Electronic Supplementary Material (ESI) for Chemical Science.
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1 General Information

Commercially available chemicals were obtained from Adamas, Acros Organics, Aldrich Chemical Co., Alfa Aesar and TCI and used as received unless otherwise stated. Anhydrous solvent, purchased from Adamas and J&K Chemical, were used as received. (*R*)-STRIP (production batch: 134900) purchased from Daicel Chiral Technologies (China) Co., Ltd. $[Ir(dF(CF_3)ppy)_2(dtbpy)]PF_6$ (production batch: BRY343) purchased from Bide Pharmaceutical Co. Ltd. All reactions were carried out using 10 mL schlenk tube at Ar atmosphere unless otherwise stated. TLC were performed on silica gel Huanghai HSGF254 plates and visualization of the developed chromatogram was performed by fluorescence quenching (λ_{max}= 254 nm). Flash chromatography was carried out on SiO_2 (silica gel 60, 200-300 mesh). Melting points are corrected and recorded using digital Büchi Melting Point Apparatus B540.

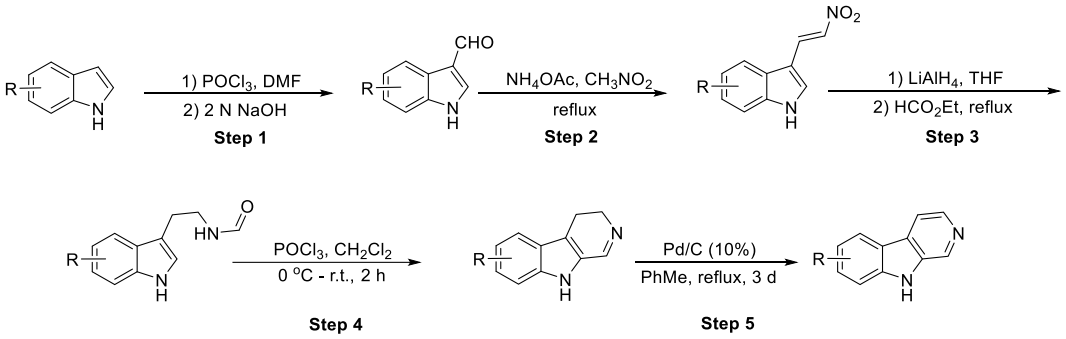
All the ^1H and ^{13}C NMR were recorded on a Bruker Avance II-400 MHz spectrometer. Solvent used for spectra was DMSO-d_6 or chloroform-*d*. ^1H chemical shifts are reported in ppm on the δ-scale relative to TMS (δ 0.00), chloroform-*d* (δ 7.26) or DMSO-d_6 (δ 2.50), and ^{13}C NMR are reported in ppm relative to chloroform-*d* (δ 77.16) or DMSO-d_6 (δ 39.52). Data for ^1H NMR are recorded as follows: chemical shift (δ, ppm) and multiplicity [s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, brs = broad singlet, coupling constant (s) in Hz, integration]. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). IR spectra were recorded on an INVENIO® FT-IR spectrometer. ESI-HRMS data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source and controlled by Xcalibur software. Enantiomeric excess (ee) value was determined by high-performance liquid chromatography (HPLC) analysis using a chiral stationary phase on Agilent Technologies 1260 Infinity II instrument in comparison with the authentic racemates. All the chiral stationary phases including Chiral NX (2), MX (2) and ND (2) used were purchased from Guangzhou FLM Scientific Instrument Co. Ltd. Optical rotations were measured using a Rudolph Autopol I polarimeter.

2 Procedure for the preparation of starting materials

2.1 General procedure for the preparation of Redox-Active Esters[1-3]

To a round bottom flask, *N*-acetyl-protected amino acid (10 mmol, 1.0 equiv.), *N*-hydroxyphthalimide (12 mmol, 1.2 equiv.) and DMAP (1.2 mmol, 0.1 equiv.) were dissolved in anhydrous CH_2Cl_2 or THF (40 mL). Then a solution of N, N′-dicyclohexylcarbodiimide (12 mmol, 1.2 equiv.) in THF or CH_2Cl_2 (10 mL) was added slowly at room temperature. The mixture was allowed to stir until all the acid was consumed (as indicated by TLC). The resulting mixture was quickly filtered and the solid residue was rinsed with more CH_2Cl_2. The filtrate was concentrated in vacuo and the products were recrystallized from CH_2Cl_2/*n*-hexane.

2.2 General procedure for the preparation of β-Carbolines[4-6]



Step 1: To a three-necked flask were added indole (1.0 equiv.) and DMF (1.0 M). Stir the mixture and cool to 0 °C, then freshly distilled $POCl_3$ (1.2 equiv.) was added slowly to the mixture in 15 min. Heat the solution at 40 °C for another 2 h, then 2 N NaOH aqueous solution was added slowly to the mixture, heat the mixture at 90 °C for another 1 h. Add EtOAc to the mixture to dissolve the solid and extract the aqueous layer with EtOAc, then wash the combined organic phase with brine. Dry the organic phase over Na_2SO_4, then separate the organic phase and evaporate under reduced pressure to obtain the product.

Step 2: To a round bottom flask were added aldehyde (1.0 equiv.), ammonium acetate (0.7 equiv.) and nitromethane (15 mL/g of aldehyde), then the reaction mixture was refluxed for 1-2 h. The solvent was removed in vacuum and the residue was washed with water, filtered and dried over infrared light to furnish the desired nitro-olefin. The crude nitro-olefin was used directly for further transformation without purification.

Step 3: Under an inert Ar atmosphere, a tetrahydrofuran solution of lithium aluminum hydride (6.0

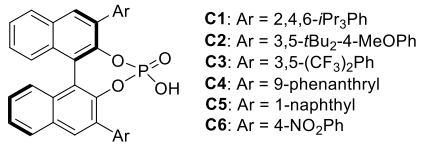
equiv., 1.0 M) was added to a tetrahydrofuran solution of nitro-olefin (1.0 equiv.) at -78 ºC. The resulting mixture was allowed to stirred for another 16 h. The reaction was quenched by dropwise addition of water until effervescence ceased. The mixture was then diluted with ethyl acetate before addition of saturated aqueous solution of Rochelle's salt and the subsequent biphasic mixture was stirred for 24 h. The organic layer was separated, aqueous phase was extracted with ethyl acetate, The organic layers were combined and washed with brine, dried over anhydrous Na_2SO_4, and concentrated to dryness. The remaining residue was purified by flash column chromatography on basic alumina (200−300 mesh) using CH_2Cl_2 and MeOH as eluents to give the corresponding tryptamines Then a mixture of the tryptamine in HCO_2Et (1.0 M) was refluxed for 16 h. The solvent was removed in vacuum. Without further purification, the corresponding N-(2-(1*H*-indol-3-yl)ethyl)-formamide was obtained.

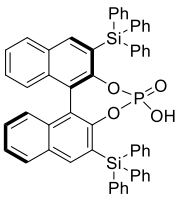
Step 4: To a round bottom flask were added *N*-formyltryptamine (10 mmol.) and CH_2Cl_2 (10 mL), cooled to 0 ºC, then freshly distilled $POCl_3$ (2.5 mL) was added slowly to the above solution. After the addition, stir the reaction mixture at room temperature for another 2 h. Concentrate in vacuo to remove unconsumed $POCl_3$ and CH_2Cl_2. Suspend the dark solid residue in ethyl acetate (50 mL) and extract with 10% AcOH/water (4 x 50 mL). Basify the combined AcOH with conc. aquous ammonia until pH = 9. Extract the precipitated solid with CH_2Cl_2 (3 x 30 mL), the organic layers were combined and washed with brine, dried over anhydrous Na_2SO_4, and concentrated to dryness.to obtain 4,9-dihydro-3*H*-pyrido[3,4-*b*]indole.

Step 5: To a round bottom flask were added 10% Pd/C (75.0 mg/mmol), 4,9-dihydro-3*H*-pyrido[3,4-*b*]indole (2.0 mmol) and toluene (15 mL), then the reaction mixture was refluxed for 3 days. The solution was filtrated and the residue was washed with a CH_2Cl_2: MeOH (9:1) solution three times, and concentrated to dryness. The remaining residue was purified by flash column chromatography on basic alumina (200−300 mesh) using *n*-hexane and EtOAc as eluents to give the corresponding products.

3 Optimization of reaction conditions

Table S1. Optimization of Reaction Conditions of **2a** to **1a**[a]





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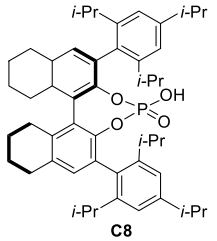

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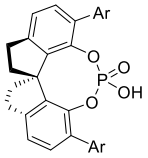

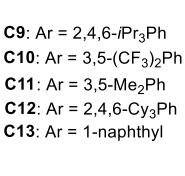

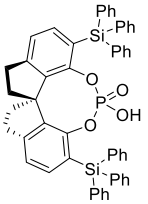

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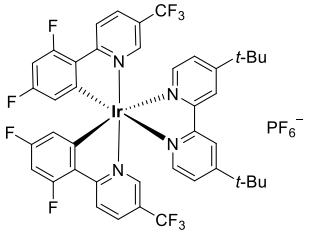


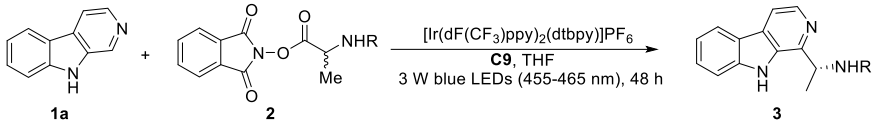
[Ir(dF(CF3)PPy)2(dtbpy)]PF6

Entry	Solvent (mL)	**CPA** (mol%)	*T* (°C)	Yield (%)[b]	ee (%)[c]
1	Dioxane (2.0)	**C1** (5)	25	92	0
2	Dioxane (2.0)	**C2** (5)	25	58	19
3	Dioxane (2.0)	**C3** (5)	25	83	41
4	Dioxane (2.0)	**C4** (5)	25	52	0
5	Dioxane (2.0)	**C5** (5)	25	66	0
6	Dioxane (2.0)	**C6** (5)	25	43	0
7	Dioxane (2.0)	**C7** (5)	25	56	43
8	Dioxane (2.0)	**C8** (5)	25	81	0
9	Dioxane (2.0)	**C9** (5)	25	91	68
10	Dioxane (2.0)	**C10** (5)	25	81	46
11	Dioxane (2.0)	**C11** (5)	25	69	9
12	Dioxane (2.0)	**C12** (5)	25	83	58
13	Dioxane (2.0)	**C13** (5)	25	80	23
14	Dioxane (2.0)	**C14** (5)	25	52	28
15	CH_2Cl_2 (2.0)	**C9** (5)	25	68	59
16	Et_2O (2.0)	**C9** (5)	25	89	76
17	CH_3CN (2.0)	**C9** (5)	25	67	44

18	DCE (2.0)	**C9** (5)	25	67	47
19	Toluene (2.0)	**C9** (5)	25	91	56
20	THF (2.0)	**C9** (5)	25	90	80
21	CPME (2.0)	**C9** (5)	25	89	77
22	MTBE (2.0)	**C9** (5)	25	91	78
23	Isopropyl ether (2.0)	**C9** (5)	25	74	73
24	2-Me-THF (2.0)	**C9** (5)	25	91	76
25	*t*-Bu-benzene (2.0)	**C9** (5)	25	84	56
26	DME (2.0)	**C9** (5)	25	92	59
27	Benzene (2.0)	**C9** (5)	25	67	31
28	Chlorobenzene (2.0)	**C9** (5)	25	78	41
29	EtOAc (2.0)	**C9** (5)	25	74	73
30	*n*-Hexane (2.0)	**C9** (5)	25	0	-
31	Cyclohexane (2.0)	**C9** (5)	25	0	-
32	THF (2.0)	**C9** (5)	5	91	83
33	THF (2.0)	**C9** (5)	-20	88	88
34	THF (2.0)	**C9** (5)	-40	85	91
35	THF (2.0)	**C9** (10)	-40	86	94
36[d]	THF (4.0)	**C9** (10)	-40	86	94

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), $[Ir(dF(CF_3)ppy)_2(dtbpy)]PF_6$ (1 mol%), 48 h. [b]Yield of isolated product. [c]Determined by HPLC analysis on a chiral stationary phase. [d]Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), 72 h. DCE = 1,2-dichloroethane. THF = tetrahydrofuran. CPME = cyclopentyl methyl ether. MTBE = tert-Butyl methyl ether. DME = 1,2-dimethoxy-ethan. 2-Me-THF = 2-methyltetrahydrofuran.

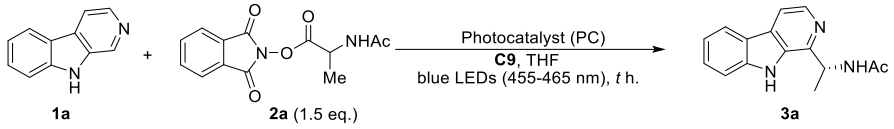
Table S2. Optimization of Reaction Conditions of **2** to **1a**[a]

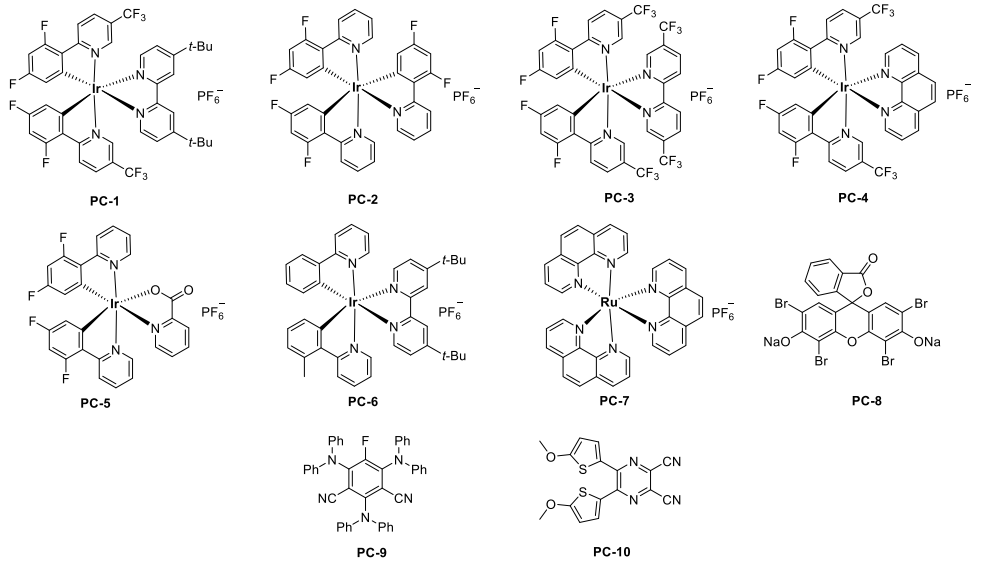


Entry	R	**3**	*T* (°C)	Yield (%)[b]	ee (%)[c]
1	Ac	**3a**	25	90	80
2	Bz	**3ab**	25	60	34
3	Boc	**3ac**	25	65	7
4	Cbz	**3ad**	25	82	26
5	formyl	**3ae**	25	81	52

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), [Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$ (1 mol%), **C9** (5 mol%), THF (2.0 mL). [b]Yield of isolated product. [c]Determined by HPLC analysis on a chiral stationary phase.

Table S3. Screening of Photocatalysist and Blue LEDs[a]

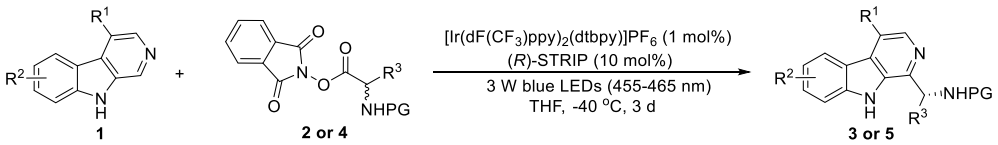




Entry	PC (mol%)	*T* (ºC)	*t* (h)	Yield (%)[b]	ee (%)[c]
1	**PC-1** (1)	-40	48	86	94%
2	**PC-2** (1)	-40	48	72	59%
3	**PC-3** (1)	-40	48	0	ND
4	**PC-4** (1)	-40	48	0	ND
5	**PC-5** (1)	-40	48	0	ND
6	**PC-6** (1)	-40	48	73	69%
7	**PC-7** (1)	-40	48	47	73%
8	**PC-8** (1)	-40	48	0	ND
9	**PC-9** (1)	-40	48	0	ND
10	**PC-10** (1)	-40	48	77	56%
11	**PC-1** (2)	5	48	86	83%
12	**PC-1** (0.5)	5	48	93	82%
13[d]	**PC-1** (1)	-40	48	84	89%
14[e]	**PC-1** (1)	-40	24	82	91%

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **C9** (5 mol%), THF (2.0 mL), 3 W Blue LEDs (455-465 nm). [b]Yield of isolated product. [c]Determined by HPLC analysis on a chiral stationary phase. [d]12 W Syn LEDs (455-465 nm). [e]40 W Kessil Blue LEDs (455-465 nm).

