Electronic Supplementary Material (ESI) for RSC Advances.

Unexpected Cyclization of *ortho*-Nitrochalcones into 2-Alkylideneindolin-3-ones

Nicolai A. Aksenov,*[a] Dmitrii A. Aksenov,[a] Nikolai A. Arutiunov,[a] Daria S. Aksenova,[a] Alexander V. Aksenov,[a] and Michael Rubin*[a,b]

a. *Department of Chemistry, North Caucasus Federal University, 1a Pushkin St., Stavropol 355009, Russian Federation.*
b. *Department of Chemistry, University of Kansas, 1567 Irving Hill Rd., Lawrence, KS 66045-7582, USA. Tel: +1-785-864- 5071, E-mail: mrubin@ku.edu*

Supporting Information

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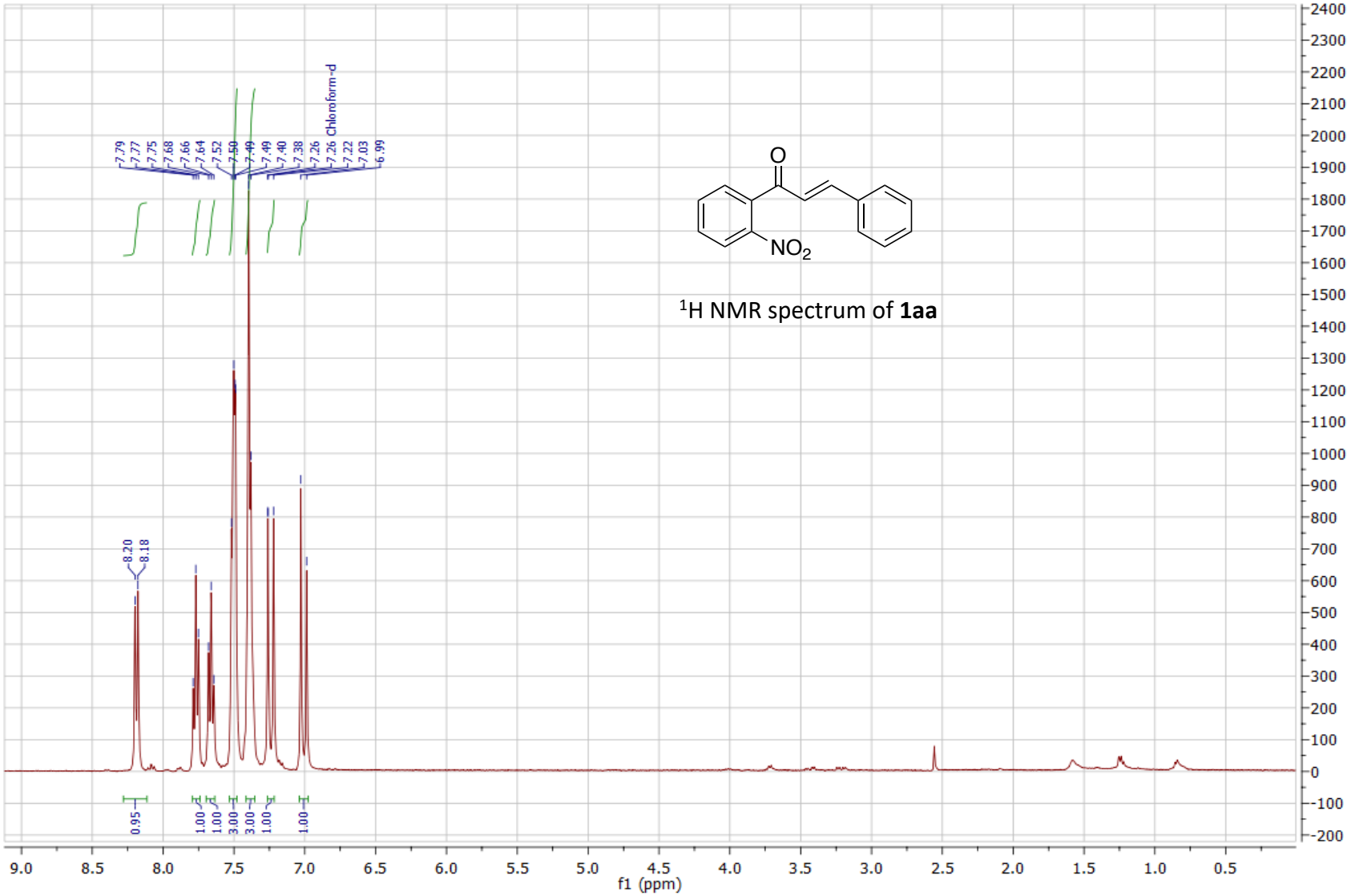
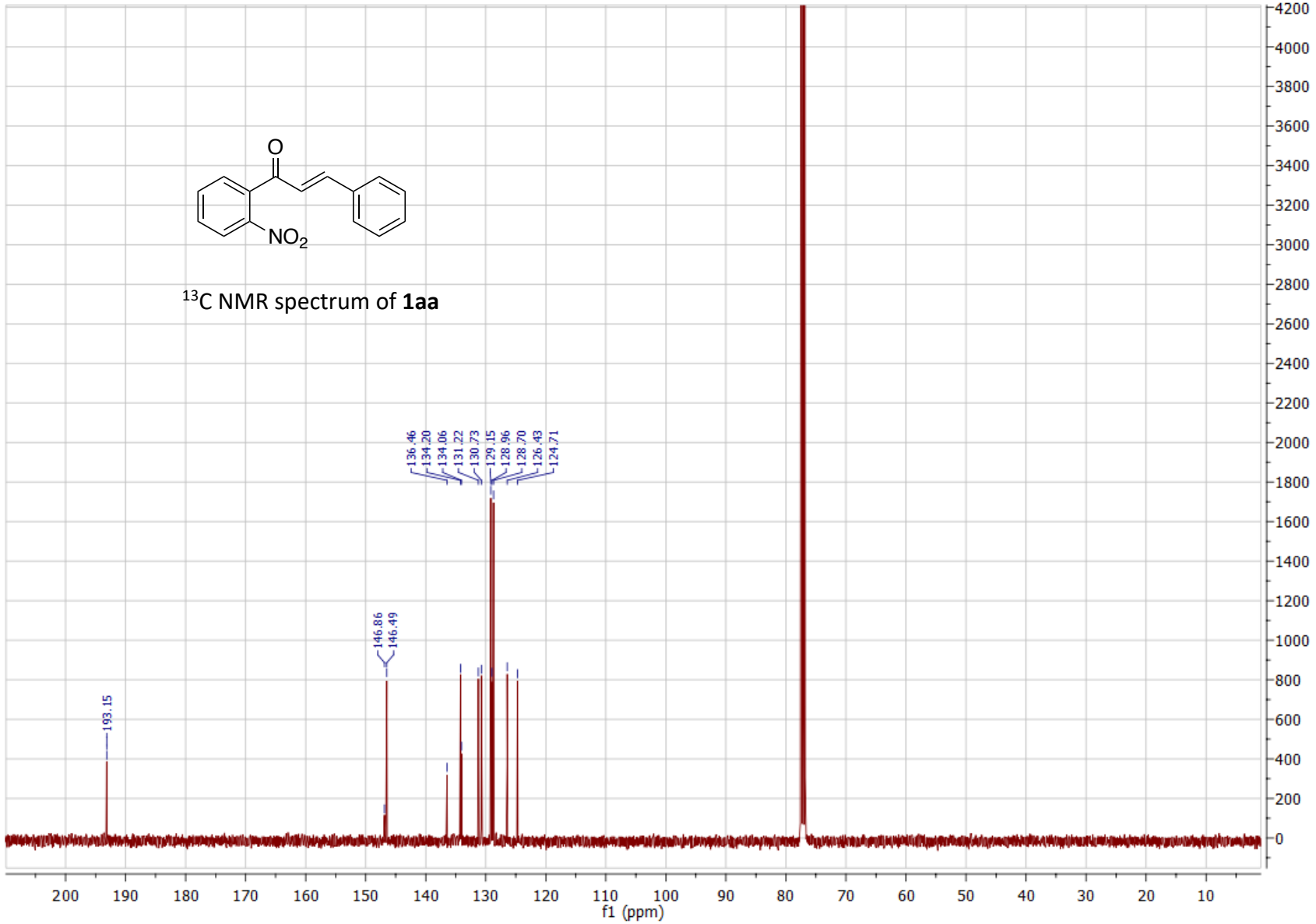
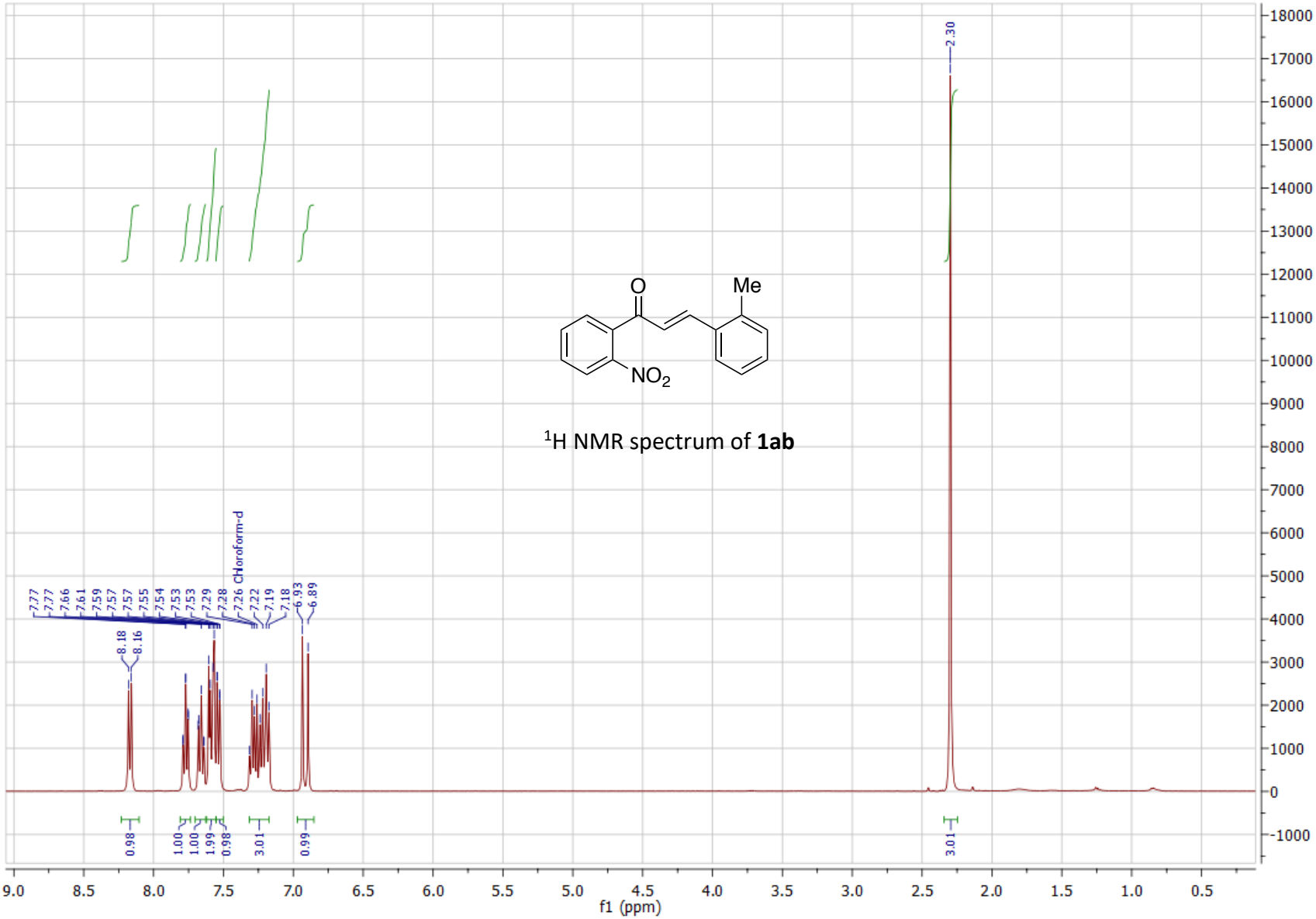
^{1}H and ^{13}C NMR spectral charts for starting chalcones



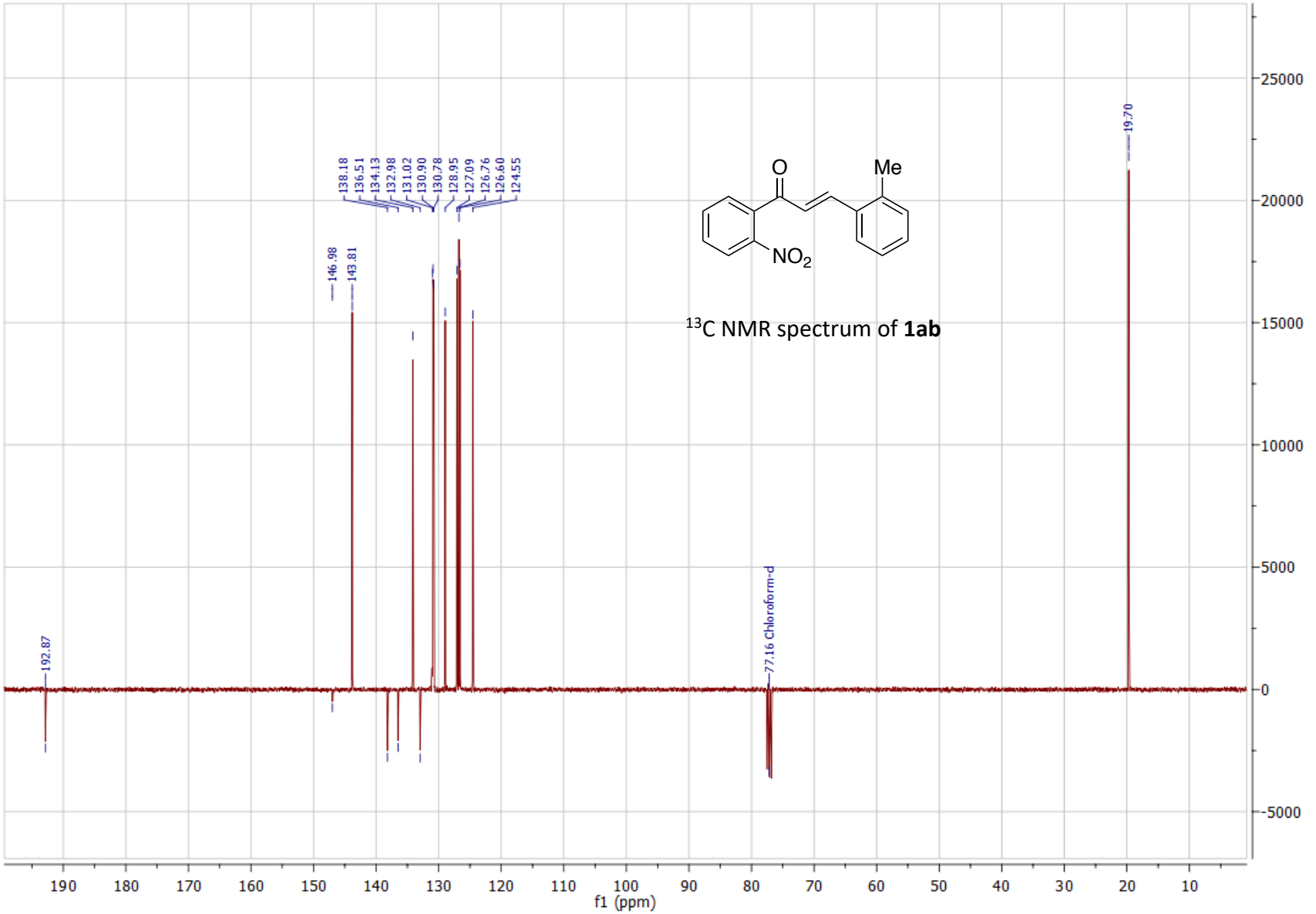
^{1}H NMR spectrum of **1aa**



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



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



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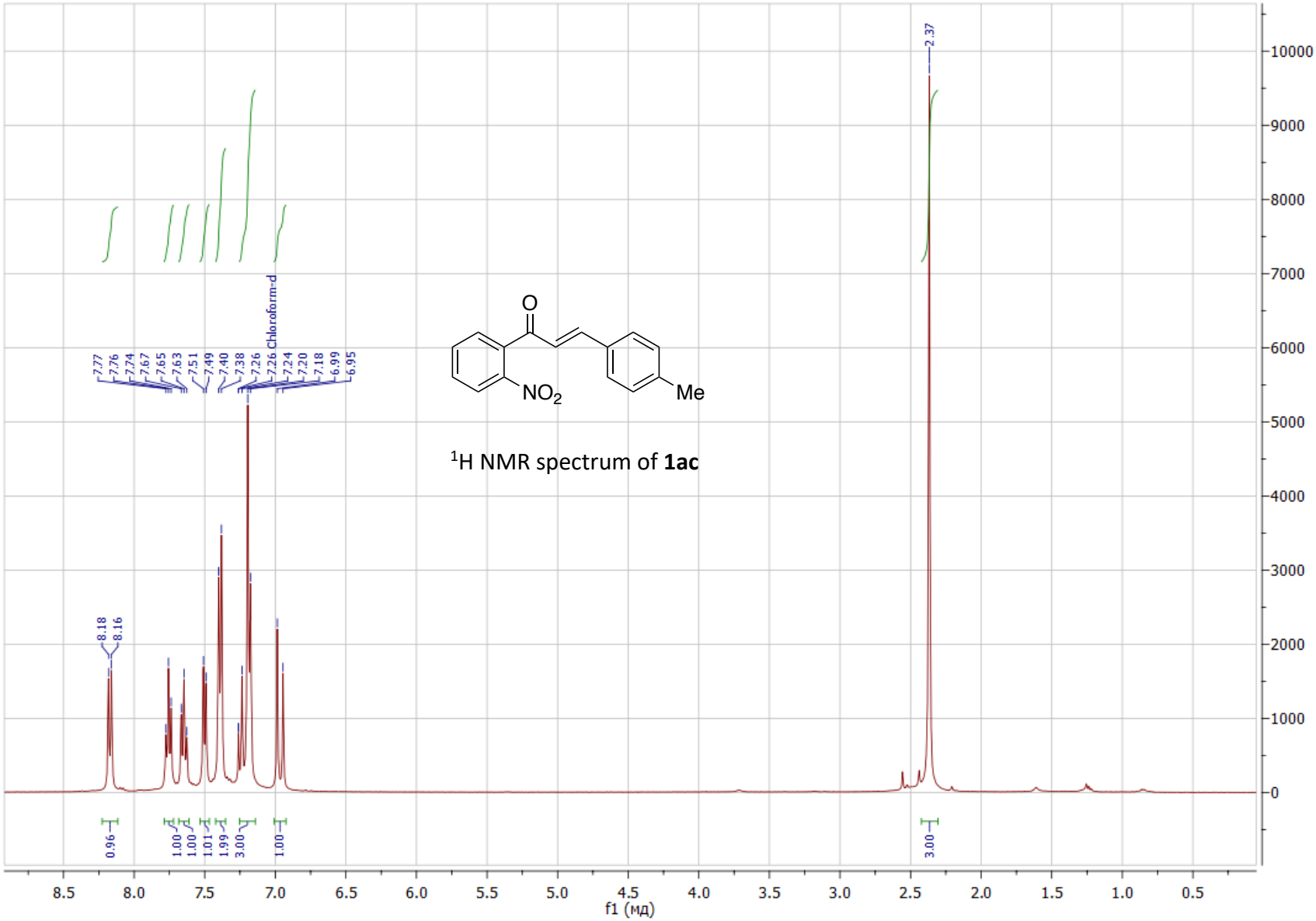

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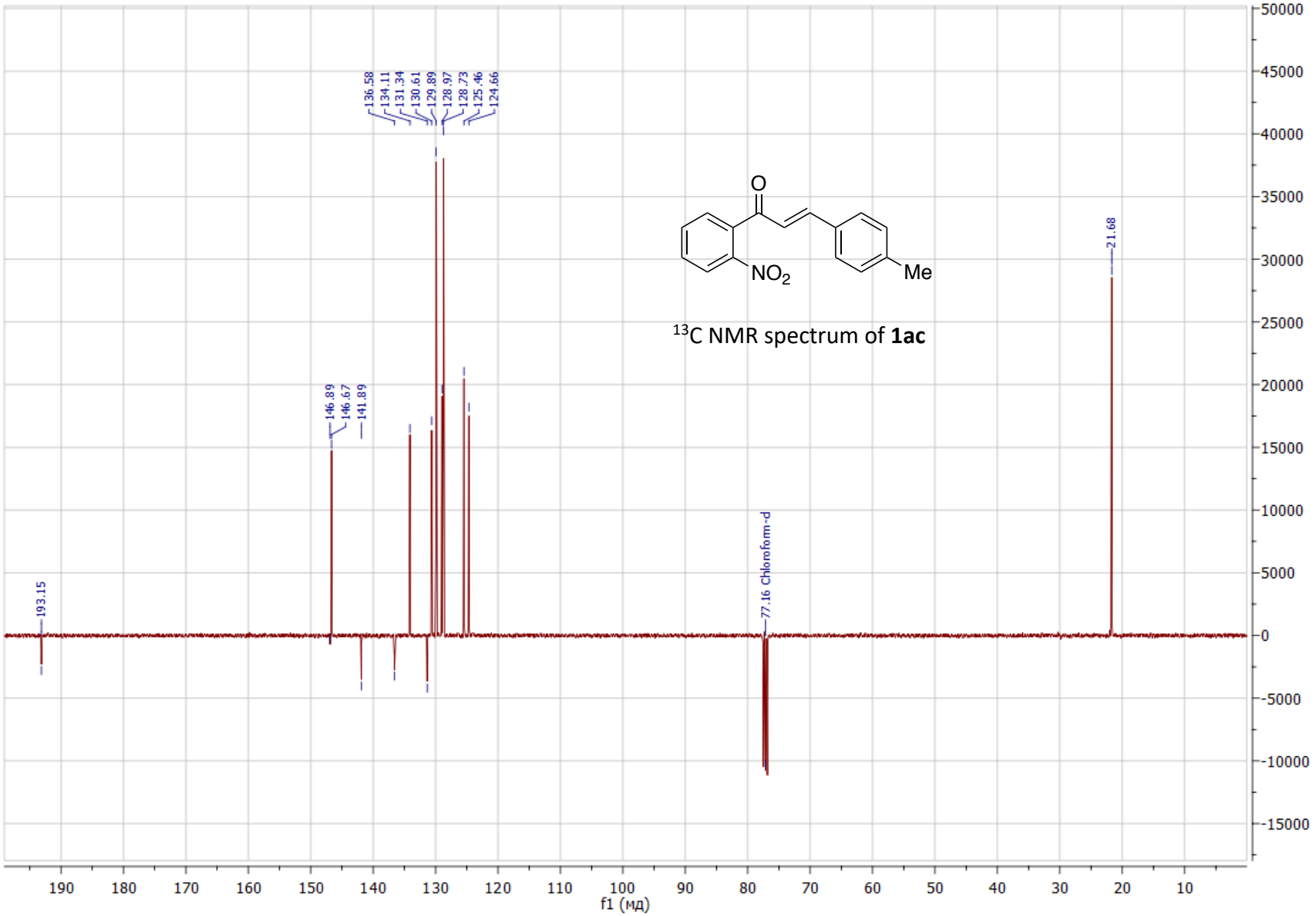

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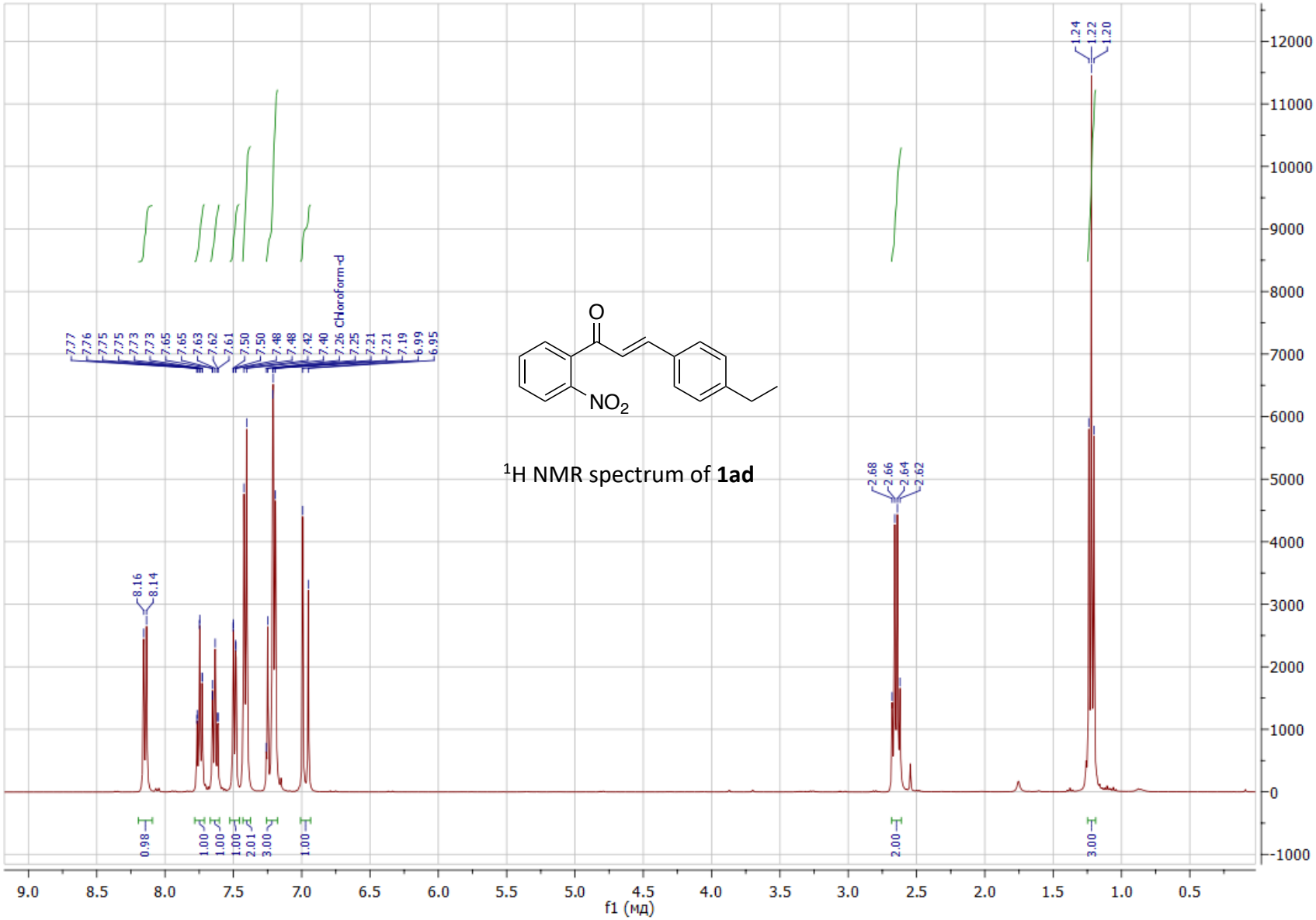

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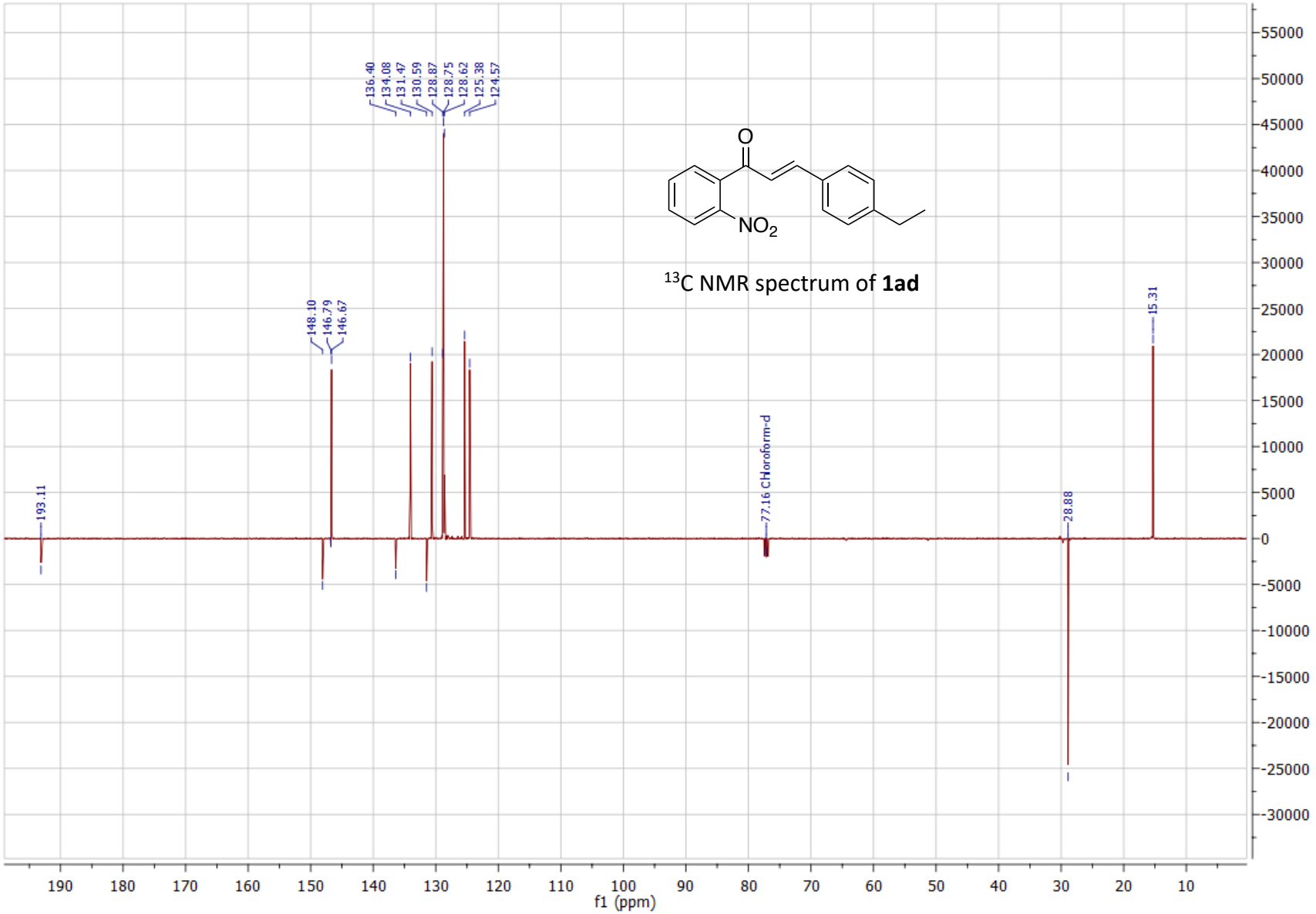

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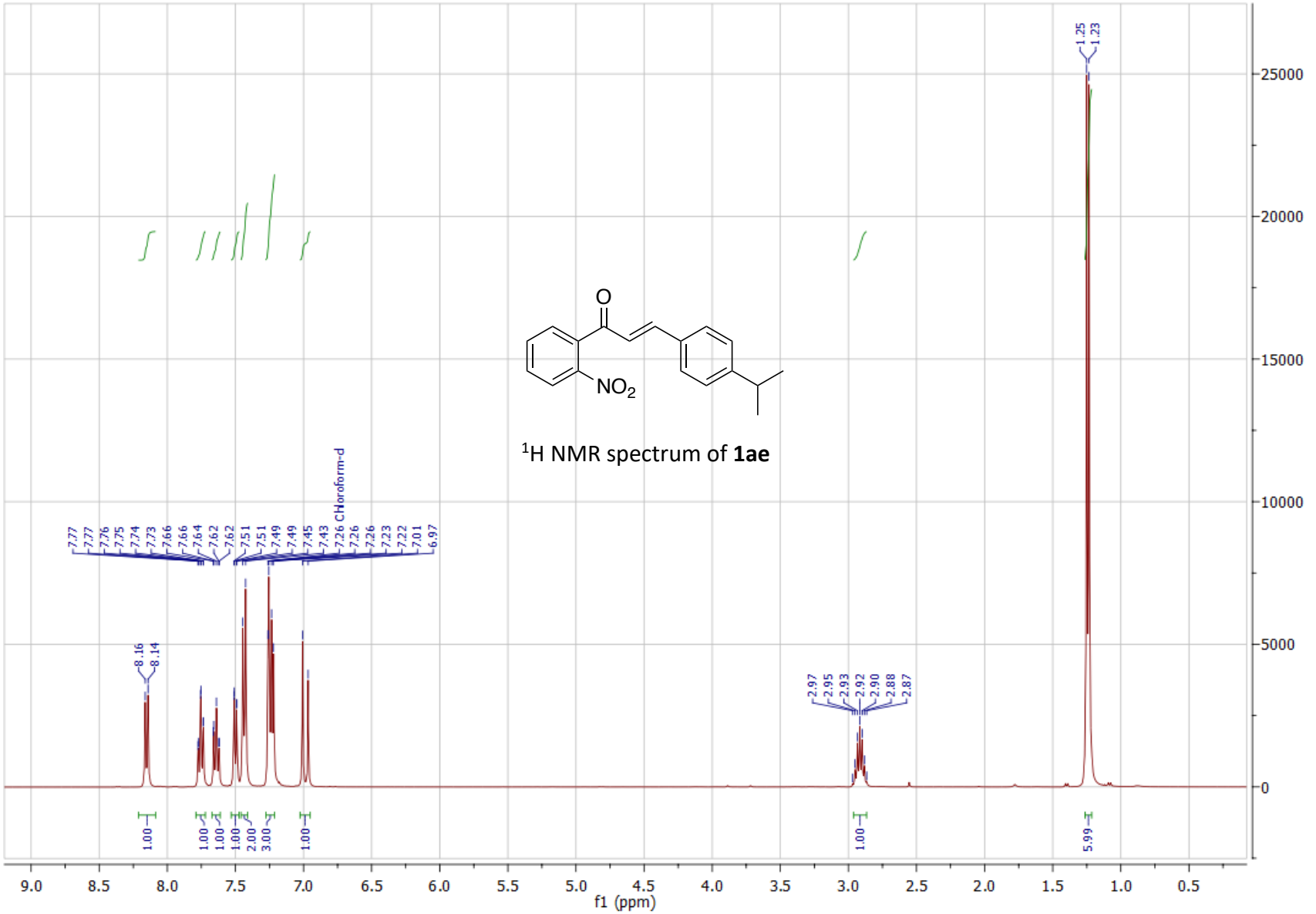

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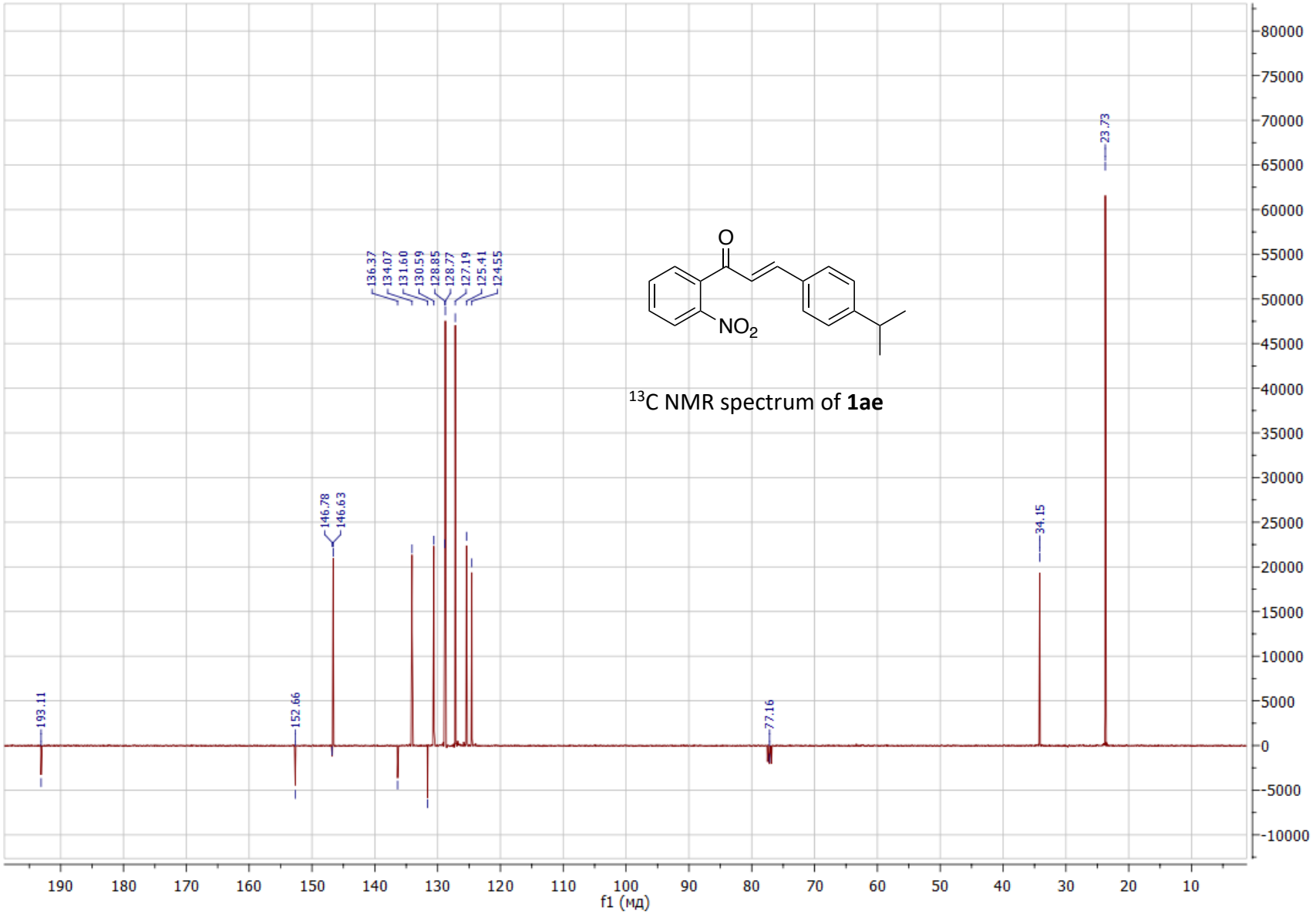

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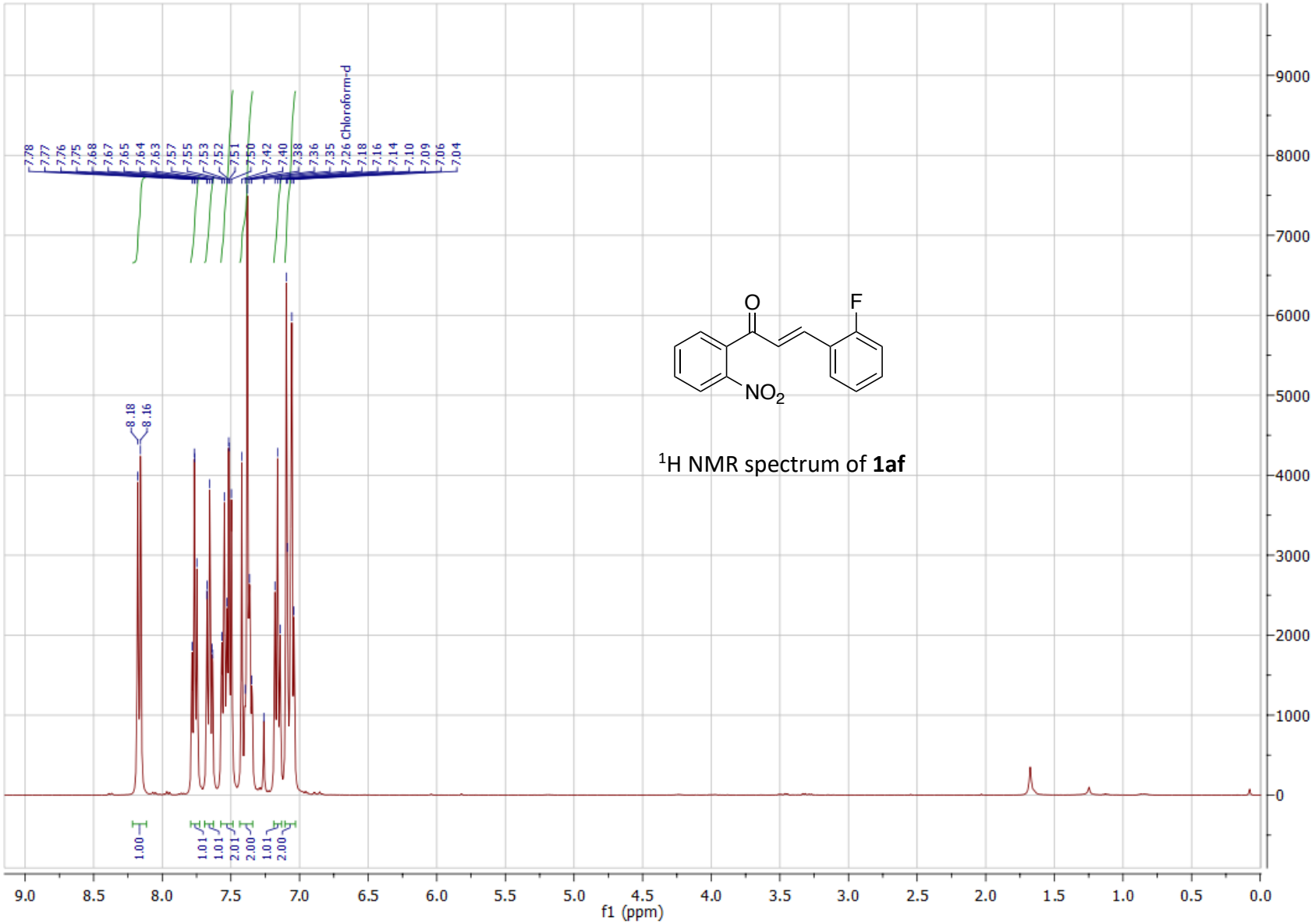

