Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Support studies toward the hicksoane alkaloids reveals cascade reactions of a (tryptophanamido)methylglycinate

Stephanie Lee, Tilo Söhnel and Jonathan Sperry*

j.sperry@auckland.ac.nz

SUPPORTING INFORMATION

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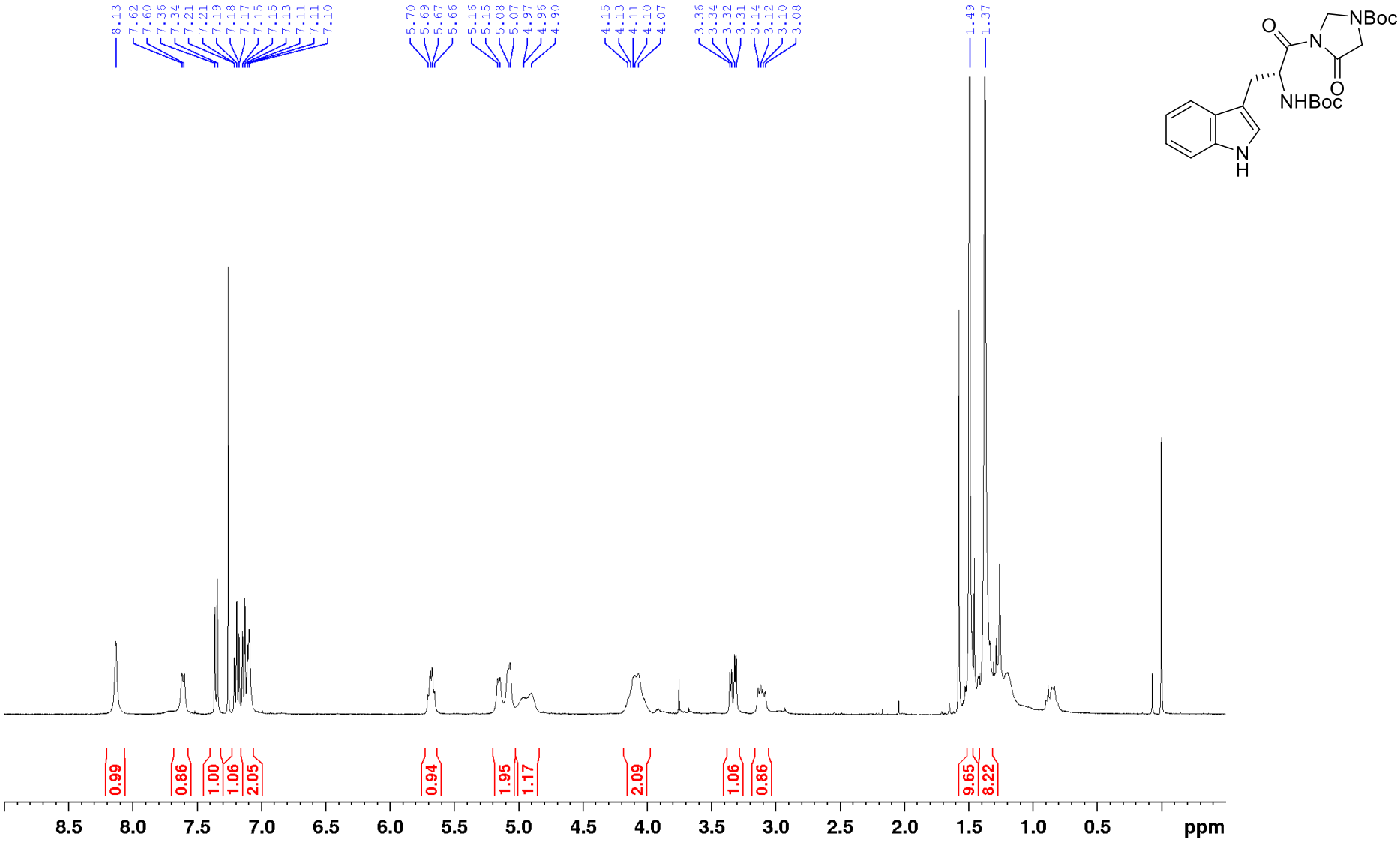
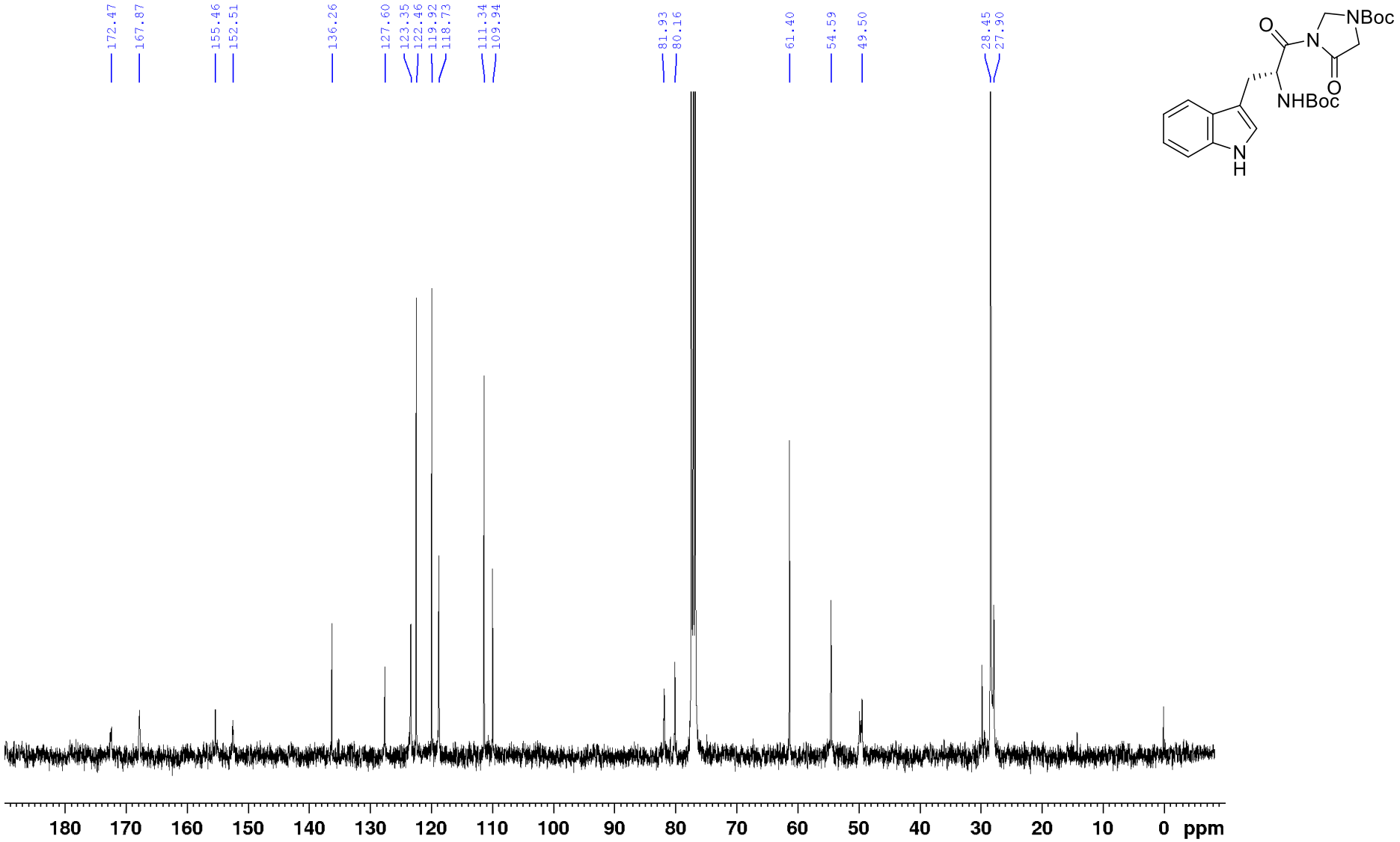
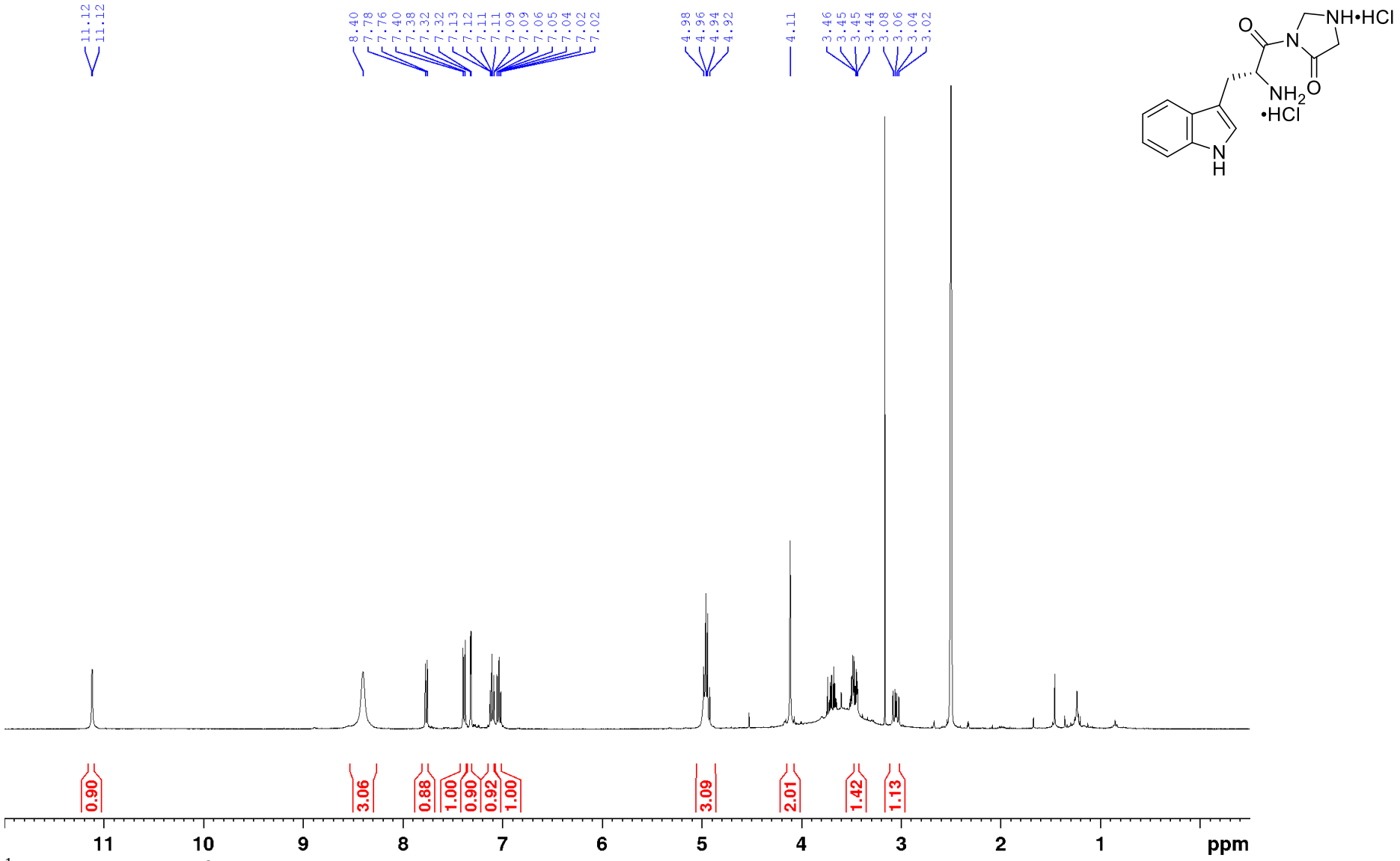


