Supporting Information

Design, Synthesis and Anti-RNA Virus Activity of 6′-Fluorinated-aristeromycin Analogues

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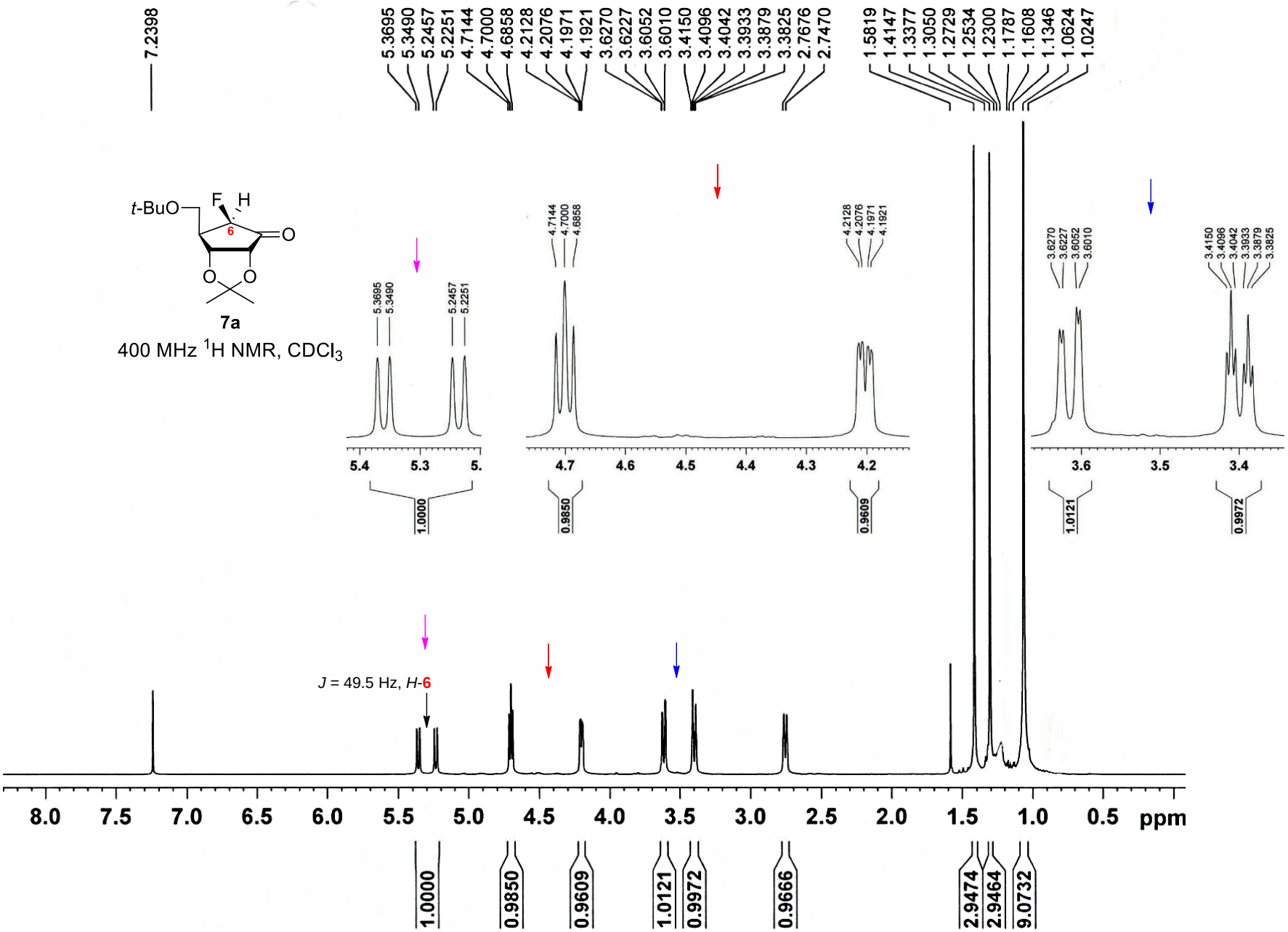
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1.2534
1.2300
1.1787
1.1608
1.1346
1.0624
1.0247
t-BuO
F
H
6
O
O
O
7a
400 MHz 1H NMR, CDCl3
J = 49.5 Hz, H-6
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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0.9666
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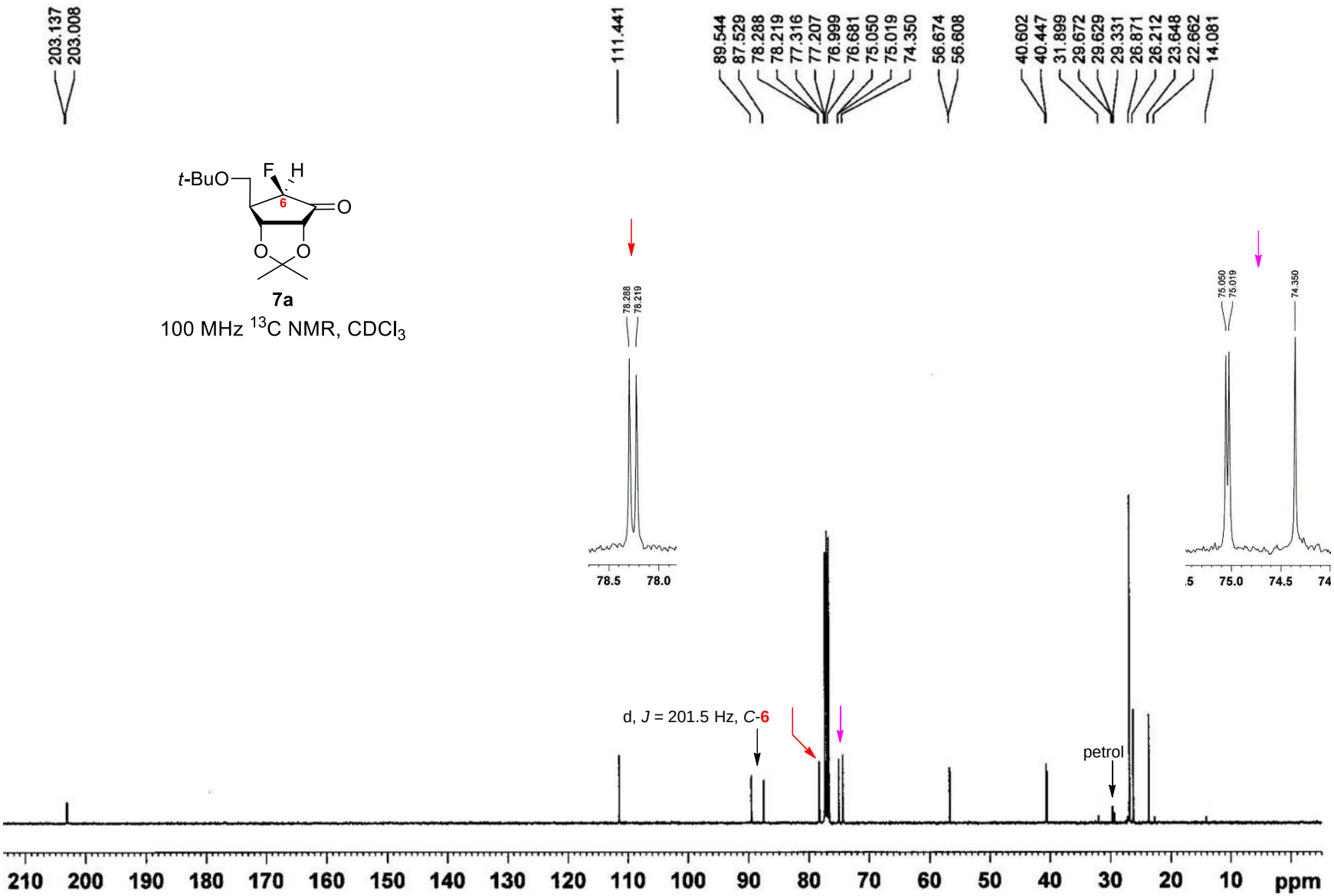
2.9464
9.0732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77.207
76.999
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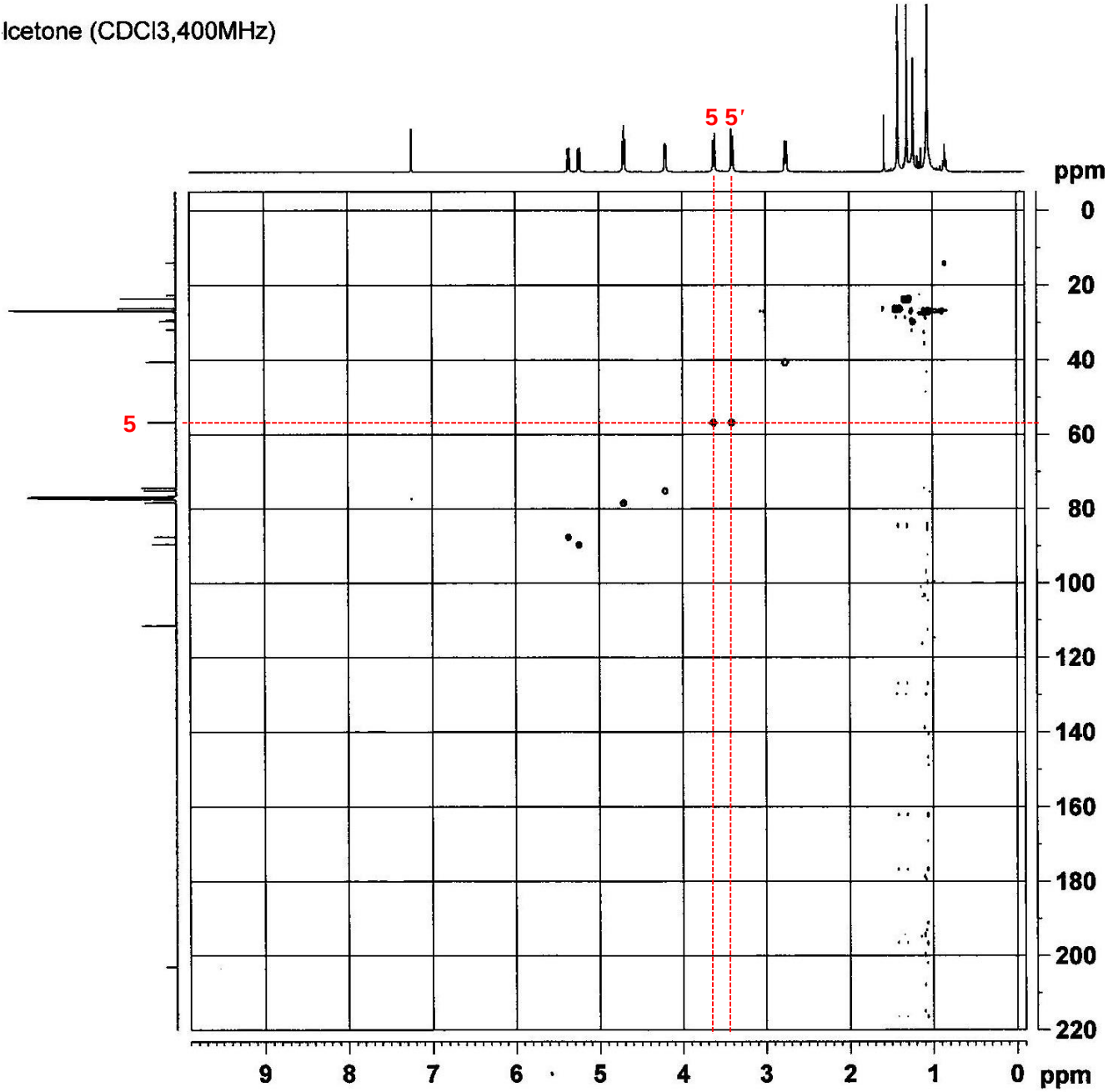
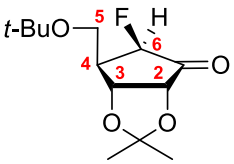
75.019
74.350
56.674
56.608
40.602
40.447
31.899
29.672
29.629
29.331
26.871
26.212
23.648
22.662
14.081
t-BuO
F
H
6
O
O
O
7a
100 MHz 13C NMR, CDCl3
78.288
78.219
78.5
78.0
75.050
75.019
74.350
75.0
74.5
d, J = 201.5 Hz, C-6
petrol
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

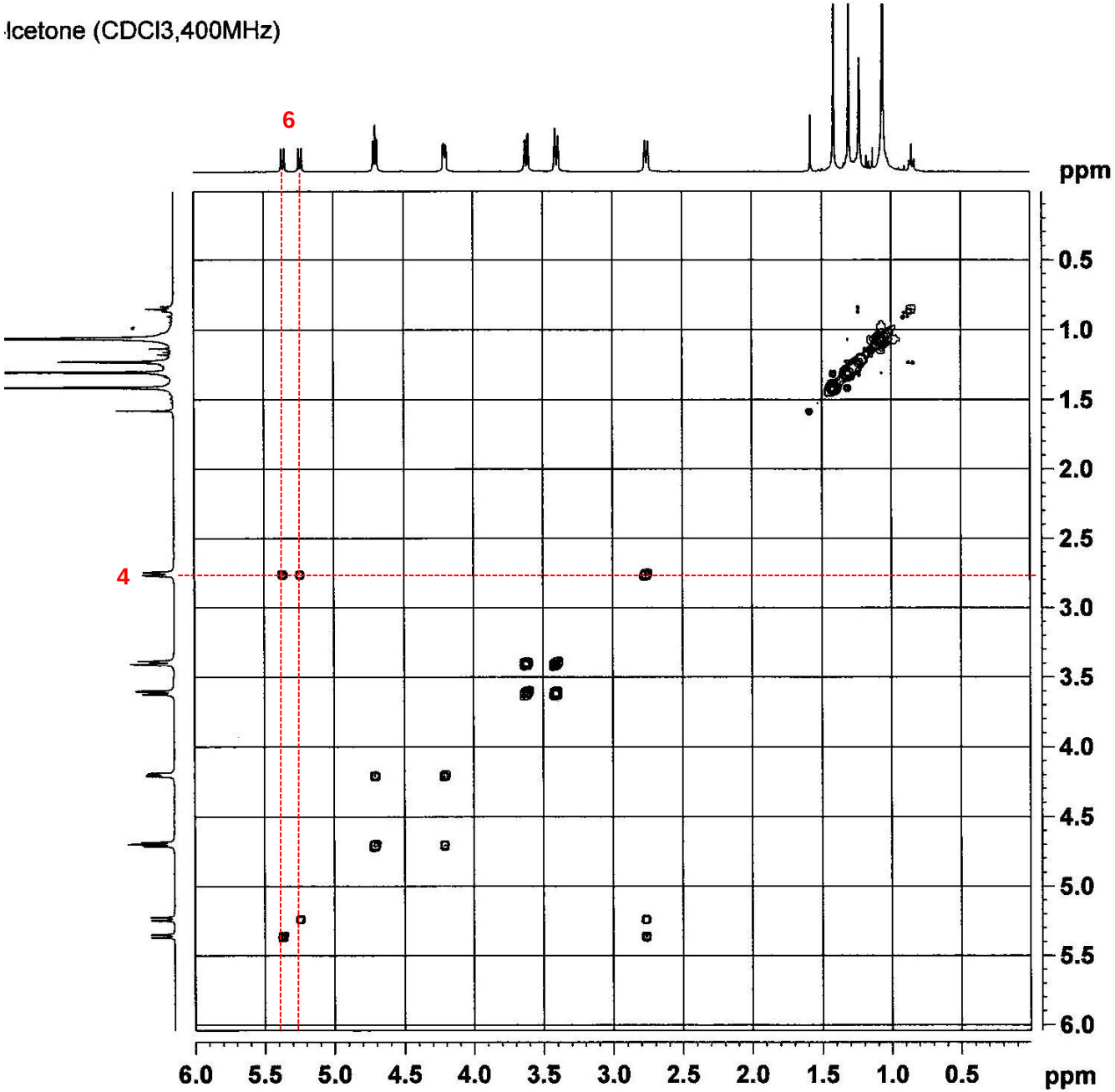
Icetone (CDCl3,400MHz)

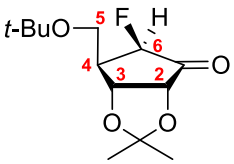




7a

400 MHz ^{1}H-^{13}C HSQC, $CDCl_3$





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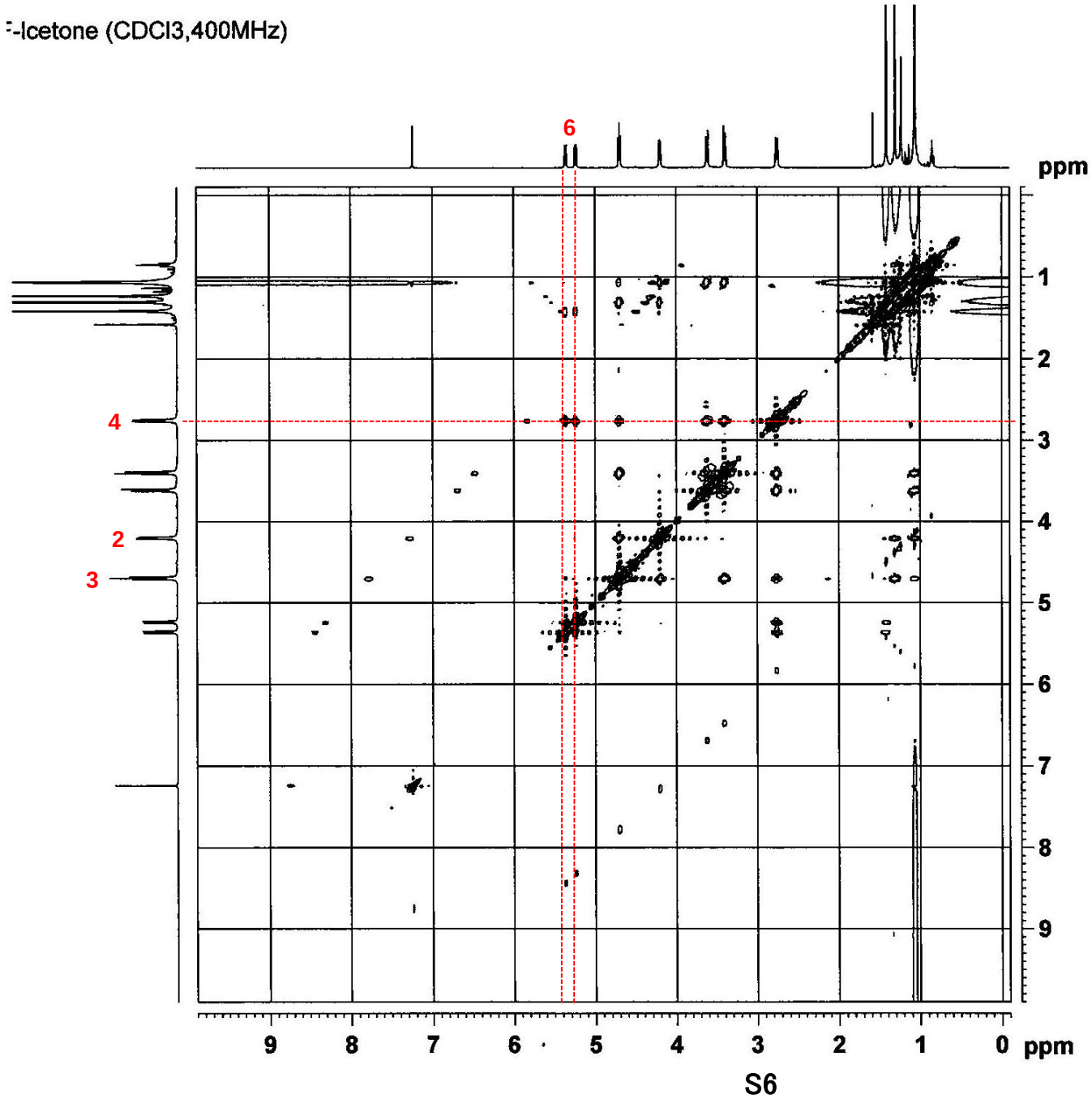
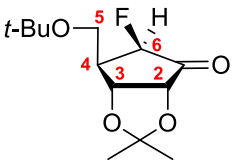
400 MHz ^{1}H-^{1}H COSY, $CDCl_3$





7a

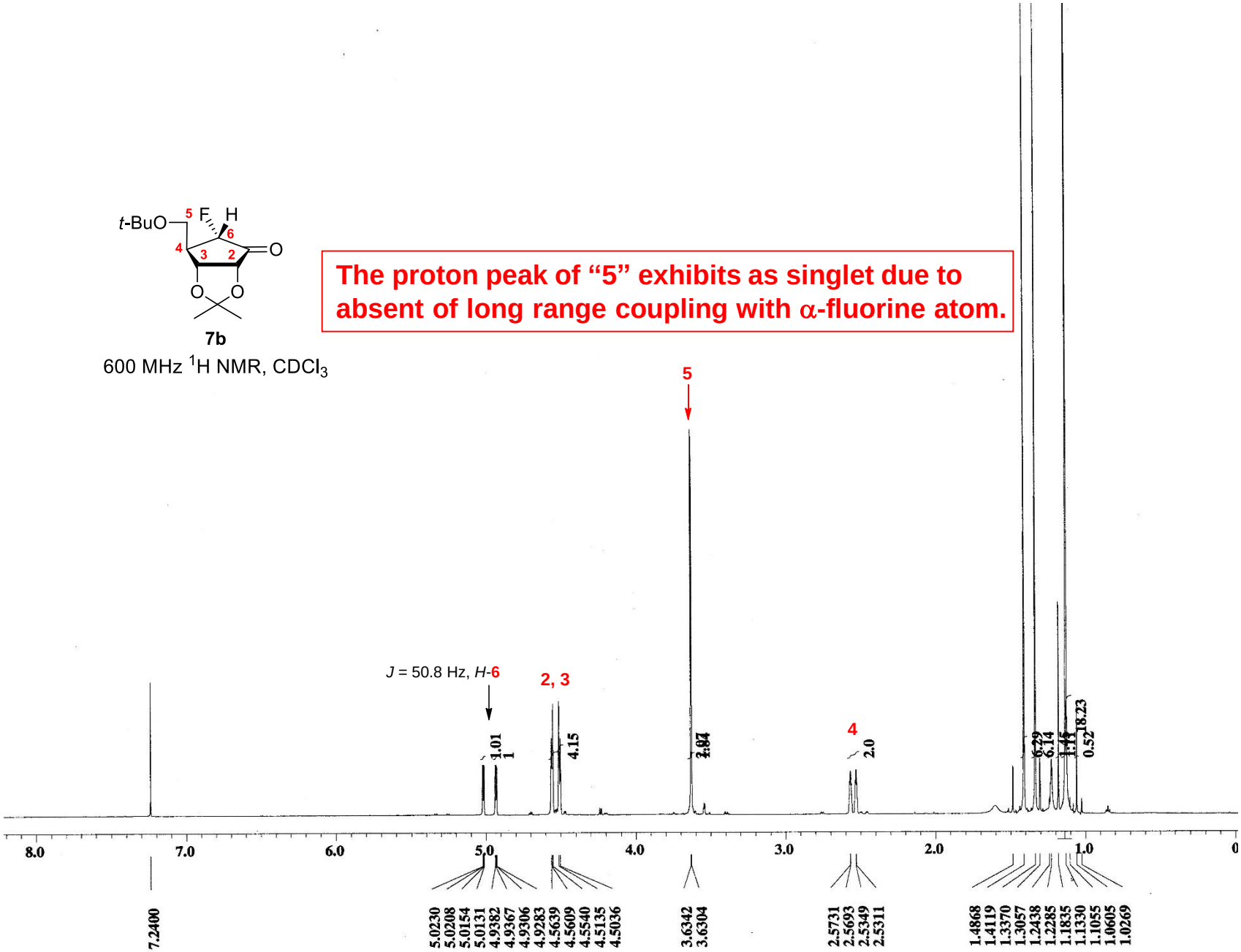
400 MHz ^{1}H-^{1}H NOESY, $CDCl_3$

4–6 NOE (O)
2–6 NOE (X)
3–6 NOE (X)

7b

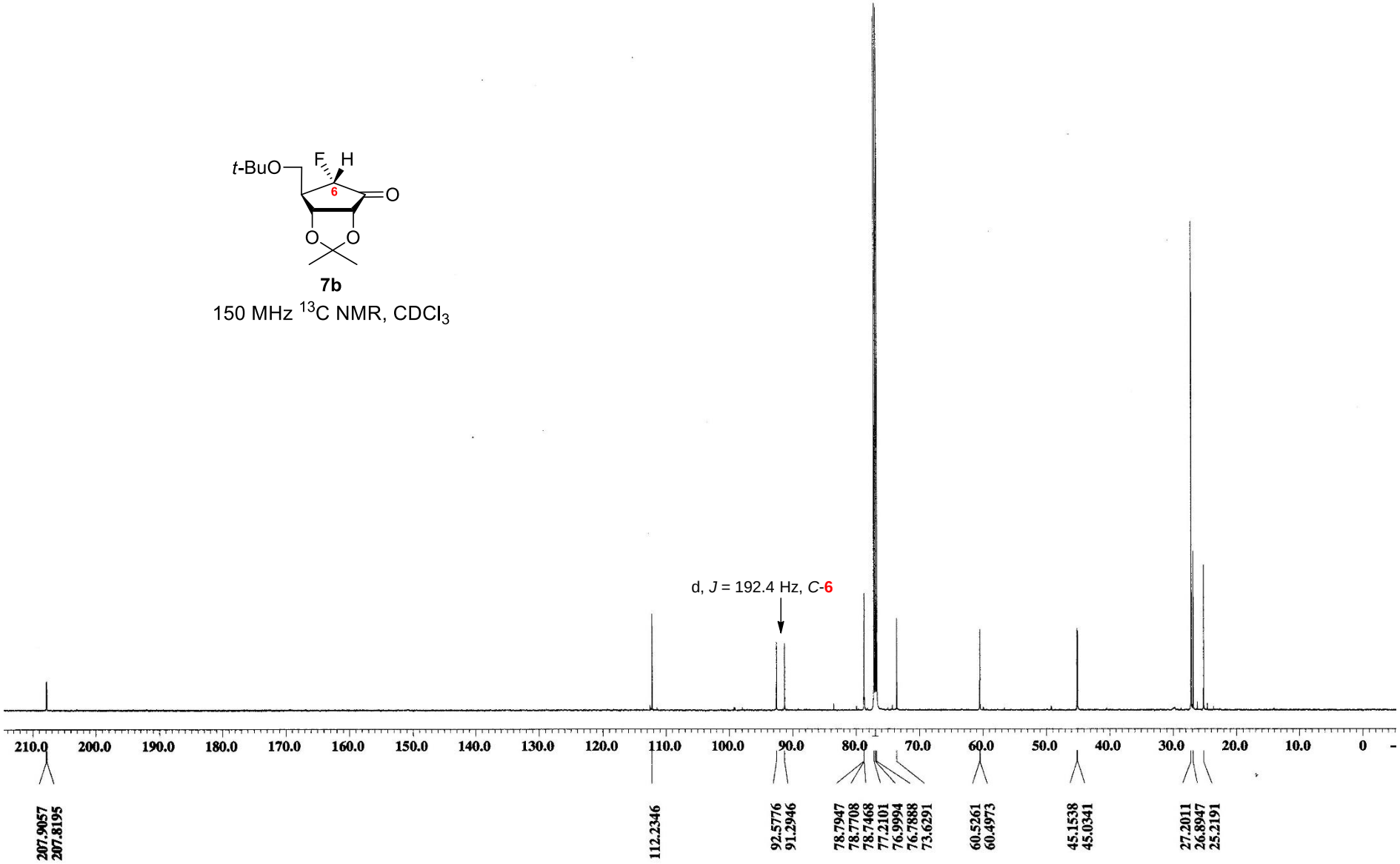
600 MHz ^{1}H NMR, $CDCl_3$

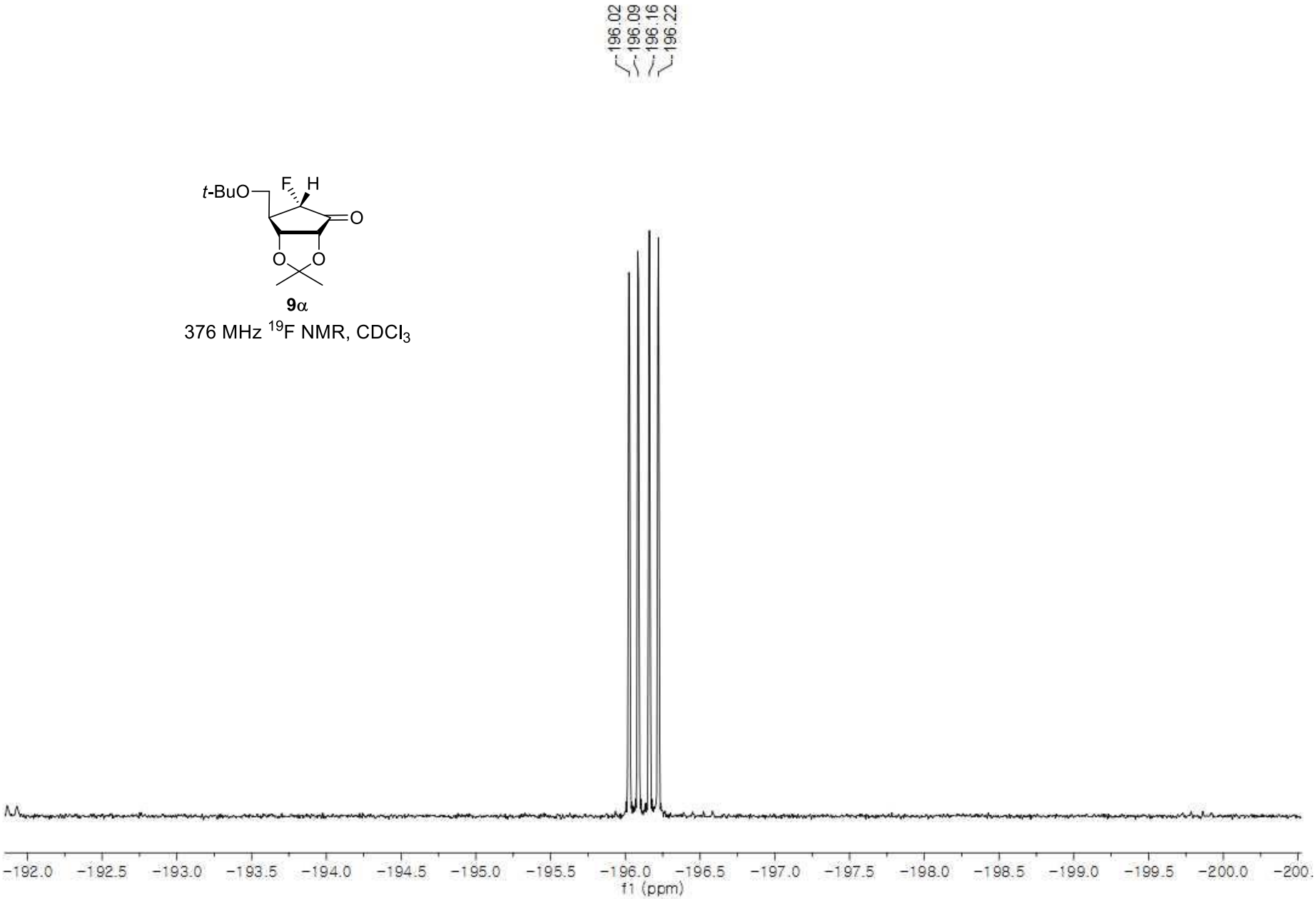
The proton peak of "5" exhibits as singlet due to absent of long range coupling with α-fluorine atom.