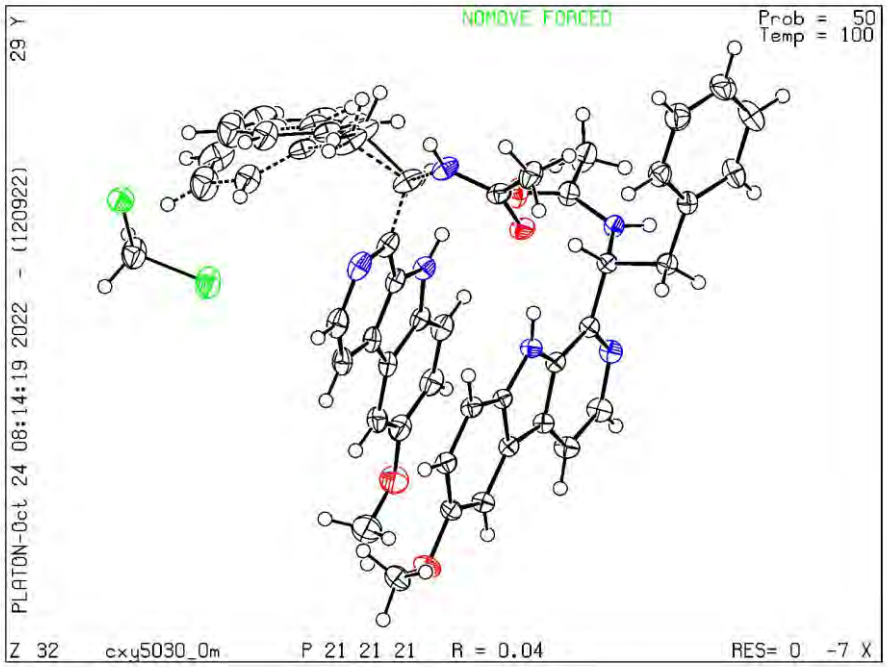
4 General procedure for Enantioselective Minisci reactions between α-amino acid-derived RAEs and β-Carbolines



Sequentially, β-Carbolines (0.20 mmol, 1.0 equiv.), redox-active ester (RAEs) (0.30 mmol, 1.5 equiv.), Ir[dF(CF_3)ppy)$_2$(dtbpy)]PF_6 (2.2 mg, 0.002 mmol, 1 mol%), and (*R*)-STRIP (14.4 mg, 0.02 mmol, 10 mol%) were added to a 10 mL Schlenk tube containing a stirrer bar. After evacuated and refilled with argon three times, anhydrous, freshly argon-sparged THF (4.0 mL) was then added via syringe. The reaction mixture was stirred under an argon atmosphere at −40 °C (the temperature was maintained in an incubator) for 1 h without light, then irradiated by 3 W blue LED (λ = 455−465 nm) for another 72 h The reaction was monitored by TLC. After completion of the reaction, the solvent was removed in vacuo and the crude residue was purified via flash column chromatography on silica gel using *n*-hexane/acetone or n-hexane/ethyl acetate as eluents to give the corresponding products **3a-3r** and **5a-5y**.

Incubator and setup of reactions:

5 X-ray data of compound 3n (CCDC number: 2215559)



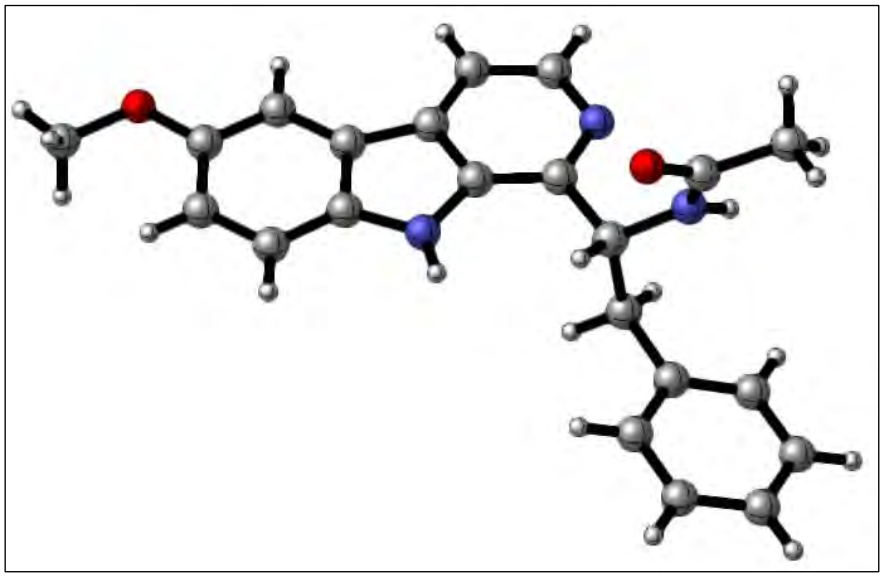


Table S4. Crystal data and structure refinement for cxy5030_0m.

Identification code	cxy5030_0m
Empirical formula	$C_{22.31}H_{21.62}Cl_{0.62}N_3O_2$
Formula weight	385.85
Temperature/K	100.0(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	14.6467(11)
b/Å	14.7349(12)

c/Å	18.2076(14)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3929.5(5)
Z	8
ρ_{calc}g/cm^3	1.304
μ/mm^{-1}	0.935
F(000)	1625.0
Crystal size/mm^3	0.26 × 0.22 × 0.18
Radiation	Ga$K\alpha$ (λ = 1.34138)
2Θ range for data collection/°	6.714 to 147.294
Index ranges	-19 ≤ h ≤ 20, -21 ≤ k ≤ 20, -25 ≤ l ≤ 26
Reflections collected	171070
Independent reflections	12007 [R_{int} = 0.0631, R_{sigma} = 0.0231]
Data/restraints/parameters	12007/15/543
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0364, wR_2 = 0.1036
Final R indexes [all data]	R_1 = 0.0385, wR_2 = 0.1048
Largest diff. peak/hole / e Å^{-3}	0.30/-0.38
Flack parameter	0.025(3)

Table S5. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for cxy5030_0m. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O2	9385.4(10)	-29.0(9)	7073.7(7)	28.6(3)
O1	6391.0(9)	5249.2(11)	5232.5(8)	35.5(3)
N3	8627.3(9)	3513.5(9)	6297.3(7)	20.0(2)
N1	7719.9(9)	5497.9(10)	4621.8(7)	21.1(2)
N2	8477.8(10)	3797.2(10)	4285.8(7)	23.5(3)
C20	9030.4(14)	8348.3(14)	5771.0(13)	36.2(4)
C19	8956.3(14)	7685.4(15)	6308.7(12)	36.1(4)
C18	9027.2(13)	6772.6(13)	6122.7(10)	30.2(3)
C17	9185.0(10)	6510.8(12)	5395.3(9)	22.5(3)
C16	9237.7(10)	5520.3(11)	5192.6(9)	22.8(3)
C3	8296.1(10)	5047.0(11)	5162.2(8)	19.5(3)
C4	8437.0(10)	4050.2(11)	4992.4(8)	19.2(3)
C8	8596.6(10)	3410.4(10)	5544.0(8)	18.4(3)
C7	8788.9(10)	2496.8(10)	5359.6(8)	19.0(3)
C10	8922.5(10)	2020.8(11)	6041.7(8)	18.7(3)
C11	9113.2(11)	1117.2(11)	6211.3(9)	21.6(3)
C12	9196.6(11)	876.3(11)	6946.7(9)	21.4(3)
C13	9539.8(14)	-305.0(13)	7810.8(10)	30.5(4)
C9	8822.2(10)	2672.4(10)	6604.8(8)	18.6(2)
C2	6809.1(11)	5566.5(13)	4700.7(10)	26.7(3)
C23	6322.9(12)	6068.1(17)	4093.3(11)	37.6(5)
C21	9199.9(16)	8096.4(14)	5051.5(12)	37.4(4)
C22	9283.1(13)	7182.8(13)	4865.3(10)	30.9(4)
C5	8671.3(13)	2927.0(13)	4109.6(9)	27.0(3)
C6	8833.8(12)	2254.0(12)	4621.9(9)	24.9(3)
C15	8930.3(11)	2431.1(11)	7339.4(8)	21.7(3)
C14	9116.0(11)	1528.2(11)	7506.3(9)	22.1(3)
O3	8018.1(10)	5103.5(9)	7045.9(7)	30.3(3)
O4	6441.9(11)	440.9(11)	4596.5(9)	39.8(3)
N5	6592.3(10)	3474.1(13)	8133.3(8)	29.1(3)
N4	6930.0(10)	5390.7(11)	7895.4(8)	27.1(3)
N6	6210.6(9)	3657.4(10)	6128.8(7)	22.8(3)
C42	2887(3)	4320(4)	6738(3)	50.3(10)
C41	3043(3)	4097(3)	7468(3)	48.2(8)
C40	3847(2)	4351(3)	7822.2(19)	36.2(5)
C39	4509.9(19)	4844(2)	7449.9(17)	26.0(5)
C38	5316(10)	5175(7)	7850(6)	36.8(18)

Table S5. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for cxy5030_0m. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C25	6229(13)	4900(30)	7490(18)	29.7(16)
C26	6405.7(11)	3883.6(13)	7493.5(9)	24.7(3)
C30	6369.8(10)	3378.1(12)	6842.6(8)	21.9(3)
C29	6506.6(10)	2426.4(12)	6854.0(9)	23.0(3)
C32	6431.3(10)	2119.0(12)	6102.4(9)	23.1(3)
C33	6500.0(12)	1265.6(13)	5767.4(10)	27.6(3)
C34	6389.2(12)	1223.5(13)	5011.0(11)	30.1(3)
C35	6690.7(18)	-357.6(17)	4969.9(15)	46.3(5)
C36	6203.0(12)	2008.5(15)	4592.7(10)	30.3(4)
C37	6137.9(11)	2848.5(13)	4916.5(9)	26.4(3)
C31	6256.6(10)	2900.8(12)	5679.9(8)	22.4(3)
C27	6737.2(12)	2563.8(14)	8140.2(9)	30.4(4)
C28	6697.7(12)	2013.5(14)	7525.7(9)	28.0(3)
C43	3551(2)	4790(2)	6367.2(19)	35.7(6)
C44	4360(2)	5060(2)	6714.2(17)	30.3(5)
C24	7776.8(12)	5459.4(12)	7632.9(9)	25.3(3)
C45	8424.1(13)	6025.6(15)	8084.1(10)	33.1(4)
Cl1	3263.7(5)	2601.8(5)	9386.6(4)	34.58(17)
Cl2	4436.8(6)	2046.6(7)	8170.6(5)	42.3(2)
C1	3370(2)	1902(3)	8611(2)	40.7(8)
C38A	5296(17)	4978(14)	7966(11)	36.8(18)
C39A	4420(3)	4458(4)	7661(3)	26.0(5)
C40A	4090(4)	3684(4)	8032(3)	36.2(5)
C41A	3300(5)	3241(5)	7794(4)	48.2(8)
C42A	2842(4)	3586(6)	7188(4)	50.3(10)
C43A	3160(5)	4304(5)	6805(4)	35.7(6)
C44A	3976(3)	4753(4)	7043(3)	30.3(5)
C25A	6210(20)	4870(50)	7530(30)	29.7(16)

Table S6. Bond Lengths for cxy5030_0m.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O2	C12	1.3817(19)	N4	C24	1.333(2)
O2	C13	1.420(2)	N4	C25A	1.460(18)
O1	C2	1.237(2)	N6	C30	1.3830(19)
N3	C8	1.3808(18)	N6	C31	1.384(2)
N3	C9	1.390(2)	C42	C41	1.388(7)
N1	C3	1.4566(19)	C42	C43	1.372(7)
N1	C2	1.346(2)	C41	C40	1.394(5)
N2	C4	1.3409(19)	C40	C39	1.389(5)
N2	C5	1.352(2)	C39	C38	1.471(14)
C20	C19	1.387(3)	C39	C44	1.394(4)
C20	C21	1.384(3)	C38	C25	1.544(16)
C19	C18	1.391(3)	C25	C26	1.52(5)
C18	C17	1.399(2)	C26	C30	1.401(2)
C17	C16	1.507(2)	C26	C25A	1.49(8)
C17	C22	1.390(2)	C30	C29	1.417(2)
C16	C3	1.546(2)	C29	C32	1.446(2)
C3	C4	1.515(2)	C29	C28	1.394(2)
C4	C8	1.397(2)	C32	C33	1.401(3)
C8	C7	1.416(2)	C32	C31	1.409(2)
C7	C10	1.440(2)	C33	C34	1.388(3)
C7	C6	1.391(2)	C34	C36	1.411(3)
C10	C11	1.395(2)	C36	C37	1.374(3)
C10	C9	1.412(2)	C37	C31	1.403(2)
C11	C12	1.391(2)	C27	C28	1.383(3)
C12	C14	1.405(2)	C43	C44	1.400(4)
C9	C15	1.393(2)	C24	C45	1.507(3)
C2	C23	1.509(2)	Cl1	C1	1.756(4)
C21	C22	1.394(3)	Cl2	C1	1.768(4)
C5	C6	1.382(2)	C38A	C39A	1.59(3)
C15	C14	1.391(2)	C38A	C25A	1.56(2)
O3	C24	1.242(2)	C39A	C40A	1.410(8)
O4	C34	1.380(2)	C39A	C44A	1.372(7)
O4	C35	1.407(3)	C40A	C41A	1.398(8)
N5	C26	1.340(2)	C41A	C42A	1.388(11)
N5	C27	1.358(3)	C42A	C43A	1.350(12)
N4	C25	1.459(11)	C43A	C44A	1.433(8)

Table S7. Bond Angles for cxy5030_0m.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C12	O2	C13	117.80(14)	C40	C39	C38	119.5(5)
C8	N3	C9	107.99(12)	C40	C39	C44	118.6(3)
C2	N1	C3	122.46(14)	C44	C39	C38	121.8(5)
C4	N2	C5	120.06(14)	C39	C38	C25	113.4(9)
C21	C20	C19	119.56(19)	N4	C25	C38	105.2(13)
C20	C19	C18	120.21(19)	N4	C25	C26	112(3)
C19	C18	C17	120.64(18)	C26	C25	C38	114(2)
C18	C17	C16	120.49(15)	N5	C26	C25	118.8(13)
C22	C17	C18	118.55(17)	N5	C26	C30	120.25(17)
C22	C17	C16	120.96(16)	N5	C26	C25A	116(2)
C17	C16	C3	113.56(13)	C30	C26	C25	120.9(13)
N1	C3	C16	109.59(12)	C30	C26	C25A	124(2)
N1	C3	C4	112.54(13)	N6	C30	C26	130.03(16)
C4	C3	C16	108.85(12)	N6	C30	C29	109.40(14)
N2	C4	C3	118.12(13)	C26	C30	C29	120.57(15)
N2	C4	C8	119.64(14)	C30	C29	C32	106.59(14)
C8	C4	C3	122.03(13)	C28	C29	C30	118.24(16)
N3	C8	C4	130.19(14)	C28	C29	C32	135.14(17)
N3	C8	C7	109.49(13)	C33	C32	C29	133.45(17)
C4	C8	C7	120.29(13)	C33	C32	C31	120.61(15)
C8	C7	C10	106.60(13)	C31	C32	C29	105.94(15)
C6	C7	C8	118.87(14)	C34	C33	C32	117.62(17)
C6	C7	C10	134.51(15)	O4	C34	C33	124.97(19)
C11	C10	C7	133.09(14)	O4	C34	C36	113.61(17)
C11	C10	C9	120.60(13)	C33	C34	C36	121.42(17)
C9	C10	C7	106.31(13)	C37	C36	C34	121.33(16)
C12	C11	C10	118.32(14)	C36	C37	C31	117.76(17)
O2	C12	C11	115.16(14)	N6	C31	C32	110.18(14)
O2	C12	C14	123.74(14)	N6	C31	C37	128.56(17)
C11	C12	C14	121.08(15)	C37	C31	C32	121.25(16)
N3	C9	C10	109.58(12)	N5	C27	C28	124.39(16)
N3	C9	C15	129.64(14)	C27	C28	C29	117.53(18)
C15	C9	C10	120.77(14)	C42	C43	C44	121.4(4)
O1	C2	N1	123.07(16)	C39	C44	C43	120.1(3)
O1	C2	C23	121.68(15)	O3	C24	N4	122.77(18)
N1	C2	C23	115.24(15)	O3	C24	C45	121.60(16)
C20	C21	C22	120.32(18)	N4	C24	C45	115.60(14)

Table S7. Bond Angles for cxy5030_0m.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C17	C22	C21	120.68(18)	Cl1	C1	Cl2	111.86(18)
N2	C5	C6	123.81(15)	C25A	C38A	C39A	117.8(16)
C5	C6	C7	117.31(16)	C40A	C39A	C38A	119.8(7)
C14	C15	C9	118.42(14)	C44A	C39A	C38A	121.0(8)
C15	C14	C12	120.78(14)	C44A	C39A	C40A	119.2(5)
C34	O4	C35	116.68(18)	C41A	C40A	C39A	120.8(6)
C26	N5	C27	118.99(16)	C42A	C41A	C40A	118.4(7)
C24	N4	C25	120.7(8)	C43A	C42A	C41A	122.1(6)
C24	N4	C25A	123.4(13)	C42A	C43A	C44A	119.6(6)
C30	N6	C31	107.88(14)	C39A	C44A	C43A	119.8(6)
C43	C42	C41	118.3(4)	N4	C25A	C26	113(4)
C42	C41	C40	121.2(4)	N4	C25A	C38A	110(2)
C39	C40	C41	120.3(3)	C26	C25A	C38A	107(4)

