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Traceless Redox-Annulations of Alicyclic Amines

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Supporting Information

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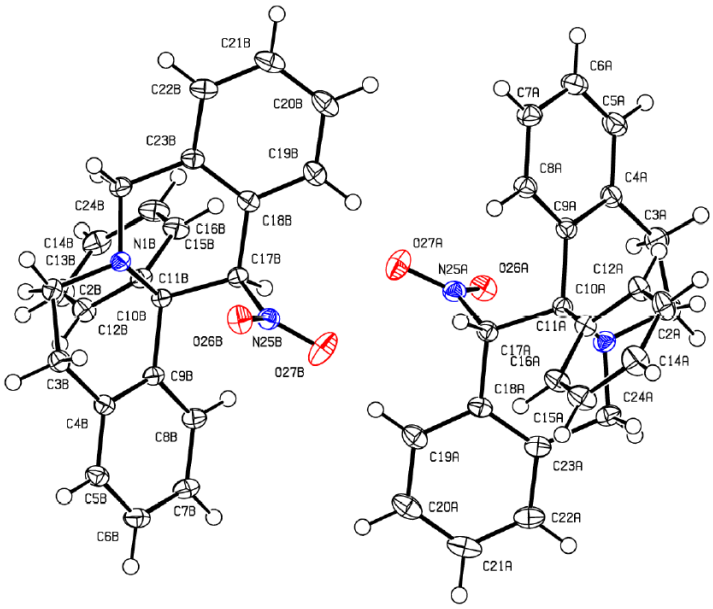
Crystallographic Summary for Compound (±)-2e



The requisite CIF of **(±)-2e** was submitted to the journal and deposited with the CCDC (deposition # 202419)

Supplementary Table 1. Crystal data and structure refinement for rick1.

Identification code	rick1	
Empirical formula	$C_{23}H_{20}N_2O_2$	
Formula weight	356.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.5713(2) Å	a= 90°.
	b = 16.9061(5) Å	b= 90°.
	c = 28.2644(8) Å	g = 90°.
Volume	3617.88(18) $Å^3$	
Z	8	
Density (calculated)	1.309 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	1504	
Crystal size	? x ? x ? mm^3	

Theta range for data collection	1.878 to 33.209°.
Index ranges	-11≤h≤11, -26≤k≤25, -41≤l≤41
Reflections collected	99381
Independent reflections	12862 [R(int) = 0.0594]
Completeness to theta = 25.000°	99.7 %
Absorption correction	"Multi
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12862 / 0 / 487
Goodness-of-fit on F^2	1.028
Final R indices [I>2sigma(I)]	R1 = 0.0404, wR2 = 0.1004 [11228]
R indices (all data)	R1 = 0.0500, wR2 = 0.1058
Absolute structure parameter	0.4(3)
Largest diff. peak and hole	0.330 and -0.198 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for rick1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O26A	2250(2)	3640(1)	7148(1)	24(1)
O26B	7224(2)	6319(1)	5680(1)	25(1)
O27A	4128(2)	4061(1)	6630(1)	30(1)
O27B	5241(2)	5950(1)	6180(1)	35(1)
N1A	2098(2)	4637(1)	8000(1)	18(1)
N1B	7425(2)	5277(1)	4842(1)	16(1)
N25A	3238(2)	4145(1)	6990(1)	20(1)
N25B	6187(2)	5834(1)	5835(1)	19(1)
C2A	2230(2)	4234(1)	8454(1)	22(1)
C2B	7349(2)	5627(1)	4370(1)	18(1)
C3A	3029(2)	3422(1)	8373(1)	25(1)
C3B	6493(2)	6436(1)	4405(1)	19(1)
C4A	4770(2)	3493(1)	8121(1)	20(1)
C4B	4746(2)	6386(1)	4655(1)	17(1)
C5A	6073(2)	2913(1)	8179(1)	24(1)
C5B	3438(2)	6953(1)	4570(1)	20(1)
C6A	7695(2)	2977(1)	7957(1)	25(1)

C6B	1804(2)	6912(1)	4789(1)	22(1)
C7A	8048(2)	3627(1)	7669(1)	23(1)
C7B	1448(2)	6302(1)	5107(1)	22(1)
C8A	6762(2)	4204(1)	7607(1)	20(1)
C8B	2742(2)	5741(1)	5198(1)	19(1)
C9A	5115(2)	4148(1)	7832(1)	17(1)
C9B	4393(2)	5773(1)	4974(1)	16(1)
C10A	3796(2)	4828(1)	7773(1)	16(1)
C10B	5706(2)	5102(1)	5063(1)	15(1)
C11A	4637(2)	5595(1)	7976(1)	17(1)
C11B	4869(2)	4323(1)	4879(1)	17(1)
C12A	5688(2)	5546(1)	8382(1)	21(1)
C12B	3940(2)	4336(1)	4450(1)	21(1)
C13A	6401(3)	6219(1)	8590(1)	26(1)
C13B	3210(2)	3651(1)	4258(1)	24(1)
C14A	6074(3)	6962(1)	8396(1)	29(1)
C14B	3385(2)	2934(1)	4494(1)	25(1)
C15A	5050(2)	7021(1)	7990(1)	26(1)
C15B	4283(2)	2911(1)	4921(1)	26(1)
C16A	4343(2)	6346(1)	7782(1)	21(1)
C16B	5017(2)	3597(1)	5112(1)	21(1)
C17A	3383(2)	4945(1)	7239(1)	17(1)
C17B	6064(2)	5020(1)	5604(1)	16(1)
C18A	1696(2)	5401(1)	7138(1)	19(1)
C18B	7727(2)	4568(1)	5726(1)	18(1)
C19A	1394(2)	5680(1)	6679(1)	23(1)
C19B	7981(2)	4303(1)	6191(1)	22(1)
C20A	-104(2)	6126(1)	6581(1)	27(1)
C20B	9462(2)	3860(1)	6305(1)	26(1)
C21A	-1288(2)	6299(1)	6942(1)	28(1)
C21B	10714(2)	3689(1)	5961(1)	26(1)
C22A	-980(2)	6032(1)	7398(1)	25(1)
C22B	10464(2)	3949(1)	5500(1)	22(1)
C23A	510(2)	5571(1)	7500(1)	21(1)
C23B	8969(2)	4388(1)	5378(1)	18(1)
C24A	834(2)	5284(1)	7996(1)	22(1)

C24B	8677(2)	4630(1)	4874(1)	19(1)

Supplementary Table 3. Bond lengths [Å] and angles [°] for rick1.

O26A-N25A	1.2198(18)
O26B-N25B	1.2175(17)
O27A-N25A	1.2278(18)
O27B-N25B	1.2267(18)
N1A-C24A	1.4538(19)
N1A-C2A	1.4571(19)
N1A-C10A	1.4727(19)
N1B-C24B	1.4501(18)
N1B-C2B	1.4588(18)
N1B-C10B	1.4741(18)
N25A-C17A	1.5282(18)
N25B-C17B	1.5259(18)
C2A-C3A	1.518(2)
C2A-H2AA	0.9900
C2A-H2AB	0.9900
C2B-C3B	1.516(2)
C2B-H2BA	0.9900
C2B-H2BB	0.9900
C3A-C4A	1.503(2)
C3A-H3AA	0.9900
C3A-H3AB	0.9900
C3B-C4B	1.503(2)
C3B-H3BA	0.9900
C3B-H3BB	0.9900
C4A-C9A	1.3992(19)
C4A-C5A	1.401(2)
C4B-C9B	1.3992(19)
C4B-C5B	1.399(2)
C5A-C6A	1.383(3)
C5A-H5AA	0.9500

C5B-C6B	1.385(2)
C5B-H5BA	0.9500
C6A-C7A	1.392(2)
C6A-H6AA	0.9500
C6B-C7B	1.394(2)
C6B-H6BA	0.9500
C7A-C8A	1.390(2)
C7A-H7AA	0.9500
C7B-C8B	1.389(2)
C7B-H7BA	0.9500
C8A-C9A	1.403(2)
C8A-H8AA	0.9500
C8B-C9B	1.402(2)
C8B-H8BA	0.9500
C9A-C10A	1.533(2)
C9B-C10B	1.5296(19)
C10A-C11A	1.553(2)
C10A-C17A	1.553(2)
C10B-C11B	1.5518(19)
C10B-C17B	1.5576(19)
C11A-C16A	1.400(2)
C11A-C12A	1.401(2)
C11B-C16B	1.397(2)
C11B-C12B	1.401(2)
C12A-C13A	1.390(2)
C12A-H12A	0.9500
C12B-C13B	1.394(2)
C12B-H12B	0.9500
C13A-C14A	1.392(2)
C13A-H13A	0.9500
C13B-C14B	1.389(2)
C13B-H13B	0.9500
C14A-C15A	1.387(2)
C14A-H14A	0.9500
C14B-C15B	1.386(2)
C14B-H14B	0.9500

C15A-C16A	1.391(2)
C15A-H15A	0.9500
C15B-C16B	1.395(2)
C15B-H15B	0.9500
C16A-H16A	0.9500
C16B-H16B	0.9500
C17A-C18A	1.518(2)
C17A-H17A	1.0000
C17B-C18B	1.513(2)
C17B-H17B	1.0000
C18A-C23A	1.392(2)
C18A-C19A	1.397(2)
C18B-C23B	1.394(2)
C18B-C19B	1.400(2)
C19A-C20A	1.389(2)
C19A-H19A	0.9500
C19B-C20B	1.386(2)
C19B-H19B	0.9500
C20A-C21A	1.389(3)
C20A-H20A	0.9500
C20B-C21B	1.388(3)
C20B-H20B	0.9500
C21A-C22A	1.384(2)
C21A-H21A	0.9500
C21B-C22B	1.388(2)
C21B-H21B	0.9500
C22A-C23A	1.401(2)
C22A-H22A	0.9500
C22B-C23B	1.397(2)
C22B-H22B	0.9500
C23A-C24A	1.505(2)
C23B-C24B	1.499(2)
C24A-H24A	0.9900
C24A-H24B	0.9900
C24B-H24C	0.9900
C24B-H24D	0.9900

C24A-N1A-C2A	113.82(12)
C24A-N1A-C10A	113.93(11)
C2A-N1A-C10A	115.32(12)
C24B-N1B-C2B	112.93(11)
C24B-N1B-C10B	113.50(11)
C2B-N1B-C10B	115.80(12)
O26A-N25A-O27A	123.86(13)
O26A-N25A-C17A	119.75(12)
O27A-N25A-C17A	116.38(13)
O26B-N25B-O27B	123.79(13)
O26B-N25B-C17B	119.51(12)
O27B-N25B-C17B	116.67(13)
N1A-C2A-C3A	108.42(12)
N1A-C2A-H2AA	110.0
C3A-C2A-H2AA	110.0
N1A-C2A-H2AB	110.0
C3A-C2A-H2AB	110.0
H2AA-C2A-H2AB	108.4
N1B-C2B-C3B	108.94(11)
N1B-C2B-H2BA	109.9
C3B-C2B-H2BA	109.9
N1B-C2B-H2BB	109.9
C3B-C2B-H2BB	109.9
H2BA-C2B-H2BB	108.3
C4A-C3A-C2A	110.44(13)
C4A-C3A-H3AA	109.6
C2A-C3A-H3AA	109.6
C4A-C3A-H3AB	109.6
C2A-C3A-H3AB	109.6
H3AA-C3A-H3AB	108.1
C4B-C3B-C2B	110.81(12)
C4B-C3B-H3BA	109.5
C2B-C3B-H3BA	109.5
C4B-C3B-H3BB	109.5
C2B-C3B-H3BB	109.5

H3BA-C3B-H3BB	108.1
C9A-C4A-C5A	119.36(15)
C9A-C4A-C3A	120.22(13)
C5A-C4A-C3A	120.41(13)
C9B-C4B-C5B	118.87(14)
C9B-C4B-C3B	120.92(13)
C5B-C4B-C3B	120.20(13)
C6A-C5A-C4A	121.16(15)
C6A-C5A-H5AA	119.4
C4A-C5A-H5AA	119.4
C6B-C5B-C4B	121.43(14)
C6B-C5B-H5BA	119.3
C4B-C5B-H5BA	119.3
C5A-C6A-C7A	119.82(15)
C5A-C6A-H6AA	120.1
C7A-C6A-H6AA	120.1
C5B-C6B-C7B	119.82(14)
C5B-C6B-H6BA	120.1
C7B-C6B-H6BA	120.1
C8A-C7A-C6A	119.52(15)
C8A-C7A-H7AA	120.2
C6A-C7A-H7AA	120.2
C8B-C7B-C6B	119.28(15)
C8B-C7B-H7BA	120.4
C6B-C7B-H7BA	120.4
C7A-C8A-C9A	121.17(14)
C7A-C8A-H8AA	119.4
C9A-C8A-H8AA	119.4
C7B-C8B-C9B	121.25(14)
C7B-C8B-H8BA	119.4
C9B-C8B-H8BA	119.4
C4A-C9A-C8A	118.97(14)
C4A-C9A-C10A	122.39(13)
C8A-C9A-C10A	118.56(12)
C4B-C9B-C8B	119.33(13)
C4B-C9B-C10B	122.06(13)

C8B-C9B-C10B	118.48(12)
N1A-C10A-C9A	110.85(11)
N1A-C10A-C11A	112.35(11)
C9A-C10A-C11A	108.58(12)
N1A-C10A-C17A	106.03(12)
C9A-C10A-C17A	109.44(11)
C11A-C10A-C17A	109.54(11)
N1B-C10B-C9B	110.81(11)
N1B-C10B-C11B	112.83(11)
C9B-C10B-C11B	107.98(11)
N1B-C10B-C17B	106.33(11)
C9B-C10B-C17B	109.88(11)
C11B-C10B-C17B	108.99(11)
C16A-C11A-C12A	117.66(13)
C16A-C11A-C10A	123.20(13)
C12A-C11A-C10A	119.09(12)
C16B-C11B-C12B	117.56(13)
C16B-C11B-C10B	123.63(13)
C12B-C11B-C10B	118.79(12)
C13A-C12A-C11A	121.21(14)
C13A-C12A-H12A	119.4
C11A-C12A-H12A	119.4
C13B-C12B-C11B	121.48(14)
C13B-C12B-H12B	119.3
C11B-C12B-H12B	119.3
C12A-C13A-C14A	120.28(15)
C12A-C13A-H13A	119.9
C14A-C13A-H13A	119.9
C14B-C13B-C12B	120.04(15)
C14B-C13B-H13B	120.0
C12B-C13B-H13B	120.0
C15A-C14A-C13A	119.25(15)
C15A-C14A-H14A	120.4
C13A-C14A-H14A	120.4
C15B-C14B-C13B	119.29(14)
C15B-C14B-H14B	120.4

C13B-C14B-H14B	120.4
C14A-C15A-C16A	120.42(15)
C14A-C15A-H15A	119.8
C16A-C15A-H15A	119.8
C14B-C15B-C16B	120.60(15)
C14B-C15B-H15B	119.7
C16B-C15B-H15B	119.7
C15A-C16A-C11A	121.17(14)
C15A-C16A-H16A	119.4
C11A-C16A-H16A	119.4
C15B-C16B-C11B	121.02(14)
C15B-C16B-H16B	119.5
C11B-C16B-H16B	119.5
C18A-C17A-N25A	107.56(11)
C18A-C17A-C10A	114.65(12)
N25A-C17A-C10A	110.41(11)
C18A-C17A-H17A	108.0
N25A-C17A-H17A	108.0
C10A-C17A-H17A	108.0
C18B-C17B-N25B	107.86(11)
C18B-C17B-C10B	114.45(12)
N25B-C17B-C10B	110.54(11)
C18B-C17B-H17B	107.9
N25B-C17B-H17B	107.9
C10B-C17B-H17B	107.9
C23A-C18A-C19A	120.39(14)
C23A-C18A-C17A	120.58(13)
C19A-C18A-C17A	118.96(14)
C23B-C18B-C19B	119.91(14)
C23B-C18B-C17B	120.66(13)
C19B-C18B-C17B	119.38(14)
C20A-C19A-C18A	120.14(16)
C20A-C19A-H19A	119.9
C18A-C19A-H19A	119.9
C20B-C19B-C18B	120.24(15)
C20B-C19B-H19B	119.9

C18B-C19B-H19B	119.9
C19A-C20A-C21A	119.66(16)
C19A-C20A-H20A	120.2
C21A-C20A-H20A	120.2
C19B-C20B-C21B	120.03(15)
C19B-C20B-H20B	120.0
C21B-C20B-H20B	120.0
C22A-C21A-C20A	120.34(15)
C22A-C21A-H21A	119.8
C20A-C21A-H21A	119.8
C20B-C21B-C22B	119.90(15)
C20B-C21B-H21B	120.1
C22B-C21B-H21B	120.1
C21A-C22A-C23A	120.55(16)
C21A-C22A-H22A	119.7
C23A-C22A-H22A	119.7
C21B-C22B-C23B	120.70(15)
C21B-C22B-H22B	119.6
C23B-C22B-H22B	119.6
C18A-C23A-C22A	118.91(15)
C18A-C23A-C24A	120.93(13)
C22A-C23A-C24A	120.15(15)
C18B-C23B-C22B	119.21(14)
C18B-C23B-C24B	120.78(13)
C22B-C23B-C24B	119.97(14)
N1A-C24A-C23A	110.91(12)
N1A-C24A-H24A	109.5
C23A-C24A-H24A	109.5
N1A-C24A-H24B	109.5
C23A-C24A-H24B	109.5
H24A-C24A-H24B	108.0
N1B-C24B-C23B	111.25(12)
N1B-C24B-H24C	109.4
C23B-C24B-H24C	109.4
N1B-C24B-H24D	109.4
C23B-C24B-H24D	109.4

H24C-C24B-H24D 108.0

Symmetry transformations used to generate equivalent atoms:

