

Synthesis of 2-(1*H*-Indol-2-yl)acetamides via Bronsted Acid-Assisted Cyclization Cascade

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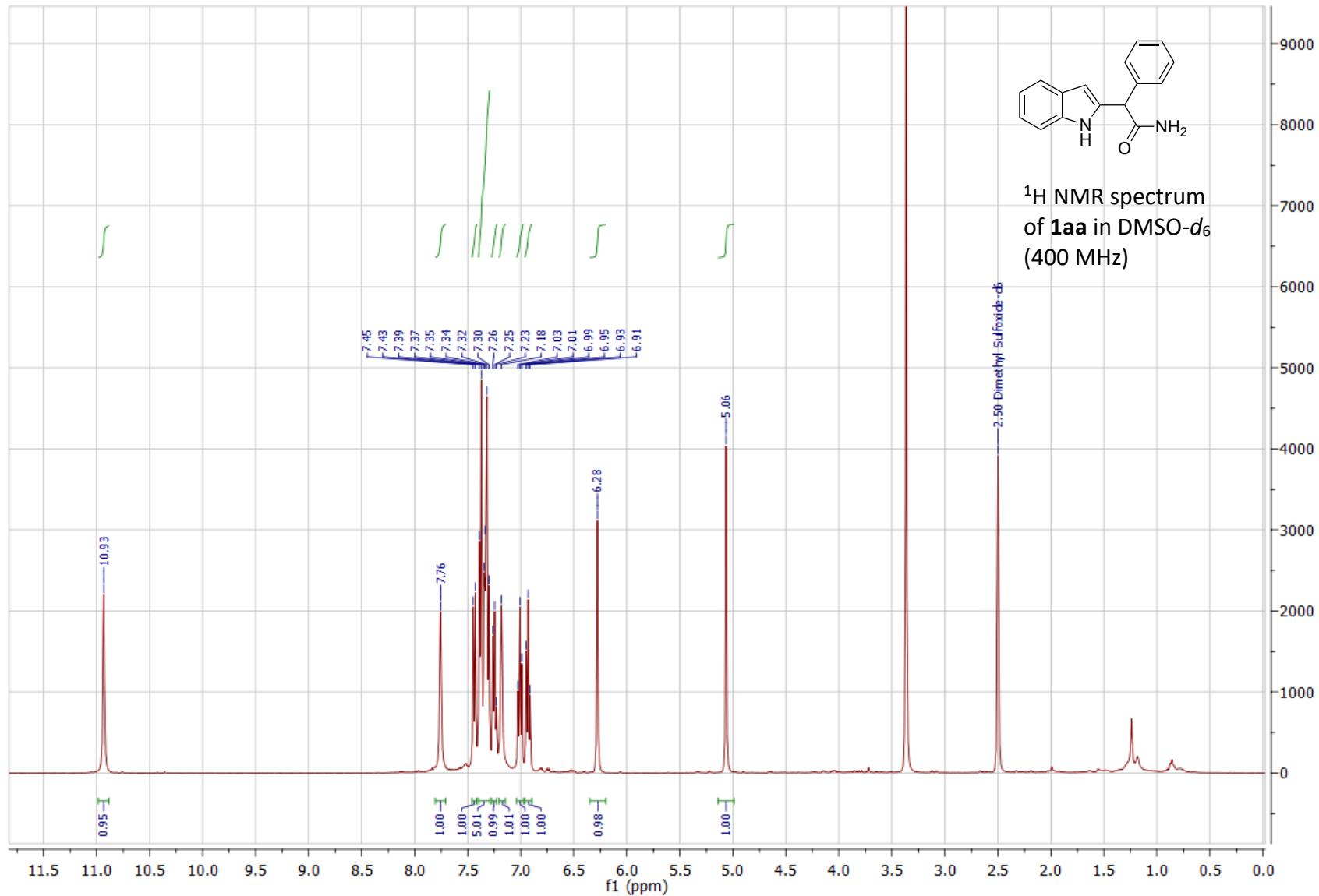
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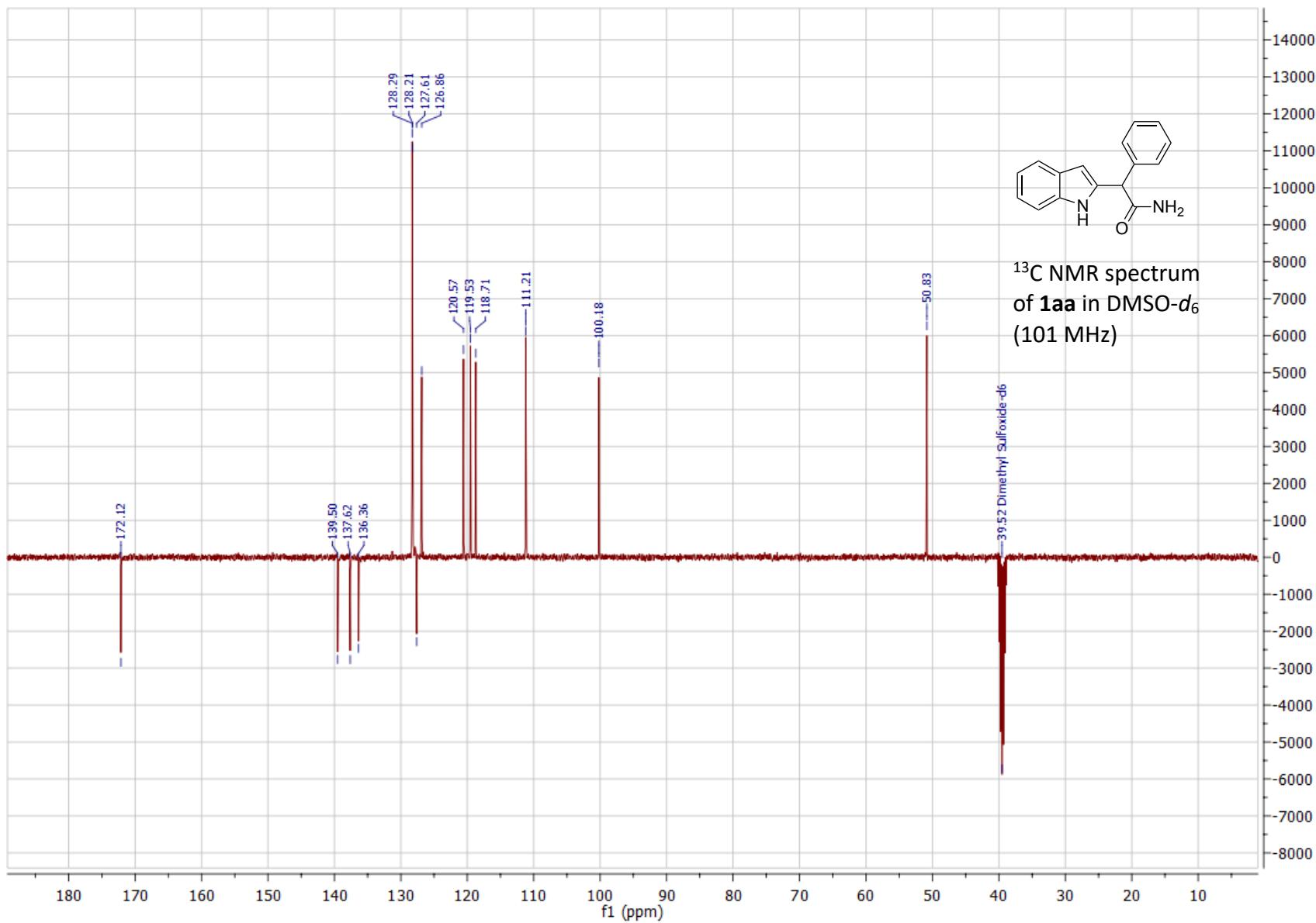
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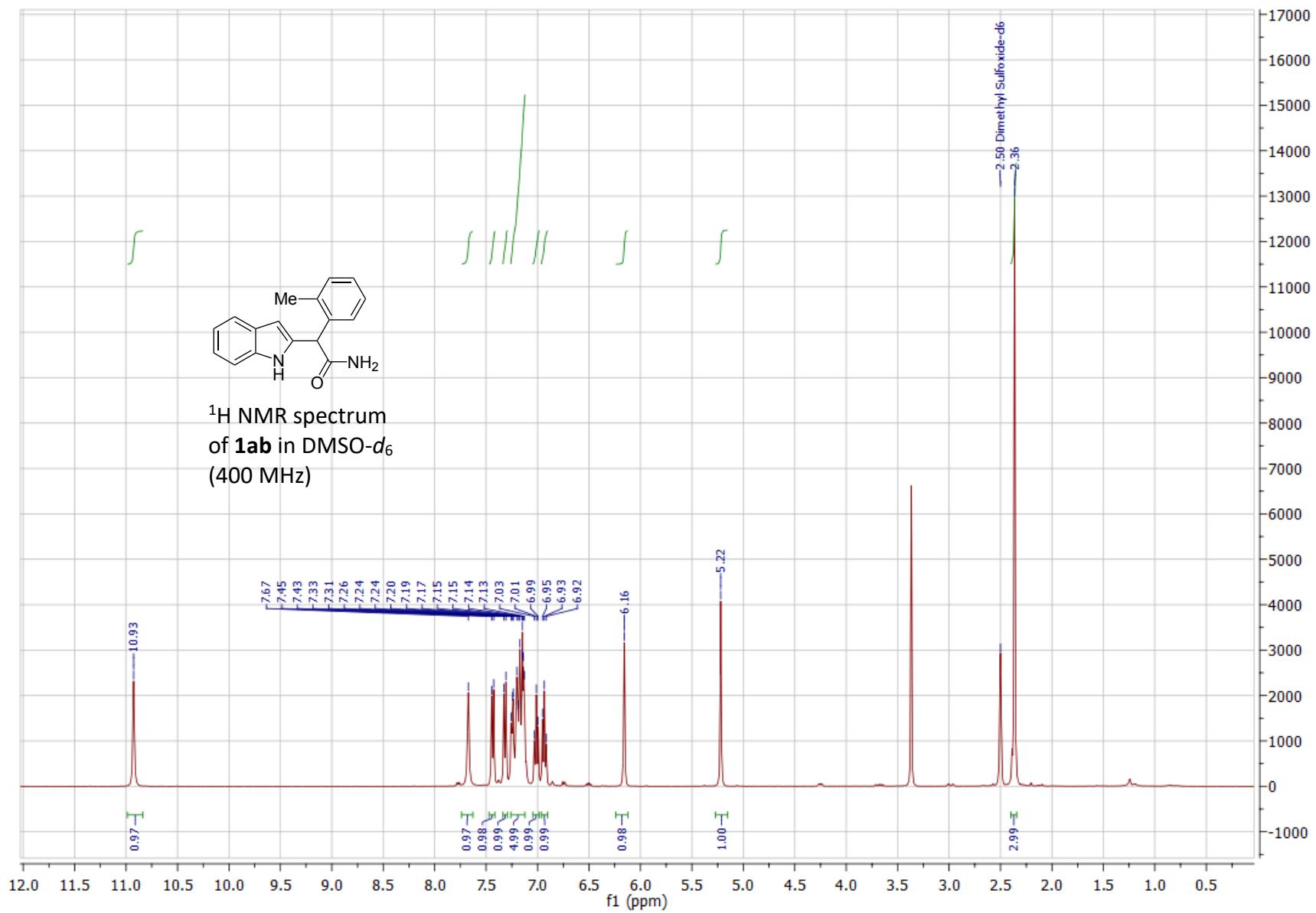
SUPPORTING INFORMATION

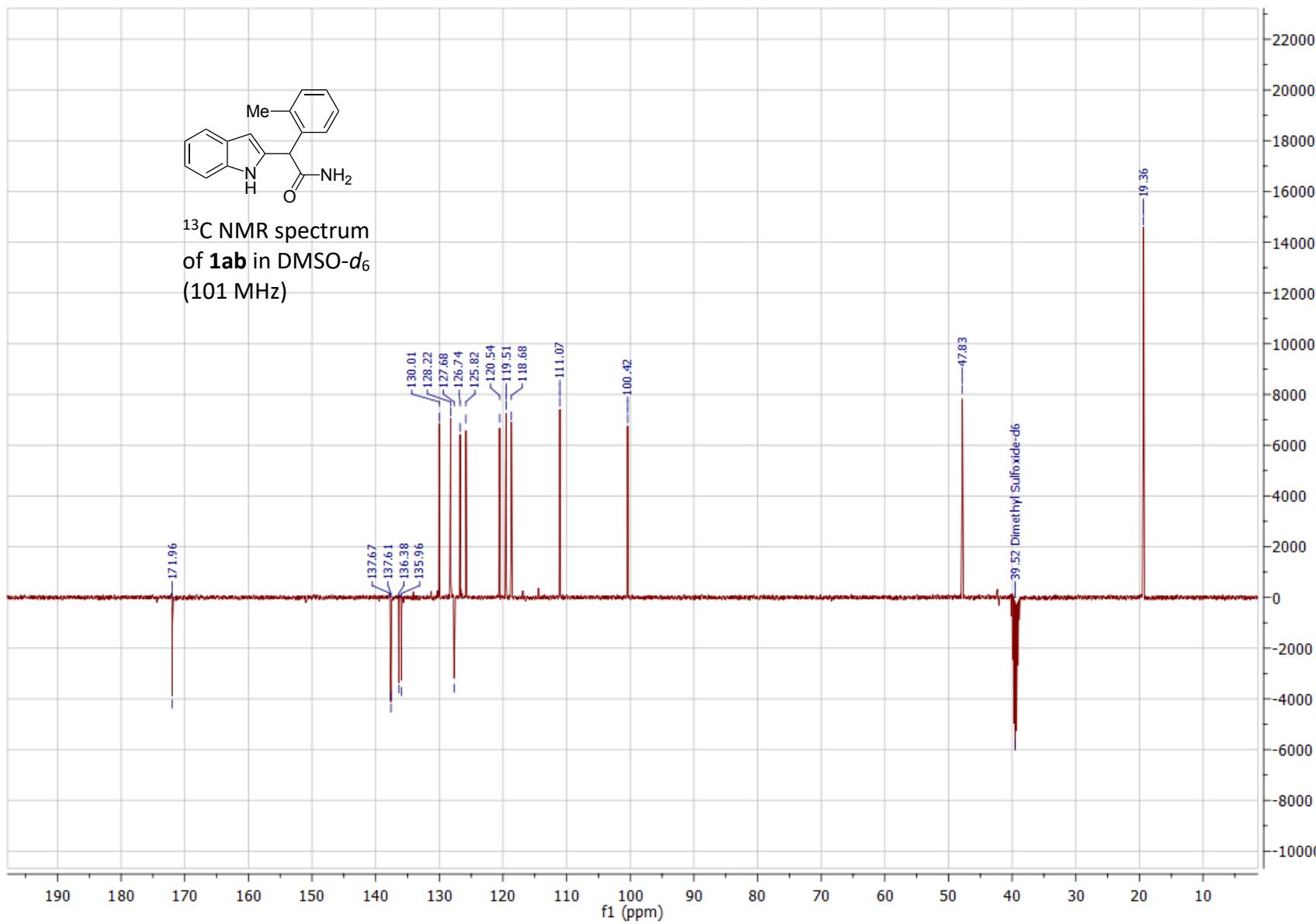
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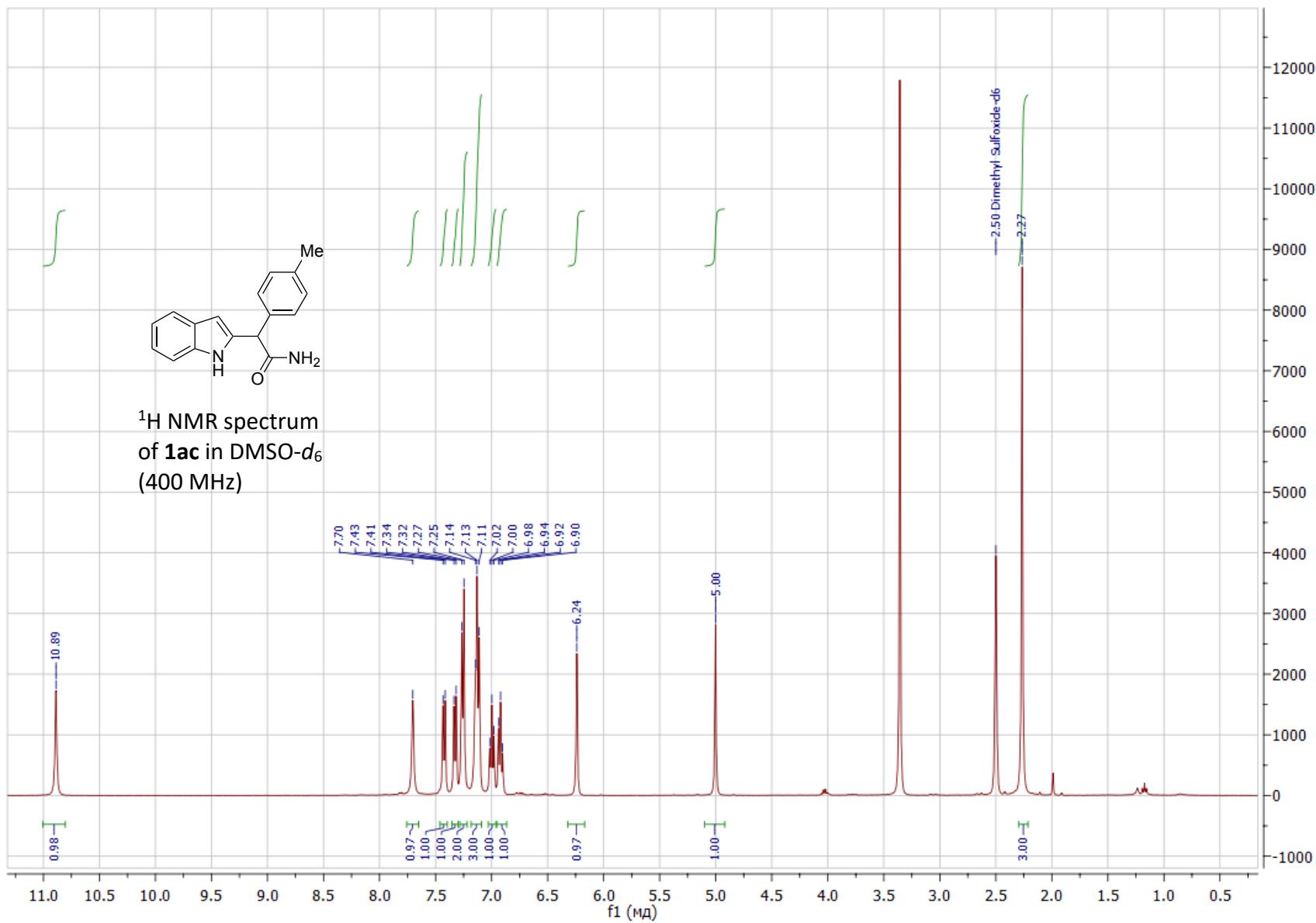
^1H and ^{13}C Spectral Charts of 2-(1*H*-Indol-2-yl)acetamides 1

