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Does electrophilic activation of nitroalkanes in polyphosphoric acid involves formation of nitrile oxides?

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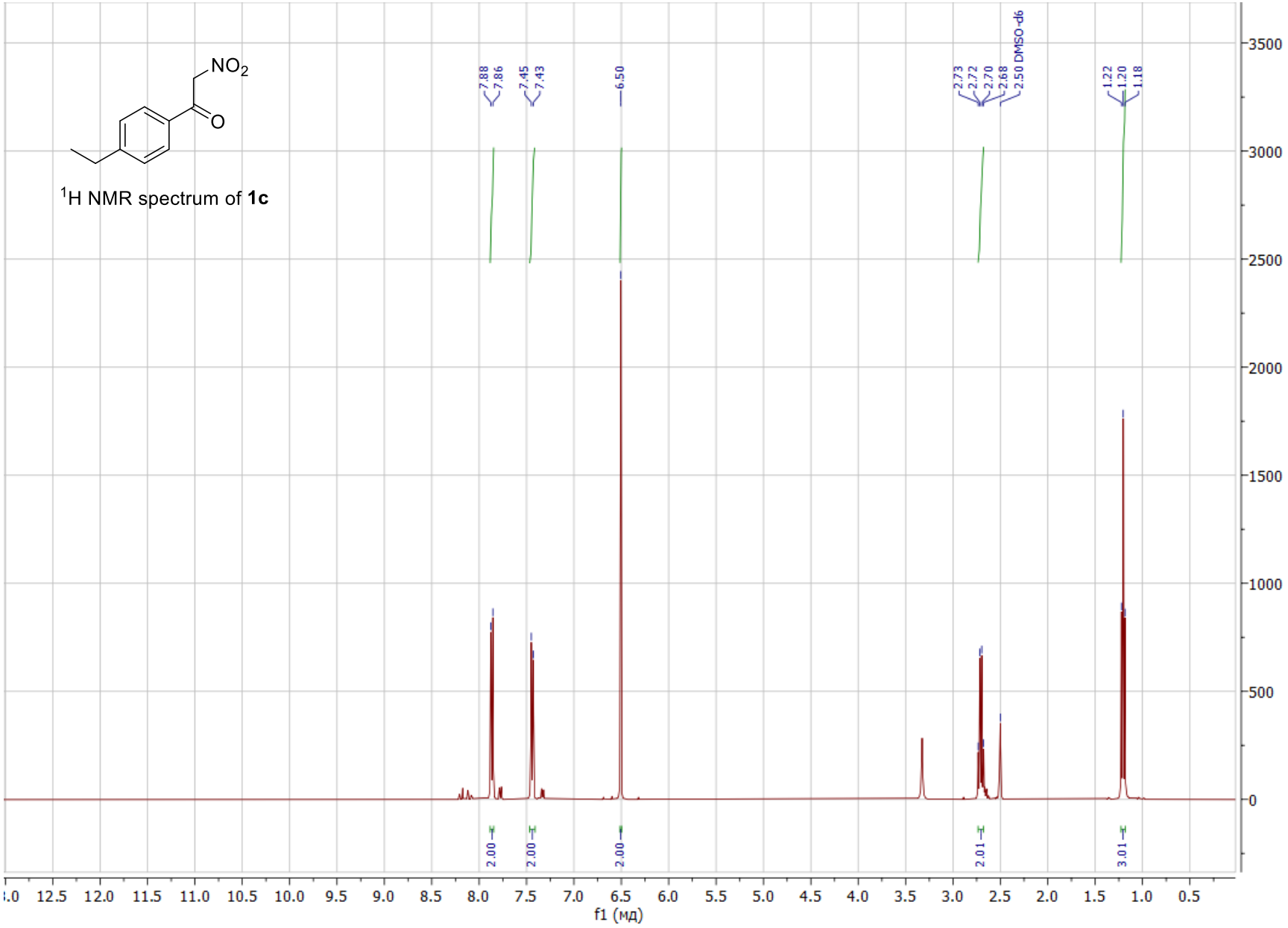
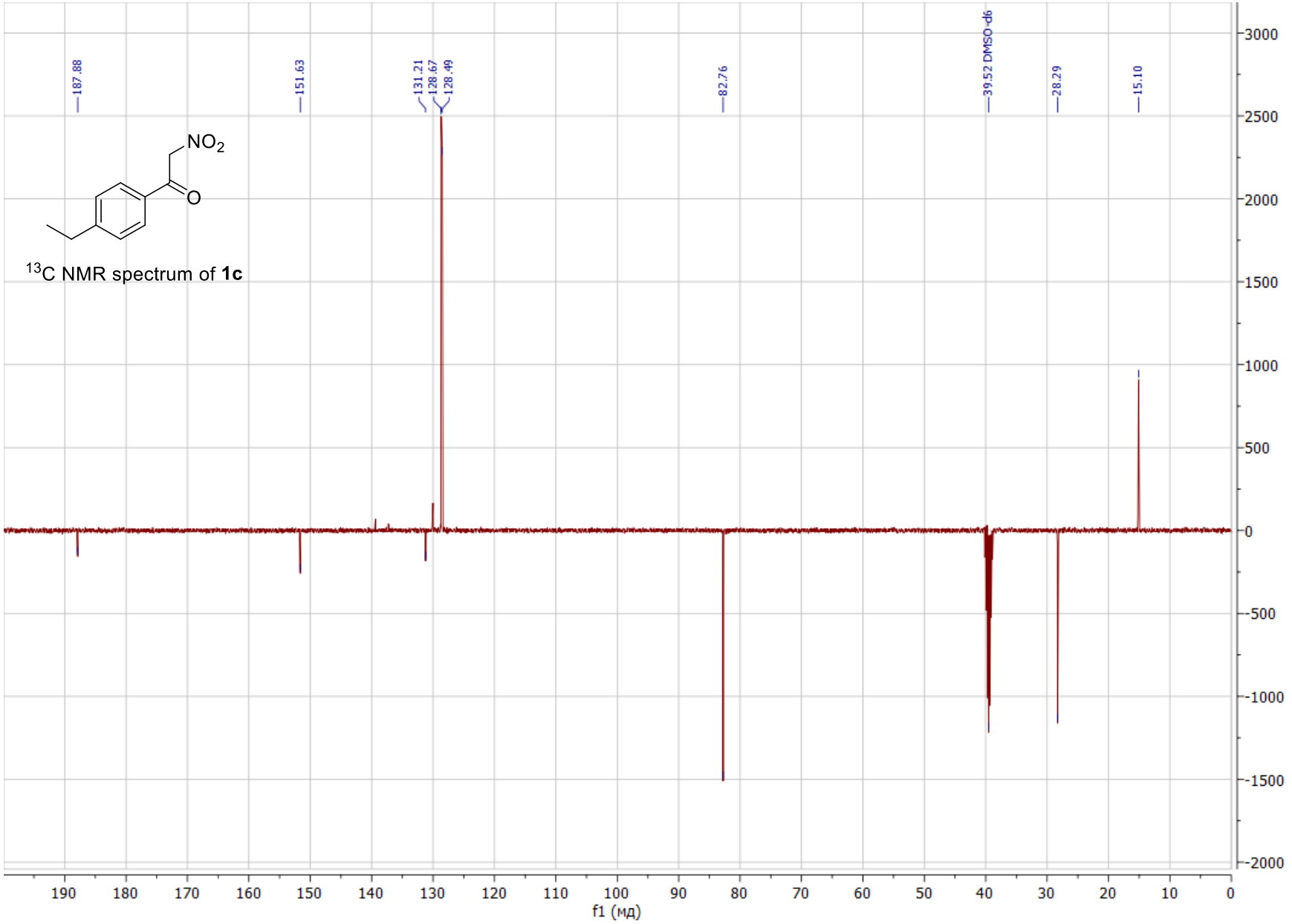
Supporting Information

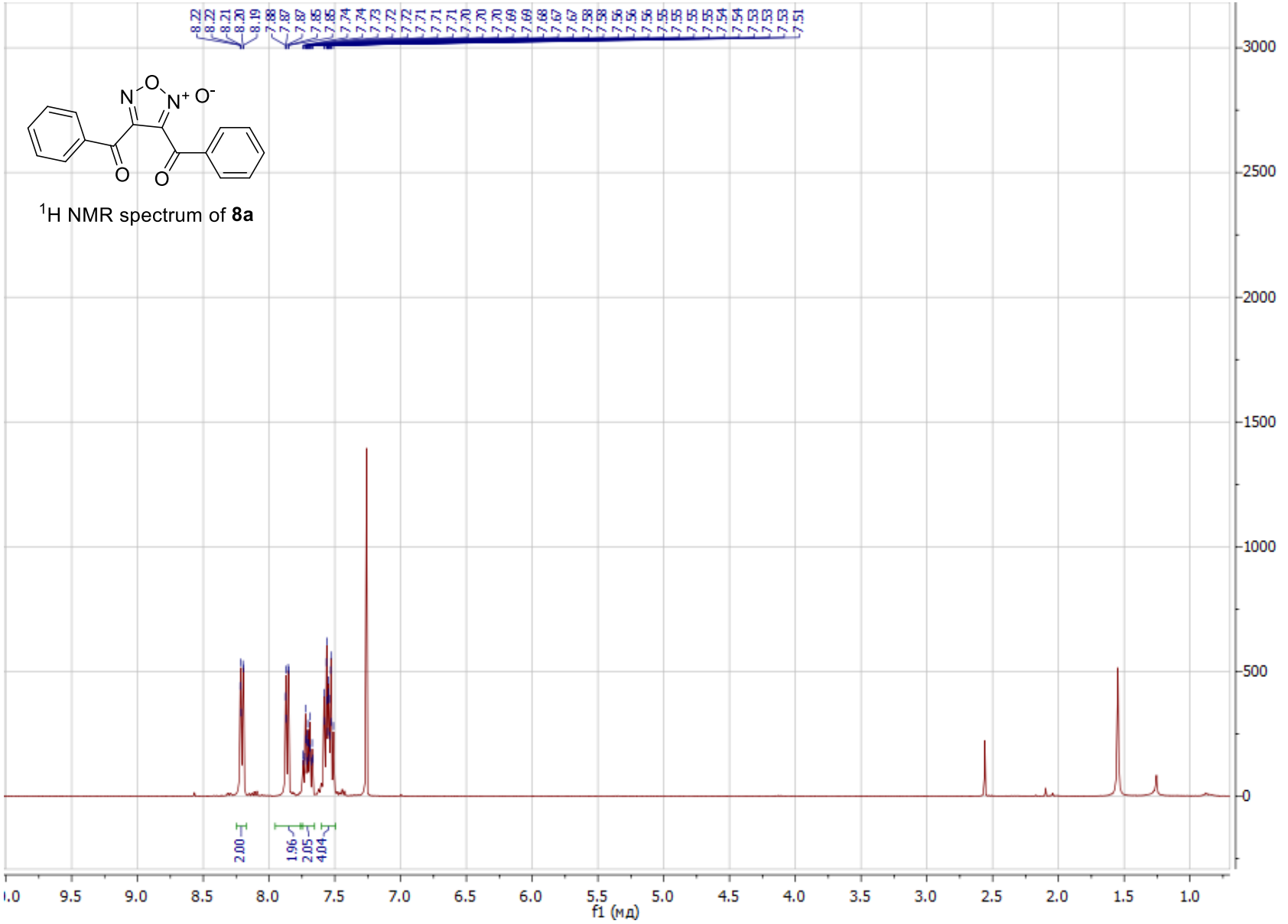
^{1}H and ^{13}C NMR spectral charts for starting 2-nitro-4'-ethylacetophenone (1c)



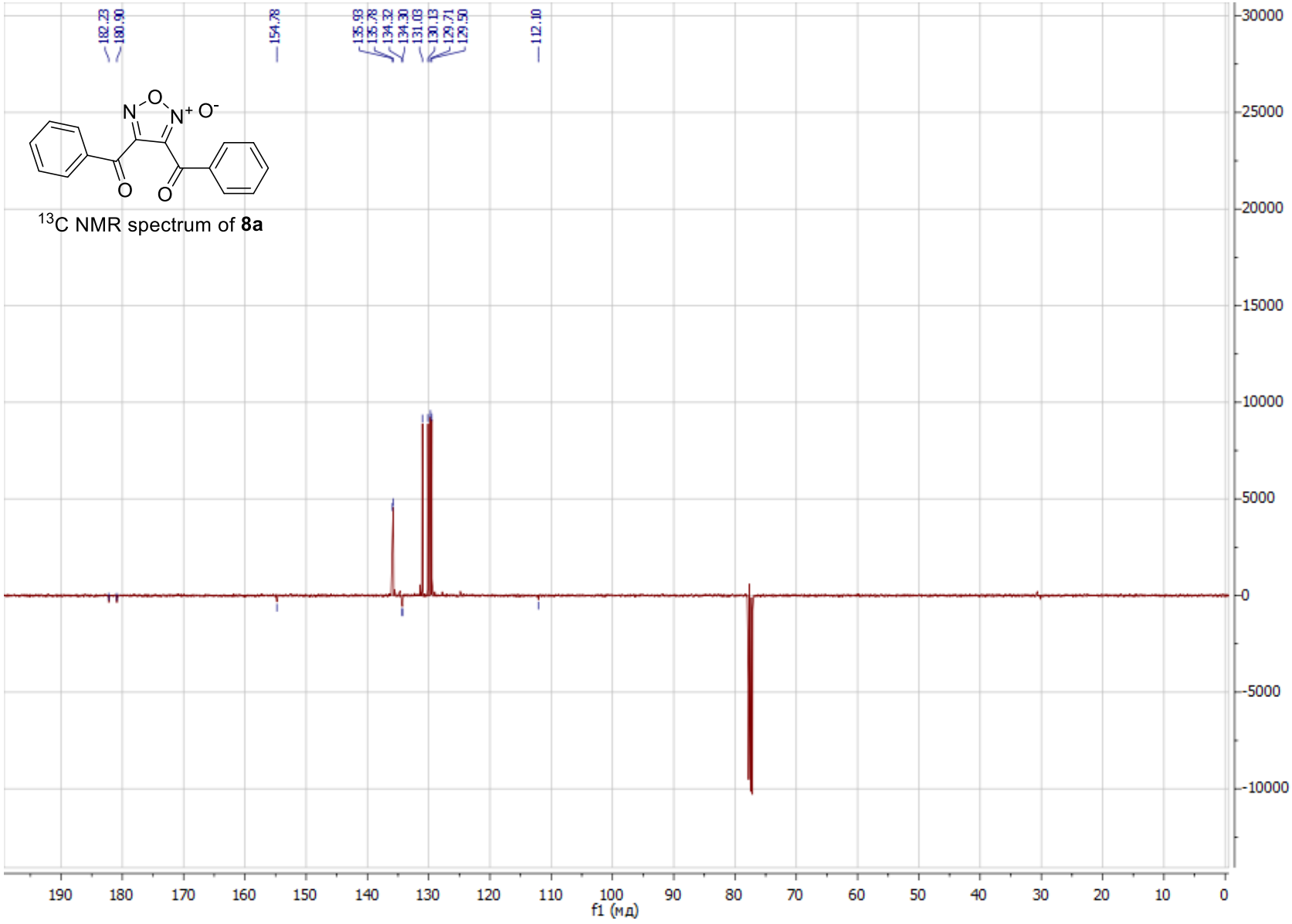


^{13}C NMR spectrum of **1c**

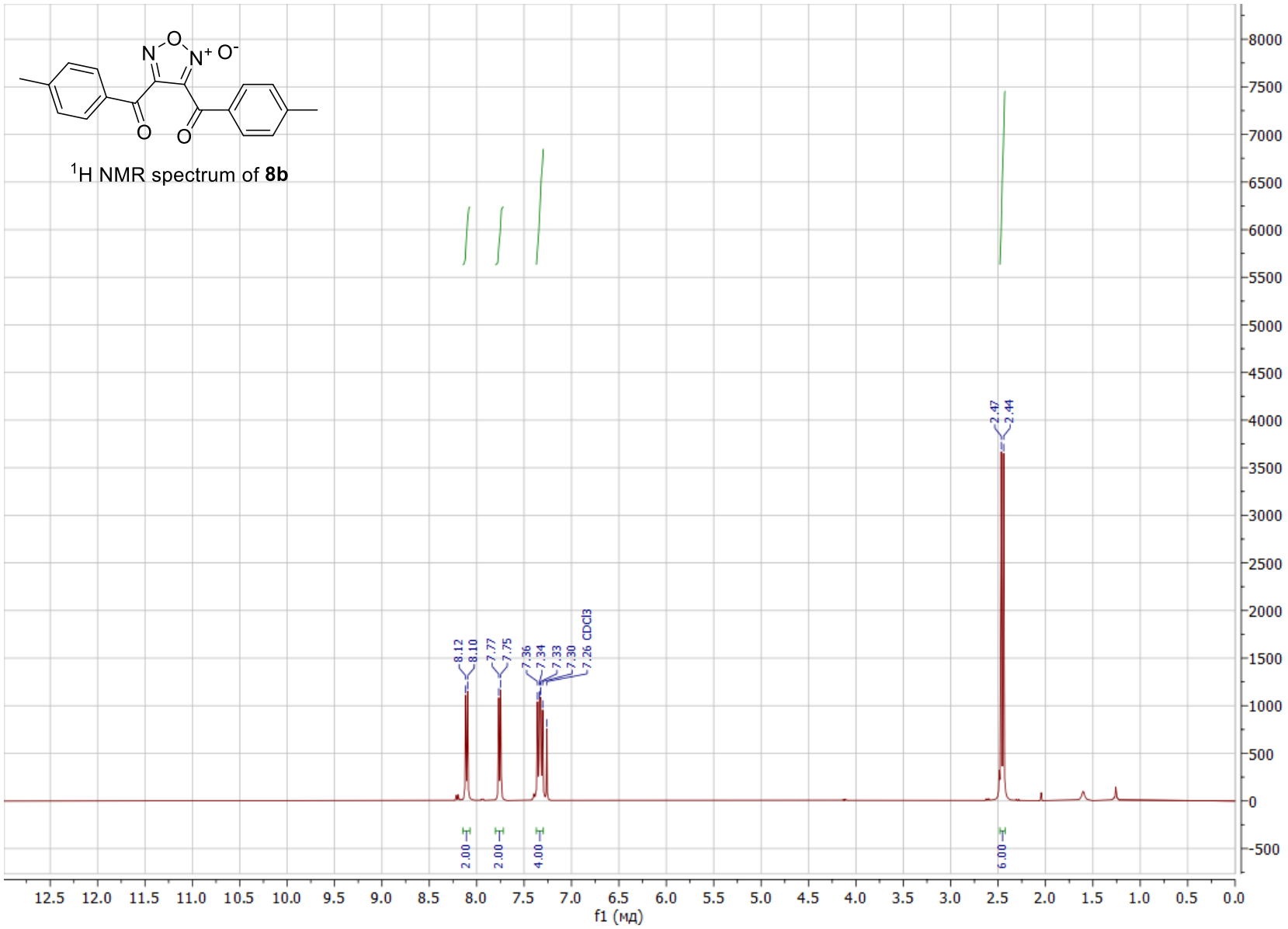
^{1}H and ^{13}C NMR spectral charts for furoxans 8



