Electronic Supplementary Material (ESI) for RSC Advances.

Preparation of Spiro[indole-3,5'-isoxazoles] via Grignard Conjugate Addition/Spirocyclization Sequence

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Supporting Information

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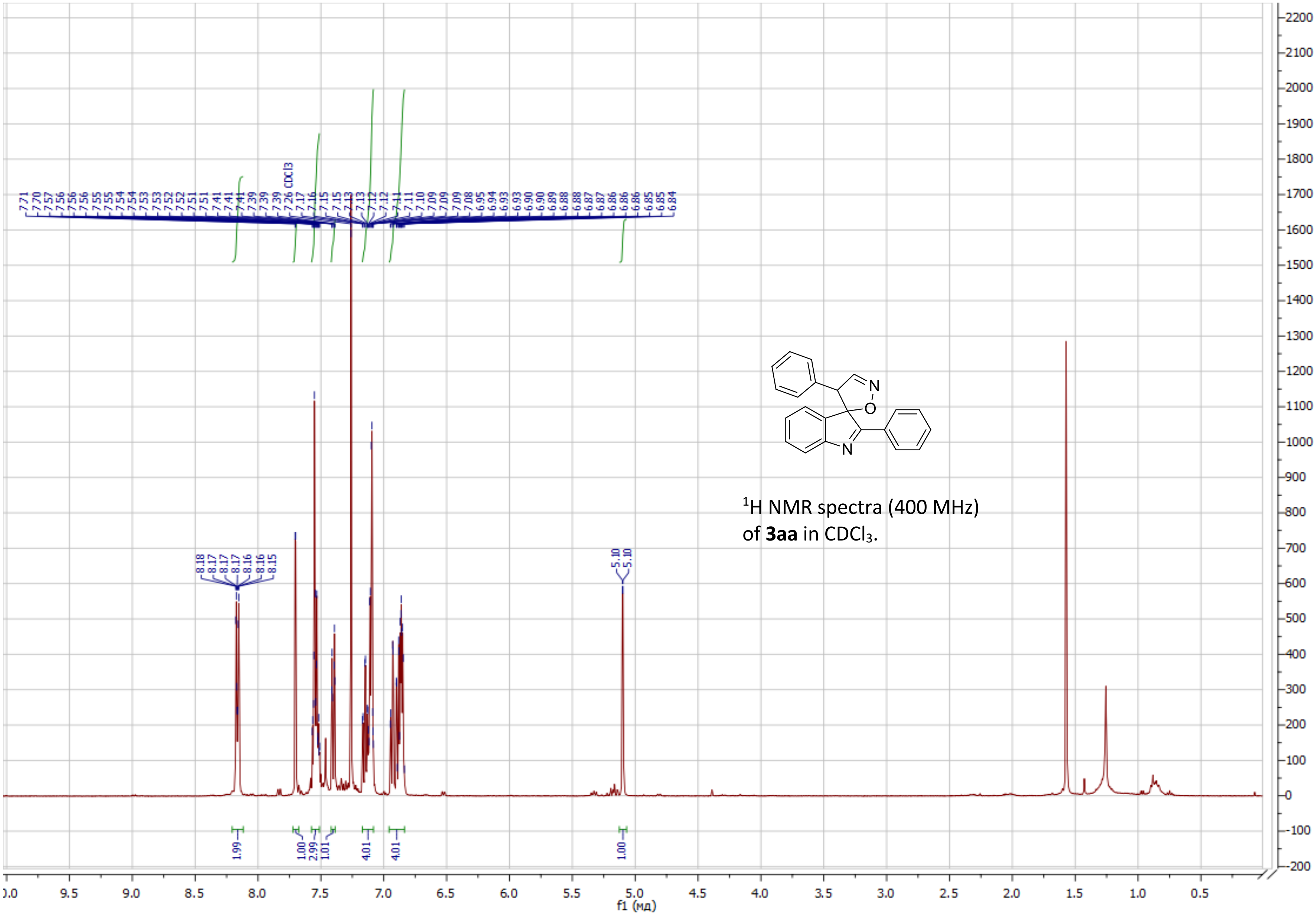
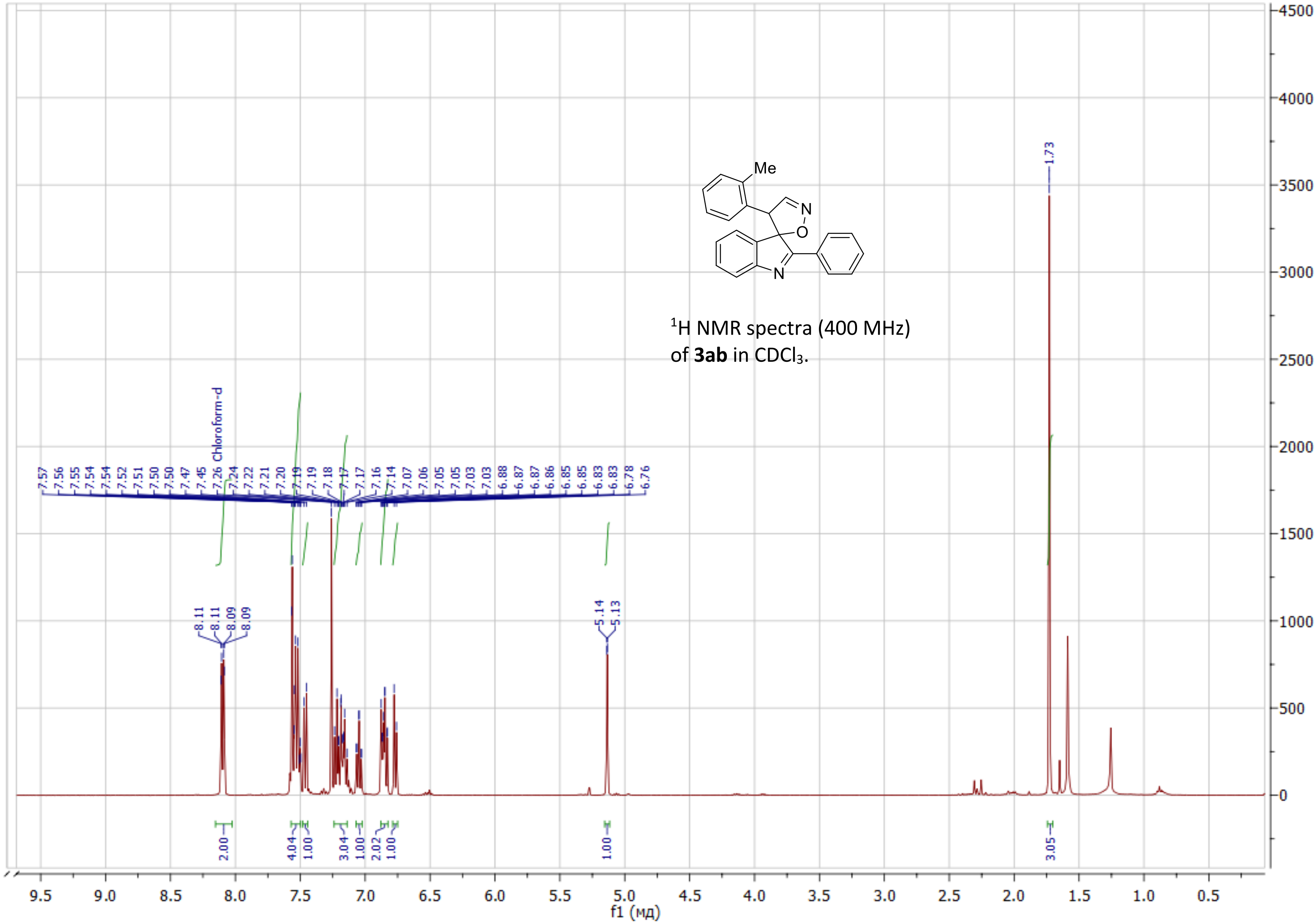
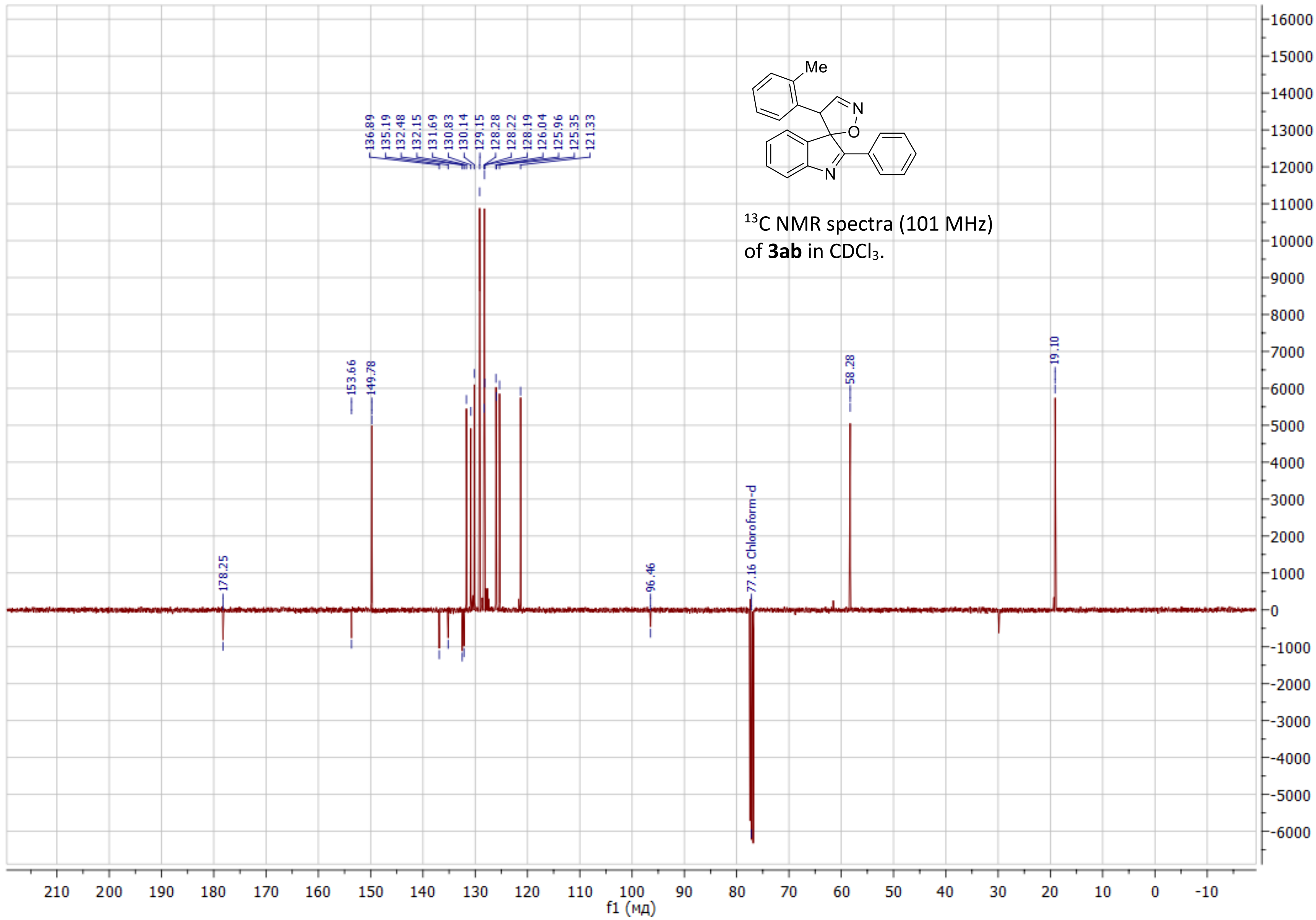
^{1}H and ^{13}C NMR Spectral Charts



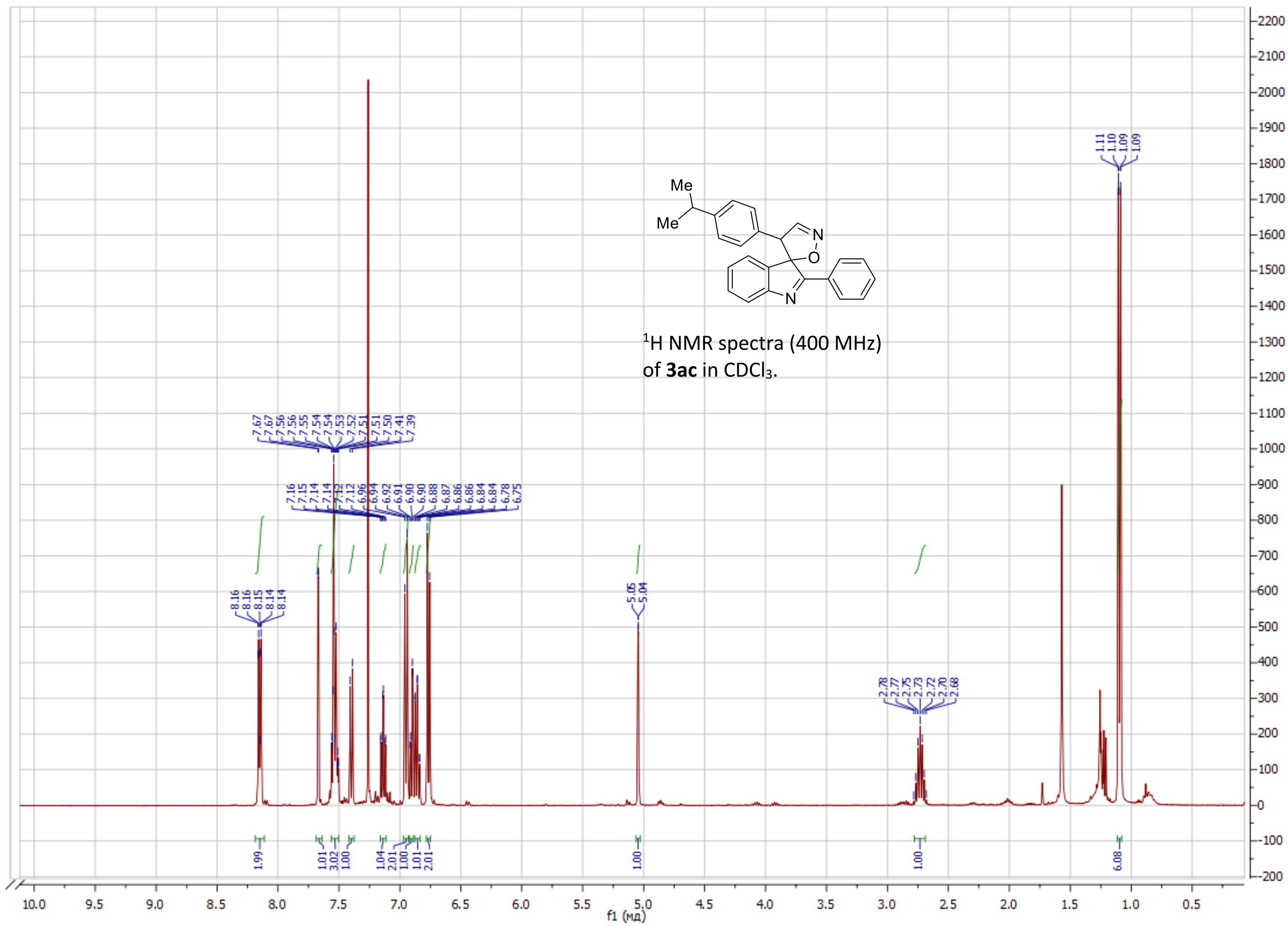
^{1}H NMR spectra (400 MHz) of **3aa** in $CDCl_3$.



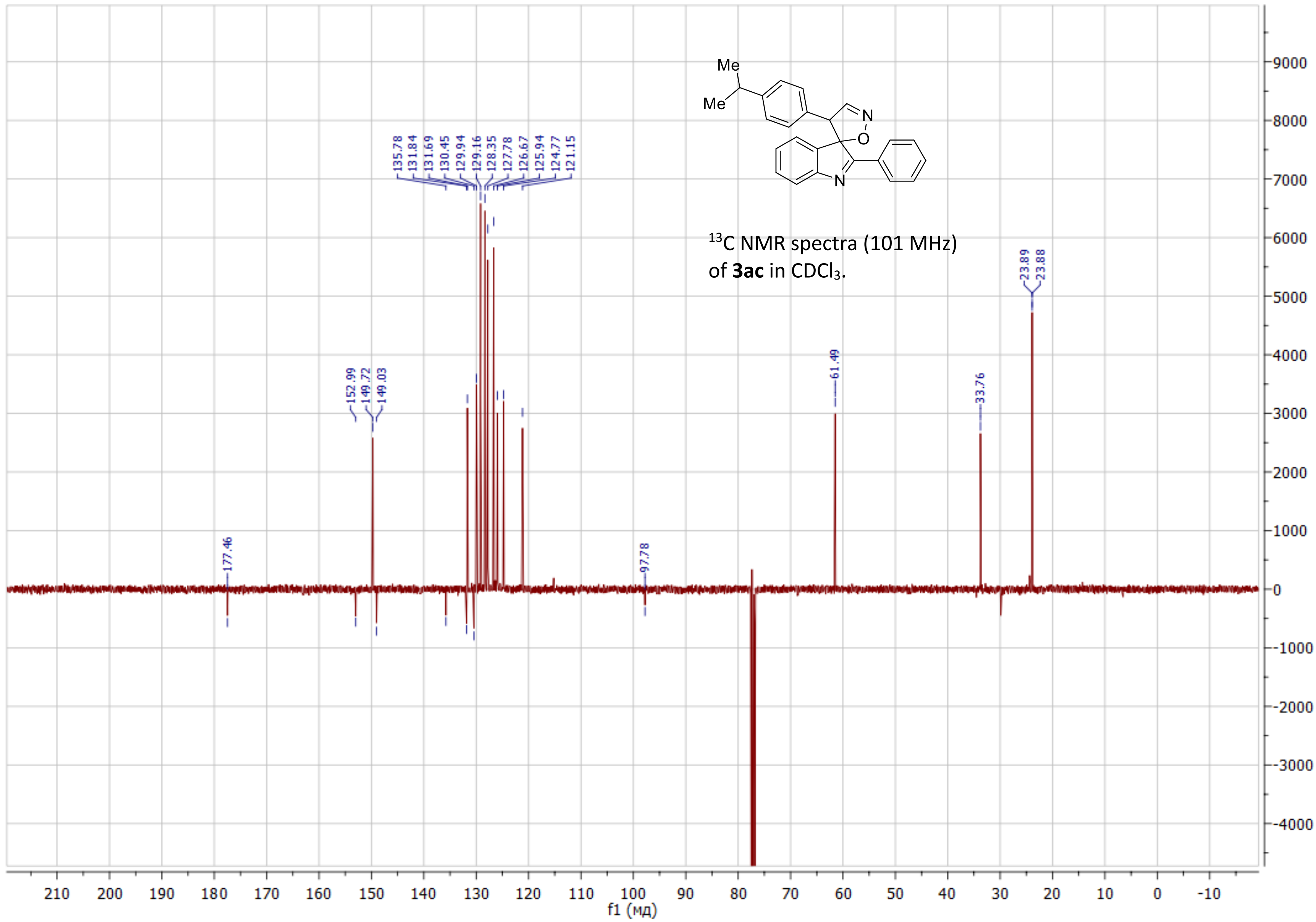
[1]H NMR spectra (400 MHz) of **3ab** in $CDCl_3$.



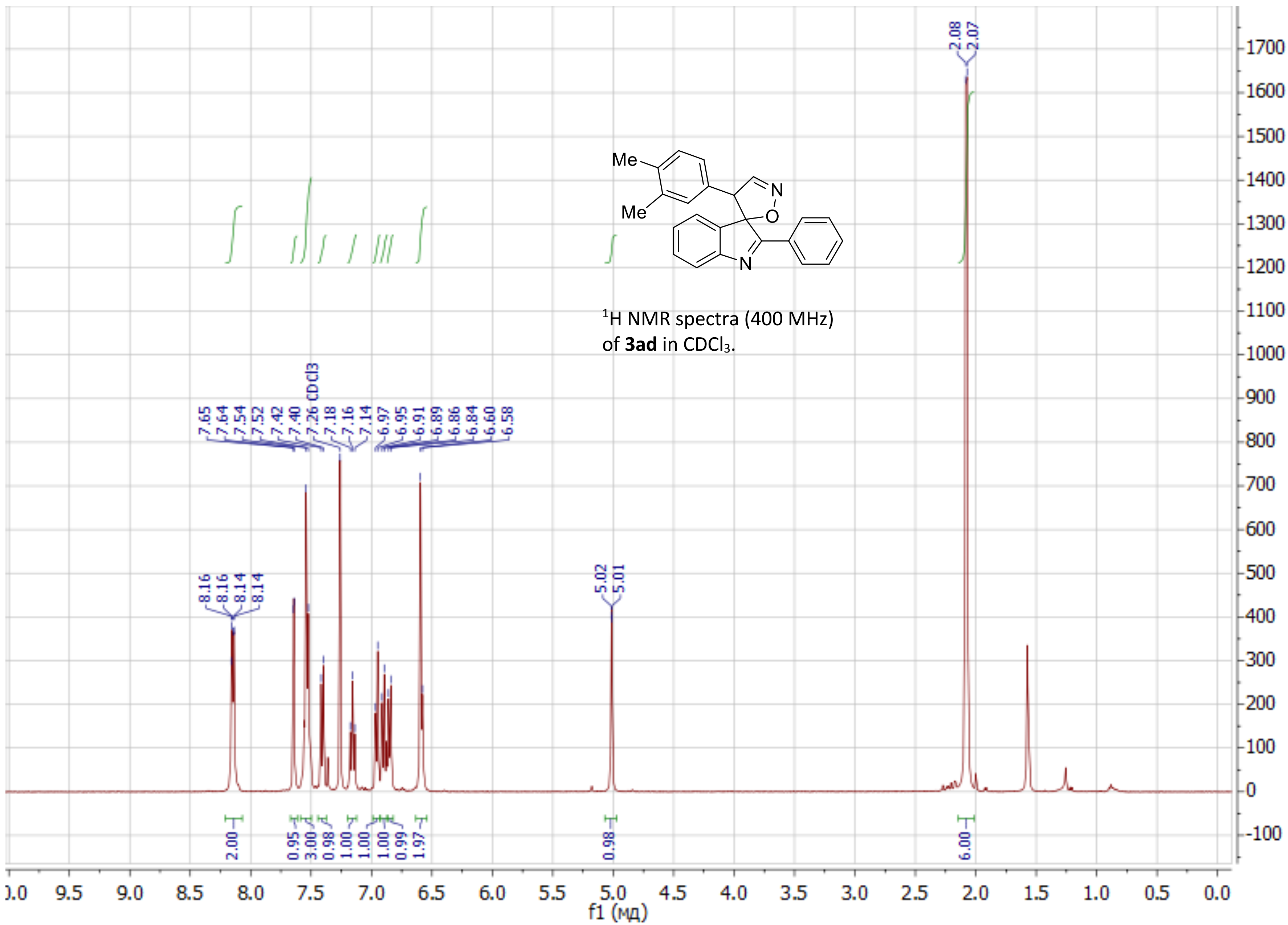
Me
N
O
N
13C NMR spectra (101 MHz) of 3ab in CDCl3.
136.89
135.19
132.48
132.15
131.69
130.83
130.14
129.15
128.28
128.22
128.19
126.04
125.96
125.35
121.33
178.25
153.66
149.78
96.46
77.16 Chloroform-d
58.28
19.10
f1 (мд)



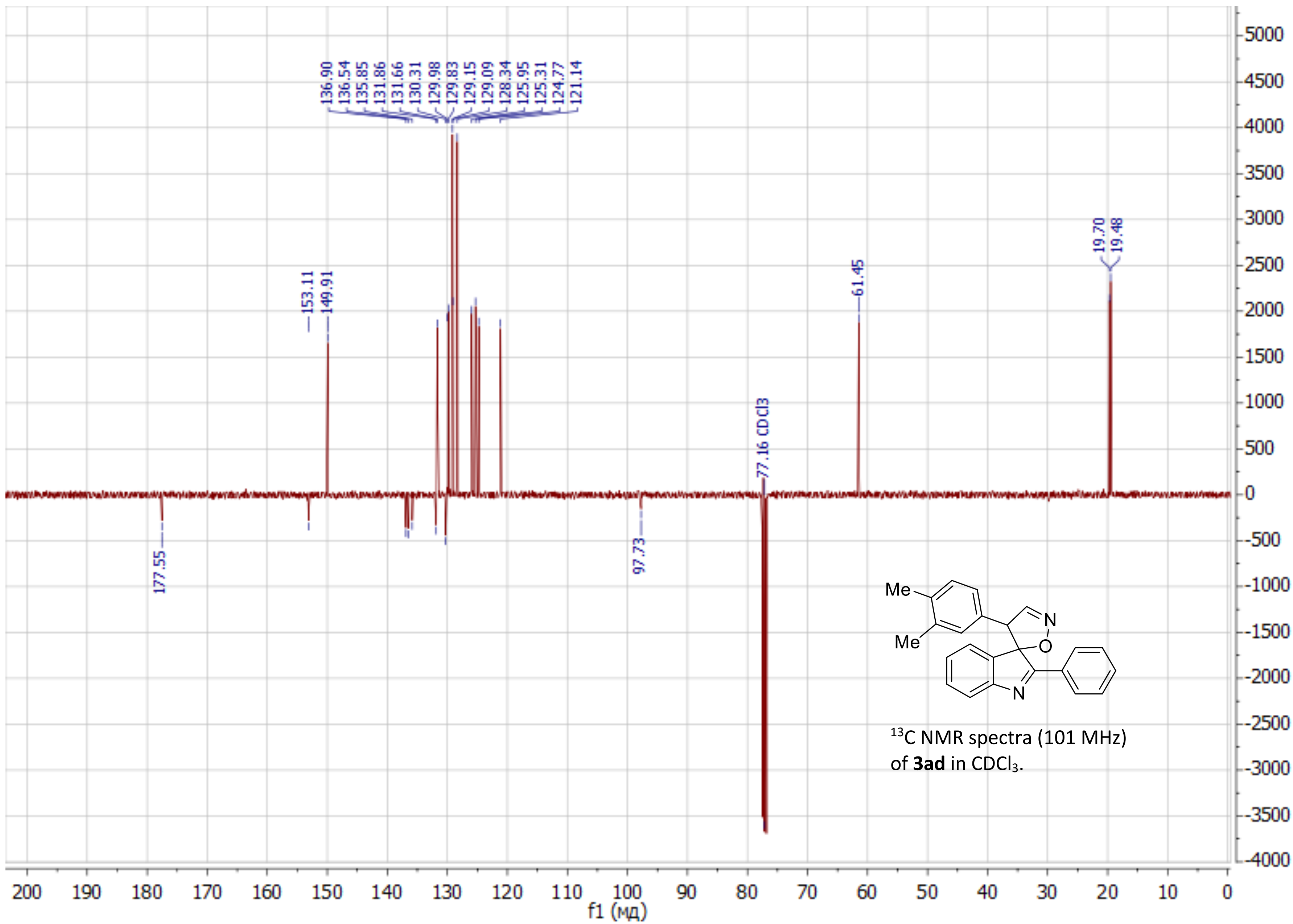
[1]H NMR spectra (400 MHz) of **3ac** in $CDCl_3$.



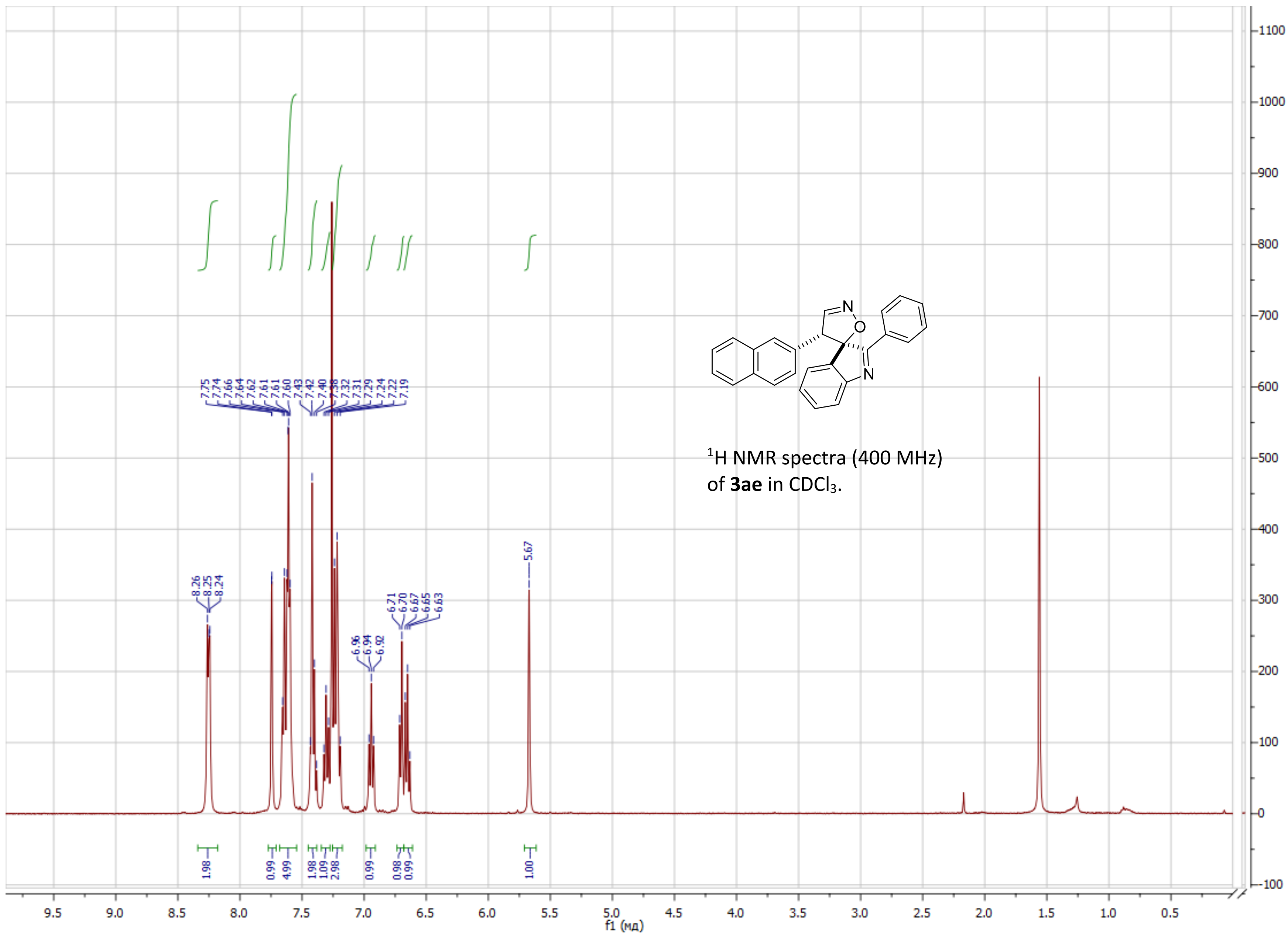
^{13}C NMR spectra (101 MHz) of **3ac** in $CDCl_3$.



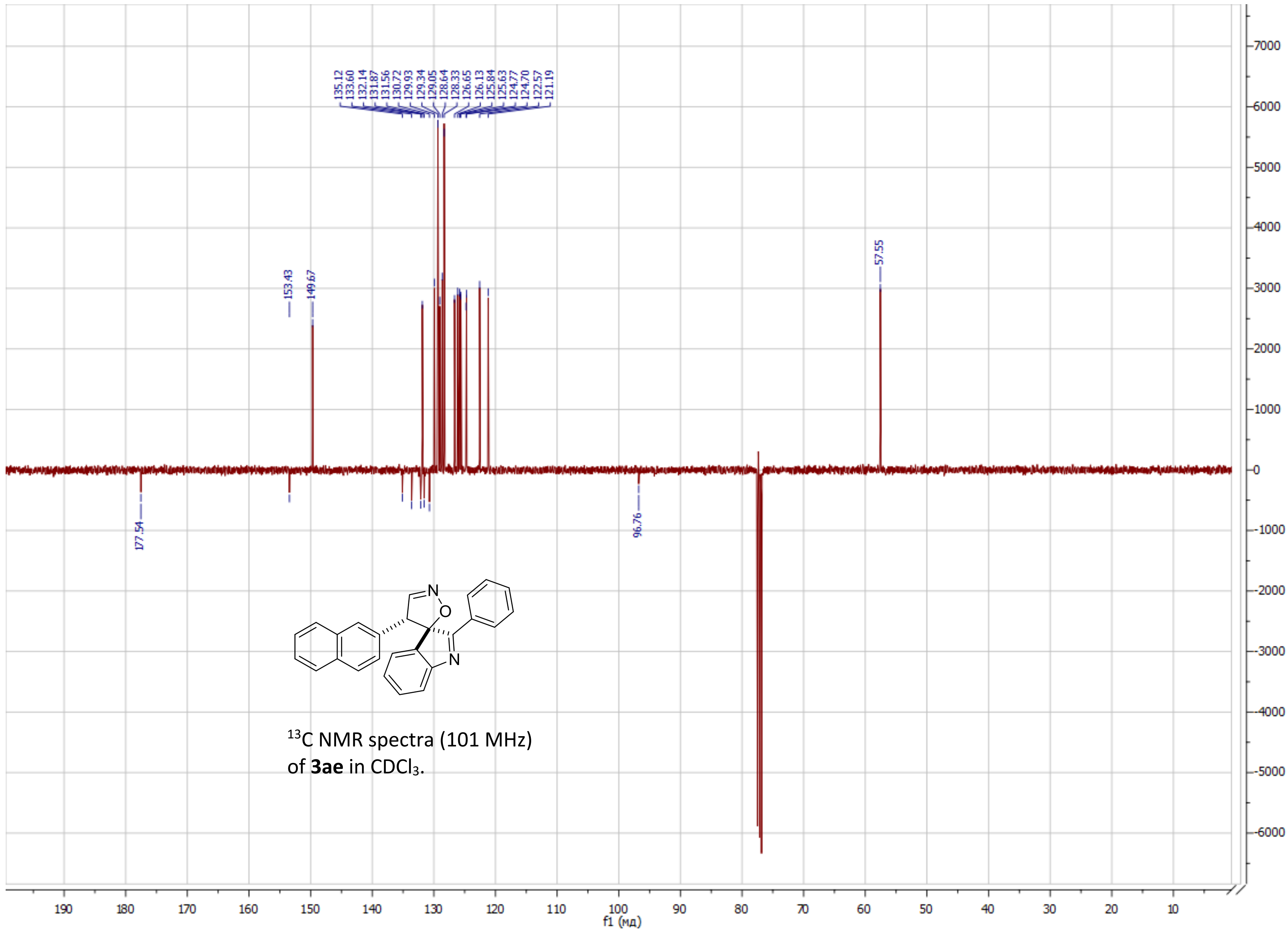
[1]H NMR spectra (400 MHz) of **3ad** in $CDCl_3$.



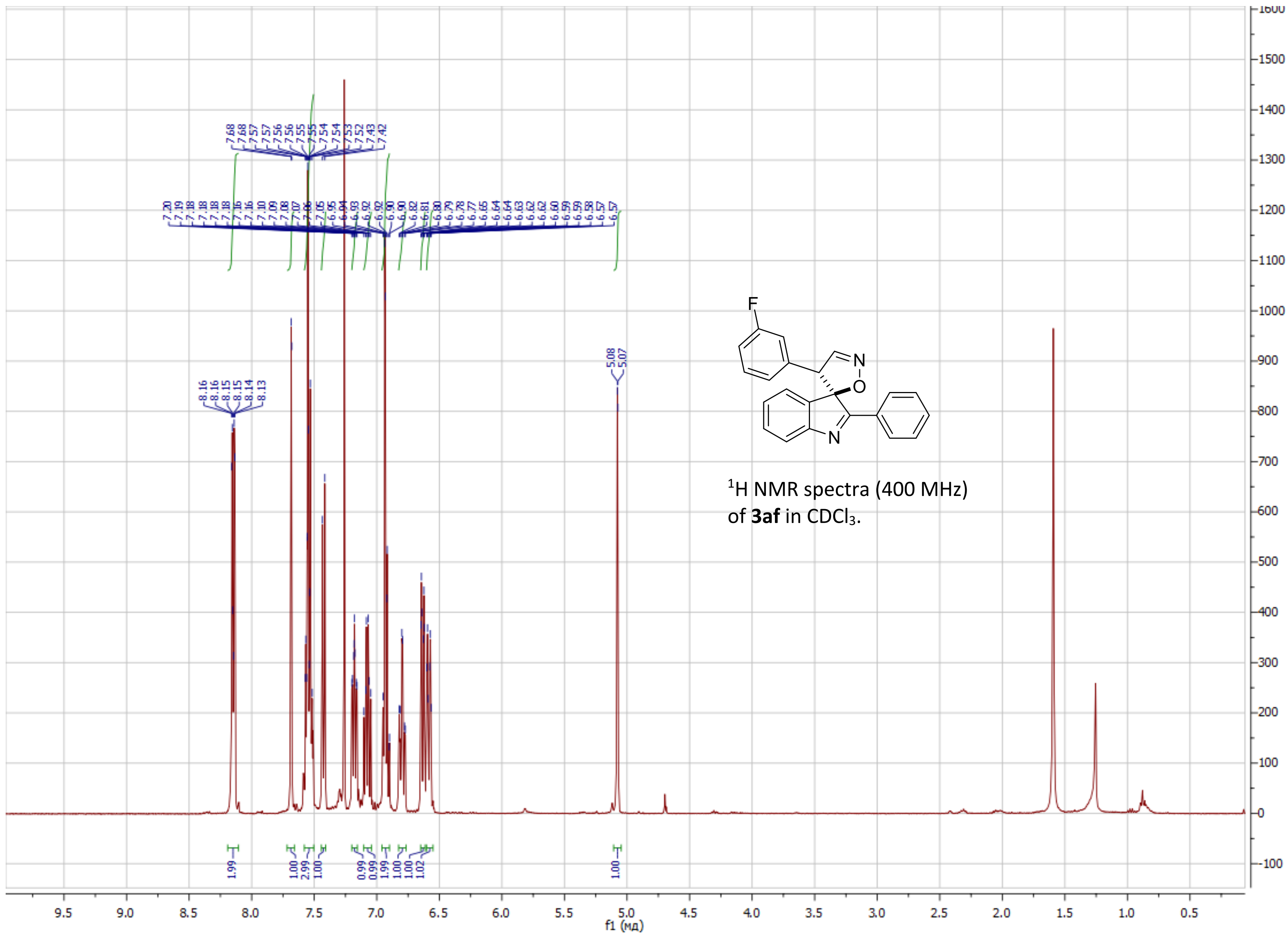
^{13}C NMR spectra (101 MHz) of **3ad** in $CDCl_3$.



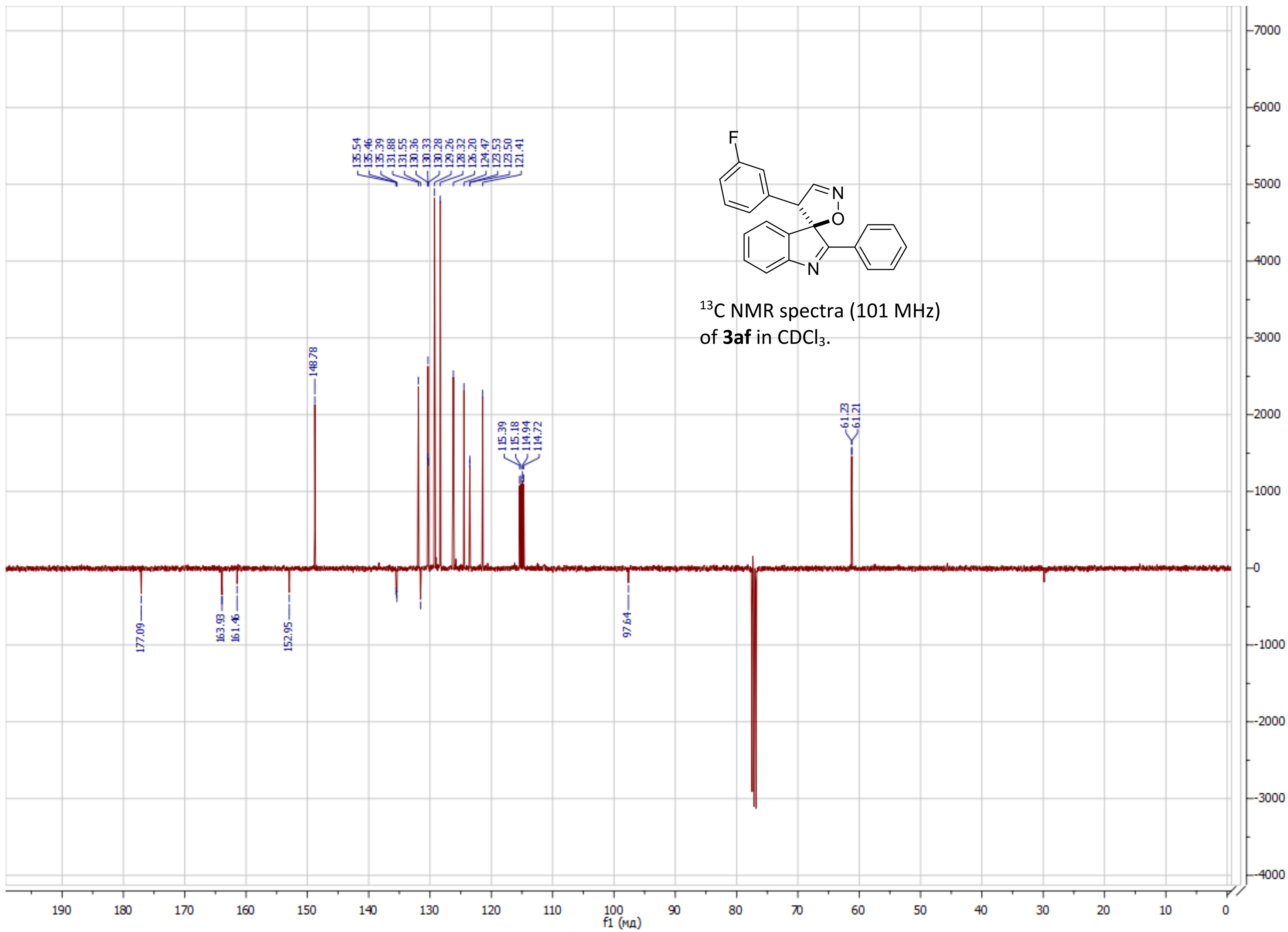
[1]H NMR spectra (400 MHz) of **3ae** in $CDCl_3$.



^{13}C NMR spectra (101 MHz)
of **3ae** in $CDCl_3$.



^1H NMR spectra (400 MHz) of **3af** in $CDCl_3$.