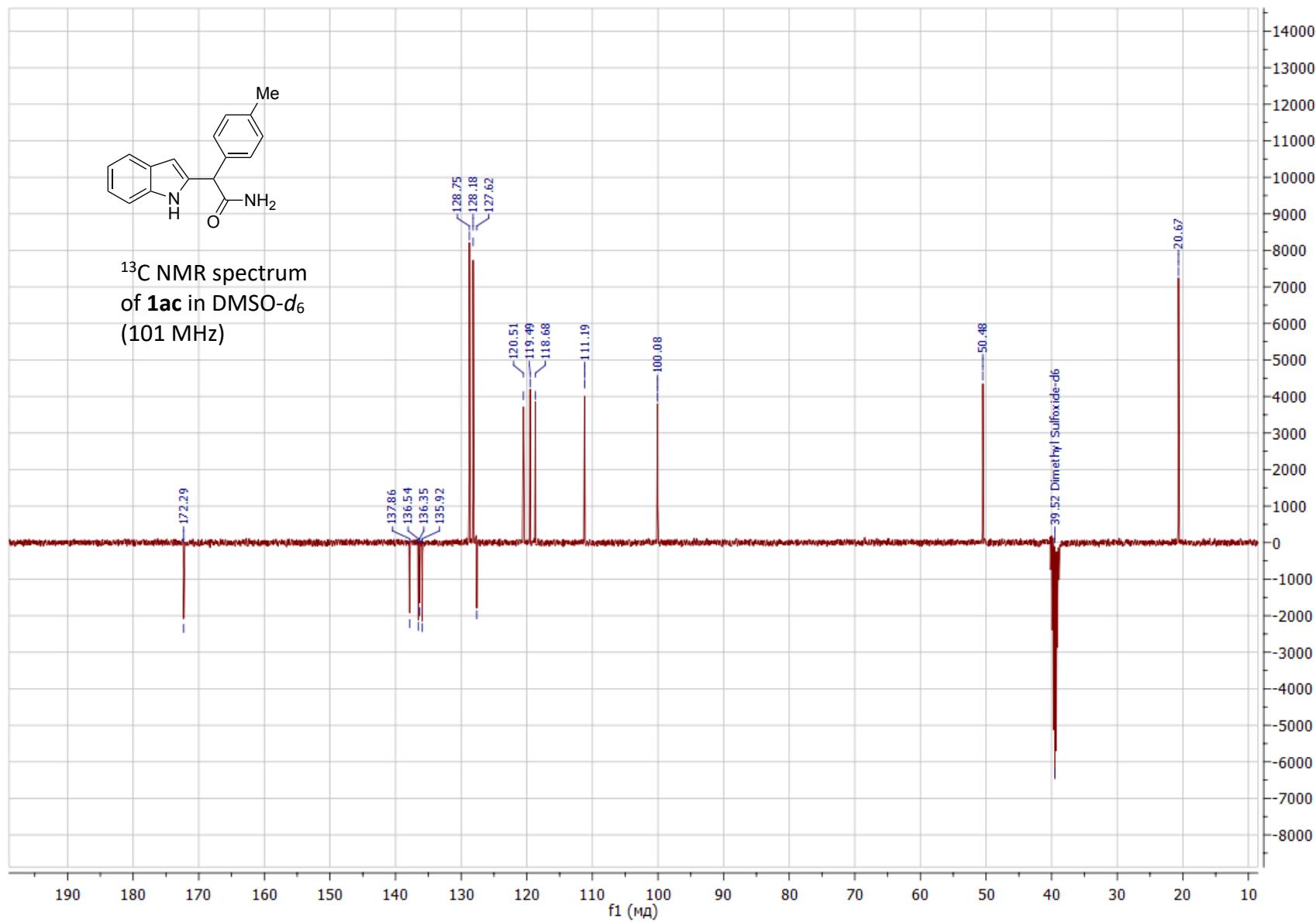


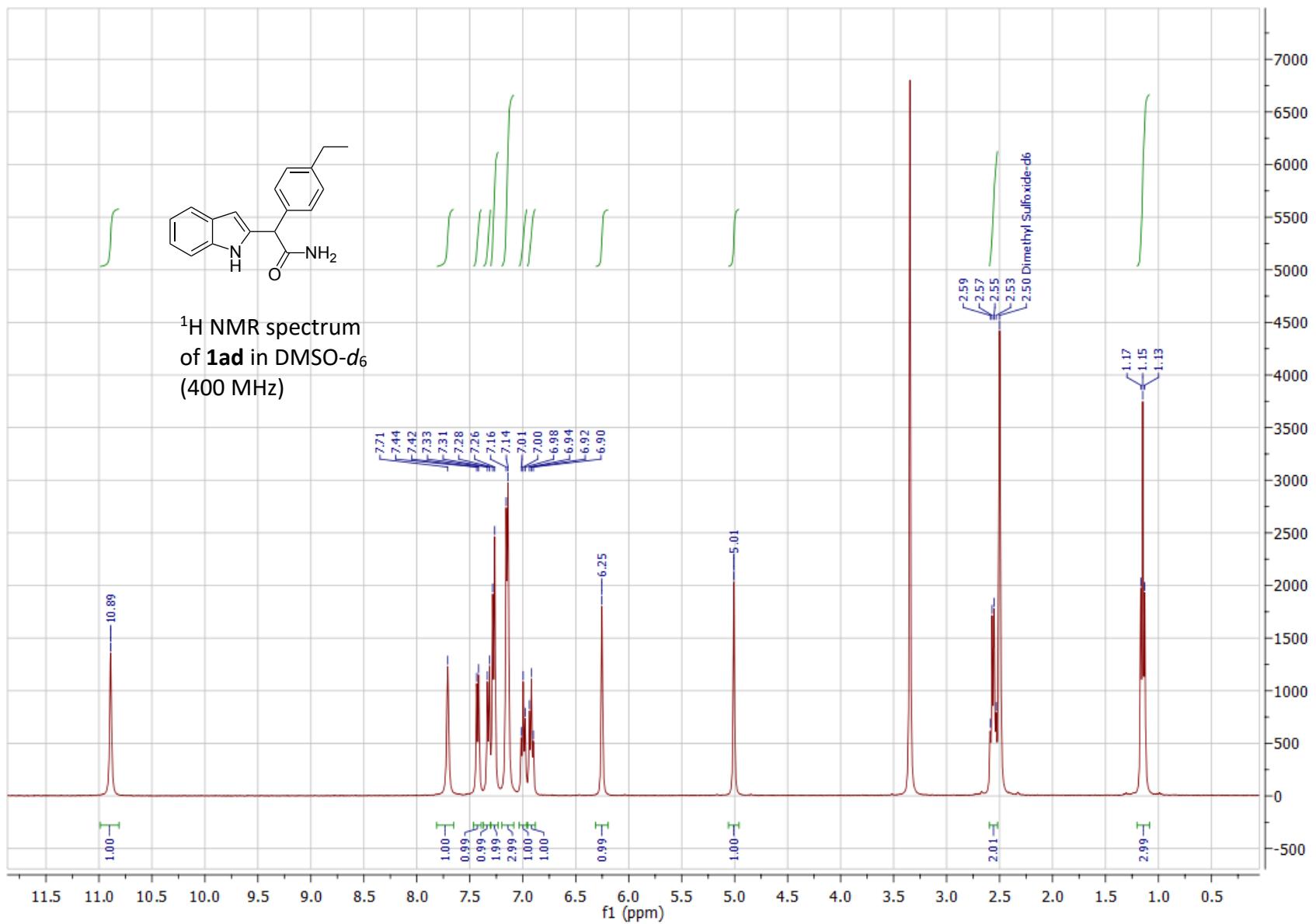


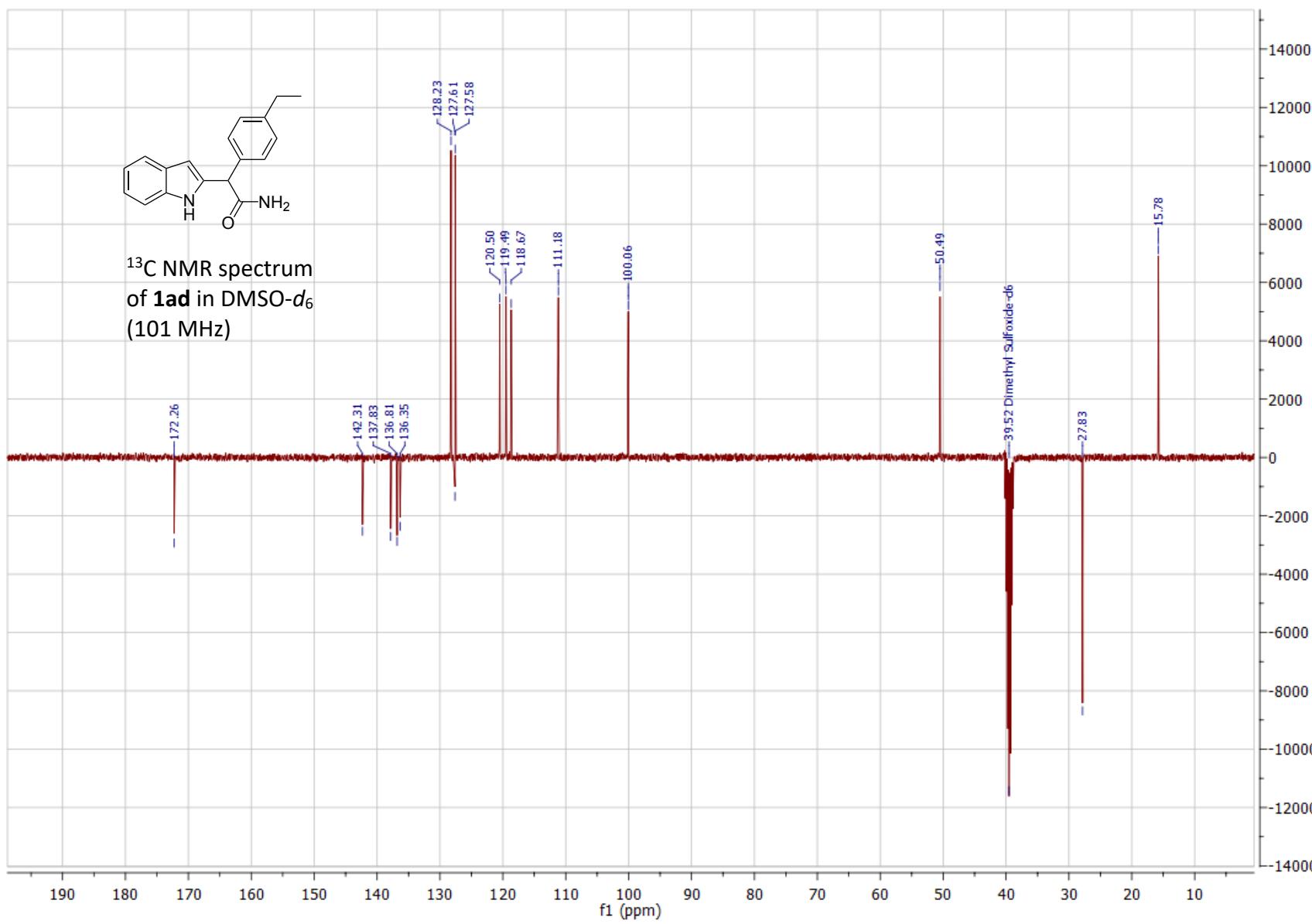


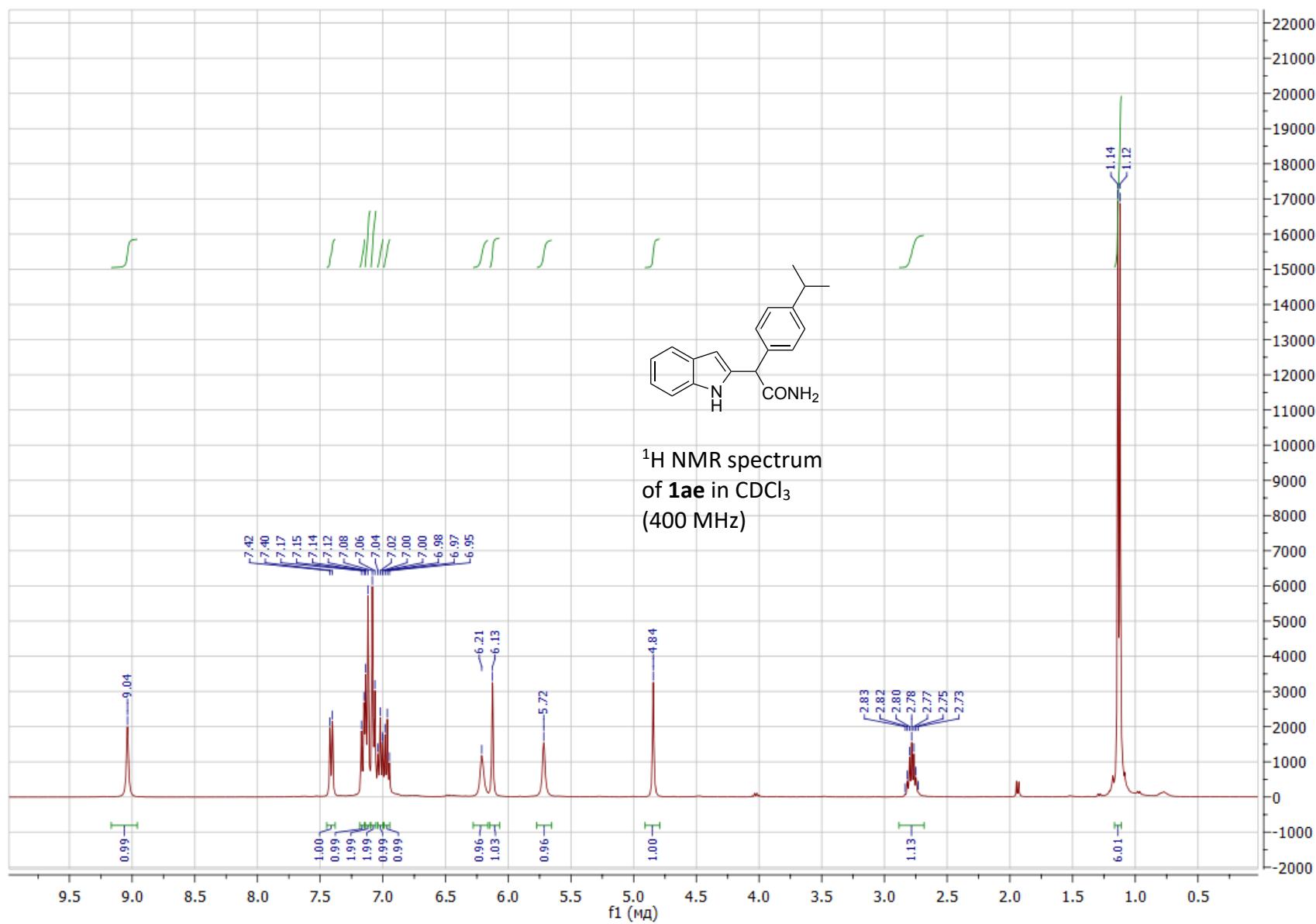


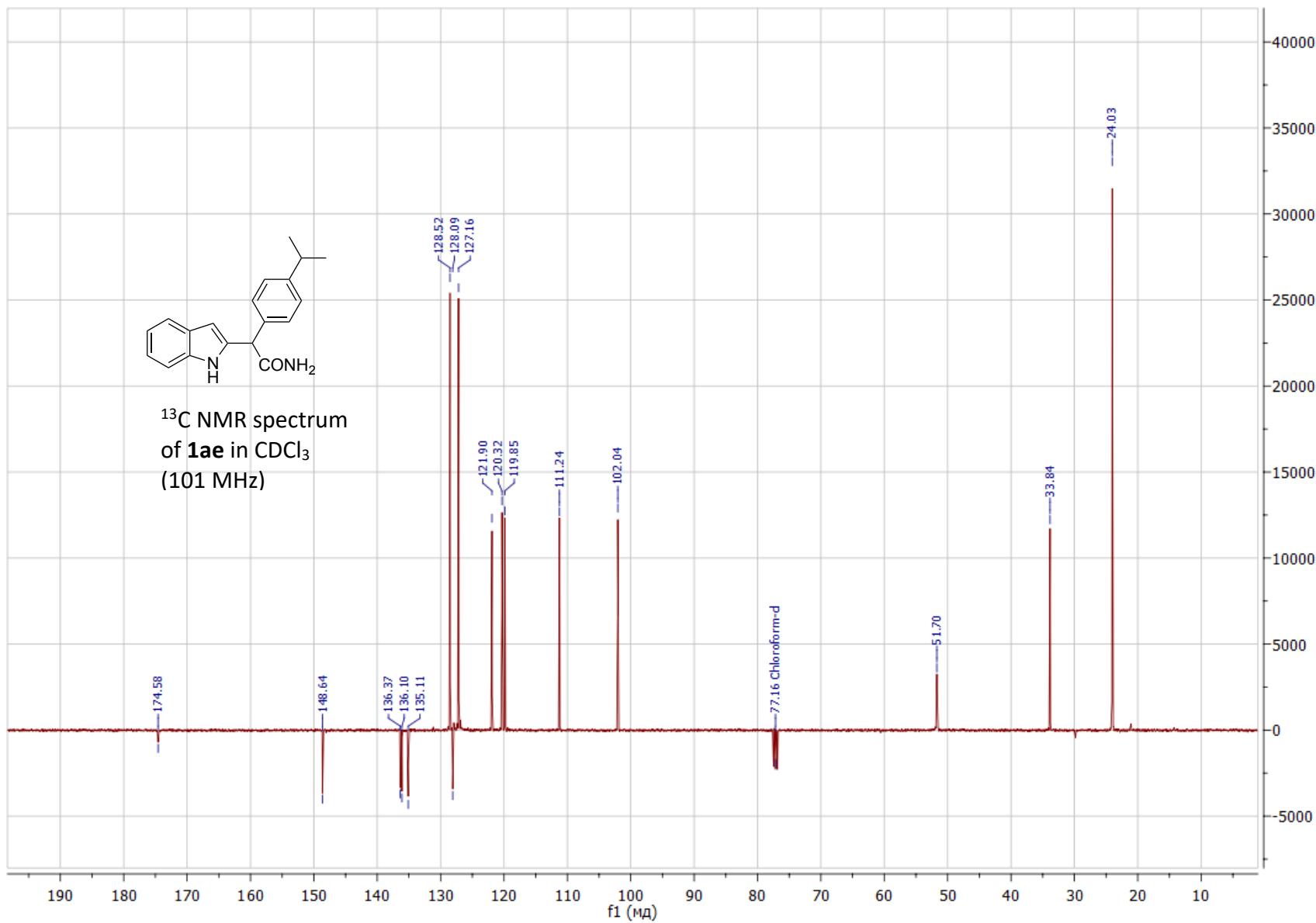


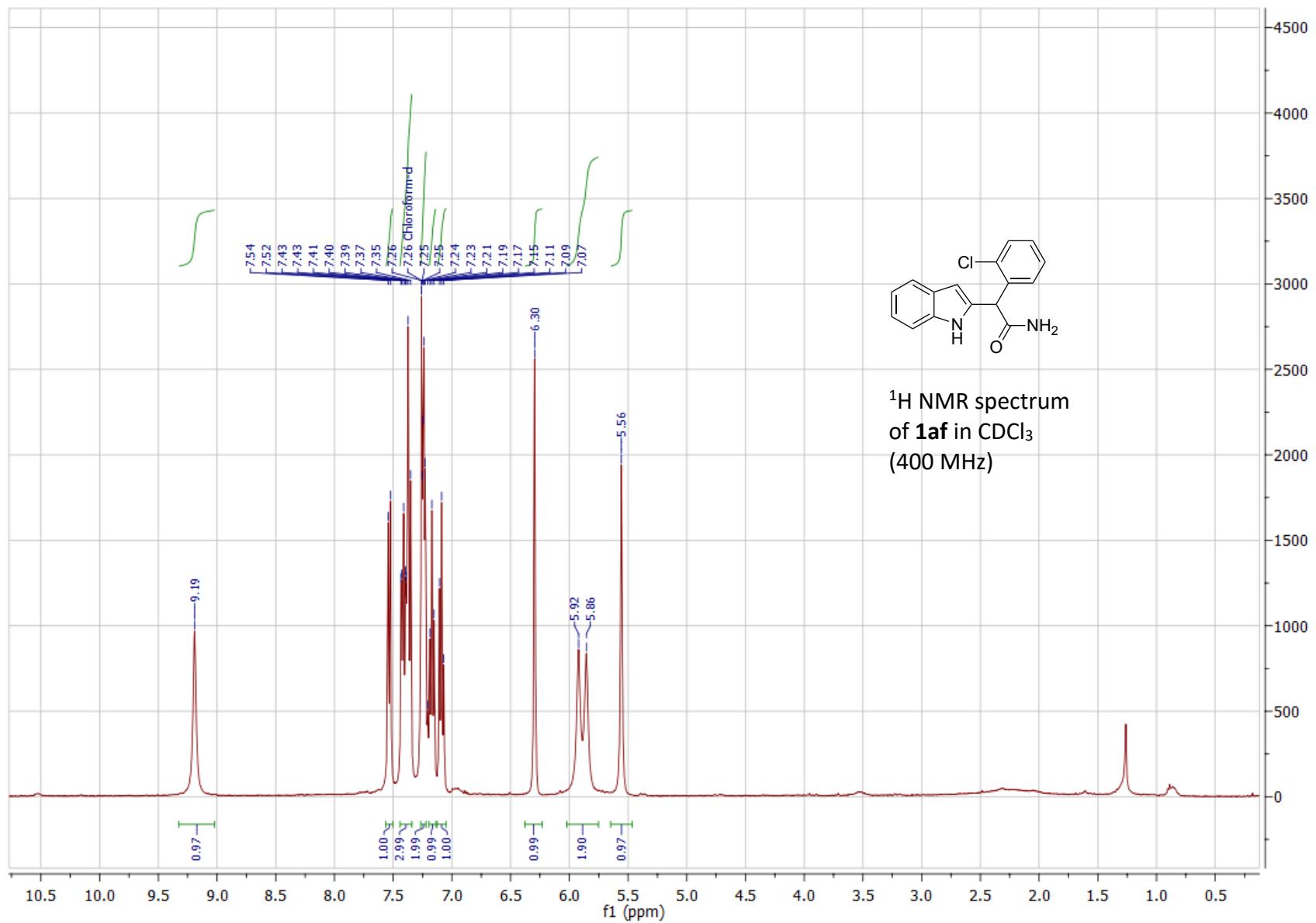


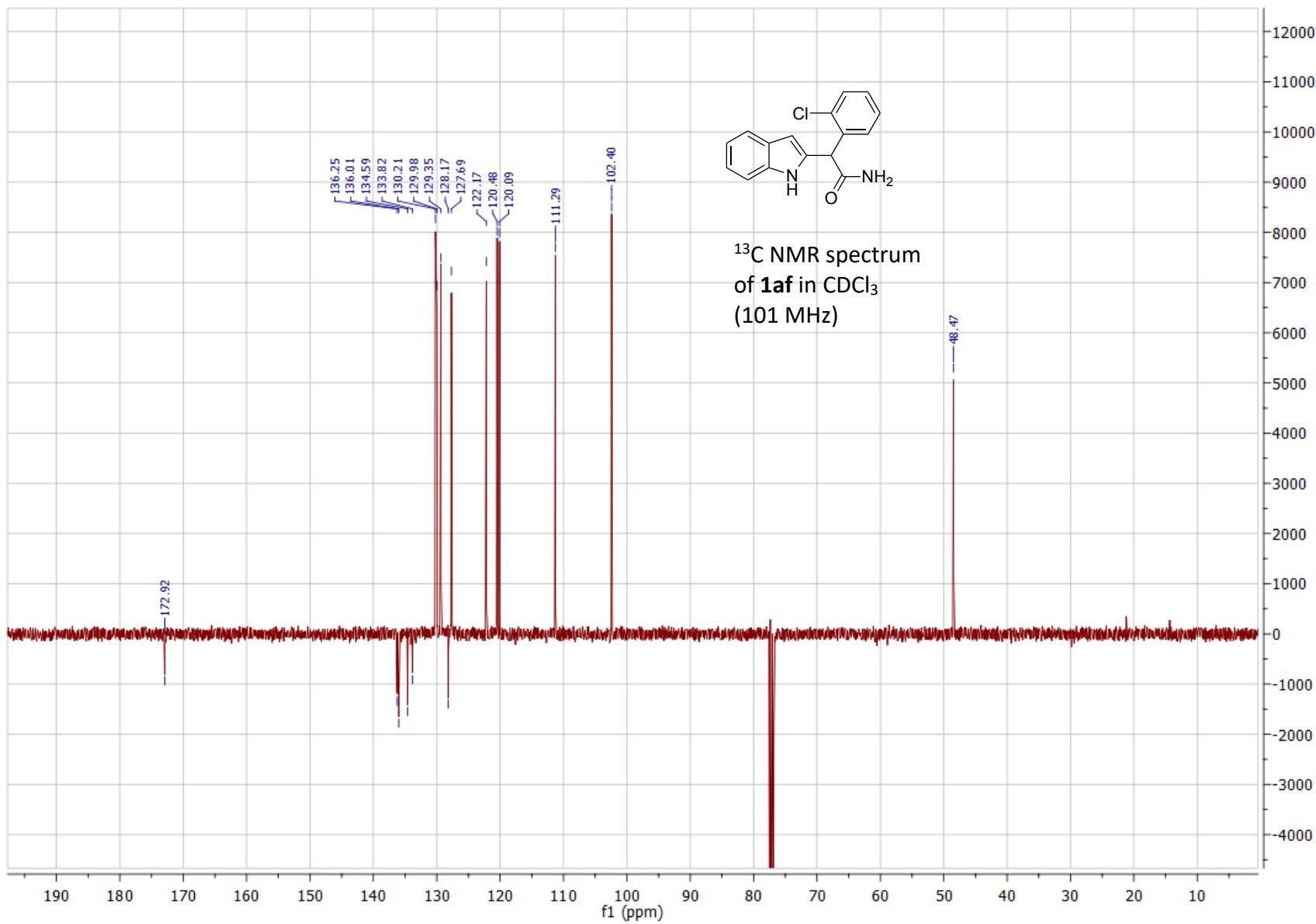


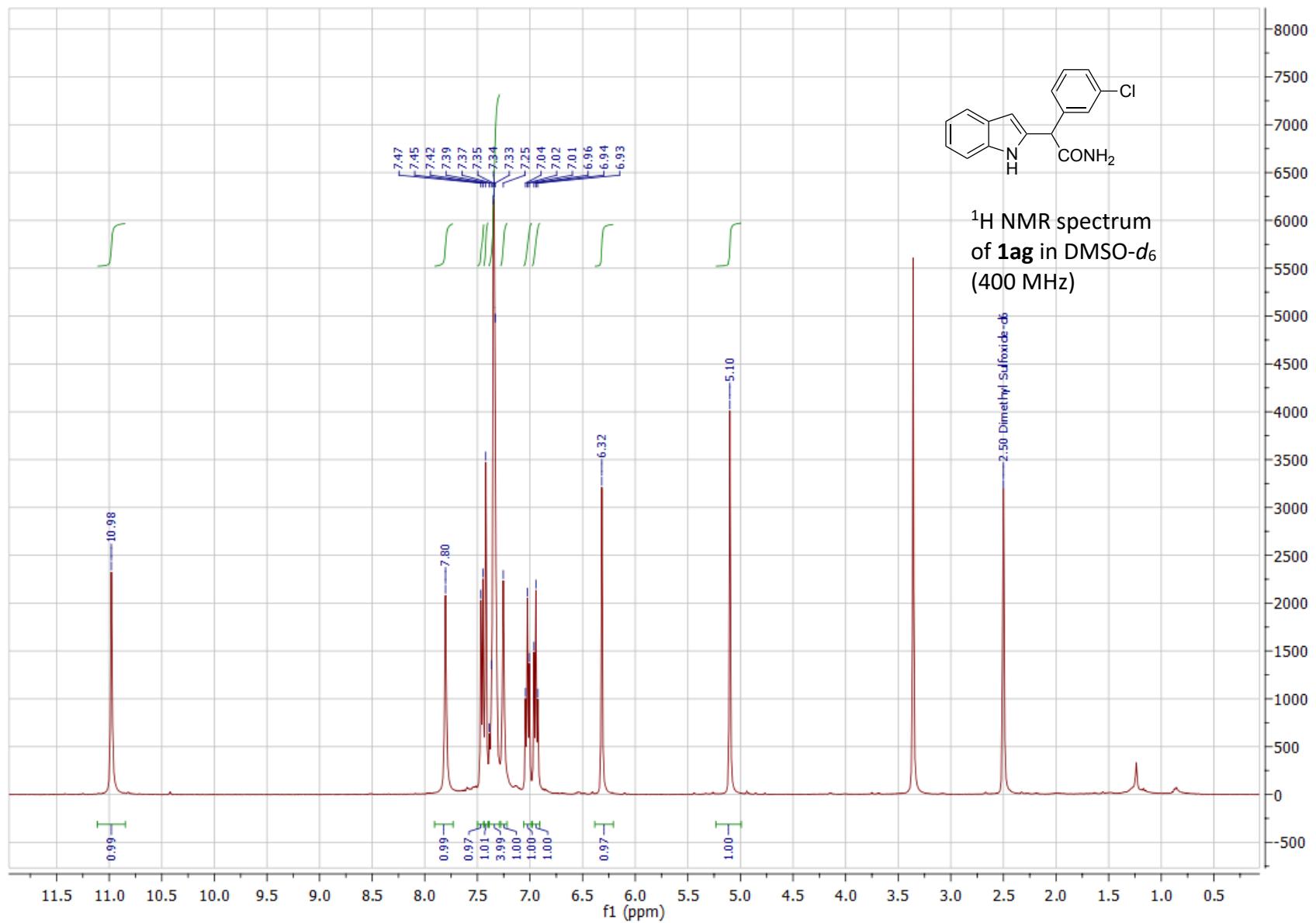


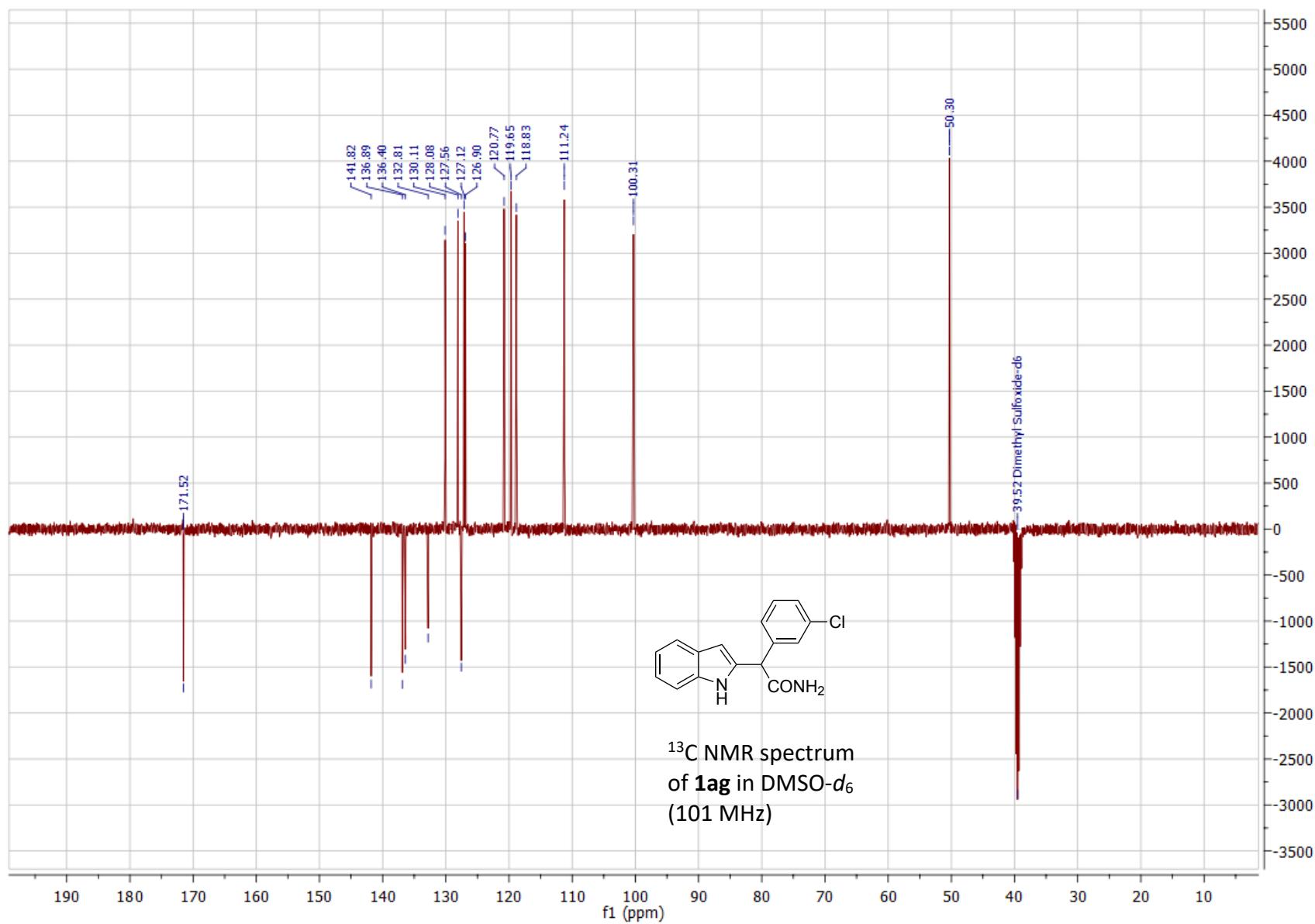


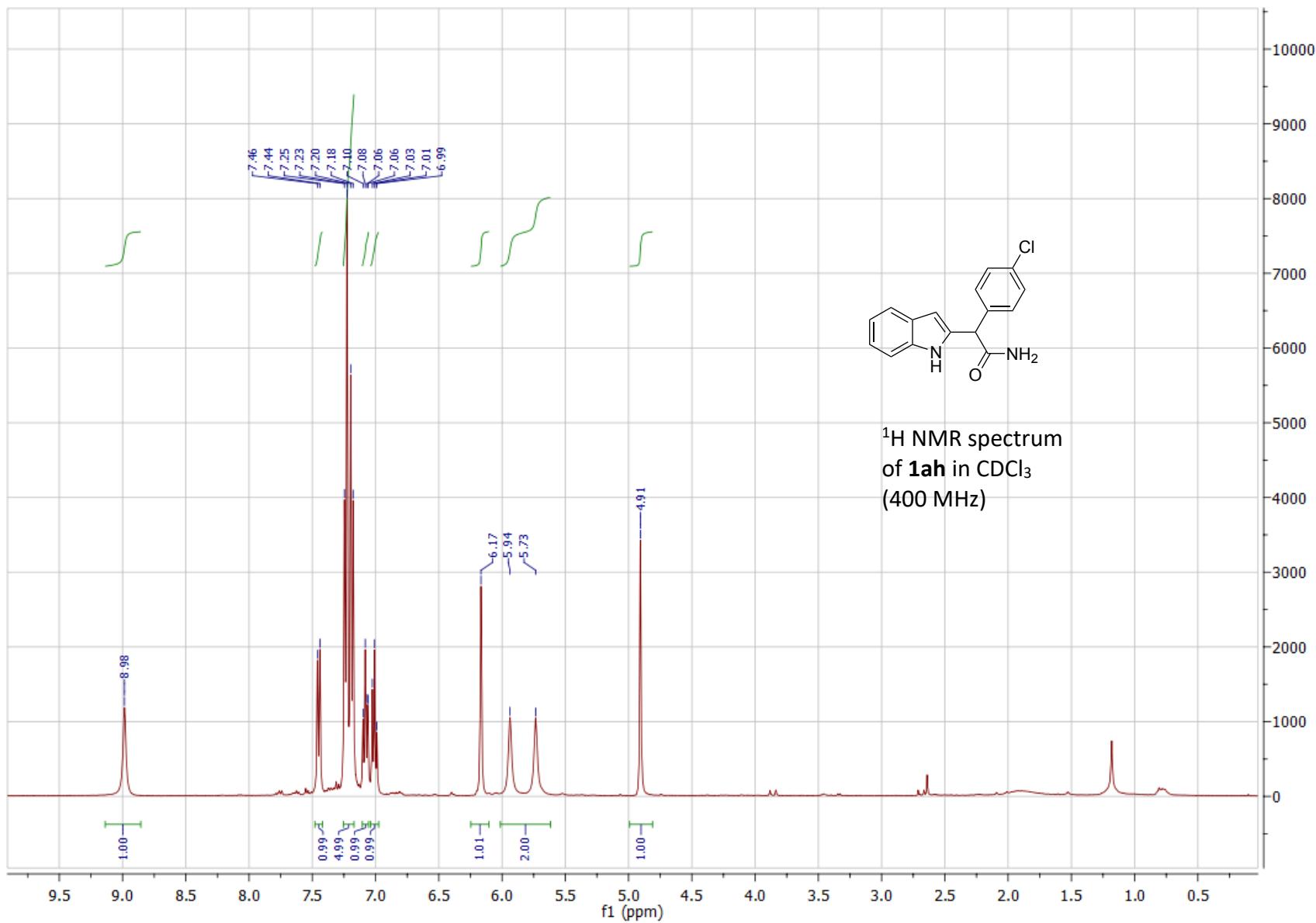


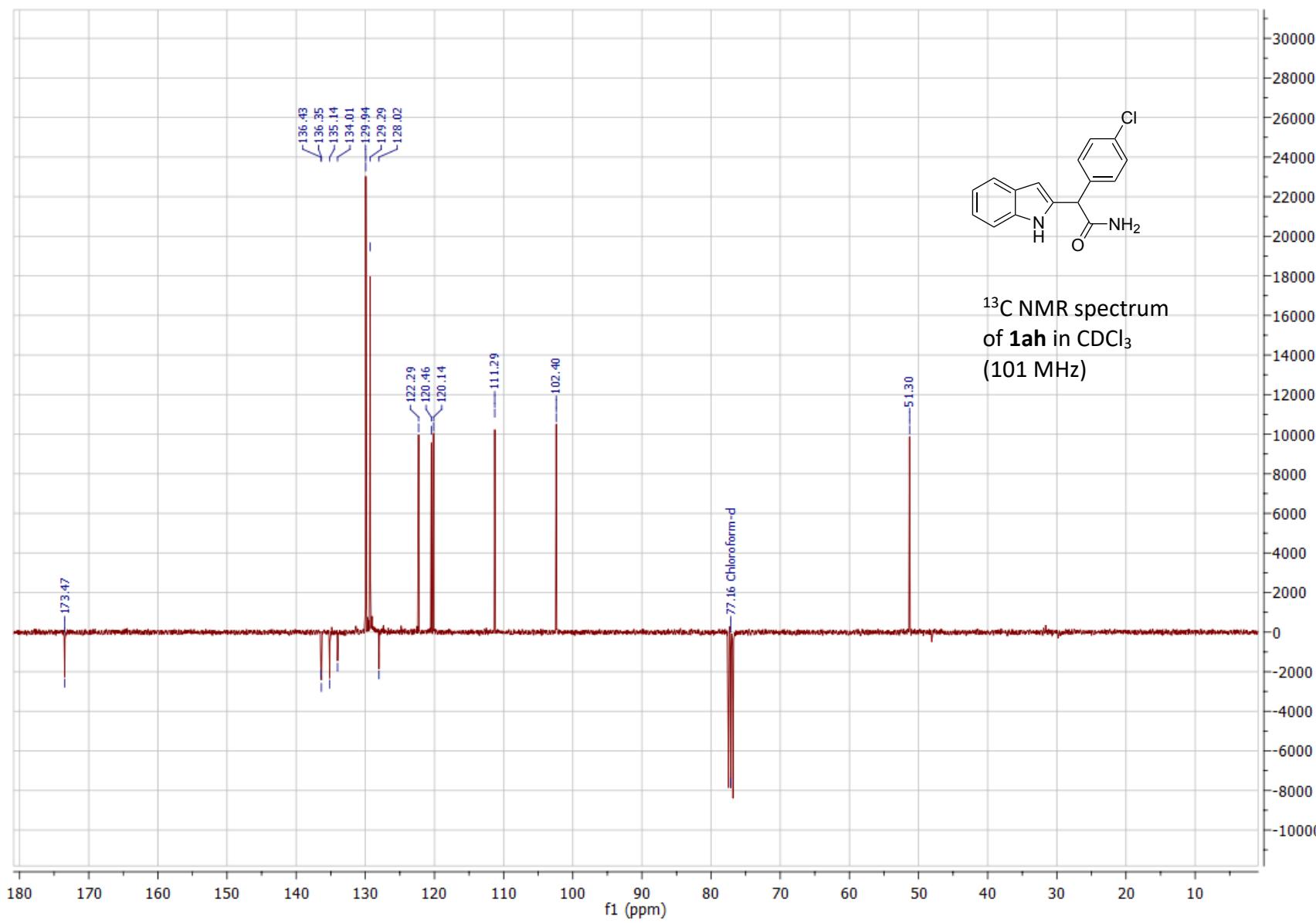


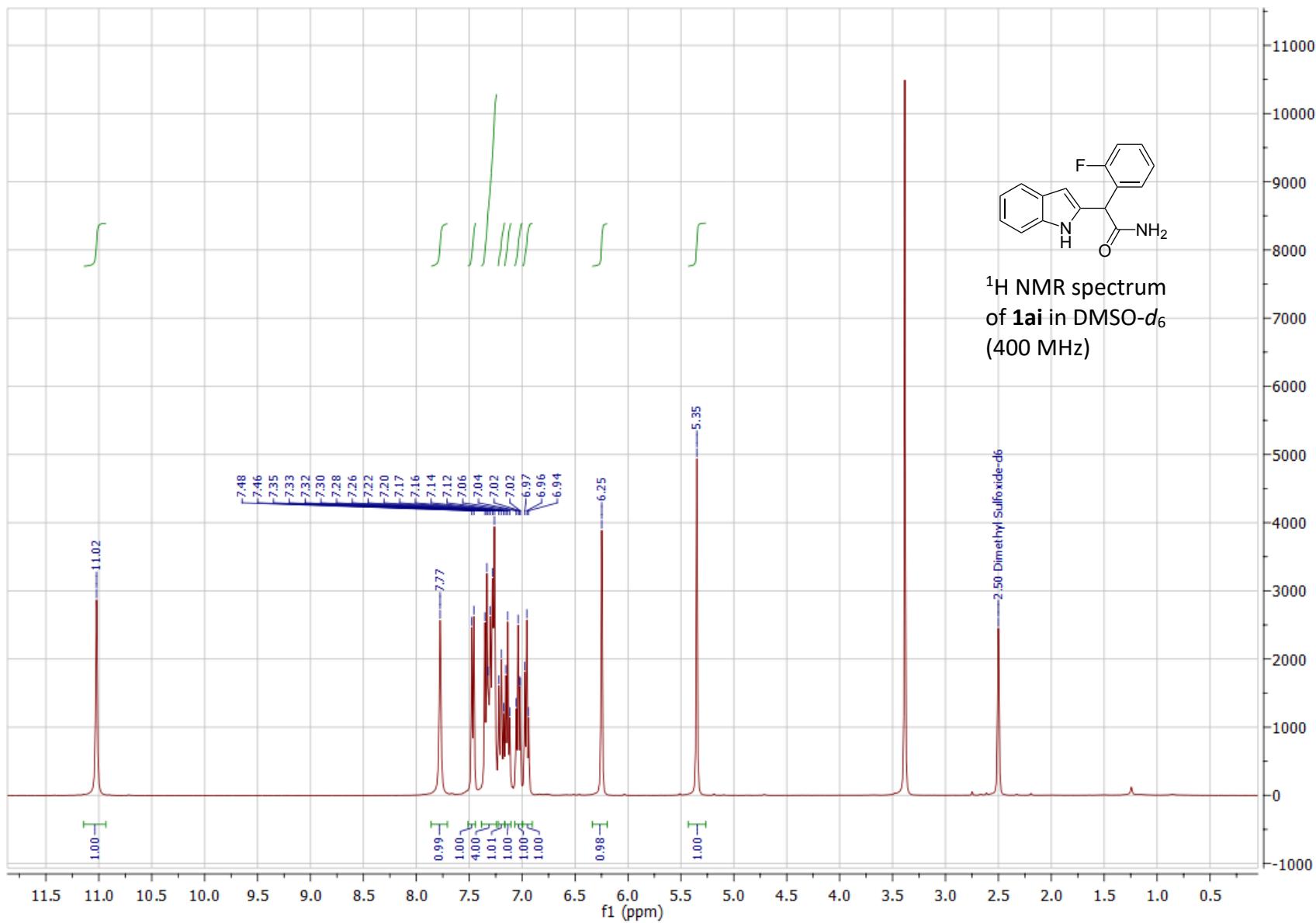


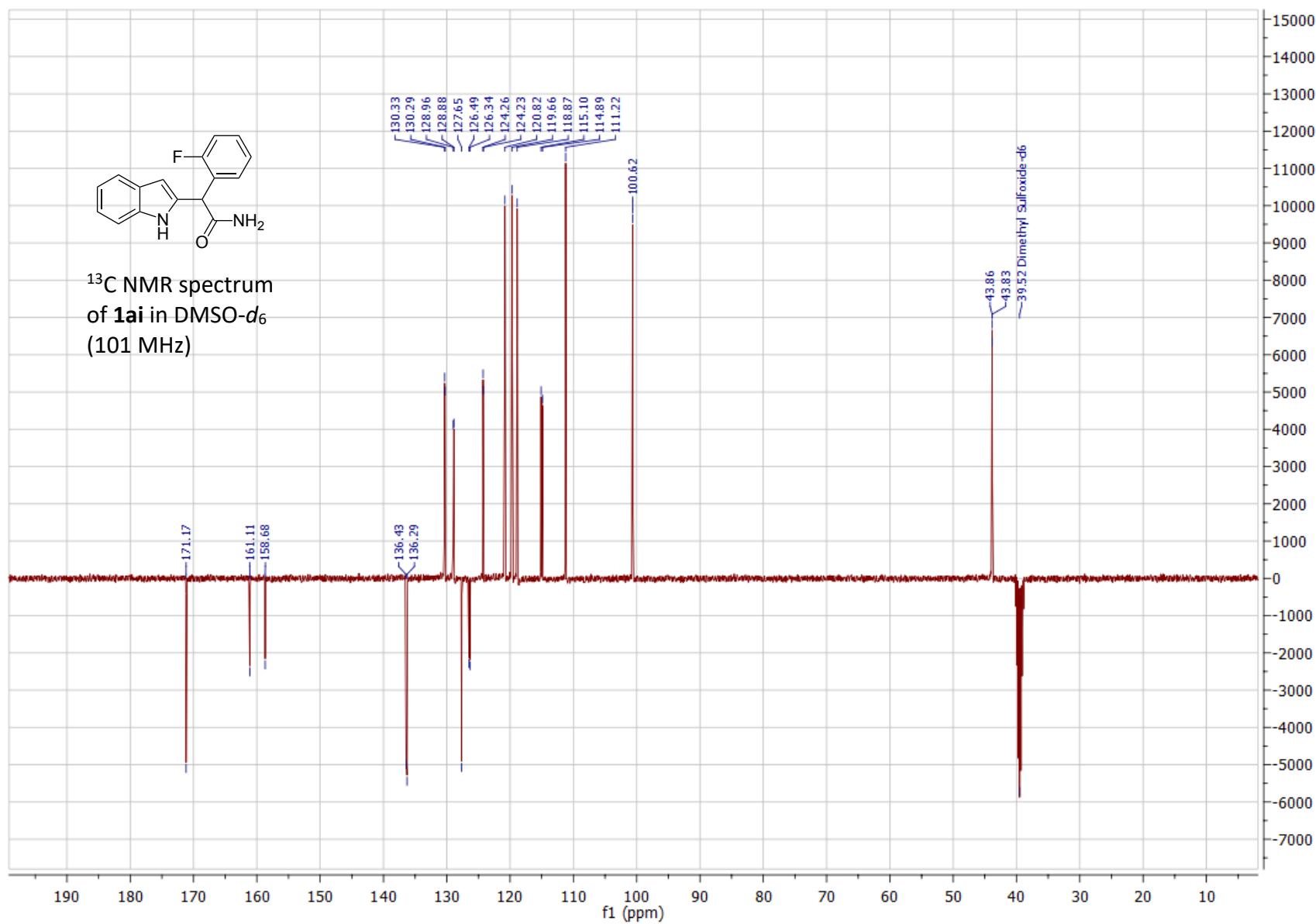


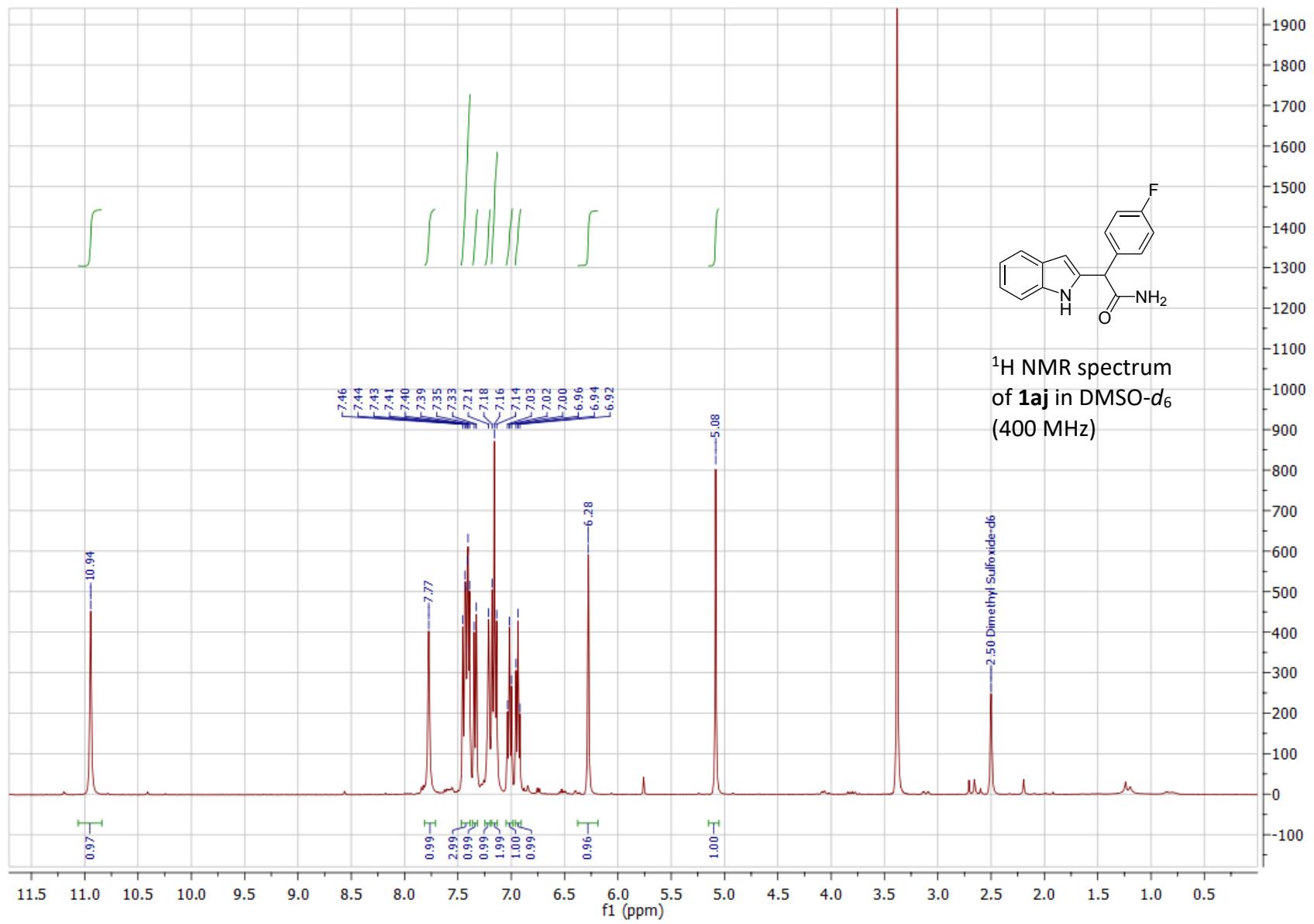


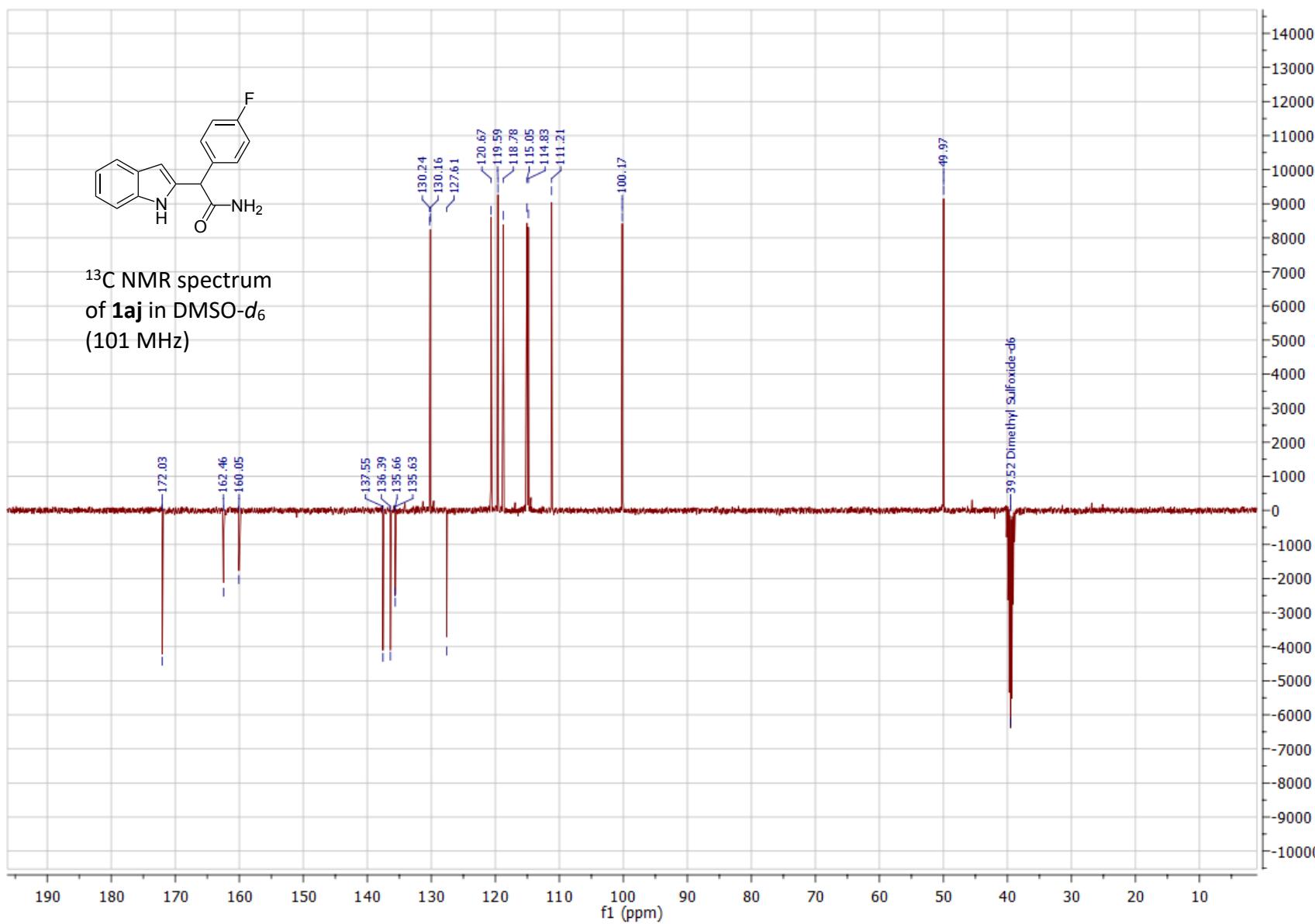


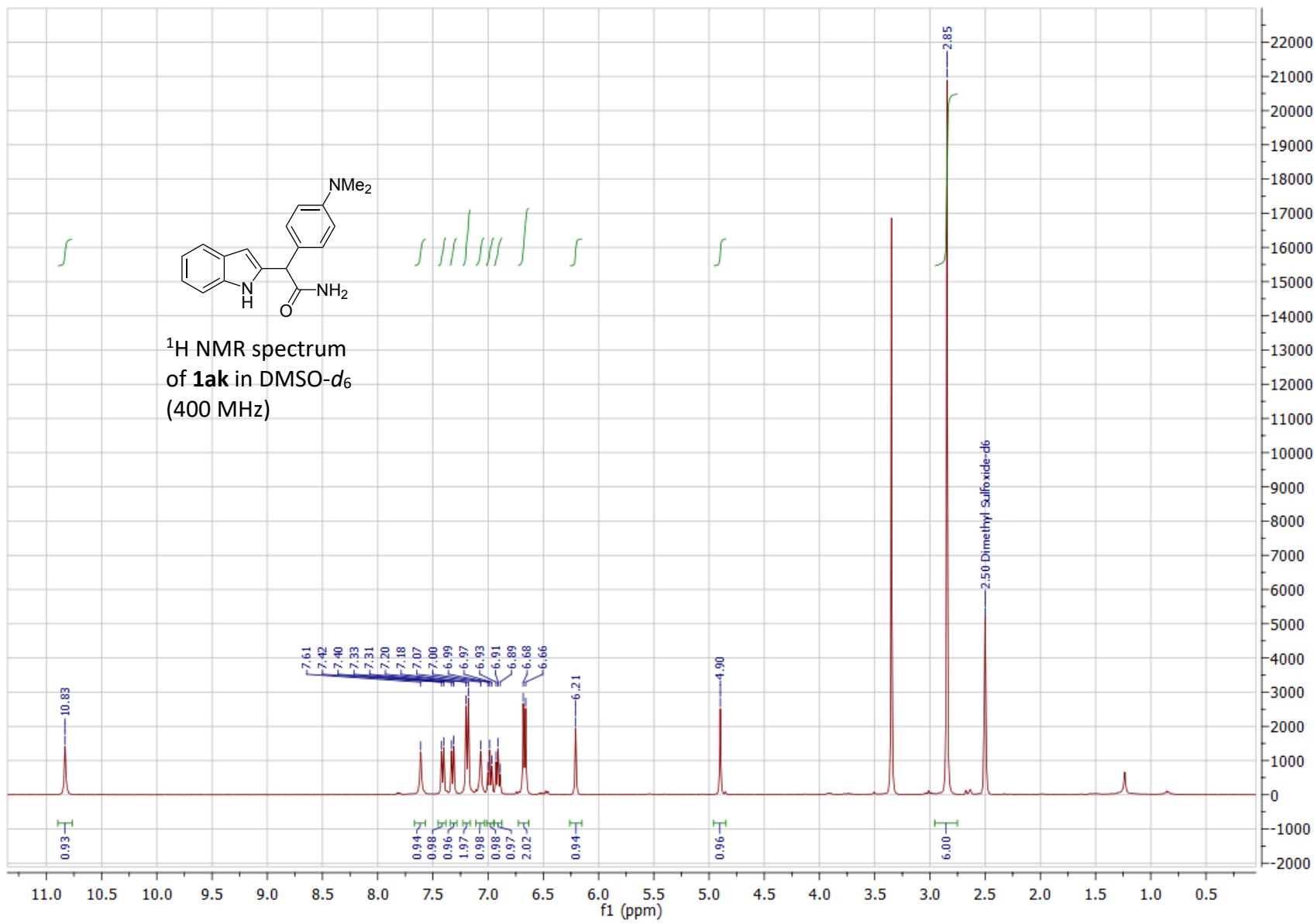


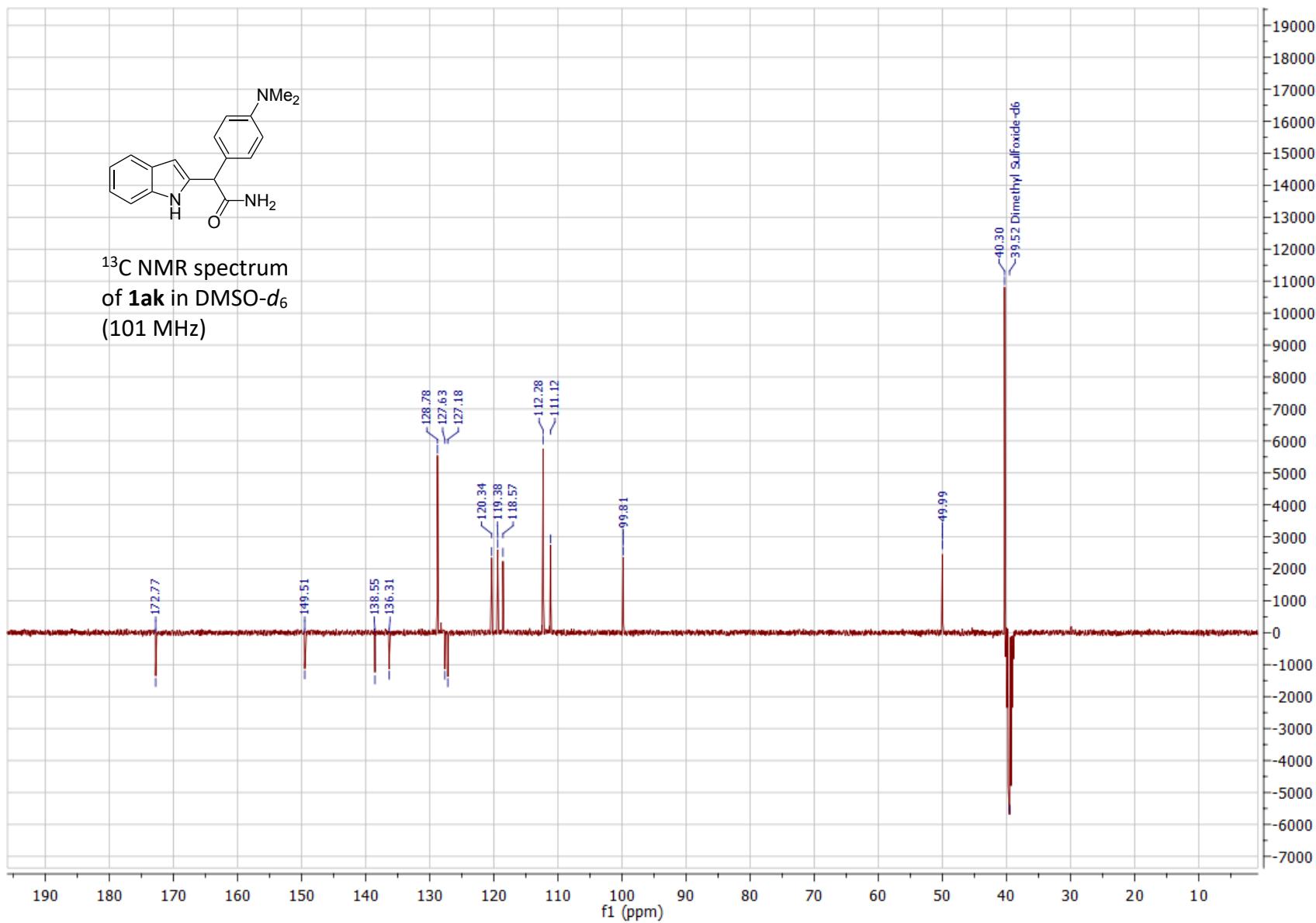


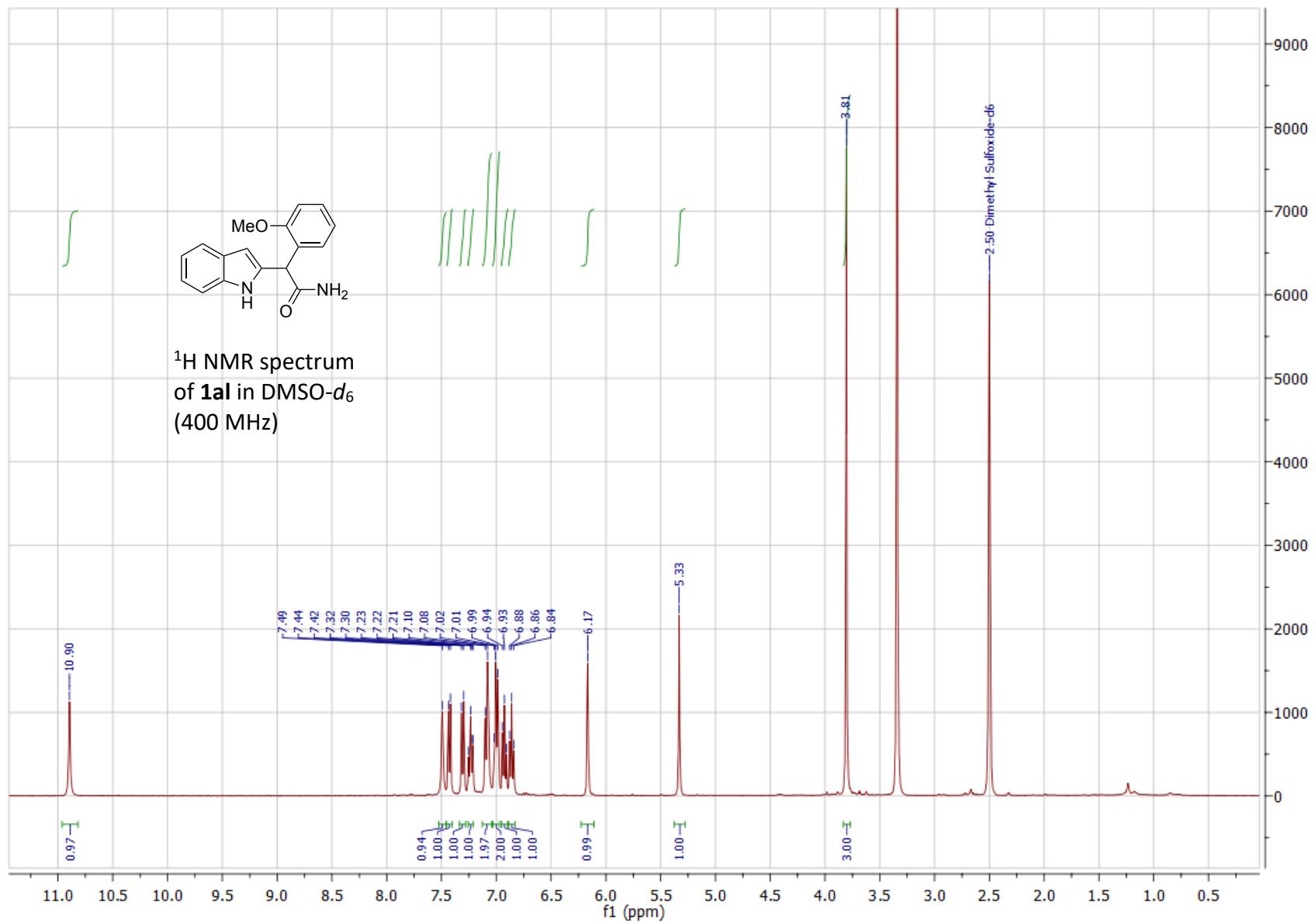


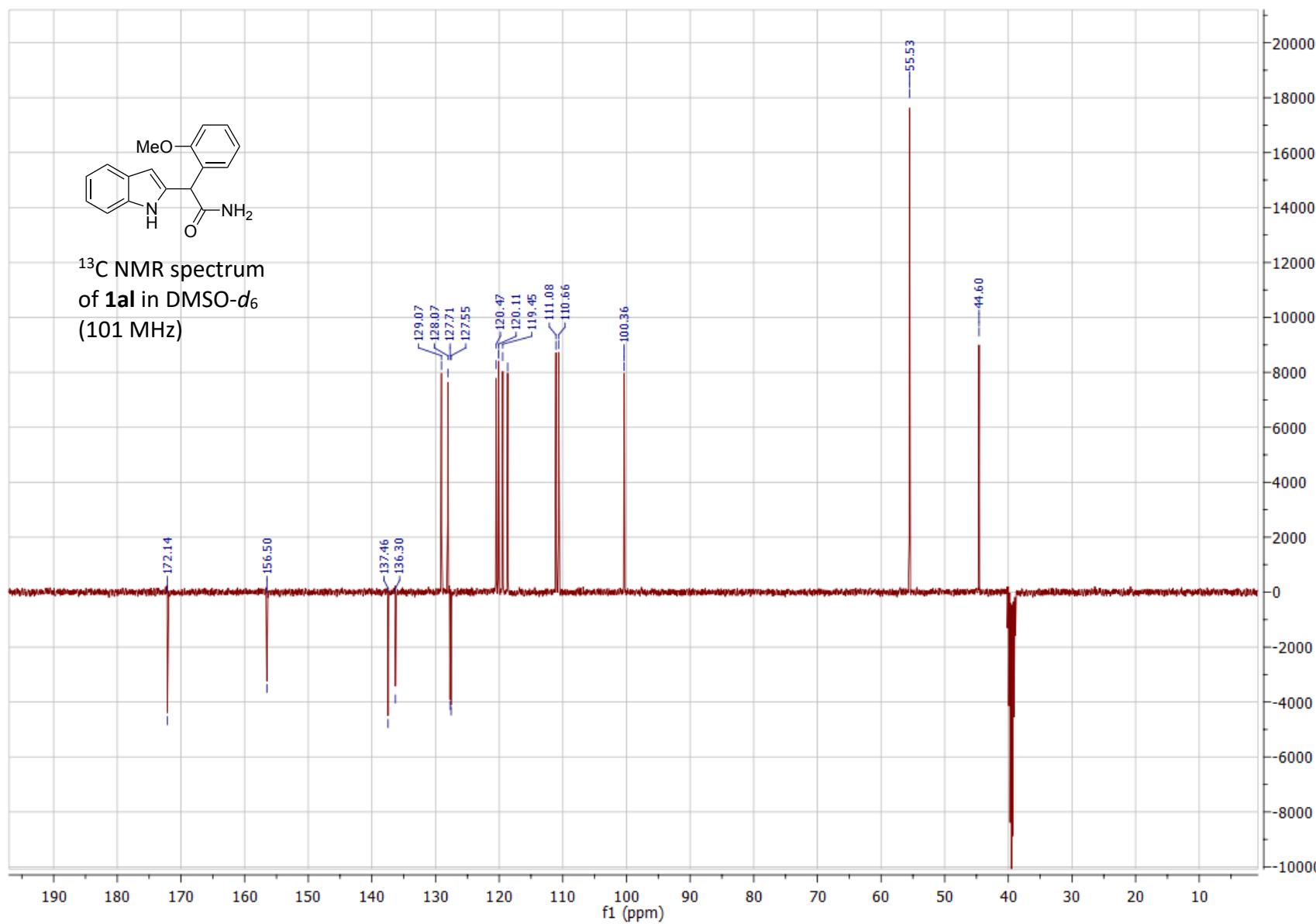


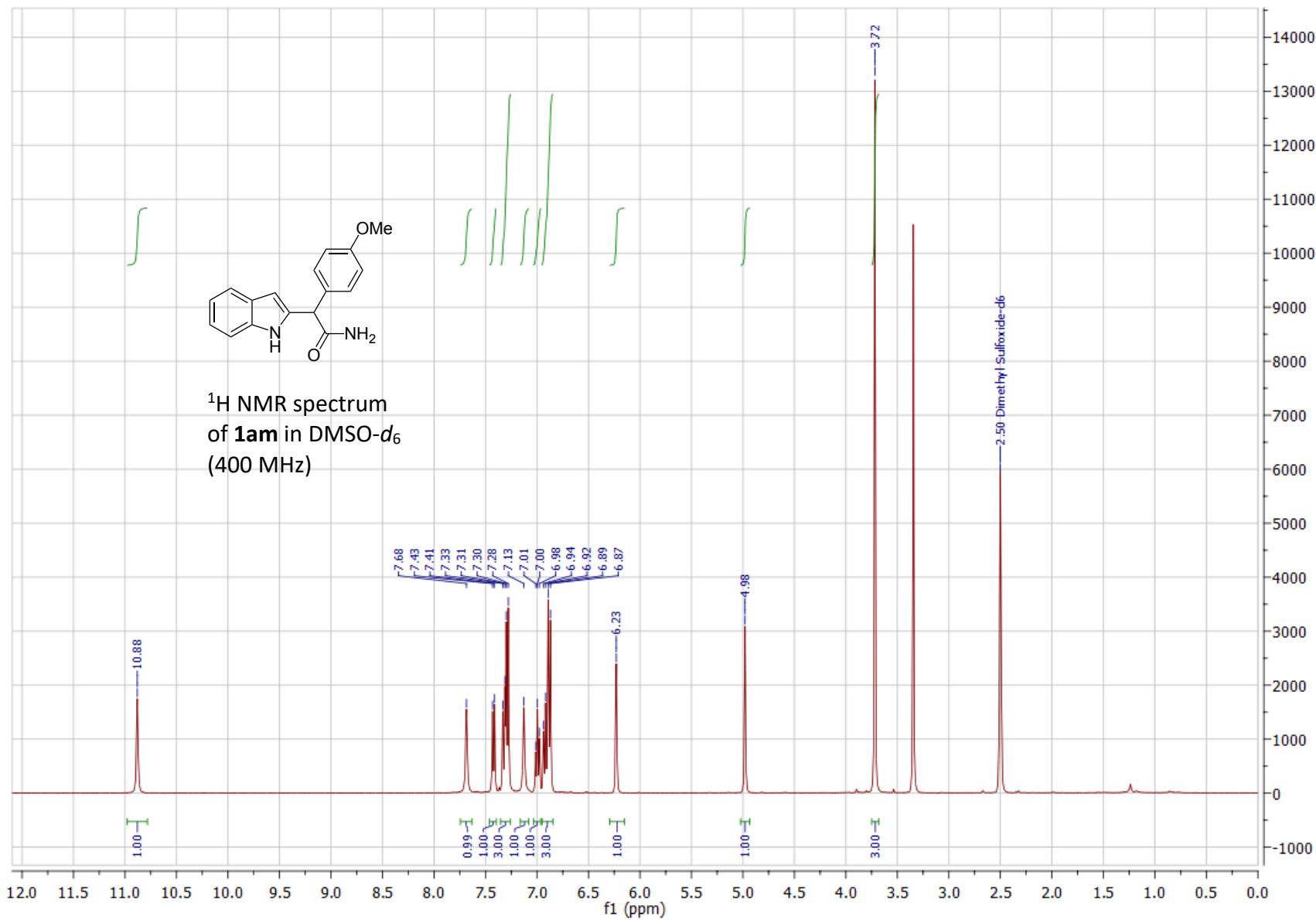


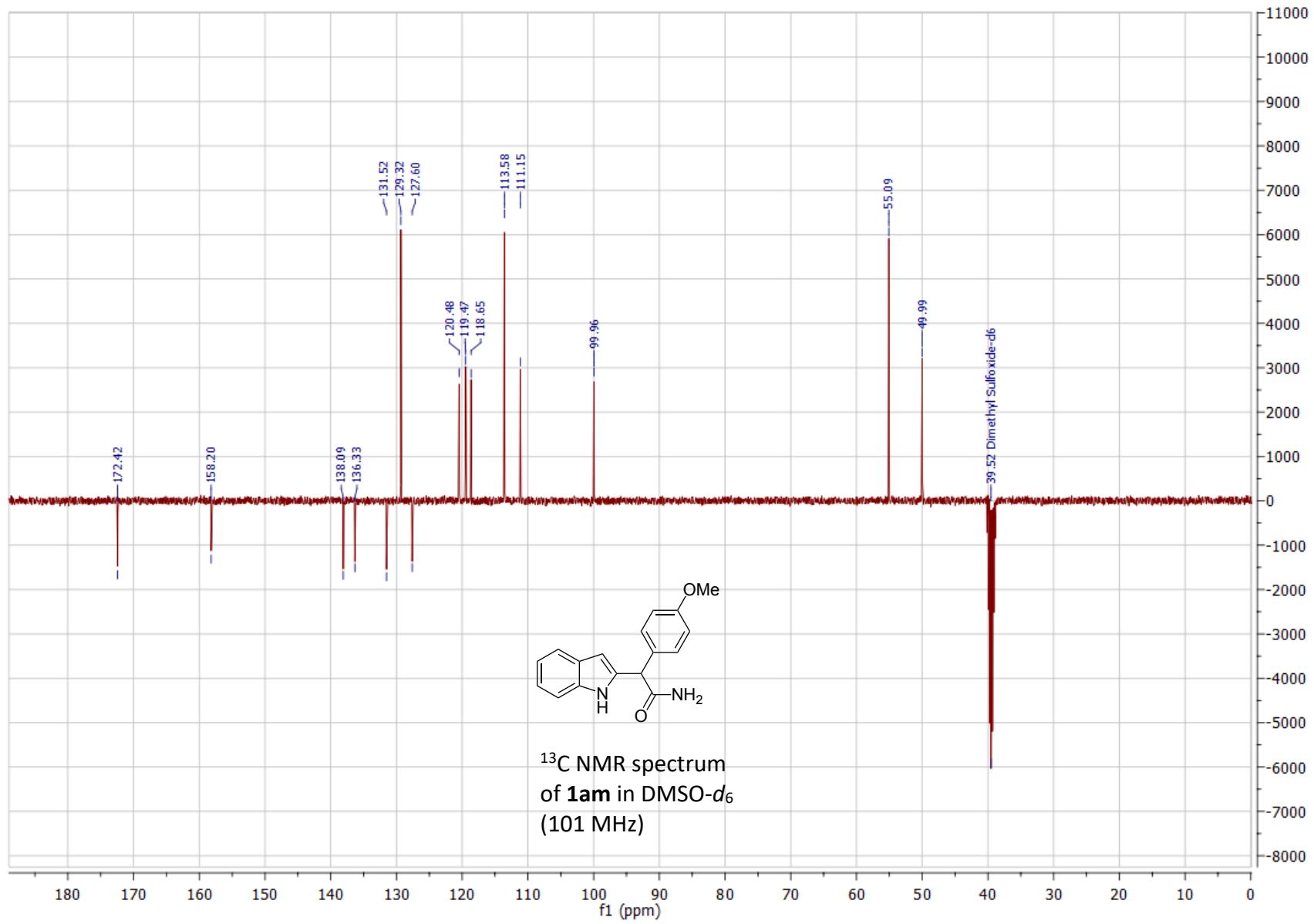


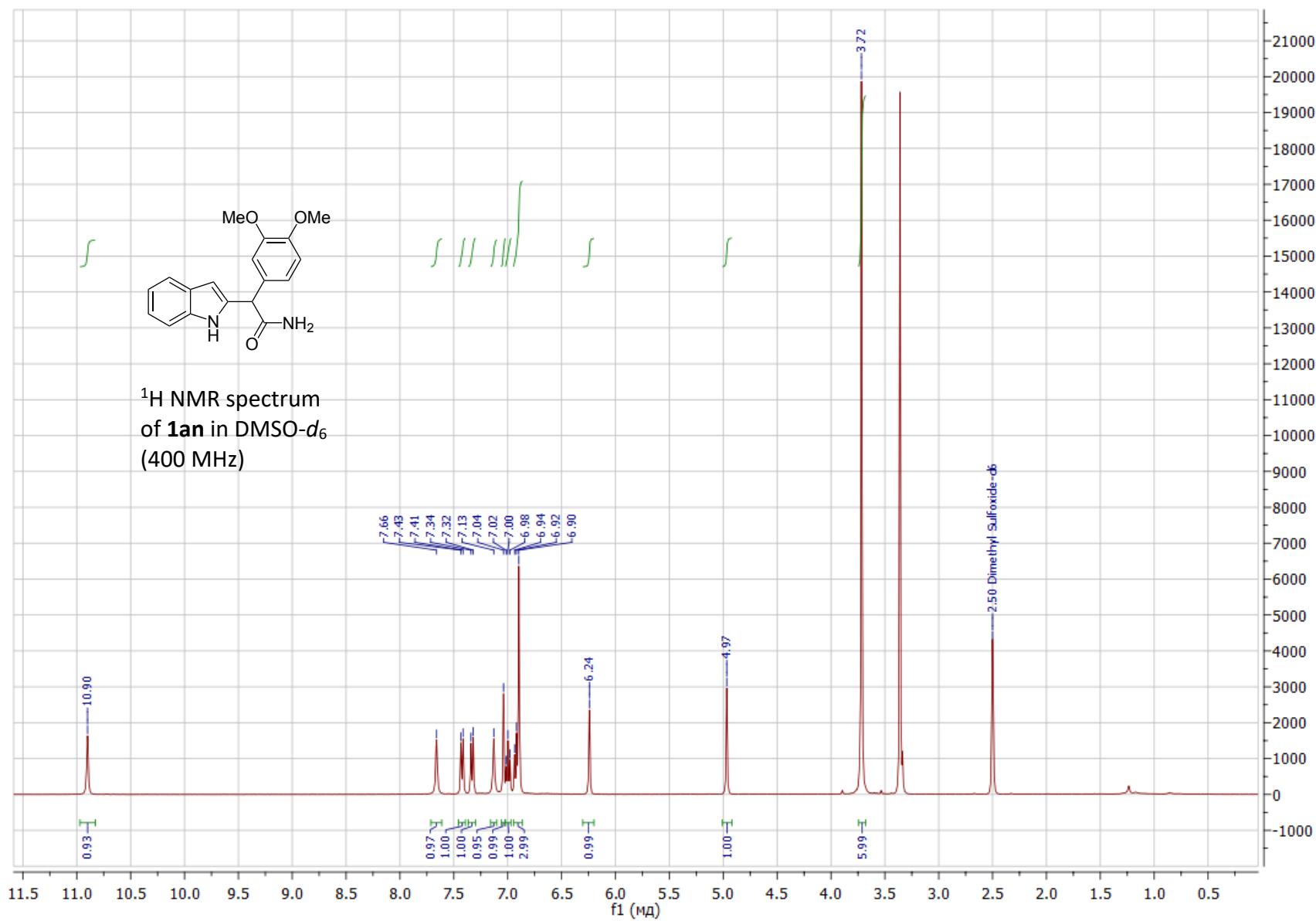


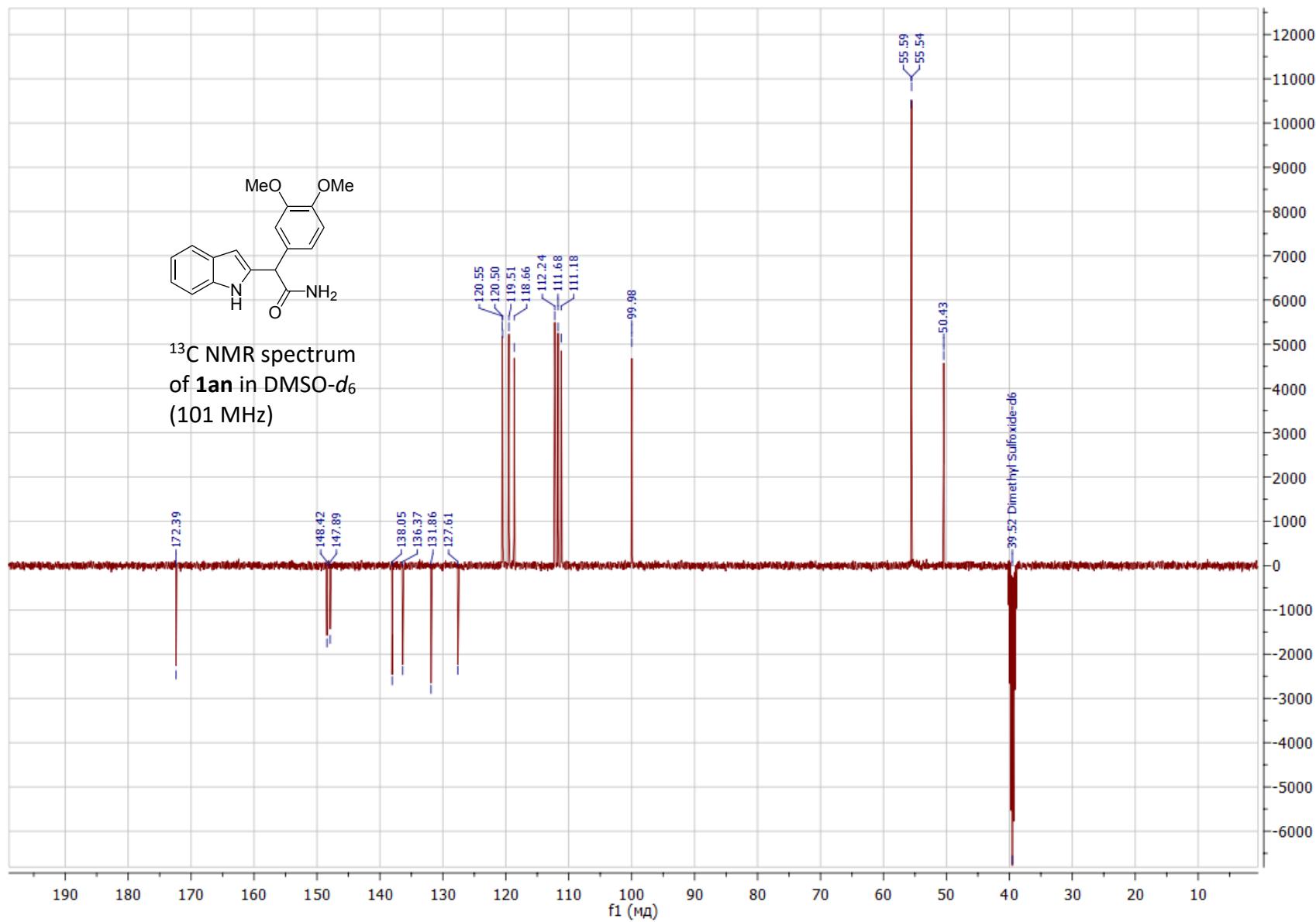


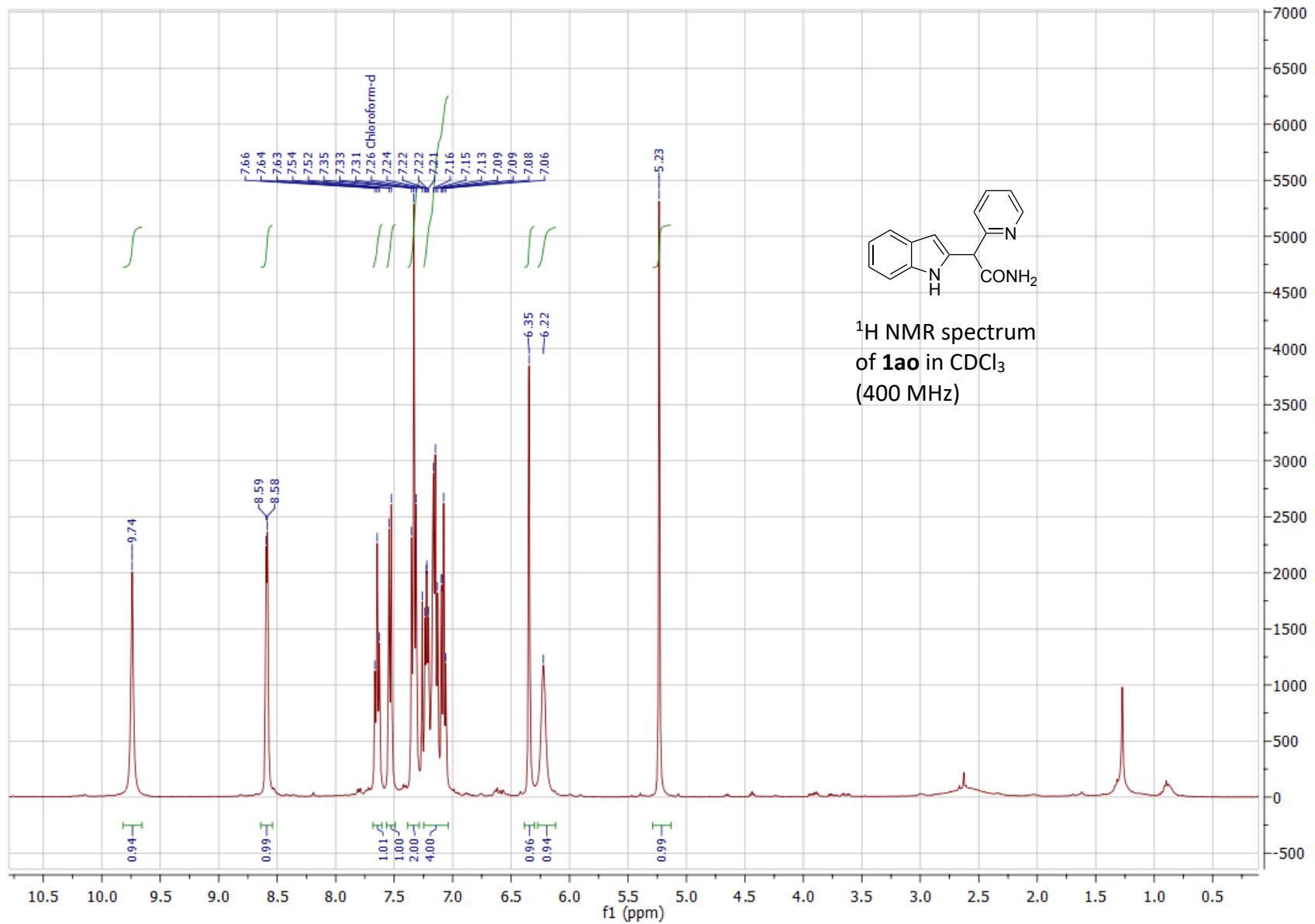


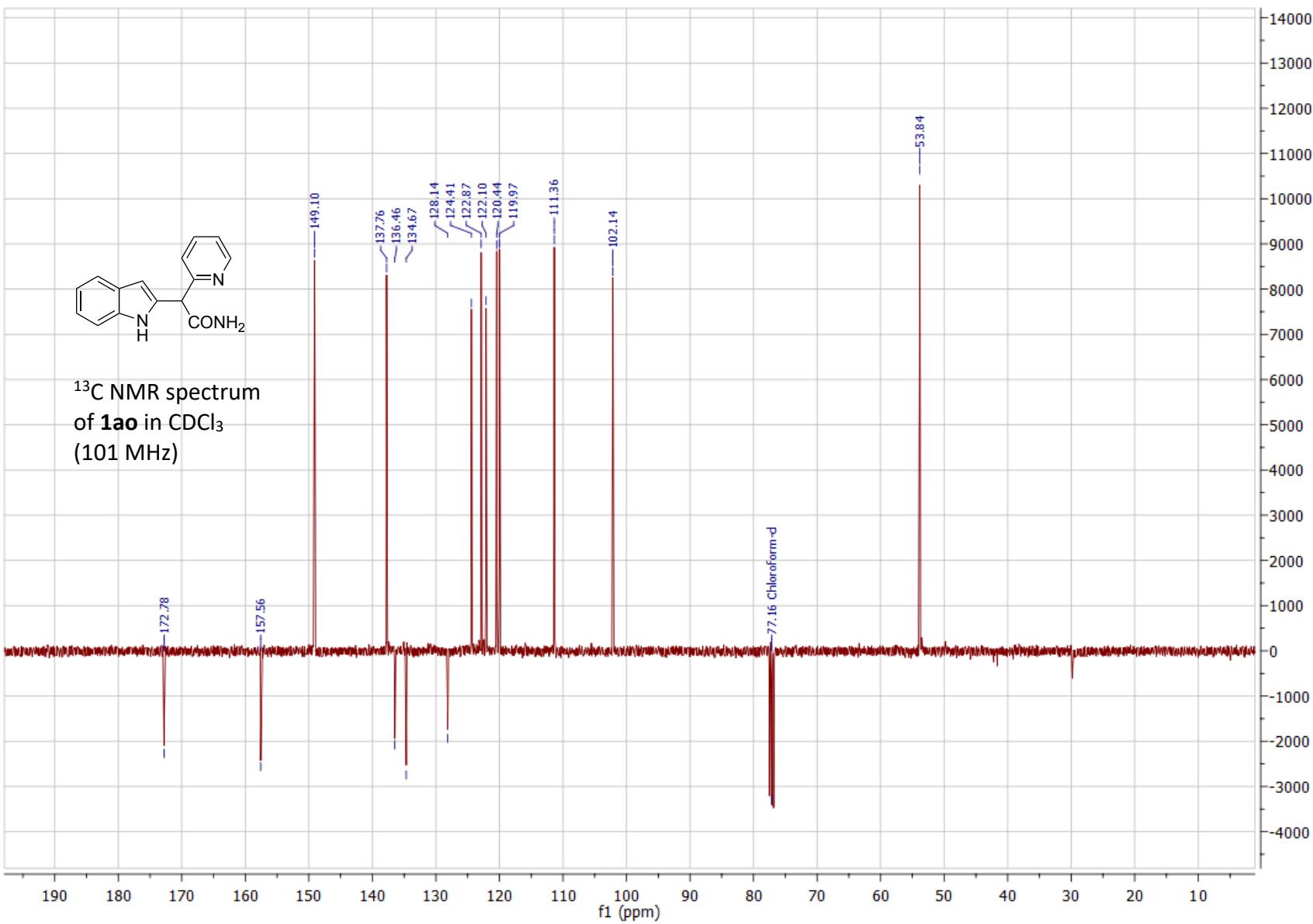


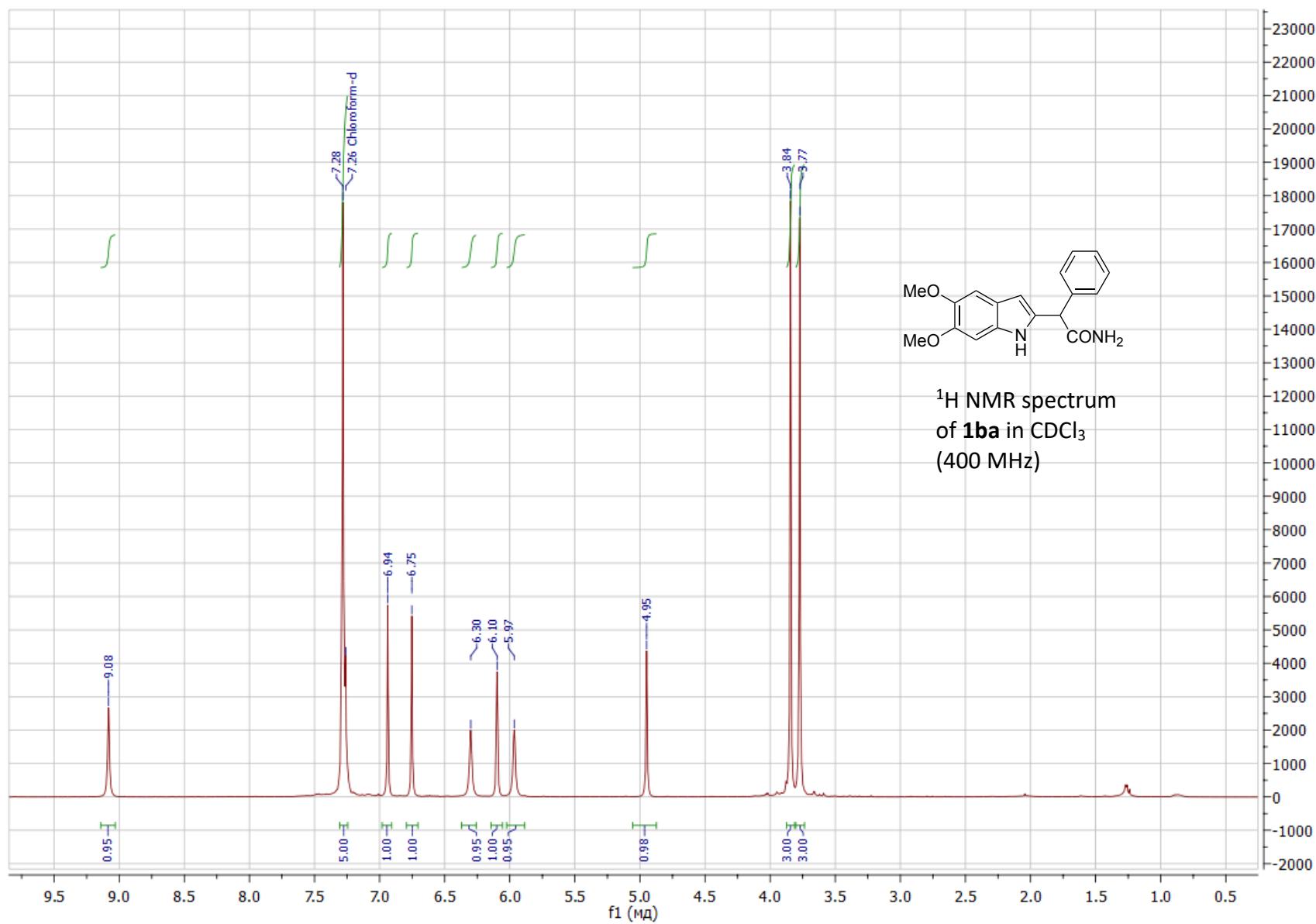


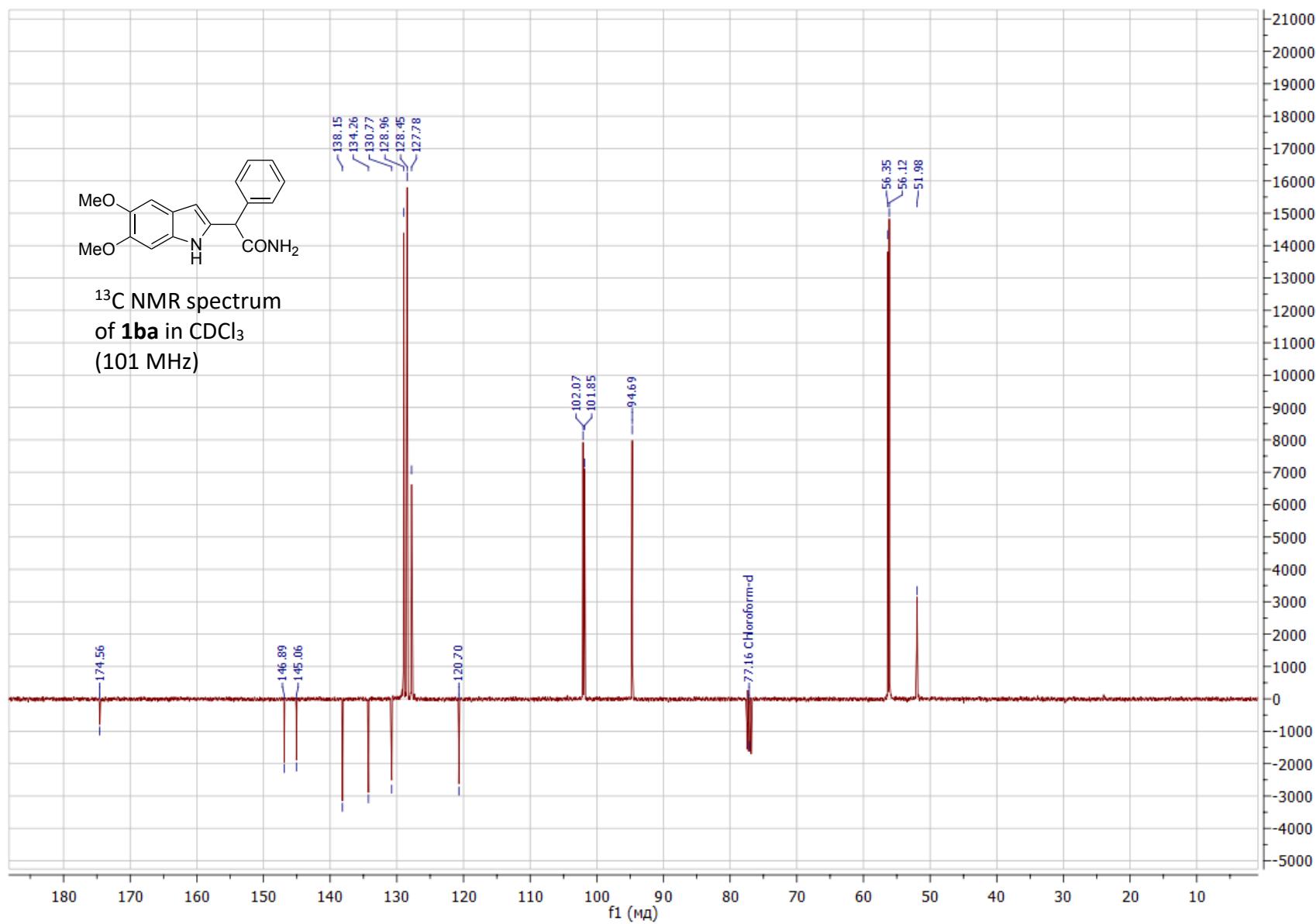


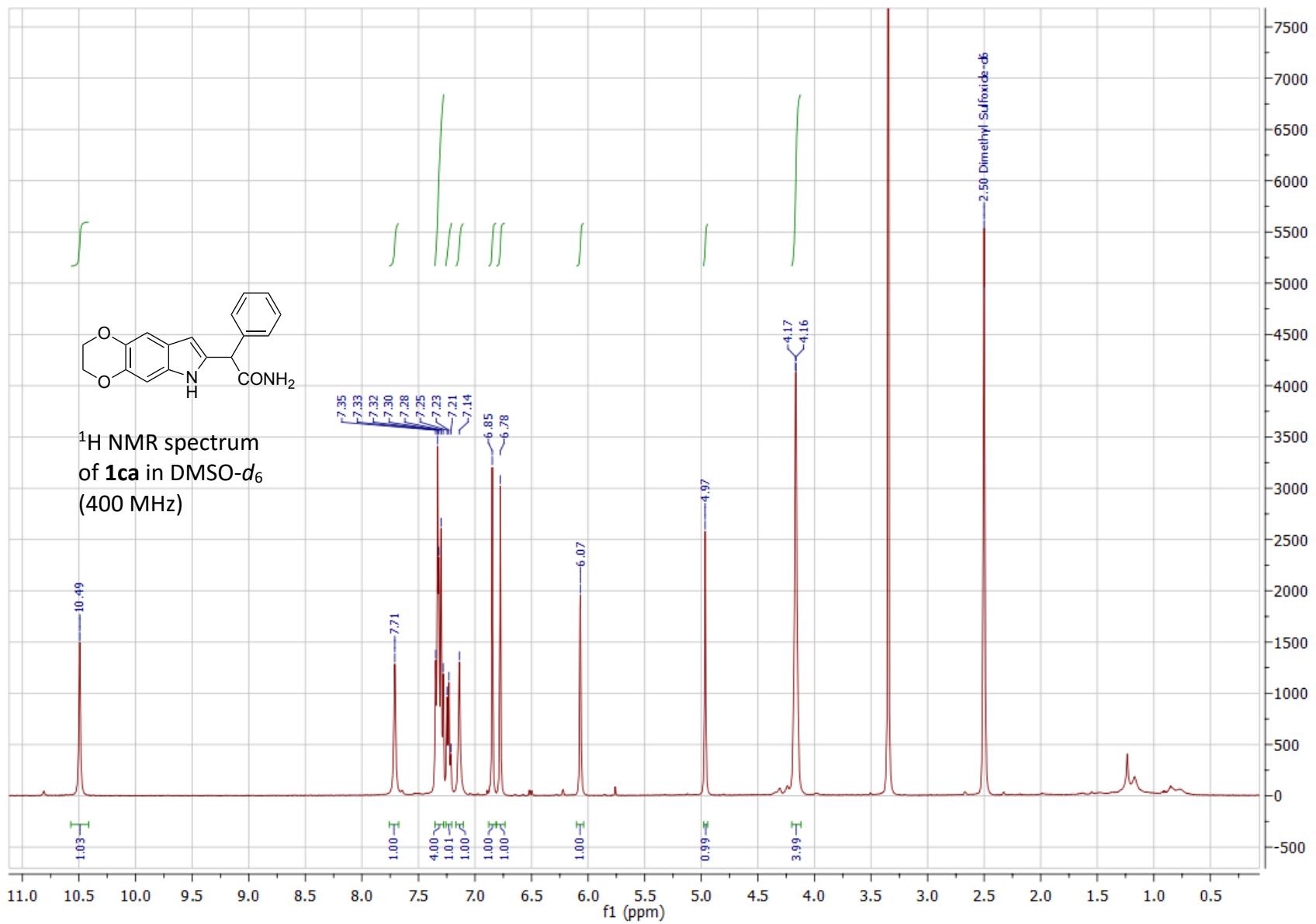


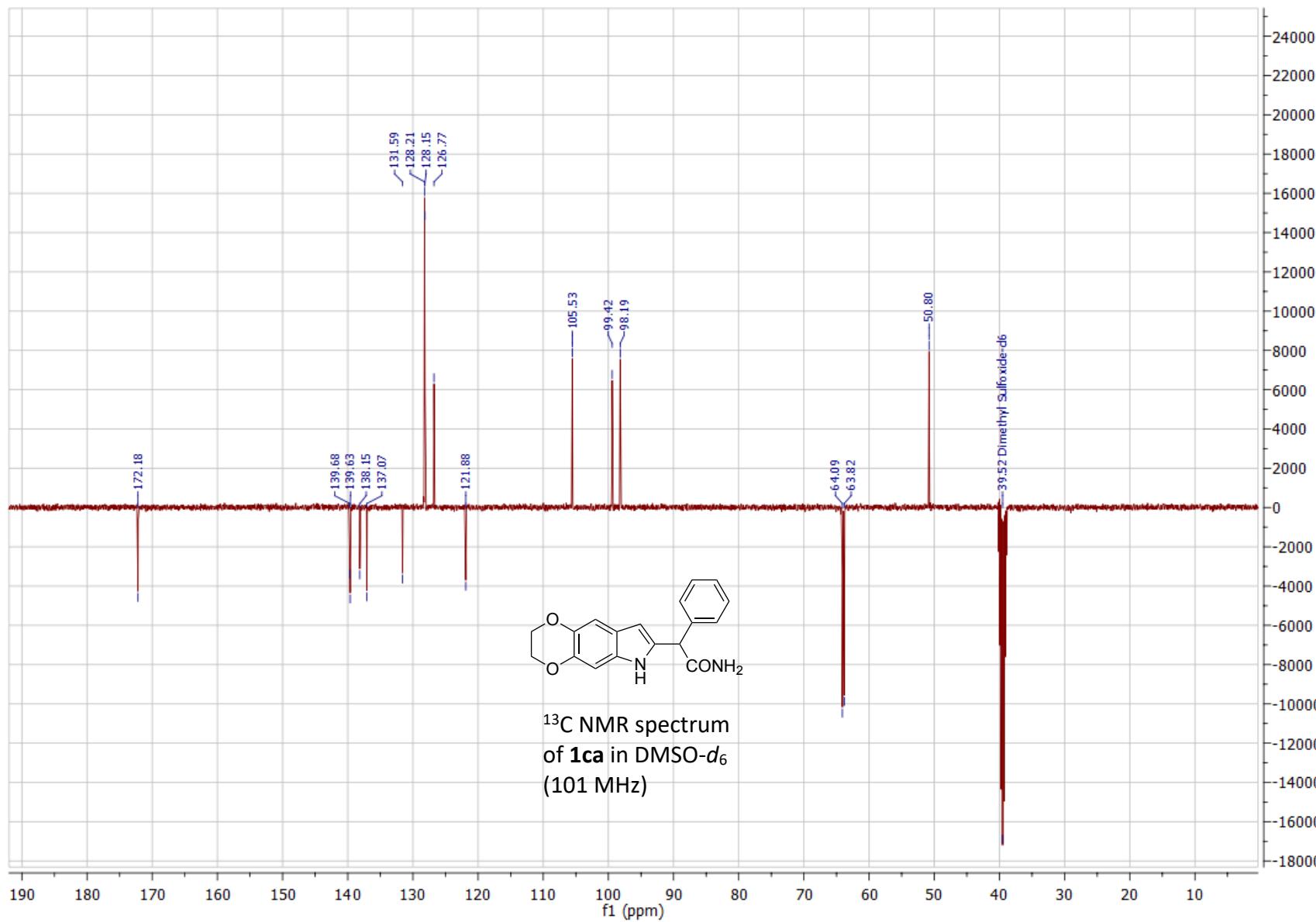


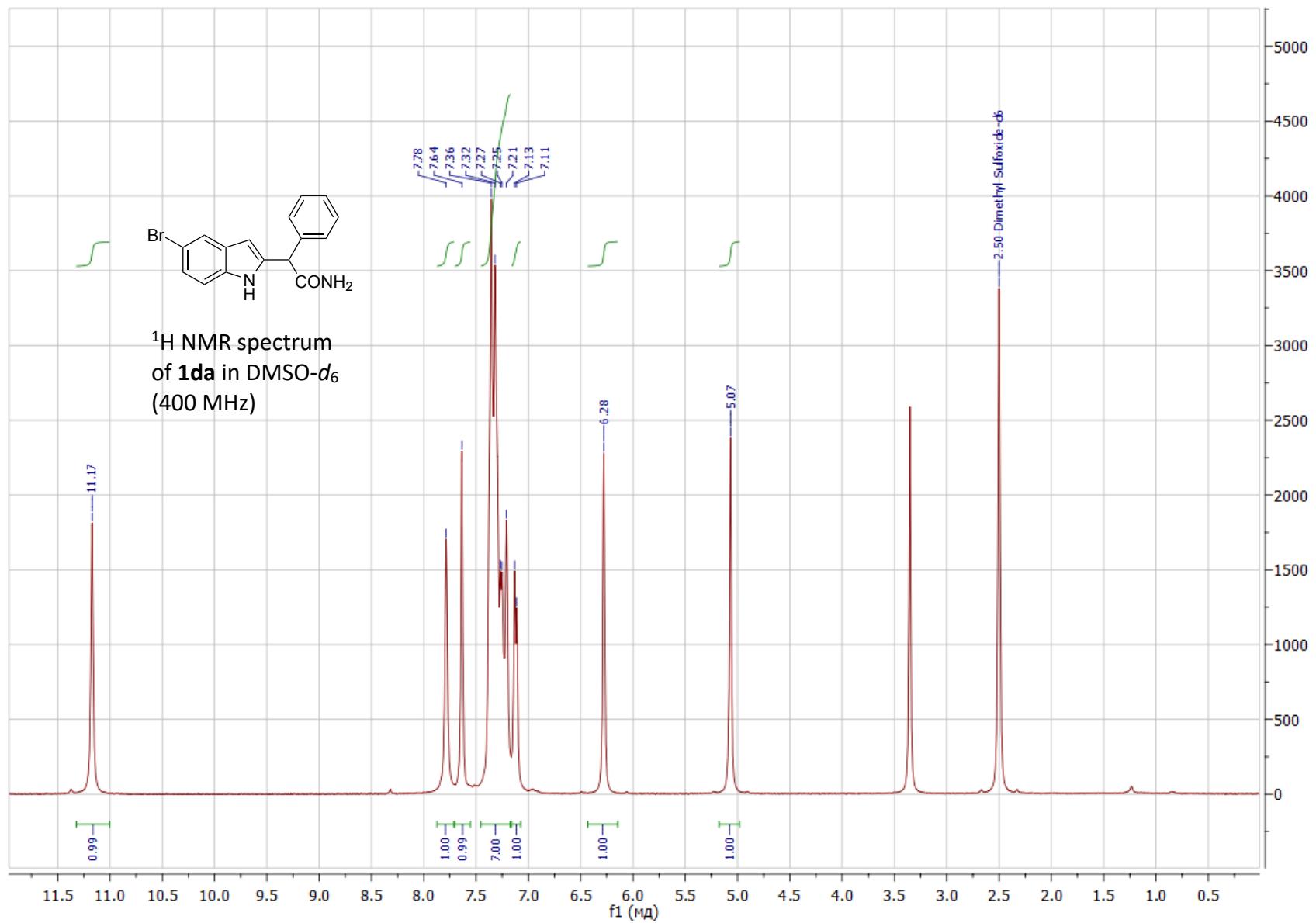


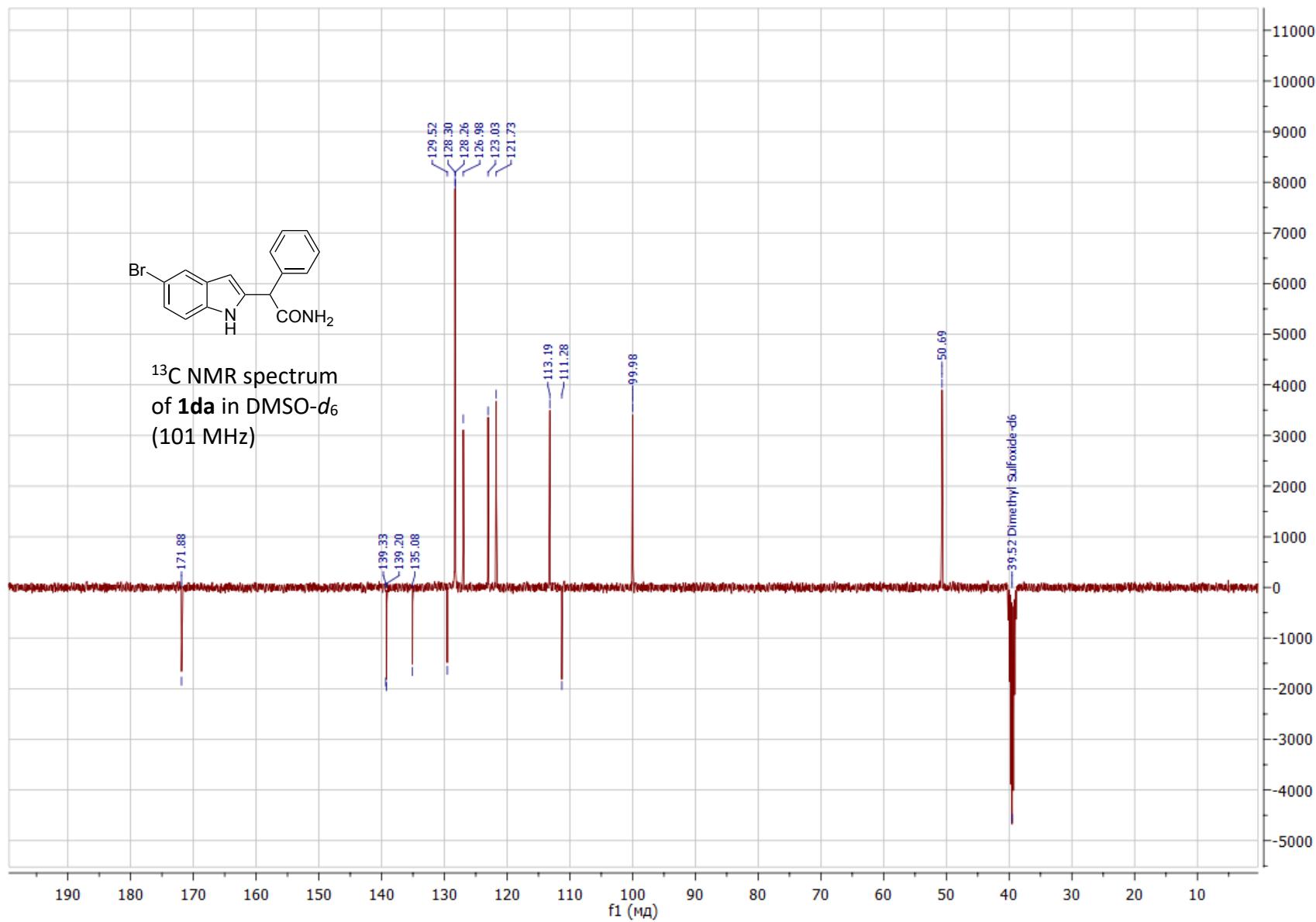




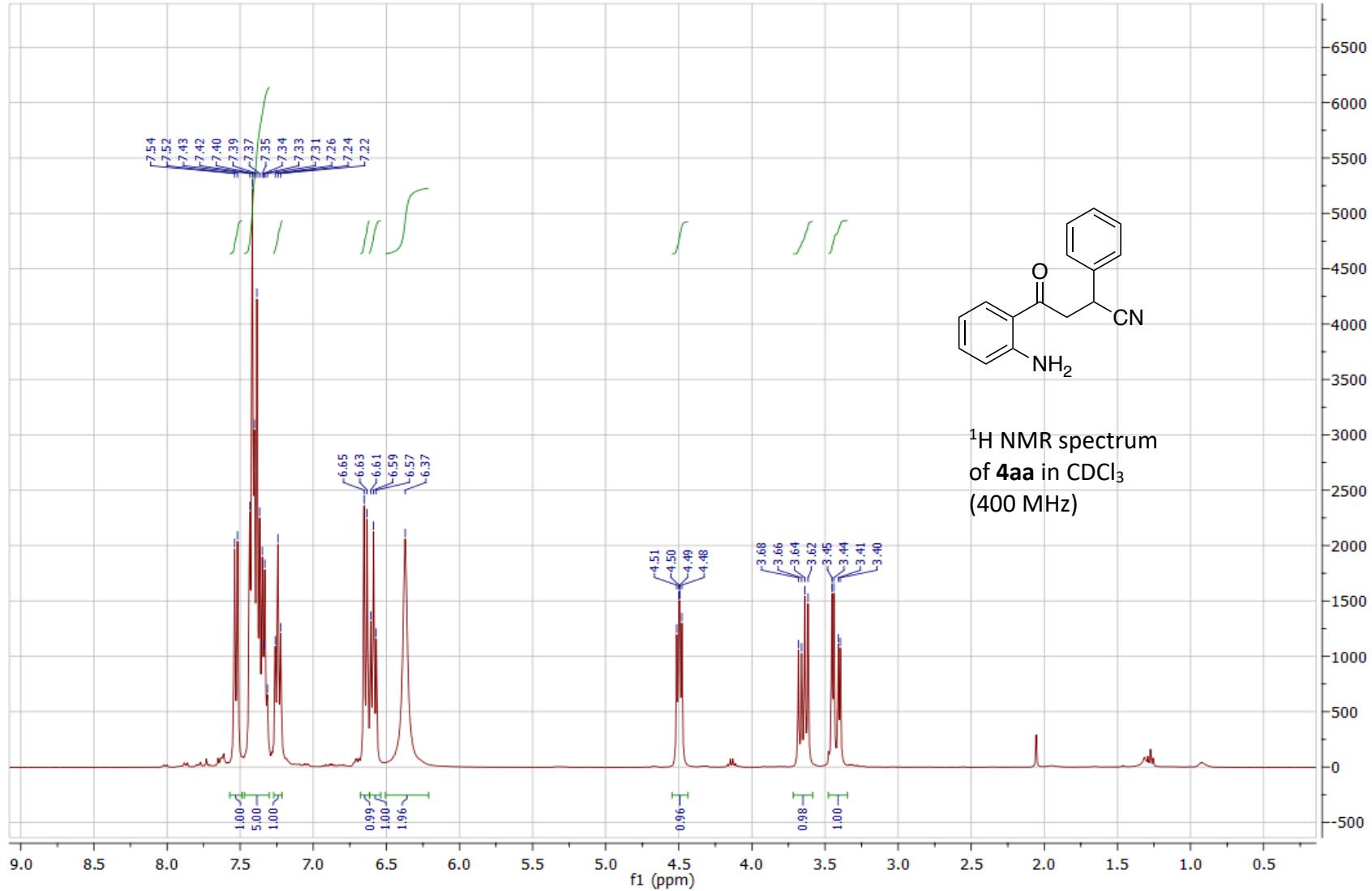


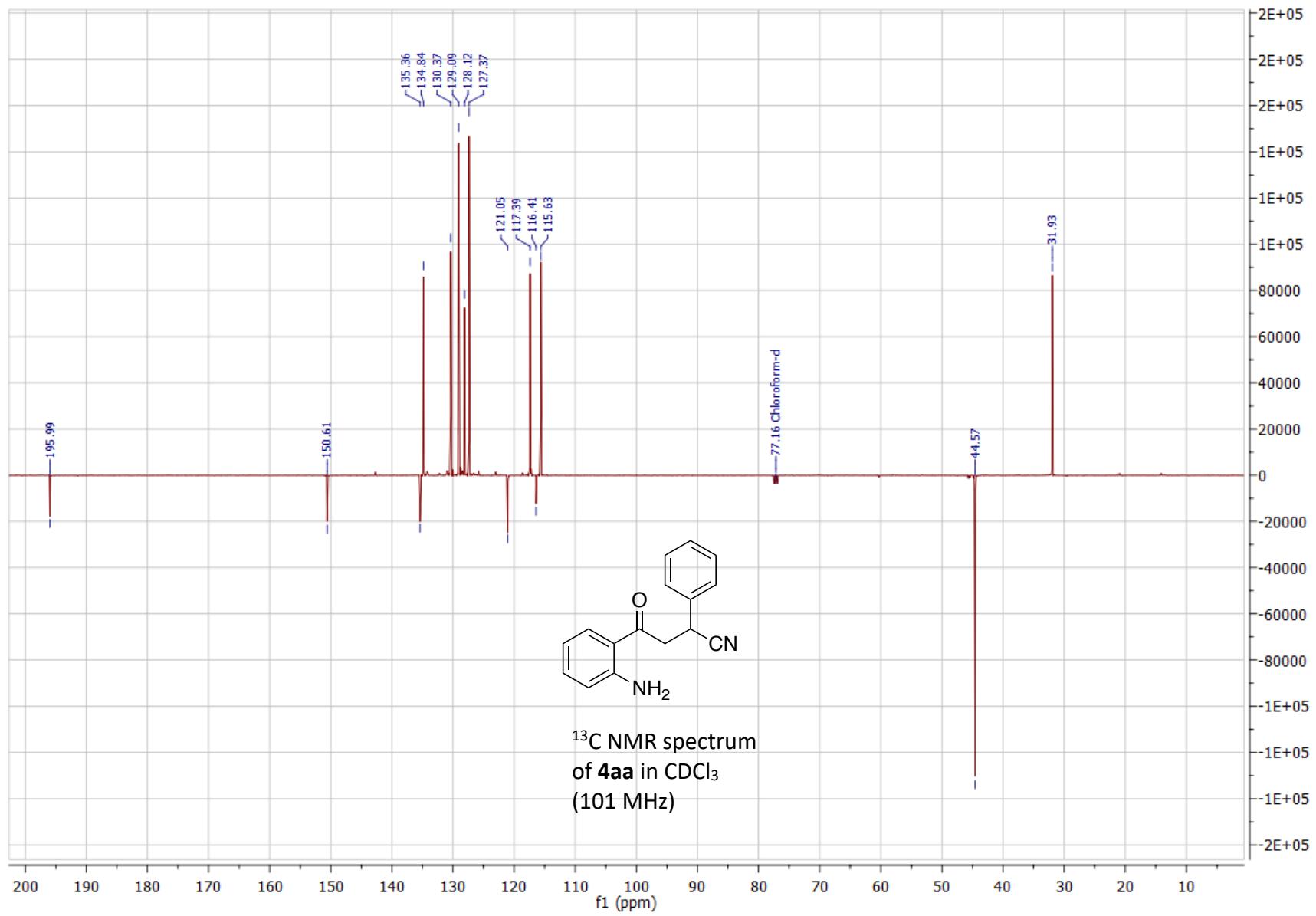


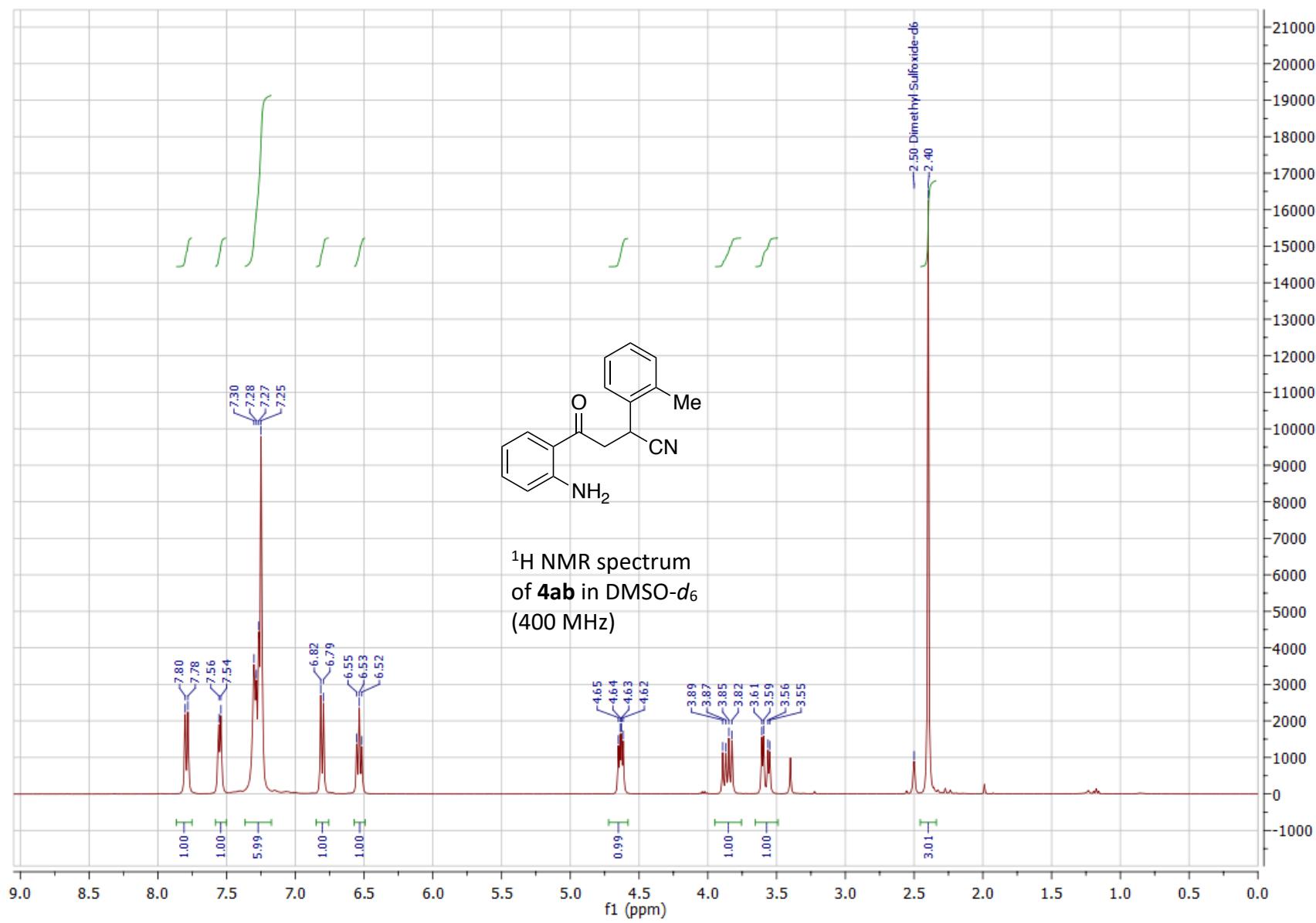


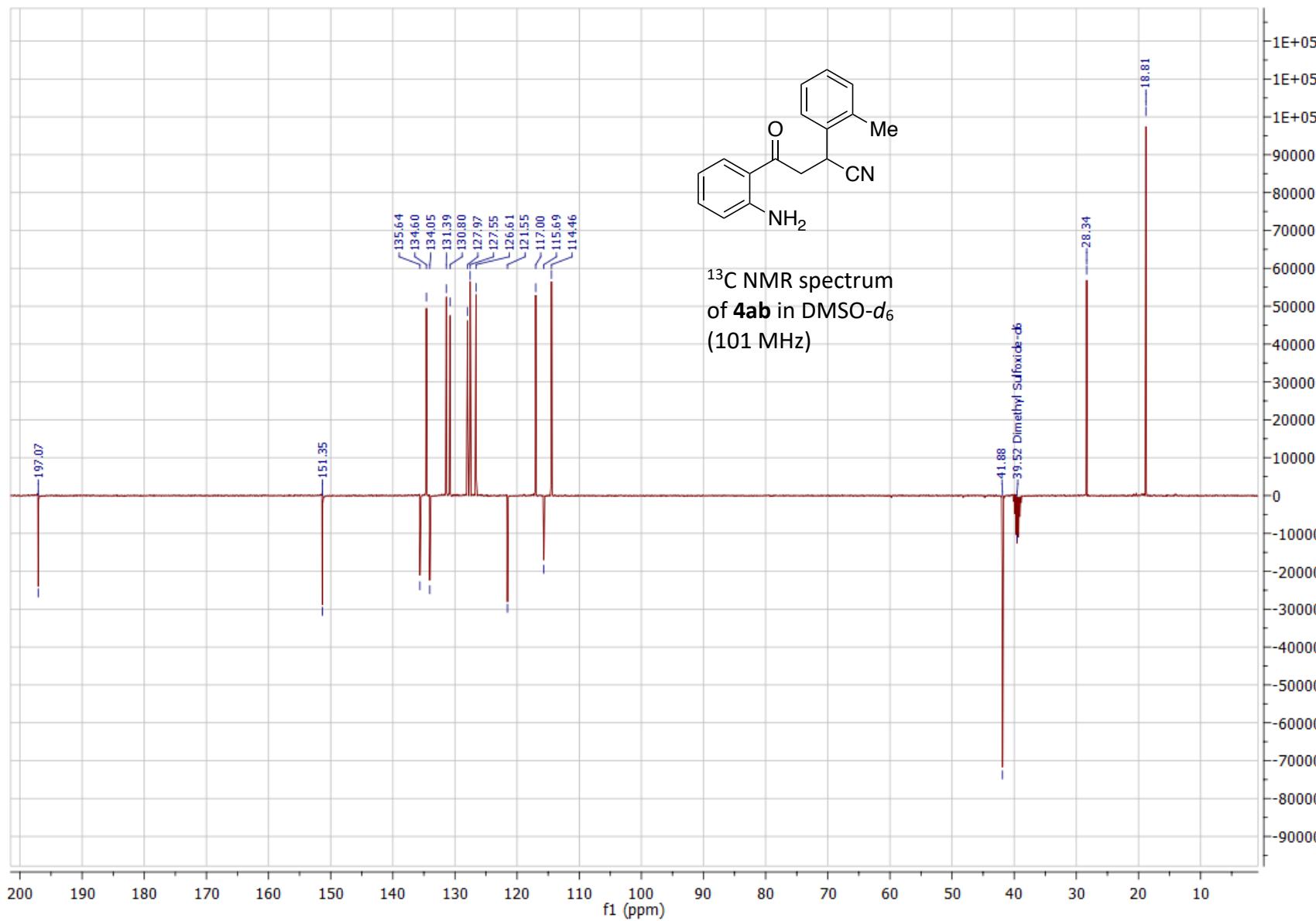


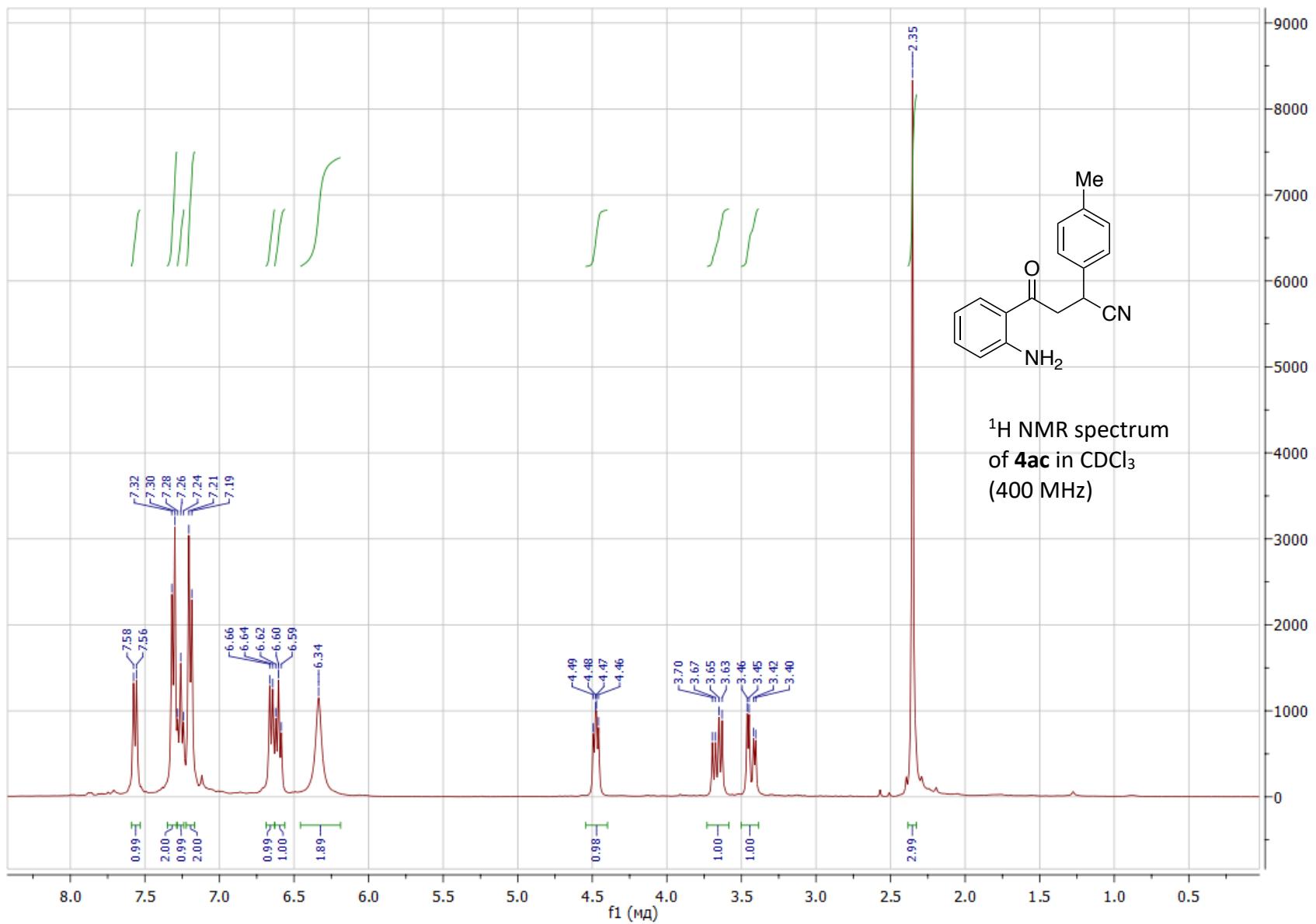
¹H and ¹³C Spectral Charts of 4-(2-aminophenyl)-4-oxo-2-arylbutanenitriles 4