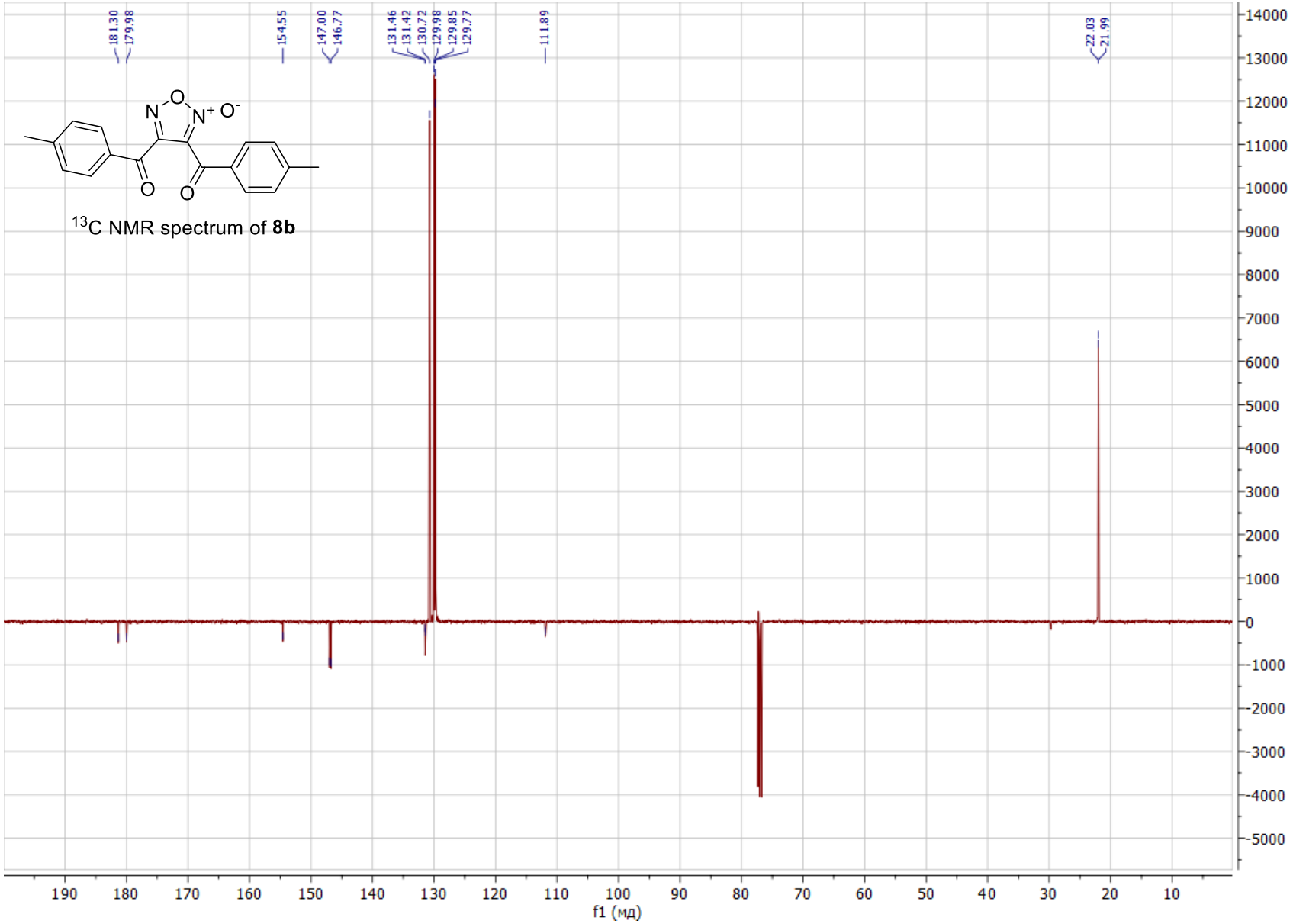
^{1}H NMR spectrum of **8a**



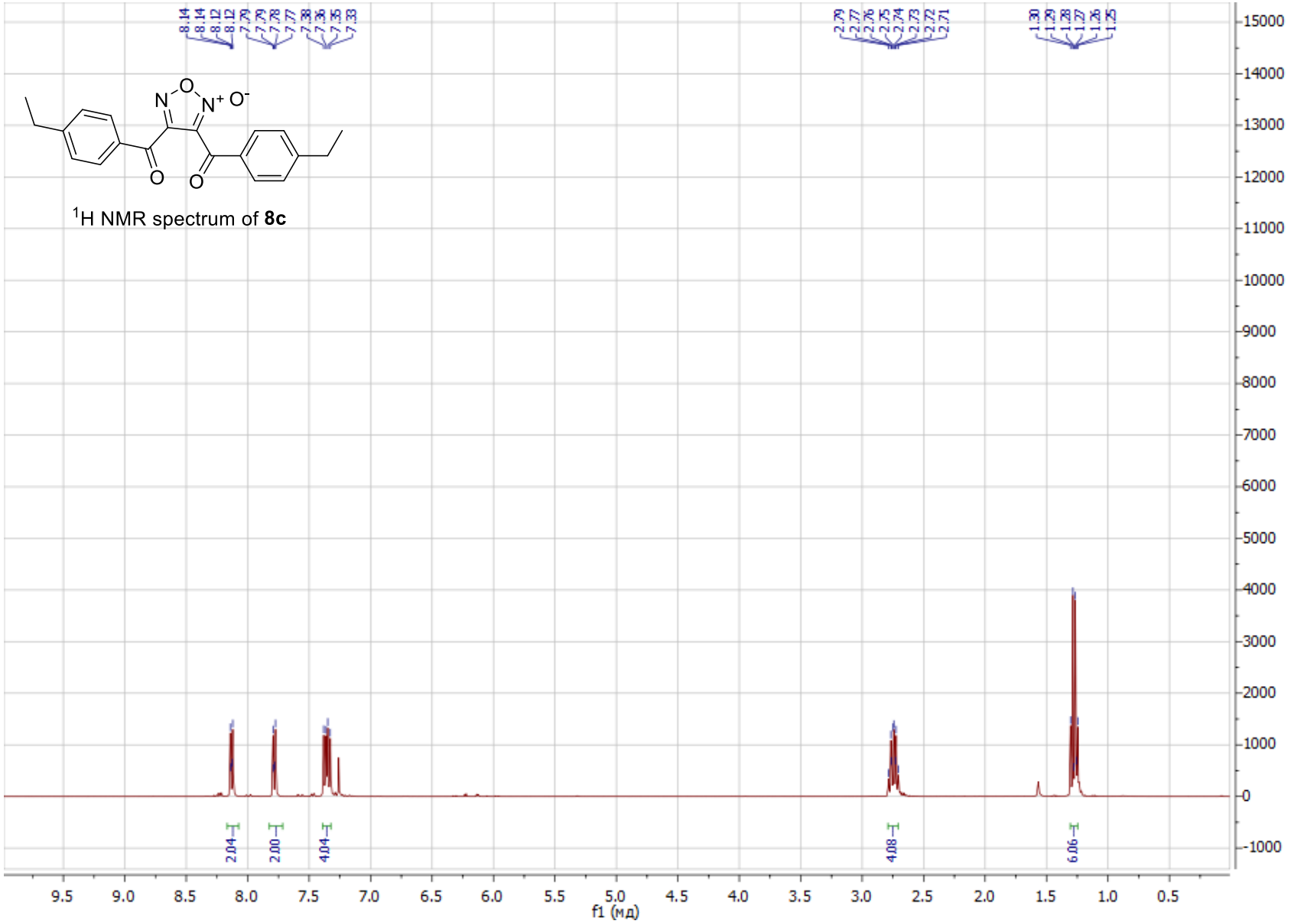
^{13}C NMR spectrum of **8a**



^{1}H NMR spectrum of **8b**

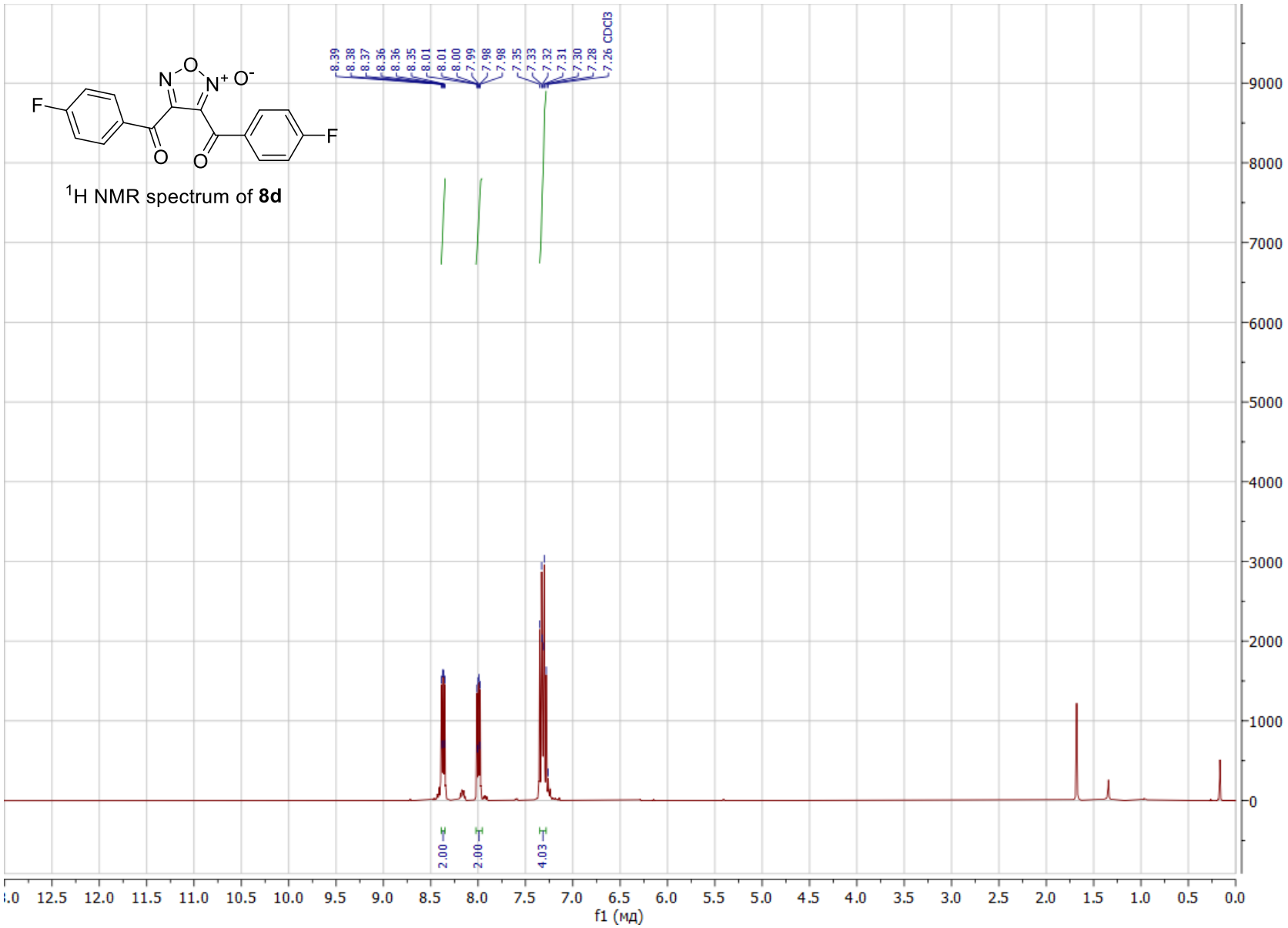


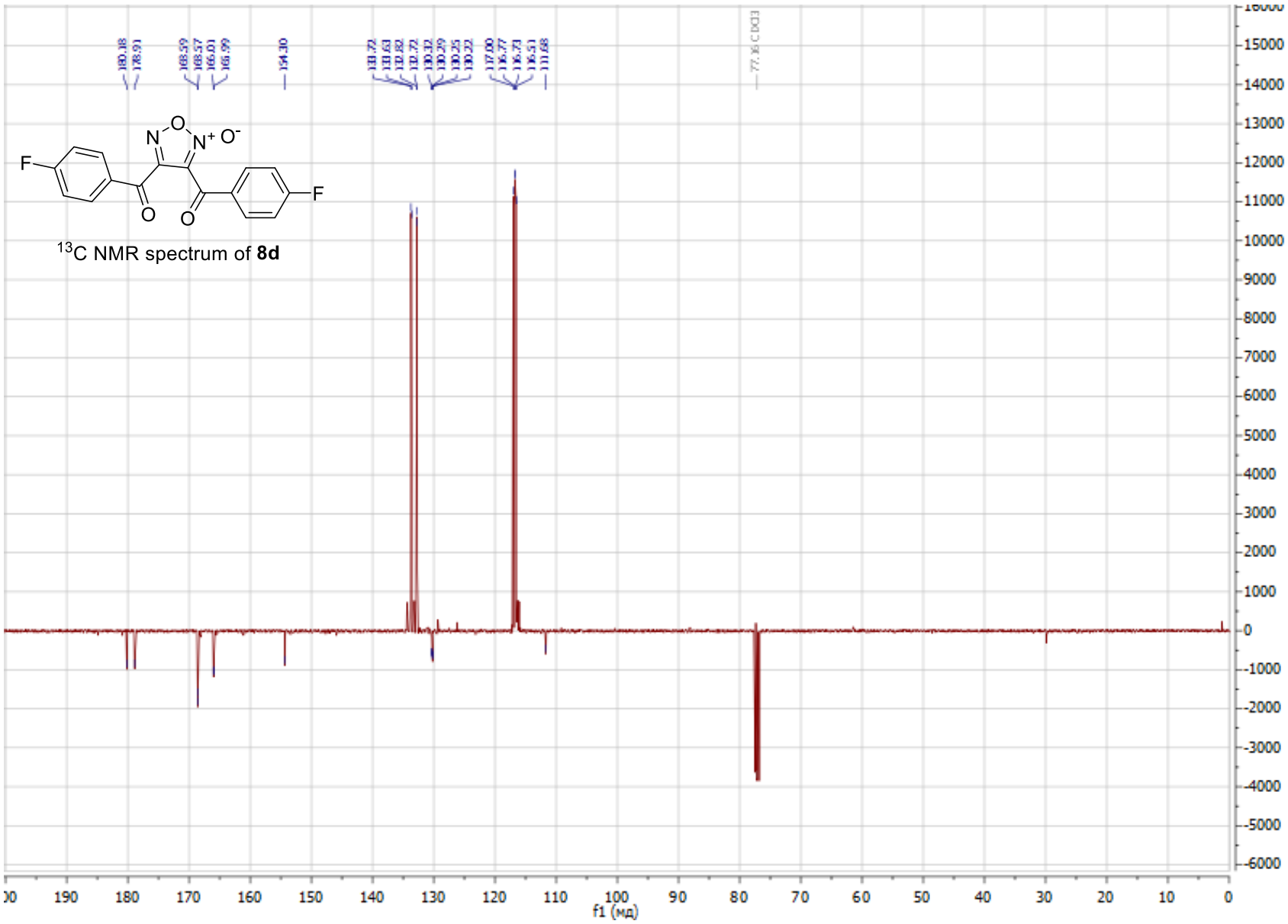
^{13}C NMR spectrum of **8b**

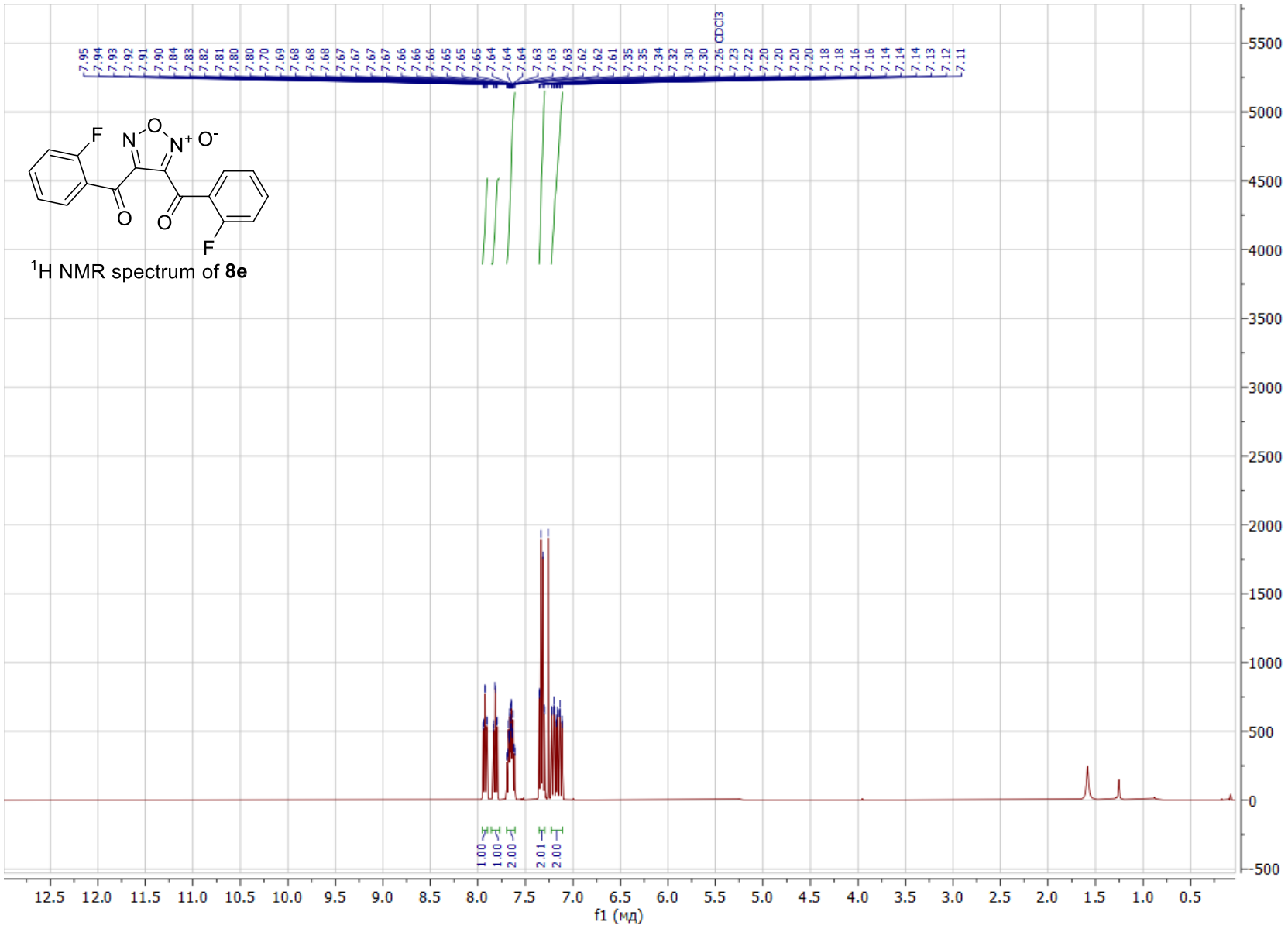


^{1}H NMR spectrum of **8c**

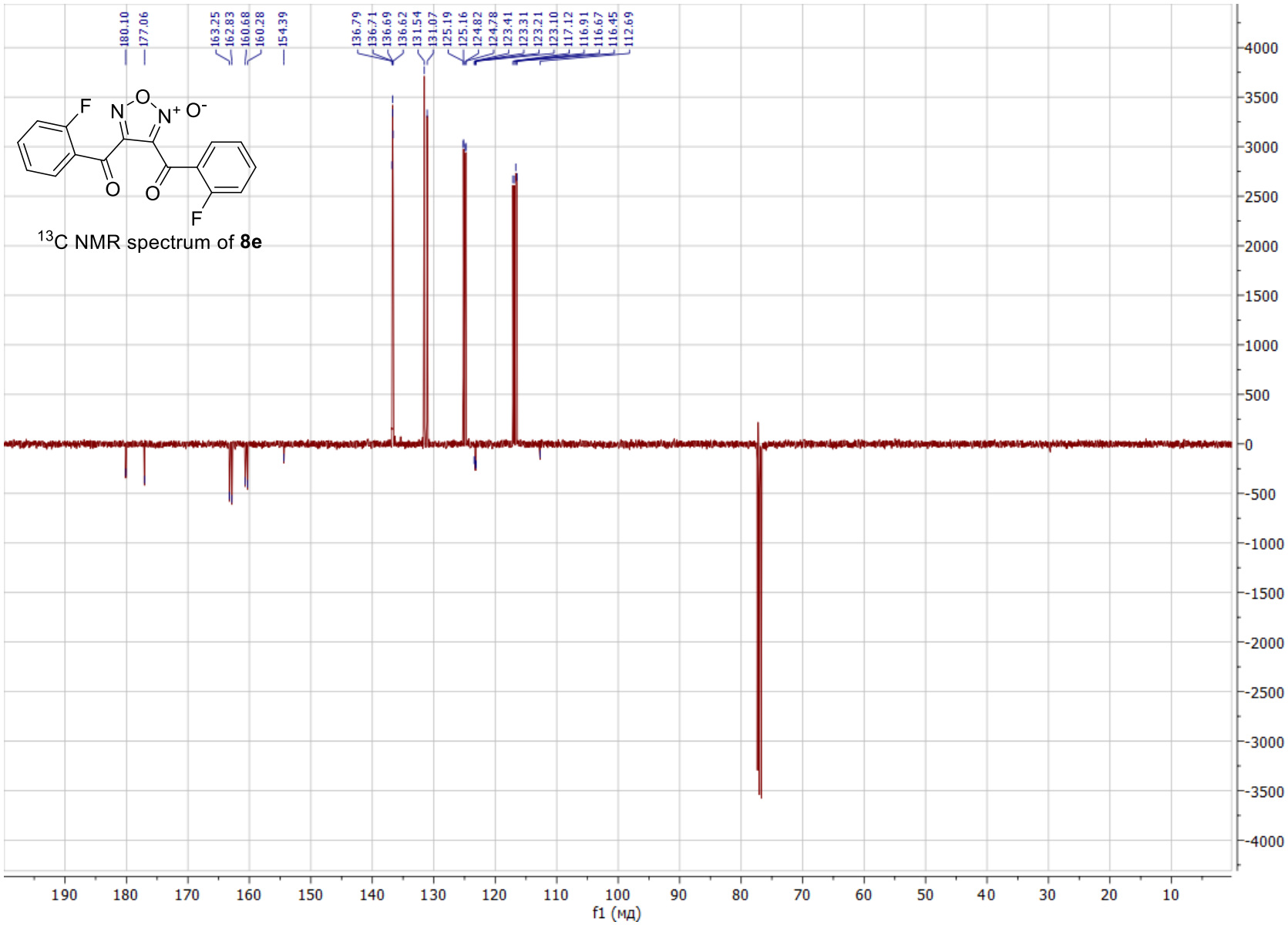
^{13}C NMR spectrum of **8c**


F
O
N
O
N+ O-
O
F
1H NMR spectrum of 8d
8.39
8.38
8.37
8.36
8.36
8.35
8.01
8.01
8.00
7.99
7.98
7.98
7.35
7.33
7.32
7.31
7.30
7.28
7.26 CDCl3
2.00
2.00
4.03
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
13.0 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
f1 (мд)

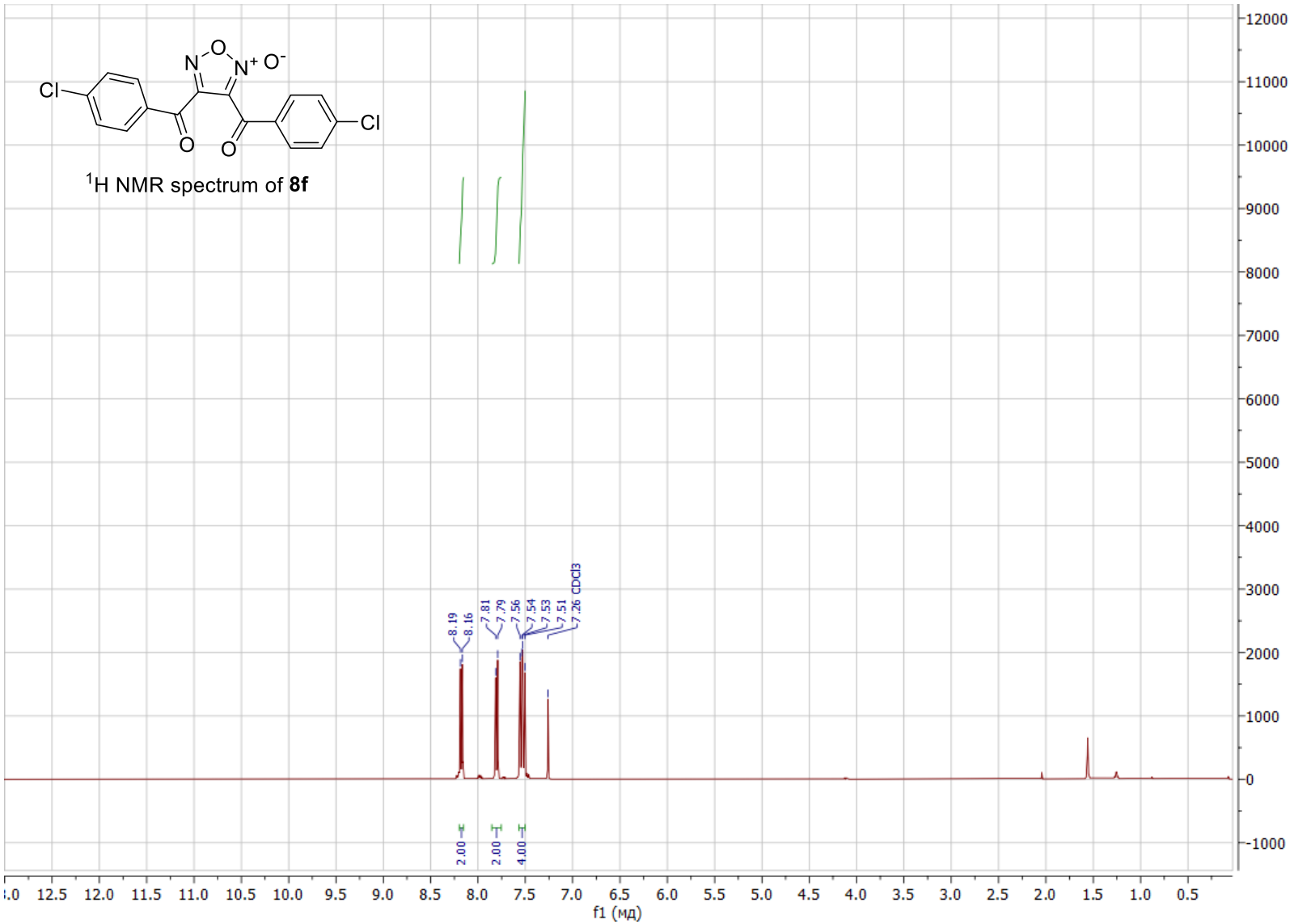

180.18
178.91
168.59
168.57
166.01
166.99
154.30
133.72
133.63
132.82
132.72
130.32
130.29
130.25
130.22
117.00
116.77
116.73
116.51
111.68
77.36 CDCl3
F
O
O
N
O
N+ O-
F
13C NMR spectrum of 8d
f1 (мд)



