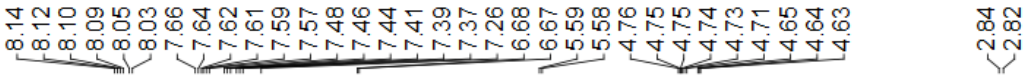
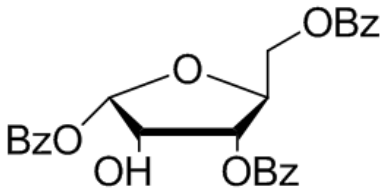
Supporting Information

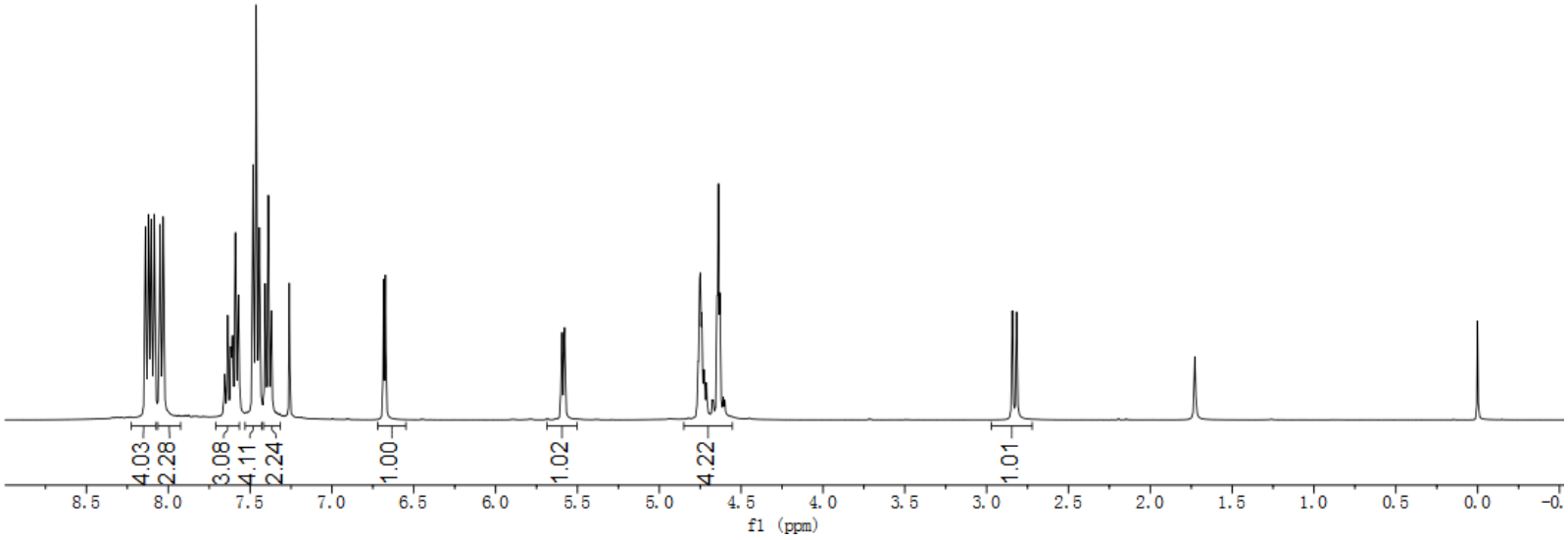
Total synthesis of 2'-*O*-methyl-β-L-arabinosyluridine and reassignment the nucleoside from *Penicillium sp.* as 2'-*O*-methyl-β-L-uridine

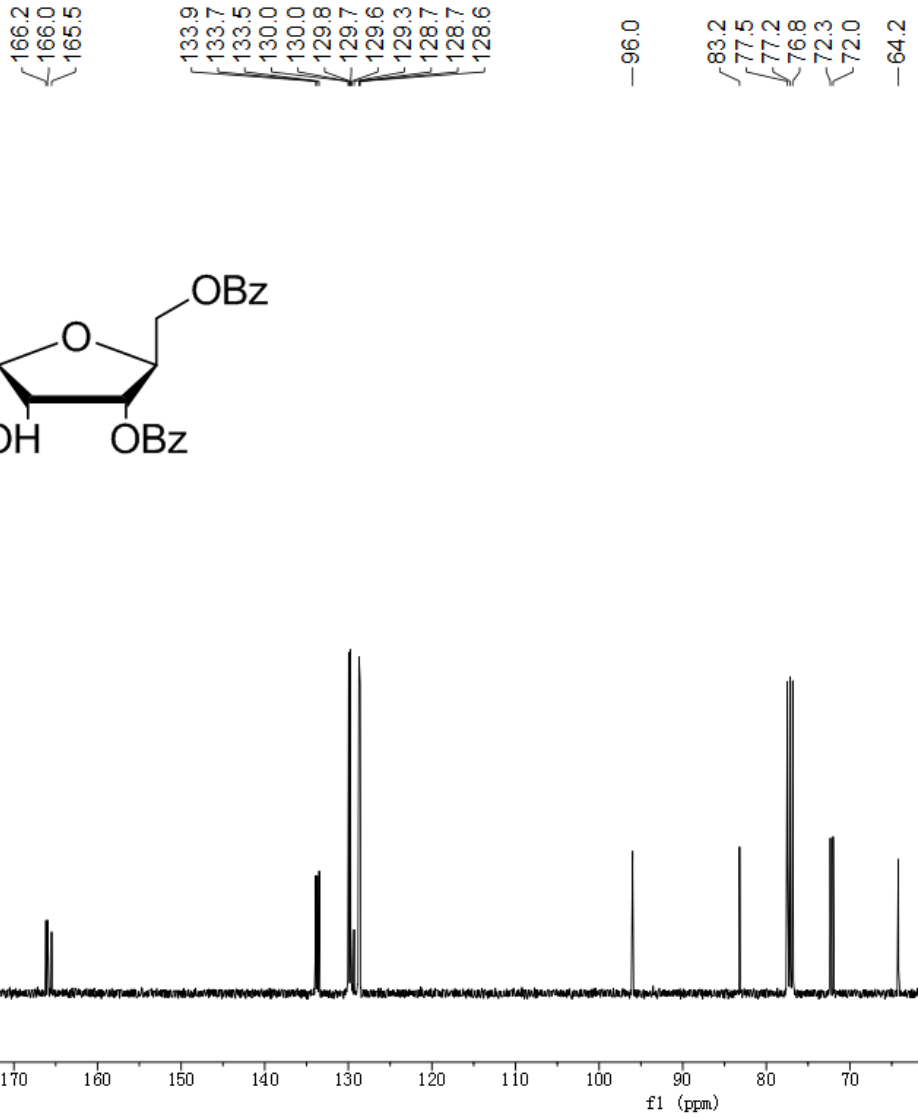
Chunyang Shen[1], Haixin Ding[1], Xueping Tao[1], Jiang Bai[1], Ban-Peng Cao*[1], Yi-Yuan Peng[2] and Qiang Xiao*[1]

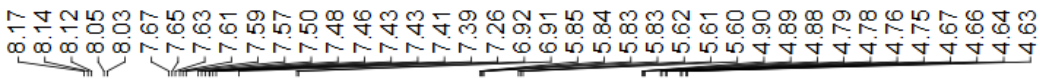
[1]Jiangxi Key Laboratory of Organic Chemistry, Jiangxi Science & Technology Normal University, 605 Fenglin Road, Nanchang, Jiangxi province, PR China 330013 and [2]Key Laboratory of Small Functional Organic Molecule, Ministry of Education, Jiangxi's Key Laboratory of Green Chemistry and College of Chemistry & Chemical Engineering, Jiangxi Normal University Nanchang, Jiangxi 330022

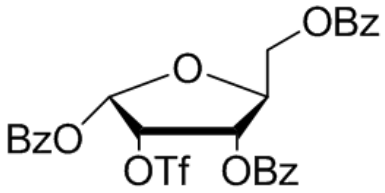

8.14
8.12
8.10
8.09
8.05
8.03
7.66
7.64
7.62
7.61
7.59
7.57
7.48
7.46
7.44
7.41
7.39
7.37
7.26
6.68
6.67
5.59
5.58
4.76
4.75
4.75
4.74
4.73
4.71
4.65
4.64
4.63
2.84
2.82