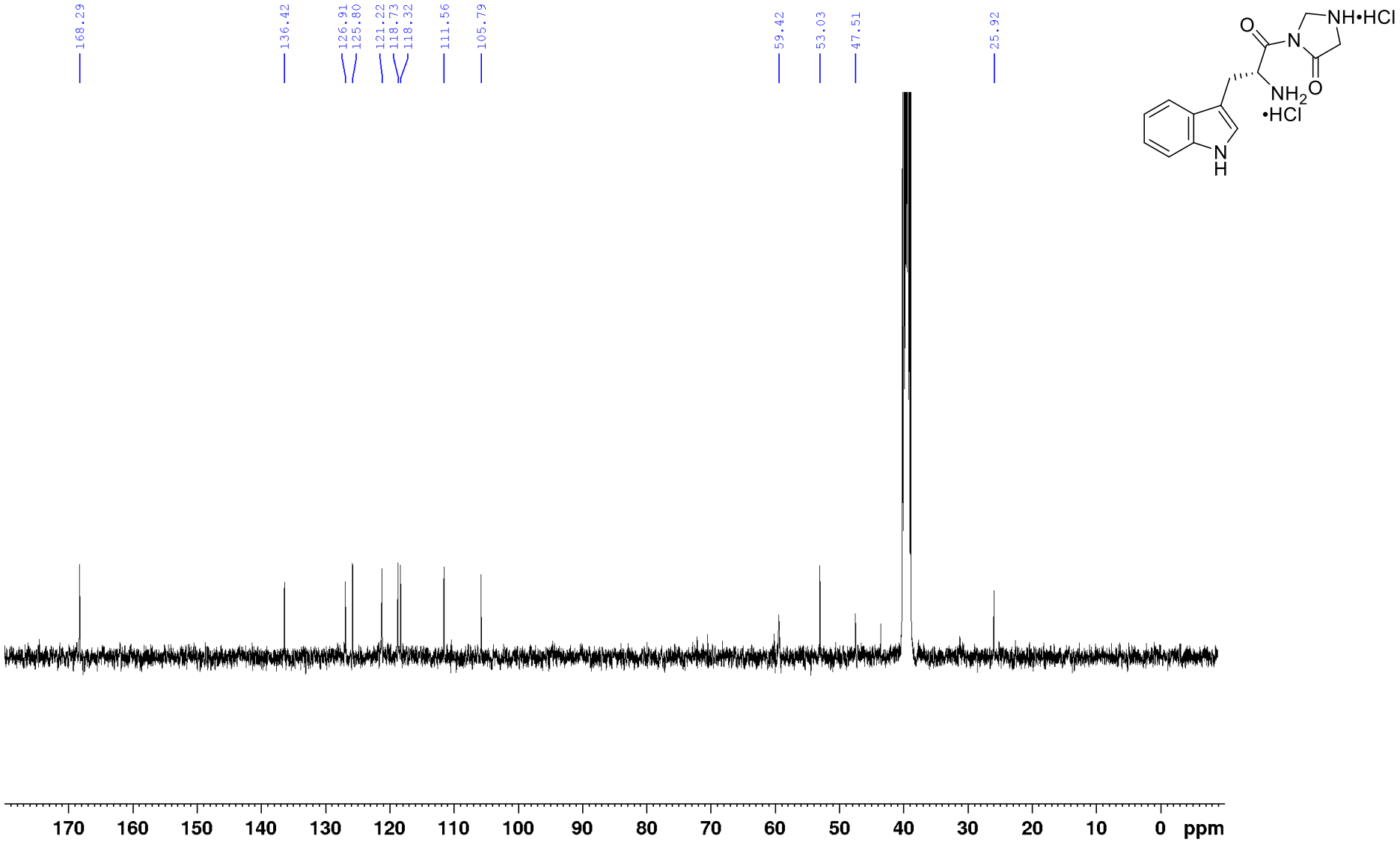
^{1}H NMR spectrum of **5** (400 MHz, $CDCl_3$).