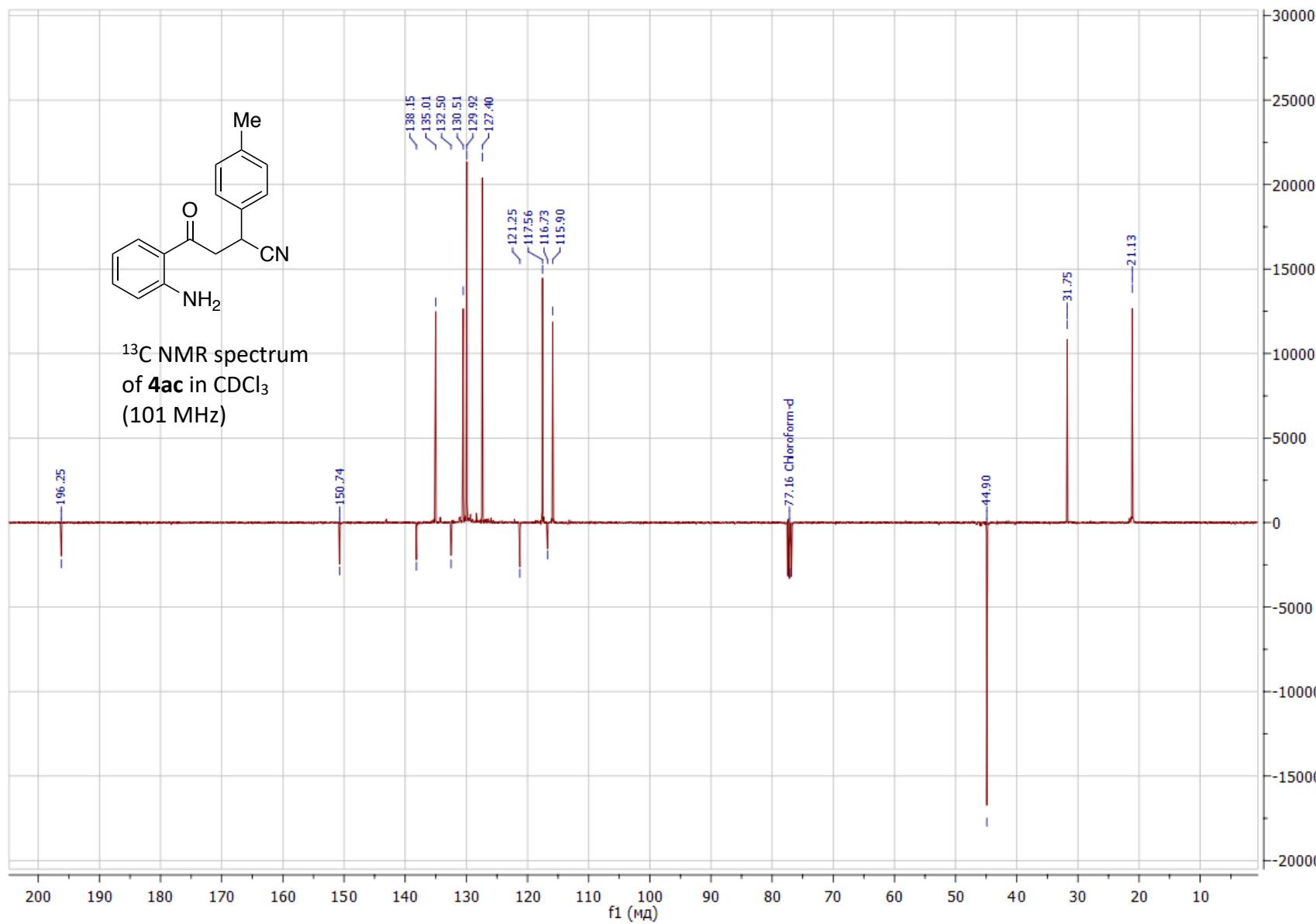


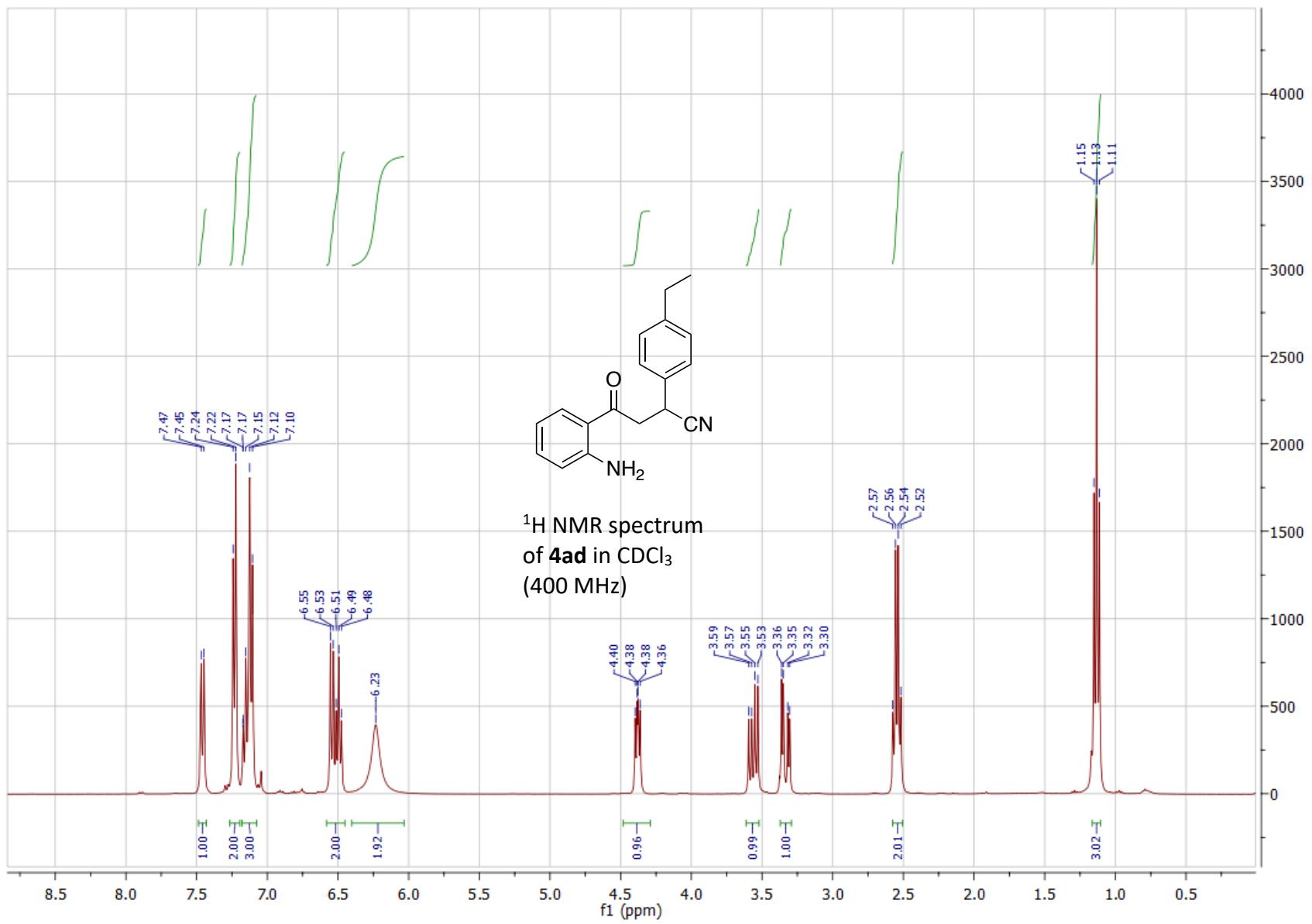


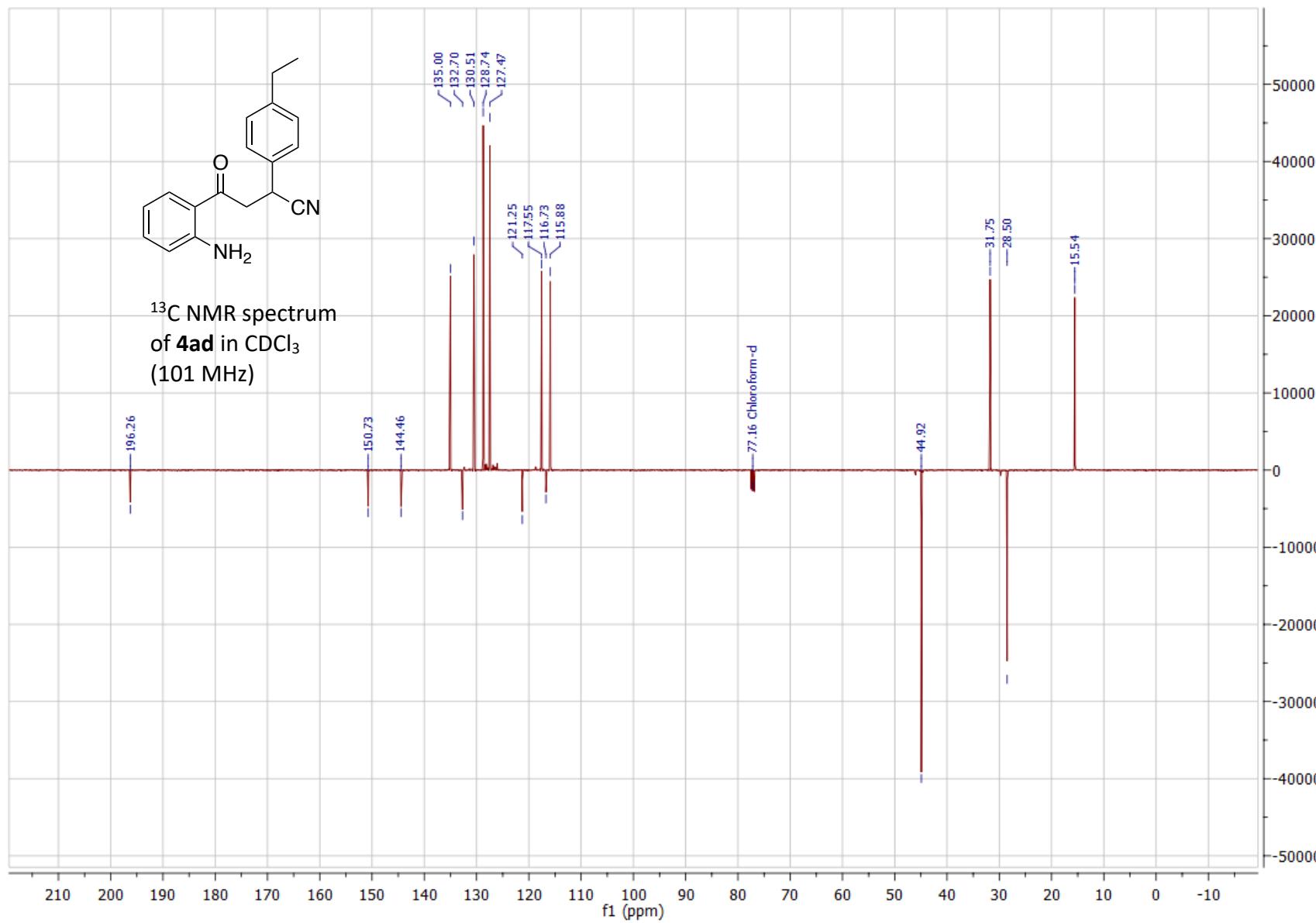


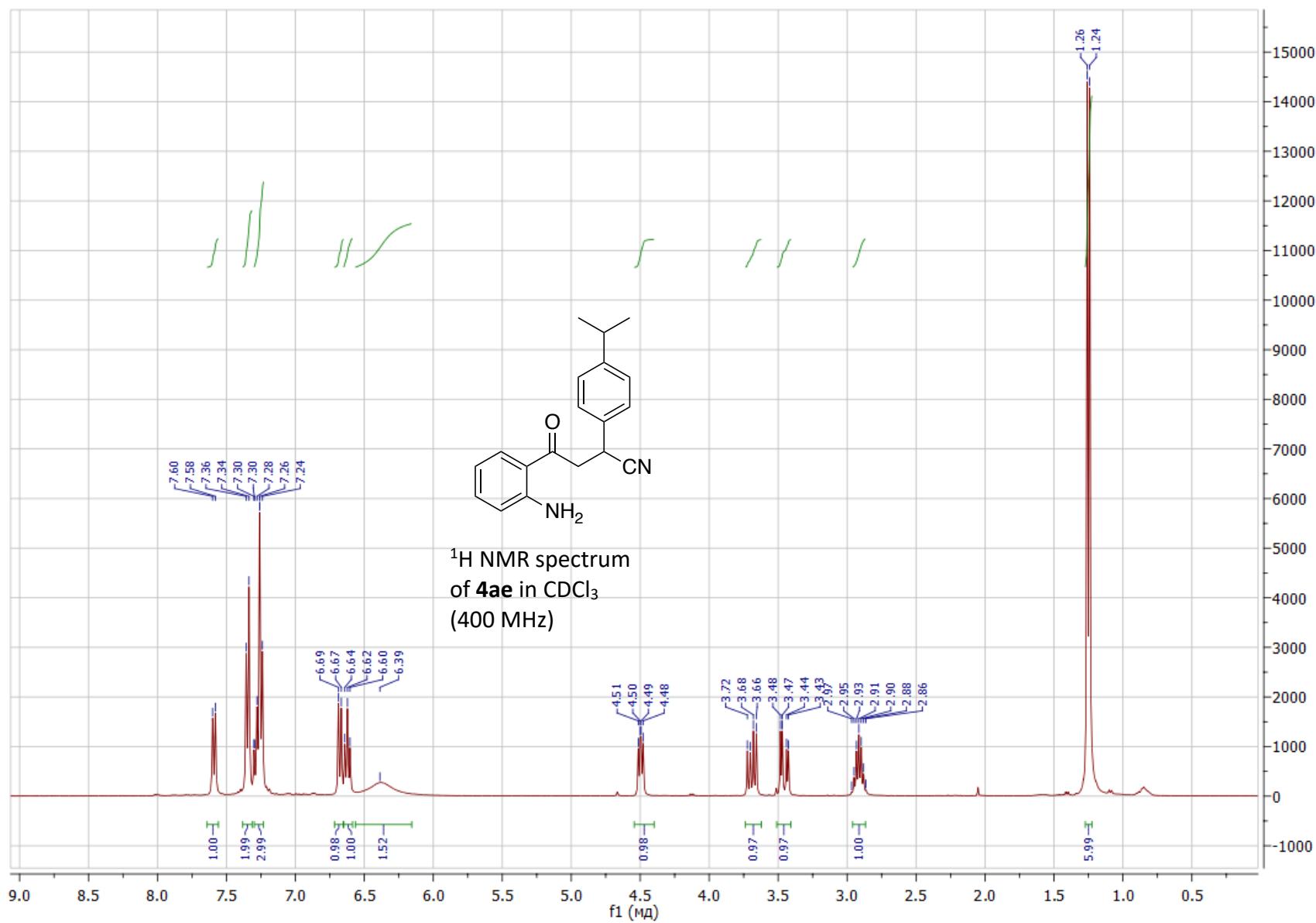


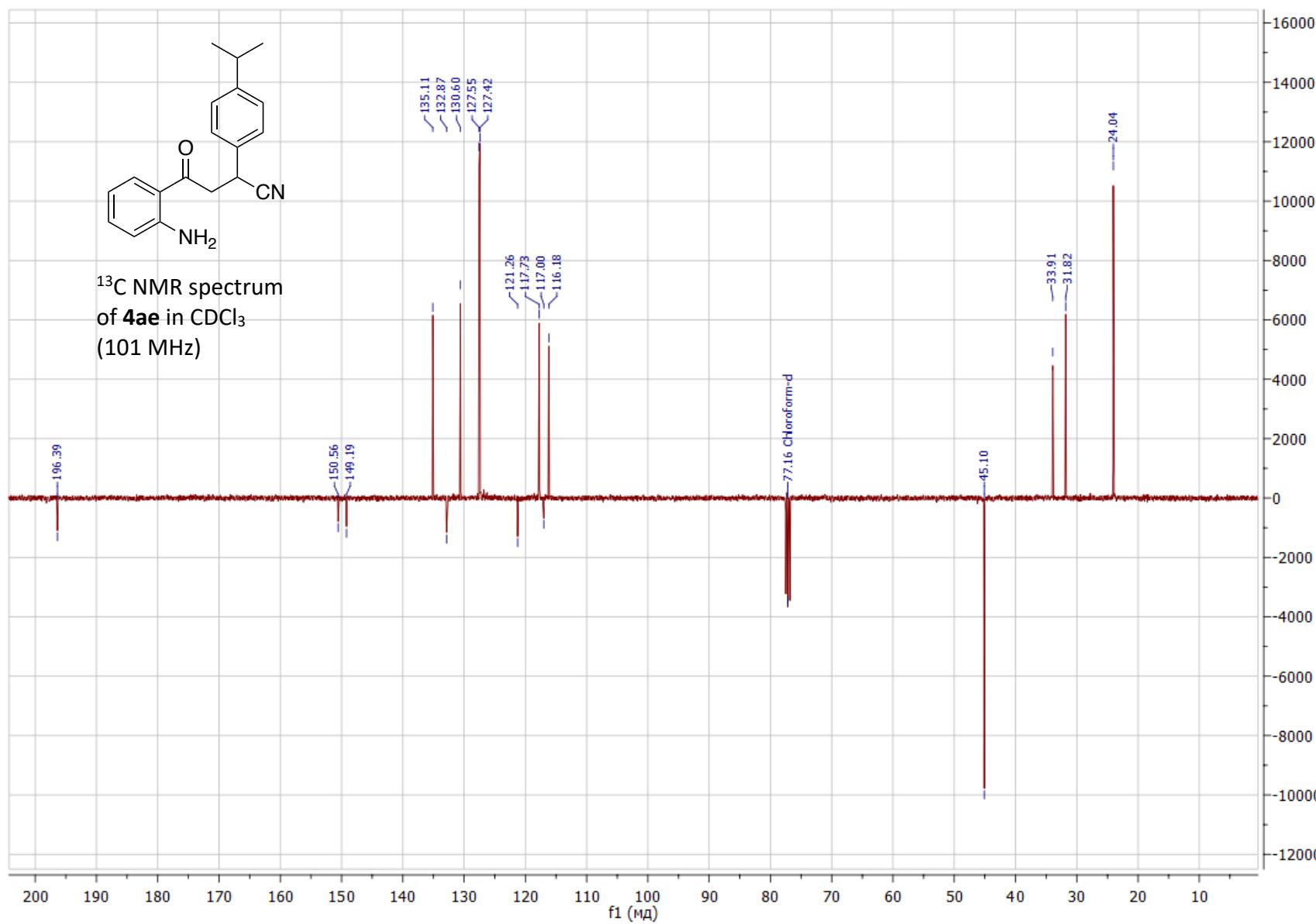


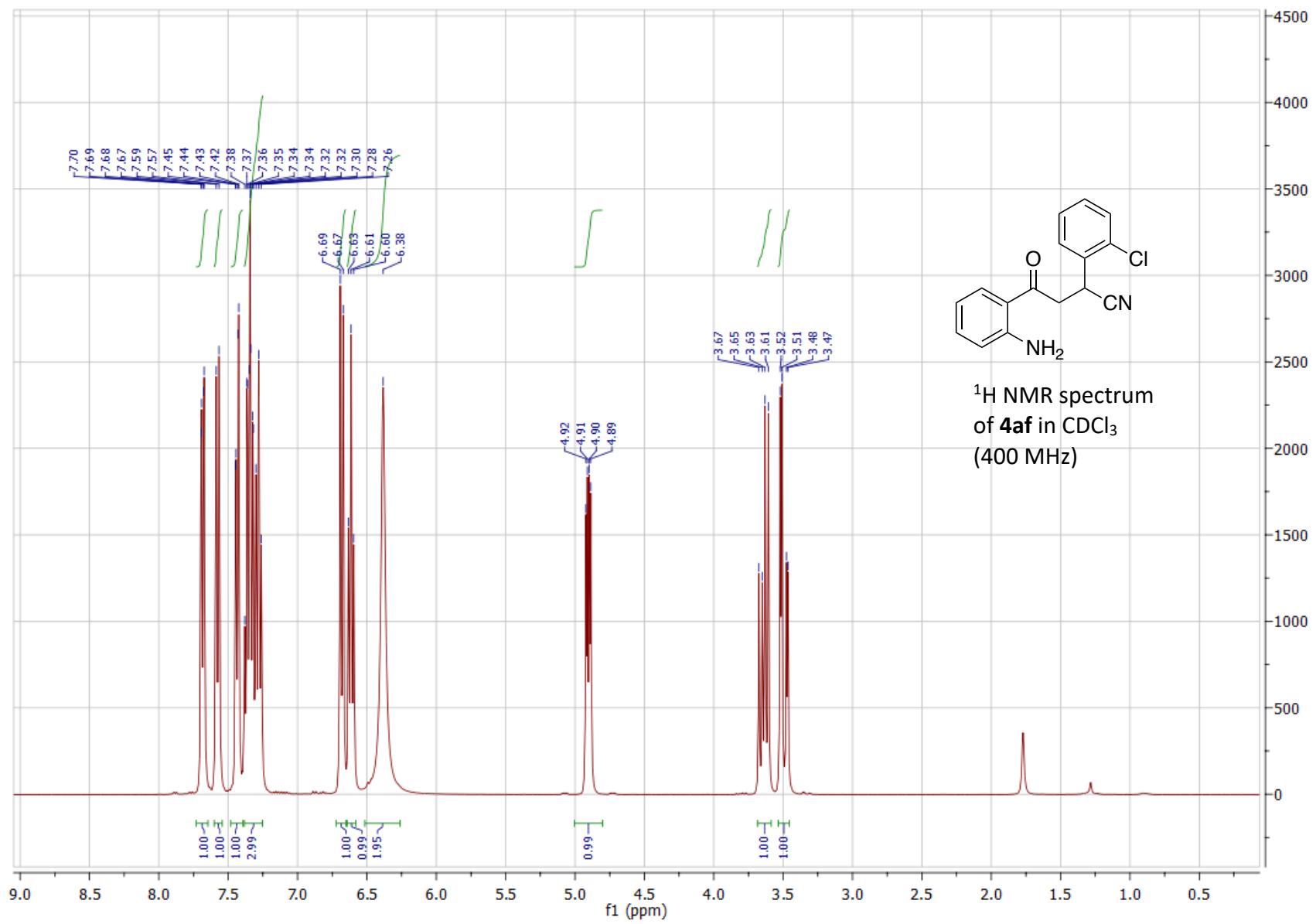


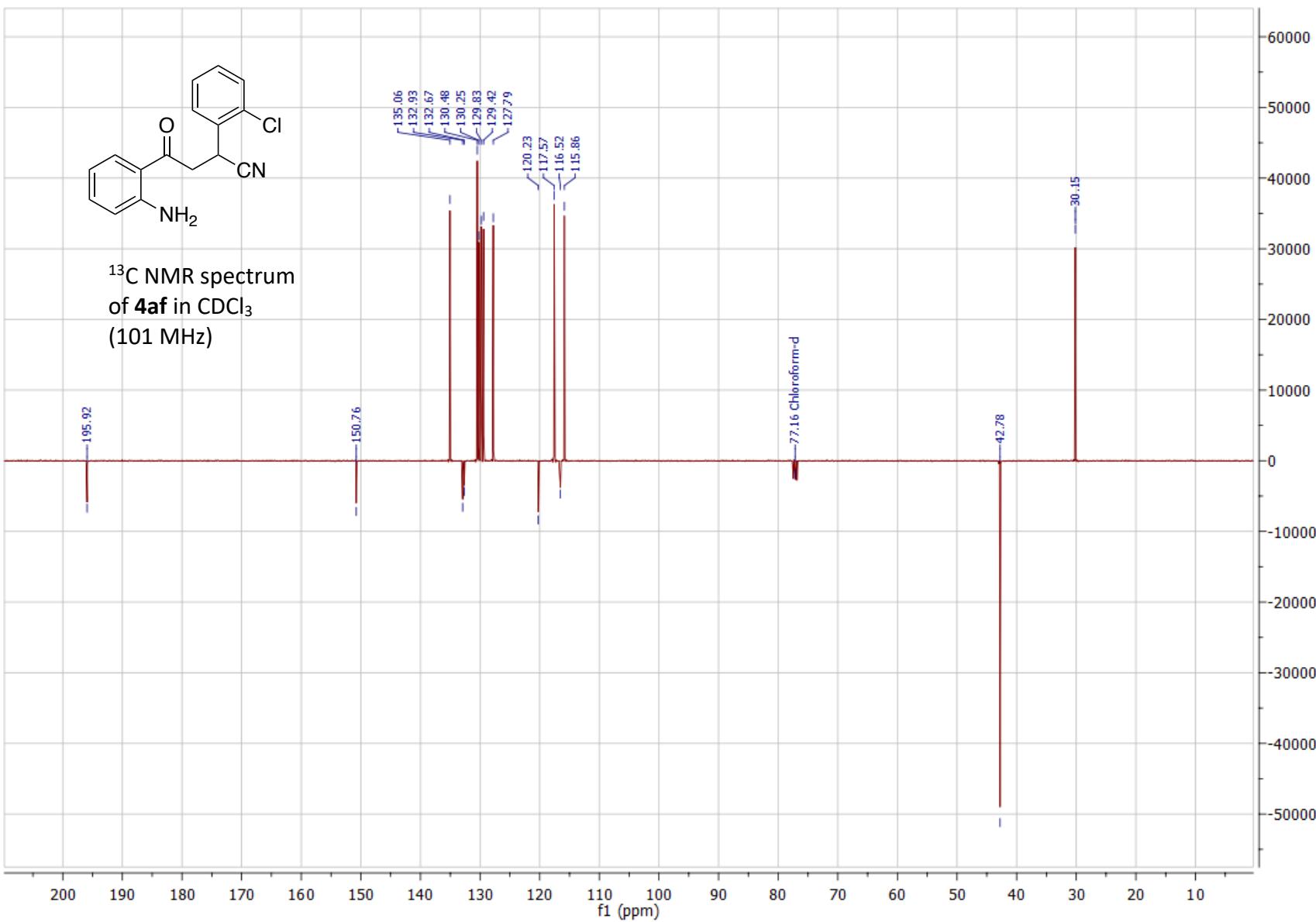


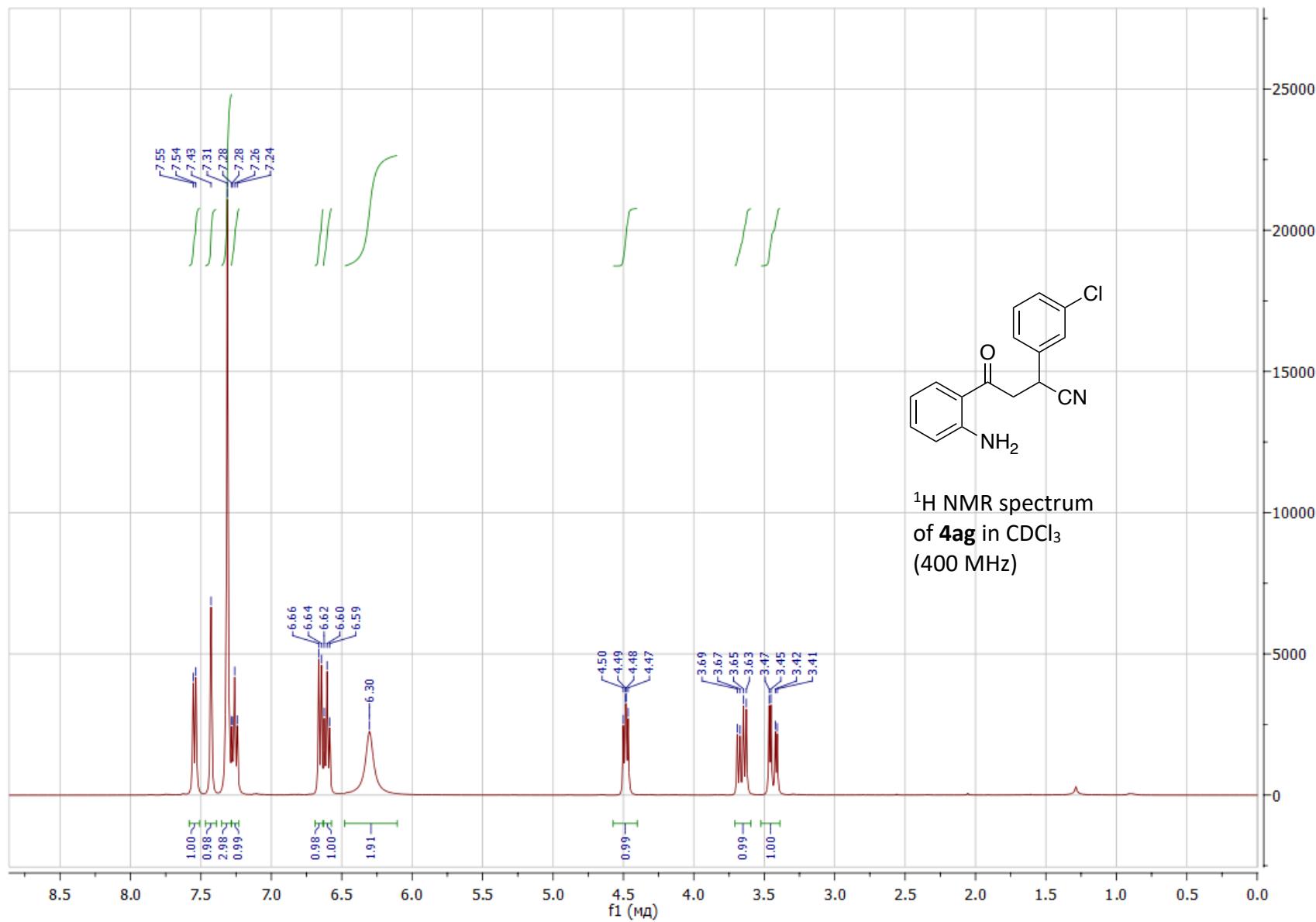


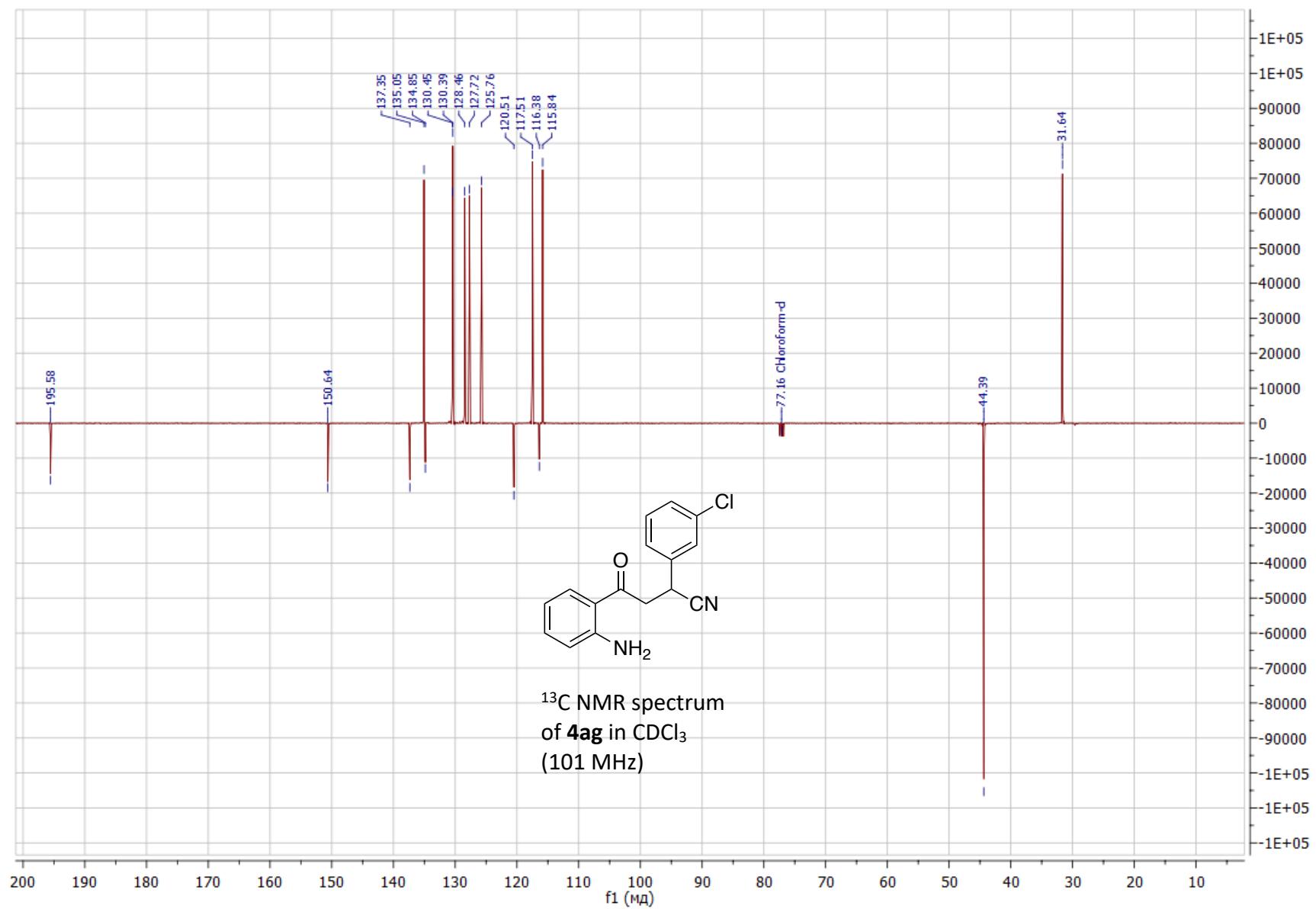


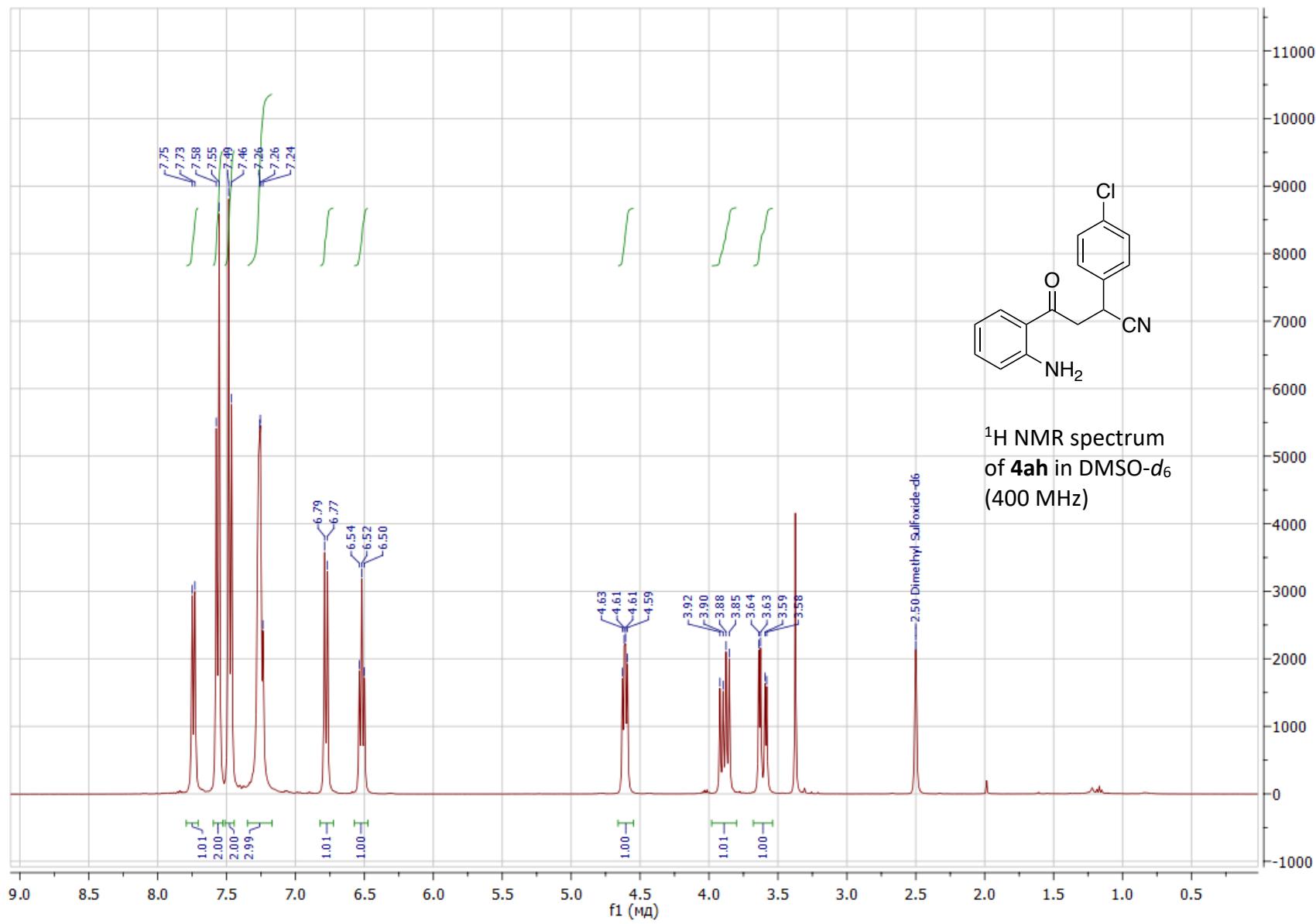


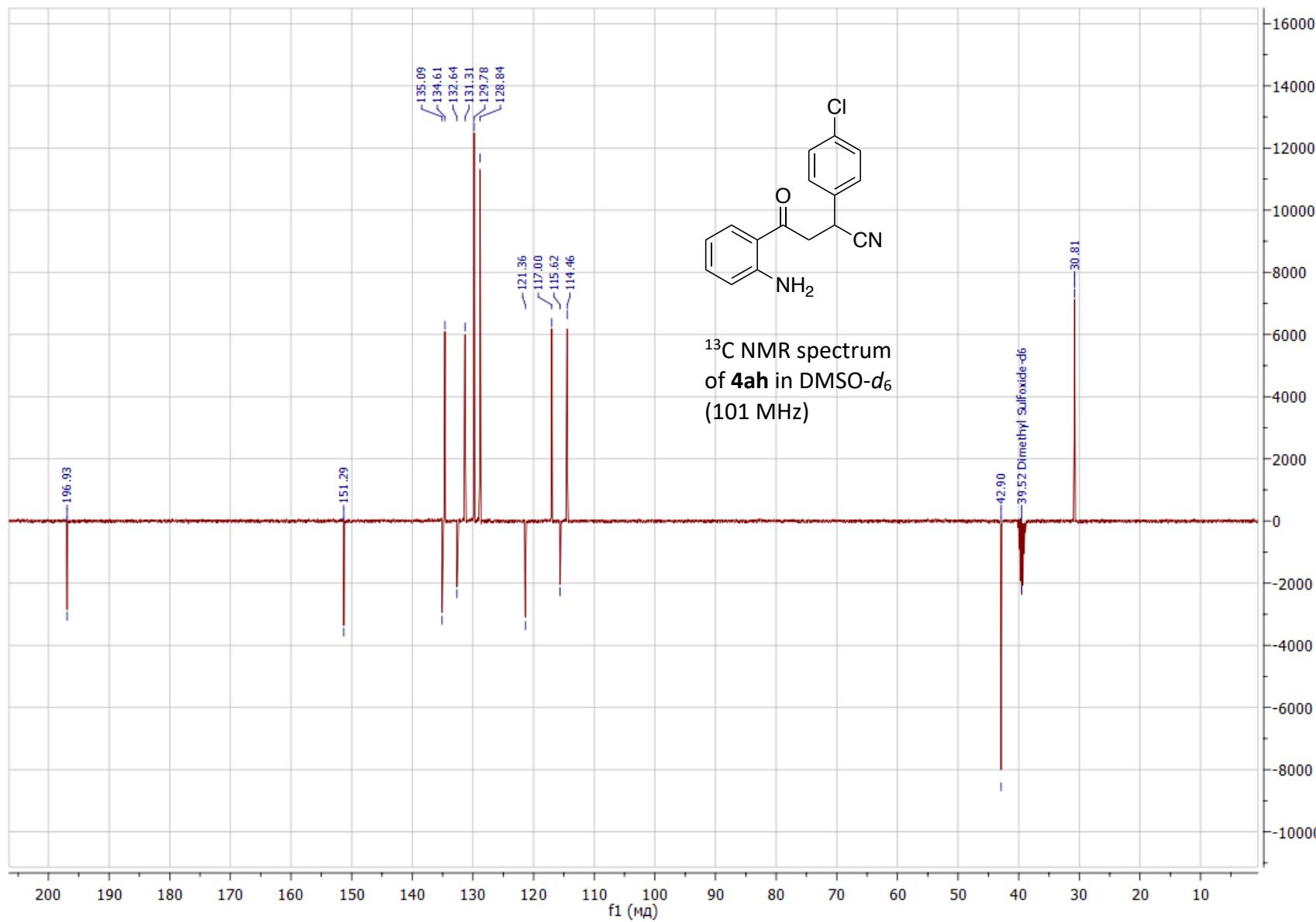


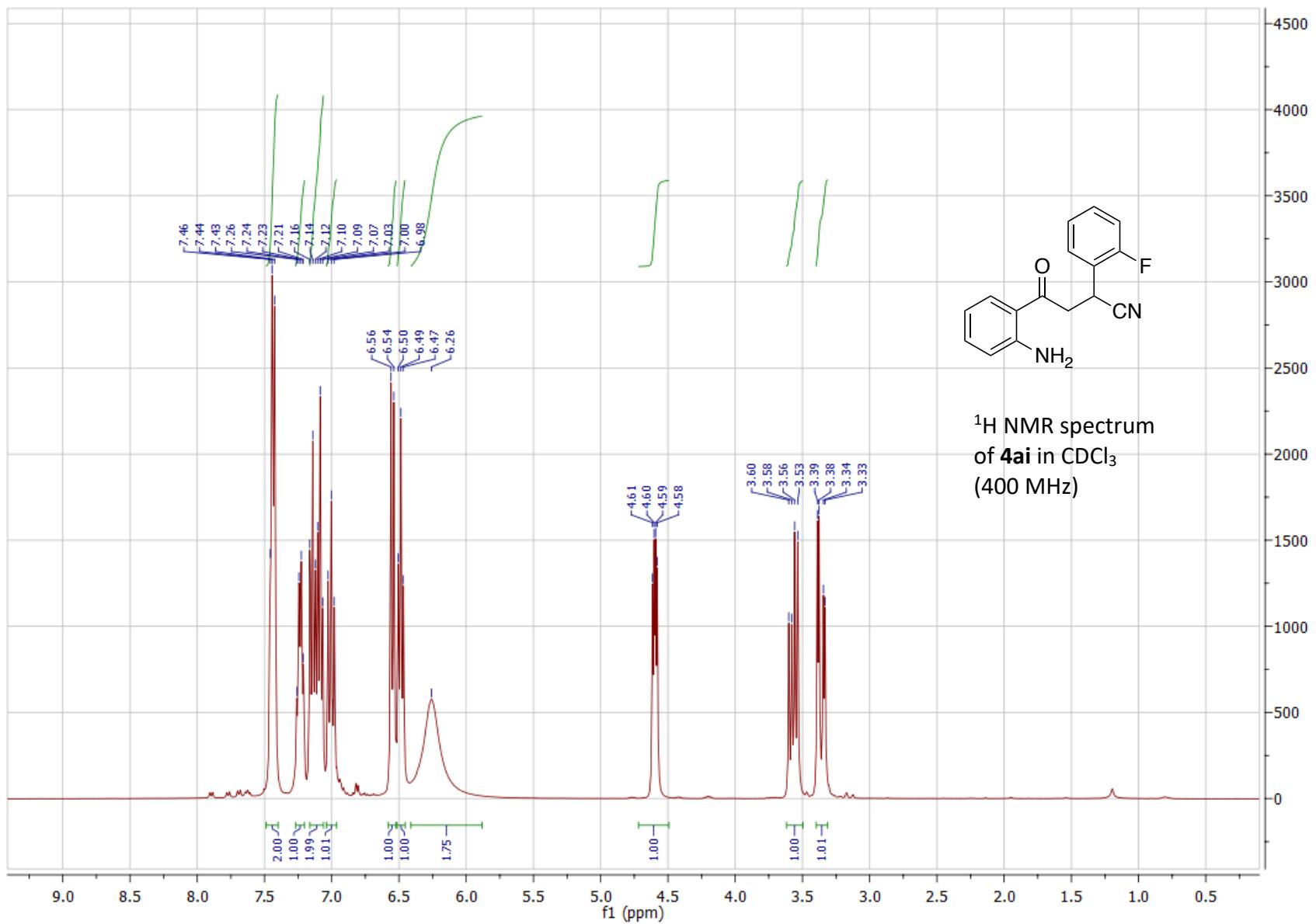


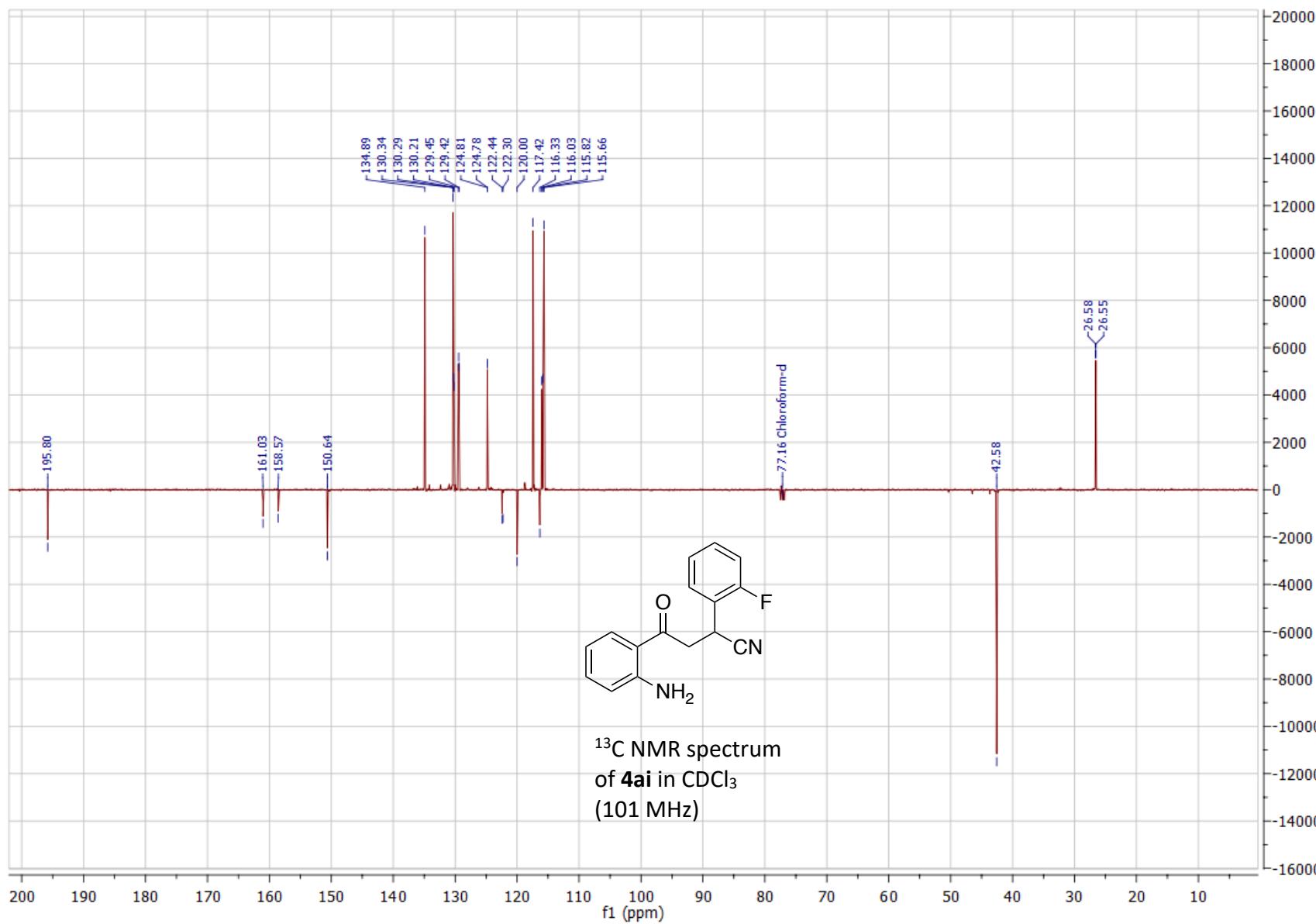


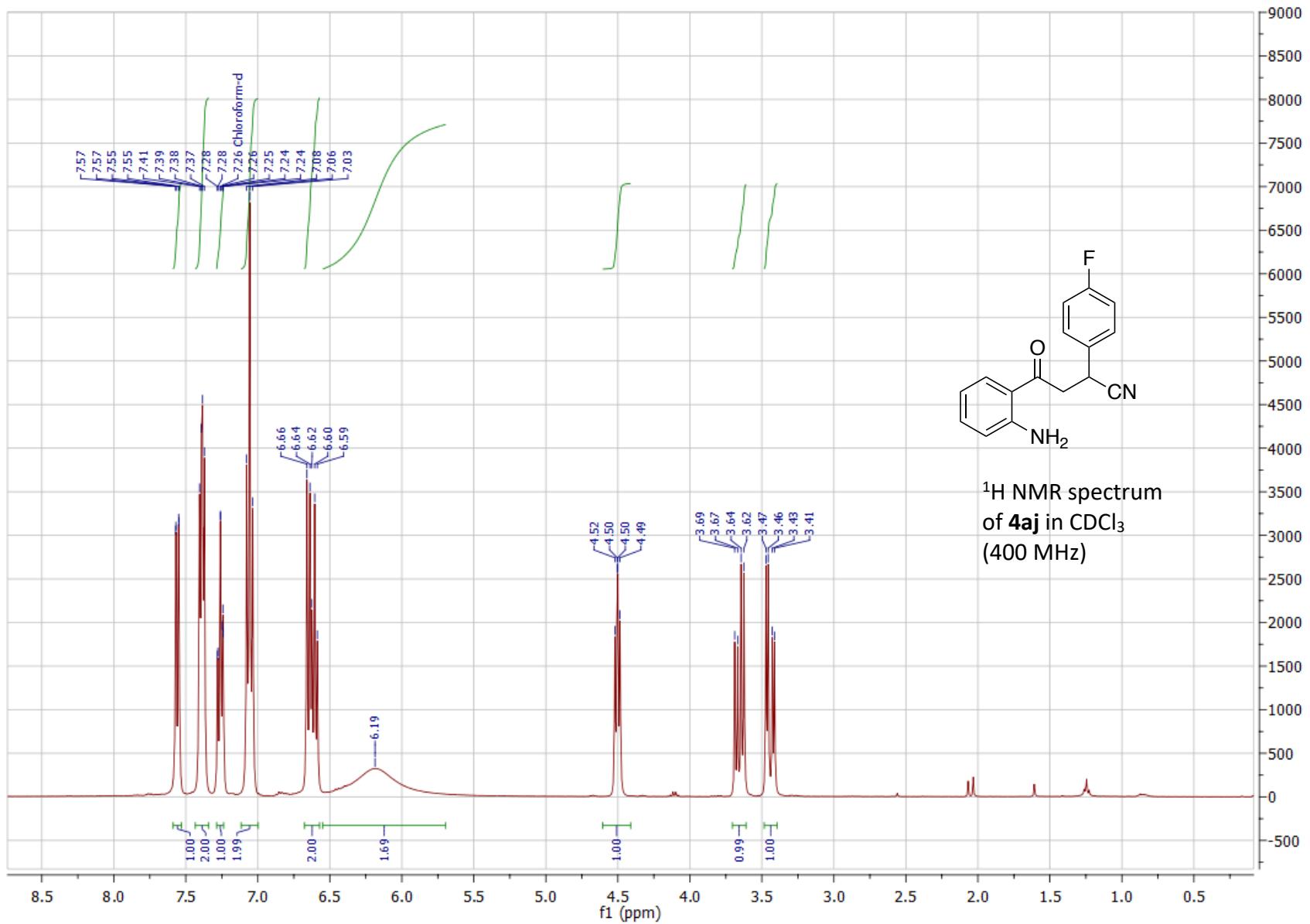


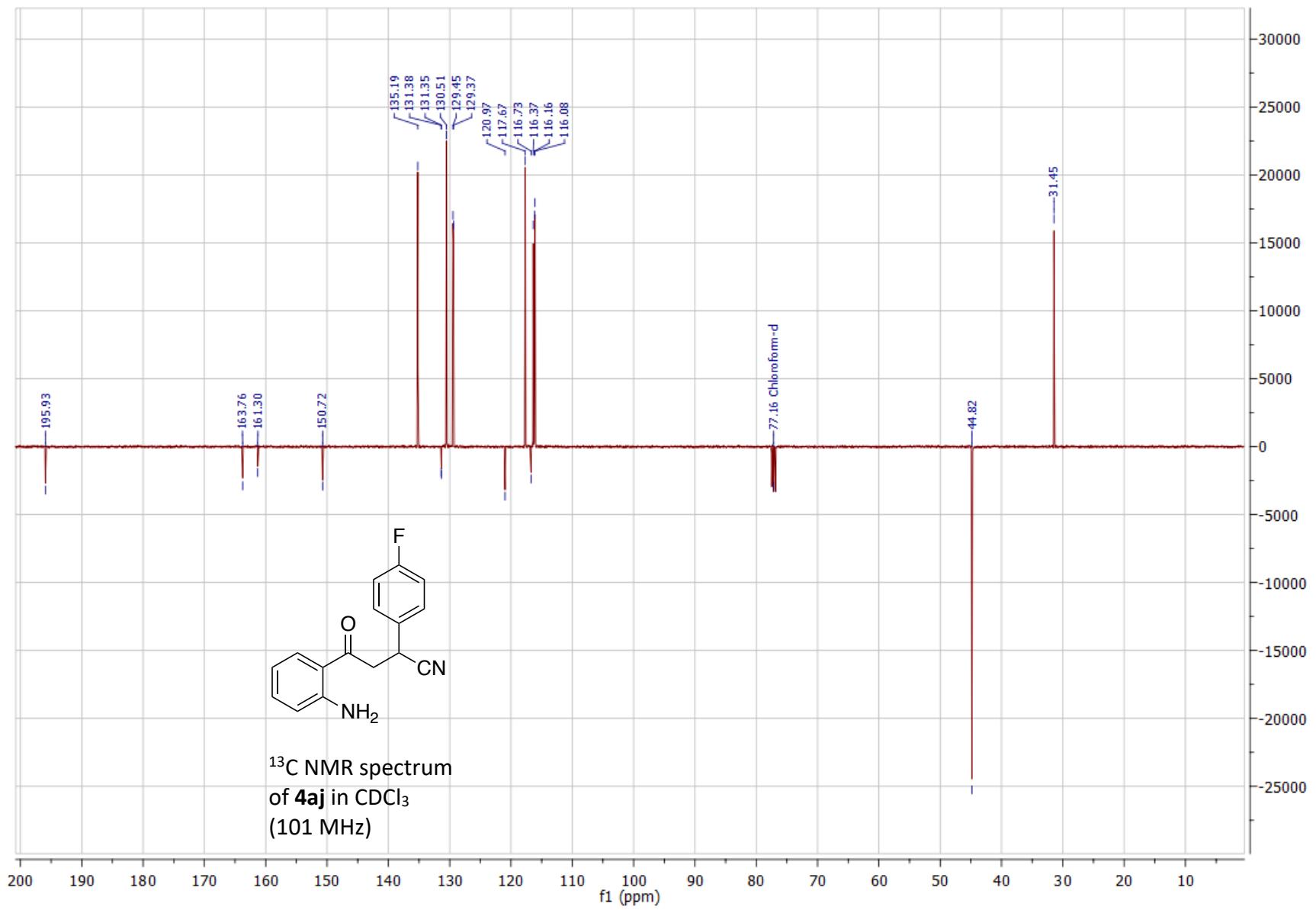


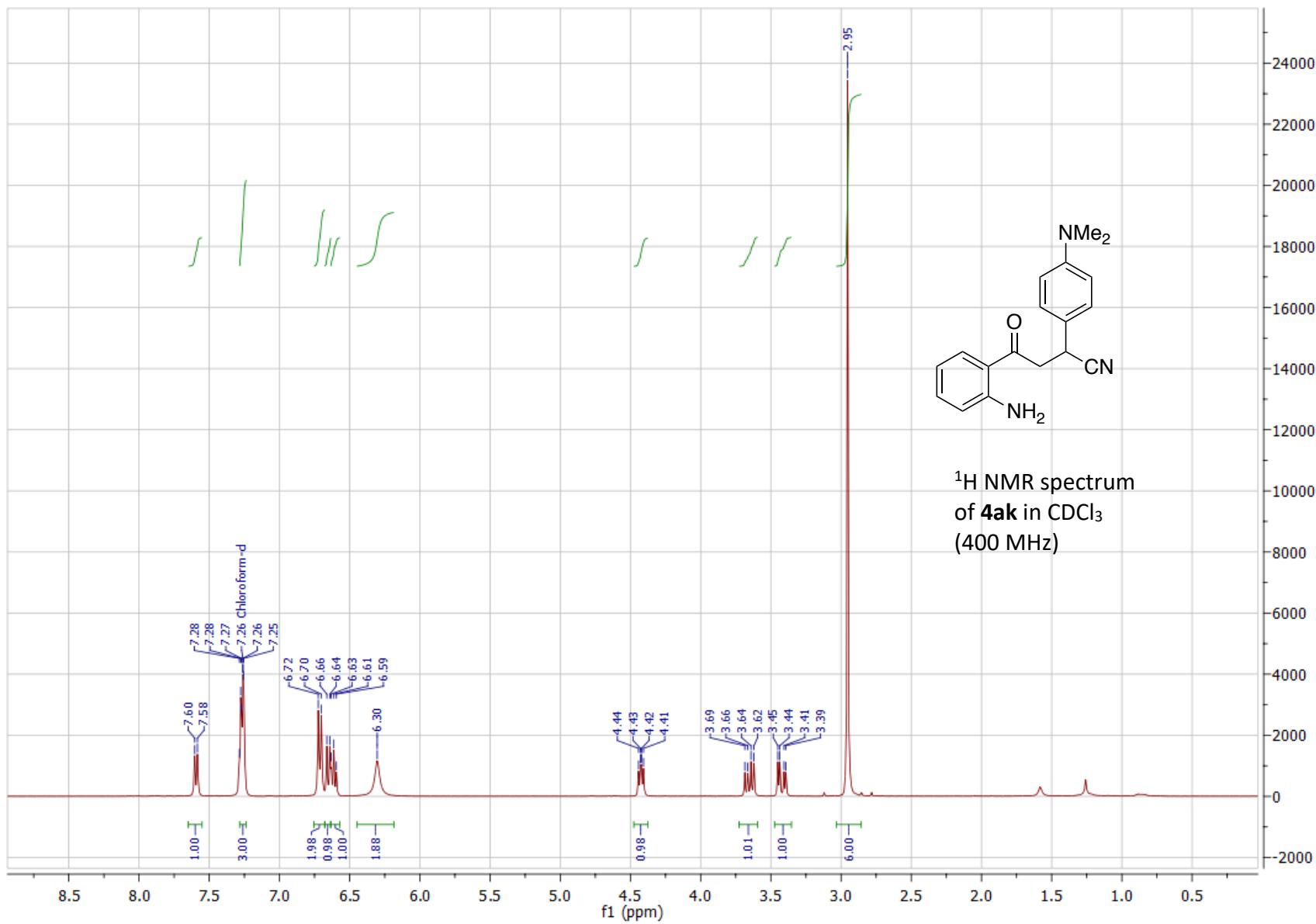


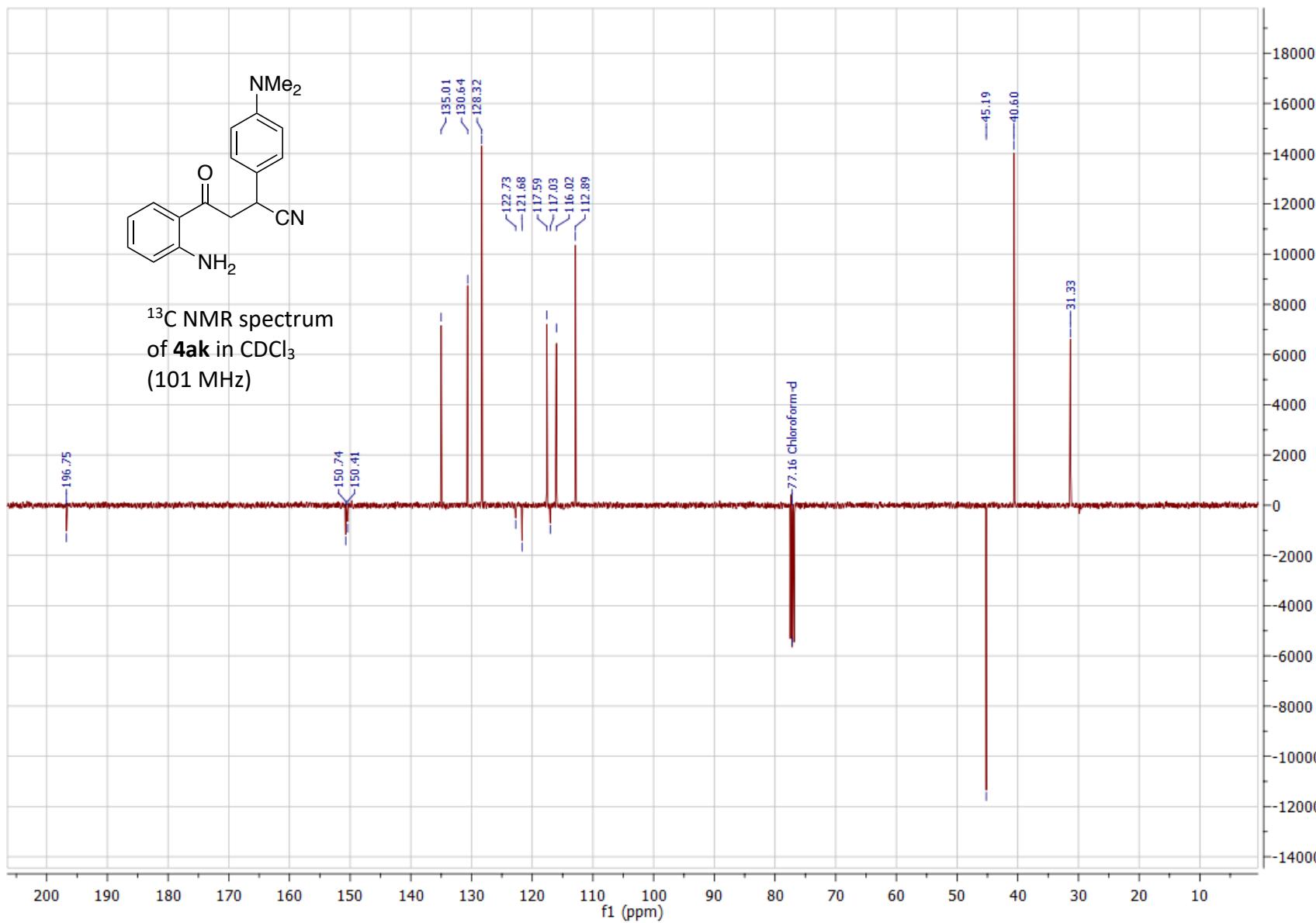


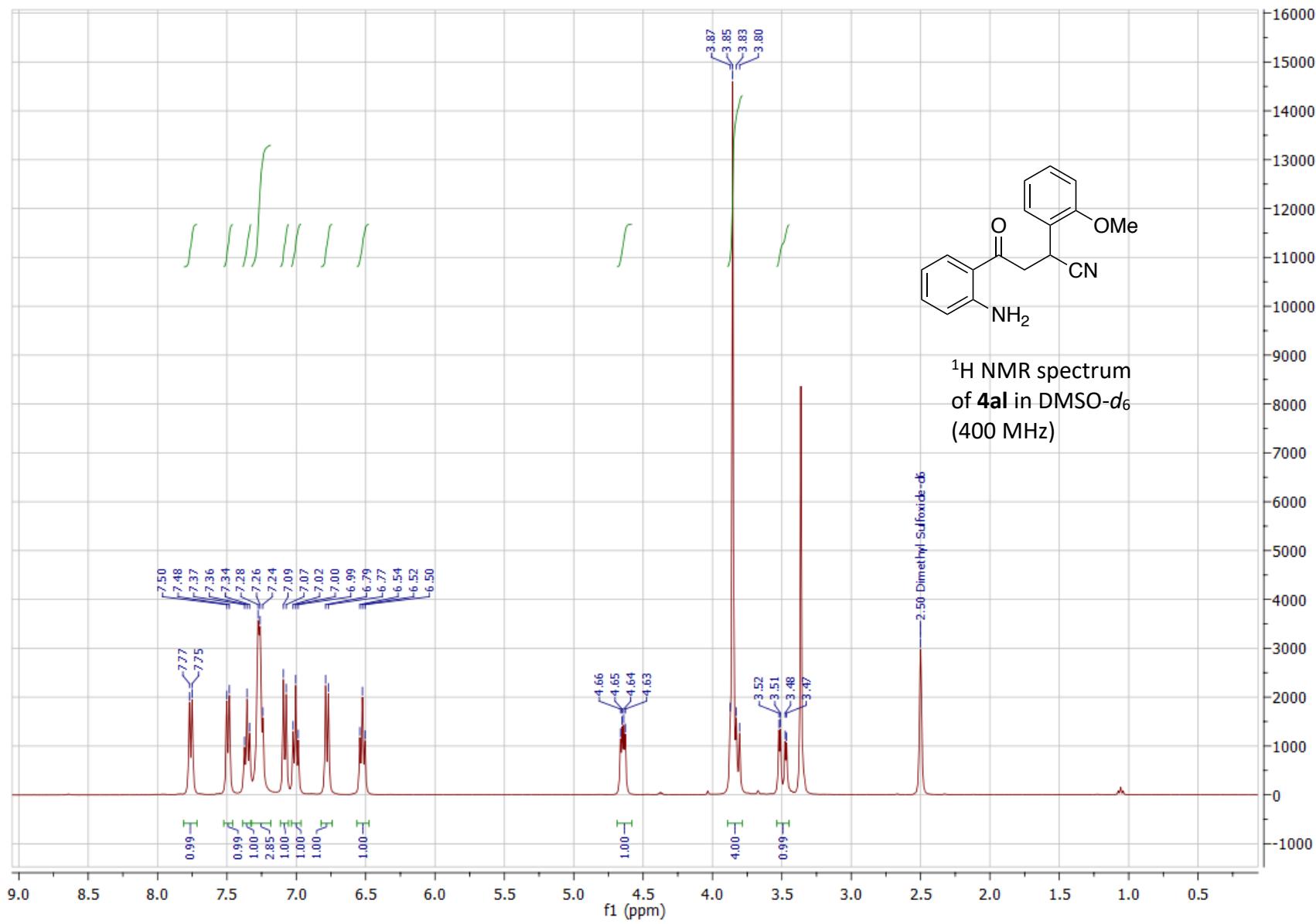


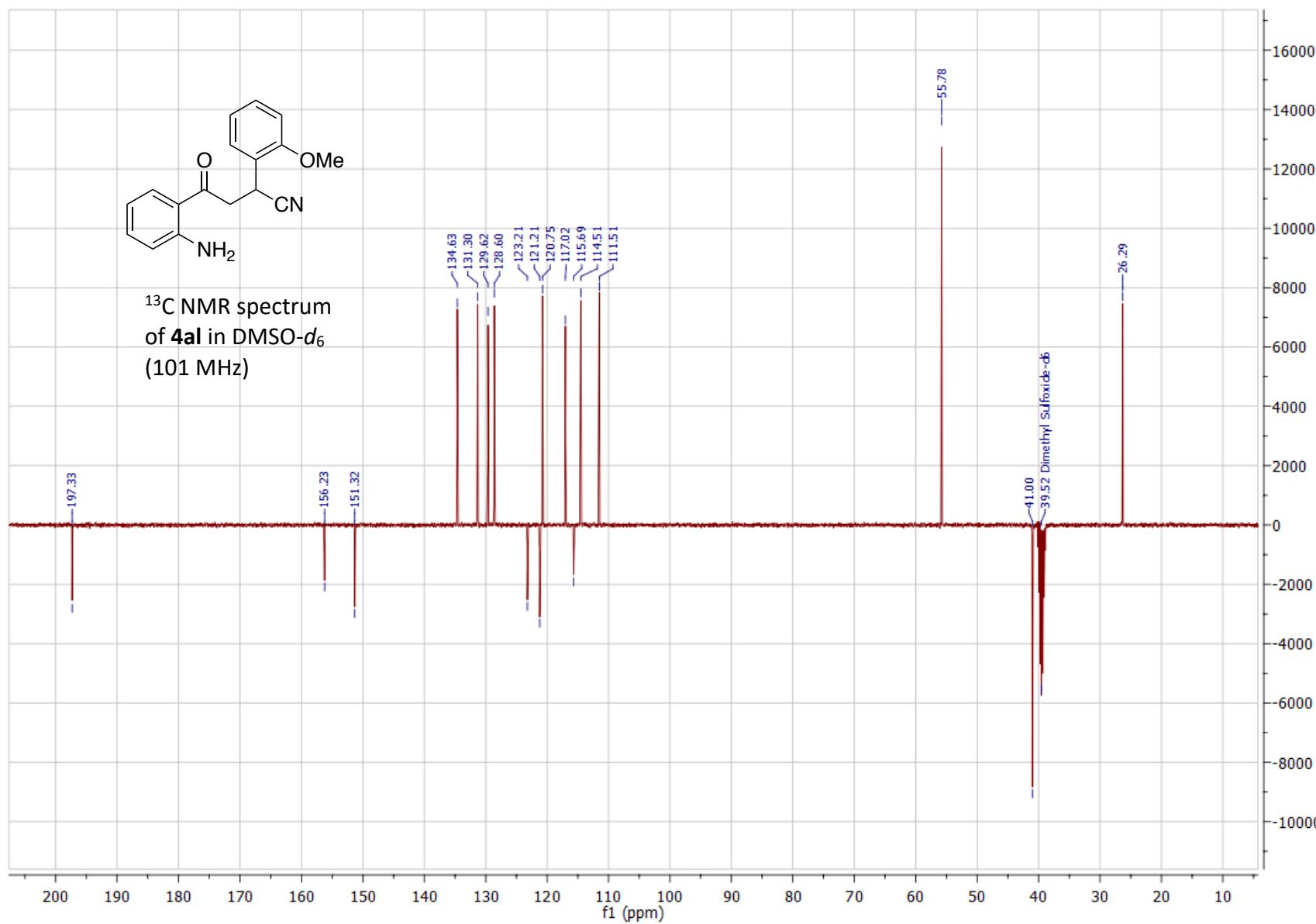


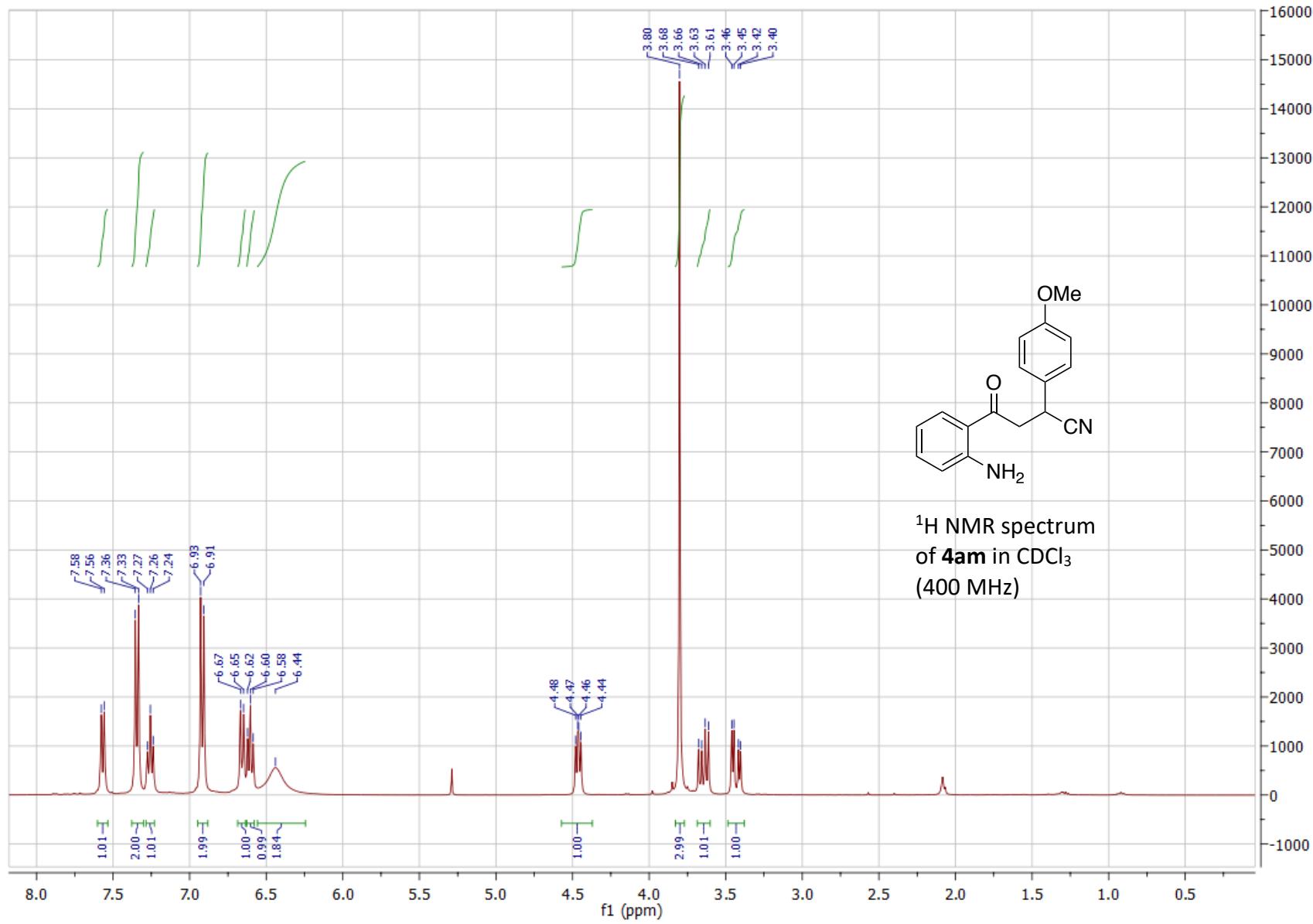


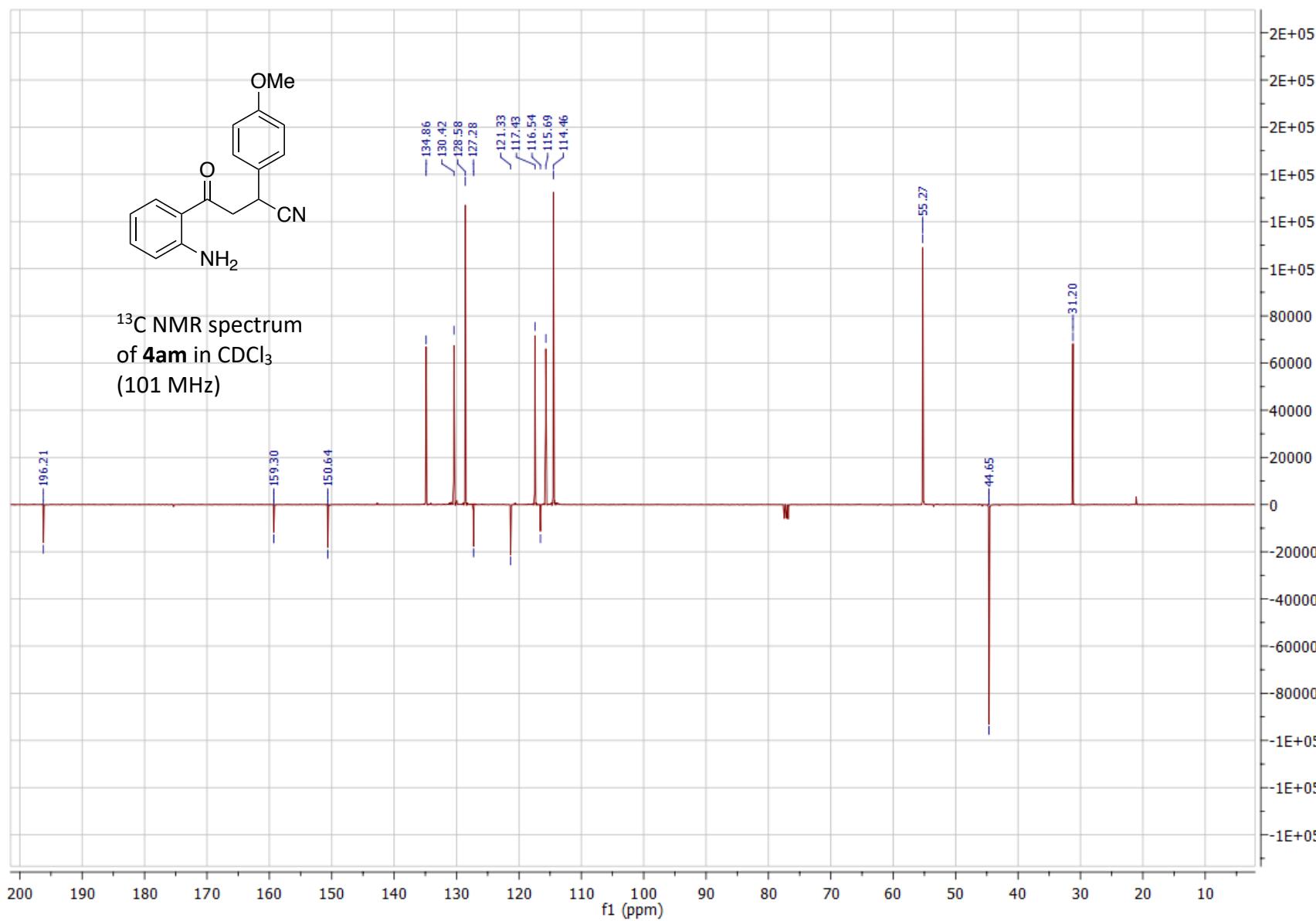


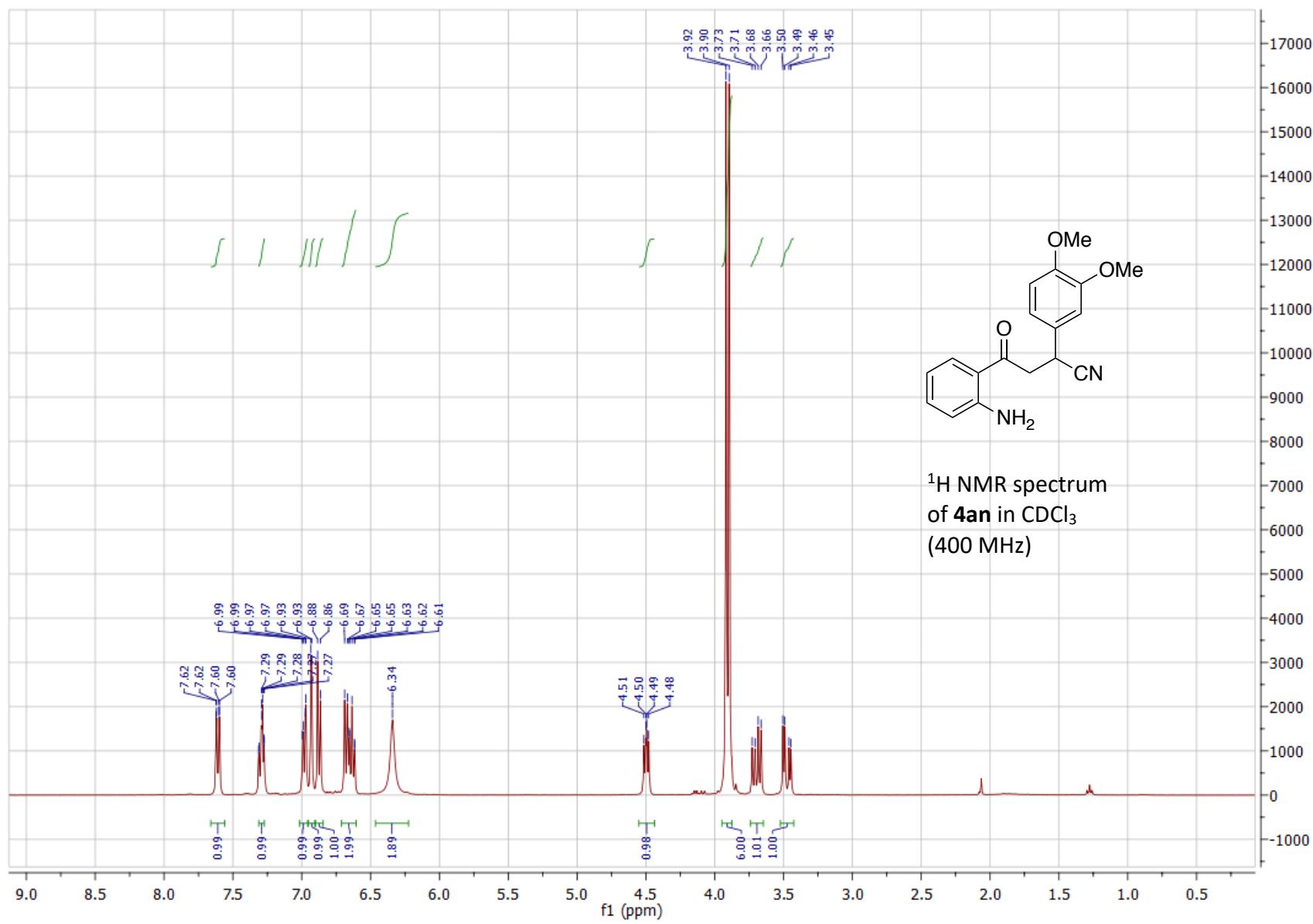


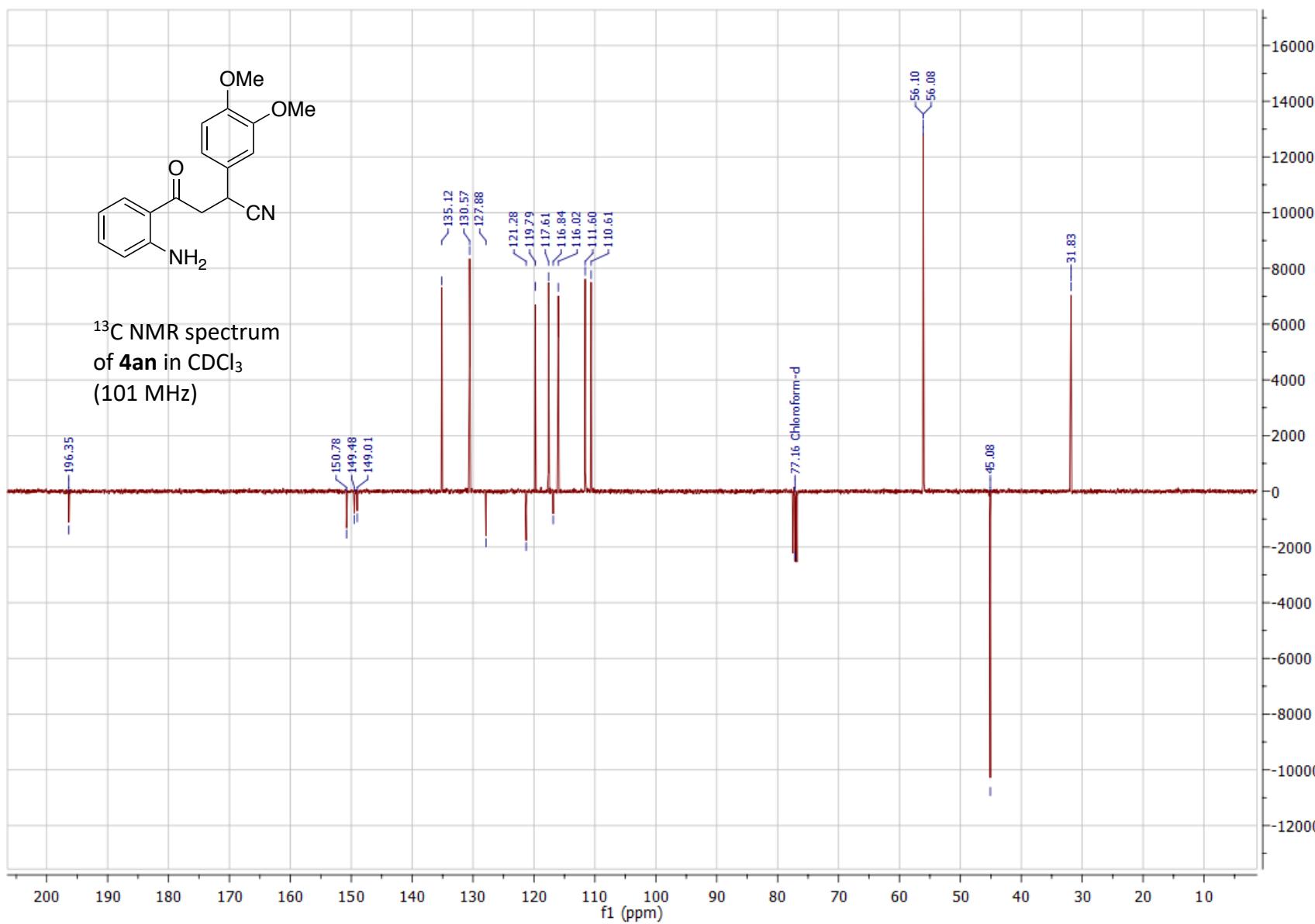


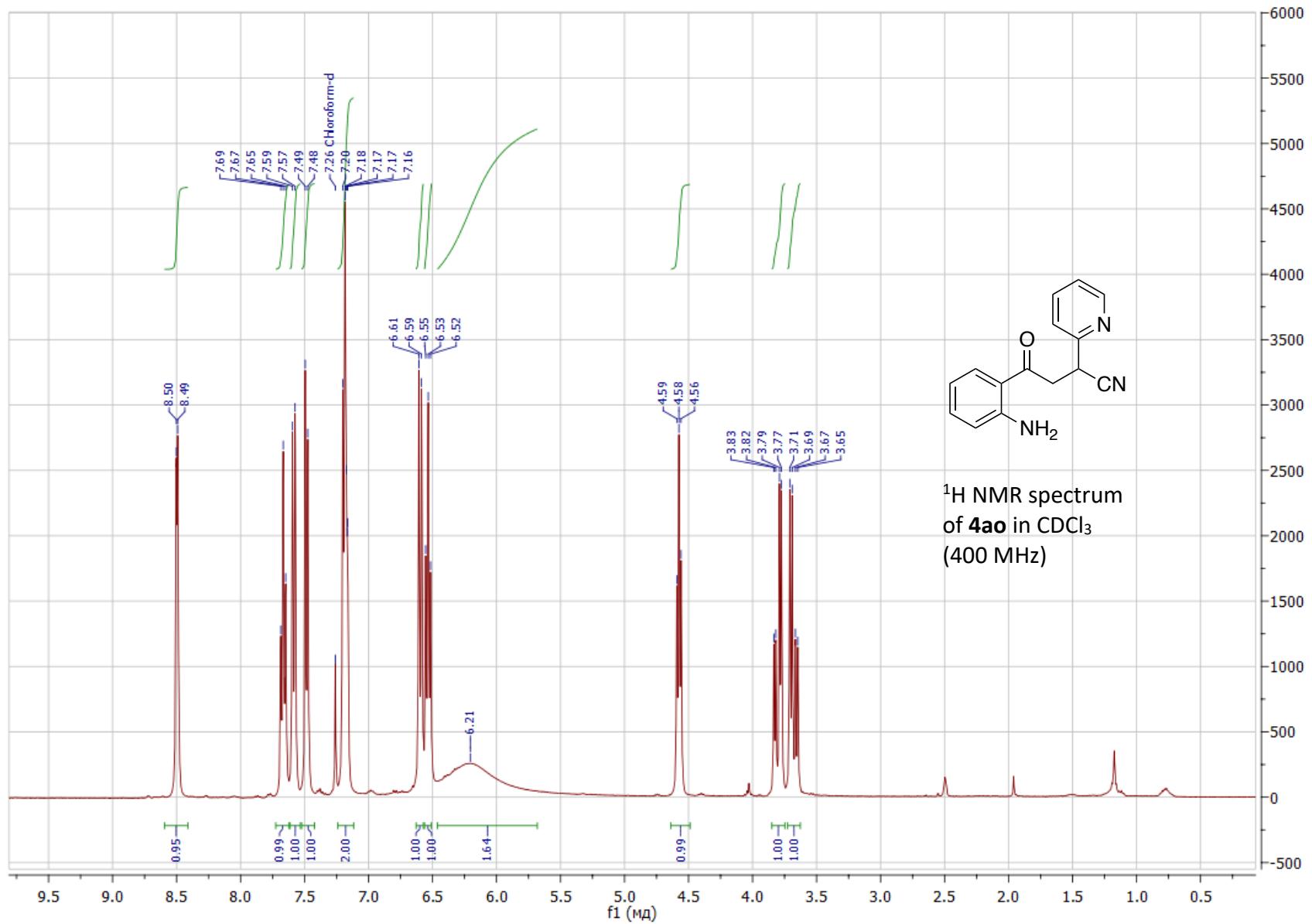


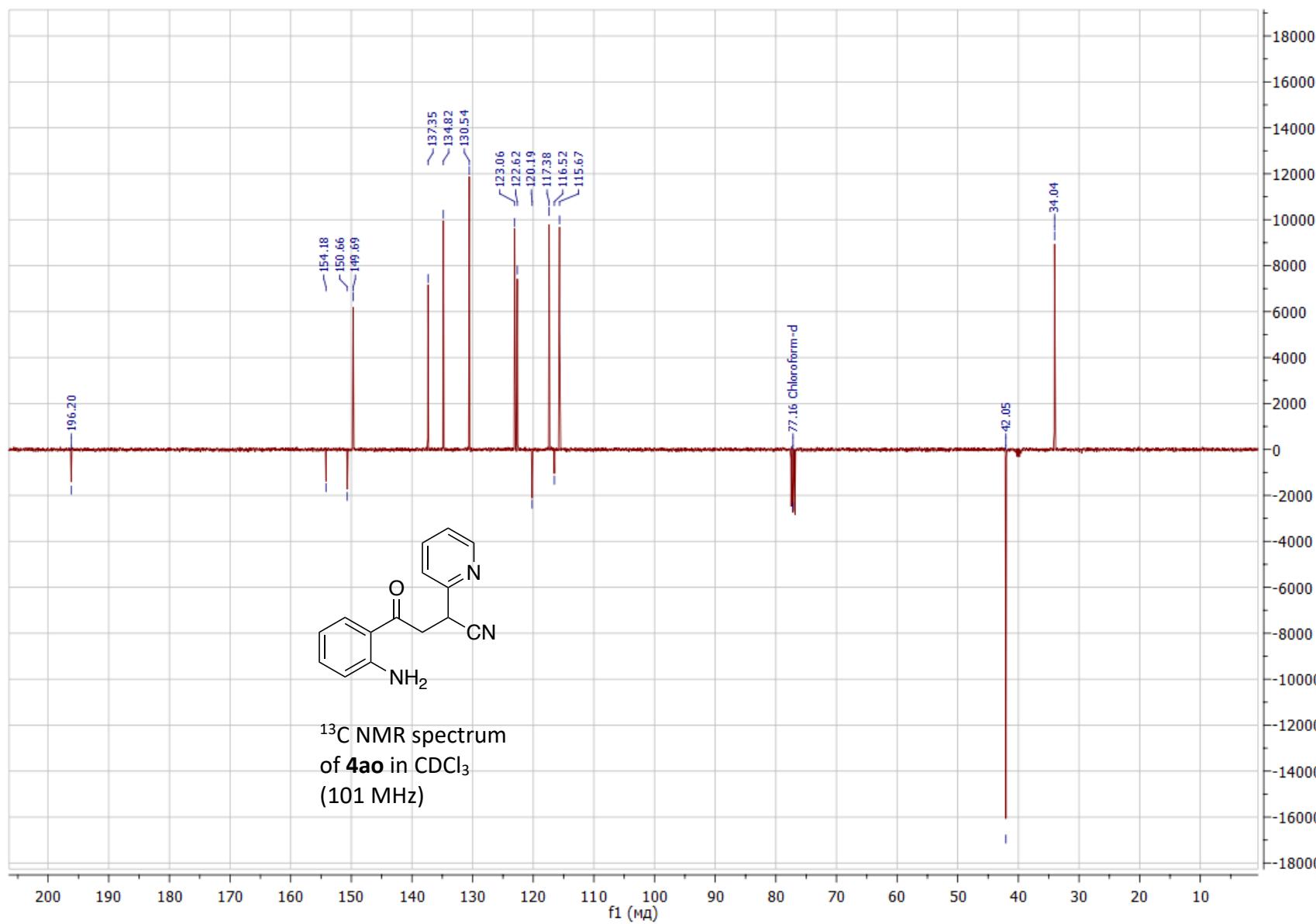


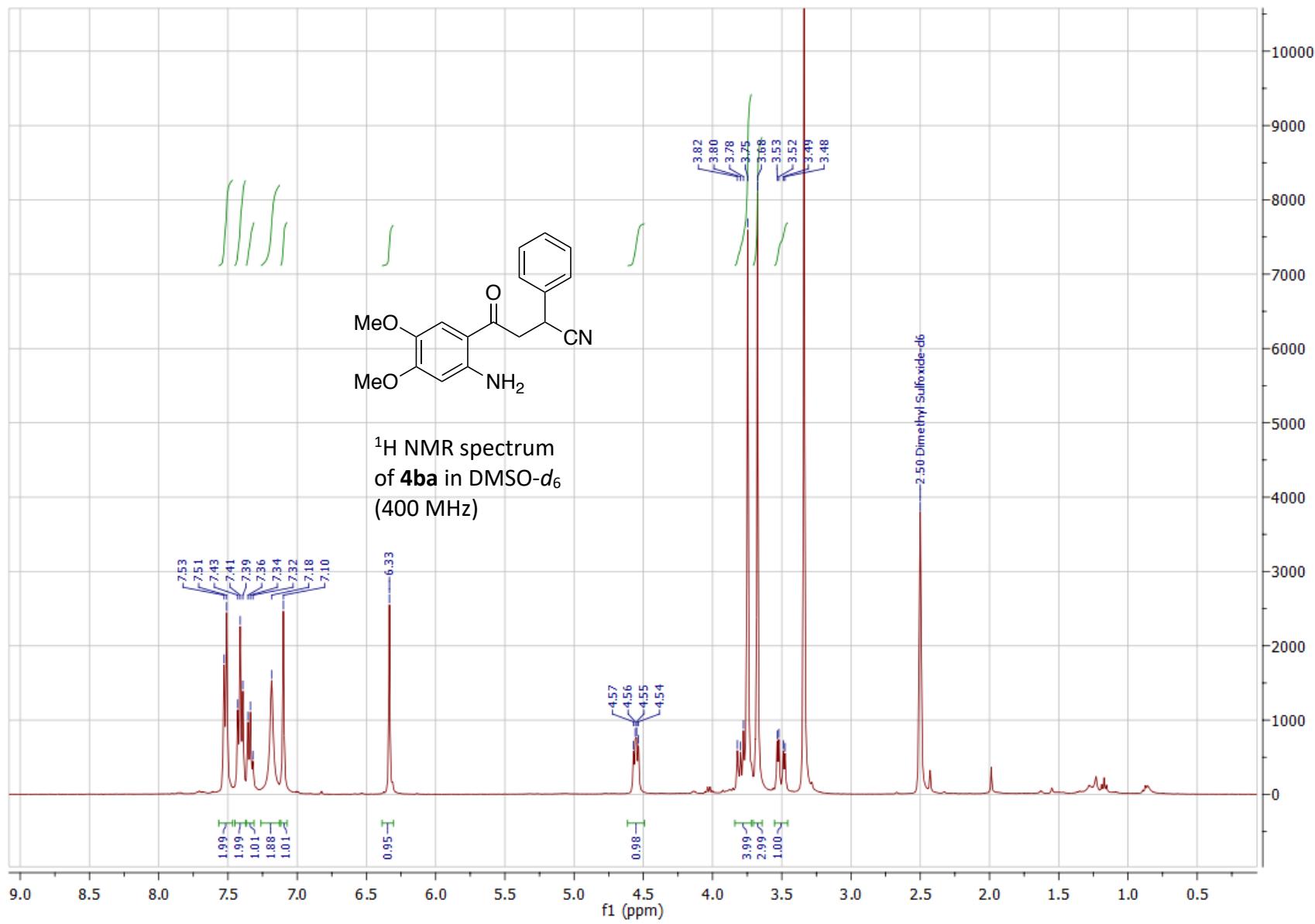


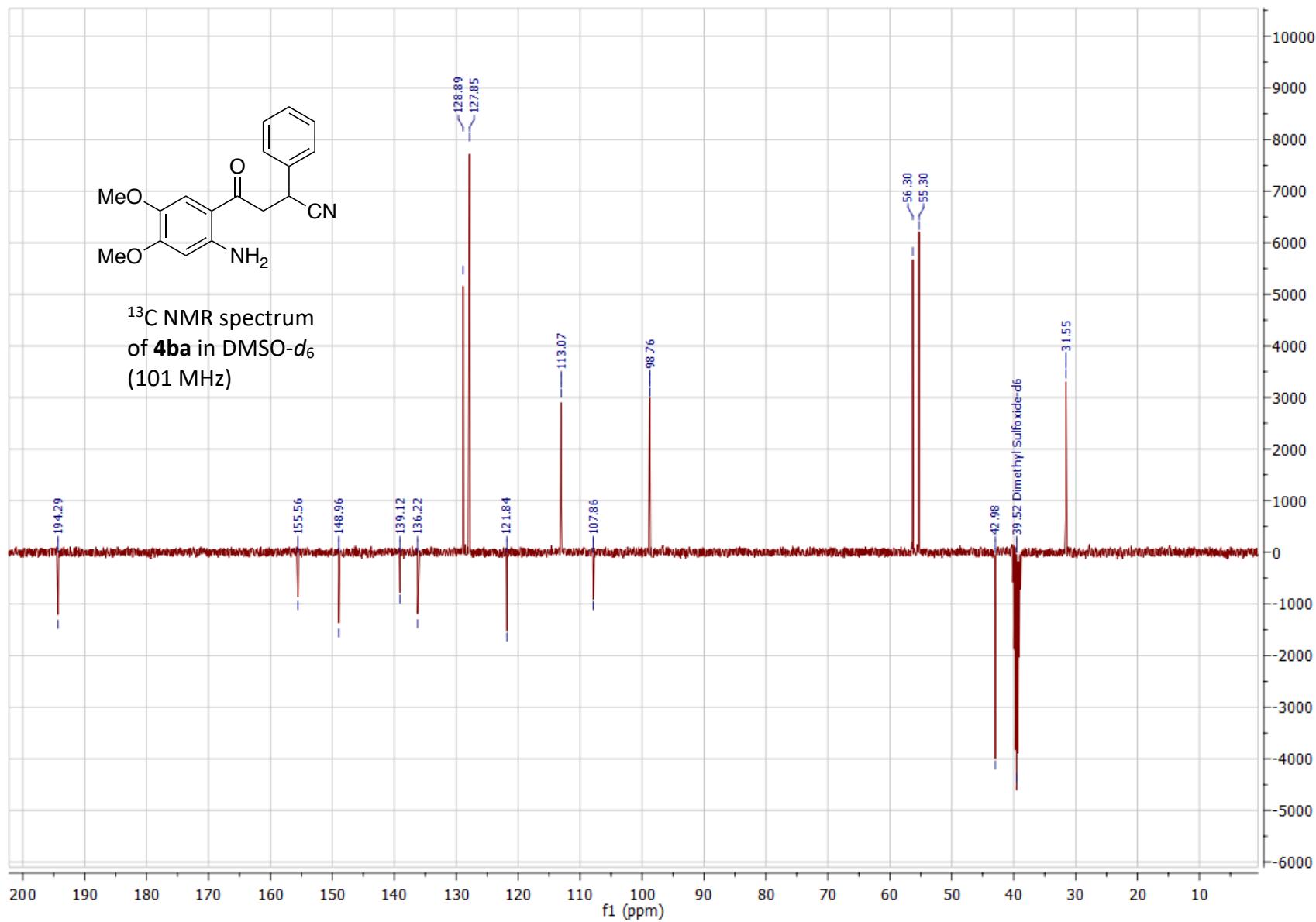


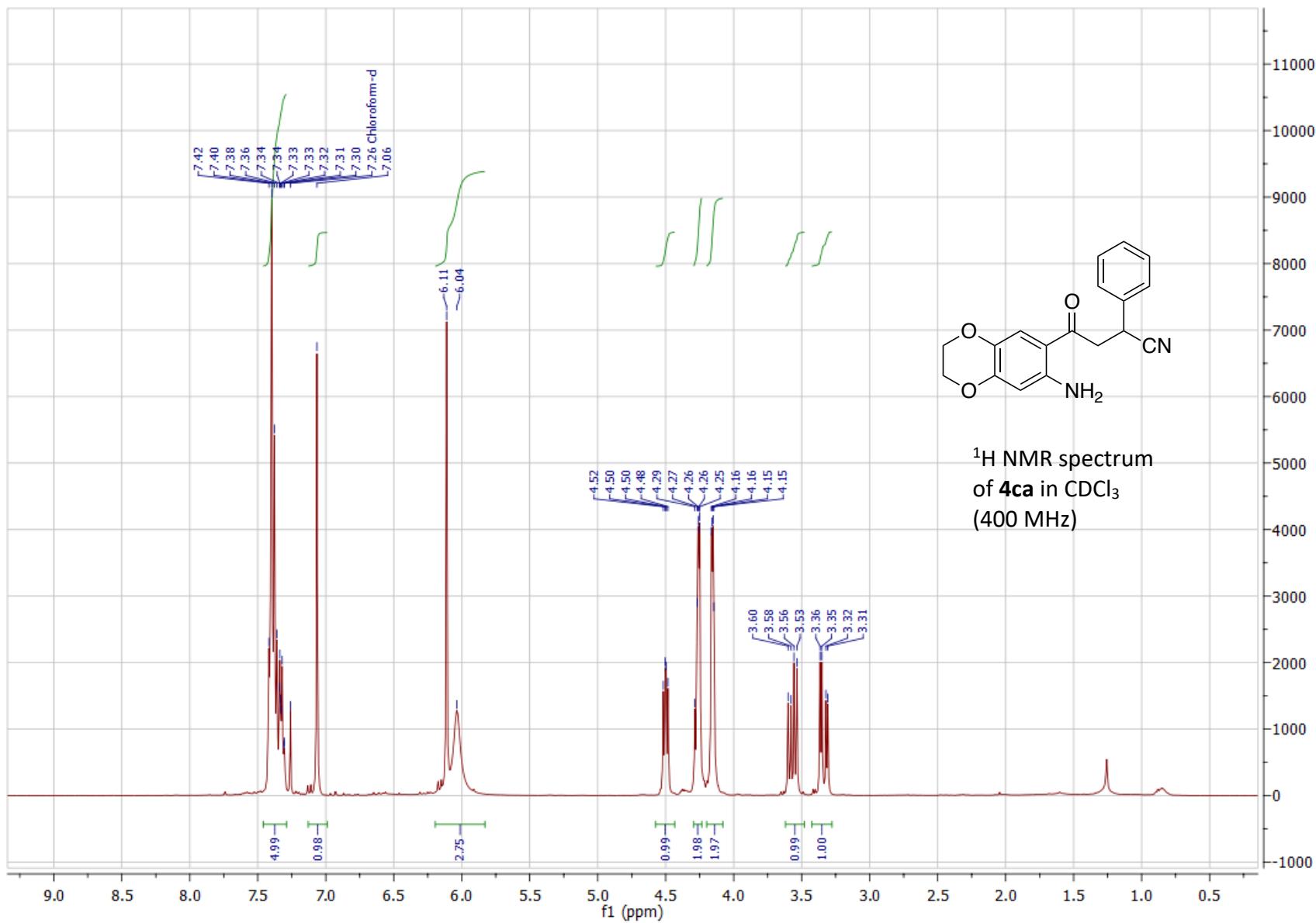


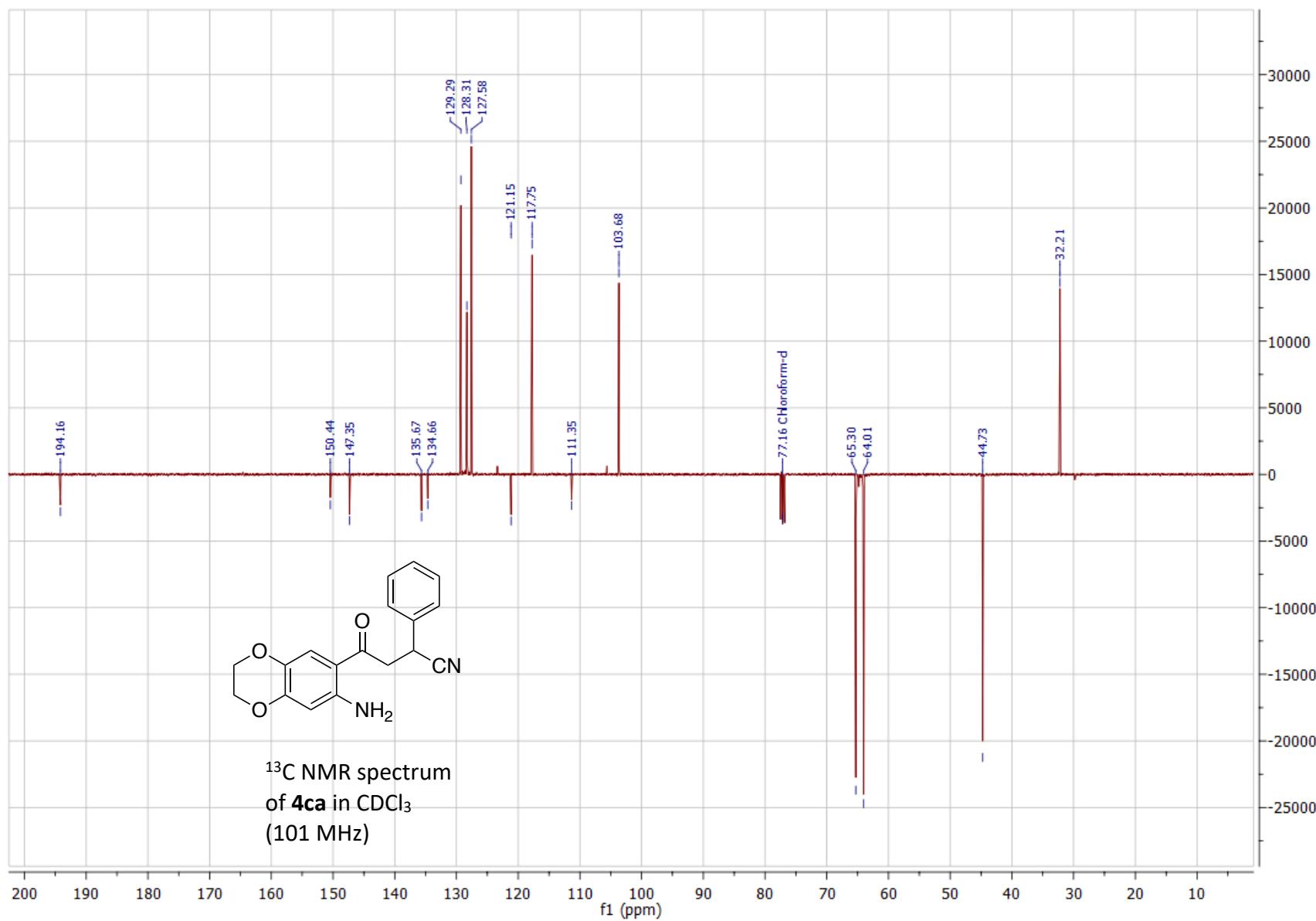


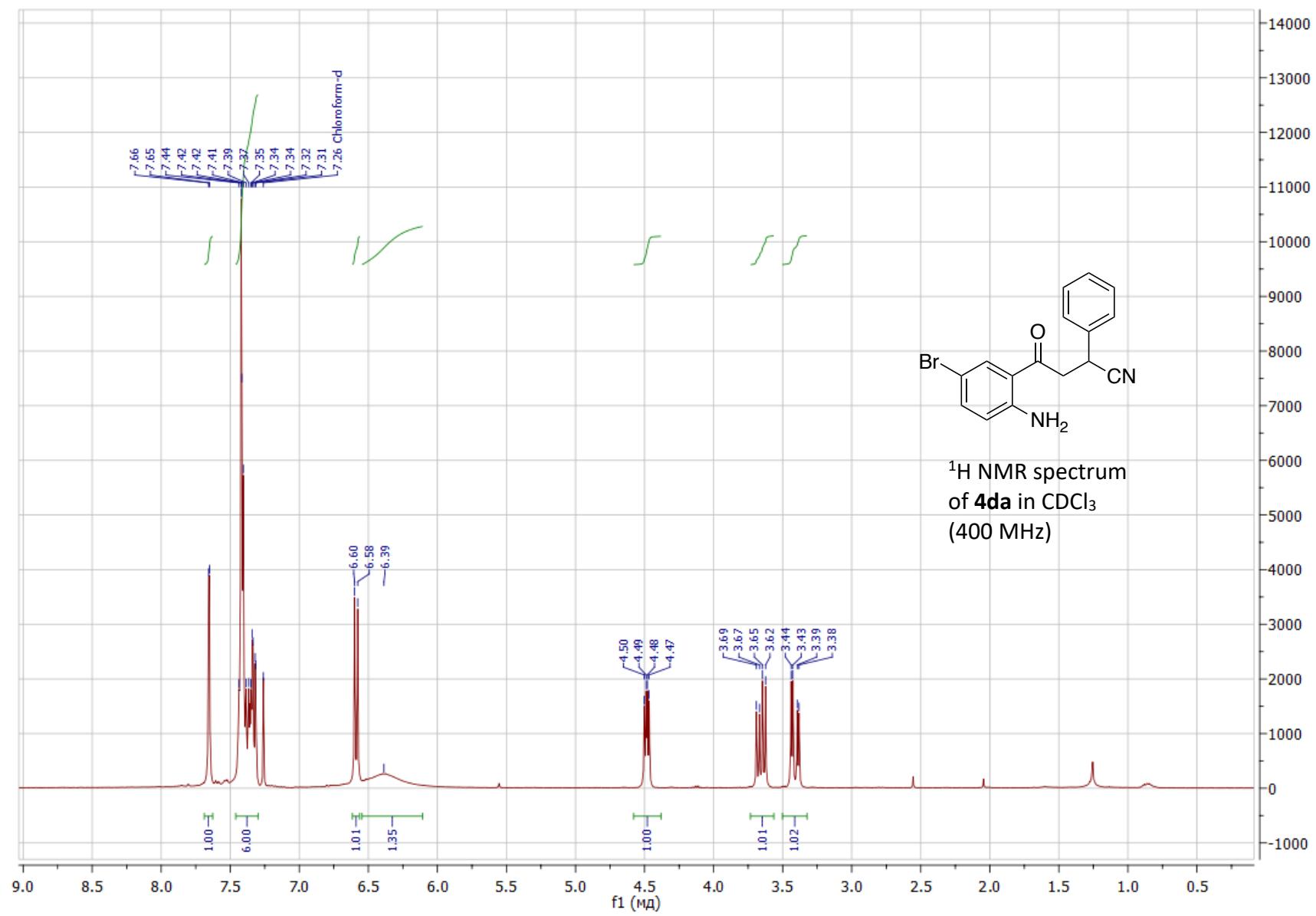


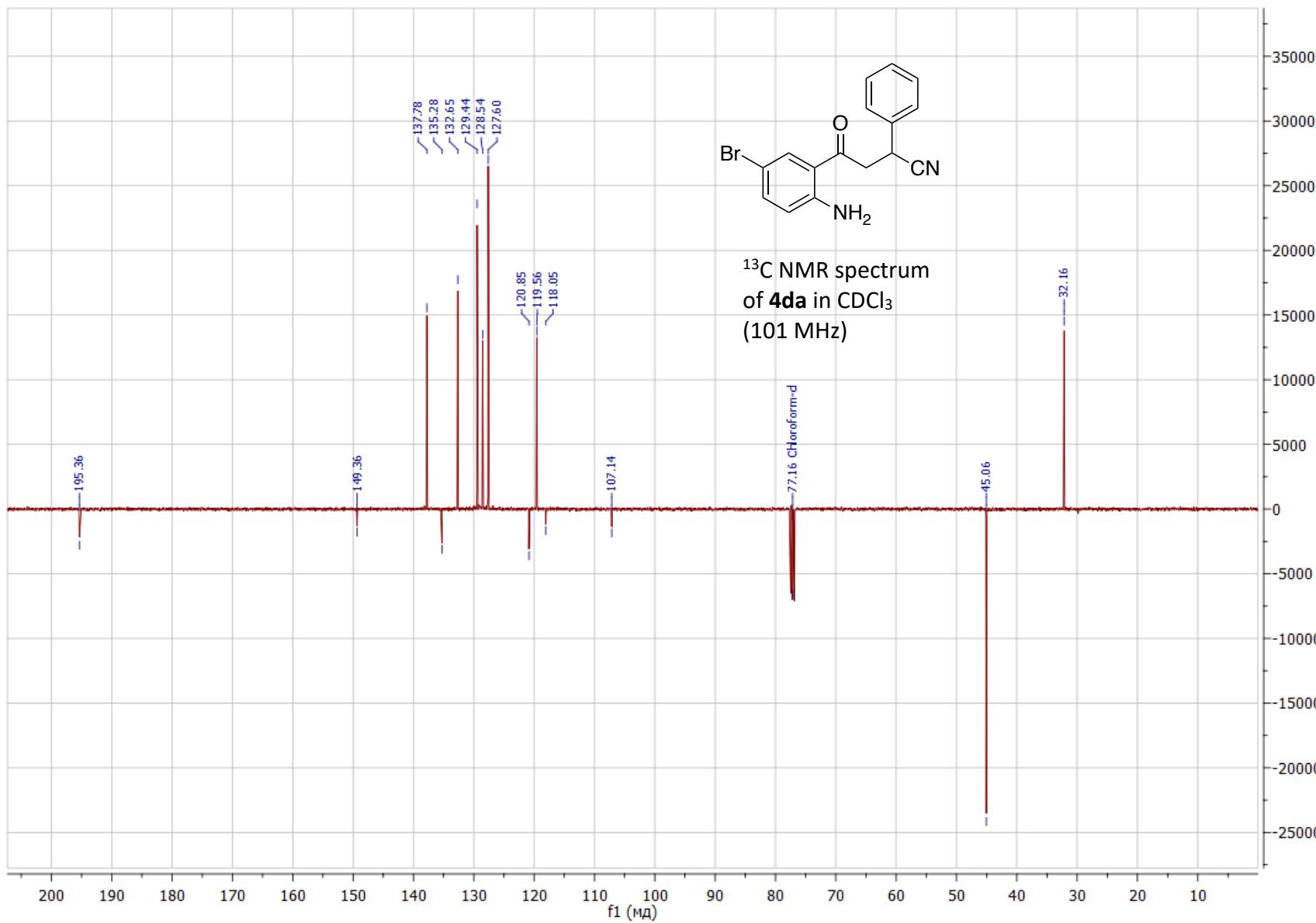




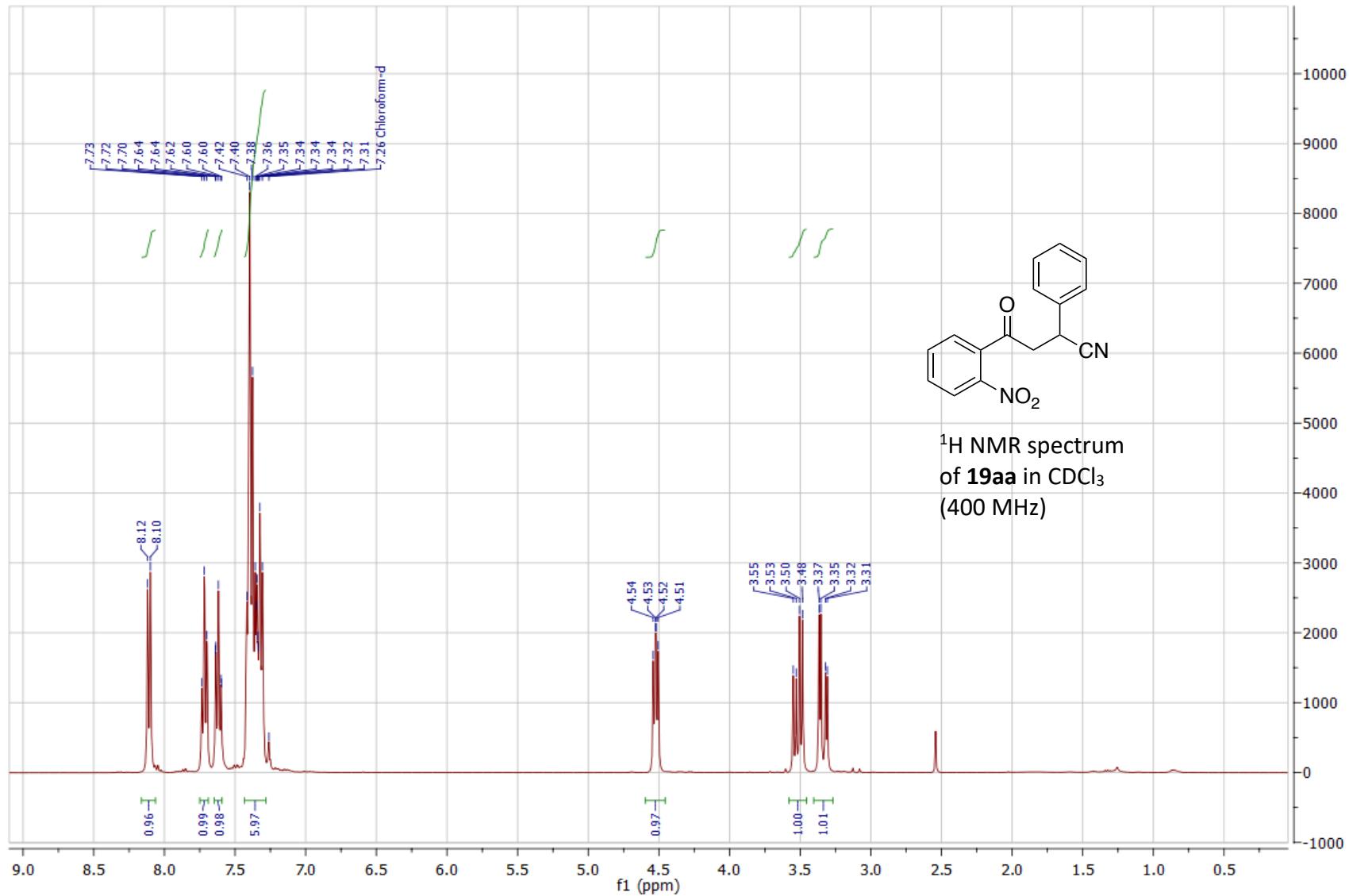


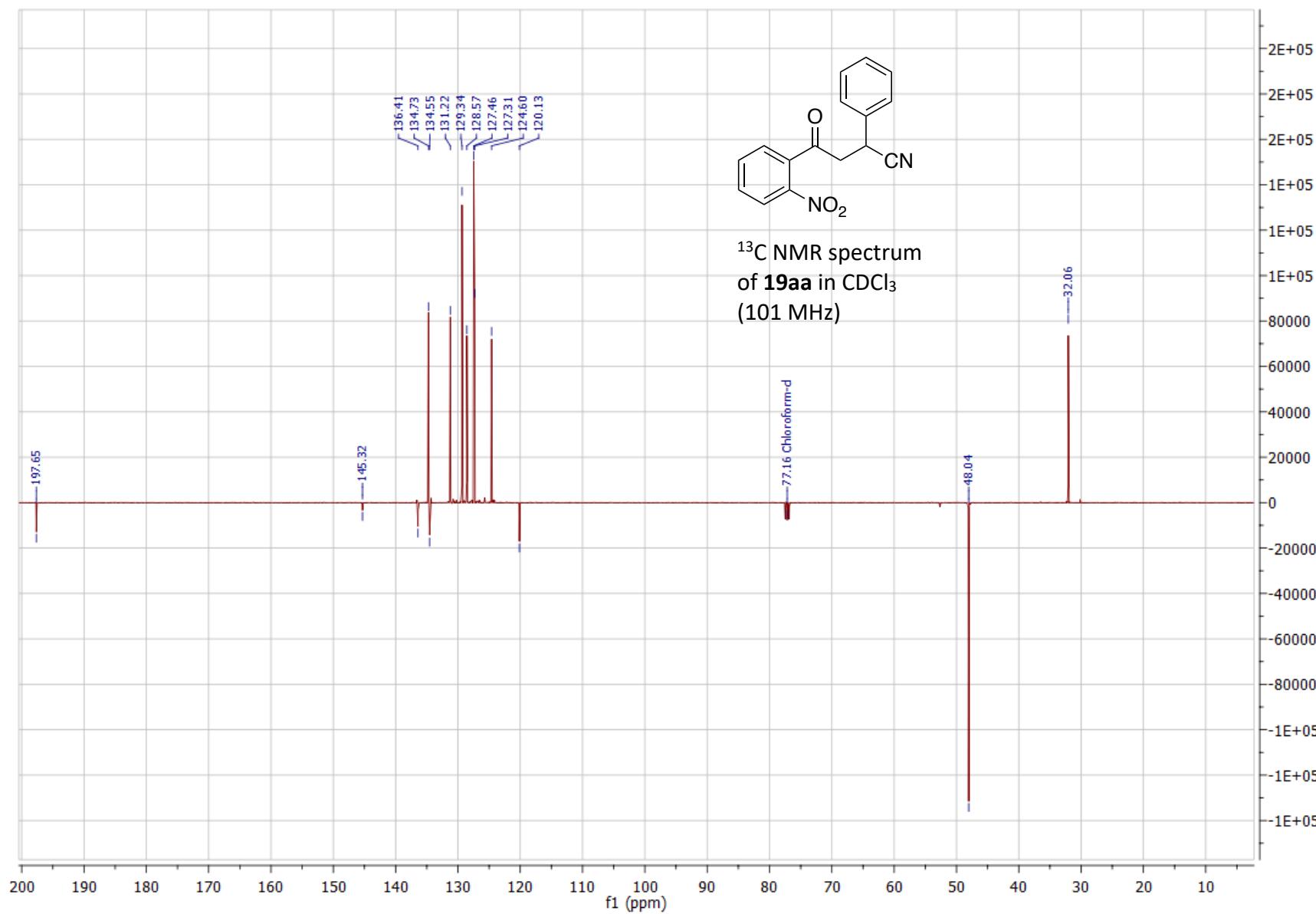


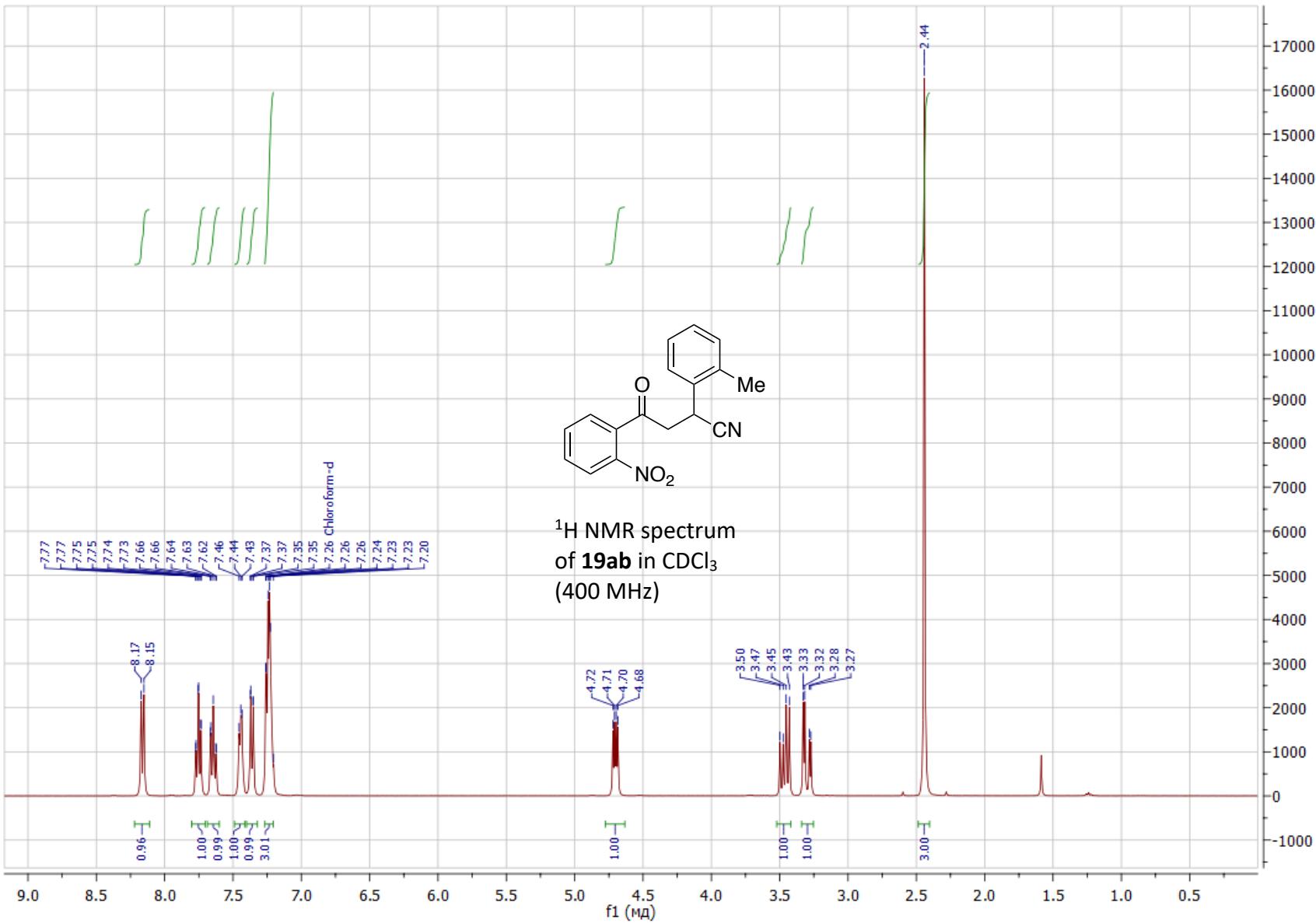


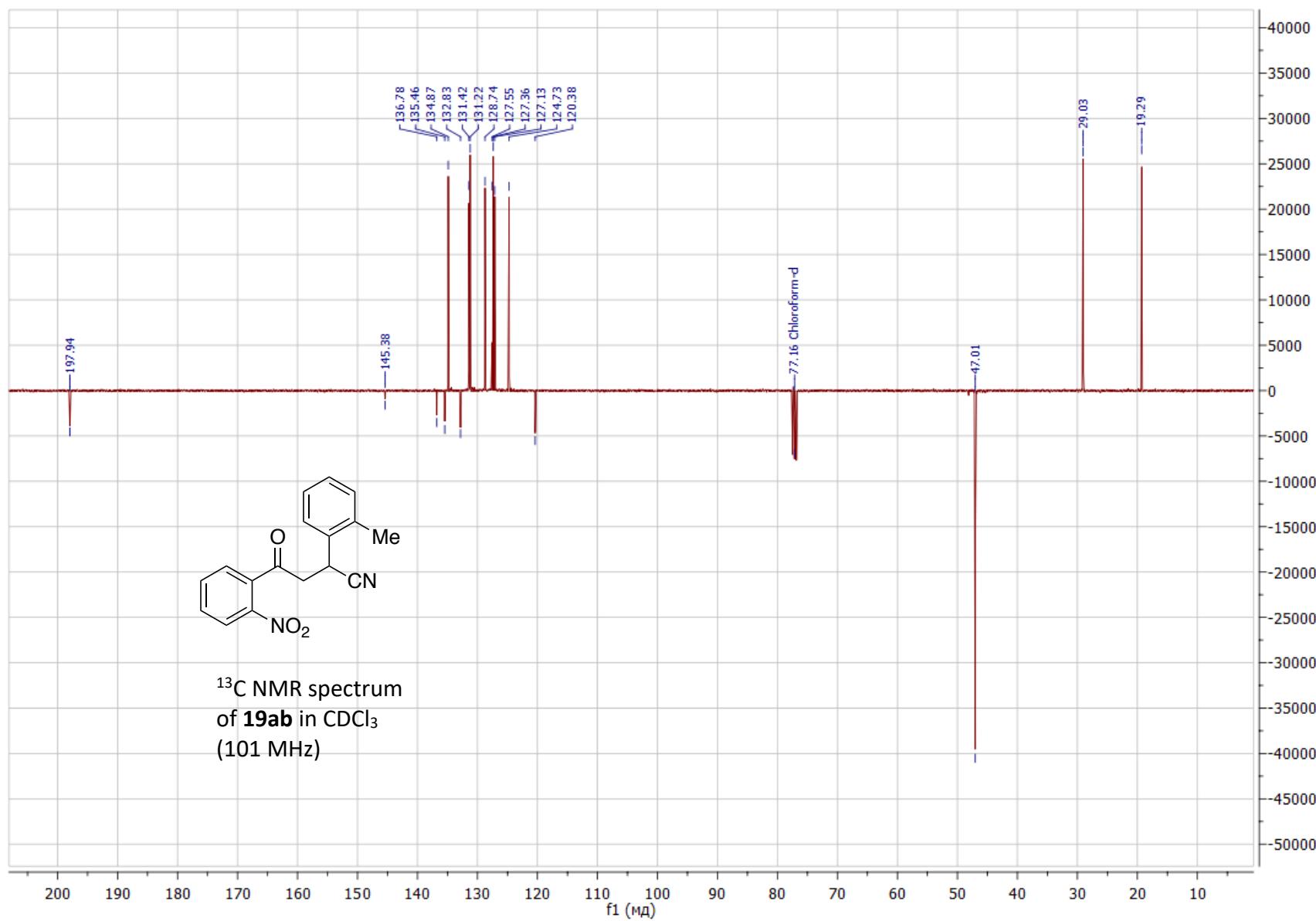


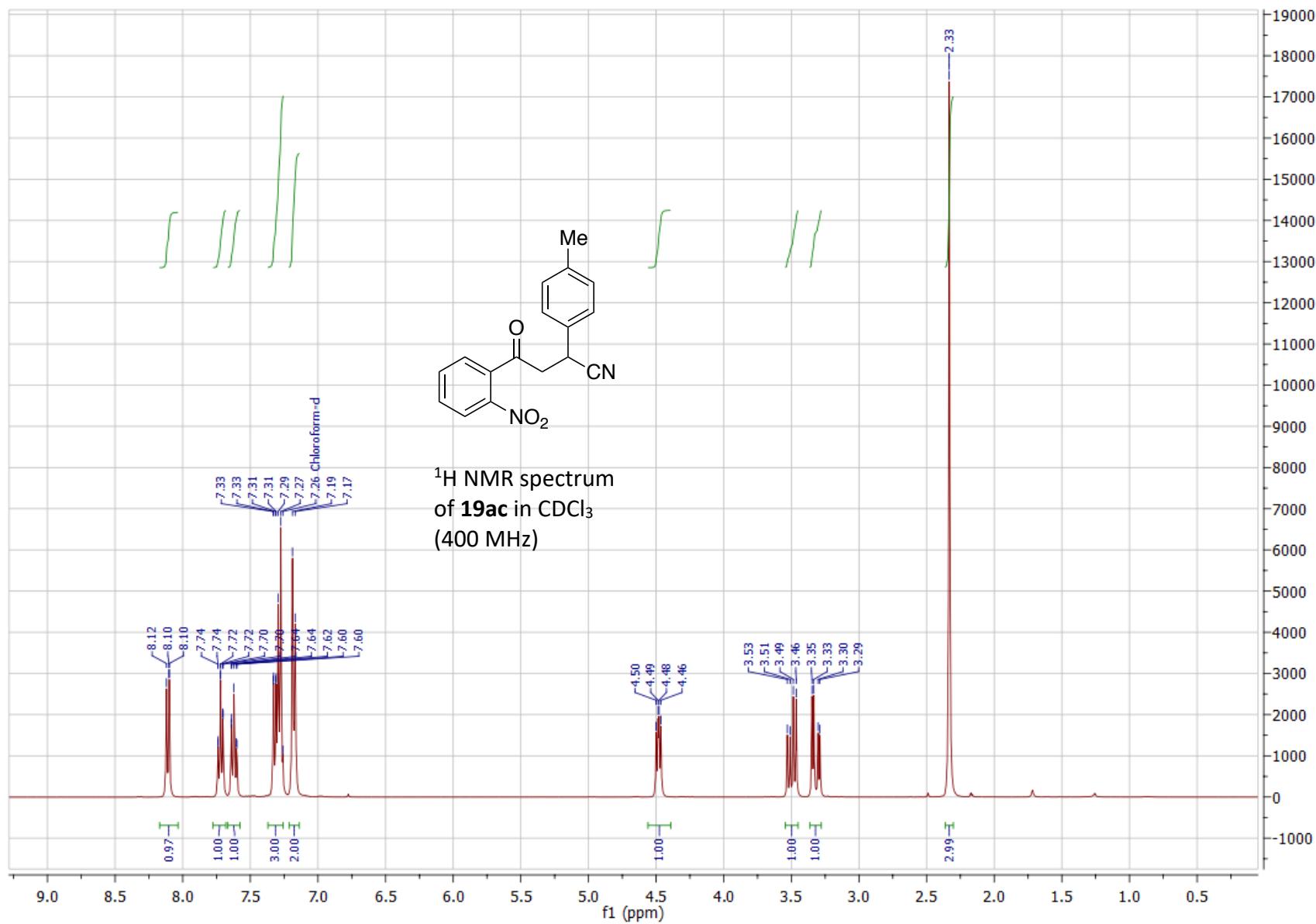
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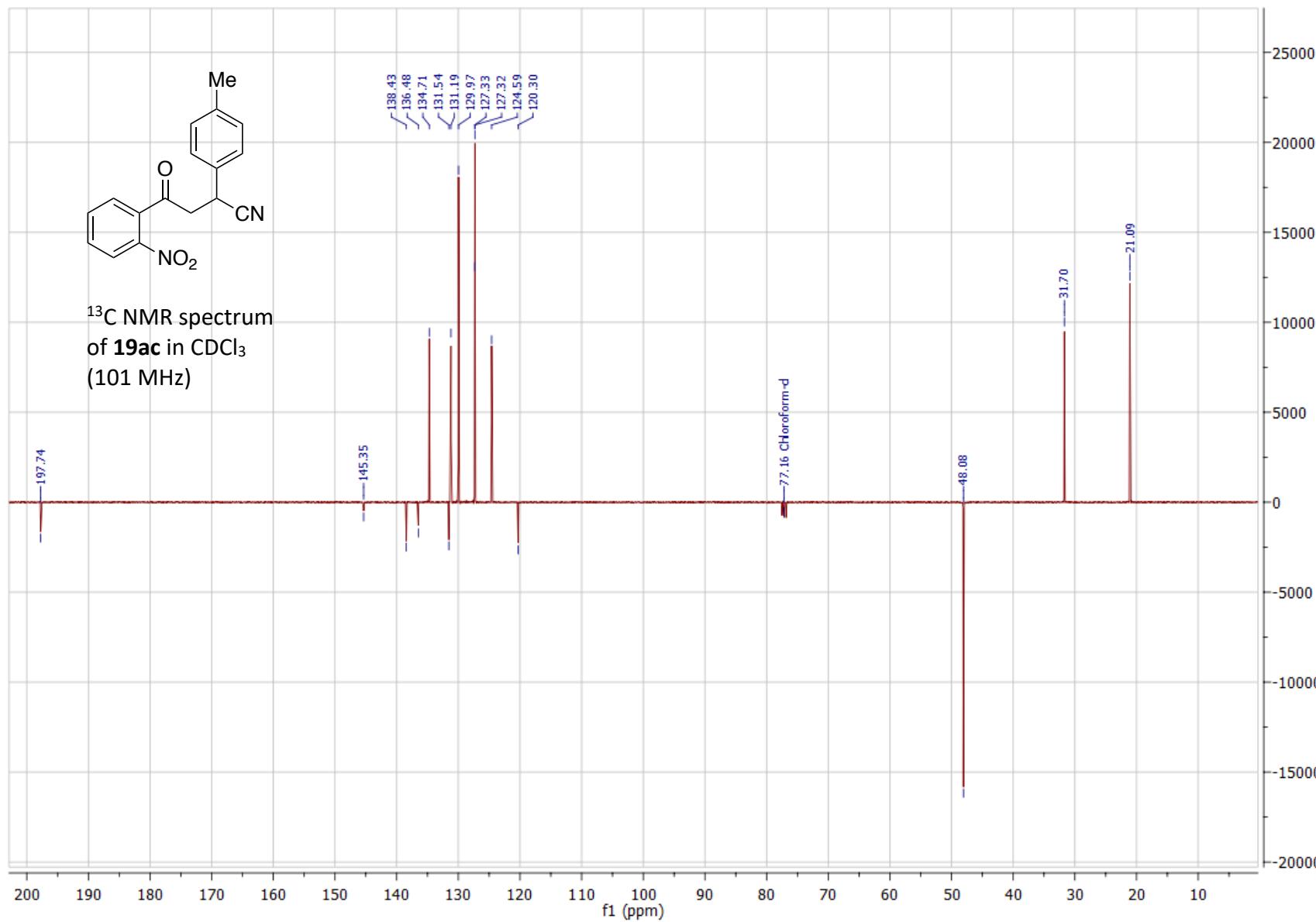