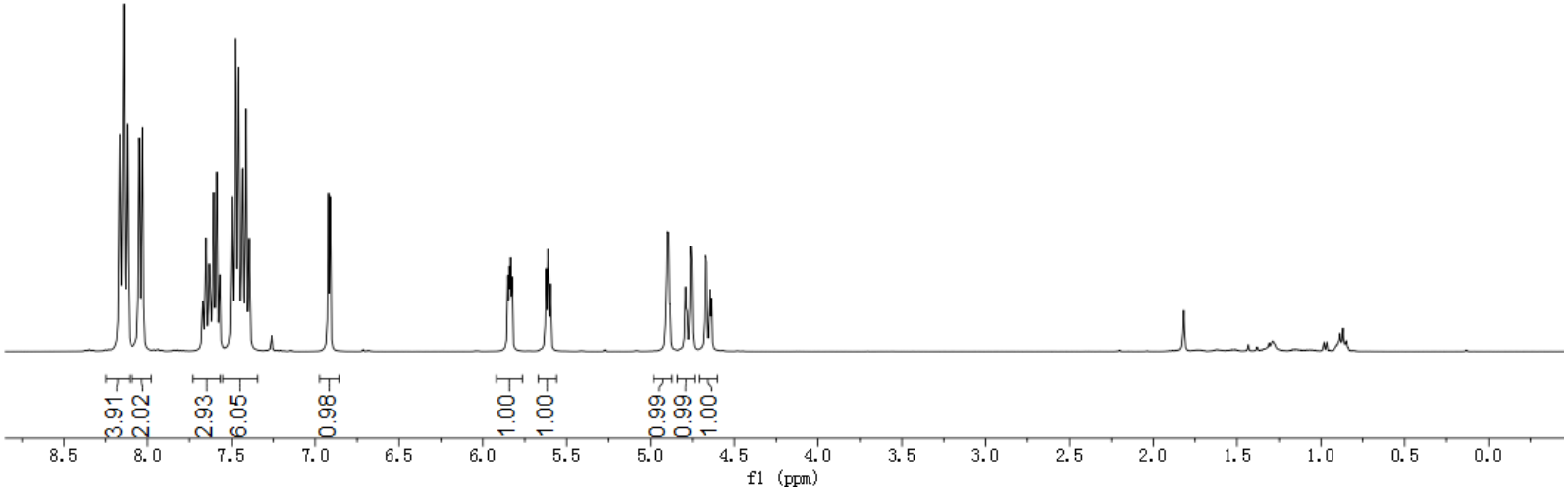

OBz
O
BzO
OH
OBz

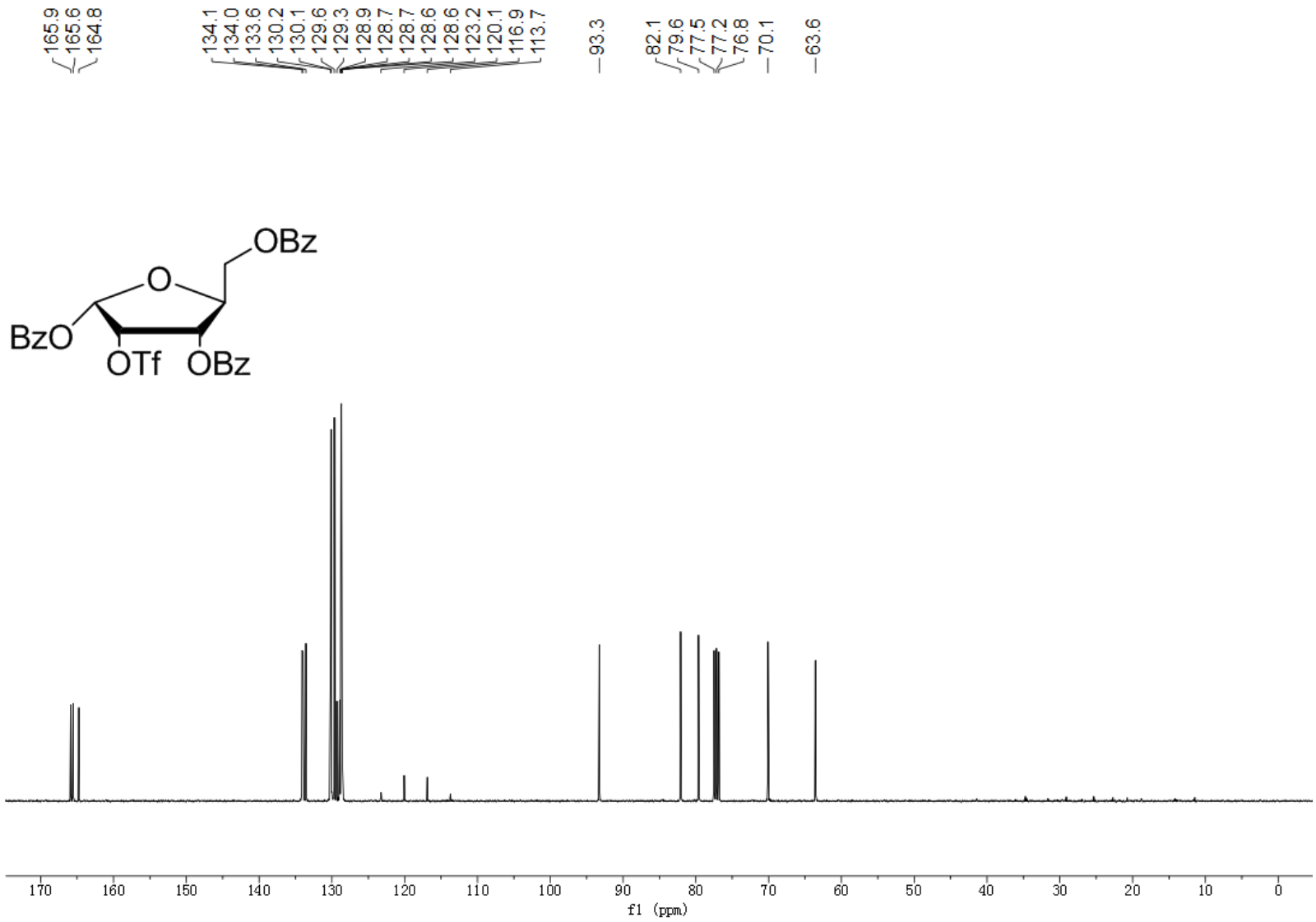

4.03
2.28
3.08
4.11
2.24
1.00
1.02
4.22
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

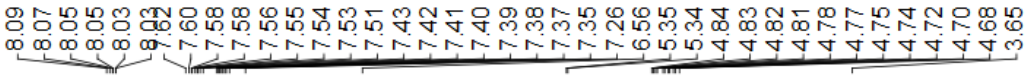

166.2
166.0
165.5
133.9
133.7
133.5
130.0
130.0
129.8
129.7
129.6
129.3
128.7
128.7
128.6
96.0
83.2
77.5
77.2
76.8
72.3
72.0
64.2
OBz
O
BzO
OH
OBz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

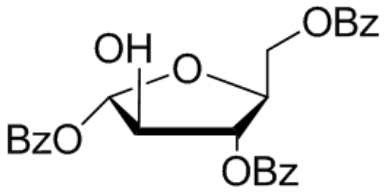

8.17
8.14
8.12
8.05
8.03
7.67
7.65
7.63
7.61
7.59
7.57
7.50
7.48
7.46
7.43
7.43
7.41
7.39
7.26
6.92
6.91
5.85
5.84
5.83
5.83
5.62
5.61
5.60
4.90
4.89
4.88
4.79
4.78
4.76
4.75
4.67
4.66
4.64
4.63

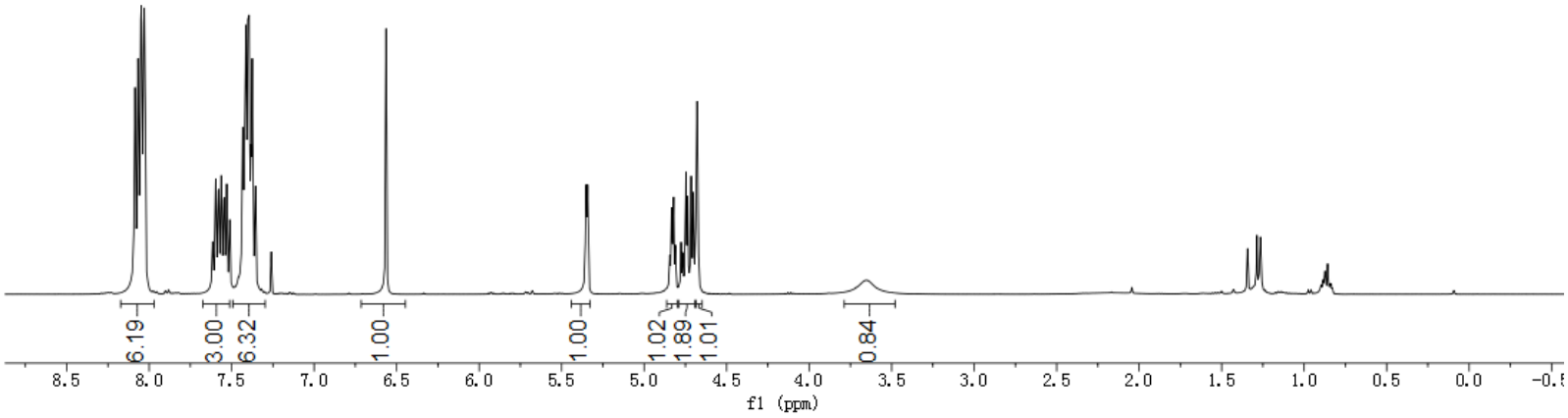

OBz
O
BzO
OTf
OBz

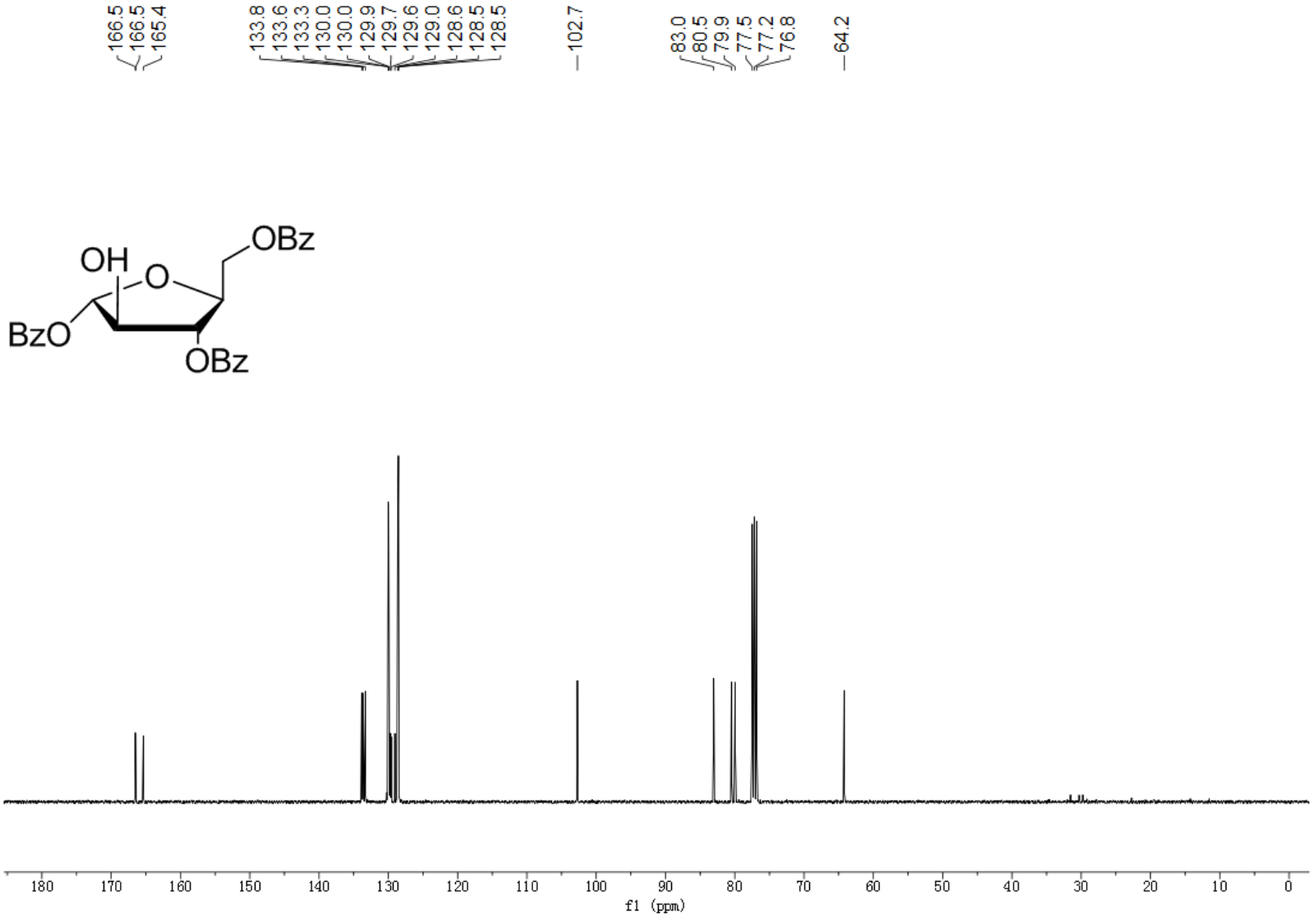

3.91
2.02
2.93
6.05
0.98
1.00
1.00
0.99
0.99
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

