Supporting Information

Formal Synthesis of Gracilamine Using Rh(I)-Catalyzed [3 + 2 + 1] Cycloaddition of 1-Yne−Vinylcyclopropanes and CO

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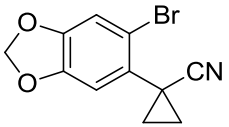
^{1}H and ^{13}C NMR spectra



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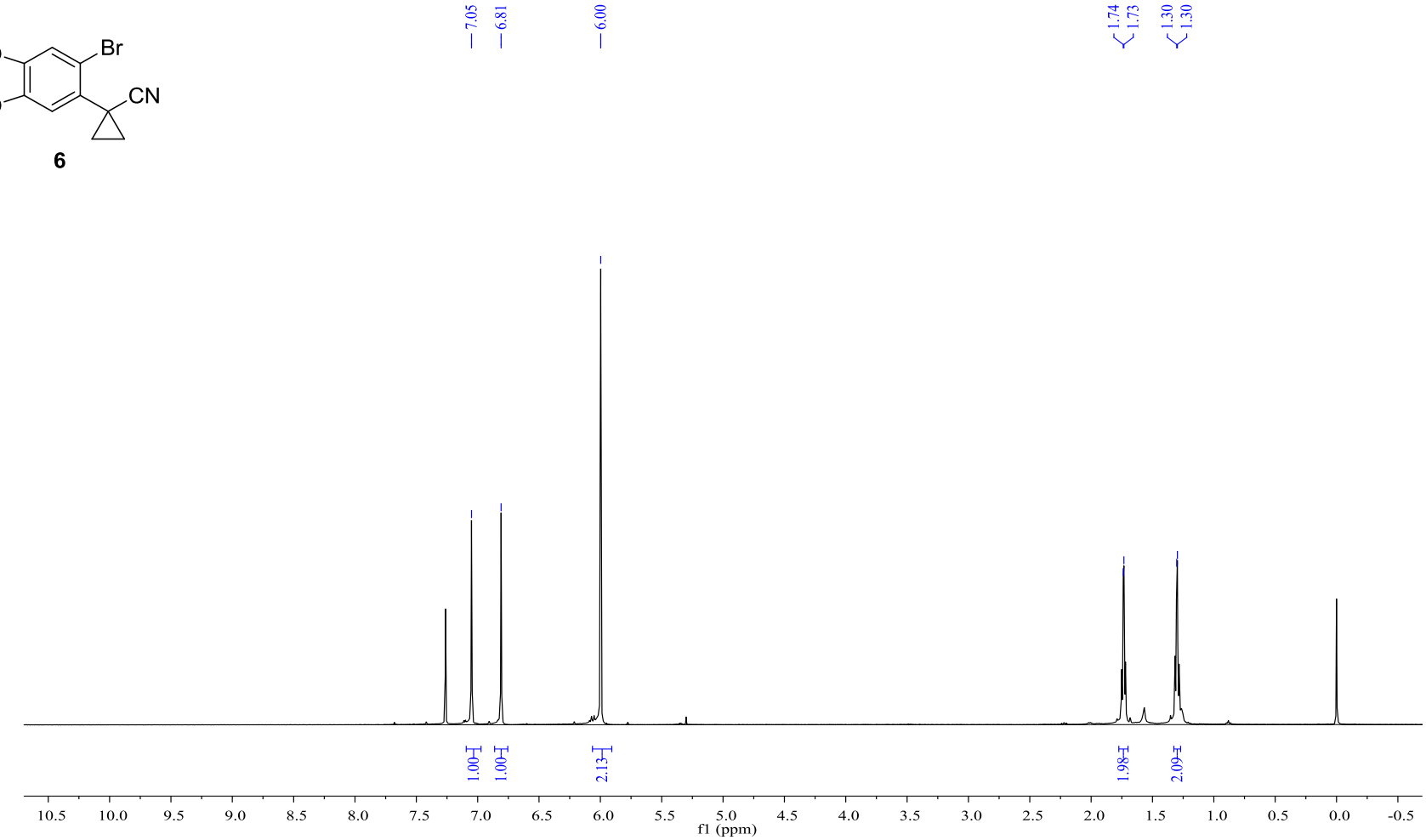

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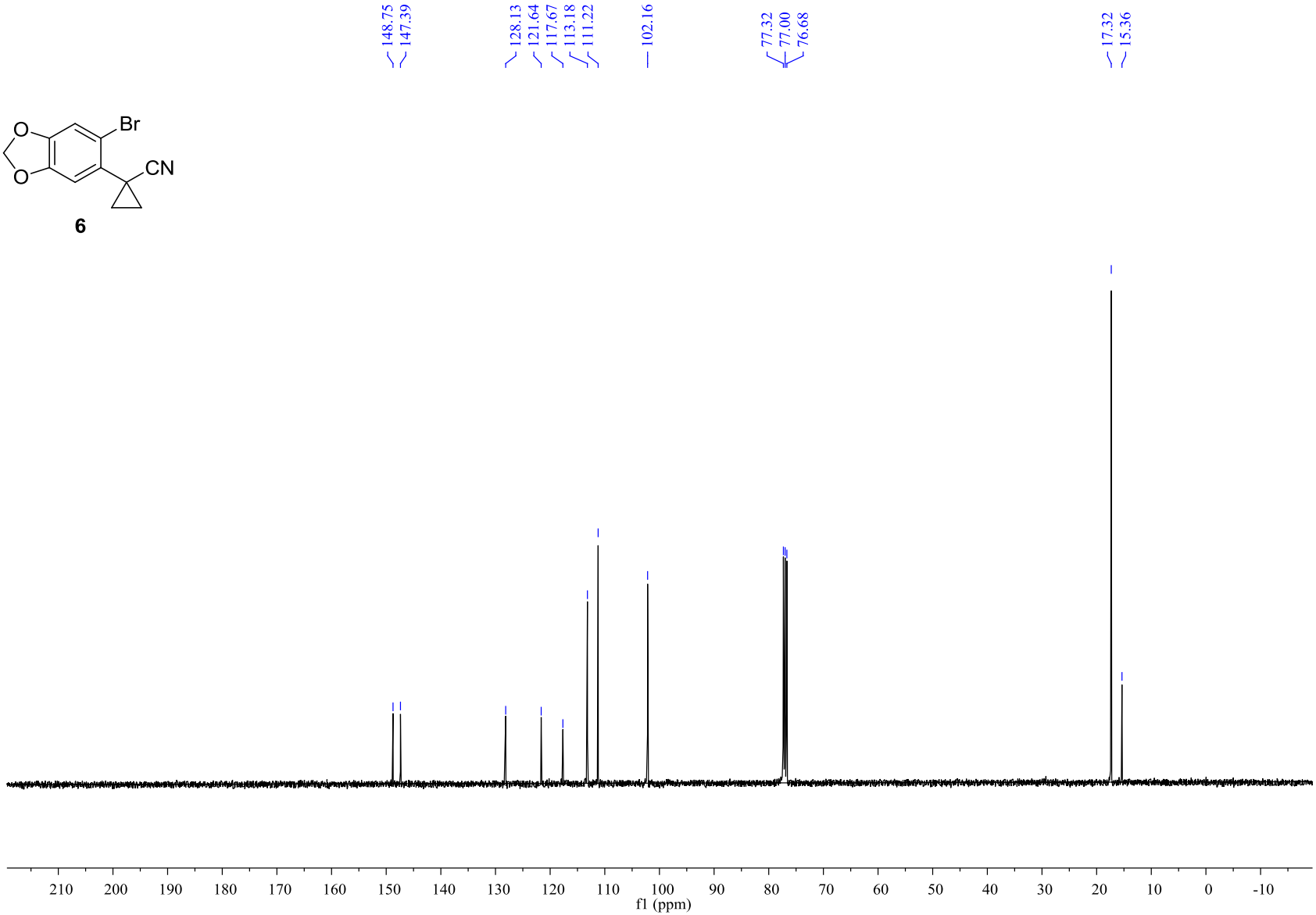
Br
CN

6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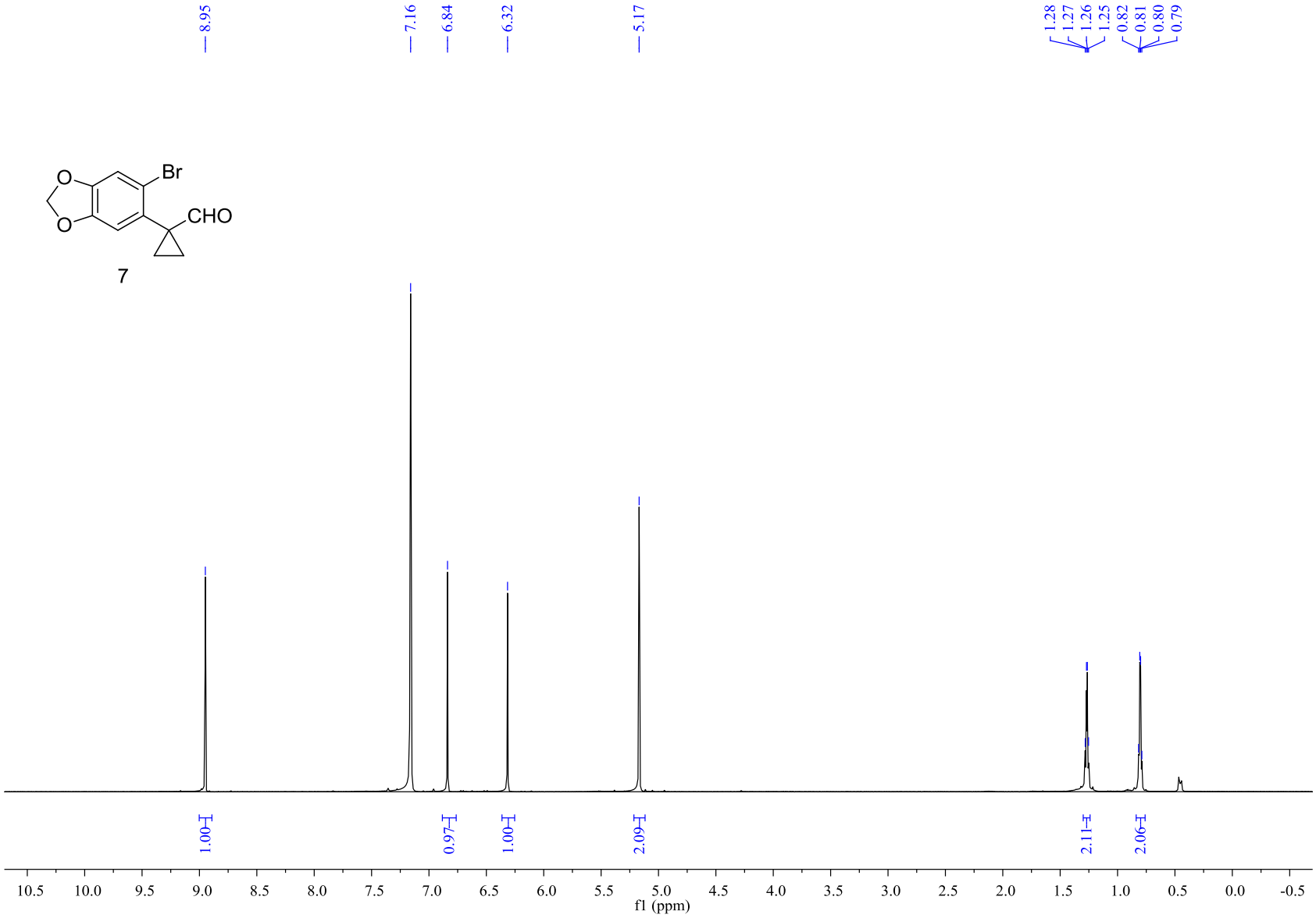
f1 (ppm)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O
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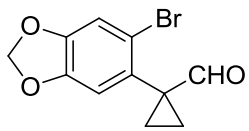
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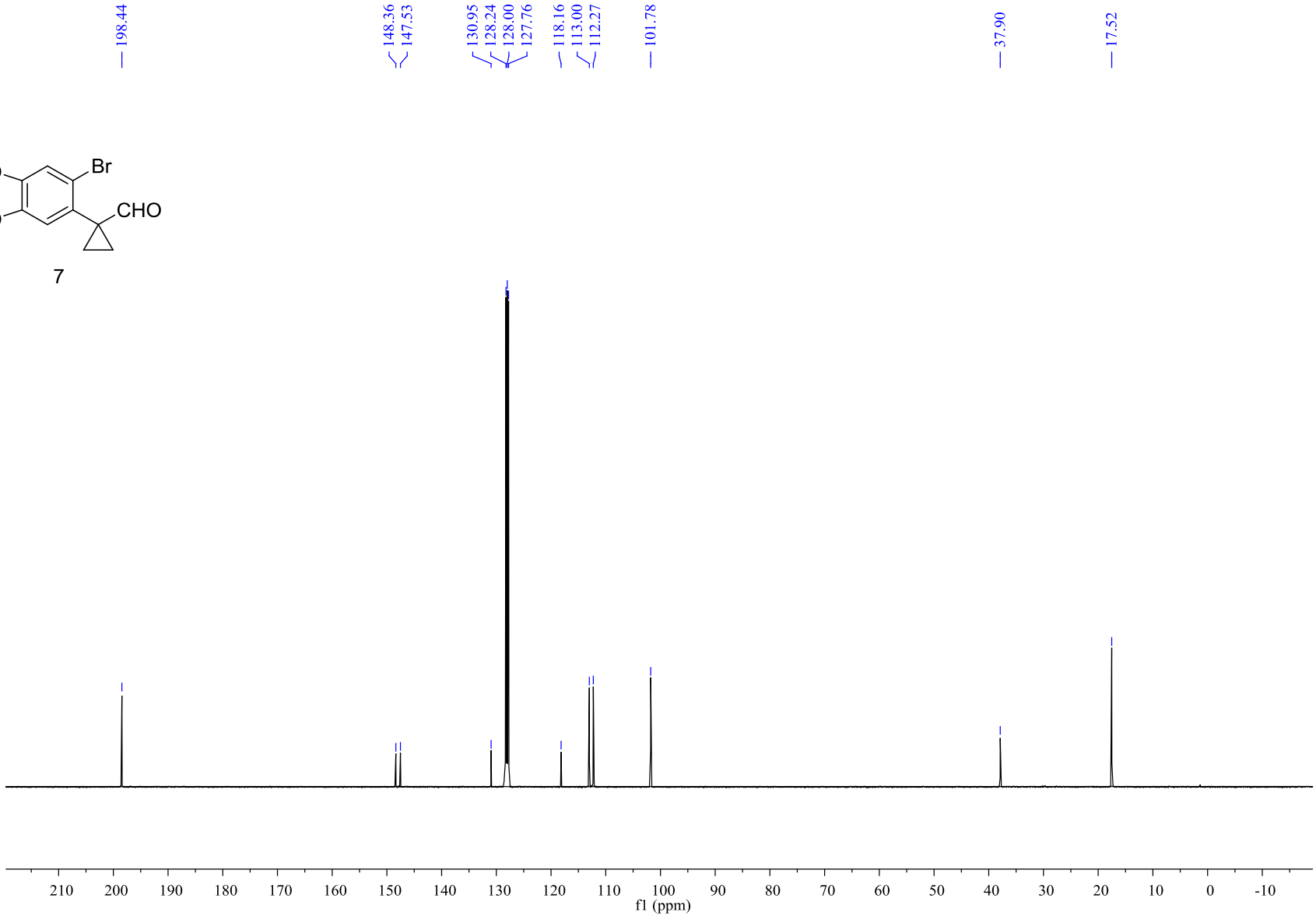
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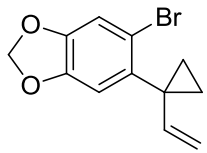
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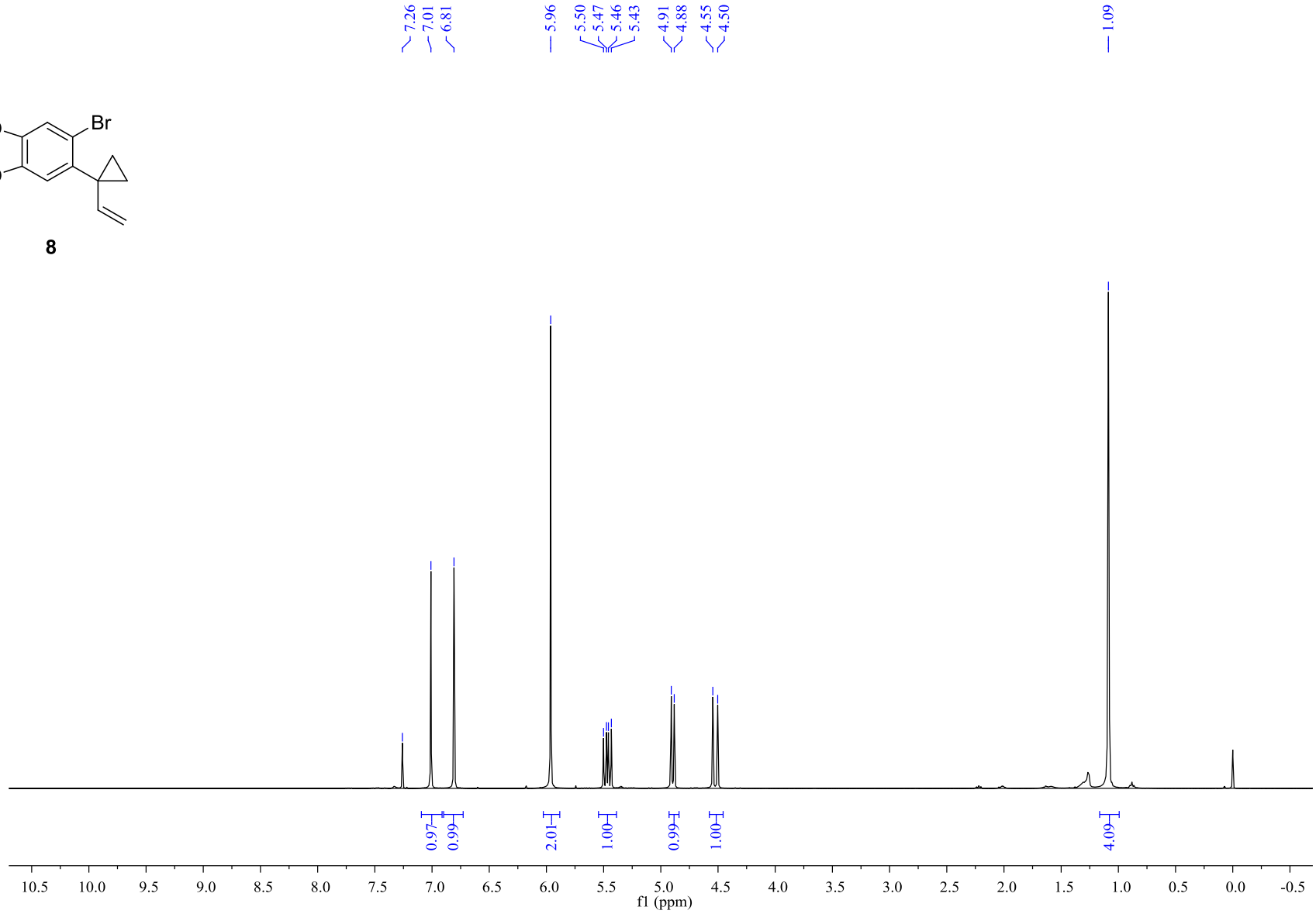
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2.01
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0.99
1.00
4.09
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f1 (ppm)

Br
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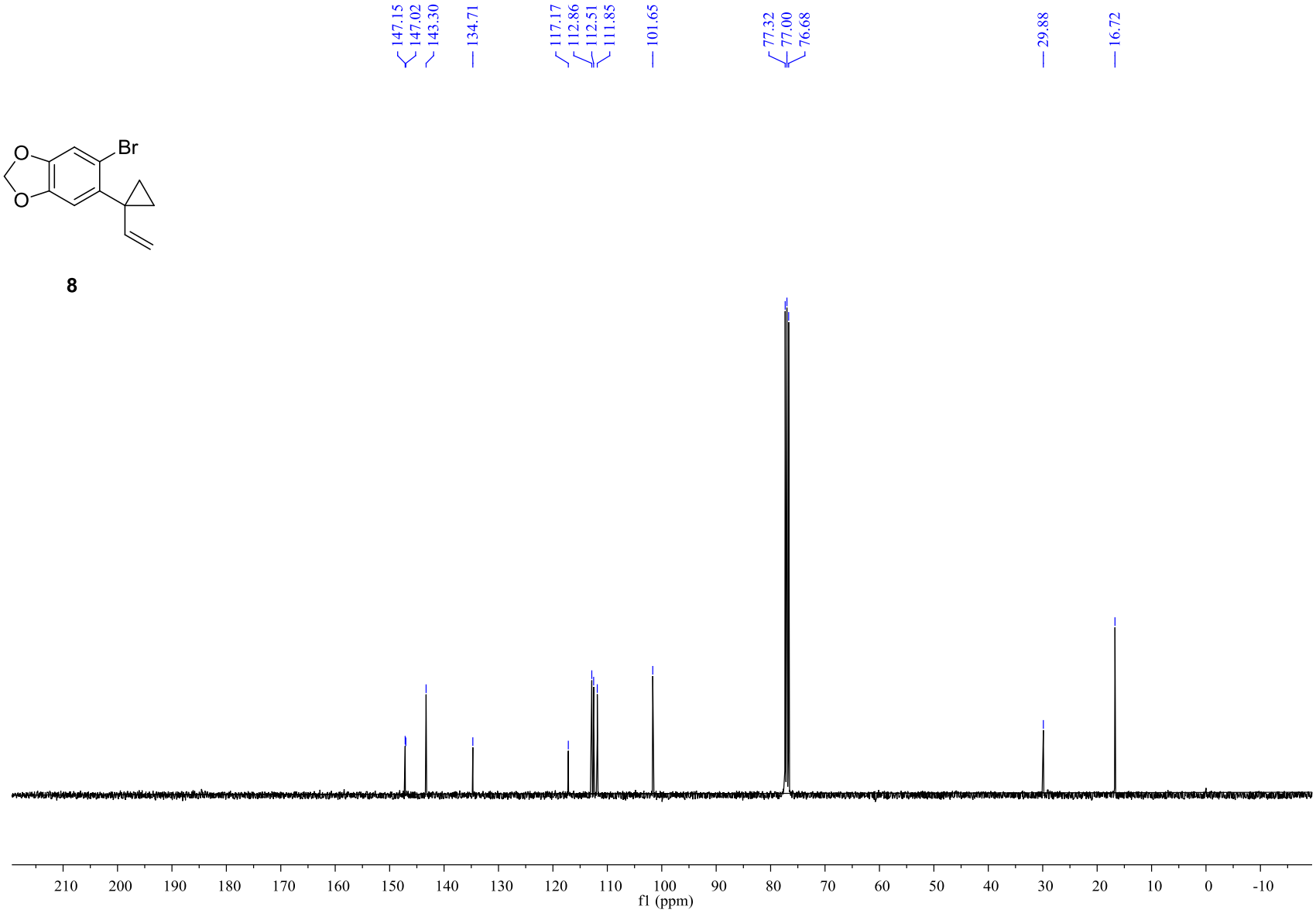
O

8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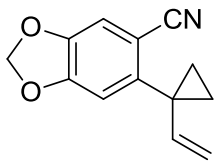
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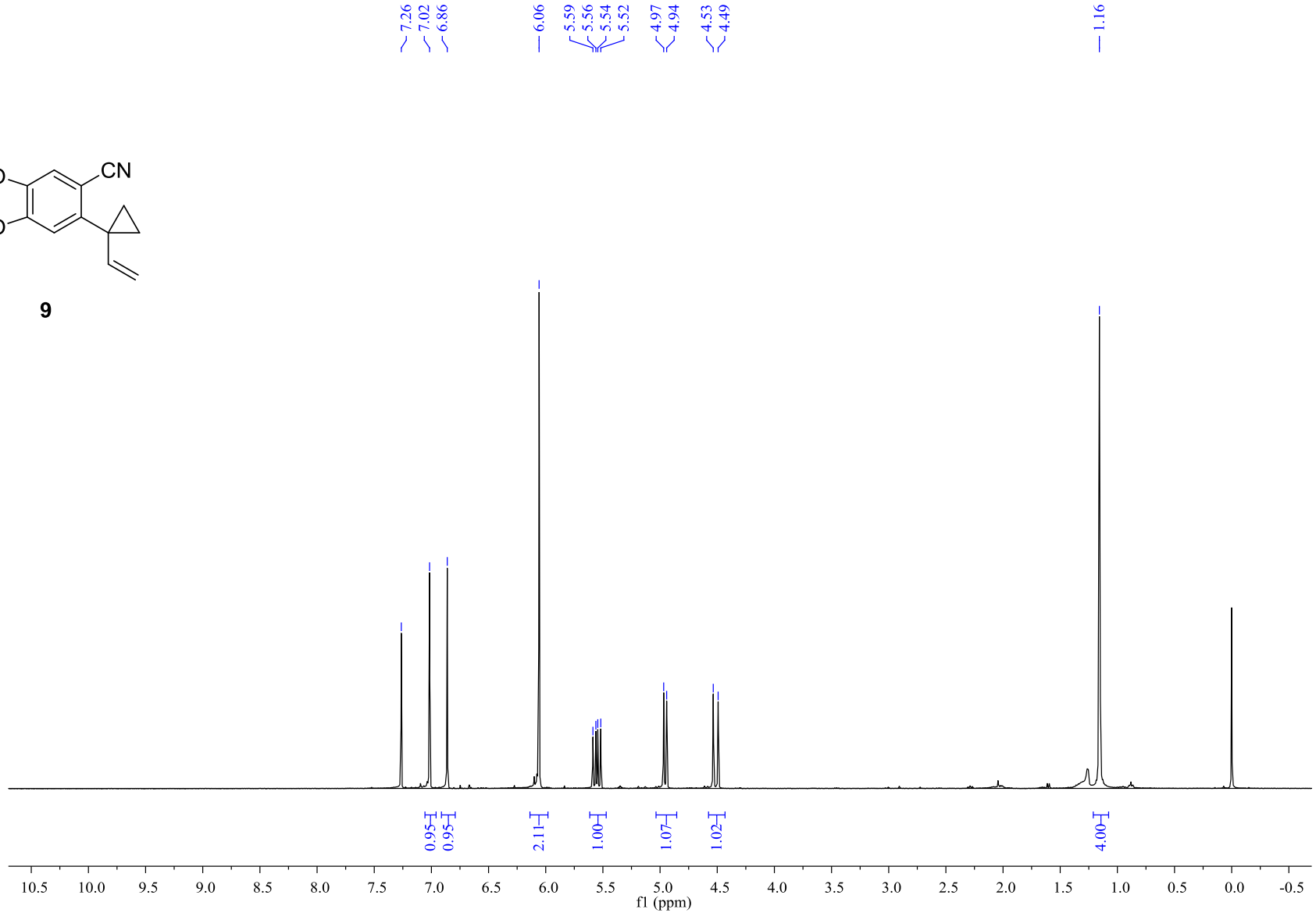
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0.95
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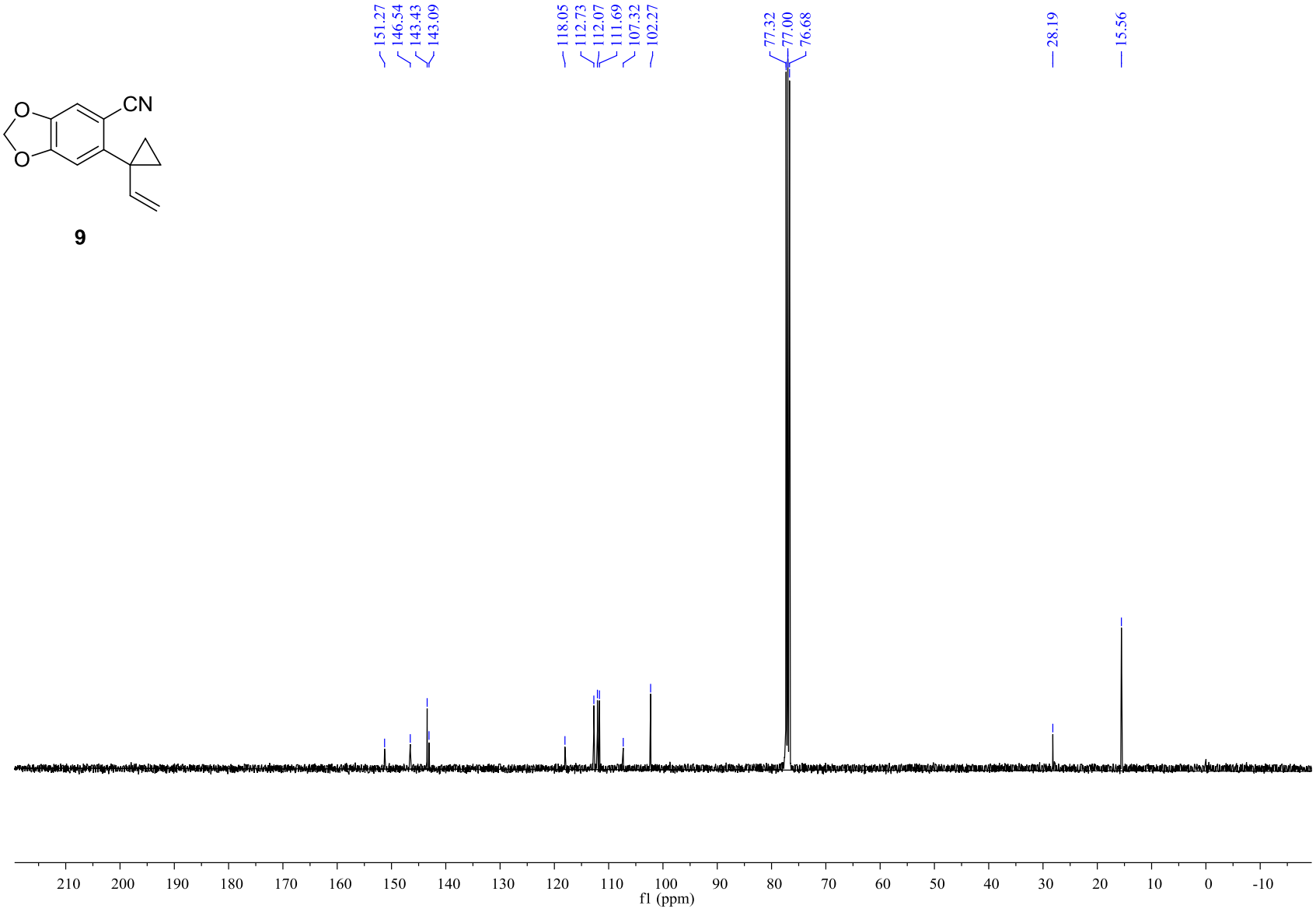
1.00
1.07
1.02
4.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

