Supporting information

Divergent total synthesis of 1,6,8a-tri-*epi*-castanospermine and 1-deoxy-6,8a-di-*epi*-castanospermine from substituted azetidin-2one (β-lactam), involving a cascade sequence of reactions as key step

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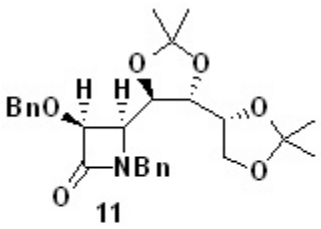
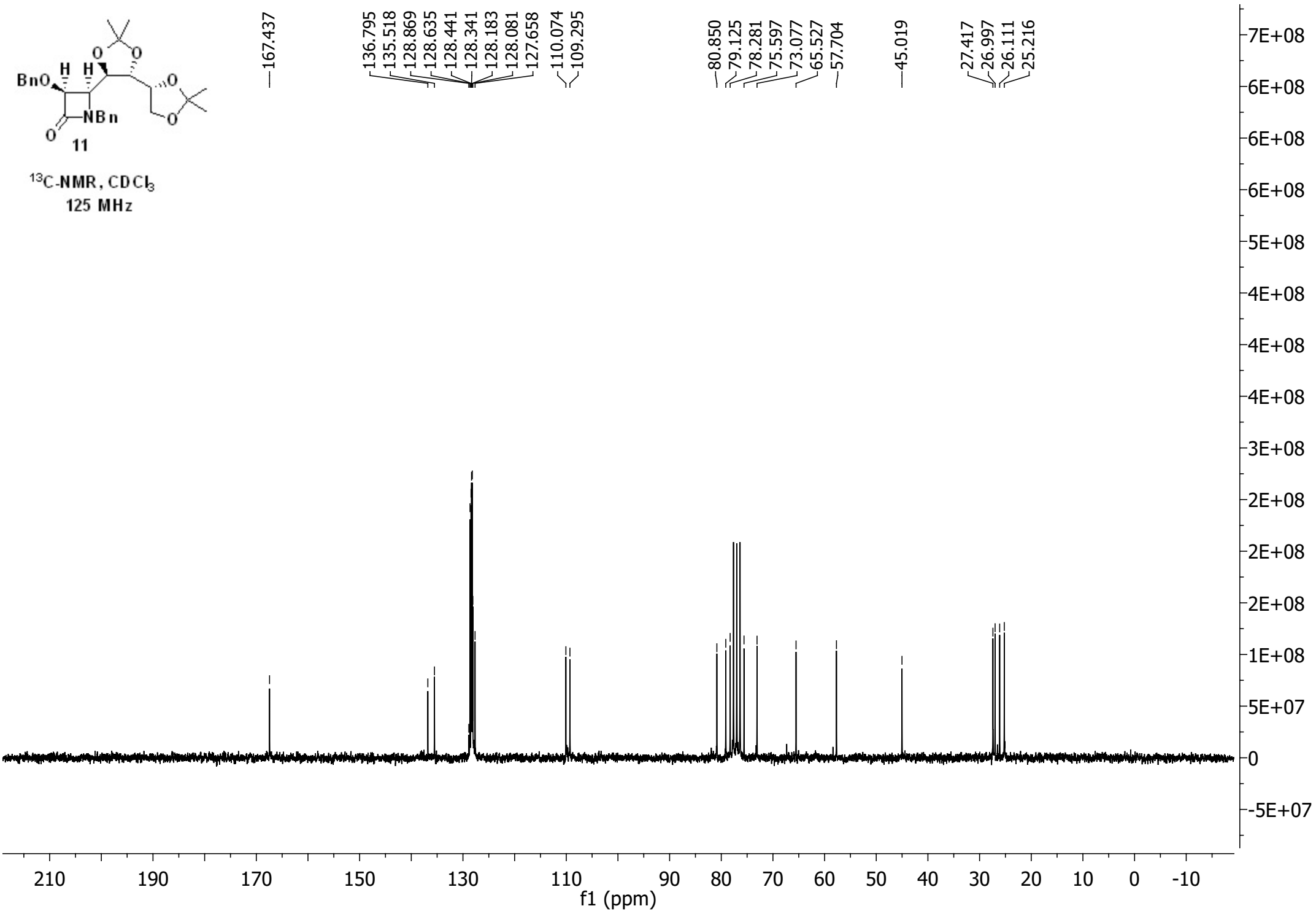
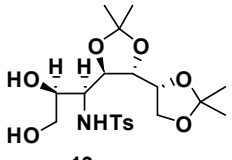


^{1}H-NMR, $CDCl_3$
500, MHz


BnO
H
H
O
O
O
O
NBn
O
11
13C-NMR, CDCl3
125 MHz
167.437
136.795
135.518
128.869
128.635
128.441
128.341
128.183
128.081
127.658
110.074
109.295
80.850
79.125
78.281
75.597
73.077
65.527
57.704
45.019
27.417
26.997
26.111
25.216
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



13

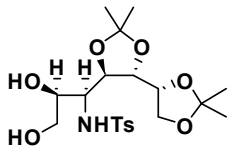
^{1}H-NMR, $CDCl_3$
500, MHz

7.783
7.742
7.329
7.286
7.252

4.126
4.101
4.090
4.059
3.833
3.805
3.795
3.795
3.780
3.780
3.759
3.745
3.717
3.693
3.678
3.648
3.599
2.423
1.455
1.358
1.283
1.229
4.021
4.002
3.984
3.964
3.918
3.887
3.845

1.8
2.0
1.3
8.0
3.0
2.6
3.0
3.9
3.3

12 11 10 9 8 7 6 5 4 3 2 1



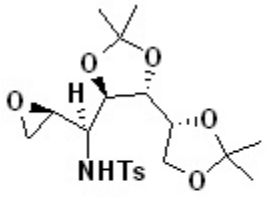
13

^{13}C-NMR, $CDCl_3$
125, MHz

143.767
137.787
127.039
127.300
129.608
129.705
109.807
110.133
77.731
76.808
71.109
68.068
62.904
80.478
54.070
26.784
26.685
26.079
25.066
21.522

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

Fri4av2#054
Dharmendra/10756

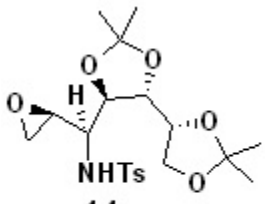


14

^{1}H-NMR, $CDCl_3$

200 MHz

Thu5av2#048
Dharmendra/10756



14

^{13}C-NMR, $CDCl_3$

50 MHz

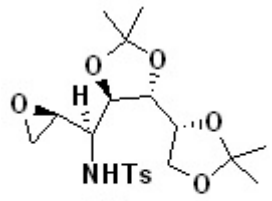
143.506
138.187
129.693
126.946
110.416
109.553
79.941
77.726
77.087
76.456
68.176
51.550
50.114
43.016
26.789
26.634
26.076
25.264
21.014

22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000

210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10

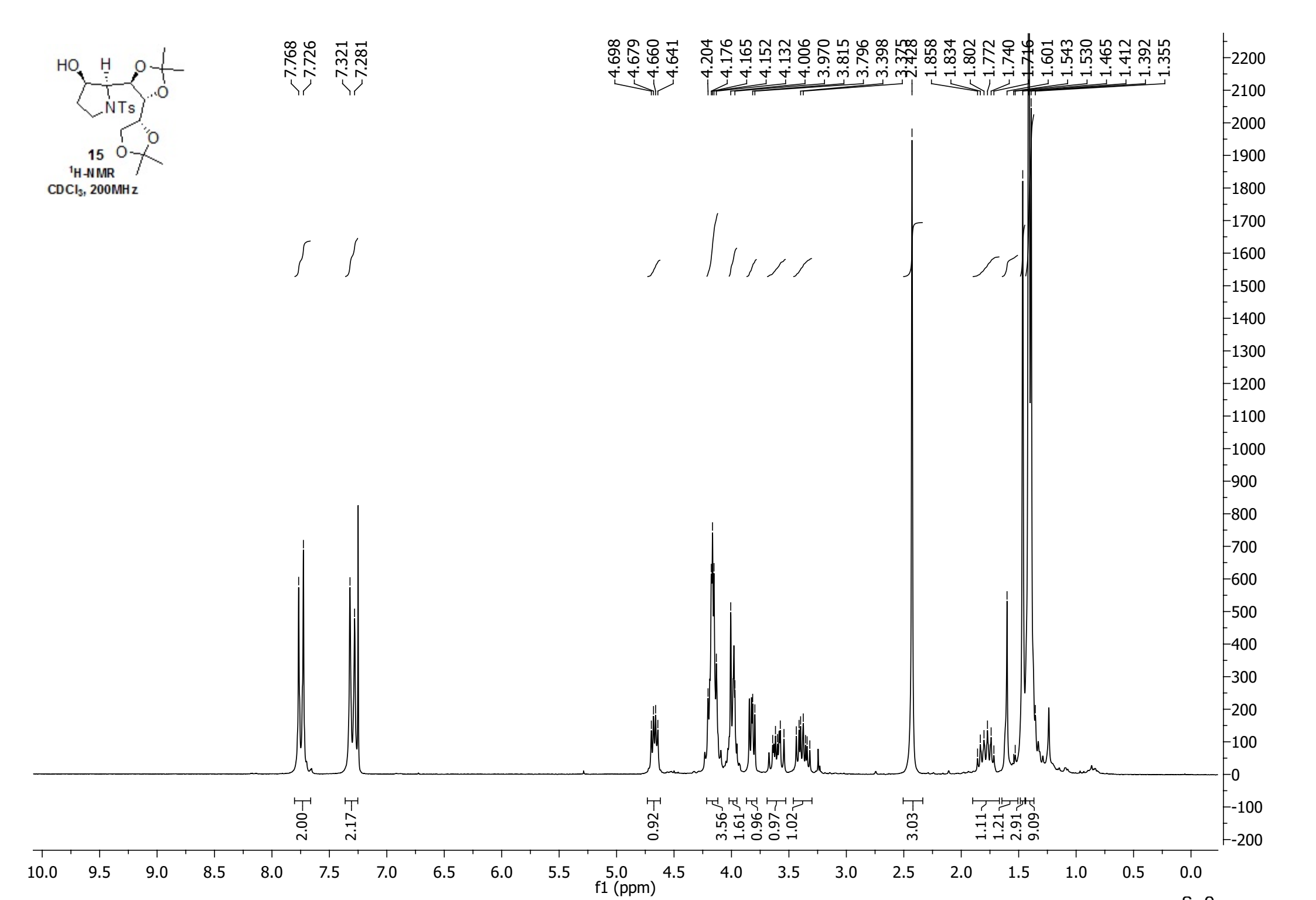
f1 (ppm)

