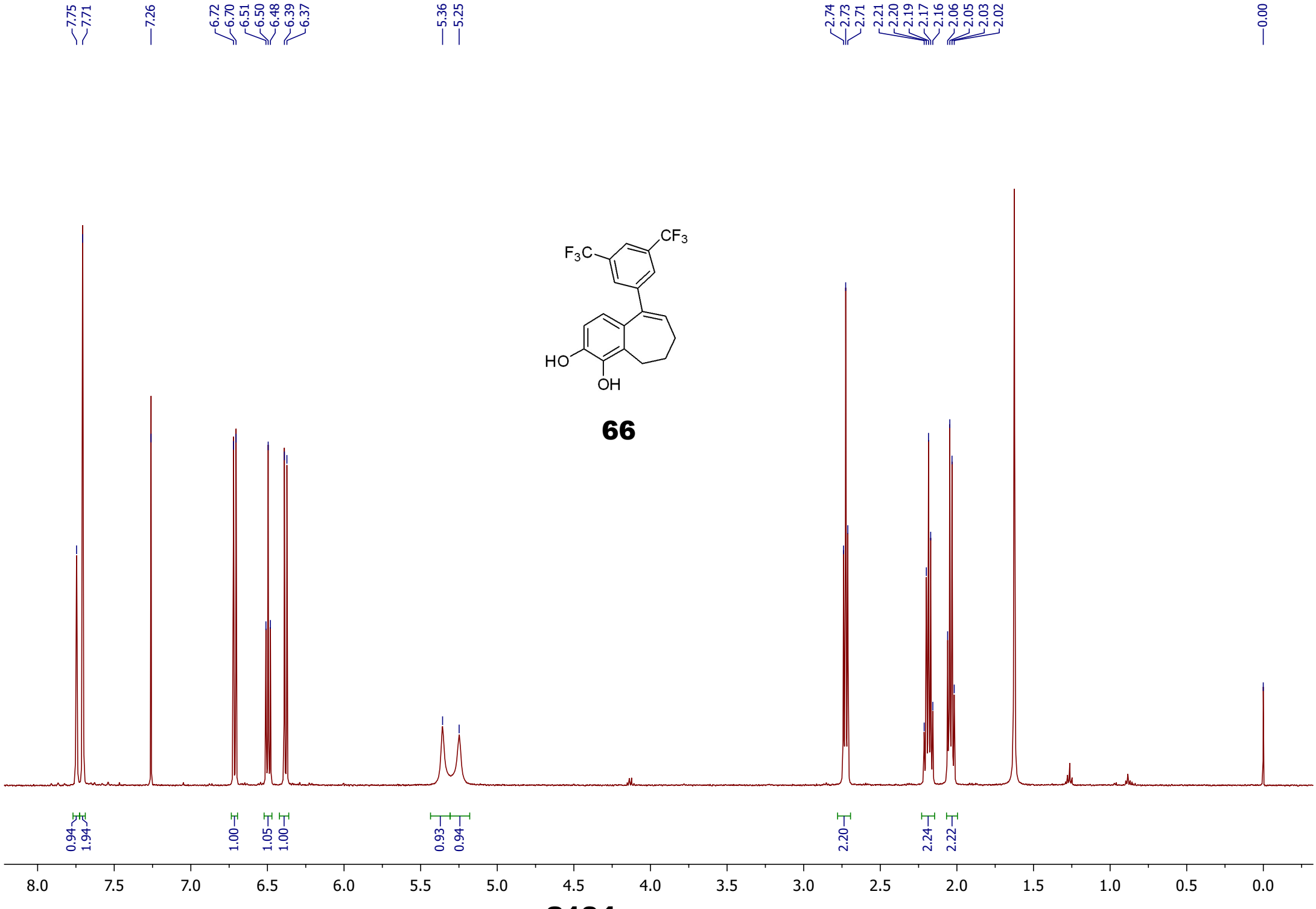
CML_III_095_Orbi_-ESI #13 RT: 0.10 AV: 1 NL:
T: FTMS - p ESI Full ms [100.00-500.00]

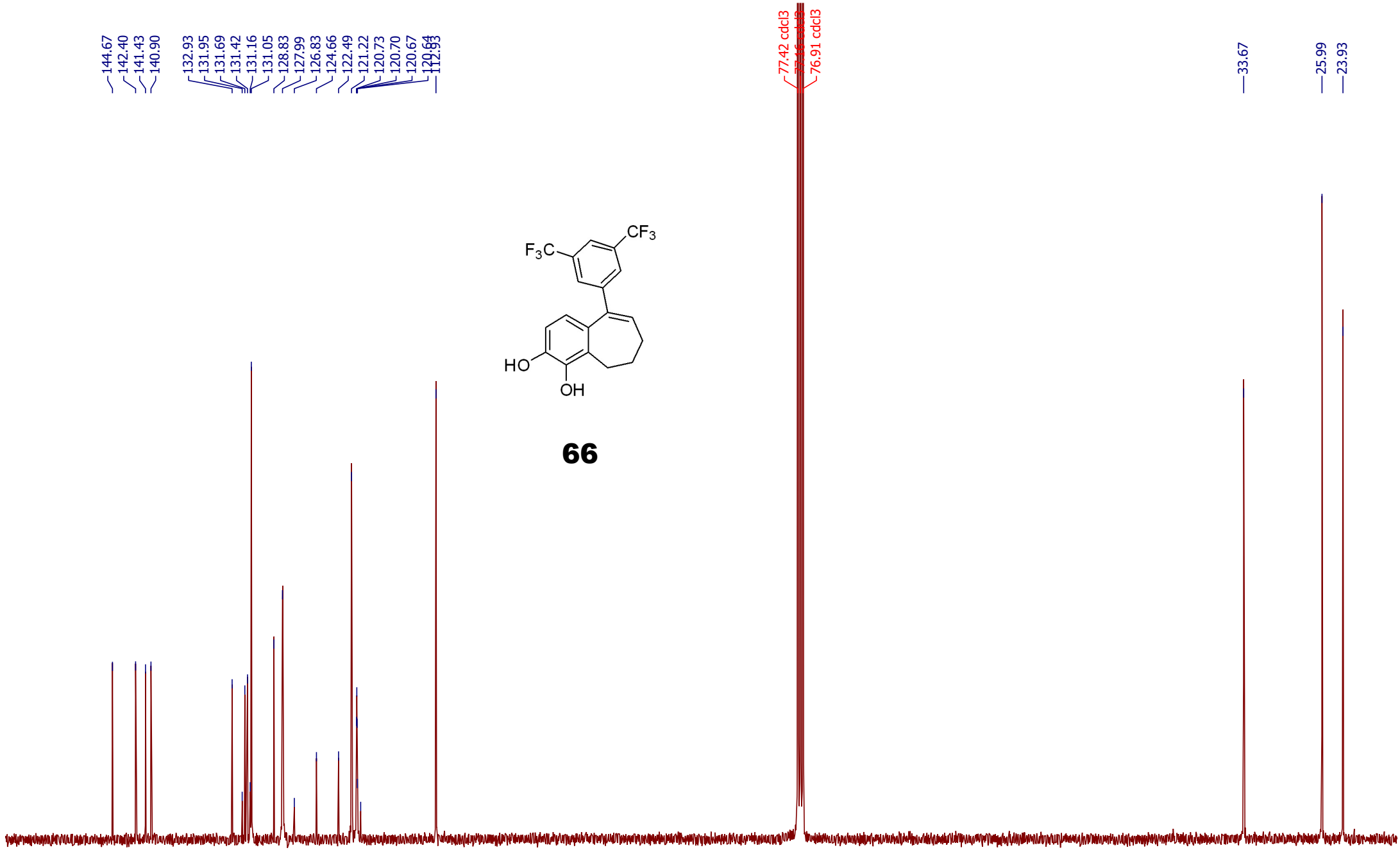


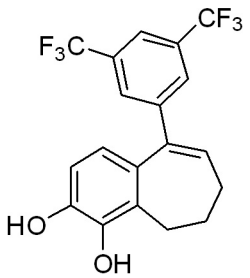
Chemical Formula: $C_{20}H_{15}F_6O_2^-$
Exact Mass: 401.0982
m/z: 401.0981 (100.0%), 402.1015 (21.6%), 403.1048 (2.2%)






7.75
7.71
7.26
6.72
6.70
6.51
6.50
6.48
6.39
6.37
5.36
5.25
2.74
2.73
2.71
2.21
2.20
2.19
2.17
2.16
2.06
2.05
2.03
2.02
0.00
F3C
CF3
HO
OH
66
0.94
1.94
1.00
1.05
1.00
0.93
0.94
2.20
2.24
2.22
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


144.67
142.40
141.43
140.90
132.93
131.95
131.69
131.42
131.16
131.05
128.83
127.99
126.83
124.66
122.49
121.22
120.73
120.70
120.67
77.42 cdcl3
76.91 cdcl3
33.67
25.99
23.93
F3C
CF3
HO
OH
66
55
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20



66

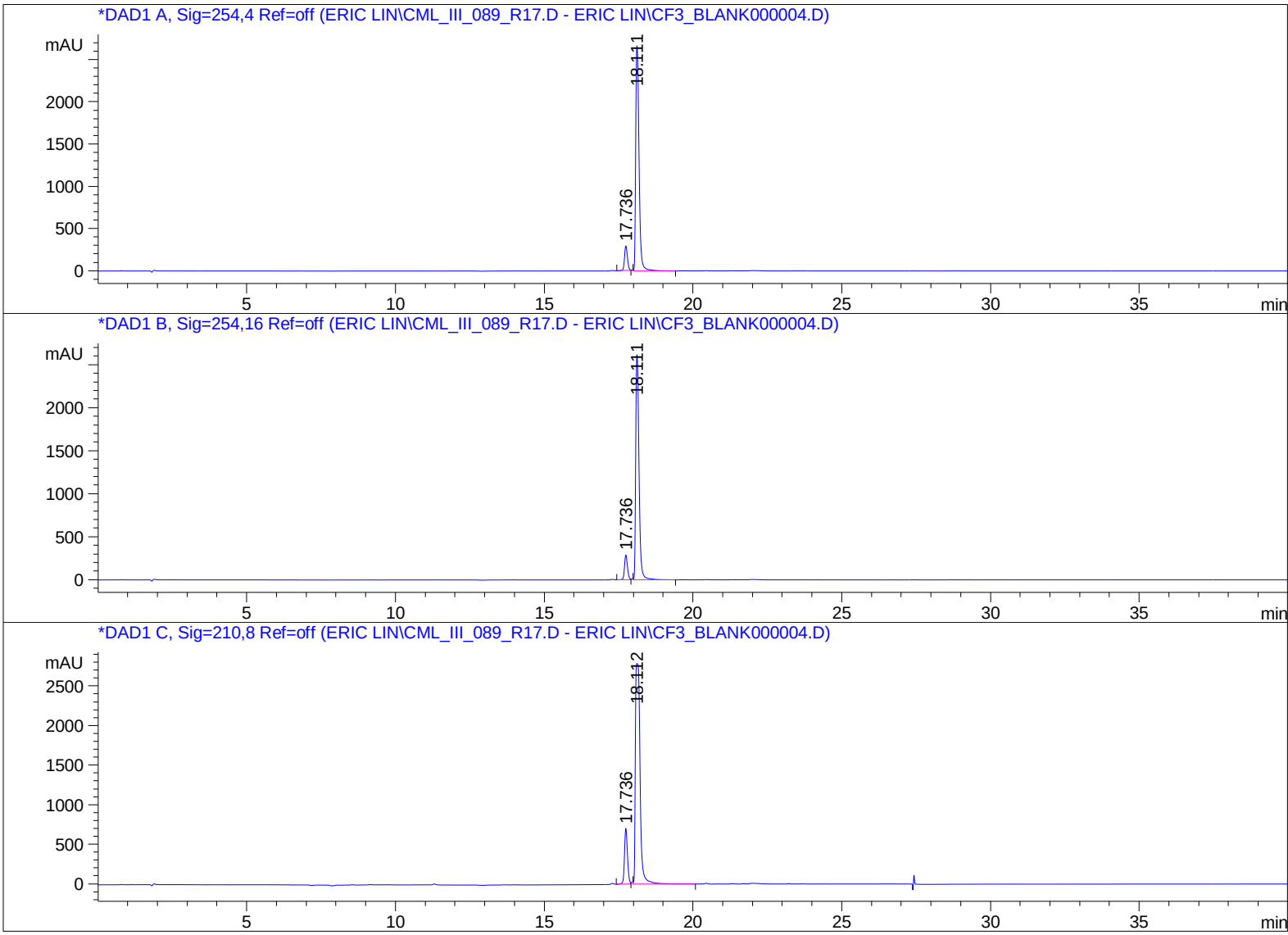
Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_089_R17.D
Sample Name: CML_III_089_r1

HPLC for compound 66

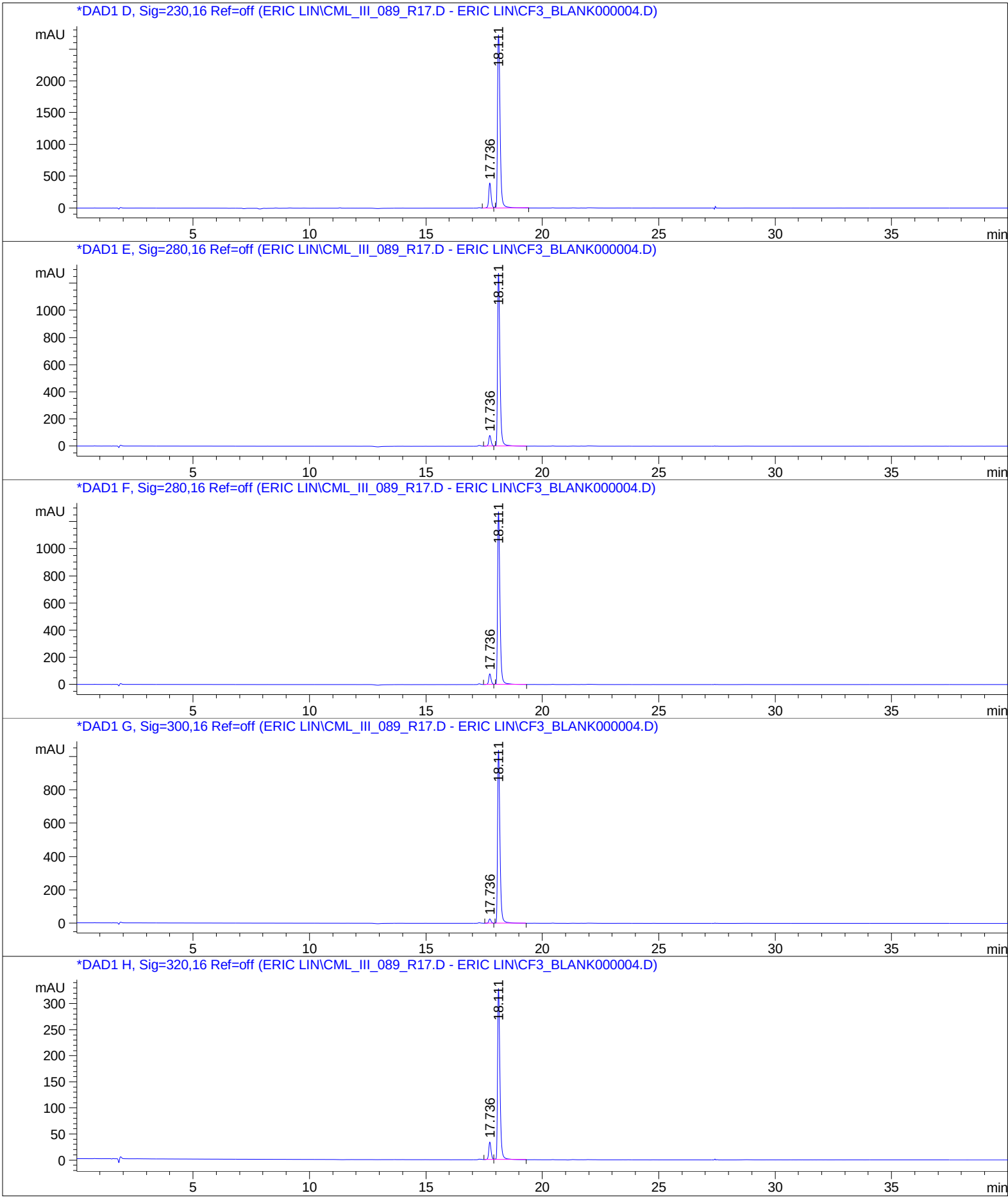
```
==========================================================================
Acq. Operator   : Eric Lin
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 3/11/2014 3:54:22 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 3/11/2014 3:42:14 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\CML_III_089_R17.D\DA.M (MASTERMETHOD.M)
Last changed    : 3/11/2014 4:44:37 PM by Eric Lin
Sample Info     : wash

                  Method:

                  0-25 min. (50:50 to 100:0) ACN:Water
                  25-30 min. (100:0) ACN:Water
                  30-35 min. (100:0 to 50:50) ACN:Water
                  35-40 min. (50:50) ACN:Water
```



Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_089_R17.D
Sample Name: CML_III_089_r1



Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_089_R17.D
Sample Name: CML_III_089_r1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1074	2060.98120	295.51819	9.0361
2	18.111	VB	0.1228	2.07474e4	2666.64331	90.9639
Totals :				2.28084e4	2962.16150	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1074	2008.27014	287.96533	9.0541
2	18.111	VB	0.1198	2.01725e4	2620.97852	90.9459
Totals :				2.21808e4	2908.94385	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1083	4991.49316	707.71631	13.9556
2	18.112	VB	0.1746	3.07754e4	2790.98706	86.0444
Totals :				3.57669e4	3498.70337	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1078	2763.69995	394.30969	11.6487
2	18.111	VB	0.1197	2.09617e4	2726.13794	88.3513
Totals :				2.37254e4	3120.44763	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1070	546.02472	78.59853	5.7074
2	18.111	VB	0.1085	9020.88184	1274.97351	94.2926
Totals :				9566.90656	1353.57204	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1070	546.02472	78.59853	5.7074
2	18.111	VB	0.1085	9020.88184	1274.97351	94.2926
Totals :				9566.90656	1353.57204	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BB	0.1042	170.52390	25.43328	2.2779
2	18.111	BB	0.1082	7315.54199	1038.30994	97.7221
Totals :				7486.06589	1063.74322	

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_III_089_R17.D
Sample Name: CML_III_089_r1

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

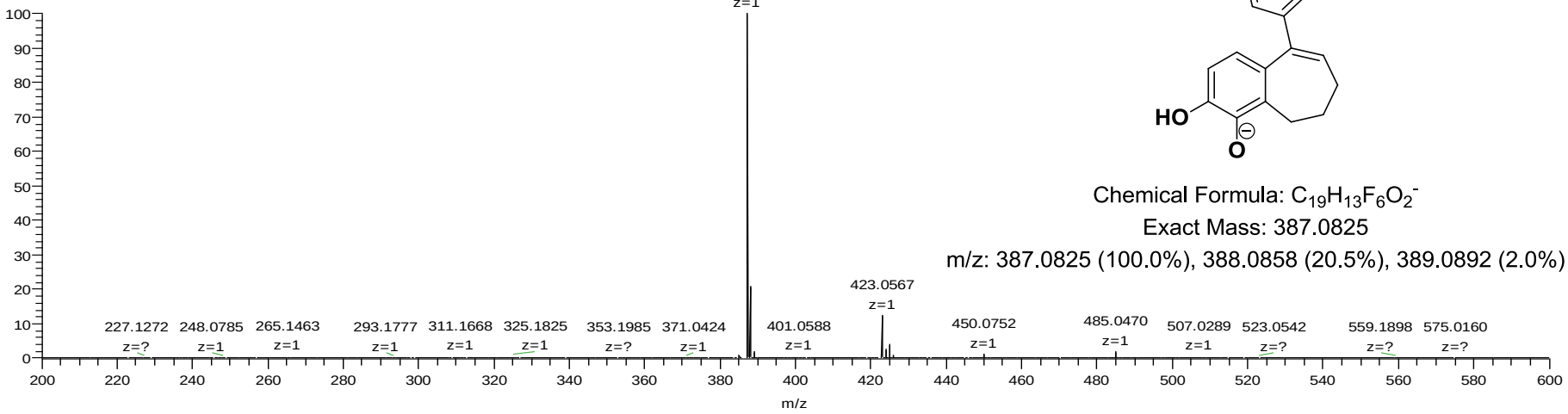
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.736	BV	0.1068	232.93462	33.65369	9.0931
2	18.111	VB	0.1086	2328.72632	328.78772	90.9069
Totals :				2561.66093	362.44141	

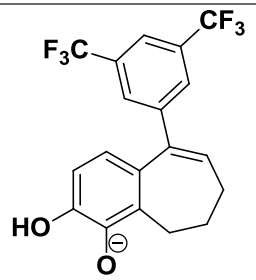
*** End of Report ***

HRMS for compound 66

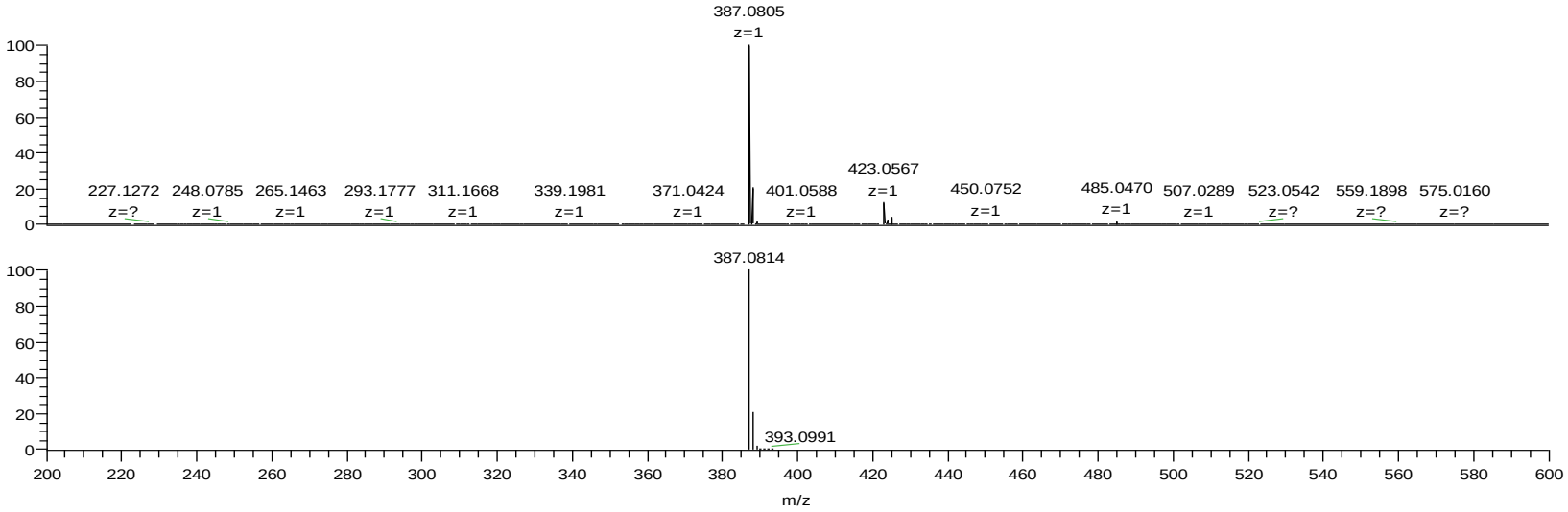
C:\Xcalibur\...\CML_III_089_Orbi_-ESI 3/17/2014 2:37:16 PM CML_III_089

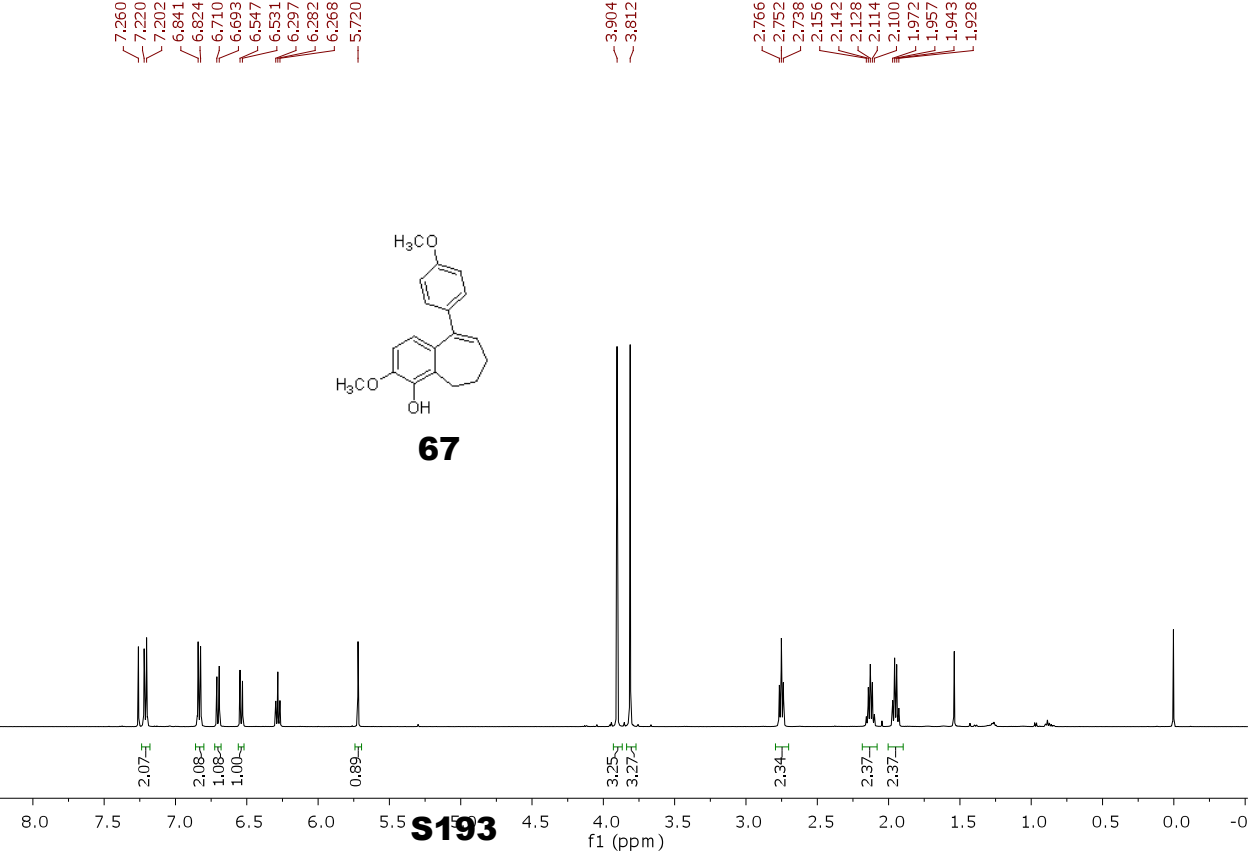
CML_III_089_Orbi_-ESI #14 RT: 0.11 AV: 1 NL:
T: FTMS - p ESI Full ms [200.00-600.00]