CN
O
O

9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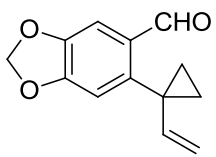
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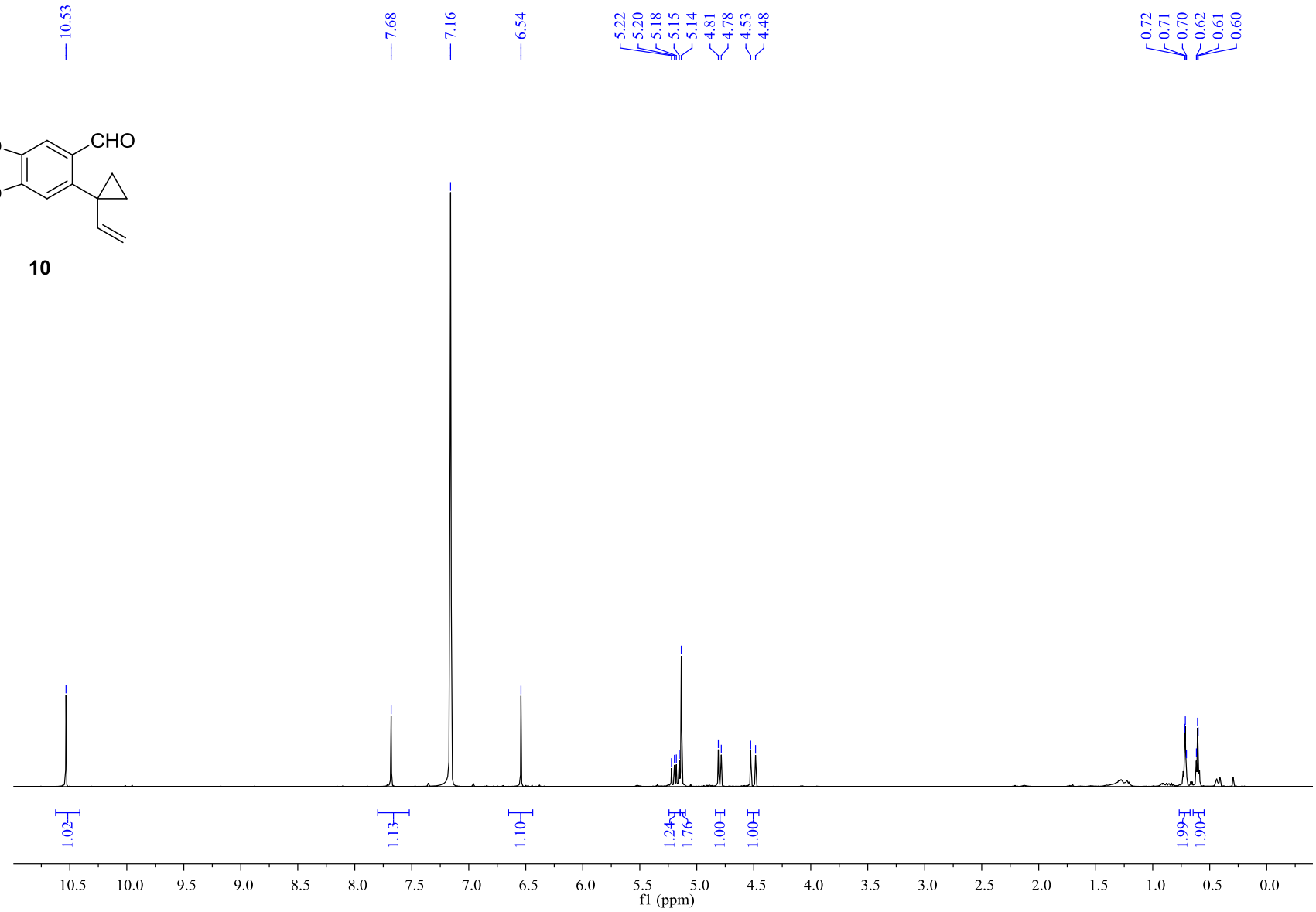
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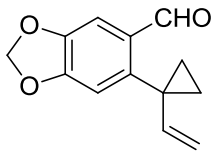
0.71
0.70
0.62
0.61
0.60
1.02
1.13
1.10
1.24
1.76
1.00
1.00
1.99
1.90
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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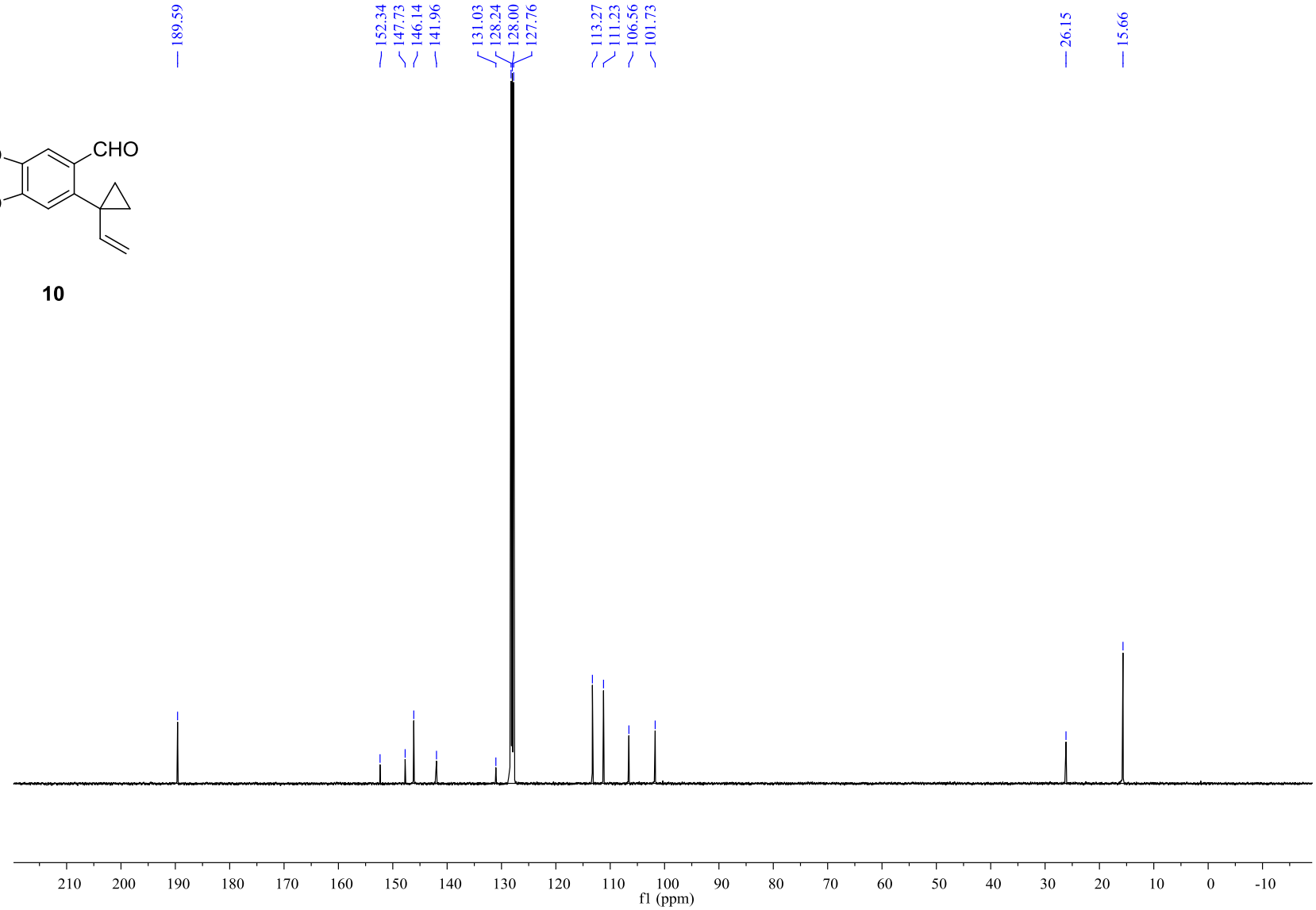
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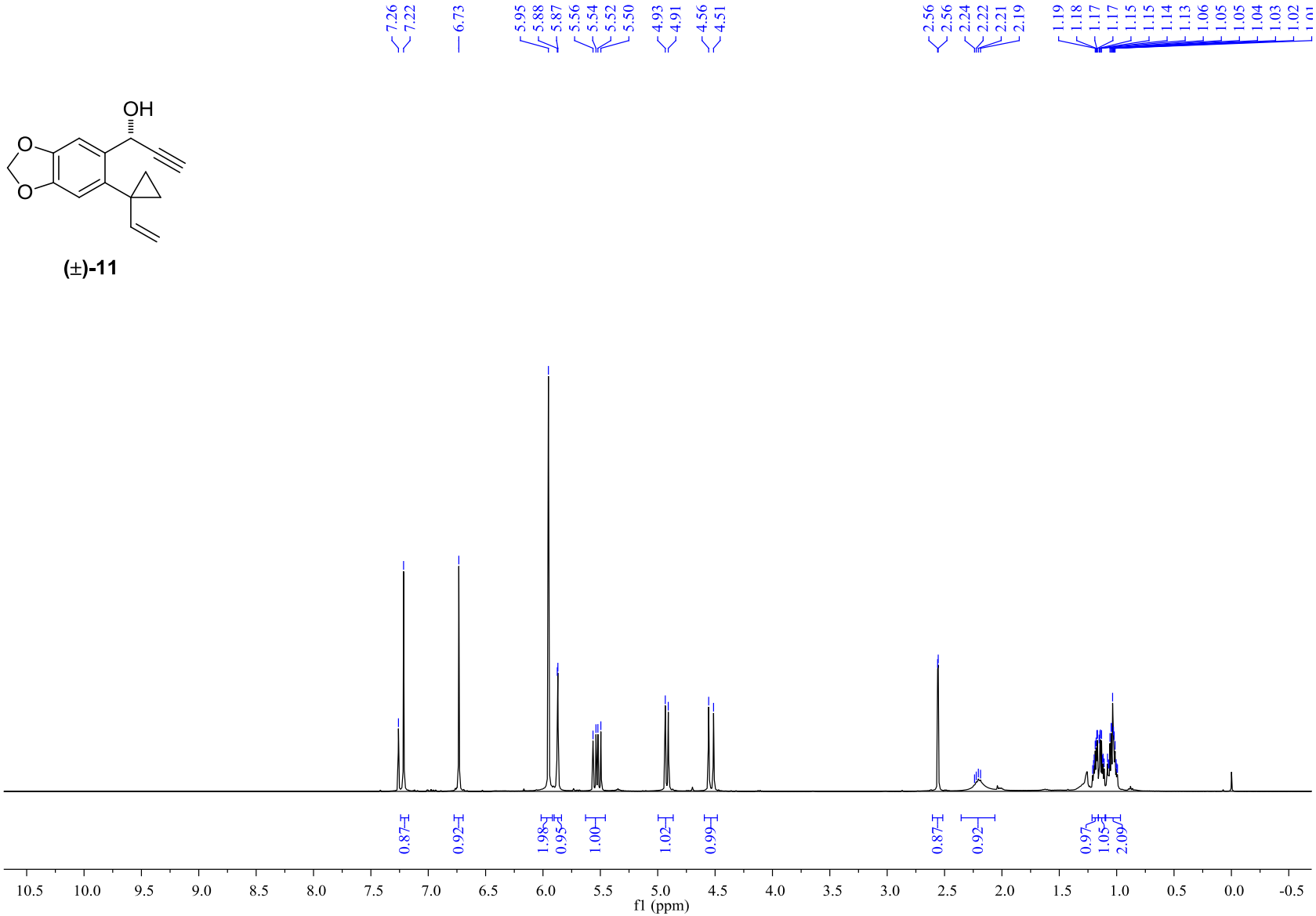
f1 (ppm)

OH
O
O

(±)-11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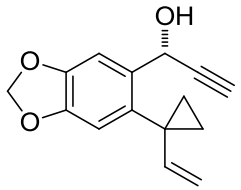
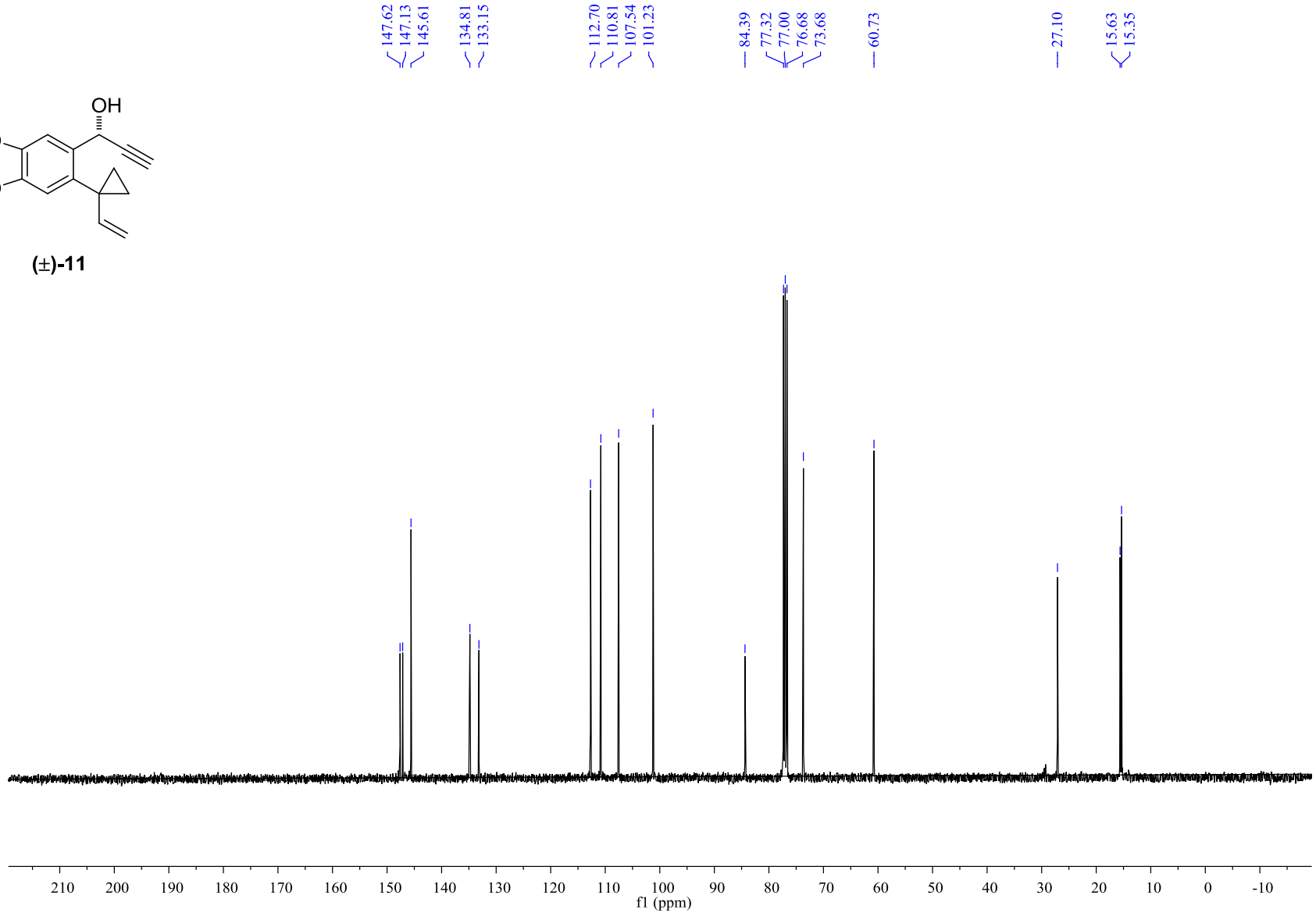
f1 (ppm)


OH
O
O

(±)-11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73.68
60.73
27.10
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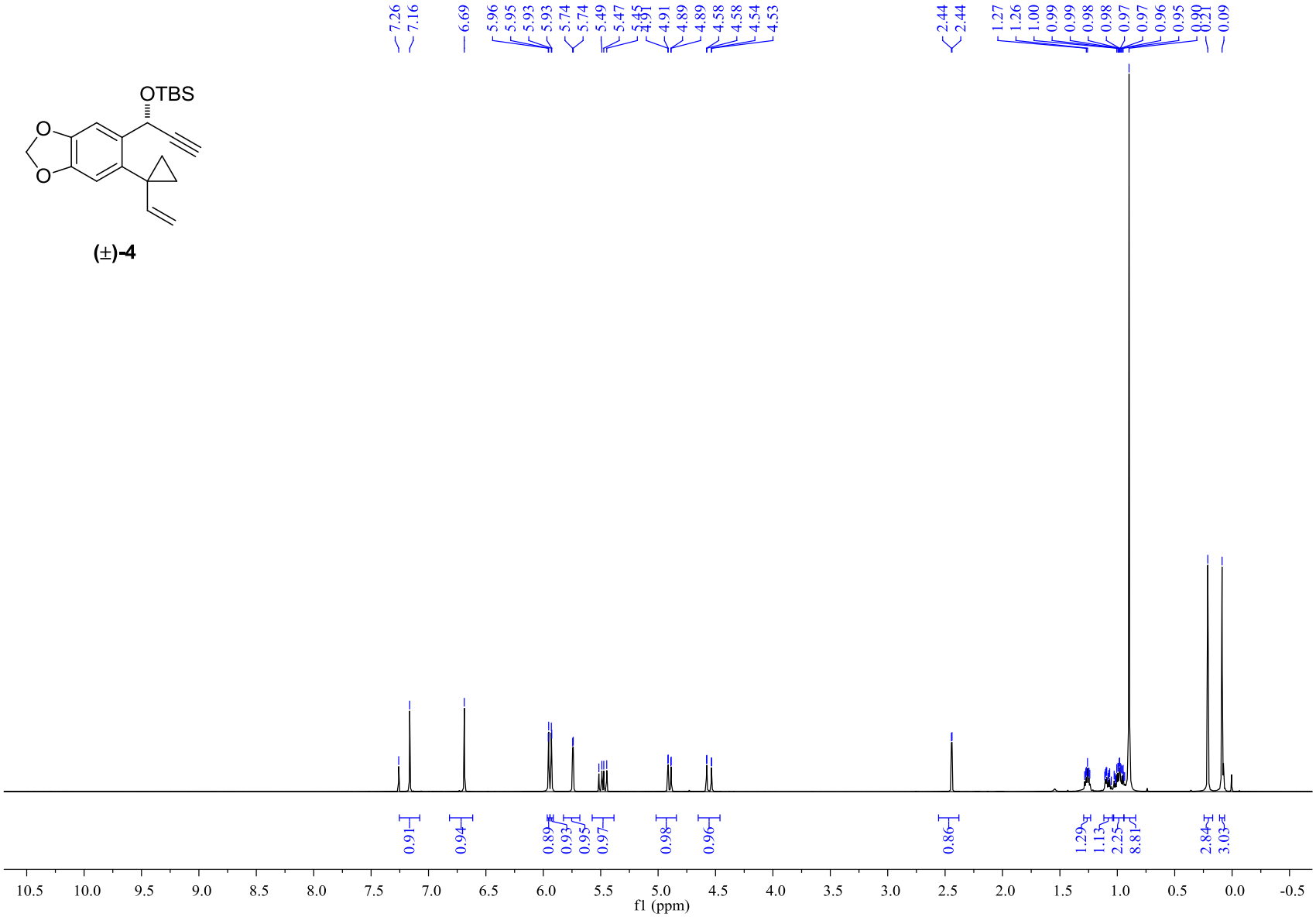
f1 (ppm)

OTBS
O
O

(±)-4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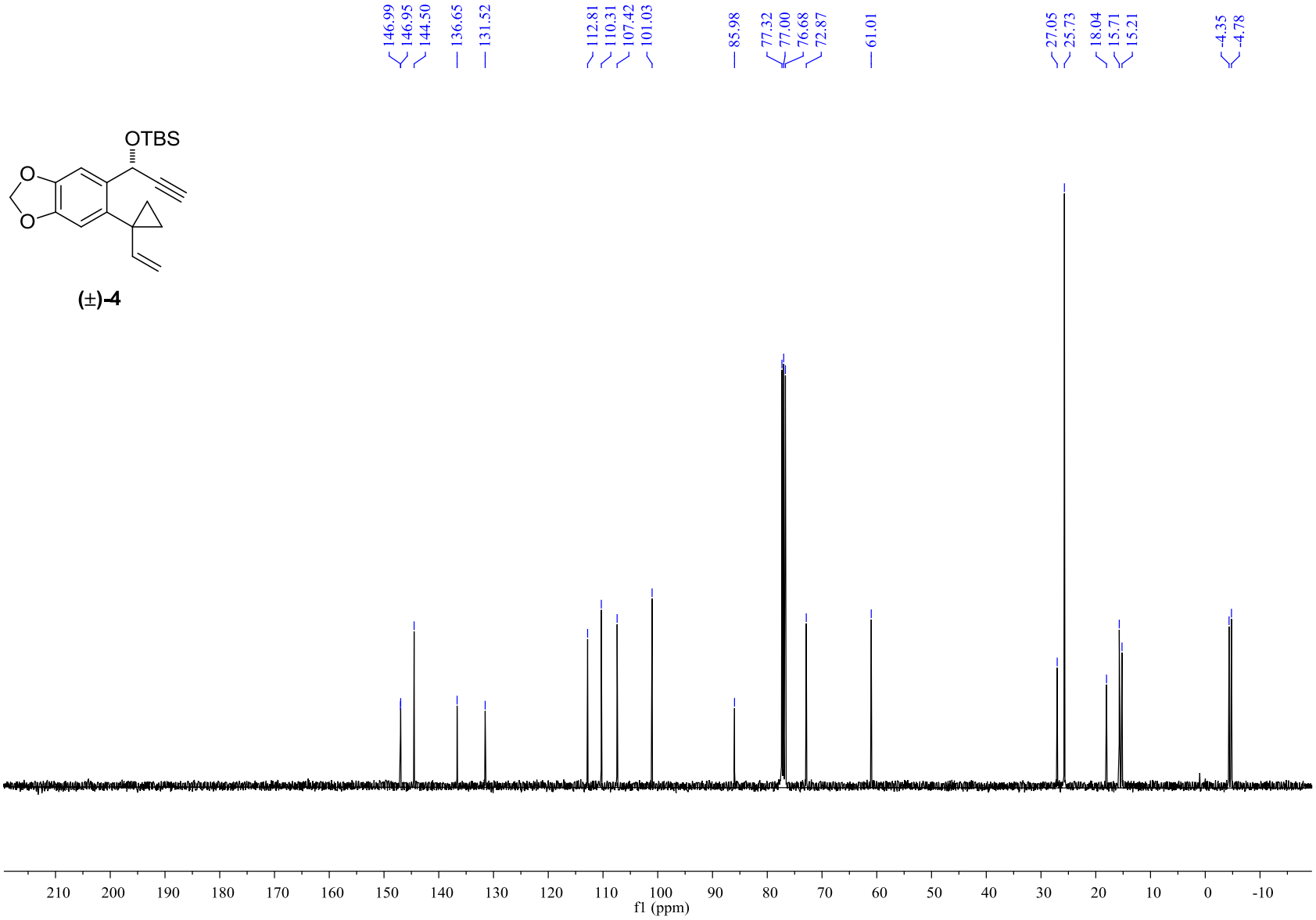
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(±)-4
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f1 (ppm)