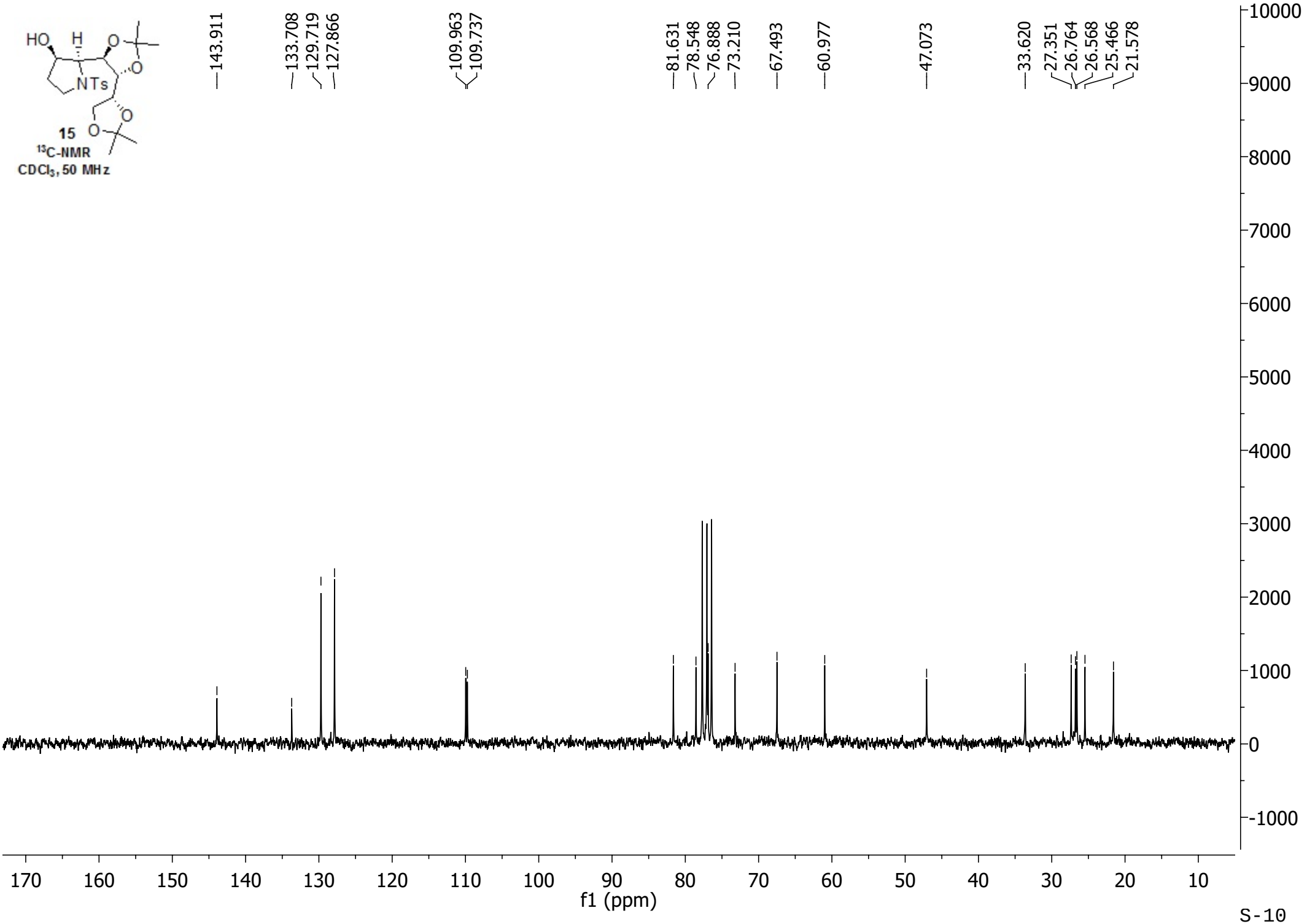
Thu5av2#048
Dharmendra/10756

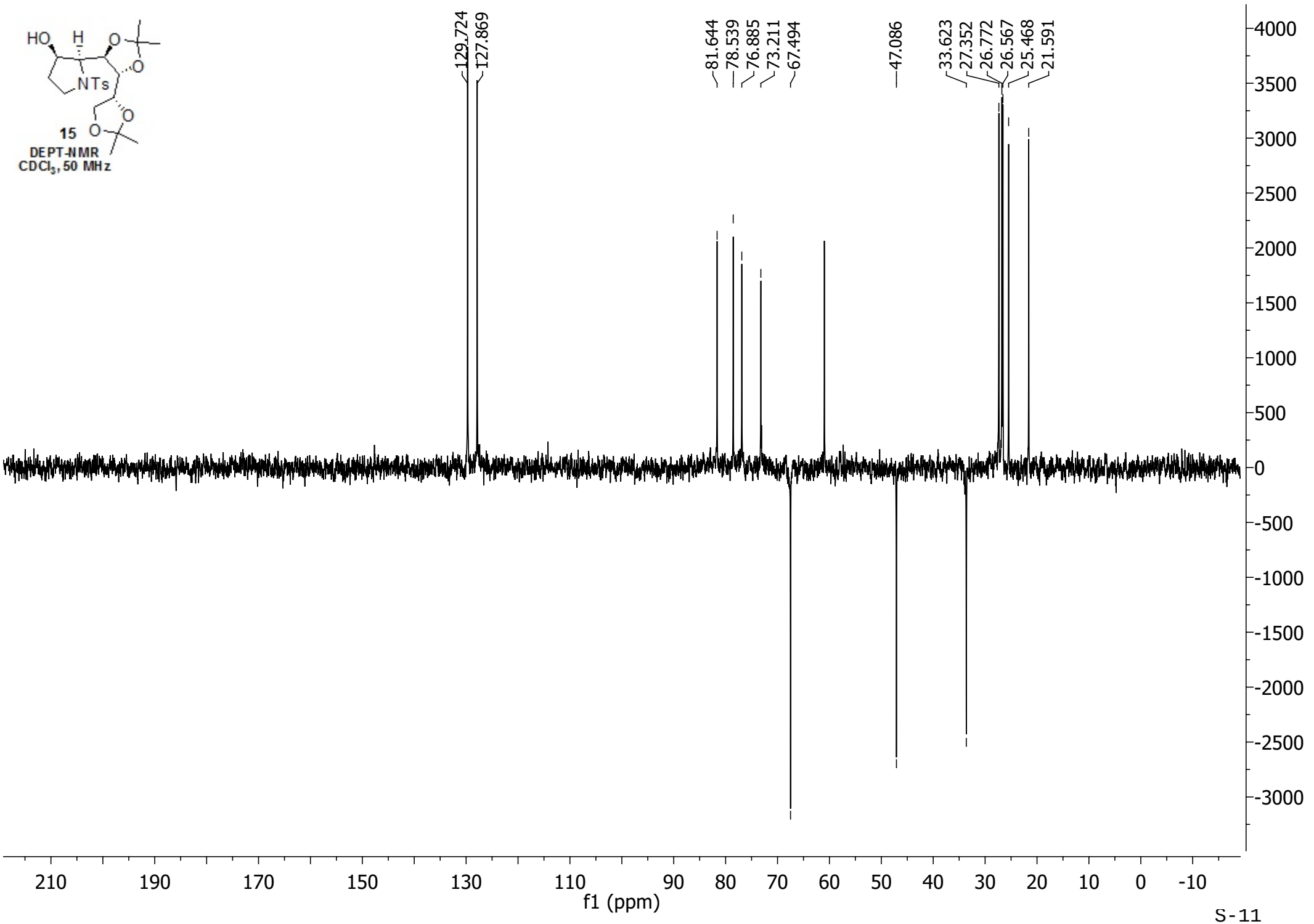


14

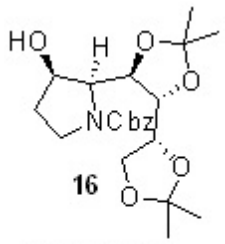
DEPT-NMR, $CDCl_3$
50 MHz


HO
H
O
O
NTs
O
O
15
1H-NMR
CDCl3, 200MHz
7.768
7.726
7.321
7.281
4.698
4.679
4.660
4.641
4.204
4.176
4.165
4.152
4.132
4.006
3.970
3.815
3.796
3.398
3.375
3.328
2.428
1.858
1.834
1.802
1.772
1.740
1.716
1.601
1.543
1.530
1.465
1.412
1.392
1.355
2.00
2.17
0.92
3.56
1.61
0.96
0.97
1.02
3.03
1.11
1.21
2.91
9.09
f1 (ppm)

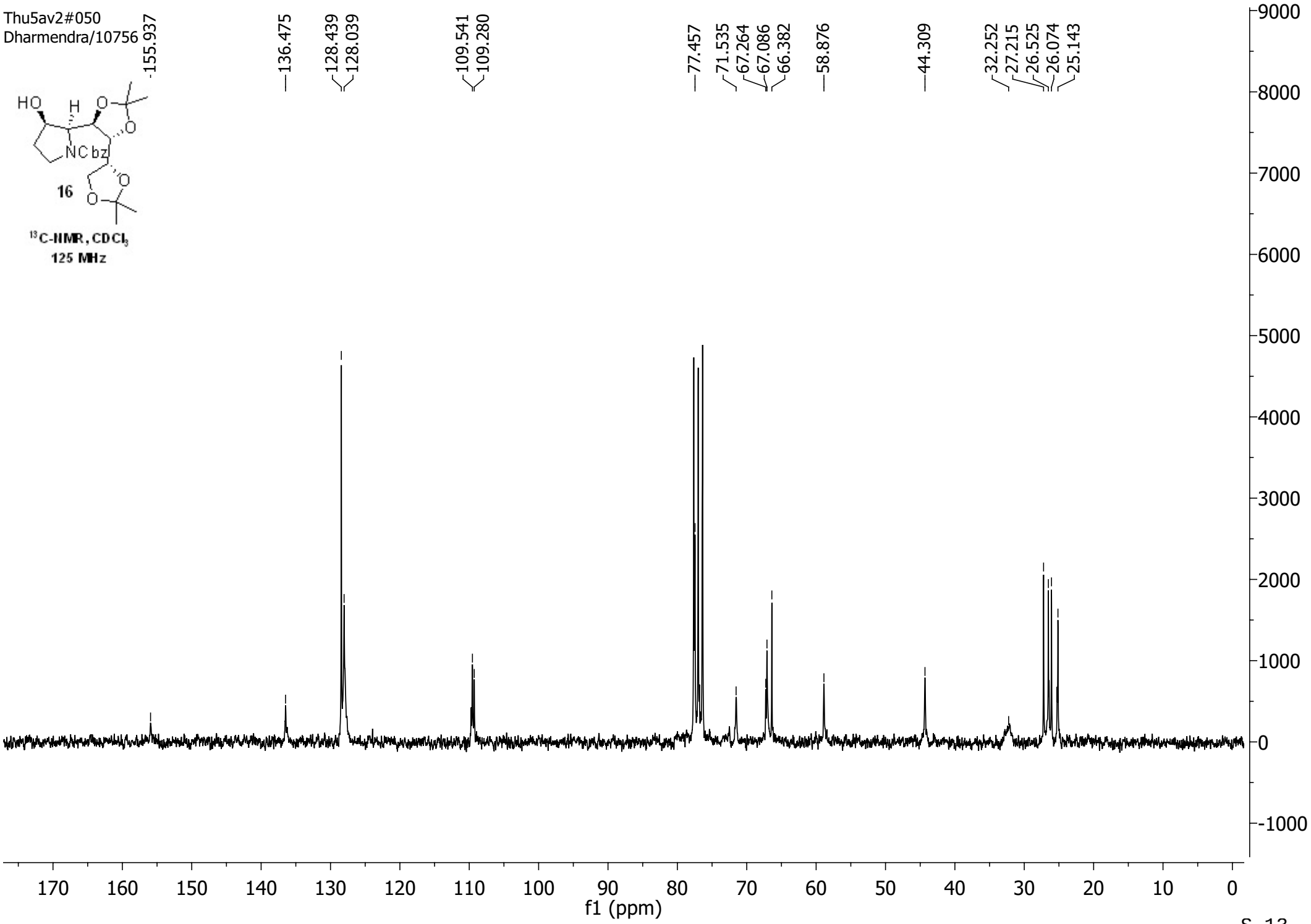

HO
H
O
O
NTs
O
O
15
13C-NMR
CDCl3, 50 MHz
143.911
133.708
129.719
127.866
109.963
109.737
81.631
78.548
76.888
73.210
67.493
60.977
47.073
33.620
27.351
26.764
26.568
25.466
21.578
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


HO
H
O
O
NTs
O
O
15
DEPT-NMR
CDCl3, 50 MHz
129.724
127.869
81.644
78.539
76.885
73.211
67.494
47.086
33.623
27.352
26.772
26.567
25.468
21.591
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
-2000
-2500
-3000
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

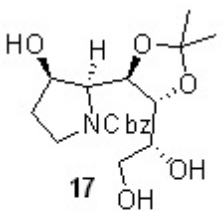
cbz protection Thu5av400#004
Tiwari
1H/cdcl3