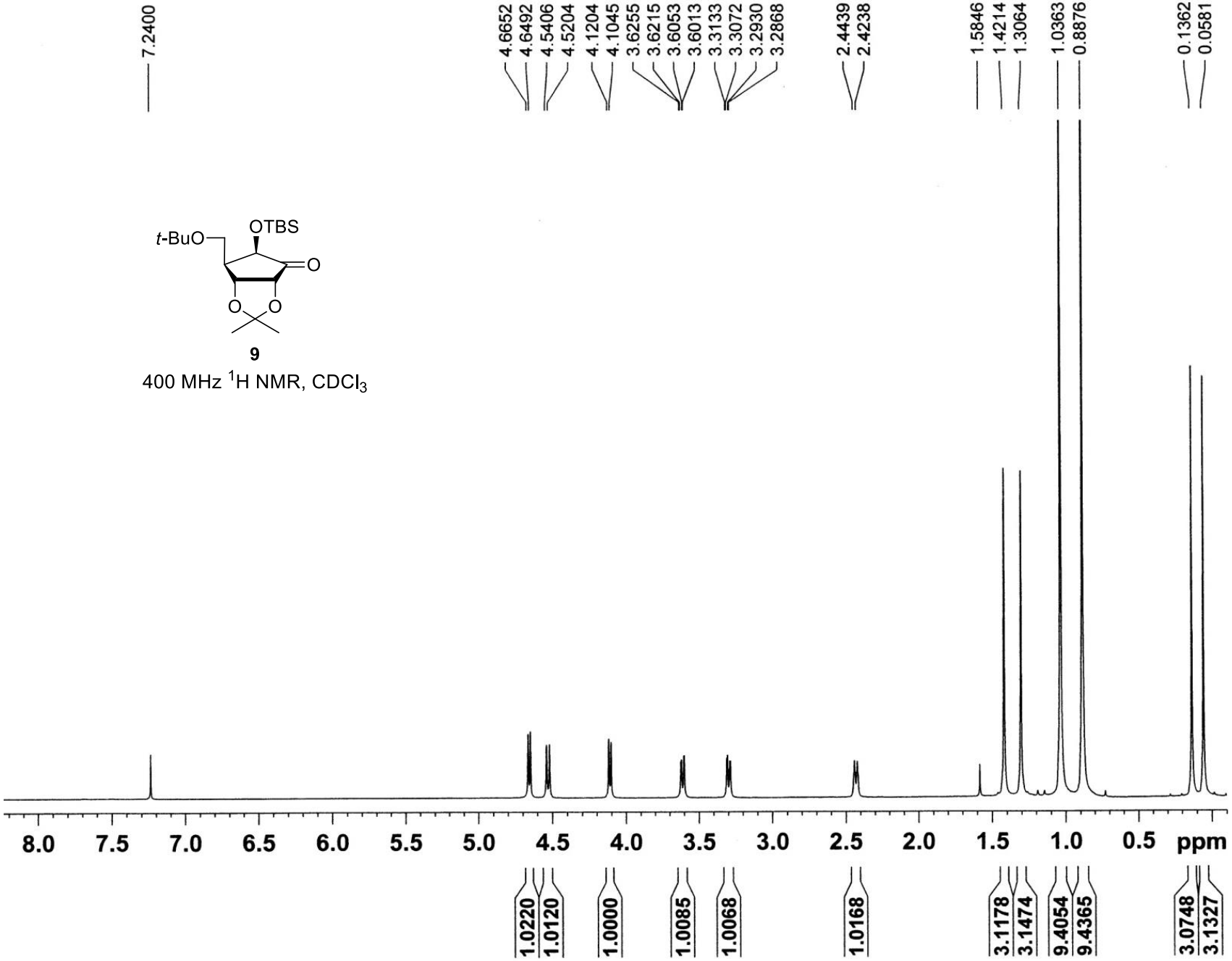


7b

150 MHz ^{13}C NMR, $CDCl_3$




196.02
196.09
196.16
196.22
t-BuO
F
H
O
O
O
9α
376 MHz 19F NMR, CDCl3
−192.0
−192.5
−193.0
−193.5
−194.0
−194.5
−195.0
−195.5
−196.0
−196.5
−197.0
−197.5
−198.0
−198.5
−199.0
−199.5
−200.0
f1 (ppm)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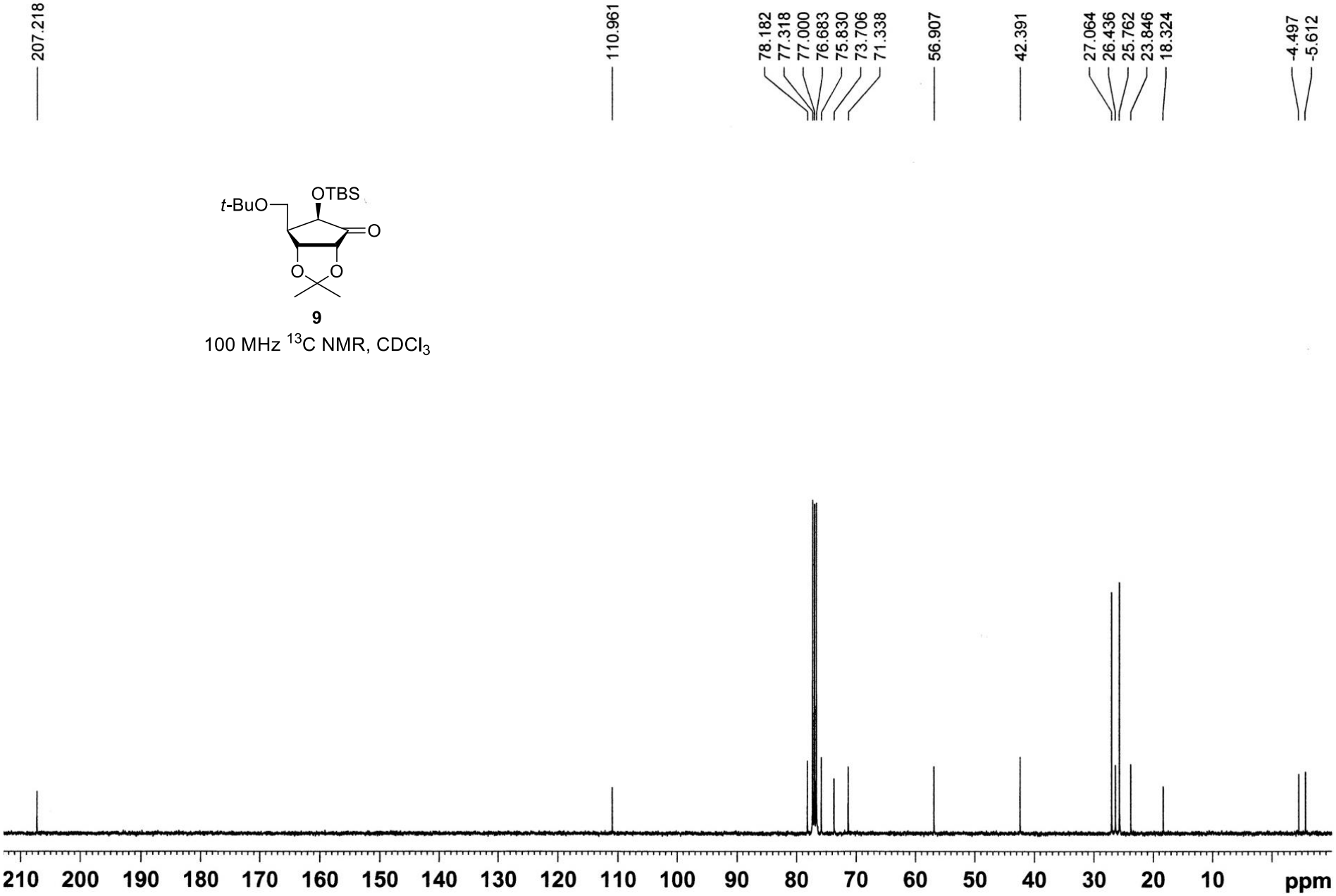
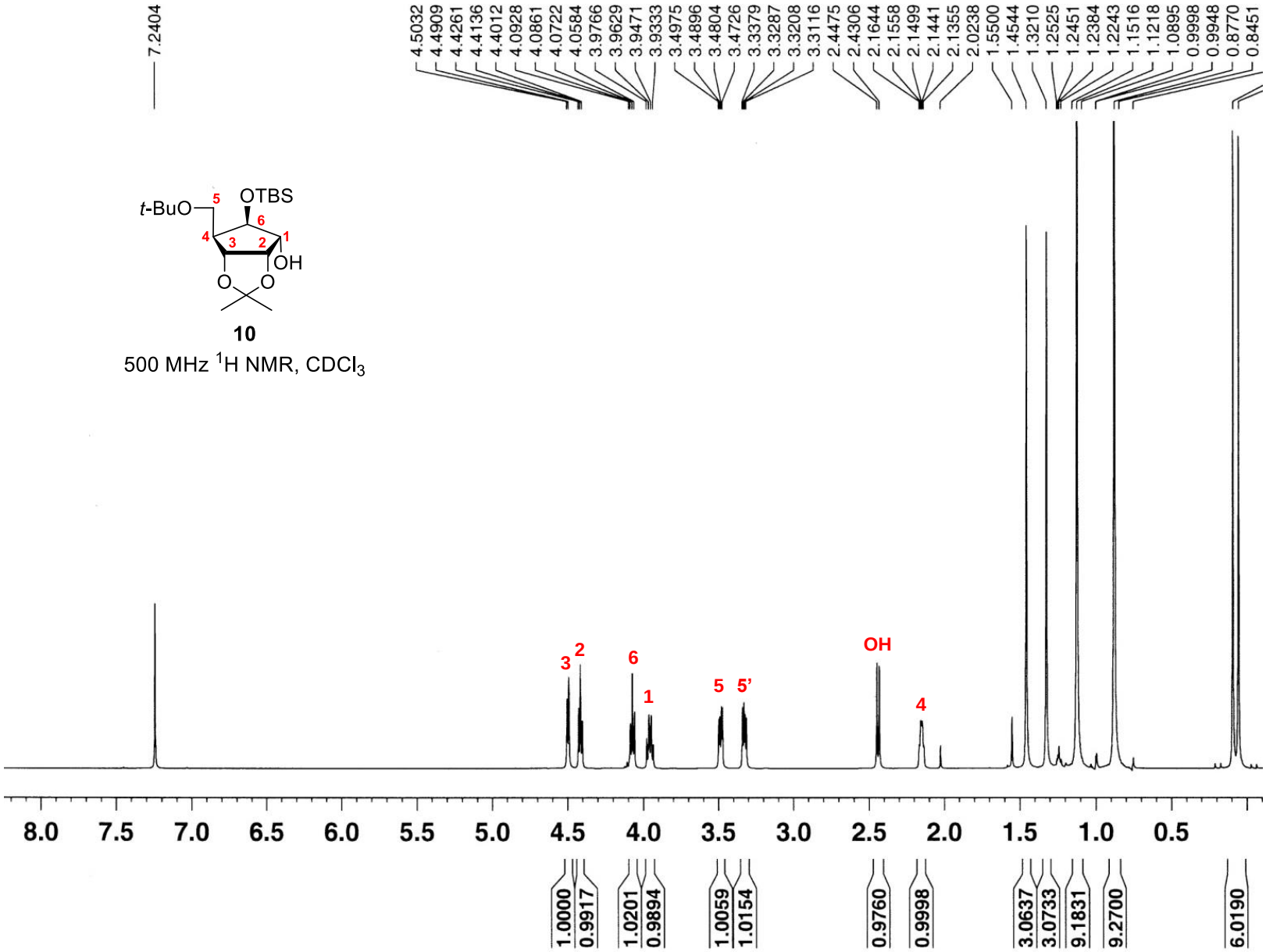
4.6652
4.6492
4.5406
4.5204
4.1204
4.1045
3.6255
3.6215
3.6053
3.6013
3.3133
3.3072
3.2930
3.2868
2.4439
2.4238
1.5846
1.4214
1.3064
1.0363
0.8876
0.1362
0.0581
OTBS
t-BuO
O
O
O
9
400 MHz 1H NMR, CDCl3
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.0220
1.0120
1.0000
1.0085
1.0068
1.0168
3.1178
3.1474
9.4054
9.4365
3.0748
3.1327



9

100 MHz ^{13}C NMR, $CDCl_3$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4.4136
4.4012
4.0928
4.0861
4.0722
4.0584
3.9766
3.9629
3.9471
3.9333
3.4975
3.4896
3.4804
3.4726
3.3379
3.3287
3.3208
3.3116
2.4475
2.4306
2.1644
2.1558
2.1499
2.1441
2.1355
2.0238
1.5500
1.4544
1.3210
1.2525
1.2451
1.2384
1.2243
1.1516
1.1218
1.0895
0.9998
0.9948
0.8770
0.8451
t-BuO
OTBS
OH
O
O
5
6
4
3
2
1
10
500 MHz 1H NMR, CDCl3
3
2
6
1
5
5'
OH
4
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
1.0000
0.9917
1.0201
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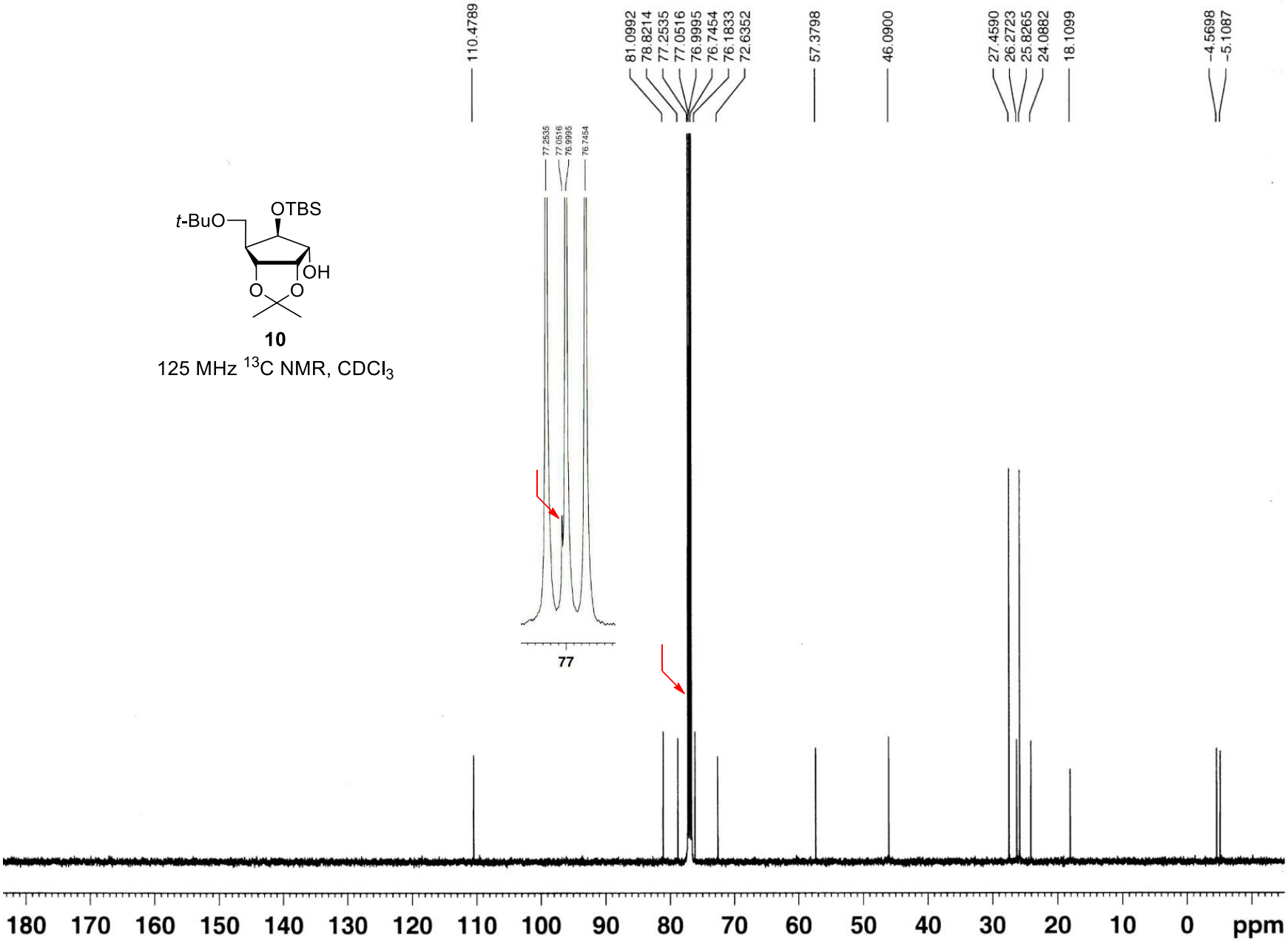
3.0733
9.1831
9.2700
6.0190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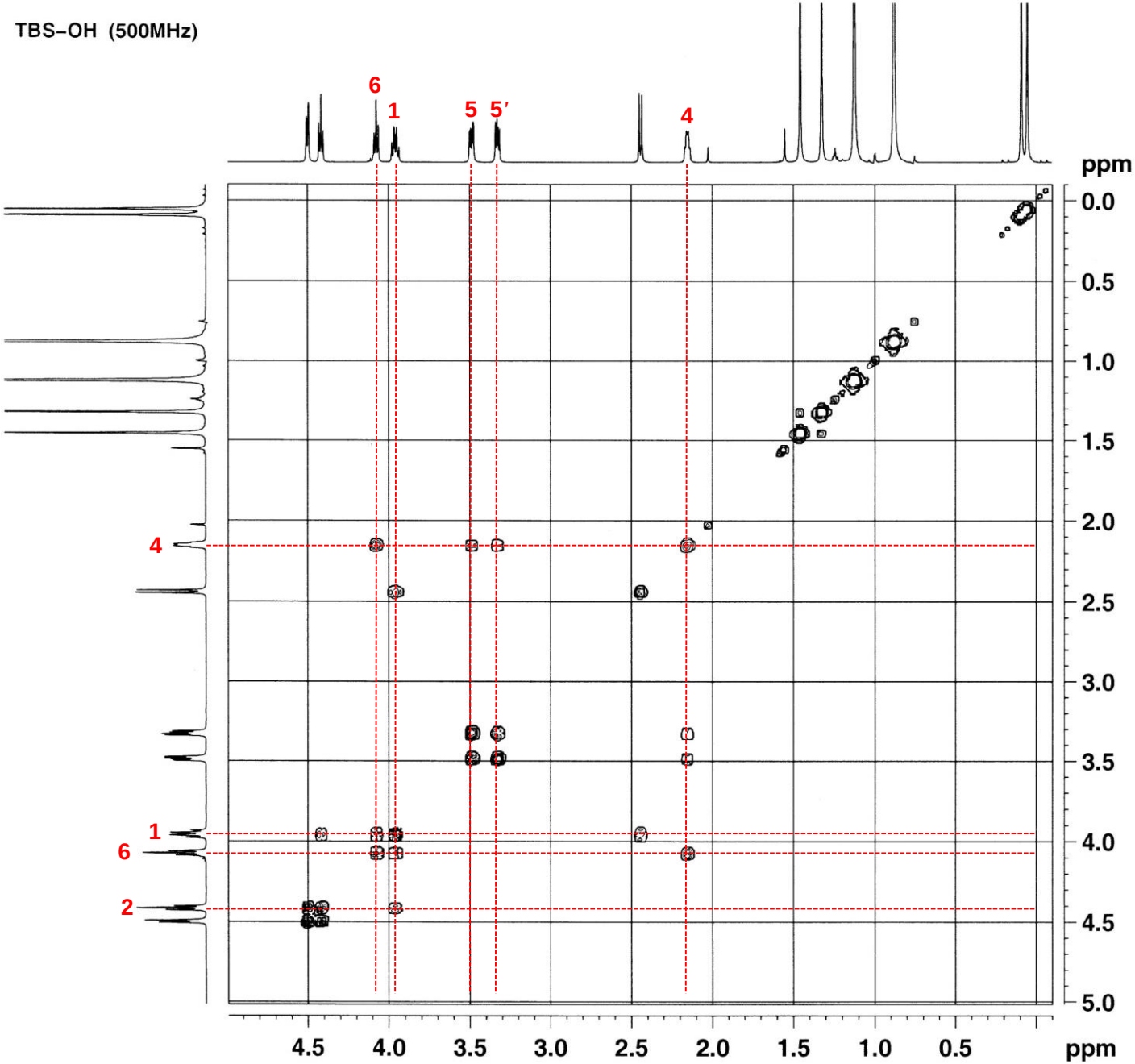
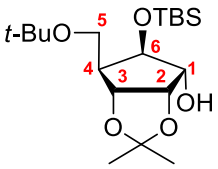
77.2535
77.0516
76.9995
76.7454
76.1833
72.6352
57.3798
46.0900
27.4590
26.2723
25.8265
24.0882
18.1099
-4.5698
-5.1087
77.2535
77.0516
76.9995
76.7454
77
t-BuO
OTBS
OH
O
O
10
125 MHz 13C NMR, CDCl3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

TBS–OH (500MHz)



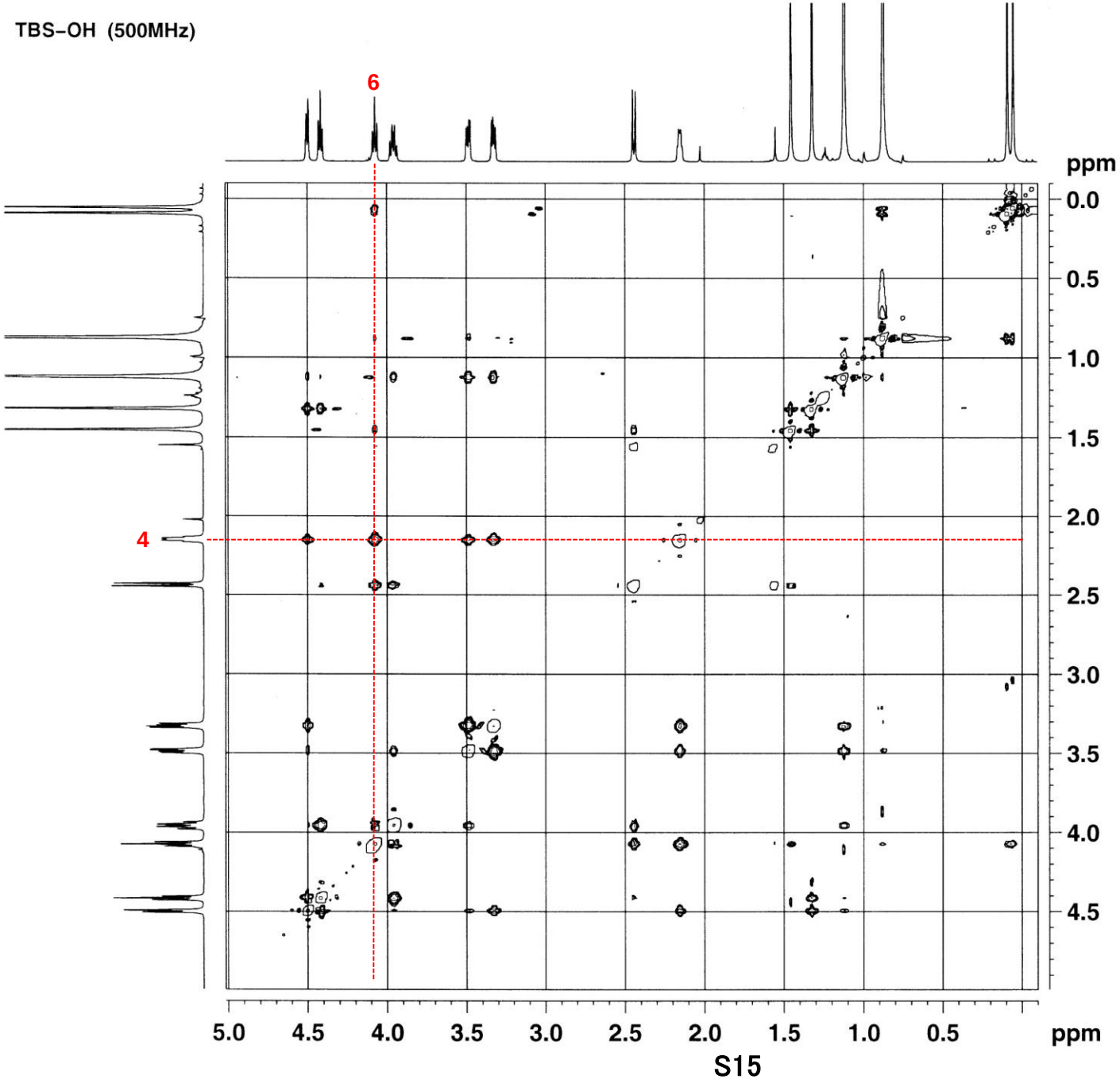


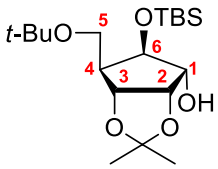
10

500 MHz ^{1}H-^{1}H COSY NMR

$CDCl_3$

TBS–OH (500MHz)



