Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Stereo- and Regio-selective Synthesis of 3'-*C*-Substituted-(*N*)-methanocarbaadenosines as Potential Anticancer Agents

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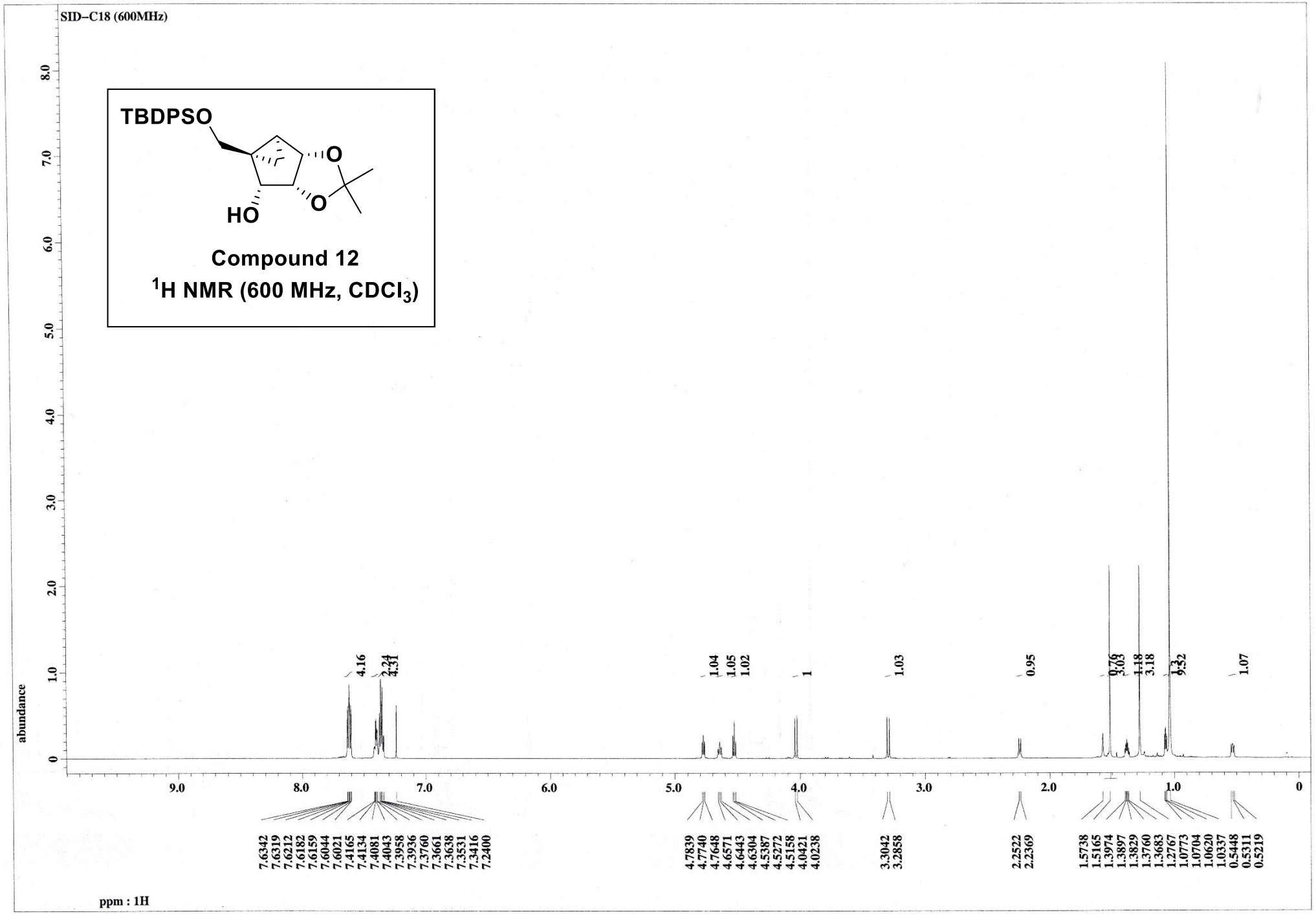
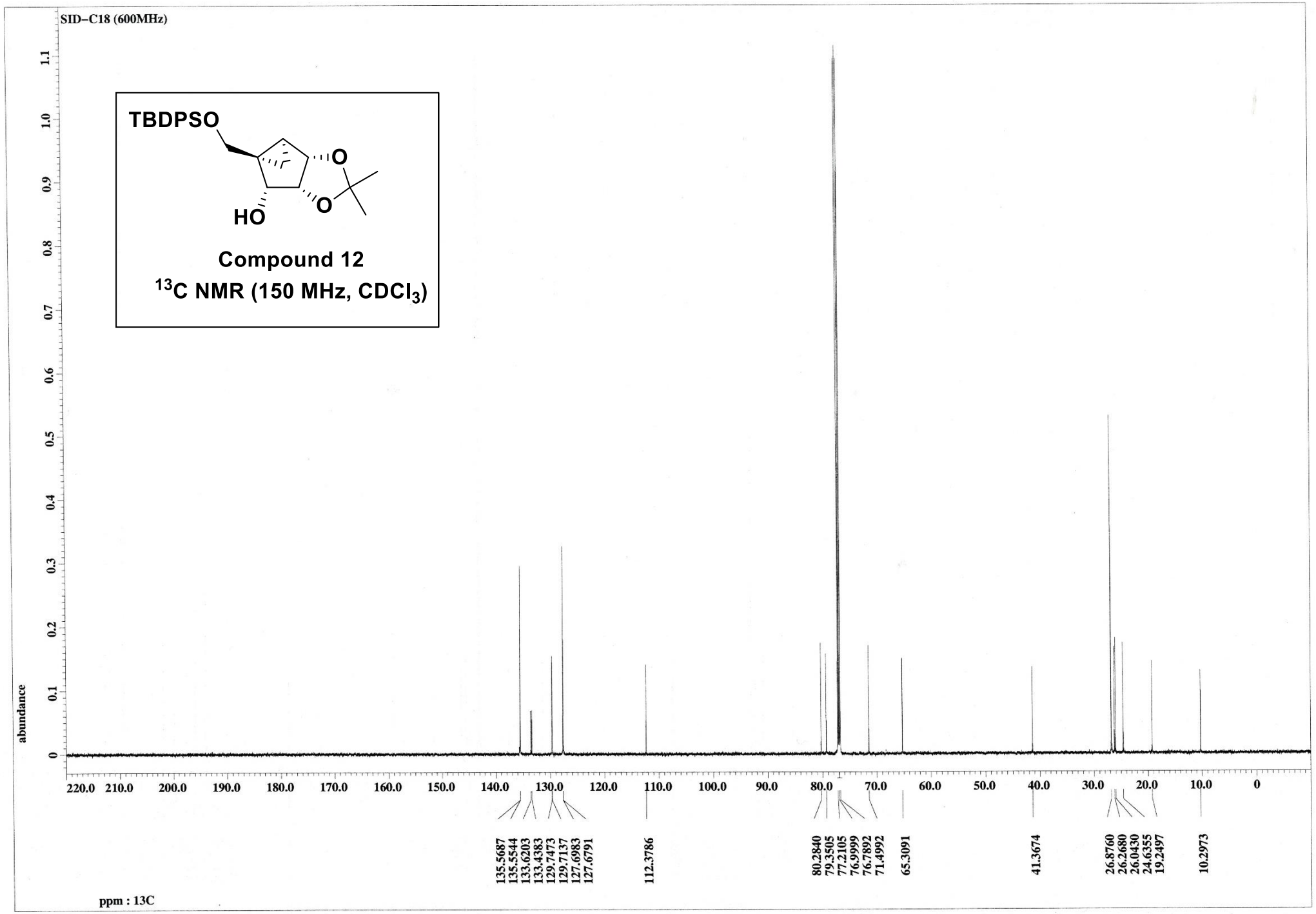
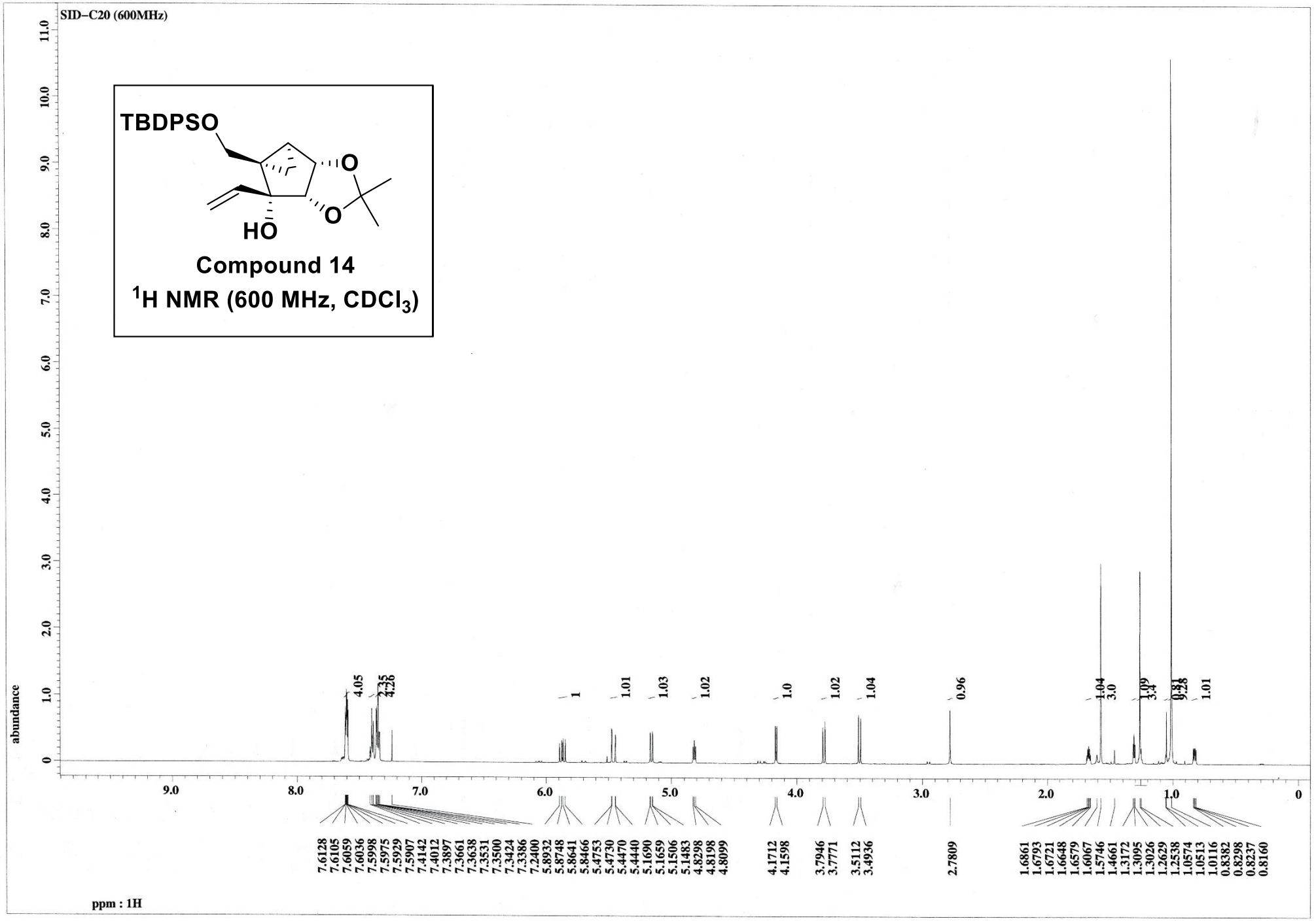
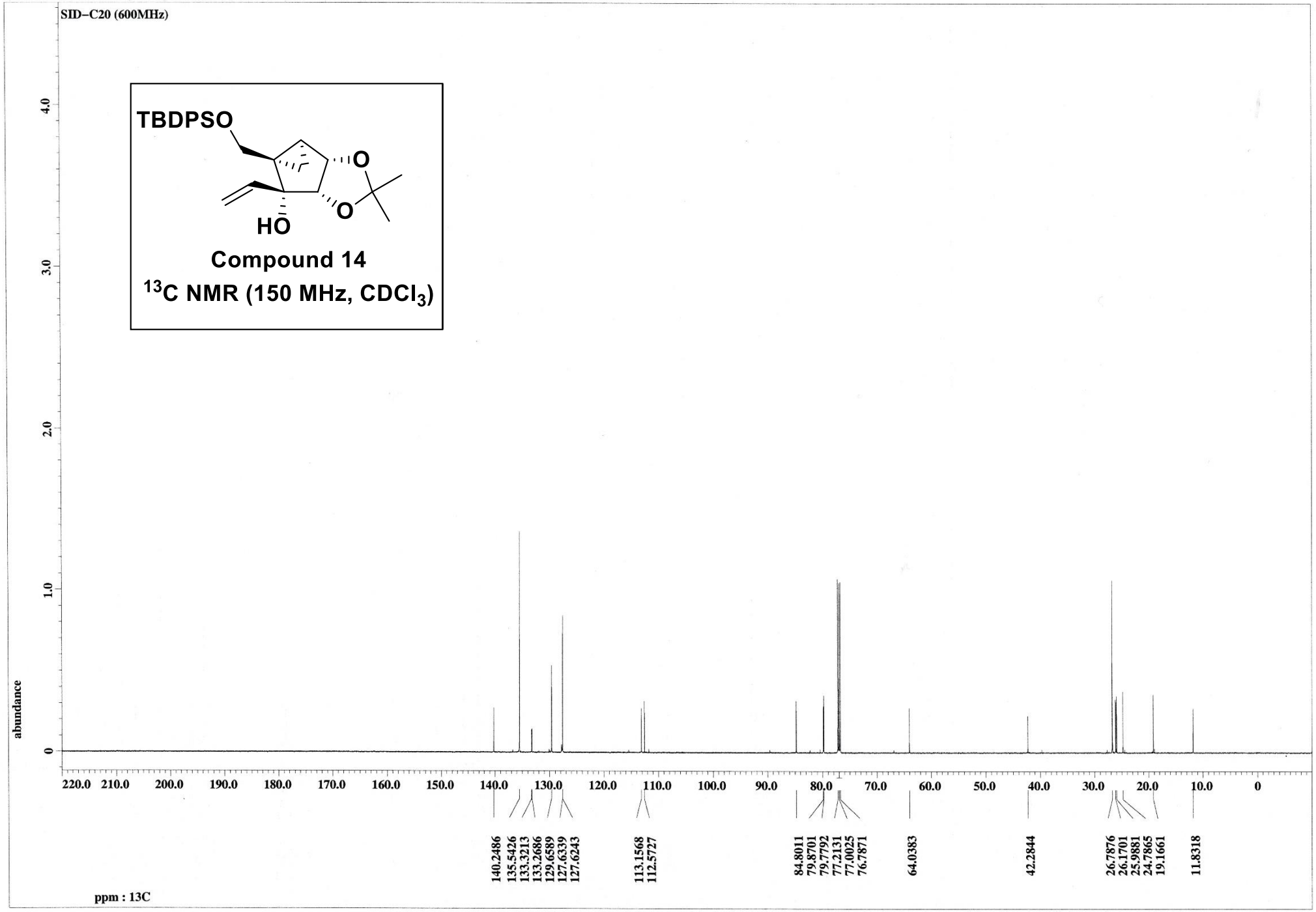
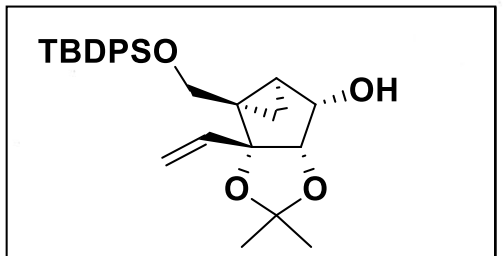

SID−C18 (600MHz)
TBDPSO
O
O
HO
Compound 12
1H NMR (600 MHz, CDCl3)
abundance
ppm : 1H
4.16
2.24
4.31
1.04
1.05
1.02
1
1.03
0.95
0.76
3.03
1.18
3.18
1.3
9.52
1.07
7.6342
7.6319
7.6212
7.6182
7.6159
7.6044
7.6021
7.4165
7.4134
7.4081
7.4043
7.3958
7.3936
7.3760
7.3661
7.3638
7.3531
7.3416
7.2400
4.7839
4.7740
4.7648
4.6571
4.6443
4.6304
4.5387
4.5272
4.5158
4.0421
4.0238
3.3042
3.2858
2.2522
2.2369
1.5738
1.5165
1.3974
1.3897
1.3829
1.3760
1.3683
1.2767
1.0773
1.0704
1.0620
1.0337
0.5448
0.5311
0.5219


SID–C18 (600MHz)
TBDPSO
HO
O
O
Compound 12
13C NMR (150 MHz, CDCl3)
abundance
ppm : 13C
135.5687
135.5544
133.6203
133.4383
129.7473
129.7137
127.6983
127.6791
112.3786
80.2840
79.3505
77.2105
76.9999
76.7892
71.4992
65.3091
41.3674
26.8760
26.2680
26.0430
24.6355
19.2497
10.2973