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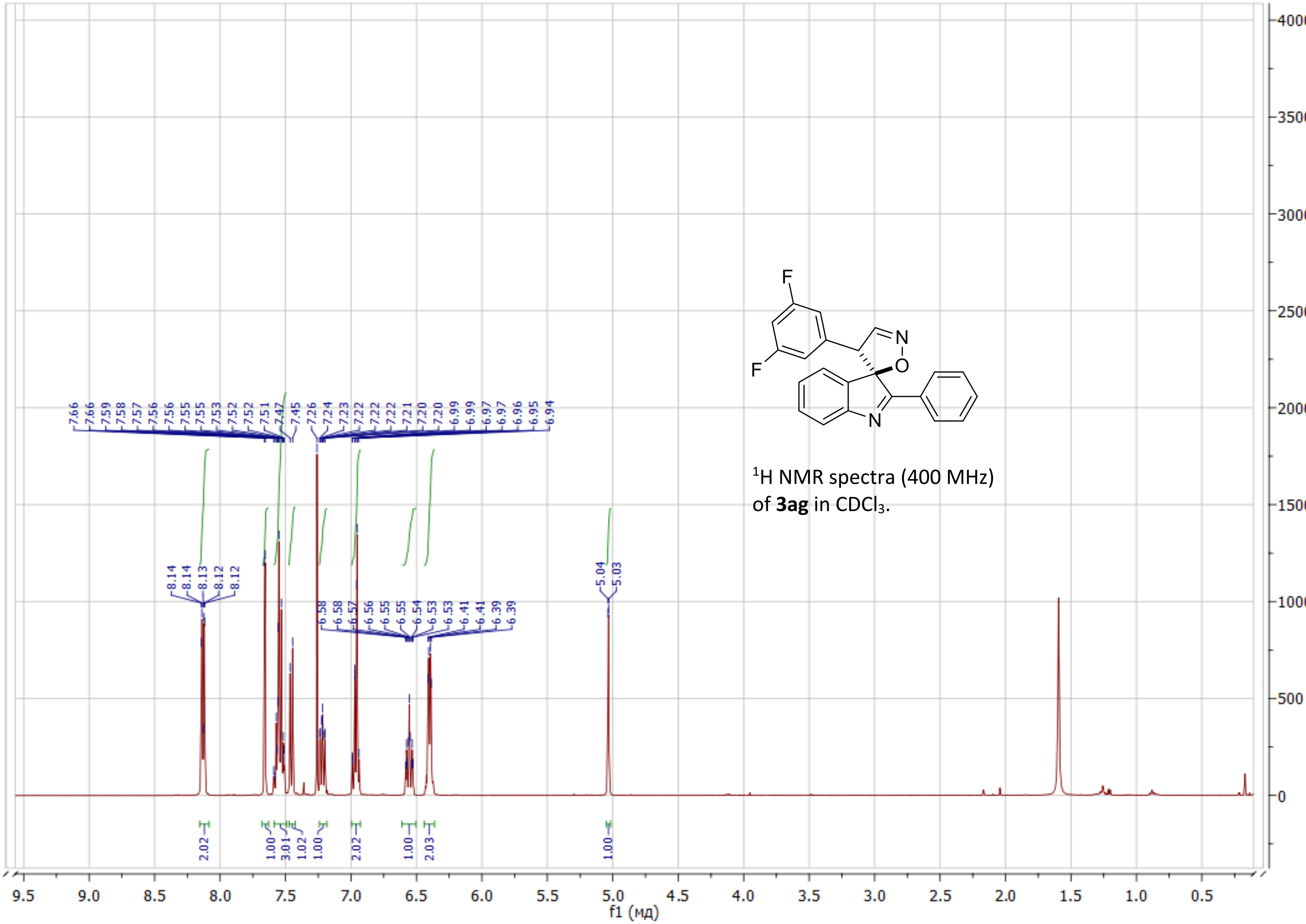
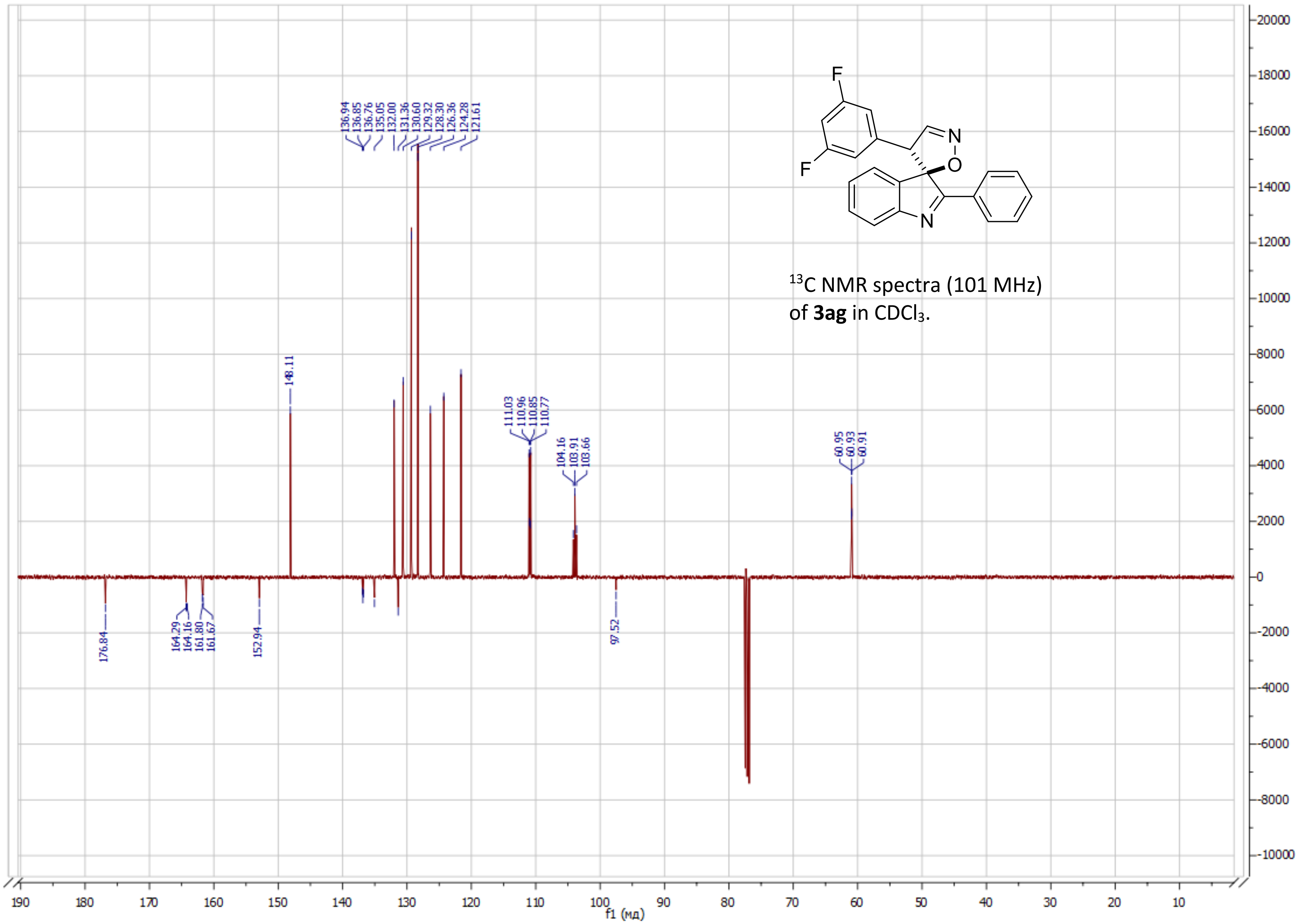
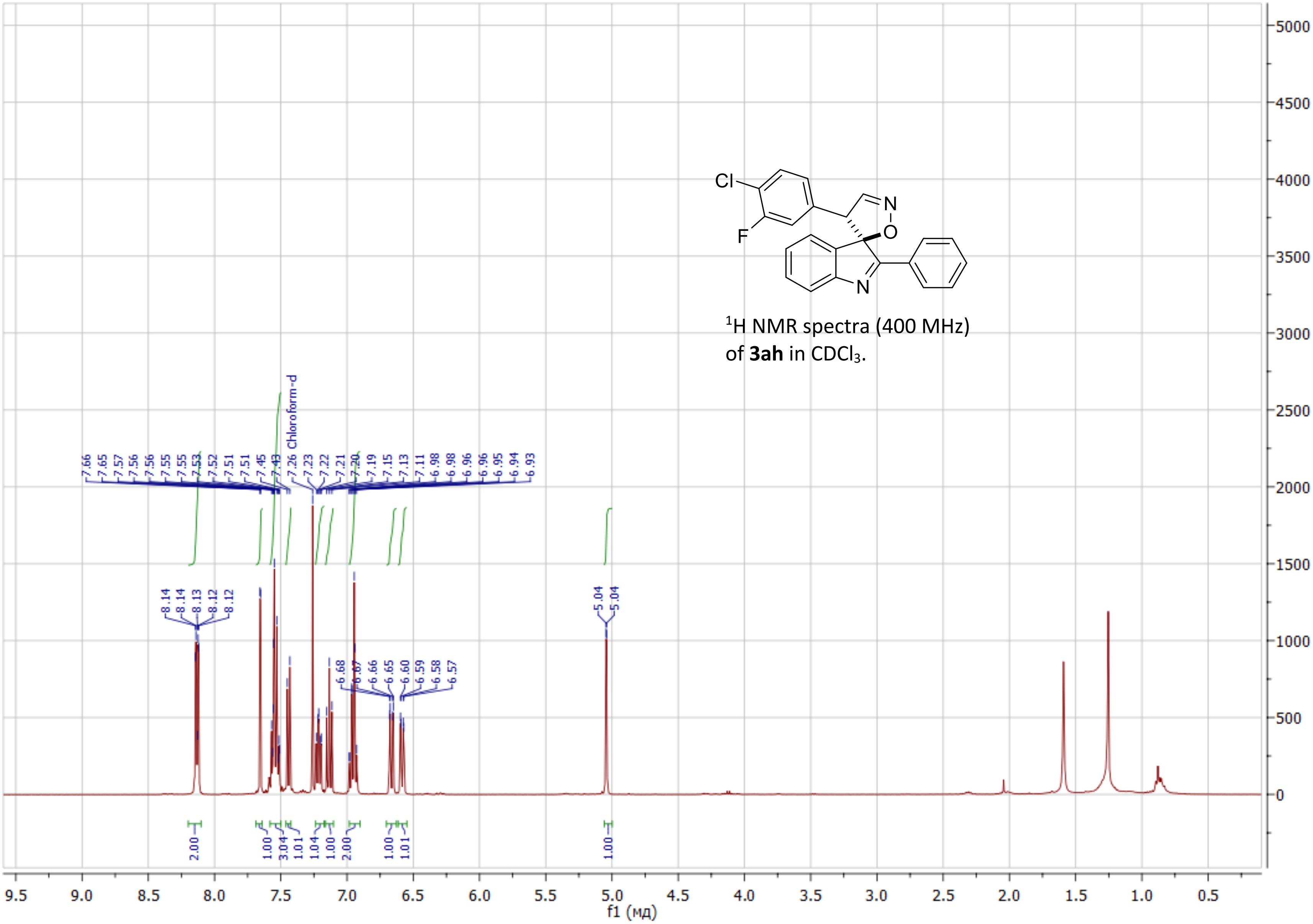


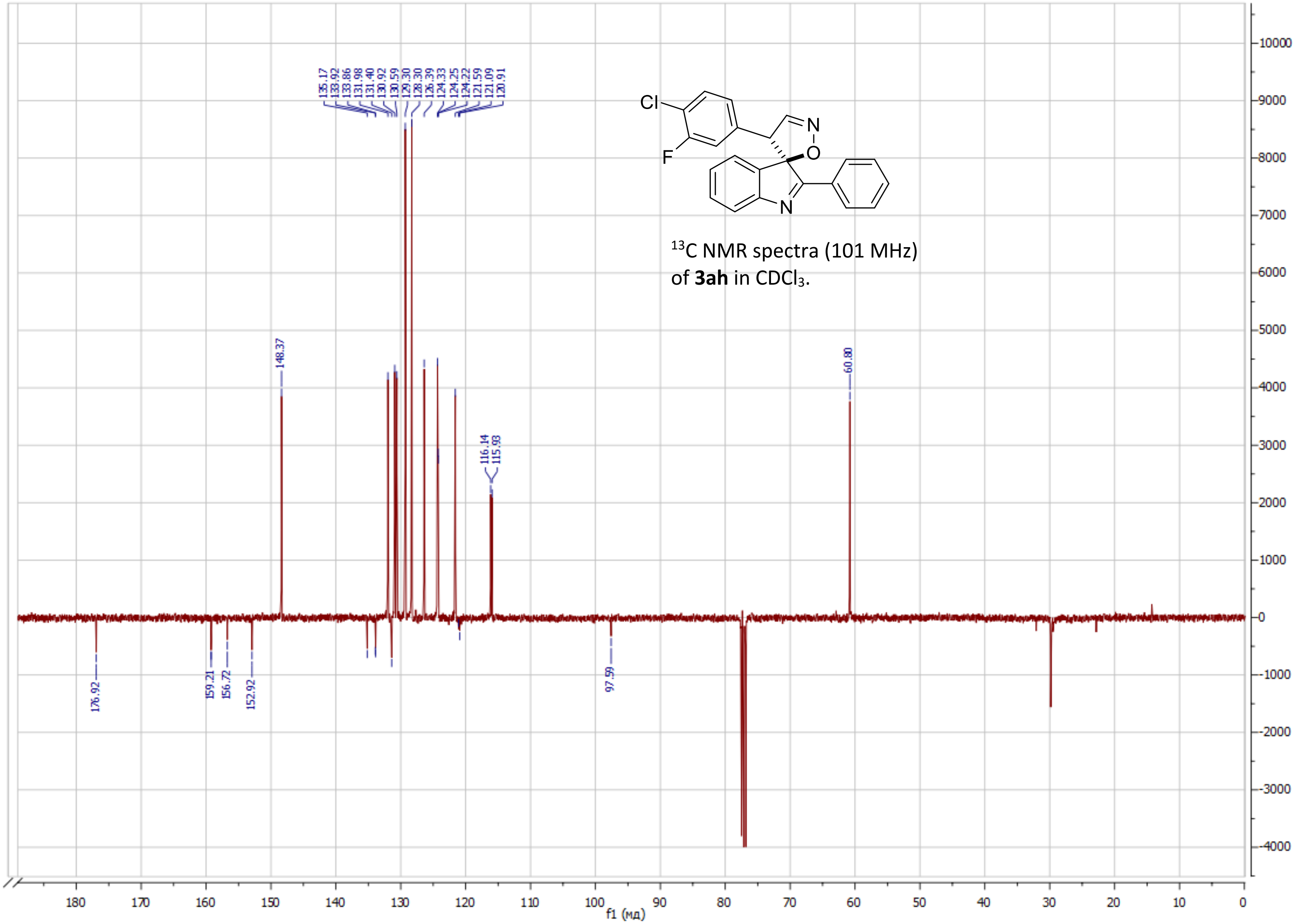
[1]H NMR spectra (400 MHz) of **3ag** in $CDCl_3$.



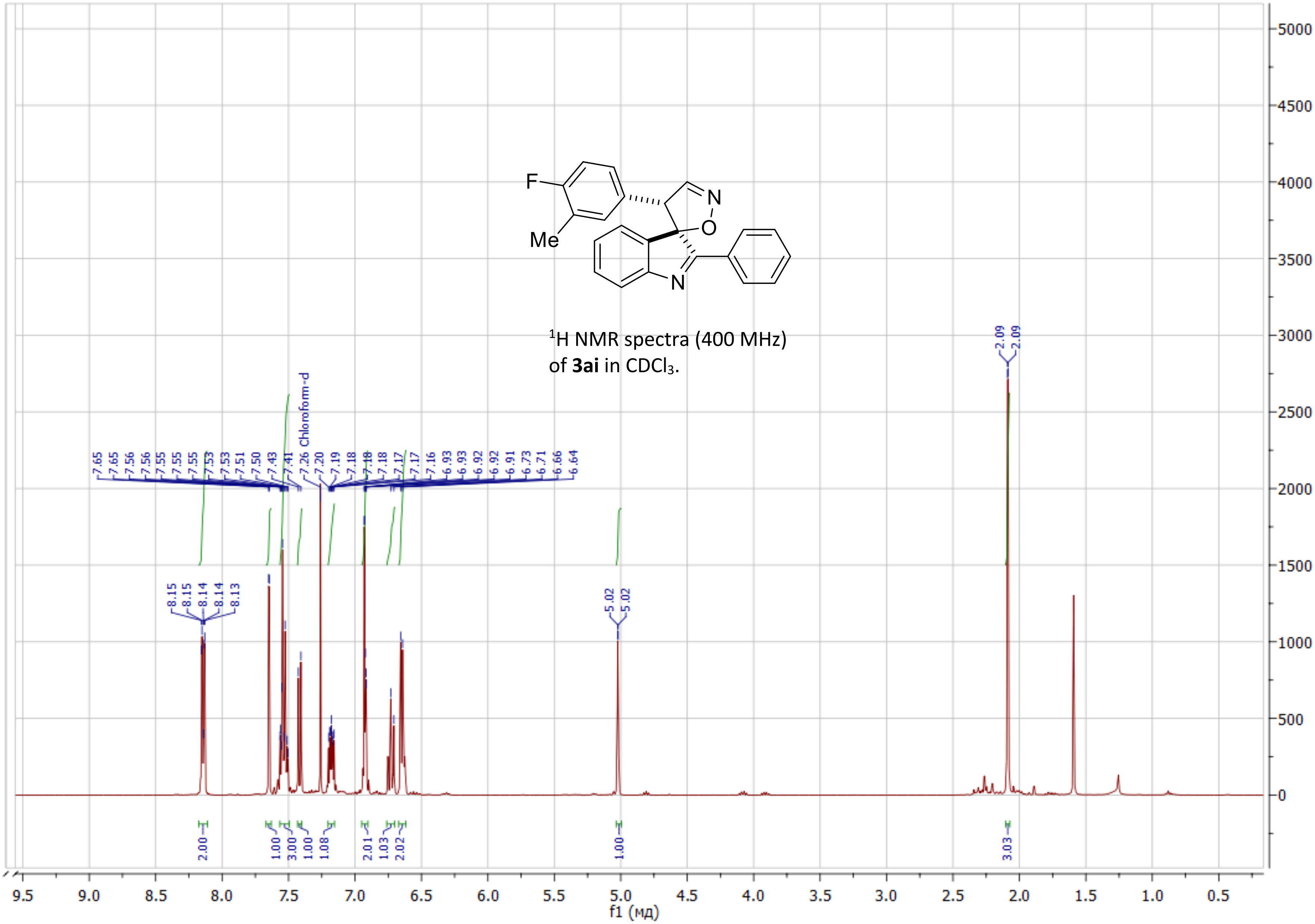
^{13}C NMR spectra (101 MHz) of **3ag** in $CDCl_3$.

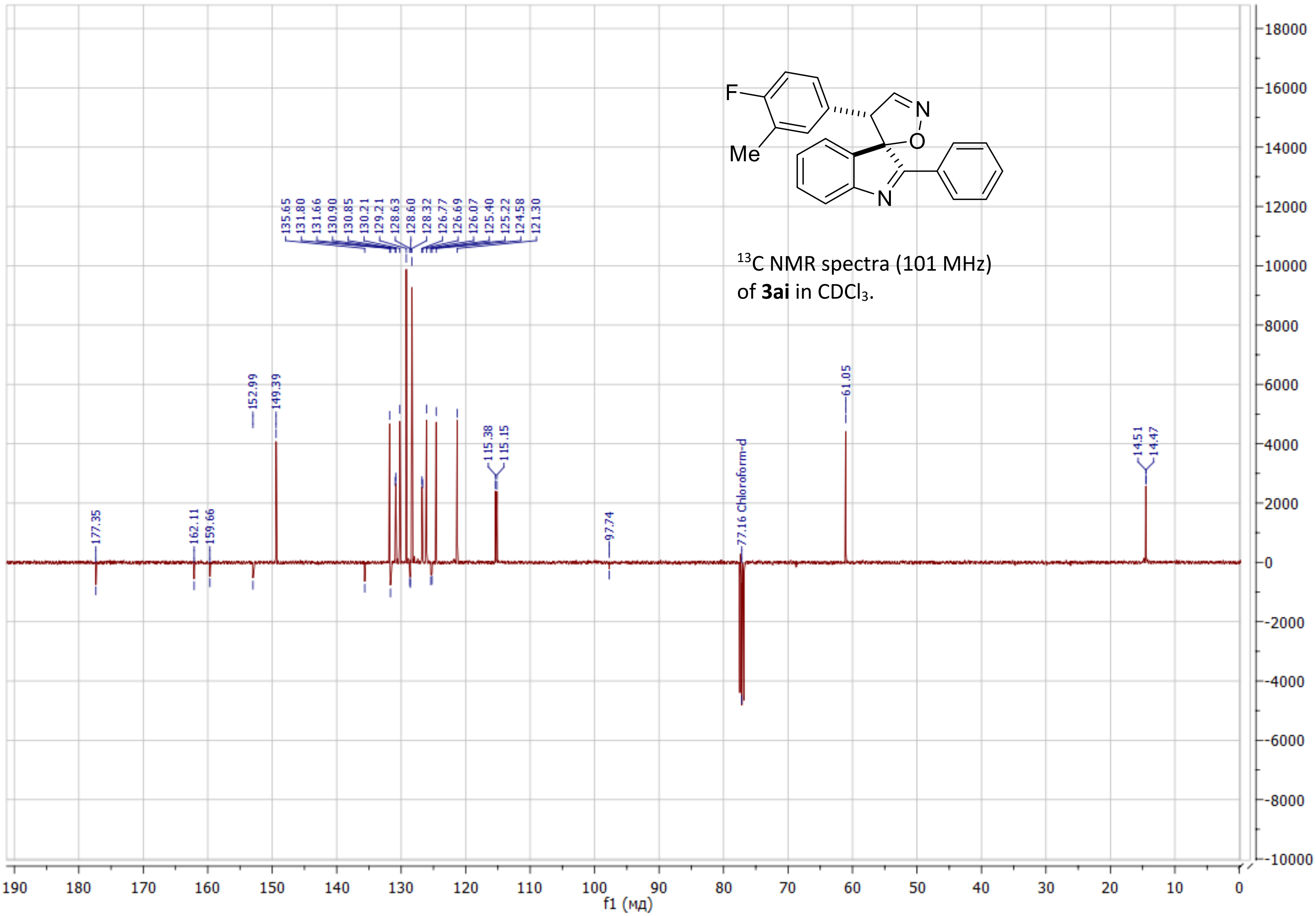


[1]H NMR spectra (400 MHz) of **3ah** in $CDCl_3$.

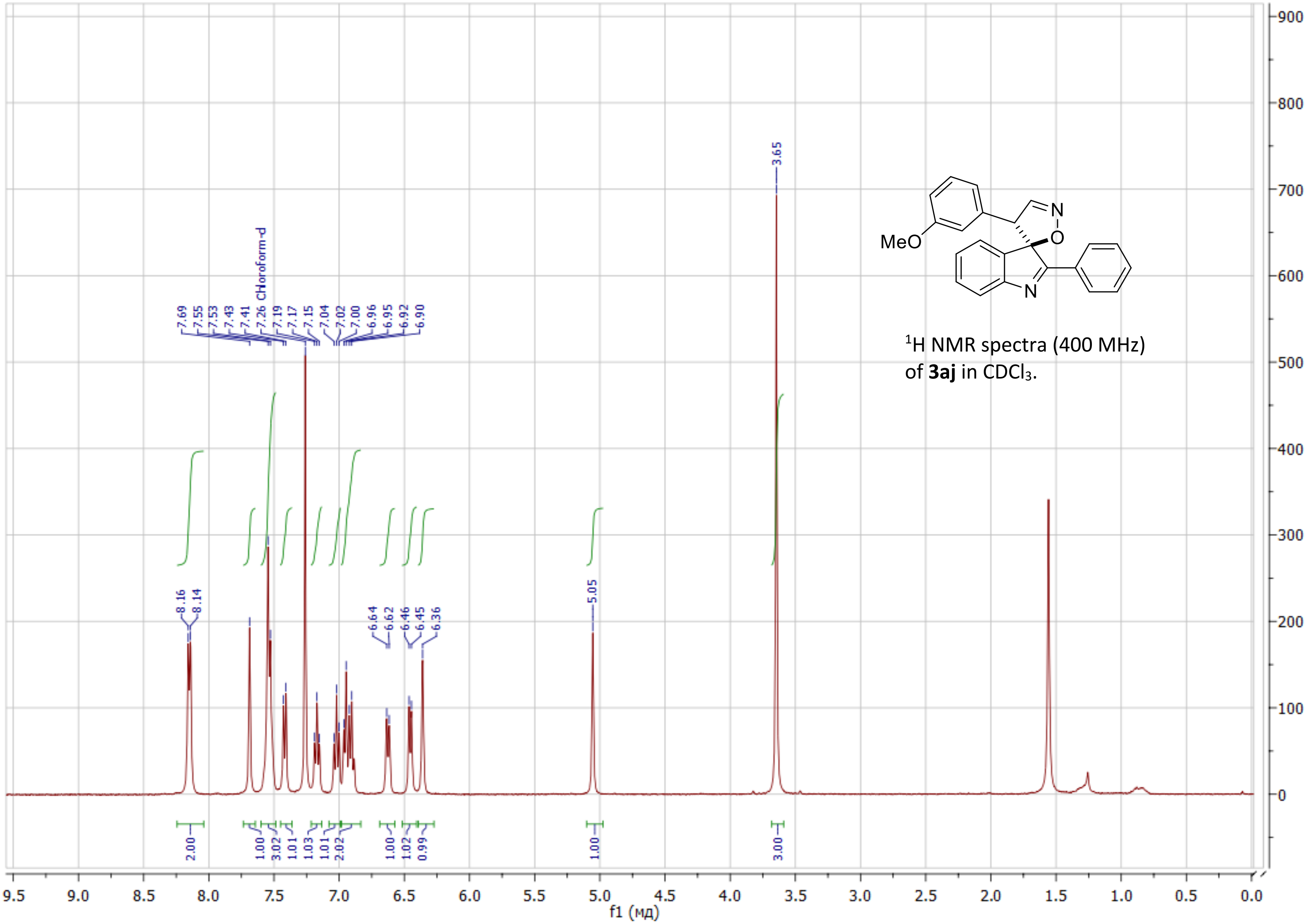


^{13}C NMR spectra (101 MHz) of **3ah** in $CDCl_3$.

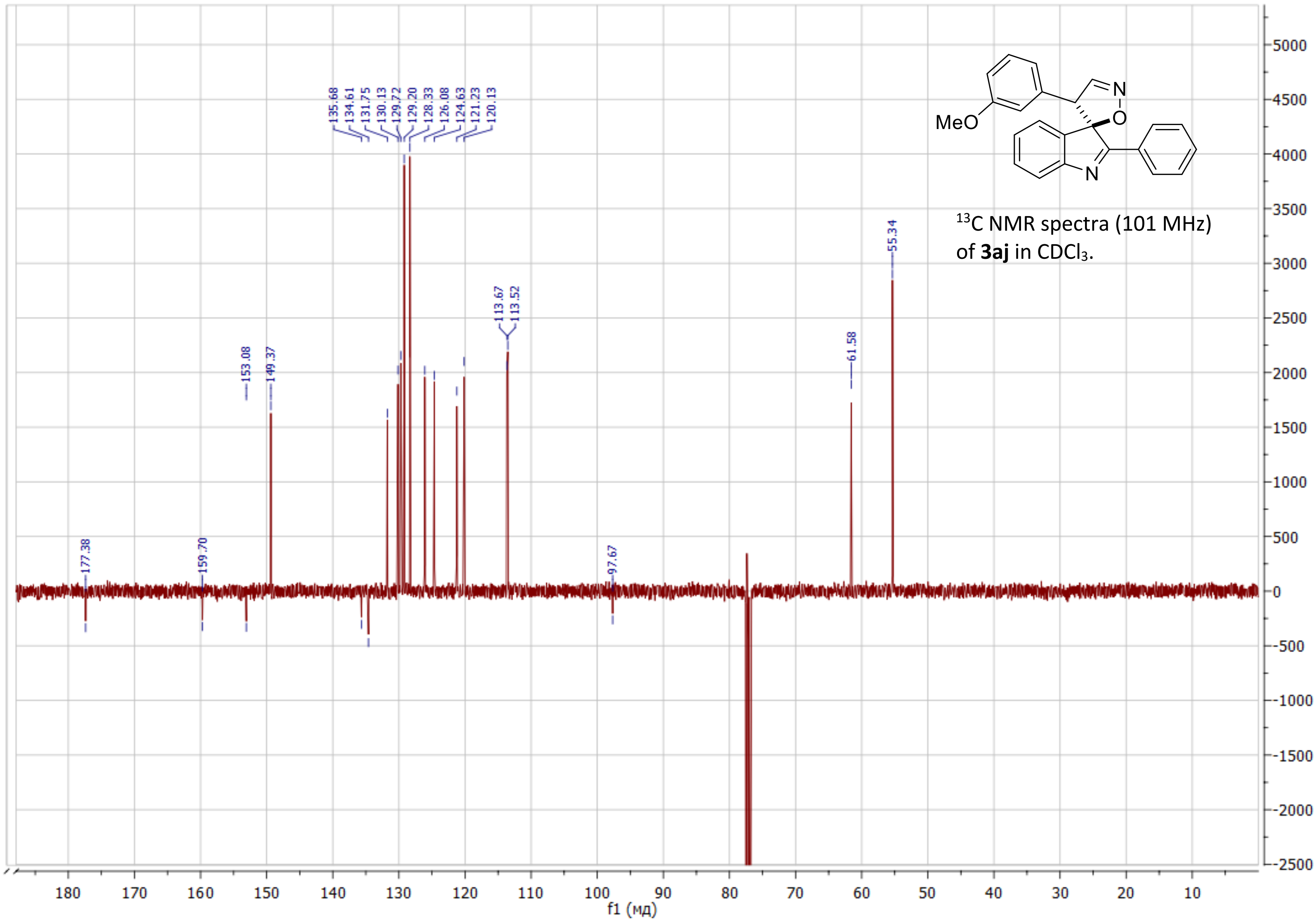

F
Me
N
O
N
1H NMR spectra (400 MHz)
of 3ai in CDCl3.
7.26 Chloroform-d
f1 (мд)



^{13}C NMR spectra (101 MHz) of **3ai** in $CDCl_3$.



[1]H NMR spectra (400 MHz) of **3aj** in $CDCl_3$.



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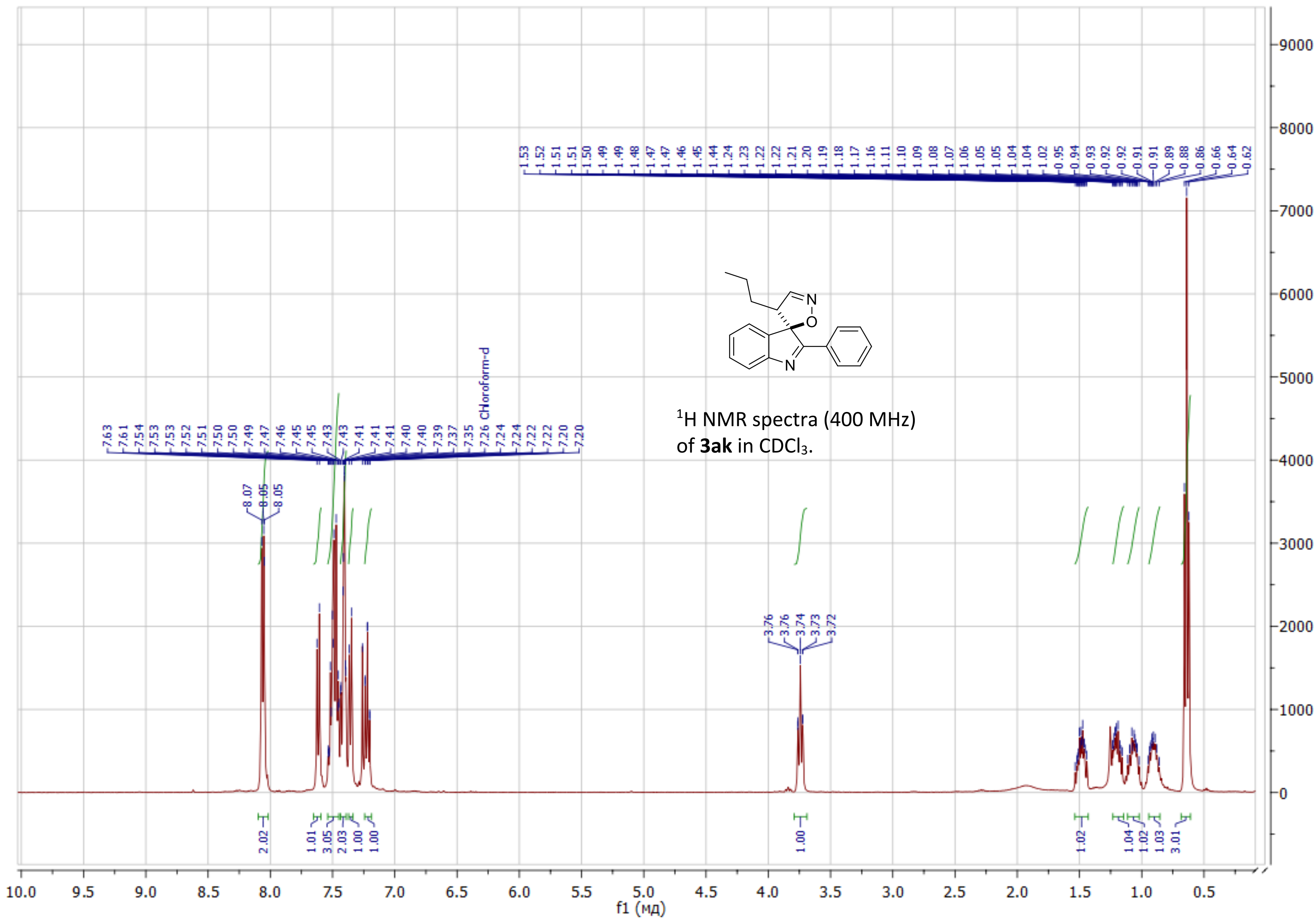
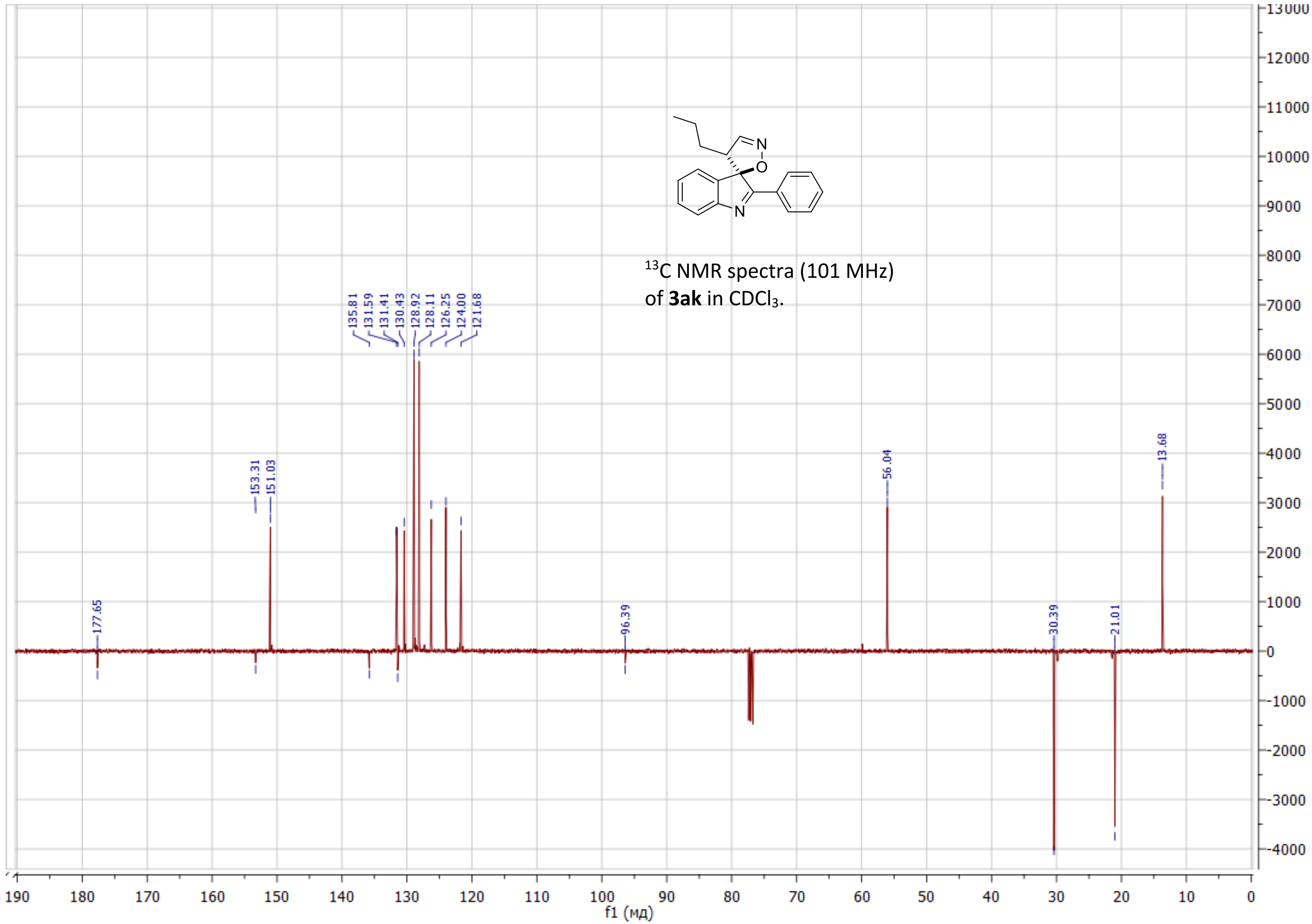
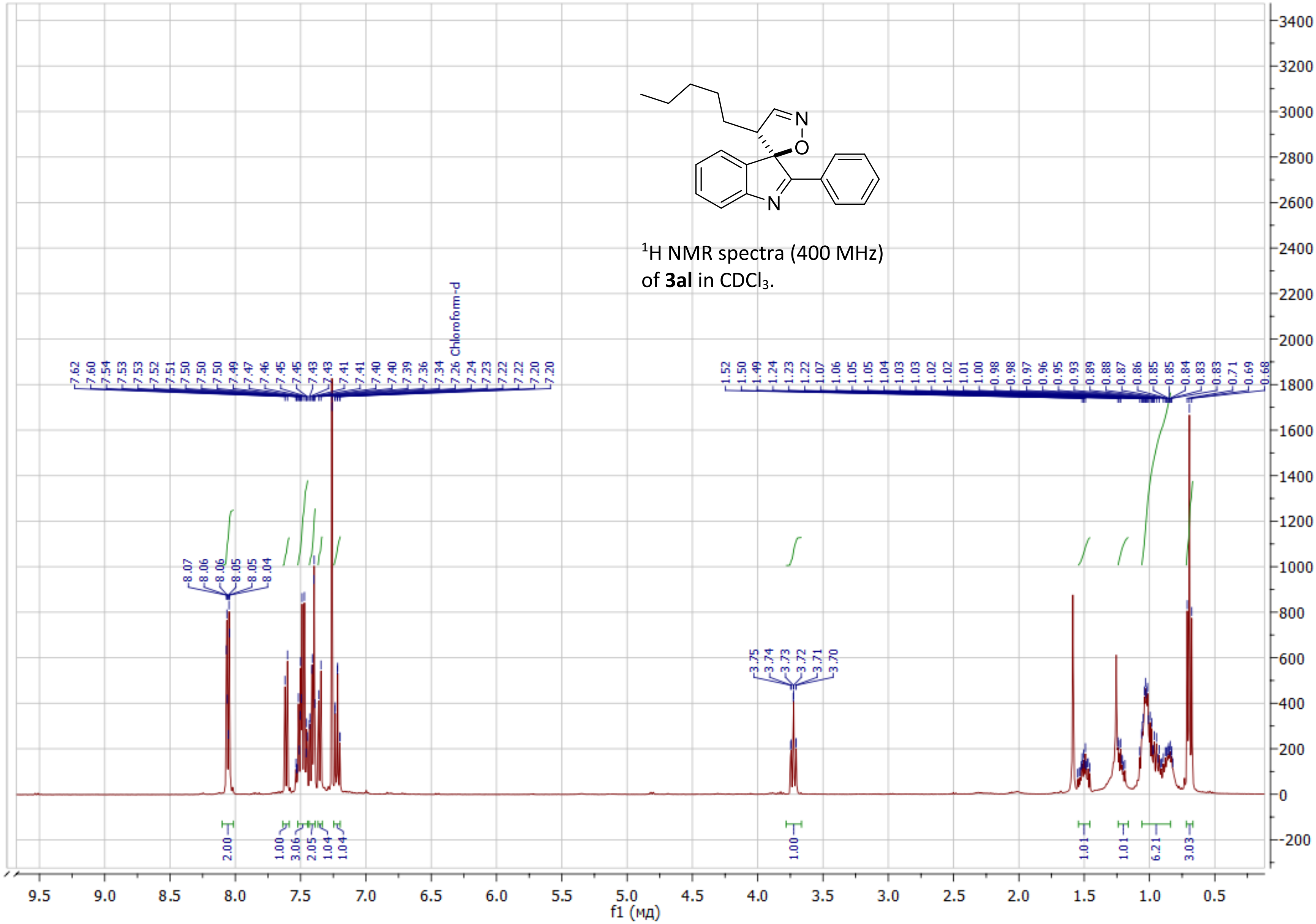


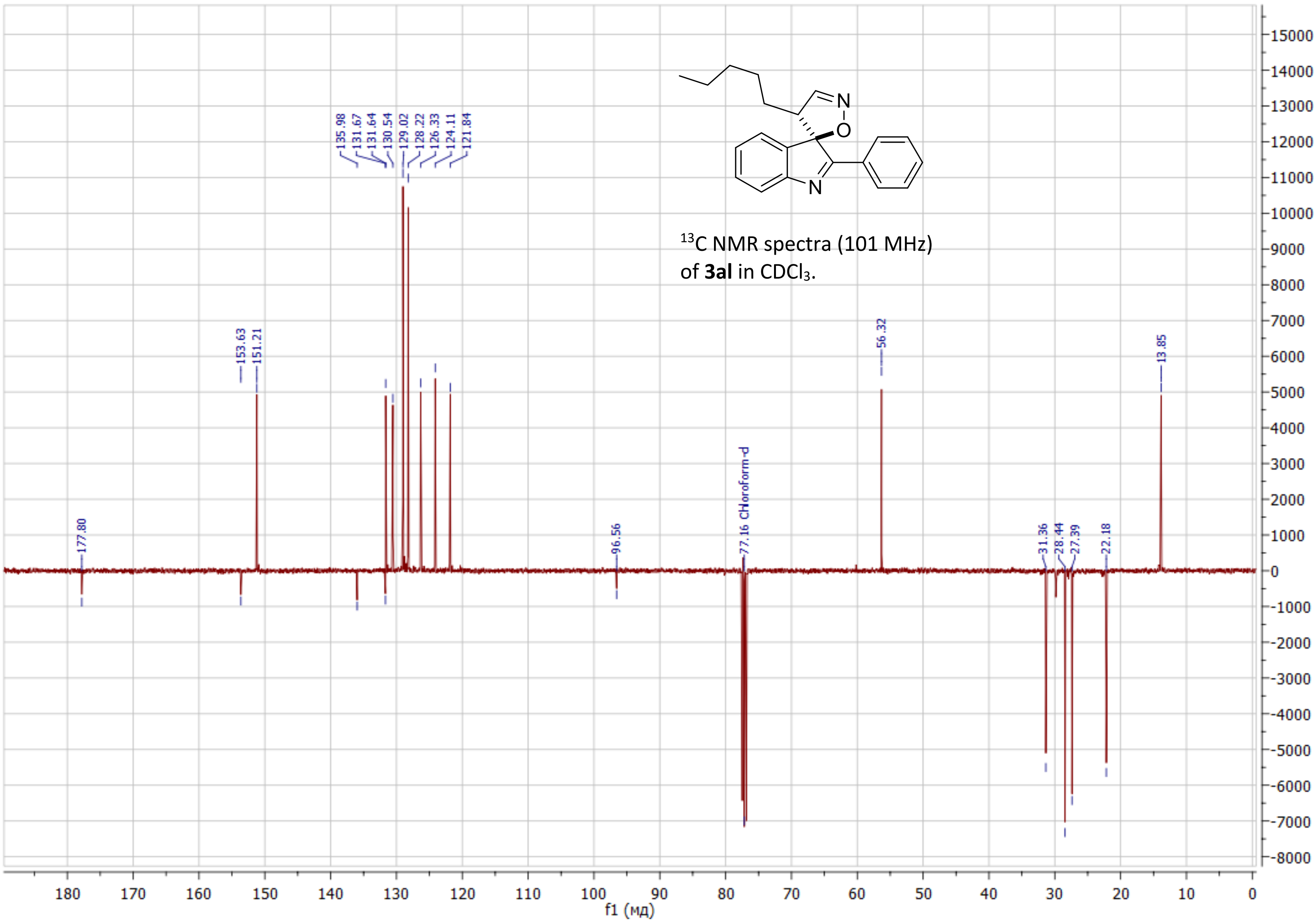
^{1}H NMR spectra (400 MHz) of **3ak** in $CDCl_3$.
f1 (мд)



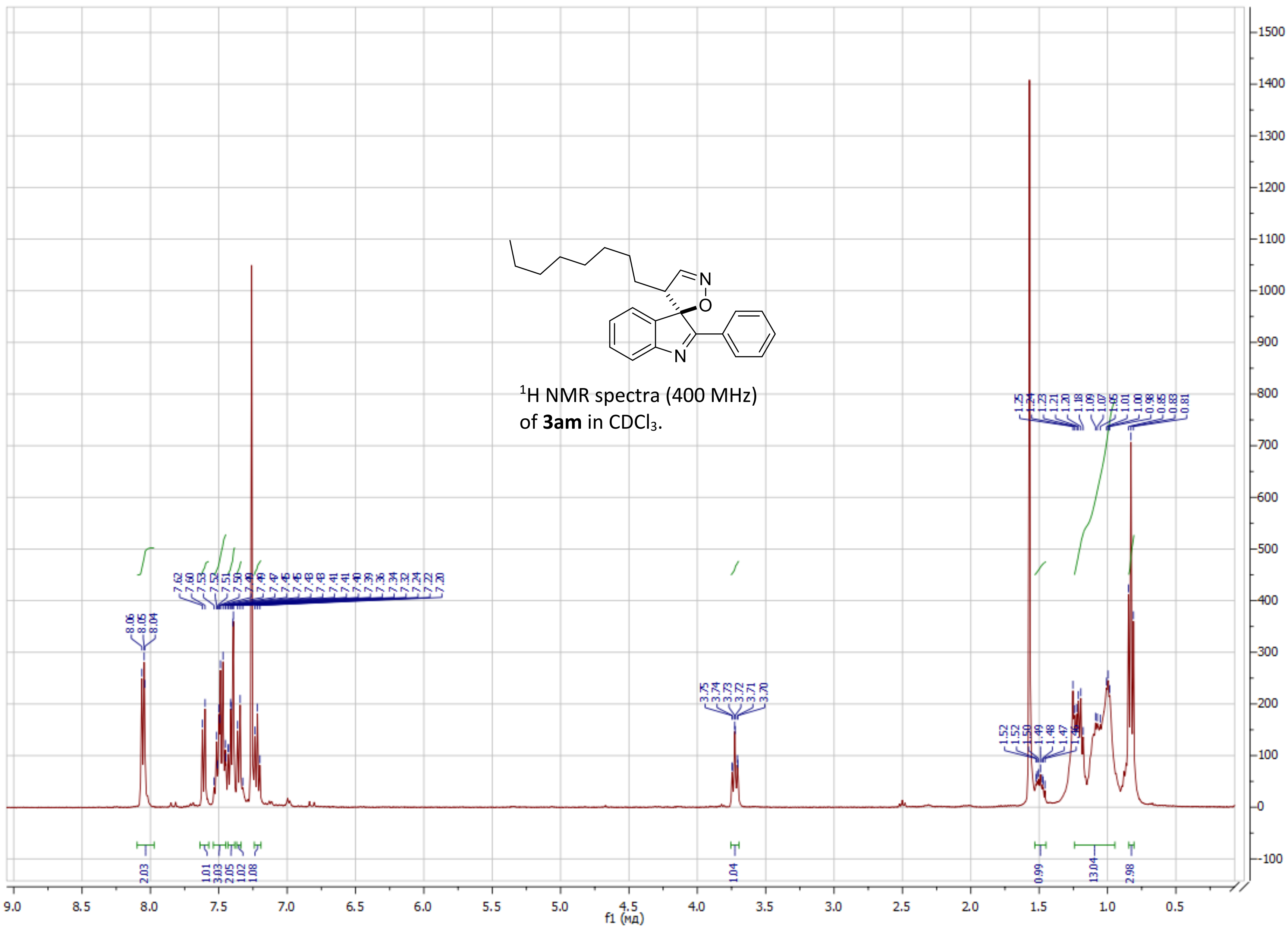
N
O
N
^{13}C NMR spectra (101 MHz)
of **3ak** in CDCl₃.
135.81
131.59
131.41
130.43
128.92
128.11
126.25
124.00
121.68
177.65
153.31
151.03
96.39
56.04
30.39
21.01
13.68
f1 (мд)



[1]H NMR spectra (400 MHz) of **3al** in $CDCl_3$.