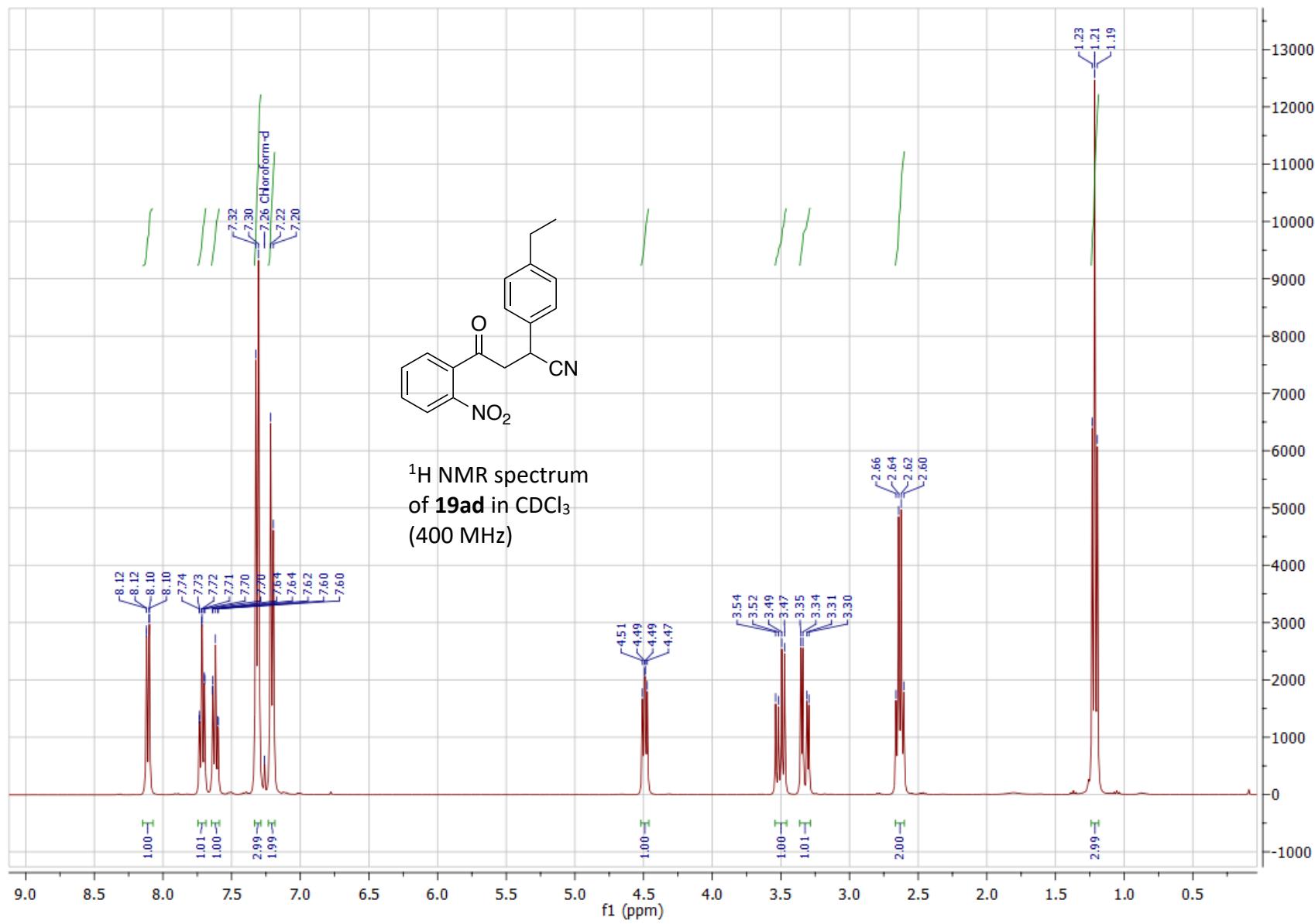


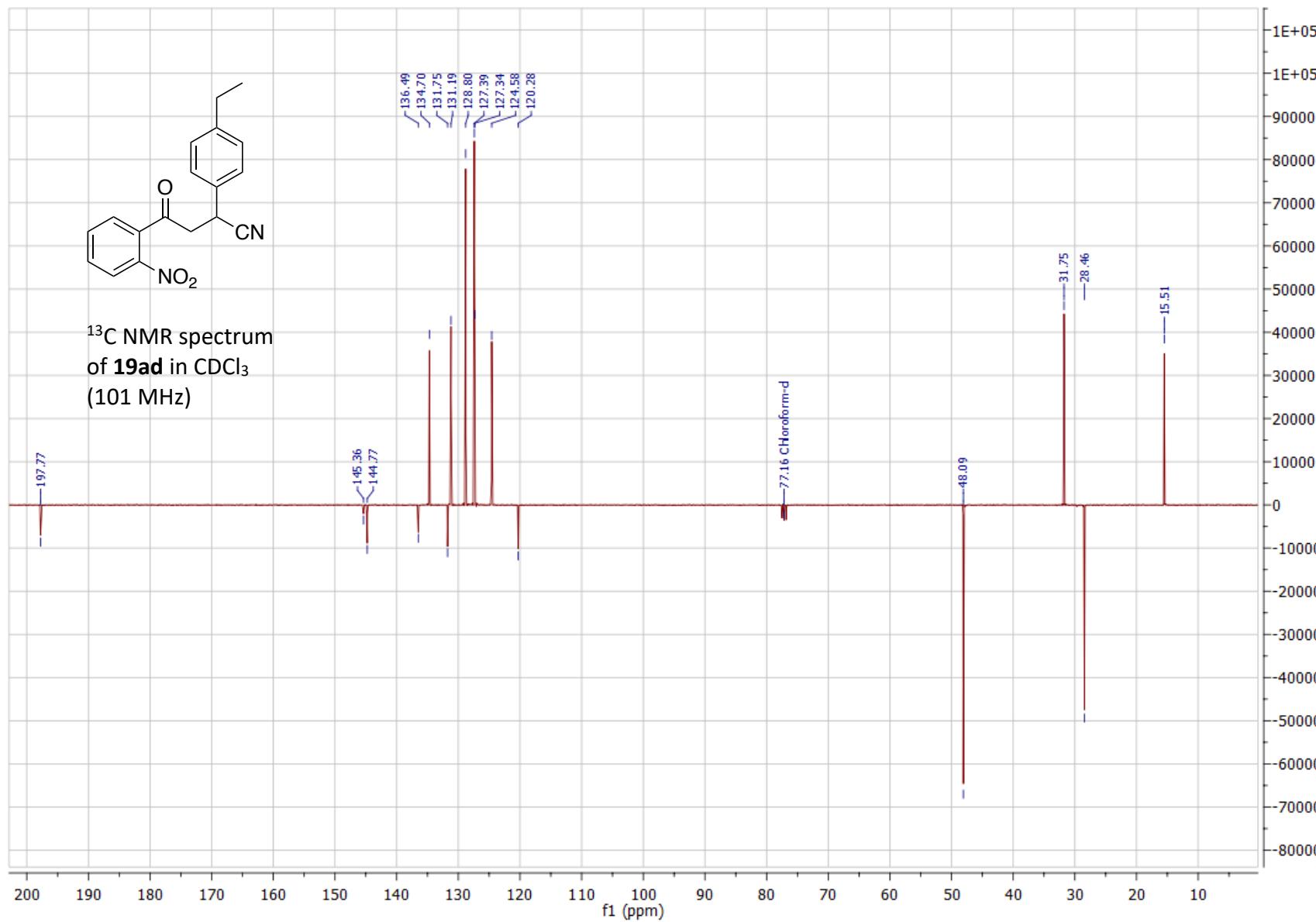


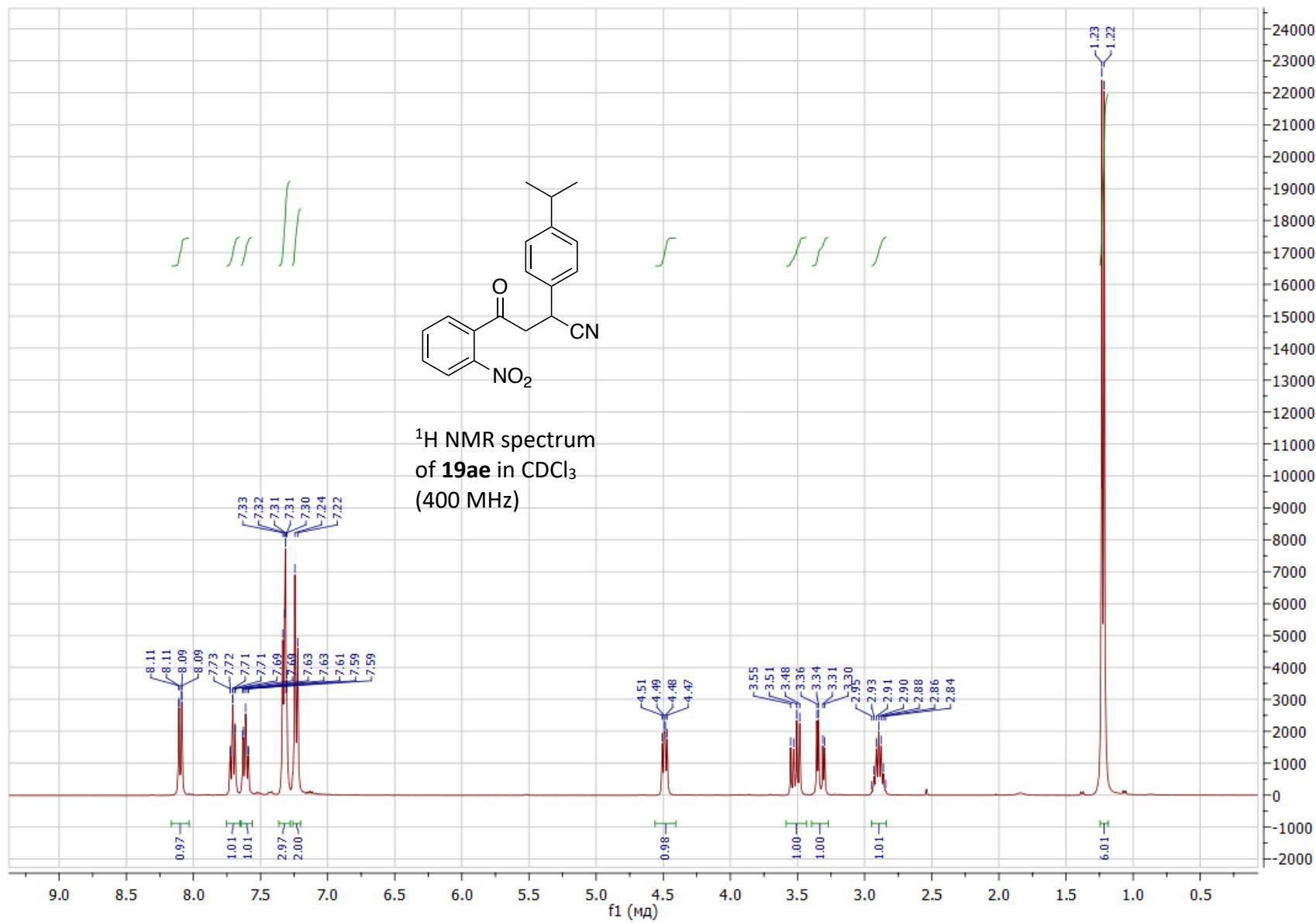


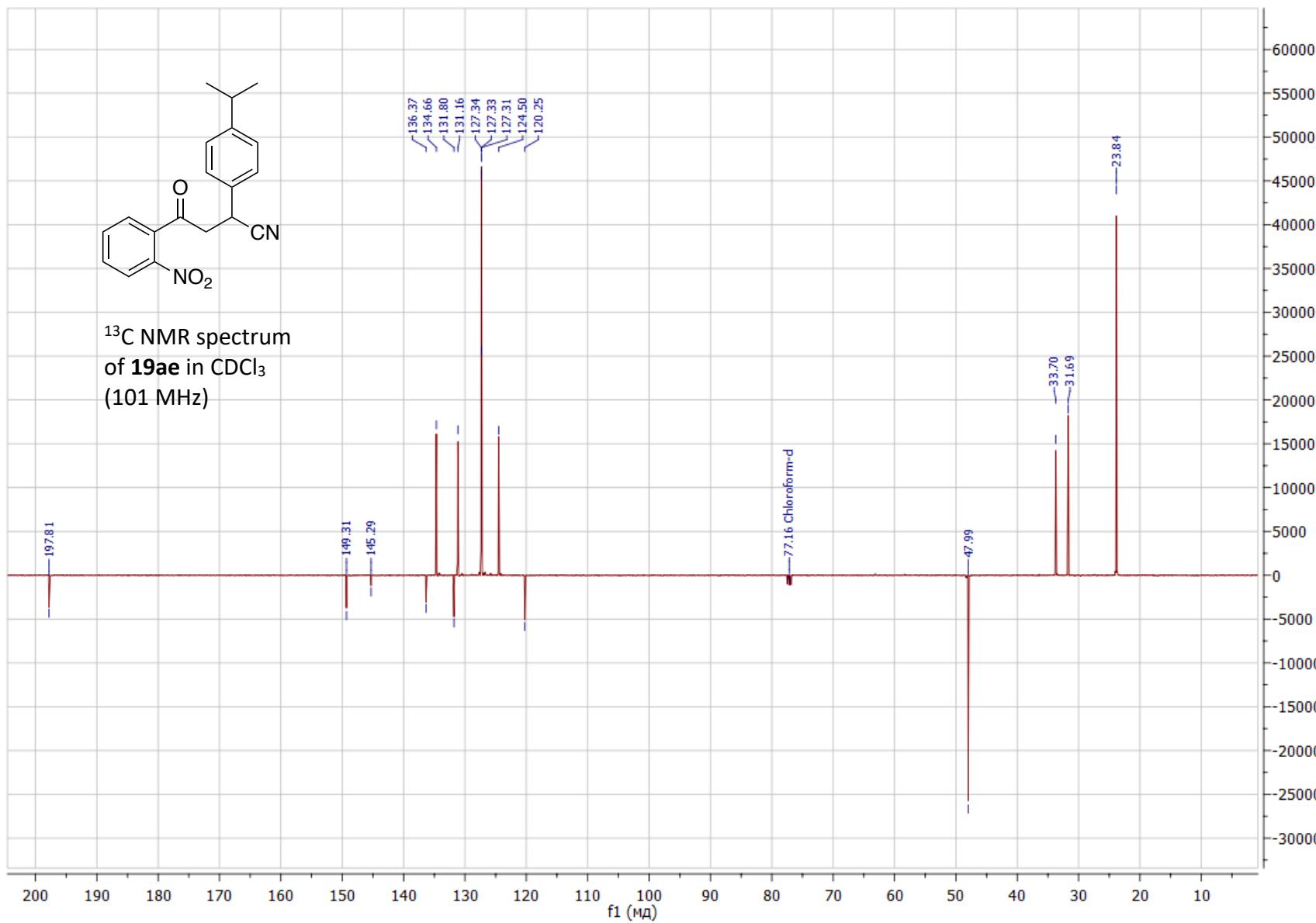


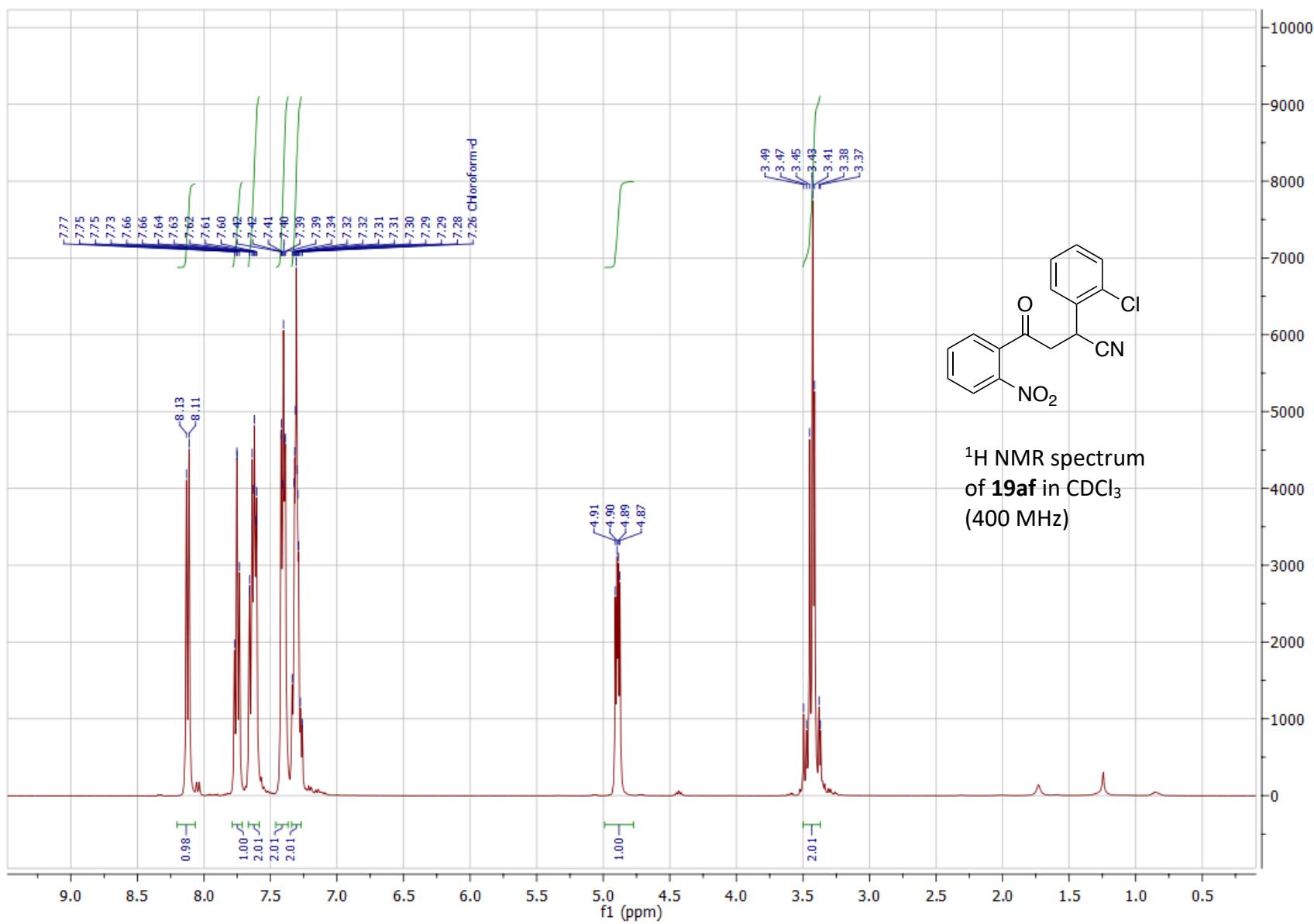


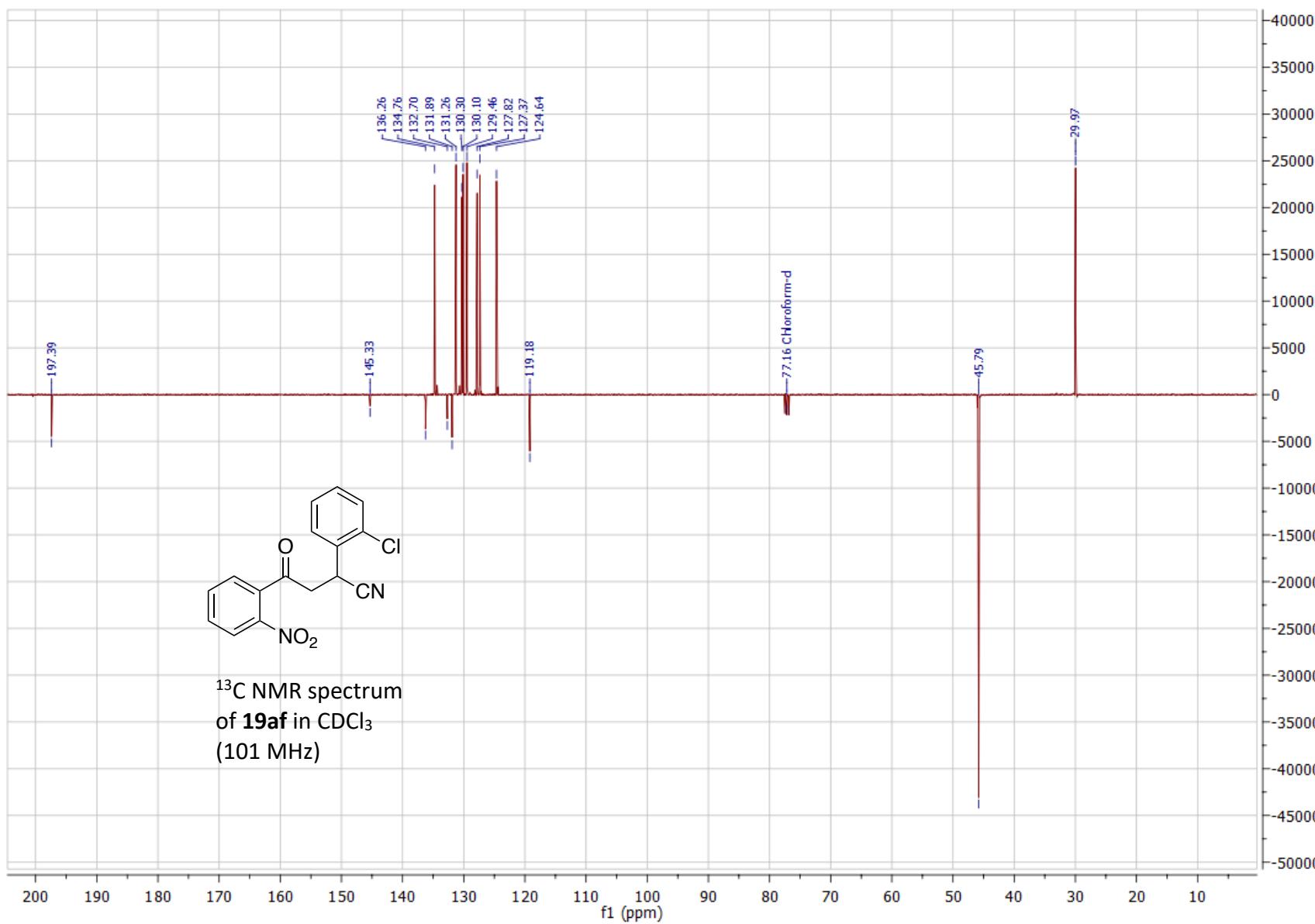


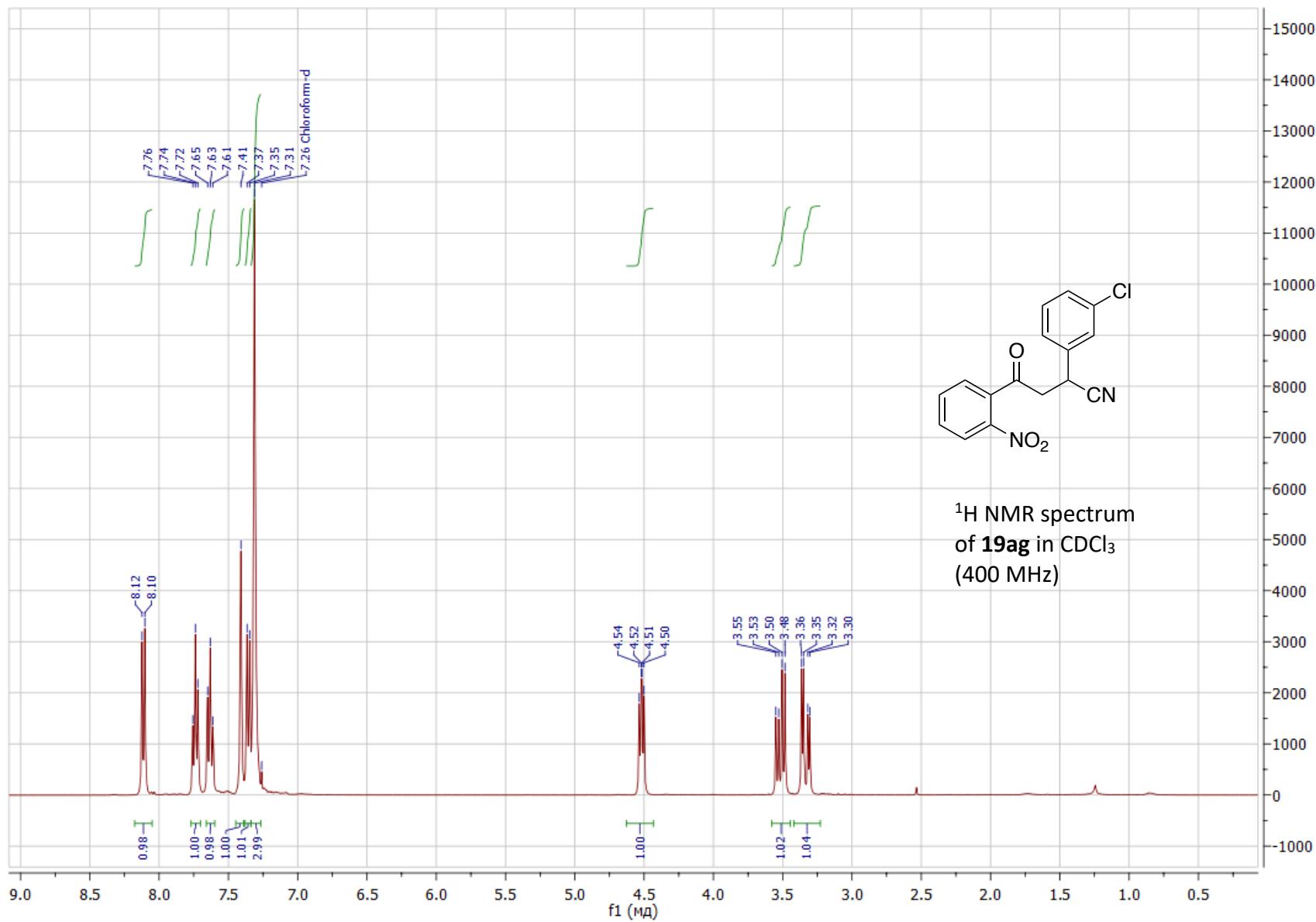


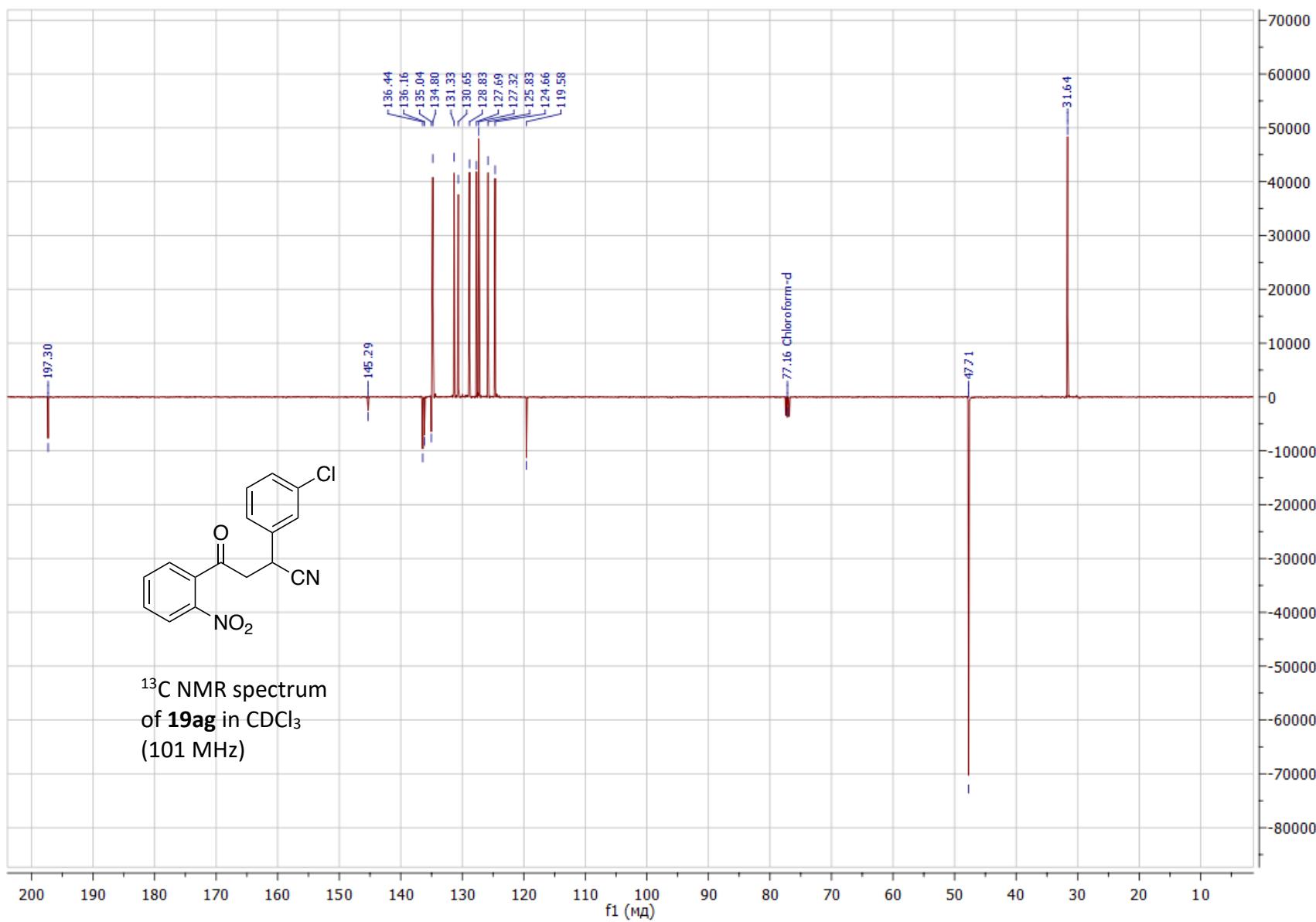


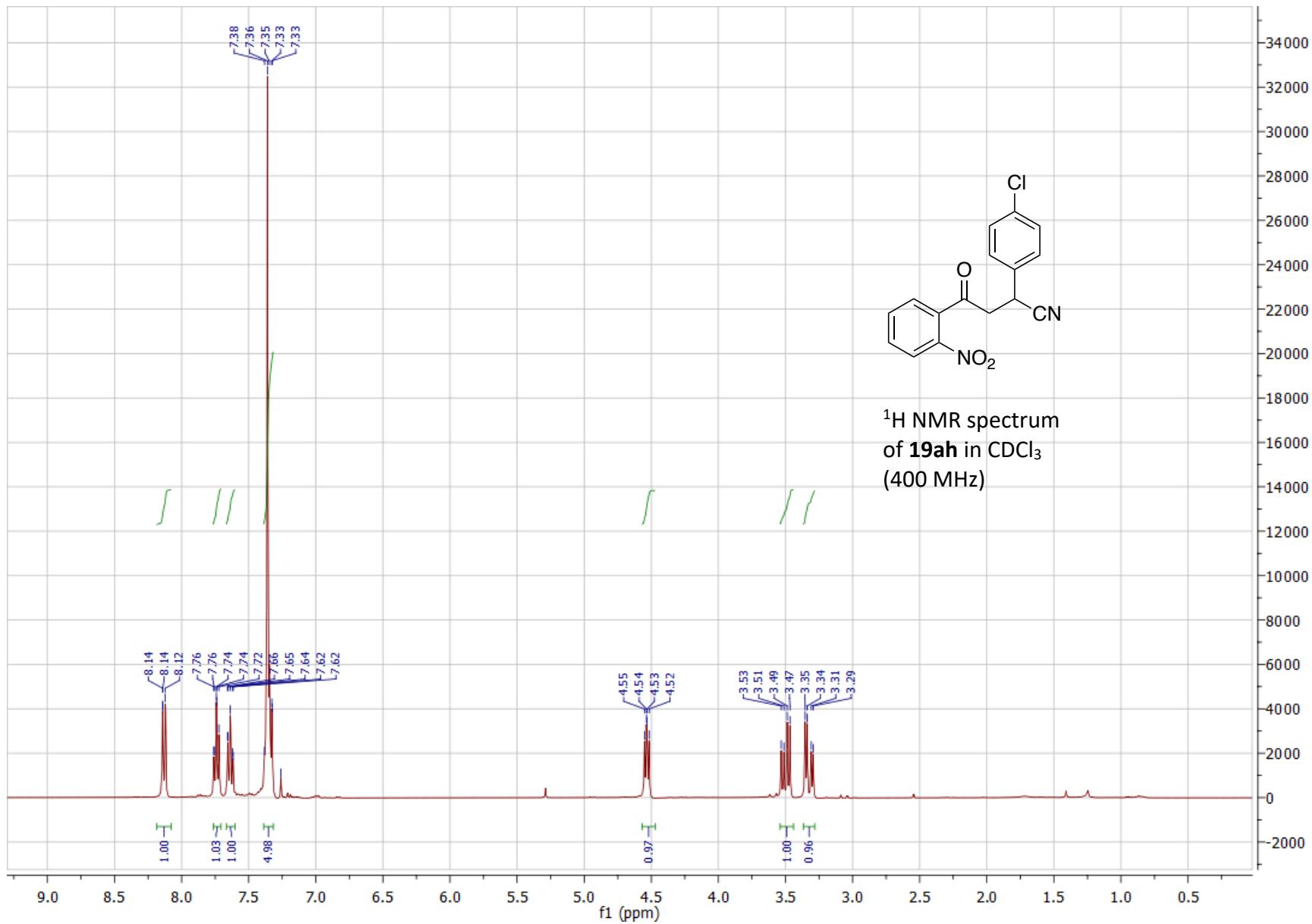


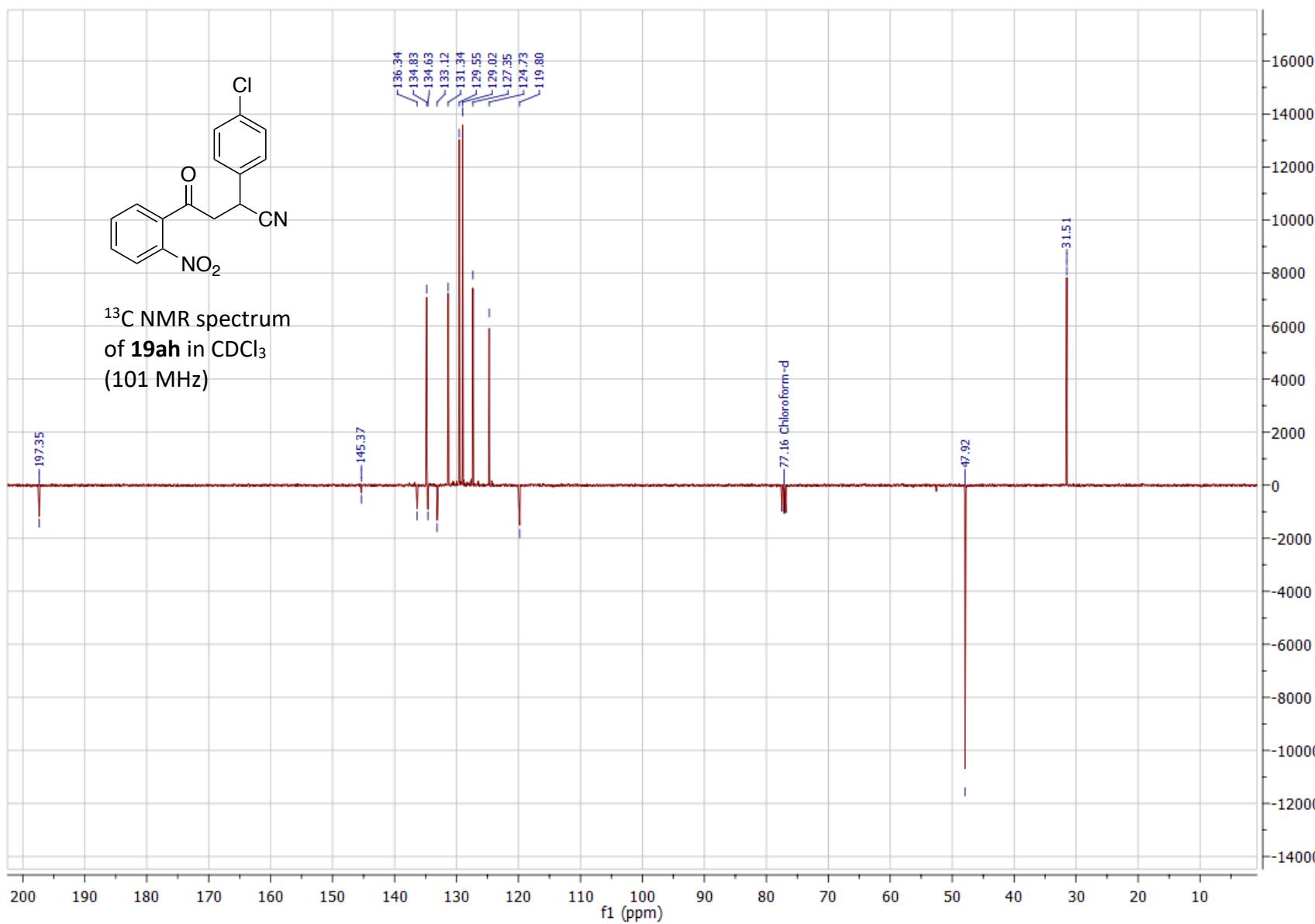


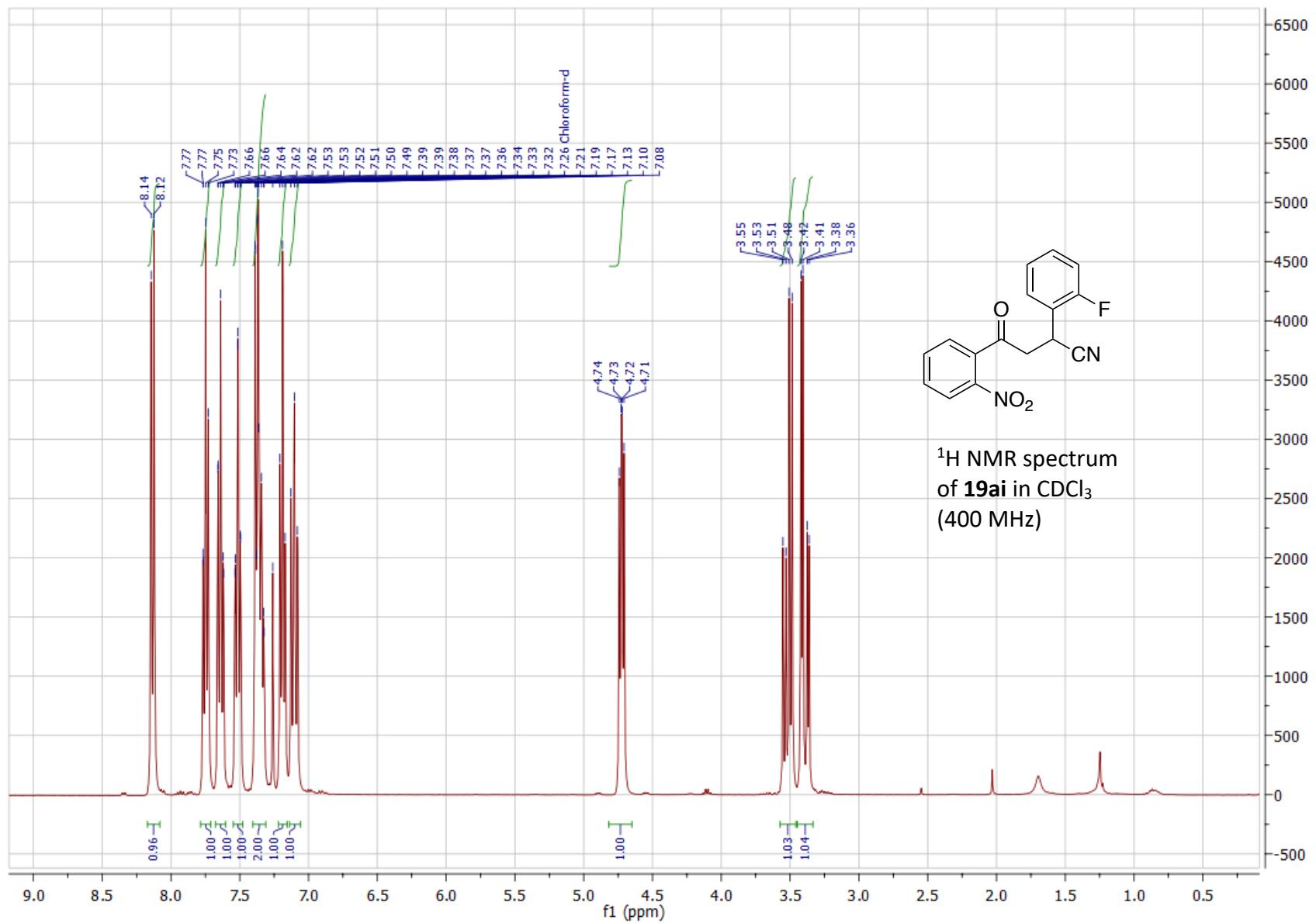


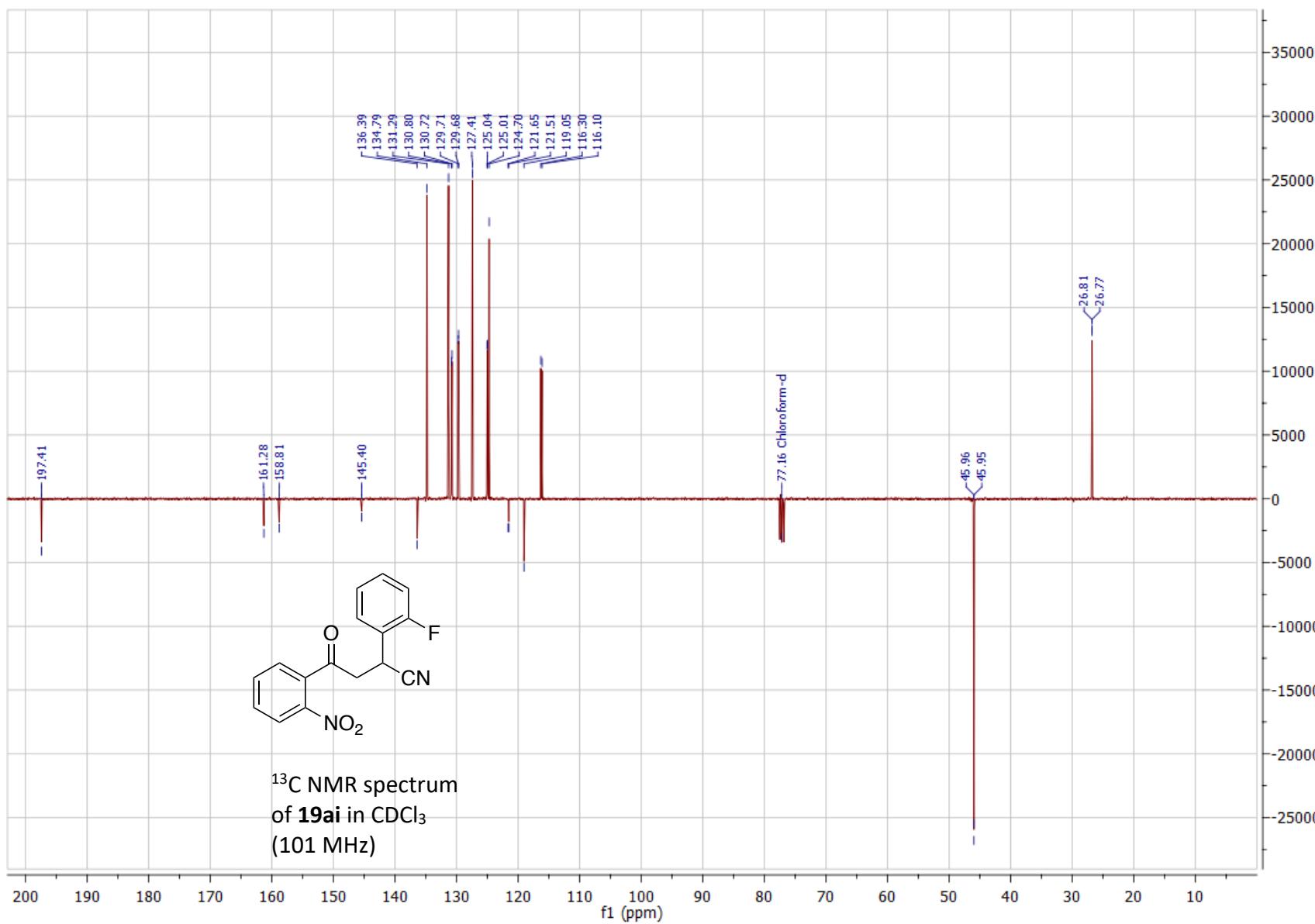


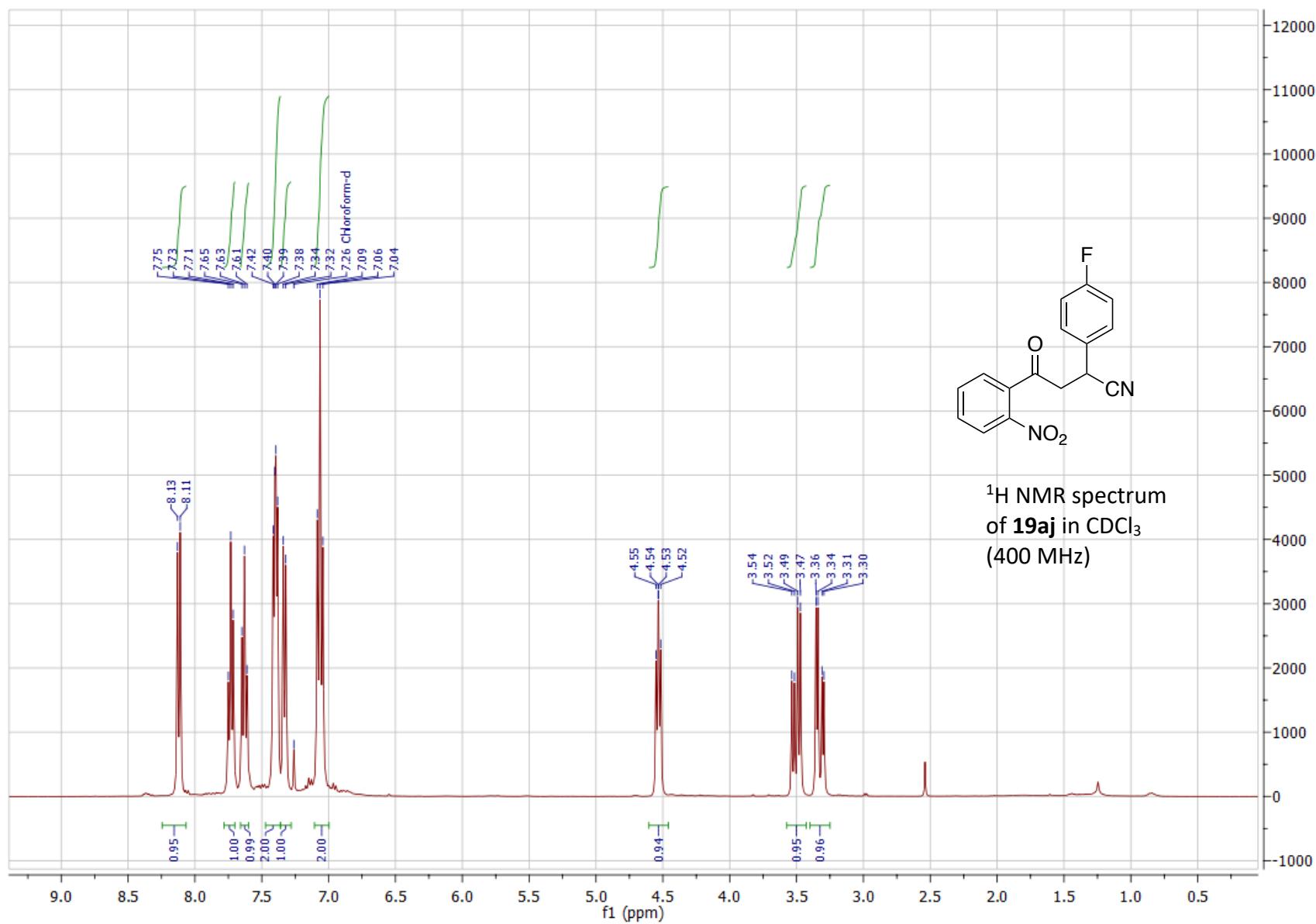


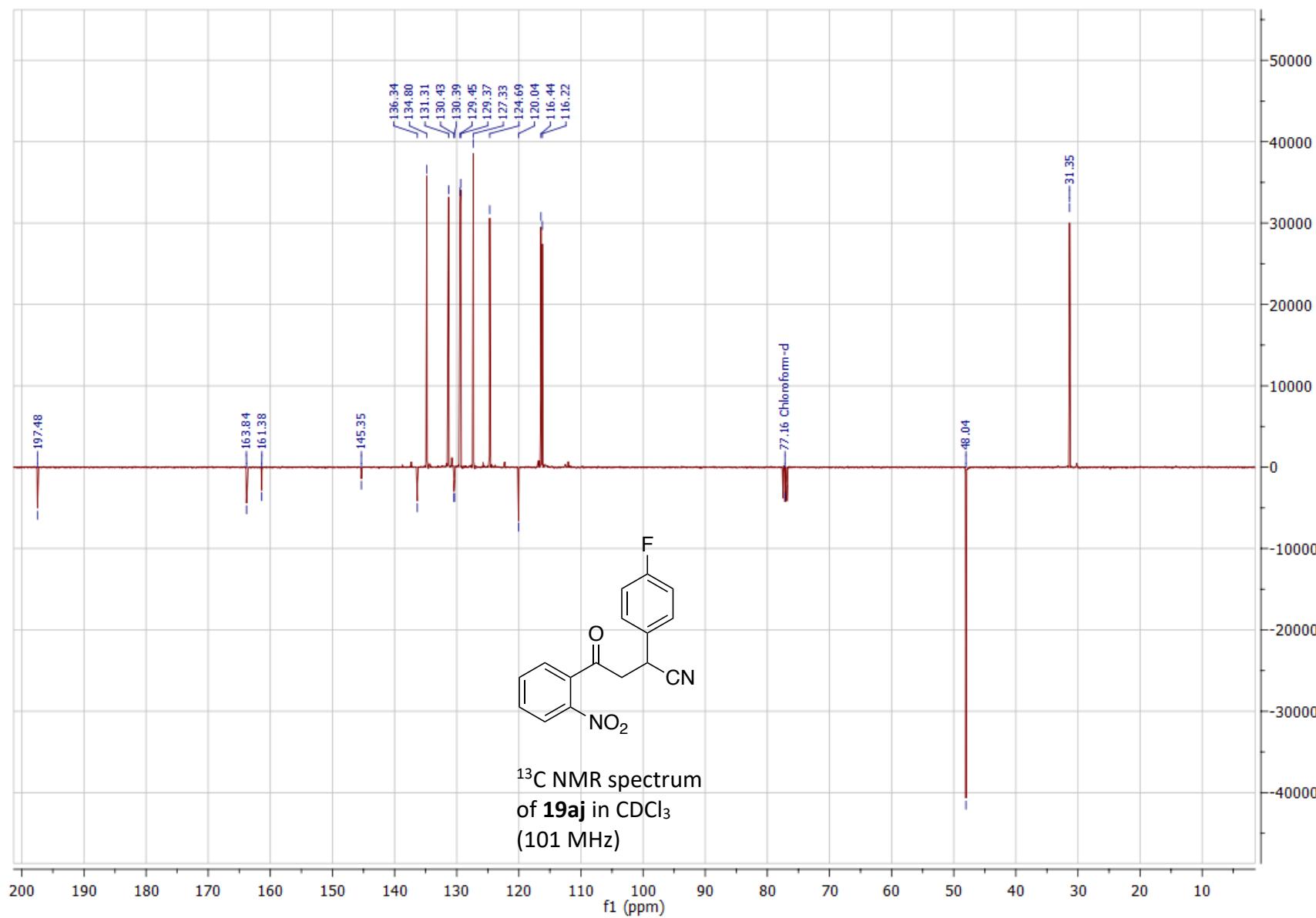


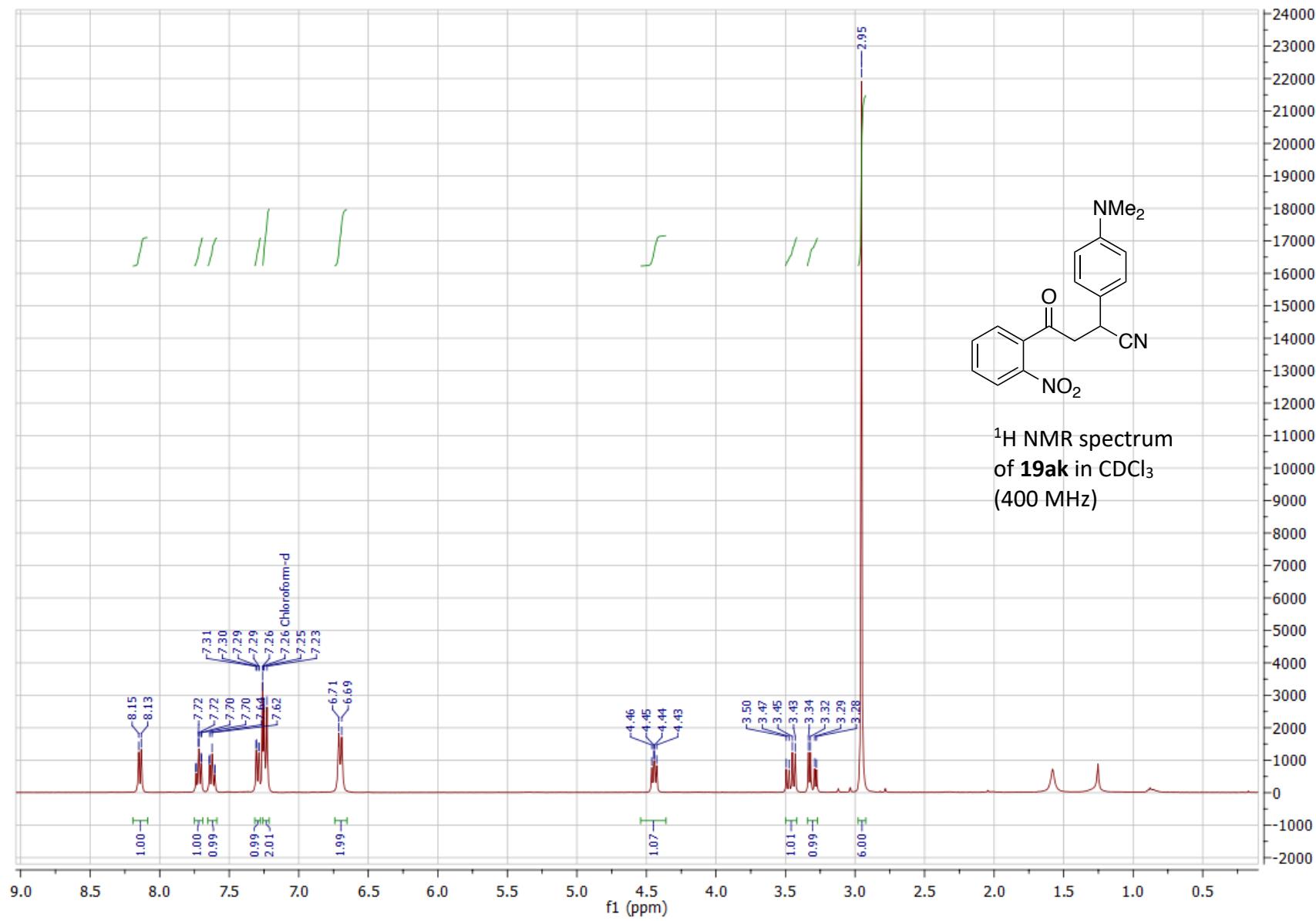


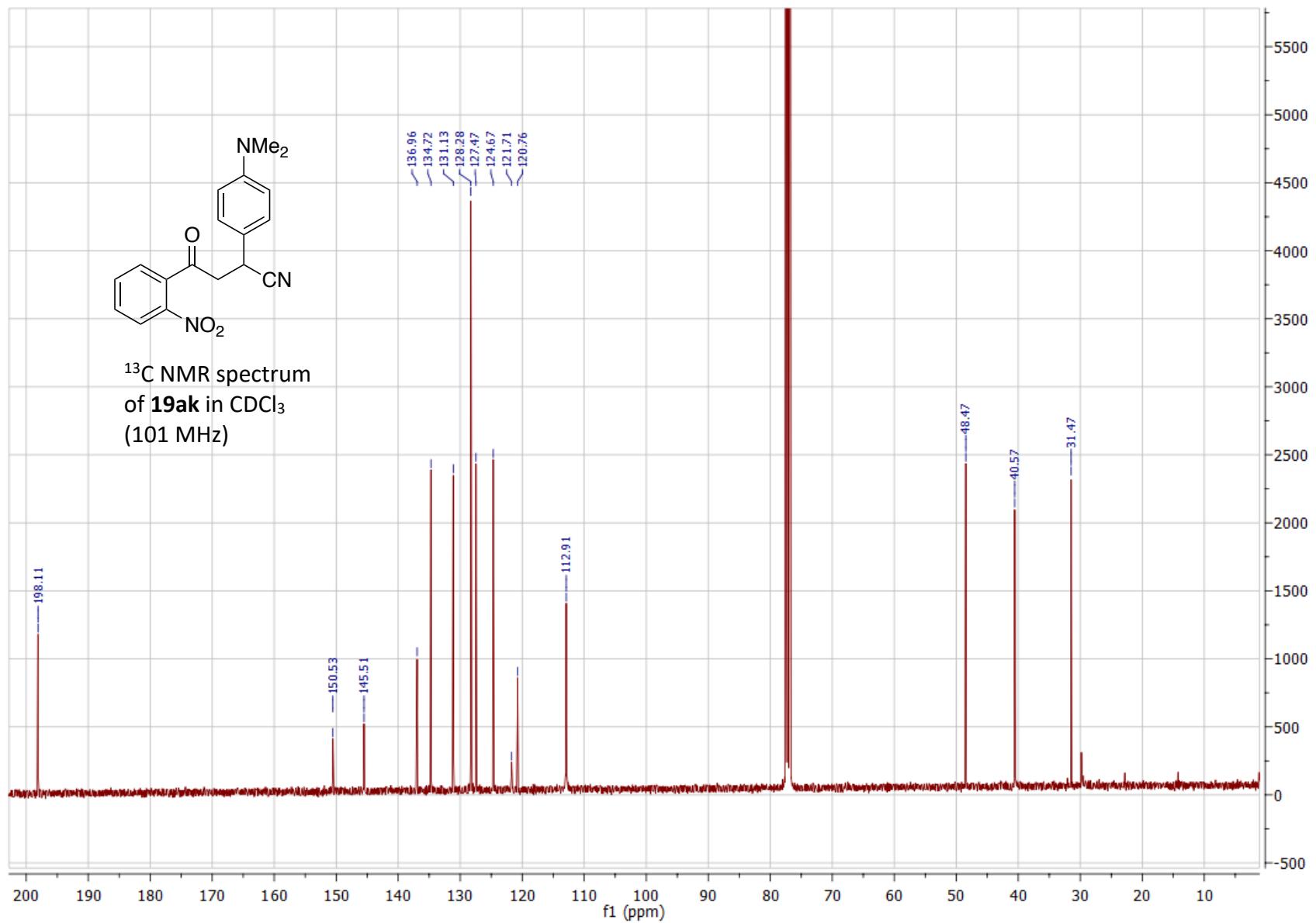


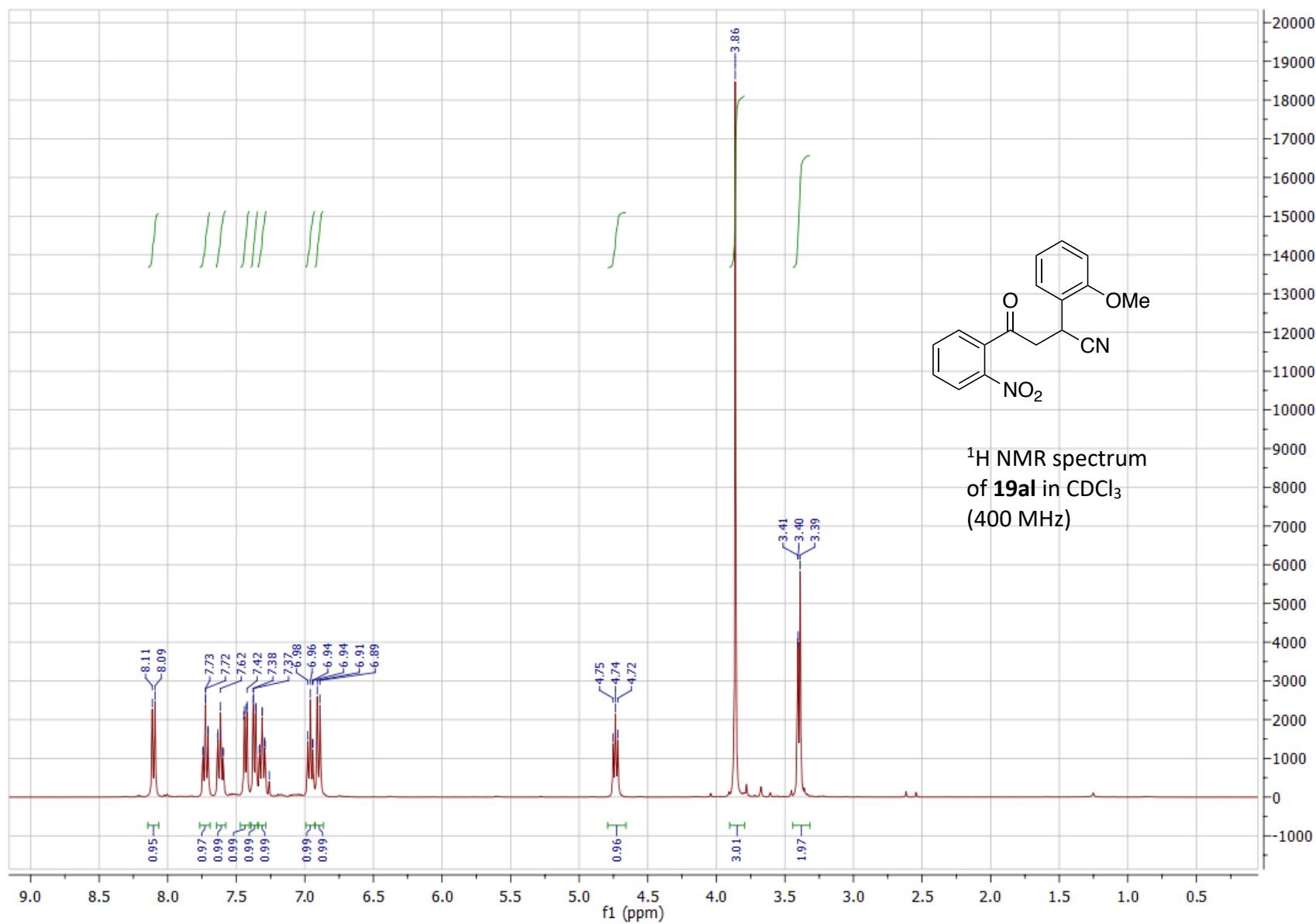


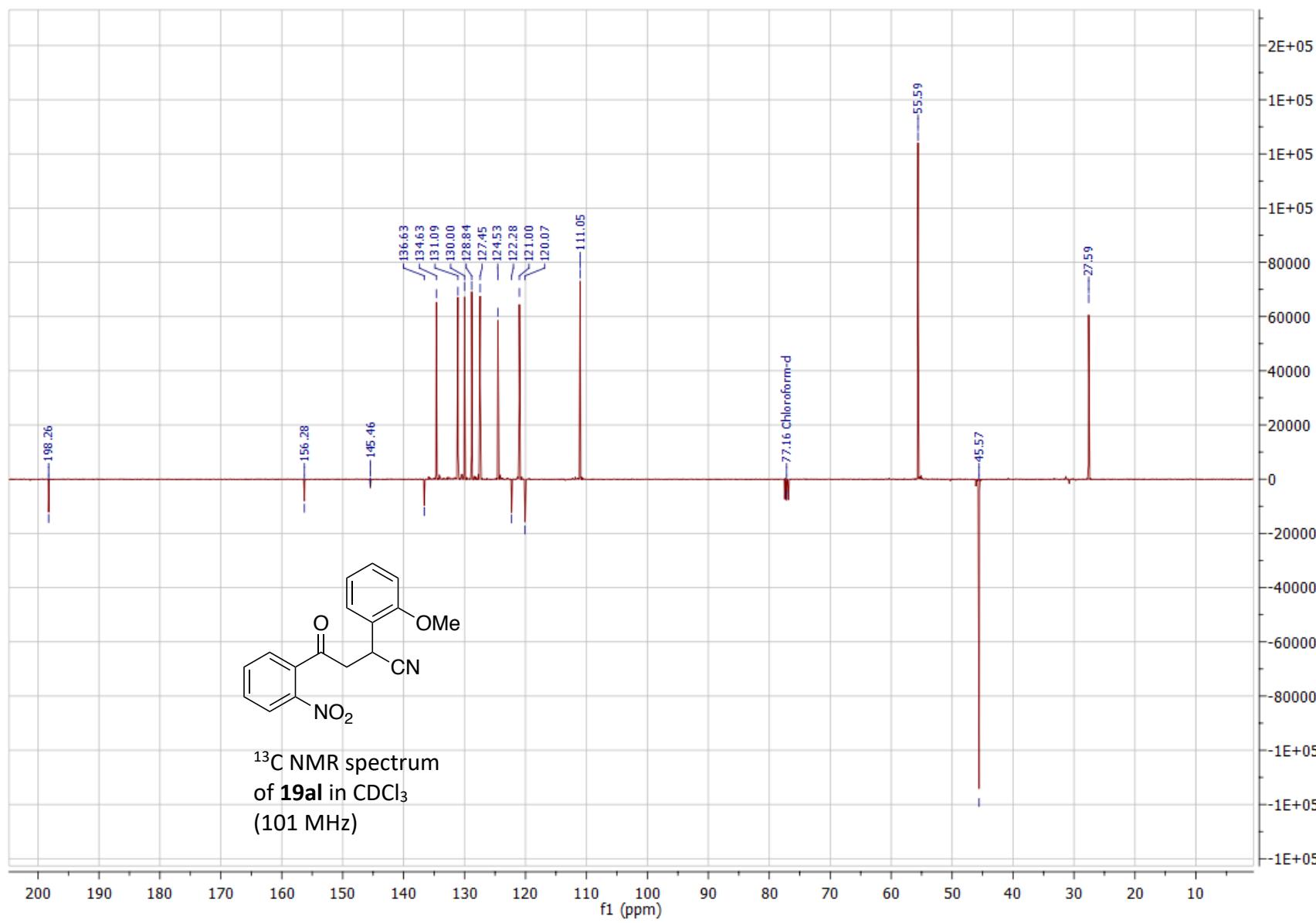


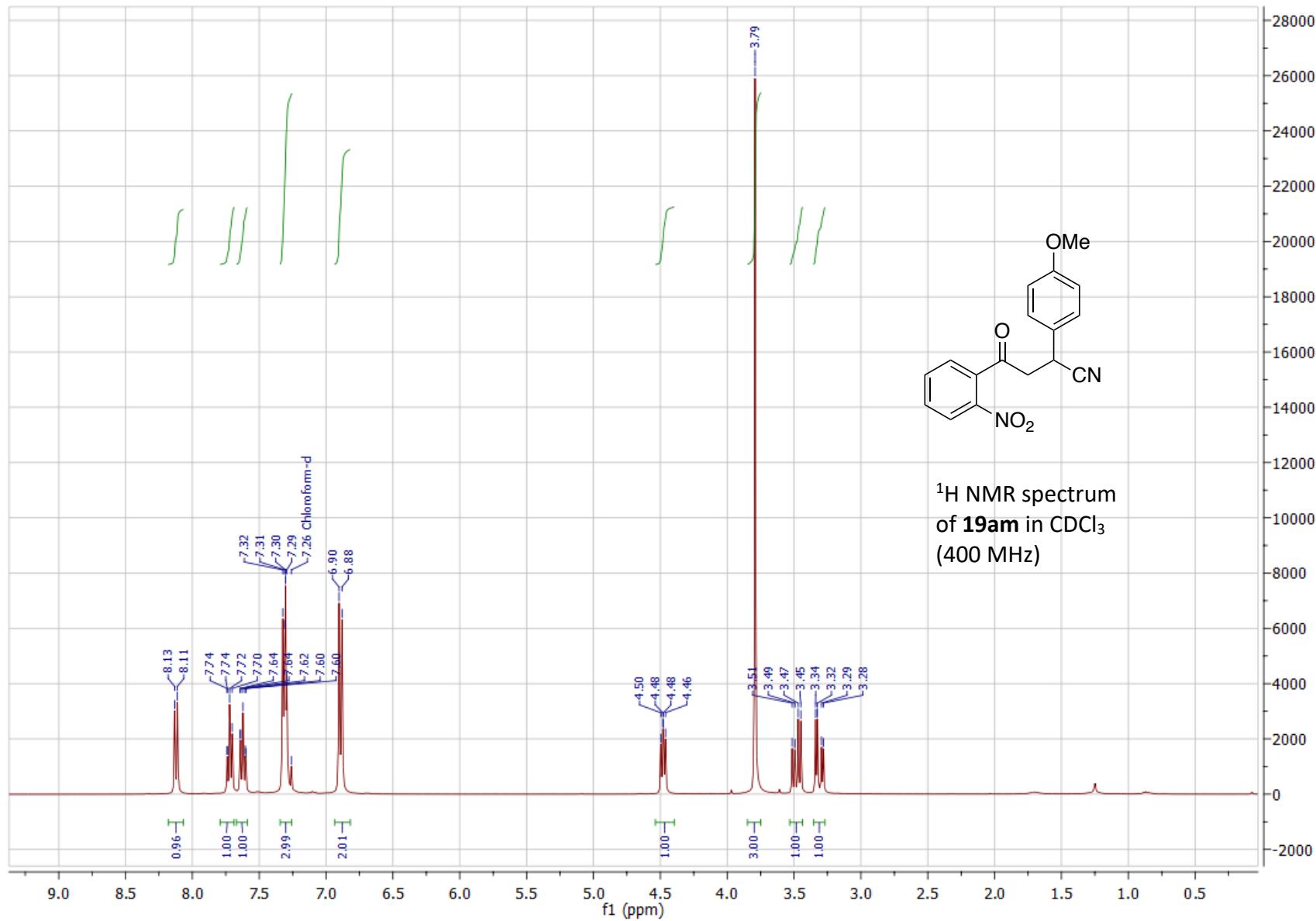


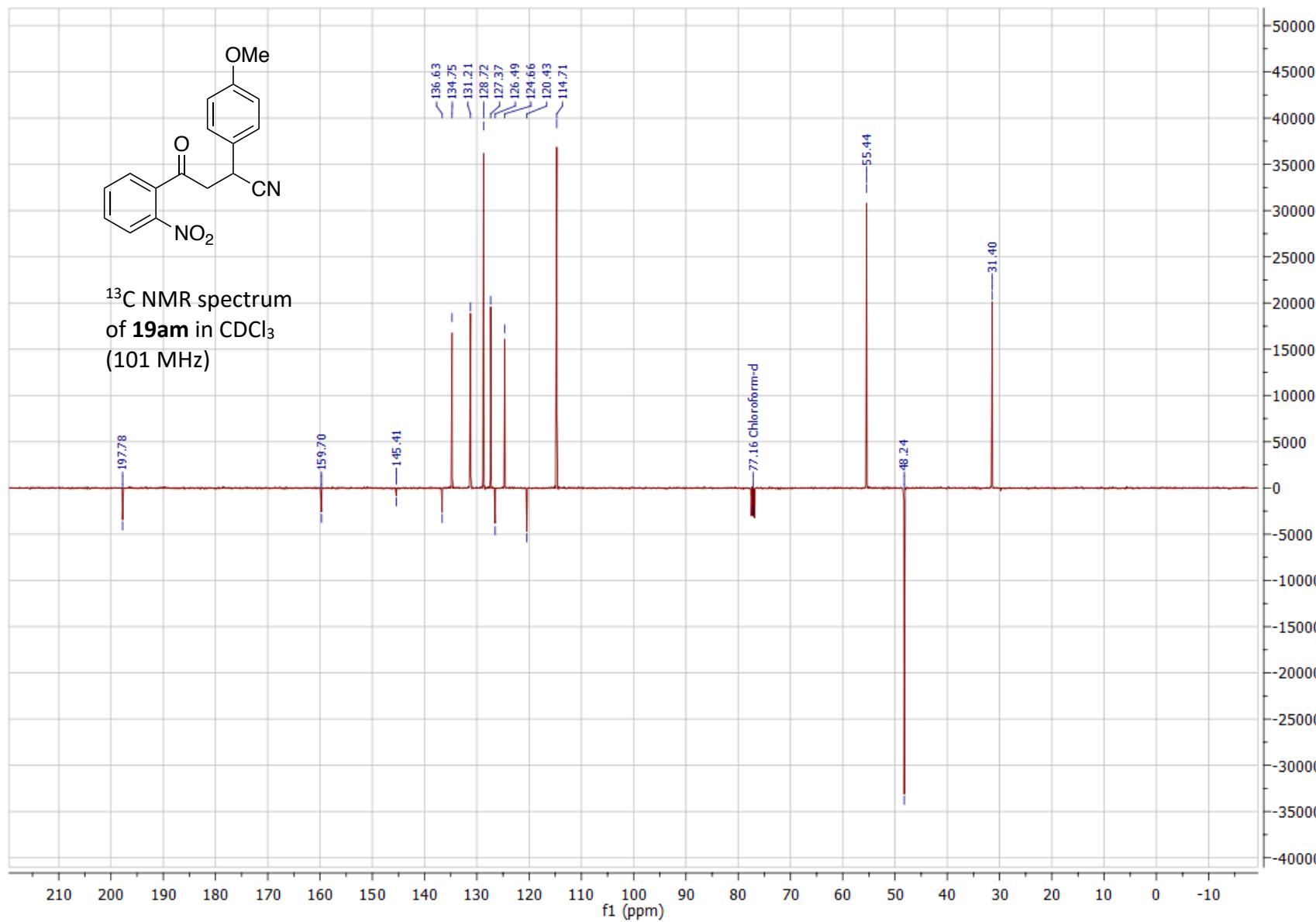


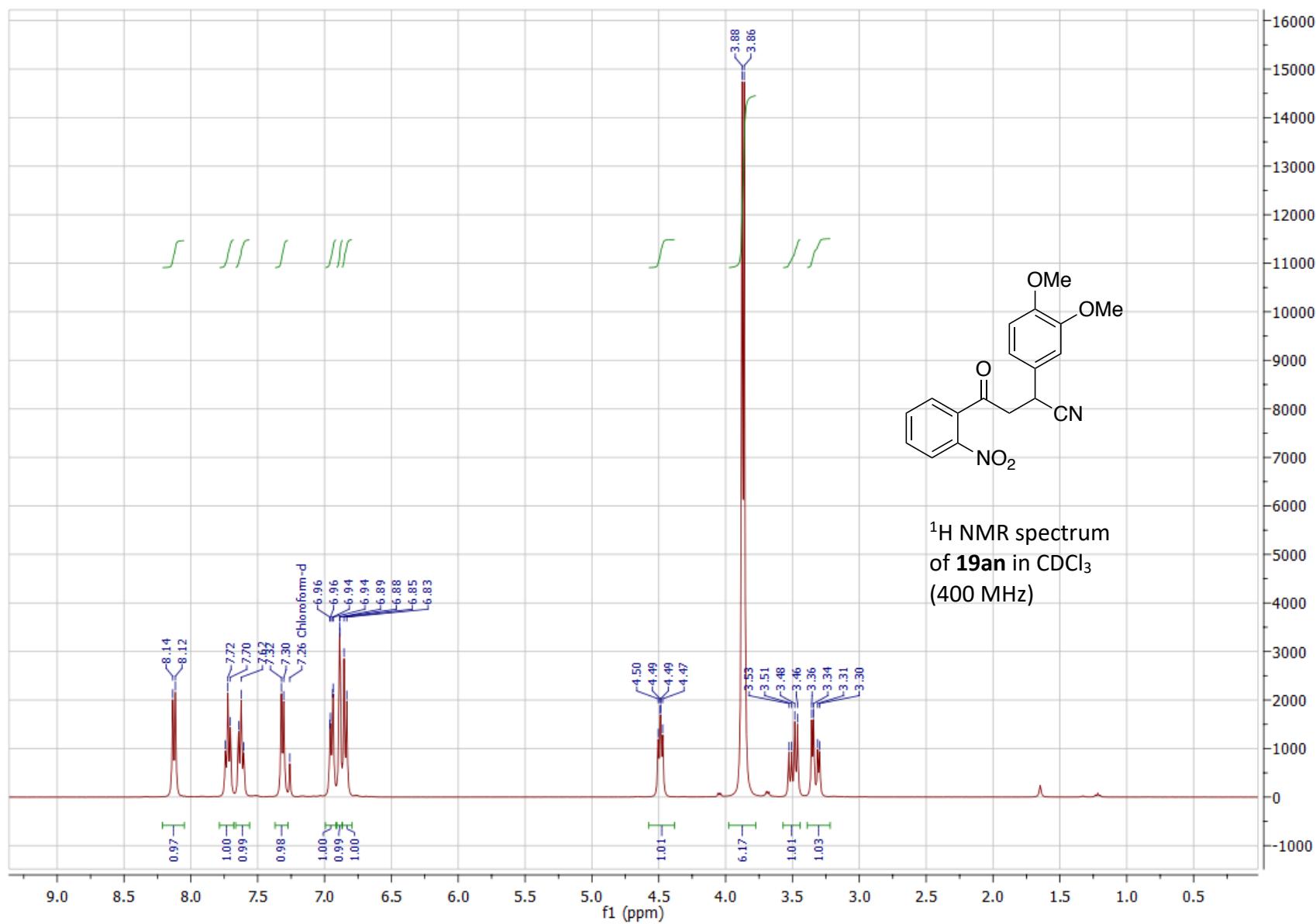


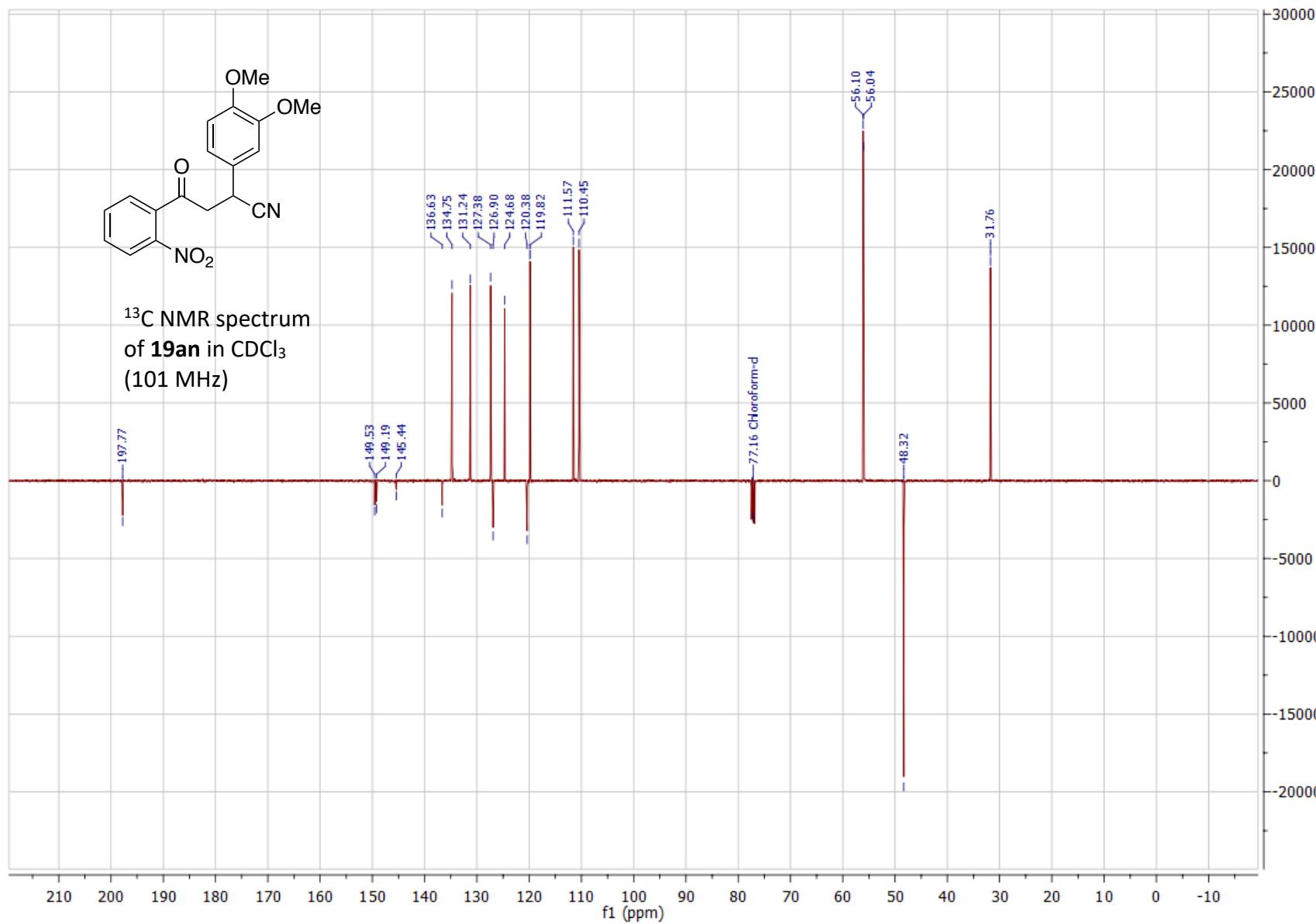


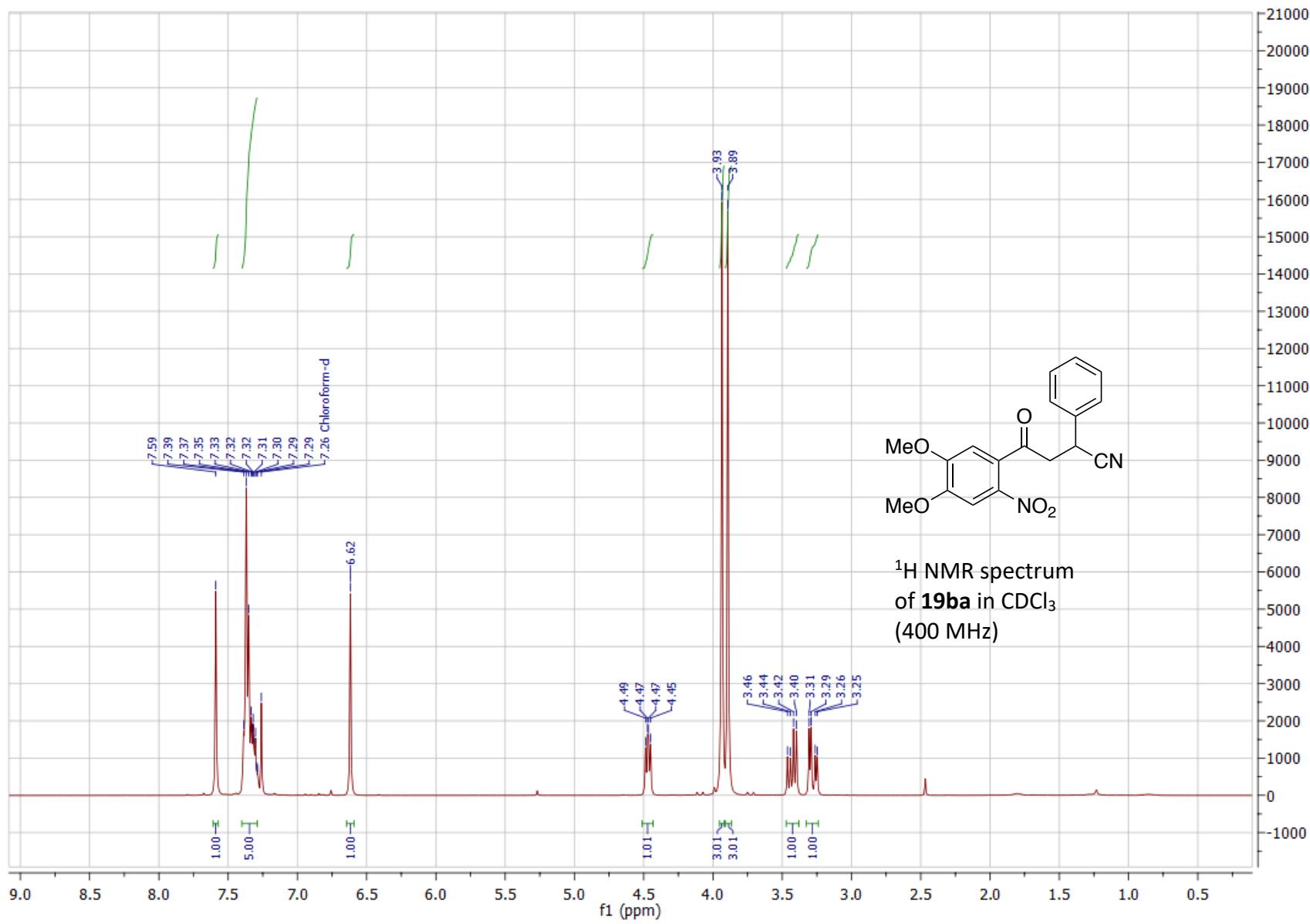


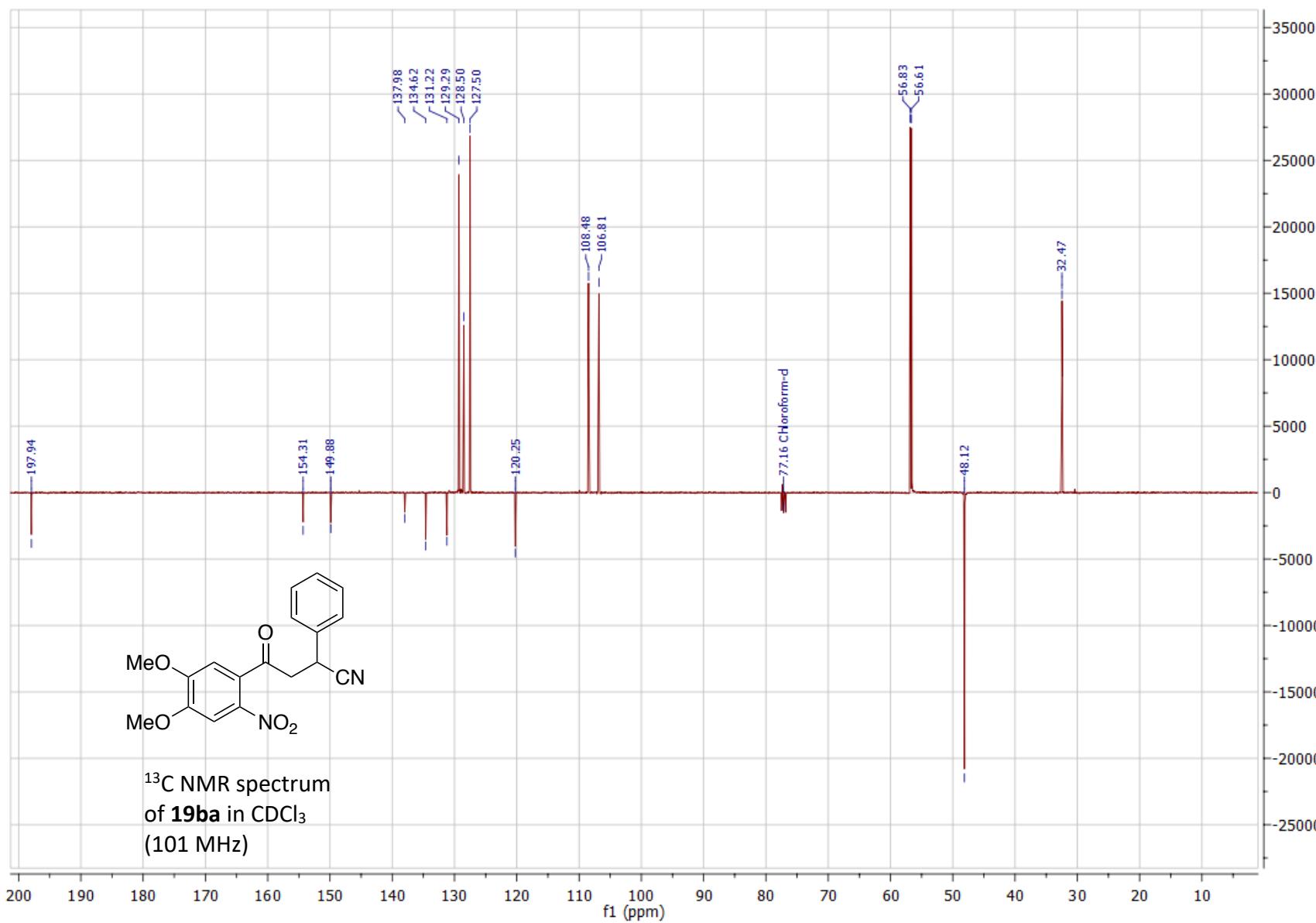


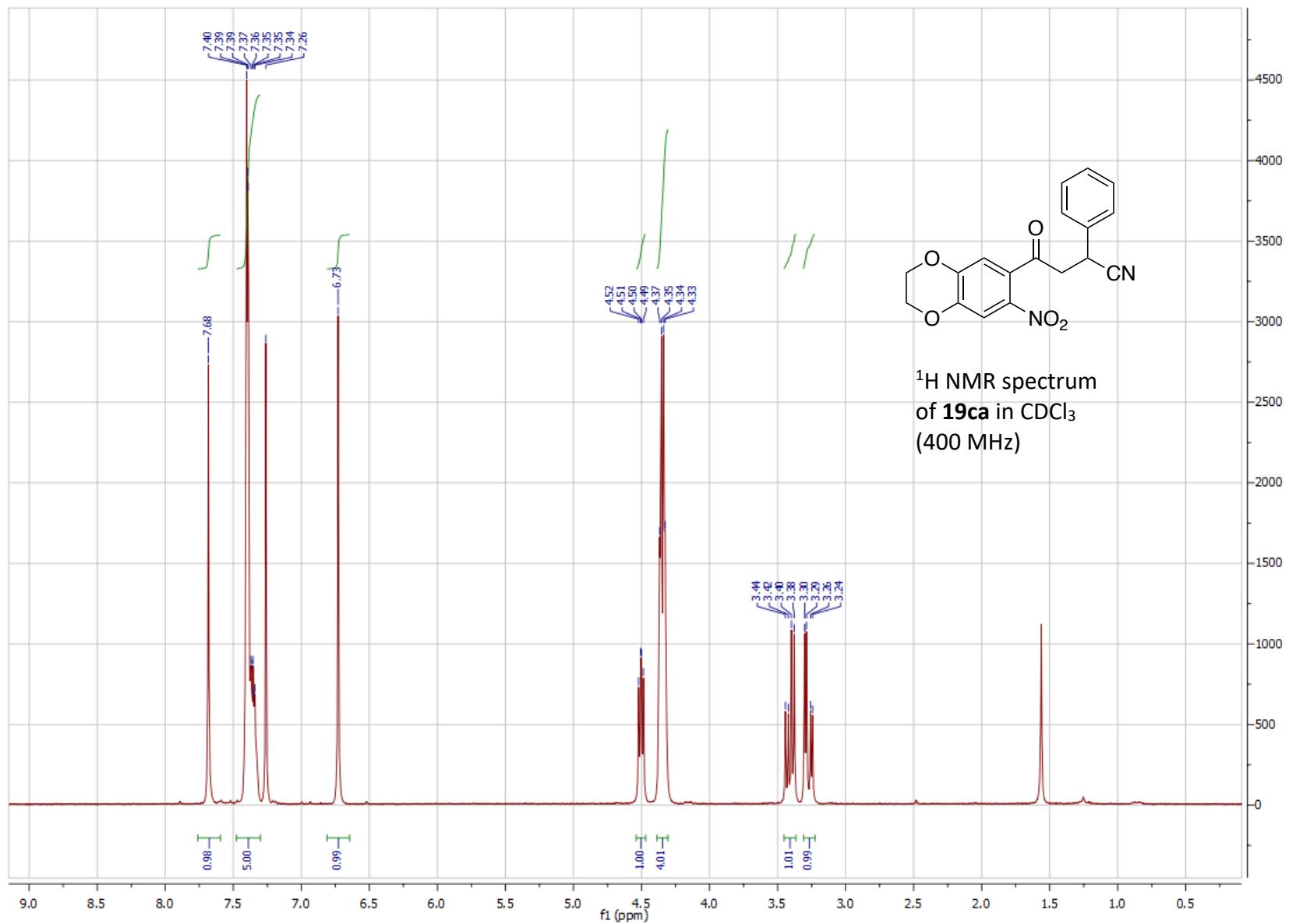


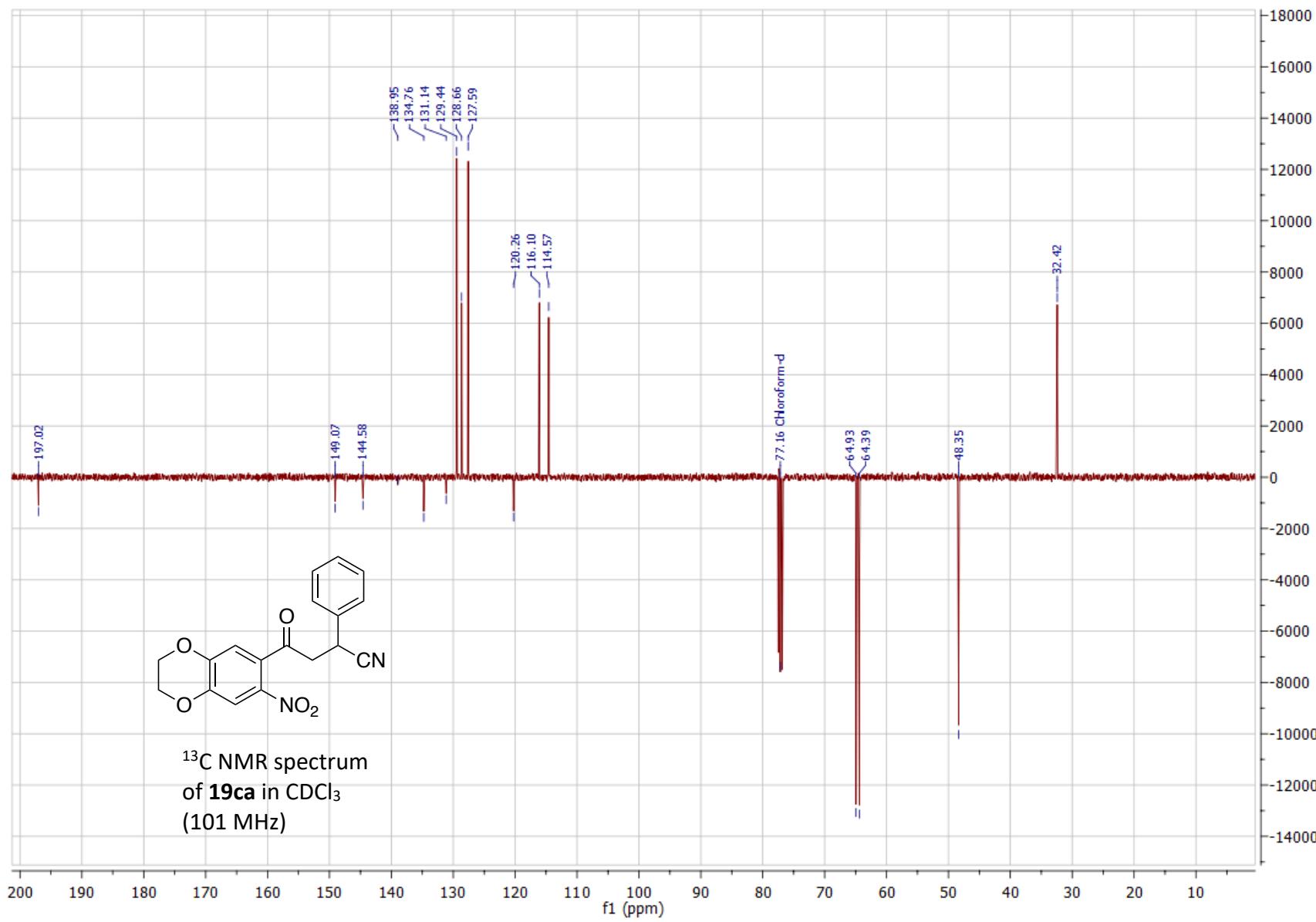




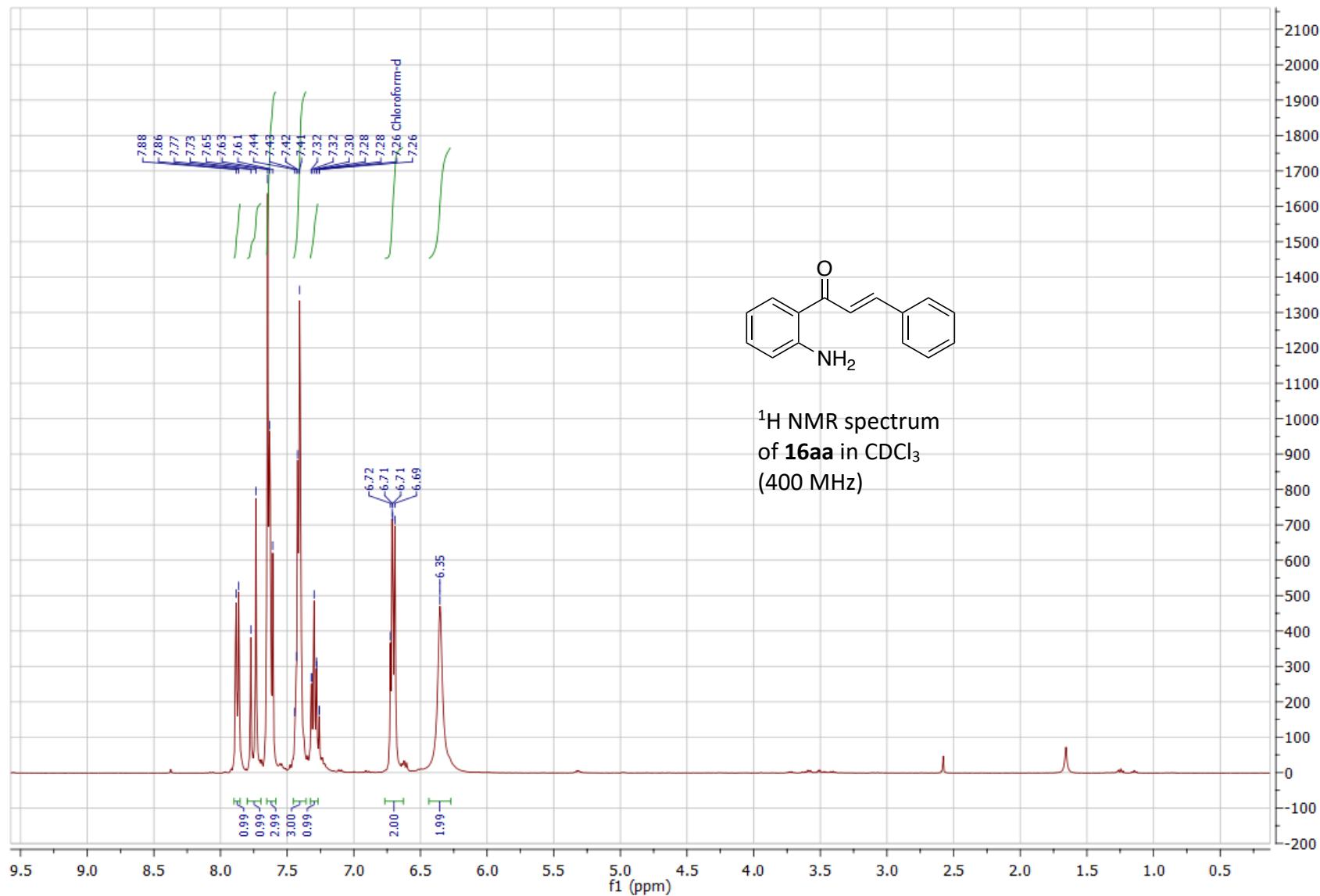


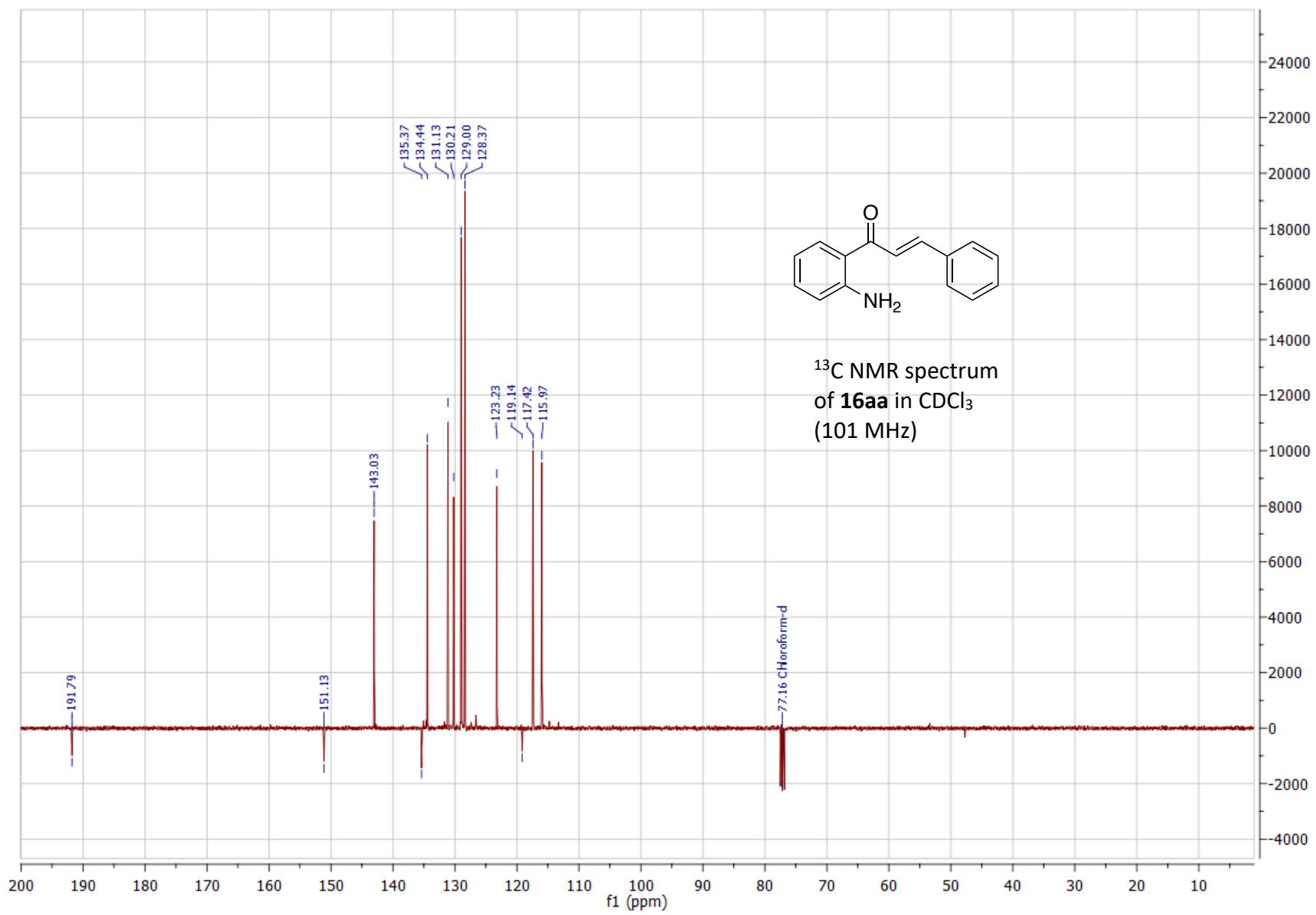


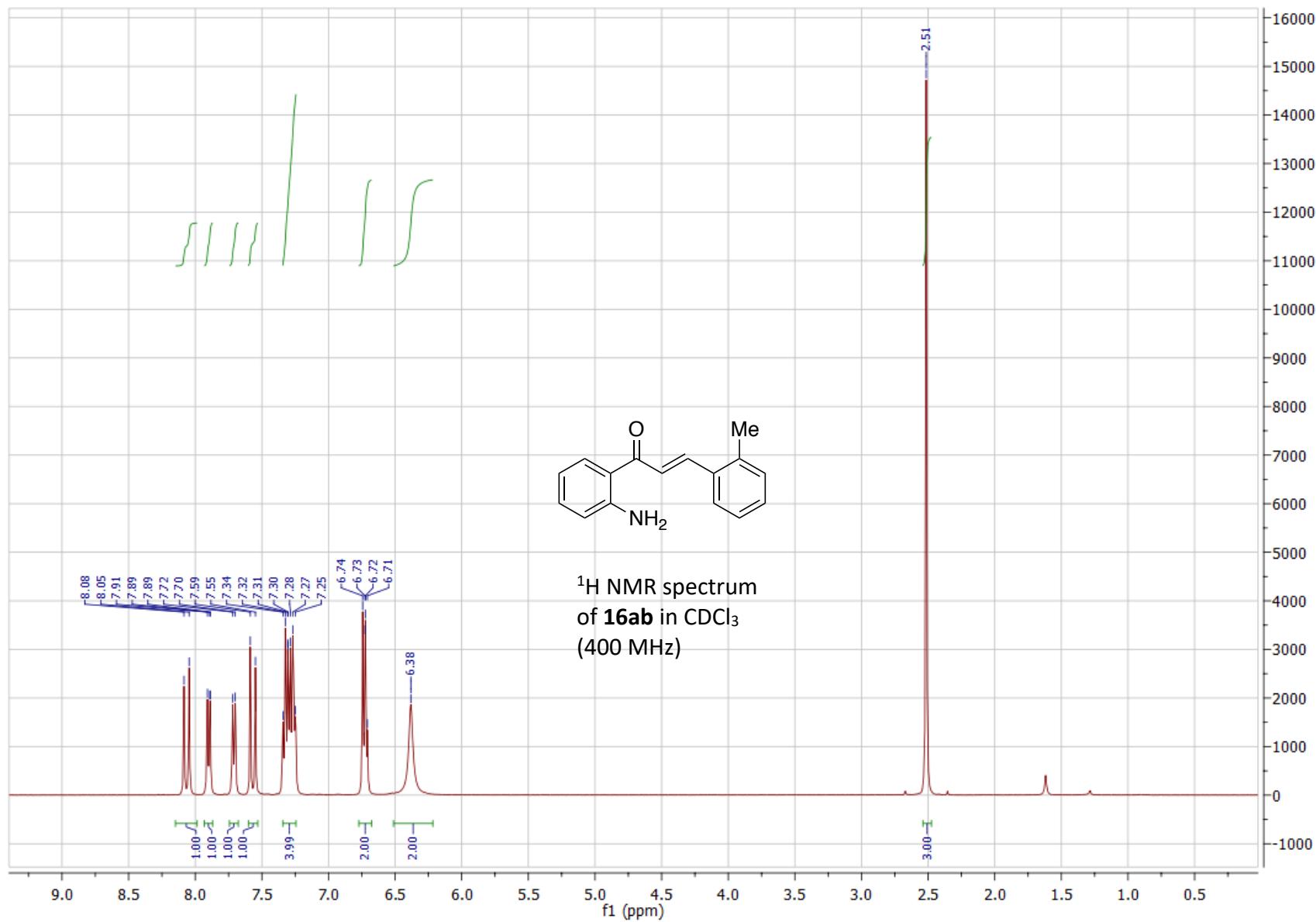


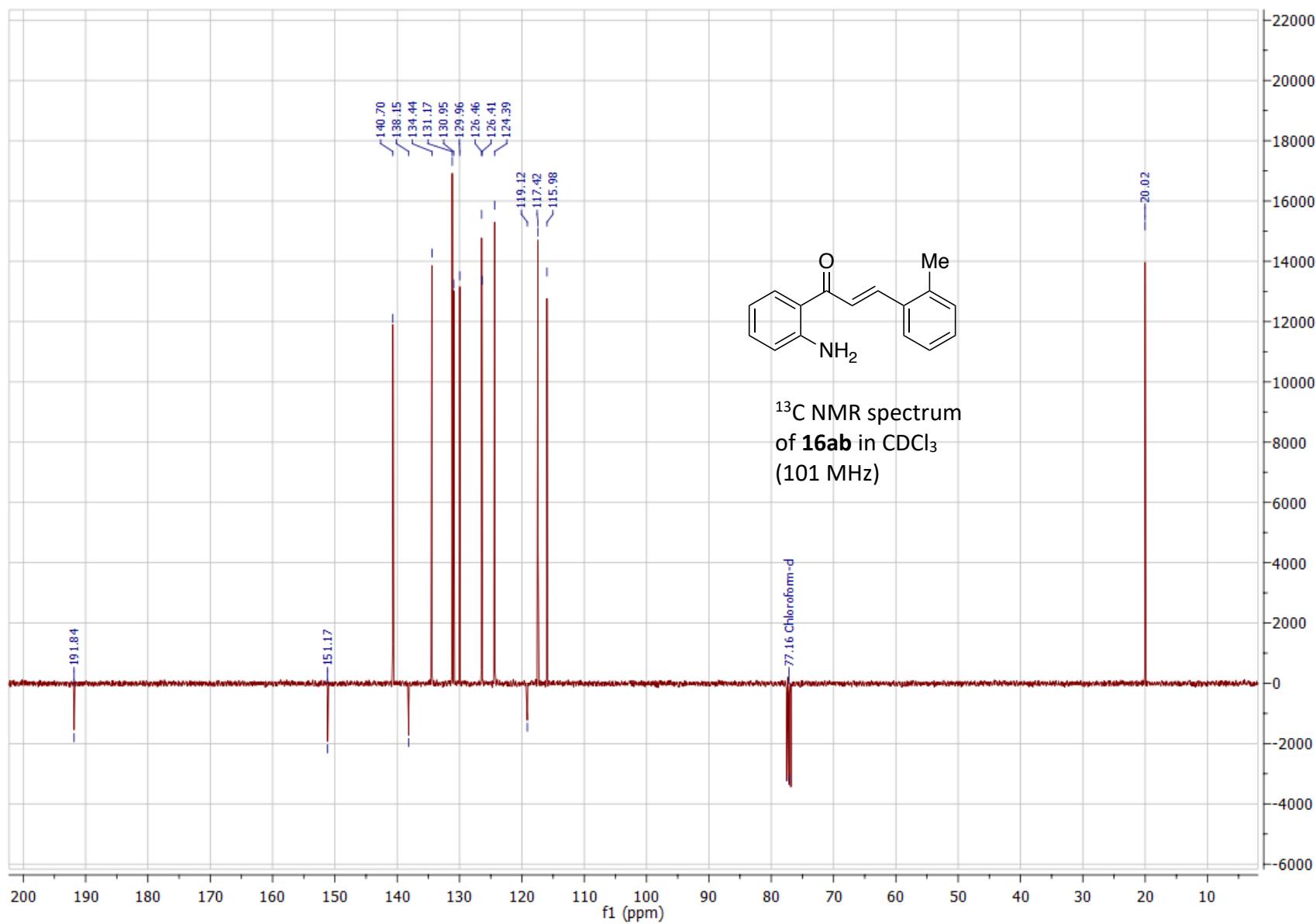


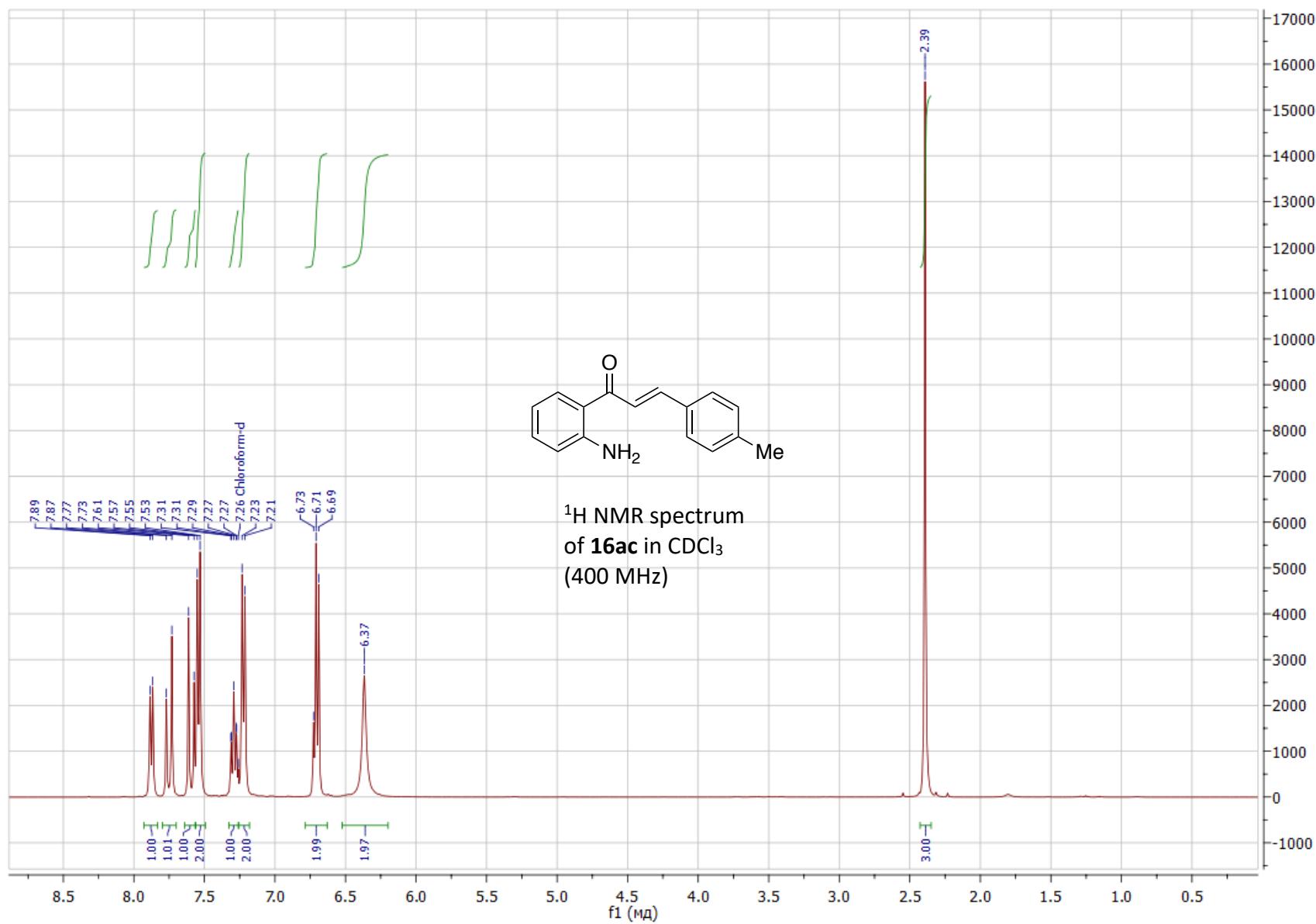
^1H and ^{13}C Spectral Charts of (E)-1-(2-aminophenyl)-3-arylprop-2-en-1-ones 16