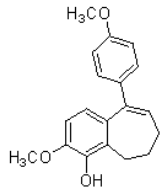


Chemical Formula: $C_{19}H_{13}F_6O_2^-$
Exact Mass: 387.0825
m/z: 387.0825 (100.0%), 388.0858 (20.5%), 389.0892 (2.0%)




7.260
7.220
7.202
6.841
6.824
6.710
6.693
6.547
6.531
6.297
6.282
6.268
5.720
3.904
3.812
2.766
2.752
2.738
2.156
2.142
2.128
2.114
2.100
1.972
1.957
1.943
1.928
H3CO
H3CO
OH
67
2.07
2.08
1.08
1.00
0.89
3.25
3.27
2.34
2.37
2.37
8.0
7.5
7.0
6.5
6.0
5.5
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

158.760
144.942
142.347
142.152
135.340
134.795
129.033
127.802
126.046
120.561
113.435
107.628
77.270
77.016
76.762
55.938
55.278
33.681
25.628
23.484



67

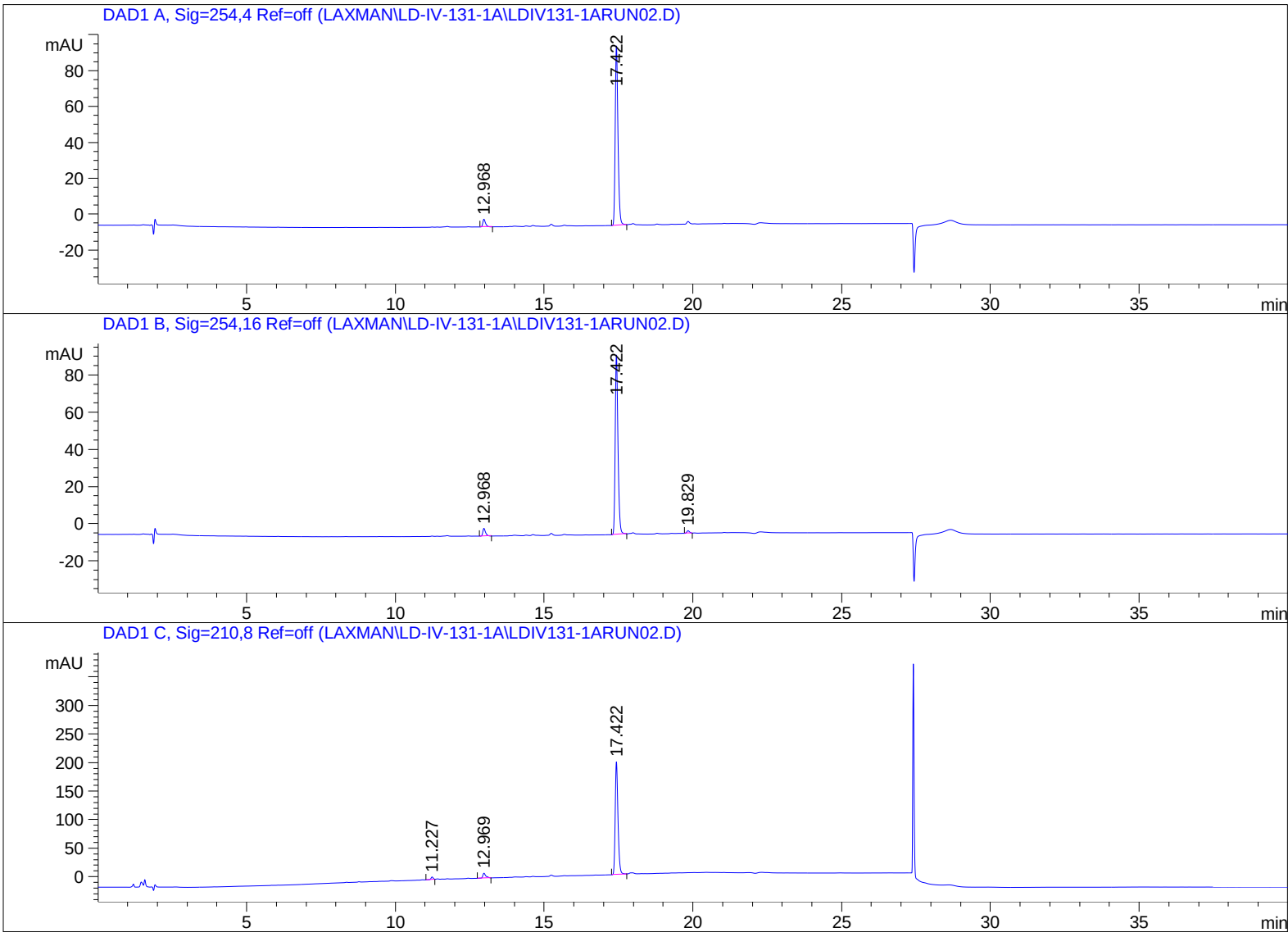
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

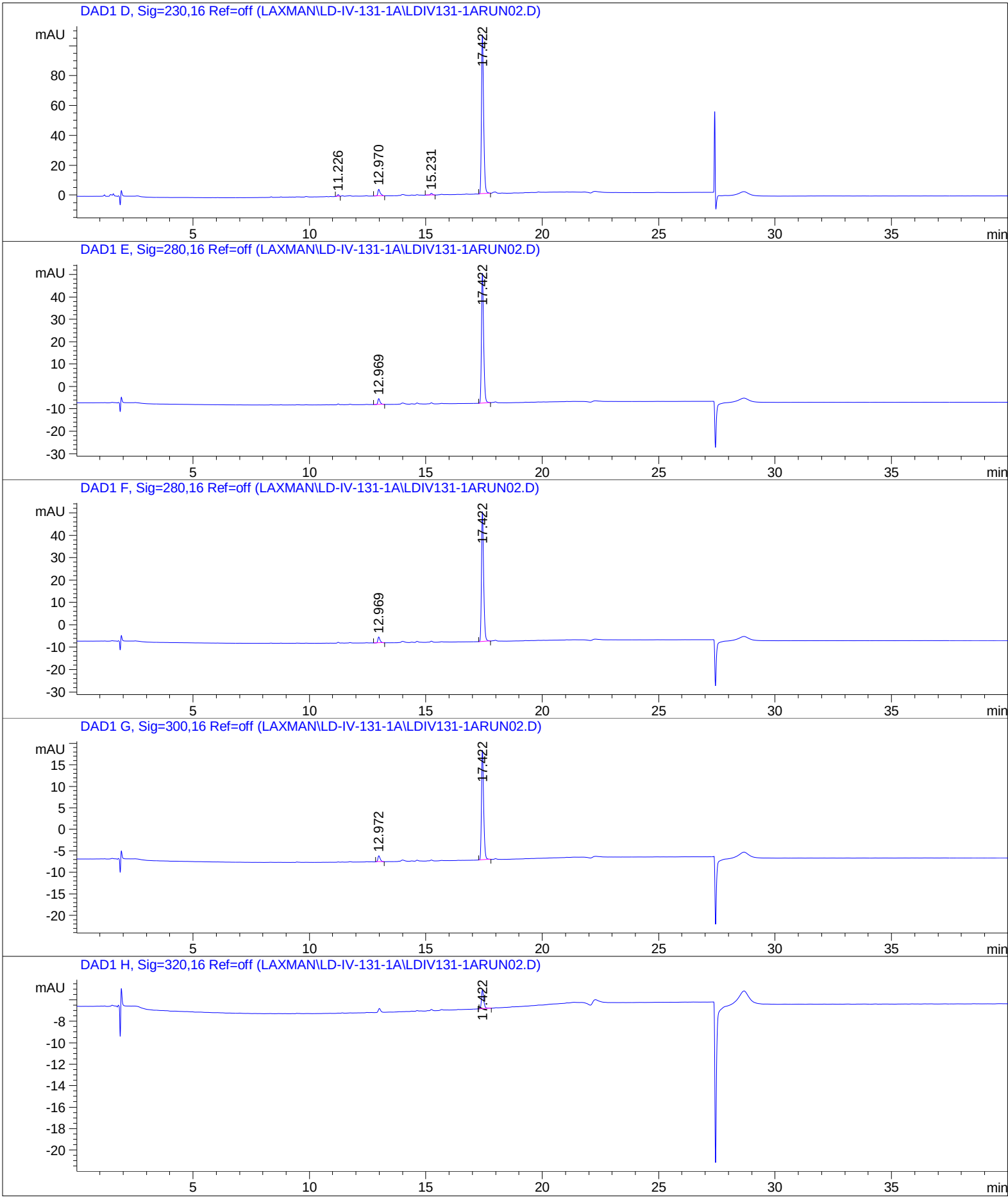
Data File C:\CHEM32\1\DATA\LAXMAN\LD-IV-131-1A\LDIV131-1ARUN02.D
Sample Name: LD-IV-131-1A-rerun-run1

HPLC for compound 67

```
==========================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/27/2014 5:37:35 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 1/27/2014 5:33:50 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-IV-131-1A\LDIV131-1ARUN02.D\DA.M (MASTERMETHOD.M)
Last changed    : 1/28/2014 10:47:18 AM by Laxman
                  (modified after loading)
Sample Info     : run1
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-IV-131-1A\LDIV131-1ARUN02.D
Sample Name: LD-IV-131-1A-rerun-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-IV-131-1A\LDIV131-1ARUN02.D
Sample Name: LD-IV-131-1A-rerun-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BB	0.0937	27.01311	4.27345	4.0000
2	17.422	BB	0.0976	648.31488	100.04857	96.0000

Totals : 675.32799 104.32202

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BB	0.0938	26.28994	4.15377	3.9796
2	17.422	BB	0.0976	626.04687	96.60343	94.7661
3	19.829	BB	0.0944	8.28639	1.33497	1.2543

Totals : 660.62320 102.09217

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.227	BV	0.0829	24.89905	4.61790	1.8119
2	12.969	BB	0.1030	61.09814	8.58504	4.4460
3	17.422	BV	0.1000	1288.22766	197.75839	93.7421

Totals : 1374.22486 210.96132

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.226	BB	0.0760	5.55864	1.15744	0.7525
2	12.970	BB	0.1029	31.45657	4.42585	4.2583
3	15.231	BB	0.1021	7.81155	1.13753	1.0575
4	17.422	BV	0.0980	693.87982	106.45760	93.9317
Totals :				738.70657	113.17843	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.969	BB	0.0958	17.15126	2.64083	4.3596
2	17.422	BB	0.0977	376.26514	58.01117	95.6404
Totals :				393.41640	60.65200	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.969	BB	0.0958	17.15126	2.64083	4.3596
2	17.422	BB	0.0977	376.26514	58.01117	95.6404
Totals :				393.41640	60.65200	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.972	BB	0.1010	9.45148	1.39534	5.3948
2	17.422	BB	0.0977	165.74431	25.52812	94.6052
Totals :				175.19579	26.92346	

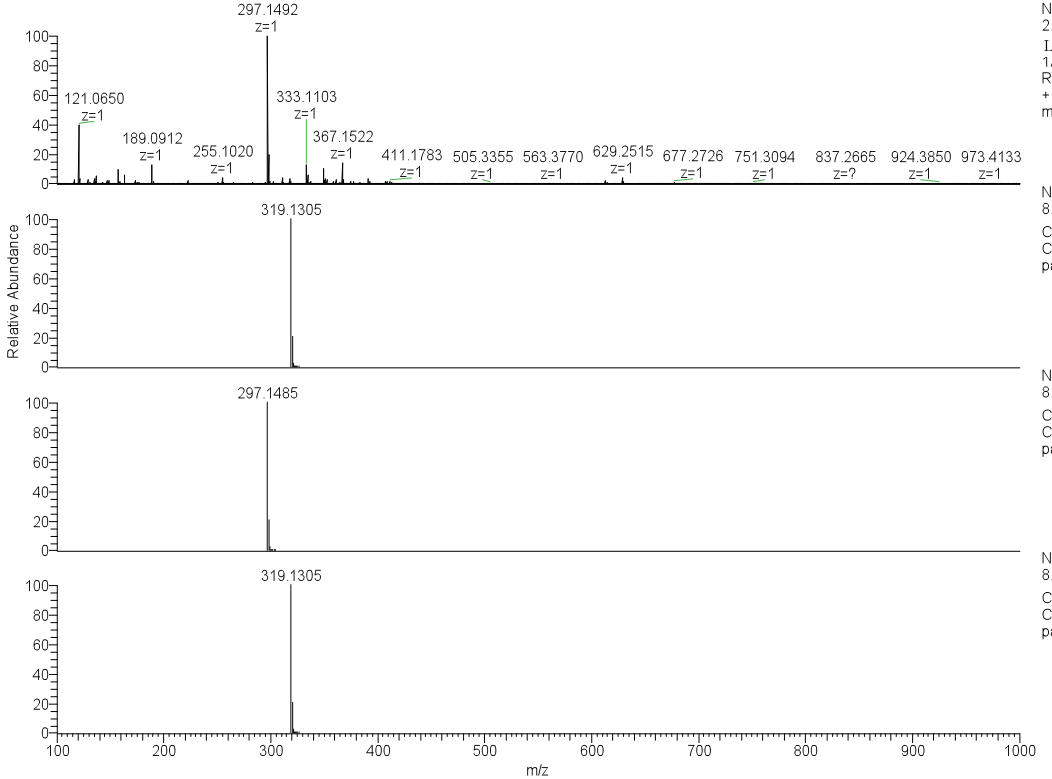
Data File C:\CHEM32\1\DATA\LAXMAN\LD-IV-131-1A\LDIV131-1ARUN02.D
Sample Name: LD-IV-131-1A-rerun-run1

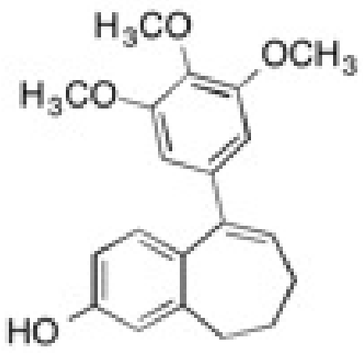
Signal 8: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.422	BB	0.1003	11.65500	1.73621	100.0000
Totals :				11.65500	1.73621	

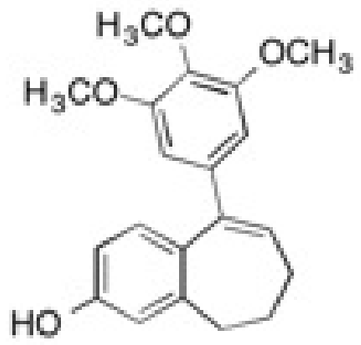
*** End of Report ***

HRMS for compound 67





68



68

154.858
152.758
143.978
142.680
138.704
137.036
132.125
130.654
127.057
115.409
112.868
105.280
60.952
56.099
35.009
32.597
25.485

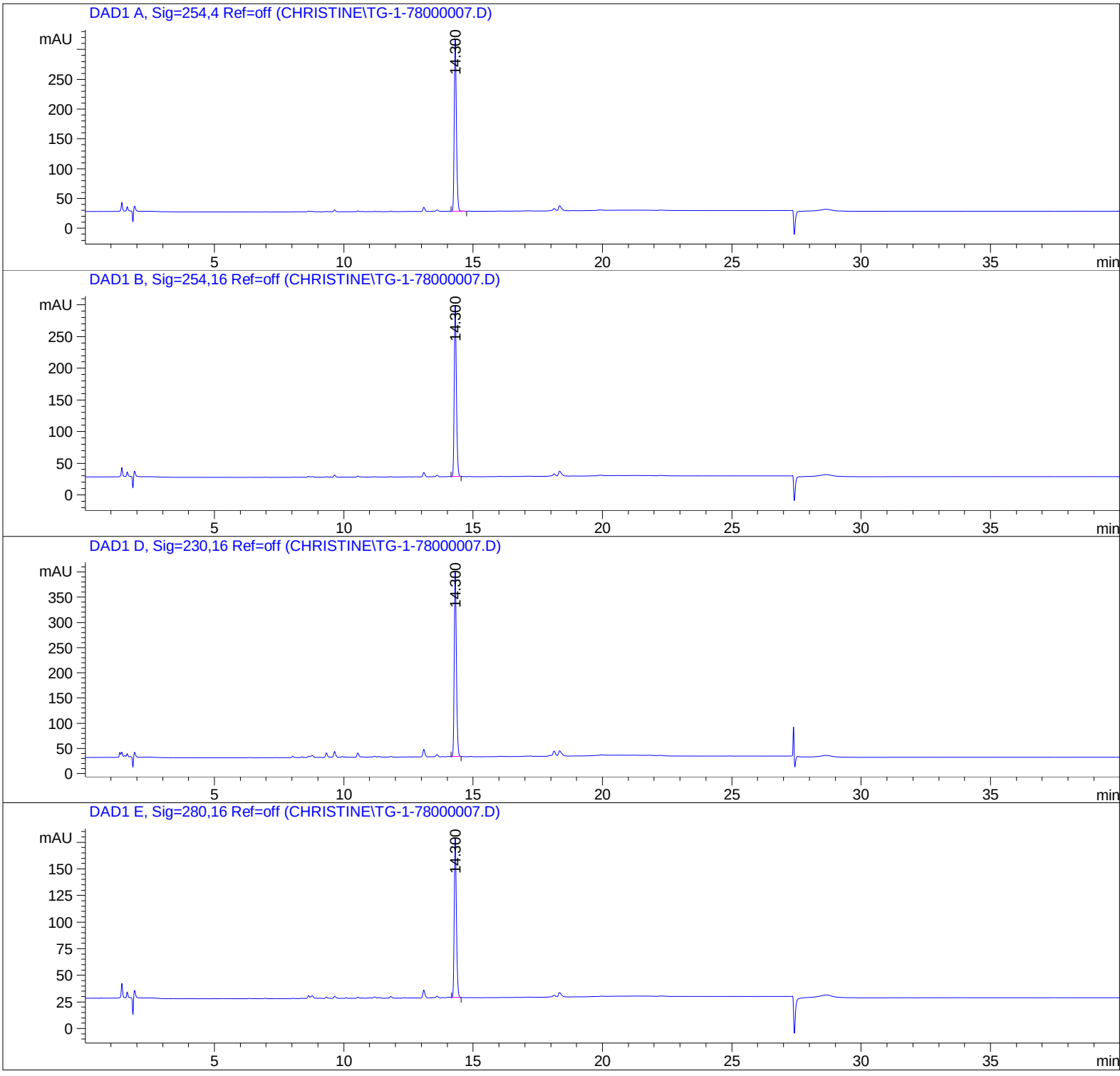
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\TG-1-78000007.D
Sample Name: TG-1-78

HPLC for compound 68

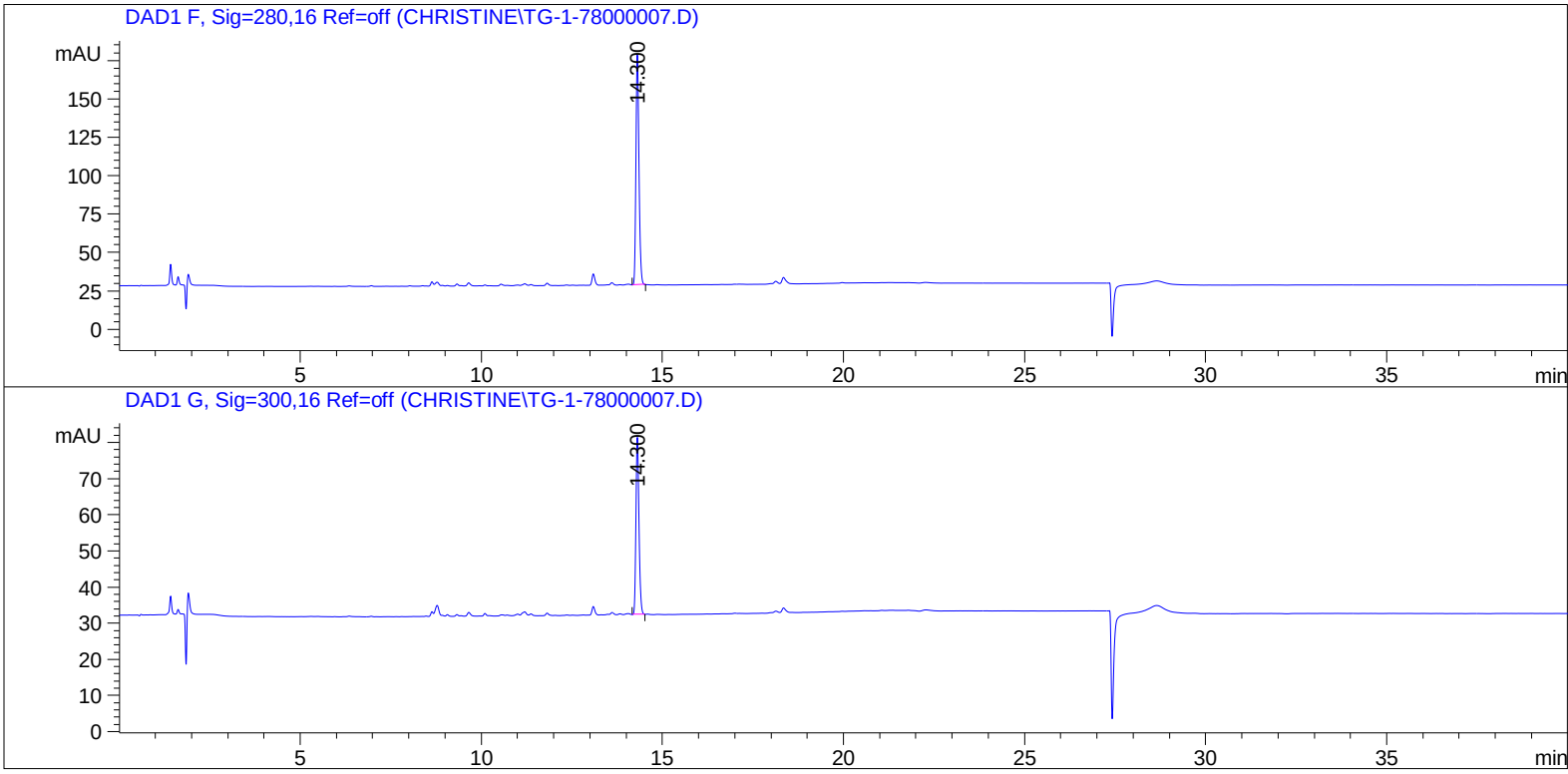
==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 10/9/2013 1:55:20 PM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 10/9/2013 1:53:39 PM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\TG-1-78000007.D\DA.M (MASTERMETHOD.M)
Last changed : 10/9/2013 3:34:35 PM by Christine



S203

Data File C:\CHEM32\1\DATA\CHRISTINE\TG-1-78000007.D
Sample Name: TG-1-78



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0879	1684.01782	289.35425	100.0000
Totals :				1684.01782	289.35425	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0877	1571.10010	270.80679	100.0000
Totals :				1571.10010	270.80679	

Data File C:\CHEM32\1\DATA\CHRISTINE\TG-1-78000007.D
Sample Name: TG-1-78

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0878	2134.54639	367.43820	100.0000
Totals :				2134.54639	367.43820	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0877	870.89191	150.12962	100.0000
Totals :				870.89191	150.12962	

Signal 5: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0877	870.89191	150.12962	100.0000
Totals :				870.89191	150.12962	

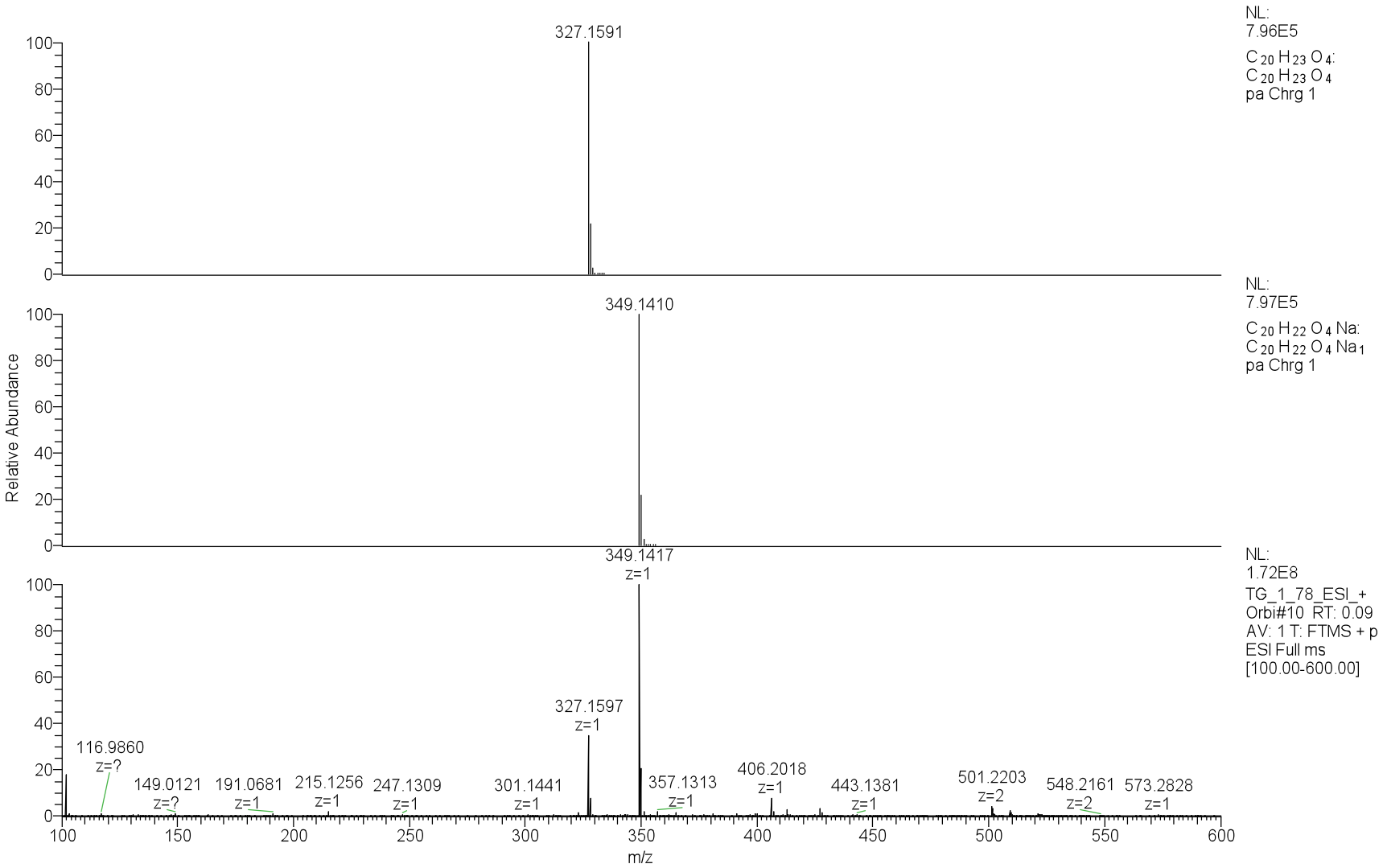
Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.300	BB	0.0877	285.43219	49.20289	100.0000
Totals :				285.43219	49.20289	

==========

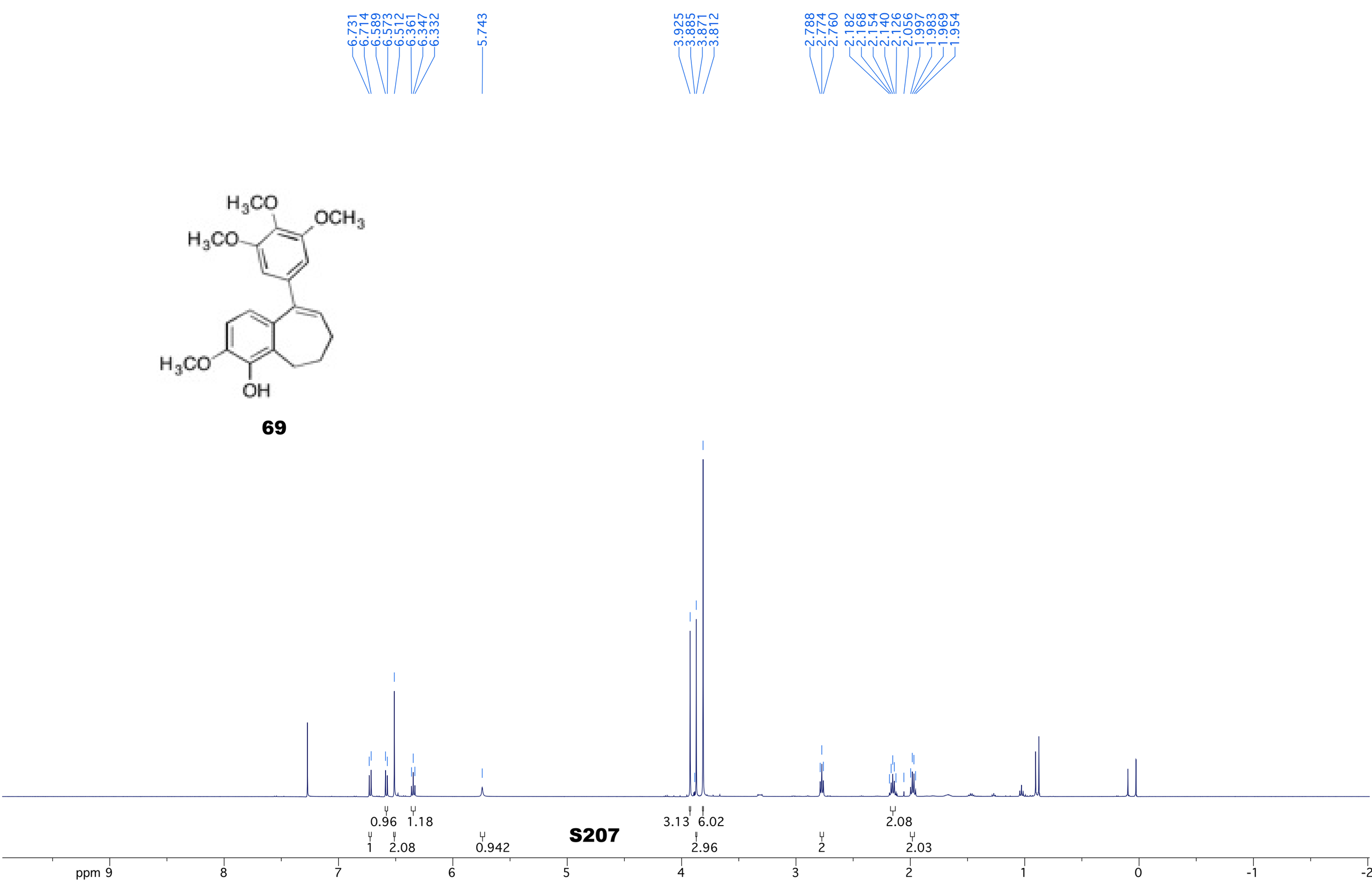
*** End of Report ***

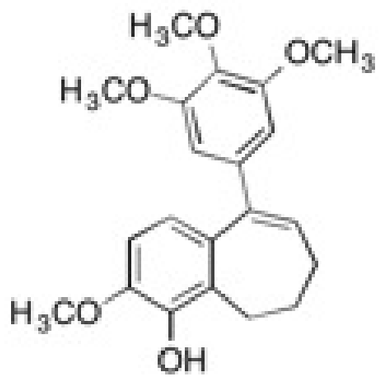
HRMS for compound 68



H3CO
OCH3
H3CO
H3CO
OH

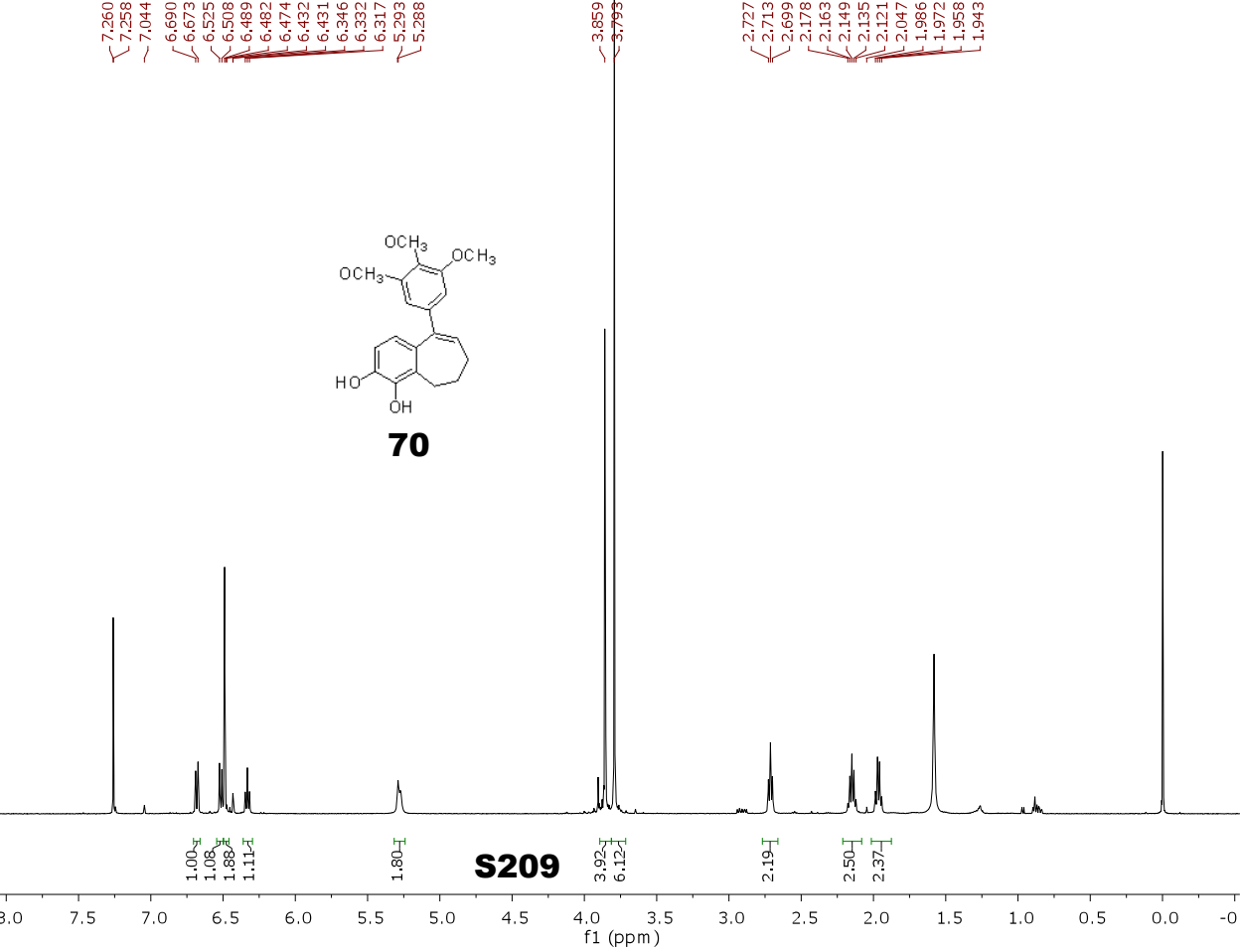
69

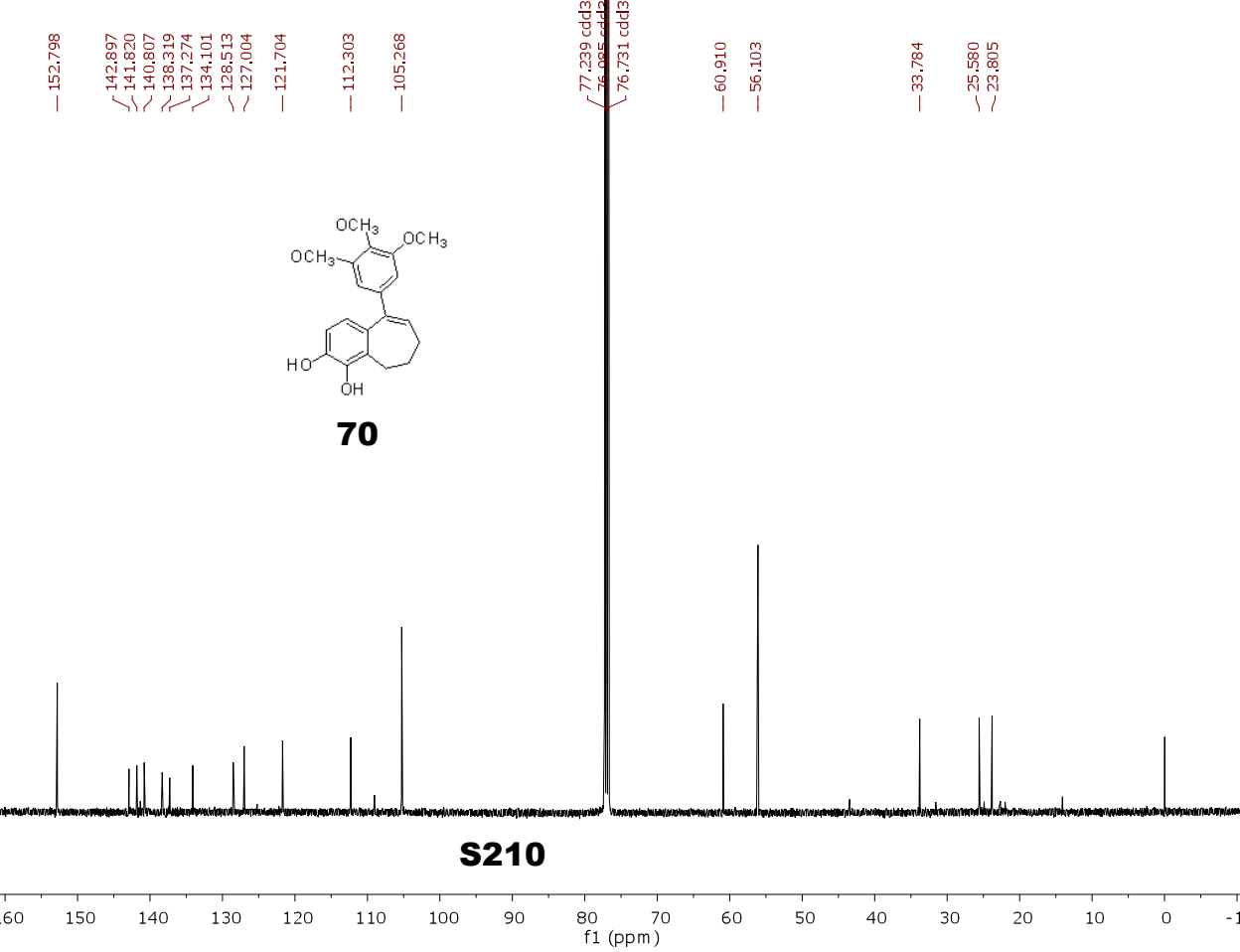

6.731
6.714
6.589
6.573
6.512
6.361
6.347
6.332
5.743
3.925
3.885
3.871
3.812
2.788
2.774
2.760
2.182
2.168
2.154
2.140
2.126
2.056
1.997
1.983
1.969
1.954
0.96
1.18
3.13
6.02
2.08
1
2.08
0.942
2.96
2
2.03
ppm 9
8
7
6
5
4
3
2
1
0
-1
-2



69

152.810
145.040
142.772
142.309
138.457
137.301
134.227
127.746
127.200
120.820
107.647
105.287
60.905
56.126
55.944
33.587
25.698
23.534


7.260
7.258
7.044
6.690
6.673
6.525
6.508
6.489
6.482
6.474
6.432
6.431
6.346
6.332
6.317
5.293
5.288
3.859
3.793
2.727
2.713
2.699
2.178
2.163
2.149
2.135
2.121
2.047
1.986
1.972
1.958
1.943
OCH3
OCH3
OCH3
HO
OH
70
1.00
1.08
1.88
1.11
1.80
S209
3.92
6.12
2.19
2.50
2.37
f1 (ppm)


152.798
142.897
141.820
140.807
138.319
137.274
134.101
128.513
127.004
121.704
112.303
105.268
77.239 cdd3
76.985 cdd2
76.731 cdd3
60.910
56.103
33.784
25.580
23.805
OCH3
OCH3
OCH3
HO
OH
70
S210
f1 (ppm)

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-73000001.D
Sample Name: LD-VI-73-1A-run1

HPLC for compound 70