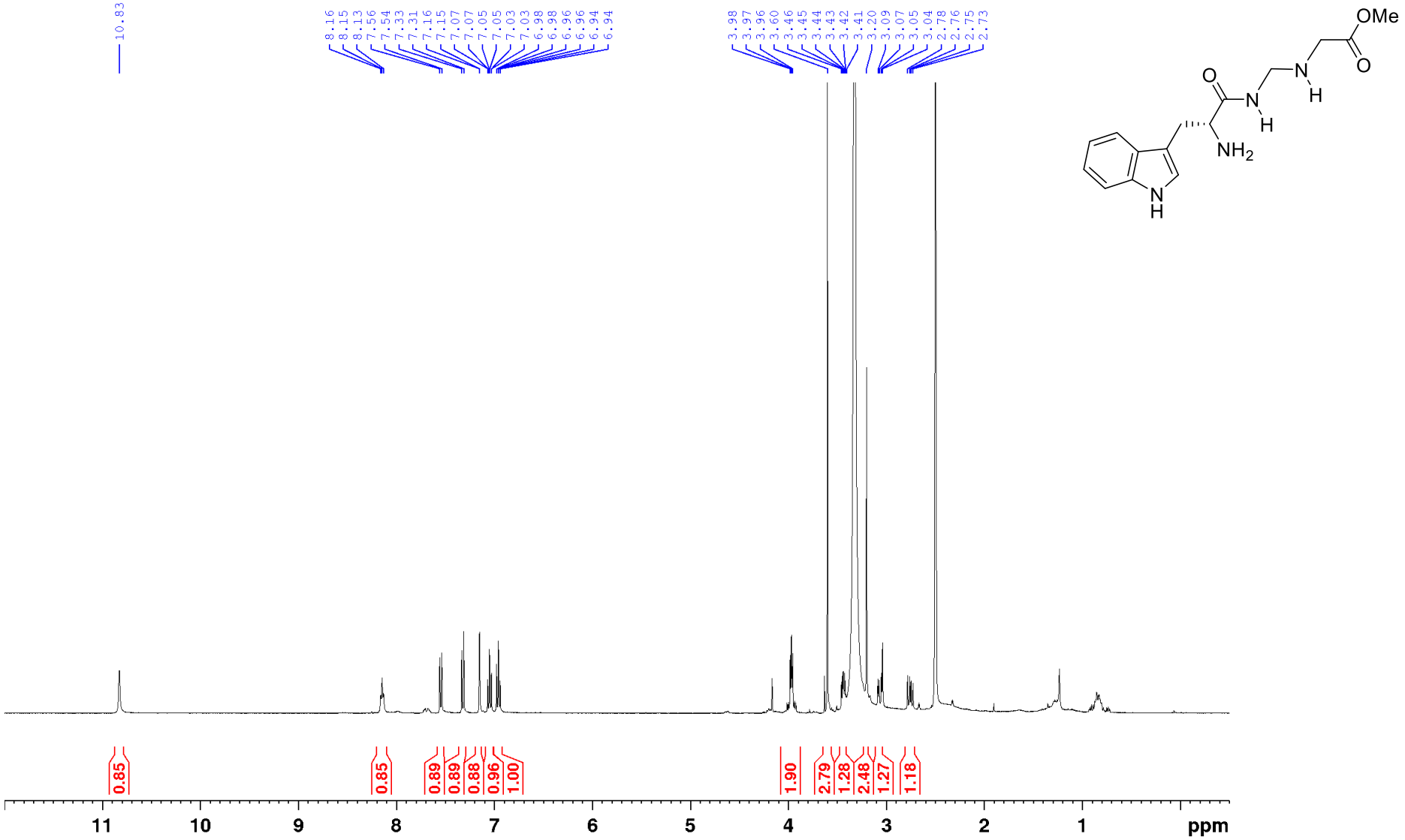
^{13}C NMR spectrum of **5** (100 MHz, $CDCl_3$).



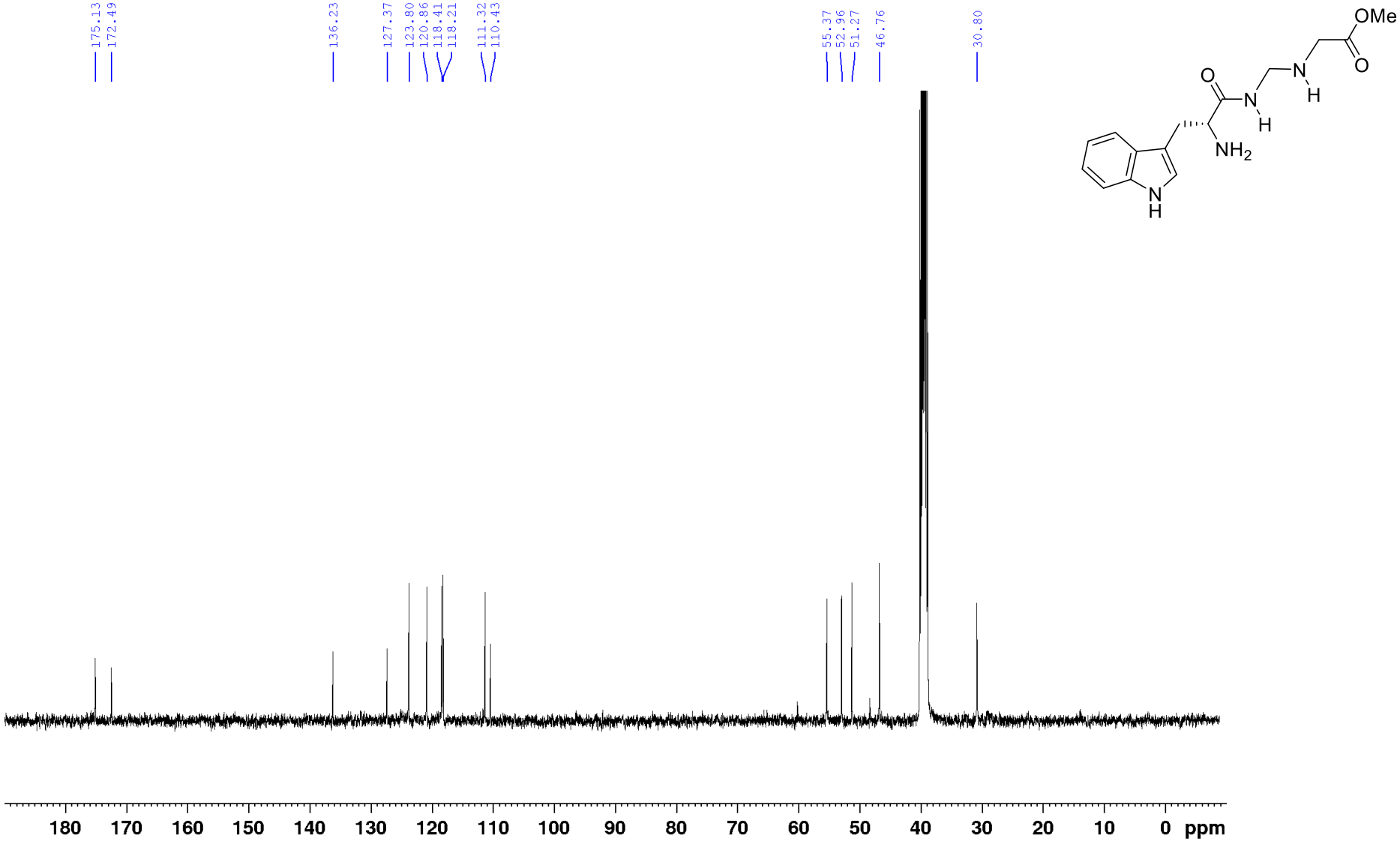
^{1}H NMR spectrum of **2•2HCl** (400 MHz, DMSO-d_6).