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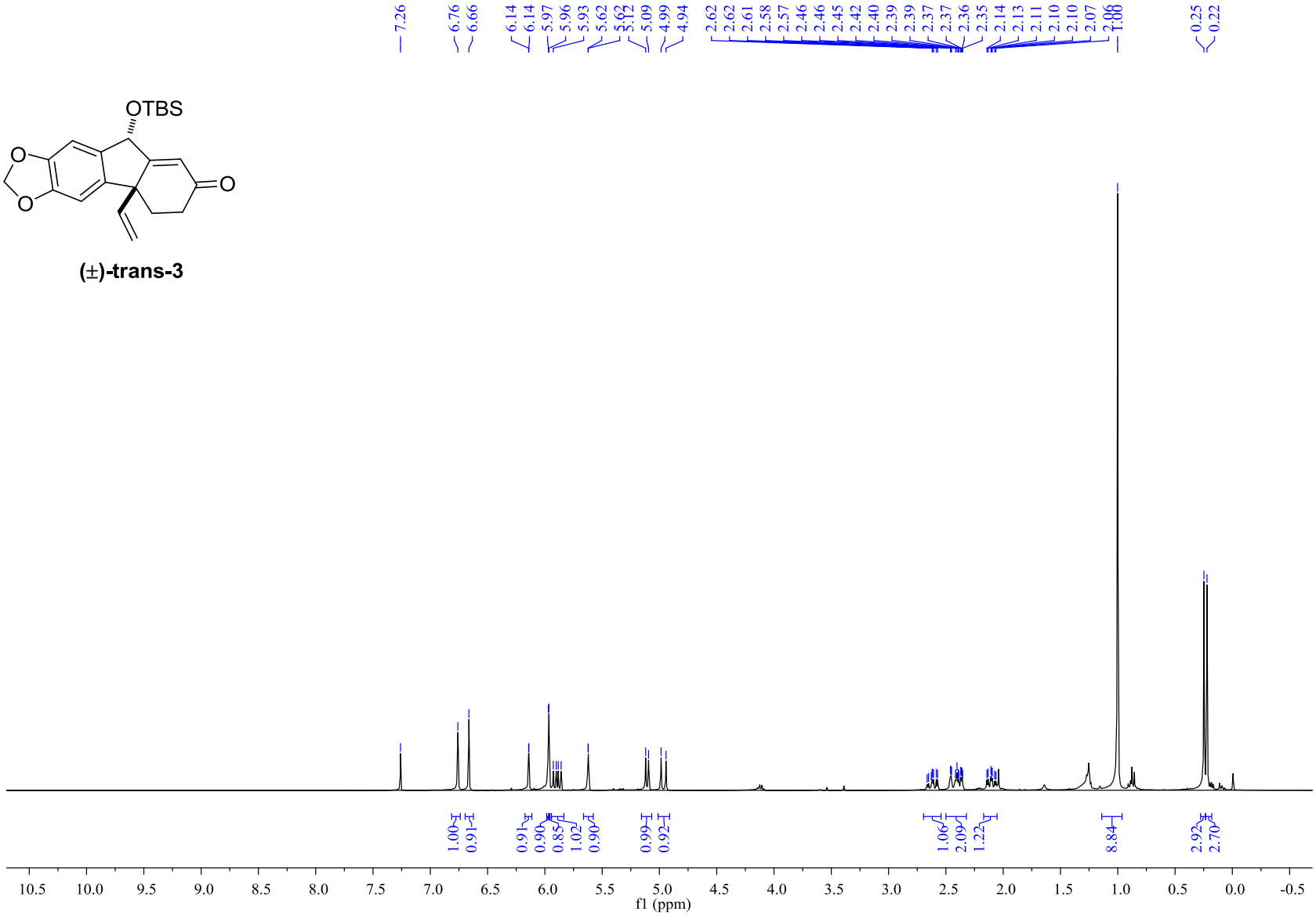
(±)-trans-3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2.35
2.14
2.13
2.11
2.10
2.10
2.07
2.06
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0.25
0.22
1.00
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0.91
0.90
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1.02
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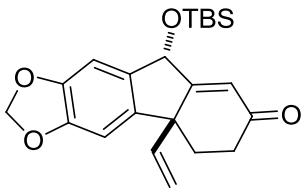
f1 (ppm)


OTBS
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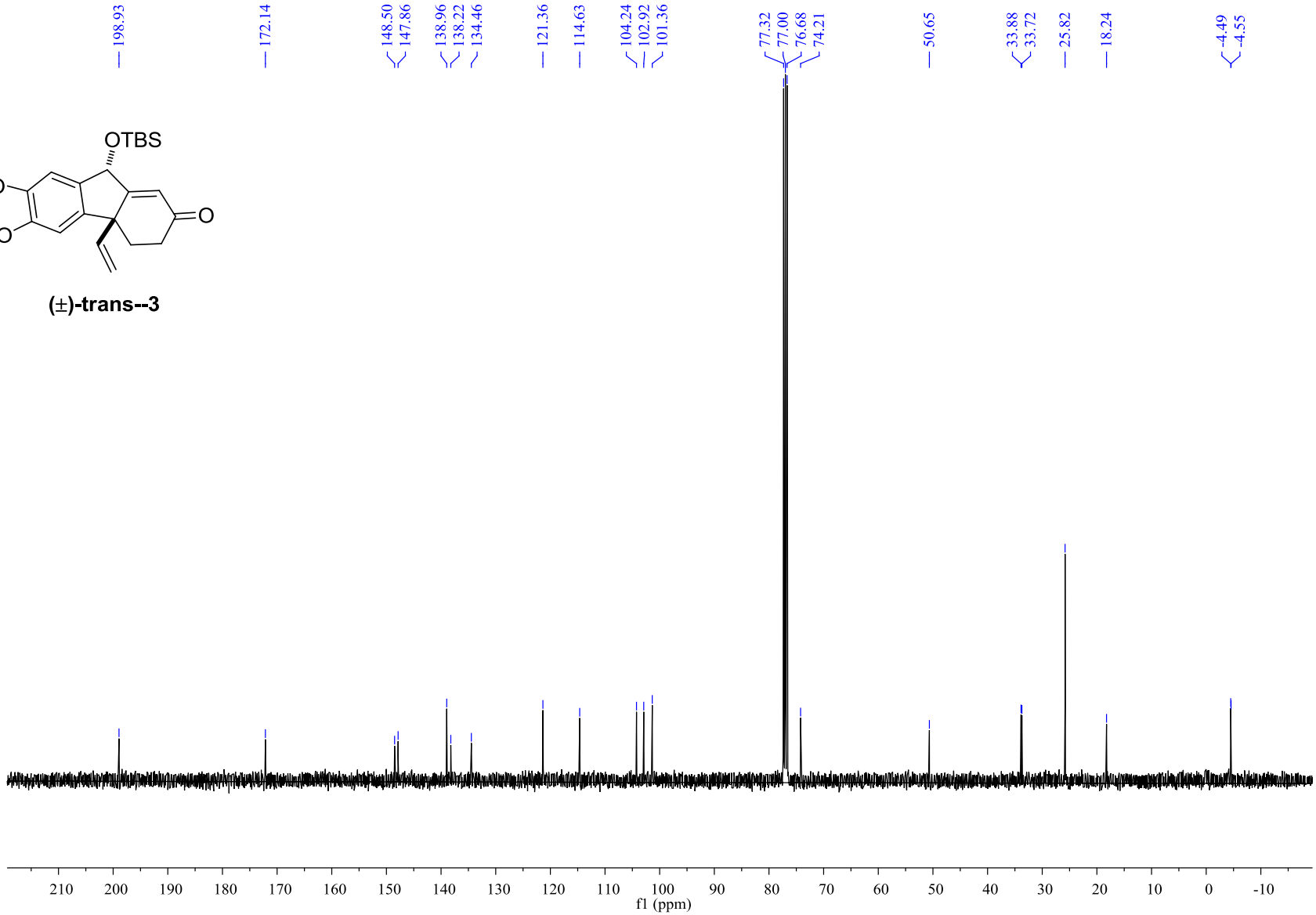
(±)-trans--3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77.32
77.00
76.68
74.21
50.65
33.88
33.72
25.82
18.24
-4.49
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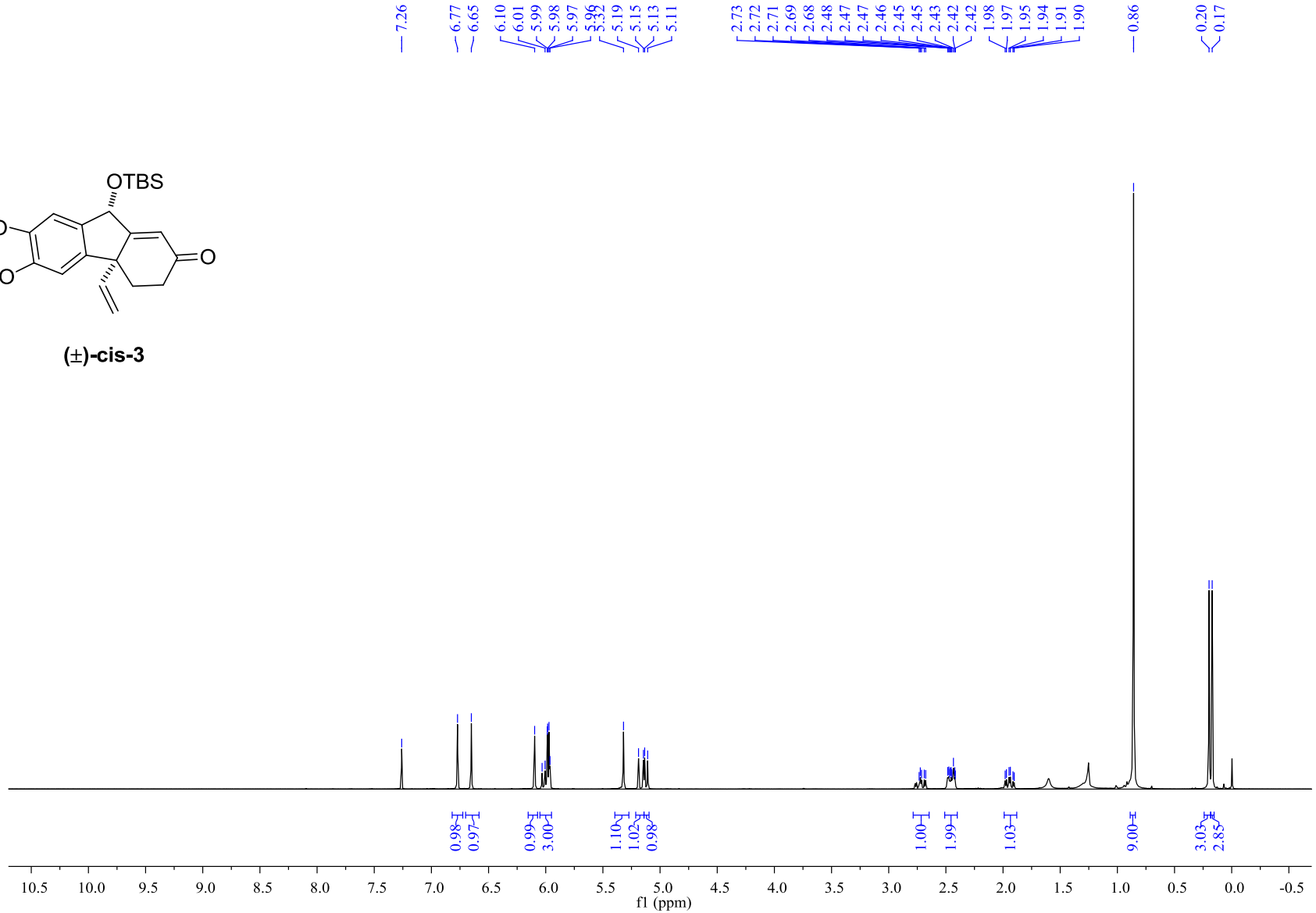
f1 (ppm)

OTBS
O
O
O

(±)-cis-3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5.13
5.11
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2.72
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2.68
2.48
2.47
2.47
2.46
2.45
2.45
2.43
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2.42
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1.97
1.95
1.94
1.91
1.90
0.86
0.20
0.17
0.98
0.97
0.99
3.00
1.10
1.02
0.98
1.00
1.99
1.03
9.00
3.03
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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
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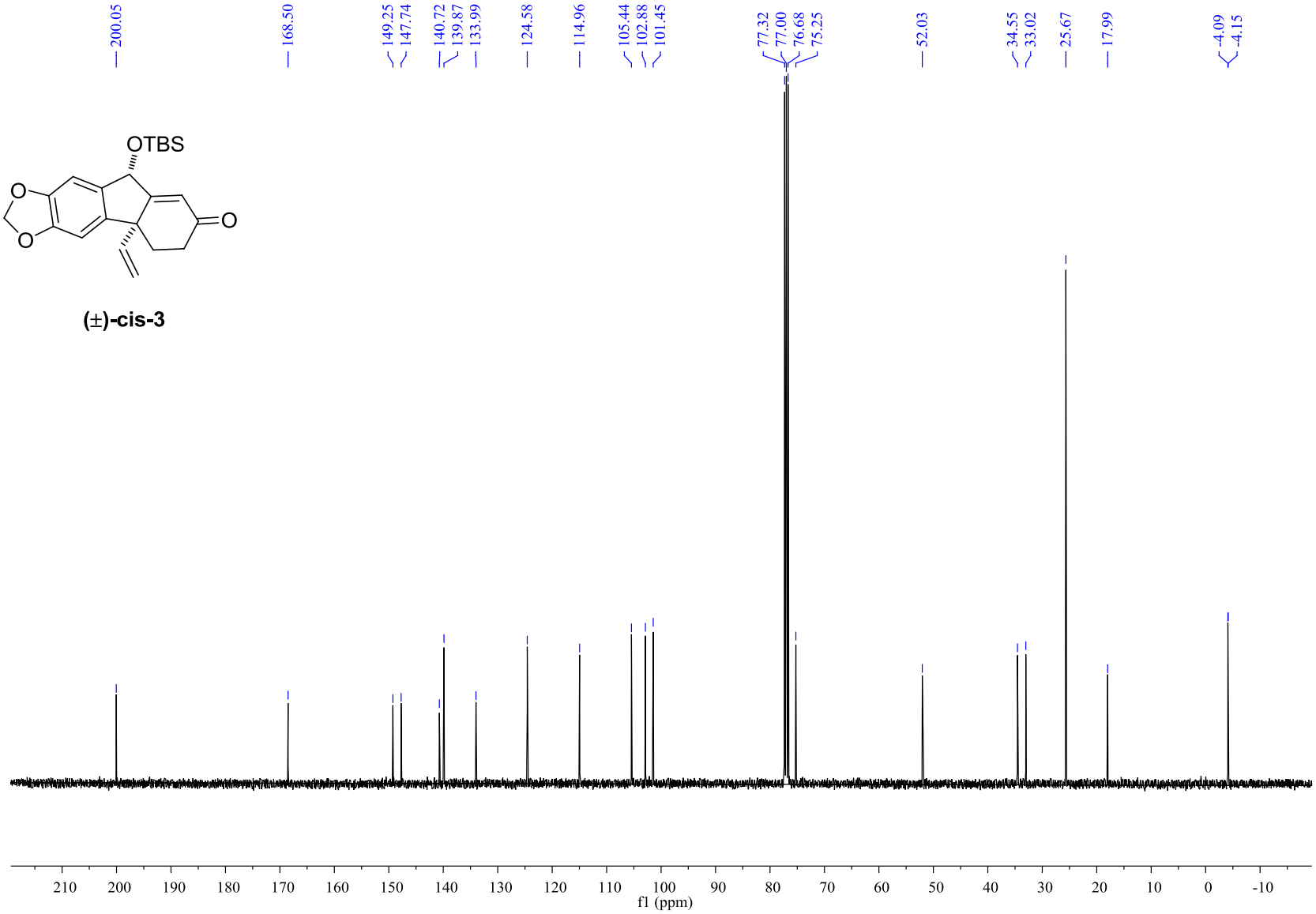
0.0
-0.5
f1 (ppm)

OTBS
O
O
O

(±)-cis-3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124.58
114.96
105.44
102.88
101.45
77.32
77.00
76.68
75.25
52.03
34.55
33.02
25.67
17.99
-4.09
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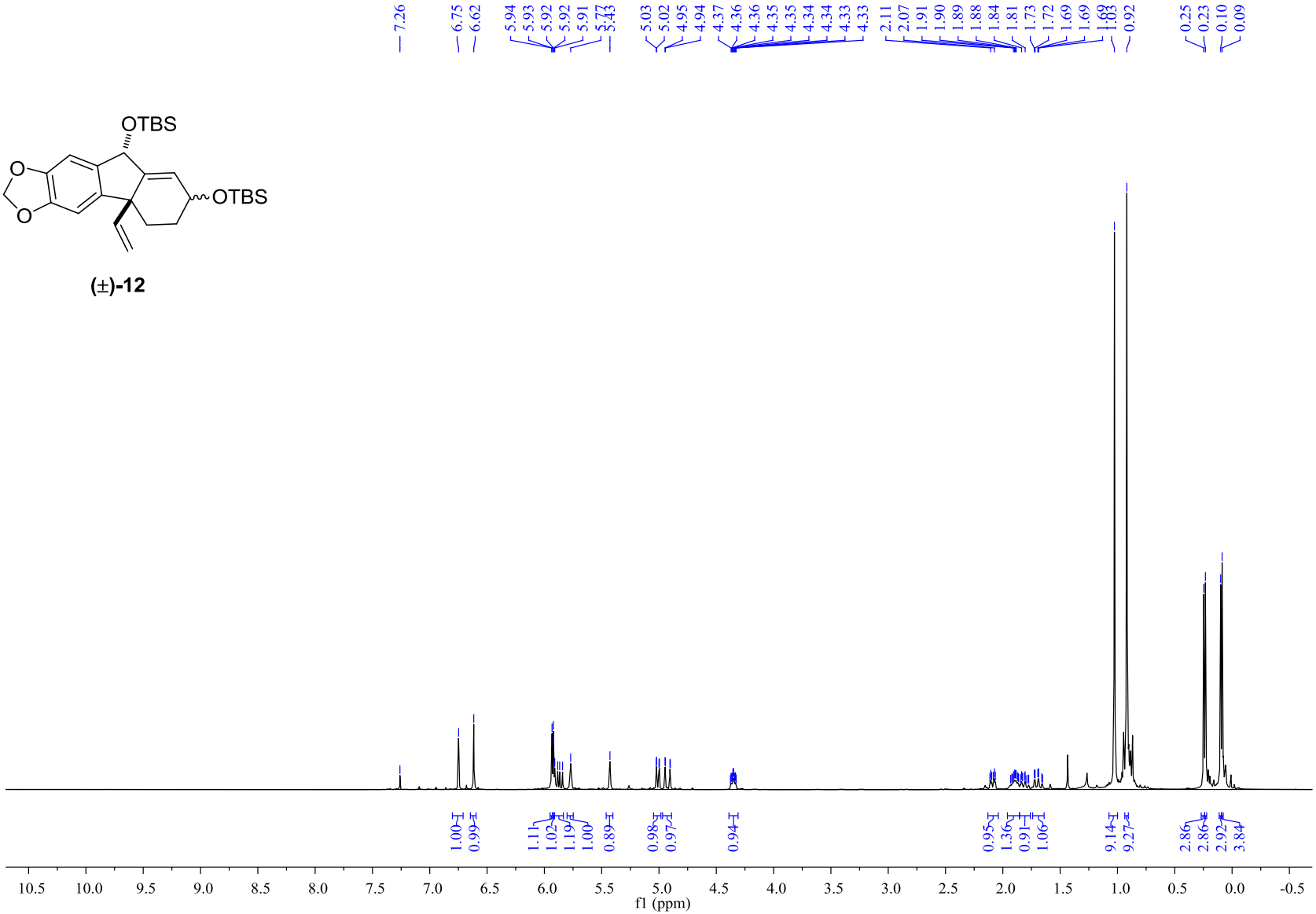
f1 (ppm)

OTBS
OTBS
O
O

(±)-12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6.75
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4.95
4.94
4.37
4.36
4.36
4.35
4.35
4.34
4.34
4.33
4.33
2.11
2.07
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1.90
1.89
1.88
1.84
1.81
1.73
1.72
1.69
1.69
1.68
1.03
0.92
0.25
0.23
0.10
0.09
1.00
0.99
1.11
1.02
1.19
1.00
0.89
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0.97
0.94
0.95
1.36
0.91
1.06
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9.27
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2.86
2.92
3.84
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9.0
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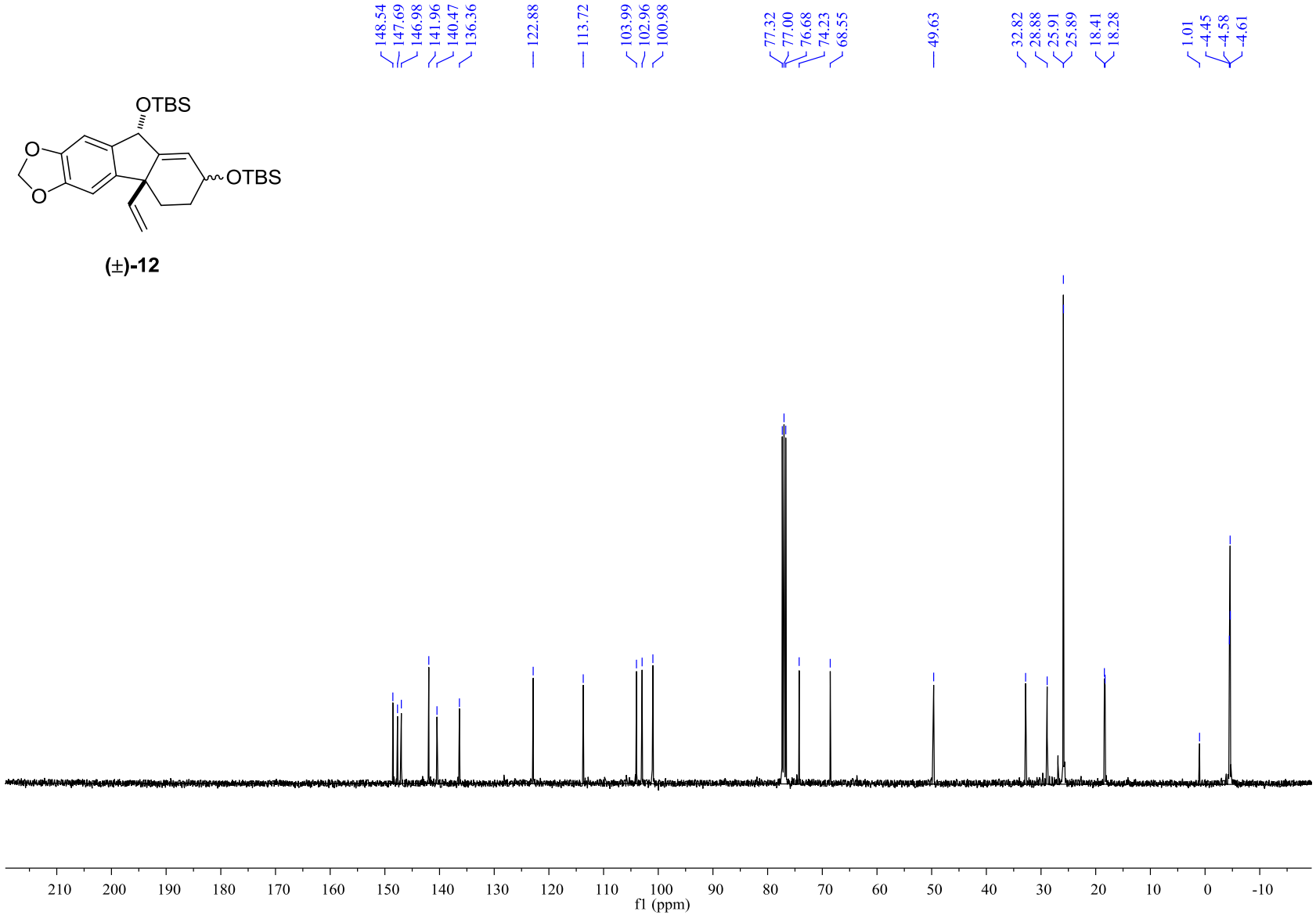
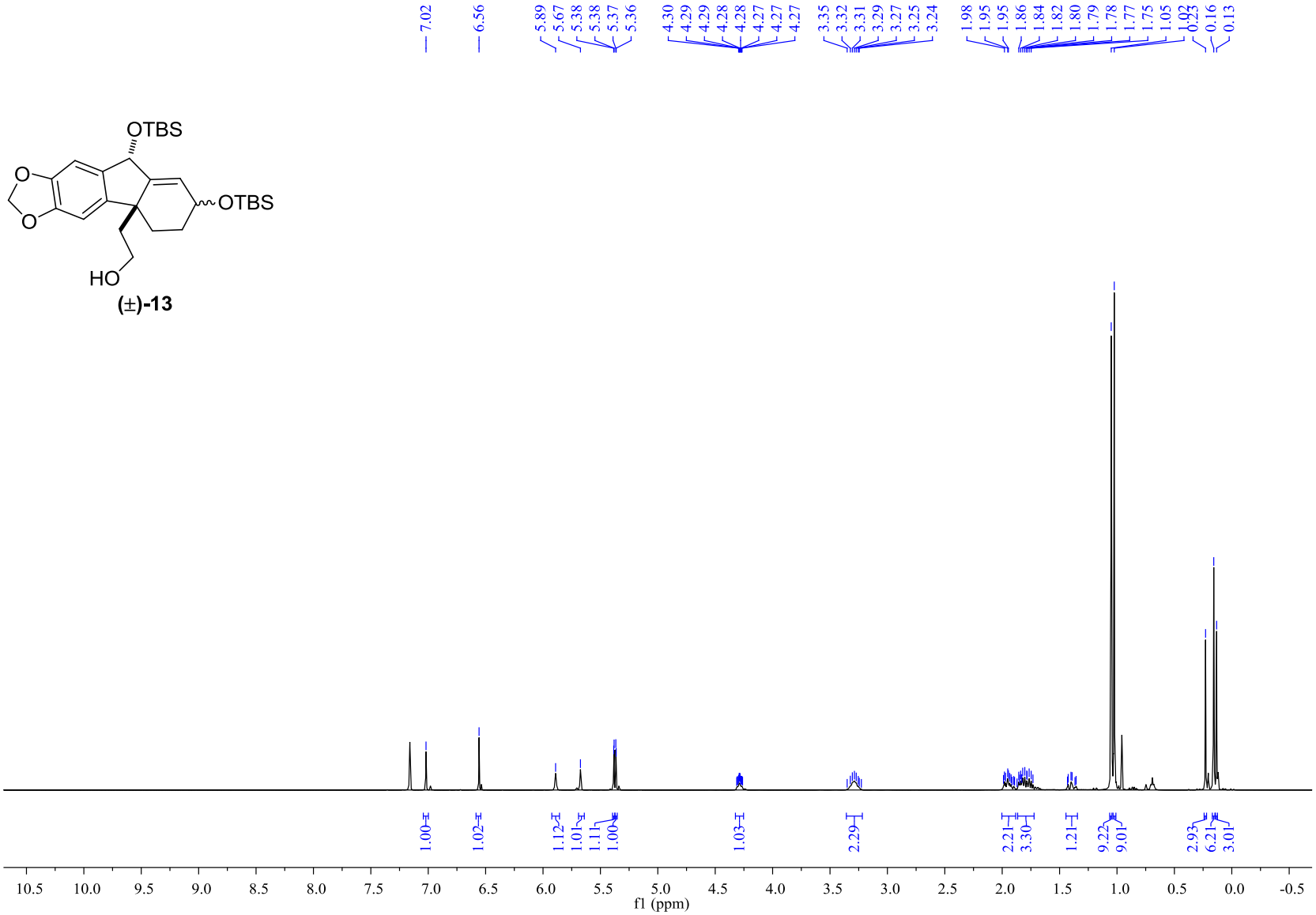
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