^{13}C NMR spectra (101 MHz) of **3al** in $CDCl_3$.



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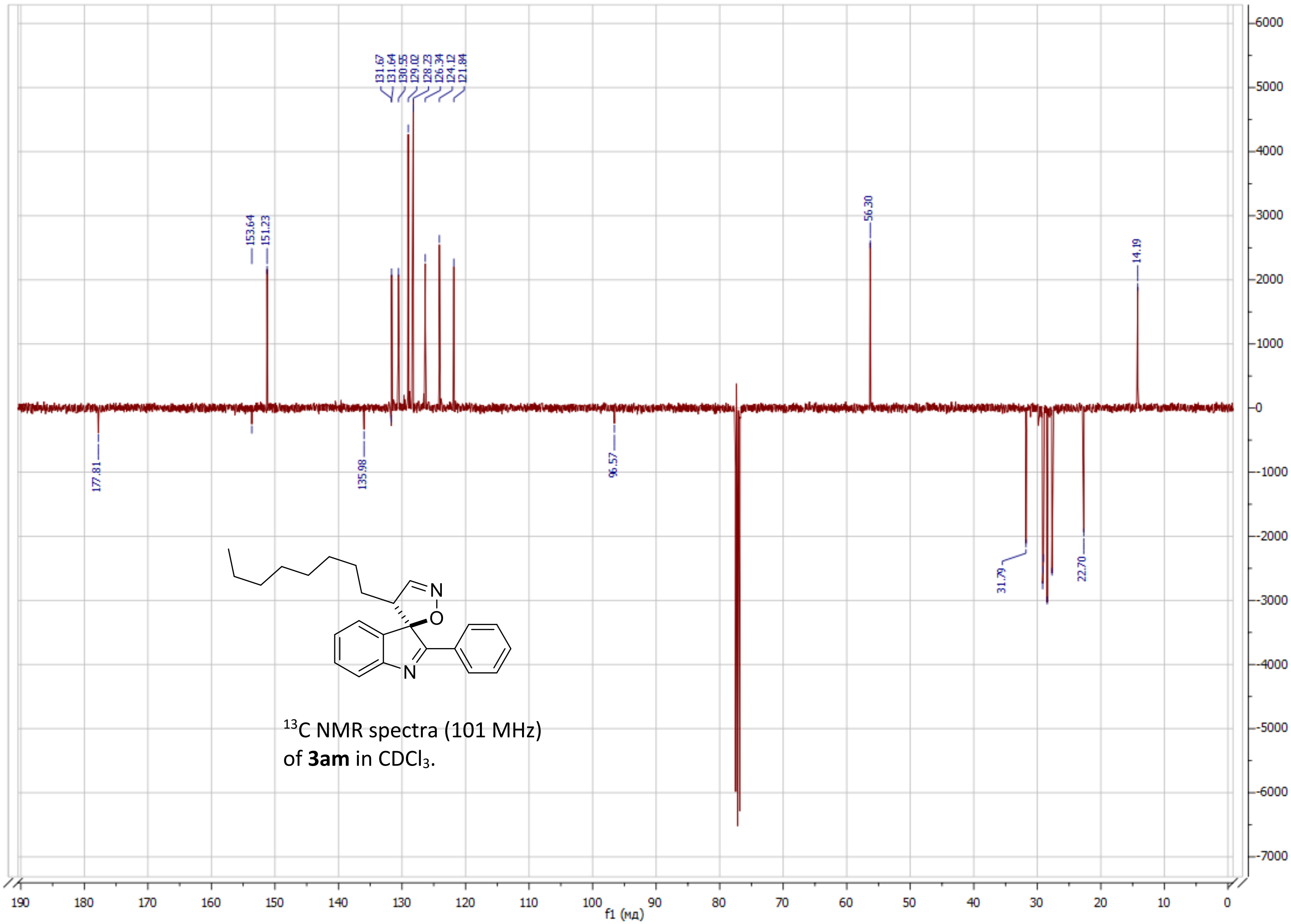
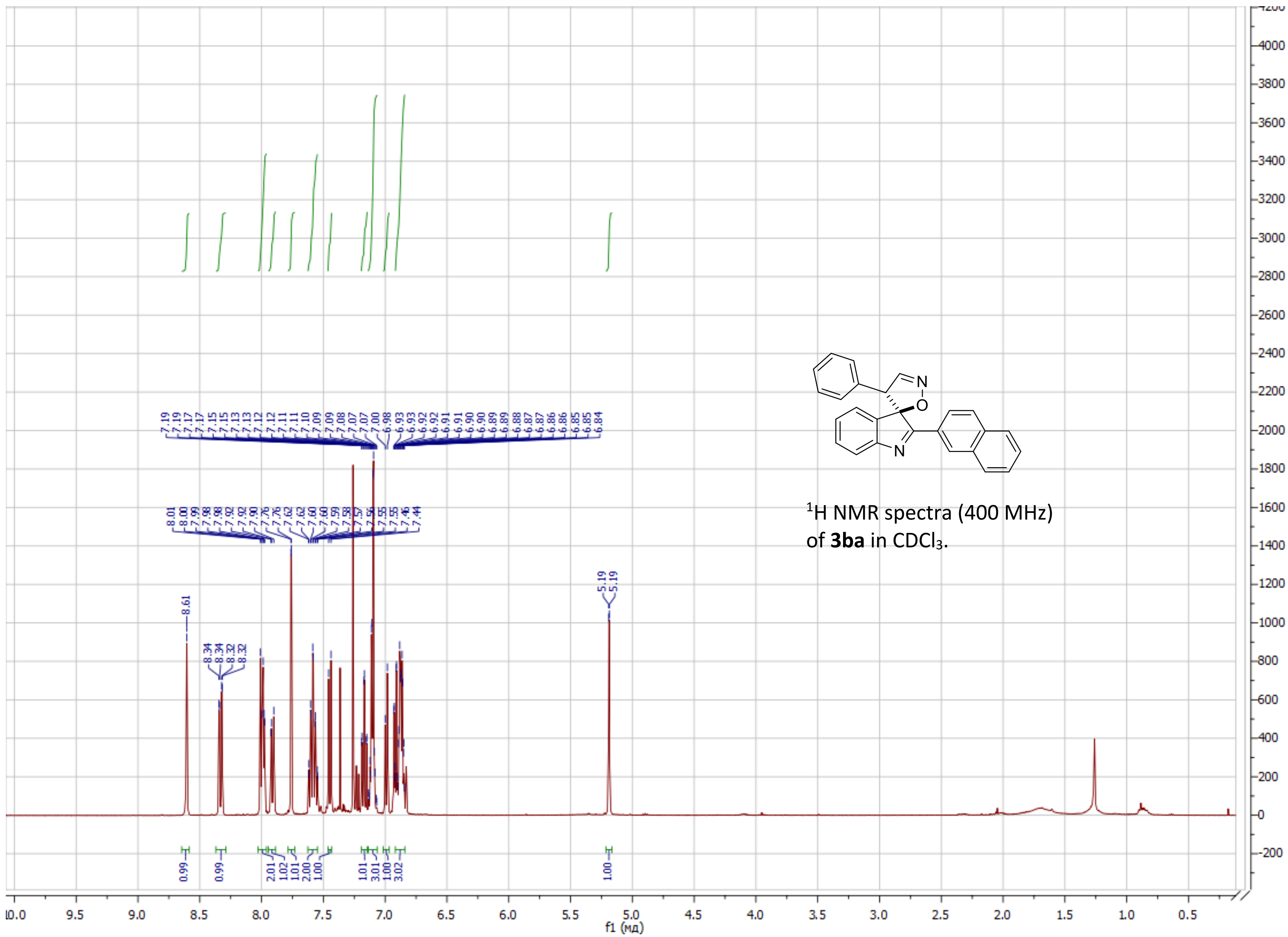
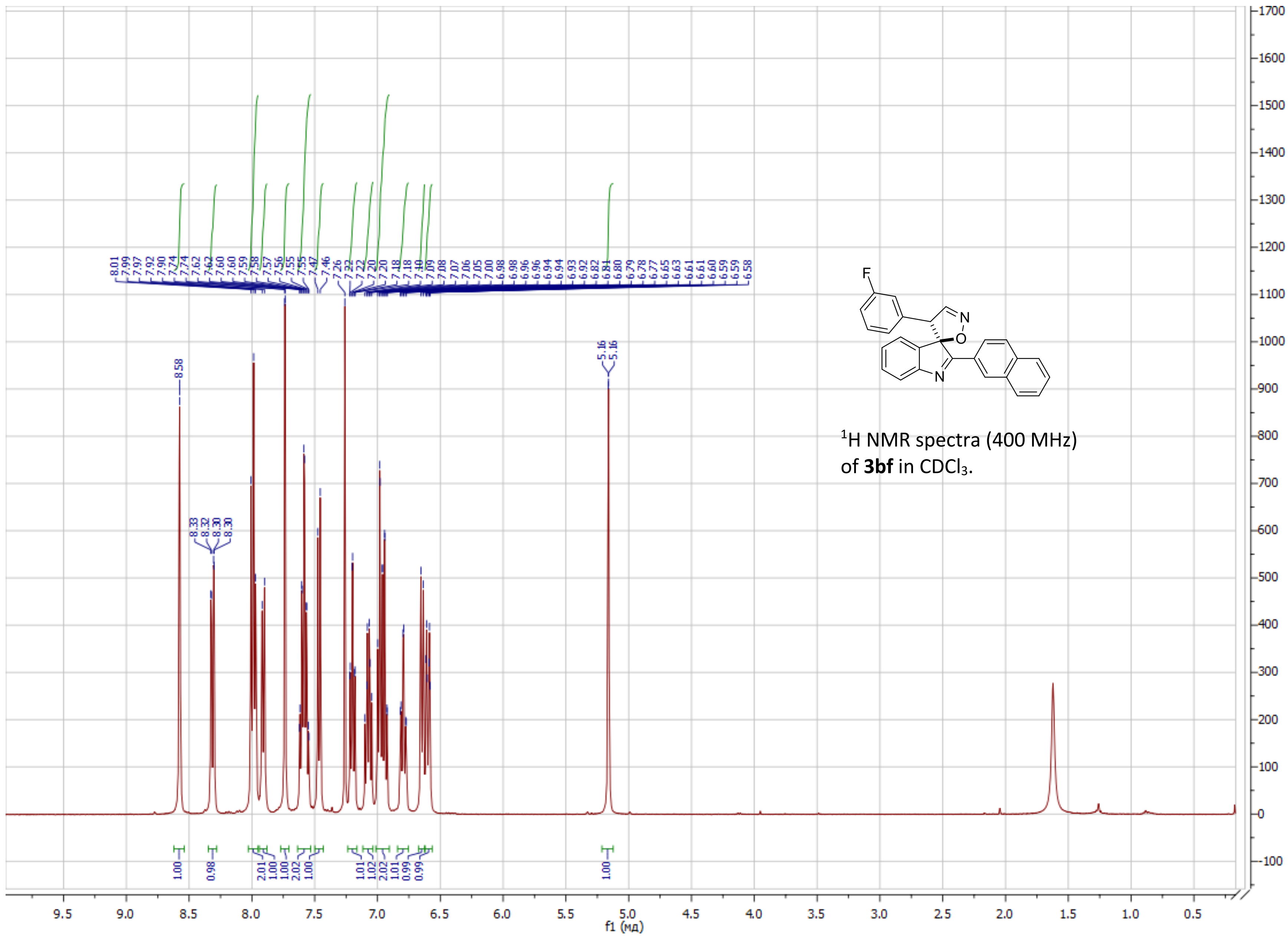


[13]C NMR spectra (101 MHz) of **3am** in $CDCl_3$.



[1]H NMR spectra (400 MHz) of **3ba** in $CDCl_3$.



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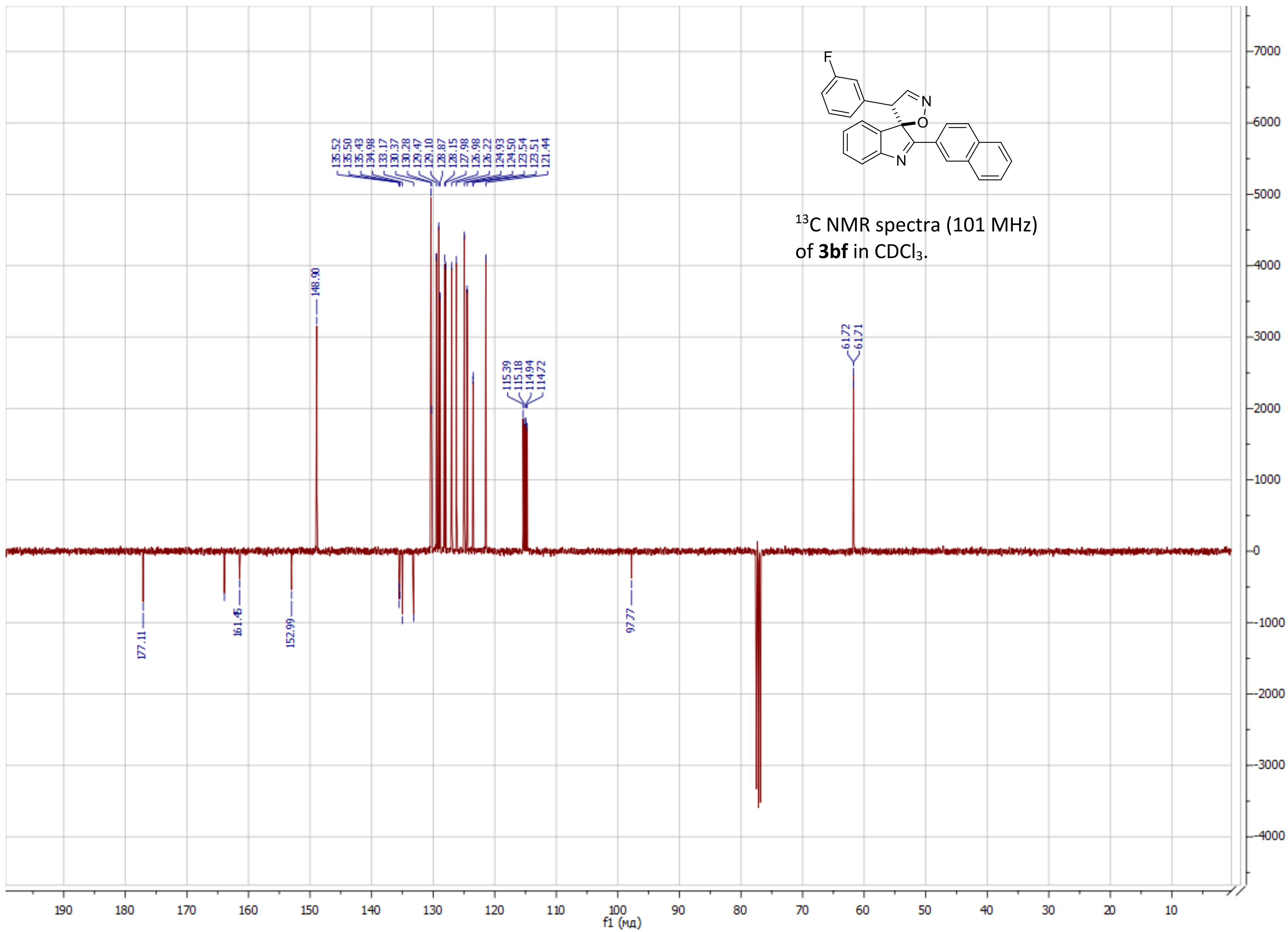
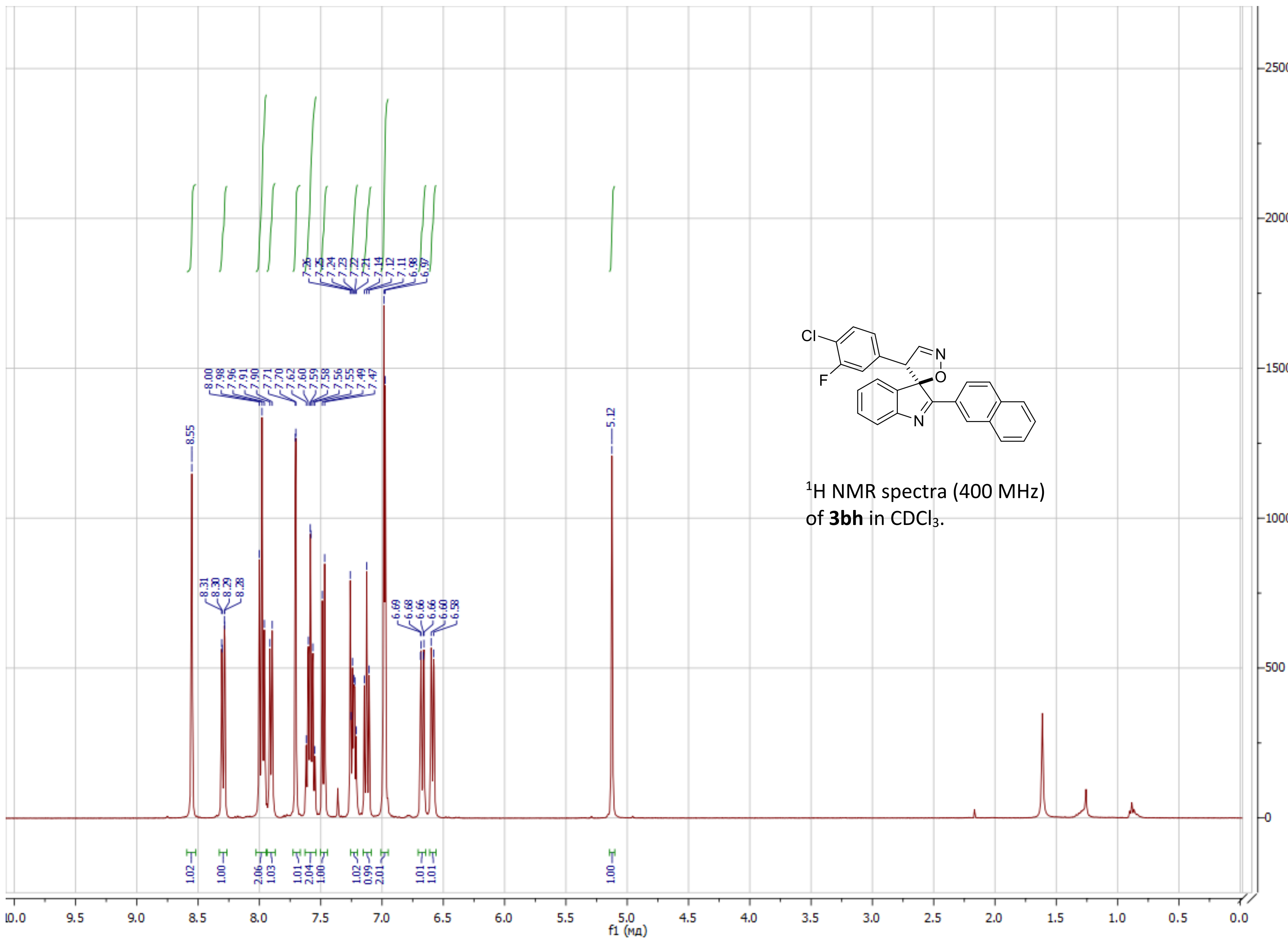
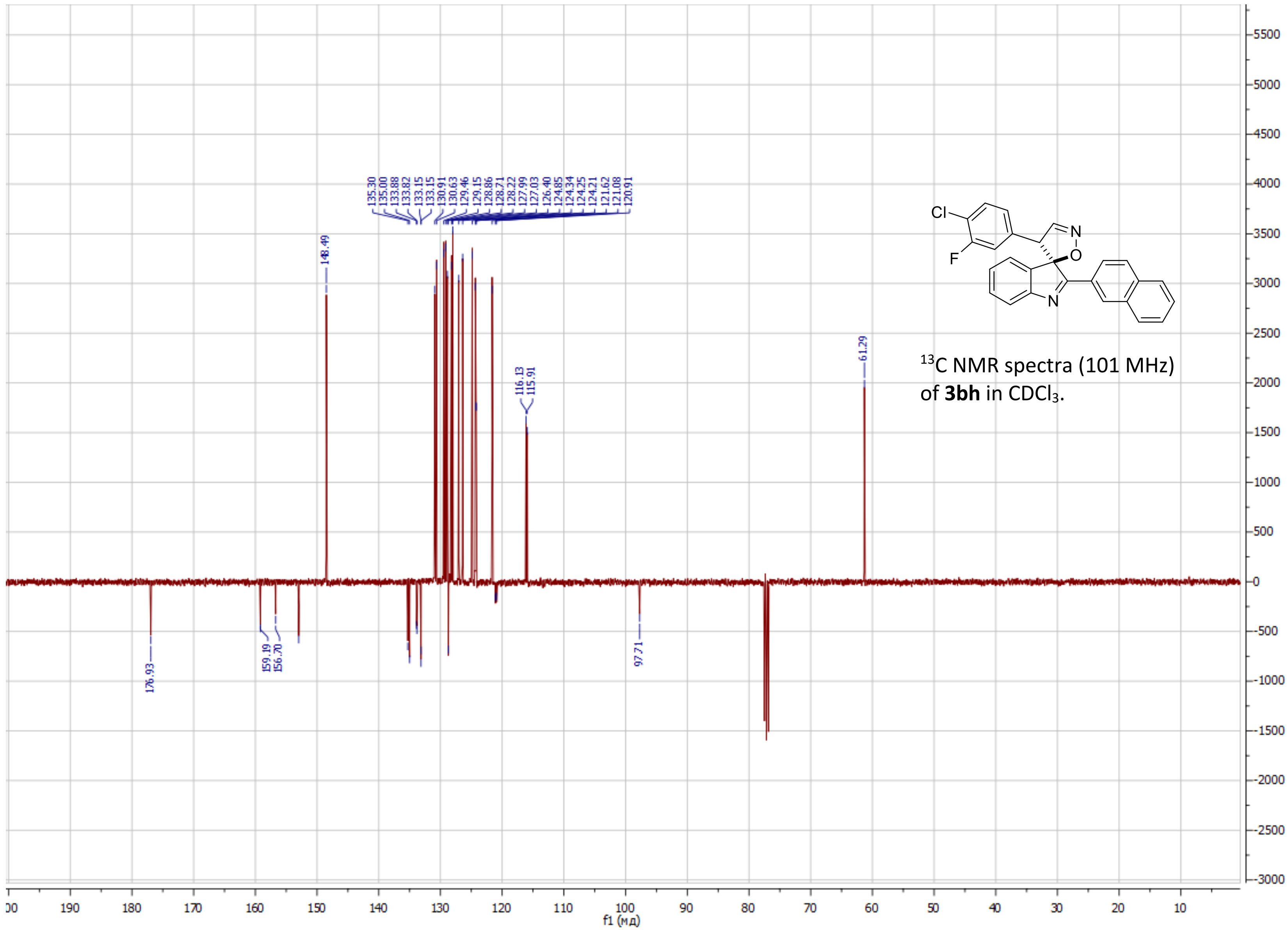


^{13}C NMR spectra (101 MHz) of **3bf** in $CDCl_3$.



^{1}H NMR spectra (400 MHz) of **3bh** in $CDCl_3$.



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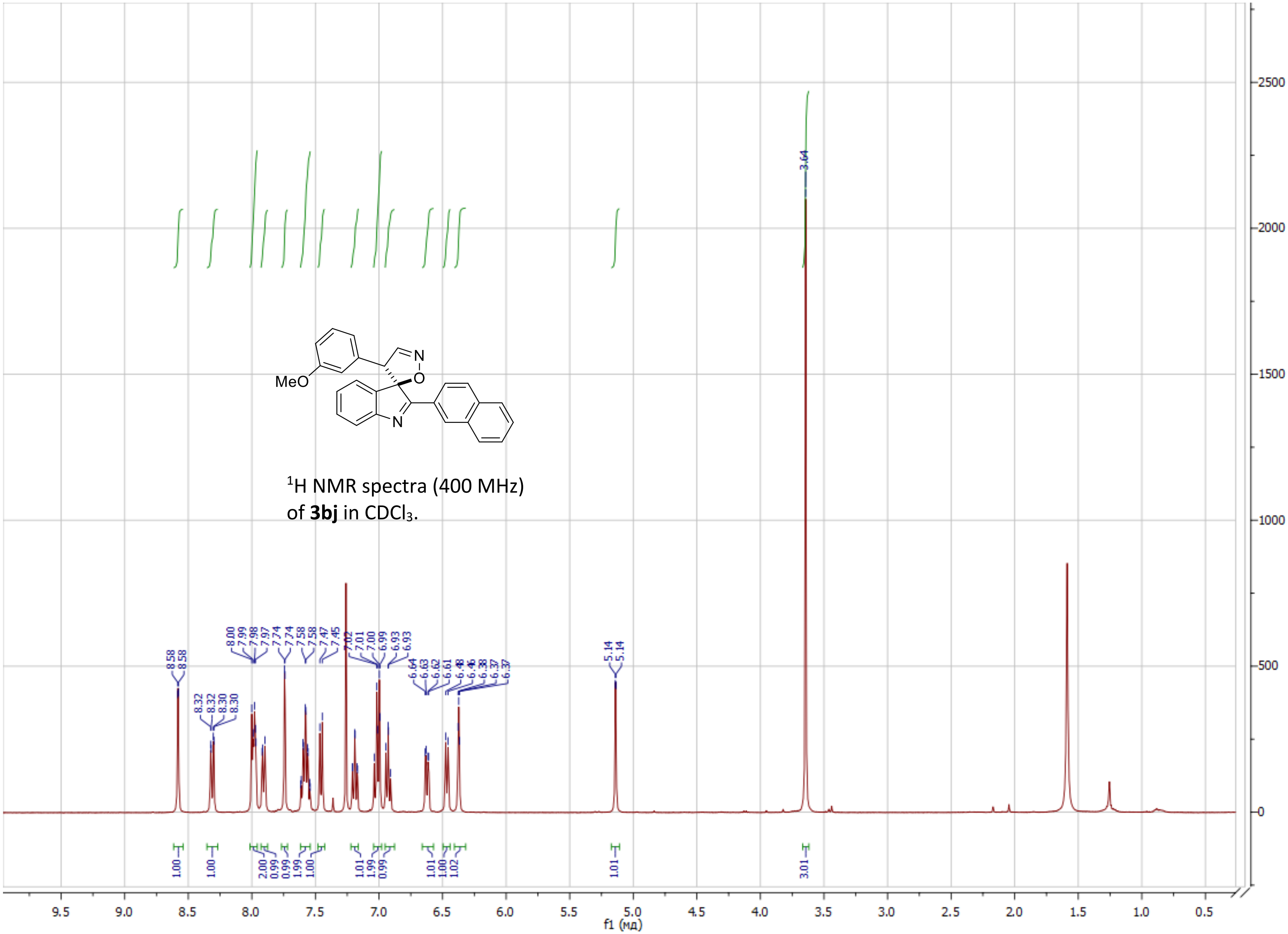
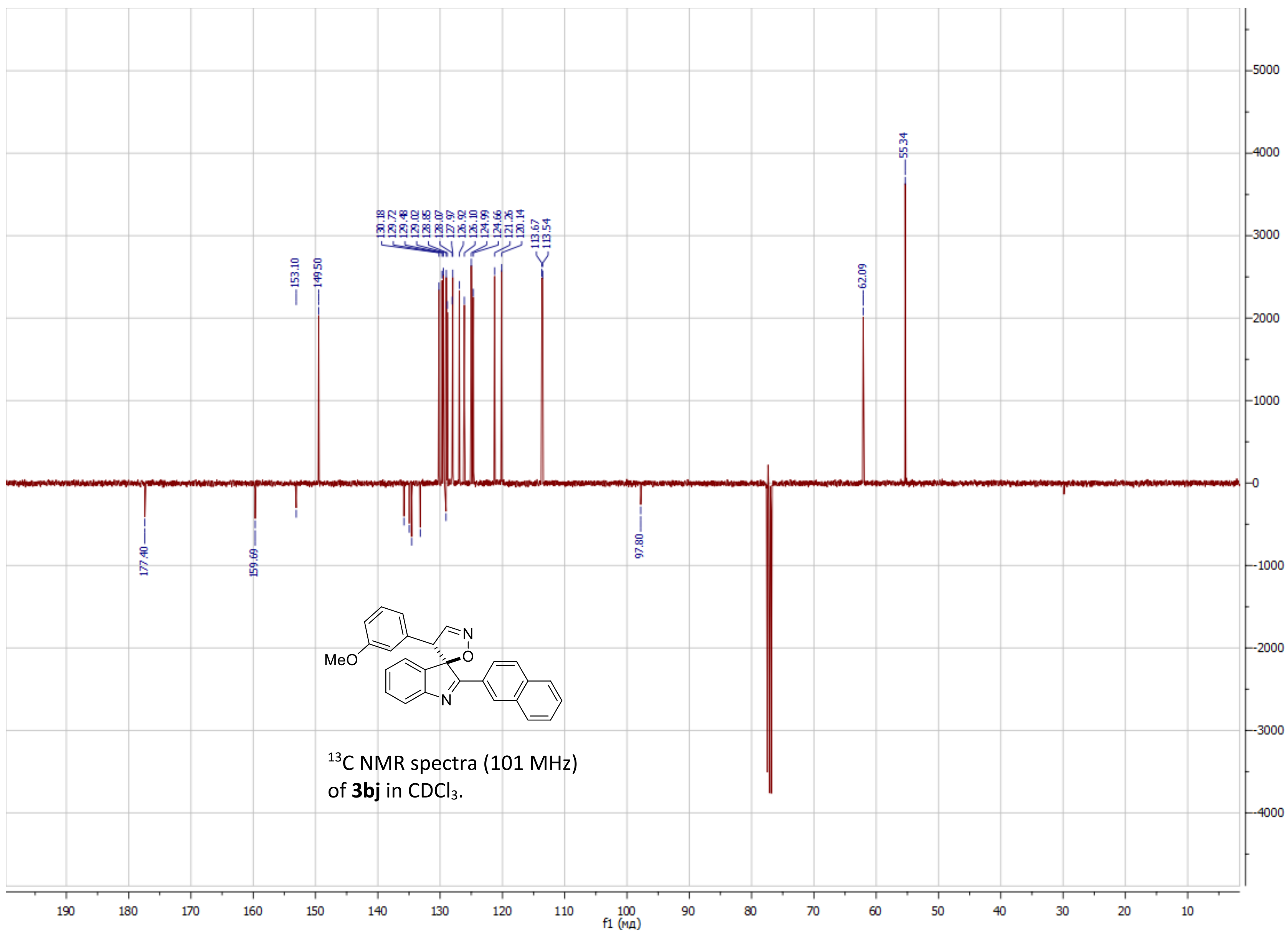
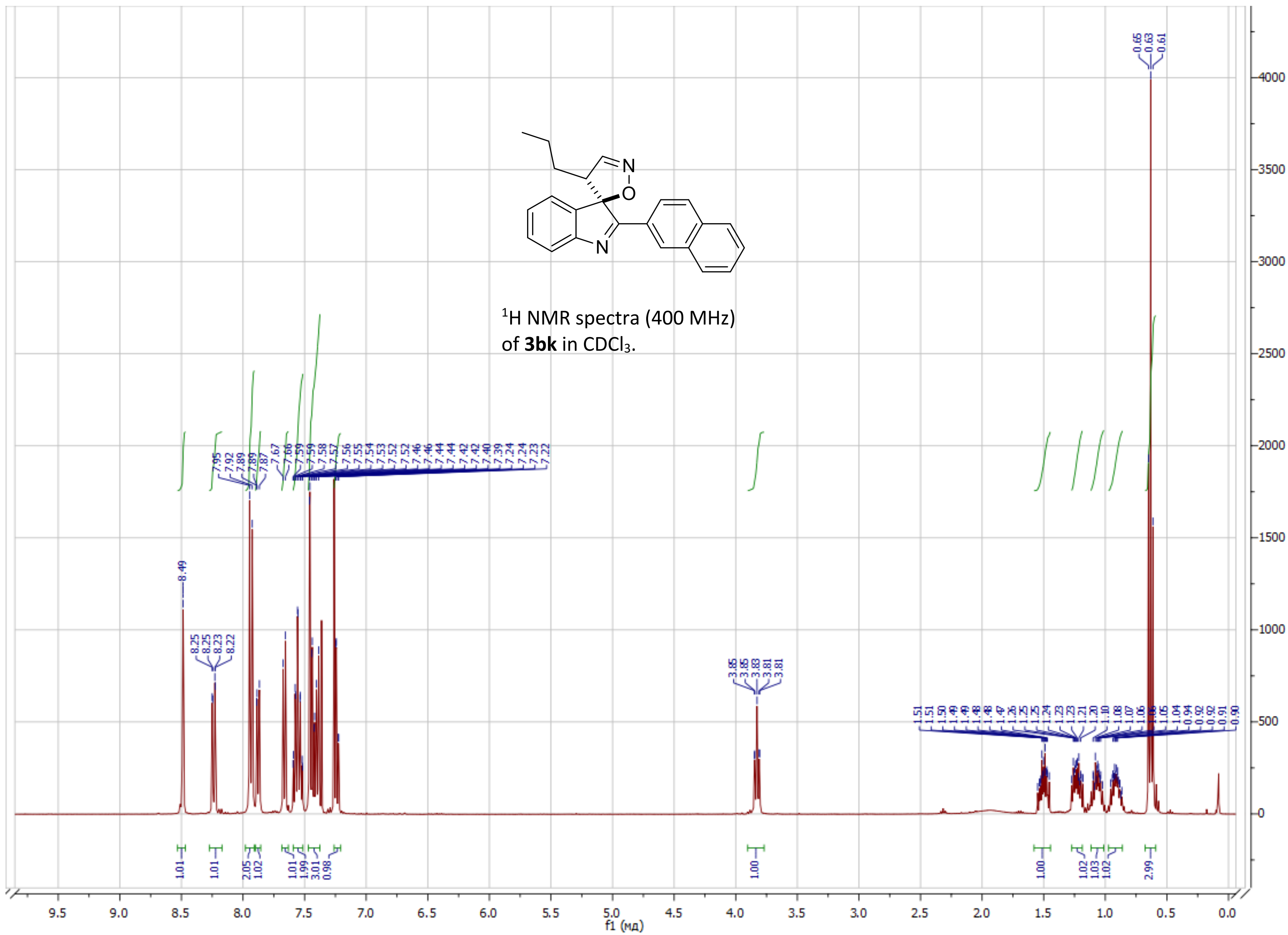


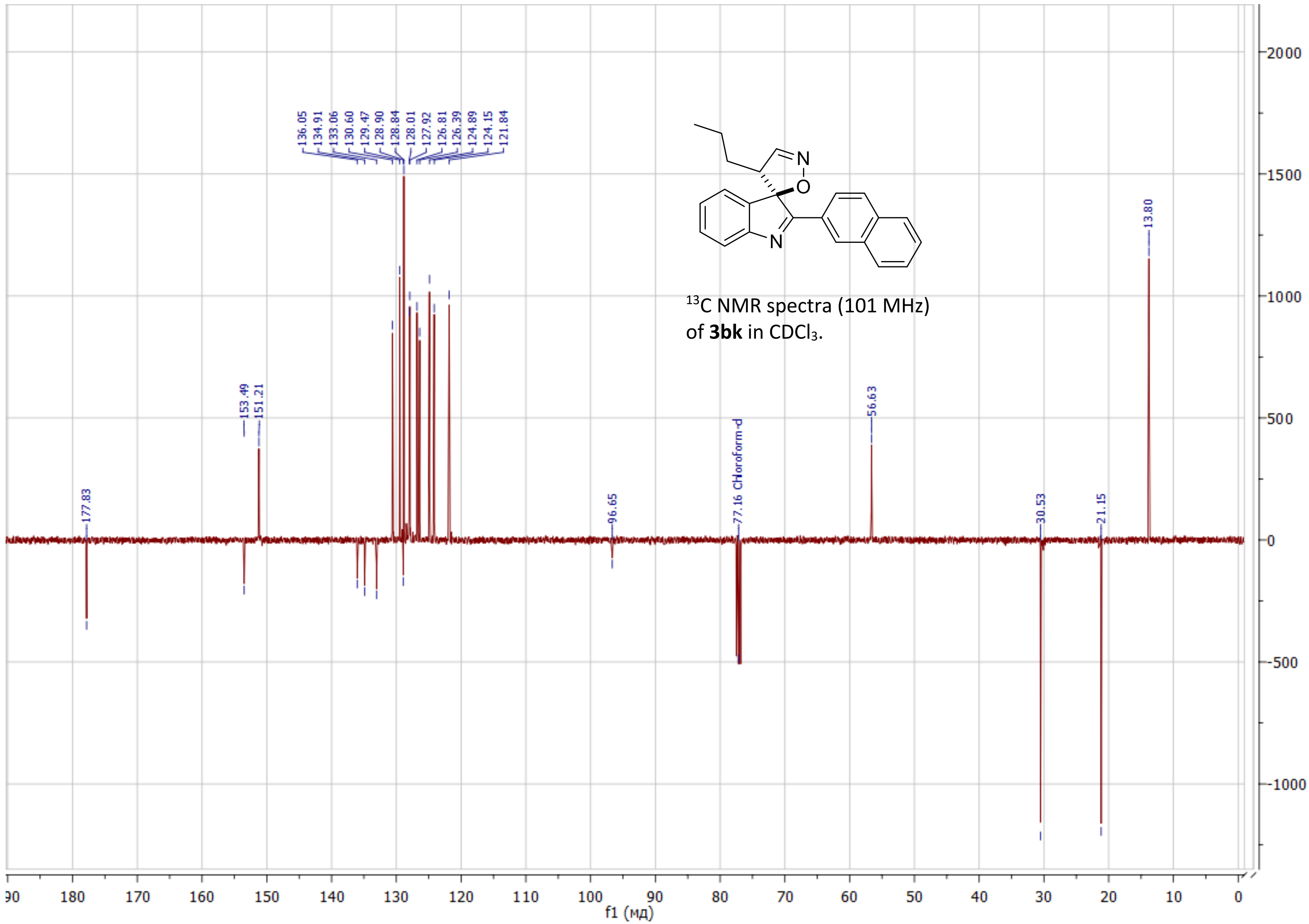
[1]H NMR spectra (400 MHz) of **3bj** in $CDCl_3$.



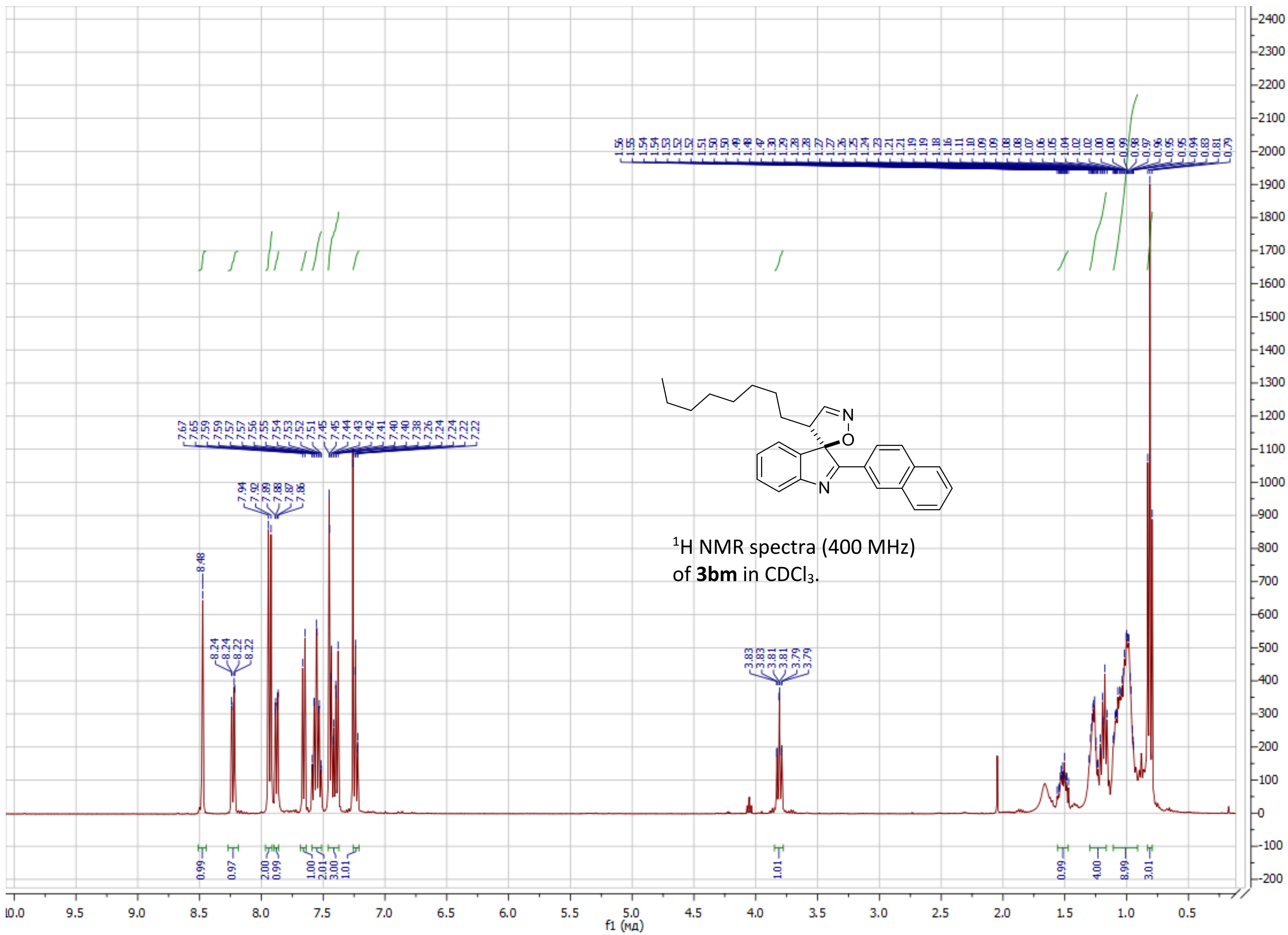
^{13}C NMR spectra (101 MHz) of **3bj** in $CDCl_3$.