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

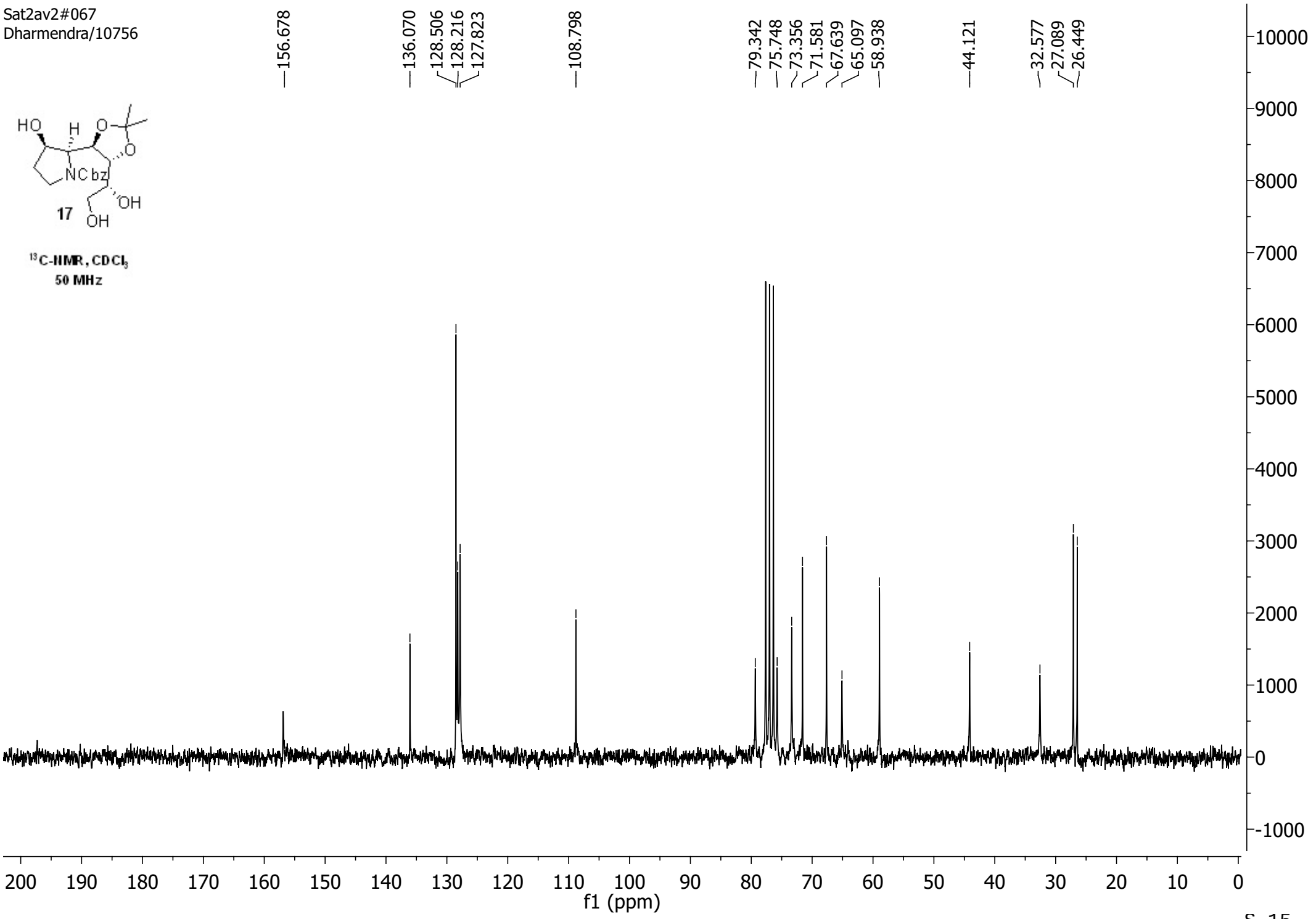

Thu5av2#050
Dharmendra/10756
155.937
136.475
128.439
128.039
109.541
109.280
77.457
71.535
67.264
67.086
66.382
58.876
44.309
32.252
27.215
26.525
26.074
25.143
HO
H
O
O
NCbz
O
O
16
13C-NMR, CDCl3
125 MHz
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Sat2av2#013
Dharmendra/10756

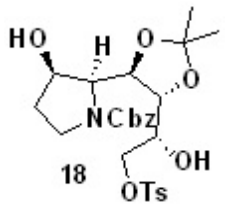


17

^{1}H-NMR, $CDCl_3$
200 MHz


Sat2av2#067
Dharmendra/10756
156.678
136.070
128.506
128.216
127.823
108.798
79.342
75.748
73.356
71.581
67.639
65.097
58.938
44.121
32.577
27.089
26.449
HO
H
O
O
NCbz
OH
OH
17
13C-NMR, CDCl3
50 MHz
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