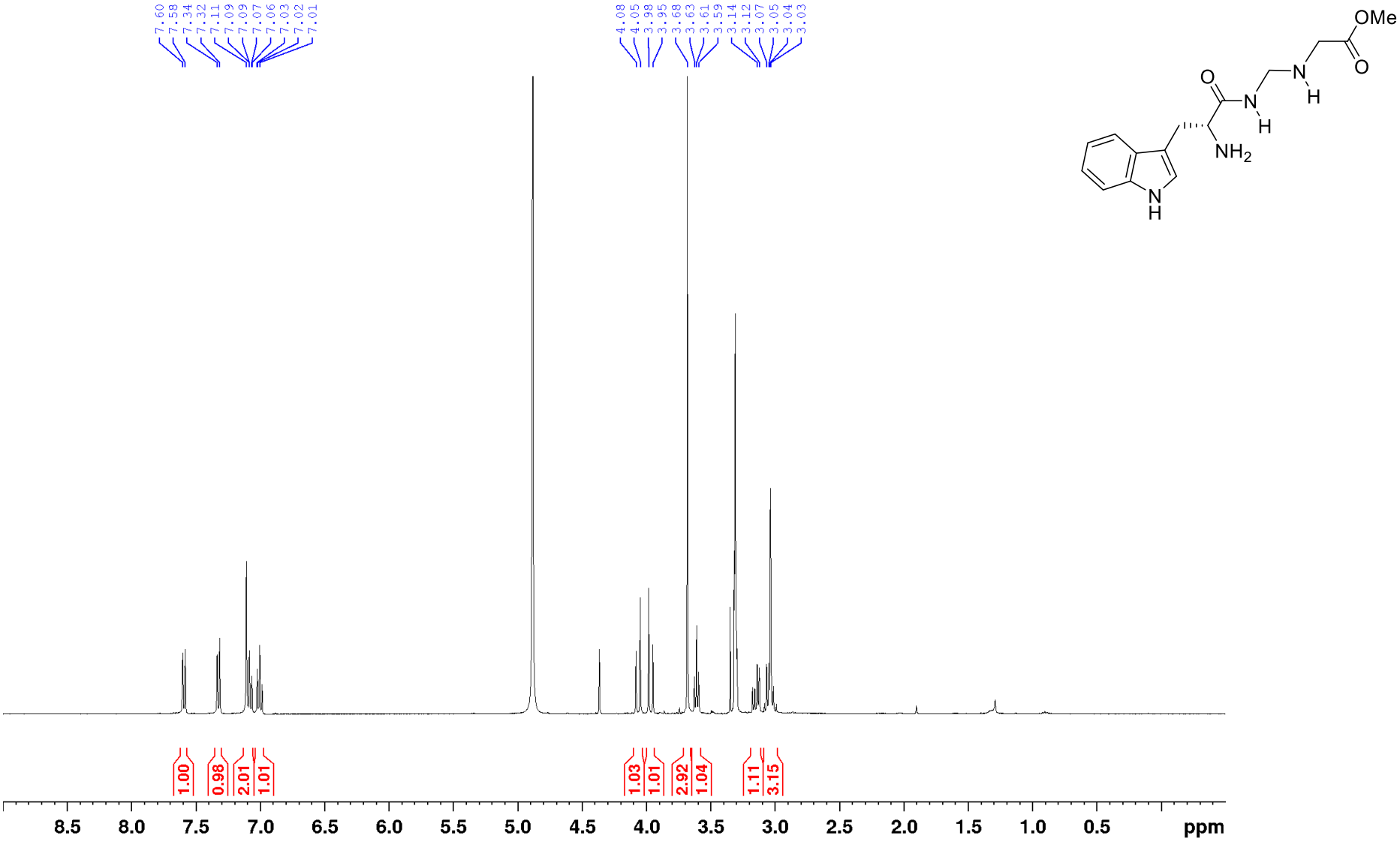
^{13}C NMR spectrum of **2•2HCl** (100 MHz, DMSO-d_6).



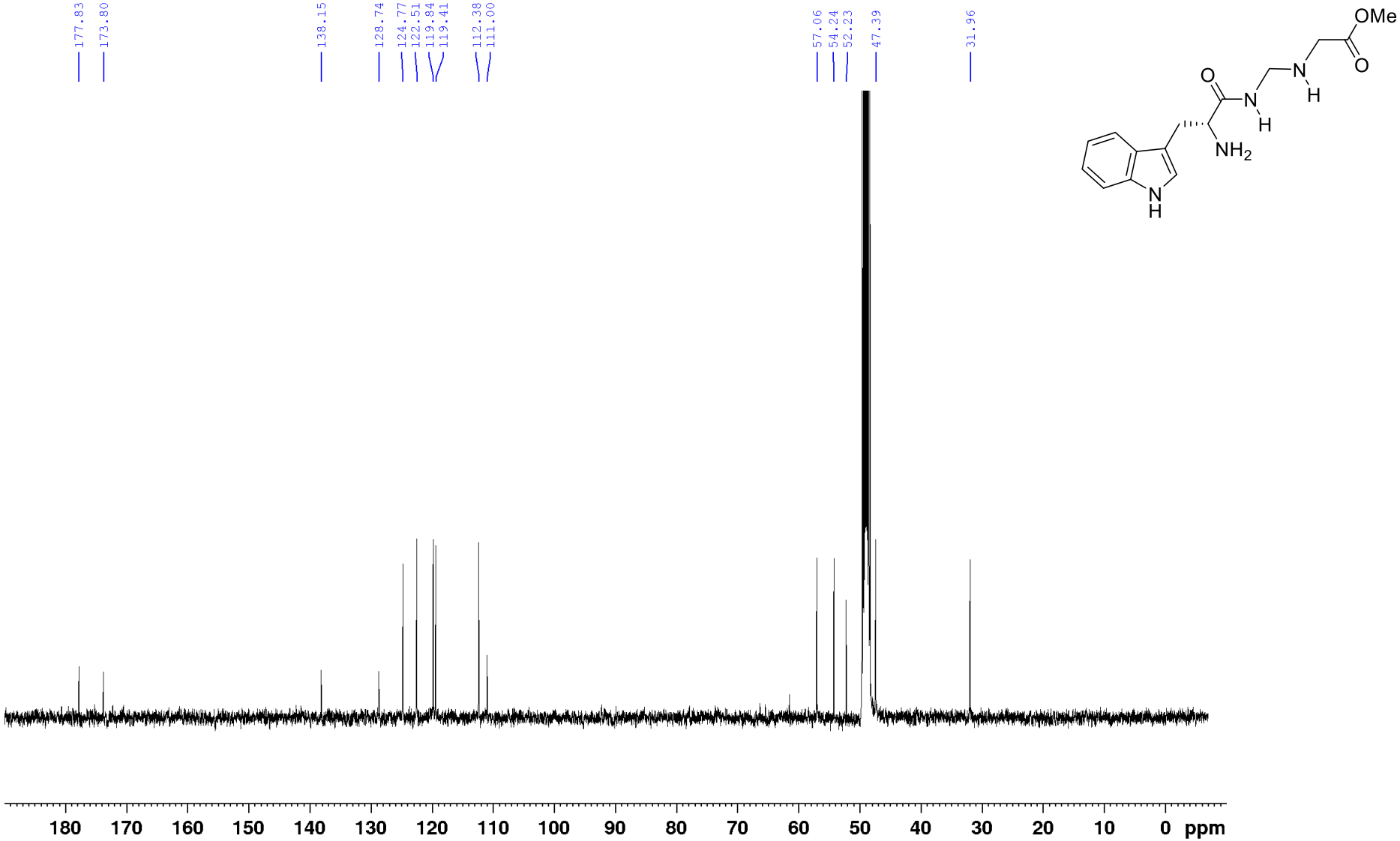
^{1}H NMR spectrum of **7** (400 MHz, DMSO-d_6).