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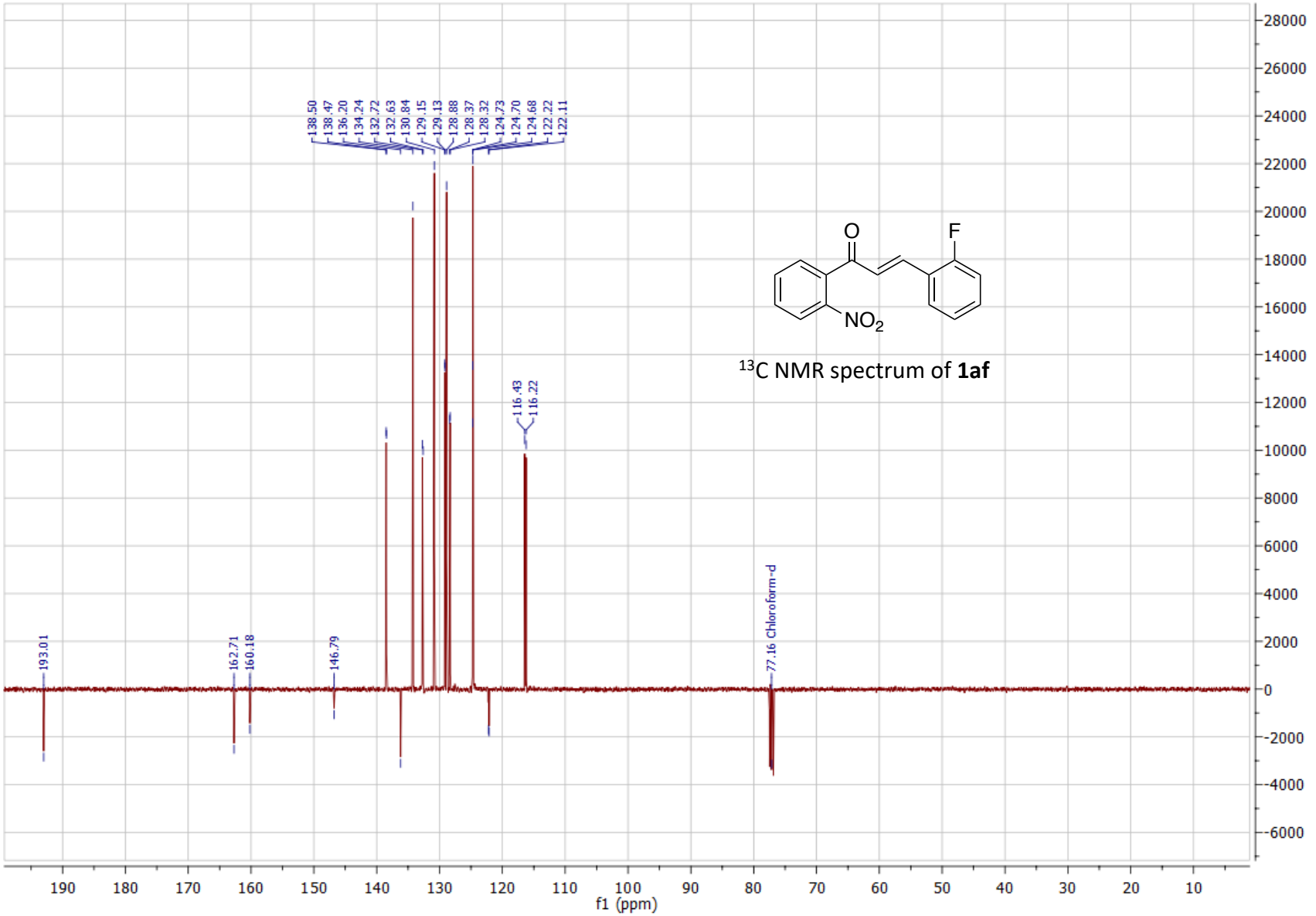

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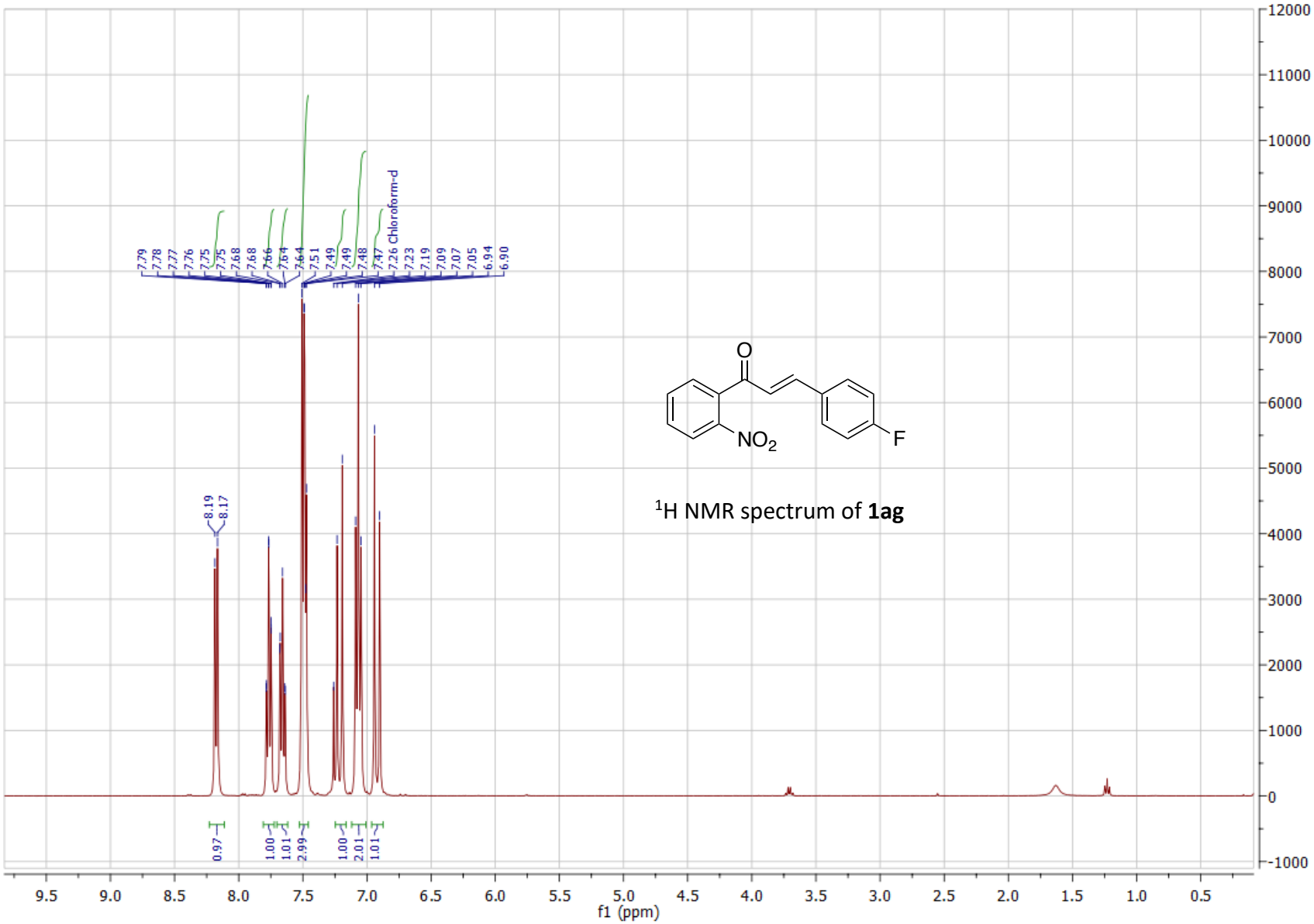

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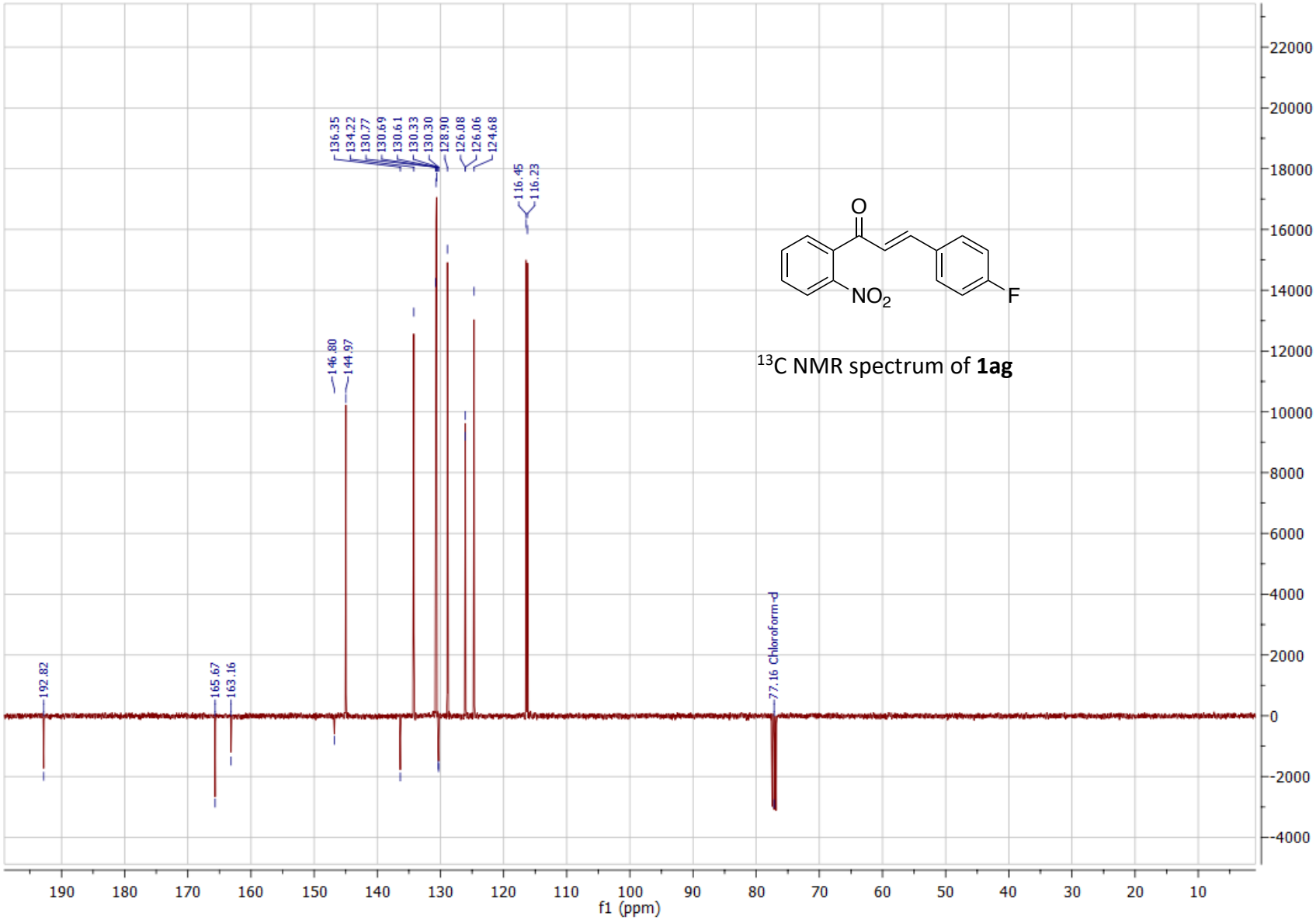

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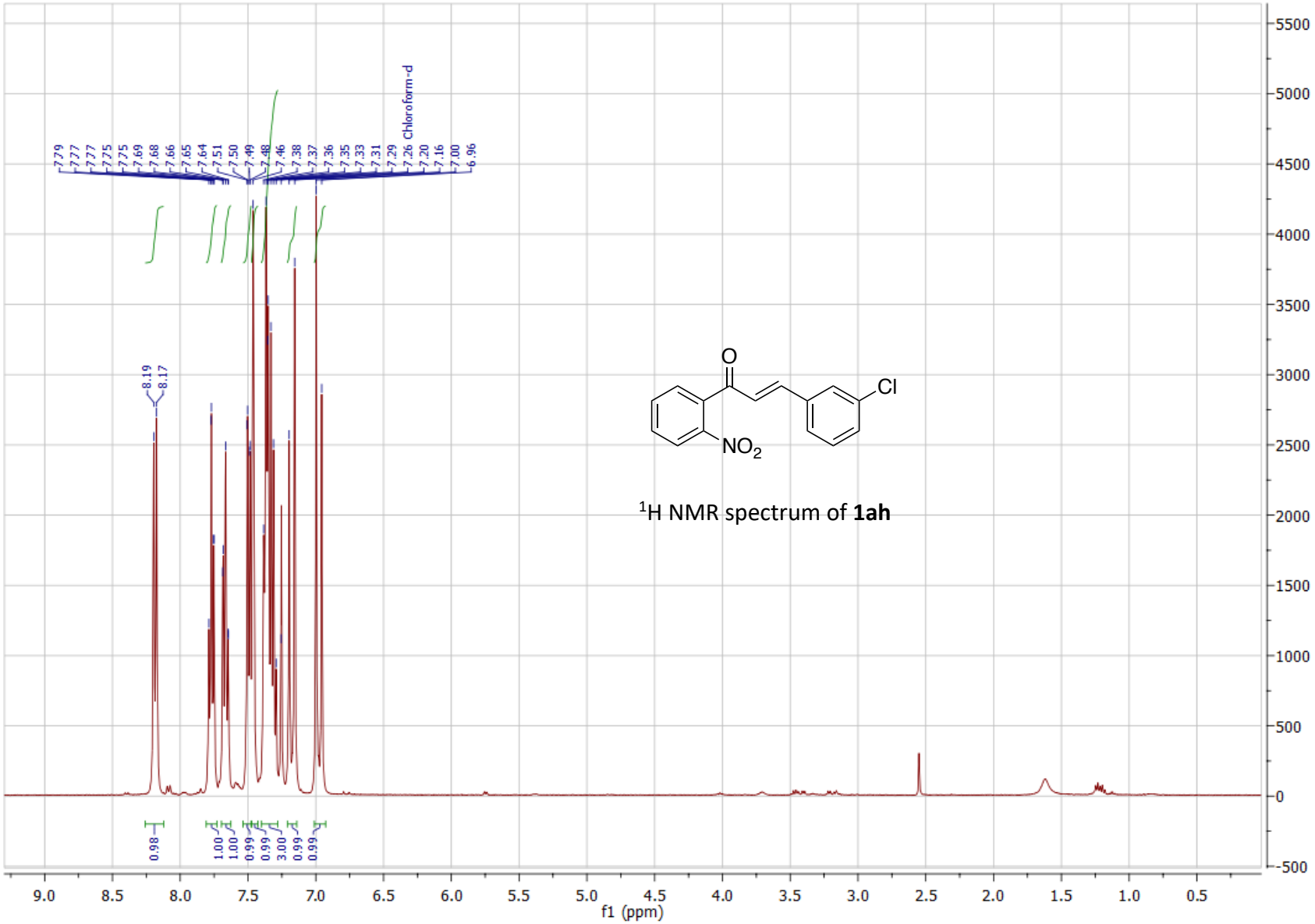

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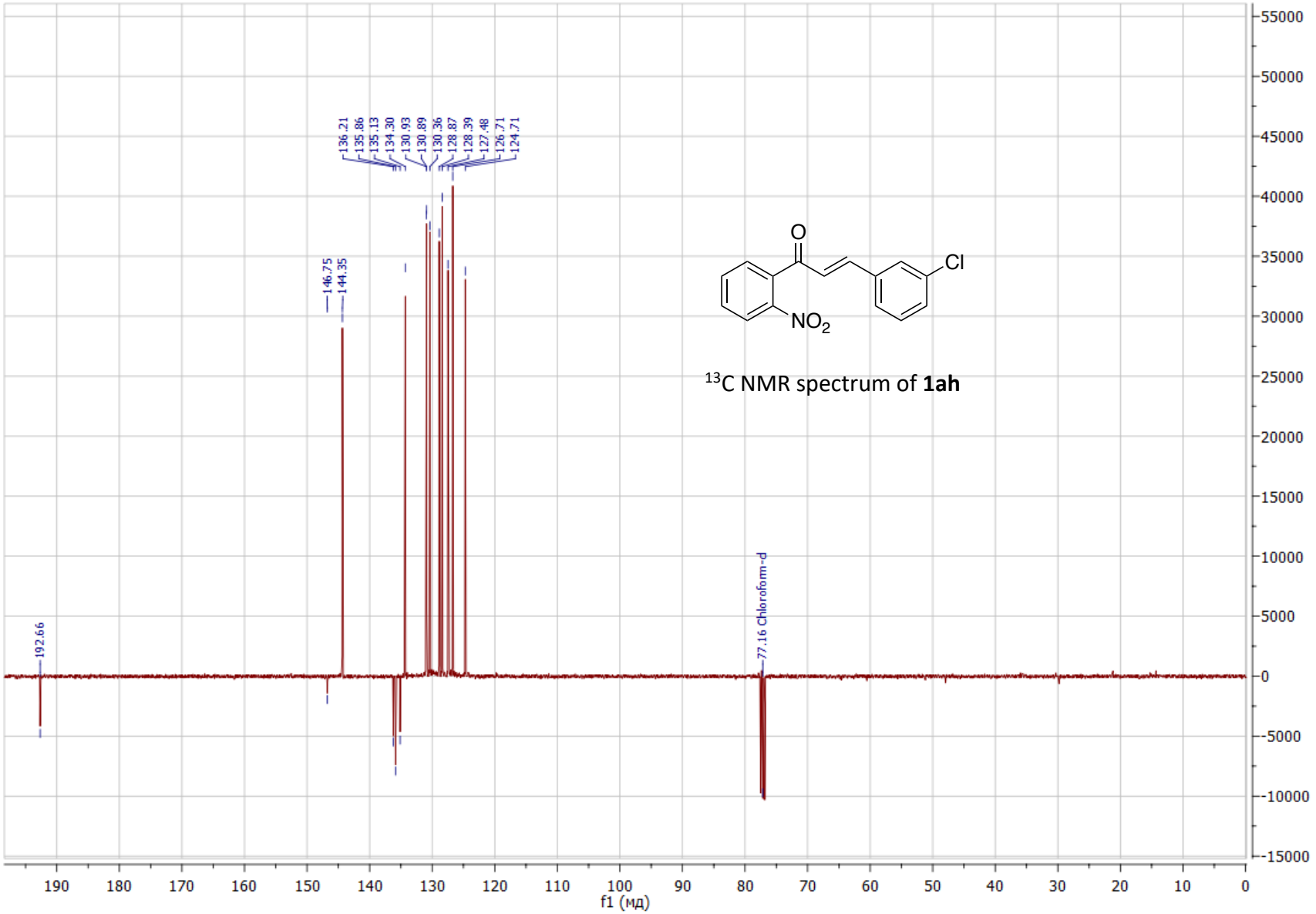

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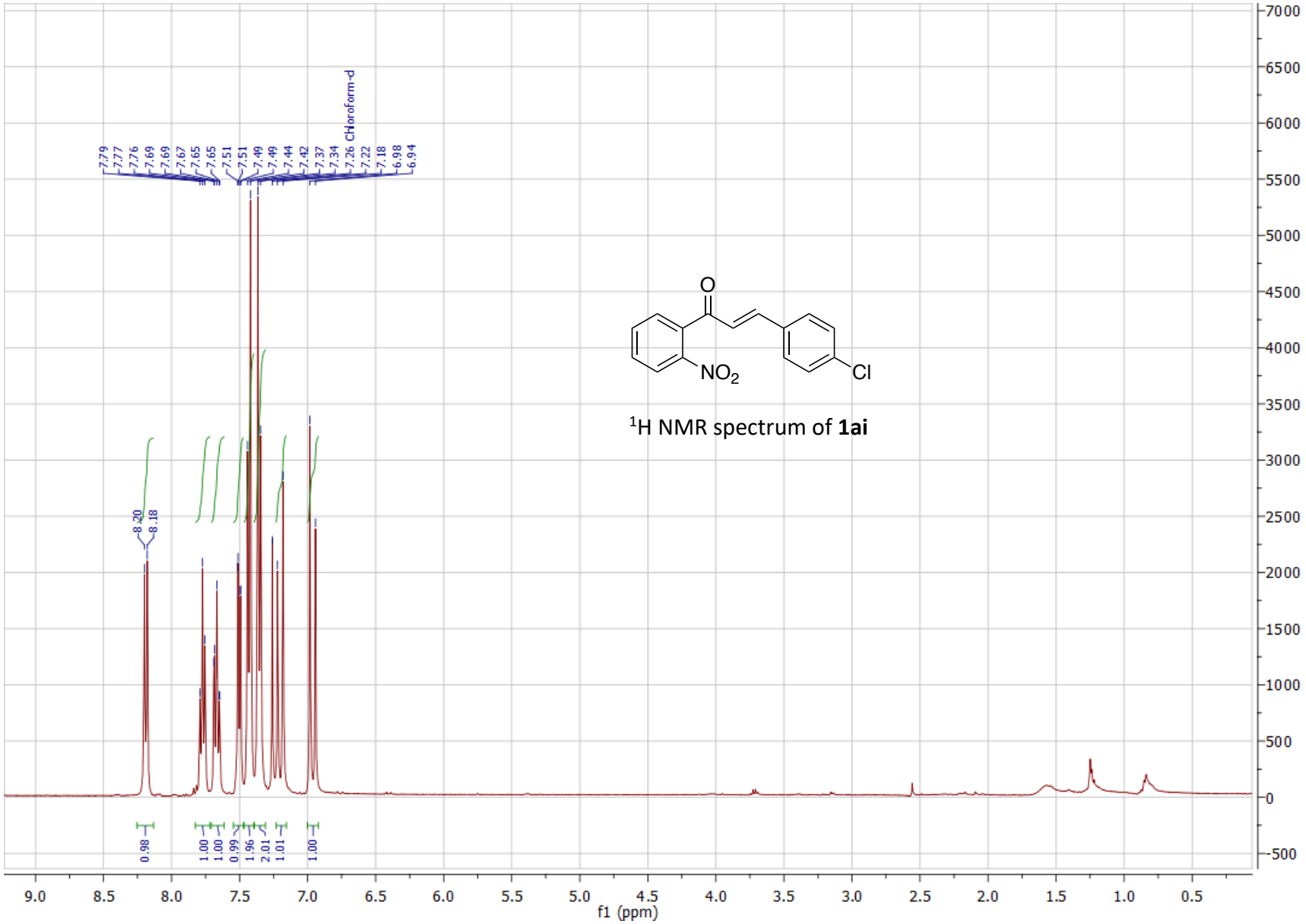

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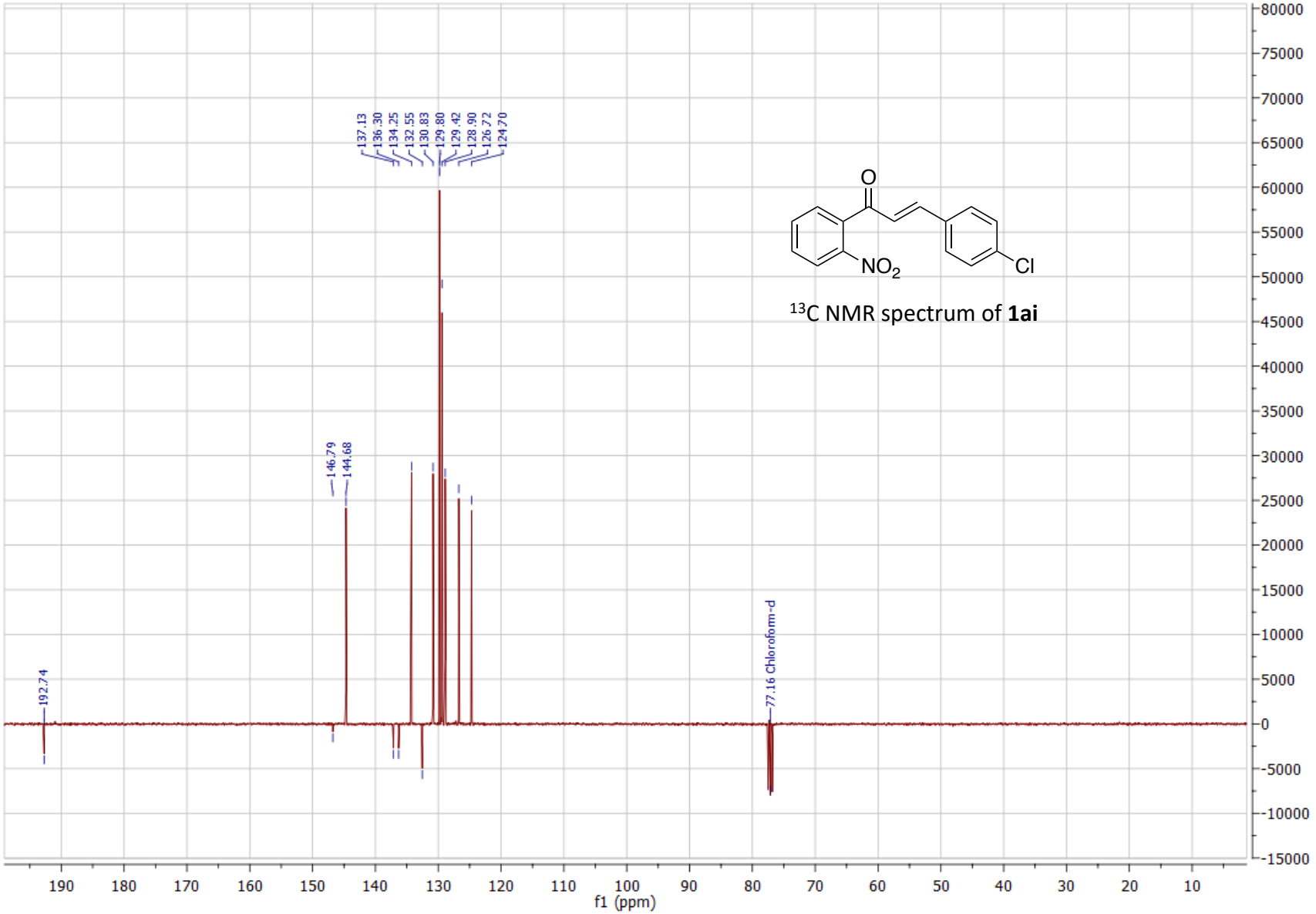

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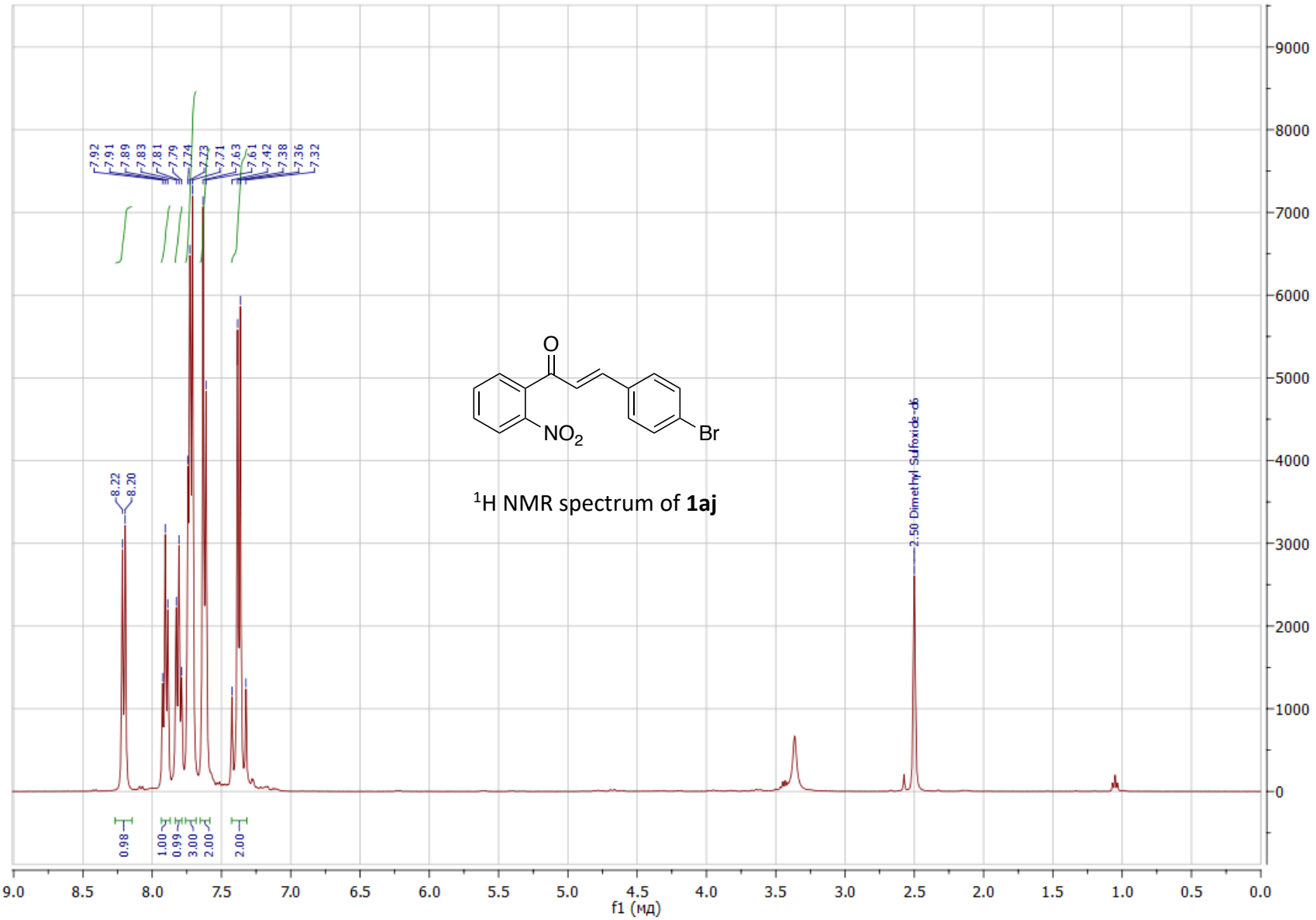

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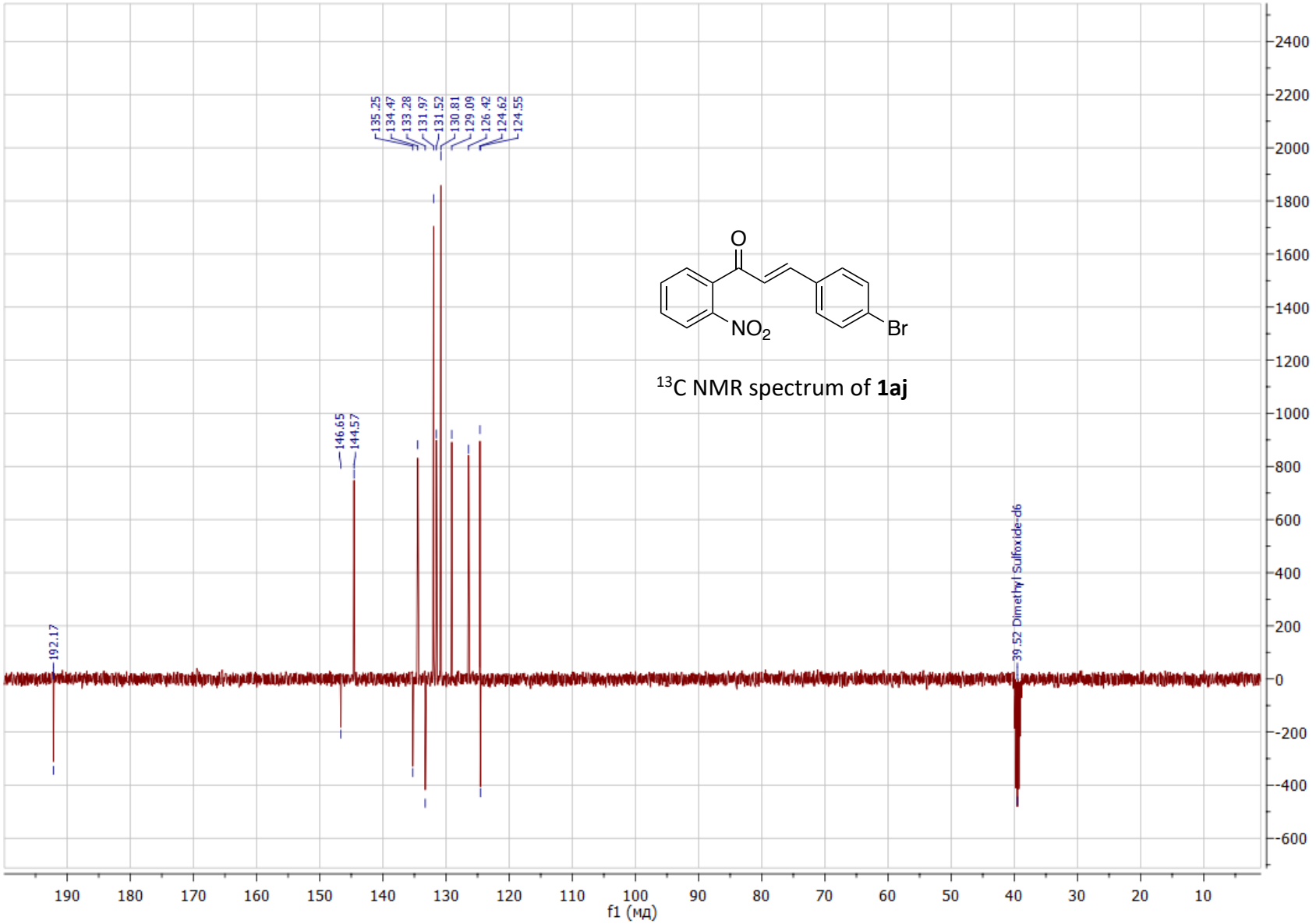

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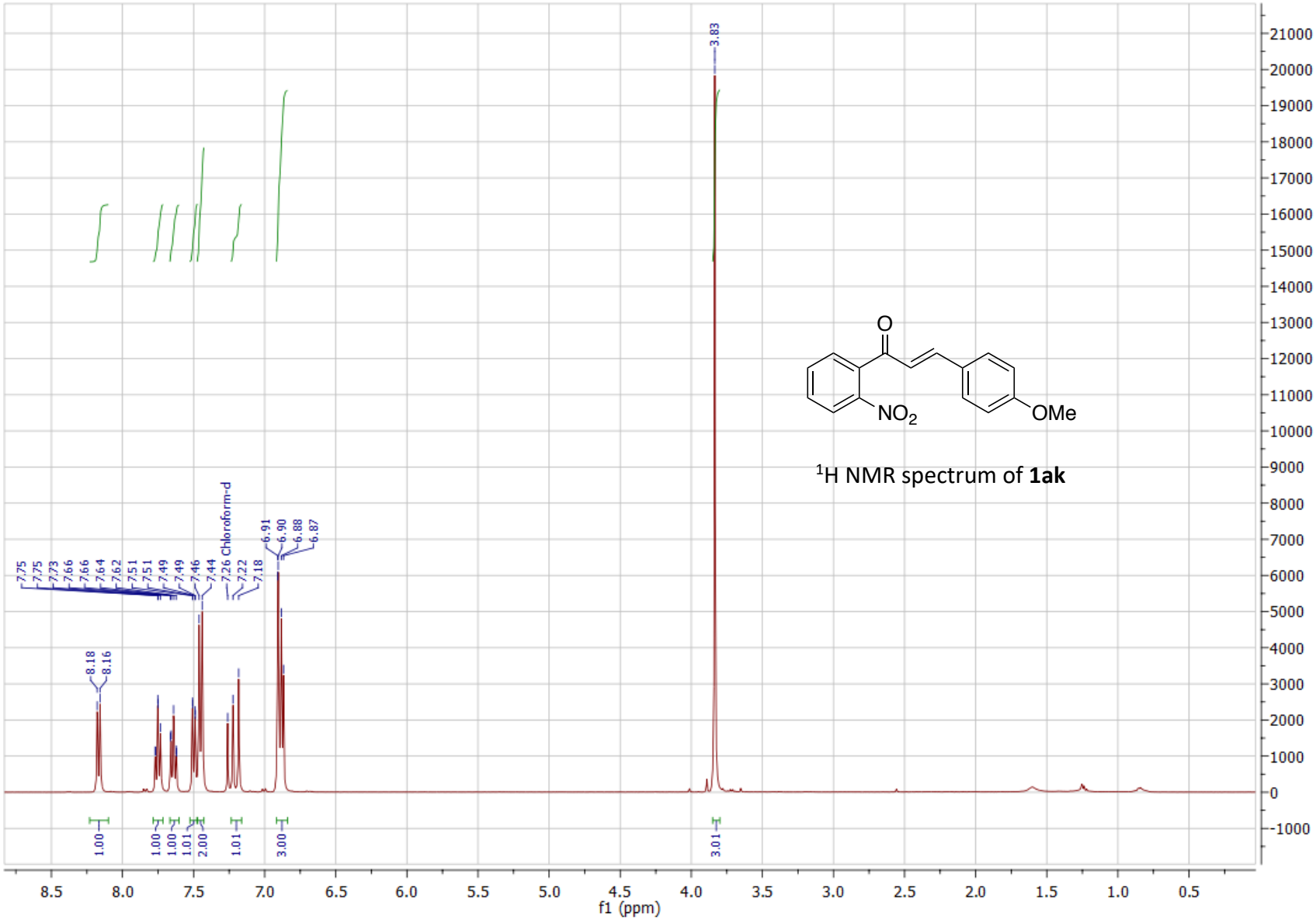

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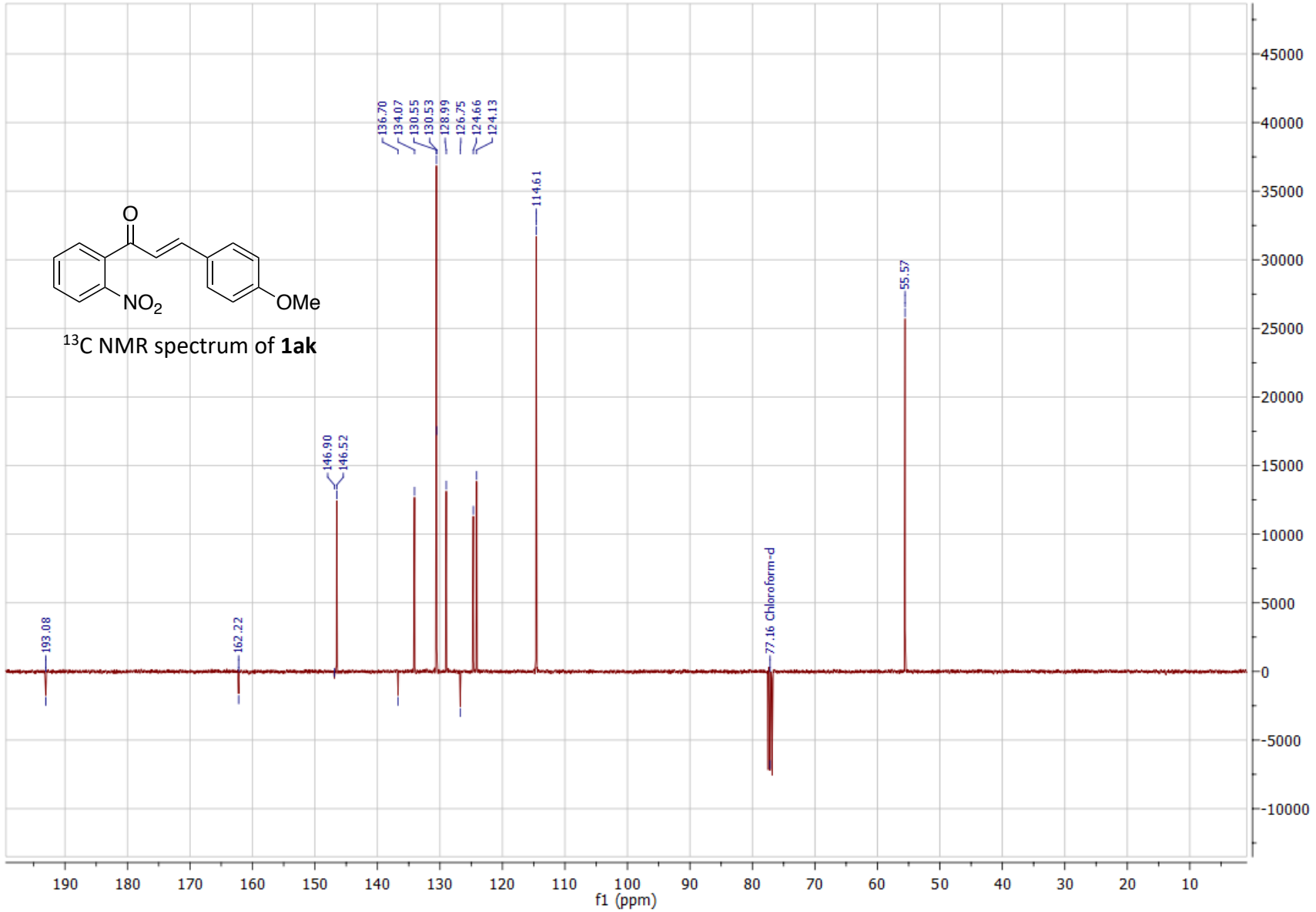

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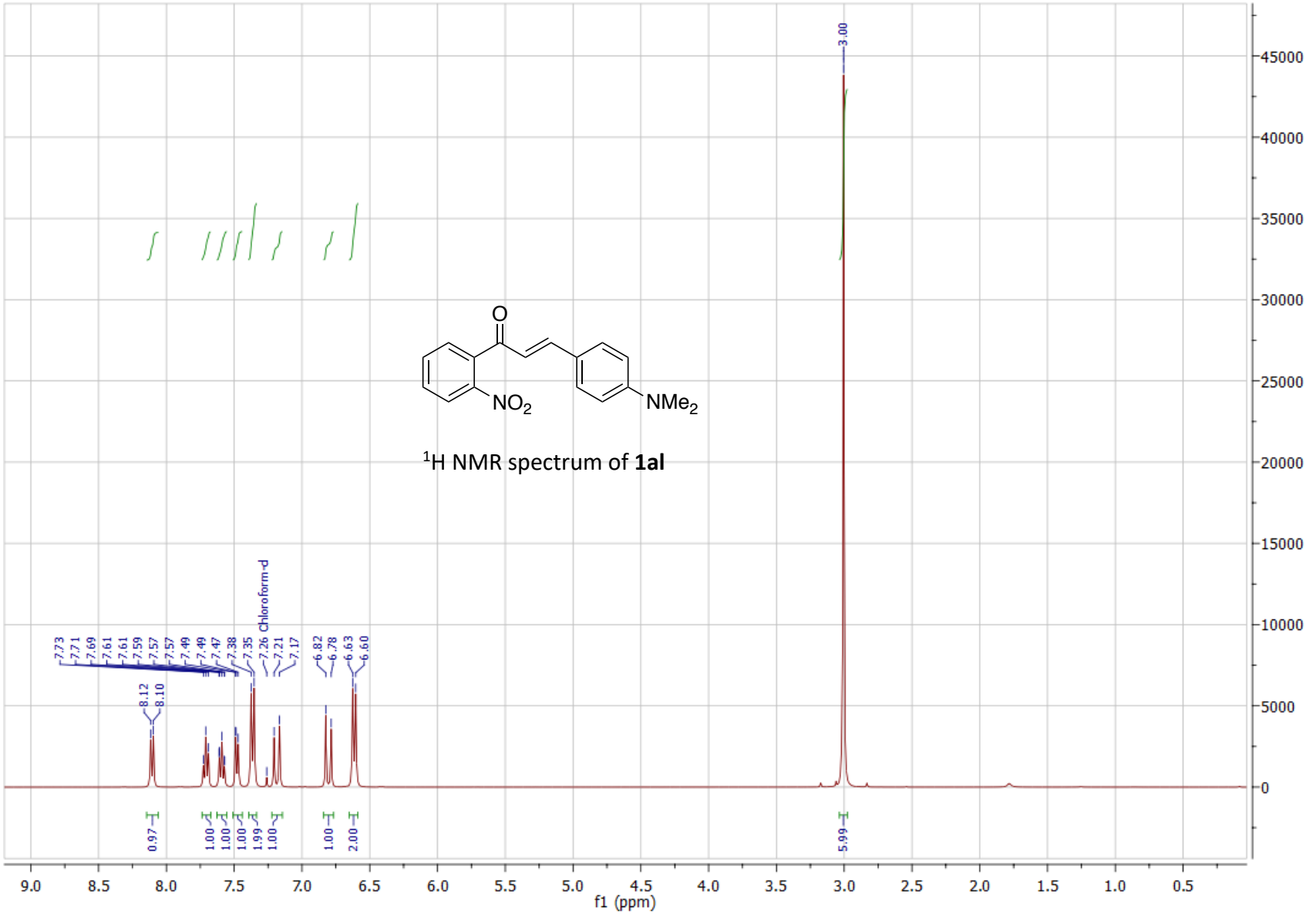