OTBS
OTBS
O
O
(±)-12
148.54
147.69
146.98
141.96
140.47
136.36
122.88
113.72
103.99
102.96
100.98
77.32
77.00
76.68
74.23
68.55
49.63
32.82
28.88
25.91
25.89
18.41
18.28
1.01
-4.45
-4.58
-4.61
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

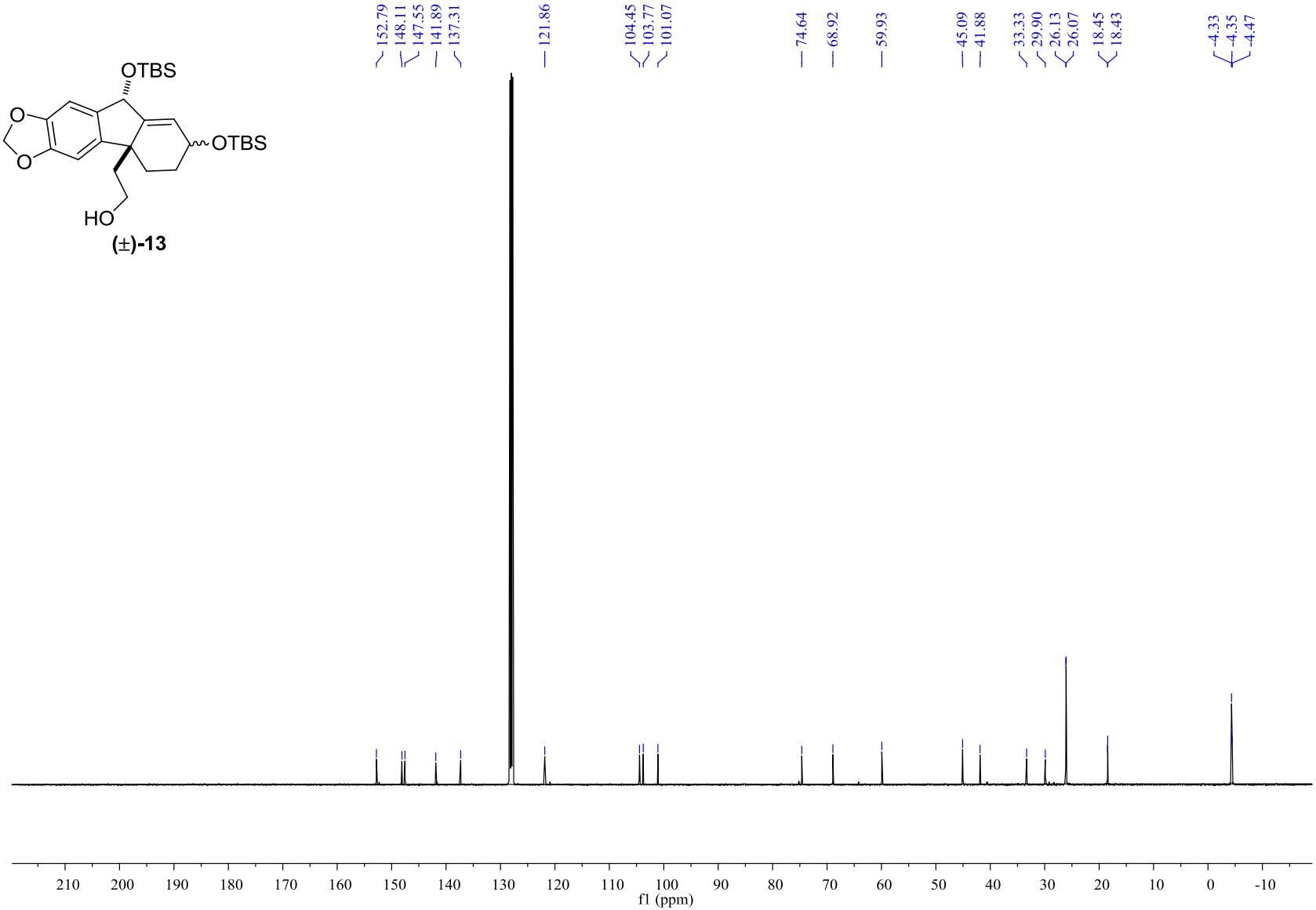
OTBS
OTBS
O
O
HO

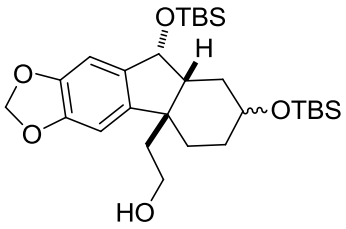
(±)-13


7.02
6.56
5.89
5.67
5.38
5.38
5.37
5.36
4.30
4.29
4.29
4.28
4.28
4.27
4.27
4.27
3.35
3.32
3.31
3.29
3.27
3.25
3.24
1.98
1.95
1.95
1.86
1.84
1.82
1.80
1.79
1.78
1.77
1.75
1.05
0.16
0.13
1.00
1.02
1.12
1.01
1.11
1.00
1.03
2.29
2.21
3.30
1.21
9.22
9.01
2.93
6.21
3.01
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)

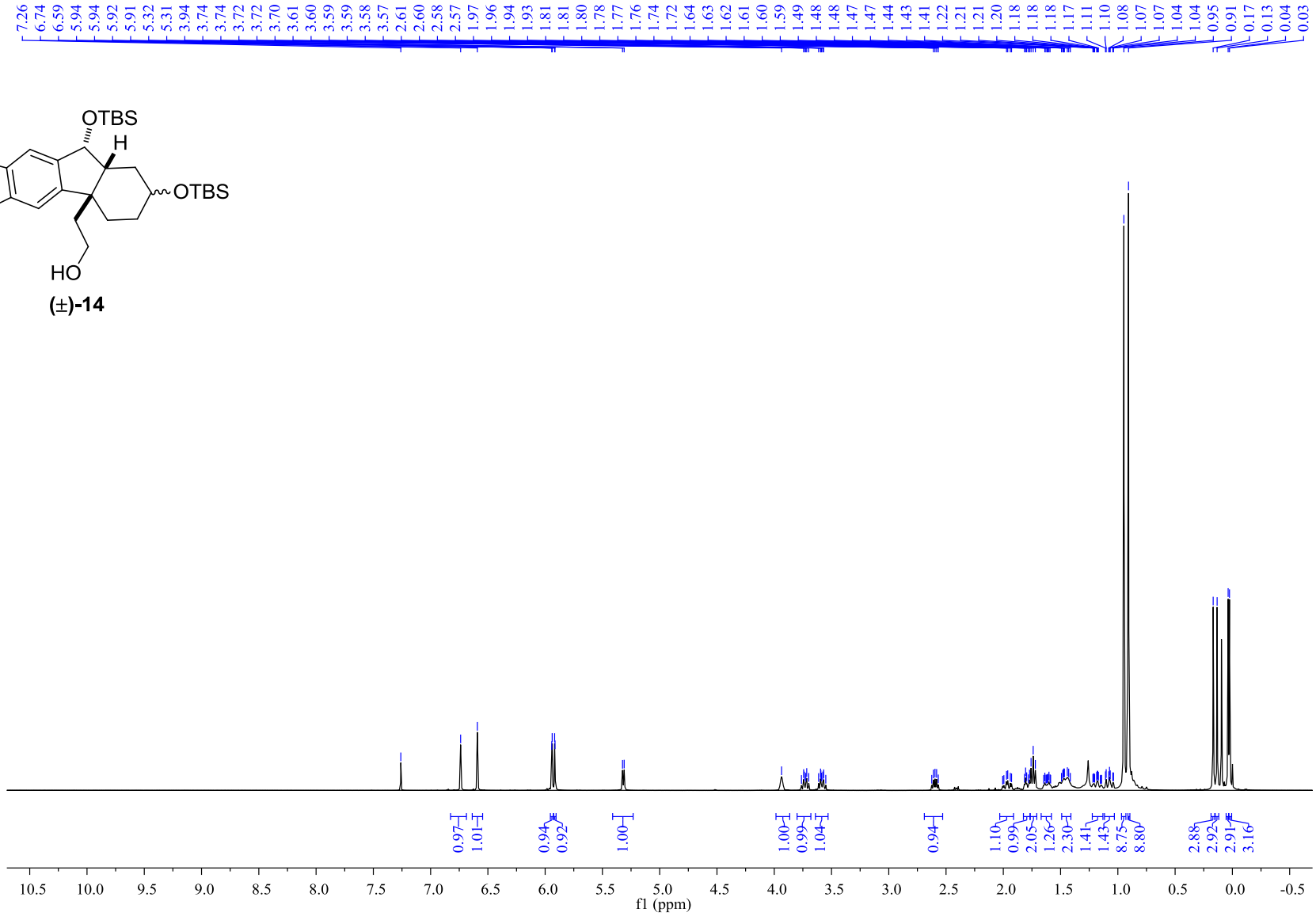
OTBS
OTBS
O
O
HO

(±)-13


152.79
148.11
147.55
141.89
137.31
121.86
104.45
103.77
101.07
74.64
68.92
59.93
45.09
41.88
33.33
29.90
26.13
26.07
18.45
18.43
-4.33
-4.35
-4.47
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

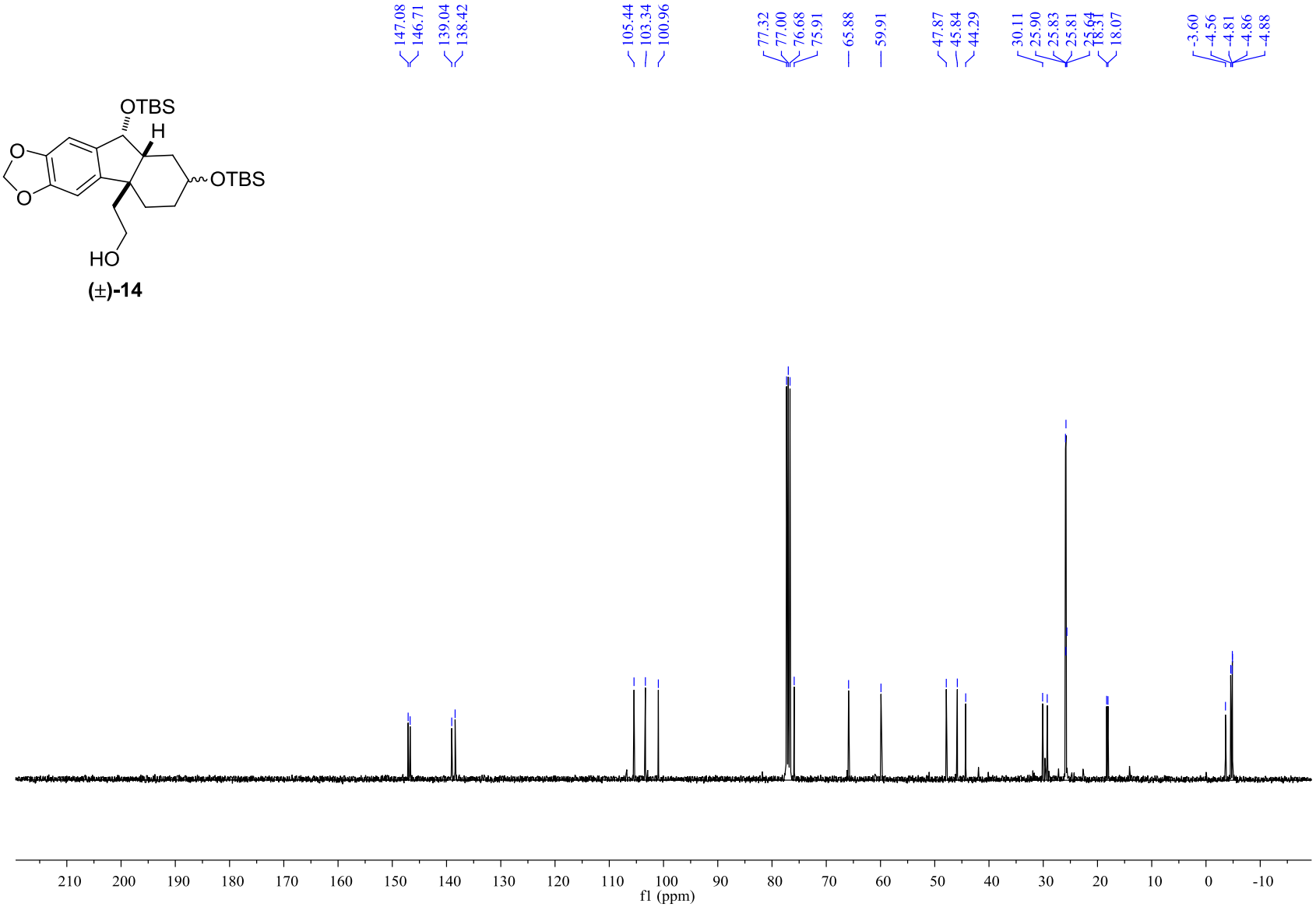

OTBS
H
OTBS
O
O
HO

(±)-14


7.26 6.74 6.59 5.94 5.94 5.92 5.91 5.32 5.31 3.94 3.74 3.74 3.72 3.72 3.70 3.61 3.60 3.59 3.59 3.58 3.57 2.61 2.60 2.58 2.57 1.97 1.96 1.94 1.93 1.81 1.81 1.80 1.78 1.77 1.76 1.74 1.72 1.64 1.63 1.62 1.61 1.60 1.59 1.49 1.48 1.48 1.47 1.47 1.44 1.43 1.41 1.22 1.21 1.21 1.20 1.18 1.18 1.18 1.17 1.11 1.10 1.08 1.07 1.07 1.04 1.04 0.95 0.91 0.17 0.13 0.04 0.03
0.97 1.01 0.94 0.92 1.00 1.00 0.99 1.04 0.94 1.10 0.99 2.05 1.26 2.30 1.41 1.43 8.75 8.80 2.88 2.92 2.91 3.16
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)

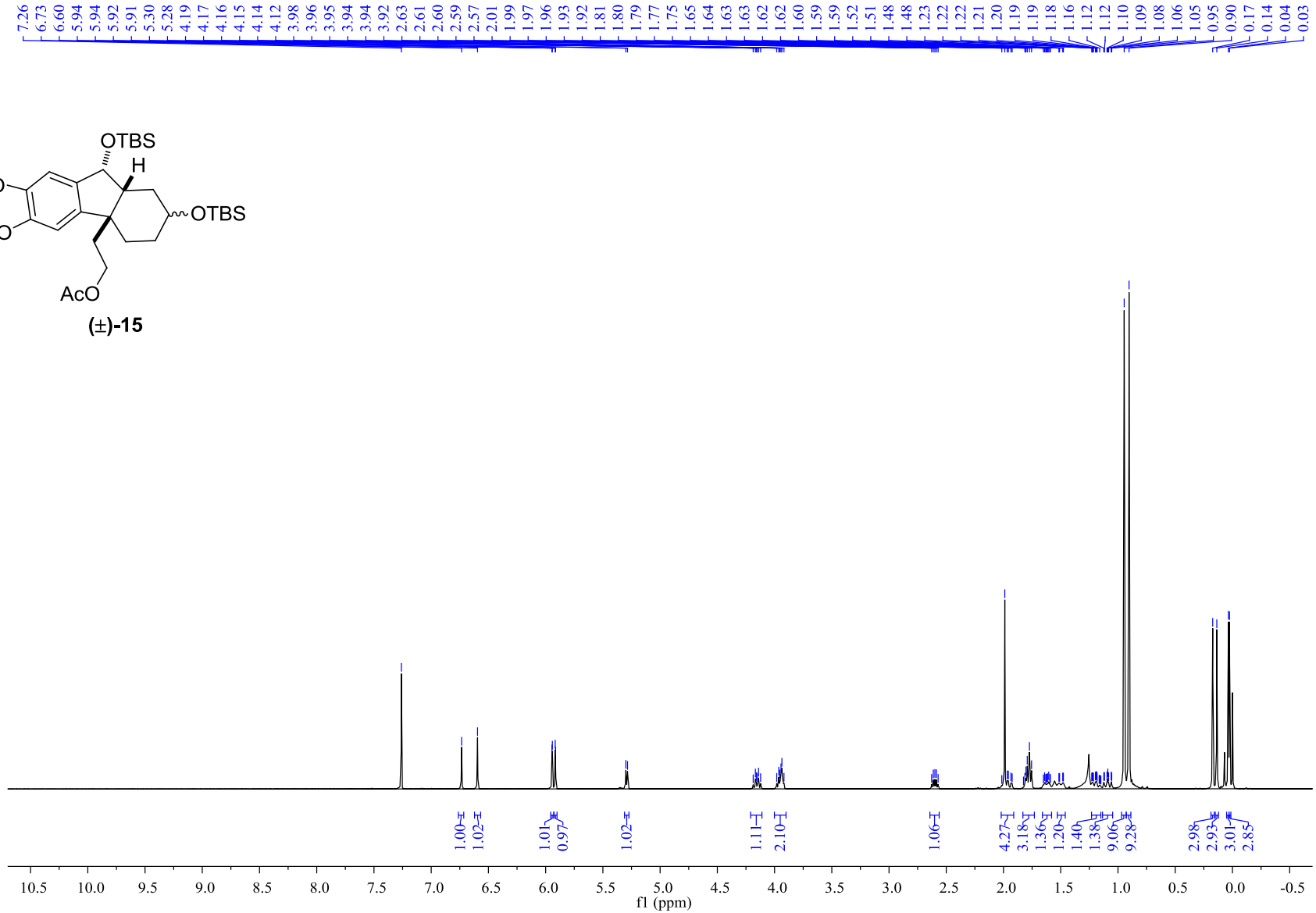
OTBS
H
OTBS
O
O
HO

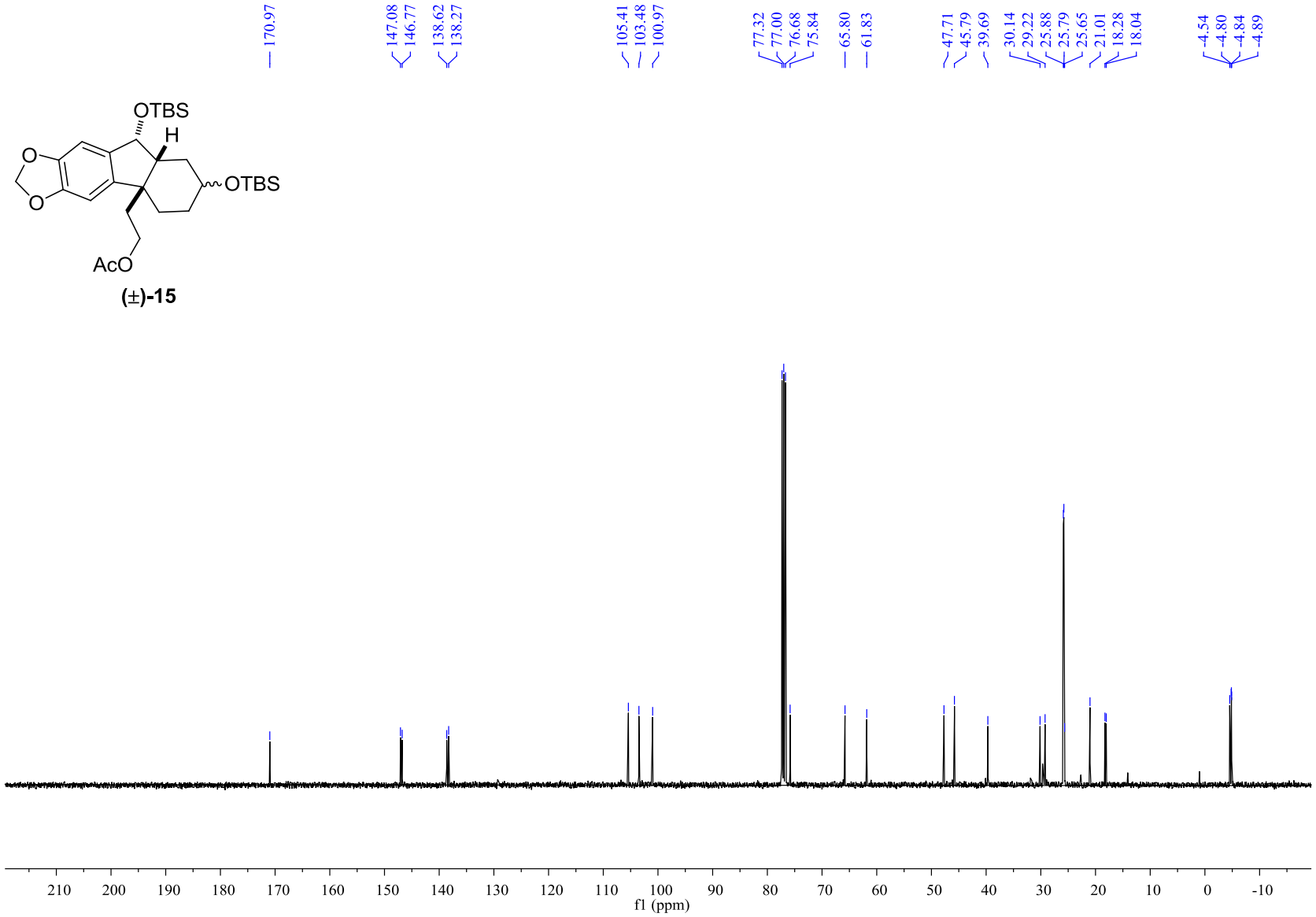
(±)-14

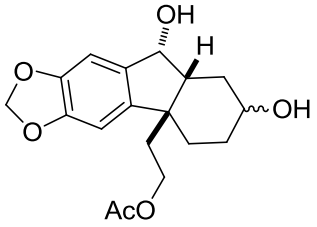

147.08
146.71
139.04
138.42
105.44
103.34
100.96
77.32
77.00
76.68
75.91
65.88
59.91
47.87
45.84
44.29
30.11
25.90
25.83
25.81
25.64
18.31
18.07
-3.60
-4.56
-4.81
-4.86
-4.88
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

OTBS
H
OTBS
O
O
AcO

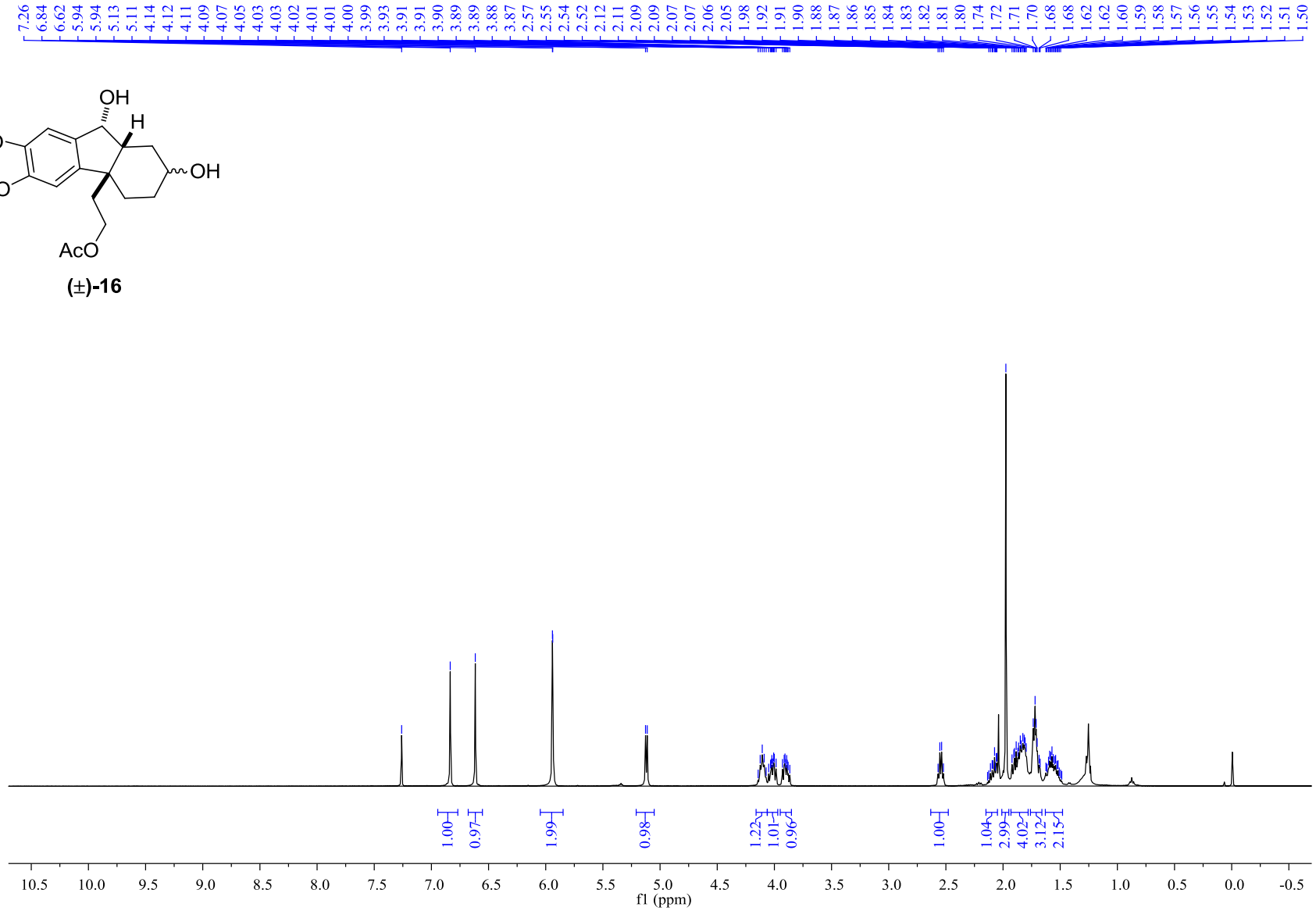
(±)-15

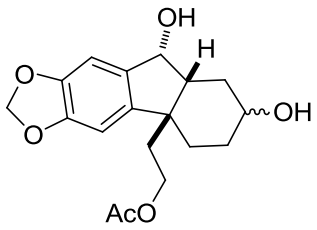

7.26 6.73 6.60 5.94 5.94 5.92 5.91 5.30 5.28 4.19 4.17 4.16 4.15 4.14 4.12 3.98 3.96 3.95 3.94 3.94 3.92 2.63 2.61 2.60 2.59 2.57 2.01 1.99 1.97 1.96 1.93 1.92 1.81 1.80 1.79 1.77 1.75 1.65 1.64 1.63 1.63 1.62 1.62 1.60 1.59 1.59 1.52 1.51 1.48 1.48 1.23 1.22 1.22 1.21 1.20 1.19 1.19 1.18 1.16 1.12 1.12 1.10 1.09 1.08 1.06 1.05 0.95 0.90 0.17 0.14 0.04 0.03
1.00 1.02 1.01 0.97 1.02 1.11 2.10 1.06 4.27 3.18 1.36 1.20 1.40 1.38 9.06 9.28 2.98 2.93 3.01 2.85
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)


170.97
147.08
146.77
138.62
138.27
105.41
103.48
100.97
77.32
77.00
76.68
75.84
65.80
61.83
47.71
45.79
39.69
30.14
29.22
25.88
25.79
25.65
21.01
18.28
18.04
-4.54
-4.80
-4.84
-4.89
OTBS
H
OTBS
O
O
AcO
(±)-15
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