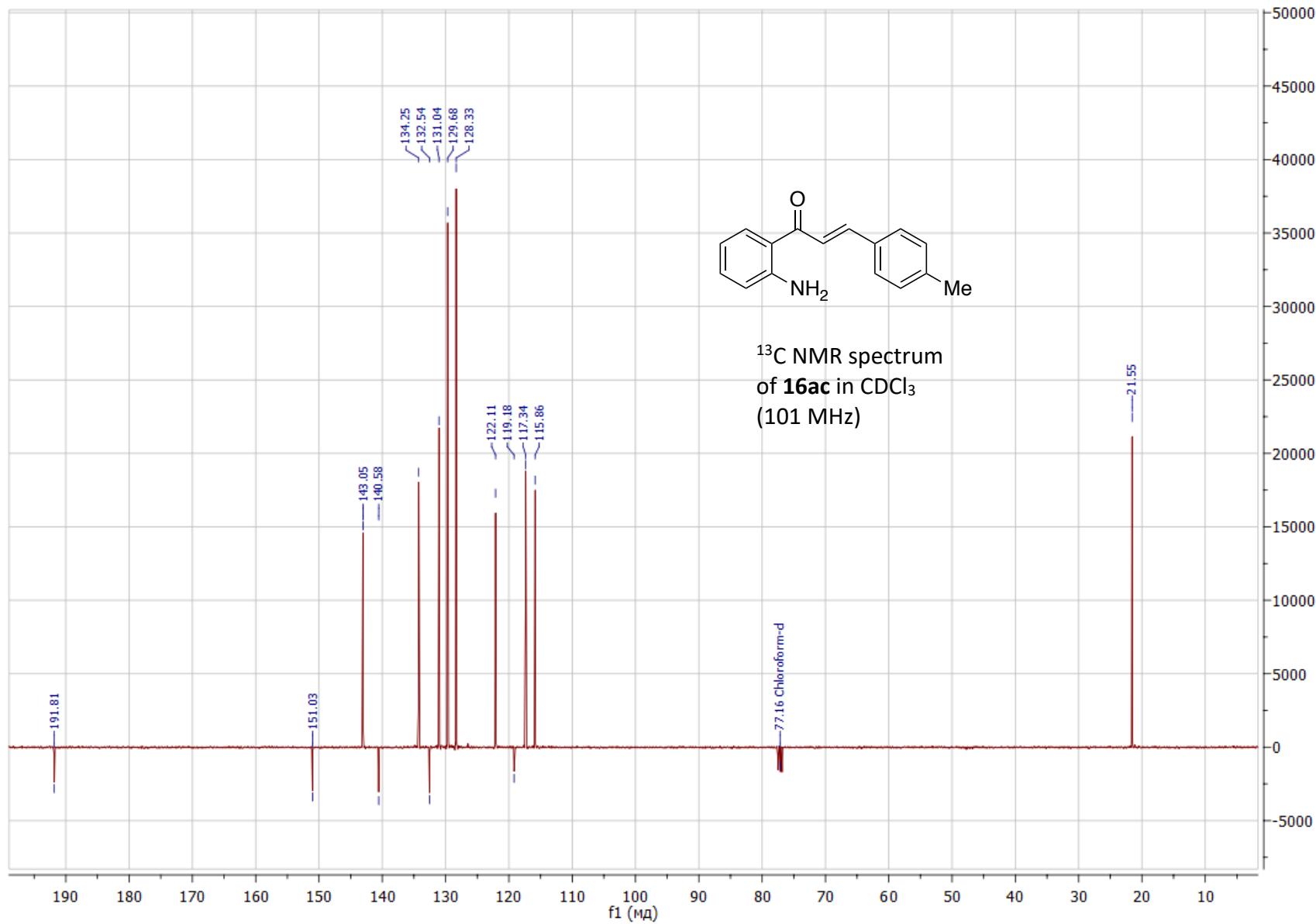


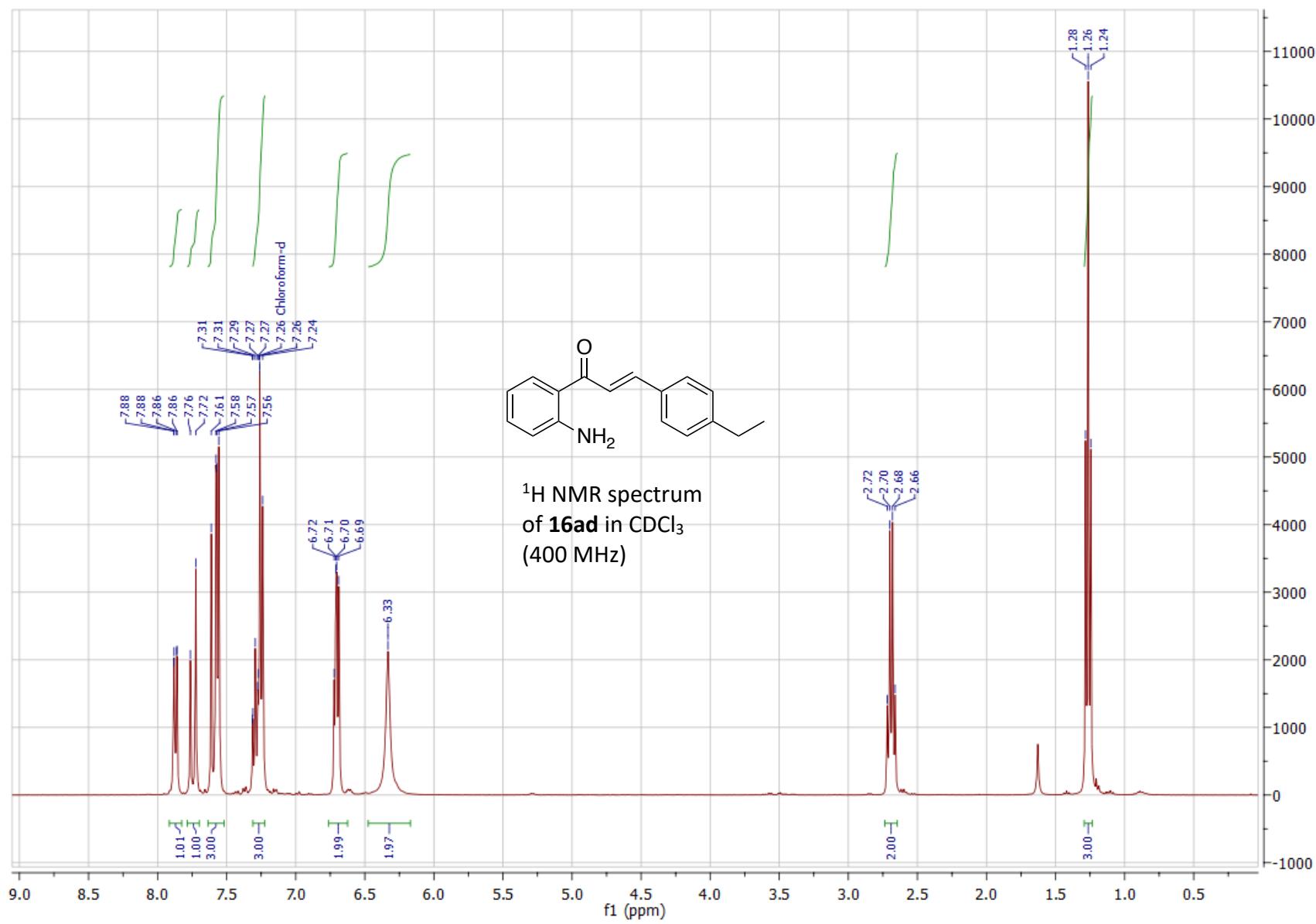


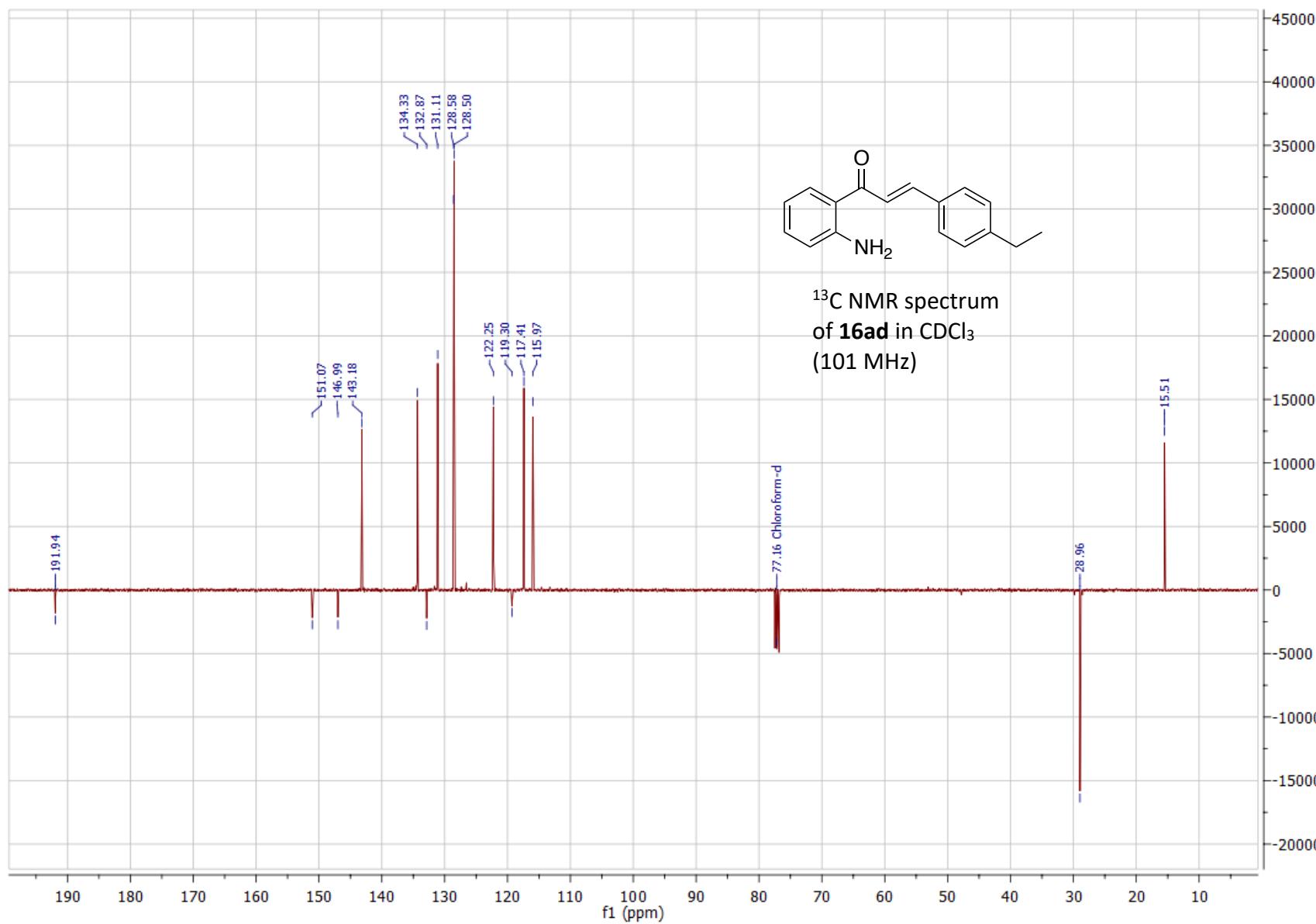


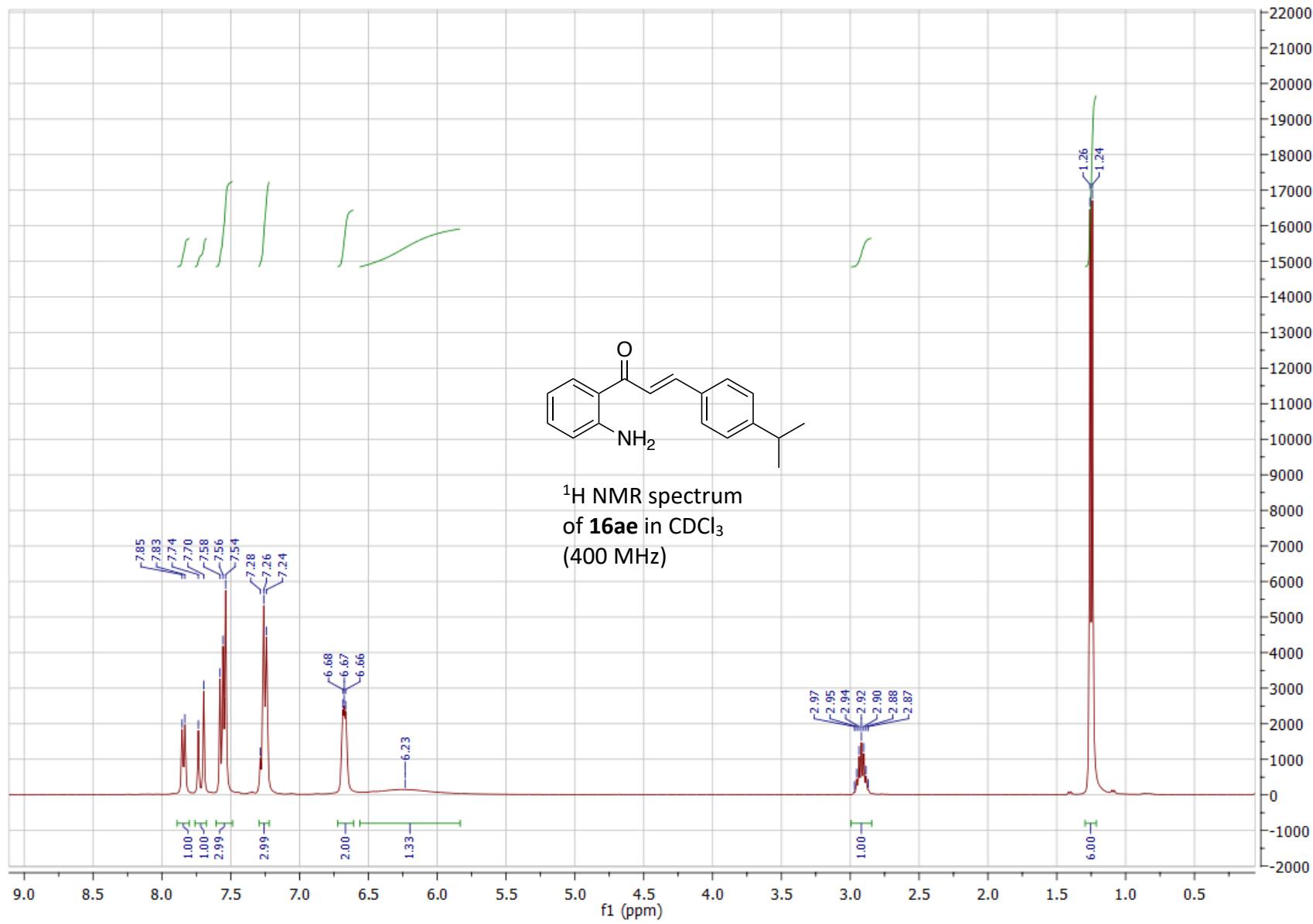


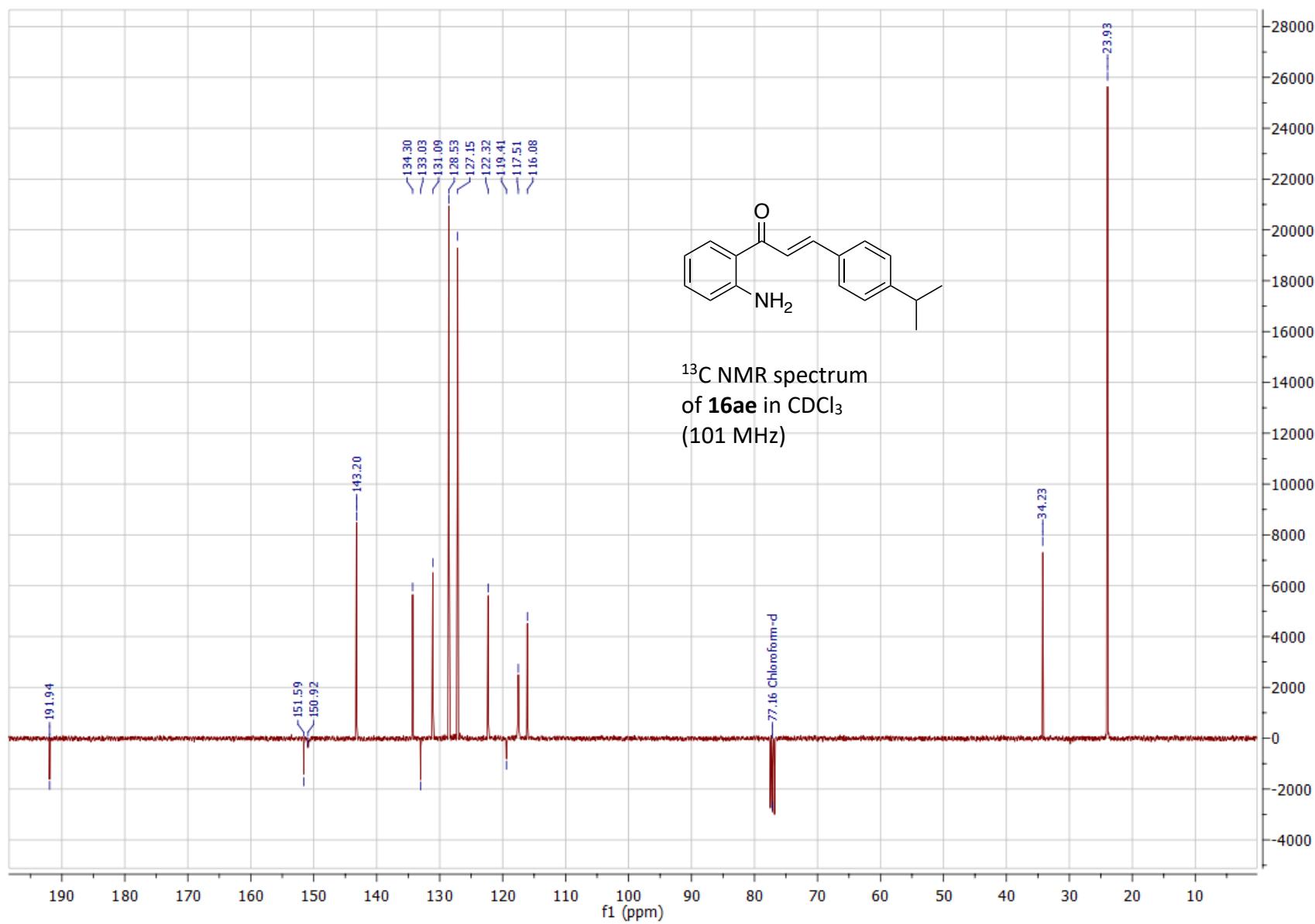


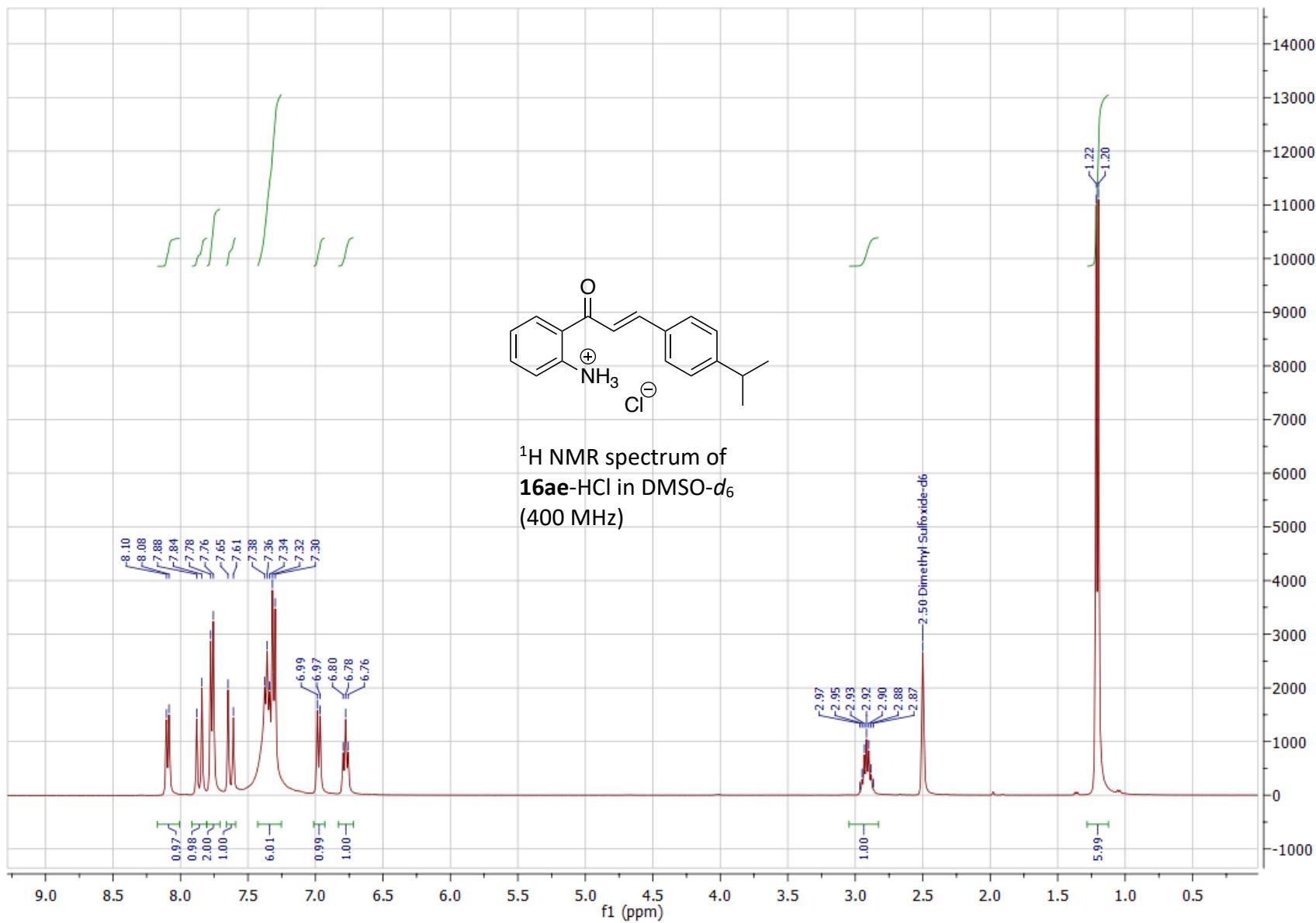


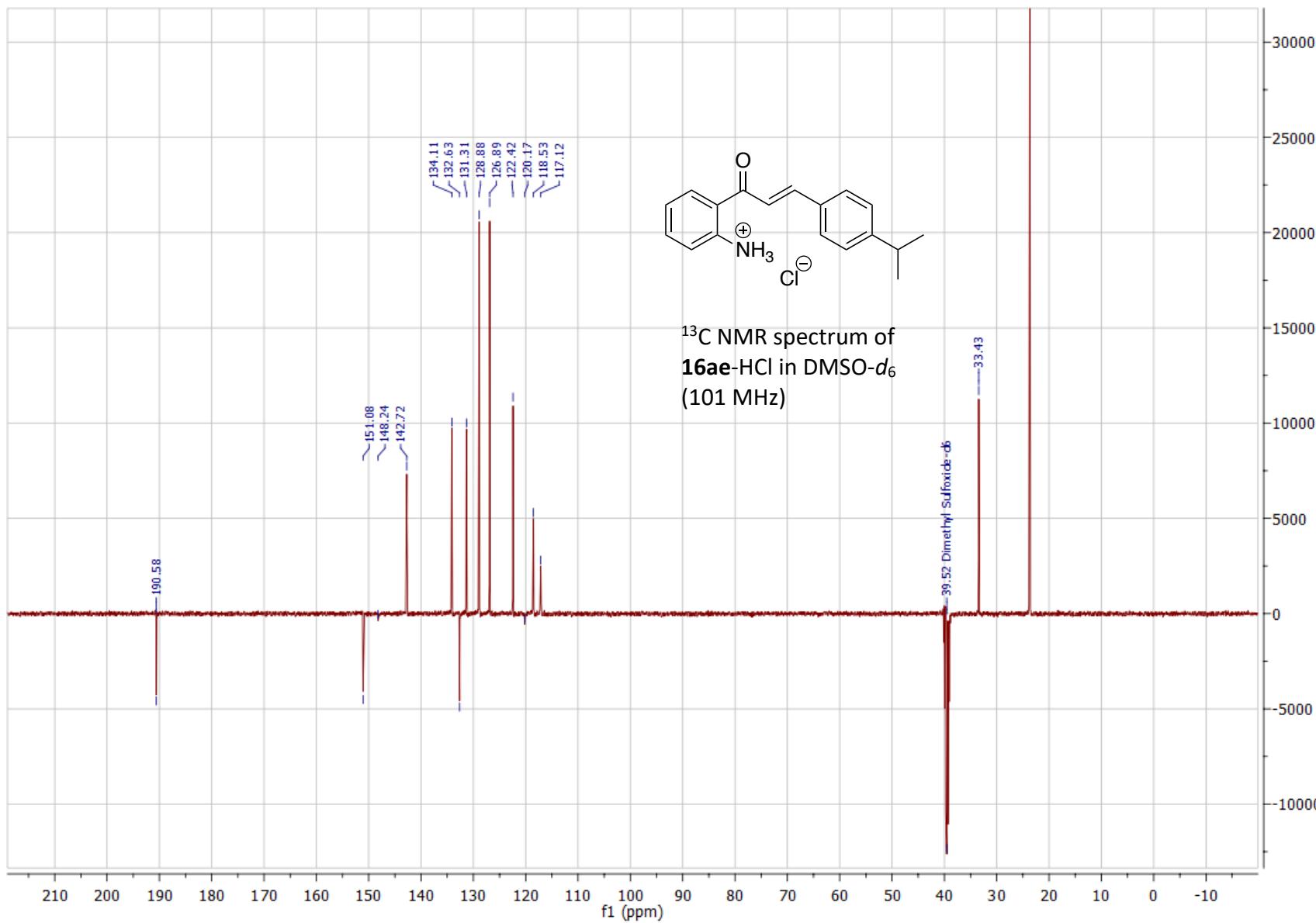


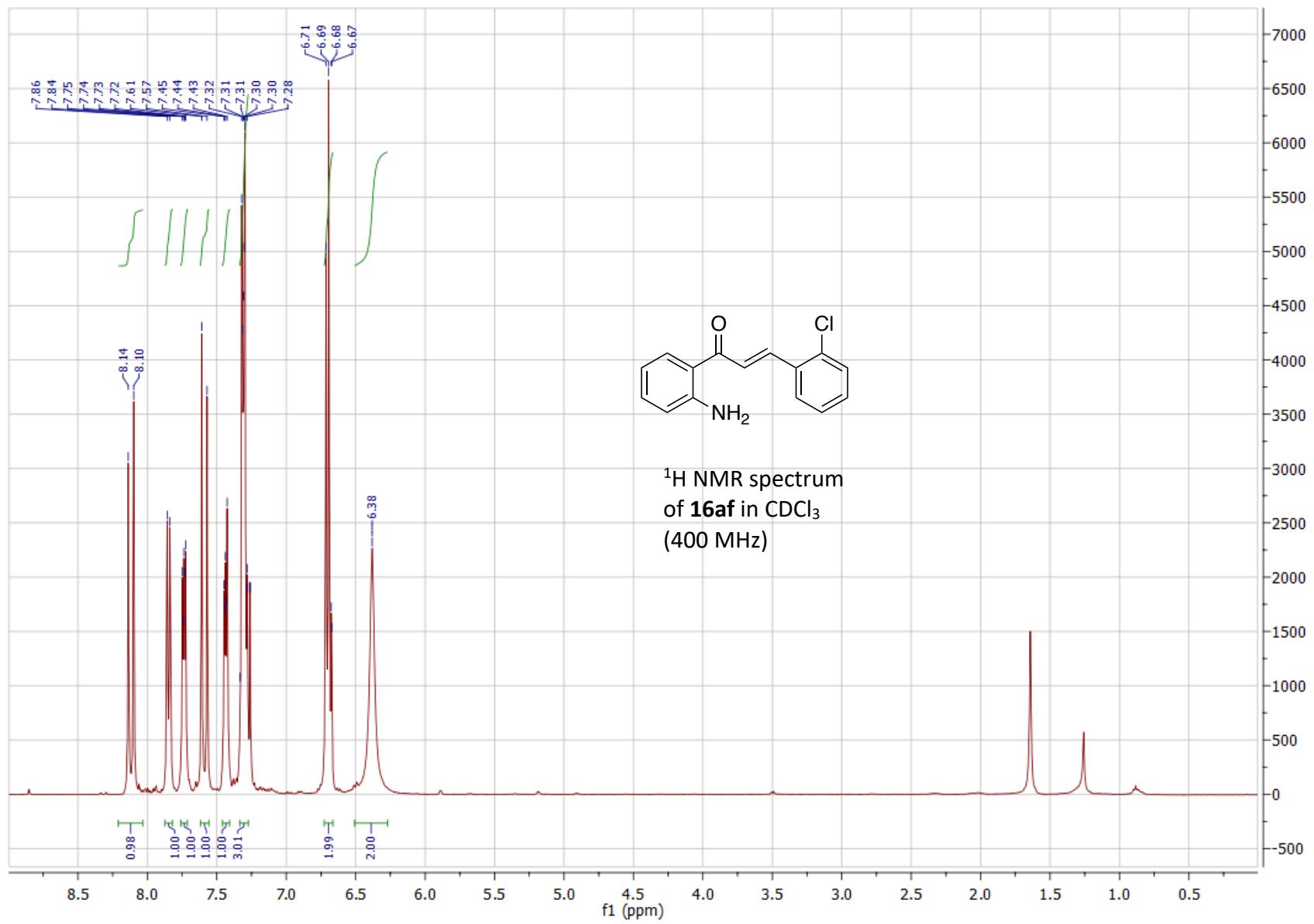


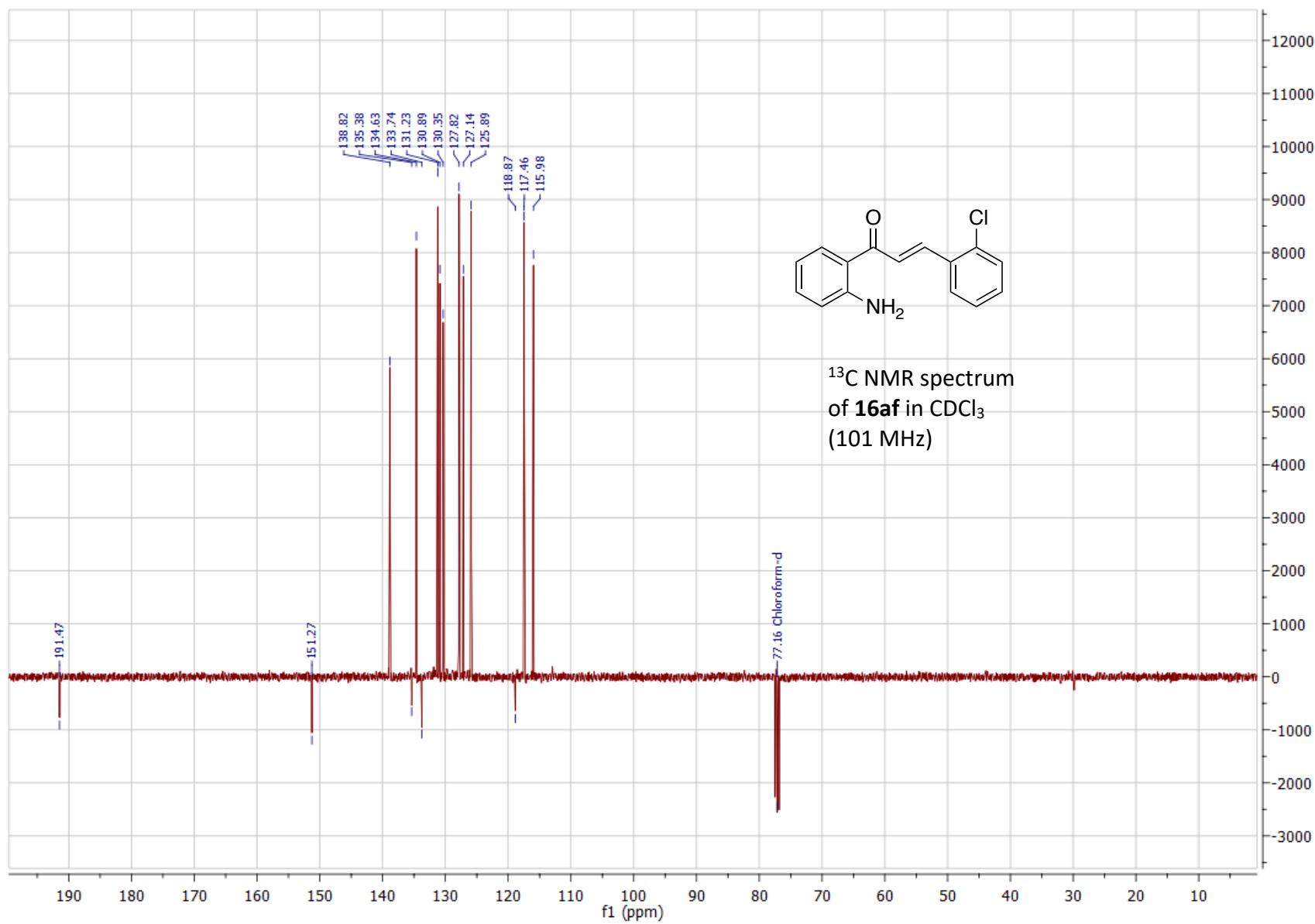


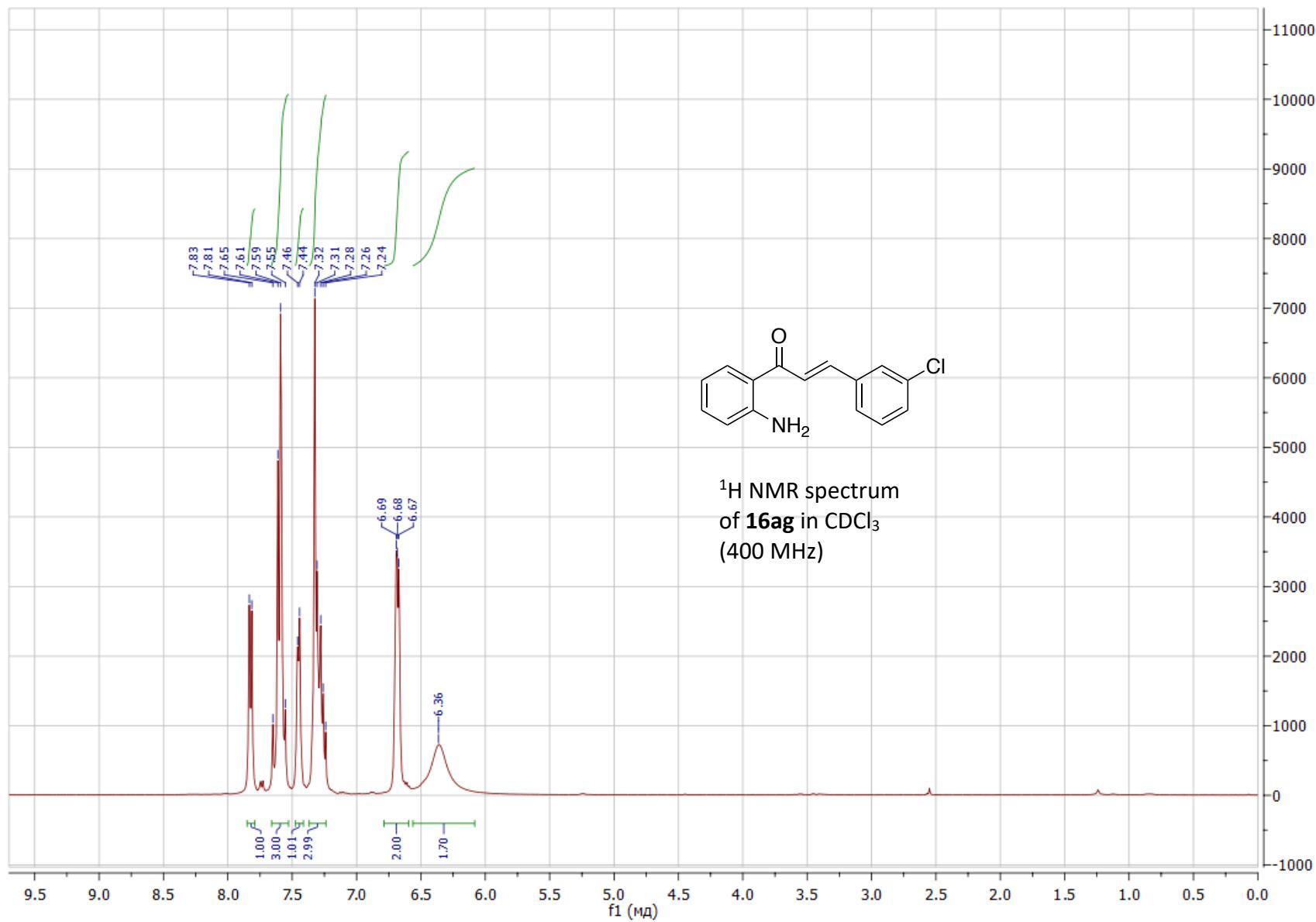


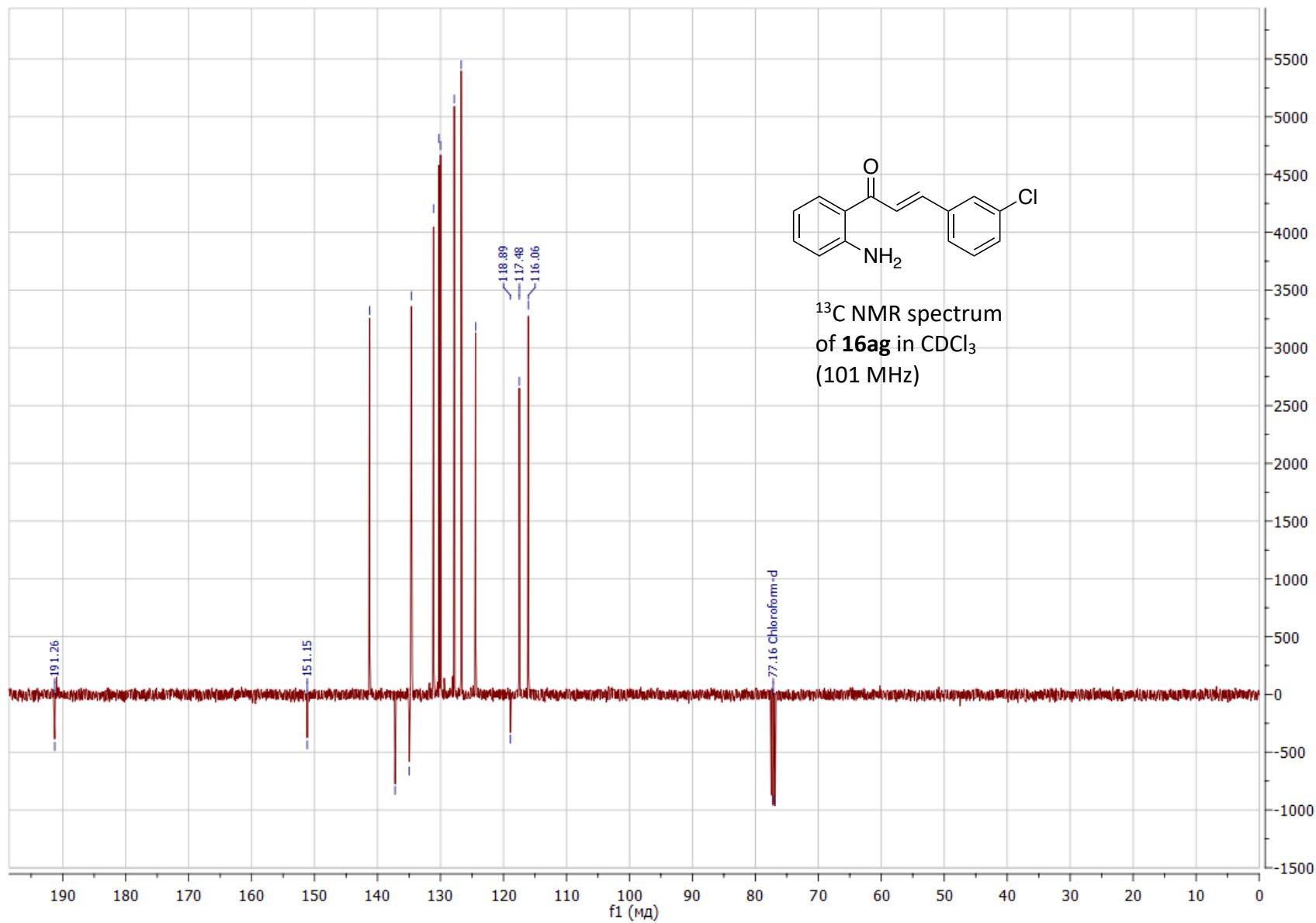


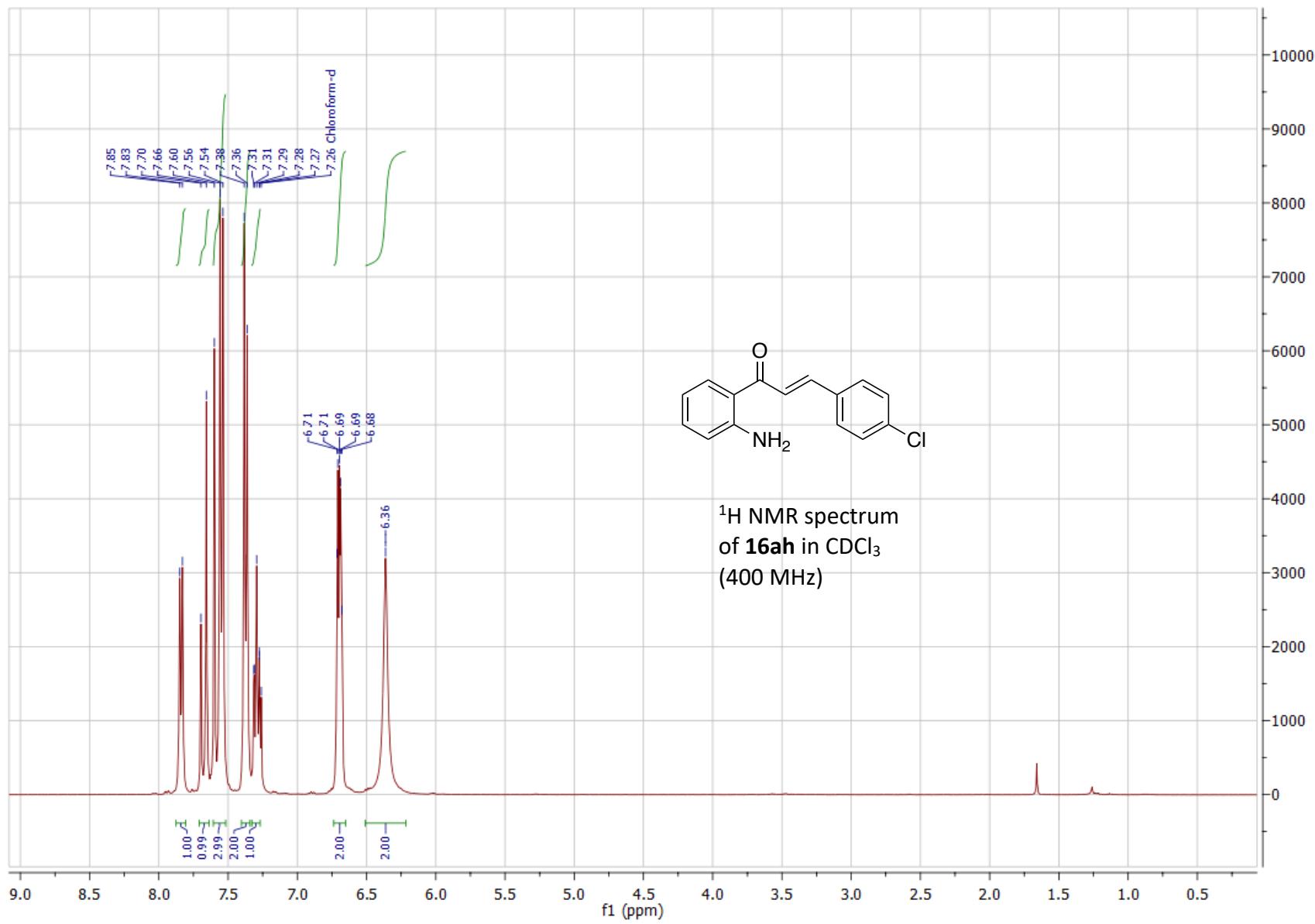


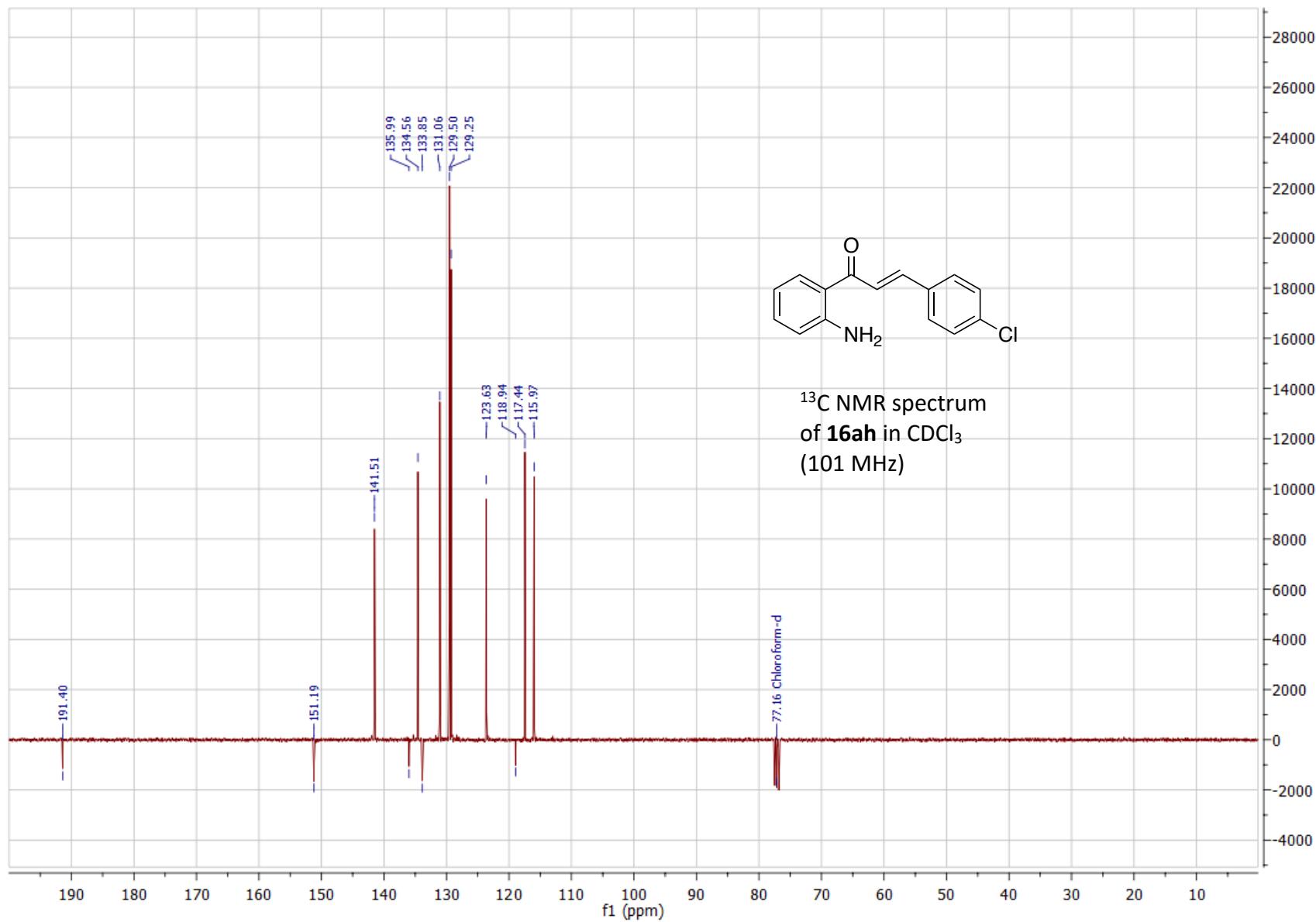


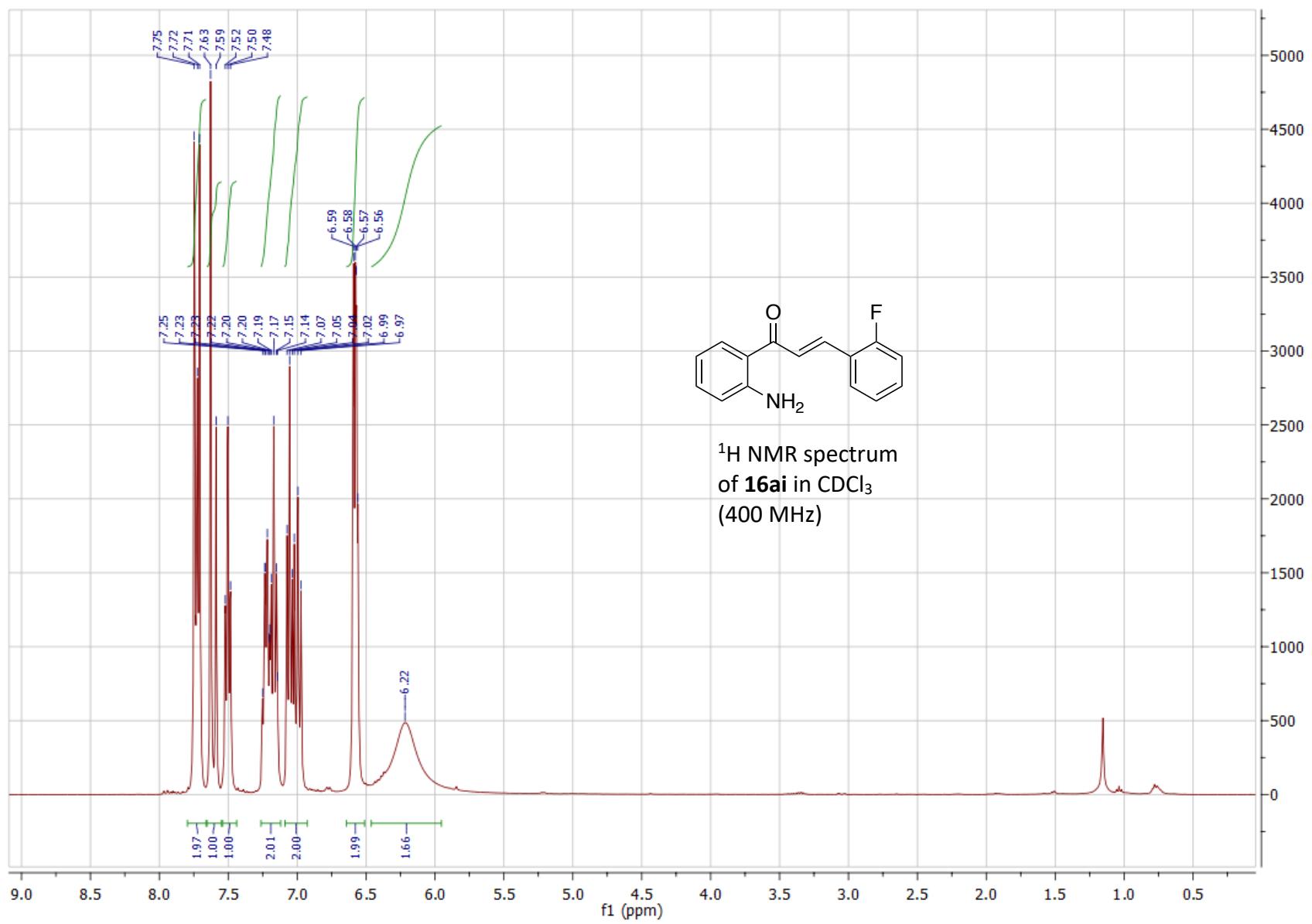


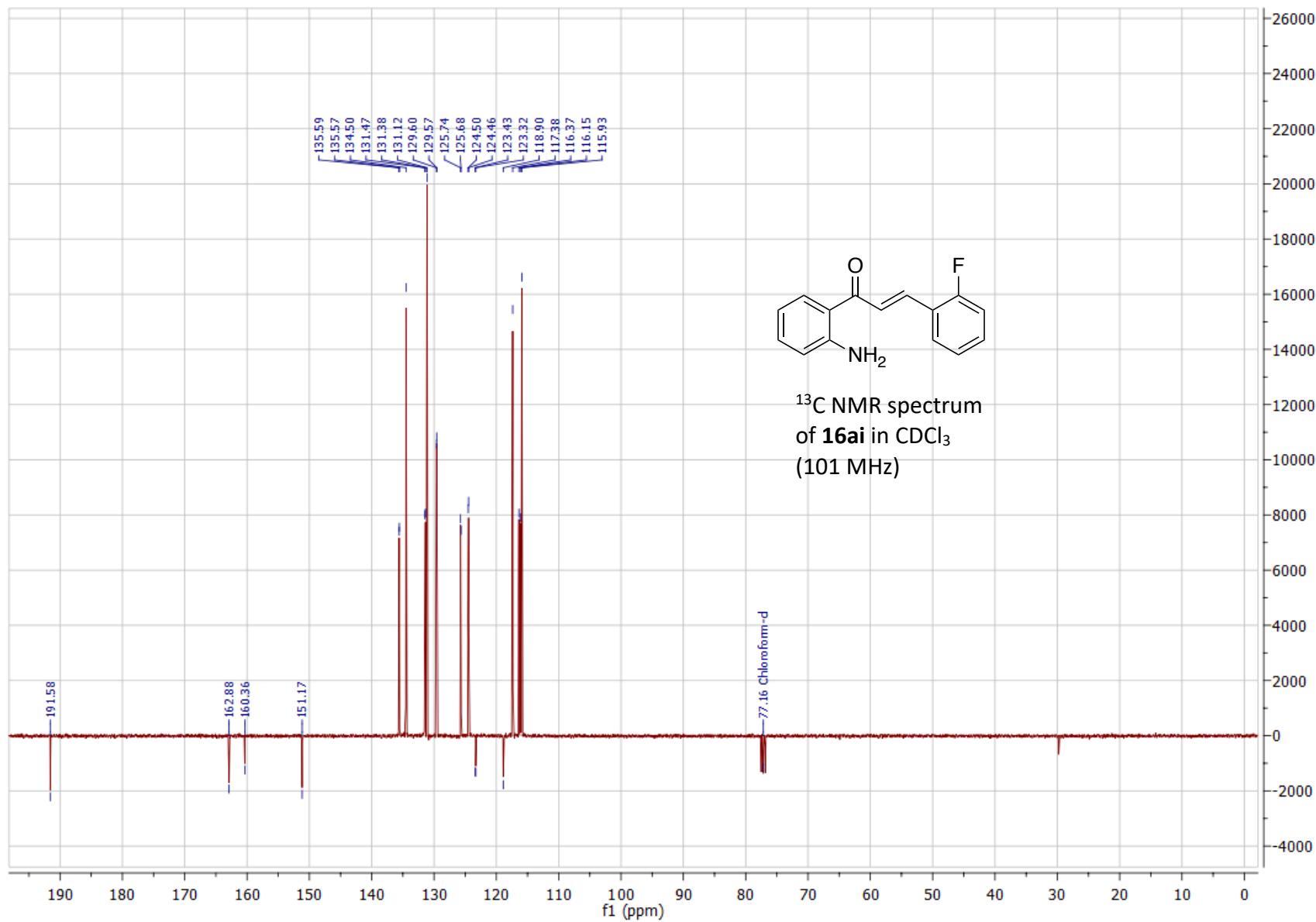


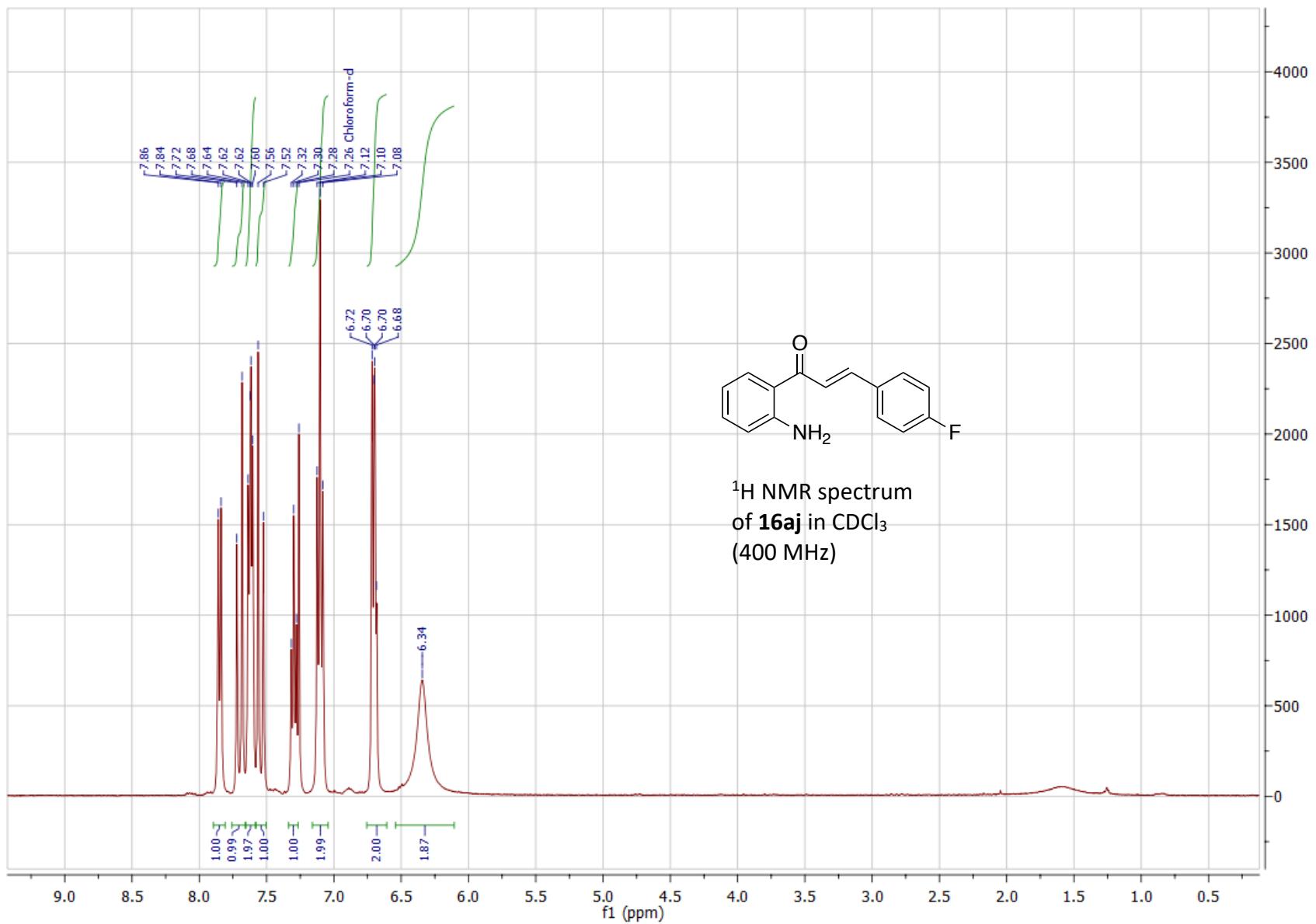


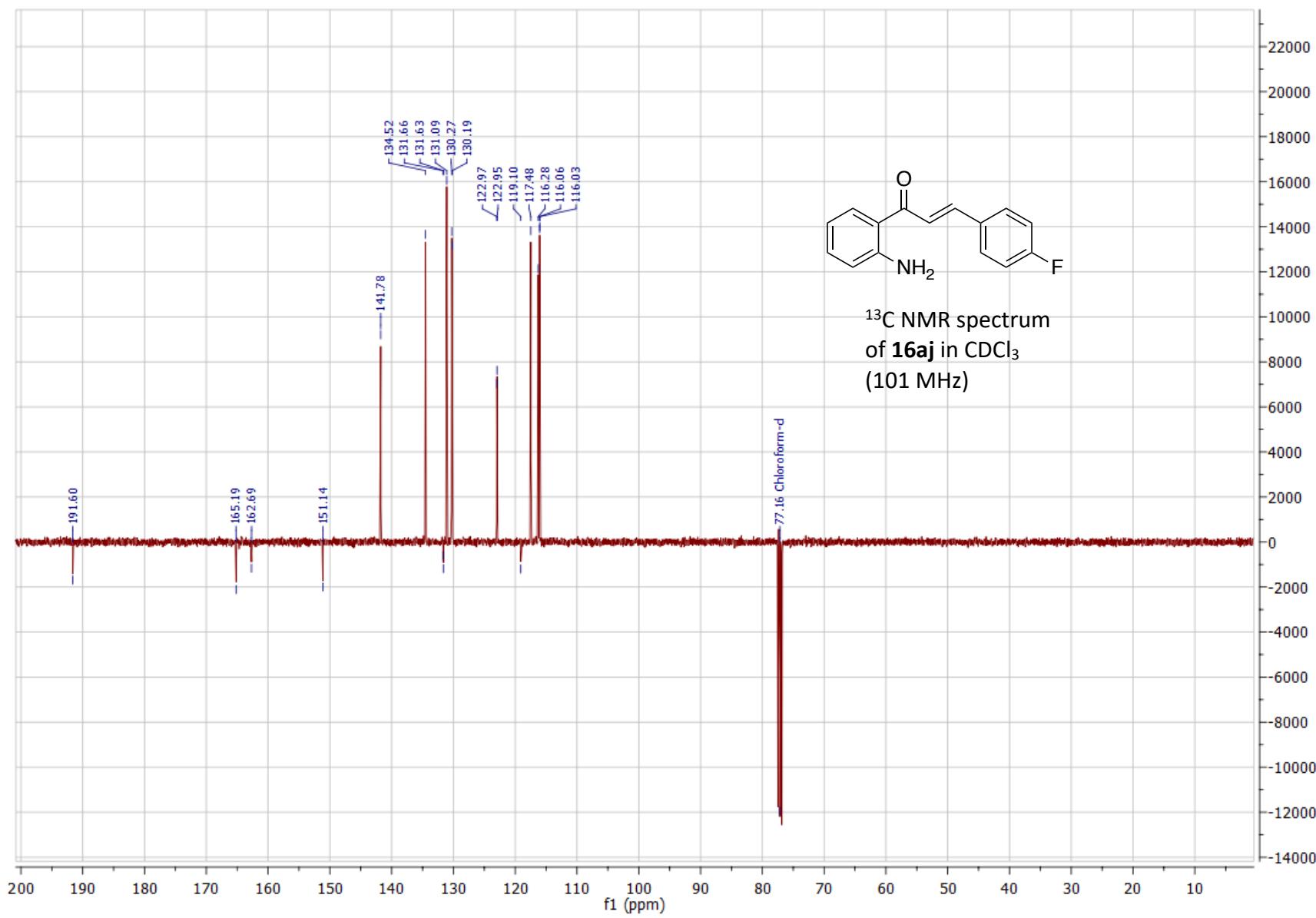


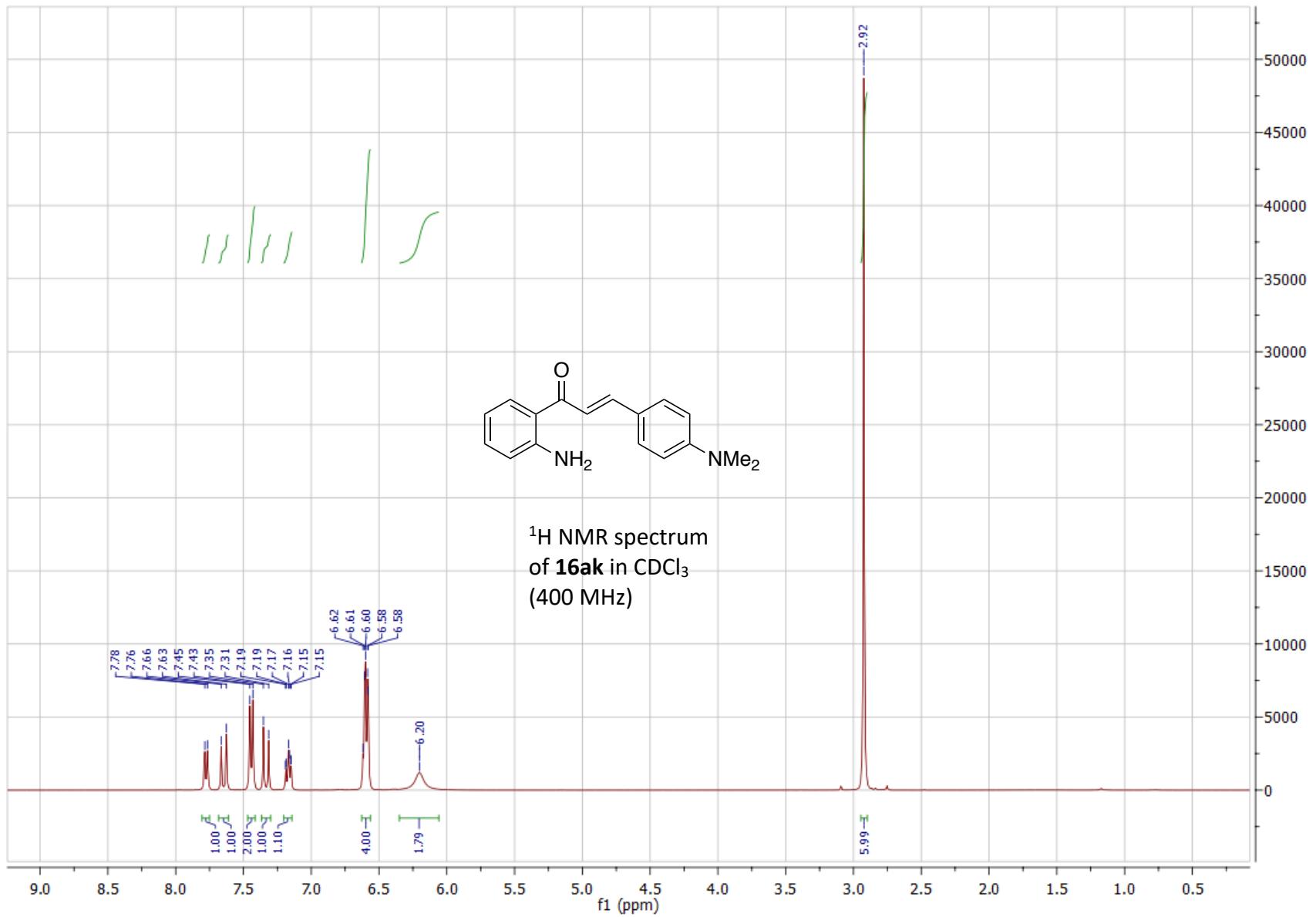


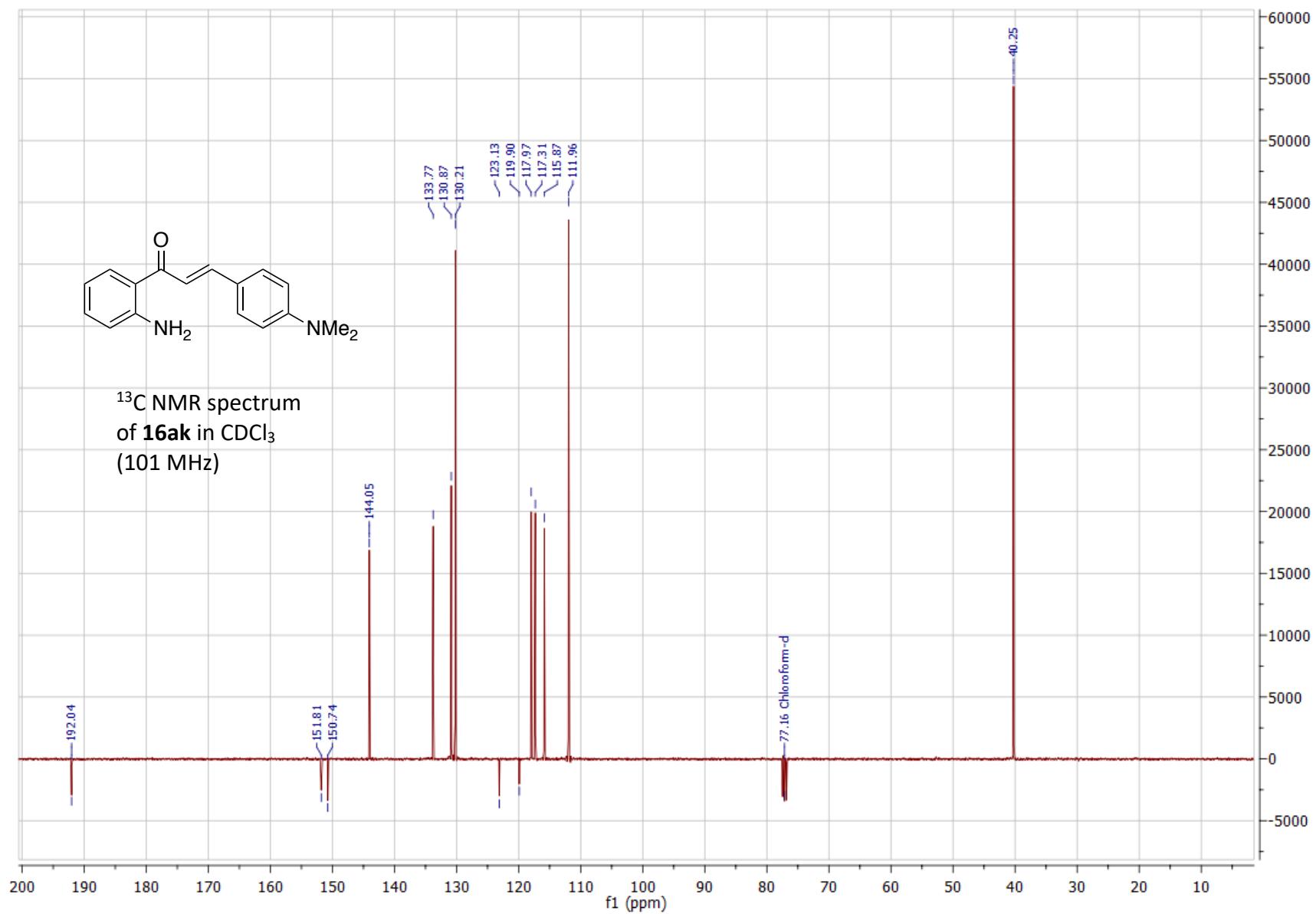


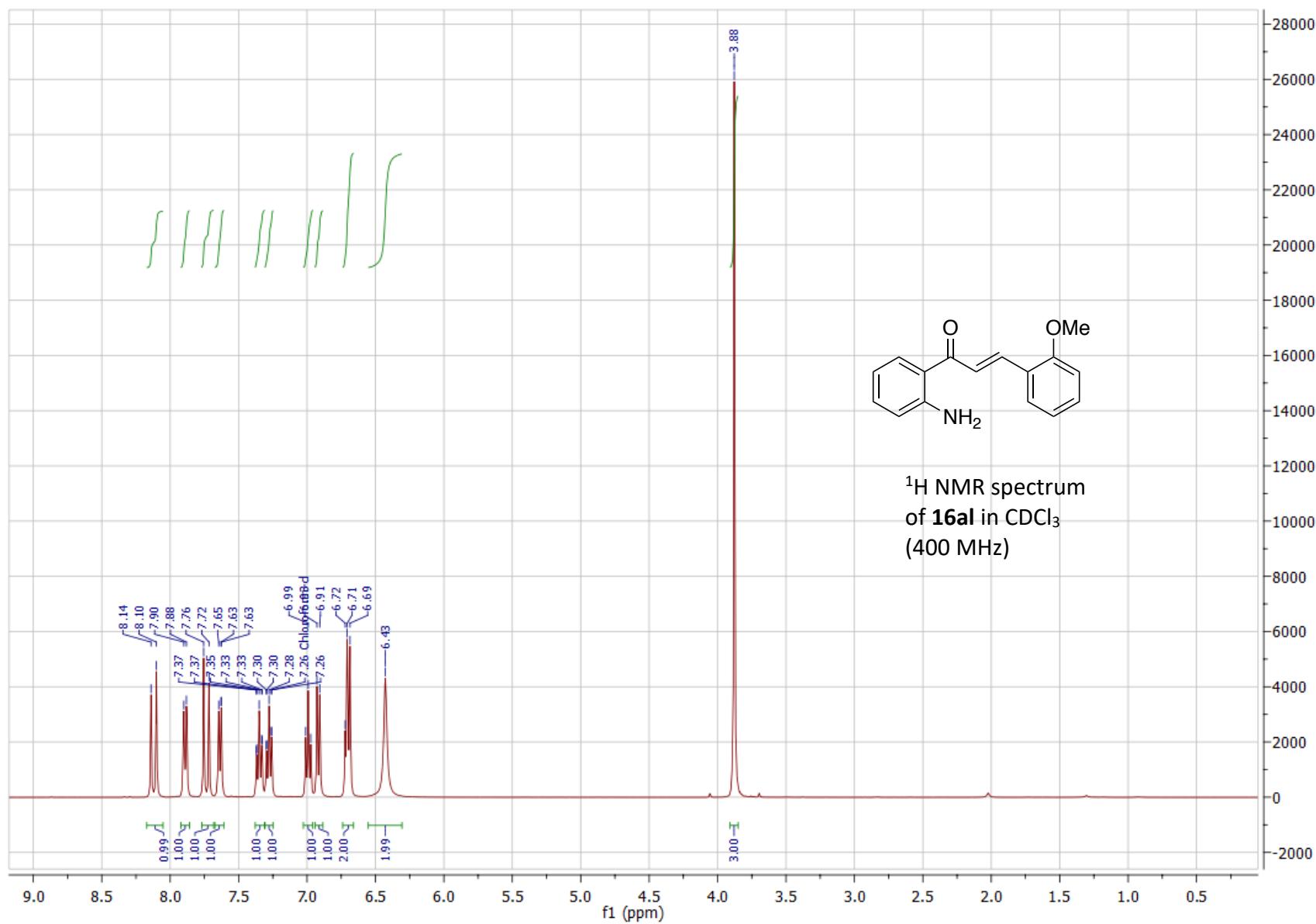


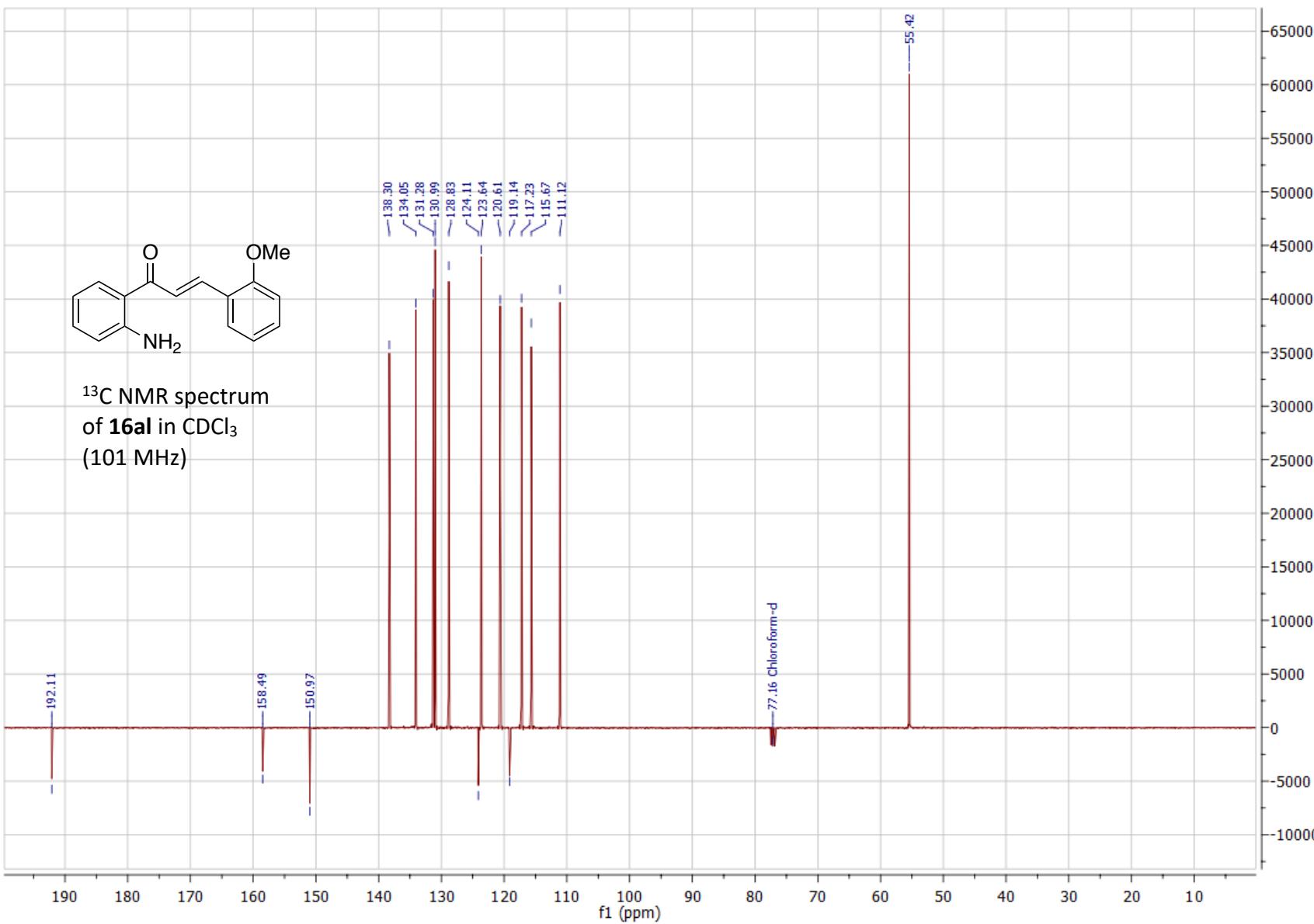


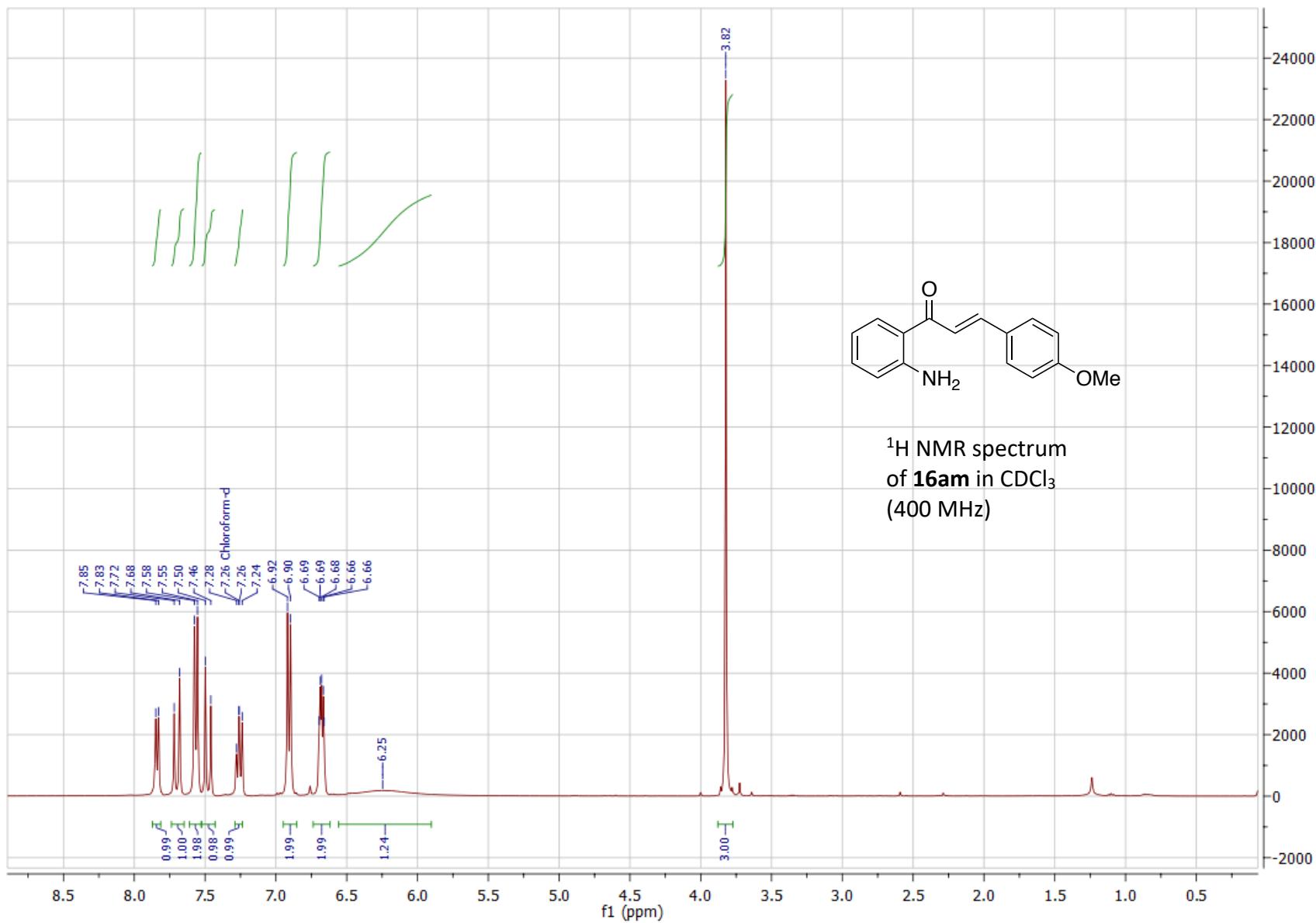


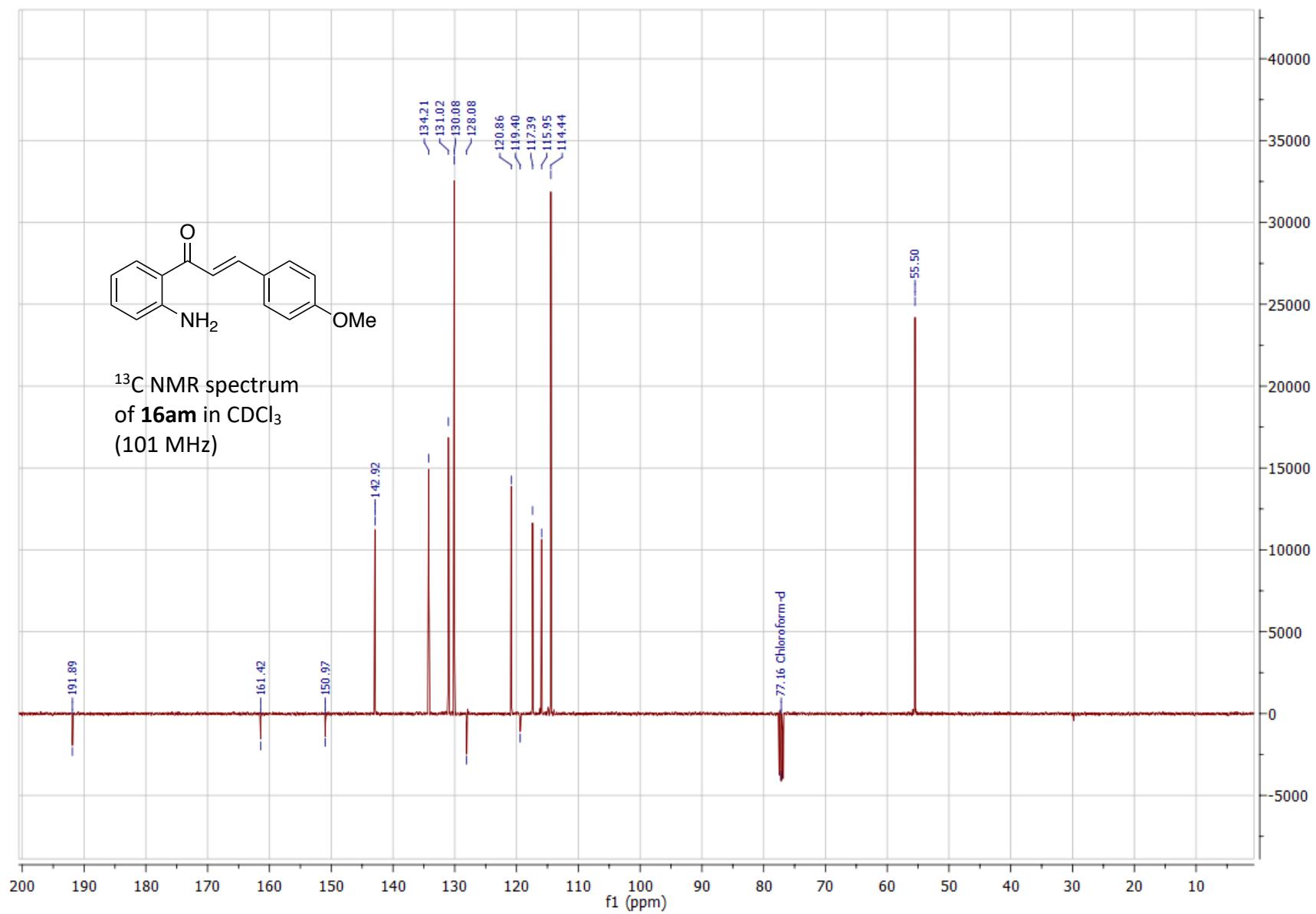