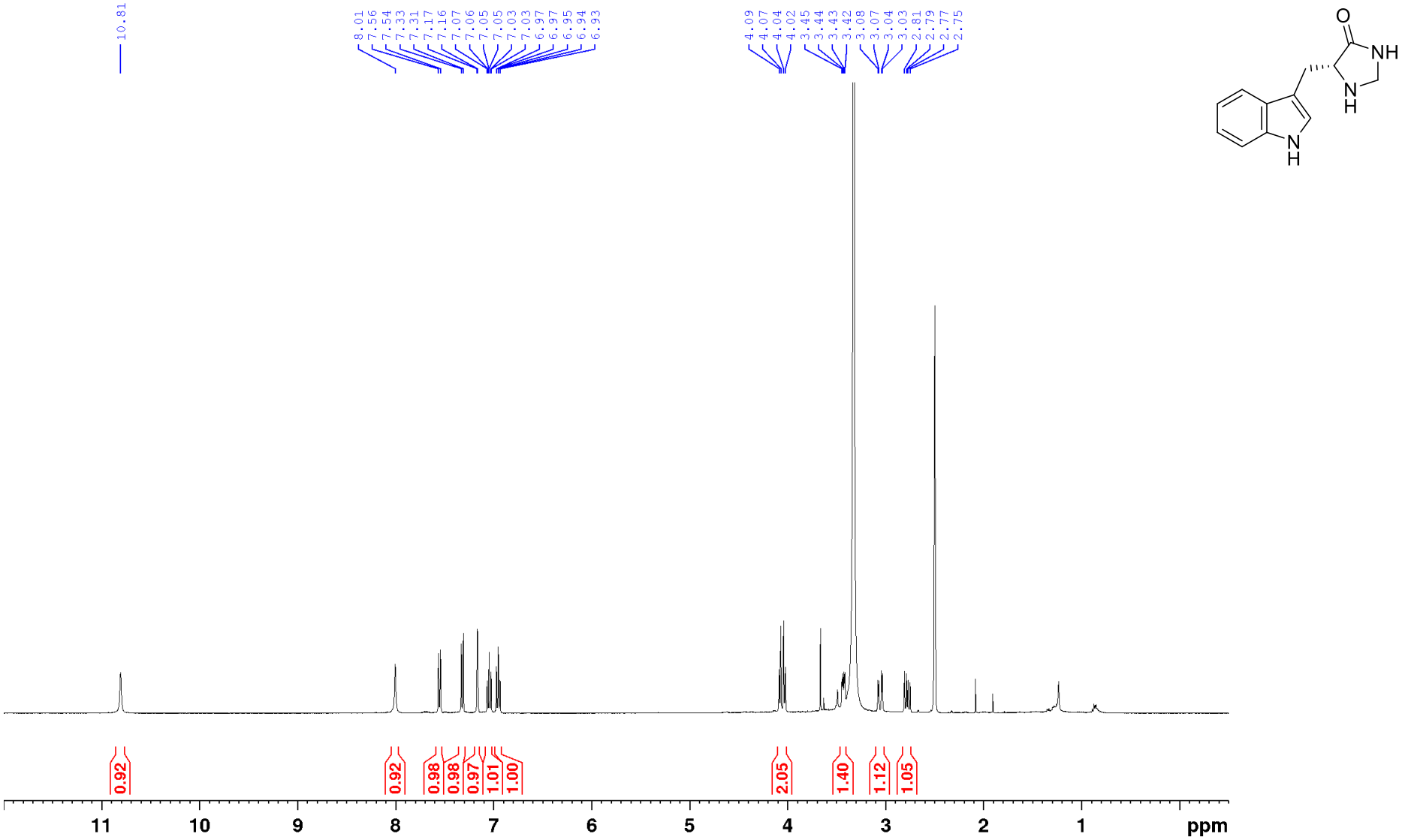
^{13}C NMR spectrum of **7** (100 MHz, DMSO-d_6).



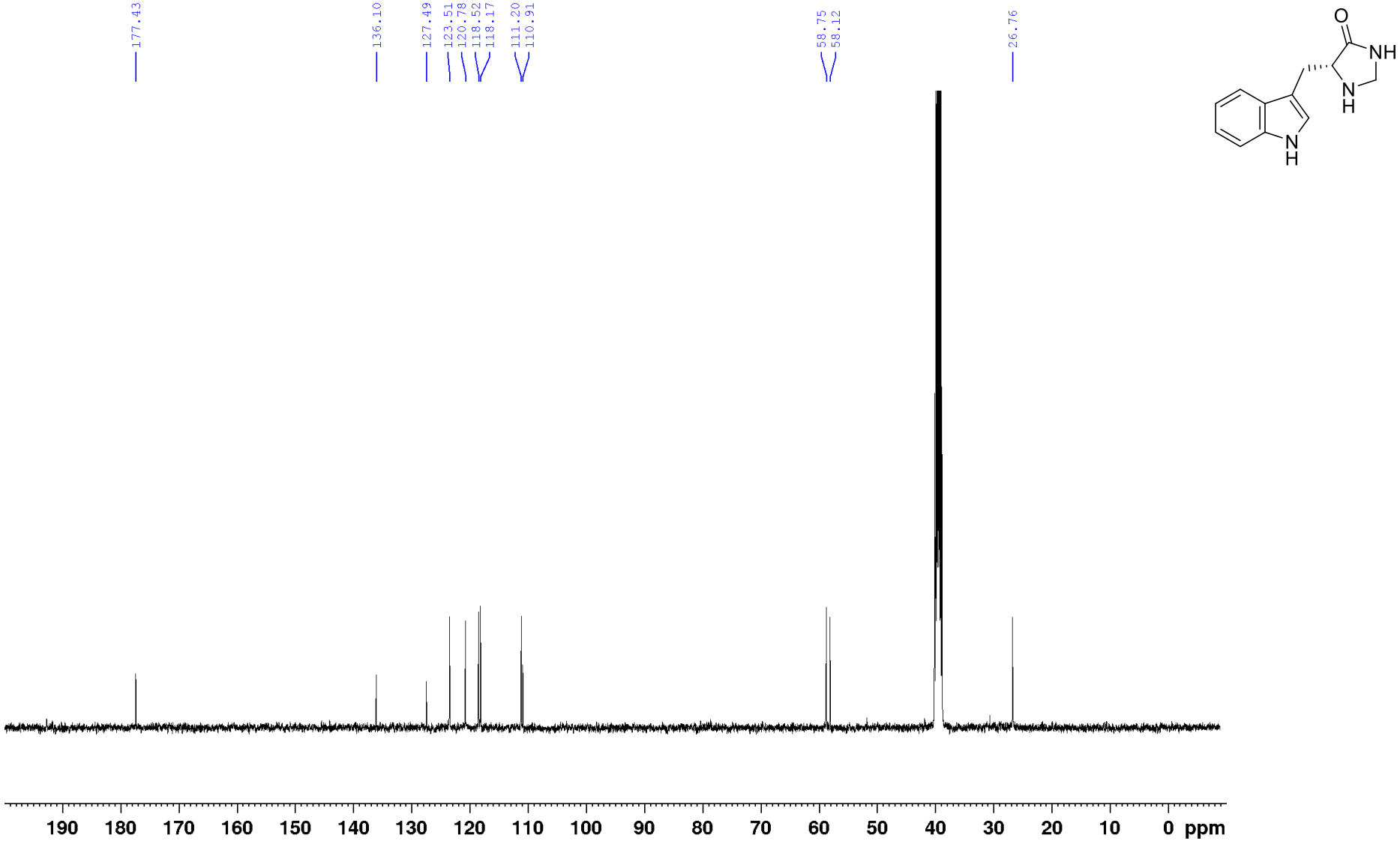
^{1}H NMR spectrum of **7** (400 MHz, CD_3OD).