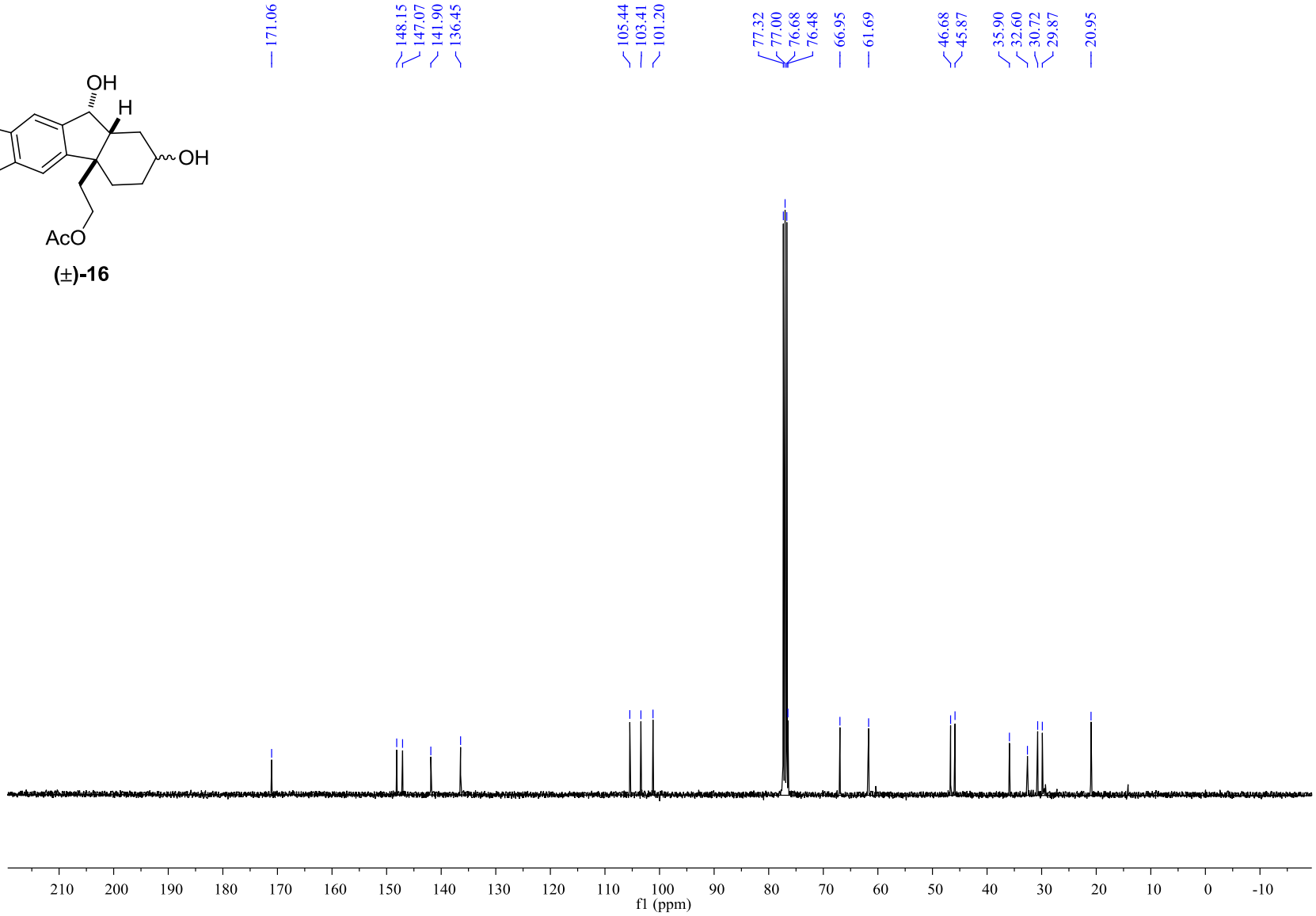

OH
H
O
O
OH
AcO

(±)-16

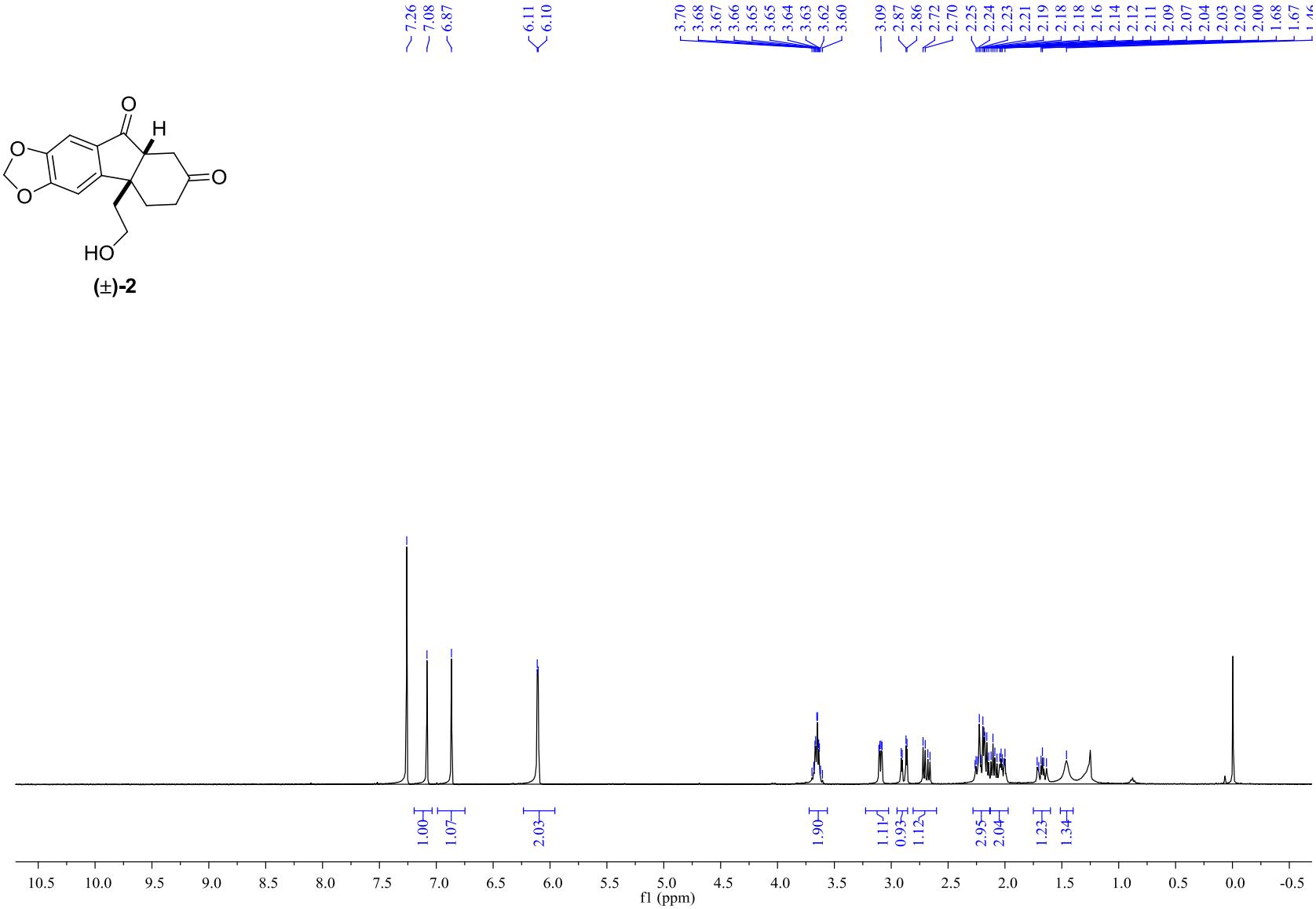

7.26 6.84 6.62 5.94 5.94 5.13 5.11 4.14 4.12 4.11 4.09 4.07 4.05 4.03 4.03 4.02 4.01 4.01 4.00 3.99 3.93 3.91 3.91 3.90 3.89 3.89 3.88 3.87 2.57 2.55 2.54 2.52 2.12 2.11 2.09 2.09 2.07 2.07 2.06 2.05 1.98 1.92 1.91 1.90 1.88 1.87 1.86 1.85 1.84 1.83 1.82 1.81 1.80 1.74 1.72 1.71 1.70 1.68 1.68 1.62 1.62 1.60 1.59 1.58 1.57 1.56 1.55 1.54 1.53 1.52 1.51 1.50
1.00 0.97 1.99 0.98 1.22 1.01 0.96 1.00 1.04 2.99 4.02 3.12 2.15
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)

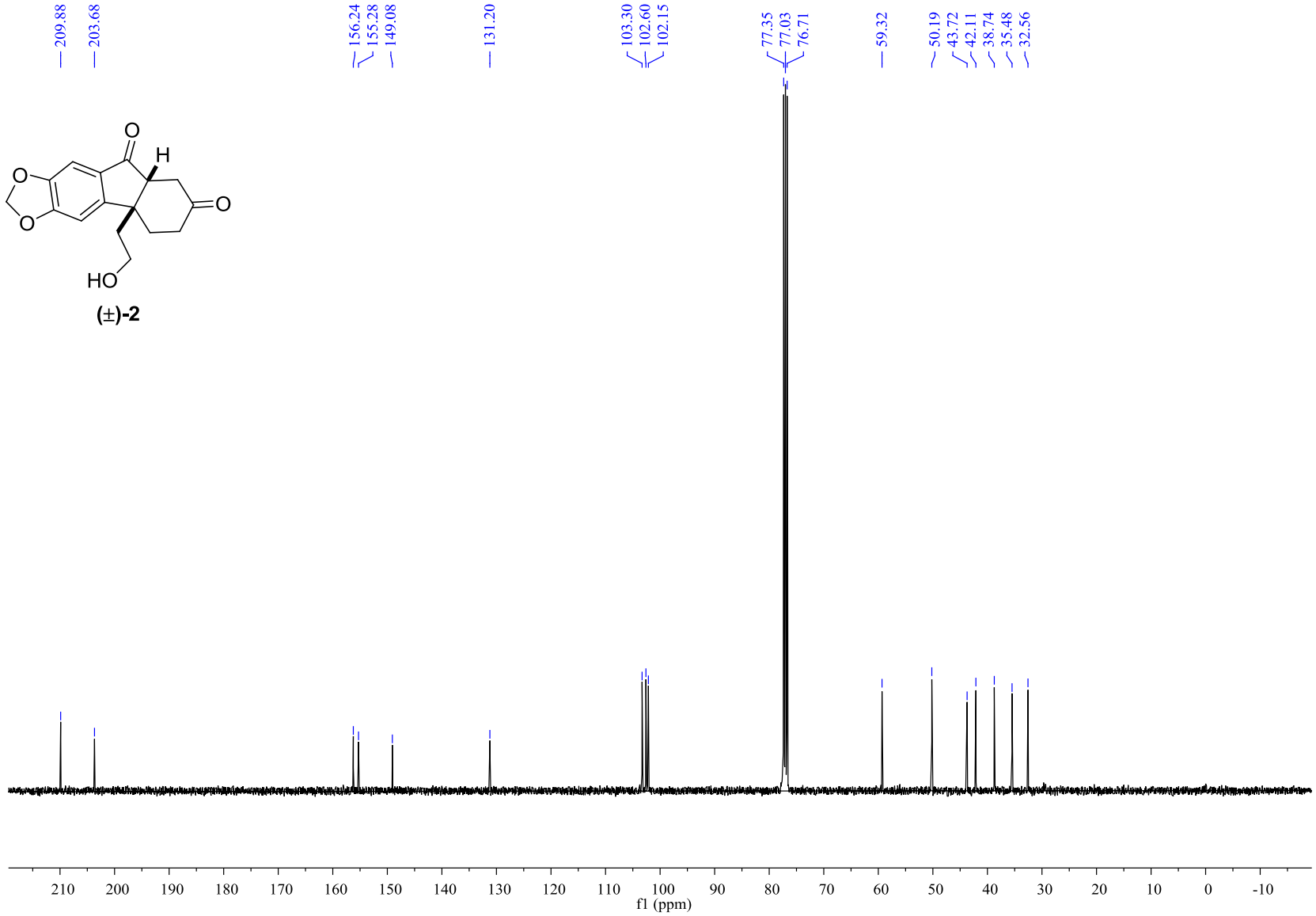

OH
H
O
O
OH
AcO

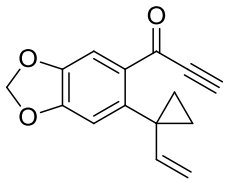
(±)-16


171.06
148.15
147.07
141.90
136.45
105.44
103.41
101.20
77.32
77.00
76.68
76.48
66.95
61.69
46.68
45.87
35.90
32.60
30.72
29.87
20.95
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

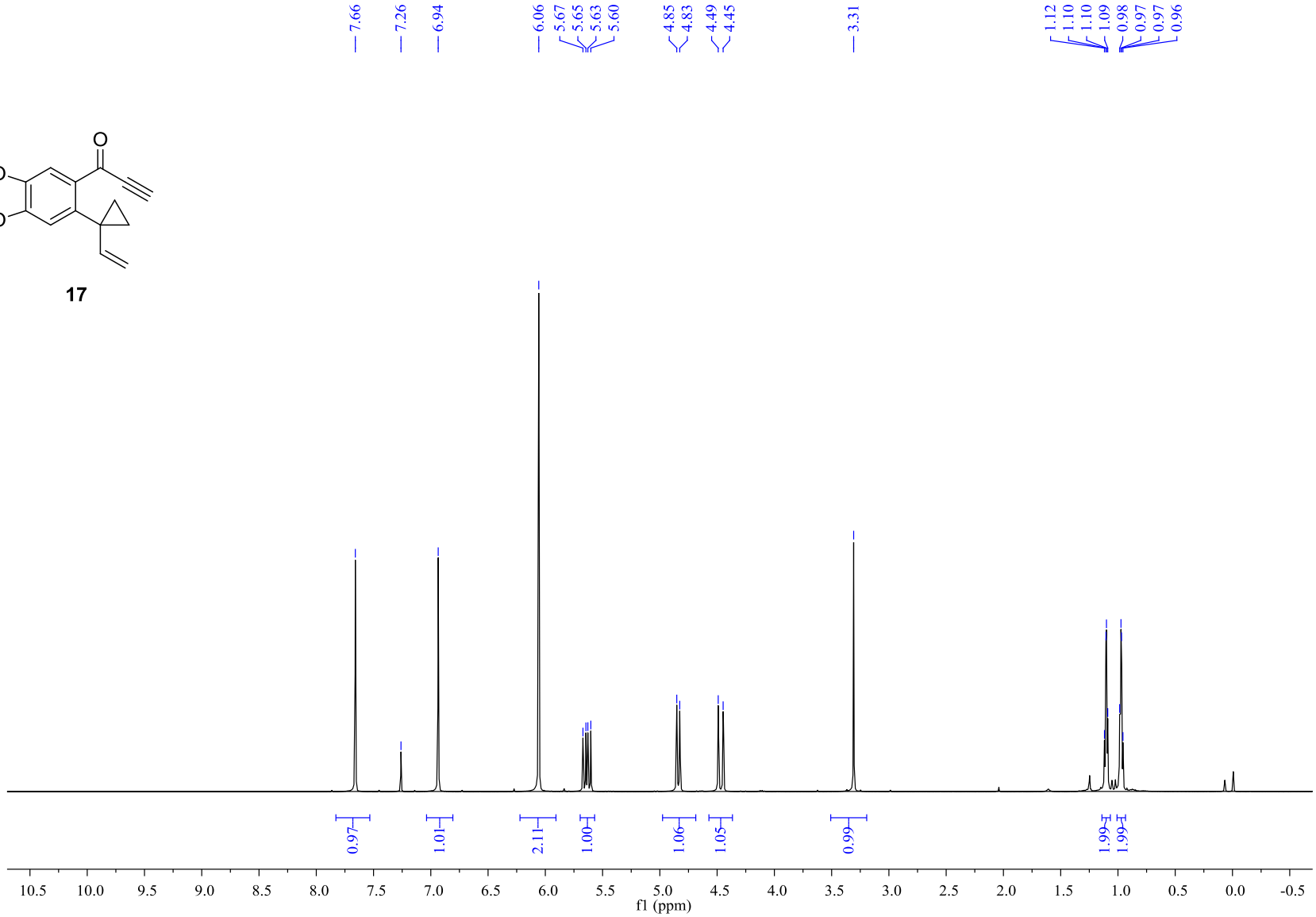
(±)-2

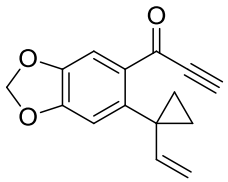

7.26
7.08
6.87
6.11
6.10
3.70
3.68
3.67
3.66
3.65
3.65
3.64
3.63
3.62
3.60
3.09
2.87
2.86
2.72
2.70
2.25
2.24
2.23
2.21
2.19
2.18
2.18
2.16
2.14
2.12
2.11
2.09
2.07
2.04
2.03
2.02
2.00
1.68
1.67
1.00
1.07
2.03
1.90
1.11
0.93
1.12
2.95
2.04
1.23
1.34
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


209.88
203.68
156.24
155.28
149.08
131.20
103.30
102.60
102.15
77.35
77.03
76.71
59.32
50.19
43.72
42.11
38.74
35.48
32.56
O
H
O
O
O
HO
(±)-2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

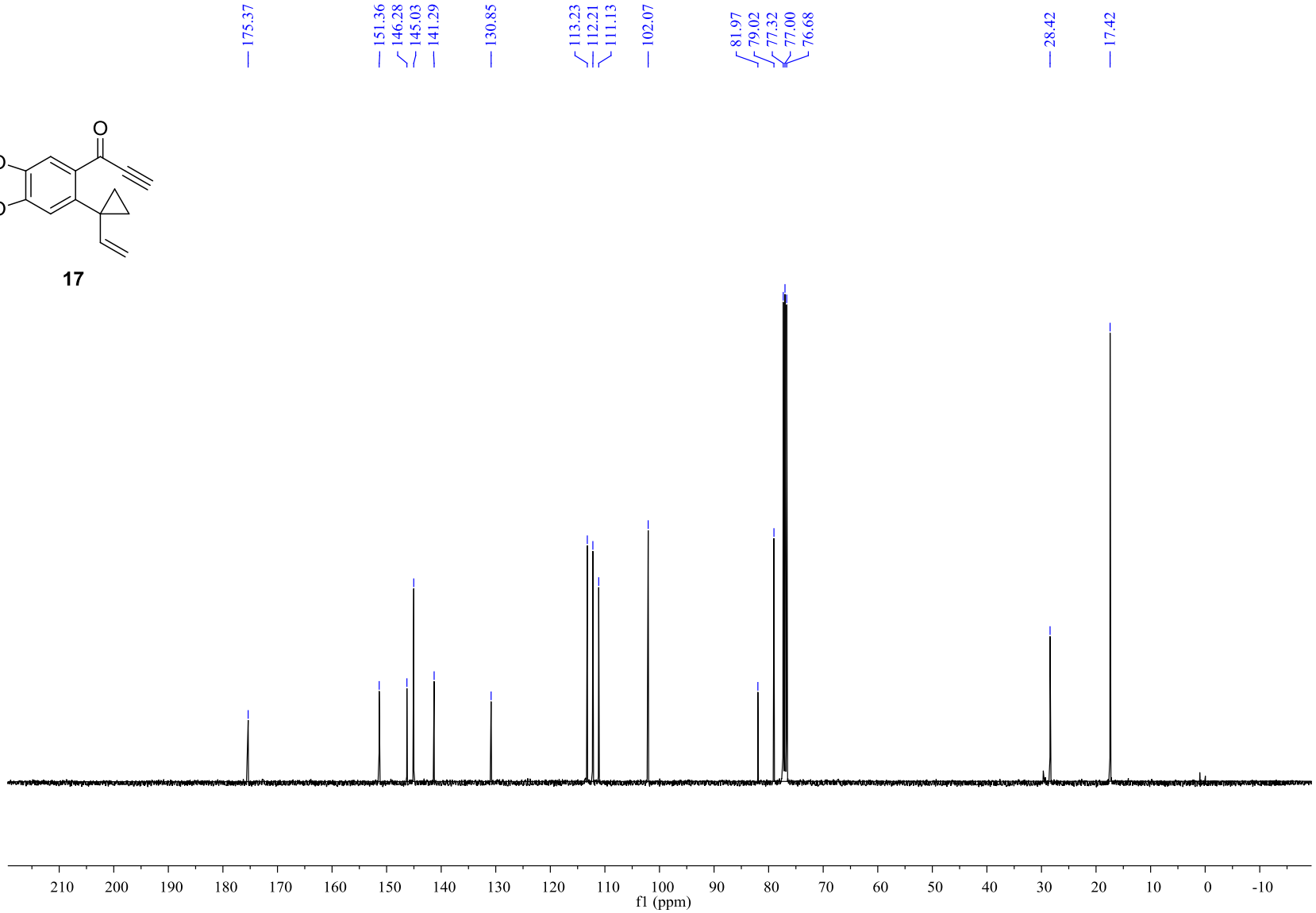


17


7.66
7.26
6.94
6.06
5.67
5.65
5.63
5.60
4.85
4.83
4.49
4.45
3.31
1.12
1.10
1.10
1.09
0.98
0.97
0.97
0.96
0.97
1.01
2.11
1.00
1.06
1.05
0.99
1.99
1.99
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)

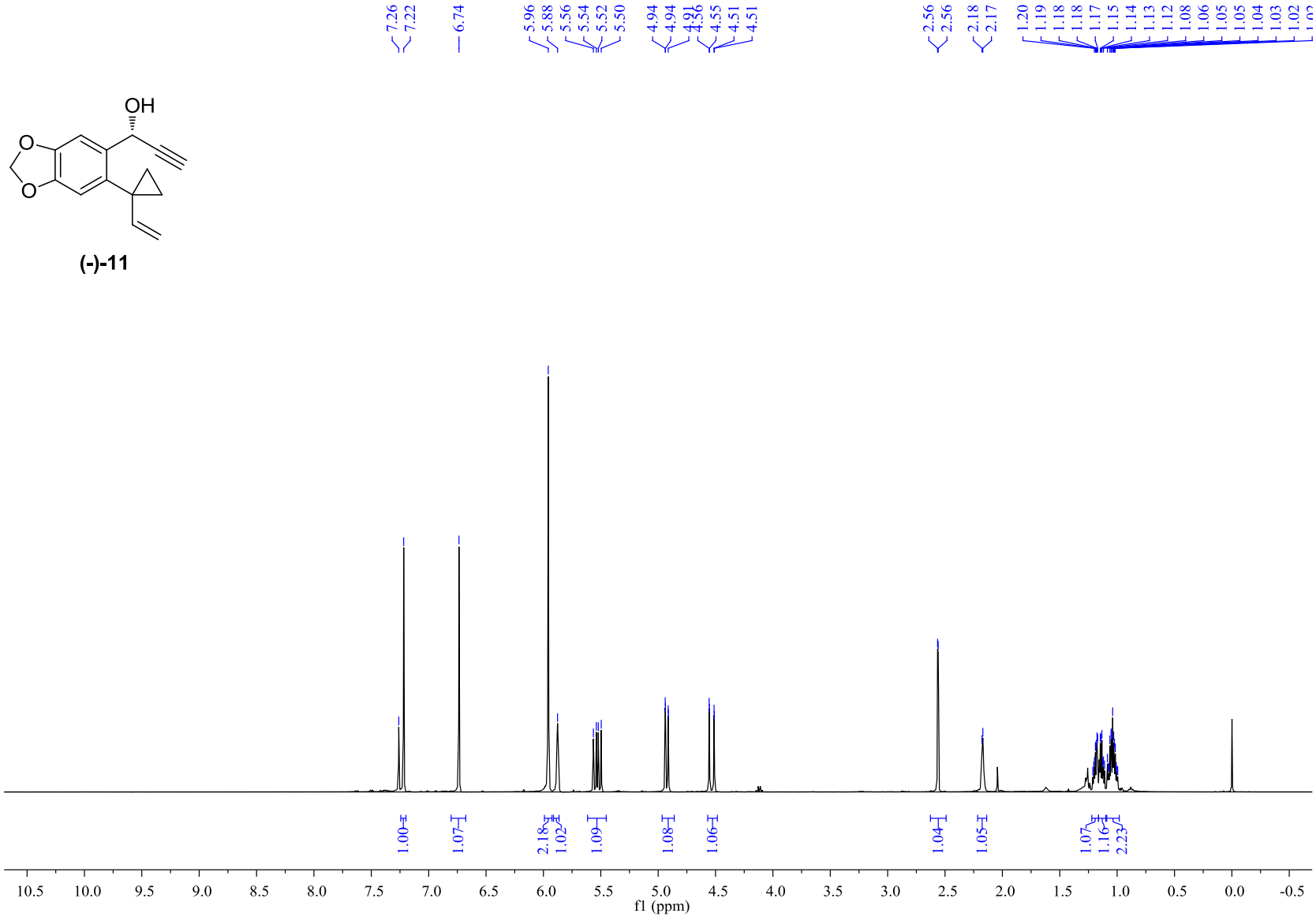


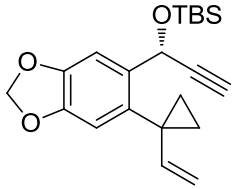
17


175.37
151.36
146.28
145.03
141.29
130.85
113.23
112.21
111.13
102.07
81.97
79.02
77.32
77.00
76.68
28.42
17.42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