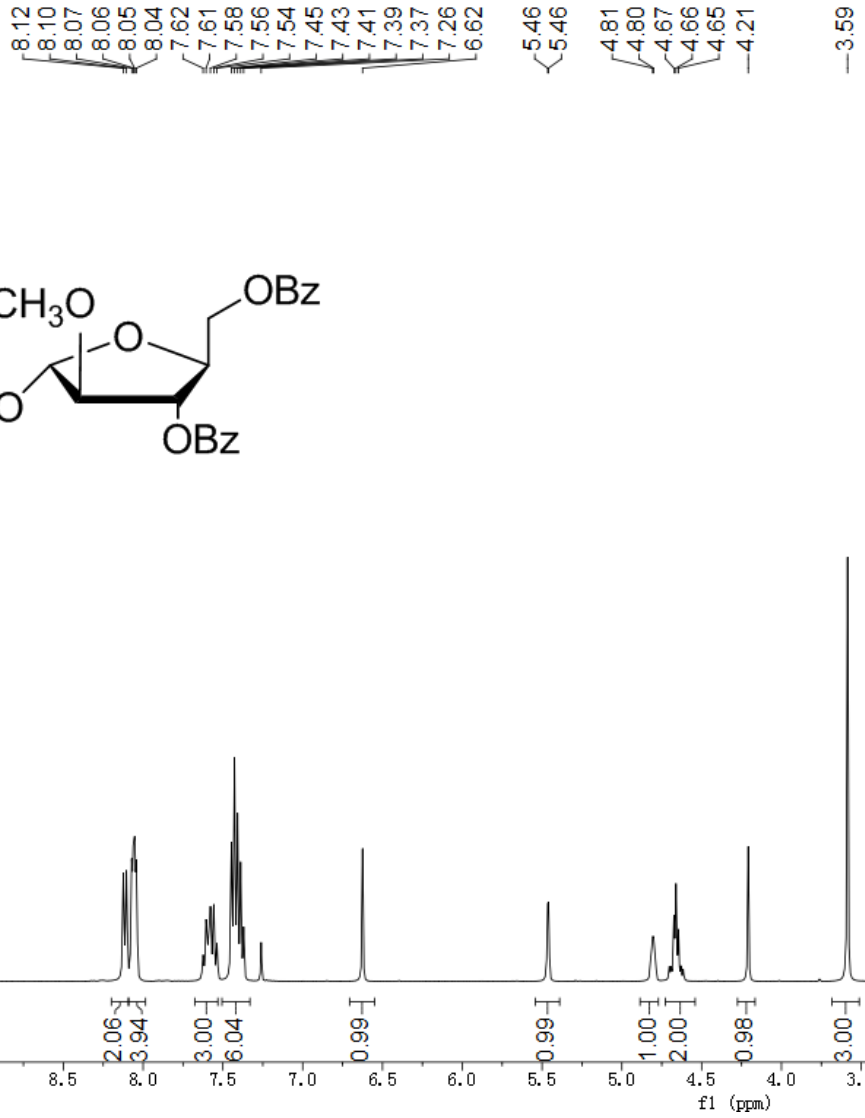

165.9
165.6
164.8
134.1
134.0
133.6
130.2
130.1
129.6
129.3
128.9
128.7
128.7
128.6
128.6
123.2
120.1
116.9
113.7
93.3
82.1
79.6
77.5
77.2
76.8
70.1
63.6
OBz
O
BzO
OTf
OBz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

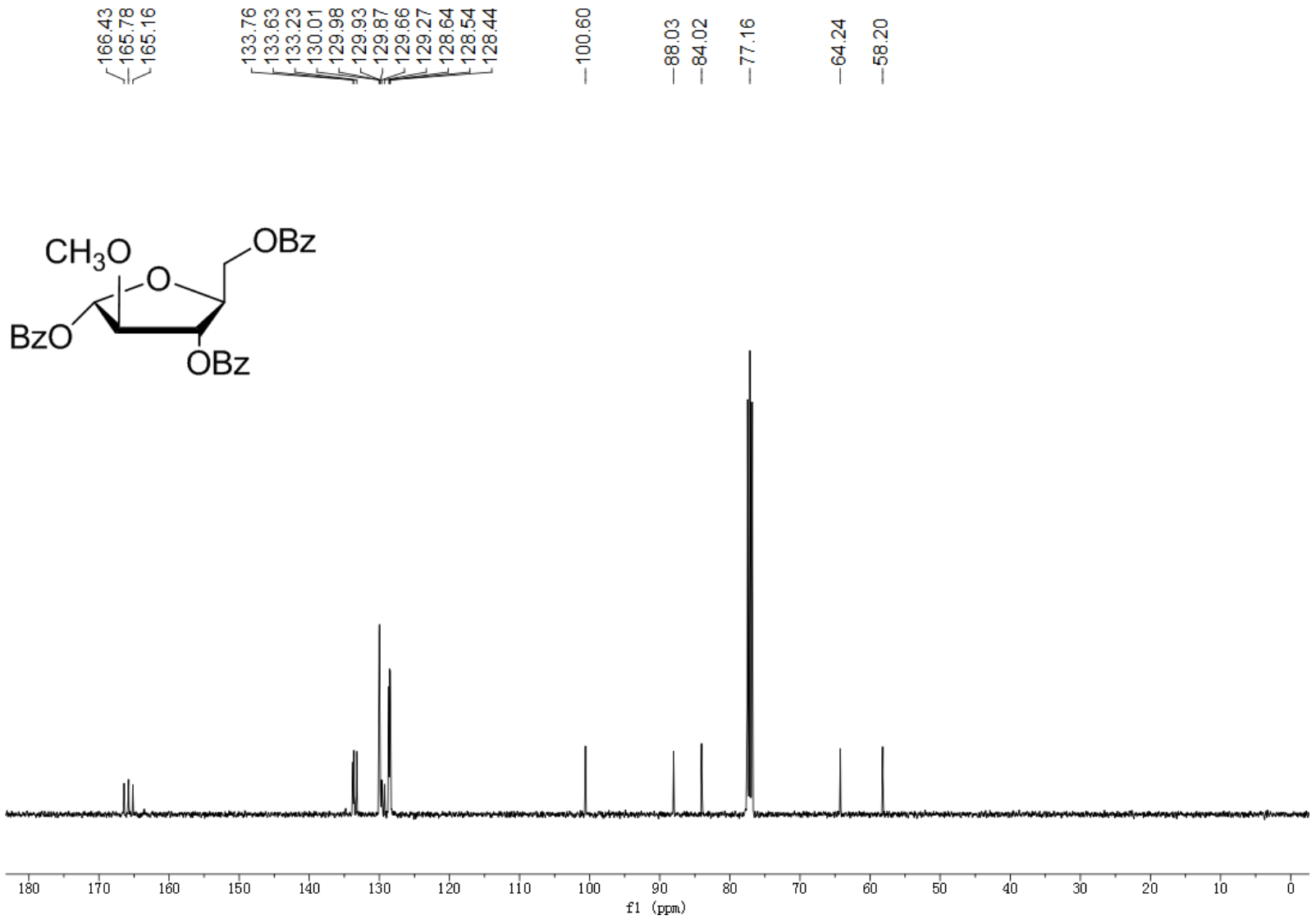

8.09
8.07
8.05
8.05
8.03
8.02
7.60
7.58
7.58
7.56
7.55
7.54
7.53
7.51
7.43
7.42
7.41
7.40
7.39
7.38
7.37
7.35
7.26
6.56
5.35
5.34
4.84
4.83
4.82
4.81
4.78
4.77
4.75
4.74
4.72
4.70
4.68
3.65