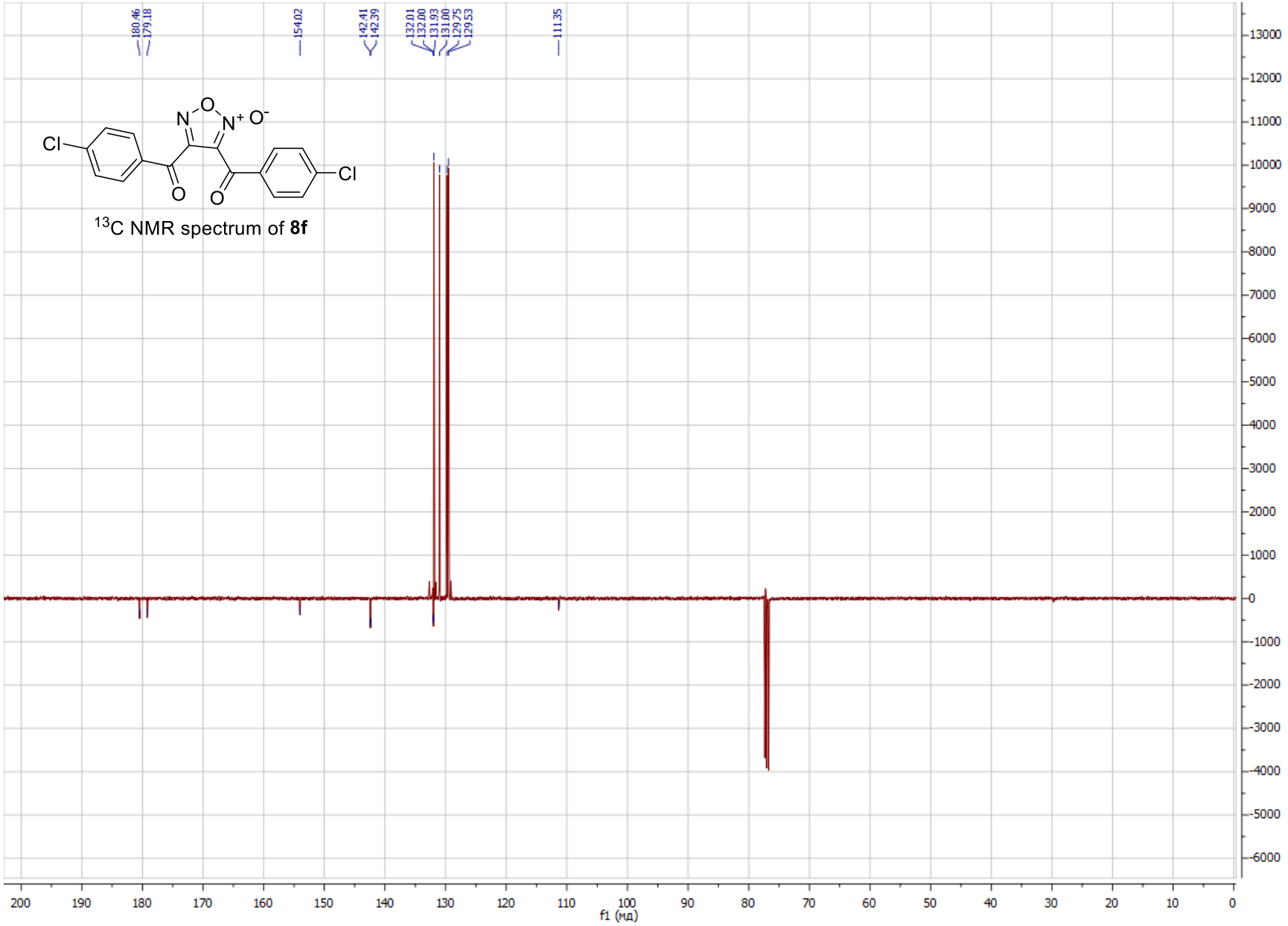
^{1}H NMR spectrum of **8e**



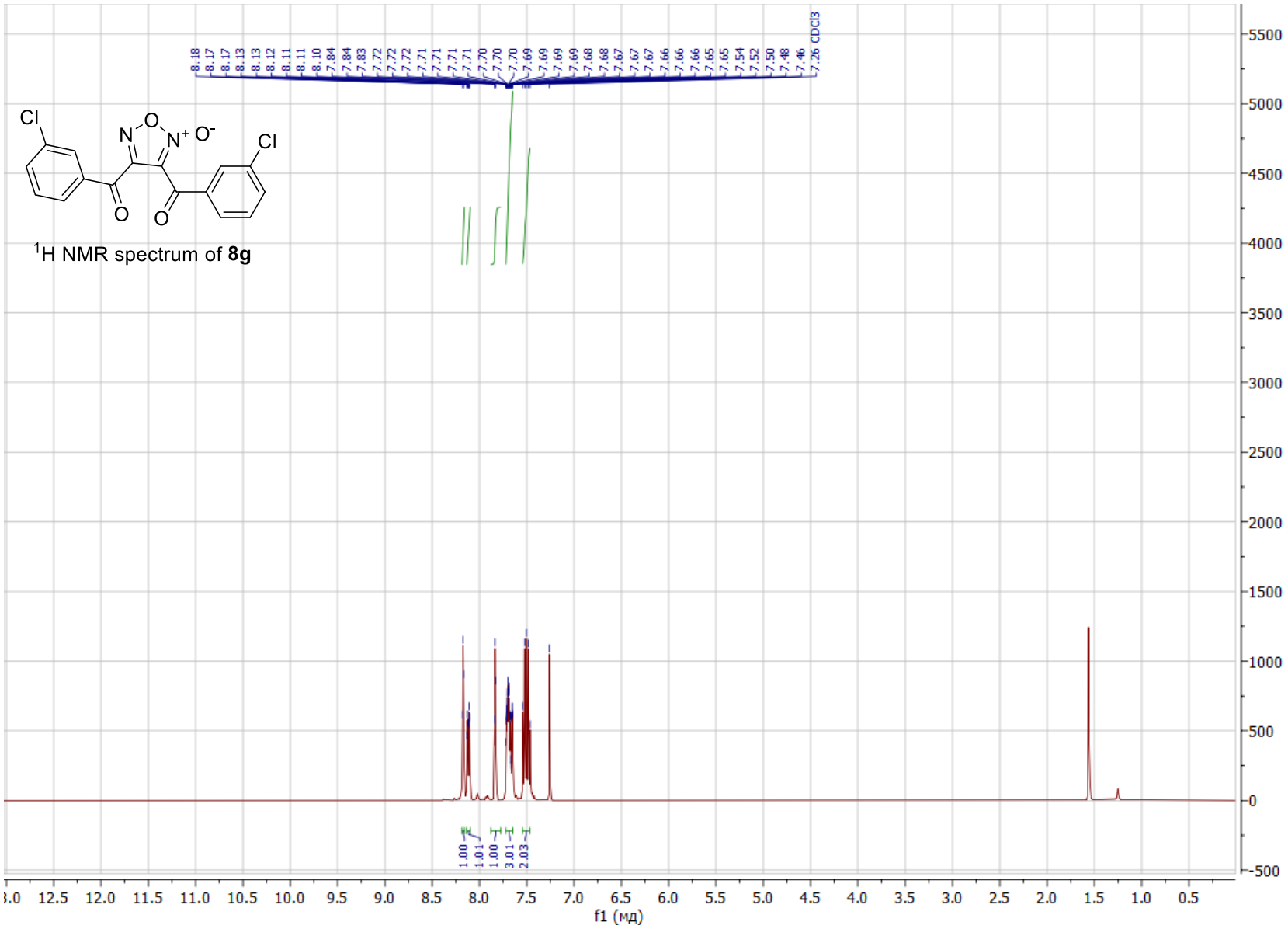
^{13}C NMR spectrum of **8e**



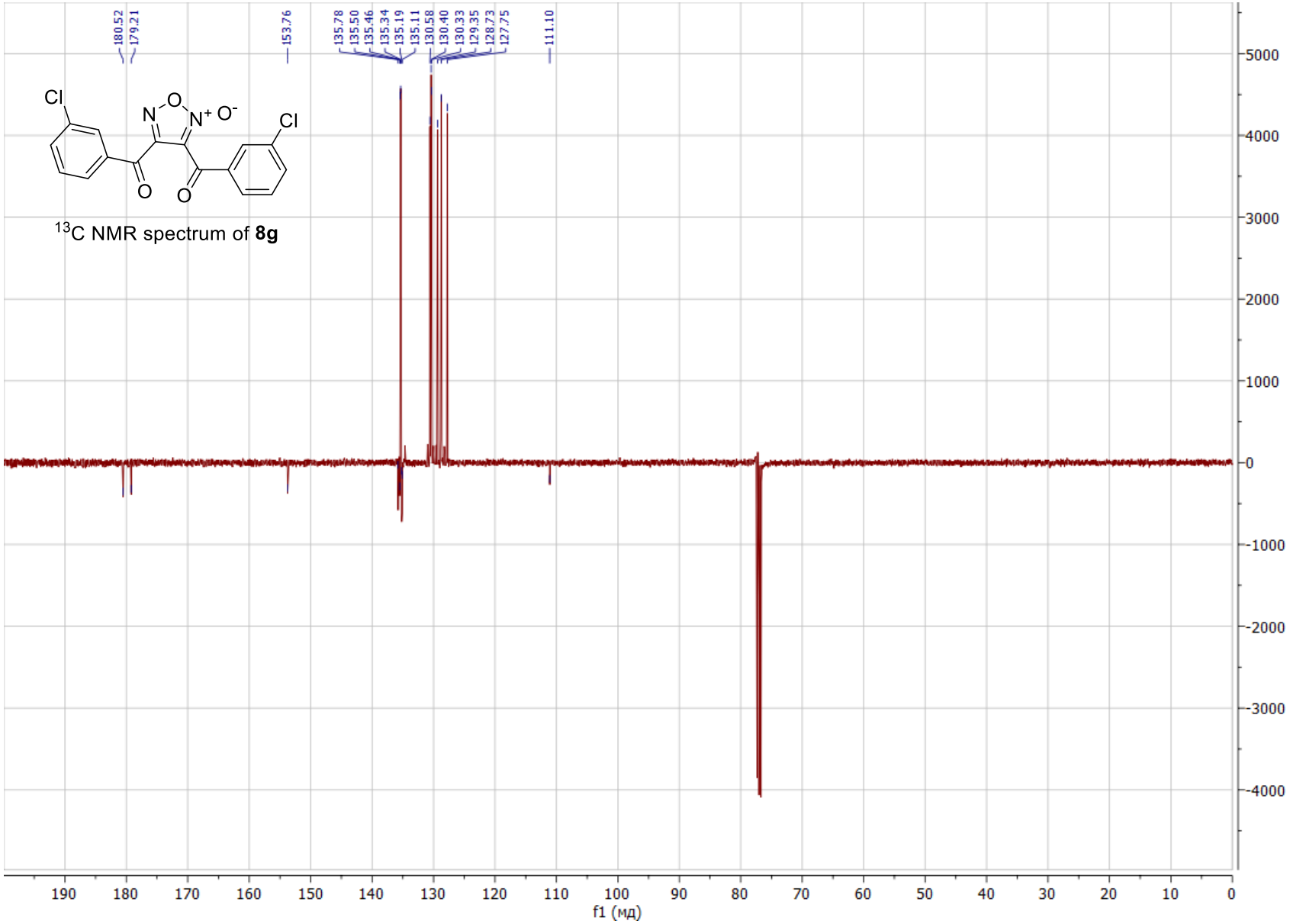
^{1}H NMR spectrum of **8f**



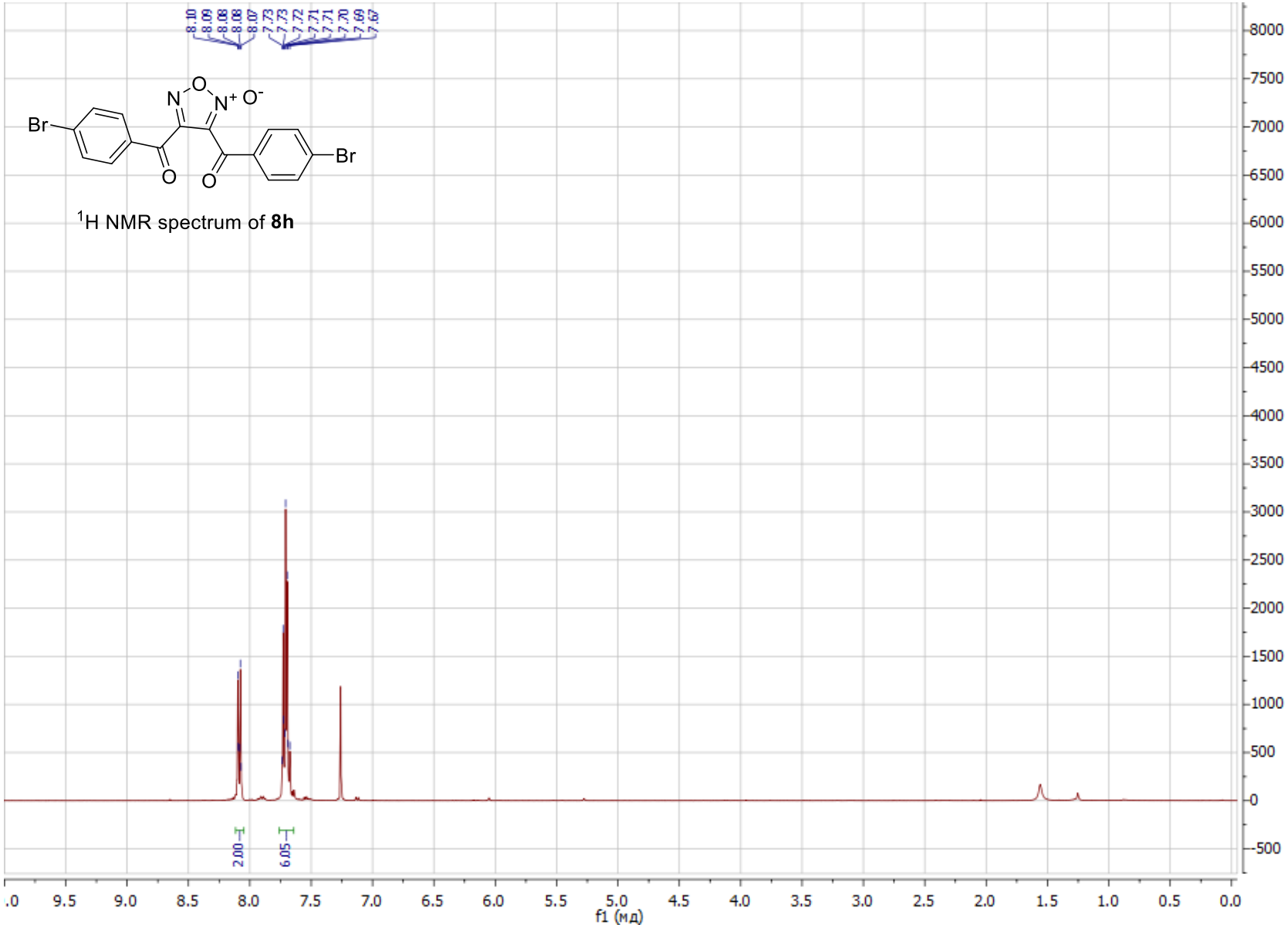
^{13}C NMR spectrum of **8f**



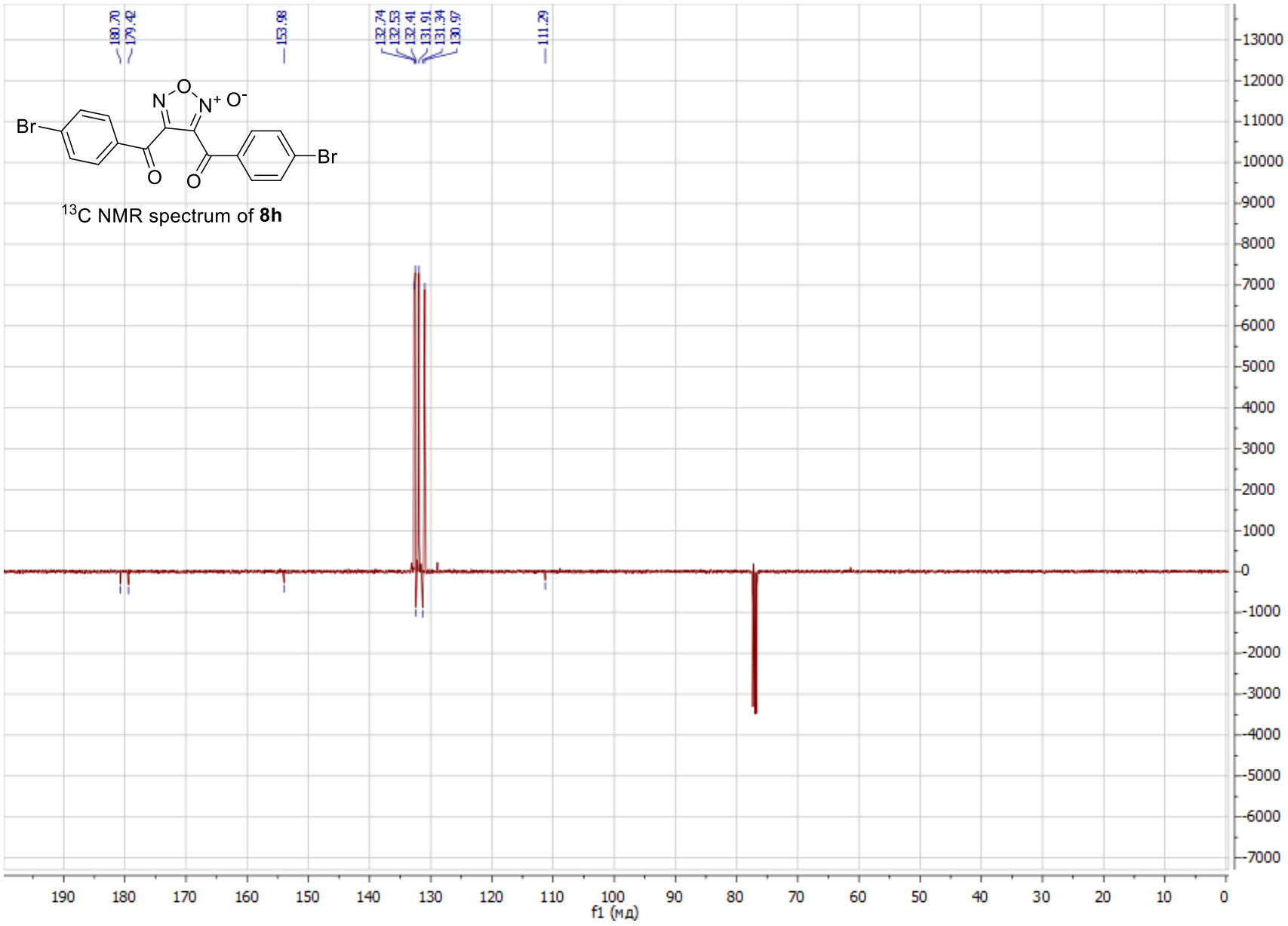
^{1}H NMR spectrum of **8g**



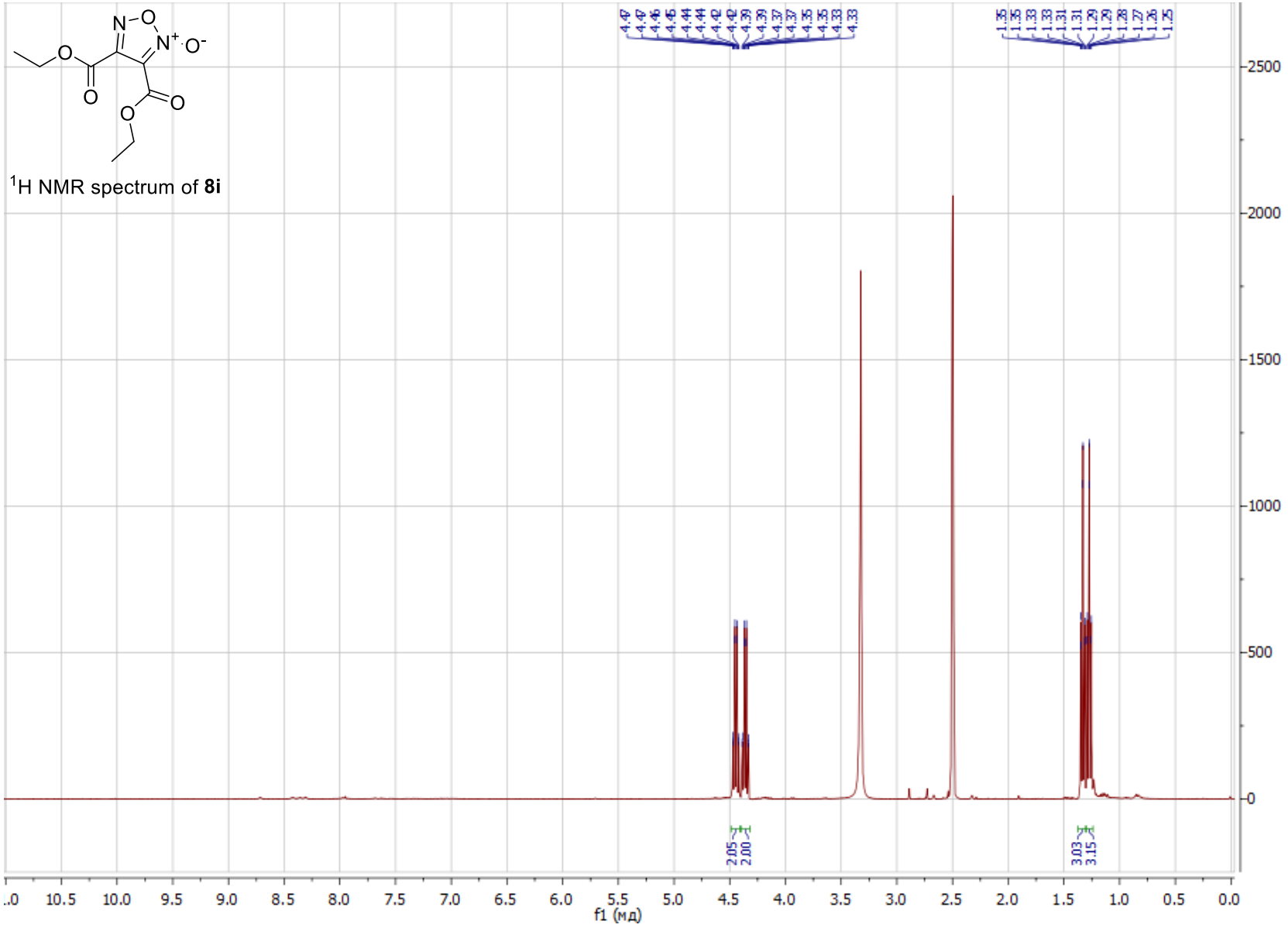
^{13}C NMR spectrum of **8g**



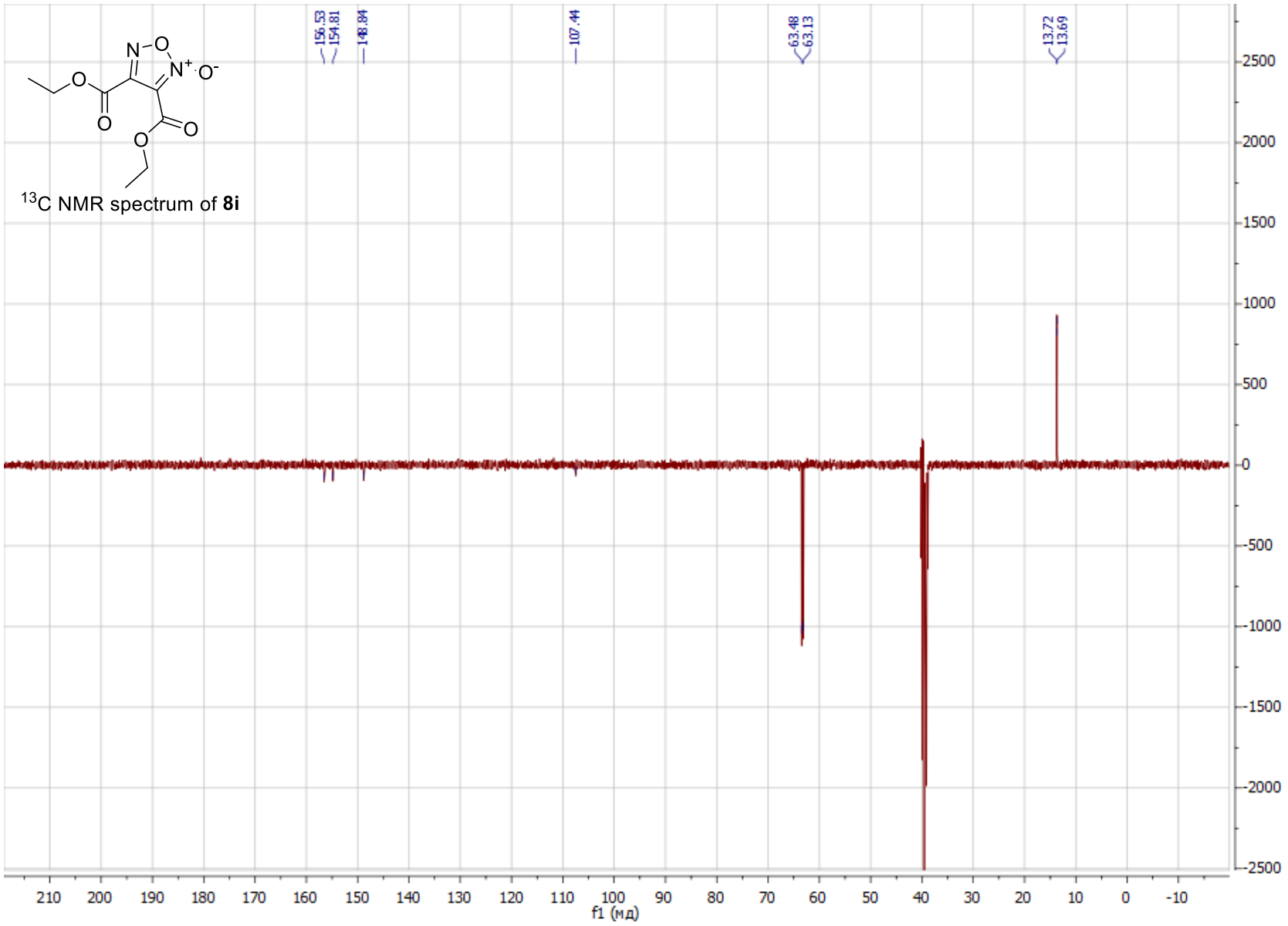
^{1}H NMR spectrum of **8h**



^{13}C NMR spectrum of **8h**

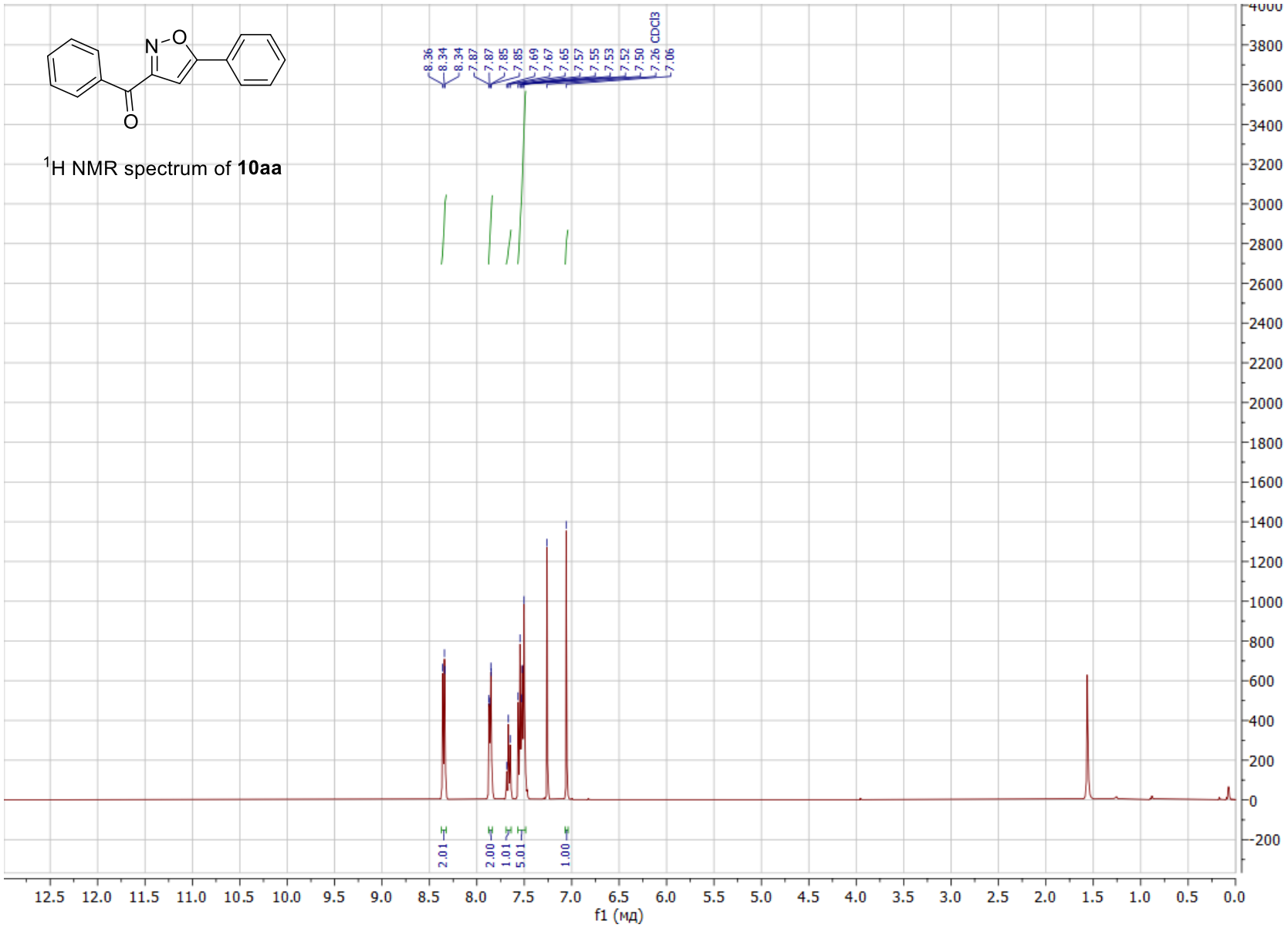


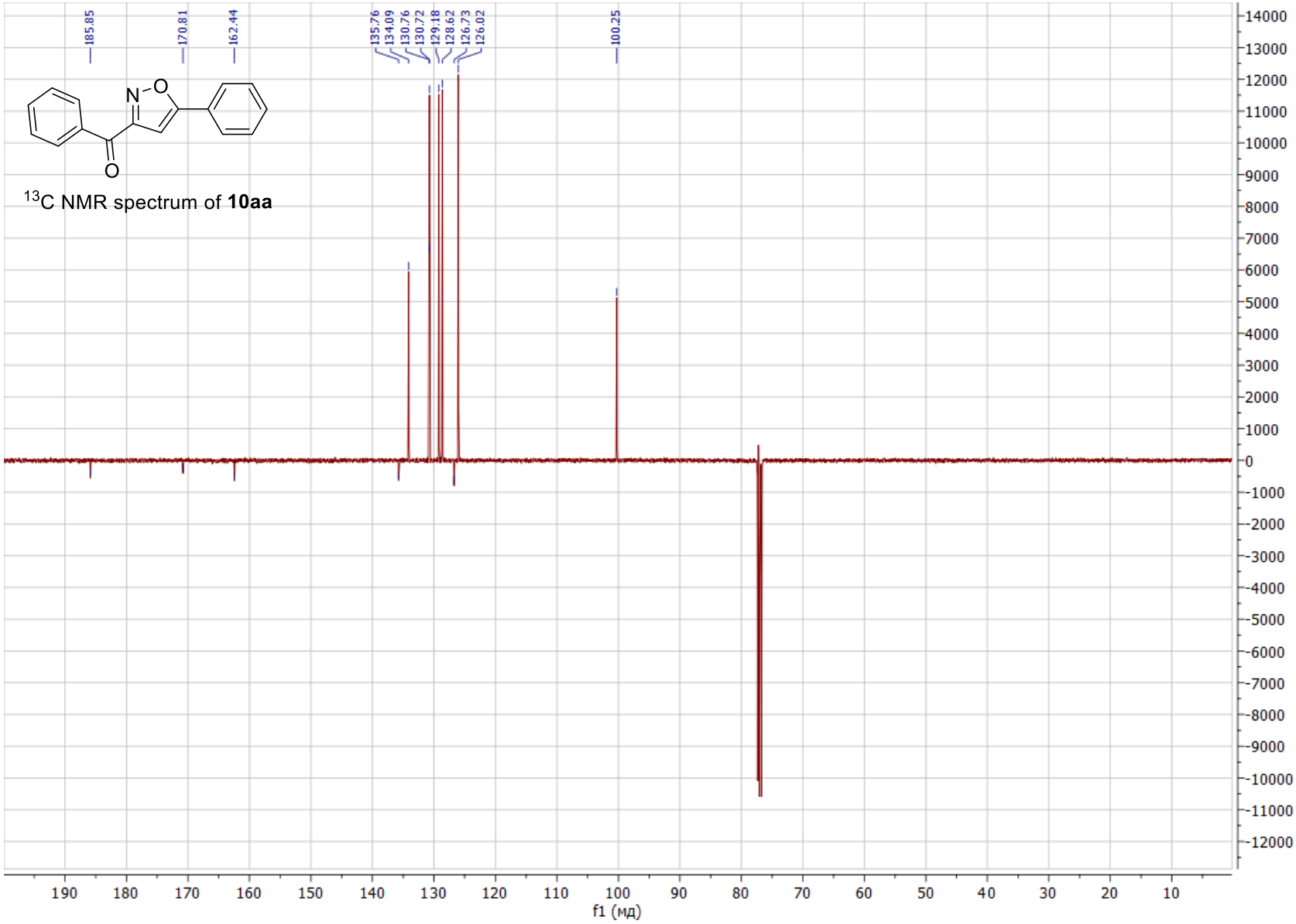
^{1}H NMR spectrum of **8i**

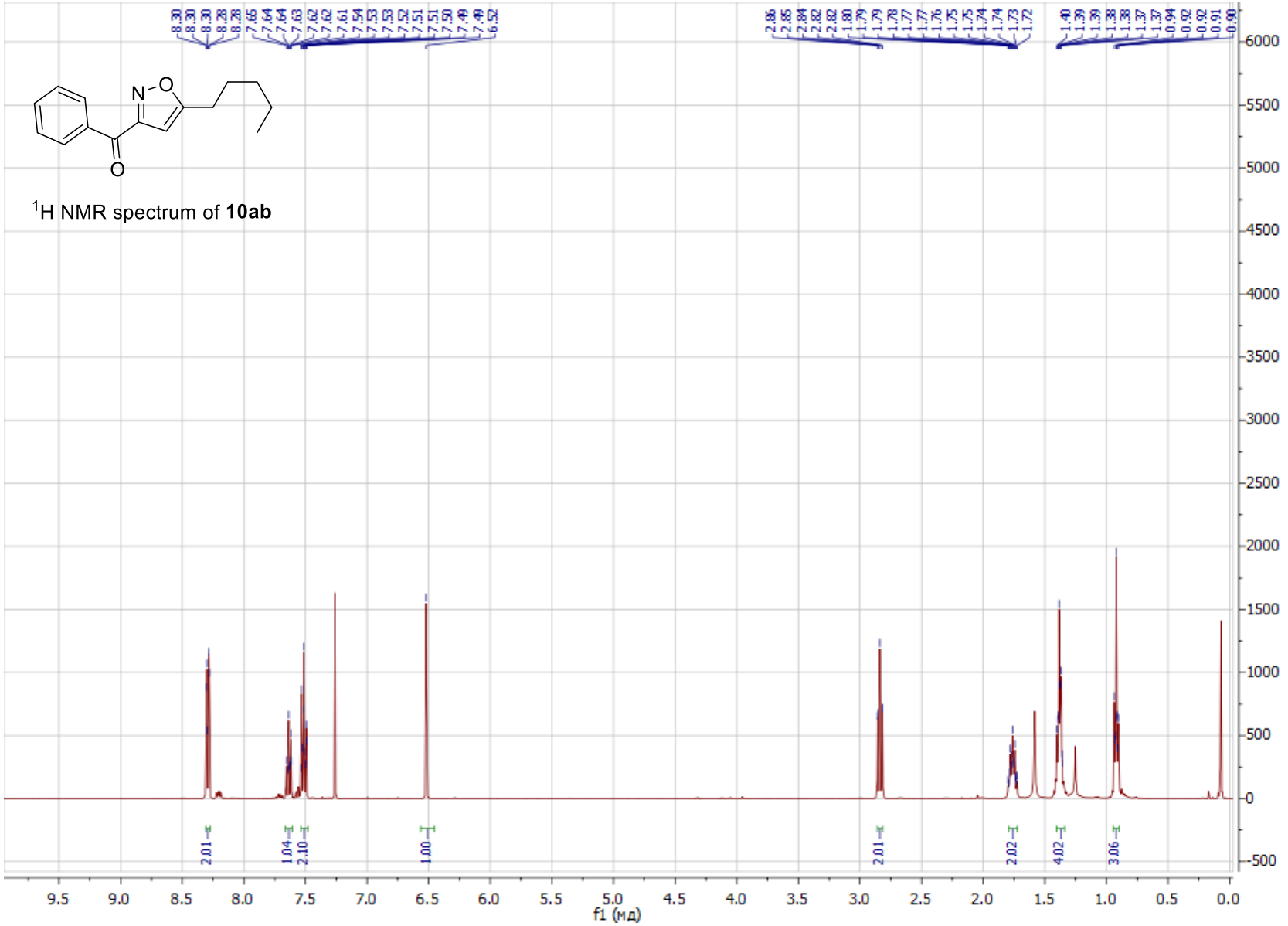


^{13}C NMR spectrum of **8i**

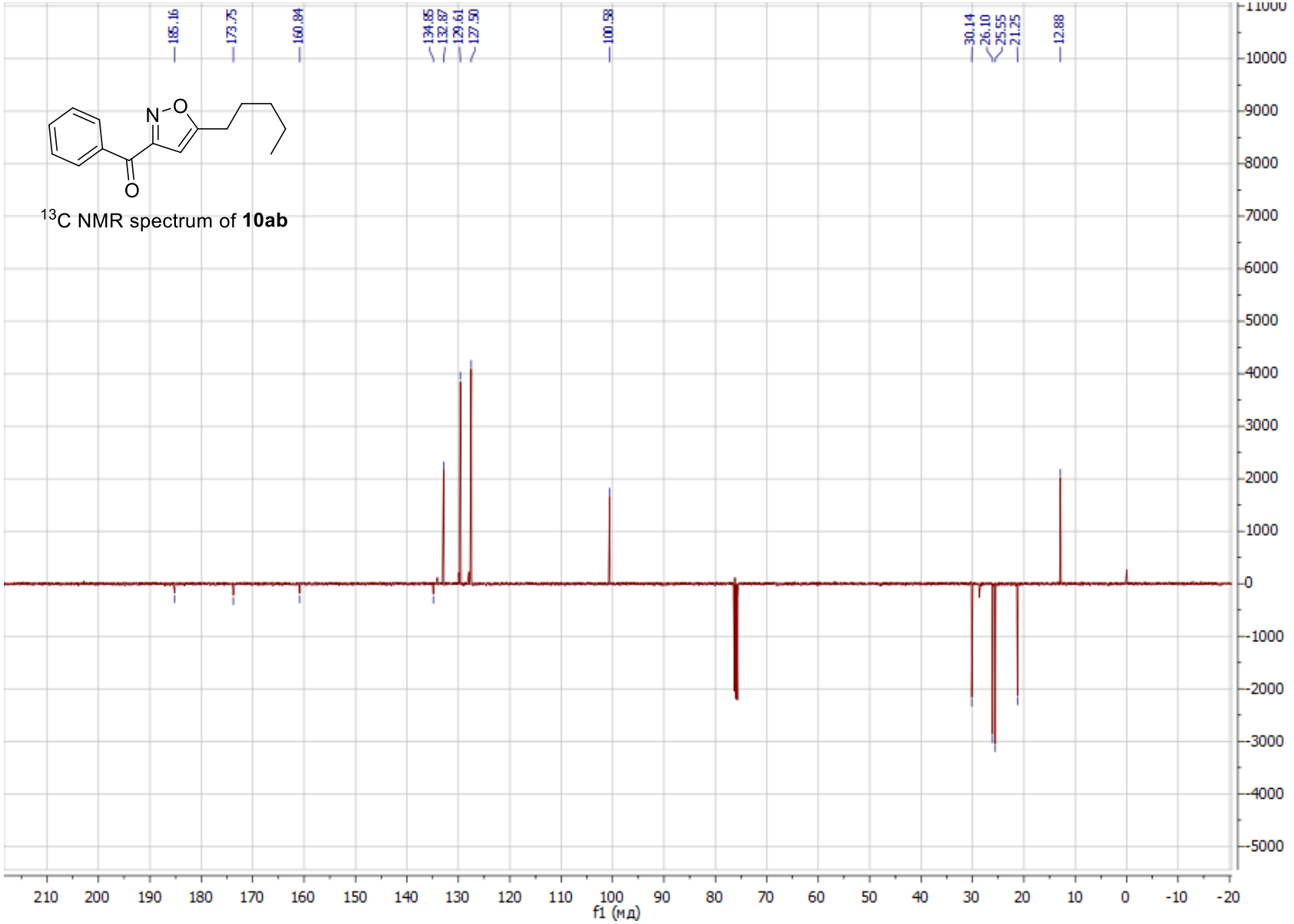
^{1}H and ^{13}C NMR spectral charts for isoxazoles 10



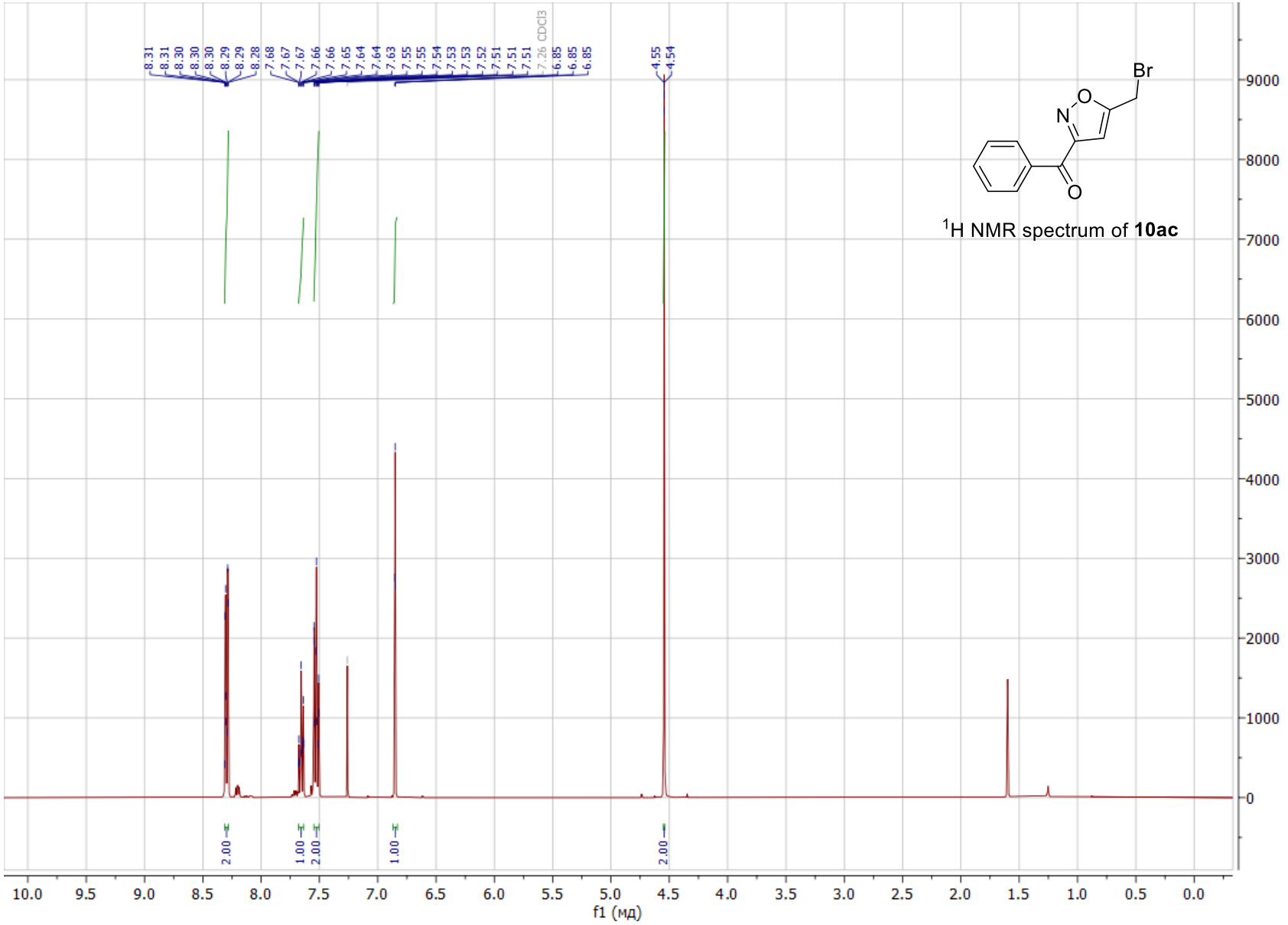

185.85
170.81
162.44
135.76
134.09
130.76
130.72
129.18
128.62
126.73
126.02
100.25
N
O
O
^{13}C NMR spectrum of **10aa**
f1 (мд)