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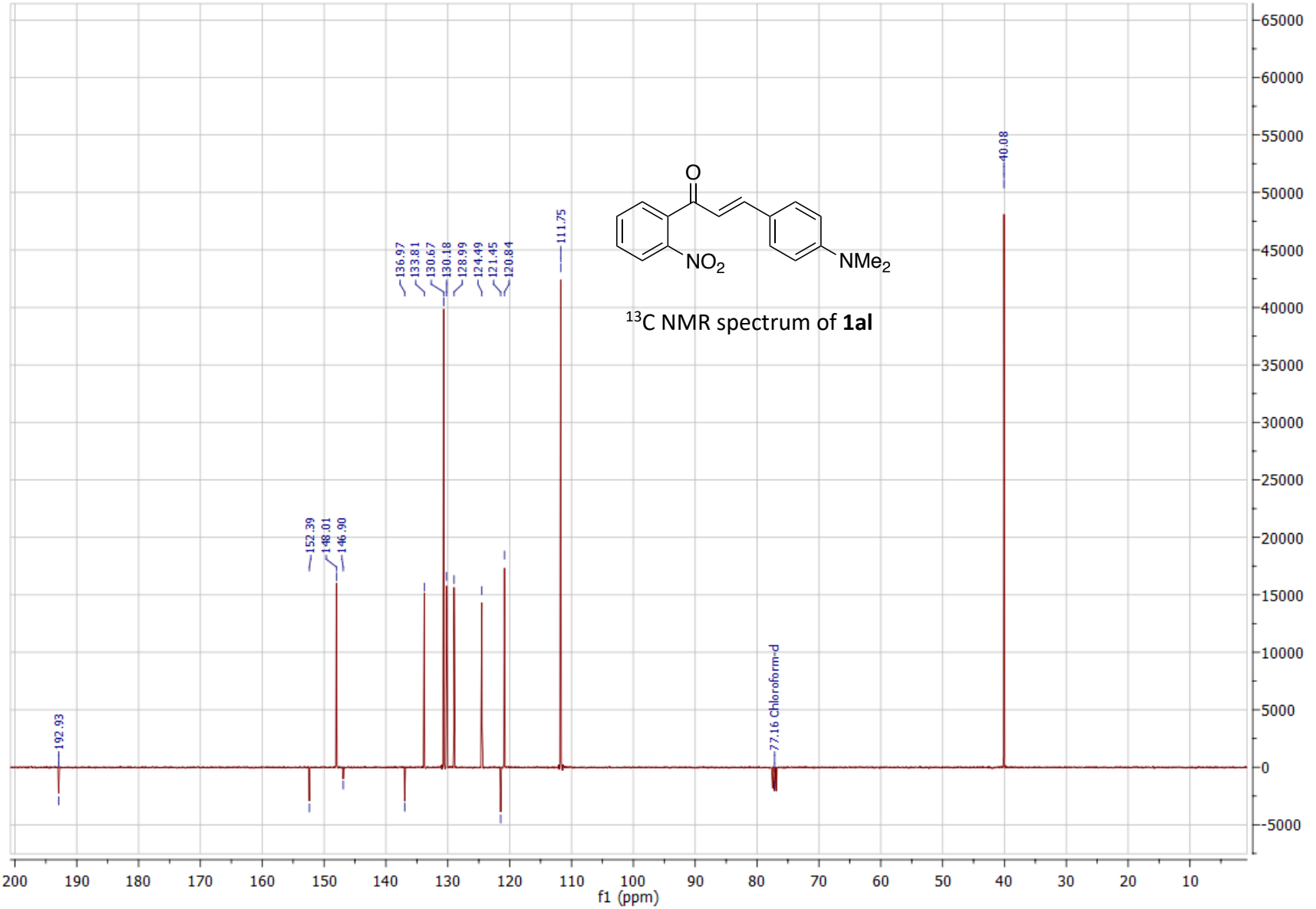

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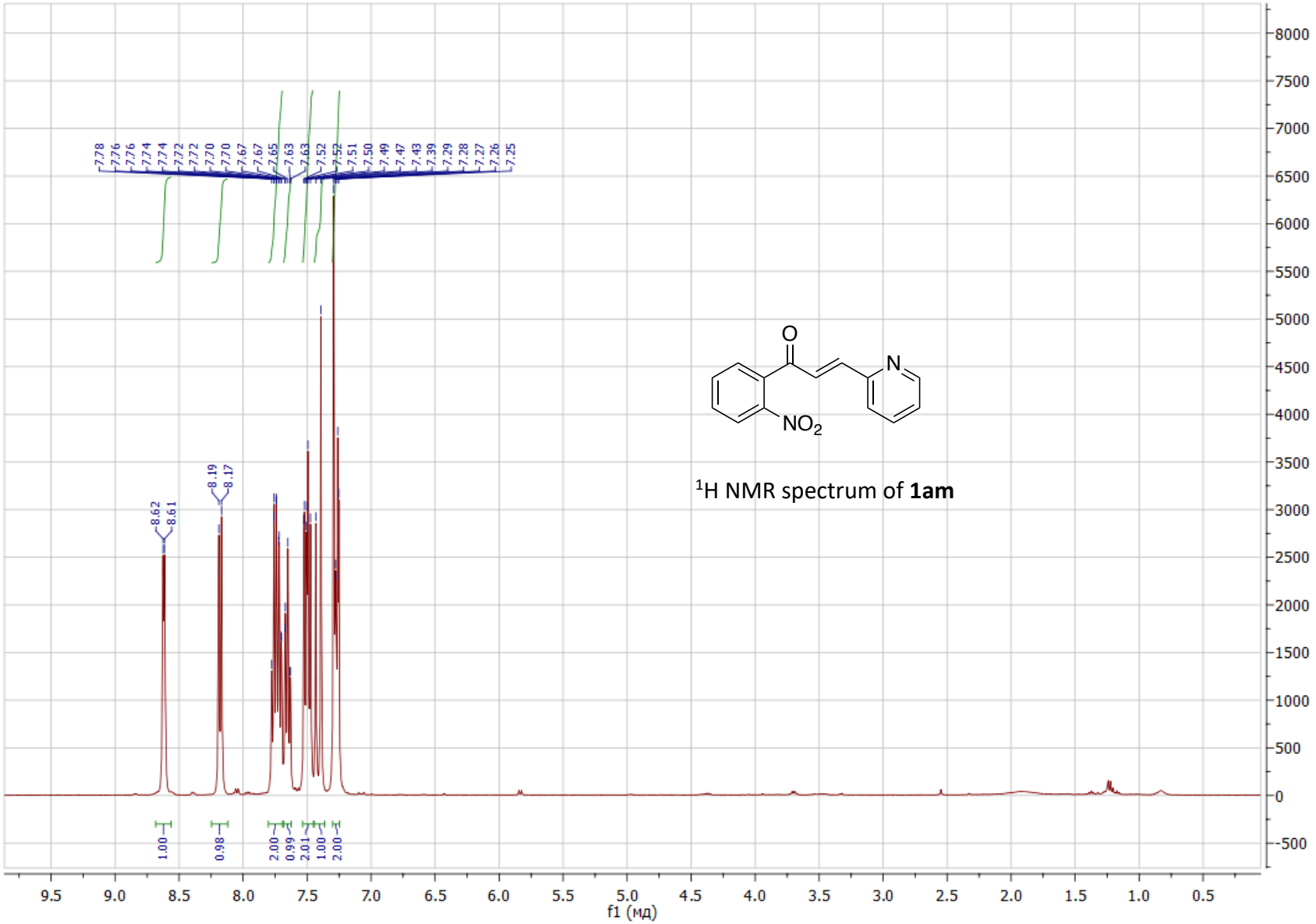

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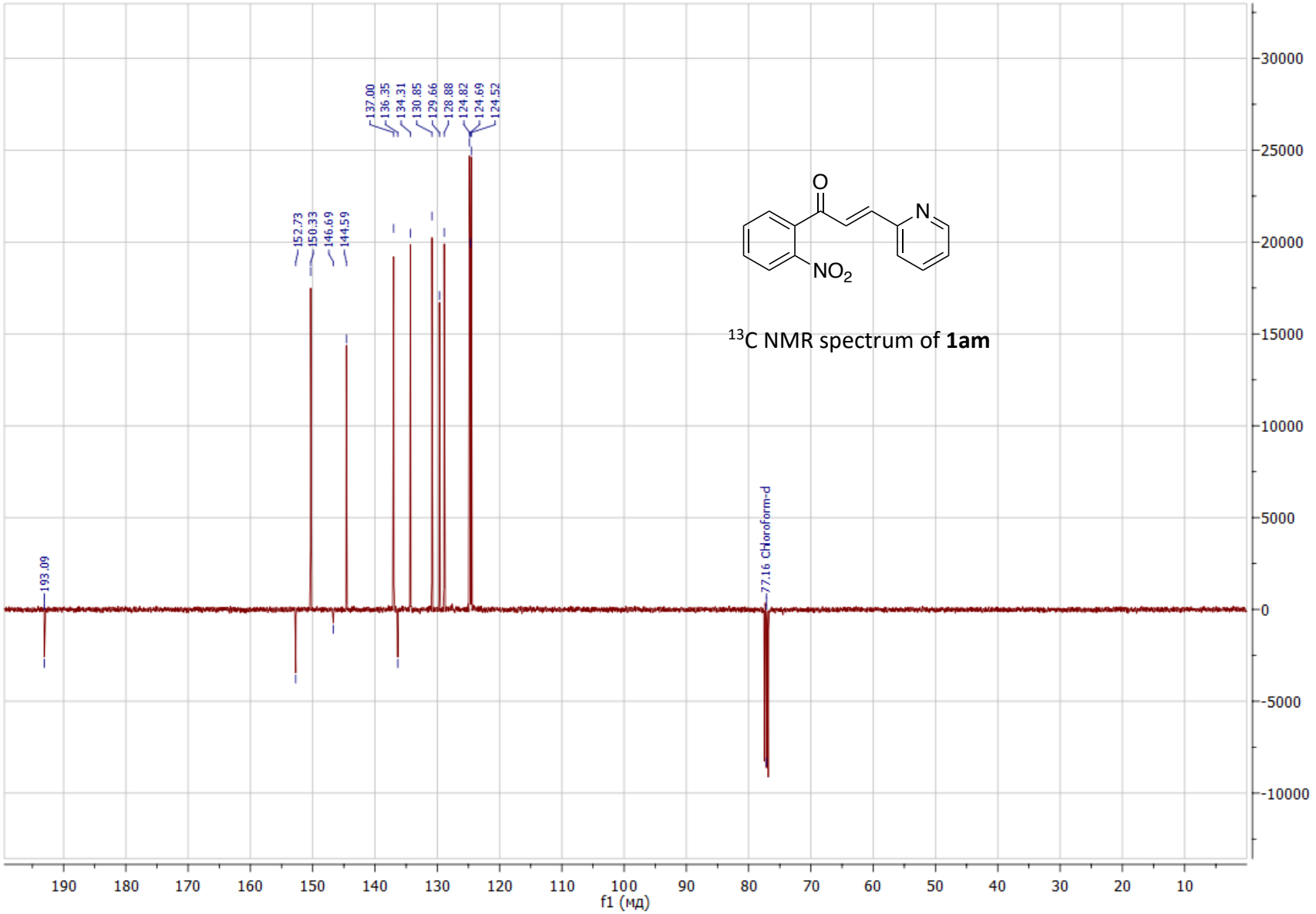

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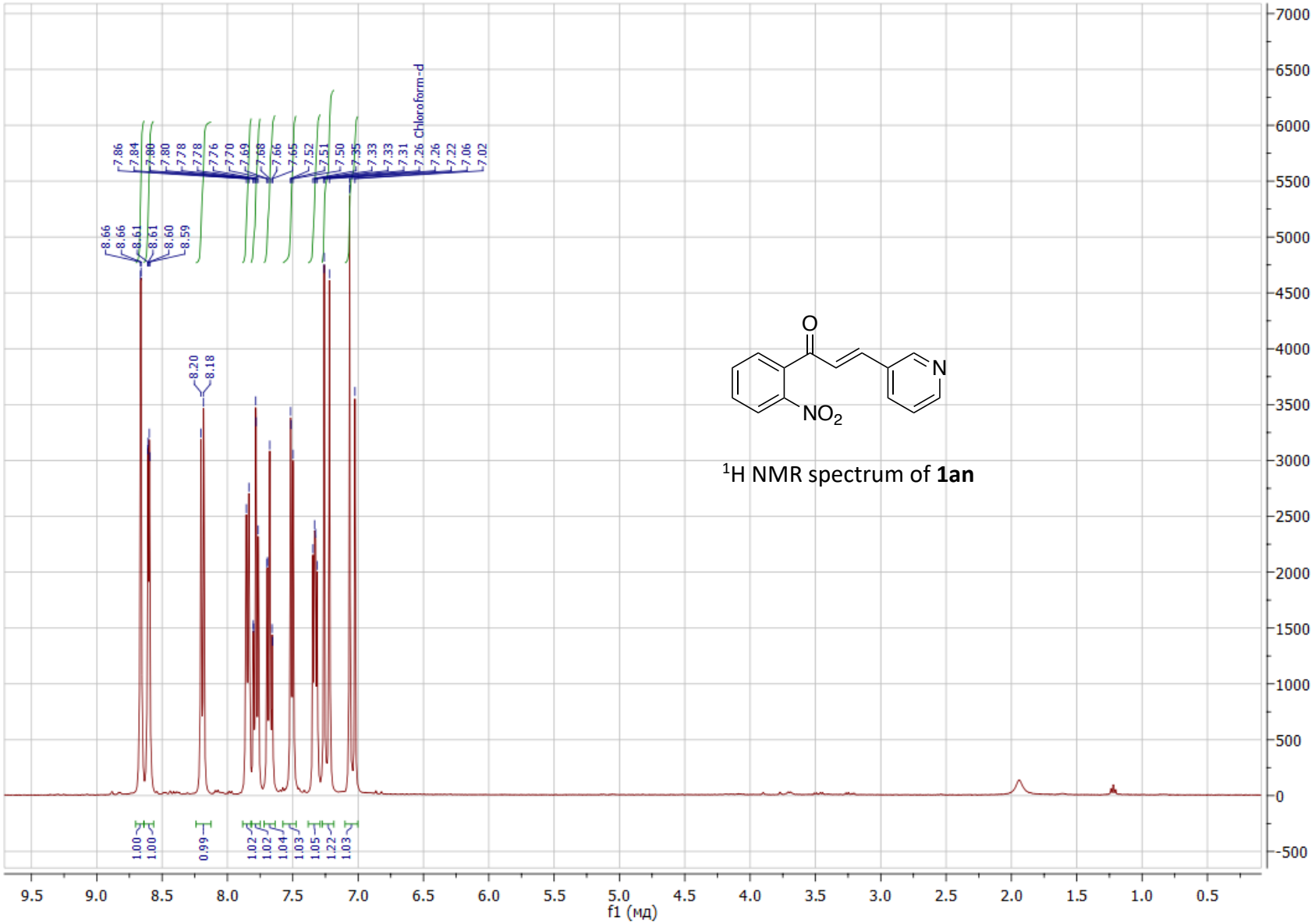

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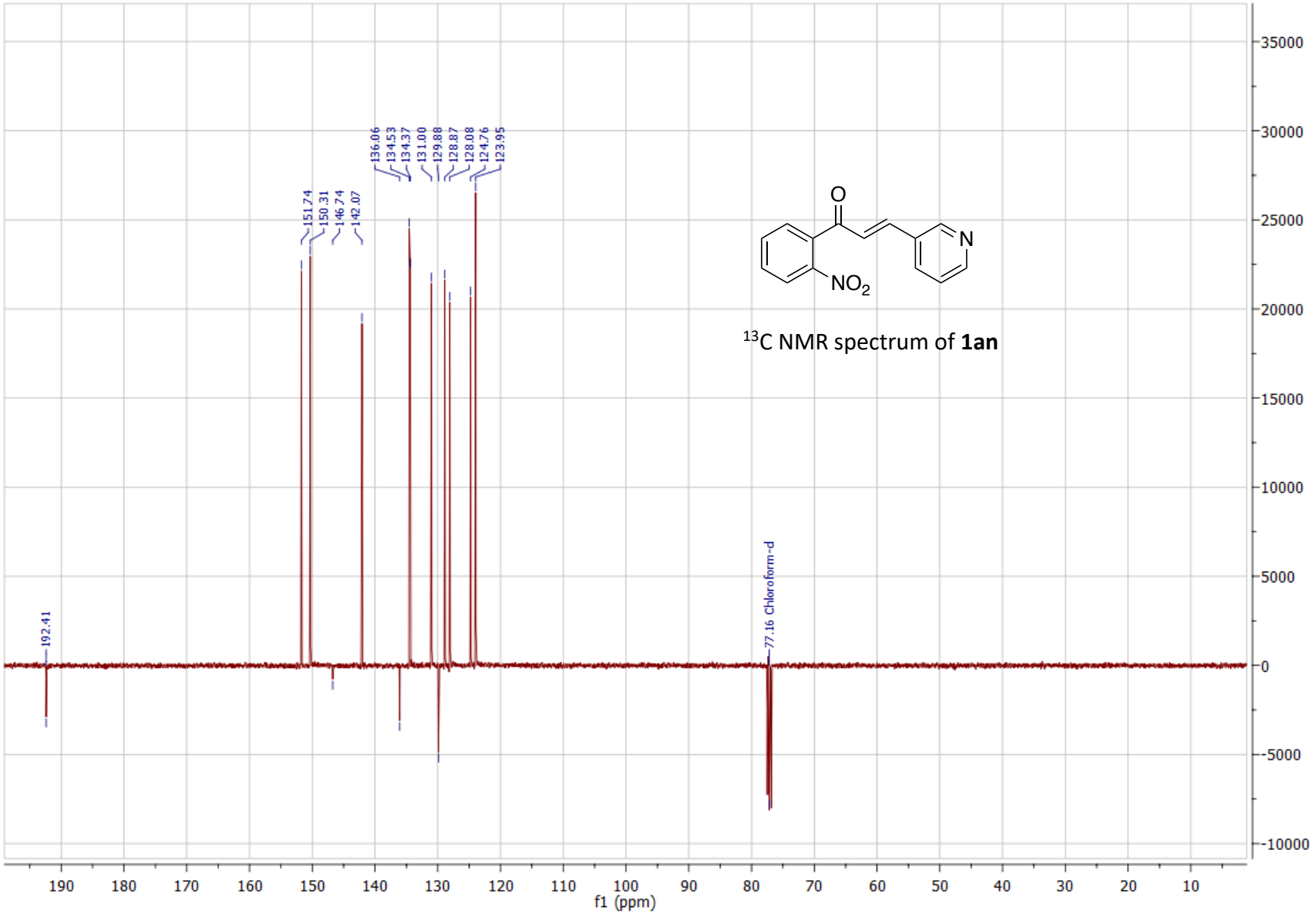

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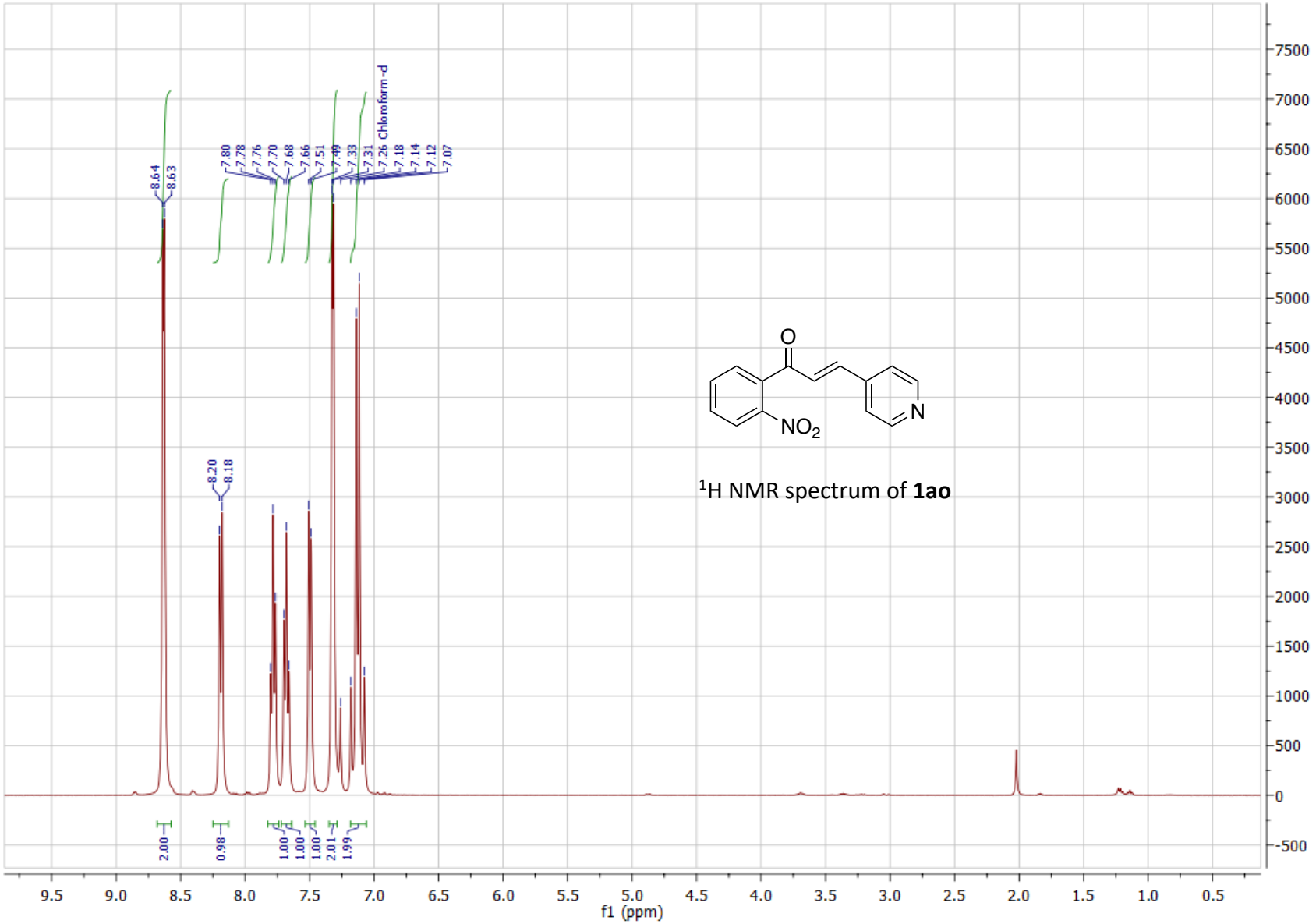

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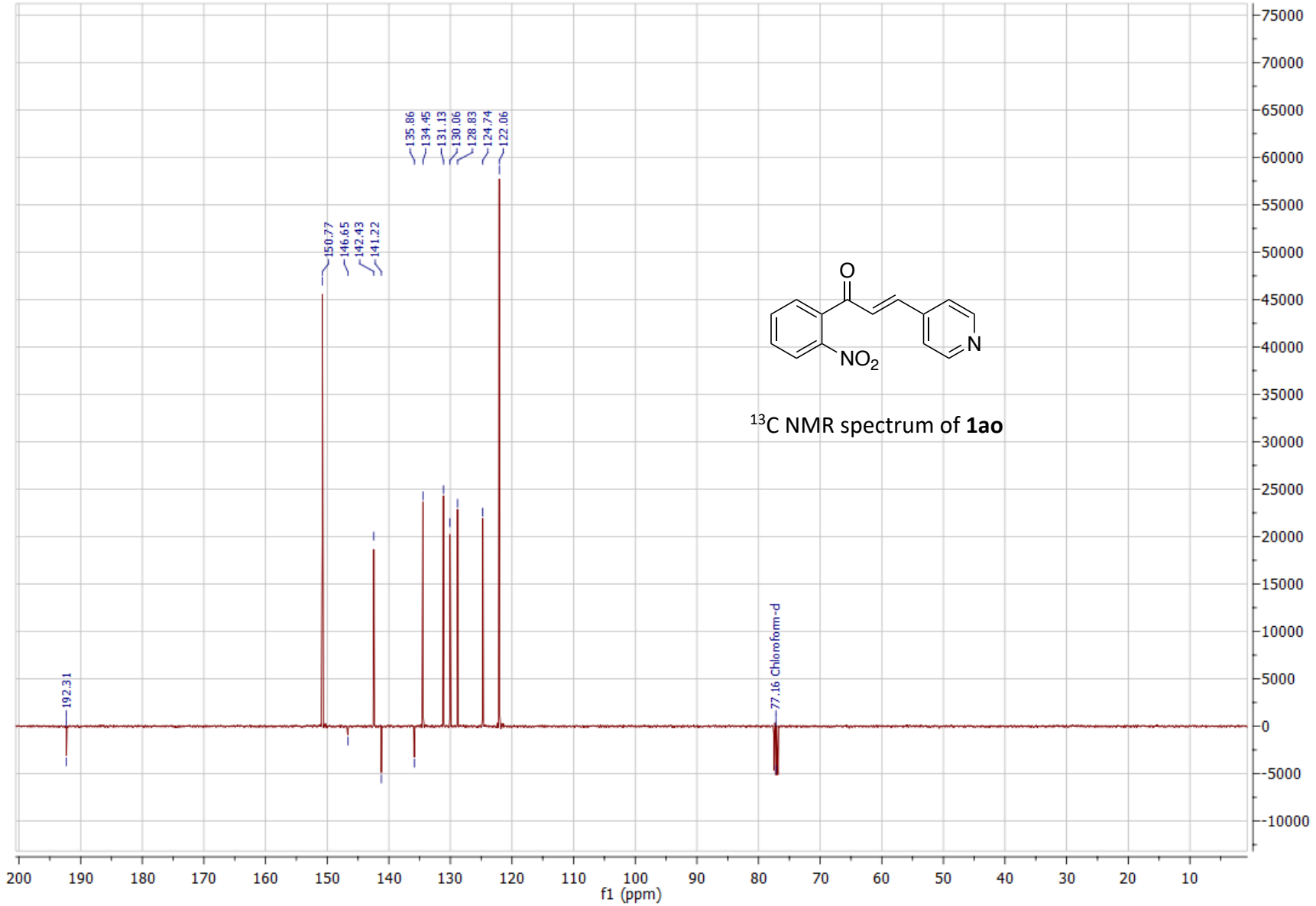
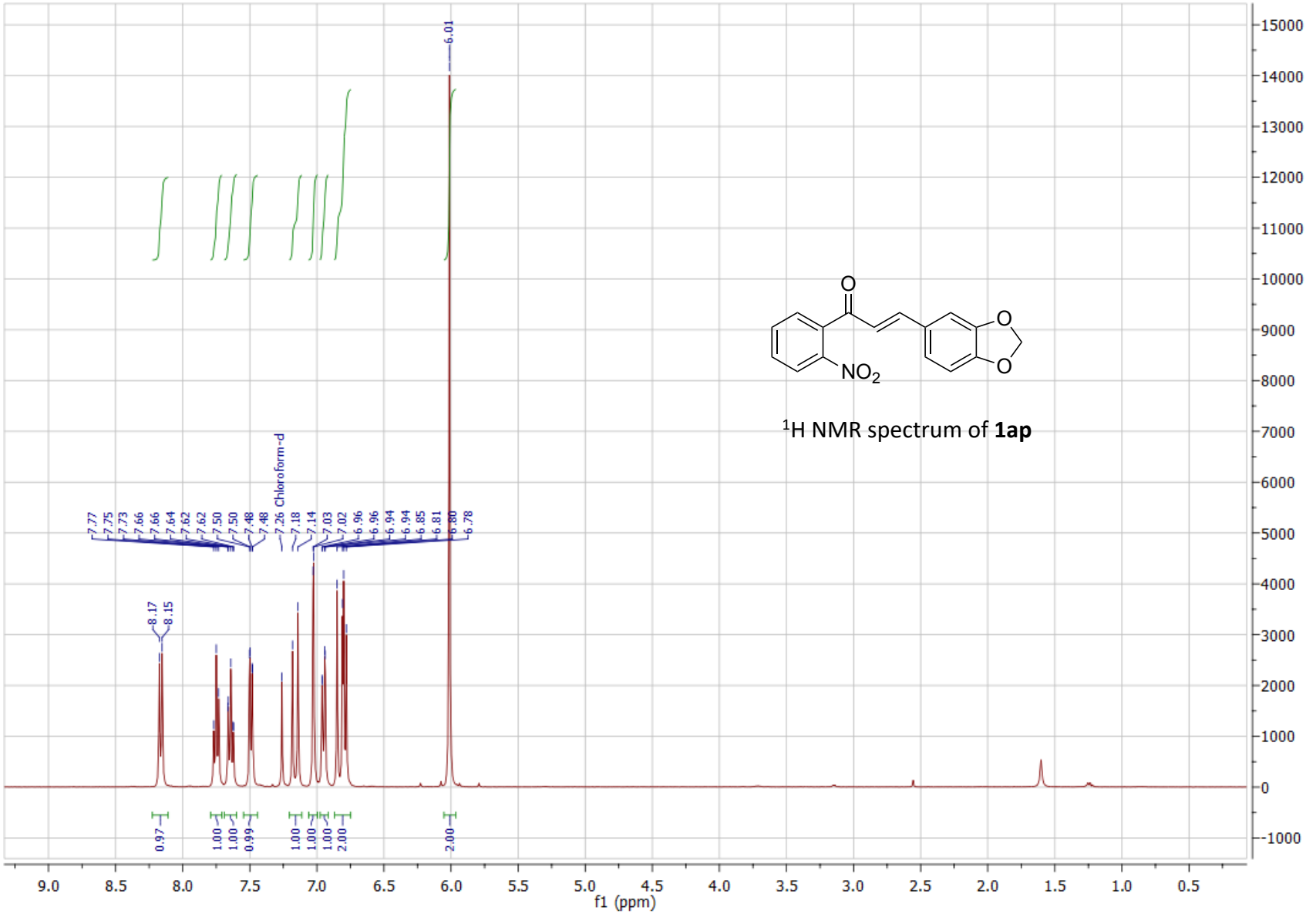


^{13}C NMR spectrum of **1ao**



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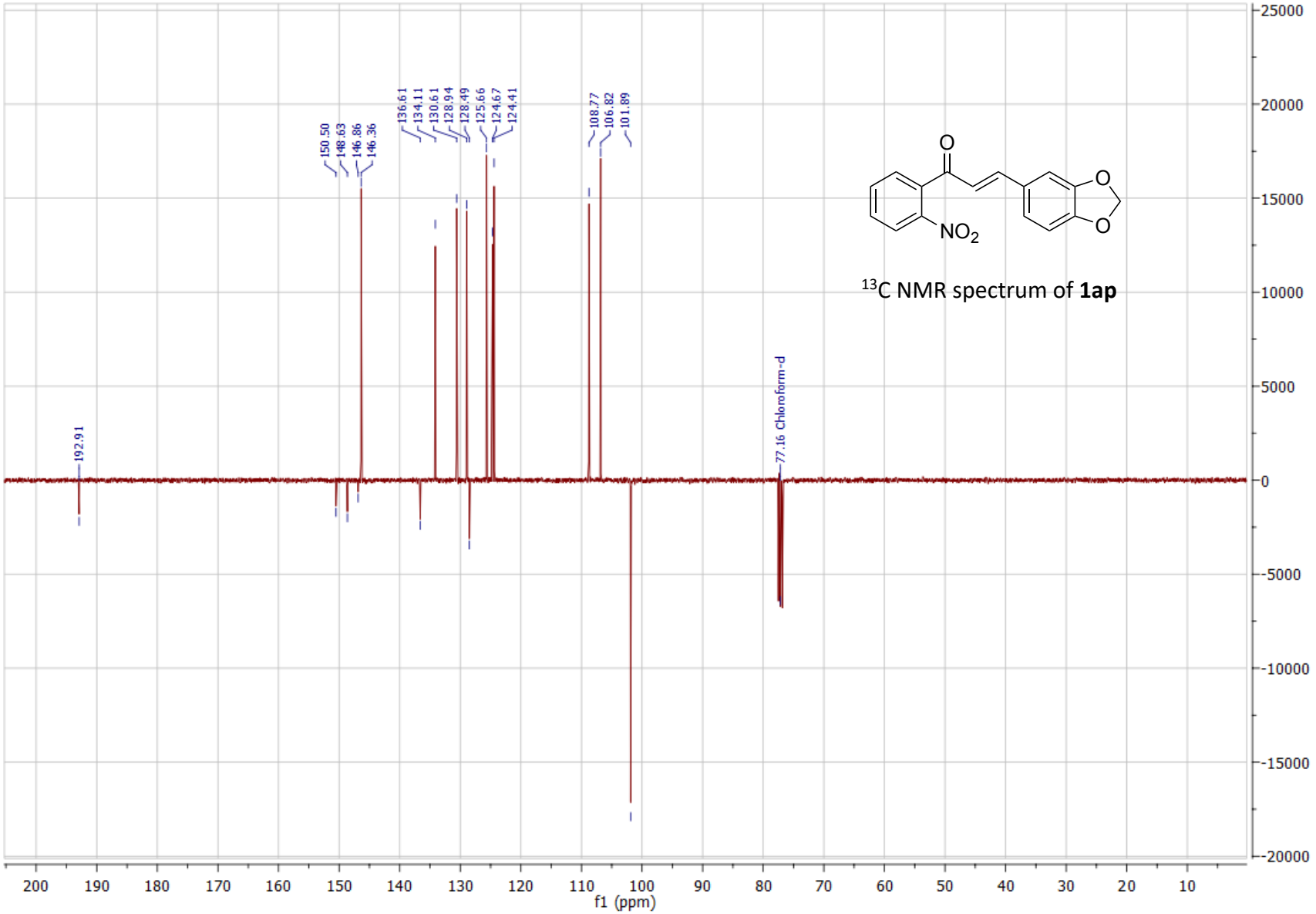
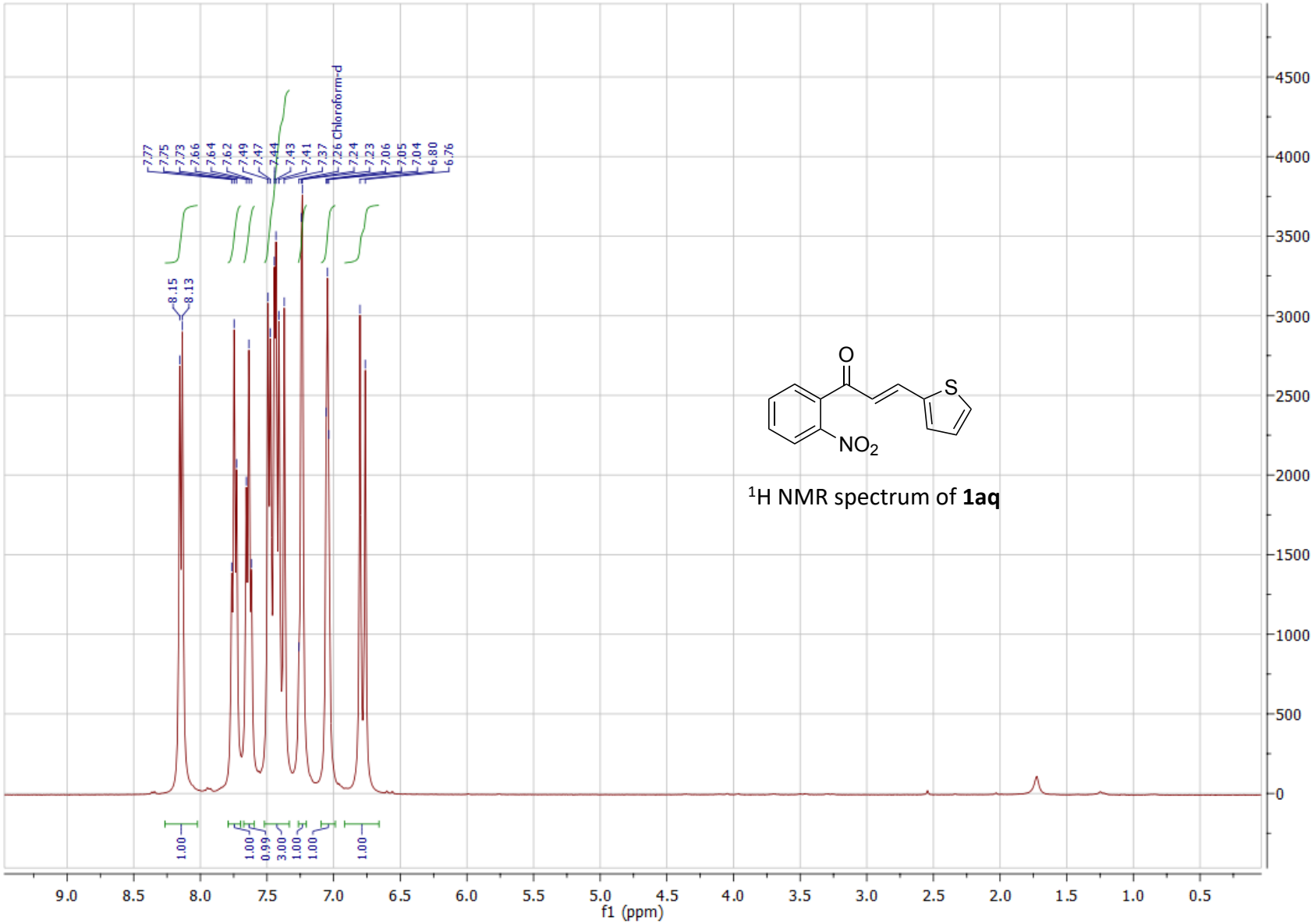
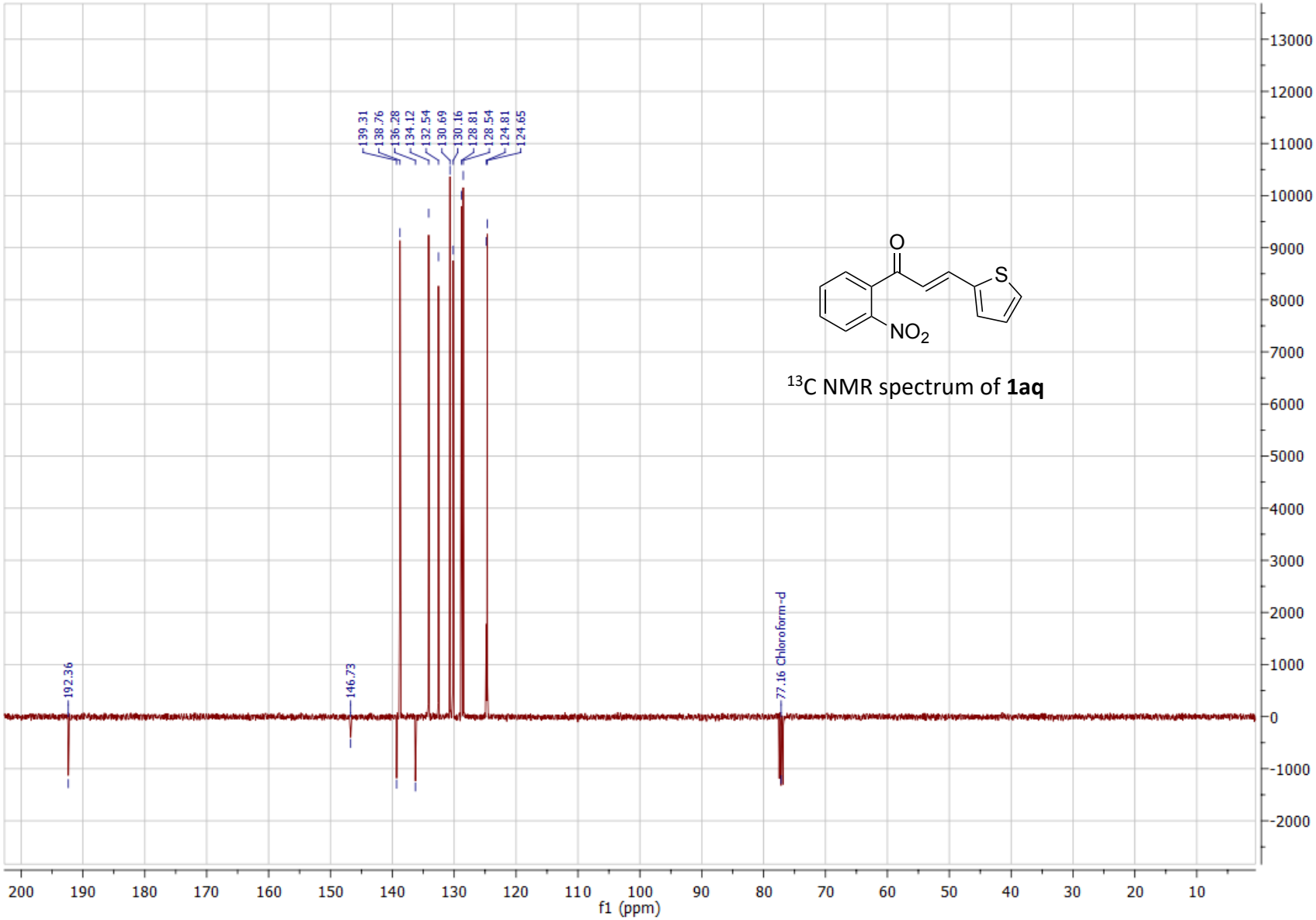