Supplementary Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for rick1. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O26A	25(1)	20(1)	27(1)	-1(1)	-3(1)	-3(1)
O26B	32(1)	20(1)	24(1)	1(1)	-1(1)	-7(1)
O27A	30(1)	36(1)	25(1)	-10(1)	5(1)	-2(1)
O27B	36(1)	34(1)	34(1)	-16(1)	13(1)	-5(1)
N1A	17(1)	18(1)	20(1)	1(1)	3(1)	1(1)
N1B	14(1)	18(1)	17(1)	1(1)	2(1)	2(1)
N25A	17(1)	21(1)	21(1)	-2(1)	-3(1)	2(1)
N25B	20(1)	18(1)	19(1)	-2(1)	-1(1)	1(1)
C2A	25(1)	23(1)	19(1)	2(1)	6(1)	0(1)
C2B	19(1)	20(1)	16(1)	0(1)	2(1)	1(1)
C3A	29(1)	21(1)	25(1)	7(1)	6(1)	1(1)
C3B	21(1)	17(1)	20(1)	2(1)	2(1)	0(1)
C4A	22(1)	18(1)	18(1)	1(1)	-1(1)	0(1)
C4B	18(1)	16(1)	17(1)	0(1)	-2(1)	0(1)
C5A	30(1)	19(1)	23(1)	3(1)	-4(1)	3(1)
C5B	22(1)	16(1)	22(1)	0(1)	-3(1)	2(1)
C6A	25(1)	23(1)	27(1)	1(1)	-5(1)	6(1)
C6B	21(1)	19(1)	27(1)	-4(1)	-5(1)	5(1)
C7A	19(1)	26(1)	25(1)	-1(1)	-1(1)	3(1)
C7B	16(1)	22(1)	28(1)	-4(1)	0(1)	3(1)
C8A	17(1)	20(1)	21(1)	2(1)	-1(1)	0(1)
C8B	17(1)	18(1)	24(1)	0(1)	1(1)	0(1)
C9A	18(1)	17(1)	16(1)	0(1)	-2(1)	1(1)
C9B	14(1)	15(1)	18(1)	-2(1)	-1(1)	1(1)
C10A	16(1)	16(1)	16(1)	1(1)	0(1)	0(1)
C10B	14(1)	15(1)	16(1)	0(1)	0(1)	2(1)

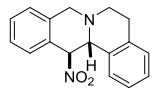
C11A	16(1)	16(1)	18(1)	1(1)	-1(1)	-1(1)
C11B	14(1)	17(1)	19(1)	-2(1)	0(1)	1(1)
C12A	25(1)	20(1)	18(1)	2(1)	-3(1)	-2(1)
C12B	22(1)	20(1)	21(1)	-1(1)	-2(1)	-2(1)
C13A	34(1)	26(1)	19(1)	0(1)	-7(1)	-5(1)
C13B	23(1)	25(1)	24(1)	-5(1)	-3(1)	-5(1)
C14A	37(1)	21(1)	28(1)	-3(1)	-7(1)	-6(1)
C14B	23(1)	20(1)	32(1)	-8(1)	-1(1)	-2(1)
C15A	29(1)	18(1)	32(1)	1(1)	-7(1)	-2(1)
C15B	24(1)	16(1)	37(1)	-1(1)	-4(1)	2(1)
C16A	22(1)	18(1)	24(1)	2(1)	-6(1)	-1(1)
C16B	20(1)	17(1)	27(1)	0(1)	-4(1)	1(1)
C17A	17(1)	17(1)	17(1)	0(1)	-2(1)	0(1)
C17B	16(1)	15(1)	17(1)	-1(1)	0(1)	0(1)
C18A	18(1)	17(1)	23(1)	-1(1)	-4(1)	-1(1)
C18B	18(1)	16(1)	19(1)	1(1)	-3(1)	-1(1)
C19A	24(1)	21(1)	24(1)	1(1)	-7(1)	-1(1)
C19B	24(1)	21(1)	20(1)	1(1)	-4(1)	-1(1)
C20A	28(1)	21(1)	32(1)	3(1)	-12(1)	-2(1)
C20B	30(1)	24(1)	25(1)	3(1)	-12(1)	-1(1)
C21A	20(1)	18(1)	45(1)	4(1)	-10(1)	1(1)
C21B	24(1)	21(1)	34(1)	1(1)	-11(1)	2(1)
C22A	18(1)	20(1)	38(1)	1(1)	-1(1)	0(1)
C22B	17(1)	19(1)	30(1)	-1(1)	-4(1)	2(1)
C23A	16(1)	18(1)	27(1)	0(1)	-2(1)	-1(1)
C23B	16(1)	17(1)	21(1)	-1(1)	-2(1)	0(1)
C24A	18(1)	22(1)	26(1)	-1(1)	3(1)	2(1)
C24B	16(1)	21(1)	20(1)	-1(1)	0(1)	3(1)

Supplementary Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for rick1.

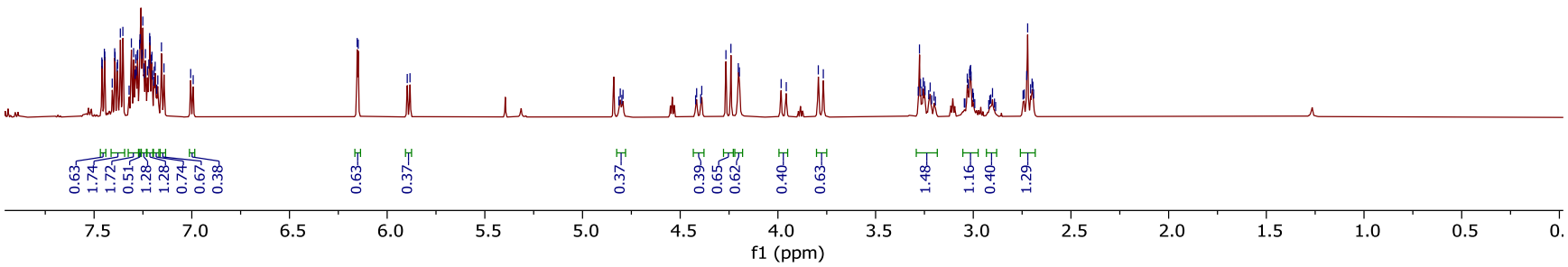
	x	y	z	U(eq)
H2AA	1043	4181	8598	27
H2AB	2984	4543	8673	27
H2BA	6654	5284	4156	22
H2BB	8557	5678	4240	22
H3AA	3205	3153	8680	30
H3AB	2210	3097	8181	30
H3BA	7285	6800	4579	23
H3BB	6314	6652	4083	23
H5AA	5839	2468	8374	29
H5BA	3676	7375	4358	24
H6AA	8566	2579	8001	30
H6BA	929	7299	4723	27
H7AA	9160	3675	7516	28
H7BA	332	6271	5259	26
H8AA	7002	4645	7409	23
H8BA	2503	5327	5416	23
H12A	5918	5043	8519	25
H12B	3804	4824	4287	25
H13A	7114	6173	8865	31
H13B	2593	3674	3966	29
H14A	6546	7424	8540	35
H14B	2894	2465	4364	30
H15A	4831	7525	7854	32
H15B	4401	2423	5085	31
H16A	3649	6396	7504	26
H16B	5626	3571	5406	25
H17A	4389	5242	7094	20
H17B	5041	4735	5748	19
H19A	2214	5566	6435	28
H19B	7136	4427	6428	26

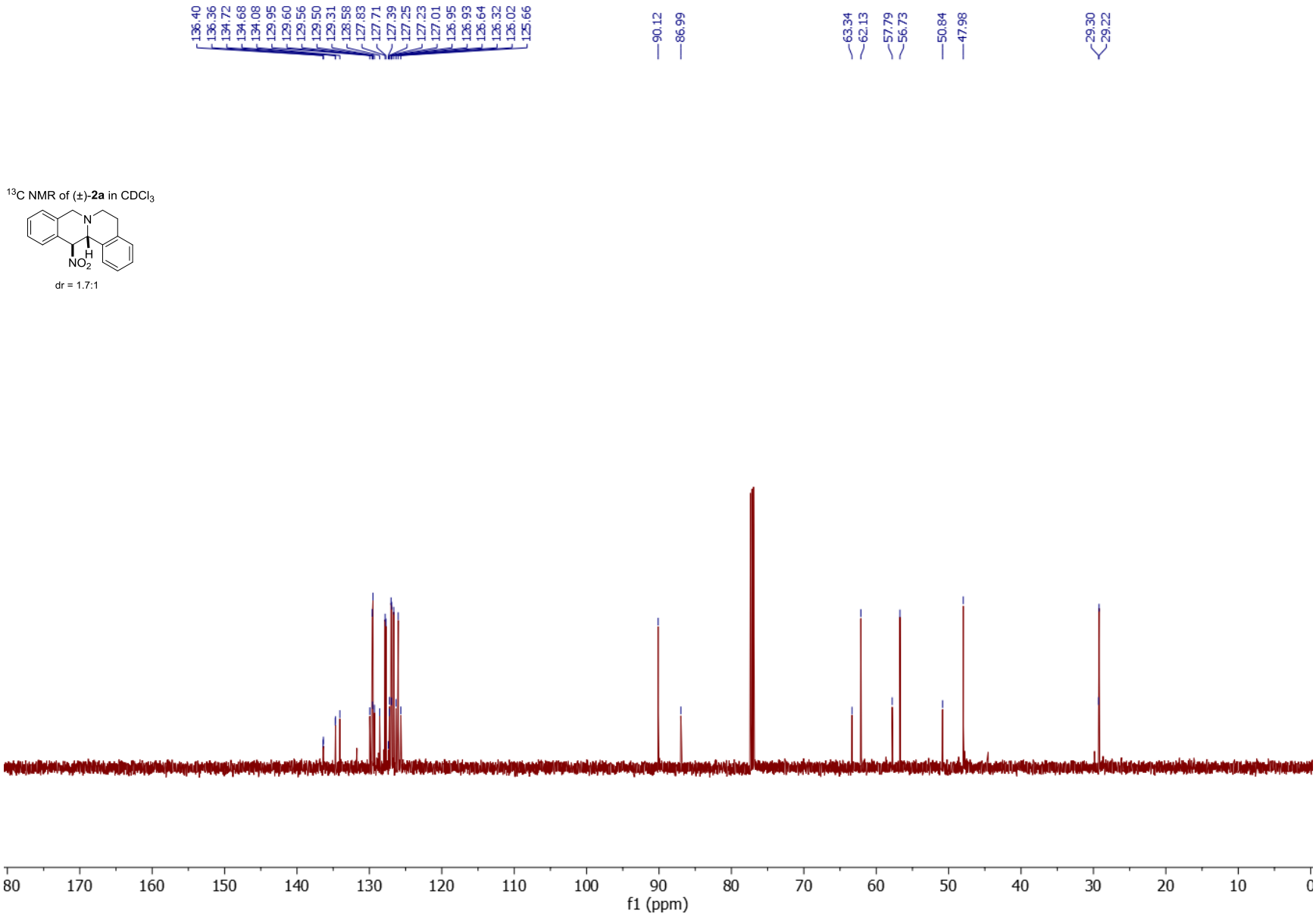
H20A	-317	6310	6269	32
H20B	9620	3674	6620	32
H21A	-2314	6603	6876	34
H21B	11739	3394	6041	31
H22A	-1786	6162	7643	30
H22B	11319	3827	5265	26
H24A	1292	5726	8191	26
H24B	-293	5104	8137	26
H24C	9815	4794	4733	22
H24D	8227	4172	4692	22