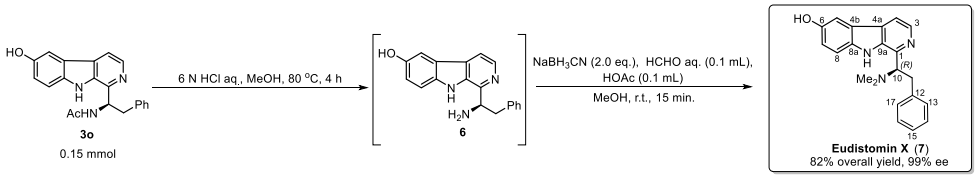
Table S8. Torsion Angles for cxy5030_0m.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O2	C12	C14	C15	179.59(15)	C41	C42	C43	C44	-1.4(6)
N3	C8	C7	C10	1.33(16)	C41	C40	C39	C38	174.6(6)
N3	C8	C7	C6	-177.11(14)	C41	C40	C39	C44	-1.3(5)
N3	C9	C15	C14	179.31(15)	C40	C39	C38	C25	127(2)
N1	C3	C4	N2	33.90(19)	C40	C39	C44	C43	0.7(4)
N1	C3	C4	C8	-151.32(13)	C39	C38	C25	N4	172.0(19)
N2	C4	C8	N3	177.19(15)	C39	C38	C25	C26	-65(3)
N2	C4	C8	C7	-0.8(2)	C38	C39	C44	C43	-175.2(6)
N2	C5	C6	C7	0.3(3)	C38	C25	C26	N5	-70.3(18)
C20	C19	C18	C17	0.8(3)	C38	C25	C26	C30	109.3(19)
C20	C21	C22	C17	0.9(3)	C25	N4	C24	O3	-1(3)
C19	C20	C21	C22	0.9(3)	C25	N4	C24	C45	177(2)
C19	C18	C17	C16	-178.44(17)	C25	C26	C30	N6	1.9(7)
C19	C18	C17	C22	1.0(3)	C25	C26	C30	C29	-178.0(7)
C18	C17	C16	C3	75.95(19)	C26	N5	C27	C28	-0.7(3)
C18	C17	C22	C21	-1.8(3)	C26	C30	C29	C32	-179.68(13)
C17	C16	C3	N1	59.37(17)	C26	C30	C29	C28	-1.4(2)
C17	C16	C3	C4	-177.16(13)	C30	N6	C31	C32	1.00(17)
C16	C17	C22	C21	177.60(17)	C30	N6	C31	C37	179.74(15)
C16	C3	C4	N2	-87.79(16)	C30	C26	C25A	N4	-128(3)
C16	C3	C4	C8	87.00(16)	C30	C26	C25A	C38A	111(3)
C3	N1	C2	O1	-0.5(3)	C30	C29	C32	C33	179.95(17)
C3	N1	C2	C23	179.11(17)	C30	C29	C32	C31	0.19(16)
C3	C4	C8	N3	2.5(2)	C30	C29	C28	C27	0.3(2)
C3	C4	C8	C7	-175.52(13)	C29	C32	C33	C34	-179.79(16)
C4	N2	C5	C6	0.2(3)	C29	C32	C31	N6	-0.73(17)
C4	C8	C7	C10	179.71(13)	C29	C32	C31	C37	-179.58(14)
C4	C8	C7	C6	1.3(2)	C32	C29	C28	C27	177.88(17)
C8	N3	C9	C10	0.29(17)	C32	C33	C34	O4	179.54(16)
C8	N3	C9	C15	179.42(15)	C32	C33	C34	C36	-0.7(2)
C8	C7	C10	C11	178.83(16)	C33	C32	C31	N6	179.47(14)
C8	C7	C10	C9	-1.11(16)	C33	C32	C31	C37	0.6(2)
C8	C7	C6	C5	-1.0(2)	C33	C34	C36	C37	1.0(3)
C7	C10	C11	C12	-179.60(16)	C34	C36	C37	C31	-0.4(3)
C7	C10	C9	N3	0.52(16)	C35	O4	C34	C33	-4.7(3)
C7	C10	C9	C15	-178.70(14)	C35	O4	C34	C36	175.56(18)
C10	C7	C6	C5	-178.89(17)	C36	C37	C31	N6	-178.98(16)

Table S8. Torsion Angles for cxy5030_0m.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C10	C11	C12	O2	179.98(14)	C36	C37	C31	C32	-0.4(2)
C10	C11	C12	C14	-1.7(2)	C31	N6	C30	C26	179.24(15)
C10	C9	C15	C14	-1.6(2)	C31	N6	C30	C29	-0.87(17)
C11	C10	C9	N3	-179.43(14)	C31	C32	C33	C34	-0.1(2)
C11	C10	C9	C15	1.3(2)	C27	N5	C26	C25	179.0(7)
C11	C12	C14	C15	1.4(2)	C27	N5	C26	C30	-0.6(2)
C13	O2	C12	C11	175.82(15)	C27	N5	C26	C25A	177.1(11)
C13	O2	C12	C14	-2.5(2)	C28	C29	C32	C33	2.2(3)
C9	N3	C8	C4	-179.19(15)	C28	C29	C32	C31	-177.62(17)
C9	N3	C8	C7	-1.01(17)	C43	C42	C41	C40	0.7(7)
C9	C10	C11	C12	0.3(2)	C44	C39	C38	C25	-57(2)
C9	C15	C14	C12	0.3(2)	C24	N4	C25	C38	-165.6(14)
C2	N1	C3	C16	-146.98(16)	C24	N4	C25	C26	70(3)
C2	N1	C3	C4	91.76(19)	C24	N4	C25A	C26	64(5)
C21	C20	C19	C18	-1.7(3)	C24	N4	C25A	C38A	-177(3)
C22	C17	C16	C3	-103.45(18)	C38A	C39A	C40A	C41A	177.2(11)
C5	N2	C4	C3	174.99(14)	C38A	C39A	C44A	C43A	-176.4(11)
C5	N2	C4	C8	0.1(2)	C39A	C38A	C25A	N4	-180(3)
C6	C7	C10	C11	-3.1(3)	C39A	C38A	C25A	C26	-57(5)
C6	C7	C10	C9	176.96(17)	C39A	C40A	C41A	C42A	-0.7(10)
O4	C34	C36	C37	-179.23(16)	C40A	C39A	C44A	C43A	3.3(7)
N5	C26	C30	N6	-178.50(15)	C40A	C41A	C42A	C43A	3.2(11)
N5	C26	C30	C29	1.6(2)	C41A	C42A	C43A	C44A	-2.5(10)
N5	C26	C25A	N4	55(3)	C42A	C43A	C44A	C39A	-0.9(9)
N5	C26	C25A	C38A	-66(3)	C44A	C39A	C40A	C41A	-2.5(8)
N5	C27	C28	C29	0.8(3)	C25A	N4	C24	O3	2(4)
N4	C25	C26	N5	48.7(19)	C25A	N4	C24	C45	180(4)
N4	C25	C26	C30	-131.7(15)	C25A	C26	C30	N6	4.0(12)
N6	C30	C29	C32	0.41(17)	C25A	C26	C30	C29	-175.9(12)
N6	C30	C29	C28	178.66(14)	C25A	C38A	C39A	C40A	107(4)
C42	C41	C40	C39	0.6(6)	C25A	C38A	C39A	C44A	-73(4)
C42	C43	C44	C39	0.7(5)					

6 Total synthesis of eudistomin X from 3o[8,9]



Deacetylation of 3o. To a solution of optically pure **3o** (51.8 mg, 0.15 mmol) in MeOH (1.5 mL) was added 6 N HCl aq. (1.5 mL) and stirred at 80 ºC for 4 h. Concentrate in vacuo to remove solvent. Suspend the residues in saturated Na_2CO_3 aq. (15 mL) and extract with CH_2Cl_2 (3 x 15 mL). The organic layers were combined and washed with brine, dried over anhydrous Na_2SO_4, and concentrated to dryness to afford pure **6**, which was used in the next step without further purification.

Synthesis of eudistomin X. To a solution of **6** in MeOH (2.0 mL) was added 37% aqueous formaldehyde (0.1 mL), AcOH (0.1 mL) and $NaBH_3CN$ (18.9 mg, 2.0 equiv.), stirred the solution at 25 ºC for 15 min. After completion of the reaction, quenched with saturated $NaHCO_3$ aq. (10 mL) and extract with CH_2Cl_2 (3 x 15 mL). The combined organic layer was washed with water (5 mL) and brine (5 mL), dried over Na_2SO_4, and concentrated in vacuo. The mixture was purified by flash column chromatography (0-3 % MeOH in CH_2Cl_2) to yield **eudistomin X (7)** (40.8 mg, 82% overall yield; > 99% ee) as an off-white amorphous solid. The structure and absolute configuration of compound **7** was confirmed by comparing with the reported spectroscopic data as shown in **Table S9** and optical rotation of **eudistomin X**.[9] **FTIR** (ν_{max}, cm^{-1}): 2992, 2916, 2848, 2359, 2340, 1699, 1544, 1457, 1375, 1108, 1055, 811, 697, 620, 518; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{22}N_3O^+$ $[M + H]^+$, 332.1757; found, 332.1753; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 14.72 min; $[\alpha]_D^{20}$ = -96.6 (c = 0.5, MeOH); Ref. 9: $[\alpha]_D^{20}$ = -106.1 (c = 0.49, MeOH).

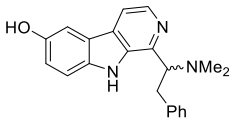
Table S9. ^1H and ^{13}C NMR data for synthetic structure of **eudistomin X** and **eudistomin X** in MeOD

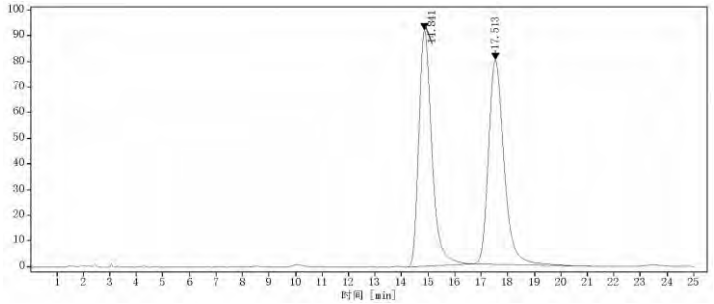
Position	δ_H (400 MHz, MeOD)	δ_H (400 MHz, MeOD)	δ_C (100 MH, MeOD)	δ_C (100 MHz, MeOD)
	Synthetic (**7**)	Nature[a]	Synthetic (**7**)	Nature[a]
1			142.9	144.7
3	8.16 (d, 5.3)	8.07 (d, 5.4)	137.2	136.9
4	7.82 (d, 5.3)	7.77 (d, 5.4)	115.2	114.9
4a			130.2	130.1
4b			122.8	122.9
5	7.46 (d, 2.4)	7.45 (d, 2.4)	106.6	106.6
6			152.3	152.2
7	7.08 (dd, 8.8, 2.4)	7.07 (dd, 8.8, 2.4)	119.7	119.5
8	7.37 (d, 8.7)	7.38 (d, 8.8)	113.4	113.5
8a			136.8	136.8
9a			137.4	137.3
10	4.38 (dd, 10.4, 4.6)	4.16 (dd, 10.4, 4.4)	70.7	71.4
11	3.50 (dd, 12.8, 10.4)	3.47 (dd, 13.1, 10.4)	37.9	38.0
	3.41 (dd, 12.8, 4.6)	3.33 (dd, 13.1, 4.4)		
12			139.0	139.9
13,17	6.84[b] (m)	6.82[b] (m)	130.3	130.2
14,16	6.94[b] (m)	6.92[b] (m)	129.0	129.0

15	6.94 (m)	6.92 (m)	127.2	126.9
$N(CH_3)_2$	2.51 (6H, s)	2.37 (6H, s)	43.2	43.5

[a]Data from Ref. 9. [b]2H.

HPLC analysis:





Entry	Retention Time	Height	Area	Area%
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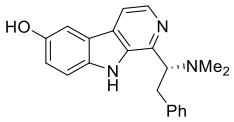
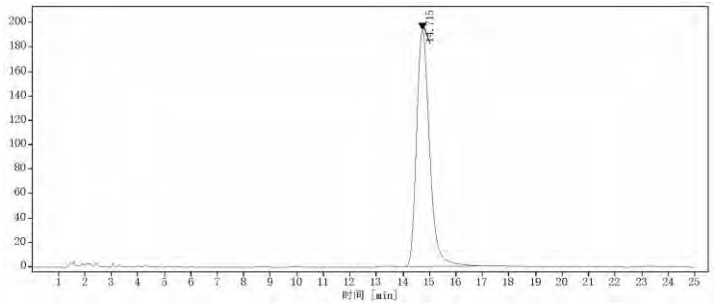
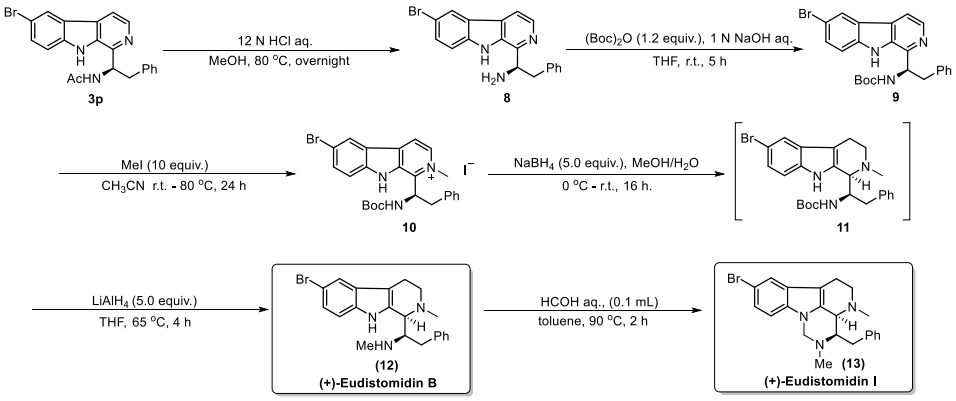
2	17.513	79.499	3242.038	50.778





Entry	Retention Time	Height	Area	Area%
1	14.715	193.705	6596.167	100

7 Total synthesis of (+)-eudistomidin B[10,11] and (+)-eudistomidin I[12,13] from 3p



Deacetylation of 3p. To a solution of **3p** (122.5 mg, 0.3 mmol, 97% ee) in MeOH (3.0 mL) was added concentrated hydrochloric acid (2.0 mL) and stirred at 80 °C overnight. Concentrate in vacuo to remove the solvent. Suspend the residues in saturated Na_2CO_3 aq. (15 mL) and extract with CH_2Cl_2 (3 x 15 mL). The organic layers were combined and washed with brine, dried over anhydrous Na_2SO_4, and concentrated to dryness to afford pure **8** (106.6 mg, 97% yield), which was used in the next step without further purification. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.43 (s, 1H), 8.37 (d, *J* = 5.3 Hz, 1H), 8.22 (d, *J* = 1.8 Hz, 1H), 7.78 (d, *J* = 5.3 Hz, 1H), 7.61 (dd, *J* = 8.6, 2.0 Hz, 1H), 7.38 (d, *J* = 8.6 Hz, 1H), 7.35 – 7.17 (m, 5H), 4.84 (dd, *J* = 10.1, 4.1 Hz, 1H), 3.51 – 3.27 (m, 1H), 2.98 (dd, *J* = 13.3, 9.4 Hz, 1H), 2.46 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.0, 138.7, 138.2, 134.5, 131.1, 129.5, 128.8, 128.5, 126.9, 124.4, 123.1, 113.8, 113.3, 112.4, 59.1, 43.3; **HRMS** (ESI-TOF) m/z: calcd for $C_{19}H_{17}BrN_3^+$ $[M + H]^+$, 366.0600; found, 366.0602.

Synthesis of *N*-Boc substituted 8. To a solution of **8** (106.6 mg, 0.29 mmol) in 3.0 mL THF and 0.6 mL 1 N NaOH aq. was added $(Boc)_2O$ (78.6 mg, 0.36 mmol) slowly and stirred at room temperature for 5 h. After completion of the reaction, quenched with saturated $NaHCO_3$ aq. (10 mL) and extract with CH_2Cl_2 (3 x 15 mL). The combined organic layer was washed with brine (5 mL), dried over Na_2SO_4, and concentrated in vacuo. The mixture was purified by flash column chromatography (15 % acetone in hexane) to yield **9** (116.0 mg, 86% yield) as a white amorphous solid. **Mp**, 247−249 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.77 (s, 1H), 8.49 (s, 1H), 8.39 – 8.20 (m, 1H), 8.06 (d, *J* = 5.3 Hz, 1H), 7.66 (d, *J* = 8.7 Hz, 1H), 7.58 (d, *J* = 8.8 Hz, 1H), 7.35 – 7.07 (m, 6H), 5.59 – 5.21 (m, 1H), 3.25 – 2.96 (m, 2H), 1.28 (s, 9H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 155.2, 145.7, 139.1, 138.2, 137.6, 133.2, 130.5, 129.4, 127.8, 127.0, 126.1, 124.3, 122.7, 114.0, 111.2, 77.9, 53.2, 28.1; **FTIR** (ν_{max}, cm^{-1}): 3180, 2984, 2903, 2389, 2342, 1673, 1542, 1475, 1271, 1161, 1053, 742, 694, 605, 556; **HRMS** (ESI-TOF) m/z: calcd for$C_{24}H_{25}BrN_3O_2^+$ $[M + H]^+$, 466.1125; found, 466.1128; **$[\alpha]_D^{20}$** = 24.8 (c = 0.3, $CHCl_3$).

Synthesis of β-carboline methiodide (10). Under conditions similar to those reported by Still.[14] To a solution of **9** (116.6 mg, 0.25 mmol) in 2.5 mL CH_3CN was added MeI (155.6 uL, 2.5 mmol) and stirred at 80 °C for 24 h. After completion of the reaction, concentrate in vacuo to remove the solvent and unconsumed MeI led to the desired β-carboline methiodide **10** with quantitative yield. **Mp**, 268−270 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 12.53 (s, 1H), 8.81 (s, 1H), 8.79 – 8.56 (m, 2H), 7.96 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.85 (d, *J* = 8.9 Hz, 2H), 7.22 (q, *J* = 7.1 Hz, 5H), 5.69 (s, 1H), 4.38 (s, 3H), 3.54 – 3.40 (m, 2H), 1.23 (s, 8H), 0.92 (s, 1H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 155.3, 142.1, 135.6, 134.6, 129.1, 128.5, 127.2, 126.0, 120.8, 117.3, 115.0, 113.8, 79.8, 51.9, 45.9, 36.0, 27.9; **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{27}BrN_3O_2^+$ $[M-I]^+$, 480.1281; found, 480.1284; **$[\alpha]_D^{20}$** = 27.3 (c = 0.3, $CHCl_3$).