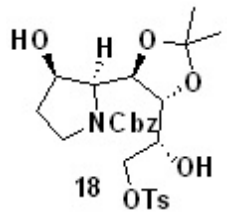
Tue4av2#010
Dharmendra/10756



18

^{1}H-NMR, $CDCl_3$
200 MHz

Sat1av2#025
Dharmendra/10756

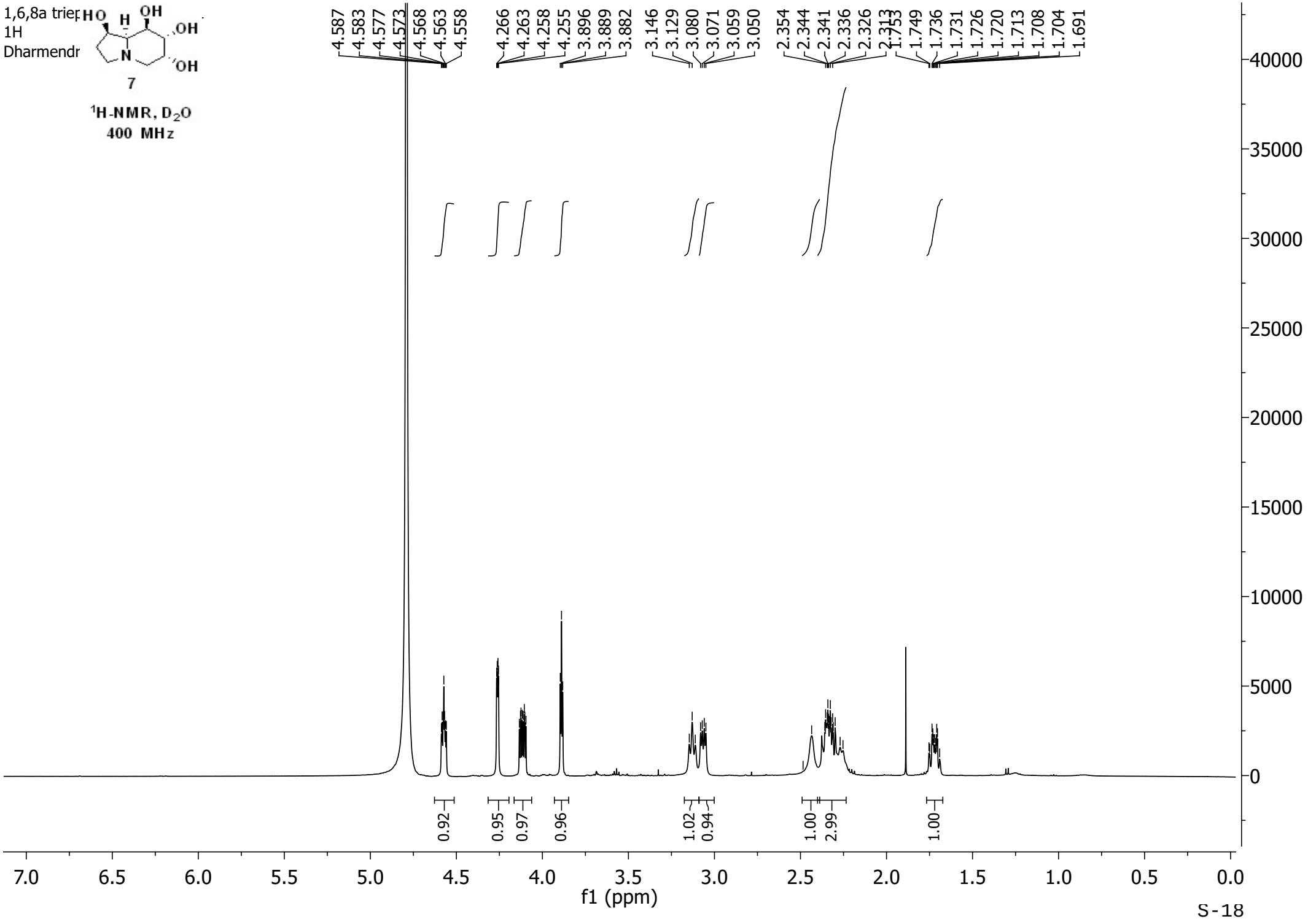


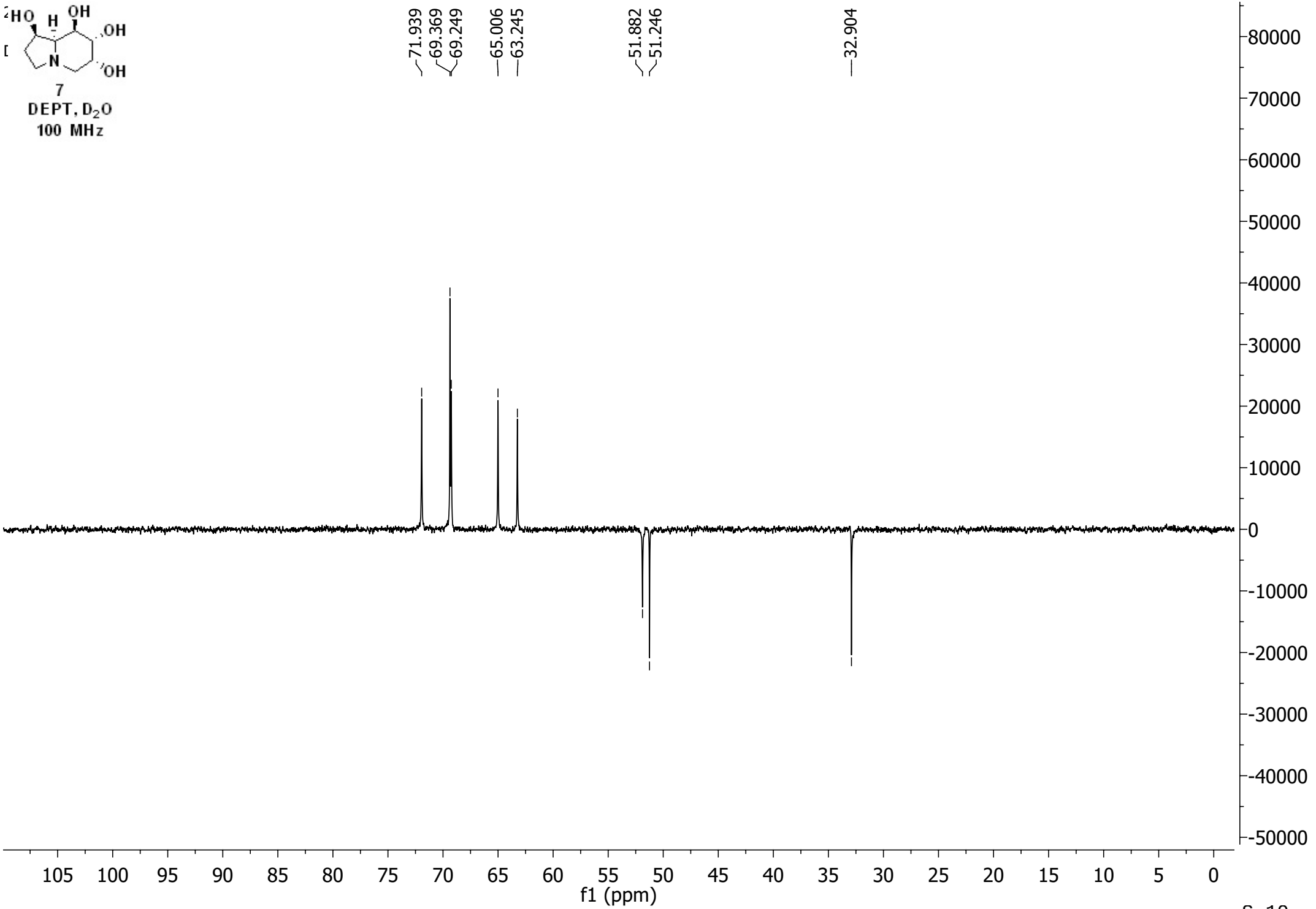
18

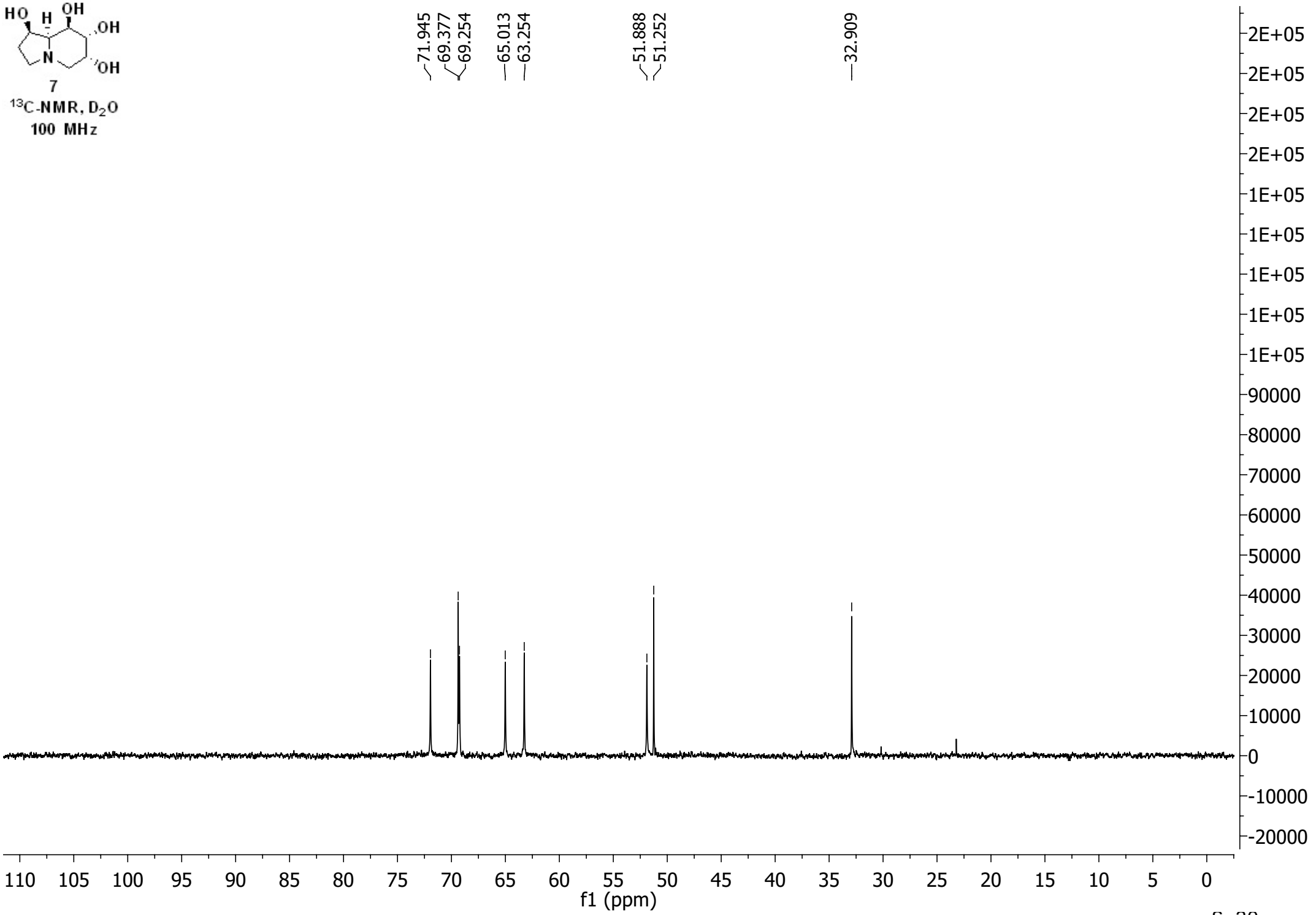
^{13}C-NMR, $CDCl_3$
50 MHz

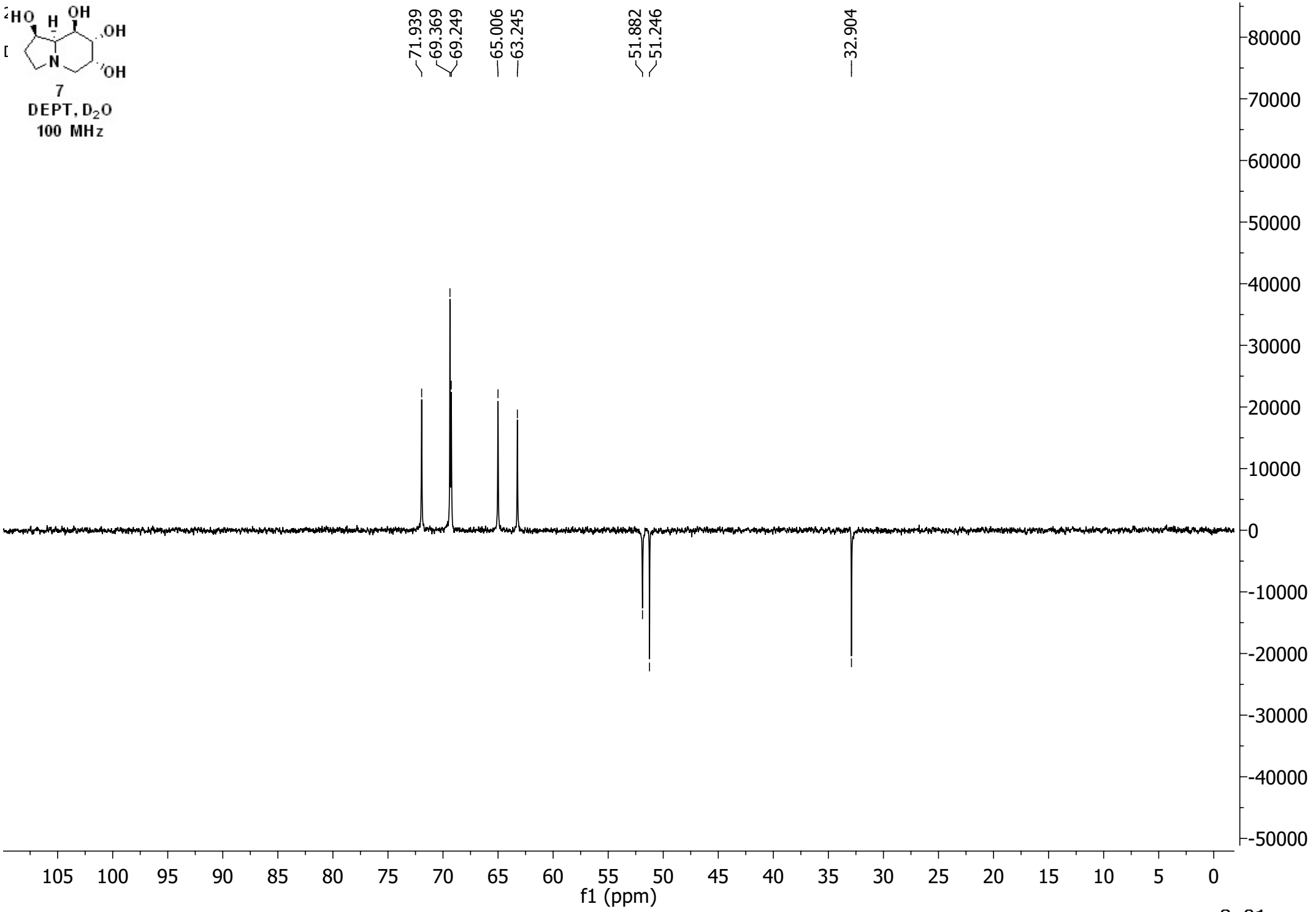
156.864
144.557
136.118
132.824
129.664
128.529
128.055
108.929
80.191
77.192
74.016
71.931
71.358
67.370
58.436
43.709
33.131
27.018
26.465
21.576

f1 (ppm)

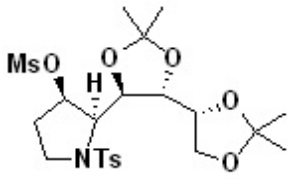

1,6,8a trie
1H
Dharmendr
HO
H
OH
OH
OH
N
7
1H-NMR, D2O
400 MHz
4.587
4.583
4.577
4.573
4.568
4.563
4.558
4.266
4.263
4.258
4.255
3.896
3.889
3.882
3.146
3.129
3.080
3.071
3.059
3.050
2.354
2.344
2.341
2.336
2.326
2.313
1.753
1.749
1.736
1.731
1.726
1.720
1.713
1.708
1.704
1.691
40000
35000
30000
25000
20000
15000
10000
5000
0
0.92
0.95
0.97
0.96
1.02
0.94
1.00
2.99
1.00
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
f1 (ppm)


HO
H
OH
OH
OH
N
7
DEPT, D2O
100 MHz
71.939
69.369
69.249
65.006
63.245
51.882
51.246
32.904
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-30000
-40000
-50000
105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


HO
H
OH
OH
OH
N
7
13C-NMR, D2O
100 MHz
71.945
69.377
69.254
65.013
63.254
51.888
51.252
32.909
2E+05
2E+05
2E+05
2E+05
1E+05
1E+05
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

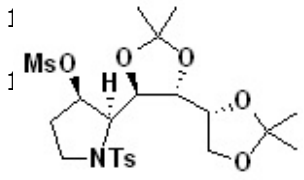

HO
H
OH
OH
OH
N
7
DEPT, D2O
100 MHz
71.939
69.369
69.249
65.006
63.245
51.882
51.246
32.904
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-30000
-40000
-50000
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)

