Progress toward a biomimetic synthesis of pegaharmaline A

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SUPPORTING INFORMATION

Contents

2,3-Dimethylpyrrolo[2,1-*b*]quinazoline-1,9-dione (1) ^{1}H NMR (400 MHz, DMSO-d_6)
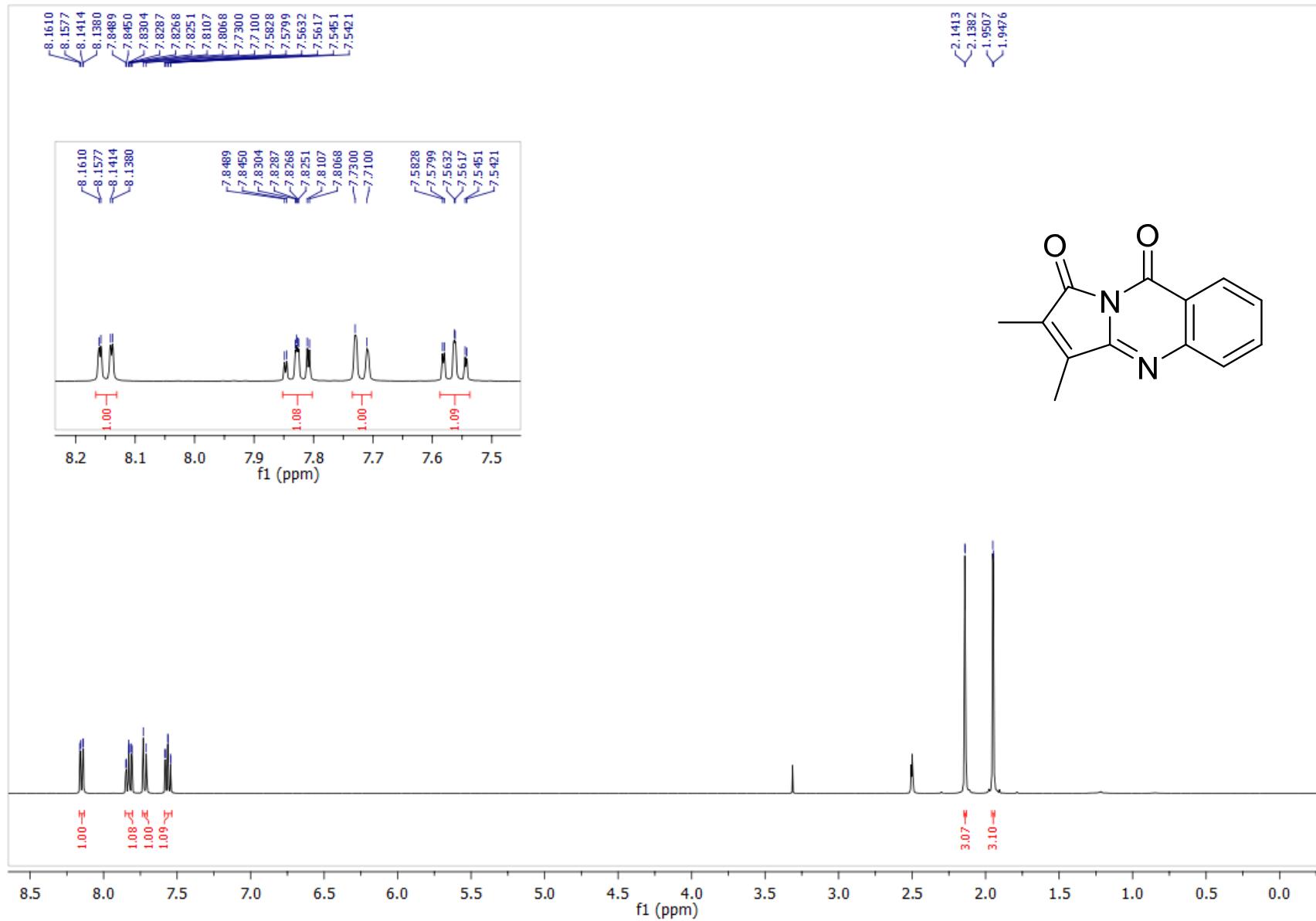
2,3-Dimethylpyrrolo[2,1-*b*]quinazoline-1,9-dione (1) ^{13}C NMR (100 MHz, DMSO-*d$_6$*)

167.0393

155.9233
153.4507

146.2706
142.4722

135.2568
133.8166

128.4881
128.3532
127.2955

122.3845

9.1373
8.4725

f1 (ppm)