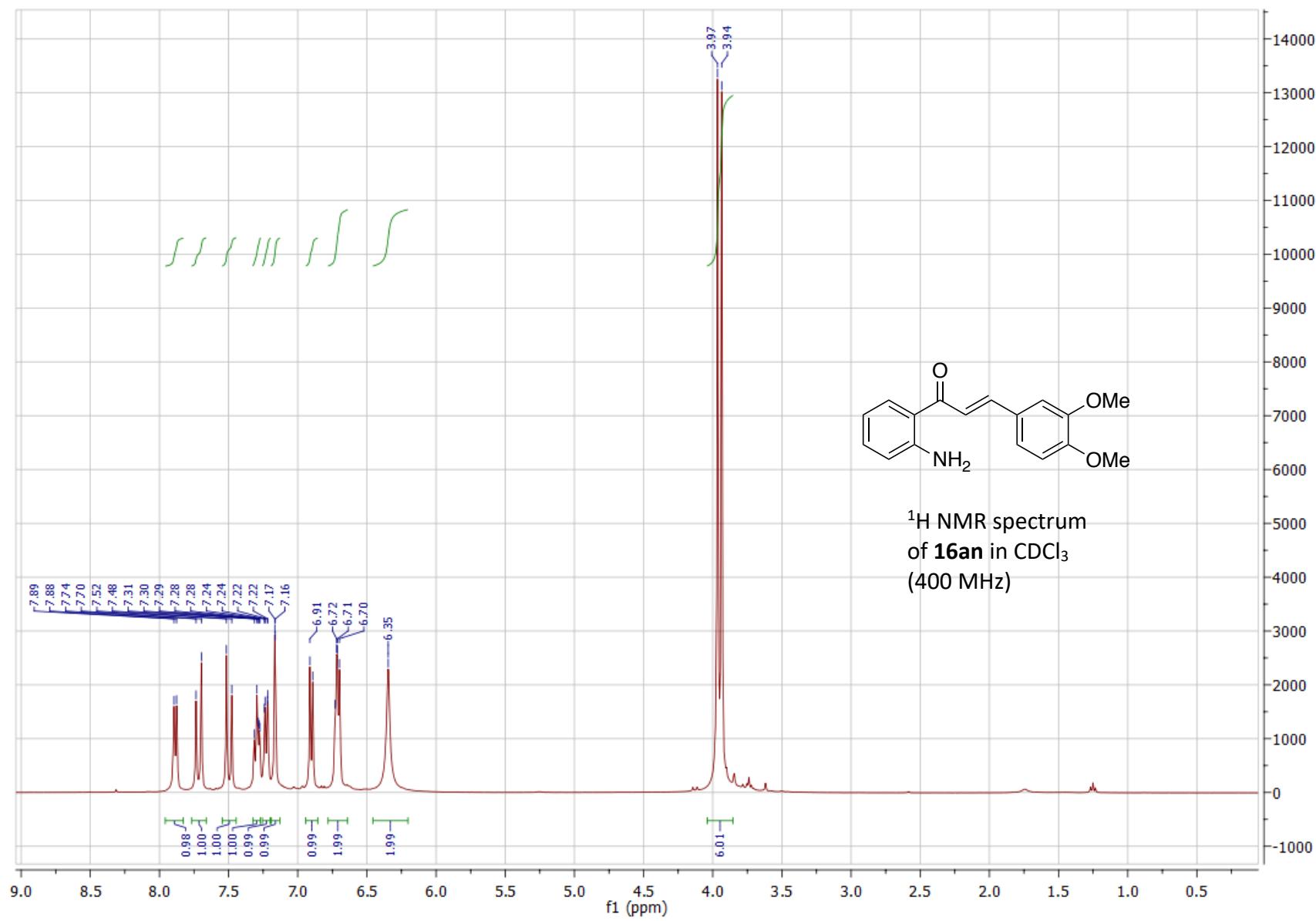


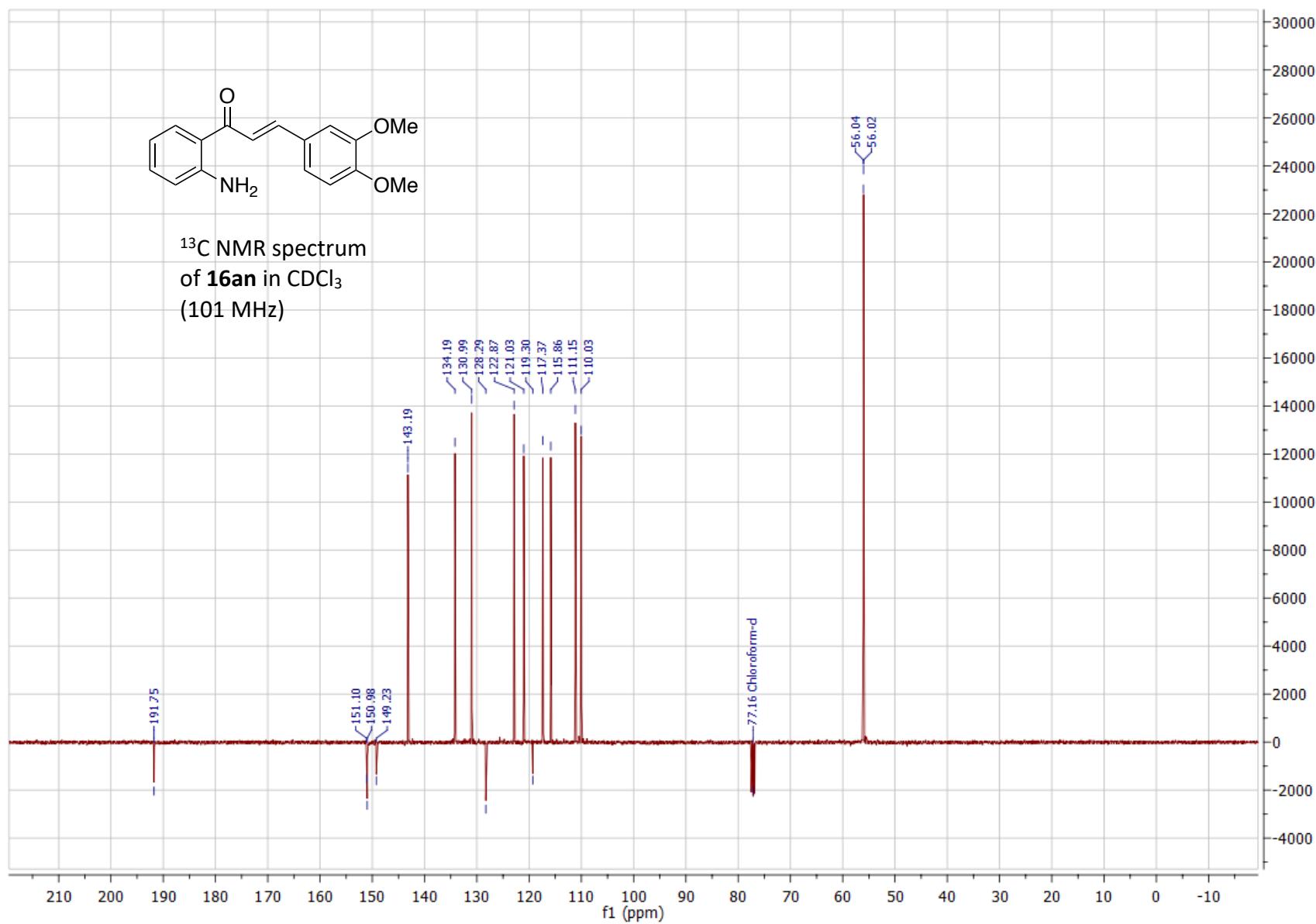


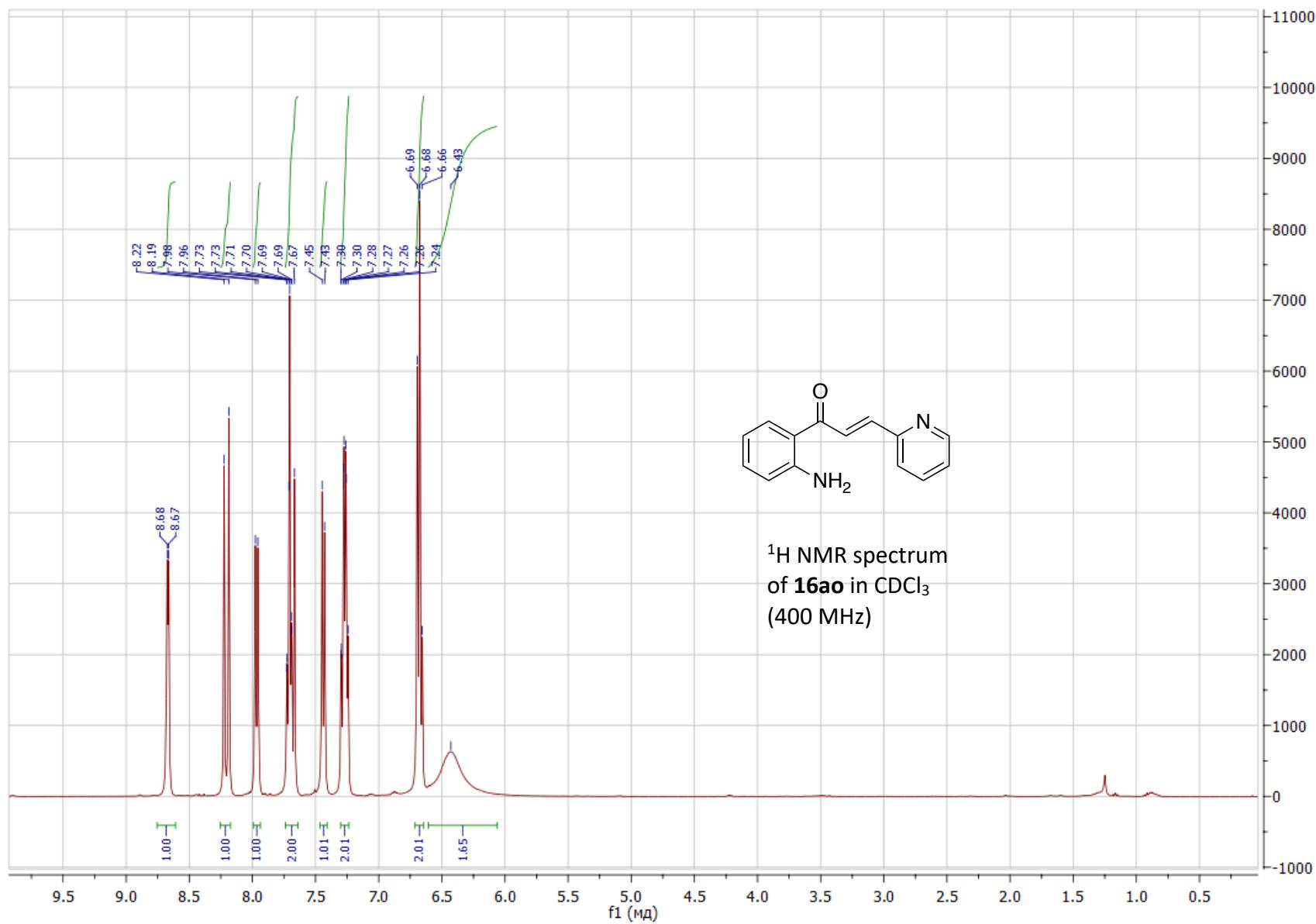


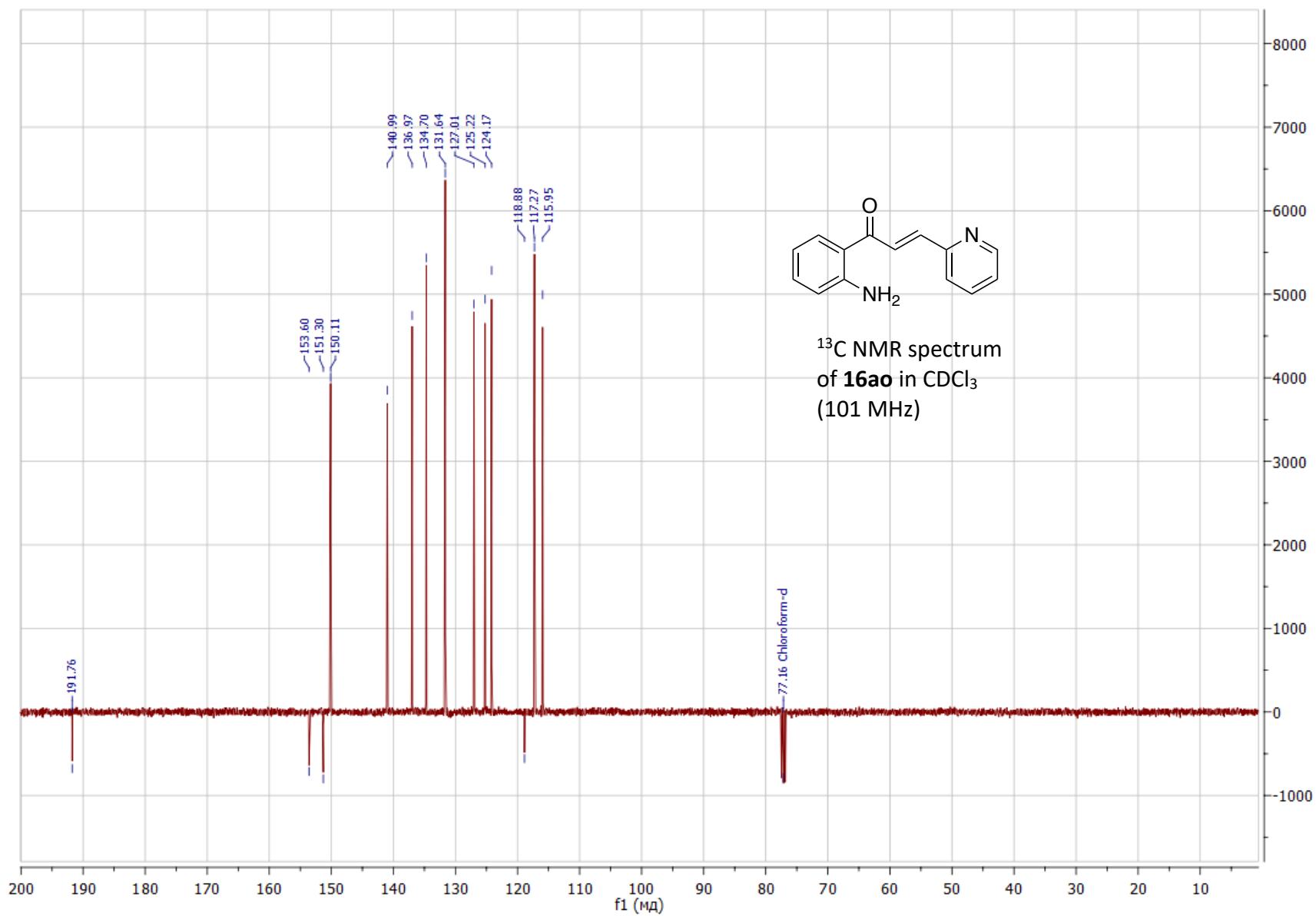


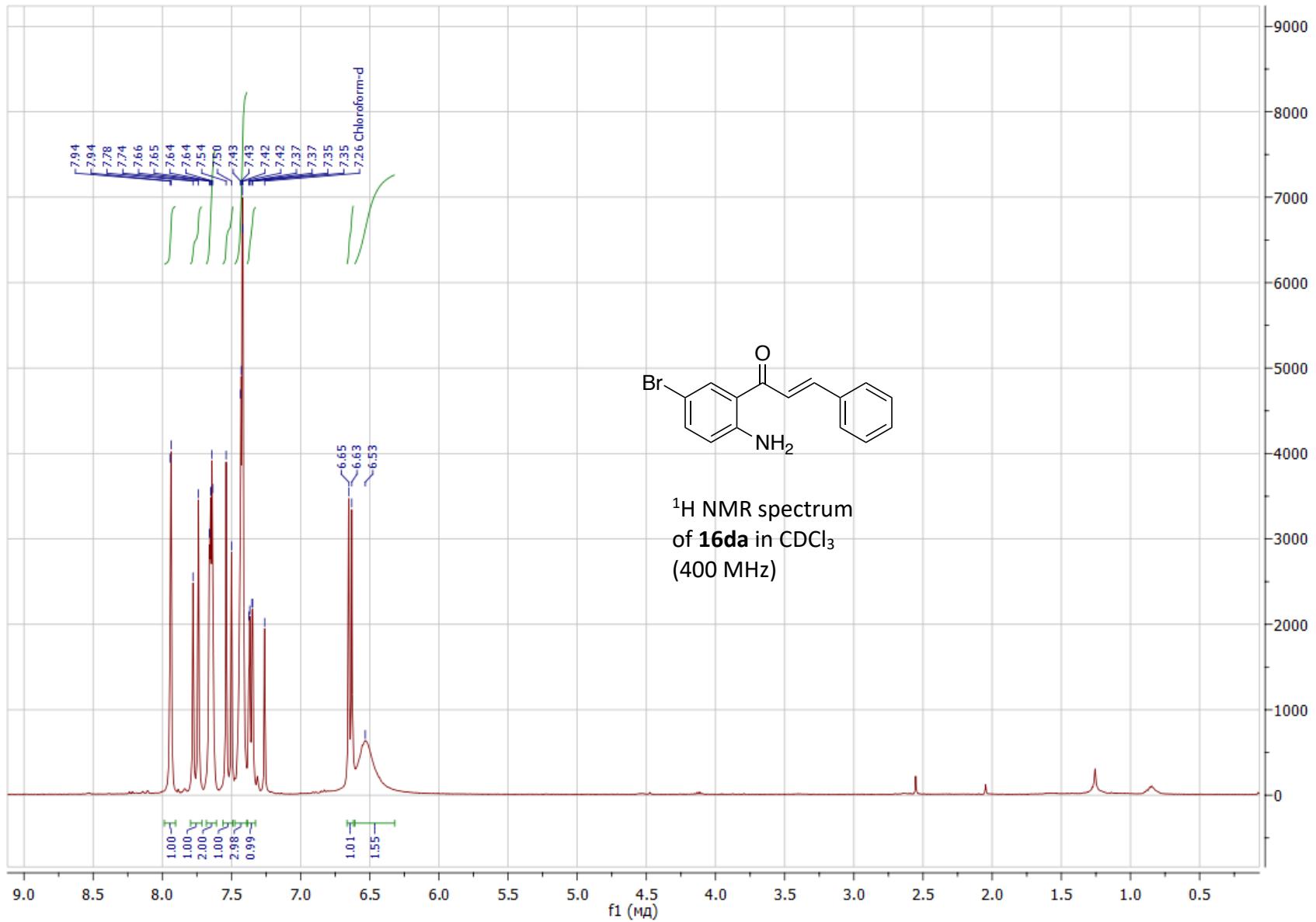


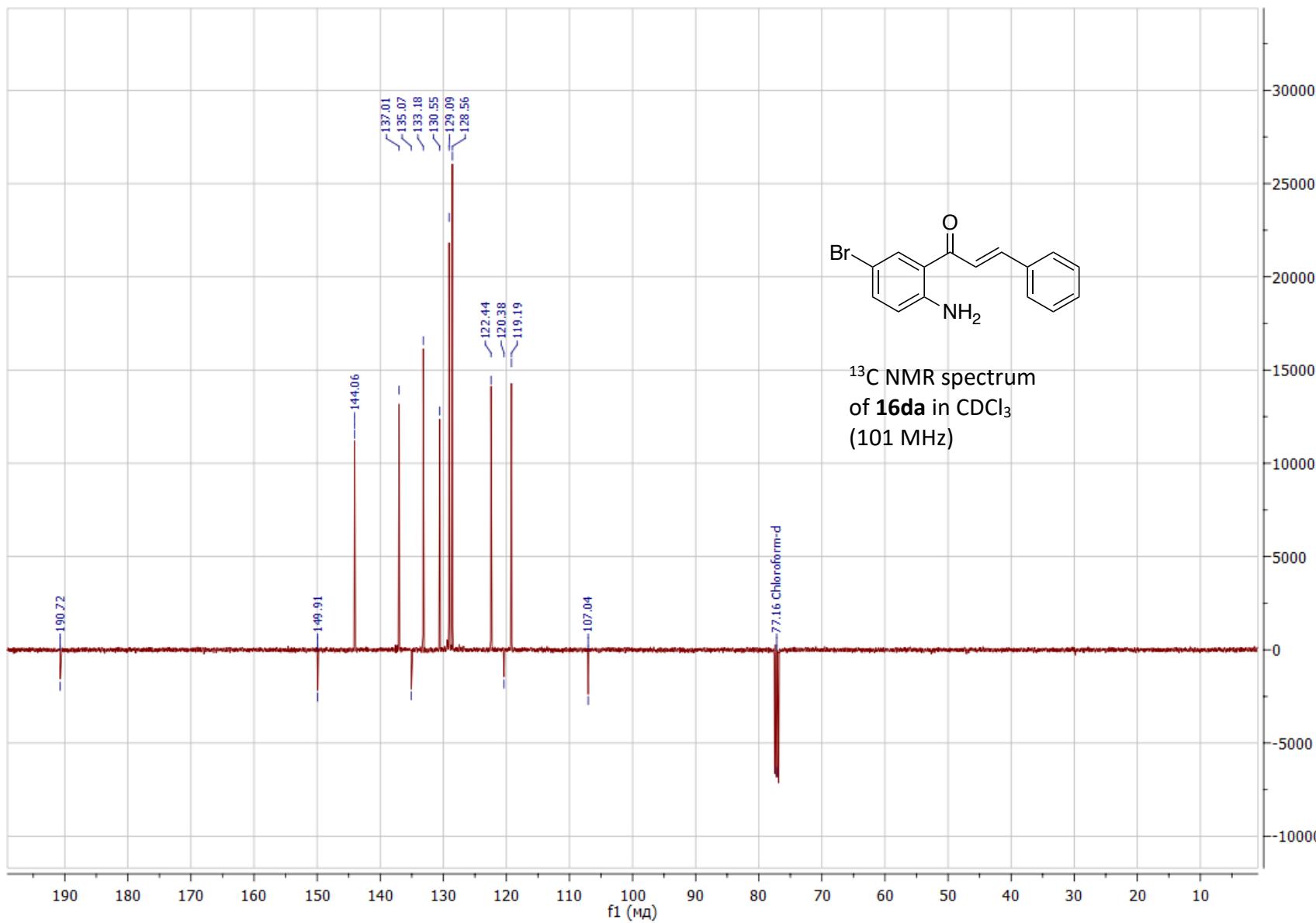




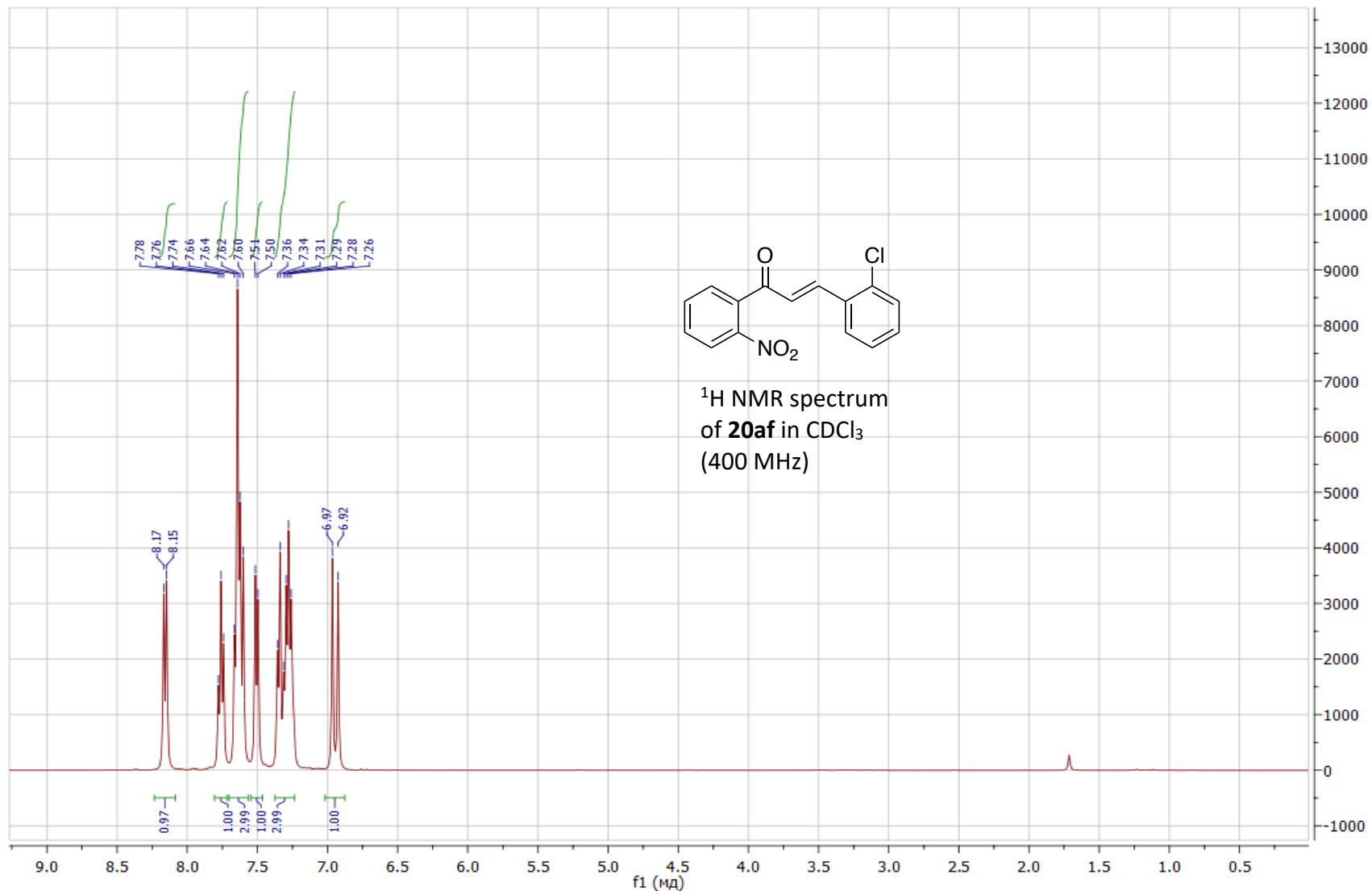


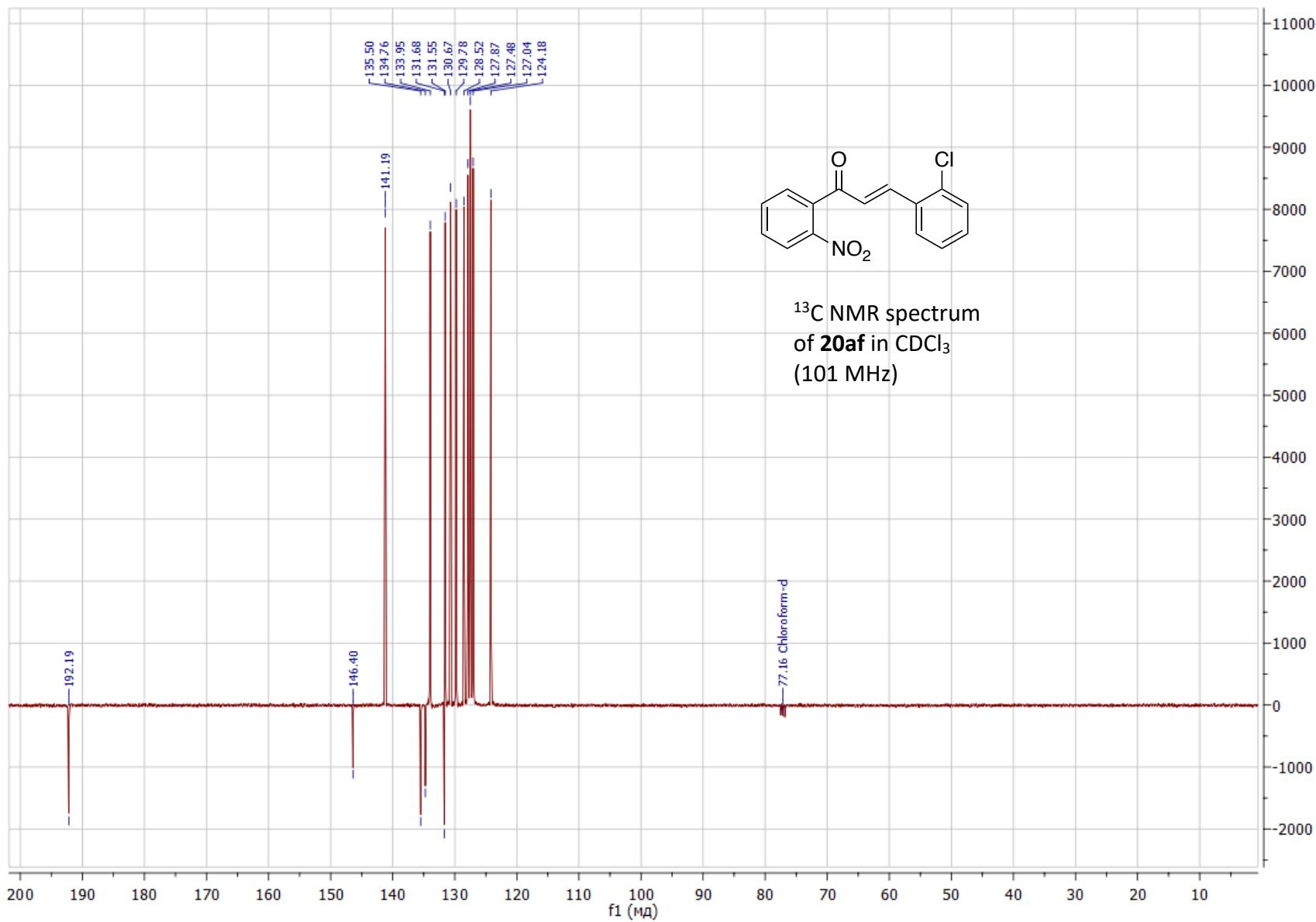


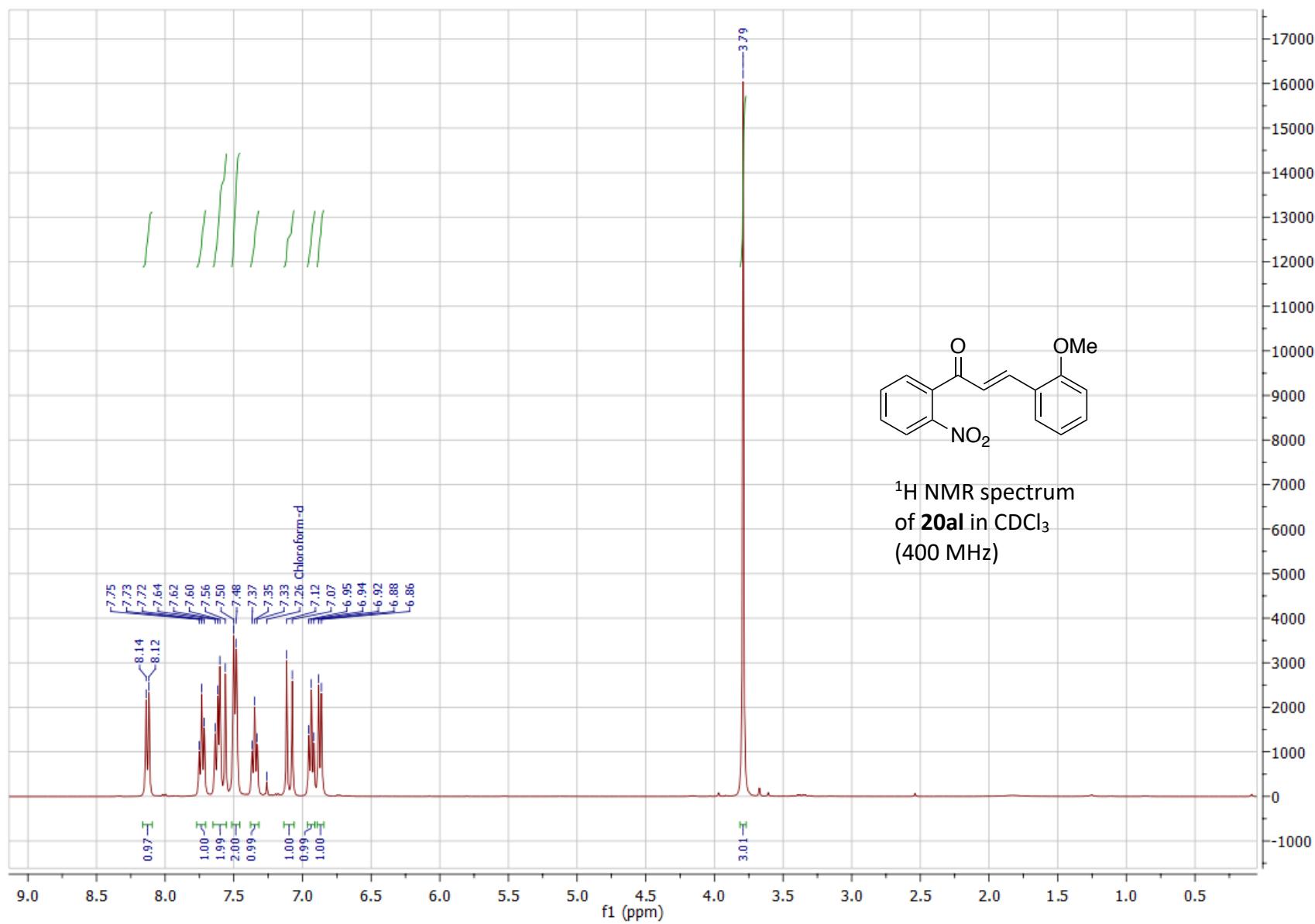


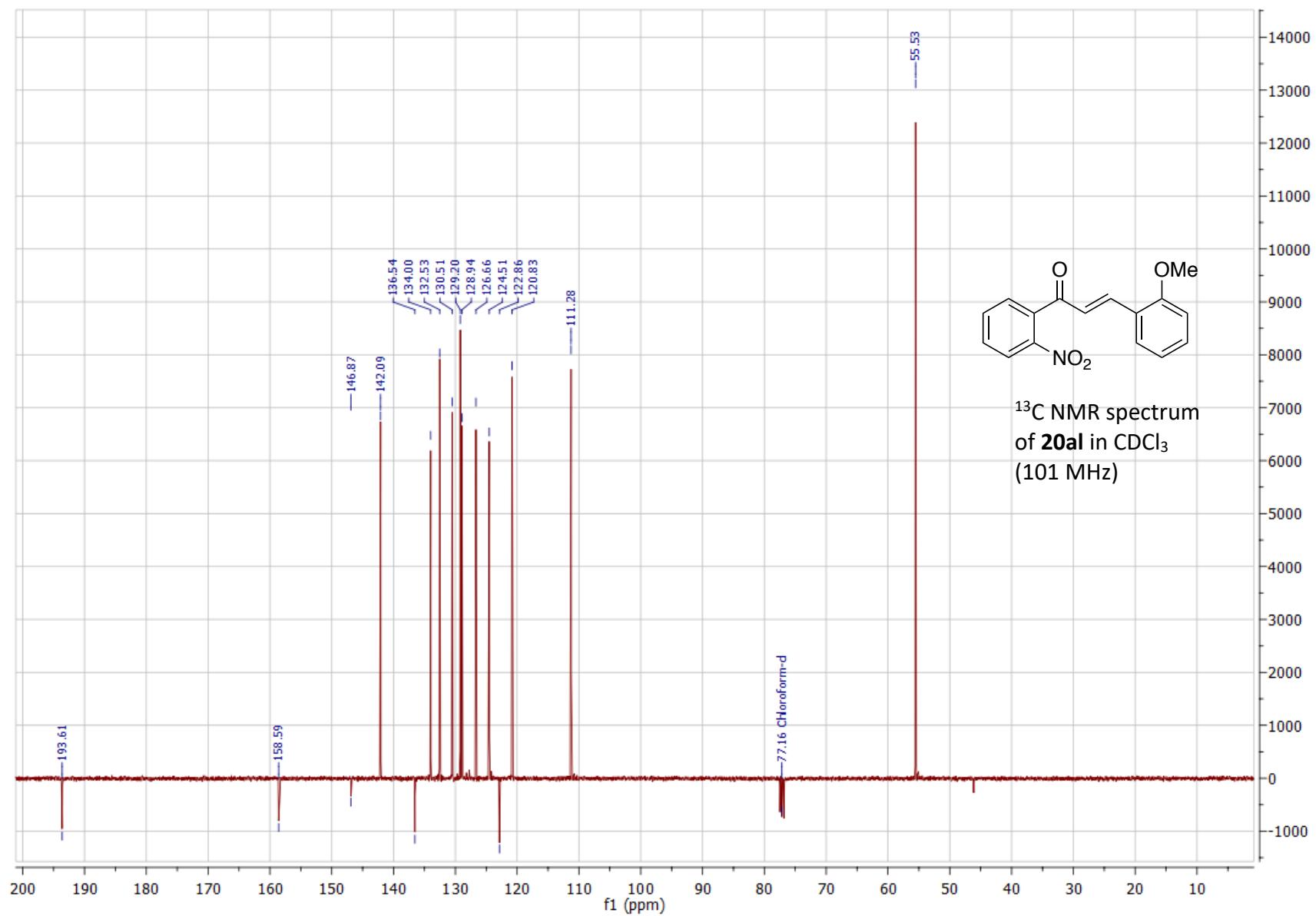


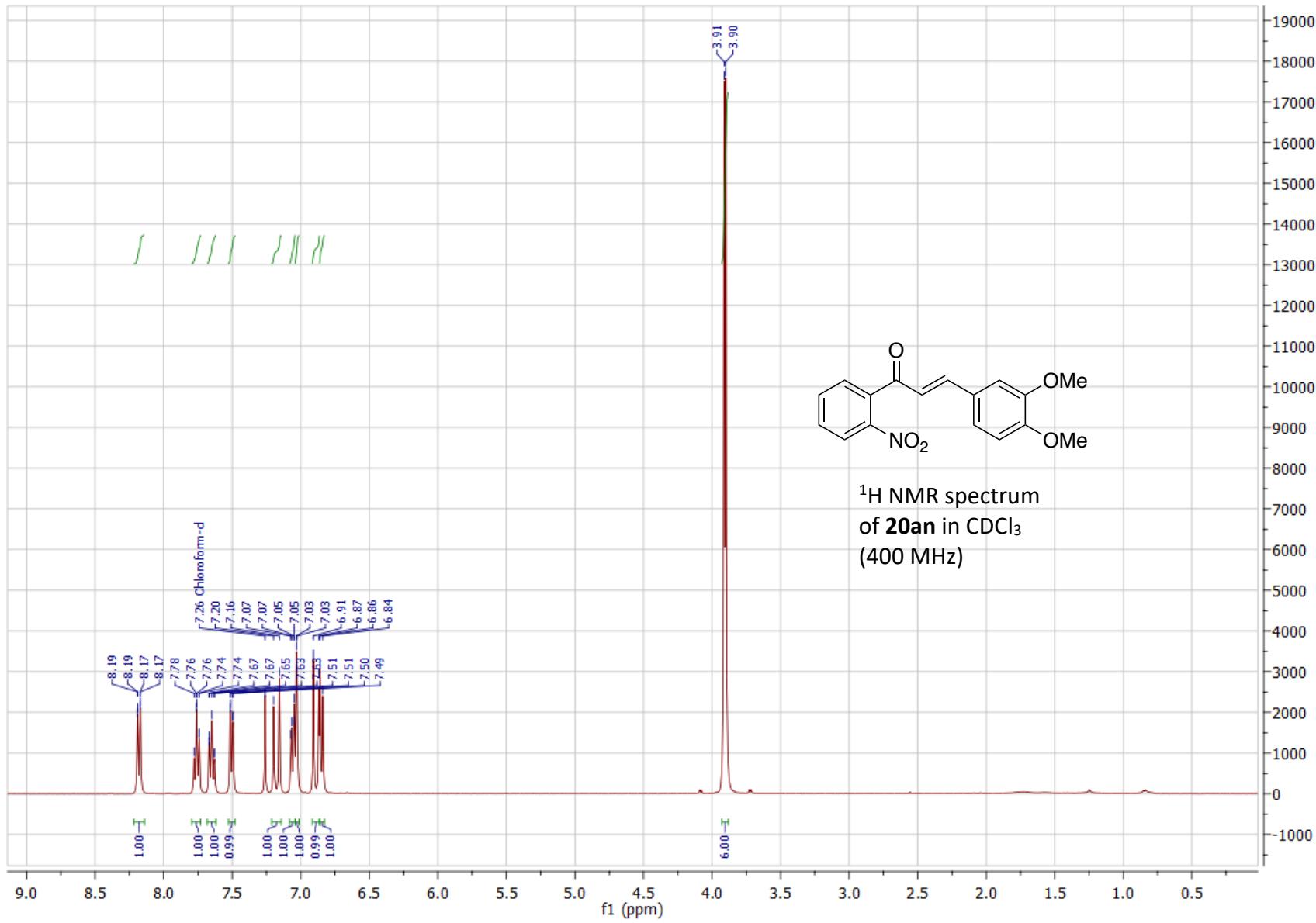
¹H and ¹³C Spectral Charts of (*E*)-1-(2-nitrophenyl)-3-arylprop-2-en-1-ones 20

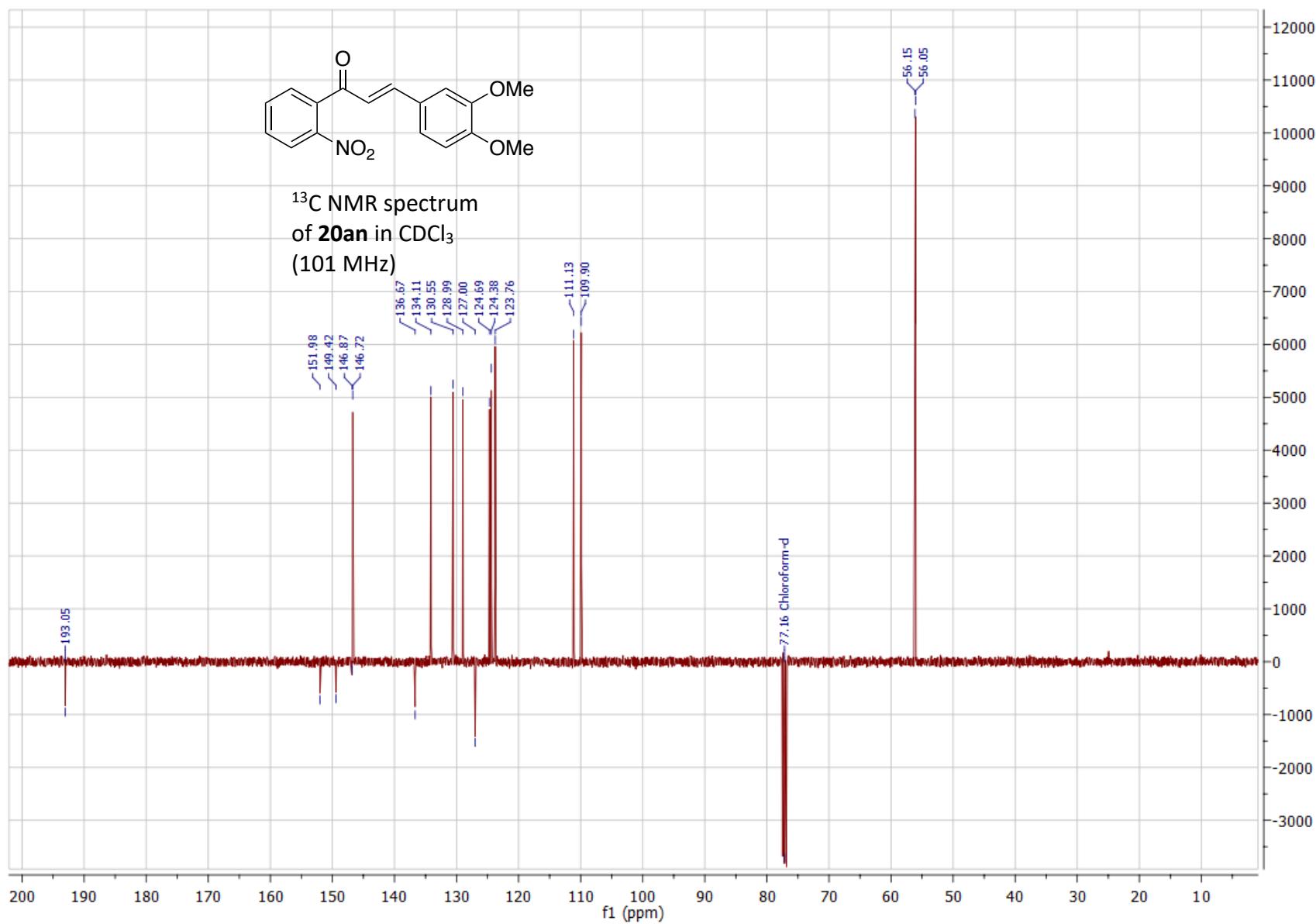


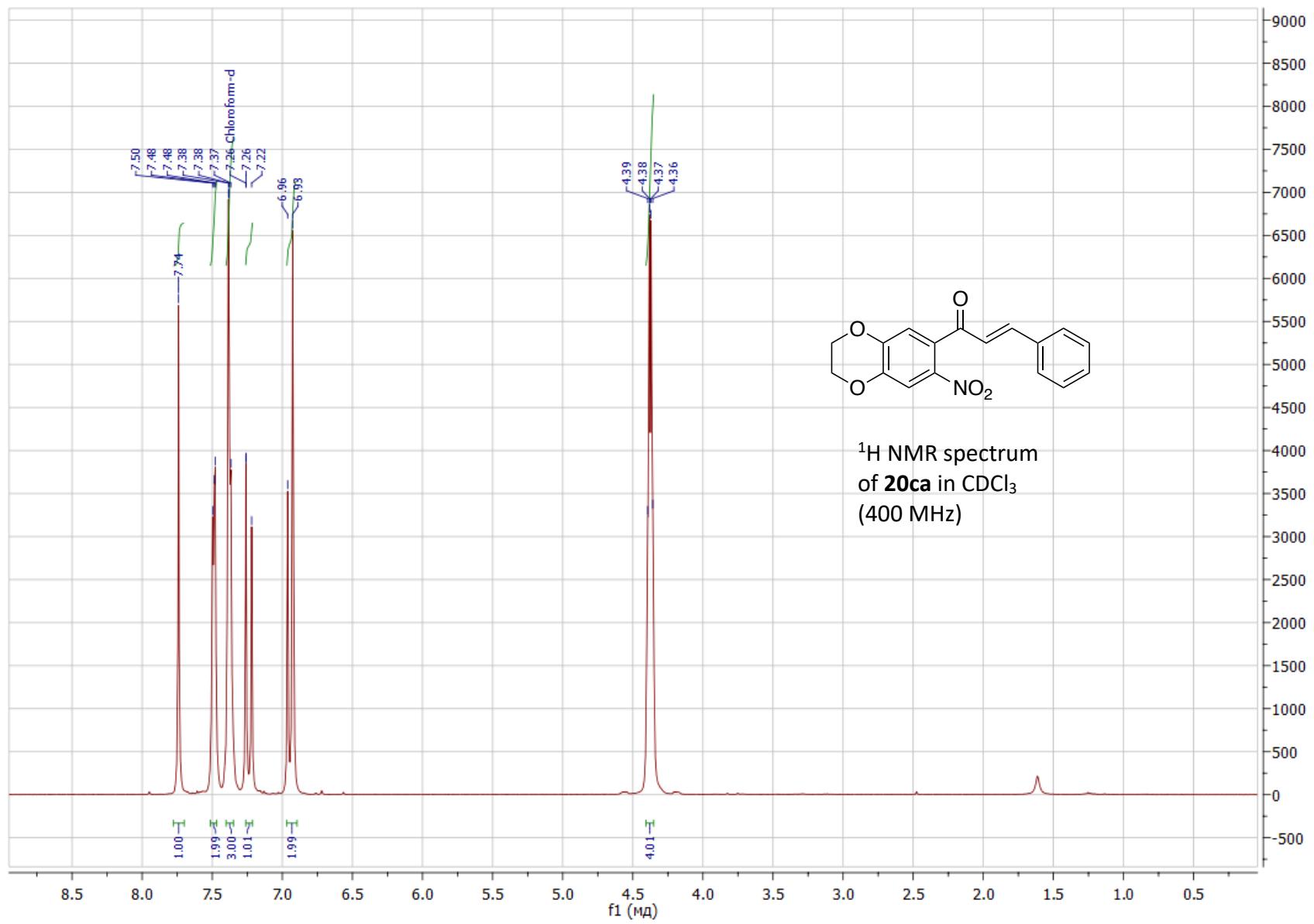


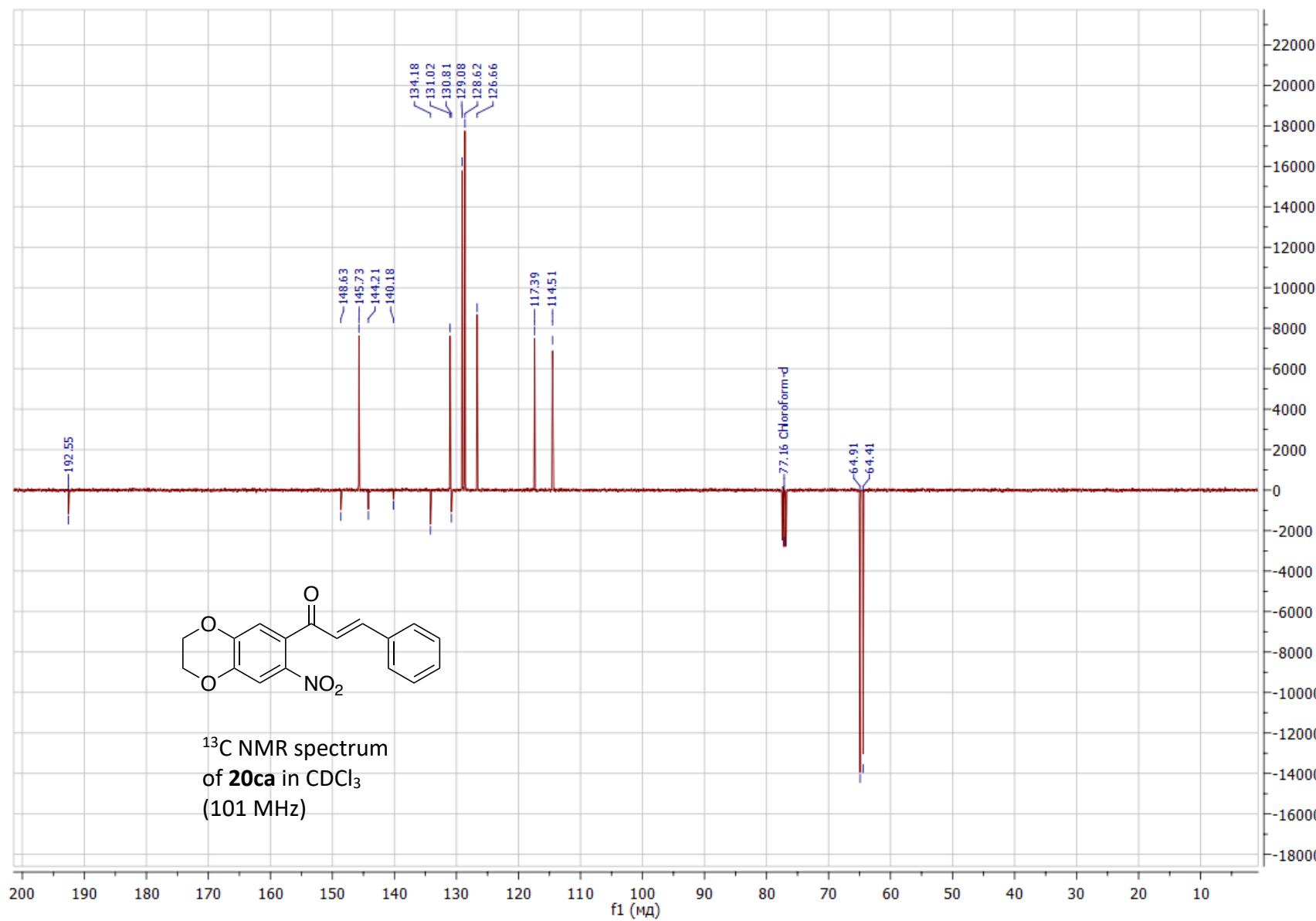




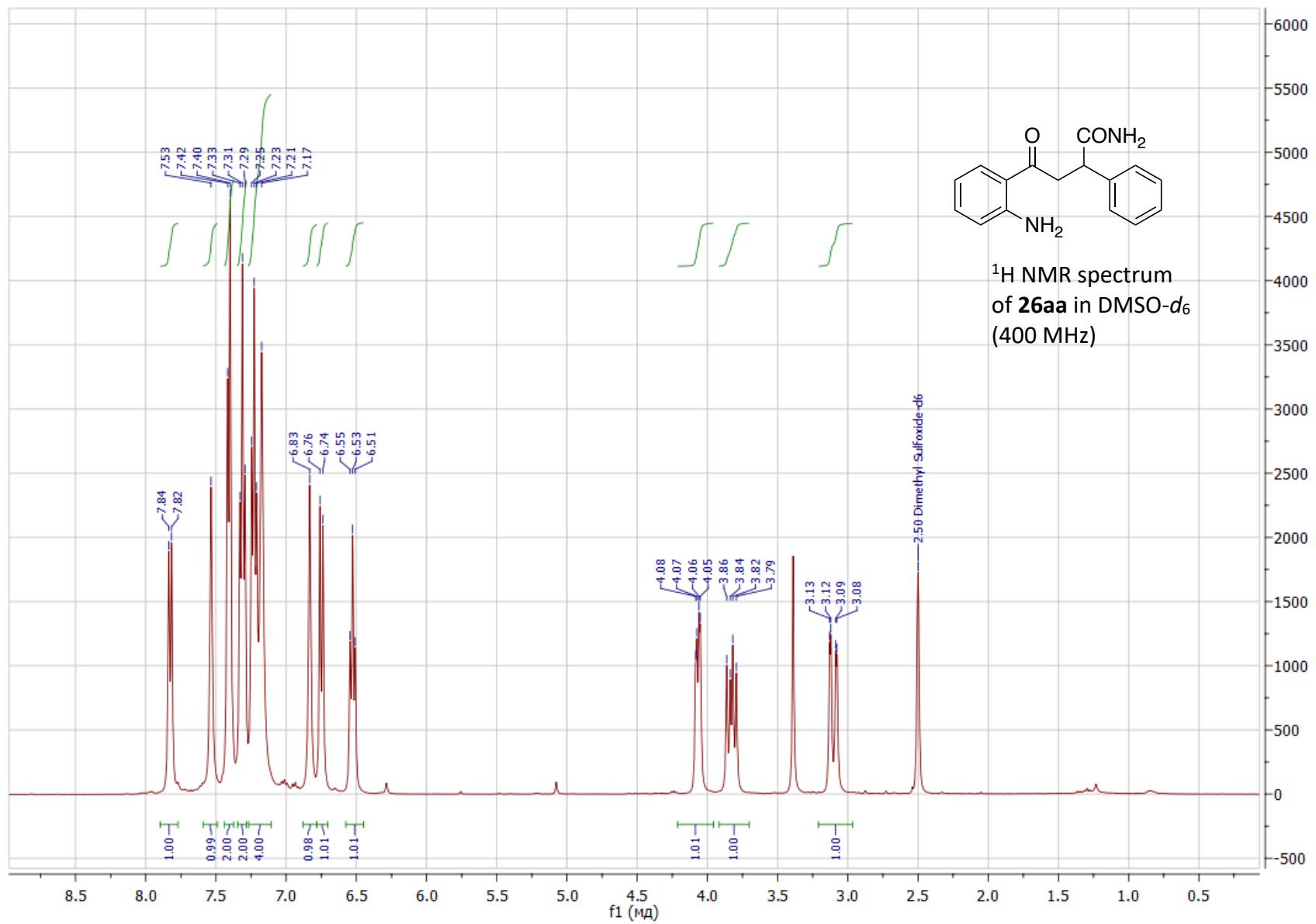


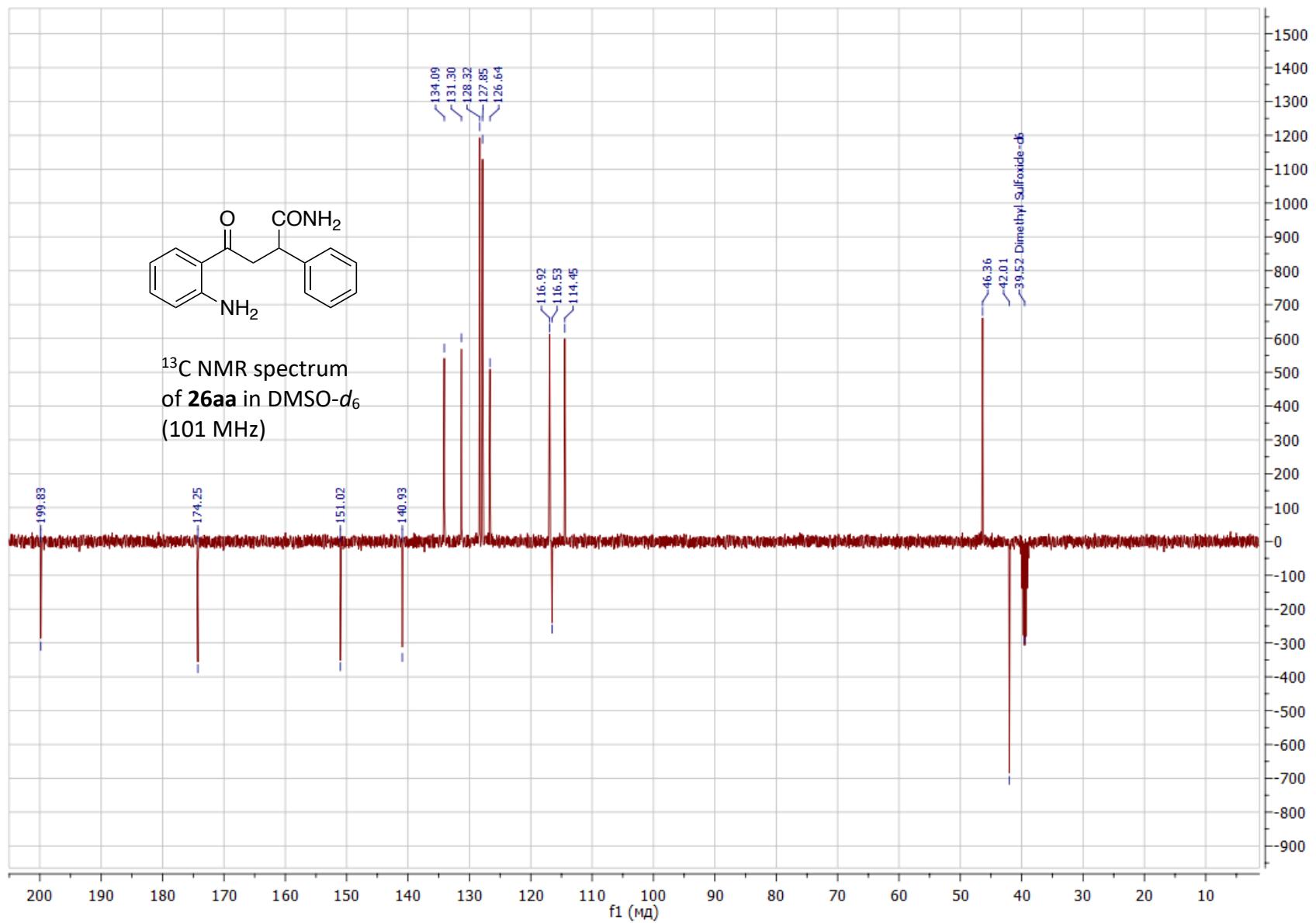


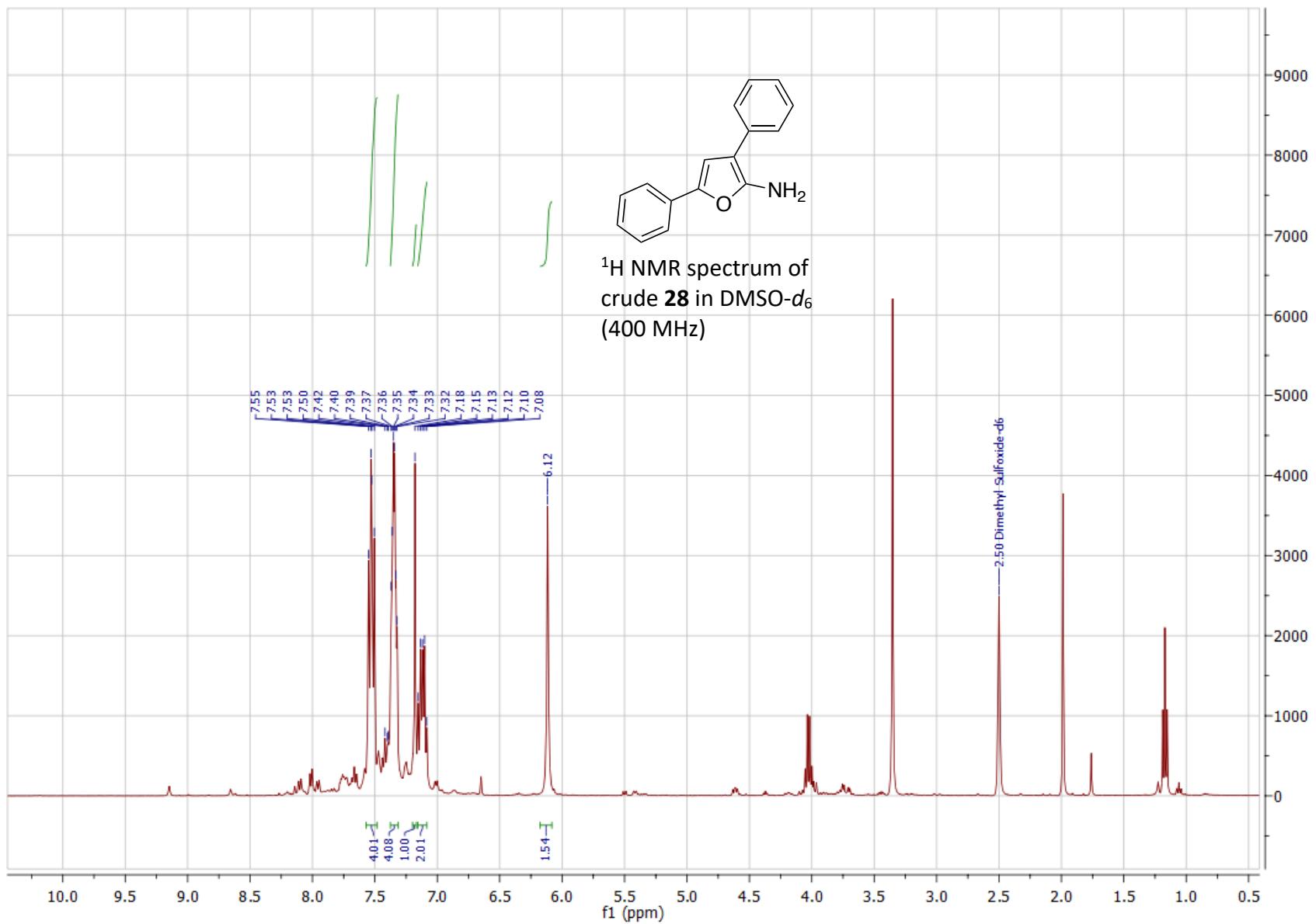


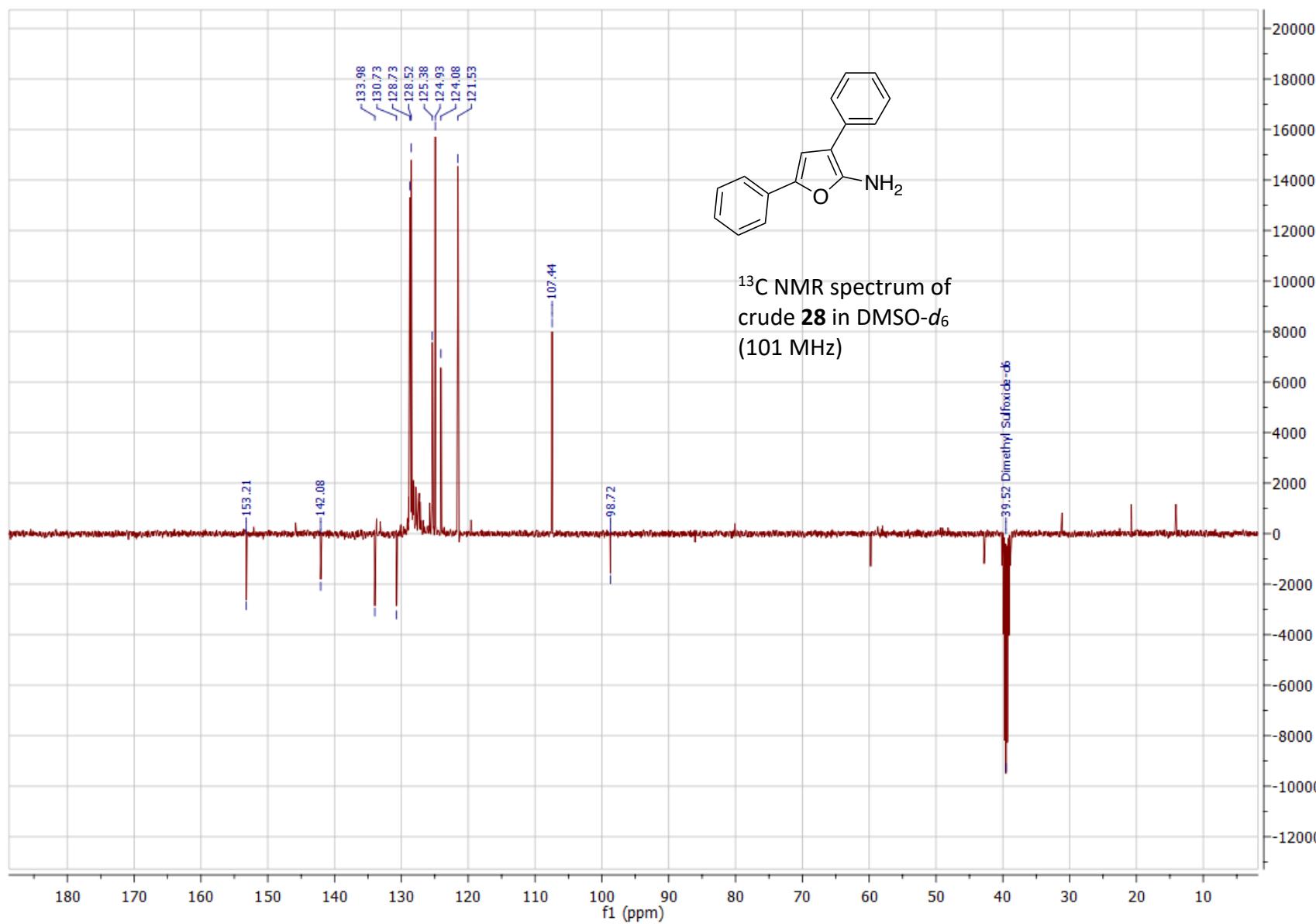


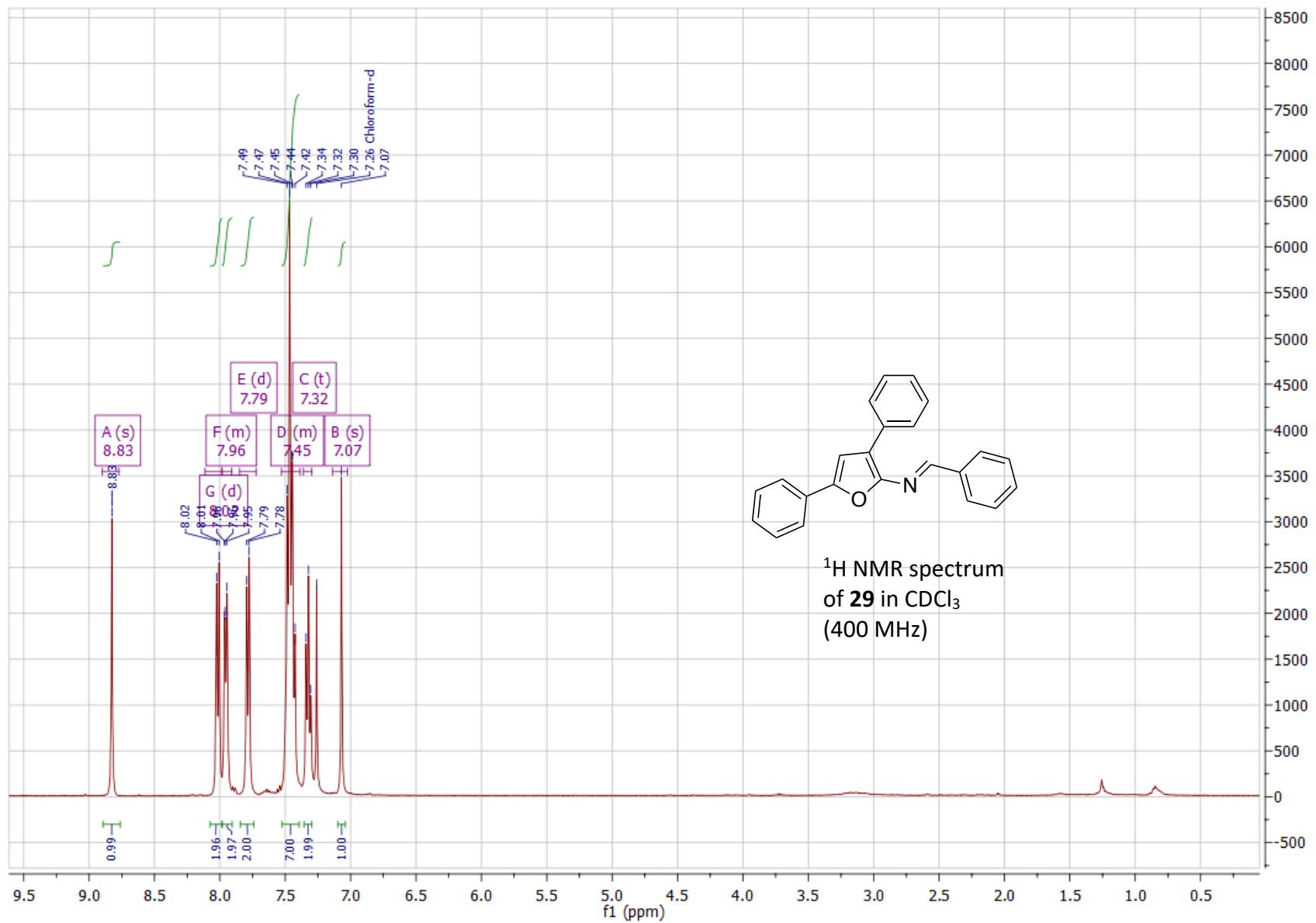
^{13}C NMR spectrum
of **20ca** in CDCl_3
(101 MHz)