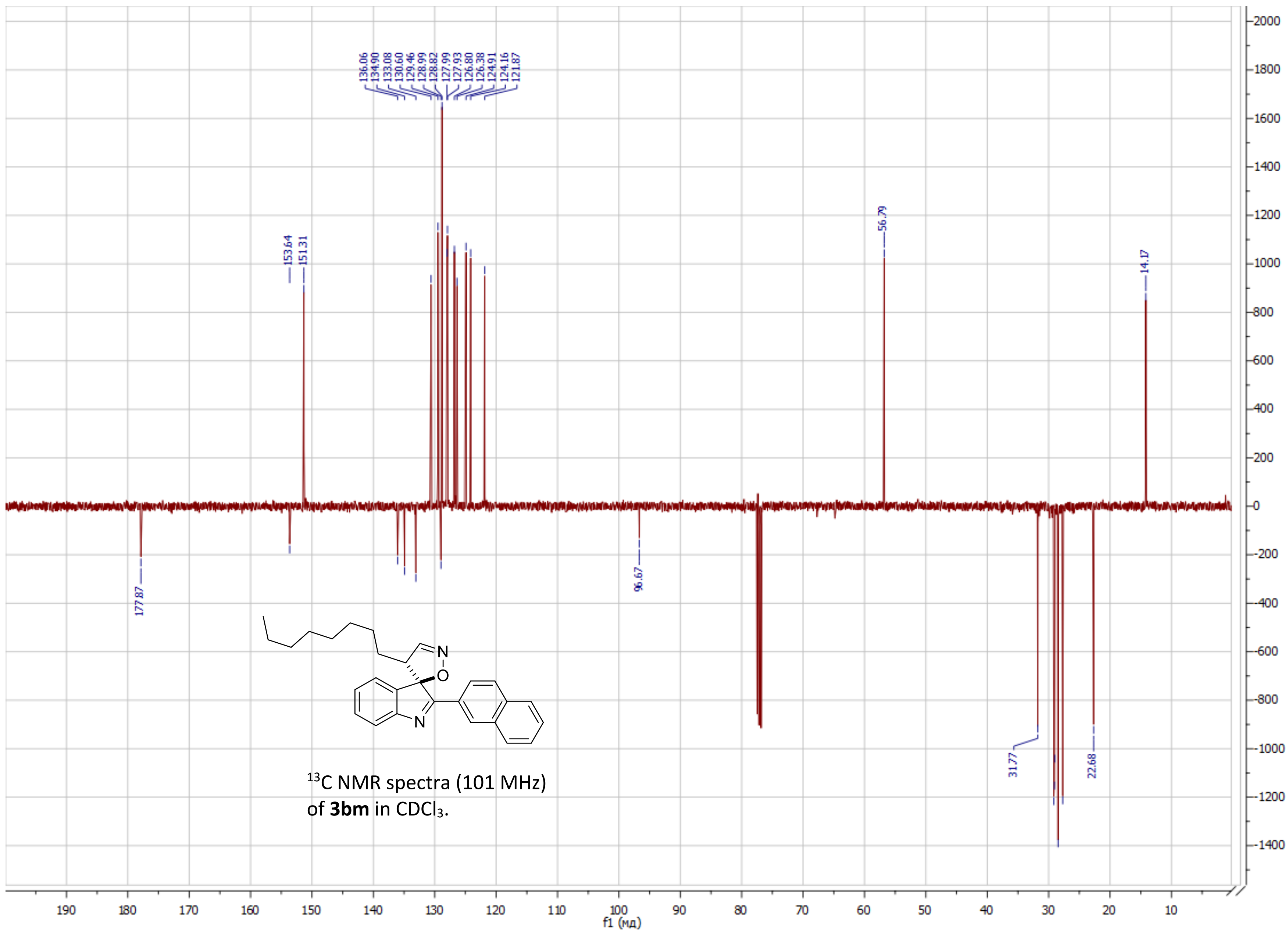
[1]H NMR spectra (400 MHz) of **3bk** in $CDCl_3$.



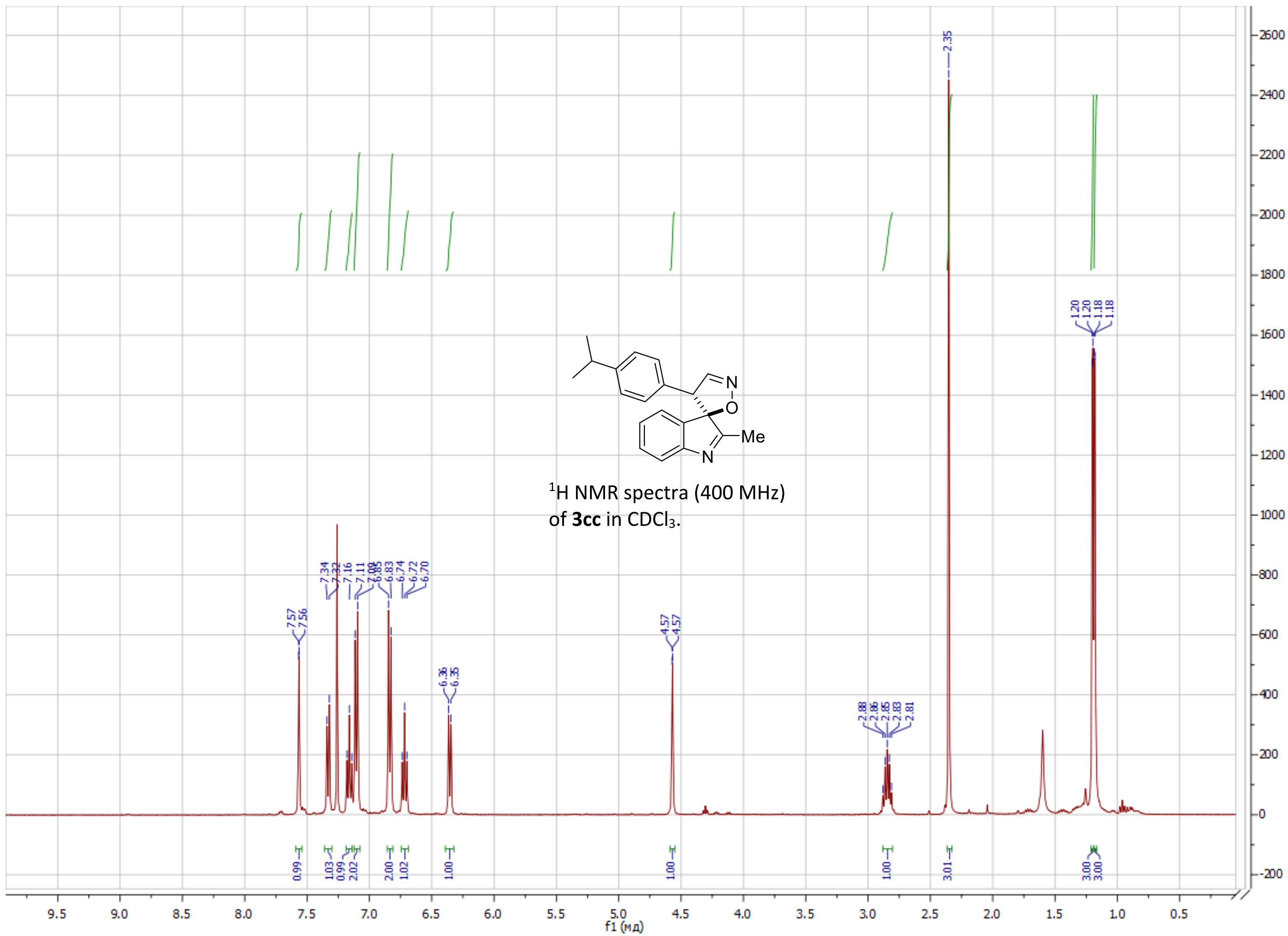
^{13}C NMR spectra (101 MHz) of **3bk** in $CDCl_3$.



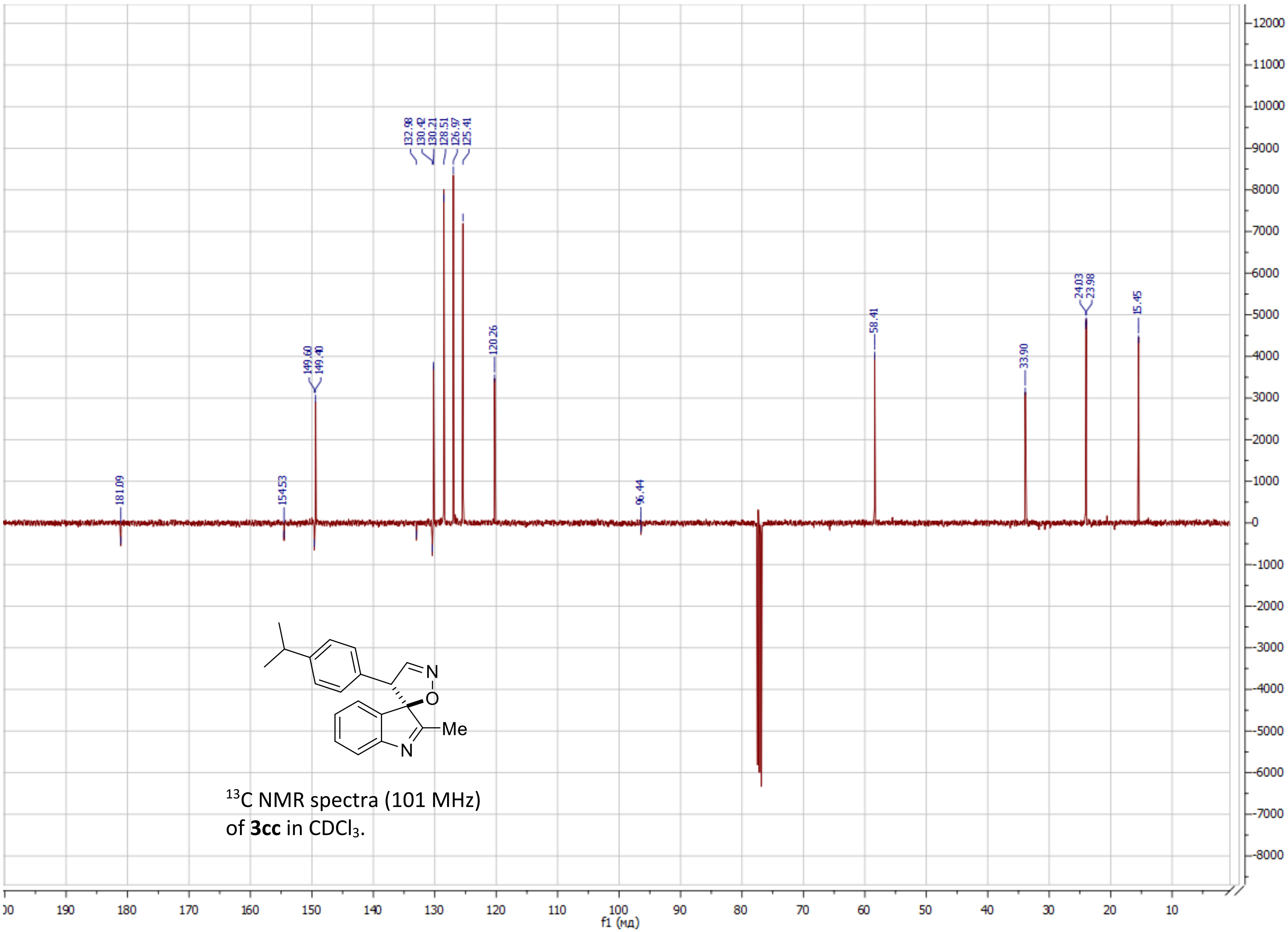
[1]H NMR spectra (400 MHz) of **3bm** in $CDCl_3$.



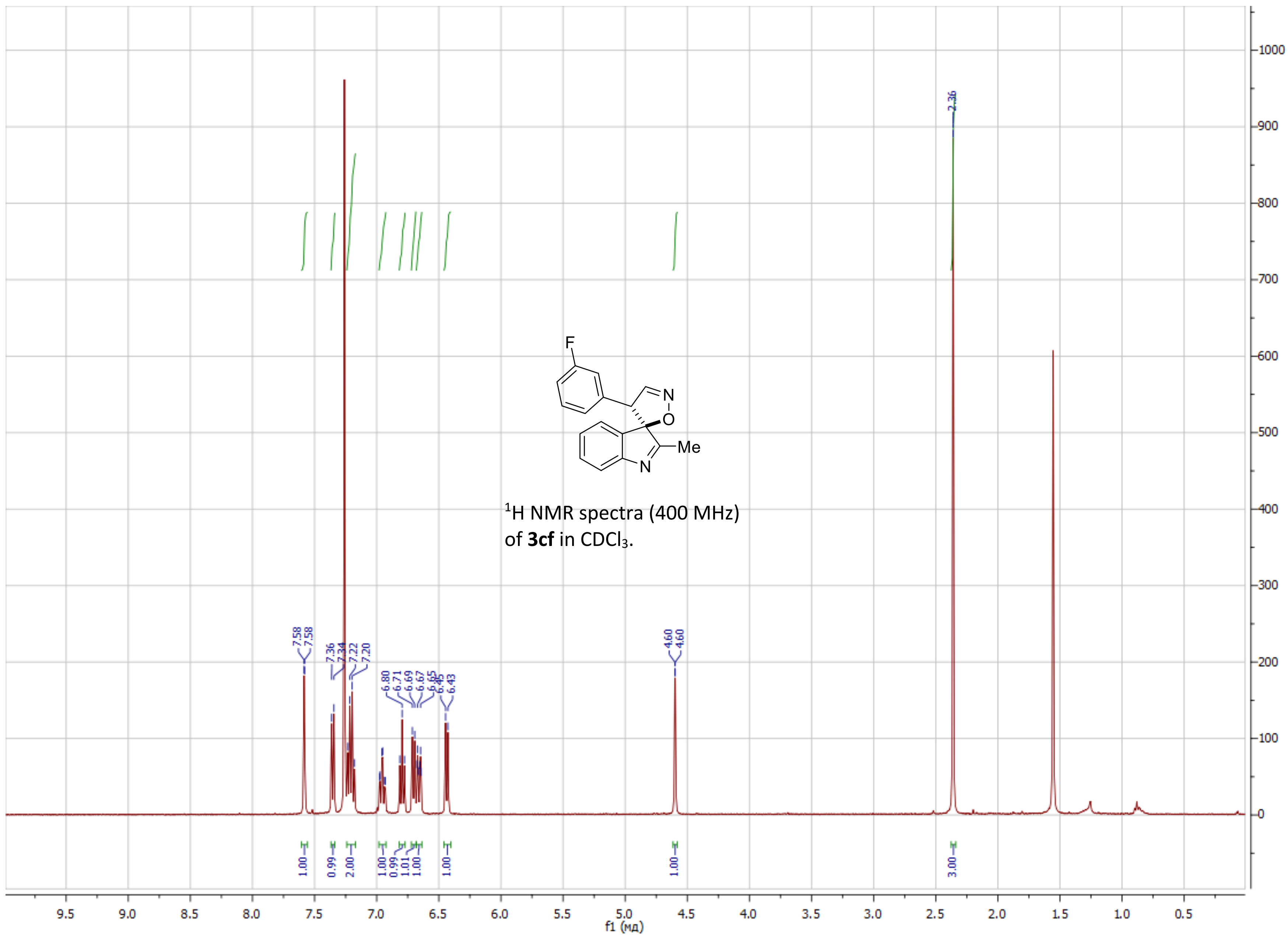
^{13}C NMR spectra (101 MHz) of **3bm** in $CDCl_3$.



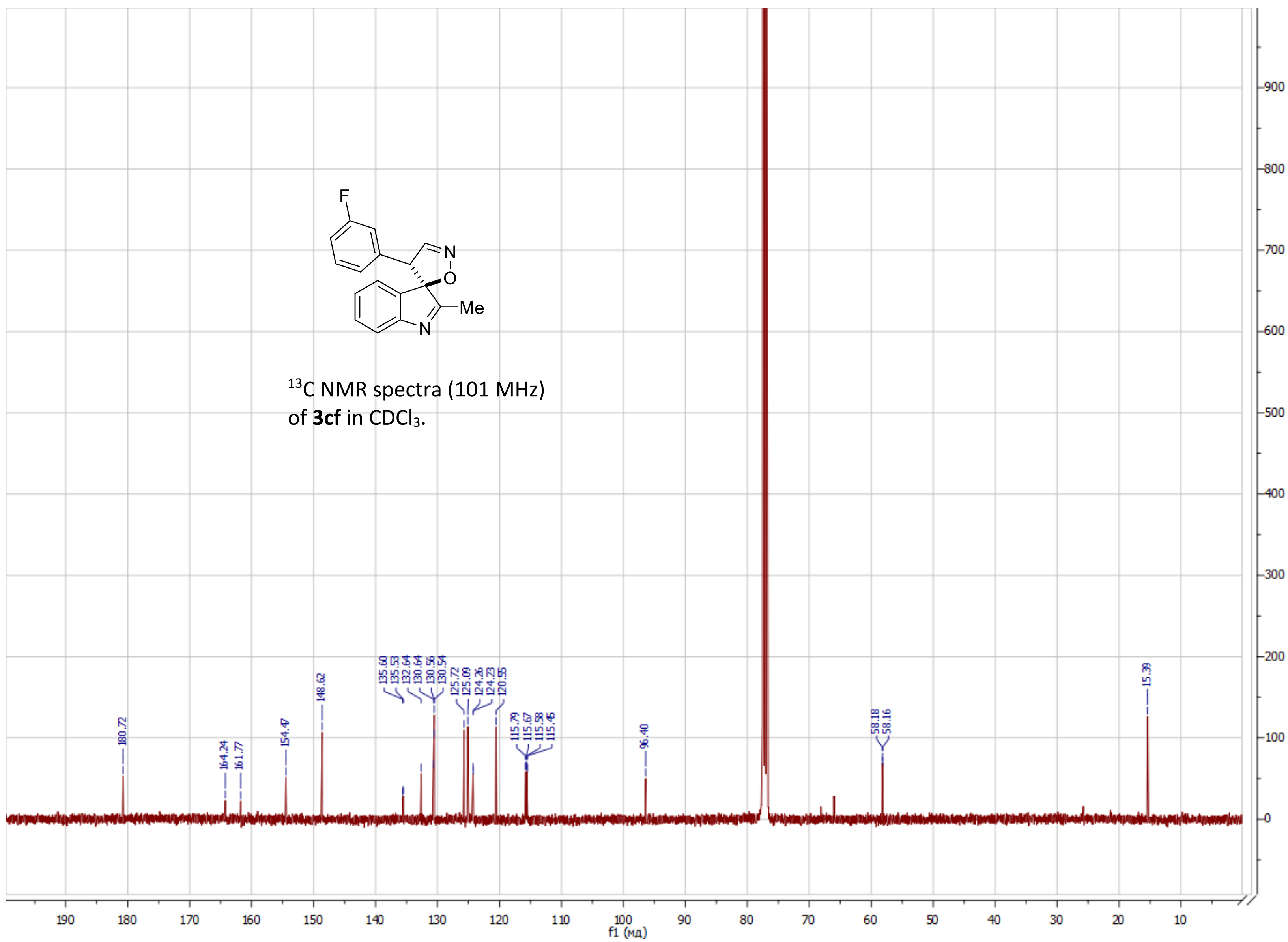
[1]H NMR spectra (400 MHz) of **3cc** in $CDCl_3$.

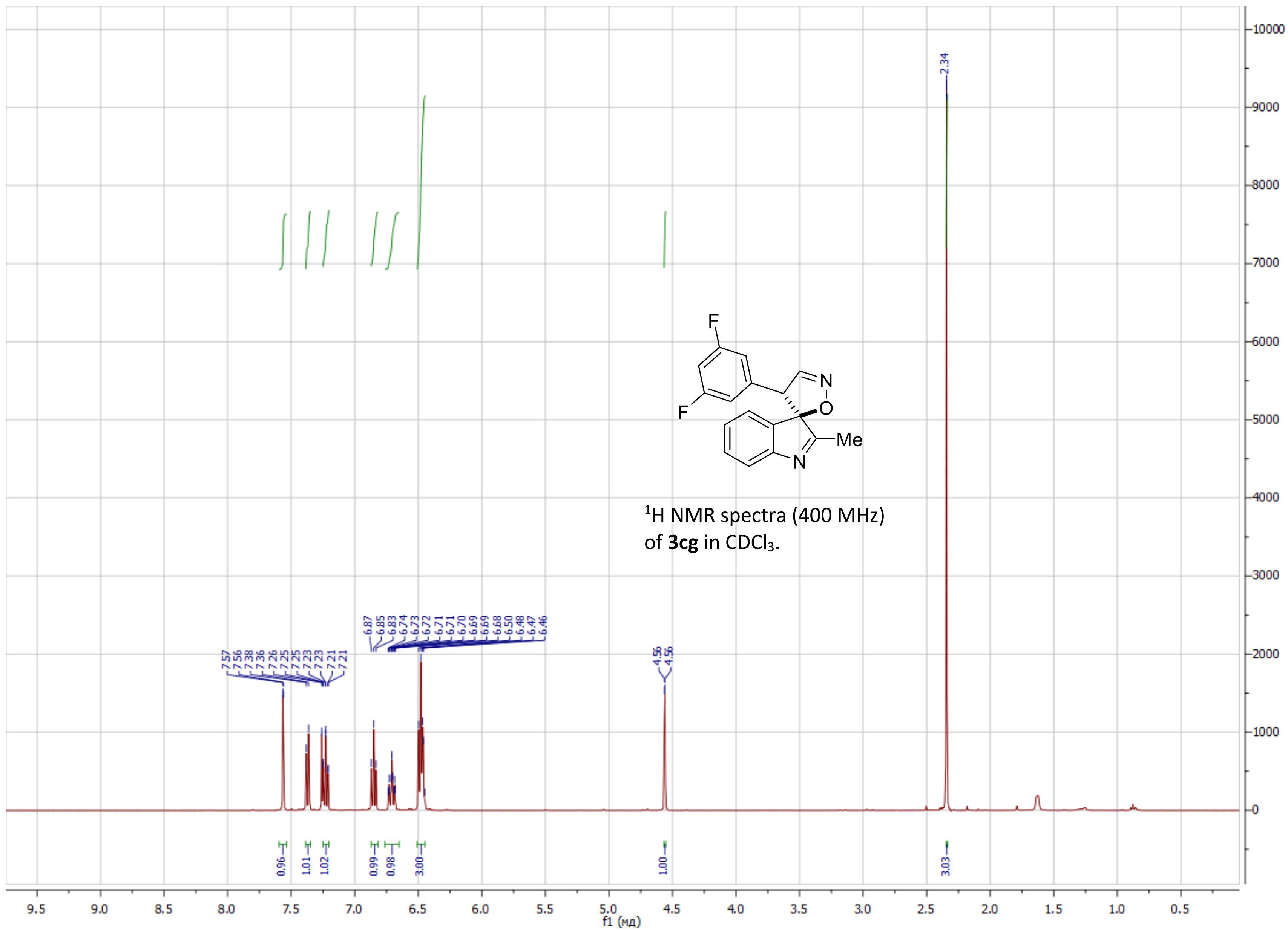


^{13}C NMR spectra (101 MHz) of **3cc** in $CDCl_3$.

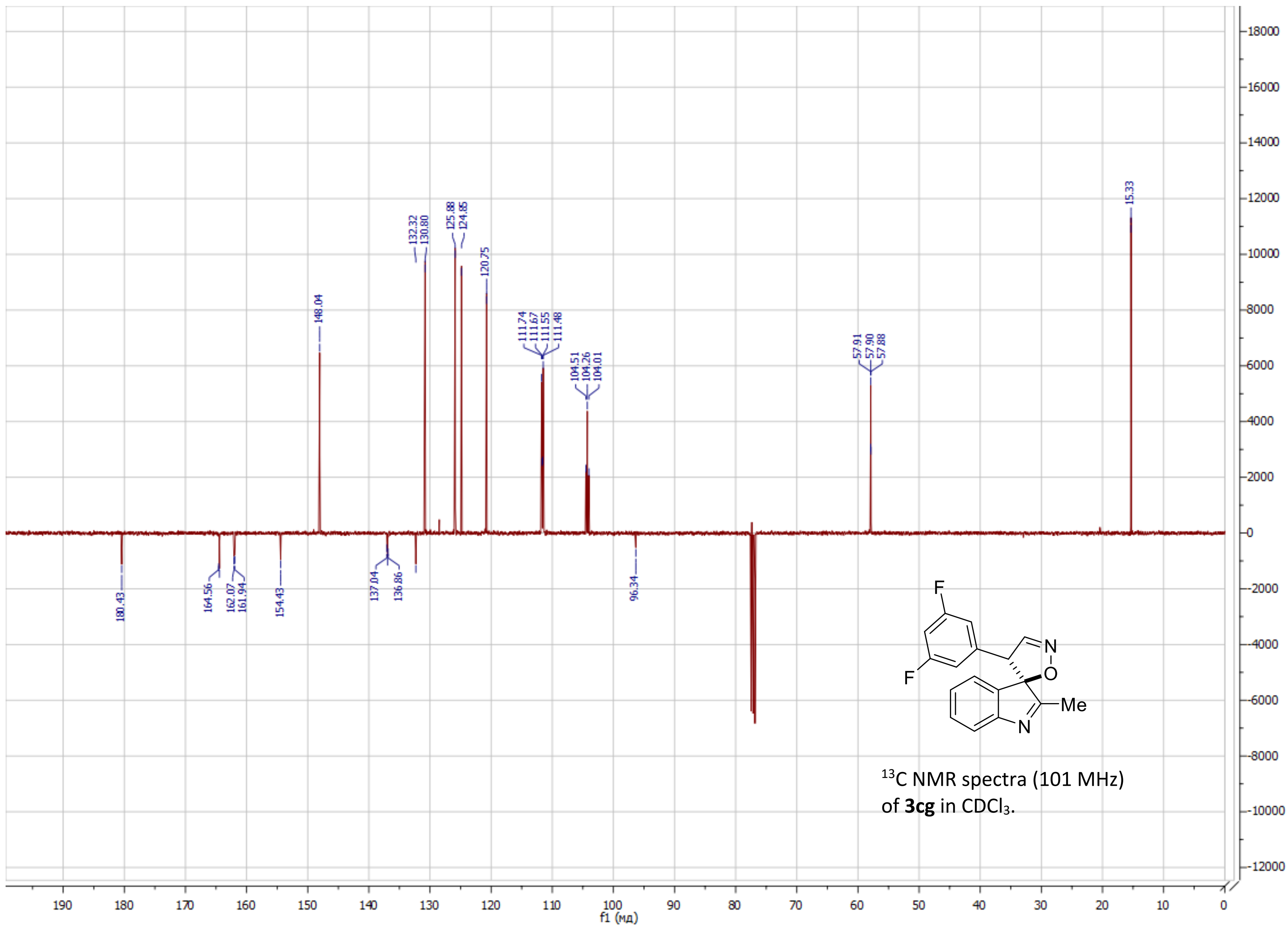


^{1}H NMR spectra (400 MHz) of **3cf** in $CDCl_3$.

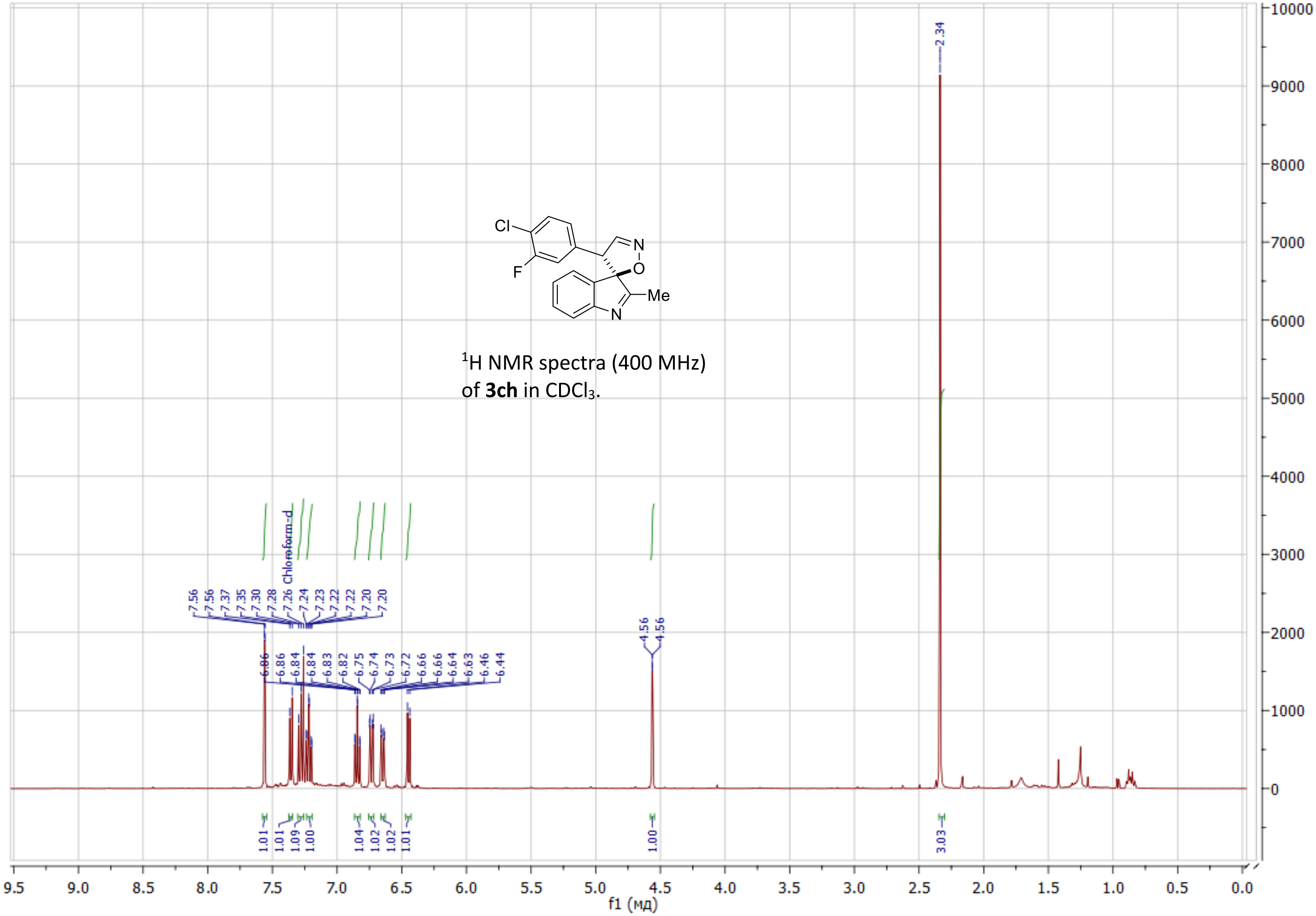

F
N
O
Me
N
^{13}C NMR spectra (101 MHz)
of **3cf** in CDCl$_3$.
180.72
164.24
161.77
154.47
148.62
135.60
135.53
132.64
130.64
130.56
130.54
125.72
125.09
124.26
124.23
120.55
115.79
115.67
115.58
115.45
96.40
58.18
58.16
15.39
f1 (мд)



[1]H NMR spectra (400 MHz)
of 3cg in CDCl₃.
F
F
N
O
N
Me
f1 (мд)



^{13}C NMR spectra (101 MHz) of **3cg** in $CDCl_3$.



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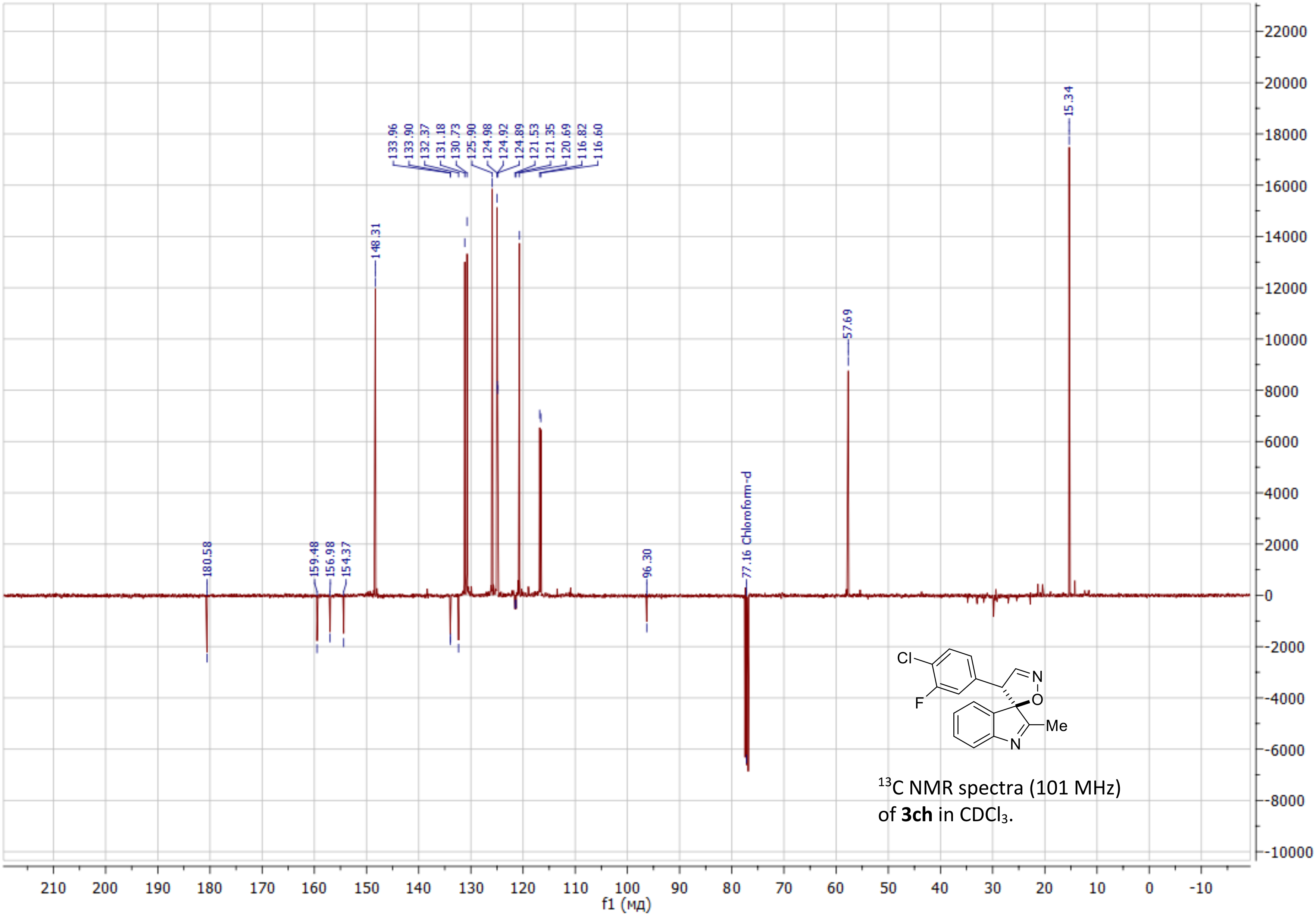
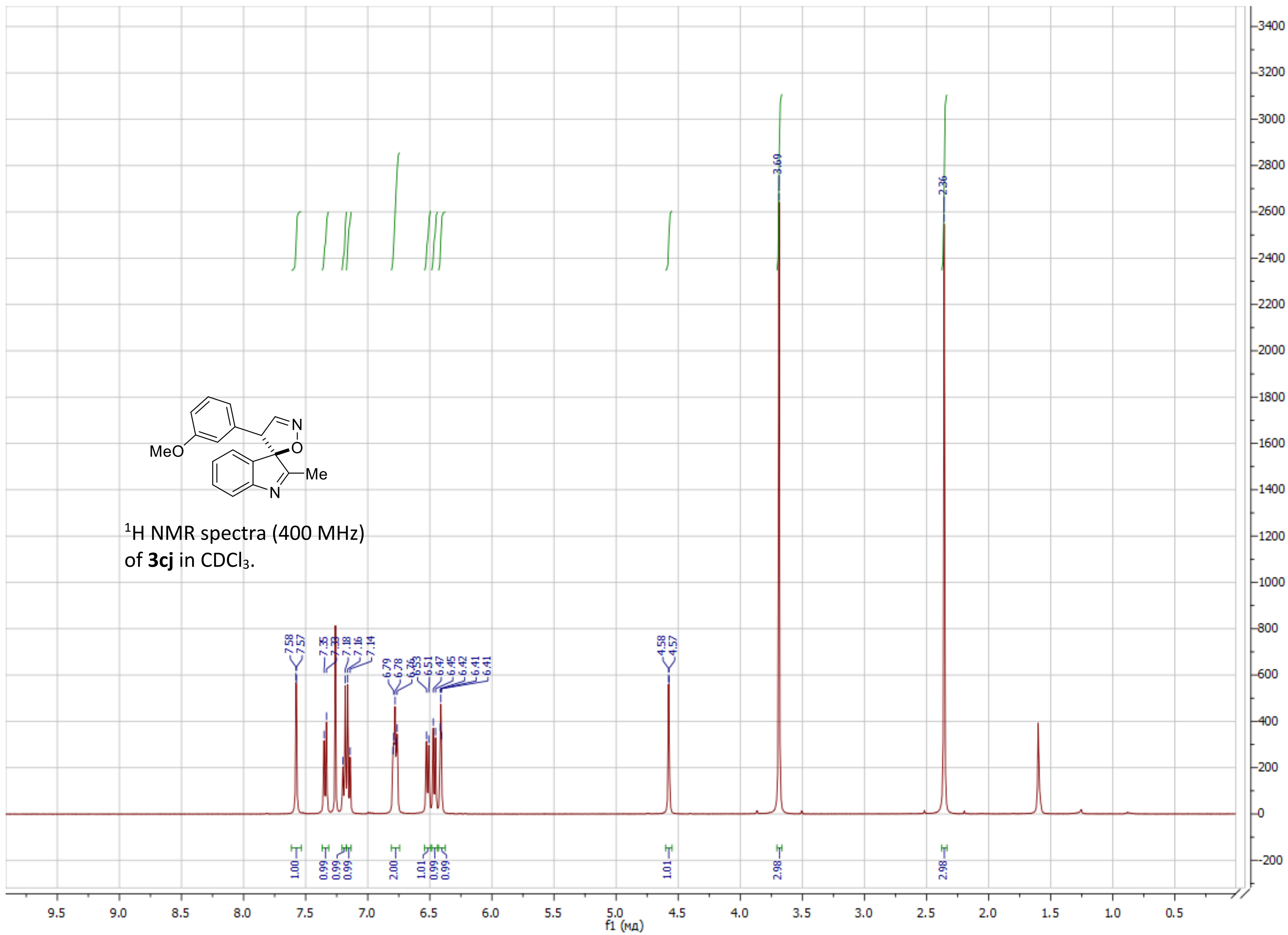
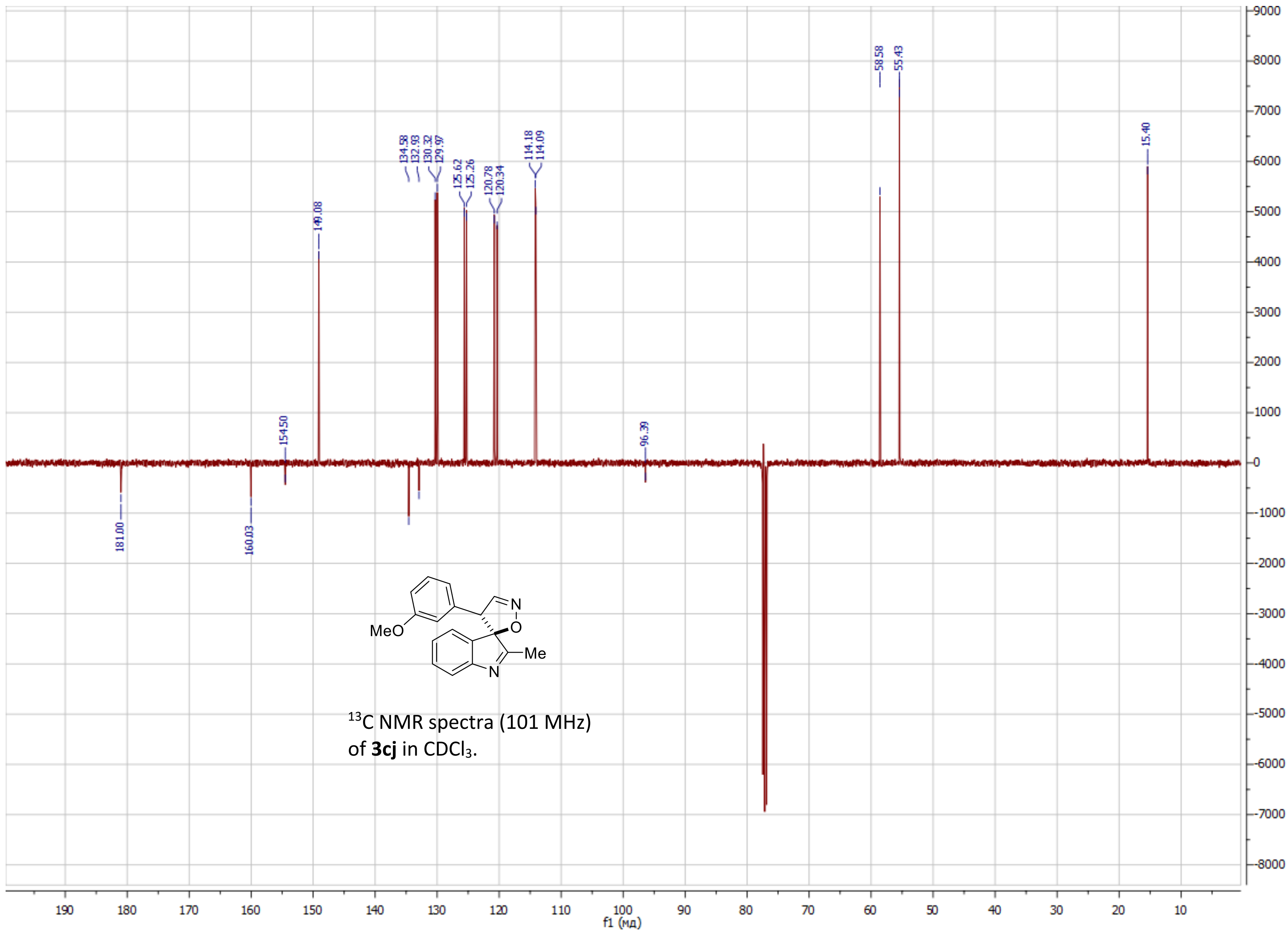
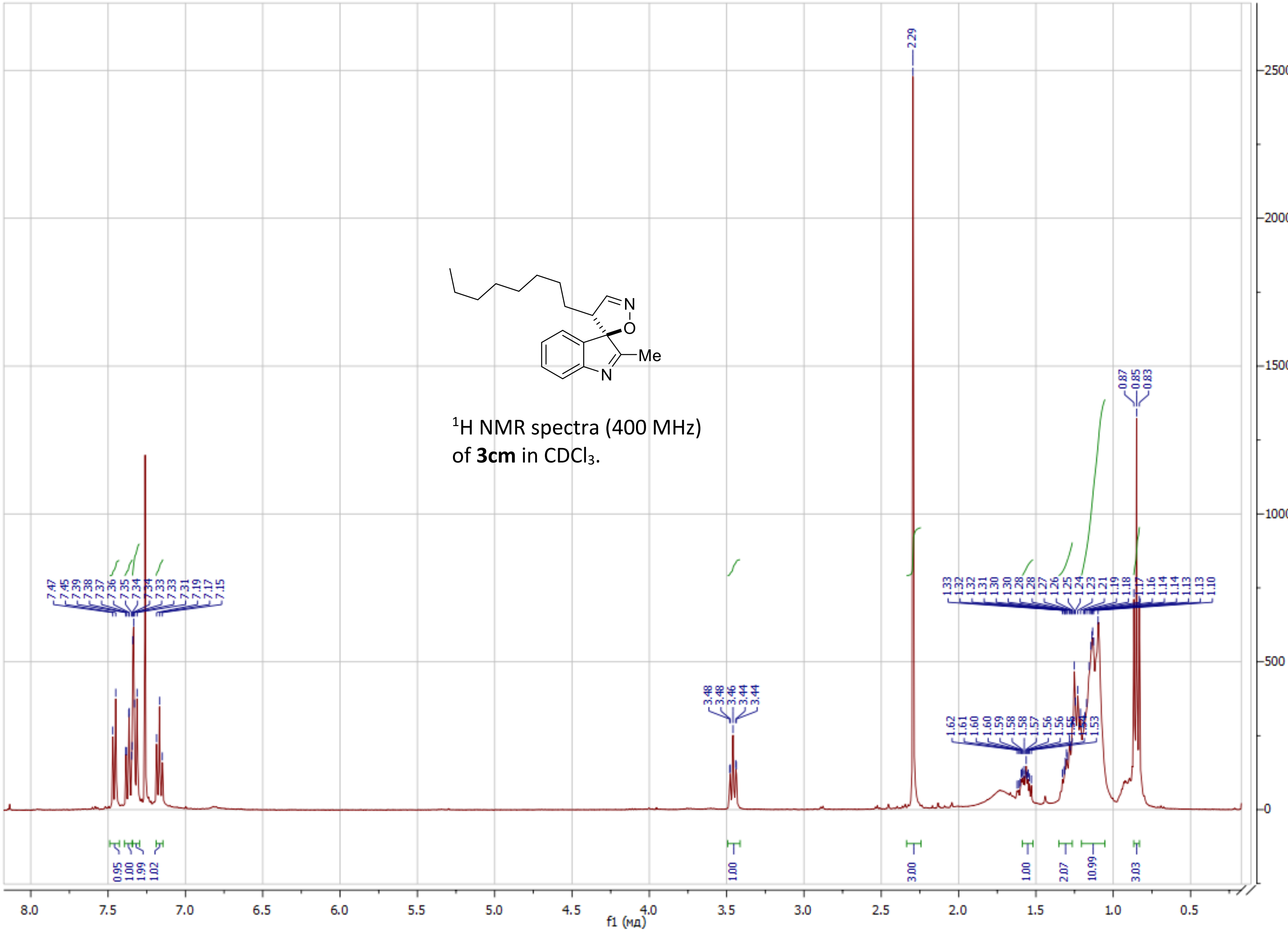


^{13}C NMR spectra (101 MHz) of **3ch** in $CDCl_3$.