Reduction of intermediate 10. Under conditions similar to those reported by Still.[14] To a solution of **10** (152.1 mg, 0.25 mmol) in a 3:1 mixture of methanol and water (4.0 mL) was added $NaBH_4$ (47.3 mg, 1.25 mmol) in portions, and stirred the solutions at room temperature for 16 h. After completion of the reaction, quenched with saturated $NaHCO_3$ aq. (10 mL) and extract with CH_2Cl_2 (3 x 15 mL). The combined organic layer was washed with brine (5 mL), dried over Na_2SO_4, and concentrated in vacuo to afford crude **11** (>10:1 d.r., according to NMR spectrum), which was used in the next step without further purification. **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{30}BrN_3O_2^+$ $[M + H]^+$, 483.1516; found, 483.1518.

Synthesis of (+)-**eudistomidin B.** To a solution of **11** (72.7 mg, 0.15 mmol) in 1.5 mL anhydrous THF was added 0.75 mL of $LiAlH_4$ (1.0 M in THF) dropwise and stirred at 65 °C for 4 h. The reaction was quenched by dropwise addition of water until effervescence ceased. The mixture was then diluted with ethyl acetate before addition of saturated aqueous solution of Rochelle's salt and the subsequent biphasic mixture was stirred for 2 h. The organic layer was separated, aqueous phase was extracted with ethyl acetate, The organic layers were combined and washed with brine, dried over anhydrous Na_2SO_4, and concentrated to dryness. The remaining residue was purified by flash column chromatography (0-3 % MeOH in CH_2Cl_2) to yield (+)-**eudistomidin B** (54.4 mg, 66% yield from **10**) as a pale yellow amorphous solid. The structure and absolute configuration of compound **12** was confirmed by comparing with the reported spectroscopic data as shown in **Table S10** and optical rotation of **eudistomidin B**.[11] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.65 (d, *J* = 1.7 Hz, 1H), 7.34 – 7.20 (m, 6H), 7.19 – 7.11 (m, 2H), 3.76 – 3.69 (m, 1H), 3.28 (dt, *J* = 11.2, 3.0 Hz, 1H), 3.22 – 3.11 (m, 2H), 2.94 – 2.86 (m, 1H), 2.72 (ddt, *J* = 14.9, 8.3, 3.7 Hz, 2H), 2.65 (s, 3H), 2.49 (s, 3H), 1.91 (t, *J* = 12.8 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.6, 134.4, 129.2, 128.8, 128.6, 126.5, 123.7, 120.6, 112.6, 112.2, 109.2, 77.5, 77.2, 76.8, 63.8, 60.6, 54.4, 44.2, 35.9, 35.3, 21.4; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{25}BrN_3^+$ $[M + H]^+$, 398.1226; found, 398.1228; **$[\alpha]_D^{22}$** = 50.0 (c = 0.2, MeOH); Ref. 10: **$[\alpha]_D^{22}$** = -54.0 (c = 0.2, MeOH).

Table S10. ^{1}H and ^{13}C NMR data for synthetic structure (**12**) of (+)-**eudistomidin B, eudistomidin B** and ^{1}H NMR data for (1*S*,10*S*)-diastereomer of **eudistomidin B** in $CDCl_3$: CF_3COOD = 10:1

Position	δ_H (400 MHz)			δ_C (100 MHz)	
	Synthetic (**12**) (1*S*,10*R*)	Nature[a] (1*R*,10*S*)	(1*S*,10*S*)-diastereomer[a]	Synthetic (**12**) (1S,10R)	Nature[a] (1R,10S)
1	5.15 (d, 3.2)	5.07 (d, 2.9)	**5.27 (d, 2.3)**	63.44	63.63
3	3.37 (dd, 13.4, 6.3)	3.30 (m)	**3.52 (m)**	46.77	45.85
	2.92[d] (m)	2.94[d] (m)	2.98 (m)		
4	2.84[d] (m)	2.86[d] (m)	2.88 (m)	15.09	14.92
	2.45 (dd, 17.3, 5.4).	2.43 (br d, 10.5)	**2.30 (dd, 16.9, 4.9)**		
4a				106.25	105.59
4b				126.60	126.61
5	7.48 (d,1.8 Hz)	7.54 (s)	7.49 (d, 2.0)	120.73	121.06
6				113.92	113.71
7	7.37 (dd, 8.6, 1.8)	7.41 (d, 8.5)	7.42 (dd, 9.0, 2.0)	127.94	127.06
8	7.31 (d, 8.8)	7.43 (d, 8.5)	7.48 (d,PDF9.0)	114.45	113.82
8a				135.65	135.53
9	10.79 (s)	11.19 (s)	11.27 (s)		
9a				121.43	122.09
10	4.18 (m)	4.01 (m)	4.10 (m)	64.25	63.86
11	3.15 (dd, 14.9, 5.3)	3.22 (m)	**3.27 (m)**	33.75	33.37

	3.04 (td, 12.7, 5.5),	3.07 (dd, 15.0, 9.4)	**3.26 (m)**		
12				132.86	133.82
13,17	6.57[b] (d, 7.2)	6.61[b] (d, 7.5)	6.50[b] (d, 7.5)	128.51	128.46
14,16	7.01[b] (t, 7.5)	7.07[b] (dd, 7.5, 7.5)	7.02[b] (d, 7.5)	129.37	128.93
15	7.10 (t, 7.3)	7.15 (dd, 7.5, 7.5)	7.12 (d, 7.5)	128.60	127.93
18	2.89[c] (s)	2.85[c] (s)	2.92[c] (s)	33.10	32.54
19	2.93[c] (s)	2.90[c] (s)	**2.94[c] (br s)**	40.47	39.81

[a]Data from Ref. 11. [b]2H. [c]3H. [d]Interchangeable.

Synthesis of (+)**-eudistomidin I.** Under conditions similar to those reported by Kobayashi.[13] To a solution of (+)-**eudistomidin B** (39.8 mg, 0.1 mmol) in toluene (2.0 mL) was added HCOH aq. (0.1 mL) and stirred at 90 °C for 1 h. The reaction mixture was concentrated in vacuo and purified by a silica gel flash column chromatography (0-20 % EtOAc in hexane) to yield **eudistomidin I** (33.6 mg, 82% yield) as a pale yellow amorphous. **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{25}BrN_3^+$ $[M + H]^+$, 410.1226; found, 410.1229; $[\alpha]_D^{24}$ = 5.1 (c = 0.5, acetone); Ref. 13: $[\alpha]_D^{24}$= -5.2 (c = 0.5, acetone). The structure and absolute configuration of compound **13** was confirmed by comparing with the reported spectroscopic data as shown in **Table S11** and optical rotation of **eudistomidin I**.[12,13] The relative stereochemistry of H-1 and H-10 in (+)-**eudistomidin I** (**13**) was elucidated from NOESY correlations as shown in **Figure S1**. NOESY cross-peaks of H_2-11/H-20a and H-13/H-20a suggested β orientations of C-11 and H-20a, while α orientations of H-1, H-10, and H-20b were deduced from NOESY correlations for H-1/H_3-18 and H_3-18/H-20b.

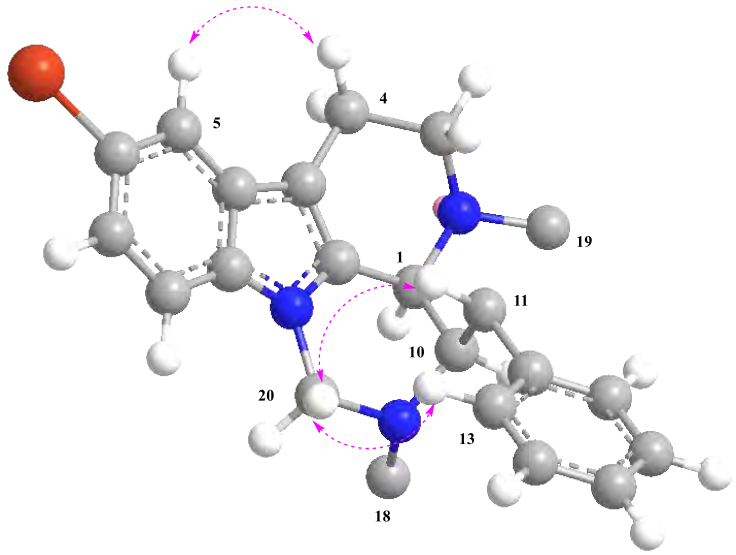


Figure S1. Selected NOESY correlations and relative stereochemistry for (+)-**Eudistomidin I** (**13**) (hydrogen atoms of methyl groups were omitted).

Table S11. ^{1}H and ^{13}C NMR data for synthetic structure (**13**) of (+)-**eudistomidin I** and **eudistomidin I** in acetone-d_6

Position	δ_H (400 MHz)	δ_H (600 MHz)	δ_C (101 MHz)	δ_C (125 MHz)
	Synthetic (**13**)	Nature[a]	Synthetic (**13**)	Nature[a]
1	3.63 (br s)	3.61 (br s)	59.0	59.2
3	2.64 (m)	2.60 (m)	55.1	55.2
	3.15 (dd, 11.5, 5.9)	3.13 (dd, 11.2, 5.1)		
4	2.69[d]	2.69[d]		

	2.91[d]	2.90[d]		
4a			107.7	107.8
4b			128.9	129.0
5	7.59 (d, 1.9)	7.59 (d, 1.8)	113.3	113.4
6			112.7	112.8
7	7.21 (dd, 8.6, 1.9)	7.20 (dd, 8.4, 1.8)	123.8	123.9
8	7.30 (dd, 8.6, 0.6)	7.29 (d, 8.4)	121.4	121.6
8a			136.7	136.8
9a			136.4	136.4
10	3.71 (m)	3.69 (m)	61.8	61.9
11	2.19 (dd, 15.2, 10.5)	2.17 (dd, 15.6, 10.8)	30.9	30.9
	2.86[e]	2.87[e]		
12			141.5	141.6
13, 17	7.26[b] (m)	7.25[b] (d, 7.8)	131.2	131.3
14, 16	7.22[b] (m)	7.23[b] (m)	129.9	130.0
15	7.15 (m)	7.13 (m)	126.5	126.6
18	2.74[c] (s)	2.72[c] (s)	42.6	42.7
19	2.40[c] (s)	2.36[c] (s)	42.6	42.7
20	4.82 (d, 11.1)	4.80 (d, 10.8)	61.3	61.4
	4.96 (d, 11.1)	4.94 (d, 10.8)		

[a]Data from Ref. 12. [b]2H. [c]3H. [d]J-values were not determined due to overlapping with other signals.

8 References

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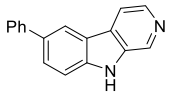
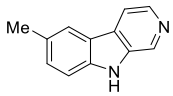
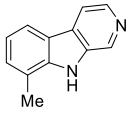
9 Characterization of Starting materials



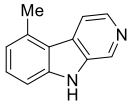
6-phenyl-9*H*-pyrido[3,4-*b*]indole (**1b**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.69 (s, 1H), 8.93 (s, 1H), 8.63 – 8.51 (m, 1H), 8.37 (d, *J* = 5.2 Hz, 1H), 8.20 (d, *J* = 5.3 Hz, 1H), 7.87 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.78 (d, *J* = 7.7 Hz, 2H), 7.68 (d, *J* = 8.5 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H).



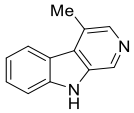
6-methyl-9*H*-pyrido[3,4-*b*]indole (**1c**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.47 (s, 1H), 8.85 (s, 1H), 8.30 (d, *J* = 5.3 Hz, 1H), 8.05 (d, *J* = 5.2 Hz, 1H), 8.02 (s, 1H), 7.48 (d, *J* = 8.3 Hz, 1H), 7.37 (dd, *J* = 8.4, 1.7 Hz, 1H), 2.48 (s, 3H).



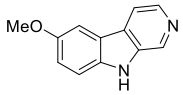
8-methyl-9*H*-pyrido[3,4-*b*]indole (**1*d***): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.56 (s, 1H), 8.91 (s, 1H), 8.33 (d, *J* = 5.2 Hz, 1H), 8.06 (dd, *J* = 9.9, 6.6 Hz, 2H), 7.35 (d, *J* = 7.1 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 2.58 (s, 3H).



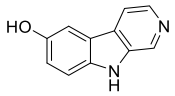
5-methyl-9H-pyrido[3,4-*b*]indole (**1e**): ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.63 (s, 1H), 8.90 (s, 1H), 8.34 (d, *J* = 5.3 Hz, 1H), 8.06 (d, *J* = 5.3 Hz, 1H), 7.43 (d, *J* = 4.3 Hz, 2H), 7.03 (t, *J* = 4.2 Hz, 1H), 2.82 (s, 3H).



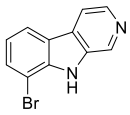
4-methyl-9*H*-pyrido[3,4-*b*]indole (**1f**): Synthesized according to Ref. 7, **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.63 (s, 1H), 8.75 (s, 1H), 8.20 (d, *J* = 7.9 Hz, 1H), 8.14 (s, 1H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.54 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 7.26 (ddd, *J* = 8.0, 7.0, 1.2 Hz, 1H), 2.78 (s, 3H).



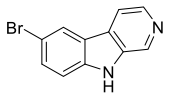
6-methoxy-9*H*-pyrido[3,4-*b*]indole (**1g**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.41 (s, 1H), 8.86 (s, 1H), 8.44 – 8.18 (m, 1H), 8.07 (d, *J* = 5.1 Hz, 1H), 7.78 (s, 1H), 7.50 (d, *J* = 8.8 Hz, 1H), 7.19 (d, *J* = 9.0 Hz, 1H), 3.86 (s, 3H).



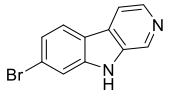
9*H*-pyrido[3,4-*b*]indol-6-ol (**1h**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 12.43 (s, 1H), 9.63 (s, 1H), 9.24 (s, 1H), 8.71 (d, *J* = 6.2 Hz, 1H), 8.50 (d, *J* = 6.2 Hz, 1H), 7.75 (d, *J* = 2.4 Hz, 1H), 7.67 (d, *J* = 8.9 Hz, 1H), 7.35 (dd, *J* = 8.9, 2.4 Hz, 1H).



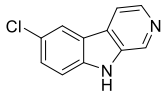
8-bromo-9*H*-pyrido[3,4-*b*]indole (**1i**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.81 (s, 1H), 8.96 (d, *J* = 1.1 Hz, 1H), 8.39 (d, *J* = 5.3 Hz, 1H), 8.28 (d, *J* = 7.8 Hz, 1H), 8.14 (dd, *J* = 5.3, 1.1 Hz, 1H), 7.78 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.20 (t, *J* = 7.8 Hz, 1H).



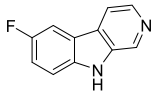
6-bromo-9*H*-pyrido[3,4-*b*]indole (**1j**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.79 (s, 1H), 8.93 (d, *J* = 1.1 Hz, 1H), 8.52 (d, *J* = 2.0 Hz, 1H), 8.36 (d, *J* = 5.3 Hz, 1H), 8.16 (d, *J* = 5.2 Hz, 1H), 7.66 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.57 (d, *J* = 8.7 Hz, 1H).



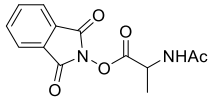
7-bromo-9*H*-pyrido[3,4-*b*]indole (**1k**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.73 (s, 1H), 8.93 (d, *J* = 1.1 Hz, 1H), 8.37 (d, *J* = 5.2 Hz, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 8.13 (d, *J* = 5.3 Hz, 1H), 7.80 (d, *J* = 1.7 Hz, 1H), 7.39 (dd, *J* = 8.4, 1.8 Hz, 1H).



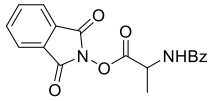
6-chloro-9*H*-pyrido[3,4-*b*]indole (**1l**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.78 (s, 1H), 9.01 – 8.85 (m, 1H), 8.47 – 8.28 (m, 2H), 8.15 (d, *J* = 5.3 Hz, 1H), 7.62 (d, *J* = 8.7 Hz, 1H), 7.55 (dd, *J* = 8.7, 2.1 Hz, 1H).



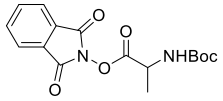
6-fluoro-9*H*-pyrido[3,4-*b*]indole (**1m**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.66 (s, 1H), 8.92 (d, *J* = 1.1 Hz, 1H), 8.33 (d, *J* = 5.2 Hz, 1H), 8.15 – 8.05 (m, 2H), 7.61 (dd, *J* = 8.9, 4.4 Hz, 1H), 7.41 (td, *J* = 9.2, 2.7 Hz, 1H).



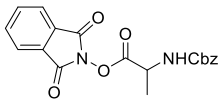
1,3-dioxoisoindolin-2-yl acetylalaninate (**2a**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.62 (d, *J* = 6.9 Hz, 1H), 7.96 (qd, *J* = 5.5, 3.3 Hz, 4H), 4.70 (p, *J* = 7.2 Hz, 1H), 1.88 (s, 3H), 1.49 (d, *J* = 7.3 Hz, 3H).



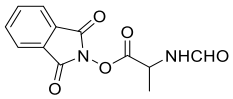
1,3-dioxoisoindolin-2-yl benzoylalaninate (**2ab**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.12 (d, *J* = 6.7 Hz, 1H), 7.99 – 7.88 (m, 6H), 7.56 (dd, *J* = 6.8, 1.9 Hz, 1H), 7.53 – 7.46 (m, 2H), 4.96 (p, *J* = 7.3 Hz, 1H), 1.64 (d, *J* = 7.4 Hz, 3H).



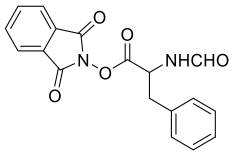
1,3-dioxoisoindolin-2-yl (tert-butoxycarbonyl)alaninate (**2ac**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.96 (qd, *J* = 7.6, 4.1 Hz, 4H), 7.70 (d, *J* = 7.3 Hz, 1H), 4.49 (p, *J* = 7.3 Hz, 1H), 1.46 (d, *J* = 7.4 Hz, 3H), 1.40 (s, 9H).



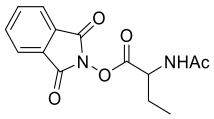
1,3-dioxoisoindolin-2-yl ((benzyloxy)carbonyl)alaninate (**2ad**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.16 (d, *J* = 7.2 Hz, 1H), 8.03 – 7.89 (m, 4H), 7.39 – 7.27 (m, 5H), 5.08 (d, *J* = 2.6 Hz, 2H), 4.61 (p, *J* = 7.3 Hz, 1H), 1.50 (d, *J* = 7.3 Hz, 3H).