^{1}H NMR of (±)-**2a** in $CDCl_3$

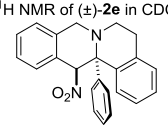


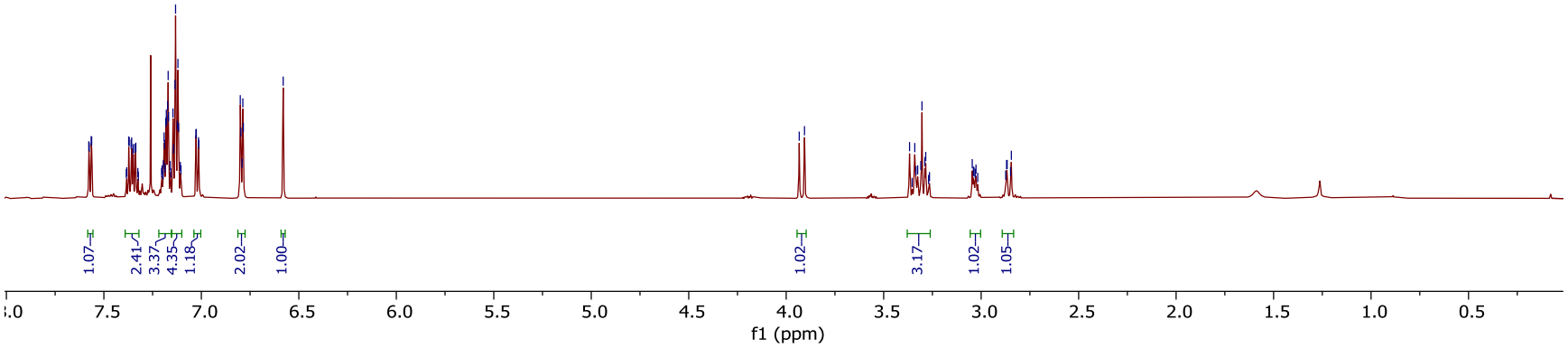
dr = 1.7:1

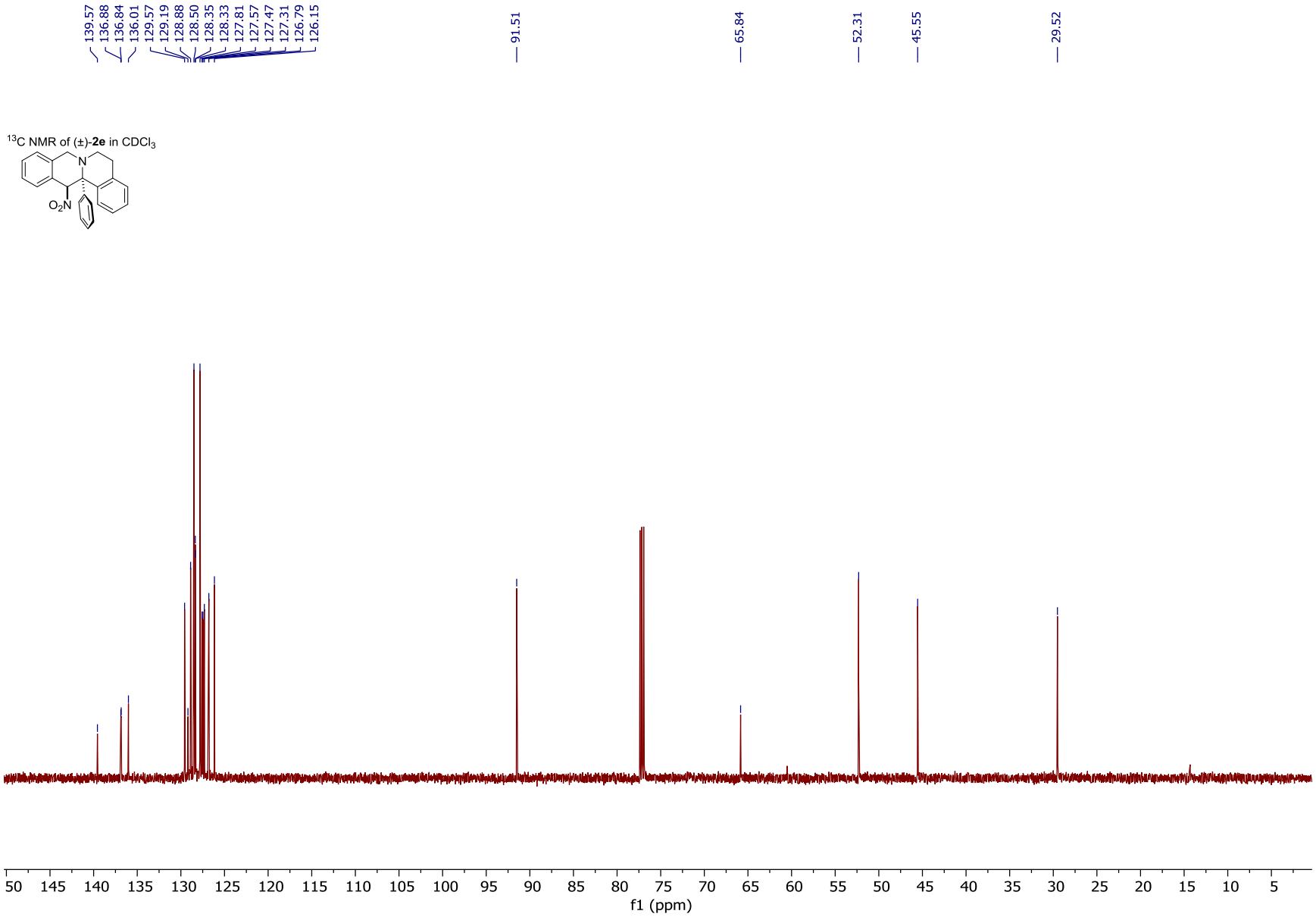


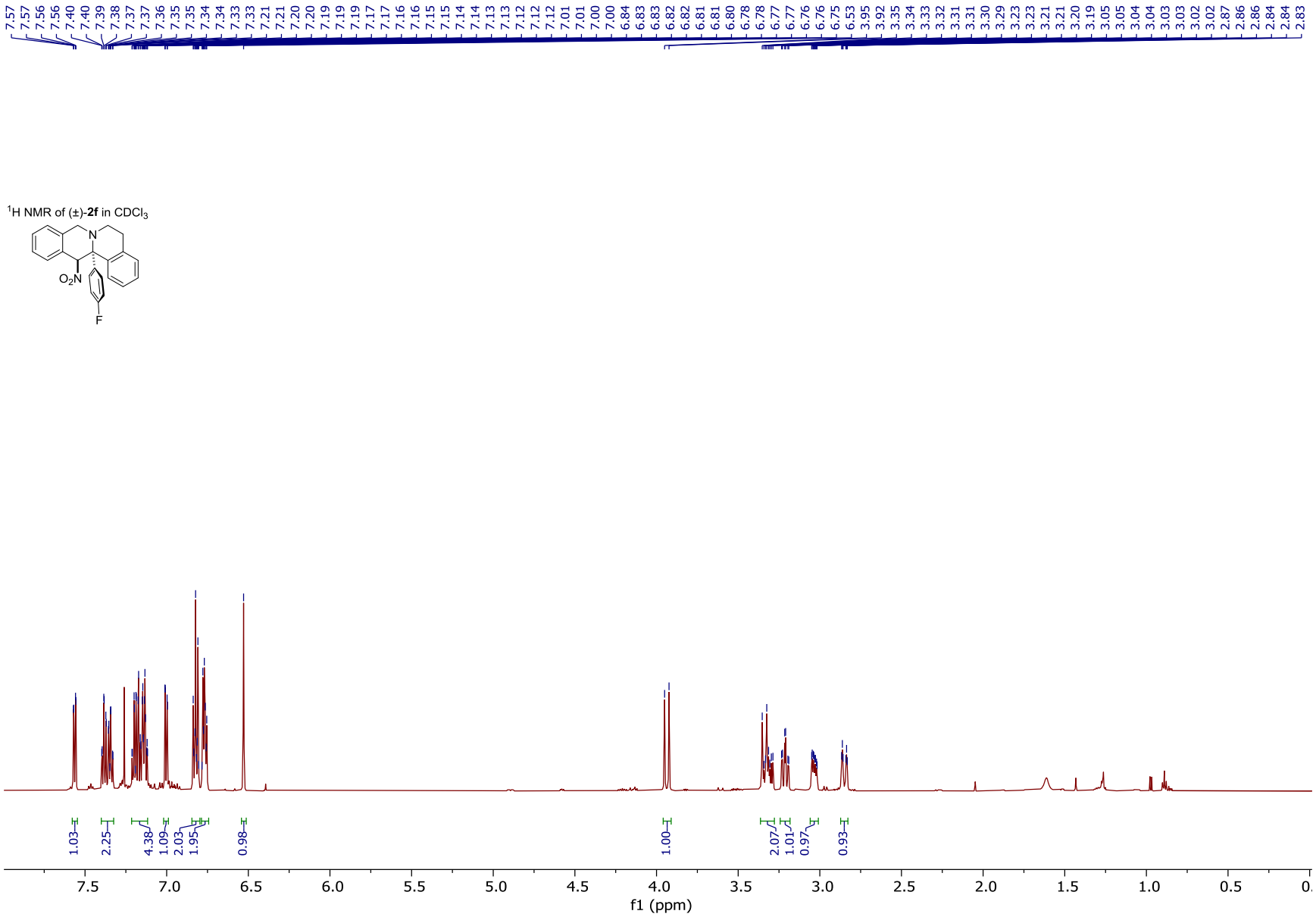

136.40
136.36
134.72
134.68
134.08
129.95
129.60
129.56
129.50
129.31
128.58
127.83
127.71
127.39
127.25
127.23
127.01
126.95
126.93
126.64
126.32
126.02
125.66
90.12
86.99
63.34
62.13
57.79
56.73
50.84
47.98
29.30
29.22
13C NMR of (±)-2a in CDCl3
N
H
NO2
dr = 1.7:1
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

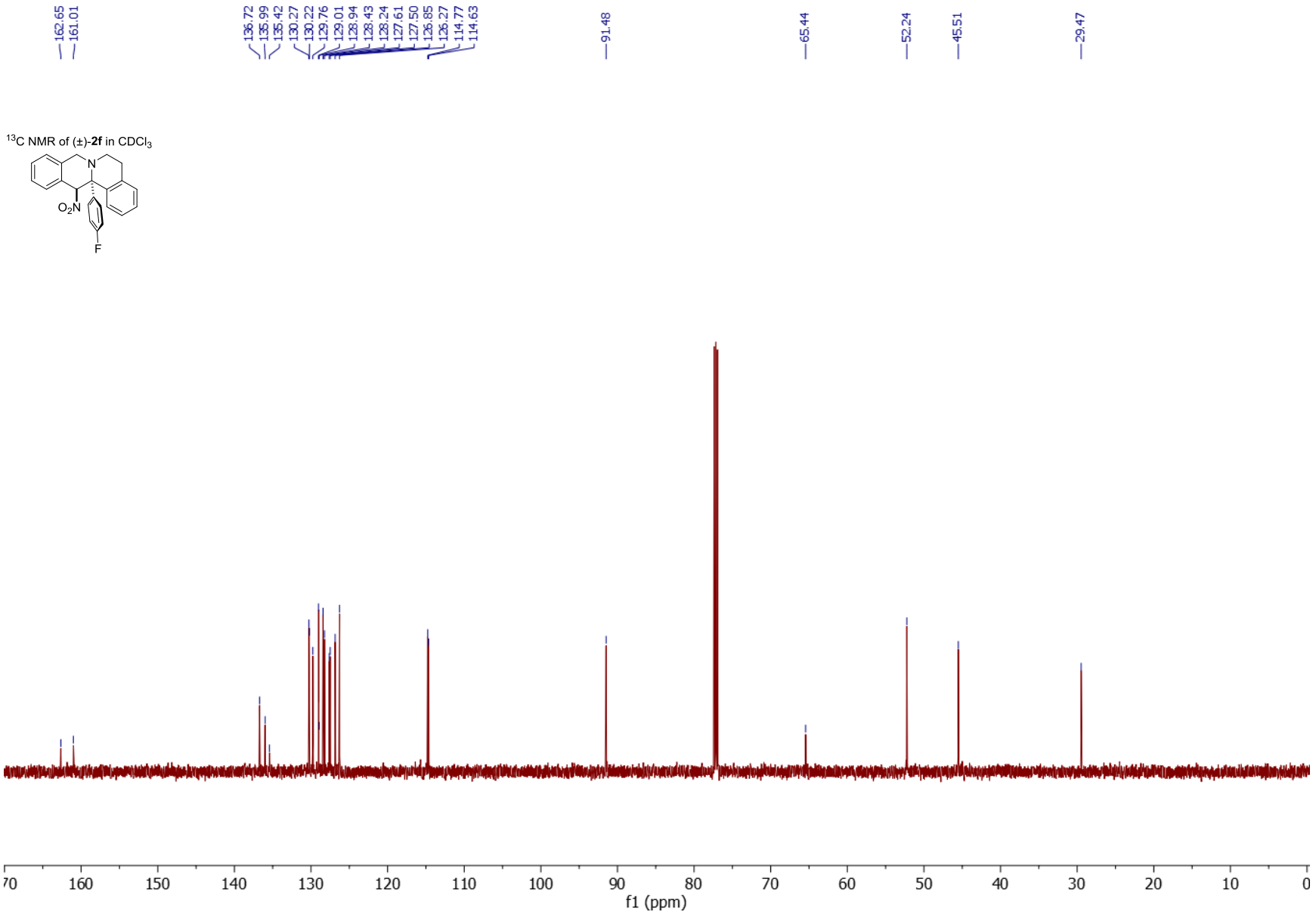
^{1}H NMR of (±)-**2e** in $CDCl_3$



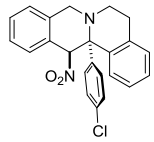


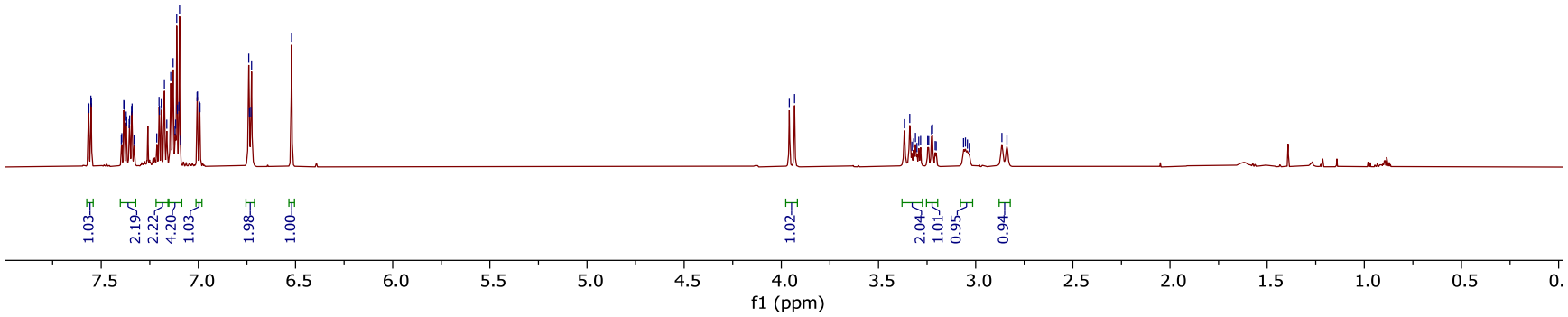

139.57
136.88
136.84
136.01
129.57
129.19
128.88
128.50
128.35
128.33
127.81
127.57
127.47
127.31
126.79
126.15
91.51
65.84
52.31
45.55
29.52
13C NMR of (±)-2e in CDCl3
O2N
50 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)

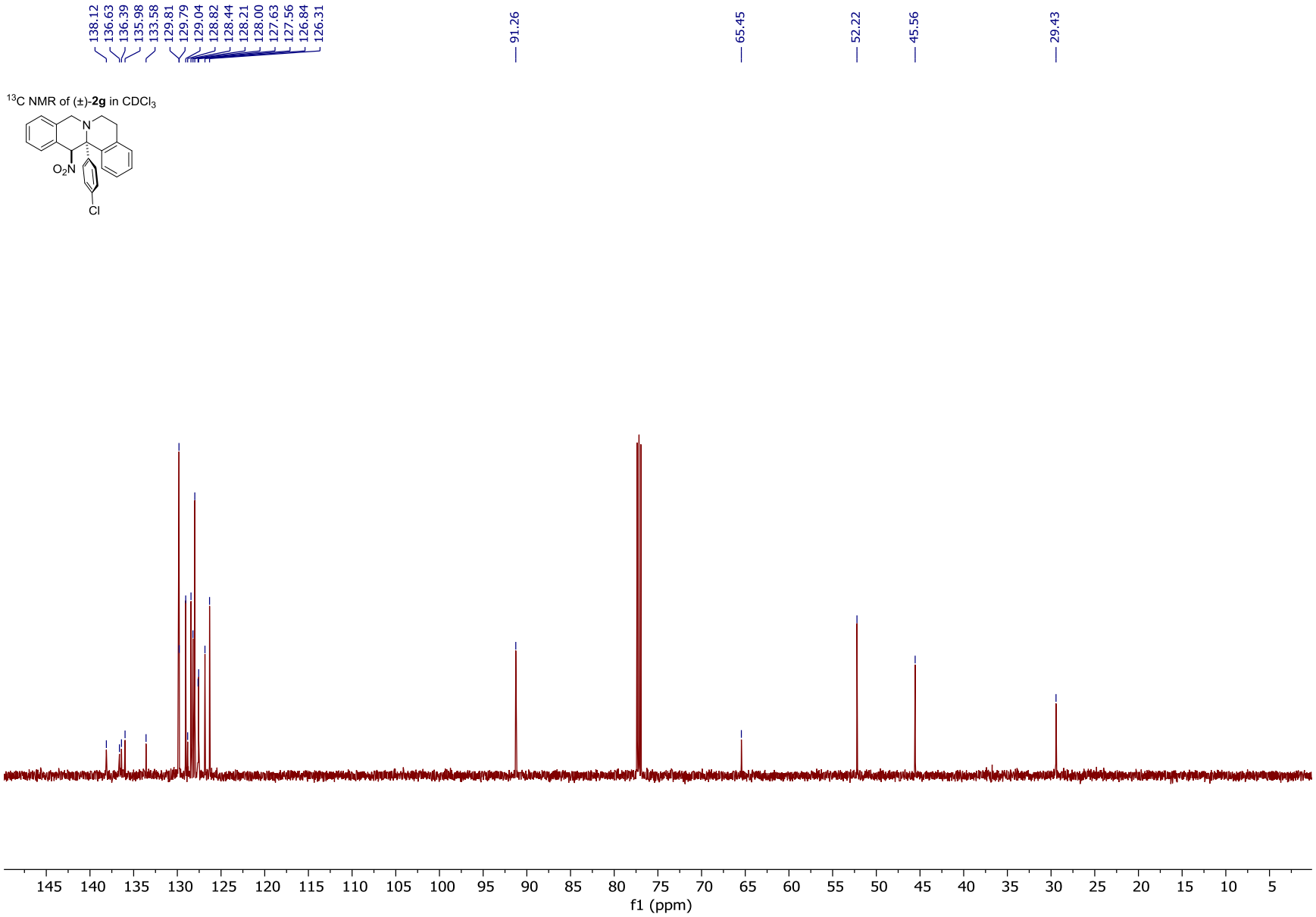

7.57 7.57 7.56 7.56 7.40 7.40 7.39 7.38 7.37 7.37 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.21 7.21 7.20 7.20 7.19 7.19 7.19 7.17 7.17 7.16 7.16 7.15 7.15 7.14 7.14 7.13 7.13 7.12 7.12 7.12 7.01 7.01 7.00 7.00 6.84 6.83 6.83 6.82 6.82 6.81 6.81 6.80 6.78 6.78 6.77 6.77 6.76 6.76 6.75 6.53 3.95 3.92 3.35 3.34 3.33 3.32 3.31 3.31 3.30 3.29 3.23 3.23 3.21 3.21 3.20 3.19 3.05 3.05 3.04 3.04 3.03 3.03 3.02 3.02 2.87 2.86 2.86 2.84 2.84 2.83
^{1}H NMR of (±)-**2f** in $CDCl_3$
N
O_2N
F
1.03 2.25 4.38 1.09 2.03 1.95 0.98 1.00 2.07 1.01 0.97 0.93
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


162.65
161.01
136.72
135.99
135.42
130.27
130.22
129.76
129.01
128.94
128.43
128.24
127.61
127.50
126.85
126.27
114.77
114.63
91.48
65.44
52.24
45.51
29.47
13C NMR of (±)-2f in CDCl3
O2N
F
f1 (ppm)

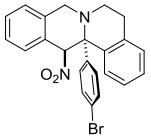
^{1}H NMR of (±)-**2g** in $CDCl_3$

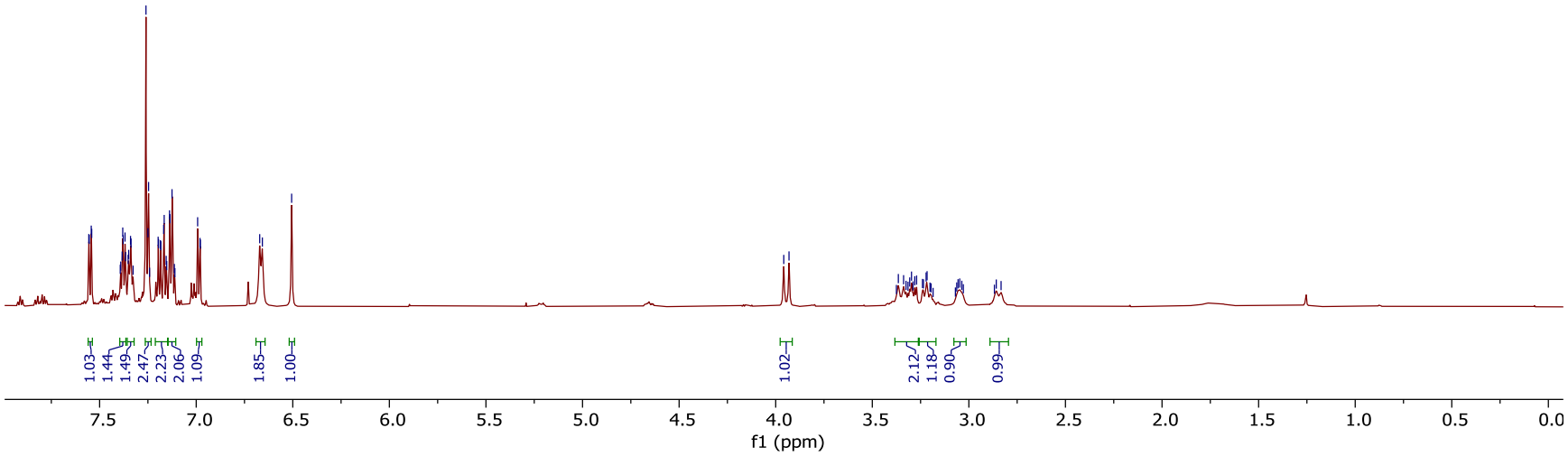


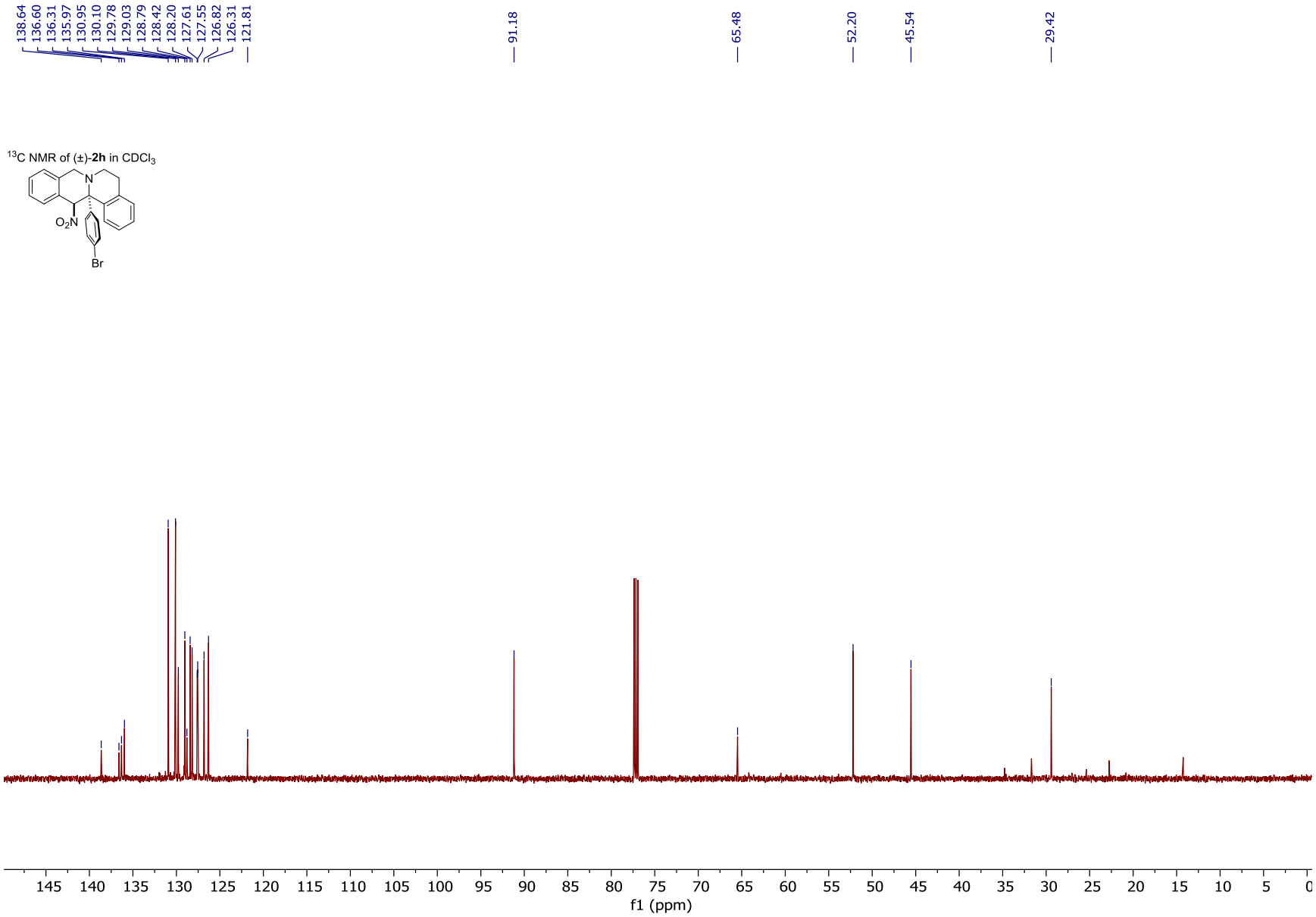



138.12
136.63
136.39
135.98
133.58
129.81
129.79
129.04
128.82
128.44
128.21
128.00
127.63
127.56
126.84
126.31
91.26
65.45
52.22
45.56
29.43
^{13}C NMR of (±)-**2g** in $CDCl_3$
N
O_2N
Cl
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)