1H, 13C dept @ 400
Deepak
1H/CDCl3



20

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



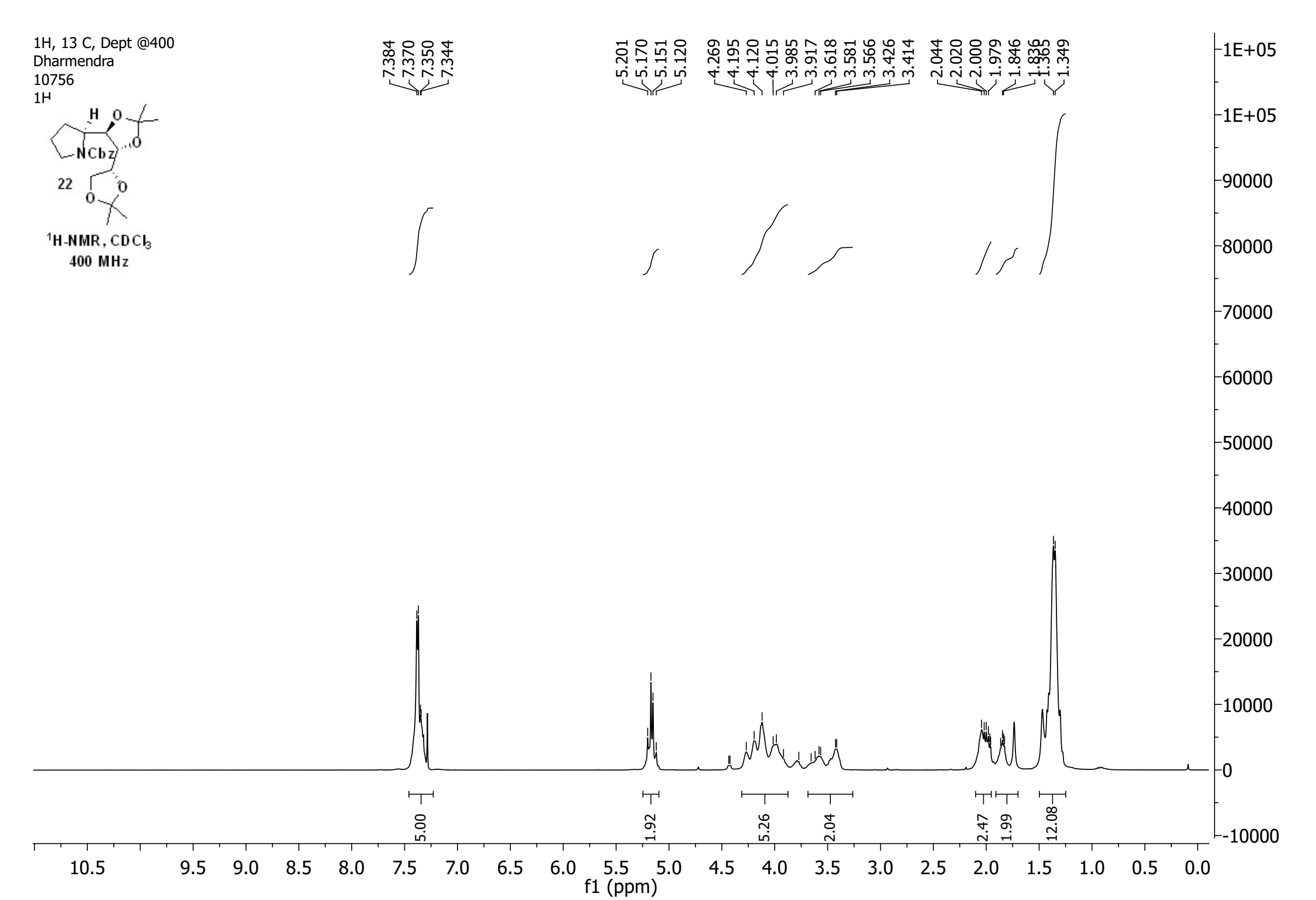
20

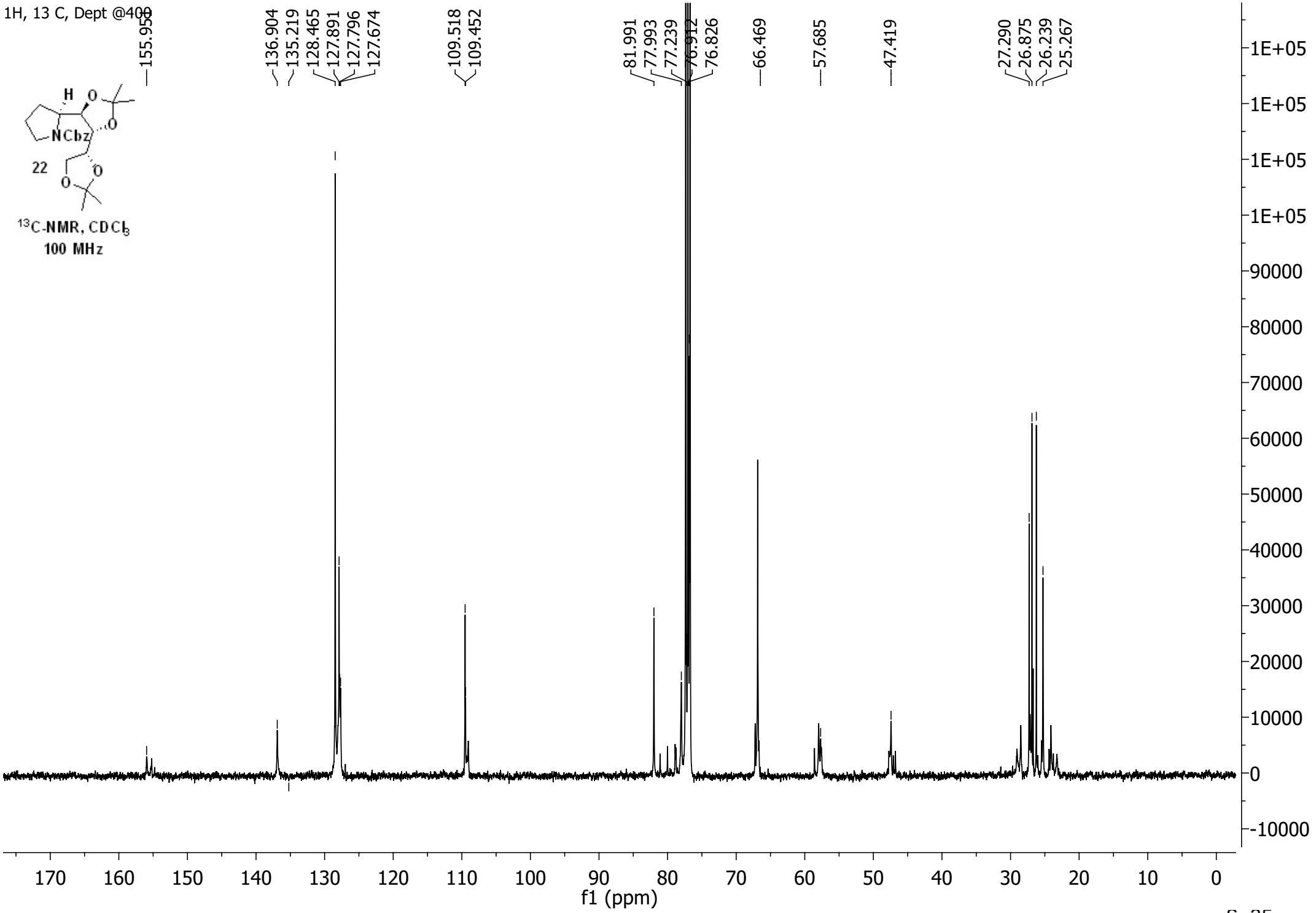
^{13}C-NMR, $CDCl_3$,
100, MHz

144.327
134.515
130.116
127.245
110.184
109.757
77.370
77.242
76.935
76.001
67.795
59.720
45.700
38.383
29.200
27.339
26.380
26.135
25.408
21.556

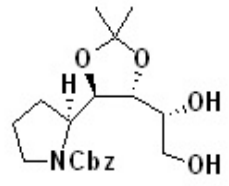
3E+05
3E+05
2E+05
2E+05
2E+05
2E+05
2E+05
1E+05
1E+05
80000
60000
40000
20000
0
-20000

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1H, 13 C, Dept @400
Dharmendra
10756
1H
NCbz
22
1H-NMR, CDCl3
400 MHz
7.384
7.370
7.350
7.344
5.201
5.170
5.151
5.120
4.269
4.195
4.120
4.015
3.985
3.917
3.618
3.581
3.566
3.426
3.414
2.044
2.020
2.000
1.979
1.846
1.836
1.365
1.349
5.00
1.92
5.26
2.04
2.47
1.99
12.08
f1 (ppm)

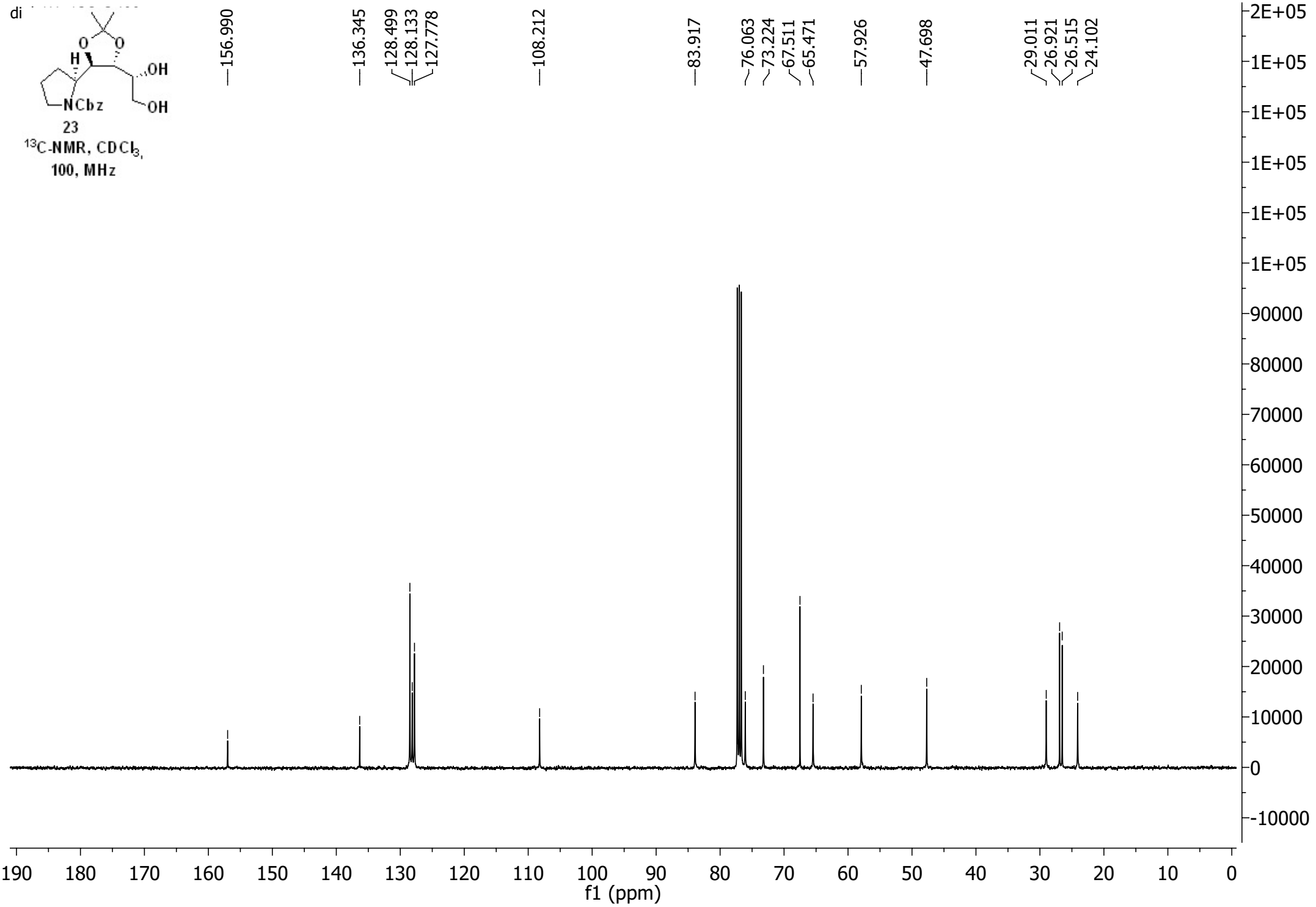

1H, 13 C, Dept @400
155.959
136.904
135.219
128.465
127.891
127.796
127.674
109.518
109.452
81.991
77.993
77.239
76.912
76.826
66.469
57.685
47.419
27.290
26.875
26.239
25.267
H
O
O
NCbz
22
O
O
O
13C-NMR, CDCl3
100 MHz
1E+05
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

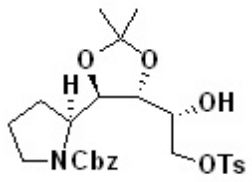
diol-1H, 13C @400
Dharmendra
10756
1l



23

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

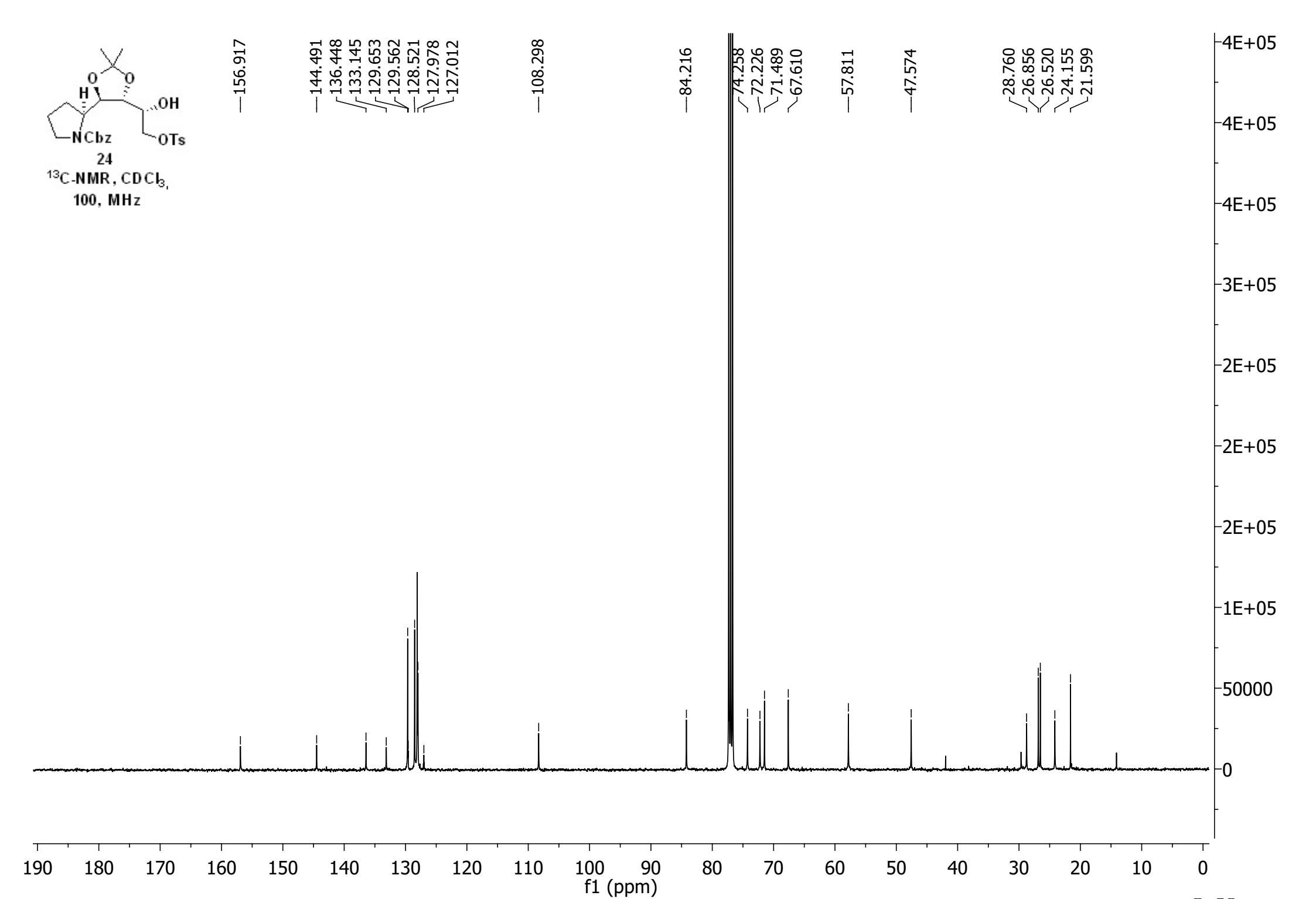

156.990
136.345
128.499
128.133
127.778
108.212
83.917
76.063
73.224
67.511
65.471
57.926
47.698
29.011
26.921
26.515
24.102
OH
OH
NCbz
23
13C-NMR, CDCl3,
100, MHz
2E+05
1E+05
1E+05
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