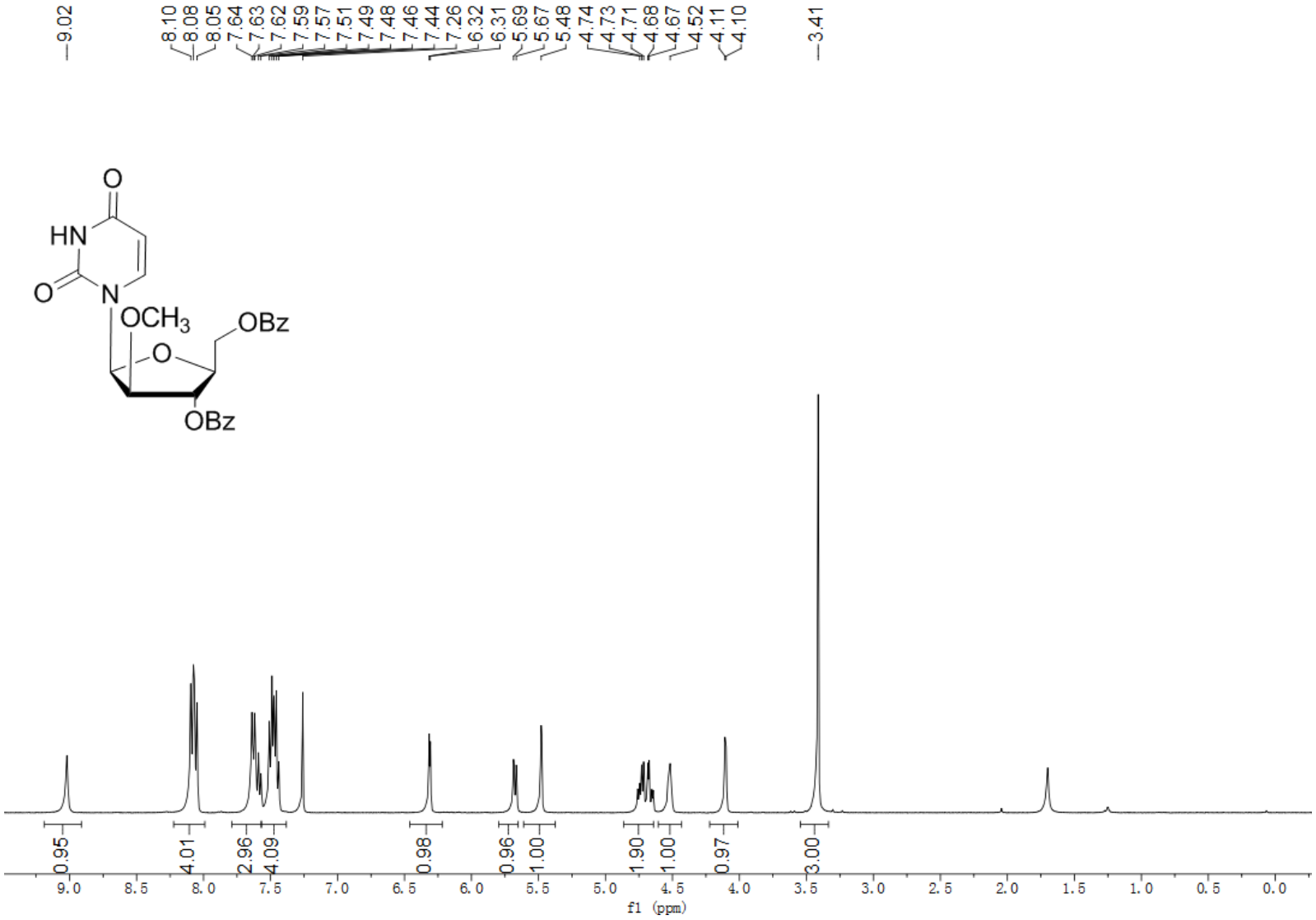

OH
O
OBz
BzO
OBz

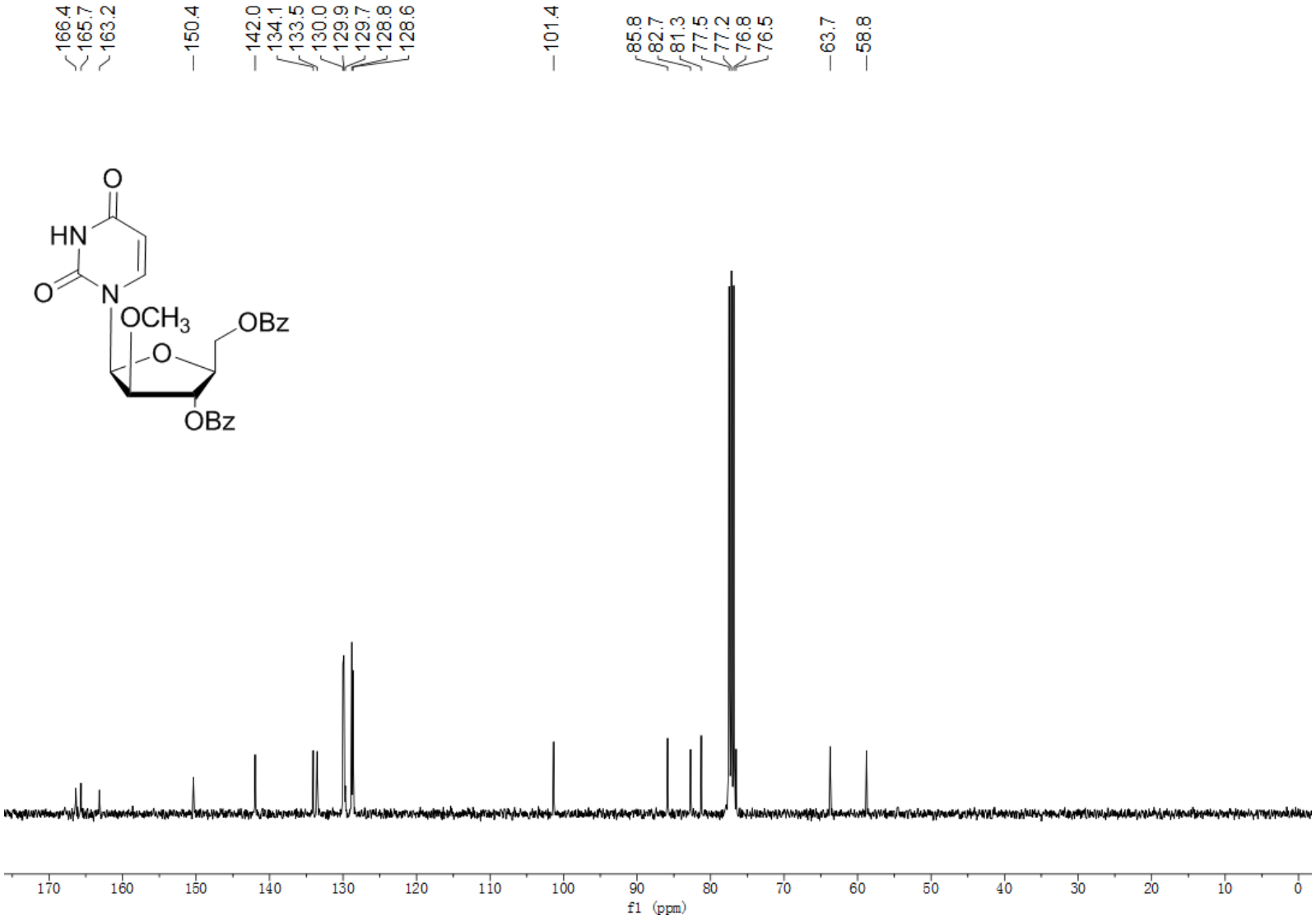

6.19
3.00
6.32
1.00
1.00
1.02
1.89
1.01
0.84
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

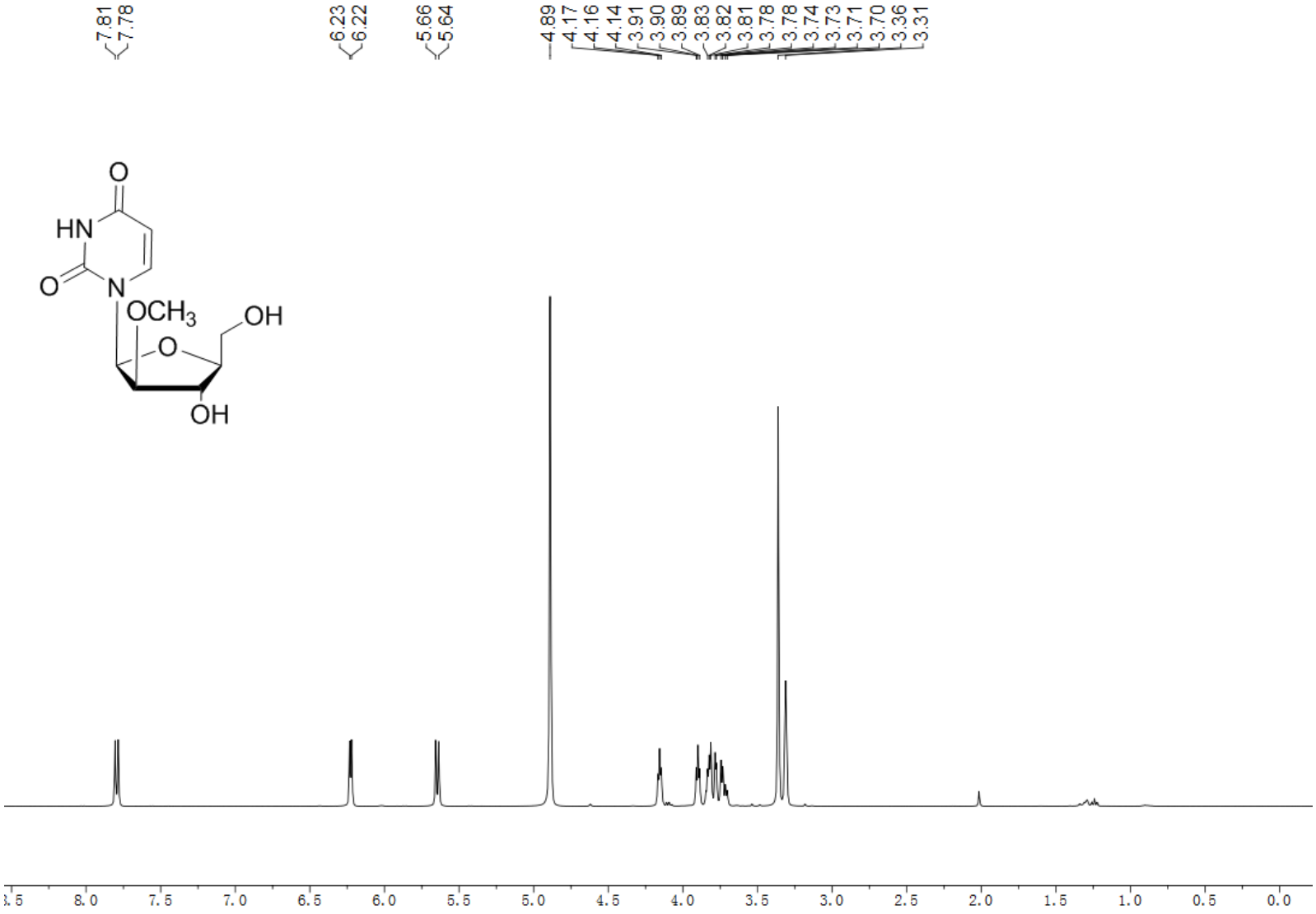

166.5
166.5
165.4
133.8
133.6
133.3
130.0
130.0
129.9
129.7
129.6
129.0
128.6
128.5
128.5
102.7
83.0
80.5
79.9
77.5
77.2
76.8
64.2
OH
O
OBz
BzO
OBz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

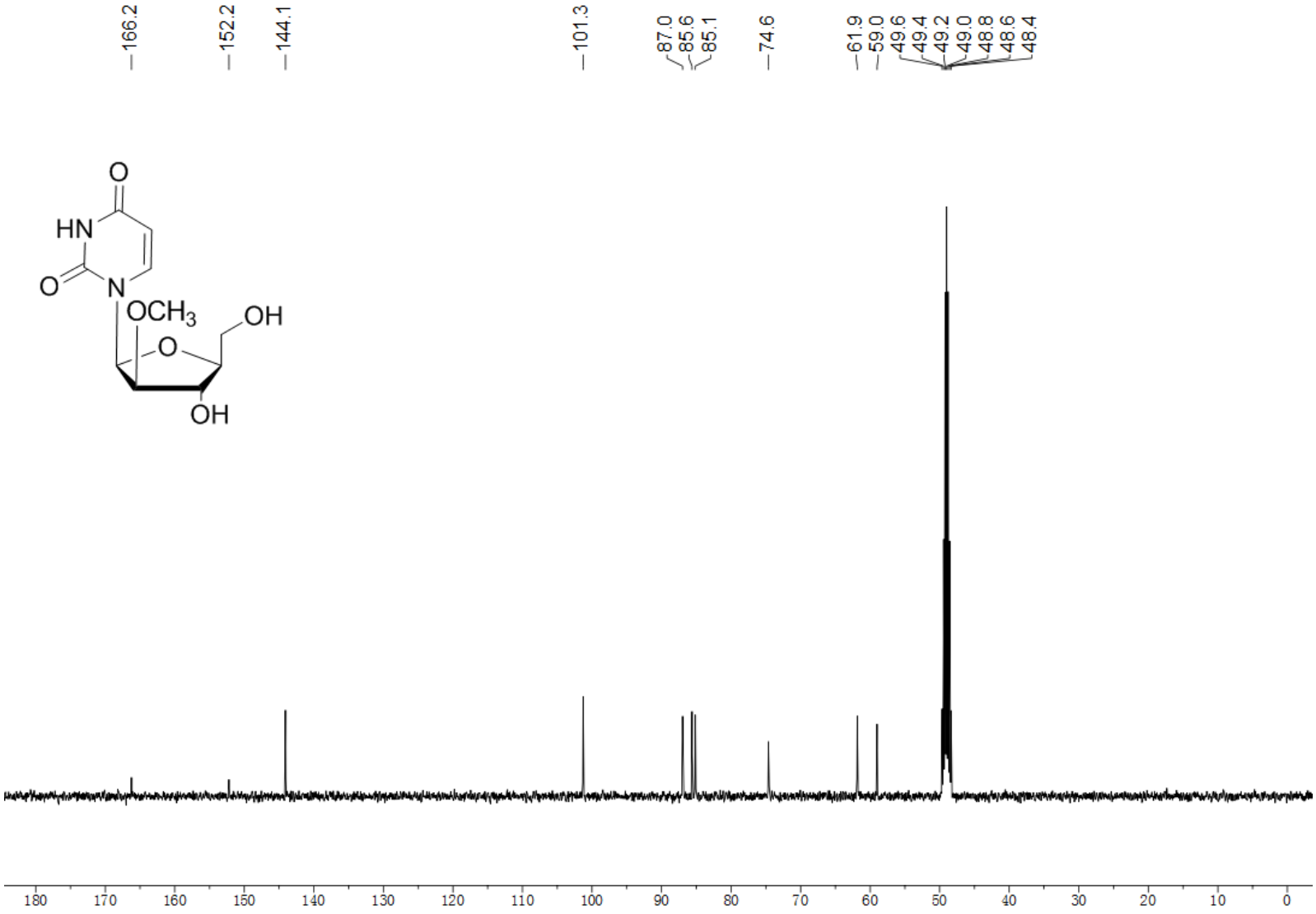

8.12
8.10
8.07
8.06
8.05
8.04
7.62
7.61
7.58
7.56
7.54
7.45
7.43
7.41
7.39
7.37
7.26
6.62
5.46
5.46
4.81
4.80
4.67
4.66
4.65
4.21
3.59
CH3O
OBz
O
BzO
OBz
2.06
3.94
3.00
6.04
0.99
0.99
1.00
2.00
0.98
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