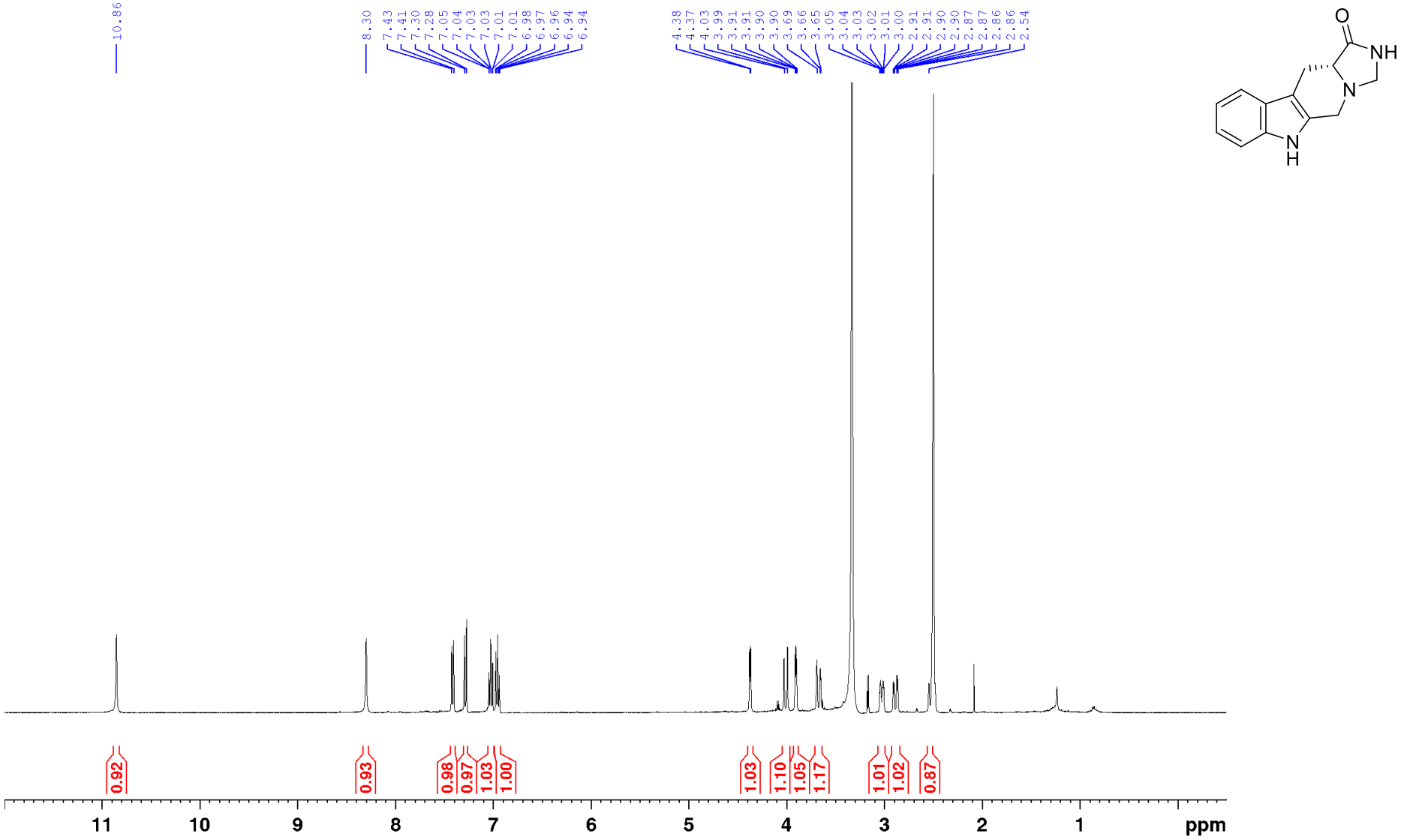
^{13}C NMR spectrum of **7** (100 MHz, CD_3OD).



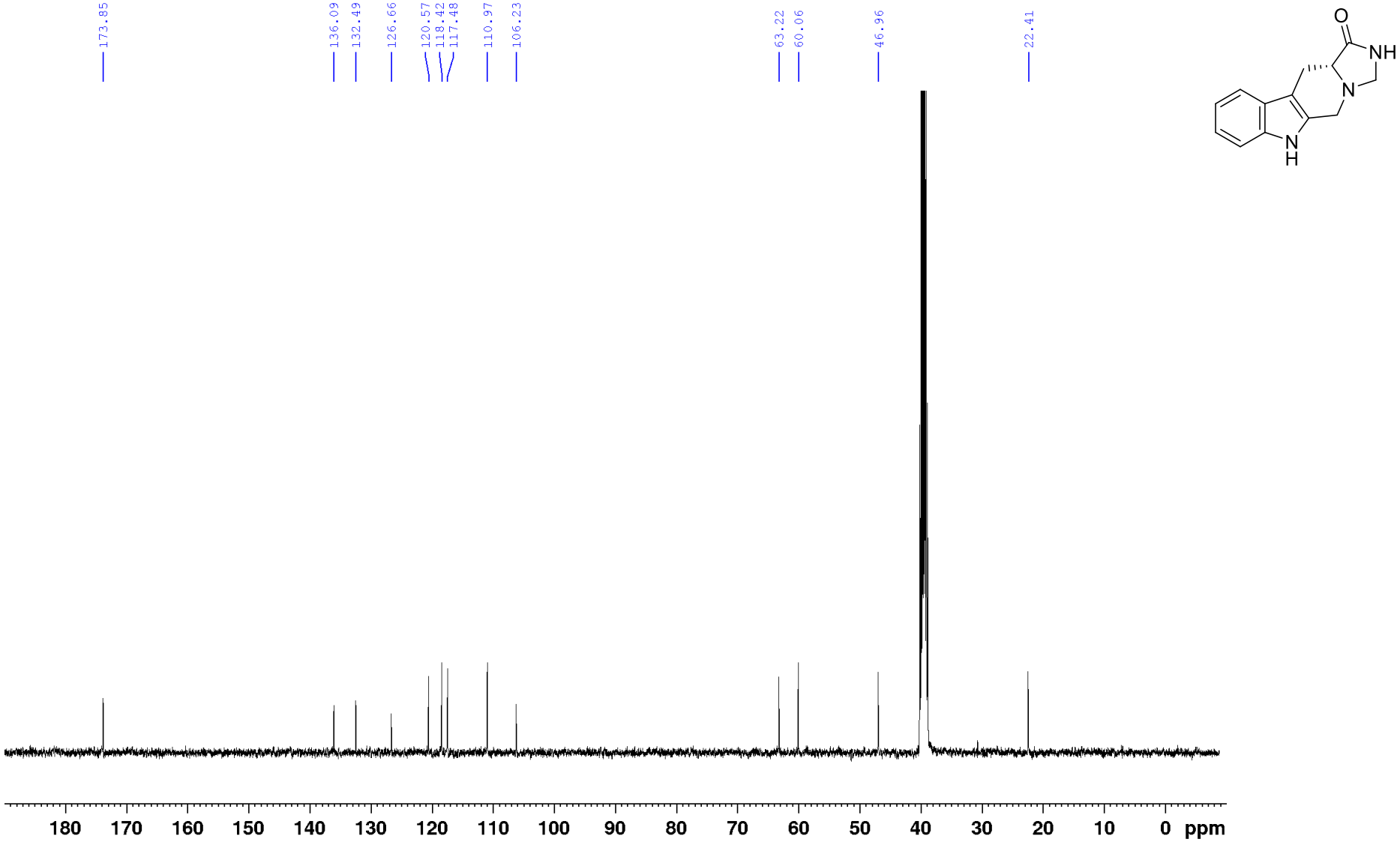
^{1}H NMR spectrum of **10** (400 MHz, DMSO-d_6).