^{13}C NMR spectra (101 MHz) of **3cj** in $CDCl_3$.



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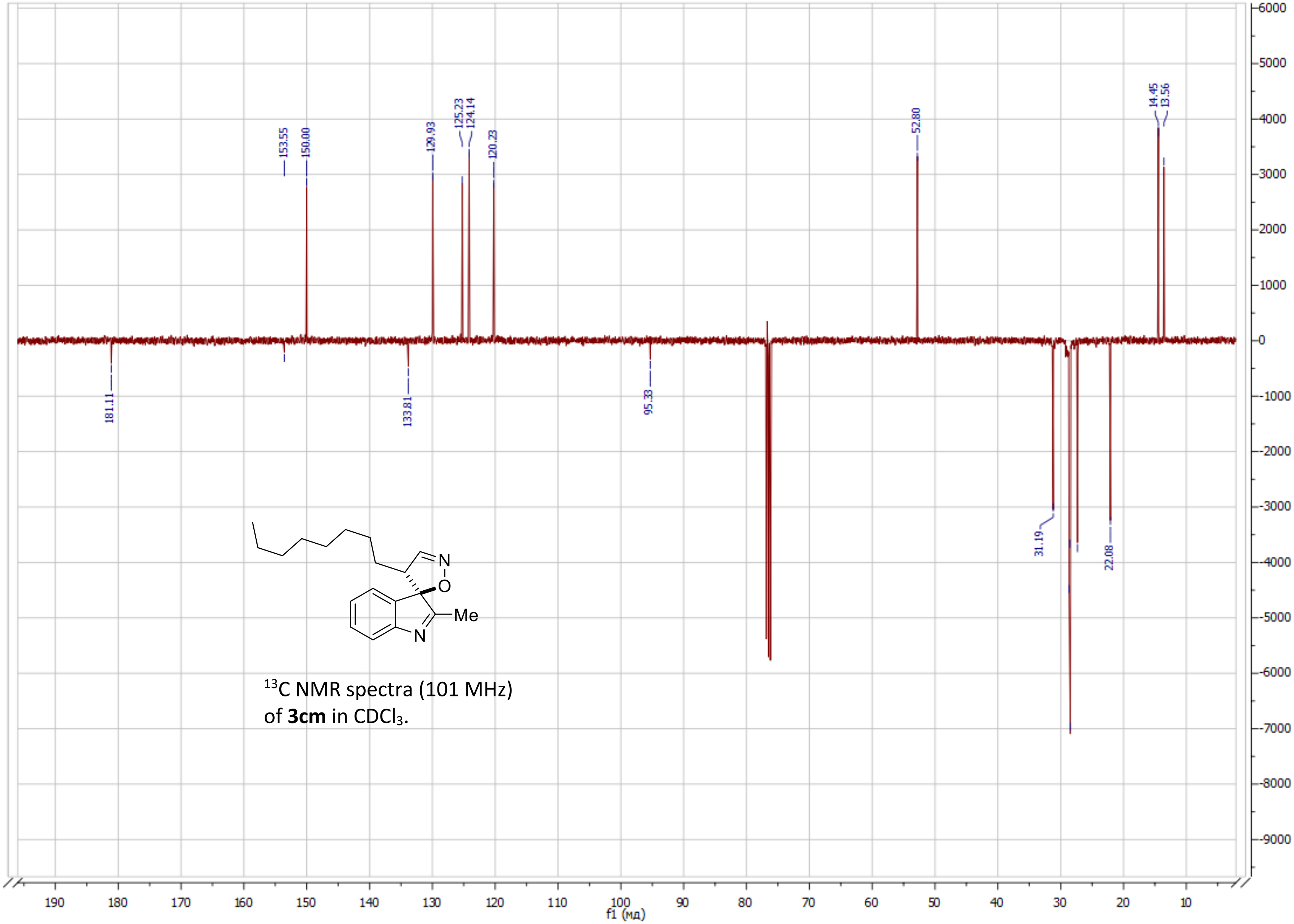
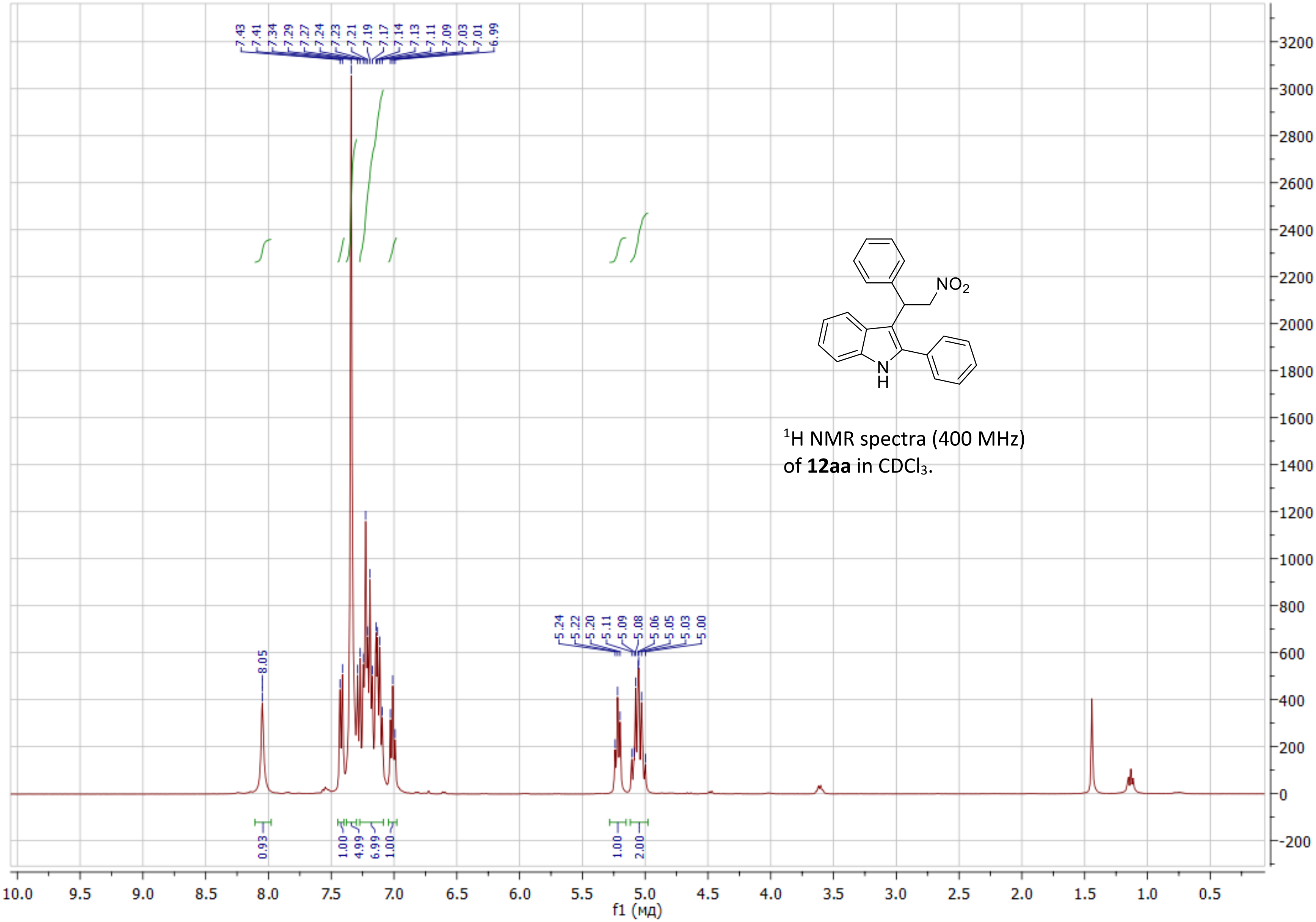
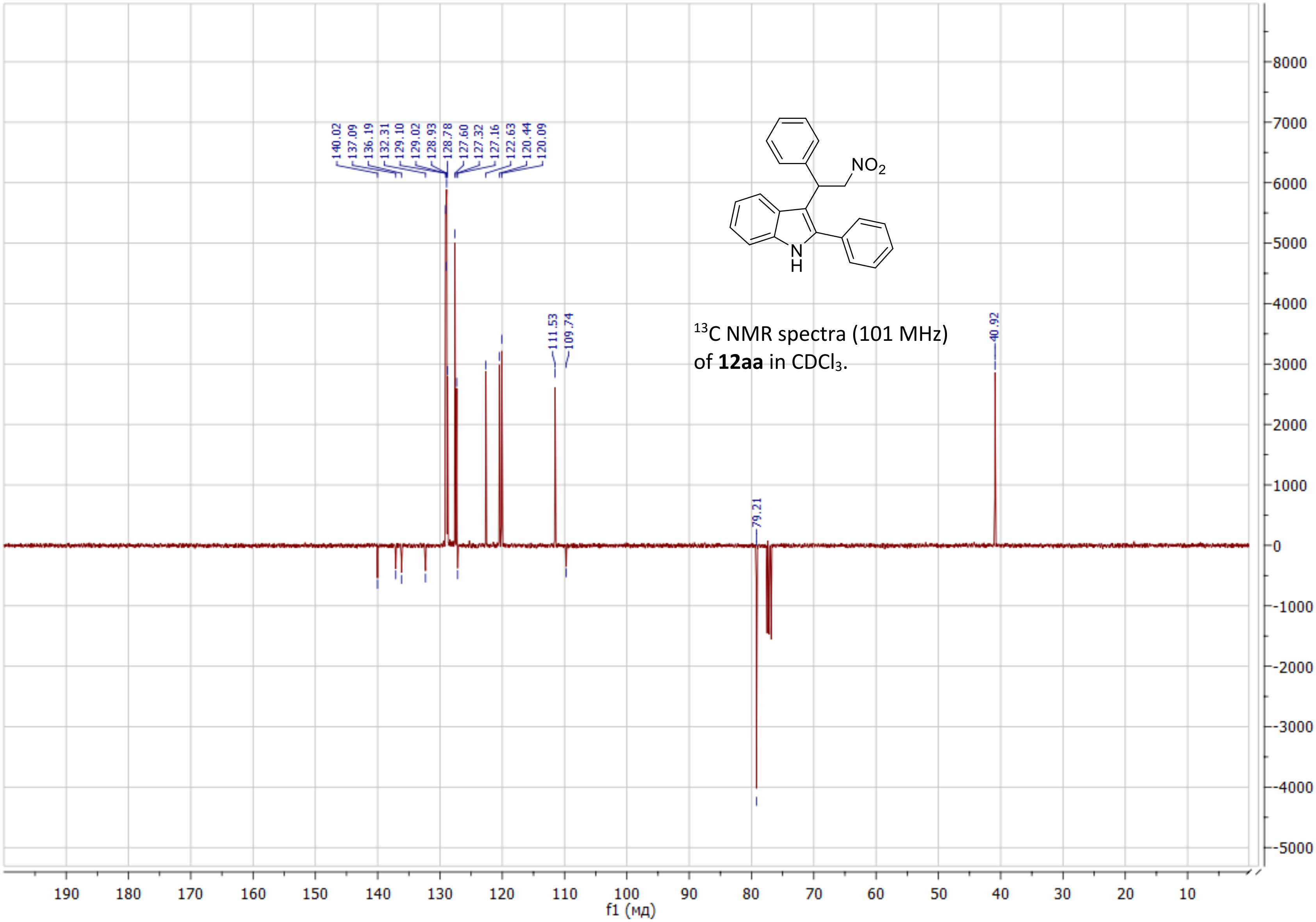


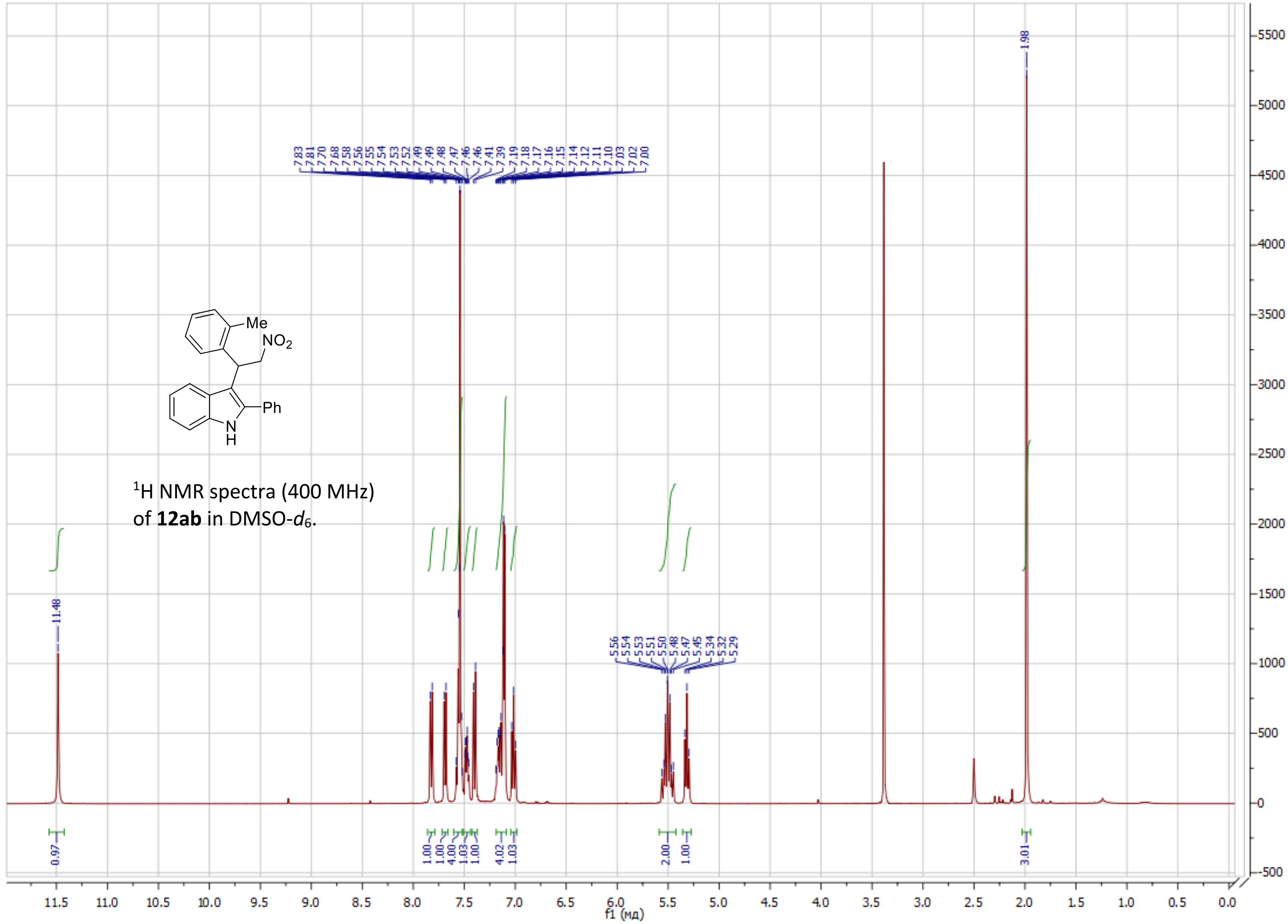
^{13}C NMR spectra (101 MHz) of **3cm** in $CDCl_3$.



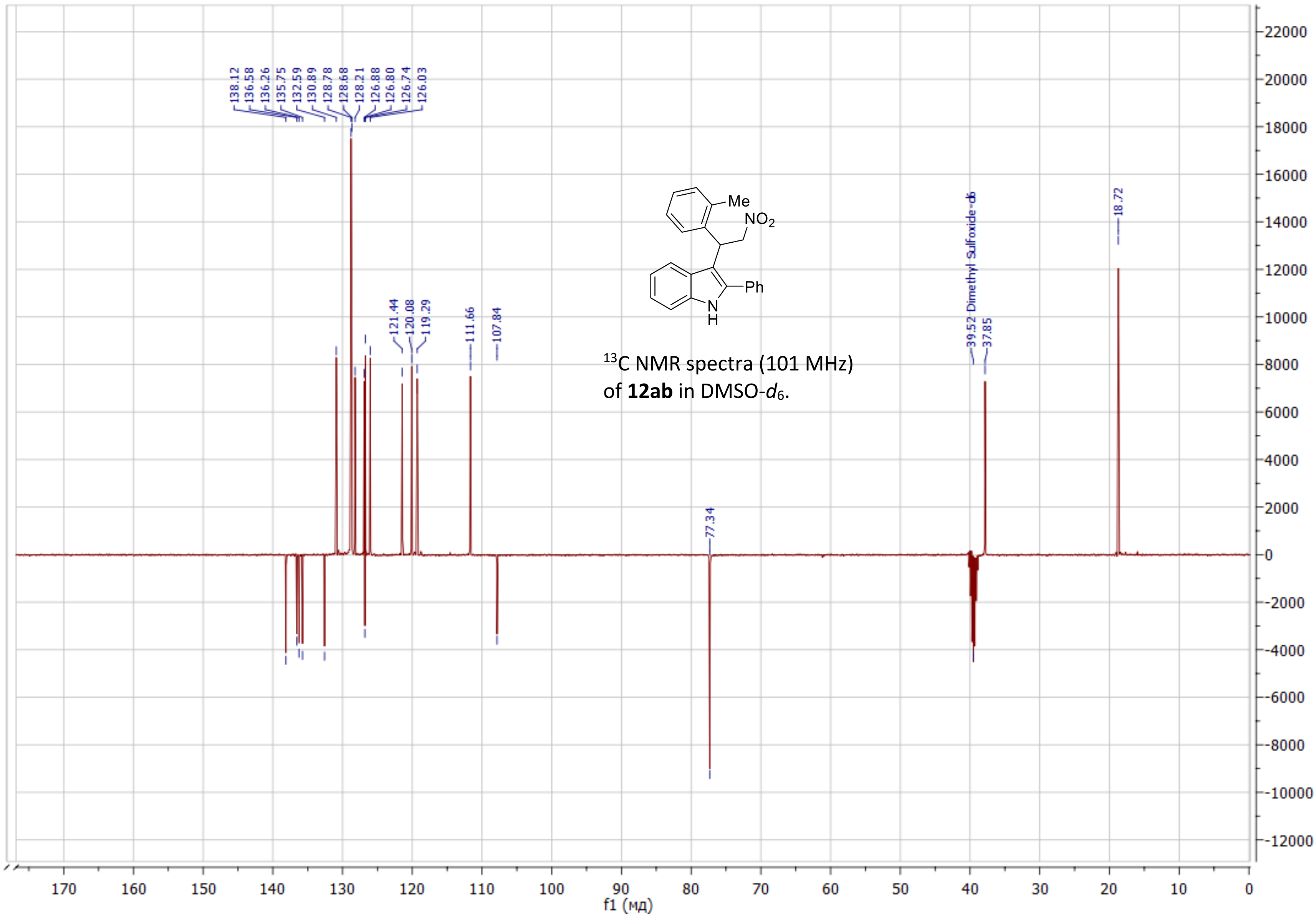
[1]H NMR spectra (400 MHz) of **12aa** in $CDCl_3$.



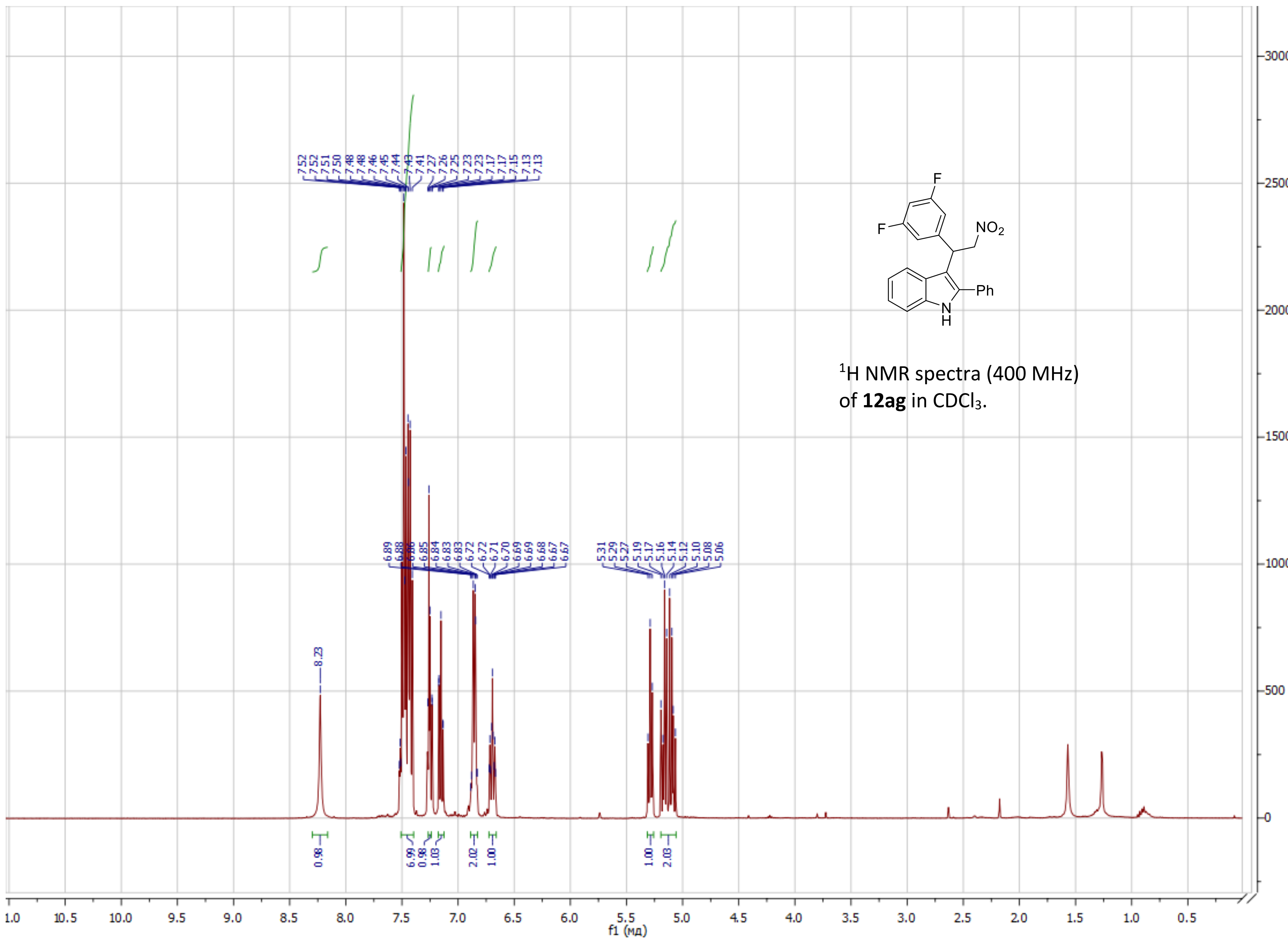
^{13}C NMR spectra (101 MHz) of **12aa** in $CDCl_3$.



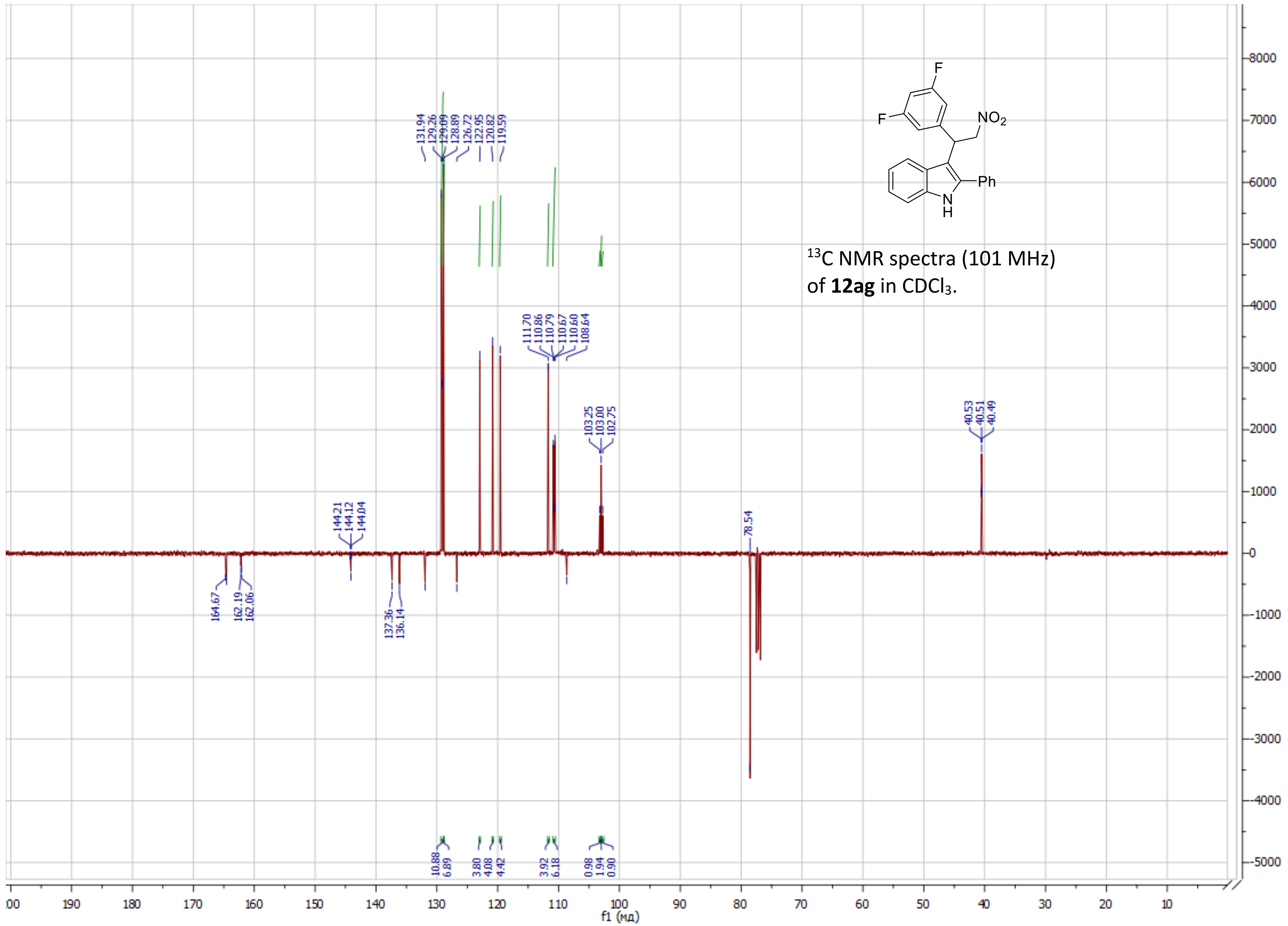
^{1}H NMR spectra (400 MHz) of **12ab** in DMSO-d_6.



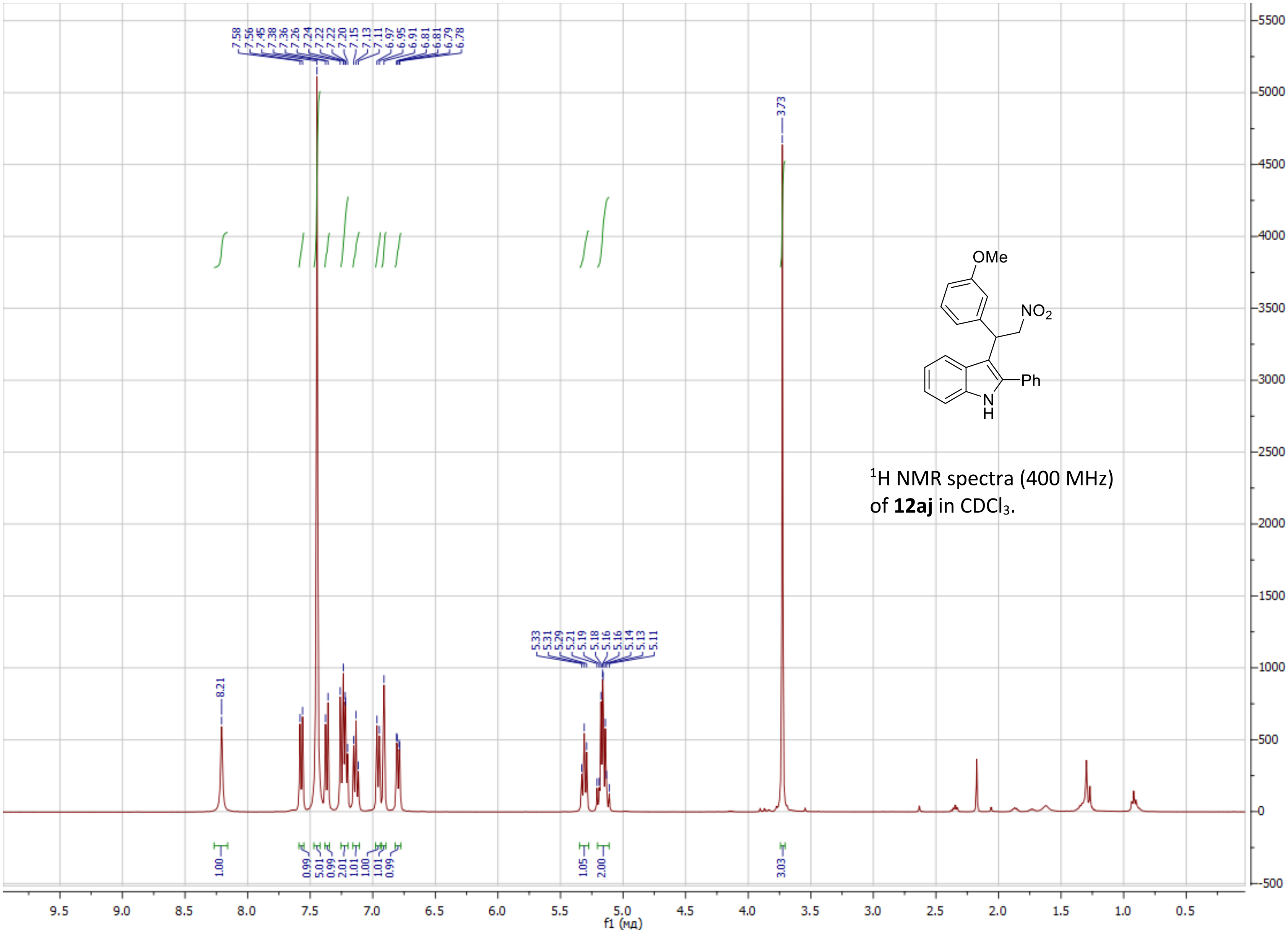
^{13}C NMR spectra (101 MHz) of **12ab** in DMSO-d_6.



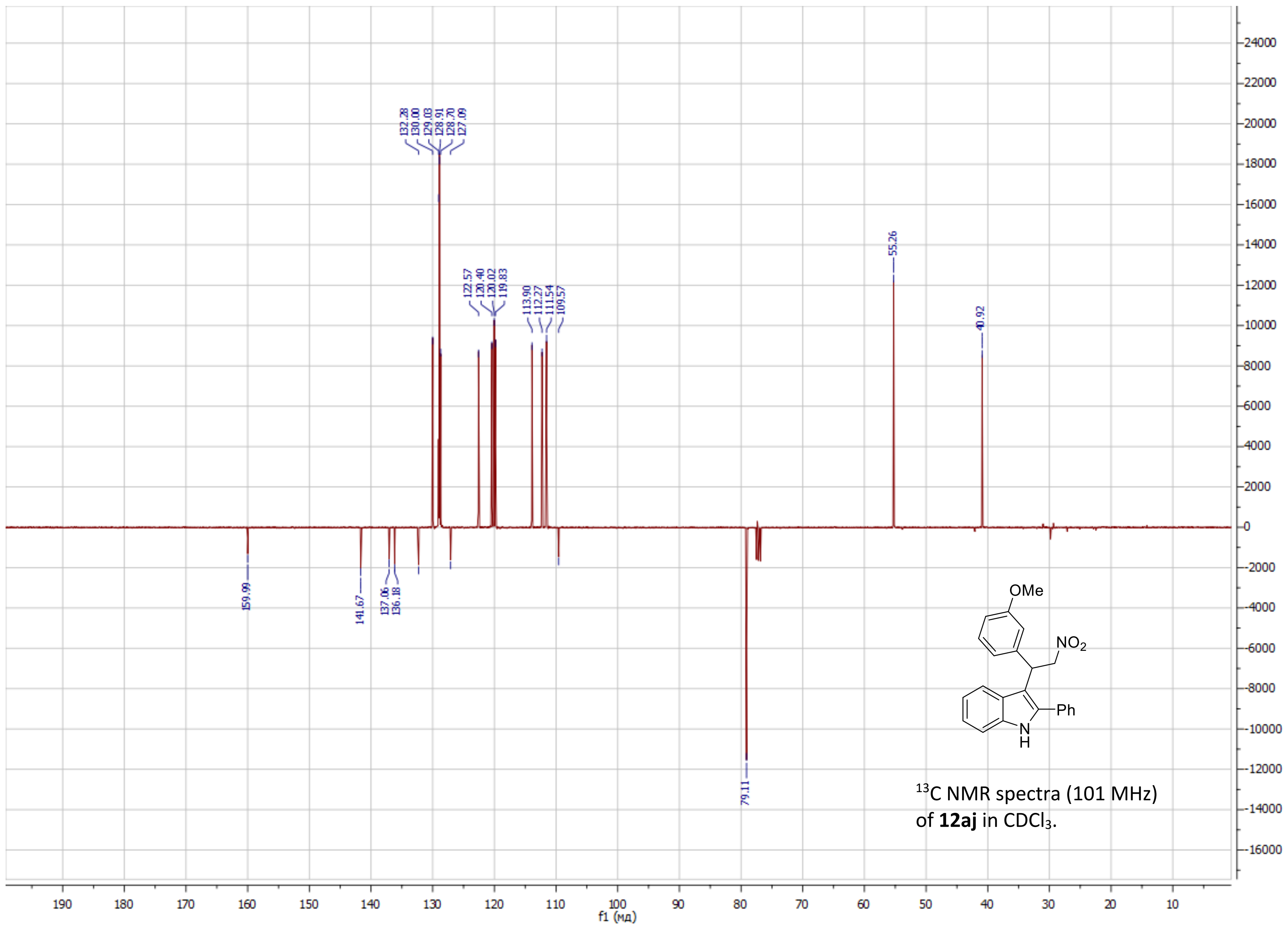
[1]H NMR spectra (400 MHz) of **12ag** in $CDCl_3$.



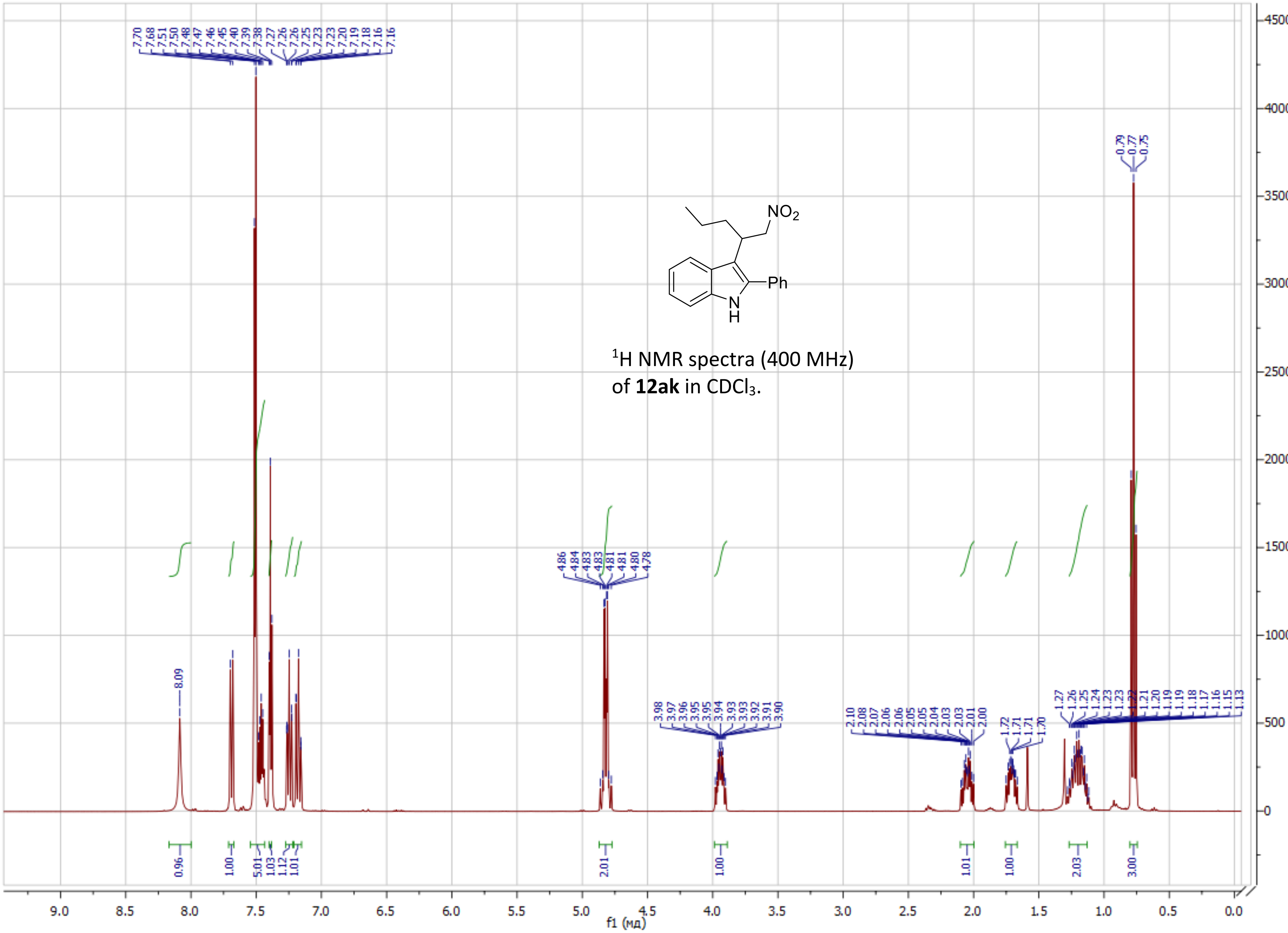
^{13}C NMR spectra (101 MHz) of **12ag** in $CDCl_3$.



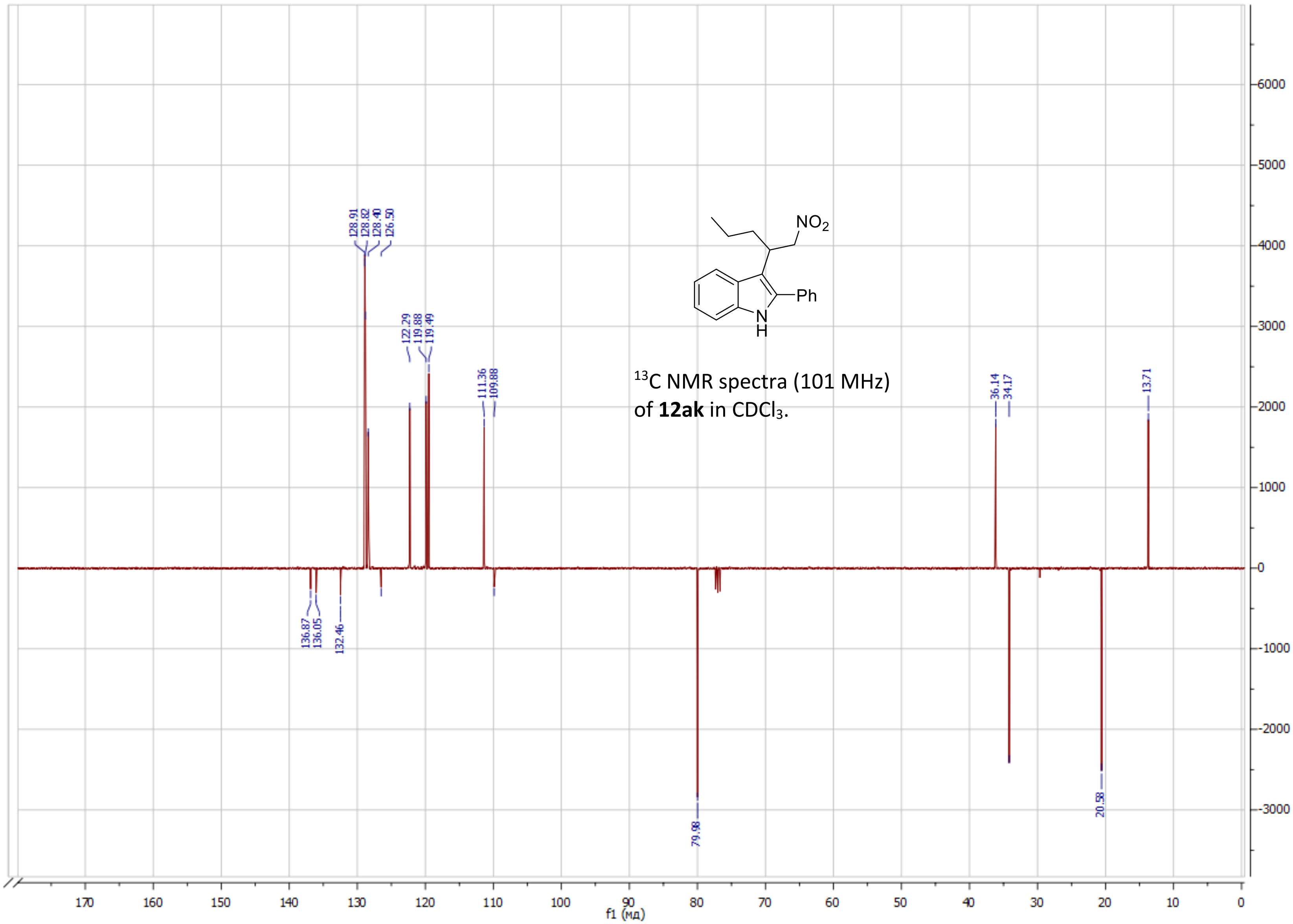
^{1}H NMR spectra (400 MHz) of **12aj** in $CDCl_3$.