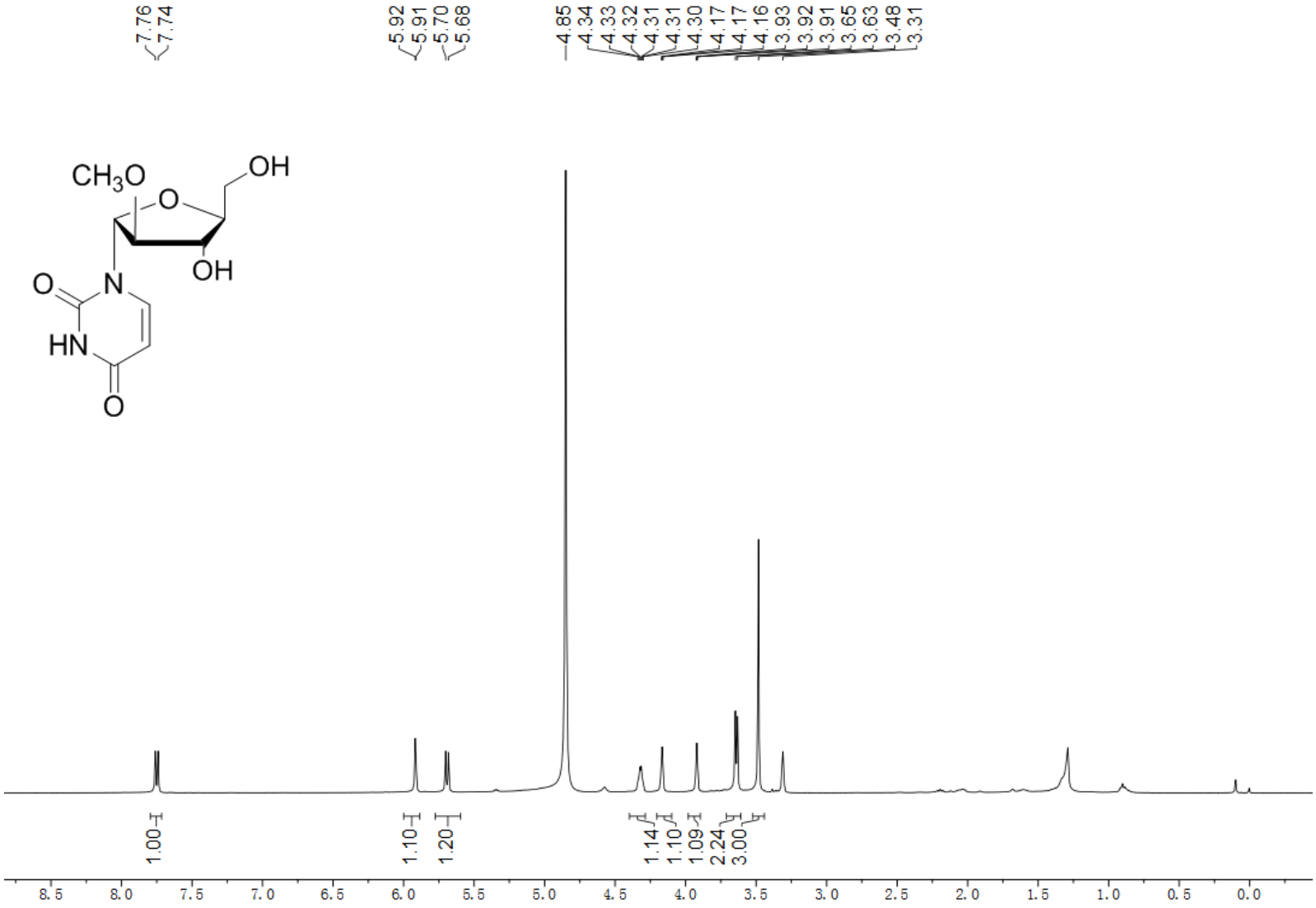

166.43
165.78
165.16
133.76
133.63
133.23
130.01
129.98
129.93
129.87
129.66
129.27
128.64
128.54
128.44
100.60
88.03
84.02
77.16
64.24
58.20
CH3O
OBz
O
BzO
OBz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

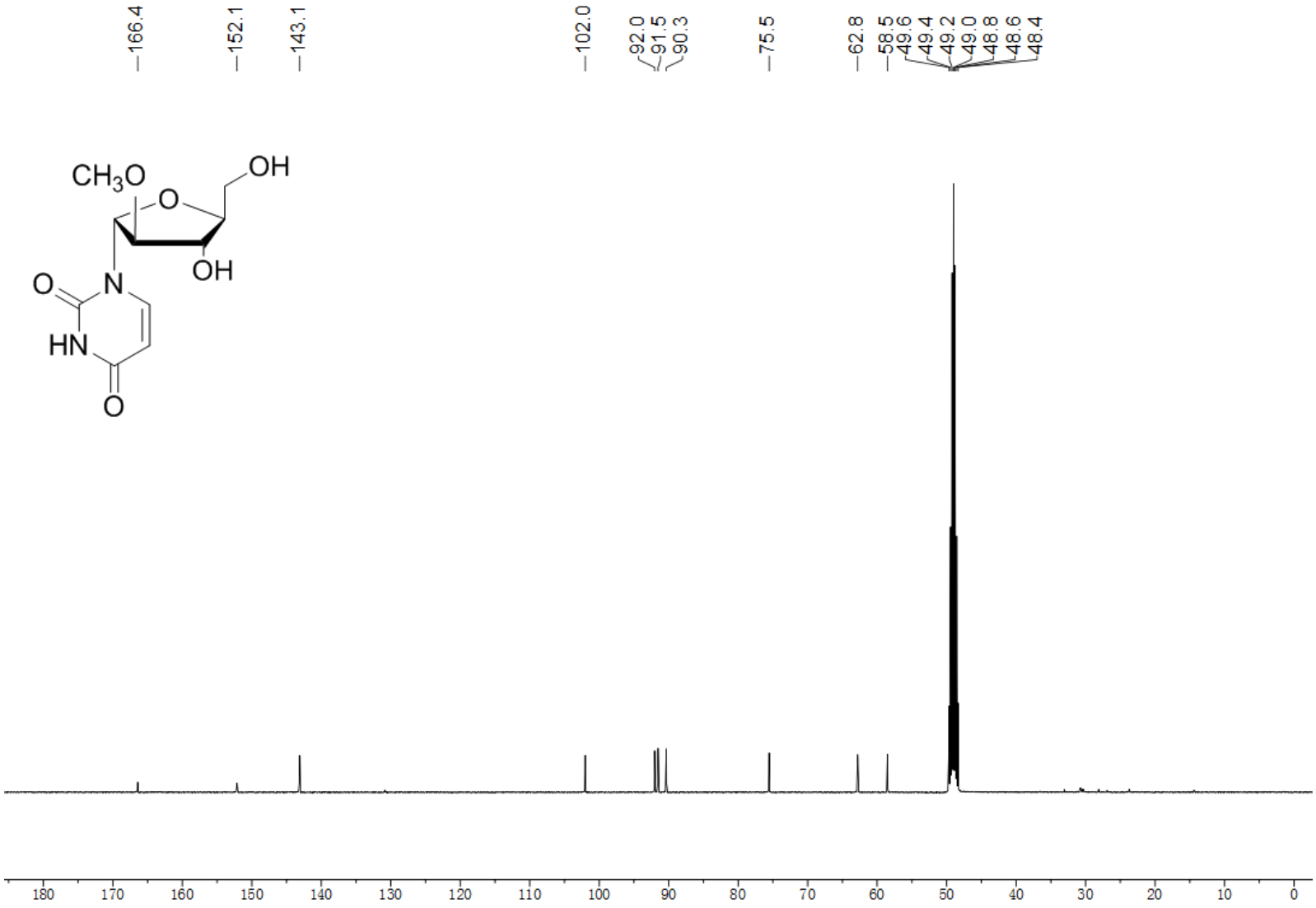

9.02
8.10
8.08
8.05
7.64
7.63
7.62
7.59
7.57
7.51
7.49
7.48
7.46
7.44
7.26
6.32
6.31
5.69
5.67
5.48
4.74
4.73
4.71
4.68
4.67
4.52
4.11
4.10
3.41
O
HN
O
N
OCH3
O
OBz
OBz
0.95
4.01
2.96
4.09
0.98
0.96
1.00
1.90
1.00
0.97
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

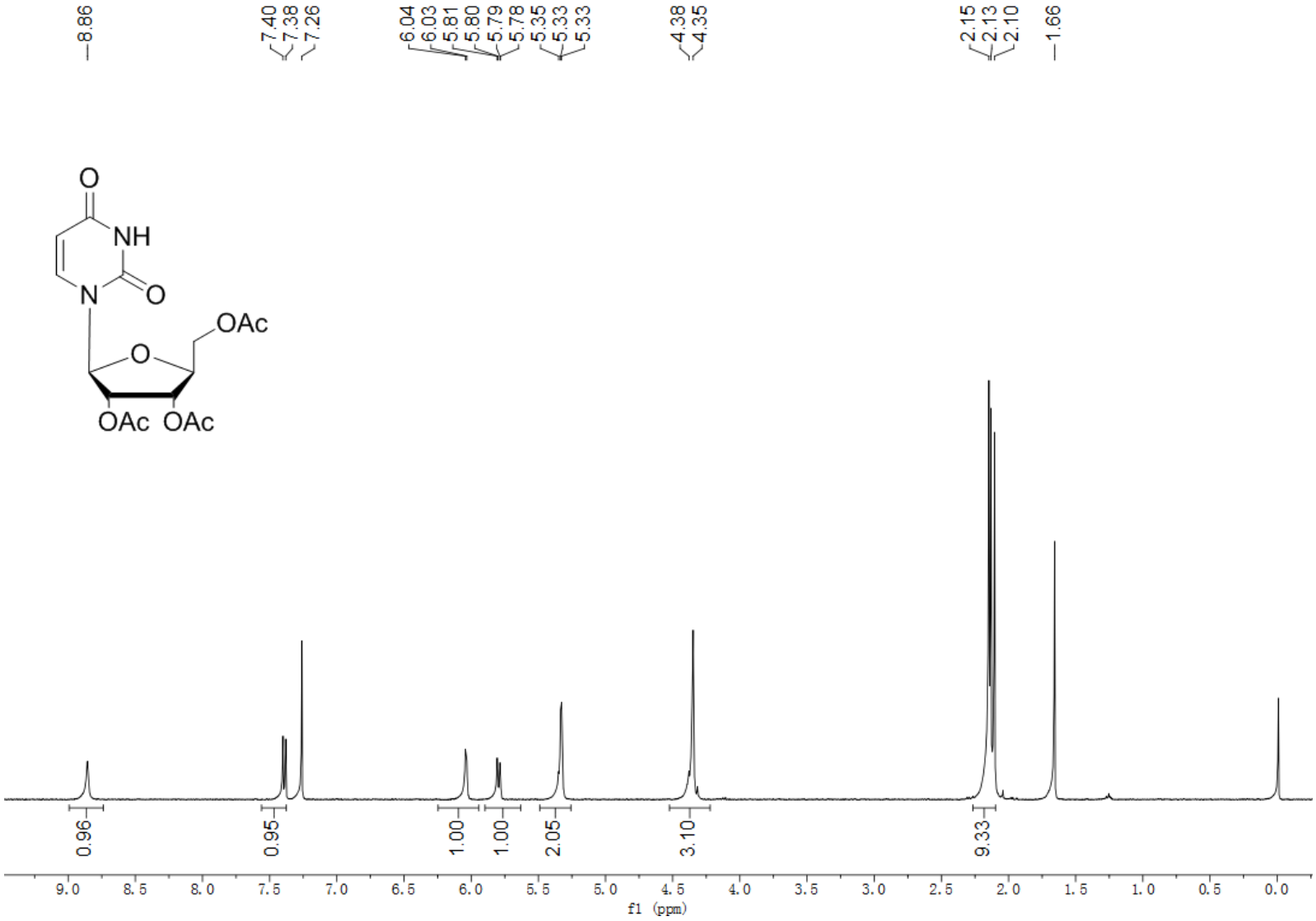

166.4
165.7
163.2
150.4
142.0
134.1
133.5
130.0
129.9
129.7
128.8
128.6
101.4
85.8
82.7
81.3
77.5
77.2
76.8
76.5
63.7
58.8
O
HN
O
N
OCH3
O
OBz
OBz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