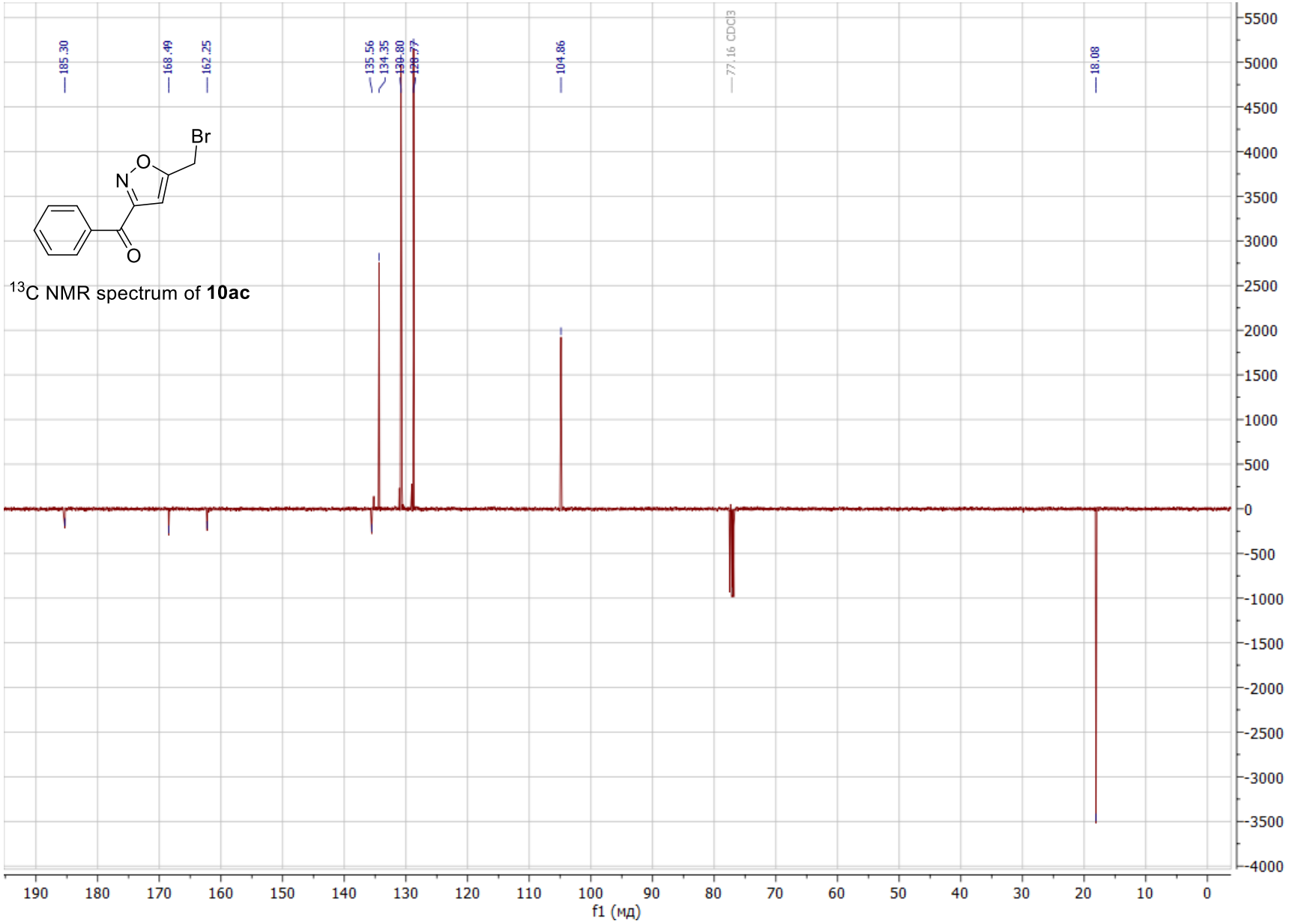
^{1}H NMR spectrum of **10ab**



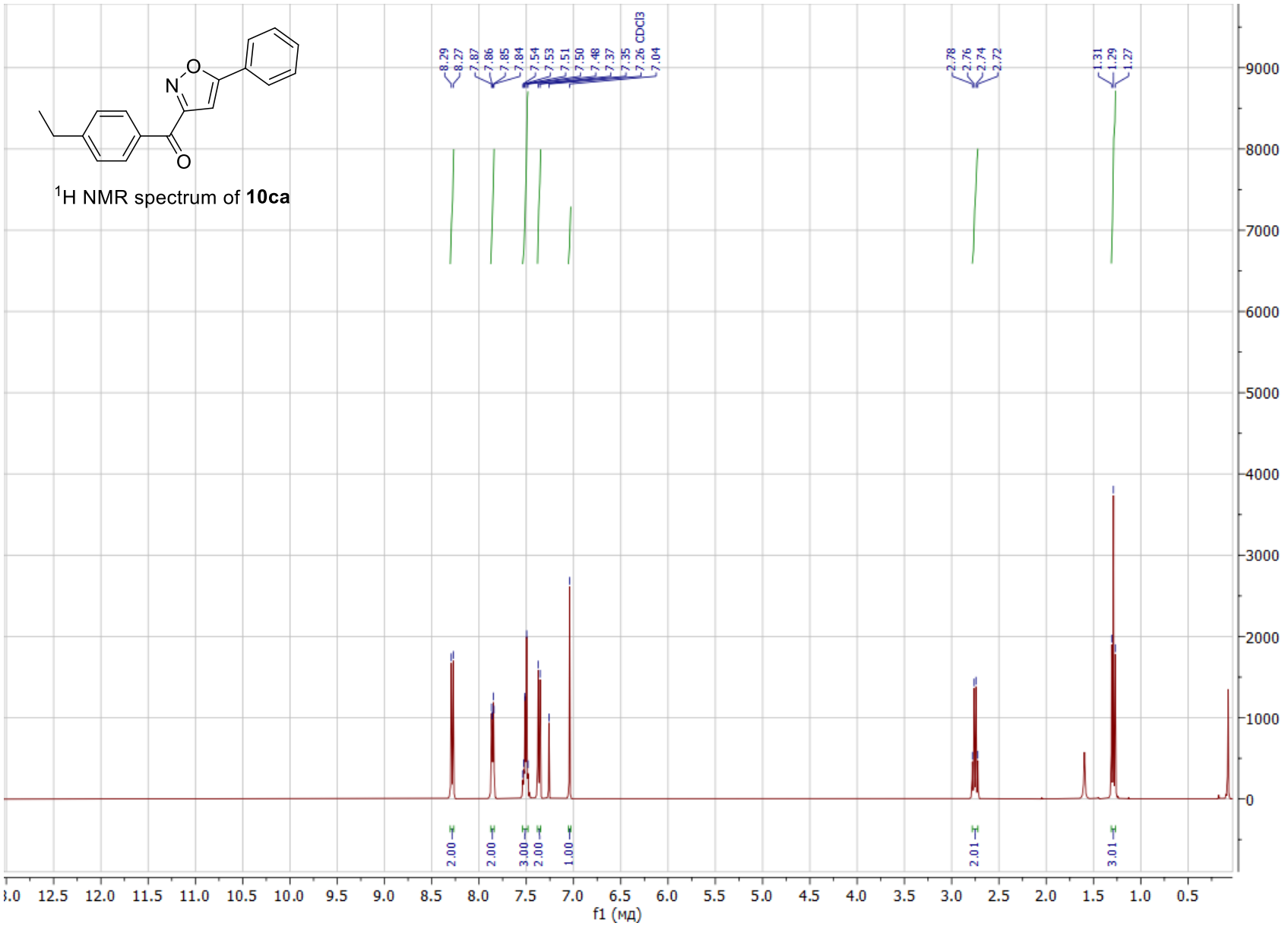
^{13}C NMR spectrum of **10ab**



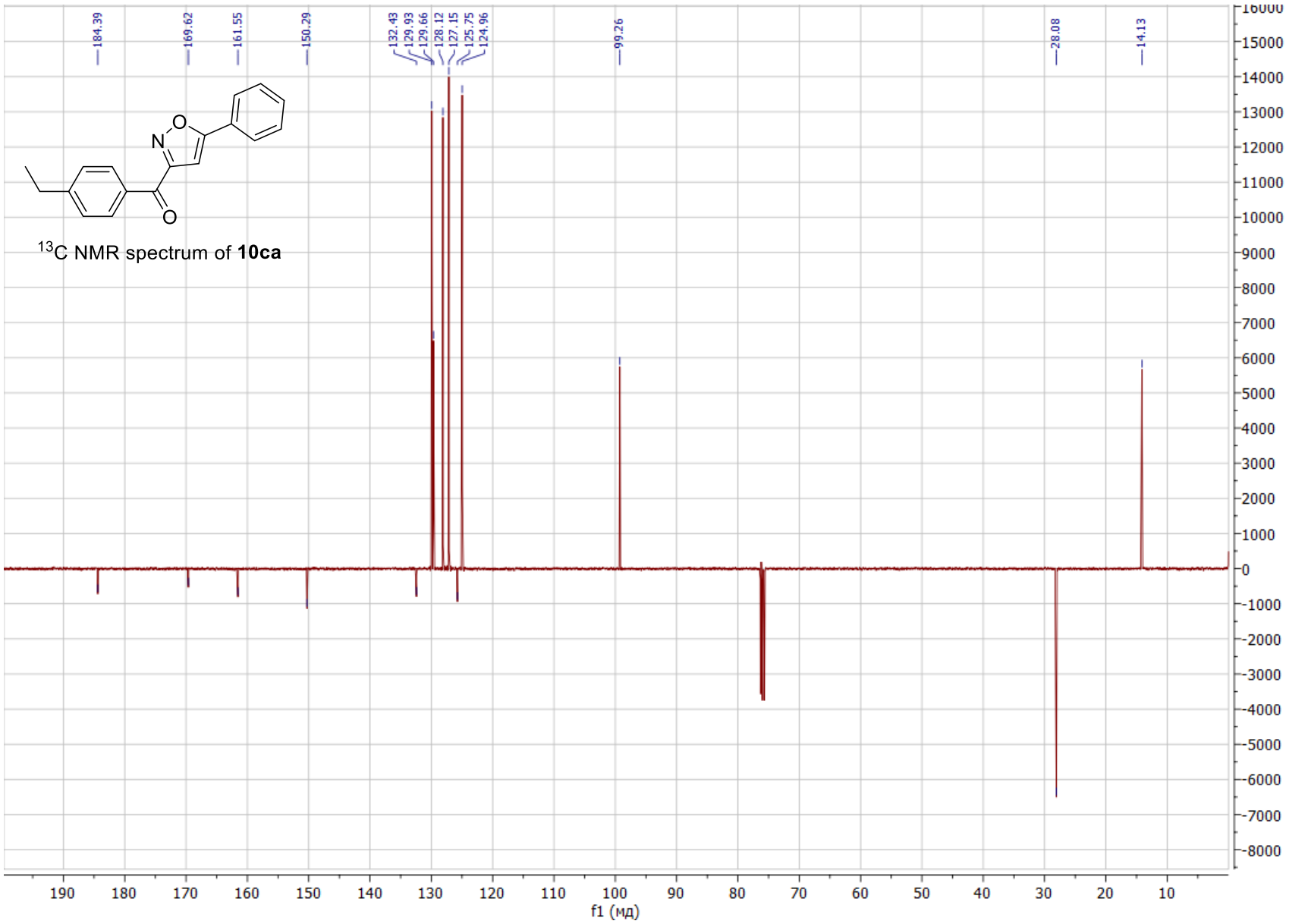
^{1}H NMR spectrum of **10ac**



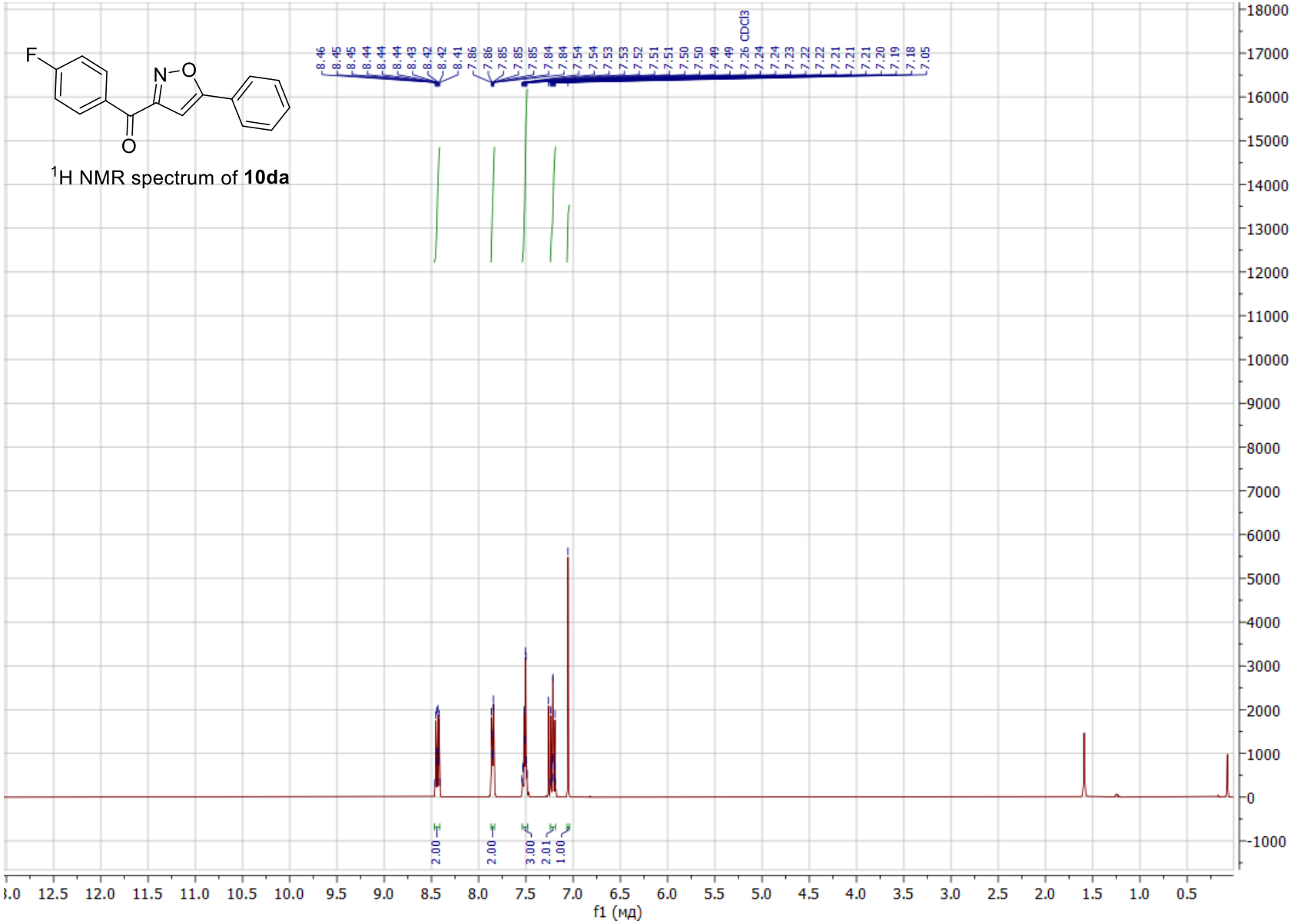
^{13}C NMR spectrum of **10ac**

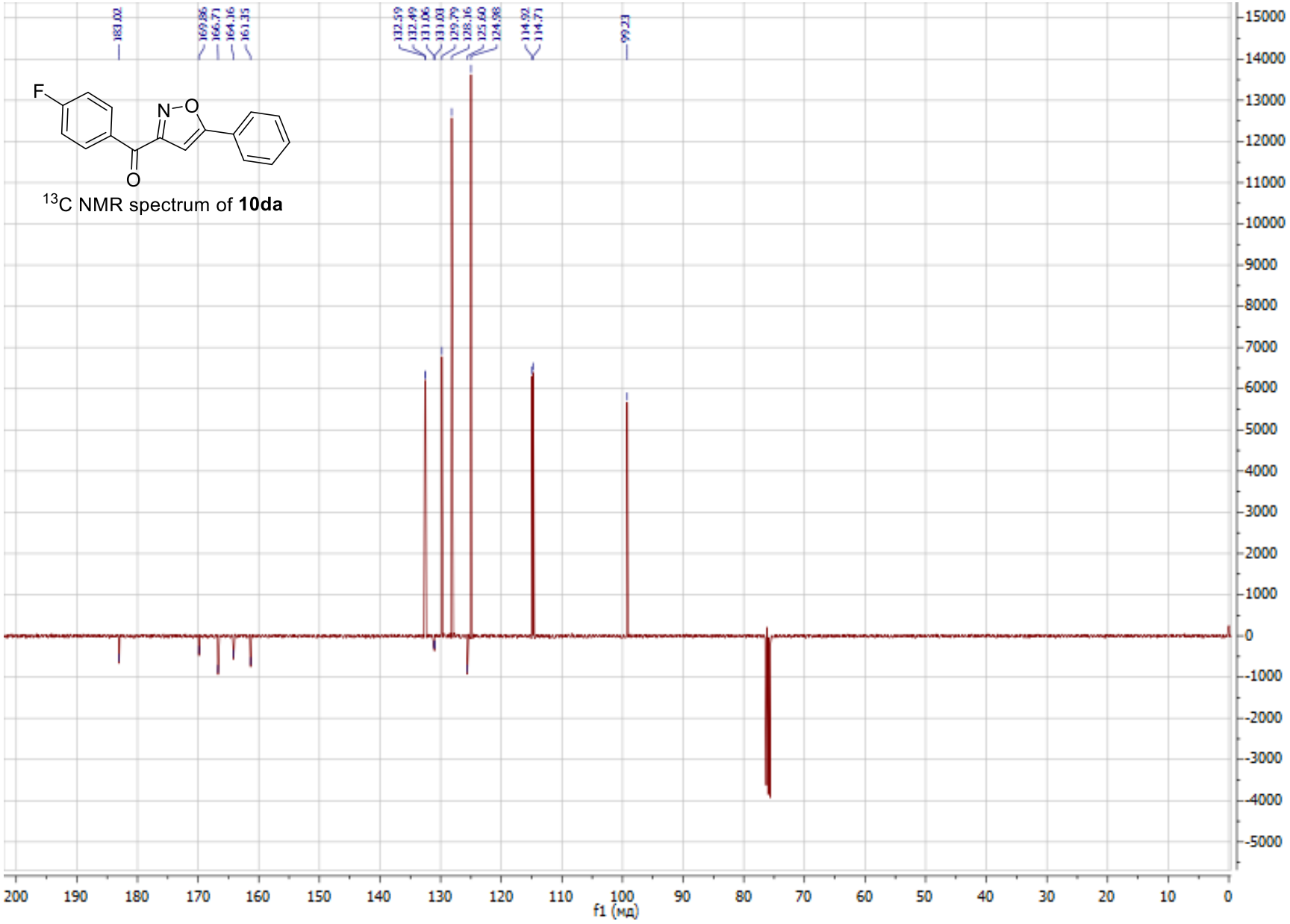


^{1}H NMR spectrum of **10ca**

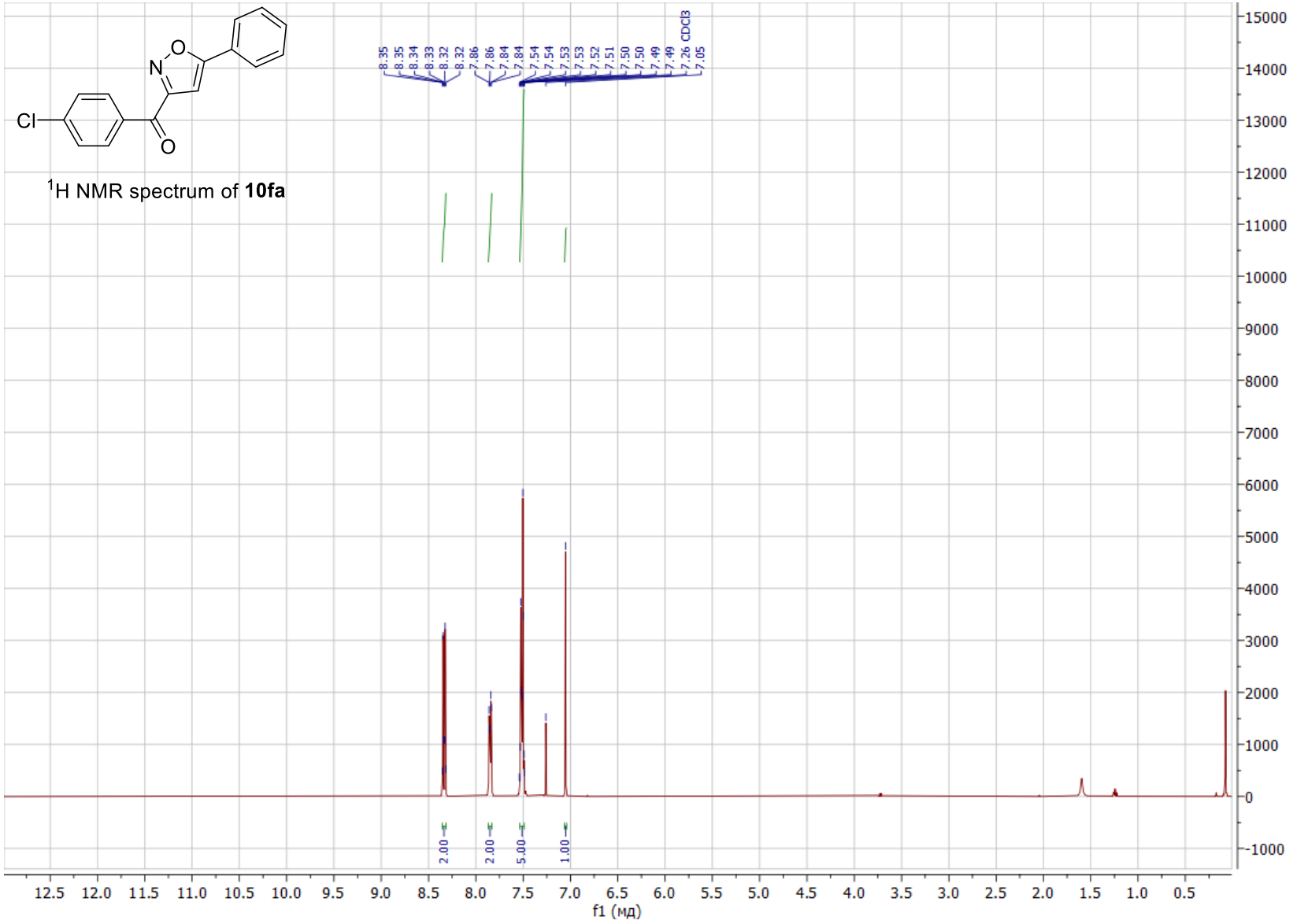


^{13}C NMR spectrum of **10ca**

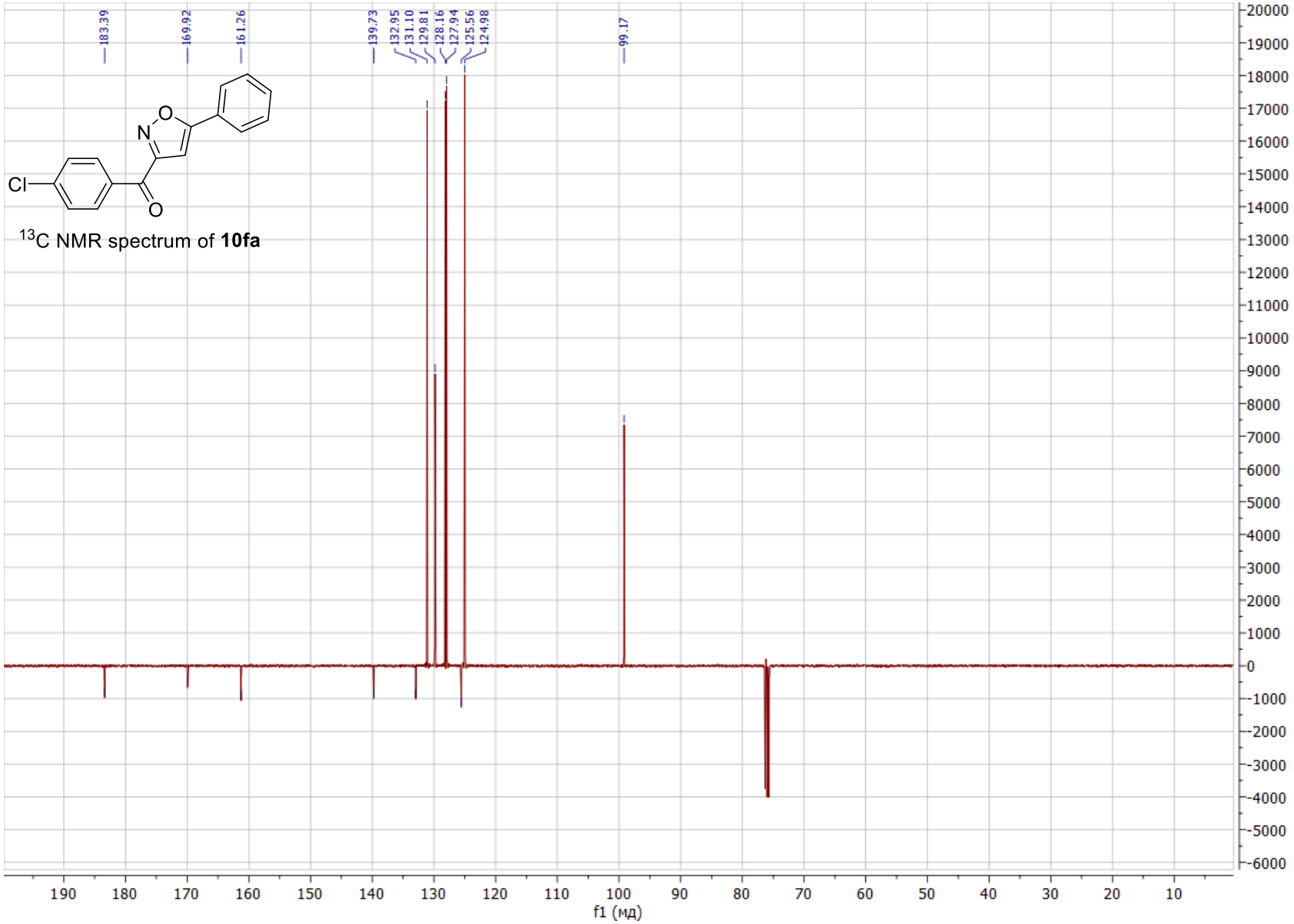

F
N
O
O
^{1}H NMR spectrum of **10da**
8.46
8.45
8.45
8.44
8.44
8.44
8.43
8.42
8.42
8.41
7.86
7.86
7.85
7.85
7.85
7.84
7.84
7.54
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.50
7.49
7.49
7.26 CDCl3
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.21
7.20
7.19
7.18
7.05
2.00
2.00
3.00
2.01
1.00
f1 (мд)



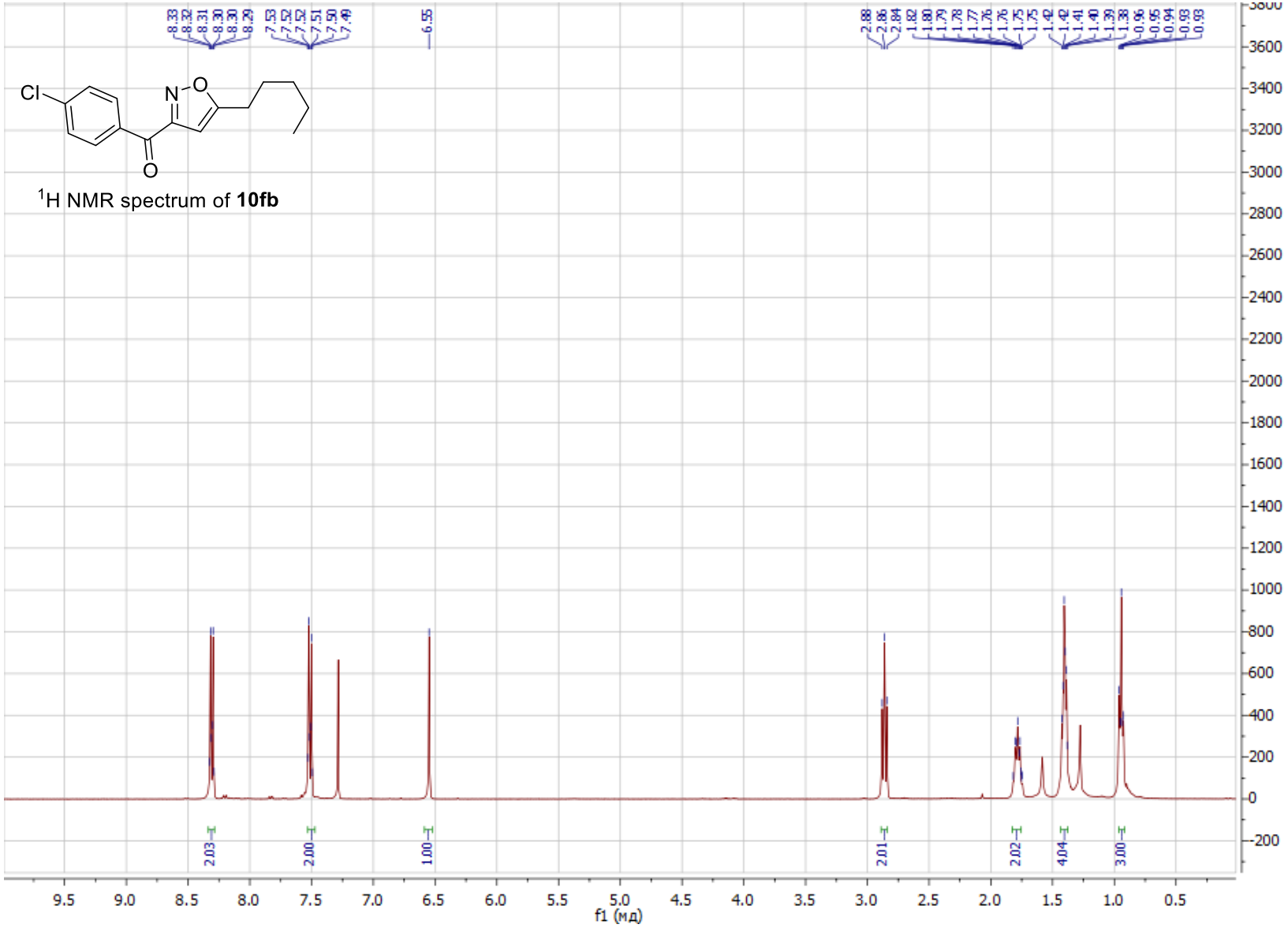
^{13}C NMR spectrum of **10da**



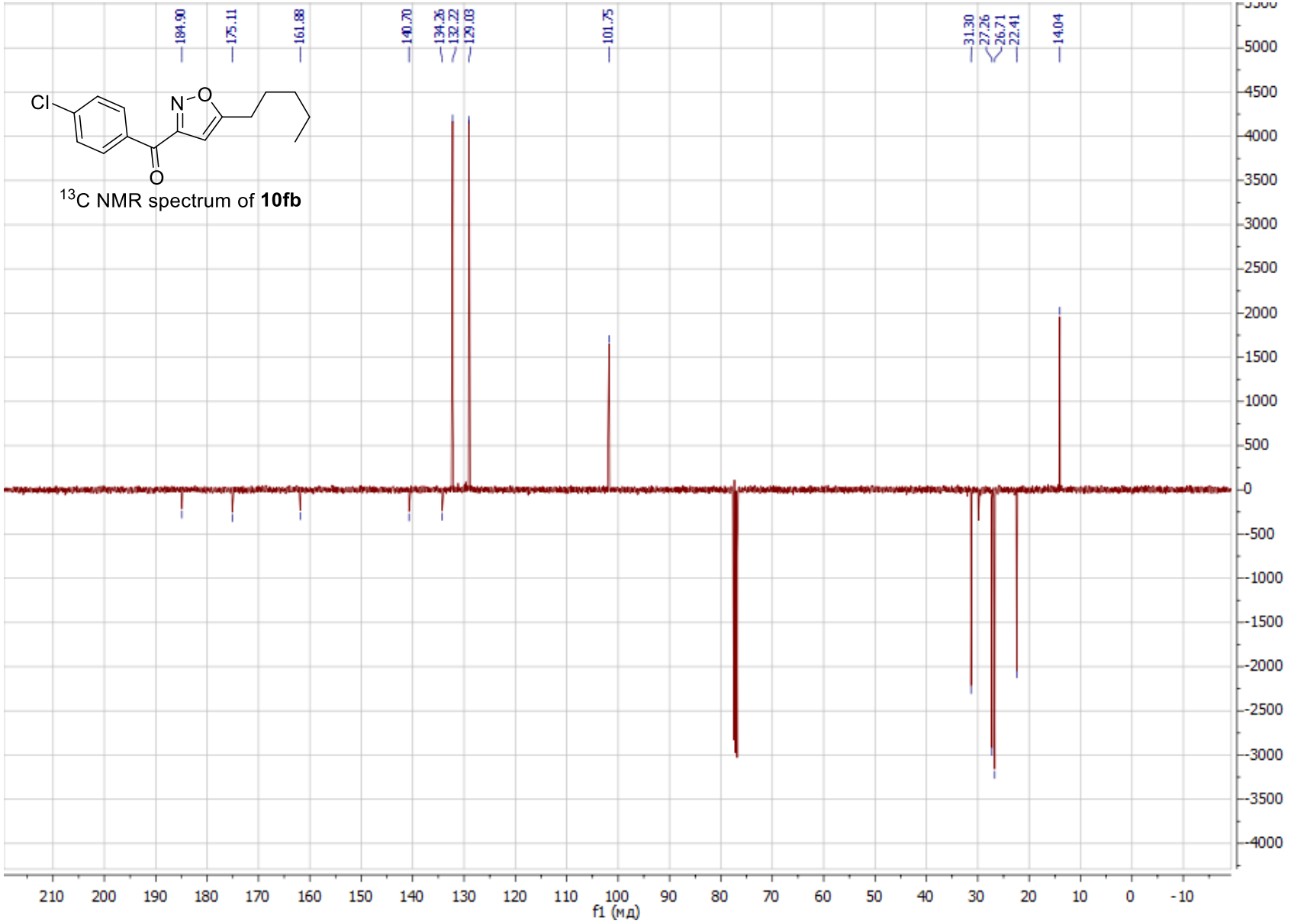
^{1}H NMR spectrum of **10fa**



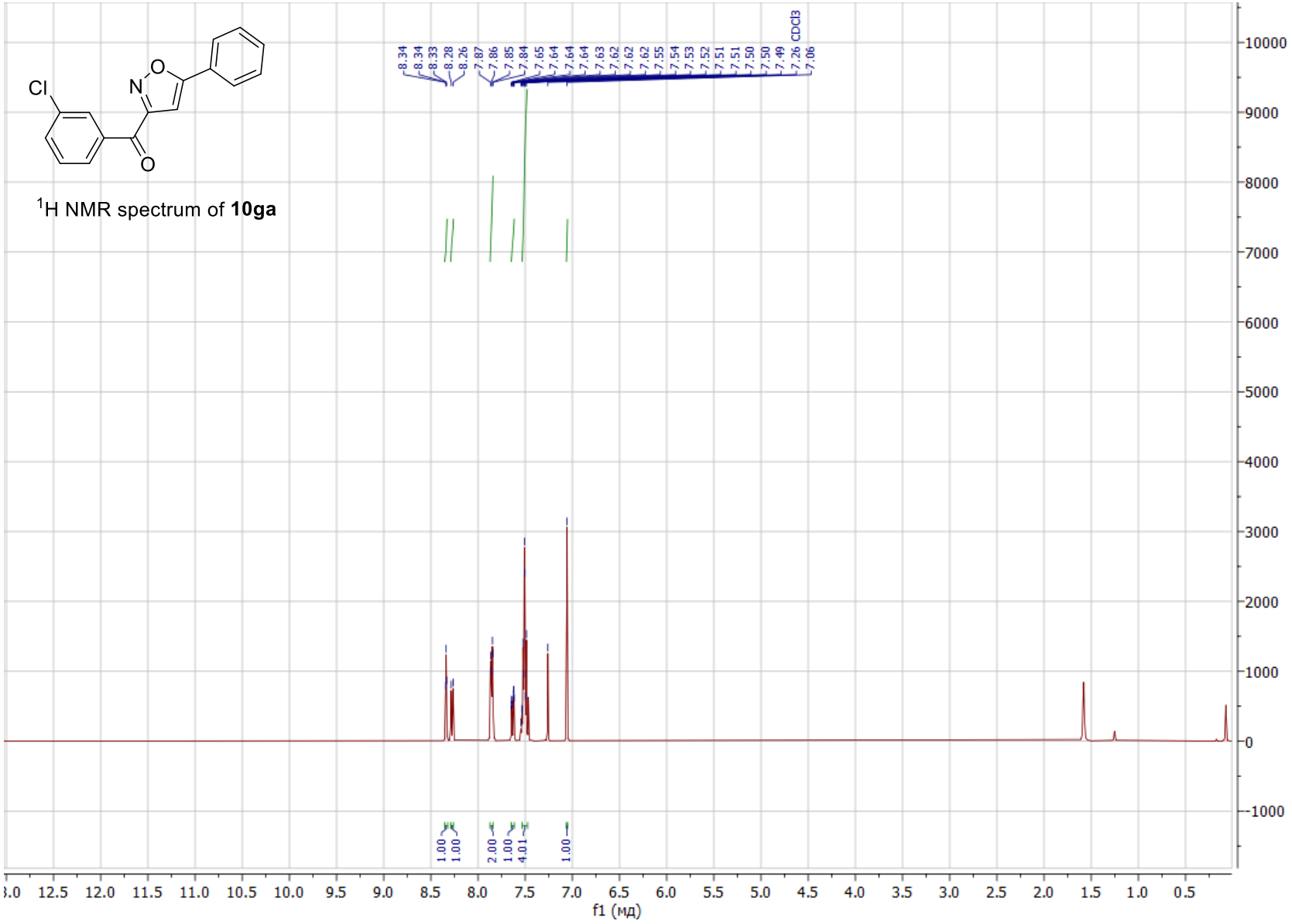
^{13}C NMR spectrum of **10fa**

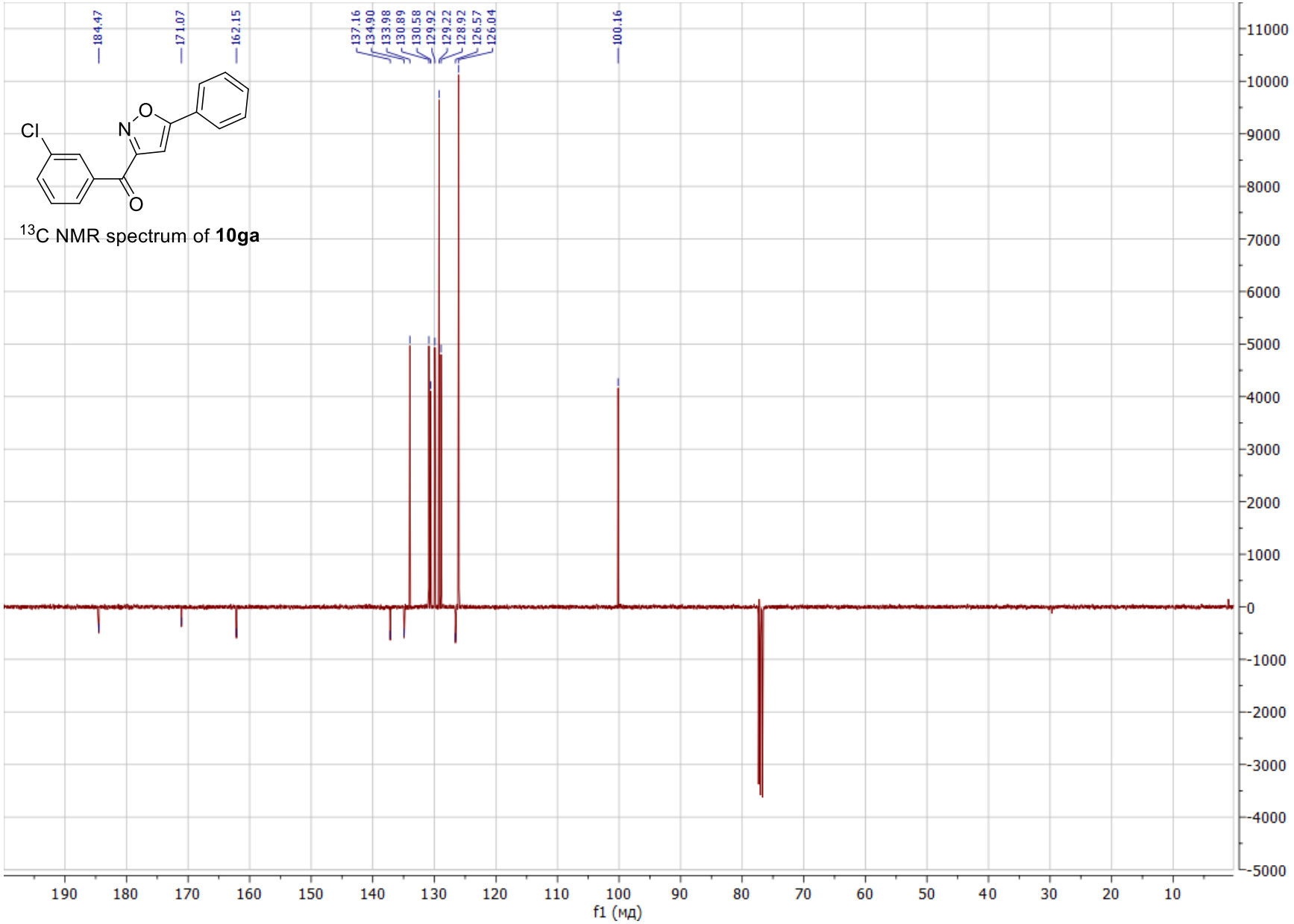


^{1}H NMR spectrum of **10fb**

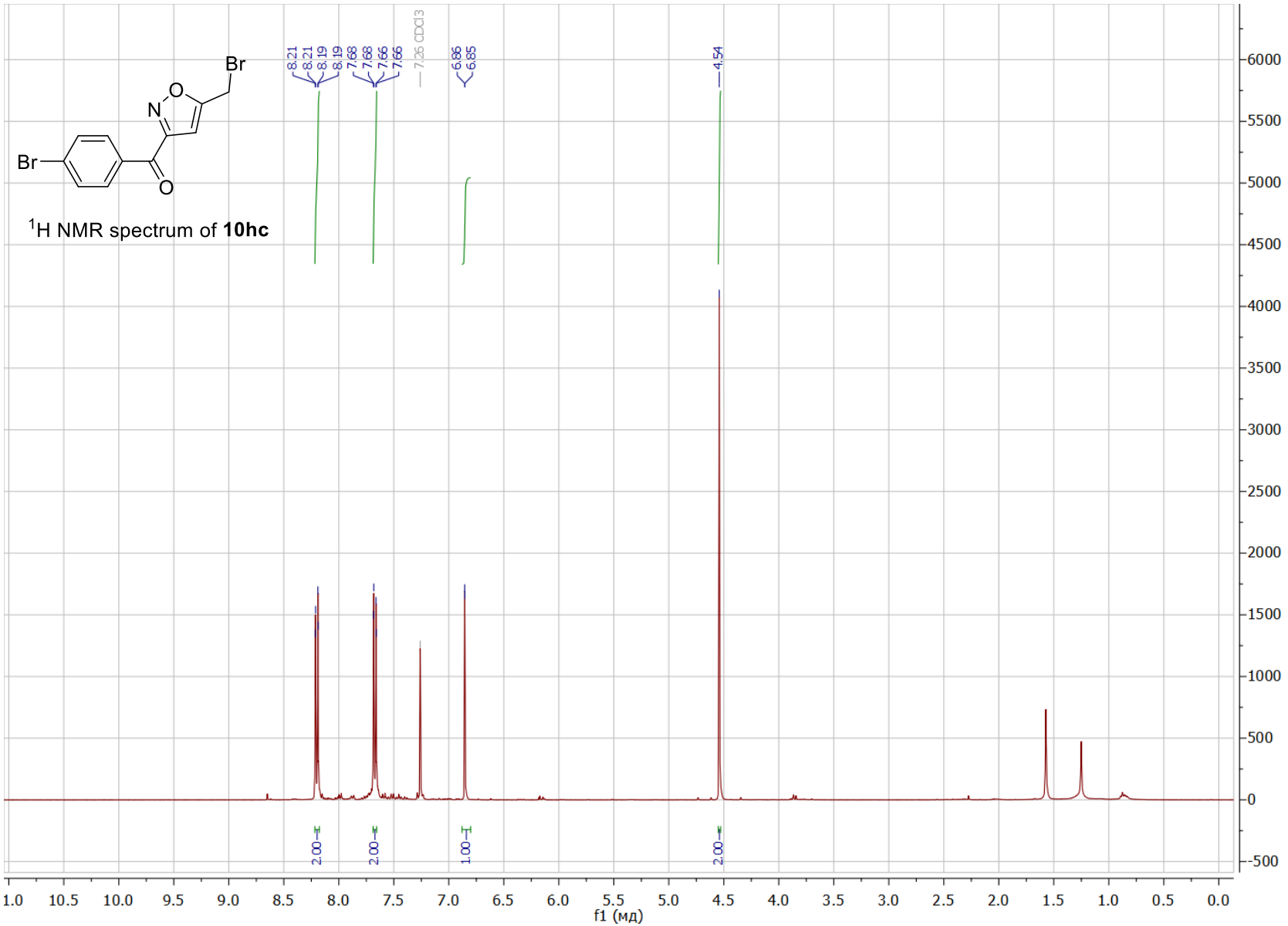


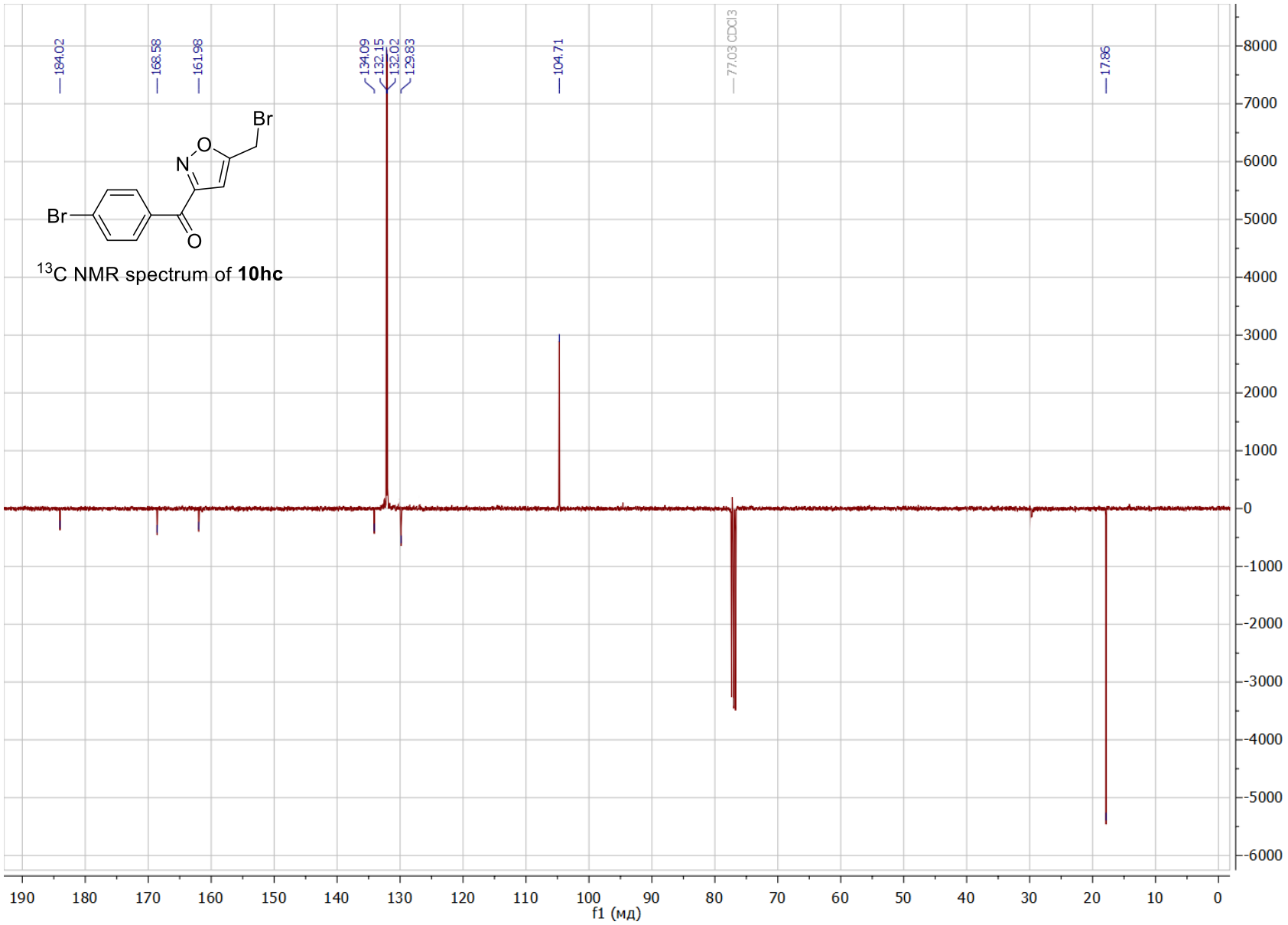
^{13}C NMR spectrum of **10fb**


Cl
N
O
O
^{1}H NMR spectrum of **10ga**
8.34
8.34
8.33
8.28
8.26
7.87
7.86
7.85
7.84
7.65
7.64
7.64
7.64
7.63
7.62
7.62
7.62
7.55
7.54
7.53
7.52
7.51
7.51
7.50
7.50
7.49
7.26 CDCl3
7.06
1.00
1.00
2.00
1.00
4.01
1.00
f1 (мд)