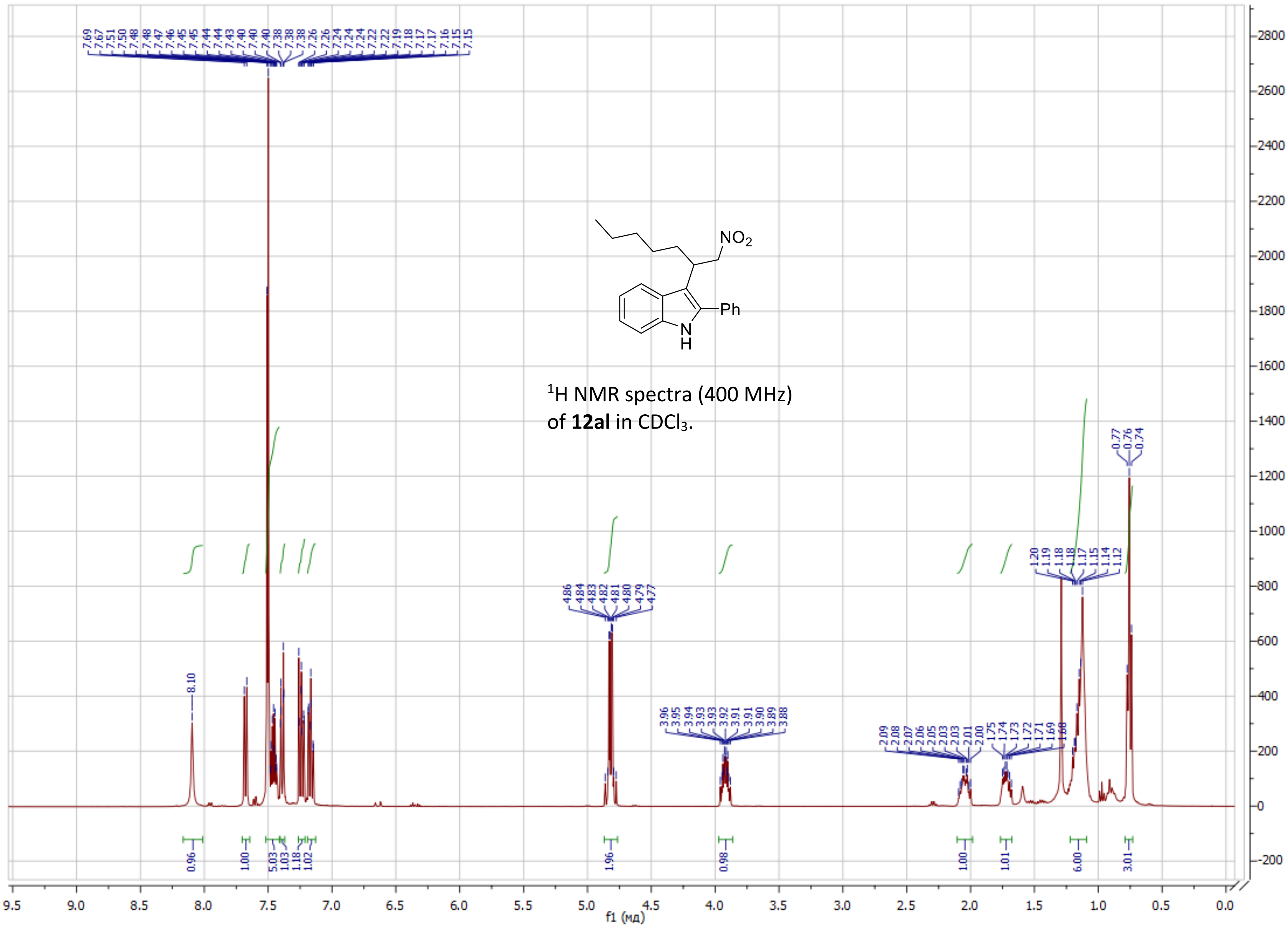


^{13}C NMR spectra (101 MHz) of **12aj** in $CDCl_3$.

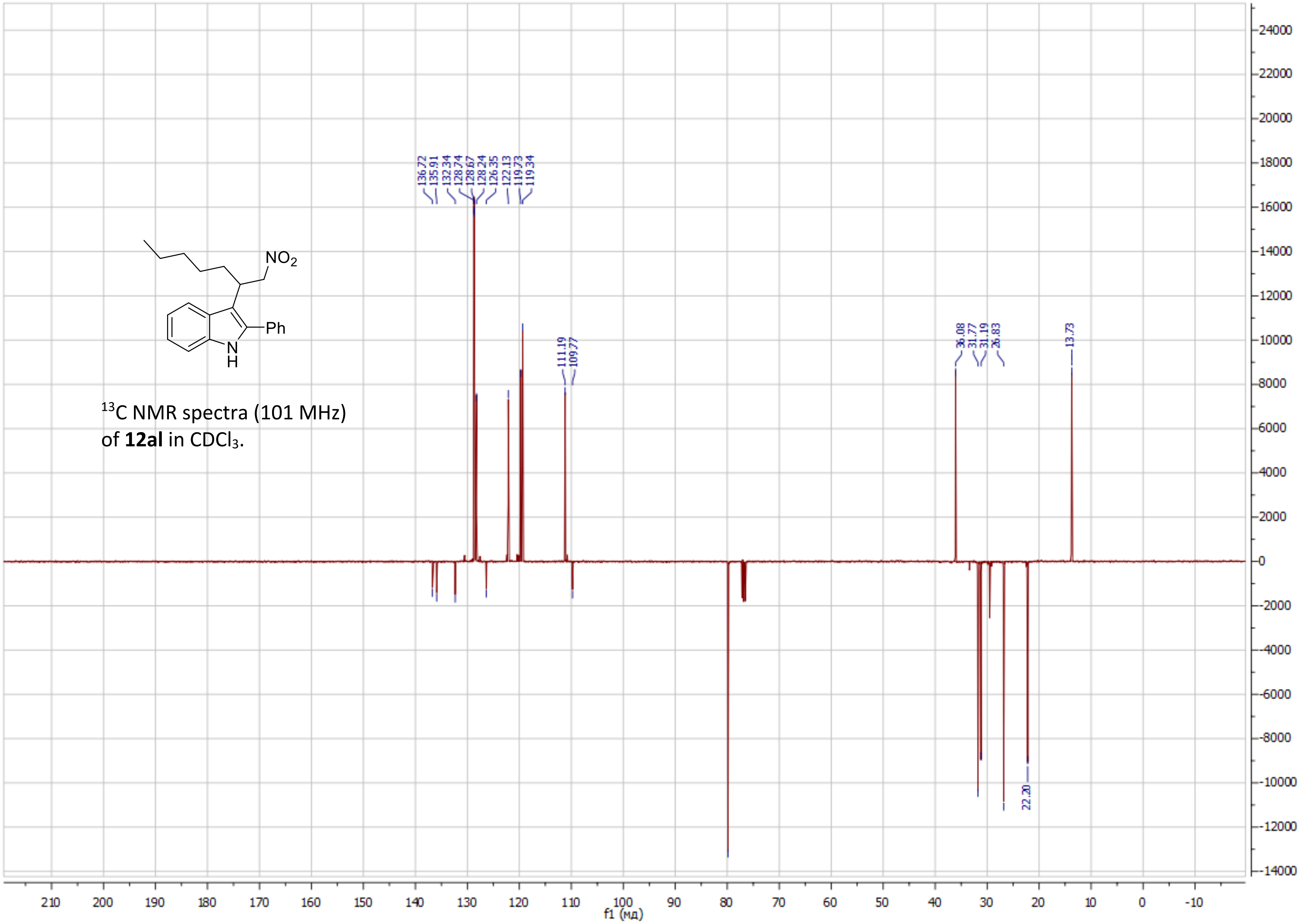


[1]H NMR spectra (400 MHz) of **12ak** in $CDCl_3$.


13C NMR spectra (101 MHz) of 12ak in CDCl3.
NO2
Ph
N
H
128.91
128.82
128.40
126.50
122.29
119.88
119.49
111.36
109.88
36.14
34.17
13.71
136.87
136.05
132.46
79.98
20.58
f1 (мд)

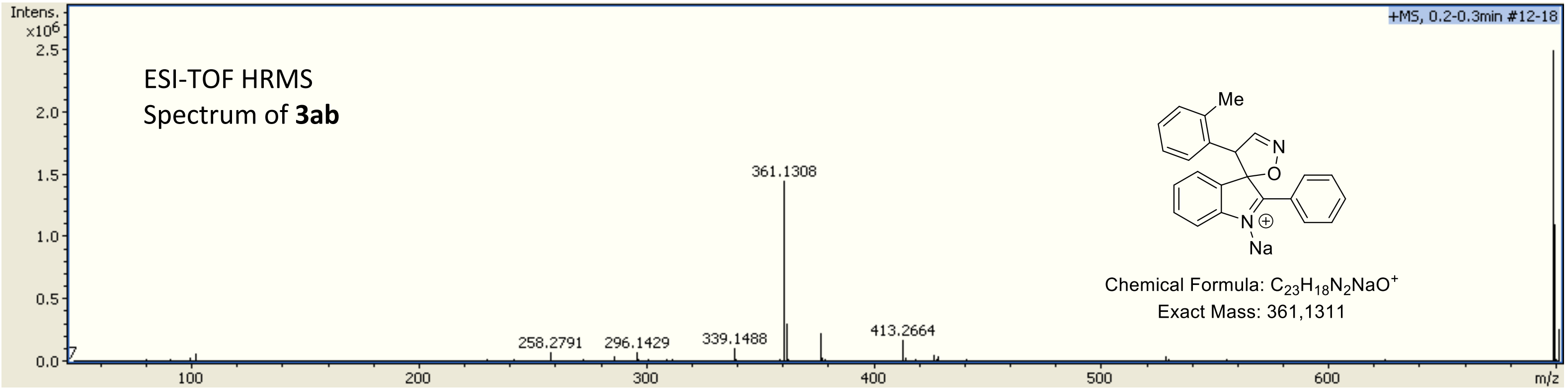


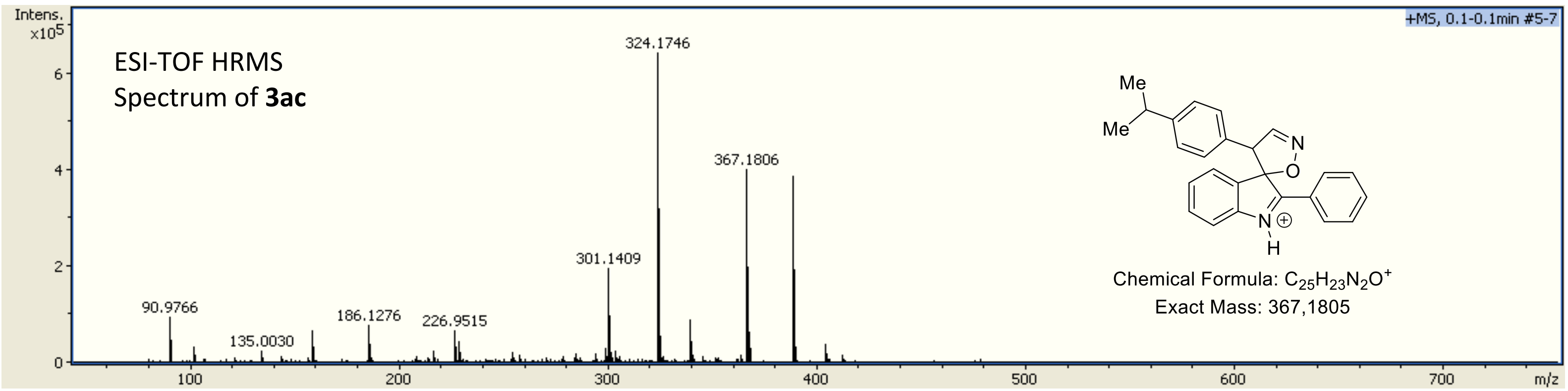
[1]H NMR spectra (400 MHz) of **12al** in $CDCl_3$.

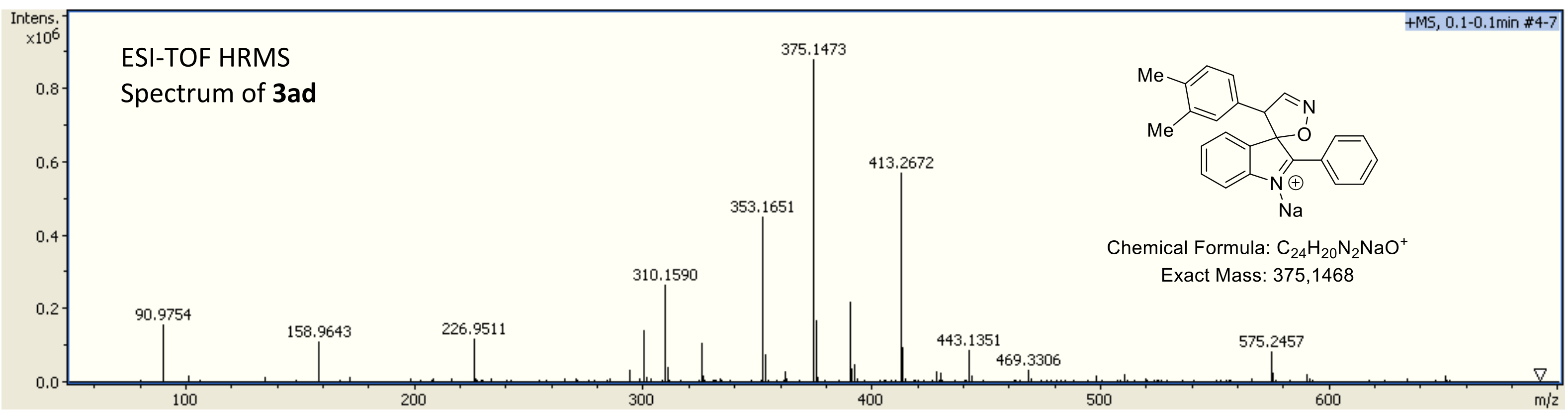


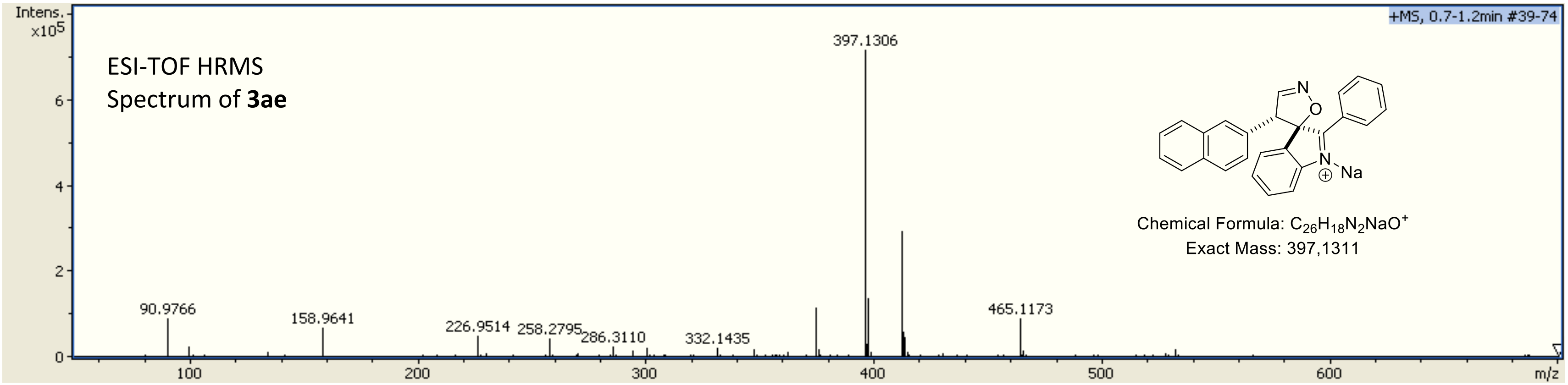
^{13}C NMR spectra (101 MHz) of **12al** in $CDCl_3$.

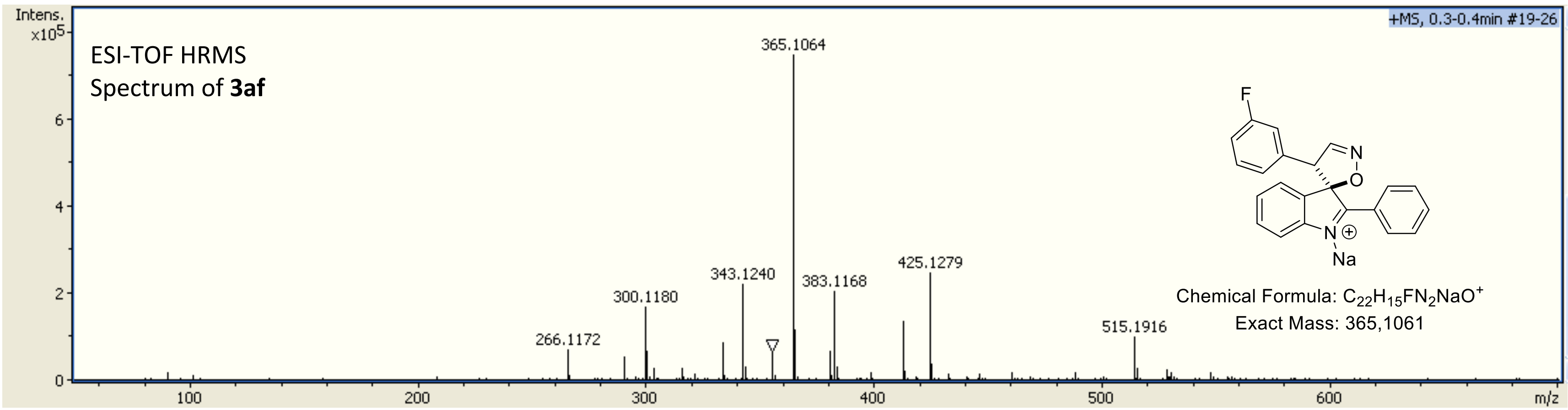
HRMS Spectral Charts

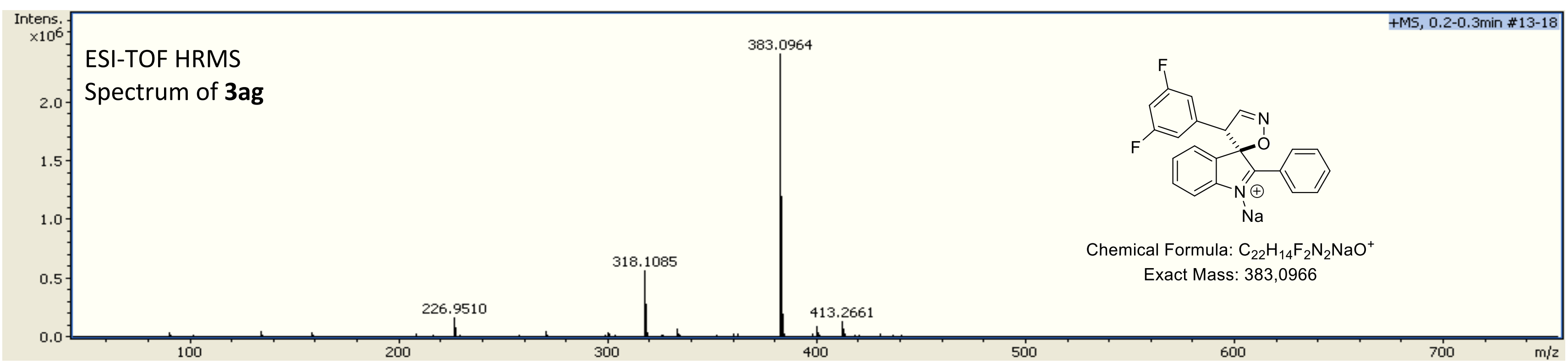


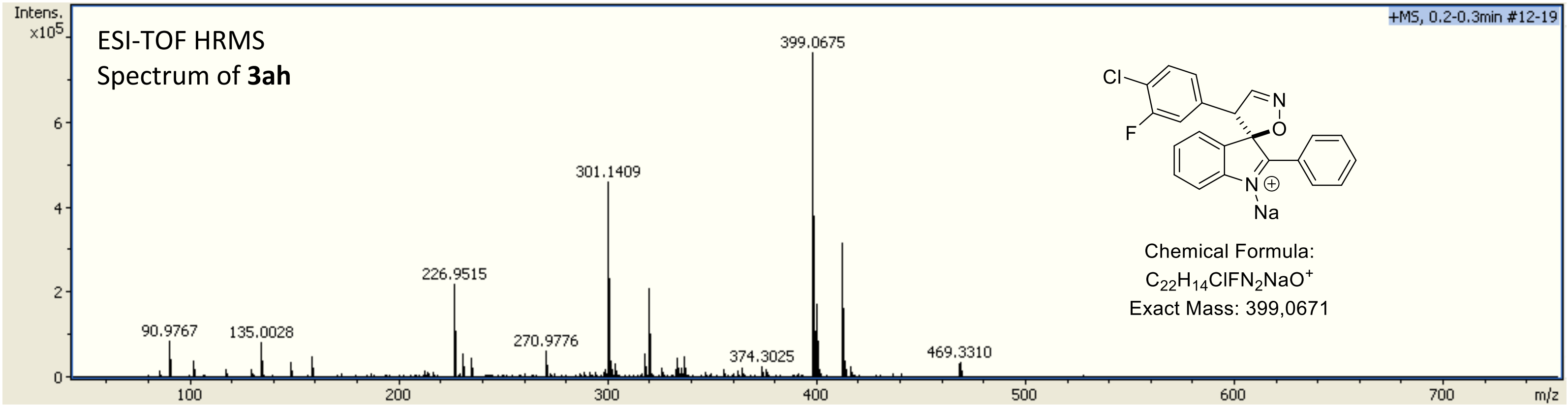


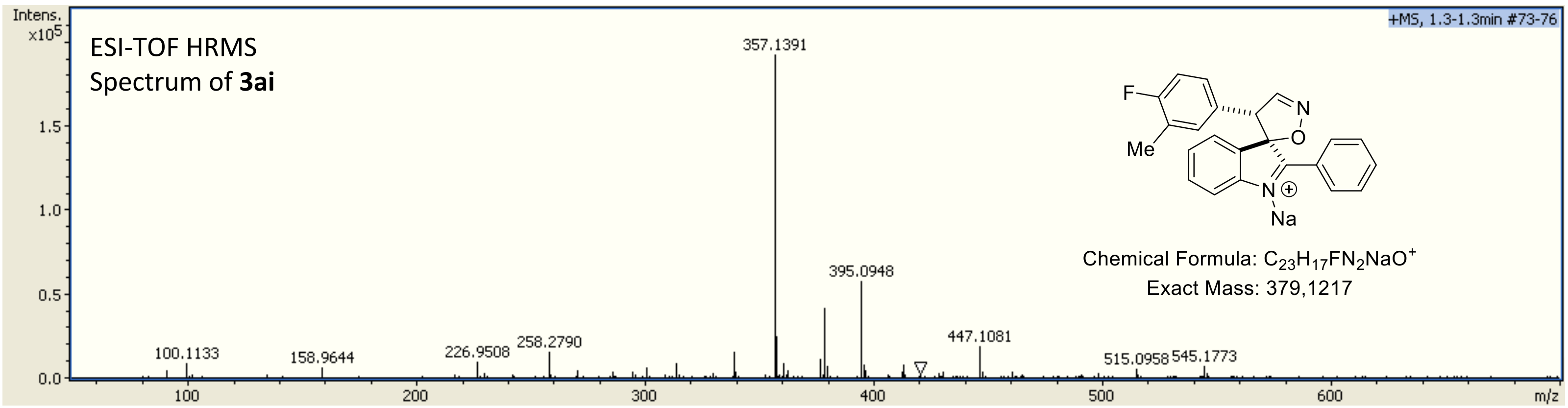


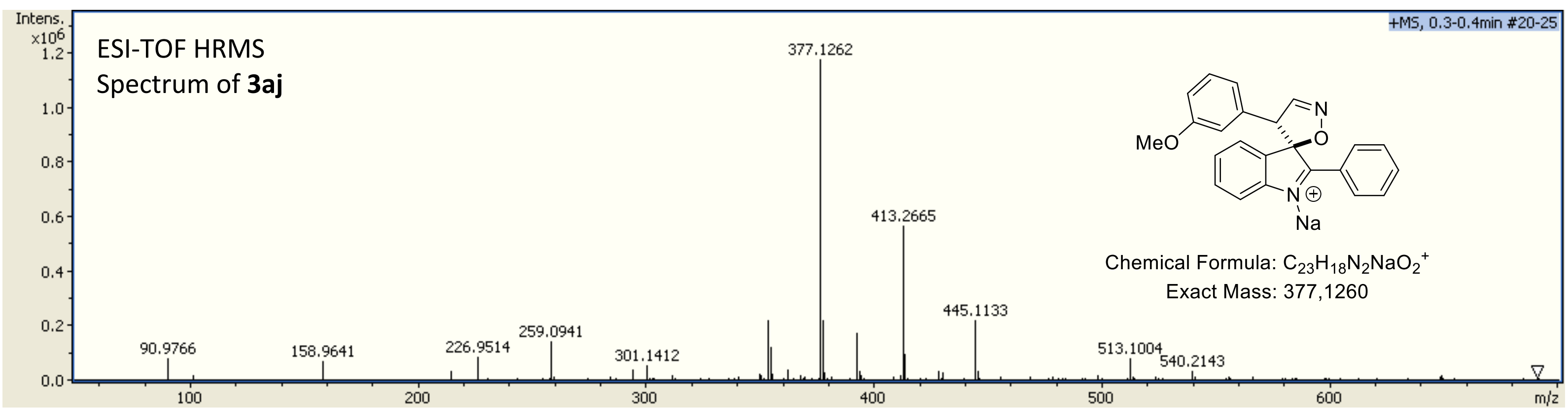

Intens. x10^5
+MS, 0.7-1.2min #39-74
ESI-TOF HRMS
Spectrum of 3ae
397.1306
90.9766
158.9641
226.9514
258.2795
286.3110
332.1435
465.1173
Na
Chemical Formula: $C_{26}H_{18}N_2NaO^+$
Exact Mass: 397,1311
m/z

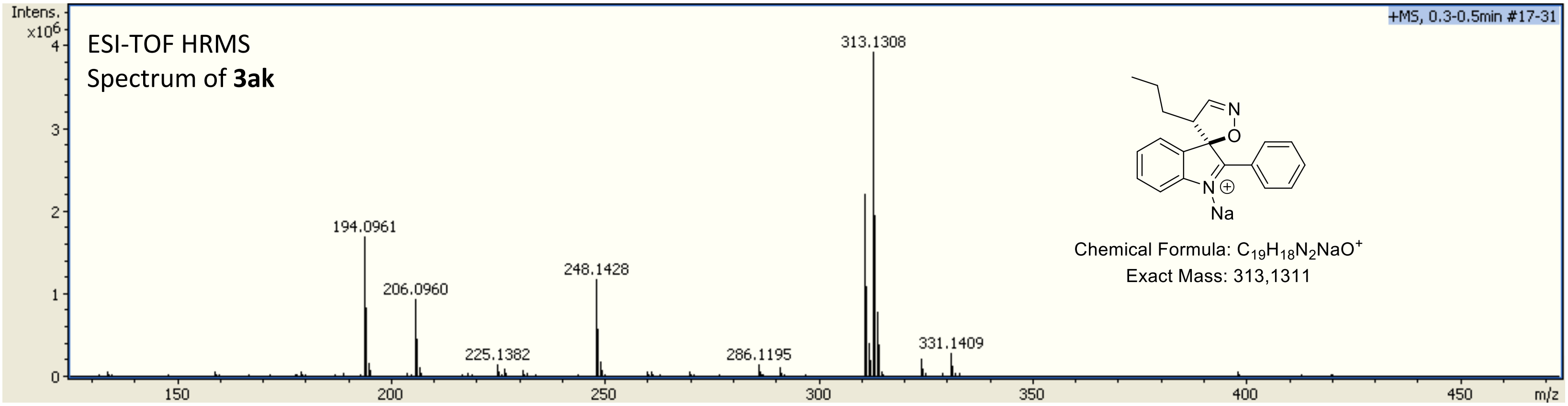

Intens. x10^5
+MS, 0.3-0.4min #19-26
ESI-TOF HRMS
Spectrum of 3af
365.1064
266.1172
300.1180
343.1240
383.1168
425.1279
515.1916
F
Na
Chemical Formula: $C_{22}H_{15}FN_2NaO^+$
Exact Mass: 365,1061
m/z

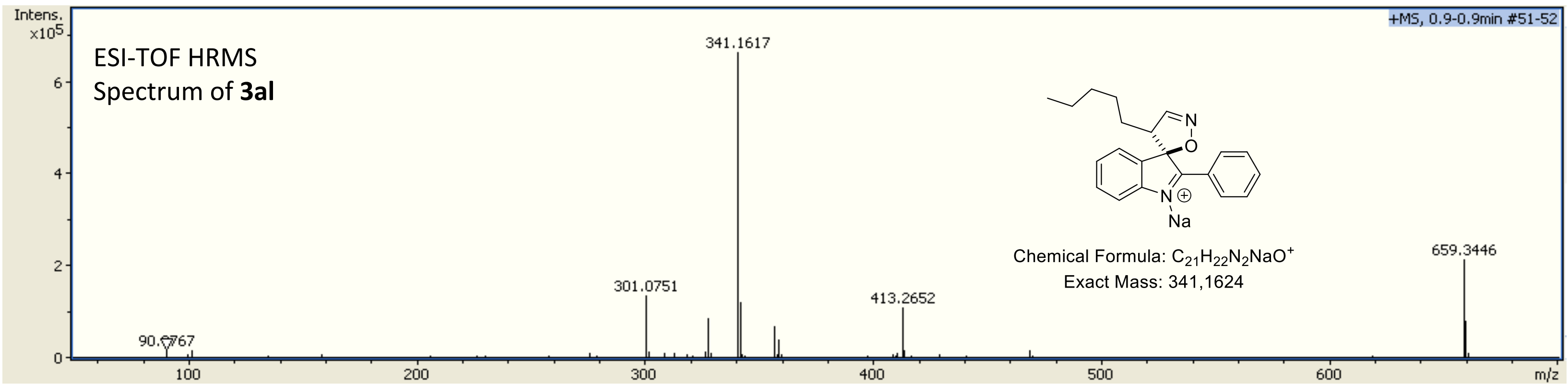

Intens. x10^6
+MS, 0.2-0.3min #13-18
ESI-TOF HRMS
Spectrum of 3ag
383.0964
226.9510
318.1085
413.2661
F
F
Na
Chemical Formula: $C_{22}H_{14}F_2N_2NaO^+$
Exact Mass: 383,0966
m/z

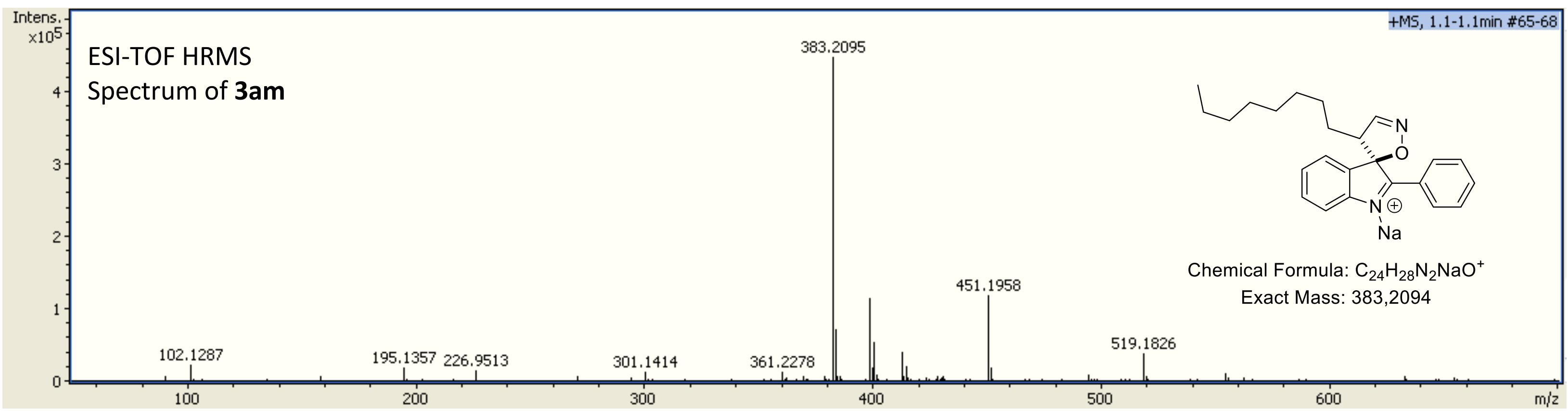

Intens.
x10^5
+MS, 0.2-0.3min #12-19
ESI-TOF HRMS
Spectrum of 3ah
399.0675
301.1409
226.9515
90.9767
135.0028
270.9776
374.3025
469.3310
Cl
F
N
O
N
Na
Chemical Formula:
C22H14ClFN2NaO+
Exact Mass: 399,0671
m/z

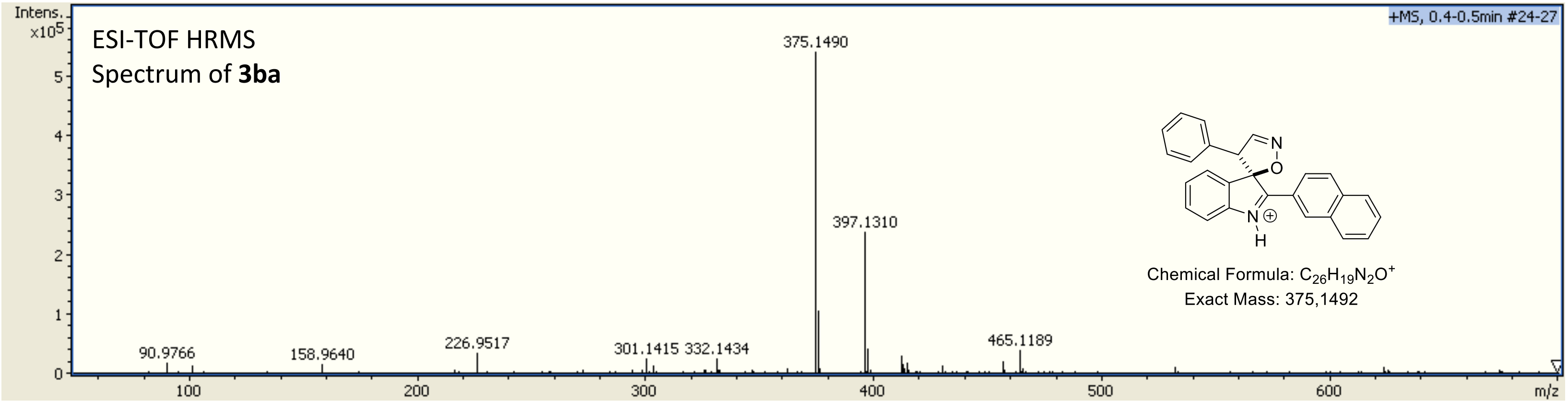

Intens.
x10^5
+MS, 1.3-1.3min #73-76
ESI-TOF HRMS
Spectrum of 3ai
357.1391
395.0948
447.1081
100.1133
158.9644
226.9508
258.2790
515.0958
545.1773
F
Me
N
O
N
Na
Chemical Formula: C23H17FN2NaO+
Exact Mass: 379,1217
m/z


Intens.
x10^6
+MS, 0.3-0.4min #20-25
ESI-TOF HRMS
Spectrum of 3aj
377.1262
413.2665
445.1133
90.9766
158.9641
226.9514
259.0941
301.1412
513.1004
540.2143
MeO
N
O
N
Na
Chemical Formula: C23H18N2NaO2+
Exact Mass: 377,1260
m/z

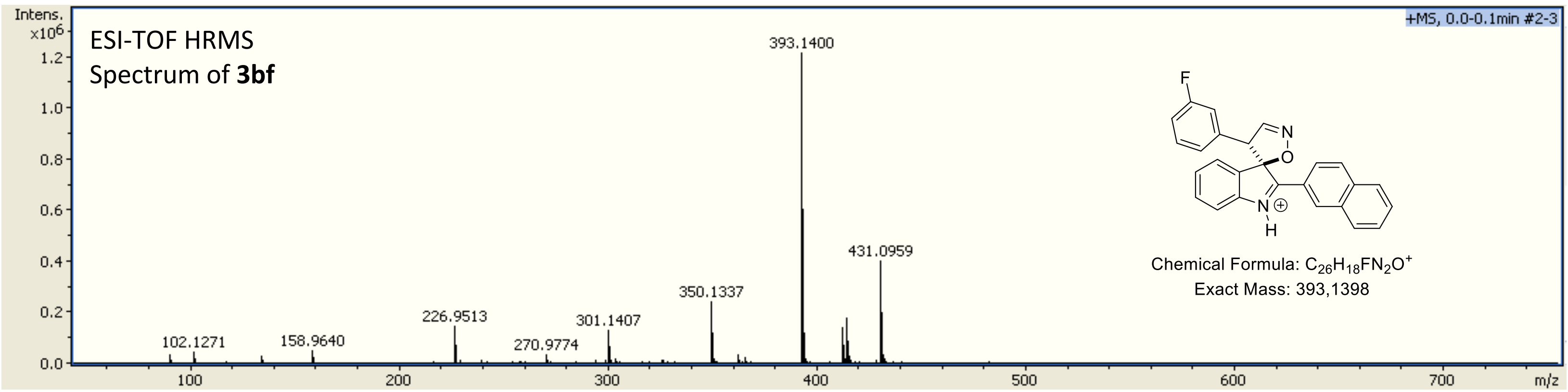

ESI-TOF HRMS
Spectrum of **3ak**
+MS, 0.3-0.5min #17-31
Intens.
x10^6
313.1308
194.0961
206.0960
248.1428
225.1382
286.1195
331.1409
Chemical Formula: $C_{19}H_{18}N_2NaO^+$
Exact Mass: 313,1311
m/z


ESI-TOF HRMS
Spectrum of **3al**
+MS, 0.9-0.9min #51-52
Intens.
x10^5
341.1617
301.0751
413.2652
659.3446
Chemical Formula: $C_{21}H_{22}N_2NaO^+$
Exact Mass: 341,1624
m/z

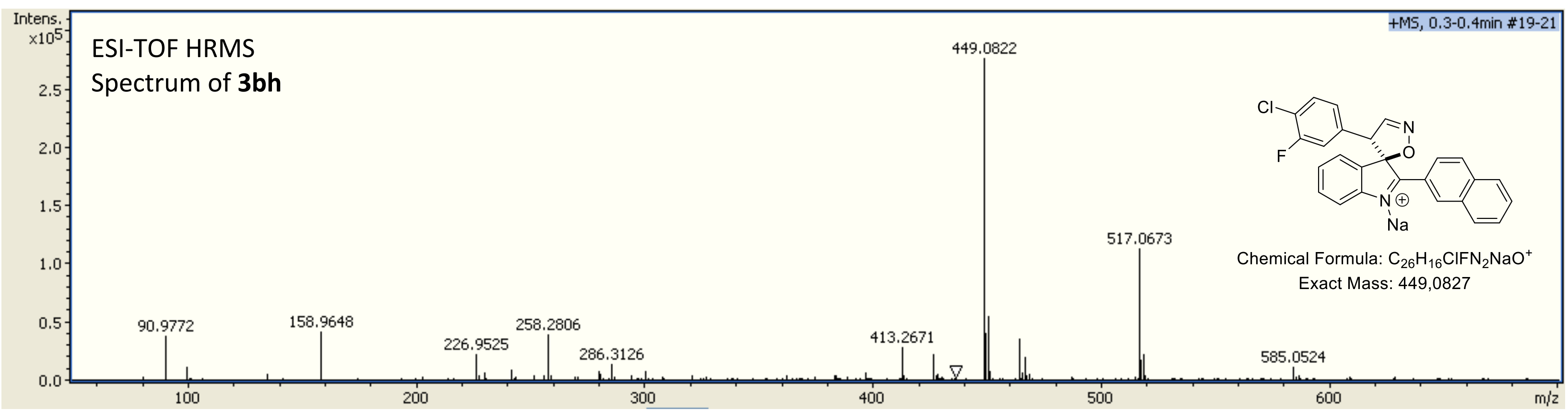

ESI-TOF HRMS
Spectrum of **3am**
+MS, 1.1-1.1min #65-68
Intens.
x10^5
383.2095
451.1958
519.1826
102.1287
195.1357
226.9513
301.1414
361.2278
Chemical Formula: $C_{24}H_{28}N_2NaO^+$
Exact Mass: 383,2094
m/z



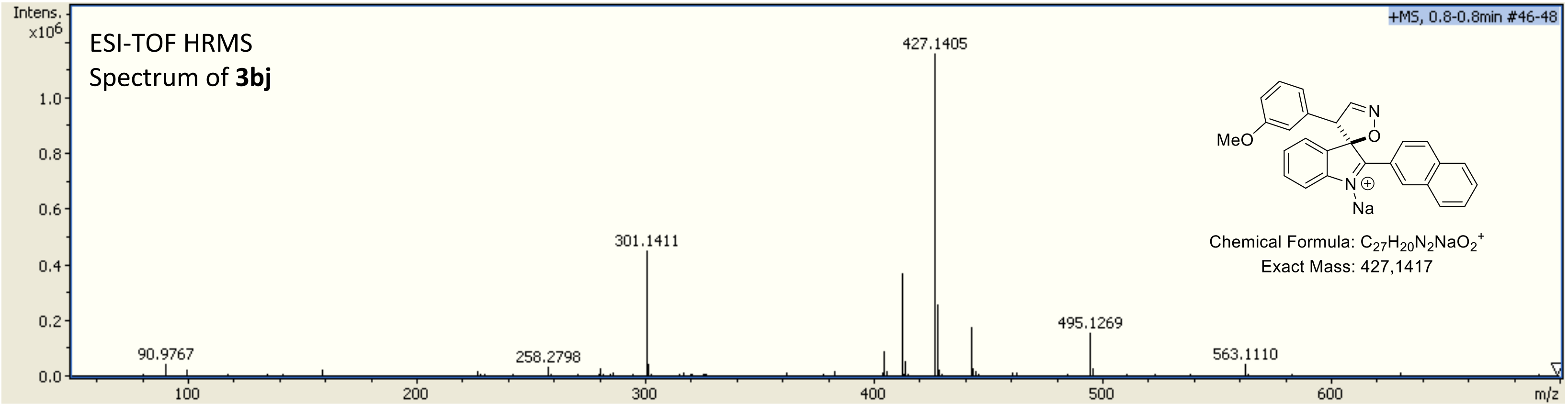
Intens. x10^5
ESI-TOF HRMS
Spectrum of **3ba**
+MS, 0.4-0.5min #24-27
375.1490
397.1310
90.9766
158.9640
226.9517
301.1415 332.1434
465.1189
Chemical Formula: $C_{26}H_{19}N_2O^+$
Exact Mass: 375,1492
m/z