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
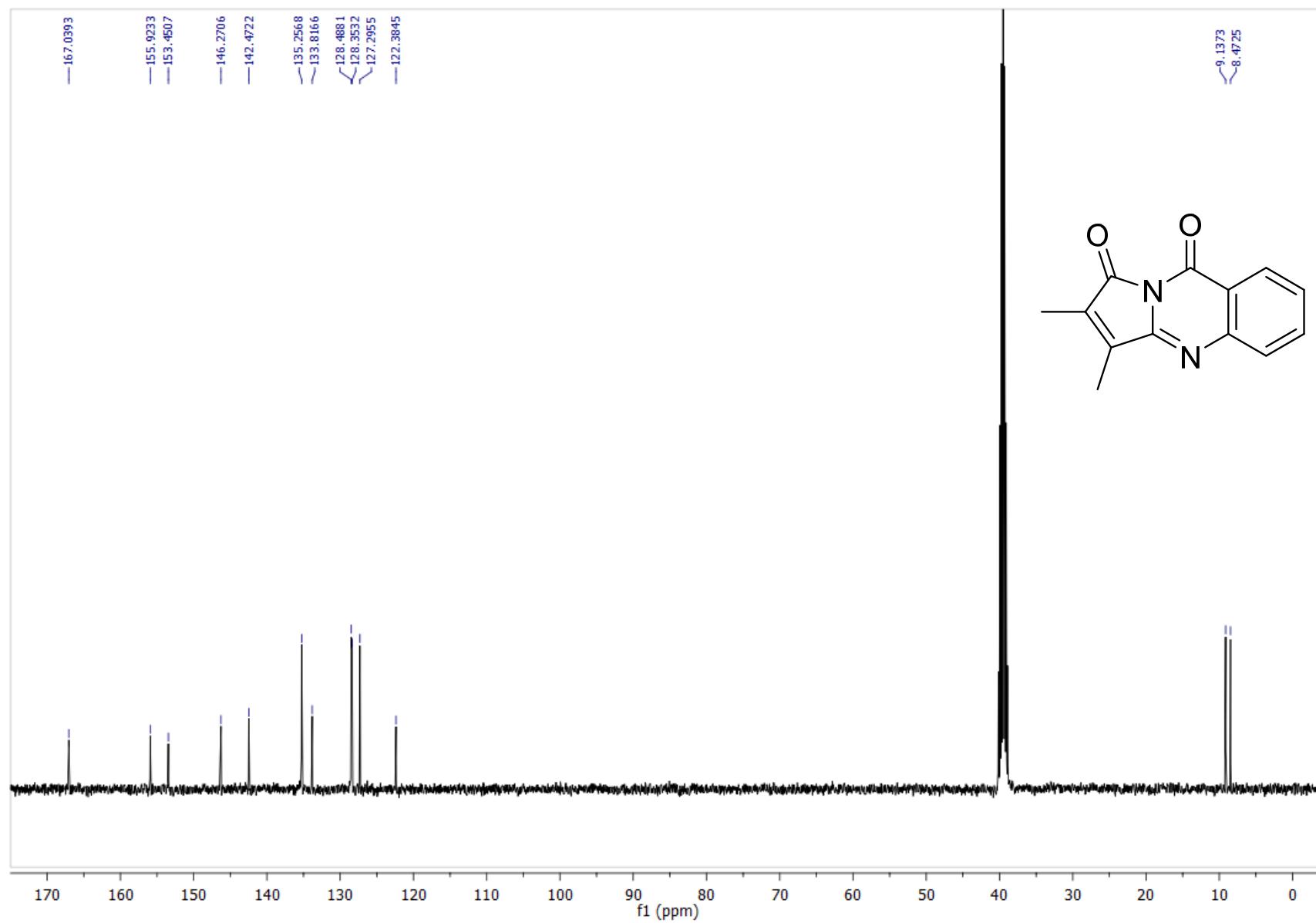
**(±)-1-(2-(6-Methoxy-1*H*-indol-3-yl)ethyl)-3,4-dimethyl-1'*H*-spiro[pyrrole-2,2'-quinazoline]-4',5(1*H*,3'*H*)-dione (7) ¹H NMR (400 MHz, DMSO-*d₆*)**
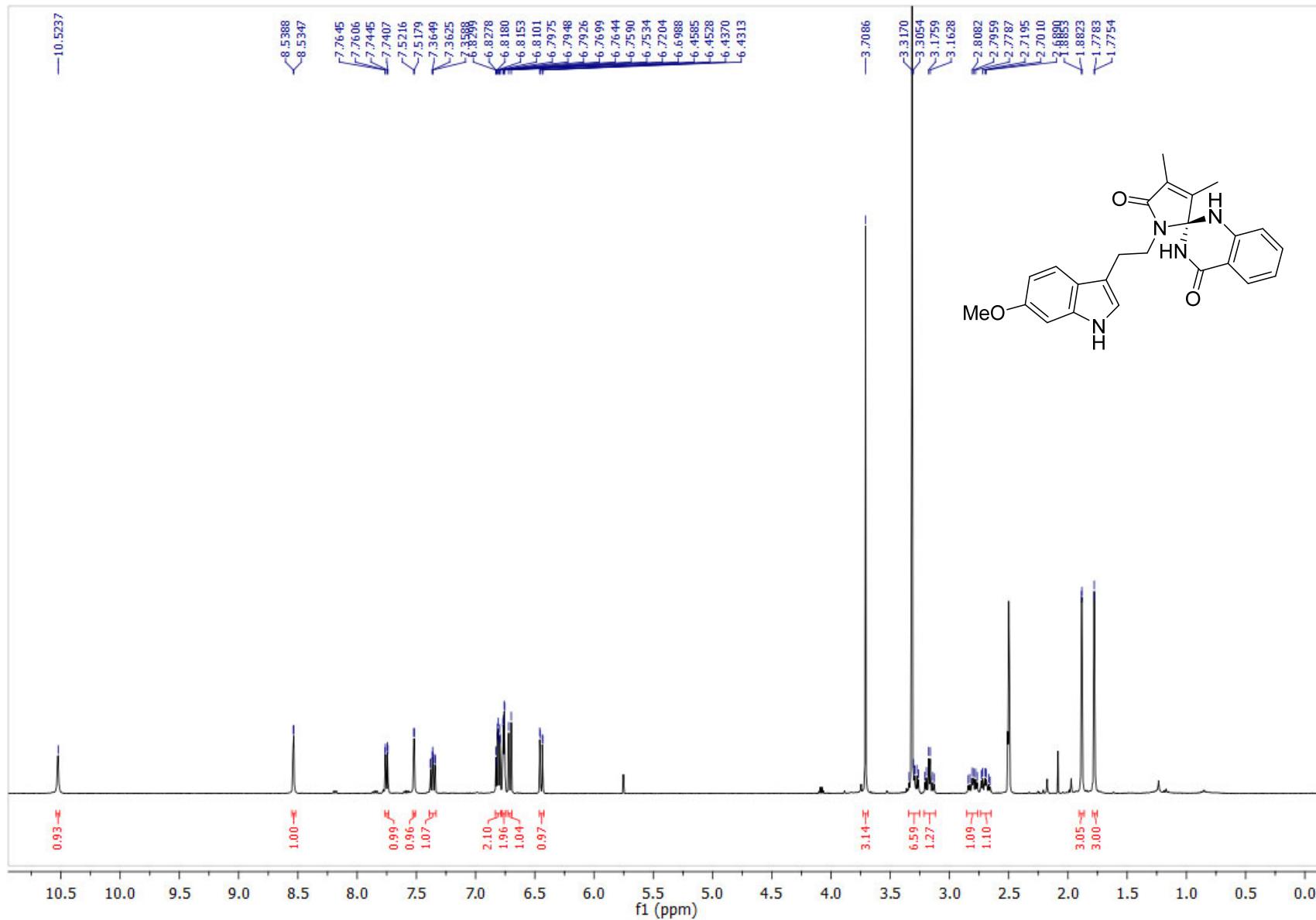
(±)-1-(2-(6-Methoxy-1*H*-indol-3-yl)ethyl)-3,4-dimethyl-1'*H*-spiro[pyrrole-2,2'-quinazoline]-4',5(1*H*,3'*H*)-dione (7) ^{13}C NMR (100 MHz, DMSO-*d₆*)
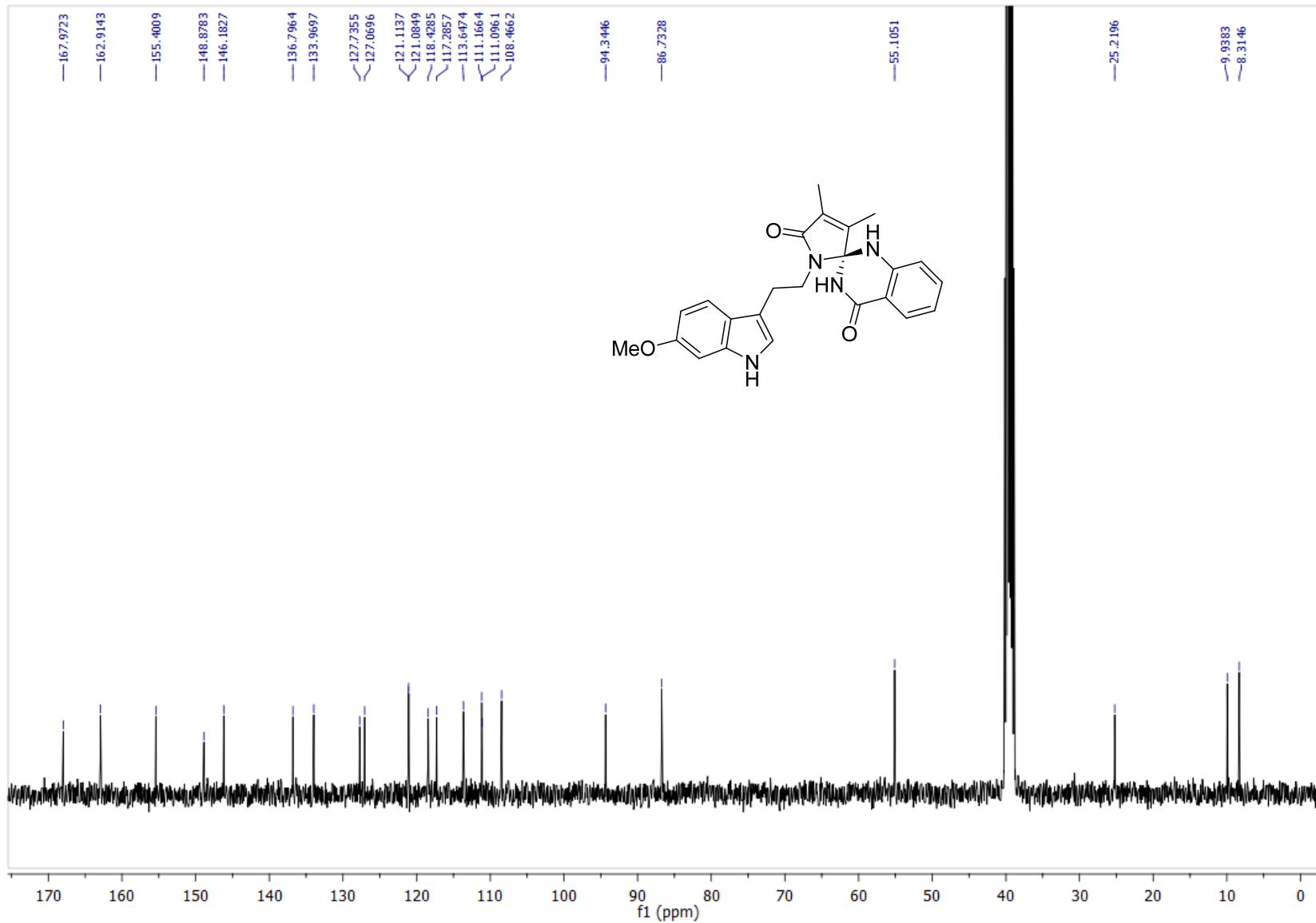