```
==========================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 9/12/2014 2:43:59 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 9/12/2014 2:31:31 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-VI-73000001.D\DA.M (MASTERMETHOD.M)
Last changed    : 9/12/2014 5:02:51 PM by Eric Lin
Sample Info     : Method-Mastermethod
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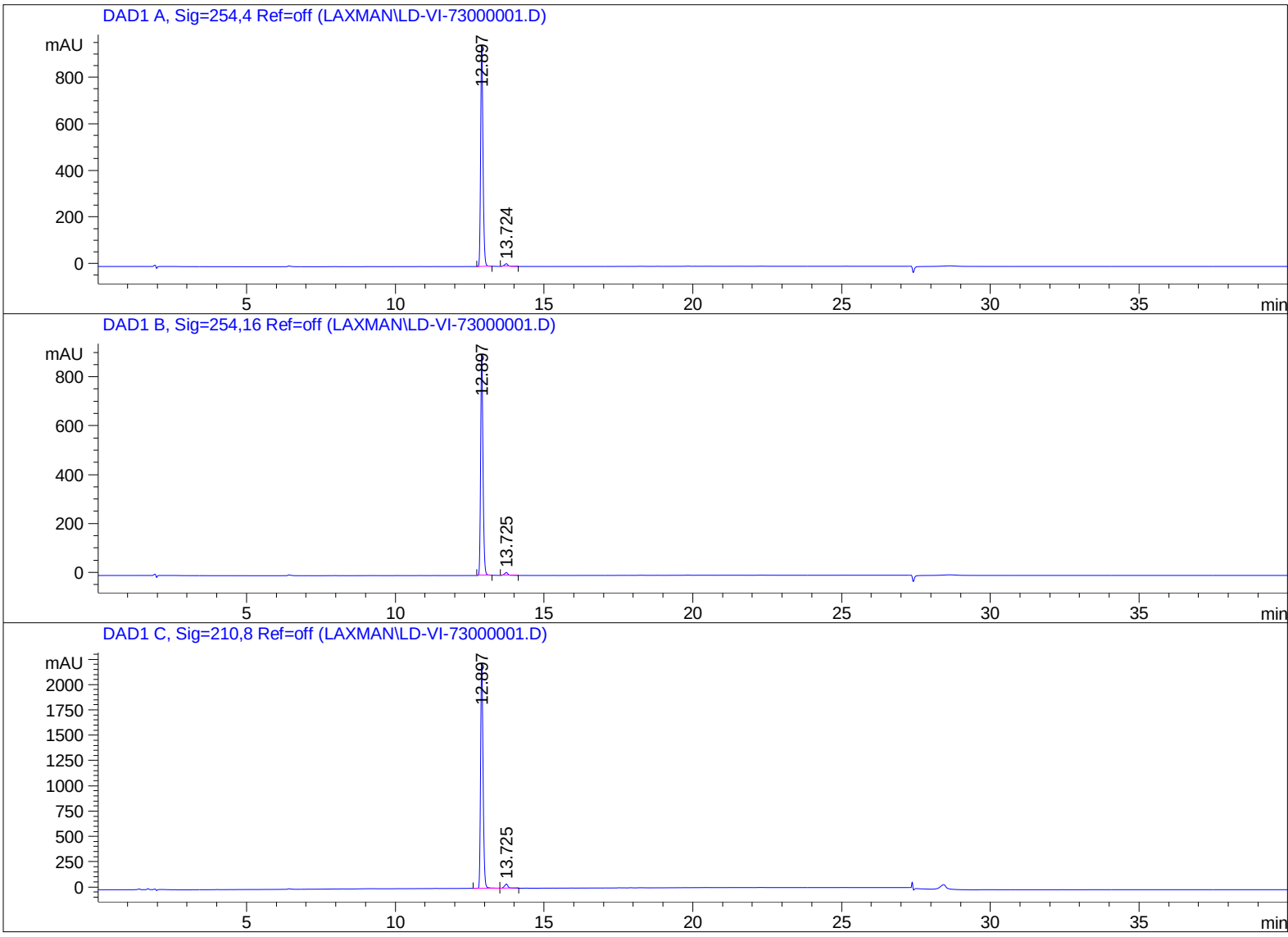
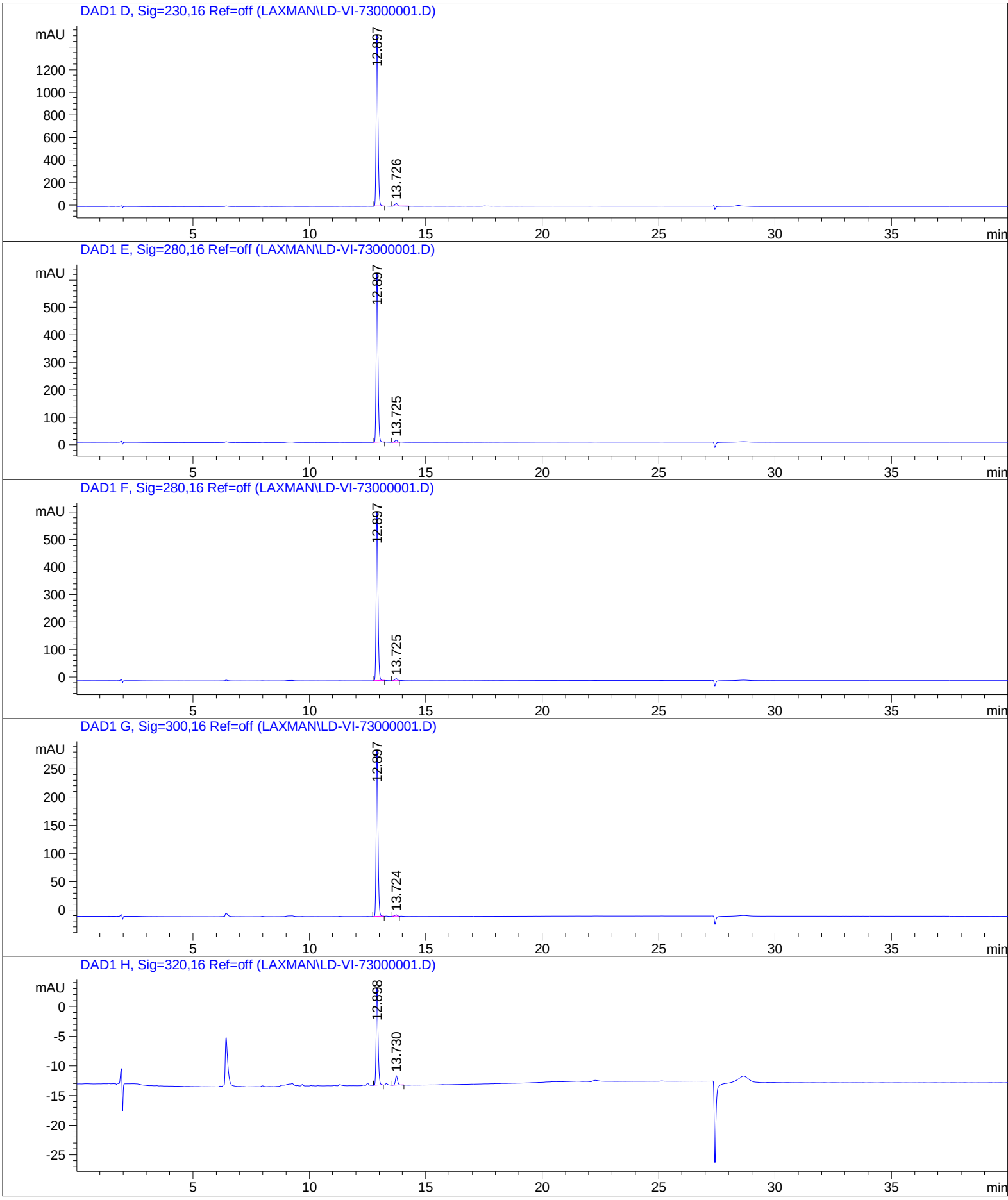


S211

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-73000001.D
Sample Name: LD-VI-73-1A-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-73000001.D
Sample Name: LD-VI-73-1A-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BB	0.0842	5219.16211	948.45355	98.1260
2	13.724	BB	0.1178	99.67384	11.89176	1.8740
Totals :				5318.83595	960.34531	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BB	0.0842	4968.39111	902.85382	98.0270
2	13.725	BB	0.1175	100.00153	11.96880	1.9730
Totals :				5068.39264	914.82262	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BV	0.0898	1.29026e4	2218.69849	97.3838
2	13.725	VB	0.1184	346.62473	41.94163	2.6162
Totals :				1.32492e4	2260.64012	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BB	0.0850	8459.47461	1519.36890	97.5900
2	13.726	BB	0.1176	208.90370	25.50353	2.4100

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-73000001.D
Sample Name: LD-VI-73-1A-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				8668.37831	1544.87242	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BB	0.0842	3387.88330	615.90149	98.2865
2	13.725	BV	0.1117	59.06462	7.51596	1.7135
Totals :				3446.94792	623.41745	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BB	0.0842	3387.88477	615.90149	98.2865
2	13.725	BV	0.1137	59.06182	7.51595	1.7135
Totals :				3446.94659	623.41744	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.897	BV	0.0844	1630.89148	295.33459	98.6910
2	13.724	BB	0.1092	21.63143	2.82855	1.3090
Totals :				1652.52291	298.16315	

Signal 8: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.898	BB	0.0895	94.41895	16.31277	90.0631
2	13.730	BB	0.0986	10.41746	1.58729	9.9369
Totals :				104.83641	17.90006	