SID–C20 (600MHz)
TBDPSO
O
O
HO
Compound 14
^{1}H NMR (600 MHz, $CDCl_3$)
abundance
ppm : 1H
4.05
2.35
4.26
1
1.01
1.03
1.02
1.0
1.02
1.04
0.96
1.04
3.0
1.09
3.4
0.81
0.88
1.01
7.6128
7.6105
7.6059
7.6036
7.5998
7.5975
7.5929
7.5907
7.4142
7.4012
7.3897
7.3661
7.3638
7.3531
7.3500
7.3424
7.3386
7.2400
5.8932
5.8748
5.8641
5.8466
5.4753
5.4730
5.4470
5.4440
5.1690
5.1659
5.1506
5.1483
4.8298
4.8198
4.8099
4.1712
4.1598
3.7946
3.7771
3.5112
3.4936
2.7809
1.6861
1.6793
1.6721
1.6648
1.6579
1.6067
1.5746
1.4661
1.3172
1.3095
1.3026
1.2629
1.2538
1.0574
1.0513
1.0116
0.8382
0.8298
0.8237
0.8160


SID–C20 (600MHz)
TBDPSO
O
O
HO
Compound 14
13C NMR (150 MHz, CDCl3)
abundance
4.0
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
140.2486
135.5426
133.3213
133.2686
129.6589
127.6339
127.6243
113.1568
112.5727
84.8011
79.8701
79.7792
77.2131
77.0025
76.7871
64.0383
42.2844
26.7876
26.1701
25.9881
24.7865
19.1661
11.8318
ppm : 13C

SID–C21 (600MHz)

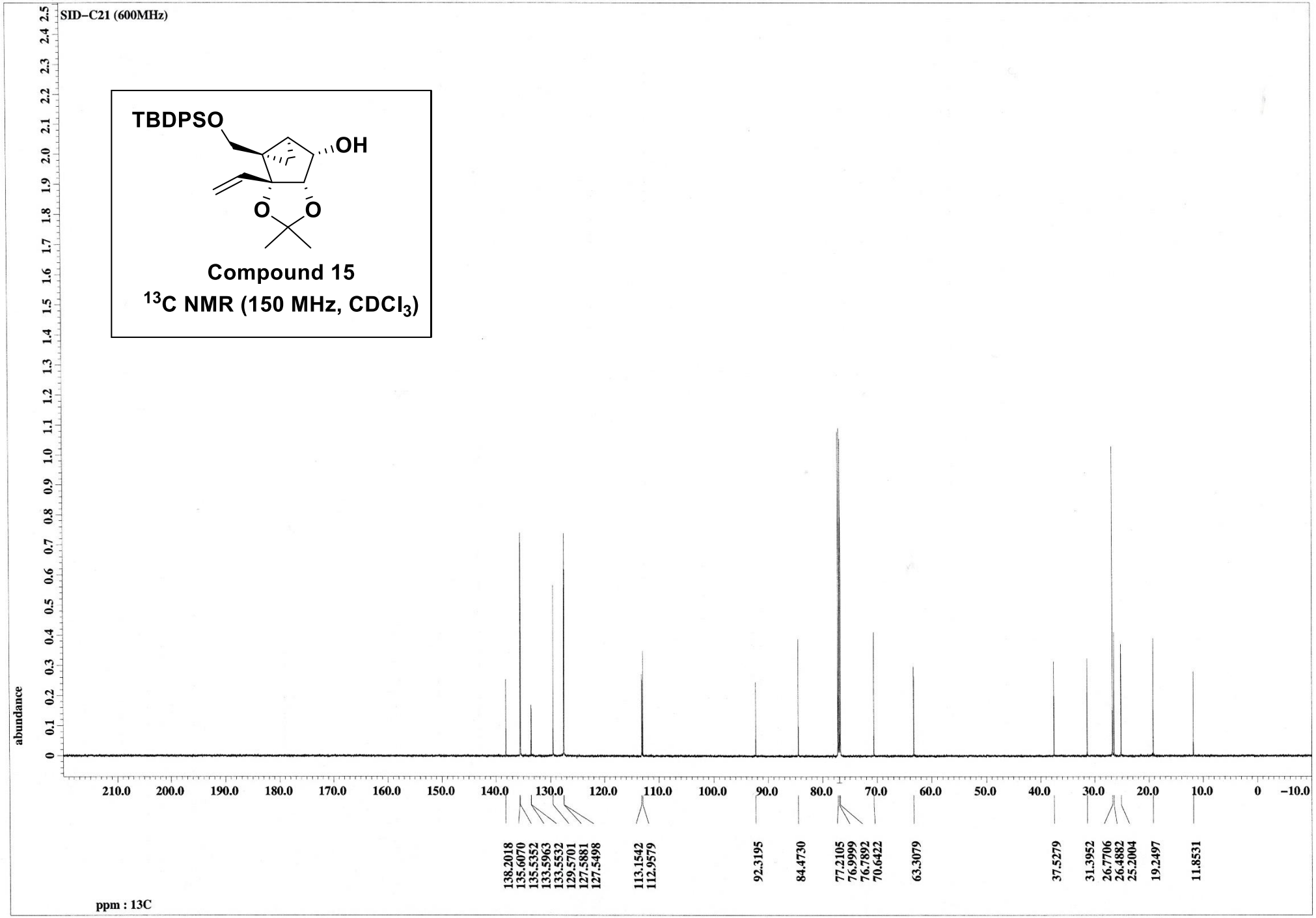


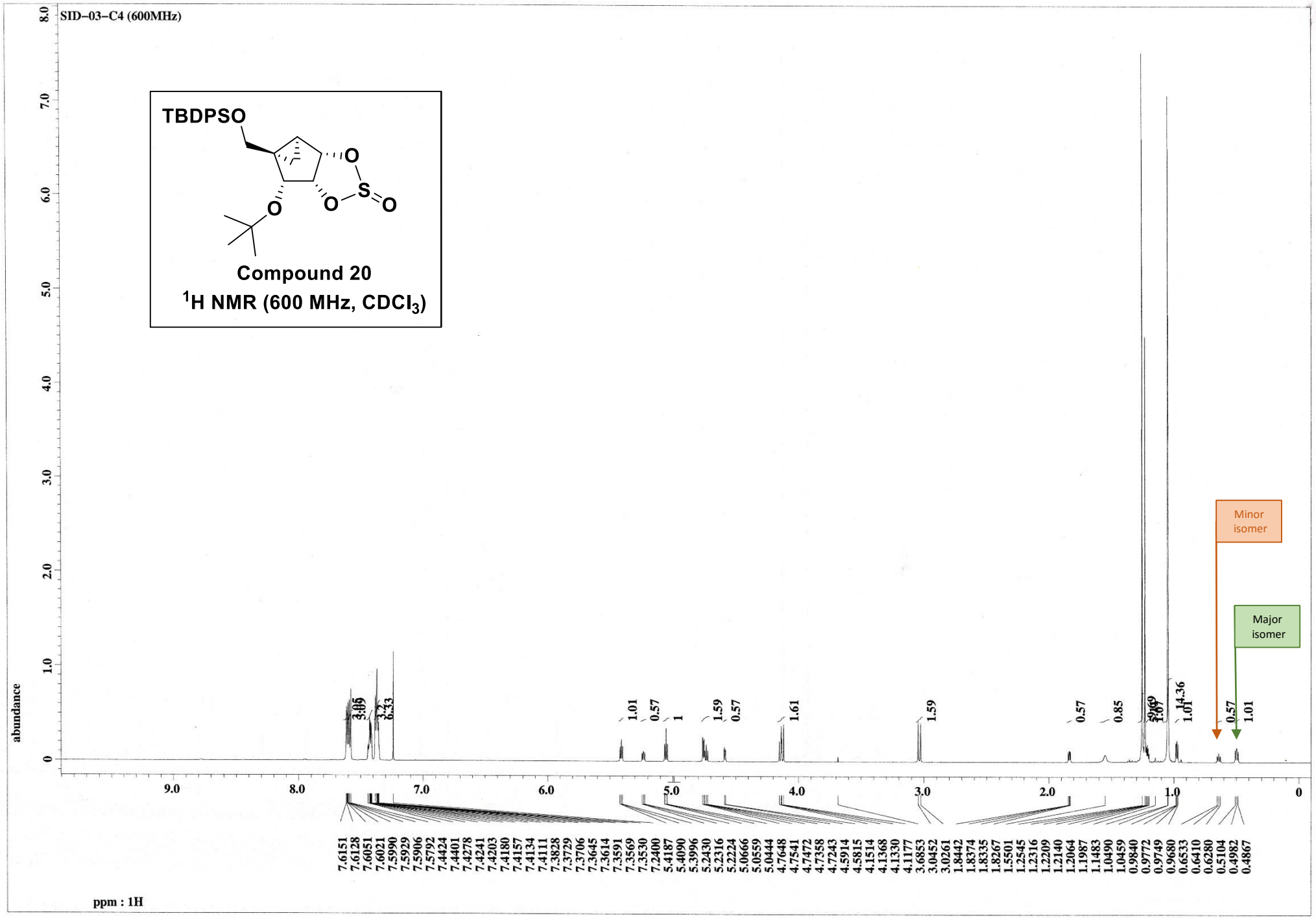
Compound 15

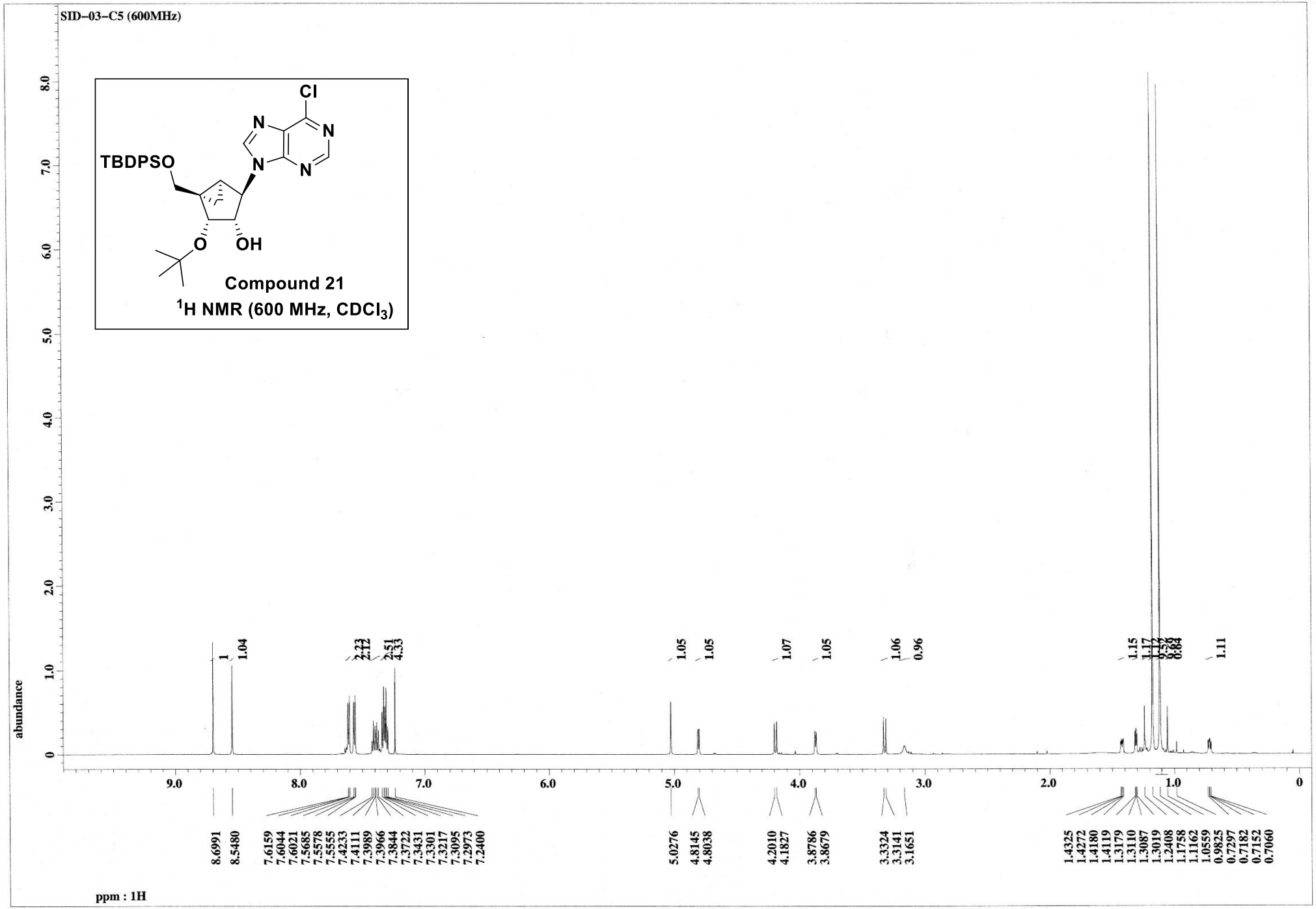
^{1}H NMR (600 MHz, $CDCl_3$)

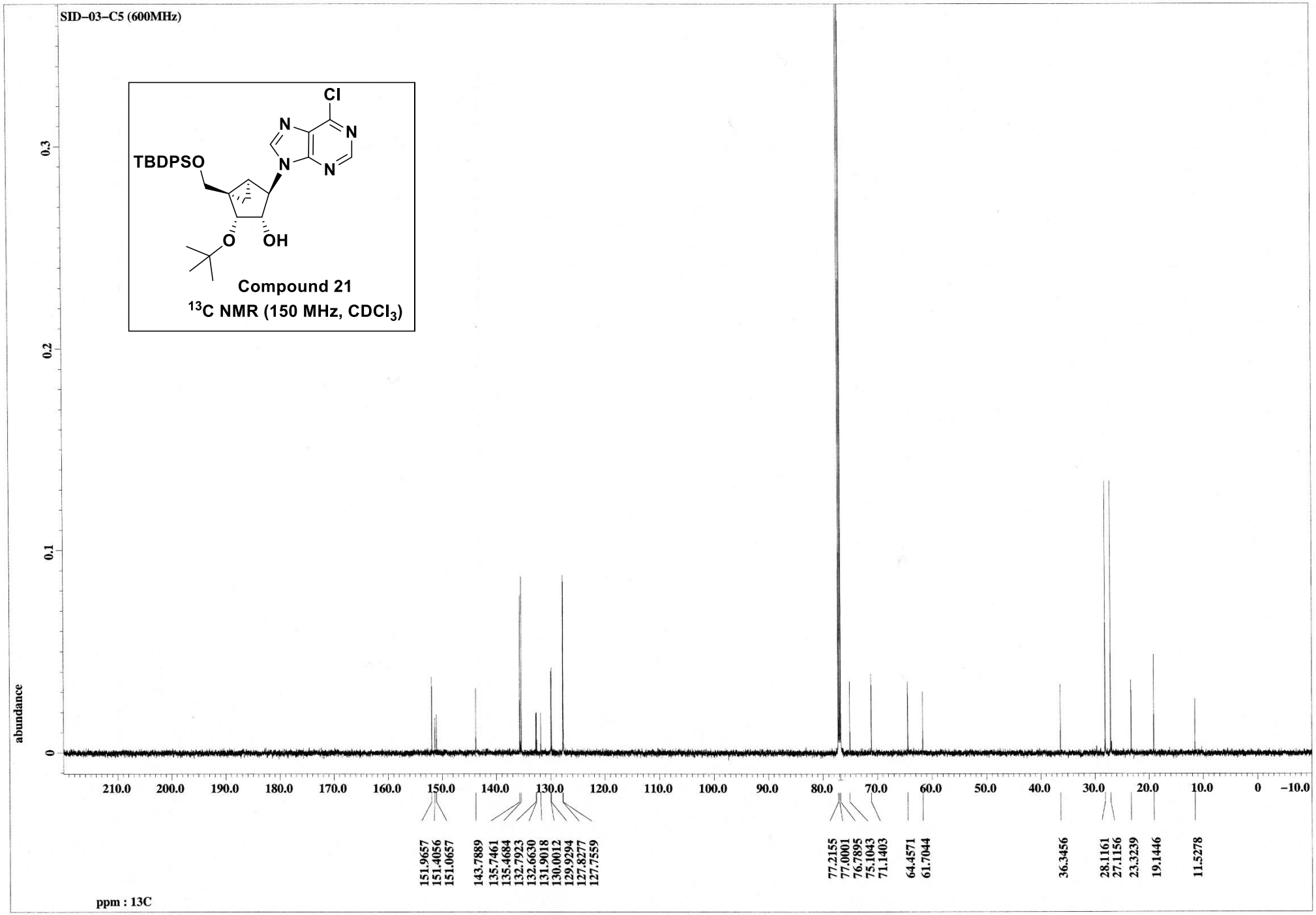
abundance

ppm : 1H

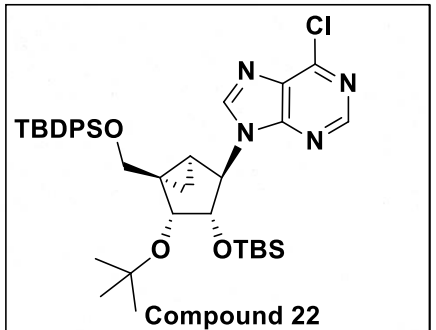

SID–C21 (600MHz)
TBDPSO
OH
O
O
Compound 15
^{13}C NMR (150 MHz, CDCl$_3$)
abundance
ppm : 13C
138.2018
135.6070
135.5352
133.5963
133.5532
129.5701
127.5881
127.5498
113.1542
112.9579
92.3195
84.4730
77.2105
76.9999
76.7892
70.6422
63.3079
37.5279
31.3952
26.7706
26.4882
25.2004
19.2497
11.8531