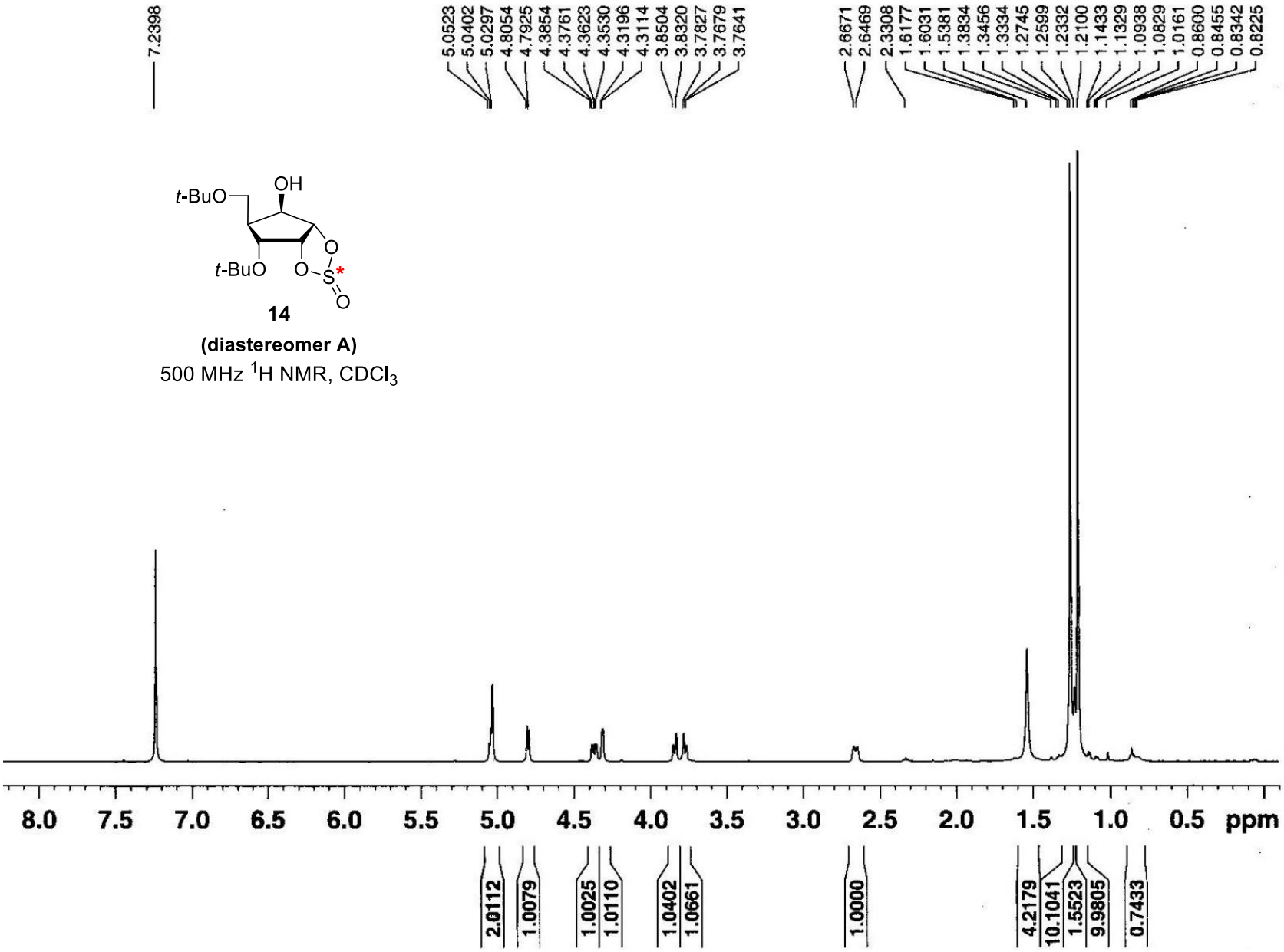


10

500 MHz ^{1}H-^{1}H NOESY NMR

$CDCl_3$

4–6 NOE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4.8054
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4.3761
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4.3530
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4.3114
3.8504
3.8320
3.7827
3.7679
3.7641
2.6671
2.6469
2.3308
1.6177
1.6031
1.5381
1.3834
1.3456
1.3334
1.2745
1.2599
1.2332
1.2100
1.1433
1.1329
1.0938
1.0829
1.0161
0.8600
0.8455
0.8342
0.8225
t-BuO
OH
t-BuO
O
O
S*
O
14
(diastereomer A)
500 MHz 1H NMR, CDCl3
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
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1.0079
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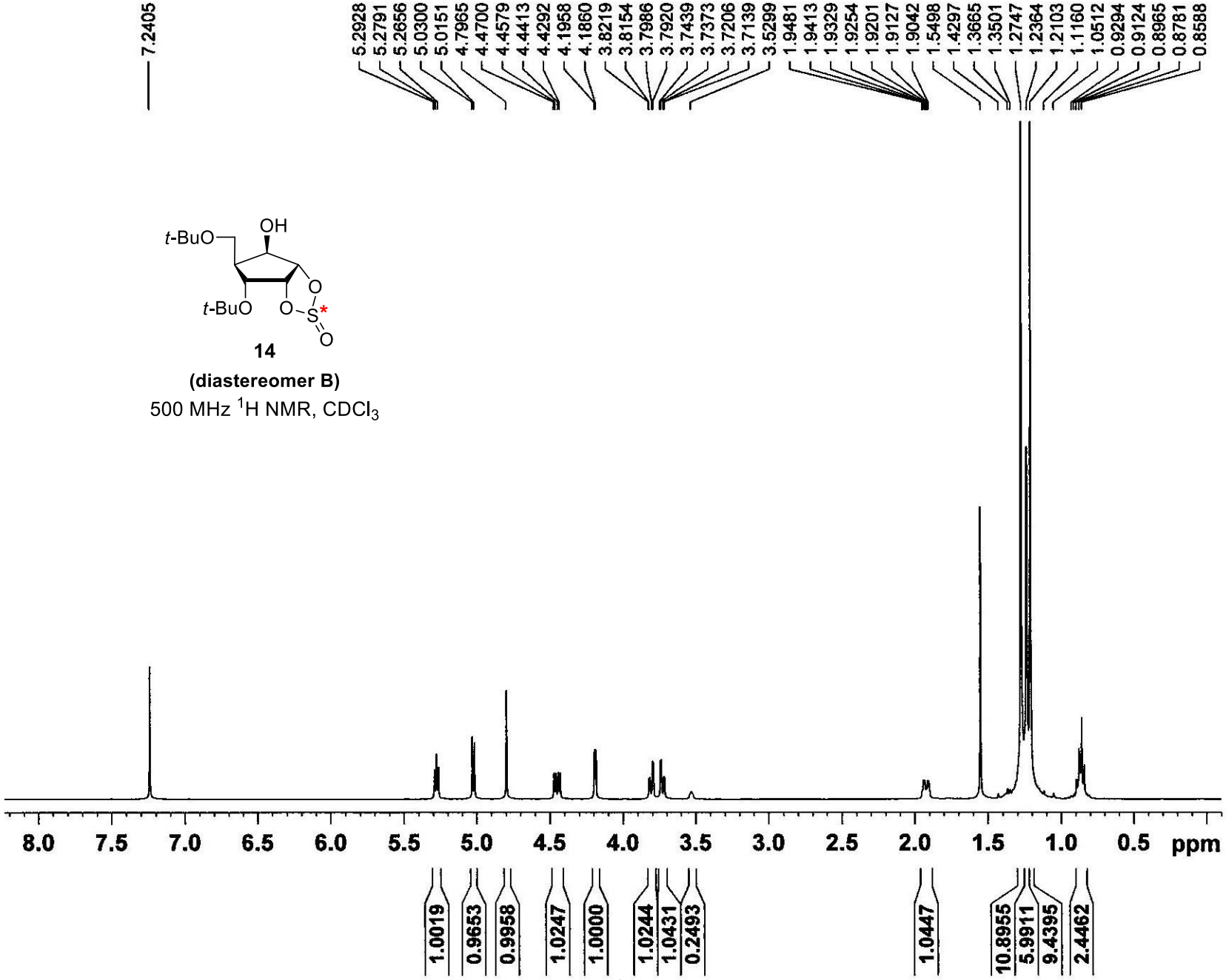
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4.1860
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3.8154
3.7986
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3.7439
3.7373
3.7206
3.7139
3.5299
1.9481
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1.9329
1.9254
1.9201
1.9127
1.9042
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1.4297
1.3665
1.3501
1.2747
1.2364
1.2103
1.1160
1.0512
0.9294
0.9124
0.8965
0.8781
0.8588
t-BuO
OH
t-BuO
O
O
S*
O
14
(diastereomer B)
500 MHz 1H NMR, CDCl3
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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0.9958
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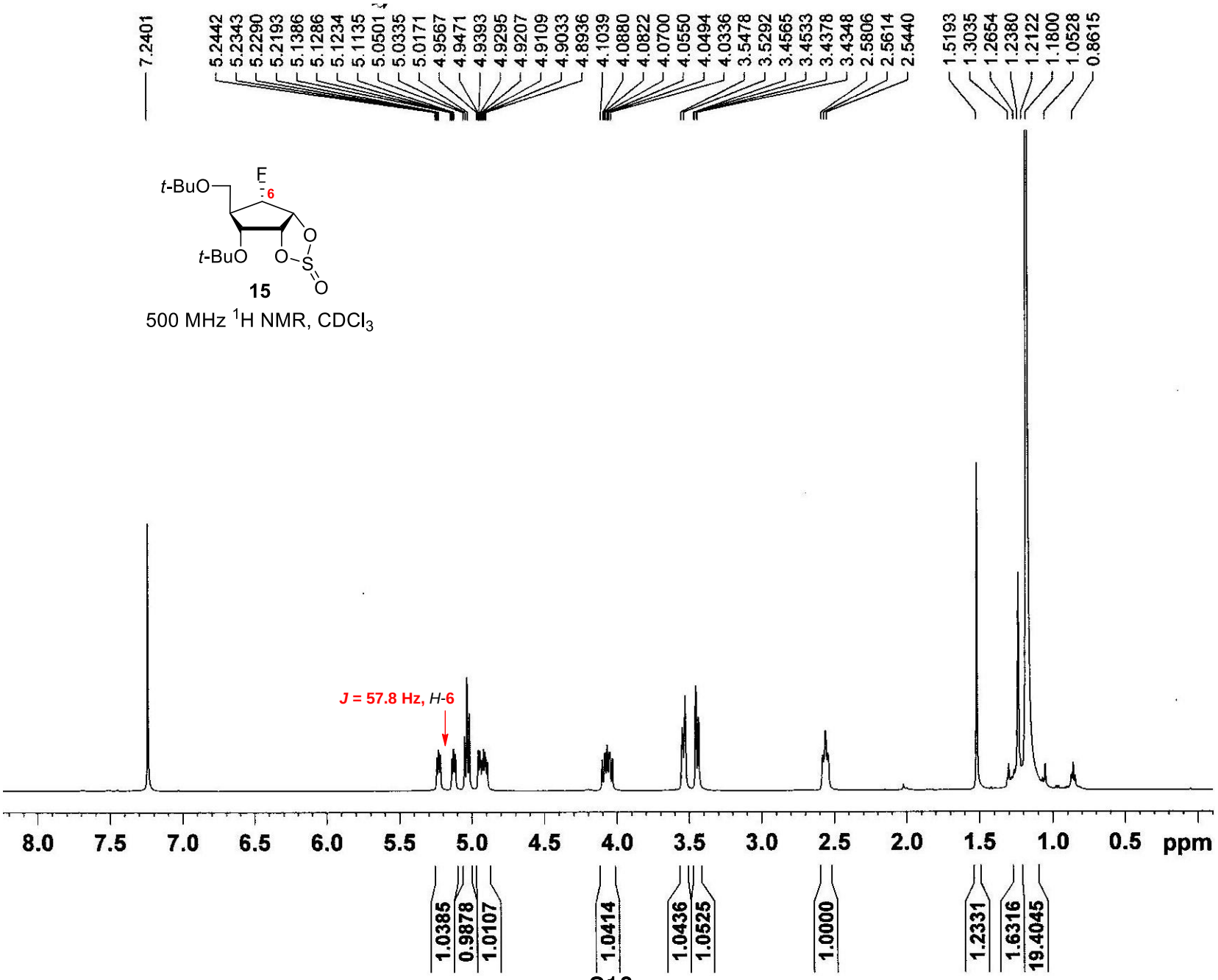
9.4395
2.4462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4.0880
4.0822
4.0700
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2.5614
2.5440
1.5193
1.3035
1.2654
1.2380
1.2122
1.1800
1.0528
0.8615
t-BuO
F
6
t-BuO
O
O
S
O
15
500 MHz 1H NMR, CDCl3
J = 57.8 Hz, H-6
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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1.0436
1.0525
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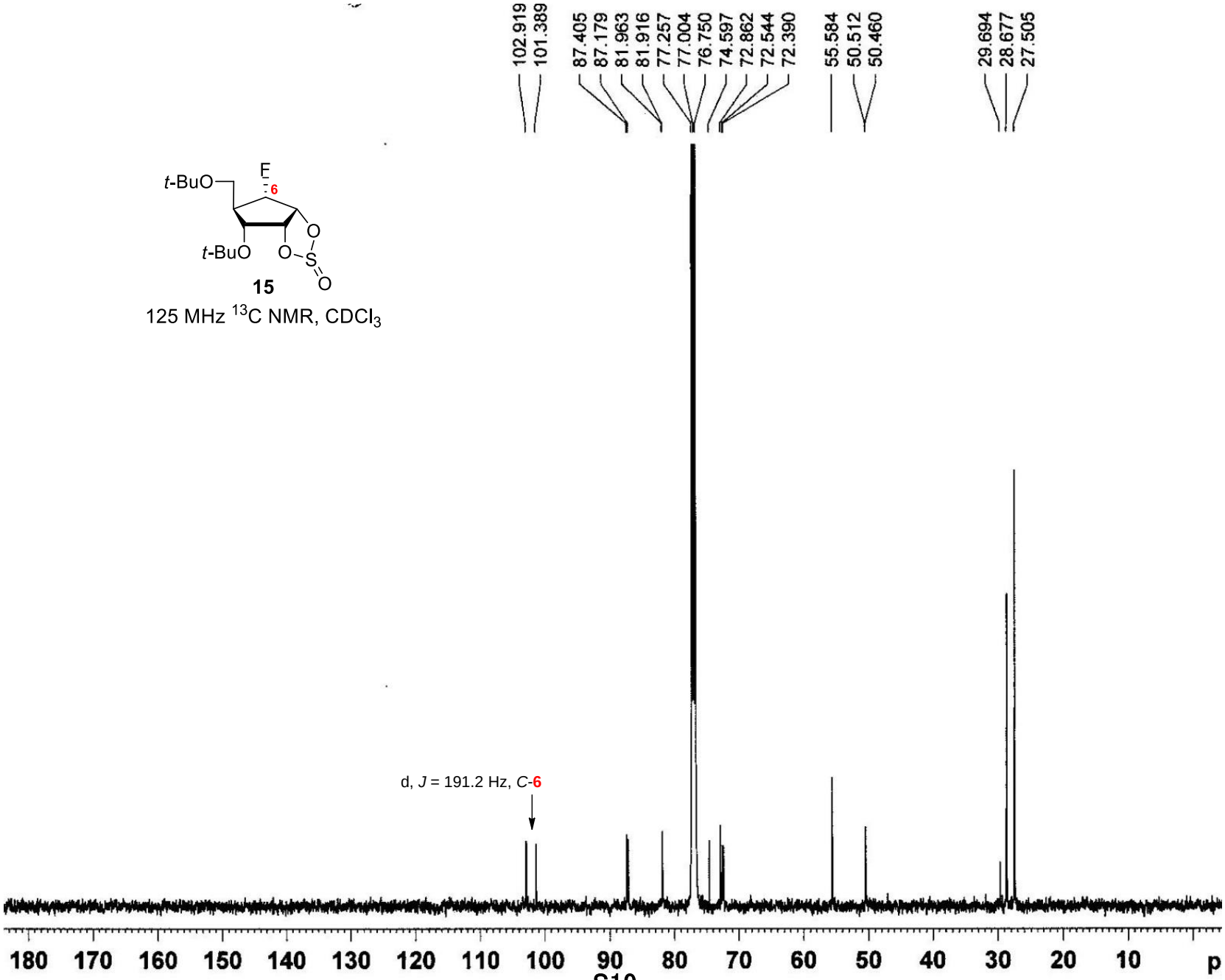
1.6316
19.4045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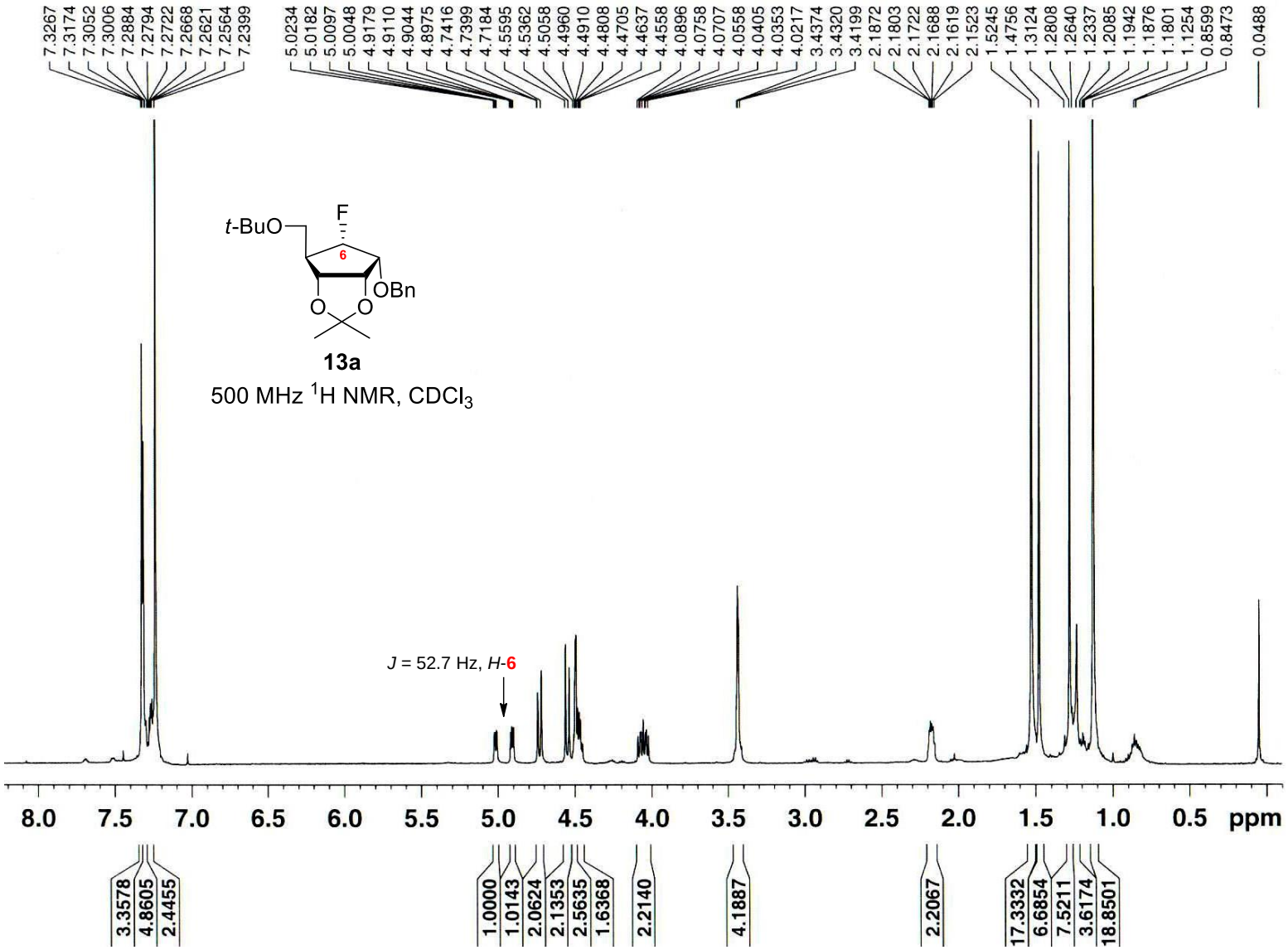
101.389
87.405
87.179
81.963
81.916
77.257
77.004
76.750
74.597
72.862
72.544
72.390
55.584
50.512
50.460
29.694
28.677
27.505
t-BuO
F
6
t-BuO
O
S
O
O
15
125 MHz 13C NMR, CDCl3
d, J = 191.2 Hz, C-6
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
p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7.3052
7.3006
7.2884
7.2794
7.2722
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7.2621
7.2564
7.2399
5.0234
5.0182
5.0097
5.0048
4.9179
4.9110
4.9044
4.8975
4.7416
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4.0405
4.0353
4.0217
3.4374
3.4320
3.4199
2.1872
2.1803
2.1722
2.1688
2.1619
2.1523
1.5245
1.4756
1.3124
1.2808
1.2640
1.2337
1.2085
1.1942
1.1876
1.1801
1.1254
0.8599
0.8473
0.0488
t-BuO
F
6
OBn
O
O
13a
500 MHz 1H NMR, CDCl3
J = 52.7 Hz, H-6
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.3578
4.8605
2.4455
1.0000
1.0143
2.0624
2.1353
2.5635
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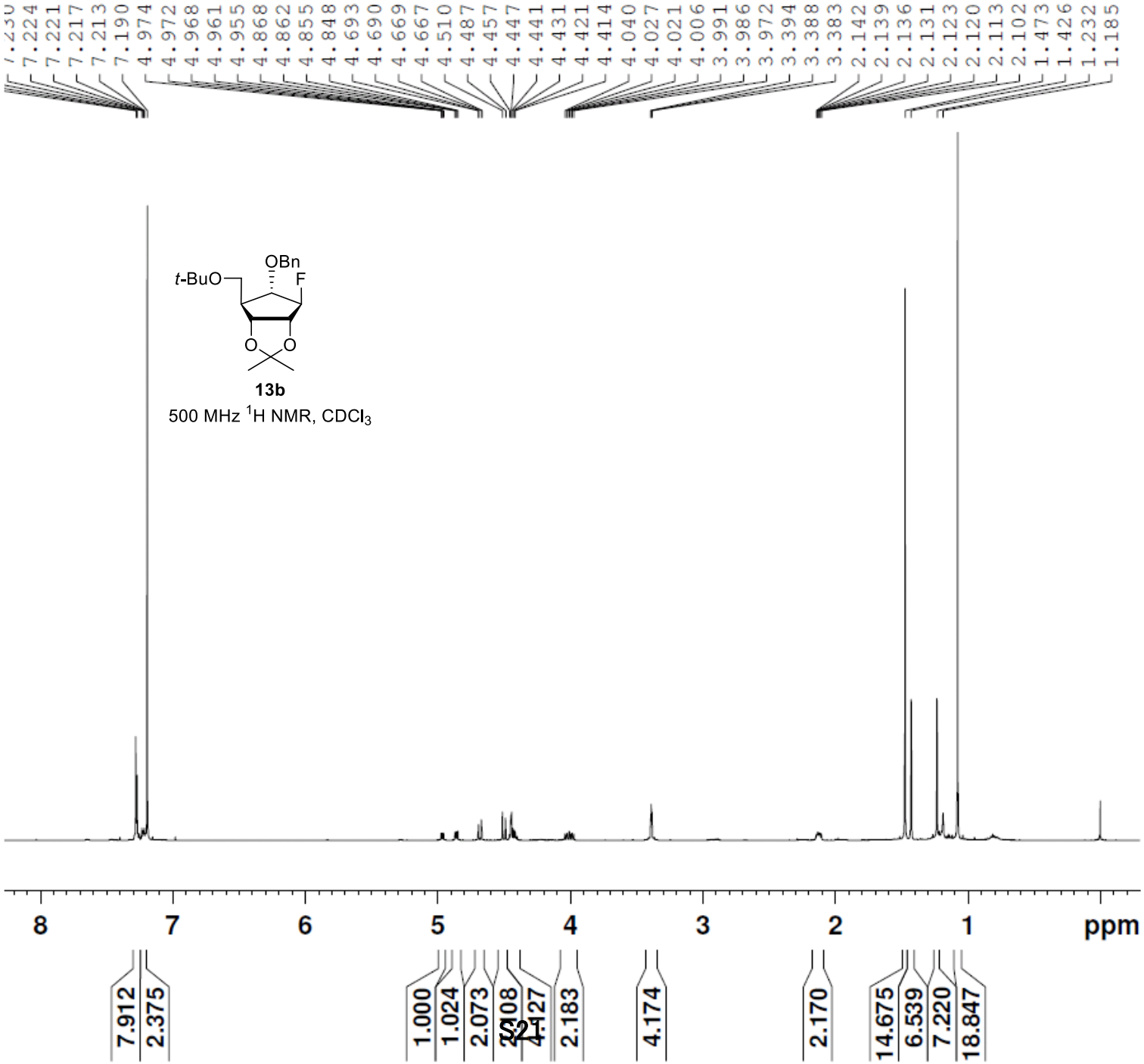
2.2140
4.1887
2.2067
17.3332
6.6854
7.5211
3.6174
18.8501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4.855
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4.690
4.669
4.667
4.510
4.487
4.457
4.447
4.441
4.431
4.421
4.414
4.040
4.027
4.021
4.006
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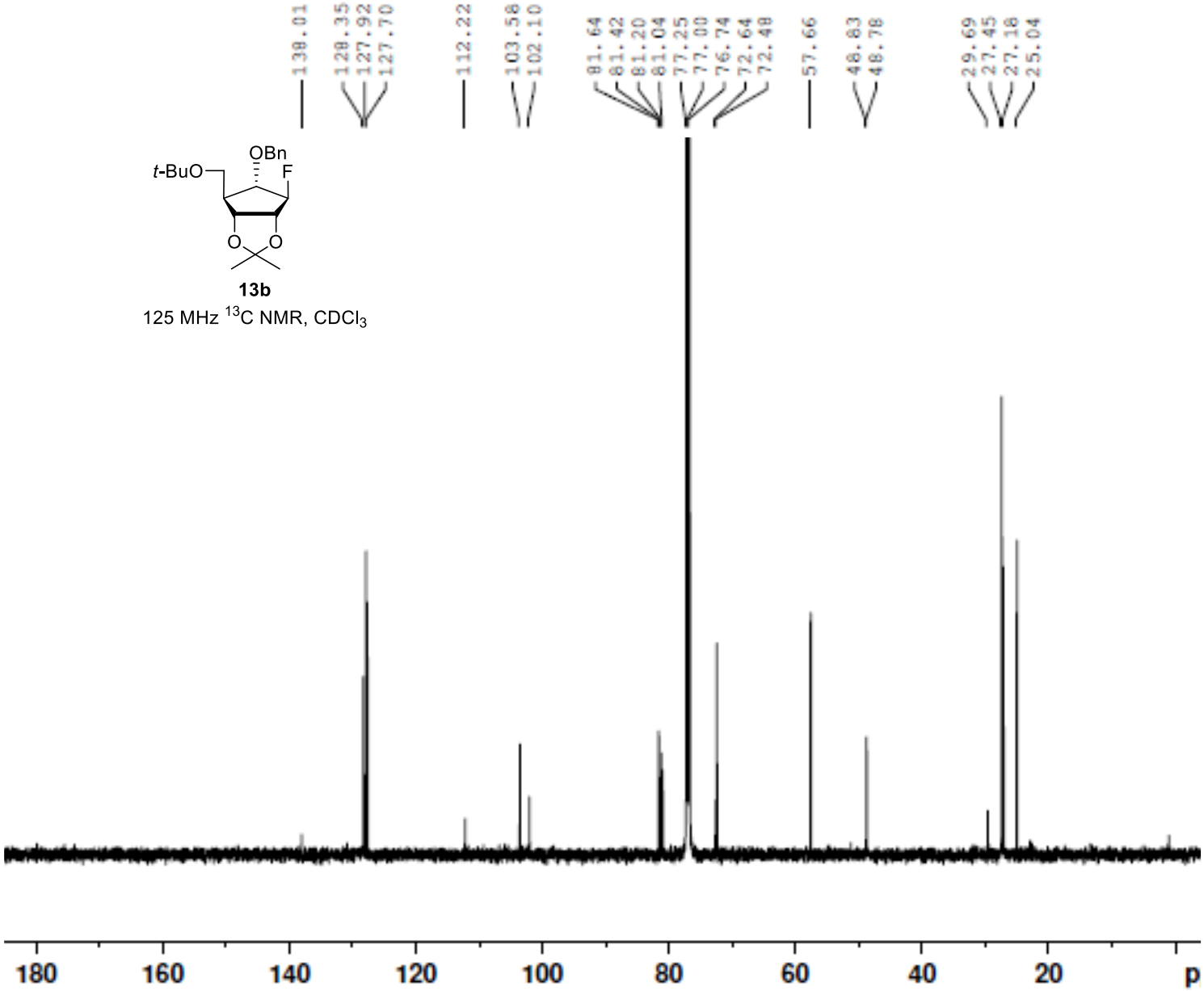
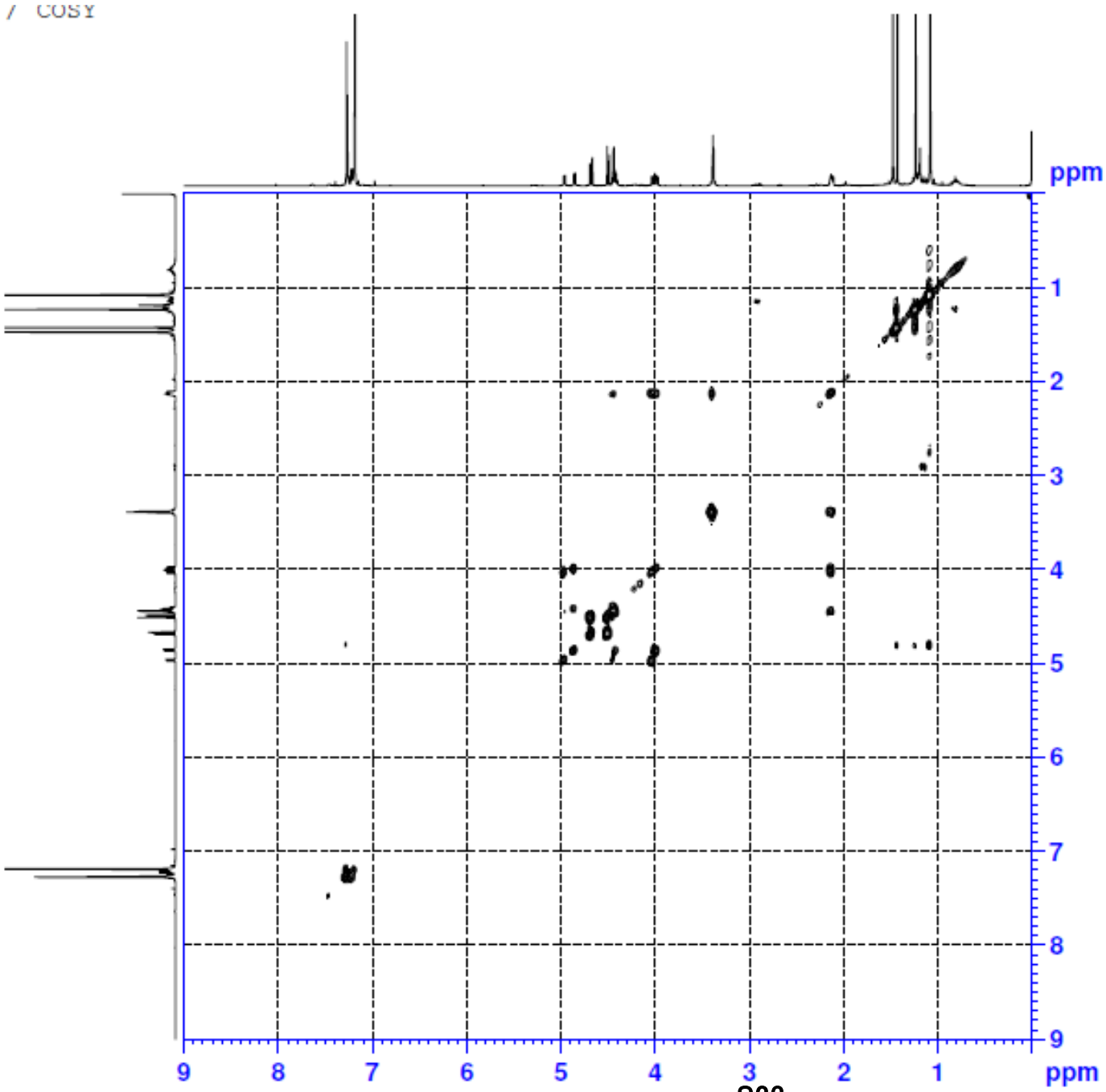
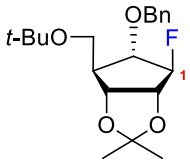
2.120
2.113
2.102
1.473
1.426
1.232
1.185
OBn
t-BuO
F
O
O
13b
500 MHz 1H NMR, CDCl3
8
7
6
5
4
3
2
1
ppm
7.912
2.375
1.000
1.024
2.073
2.008
4.427
2.183
4.174
2.170
14.675
6.539
7.220
18.847