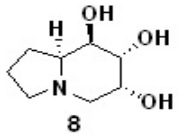
24

^{1}H-NMR, $CDCl_3$,

400, MHz

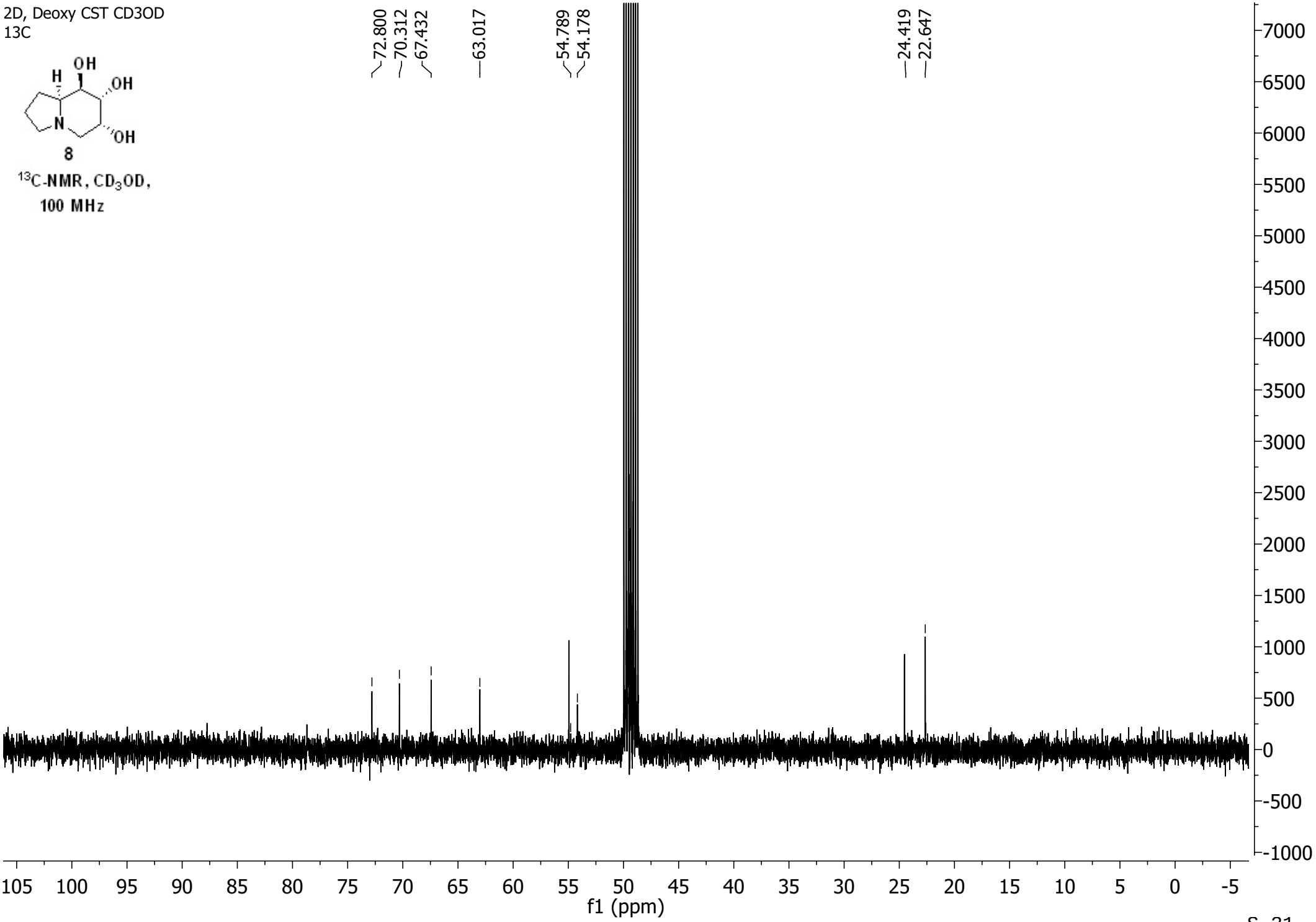

156.917
144.491
136.448
133.145
129.653
129.562
128.521
127.978
127.012
108.298
84.216
74.258
72.226
71.489
67.610
57.811
47.574
28.760
26.856
26.520
24.155
21.599
OH
OTs
NCbz
24
13C-NMR, CDCl3,
100, MHz
4E+05
4E+05
4E+05
3E+05
2E+05
2E+05
2E+05
1E+05
50000
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

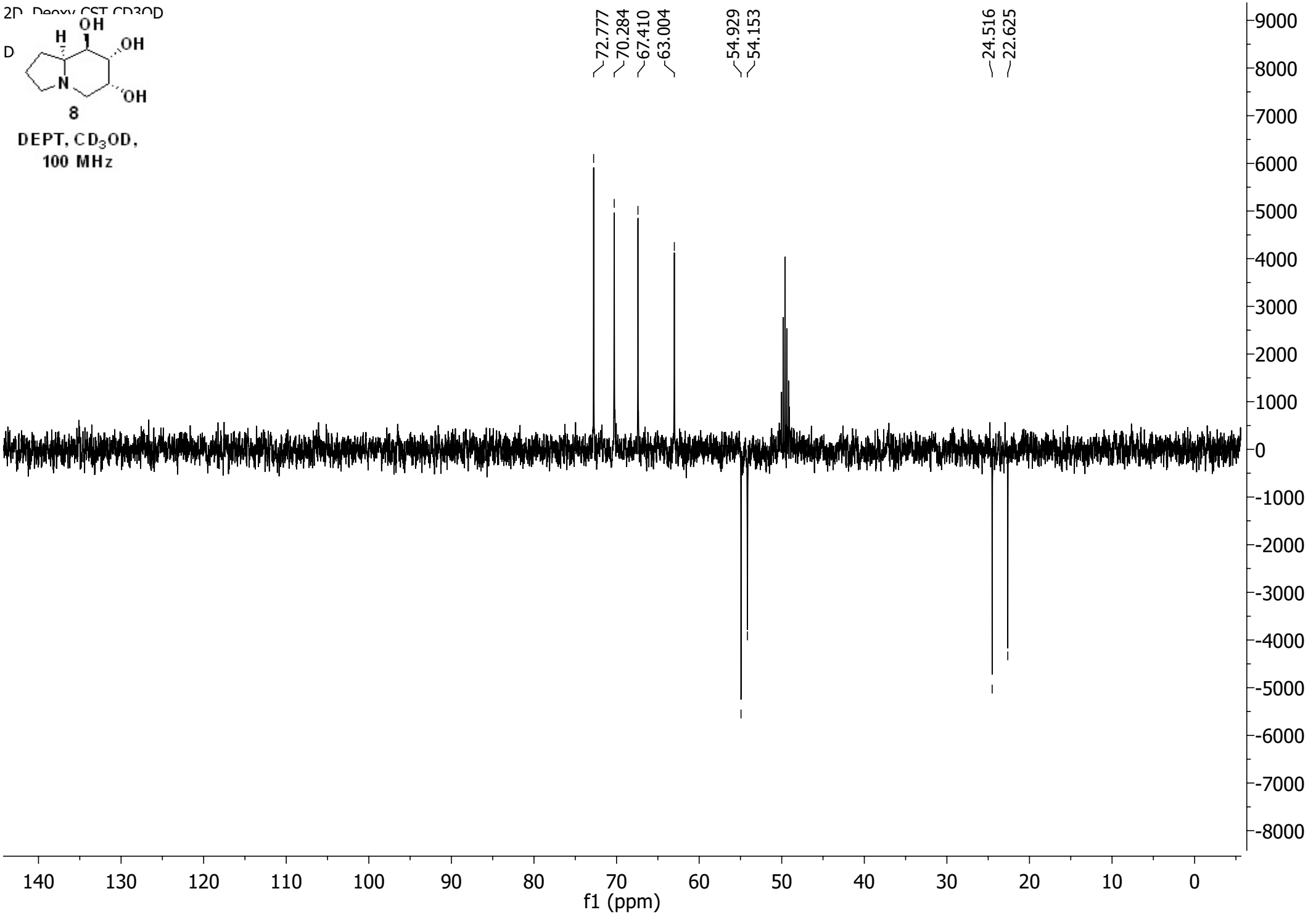
2D, Deoxy CST CD3OD
Dharmendra
1H



8

^{1}H-NMR, CD_3OD,
400 MHz


2D, Deoxy CST CD3OD
13C
OH
H
OH
N
OH
8
^{13}C-NMR, CD_3OD,
100 MHz
72.800
70.312
67.432
63.017
54.789
54.178
24.419
22.647
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5
f1 (ppm)

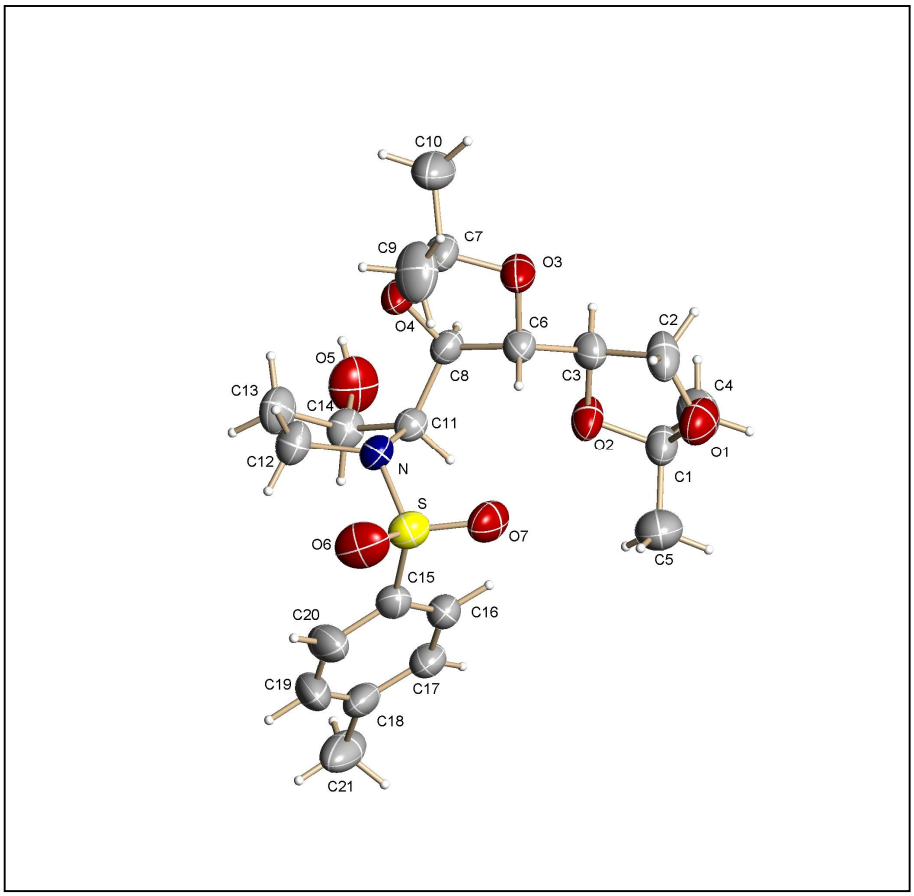

2D_Deoxy CST CD3OD
D
OH
H
OH
N
OH
8
DEPT, CD3OD,
100 MHz
72.777
70.284
67.410
63.004
54.929
54.153
24.516
22.625
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000
-5000
-6000
-7000
-8000
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

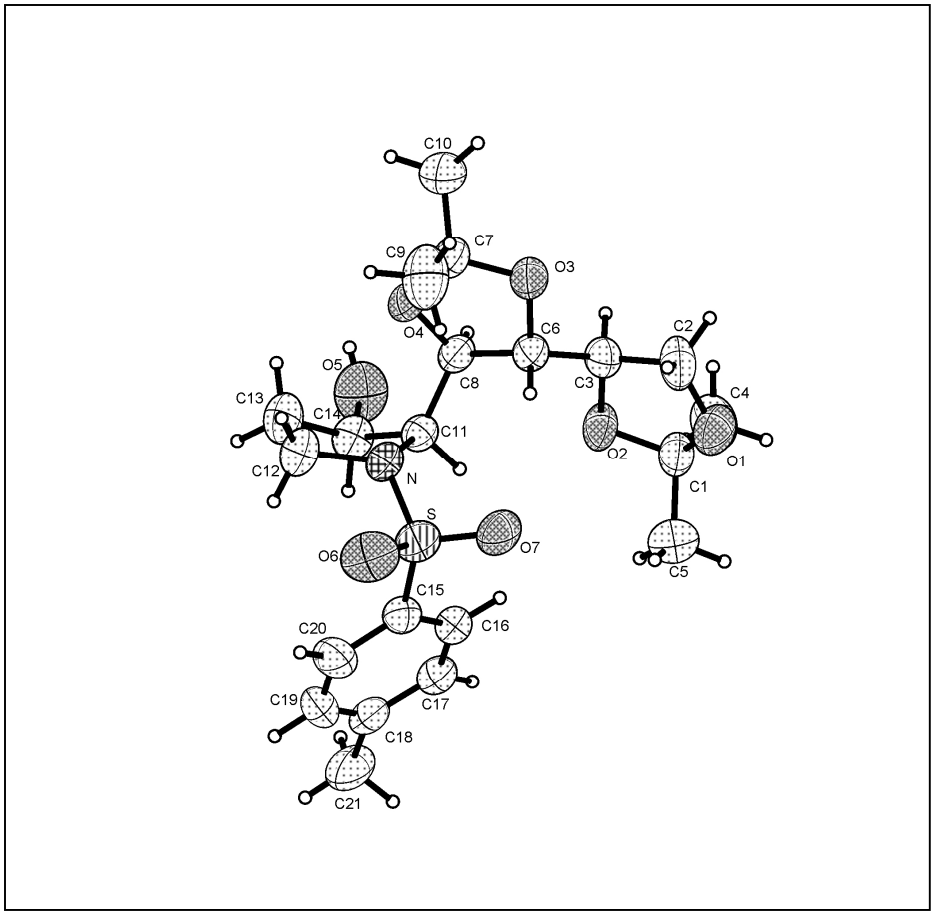
X-ray Crystal Structure Analysis For DTI-357