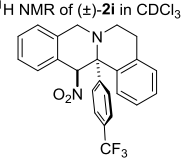
^{1}H NMR of (±)-**2h** in $CDCl_3$

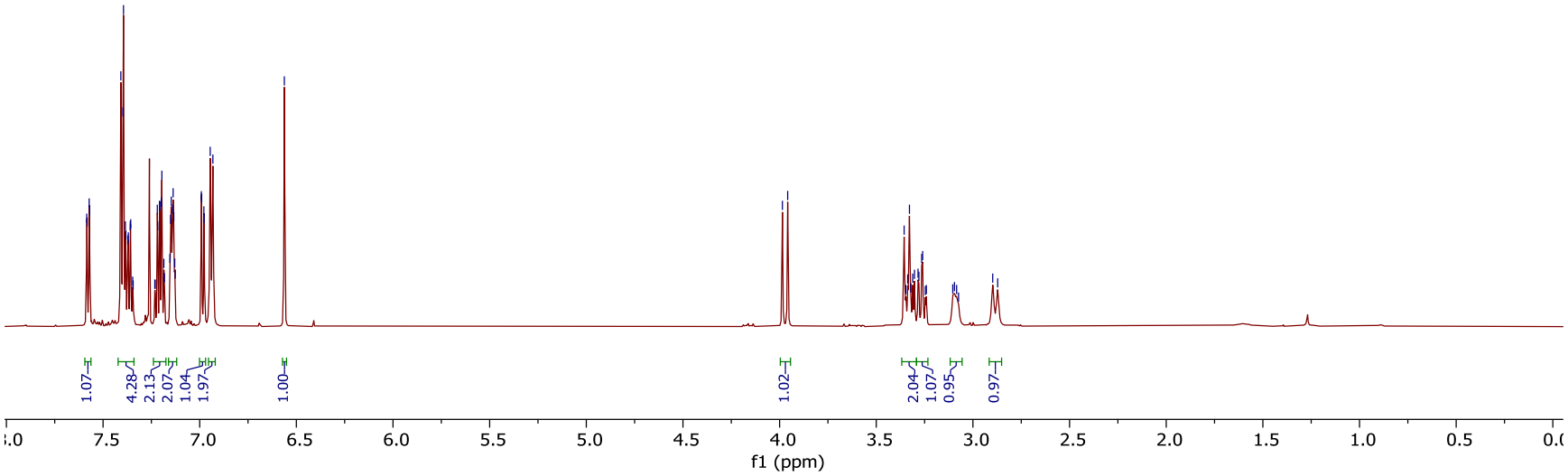


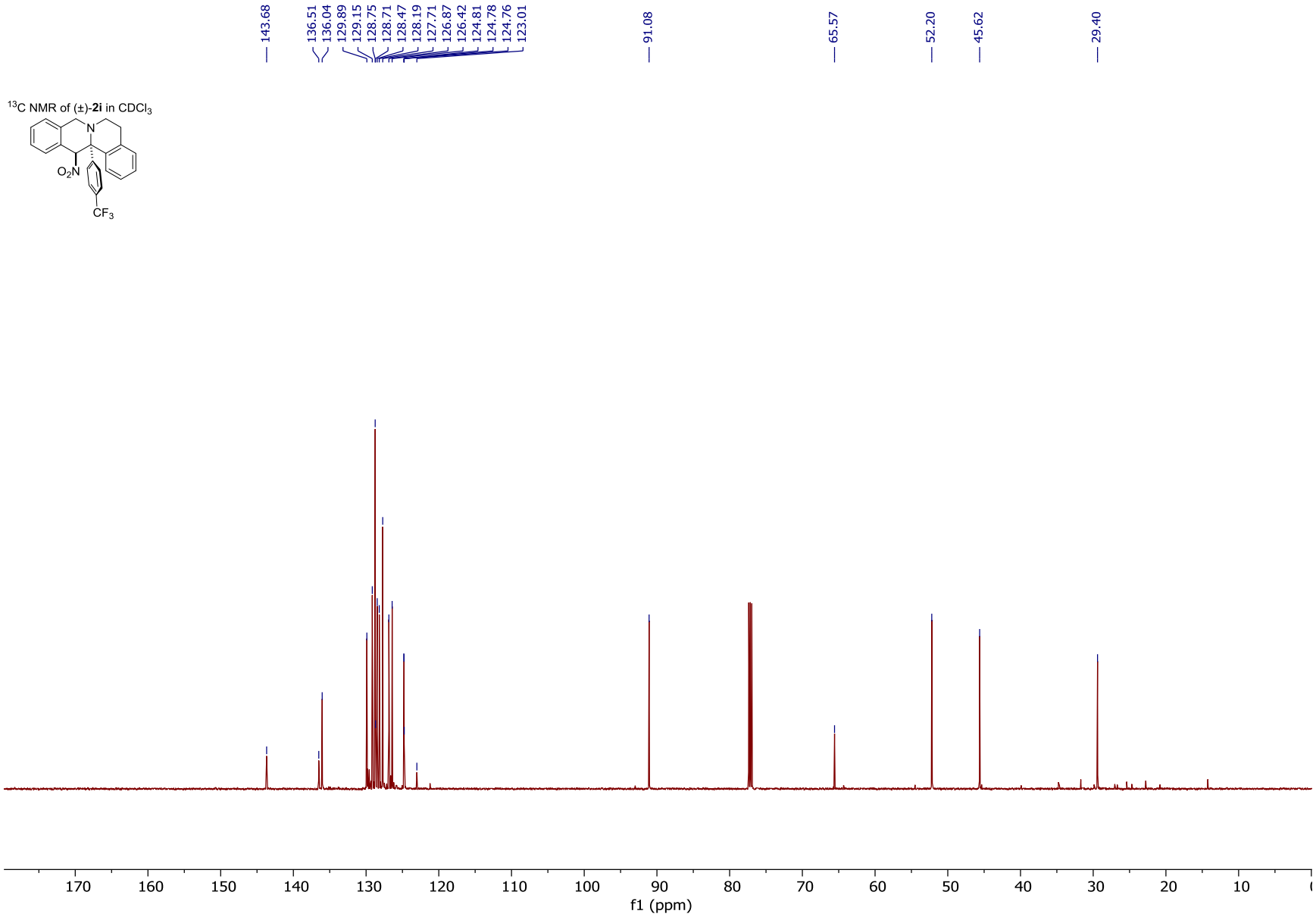



138.64
136.60
136.31
135.97
130.95
130.10
129.78
129.03
128.79
128.42
128.20
127.61
127.55
126.82
126.31
121.81
91.18
65.48
52.20
45.54
29.42
13C NMR of (±)-2h in CDCl3
N
O2N
Br
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

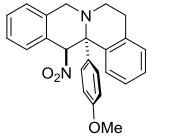
^{1}H NMR of (±)-**2i** in $CDCl_3$

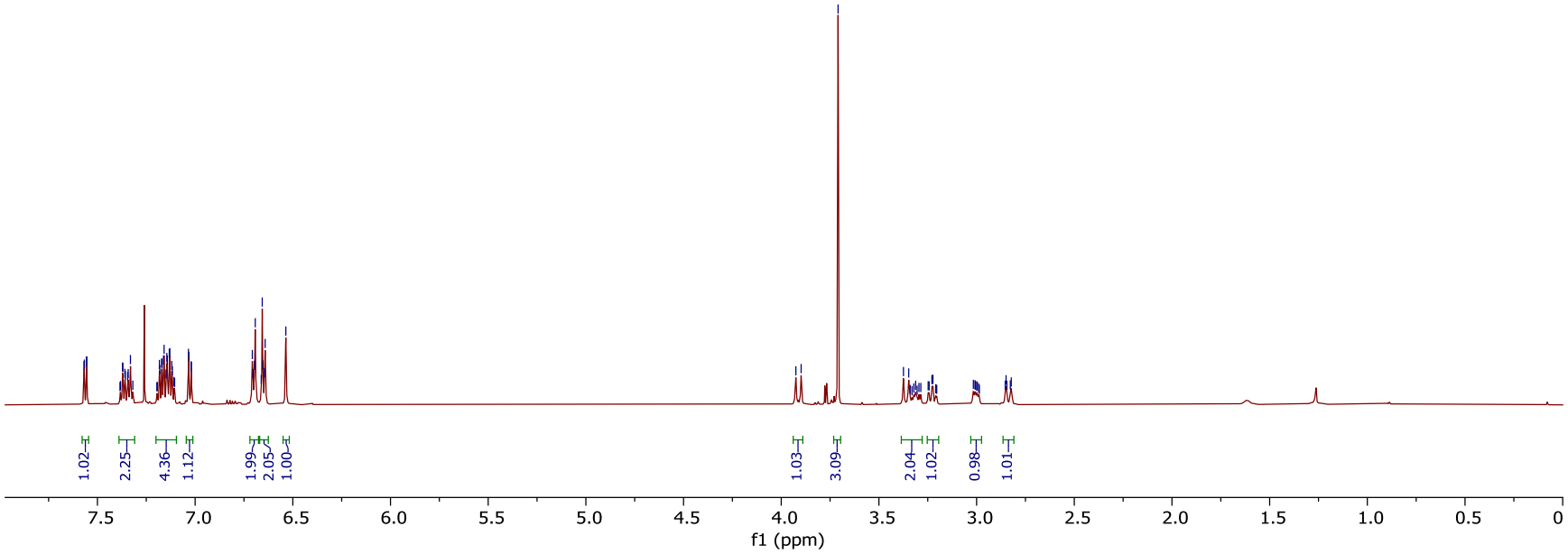


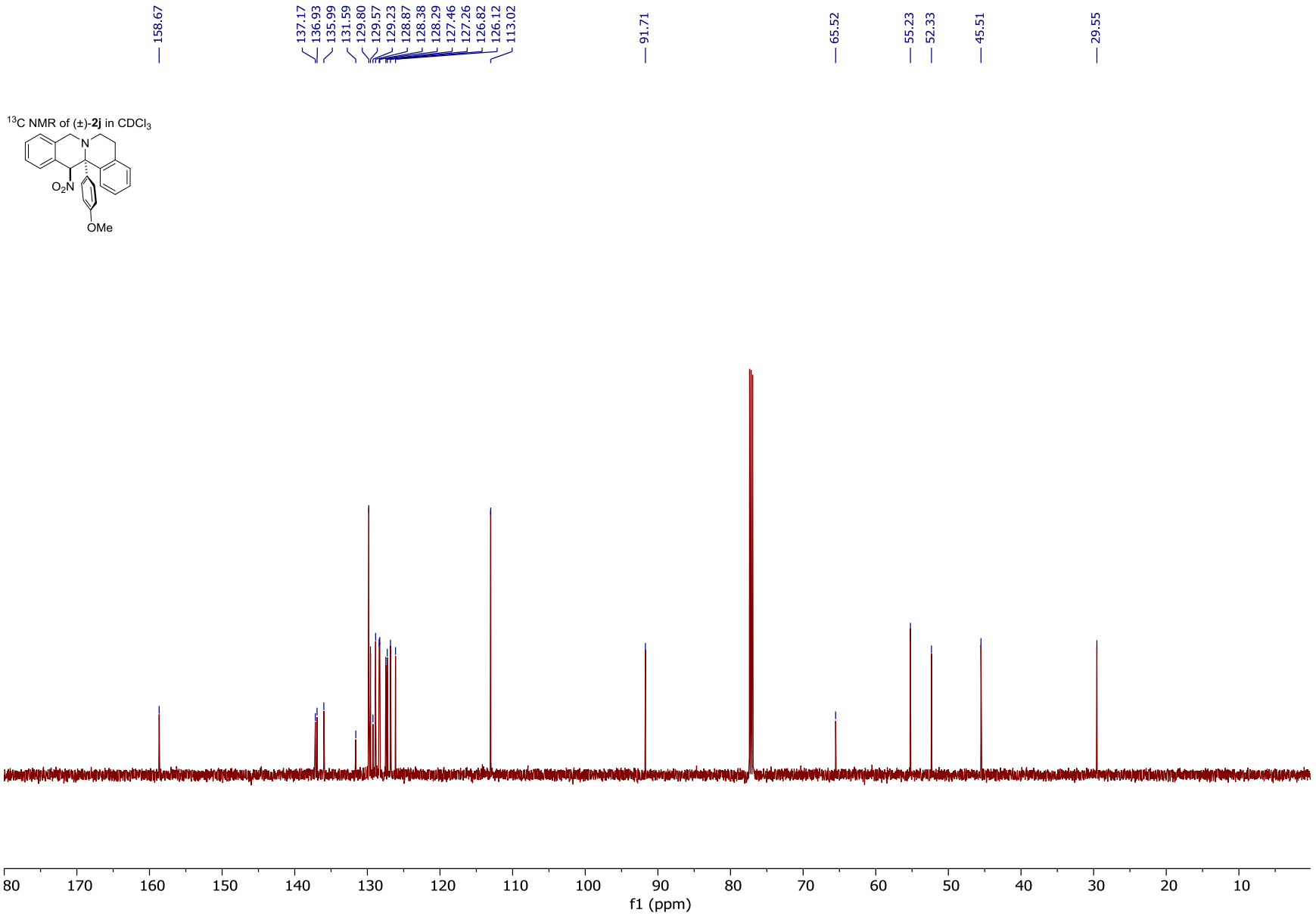



143.68
136.51
136.04
129.89
129.15
128.75
128.71
128.47
128.19
127.71
126.87
126.42
124.81
124.78
124.76
123.01
91.08
65.57
52.20
45.62
29.40
13C NMR of (±)-2i in CDCl3
O2N
CF3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

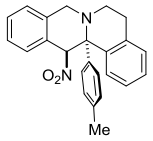
^{1}H NMR of (±)-**2j** in $CDCl_3$

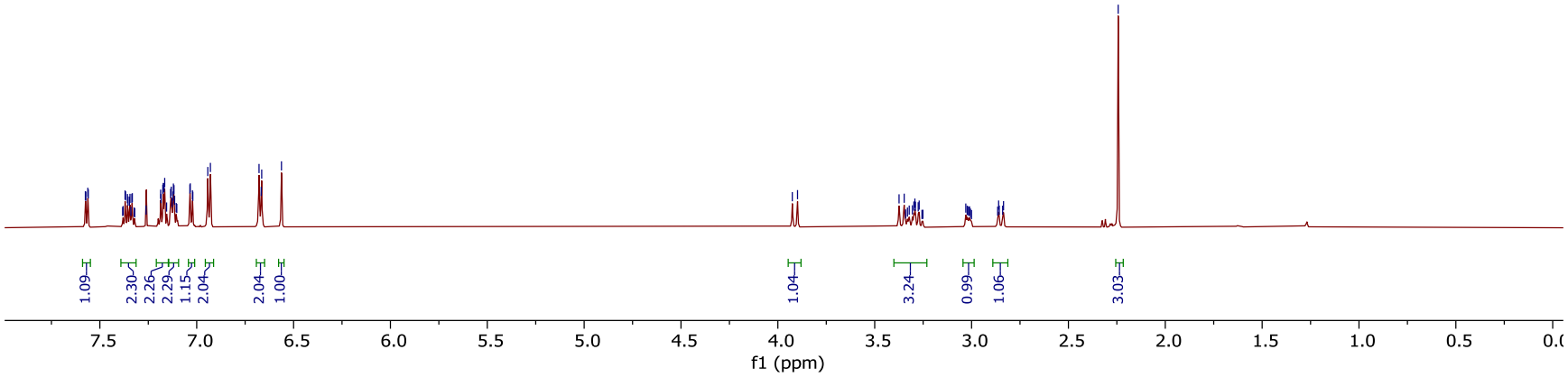




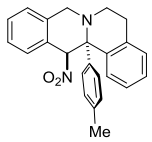

158.67
137.17
136.93
135.99
131.59
129.80
129.57
129.23
128.87
128.38
128.29
127.46
127.26
126.82
126.12
113.02
91.71
65.52
55.23
52.33
45.51
29.55
13C NMR of (±)-2j in CDCl3
O2N
OMe
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