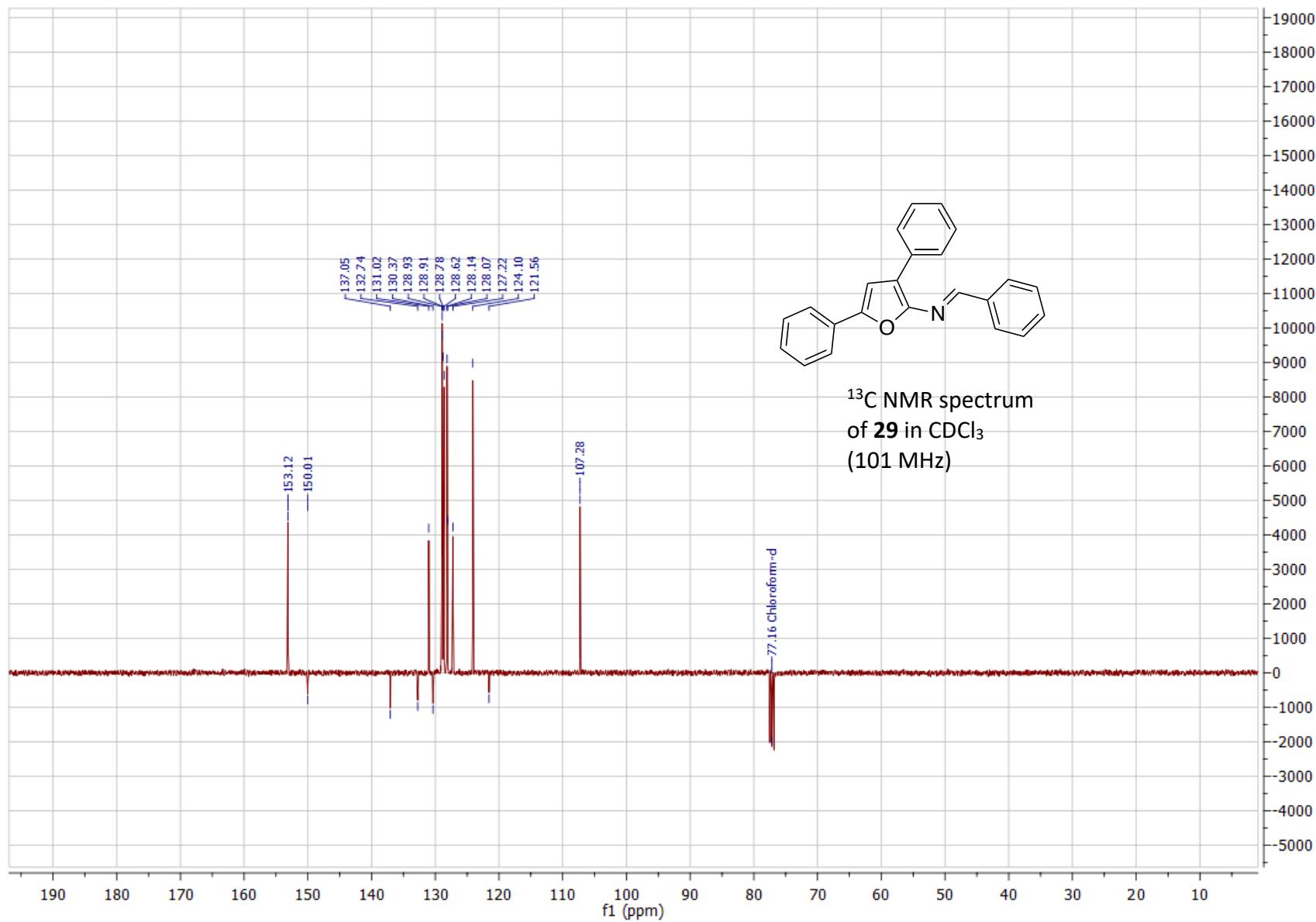


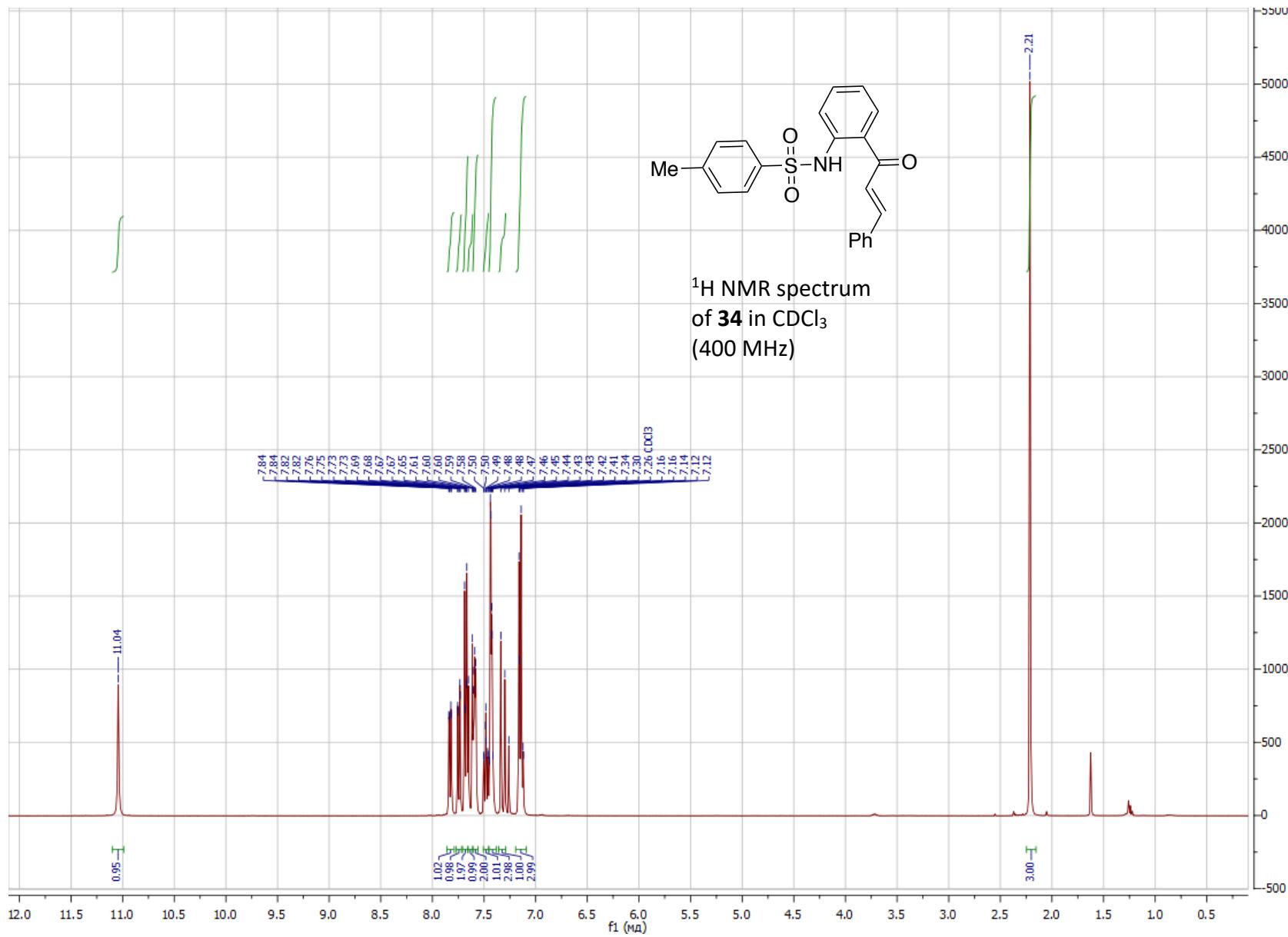


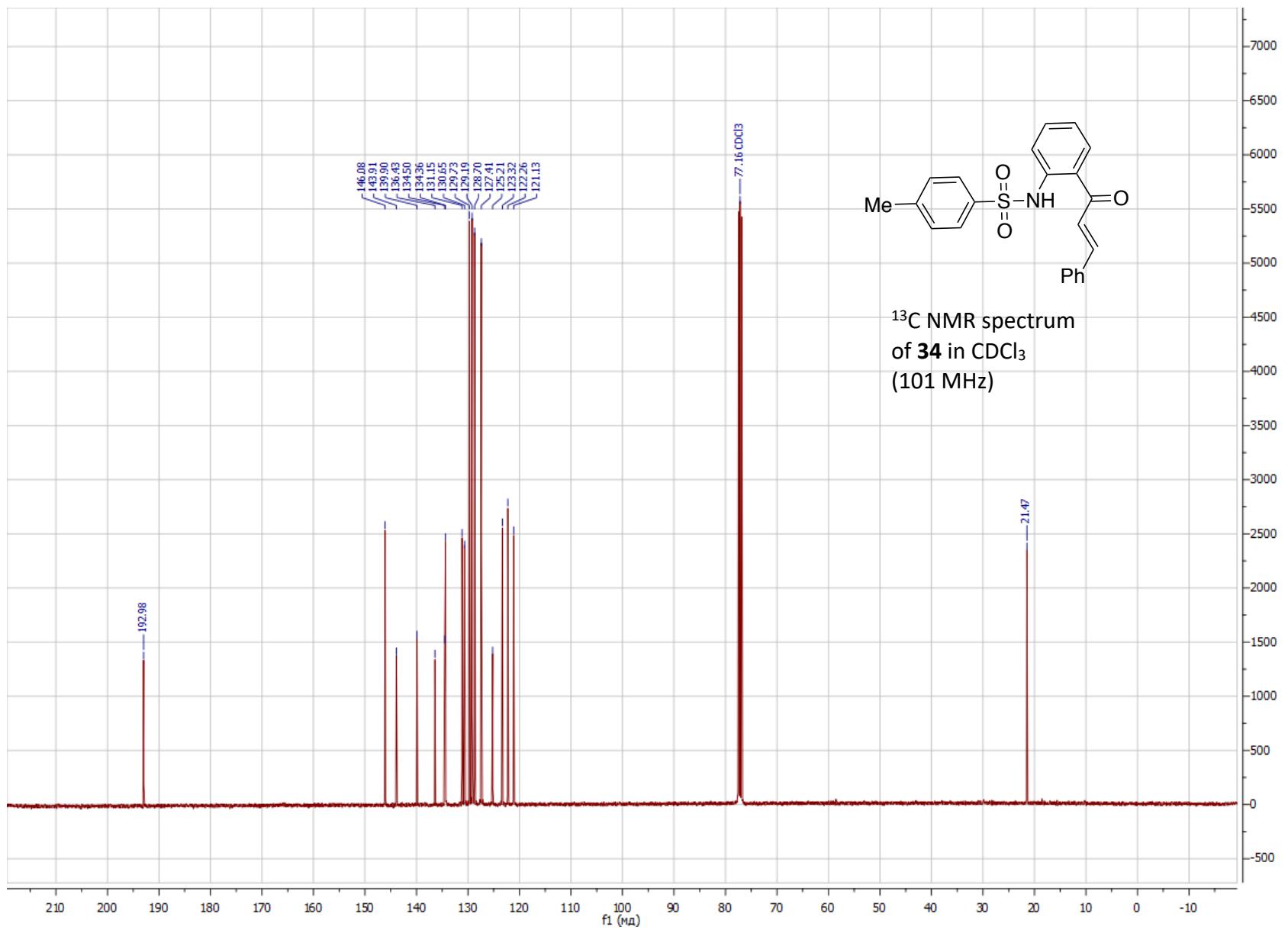


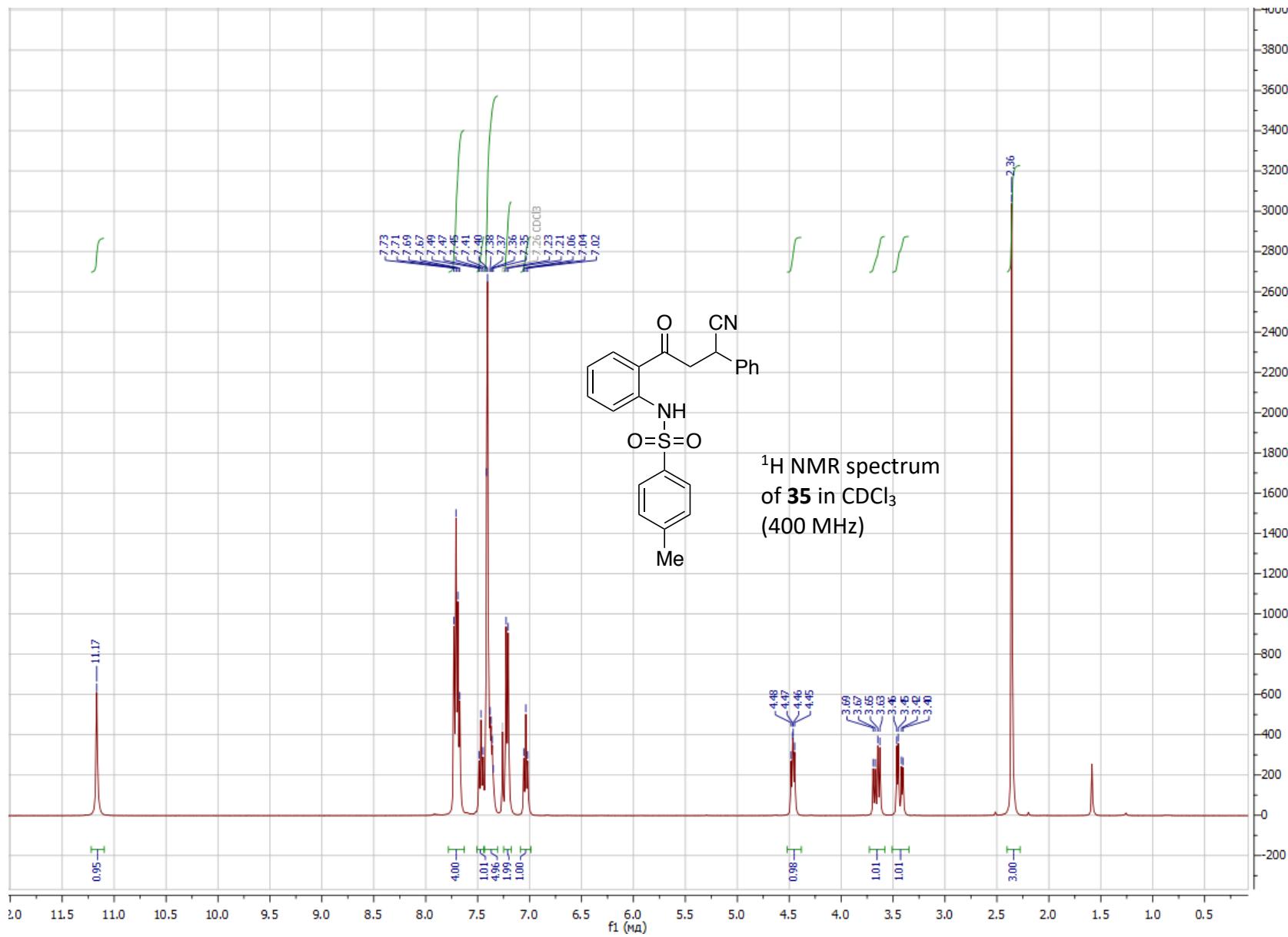


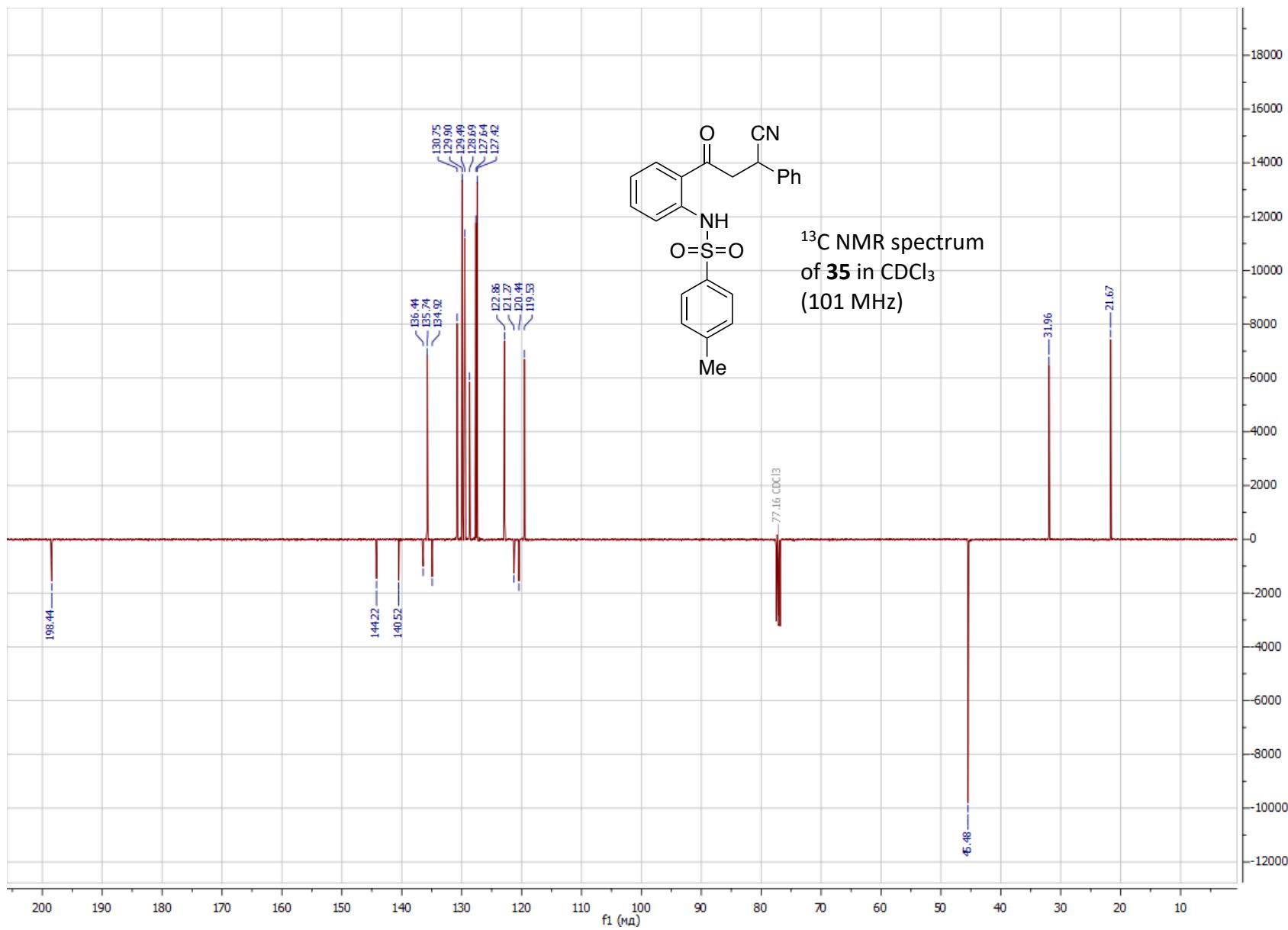


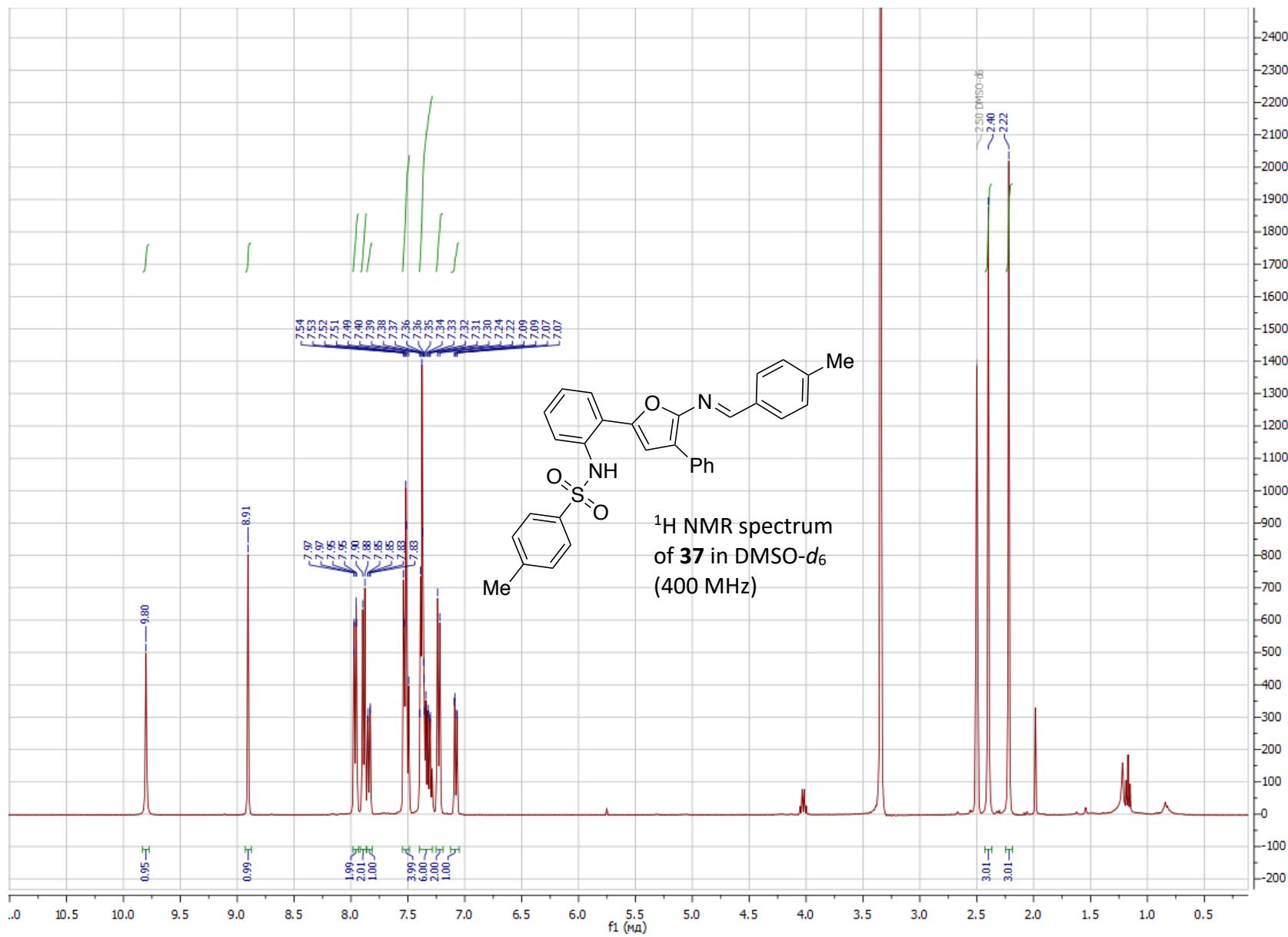


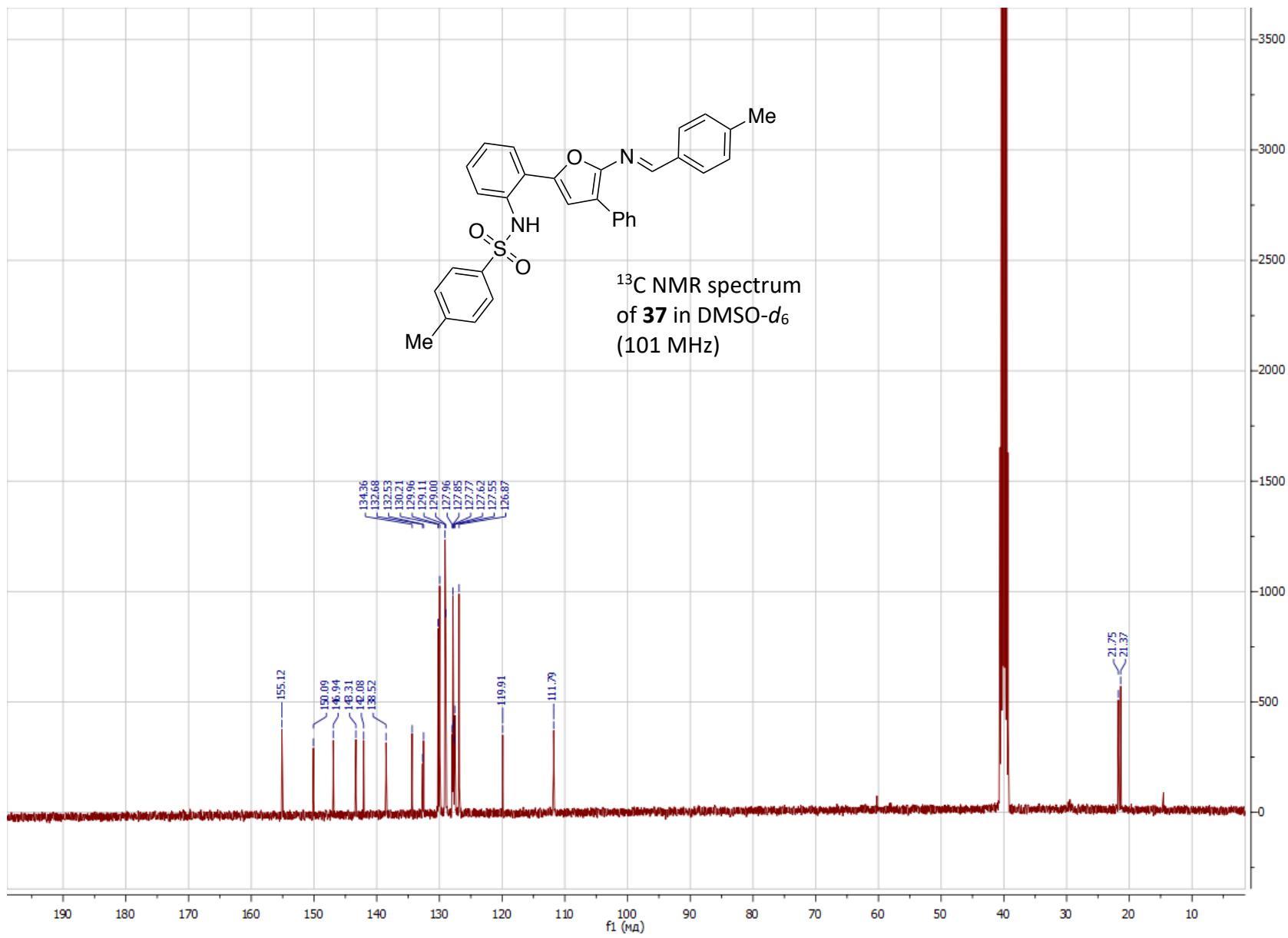


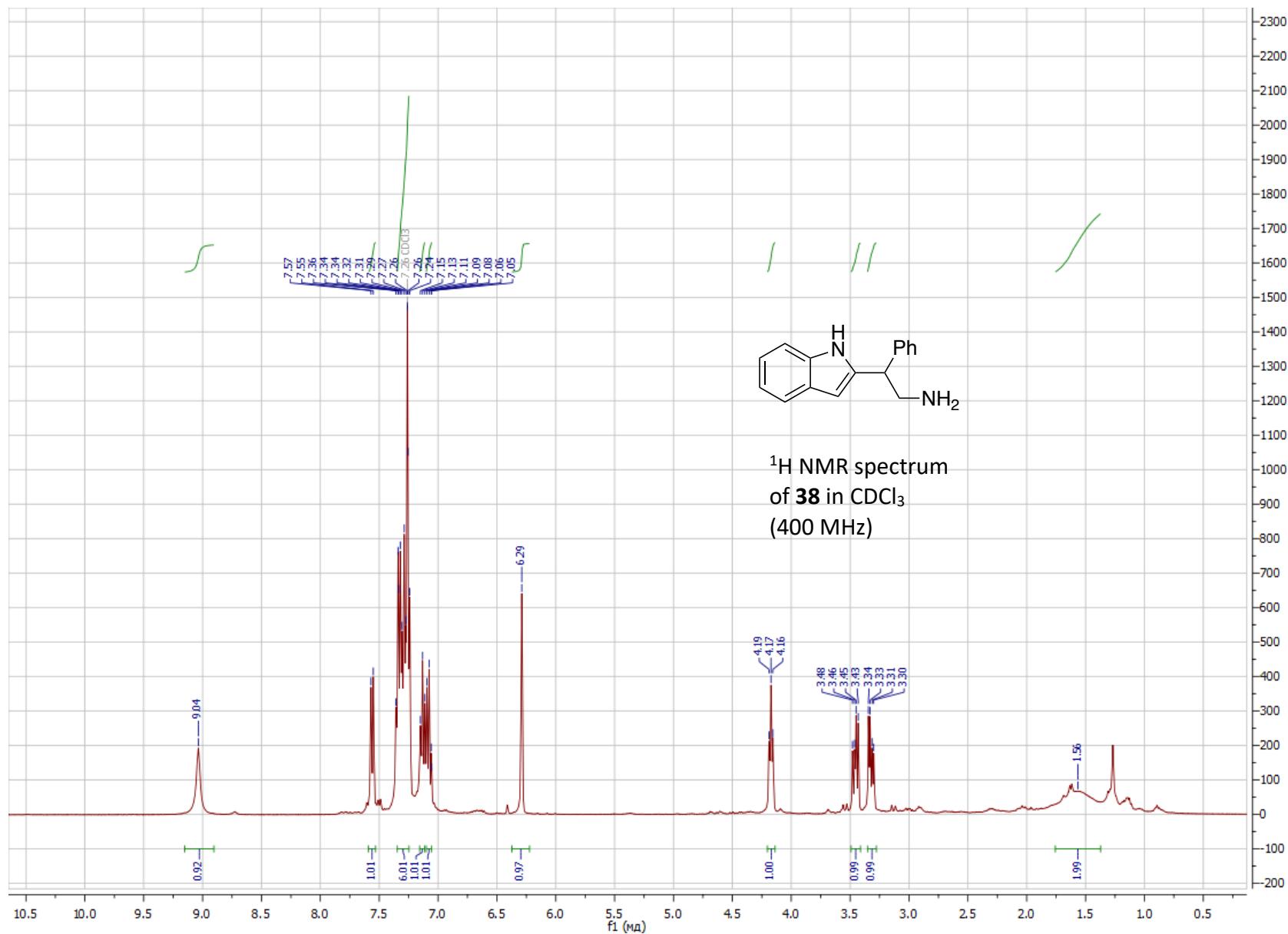


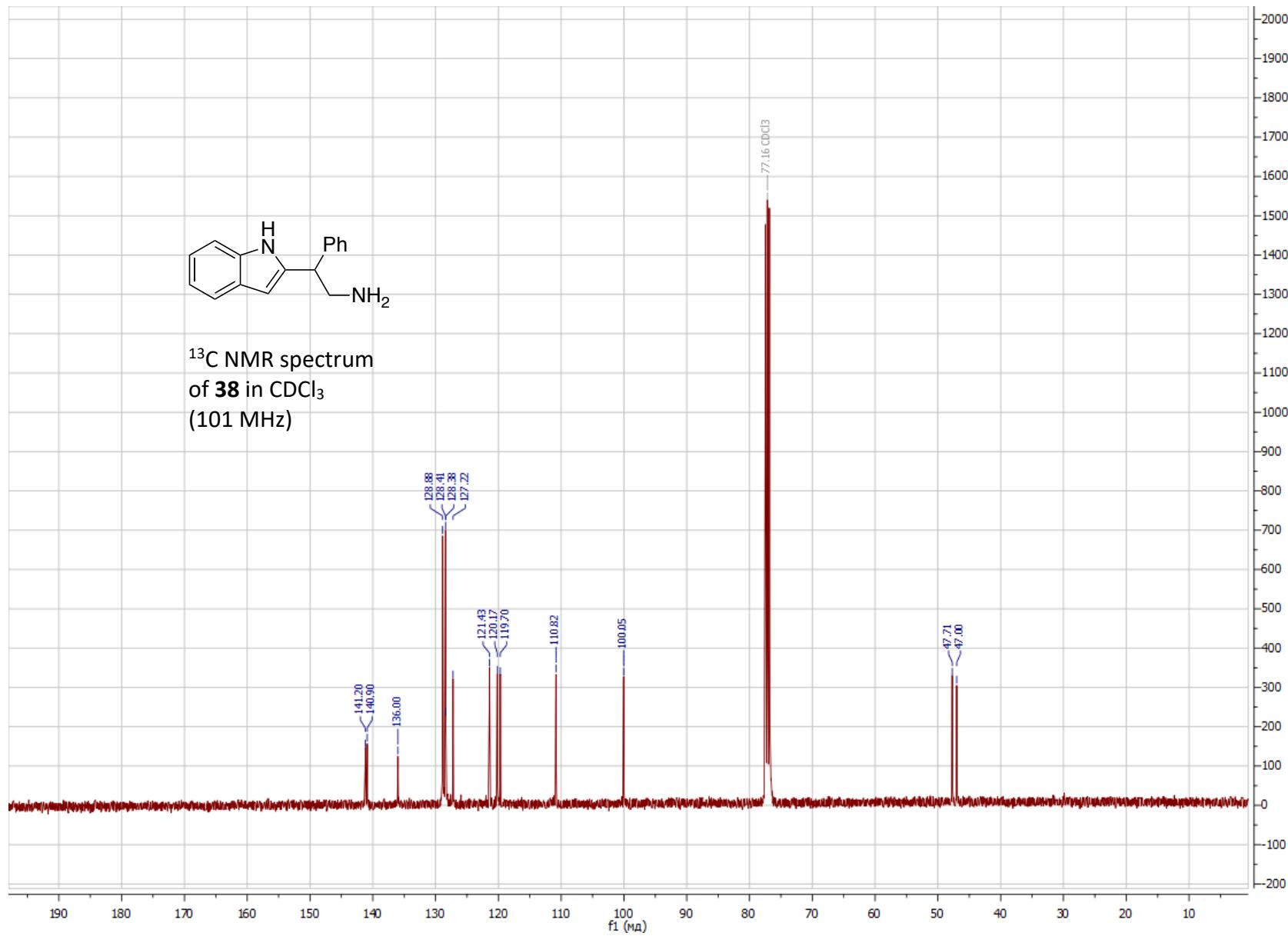


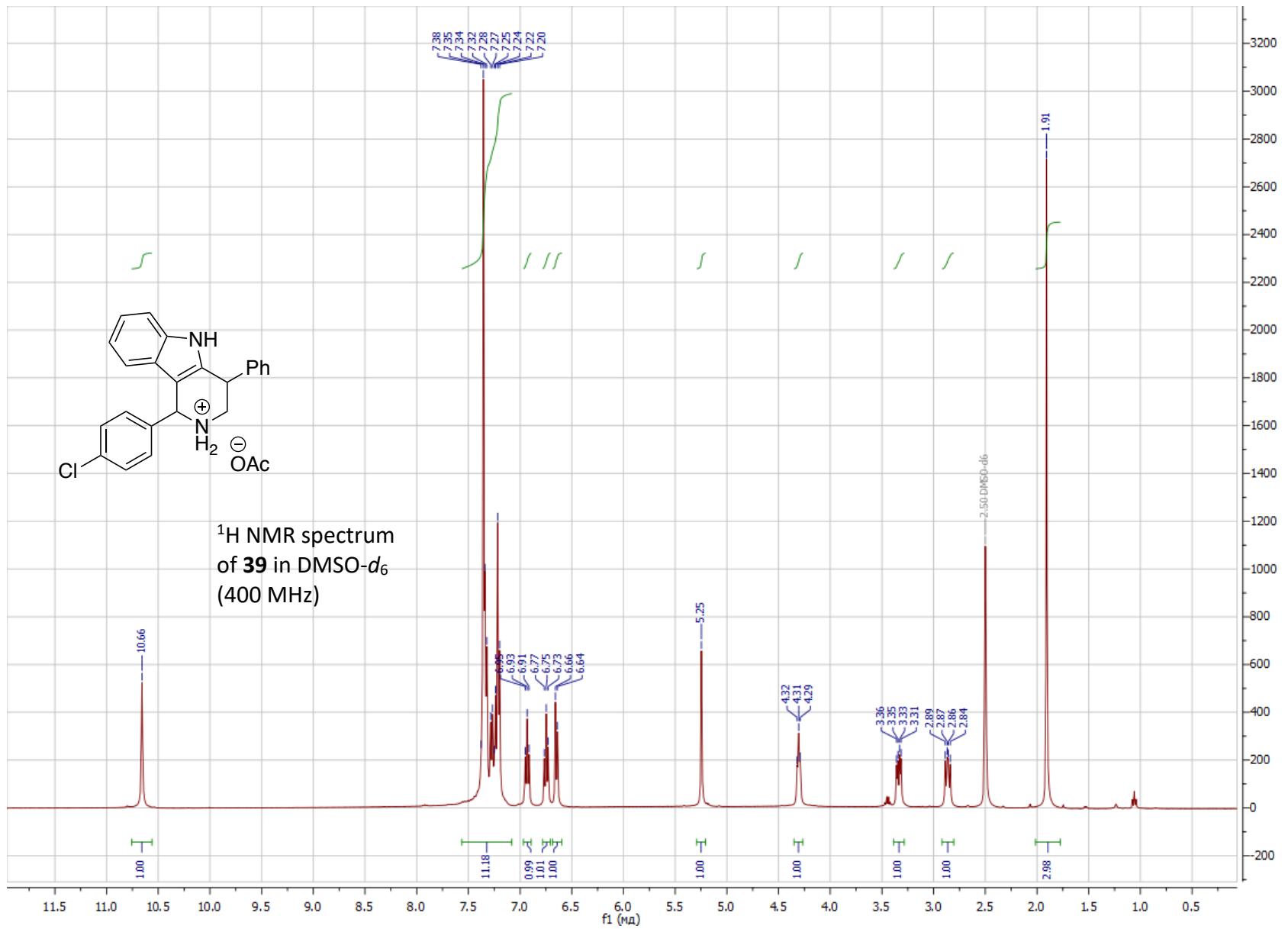


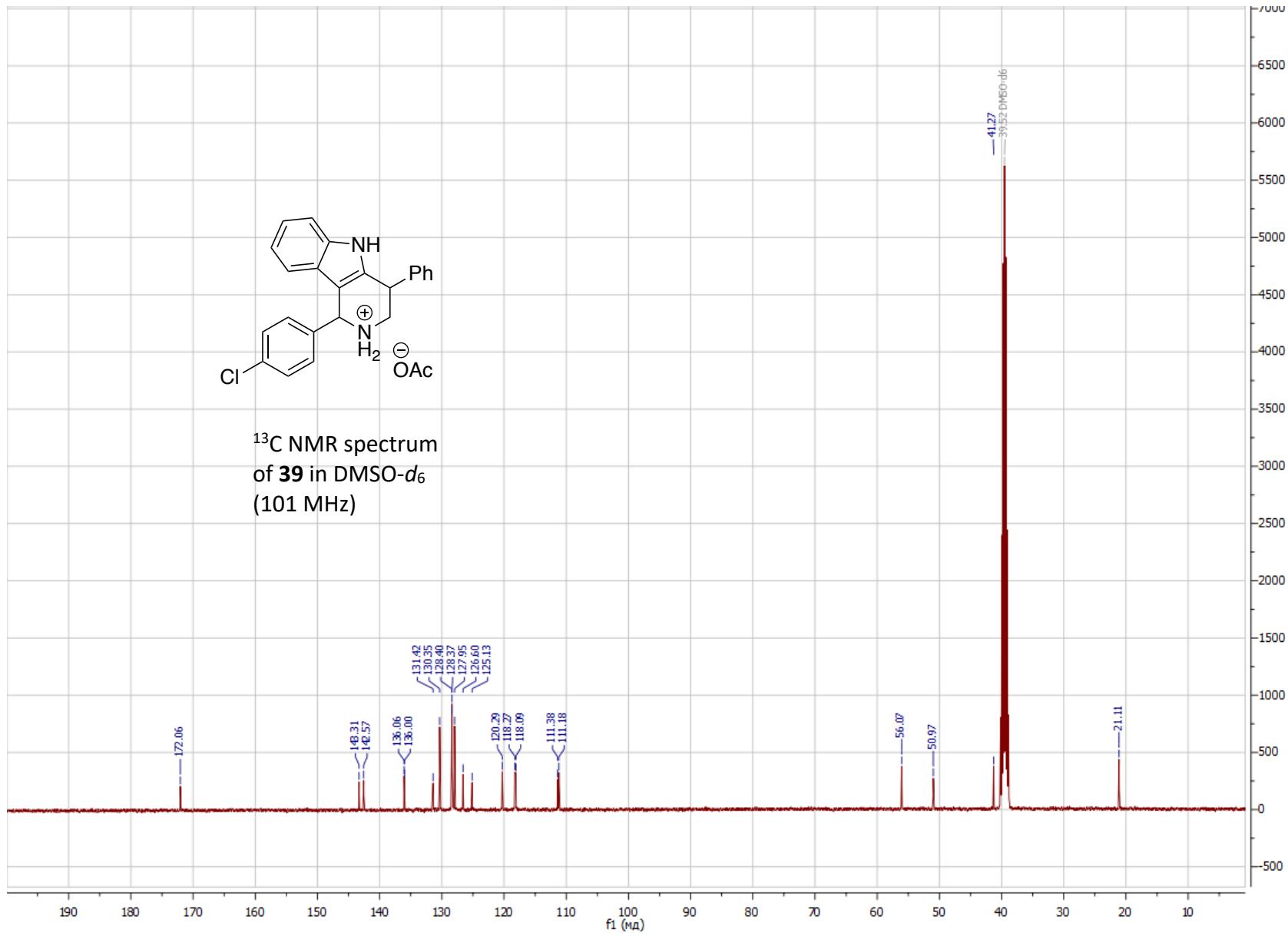


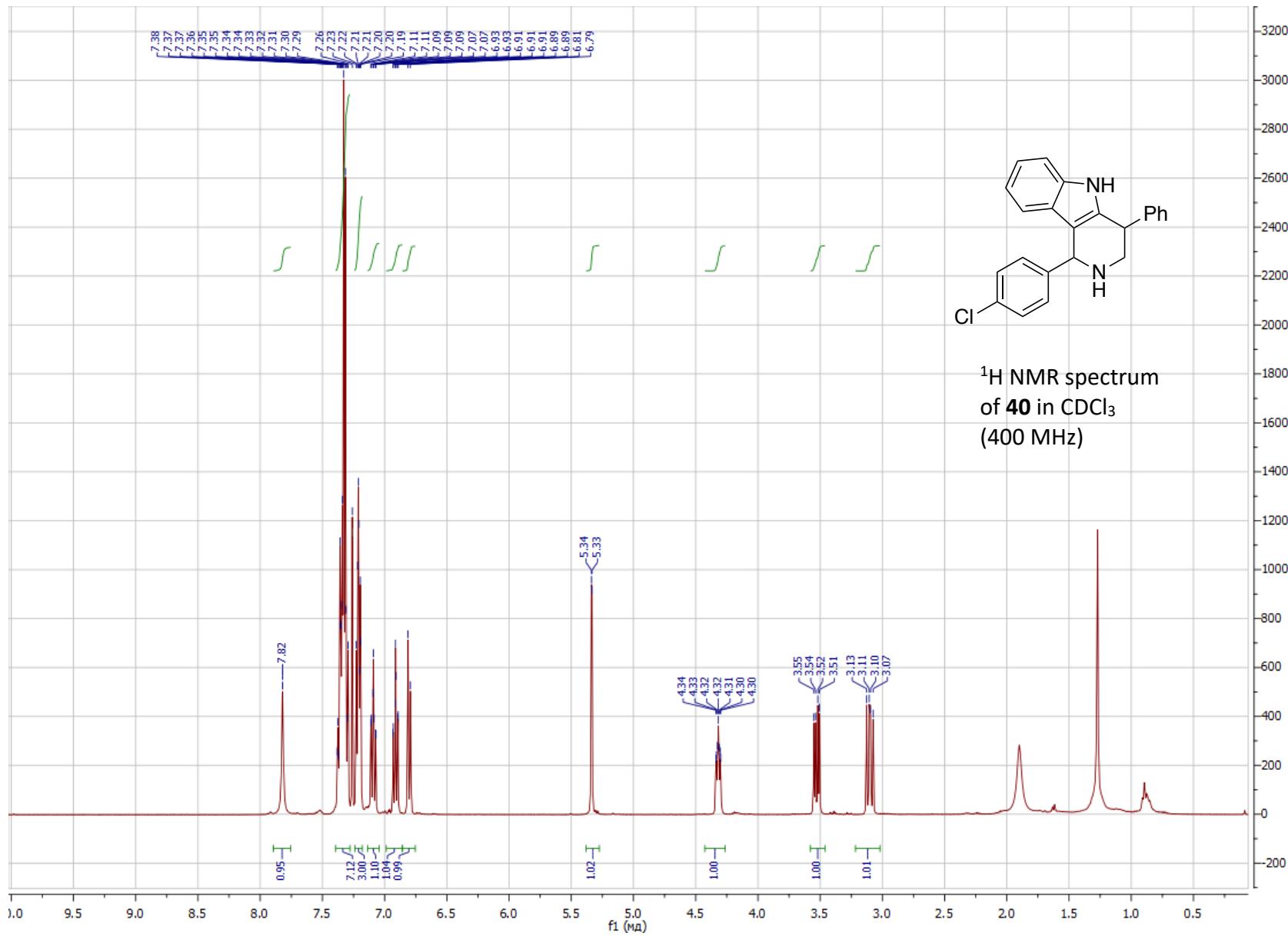


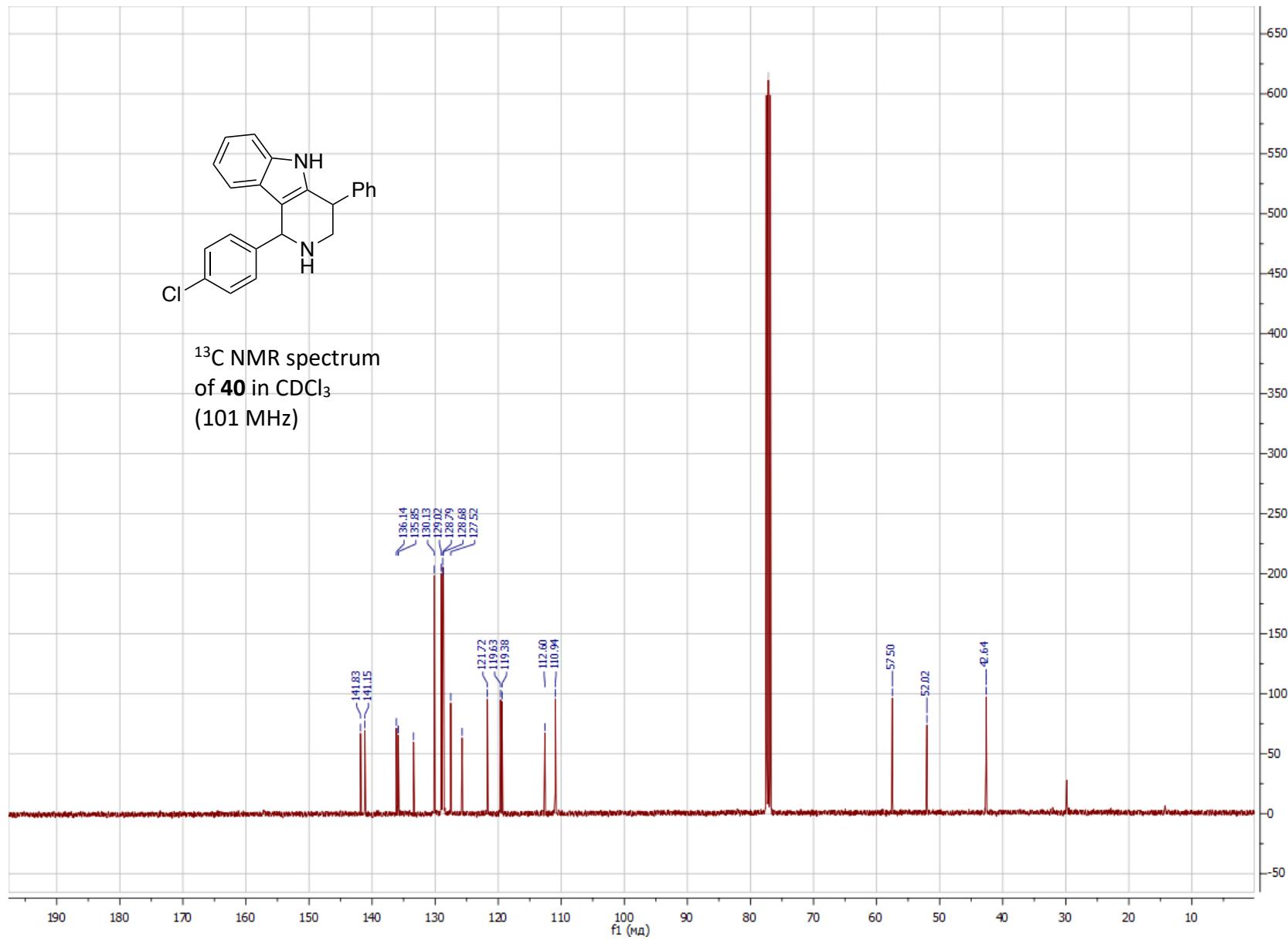




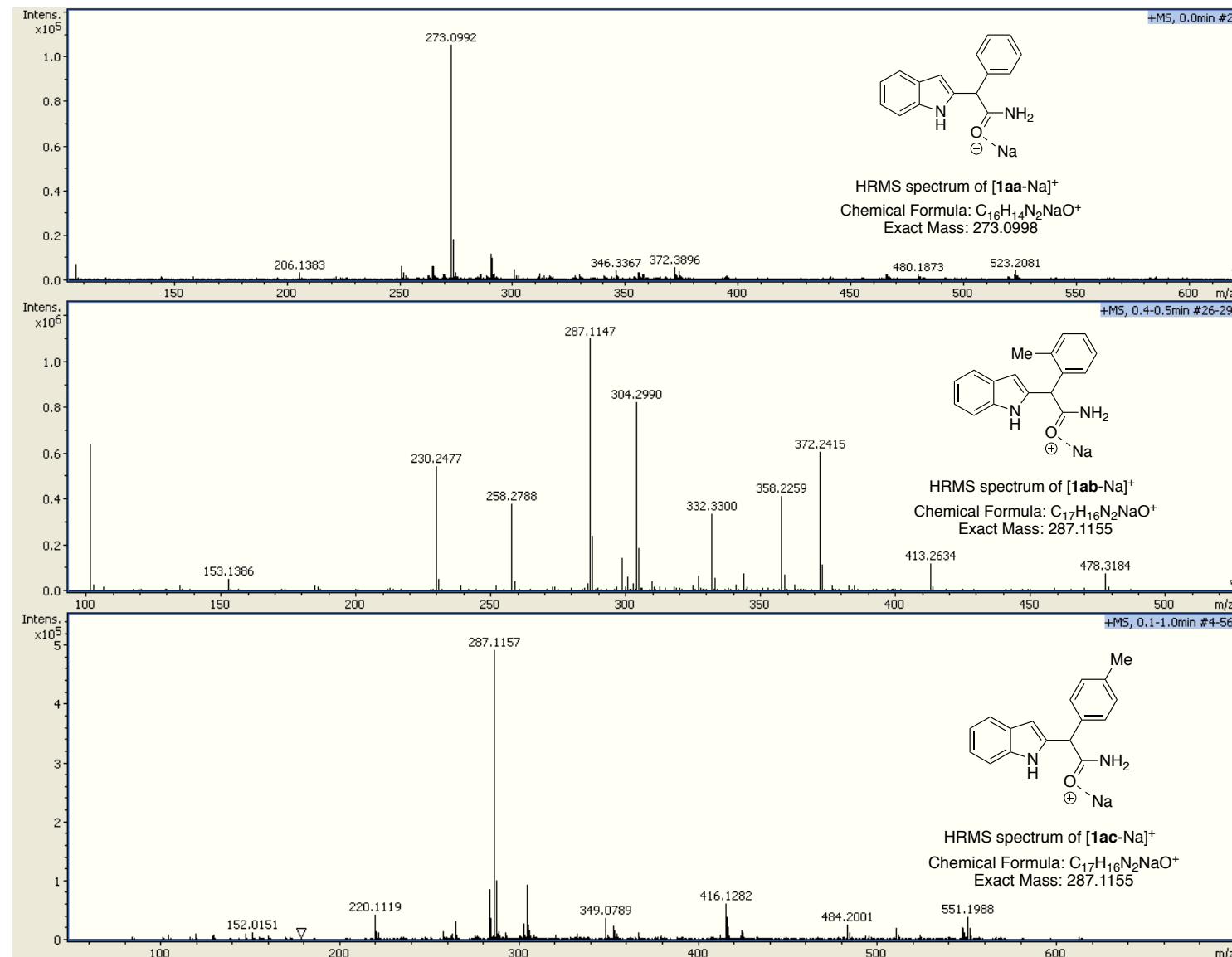


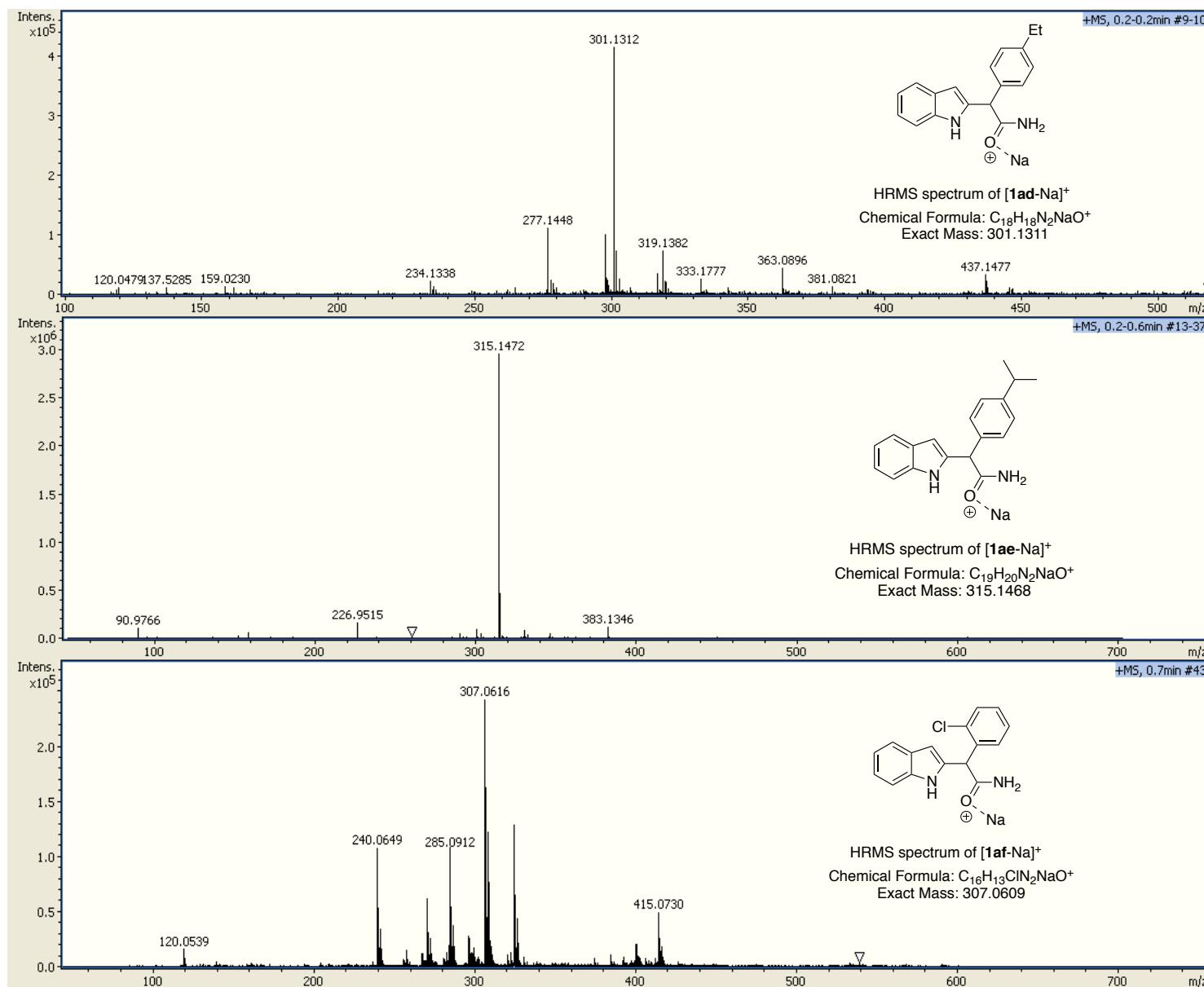


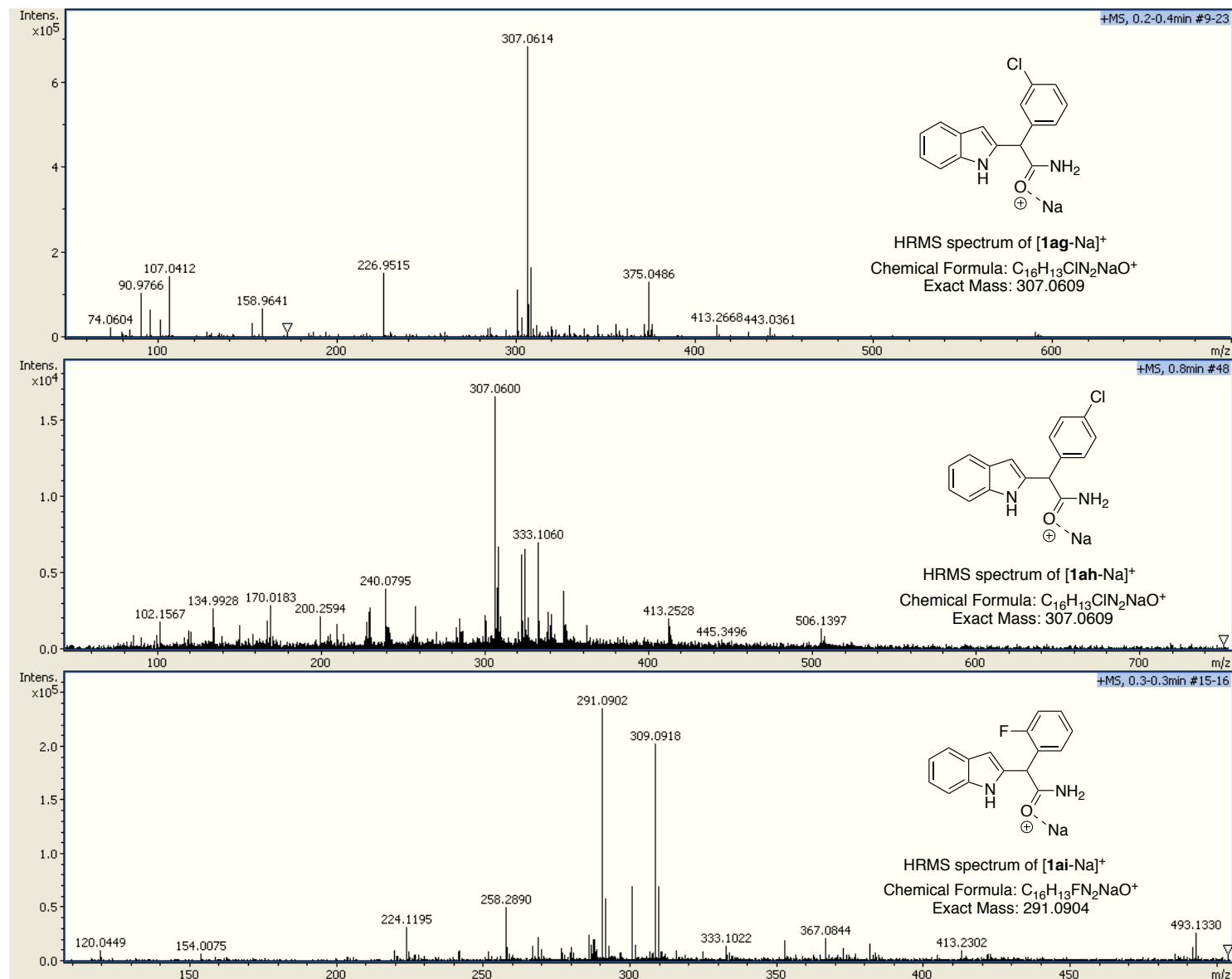


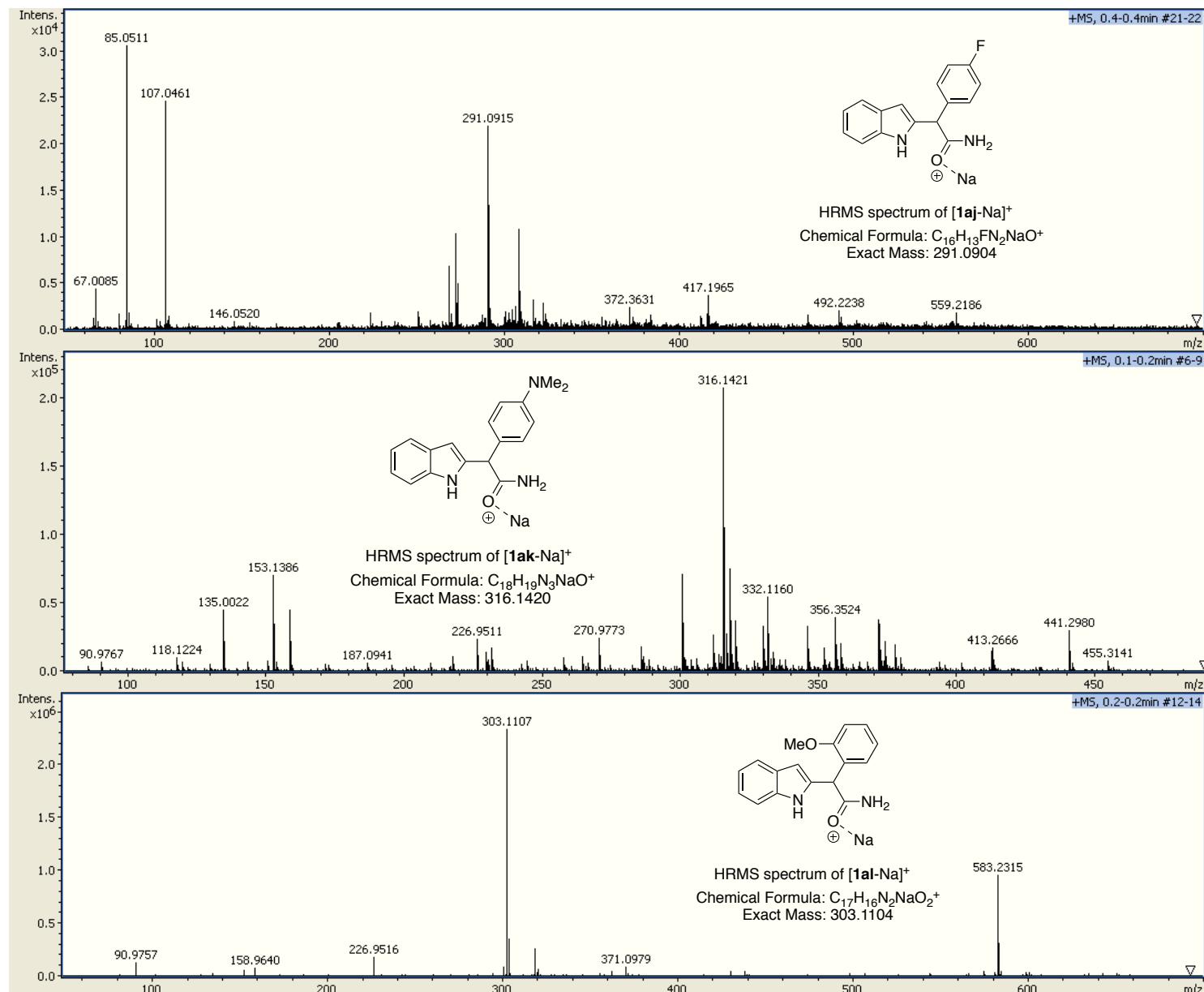


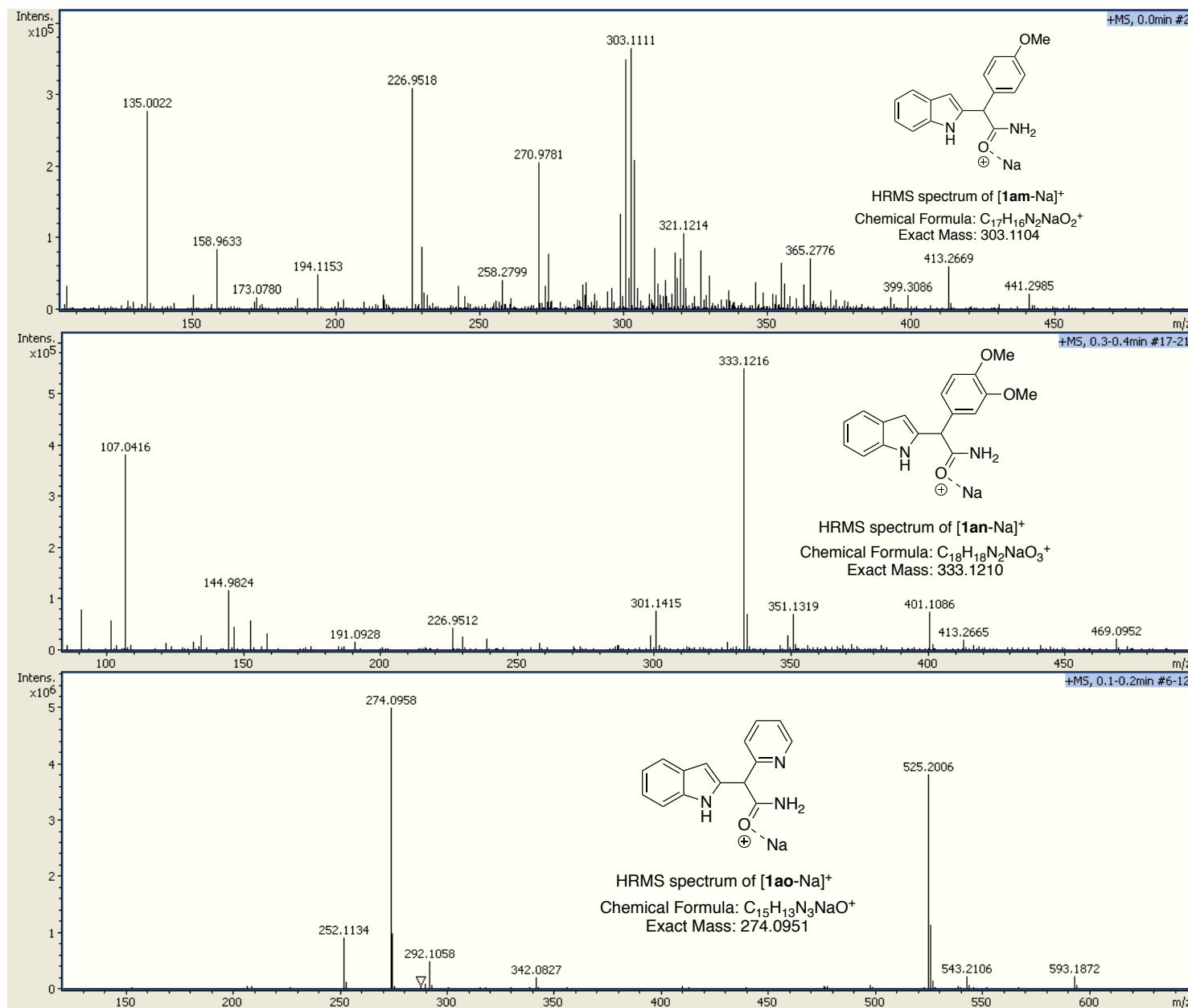
HRMS Spectral Charts of 2-(1*H*-Indol-2-yl)acetamides 1

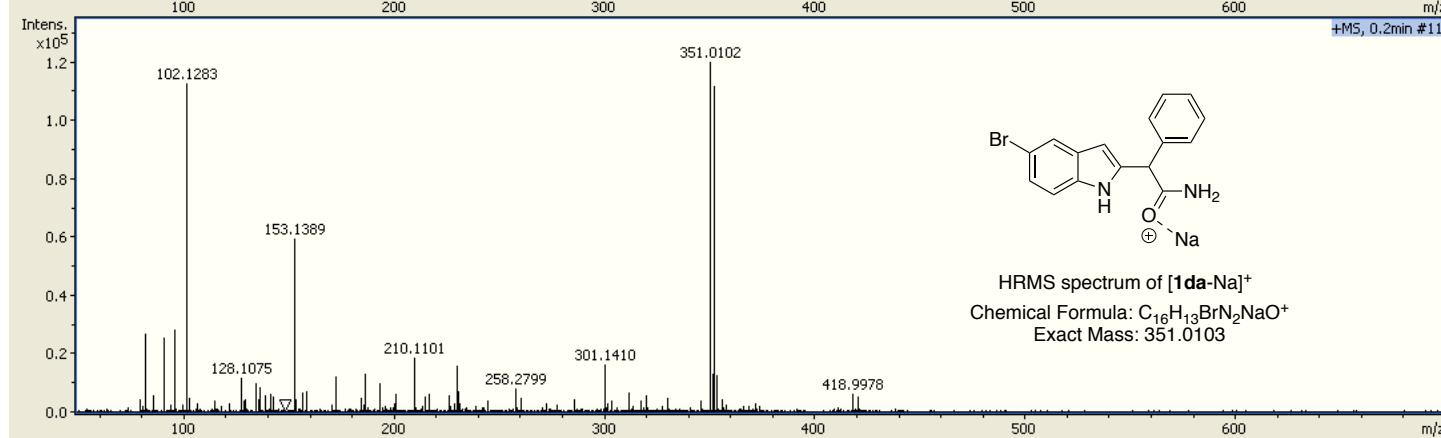
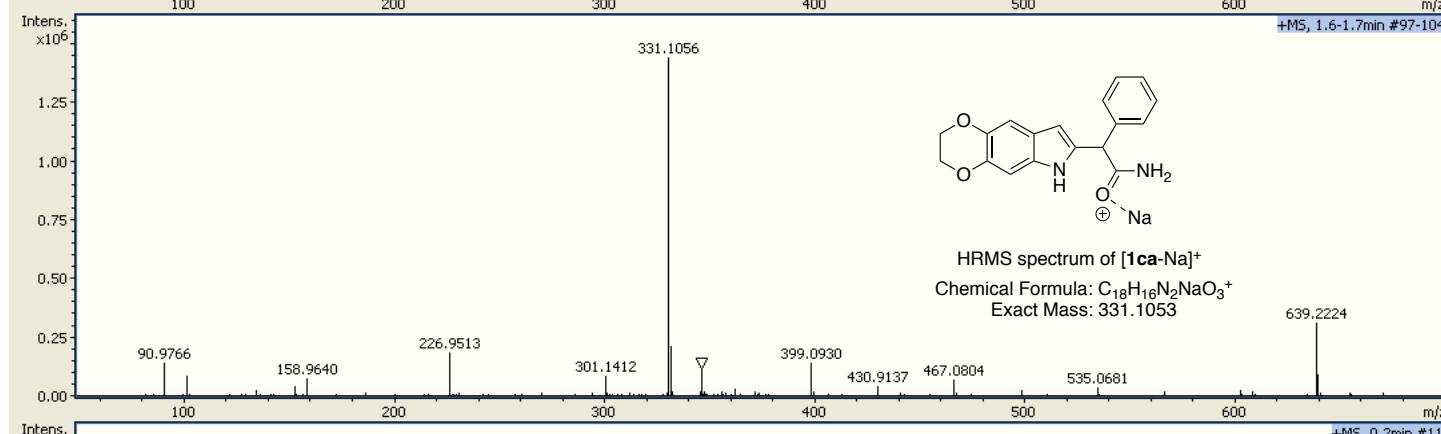
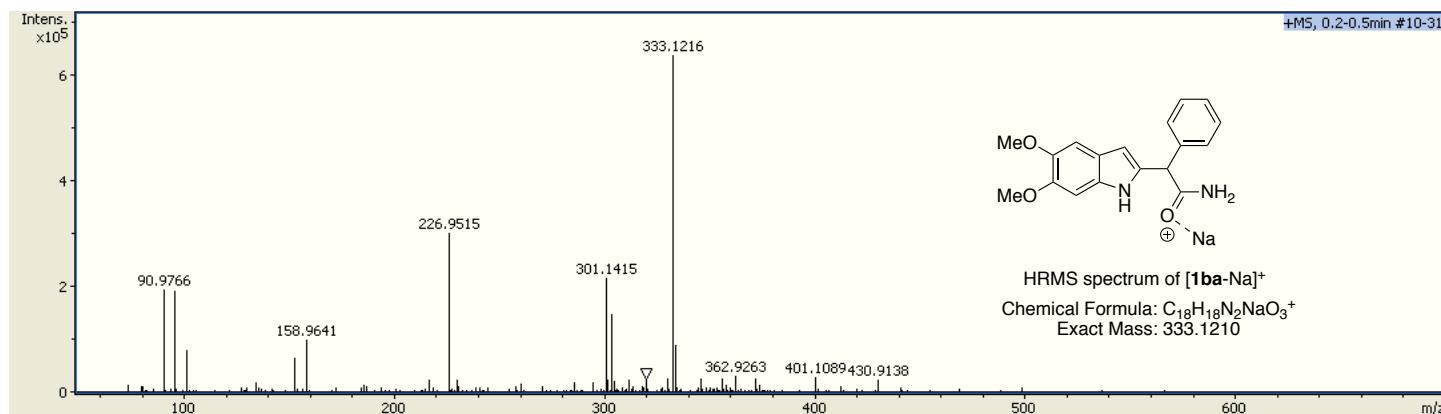




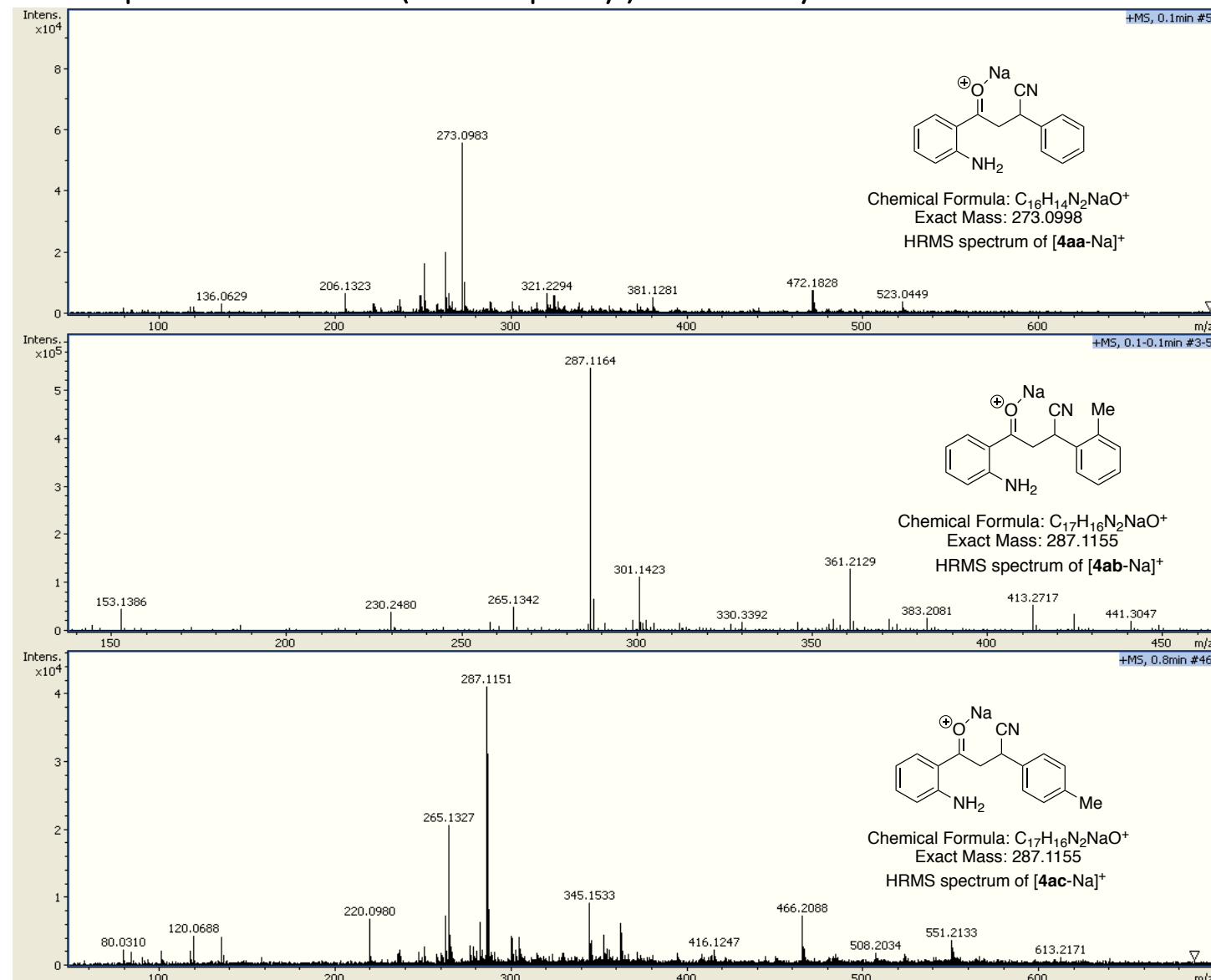


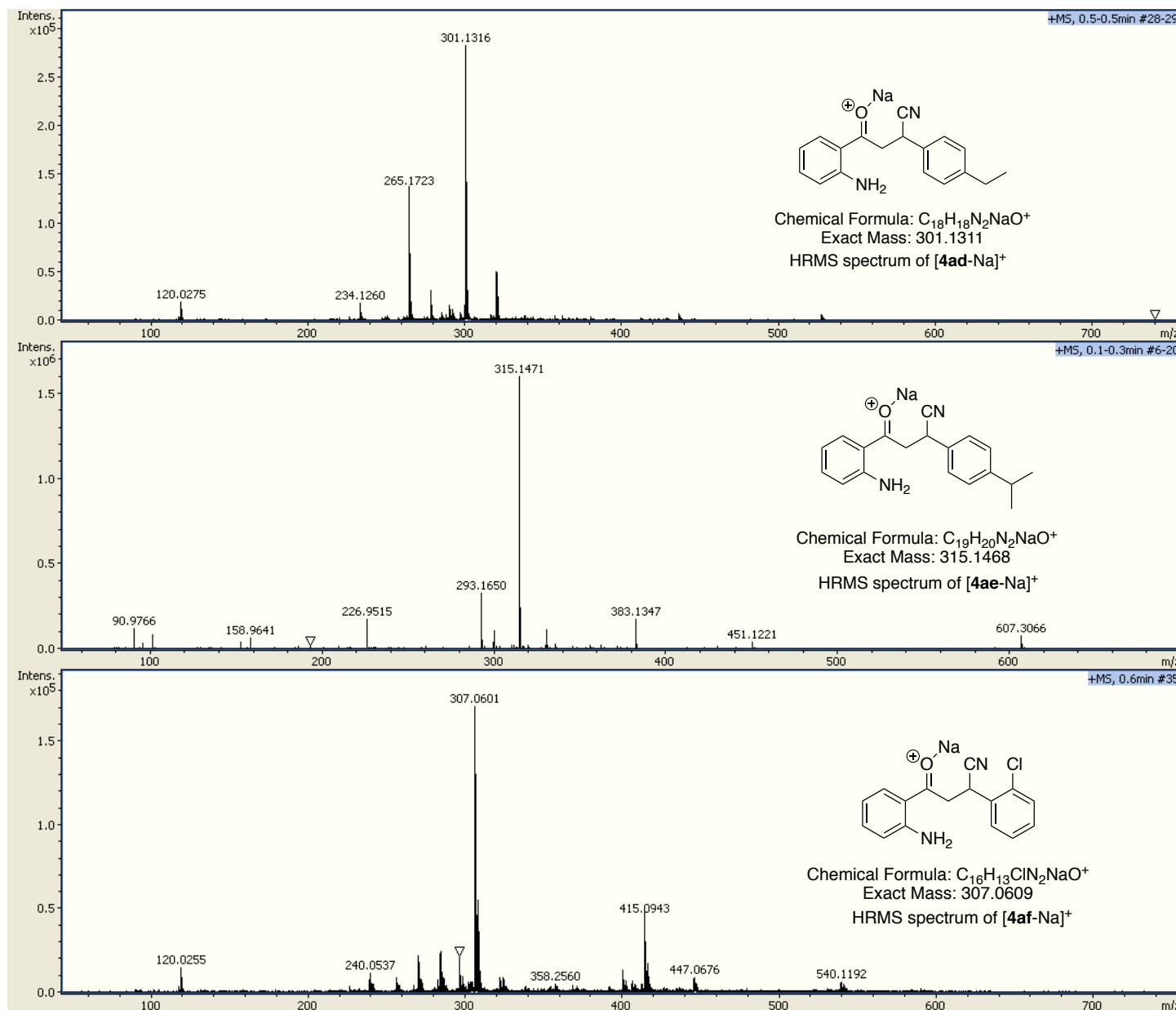


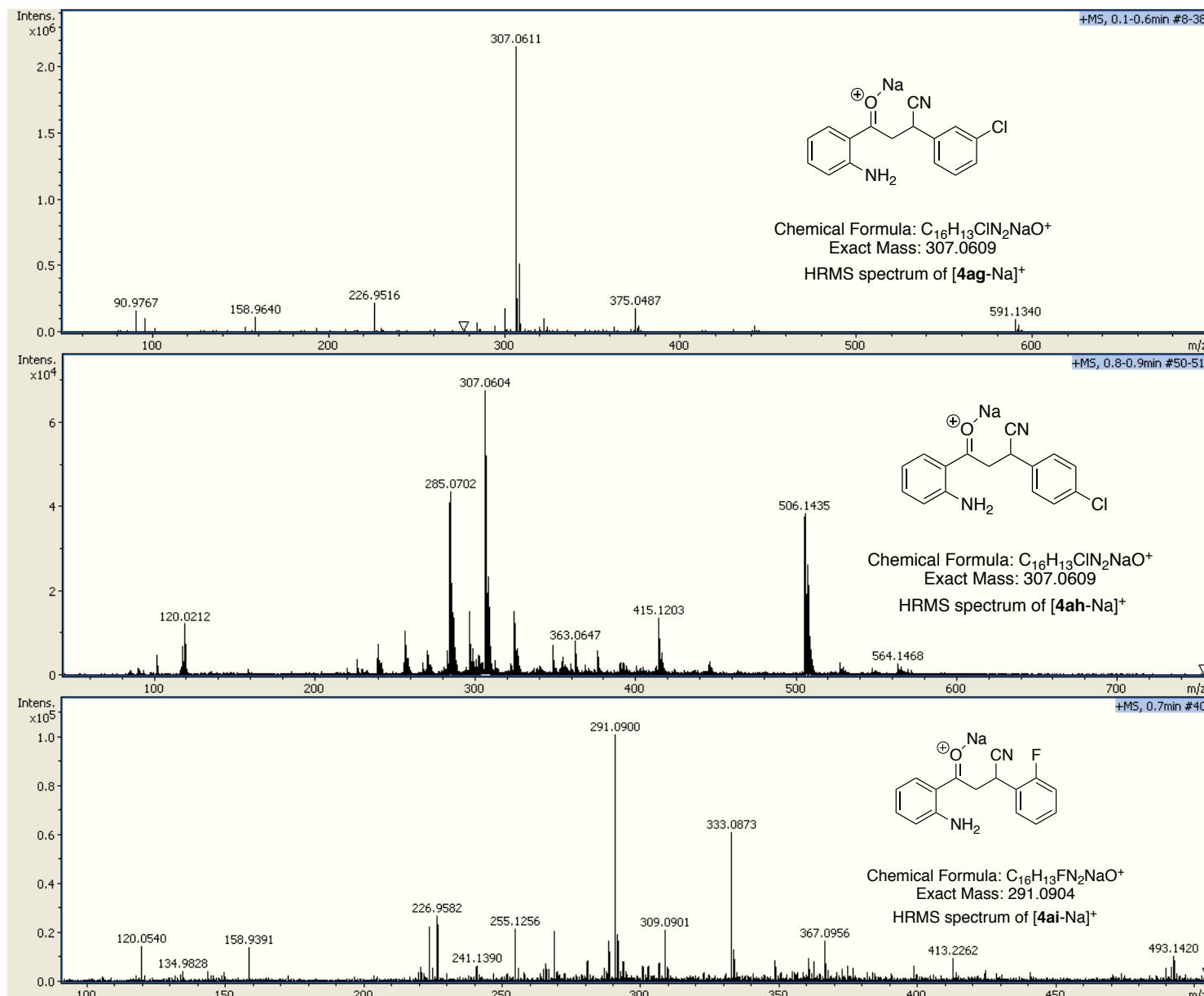


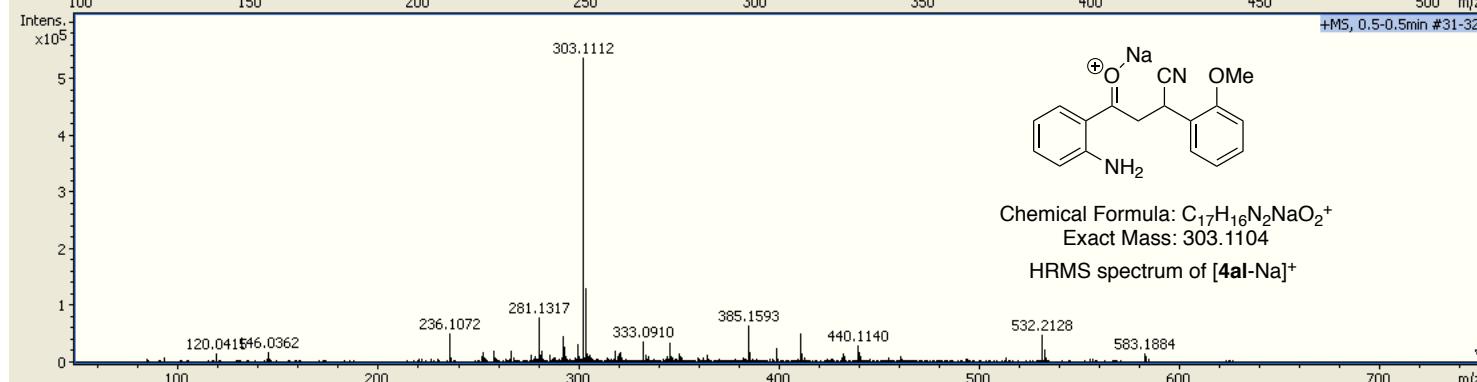
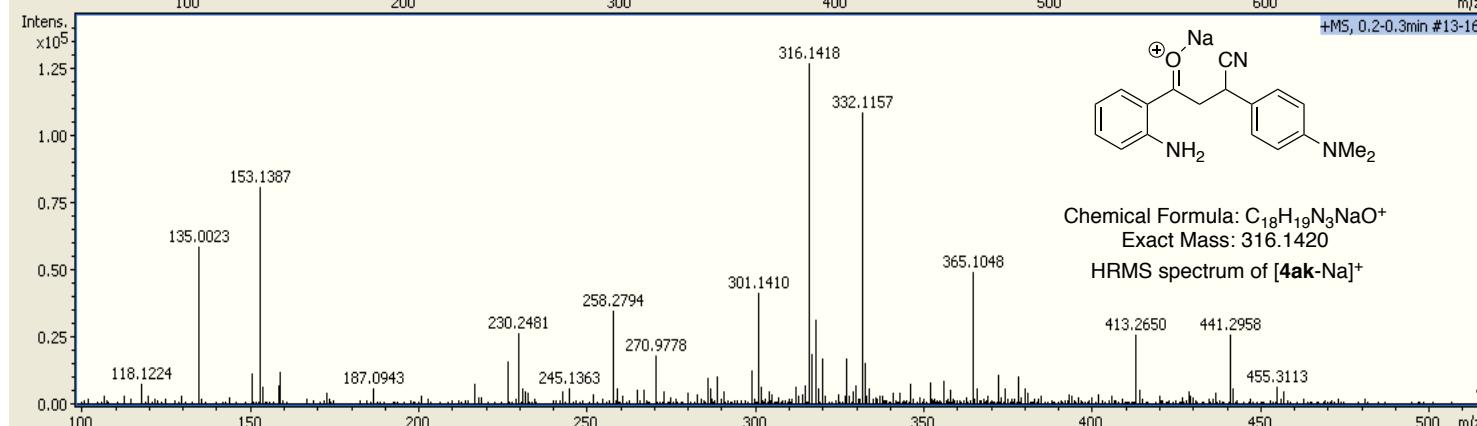
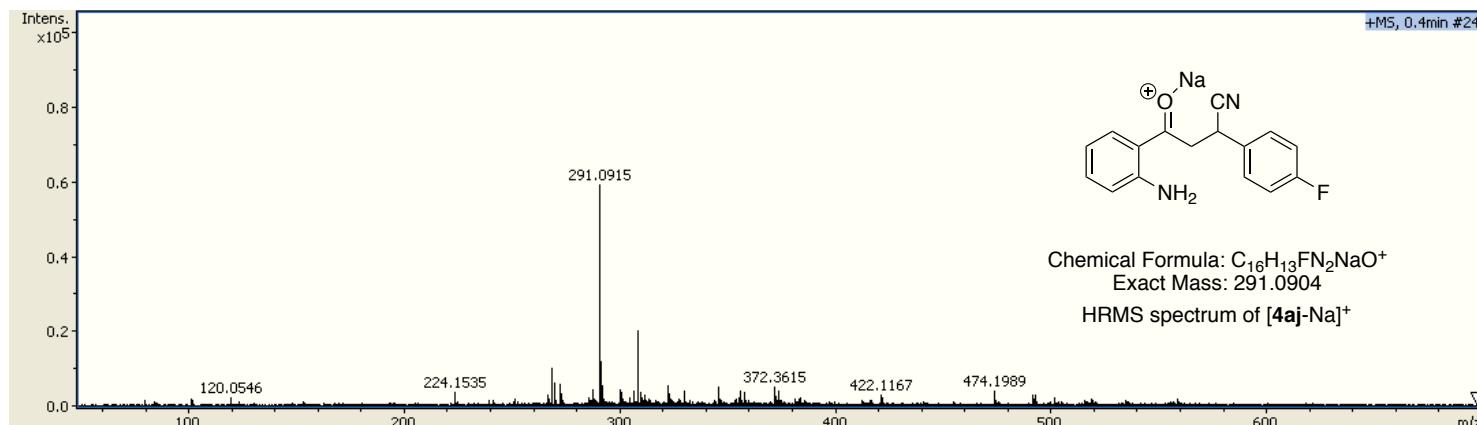