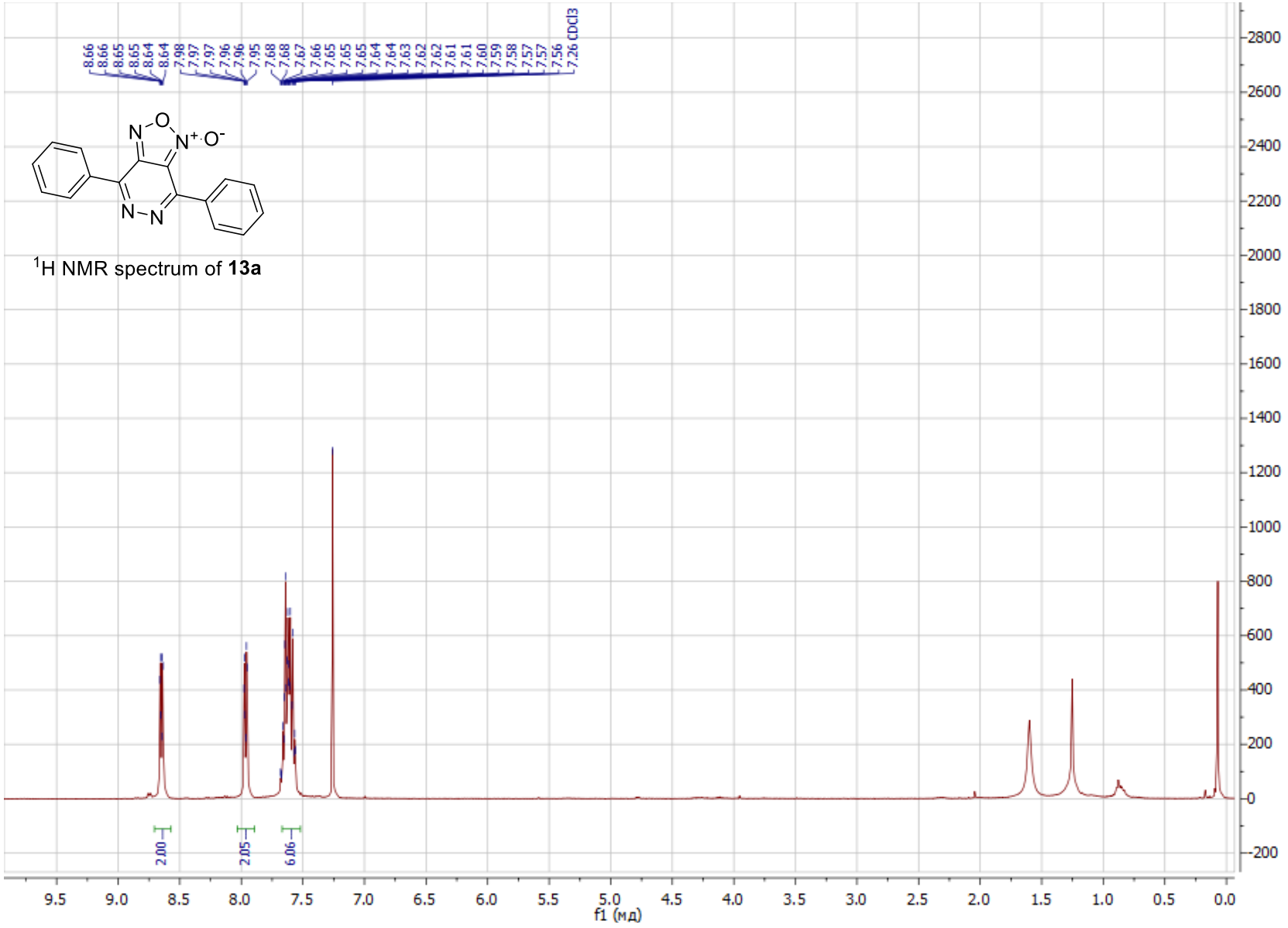


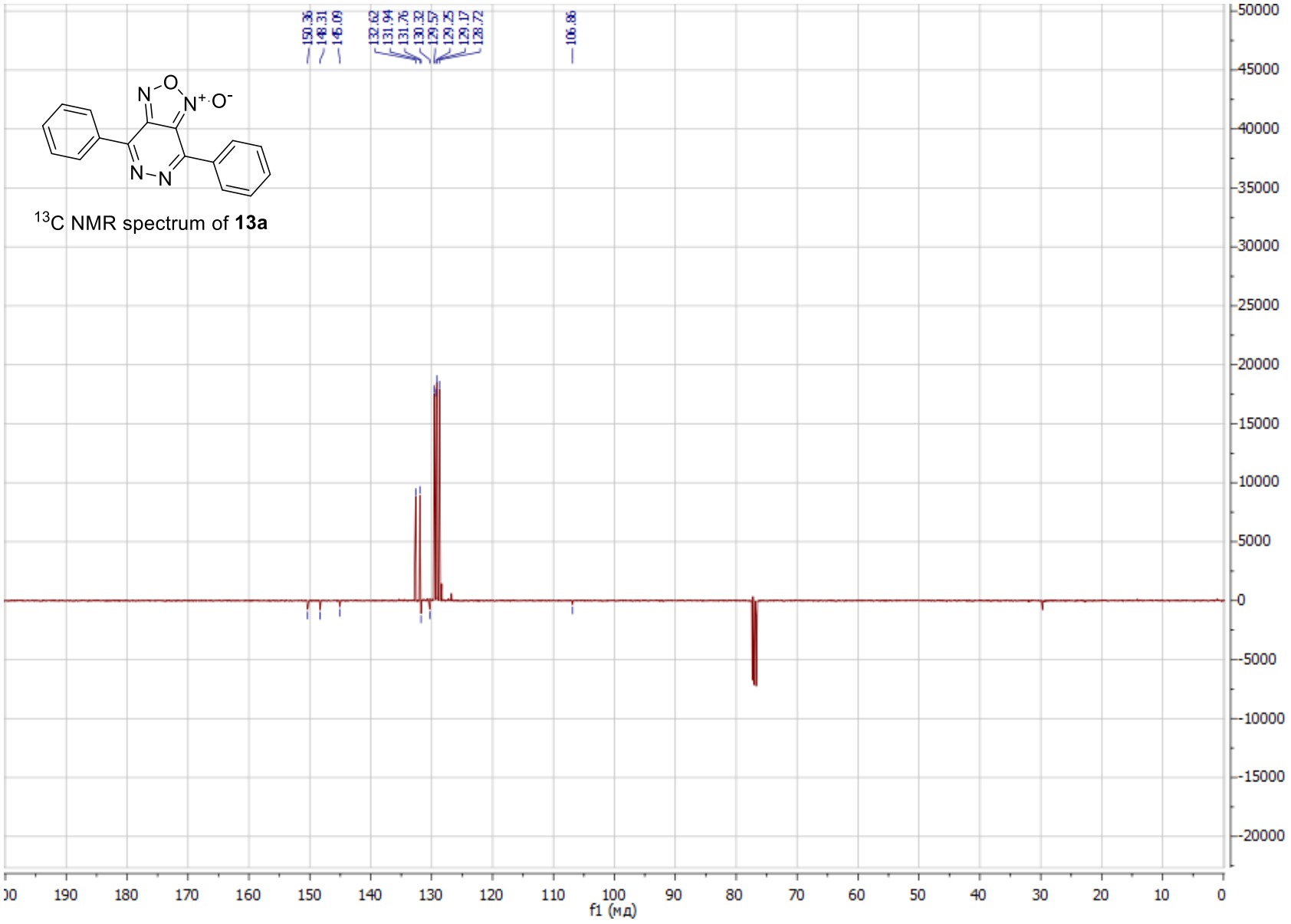
^{13}C NMR spectrum of **10ga**


Br
O
N
O
Br
^{1}H NMR spectrum of **10hc**
8.21
8.21
8.19
8.19
7.68
7.68
7.66
7.66
7.26 CDCl3
6.86
6.85
4.54
2.00
2.00
1.00
2.00
f1 (мд)


184.02
168.58
161.98
134.09
132.15
132.02
129.83
104.71
77.03 CDCl3
17.86
Br
O
N
O
Br
^{13}C NMR spectrum of **10hc**
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000
-5000
-6000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (мд)

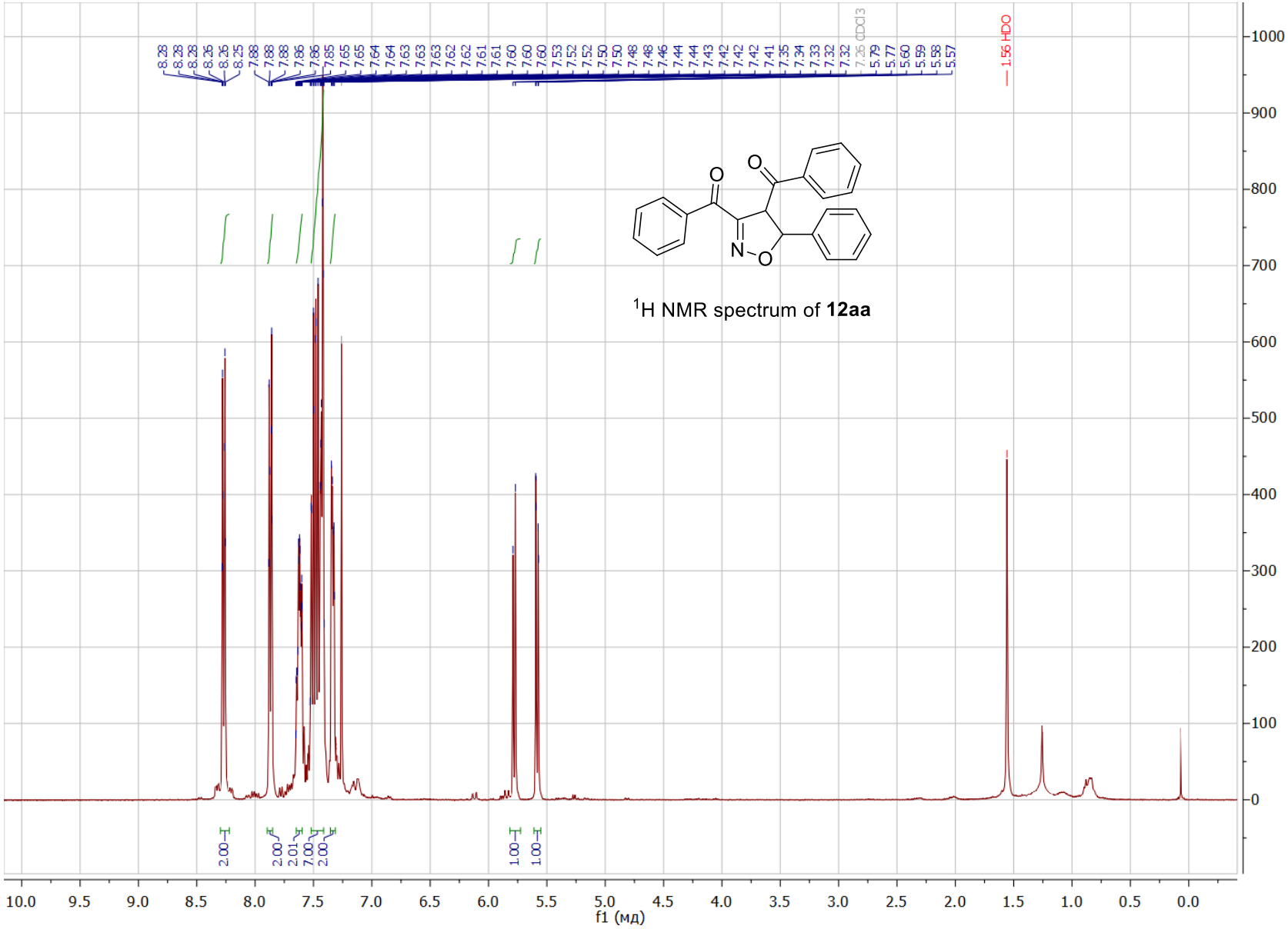
^{1}H and ^{13}C NMR spectral charts for 4,7-diphenyl-[1,2,5]oxadiazolo[3,4-d]pyridazine 1-oxide (13a):

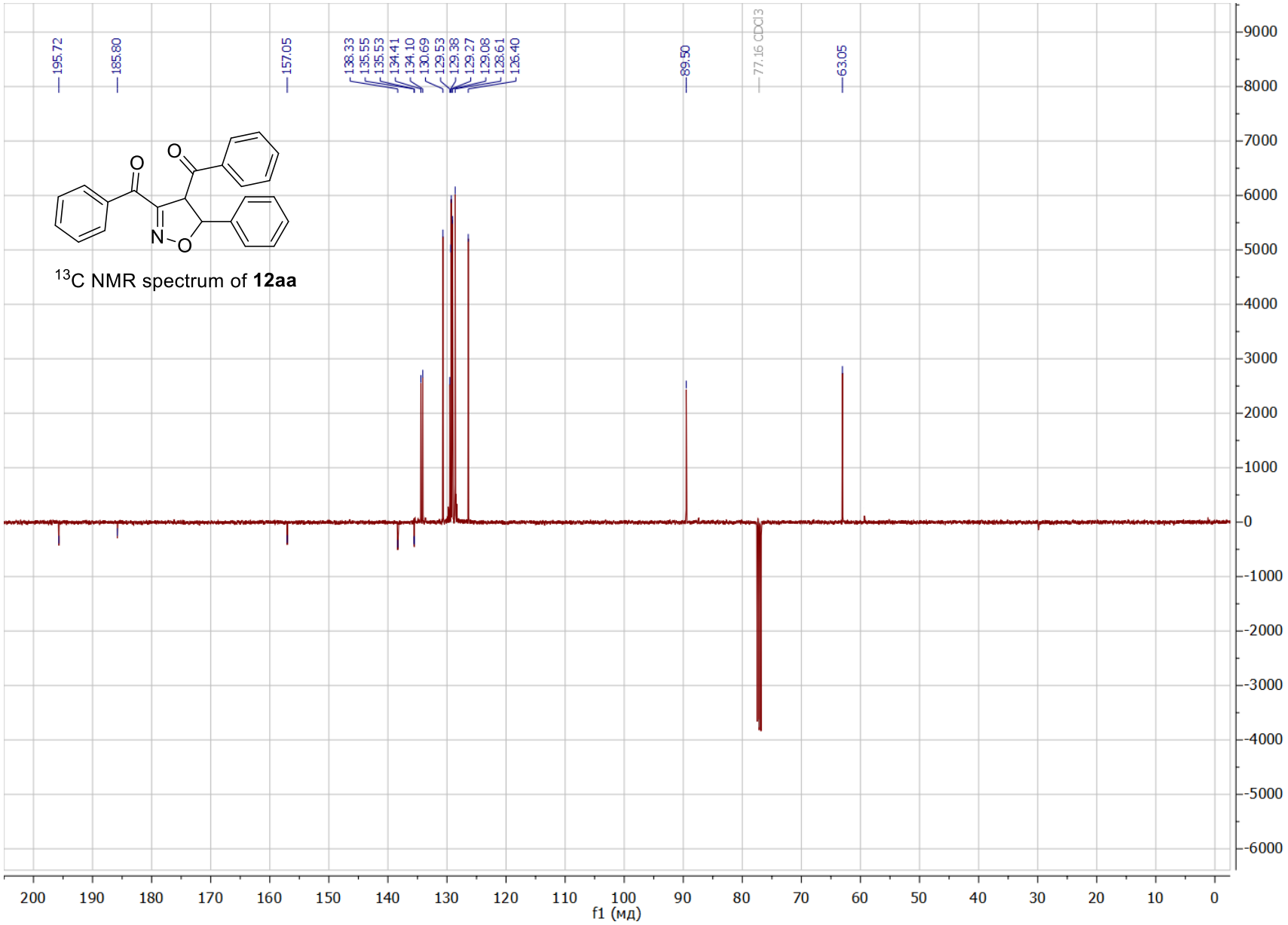




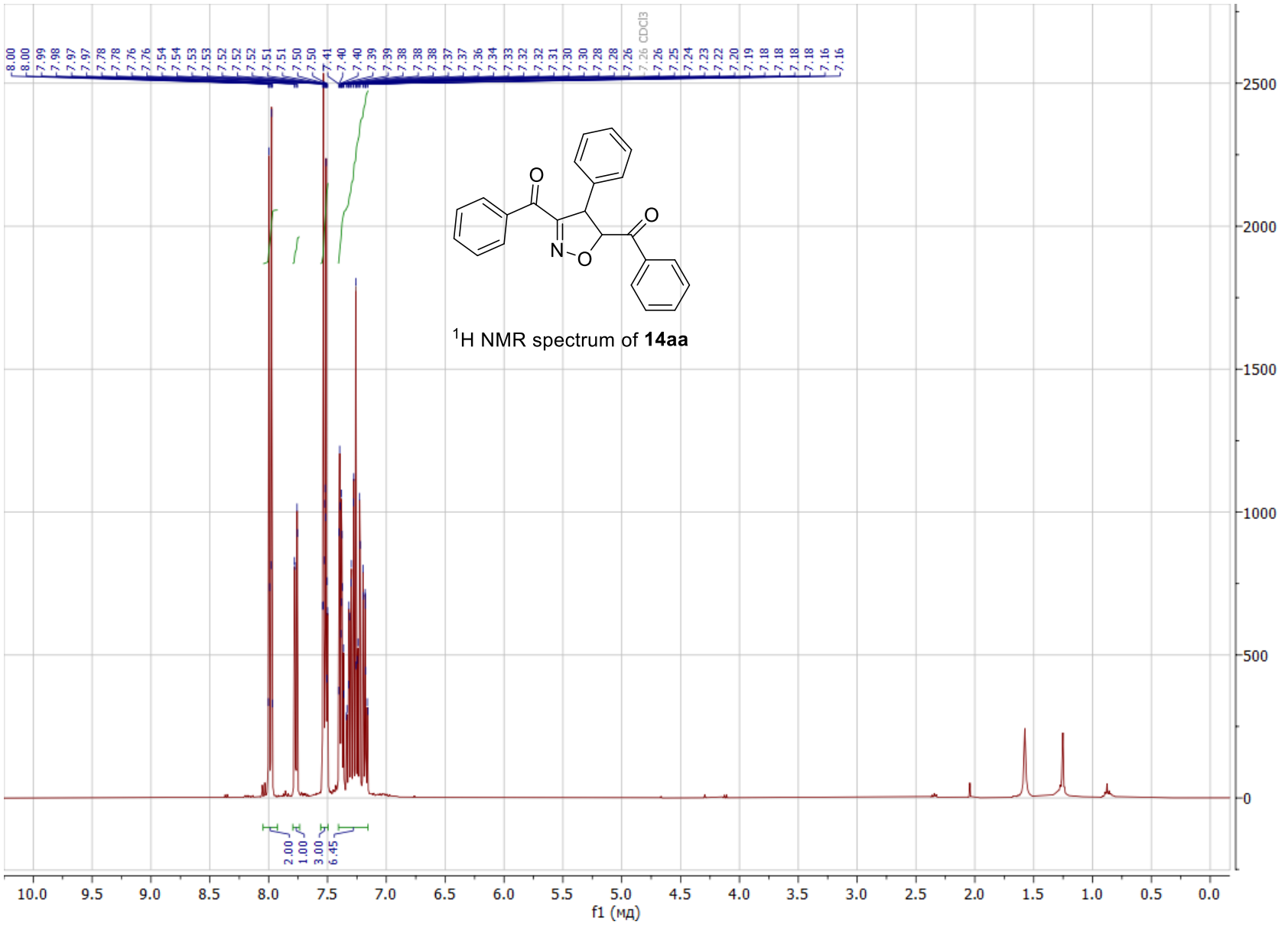
^{13}C NMR spectrum of **13a**

^{1}H and ^{13}C NMR spectral charts for cycloadducts with chalcones 12aa, 14aa, 15fb:

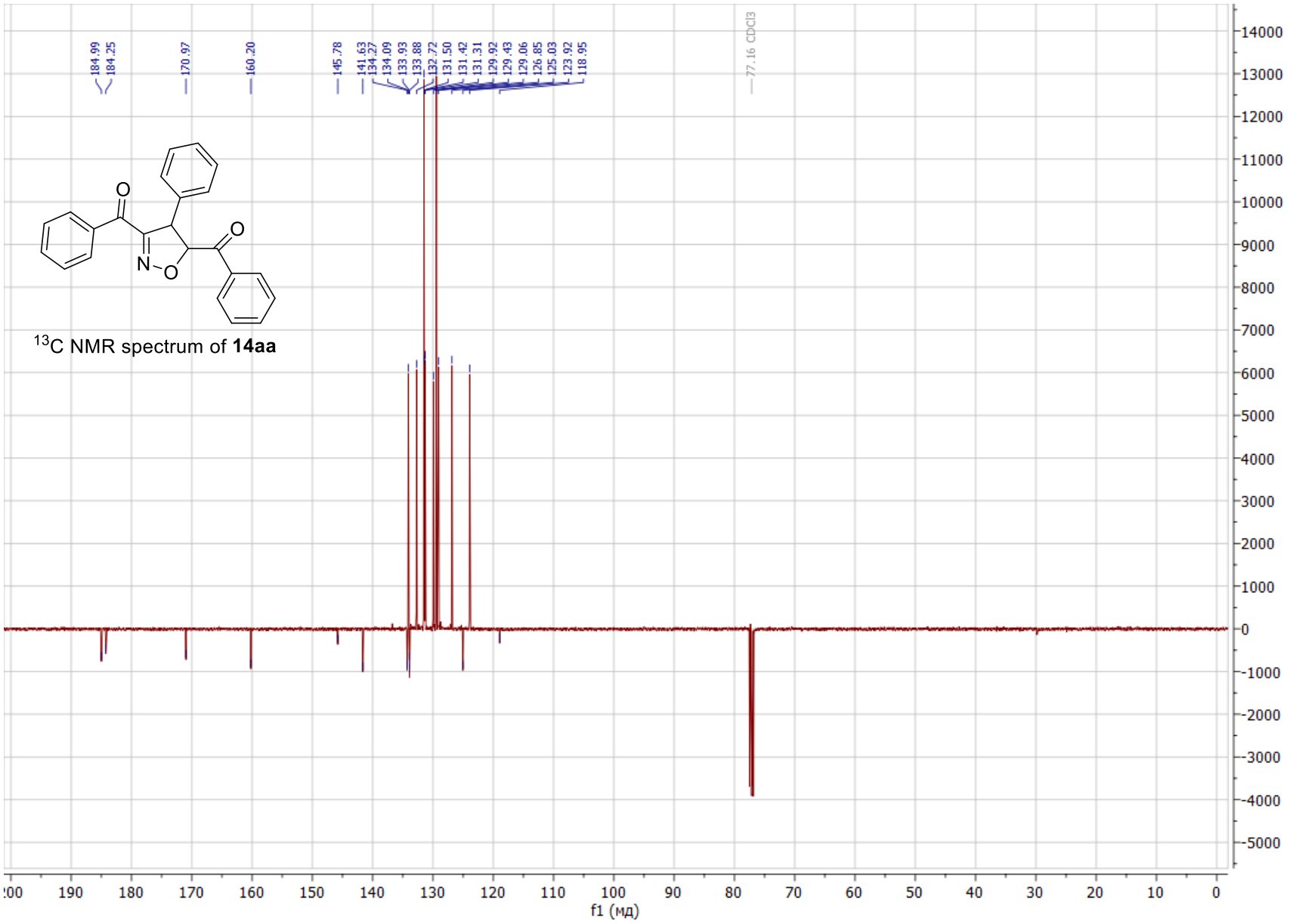




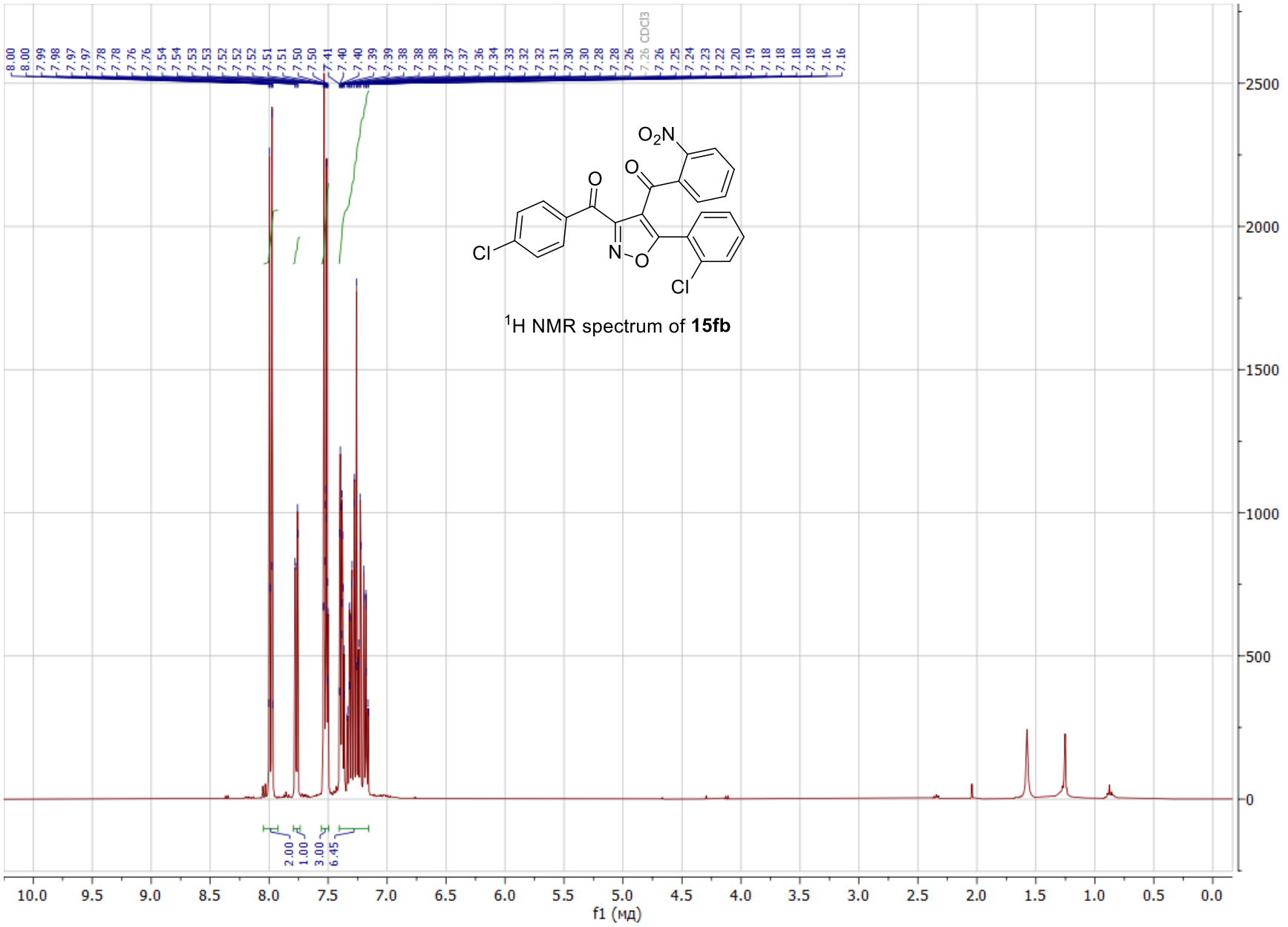
^{13}C NMR spectrum of **12aa**



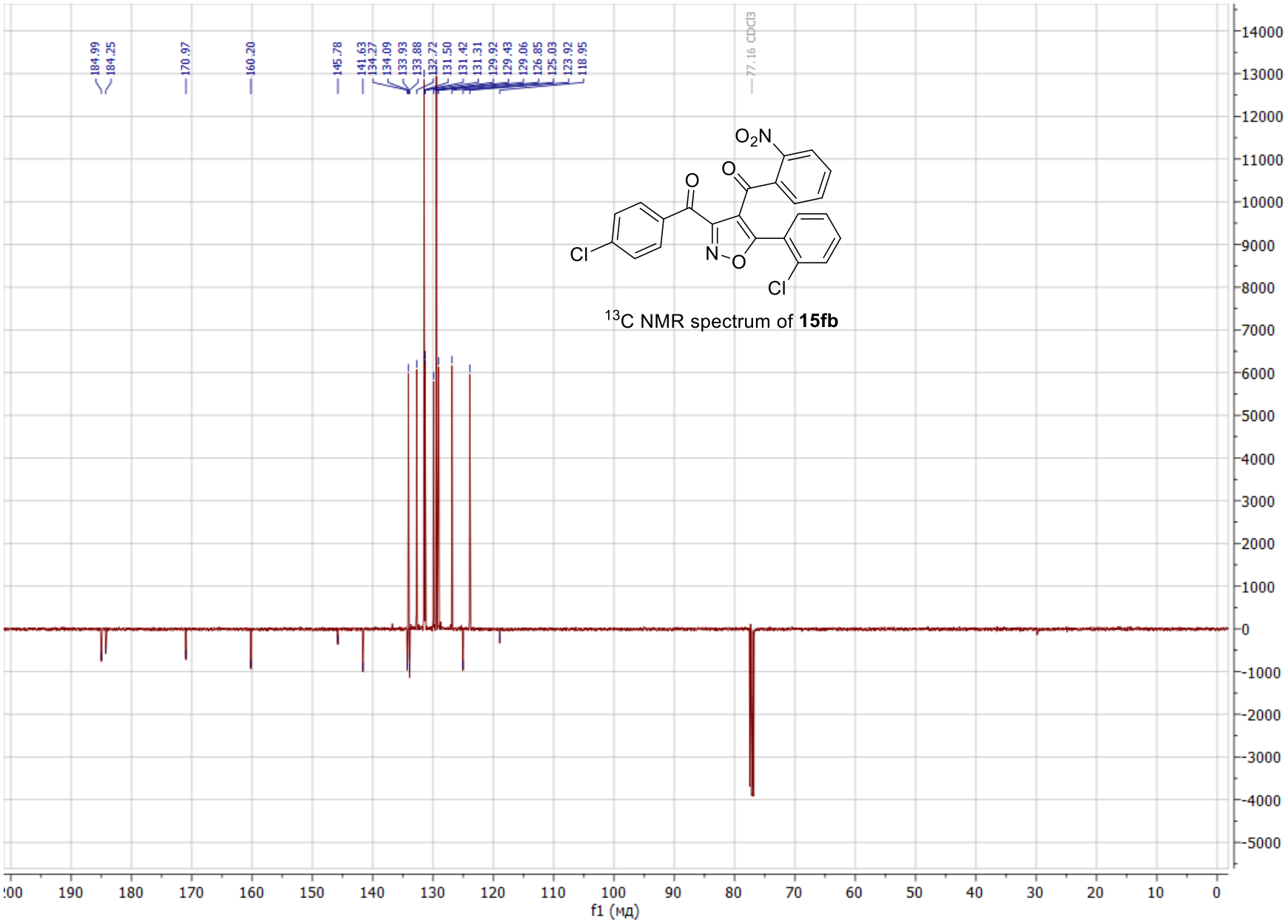
^{1}H NMR spectrum of **14aa**



^{13}C NMR spectrum of **14aa**

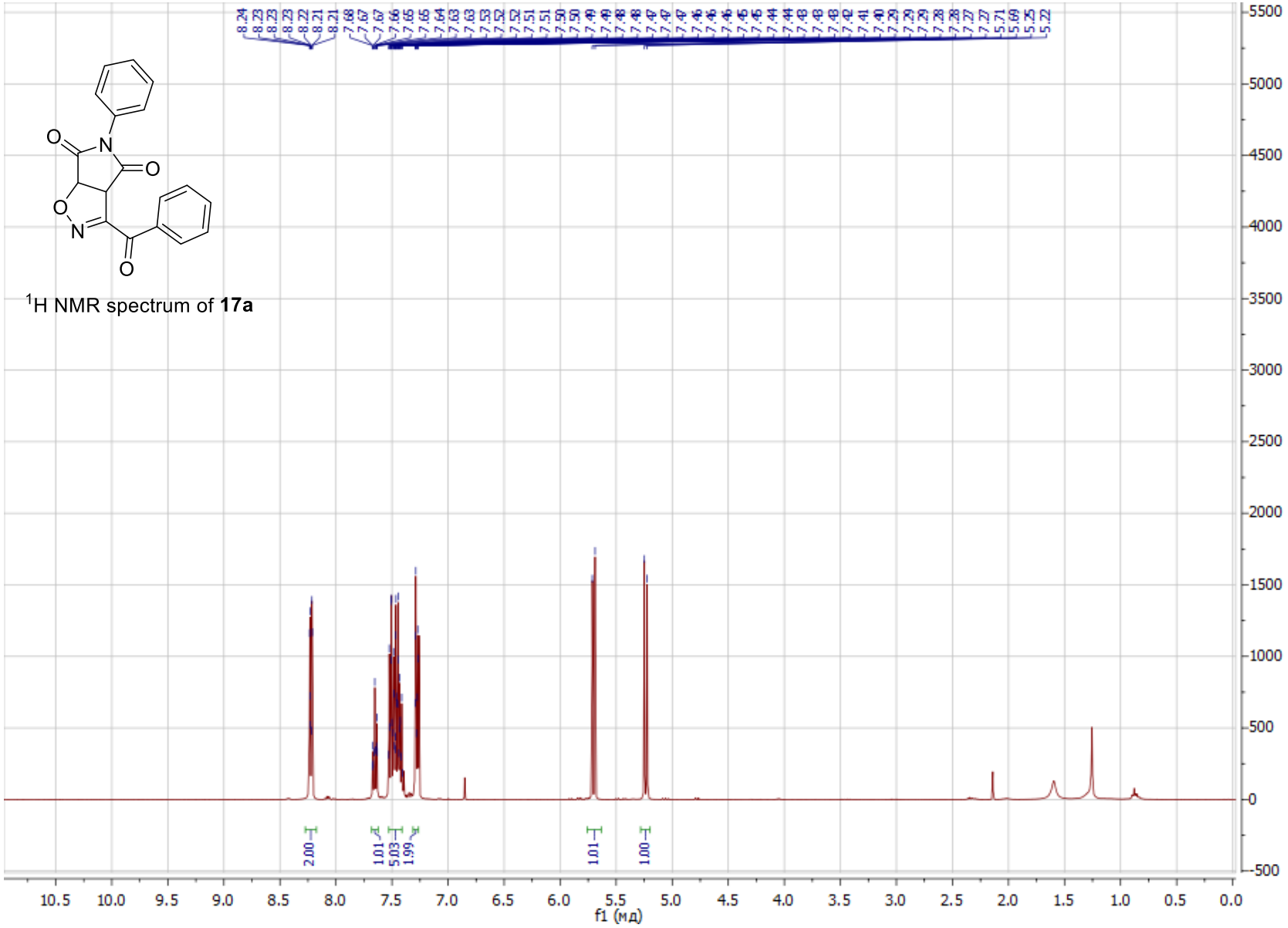


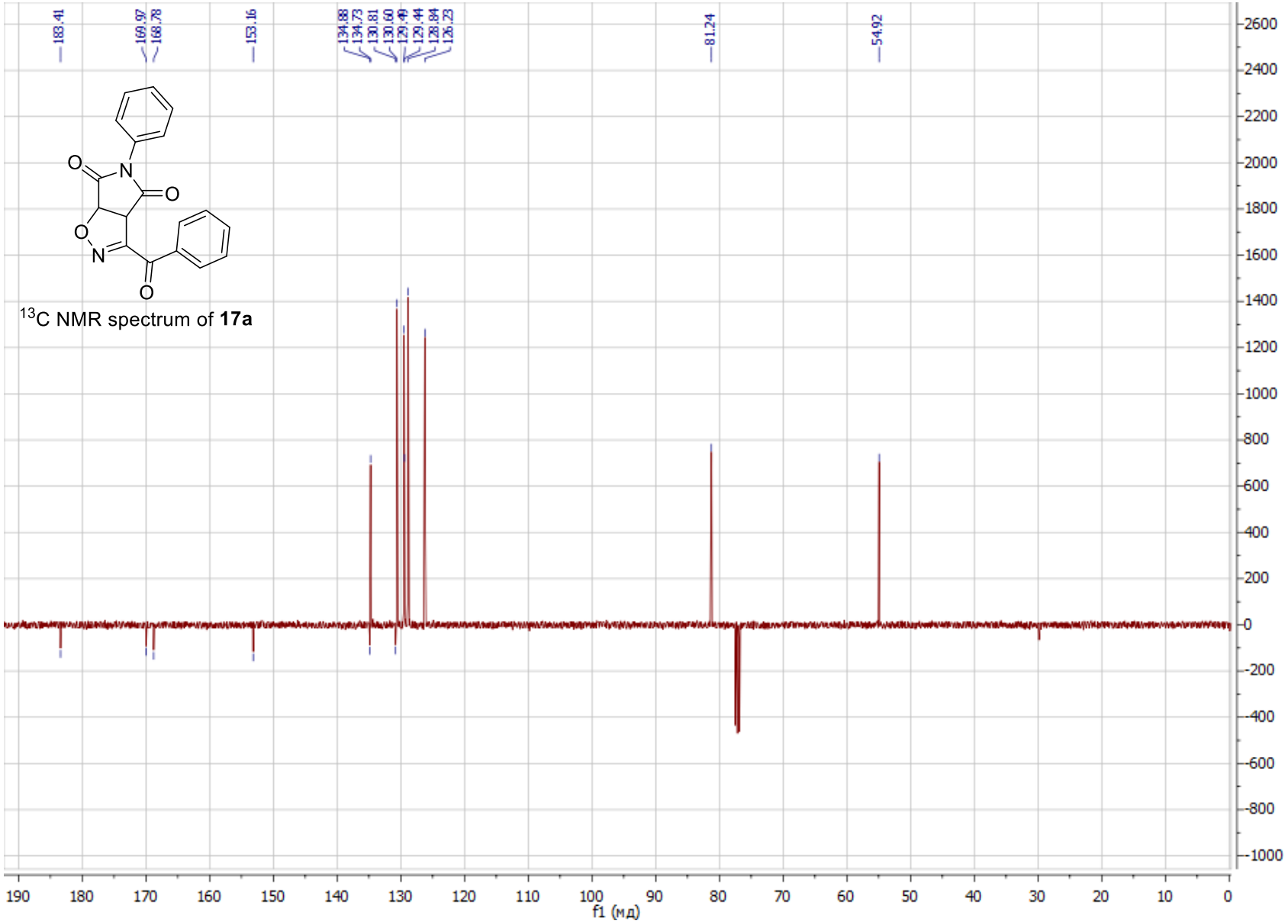
^{1}H NMR spectrum of **15fb**



^{13}C NMR spectrum of **15fb**

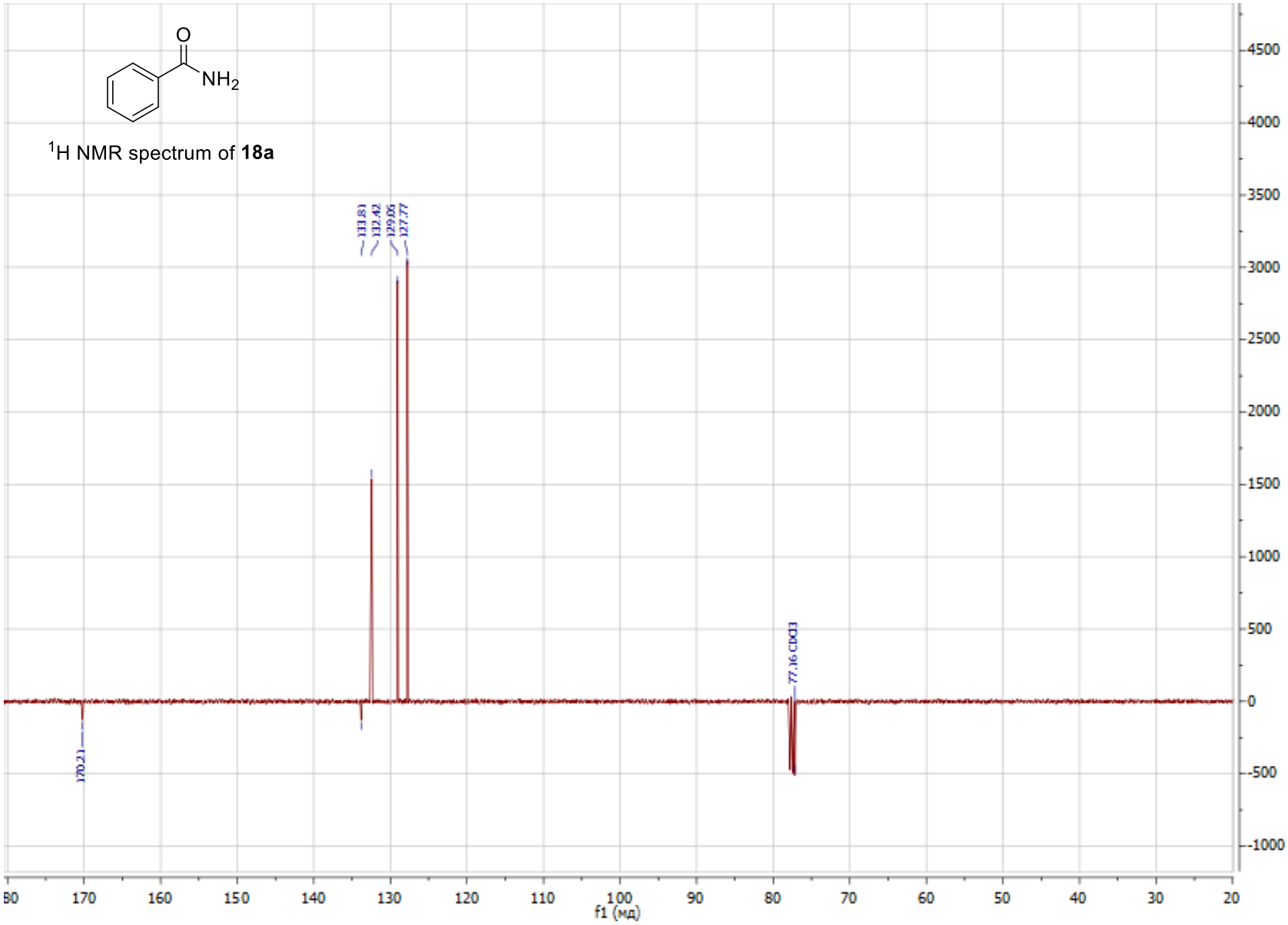
^{1}H and ^{13}C NMR spectral charts for 3-Benzoyl-5-phenyl-3a,6a-dihydro-4H-pyrrolo[3,4-d]isoxazole-4,6(5H)-dione (17a):