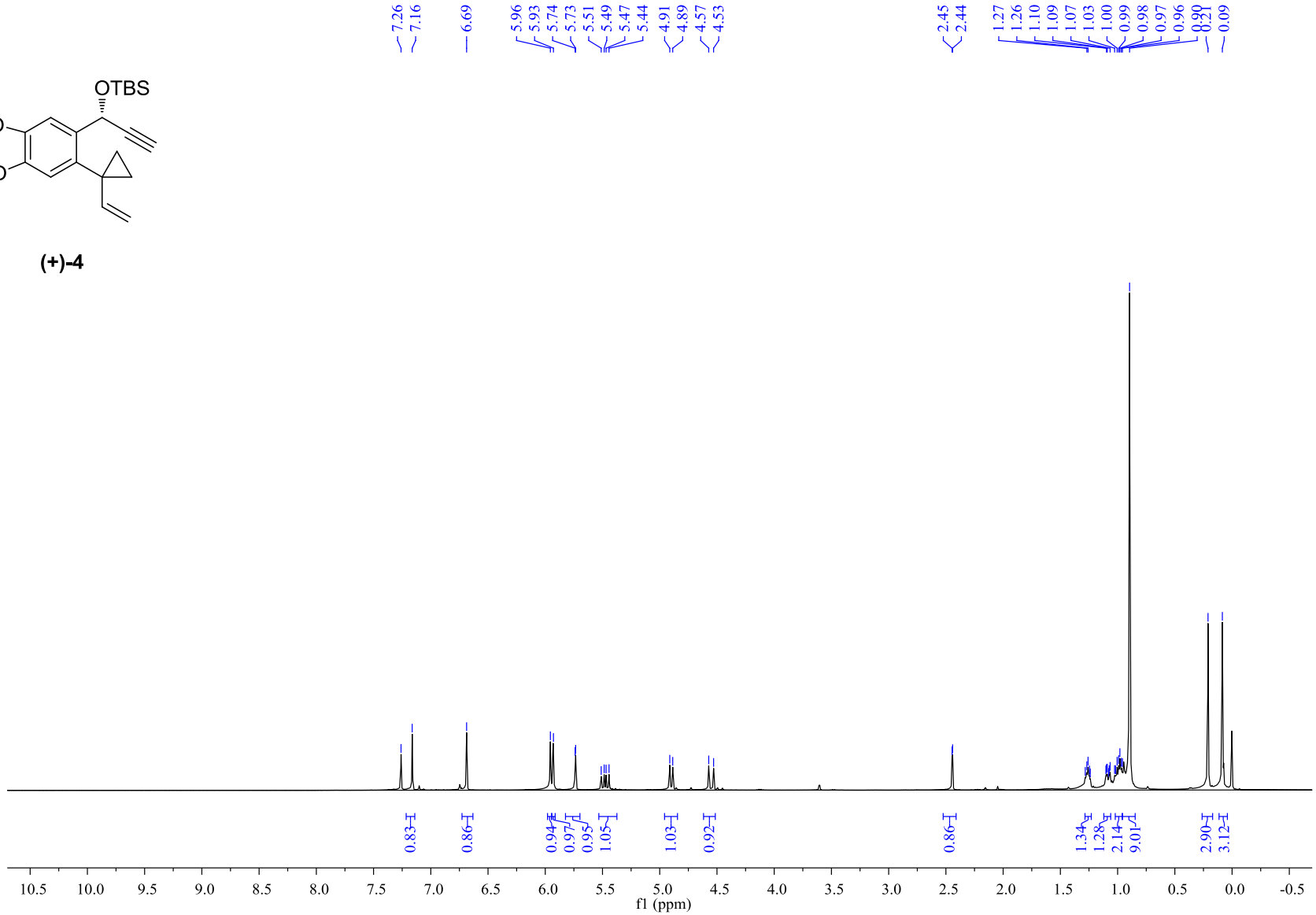
OH
O
O

(-)-11


7.26
7.22
6.74
5.96
5.88
5.56
5.54
5.52
5.50
4.94
4.94
4.91
4.56
4.55
4.51
4.51
2.56
2.56
2.18
2.17
1.20
1.19
1.18
1.18
1.17
1.15
1.14
1.13
1.12
1.08
1.06
1.05
1.05
1.04
1.03
1.02
1.00
1.07
2.18
1.02
1.09
1.08
1.06
1.04
1.05
1.07
1.16
2.23
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)

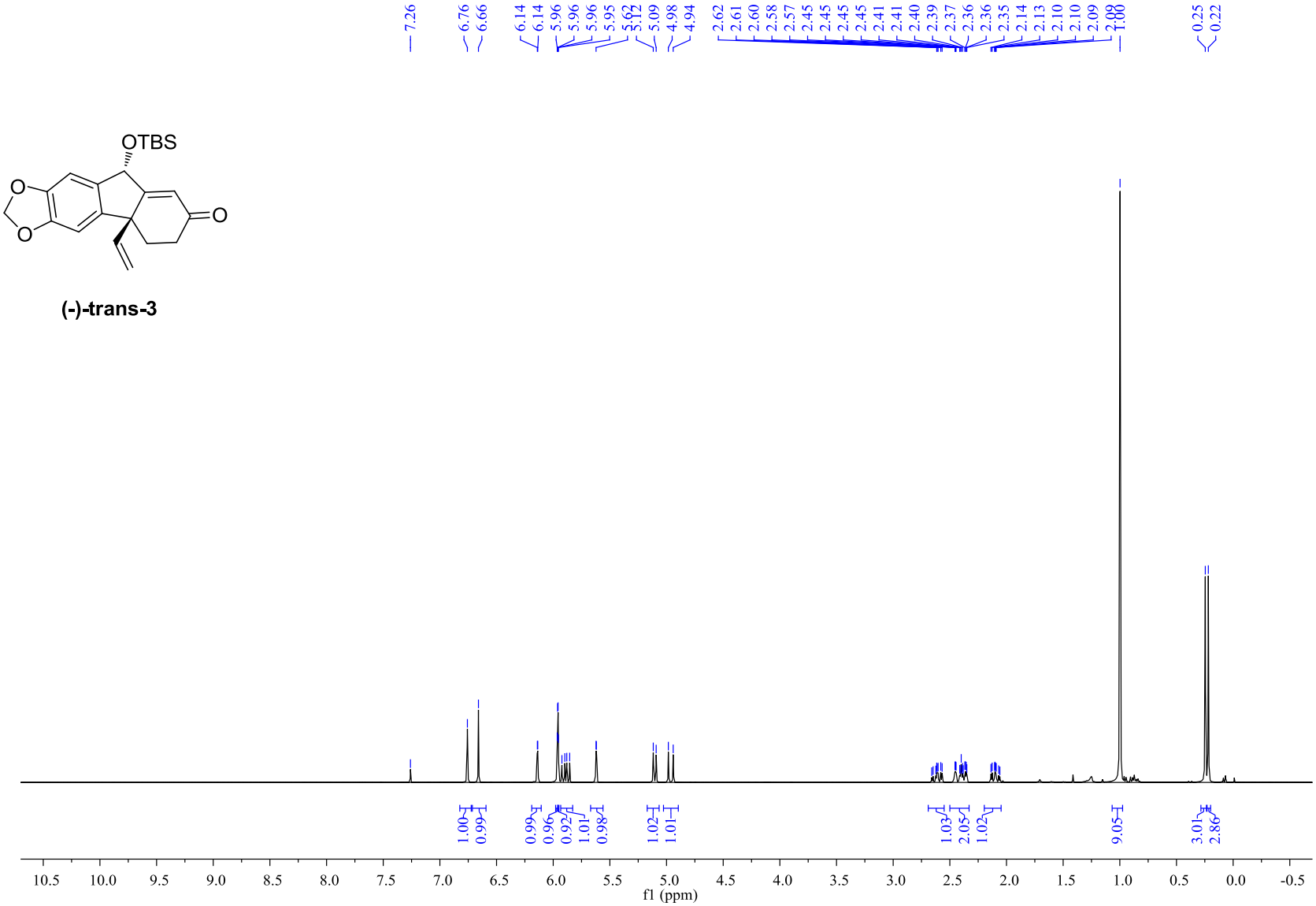

OTBS
O
O

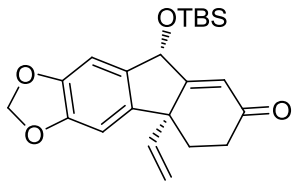
(+)-4


7.26
7.16
6.69
5.96
5.93
5.74
5.73
5.51
5.49
5.47
5.44
4.91
4.89
4.57
4.53
2.45
2.44
1.27
1.26
1.10
1.09
1.07
1.03
1.00
0.99
0.98
0.97
0.96
0.90
0.21
0.09
0.83
0.86
0.94
0.97
0.95
1.05
1.03
0.92
0.86
1.34
1.28
2.14
9.01
2.90
3.12
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)

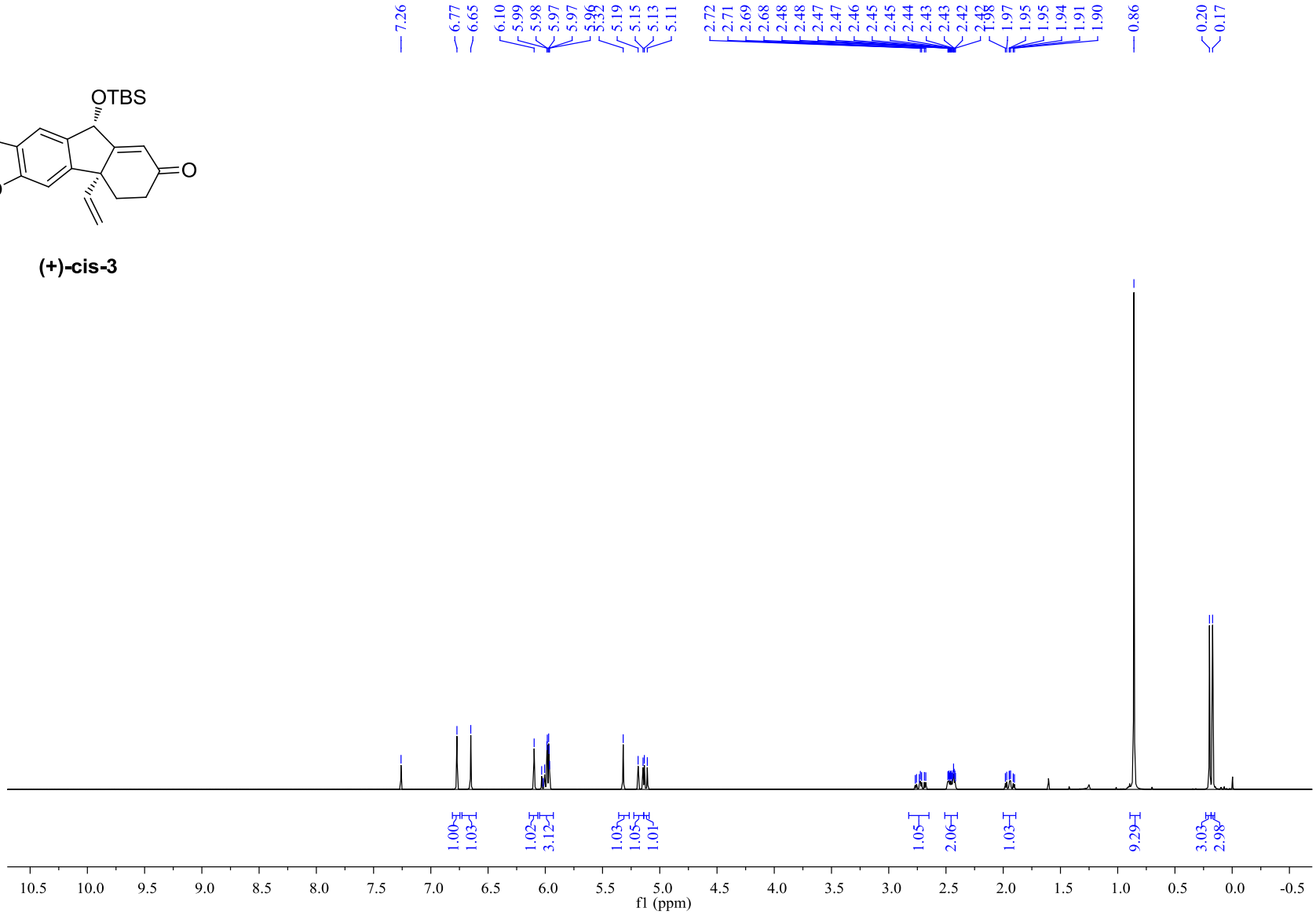
OTBS
O
O
O

(-)-trans-3


7.26
6.76
6.66
6.14
6.14
5.96
5.96
5.96
5.95
5.62
5.12
5.09
4.98
4.94
2.62
2.61
2.60
2.58
2.57
2.45
2.45
2.45
2.41
2.41
2.40
2.39
2.37
2.36
2.36
2.35
2.14
2.13
2.10
2.10
2.09
2.08
1.00
0.25
0.22
1.00
0.99
0.99
0.96
0.92
1.01
0.98
1.02
1.01
1.03
2.05
1.02
9.05
3.01
2.86
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)

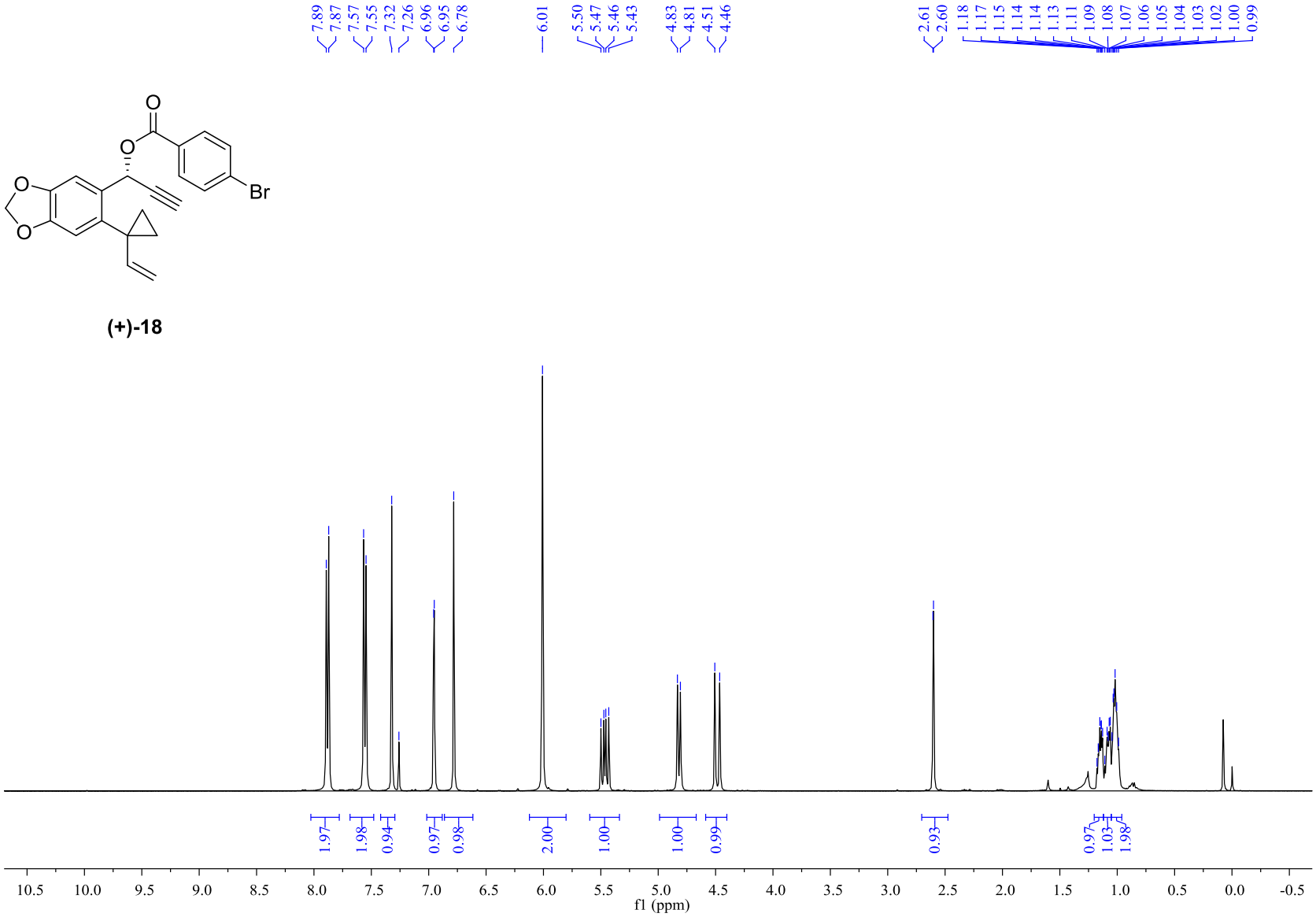

OTBS
O
O
O

(+)-cis-3


7.26
6.77
6.65
6.10
5.99
5.98
5.97
5.97
5.19
5.15
5.13
5.11
2.72
2.71
2.69
2.68
2.48
2.48
2.47
2.47
2.46
2.45
2.45
2.44
2.43
2.43
2.42
1.97
1.95
1.95
1.94
1.91
1.90
0.86
0.20
0.17
1.00
1.03
1.02
3.12
1.03
1.05
1.01
1.05
2.06
1.03
9.29
3.03
2.98
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)

O
O
O
O
Br

(+)-18


7.89
7.87
7.57
7.55
7.32
7.26
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6.95
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6.01
5.50
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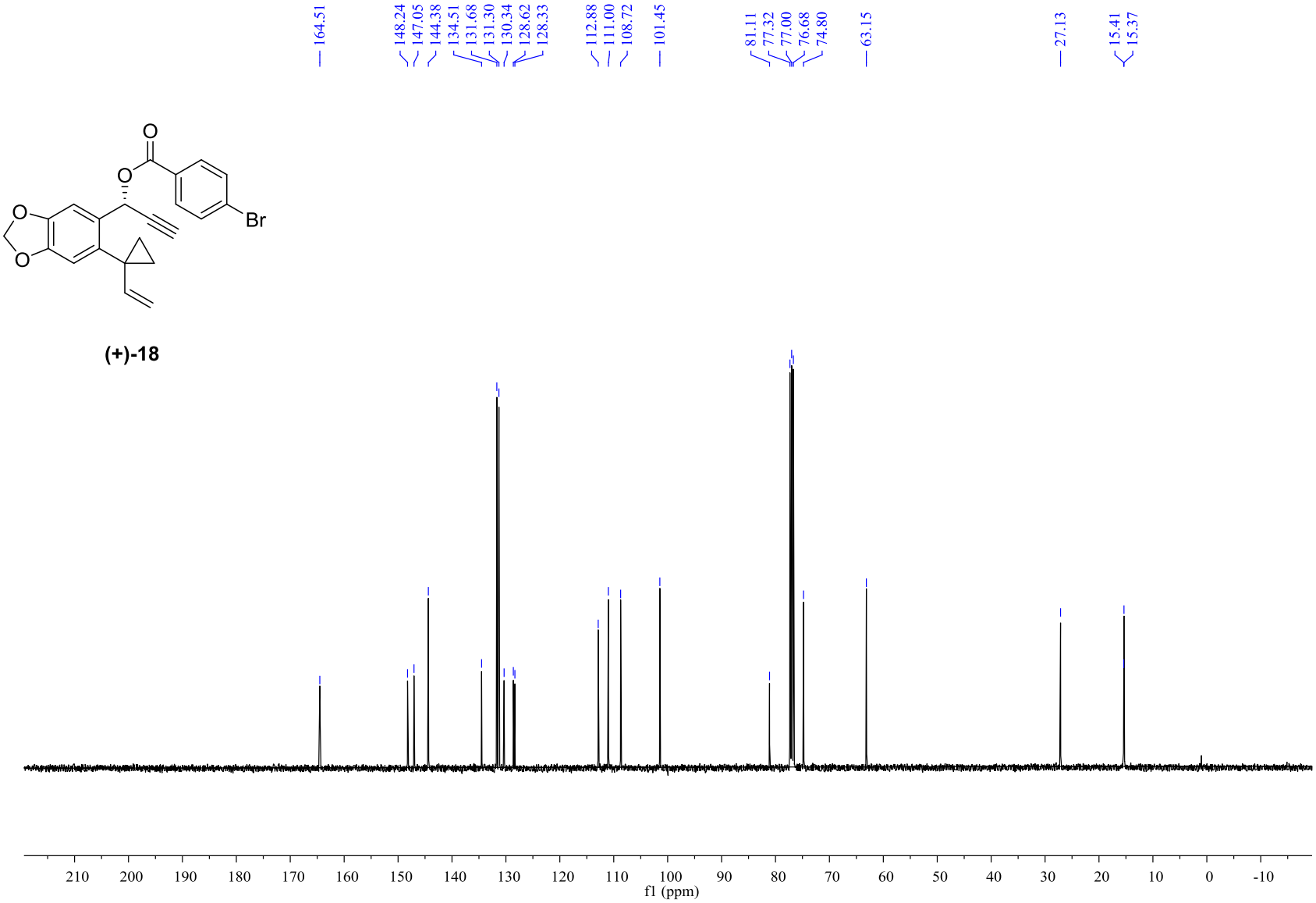
5.46
5.43
4.83
4.81
4.51
4.46
2.61
2.60
1.18
1.17
1.15
1.14
1.14
1.13
1.11
1.09
1.08
1.07
1.06
1.05
1.04
1.03
1.02
1.00
0.99
1.97
1.98
0.94
0.97
0.98
2.00
1.00
1.00
0.99
0.93
0.97
1.03
1.98
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