Crystal Data: Single crystals of the compound were grown by slow evaporation of the solution in dichloromethane and pet-ether. Colourless crystal of approximate size 0.23 x 0.09 x 0.02 mm, was used for data collection on *Bruker SMART APEX* CCD diffractometer using Mo K_α radiation with fine focus tube with 50kV and 30mA. Crystal to detector distance 6.05 cm, 512 x 512 pixels / frame, hemisphere data acquisition. Total frames = 1271, Oscillation / frame -0.3º, exposure / frame = 15.0 sec / frame, maximum detector swing angle = ï 30.0º, beam center = (260.2, 252.5), in plane spot width = 1.24, SAINT integration, θ range = 2.09 to 25.0 º, completeness to θ of 25.0 º is 100.0 %. SADABS correction applied , C21 H31 N O7 S, *M* = 441.53. Crystals belong to Orthorhombic, space group $P2_12_12_1$, a = 6.7501(5), *b* = b = 17.434(1) , *c* = c = 19.510(1) Å, *V* = 2296.1(3) $Å^3$, *Z* = 4, D_c = 1.277 g /cc, μ (MoK_α) = 0.181 mm^{-1}, *T* = 293(2) K, 11648 reflections measured, 4023 unique [I>2σ(I)], R value 0.0448, wR2 = 0.0997. All the data were corrected for Lorentzian, polarisation and absorption effects. SHELX-97 (ShelxTL)[ref] was used for structure solution and full matrix least squares refinement on F^2. Hydrogen atoms were included in the refinement as per the riding model. Largest diff. peak and hole 0.269 and -0.135 e. $Å^{ï\,3}$. Data collection and refinement parameters are listed in table 1.

X-ray analysis revealed the relative conformation of the molecule as C3, C6, c8 ,C11 and C14 as R, R, R, S and R configuration respectively.

Caption to Fig 1.: ORTEP diagram of the molecule. Ellipsoids are drawn at 40% probability.


C10
C7
C9
O3
O4
C6
C2
O5
C8
C3
C4
C13
C14
C11
O2
O1
C12
N
C1
S
O6
O7
C5
C15
C20
C16
C19
C17
C18
C21


C10
C7
C9
O3
O4
C6
C2
O5
C8
C3
C4
C13
C14
C11
O2
O1
C12
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C1
S
O6
O7
C5
C15
C20
C16
C19
C17
C18
C21

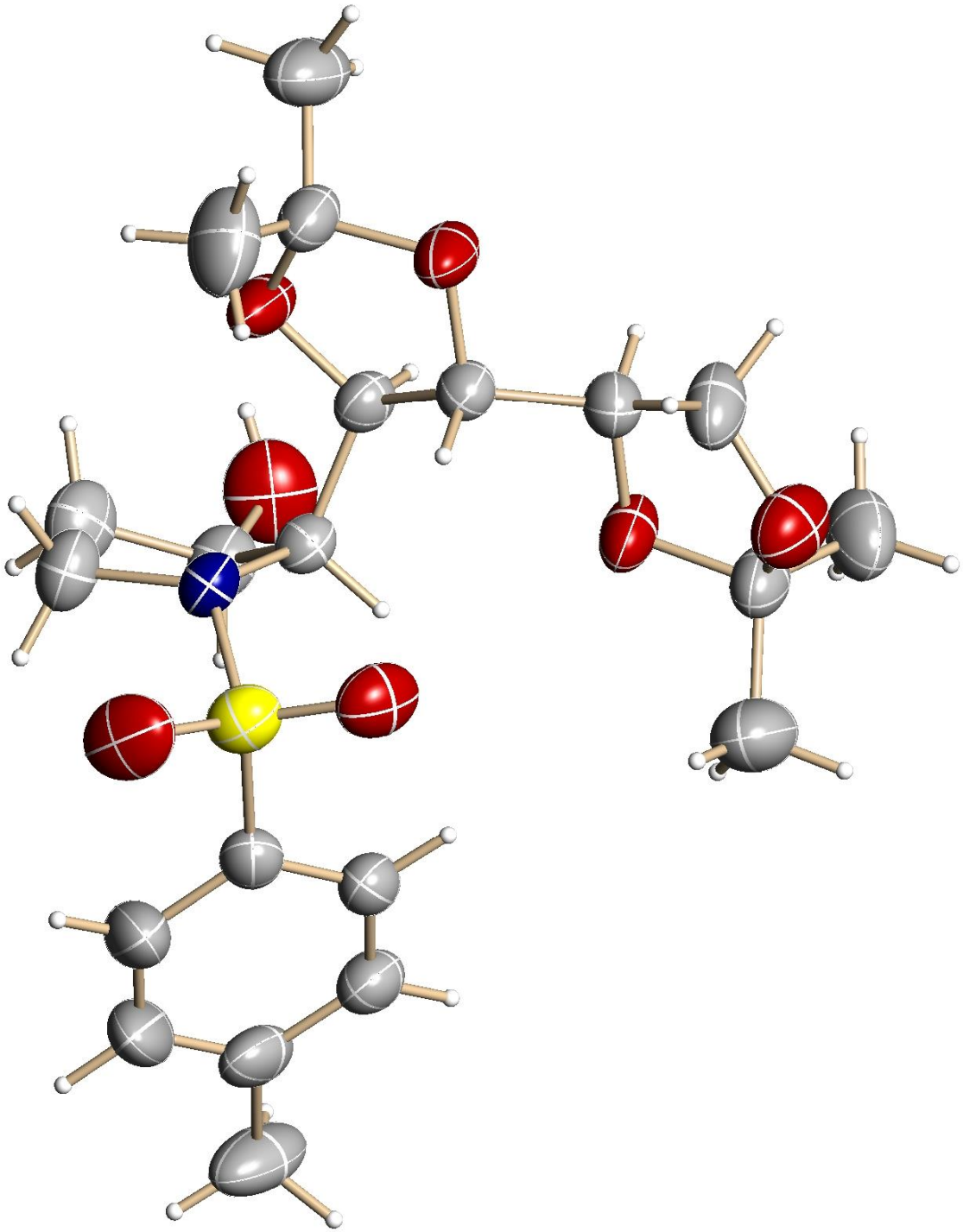

Table 1. Crystal data and structure refinement for dti357.

Empirical formula	C21 H31 N O7 S	
Formula weight	441.53	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.7501(5) Å	α= 90°.
	b = 17.4344(13) Å	β= 90°.
	c = 19.510(1) Å	γ = 90°.
Volume	2296.1(3) $Å^3$	
Z	4	
Density (calculated)	1.277 g/cc	
Absorption coefficient	0.181 mm^{-1}	
F(000)	944	
Crystal size	0.23 x 0.09 x 0.02 mm^3	
Theta range for data collection	2.09 to 25.00°.	
Index ranges	-7<=h<=8, -20<=k<=19, -23<=l<=22	
Reflections collected	11648	
Independent reflections	4023 [R(int) = 0.0344]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9957 and 0.9588	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4023 / 0 / 277	
Goodness-of-fit on F^2	1.024	
Final R indices [I>2sigma(I)]	R1 = 0.0448, wR2 = 0.0997	
R indices (all data)	R1 = 0.0739, wR2 = 0.1110	
Absolute structure parameter	-0.02(10)	
Largest diff. peak and hole	0.269 and -0.135 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for dti357. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S	921(1)	789(1)	1026(1)	64(1)
O(1)	4885(4)	-1188(1)	-837(1)	85(1)
O(2)	5799(3)	-669(1)	168(1)	68(1)
O(3)	2700(3)	-1970(1)	1111(1)	70(1)
O(4)	3031(4)	-1217(1)	2060(1)	72(1)
O(5)	6808(4)	88(2)	2325(2)	103(1)
O(6)	-1015(4)	986(1)	1268(1)	93(1)
O(7)	1171(4)	388(1)	398(1)	77(1)
N	1904(4)	266(1)	1629(1)	53(1)
C(1)	6295(5)	-694(2)	-534(1)	66(1)
C(2)	4201(6)	-1699(2)	-338(2)	82(1)
C(3)	4945(5)	-1383(2)	341(2)	60(1)
C(4)	8375(6)	-995(2)	-622(2)	96(1)
C(5)	6059(9)	100(2)	-824(2)	109(1)
C(6)	3368(4)	-1244(1)	875(1)	52(1)
C(7)	2039(6)	-1884(2)	1797(2)	66(1)
C(8)	4152(5)	-863(1)	1527(1)	52(1)
C(9)	-133(7)	-1756(3)	1826(2)	109(2)
C(10)	2752(7)	-2577(2)	2197(2)	98(1)
C(11)	3960(5)	6(1)	1547(1)	47(1)
C(12)	1583(5)	510(2)	2353(2)	72(1)
C(13)	3512(5)	409(2)	2705(2)	77(1)
C(14)	5015(5)	406(2)	2141(2)	61(1)
C(15)	2332(5)	1634(2)	977(2)	57(1)
C(16)	4078(5)	1643(2)	605(1)	60(1)
C(17)	5312(6)	2266(2)	632(1)	66(1)
C(18)	4856(6)	2896(2)	1027(2)	76(1)
C(19)	3081(8)	2891(2)	1375(2)	99(1)
C(20)	1841(6)	2270(2)	1358(2)	88(1)
C(21)	6290(8)	3565(2)	1078(2)	109(2)

Table 3. Bond lengths [Å] and angles [°] for dti357.

S-O(7)	1.421(2)
S-O(6)	1.431(3)
S-N	1.630(2)
S-C(15)	1.756(3)
O(1)-C(2)	1.399(4)
O(1)-C(1)	1.413(4)
O(2)-C(1)	1.409(3)
O(2)-C(3)	1.413(3)
O(3)-C(7)	1.418(4)
O(3)-C(6)	1.421(3)
O(4)-C(8)	1.427(3)
O(4)-C(7)	1.436(4)
O(5)-C(14)	1.378(4)
O(5)-H(5)	0.8200
N-C(11)	1.469(4)
N-C(12)	1.490(4)
C(1)-C(5)	1.504(5)
C(1)-C(4)	1.509(5)
C(2)-C(3)	1.519(4)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(6)	1.510(4)
C(3)-H(3)	0.9800
C(4)-H(4A)	0.9600
C(4)-H(4B)	0.9600
C(4)-H(4C)	0.9600
C(5)-H(5A)	0.9600
C(5)-H(5B)	0.9600
C(5)-H(5C)	0.9600
C(6)-C(8)	1.530(4)
C(6)-H(6)	0.9800
C(7)-C(9)	1.484(5)
C(7)-C(10)	1.518(5)
C(8)-C(11)	1.521(3)

C(8)-H(8)	0.9800
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(9)-H(9C)	0.9600
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-C(14)	1.529(4)
C(11)-H(11)	0.9800
C(12)-C(13)	1.482(5)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-C(14)	1.497(5)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-H(14)	0.9800
C(15)-C(20)	1.376(4)
C(15)-C(16)	1.384(4)
C(16)-C(17)	1.370(4)
C(16)-H(16)	0.9300
C(17)-C(18)	1.375(4)
C(17)-H(17)	0.9300
C(18)-C(19)	1.377(5)
C(18)-C(21)	1.519(5)
C(19)-C(20)	1.369(5)
C(19)-H(19)	0.9300
C(20)-H(20)	0.9300
C(21)-H(21A)	0.9600
C(21)-H(21B)	0.9600
C(21)-H(21C)	0.9600

O(7)-S-O(6)	120.72(15)
O(7)-S-N	107.44(13)
O(6)-S-N	105.59(14)
O(7)-S-C(15)	107.47(14)
O(6)-S-C(15)	108.15(15)