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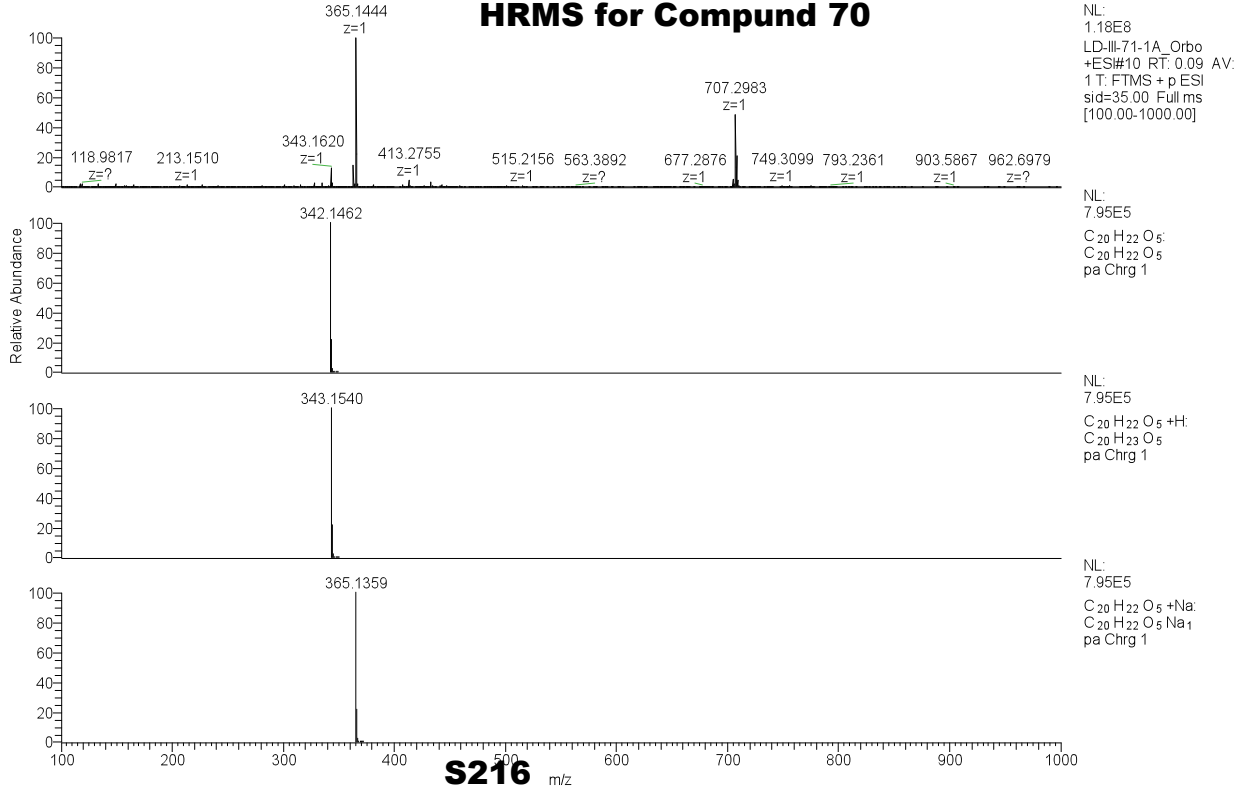
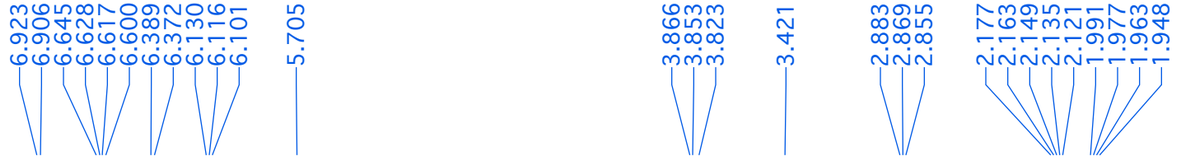
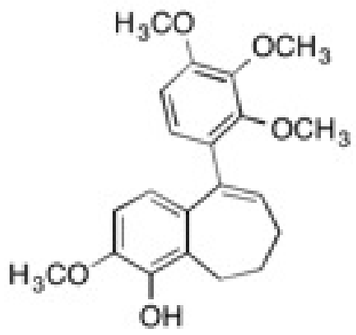
*** End of Report ***

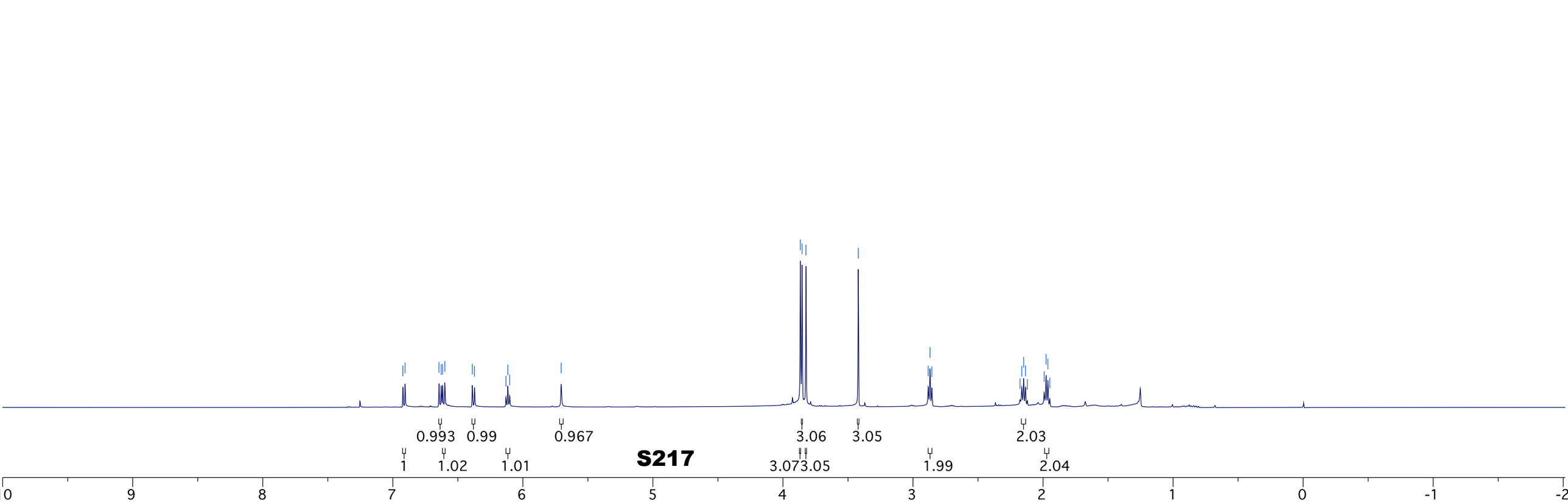
HRMS for Compund 70




6.923
6.906
6.645
6.628
6.617
6.600
6.389
6.372
6.130
6.116
6.101
5.705
3.866
3.853
3.823
3.421
2.883
2.869
2.855
2.177
2.163
2.149
2.135
2.121
1.991
1.977
1.963
1.948

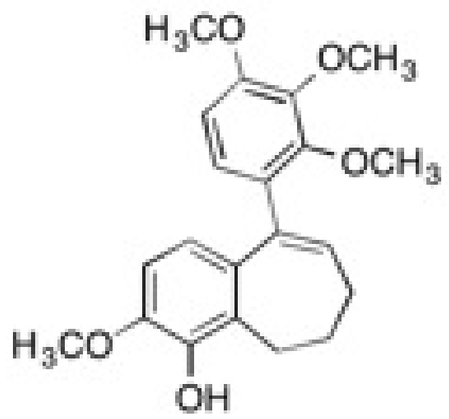

H3CO
OCH3
OCH3
H3CO
OH

71


0.993
0.99
0.967
3.06
3.05
2.03
1
1.02
1.01
3.07
3.05
1.99
2.04
10
9
8
7
6
5
4
3
2
1
0
-1
-2

S217

153.038
151.752
144.668
142.390
142.368
139.993
136.245
130.950
128.561
126.813
125.000
119.014
107.257
106.624
60.694
60.461
55.938
55.860
33.606
25.586
23.436



71

S218

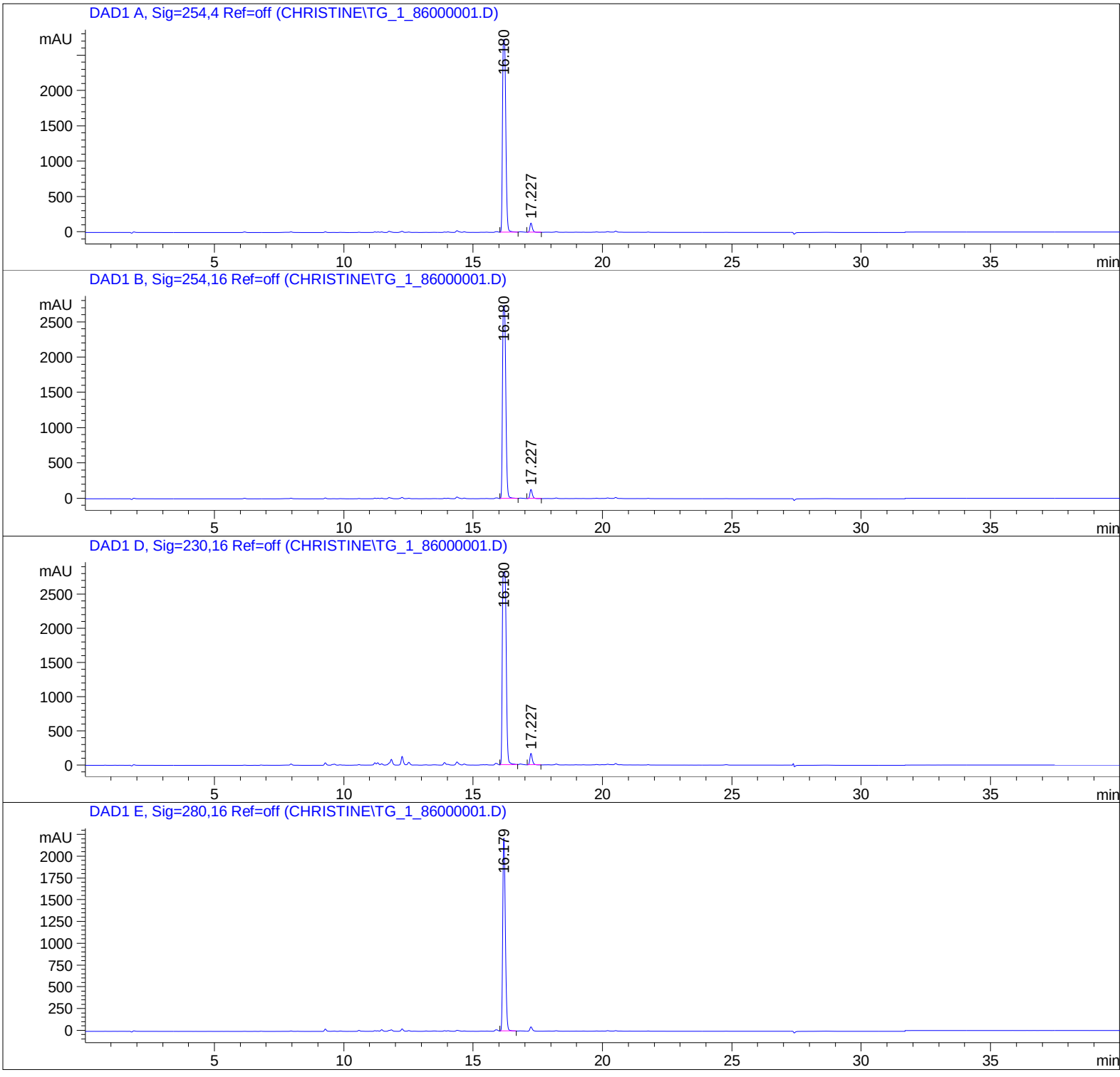
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_86000001.D
Sample Name: TG_1_86

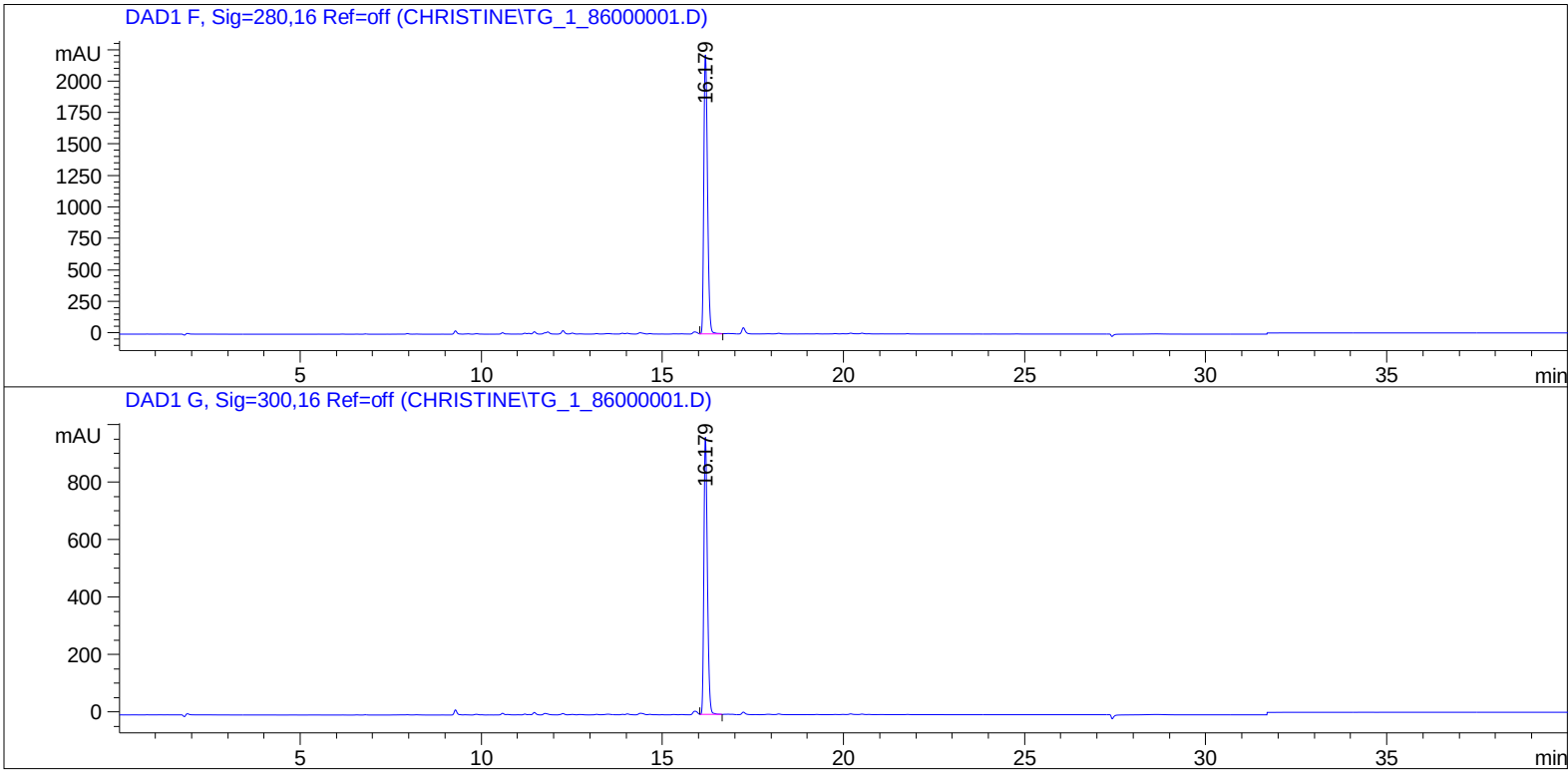
HPLC of compound 71

==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/19/2014 11:07:23 AM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 5/19/2014 10:53:24 AM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\TG_1_86000001.D\DA.M (MASTERMETHOD.M)
Last changed : 5/19/2014 12:11:57 PM by Christine



Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_86000001.D
Sample Name: TG_1_86



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.180	VV	0.1421	2.38945e4	2730.56470	96.5205
2	17.227	VB	0.1009	861.36761	130.67624	3.4795

Totals : 2.47558e4 2861.24094

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.180	VV	0.1423	2.39879e4	2735.90332	96.5594
2	17.227	VB	0.1008	854.72571	129.77695	3.4406

Totals :	2.48426e4	2865.68027

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.180	BV	0.1687	2.92081e4	2822.58813	96.3796
2	17.227	BB	0.1002	1097.15686	168.01945	3.6204
Totals :				3.03053e4	2990.60759	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.179	VV	0.1104	1.57028e4	2223.08569	100.0000
Totals :				1.57028e4	2223.08569	

Signal 5: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.179	VV	0.1104	1.57028e4	2223.08569	100.0000
Totals :				1.57028e4	2223.08569	

Signal 6: DAD1 G, Sig=300,16 Ref=off

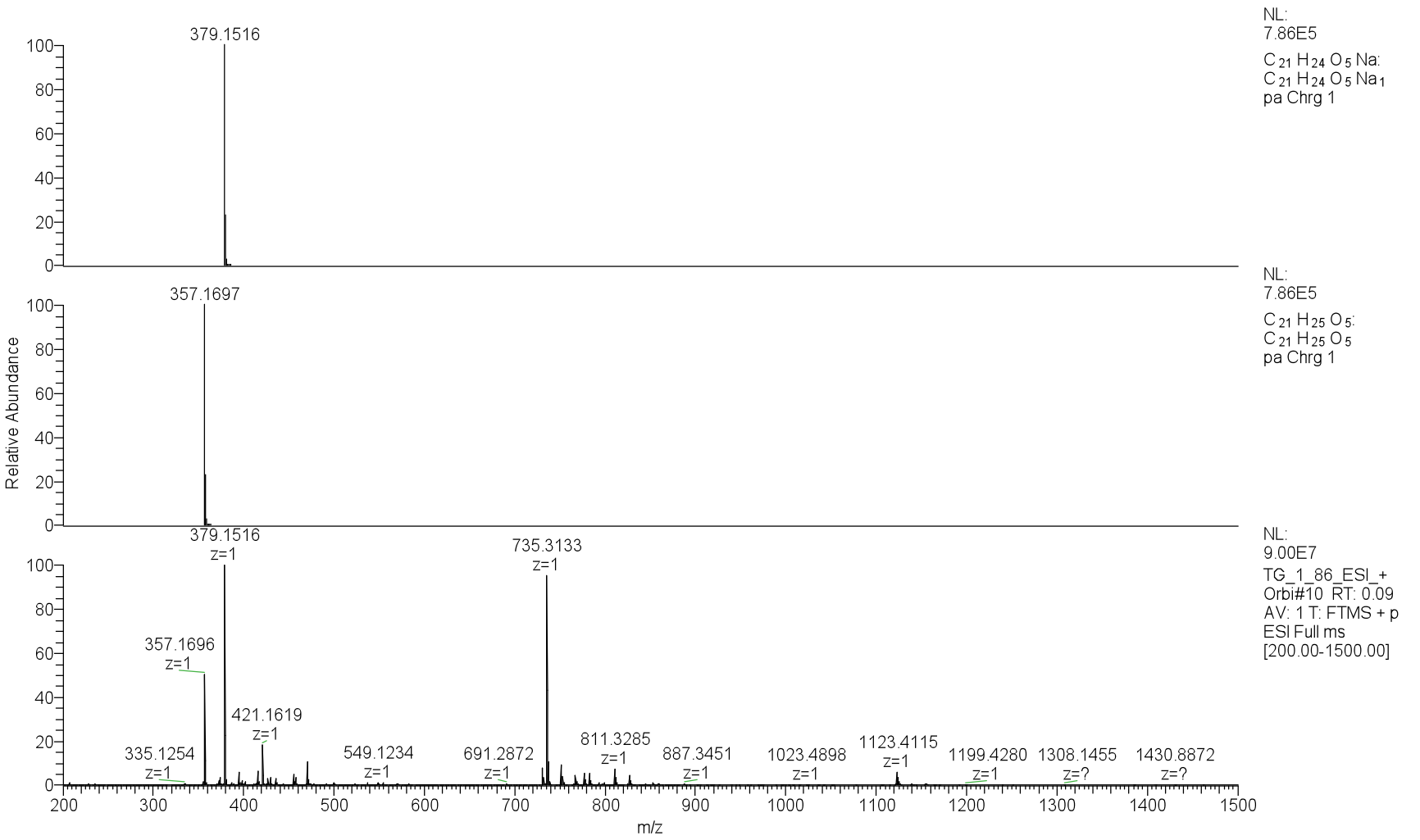
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.179	VV	0.1017	6443.40820	967.43469	100.0000
Totals :				6443.40820	967.43469	

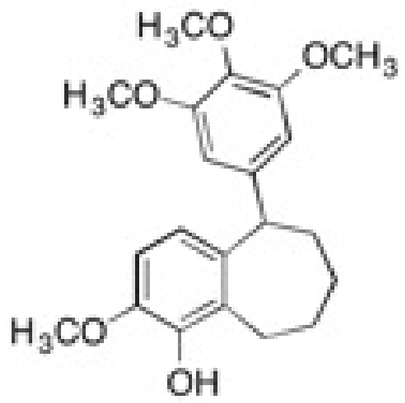
==========

*** End of Report ***

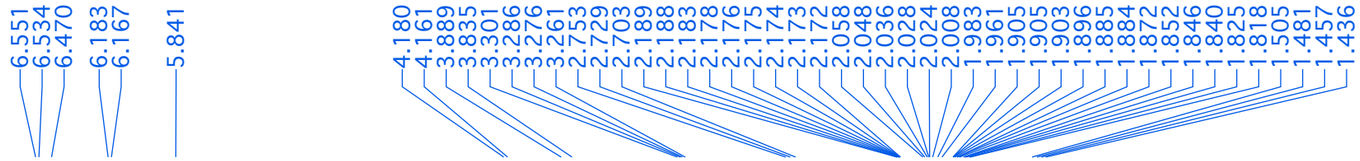
HRMS of compound 71

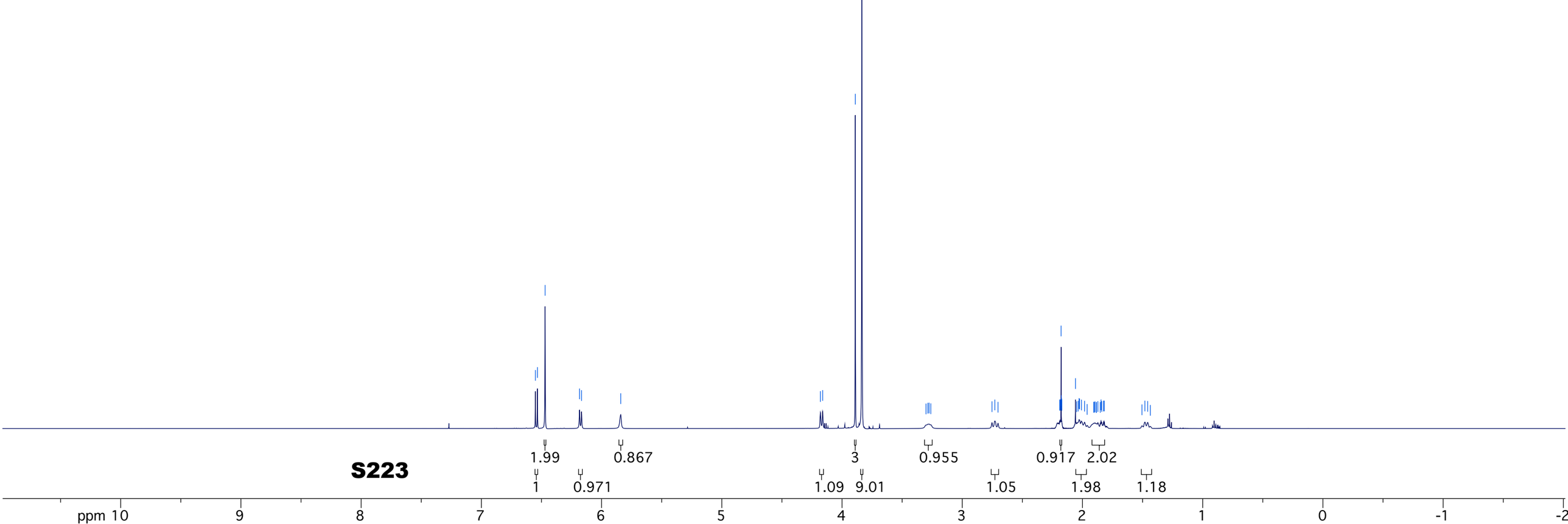
C:\Xcalibur\...\TG_1_86_ESI_+Orbi 1/13/2014 1:41:22 PM TG-1-86



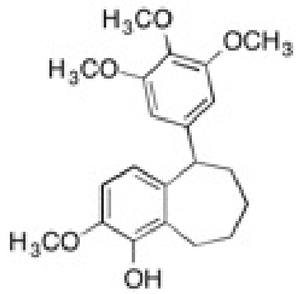

H3CO
OCH3
H3CO
H3CO
OH

72


6.551
6.534
6.470
6.183
6.167
5.841
4.180
4.161
3.889
3.835
3.301
3.286
3.276
3.261
2.753
2.729
2.703
2.189
2.188
2.183
2.178
2.176
2.175
2.174
2.173
2.172
2.058
2.048
2.036
2.028
2.024
2.008
1.983
1.961
1.905
1.905
1.903
1.896
1.885
1.884
1.872
1.852
1.846
1.840
1.825
1.818
1.505
1.481
1.457
1.436


1.99
0.867
3
0.955
0.917
2.02
1
0.971
1.09
9.01
1.05
1.98
1.18
ppm 10
9
8
7
6
5
4
3
2
1
0
-1
-2

S223



72

153.081
144.696
142.609
141.167
139.647
136.158
128.389
118.565
107.130
105.663
60.873
56.072
55.862
49.535
34.679
30.626
27.194
25.365