(±)-1-(2-(Indol-3-yl)ethyl)-3,4-dimethyl-1'*H*-spiro[pyrrole-2,2'-quinazoline]-4',5(1*H*,3'*H*)-dione (8) ¹H NMR (400 MHz, DMSO-*d₆*)
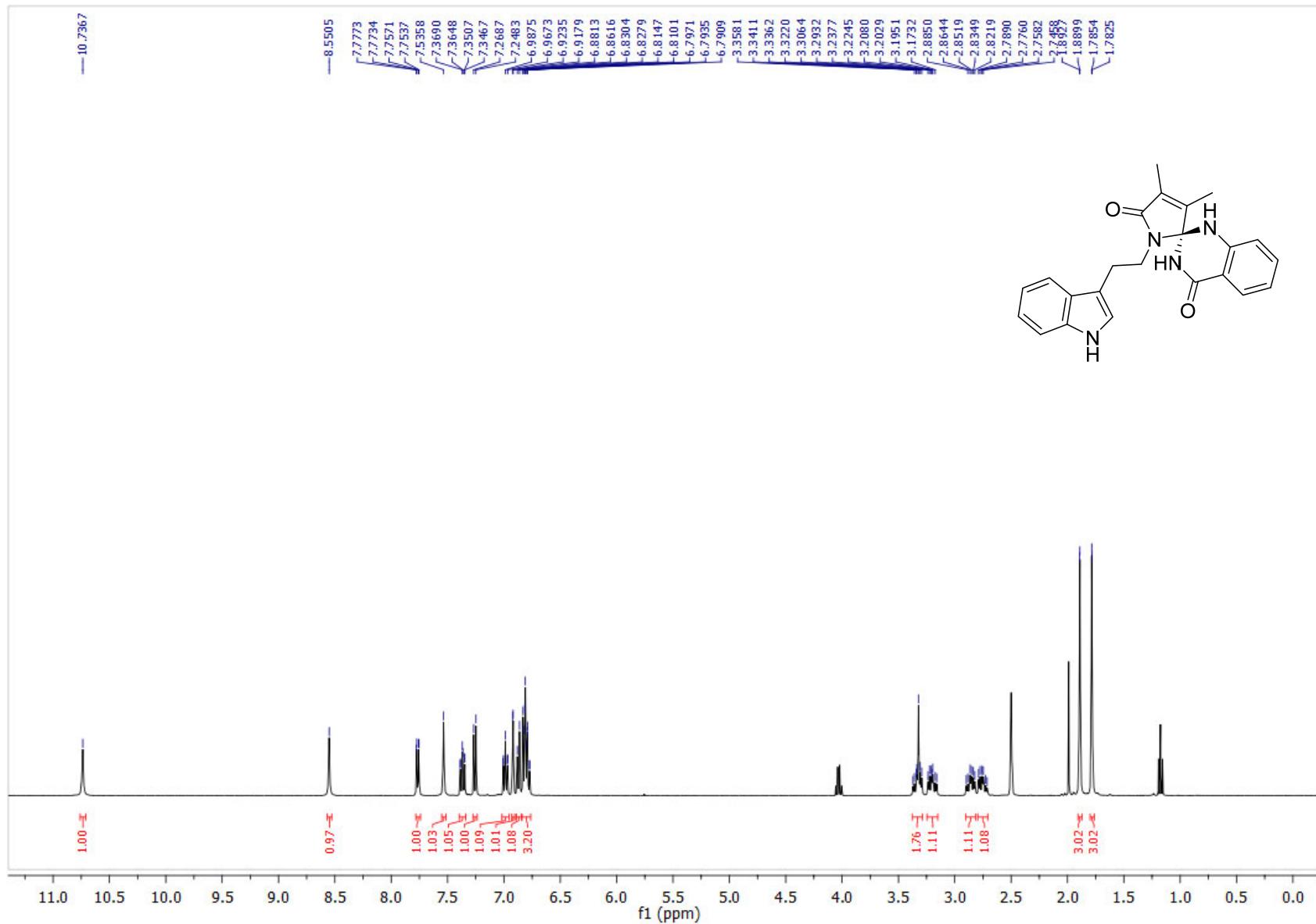
(±)-1-(2-(Indol-3-yl)ethyl)-3,4-dimethyl-1'*H*-spiro[pyrrole-2,2'-quinazoline]-4',5(1*H*,3'*H*)-dione (8) ^{13}C NMR (100 MHz, DMSO-d_6)
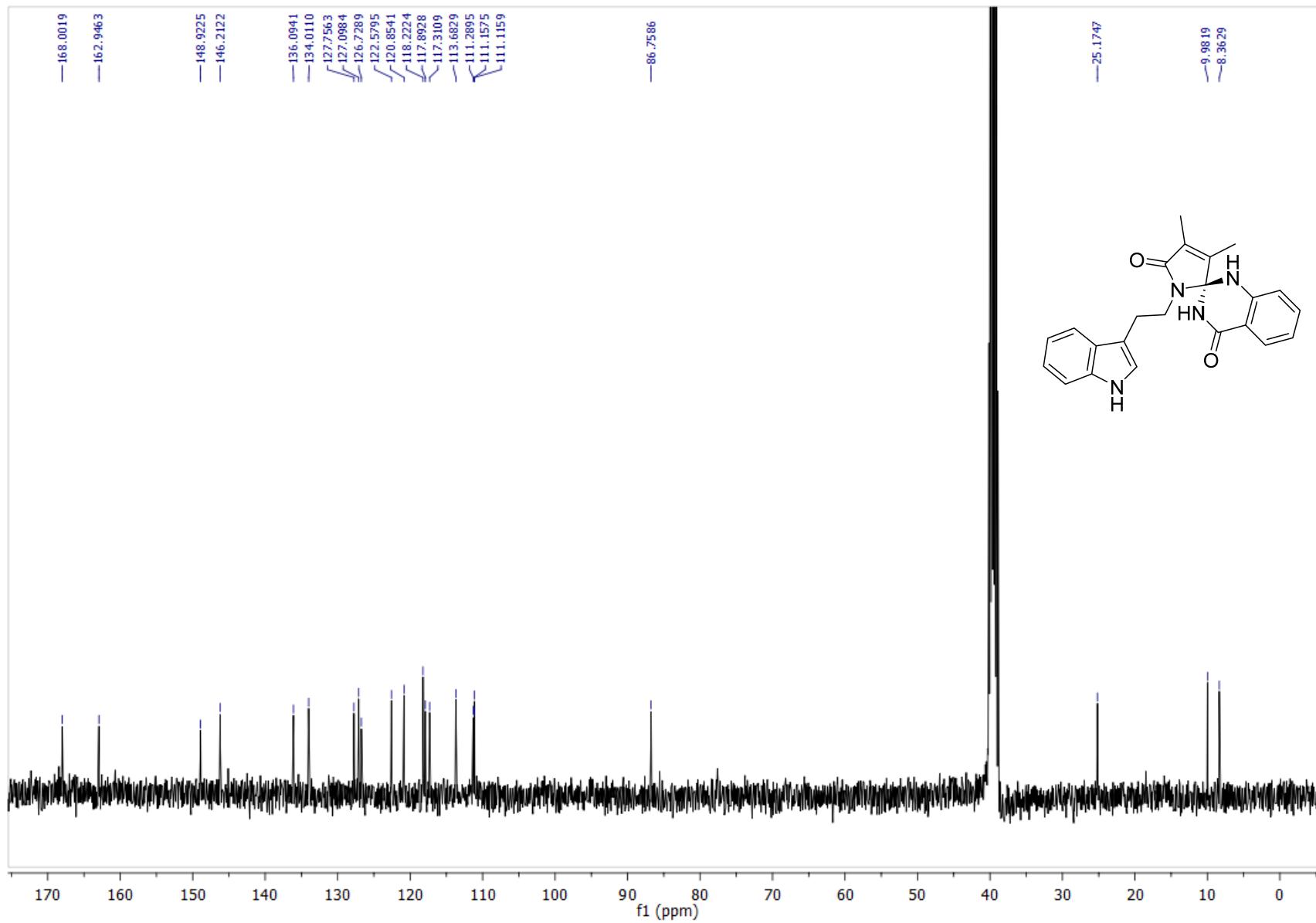
(±)-1-Hydroxy-2,3-dimethylpyrrolo[2,1-*b*]quinazolin-9(1*H*)-one (10) ¹H NMR (400 MHz, DMSO-*d₆*)
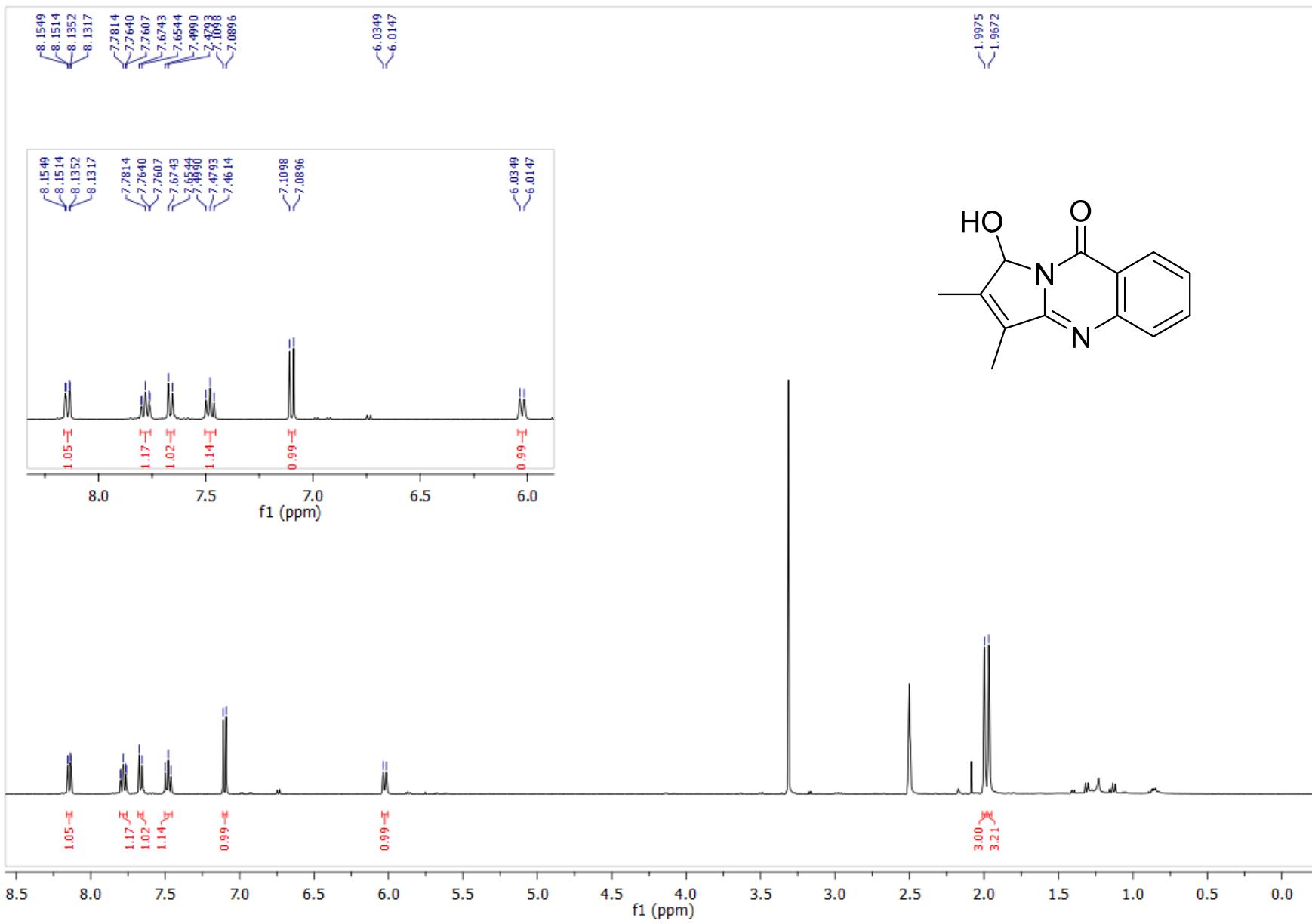
(±)-1-Hydroxy-2,3-dimethylpyrrolo[2,1-*b*]quinazolin-9(1*H*)-one (10) ^{13}C NMR (100 MHz, DMSO-*d₆*)