^{13}C NMR spectrum of **1ap**



^{1}H NMR spectrum of **1aq**



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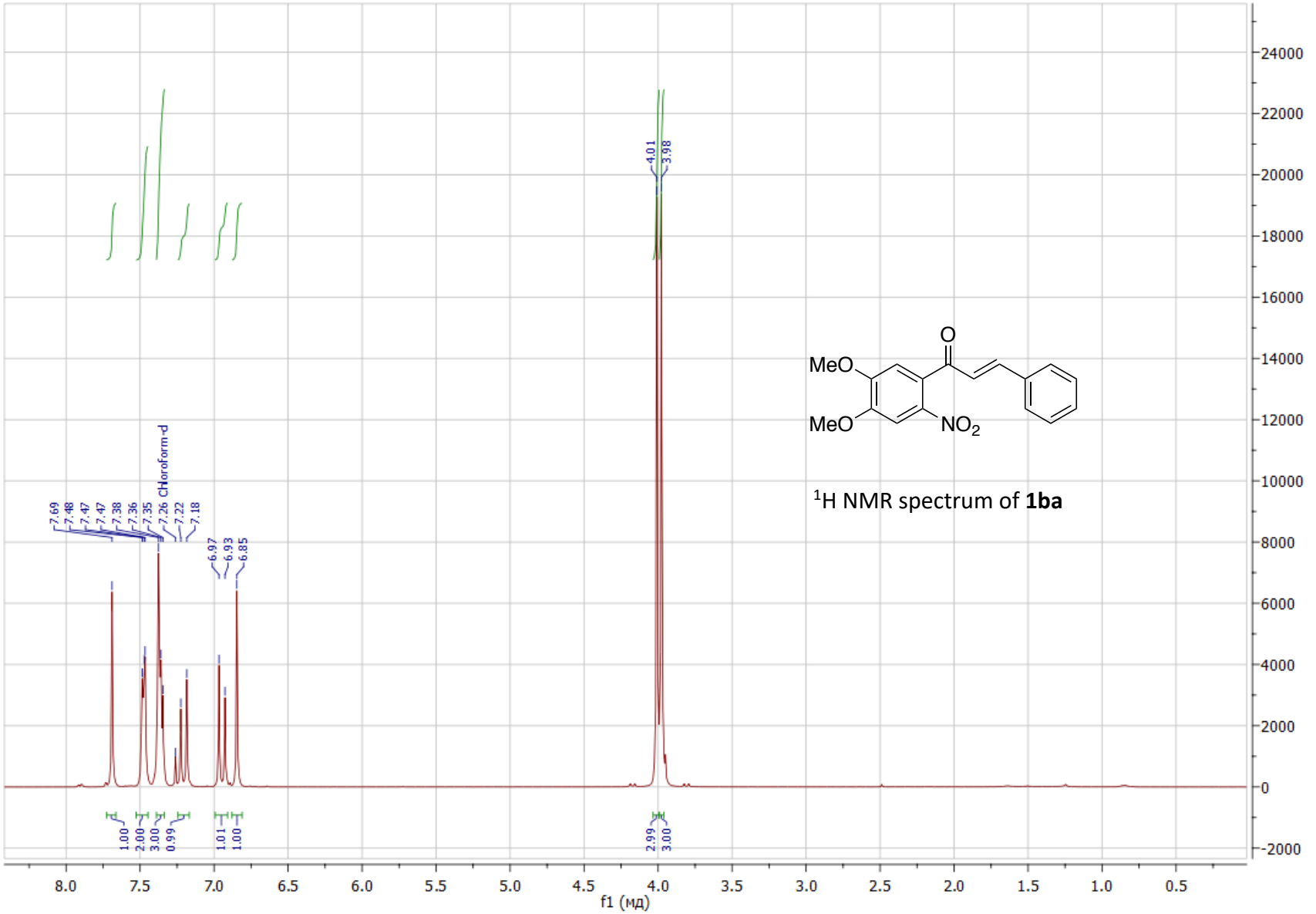

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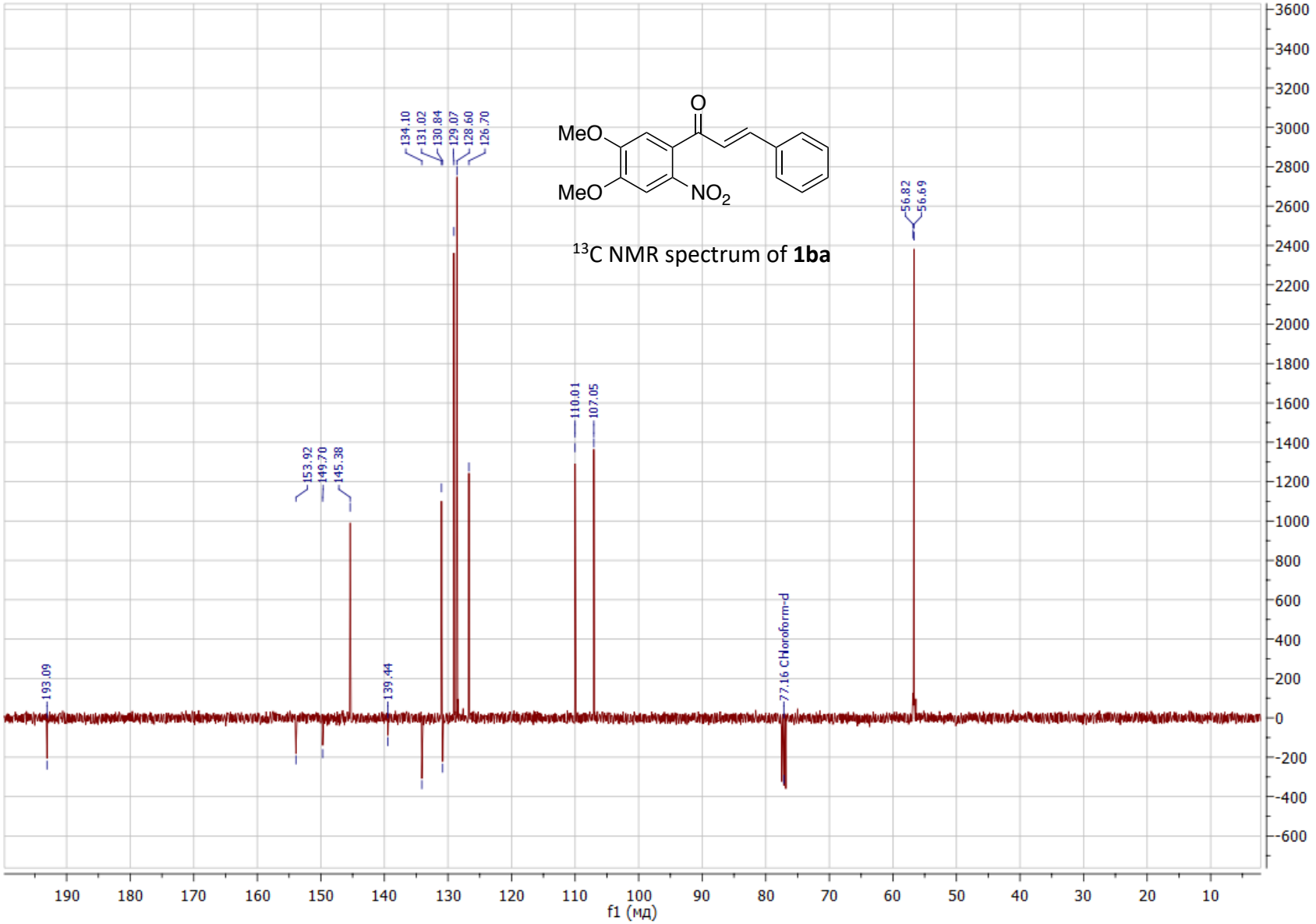
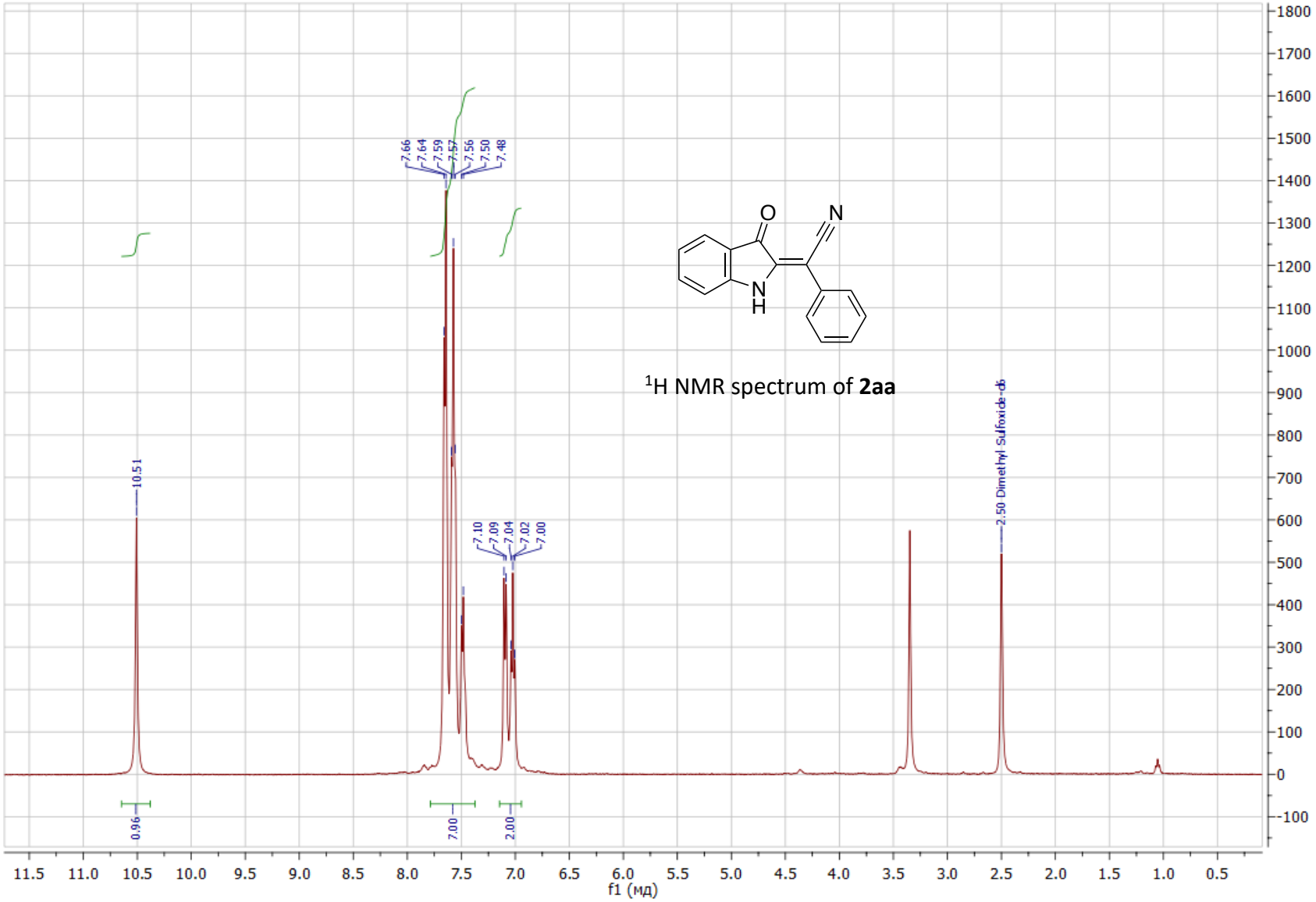


^{13}C NMR spectrum of **1ba**

^{1}H and ^{13}C NMR spectral charts for (*E*)-2-(3-oxoindolin-2-ylidene)-2-arylacetonitriles



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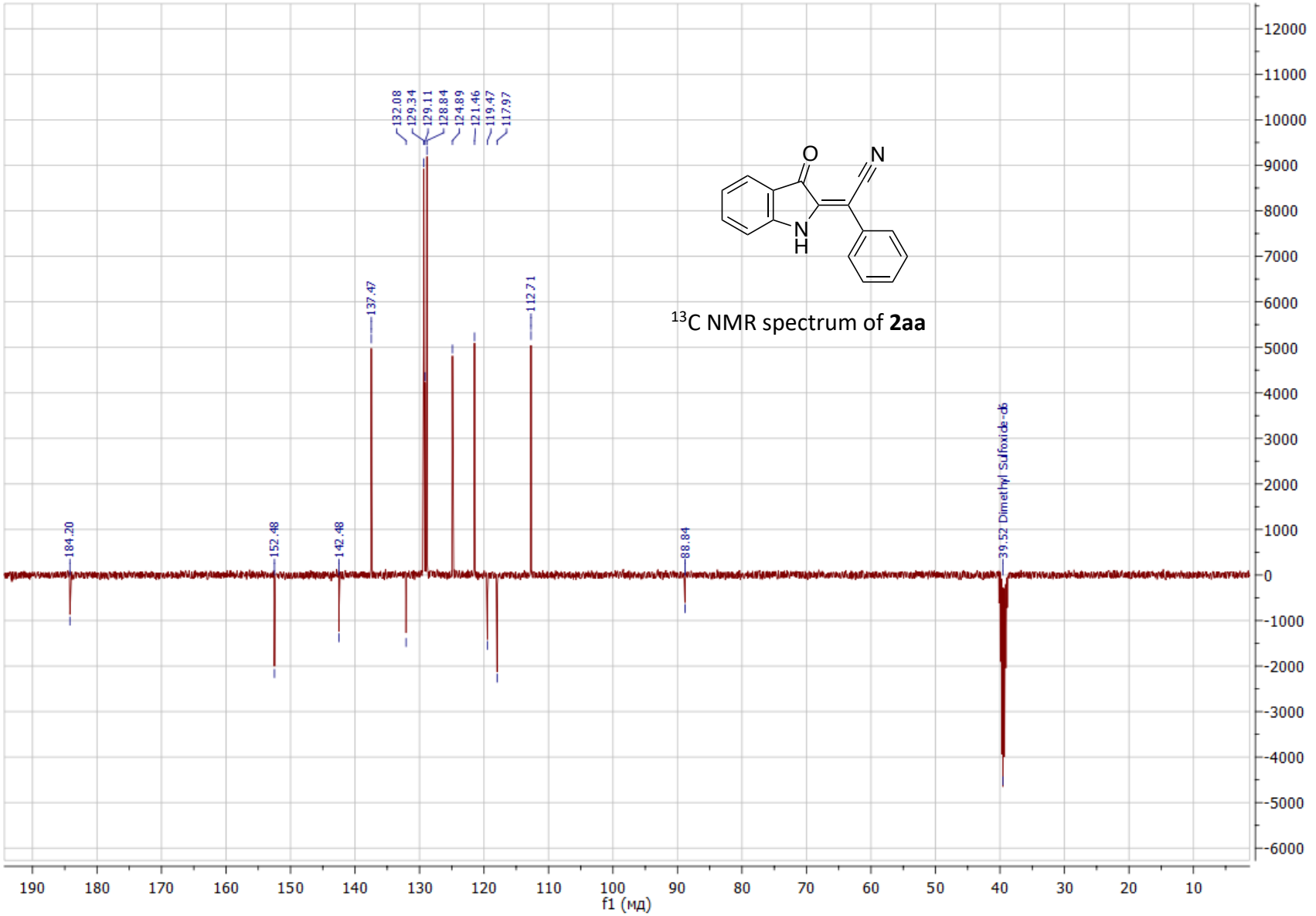

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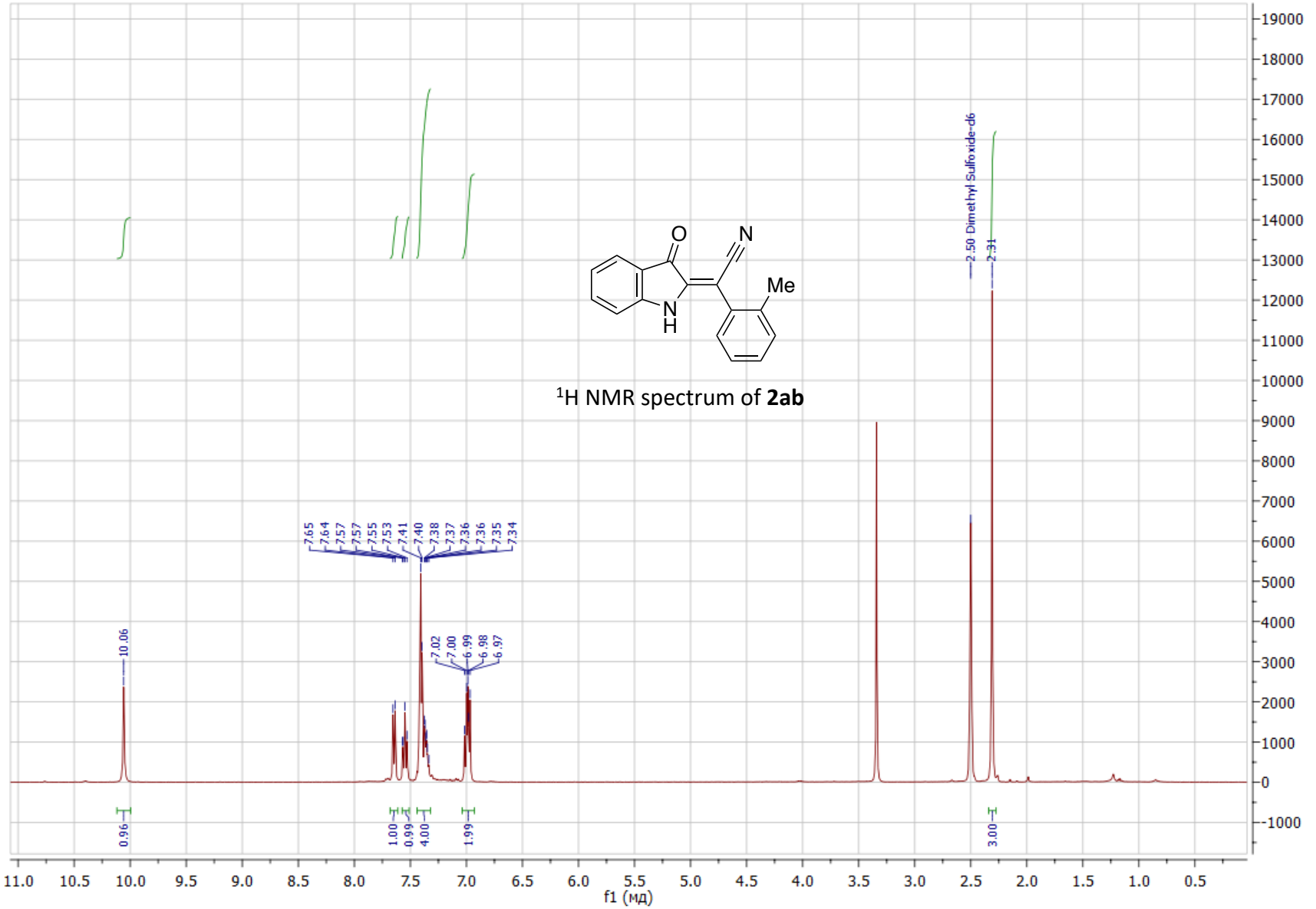
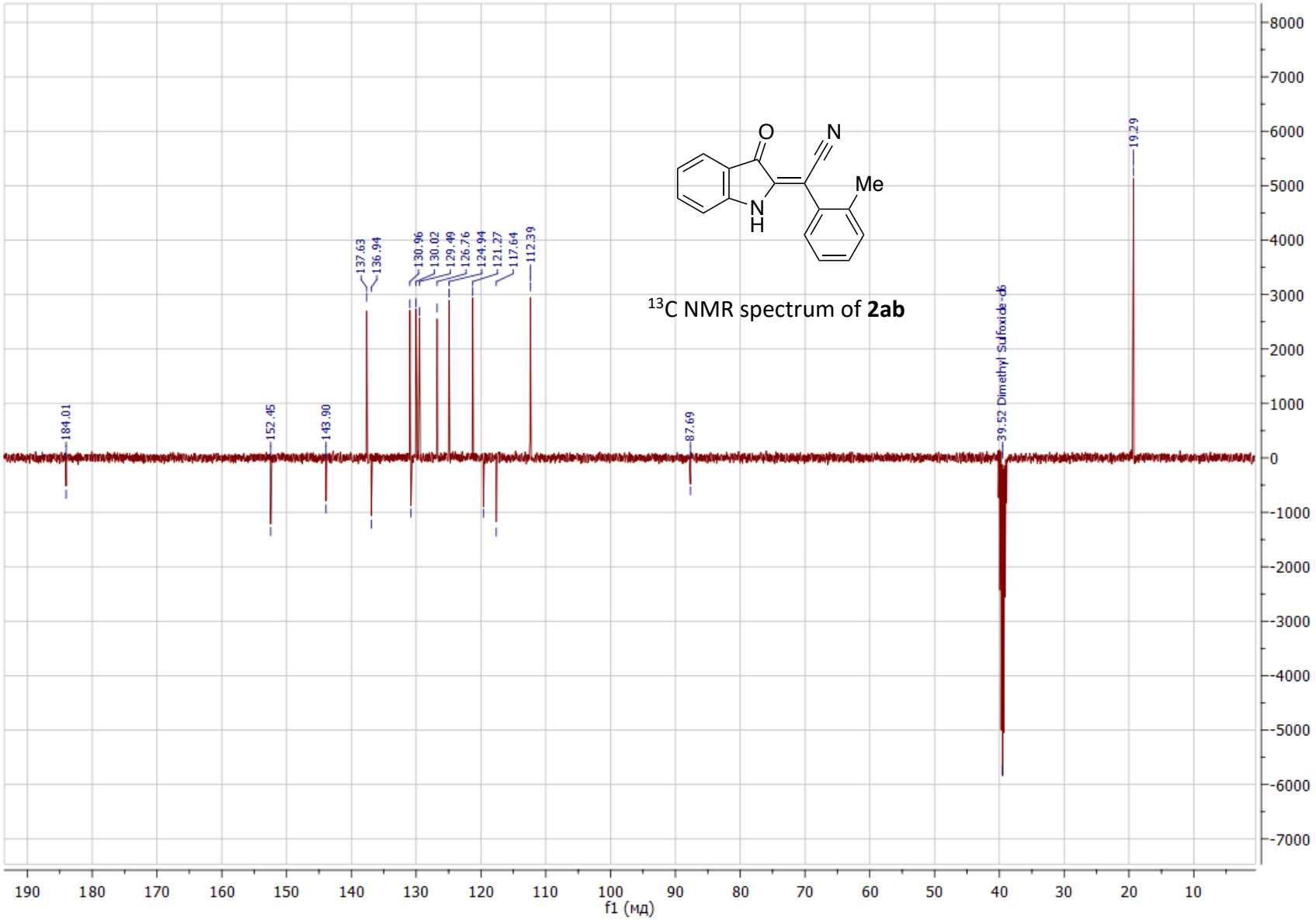
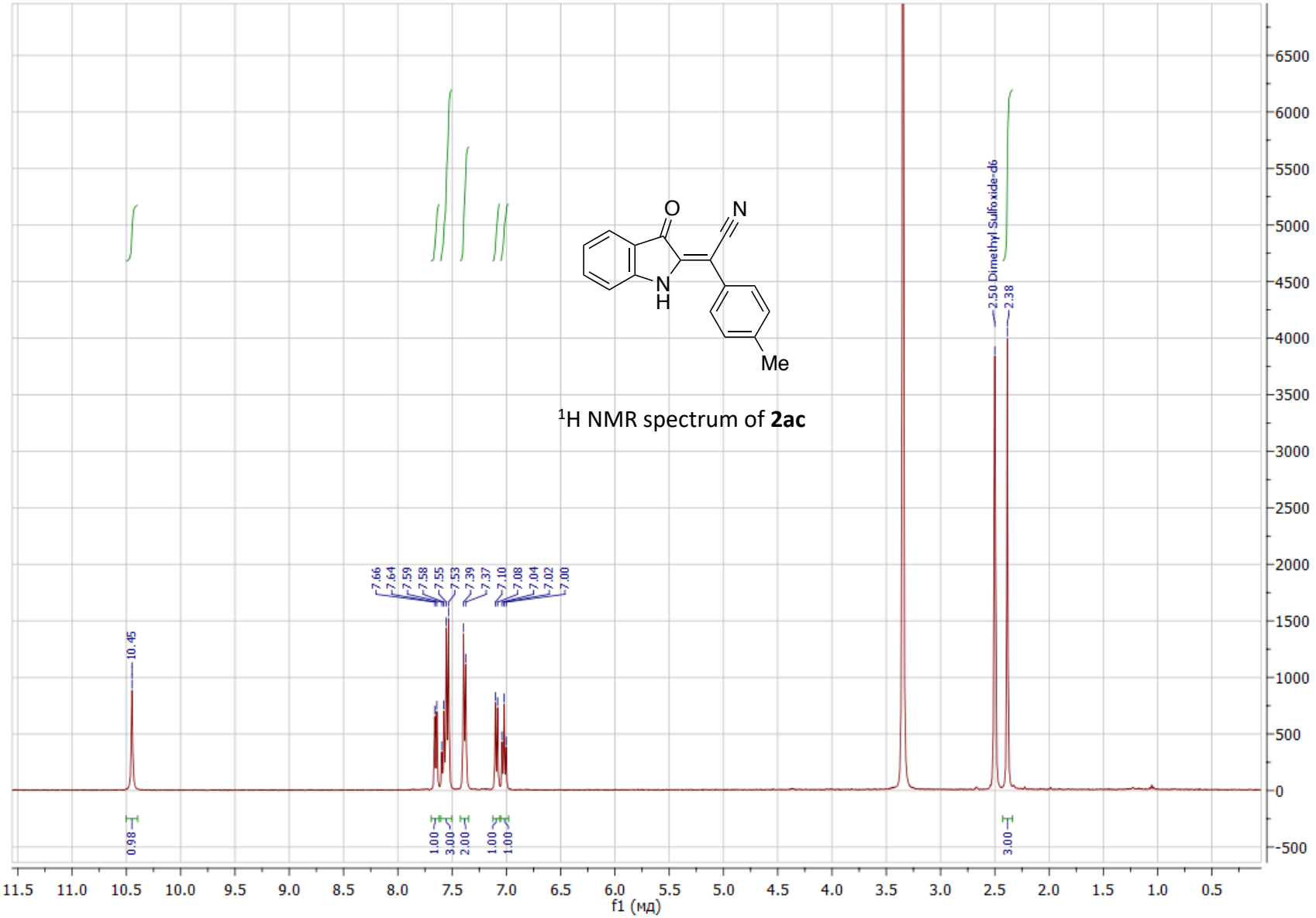


^{1}H NMR spectrum of **2ab**


O
N
Me
N
H
^{13}C NMR spectrum of **2ab**
184.01
152.45
143.90
137.63
136.94
130.96
130.02
129.49
126.76
124.94
121.27
117.64
112.39
87.69
39.52 Dimethyl Sulfoxide-d6
19.29
f1 (мд)



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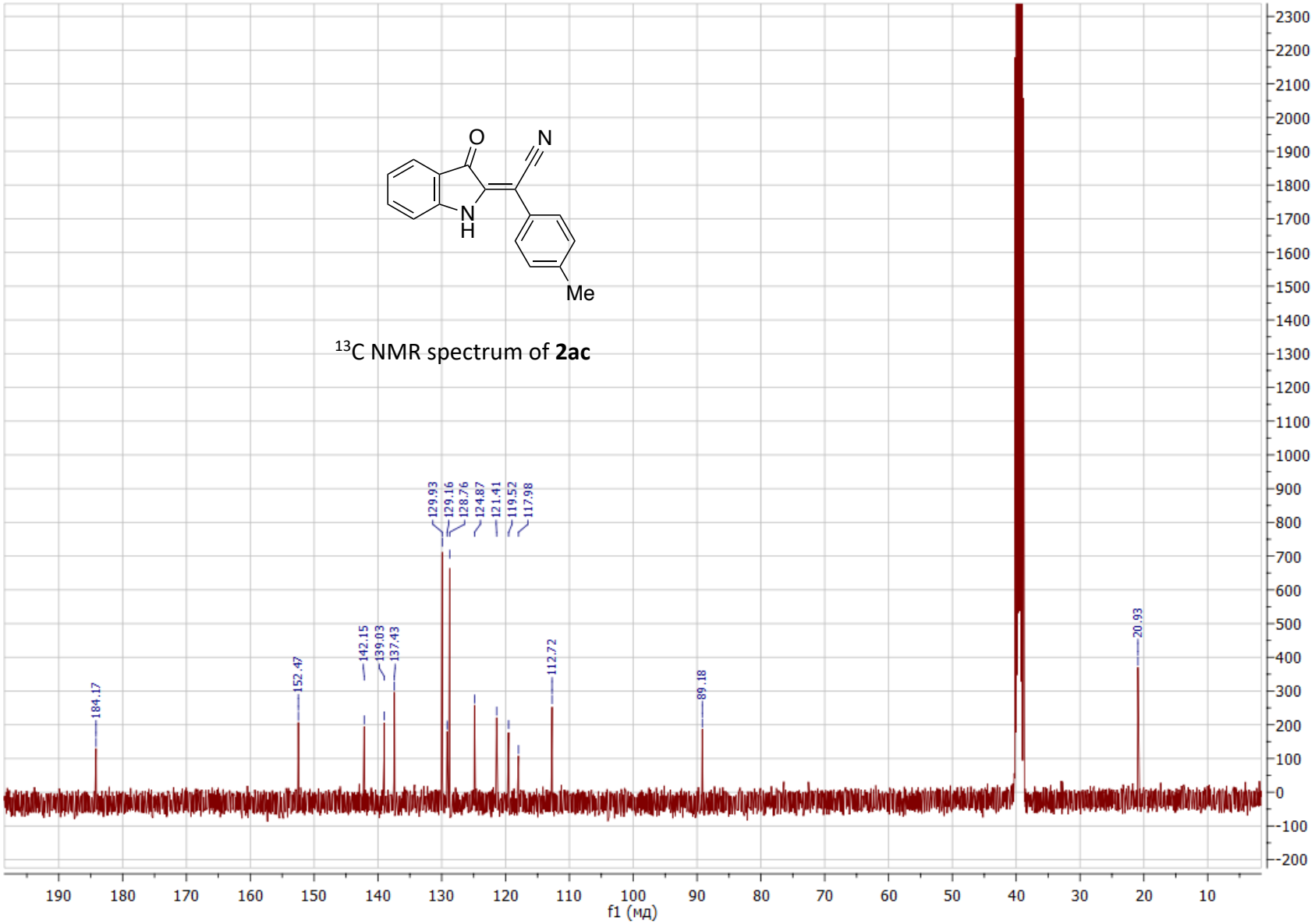

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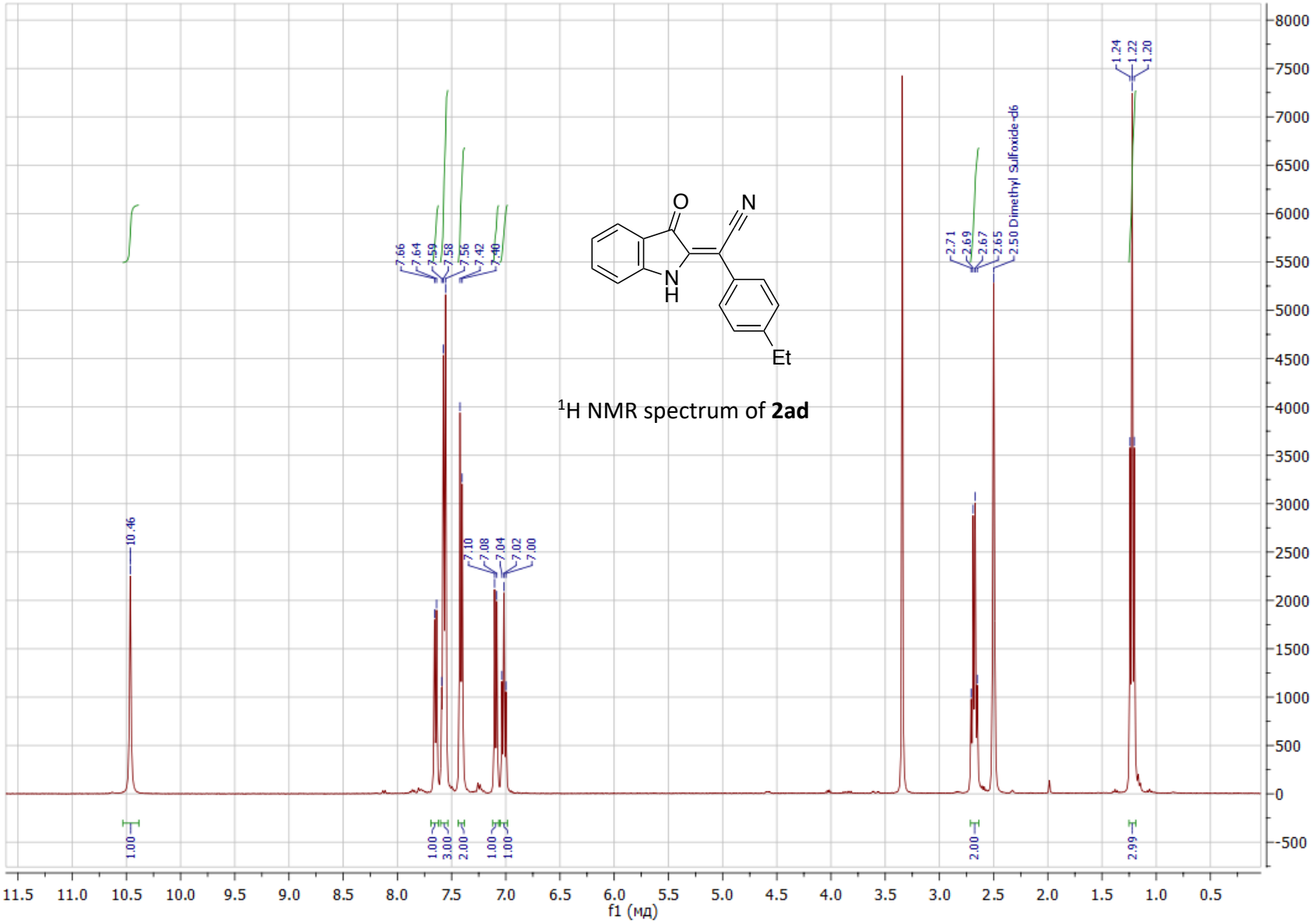

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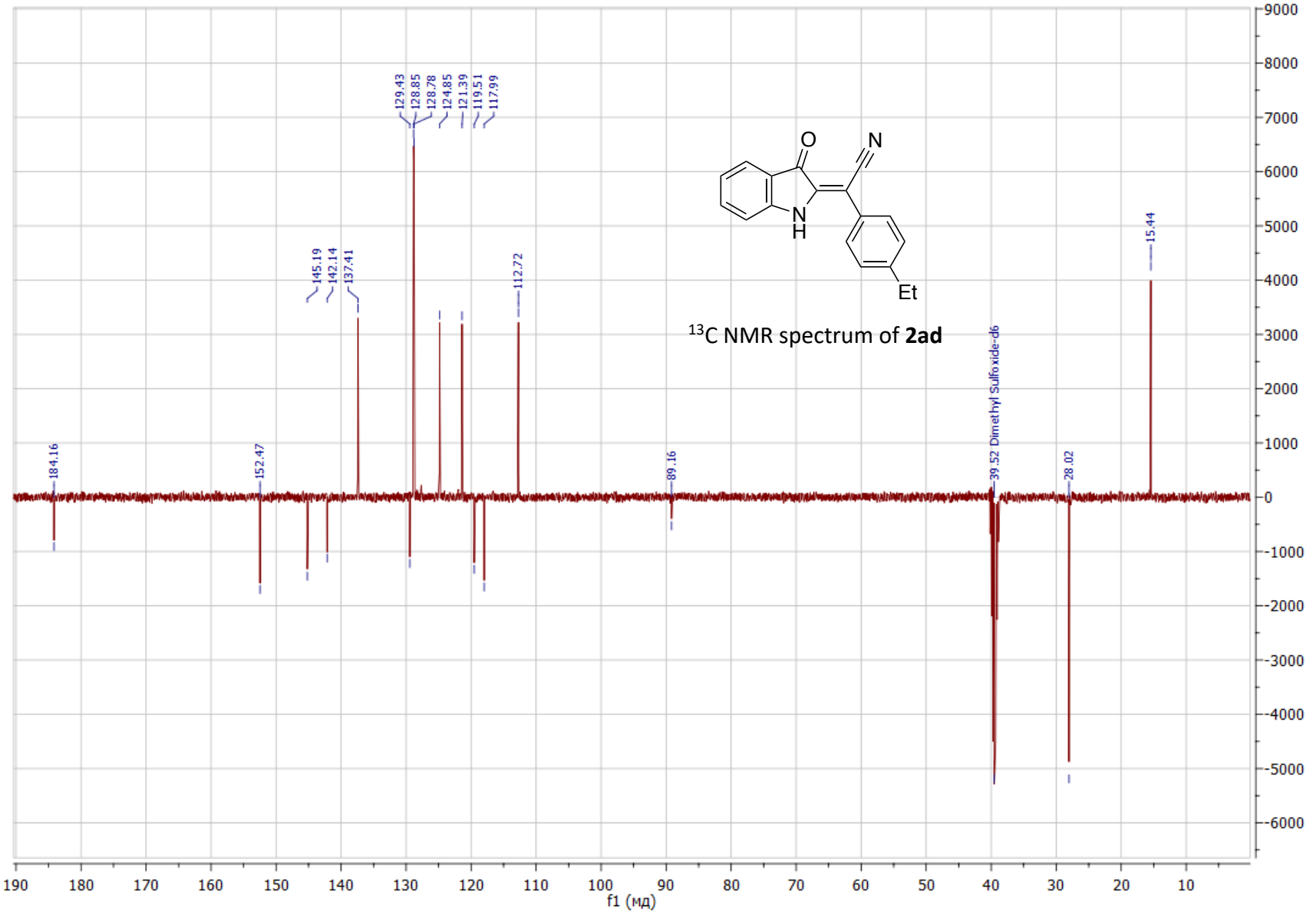
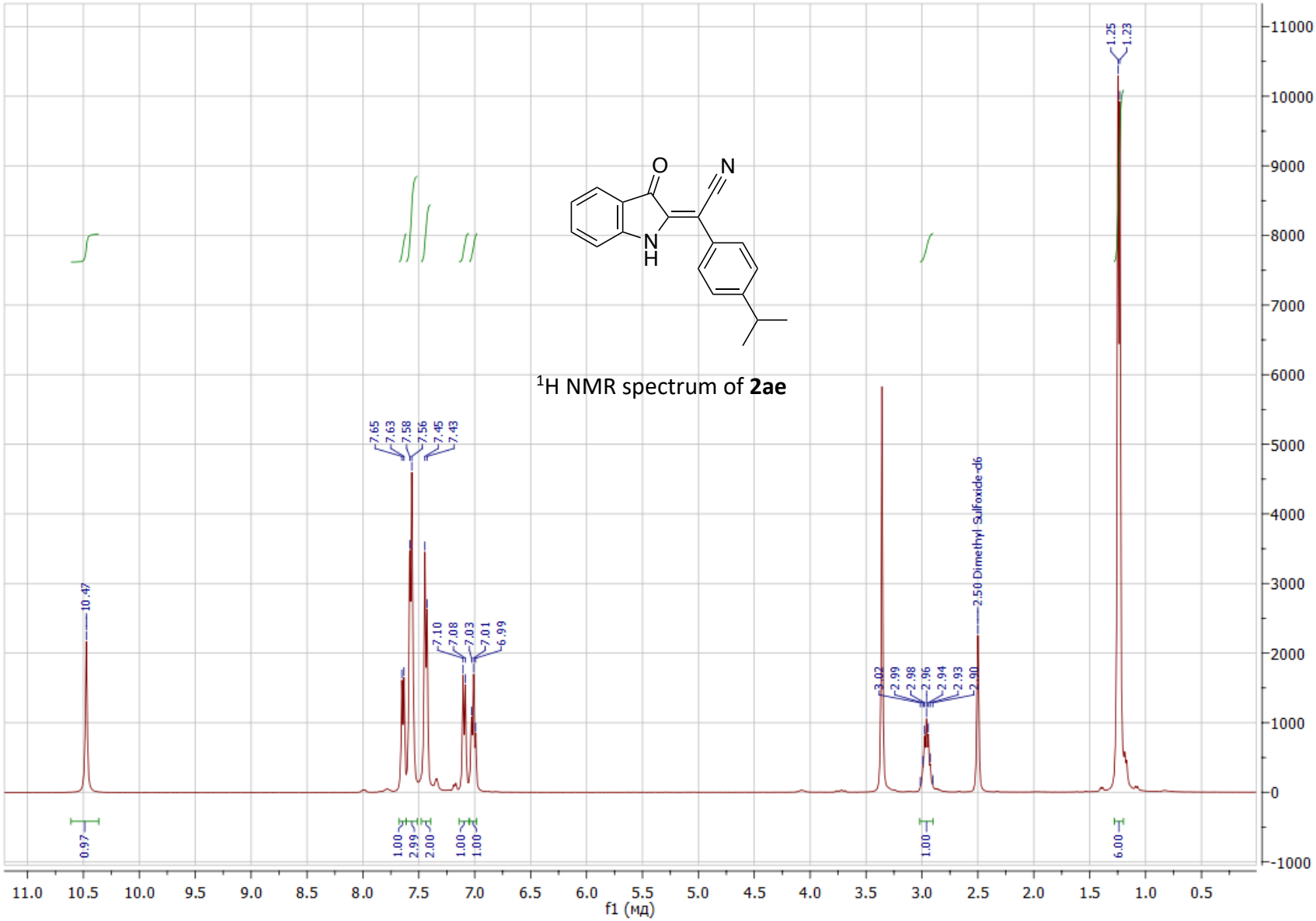
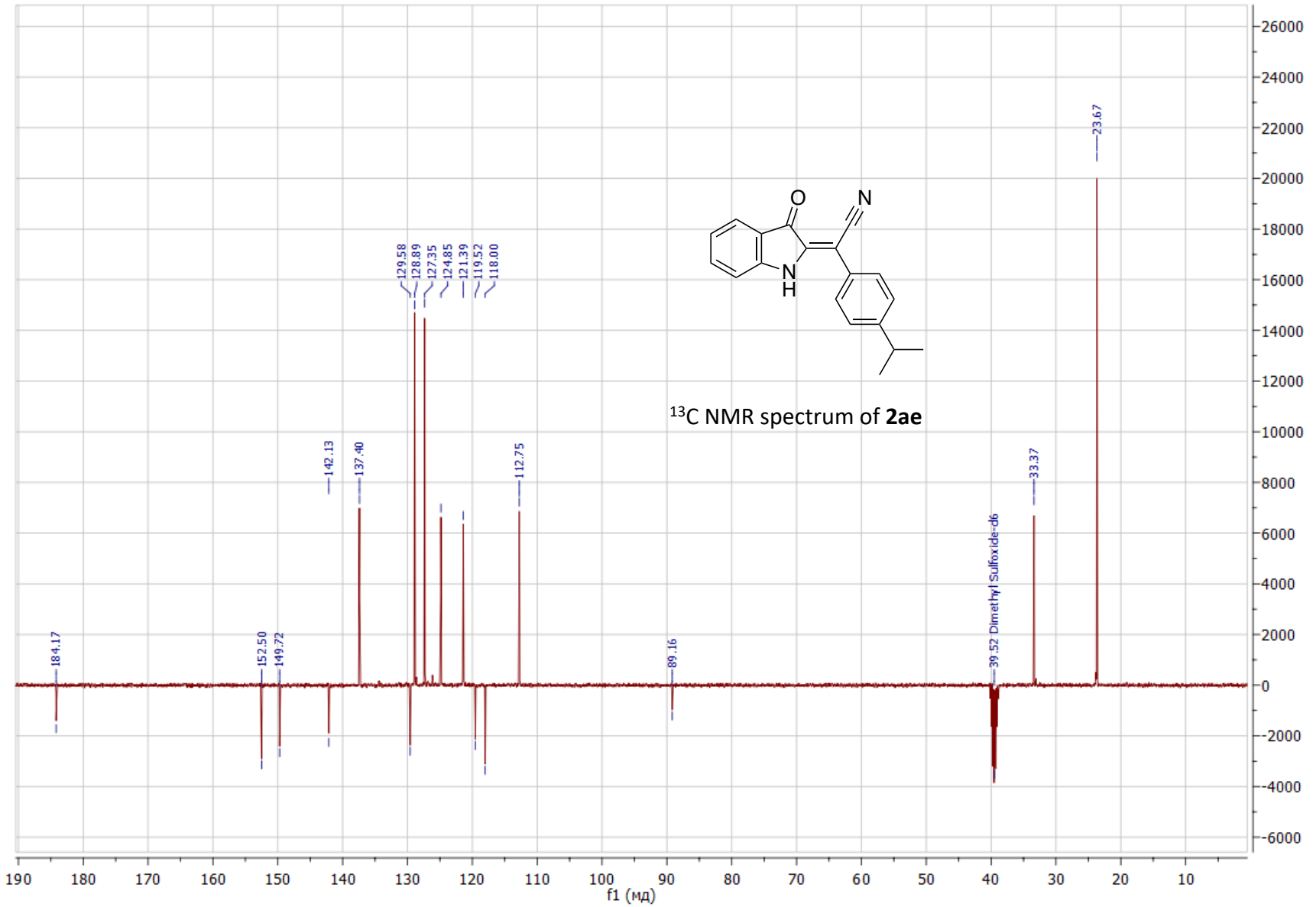