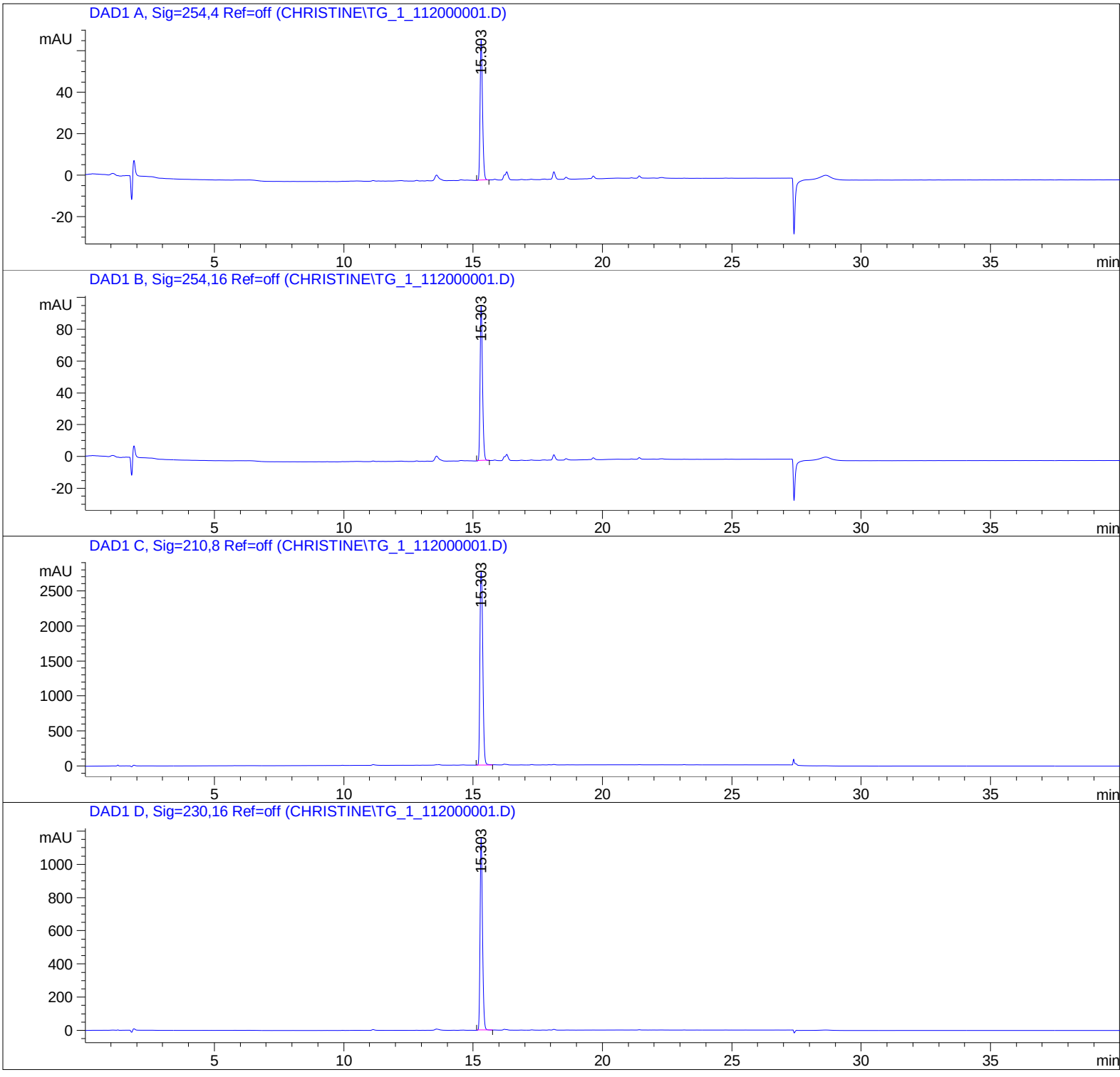
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_112000001.D
Sample Name: TG_1_112

HPLC of compound 72

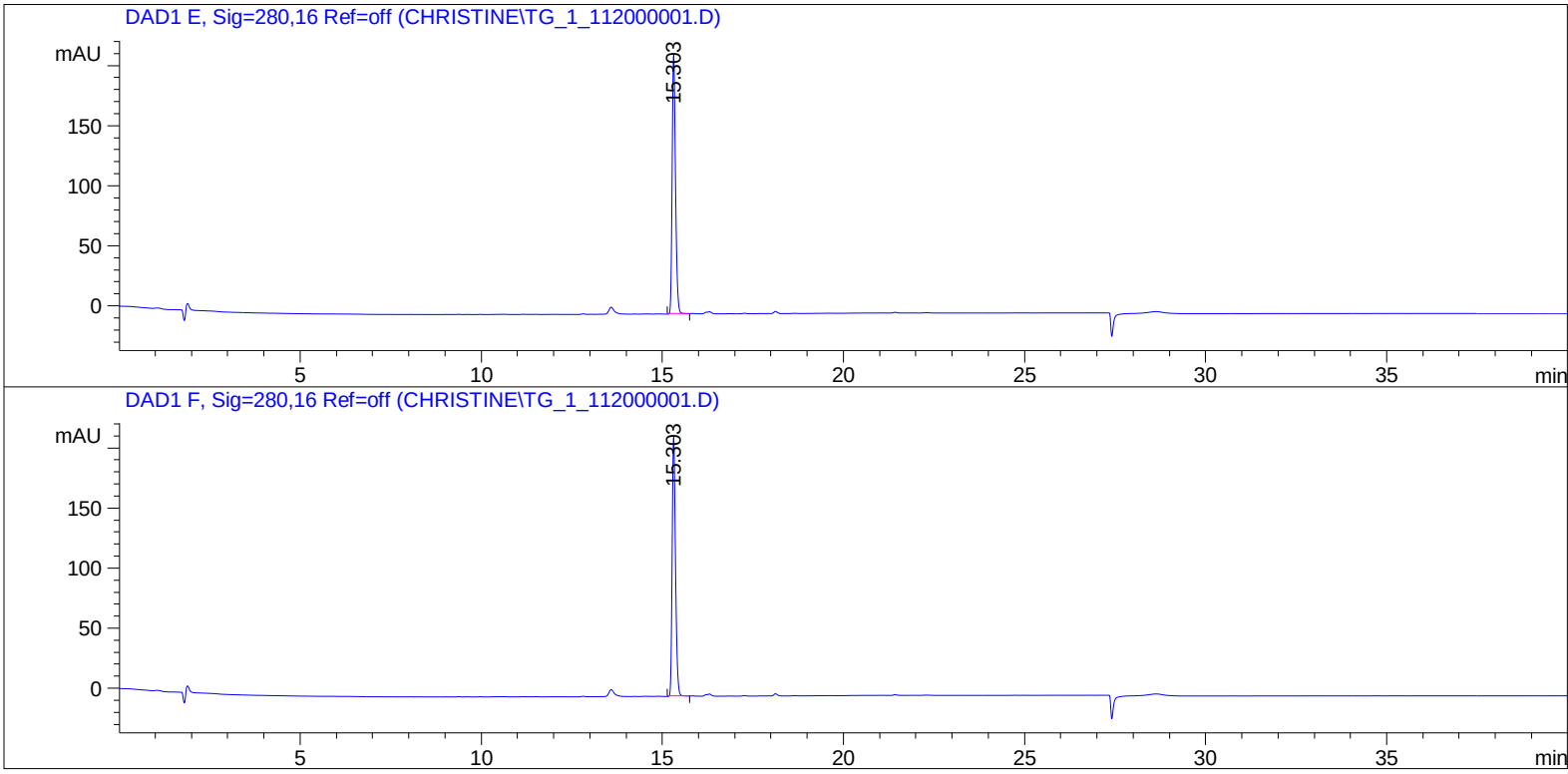
==========

Acq. Operator : Christine
Acq. Instrument : Instrument 1 Location : -
Injection Date : 6/5/2014 11:26:01 AM
Acq. Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed : 6/5/2014 11:13:29 AM by Christine
Analysis Method : C:\CHEM32\1\DATA\CHRISTINE\TG_1_112000001.D\DA.M (MASTERMETHOD.M)
Last changed : 6/10/2014 10:09:52 AM by Eric Lin



S225

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_112000001.D
Sample Name: TG_1_112



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BB	0.1000	442.72894	67.97310	100.0000
Totals :				442.72894	67.97310	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BB	0.0995	631.04565	97.49252	100.0000
Totals :				631.04565	97.49252	

Data File C:\CHEM32\1\DATA\CHRISTINE\TG_1_112000001.D
Sample Name: TG_1_112

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BV	0.1224	2.13590e4	2755.23682	100.0000
Totals :				2.13590e4	2755.23682	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BB	0.0996	7499.44434	1157.97766	100.0000
Totals :				7499.44434	1157.97766	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BB	0.0987	1380.06250	215.62195	100.0000
Totals :				1380.06250	215.62195	

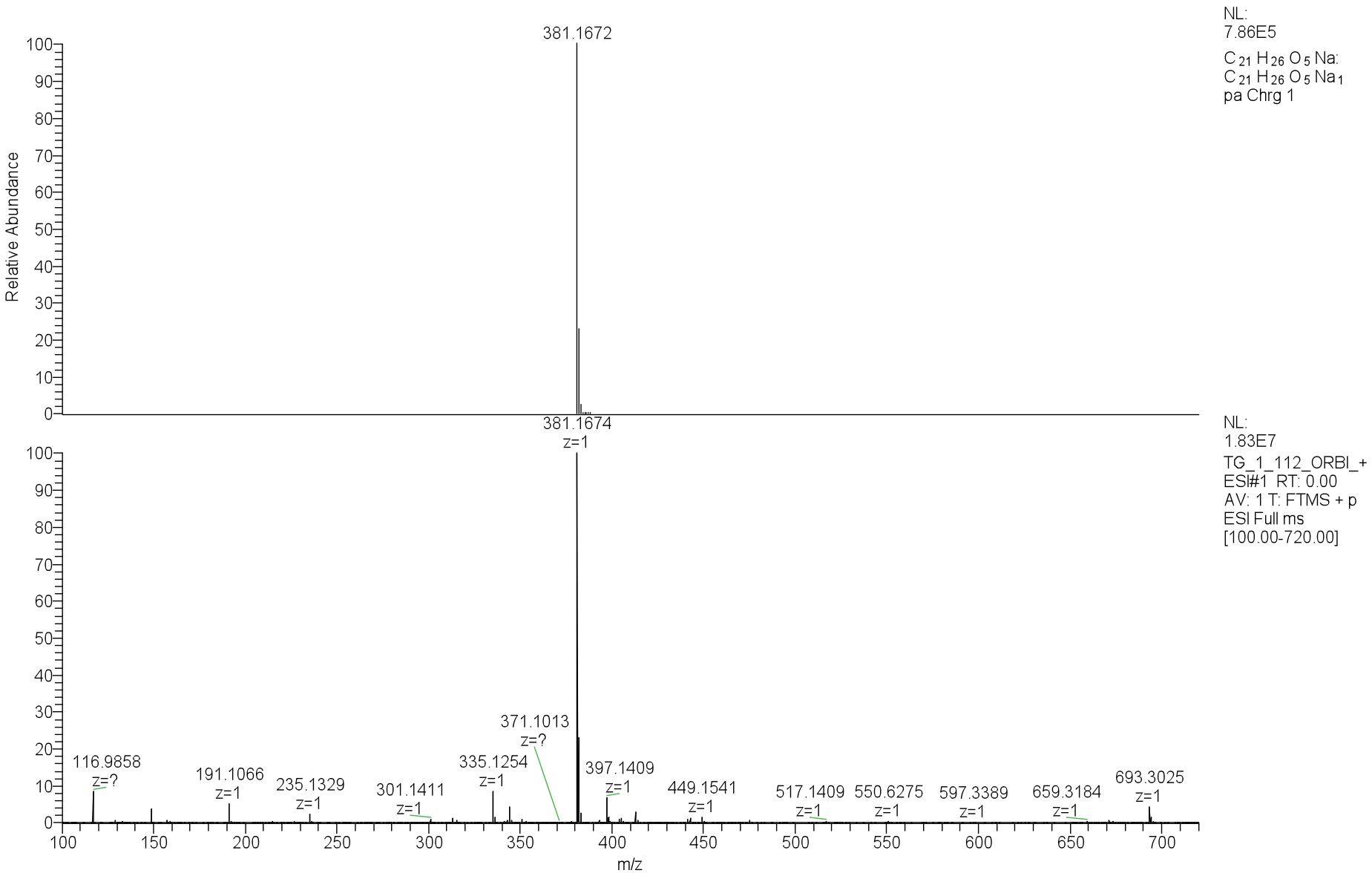
Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.303	BB	0.0987	1380.06250	215.62195	100.0000
Totals :				1380.06250	215.62195	

==========

*** End of Report ***

HRMS of compound 72



X-ray Crystallographic Analysis:

X-ray crystallographic analysis of compound **72**.[S1] Crystallographic data were collected on a crystal of **72** with dimensions 0.30 x 0.17 x 0.14 mm^3. Data were collected at 150 K on a Bruker X8 Apex using Mo KR radiation (λ = 0.71073 Å). The structure was solved by direct methods after correction of the data using SADABS. Crystallographic data and definement details for the complex mentioned herein is found in the Supporting Information (Table S1-S5). The thermal ellipsoid plots at 50% probability for compound **73** is displayed in Figure S1. All data were processed using the Bruker AXS SHELXTL software, version 6.10.

Figure S1. X-ray crystallogaphy of compound **72**

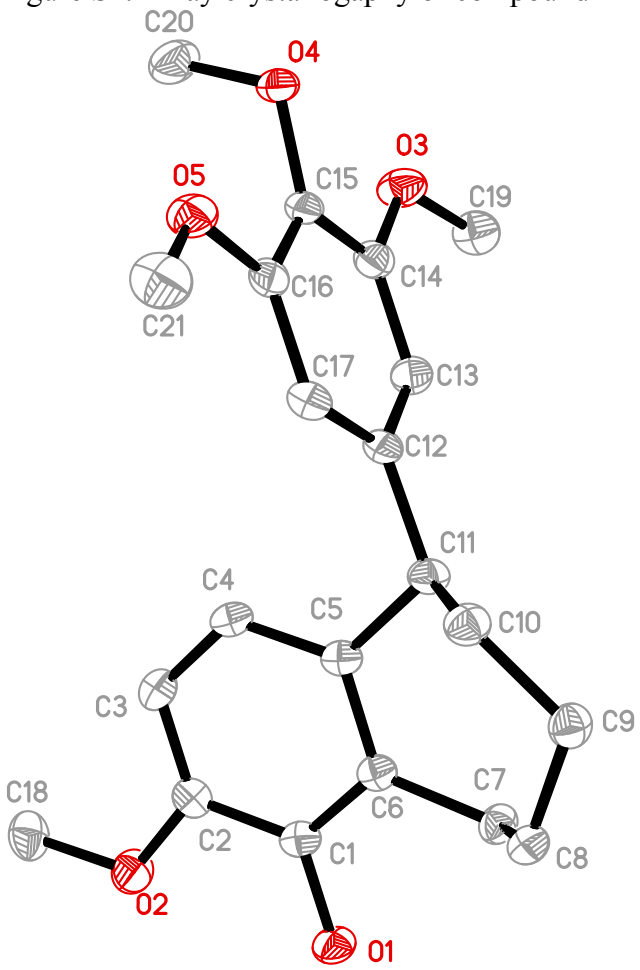


Table S1. Crystal data and structure refinement for Compound 72

Identification code	KP69	
Empirical formula	C21 H26 O5	
Formula weight	358.42	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P c a 21	
Unit cell dimensions	a = 16.5289(7) Å	α= 90°.
	b = 11.7370(4) Å	β= 90°.
	c = 9.4340(4) Å	γ = 90°.
Volume	1830.19(13) $Å^3$	
Z	4	
Density (calculated)	1.301 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	768	
Crystal size	0.303 x 0.165 x 0.144 mm^3	
Theta range for data collection	5.211 to 25.675°.	
Index ranges	-20<=h<=18, -14<=k<=12, -11<=l<=11	
Reflections collected	15353	
Independent reflections	3439 [R(int) = 0.0219]	
Completeness to theta = 25.242°	98.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.987 and 0.981	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3439 / 1 / 238	
Goodness-of-fit on F^2	1.040	
Final R indices [I>2sigma(I)]	R1 = 0.0270, wR2 = 0.0688	
R indices (all data)	R1 = 0.0283, wR2 = 0.0696	
Absolute structure parameter	0.27(19)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.164 and -0.148 e.$Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Compound 72. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	1246(1)	1871(1)	4248(2)	24(1)
O(2)	1430(1)	2238(1)	1512(2)	29(1)
O(3)	3644(1)	8680(1)	4127(2)	28(1)
O(4)	2556(1)	10180(1)	3151(2)	24(1)
O(5)	990(1)	9662(1)	2973(2)	26(1)
C(1)	1350(1)	2969(2)	3795(2)	20(1)
C(2)	1457(1)	3188(2)	2347(2)	20(1)
C(3)	1576(1)	4290(2)	1891(2)	22(1)
C(4)	1583(1)	5174(2)	2885(2)	21(1)
C(5)	1469(1)	4976(2)	4319(2)	19(1)
C(6)	1353(1)	3842(2)	4791(2)	18(1)
C(7)	1236(1)	3543(2)	6335(2)	21(1)
C(8)	465(1)	4047(2)	6998(2)	22(1)
C(9)	551(1)	5284(2)	7436(2)	23(1)
C(10)	674(1)	6106(2)	6204(2)	23(1)
C(11)	1482(1)	5954(2)	5399(2)	21(1)
C(12)	1759(1)	7070(2)	4737(2)	20(1)
C(13)	2579(1)	7317(2)	4714(2)	22(1)
C(14)	2852(1)	8337(2)	4136(2)	22(1)
C(15)	2300(1)	9113(2)	3569(2)	20(1)
C(16)	1482(1)	8849(2)	3561(2)	21(1)
C(17)	1208(1)	7832(2)	4148(2)	22(1)
C(18)	1560(1)	2377(2)	33(2)	30(1)
C(19)	4209(1)	7955(2)	4832(2)	29(1)
C(20)	2828(2)	10219(2)	1717(2)	33(1)
C(21)	138(1)	9516(2)	3108(3)	34(1)

Table S3. Bond lengths [Å] and angles [°] for Compound 72.

O(1)-C(1)	1.369(2)
O(2)-C(2)	1.365(2)
O(2)-C(18)	1.421(2)
O(3)-C(14)	1.370(2)
O(3)-C(19)	1.428(3)
O(4)-C(15)	1.379(2)
O(4)-C(20)	1.427(3)
O(5)-C(16)	1.371(2)
O(5)-C(21)	1.425(3)
C(1)-C(6)	1.390(3)
C(1)-C(2)	1.401(3)
C(2)-C(3)	1.378(3)
C(3)-C(4)	1.398(3)
C(4)-C(5)	1.386(3)
C(5)-C(6)	1.416(2)
C(5)-C(11)	1.535(3)
C(6)-C(7)	1.511(3)
C(7)-C(8)	1.537(3)
C(8)-C(9)	1.516(3)
C(9)-C(10)	1.524(3)
C(10)-C(11)	1.547(3)
C(11)-C(12)	1.522(3)
C(12)-C(13)	1.386(3)
C(12)-C(17)	1.392(3)
C(13)-C(14)	1.391(3)
C(14)-C(15)	1.395(3)
C(15)-C(16)	1.387(3)
C(16)-C(17)	1.392(3)

C(2)-O(2)-C(18)	117.91(17)
C(14)-O(3)-C(19)	116.57(16)
C(15)-O(4)-C(20)	113.41(15)
C(16)-O(5)-C(21)	117.79(16)
O(1)-C(1)-C(6)	118.90(17)

O(1)-C(1)-C(2)	119.50(17)
C(6)-C(1)-C(2)	121.59(17)
O(2)-C(2)-C(3)	126.22(18)
O(2)-C(2)-C(1)	114.18(17)
C(3)-C(2)-C(1)	119.60(18)
C(2)-C(3)-C(4)	119.23(18)
C(5)-C(4)-C(3)	121.97(18)
C(4)-C(5)-C(6)	118.87(17)
C(4)-C(5)-C(11)	121.39(17)
C(6)-C(5)-C(11)	119.73(17)
C(1)-C(6)-C(5)	118.74(17)
C(1)-C(6)-C(7)	118.67(17)
C(5)-C(6)-C(7)	122.59(17)
C(6)-C(7)-C(8)	114.15(16)
C(9)-C(8)-C(7)	113.66(16)
C(8)-C(9)-C(10)	114.30(17)
C(9)-C(10)-C(11)	114.53(16)
C(12)-C(11)-C(5)	112.05(16)
C(12)-C(11)-C(10)	111.19(15)
C(5)-C(11)-C(10)	113.60(15)
C(13)-C(12)-C(17)	119.96(17)
C(13)-C(12)-C(11)	118.70(16)
C(17)-C(12)-C(11)	121.33(17)
C(12)-C(13)-C(14)	120.18(17)
O(3)-C(14)-C(13)	124.41(18)
O(3)-C(14)-C(15)	115.49(17)
C(13)-C(14)-C(15)	120.05(18)
O(4)-C(15)-C(16)	120.02(17)
O(4)-C(15)-C(14)	120.12(17)
C(16)-C(15)-C(14)	119.54(17)
O(5)-C(16)-C(15)	115.12(17)
O(5)-C(16)-C(17)	124.41(18)
C(15)-C(16)-C(17)	120.46(18)
C(16)-C(17)-C(12)	119.78(18)

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Compound 72. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	36(1)	14(1)	24(1)	0(1)	3(1)	0(1)
O(2)	45(1)	21(1)	22(1)	-4(1)	2(1)	2(1)
O(3)	22(1)	26(1)	37(1)	6(1)	-1(1)	-5(1)
O(4)	38(1)	15(1)	21(1)	0(1)	3(1)	-5(1)
O(5)	28(1)	18(1)	34(1)	2(1)	-5(1)	4(1)
C(1)	17(1)	18(1)	25(1)	2(1)	1(1)	1(1)
C(2)	18(1)	21(1)	22(1)	-3(1)	1(1)	2(1)
C(3)	23(1)	24(1)	19(1)	3(1)	2(1)	2(1)
C(4)	21(1)	16(1)	24(1)	3(1)	2(1)	-1(1)
C(5)	16(1)	17(1)	23(1)	1(1)	1(1)	0(1)
C(6)	14(1)	19(1)	22(1)	2(1)	1(1)	1(1)
C(7)	25(1)	18(1)	19(1)	2(1)	1(1)	2(1)
C(8)	25(1)	22(1)	20(1)	2(1)	2(1)	-4(1)
C(9)	24(1)	24(1)	21(1)	-1(1)	4(1)	1(1)
C(10)	25(1)	18(1)	25(1)	0(1)	1(1)	1(1)
C(11)	23(1)	15(1)	23(1)	1(1)	-1(1)	-1(1)
C(12)	27(1)	15(1)	20(1)	-3(1)	0(1)	-3(1)
C(13)	26(1)	17(1)	24(1)	0(1)	-1(1)	2(1)
C(14)	24(1)	21(1)	21(1)	-2(1)	3(1)	-3(1)
C(15)	30(1)	14(1)	17(1)	-2(1)	2(1)	-5(1)
C(16)	28(1)	15(1)	19(1)	-3(1)	-2(1)	2(1)
C(17)	22(1)	18(1)	25(1)	-3(1)	-1(1)	-2(1)
C(18)	36(1)	32(1)	22(1)	-7(1)	1(1)	4(1)
C(19)	23(1)	30(1)	35(1)	2(1)	0(1)	0(1)
C(20)	47(1)	26(1)	26(1)	3(1)	10(1)	-3(1)
C(21)	29(1)	28(1)	46(1)	-2(1)	-4(1)	8(1)

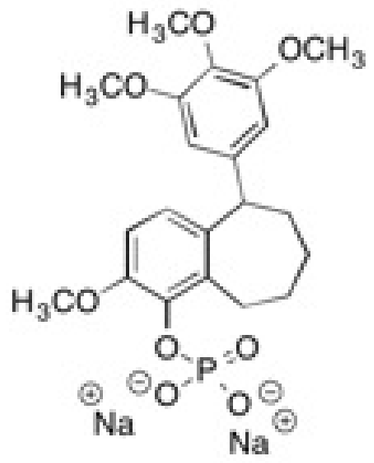
Table 5. Hydrogen bonds for 72 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(2)	0.85(3)	2.17(3)	2.635(2)	115(2)
O(1)-H(1)...O(4)#1	0.85(3)	2.47(3)	3.115(2)	133(2)
O(1)-H(1)...O(5)#1	0.85(3)	2.25(3)	2.8890(19)	133(2)

Symmetry transformations used to generate equivalent atoms:

#1 x,y-1,z

Crystallographic data for structure **72** (deposition number CCDC 1037721) reported in this paper have been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-(0) 1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).