^{1}H NMR of (±)-**2k** in $CDCl_3$

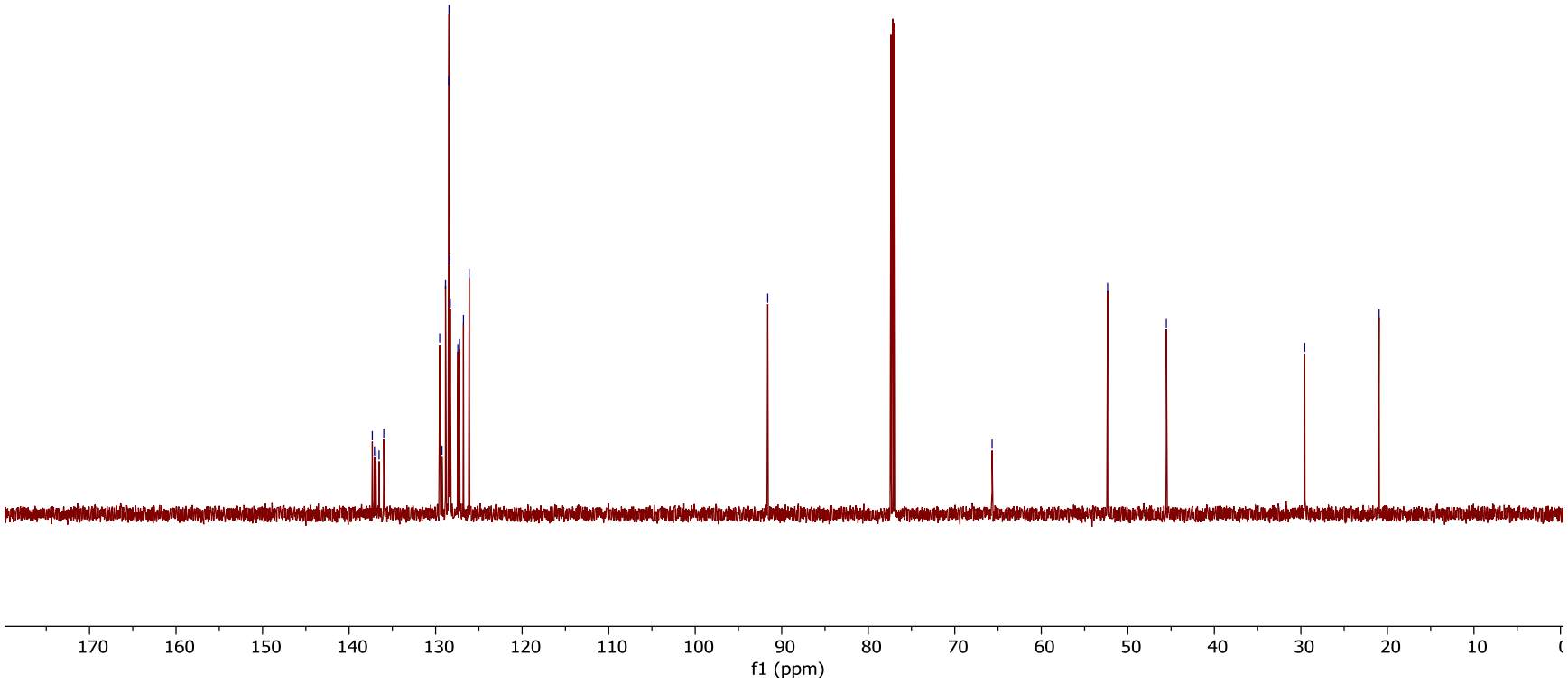




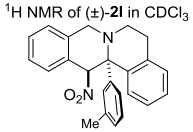
^{13}C NMR of (±)-**2k** in $CDCl_3$

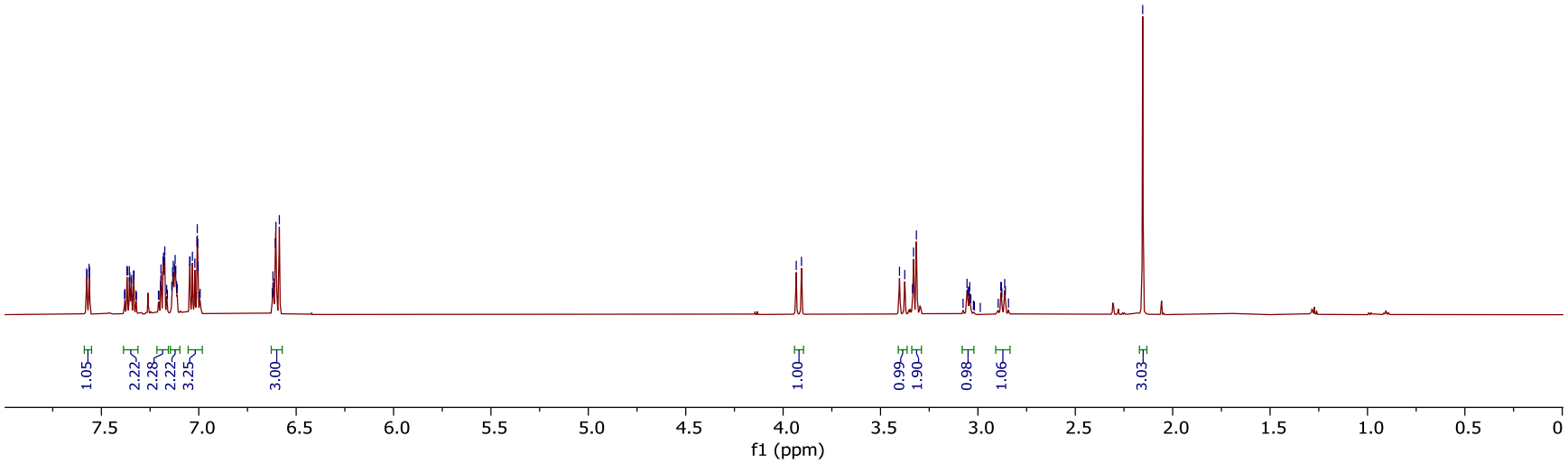


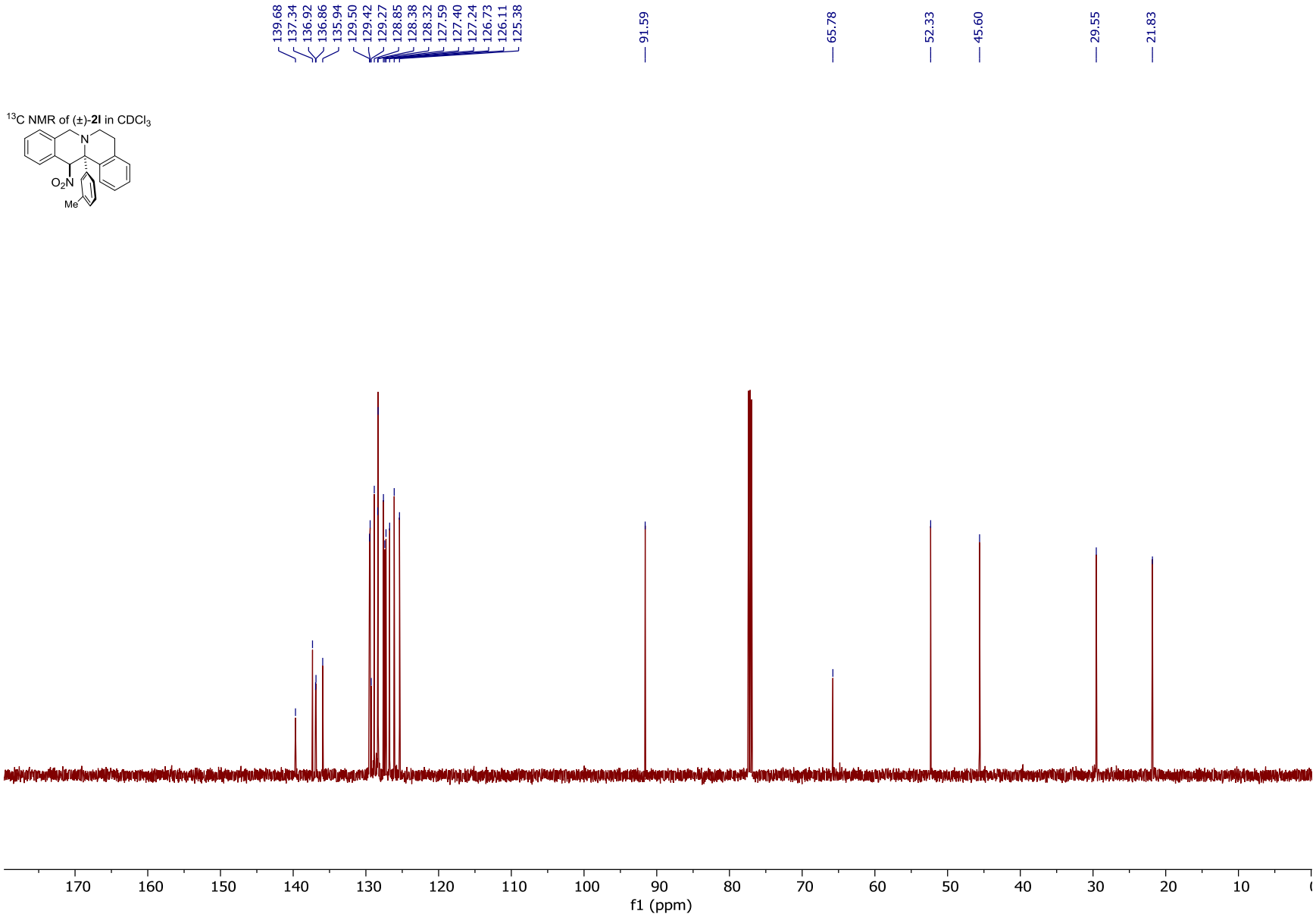
137.32
137.07
136.90
136.54
135.99
129.53
129.26
128.86
128.49
128.46
128.36
128.31
127.43
127.24
126.78
126.13
91.62
65.69
52.34
45.55
29.55
20.96



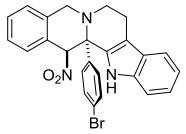
^{1}H NMR of (±)-**2l** in $CDCl_3$

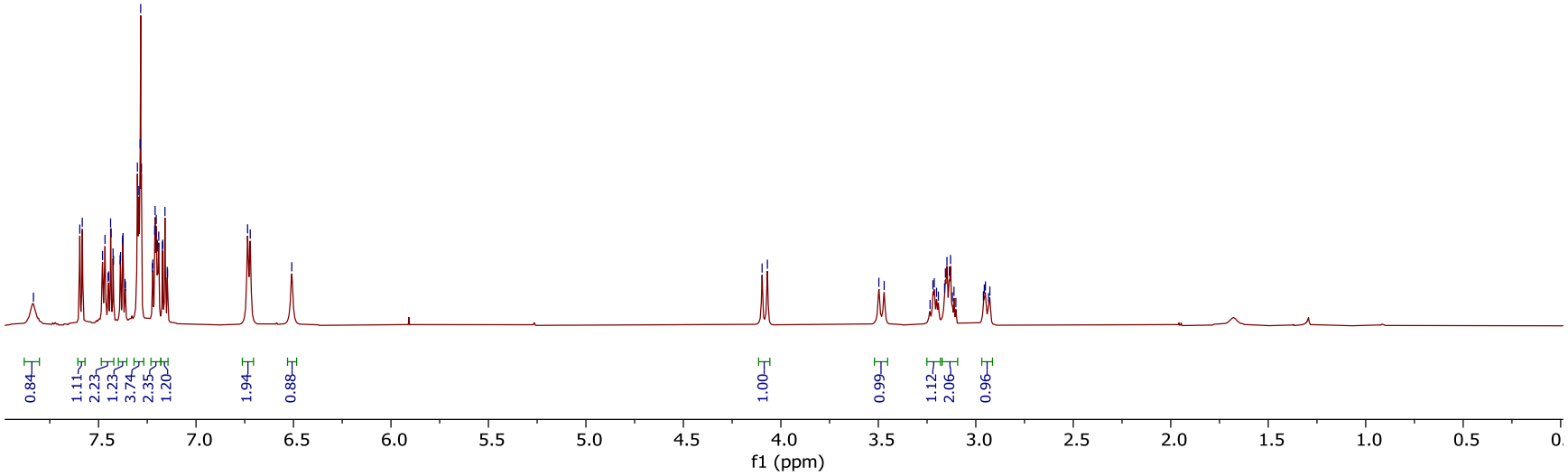


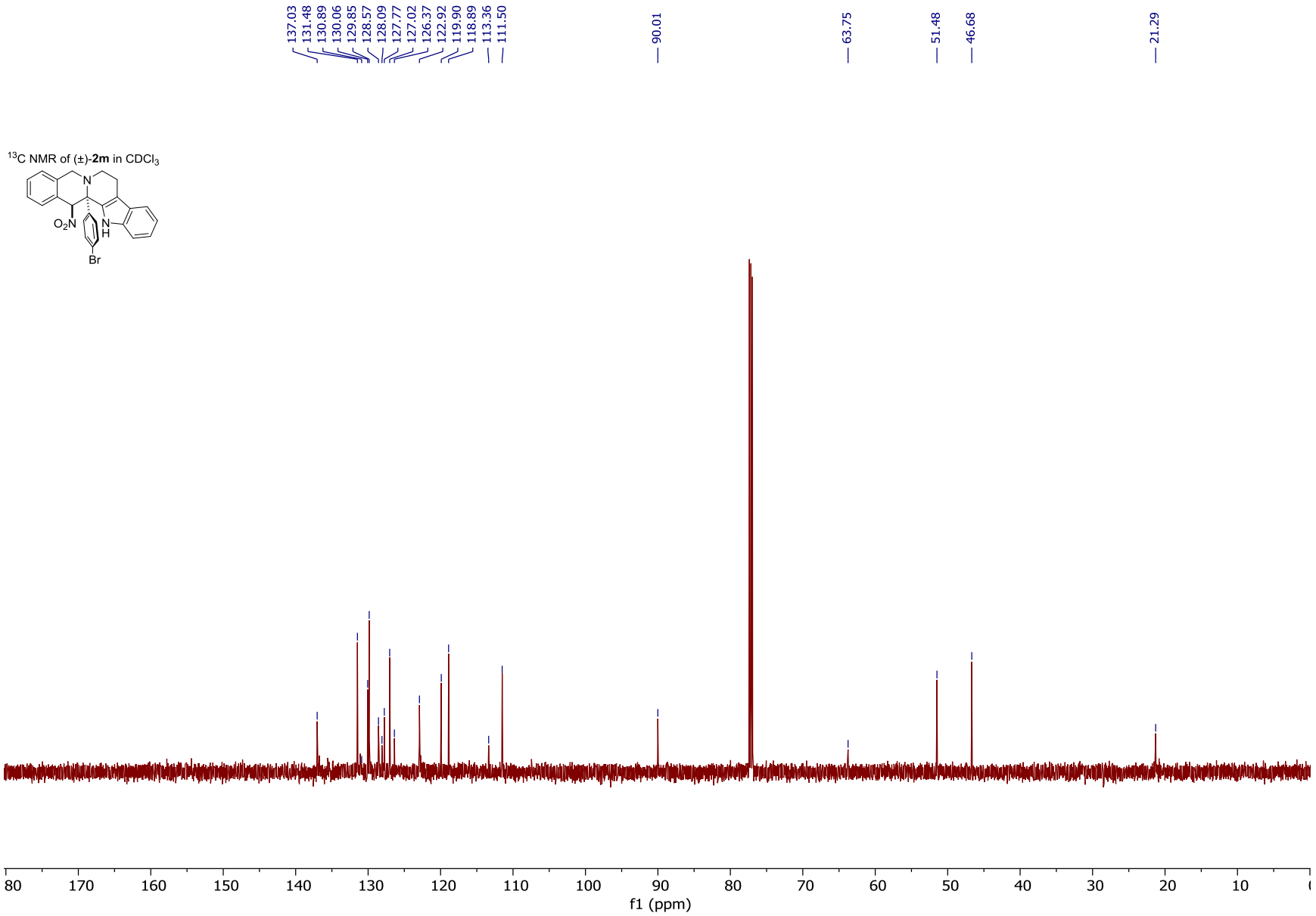



139.68
137.34
136.92
136.86
135.94
129.50
129.42
129.27
128.85
128.38
128.32
127.59
127.40
127.24
126.73
126.11
125.38
91.59
65.78
52.33
45.60
29.55
21.83
13C NMR of (±)-2l in CDCl3
N
O2N
Me
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