N-S-C(15)	106.71(13)
C(2)-O(1)-C(1)	108.6(2)
C(1)-O(2)-C(3)	107.6(2)
C(7)-O(3)-C(6)	108.1(2)
C(8)-O(4)-C(7)	109.7(2)
C(14)-O(5)-H(5)	109.5
C(11)-N-C(12)	109.3(2)
C(11)-N-S	118.54(18)
C(12)-N-S	117.7(2)
O(2)-C(1)-O(1)	105.4(2)
O(2)-C(1)-C(5)	108.2(3)
O(1)-C(1)-C(5)	109.3(3)
O(2)-C(1)-C(4)	110.0(3)
O(1)-C(1)-C(4)	111.5(3)
C(5)-C(1)-C(4)	112.1(3)
O(1)-C(2)-C(3)	105.5(2)
O(1)-C(2)-H(2A)	110.6
C(3)-C(2)-H(2A)	110.6
O(1)-C(2)-H(2B)	110.6
C(3)-C(2)-H(2B)	110.6
H(2A)-C(2)-H(2B)	108.8
O(2)-C(3)-C(6)	108.1(2)
O(2)-C(3)-C(2)	104.2(2)
C(6)-C(3)-C(2)	115.3(3)
O(2)-C(3)-H(3)	109.7
C(6)-C(3)-H(3)	109.7
C(2)-C(3)-H(3)	109.7
C(1)-C(4)-H(4A)	109.5
C(1)-C(4)-H(4B)	109.5
H(4A)-C(4)-H(4B)	109.5
C(1)-C(4)-H(4C)	109.5
H(4A)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
C(1)-C(5)-H(5A)	109.5
C(1)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5

C(1)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
O(3)-C(6)-C(3)	107.7(2)
O(3)-C(6)-C(8)	103.1(2)
C(3)-C(6)-C(8)	113.6(2)
O(3)-C(6)-H(6)	110.7
C(3)-C(6)-H(6)	110.7
C(8)-C(6)-H(6)	110.7
O(3)-C(7)-O(4)	106.1(2)
O(3)-C(7)-C(9)	111.3(3)
O(4)-C(7)-C(9)	109.1(3)
O(3)-C(7)-C(10)	107.5(3)
O(4)-C(7)-C(10)	108.2(3)
C(9)-C(7)-C(10)	114.4(3)
O(4)-C(8)-C(11)	111.6(2)
O(4)-C(8)-C(6)	103.5(2)
C(11)-C(8)-C(6)	115.1(2)
O(4)-C(8)-H(8)	108.8
C(11)-C(8)-H(8)	108.8
C(6)-C(8)-H(8)	108.8
C(7)-C(9)-H(9A)	109.5
C(7)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(7)-C(10)-H(10A)	109.5
C(7)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
N-C(11)-C(8)	113.0(2)
N-C(11)-C(14)	102.5(2)
C(8)-C(11)-C(14)	115.7(2)

N-C(11)-H(11)	108.5
C(8)-C(11)-H(11)	108.5
C(14)-C(11)-H(11)	108.5
C(13)-C(12)-N	106.1(3)
C(13)-C(12)-H(12A)	110.5
N-C(12)-H(12A)	110.5
C(13)-C(12)-H(12B)	110.5
N-C(12)-H(12B)	110.5
H(12A)-C(12)-H(12B)	108.7
C(12)-C(13)-C(14)	104.8(3)
C(12)-C(13)-H(13A)	110.8
C(14)-C(13)-H(13A)	110.8
C(12)-C(13)-H(13B)	110.8
C(14)-C(13)-H(13B)	110.8
H(13A)-C(13)-H(13B)	108.9
O(5)-C(14)-C(13)	113.9(3)
O(5)-C(14)-C(11)	115.0(3)
C(13)-C(14)-C(11)	104.1(3)
O(5)-C(14)-H(14)	107.8
C(13)-C(14)-H(14)	107.8
C(11)-C(14)-H(14)	107.8
C(20)-C(15)-C(16)	118.6(3)
C(20)-C(15)-S	121.0(3)
C(16)-C(15)-S	120.0(2)
C(17)-C(16)-C(15)	120.5(3)
C(17)-C(16)-H(16)	119.8
C(15)-C(16)-H(16)	119.8
C(16)-C(17)-C(18)	121.2(3)
C(16)-C(17)-H(17)	119.4
C(18)-C(17)-H(17)	119.4
C(17)-C(18)-C(19)	117.7(3)
C(17)-C(18)-C(21)	120.5(4)
C(19)-C(18)-C(21)	121.8(3)
C(20)-C(19)-C(18)	121.7(3)
C(20)-C(19)-H(19)	119.1
C(18)-C(19)-H(19)	119.1

C(19)-C(20)-C(15)	120.2(3)
C(19)-C(20)-H(20)	119.9
C(15)-C(20)-H(20)	119.9
C(18)-C(21)-H(21A)	109.5
C(18)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5
C(18)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for dti357. 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}
S	55(1)	70(1)	65(1)	10(1)	-6(1)	-2(1)
O(1)	104(2)	92(2)	59(1)	-5(1)	-4(1)	-23(2)
O(2)	80(2)	74(1)	50(1)	-11(1)	19(1)	-23(1)
O(3)	98(2)	54(1)	57(1)	-4(1)	18(1)	-22(1)
O(4)	115(2)	54(1)	47(1)	2(1)	11(1)	-22(1)
O(5)	73(2)	136(2)	99(2)	-14(2)	-29(2)	-2(2)
O(6)	49(1)	111(2)	118(2)	23(2)	0(1)	9(1)
O(7)	96(2)	78(1)	56(1)	3(1)	-20(1)	-15(1)
N	53(2)	60(1)	45(1)	5(1)	4(1)	-4(1)
C(1)	82(3)	64(2)	51(2)	-9(1)	15(2)	-13(2)
C(2)	93(3)	84(2)	67(2)	-25(2)	28(2)	-27(2)
C(3)	65(2)	57(2)	56(2)	-3(1)	14(2)	2(2)
C(4)	85(3)	96(3)	108(3)	-17(2)	37(2)	-8(2)
C(5)	157(4)	77(2)	93(3)	14(2)	17(3)	2(3)
C(6)	57(2)	50(2)	48(2)	0(1)	7(1)	-4(1)
C(7)	87(3)	61(2)	50(2)	2(1)	15(2)	-11(2)
C(8)	60(2)	56(2)	42(2)	5(1)	1(1)	-2(2)
C(9)	84(3)	146(4)	96(3)	-21(3)	29(2)	-13(3)
C(10)	155(4)	62(2)	78(2)	6(2)	3(3)	-10(2)
C(11)	46(2)	50(2)	44(2)	3(1)	0(1)	-6(2)
C(12)	71(2)	91(2)	53(2)	-1(2)	10(2)	-2(2)
C(13)	85(3)	92(2)	56(2)	-12(2)	-6(2)	-8(2)
C(14)	52(2)	71(2)	59(2)	-3(2)	-4(2)	-11(2)
C(15)	62(2)	55(2)	52(2)	5(1)	2(2)	10(2)
C(16)	78(2)	54(2)	48(2)	3(1)	8(2)	3(2)
C(17)	85(3)	61(2)	53(2)	8(2)	8(2)	-7(2)
C(18)	122(3)	61(2)	46(2)	12(2)	2(2)	-9(2)
C(19)	171(5)	51(2)	75(3)	-8(2)	38(3)	8(2)
C(20)	113(3)	60(2)	90(3)	7(2)	40(2)	17(2)
C(21)	182(5)	75(2)	69(2)	9(2)	-7(3)	-52(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for dti357.

	x	y	z	U(eq)
H(5)	6615	-291	2565	154
H(2A)	4723	-2209	-418	98
H(2B)	2765	-1723	-341	98
H(3)	5966	-1724	528	71
H(4A)	9292	-653	-403	144
H(4B)	8681	-1029	-1101	144
H(4C)	8475	-1494	-417	144
H(5A)	4689	247	-805	164
H(5B)	6498	104	-1292	164
H(5C)	6837	455	-562	164
H(6)	2267	-948	682	62
H(8)	5550	-1000	1584	63
H(9A)	-494	-1362	1506	163
H(9B)	-502	-1601	2280	163
H(9C)	-808	-2223	1710	163
H(10A)	2258	-3036	1986	147
H(10B)	2275	-2547	2660	147
H(10C)	4174	-2587	2198	147
H(11)	4477	214	1115	56
H(12A)	1166	1042	2372	86
H(12B)	573	195	2568	86
H(13A)	3542	-70	2956	93
H(13B)	3756	828	3021	93
H(14)	5256	939	2006	73
H(16)	4416	1224	335	72
H(17)	6480	2264	379	79
H(19)	2717	3320	1628	119
H(20)	662	2278	1604	105
H(21A)	7449	3407	1327	163
H(21B)	5664	3982	1314	163

H(21C)	6665	3727	626	163

Table 6. Torsion angles [°] for dti357.

O(7)-S-N-C(11)	53.2(2)
O(6)-S-N-C(11)	-176.8(2)
C(15)-S-N-C(11)	-61.8(2)
O(7)-S-N-C(12)	-171.6(2)
O(6)-S-N-C(12)	-41.5(3)
C(15)-S-N-C(12)	73.4(2)
C(3)-O(2)-C(1)-O(1)	30.6(3)
C(3)-O(2)-C(1)-C(5)	147.5(3)
C(3)-O(2)-C(1)-C(4)	-89.8(3)
C(2)-O(1)-C(1)-O(2)	-26.2(3)
C(2)-O(1)-C(1)-C(5)	-142.4(3)
C(2)-O(1)-C(1)-C(4)	93.2(3)
C(1)-O(1)-C(2)-C(3)	12.0(4)
C(1)-O(2)-C(3)-C(6)	-145.7(3)
C(1)-O(2)-C(3)-C(2)	-22.6(3)
O(1)-C(2)-C(3)-O(2)	6.5(3)
O(1)-C(2)-C(3)-C(6)	124.8(3)
C(7)-O(3)-C(6)-C(3)	151.5(3)
C(7)-O(3)-C(6)-C(8)	31.1(3)
O(2)-C(3)-C(6)-O(3)	-172.5(2)
C(2)-C(3)-C(6)-O(3)	71.4(3)
O(2)-C(3)-C(6)-C(8)	-59.0(3)
C(2)-C(3)-C(6)-C(8)	-175.1(2)
C(6)-O(3)-C(7)-O(4)	-23.3(4)
C(6)-O(3)-C(7)-C(9)	95.1(3)
C(6)-O(3)-C(7)-C(10)	-138.9(3)
C(8)-O(4)-C(7)-O(3)	4.9(3)
C(8)-O(4)-C(7)-C(9)	-115.0(3)
C(8)-O(4)-C(7)-C(10)	120.0(3)
C(7)-O(4)-C(8)-C(11)	138.0(3)
C(7)-O(4)-C(8)-C(6)	13.7(3)
O(3)-C(6)-C(8)-O(4)	-27.0(3)
C(3)-C(6)-C(8)-O(4)	-143.2(2)
O(3)-C(6)-C(8)-C(11)	-149.1(3)

C(3)-C(6)-C(8)-C(11)	94.7(3)
C(12)-N-C(11)-C(8)	102.7(3)
S-N-C(11)-C(8)	-118.6(2)
C(12)-N-C(11)-C(14)	-22.5(3)
S-N-C(11)-C(14)	116.3(2)
O(4)-C(8)-C(11)-N	-46.3(3)
C(6)-C(8)-C(11)-N	71.4(3)
O(4)-C(8)-C(11)-C(14)	71.4(4)
C(6)-C(8)-C(11)-C(14)	-171.0(3)
C(11)-N-C(12)-C(13)	1.4(3)
S-N-C(12)-C(13)	-137.7(2)
N-C(12)-C(13)-C(14)	21.0(4)
C(12)-C(13)-C(14)-O(5)	-161.0(3)
C(12)-C(13)-C(14)-C(11)	-34.9(3)
N-C(11)-C(14)-O(5)	160.4(3)
C(8)-C(11)-C(14)-O(5)	37.0(4)
N-C(11)-C(14)-C(13)	35.0(3)
C(8)-C(11)-C(14)-C(13)	-88.3(3)
O(7)-S-C(15)-C(20)	154.3(3)
O(6)-S-C(15)-C(20)	22.5(3)
N-S-C(15)-C(20)	-90.7(3)
O(7)-S-C(15)-C(16)	-32.3(3)
O(6)-S-C(15)-C(16)	-164.1(2)
N-S-C(15)-C(16)	82.7(3)
C(20)-C(15)-C(16)-C(17)	1.7(4)
S-C(15)-C(16)-C(17)	-171.8(2)
C(15)-C(16)-C(17)-C(18)	0.0(4)
C(16)-C(17)-C(18)-C(19)	-2.3(5)
C(16)-C(17)-C(18)-C(21)	177.0(3)
C(17)-C(18)-C(19)-C(20)	2.9(5)
C(21)-C(18)-C(19)-C(20)	-176.4(3)
C(18)-C(19)-C(20)-C(15)	-1.2(6)
C(16)-C(15)-C(20)-C(19)	-1.2(5)
S-C(15)-C(20)-C(19)	172.3(3)