SID–03–C4 (600MHz)
TBDPSO
O
S
O
O
O
Compound 20
^{1}H NMR (600 MHz, $CDCl_3$)
Minor isomer
Major isomer
abundance
ppm : 1H


SID–03–C5 (600MHz)
Cl
N
N
N
N
TBDPSO
O
OH
Compound 21
^{1}H NMR (600 MHz, $CDCl_3$)
abundance
ppm : 1H


SID–03–C5 (600MHz)
Cl
N
N
N
N
TBDPSO
O
OH
Compound 21
^{13}C NMR (150 MHz, $CDCl_3$)
abundance
0
0.1
0.2
0.3
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
151.9657
151.4056
151.0657
143.7889
135.7461
135.4684
132.7923
132.6630
131.9018
130.0012
129.9294
127.8277
127.7559
77.2155
77.0001
76.7895
75.1043
71.1403
64.4571
61.7044
36.3456
28.1161
27.1156
23.3239
19.1446
11.5278
ppm : 13C

SID-03-C6 (600MHz)

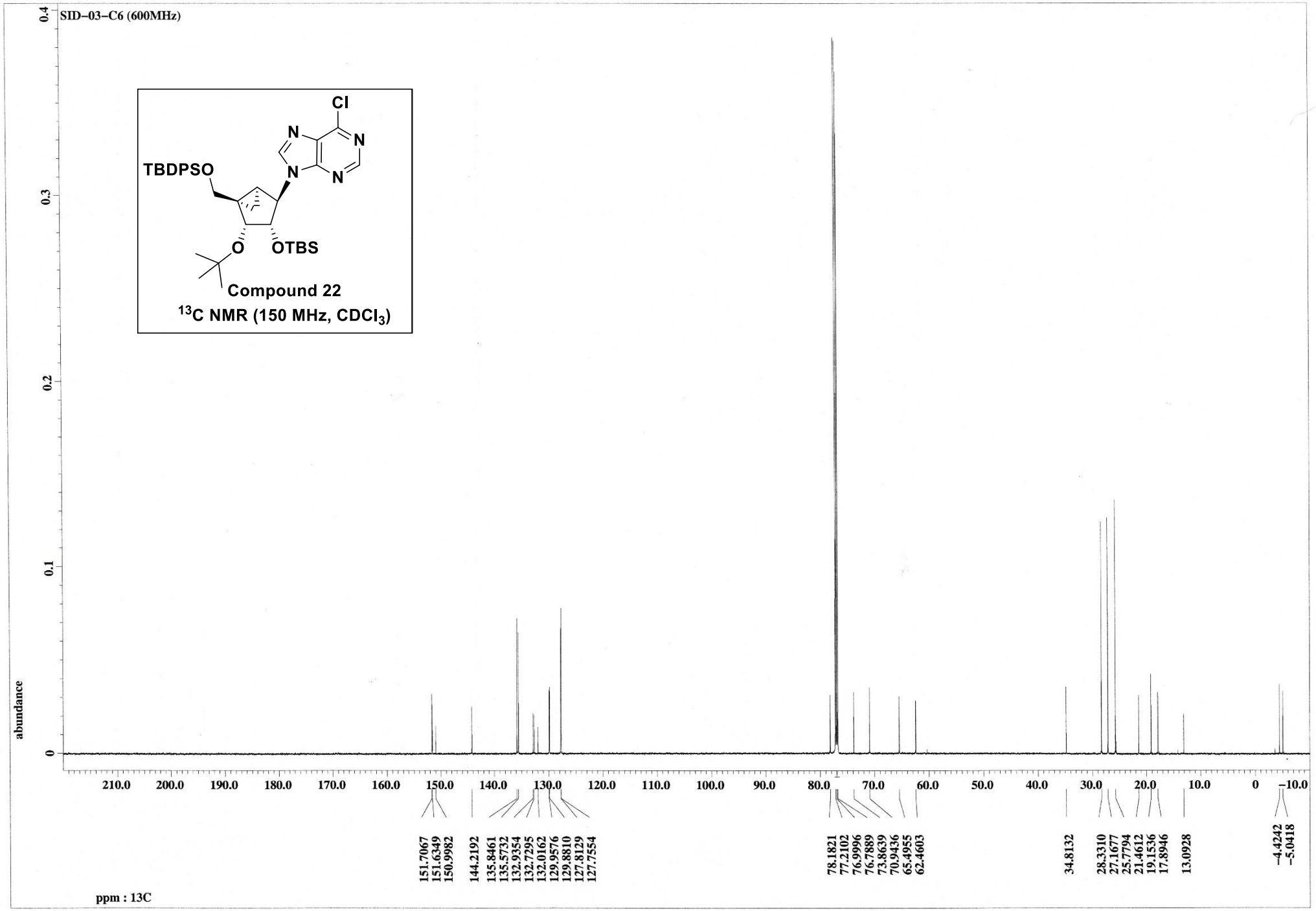


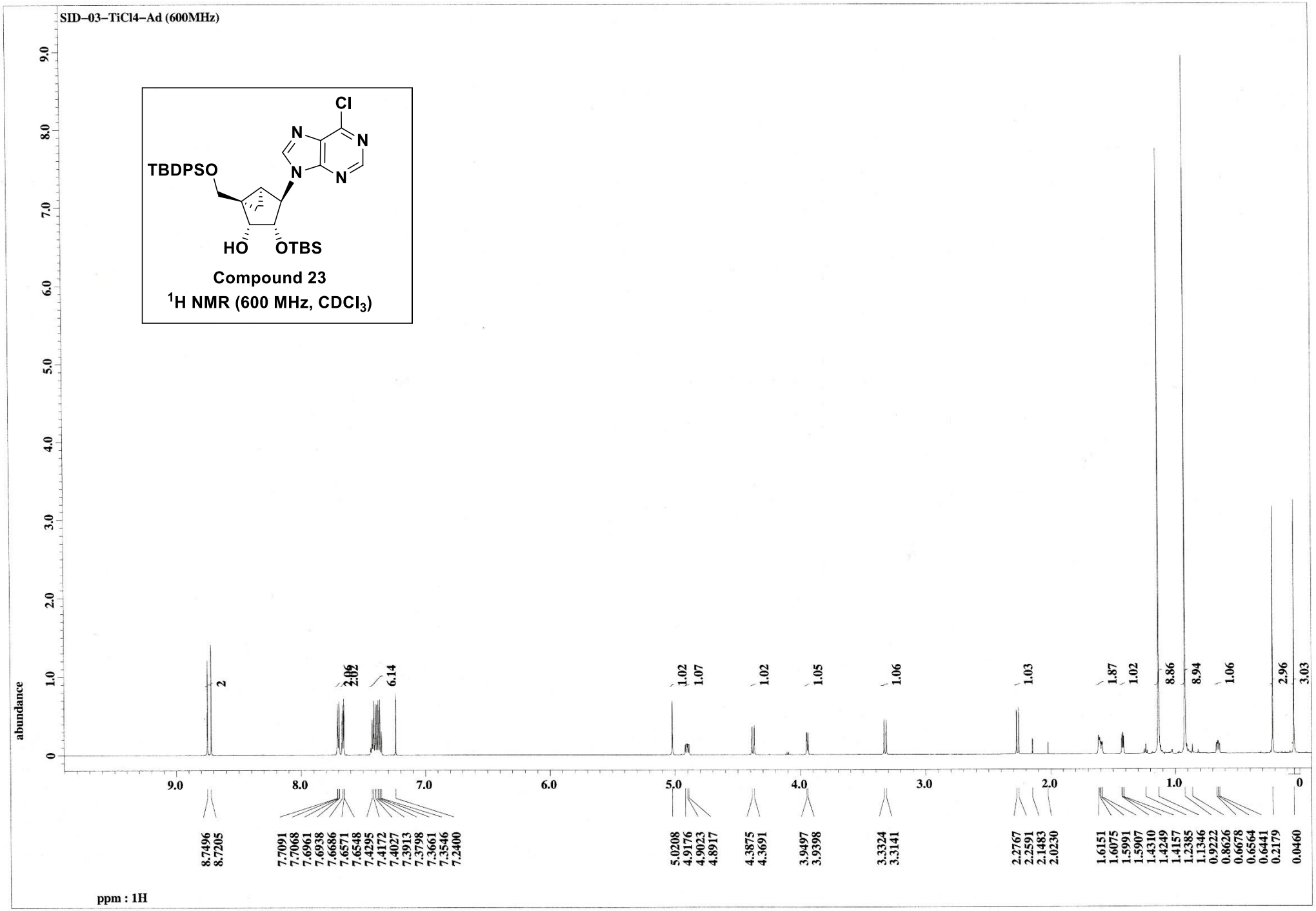
Compound 22

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

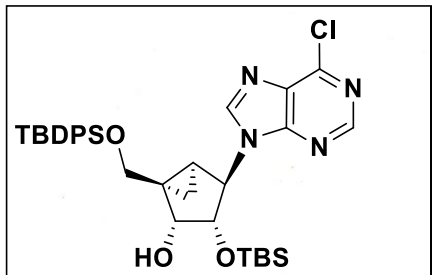
abundance

ppm : 1H


SID−03−C6 (600MHz)
Cl
N
N
N
N
TBDPSO
O
OTBS
Compound 22
^{13}C NMR (150 MHz, $CDCl_3$)
abundance
0.4
0.3
0.2
0.1
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 −10.0
151.7067
151.6349
150.9982
144.2192
135.8461
135.5732
132.9354
132.7295
132.0162
129.9576
129.8810
127.8129
127.7554
78.1821
77.2102
76.9996
76.7889
73.8639
70.9436
65.4955
62.4603
34.8132
28.3310
27.1677
25.7794
21.4612
19.1536
17.8946
13.0928
−4.4242
−5.0418
ppm : 13C


SID–03–TiCl4–Ad (600MHz)
Cl
N
N
N
N
TBDPSO
HO
OTBS
Compound 23
1H NMR (600 MHz, CDCl3)
abundance
ppm : 1H
8.7496
8.7205
7.7091
7.7068
7.6961
7.6938
7.6686
7.6571
7.6548
7.4295
7.4172
7.4027
7.3913
7.3798
7.3661
7.3546
7.2400
5.0208
4.9176
4.9023
4.8917
4.3875
4.3691
3.9497
3.9398
3.3324
3.3141
2.2767
2.2591
2.1483
2.0230
1.6151
1.6075
1.5991
1.5907
1.4310
1.4249
1.4157
1.2385
1.1346
0.9222
0.8626
0.6678
0.6564
0.6441
0.2179
0.0460

SID–03–TiCl4–Ad (600MHz)



Compound 23

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

abundance

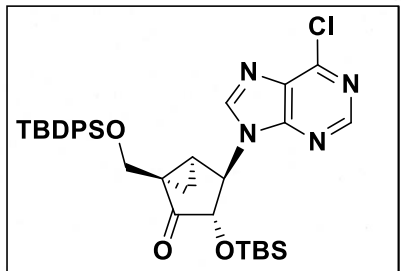
0 0.1 0.2 0.3 0.4 0.5

220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0

151.8076
151.3480
151.1517
143.6690
135.6980
135.5065
133.0171
132.5623
131.8968
129.9196
129.8766
127.8563
77.2872
77.2106
76.9999
76.7845
70.8003
64.9644
61.6037
36.7380
26.9909
25.7318
24.0036
19.2497
17.9380
10.5510
−4.6010
−5.3574

ppm : 13C

SID–03–C8 (600MHz)

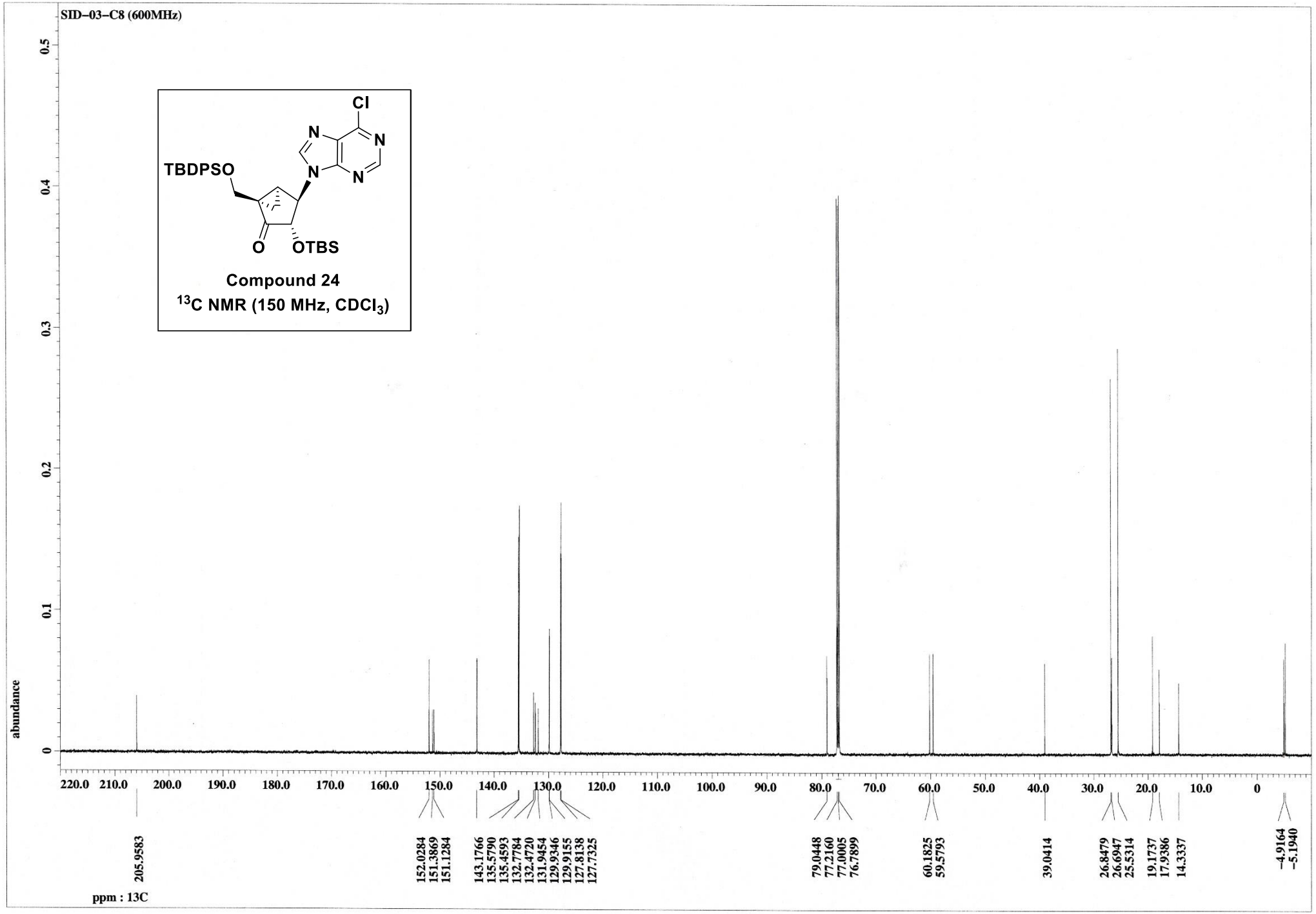


Compound 24

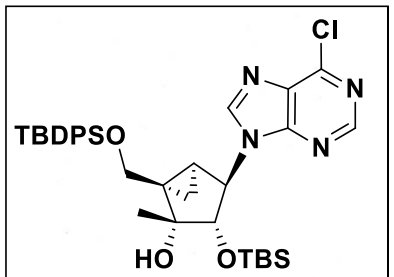
^{1}H NMR (600 MHz, $CDCl_3$)

abundance

ppm : 1H


SID--03--C8 (600MHz)
Cl
N
N
N
N
TBDPSO
O
OTBS
Compound 24
13C NMR (150 MHz, CDCl3)
abundance
0.5
0.4
0.3
0.2
0.1
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
205.9583
152.0284
151.3869
151.1284
143.1766
135.5790
135.4593
132.7784
132.4720
131.9454
129.9346
129.9155
127.8138
127.7325
79.0448
77.2160
77.0005
76.7899
60.1825
59.5793
39.0414
26.8479
26.6947
25.5314
19.1737
17.9386
14.3337
-4.9164
-5.1940
ppm : 13C

SID–03–Me–UPS (600MHz)