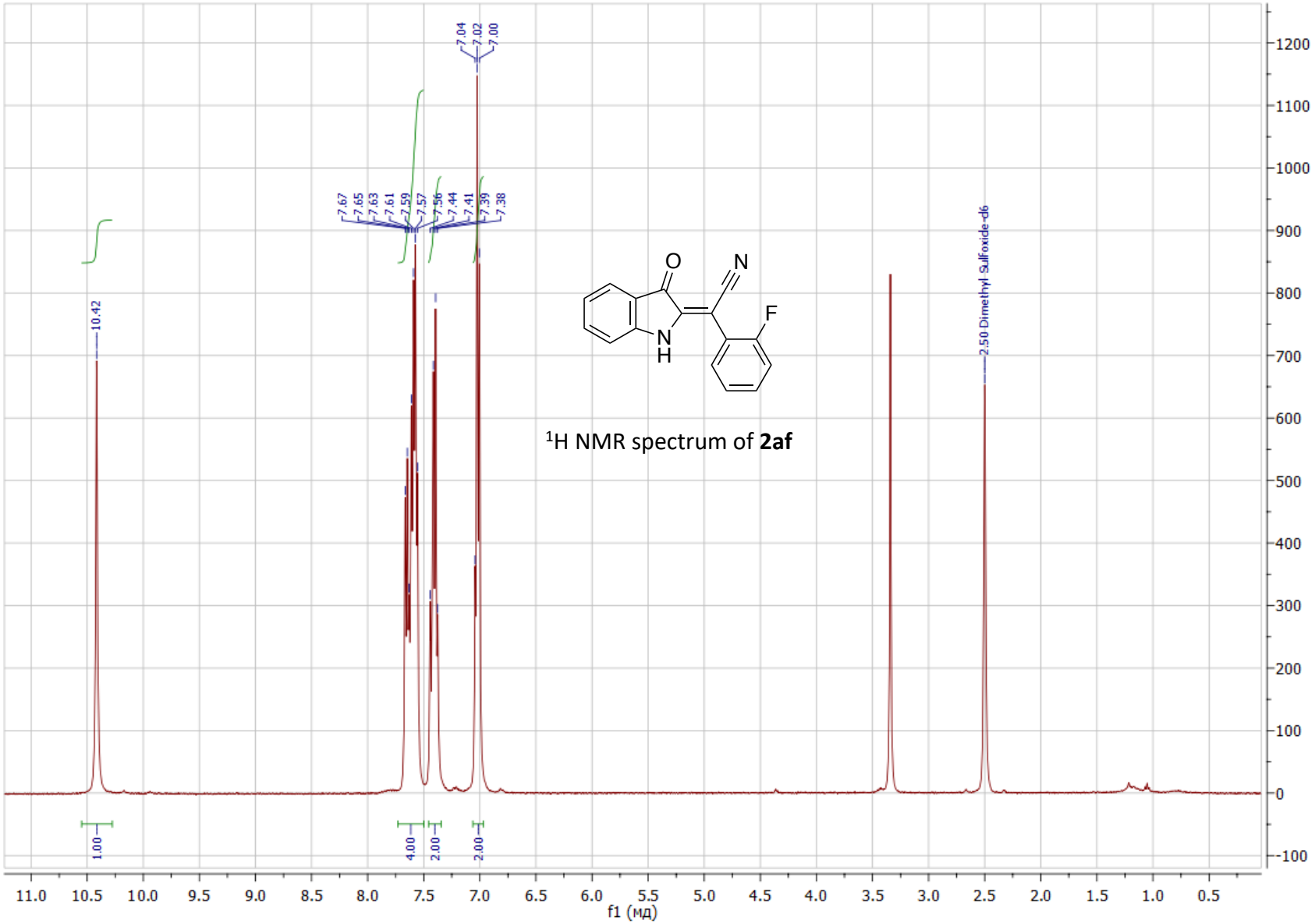
^{13}C NMR spectrum of **2ad**



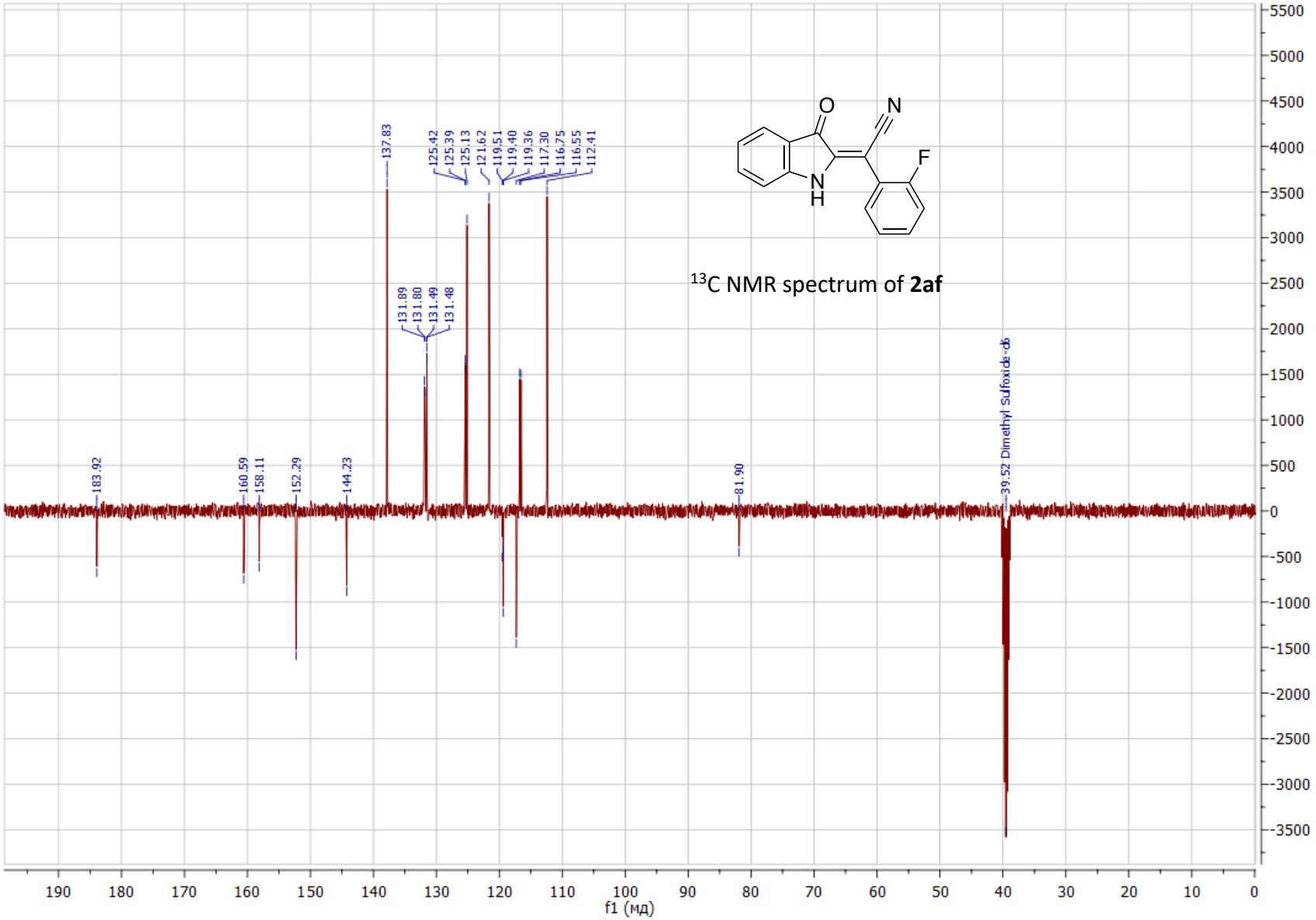
^{1}H NMR spectrum of **2ae**



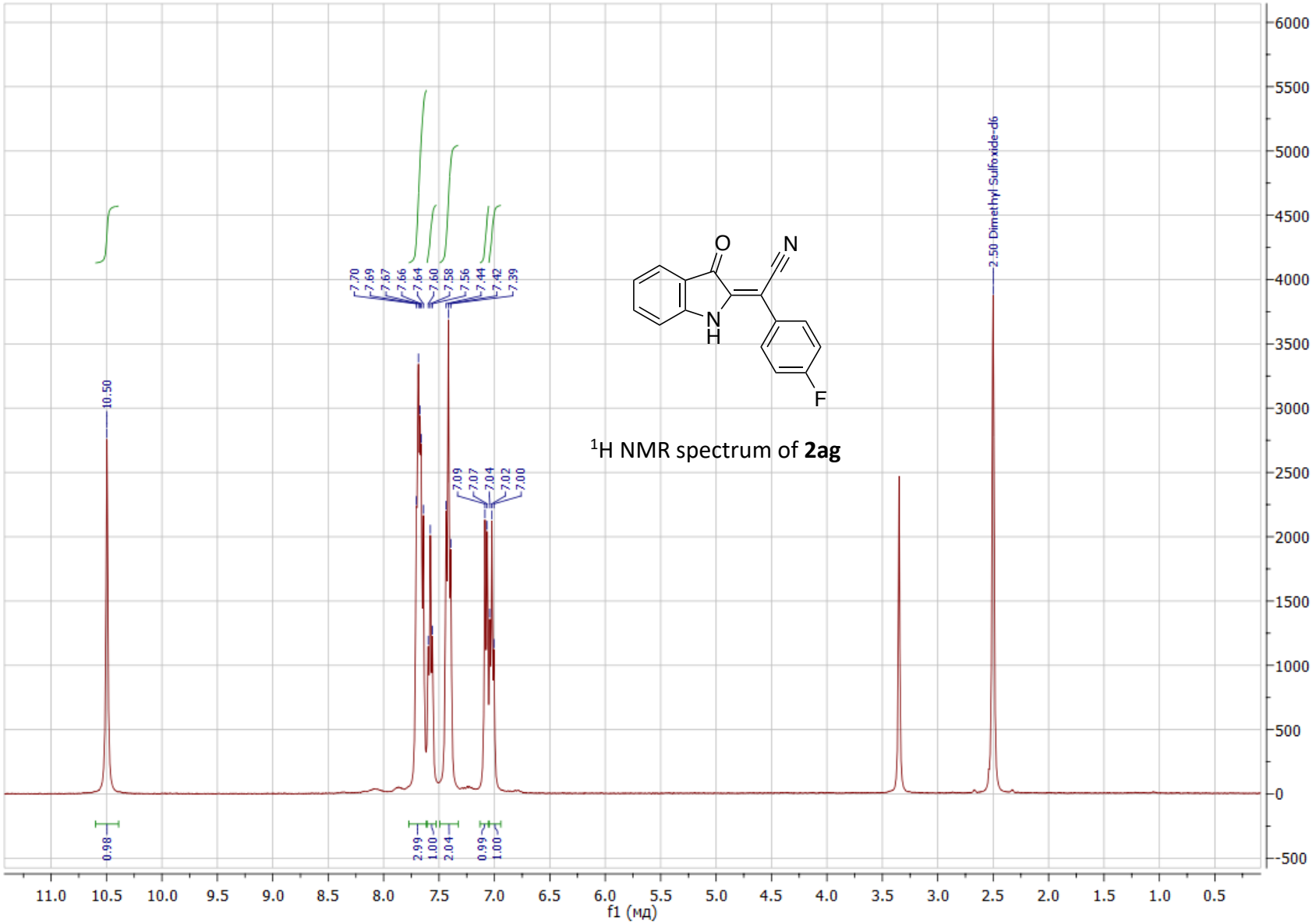
^{13}C NMR spectrum of **2ae**



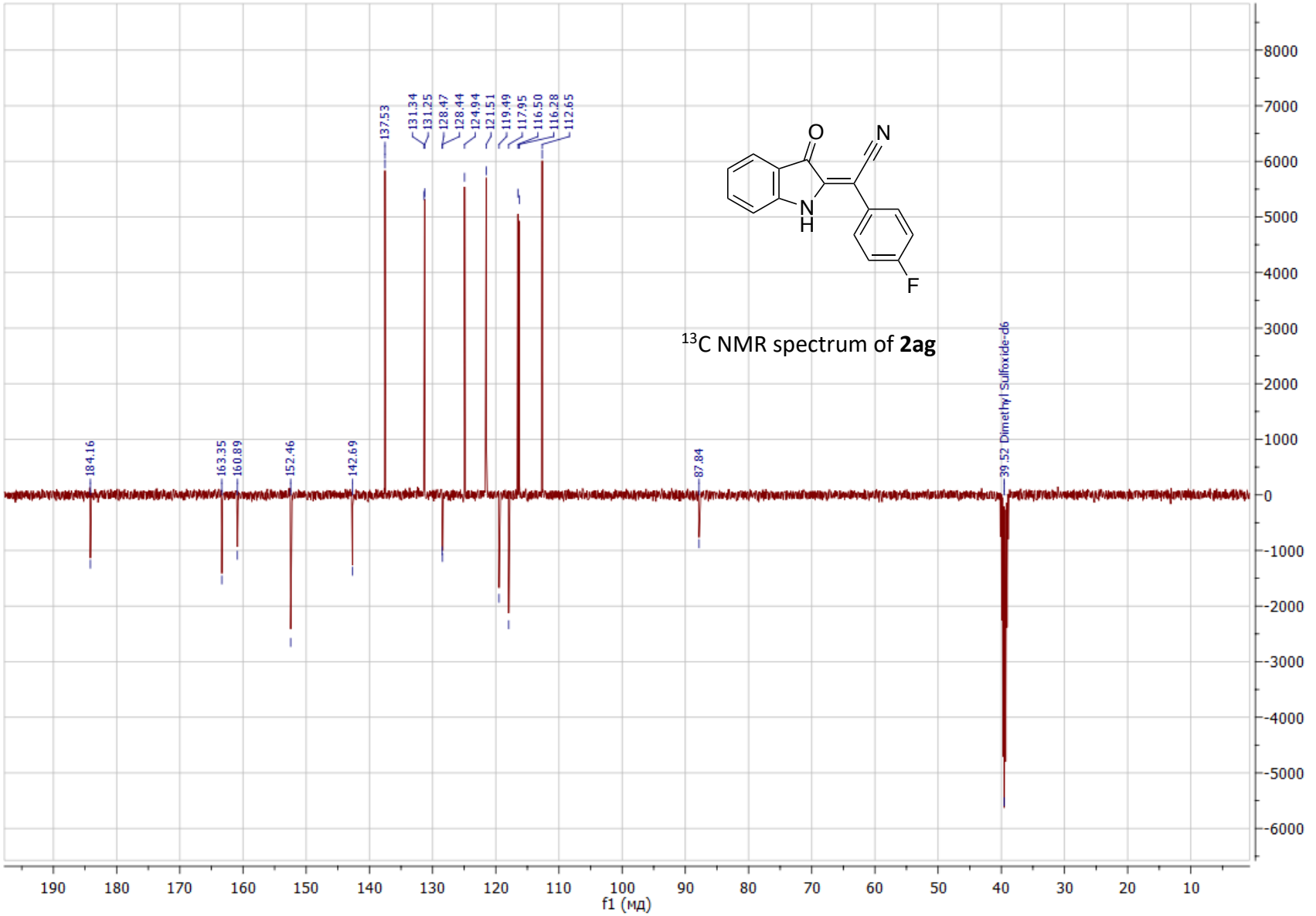
^{1}H NMR spectrum of **2af**



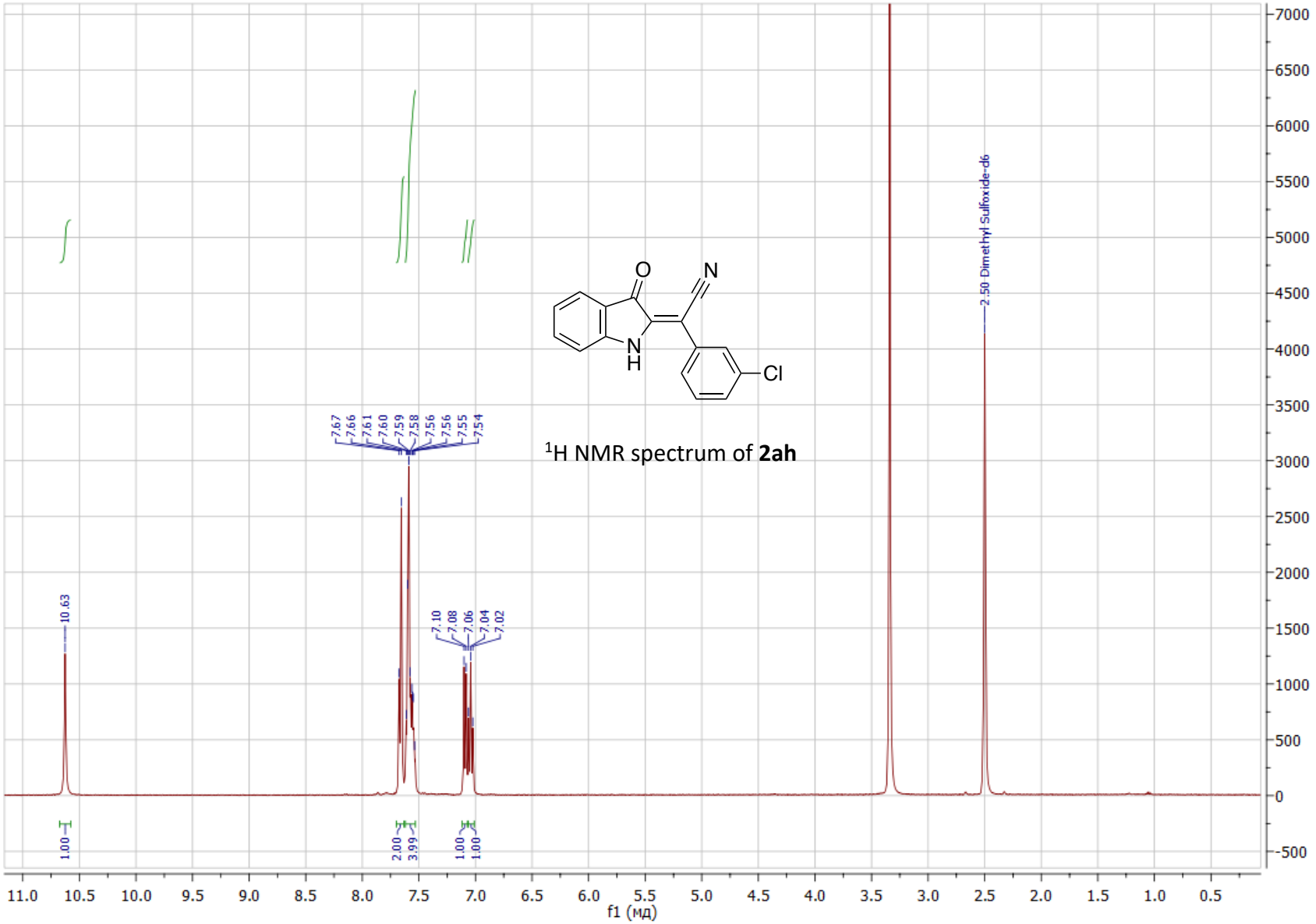
^{13}C NMR spectrum of **2af**

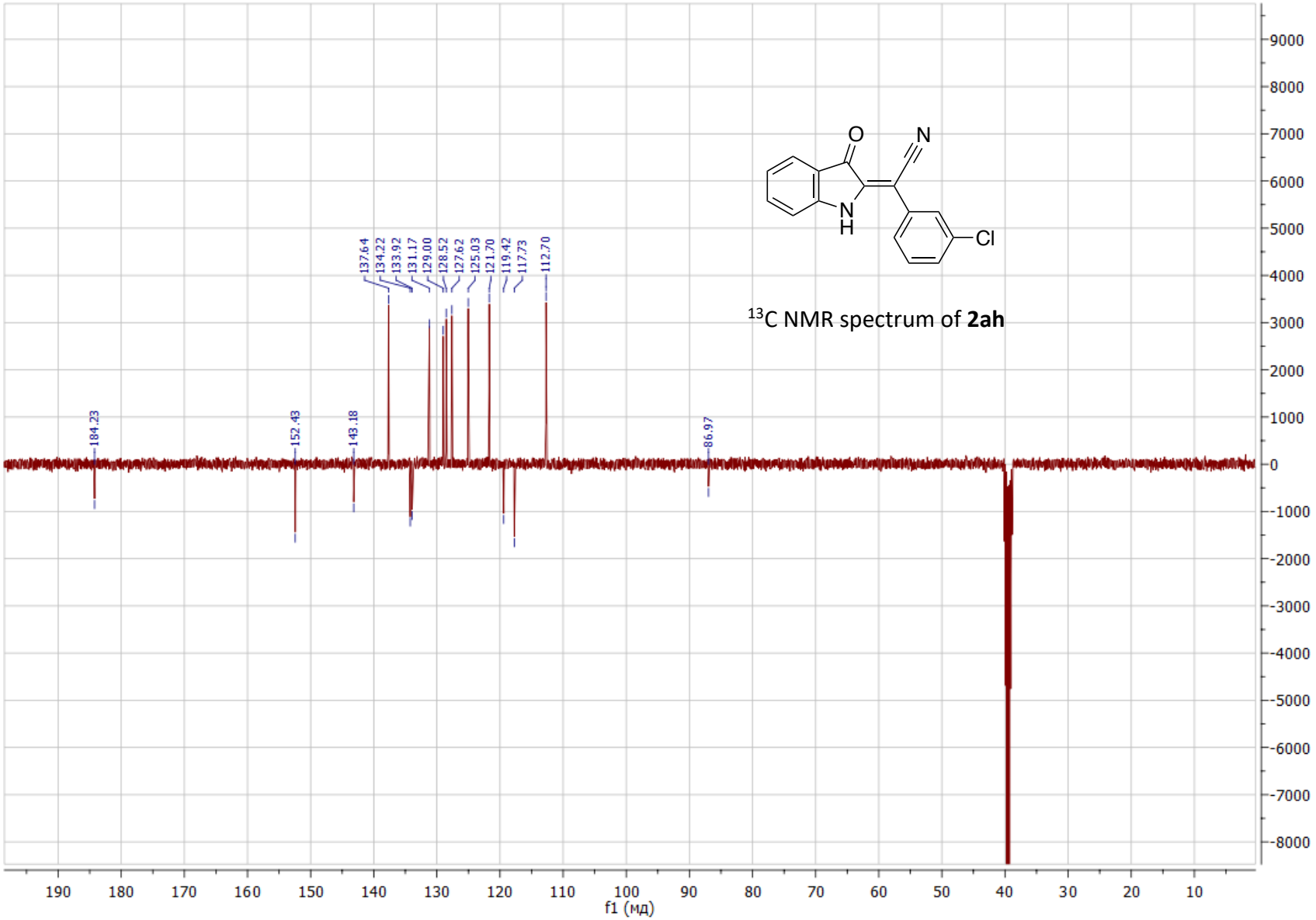


^{1}H NMR spectrum of **2ag**

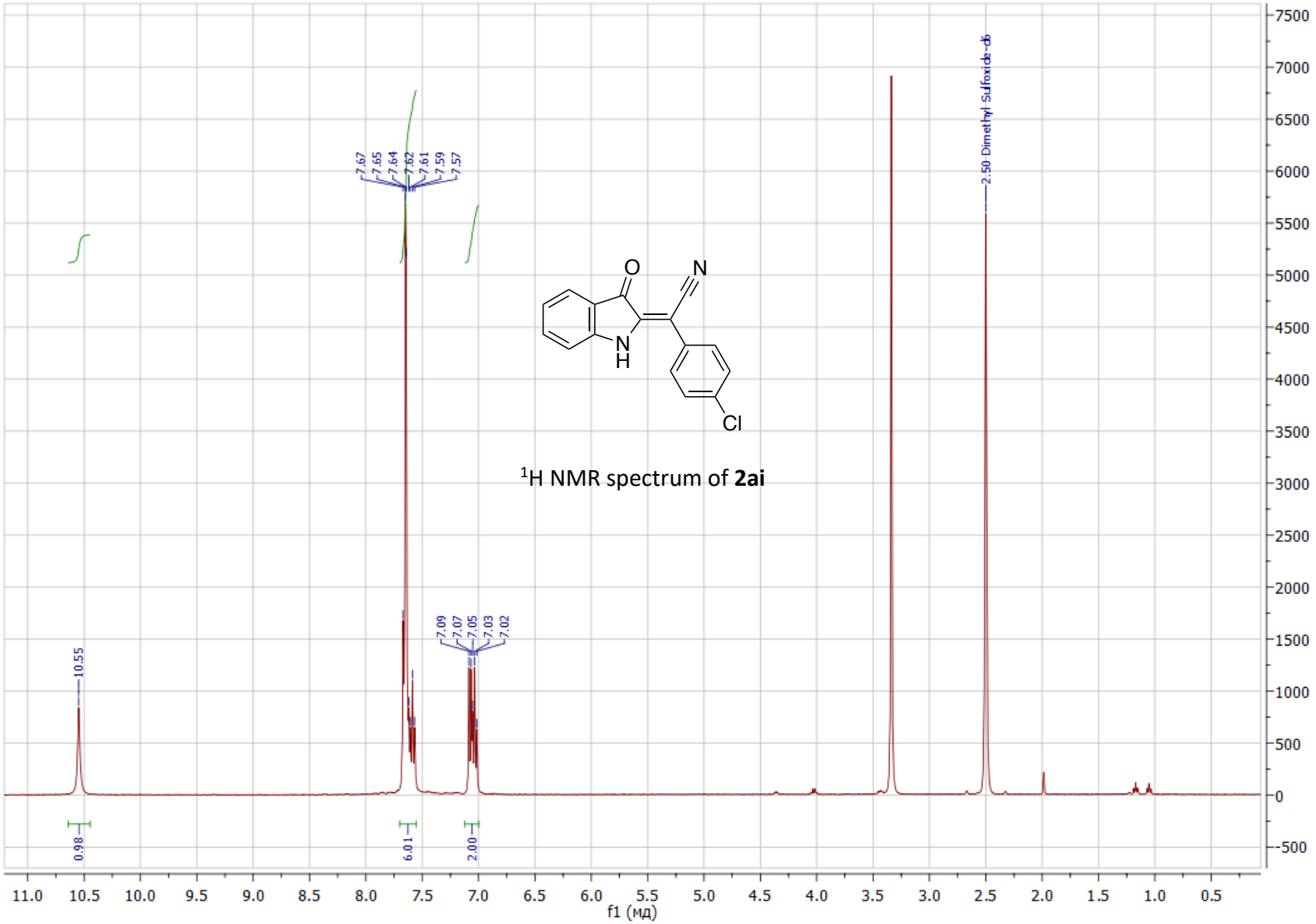


^{13}C NMR spectrum of **2ag**

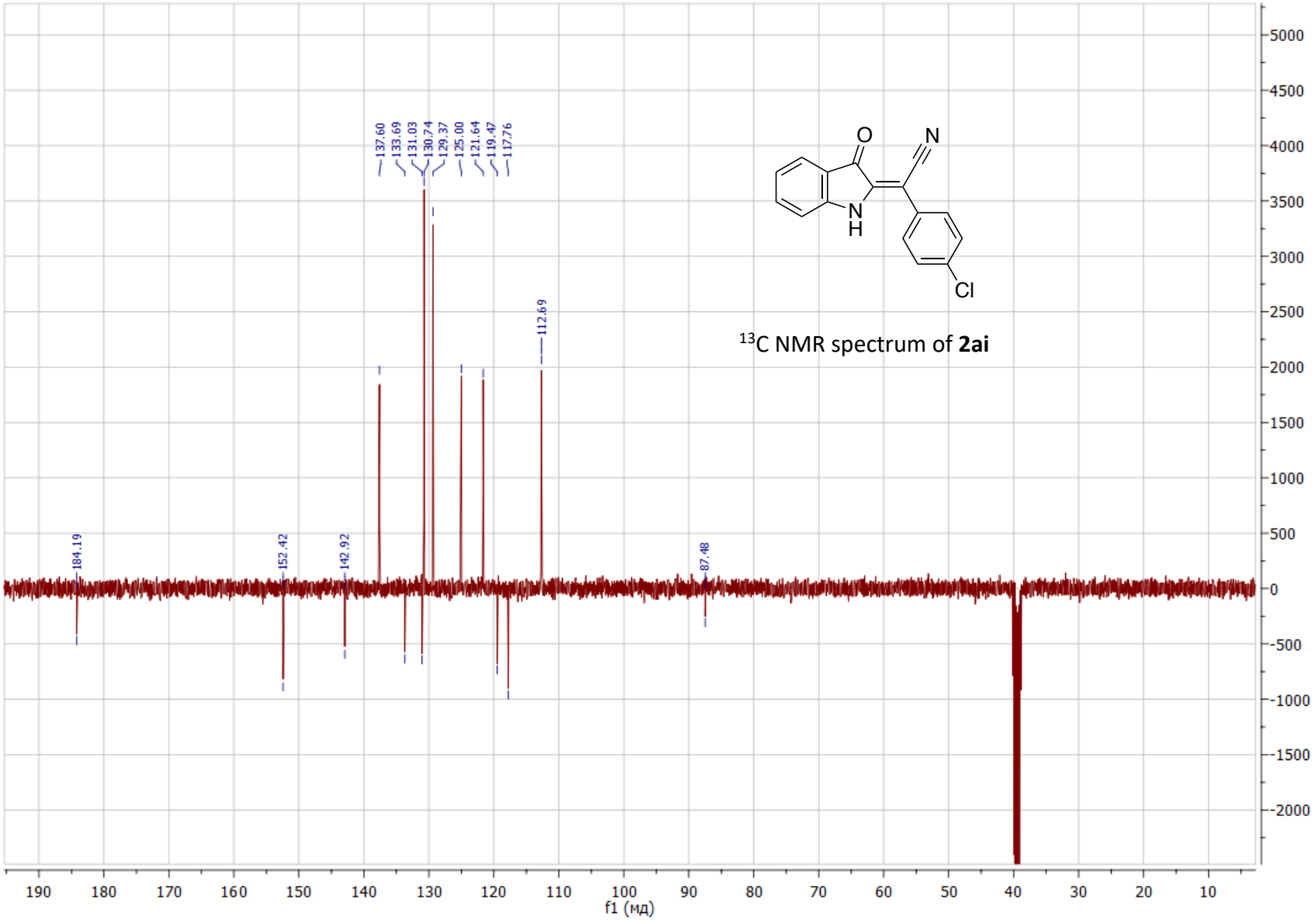

O
N
N
H
Cl
^{1}H NMR spectrum of **2ah**
10.63
7.67
7.66
7.61
7.60
7.59
7.58
7.56
7.56
7.55
7.54
7.10
7.08
7.06
7.04
7.02
2.50 Dimethyl Sulfoxide-d6
1.00
2.00
3.99
1.00
1.00
f1 (мд)