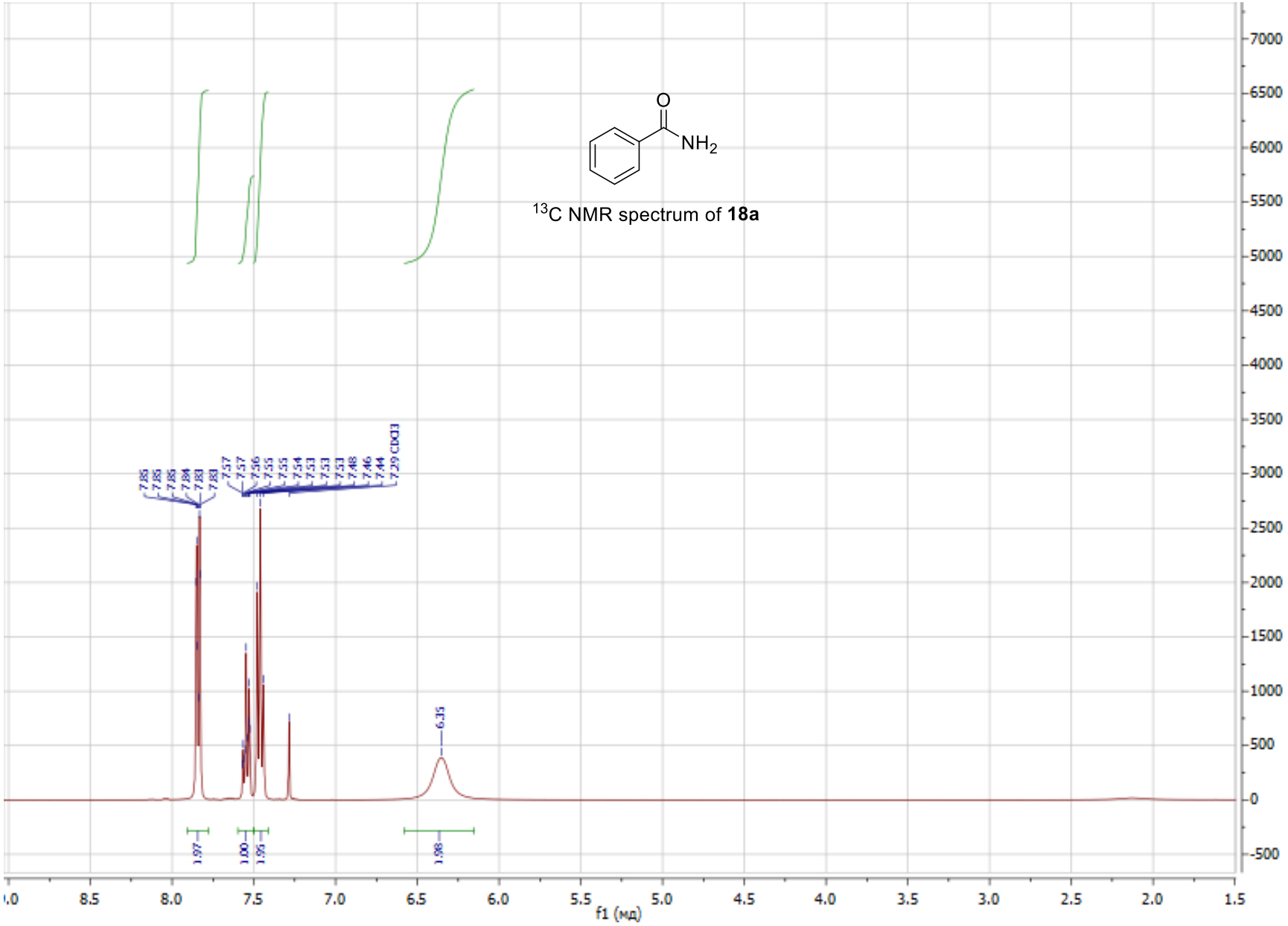




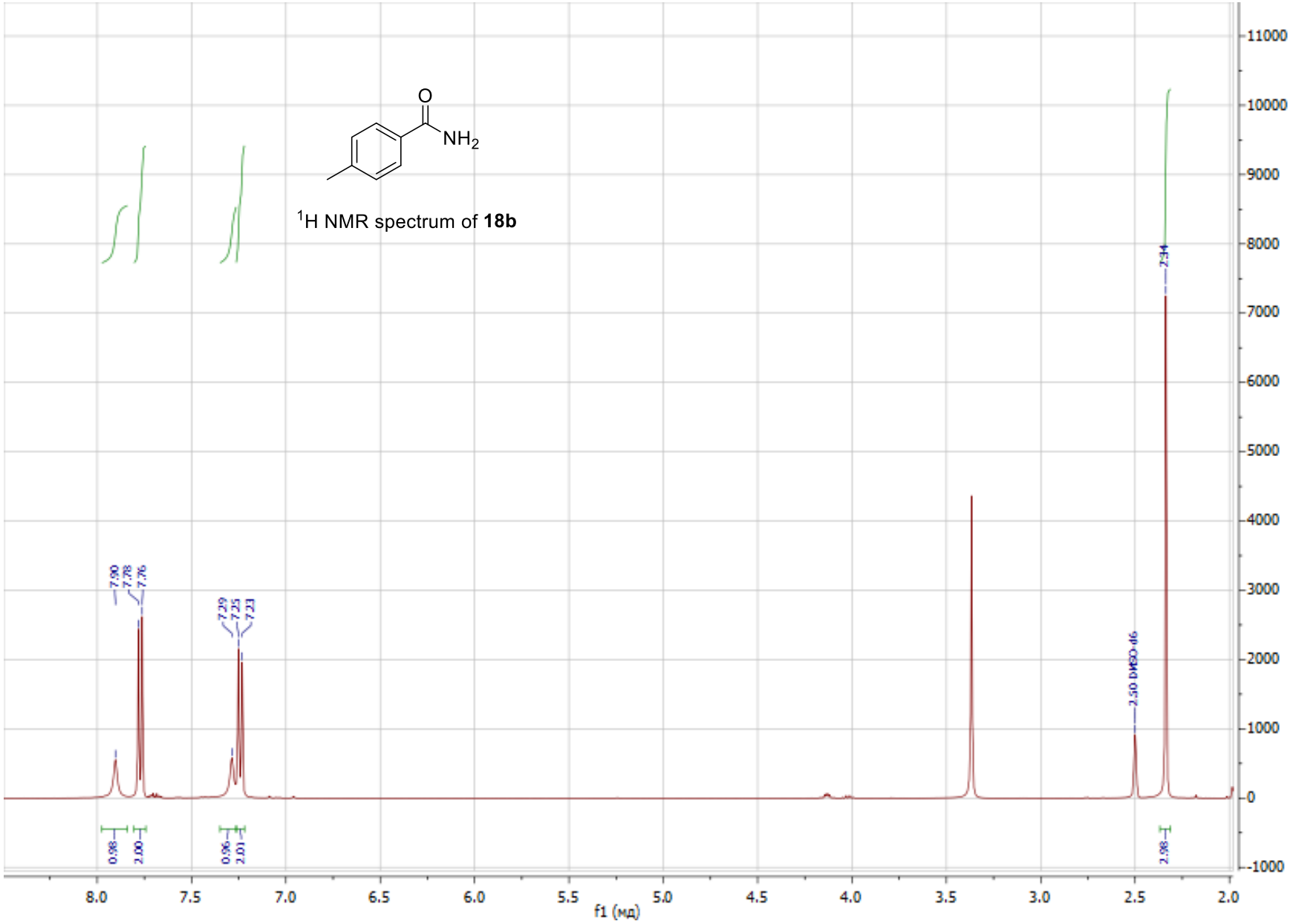
^{13}C NMR spectrum of **17a**

^{1}H and ^{13}C NMR spectral charts for benzamides (18)

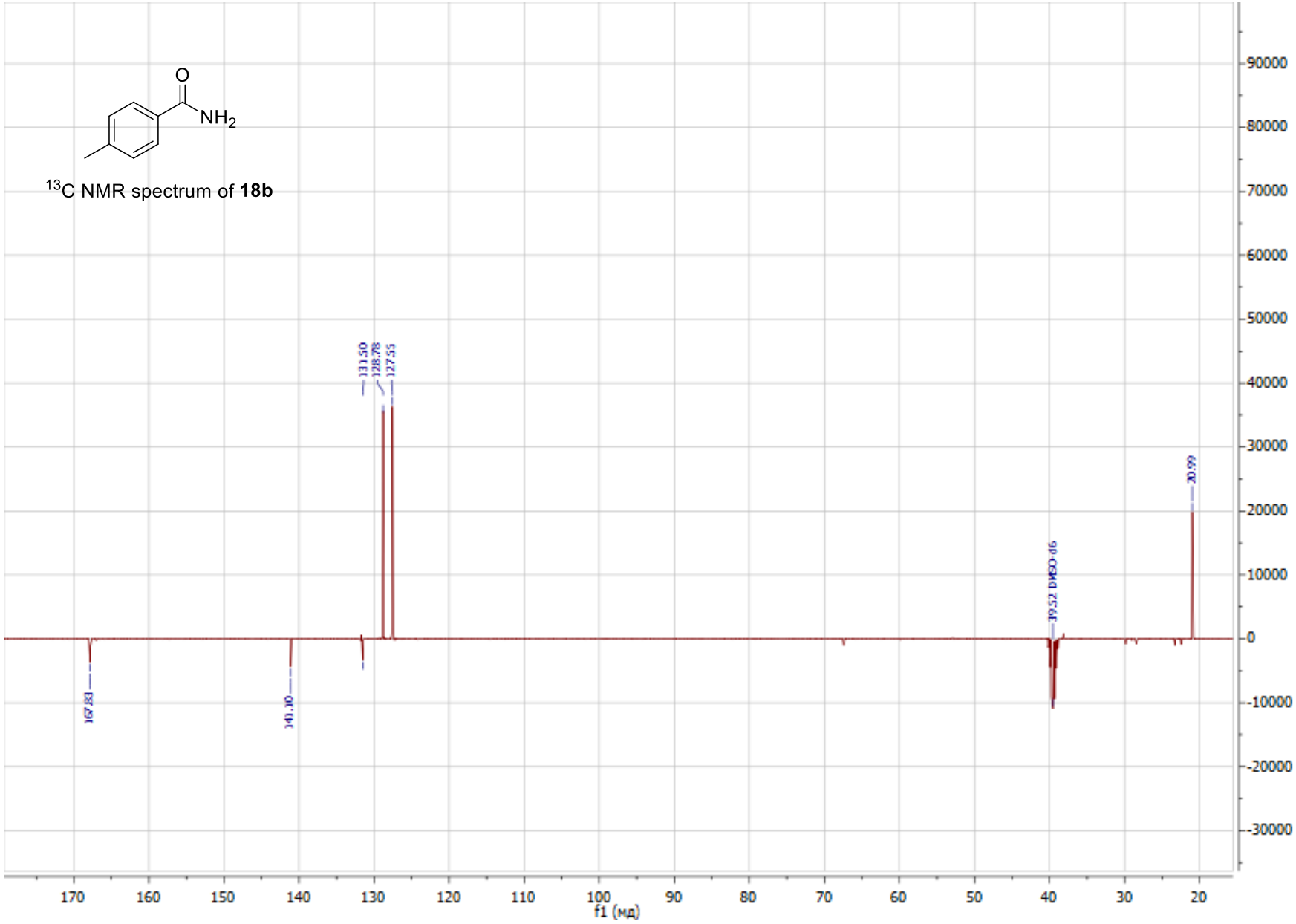




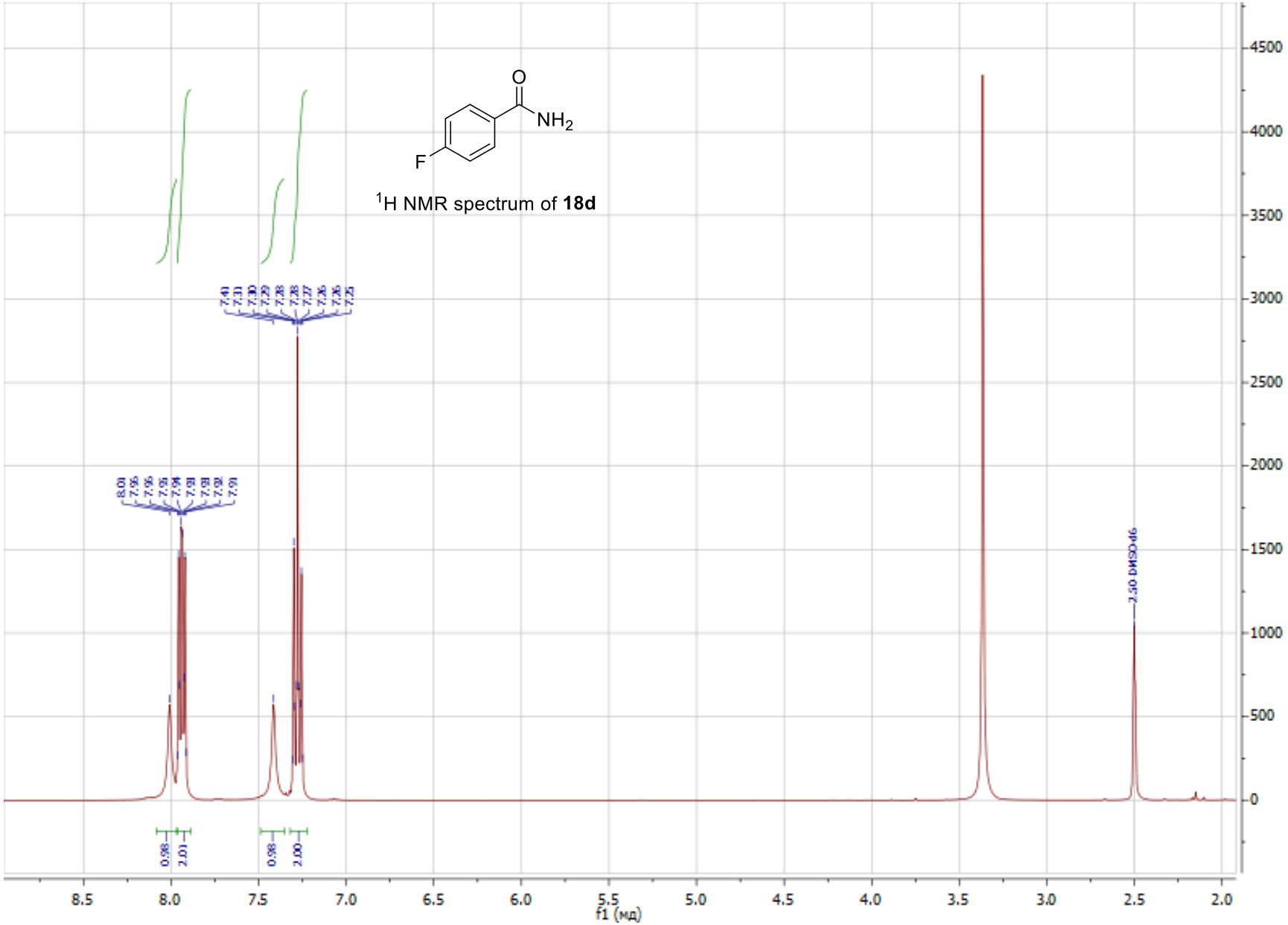
^{13}C NMR spectrum of **18a**

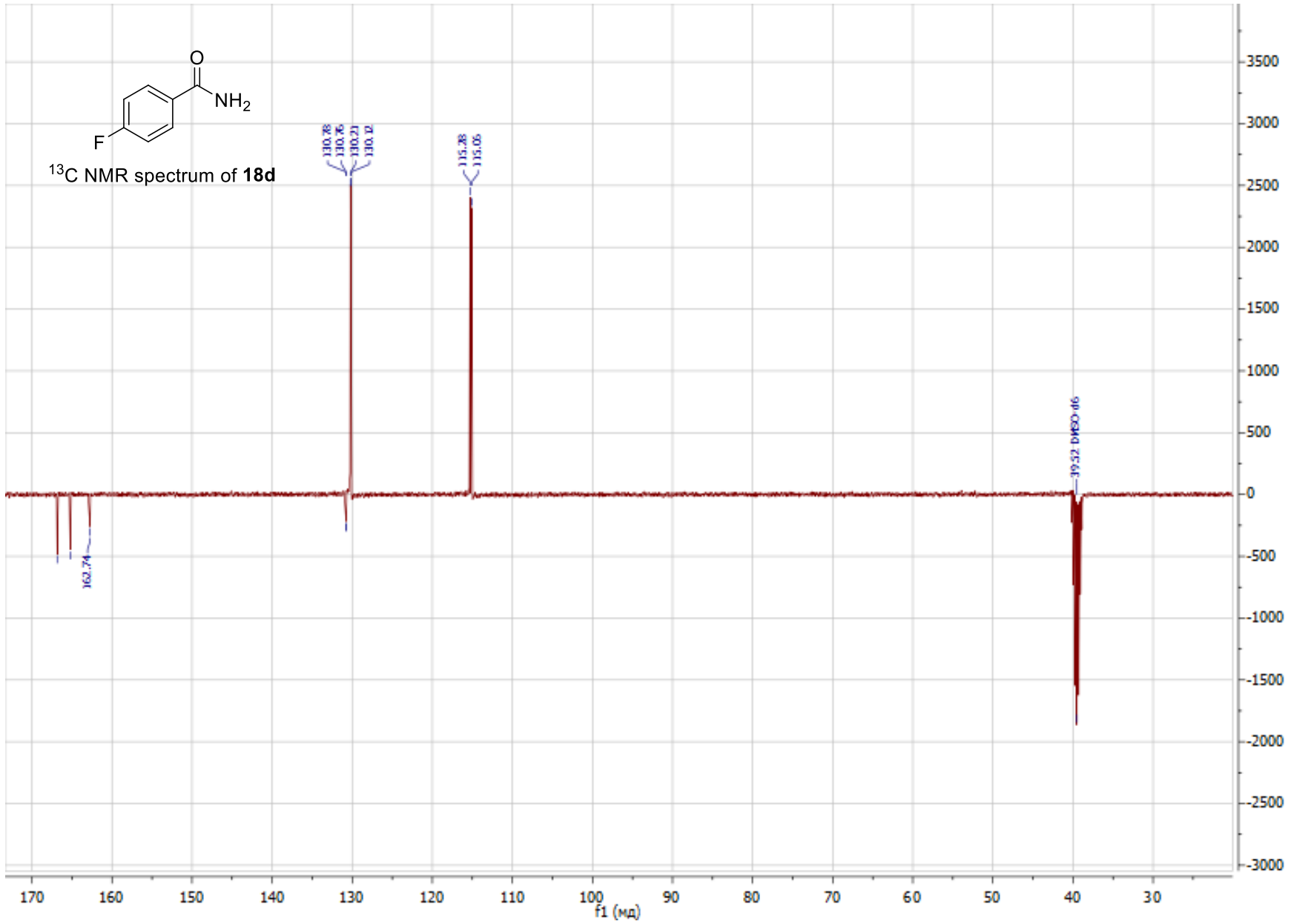


^{1}H NMR spectrum of **18b**

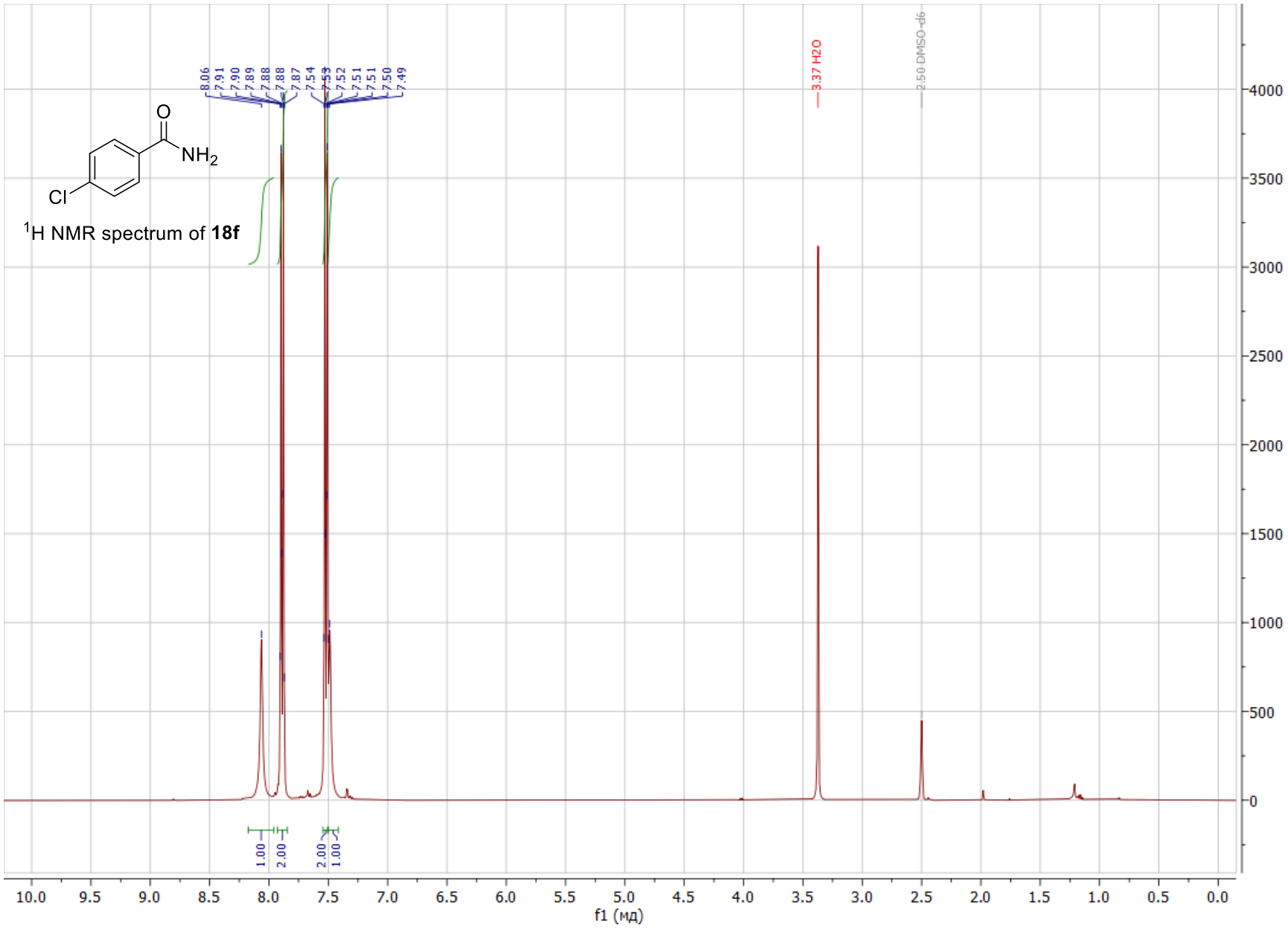


^{13}C NMR spectrum of **18b**

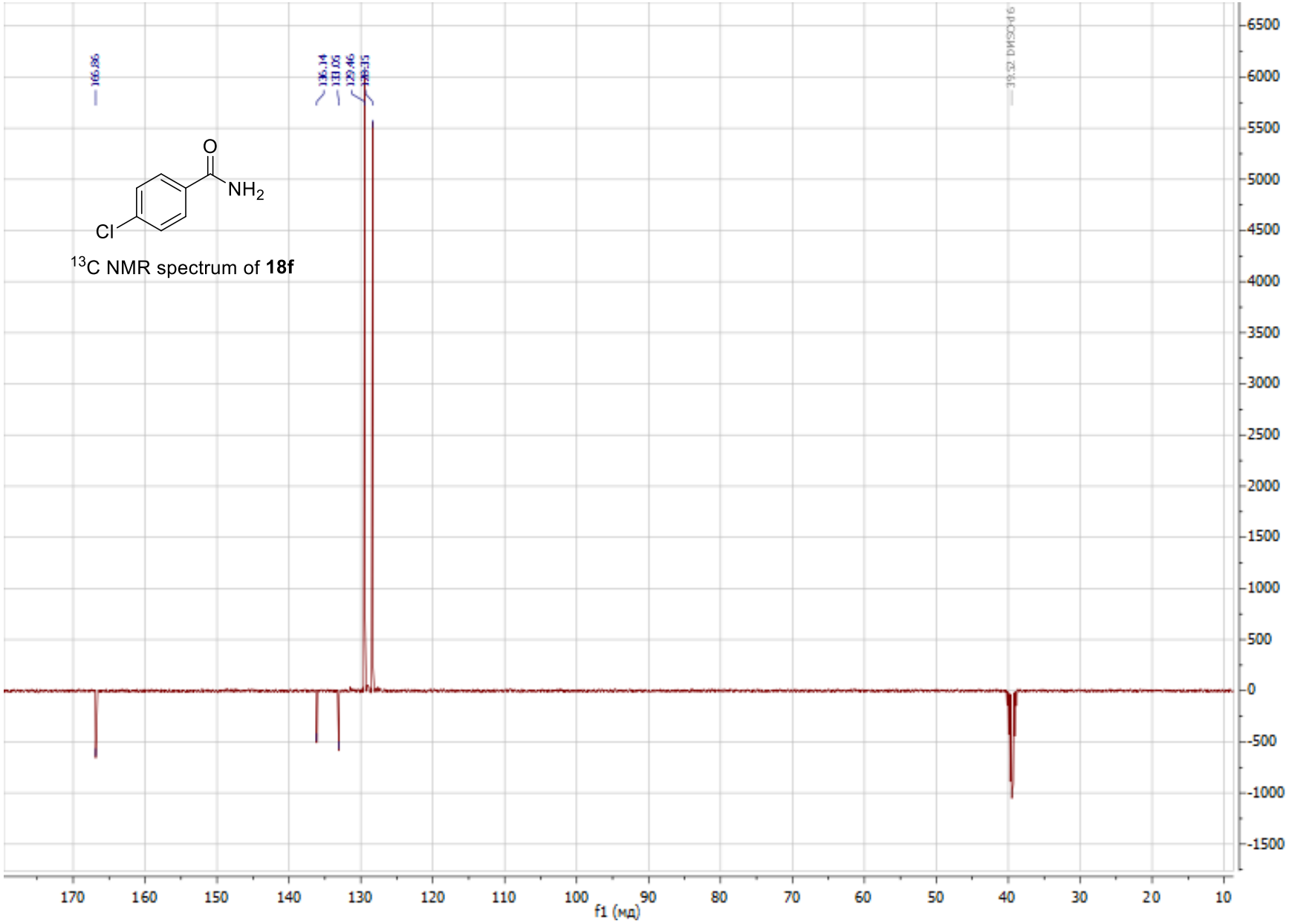

O
NH2
F
1H NMR spectrum of 18d
8.01
7.95
7.95
7.95
7.94
7.93
7.93
7.92
7.91
7.41
7.31
7.30
7.29
7.28
7.28
7.27
7.26
7.26
7.25
2.50 DMSO-d6
0.98
2.01
0.98
2.00
4500
4000
3500
3000
2500
2000
1500
1000
500
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (м.д)