Compound 25a

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

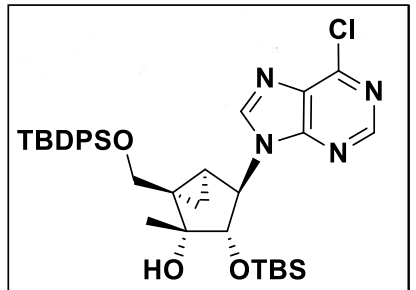
abundance

1 1.04 2.18 2.2 6.68 1.05 1.07 1.06 1.08 1.02 1.07 9.72

8.6928 8.4285 7.6852 7.6737 7.6493 7.6386 7.4567 7.4445 7.4323 7.4071 7.3941 7.3803 7.3681 7.2398 4.7868 4.2535 4.2344 3.7646 3.6271 3.6088 2.6821 1.7302 1.7157 1.3513 1.3437 1.3360 1.2879 1.2367 1.1114 0.9067 0.8853 0.8654 0.8540 0.8418 0.1259 0.0526

parts per Million : 1H

SID–03–Me–UPS (600MHz)



Compound 25a

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

abundance

parts per Million : 13C

151.6786
151.5685
151.1328
143.6166
135.6408
135.4781
133.0030
132.7923
132.0742
130.0252
129.9008
127.8709
127.8470
84.3440
77.2108
77.0768
77.0002
76.7895
63.9115
62.7242
38.8782
29.6338
29.5955
29.2939
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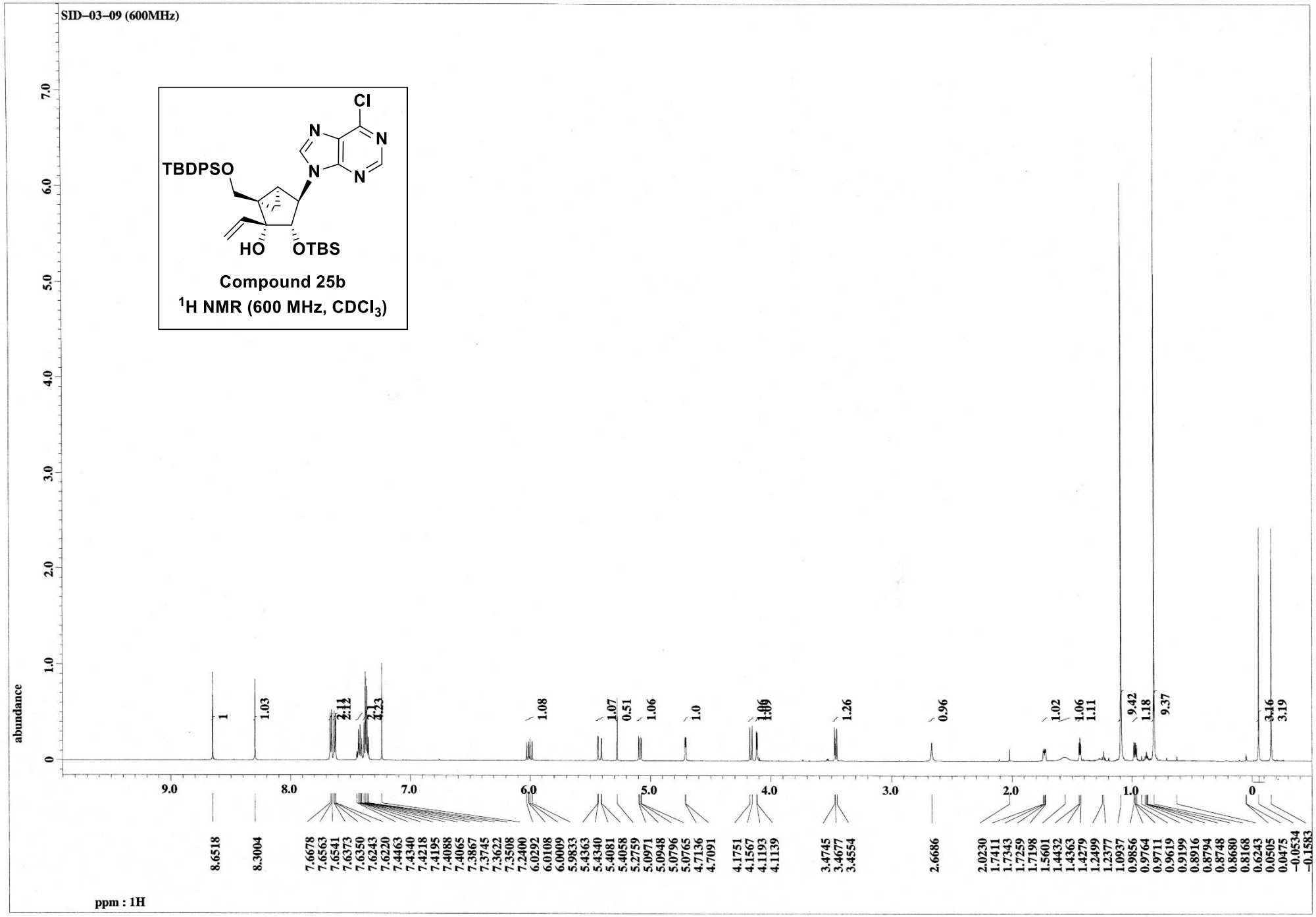
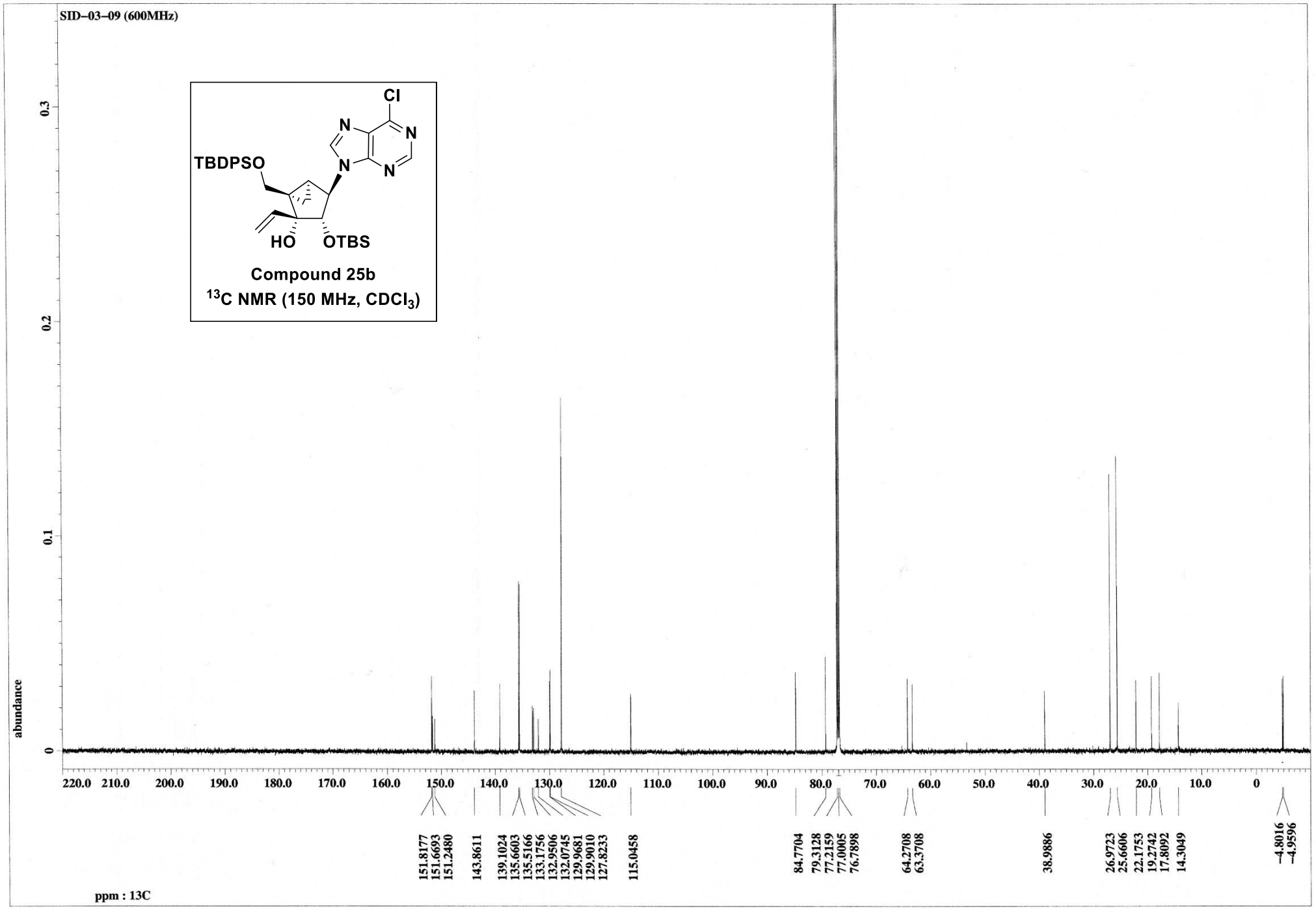
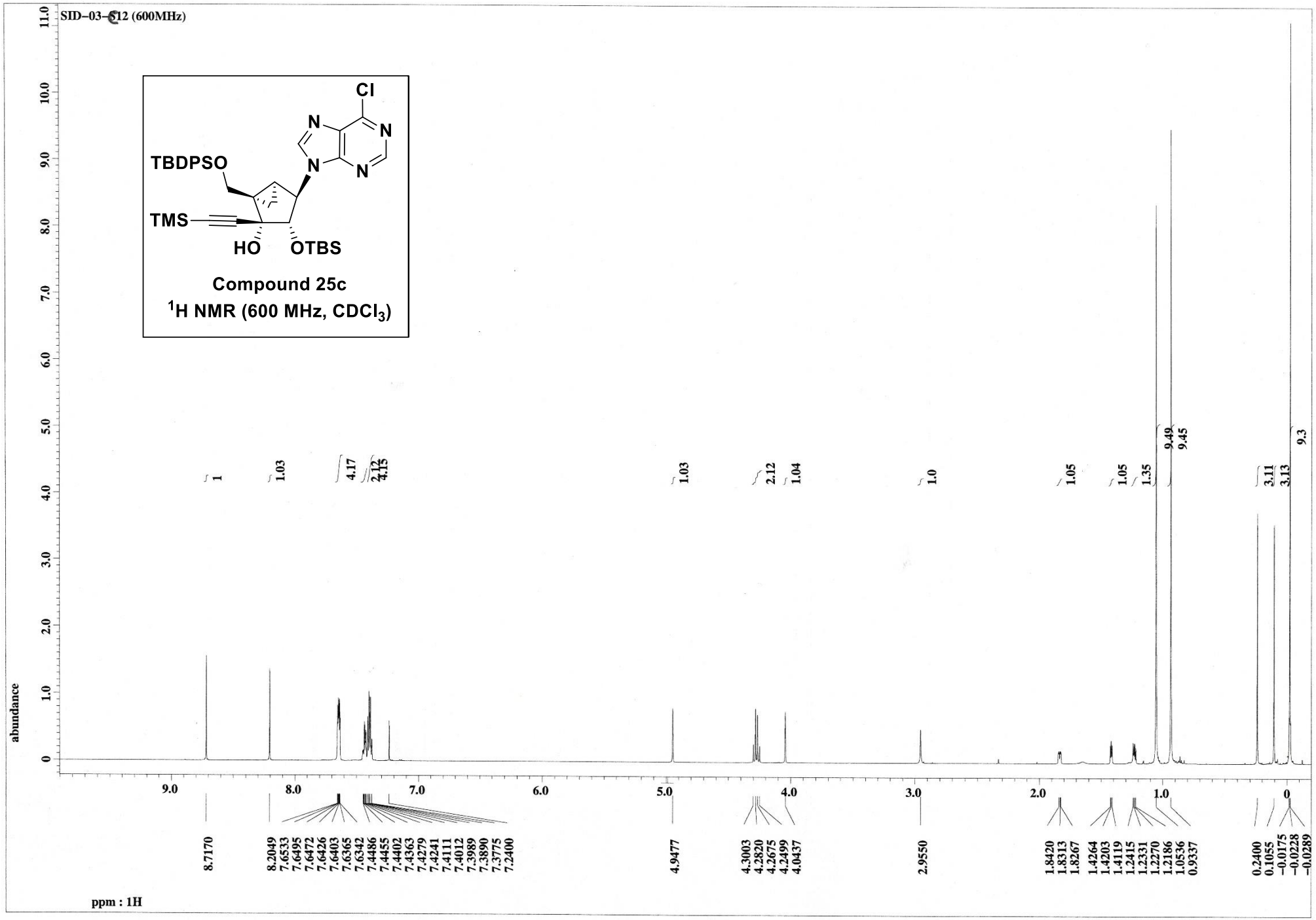
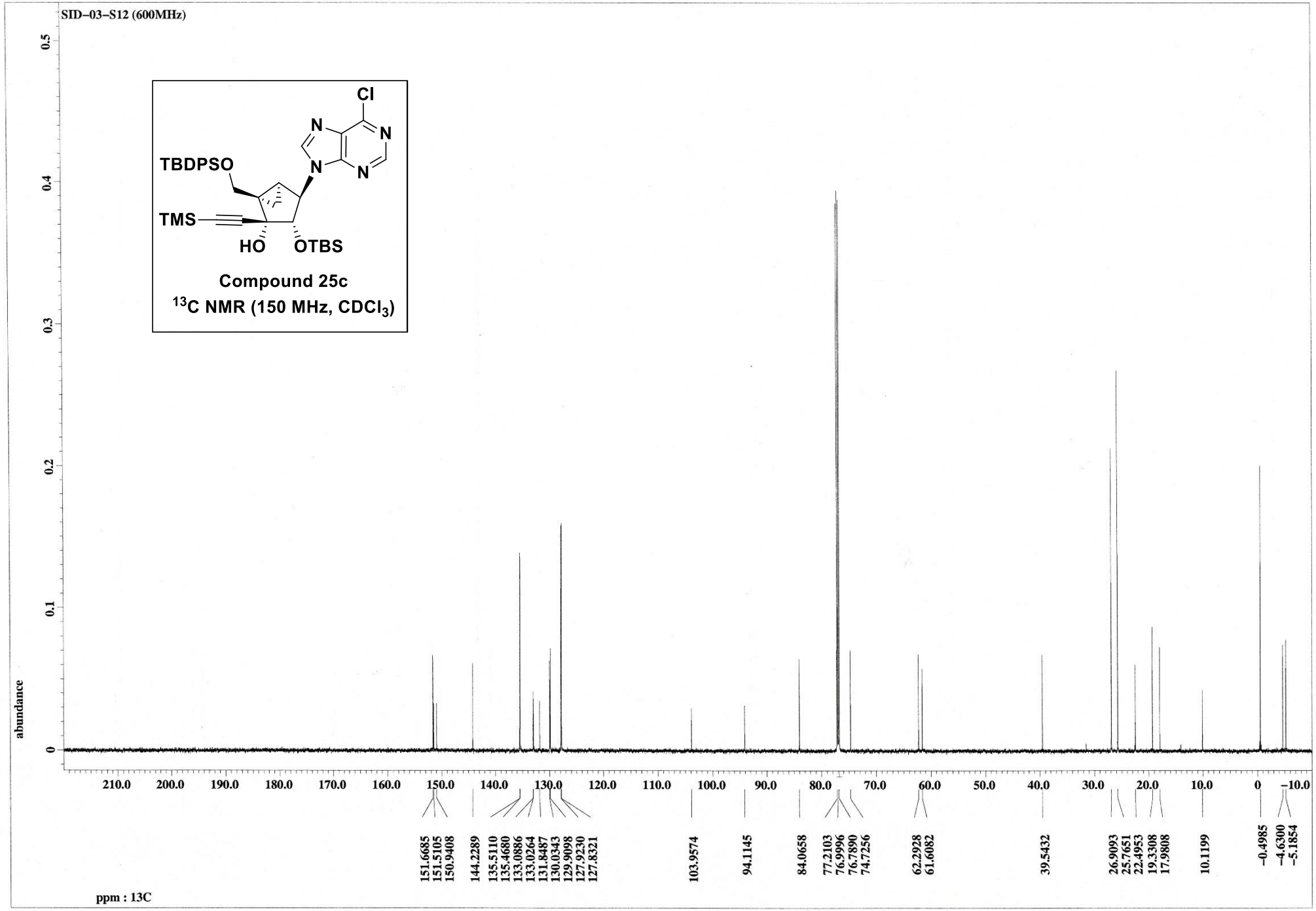
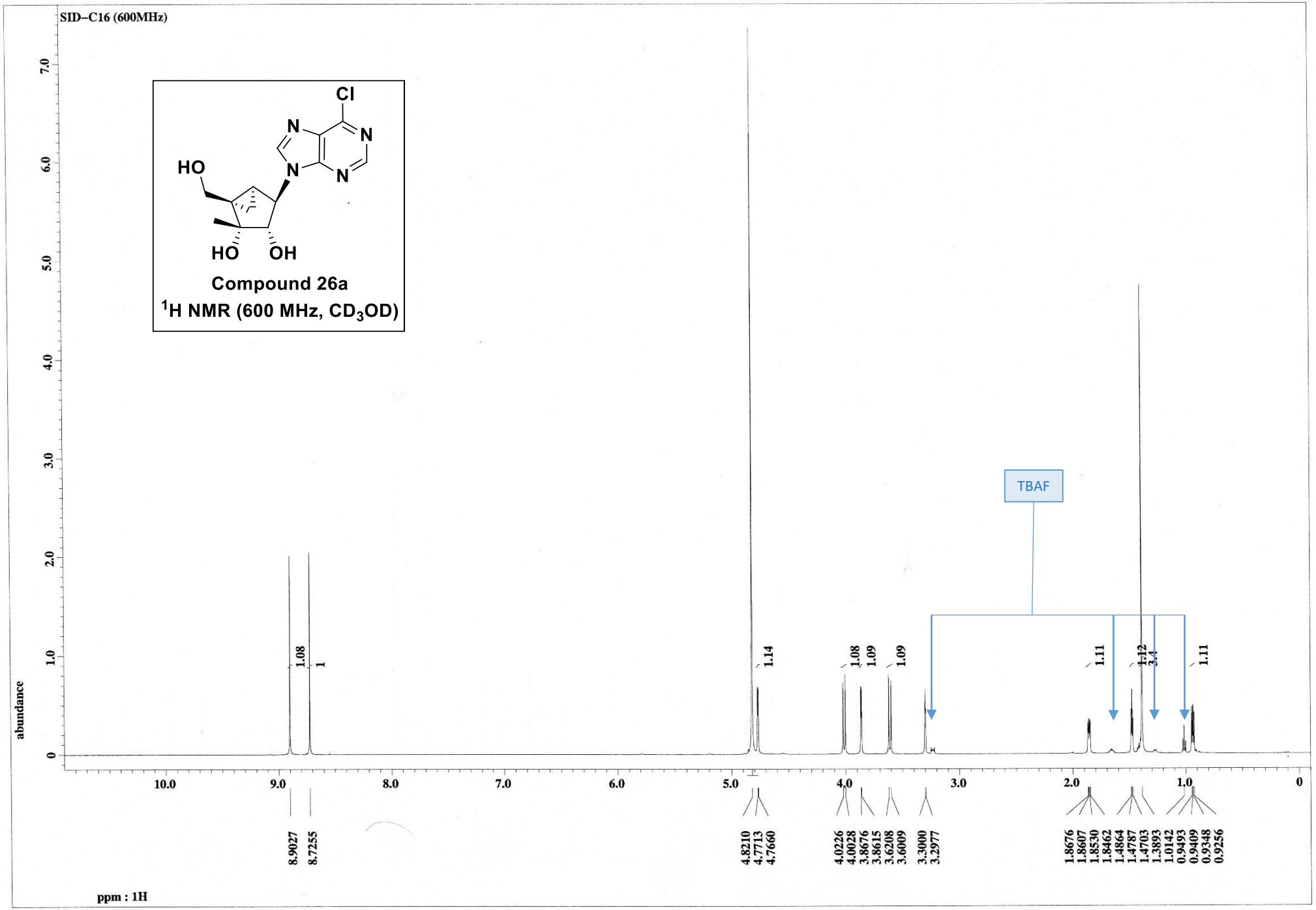
19.1590
17.8999
13.0551
−4.6439
−5.2231