O
O
O
O
Br

(+)-18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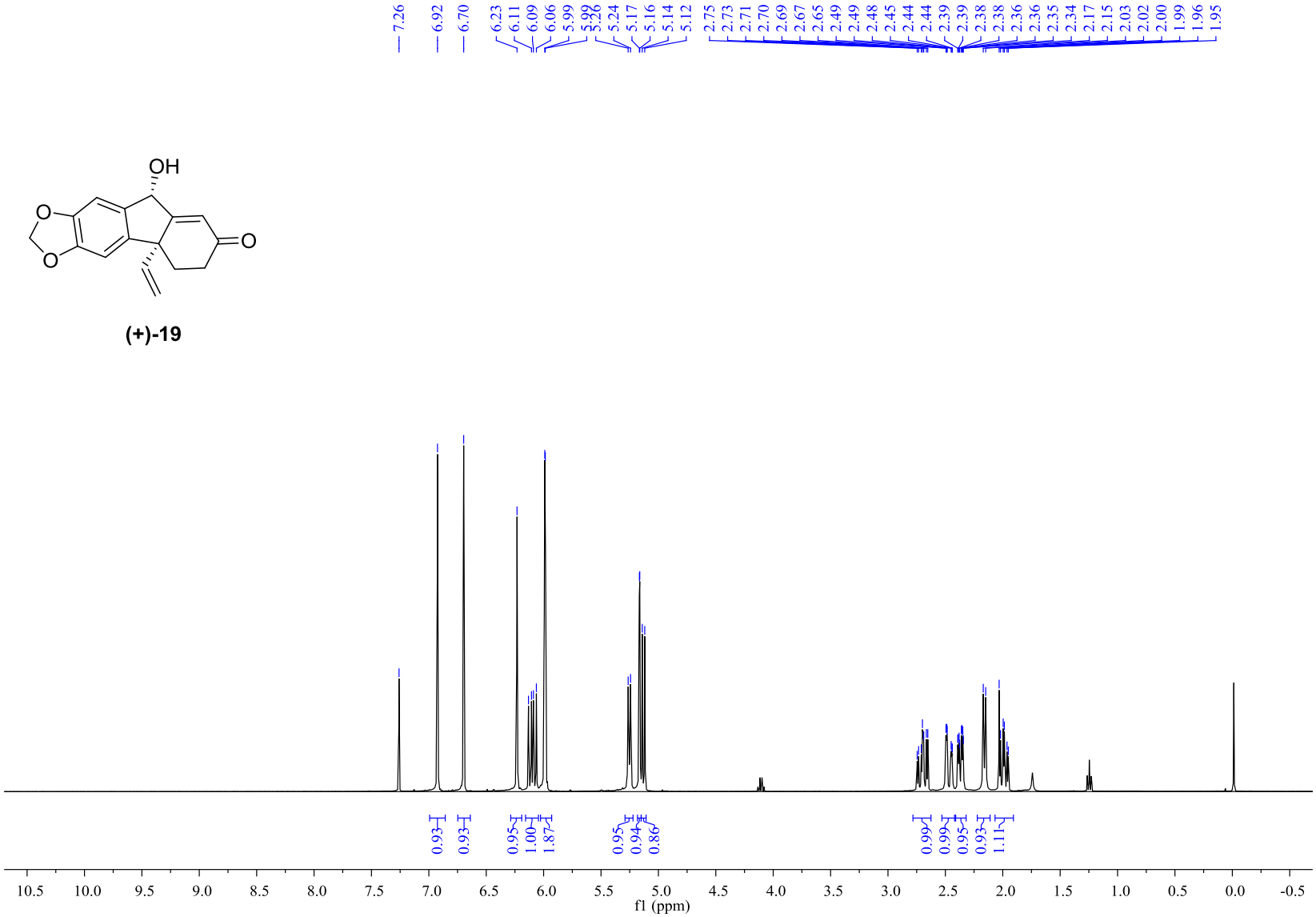
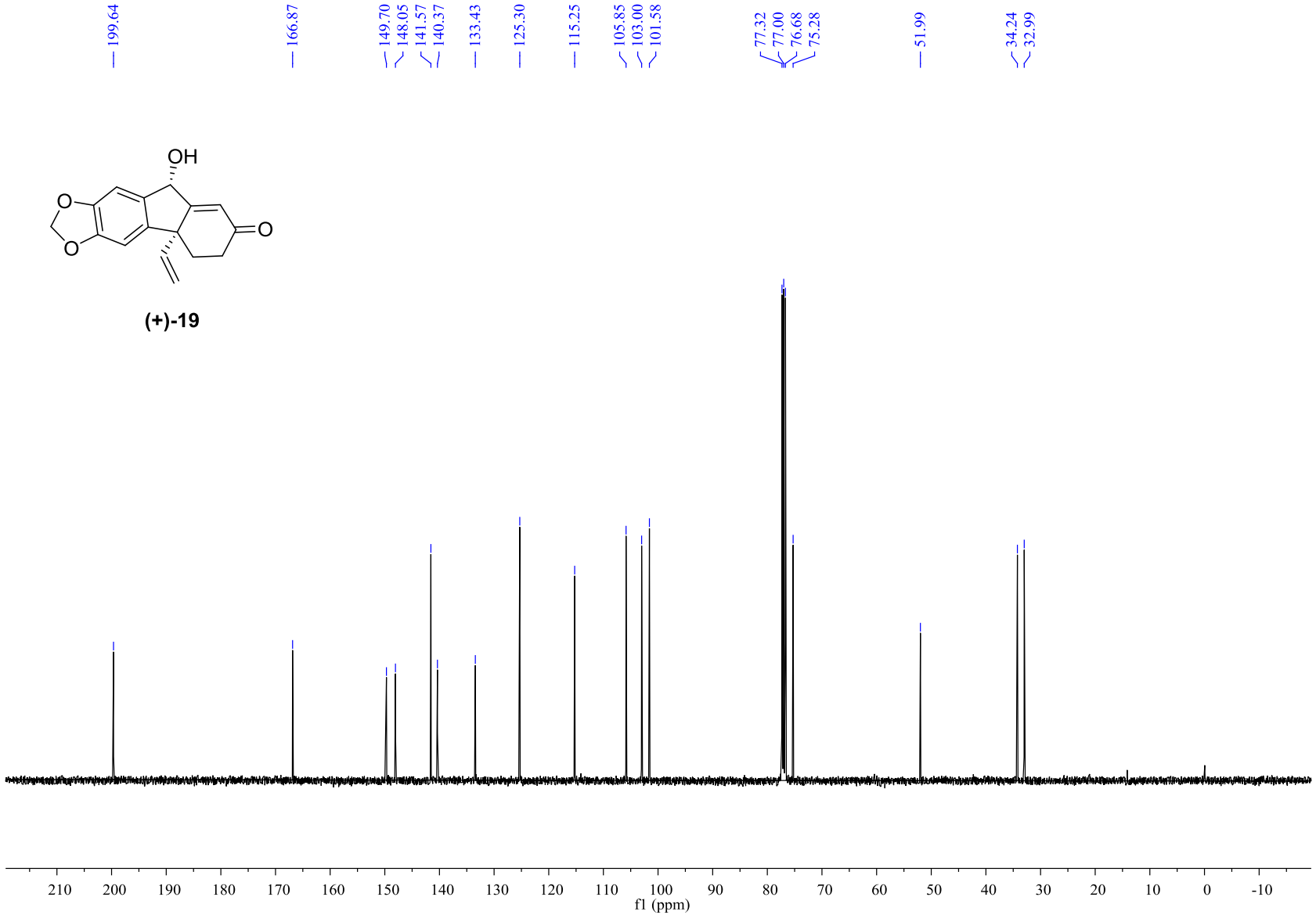
147.05
144.38
134.51
131.68
131.30
130.34
128.62
128.33
112.88
111.00
108.72
101.45
81.11
77.32
77.00
76.68
74.80
63.15
27.13
15.41
15.37
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.26
6.92
6.70
6.23
6.11
6.09
6.06
5.99
5.99
5.26
5.24
5.17
5.16
5.14
5.12
2.75
2.73
2.71
2.70
2.69
2.67
2.65
2.49
2.49
2.48
2.45
2.44
2.44
2.44
2.39
2.39
2.38
2.38
2.36
2.36
2.35
2.34
2.17
2.15
2.03
2.02
2.00
1.99
1.96
1.95
OH
O
O
O
(+)-19
0.93
0.93
0.95
1.00
1.87
0.95
0.94
0.86
0.99
0.99
0.95
0.93
1.11
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)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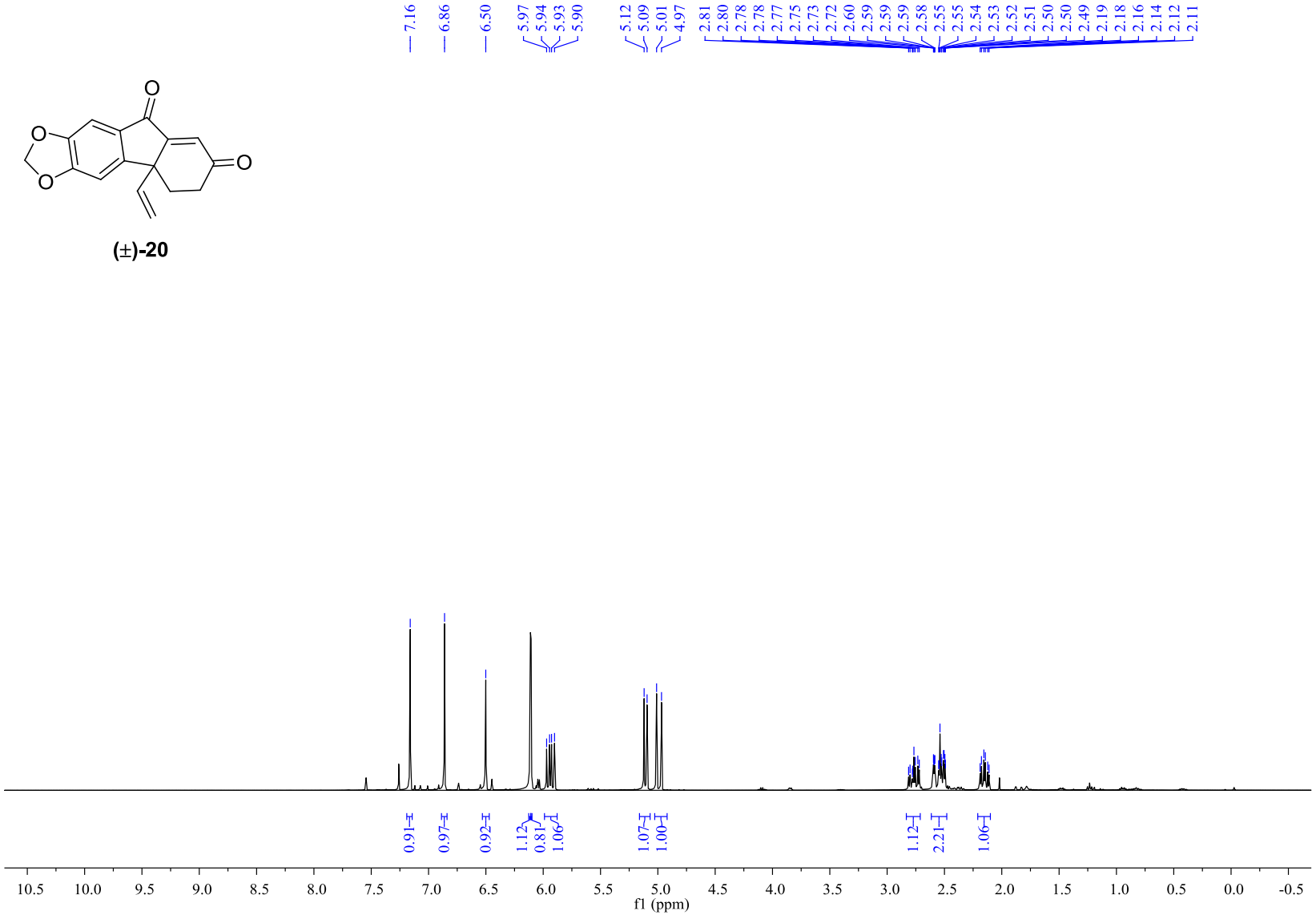
140.37
133.43
125.30
115.25
105.85
103.00
101.58
77.32
77.00
76.68
75.28
51.99
34.24
32.99
OH
O
O
O
(+)-19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.16
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5.94
5.93
5.90
5.12
5.09
5.01
4.97
2.81
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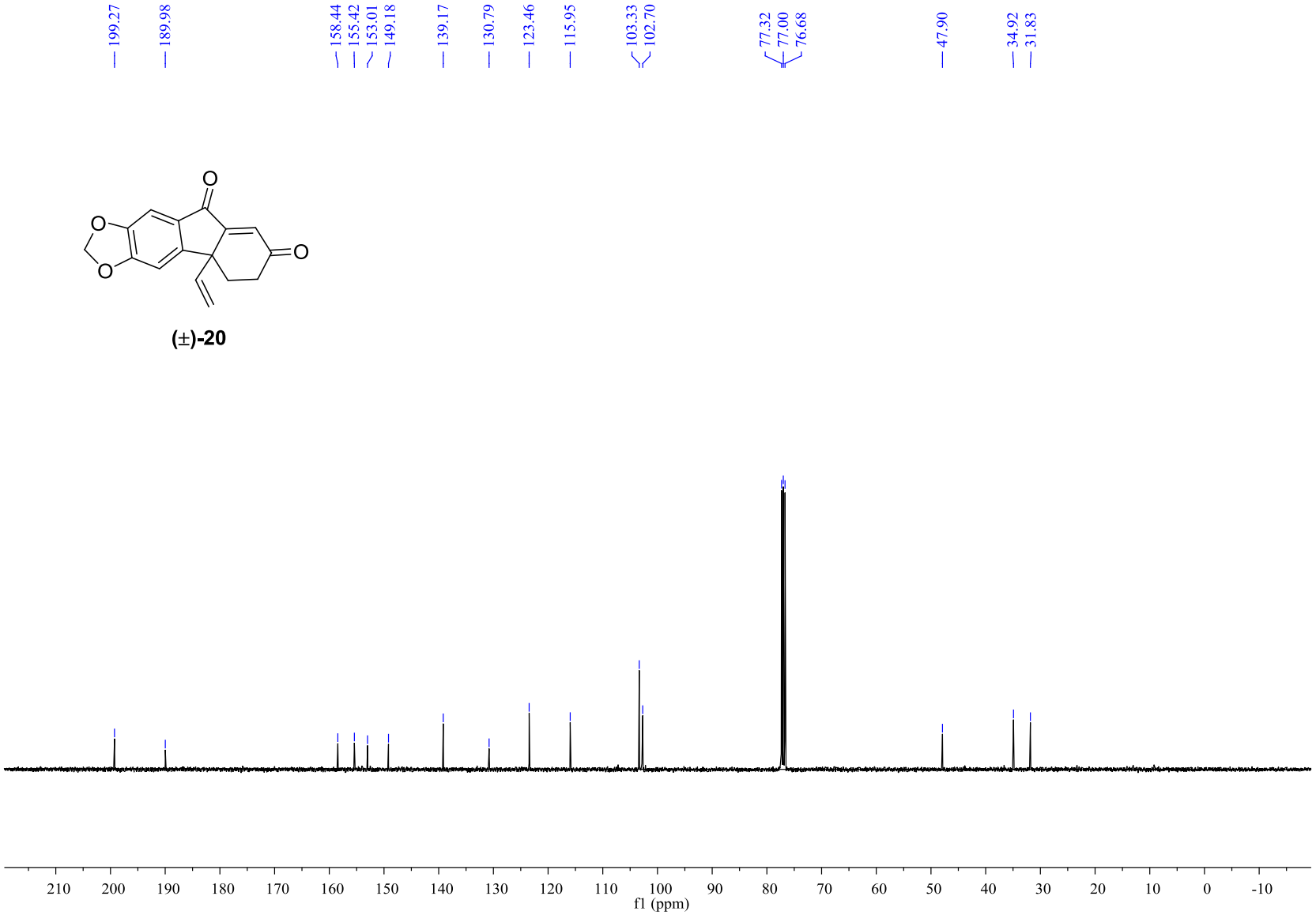
2.78
2.78
2.77
2.75
2.73
2.72
2.60
2.59
2.59
2.59
2.58
2.55
2.55
2.54
2.53
2.52
2.51
2.50
2.50
2.49
2.19
2.18
2.16
2.14
2.12
2.11
O
O
O
O
0.91
0.97
0.92
1.12
0.81
1.06
1.07
1.00
1.12
2.21
1.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

(±)-20


199.27
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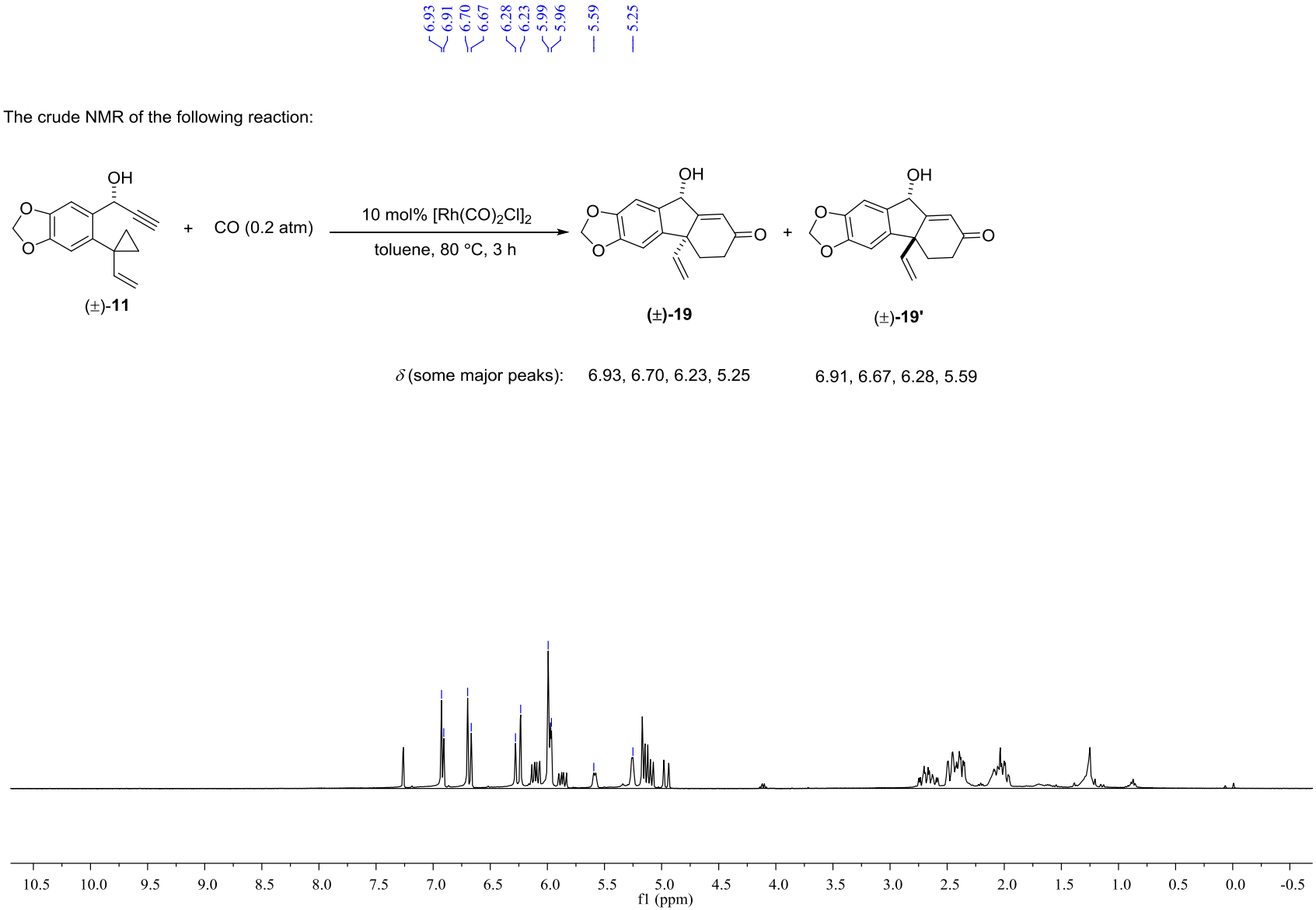
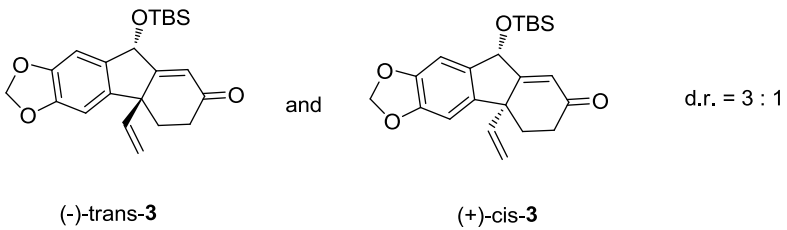
158.44
155.42
153.01
149.18
139.17
130.79
123.46
115.95
103.33
102.70
77.32
77.00
76.68
47.90
34.92
31.83
O
O
O
O
(±)-20
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

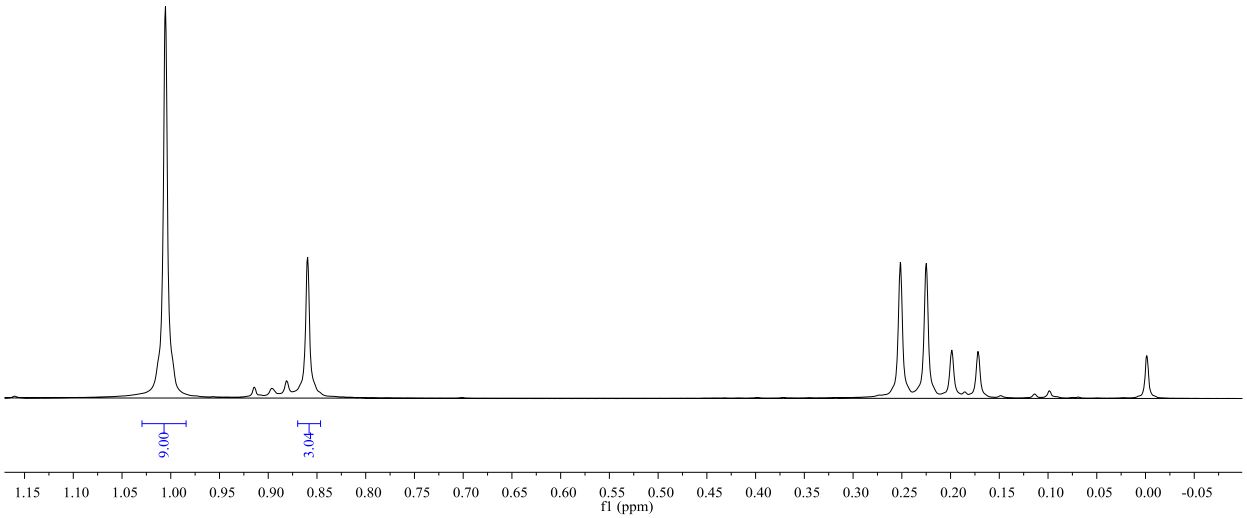
The crude NMR of the following reaction:

δ (some major peaks): 6.93, 6.70, 6.23, 5.25 6.91, 6.67, 6.28, 5.59

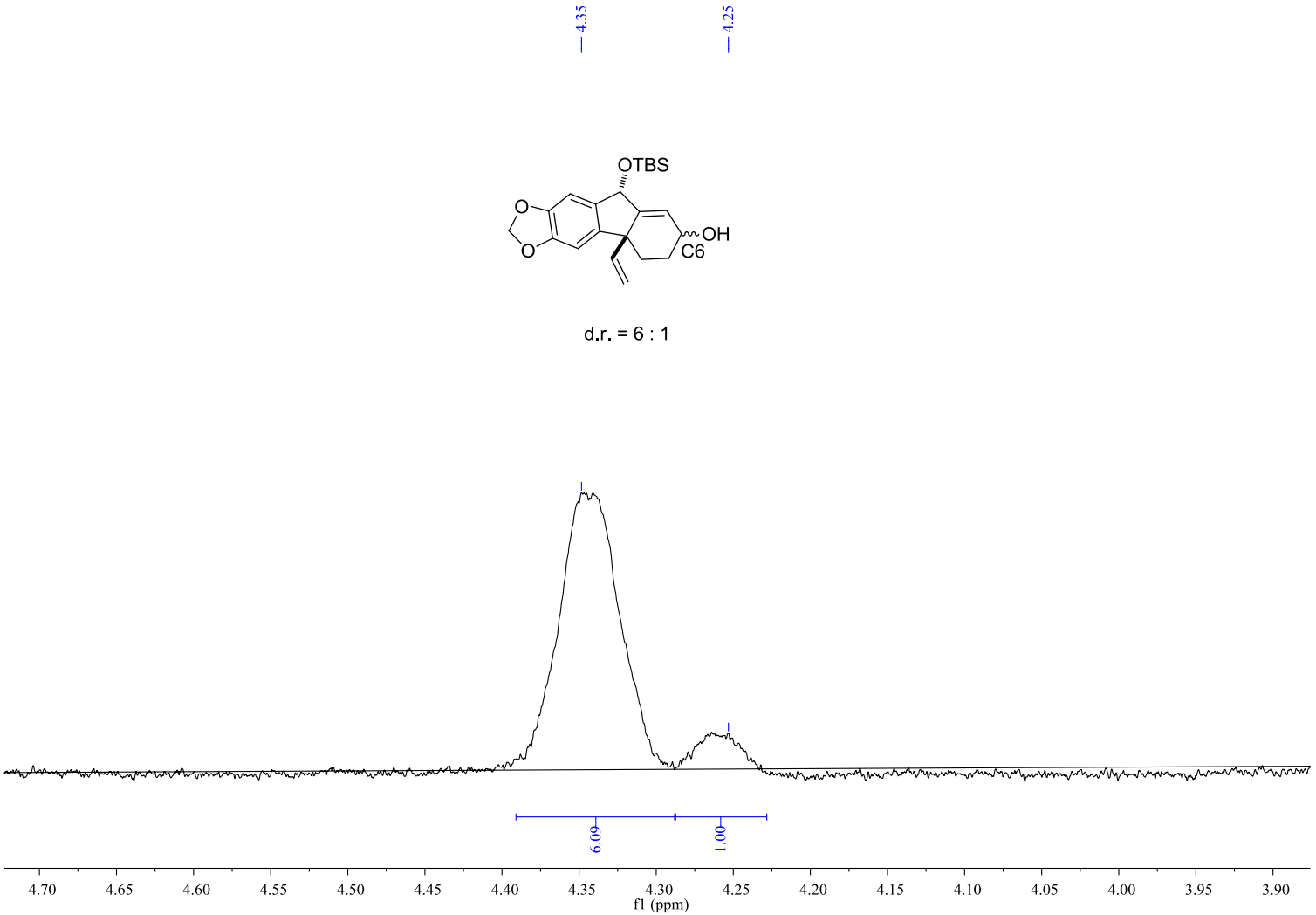


Crude ^{1}H NMR

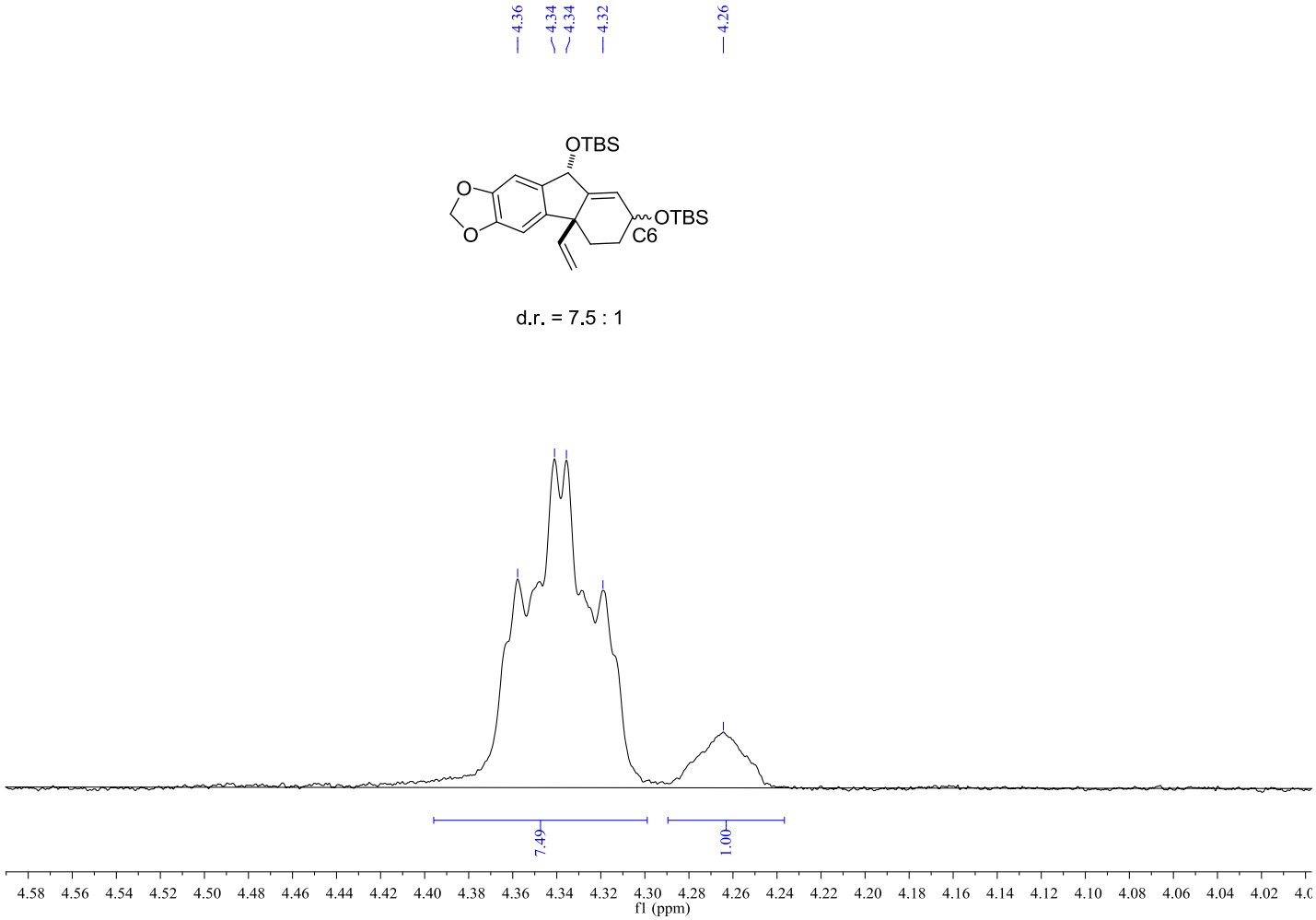

OTBS
O
O
O
and
OTBS
O
O
O
d.r. = 3 : 1
(-)-trans-3
(+)-cis-3


9.00
3.04
1.15 1.10 1.05 1.00 0.95 0.90 0.85 0.80 0.75 0.70 0.65 0.60 0.55 0.50 0.45 0.40 0.35 0.30 0.25 0.20 0.15 0.10 0.05 0.00 -0.05
f1 (ppm)

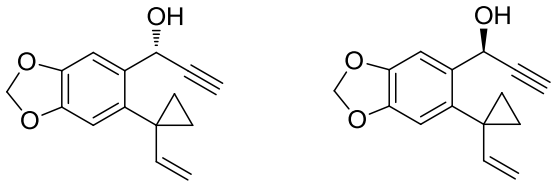
Crude ^{1}H NMR


4.35
4.25
OTBS
O
O
OH
C6
d.r. = 6 : 1
6.09
1.00
4.70
4.65
4.60
4.55
4.50
4.45
4.40
4.35
4.30
4.25
4.20
4.15
4.10
4.05
4.00
3.95
3.90
f1 (ppm)

Crude ^{1}H NMR of (±)-**12**

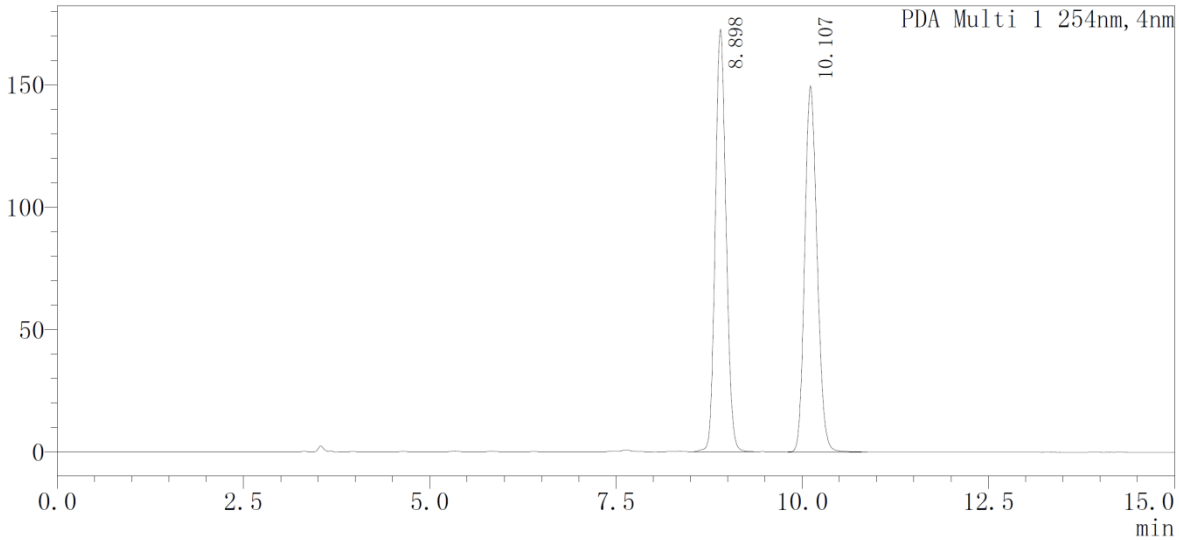


HPLC spectra

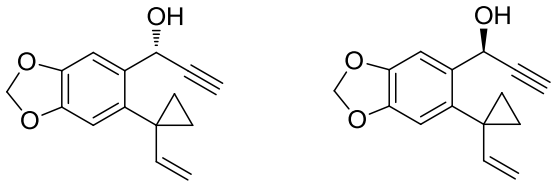


(-)-11 (+)-11

50 : 50

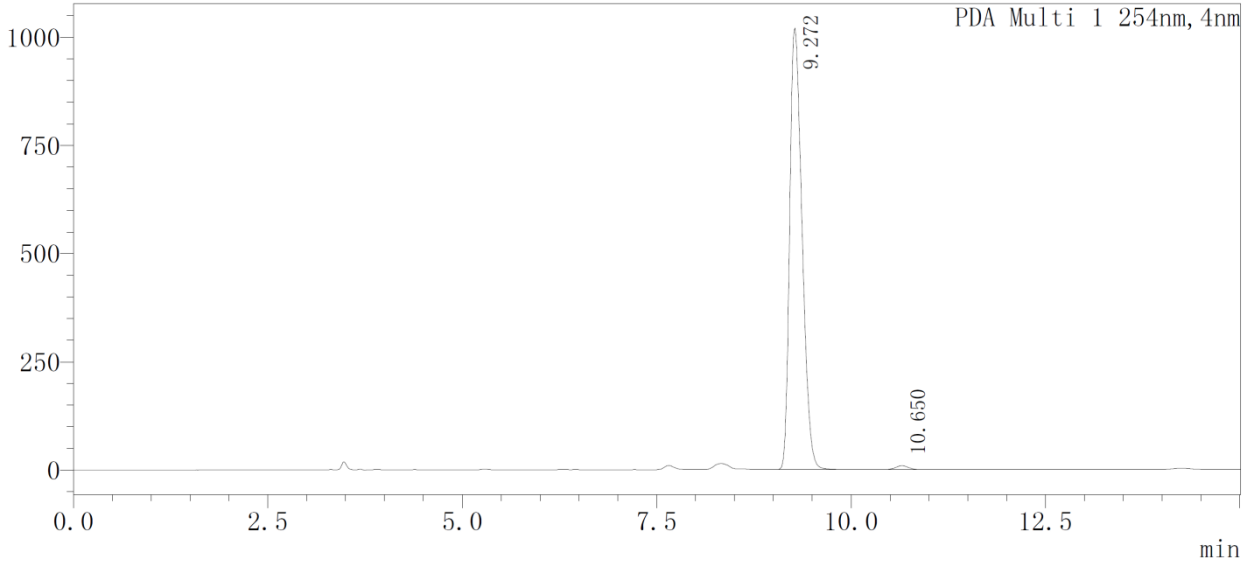


Peak	RetTime	Area	Height	Concentration
1	8.898	1739239	172664	50.101
2	10.107	1732245	149604	49.899
total		3471484	322268	



(-)-**11** (+)-**11**

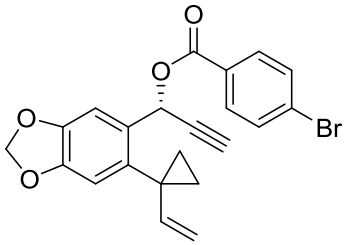
99 : **1**



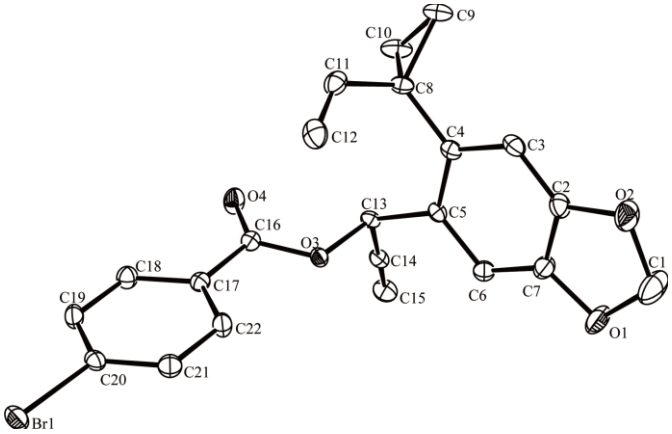
Peak	RetTime	Area	Height	Concentration
1	9.272	11296413	1019920	99.081
2	10.650	104816	8939	0.919
total		11401229	1028859	

X-ray data

X-ray data of (+)-18 (exp_6570).:



(+)-18



Ellipsoids are drawn at 30% probability

Table 1 Crystal data and structure refinement for exp_6570.