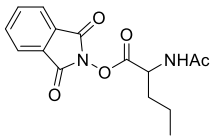
1,3-dioxoisoindolin-2-yl formylalaninate (**2ae**): **Mp**, 168−170 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.89 (d, *J* = 7.2 Hz, 1H), 8.12 (s, 1H), 8.04 – 7.86 (m, 4H), 4.84 (p, *J* = 7.3 Hz, 1H), 1.51 (d, *J* = 7.3 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.6, 161.6, 161.1, 135.6, 128.2, 124.0, 44.4, 17.1; **FTIR** (ν_{max}, cm^{-1}): 3295, 2335, 1747, 1655, 1537, 1380, 1062, 876, 518; **HRMS** (ESI-TOF) m/z: calcd for $C_{12}H_{11}N_2O_5^+$ $[M + H]^+$, 263.0662; found, 263.0664.



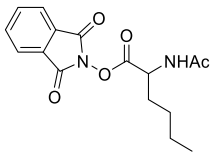
1,3-dioxoisoindolin-2-yl formylphenylalaninate (**2b**): **Mp**, 151−153 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.87 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.08 (d, *J* = 1.4 Hz, 1H), 8.04 – 7.91 (m, 4H), 7.42 – 7.29 (m, 4H), 7.29 – 7.20 (m, 1H), 5.11 (ddd, *J* = 9.5, 8.0, 4.8 Hz, 1H), 3.38 – 3.26 (m, 1H), 3.10 (dd, *J* = 14.0, 9.8 Hz, 1H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.4, 161.5, 161.3, 135.9, 135.6, 129.3, 128.4, 128.2, 126.9, 124.1, 49.9, 36.4; **FTIR** (ν_{max}, cm^{-1}): 3325, 2382, 1737, 1655, 1520, 1387, 1058, 970, 879, 700, 499; **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{15}N_2O_5^+$ $[M + H]^+$, 339.0975; found, 339.0978.



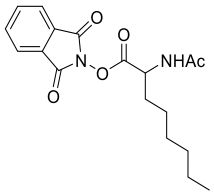
1,3-dioxoisoindolin-2-yl 2-acetamidobutanoate (**4a**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.56 (d, *J* = 7.2 Hz, 1H), 8.04 – 7.86 (m, 4H), 4.62 (td, *J* = 7.6, 5.7 Hz, 1H), 1.91 (s, 3H), 1.90 – 1.77 (m, 2H), 1.04 (t, *J* = 7.4 Hz, 3H).



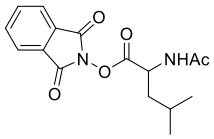
1,3-dioxoisoindolin-2-yl 2-acetamidopentanoate (**4b**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.57 (d, *J* = 7.3 Hz, 1H), 7.96 (d, *J* = 4.0 Hz, 4H), 4.67 (td, *J* = 8.0, 5.6 Hz, 1H), 1.90 (s, 3H), 1.82 (ddd, *J* = 14.7, 10.7, 7.1 Hz, 2H), 1.48 (q, *J* = 7.6 Hz, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).



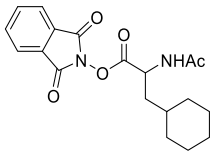
1,3-dioxoisoindolin-2-yl 2-acetamidohexanoate (**4c**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.56 (d, J = 7.3 Hz, 1H), 8.05 – 7.86 (m, 4H), 4.65 (td, J = 8.0, 5.6 Hz, 1H), 1.90 (s, 3H), 1.89 – 1.74 (m, 2H), 1.53 – 1.39 (m, 2H), 1.34 (tdd, J = 10.9, 8.3, 5.5 Hz, 2H), 0.91 (t, J = 7.2 Hz, 3H).



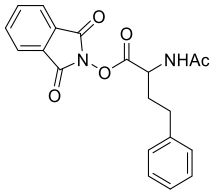
1,3-dioxoisoindolin-2-yl 2-acetamidooctanoate (**4d**): **Mp**, 137−139 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.57 (d, J = 7.3 Hz, 1H), 7.96 (q, J = 4.9 Hz, 4H), 4.65 (q, J = 7.0 Hz, 1H), 2.01 – 1.66 (m, 5H), 1.46 (p, J = 7.1 Hz, 2H), 1.30 (d, J = 8.8 Hz, 6H), 0.88 (t, J = 6.5 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 169.6, 161.6, 135.5, 128.2, 124.0, 50.1, 40.1, 31.0, 28.2, 24.8, 22.1, 22.0, 13.9; **FTIR** (ν_{max}, cm^{-1}): 3291, 2930, 2382, 1750, 1649, 1541, 1377, 1094, 878, 590; **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{23}N_2O_5^+$ $[M + H]^+$, 347.1601; found, 347.1604.



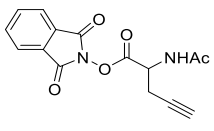
1,3-dioxoisoindolin-2-yl acetylleucinate (**4e**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.59 (d, J = 7.5 Hz, 1H), 8.01 – 7.89 (m, 4H), 4.69 (ddd, J = 9.2, 7.5, 5.6 Hz, 1H), 1.90 (s, 3H), 1.85 – 1.66 (m, 3H), 0.96 (d, J = 6.3 Hz, 3H), 0.91 (d, J = 6.4 Hz, 3H).



1,3-dioxoisoindolin-2-yl 2-acetamido-3-cyclohexylpropanoate (**4f**): **Mp**, 140−142 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.58 (d, J = 7.4 Hz, 1H), 7.96 (q, J = 4.6 Hz, 4H), 4.71 (q, J = 7.5 Hz, 1H), 1.90 (s, 3H), 1.83 – 1.55 (m, 7H), 1.56 – 1.39 (m, 1H), 1.18 (p, J = 12.9 Hz, 3H), 1.08 – 0.80 (m, 2H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 170.0, 169.7, 161.6, 135.5, 128.2, 124.0, 47.8, 38.3, 33.3, 32.9, 31.5, 25.9, 25.7, 25.5, 22.1; **FTIR** (ν_{max}, cm^{-1}): 3325, 2925, 2392, 1750, 1653, 1544, 1375, 1084, 874, 692, 598; **HRMS** (ESI-TOF) m/z: calcd for $C_{19}H_{23}N_2O_5^+$ $[M + H]^+$, 359.1601; found, 359.1603.

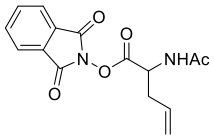


1,3-dioxoisoindolin-2-yl 2-acetamido-4-phenylbutanoate (**4g**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.69 (d, J = 7.2 Hz, 1H), 8.03 – 7.89 (m, 4H), 7.33 (t, J = 7.5 Hz, 2H), 7.29 – 7.16 (m, 3H), 4.71 – 4.54 (m, 1H), 2.79 (t, J = 7.9 Hz, 2H), 2.22 – 2.02 (m, 2H), 1.94 (s, 3H).

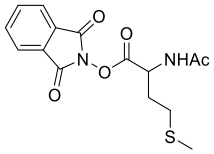


1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-ynoate (**4h**): **Mp**, 168−170 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.79 (d, J = 7.8 Hz, 1H), 7.96 (hept, J = 3.8 Hz, 4H), 4.90 (q, J = 7.1 Hz, 1H), 3.03 (d, J =

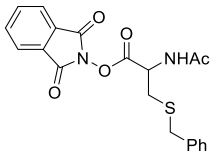
2.6 Hz, 1H), 2.86 – 2.65 (m, 2H), 1.93 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 167.8, 161.4, 135.6, 128.2, 124.0, 78.9, 74.1, 49.3, 22.1, 21.1; **FTIR** (ν_{max}, cm^{-1}): 3348, 3279, 2971, 2959, 1750, 1650, 1532, 1355, 1108, 1031, 983, 871, 582; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{13}N_2O_5^+$ $[M + H]^+$, 301.0819; found, 301.0821.



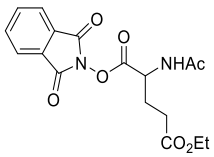
1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-enoate (**4i**): **Mp**, 138−140 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.60 (d, *J* = 7.6 Hz, 1H), 7.97 (tdd, *J* = 9.0, 5.3, 3.3 Hz, 4H), 5.86 (ddt, *J* = 17.1, 10.1, 6.9 Hz, 1H), 5.36 – 5.10 (m, 2H), 4.77 (td, *J* = 8.0, 5.5 Hz, 1H), 2.72 – 2.61 (m, 1H), 2.56 (q, *J* = 7.3 Hz, 1H), 1.90 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 168.9, 161.6, 135.6, 132.5, 128.2, 124.0, 119.2, 50.0, 35.2, 22.1. **FTIR** (ν_{max}, cm^{-1}): 3325, 2389, 1788, 1735, 1646, 1541, 1375, 1193, 1099, 874, 597, 516; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{15}N_2O_5^+$ $[M + H]^+$, 303.0975; found, 303.0978.



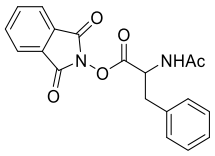
1,3-dioxoisoindolin-2-yl acetylmethioninate (**4j**): **^{1}H NMR** (400 MHz, DMSO) δ 8.62 (d, *J* = 7.2 Hz, 1H), 8.02 – 7.89 (m, 4H), 4.83 (ddd, *J* = 8.5, 7.2, 5.3 Hz, 1H), 2.69 – 2.61 (m, 2H), 2.16 – 2.06 (m, 2H), 2.09 (s, 3H), 1.91 (s, 3H).



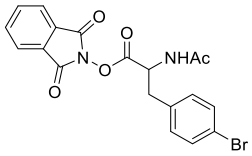
1,3-dioxoisoindolin-2-yl *N*-acetyl-S-benzylcysteinate (**4k**): **Mp**, 149−151 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.91 (d, *J* = 7.9 Hz, 1H), 7.13 (q, *J* = 4.9 Hz, 4H), 6.50 (d, *J* = 5.8 Hz, 4H), 6.46 – 6.38 (m, 1H), 4.09 (td, *J* = 8.5, 5.1 Hz, 1H), 2.14 (dd, *J* = 14.1, 5.1 Hz, 1H), 2.09 – 1.93 (m, 1H), 1.67 (s, 2H), 1.10 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 168.2, 161.5, 137.9, 135.6, 128.9, 128.5, 128.1, 127.0, 124.0, 50.0, 35.3, 32.0, 22.2; **FTIR** (ν_{max}, cm^{-1}):2988, 2905, 2387, 2344., 1753, 1700, 1651, 1559, 1544, 1459, 1078, 658; **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{19}N_2O_5S^+$ $[M + H]^+$, 399.1009; found, 399.1013.



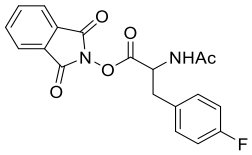
1-(1,3-dioxoisoindolin-2-yl) 5-ethyl acetylglutamate (**4l**): **Mp**, 136−138 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.61 (d, *J* = 7.3 Hz, 1H), 7.97 (q, *J* = 6.1 Hz, 4H), 4.75 (dt, *J* = 13.1, 6.4 Hz, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 2.55 (q, *J* = 6.4 Hz, 2H), 2.17 (dq, *J* = 13.9, 7.0 Hz, 1H), 2.03 (dp, *J* = 14.7, 7.0 Hz, 1H), 1.90 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 171.9, 169.8, 169.3, 161.6, 135.6, 128.2, 124.0, 60.1, 49.4, 29.4, 26.2, 22.1, 14.1; **FTIR** (ν_{max}, cm^{-1}): 3300, 2982, 2381, 2332, 1738, 1655, 1544, 1373, 1255, 1188, 1086, 953, 878, 694, 517; **HRMS** (ESI-TOF) m/z: calcd for $C_{17}H_{19}N_2O_7^+$ $[M + H]^+$, 363.1187; found, 363.1188.



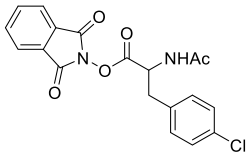
1,3-dioxoisoindolin-2-yl acetylphenylalaninate (**4m**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.68 (d, J = 8.0 Hz, 1H), 8.03 – 7.88 (m, 4H), 7.39 – 7.28 (m, 4H), 7.28 – 7.22 (m, 1H), 4.95 (ddd, J = 9.8, 8.0, 4.9 Hz, 1H), 3.27 (dd, J = 14.0, 4.9 Hz, 2H), 3.06 (dd, J = 14.0, 10.0 Hz, 1H), 1.82 (s, 3H).



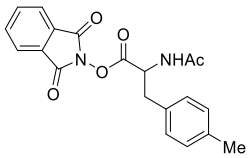
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-bromophenyl)propanoate (**4n**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.69 (d, J = 8.1 Hz, 1H), 7.97 (dtt, J = 8.9, 5.8, 3.4 Hz, 4H), 7.50 (d, J = 8.1 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 4.98 (ddd, J = 9.9, 8.1, 5.0 Hz, 1H), 3.25 (dd, J = 13.9, 5.0 Hz, 1H), 3.05 (dd, J = 13.9, 10.0 Hz, 1H), 1.83 (s, 3H).



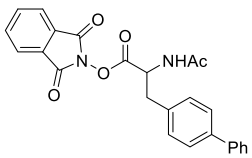
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-fluorophenyl)propanoate (**4o**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.67 (d, J = 8.1 Hz, 1H), 7.97 (dtt, J = 8.9, 5.8, 3.4 Hz, 4H), 7.45 – 7.33 (m, 2H), 7.20 – 7.06 (m, 2H), 4.96 (ddd, J = 9.9, 8.0, 5.0 Hz, 1H), 3.25 (dd, J = 13.9, 5.1 Hz, 1H), 3.05 (dd, J = 14.0, 10.0 Hz, 1H), 1.82 (s, 3H).



1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-chlorophenyl)propanoate (**4p**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.68 (d, J = 8.1 Hz, 1H), 8.05 – 7.87 (m, 4H), 7.45 – 7.29 (m, 4H), 4.98 (ddd, J = 9.9, 8.1, 5.0 Hz, 1H), 3.26 (dd, J = 13.9, 5.0 Hz, 1H), 3.06 (dd, J = 13.9, 10.0 Hz, 1H), 1.83 (s, 3H).

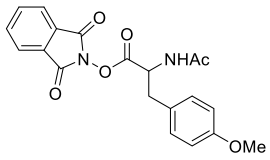


1,3-dioxoisoindolin-2-yl 2-acetamido-3-(p-tolyl)propanoate (**4q**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.65 (d, J = 8.0 Hz, 1H), 8.02 – 7.90 (m, 4H), 7.23 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 7.7 Hz, 2H), 4.90 (ddd, J = 9.8, 8.0, 4.9 Hz, 1H), 3.27 – 3.16 (m, 1H), 3.01 (dd, J = 14.0, 9.9 Hz, 1H), 2.27 (s, 3H), 1.83 (s, 3H).

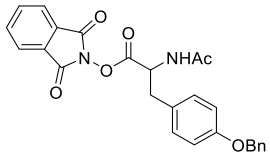


1,3-dioxoisoindolin-2-yl 3-([1,1'-biphenyl]-4-yl)-2-acetamidopropanoate (**4r**): **Mp**, 220−222 °C; **^{1}H NM**R (400 MHz, DMSO-d_6) δ 8.73 (d, J = 8.0 Hz, 1H), 8.03 – 7.93 (m, 4H), 7.70 – 7.65 (m, 2H),

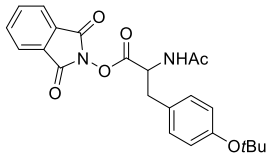
7.65 – 7.61 (m, 2H), 7.46 (ddd, *J* = 8.0, 4.6, 1.9 Hz, 4H), 7.39 – 7.32 (m, 1H), 5.00 (ddd, *J* = 9.9, 7.9, 4.8 Hz, 1H), 3.30 (d, *J* = 4.9 Hz, 1H), 3.11 (dd, *J* = 13.9, 9.9 Hz, 1H), 1.85 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 169.0, 161.6, 139.8, 138.7, 135.6, 135.5, 129.8, 128.9, 128.2, 127.4, 126.6, 126.5, 124.0, 51.5, 36.1, 22.2; **FTIR** (ν_{max}, cm^{-1}): 3338, 2985, 2359, 1753, 1655, 1525, 1378, 1100, 971, 880, 755, 690, 592; **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{21}N_2O_5^+$ $[M + H]^+$, 429.1445; found, 429.1448.



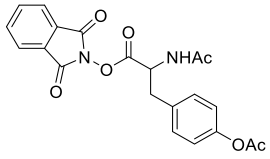
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-methoxyphenyl)propanoate (**4s**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.65 (d, *J* = 7.8 Hz, 1H), 7.96 (dt, *J* = 13.2, 4.2 Hz, 4H), 7.28 (d, *J* = 7.9 Hz, 2H), 6.87 (d, *J* = 8.1 Hz, 2H), 4.90 (td, *J* = 8.9, 4.5 Hz, 1H), 3.73 (s, 3H), 3.21 (dd, *J* = 14.1, 5.0 Hz, 1H), 3.10 – 2.90 (m, 1H), 1.84 (s, 3H).