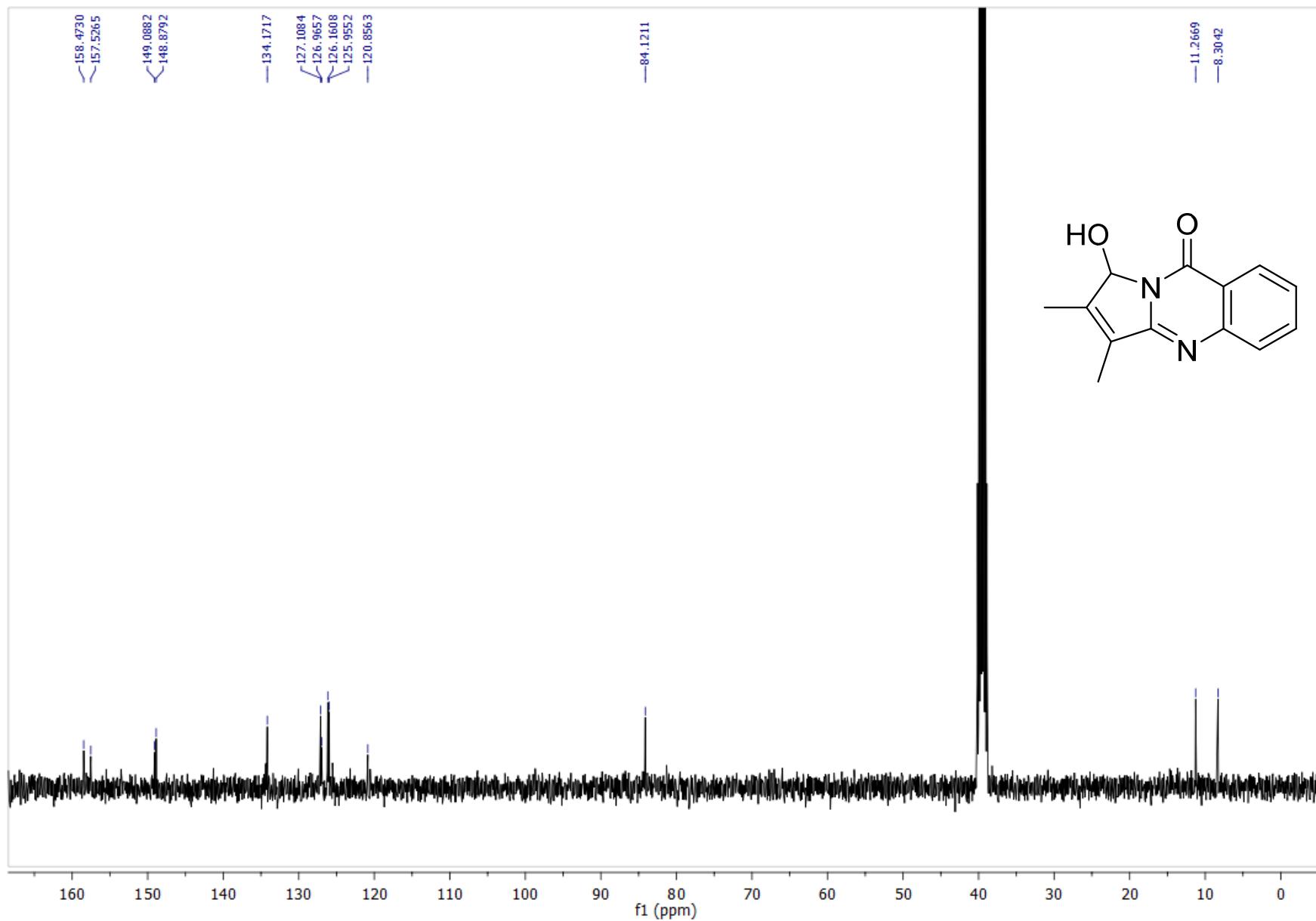
(±)-*N*-(Indol-3-yl)ethyl)-2-methyl-3-(4-oxo-3,4-dihydroquinazolin-2-yl)butanamide (13A) ¹H NMR (400 MHz, DMSO-*d₆*)
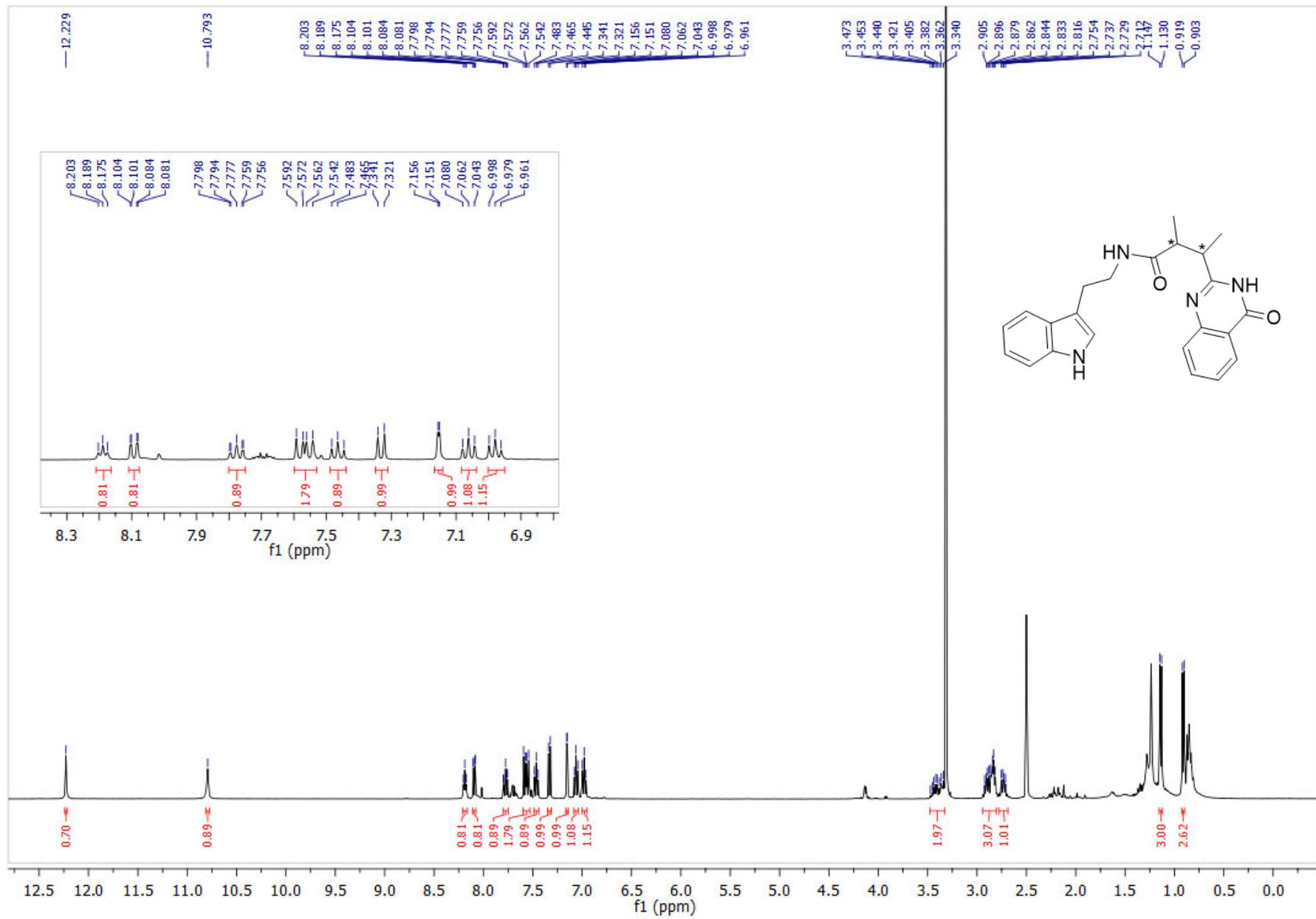
(±)-*N*-(Indol-3-yl)ethyl)-2-methyl-3-(4-oxo-3,4-dihydroquinazolin-2-yl)butanamide (13A) ^{13}C NMR (100 MHz, DMSO-*d₆*)
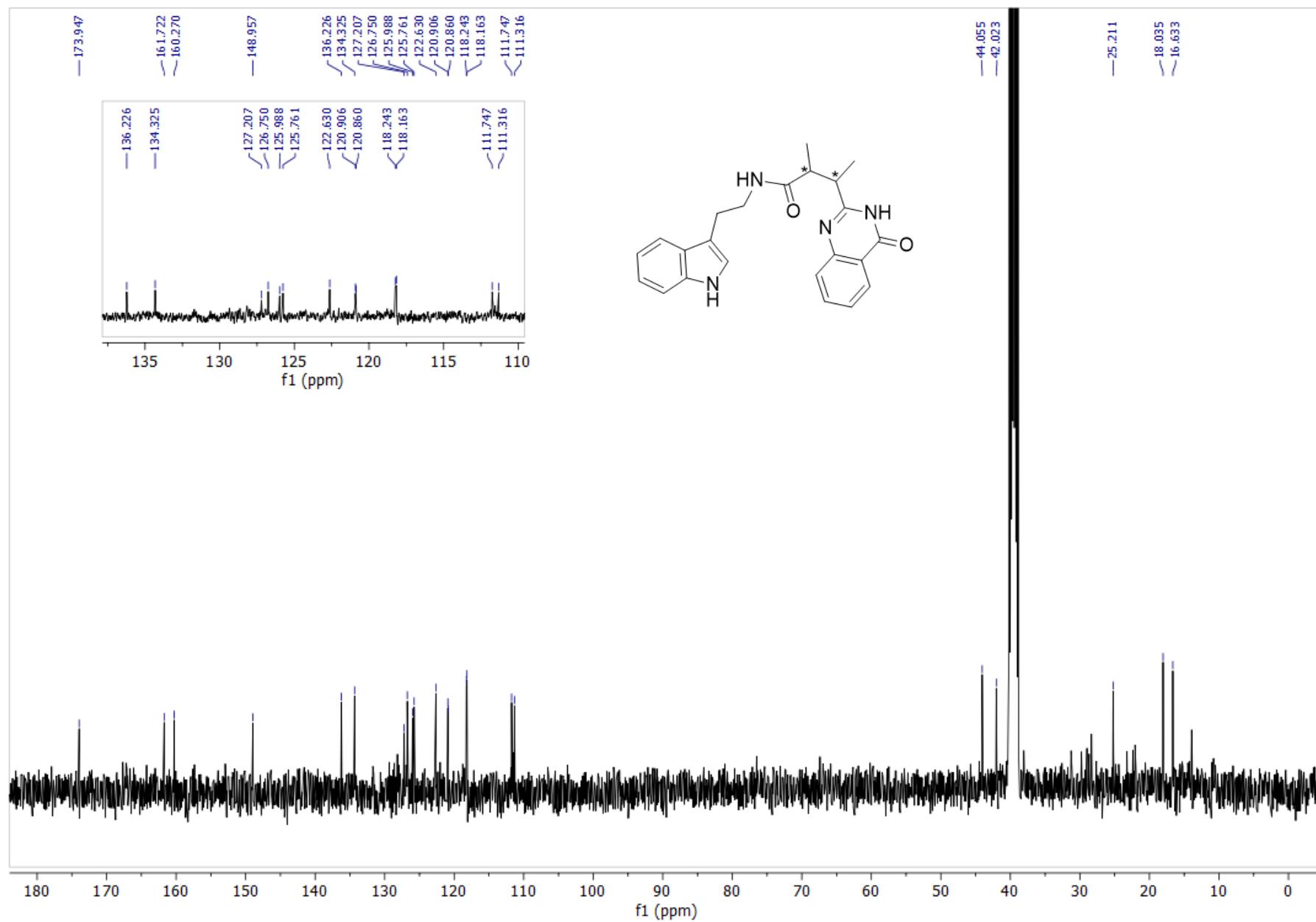
(±)-N-(2-(Indol-3-yl)ethyl)-2-methyl-3-(4-oxo-3,4-dihydroquinazolin-2-yl)butanamide (13B) ¹H NMR (400 MHz, DMSO-d₆)