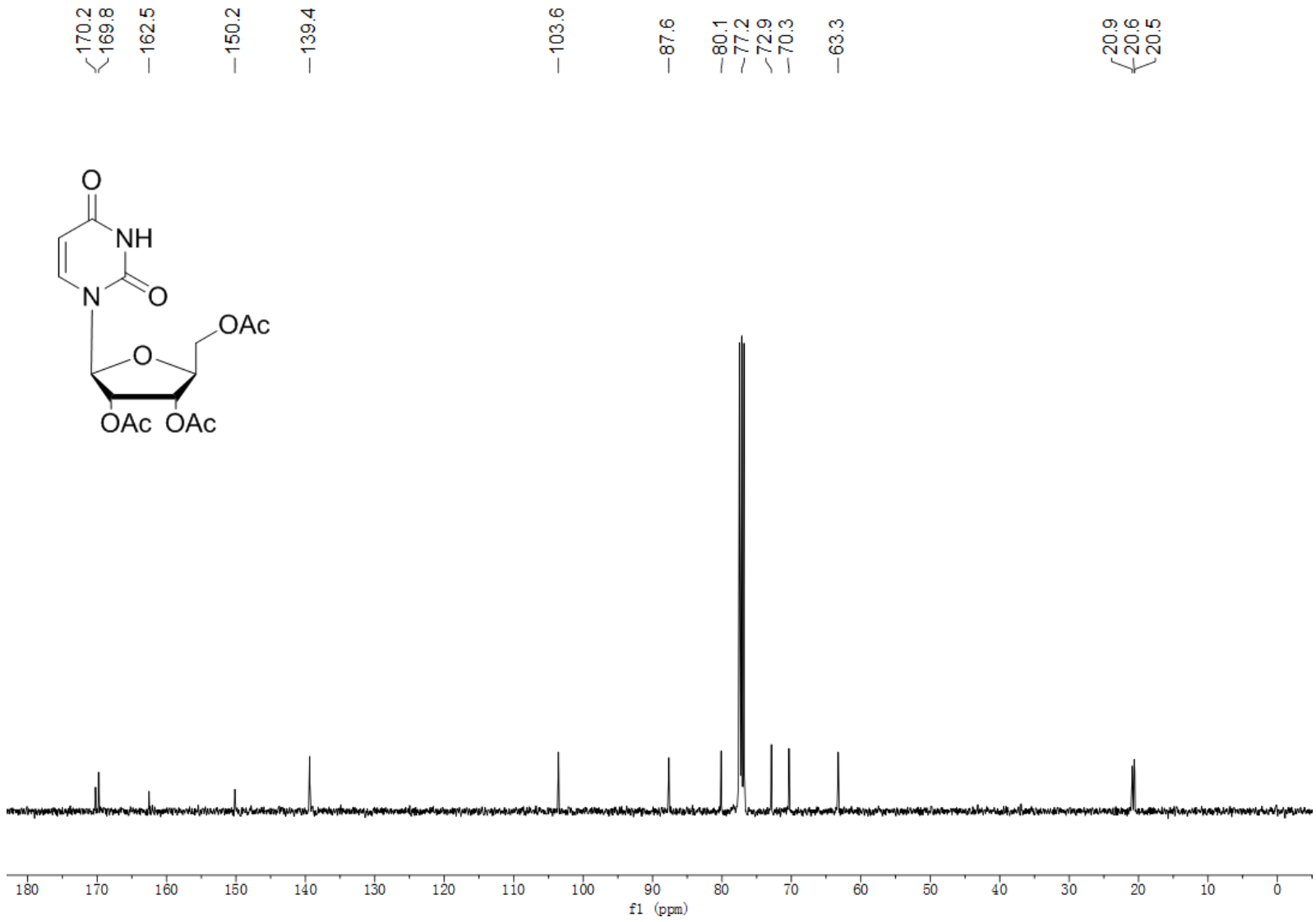

7.81
7.78
6.23
6.22
5.66
5.64
4.89
4.17
4.16
4.14
3.91
3.90
3.89
3.83
3.82
3.81
3.78
3.78
3.74
3.73
3.71
3.70
3.36
3.31
O
HN
O
N
OCH3
O
OH
OH
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

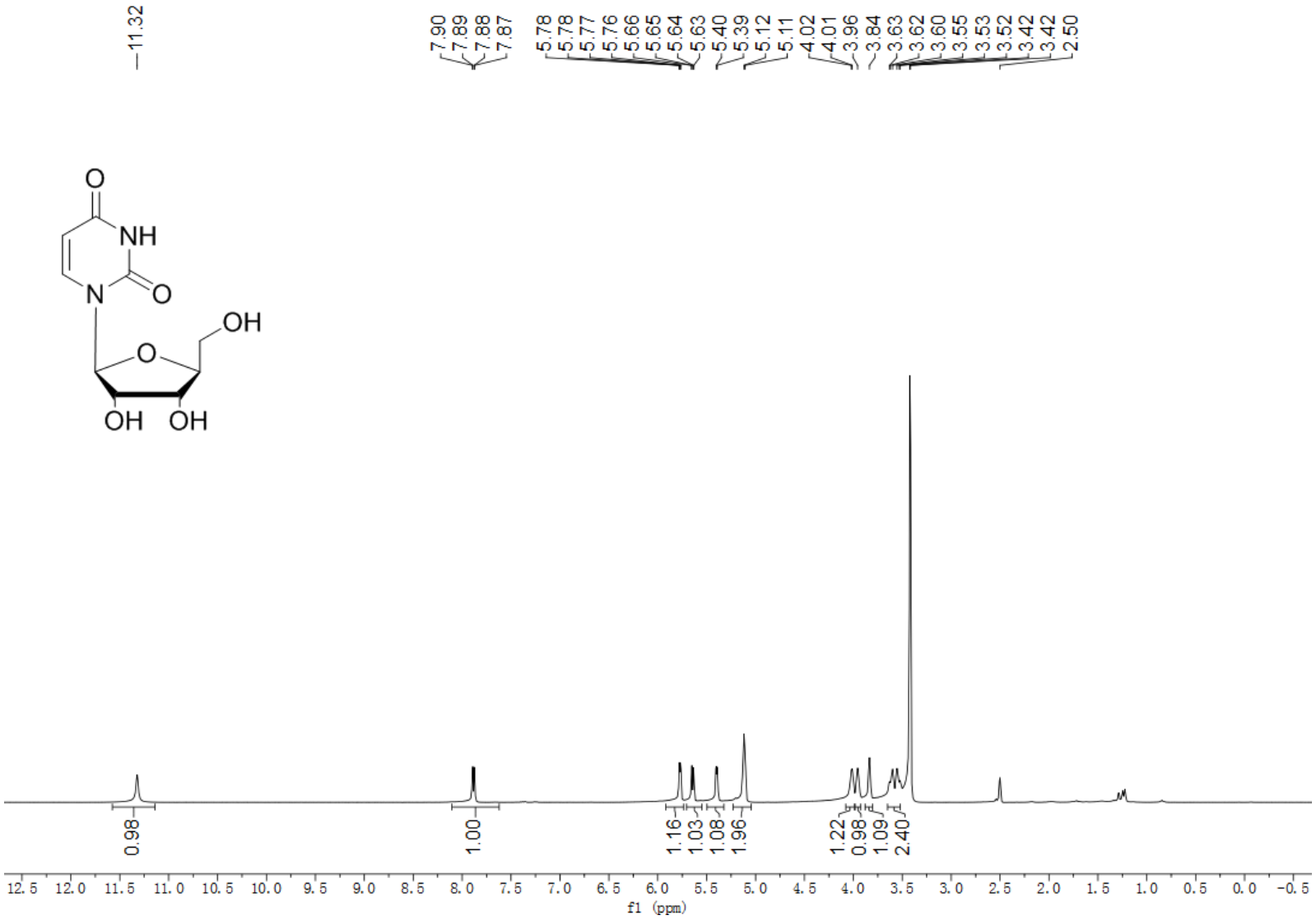

166.2
152.2
144.1
101.3
87.0
85.6
85.1
74.6
61.9
59.0
49.6
49.4
49.2
49.0
48.8
48.6
48.4
O
HN
O
N
OCH3
O
OH
OH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