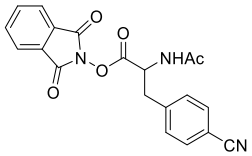
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(benzyloxy)phenyl)propanoate (**4t**): **Mp**, 218−220 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.64 (d, *J* = 8.0 Hz, 1H), 8.02 – 7.92 (m, 4H), 7.45 (d, *J* = 7.0 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 2H), 7.33 (d, *J* = 7.1 Hz, 1H), 7.30 – 7.24 (m, 2H), 6.95 (d, *J* = 8.6 Hz, 2H), 5.08 (s, 2H), 4.89 (ddd, *J* = 9.7, 7.9, 4.8 Hz, 1H), 3.20 (dd, *J* = 14.0, 4.9 Hz, 1H), 2.99 (dd, *J* = 14.0, 9.9 Hz, 1H), 1.83 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.6, 169.0, 161.6, 157.3, 137.1, 135.6, 134.5, 130.4, 130.3, 128.4, 128.3, 128.2, 127.8, 127.7, 127.7, 124.0, 122.9, 114.6, 114.4, 69.1, 51.8, 35.7, 22.1; **FTIR** (ν_{max}, cm^{-1}): 3334, 2990, 2901, 2359, 2340, 1755, 1659, 1521, 1375, 1251, 1185, 1095, 732, 698, 588; **HRMS** (ESI-TOF) m/z: calcd for $C_{26}H_{23}N_2O_6^+$ $[M + H]^+$, 459.1551; found, 459.1554.



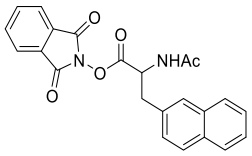
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(tert-butoxy)phenyl)propanoate (**4u**): **Mp**, 162−164 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.66 (d, *J* = 8.0 Hz, 1H), 8.05 – 7.87 (m, 4H), 7.25 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 4.92 (ddd, *J* = 9.8, 8.0, 5.0 Hz, 1H), 3.22 (dd, *J* = 14.0, 5.0 Hz, 1H), 3.07 – 2.96 (m, 1H), 1.82 (s, 3H), 1.27 (s, 9H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.6, 169.0, 164.1, 161.6, 153.9, 135.6, 134.5, 130.8, 129.9, 129.8, 129.6, 128.8, 128.2, 124.0, 123.5, 123.4, 123.0, 77.8, 51.6, 35.9, 28.6, 22.1; **FTIR** (ν_{max}, cm^{-1}): 3334, 2919, 2383, 1748, 1657, 1542, 1385, 1161, 1084, 876, 696, 515; **HRMS** (ESI-TOF) m/z: calcd for $C_{23}H_{25}N_2O_6^+$ $[M + H]^+$, 425.1707; found, 425.1711.



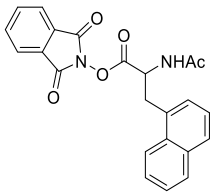
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-acetoxyphenyl)propanoate (**4v**) **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.69 (d, *J* = 8.0 Hz, 1H), 8.04 – 7.89 (m, 4H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 8.5 Hz, 2H), 4.96 (ddd, *J* = 10.0, 8.0, 4.8 Hz, 1H), 3.27 (dd, *J* = 14.0, 4.9 Hz, 1H), 3.07 (dd, *J* = 14.0, 9.9 Hz, 1H), 2.26 (s, 3H), 1.83 (s, 3H).



1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-cyanophenyl)propanoate (**4w**): **Mp**, 206−208 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.71 (d, J = 8.1 Hz, 1H), 7.97 (q, J = 5.6 Hz, 4H), 7.79 (d, J = 7.9 Hz, 2H), 7.57 (d, J = 7.5 Hz, 2H), 5.07 (q, J = 8.5 Hz, 1H), 3.37 (d, J = 5.6 Hz, 1H), 3.16 (t, J = 12.0 Hz, 1H), 1.81 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.6, 168.7, 161.5, 142.2, 135.6, 132.2, 130.4, 128.1, 124.1, 118.8, 109.8, 50.9, 36.3, 22.1; **FTIR** (ν_{max}, cm^{-1}): 3328, 2982, 2919, 2338, 2234, 1790, 1746, 1650, 1528, 1353, 1184, 1098, 973, 875, 698, 542; **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{16}N_3O_5^+$ $[M + H]^+$, 378.1084; found, 378.1086.

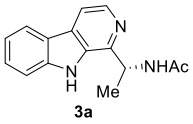


1,3-dioxoisoindolin-2-yl 2-acetamido-3-(naphthalen-2-yl)propanoate (**4x**): **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.75 (d, J = 8.0 Hz, 1H), 8.04 – 7.92 (m, 4H), 7.92 – 7.83 (m, 4H), 7.57 – 7.44 (m, 3H), 5.09 (ddd, J = 9.6, 7.9, 5.1 Hz, 1H), 3.45 (dd, J = 14.0, 5.1 Hz, 1H), 3.25 (dd, J = 14.0, 9.7 Hz, 1H), 1.82 (s, 3H).



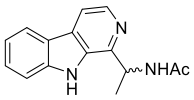
1,3-dioxoisoindolin-2-yl 2-acetamido-3-(naphthalen-1-yl)propanoate (**4y**): **Mp**, 218−220 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.86 (d, J = 8.0 Hz, 1H), 8.12 (d, J = 8.5 Hz, 1H), 8.04 – 7.92 (m, 5H), 7.87 (d, J = 8.2 Hz, 1H), 7.64 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.60 – 7.51 (m, 2H), 7.47 (dd, J = 8.2, 7.0 Hz, 1H), 5.06 (ddd, J = 9.8, 7.9, 4.6 Hz, 1H), 3.82 (dd, J = 14.3, 4.6 Hz, 1H), 3.48 (dd, J = 14.4, 9.8 Hz, 1H), 1.82 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.7, 169.1, 161.6, 135.6, 133.5, 132.0, 131.1, 128.9, 128.2, 127.9, 127.8, 126.6, 125.8, 125.5, 124.1, 122.8, 50.8, 34.0, 22.1; **FTIR** (ν_{max}, cm^{-1}): 3298, 2992, 2359, 1744, 1657, 1548, 1373, 1084, 875, 698, 523; **HRMS** (ESI-TOF) m/z: calcd for $C_{23}H_{19}N_2O_5^+$ $[M + H]^+$, 403.1288; found, 403.1291.

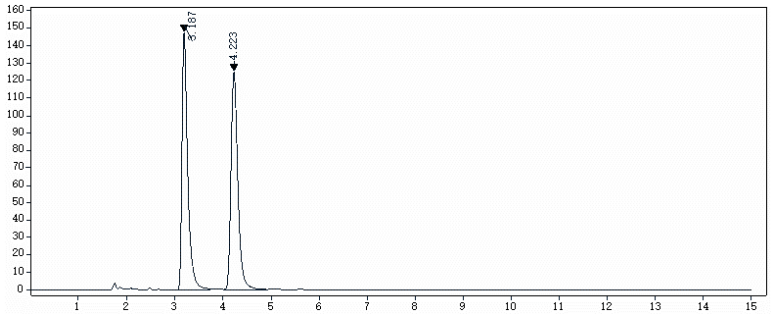
10 Characterization of Products



3a

(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3a**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 43.6 mg, 86% yield; 94% ee). **Mp**: 220−222 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.62 (s, 1H), 8.48 (d, J = 7.8 Hz, 1H), 8.30 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.3 Hz, 1H), 7.61 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.24 (t, J = 7.4 Hz, 1H), 5.62 (p, J = 7.0 Hz, 1H), 1.89 (s, 3H), 1.48 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 146.0, 140.5, 137.2, 132.5, 128.1, 128.0, 121.7, 120.9, 119.4, 113.7, 112.1, 45.9, 22.7, 20.5; **FTIR** (ν_{max}, cm^{-1}): 3172, 2979, 1649, 1626, 1566, 1505, 1430, 1377, 1313, 1287, 1238, 1165, 1149, 746, 737, 624, 587; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{16}N_3O^+$ $[M + H]^+$, 254.1288; found, 254.1285; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.17 min, t_r (minor) = 4.22 min; **$[\alpha]_D^{20}$** = 66.1 (c = 0.3, $CHCl_3$).





Entry	Retention Time	Height	Area	Area%
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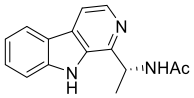
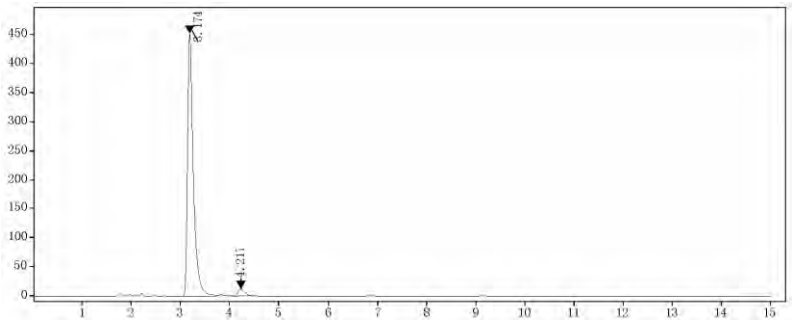
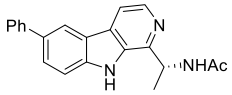
2	4.223	125.147	1231.734	50.057



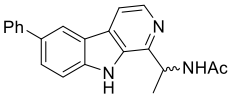


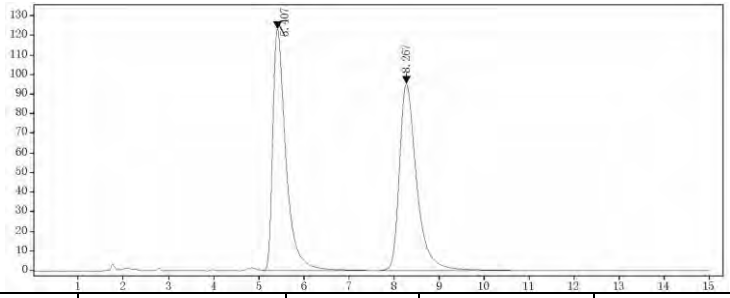
Entry	Retention Time	Height	Area	Area%
1	3.174	452.412	3713.512	97.041
2	4.217	11.712	113.219	2.959



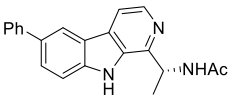
3b

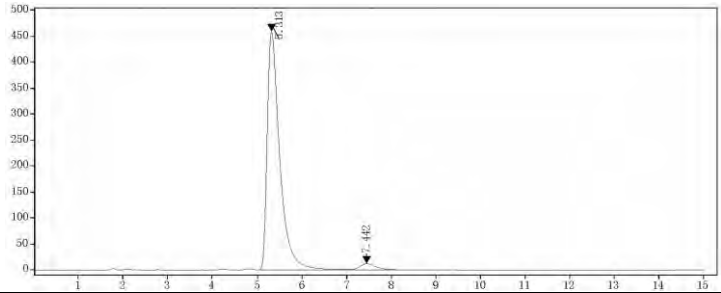
(*R*)-*N*-(1-(6-phenyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3b**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 50.7 mg, 77% yield; 93% ee). **Mp**: 176−178 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.68 (s, 1H), 8.56 (d, J = 1.9 Hz, 1H), 8.49 (d, J = 7.8 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.12 (d, J = 5.3 Hz, 1H), 7.87 (dd, J = 8.6, 1.9 Hz, 1H), 7.78 (dt, J = 6.3, 1.3 Hz, 2H), 7.69 (d, J = 8.5 Hz, 1H), 7.49 (t, J = 7.8 Hz, 2H), 7.40 – 7.30 (m, 1H), 5.63 (p, J = 6.9 Hz, 1H), 1.90 (s, 3H), 1.50 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 146.2, 140.8, 140.0, 137.4, 133.0, 131.8, 128.9, 128.2, 127.2, 126.7, 126.7, 121.6, 119.7, 113.9, 112.5, 45.9, 22.7, 20.5; **FTIR** (ν_{max}, cm^{-1}): 3220, 3177, 2988, 2906, 2384, 2347, 1650, 1558, 1493, 1375, 1239, 1062, 813, 748, 700, 598; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{20}N_3O^+$ [M + H]$^+$, 330.1601; found, 330.1595; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 5.31 min, t_r (minor) = 7.44 min; **[α]$_D^{20}$** = 64.9 (c = 0.3, $CHCl_3$).



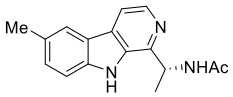


Entry	Retention Time	Height	Area	Area%
1	5.407	123.377	2509.735	49.807
2	8.267	95.286	2529.161	50.193



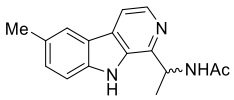


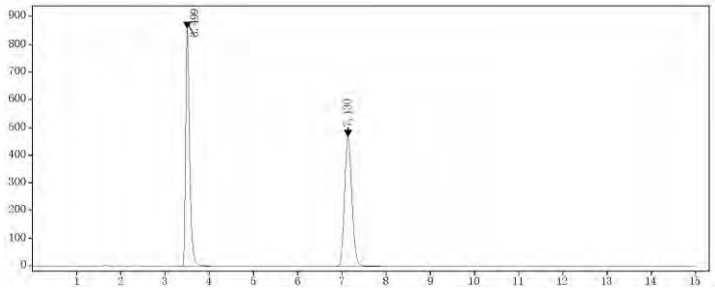
Entry	Retention Time	Height	Area	Area%
1	5.313	459.175	8991.402	96.471
2	7.442	12.318	328.904	3.529



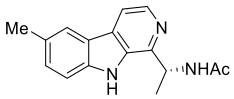
3c

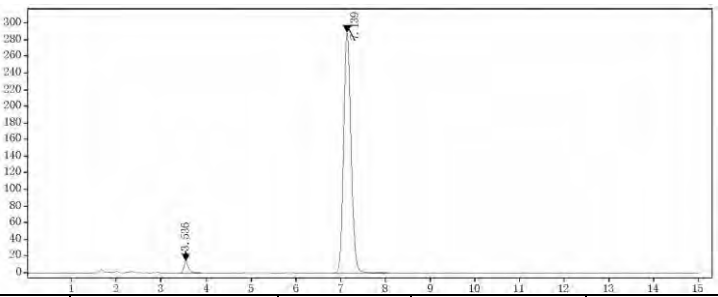
(*R*)-*N*-(1-(6-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3c**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 43.8 mg, 82% yield; 94% ee). **Mp**: 177−179 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.45 (s, 1H), 8.44 (d, J = 7.8 Hz, 1H), 8.27 (d, J = 5.2 Hz, 1H), 7.99 (s, 1H), 7.96 (d, J = 5.2 Hz, 1H), 7.50 (d, J = 8.3 Hz, 1H), 7.43 – 7.28 (m, 1H), 5.60 (p, J = 7.0 Hz, 1H), 2.47 (s, 3H), 1.89 (s, 3H), 1.48 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.8, 145.9, 138.8, 137.0, 132.7, 129.6, 128.1, 127.7, 121.1, 121.0, 113.6, 111.8, 45.9, 22.7, 21.0, 20.5; **FTIR** (ν_{max}, cm^{-1}): 3157, 2983, 2923, 2858, 2378, 1658, 1560, 1508, 1448, 1371, 1296, 1242, 1152, 1059, 793, 620, 564; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O^+$ [M + H]$^+$, 268.1444; found, 268.1441; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 7.14 min, t_r (minor) = 3.54 min; **[α]$_D^{20}$** = 41.4 (c = 0.3, $CHCl_3$).



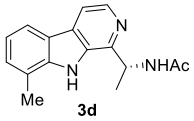


Entry	Retention Time	Height	Area	Area%
1	3.499	856.564	5380.467	49.998
2	7.130	466.334	5380.976	50.002



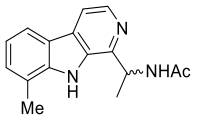


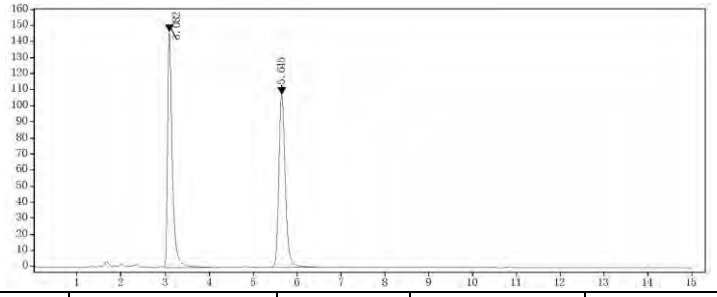
Entry	Retention Time	Height	Area	Area%
1	3.536	13.984	101.150	2.924
2	7.139	288.735	3357.949	97.076



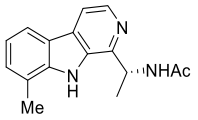
3d

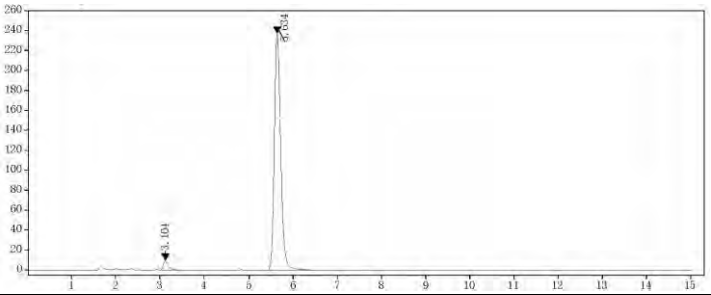
(*R*)-*N*-(1-(8-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3d**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 40.1 mg, 75% yield; 94% ee). **Mp**: 230−232 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.26 (s, 1H), 8.51 (d, J = 8.0 Hz, 1H), 8.29 (d, J = 5.2 Hz, 1H), 8.03 (d, J = 7.9 Hz, 1H), 7.99 (d, J = 5.2 Hz, 1H), 7.34 (d, J = 7.1 Hz, 1H), 7.15 (t, J = 7.5 Hz, 1H), 5.78 (p, J = 6.9 Hz, 1H), 2.62 (s, 3H), 1.91 (s, 3H), 1.49 (d, J = 6.7 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 146.2, 139.8, 137.3, 132.6, 128.5, 128.4, 121.4, 120.6, 119.6, 119.0, 113.7, 45.8, 22.7, 20.5, 17.2; **FTIR** (ν_{max}, cm^{-1}): 3380, 3270, 2919, 2382, 1653, 1542, 1433, 1372, 1288, 1232, 1029, 992, 820, 765, 559; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O^+$ [M + H]$^+$, 268.1444; found, 268.1440; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 μm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 5.63 min, t_r (minor) = 3.10 min; **[α]$_D^{20}$** = 64.4 (c = 0.3, $CHCl_3$).