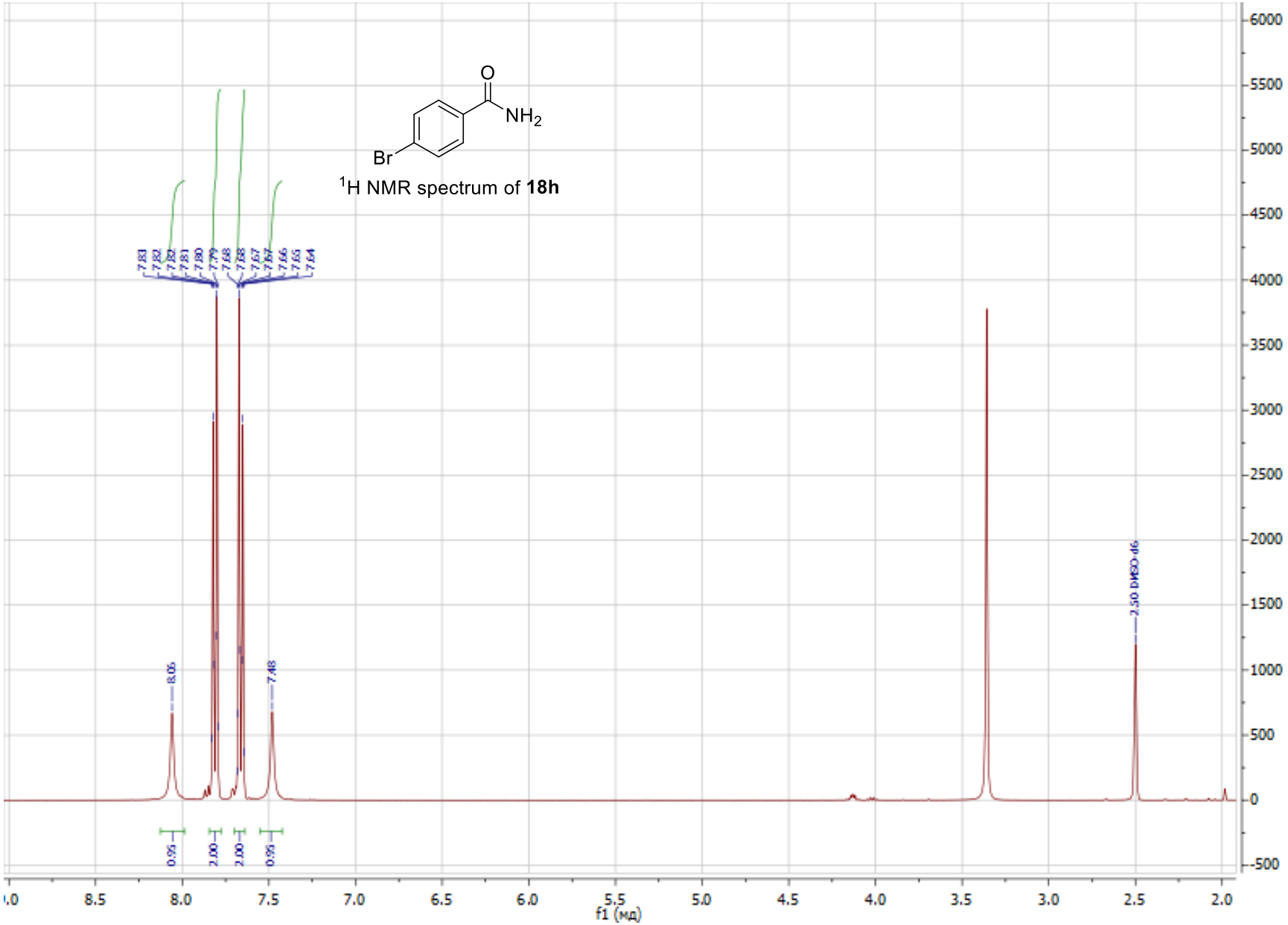
^{13}C NMR spectrum of **18d**

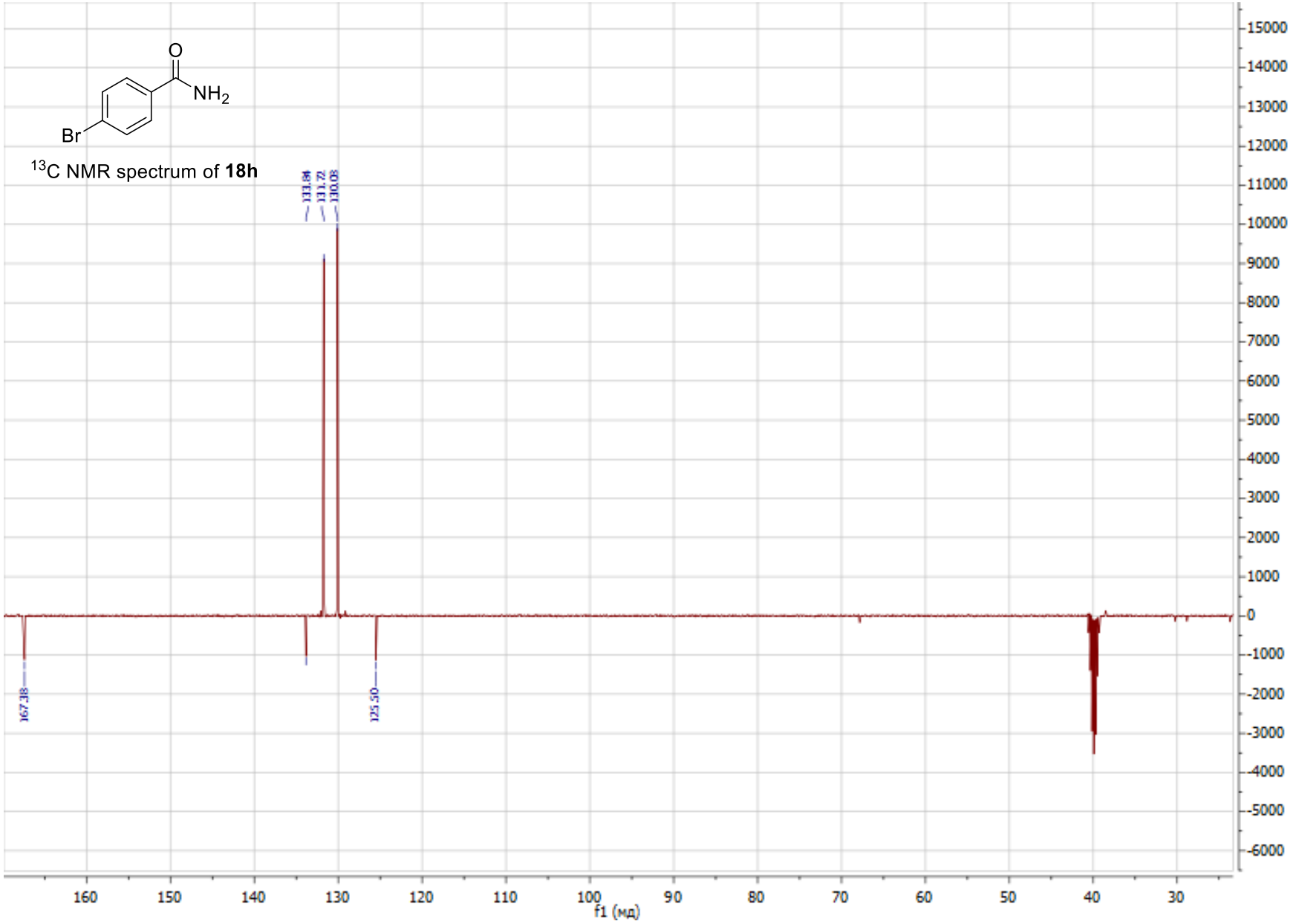


^{1}H NMR spectrum of **18f**

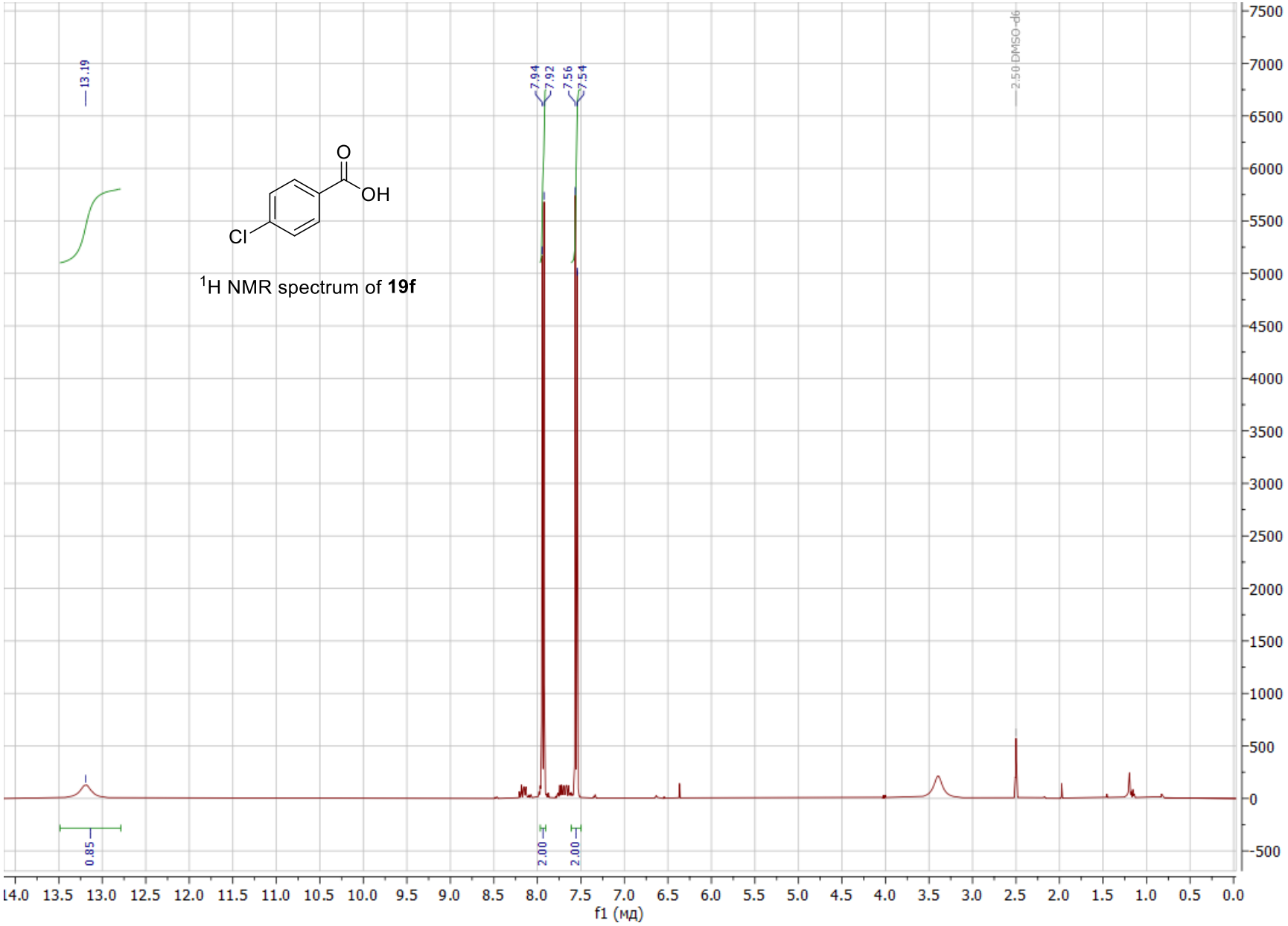


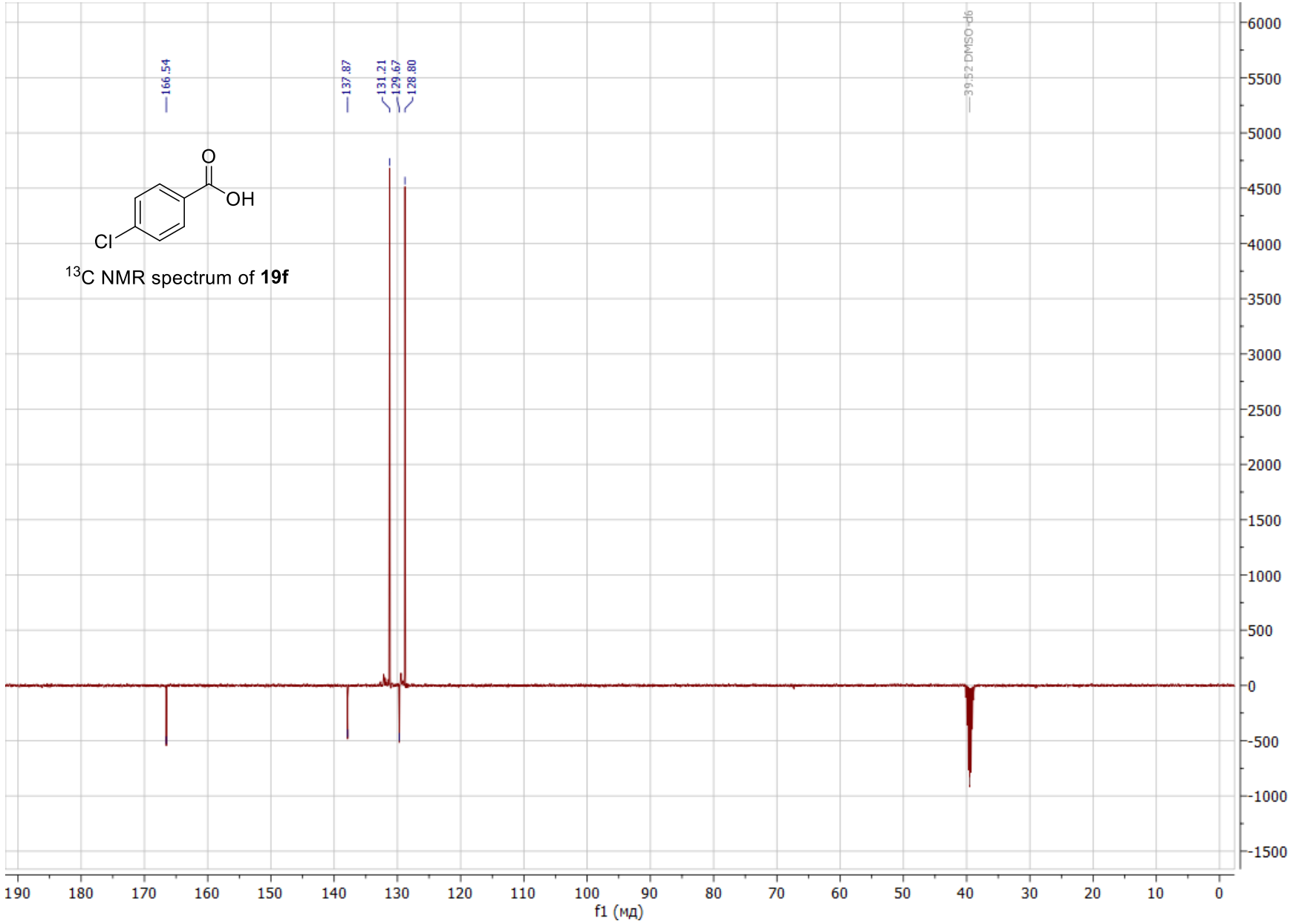
^{13}C NMR spectrum of **18f**


O
NH2
Br
1H NMR spectrum of 18h
7.83
7.82
7.82
7.81
7.80
7.79
7.68
7.68
7.67
7.67
7.66
7.65
7.64
8.05
7.48
2.50 DMSO-d6
0.95
2.00
2.00
0.99
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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
f1 (м.д)


O
NH2
Br
13C NMR spectrum of 18h
133.84
131.72
130.08
167.38
125.50
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000
-5000
-6000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (мд)

^{1}H and ^{13}C NMR spectral charts for starting 4-chlorobenzoic acid (19f)

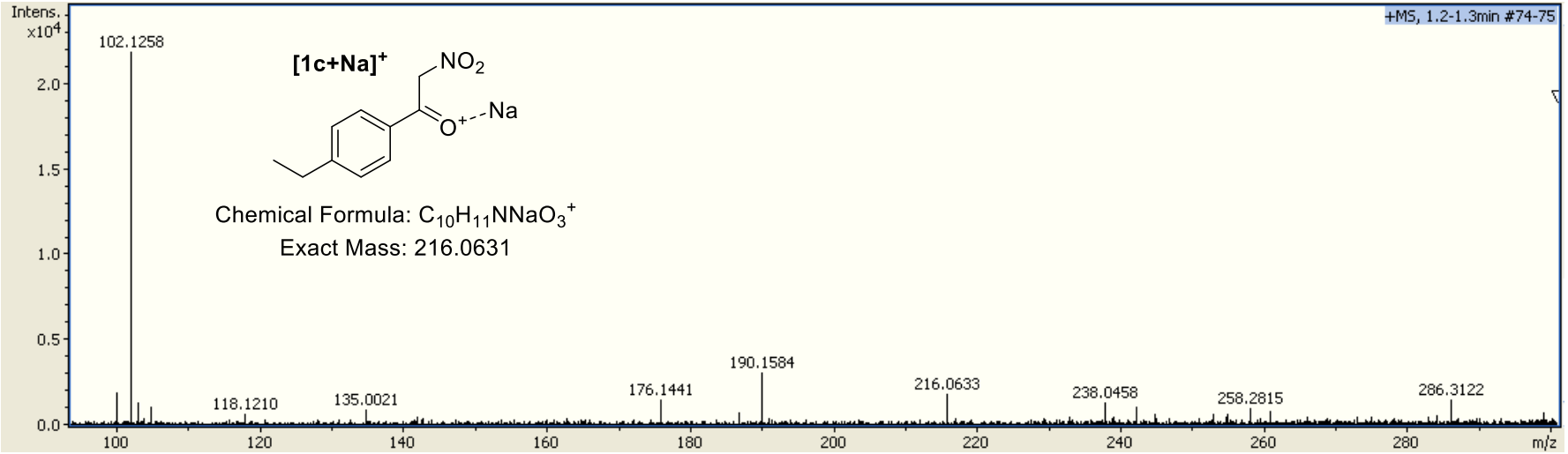




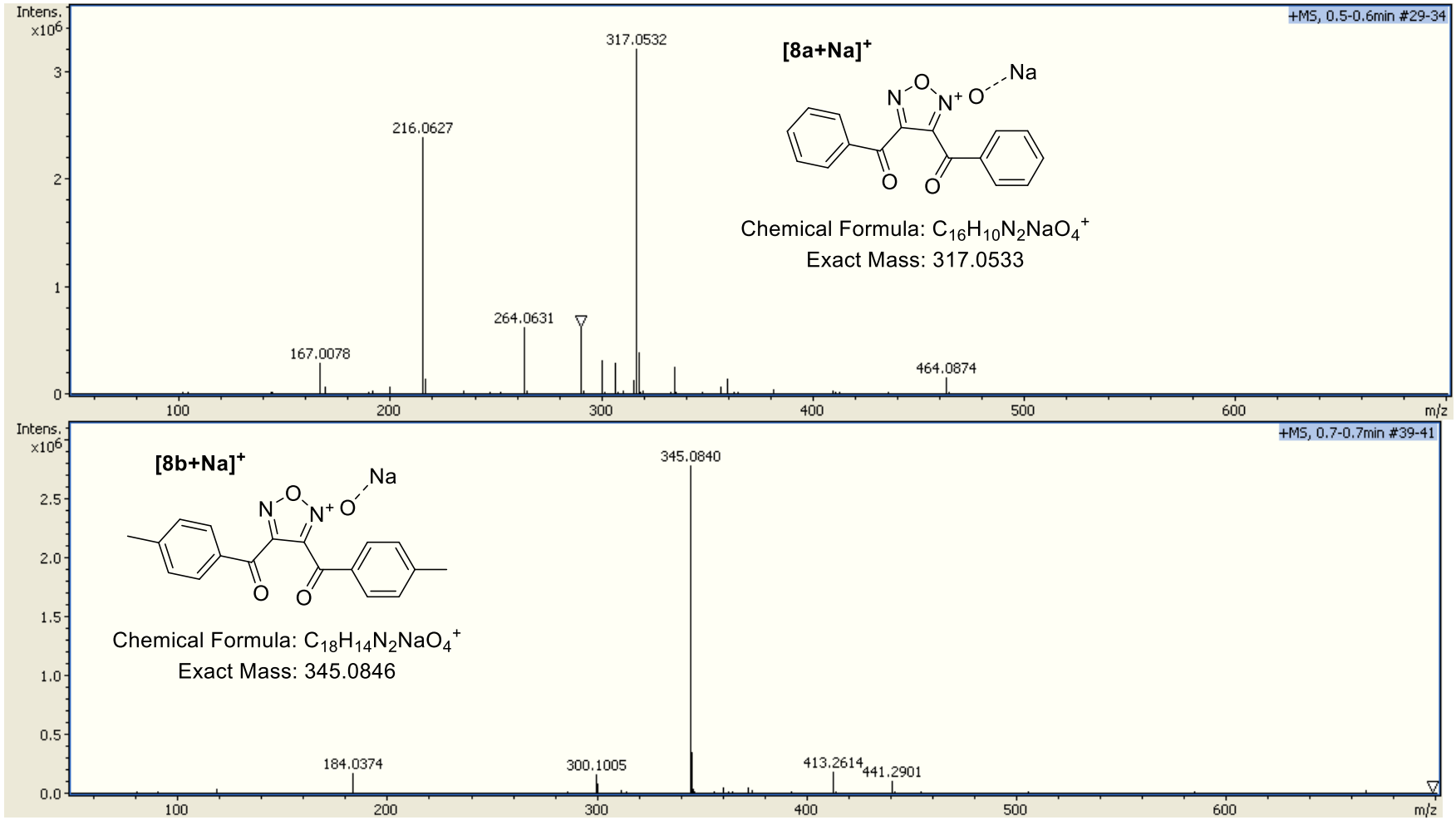
^{13}C NMR spectrum of **19f**

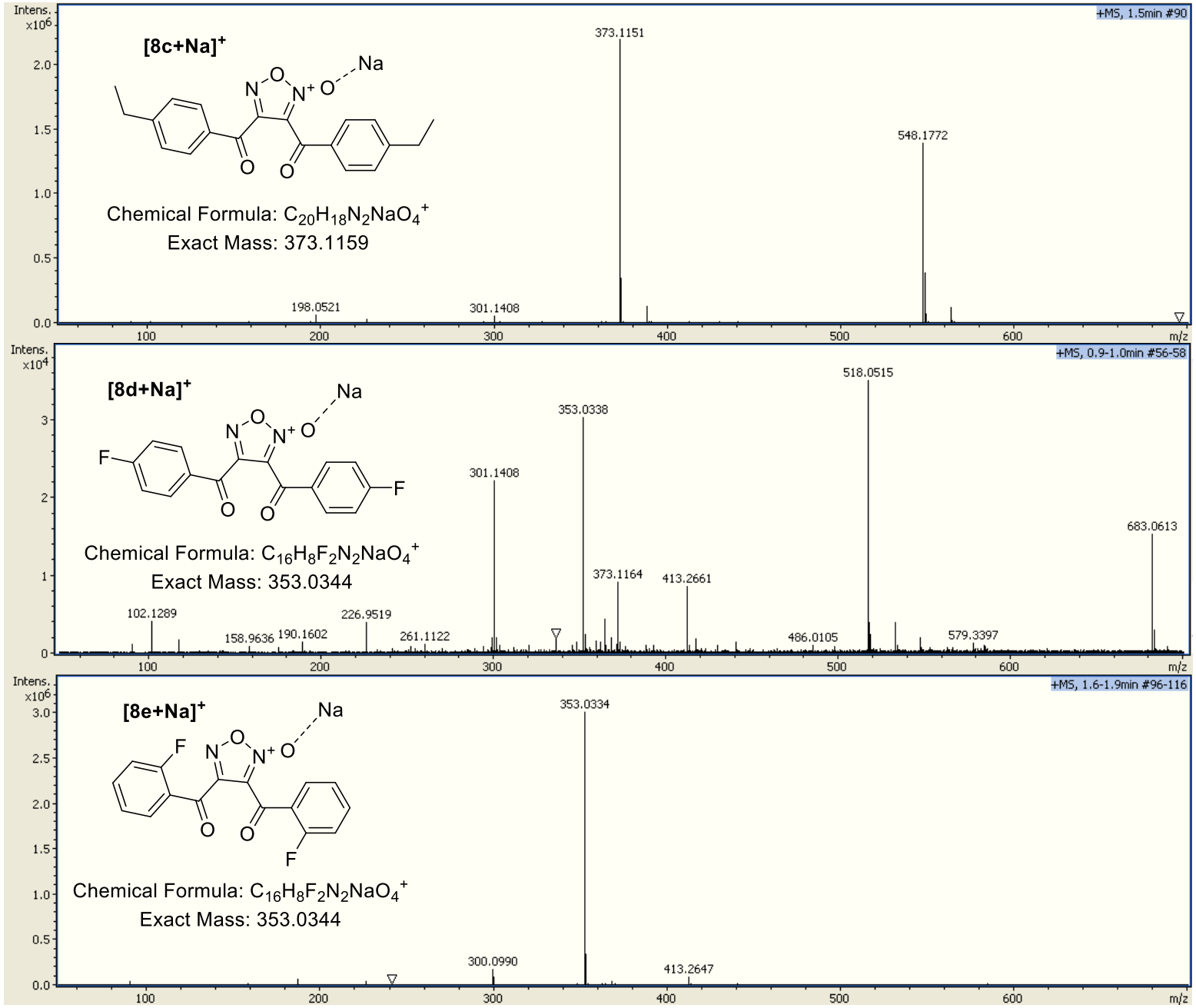
HRMS spectral charts

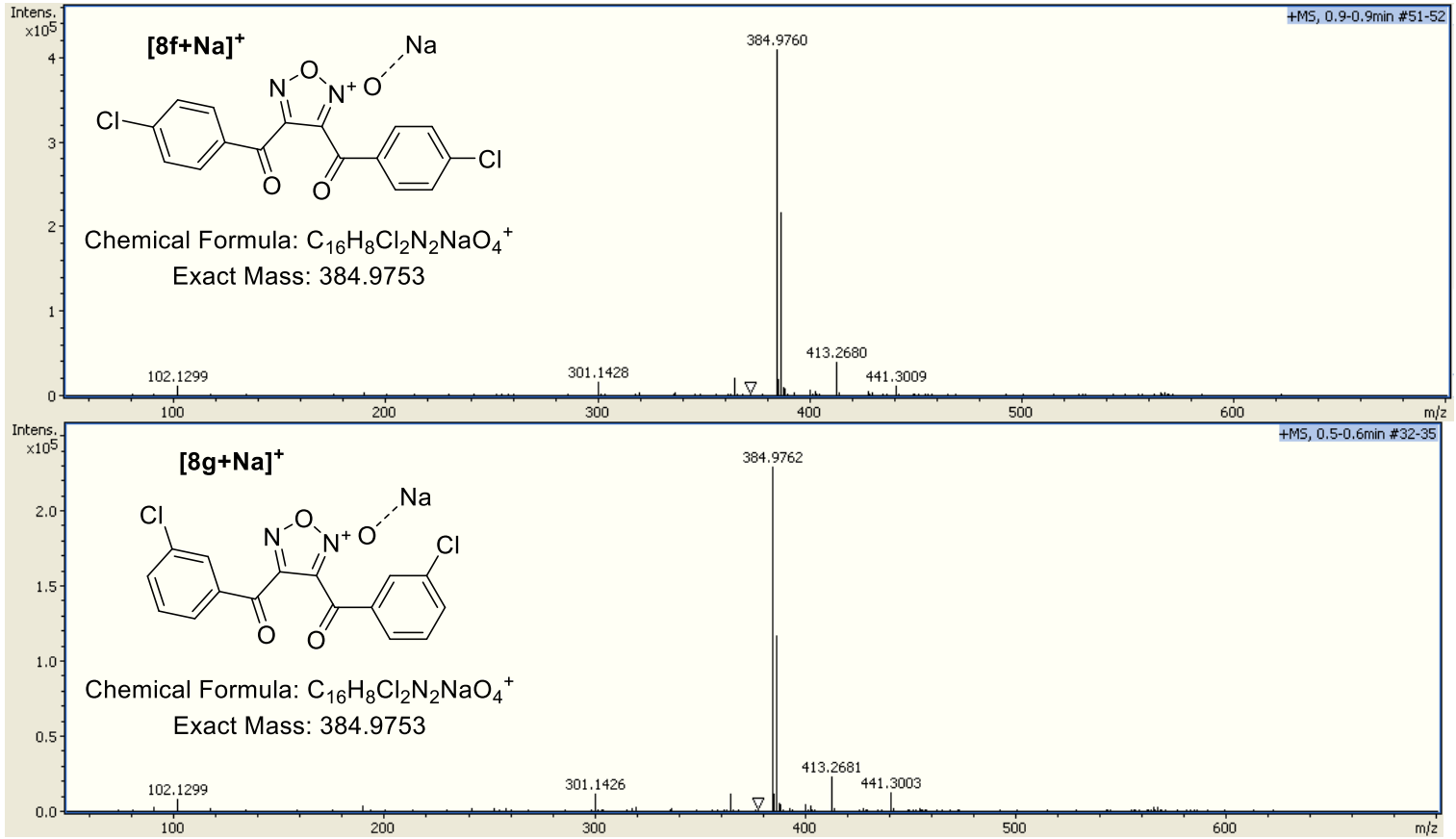
HRMS spectral charts for starting 2-nitro-4'-ethylacetophenone (1c)

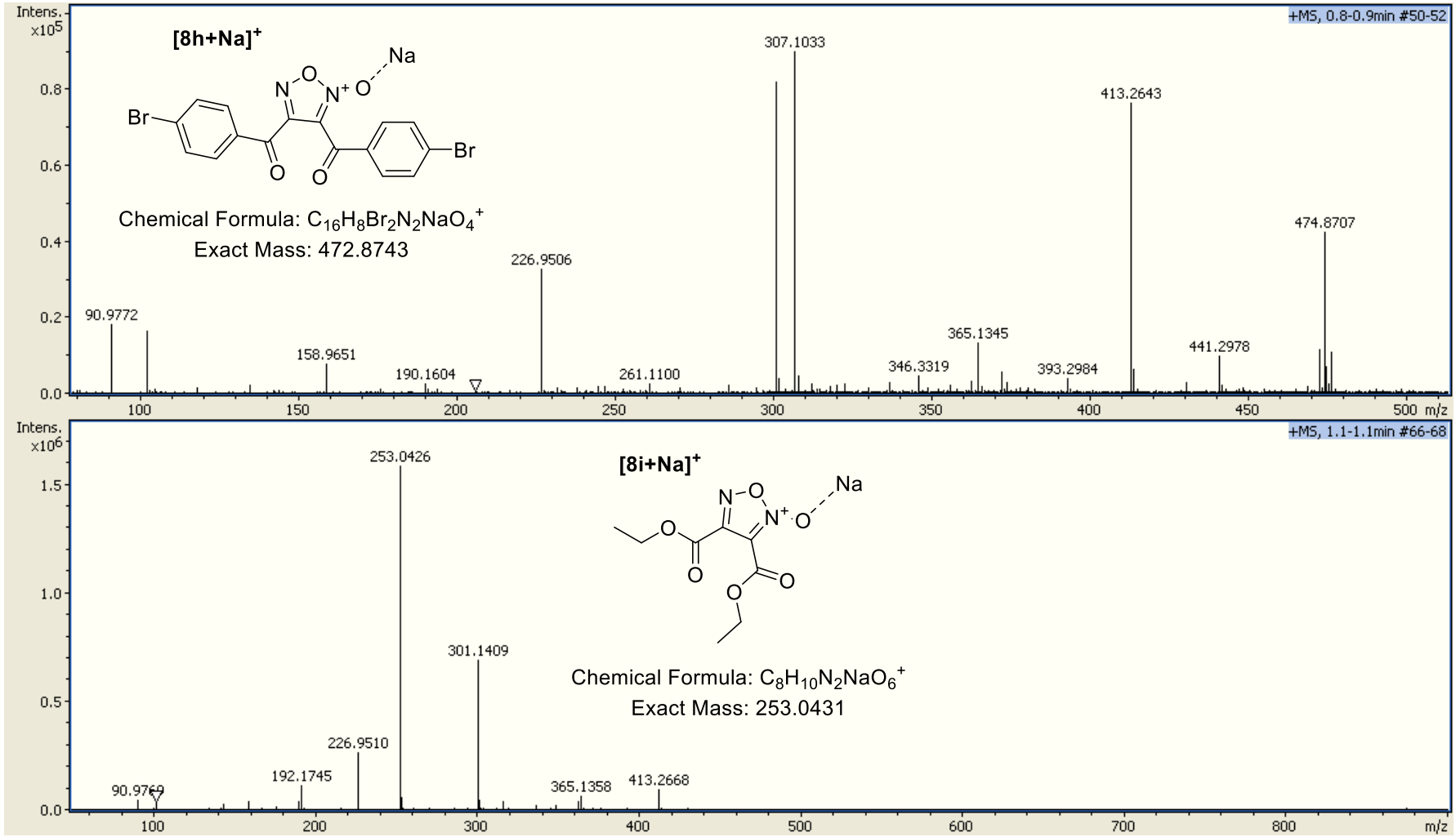


HRMS spectral charts for furoxans 8

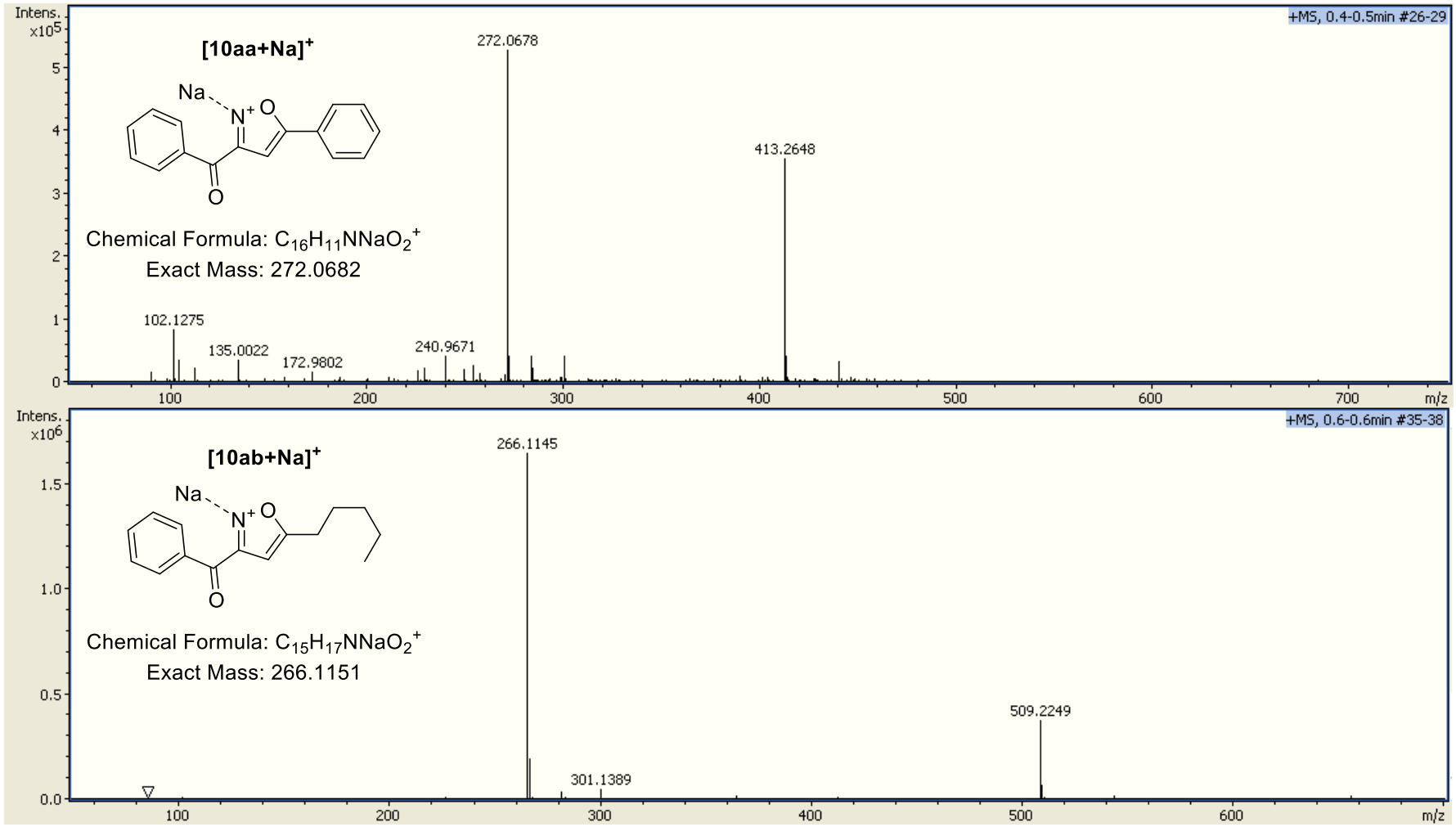


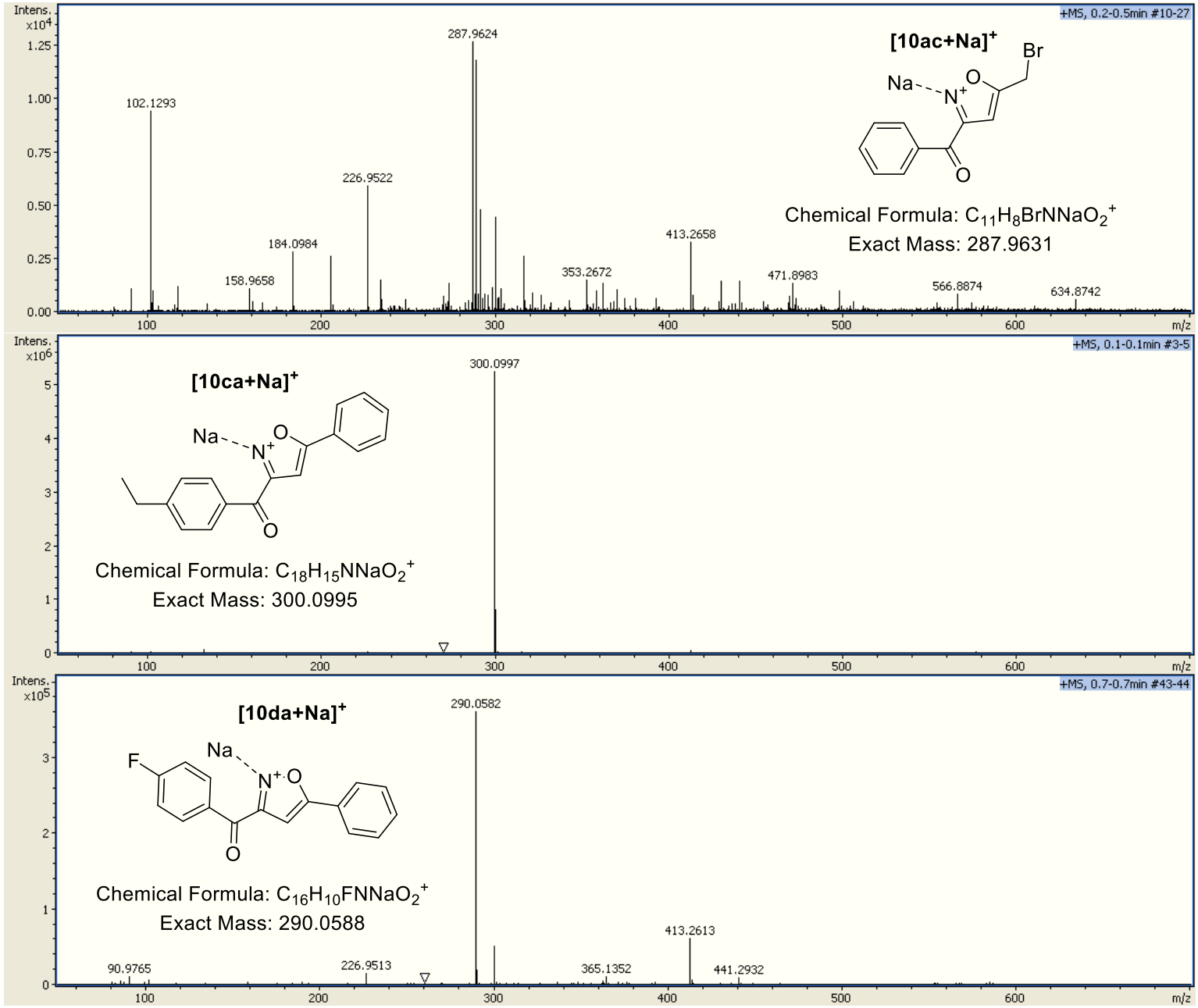

[8c+Na]+
Chemical Formula: C20H18N2NaO4+
Exact Mass: 373.1159
+MS, 1.5min #90
373.1151
548.1772
198.0521
301.1408
[8d+Na]+
Chemical Formula: C16H8F2N2NaO4+
Exact Mass: 353.0344
+MS, 0.9-1.0min #56-58
518.0515
353.0338
301.1408
683.0613
373.1164
413.2661
102.1289
158.9636
190.1602
226.9519
261.1122
486.0105
579.3397
[8e+Na]+
Chemical Formula: C16H8F2N2NaO4+
Exact Mass: 353.0344
+MS, 1.6-1.9min #96-116
353.0334
300.0990
413.2647
Intens.
m/z