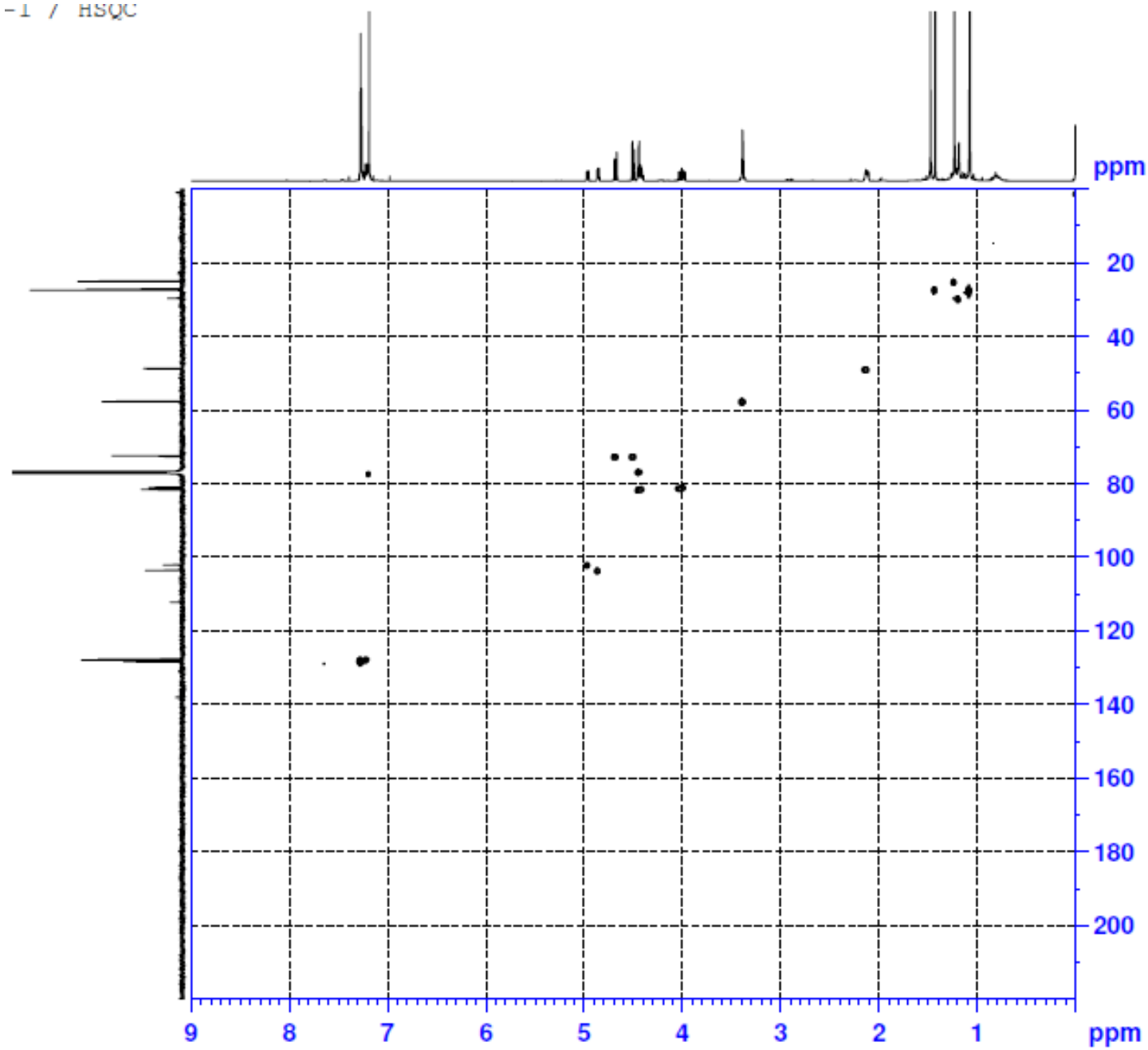

138.01
128.35
127.92
127.70
112.22
103.58
102.10
81.64
81.42
81.20
81.04
77.25
77.00
76.74
72.64
72.48
57.66
48.83
48.78
29.69
27.45
27.18
25.04
OBn
t-BuO
F
O
O
13b
125 MHz 13C NMR, CDCl3
180
160
140
120
100
80
60
40
20
p