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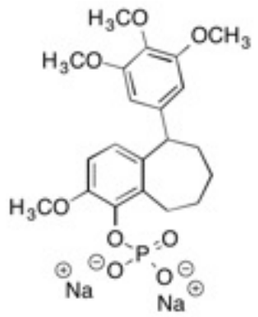

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140.418
139.045
137.262
137.240
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121.761
108.633
105.937
60.830
55.372
48.617
33.706
29.962
26.619

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

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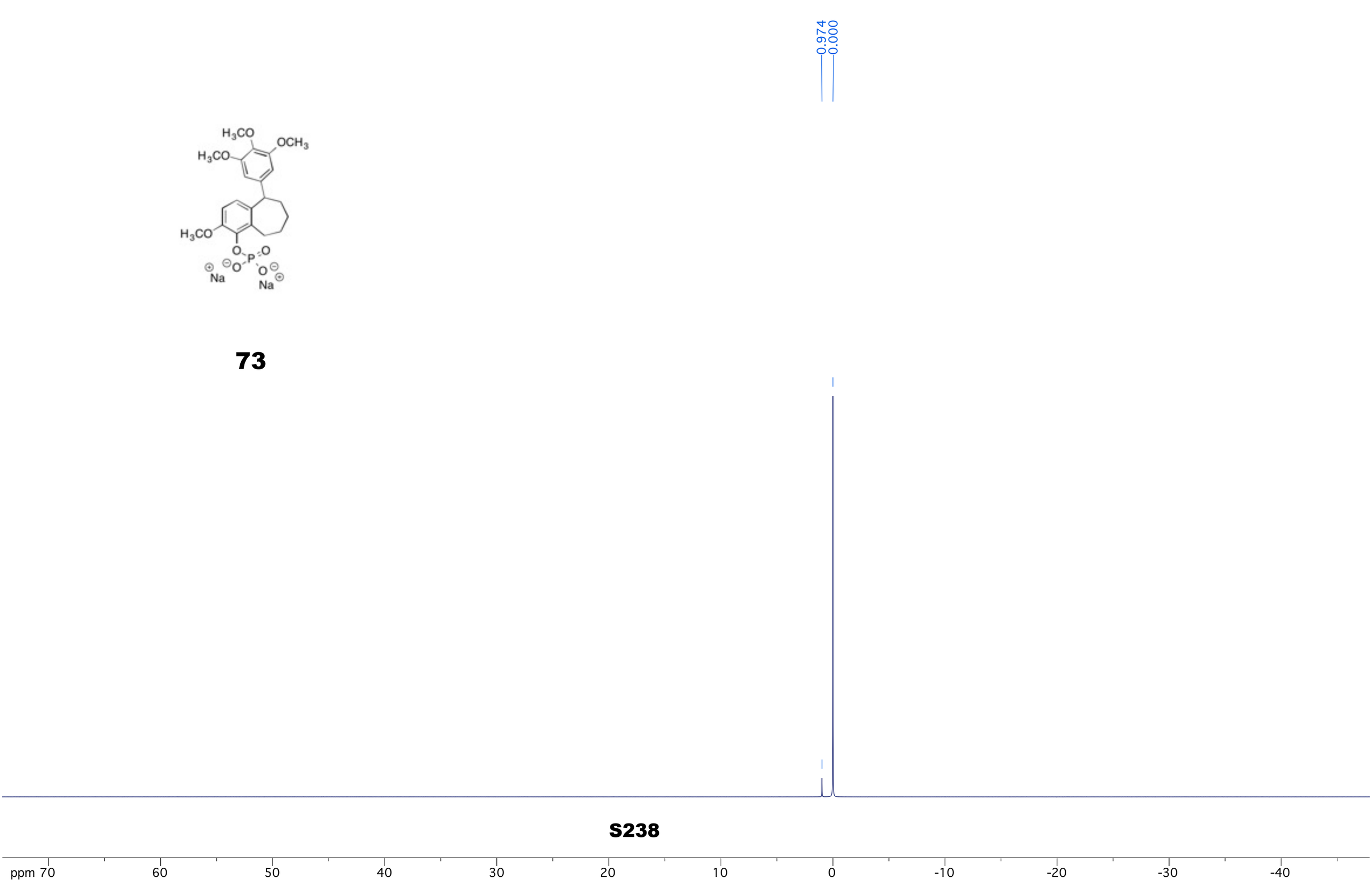
H3CO
O
P
O
O
O
Na
Na

73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S238

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HPLC of compound 73

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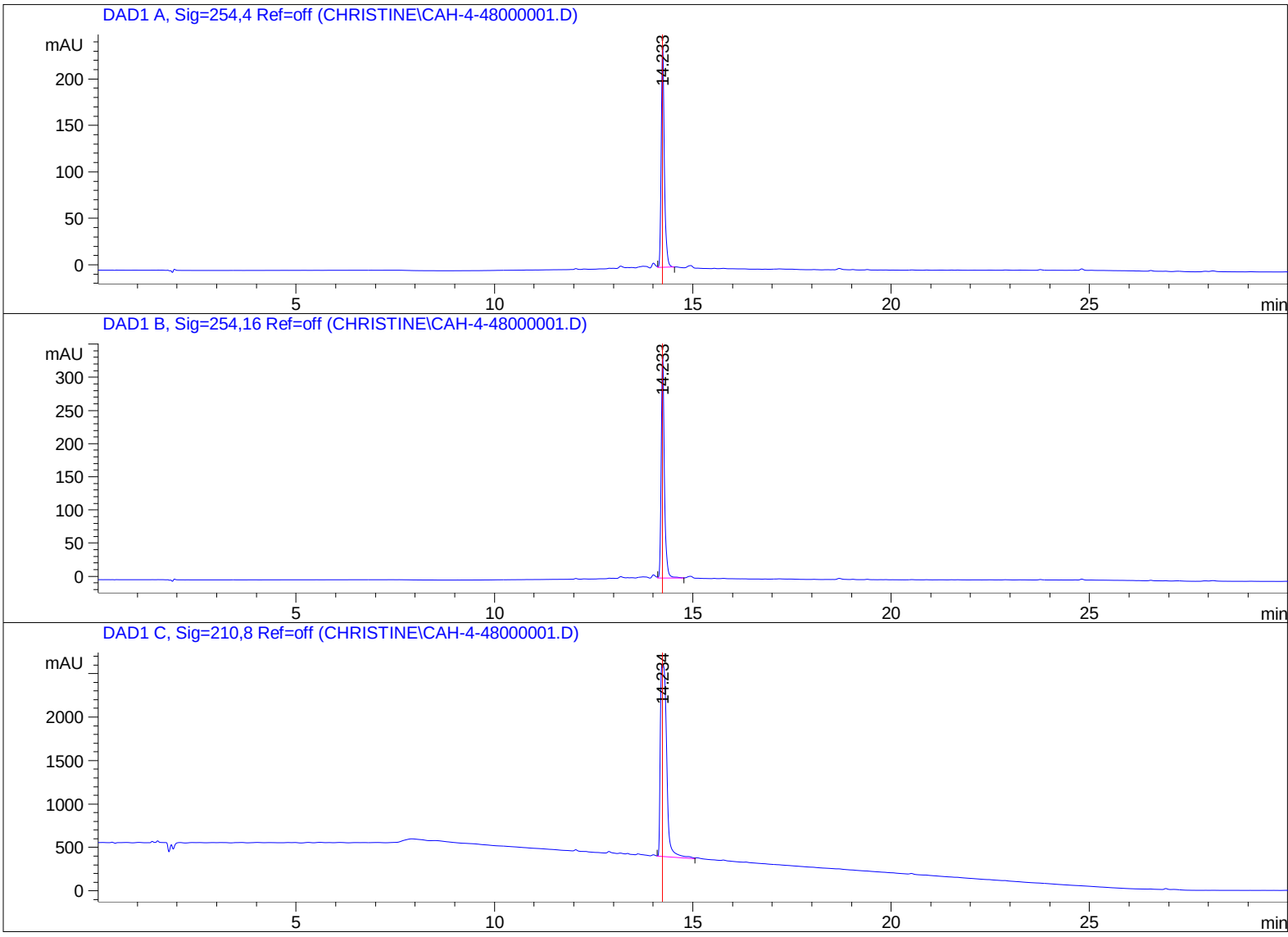

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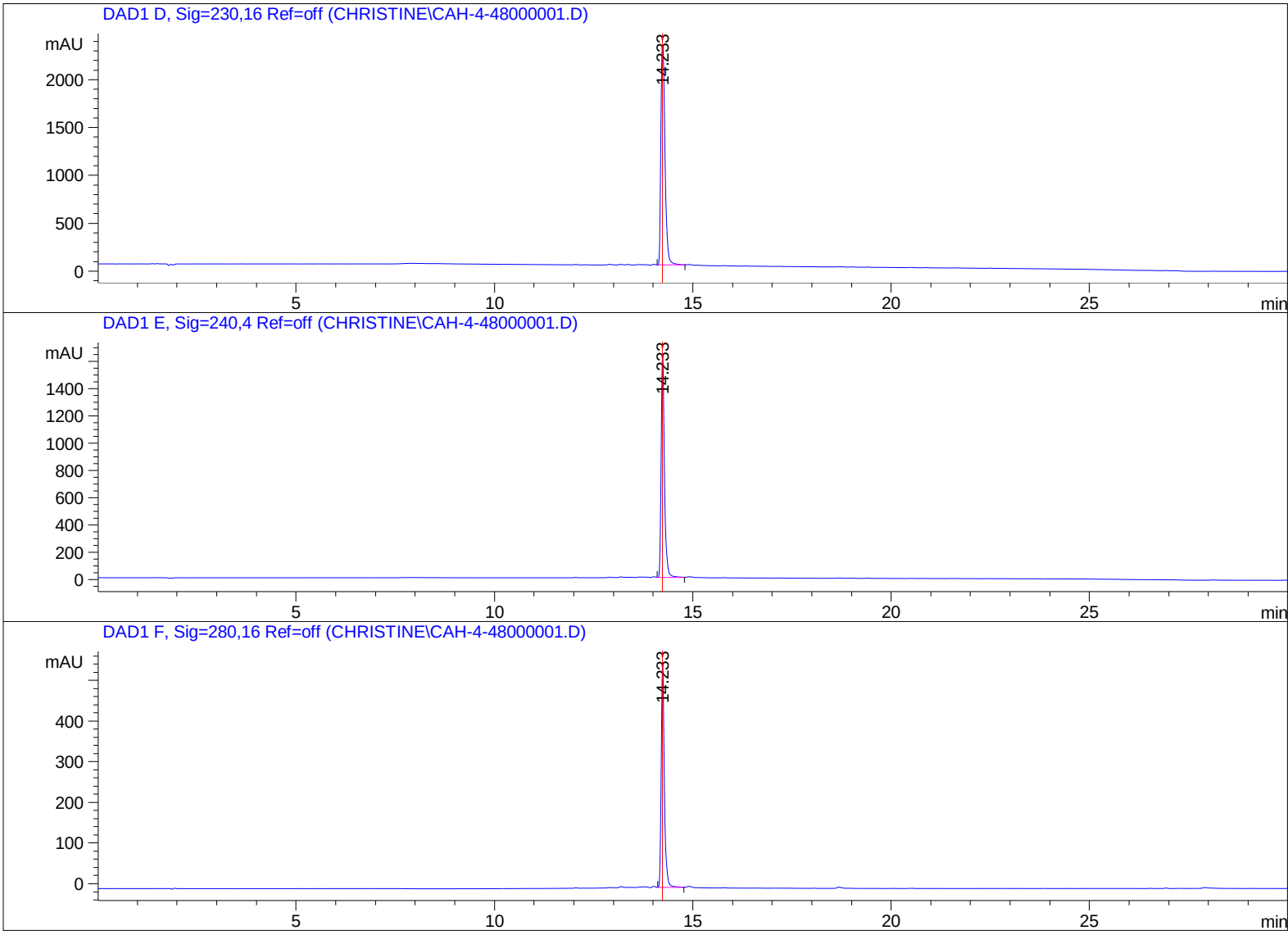

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Area Percent Report

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Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	VB	0.0823	1316.60486	238.95862	100.0000

Totals : 1316.60486 238.95862

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Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	VV	0.0833	1889.52271	337.36499	100.0000
Totals :				1889.52271	337.36499	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.234	VV	0.1789	2.45007e4	2218.16870	100.0000
Totals :				2.45007e4	2218.16870	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	VV	0.1151	1.67958e4	2302.03564	100.0000
Totals :				1.67958e4	2302.03564	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	VV	0.0897	9544.16406	1644.86255	100.0000
Totals :				9544.16406	1644.86255	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	VV	0.0826	3068.52466	554.10748	100.0000
Totals :				3068.52466	554.10748	

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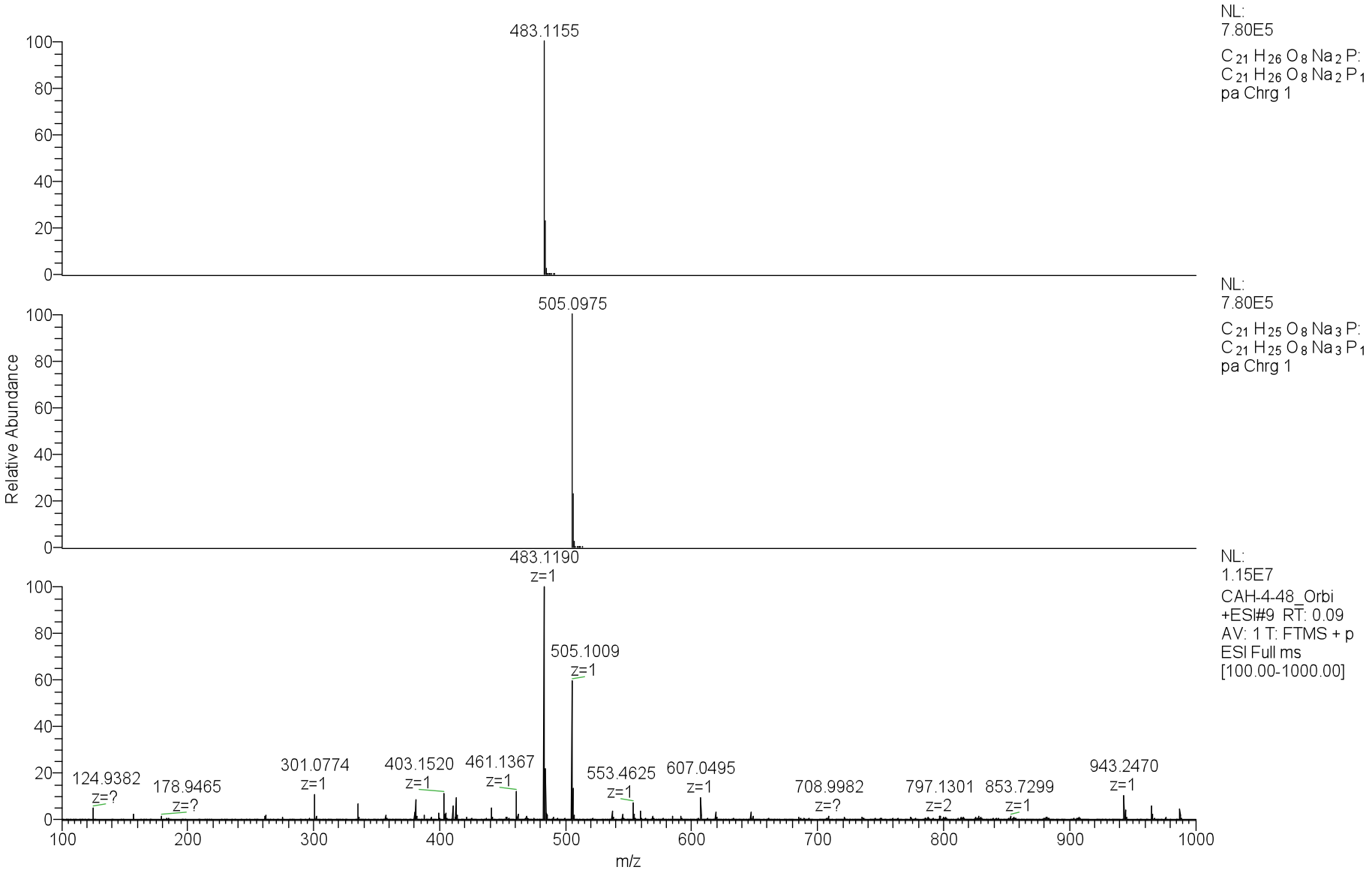
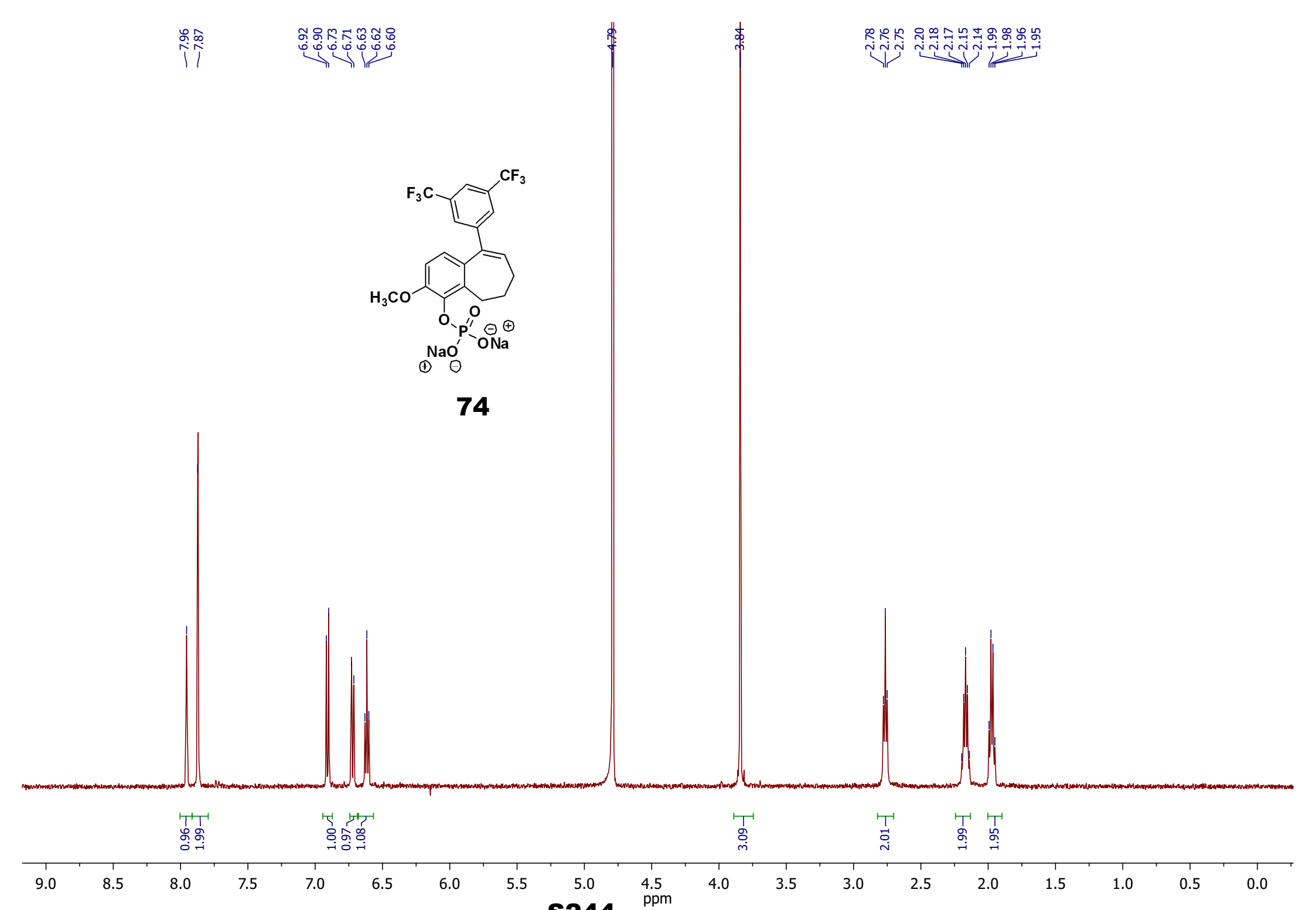
*** End of Report ***

HRMS of compound 73

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2.75
2.20
2.18
2.17
2.15
2.14
1.99
1.98
1.96
1.95
F3C
CF3
H3CO
O
O
P
O
Na
NaO
0.96
1.99
1.00
0.97
1.08
3.09
2.01
1.99
1.95
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8.0
7.5
7.0
6.5
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5.5
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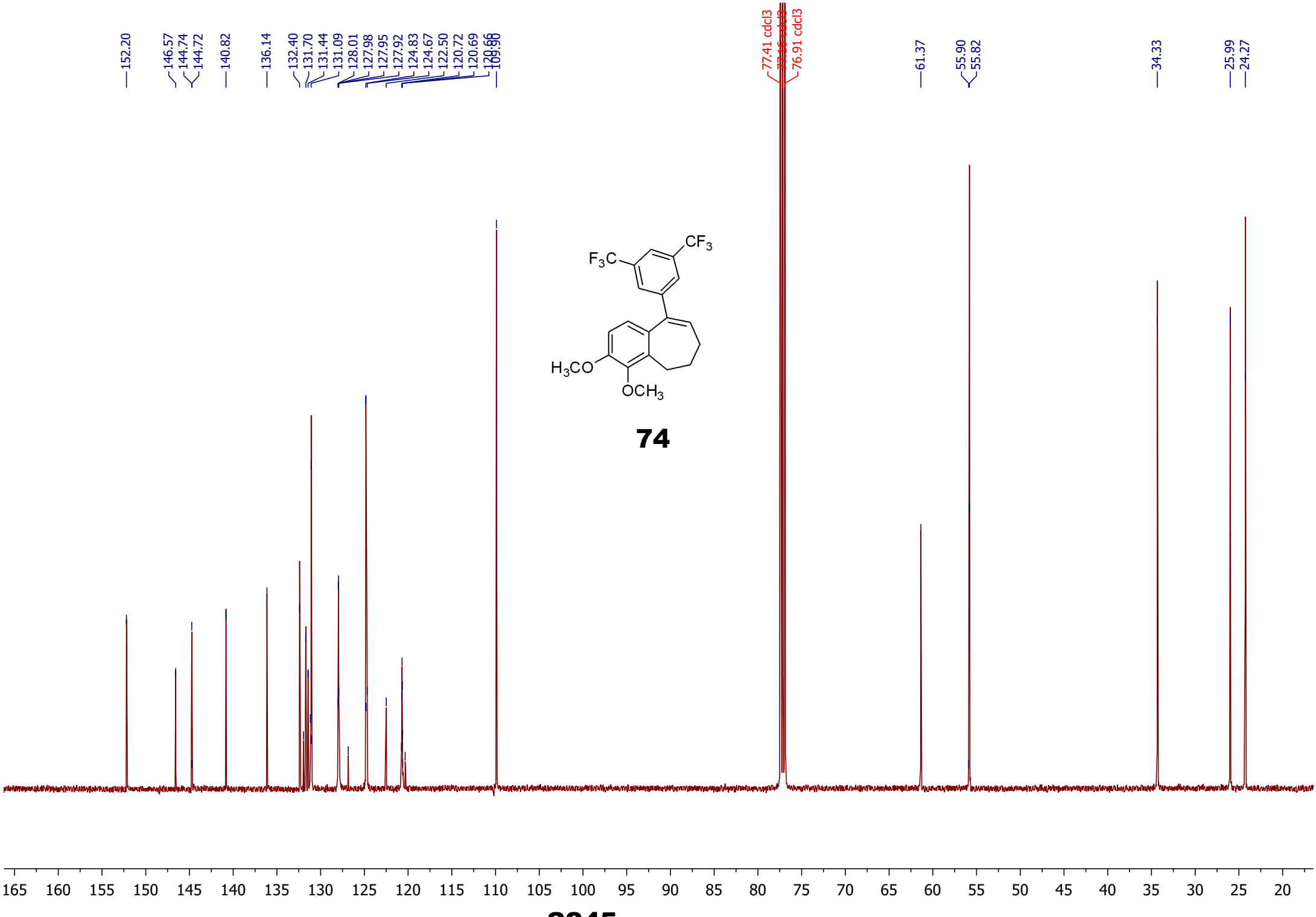
ppm

74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77.41 cdcl3
76.91 cdcl3
61.37
55.90
55.82
34.33
25.99
24.27
F_3C
CF_3
H_3CO
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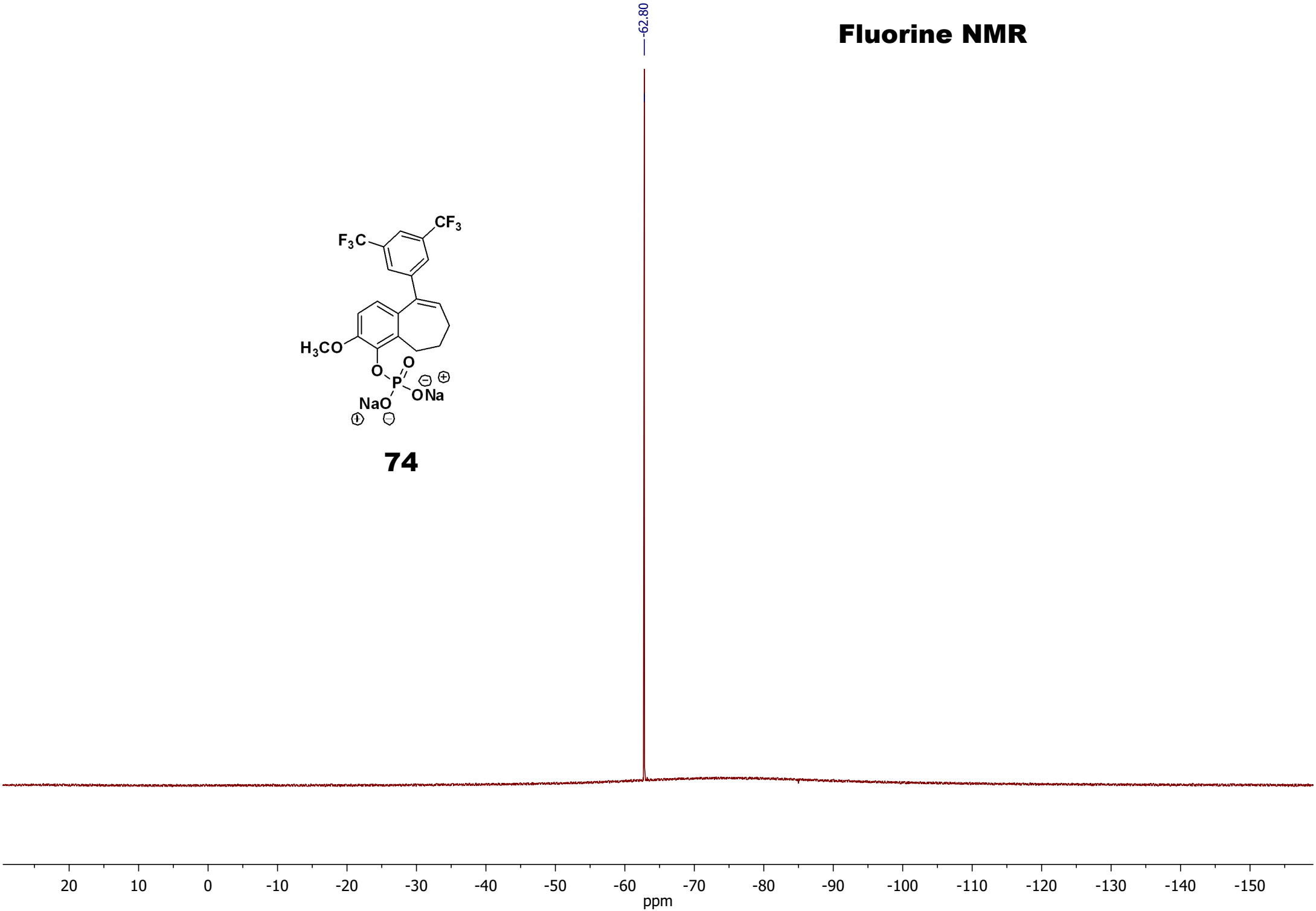
Fluorine NMR