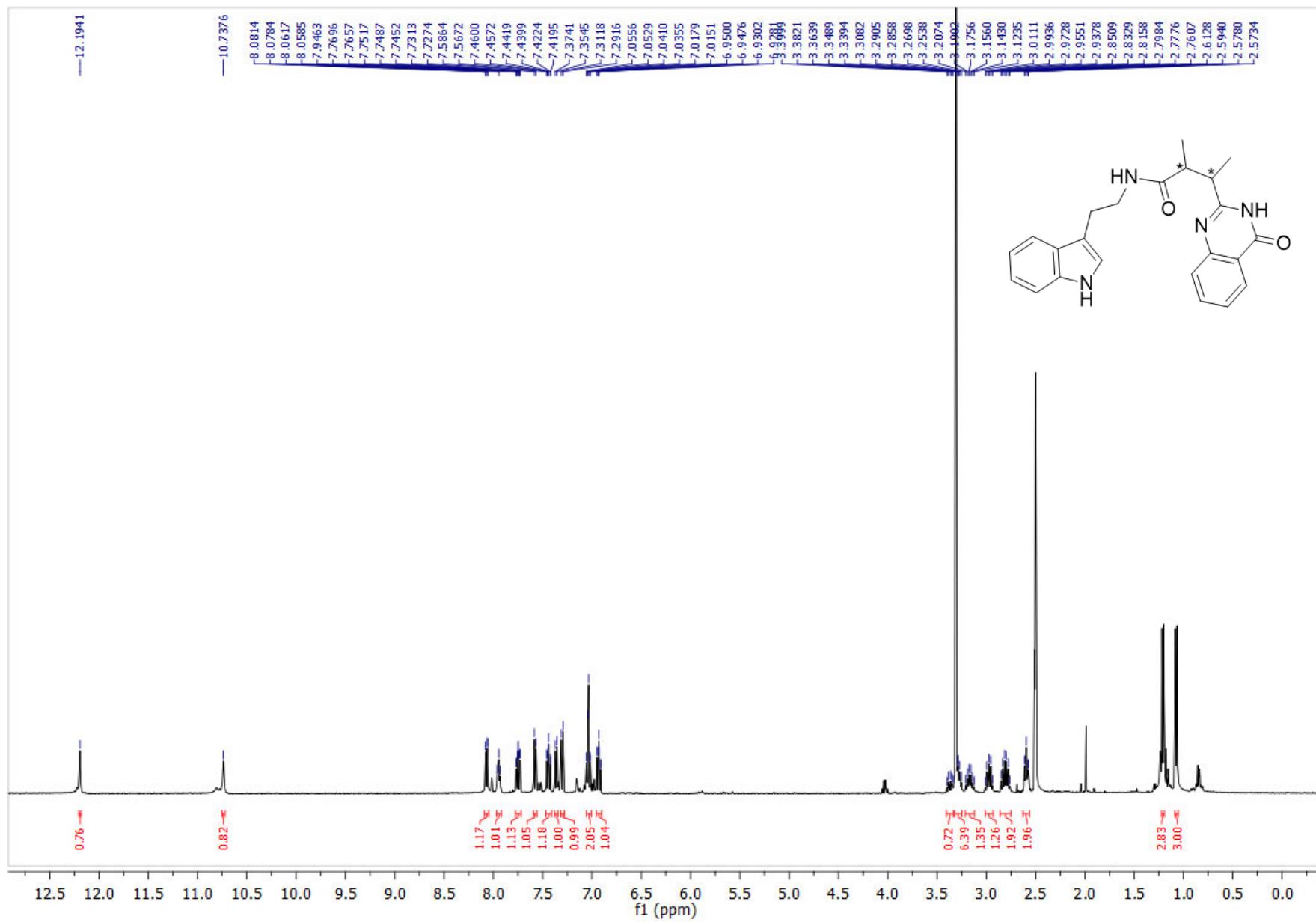
**(±)-*N*-(2-(Indol-3-yl)ethyl)-2-methyl-3-(4-oxo-3,4-dihydroquinazolin-2-yl)butanamide (13B) ¹³C NMR (100 MHz, DMSO-*d₆*)**
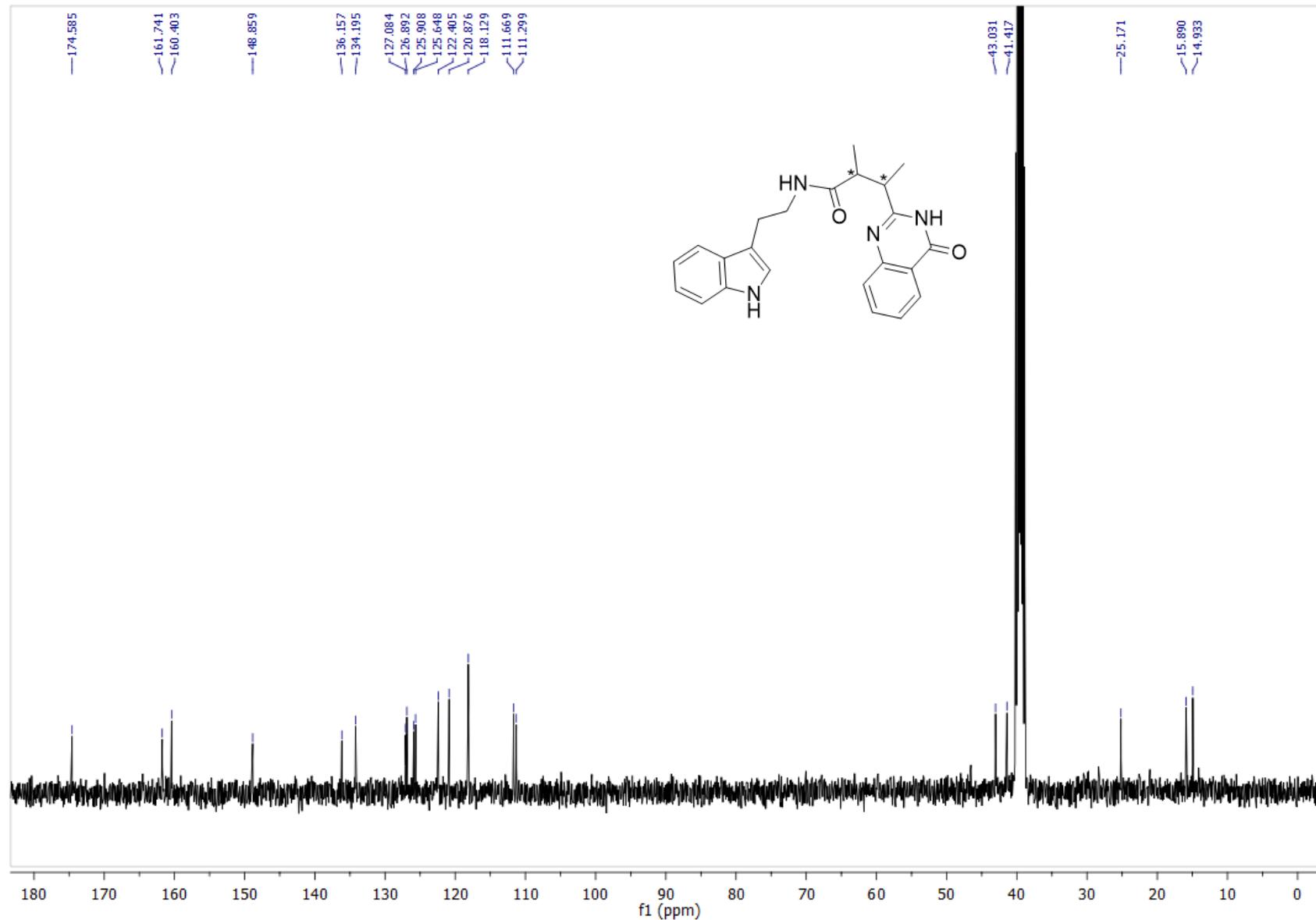
(±)-2-(2-(2,3,4,9-Tetrahydropyrido[3,4-b]indol-1-yl)ethyl)quinazolin-4 (3H)-one (23) ^{1}H NMR (300 MHz, DMSO-d_6)
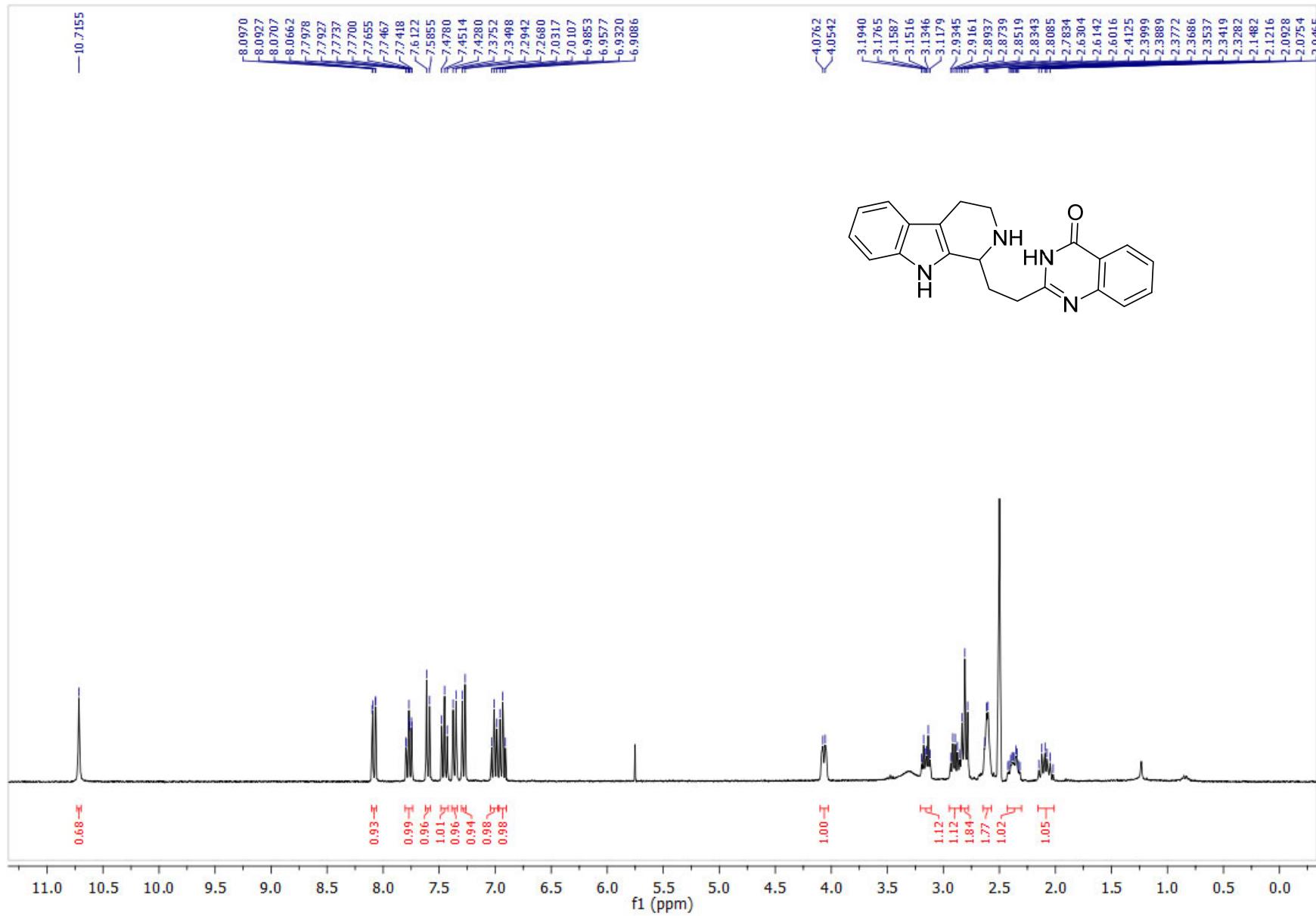
(±)-2-(2-(2,3,4,9-Tetrahydropyrido[3,4-b]indol-1-yl)ethyl)quinazolin-4(3H)-one (23) ^{13}C NMR (75 MHz, DMSO-d_6)
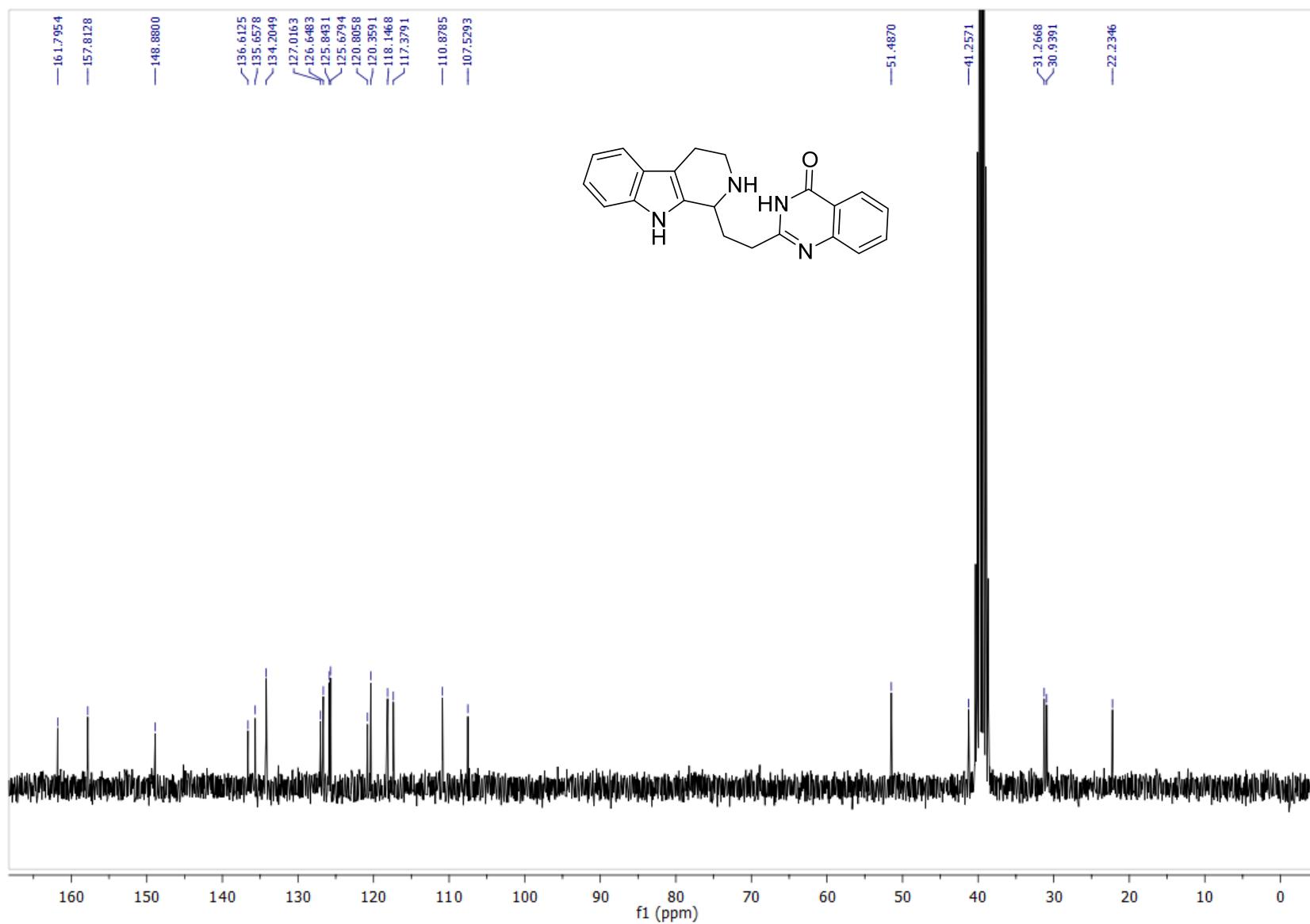