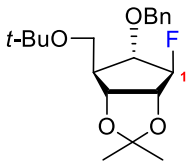




13b

500 MHz ^{1}H-^{1}H COSY NMR
$CDCl_3$

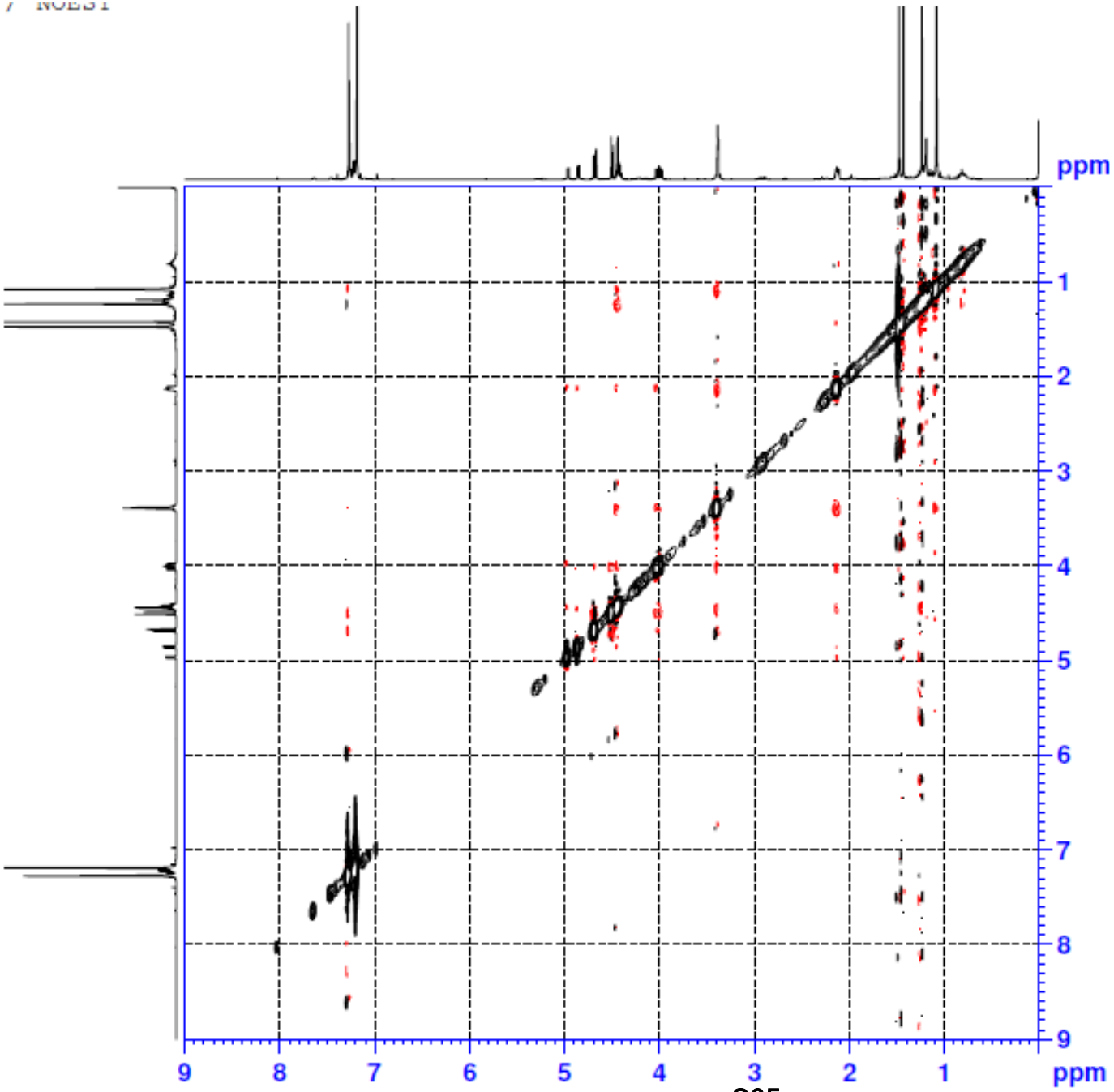


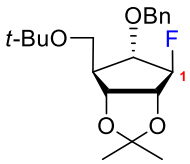


13b

500 MHz ^{1}H-^{13}C HSQC NMR

$CDCl_3$





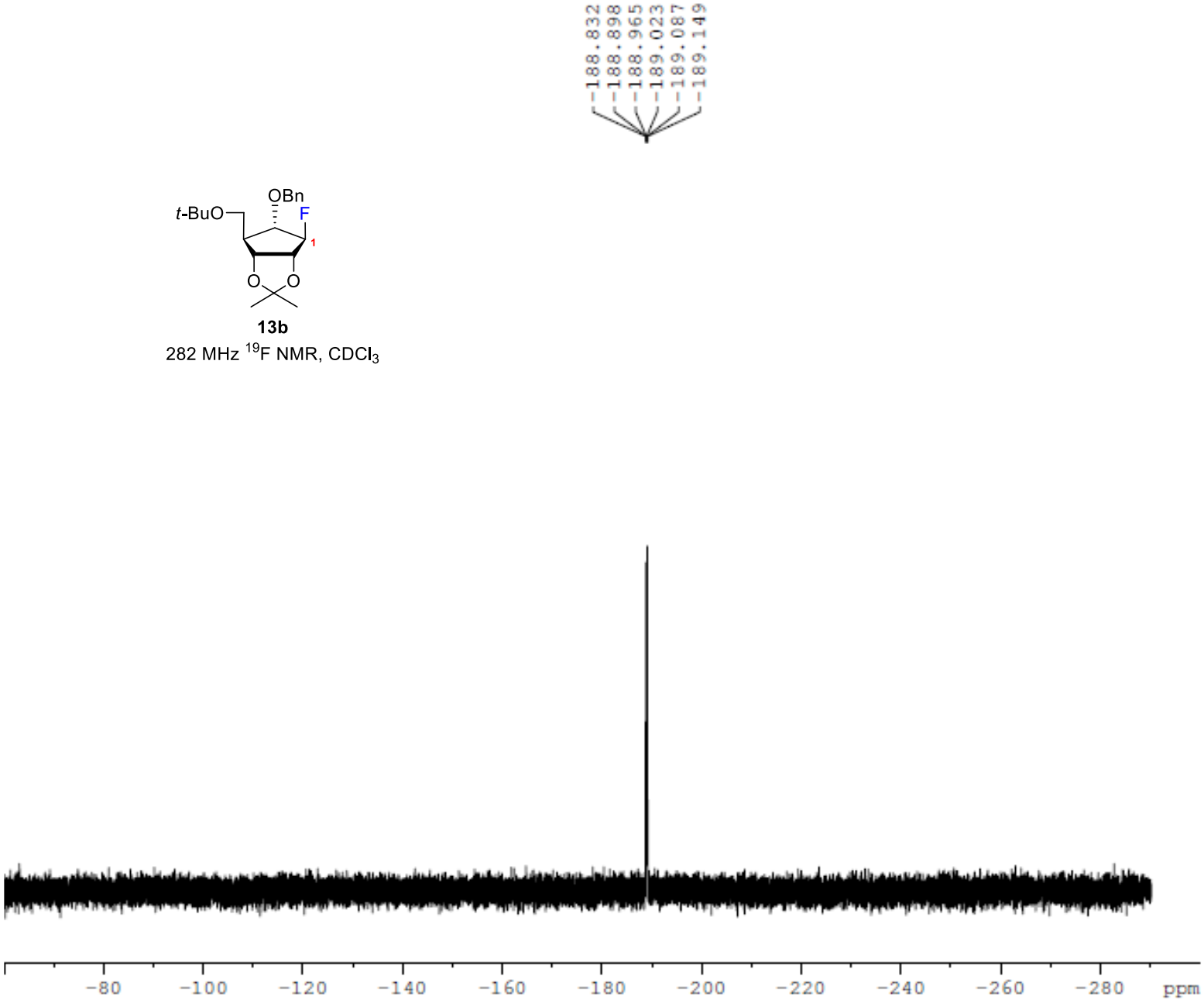
13b

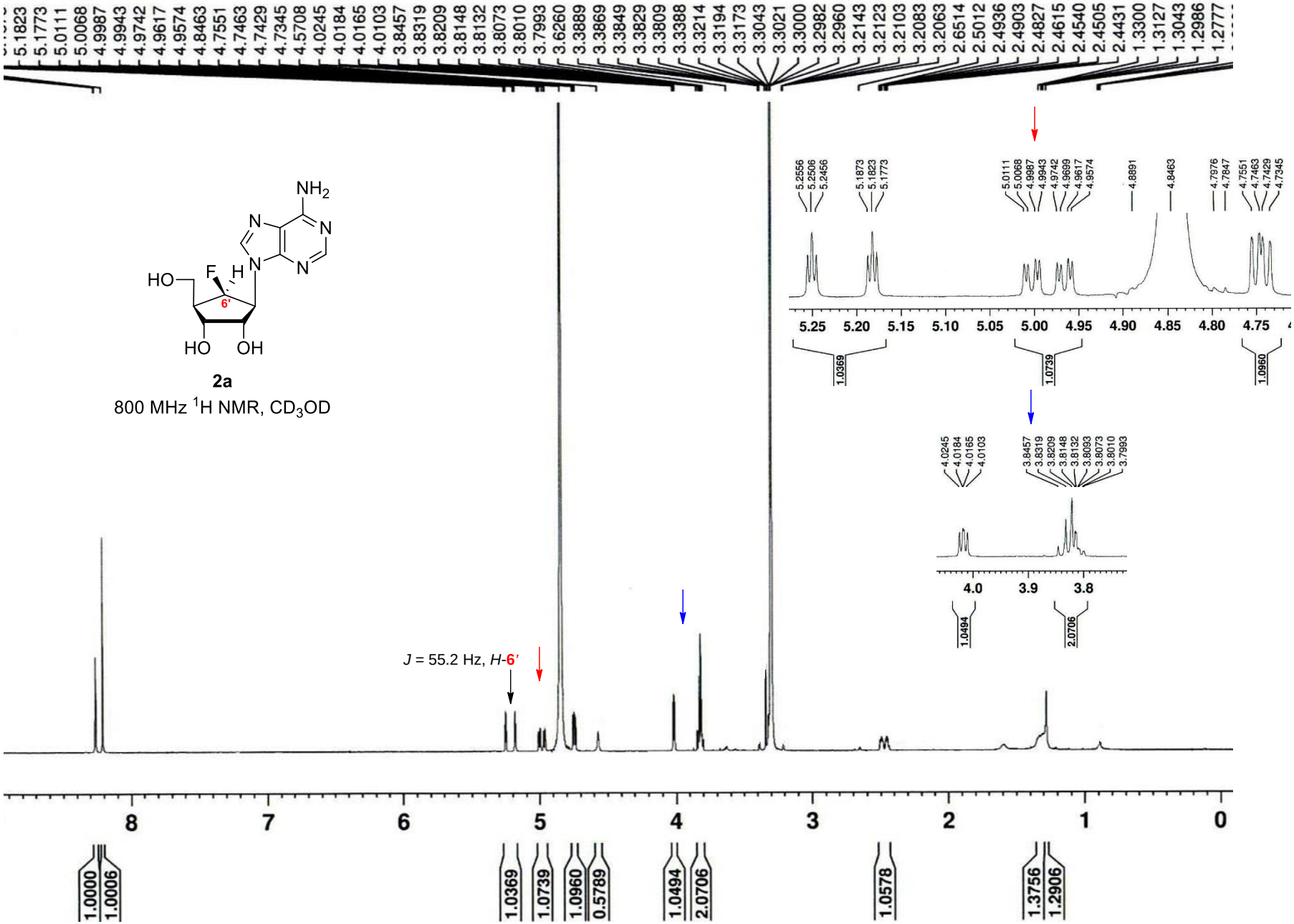
500 MHz ^{1}H-^{1}H NOESY NMR

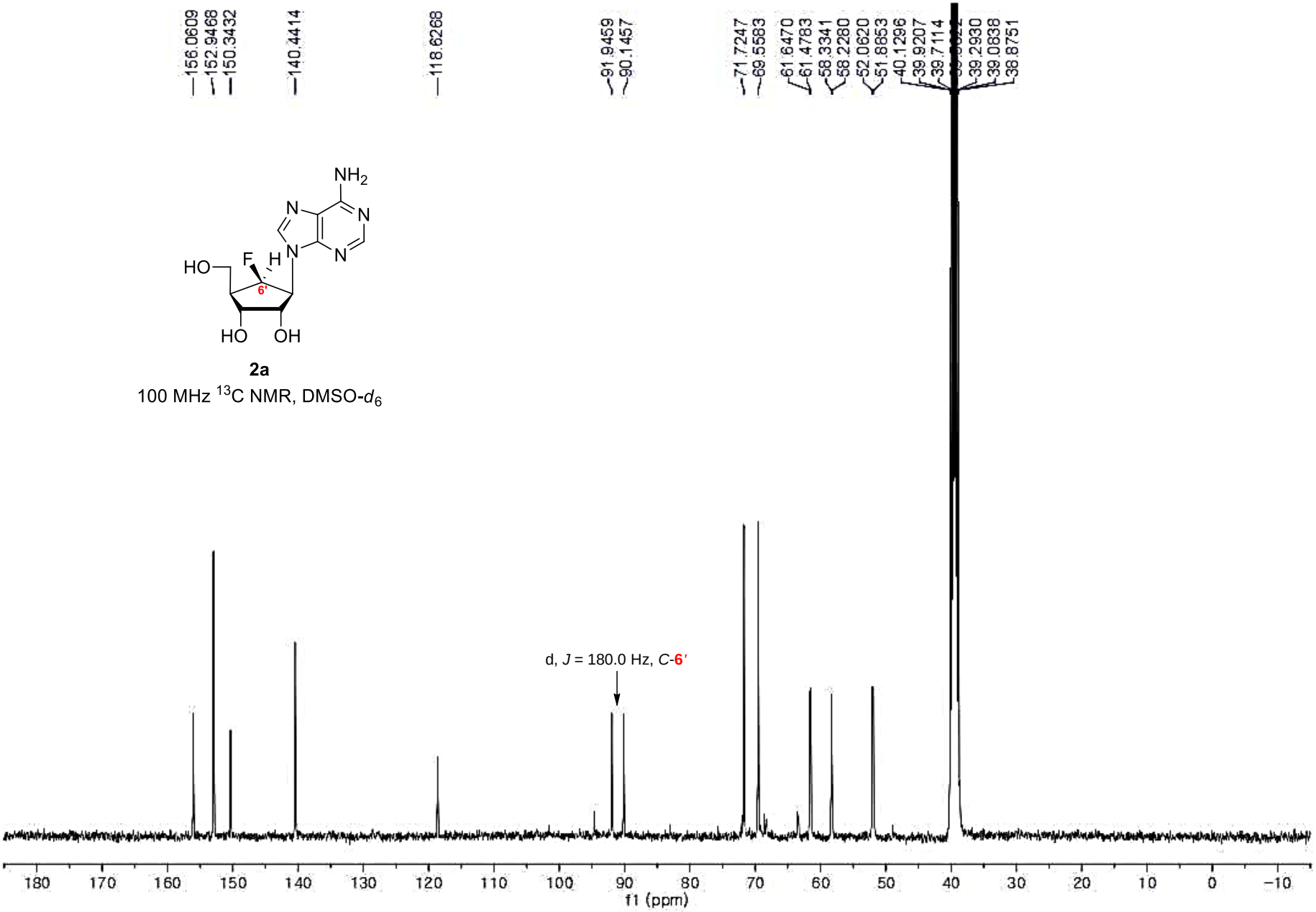
$CDCl_3$

13b

282 MHz ^{19}F NMR, $CDCl_3$

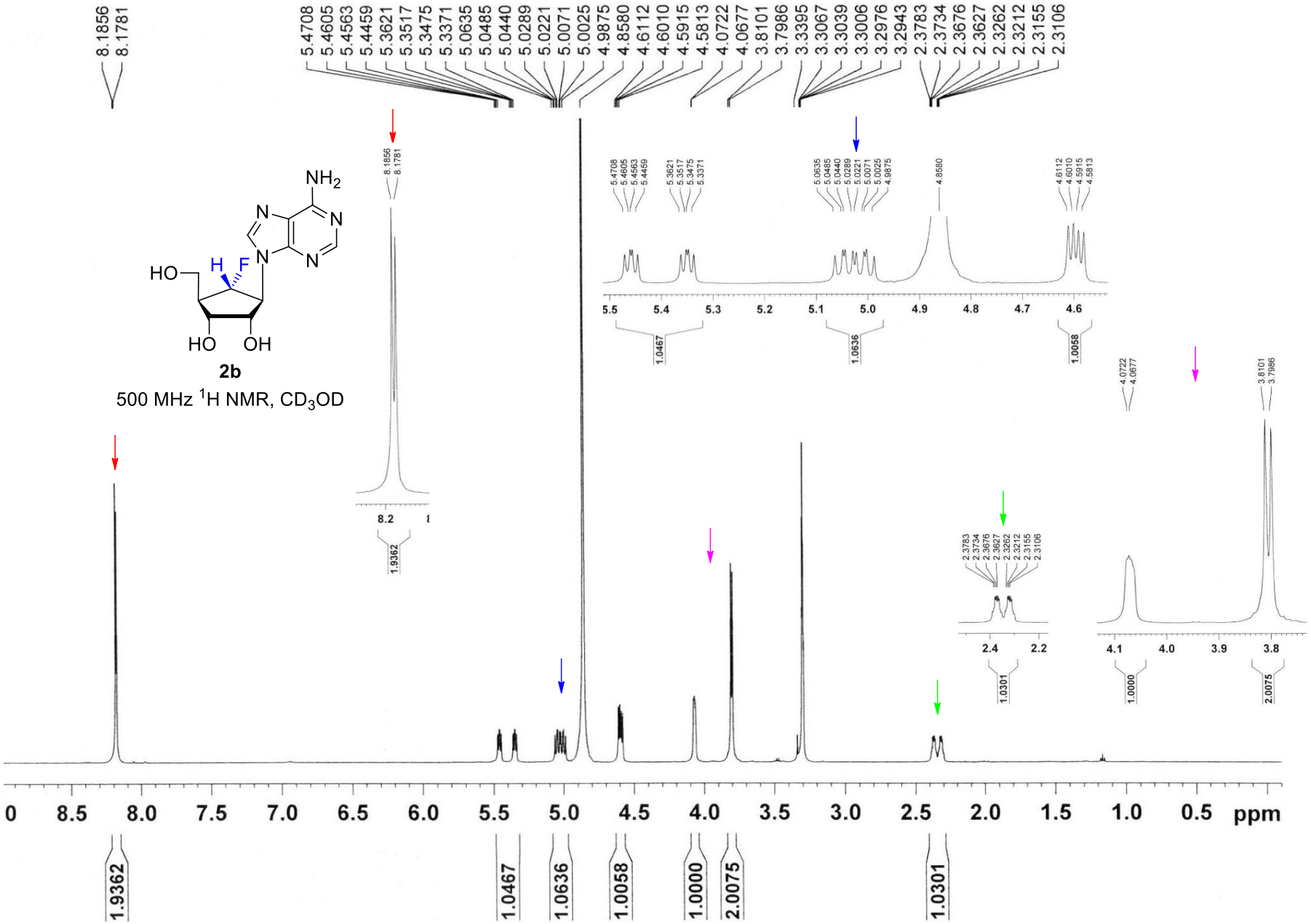


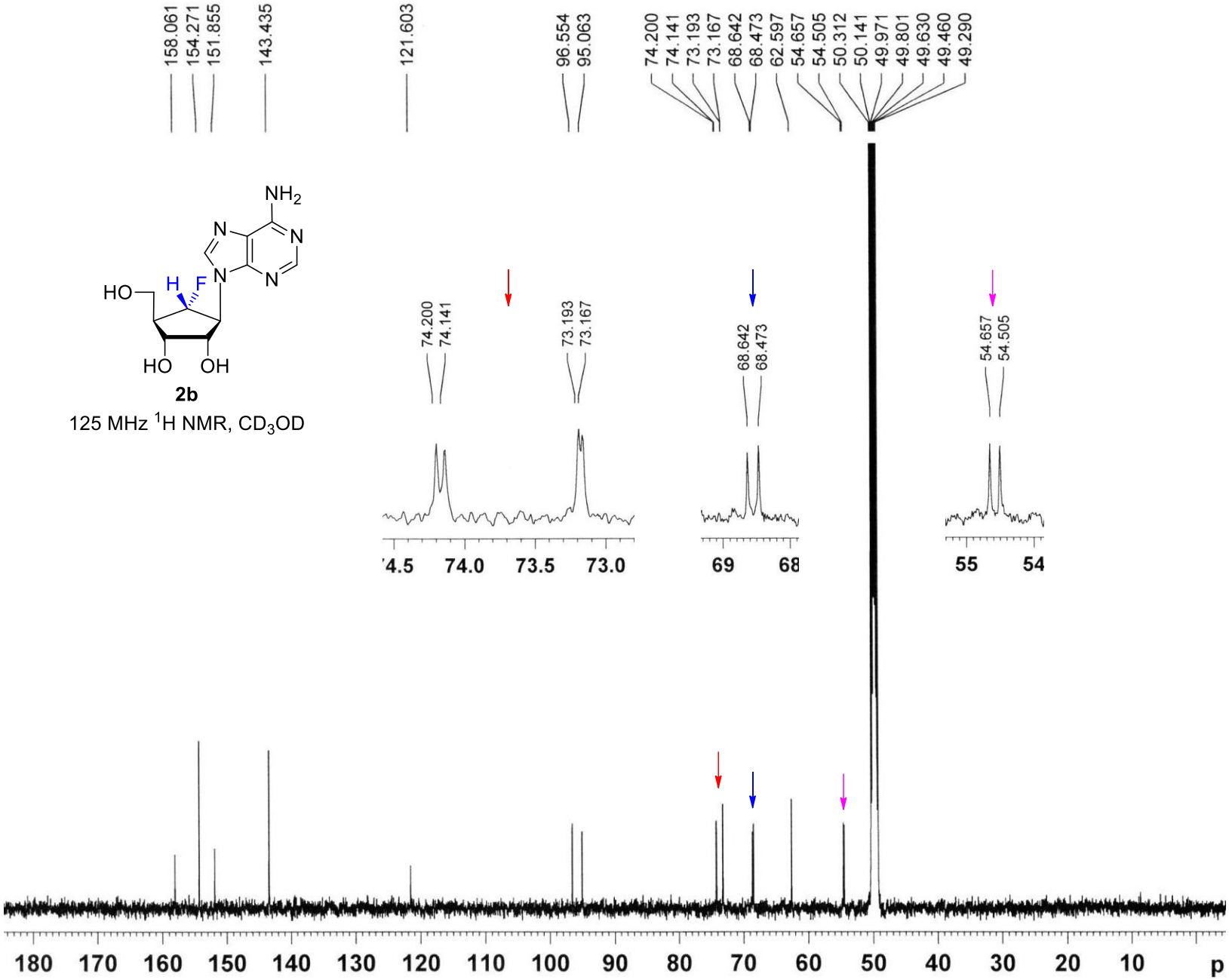

NH2
N
N
N
N
HO
F
H
6'
HO
OH
2a
800 MHz 1H NMR, CD3OD
J = 55.2 Hz, H-6'
8
7
6
5
4
3
2
1
0
1.0000
1.0006
1.0369
1.0739
1.0960
0.5789
1.0494
2.0706
1.0578
1.3756
1.2906

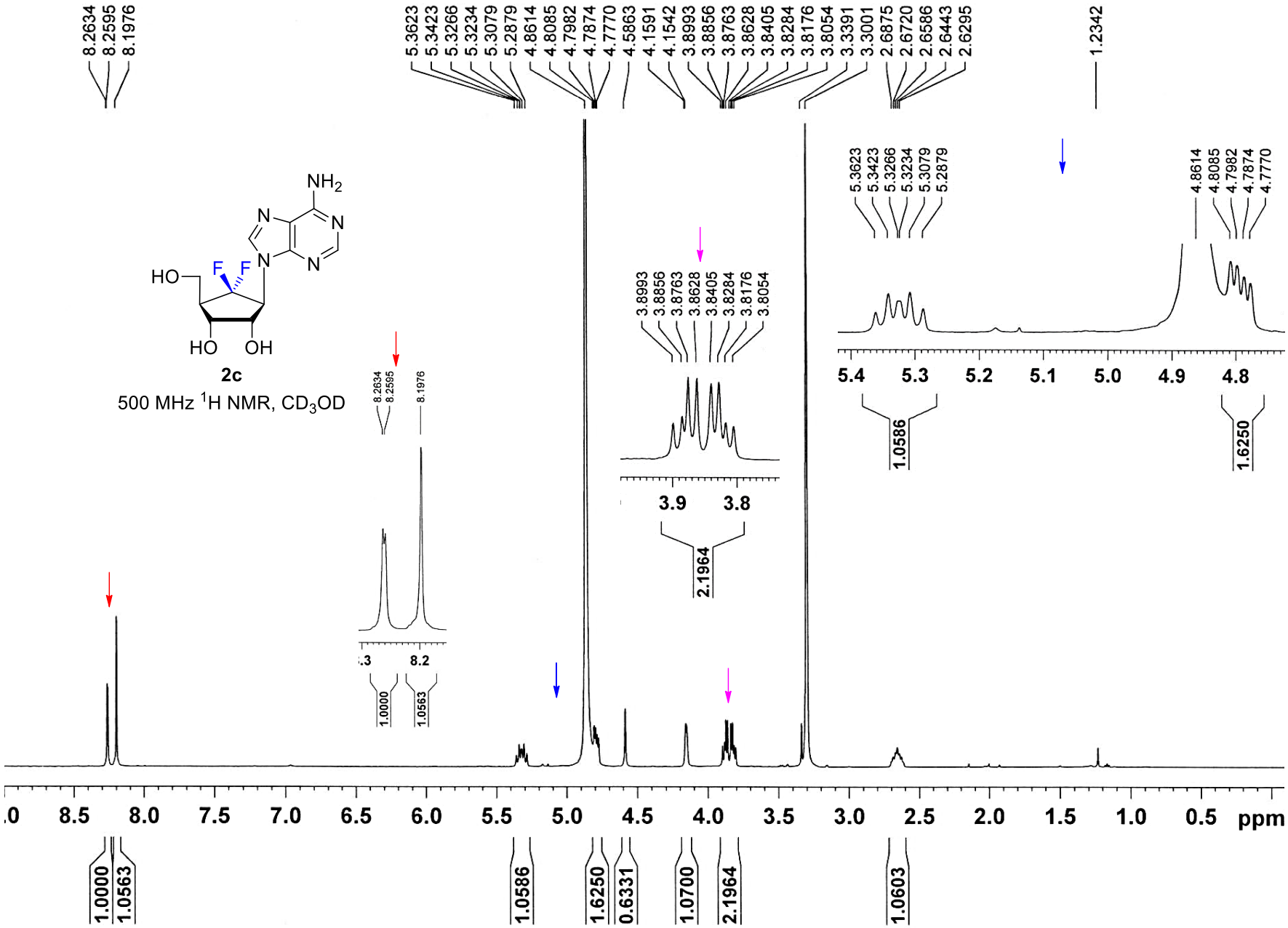


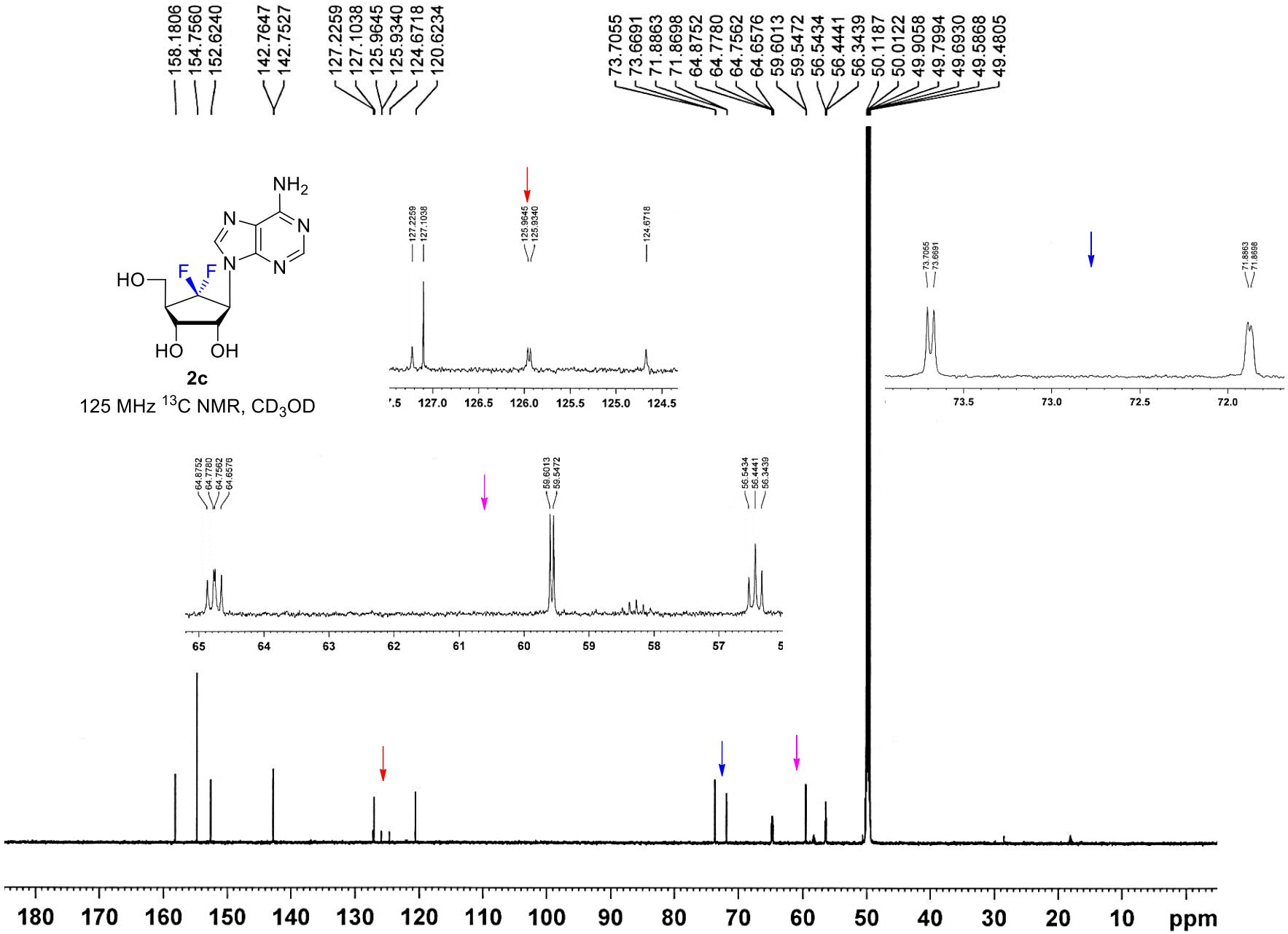
2a

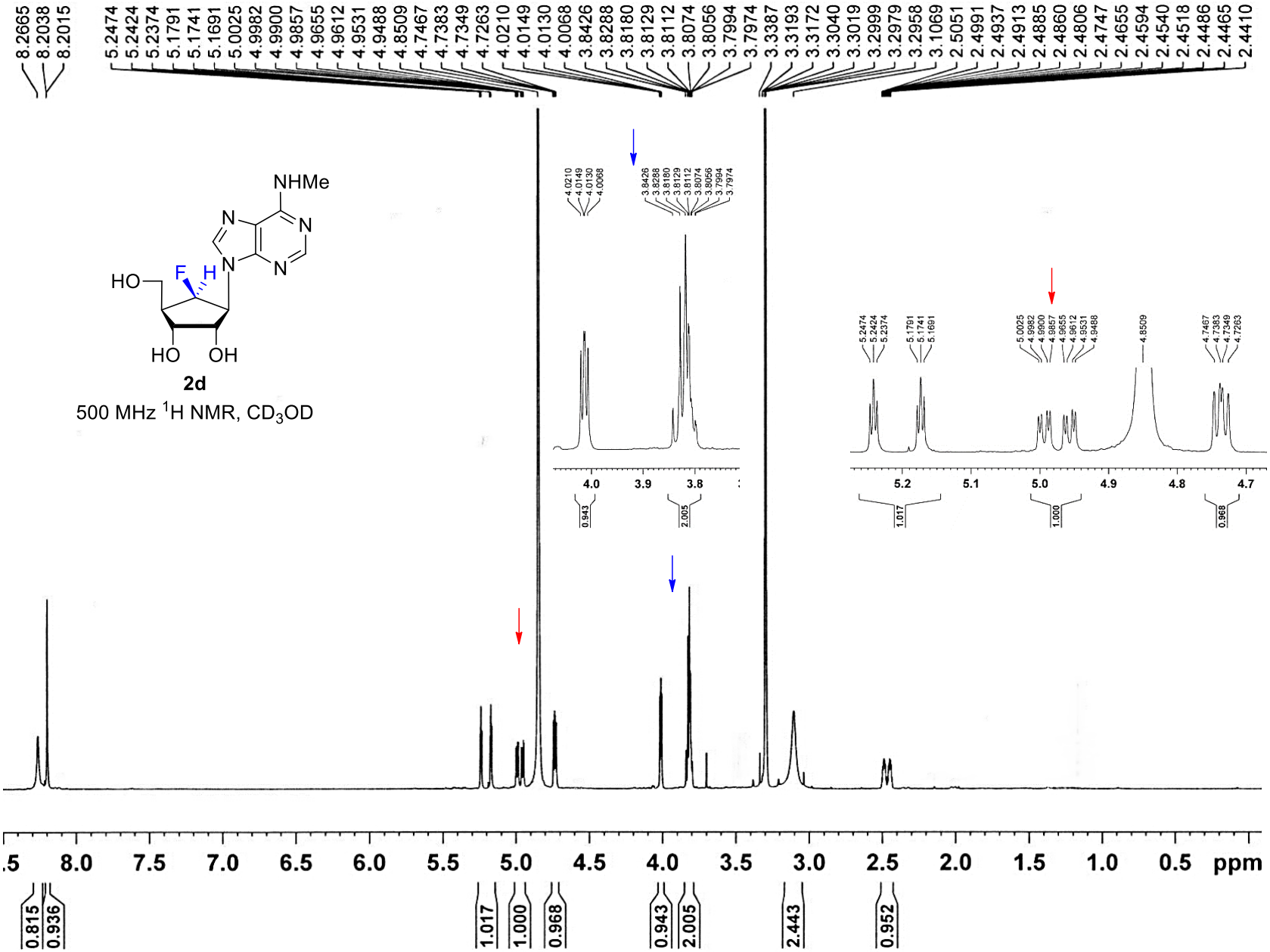
100 MHz ^{13}C NMR, DMSO-d_6

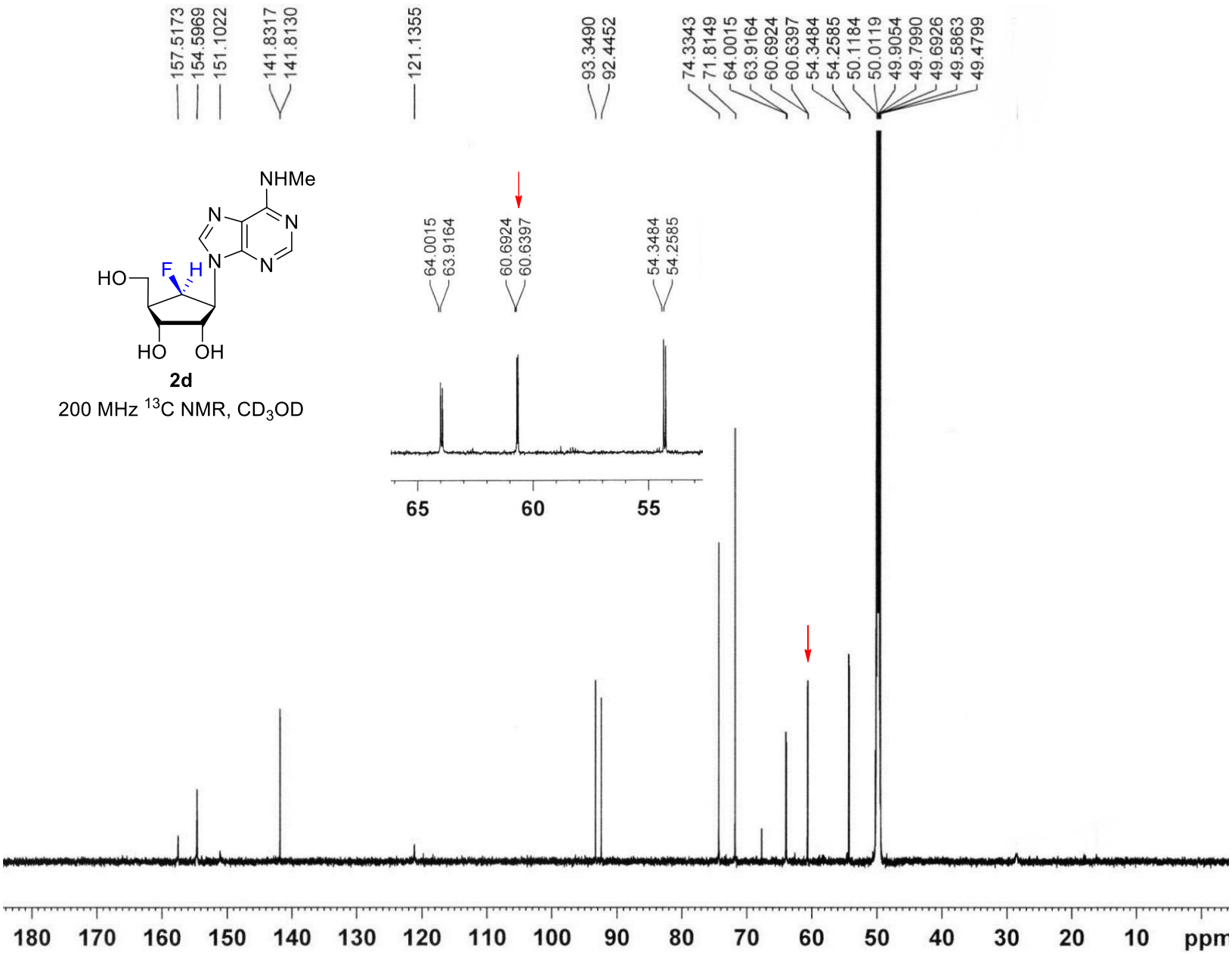

2b
500 MHz ^{1}H NMR, CD_3OD
ppm

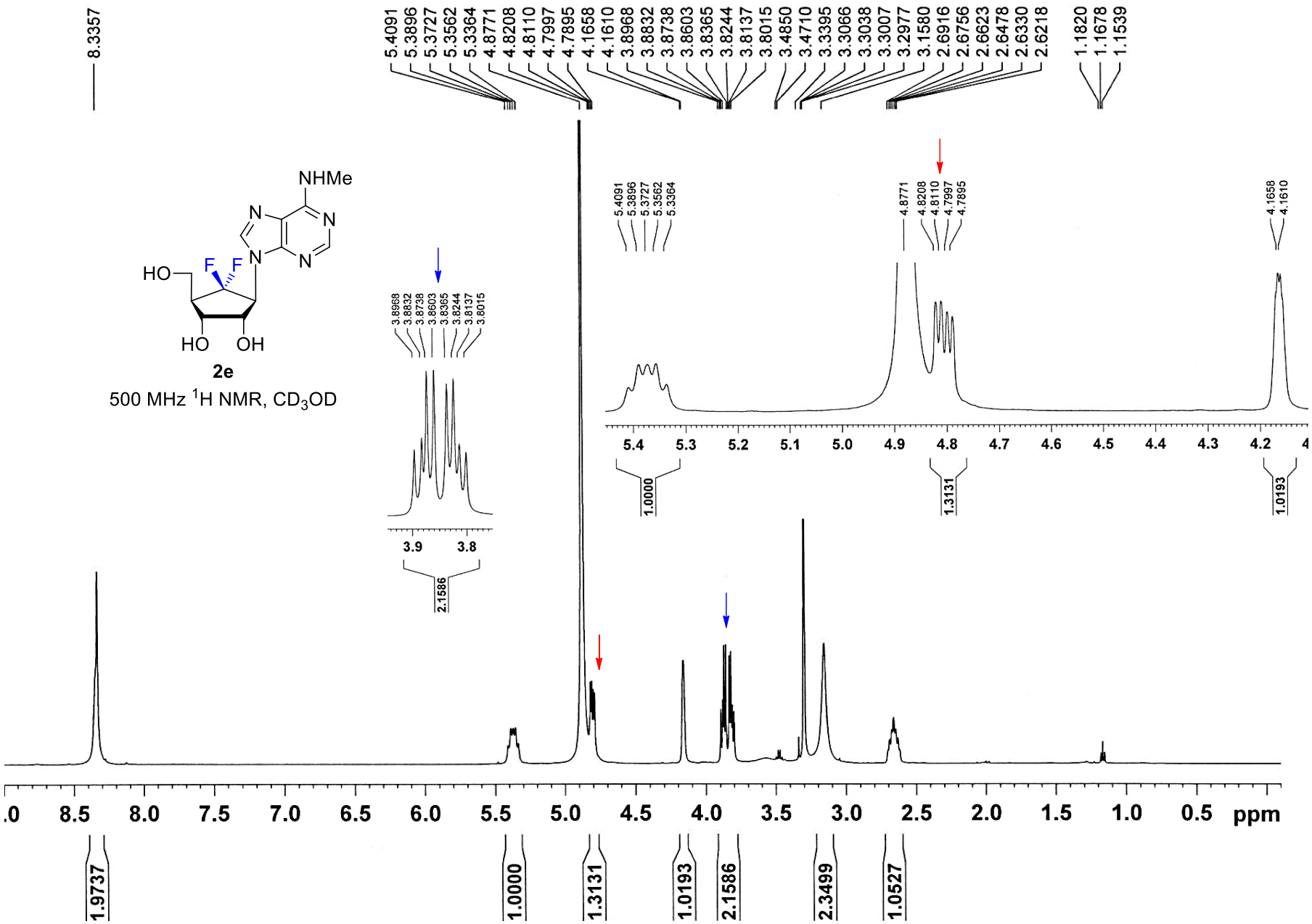

158.061
154.271
151.855
143.435
121.603
96.554
95.063
74.200
74.141
73.193
73.167
68.642
68.473
62.597
54.657
54.505
50.312
50.141
49.971
49.801
49.630
49.460
49.290
NH2
N
N
N
N
HO
H
F
HO
OH
2b
125 MHz 1H NMR, CD3OD
74.200
74.141
73.193
73.167
68.642
68.473
54.657
54.505
74.5
74.0
73.5
73.0
69
68
55
54
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
p