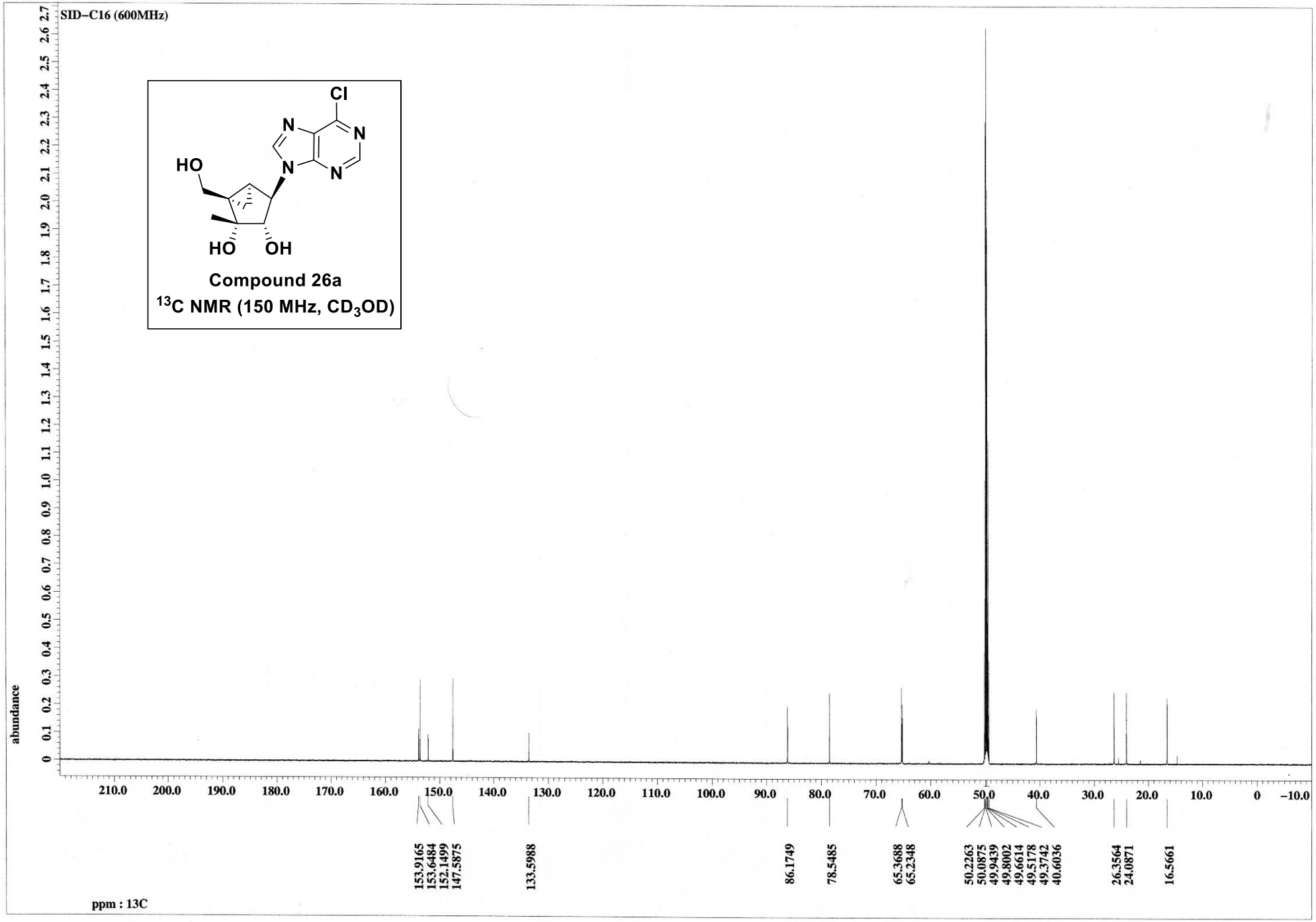

SID–03–09 (600MHz)
Cl
N
N
N
N
TBDPSO
HO
OTBS
Compound 25b
^{1}H NMR (600 MHz, $CDCl_3$)
abundance
ppm : 1H

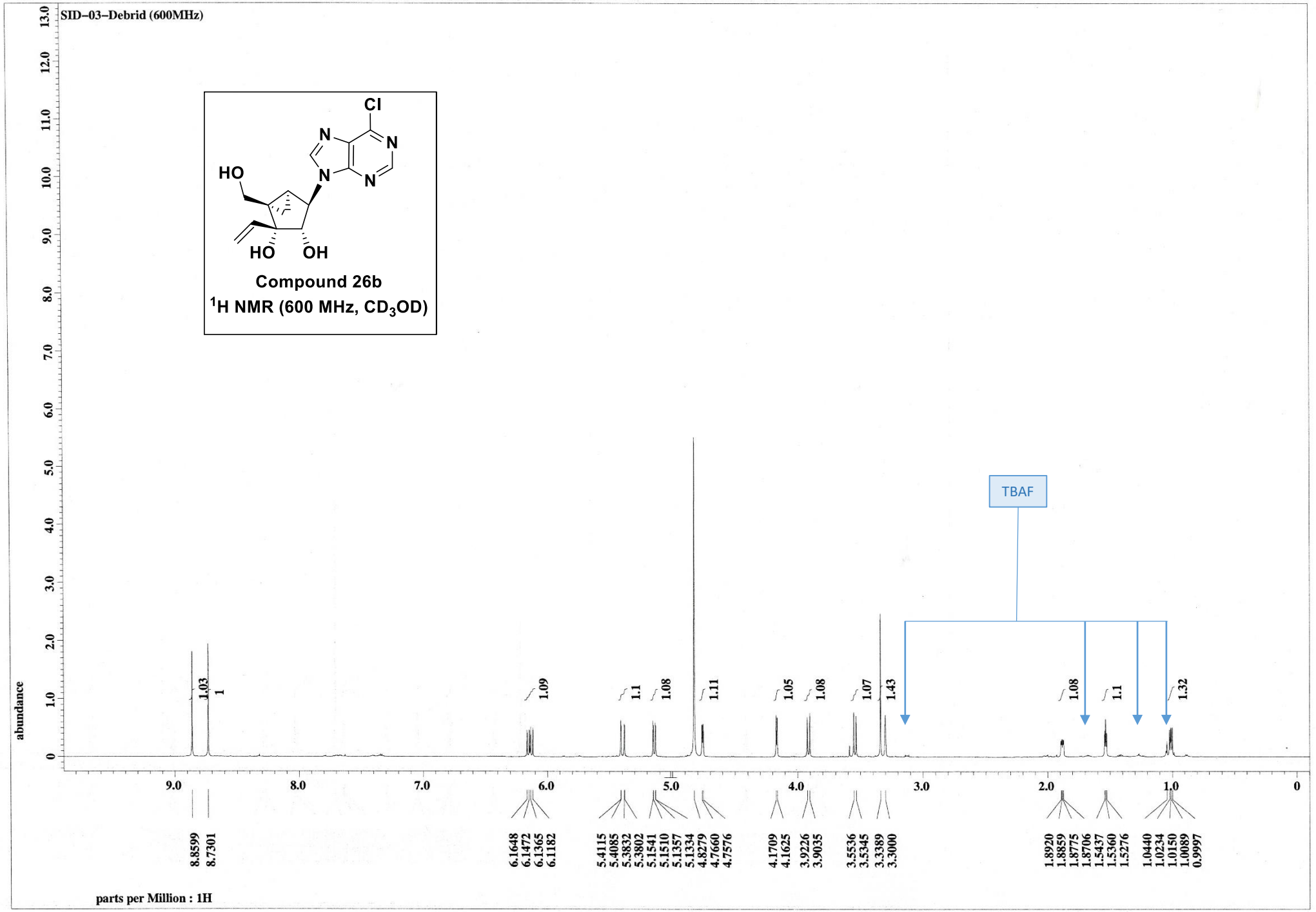

SID–03–09 (600MHz)
Cl
N
N
N
N
TBDPSO
HO
OTBS
Compound 25b
^{13}C NMR (150 MHz, $CDCl_3$)
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.8177
151.6693
151.2480
143.8611
139.1024
135.6603
135.5166
133.1756
132.9506
132.0745
129.9681
129.9010
127.8233
115.0458
84.7704
79.3128
77.2159
77.0005
76.7898
64.2708
63.3708
38.9886
26.9723
25.6606
22.1753
19.2742
17.8092
14.3049
–4.8016
–4.9596
ppm : 13C

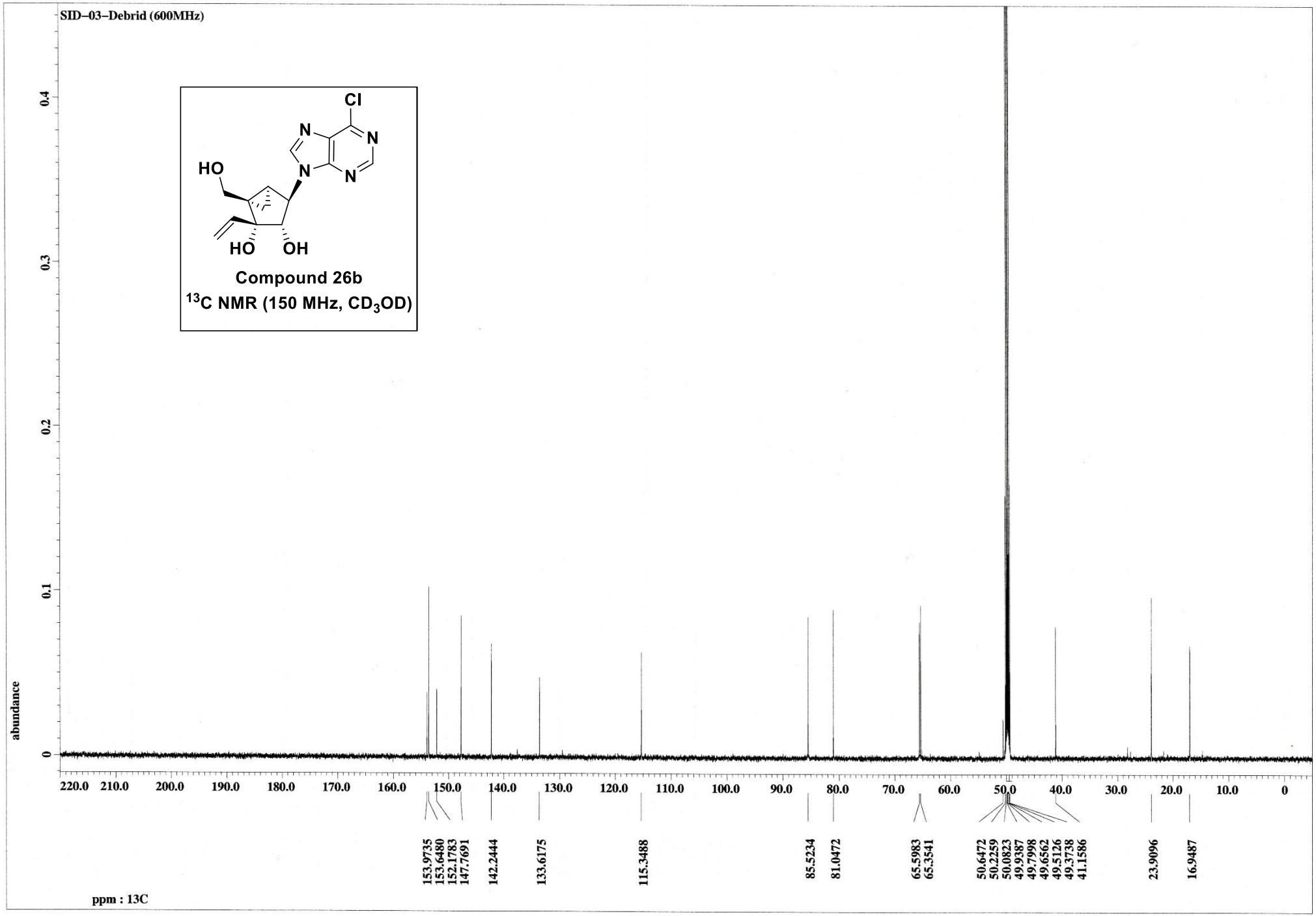

SID–03–512 (600MHz)
Cl
N
N
N
N
TBDPSO
TMS
HO
OTBS
Compound 25c
^{1}H NMR (600 MHz, $CDCl_3$)
abundance
ppm : 1H
1
1.03
4.17
2.12
4.15
1.03
2.12
1.04
1.0
1.05
1.05
1.35
9.49
9.45
3.11
3.13
9.3
8.7170
8.2049
7.6533
7.6495
7.6472
7.6426
7.6403
7.6365
7.6342
7.4486
7.4455
7.4402
7.4363
7.4279
7.4241
7.4111
7.4012
7.3989
7.3890
7.3775
7.2400
4.9477
4.3003
4.2820
4.2675
4.2499
4.0437
2.9550
1.8420
1.8313
1.8267
1.4264
1.4203
1.4119
1.2415
1.2331
1.2270
1.2186
1.0536
0.9337
0.2400
0.1055
−0.0175
−0.0228
−0.0289