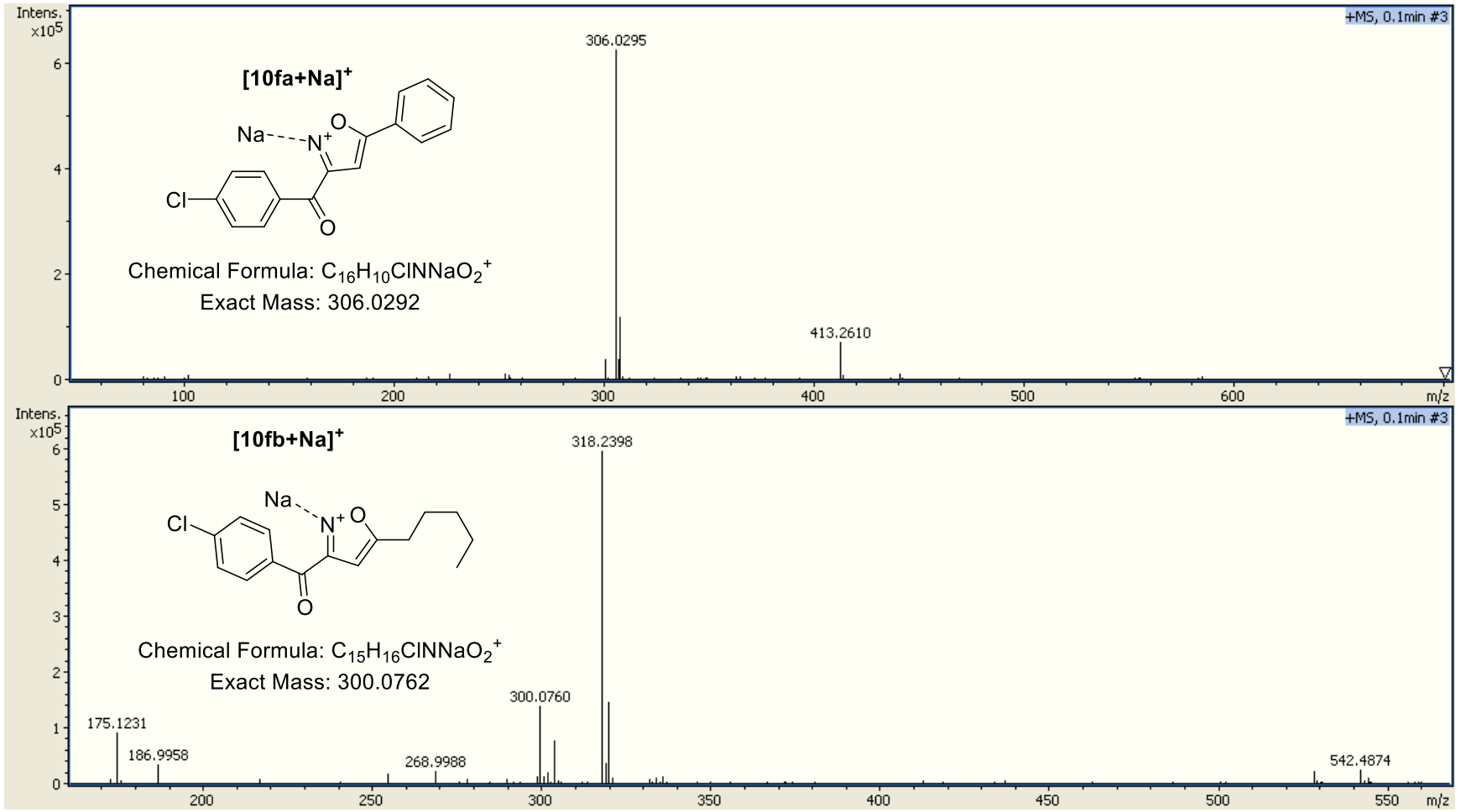

Intens.
x10^5
+MS, 0.9-0.9min #51-52
[8f+Na]+
Na
Cl
Cl
O
O
O
N
N+
O
Chemical Formula: C16H8Cl2N2NaO4+
Exact Mass: 384.9753
384.9760
413.2680
441.3009
301.1428
102.1299
m/z
Intens.
x10^5
+MS, 0.5-0.6min #32-35
[8g+Na]+
Na
Cl
Cl
O
O
O
N
N+
O
Chemical Formula: C16H8Cl2N2NaO4+
Exact Mass: 384.9753
384.9762
413.2681
441.3003
301.1426
102.1299
m/z

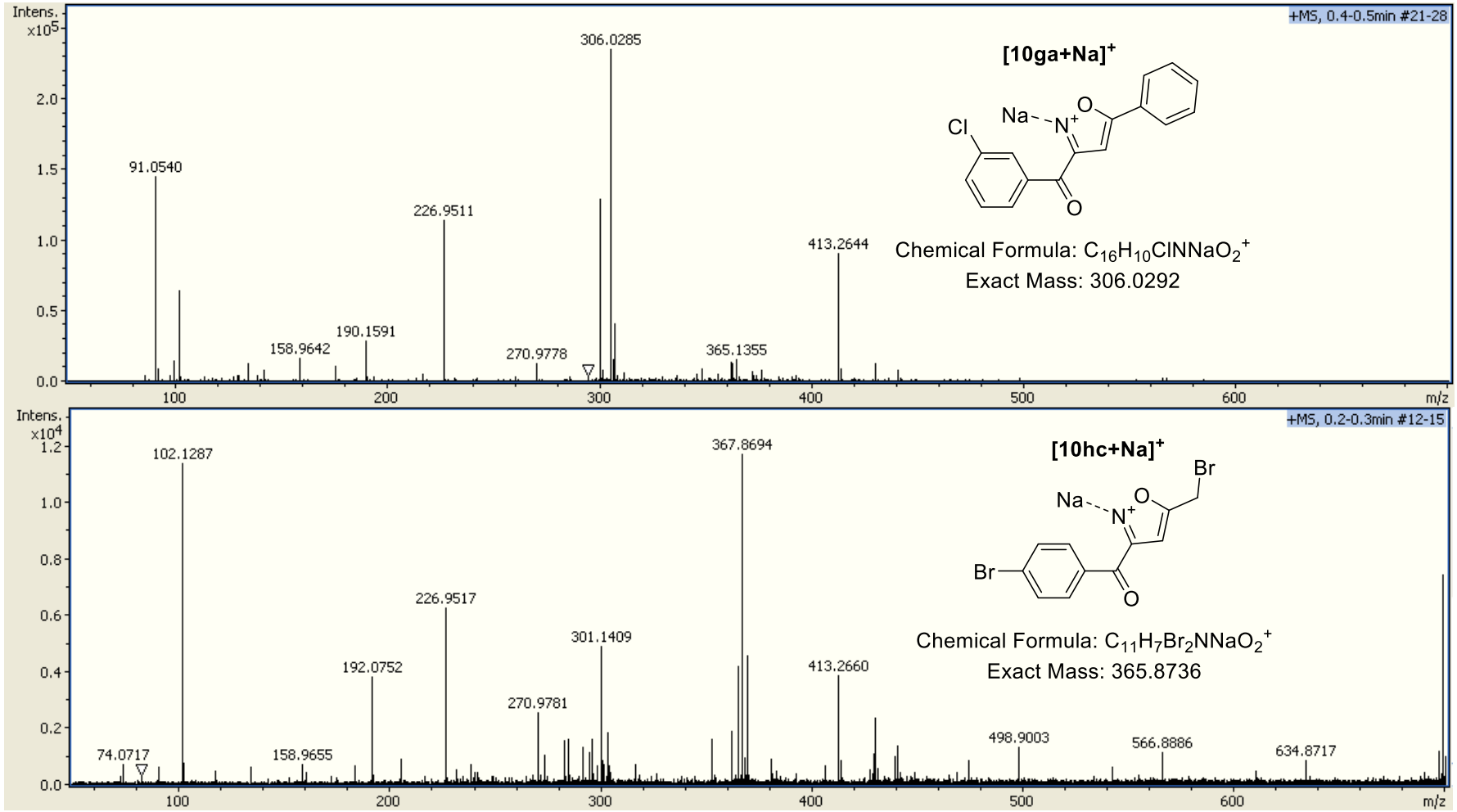

Intens.
x10^5
+MS, 0.8-0.9min #50-52
[8h+Na]+
Chemical Formula: C16H8Br2N2NaO4+
Exact Mass: 472.8743
307.1033
413.2643
474.8707
226.9506
90.9772
158.9651
190.1604
261.1100
346.3319
365.1345
393.2984
441.2978
m/z
Intens.
x10^6
+MS, 1.1-1.1min #66-68
[8i+Na]+
Chemical Formula: C8H10N2NaO6+
Exact Mass: 253.0431
253.0426
301.1409
226.9510
192.1745
90.9769
365.1358
413.2668
m/z

HRMS spectral charts for isoxazoles 10

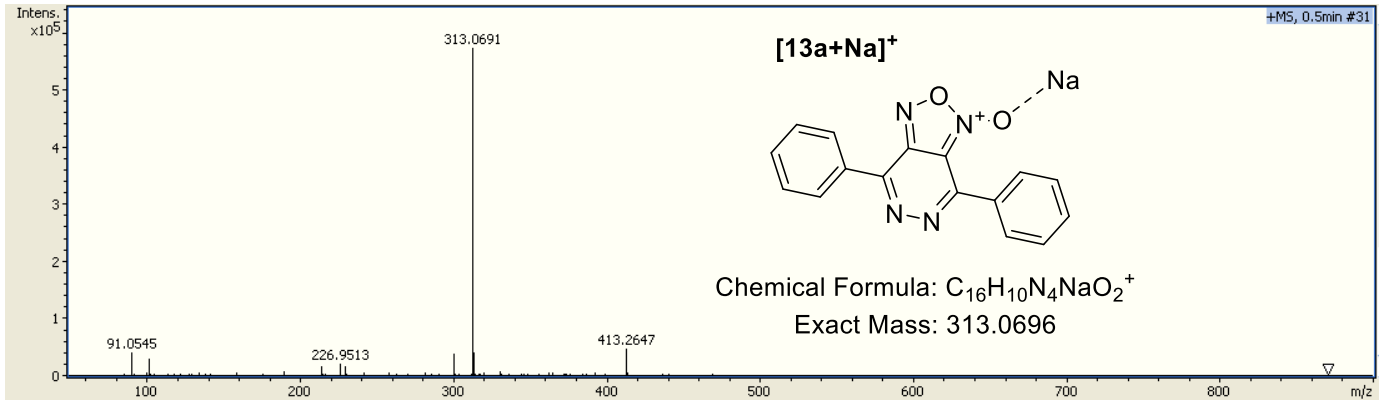



Intens.
x10^4
+MS, 0.2-0.5min #10-27
[10ac+Na]+
Br
Na
O
N+
O
287.9624
102.1293
226.9522
184.0984
158.9658
353.2672
413.2658
471.8983
566.8874
634.8742
Chemical Formula: C11H8BrNNaO2+
Exact Mass: 287.9631
m/z
Intens.
x10^6
+MS, 0.1-0.1min #3-5
[10ca+Na]+
Na
O
N+
O
300.0997
Chemical Formula: C18H15NNaO2+
Exact Mass: 300.0995
m/z
Intens.
x10^5
+MS, 0.7-0.7min #43-44
[10da+Na]+
F
Na
N+
O
O
290.0582
413.2613
90.9765
226.9513
365.1352
441.2932
Chemical Formula: C16H10FNNaO2+
Exact Mass: 290.0588
m/z

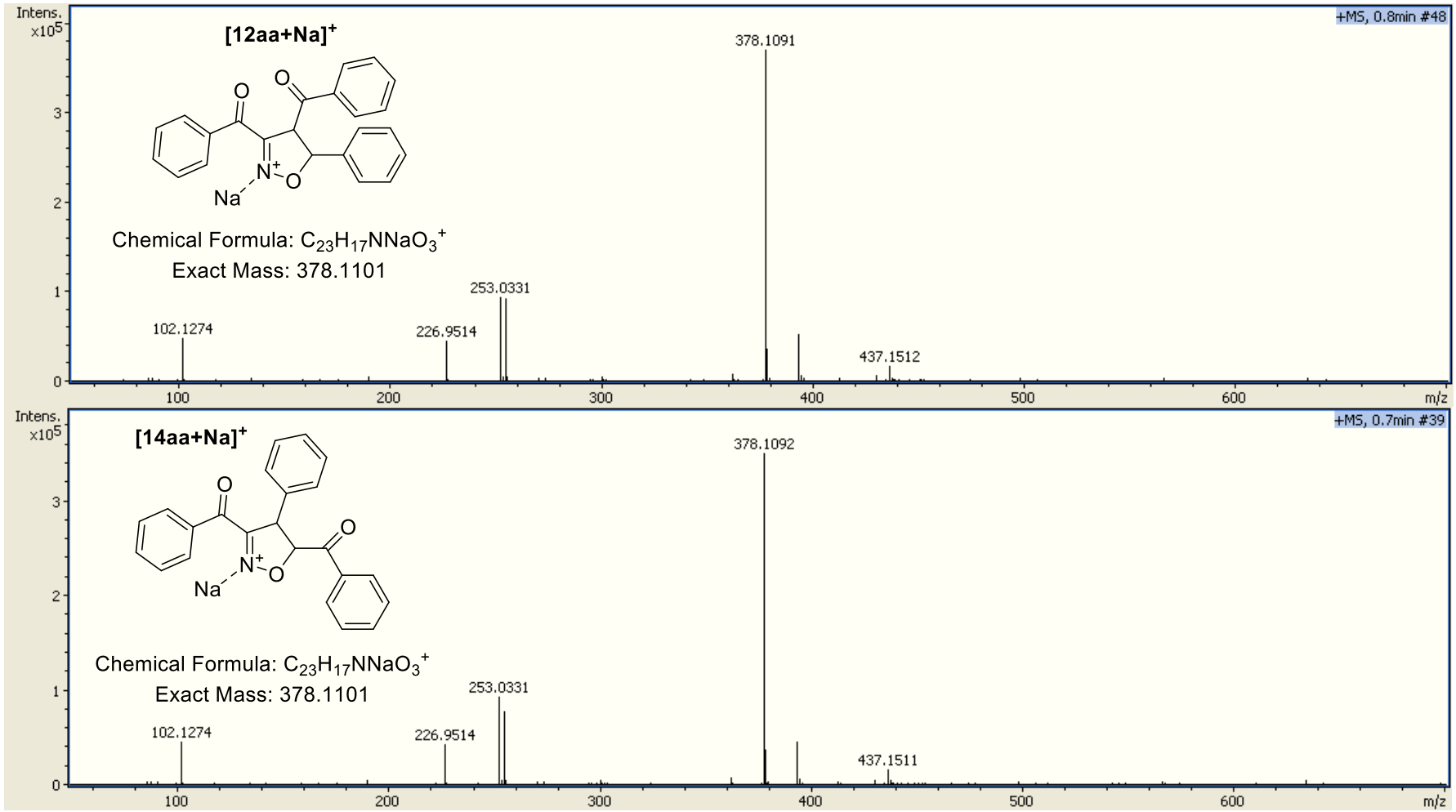

Intens.
x10^5
+MS, 0.1min #3
306.0295
[10fa+Na]+
Na
O
N+
Cl
O
Chemical Formula: C16H10ClNNaO2+
Exact Mass: 306.0292
413.2610
m/z
Intens.
x10^5
+MS, 0.1min #3
[10fb+Na]+
318.2398
Na
Cl
N+
O
O
Chemical Formula: C15H16ClNNaO2+
Exact Mass: 300.0762
300.0760
175.1231
186.9958
268.9988
542.4874
m/z

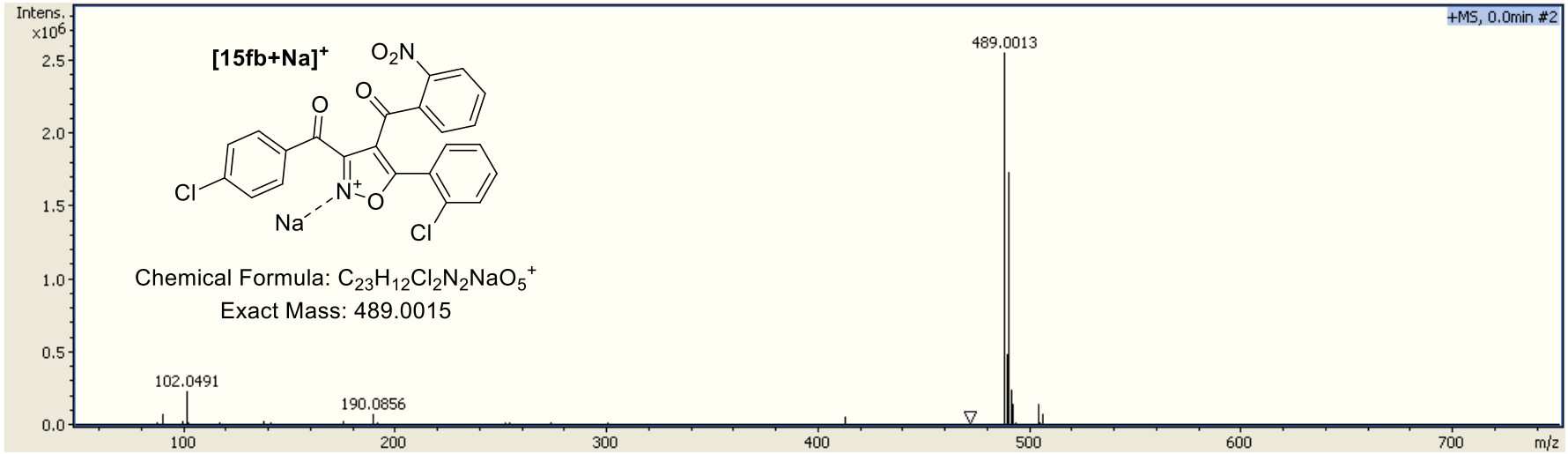

Intens.
x10^5
+MS, 0.4-0.5min #21-28
306.0285
[10ga+Na]+
91.0540
226.9511
413.2644
190.1591
158.9642
270.9778
365.1355
Chemical Formula: C16H10ClNNaO2+
Exact Mass: 306.0292
m/z
Intens.
x10^4
+MS, 0.2-0.3min #12-15
102.1287
367.8694
[10hc+Na]+
226.9517
301.1409
192.0752
413.2660
270.9781
74.0717
158.9655
498.9003
566.8886
634.8717
Chemical Formula: C11H7Br2NNaO2+
Exact Mass: 365.8736
m/z

HRMS spectral charts for 4,7-diphenyl-[1,2,5]oxadiazolo[3,4-d]pyridazine 1-oxide (13a):

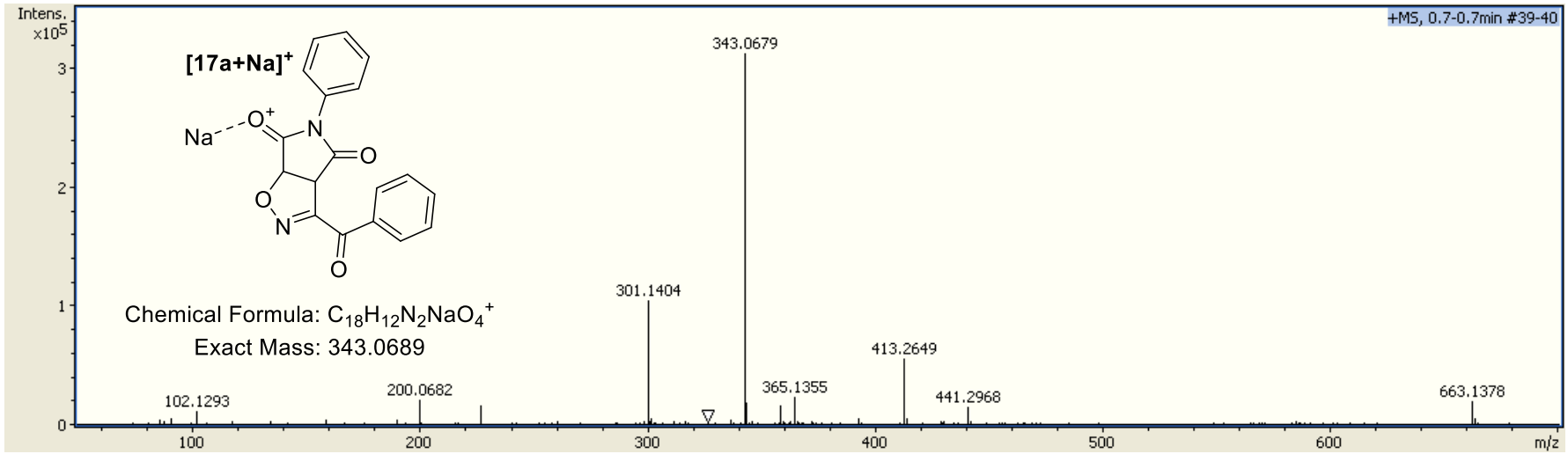


HRMS spectral charts for cycloadducts with chalcones 12aa, 14aa, 15fb:





HRMS spectral charts for 3-benzoyl-5-phenyl-3a,6a-dihydro-4H-pyrrolo[3,4-d]isoxazole-4,6(5H)-dione (17a):



X-Ray crystallography data

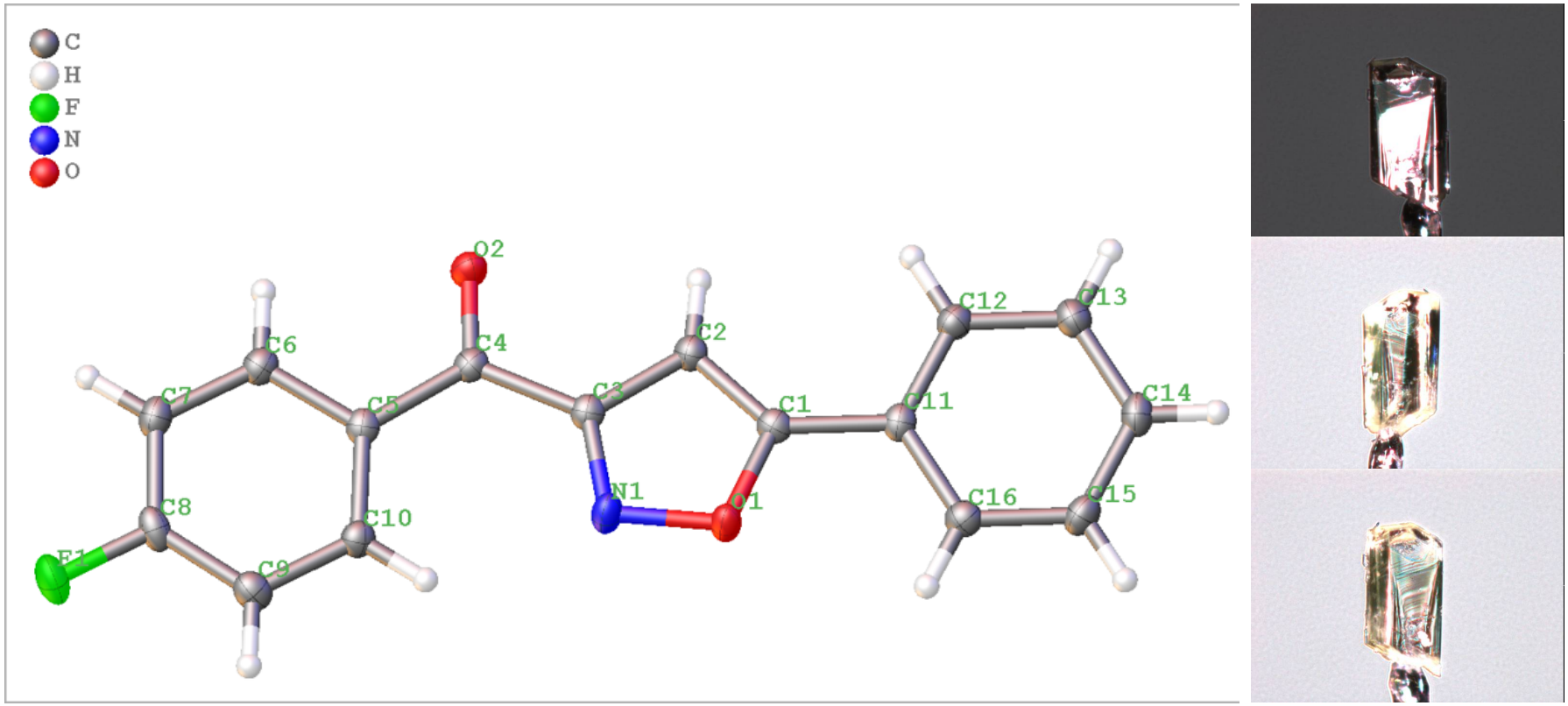


Figure S1. ORTEP drawing of the crystal structure (left) and microphotography of the single crystal of compound **10da** used for X-Ray diffraction analysis (right)

Table S1 Crystal data and structure refinement for 10da.

Identification code	ANNA_KUR182_3
Empirical formula	$C_{16}H_{10}FNO_2$
Formula weight	267.25
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.77617(12)
b/Å	28.7775(6)
c/Å	7.41435(13)
α/°	90
β/°	95.1230(19)
γ/°	90
Volume/Å^3	1227.52(4)
Z	4
ρ_{calc}g/cm^3	1.446
μ/mm^{-1}	0.888
F(000)	552.0
Crystal size/mm^3	0.576 × 0.289 × 0.124
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	12.302 to 152.478
Index ranges	$-7 \leq h \leq 7$, $-35 \leq k \leq 36$, $-6 \leq l \leq 9$
Reflections collected	12715
Independent reflections	2566 [R_{int} = 0.0452, R_{sigma} = 0.0238]
Data/restraints/parameters	2566/0/169
Goodness-of-fit on F^2	1.072
Final R indexes [I>=2σ (I)]	R_1 = 0.0513, wR_2 = 0.1360
Final R indexes [all data]	R_1 = 0.0533, wR_2 = 0.1374
Largest diff. peak/hole / e Å^{-3}	0.38/-0.31

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 10da. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
F1	14576.8(19)	3189.4(4)	5379.5(14)	28.6(3)
O2	7051(2)	4471.0(4)	1342.2(16)	23.0(3)
O1	11058(2)	5764.8(4)	2646.6(16)	20.9(3)
N1	11294(3)	5288.9(5)	2987.6(19)	21.6(3)
C4	8893(3)	4587.8(5)	2164(2)	16.6(3)
C8	13225(3)	3535.0(6)	4622(2)	20.4(3)
C2	7877(3)	5435.5(5)	1330(2)	17.2(3)
C3	9396(3)	5099.7(5)	2194.9(19)	16.3(3)
C11	8482.1(17)	6329.0(2)	1154.6(13)	16.7(3)
C16	10154.3(14)	6672.9(3)	1492.5(13)	19.6(3)
C15	9661.5(16)	7128.7(3)	969.8(14)	21.5(3)
C14	7496.6(18)	7240.6(2)	109.1(14)	21.4(4)
C13	5824.4(14)	6896.6(3)	-228.9(14)	21.6(3)
C12	6317.1(15)	6440.8(3)	293.9(14)	19.3(3)
C10	12707(3)	4333.1(5)	3866(2)	18.1(3)
C1	8998(3)	5844.0(5)	1653.6(19)	16.3(3)
C9	14093(3)	3979.6(6)	4662(2)	20.7(3)
C7	11029(3)	3420.7(5)	3839(2)	20.3(3)
C5	10482(3)	4234.2(5)	3061.8(19)	16.3(3)
C6	9665(3)	3774.4(5)	3054(2)	18.8(3)

Table S3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 10da. 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}
F1	34.3(6)	21.2(5)	28.7(5)	1.4(4)	-5.6(4)	9.6(4)
O2	21.2(6)	19.4(6)	27.3(6)	1.4(4)	-4.4(5)	-2.7(4)
O1	22.7(6)	14.2(5)	24.0(6)	1.2(4)	-7.1(5)	-1.2(4)
N1	26.2(7)	13.7(6)	23.8(7)	0.7(5)	-4.2(5)	-1.4(5)
C4	18.7(7)	17.7(7)	13.7(7)	-0.5(5)	2.8(5)	-1.5(6)
C8	28.1(9)	19.0(8)	14.0(7)	0.2(6)	0.8(6)	8.0(6)
C2	19.7(7)	17.2(7)	14.7(7)	0.3(5)	1.2(5)	-0.1(6)
C3	19.7(7)	17.8(7)	11.4(7)	-0.8(5)	2.1(5)	0.0(6)
C11	21.9(8)	16.2(7)	12.3(7)	-0.4(5)	3.0(6)	0.1(6)
C16	20.5(8)	21.1(8)	16.8(7)	0.1(6)	0.3(6)	-1.1(6)
C15	26.8(8)	17.6(7)	20.2(8)	-1.3(6)	1.9(6)	-4.6(6)
C14	29.3(9)	15.6(7)	19.7(8)	0.8(6)	3.5(6)	2.2(6)
C13	21.9(8)	20.8(8)	22.1(8)	0.9(6)	1.6(6)	2.6(6)
C12	20.5(8)	17.8(7)	19.7(7)	-1.3(6)	2.3(6)	-1.7(6)
C10	20.9(8)	17.1(7)	16.4(7)	-2.0(5)	1.1(6)	-0.5(6)
C1	18.1(7)	19.2(8)	11.5(6)	-0.5(5)	1.1(5)	0.4(6)
C9	21.7(8)	22.8(8)	17.3(7)	-2.7(6)	-0.8(6)	2.5(6)
C7	27.5(8)	16.0(7)	17.8(7)	-0.2(6)	3.7(6)	-0.7(6)
C5	20.7(8)	17.1(7)	11.3(6)	-0.8(5)	2.6(5)	0.3(6)
C6	21.9(8)	18.5(8)	16.3(7)	-1.4(6)	2.2(6)	-1.4(6)

Table S4 Bond Lengths for 10da.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
F1	C8	1.3548(18)	C11	C16	1.3900
O2	C4	1.225(2)	C11	C12	1.3900
O1	N1	1.3970(17)	C11	C1	1.4674(17)
O1	C1	1.3615(19)	C16	C15	1.3900
N1	C3	1.315(2)	C15	C14	1.3900
C4	C3	1.501(2)	C14	C13	1.3900
C4	C5	1.487(2)	C13	C12	1.3900
C8	C9	1.374(2)	C10	C9	1.393(2)
C8	C7	1.387(2)	C10	C5	1.397(2)
C2	C3	1.419(2)	C7	C6	1.383(2)
C2	C1	1.353(2)	C5	C6	1.405(2)

Table S5 Bond Angles for 10da.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	O1	N1	109.17(12)	C11	C16	C15	120.0
C3	N1	O1	105.15(12)	C14	C15	C16	120.0
O2	C4	C3	115.81(14)	C15	C14	C13	120.0
O2	C4	C5	120.59(14)	C14	C13	C12	120.0
C5	C4	C3	123.60(14)	C13	C12	C11	120.0
F1	C8	C9	118.64(15)	C9	C10	C5	120.29(15)
F1	C8	C7	118.11(15)	O1	C1	C11	116.33(12)
C9	C8	C7	123.25(15)	C2	C1	O1	109.37(13)
C1	C2	C3	104.16(14)	C2	C1	C11	134.30(14)
N1	C3	C4	124.47(14)	C8	C9	C10	118.45(16)
N1	C3	C2	112.15(14)	C6	C7	C8	117.85(15)
C2	C3	C4	123.37(14)	C10	C5	C4	124.01(14)
C16	C11	C12	120.0	C10	C5	C6	119.33(14)
C16	C11	C1	120.64(9)	C6	C5	C4	116.65(14)
C12	C11	C1	119.34(9)	C7	C6	C5	120.84(15)

Table S6 Torsion Angles for 10da.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
F1	C8	C9	C10	179.37(13)	C16	C15	C14	C13	0.0
F1	C8	C7	C6	-179.43(13)	C15	C14	C13	C12	0.0
O2	C4	C3	N1	-179.10(15)	C14	C13	C12	C11	0.0
O2	C4	C3	C2	-0.4(2)	C12	C11	C16	C15	0.0
O2	C4	C5	C10	173.32(14)	C12	C11	C1	O1	-173.83(9)
O2	C4	C5	C6	-5.6(2)	C12	C11	C1	C2	7.2(2)
O1	N1	C3	C4	178.64(13)	C10	C5	C6	C7	0.4(2)
O1	N1	C3	C2	-0.19(17)	C1	O1	N1	C3	0.17(16)
N1	O1	C1	C2	-0.09(17)	C1	C2	C3	N1	0.14(18)
N1	O1	C1	C11	-179.31(11)	C1	C2	C3	C4	-178.71(13)
C4	C5	C6	C7	179.33(13)	C1	C11	C16	C15	178.32(11)
C8	C7	C6	C5	-0.3(2)	C1	C11	C12	C13	-178.34(10)
C3	C4	C5	C10	-6.9(2)	C9	C8	C7	C6	0.4(2)
C3	C4	C5	C6	174.22(13)	C9	C10	C5	C4	-179.30(14)
C3	C2	C1	O1	-0.02(17)	C9	C10	C5	C6	-0.4(2)
C3	C2	C1	C11	179.00(15)	C7	C8	C9	C10	-0.4(2)
C11	C16	C15	C14	0.0	C5	C4	C3	N1	1.1(2)
C16	C11	C12	C13	0.0	C5	C4	C3	C2	179.77(14)
C16	C11	C1	O1	7.84(16)	C5	C10	C9	C8	0.4(2)
C16	C11	C1	C2	-171.14(15)					

Table S7 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for 10da.

Atom	*x*	*y*	*z*	U(eq)
H2	6440.04	5386.07	688.25	21
H16	11602.73	6598.05	2068.4	23
H15	10780.28	7358.82	1195.86	26
H14	7166.9	7545.53	-240.69	26
H13	4375.94	6971.48	-804.73	26
H12	5198.35	6210.71	67.8	23
H10	13264.84	4636.27	3870.07	22
H9	15572.31	4043.14	5206.83	25
H7	10490.01	3116.2	3841.4	24
H6	8187.86	3706.87	2513.62	23

Experimental

Single crystals of $C_{16}H_{10}FNO_2$ **10da** were prepared by cycloaddition reaction in PPA. A suitable crystal was selected and mounted on the glass stick by acrylic **glue** on a SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2[1], the structure was solved with the SHELXD[2] structure solution program using Dual Space and refined with the SHELXL[3] refinement package using Least Squares minimisation.

Crystal structure determination of [10da]

Crystal Data for $C_{16}H_{10}FNO_2$ (M =267.25 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 5.77617(12) Å, b = 28.7775(6) Å, c = 7.41435(13) Å, β = 95.1230(19)°, V = 1227.52(4) Å^3, Z = 4, T = 100.00(10) K, μ(Cu Kα) = 0.888 mm^{-1}, *Dcalc* = 1.446 g/cm^3, 12715 reflections measured (12.302° ≤ 2Θ ≤ 152.478°), 2566 unique (R_{int} = 0.0452, R_{sigma} = 0.0238) which were used in all calculations. The final R_1 was 0.0513 (I > 2σ(I)) and wR_2 was 0.1374 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2.a Aromatic/amide H refined with riding coordinates:
 C2(H2), C16(H16), C15(H15), C14(H14), C13(H13), C12(H12), C10(H10), C9(H9),
 C7(H7), C6(H6)
2.b Fitted hexagon refined as free rotating group:
 C11(C16,C15,C14,C13,C12)

References

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2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.