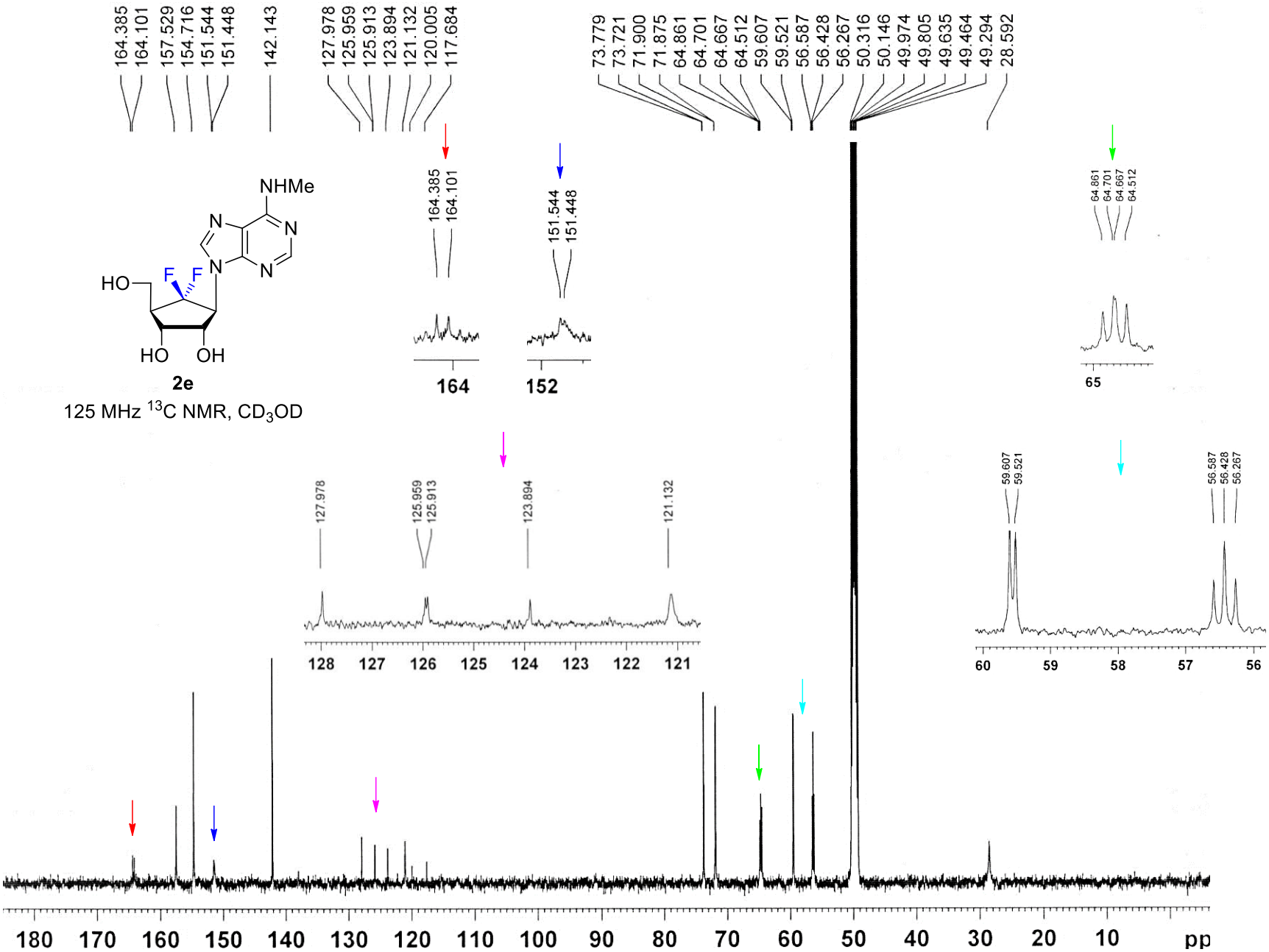

2c
500 MHz 1H NMR, CD3OD
NH2
HO
F
F
HO
OH
8.2634
8.2595
8.1976
5.3623
5.3423
5.3266
5.3234
5.3079
5.2879
4.8614
4.8085
4.7982
4.7874
4.7770
4.5863
4.1591
4.1542
3.8993
3.8856
3.8763
3.8628
3.8405
3.8284
3.8176
3.8054
3.3391
3.3001
2.6875
2.6720
2.6586
2.6443
2.6295
1.2342
1.0000
1.0563
1.0586
1.6250
0.6331
1.0700
2.1964
1.0603
ppm

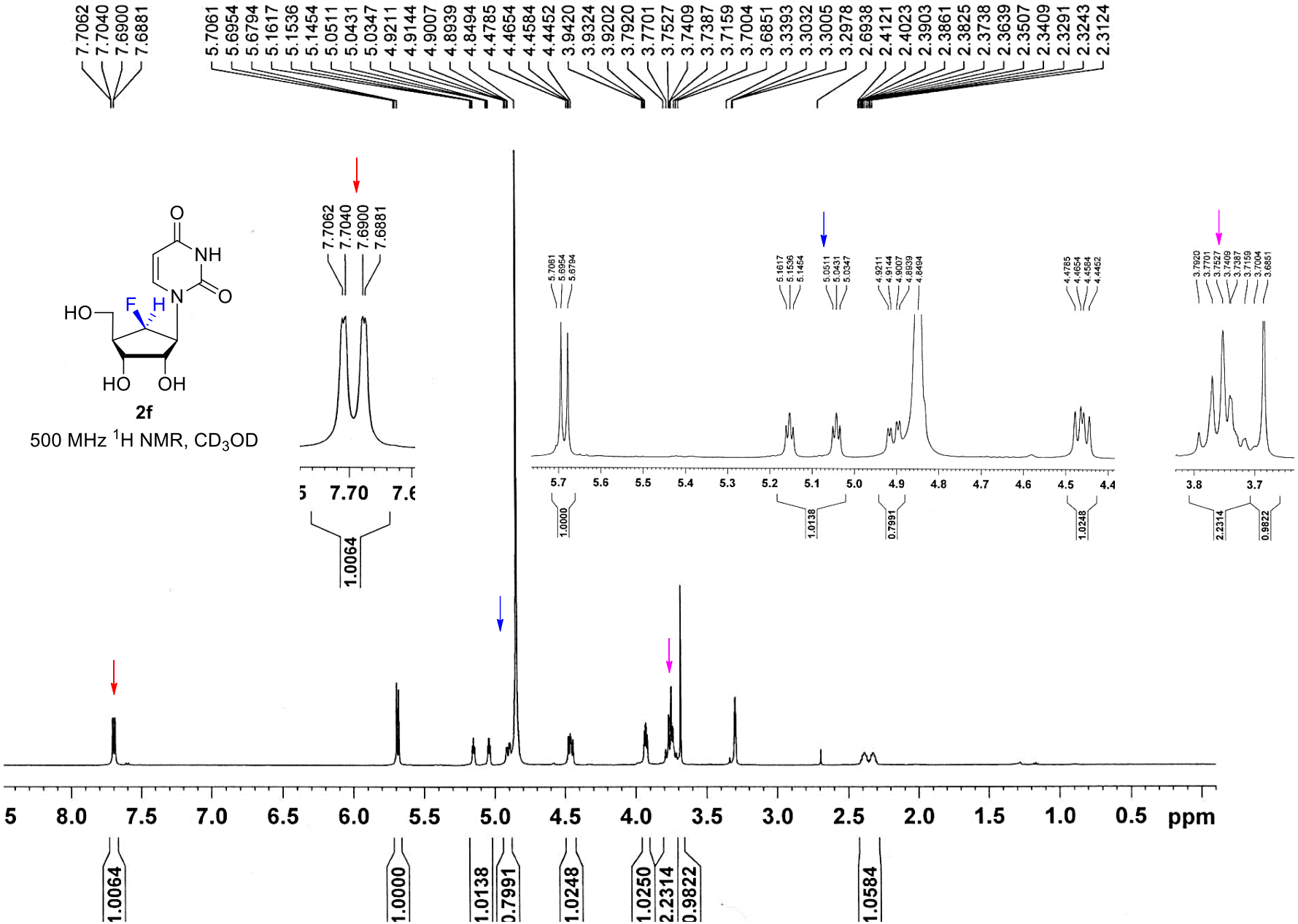

158.1806
154.7560
152.6240
142.7647
142.7527
127.2259
127.1038
125.9645
125.9340
124.6718
120.6234
73.7055
73.6691
71.8863
71.8698
64.8752
64.7780
64.7562
64.6576
59.6013
59.5472
56.5434
56.4441
56.3439
50.1187
50.0122
49.9058
49.7994
49.6930
49.5868
49.4805
NH2
N
N
N
N
HO
F
F
HO
OH
2c
125 MHz 13C NMR, CD3OD
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

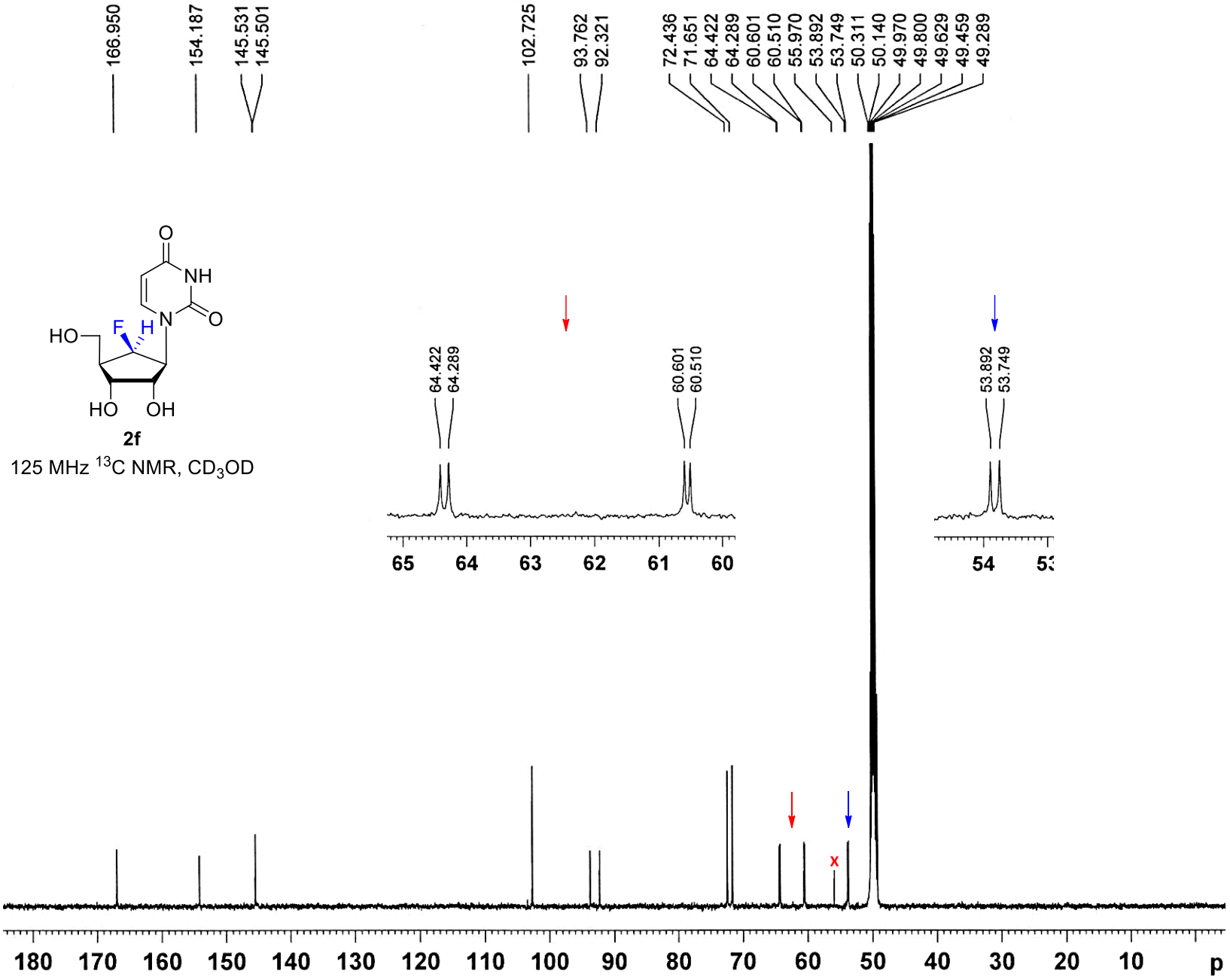

NHMe
N
N
N
N
HO
F
H
HO
OH
2d
500 MHz 1H NMR, CD3OD
8.2665
8.2038
8.2015
5.2474
5.2424
5.2374
5.1791
5.1741
5.1691
5.0025
4.9982
4.9900
4.9857
4.9655
4.9612
4.9531
4.9488
4.8509
4.7467
4.7383
4.7349
4.7263
4.0210
4.0149
4.0130
4.0068
3.8426
3.8288
3.8180
3.8129
3.8112
3.8074
3.8056
3.7994
3.7974
3.3387
3.3193
3.3172
3.3040
3.3019
3.2999
3.2979
3.2958
3.1069
2.5051
2.4991
2.4937
2.4913
2.4885
2.4860
2.4806
2.4747
2.4655
2.4594
2.4540
2.4518
2.4486
2.4465
2.4410
ppm
0.815
0.936
1.017
1.000
0.968
0.943
2.005
2.443
0.952

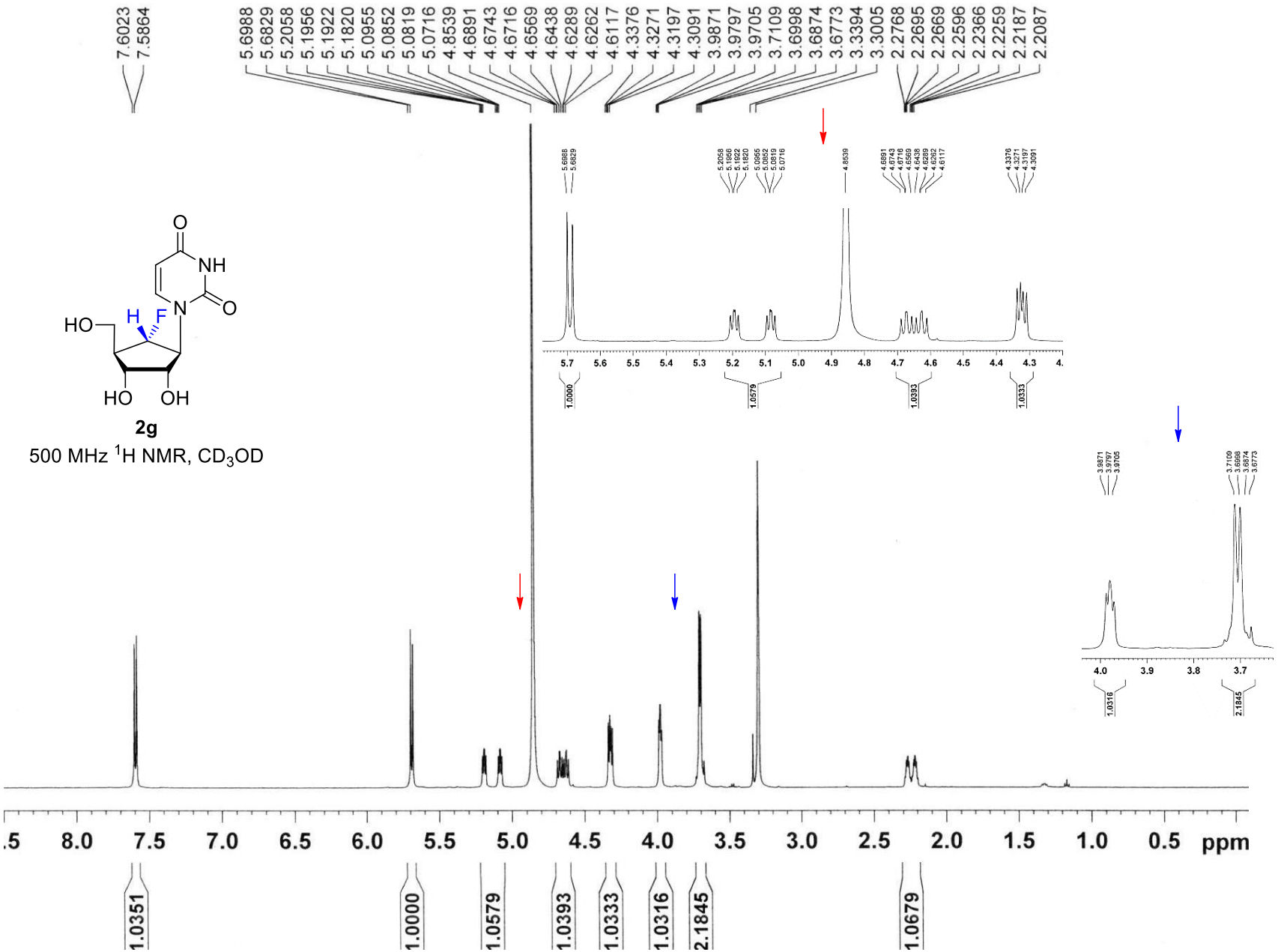

157.5173
154.5969
151.1022
141.8317
141.8130
121.1355
93.3490
92.4452
74.3343
71.8149
64.0015
63.9164
60.6924
60.6397
54.3484
54.2585
50.1184
50.0119
49.9054
49.7990
49.6926
49.5863
49.4799
NHMe
N
N
N
N
HO
F
H
HO
OH
2d
200 MHz 13C NMR, CD3OD
64.0015
63.9164
60.6924
60.6397
54.3484
54.2585
65
60
55
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


NHMe
N
N
N
N
HO
F
F
HO
OH
2e
500 MHz ^{1}H NMR, CD_3OD
8.3357
5.4091
5.3896
5.3727
5.3562
5.3364
4.8771
4.8208
4.8110
4.7997
4.7895
4.1658
4.1610
3.8968
3.8832
3.8738
3.8603
3.8365
3.8244
3.8137
3.8015
3.4850
3.4710
3.3395
3.3066
3.3038
3.3007
3.2977
3.1580
2.6916
2.6756
2.6623
2.6478
2.6330
2.6218
1.1820
1.1678
1.1539
1.0000
1.3131
1.0193
2.1586
1.9737
2.3499
1.0527
ppm


164.385
164.101
157.529
154.716
151.544
151.448
142.143
127.978
125.959
125.913
123.894
121.132
120.005
117.684
73.779
73.721
71.900
71.875
64.861
64.701
64.667
64.512
59.607
59.521
56.587
56.428
56.267
50.316
50.146
49.974
49.805
49.635
49.464
49.294
28.592
NHMe
N
N
N
N
HO
F
F
HO
OH
2e
125 MHz 13C NMR, CD3OD
164
152
65
128
127
126
125
124
123
122
121
60
59
58
57
56
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
pp

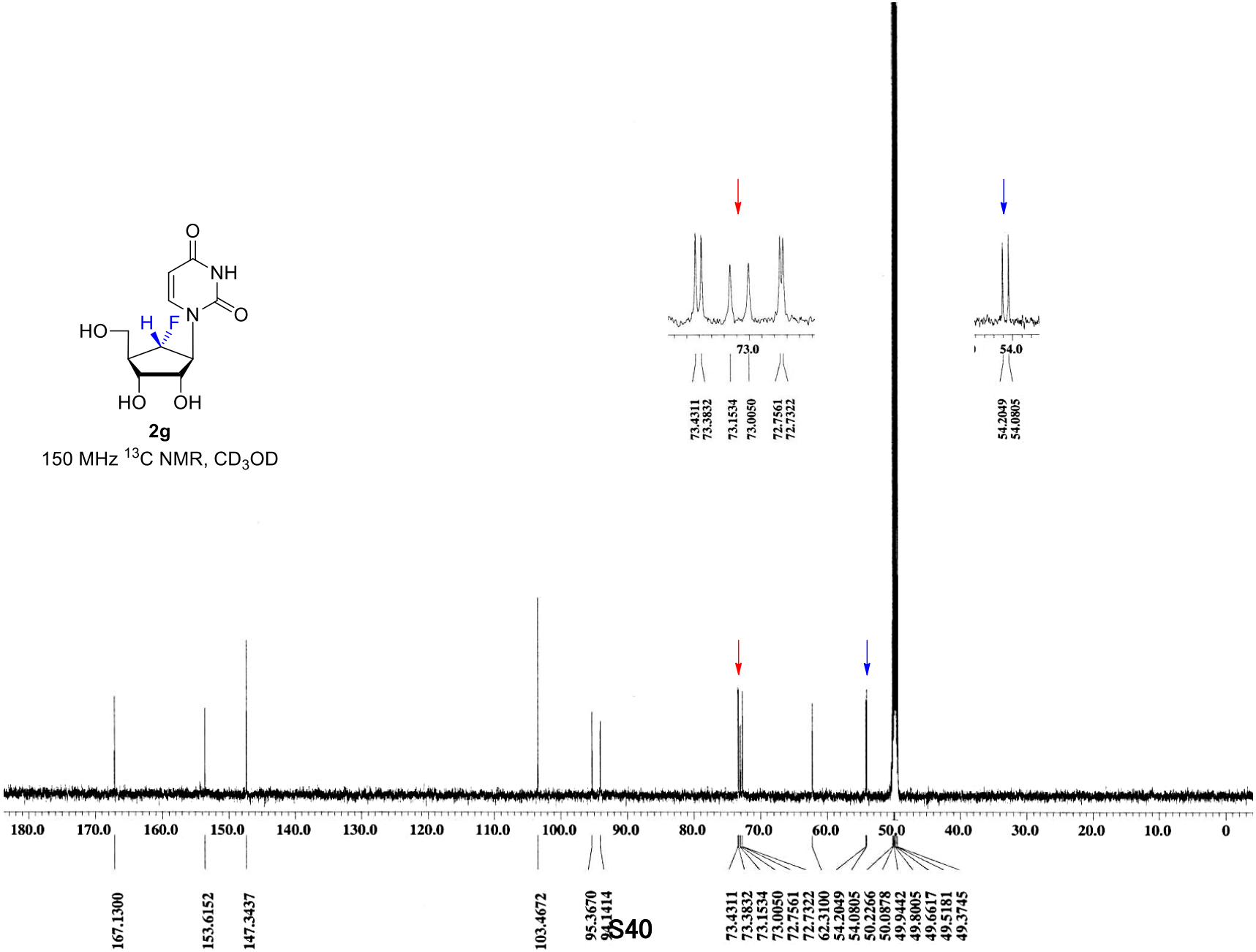

2f
500 MHz 1H NMR, CD3OD
ppm

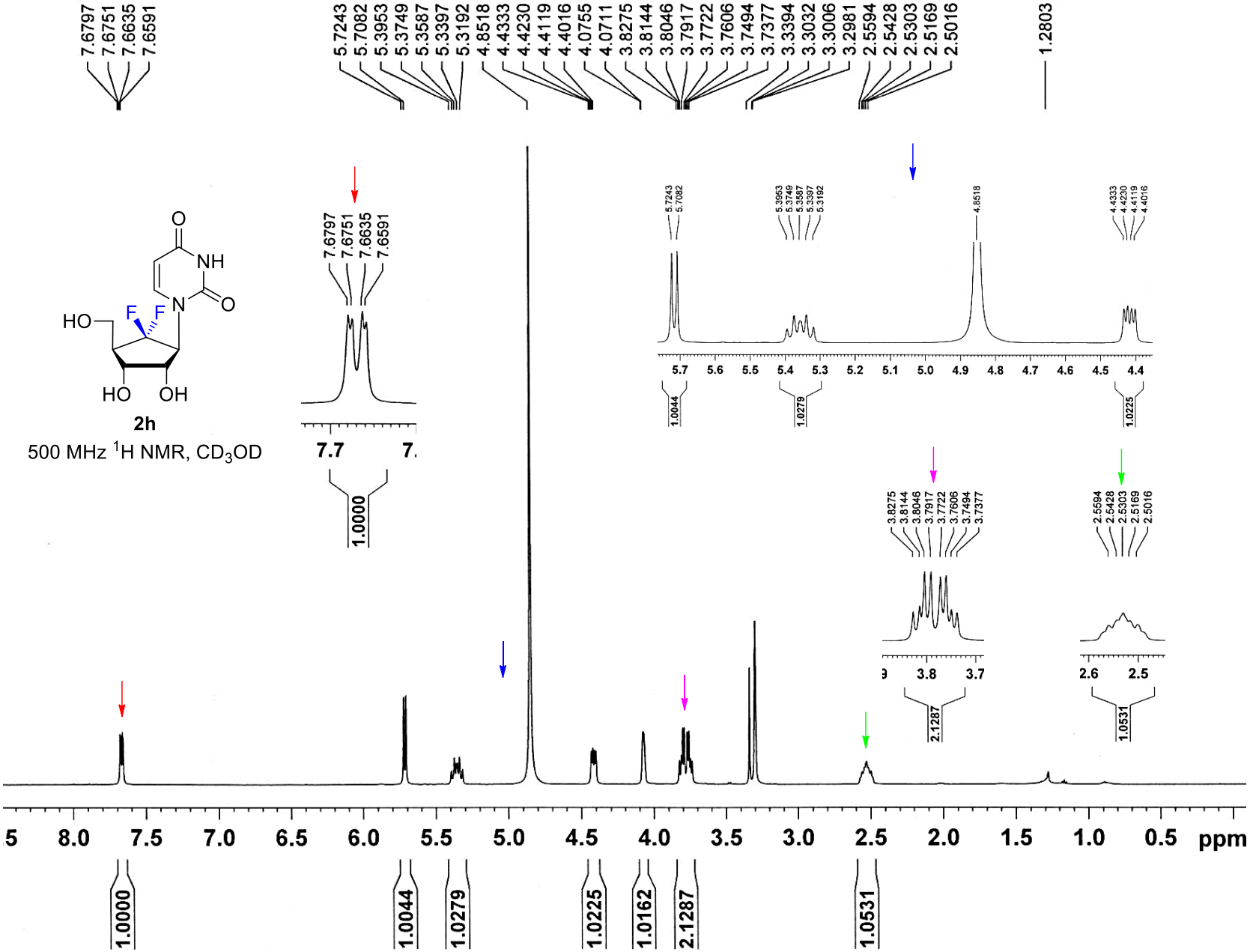

166.950
154.187
145.531
145.501
102.725
93.762
92.321
72.436
71.651
64.422
64.289
60.601
60.510
55.970
53.892
53.749
50.311
50.140
49.970
49.800
49.629
49.459
49.289
O
NH
O
N
HO
F
H
HO
OH
2f
125 MHz 13C NMR, CD3OD
64.422
64.289
60.601
60.510
53.892
53.749
65
64
63
62
61
60
54
53
x
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
p