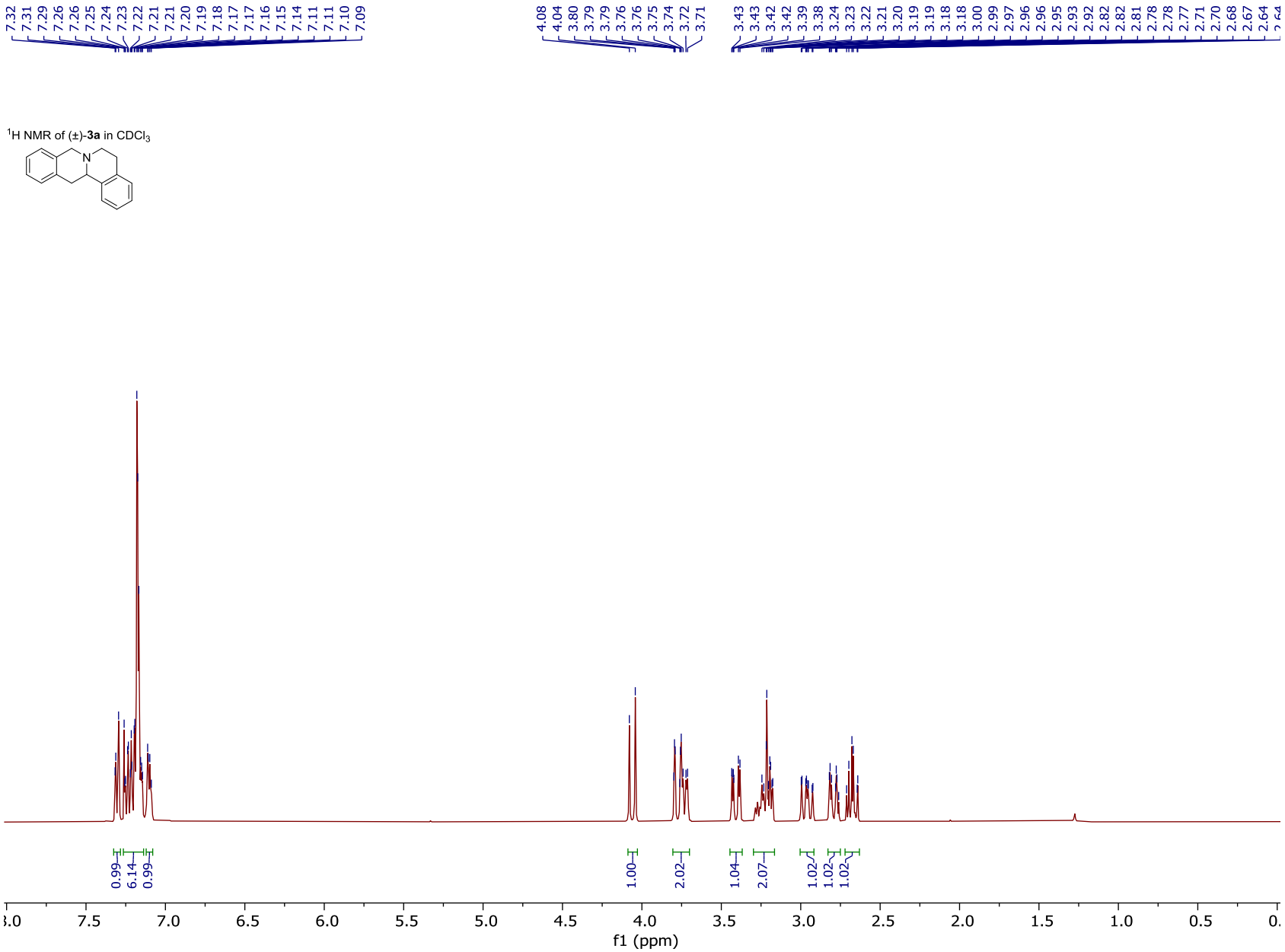
^{1}H NMR of (±)-**2m** in $CDCl_3$

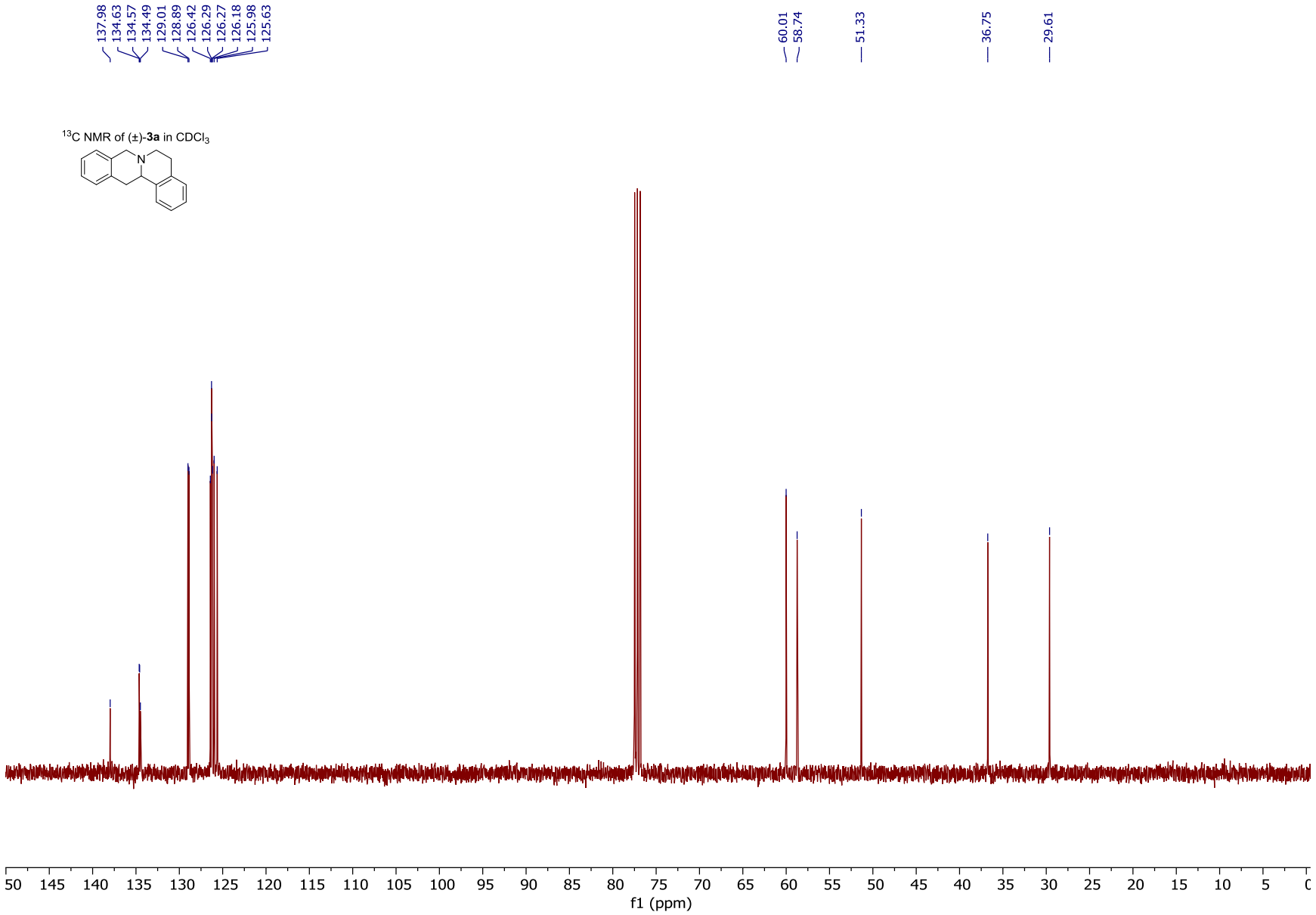


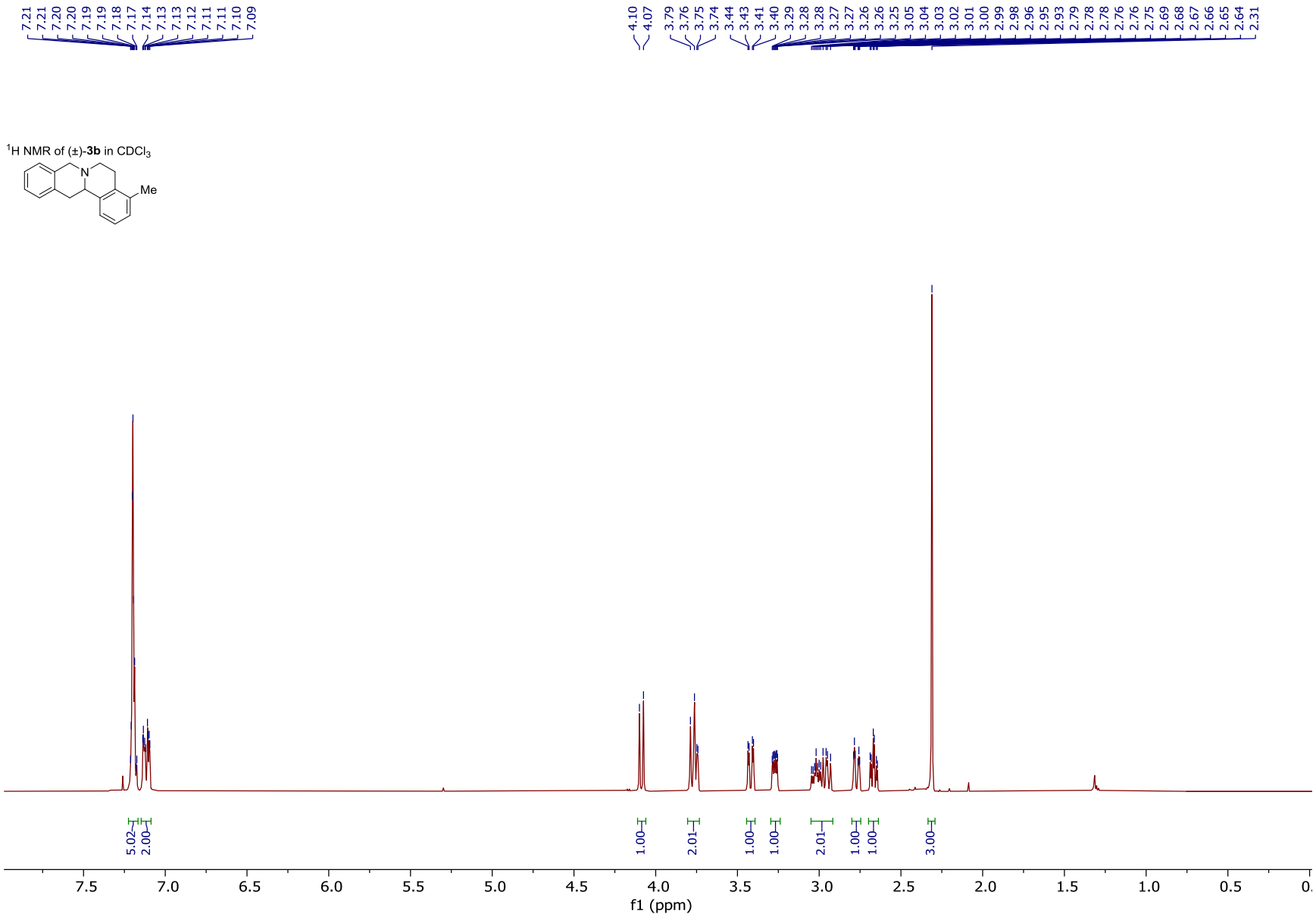


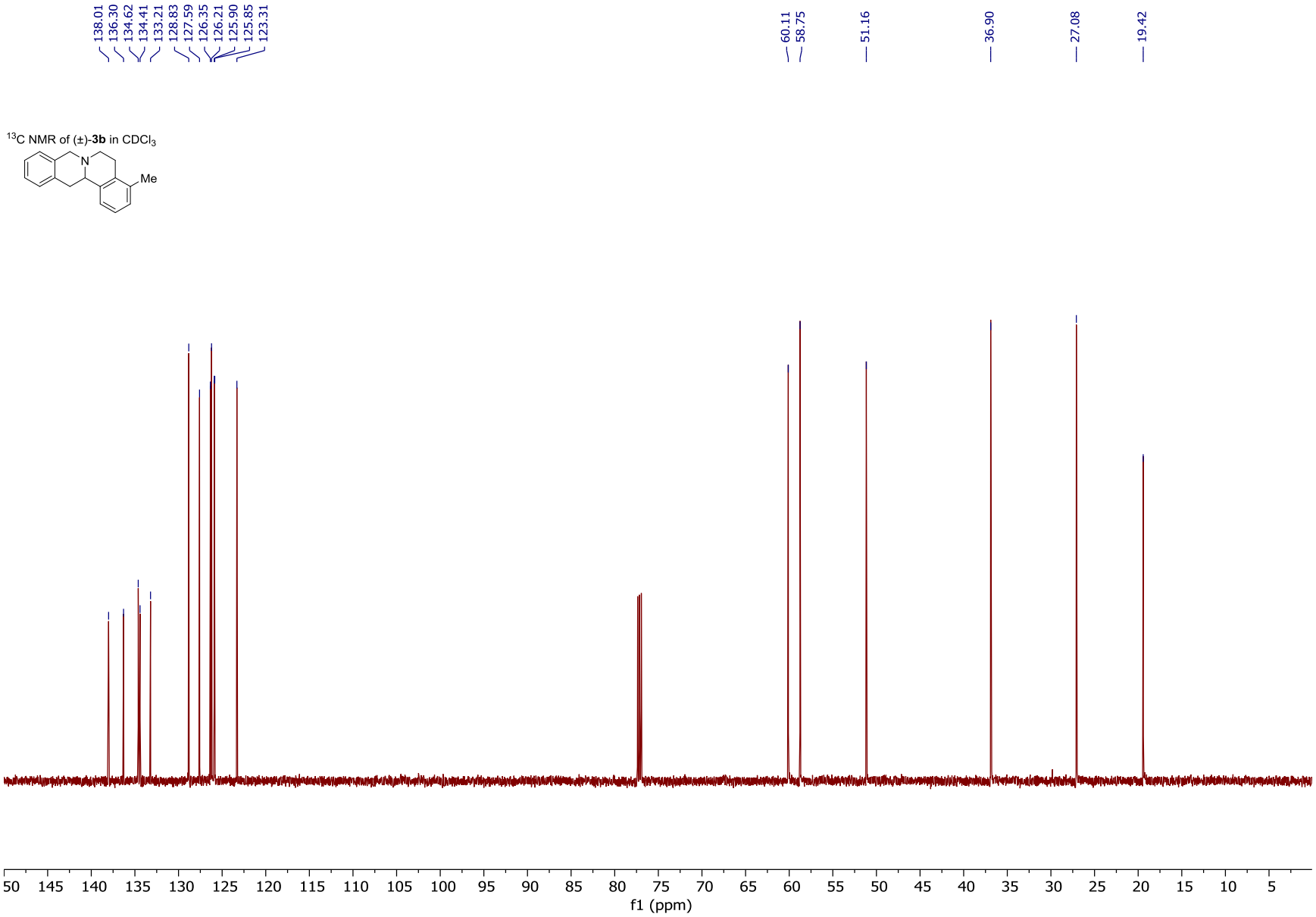

137.03
131.48
130.89
130.06
129.85
128.57
128.09
127.77
127.02
126.37
122.92
119.90
118.89
113.36
111.50
90.01
63.75
51.48
46.68
21.29
13C NMR of (±)-2m in CDCl3
O2N
N
H
Br
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

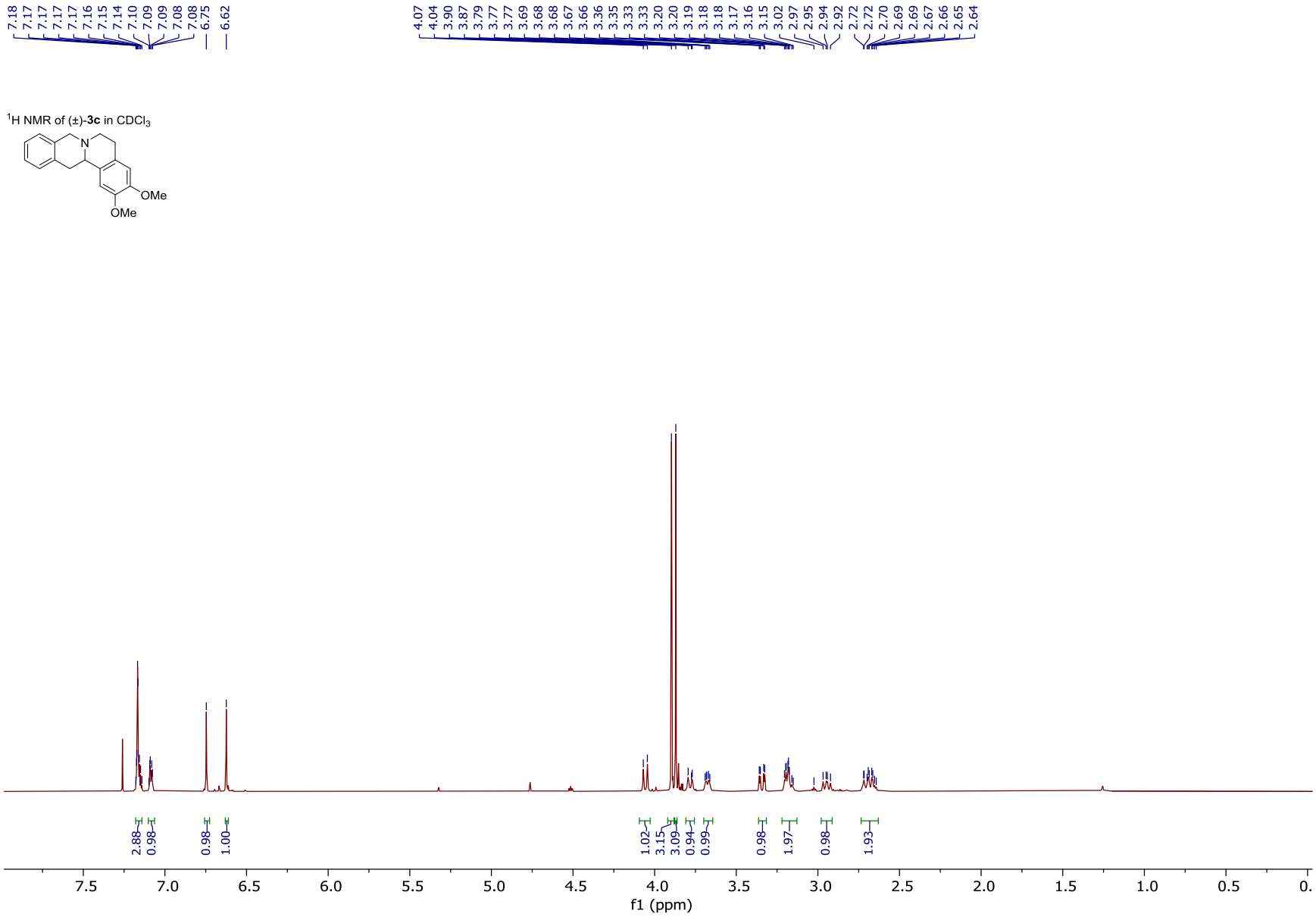
^{1}H NMR of (±)-**3a** in $CDCl_3$




137.98
134.63
134.57
134.49
129.01
128.89
126.42
126.29
126.27
126.18
125.98
125.63
60.01
58.74
51.33
36.75
29.61
13C NMR of (±)-3a in CDCl3
N
50 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