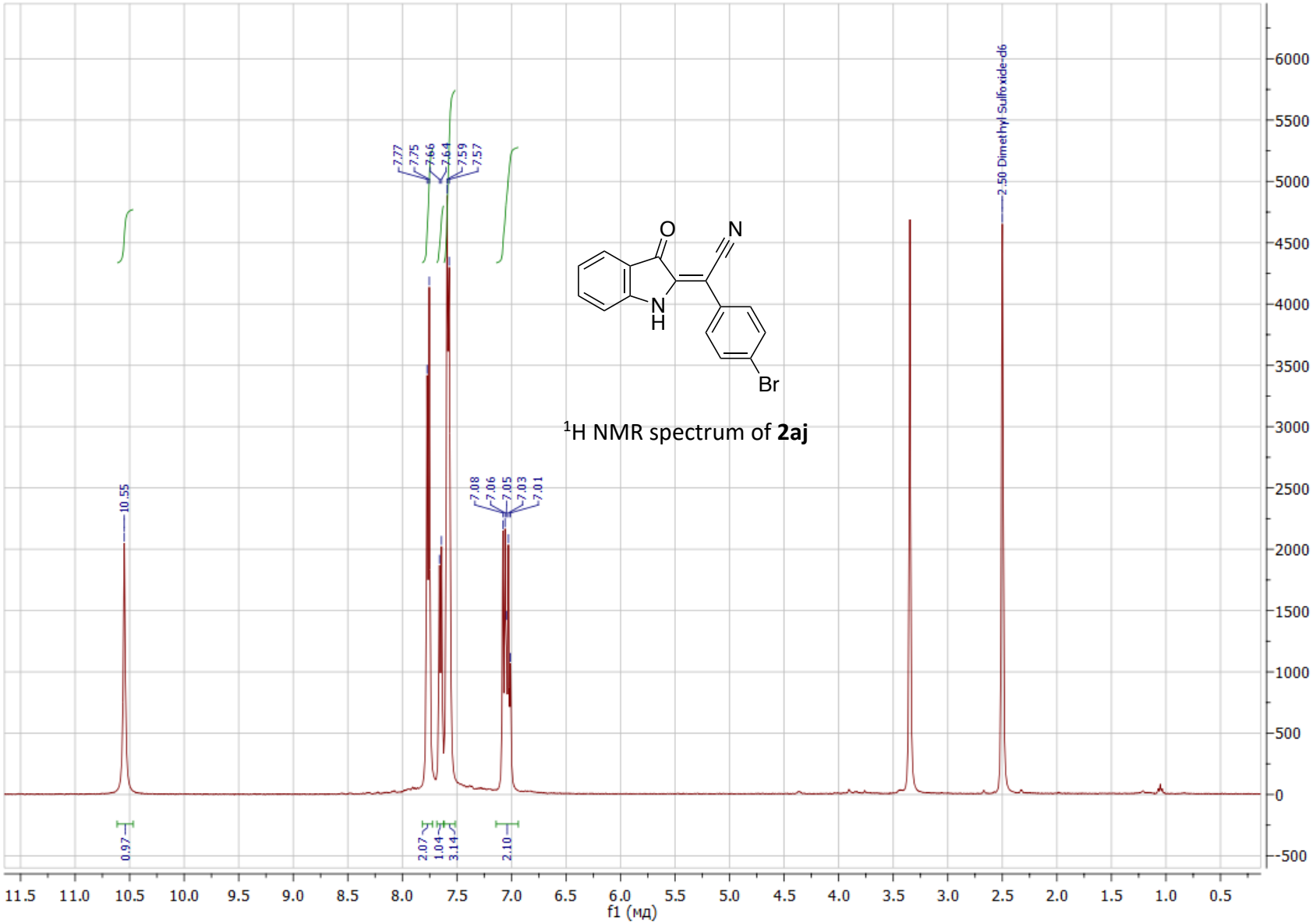
^{13}C NMR spectrum of **2ah**



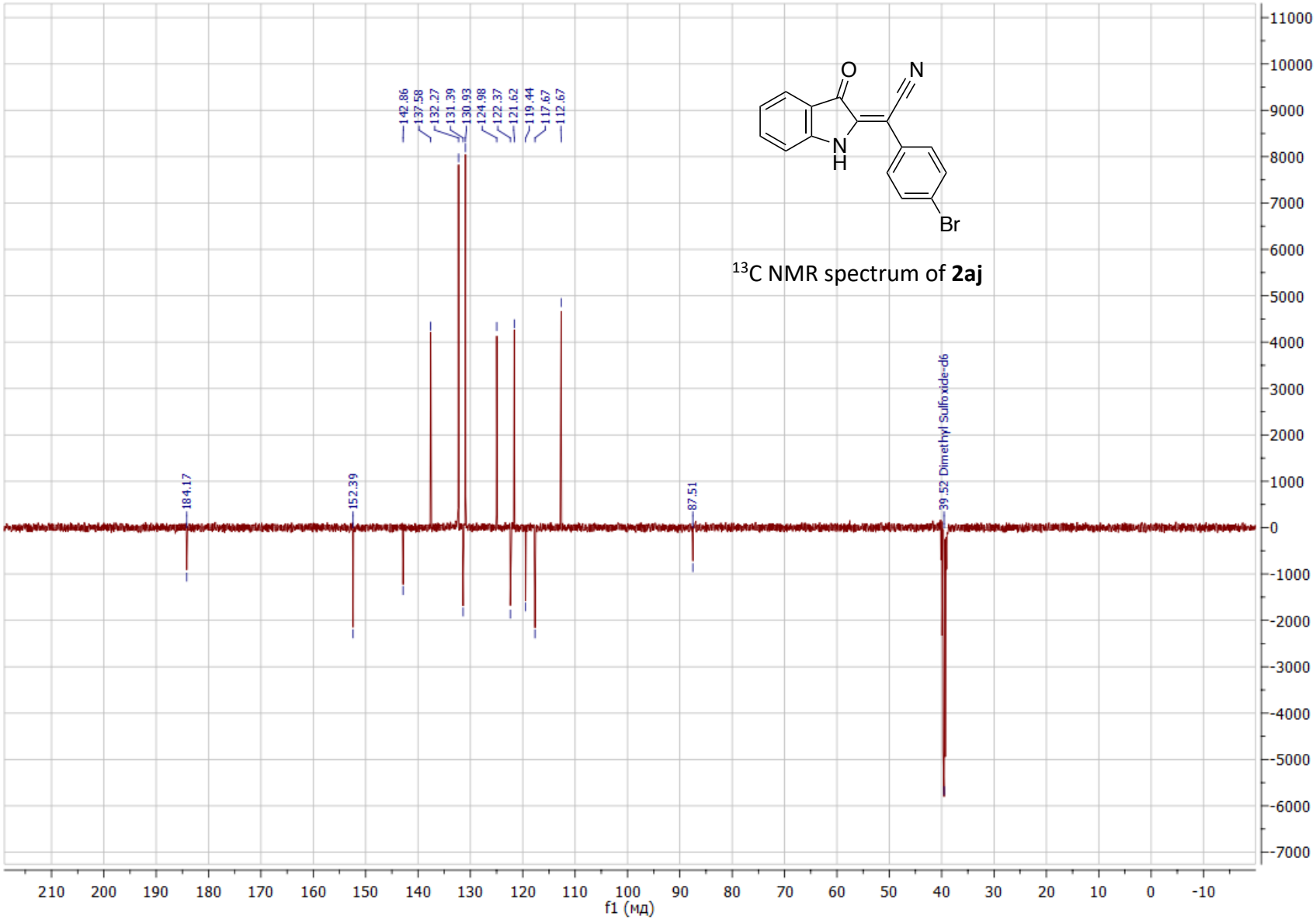
^{1}H NMR spectrum of **2ai**



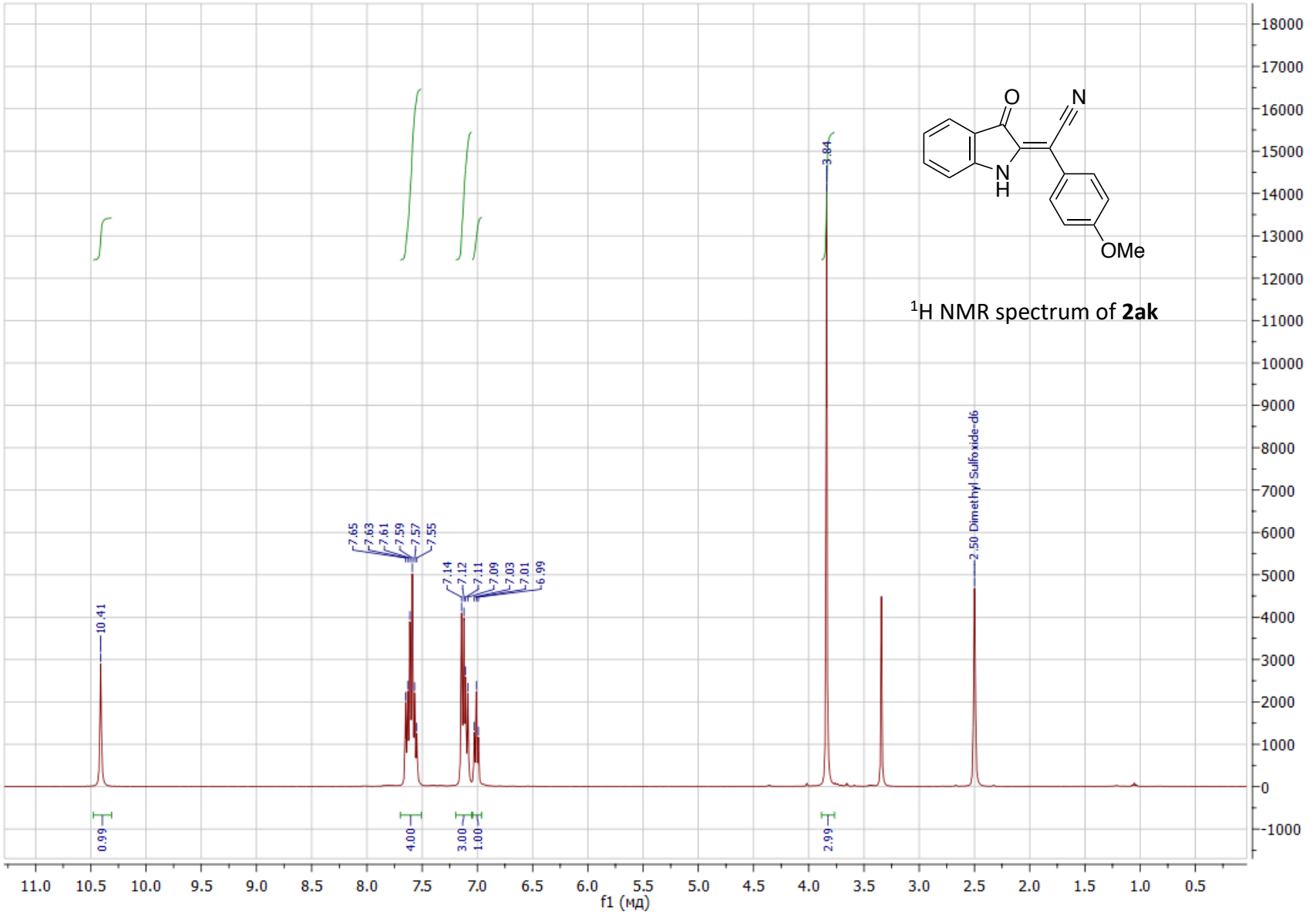
^{13}C NMR spectrum of **2ai**



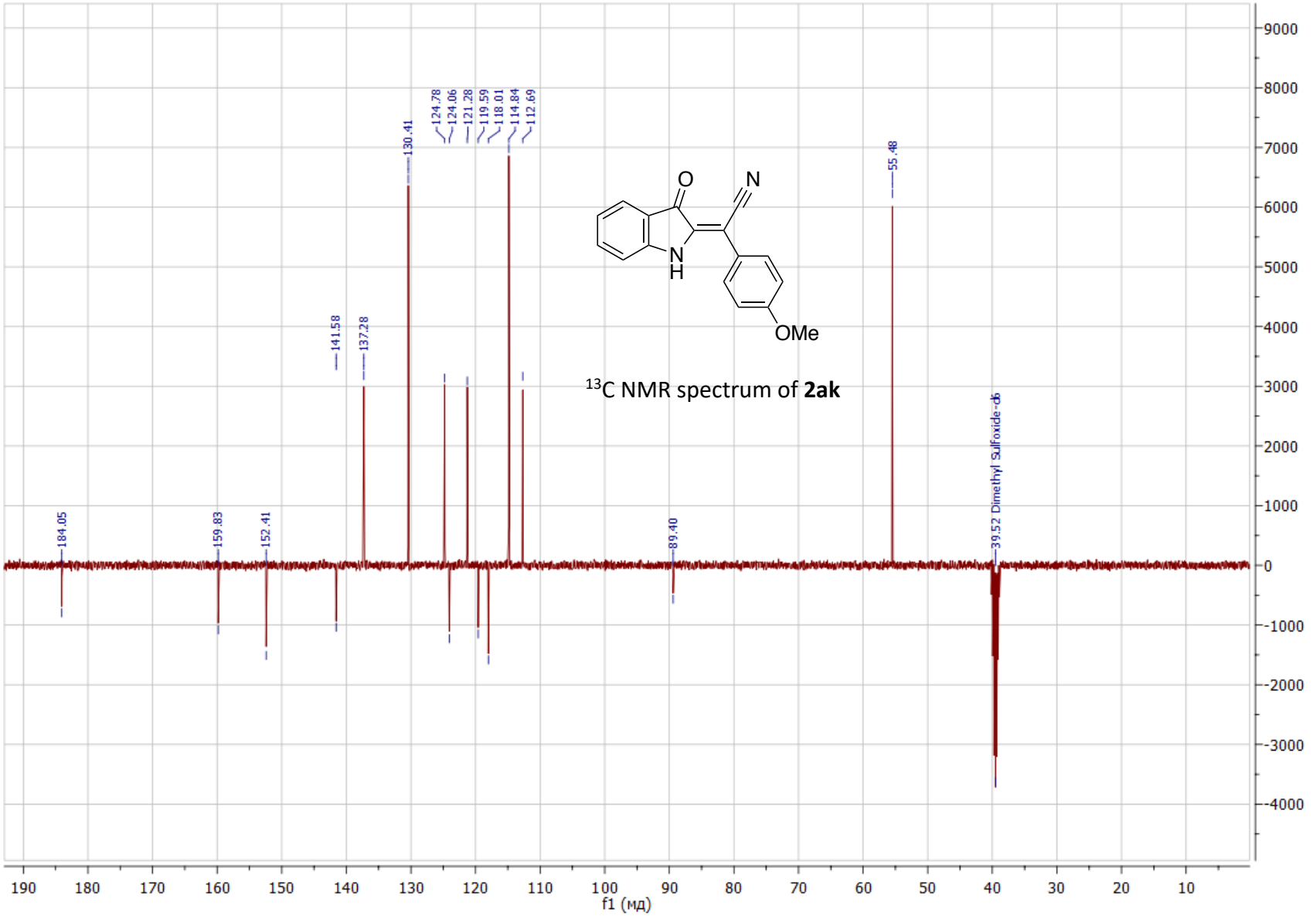
^{1}H NMR spectrum of **2aj**



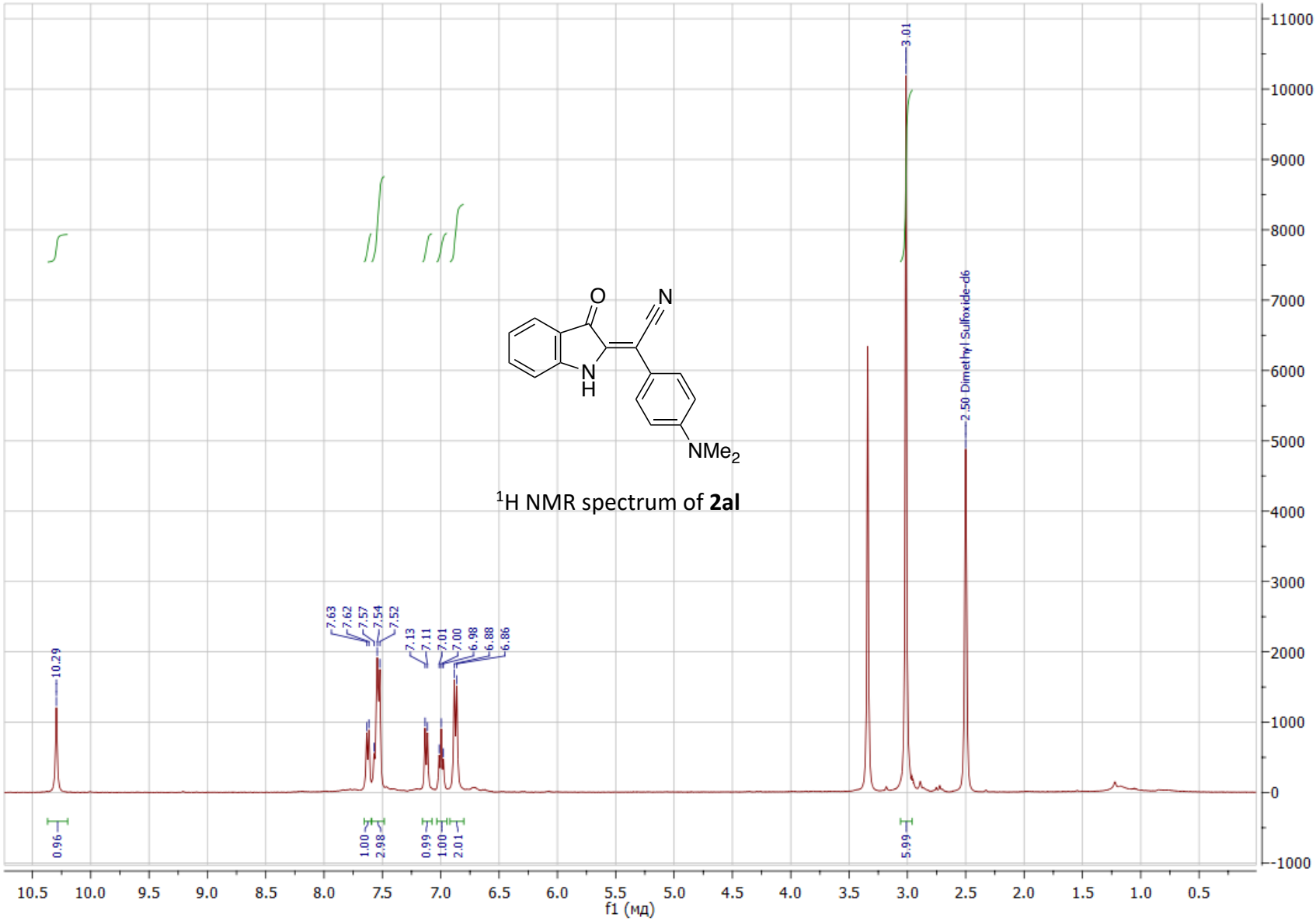
^{13}C NMR spectrum of **2aj**



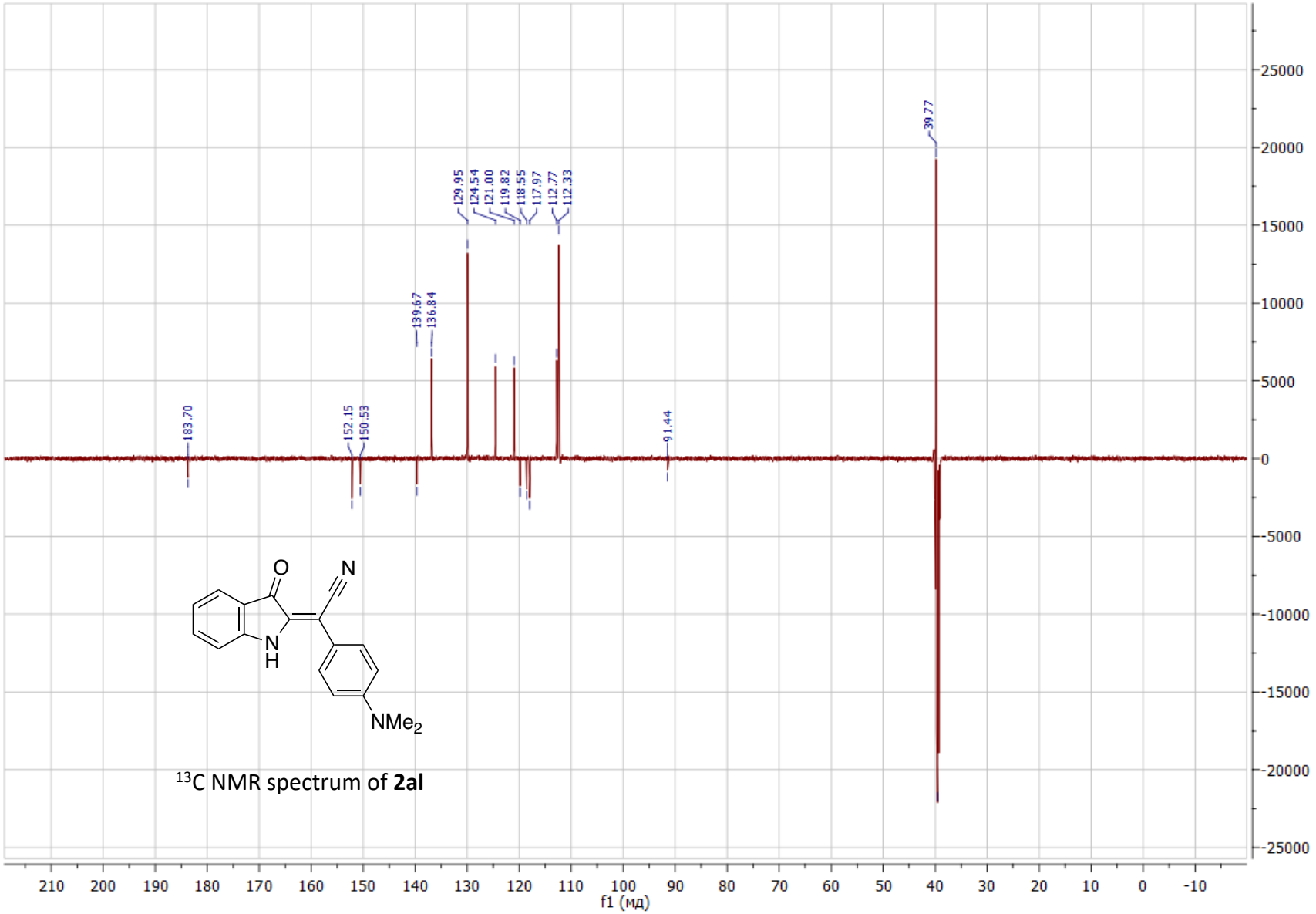
^{1}H NMR spectrum of **2ak**



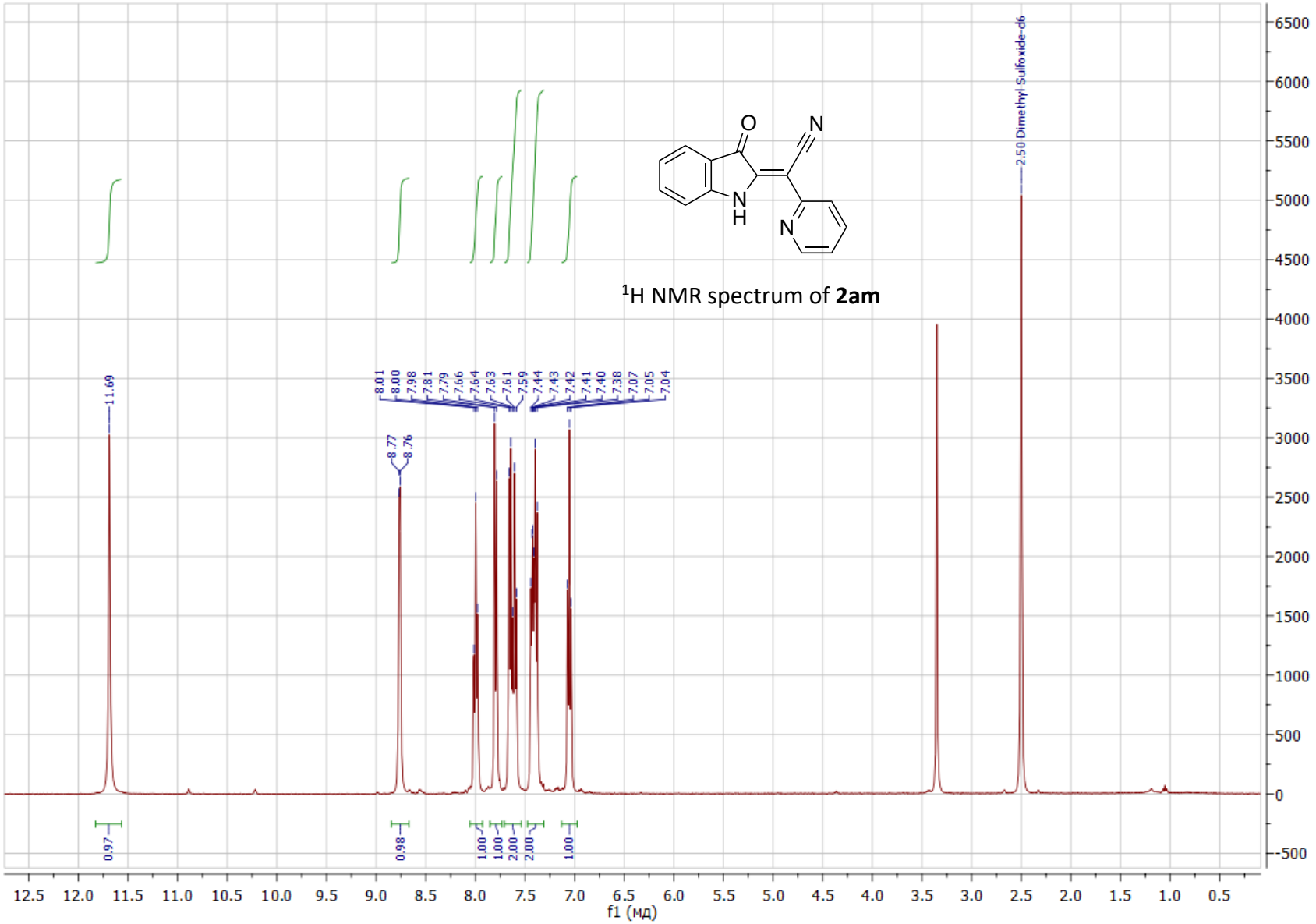
^{13}C NMR spectrum of **2ak**



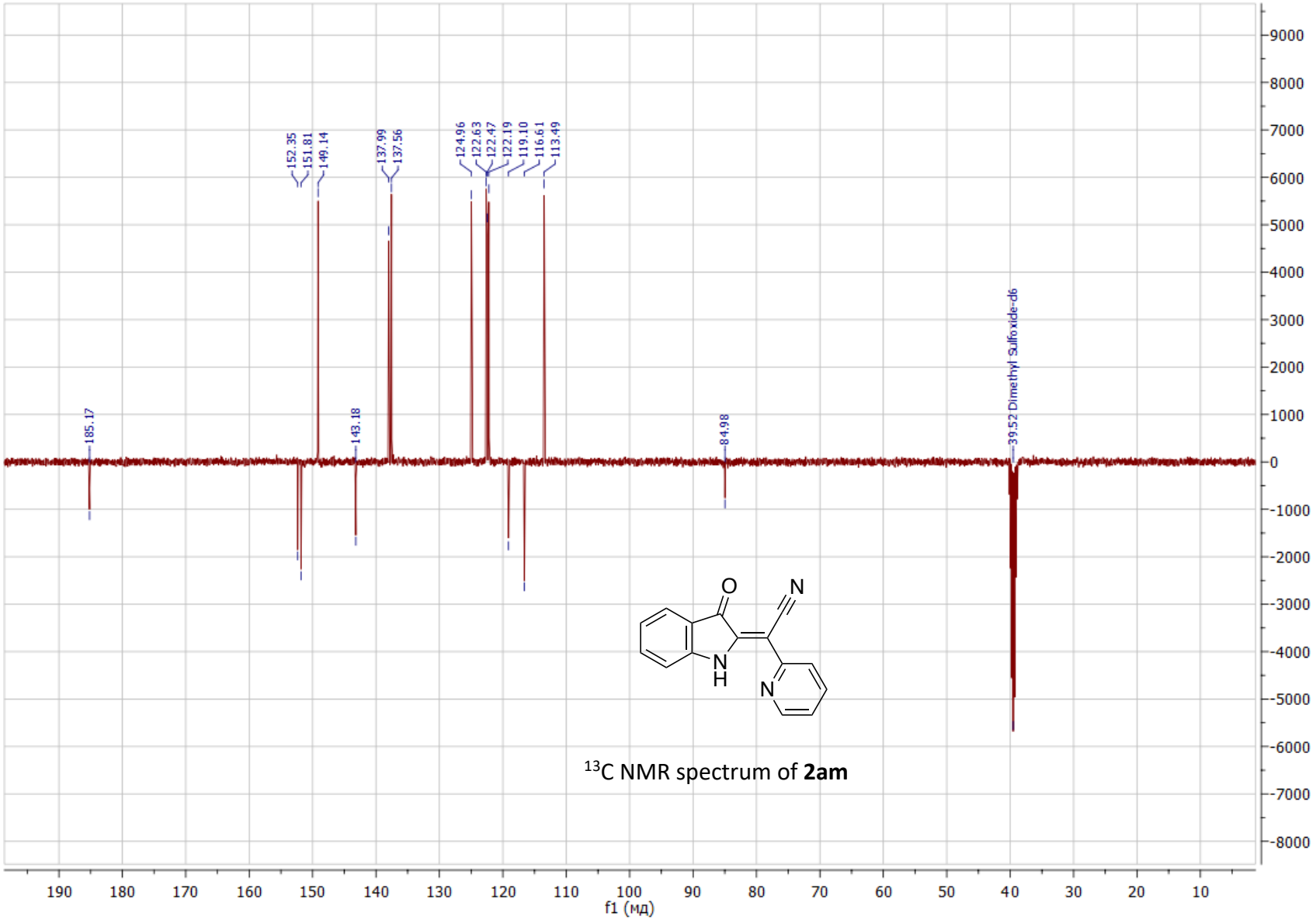
^{1}H NMR spectrum of **2al**



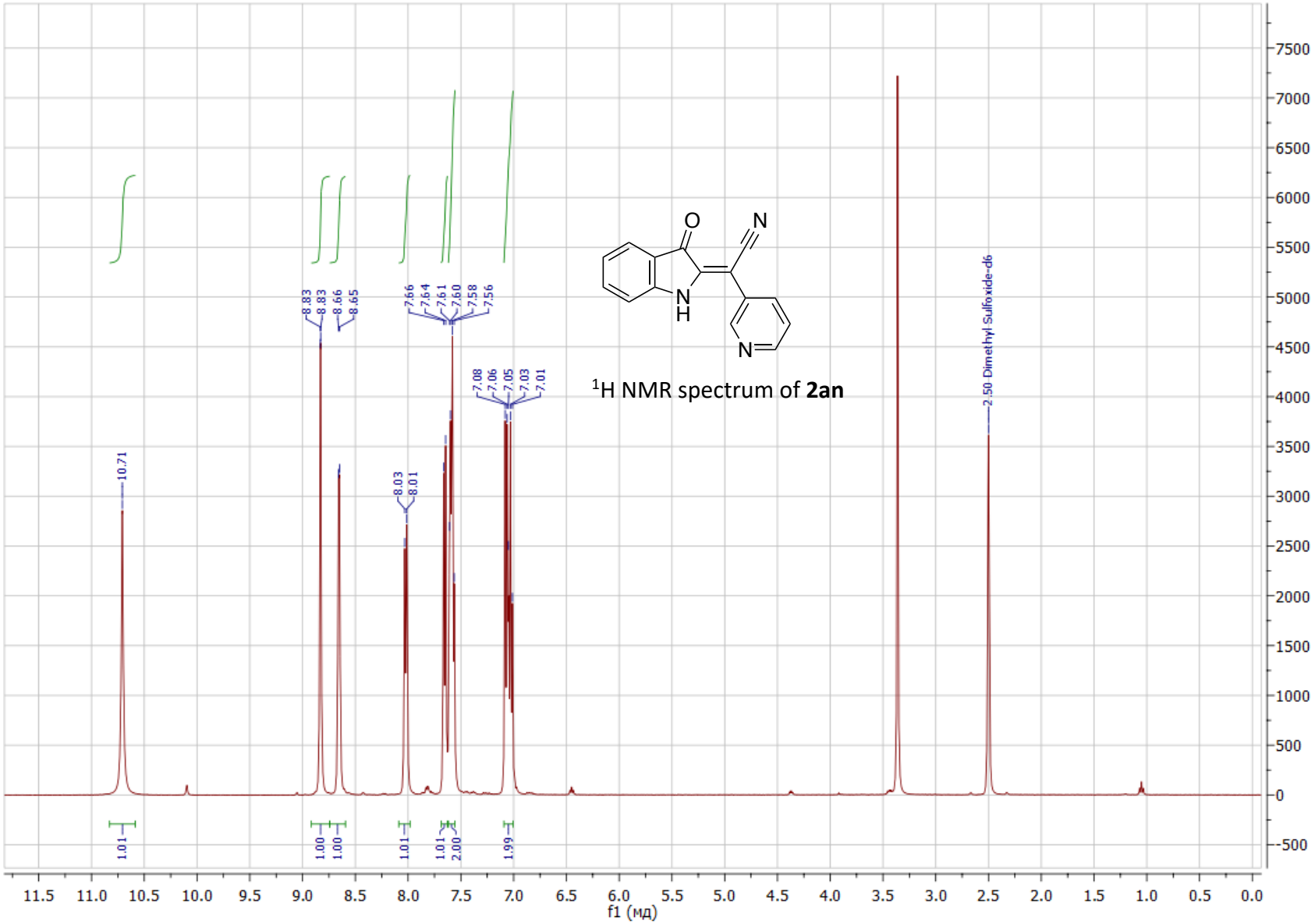
^{13}C NMR spectrum of **2al**

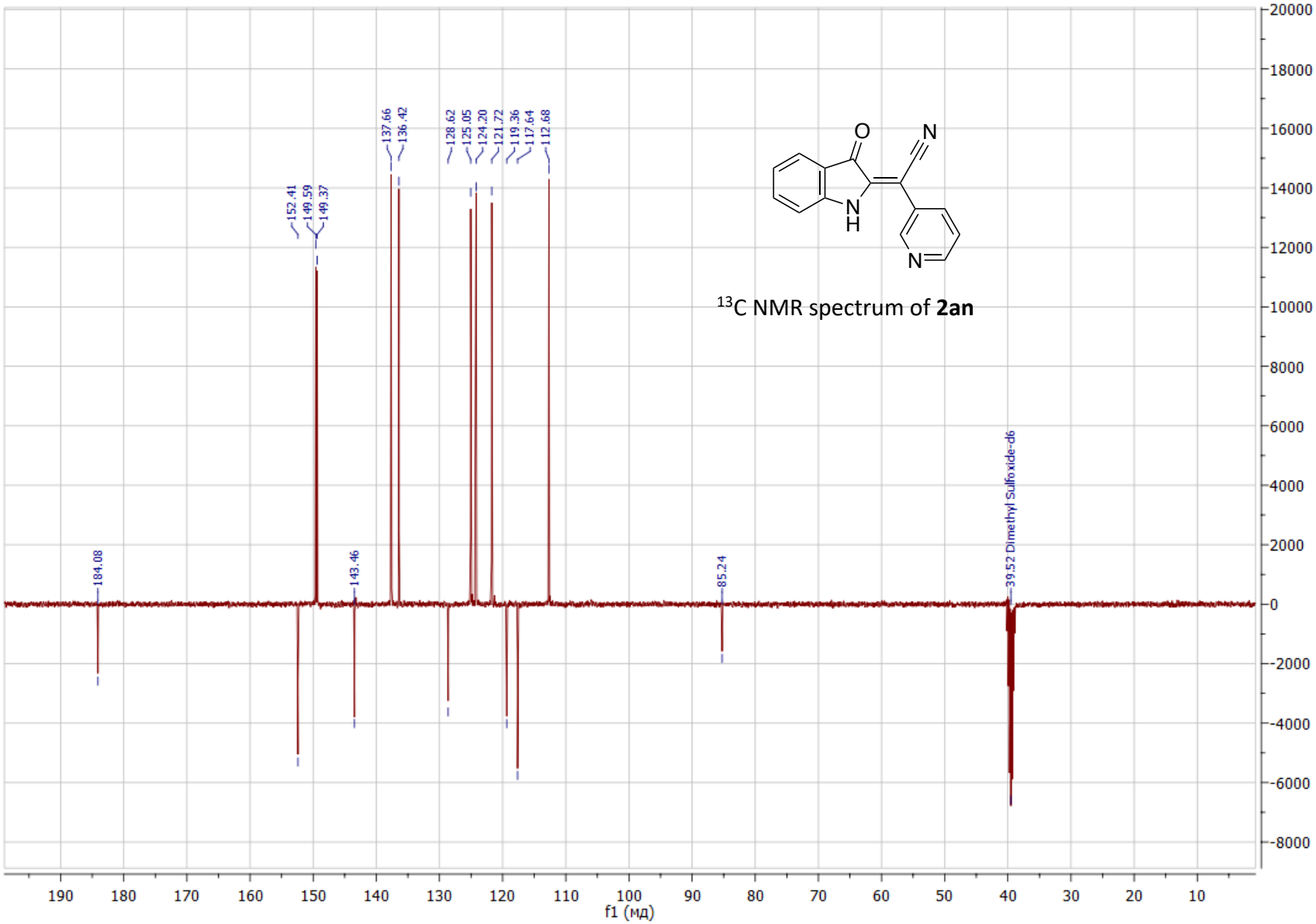


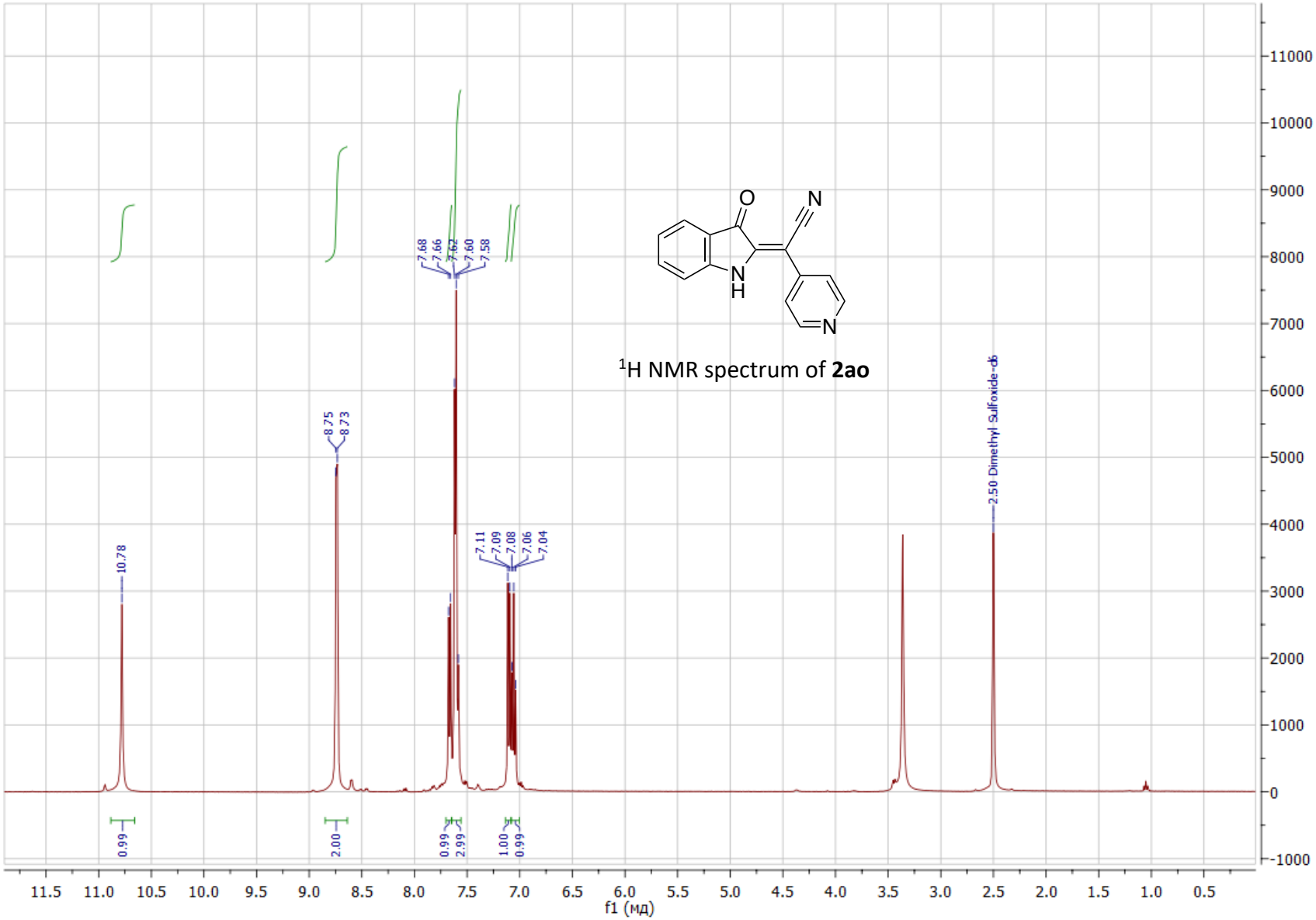
^{1}H NMR spectrum of **2am**

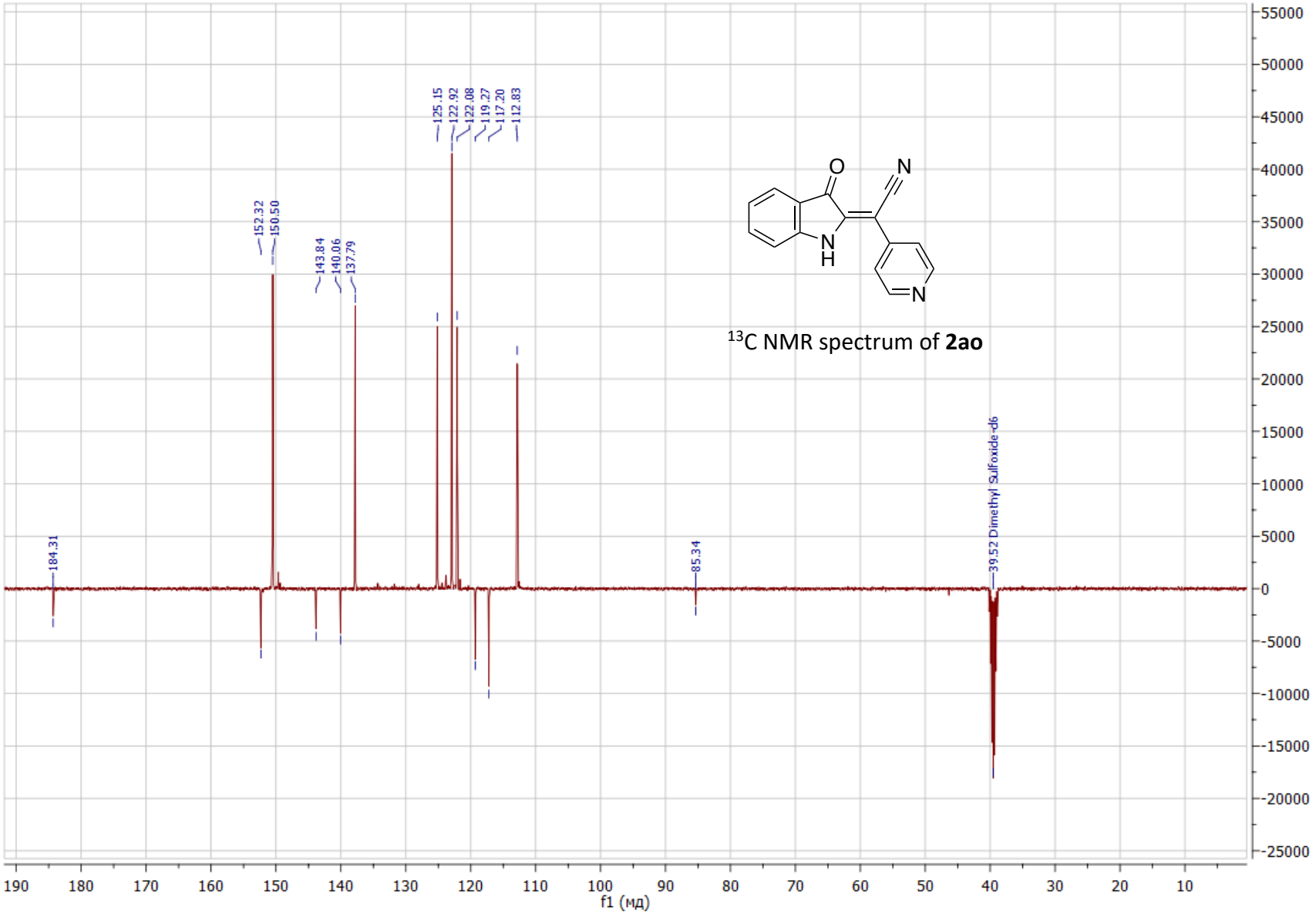


^{13}C NMR spectrum of **2am**

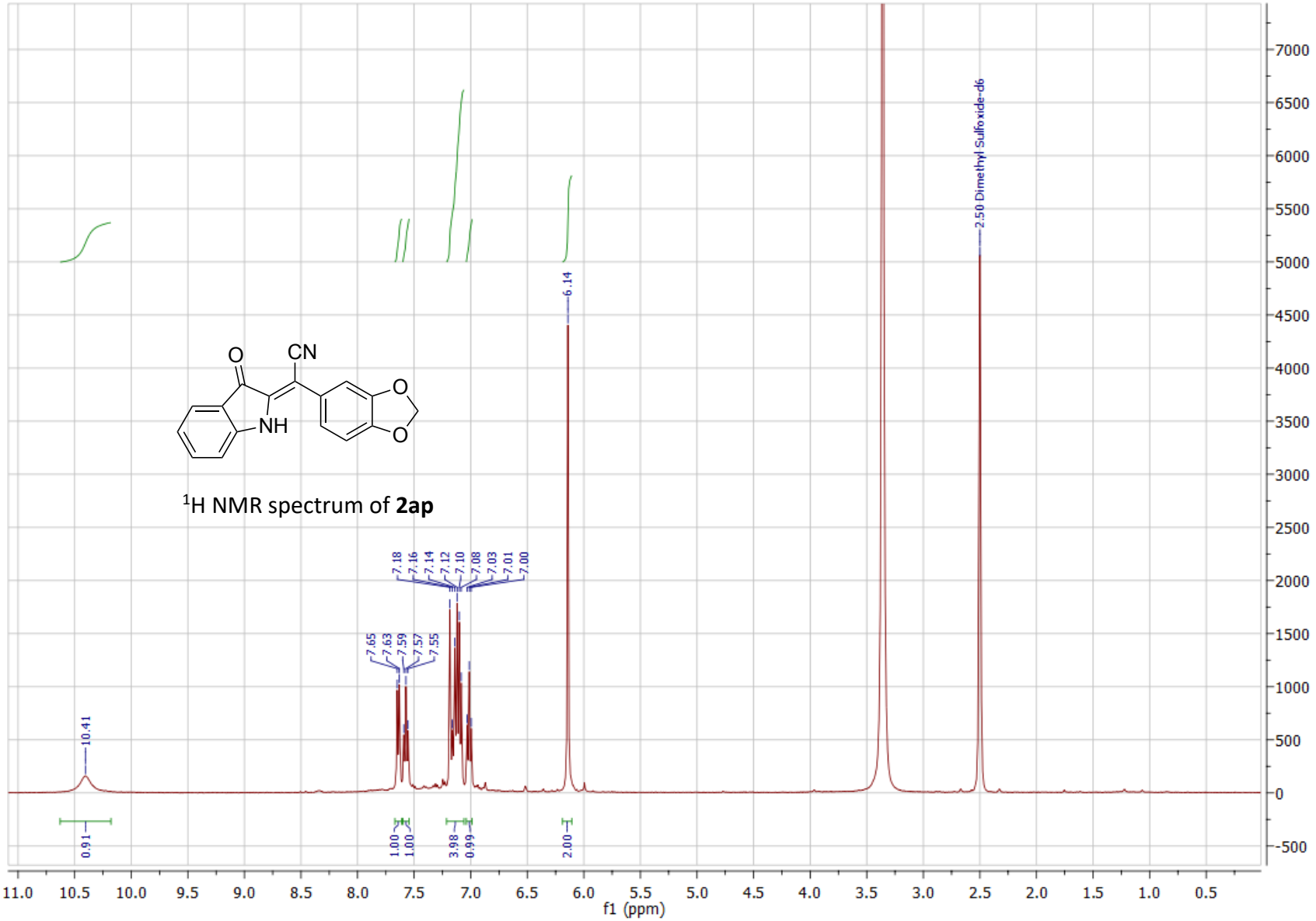

O
N
N
H
N
^{1}H NMR spectrum of **2an**
8.83
8.83
8.66
8.65
7.66
7.64
7.61
7.60
7.58
7.56
7.08
7.06
7.05
7.03
7.01
8.03
8.01
10.71
2.50 Dimethyl Sulfoxide-d6
1.01
1.00
1.00
1.01
1.01
2.00
1.99
f1 (мд)


O
N
N
H
N
^{13}C NMR spectrum of **2an**
152.41
149.59
149.37
137.66
136.42
128.62
125.05
124.20
121.72
119.36
117.64
112.68
184.08
143.46
85.24
39.52 Dimethyl Sulfoxide-d6
f1 (мд)