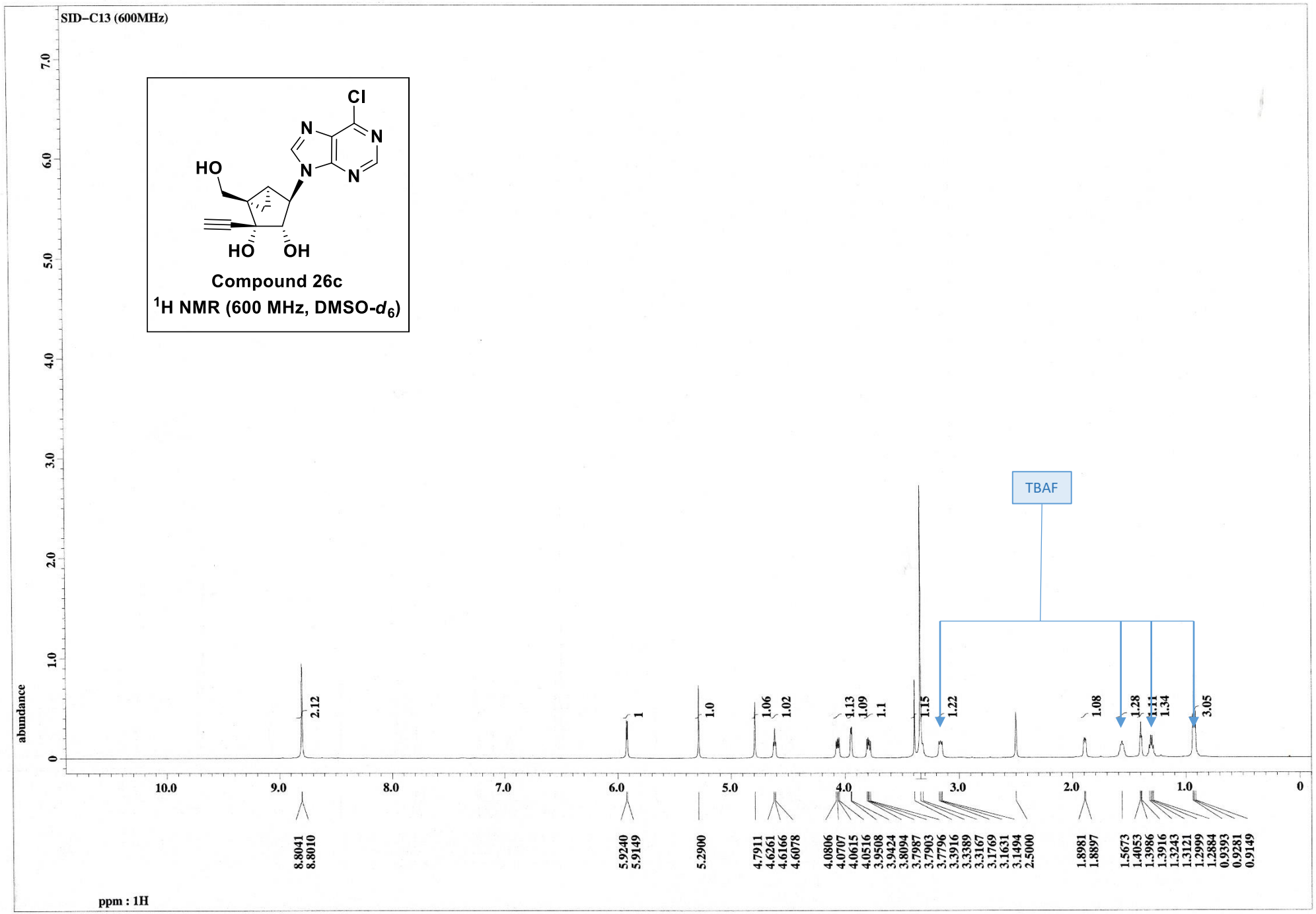

SID-03-S12 (600MHz)
Cl
N
N
N
N
TBDPSO
TMS
HO
OTBS
Compound 25c
13C NMR (150 MHz, CDCl3)
abundance
0.5
0.4
0.3
0.2
0.1
0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
151.6685
151.5105
150.9408
144.2289
135.5110
135.4680
133.0886
133.0264
131.8487
130.0343
129.9098
127.9230
127.8321
103.9574
94.1145
84.0658
77.2103
76.9996
76.7890
74.7256
62.2928
61.6082
39.5432
26.9093
25.7651
22.4953
19.3308
17.9808
10.1199
-0.4985
-4.6300
-5.1854
ppm : 13C

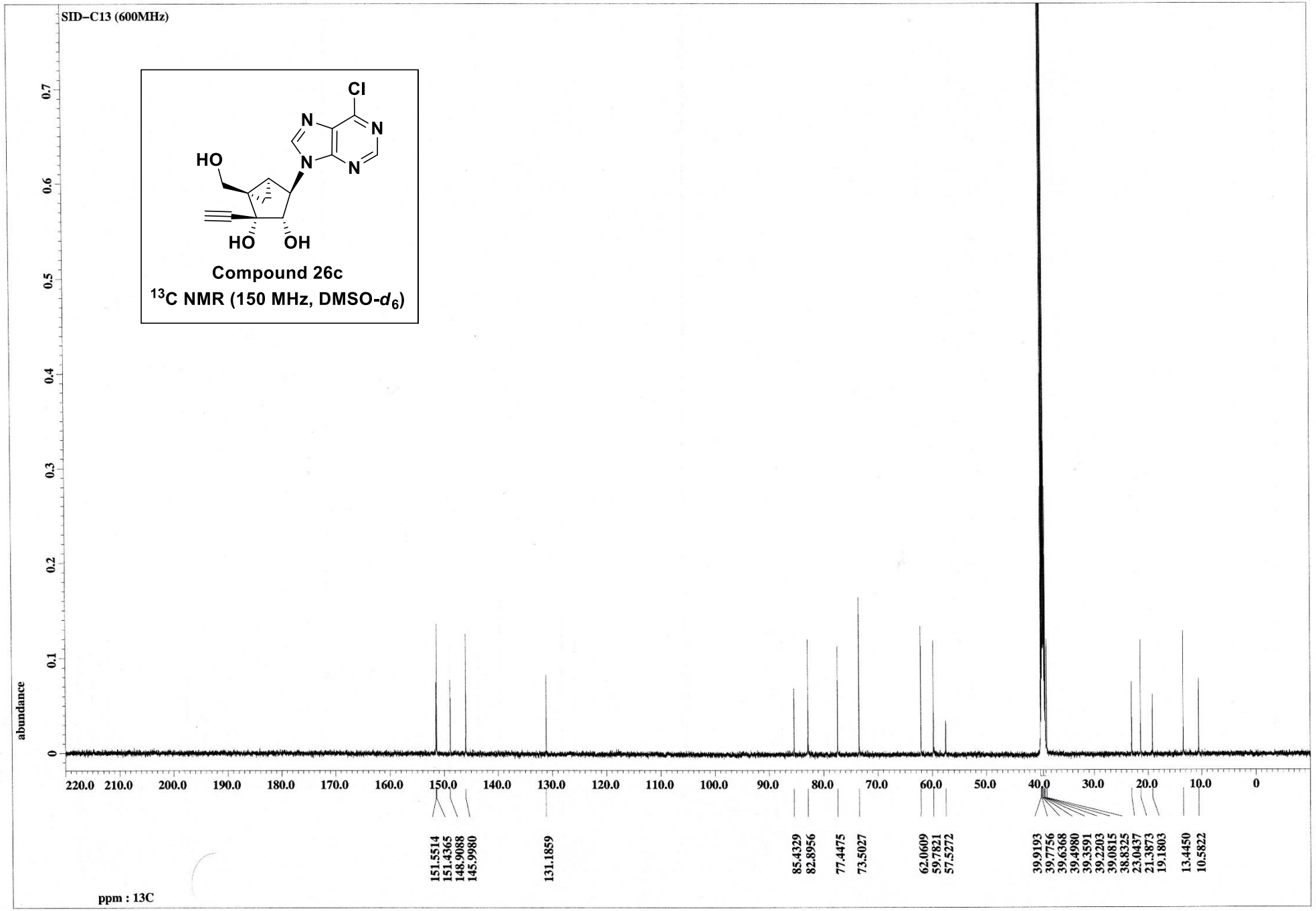

SID–C16 (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26a
^{1}H NMR (600 MHz, CD_3OD)
TBAF
abundance
ppm : 1H
8.9027
8.7255
4.8210
4.7713
4.7660
4.0226
4.0028
3.8676
3.8615
3.6208
3.6009
3.3000
3.2977
1.8676
1.8607
1.8530
1.8462
1.4864
1.4787
1.4703
1.3893
1.0142
0.9493
0.9409
0.9348
0.9256

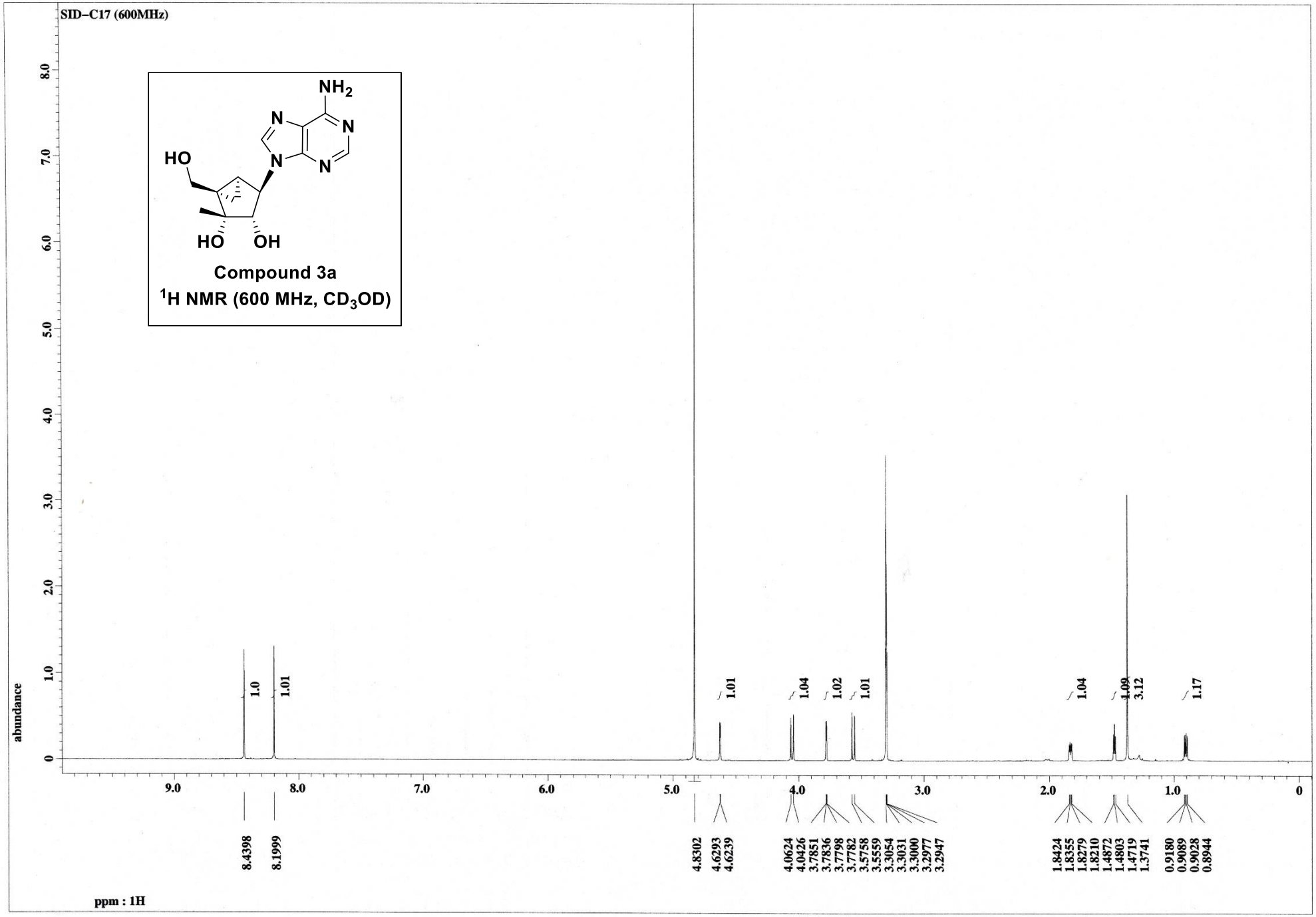

SID–C16 (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26a
13C NMR (150 MHz, CD3OD)
abundance
ppm : 13C
153.9165
153.6484
152.1499
147.5875
133.5988
86.1749
78.5485
65.3688
65.2348
50.2263
50.0875
49.9439
49.8002
49.6614
49.5178
49.3742
40.6036
26.3564
24.0871
16.5661

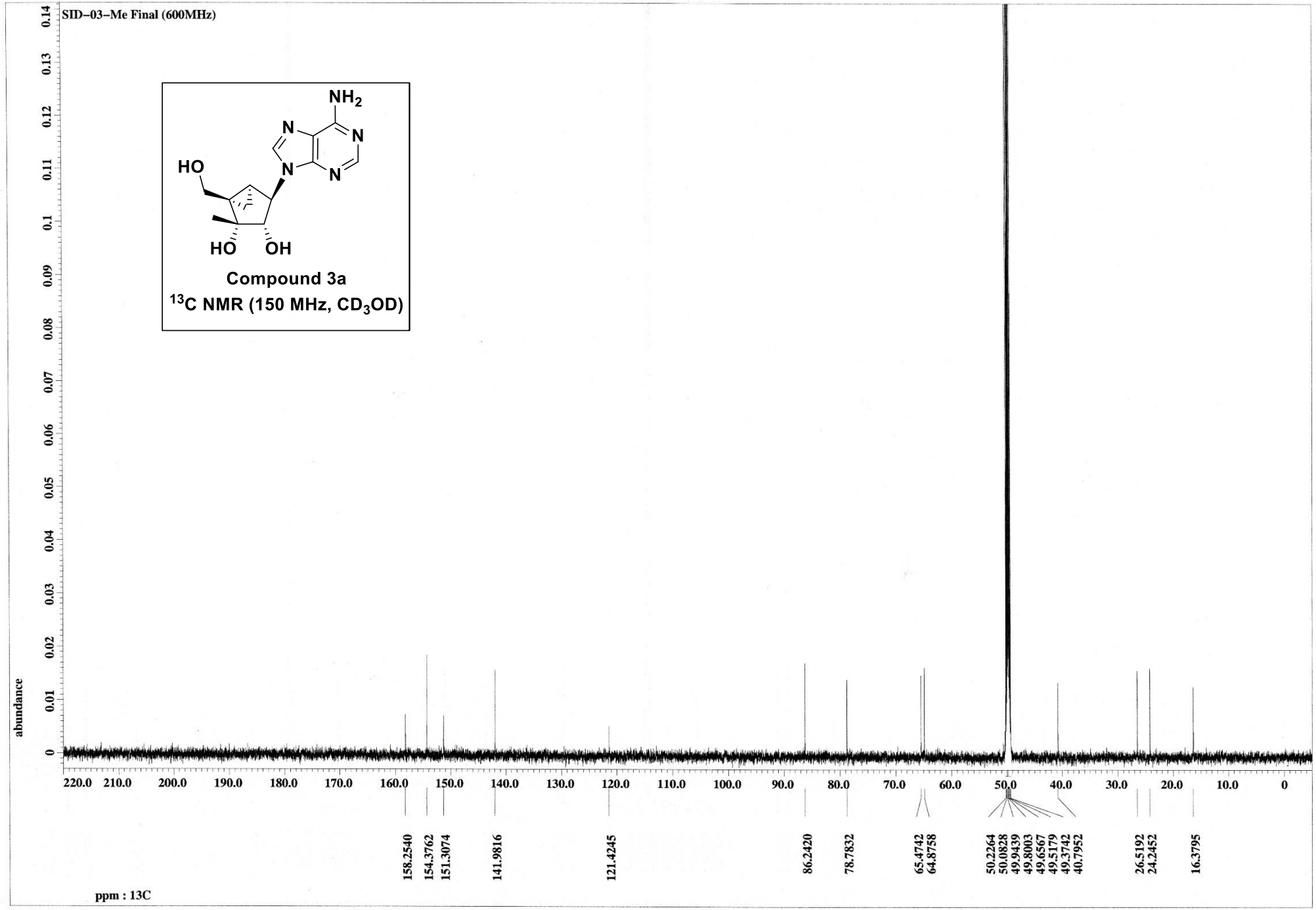

SID–03–Debrid (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26b
1H NMR (600 MHz, CD3OD)
TBAF
abundance
parts per Million : 1H
8.8599
8.7301
6.1648
6.1472
6.1365
6.1182
5.4115
5.4085
5.3832
5.3802
5.1541
5.1510
5.1357
5.1334
4.8279
4.7660
4.7576
4.1709
4.1625
3.9226
3.9035
3.5536
3.5345
3.3389
3.3000
1.8920
1.8859
1.8775
1.8706
1.5437
1.5360
1.5276
1.0440
1.0234
1.0150
1.0089
0.9997