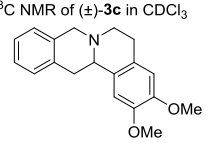

7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.14
7.13
7.13
7.12
7.11
7.11
7.10
7.09
4.10
4.07
3.79
3.76
3.75
3.74
3.44
3.43
3.41
3.40
3.29
3.28
3.28
3.27
3.27
3.26
3.26
3.25
3.05
3.04
3.03
3.02
3.01
3.00
2.99
2.98
2.96
2.95
2.93
2.79
2.78
2.78
2.76
2.76
2.75
2.69
2.68
2.67
2.66
2.65
2.64
2.31
^{1}H NMR of (±)-**3b** in $CDCl_3$
N
Me
5.02
2.00
1.00
2.01
1.00
1.00
2.01
1.00
1.00
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

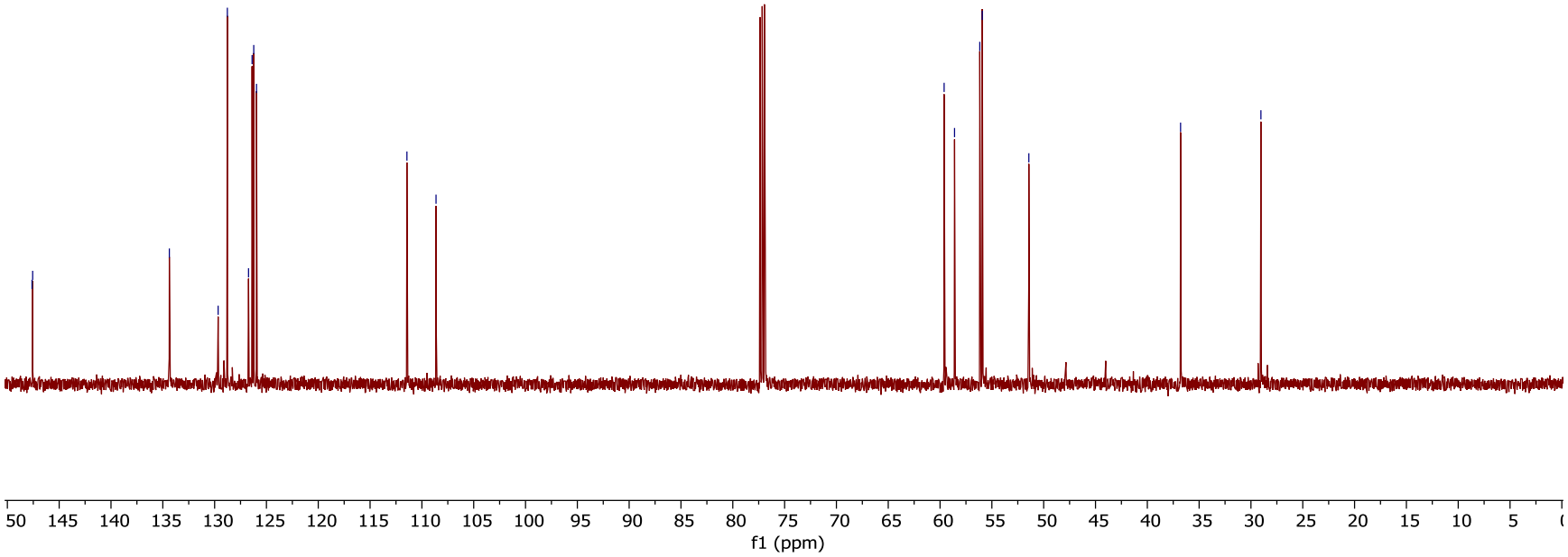

138.01
136.30
134.62
134.41
133.21
128.83
127.59
126.35
126.21
125.90
125.85
123.31
60.11
58.75
51.16
36.90
27.08
19.42
^{13}C NMR of (±)-**3b** in $CDCl_3$
N
Me
50 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)


7.18
7.17
7.17
7.17
7.17
7.16
7.15
7.14
7.10
7.09
7.09
7.08
7.08
6.75
6.62
4.07
4.04
3.90
3.87
3.79
3.77
3.77
3.69
3.68
3.68
3.67
3.66
3.36
3.35
3.33
3.33
3.20
3.20
3.19
3.18
3.18
3.17
3.16
3.15
3.02
2.97
2.95
2.94
2.92
2.72
2.72
2.70
2.69
2.69
2.67
2.66
2.65
2.64
1H NMR of (±)-3c in CDCl3
N
OMe
OMe
2.88
0.98
0.98
1.00
1.02
3.15
3.09
0.94
0.99
0.98
1.97
0.98
1.93
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

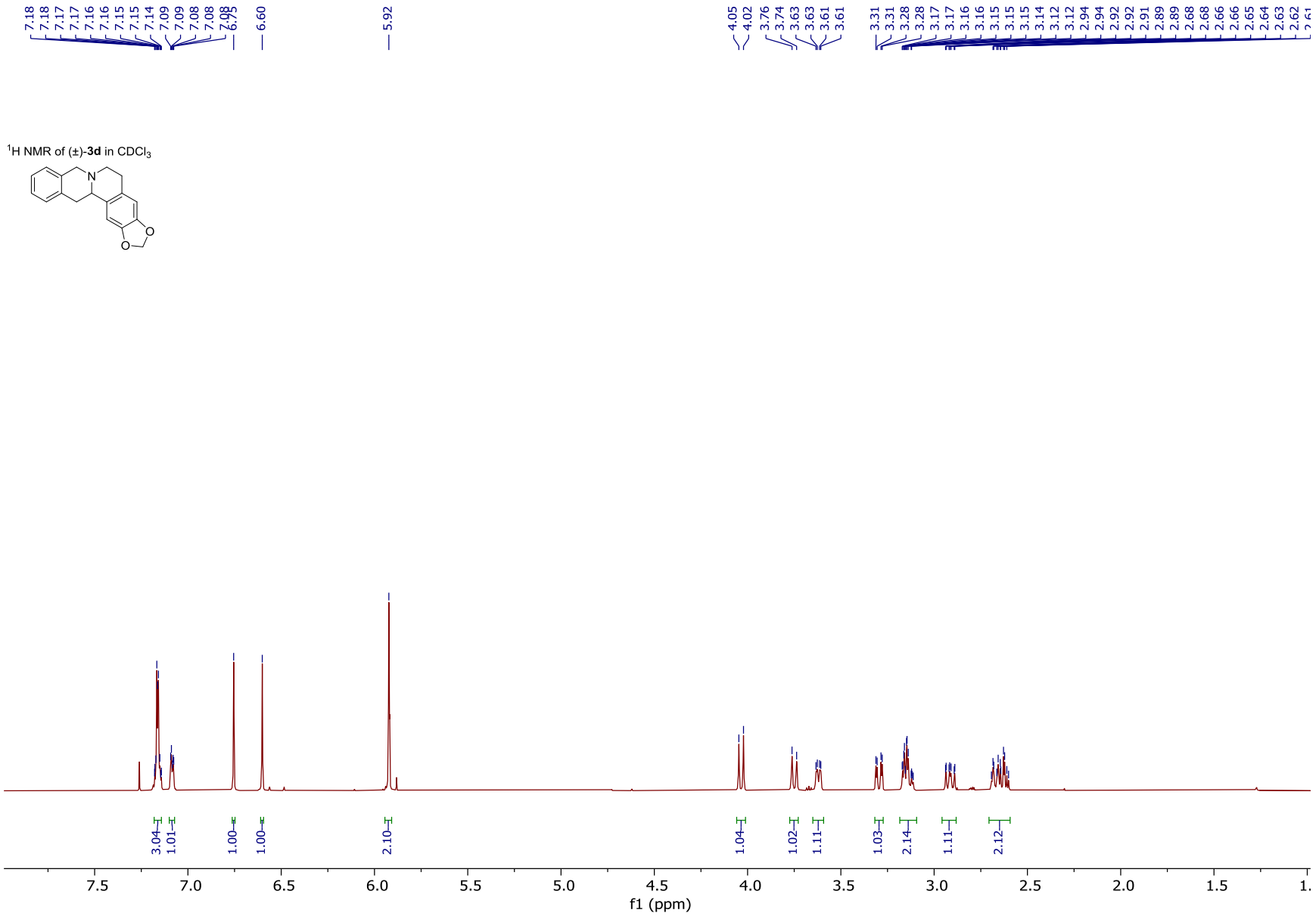
147.62
147.56
134.37
129.66
128.77
126.73
126.39
126.22
125.96
111.44
108.63
59.61
58.60
56.17
55.92
51.43
36.79
29.04

^{13}C NMR of (±)-**3c** in $CDCl_3$

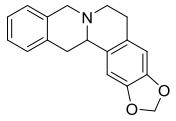




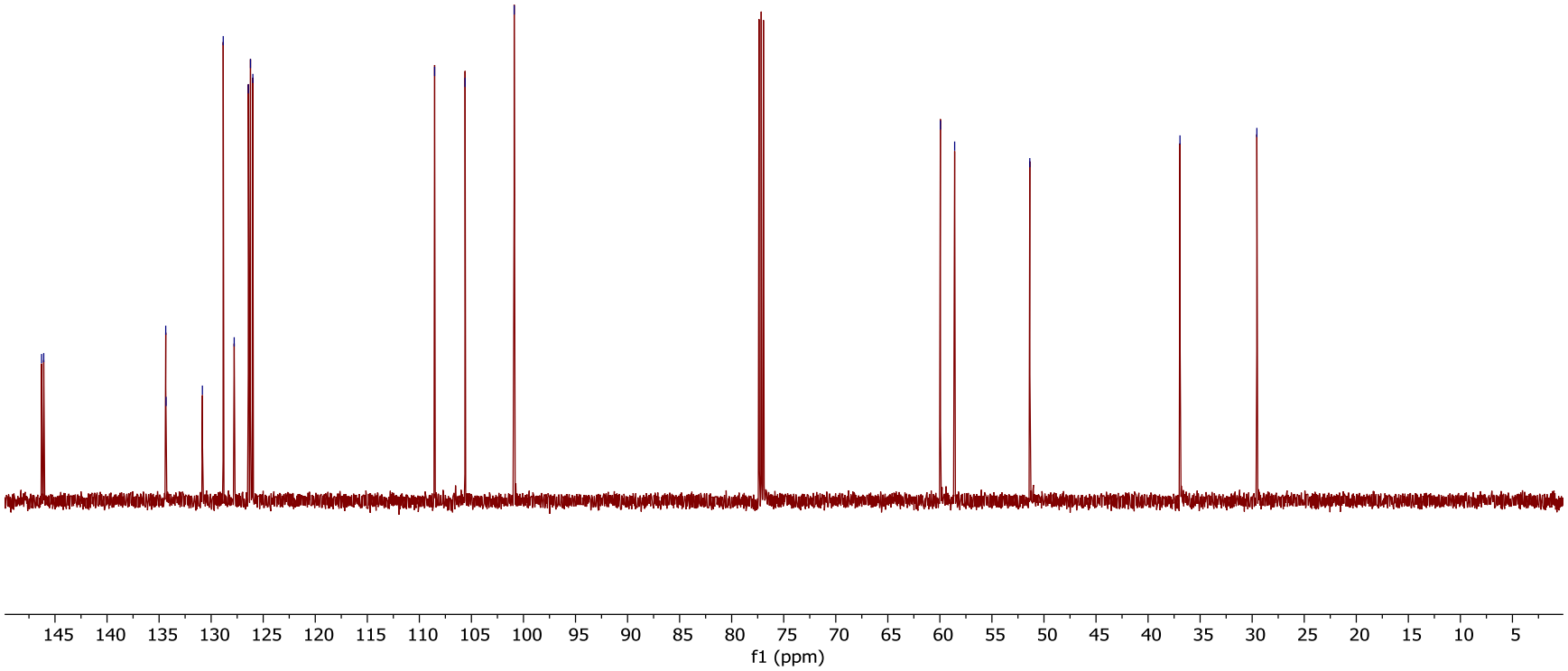
^{1}H NMR of (±)-**3d** in $CDCl_3$



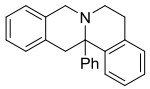
^{13}C NMR of (±)-**3d** in $CDCl_3$

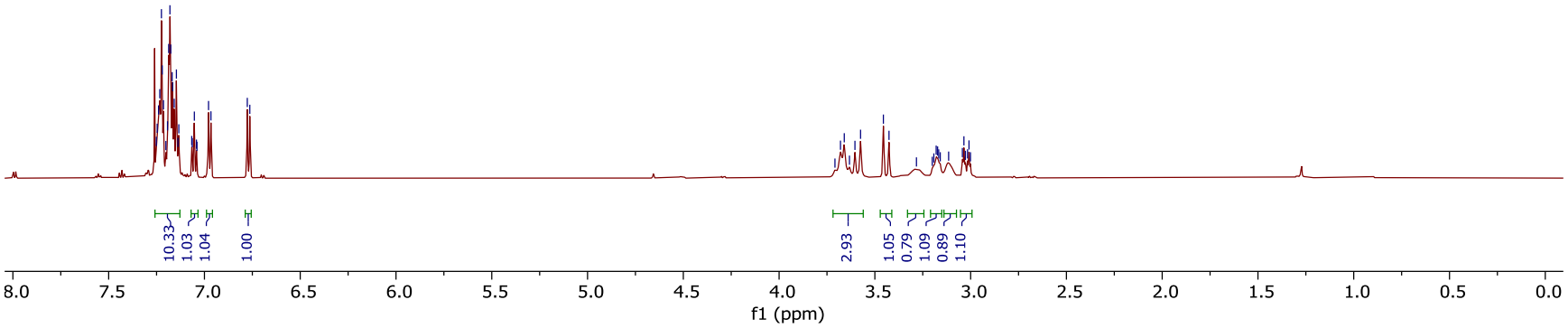


146.29
146.08
134.36
134.33
130.84
128.84
127.79
126.43
126.22
125.99
108.53
105.61
100.89
59.95
58.58
51.36
36.94
29.55