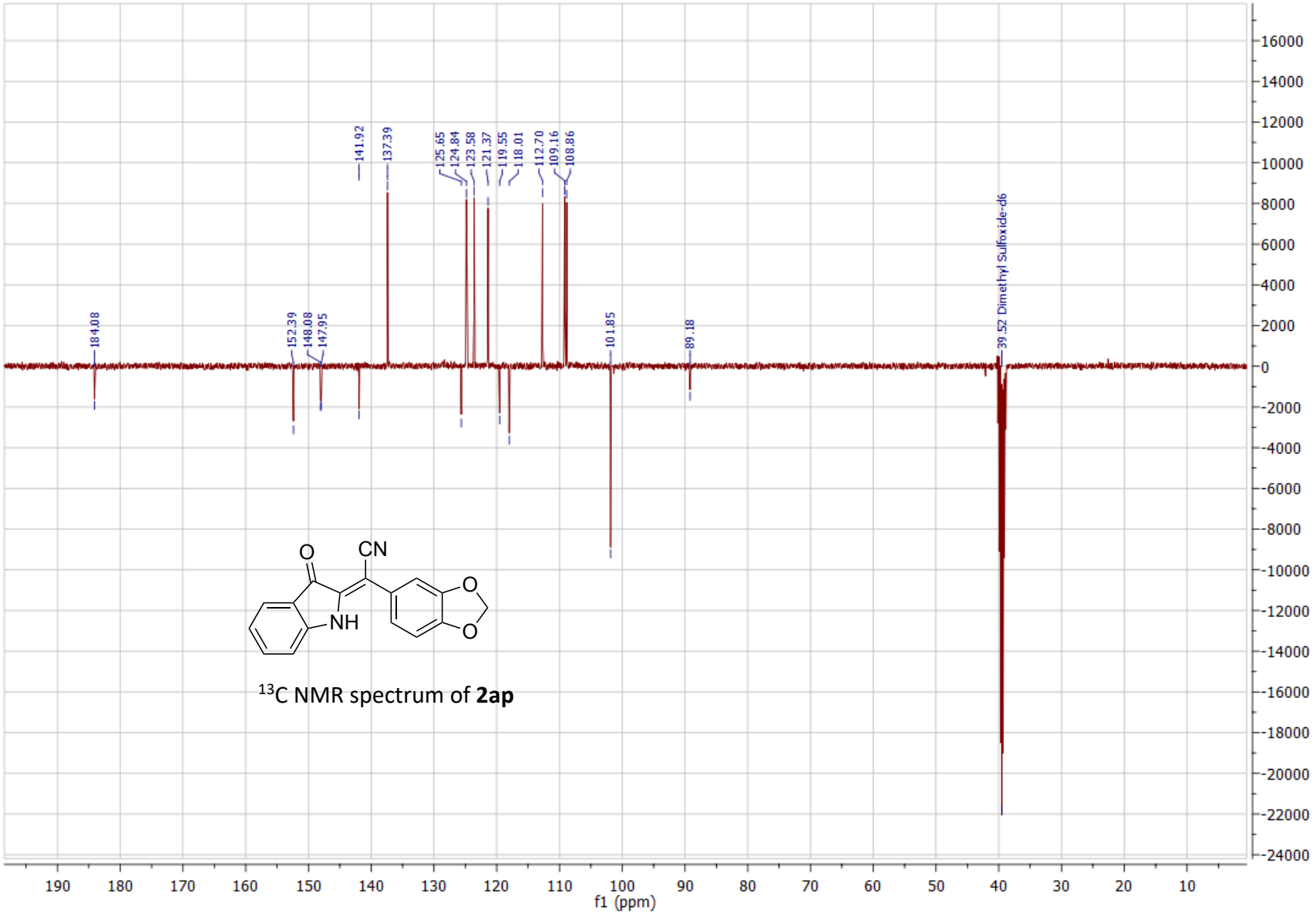

O
N
N
H
N
[1]H NMR spectrum of **2ao**
10.78
8.75
8.73
7.68
7.66
7.62
7.60
7.58
7.11
7.09
7.08
7.06
7.04
2.50 Dimethyl Sulfoxide-d6
0.99
2.00
0.99
2.99
1.00
0.99
f1 (мд)



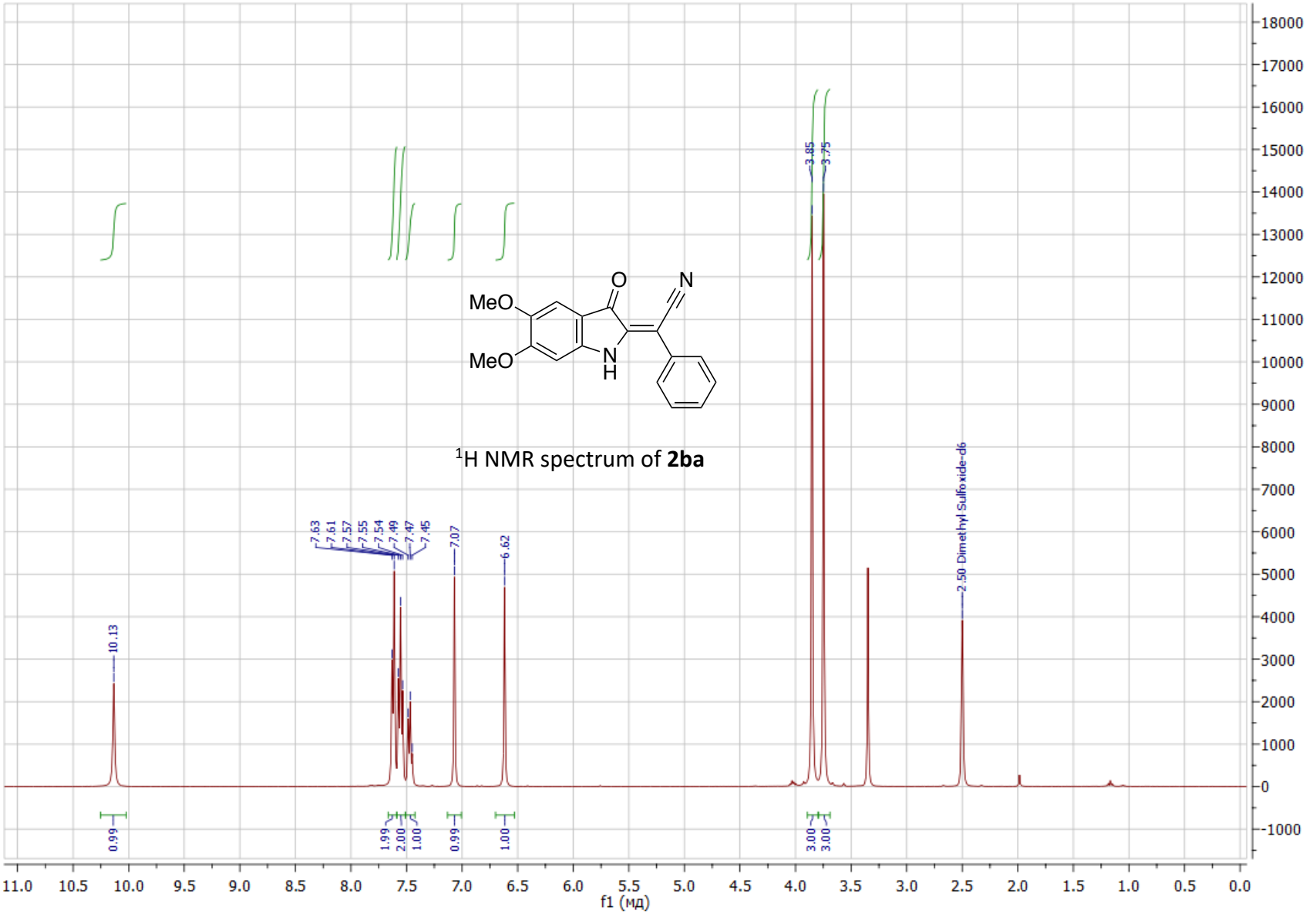
^{13}C NMR spectrum of **2ao**

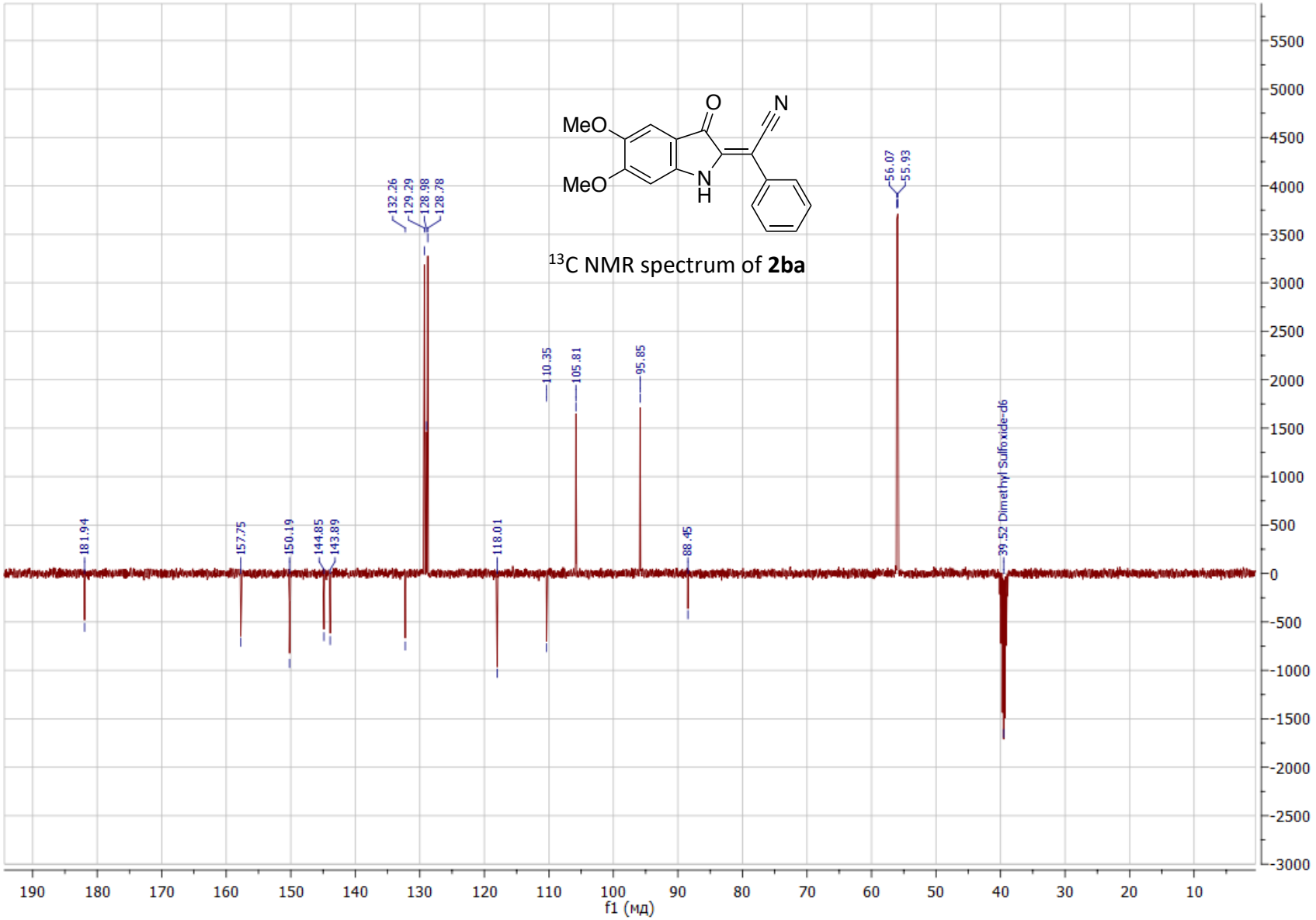


^{1}H NMR spectrum of **2ap**



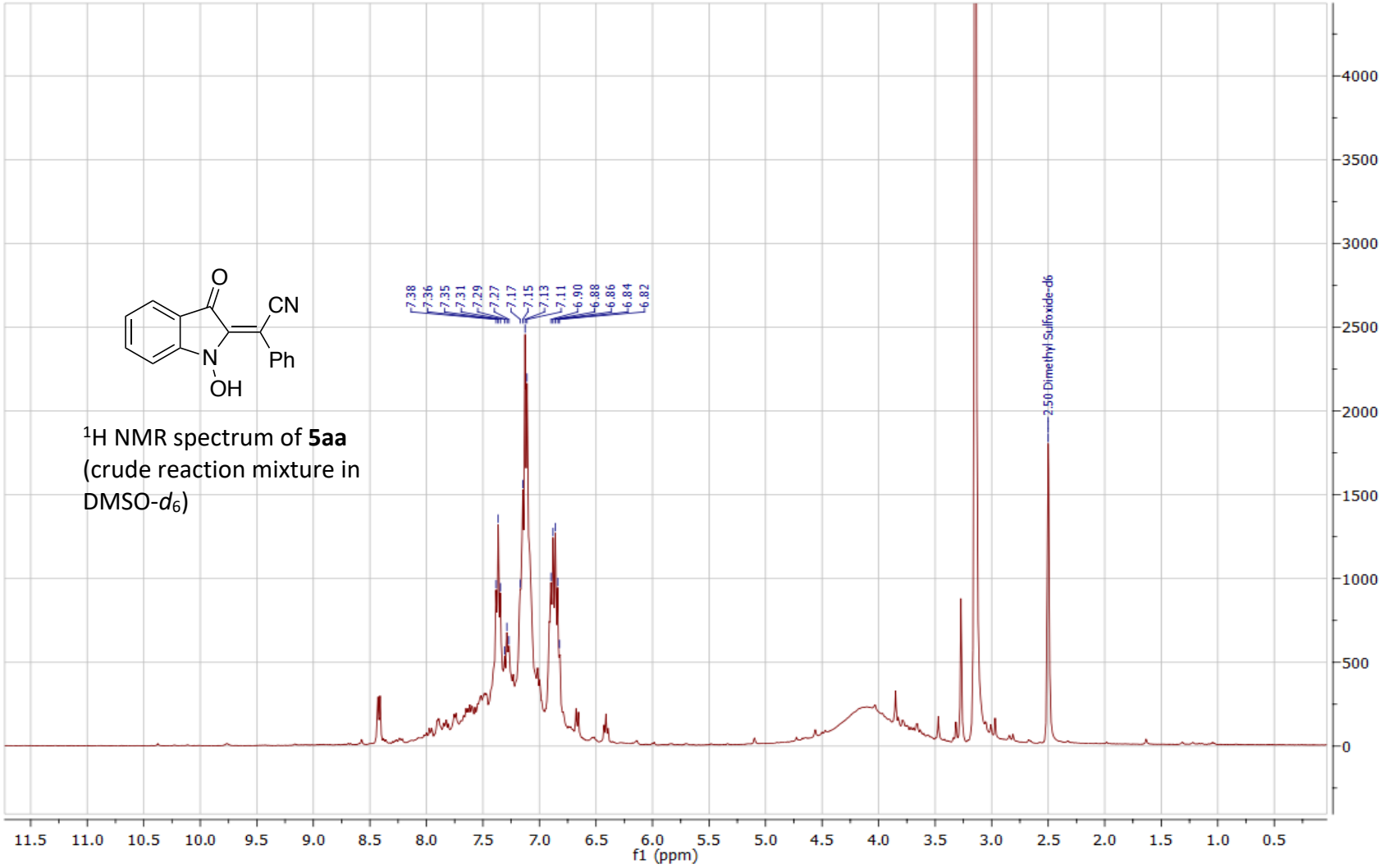
^{13}C NMR spectrum of **2ap**


MeO
MeO
O
N
N
H
^{1}H NMR spectrum of **2ba**
10.13
7.63
7.61
7.57
7.55
7.54
7.49
7.47
7.45
7.07
6.62
3.85
3.75
2.50 Dimethyl Sulfoxide-d6
0.99
1.99
2.00
1.00
0.99
1.00
3.00
3.00
f1 (мд)

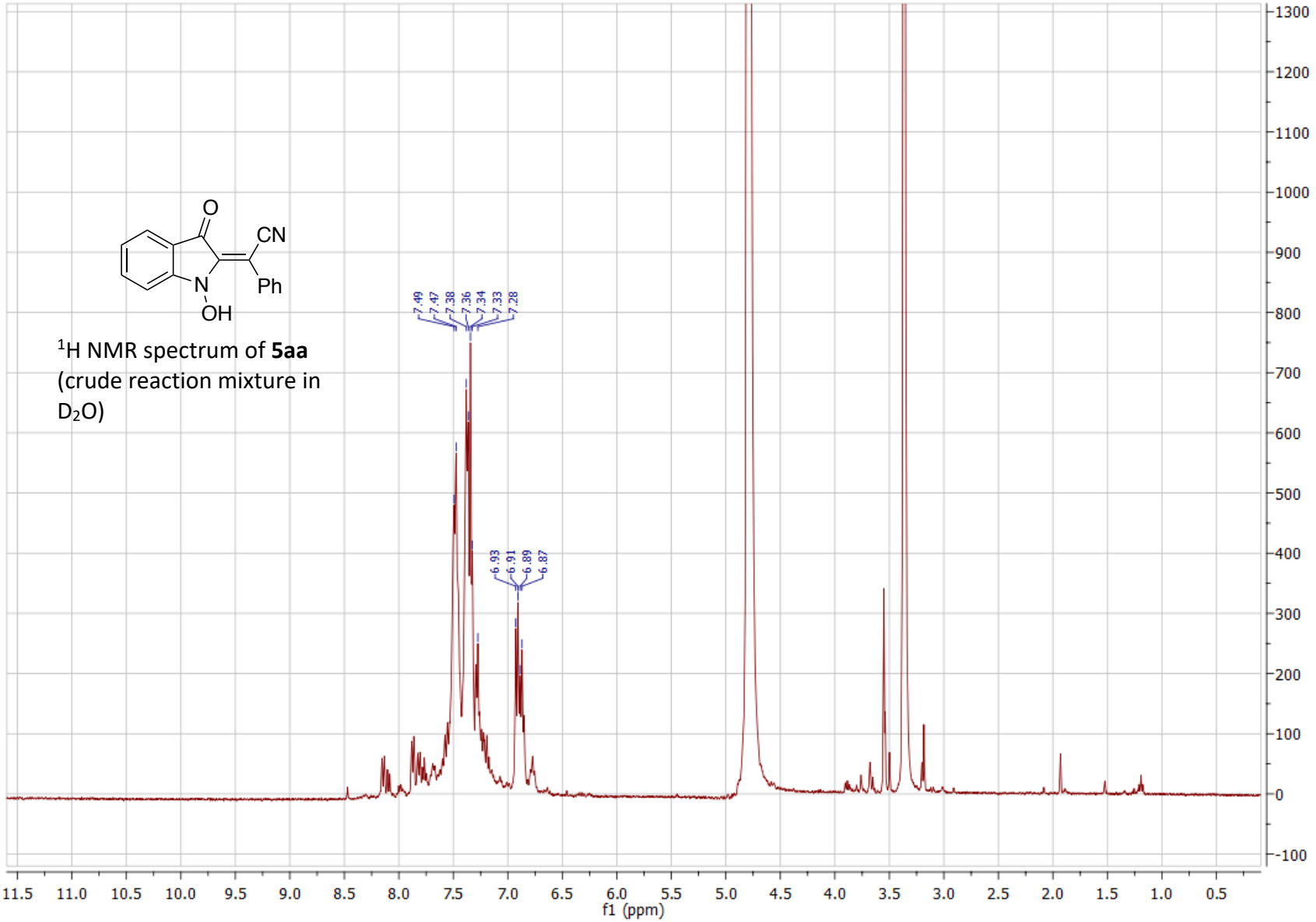


^{13}C NMR spectrum of **2ba**

^{1}H and spectral charts of intermediate 5aa (in crude reaction mixture)

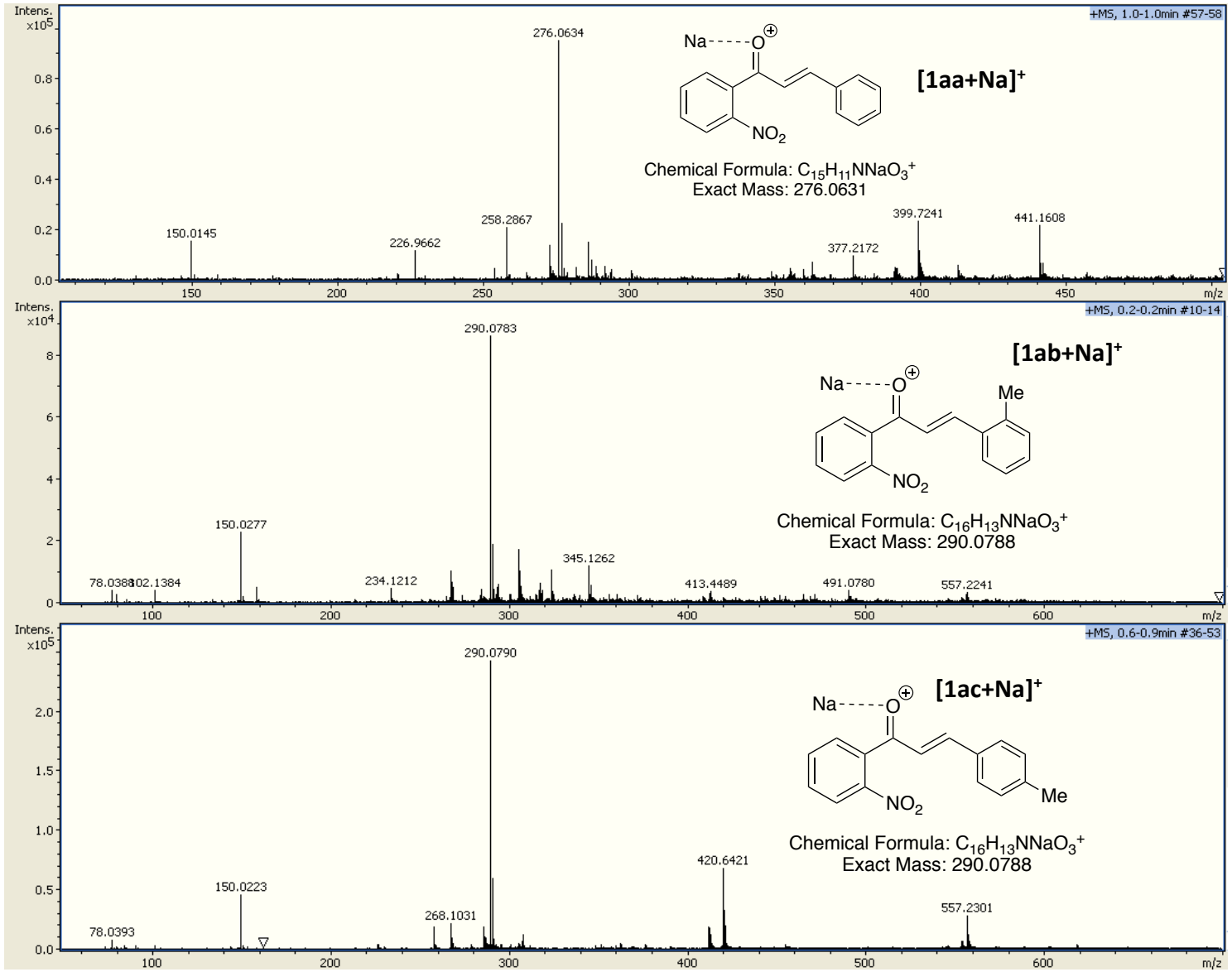


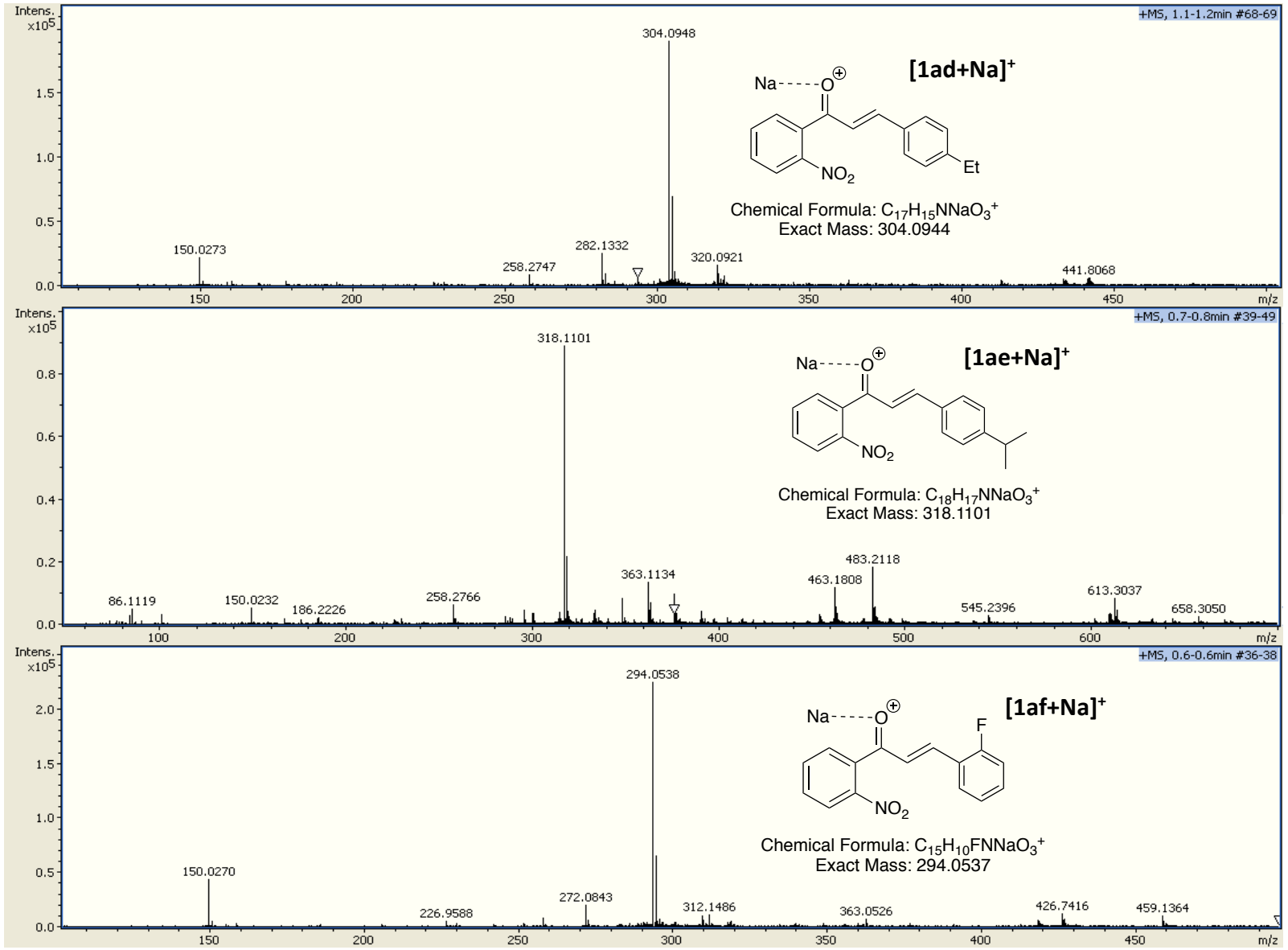
^{1}H NMR spectrum of **5aa** (crude reaction mixture in DMSO-d_6)

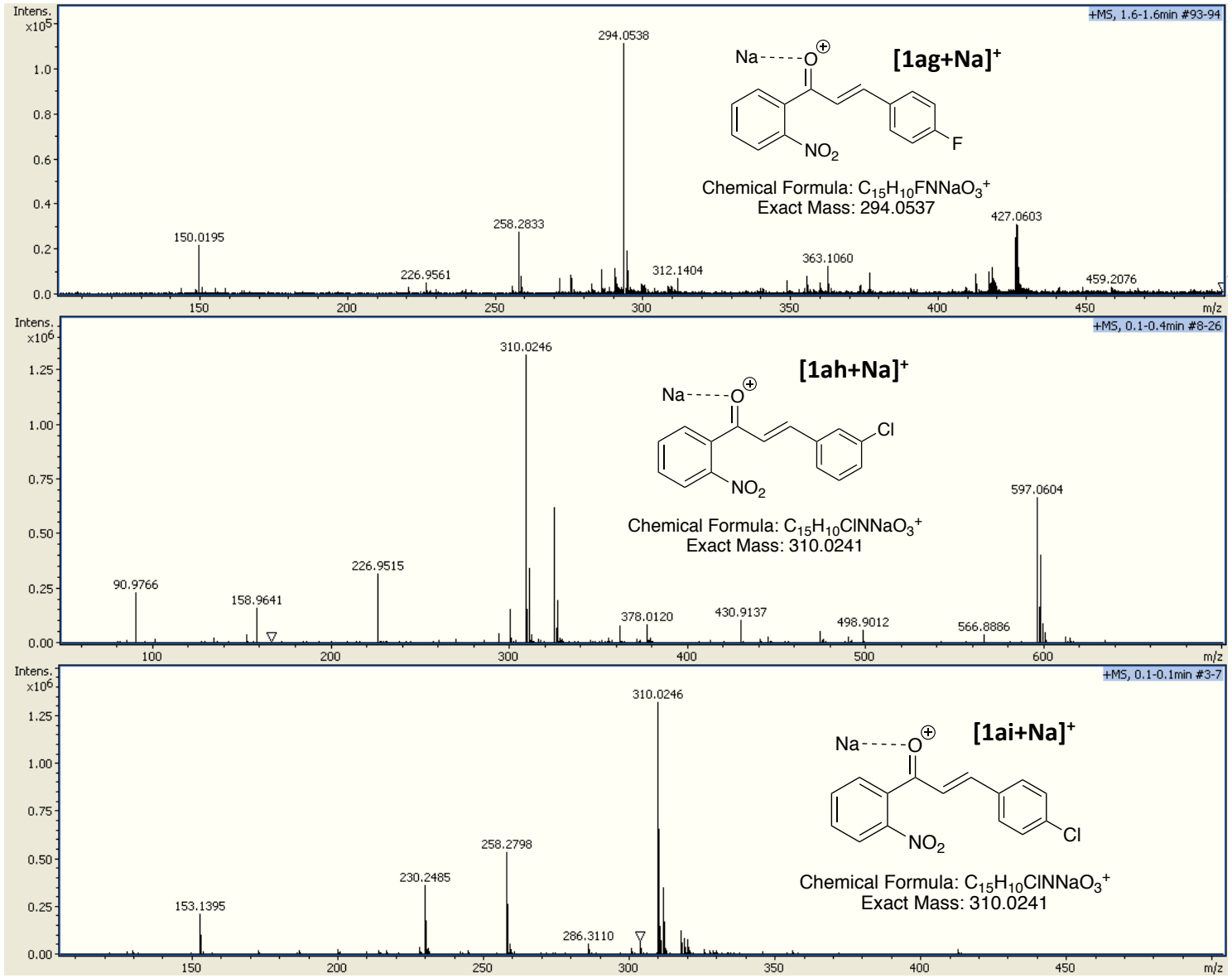


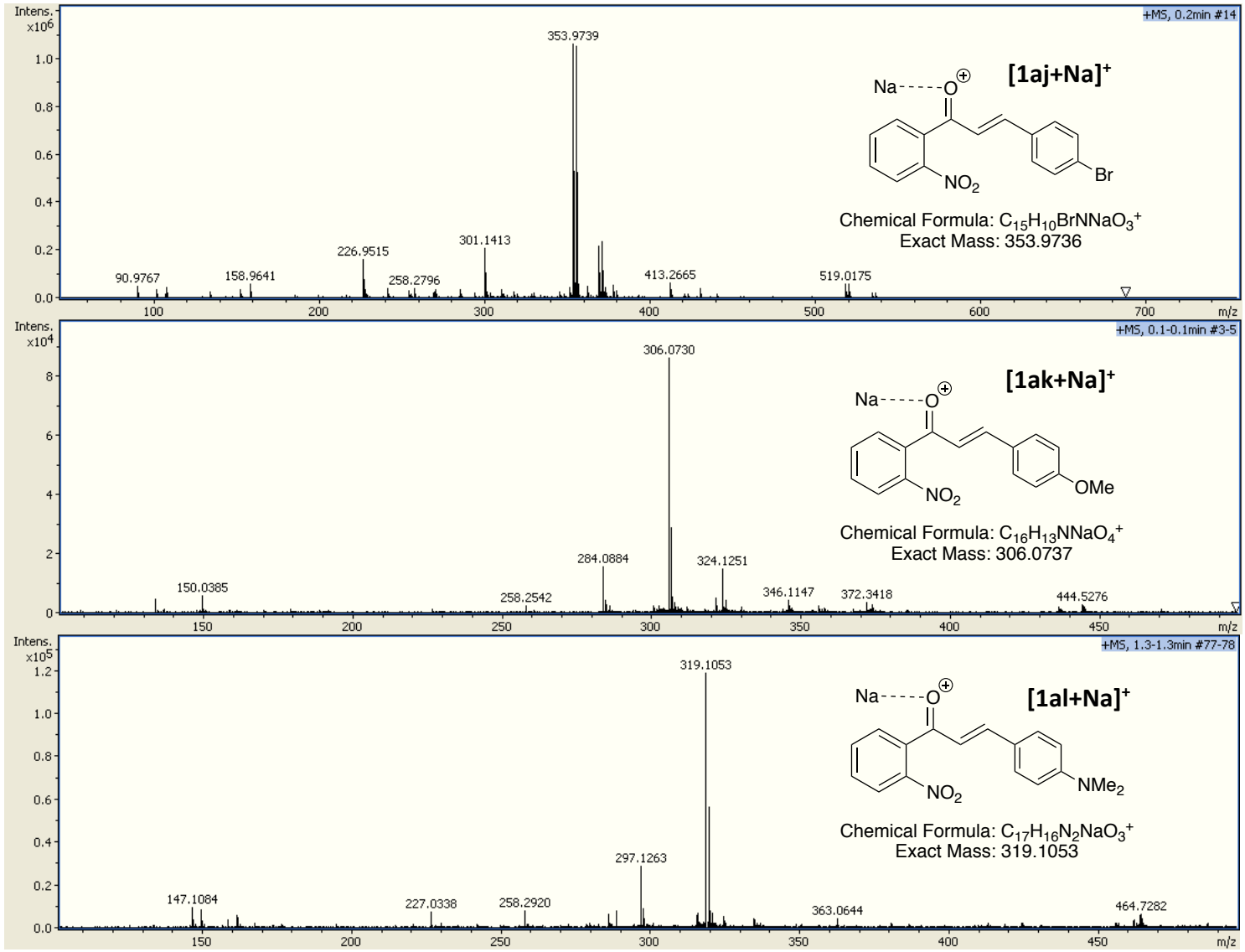
^{1}H NMR spectrum of **5aa** (crude reaction mixture in D_2O)

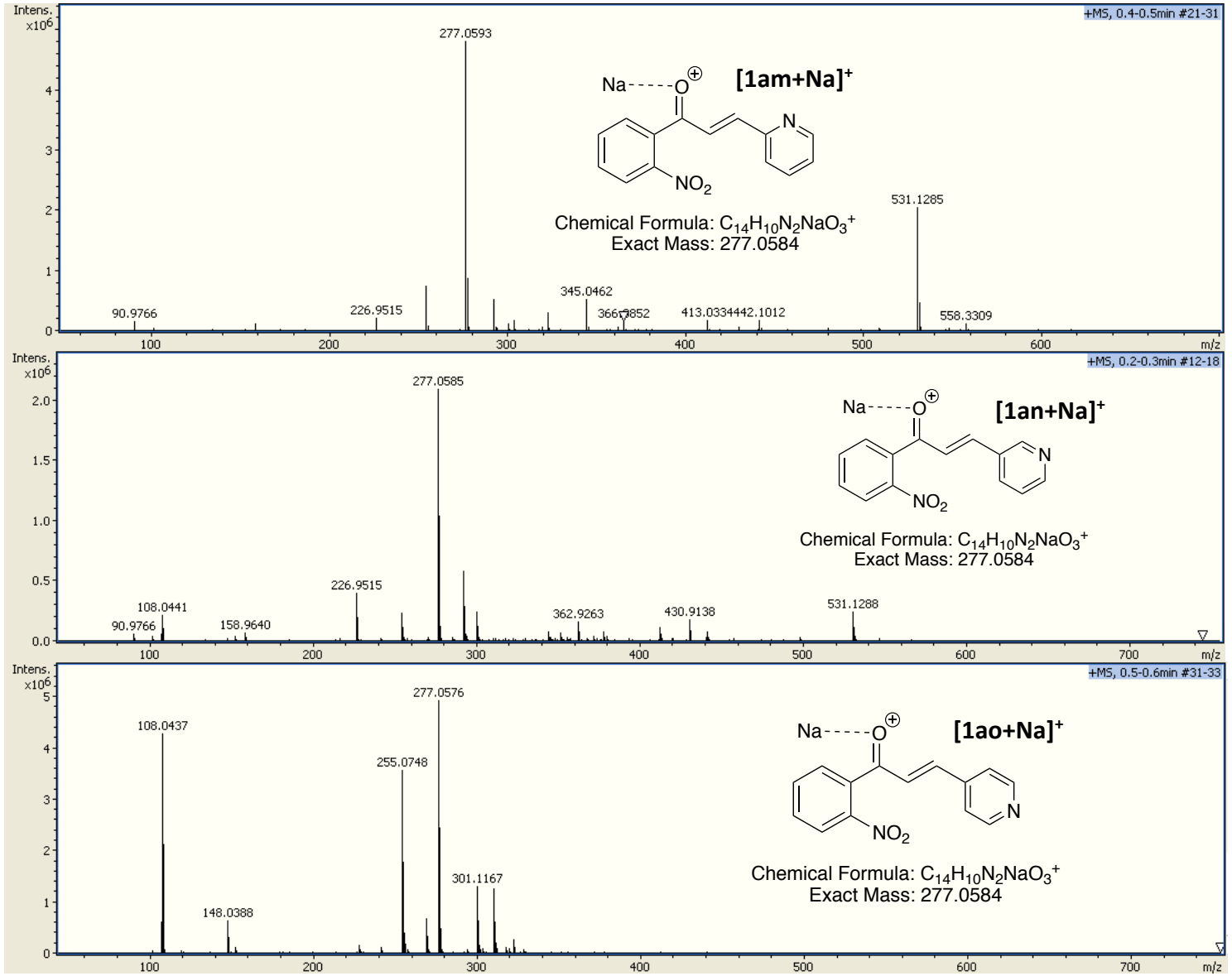
HRMS spectral charts for starting chalcones

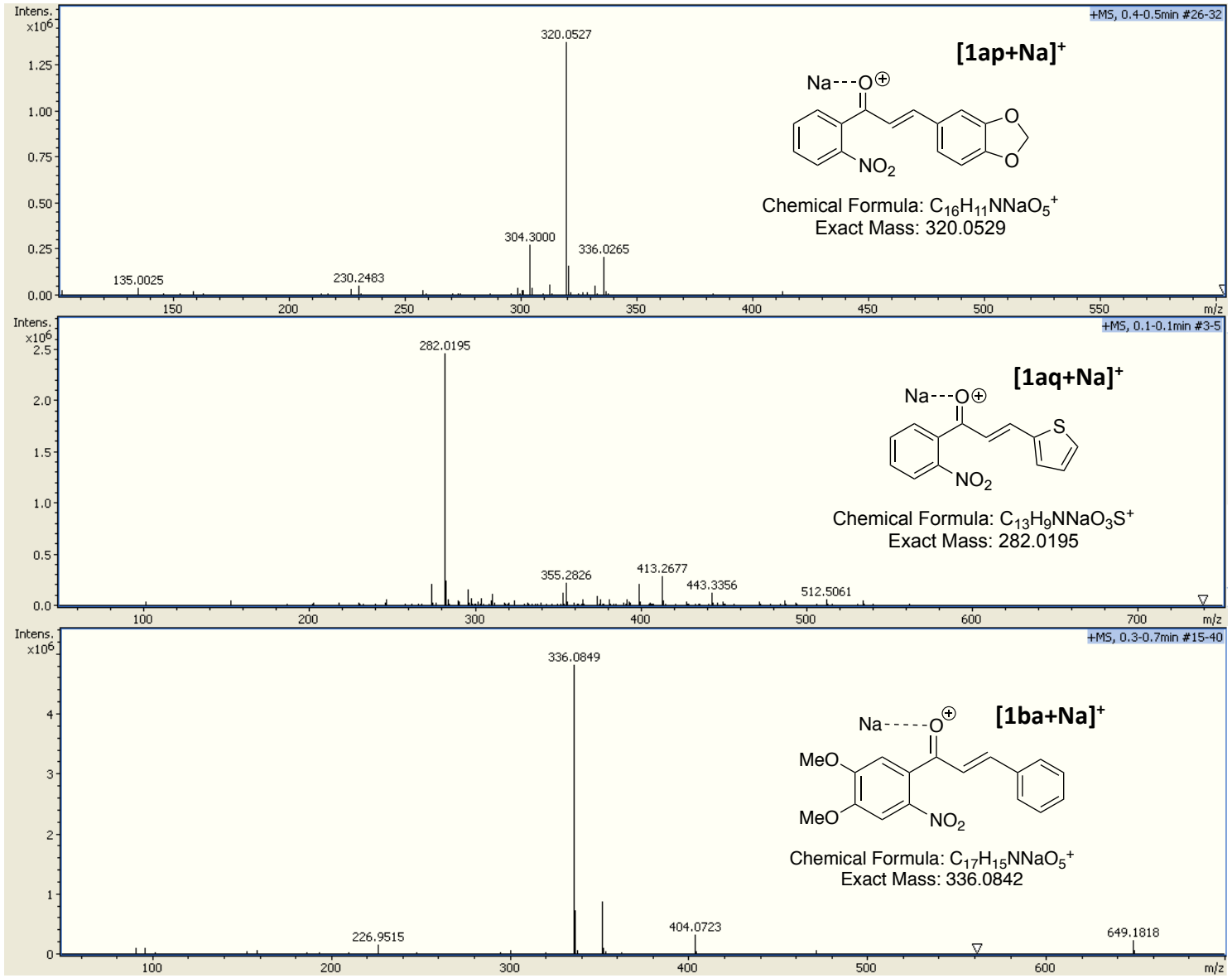



+MS, 1.1-1.2min #68-69
304.0948
[1ad+Na]+
Chemical Formula: C17H15NNaO3+
Exact Mass: 304.0944
150.0273
258.2747
282.1332
320.0921
441.8068
+MS, 0.7-0.8min #39-49
318.1101
[1ae+Na]+
Chemical Formula: C18H17NNaO3+
Exact Mass: 318.1101
86.1119
150.0232
186.2226
258.2766
363.1134
463.1808
483.2118
545.2396
613.3037
658.3050
+MS, 0.6-0.6min #36-38
294.0538
[1af+Na]+
Chemical Formula: C15H10FNNaO3+
Exact Mass: 294.0537
150.0270
226.9588
272.0843
312.1486
363.0526
426.7416
459.1364
Intens.
m/z