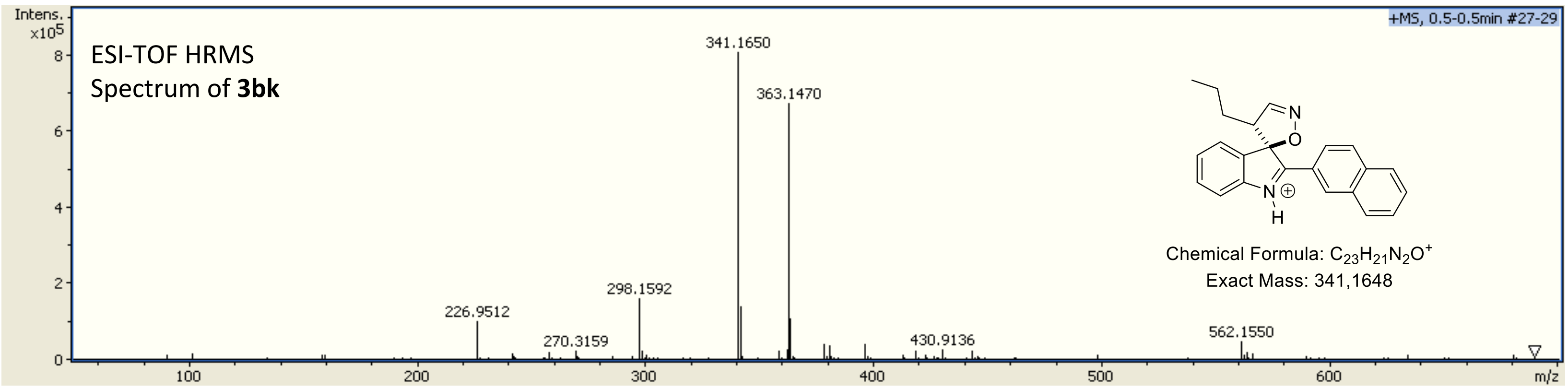


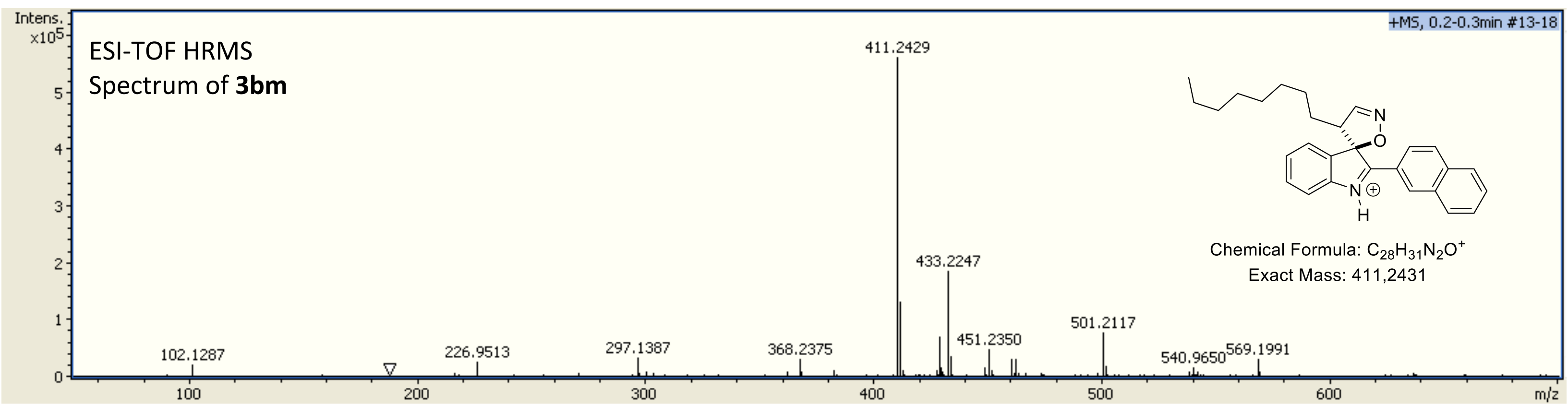
Intens. x10^6
ESI-TOF HRMS
Spectrum of **3bf**
+MS, 0.0-0.1min #2-3
393.1400
431.0959
350.1337
102.1271
158.9640
226.9513
270.9774
301.1407
Chemical Formula: $C_{26}H_{18}FN_2O^+$
Exact Mass: 393,1398
m/z

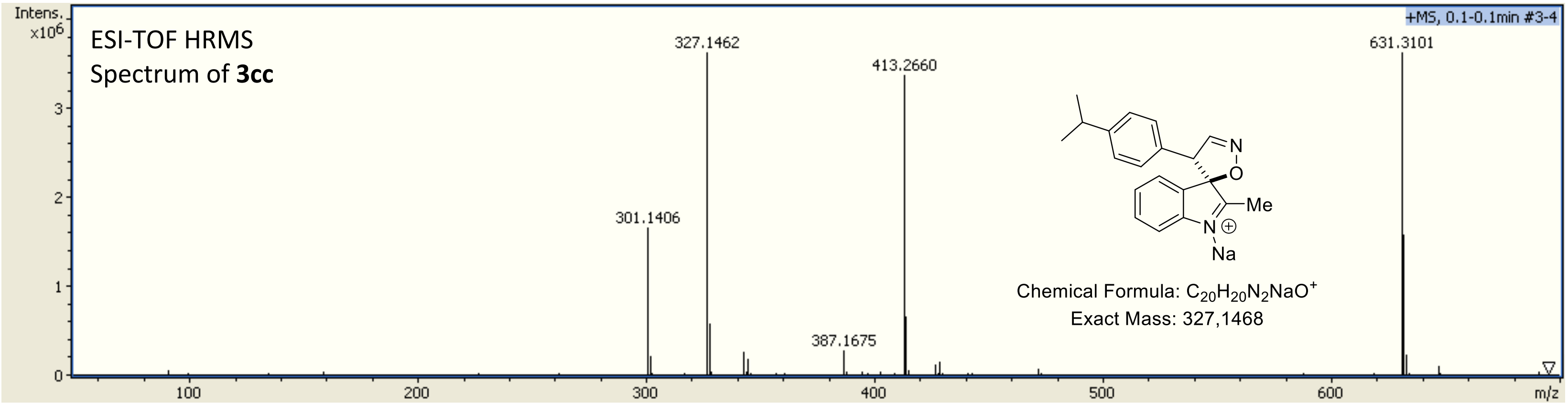


Intens. x10^5
ESI-TOF HRMS
Spectrum of **3bh**
+MS, 0.3-0.4min #19-21
449.0822
517.0673
90.9772
158.9648
226.9525
258.2806
286.3126
413.2671
585.0524
Chemical Formula: $C_{26}H_{16}ClFN_2NaO^+$
Exact Mass: 449,0827
m/z

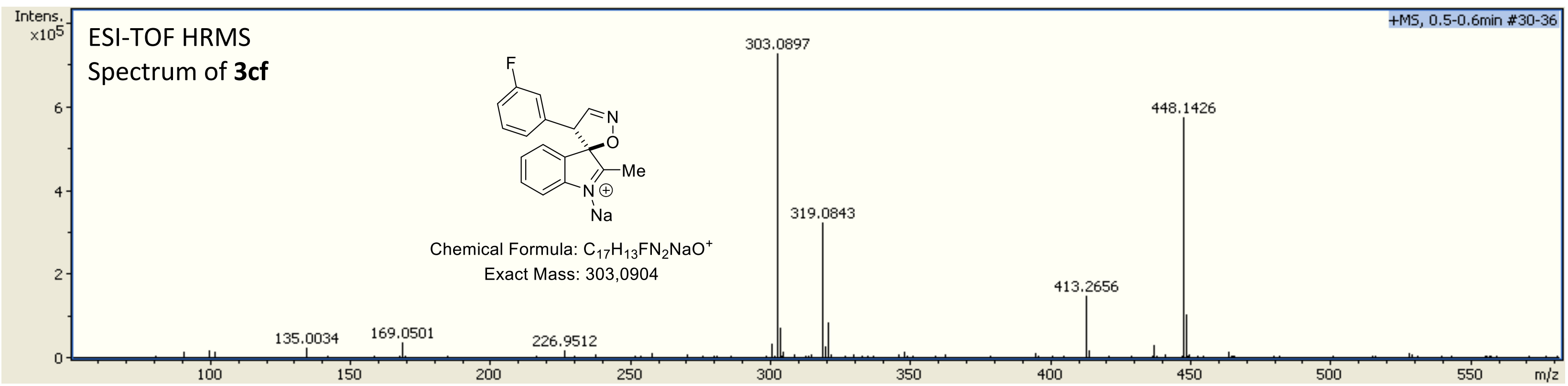

Intens.
x10^6
ESI-TOF HRMS
Spectrum of 3bj
+MS, 0.8-0.8min #46-48
427.1405
301.1411
90.9767
258.2798
495.1269
563.1110
MeO
N
O
N
Na
Chemical Formula: C27H20N2NaO2+
Exact Mass: 427,1417
m/z


Intens.
x10^5
ESI-TOF HRMS
Spectrum of 3bk
+MS, 0.5-0.5min #27-29
341.1650
363.1470
298.1592
226.9512
270.3159
430.9136
562.1550
N
O
N
H
Chemical Formula: C23H21N2O+
Exact Mass: 341,1648
m/z

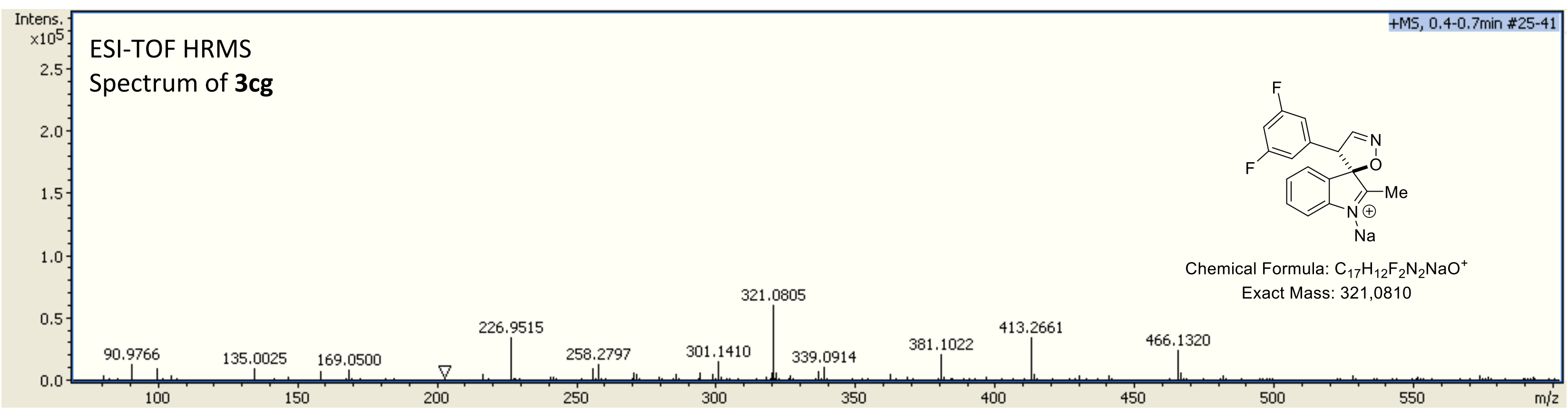

Intens.
x10^5
ESI-TOF HRMS
Spectrum of 3bm
+MS, 0.2-0.3min #13-18
411.2429
433.2247
501.2117
451.2350
102.1287
226.9513
297.1387
368.2375
540.9650
569.1991
N
O
N
H
Chemical Formula: C28H31N2O+
Exact Mass: 411,2431
m/z



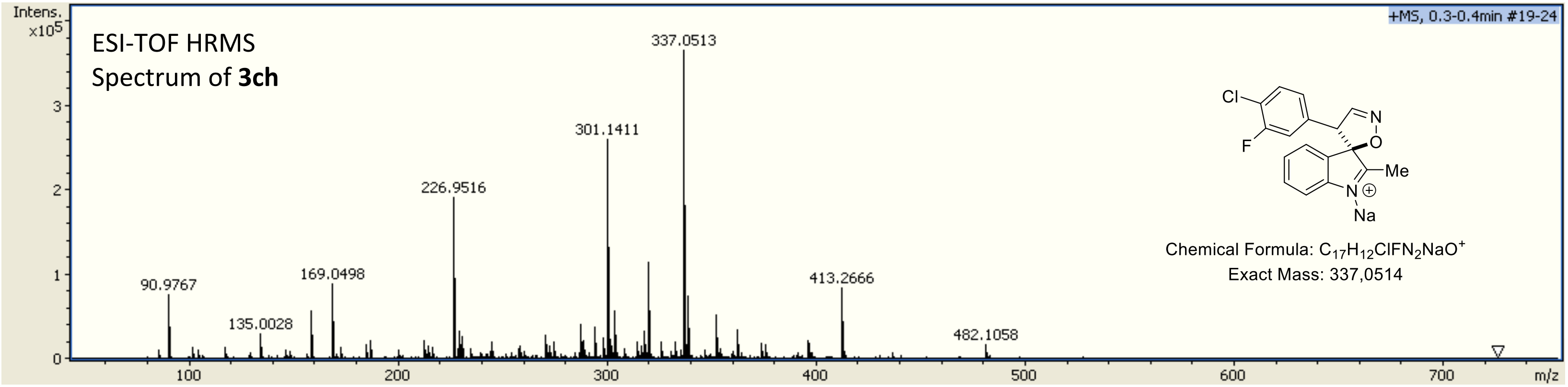
Intens.
x10^6
ESI-TOF HRMS
Spectrum of 3cc
+MS, 0.1-0.1min #3-4
327.1462
413.2660
631.3101
301.1406
387.1675
Me
N
O
N
Na
Chemical Formula: $C_{20}H_{20}N_2NaO^+$
Exact Mass: 327,1468
m/z

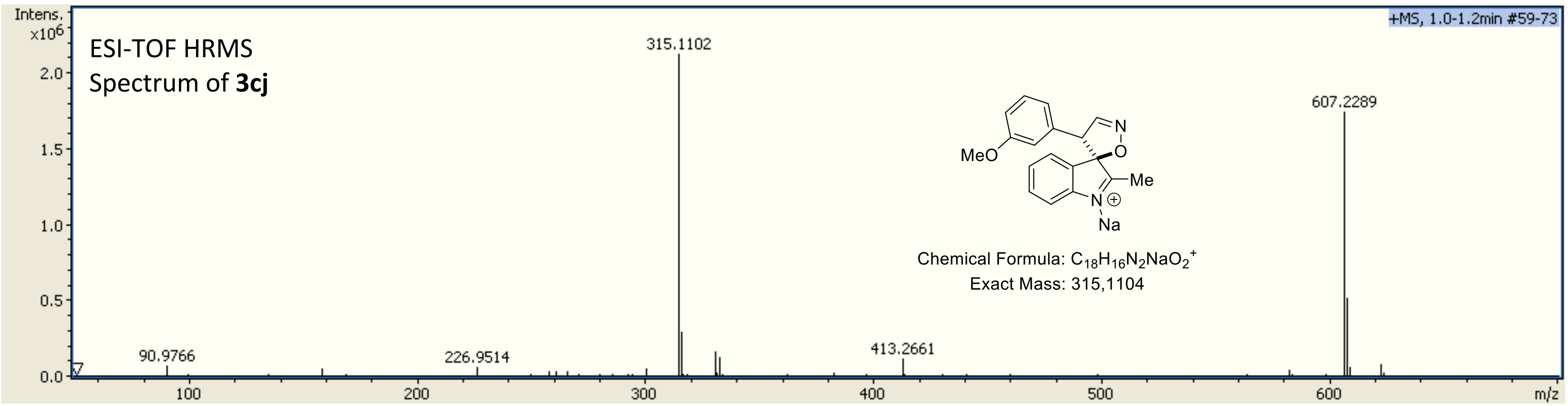


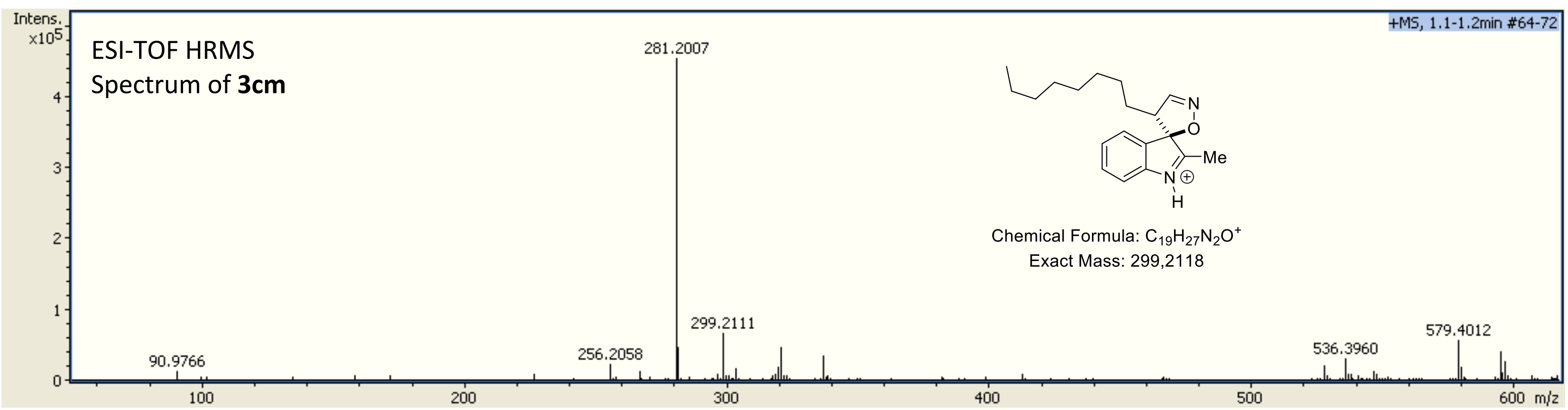
Intens.
x10^5
ESI-TOF HRMS
Spectrum of 3cf
+MS, 0.5-0.6min #30-36
F
N
O
Me
N
Na
303.0897
448.1426
319.0843
413.2656
135.0034
169.0501
226.9512
Chemical Formula: $C_{17}H_{13}FN_2NaO^+$
Exact Mass: 303,0904
m/z

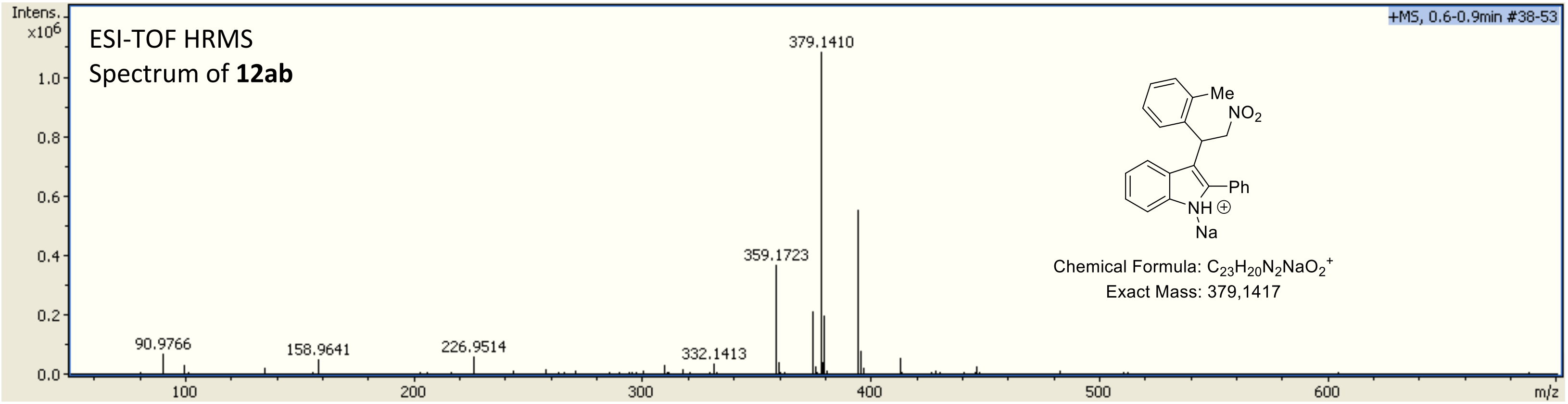


Intens.
x10^5
ESI-TOF HRMS
Spectrum of 3cg
+MS, 0.4-0.7min #25-41
F
F
N
O
Me
N
Na
Chemical Formula: $C_{17}H_{12}F_2N_2NaO^+$
Exact Mass: 321,0810
321.0805
226.9515
413.2661
466.1320
381.1022
90.9766
135.0025
169.0500
258.2797
301.1410
339.0914
m/z

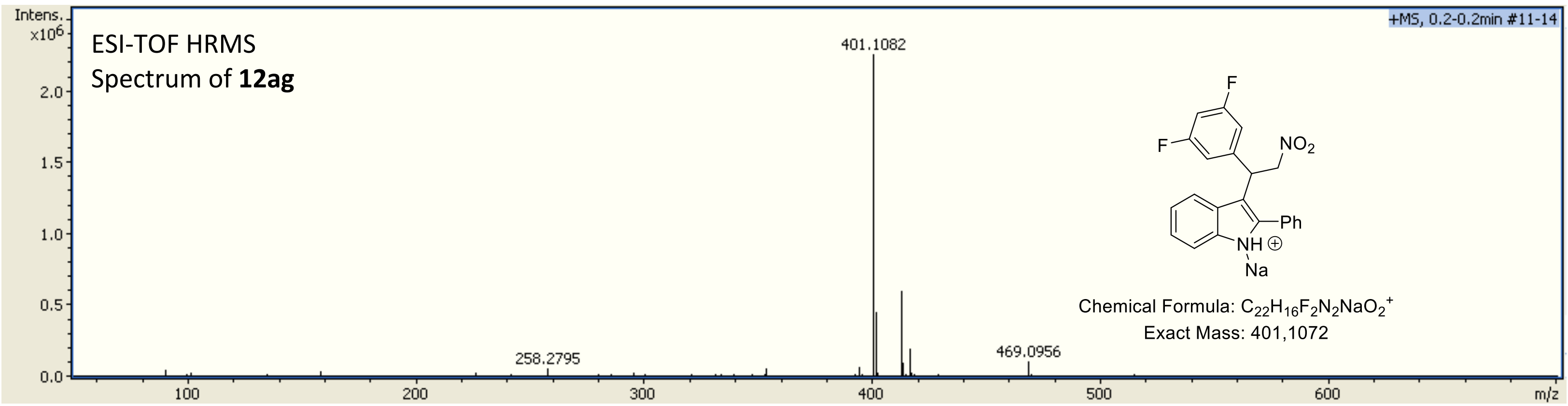

Intens.
x10^5
+MS, 0.3-0.4min #19-24
ESI-TOF HRMS
Spectrum of 3ch
337.0513
301.1411
226.9516
169.0498
90.9767
135.0028
413.2666
482.1058
Chemical Formula: C17H12ClFN2NaO+
Exact Mass: 337,0514
m/z


Intens.
x10^6
+MS, 1.0-1.2min #59-73
ESI-TOF HRMS
Spectrum of 3cj
315.1102
607.2289
90.9766
226.9514
413.2661
Chemical Formula: C18H16N2NaO2+
Exact Mass: 315,1104
m/z

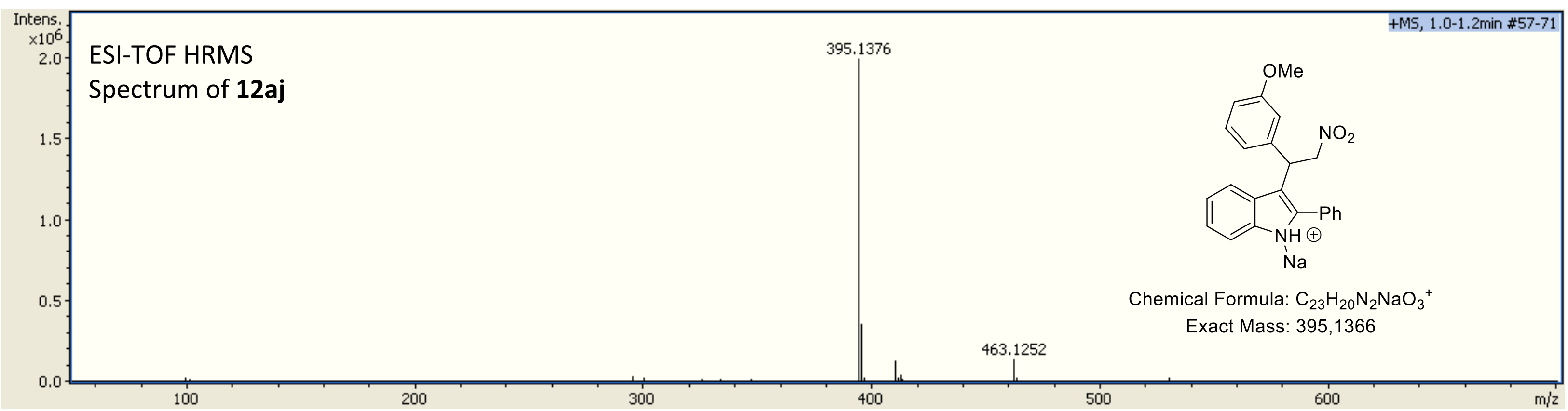

Intens.
x10^5
+MS, 1.1-1.2min #64-72
ESI-TOF HRMS
Spectrum of 3cm
281.2007
299.2111
256.2058
90.9766
536.3960
579.4012
Chemical Formula: C19H27N2O+
Exact Mass: 299,2118
m/z



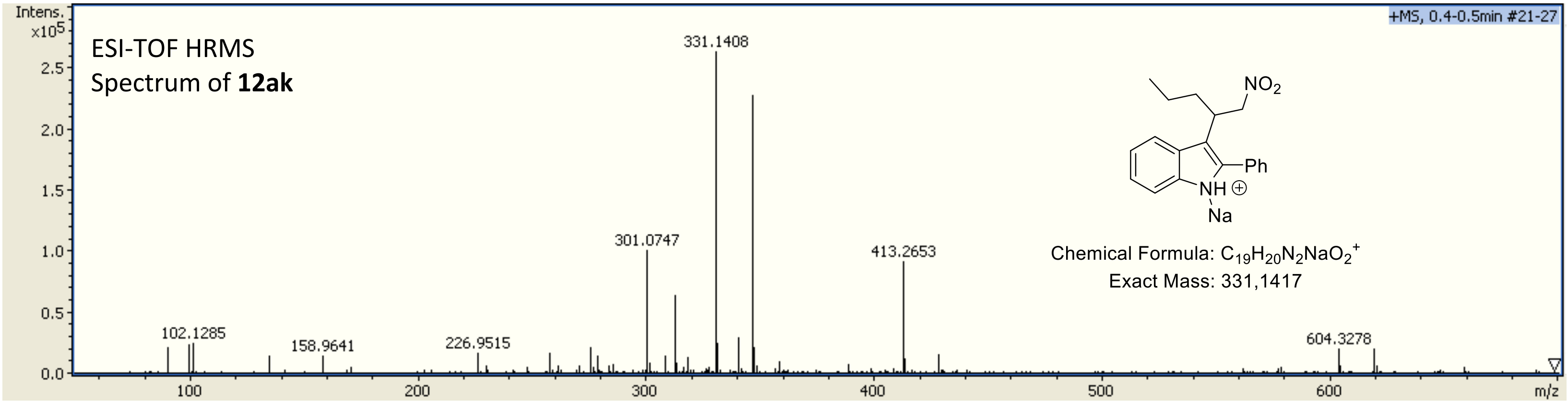
ESI-TOF HRMS
Spectrum of 12ab
+MS, 0.6-0.9min #38-53
379.1410
359.1723
90.9766
158.9641
226.9514
332.1413
Me
NO2
Ph
NH ⊕
Na
Chemical Formula: C23H20N2NaO2+
Exact Mass: 379,1417



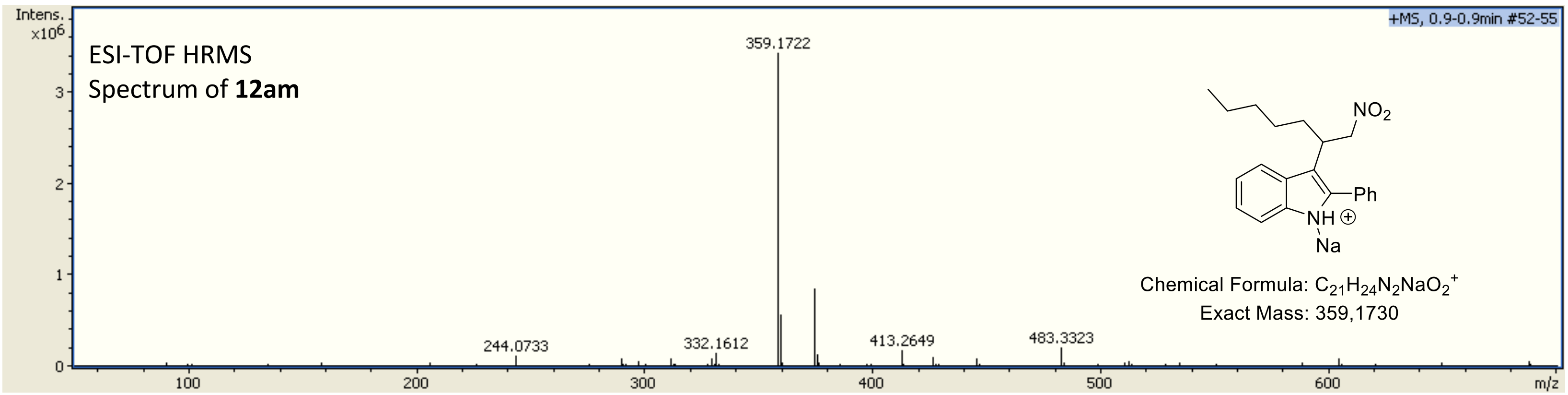
ESI-TOF HRMS
Spectrum of 12ag
+MS, 0.2-0.2min #11-14
401.1082
258.2795
469.0956
F
F
NO2
Ph
NH ⊕
Na
Chemical Formula: C22H16F2N2NaO2+
Exact Mass: 401,1072



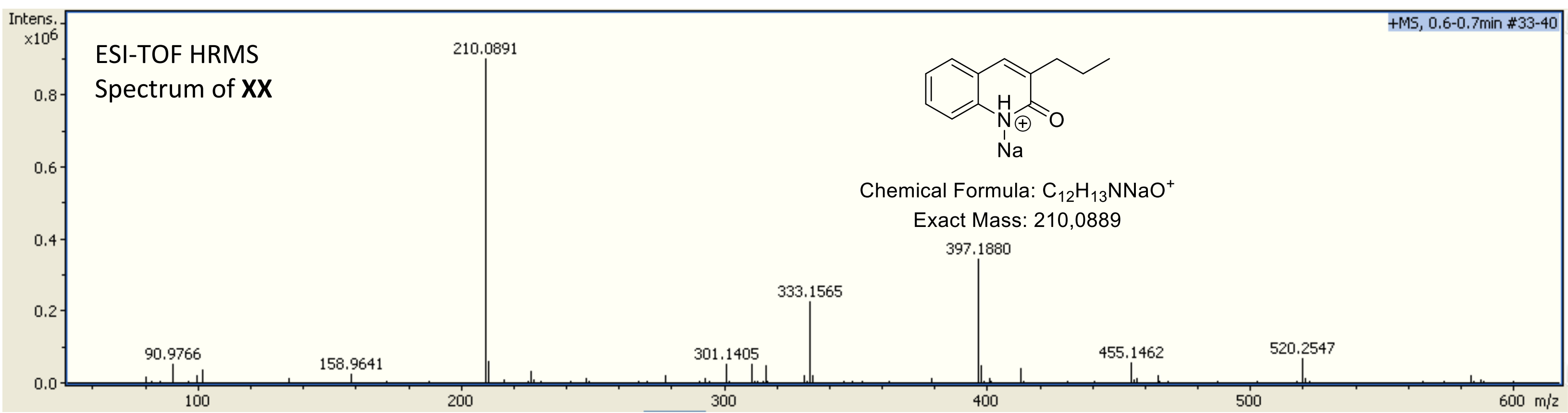
ESI-TOF HRMS
Spectrum of 12aj
+MS, 1.0-1.2min #57-71
395.1376
463.1252
OMe
NO2
Ph
NH ⊕
Na
Chemical Formula: C23H20N2NaO3+
Exact Mass: 395,1366



Intens.
x10^5
+MS, 0.4-0.5min #21-27
ESI-TOF HRMS
Spectrum of **12ak**
331.1408
301.0747
413.2653
102.1285
158.9641
226.9515
604.3278
NO2
Ph
NH ⊕
Na
Chemical Formula: $C_{19}H_{20}N_2NaO_2^+$
Exact Mass: 331,1417
m/z



Intens.
x10^6
+MS, 0.9-0.9min #52-55
ESI-TOF HRMS
Spectrum of **12am**
359.1722
244.0733
332.1612
413.2649
483.3323
NO2
Ph
NH ⊕
Na
Chemical Formula: $C_{21}H_{24}N_2NaO_2^+$
Exact Mass: 359,1730
m/z



Intens.
x10^6
+MS, 0.6-0.7min #33-40
ESI-TOF HRMS
Spectrum of **XX**
210.0891
397.1880
333.1565
90.9766
158.9641
301.1405
455.1462
520.2547
H
N⊕
O
Na
Chemical Formula: $C_{12}H_{13}NNaO^+$
Exact Mass: 210,0889
m/z

X-Ray Crystallography Data for compound **3ag**

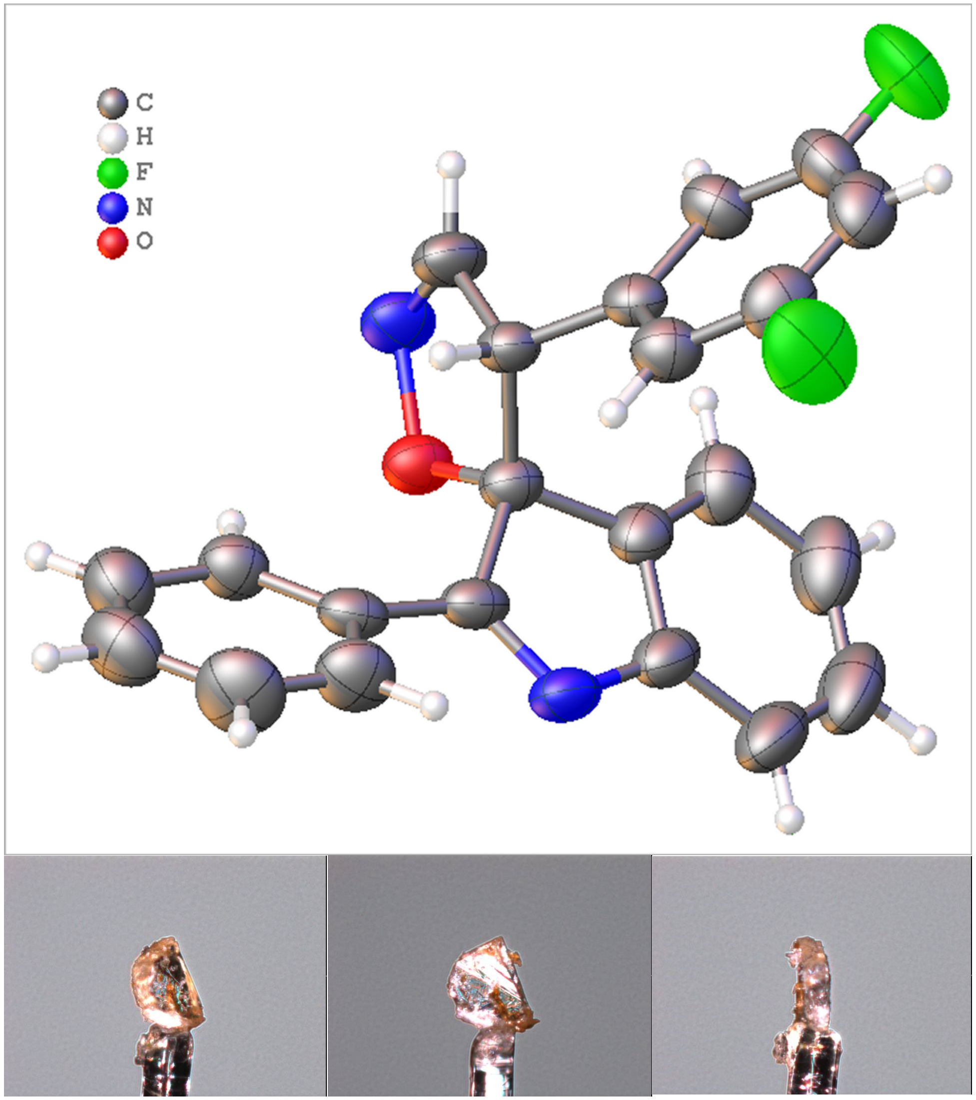


Figure S1. ORTEP drawing and sample microphotographs of the sample crystal

Table S1. Crystal data and structure refinement for 3ag.

Identification code	ANNA_ROS534_11
Empirical formula	$C_{22}H_{14}F_2N_2O$
Formula weight	360.35
Temperature/K	104(6)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	20.4243(4)
b/Å	15.3838(2)
c/Å	11.4158(2)
α/°	90
β/°	103.3576(18)
γ/°	90
Volume/Å^3	3489.86(10)
Z	8
ρ_{calc}g/cm^3	1.372
μ/mm^{-1}	0.833
F(000)	1488.0
Crystal size/mm^3	0.359 × 0.266 × 0.122
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	4.446 to 153.222
Index ranges	$-25 \leq h \leq 25$, $-14 \leq k \leq 19$, $-14 \leq l \leq 13$
Reflections collected	37744
Independent reflections	7322 [R_{int} = 0.0317, R_{sigma} = 0.0196]
Data/restraints/parameters	7322/0/542
Goodness-of-fit on F^2	1.025
Final R indexes [I>=2σ (I)]	R_1 = 0.0439, wR_2 = 0.1158
Final R indexes [all data]	R_1 = 0.0507, wR_2 = 0.1230
Largest diff. peak/hole / e Å^{-3}	0.15/-0.21

Table S2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 3ag. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
$O_{(1)}$	7055.1(5)	4446.3(6)	1974.4(9)	47.3(2)
$F_{(4)}$	5042.7(6)	3310.2(7)	7844.3(12)	82.6(3)
$F_{(3)}$	4900.2(6)	6218.3(8)	8941.4(12)	84.2(3)
$F_{(1)}$	9784.8(6)	6672.4(8)	4443.0(12)	89.1(4)
$O_{(2)}$	7876.3(6)	5305.1(7)	7647.1(12)	64.3(3)
$F_{(2)}$	9533.2(7)	4607.9(9)	7296.7(11)	94.2(4)
$N_{(4)}$	7115.6(7)	7225.4(7)	7983.9(11)	53.1(3)
$N_{(1)}$	7914.4(6)	2778.3(7)	3624.5(13)	54.1(3)
$N_{(2)}$	6975.5(7)	5364.3(8)	1958.0(13)	56.2(3)
$N_{(3)}$	7924.8(8)	4423.5(8)	8031.3(15)	65.7(4)
$C_{(9)}$	7699.0(7)	5013.8(7)	3805.8(12)	41.5(3)
$C_{(2)}$	7614.4(7)	4210.2(8)	2965.9(11)	41.0(3)
$C_{(1)}$	7476.1(7)	3380.4(8)	3620.1(12)	43.9(3)
$C_{(33)}$	6343.1(7)	4929.0(8)	8701.9(11)	44.4(3)
$C_{(11)}$	8402.4(7)	5230.2(8)	4502.8(12)	41.1(3)
$C_{(17)}$	6926.6(7)	3276.1(8)	4241.4(12)	44.8(3)
$C_{(18)}$	6294.3(8)	3635.7(9)	3779.3(13)	50.8(3)
$C_{(19)}$	5772.8(9)	3480.3(11)	4341.5(16)	60.8(4)
$C_{(20)}$	5880.6(10)	2985.8(12)	5373.0(17)	68.3(4)
$C_{(21)}$	6505.6(11)	2636.5(14)	5836.0(18)	76.2(5)
$C_{(22)}$	7027.3(9)	2776.9(11)	5281.8(15)	62.0(4)
$C_{(3)}$	8207.1(7)	3917.6(9)	2490.7(13)	46.8(3)
$C_{(25)}$	6781.2(7)	5964.9(8)	6874.8(12)	46.0(3)
$C_{(26)}$	6688.7(7)	6856.2(9)	6939.7(13)	48.4(3)
$C_{(31)}$	7093.5(7)	5059.7(8)	8872.2(12)	45.8(3)
$C_{(16)}$	8786.7(8)	5860.8(9)	4108.8(13)	50.5(3)
$C_{(24)}$	7306.4(7)	5713.1(8)	7980.4(12)	45.4(3)
$C_{(23)}$	7488.8(8)	6614.0(8)	8544.7(13)	48.0(3)
$C_{(34)}$	6030.0(8)	4140.8(9)	8334.5(13)	51.1(3)
$C_{(4)}$	8354.0(7)	3073.0(9)	2902.2(15)	53.7(3)

$C_{(38)}$	5954.9(8)	5630.5(9)	8915.7(13)	52.2(3)
$C_{(12)}$	8657.5(8)	4802.1(9)	5582.9(13)	50.9(3)
$C_{(10)}$	7333.9(8)	5670.9(9)	2920.8(15)	55.2(4)
$C_{(37)}$	5272.2(9)	5529.4(11)	8740.3(14)	58.0(4)
$C_{(32)}$	7505.1(8)	4290.3(9)	8667.7(16)	58.9(4)
$C_{(15)}$	9411.9(8)	6052.0(11)	4817.6(15)	57.7(4)
$C_{(35)}$	5342.0(9)	4082.8(10)	8192.3(14)	57.5(4)
$C_{(13)}$	9287.7(9)	5023.7(11)	6239.2(14)	59.5(4)
$C_{(36)}$	4941.6(9)	4759.9(12)	8384.7(14)	60.7(4)
$C_{(14)}$	9683.2(8)	5650.4(12)	5888.9(16)	62.1(4)
$C_{(27)}$	6260.0(9)	7302.4(11)	6025.5(16)	61.6(4)
$C_{(39)}$	7991(4)	6853(6)	9693(8)	40.1(13)
$C_{(40)}$	7885(4)	7577(5)	10335(6)	46.7(12)
$C_{(41)}$	8372(4)	7820(4)	11334(6)	59.7(14)
$C_{(42)}$	8965(5)	7343(6)	11684(9)	69.5(17)
$C_{(43)}$	9053(5)	6610(4)	11016(8)	71.3(18)
$C_{(44)}$	8562(5)	6362(5)	10024(9)	62.1(15)
$C_{(30)}$	6451.5(9)	5494.5(11)	5895.8(14)	60.3(4)
$C_{(8)}$	8534.0(8)	4297.7(12)	1696.8(15)	60.8(4)
$C_{(28)}$	5937.2(9)	6826.4(13)	5021.6(16)	69.8(5)
$C_{(29)}$	6029.0(10)	5939.9(14)	4959.5(15)	71.8(5)
$C_{(5)}$	8835.8(9)	2584.0(12)	2529(2)	73.1(5)
$C_{(7)}$	9013.7(9)	3800.7(16)	1304.9(18)	76.8(5)
$C_{(6)}$	9159.6(9)	2961.6(16)	1718(2)	82.0(6)
$C_{(50)}$	8054(7)	7312(11)	10387(11)	71(3)
$C_{(49)}$	8591(9)	7498(14)	11364(9)	88(5)
$C_{(48)}$	9153(10)	7018(16)	11470(12)	98(5)
$C_{(47)}$	9224(7)	6367(13)	10664(15)	97(4)
$C_{(46)}$	8682(7)	6198(11)	9712(16)	78(3)
$C_{(45)}$	8067(9)	6665(10)	9511(14)	60(3)

Table S3. Anisotropic Displacement Parameters (Å^2×10^3) for 3ag. The Anisotropic displacement factor exponent takes the form: -2π^2[h^{2}a*^{2}U$_{11}$+2hka*b*U$_{12}$+…].