74



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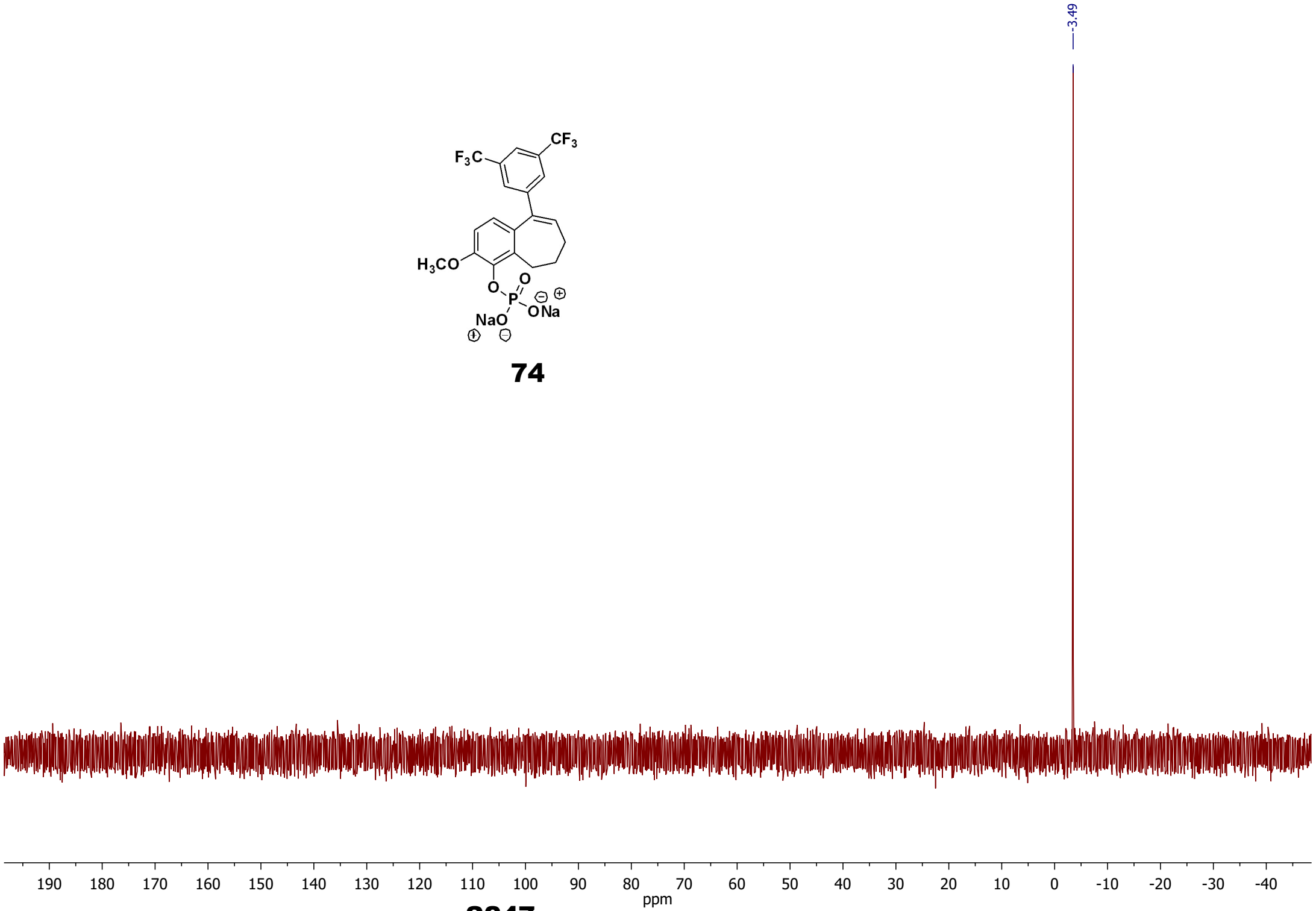
P
O
ONa
NaO

74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-40
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HPLC of compound 74

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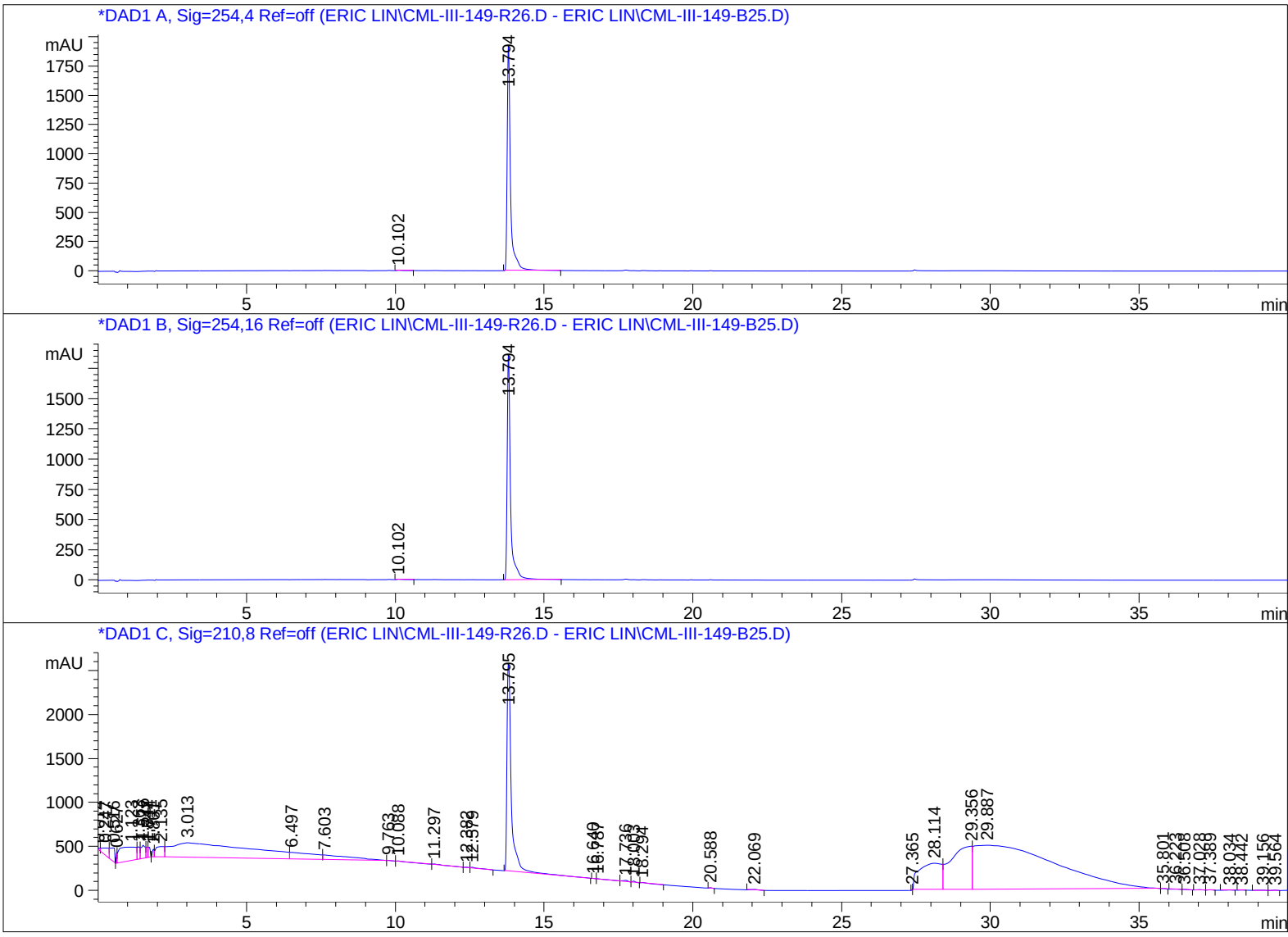

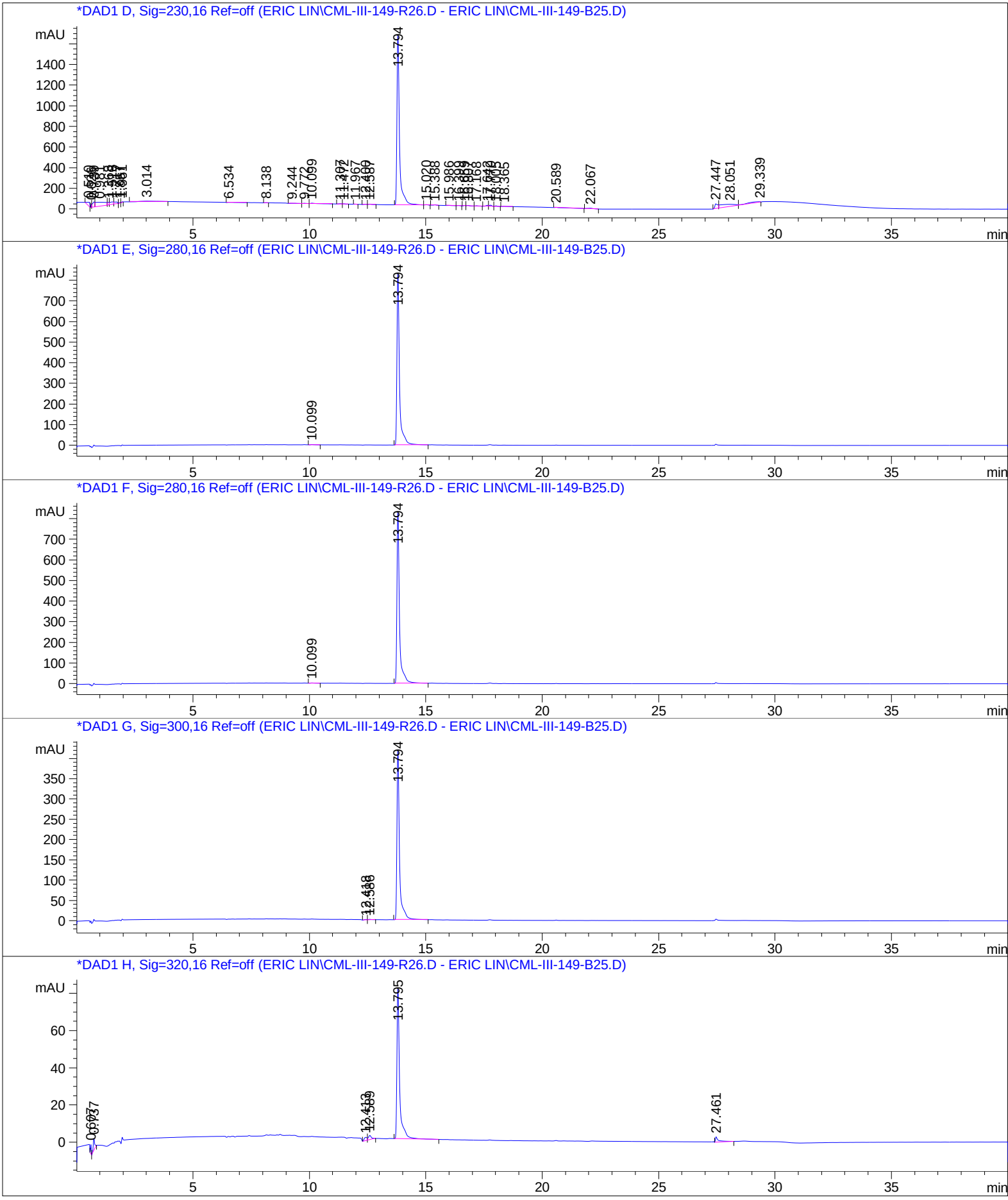
*DAD1 D, Sig=230,16 Ref=off (ERIC LIN\CML-III-149-R26.D - ERIC LIN\CML-III-149-B25.D)
mAU
13.794
3.014
6.534
8.138
9.244
10.099
20.589
22.067
27.447
28.051
29.339
min
*DAD1 E, Sig=280,16 Ref=off (ERIC LIN\CML-III-149-R26.D - ERIC LIN\CML-III-149-B25.D)
mAU
13.794
10.099
min
*DAD1 F, Sig=280,16 Ref=off (ERIC LIN\CML-III-149-R26.D - ERIC LIN\CML-III-149-B25.D)
mAU
13.794
10.099
min
*DAD1 G, Sig=300,16 Ref=off (ERIC LIN\CML-III-149-R26.D - ERIC LIN\CML-III-149-B25.D)
mAU
13.794
min
*DAD1 H, Sig=320,16 Ref=off (ERIC LIN\CML-III-149-R26.D - ERIC LIN\CML-III-149-B25.D)
mAU
13.795
27.461
min

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-III-149-R26.D
Sample Name: CML-III-149-r2

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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.102	VB	0.1632	33.33345	2.91925	0.2180
2	13.794	VB	0.1164	1.52597e4	1926.53333	99.7820
Totals :				1.52930e4	1929.45257	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

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1	10.102	VB	0.1638	32.29185	2.81660	0.2186
2	13.794	VB	0.1162	1.47398e4	1866.08594	99.7814
Totals :				1.47721e4	1868.90253	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.014	BV	0.1264	116.59129	15.36866	0.0538
2	0.247	VV	0.2472	1332.98865	78.86525	0.6147
3	0.516	VB	0.1533	1552.54041	154.45248	0.7159
4	0.627	BV	0.0341	268.13446	119.93959	0.1236
5	1.123	VV	0.5253	6208.21094	153.02040	2.8628
6	1.363	VV	0.0915	939.56226	141.51839	0.4333
7	1.516	VV	0.1314	1505.25952	149.60490	0.6941
8	1.647	VV	0.0561	475.18509	120.27156	0.2191
9	1.714	VB	0.0678	516.40662	116.22965	0.2381
10	1.861	BV	0.0728	400.88586	88.58305	0.1849
11	2.135	VV	0.3148	2428.47144	120.64979	1.1198

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-III-149-R26.D
Sample Name: CML-III-149-r2

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
12	3.013	VV	2.2150	3.12489e4	168.24092	14.4098
13	6.497	VV	0.6875	4282.35107	75.98831	1.9747
14	7.603	VB	0.7892	3460.69922	52.62601	1.5958
15	9.763	BV	0.1387	32.96905	3.29470	0.0152
16	10.088	VV	0.3678	200.97350	6.81970	0.0927
17	11.297	VB	0.3651	246.68819	8.53868	0.1138
18	12.382	BV	0.1599	44.04943	4.35936	0.0203
19	12.579	VB	0.2161	158.32291	9.61325	0.0730
20	13.795	BB	0.1546	2.43593e4	2356.12231	11.2329
21	16.640	BV	0.0861	8.61282	1.51916	3.972e-3
22	16.787	VV	0.3534	50.88361	1.79118	0.0235
23	17.736	VV	0.1681	152.08693	13.23410	0.0701
24	18.003	VB	0.0973	85.28777	13.21263	0.0393
25	18.294	BB	0.1673	79.26408	6.19386	0.0366
26	20.588	BB	0.0775	27.55946	5.59449	0.0127
27	22.069	BB	0.2297	115.85145	7.27799	0.0534
28	27.365	BB	0.0195	6.61201	5.81724	3.049e-3
29	28.114	BV	0.7154	1.57789e4	300.12476	7.2761
30	29.356	VV	0.7563	2.42025e4	491.68054	11.1606
31	29.887	VB	2.3219	9.61836e4	501.41739	44.3533
32	35.801	BB	0.1517	22.13400	2.31238	0.0102
33	36.223	BV	0.2609	55.02095	3.04299	0.0254
34	36.508	VB	0.1819	36.04284	3.00632	0.0166
35	37.028	BV	0.1866	53.60883	4.38545	0.0247
36	37.389	VB	0.1591	49.88113	5.14453	0.0230
37	38.034	VB	0.1951	36.97443	2.81737	0.0171
38	38.442	BB	0.1327	14.78003	1.82136	6.816e-3
39	39.156	BB	0.2580	71.35019	3.96372	0.0329
40	39.564	BB	0.2068	48.35715	4.00710	0.0223
Totals :				2.16858e5	5322.47151	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.510	BB	0.1227	281.79205	33.32289	1.5359
2	0.626	BV	0.0323	62.86551	30.14817	0.3426
3	0.739	VV	0.1042	377.07275	48.78299	2.0552
4	0.981	VV	0.3857	1086.44214	37.98767	5.9214
5	1.368	VV	0.0797	151.39351	26.08789	0.8251
6	1.518	VV	0.1373	272.69882	25.79583	1.4863
7	1.717	VB	0.1341	182.61948	18.04617	0.9953
8	1.857	BB	0.0617	27.95403	7.81287	0.1524
9	1.961	BB	0.0545	20.80817	5.99101	0.1134
10	3.014	BB	0.6050	267.75238	5.56837	1.4593

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-III-149-R26.D
Sample Name: CML-III-149-r2

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
11	6.534	BV	0.3767	57.15169	1.91202	0.3115
12	8.138	BB	0.0794	5.82987	1.14436	0.0318
13	9.244	BV	0.3169	31.18376	1.26792	0.1700
14	9.772	VV	0.1416	27.56984	2.68903	0.1503
15	10.099	VB	0.2091	71.74109	4.67413	0.3910
16	11.307	BV	0.1256	22.51918	2.69070	0.1227
17	11.472	VB	0.1012	16.75818	2.46791	0.0913
18	11.967	VB	0.0976	9.49871	1.50531	0.0518
19	12.400	BV	0.1513	21.66013	2.31095	0.1181
20	12.587	VB	0.1309	38.09042	4.15847	0.2076
21	13.794	VV	0.1147	1.28217e4	1649.89600	69.8822
22	15.020	VB	0.1292	30.51363	3.66408	0.1663
23	15.388	BB	0.1131	32.63912	4.37035	0.1779
24	15.986	BV	0.2081	21.80622	1.47846	0.1189
25	16.399	VB	0.1185	27.55081	3.40261	0.1502
26	16.659	BV	0.0876	26.69537	4.74829	0.1455
27	16.807	VB	0.1090	40.28115	5.52840	0.2195
28	17.168	BB	0.1287	25.67058	2.86103	0.1399
29	17.642	BV	0.1179	75.71060	9.61090	0.4126
30	17.770	VV	0.1174	90.70266	11.33053	0.4944
31	18.005	VB	0.1042	39.87554	5.66185	0.2173
32	18.365	BB	0.1760	27.57606	2.03578	0.1503
33	20.589	BB	0.1915	38.05714	2.58892	0.2074
34	22.067	BB	0.2146	74.61647	4.87451	0.4067
35	27.447	BV	0.1212	421.20547	46.70127	2.2957
36	28.051	VV	0.6665	1109.79724	22.15031	6.0487
37	29.339	VB	1.1831	409.78696	4.76587	2.2335
Totals :				1.83476e4	2050.03383	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.099	BB	0.1567	12.72469	1.13608	0.1970
2	13.794	BB	0.1140	6446.57959	835.78650	99.8030
Totals :				6459.30428	836.92258	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.099	BB	0.1567	12.72469	1.13608	0.1970
2	13.794	BB	0.1140	6446.57959	835.78650	99.8030
Totals :				6459.30428	836.92258	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.418	BV	0.1436	13.22460	1.48978	0.4033
2	12.586	VB	0.1478	14.48388	1.36451	0.4417
3	13.794	BB	0.1144	3251.21021	419.63831	99.1550
Totals :				3278.91869	422.49259	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.607	BB	0.0301	9.38681	4.94999	1.2174
2	0.737	BB	0.0659	29.25878	6.58113	3.7946
3	12.413	BV	0.1398	15.82891	1.84999	2.0529
4	12.589	VB	0.1340	22.26375	2.36228	2.8874
5	13.795	BB	0.1197	664.22479	80.99686	86.1439
6	27.461	BB	0.1347	30.10082	2.90944	3.9038
Totals :				771.06386	99.64970	

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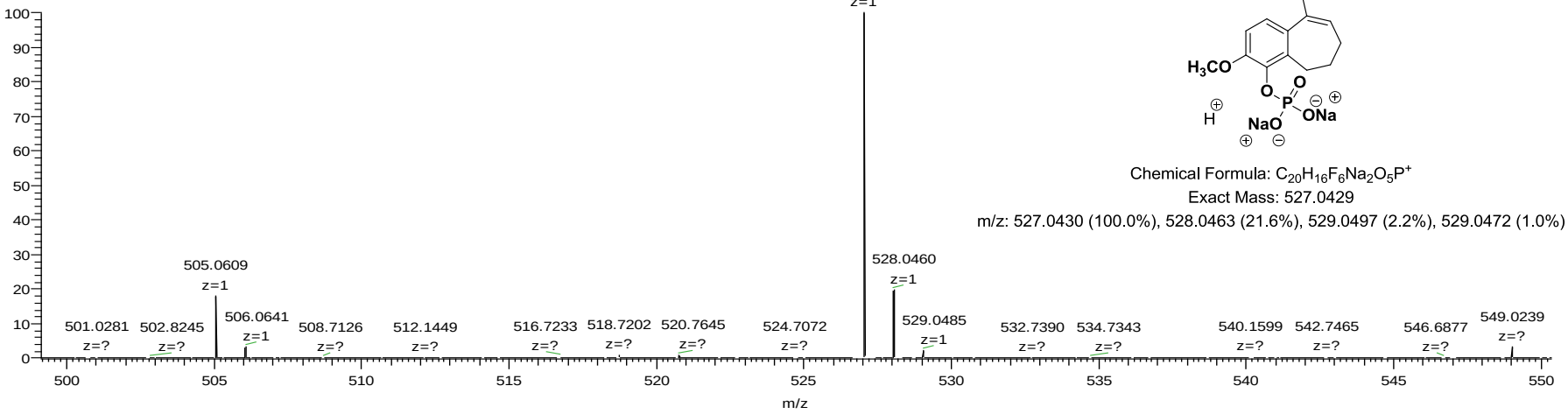
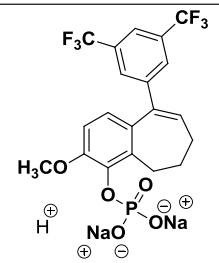
*** End of Report ***

HRMS of compound 74

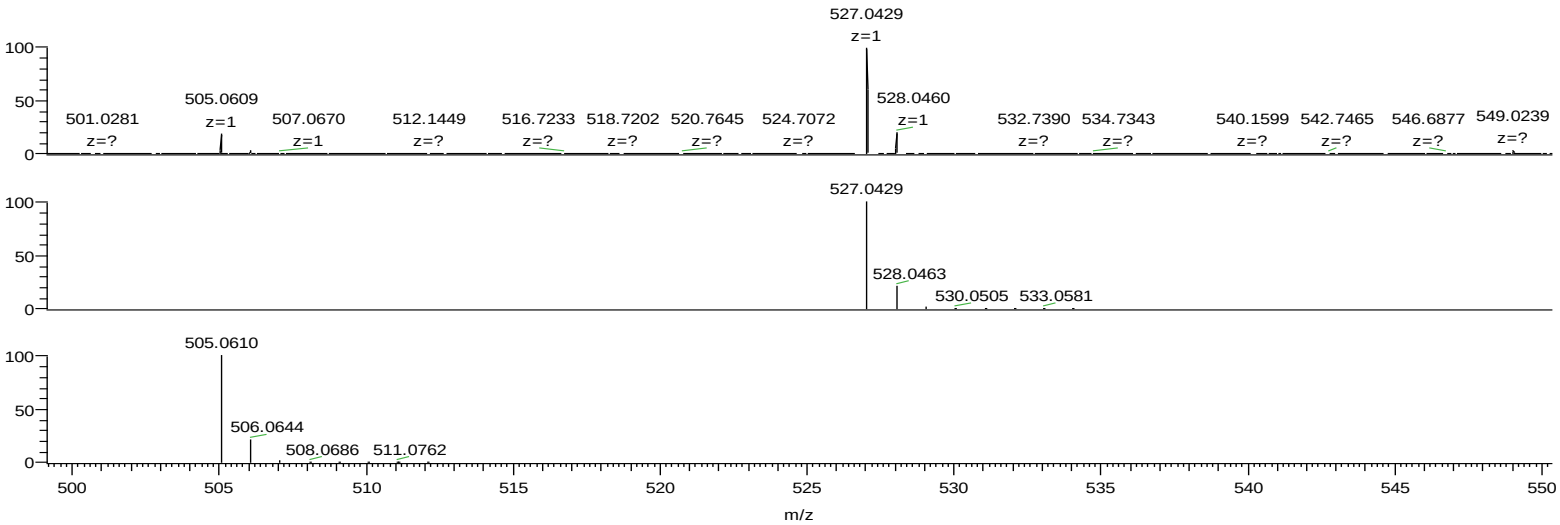
C:\Xcalibur\...\CML_III_141_Orbi_+ESI 7/23/2014 4:29:51 PM CML_III_141

CML_III_141_Orbi_+ESI #12 RT: 0.09 AV: 1 NL
T: FTMS + p ESI Full ms [100.00-1000.00]





Chemical Formula: $C_{20}H_{16}F_6Na_2O_5P^+$
Exact Mass: 527.0429
m/z: 527.0430 (100.0%), 528.0463 (21.6%), 529.0497 (2.2%), 529.0472 (1.0%)



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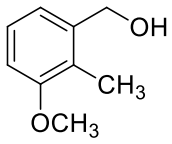
1.82E7
CML_III_141_Orbi_+ESI#12 RT: 0.09 AV: 1 T: FTMS + p ESI Full ms [100.00-1000.00]

NL:
7.95E5
$C_{20}H_{15}F_6Na_2O_5P$ +H:
$C_{20}H_{16}F_6Na_2O_5P_1$
pa Chrg 1

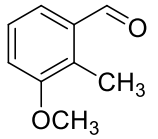
NL:
7.95E5
$C_{20}H_{16}F_6NaO_5P$ +H:
$C_{20}H_{17}F_6Na_1O_5P_1$
pa Chrg 1

Alternative Synthesis for Compound **30**



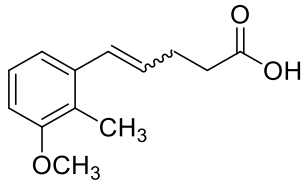
3-Methoxy-2-methylbenzoic acid

To a well-stirred and pre-cooled 0° C solution of 3-methoxy-2-methylbenzoic acid (500 mg, 3 mmol) in THF (150 mL), $LiAlH_4$ (1.13 mL, 2.0 M) was added, and the reaction was stirred warming from 0° C to room temperature over 12 h. The reaction mixture was quenched with 7 mL of 20% H_2O in THF added dropwise, and then 10 mL of 15-20% NaOH was added. After filtering through Celite®, the filtrate was extracted with EtOAc (3 x 50 mL). The combined organic phase was dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B → 60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording the alcohol (0.40 g, 2.67mmol, 89%) as a white powder. ^{1}H NMR (CDCl3, 500 MHz) δ 7.17 (1H, dd, *J* = 5.0 Hz, 10.0 Hz), 6.97 (1H, d, *J* = 5.0 Hz), 6.82 (1H, d, *J* = 10.0 Hz), 4.66 (2H, s), 3.83 (3H, s), 2.21 (3H, s). ^{13}C NMR (CDCl3, 125 MHz) δ 157.7, 139.9, 126.3, 124.7, 120.0, 109.8, 63.5, 55.6, 10.8.