Identification code	exp_6570
Empirical formula	C22H17BrO4
Formula weight	425.26
Temperature/K	180.00(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	12.49463(6)

b/Å	6.03595(5)
c/Å	12.70711(6)
α/°	90
β/°	99.5432(5)
γ/°	90
Volume/$Å^3$	945.069(11)
Z	2
ρ_{calc}g/cm^3	1.494
μ/mm^{-1}	3.175
F(000)	432.0
Crystal size/mm^3	0.1 × 0.1 × 0.05
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.054 to 133.108
Index ranges	$-14 \leq h \leq 14$, $-7 \leq k \leq 5$, $-15 \leq l \leq 15$
Reflections collected	69848
Independent reflections	3042 [R_{int} = 0.0473, R_{sigma} = 0.0125]

Data/restraints/parameters	3042/1/245
Goodness-of-fit on F^2	1.076
Final R indexes [I>=2σ (I)]	R_1 = 0.0260, wR_2 = 0.0684
Final R indexes [all data]	R_1 = 0.0261, wR_2 = 0.0684
Largest diff. peak/hole / e $Å^{-3}$	0.58/-0.37
Flack parameter	-0.024(7)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_6570. Ueq is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
Br1	14662.0(2)	9456.8(8)	8501.0(2)	32.79(16)
C1	5872(5)	8720(11)	4133(4)	69(2)
C2	6442(3)	7271(7)	5741(3)	30.0(8)
C3	6592(3)	6684(6)	6791(3)	27.6(8)
C4	7383(2)	5069(5)	7149(2)	21.9(8)

C5	7993(2)	4160(6)	6438(2)	20.7(7)
C6	7810(2)	4754(8)	5351(2)	28.8(8)
C7	7035(3)	6324(7)	5031(3)	31.4(8)
C8	7548(2)	4428(8)	8312(2)	25.3(6)
C9	6534(3)	3919(7)	8781(3)	38.7(11)
C10	7287(3)	2078(8)	8594(3)	39.4(10)
C11	8430(3)	5576(7)	9044(3)	31.7(8)
C12	9068(3)	7157(7)	8785(3)	38.2(9)
C13	8927(2)	2599(6)	6827(2)	21.1(7)
C14	9093(2)	880(6)	6063(2)	24.4(7)
C15	9256(3)	-458(9)	5430(3)	34.5(7)
C16	10704(3)	3281(6)	7801(2)	22.5(7)
C17	11641(2)	4839(6)	7945(2)	21.7(7)
C18	12585(3)	4168(7)	8624(2)	28.0(8)
C19	13488(3)	5534(7)	8779(3)	30.5(8)
C20	13428(2)	7552(6)	8265(2)	25.1(7)

C21	12505(3)	8266(7)	7599(3)	29.5(8)
C22	11609(3)	6874(6)	7441(3)	28.3(8)
O1	6736(3)	7254(6)	4036(2)	50.6(9)
O2	5734(2)	8819(6)	5220(2)	48.3(9)
O3	9904.8(16)	3994(4)	7016.9(16)	22.0(6)
O4	10651(2)	1607(5)	8295(2)	35.8(6)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_6570. The Anisotropic displacement factor exponent takes the form: -

$2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	22.26(18)	41.5(3)	35.8(2)	-9.08(18)	8.33(11)	-7.66(17)
C1	87(4)	84(5)	38(2)	16(2)	12(2)	56(3)
C2	25.4(16)	31(2)	33.2(17)	-0.6(15)	3.1(13)	7.4(14)
C3	21.1(15)	31(2)	32.0(16)	-5.8(14)	8.3(12)	0.5(14)
C4	20.9(13)	20(2)	25.9(14)	-4.3(12)	6.2(11)	-5.3(11)

C5	17.7(12)	20(2)	25.0(13)	-2.3(13)	3.7(10)	-1.3(13)
C6	27.2(14)	36(2)	24.1(13)	-2.6(16)	6.5(11)	3.2(16)
C7	33.4(18)	37(2)	23.7(16)	1.7(15)	3.1(13)	7.2(16)
C8	27.9(14)	25.6(18)	24.9(13)	-4.2(18)	12.1(10)	0.1(19)
C9	39.1(19)	46(3)	36.7(17)	2.1(17)	23.3(15)	-4.4(17)
C10	52(2)	32(3)	41(2)	3.7(17)	27.6(18)	-2.7(19)
C11	36.2(18)	34(2)	24.6(15)	-3.2(14)	2.7(13)	6.7(17)
C12	35.5(19)	37(3)	39.6(19)	-11.4(18)	-0.3(15)	-3.8(17)
C13	20.3(14)	19.2(19)	24.9(14)	1.6(12)	7.2(11)	-2.6(12)
C14	20.8(14)	22(2)	31.0(16)	2.7(15)	7.0(12)	0.6(13)
C15	35.9(16)	30(2)	39.5(16)	-6(2)	11.7(12)	2(2)
C16	24.0(15)	21(2)	22.4(14)	-1.2(14)	3.7(11)	2.4(13)
C17	23.8(13)	21(2)	20.2(12)	-2.4(13)	4.2(10)	1.0(13)
C18	27.9(15)	26(2)	28.8(14)	1.8(16)	2.4(11)	0.6(16)
C19	20.8(15)	41(2)	27.6(15)	-2.9(15)	-1.0(12)	3.9(15)
C20	20.8(14)	32(2)	23.1(14)	-8.0(14)	6.5(11)	-2.7(14)

C21	30.4(17)	27(2)	31.0(16)	4.0(15)	4.7(13)	-2.6(16)
C22	22.4(16)	31(2)	29.5(15)	0.8(14)	-1.0(12)	-0.2(14)
O1	58.9(18)	69(2)	23.8(12)	9.1(14)	4.8(12)	33.5(16)
O2	47.0(15)	56(3)	40.5(14)	3.5(13)	4.3(12)	29.1(14)
O3	17.6(9)	22.9(17)	25.3(9)	3.3(9)	2.6(7)	-1.1(9)
O4	33.7(13)	29.6(16)	40.6(13)	13.7(12)	-3.8(10)	-3.9(11)

Table 4 Bond Lengths for exp_6570.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C20	1.907(3)	C8	C11	1.489(5)
C1	O1	1.417(6)	C9	C10	1.499(6)
C1	O2	1.422(5)	C11	C12	1.320(6)
C2	C3	1.363(5)	C13	C14	1.459(5)
C2	C7	1.382(5)	C13	O3	1.471(4)
C2	O2	1.378(5)	C14	C15	1.181(6)
C3	C4	1.409(5)	C16	C17	1.489(5)

C4	C5	1.388(4)	C16	O3	1.358(4)
C4	C8	1.508(4)	C16	O4	1.197(4)
C5	C6	1.408(4)	C17	C18	1.402(4)
C5	C13	1.517(4)	C17	C22	1.383(5)
C6	C7	1.368(5)	C18	C19	1.385(5)
C7	O1	1.377(4)	C19	C20	1.378(6)
C8	C9	1.518(4)	C20	C21	1.382(5)
C8	C10	1.512(7)	C21	C22	1.387(5)

Table 5 Bond Angles for exp_6570.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	C1	O2	109.1(3)	C9	C10	C8	60.5(2)
C3	C2	C7	121.9(3)	C12	C11	C8	126.6(3)
C3	C2	O2	128.3(3)	C14	C13	C5	114.3(3)
O2	C2	C7	109.8(3)	C14	C13	O3	108.1(2)
C2	C3	C4	118.1(3)	O3	C13	C5	105.7(3)

C3	C4	C8	117.9(3)	C15	C14	C13	177.4(4)
C5	C4	C3	119.6(3)	O3	C16	C17	110.9(3)
C5	C4	C8	122.4(3)	O4	C16	C17	125.4(3)
C4	C5	C6	121.4(3)	O4	C16	O3	123.7(3)
C4	C5	C13	120.6(3)	C18	C17	C16	117.3(3)
C6	C5	C13	117.9(3)	C22	C17	C16	122.9(3)
C7	C6	C5	117.3(3)	C22	C17	C18	119.9(3)
C6	C7	C2	121.5(3)	C19	C18	C17	119.9(4)
C6	C7	O1	128.7(3)	C20	C19	C18	118.7(3)
O1	C7	C2	109.7(3)	C19	C20	Br1	118.6(2)
C4	C8	C9	116.7(3)	C19	C20	C21	122.7(3)
C4	C8	C10	118.2(3)	C21	C20	Br1	118.6(3)
C10	C8	C9	59.3(3)	C20	C21	C22	118.1(3)
C11	C8	C4	117.3(3)	C17	C22	C21	120.8(3)
C11	C8	C9	115.4(3)	C7	O1	C1	105.6(3)
C11	C8	C10	117.1(3)	C2	O2	C1	105.4(3)

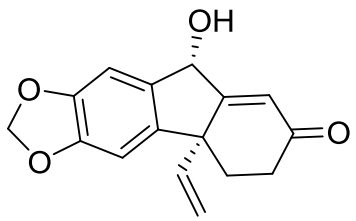
C10	C9	C8	60.1(3)	C16	O3	C13	115.4(2)

Table 6 Hydrogen Atom Coordinates (Å×104) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_6570.

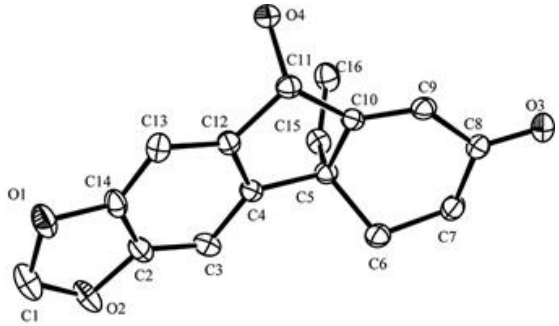
Atom	x	y	z	U(eq)
H1A	5195	8191	3687	83
H1B	6037	10215	3882	83
H3	6175	7344	7267	33
H6	8209	4090	4859	35
H9A	6523	4372	9528	46
H9B	5826	4014	8301	46
H10A	7040	1041	8000	47
H10B	7737	1399	9226	47
H11	8544	5124	9770	38
H12A	8986	7668	8069	46
H12B	9609	7784	9315	46

H13	8813	1896	7512	25
H15	9387	-1534	4920	41
H18	12607	2777	8977	34
H19	14136	5090	9231	37
H21	12484	9671	7258	35
H22	10967	7323	6982	34

X-ray data of (+)-19 (exp_6323):



(+)-19



Ellipsoids are drawn at 30% probability

Table 1 Crystal data and structure refinement for exp_6323.

Identification code	exp_6323
Empirical formula	C16H14O4
Formula weight	270.27

Temperature/K	180.01(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.9516(7)
b/Å	10.8253(8)
c/Å	27.1895(19)
α / °	90
β / °	90
γ / °	90
Volume/Å^3	2634.8(3)
Z	8
ρ_{calc}g/cm^3	1.363
μ /mm^{-1}	0.098
F(000)	1136.0
Crystal size/mm^3	0.1 × 0.1 × 0.05

Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.862 to 52.042
Index ranges	$-10 \leqslant h \leqslant 11$, $-13 \leqslant k \leqslant 11$, $-33 \leqslant l \leqslant 27$
Reflections collected	15976
Independent reflections	5175 [R_{int} = 0.0616, R_{sigma} = 0.0752]
Data/restraints/parameters	5175/0/363
Goodness-of-fit on F2	0.991
Final R indexes [I>=2σ (I)]	R1 = 0.0465, wR2 = 0.0790
Final R indexes [all data]	R1 = 0.0853, wR2 = 0.0918
Largest diff. peak/hole / e Å-3	0.15/-0.17
Flack parameter	0.3(8)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_6323. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
C1	4033(6)	5421(4)	8357.6(16)	66.8(13)
C2	2683(5)	6584(3)	8865.1(12)	38.3(9)
C3	1616(4)	7028(3)	9177.9(11)	37.0(9)
C4	1829(4)	8246(3)	9332.5(11)	29.0(8)
C5	840(4)	8971(3)	9686.5(11)	29.6(8)
C6	-818(4)	8997(3)	9552.7(12)	37.5(9)
C7	-1619(4)	9946(3)	9874.7(13)	40.4(9)
C8	-866(4)	11177(3)	9888.6(12)	37.2(9)
C9	669(4)	11265(3)	9706.9(11)	33.5(8)
C10	1466(4)	10254(3)	9624.9(11)	28.8(8)
C11	3021(4)	10217(3)	9403.8(11)	32.2(8)
C12	3053(4)	8933(3)	9183.2(10)	30.2(8)
C13	4133(4)	8455(3)	8864.5(11)	35.6(9)
C14	3897(4)	7264(3)	8714.3(11)	37.6(9)
C15	1035(4)	8440(3)	10199.3(11)	35.1(9)

C16	1755(5)	8940(3)	10565.7(12)	43.0(9)
C17	-3869(5)	2405(4)	9061.9(14)	52.3(11)
C18	-2280(4)	1473(3)	8548.4(11)	35.3(9)
C19	-1581(4)	656(3)	8239.4(11)	34.3(9)
C20	-114(4)	964(3)	8117.6(10)	29.9(8)
C21	913(4)	265(3)	7770.8(11)	31.6(8)
C22	1036(4)	-1119(3)	7883.0(12)	37.6(9)
C23	2314(5)	-1668(3)	7593.5(12)	48.1(10)
C24	3752(5)	-981(4)	7641.6(13)	52.6(11)
C25	3700(4)	300(4)	7825.1(12)	45.3(10)
C26	2391(4)	878(3)	7880.1(11)	34.8(9)
C27	2154(4)	2141(3)	8104.1(12)	37.8(9)
C28	575(4)	2027(3)	8294.3(11)	33.0(8)
C29	-167(4)	2843(3)	8607.9(11)	37.7(9)
C30	-1599(4)	2531(3)	8720.5(12)	38.1(9)
C31	397(4)	444(3)	7244.8(11)	37.3(9)

C32	1141(5)	979(3)	6886.4(13)	48.8(10)
O1	4779(3)	6570(2)	8404.7(8)	52.6(7)
O2	2749(3)	5410(2)	8666.2(9)	55.9(8)
O3	-1513(3)	12093(2)	10051.4(10)	54.0(7)
O4	4184(3)	10335(2)	9761.2(8)	42.5(6)
O5	-2621(3)	3199(2)	8999.2(9)	56.9(8)
O6	-3738(3)	1410(2)	8717.3(9)	52.5(7)
O7	4949(4)	-1452(3)	7532.5(12)	84.6(11)
O8	2209(3)	3124(2)	7753.9(9)	46.7(7)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_6323. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U11	U22	U33	U23	U13	U12
C1	88(4)	44(3)	68(3)	-18(2)	11(3)	13(3)
C2	53(3)	29.0(18)	32.7(18)	-4.4(16)	-6.7(19)	7.2(19)

C3	46(3)	34(2)	30.6(18)	-2.4(16)	-3.5(17)	-4.7(18)
C4	34(2)	25.4(17)	27.8(16)	0.1(14)	-4.4(16)	0.4(16)
C5	29(2)	28.3(18)	31.4(17)	-0.6(15)	1.4(16)	-3.3(16)
C6	34(2)	29.9(19)	49(2)	1.4(17)	-2.9(18)	-4.6(17)
C7	29(2)	37(2)	55(2)	0.1(17)	-3.2(19)	1.5(17)
C8	33(2)	35(2)	44(2)	-2.2(17)	-9.1(18)	5.3(18)
C9	31(2)	27.1(19)	41.9(19)	1.4(16)	-5.3(17)	-4.3(16)
C10	32(2)	26.6(19)	27.7(17)	-1.5(15)	-3.4(15)	-1.1(16)
C11	32(2)	30.6(18)	34.0(17)	1.2(15)	-1.9(17)	-3.5(16)
C12	35(2)	31.0(18)	25.0(16)	0.9(15)	-3.8(16)	4.7(17)
C13	37(2)	40(2)	29.2(17)	6.3(16)	3.8(16)	6.7(18)
C14	48(3)	41(2)	24.3(17)	-4.9(16)	-1.0(18)	14.6(19)
C15	41(2)	30.7(18)	34.0(18)	1.9(16)	7.7(17)	-2.7(17)
C16	52(3)	43(2)	33.6(19)	0.4(18)	2.5(19)	7.5(19)
C17	44(3)	62(3)	51(2)	-2(2)	16(2)	7(2)
C18	30(2)	42(2)	34.0(19)	1.3(17)	7.3(17)	-3.2(17)

C19	37(2)	33.7(19)	32.2(18)	-0.1(16)	-0.2(17)	-5.2(17)
C20	30(2)	33.0(18)	26.1(17)	1.9(15)	0.6(15)	-0.8(17)
C21	30(2)	34.1(19)	31.1(17)	-0.8(15)	-1.0(16)	1.9(16)
C22	39(2)	37(2)	36.9(18)	-1.0(16)	-0.1(17)	3.7(18)
C23	60(3)	48(2)	36(2)	1.5(18)	-2(2)	19(2)
C24	55(3)	65(3)	38(2)	21(2)	16(2)	27(2)
C25	31(2)	64(3)	40(2)	15.8(19)	5.3(17)	7(2)
C26	33(2)	45(2)	26.4(16)	8.1(16)	-1.0(16)	1.5(18)
C27	33(2)	43(2)	37.8(18)	2.7(18)	-2.6(18)	-6.7(17)
C28	30(2)	38(2)	31.5(17)	0.2(16)	-0.2(16)	-4.6(17)
C29	48(3)	35(2)	30.4(18)	-7.8(16)	2.3(18)	-8.5(18)
C30	44(3)	38(2)	32.1(19)	-1.9(17)	9.2(18)	1.5(19)
C31	38(2)	40(2)	34.0(19)	-4.7(17)	-1.9(17)	9.5(17)
C32	54(3)	58(2)	35.2(19)	2.1(19)	-4(2)	5(2)
O1	66(2)	51.8(16)	40.2(13)	-11.7(13)	7.8(14)	15.2(15)
O2	81(2)	35.4(14)	51.5(15)	-17.1(13)	7.3(16)	3.4(15)

O3	39.3(17)	44.4(16)	78.3(18)	-16.5(15)	-2.6(15)	10.5(13)
O4	31.9(15)	44.5(15)	51.0(14)	-14.1(12)	-2.7(12)	-2.9(13)
O5	62(2)	52.2(15)	56.4(15)	-13.8(14)	28.2(15)	-4.0(16)
O6	38.8(17)	63.8(17)	54.9(15)	-11.9(14)	17.5(13)	-8.6(14)
O7	68(2)	84(2)	102(2)	40.8(18)	48.6(19)	43(2)
O8	34.9(16)	50.0(15)	55.2(15)	5.6(13)	8.1(13)	-8.2(14)

Table 4 Bond Lengths for exp_6323.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	O1	1.418(5)	C17	O5	1.420(5)
C1	O2	1.423(5)	C17	O6	1.433(4)
C2	C3	1.366(5)	C18	C19	1.371(4)
C2	C14	1.376(5)	C18	C30	1.380(5)
C2	O2	1.382(4)	C18	O6	1.385(4)
C3	C4	1.397(4)	C19	C20	1.394(5)
C4	C5	1.525(5)	C20	C21	1.520(4)

C4	C12	1.385(5)	C20	C28	1.391(4)
C5	C6	1.528(5)	C21	C22	1.532(4)
C5	C10	1.507(4)	C21	C26	1.509(5)
C5	C15	1.518(4)	C21	C31	1.516(4)
C6	C7	1.528(4)	C22	C23	1.511(5)
C7	C8	1.494(5)	C23	C24	1.492(6)
C8	C9	1.463(5)	C24	C25	1.475(6)
C8	O3	1.230(4)	C24	O7	1.223(5)
C9	C10	1.325(4)	C25	C26	1.337(5)
C10	C11	1.517(5)	C26	C27	1.512(5)
C11	C12	1.514(4)	C27	C28	1.510(5)
C11	O4	1.430(4)	C27	O8	1.429(4)
C12	C13	1.398(4)	C28	C29	1.396(4)
C13	C14	1.368(5)	C29	C30	1.360(5)
C14	O1	1.377(4)	C30	O5	1.390(4)
C15	C16	1.304(5)	C31	C32	1.315(5)

Table 5 Bond Angles for exp_6323.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	C1	O2	109.5(3)	C19	C18	O6	127.0(3)
C3	C2	C14	123.4(3)	C30	C18	O6	110.2(3)
C3	C2	O2	126.6(4)	C18	C19	C20	114.9(3)
C14	C2	O2	110.0(3)	C19	C20	C21	126.7(3)
C2	C3	C4	115.1(3)	C28	C20	C19	122.2(3)
C3	C4	C5	126.6(3)	C28	C20	C21	111.0(3)
C12	C4	C3	121.8(3)	C20	C21	C22	114.0(3)
C12	C4	C5	111.6(3)	C26	C21	C20	100.9(3)
C4	C5	C6	115.0(3)	C26	C21	C22	109.1(3)
C10	C5	C4	100.9(3)	C26	C21	C31	113.4(3)
C10	C5	C6	108.5(3)	C31	C21	C20	109.7(3)
C10	C5	C15	114.1(3)	C31	C21	C22	109.6(3)
C15	C5	C4	108.5(3)	C23	C22	C21	109.6(3)

C15	C5	C6	109.7(3)	C24	C23	C22	114.3(3)
C5	C6	C7	109.4(3)	C25	C24	C23	118.1(4)
C8	C7	C6	113.7(3)	O7	C24	C23	121.8(4)
C9	C8	C7	118.2(3)	O7	C24	C25	120.1(4)
O3	C8	C7	121.0(3)	C26	C25	C24	120.4(4)
O3	C8	C9	120.7(3)	C21	C26	C27	110.7(3)
C10	C9	C8	120.6(3)	C25	C26	C21	122.7(3)
C5	C10	C11	111.2(3)	C25	C26	C27	126.3(3)
C9	C10	C5	122.8(3)	C28	C27	C26	101.3(3)
C9	C10	C11	125.6(3)	O8	C27	C26	113.6(3)
C12	C11	C10	101.4(3)	O8	C27	C28	108.8(3)
O4	C11	C10	113.4(2)	C20	C28	C27	111.4(3)
O4	C11	C12	109.7(3)	C20	C28	C29	121.5(3)
C4	C12	C11	111.2(3)	C29	C28	C27	127.1(3)
C4	C12	C13	122.0(3)	C30	C29	C28	115.4(3)
C13	C12	C11	126.8(3)	C18	C30	O5	109.0(3)

C14	C13	C12	115.3(3)	C29	C30	C18	123.1(3)
C2	C14	O1	110.1(3)	C29	C30	O5	127.9(3)
C13	C14	C2	122.5(3)	C32	C31	C21	126.9(4)
C13	C14	O1	127.4(4)	C14	O1	C1	105.3(3)
C16	C15	C5	126.9(3)	C2	O2	C1	105.0(3)
O5	C17	O6	108.2(3)	C30	O5	C17	105.6(3)
C19	C18	C30	122.8(3)	C18	O6	C17	104.9(3)

Table 6 Hydrogen Atom Coordinates (Å$\times 10^4$) and Isotropic Displacement Parameters (Å$^2\times 10^3$) for exp_6323.

Atom	x	y	z	U(eq)
H1A	4715	4741	8452	80
H1B	3730	5293	8011	80
H3	791	6543	9283	44
H6A	-1261	8170	9604	45
H6B	-937	9219	9202	45

H7A	-2650	10055	9751	49
H7B	-1687	9620	10214	49
H9	1098	12053	9647	40
H11	3129	10858	9141	39
H13	4972	8923	8760	43
H15	585	7660	10260	42
H16A	2226	9720	10525	52
H16B	1808	8525	10873	52
H17A	-4807	2866	9003	63
H17B	-3889	2078	9402	63
H19	-2059	-65	8117	41
H22A	1210	-1242	8239	45
H22B	92	-1538	7793	45
H23A	2472	-25307	704	58
H23B	2031	-1695	7242	58
H25	4602	717	7905	54

H27	2871	2290	8380	45
H29	296	3566	8734	45
H31	-568	137	7166	45
H32A	2111	1302	6946	59
H32B	709	1042	6568	59
H4	4168	11051	9881	64
H8	3079	3182	7640	70