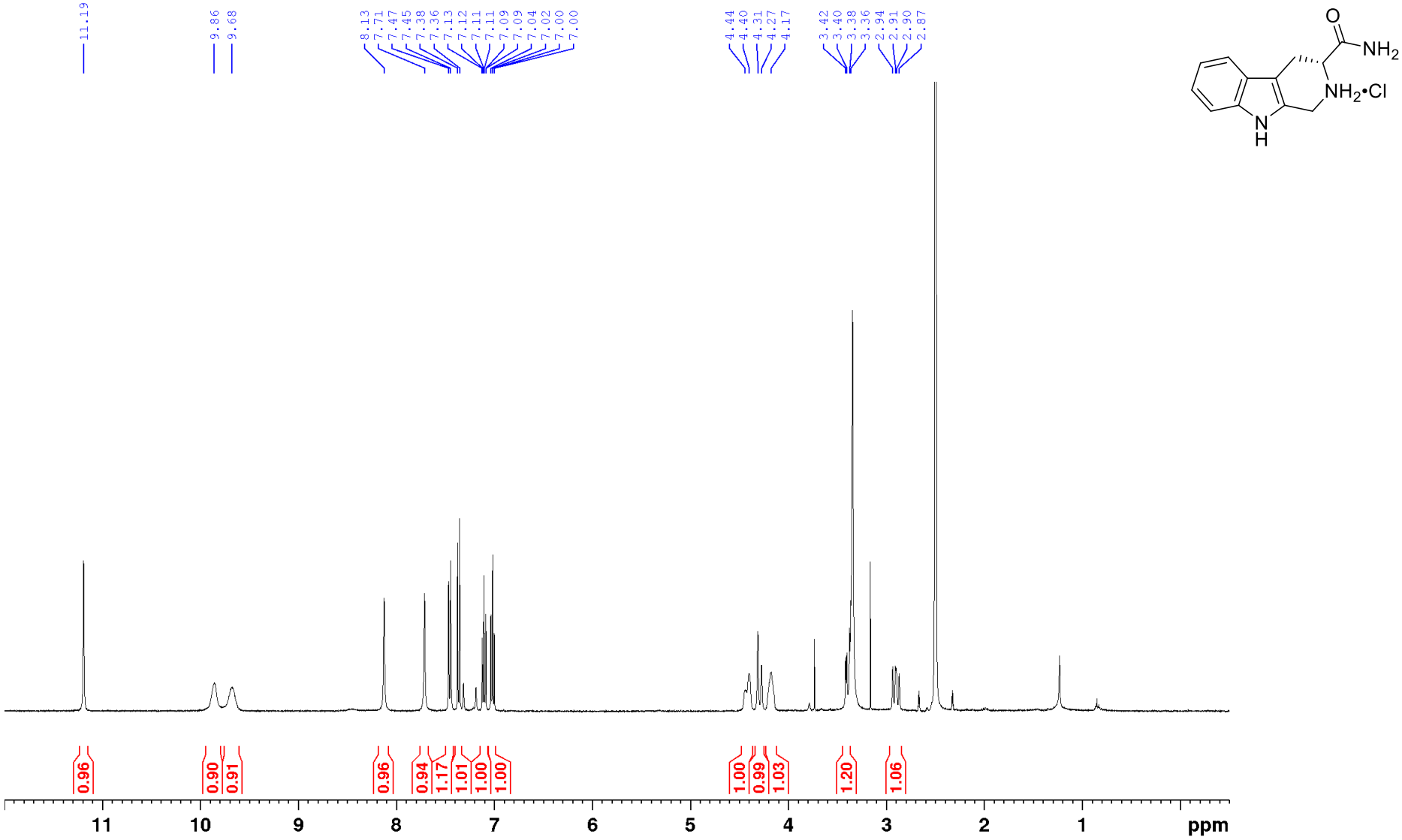
^{13}C NMR spectrum of **10** (100 MHz, DMSO-d_6).



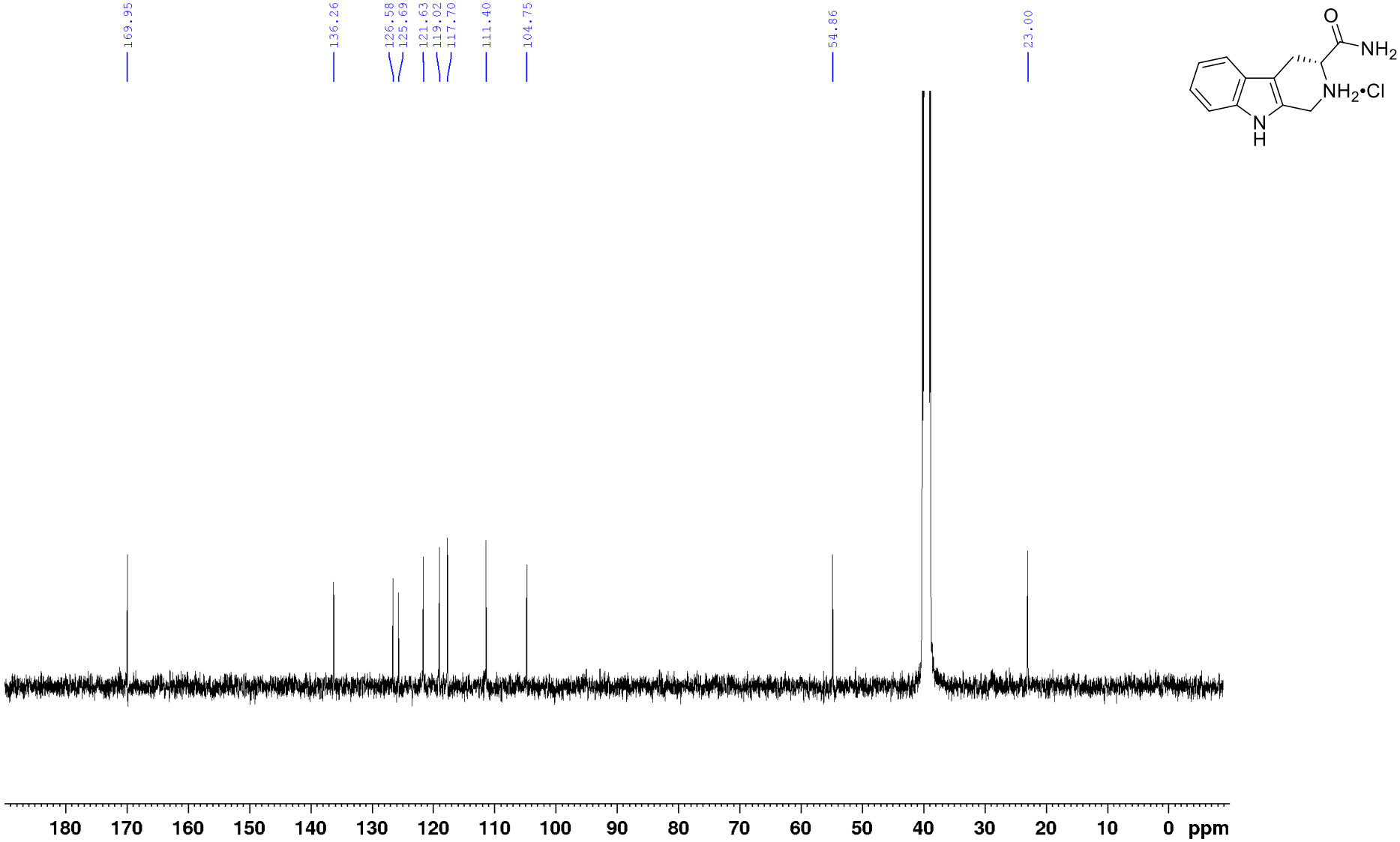
^{1}H NMR spectrum of **9** (400 MHz, DMSO-d_6).



^{13}C NMR spectrum of **9** (100 MHz, DMSO-d_6).