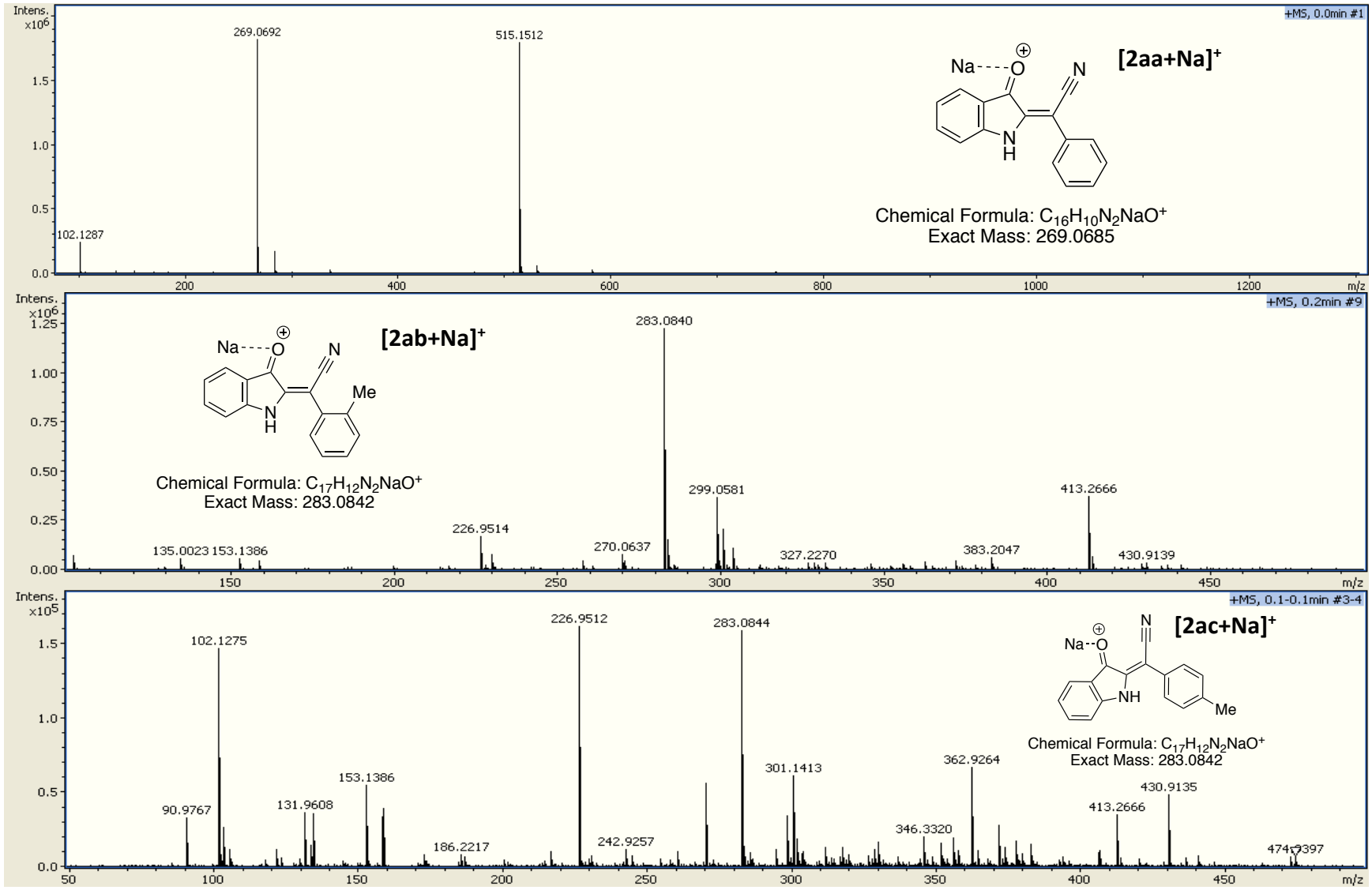

+MS, 1.6-1.6min #93-94
Intens.
x10^5
294.0538
[1ag+Na]+
Na
O
NO2
F
Chemical Formula: C15H10FNNaO3+
Exact Mass: 294.0537
150.0195
258.2833
226.9561
312.1404
363.1060
427.0603
459.2076
m/z
+MS, 0.1-0.4min #8-26
Intens.
x10^6
310.0246
[1ah+Na]+
Cl
Chemical Formula: C15H10ClNNaO3+
Exact Mass: 310.0241
597.0604
226.9515
90.9766
158.9641
378.0120
430.9137
498.9012
566.8886
+MS, 0.1-0.1min #3-7
Intens.
x10^6
310.0246
[1ai+Na]+
Chemical Formula: C15H10ClNNaO3+
Exact Mass: 310.0241
258.2798
230.2485
153.1395
286.3110

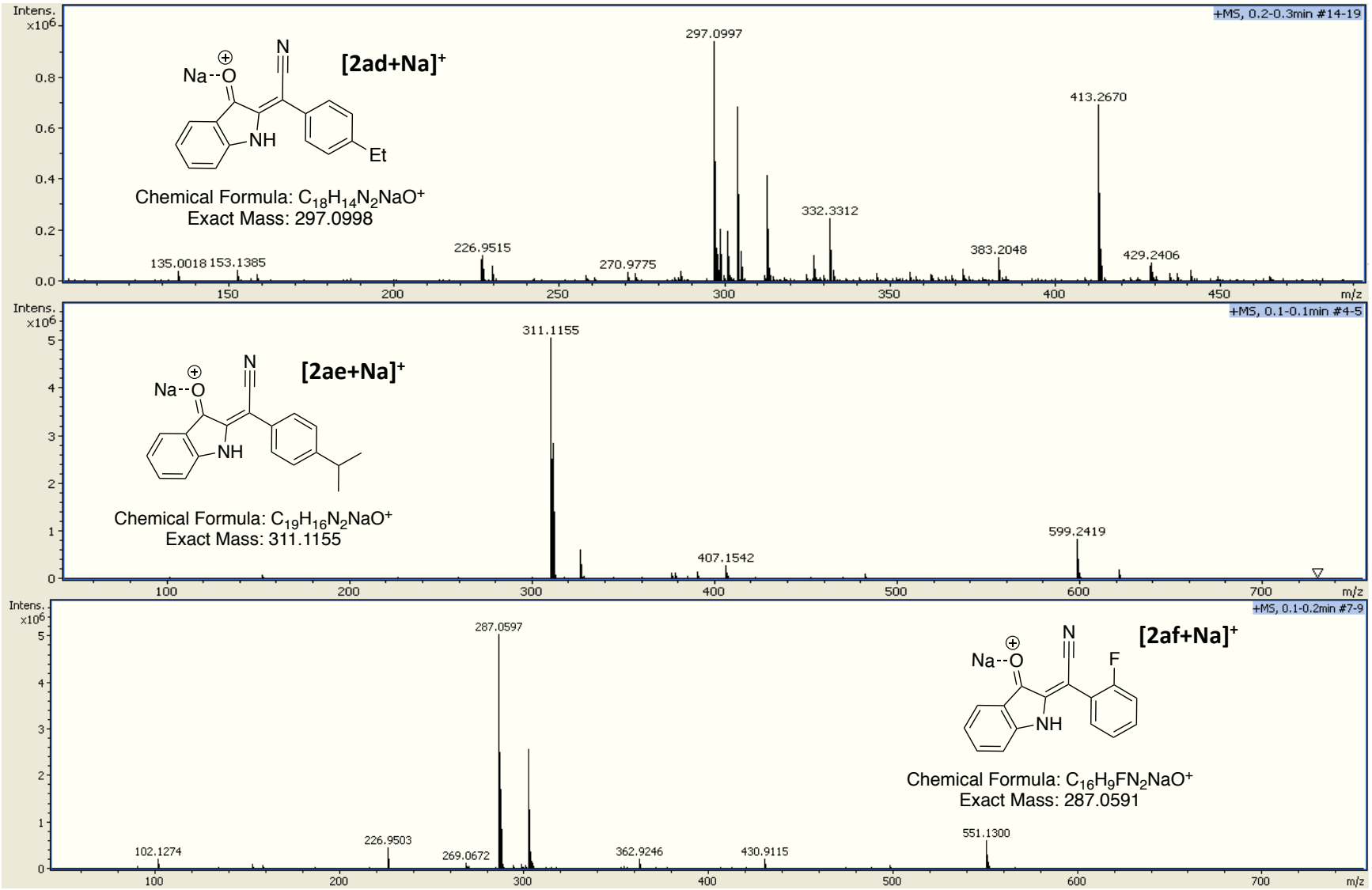

+MS, 0.2min #14
353.9739
301.1413
226.9515
90.9767
158.9641
258.2796
413.2665
519.0175
[1aj+Na]+
Chemical Formula: C15H10BrNNaO3+
Exact Mass: 353.9736
+MS, 0.1-0.1min #3-5
306.0730
284.0884
324.1251
150.0385
258.2542
346.1147
372.3418
444.5276
[1ak+Na]+
Chemical Formula: C16H13NNaO4+
Exact Mass: 306.0737
+MS, 1.3-1.3min #77-78
319.1053
297.1263
147.1084
227.0338
258.2920
363.0644
464.7282
[1al+Na]+
Chemical Formula: C17H16N2NaO3+
Exact Mass: 319.1053

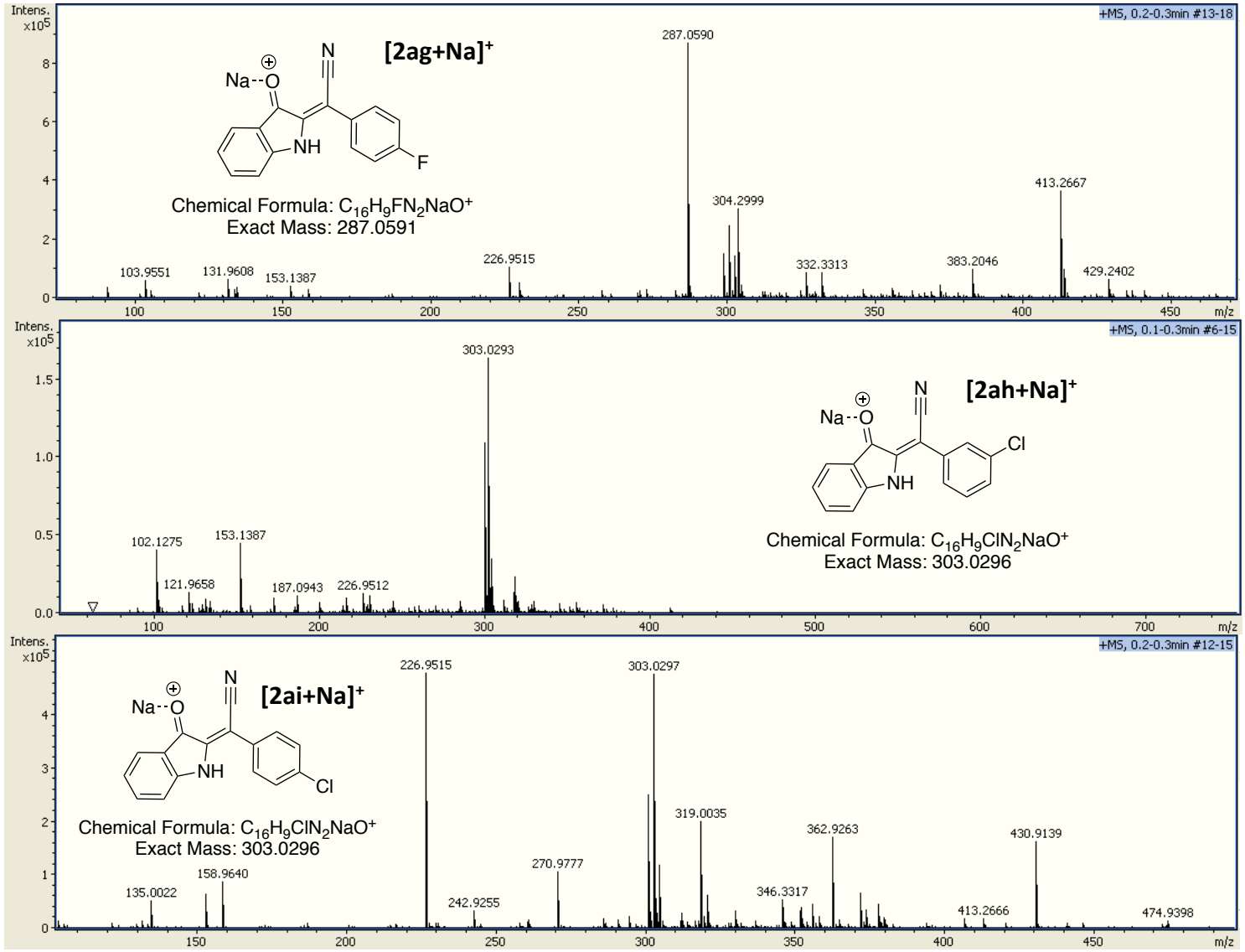

Intens.
x10^6
+MS, 0.4-0.5min #21-31
277.0593
[1am+Na]+
Na
O
N
NO2
Chemical Formula: C14H10N2NaO3+
Exact Mass: 277.0584
531.1285
345.0462
90.9766
226.9515
366.3852
413.0334442.1012
558.3309
m/z
+MS, 0.2-0.3min #12-18
277.0585
[1an+Na]+
Chemical Formula: C14H10N2NaO3+
Exact Mass: 277.0584
226.9515
108.0441
90.9766
158.9640
362.9263
430.9138
531.1288
+MS, 0.5-0.6min #31-33
277.0576
108.0437
[1ao+Na]+
255.0748
Chemical Formula: C14H10N2NaO3+
Exact Mass: 277.0584
301.1167
148.0388

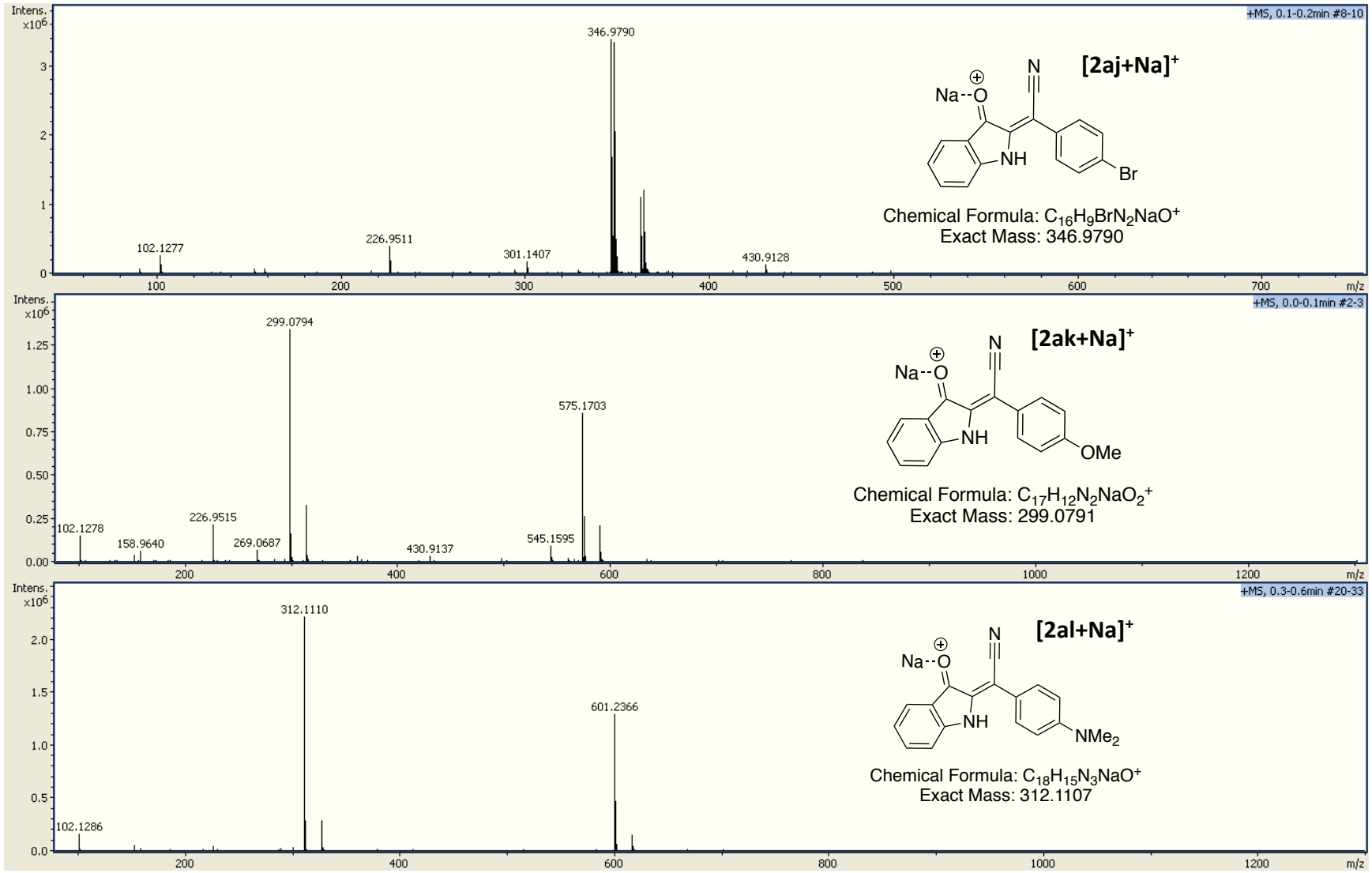

Intens.
x10^6
+MS, 0.4-0.5min #26-32
320.0527
304.3000
336.0265
135.0025
230.2483
[1ap+Na]+
Na---O
NO2
O
O
Chemical Formula: C16H11NNaO5+
Exact Mass: 320.0529
m/z
+MS, 0.1-0.1min #3-5
282.0195
355.2826
413.2677
443.3356
512.5061
[1aq+Na]+
Na---O
S
NO2
Chemical Formula: C13H9NNaO3S+
Exact Mass: 282.0195
+MS, 0.3-0.7min #15-40
336.0849
226.9515
404.0723
649.1818
[1ba+Na]+
Na-----O
MeO
MeO
NO2
Chemical Formula: C17H15NNaO5+
Exact Mass: 336.0842

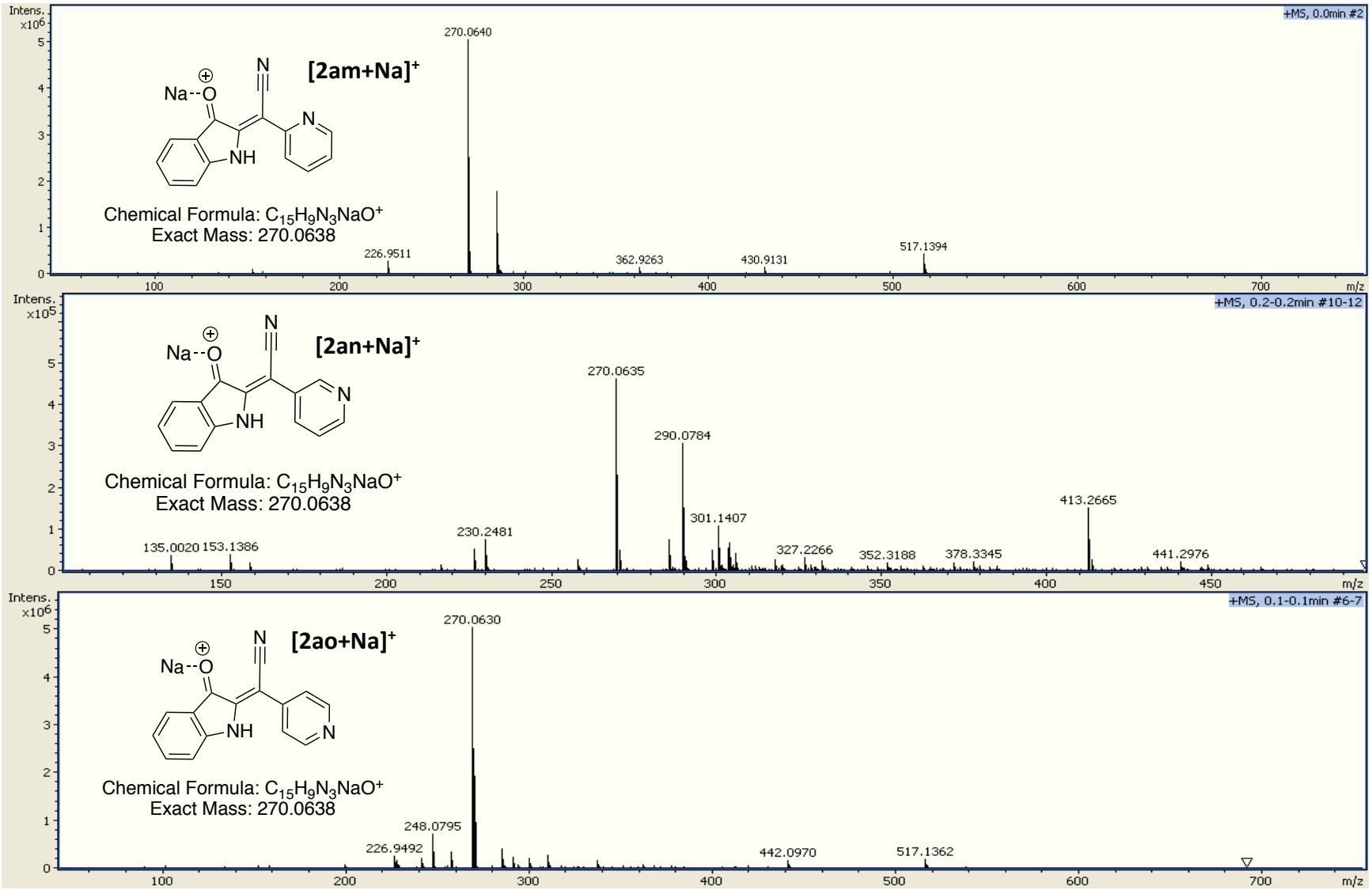
HRMS spectral charts for (*E*)-2-(3-oxoindolin-2-ylidene)-2-arylacetonitriles

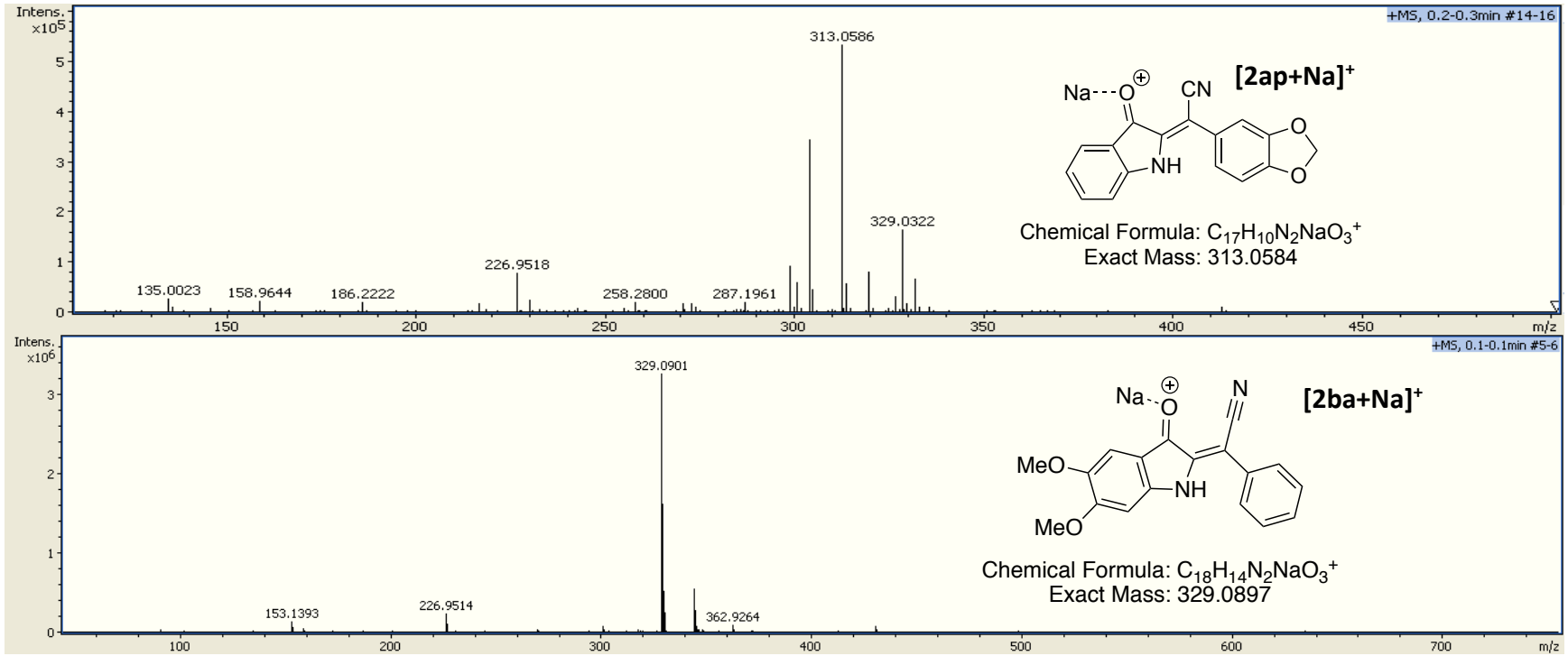



Intens.
x10^6
+MS, 0.2-0.3min #14-19
[2ad+Na]+
Chemical Formula: C18H14N2NaO+
Exact Mass: 297.0998
297.0997
413.2670
332.3312
226.9515
383.2048
429.2406
270.9775
135.0018
153.1385
m/z
+MS, 0.1-0.1min #4-5
[2ae+Na]+
Chemical Formula: C19H16N2NaO+
Exact Mass: 311.1155
311.1155
599.2419
407.1542
+MS, 0.1-0.2min #7-9
[2af+Na]+
Chemical Formula: C16H9FN2NaO+
Exact Mass: 287.0591
287.0597
551.1300
226.9503
102.1274
269.0672
362.9246
430.9115

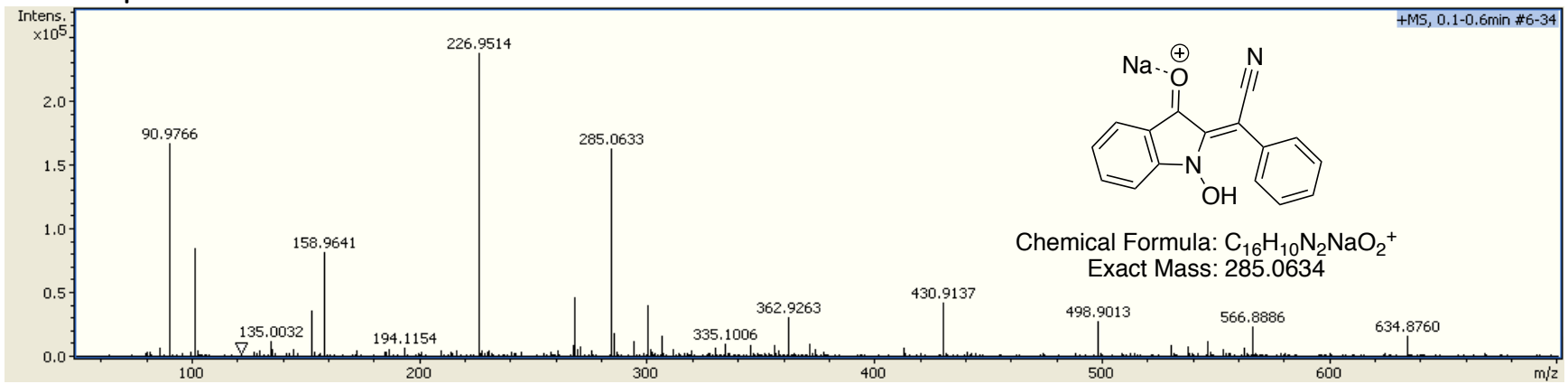

Intens.
x10^5
+MS, 0.2-0.3min #13-18
[2ag+Na]+
Chemical Formula: C16H9FN2NaO+
Exact Mass: 287.0591
287.0590
304.2999
413.2667
226.9515
332.3313
383.2046
429.2402
103.9551
131.9608
153.1387
m/z
+MS, 0.1-0.3min #6-15
[2ah+Na]+
Chemical Formula: C16H9ClN2NaO+
Exact Mass: 303.0296
303.0293
102.1275
153.1387
121.9658
187.0943
226.9512
+MS, 0.2-0.3min #12-15
[2ai+Na]+
Chemical Formula: C16H9ClN2NaO+
Exact Mass: 303.0296
226.9515
303.0297
319.0035
362.9263
430.9139
270.9777
158.9640
135.0022
242.9255
346.3317
413.2666
474.9398


+MS, 0.1-0.2min #8-10
Intens.
x10^6
346.9790
102.1277
226.9511
301.1407
430.9128
m/z
[2aj+Na]+
Chemical Formula: C16H9BrN2NaO+
Exact Mass: 346.9790
+MS, 0.0-0.1min #2-3
299.0794
575.1703
102.1278
158.9640
226.9515
269.0687
430.9137
545.1595
[2ak+Na]+
Chemical Formula: C17H12N2NaO2+
Exact Mass: 299.0791
+MS, 0.3-0.6min #20-33
312.1110
601.2366
102.1286
[2al+Na]+
Chemical Formula: C18H15N3NaO+
Exact Mass: 312.1107


Intens.
x10^6
+MS, 0.0min #2
[2am+Na]+
Chemical Formula: C15H9N3NaO+
Exact Mass: 270.0638
270.0640
226.9511
362.9263
430.9131
517.1394
m/z
Intens.
x10^5
+MS, 0.2-0.2min #10-12
[2an+Na]+
Chemical Formula: C15H9N3NaO+
Exact Mass: 270.0638
270.0635
290.0784
413.2665
301.1407
230.2481
135.0020
153.1386
327.2266
352.3188
378.3345
441.2976
m/z
Intens.
x10^6
+MS, 0.1-0.1min #6-7
[2ao+Na]+
Chemical Formula: C15H9N3NaO+
Exact Mass: 270.0638
270.0630
248.0795
226.9492
442.0970
517.1362
m/z



HRMS spectral chart for intermediate 5aa



X-Ray crystallography data

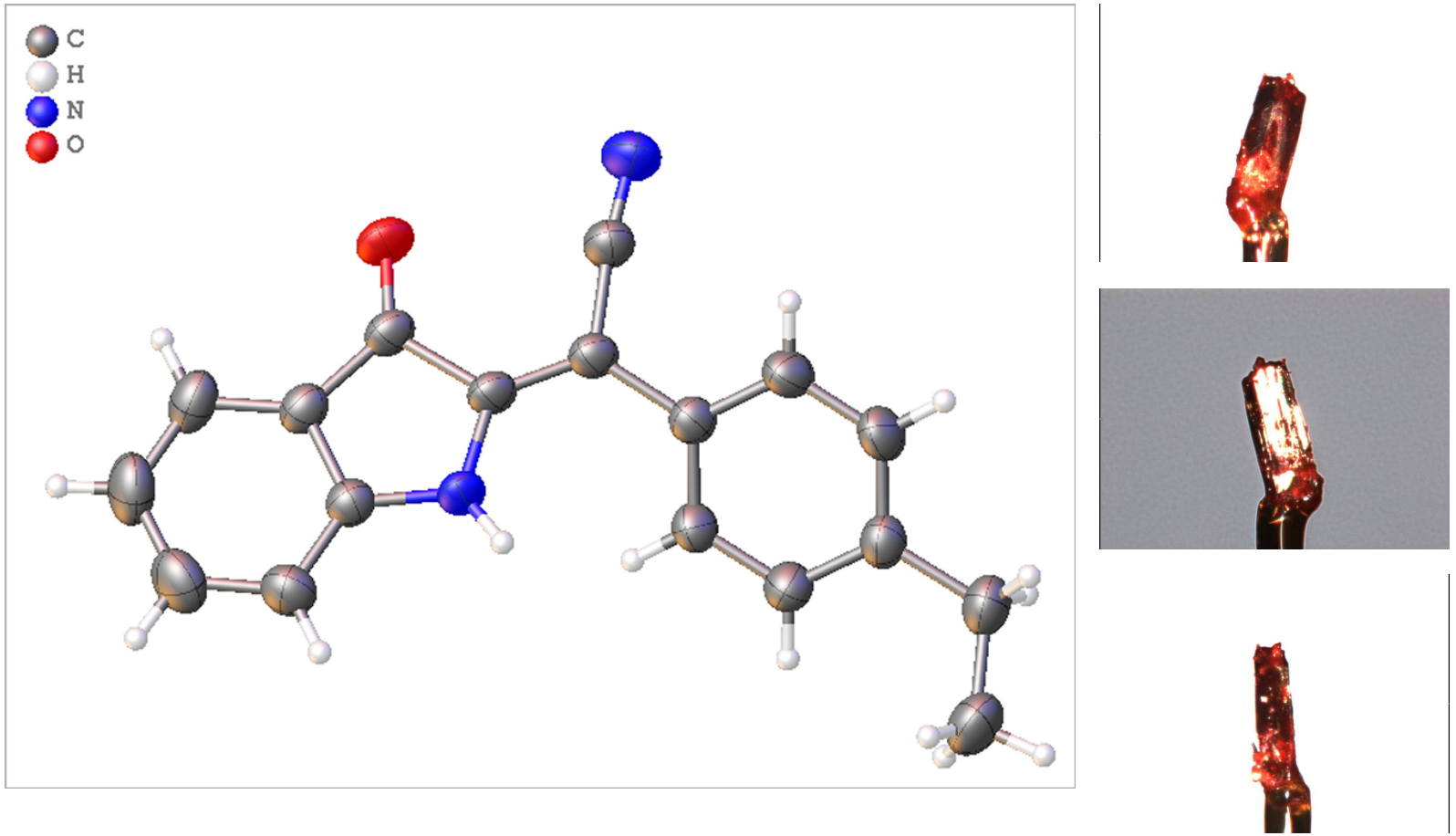


Figure S1. ORTEP drawing of the crystal structure (left) and microphotography of the single crystal of compound **2ad** used for X-Ray diffraction analysis (right)

Table S1 Crystal data and structure refinement for compound 2ad.

Identification code	ANNA1120_ISAT(da_FINAL)_3
Empirical formula	$C_{18}H_{14}N_2O$
Formula weight	274.31
Temperature/K	293(2)
Crystal system	monoclinic
Space group	C2/c
a/Å	20.6798(2)
b/Å	11.21770(10)
c/Å	12.58050(10)
α/°	90
β/°	102.8320(10)
γ/°	90
Volume/Å^3	2845.53(5)
Z	8
ρ_{calc}g/cm^3	1.281
μ/mm^{-1}	0.640
F(000)	1152.0
Crystal size/mm^3	0.394 × 0.208 × 0.107
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.772 to 152.69
Index ranges	-26 ≤ h ≤ 23, -14 ≤ k ≤ 14, -12 ≤ l ≤ 15
Reflections collected	15274
Independent reflections	2976 [R_{int} = 0.0160, R_{sigma} = 0.0101]
Data/restraints/parameters	2976/0/195
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0390, wR_2 = 0.1083
Final R indexes [all data]	R_1 = 0.0408, wR_2 = 0.1106
Largest diff. peak/hole / e Å^{-3}	0.20/-0.16

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

Atom	*x*	*y*	*z*	U(eq)
O1	4123.7(5)	4620.1(9)	2737.8(6)	56.5(2)
N1	4063.9(5)	5158.3(9)	5445.9(7)	44.9(2)
C8	4260.1(5)	6266.7(10)	5135.4(8)	41.1(2)
C1	3989.5(5)	4360.9(10)	4599.3(8)	39.8(2)
C2	4148.6(5)	5034.5(10)	3643.8(8)	41.2(2)
C9	3785.7(6)	3211.8(10)	4590.0(8)	41.0(2)
C11	3524.5(5)	2619.2(10)	5458.7(8)	40.6(2)
N2	3853.6(7)	1795.1(11)	3010.0(9)	67.2(3)
C3	4303.2(5)	6241.0(10)	4043.8(8)	42.1(2)
C16	3151.8(6)	3247.7(10)	6067.0(9)	44.1(3)
C14	2997.6(6)	1474.6(10)	7075.4(9)	46.6(3)
C10	3826.5(6)	2473.5(11)	3675.2(9)	47.9(3)
C15	2895.6(6)	2684.0(11)	6859.5(10)	47.3(3)
C12	3615.1(7)	1401.6(11)	5655.8(10)	51.0(3)
C7	4388.8(7)	7295.3(11)	5752.1(10)	52.4(3)
C13	3358.6(7)	844.4(11)	6451.5(10)	54.0(3)
C4	4458.6(7)	7268.6(12)	3532.9(11)	55.3(3)
C6	4547.0(8)	8301.1(13)	5230.4(13)	63.8(4)
C17	2719.6(8)	844.4(13)	7940.1(12)	61.2(3)
C5	4577.5(8)	8300.7(13)	4137.3(13)	66.8(4)
C18	2625.9(10)	1595.1(16)	8882.6(13)	74.2(4)

Table S3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for compound 2ad. 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}
O1	83.6(6)	60.0(5)	29.0(4)	-1.7(3)	19.0(4)	-2.8(4)
N1	69.3(6)	40.7(5)	27.4(4)	-0.8(3)	16.0(4)	-3.9(4)
C8	45.9(5)	42.4(6)	35.2(5)	0.3(4)	9.7(4)	-1.1(4)
C1	51.8(6)	42.2(5)	26.3(4)	0.6(4)	10.6(4)	1.9(4)
C2	48.8(6)	47.6(6)	28.0(5)	2.7(4)	10.1(4)	0.4(4)
C9	51.7(6)	41.1(5)	30.6(5)	-0.6(4)	9.8(4)	1.9(4)
C11	50.8(6)	37.6(5)	33.3(5)	2.0(4)	8.8(4)	0.8(4)
N2	95.8(9)	58.6(7)	53.4(6)	-18.3(5)	30.0(6)	-15.4(6)
C3	45.1(5)	47.9(6)	33.7(5)	2.7(4)	9.6(4)	-3.8(4)
C16	54.2(6)	34.4(5)	45.6(6)	4.4(4)	15.4(5)	2.9(4)
C14	56.6(6)	40.9(6)	43.0(6)	4.8(4)	12.3(5)	-4.8(5)
C10	61.1(7)	46.4(6)	37.6(5)	-3.7(5)	14.3(5)	-5.7(5)
C15	54.8(6)	41.5(6)	49.9(6)	1.9(5)	20.7(5)	1.2(5)
C12	71.2(8)	38.8(6)	46.6(6)	0.5(5)	20.6(5)	7.6(5)
C7	61.9(7)	48.6(7)	46.4(6)	-7.7(5)	11.4(5)	-4.6(5)
C13	77.6(8)	34.4(6)	52.1(7)	6.2(5)	19.1(6)	4.0(5)
C4	58.7(7)	60.9(8)	46.6(6)	9.9(5)	12.3(5)	-14.2(6)
C6	70.5(8)	47.5(7)	69.7(9)	-7.4(6)	7.9(7)	-13.3(6)
C17	78.8(9)	51.5(7)	58.2(7)	8.8(6)	25.8(7)	-9.5(6)
C5	74.2(9)	54.3(8)	69.6(9)	10.3(7)	11.3(7)	-22.1(7)
C18	99.7(12)	74.6(10)	56.1(8)	6.3(7)	34.1(8)	-14.0(9)

Table S4. Bond Lengths for compound 2ad.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C2	1.2214(13)	N2	C10	1.1417(16)
N1	C8	1.3905(14)	C3	C4	1.3917(16)
N1	C1	1.3730(13)	C16	C15	1.3818(15)
C8	C3	1.3962(14)	C14	C15	1.3904(17)
C8	C7	1.3828(16)	C14	C13	1.3905(18)
C1	C2	1.5165(14)	C14	C17	1.5138(16)
C1	C9	1.3554(16)	C12	C13	1.3821(17)
C2	C3	1.4545(16)	C7	C6	1.3807(19)
C9	C11	1.4800(14)	C4	C5	1.377(2)
C9	C10	1.4355(15)	C6	C5	1.390(2)
C11	C16	1.3924(15)	C17	C18	1.502(2)
C11	C12	1.3933(16)			

Table S5. Bond Angles for compound 2ad.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	N1	C8	110.74(9)	C8	C3	C2	107.60(9)
N1	C8	C3	110.13(9)	C4	C3	C8	120.59(11)
C7	C8	N1	128.34(10)	C4	C3	C2	131.80(10)
C7	C8	C3	121.51(11)	C15	C16	C11	120.84(10)
N1	C1	C2	106.47(9)	C15	C14	C13	117.58(11)
C9	C1	N1	127.40(9)	C15	C14	C17	122.05(11)
C9	C1	C2	126.07(9)	C13	C14	C17	120.36(11)
O1	C2	C1	125.34(11)	N2	C10	C9	173.42(13)
O1	C2	C3	129.60(10)	C16	C15	C14	121.44(11)
C3	C2	C1	105.02(8)	C13	C12	C11	120.94(11)
C1	C9	C11	125.77(9)	C6	C7	C8	116.85(12)
C1	C9	C10	118.82(10)	C12	C13	C14	121.30(11)
C10	C9	C11	115.40(10)	C5	C4	C3	118.21(12)
C16	C11	C9	121.05(10)	C7	C6	C5	122.51(13)
C16	C11	C12	117.88(10)	C18	C17	C14	116.21(12)
C12	C11	C9	121.00(10)	C4	C5	C6	120.31(12)

Table S6. Hydrogen Bonds for compound 2ad.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N1	H1	O1[1]	0.869(16)	2.053(16)	2.8692(12)	156.1(14)

[1]+X,1-Y,1/2+Z

Table S7. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 2ad.

Atom	*x*	*y*	*z*	U(eq)
H16	3074.37	4057.21	5938.31	53
H15	2649.6	3123.13	7257.29	57
H12	3851.54	957.58	5245.99	61
H7	4369.61	7308.9	6483.58	63
H13	3428.83	32	6572.1	65
H4	4481.4	7258.54	2802.96	66
H6	4636.7	9005.74	5625.15	77
H17A	2293.92	503.11	7593.6	73
H17B	3013.33	189.36	8225.24	73
H5	4678.7	9000.72	3812.98	80
H18A	2276.4	2160.34	8634.28	111
H18B	3030.09	2012.44	9185.52	111
H18C	2511.26	1092.63	9430.45	111
H1	4047(7)	5008(14)	6117(13)	57(4)

Experimental

Single crystals of $C_{18}H_{14}N_2O$ **2ad** were obtained via crystallization from ethanol. A suitable crystal was selected and mounted on the glass stick by acrylic glue, and the diffraction was measured on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer. The crystal was kept at 293(2) K during data collection. The structure was solved using Olex2 software,[S1] with the ShelXT [S2] structure solution algorithm using Intrinsic Phasing and refined with the ShelXL [S3] refinement package using Least Squares minimisation.

Crystal structure determination of compound 2ad.

Crystal Data for $C_{18}H_{14}N_2O$ (M =274.31 g/mol): monoclinic, space group C2/c (no. 15), a = 20.6798(2) Å, b = 11.21770(10) Å, c = 12.58050(10) Å, β = 102.8320(10)°, V = 2845.53(5) Å^3, Z = 8, T = 293(2) K, μ(CuKα) = 0.640 mm^{-1}, *Dcalc* = 1.281 g/cm^3, 15274 reflections measured (8.772° ≤ 2Θ ≤ 152.69°), 2976 unique (R_{int} = 0.0160, R_{sigma} = 0.0101) which were used in all calculations. The final R_1 was 0.0390 (I > 2σ(I)) and wR_2 was 0.1106 (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, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 C17(H17A,H17B)
2.b Aromatic/amide H refined with riding coordinates:
 C16(H16), C15(H15), C12(H12), C7(H7), C13(H13), C4(H4), C6(H6), C5(H5)
2.c Idealised Me refined as rotating group:
 C18(H18A,H18B,H18C)
```

This report has been created with Olex2, compiled on 2018.05.29 svn.r3508 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.

References

(S1) Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX 2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Cryst*. **2009**, *42*, 339-341.

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