2g
500 MHz ^{1}H NMR, CD_3OD
ppm

2g

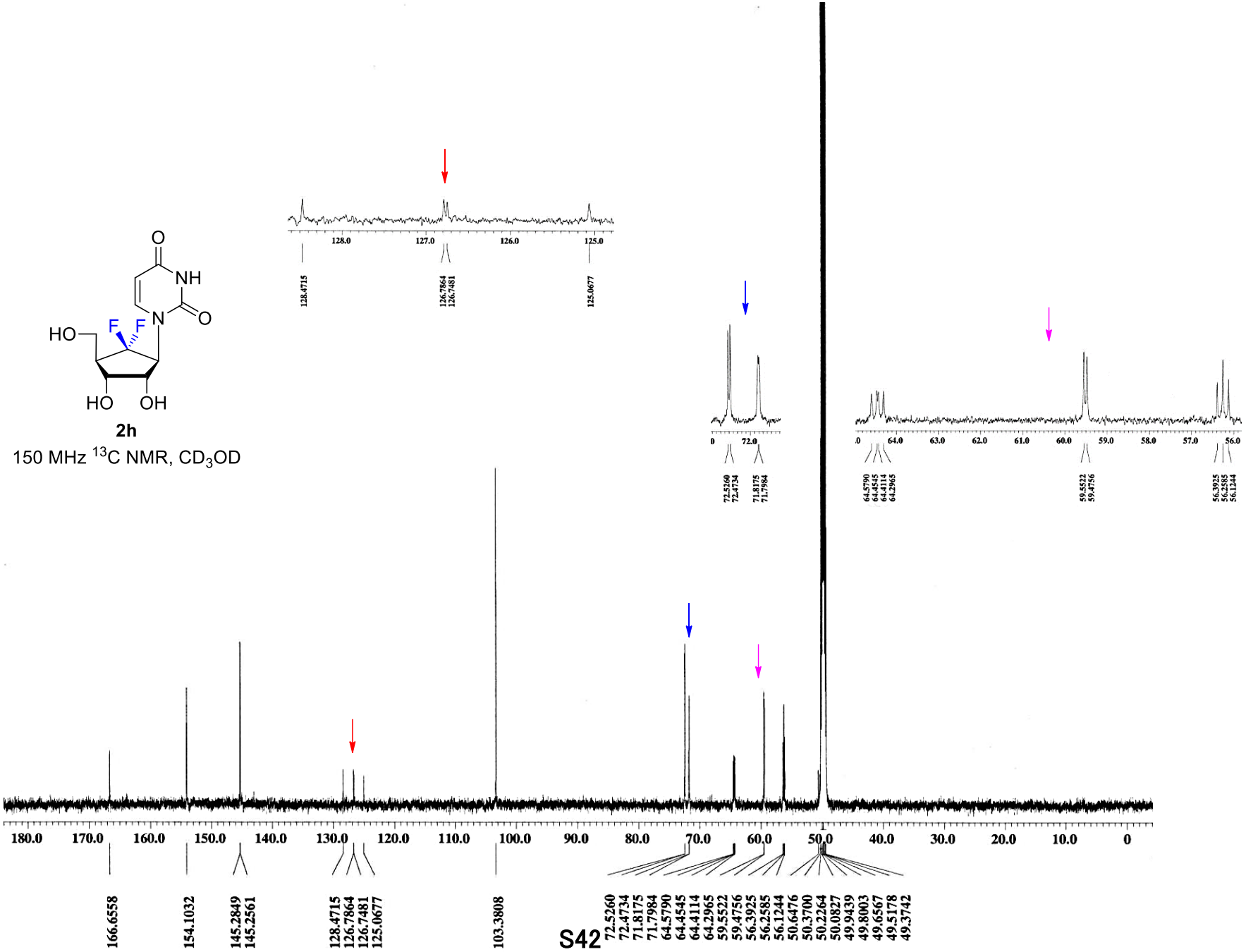
150 MHz ^{13}C NMR, CD_3OD

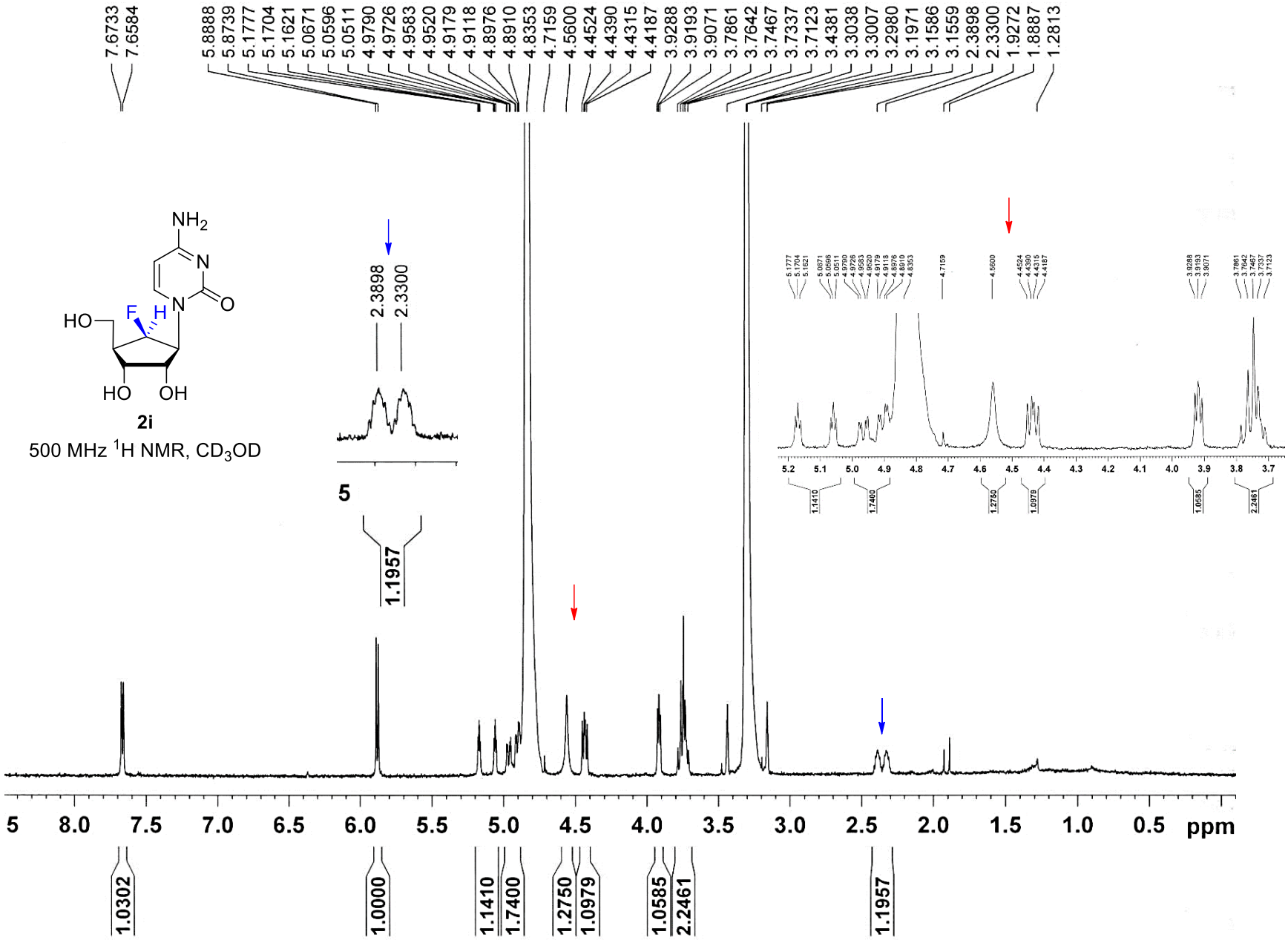


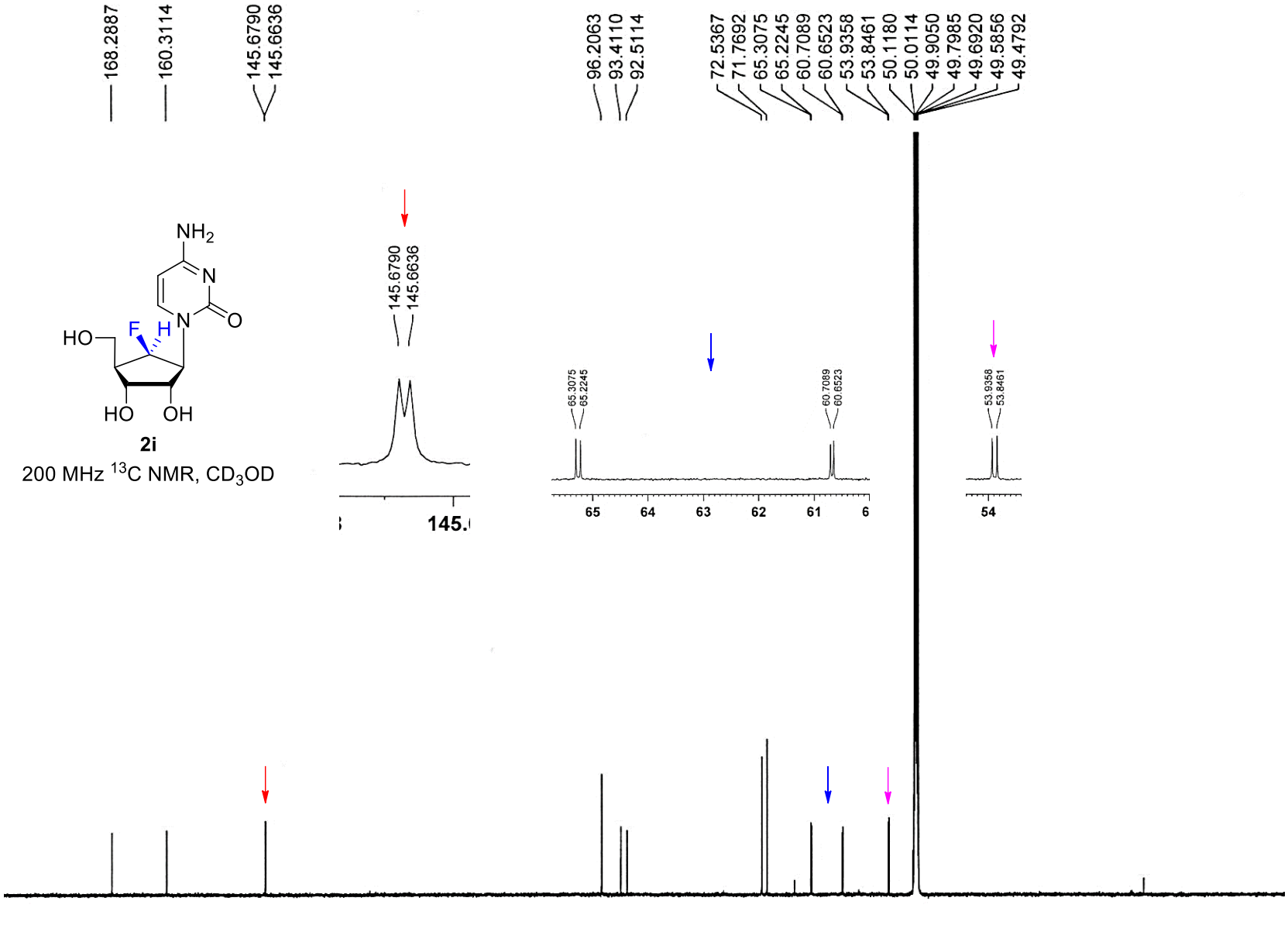

7.6797
7.6751
7.6635
7.6591
5.7243
5.7082
5.3953
5.3749
5.3587
5.3397
5.3192
4.8518
4.4333
4.4230
4.4119
4.4016
4.0755
4.0711
3.8275
3.8144
3.8046
3.7917
3.7722
3.7606
3.7494
3.7377
3.3394
3.3032
3.3006
3.2981
2.5594
2.5428
2.5303
2.5169
2.5016
1.2803
O
NH
N
O
HO
F
F
HO
OH
2h
500 MHz 1H NMR, CD3OD
1.0000
1.0044
1.0279
1.0225
2.1287
1.0531
1.0162
ppm

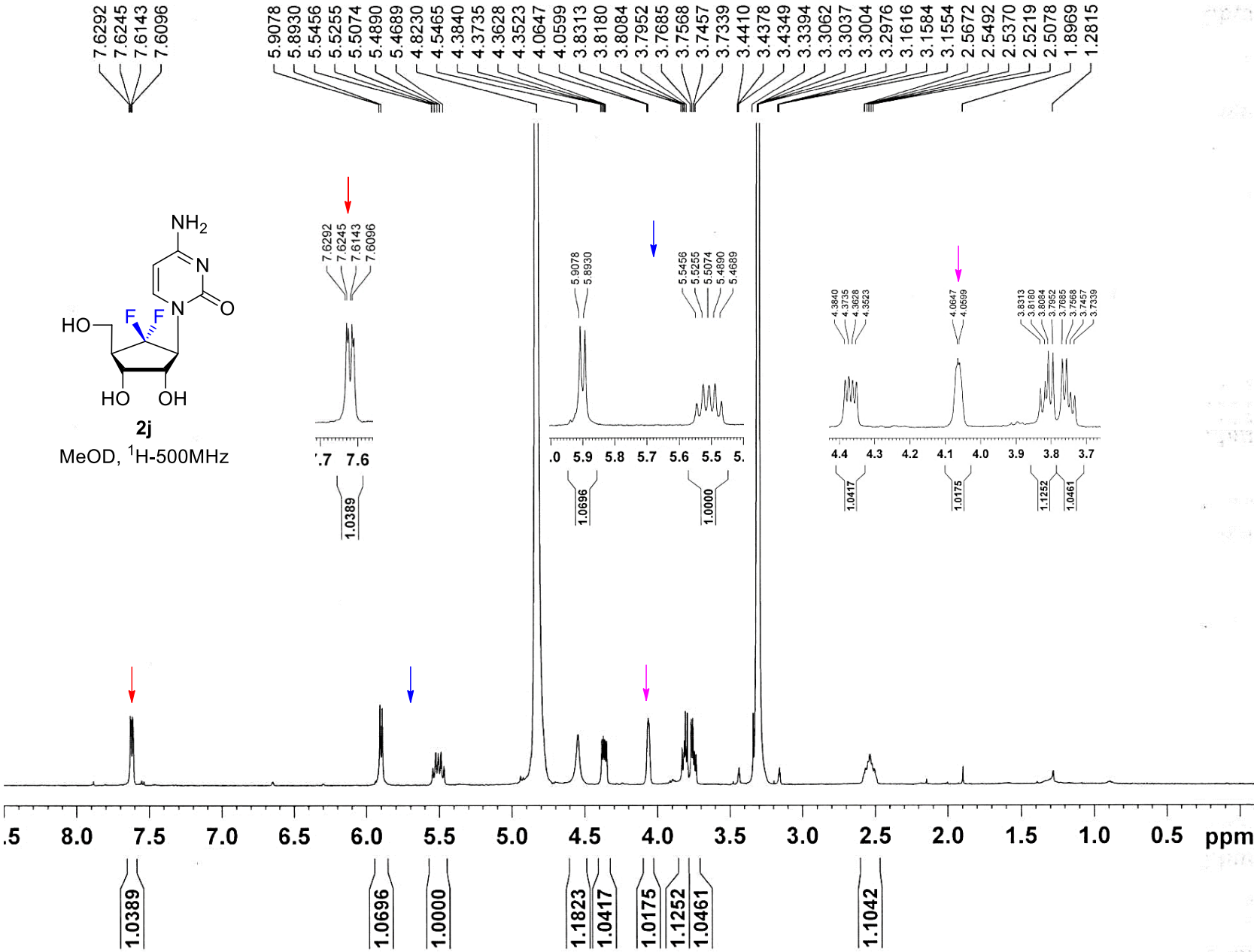
2h

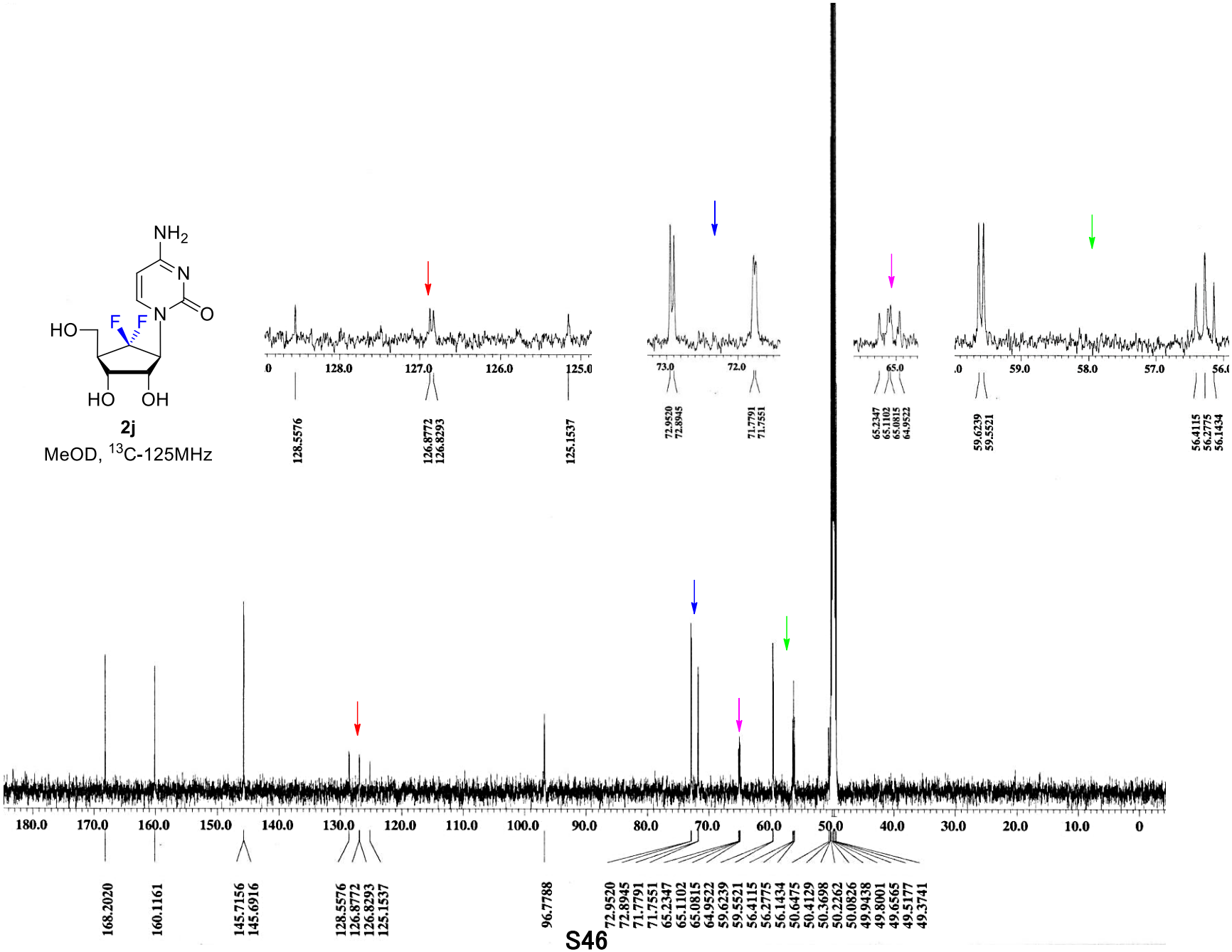
150 MHz ^{13}C NMR, CD_3OD




2i
500 MHz 1H NMR, CD3OD
5
ppm


168.2887
160.3114
145.6790
145.6636
96.2063
93.4110
92.5114
72.5367
71.7692
65.3075
65.2245
60.7089
60.6523
53.9358
53.8461
50.1180
50.0114
49.9050
49.7985
49.6920
49.5856
49.4792
NH2
N
N
O
HO
F
H
HO
OH
2i
200 MHz 13C NMR, CD3OD
145.6790
145.6636
65.3075
65.2245
60.7089
60.6523
53.9358
53.8461
65
64
63
62
61
54
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


2j
MeOD, 1H-500MHz
ppm


NH2
N
O
N
HO
F
F
HO
OH
2j
MeOD, 13C-125MHz
128.5576
126.8772
126.8293
125.1537
72.9520
72.8945
71.7791
71.7551
65.2347
65.1102
65.0815
64.9522
59.6239
59.5521
56.4115
56.2775
56.1434
168.2020
160.1161
145.7156
145.6916
96.7788
72.9520
72.8945
71.7791
71.7551
65.2347
65.1102
65.0815
64.9522
59.6239
59.5521
56.4115
56.2775
56.1434
50.6475
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50.3698
50.2262
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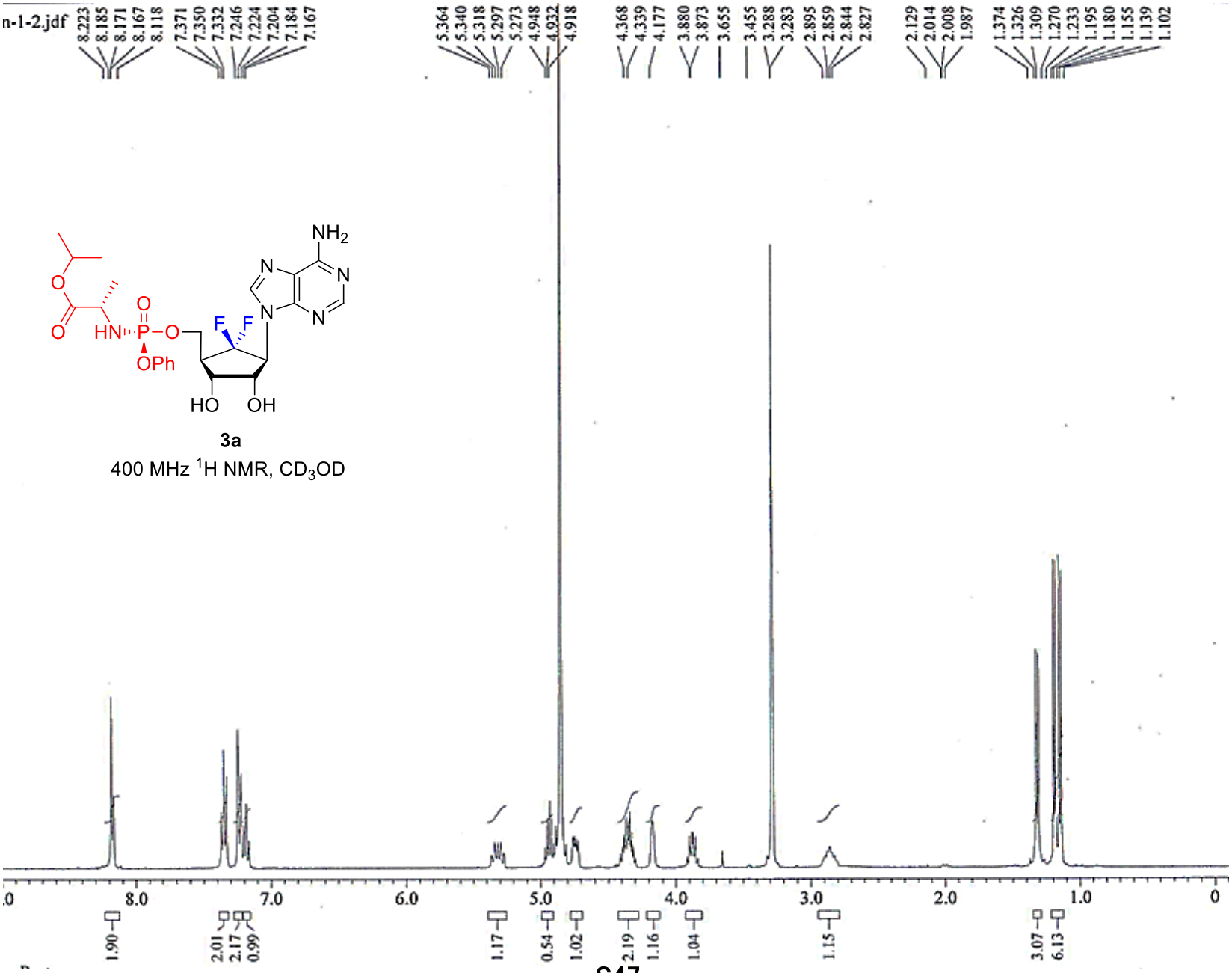
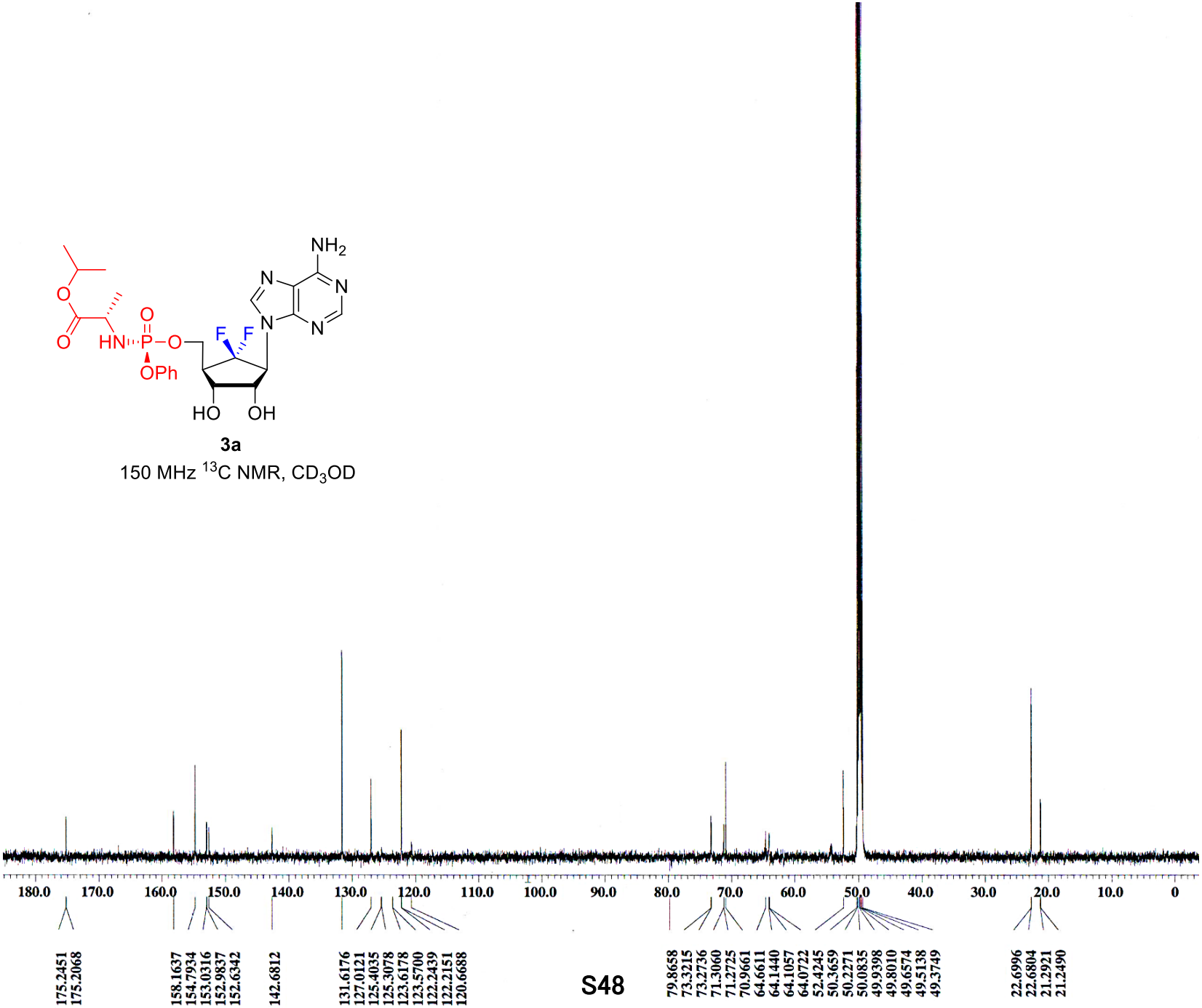
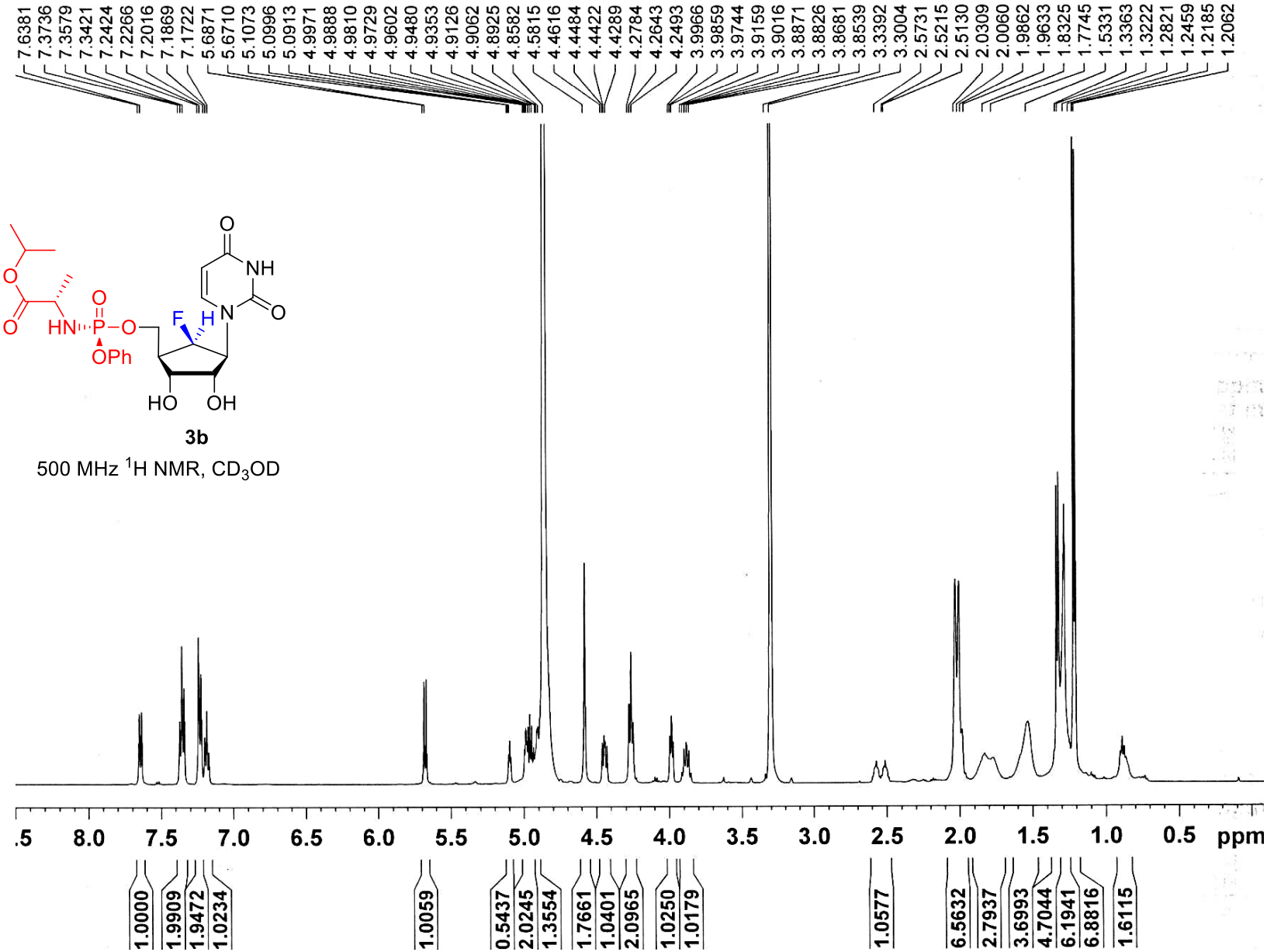
49.9438
49.8001
49.6565
49.5177
49.3741


n-1-2.jdf
8.223 8.185 8.171 8.167 8.118 7.371 7.350 7.332 7.246 7.224 7.204 7.184 7.167
5.364 5.340 5.318 5.297 5.273 4.948 4.932 4.918 4.368 4.339 4.177 3.880 3.873 3.655 3.455 3.288 3.283 2.895 2.859 2.844 2.827 2.129 2.014 2.008 1.987 1.374 1.326 1.309 1.270 1.233 1.195 1.180 1.155 1.139 1.102
NH2
N
N
N
N
O
O
HN
P
O
O
OPh
F
F
HO
OH
3a
400 MHz 1H NMR, CD3OD
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.90
2.01
2.17
0.99
1.17
0.54
1.02
2.19
1.16
1.04
1.15
3.07
6.13