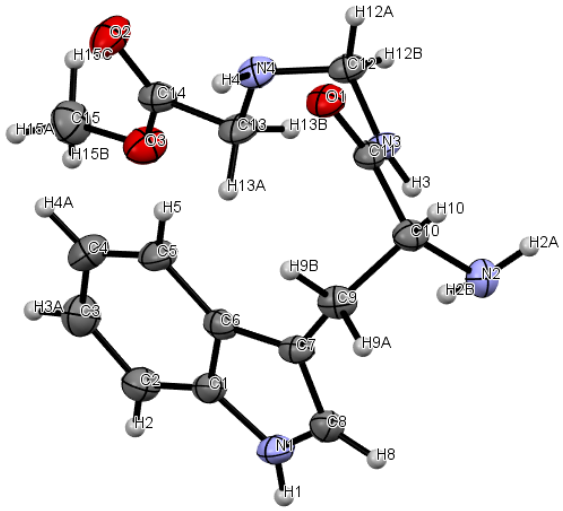


^{1}H NMR spectrum of **8•HCl** (400 MHz, DMSO-d_6).



^{13}C NMR spectrum of **8•HCl** (100 MHz, DMSO-d_6).

Crystallographic Data for 7

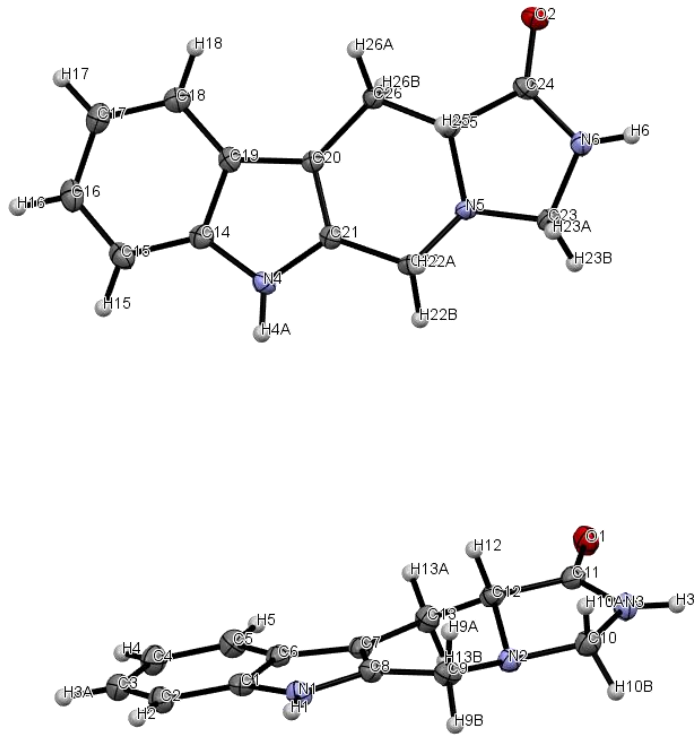


ORTEP representation of **7**.

Table S1. Crystal data and structure refinement for **7**.

CCDC number	2281801
Identification code	SYL-06-20_01
Empirical formula	$C_{15}H_{20}N_4O_3$
Formula weight	304.35
Temperature/K	120(1)
Crystal system	monoclinic
Space group	C2
a/Å	16.2851(13)
b/Å	5.2346(4)
c/Å	18.9492(16)
α/°	90
β/°	112.304(10)
γ/°	90
Volume/Å^3	1494.5(2)
Z	4
ρ_{calc}mg/mm^3	1.353
μ/mm^{-1}	0.794
F(000)	648.0
Crystal size/mm^3	0.2 × 0.05 × 0.04
2Θ range for data collection	11.748 to 135.458°
Index ranges	$-19 \leq h \leq 12$, $-6 \leq k \leq 5$, $-18 \leq l \leq 22$
Reflections collected	4802
Independent reflections	2156[R(int) = 0.0507]
Data/restraints/parameters	2156/4/206
Goodness-of-fit on F^2	1.043
Final R indexes [I>=2σ (I)]	R_1 = 0.0514, wR_2 = 0.1298
Final R indexes [all data]	R_1 = 0.0636, wR_2 = 0.1390
Largest diff. peak/hole / e Å^{-3}	0.37/-0.26
Flack parameter	0.0(3)
Crystallisation solvent system	Methanol/Dichloromethane (Slow evaporation)

Crystallographic Data for 9

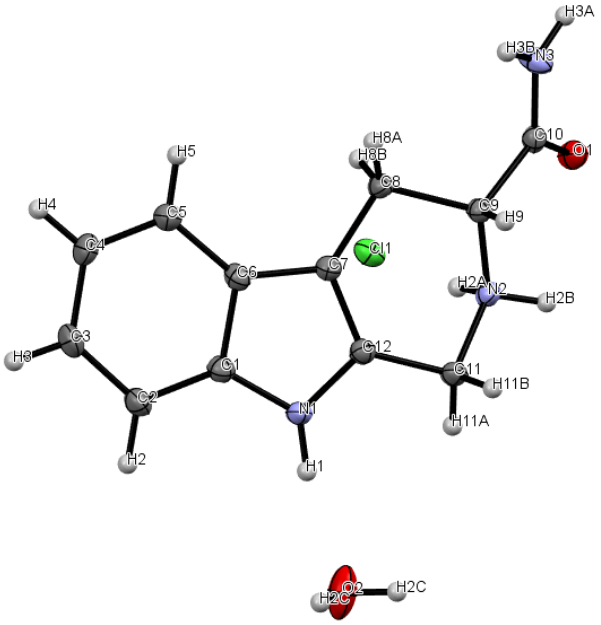


ORTEP representation with labels of two molecules of **9**.

Table S2. Crystal data and structure refinement for **9**.

CCDC number	2281800
Identification code	SYL-06-08
Empirical formula	$C_{26}H_{26}N_6O_2$
Formula weight	454.53
Temperature/K	113(1)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.45921(12)
b/Å	15.7500(3)
c/Å	21.1734(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2154.03(7)
Z	4
ρ_{calc}mg/mm^3	1.402
μ/mm^{-1}	0.743
F(000)	960.0
Crystal size/mm^3	0.35 × 0.03 × 0.03
2Θ range for data collection	6.994 to 145.042°
Index ranges	$-7 \leq h \leq 7$, $-13 \leq k \leq 18$, $-25 \leq l \leq 25$
Reflections collected	9625
Independent reflections	3813[R(int) = 0.0388]
Data/restraints/parameters	3813/0/307
Goodness-of-fit on F^2	1.078
Final R indexes [I>=2σ (I)]	R_1 = 0.0365, wR_2 = 0.0914
Final R indexes [all data]	R_1 = 0.0395, wR_2 = 0.0926
Largest diff. peak/hole / e Å^{-3}	0.17/-0.26
Flack parameter	0.18(17)
Crystallisation solvent system	Methanol (Slow evaporation)

Crystallographic Data for 8•HCl



ORTEP representation of **8•HCl** (co-crystallised with one molecule of water).

Table S3. Crystal data and structure refinement for **8•HCl**.

CCDC number	2281802
Identification code	SYL-06-23_2
Empirical formula	$C_{12}H_{15}ClN_3O_{1.5}$
Formula weight	260.72
Temperature/K	120(1)
Crystal system	monoclinic
Space group	C2
a/Å	12.4988(3)
b/Å	7.09306(15)
c/Å	13.9973(3)
α/°	90
β/°	102.543(2)
γ/°	90
Volume/Å^3	1211.31(4)
Z	4
ρ_{calc}mg/mm^3	1.430
μ/mm^{-1}	2.742
F(000)	548.0
Crystal size/mm^3	0.35 × 0.04 × 0.04
2Θ range for data collection	6.47 to 144.312°
Index ranges	$-15 \leq h \leq 15$, $-8 \leq k \leq 8$, $-17 \leq l \leq 15$
Reflections collected	4201
Independent reflections	1911[R(int) = 0.0306]
Data/restraints/parameters	1911/2/162
Goodness-of-fit on F^2	1.073
Final R indexes [I>=2σ (I)]	R_1 = 0.0281, wR_2 = 0.0734
Final R indexes [all data]	R_1 = 0.0285, wR_2 = 0.0739
Largest diff. peak/hole / e Å^{-3}	0.22/-0.18
Flack parameter	-0.011(11)
Crystallisation solvent system	Methanol/Ethanol (Slow evaporation)