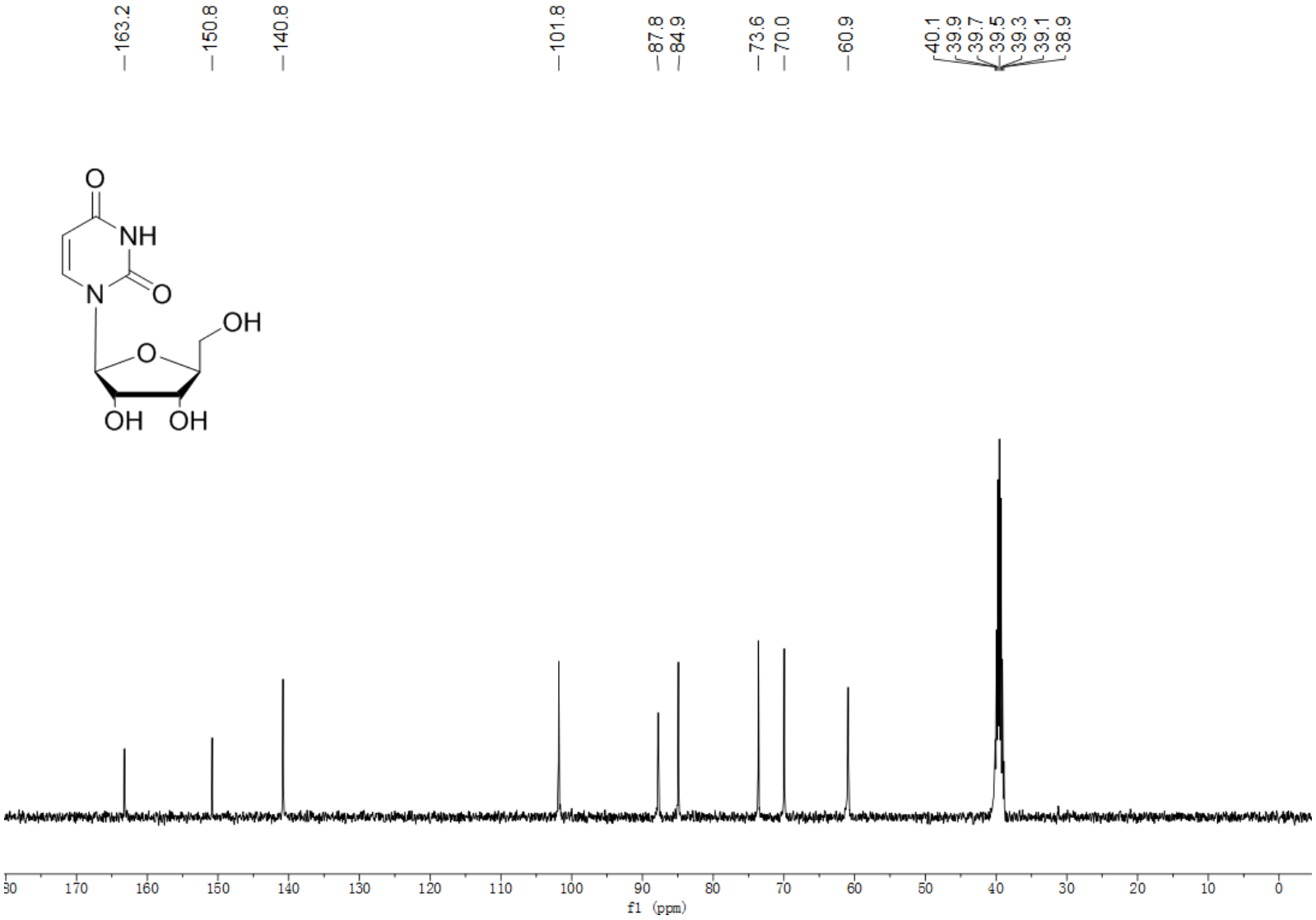

7.76
7.74
5.92
5.91
5.70
5.68
4.85
4.34
4.33
4.32
4.31
4.31
4.30
4.17
4.17
4.16
3.93
3.92
3.91
3.65
3.63
3.48
3.31
CH3O
O
OH
OH
O
N
HN
O
1.00
1.10
1.20
1.14
1.10
1.09
2.24
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

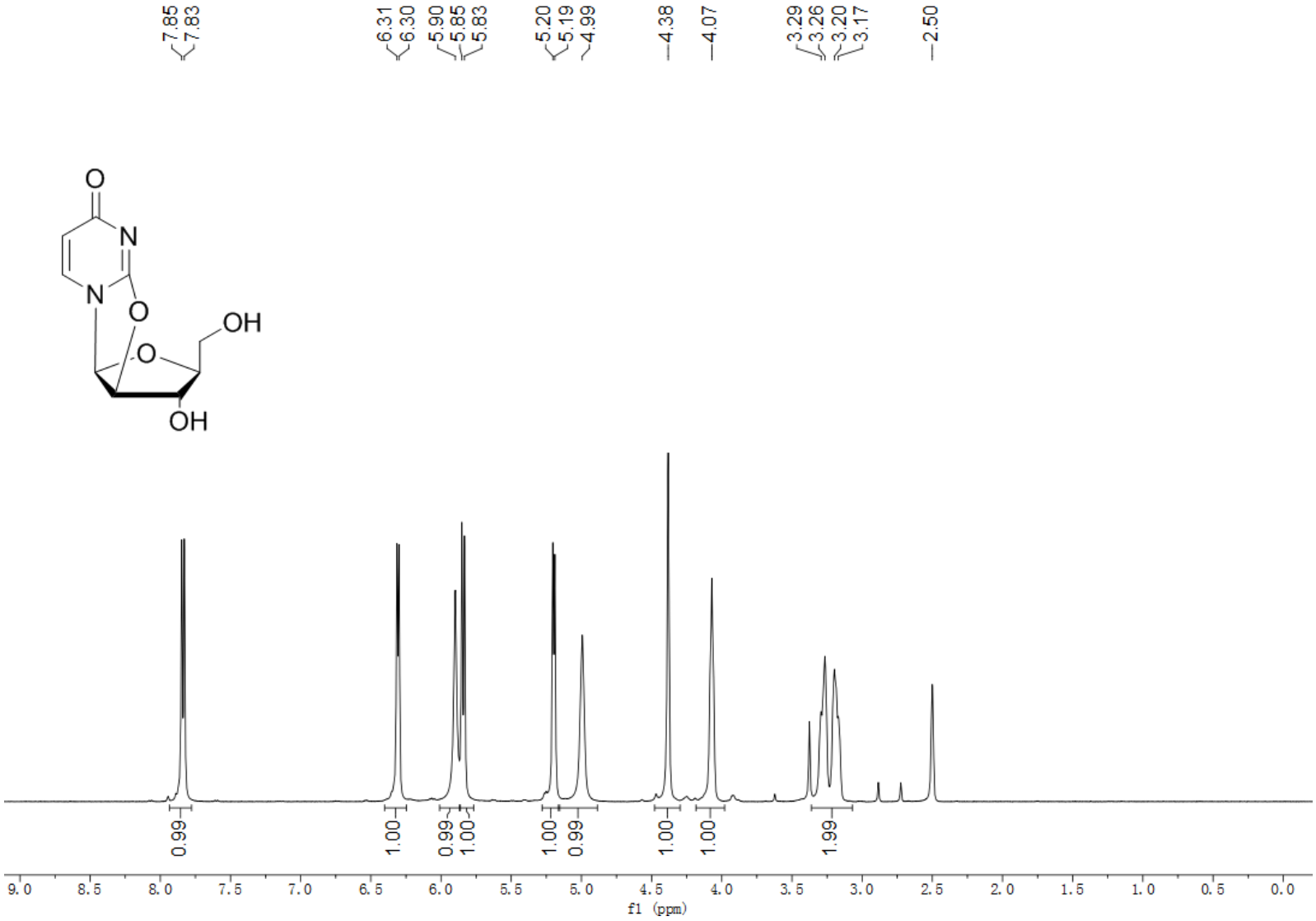

166.4
152.1
143.1
102.0
92.0
91.5
90.3
75.5
62.8
58.5
49.6
49.4
49.2
49.0
48.8
48.6
48.4
CH3O
O
OH
OH
O
N
HN
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

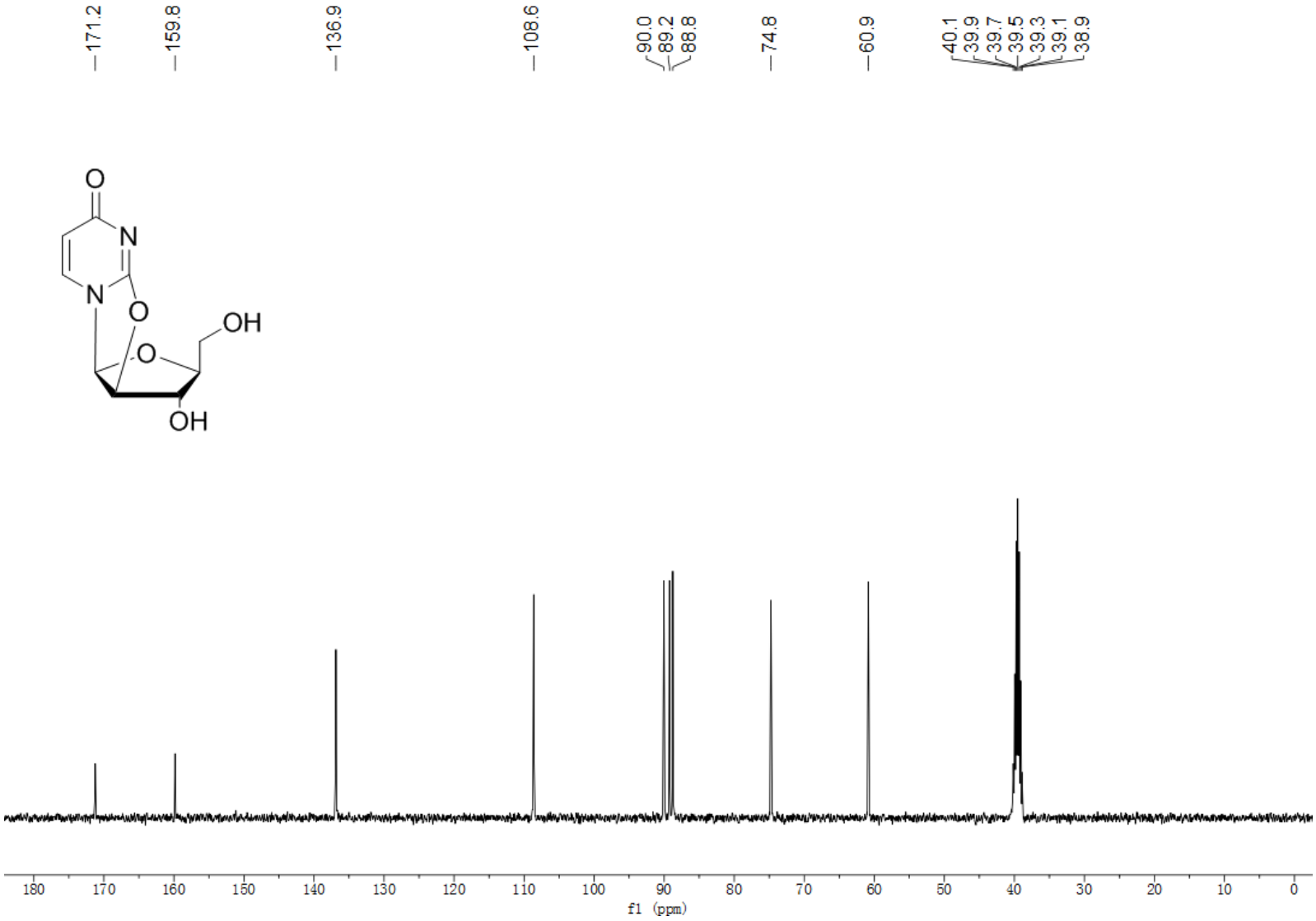

8.86
7.40
7.38
7.26
6.04
6.03
5.81
5.80
5.79
5.78
5.35
5.33
5.33
4.38
4.35
2.15
2.13
2.10
1.66
O
NH
O
N
O
OAc
O
OAc OAc
0.96
0.95
1.00
1.00
2.05
3.10
9.33
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