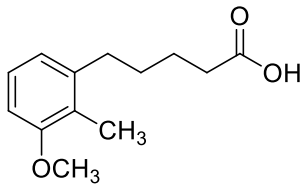
3-Methoxy-2-methylbenzaldehyde

To a well-stirred pyridinium chlorochromate (PCC) in CH_2Cl_2 (40 mL) solution, (3-methoxy-2-methylphenyl)methanol (3.22 g, 21.2 mmol) dissolved in CH_2Cl_2 (25 mL) was slowly added. The reaction mixture was stirred at room temperature for 12 h. The mixture was filtered through Celite®, and the Celite® was washed thoroughly with CH_2Cl_2 (2 x 30 mL). The filtrate was concentrated under reduced pressure and purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B → 60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording the aldehyde (2.70 g, 18 mmol, 85%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.31 (1H, s), 7.41 (1H, d, *J* = 10.0 Hz), 7.30 (1H, dd, *J* = 5.0 Hz, 10.0 Hz), 7.06 (1H, d, *J* = 10.0 Hz), 3.86 (3H, s), 2.53 (3H, s). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 192.7, 158.1, 135.1, 129.6, 126.6, 123.0, 115.2, 55.9, 10.4.



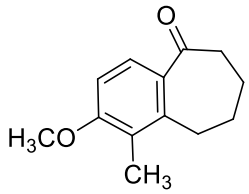
5-(3′-methoxy-2′-methylphenyl)pent-4-enoic acid

A mixture of 3-(carboxypropyl)triphenylphosphonium bromide (7.8 g, 18.2 mmol) and potassium *tert*-butoxide (4.5 g, 40.1 mmol) in THF (150 mL) was stirred for 1 h at room temperature. A 3-methoxy-2-methylbenzaldehyde (2.70 g, 18 mmol) solution in THF (20 mL) was added dropwise to the reaction mixture. The reaction was quenched with 2 M HCl (30mL), then extracted with EtOAc (3 x 50 mL). The organic extract was washed with brine, dried with Na_2SO_4, concentrated under reduced pressure, and then purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B → 60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording 5-(3′-methoxy-2′-methylphenyl)pent-4-enoic acid (*E* & *Z*) (3.04 g, 13.8 mmol, 75%) as a pale yellow oil. NMR data was collected after the next step.



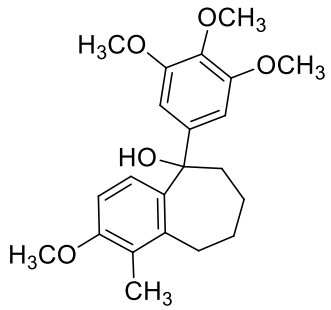
5-(3′-methoxy-2′-methylphenyl)pentanoic acid

5-(3′-Methoxy-2′-methylphenyl)pent-4-enoic acid (3.04 g, 13.8 mmol) was mixed with Pd/C powder quickly, and the reaction vessel was purged with nitrogen. Methanol (40 mL) was slowly added, and the reaction vessel was purged again with nitrogen. The reaction was stirred for 12 h at room temperature. The suspension was filtered through Celite®, and the Celite® was rinsed with EtOAc. The filtrate (combined MeOH and EtOAc) was concentrated under reduced pressure and purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B→60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording 5-(3-methoxy-2-methylphenyl)pentanoic acid (2.1 g, 9.6 mmol 70%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.42 (1H, dd, *J* =10.0 Hz, 5.0 Hz), 7.15 (1H, d, *J* = 5.0 Hz), 7.05 (1H, d, *J* =10.0 Hz), 4.15 (3H, s), 2.97 (2H, m), 2.73 (2H, t, *J* =5.0 Hz), 2.51 (3H, s), 2.06 (2H, m), 1.96 (2H, m). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 180.1, 157.7, 141.6, 125.9, 124.6, 121.6, 107.9, 55.6, 34.0, 33.2, 29.9, 24.6, 11.2.



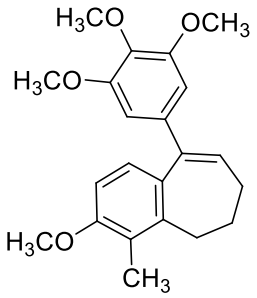
1,2-Dimethoxy-6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-one

Eaton's reagent (42 mL) was added to 5-(3´-methoxy-2´-methylphenyl)pentanoic acid (2.1 g, 9.6 mmol). The mixture was sonicated until dissolved and stirred at room temperature for 12 h. Ice was poured into the reaction flask, and the mixture was neutralized with sat. $NaHCO_3$ and extracted with EtOAc. The organic layer was dried with Na_2SO_4, concentrated under reduced pressure, and purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B→60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording the ketone product (1.87 g, 9.1 mmol, 94%) as a pale yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.53 (1H, d, J = 10.0 Hz), 6.76 (1H, d, J = 10.0 Hz), 3.85 (3H, s), 2.88 (2H, m), 2.65 (2H, m), 2.21 (3H, s), 1.81 (2H, m), 1.73 (2H, m). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 206.5, 160.5, 140.5, 132.7, 127.5, 123.9, 107.8, 55.6, 40.5, 27.1, 24.2, 20.6, 11.2.



2-Methoxy-1-methyl-5-(3´,4´,5´-trimethoxyphenyl)-6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-ol

5-bromo-1,2,3-trimethoxybenzene (3.39 g, 13.7 mmol) was dissolved in THF (40 mL) and cooled to -78 °C. *n*-BuLi (5.49 mL, 2.5 M) was added dropwise, and the mixture was stirred at -78° C. After 1 h, the ketone (1.87 g, 9.1 mmol) in THF was added dropwise to the reaction flask. The reaction was allowed to stir for 12 h warming to room temperature. The reaction was quenched with water (100 mL) and extracted with EtOAc. The organic layer was dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was further purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 10% A/ 93% B (3 CV), 10% A/ 93% B → 60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280nm] affording the tertiary alcohol (2.38 g, 6.4 mmol, 70%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.35 (1H, d, J = 10.0 Hz), 6.71 (1H, d, J = 10.0 Hz), 6.53 (2H, s), 3.84 (3H, s), 3.83 (3H, s), 3.75 (6H, s), 2.97 (2H, m), 2.55 (2H, m), 2.22 (3H, s), 1.84, (2H, m), 1.30, (2H, m). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 156.7, 153.0, 142.4, 140.7, 137.6, 137.2, 125.3, 124.0, 107.2, 104.2, 80.0, 60.8, 56.1, 55.4, 41.4, 28.5, 26.1, 25.8, 11.9.



3-Methoxy-4-methyl-9-(3′,4′,5′-trimethoxyphenyl)-6,7-dihydro-5*H*-benzo[7]annulene
The tertiary alcohol (2.38 g, 6.4 mmol) was dissolved in acetic acid (15 mL) and stirred for 6 h. The reaction was quenched with water (100 mL) and then extracted with EtOAc. The organic phase was washed with brine, dried with Na_2SO_4, concentrated and purified by flash chromatography using a prepacked 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7% A/ 93% B (3 CV), 7% A/ 93% B → 60% A/ 40% B (10 CV), 60% A/ 40% B (1 CV); flow rate: 25 mL/min; monitored at 254 and 280 nm] affording **26** (1.78 g, 5.0 mmol, 78%) as a white powder. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.86 (1H, d, J = 10.0 Hz), 6.70 (1H, d, J =10.0 Hz), 6.52 (2H, s), 6.32 (1H, t, J = 7.5 Hz), 3.86 (3H, s), 3.84 (3H, s), 3.80 (6H, s), 2.68 (2H, t, J = 6.5 Hz), 2.29 (3H, s), 2.12 (2H, p, J = 7.0 Hz), 1.91 (2H, q, J = 7.5 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 156.5, 152.8, 143.5, 141.7, 138.6, 137.3, 133.0, 127.4, 126.5, 123.2, 107.4, 105.3, 60.9, 56.1, 55.5, 34.0, 27.7, 25.5, 11.8. HRMS: Obsvd 355.1906 [M+H]+, calcd for $C_{22}H_{27}O_5$: 355.1904. HPLC (by method B): 19.87 min.

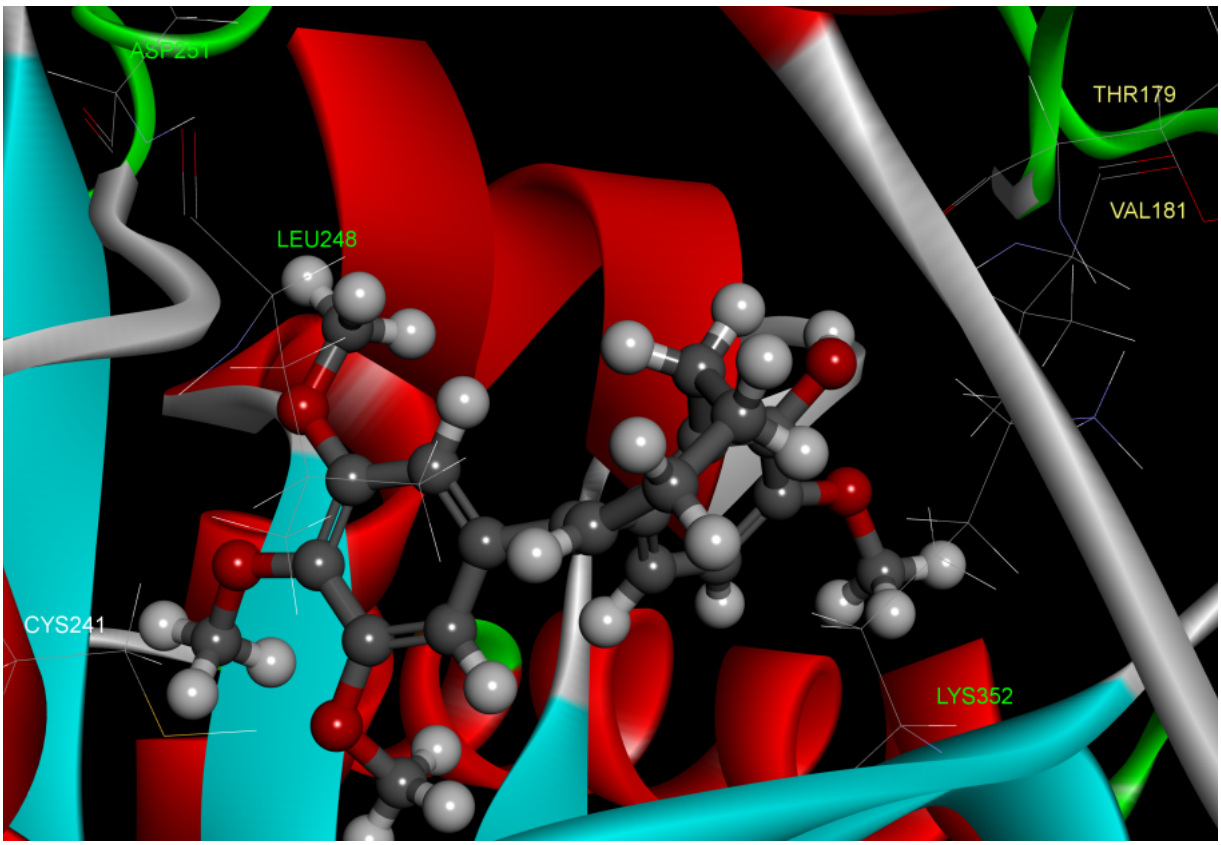
Compound	Inhibition of tubulin polymerization IC_{50} (μM) ± SD	% Inhibition of colchicine binding ± SD	GI_{50} (μM) SRB assay[a]		
			SK-OV-3	NCI-H460	DU-145
CA4	1.0[b]	84 ± 3 (1 μM), 98 ± 0.007 (5 μM)	0.00455 ± 0.00211	0.00223[c]	0.00327 ± 0.00215[c]
CA4P	>40[b]	nr	0.00119 ± 0.00124	0.00194[c]	0.00323 ± 0.00147[c]
KGP18	1.4[d]	nr	0.0000543[e]	0.0000418[e]	0.0000249[e]
28	>20	nr	32.7 ± 3.92	37.5 ± 4.21	89.3 ± 30.7
29	1.0 ± 0.02	37 ± 5 (1 μM), 72 ± 0.8 (5 μM)	0.0516 ± 0.0315	0.0527 ± 0.0184	0.0619 ± 0.00509
30	1.6 ± 0.2	65 ± 0.6 (5 μM)	0.330 ± 0.00624	0.422 ± 0.0104	0.644 ± 0.193
31	>20	nr	0.568 ± 0.0718	0.763 ± 0.130	1.51 ± 0.741
32	>20	nr	2.96 ± 0.804	3.32 ± 0.459	6.03 ± 0.0974
33	>20	nr	11.5 ± 5.87	16.1 ± 0.212	12.2 ± 7.59
34	>20	nr	31.1 ± 6.20	25.5 ± 4.01	52.1 ± 2.96
35	3.1 ± 0.03	30 ± 4 (5 μM), 56 ± 4 (50 μM)	0.277 ± 0.294	0.593 ± 0.109	0.708 ± 0.343
36	>20	nr	20.5 ± 13.6	33.4 ± 2.41	48.3 ± 38.3
37	>20	nr	40.7 ± 11.6	57.7 ± 19.1	68.7 ± 18.1
61	>20	nr	6.96 ± 0.503	10.5 ± 0.768	26.2 ± 7.83
62	1.2 ± 0.007	36 ± 5 (1 μM), 69 ± 3 (5 μM)	0.0432 ± 0.00826	0.120 ± 0.0179	0.0562 ±0.0269
64	>20	nr	0.557 ± 0.0358	0.652 ± 0.0543	4.40 ± 1.97
65	3.8 ± 0.3	8.5 ± 4 (5 μM), 37 ± 5 (50 μM)	4.81 ± 1.55	4.39 ± 1.36	4.92 ± 0.267
66	>20	nr	16.8 ± 9.60	25.0 ± 3.09	21.8 ± 0.607
67	7.4 ± 0.06	nr	18.4 ± 19.3	10.6 ± 5.26	8.59 ± 6.32
68	2.7 ± 0.1	27 ± 5 (5 μM)	0.527 ± 0.00634	0.647 ± 0.0160	1.02 ± 0.100
70	7.7 ± 0.2	nr	0.346 ± 0.127	0.691 ± 0.219	1.53 ± 1.02

71	11 ± 0.4	nr	3.53 ± 0.270	4.24 ± 0.208	7.54 ± 5.49
72	0.70 ± 0.1	21 ± 0.9 (1 μM), 67 ± 0.6 (5 μM)	0.408 ± 0.0883	0.141 ± 0.153	0.570 ± 0.147
73	>20	nr	0.357 ± 0.119	0.145 ± 0.161	0.753 ± 0.246
74	>20	nr	17.2 ± 4.94	16.3 ± 1.17	17.5 ± 1.55

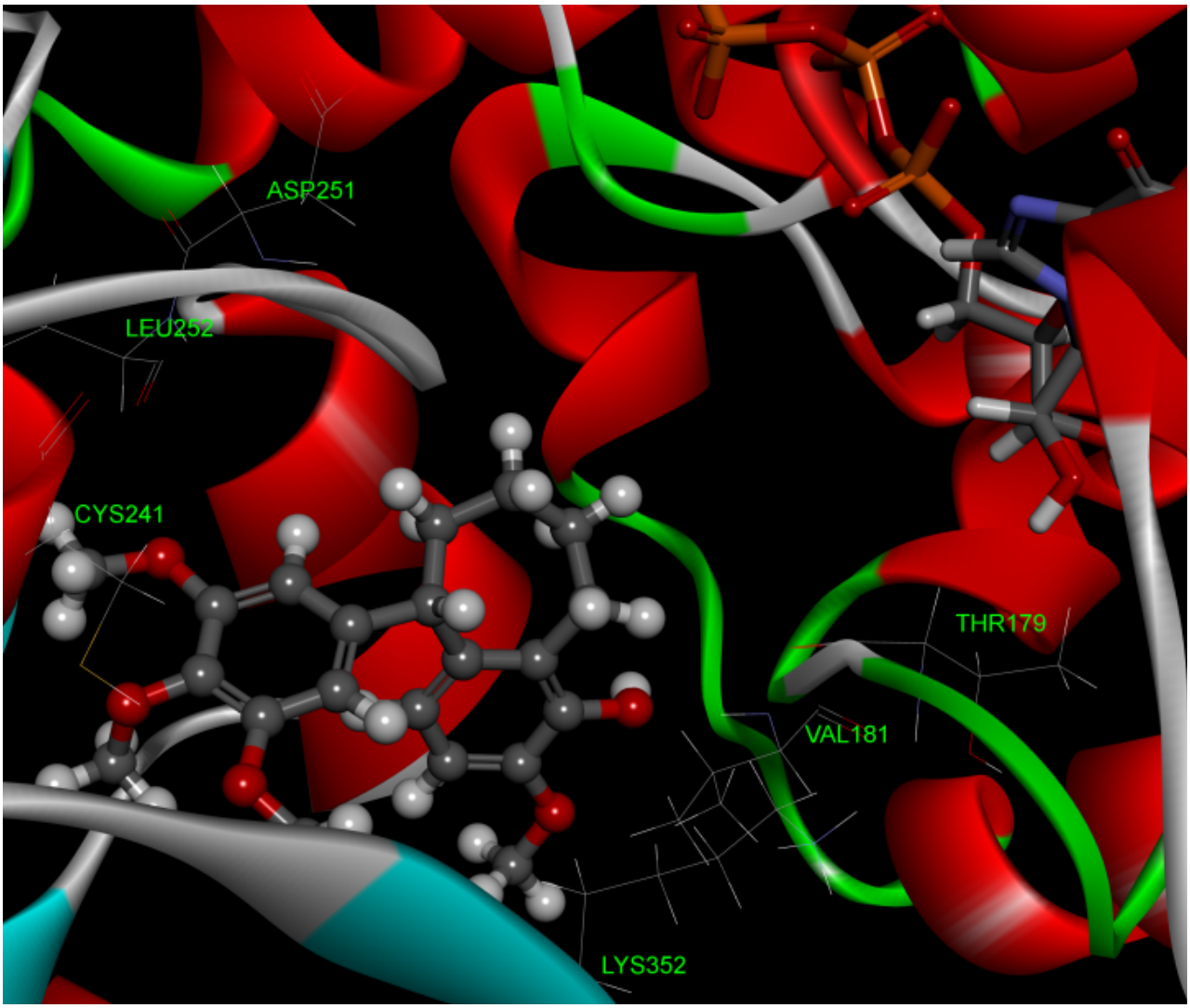
Molecular docking

Discovery Studio Client 4.5 (Accelrys) was used to carry out molecular docking studies on several analogues that were active inhibitors of tubulin polymerization (compounds **29**, **62** and **72**), the lead benzosuberene compound **KGP18**, colchicine, and compound **33** (IC_{50} > 20 μM in the tubulin polymerization assay). The X-ray structure of of *N*-deacetyl-*N*-(2-mercaptoacetyl)-colchicine (DAMA-colchicine) in the structure co-crystallized with tubulin (1SA0) was the starting structure for these studies. The protein was prepared and DAMA-colchicine was removed, and then docked (CDocker) to validate the docking procedure and parameters. There was excellent agreement between the docked and X-ray crystal structure of the ligand bound to tubulin.

The trimethoxyphenyl ring of colchicine, **KGP18** and all three active analogues was docked in a similar position to that of the trimethoxyphenyl moiety of *N*-deacetyl-*N*-(2-mercaptoacetyl)-colchicine in the structure co-crystallized with tubulin, and very close to the Cys241 residue of the beta subunit.

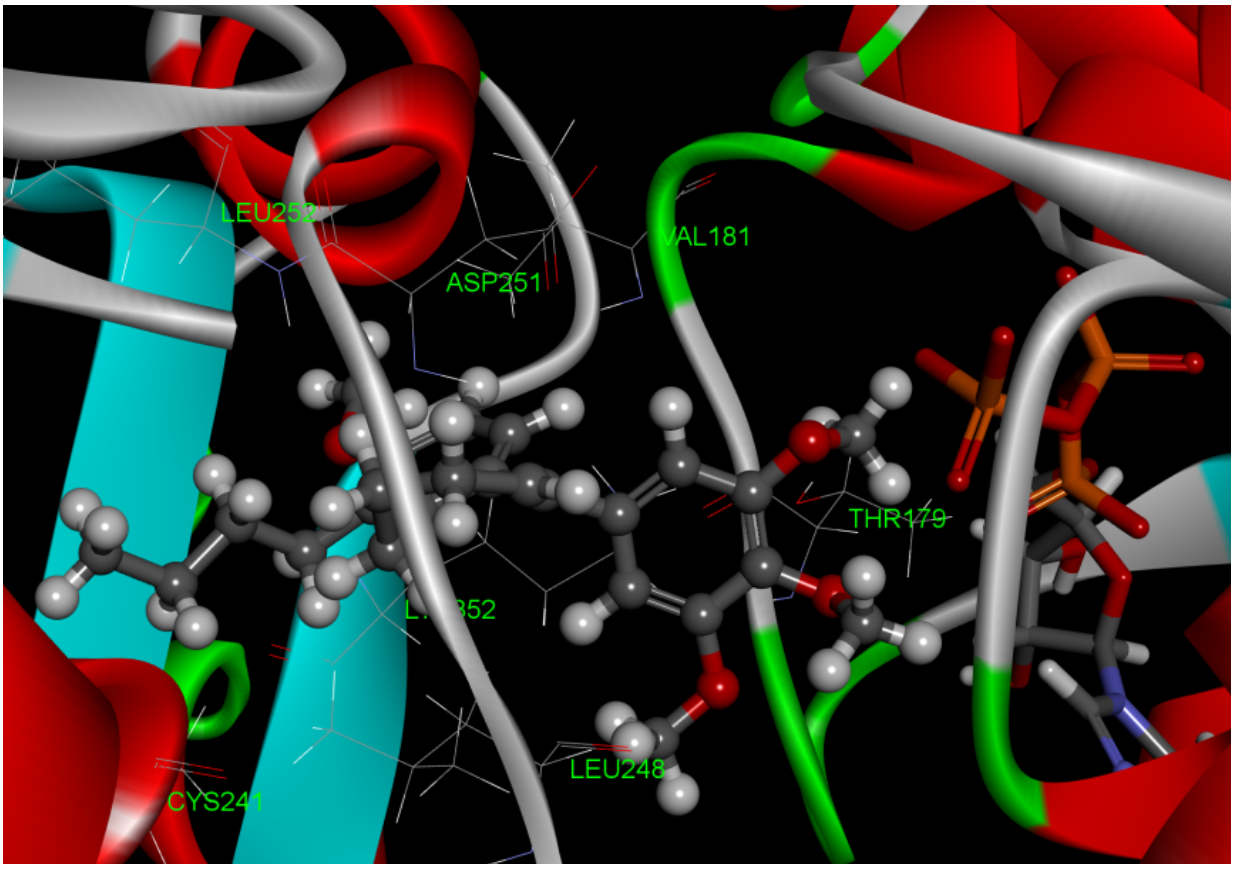


KGP18 (ball and stick) docked at the DAMA-colchicine binding site of tubulin (1SA0). THR179 and VAL181 (on the right side of the figure) are part of the alpha subunit. All other labeled residues are found in the tubulin beta subunit.



Compound **72** (ball and stick) docked at the DAMA-colchicine binding site of tubulin (1SA0). THR179 and VAL181 (on the right side of the figure) are part of the alpha subunit. All other labeled residues are found in the tubulin beta subunit.

In contrast, docking placed multiple top conformations of compound **33** (IC_{50} > 20 μM in the tubulin polymerization assay) with its trimethoxyphenyl ring outside of this pocket. In one top conformation shown below, the butyl chain is placed in the location of the trimethoxyphenyl ring of DAMA-colchicine, and the trimethoxyphenyl moiety of compound **33** is displaced toward the alpha subunit of tubulin.



Compound **33** (ball and stick) docked at the DAMA-colchicine binding site of tubulin (1SA0). THR179 and VAL181 (on the right side of the figure) are part of the alpha subunit. All other labeled residues are found in the tubulin beta subunit.

Insight into tubulin regulation from a complex with colchicine and a stathmin-like domain.
Ravelli, R.B., Gigant, B., Curmi, P.A., Jourdain, I., Lachkar, S., Sobel, A., Knossow, M.
(2004) Nature **428:** 198-202
PubMed: 15014504
DOI: 10.1038/nature02393