3a

150 MHz ^{13}C NMR, CD_3OD

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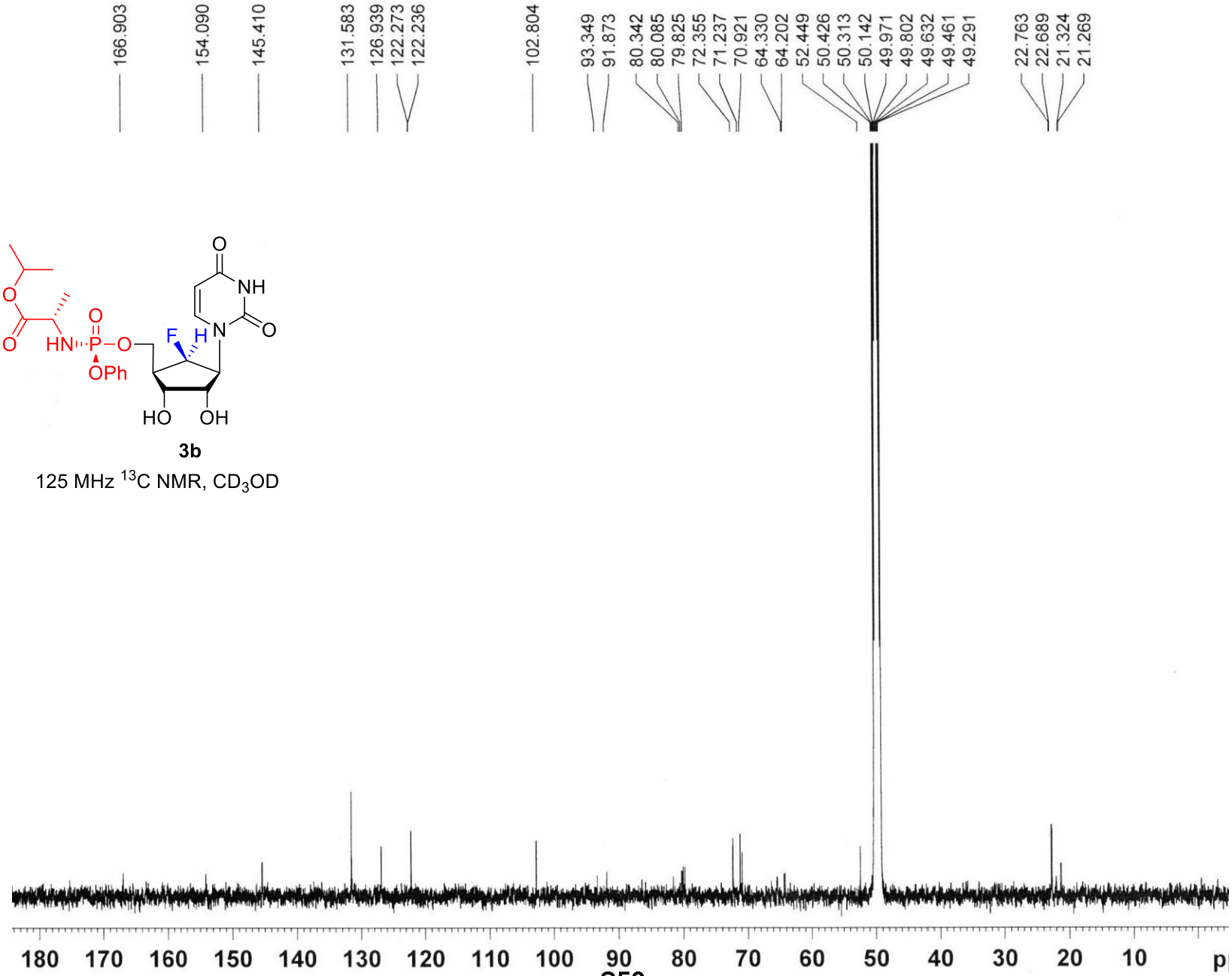
7.3736
7.3579
7.3421
7.2424
7.2266
7.2016
7.1869
7.1722
5.6871
5.6710
5.1073
5.0996
5.0913
4.9971
4.9888
4.9810
4.9729
4.9602
4.9480
4.9353
4.9126
4.9062
4.8925
4.8582
4.5815
4.4616
4.4484
4.4422
4.4289
4.2784
4.2643
4.2493
3.9966
3.9859
3.9744
3.9159
3.9016
3.8871
3.8826
3.8681
3.8539
3.3392
3.3004
2.5731
2.5215
2.5130
2.0309
2.0060
1.9862
1.9633
1.8325
1.7745
1.5331
1.3363
1.3222
1.2821
1.2459
1.2185
1.2062
O
NH
O
N
F
H
O
HN
P
O
OPh
O
HO
OH
3b
500 MHz 1H NMR, CD3OD
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.0000
1.9909
1.9472
1.0234
1.0059
0.5437
2.0245
1.3554
1.7661
1.0401
2.0965
1.0250
1.0179
1.0577
6.5632
2.7937
3.6993
4.7044
6.1941
6.8816
1.6115

3b

125 MHz ^{13}C NMR, CD_3OD



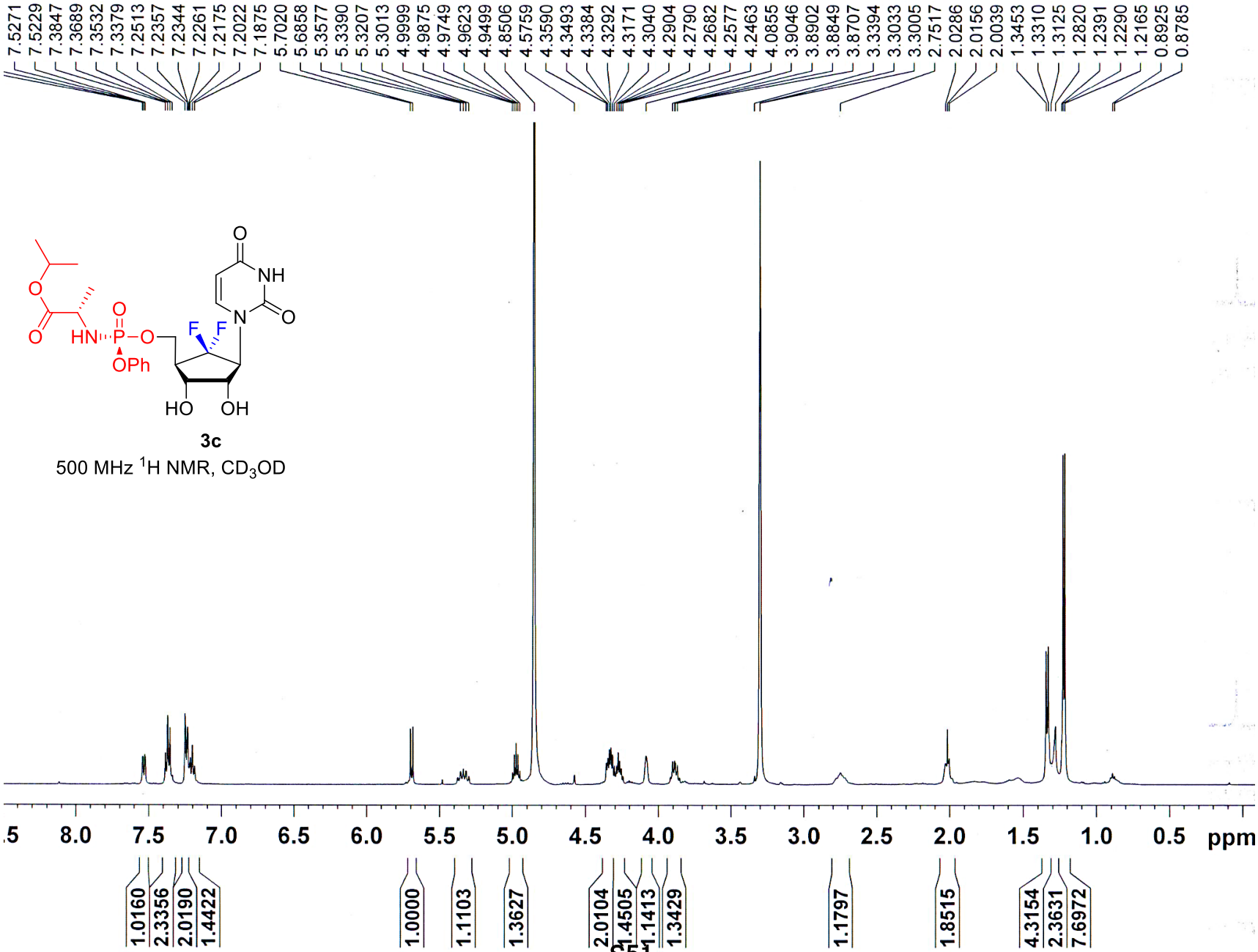
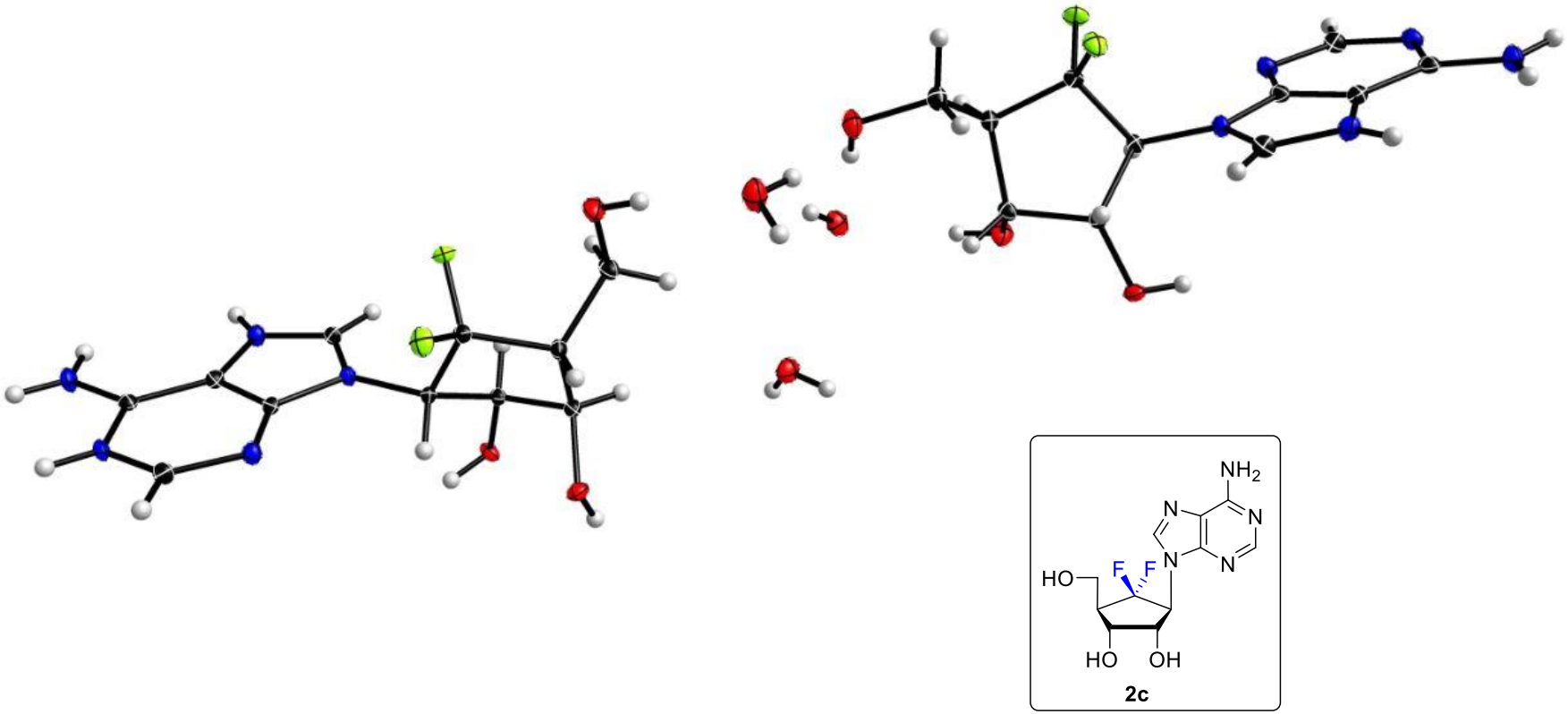
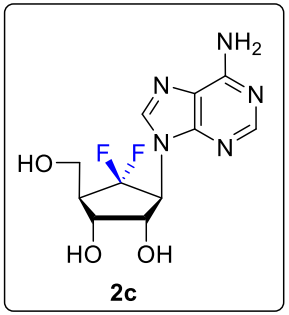

7.5271 7.5229 7.3847 7.3689 7.3532 7.3379 7.2513 7.2357 7.2344 7.2261 7.2175 7.2022 7.1875 5.7020 5.6858 5.3577 5.3390 5.3207 5.3013 4.9999 4.9875 4.9749 4.9623 4.9499 4.8506 4.5759 4.3590 4.3493 4.3384 4.3292 4.3171 4.3040 4.2904 4.2790 4.2682 4.2577 4.2463 4.0855 3.9046 3.8902 3.8849 3.8707 3.3394 3.3033 3.3005 2.7517 2.0286 2.0156 2.0039 1.3453 1.3310 1.3125 1.2820 1.2391 1.2290 1.2165 0.8925 0.8785
O
NH
O
N
F F
O
O
HN
P
O
OPh
HO OH
3c
500 MHz 1H NMR, CD3OD
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.0160 2.3356 2.0190 1.4422
1.0000
1.1103
1.3627
2.0104 1.4505 1.1413 1.3429
1.1797
1.8515
4.3154 2.3631 7.6972

Fig S1. ORTEP diagram of compound **2c** showing thermal ellipsoid at 50% probability

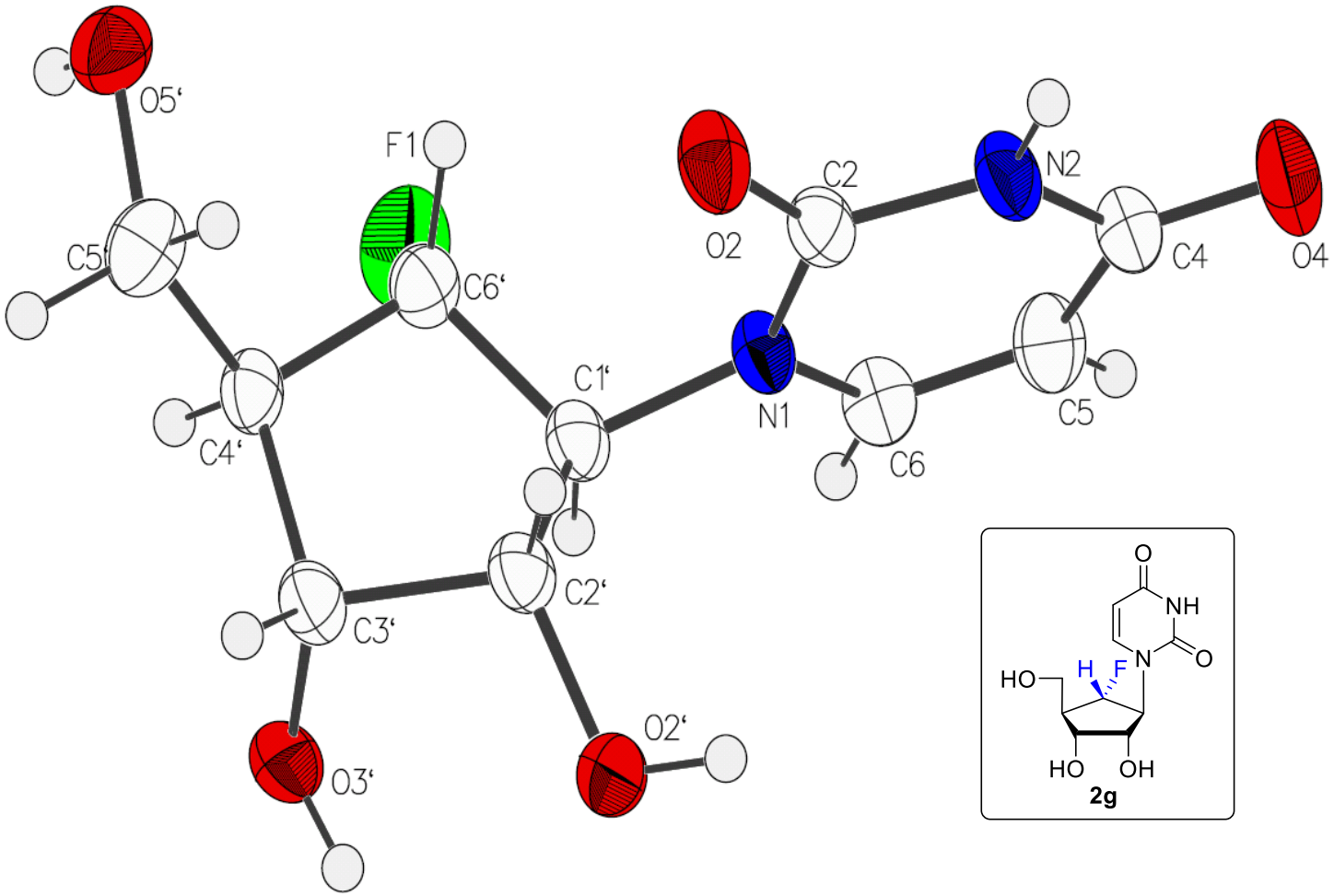


Single-Crystal X-ray crystallography data of compound 2c

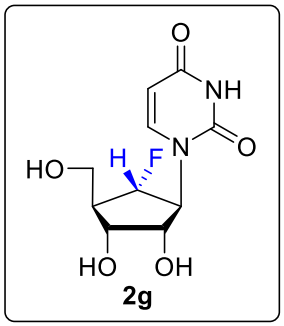


Crystal structure data for $C_{11}H_{13}F_2N_5O_{3,}1.5(H_2O)$ **(2c)** are as follows: M_r = 328.29, T = 99.9 (5) K, monoclinic, space group $P2_1$ (no. 4), a = 10.20373(8) Å, b = 6.97368(6) Å, c = 18.69692(15) Å, α = 90°, β = 96.8278(7)°, g = 90° V = 1320.992(19) Å^3, Z = 4, ρ_{calc} = 1.651 gcm^{-3}, μ = 1.280 mm^{-1}, F(000) = 684.0, crystal dimension 0.19 × 0.07 × 0.02 mm^3, radiation CuK_α (λ = 1.54184). Of 27411 reflections collected in the 2θ range from 4.76 to 153.162° using an ω scan on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer, 5504 were unique reflections (R_{int} = 0.0329, R_{sigma} = 0.0221). Using Olex2, the structure was solved with the ShelXT structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization. Final R indexes [all data] *R1* = 0.0241, *wR2* = 0.0659, GOF = 1.027, and maxmin^{-1} residual electron density 0.24/-0.20 eÅ^{-3}. Flack × parameter = 0.04(3). Further details of the crystal structure investigation(s) may be obtained from the Cambridge Crystallographic Data Centre (CCDC, 12 Union Road, Cambridge, CB2 1EZ (UK); Tel: (+44)1223-336-408, Fax: (+44)1223-336-033, e-mail: deposit@ccdc.cam.ac.kr) using no. **CCDC 1914672**.

Fig S2. ORTEP diagram of compound **2g** showing thermal ellipsoid at 50% probability

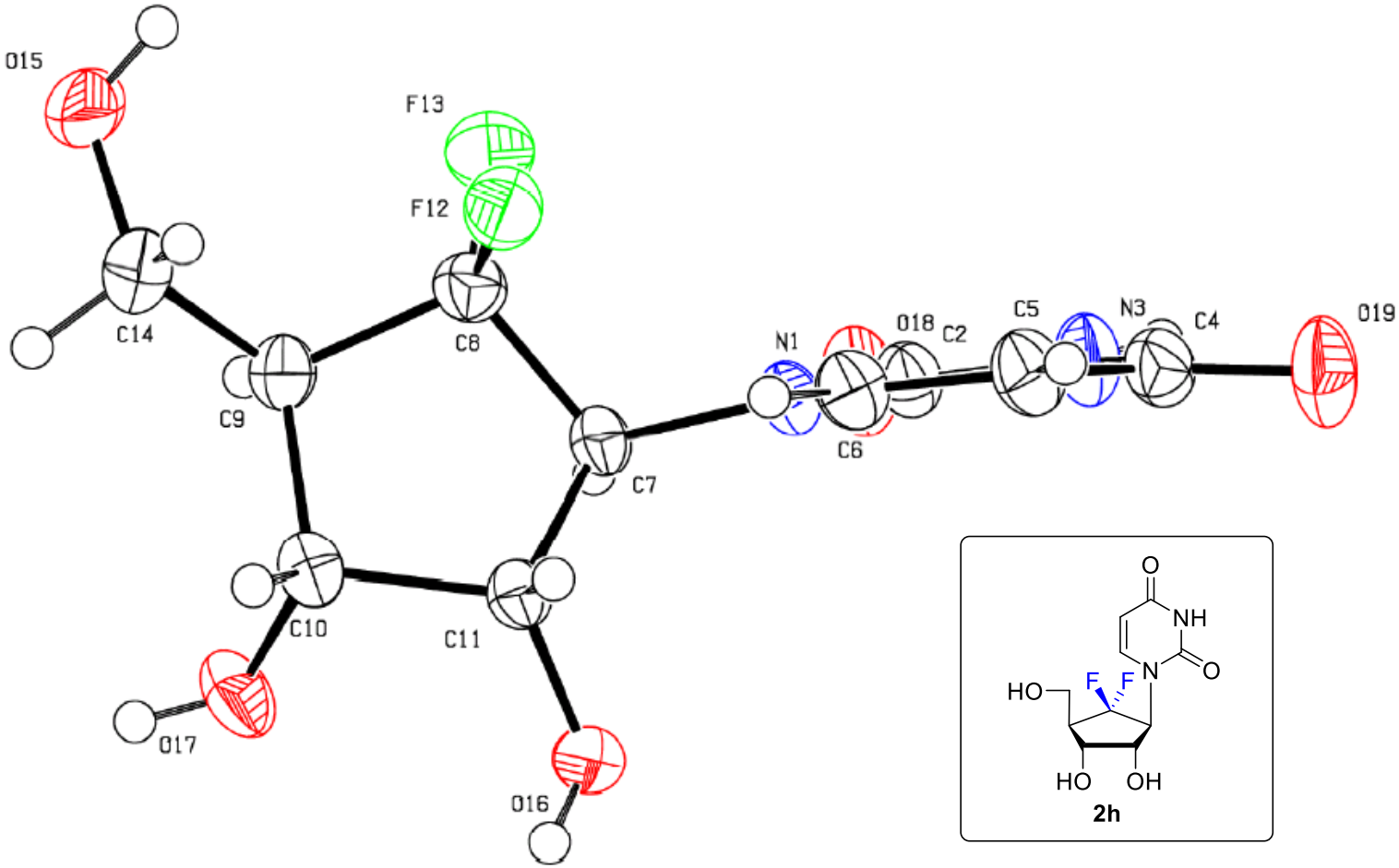


Single-Crystal X-ray crystallography data of compound 2g

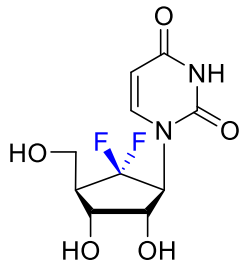


Crystal structure data for $C_{10}H_{13}FN_2O_5$ **(2g)** are as follows: M_r = 260.22, T = 295.71 (13) K, trigonal, space group $P3_221$, a = 6.6465(2) Å, b = 6.6465(2) Å, c = 43.3632(14) Å, α = 90°, β = 90°, γ = 120°, V = 1658.97(13) Å^3, Z = 6, ρ_{calc} = 1.563 gcm^{-3}, μ = 1.183 mm^{-1}, F(000) = 816.0, crystal dimension 0.242 × 0.067 × 0.034 mm^3, radiation CuK_α (λ = 1.54184). Of 29371 reflections collected in the 2θ range from 12.246 to 154.882° using an ω scan on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer, 2346 were unique reflections (R_{int} = 0.0654, R_{sigma} = 0.0230). Using Olex2, the structure was solved with the ShelXT structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization. Final R indexes [all data] *R1* = 0.0273, *wR2* = 0.0779, GOF = 1.062, and maxmin^{-1} residual electron density 0.14/-0.16 eÅ^{-3}. Flack × parameter = 0.13(10). Further details of the crystal structure investigation(s) may be obtained from the Cambridge Crystallographic Data Centre (CCDC, 12 Union Road, Cambridge, CB2 1EZ (UK); Tel: (+44)1223-336-408, Fax: (+44)1223-336-033, e-mail: deposit@ccdc.cam.ac.kr) using no. **CCDC 1871331**

Fig S3. ORTEP diagram of compound **2h** showing thermal ellipsoid at 50% probability



Single-Crystal X-ray crystallography data of compound 2h



2h

Crystal structure data for $C_{10}H_{13}F_2N_2O_5$ **(2h)** are as follows: M_r = 278.21, T = 293.55 (10) K, orthorhombic, space group $P2_12_12_1$, a = 5.8735(2) Å, b = 13.5166(2) Å, c = 14.1374(14) Å, α = 90°, β=90°, γ = 90°, V=1122.36 (7) Å^3, Z = 4, ρ_{calc}= 1.6463 gcm^{-3}, μ = 1.343 mm^{-1}, F(000)=578.6, crystal dimension 0.261 x 0.21 x 0.074 mm^3, radiation CuK_α (λ=1.54184). Of 3965 reflections collected in the 2θ range from 9.06 to 147.32° using an ω scan on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer, 2190 were unique reflections (R_{int} = 0.0191, R_{sigma} = 0.0258). Using Olex2, the structure was solved with the ShelXT structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization. Final R indexes [all data] *R1*=0.0318, *wR2*= 0.0813, GOF=1.065, and maxmin^{-1} residual electron density 0.17/-0.19 eÅ^{-3}. Flack *x* parameter= 0.02(13). Further details of the crystal structure investigation(s) may be obtained from the Cambridge Crystallographic Data Centre (CCDC, 12 Union Road, Cambridge, CB2 1EZ (UK); Tel: (+44)1223-336-408, Fax: (+44)1223-336-033, e-mail: deposit@ccdc.cam.ac.kr) using no. **CCDC 1871332**.