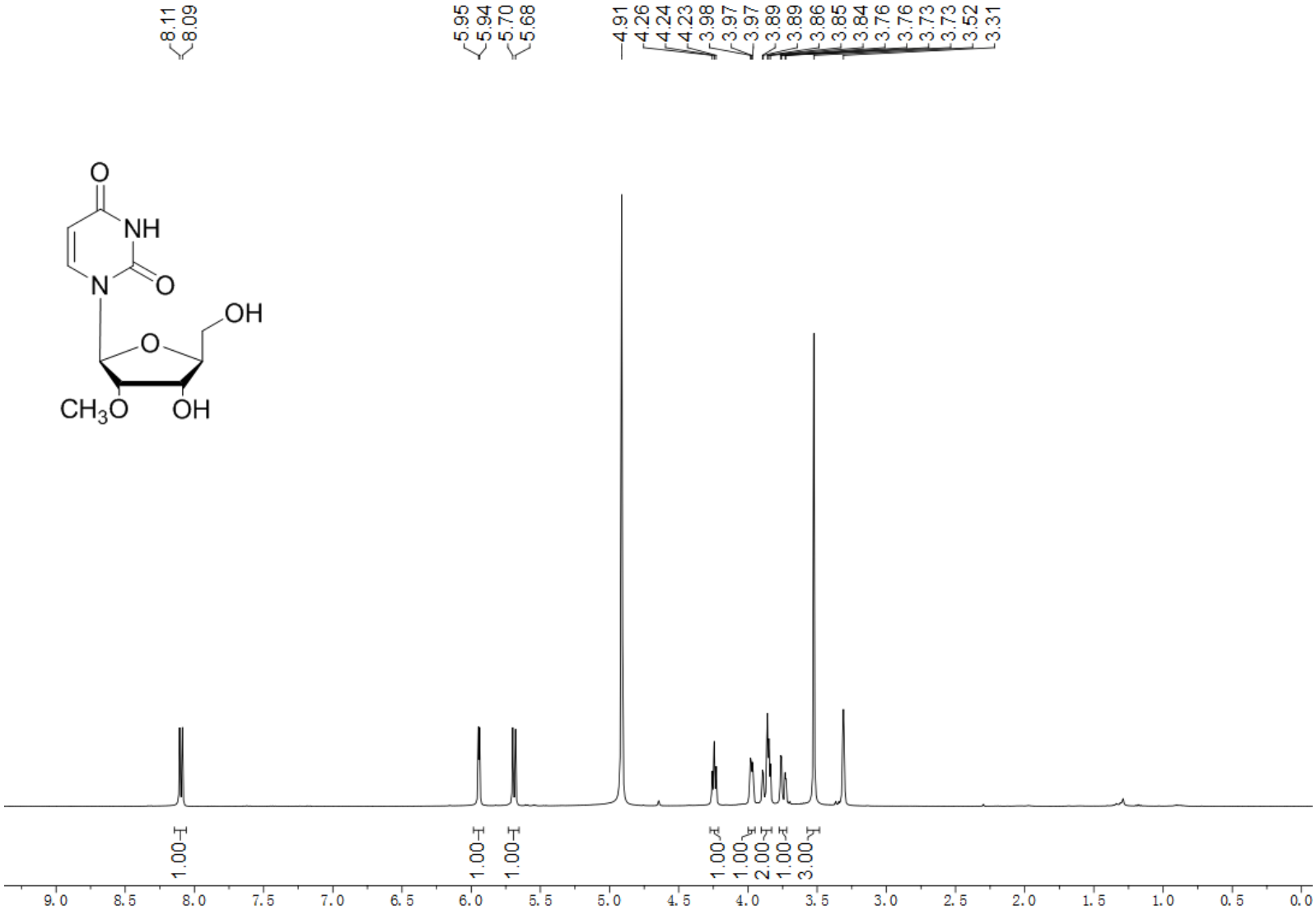

170.2
169.8
162.5
150.2
139.4
103.6
87.6
80.1
77.2
72.9
70.3
63.3
20.9
20.6
20.5
O
NH
O
N
O
OAc
O
OAc
OAc
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.32
7.90
7.89
7.88
7.87
5.78
5.78
5.77
5.76
5.66
5.65
5.64
5.63
5.40
5.39
5.12
5.11
4.02
4.01
3.96
3.84
3.63
3.62
3.60
3.55
3.53
3.52
3.42
3.42
2.50
O
NH
O
N
OH
O
OH
OH
0.98
1.00
1.16
1.03
1.08
1.96
1.22
0.98
1.09
2.40
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


163.2
150.8
140.8
101.8
87.8
84.9
73.6
70.0
60.9
40.1
39.9
39.7
39.5
39.3
39.1
38.9
O
NH
N
O
OH
O
OH
OH
f1 (ppm)


7.85
7.83
6.31
6.30
5.90
5.85
5.83
5.20
5.19
4.99
4.38
4.07
3.29
3.26
3.20
3.17
2.50
O
N
N
O
O
OH
OH
0.99
1.00
0.99
1.00
1.00
0.99
1.00
1.00
1.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.2
159.8
136.9
108.6
90.0
89.2
88.8
74.8
60.9
40.1
39.9
39.7
39.5
39.3
39.1
38.9
O
N
N
O
O
OH
OH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.11
8.09
5.95
5.94
5.70
5.68
4.91
4.26
4.24
4.23
3.98
3.97
3.97
3.89
3.89
3.86
3.85
3.84
3.76
3.76
3.73
3.73
3.52
3.31
O
NH
N
O
OH
O
CH3O
OH
1.00
1.00
1.00
1.00
1.00
2.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

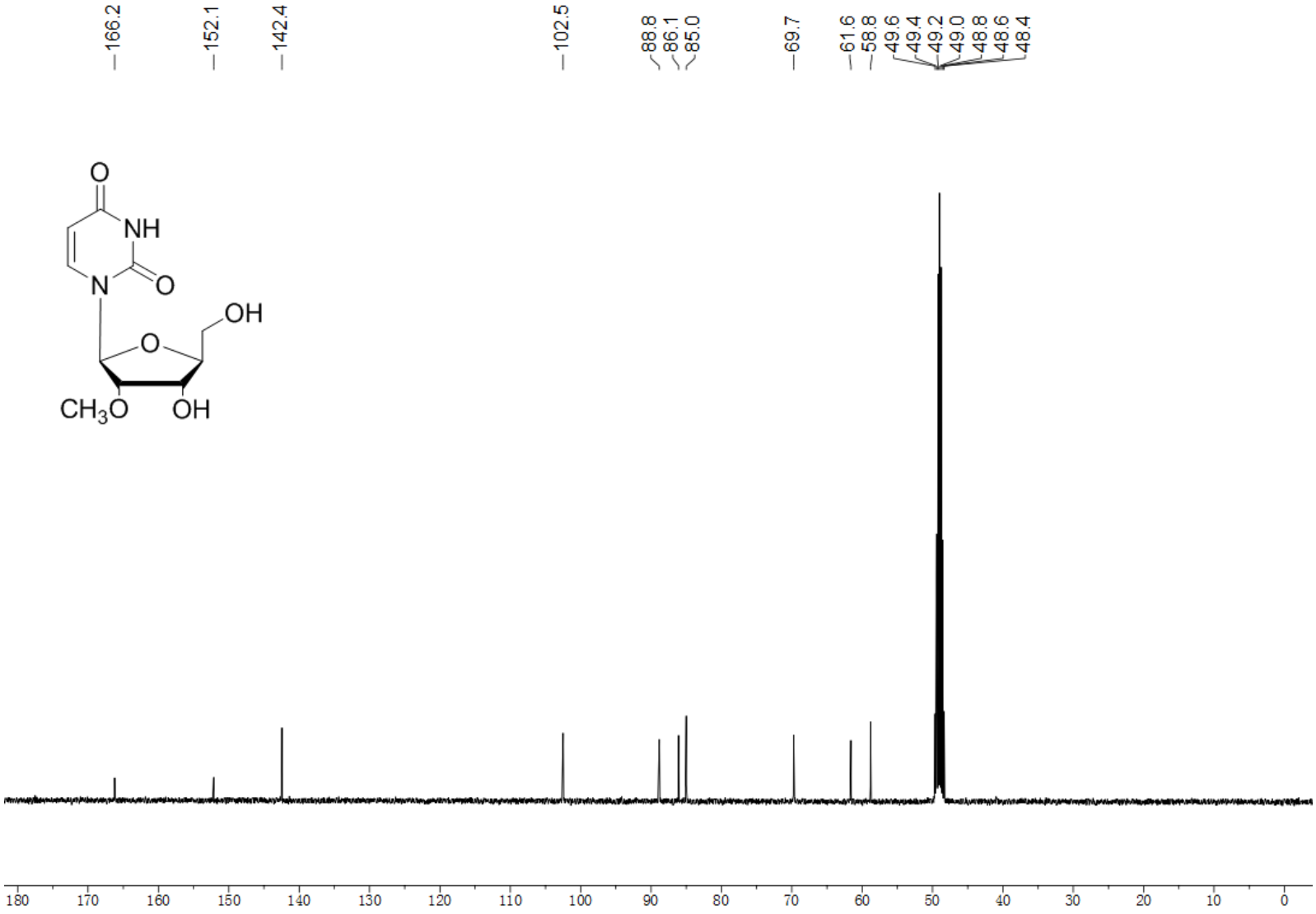

166.2
152.1
142.4
102.5
88.8
86.1
85.0
69.7
61.6
58.8
49.6
49.4
49.2
49.0
48.8
48.6
48.4
O
NH
N
O
OH
O
CH3O
OH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Table S1 Crystal data and structure refinement for 6.

Identification code	1
Empirical formula	$C_{26}H_{22}O_8$
Formula weight	462.43
Temperature/K	296(2)
Crystal system	orthorhombic
Space group	P2(1)2(1)2(1)
a/Å	5.8763(16)
b/Å	17.706(5)
c/Å	24.914(7)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2592.2(12)
Z	4
ρ_{calc}g/cm^3	1.185
μ/mm^{-1}	0.088
F(000)	968
Crystal size/mm^3	0.280 x 0.260 x 0.240
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.418 to 49.99
Index ranges	-6 ≤ h ≤ 6, -11 ≤ k ≤ 21, -29 ≤ l ≤ 26
Reflections collected	10840
Independent reflections	4518 [R(int) = 0.0398]
Data/restraints/parameters	4518/0/307
Goodness-of-fit on F^2	1.024
Final R indexes [I>=2σ (I)]	R_1 = 0.0698, wR_2 = 0.1931
Final R indexes [all data]	R_1 = 0.1116, wR_2 = 0.2253
Largest diff. peak/hole / e Å^{-3}	0.442/-0.263

Table S2 Crystal data and structure refinement for 12.

Identification code	1
Empirical formula	$C_{18}H_{20}N_4O_{10}$
Formula weight	452.38
Temperature/K	296(2)
Crystal system	orthorhombic
Space group	P2(1)2(1)2(1)
a/Å	7.4296(7)
b/Å	13.6558(12)
c/Å	18.1898(16)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1845.5(3)
Z	4
ρ_{calc}g/cm^3	1.628
μ/mm^{-1}	0.135
F(000)	944
Crystal size/mm^3	0.260 x 0.200 x 0.170
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.382 to 51.99
Index ranges	-8 ≤ h ≤ 9, -16 ≤ k ≤ 15, -22 ≤ l ≤ 19
Reflections collected	10220
Independent reflections	3616 [R(int) = 0.0213]
Data/restraints/parameters	3616/0/298
Goodness-of-fit on F^2	0.930
Final R indexes [I>=2σ (I)]	R_1 = 0.0278, wR_2 = 0.0693
Final R indexes [all data]	R_1 = 0.0311, wR_2 = 0.0719
Largest diff. peak/hole / e Å^{-3}	0.198/-0.144