XRD data table

	(±)-7	10	23
CCDC number	1859965	1859974	2087701
Empirical formula	$C_{23}H_{22}N_4O_2$	$C_{13}H_{12}N_2O_2$	$C_{21}H_{20}N_4O$
Formula weight	386.44	228.25	344.41
Temperature/K	109(2)	99.9(5)	100.0(1)
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/n$
a/Å	12.0831(2)	15.7052(8)	13.5402(3)
b/Å	9.0813(2)	5.0360(2)	7.8769(2)
c/Å	18.4230(3)	13.3544(6)	16.4206(4)
α/°	90	90	90
β/°	102.333(2)	90.211(4)	105.285(2)
γ/°	90	90	90
Volume/Å³	1974.91(6)	1056.21(8)	1689.38(7)
Z	4	4	4
ρ_{calc}g/cm³	1.300	1.435	1.354
μ/mm⁻¹	0.685	0.807	0.686
F(000)	816	480.0	728.0
Crystal size/mm³	0.32 × 0.14 × 0.1	0.16 × 0.14 × 0.1	0.05 × 0.05 × 0.01
Radiation	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	12.298 to 135.474	11.268 to 135.436	7.552 to 135.346
Index ranges	$-14 \leq h \leq 14, -10 \leq k \leq 9, -22 \leq l \leq 21$	$-17 \leq h \leq 18, -6 \leq k \leq 6, 16 \leq l \leq 15$	$-16 \leq h \leq 15, -9 \leq k \leq 9, -19 \leq l \leq 19$
Reflections collected	13539	8761	19105
Independent reflections	3567 [R_{int} = 0.0341, R_{sigma} = 0.0297]	1894 [R_{int} =0.0308 R_{sigma} = 0.0228]	3053 [R_{int} = 0.0602, R_{sigma} = 0.0344]
Data/restraints/parameters	3567/0/272	1894/0/157	3053/0/243
Goodness-of-fit on F²	1.067	1.071	1.056
Final R indexes [I>=2σ (I)]	R_1 = 0.0366, wR_2 = 0.0941	R_1 = 0.0544, wR_2 = 0.1524	R_1 = 0.0447, wR_2 = 0.1213
Final R indexes [all data]	R_1 = 0.0418, wR_2 = 0.0974	R_1 = 0.0565, wR_2 = 0.1545	R_1 = 0.0473, wR_2 = 0.1244
Largest diff. peak/hole / e Å⁻³	0.18/-0.22	0.31/-0.36	0.23/-0.38