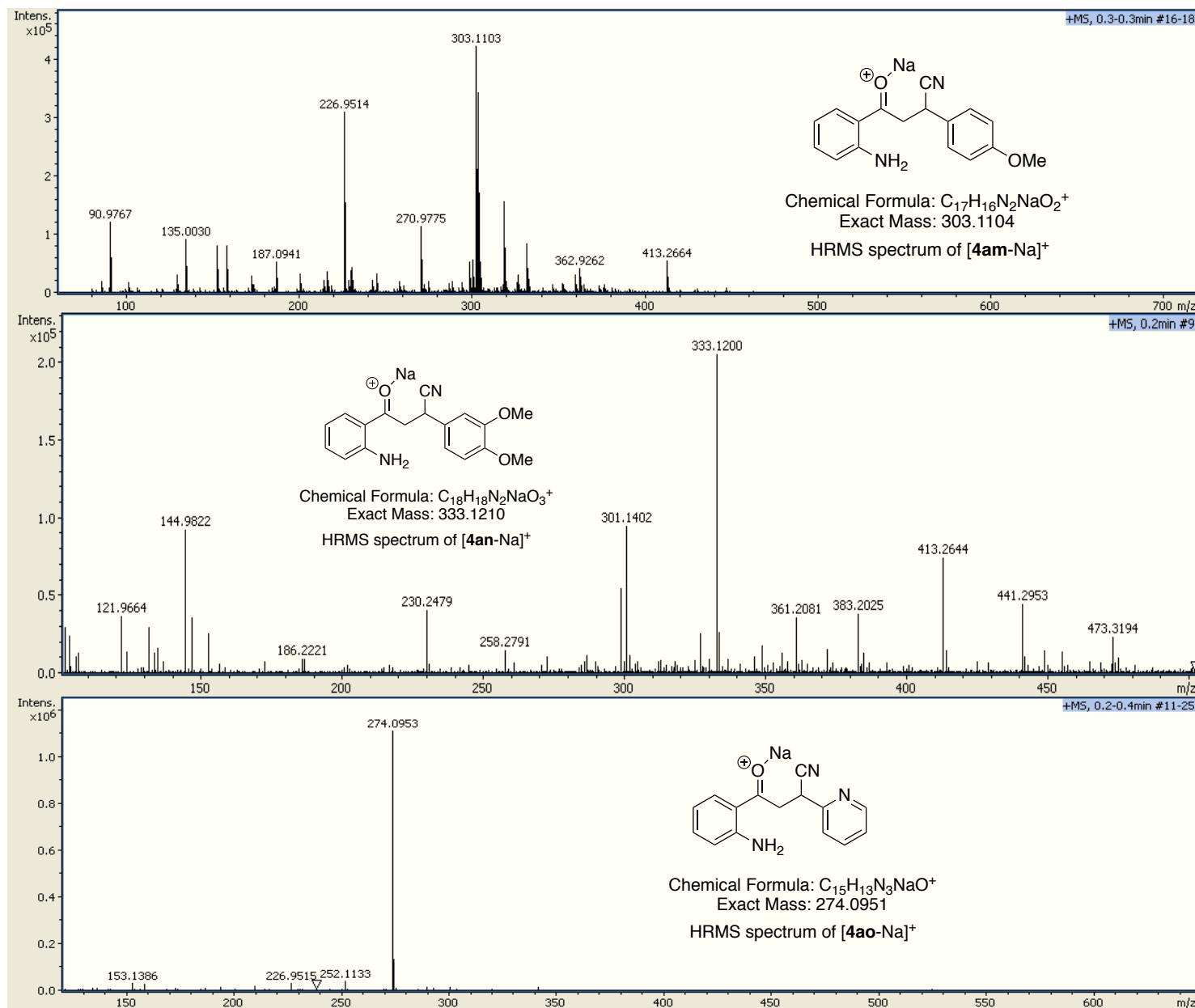
HRMS Spectral Charts of 4-(2-aminophenyl)-4-oxo-2-arylbutanenitriles 4

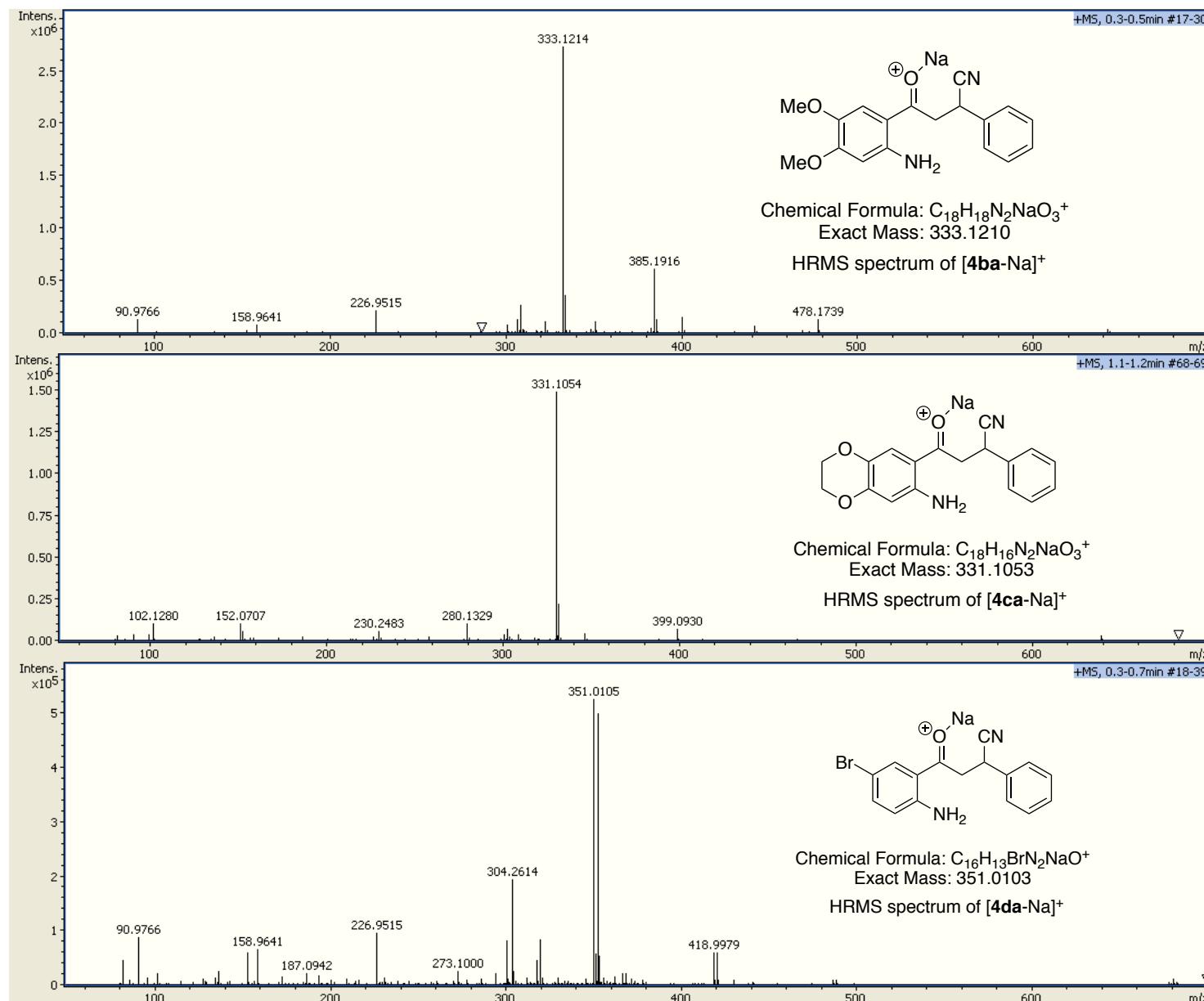




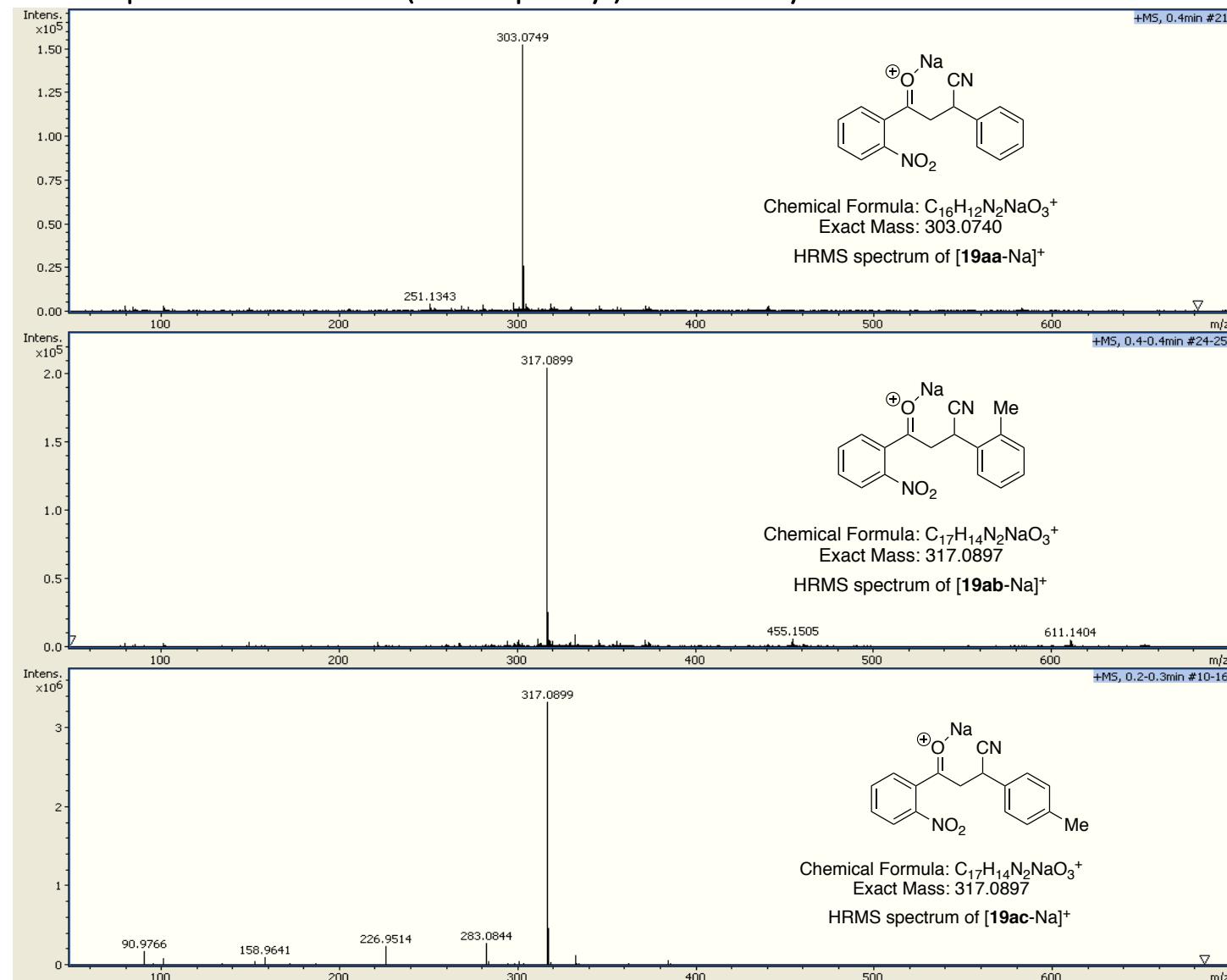


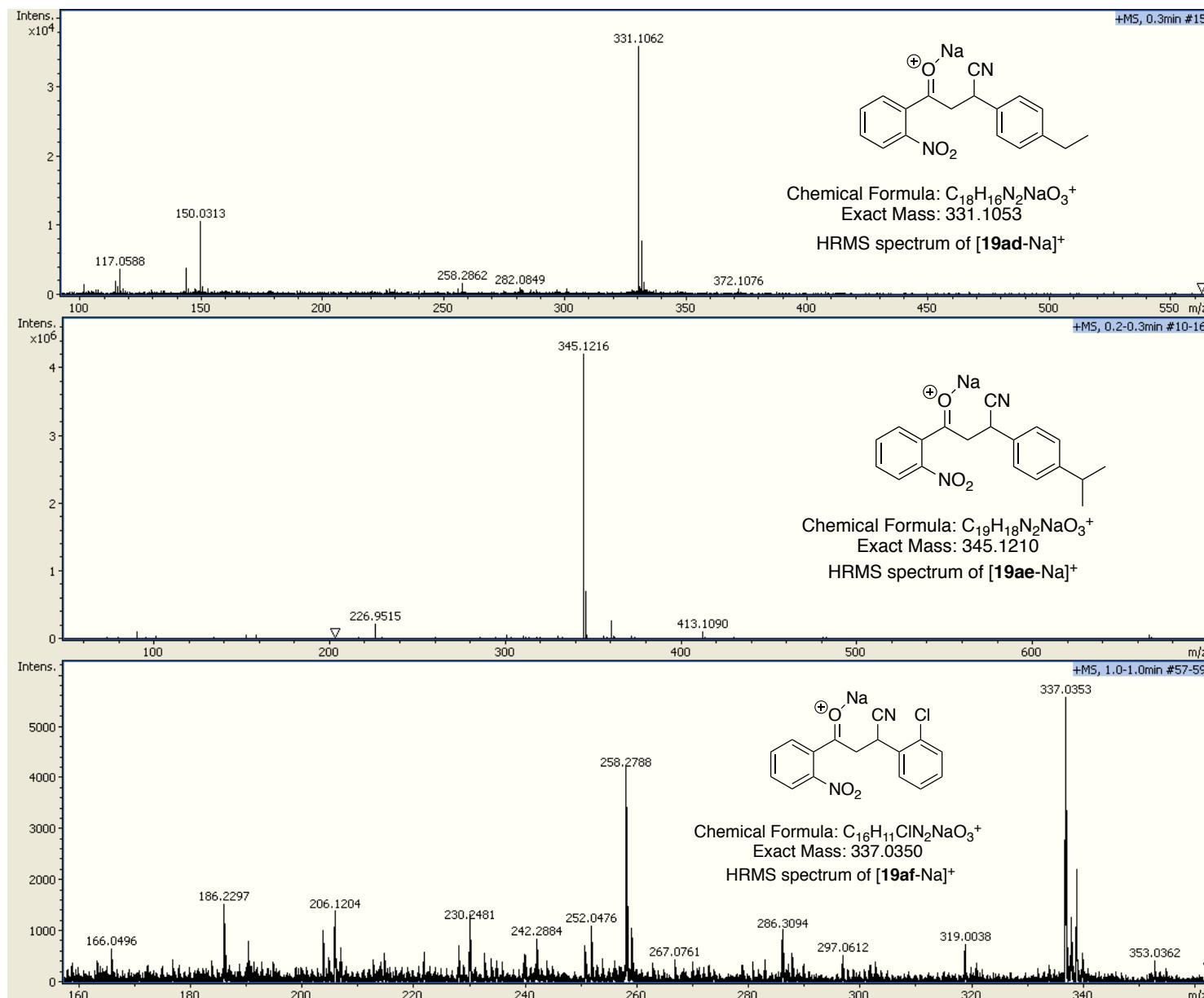


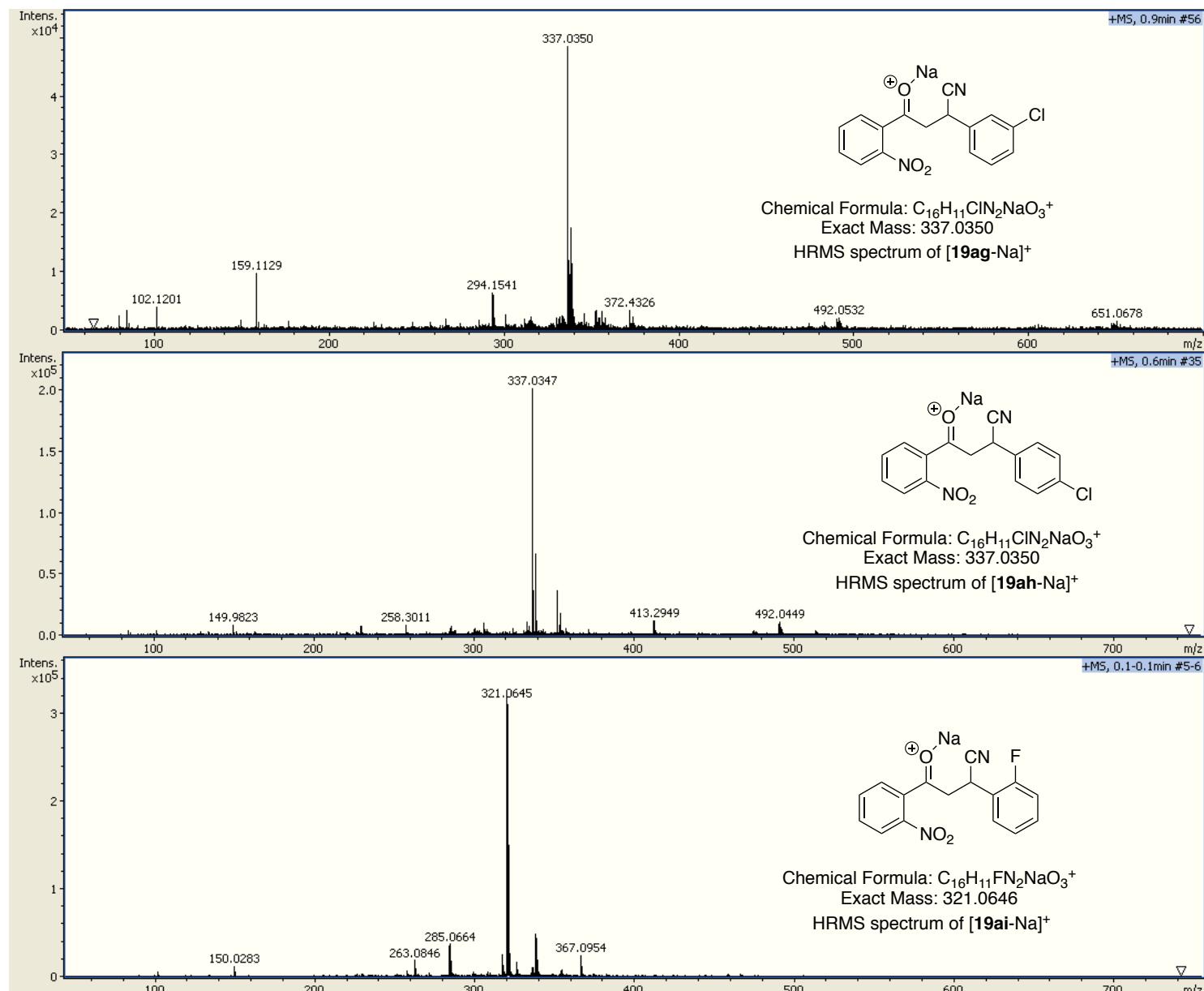


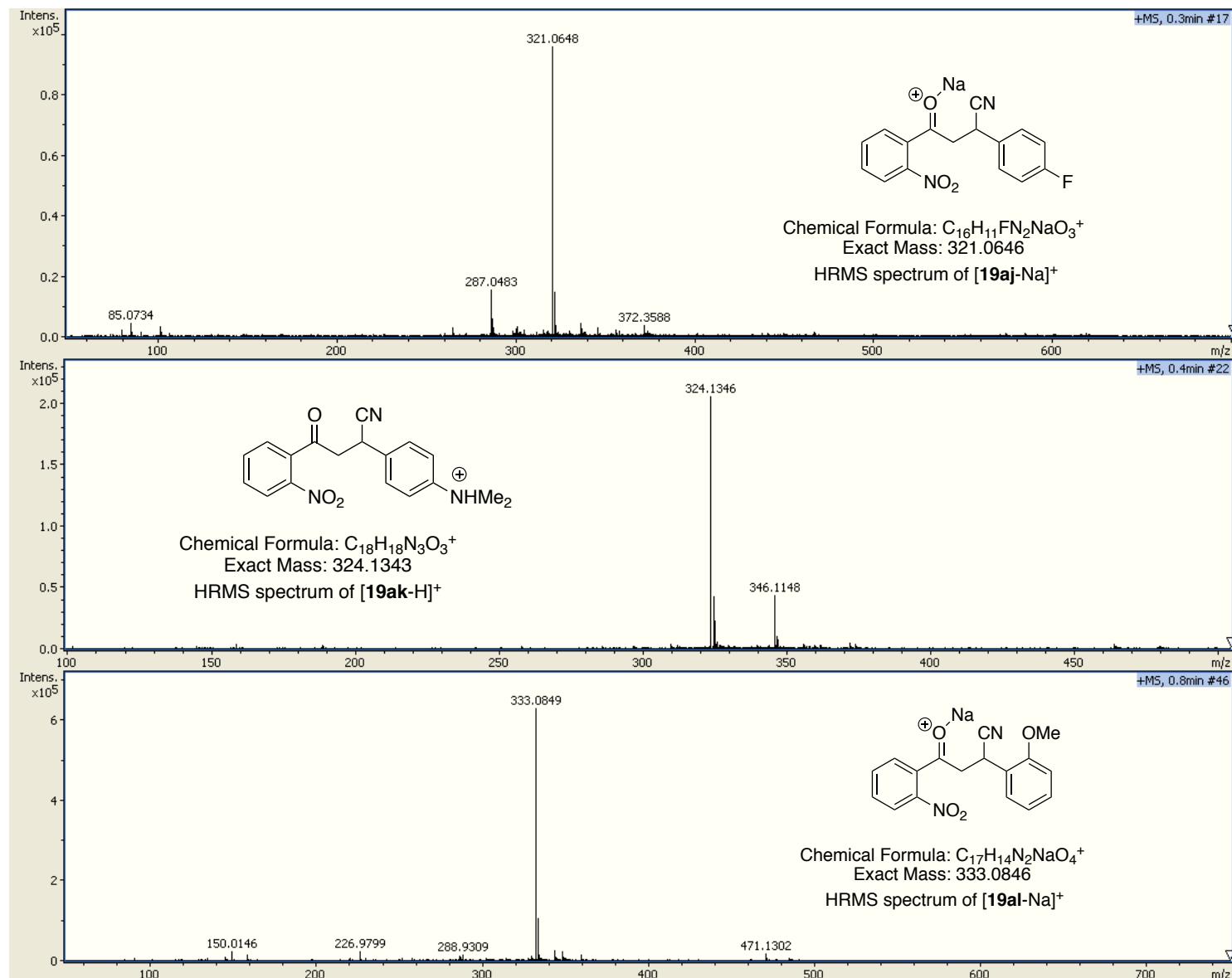


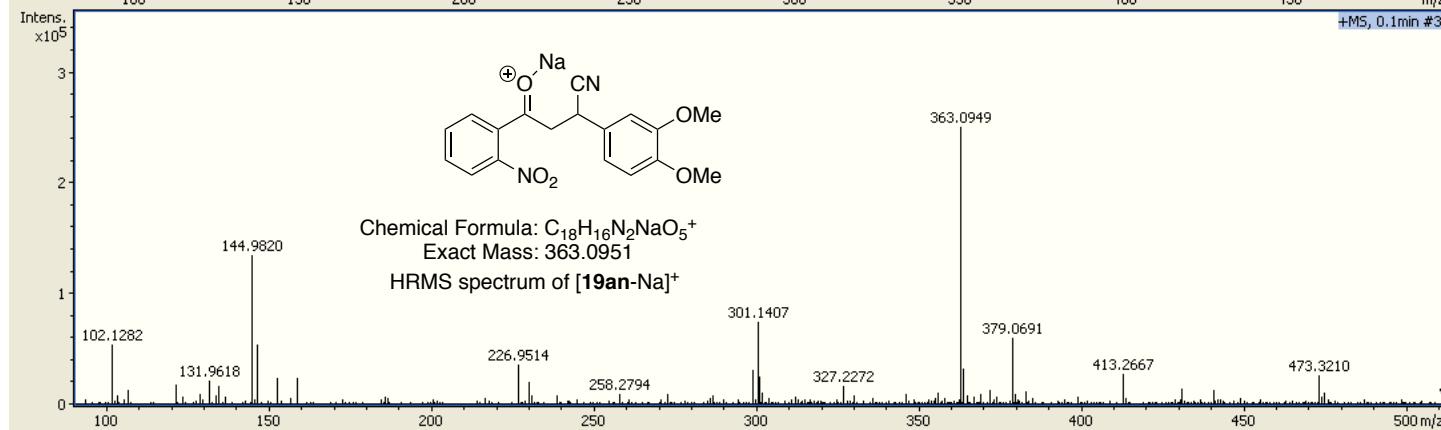
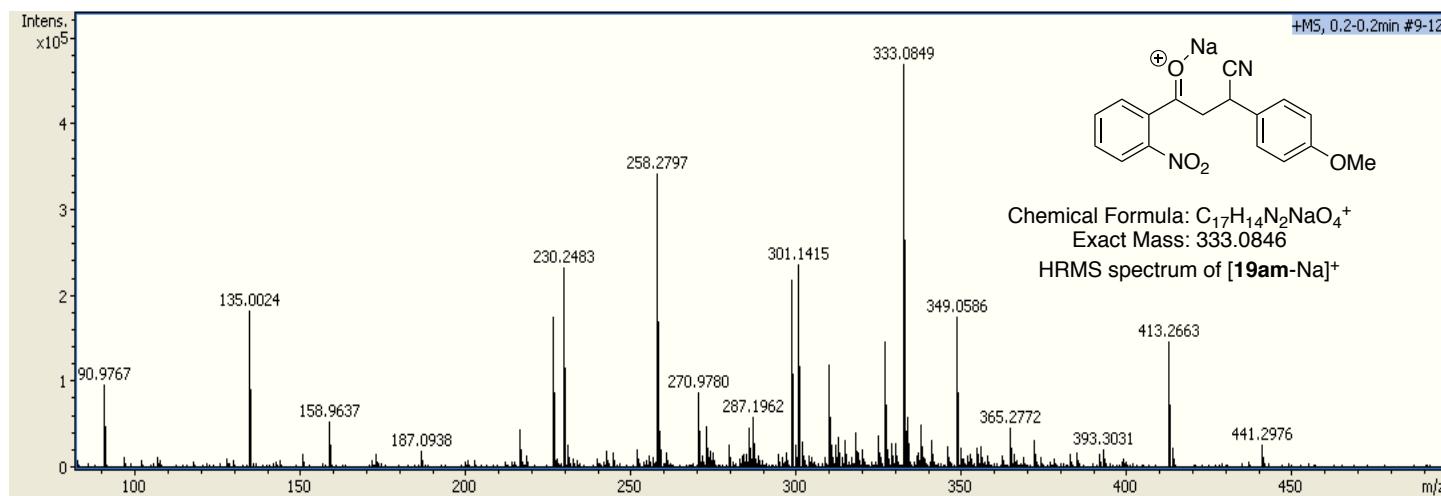
HRMS Spectral Charts of 4-(2-nitrophenyl)-4-oxo-2-arylbutanenitriles 19

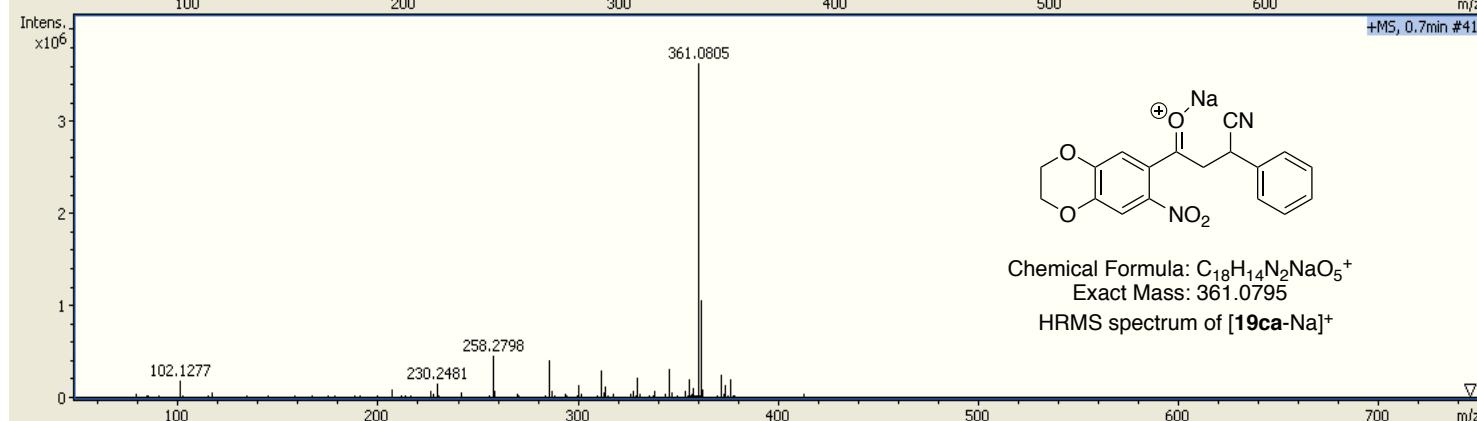
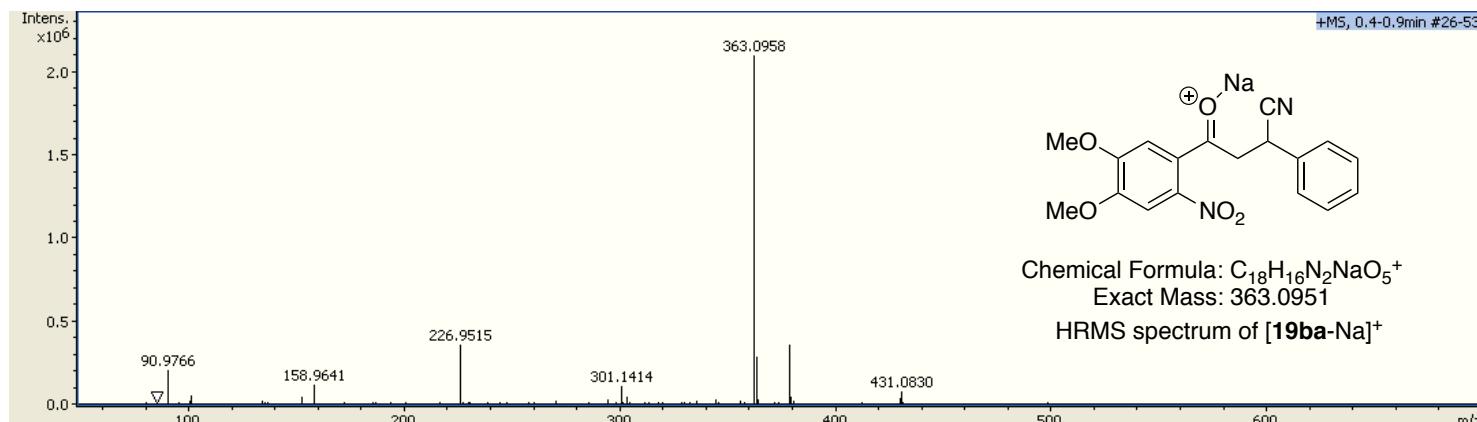




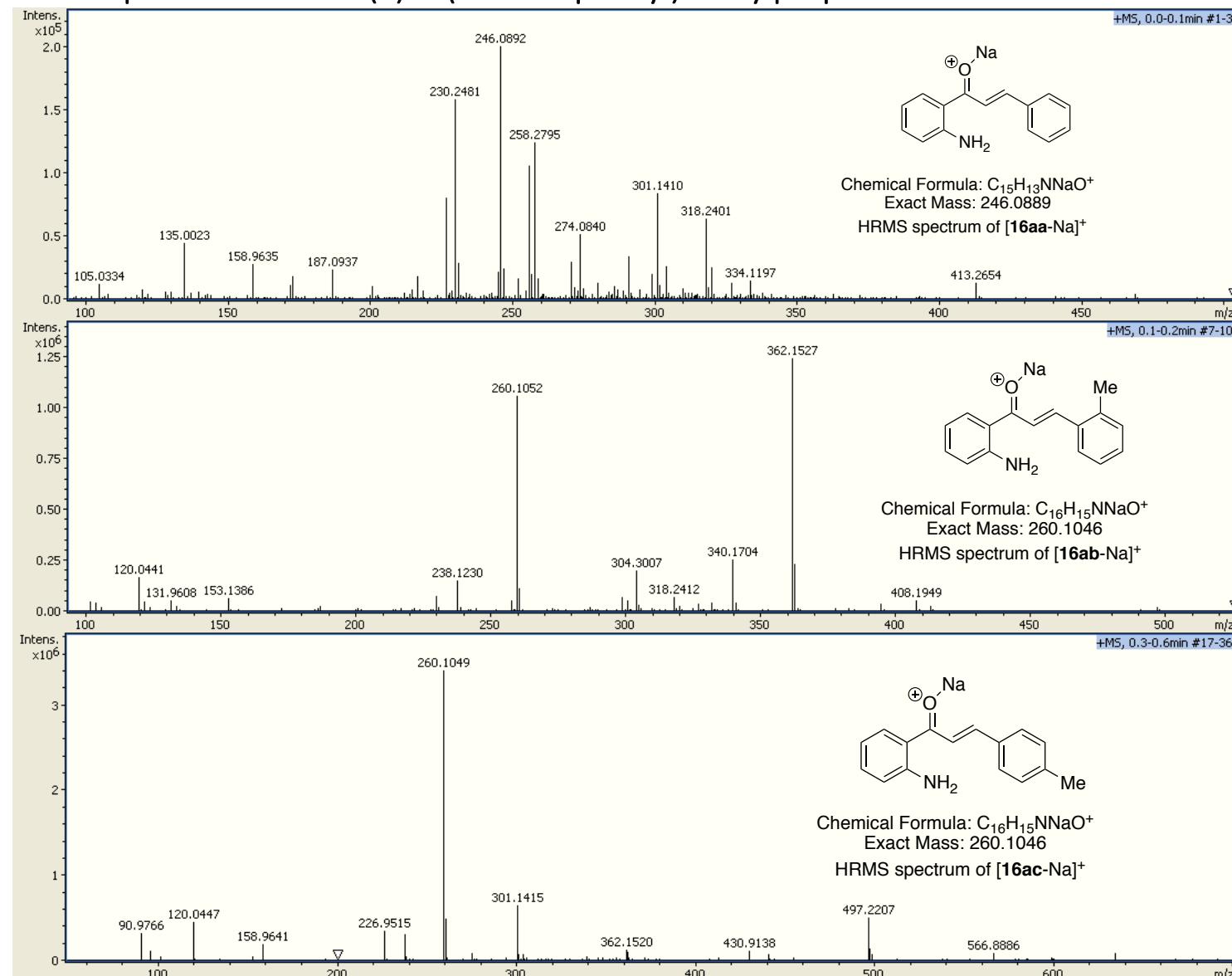


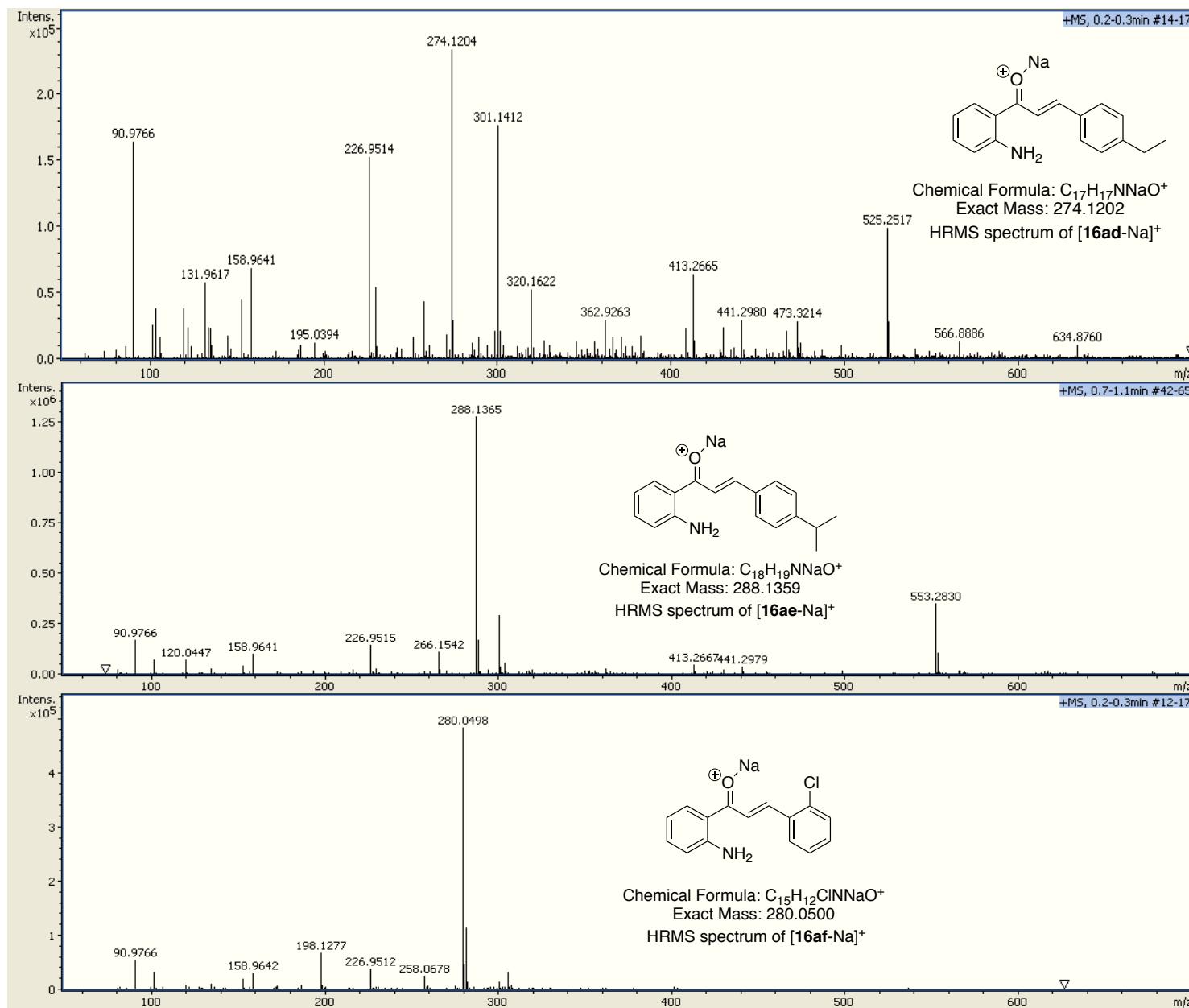


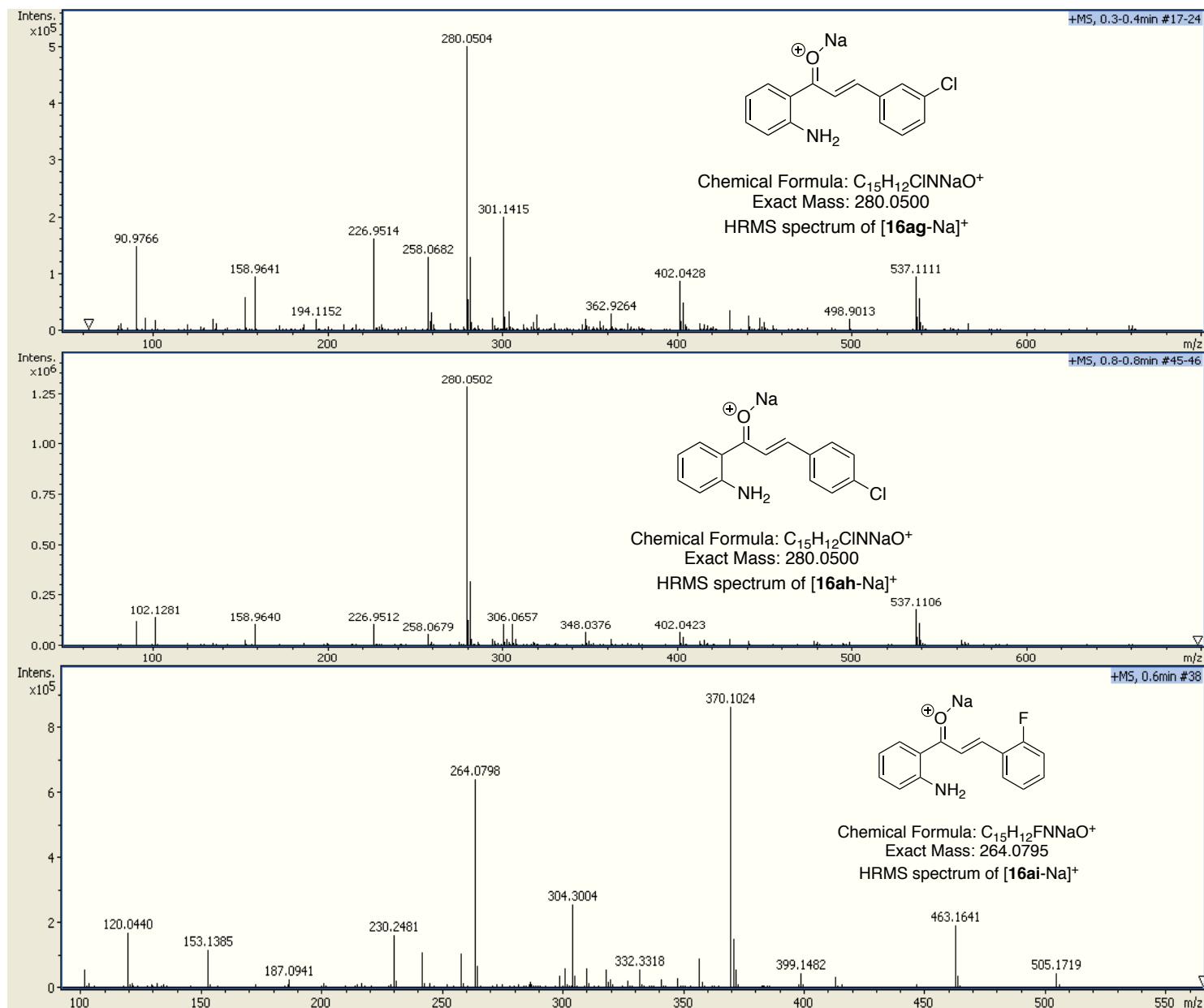


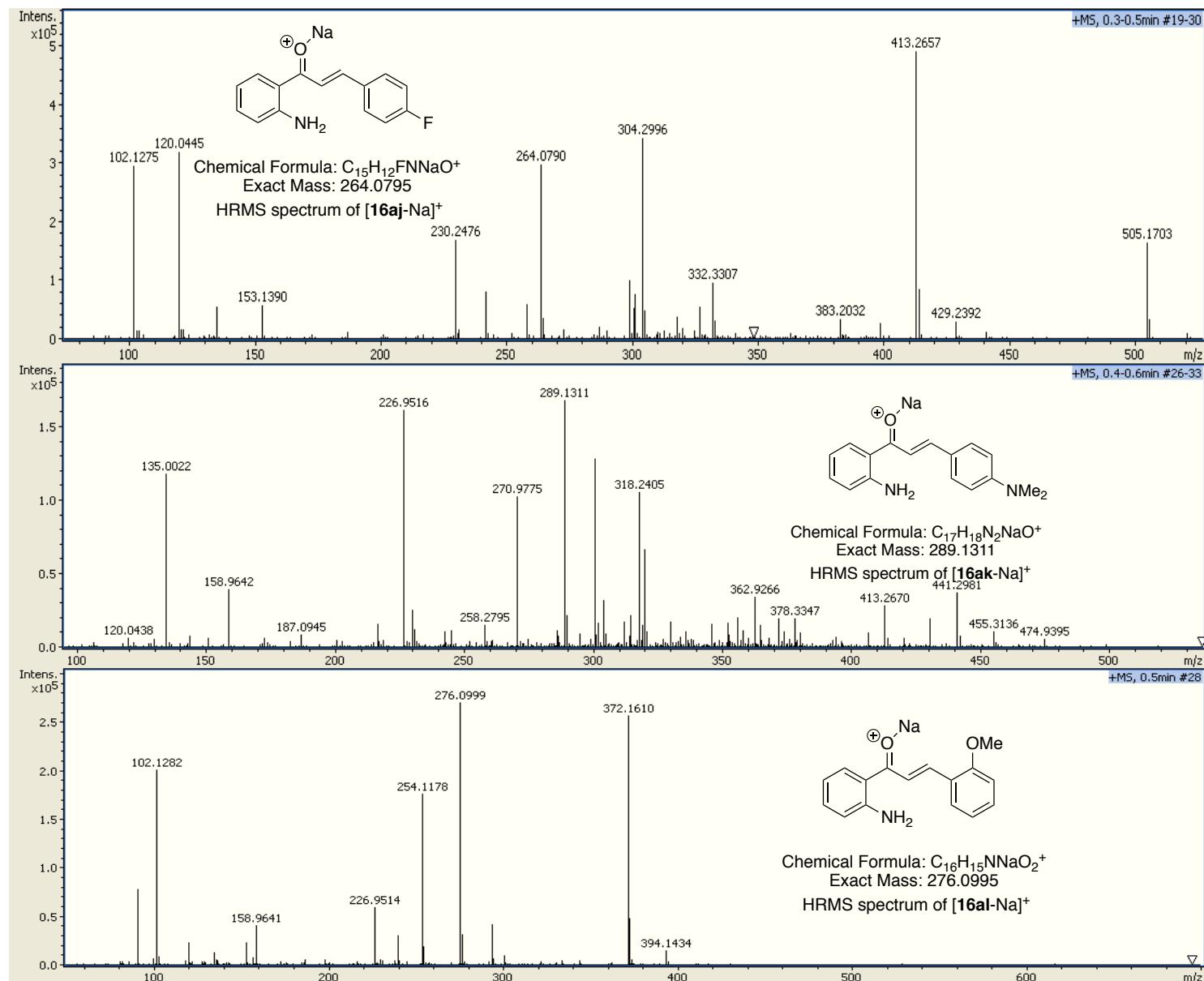


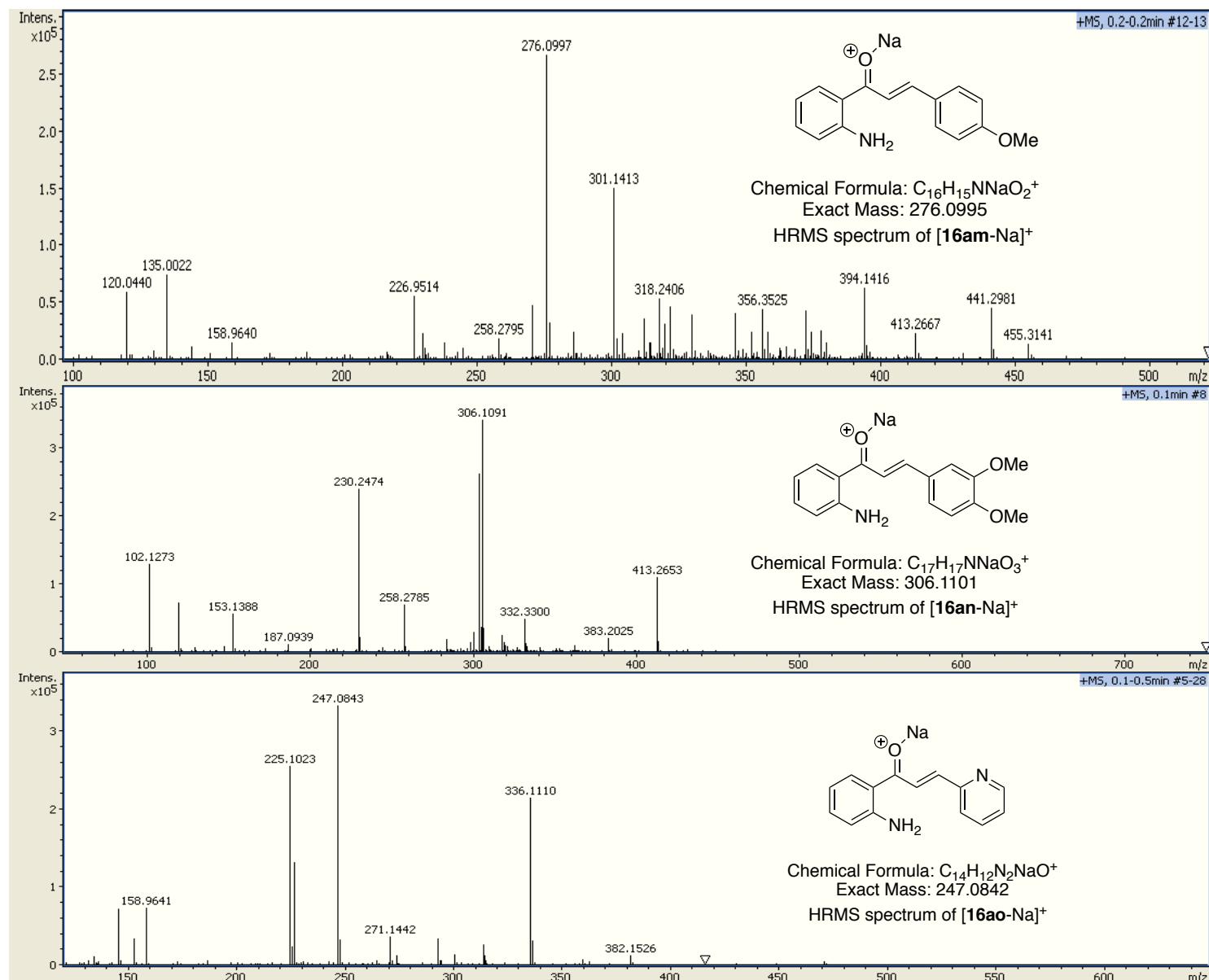
HRMS Spectral Charts of (E)-1-(2-aminophenyl)-3-arylprop-2-en-1-ones 16

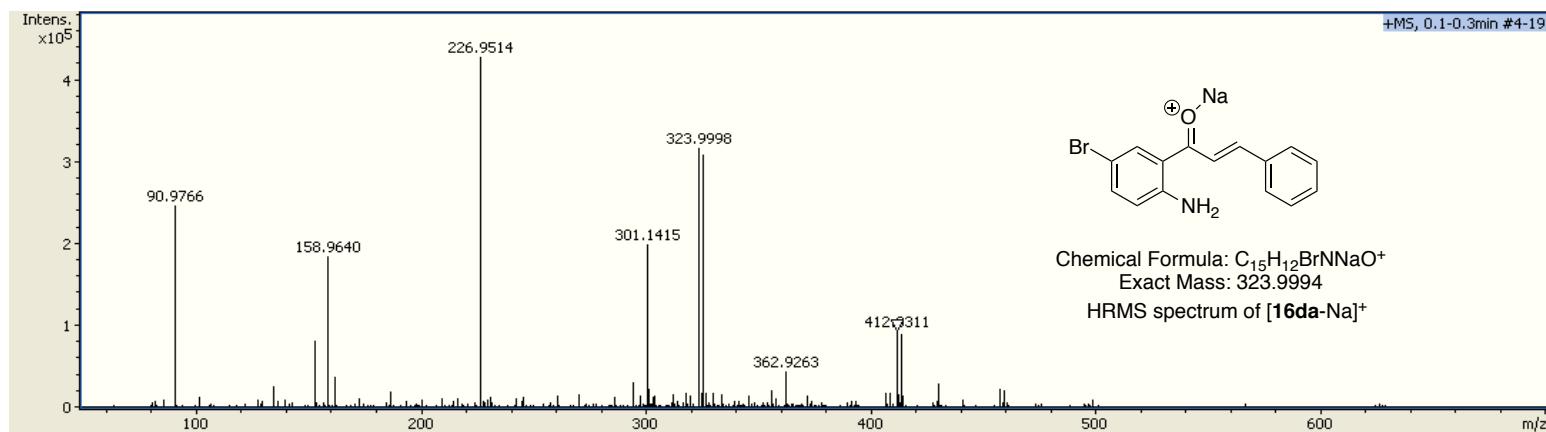




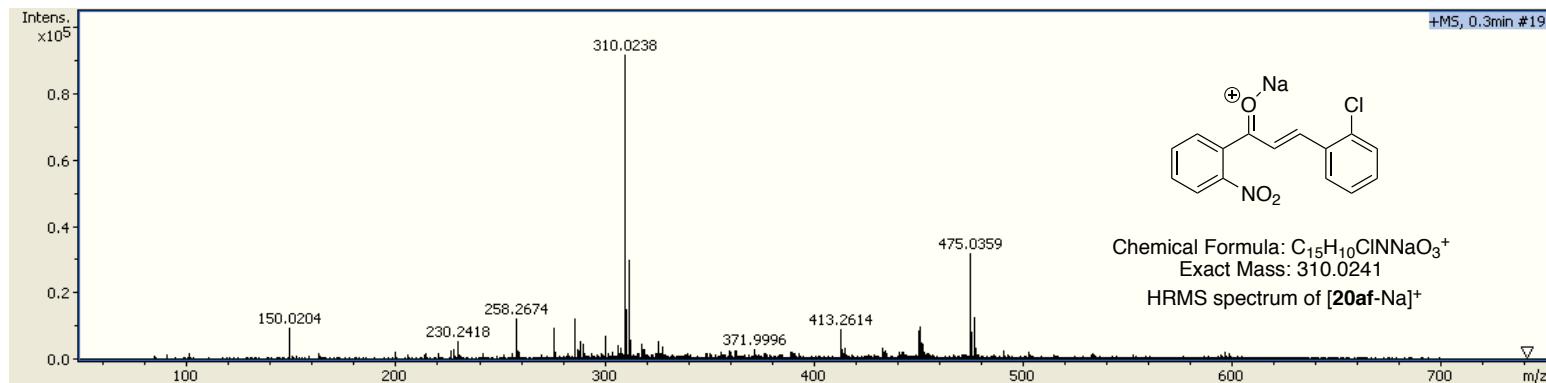


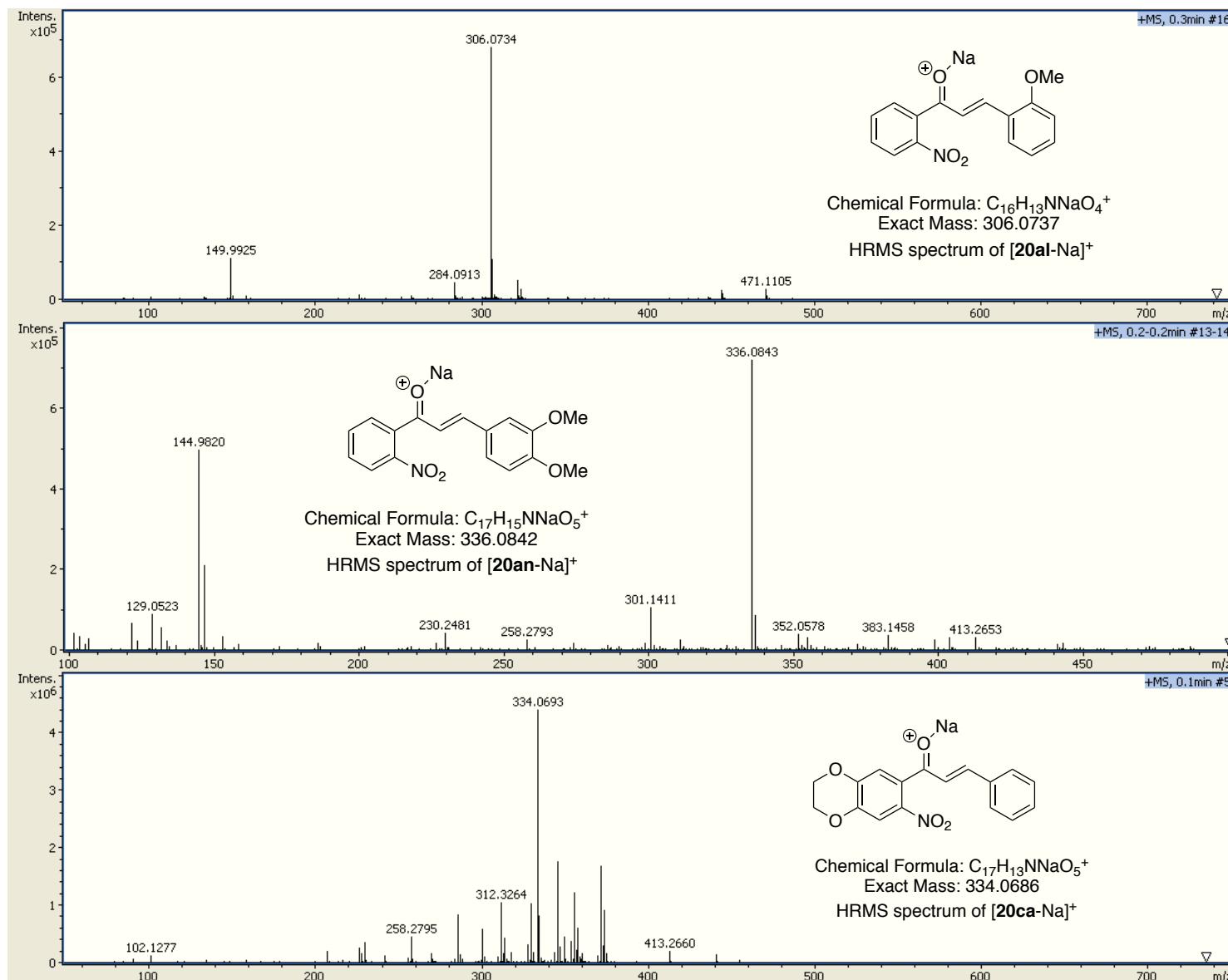


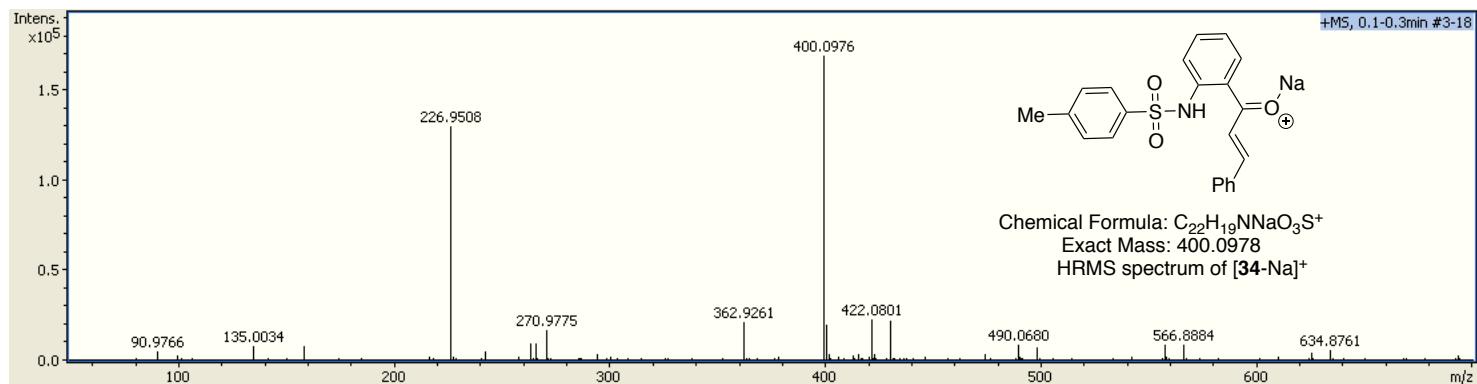
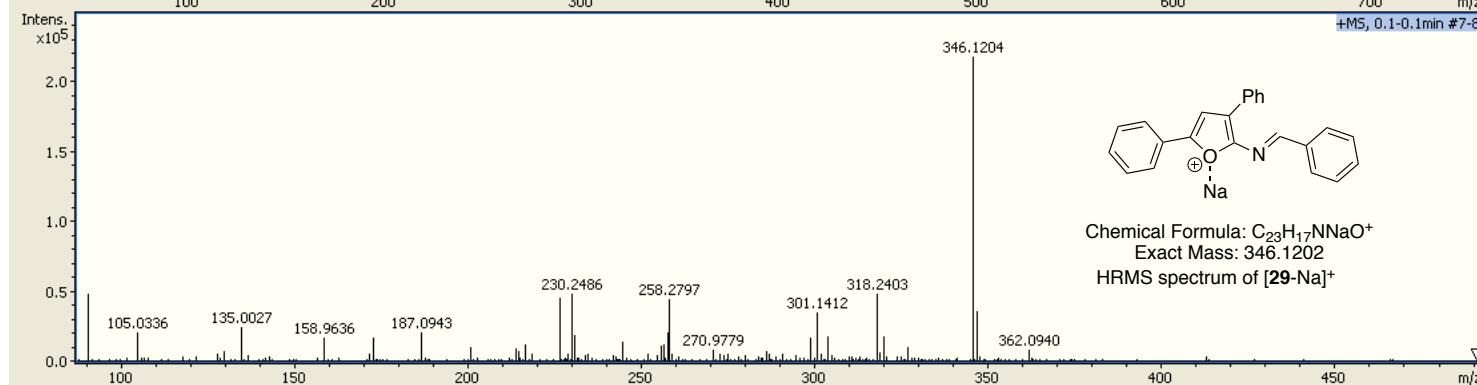
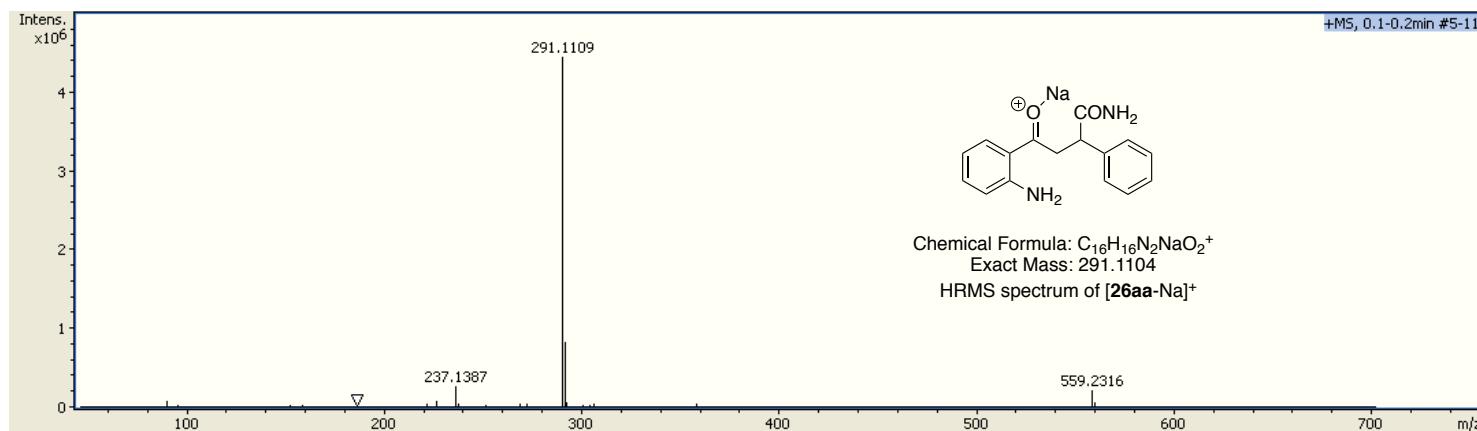


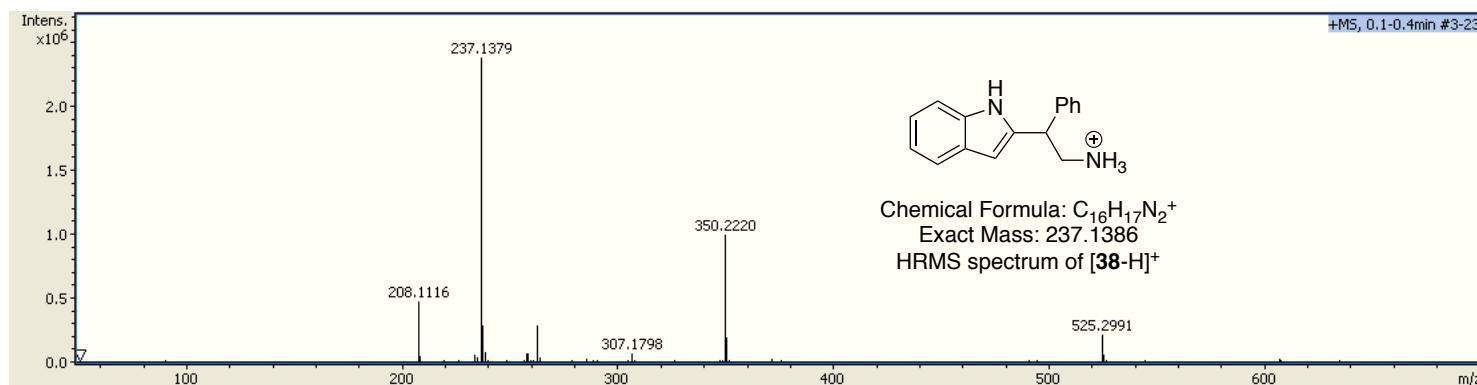
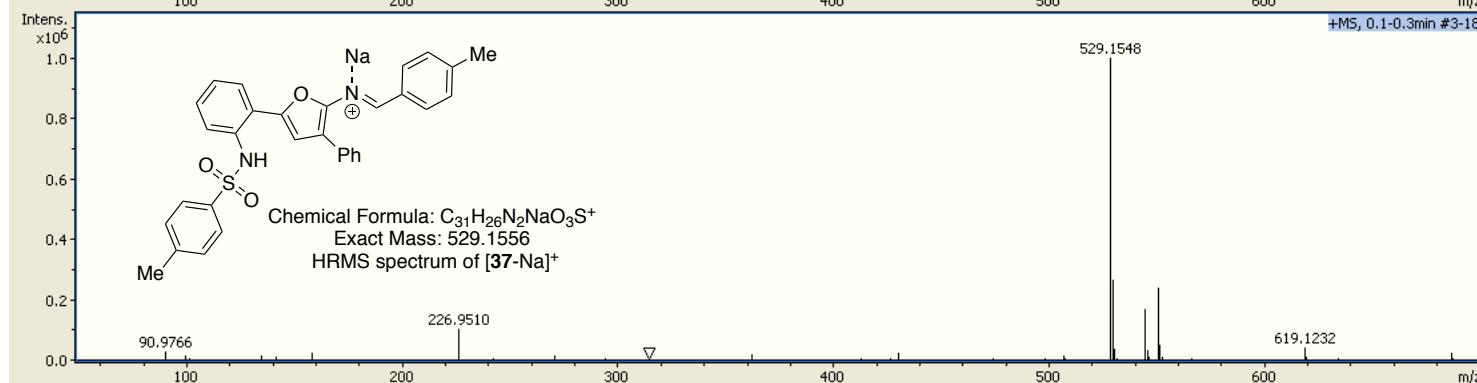
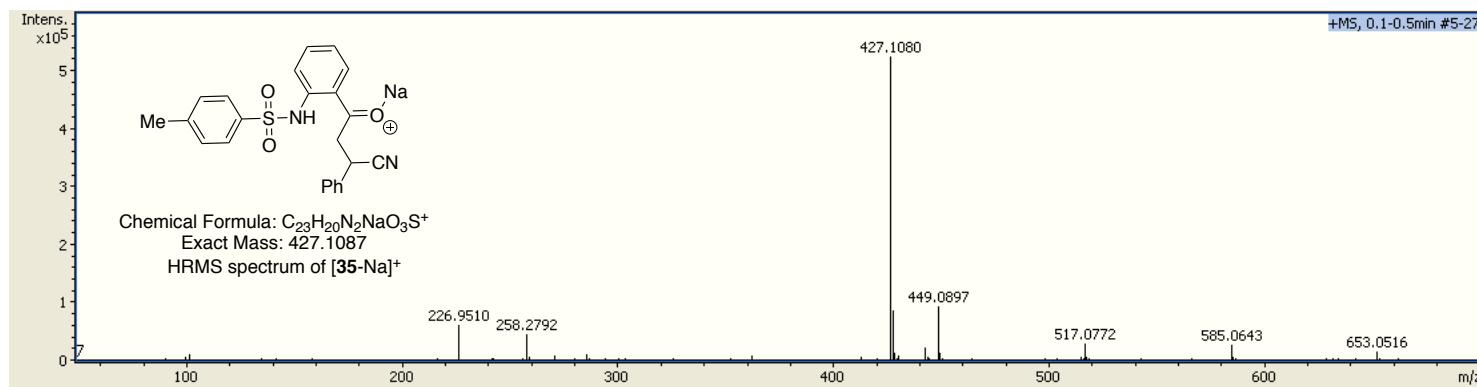


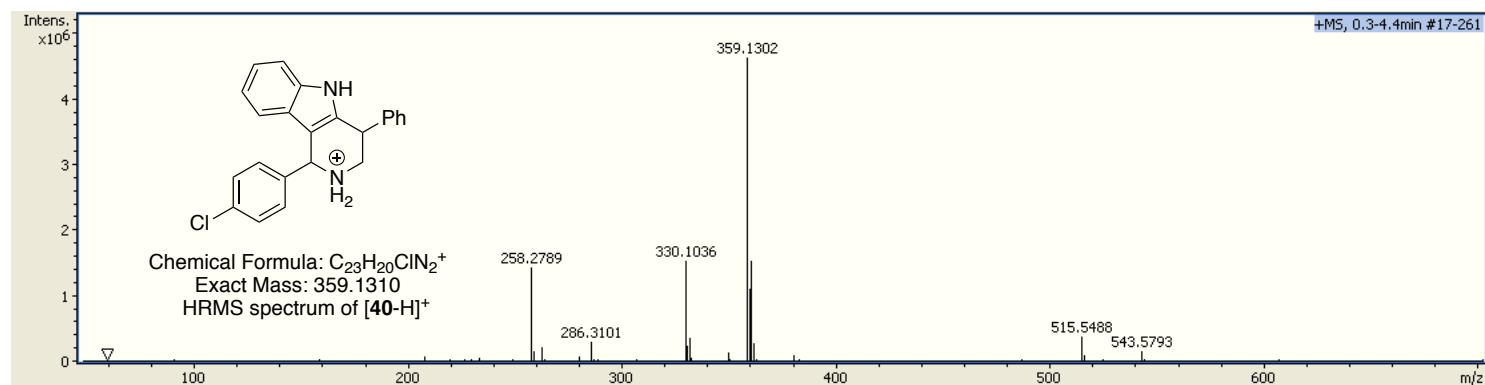
HRMS Spectral Charts of (*E*)-1-(2-nitrophenyl)-3-arylprop-2-en-1-ones 20







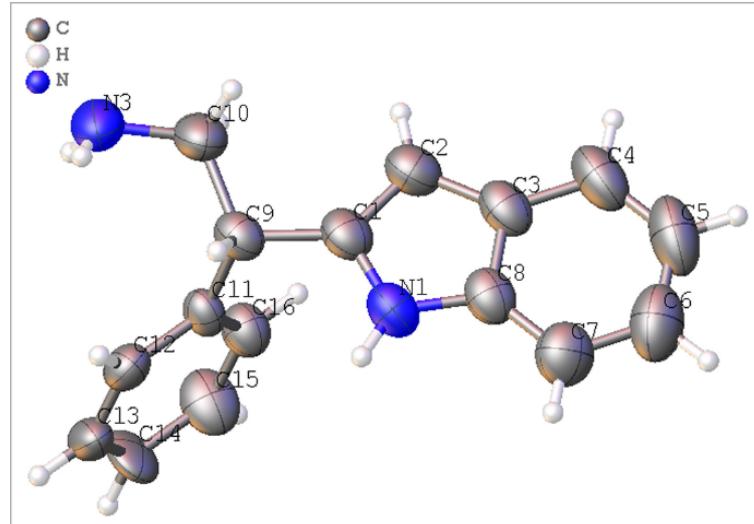




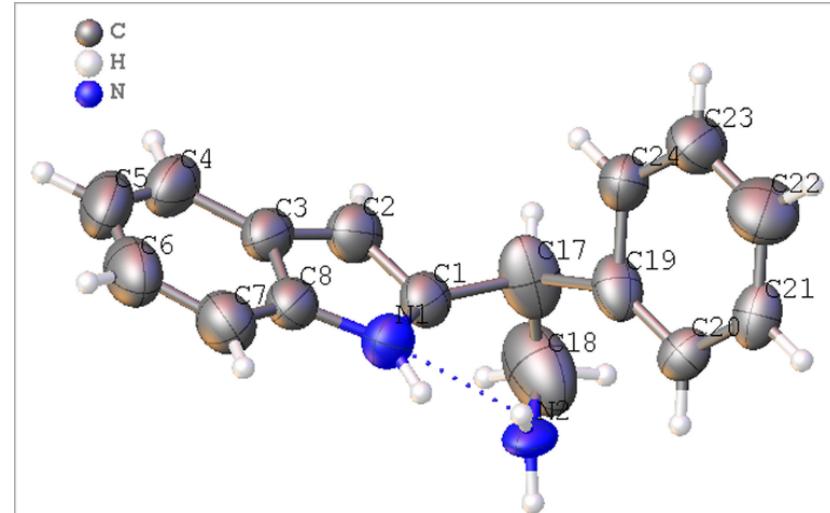
X-Ray Crystallography data for amine 38

ORTEP Drawing

Residue 1



Residue 2



Sample Pictures of crystal



Table S1. Crystal data and structure refinement for 38.

Identification code	ANNA1229_tryptamine_2
Empirical formula	C ₁₆ H ₁₆ N ₂
Formula weight	236.31
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	Pbca
a/Å	11.62377(13)
b/Å	11.62379(12)
c/Å	19.3359(2)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	2612.52(5)
Z	8
ρ _{calc} g/cm ³	1.202
μ/mm ⁻¹	0.550
F(000)	1008.0
Crystal size/mm ³	0.421 × 0.232 × 0.121
Radiation	CuKα ($\lambda = 1.54184$)
2Θ range for data collection/°	9.148 to 152.952
Index ranges	-14 ≤ h ≤ 14, -14 ≤ k ≤ 12, -24 ≤ l ≤ 24
Reflections collected	31528
Independent reflections	2722 [$R_{\text{int}} = 0.0157$, $R_{\text{sigma}} = 0.0057$]
Data/restraints/parameters	2722/0/235
Goodness-of-fit on F ²	1.045
Final R indexes [I>=2σ (I)]	$R_1 = 0.0430$, $wR_2 = 0.1305$
Final R indexes [all data]	$R_1 = 0.0466$, $wR_2 = 0.1348$
Largest diff. peak/hole / e Å ⁻³	0.23/-0.16

Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for ANNA1229_triptamine_2. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	$U(\text{eq})$
N ₍₁₎	4277.3(10)	3324.9(9)	6632.4(6)	59.1(3)
C ₍₁₁₎	5902(5)	5355(4)	6514(3)	44.4(15)
C ₍₁₂₎	5670(9)	6410(5)	6823(3)	53.6(13)
C ₍₁₃₎	5545(9)	7388(3)	6417(5)	61.8(15)
C ₍₁₄₎	5651(6)	7312(3)	5702(4)	71.3(19)
C ₍₁₅₎	5882(11)	6258(4)	5394(3)	73.7(16)
C ₍₁₆₎	6008(11)	5279(4)	5800(3)	55.7(13)
N ₍₃₎	7929.6(17)	4924.6(14)	7511.7(11)	76.0(5)
C ₍₈₎	3892.9(12)	2323.8(11)	6337.8(6)	57.8(3)
C ₍₃₎	4862.4(13)	1650.7(11)	6176.8(7)	60.0(3)
C ₍₁₎	5448.1(12)	3307.9(10)	6668.0(6)	56.2(3)
C ₍₂₎	5843.8(13)	2300.9(11)	6389.9(7)	62.7(4)
C ₍₉₎	6038.9(18)	4340.8(13)	6974.1(7)	51.2(4)
C ₍₇₎	2783.3(15)	1969.4(15)	6188.2(8)	76.2(4)
C ₍₄₎	4689.1(18)	575.2(13)	5870.2(9)	78.8(5)
C ₍₁₀₎	7299.6(18)	4058.4(14)	7125.4(11)	67.9(5)
C ₍₅₎	3585(2)	222.8(15)	5723.8(10)	92.3(6)
C ₍₆₎	2643.8(19)	912.2(16)	5876.0(10)	90.4(6)
C ₍₁₇₎	6549(17)	4133(16)	6735(16)	83(6)
C ₍₁₈₎	6660(30)	4190(30)	7469(16)	128(11)
N ₍₂₎	5526(12)	4517(9)	7614(5)	59(4)
C ₍₁₉₎	5960(5)	5429(5)	6500(3)	61(2)
C ₍₂₄₎	6343(11)	5370(5)	5820(3)	59.5(14)
C ₍₂₃₎	6214(12)	6312(6)	5384(3)	76.1(17)
C ₍₂₂₎	5702(7)	7313(4)	5628(4)	92(3)
C ₍₂₁₎	5319(8)	7372(4)	6308(5)	56.7(13)
C ₍₂₀₎	5448(8)	6430(5)	6744(4)	47.9(12)

Table S3. Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for ANNA1229_tryptamine_2. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + 2hka^*b^*U_{12} + \dots]$.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
N ₍₁₎	70.5(7)	46.4(6)	60.4(6)	-1.0(5)	6.8(5)	2.4(5)
C ₍₁₁₎	47(3)	38(2)	48(3)	-8(2)	5(2)	0.6(18)
C ₍₁₂₎	47(3)	58(2)	56(2)	-5.1(16)	-12.6(17)	0.1(16)
C ₍₁₃₎	58(3)	51(2)	77(3)	-13.7(17)	-27(2)	11.1(17)
C ₍₁₄₎	105(5)	27(3)	82(4)	17(2)	-17(3)	3(2)
C ₍₁₅₎	106(4)	66(3)	50(2)	10.0(18)	9(2)	-7(2)
C ₍₁₆₎	67(3)	49.8(19)	50.2(18)	-2.4(13)	2.9(17)	-1.4(17)
N ₍₃₎	82.5(10)	58.8(9)	86.8(11)	2.5(8)	-31.5(9)	2.5(7)
C ₍₈₎	74.6(8)	49.6(7)	49.3(6)	4.1(5)	4.7(5)	-3.7(6)
C ₍₃₎	83.6(9)	44.8(7)	51.7(6)	4.2(5)	5.2(6)	2.4(6)
C ₍₁₎	70.5(8)	44.6(6)	53.4(6)	7.9(5)	0.1(5)	1.3(5)
C ₍₂₎	70.3(8)	51.6(7)	66.3(8)	6.1(6)	2.1(6)	7.4(6)
C ₍₉₎	63.6(10)	44.9(7)	45.0(7)	1.1(5)	2.0(6)	5.1(7)
C ₍₇₎	78.2(10)	75.3(10)	75.1(9)	4.7(8)	5.2(7)	-11.4(8)
C ₍₄₎	119.3(13)	47.9(7)	69.1(9)	-3.8(7)	6.3(9)	3.7(8)
C ₍₁₀₎	75.3(12)	53.1(9)	75.4(11)	2.1(8)	-20.2(9)	10.4(8)
C ₍₅₎	142.7(17)	60.2(9)	73.9(10)	-11.0(8)	1.3(11)	-25.8(10)
C ₍₆₎	108.2(14)	85.7(12)	77.2(10)	-0.1(9)	-3.9(10)	-35.4(11)
C ₍₁₇₎	51(9)	67(10)	132(18)	23(11)	-12(10)	11(7)
C ₍₁₈₎	120(20)	140(20)	120(20)	31(18)	-31(18)	-51(19)
N ₍₂₎	101(9)	50(6)	25(4)	0(4)	14(4)	1(5)
C ₍₁₉₎	71(4)	52(3)	59(4)	17(3)	-20(3)	-10(3)
C ₍₂₄₎	67(4)	47.6(18)	64(2)	-2.5(15)	5(2)	-4.1(18)
C ₍₂₃₎	102(4)	63(3)	63(3)	8(2)	10(2)	-1(2)
C ₍₂₂₎	120(7)	94(7)	62(3)	15(3)	-9(3)	-2(5)
C ₍₂₁₎	54(3)	44.7(19)	72(3)	-1.5(15)	-22(2)	1.2(14)
C ₍₂₀₎	44(2)	51(2)	48.3(18)	-5.9(15)	-9.8(15)	3.7(14)

Table S4. Bond Lengths for ANNA1229_tryptamine_2.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N ₍₁₎	C ₍₈₎	1.3705(17)	C ₍₁₎	C ₍₉₎	1.5044(19)
N ₍₁₎	C ₍₁₎	1.3628(18)	C ₍₁₎	C ₍₁₇₎	1.604(18)
C ₍₁₁₎	C ₍₁₂₎	1.3900	C ₍₉₎	C ₍₁₀₎	1.530(3)
C ₍₁₁₎	C ₍₁₆₎	1.3900	C ₍₇₎	C ₍₆₎	1.379(2)
C ₍₁₁₎	C ₍₉₎	1.485(5)	C ₍₄₎	C ₍₅₎	1.377(3)
C ₍₁₂₎	C ₍₁₃₎	1.3900	C ₍₅₎	C ₍₆₎	1.388(3)
C ₍₁₃₎	C ₍₁₄₎	1.3900	C ₍₁₇₎	C ₍₁₈₎	1.43(5)
C ₍₁₄₎	C ₍₁₅₎	1.3900	C ₍₁₇₎	C ₍₁₉₎	1.715(17)
C ₍₁₅₎	C ₍₁₆₎	1.3900	C ₍₁₈₎	N ₍₂₎	1.40(4)
N ₍₃₎	C ₍₁₀₎	1.452(2)	C ₍₁₉₎	C ₍₂₄₎	1.3900
C ₍₈₎	C ₍₃₎	1.407(2)	C ₍₁₉₎	C ₍₂₀₎	1.3900
C ₍₈₎	C ₍₇₎	1.384(2)	C ₍₂₄₎	C ₍₂₃₎	1.3900
C ₍₃₎	C ₍₂₎	1.429(2)	C ₍₂₃₎	C ₍₂₂₎	1.3900
C ₍₃₎	C ₍₄₎	1.3981(19)	C ₍₂₂₎	C ₍₂₁₎	1.3900
C ₍₁₎	C ₍₂₎	1.3678(18)	C ₍₂₁₎	C ₍₂₀₎	1.3900

Table S5. Bond Angles for ANNA1229_tryptamine_2.

Atom	Atom	Atom	Angle/ ^o	Atom	Atom	Atom	Angle/ ^o
C ₍₁₎	N ₍₁₎	C ₍₈₎	109.53(11)	C ₍₁₁₎	C ₍₉₎	C ₍₁₎	110.4(3)
C ₍₁₂₎	C ₍₁₁₎	C ₍₁₆₎	120.0	C ₍₁₁₎	C ₍₉₎	C ₍₁₀₎	112.8(3)
C ₍₁₂₎	C ₍₁₁₎	C ₍₉₎	117.6(3)	C ₍₁₎	C ₍₉₎	C ₍₁₀₎	109.95(14)
C ₍₁₆₎	C ₍₁₁₎	C ₍₉₎	122.4(3)	C ₍₆₎	C ₍₇₎	C ₍₈₎	117.80(17)
C ₍₁₃₎	C ₍₁₂₎	C ₍₁₁₎	120.0	C ₍₅₎	C ₍₄₎	C ₍₃₎	119.18(17)
C ₍₁₂₎	C ₍₁₃₎	C ₍₁₄₎	120.0	N ₍₃₎	C ₍₁₀₎	C ₍₉₎	115.64(15)
C ₍₁₅₎	C ₍₁₄₎	C ₍₁₃₎	120.0	C ₍₄₎	C ₍₅₎	C ₍₆₎	121.30(16)
C ₍₁₄₎	C ₍₁₅₎	C ₍₁₆₎	120.0	C ₍₇₎	C ₍₆₎	C ₍₅₎	120.98(18)
C ₍₁₅₎	C ₍₁₆₎	C ₍₁₁₎	120.0	C ₍₁₎	C ₍₁₇₎	C ₍₁₉₎	100.7(9)
N ₍₁₎	C ₍₈₎	C ₍₃₎	107.64(12)	C ₍₁₈₎	C ₍₁₇₎	C ₍₁₎	100(2)
N ₍₁₎	C ₍₈₎	C ₍₇₎	130.03(14)	C ₍₁₈₎	C ₍₁₇₎	C ₍₁₉₎	105(3)
C ₍₇₎	C ₍₈₎	C ₍₃₎	122.31(13)	N ₍₂₎	C ₍₁₈₎	C ₍₁₇₎	97(2)
C ₍₈₎	C ₍₃₎	C ₍₂₎	106.35(11)	C ₍₂₄₎	C ₍₁₉₎	C ₍₁₇₎	94.6(12)
C ₍₄₎	C ₍₃₎	C ₍₈₎	118.41(15)	C ₍₂₄₎	C ₍₁₉₎	C ₍₂₀₎	120.0
C ₍₄₎	C ₍₃₎	C ₍₂₎	135.24(15)	C ₍₂₀₎	C ₍₁₉₎	C ₍₁₇₎	144.7(12)
N ₍₁₎	C ₍₁₎	C ₍₂₎	109.17(12)	C ₍₂₃₎	C ₍₂₄₎	C ₍₁₉₎	120.0
N ₍₁₎	C ₍₁₎	C ₍₉₎	117.65(13)	C ₍₂₂₎	C ₍₂₃₎	C ₍₂₄₎	120.0
N ₍₁₎	C ₍₁₎	C ₍₁₇₎	142.3(8)	C ₍₂₃₎	C ₍₂₂₎	C ₍₂₁₎	120.0
C ₍₂₎	C ₍₁₎	C ₍₉₎	133.17(15)	C ₍₂₀₎	C ₍₂₁₎	C ₍₂₂₎	120.0
C ₍₂₎	C ₍₁₎	C ₍₁₇₎	106.0(10)	C ₍₂₁₎	C ₍₂₀₎	C ₍₁₉₎	120.0
C ₍₁₎	C ₍₂₎	C ₍₃₎	107.31(13)				

Table S6. Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for ANNA1229_triptamine_2.

Atom	x	y	z	U(eq)
H ₍₁₂₎	5598.86	6460.39	7300.82	64
H ₍₁₃₎	5389.65	8094.25	6623.27	74
H ₍₁₄₎	5566.98	7967.43	5430.61	86
H ₍₁₅₎	5953.52	6206.76	4915.51	88
H ₍₁₆₎	6162.74	4572.88	5593.05	67
H ₍₂₎	6609.59	2080.26	6347.39	75
H ₍₉₎	5662.58	4518.02	7414.91	61
H ₍₇₎	2153.01	2429.15	6294.8	91
H ₍₄₎	5312.1	103.76	5766.53	95
H _(10A)	7328.48	3341.8	7381.88	82
H _(10B)	7691.18	3933.14	6688.87	82
H ₍₅₎	3467.61	-491.35	5518.75	111
H ₍₆₎	1907.89	657.35	5765.74	108
H ₍₁₇₎	7236.02	3894.52	6479.32	100
H _(18A)	6852.69	3445.75	7668.77	153
H _(18B)	7216.21	4759.71	7614.39	153
H _(2A)	5042.64	4612.59	7281.91	70
H _(2B)	5312.83	4624.08	8034.92	70
H ₍₁₎	3852(15)	3884(15)	6836(10)	84(5)
H ₍₂₄₎	6685.94	4699.75	5656.34	71
H ₍₂₃₎	6470.55	6272.26	4928.87	91
H ₍₂₂₎	5615.67	7943.54	5336.42	110
H ₍₂₁₎	4976.18	8042.32	6471.45	68
H ₍₂₀₎	5191.56	6469.81	7198.93	57
H _(3A)	8090(20)	5470(20)	7252(12)	103(9)
H _(3B)	7480(20)	5200(20)	7783(12)	85(8)

Table S7. Atomic Occupancy for ANNA1229_tryptamine_2.

Atom	<i>Occupancy</i>	Atom	<i>Occupancy</i>	Atom	<i>Occupancy</i>
C ₍₁₁₎	0.51(3)	C ₍₁₂₎	0.51(3)	H ₍₁₂₎	0.51(3)
C ₍₁₃₎	0.51(3)	H ₍₁₃₎	0.51(3)	C ₍₁₄₎	0.51(3)
H ₍₁₄₎	0.51(3)	C ₍₁₅₎	0.51(3)	H ₍₁₅₎	0.51(3)
C ₍₁₆₎	0.51(3)	H ₍₁₆₎	0.51(3)	N ₍₃₎	0.900(4)
C ₍₉₎	0.900(4)	H ₍₉₎	0.900(4)	C ₍₁₀₎	0.900(4)
H _(10A)	0.900(4)	H _(10B)	0.900(4)	C ₍₁₇₎	0.100(4)
H ₍₁₇₎	0.100(4)	C ₍₁₈₎	0.100(4)	H _(18A)	0.100(4)
H _(18B)	0.100(4)	N ₍₂₎	0.100(4)	H _(2A)	0.09(3)
H _(2B)	0.1	C ₍₁₉₎	0.49(3)	C ₍₂₄₎	0.49(3)
H ₍₂₄₎	0.49(3)	C ₍₂₃₎	0.49(3)	H ₍₂₃₎	0.49(3)
C ₍₂₂₎	0.49(3)	H ₍₂₂₎	0.49(3)	C ₍₂₁₎	0.49(3)
H ₍₂₁₎	0.49(3)	C ₍₂₀₎	0.49(3)	H ₍₂₀₎	0.49(3)
H _(3A)	0.91(3)	H _(3B)	0.9		

Experimental

Single crystals of C₁₆H₁₆N₂ (**38**) [ANNA1229_triptamine_2] were prepared by slow evaporation of chloroform solution. A suitable crystal was selected and mounted by acrylic glue on the glass stick on a SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer. The crystal was kept at 293(2) 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 [ANNA1229_triptamine_2]

Crystal Data for C₁₆H₁₆N₂ ($M=236.31\text{ g/mol}$): orthorhombic, space group Pbca (no. 61), $a = 11.62377(13)\text{ \AA}$, $b = 11.62379(12)\text{ \AA}$, $c = 19.3359(2)\text{ \AA}$, $V = 2612.52(5)\text{ \AA}^3$, $Z = 8$, $T = 293(2)\text{ K}$, $\mu(\text{CuK}\alpha) = 0.550\text{ mm}^{-1}$, $D_{\text{calc}} = 1.202\text{ g/cm}^3$, 31528 reflections measured ($9.148^\circ \leq 2\Theta \leq 152.952^\circ$), 2722 unique ($R_{\text{int}} = 0.0157$, $R_{\text{sigma}} = 0.0057$) which were used in all calculations. The final R_1 was 0.0430 ($I > 2\sigma(I)$) and wR_2 was 0.1348 (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, All N(H,H) groups

2. Others

Sof(C17)=Sof(H17)=Sof(C18)=Sof(H18A)=Sof(H18B)=Sof(N2)=1-FVAR(1)

Sof(N3)=Sof(C9)=Sof(H9)=Sof(C10)=Sof(H10A)=Sof(H10B)=FVAR(1)

Sof(C19)=Sof(C24)=Sof(H24)=Sof(C23)=Sof(H23)=Sof(C22)=Sof(H22)=Sof(C21)=

Sof(H21)=Sof(C20)=Sof(H20)=1-FVAR(2)

Sof(C11)=Sof(C12)=Sof(H12)=Sof(C13)=Sof(H13)=Sof(C14)=Sof(H14)=Sof(C15)=

Sof(H15)=Sof(C16)=Sof(H16)=FVAR(2)

Sof(H3A)=1-FVAR(3)

Sof(H2A)=FVAR(3)

Fixed Sof: H2B(0.1) H3B(0.9)

3.a Ternary CH refined with riding coordinates:

C9(H9), C17(H17)

3.b Secondary CH₂ refined with riding coordinates:

C10(H10A,H10B), C18(H18A,H18B)

3.c Aromatic/amide H refined with riding coordinates:

C12(H12), C13(H13), C14(H14), C15(H15), C16(H16), C2(H2), C7(H7), C4(H4),
C5(H5), C6(H6), C24(H24), C23(H23), C22(H22), C21(H21), C20(H20)

3.d Fitted hexagon refined as free rotating group:

C11(C12,C13,C14,C15,C16), C19(C24,C23,C22,C21,C20)

3.e X=CH₂ refined with riding coordinates:

N2(H2A,H2B)

References Cited

- (S1) Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. OLEX2: a Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Cryst.* **2009**, *42*, 339-341.
- (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.