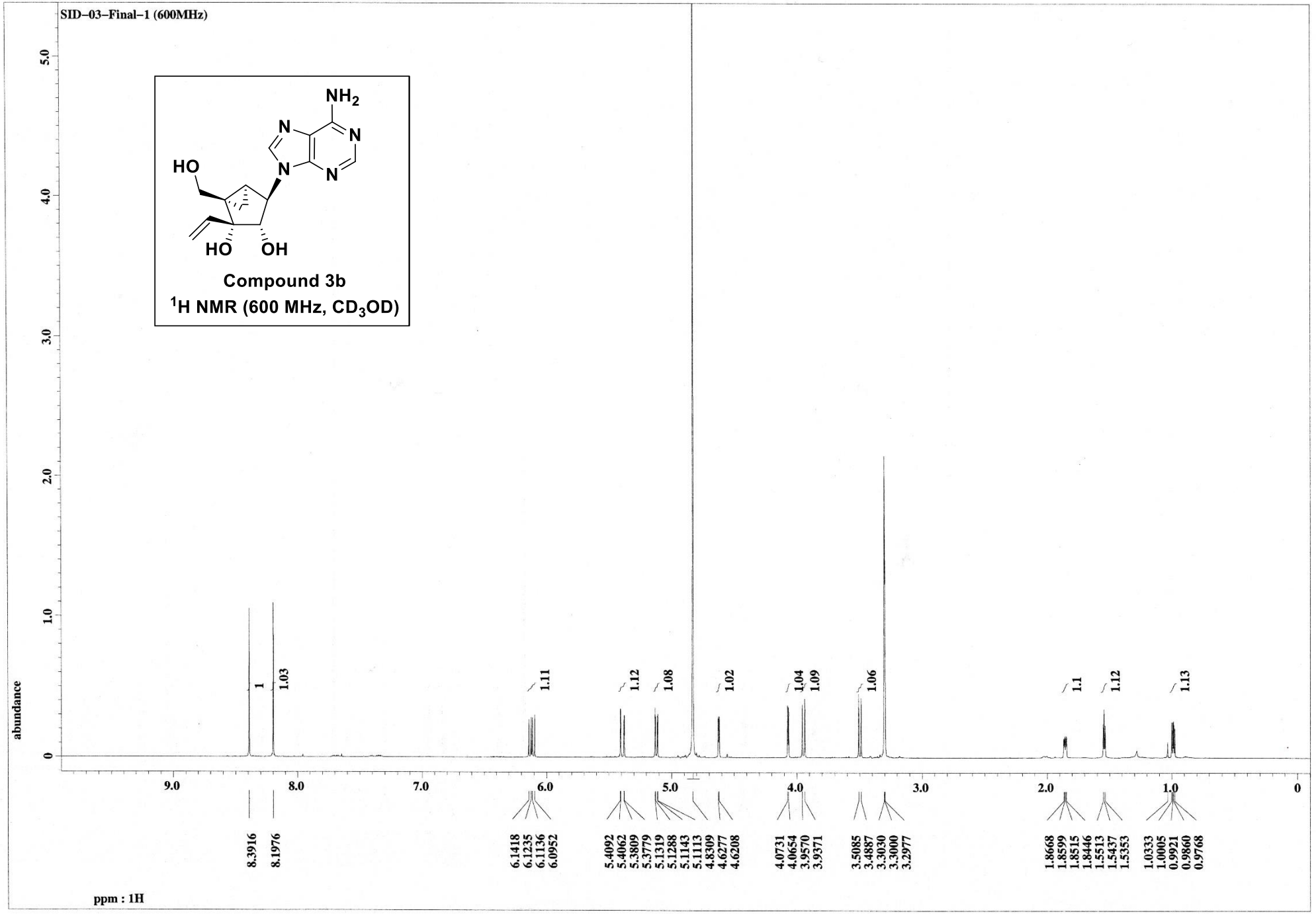

SID–03–Debrid (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26b
13C NMR (150 MHz, CD3OD)
abundance
0
0.1
0.2
0.3
0.4
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
153.9735
153.6480
152.1783
147.7691
142.2444
133.6175
115.3488
85.5234
81.0472
65.5983
65.3541
50.6472
50.2259
50.0823
49.9387
49.7998
49.6562
49.5126
49.3738
41.1586
23.9096
16.9487
ppm : 13C


SID–C13 (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26c
^{1}H NMR (600 MHz, DMSO-d_6)
TBAF
abundance
ppm : 1H
8.8041
8.8010
5.9240
5.9149
5.2900
4.7911
4.6261
4.6166
4.6078
4.0806
4.0707
4.0615
4.0516
3.9508
3.9424
3.8094
3.7987
3.7903
3.7796
3.3916
3.3389
3.3167
3.1769
3.1631
3.1494
2.5000
1.8981
1.8897
1.5673
1.4053
1.3986
1.3916
1.3243
1.3121
1.2999
1.2884
0.9393
0.9281
0.9149

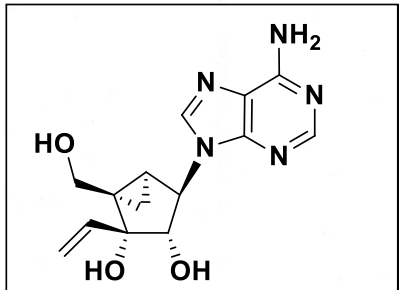

SID−C13 (600MHz)
Cl
N
N
N
N
HO
HO
OH
Compound 26c
^{13}C NMR (150 MHz, DMSO-d_6)
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.5514
151.4365
148.9088
145.9980
131.1859
85.4329
82.8956
77.4475
73.5027
62.0609
59.7821
57.5272
39.9193
39.7756
39.6368
39.4980
39.3591
39.2203
39.0815
38.8325
23.0437
21.3873
19.1803
13.4450
10.5822
ppm : 13C


SID–C17 (600MHz)
NH2
N
N
N
N
HO
HO
OH
Compound 3a
1H NMR (600 MHz, CD3OD)
abundance
ppm : 1H
1.0
1.01
1.01
1.04
1.02
1.01
1.04
1.09
3.12
1.17
8.4398
8.1999
4.8302
4.6293
4.6239
4.0624
4.0426
3.7851
3.7836
3.7798
3.7782
3.5758
3.5559
3.3054
3.3031
3.3000
3.2977
3.2947
1.8424
1.8355
1.8279
1.8210
1.4872
1.4803
1.4719
1.3741
0.9180
0.9089
0.9028
0.8944


SID–03–Me Final (600MHz)
NH2
N
N
N
N
HO
HO
OH
Compound 3a
13C NMR (150 MHz, CD3OD)
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
0.13
0.14
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
158.2540
154.3762
151.3074
141.9816
121.4245
86.2420
78.7832
65.4742
64.8758
50.2264
50.0828
49.9439
49.8003
49.6567
49.5179
49.3742
40.7952
26.5192
24.2452
16.3795
ppm : 13C


SID–03–Final–1 (600MHz)
NH2
N
N
N
N
HO
HO
OH
Compound 3b
1H NMR (600 MHz, CD3OD)
abundance
ppm : 1H
1
1.03
1.11
1.12
1.08
1.02
1.04
1.09
1.06
1.1
1.12
1.13
8.3916
8.1976
6.1418
6.1235
6.1136
6.0952
5.4092
5.4062
5.3809
5.3779
5.1319
5.1288
5.1143
5.1113
4.8309
4.6277
4.6208
4.0731
4.0654
3.9570
3.9371
3.5085
3.4887
3.3030
3.3000
3.2977
1.8668
1.8599
1.8515
1.8446
1.5513
1.5437
1.5353
1.0333
1.0005
0.9921
0.9860
0.9768

SID–03–Final–1 (600MHz)



Compound 3b

^{13}C NMR (150 MHz, CD_3OD)

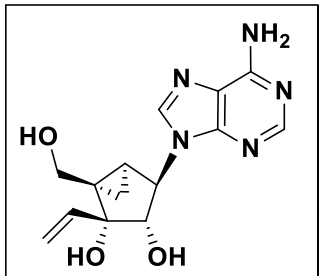
abundance

0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17

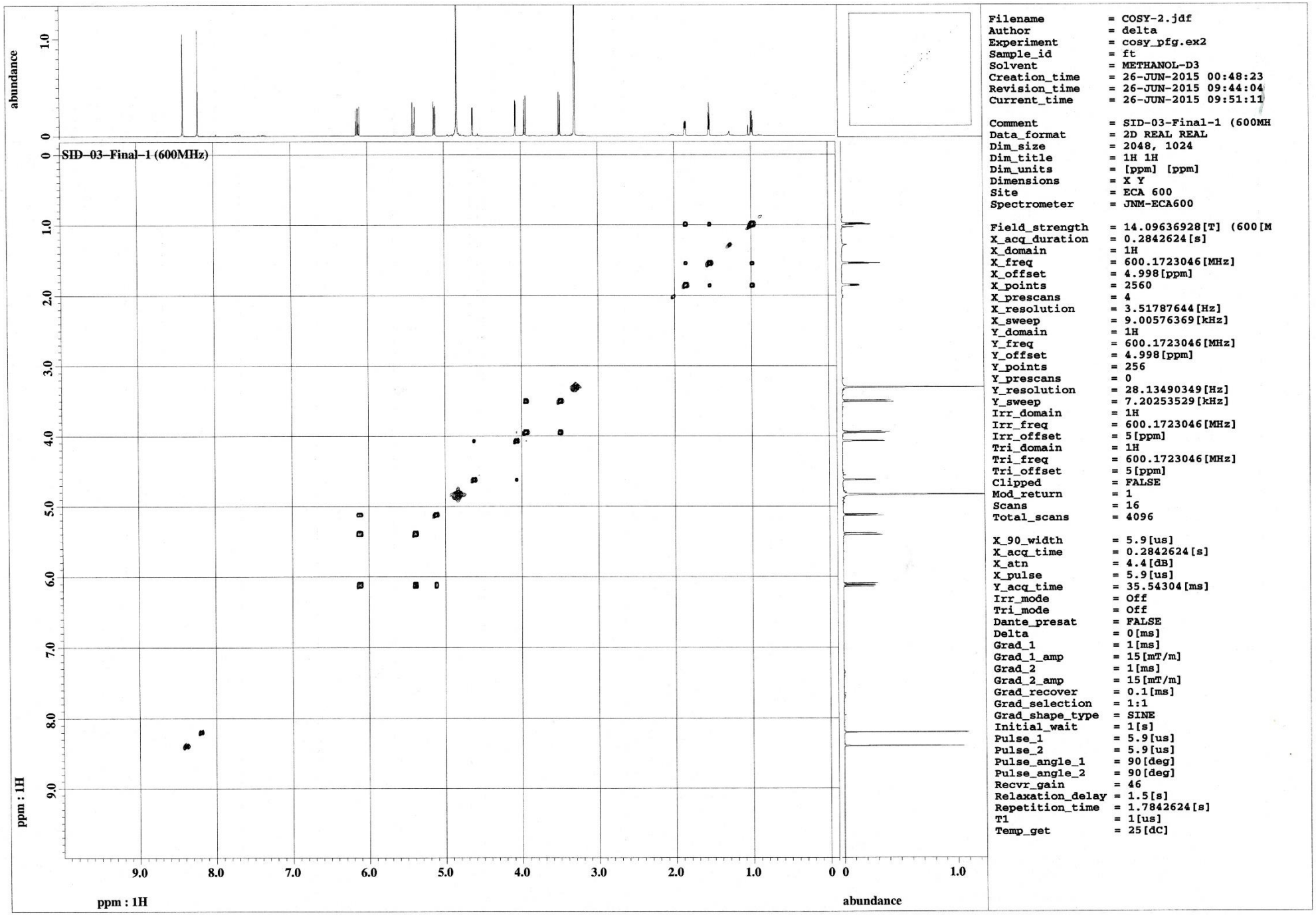
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0

158.2676
154.3802
151.3690
142.4644
142.1293
121.4908
115.0087
85.7098
81.3342
65.5549
65.2916
50.2257
50.0821
49.9432
49.7996
49.6560
49.5171
49.3735
41.4169
24.1775
16.7714

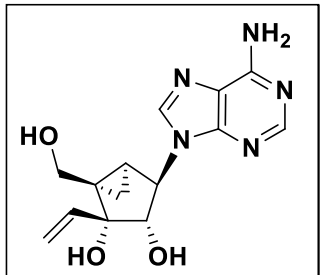
ppm : 13C



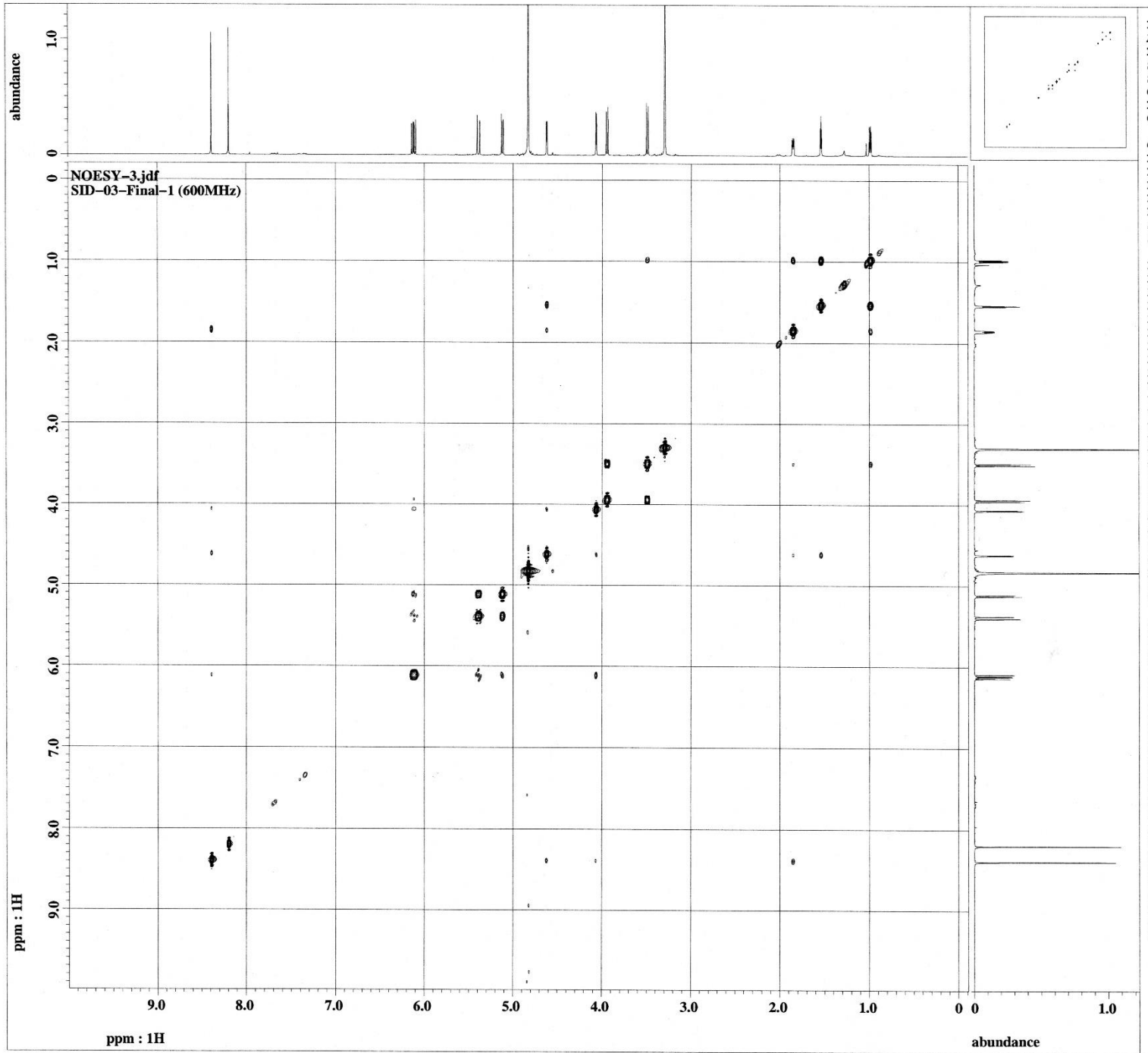
Compound 3b
COSY
(600 MHz, CD_3OD)



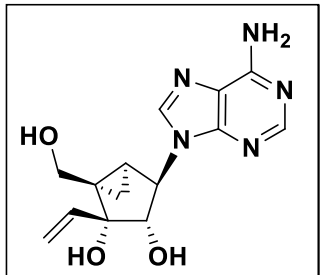

```
Filename          = COSY-2.jdf
Author            = delta
Experiment        = cosy_pfg.ex2
Sample_id         = ft
Solvent           = METHANOL-D3
Creation_time     = 26-JUN-2015 00:48:23
Revision_time     = 26-JUN-2015 09:44:04
Current_time      = 26-JUN-2015 09:51:11

Comment           = SID-03-Final-1 (600MH
Data_format       = 2D REAL REAL
Dim_size          = 2048, 1024
Dim_title         = 1H 1H
Dim_units         = [ppm] [ppm]
Dimensions        = X Y
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.2842624[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 4.998[ppm]
X_points          = 2560
X_prescans        = 4
X_resolution      = 3.51787644[Hz]
X_sweep           = 9.00576369[kHz]
Y_domain          = 1H
Y_freq            = 600.1723046[MHz]
Y_offset          = 4.998[ppm]
Y_points          = 256
Y_prescans        = 0
Y_resolution      = 28.13490349[Hz]
Y_sweep           = 7.20253529[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 4096

X_90_width        = 5.9[us]
X_acq_time        = 0.2842624[s]
X_atn             = 4.4[dB]
X_pulse           = 5.9[us]
Y_acq_time        = 35.54304[ms]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Delta             = 0[ms]
Grad_1            = 1[ms]
Grad_1_amp        = 15[mT/m]
Grad_2            = 1[ms]
Grad_2_amp        = 15[mT/m]
Grad_recover      = 0.1[ms]
Grad_selection    = 1:1
Grad_shape_type   = SINE
Initial_wait      = 1[s]
Pulse_1           = 5.9[us]
Pulse_2           = 5.9[us]
Pulse_angle_1     = 90[deg]
Pulse_angle_2     = 90[deg]
Recvr_gain        = 46
Relaxation_delay  = 1.5[s]
Repetition_time   = 1.7842624[s]
T1                = 1[us]
Temp_get          = 25[dC]
```