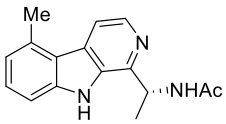


Entry	Retention Time	Height	Area	Area%
1	3.082	56.566	437.870	50.069
2	5.645	42.1330	436.660	49.931



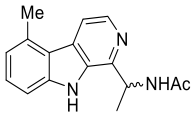


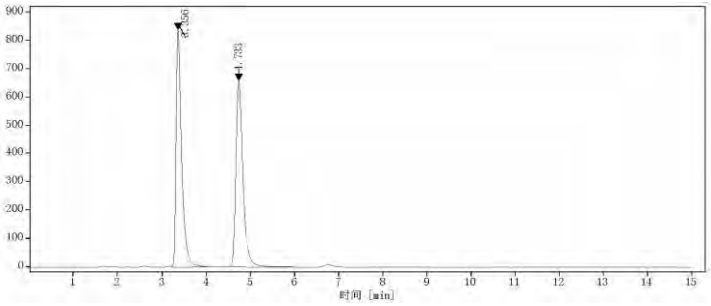
Entry	Retention Time	Height	Area	Area%
1	3.104	3.661	31.016	3.169
2	5.634	93.214	947.577	96.831



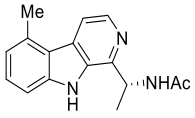
3e

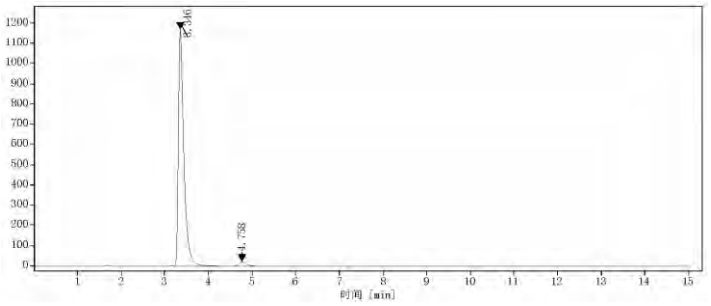
(*R*)-*N*-(1-(5-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3e**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 43.3 mg, 81% yield; 95% ee). **Mp**: 192−194 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.66 (s, 1H), 8.51 (d, J = 7.8 Hz, 1H), 8.30 (d, J = 5.3 Hz, 1H), 7.97 (d, J = 5.5 Hz, 1H), 7.43 (d, J = 7.3 Hz, 2H), 7.02 (d, J = 6.4 Hz, 1H), 5.74 – 5.50 (m, 1H), 2.80 (s, 3H), 1.89 (s, 3H), 1.47 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 145.8, 140.6, 137.3, 133.9, 132.4, 128.1, 127.9, 120.6, 119.7, 115.4, 109.6, 45.9, 22.7, 20.6, 20.2; **FTIR** (ν_{max}, cm^{-1}): 2974, 2828, 2382, 2148, 1701, 1648, 1541, 1500, 1473, 1457, 1238, 1089, 1048, 738, 639, 540; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O^+$ [M + H]$^+$, 268.1444; found, 268.1440; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 3.35 min, t_r (minor) = 4.76 min; **[α]$_D^{20}$** = 55.2 (c = 0.3, $CHCl_3$).



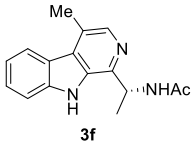


Entry	Retention Time	Height	Area	Area%
1	3.356	836.973	7302.026	49.992
2	4.733	656.555	7304.381	50.008



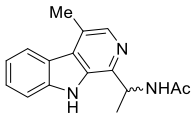


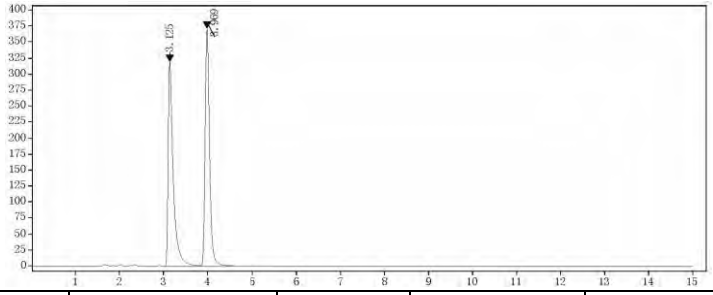
Entry	Retention Time	Height	Area	Area%
1	3.346	1169.016	10107.821	97.275
2	4.758	22.557	283.183	2.725



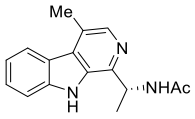
3f

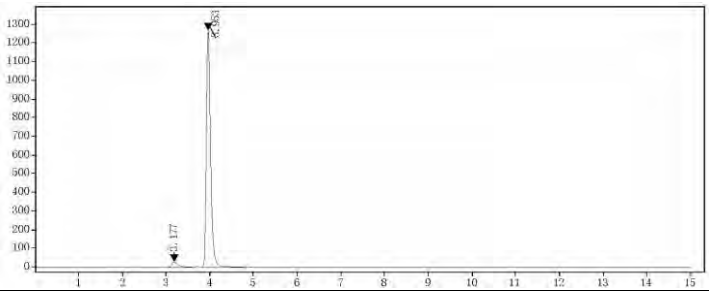
(*R*)-*N*-(1-(4-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3f**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 40.6 mg, 76% yield; 94% ee). **Mp**: 219−221 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.60 (s, 1H), 8.43 (d, J = 7.8 Hz, 1H), 8.19 (d, J = 7.9 Hz, 1H), 8.10 (d, J = 1.0 Hz, 1H), 7.68 – 7.59 (m, 1H), 7.54 (ddd, J = 8.3, 7.0, 1.2 Hz, 1H), 7.25 (ddd, J = 8.0, 7.0, 1.1 Hz, 1H), 5.59 (p, J = 6.9 Hz, 1H), 2.76 (d, J = 0.7 Hz, 3H), 1.88 (s, 3H), 1.47 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.8, 143.8, 140.4, 137.6, 132.0, 127.4, 126.6, 125.3, 123.1, 121.4, 119.4, 111.9, 45.6, 22.7, 20.5, 16.8; **FTIR** (ν_{max}, cm^{-1}): 3298, 3155, 2920, 2879, 2384, 2337, 1653, 1459, 1273, 1108, 731, 520; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O^+$ [M + H]$^+$, 268.1444; found, 268.1440; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 3.95 min, t_r (minor) = 3.18 min; **[α]$_D^{20}$** = 40.3 (c = 0.3, $CHCl_3$).



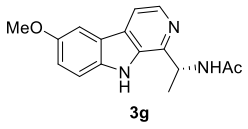


Entry	Retention Time	Height	Area	Area%
1	3.125	319.183	2578.978	49.722
2	3.969	371.207	2607.772	50.278



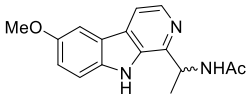


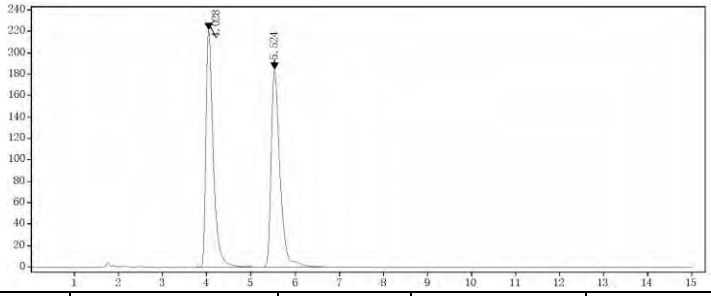
Entry	Retention Time	Height	Area	Area%
1	3.177	28.453	284.156	3.216
2	3.953	1269.584	8550.2555	96.784



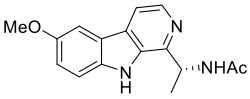
3g

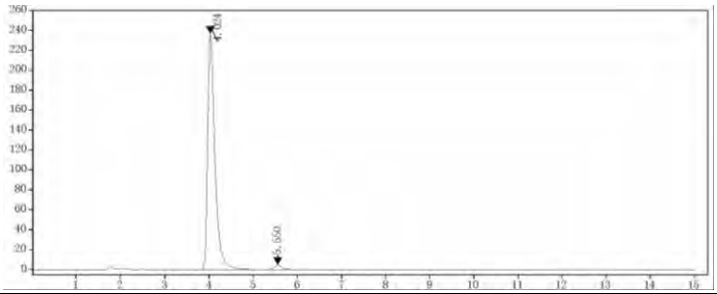
(*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3g**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 40.8 mg, 72% yield; 96% ee). **Mp**: 175−177 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.40 (s, 1H), 8.44 (d, J = 7.8 Hz, 1H), 8.26 (d, J = 5.3 Hz, 1H), 7.99 (d, J = 5.3 Hz, 1H), 7.76 (d, J = 2.5 Hz, 1H), 7.52 (d, J = 8.9 Hz, 1H), 7.19 (dd, J = 8.8, 2.5 Hz, 1H), 5.60 (p, J = 7.0 Hz, 1H), 3.86 (s, 3H), 1.90 (s, 3H), 1.48 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.8, 153.4, 146.0, 136.6, 135.3, 133.1, 127.8, 121.2, 118.1, 113.7, 112.9, 103.4, 55.6, 45.9, 22.7, 20.4; **FTIR** (ν_{max}, cm^{-1}): 3228, 3179, 2925, 2850, 2384, 1641, 1558, 1500, 1437, 1377, 1287, 1290, 1210, 1157, 1024, 804, 625, 557; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O_2^+$ [M + H]$^+$, 284.1394; found, 284.1390; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 4.02 min, t_r (minor) = 5.55 min; **[α]$_D^{20}$** = 51.7 (c = 0.3, $CHCl_3$).



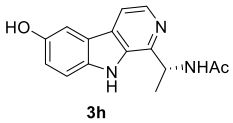


Entry	Retention Time	Height	Area	Area%
1	4.028	221.358	2561.607	49.909
2	5.524	184.128	2570.966	50.091



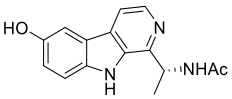


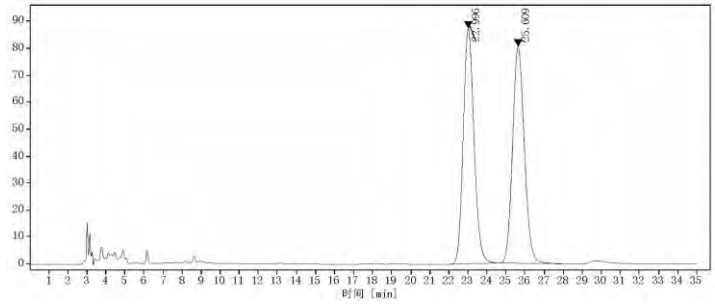
Entry	Retention Time	Height	Area	Area%
1	4.024	237.214	2752.534	97.928
2	5.550	4.672	58.246	2.072



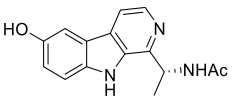
3h

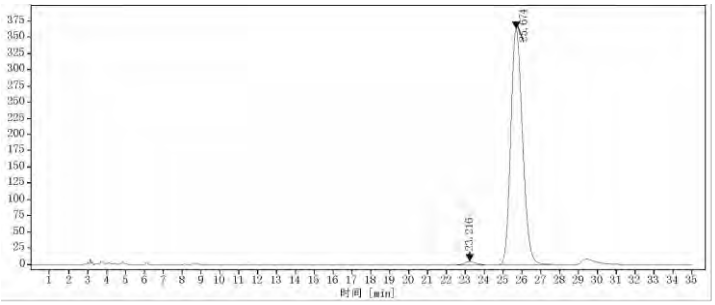
(*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3h**) was prepared as a brown amorphous powder according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 33.9 mg, 63% yield; 97% ee). **Mp**: 259−261 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.28 (s, 1H), 9.17 (s, 1H), 8.46 (d, J = 7.8 Hz, 1H), 8.21 (d, J = 5.2 Hz, 1H), 7.90 (d, J = 5.2 Hz, 1H), 7.48 (d, J = 2.3 Hz, 1H), 7.42 (d, J = 8.7 Hz, 1H), 7.06 (dd, J = 8.7, 2.4 Hz, 1H), 5.56 (p, J = 6.9 Hz, 1H), 1.88 (s, 3H), 1.45 (d, J = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 150.9, 145.9, 136.4, 134.6, 133.1, 127.6, 121.6, 118.2, 113.7, 112.6, 105.6, 45.9, 22.7, 20.5; **FTIR** (ν_{max}, cm^{-1}): 2925, 2855, 2381, 1737, 1645, 1464, 1375, 1268, 1025, 820, 759, 723; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{16}N_3O_2^+$ [M + H]$^+$, 270.1237; found, 270.1233; **HPLC analysis:** Chiral NY(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 0.5 mL/min, 25 ºC, 254 nm), t_r (major) = 6.77 min, t_r (minor) = 8.94 min; **[α]$_D^{20}$** = 9.3 (c = 0.3, $CHCl_3$).



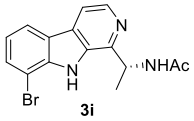


Entry	Retention Time	Height	Area	Area%
1	22.996	87.175	3430.610	49.856
2	25.609	80.307	3450.481	50.144



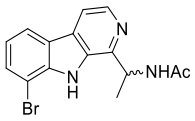


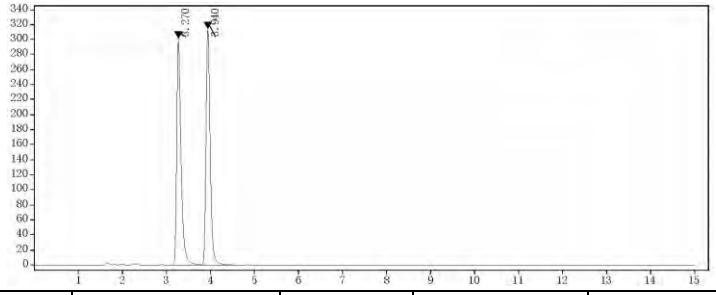
Entry	Retention Time	Height	Area	Area%
1	23.216	5.243	218.059	1.361
2	25.674	362.874	15804.163	98.639



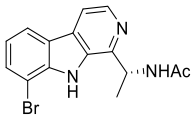
3i

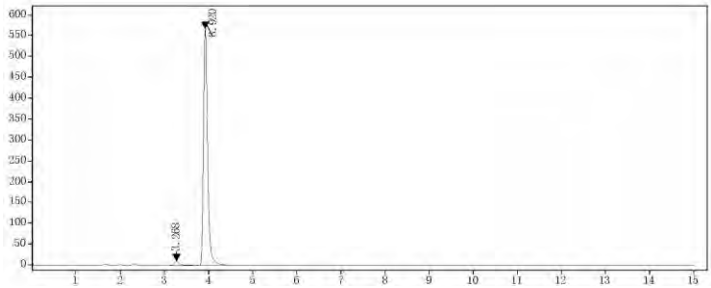
(*R*)-*N*-(1-(8-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3i**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 51.2 mg, 77% yield; 96% ee). **Mp**: 221−223 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.45 (s, 1H), 8.58 (d, J = 8.1 Hz, 1H), 8.36 (d, J = 5.3 Hz, 1H), 8.25 (d, J = 7.8 Hz, 1H), 8.05 (d, J = 5.2 Hz, 1H), 7.77 (d, J = 7.6 Hz, 1H), 7.20 (t, J = 7.8 Hz, 1H), 5.92 – 5.72 (m, 1H), 1.90 (s, 3H), 1.53 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.5, 146.9, 138.8, 138.1, 132.9, 130.6, 128.4, 122.9, 121.1, 120.9, 114.1, 104.5, 45.7, 22.6, 19.6; **FTIR** (ν_{max}, cm^{-1}): 3208, 3157, 2983, 2903, 2383, 2339, 1650, 1542, 1492, 1426, 1370, 1320, 1283, 1133, 835, 769, 675, 600; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{15}BrN_3O^+$ [M + H]$^+$, 332.0393; found, 332.0388; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.92 min, t_r (minor) = 3.27 min; **[α]$_D^{20}$** = 87.1 (c = 0.3, $CHCl_3$).



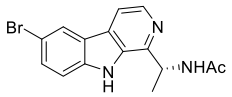


Entry	Retention Time	Height	Area	Area%
1	3.270	301.690	2080.315	49.760
2	3.940	314.211	2100.365	50.240



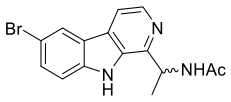


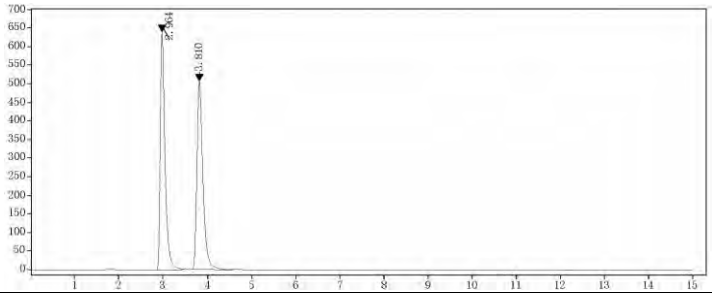
Entry	Retention Time	Height	Area	Area%
1	3.268	9.948	84.18	2.249
2	3.920	566.548	3658.575	97.751



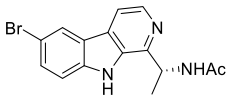
3j

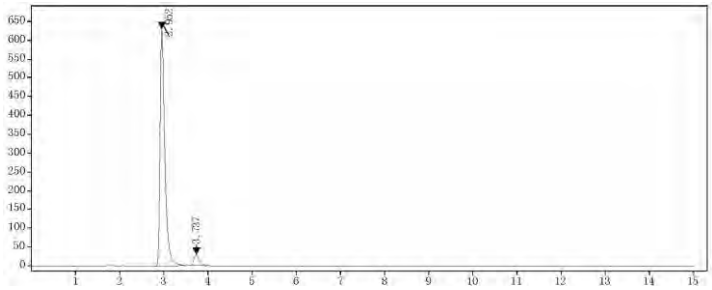
(*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3j**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 51.8 mg, 78% yield; 90% ee). **Mp**: 188−190 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.72 (s, 1H), 8.47 (d, J = 2.0 Hz, 1H), 8.40 (d, J = 7.7 Hz, 1H), 8.32 (d, J = 5.3 Hz, 1H), 8.06 (d, J = 5.2 Hz, 1H), 7.65 (dd, J = 8.7, 2.0 Hz, 1H), 7.58 (d, J = 8.7 Hz, 1H), 5.60 (p, J = 7.0 Hz, 1H), 1.89 (s, 3H), 1.49 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.8, 146.3, 139.1, 137.5, 132.8, 130.4, 126.9, 124.1, 122.8, 114.0, 113.9, 111.2, 45.9, 22.6, 20.2; **FTIR** (ν_{max}, cm^{-1}): 3218, 3165, 2979, 2850, 1653, 1556, 1495, 1375, 1275, 1239, 1070, 818, 801, 610, 582; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{15}BrN_3O^+$ [M + H]$^+$, 332.0393; found, 332.0390; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 2.95 min, t_r (minor) = 3.74 min; **[α]$_D^{20}$** = 43.4 (c = 0.3, $CHCl_3$).