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
$O_{(1)}$	52.4(5)	37.4(5)	47.0(5)	2.4(4)	1.2(4)	2.9(4)
$F_{(4)}$	78.8(7)	65.0(6)	94.6(8)	-2.6(5)	0.4(6)	-26.5(5)
$F_{(3)}$	80.2(7)	80.5(7)	95.7(8)	-2.6(6)	28.2(6)	27.7(6)
$F_{(1)}$	78.2(7)	94.5(8)	95.4(8)	5.5(7)	21.4(6)	-43.3(6)
$O_{(2)}$	73.6(7)	49.3(6)	77.8(7)	12.6(5)	33.2(6)	16.2(5)
$F_{(2)}$	88.5(8)	110.4(10)	67.3(7)	26.4(6)	-15.8(6)	0.3(7)
$N_{(4)}$	67.4(8)	32.4(5)	54.9(7)	0.8(5)	4.6(6)	-4.4(5)
$N_{(1)}$	55.7(7)	31.2(5)	70.8(8)	-2.3(5)	5.4(6)	4.8(5)
$N_{(2)}$	59.0(7)	37.8(6)	65.6(8)	10.8(5)	1.5(6)	5.3(5)
$N_{(3)}$	71.9(9)	41.3(7)	84.9(10)	1.9(6)	20.1(8)	13.6(6)
$C_{(9)}$	46.8(7)	28.3(5)	48.9(7)	-1.1(5)	9.9(5)	1.3(5)
$C_{(2)}$	45.1(6)	31.9(6)	43.2(6)	-0.6(5)	4.2(5)	2.4(5)
$C_{(1)}$	50.4(7)	27.7(5)	47.7(7)	-2.2(5)	-0.7(5)	-0.3(5)
$C_{(33)}$	57.8(8)	36.6(6)	36.8(6)	1.9(5)	6.8(5)	-0.8(5)
$C_{(11)}$	47.6(7)	31.3(5)	44.6(6)	-4.7(5)	11.1(5)	0.2(5)
$C_{(17)}$	54.3(7)	28.5(5)	47.1(7)	-1.2(5)	2.6(6)	-5.2(5)
$C_{(18)}$	54.8(8)	44.6(7)	50.1(7)	4.8(6)	6.5(6)	-1.7(6)
$C_{(19)}$	55.8(8)	61.8(9)	63.4(9)	0.0(7)	11.0(7)	-6.0(7)
$C_{(20)}$	75.4(11)	66.5(10)	67.1(10)	3.7(8)	24.7(9)	-15.6(8)
$C_{(21)}$	91.5(13)	73.8(11)	63.7(10)	24.8(9)	18.7(10)	-3.1(10)
$C_{(22)}$	70.4(10)	52.4(8)	58.4(9)	15.2(7)	5.2(7)	2.6(7)
$C_{(3)}$	47.1(7)	42.8(7)	48.5(7)	-10.5(5)	6.9(6)	0.9(5)
$C_{(25)}$	58.3(8)	36.5(6)	43.0(6)	1.4(5)	11.7(6)	-2.9(5)
$C_{(26)}$	54.2(7)	37.9(6)	50.5(7)	2.7(5)	6.9(6)	0.3(5)
$C_{(31)}$	58.2(8)	30.8(6)	45.6(7)	3.1(5)	6.1(6)	-0.1(5)
$C_{(16)}$	56.9(8)	44.5(7)	50.5(7)	0.8(6)	13.1(6)	-7.9(6)
$C_{(24)}$	53.8(7)	32.9(6)	49.2(7)	2.1(5)	11.4(6)	2.5(5)
$C_{(23)}$	56.8(8)	36.6(6)	48.1(7)	3.3(5)	7.2(6)	-7.3(5)
$C_{(34)}$	63.6(9)	38.1(6)	48.5(7)	0.6(5)	6.4(6)	-1.7(6)
$C_{(4)}$	49.2(7)	41.5(7)	65.2(9)	-13.1(6)	2.5(6)	6.0(6)
$C_{(38)}$	66.4(9)	41.8(7)	48.0(7)	-2.3(5)	12.6(6)	2.8(6)

$C_{(12)}$	57.6(8)	44.6(7)	49.1(7)	1.8(6)	9.3(6)	-0.2(6)
$C_{(10)}$	59.1(8)	31.0(6)	69.6(9)	2.3(6)	2.5(7)	4.8(6)
$C_{(37)}$	65.1(9)	60.3(9)	49.1(8)	4.6(6)	14.2(7)	13.5(7)
$C_{(32)}$	63.6(9)	32.9(6)	77.5(10)	7.0(6)	11.1(8)	5.3(6)
$C_{(15)}$	56.1(8)	55.2(8)	64.7(9)	-7.6(7)	19.9(7)	-15.4(7)
$C_{(35)}$	64.5(9)	53.2(8)	49.1(7)	4.4(6)	1.7(7)	-12.4(7)
$C_{(13)}$	62.6(9)	62.5(9)	48.2(8)	1.6(6)	2.2(7)	7.4(7)
$C_{(36)}$	56.1(8)	73.9(10)	49.8(8)	11.9(7)	7.5(6)	-1.2(7)
$C_{(14)}$	51.6(8)	68.5(10)	61.9(9)	-15.2(8)	4.4(7)	-2.5(7)
$C_{(27)}$	63.2(9)	50.8(8)	66.3(10)	13.1(7)	5.8(8)	6.3(7)
$C_{(39)}$	54(2)	29(3)	35(3)	-4(2)	4.4(17)	-9.8(19)
$C_{(40)}$	54(2)	41(2)	45.2(18)	-4.4(16)	10.7(16)	-5.9(15)
$C_{(41)}$	72(3)	55(2)	52.0(18)	-5.3(18)	13.9(19)	-16.0(19)
$C_{(42)}$	71(4)	72(3)	55(3)	4(3)	-7(2)	-23(3)
$C_{(43)}$	64(3)	63(3)	78(4)	7(3)	-5(2)	2(2)
$C_{(44)}$	66(4)	46(2)	67(3)	4.4(19)	0(2)	5.8(19)
$C_{(30)}$	81.4(11)	50.3(8)	47.4(8)	-5.6(6)	11.2(7)	-10.3(7)
$C_{(8)}$	56.4(8)	71.7(10)	54.9(8)	-4.8(7)	13.8(7)	0.8(7)
$C_{(28)}$	61.4(9)	82.0(12)	58.0(9)	19.2(8)	-2.5(7)	-2.3(8)
$C_{(29)}$	80.5(12)	81.4(12)	47.3(8)	-2.1(8)	1.8(8)	-18.0(9)
$C_{(5)}$	59.2(9)	59.8(10)	95.0(13)	-24.9(9)	6.9(9)	14.7(8)
$C_{(7)}$	58.0(10)	108.8(16)	66.5(10)	-19.5(10)	20.2(8)	2.9(10)
$C_{(6)}$	54.2(9)	99.7(15)	90.4(14)	-39.5(12)	13.2(9)	15.1(10)
$C_{(50)}$	71(6)	69(7)	69(4)	9(5)	9(4)	-15(4)
$C_{(49)}$	98(10)	107(10)	52(4)	-14(6)	6(5)	-50(9)
$C_{(48)}$	97(9)	126(14)	59(6)	18(7)	-8(5)	-53(8)
$C_{(47)}$	71(5)	114(9)	91(7)	34(6)	-13(4)	-10(5)
$C_{(46)}$	58(5)	74(7)	88(8)	23(5)	-14(4)	-6(4)
$C_{(45)}$	92(6)	38(5)	49(4)	-7(3)	17(4)	-21(4)

Table S4. Bond Lengths for 3ag.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
$O_{(1)}$	$N_{(2)}$	1.4211(15)	$C_{(25)}$	$C_{(30)}$	1.371(2)
$O_{(1)}$	$C_{(2)}$	1.4562(15)	$C_{(26)}$	$C_{(27)}$	1.380(2)
$F_{(4)}$	$C_{(35)}$	1.3534(18)	$C_{(31)}$	$C_{(24)}$	1.5622(18)
$F_{(3)}$	$C_{(37)}$	1.3542(19)	$C_{(31)}$	$C_{(32)}$	1.501(2)
$F_{(1)}$	$C_{(15)}$	1.3506(18)	$C_{(16)}$	$C_{(15)}$	1.376(2)
$O_{(2)}$	$N_{(3)}$	1.4218(17)	$C_{(24)}$	$C_{(23)}$	1.5374(18)
$O_{(2)}$	$C_{(24)}$	1.4484(18)	$C_{(23)}$	$C_{(39)}$	1.511(8)
$F_{(2)}$	$C_{(13)}$	1.3559(19)	$C_{(23)}$	$C_{(45)}$	1.420(18)
$N_{(4)}$	$C_{(26)}$	1.4226(18)	$C_{(34)}$	$C_{(35)}$	1.380(2)
$N_{(4)}$	$C_{(23)}$	1.2845(19)	$C_{(4)}$	$C_{(5)}$	1.381(2)
$N_{(1)}$	$C_{(1)}$	1.2872(18)	$C_{(38)}$	$C_{(37)}$	1.371(2)
$N_{(1)}$	$C_{(4)}$	1.426(2)	$C_{(12)}$	$C_{(13)}$	1.374(2)
$N_{(2)}$	$C_{(10)}$	1.263(2)	$C_{(37)}$	$C_{(36)}$	1.377(2)
$N_{(3)}$	$C_{(32)}$	1.262(2)	$C_{(15)}$	$C_{(14)}$	1.368(3)
$C_{(9)}$	$C_{(2)}$	1.5495(16)	$C_{(35)}$	$C_{(36)}$	1.373(3)
$C_{(9)}$	$C_{(11)}$	1.5099(18)	$C_{(13)}$	$C_{(14)}$	1.375(3)
$C_{(9)}$	$C_{(10)}$	1.5002(18)	$C_{(27)}$	$C_{(28)}$	1.391(3)
$C_{(2)}$	$C_{(1)}$	1.5377(17)	$C_{(39)}$	$C_{(40)}$	1.379(7)
$C_{(2)}$	$C_{(3)}$	1.5052(19)	$C_{(39)}$	$C_{(44)}$	1.367(11)
$C_{(1)}$	$C_{(17)}$	1.468(2)	$C_{(40)}$	$C_{(41)}$	1.381(11)
$C_{(33)}$	$C_{(31)}$	1.513(2)	$C_{(41)}$	$C_{(42)}$	1.393(9)
$C_{(33)}$	$C_{(34)}$	1.3896(19)	$C_{(42)}$	$C_{(43)}$	1.396(9)
$C_{(33)}$	$C_{(38)}$	1.394(2)	$C_{(43)}$	$C_{(44)}$	1.383(12)
$C_{(11)}$	$C_{(16)}$	1.3866(19)	$C_{(30)}$	$C_{(29)}$	1.389(3)
$C_{(11)}$	$C_{(12)}$	1.3888(19)	$C_{(8)}$	$C_{(7)}$	1.396(3)
$C_{(17)}$	$C_{(18)}$	1.392(2)	$C_{(28)}$	$C_{(29)}$	1.381(3)
$C_{(17)}$	$C_{(22)}$	1.389(2)	$C_{(5)}$	$C_{(6)}$	1.384(3)
$C_{(18)}$	$C_{(19)}$	1.385(2)	$C_{(7)}$	$C_{(6)}$	1.383(3)
$C_{(19)}$	$C_{(20)}$	1.376(3)	$C_{(50)}$	$C_{(49)}$	1.402(17)
$C_{(20)}$	$C_{(21)}$	1.373(3)	$C_{(50)}$	$C_{(45)}$	1.414(12)
$C_{(21)}$	$C_{(22)}$	1.376(3)	$C_{(49)}$	$C_{(48)}$	1.346(16)
$C_{(3)}$	$C_{(4)}$	1.391(2)	$C_{(48)}$	$C_{(47)}$	1.390(14)

$C_{(3)}$	$C_{(8)}$	1.374(2)	$C_{(47)}$	$C_{(46)}$	1.385(17)
$C_{(25)}$	$C_{(26)}$	1.3884(19)	$C_{(46)}$	$C_{(45)}$	1.419(17)
$C_{(25)}$	$C_{(24)}$	1.5060(19)			

Table S5. Bond Angles for 3ag.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
$N_{(2)}$	$O_{(1)}$	$C_{(2)}$	108.97(9)	$N_{(4)}$	$C_{(23)}$	$C_{(24)}$	113.13(12)
$N_{(3)}$	$O_{(2)}$	$C_{(24)}$	109.59(11)	$N_{(4)}$	$C_{(23)}$	$C_{(39)}$	117.4(4)
$C_{(23)}$	$N_{(4)}$	$C_{(26)}$	107.67(11)	$N_{(4)}$	$C_{(23)}$	$C_{(45)}$	129.6(7)
$C_{(1)}$	$N_{(1)}$	$C_{(4)}$	107.40(12)	$C_{(39)}$	$C_{(23)}$	$C_{(24)}$	129.2(4)
$C_{(10)}$	$N_{(2)}$	$O_{(1)}$	108.55(11)	$C_{(45)}$	$C_{(23)}$	$C_{(24)}$	116.9(7)
$C_{(32)}$	$N_{(3)}$	$O_{(2)}$	108.93(12)	$C_{(35)}$	$C_{(34)}$	$C_{(33)}$	118.50(14)
$C_{(11)}$	$C_{(9)}$	$C_{(2)}$	117.29(11)	$C_{(3)}$	$C_{(4)}$	$N_{(1)}$	112.13(13)
$C_{(10)}$	$C_{(9)}$	$C_{(2)}$	98.85(11)	$C_{(5)}$	$C_{(4)}$	$N_{(1)}$	126.40(16)
$C_{(10)}$	$C_{(9)}$	$C_{(11)}$	118.08(11)	$C_{(5)}$	$C_{(4)}$	$C_{(3)}$	121.21(18)
$O_{(1)}$	$C_{(2)}$	$C_{(9)}$	103.72(9)	$C_{(37)}$	$C_{(38)}$	$C_{(33)}$	119.15(14)
$O_{(1)}$	$C_{(2)}$	$C_{(1)}$	112.67(10)	$C_{(13)}$	$C_{(12)}$	$C_{(11)}$	118.57(14)
$O_{(1)}$	$C_{(2)}$	$C_{(3)}$	110.25(11)	$N_{(2)}$	$C_{(10)}$	$C_{(9)}$	115.62(12)
$C_{(1)}$	$C_{(2)}$	$C_{(9)}$	111.63(11)	$F_{(3)}$	$C_{(37)}$	$C_{(38)}$	118.55(15)
$C_{(3)}$	$C_{(2)}$	$C_{(9)}$	118.53(11)	$F_{(3)}$	$C_{(37)}$	$C_{(36)}$	117.83(16)
$C_{(3)}$	$C_{(2)}$	$C_{(1)}$	100.37(10)	$C_{(38)}$	$C_{(37)}$	$C_{(36)}$	123.62(15)
$N_{(1)}$	$C_{(1)}$	$C_{(2)}$	113.19(13)	$N_{(3)}$	$C_{(32)}$	$C_{(31)}$	116.16(13)
$N_{(1)}$	$C_{(1)}$	$C_{(17)}$	121.86(12)	$F_{(1)}$	$C_{(15)}$	$C_{(16)}$	118.64(15)
$C_{(17)}$	$C_{(1)}$	$C_{(2)}$	124.88(11)	$F_{(1)}$	$C_{(15)}$	$C_{(14)}$	117.42(15)
$C_{(34)}$	$C_{(33)}$	$C_{(31)}$	122.45(13)	$C_{(14)}$	$C_{(15)}$	$C_{(16)}$	123.93(15)
$C_{(34)}$	$C_{(33)}$	$C_{(38)}$	119.20(14)	$F_{(4)}$	$C_{(35)}$	$C_{(34)}$	118.03(15)
$C_{(38)}$	$C_{(33)}$	$C_{(31)}$	118.35(12)	$F_{(4)}$	$C_{(35)}$	$C_{(36)}$	117.91(16)
$C_{(16)}$	$C_{(11)}$	$C_{(9)}$	121.63(12)	$C_{(36)}$	$C_{(35)}$	$C_{(34)}$	124.06(15)
$C_{(16)}$	$C_{(11)}$	$C_{(12)}$	119.70(13)	$F_{(2)}$	$C_{(13)}$	$C_{(12)}$	118.39(16)
$C_{(12)}$	$C_{(11)}$	$C_{(9)}$	118.64(12)	$F_{(2)}$	$C_{(13)}$	$C_{(14)}$	117.93(15)
$C_{(18)}$	$C_{(17)}$	$C_{(1)}$	121.29(12)	$C_{(12)}$	$C_{(13)}$	$C_{(14)}$	123.68(15)
$C_{(22)}$	$C_{(17)}$	$C_{(1)}$	119.65(14)	$C_{(35)}$	$C_{(36)}$	$C_{(37)}$	115.47(15)

$C_{(22)}$	$C_{(17)}$	$C_{(18)}$	119.00(15)	$C_{(15)}$	$C_{(14)}$	$C_{(13)}$	115.64(14)
$C_{(19)}$	$C_{(18)}$	$C_{(17)}$	120.08(14)	$C_{(26)}$	$C_{(27)}$	$C_{(28)}$	117.27(15)
$C_{(20)}$	$C_{(19)}$	$C_{(18)}$	120.24(16)	$C_{(40)}$	$C_{(39)}$	$C_{(23)}$	119.9(7)
$C_{(21)}$	$C_{(20)}$	$C_{(19)}$	119.76(17)	$C_{(44)}$	$C_{(39)}$	$C_{(23)}$	118.1(6)
$C_{(20)}$	$C_{(21)}$	$C_{(22)}$	120.75(16)	$C_{(44)}$	$C_{(39)}$	$C_{(40)}$	121.9(7)
$C_{(21)}$	$C_{(22)}$	$C_{(17)}$	120.16(16)	$C_{(39)}$	$C_{(40)}$	$C_{(41)}$	119.1(5)
$C_{(4)}$	$C_{(3)}$	$C_{(2)}$	106.64(13)	$C_{(40)}$	$C_{(41)}$	$C_{(42)}$	120.4(5)
$C_{(8)}$	$C_{(3)}$	$C_{(2)}$	131.62(14)	$C_{(41)}$	$C_{(42)}$	$C_{(43)}$	118.9(6)
$C_{(8)}$	$C_{(3)}$	$C_{(4)}$	121.36(15)	$C_{(44)}$	$C_{(43)}$	$C_{(42)}$	120.6(6)
$C_{(26)}$	$C_{(25)}$	$C_{(24)}$	106.78(12)	$C_{(39)}$	$C_{(44)}$	$C_{(43)}$	119.1(6)
$C_{(30)}$	$C_{(25)}$	$C_{(26)}$	121.17(14)	$C_{(25)}$	$C_{(30)}$	$C_{(29)}$	117.89(15)
$C_{(30)}$	$C_{(25)}$	$C_{(24)}$	131.93(13)	$C_{(3)}$	$C_{(8)}$	$C_{(7)}$	117.55(18)
$C_{(25)}$	$C_{(26)}$	$N_{(4)}$	111.98(12)	$C_{(29)}$	$C_{(28)}$	$C_{(27)}$	121.21(15)
$C_{(27)}$	$C_{(26)}$	$N_{(4)}$	126.44(13)	$C_{(28)}$	$C_{(29)}$	$C_{(30)}$	121.01(16)
$C_{(27)}$	$C_{(26)}$	$C_{(25)}$	121.42(14)	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	117.52(18)
$C_{(33)}$	$C_{(31)}$	$C_{(24)}$	115.34(11)	$C_{(6)}$	$C_{(7)}$	$C_{(8)}$	120.9(2)
$C_{(32)}$	$C_{(31)}$	$C_{(33)}$	117.53(12)	$C_{(7)}$	$C_{(6)}$	$C_{(5)}$	121.44(17)
$C_{(32)}$	$C_{(31)}$	$C_{(24)}$	98.96(12)	$C_{(49)}$	$C_{(50)}$	$C_{(45)}$	124.6(11)
$C_{(15)}$	$C_{(16)}$	$C_{(11)}$	118.47(14)	$C_{(48)}$	$C_{(49)}$	$C_{(50)}$	116.9(9)
$O_{(2)}$	$C_{(24)}$	$C_{(25)}$	110.57(12)	$C_{(49)}$	$C_{(48)}$	$C_{(47)}$	123.6(10)
$O_{(2)}$	$C_{(24)}$	$C_{(31)}$	104.71(10)	$C_{(46)}$	$C_{(47)}$	$C_{(48)}$	117.9(10)
$O_{(2)}$	$C_{(24)}$	$C_{(23)}$	111.66(12)	$C_{(47)}$	$C_{(46)}$	$C_{(45)}$	123.3(11)
$C_{(25)}$	$C_{(24)}$	$C_{(31)}$	117.39(12)	$C_{(50)}$	$C_{(45)}$	$C_{(23)}$	116.1(12)
$C_{(25)}$	$C_{(24)}$	$C_{(23)}$	100.13(10)	$C_{(50)}$	$C_{(45)}$	$C_{(46)}$	113.7(13)
$C_{(23)}$	$C_{(24)}$	$C_{(31)}$	112.58(11)	$C_{(46)}$	$C_{(45)}$	$C_{(23)}$	130.1(9)

Table S6. Torsion Angles for 3ag.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
$O_{(1)}$	$N_{(2)}$	$C_{(10)}$	$C_{(9)}$	-3.0(2)	$C_{(25)}$	$C_{(24)}$	$C_{(23)}$	$C_{(39)}$	179.9(5)
$O_{(1)}$	$C_{(2)}$	$C_{(1)}$	$N_{(1)}$	122.10(12)	$C_{(25)}$	$C_{(24)}$	$C_{(23)}$	$C_{(45)}$	-168.7(8)
$O_{(1)}$	$C_{(2)}$	$C_{(1)}$	$C_{(17)}$	-60.95(16)	$C_{(25)}$	$C_{(30)}$	$C_{(29)}$	$C_{(28)}$	-0.9(3)
$O_{(1)}$	$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	-121.21(12)	$C_{(26)}$	$N_{(4)}$	$C_{(23)}$	$C_{(24)}$	-5.25(18)

$O_{(1)}$ $C_{(2)}$ $C_{(3)}$ $C_{(8)}$	51.64(19)	$C_{(26)}$ $N_{(4)}$ $C_{(23)}$ $C_{(39)}$	179.8(4)
$F_{(4)}$ $C_{(35)}$ $C_{(36)}$ $C_{(37)}$	179.83(14)	$C_{(26)}$ $N_{(4)}$ $C_{(23)}$ $C_{(45)}$	168.2(9)
$F_{(3)}$ $C_{(37)}$ $C_{(36)}$ $C_{(35)}$	179.96(14)	$C_{(26)}$ $C_{(25)}$ $C_{(24)}$ $O_{(2)}$	-121.47(13)
$F_{(1)}$ $C_{(15)}$ $C_{(14)}$ $C_{(13)}$	179.93(15)	$C_{(26)}$ $C_{(25)}$ $C_{(24)}$ $C_{(31)}$	118.53(13)
$O_{(2)}$ $N_{(3)}$ $C_{(32)}$ $C_{(31)}$	-1.3(2)	$C_{(26)}$ $C_{(25)}$ $C_{(24)}$ $C_{(23)}$	-3.60(15)
$O_{(2)}$ $C_{(24)}$ $C_{(23)}$ $N_{(4)}$	122.70(14)	$C_{(26)}$ $C_{(25)}$ $C_{(30)}$ $C_{(29)}$	1.0(3)
$O_{(2)}$ $C_{(24)}$ $C_{(23)}$ $C_{(39)}$	-63.1(5)	$C_{(26)}$ $C_{(27)}$ $C_{(28)}$ $C_{(29)}$	1.7(3)
$O_{(2)}$ $C_{(24)}$ $C_{(23)}$ $C_{(45)}$	-51.7(8)	$C_{(31)}$ $C_{(33)}$ $C_{(34)}$ $C_{(35)}$	-179.44(13)
$F_{(2)}$ $C_{(13)}$ $C_{(14)}$ $C_{(15)}$	-179.82(16)	$C_{(31)}$ $C_{(33)}$ $C_{(38)}$ $C_{(37)}$	178.57(13)
$N_{(4)}$ $C_{(26)}$ $C_{(27)}$ $C_{(28)}$	173.49(16)	$C_{(31)}$ $C_{(24)}$ $C_{(23)}$ $N_{(4)}$	-119.85(14)
$N_{(4)}$ $C_{(23)}$ $C_{(39)}$ $C_{(40)}$	23.7(9)	$C_{(31)}$ $C_{(24)}$ $C_{(23)}$ $C_{(39)}$	54.4(5)
$N_{(4)}$ $C_{(23)}$ $C_{(39)}$ $C_{(44)}$	-152.7(7)	$C_{(31)}$ $C_{(24)}$ $C_{(23)}$ $C_{(45)}$	65.8(8)
$N_{(4)}$ $C_{(23)}$ $C_{(45)}$ $C_{(50)}$	37.4(16)	$C_{(16)}$ $C_{(11)}$ $C_{(12)}$ $C_{(13)}$	0.5(2)
$N_{(4)}$ $C_{(23)}$ $C_{(45)}$ $C_{(46)}$	-138.6(14)	$C_{(16)}$ $C_{(15)}$ $C_{(14)}$ $C_{(13)}$	-0.1(3)
$N_{(1)}$ $C_{(1)}$ $C_{(17)}$ $C_{(18)}$	-145.31(14)	$C_{(24)}$ $O_{(2)}$ $N_{(3)}$ $C_{(32)}$	-7.51(19)
$N_{(1)}$ $C_{(1)}$ $C_{(17)}$ $C_{(22)}$	31.74(19)	$C_{(24)}$ $C_{(25)}$ $C_{(26)}$ $N_{(4)}$	1.02(17)
$N_{(1)}$ $C_{(4)}$ $C_{(5)}$ $C_{(6)}$	172.78(16)	$C_{(24)}$ $C_{(25)}$ $C_{(26)}$ $C_{(27)}$	176.77(15)
$N_{(2)}$ $O_{(1)}$ $C_{(2)}$ $C_{(9)}$	19.56(14)	$C_{(24)}$ $C_{(25)}$ $C_{(30)}$ $C_{(29)}$	-174.50(17)
$N_{(2)}$ $O_{(1)}$ $C_{(2)}$ $C_{(1)}$	140.44(12)	$C_{(24)}$ $C_{(31)}$ $C_{(32)}$ $N_{(3)}$	8.55(18)
$N_{(2)}$ $O_{(1)}$ $C_{(2)}$ $C_{(3)}$	-108.33(12)	$C_{(24)}$ $C_{(23)}$ $C_{(39)}$ $C_{(40)}$	-150.4(5)
$N_{(3)}$ $O_{(2)}$ $C_{(24)}$ $C_{(25)}$	-114.91(13)	$C_{(24)}$ $C_{(23)}$ $C_{(39)}$ $C_{(44)}$	33.3(10)
$N_{(3)}$ $O_{(2)}$ $C_{(24)}$ $C_{(31)}$	12.43(16)	$C_{(24)}$ $C_{(23)}$ $C_{(45)}$ $C_{(50)}$	-149.3(9)
$N_{(3)}$ $O_{(2)}$ $C_{(24)}$ $C_{(23)}$	134.53(13)	$C_{(24)}$ $C_{(23)}$ $C_{(45)}$ $C_{(46)}$	34.7(18)
$C_{(9)}$ $C_{(2)}$ $C_{(1)}$ $N_{(1)}$	-121.66(13)	$C_{(23)}$ $N_{(4)}$ $C_{(26)}$ $C_{(25)}$	2.67(19)
$C_{(9)}$ $C_{(2)}$ $C_{(1)}$ $C_{(17)}$	55.29(16)	$C_{(23)}$ $N_{(4)}$ $C_{(26)}$ $C_{(27)}$	-172.82(16)
$C_{(9)}$ $C_{(2)}$ $C_{(3)}$ $C_{(4)}$	119.55(13)	$C_{(23)}$ $C_{(39)}$ $C_{(40)}$ $C_{(41)}$	-175.5(6)
$C_{(9)}$ $C_{(2)}$ $C_{(3)}$ $C_{(8)}$	-67.6(2)	$C_{(23)}$ $C_{(39)}$ $C_{(44)}$ $C_{(43)}$	174.8(7)
$C_{(9)}$ $C_{(11)}$ $C_{(16)}$ $C_{(15)}$	177.07(13)	$C_{(34)}$ $C_{(33)}$ $C_{(31)}$ $C_{(24)}$	114.23(14)
$C_{(9)}$ $C_{(11)}$ $C_{(12)}$ $C_{(13)}$	-177.52(13)	$C_{(34)}$ $C_{(33)}$ $C_{(31)}$ $C_{(32)}$	-2.0(2)
$C_{(2)}$ $O_{(1)}$ $N_{(2)}$ $C_{(10)}$	-11.17(17)	$C_{(34)}$ $C_{(33)}$ $C_{(38)}$ $C_{(37)}$	-1.0(2)
$C_{(2)}$ $C_{(9)}$ $C_{(11)}$ $C_{(16)}$	98.18(15)	$C_{(34)}$ $C_{(35)}$ $C_{(36)}$ $C_{(37)}$	0.0(2)
$C_{(2)}$ $C_{(9)}$ $C_{(11)}$ $C_{(12)}$	-83.86(16)	$C_{(4)}$ $N_{(1)}$ $C_{(1)}$ $C_{(2)}$	-5.39(15)
$C_{(2)}$ $C_{(9)}$ $C_{(10)}$ $N_{(2)}$	14.51(18)	$C_{(4)}$ $N_{(1)}$ $C_{(1)}$ $C_{(17)}$	177.55(12)
$C_{(2)}$ $C_{(1)}$ $C_{(17)}$ $C_{(18)}$	37.99(18)	$C_{(4)}$ $C_{(3)}$ $C_{(8)}$ $C_{(7)}$	0.8(2)

$C_{(2)}$	$C_{(1)}$	$C_{(17)}$	$C_{(22)}$	-144.96(14)	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	$C_{(7)}$	0.8(3)
$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	$N_{(1)}$	-0.65(16)	$C_{(38)}$	$C_{(33)}$	$C_{(31)}$	$C_{(24)}$	-65.32(16)
$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	$C_{(5)}$	173.86(14)	$C_{(38)}$	$C_{(33)}$	$C_{(31)}$	$C_{(32)}$	178.49(13)
$C_{(2)}$	$C_{(3)}$	$C_{(8)}$	$C_{(7)}$	-171.16(15)	$C_{(38)}$	$C_{(33)}$	$C_{(34)}$	$C_{(35)}$	0.1(2)
$C_{(1)}$	$N_{(1)}$	$C_{(4)}$	$C_{(3)}$	3.83(16)	$C_{(38)}$	$C_{(37)}$	$C_{(36)}$	$C_{(35)}$	-0.9(2)
$C_{(1)}$	$N_{(1)}$	$C_{(4)}$	$C_{(5)}$	-170.34(15)	$C_{(12)}$	$C_{(11)}$	$C_{(16)}$	$C_{(15)}$	-0.9(2)
$C_{(1)}$	$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	-2.19(13)	$C_{(12)}$	$C_{(13)}$	$C_{(14)}$	$C_{(15)}$	-0.4(3)
$C_{(1)}$	$C_{(2)}$	$C_{(3)}$	$C_{(8)}$	170.67(15)	$C_{(10)}$	$C_{(9)}$	$C_{(2)}$	$O_{(1)}$	-19.16(13)
$C_{(1)}$	$C_{(17)}$	$C_{(18)}$	$C_{(19)}$	175.82(13)	$C_{(10)}$	$C_{(9)}$	$C_{(2)}$	$C_{(1)}$	-140.74(12)
$C_{(1)}$	$C_{(17)}$	$C_{(22)}$	$C_{(21)}$	-176.54(16)	$C_{(10)}$	$C_{(9)}$	$C_{(2)}$	$C_{(3)}$	103.41(13)
$C_{(33)}$	$C_{(31)}$	$C_{(24)}$	$O_{(2)}$	-138.18(12)	$C_{(10)}$	$C_{(9)}$	$C_{(11)}$	$C_{(16)}$	-19.95(19)
$C_{(33)}$	$C_{(31)}$	$C_{(24)}$	$C_{(25)}$	-15.15(17)	$C_{(10)}$	$C_{(9)}$	$C_{(11)}$	$C_{(12)}$	158.00(13)
$C_{(33)}$	$C_{(31)}$	$C_{(24)}$	$C_{(23)}$	100.32(14)	$C_{(32)}$	$C_{(31)}$	$C_{(24)}$	$O_{(2)}$	-11.85(14)
$C_{(33)}$	$C_{(31)}$	$C_{(32)}$	$N_{(3)}$	133.36(16)	$C_{(32)}$	$C_{(31)}$	$C_{(24)}$	$C_{(25)}$	111.18(13)
$C_{(33)}$	$C_{(34)}$	$C_{(35)}$	$F_{(4)}$	-179.44(13)	$C_{(32)}$	$C_{(31)}$	$C_{(24)}$	$C_{(23)}$	-133.35(12)
$C_{(33)}$	$C_{(34)}$	$C_{(35)}$	$C_{(36)}$	0.4(2)	$C_{(27)}$	$C_{(28)}$	$C_{(29)}$	$C_{(30)}$	-0.5(3)
$C_{(33)}$	$C_{(38)}$	$C_{(37)}$	$F_{(3)}$	-179.44(13)	$C_{(39)}$	$C_{(40)}$	$C_{(41)}$	$C_{(42)}$	0.5(10)
$C_{(33)}$	$C_{(38)}$	$C_{(37)}$	$C_{(36)}$	1.5(2)	$C_{(40)}$	$C_{(39)}$	$C_{(44)}$	$C_{(43)}$	-1.5(14)
$C_{(11)}$	$C_{(9)}$	$C_{(2)}$	$O_{(1)}$	-147.22(11)	$C_{(40)}$	$C_{(41)}$	$C_{(42)}$	$C_{(43)}$	-0.8(11)
$C_{(11)}$	$C_{(9)}$	$C_{(2)}$	$C_{(1)}$	91.20(14)	$C_{(41)}$	$C_{(42)}$	$C_{(43)}$	$C_{(44)}$	0.0(12)
$C_{(11)}$	$C_{(9)}$	$C_{(2)}$	$C_{(3)}$	-24.65(17)	$C_{(42)}$	$C_{(43)}$	$C_{(44)}$	$C_{(39)}$	1.1(13)
$C_{(11)}$	$C_{(9)}$	$C_{(10)}$	$N_{(2)}$	142.02(15)	$C_{(44)}$	$C_{(39)}$	$C_{(40)}$	$C_{(41)}$	0.7(12)
$C_{(11)}$	$C_{(16)}$	$C_{(15)}$	$F_{(1)}$	-179.31(14)	$C_{(30)}$	$C_{(25)}$	$C_{(26)}$	$N_{(4)}$	-175.48(14)
$C_{(11)}$	$C_{(16)}$	$C_{(15)}$	$C_{(14)}$	0.7(3)	$C_{(30)}$	$C_{(25)}$	$C_{(26)}$	$C_{(27)}$	0.3(2)
$C_{(11)}$	$C_{(12)}$	$C_{(13)}$	$F_{(2)}$	179.61(14)	$C_{(30)}$	$C_{(25)}$	$C_{(24)}$	$O_{(2)}$	54.5(2)
$C_{(11)}$	$C_{(12)}$	$C_{(13)}$	$C_{(14)}$	0.2(3)	$C_{(30)}$	$C_{(25)}$	$C_{(24)}$	$C_{(31)}$	-65.5(2)
$C_{(17)}$	$C_{(18)}$	$C_{(19)}$	$C_{(20)}$	1.4(2)	$C_{(30)}$	$C_{(25)}$	$C_{(24)}$	$C_{(23)}$	172.38(17)
$C_{(18)}$	$C_{(17)}$	$C_{(22)}$	$C_{(21)}$	0.6(2)	$C_{(8)}$	$C_{(3)}$	$C_{(4)}$	$N_{(1)}$	-174.40(13)
$C_{(18)}$	$C_{(19)}$	$C_{(20)}$	$C_{(21)}$	-0.8(3)	$C_{(8)}$	$C_{(3)}$	$C_{(4)}$	$C_{(5)}$	0.1(2)
$C_{(19)}$	$C_{(20)}$	$C_{(21)}$	$C_{(22)}$	0.2(3)	$C_{(8)}$	$C_{(7)}$	$C_{(6)}$	$C_{(5)}$	0.2(3)
$C_{(20)}$	$C_{(21)}$	$C_{(22)}$	$C_{(17)}$	0.0(3)	$C_{(50)}$	$C_{(49)}$	$C_{(48)}$	$C_{(47)}$	0.6(16)
$C_{(22)}$	$C_{(17)}$	$C_{(18)}$	$C_{(19)}$	-1.2(2)	$C_{(49)}$	$C_{(50)}$	$C_{(45)}$	$C_{(23)}$	-176.0(9)
$C_{(3)}$	$C_{(2)}$	$C_{(1)}$	$N_{(1)}$	4.85(14)	$C_{(49)}$	$C_{(50)}$	$C_{(45)}$	$C_{(46)}$	0.7(19)
$C_{(3)}$	$C_{(2)}$	$C_{(1)}$	$C_{(17)}$	-178.20(12)	$C_{(49)}$	$C_{(48)}$	$C_{(47)}$	$C_{(46)}$	-0.8(18)

$C_{(3)}$ $C_{(4)}$ $C_{(5)}$ $C_{(6)}$	-0.9(2)	$C_{(48)}$ $C_{(47)}$ $C_{(46)}$ $C_{(45)}$	1(2)
$C_{(3)}$ $C_{(8)}$ $C_{(7)}$ $C_{(6)}$	-1.0(3)	$C_{(47)}$ $C_{(46)}$ $C_{(45)}$ $C_{(23)}$	175.1(12)
$C_{(25)}$ $C_{(26)}$ $C_{(27)}$ $C_{(28)}$	-1.6(3)	$C_{(47)}$ $C_{(46)}$ $C_{(45)}$ $C_{(50)}$	-1(2)
$C_{(25)}$ $C_{(24)}$ $C_{(23)}$ $N_{(4)}$	5.63(17)	$C_{(45)}$ $C_{(50)}$ $C_{(49)}$ $C_{(48)}$	-0.5(17)

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

Atom	*x*	*y*	*z*	U(eq)
$H_{(9)}$	7426.03	4911.75	4394.59	50
$H_{(18)}$	6221.75	3980.7	3092.39	61
$H_{(19)}$	5348.36	3711.05	4020.74	73
$H_{(20)}$	5531.45	2888.57	5755.06	82
$H_{(21)}$	6577.07	2301.28	6531.94	91
$H_{(22)}$	7448.3	2537	5604.52	74
$H_{(31)}$	7273.06	5270.99	9693.53	55
$H_{(16)}$	8625.75	6148	3382.43	61
$H_{(34)}$	6278.65	3663.27	8187.75	61
$H_{(38)}$	6156.37	6160.48	9174.11	63
$H_{(12)}$	8407.18	4374.59	5856.25	61
$H_{(10)}$	7369.11	6265.25	3071.46	66
$H_{(32)}$	7453.76	3746.63	8991.4	71
$H_{(36)}$	4478.52	4702.69	8281.89	73
$H_{(14)}$	10107.55	5791.91	6350.2	75
$H_{(27)}$	6189.84	7897.02	6078.38	74
$H_{(40)}$	7490.92	7898.46	10098.53	56
$H_{(41)}$	8304.22	8305.5	11777.31	72
$H_{(42)}$	9295.92	7509.69	12350.54	83
$H_{(43)}$	9446.82	6285.71	11241.04	86
$H_{(44)}$	8618.35	5867.69	9588.88	74
$H_{(30)}$	6508.77	4896.2	5859.36	72
$H_{(8)}$	8438.99	4865.18	1430.61	73
$H_{(28)}$	5654.36	7110.47	4380.59	84
$H_{(29)}$	5804.55	5636.2	4280.63	86

$H_{(5)}$	8938.6	2022.04	2812.97	88
$H_{(7)}$	9238.49	4037.01	758.07	92
$H_{(6)}$	9482.28	2644.2	1445.31	98
$H_{(50)}$	7661.94	7635.48	10308.26	85
$H_{(49)}$	8560.61	7933.52	11914.39	105
$H_{(48)}$	9513.2	7128.78	12116.6	118
$H_{(47)}$	9622.47	6054.4	10762.35	117
$H_{(46)}$	8723.54	5756.92	9176.63	94

Table S8. Atomic Occupancy for ANNA_ROS534_11.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
$C_{(39)}$	0.58(2)	$C_{(40)}$	0.58(2)	$H_{(40)}$	0.58(2)
$C_{(41)}$	0.58(2)	$H_{(41)}$	0.58(2)	$C_{(42)}$	0.58(2)
$H_{(42)}$	0.58(2)	$C_{(43)}$	0.58(2)	$H_{(43)}$	0.58(2)
$C_{(44)}$	0.58(2)	$H_{(44)}$	0.58(2)	$C_{(50)}$	0.42(2)
$H_{(50)}$	0.42(2)	$C_{(49)}$	0.42(2)	$H_{(49)}$	0.42(2)
$C_{(48)}$	0.42(2)	$H_{(48)}$	0.42(2)	$C_{(47)}$	0.42(2)
$H_{(47)}$	0.42(2)	$C_{(46)}$	0.42(2)	$H_{(46)}$	0.42(2)
$C_{(45)}$	0.42(2)				

Experimental

Single crystals of $C_{22}H_{14}F_2N_2O$ (3ag) were prepared by slow evaporation of saturated solution in EtOAc/hexane. A suitable crystal was selected and mounted with acrylic glue on a glass stick in a SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer. The crystal was kept at 104(6) K during data collection. Using Olex2,[S1] the structure was solved with the ShelXT[S2] structure solution program using Intrinsic Phasing and refined with the ShelXL[S3] refinement package using Least Squares minimization.

Crystal structure determination of [ANNA_ROS534_11]

Crystal Data for $C_{22}H_{14}F_2N_2O$ (M =360.35 g/mol): monoclinic, space group P2₁/c (no. 14), a = 20.4243(4) Å, b = 15.3838(2) Å, c = 11.4158(2) Å, β = 103.3576(18)°, V = 3489.86(10) Å^3, Z = 8, T = 104(6) K, μ(CuKα) = 0.833 mm^{-1}, $Dcalc$ = 1.372 g/cm^3, 37744 reflections measured (4.446° ≤ 2Θ ≤ 153.222°), 7322 unique (R_{int} = 0.0317, R_{sigma} = 0.0196) which were used in all calculations. The final R_1 was 0.0439 (I > 2σ(I)) and wR_2 was 0.1230 (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. Others
 Sof(C50)=Sof(H50)=Sof(C49)=Sof(H49)=Sof(C48)=Sof(H48)=Sof(C47)=Sof(H47)=
 Sof(C46)=Sof(H46)=Sof(C45)=1-FVAR(1)
 Sof(C39)=Sof(C40)=Sof(H40)=Sof(C41)=Sof(H41)=Sof(C42)=Sof(H42)=Sof(C43)=
 Sof(H43)=Sof(C44)=Sof(H44)=FVAR(1)
3.a Ternary CH refined with riding coordinates:
 C9(H9), C31(H31)
3.b Aromatic/amide H refined with riding coordinates:
 C18(H18), C19(H19), C20(H20), C21(H21), C22(H22), C16(H16), C34(H34),
 C38(H38), C12(H12), C10(H10), C32(H32), C36(H36), C14(H14), C27(H27), C40(H40),
  C41(H41), C42(H42), C43(H43), C44(H44), C30(H30), C8(H8), C28(H28), C29(H29),
 C5(H5), C7(H7), C6(H6), C50(H50), C49(H49), C48(H48), C47(H47), C46(H46)
```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(S2) Sheldrick, G.M. SHELXT - Integrated Space-Group and Crystal-Structure Determination. *Acta Cryst*. **2015**, *A71*, 3-8.

(S3) Sheldrick, G.M. Crystal Structure Refinement with SHELXL. *Acta Cryst*. **2015**, *C71*, 3-8.