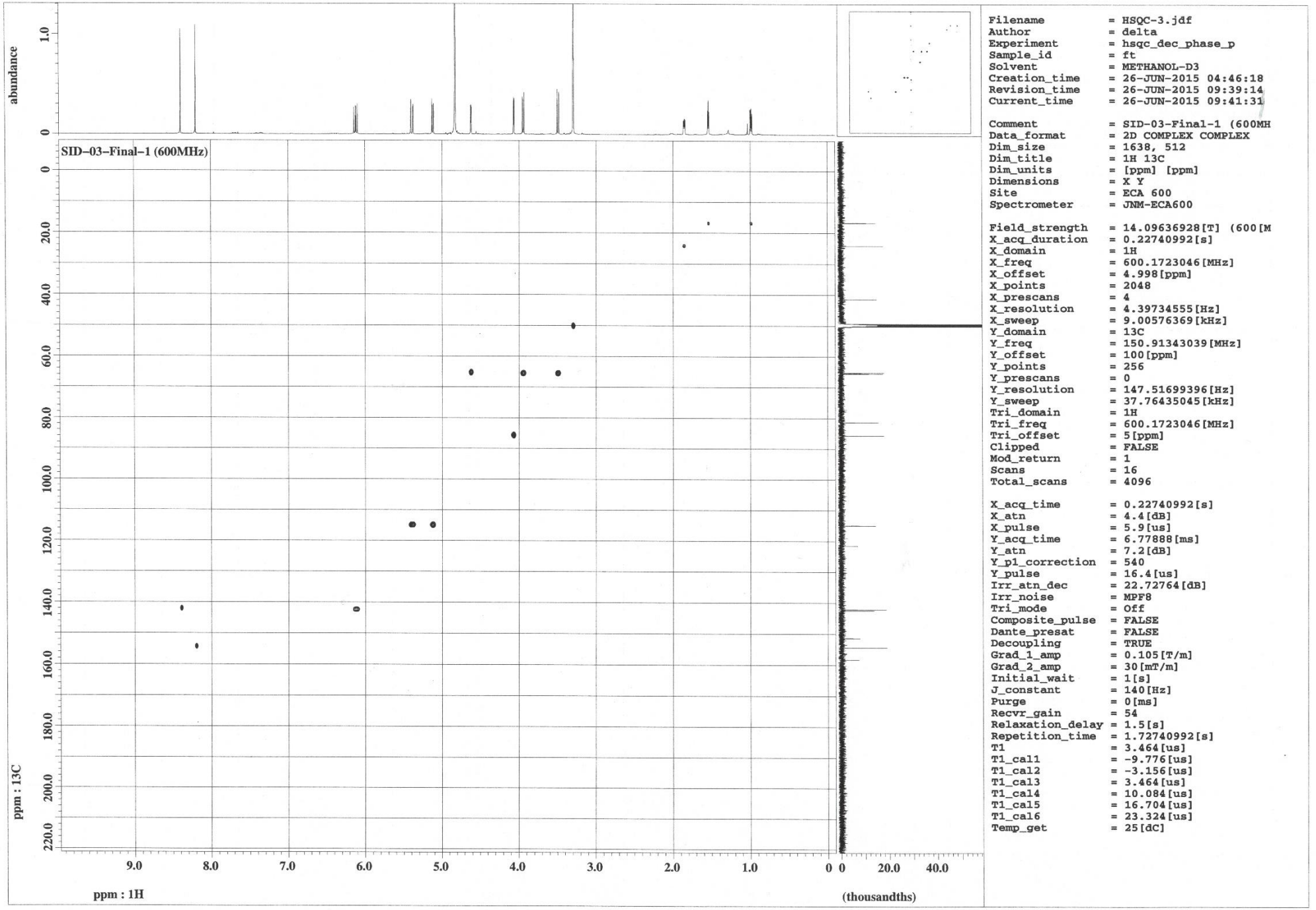
Compound 3b
NOESY
(600 MHz, CD_3OD)



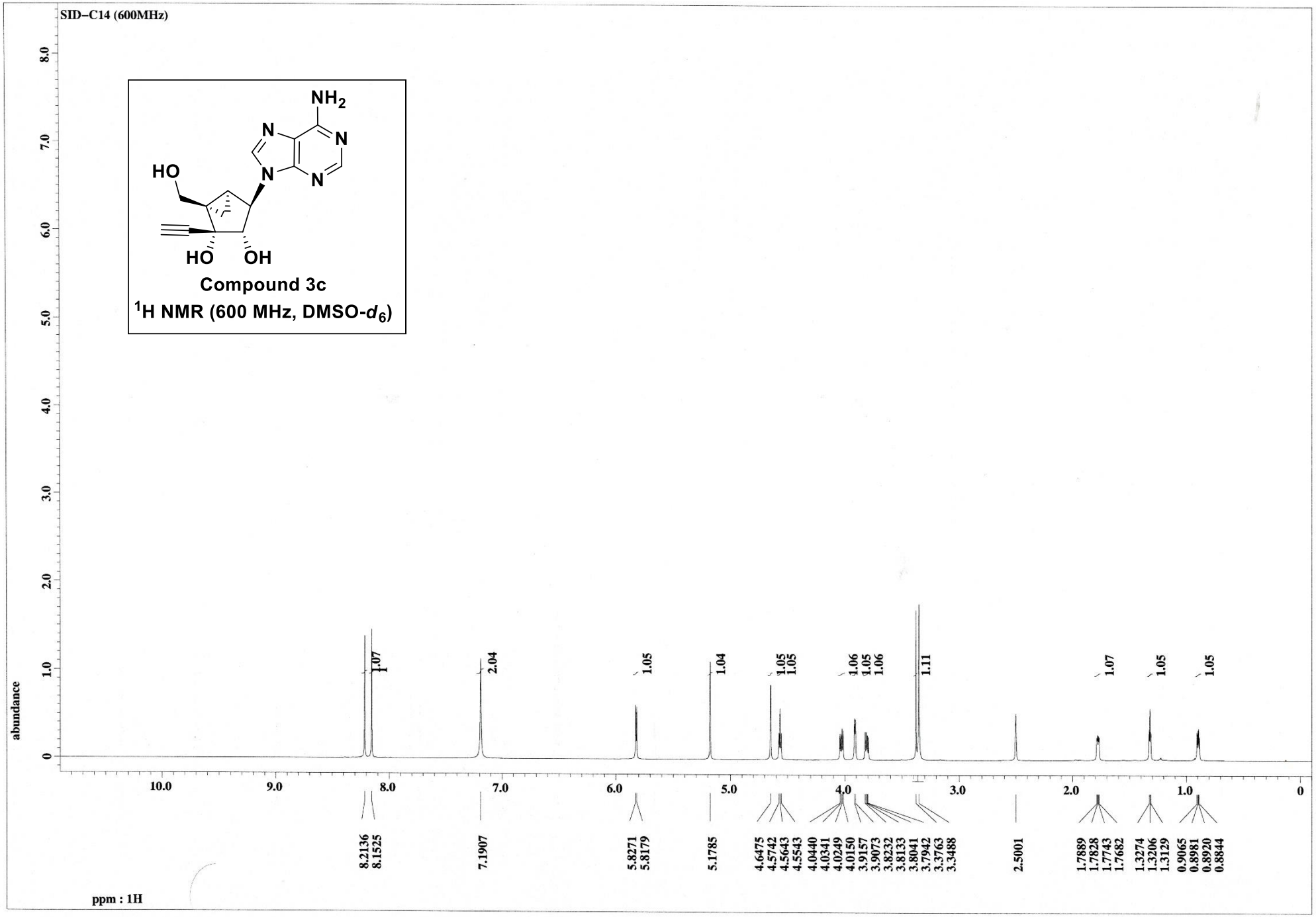

```
Filename          = NOESY-3.jdf
Author            = delta
Experiment        = noesy_phase_pfgz
Sample_id         = ft
Solvent           = METHANOL-D3
Creation_time     = 25-JUN-2015 22:45:04
Revision_time     = 26-JUN-2015 09:56:35
Current_time      = 26-JUN-2015 10:05:27

Comment           = SID-03-Final-1 (600MH
Data_format       = 2D REAL REAL
Dim_size          = 1638, 512
Dim_title         = 1H 1H
Dim_units         = [ppm] [ppm]
Dimensions        = X Y
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.22740992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 4.998[ppm]
X_points          = 2048
X_prescans        = 4
X_resolution      = 4.39734555[Hz]
X_sweep           = 9.00576369[kHz]
Y_domain          = 1H
Y_freq            = 600.1723046[MHz]
Y_offset          = 4.998[ppm]
Y_points          = 256
Y_prescans        = 0
Y_resolution      = 28.13490349[Hz]
Y_sweep           = 7.20253529[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 12
Total_scans       = 3072

X_acq_time        = 0.22740992[s]
X_atn             = 4.4[dB]
X_pulse           = 5.9[us]
Y_acq_time        = 35.54304[ms]
Y_p1_correction   = 180
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Grad_1            = 1[ms]
Grad_1_amp        = 45[mT/m]
Initial_wait      = 1[s]
Mix_time          = 0.8[s]
Recvr_gain        = 46
Relaxation_delay  = 1.5[s]
Repetition_time   = 1.72740992[s]
Scramble          = 1[ms]
T1                = 61.868[us]
T1_cal1           = 61.868[us]
T1_cal2           = 0.131288[ms]
T1_cal3           = 0.200708[ms]
T1_cal4           = 0.270128[ms]
T1_cal5           = 0.339548[ms]
T1_cal6           = 0.408968[ms]
Temp_get          = 25[dC]
```

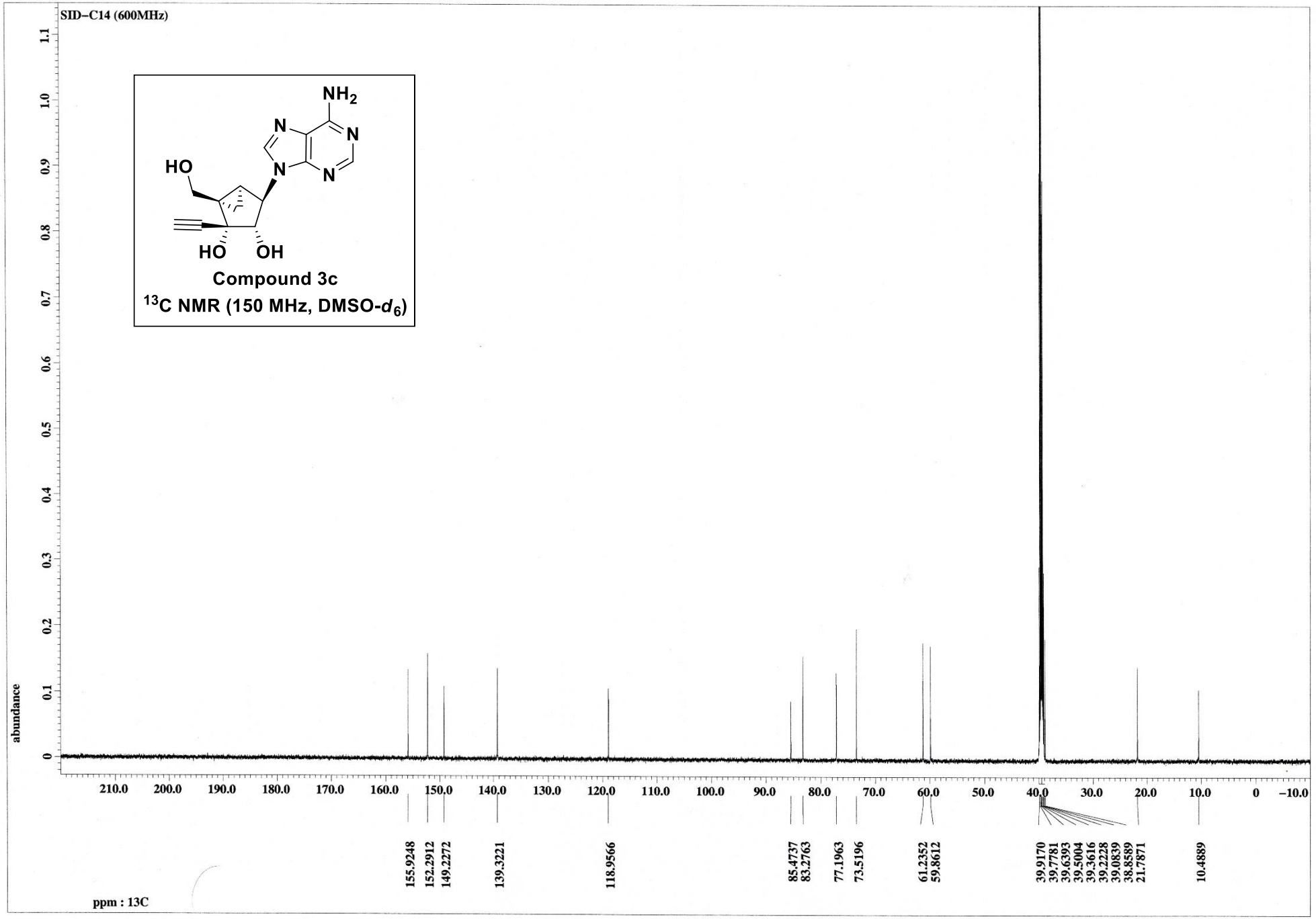


Compound 3b
HSQC
(600 MHz, CD_3OD)




```
Filename          = HSQC-3.jdf
Author            = delta
Experiment        = hsqc_dec_phase_p
Sample_id         = ft
Solvent           = METHANOL-D3
Creation_time     = 26-JUN-2015 04:46:18
Revision_time     = 26-JUN-2015 09:39:14
Current_time      = 26-JUN-2015 09:41:31

Comment           = SID-03-Final-1 (600MH
Data_format       = 2D COMPLEX COMPLEX
Dim_size          = 1638, 512
Dim_title         = 1H 13C
Dim_units         = [ppm] [ppm]
Dimensions        = X Y
Site              = ECA 600
Spectrometer      = JNM-ECA600

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.22740992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 4.998[ppm]
X_points          = 2048
X_prescans        = 4
X_resolution      = 4.39734555[Hz]
X_sweep           = 9.00576369[kHz]
Y_domain          = 13C
Y_freq            = 150.91343039[MHz]
Y_offset          = 100[ppm]
Y_points          = 256
Y_prescans        = 0
Y_resolution      = 147.51699396[Hz]
Y_sweep           = 37.76435045[kHz]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 4096

X_acq_time        = 0.22740992[s]
X_atn             = 4.4[dB]
X_pulse           = 5.9[us]
Y_acq_time        = 6.77888[ms]
Y_atn             = 7.2[dB]
Y_p1_correction   = 540
Y_pulse           = 16.4[us]
Irr_atn_dec       = 22.72764[dB]
Irr_noise         = MPF8
Tri_mode          = Off
Composite_pulse   = FALSE
Dante_presat      = FALSE
Decoupling        = TRUE
Grad_1_amp        = 0.105[T/m]
Grad_2_amp        = 30[mT/m]
Initial_wait      = 1[s]
J_constant        = 140[Hz]
Purge             = 0[ms]
Recvr_gain        = 54
Relaxation_delay  = 1.5[s]
Repetition_time   = 1.72740992[s]
T1                = 3.464[us]
T1_cal1           = -9.776[us]
T1_cal2           = -3.156[us]
T1_cal3           = 3.464[us]
T1_cal4           = 10.084[us]
T1_cal5           = 16.704[us]
T1_cal6           = 23.324[us]
Temp_get          = 25[dC]
```


SID–C14 (600MHz)
NH2
N
N
N
N
HO
HO
OH
Compound 3c
1H NMR (600 MHz, DMSO-d6)
abundance
ppm : 1H
8.2136
8.1525
7.1907
5.8271
5.8179
5.1785
4.6475
4.5742
4.5643
4.5543
4.0440
4.0341
4.0249
4.0150
3.9157
3.9073
3.8232
3.8133
3.8041
3.7942
3.7763
3.3488
2.5001
1.7889
1.7828
1.7743
1.7682
1.3274
1.3206
1.3129
0.9065
0.8981
0.8920
0.8844


SID−C14 (600MHz)
NH2
N
N
N
N
HO
HO
OH
Compound 3c
13C NMR (150 MHz, DMSO-d6)
abundance
ppm : 13C
155.9248
152.2912
149.2272
139.3221
118.9566
85.4737
83.2763
77.1963
73.5196
61.2352
59.8612
39.9170
39.7781
39.6393
39.5004
39.3616
39.2228
39.0839
38.8589
21.7871
10.4889