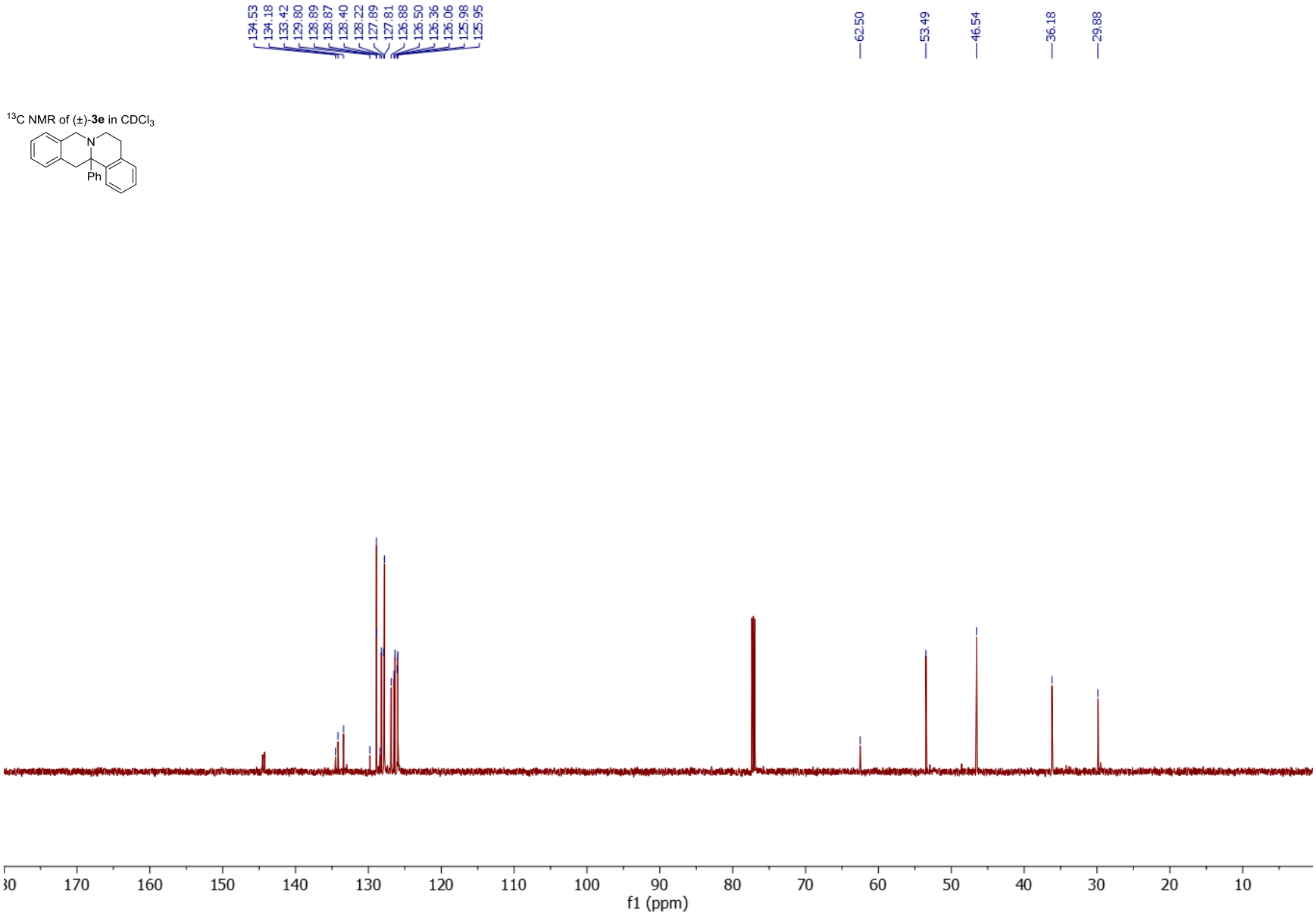


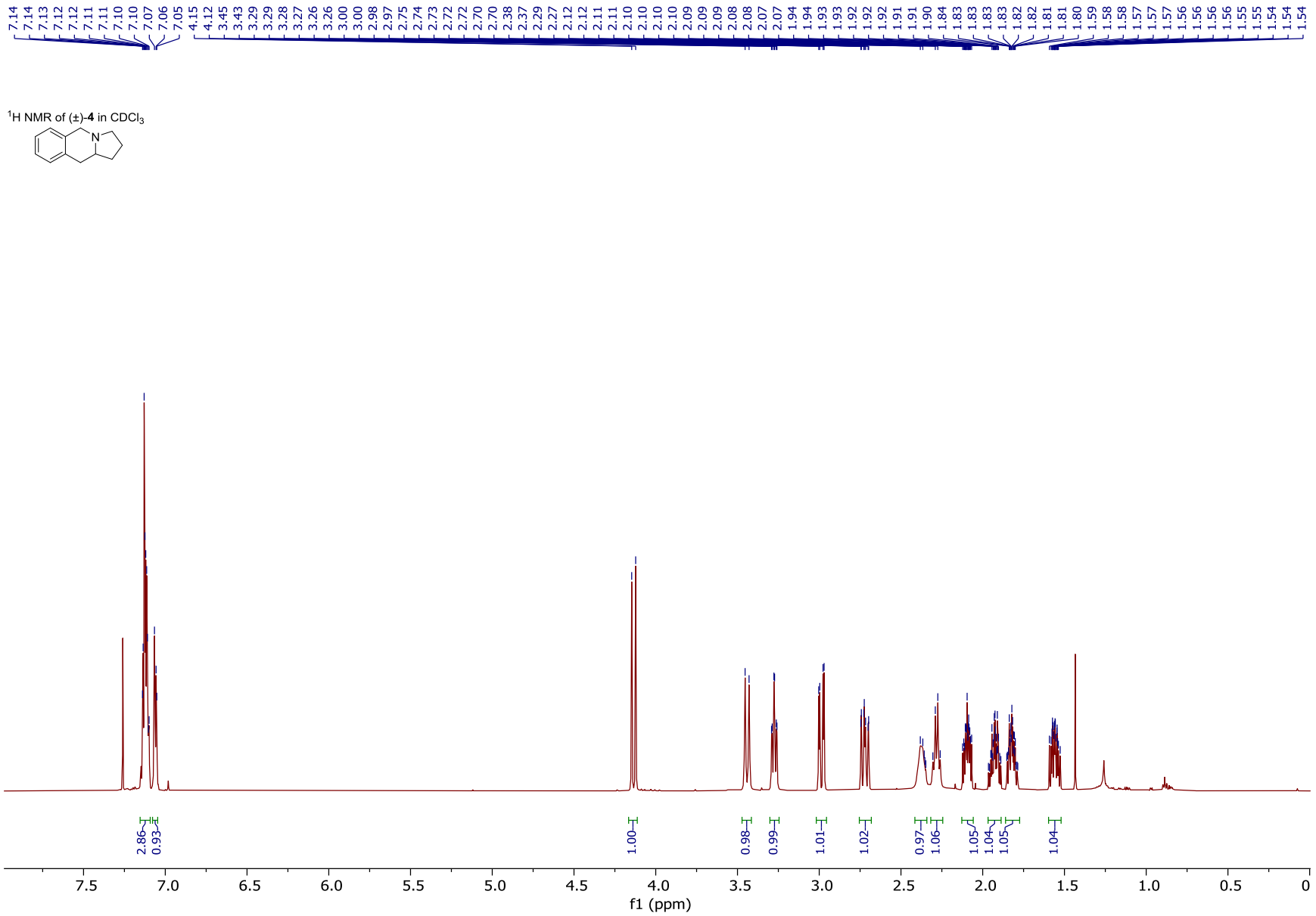
^{1}H NMR of (±)-**3e** in $CDCl_3$

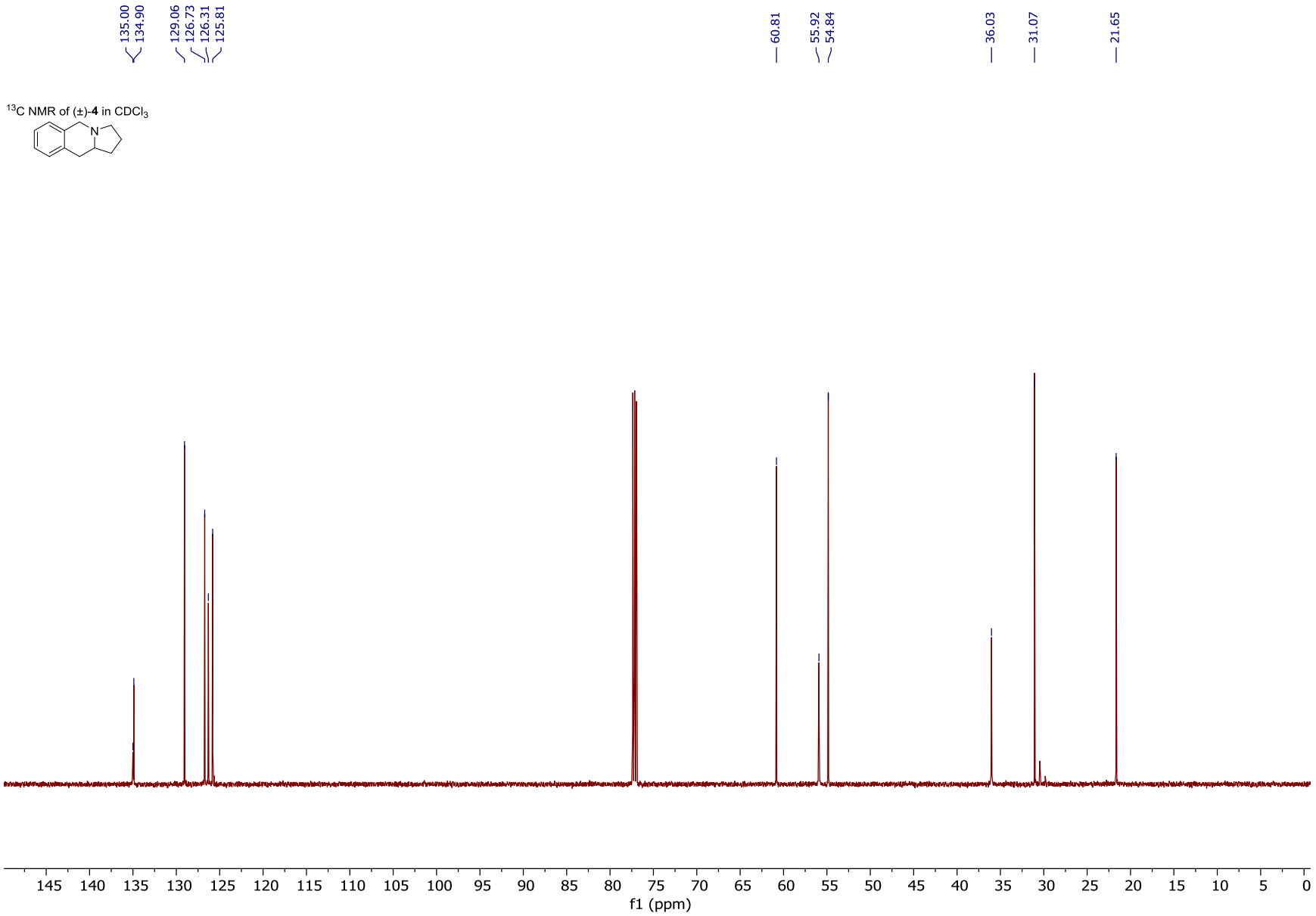


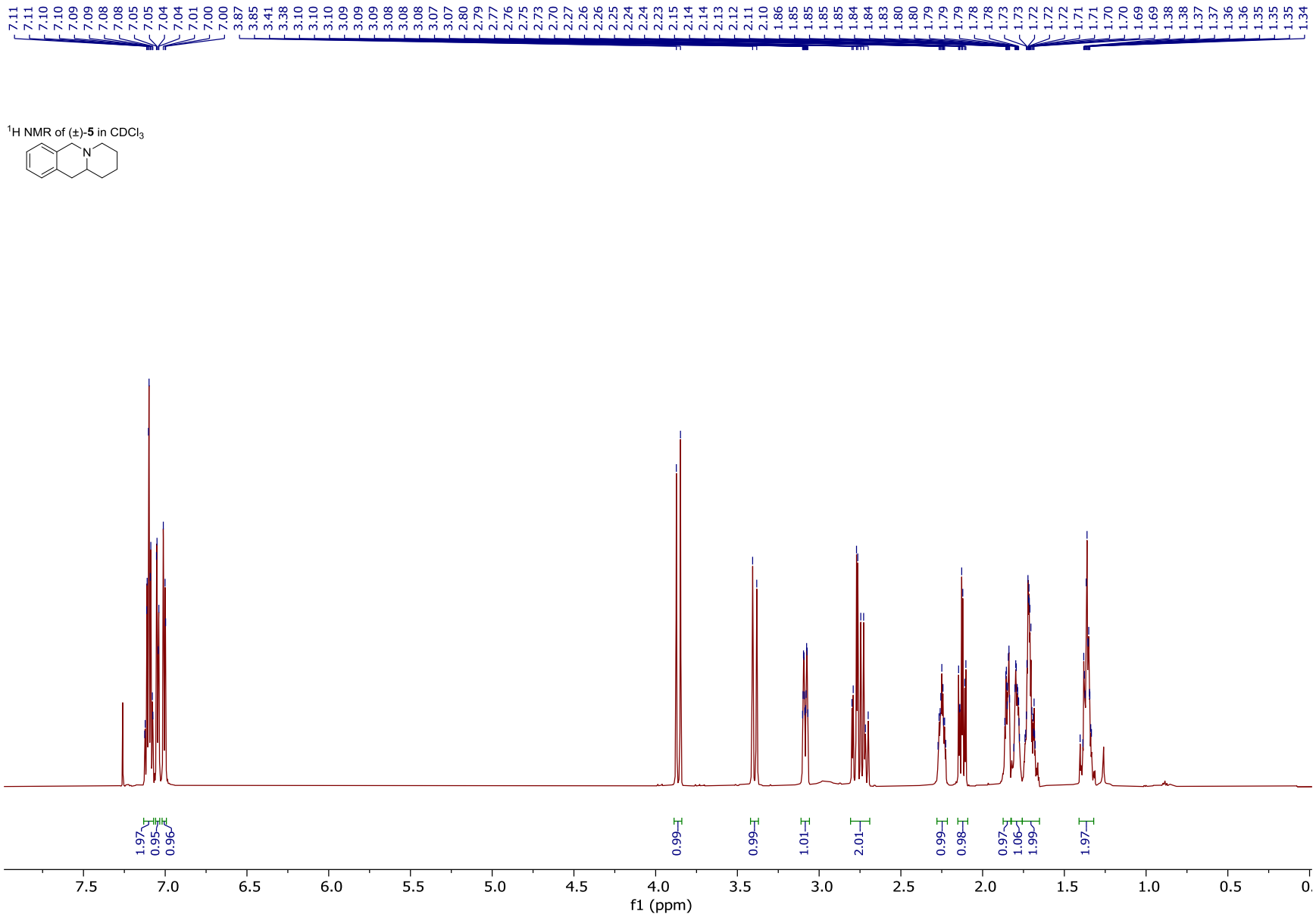


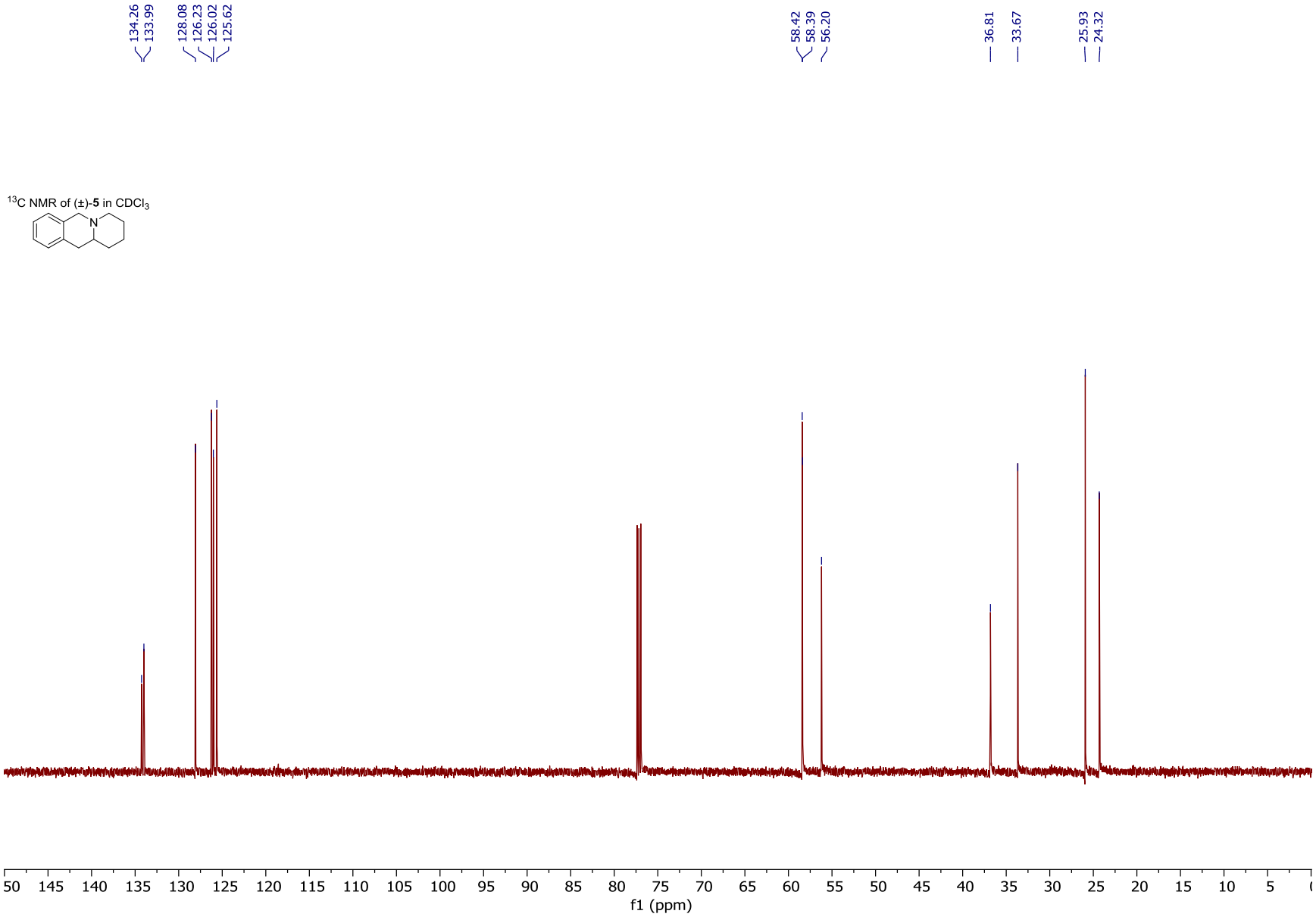
^{13}C NMR of (±)-**3e** in $CDCl_3$




7.14 7.14 7.13 7.12 7.12 7.11 7.11 7.10 7.10 7.07 7.06 7.05 4.15 4.12 3.45 3.43 3.29 3.29 3.28 3.27 3.26 3.26 3.00 3.00 2.98 2.97 2.75 2.74 2.73 2.72 2.72 2.70 2.70 2.38 2.37 2.29 2.27 2.12 2.12 2.11 2.11 2.10 2.10 2.10 2.10 2.09 2.09 2.09 2.08 2.08 2.07 2.07 1.94 1.94 1.93 1.93 1.92 1.92 1.92 1.91 1.91 1.90 1.84 1.83 1.83 1.83 1.82 1.82 1.81 1.81 1.80 1.59 1.58 1.58 1.57 1.57 1.57 1.56 1.56 1.56 1.55 1.55 1.54 1.54 1.54
^{1}H NMR of (±)-**4** in $CDCl_3$
N
2.86 0.93 1.00 0.98 0.99 1.01 1.02 0.97 1.06 1.05 1.04 1.05 1.04
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
f1 (ppm)


135.00
134.90
129.06
126.73
126.31
125.81
60.81
55.92
54.84
36.03
31.07
21.65
^{13}C NMR of (±)-**4** in $CDCl_3$
N
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


7.11 7.11 7.10 7.10 7.09 7.09 7.08 7.08 7.05 7.05 7.04 7.04 7.01 7.00 7.00 3.87 3.85 3.41 3.38 3.10 3.10 3.10 3.09 3.09 3.09 3.08 3.08 3.08 3.07 3.07 2.80 2.79 2.77 2.76 2.75 2.73 2.70 2.27 2.26 2.26 2.25 2.24 2.24 2.23 2.15 2.14 2.14 2.13 2.12 2.11 2.10 1.86 1.85 1.85 1.85 1.85 1.84 1.84 1.83 1.80 1.80 1.79 1.79 1.79 1.78 1.78 1.73 1.73 1.72 1.72 1.72 1.71 1.71 1.70 1.70 1.69 1.69 1.38 1.38 1.37 1.37 1.36 1.36 1.35 1.35 1.35 1.34
1H NMR of (±)-5 in CDCl3
N
1.97 0.95 0.96 0.99 0.99 1.01 2.01 0.99 0.98 0.97 1.06 1.99 1.97
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


134.26
133.99
128.08
126.23
126.02
125.62
58.42
58.39
56.20
36.81
33.67
25.93
24.32
^{13}C NMR of (±)-**5** in CDCl$_3$
N
50 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)