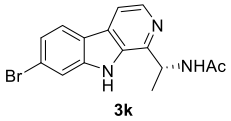


Entry	Retention Time	Height	Area	Area%
1	2.964	639.493	4782.509	50.017
2	3.810	505.904	4779.266	49.983



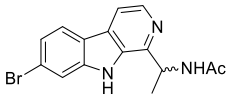


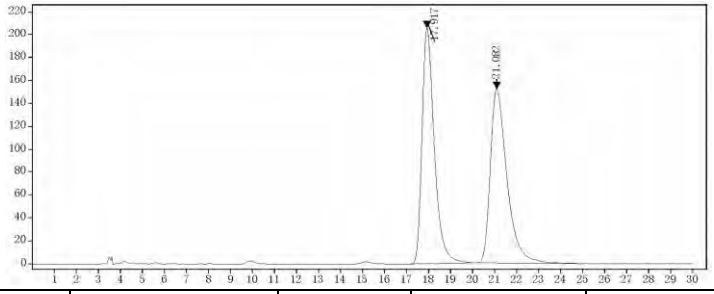
Entry	Retention Time	Height	Area	Area%
1	2.952	630.118	4724.377	94.811
2	3.737	28.962	258.570	5.189



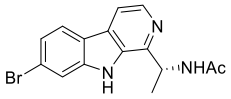
3k

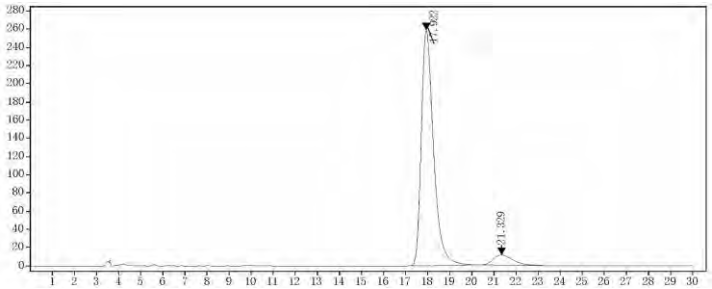
(*R*)-*N*-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3k**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 53.2 mg, 80% yield; 87% ee). **Mp**: 245−247 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.74 (s, 1H), 8.46 (d, J = 7.8 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 8.03 (d, J = 5.3 Hz, 1H), 7.78 (d, J = 1.7 Hz, 1H), 7.38 (dd, J = 8.4, 1.8 Hz, 1H), 5.59 (p, J = 6.9 Hz, 1H), 1.88 (s, 3H), 1.48 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 146.3, 141.3, 137.9, 132.7, 127.5, 123.5, 122.4, 120.8, 120.1, 114.6, 113.7, 45.9, 22.6, 20.4; **FTIR** (ν_{max}, cm^{-1}): 3202, 2988, 2974, 2901, 2382, 2342, 1648, 1623, 1560, 1542, 1421, 1375, 1314, 1245, 1078, 1048, 841, 790, 640, 583; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{15}BrN_3O^+$ $[M + H]^+$, 332.0393; found, 332.0388; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 ºC, 254 nm), t_r (major) = 17.92 min, t_r (minor) = 21.33 min; **$[\alpha]_D^{20}$** = -65.3 (c = 0.3, $CHCl_3$).



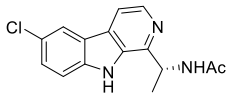


Entry	Retention Time	Height	Area	Area%
1	17.917	205.546	7958.969	49.914
2	21.082	151.783	7986.241	50.086



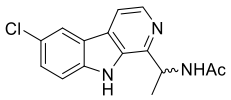


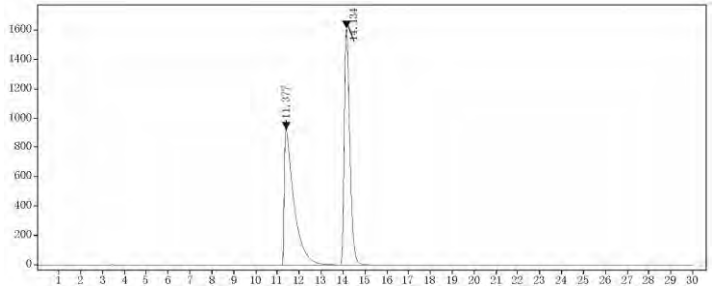
Entry	Retention Time	Height	Area	Area%
1	17.922	258.978	9951.482	93.439
2	21.329	11.121	698.789	6.561



3l

(*R*)-*N*-(1-(6-chloro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3l**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 50.0 mg, 87% yield; 86% ee). **Mp**: 202−204 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.78 (s, 1H), 8.47 (d, J = 7.7 Hz, 1H), 8.33 (dd, J = 10.2, 3.7 Hz, 2H), 8.06 (d, J = 5.2 Hz, 1H), 7.63 (d, J = 8.7 Hz, 1H), 7.54 (dd, J = 8.6, 2.1 Hz, 1H), 5.60 (p, J = 7.0 Hz, 1H), 1.89 (s, 3H), 1.48 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 146.5, 138.9, 137.5, 133.1, 128.0, 127.1, 123.6, 122.2, 121.2, 114.0, 113.7, 45.9, 22.6, 20.4; **FTIR** (ν_{max}, cm^{-1}): 3397, 3279, 2905, 2371, 2287, 2134, 1648, 1387, 1256, 1140, 1021, 985, 782, 576; **HRMS** (ESI-TOF) m/z: calcd for $C_{15}H_{15}ClN_3O^+$ $[M + H]^+$, 288.0898; found, 288.0894; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 14.24 min, t_r (minor) = 12.17 min; **[α]$_D^{20}$** = 48.7 (c = 0.3, $CHCl_3$).





Entry	Retention Time	Height	Area	Area%
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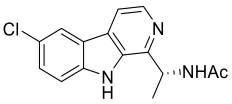
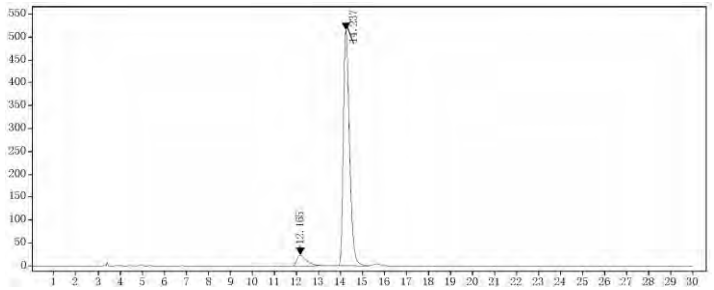
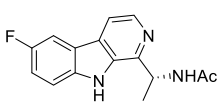
2	14.134	1612.009	28440.597	49.989



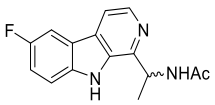


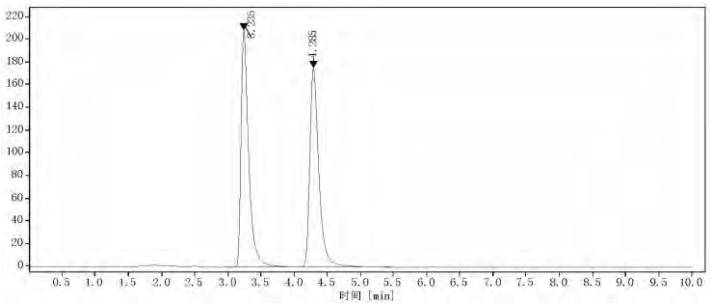
Entry	Retention Time	Height	Area	Area%
1	12.165	23.513	743.047	6.923
2	14.237	514.738	9989.662	93.077



3m

(*R*)-*N*-(1-(6-fluoro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3m**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 45.5 mg, 84% yield; 91% ee). **mp** 206−208 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.60 (s, 1H), 8.39 (d, *J* = 7.8 Hz, 1H), 8.30 (d, *J* = 5.3 Hz, 1H), 8.14 – 7.92 (m, 2H), 7.62 (dd, *J* = 8.9, 4.4 Hz, 1H), 7.40 (td, *J* = 9.3, 2.6 Hz, 1H), 5.60 (p, *J* = 7.0 Hz, 1H), 1.90 (s, 3H), 1.49 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.8, 156.5 (d, J^1 = 235.3 Hz), 146.4, 137.0, 136.9, 133.4, 127.6 (d, J^4 = 4.0 Hz) 121.2 (d, J^3 = 10.1 Hz), 116.1 (J^2 = 25.2 Hz), 113.8, 113.1 (J^3 = 9.1 Hz), 106.8 (J^2 = 24.2 Hz), 45.9, 22.6, 20.2. **^{19}F NMR** (376 MHz, DMSO) δ -123.87 (sextet); **FTIR** (ν_{max}, cm^{-1}): 3218, 3175, 3102, 3037, 2838, 2357, 1655, 1580, 1505, 1283, 1159, 816, 620; HRMS (ESI-TOF) m/z: calcd for $C_{15}H_{15}FN_3O^+$ $[M + H]^+$, 272.1194; found, 272.1190; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.22 min, t_r (minor) = 4.28 min; **$[\alpha]_D^{20}$** = 59.7 (c = 0.3, $CHCl_3$).





Entry	Retention Time	Height	Area	Area%
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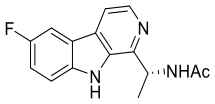
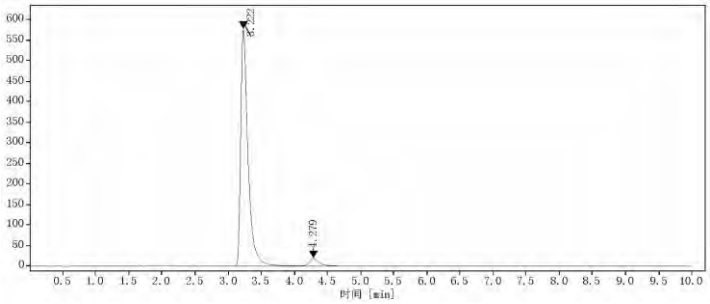
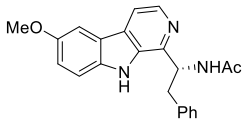
2	4.285	174.687	1655.082	49.922



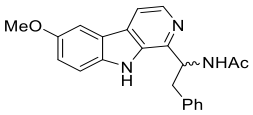


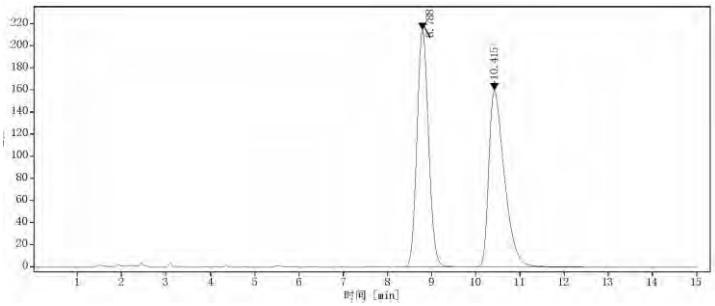
Entry	Retention Time	Height	Area	Area%
1	3.222	578.451	4452.078	95.551
2	4.279	19.117	207.311	4.449



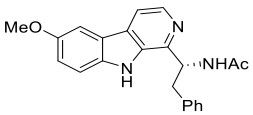
3n

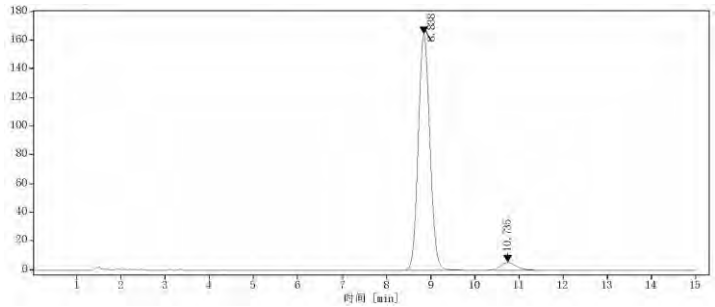
(*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3n**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 59.7 mg, 83% yield; 92% ee). **Mp**: 189−191 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.40 (s, 1H), 8.55 (d, J = 8.4 Hz, 1H), 8.28 (d, J = 5.3 Hz, 1H), 8.00 (d, J = 5.3 Hz, 1H), 7.75 (d, J = 2.5 Hz, 1H), 7.52 (d, J = 8.8 Hz, 1H), 7.26 (d, J = 7.4 Hz, 2H), 7.19 (dt, J = 8.9, 6.4 Hz, 3H), 7.12 (t, J = 7.2 Hz, 1H), 5.77 (td, J = 8.5, 5.7 Hz, 1H), 3.85 (s, 3H), 3.19 (qd, J = 13.7, 7.3 Hz, 2H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 153.3, 145.1, 138.5, 136.7, 135.4, 133.6, 129.4, 127.8, 126.0, 121.1, 118.2, 113.8, 112.9, 103.4, 55.6, 51.2, 38.8, 22.5.; **FTIR** (ν_{max}, cm^{-1}): 3211, 3177, 3032, 2991, 2382, 1655, 1575, 1498, 1377, 1290, 1210, 1171, 1031, 813, 731, 620; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{22}N_3O_2$+ $[M + H]^+$, 360.1707; found, 360.1701; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 8.84 min, t_r (minor) = 10.74 min; **[α]$_D^{20}$** = 40.1 (c = 0.3, $CHCl_3$).



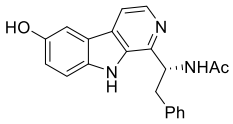


Entry	Retention Time	Height	Area	Area%
1	8.788	214.163	3909.052	49.927
2	10.415	159.330	3920.541	50.073



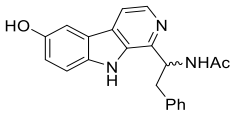


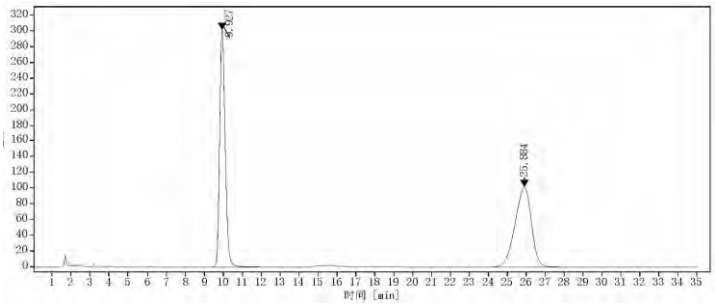
Entry	Retention Time	Height	Area	Area%
1	8.838	164.703	3007.525	95.755
2	10.735	5.267	133.338	4.245



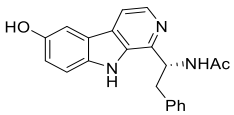
3o

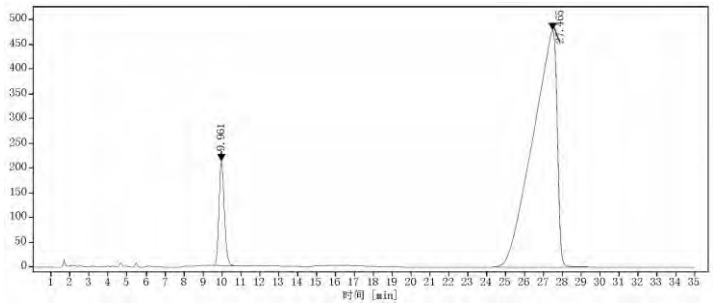
(*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3o**) was prepared as a brown amorphous powder according to the General Procedure I (purification by flash column chromatography: 30% acetone in hexanes, 60.1 mg, 87% yield; 82% ee). **Mp**: 251−252 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.23 (s, 1H), 9.12 (s, 1H), 8.51 (d, J = 8.5 Hz, 1H), 8.24 (d, J = 5.2 Hz, 1H), 7.90 (d, J = 5.2 Hz, 1H), 7.48 (d, J = 2.4 Hz, 1H), 7.43 (d, J = 8.7 Hz, 1H), 7.26 (d, J = 7.0 Hz, 2H), 7.20 (t, J = 7.5 Hz, 2H), 7.12 (t, J = 7.1 Hz, 1H), 7.06 (dd, J = 8.7, 2.4 Hz, 1H), 5.76 (td, J = 8.7, 5.8 Hz, 1H), 3.29 – 3.09 (m, 2H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 150.9, 144.9, 138.5, 136.5, 134.6, 133.6, 129.5, 127.9, 127.7, 126.1, 121.5, 118.2, 113.8, 112.6, 105.5, 51.2, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3385, 3289, 2382, 1984, 1650, 1570, 1495, 1379, 1239, 1208, 1024, 992, 820, 648, 582; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{20}N_3O_2^+$ [M + H]$^+$, 346.1550; found, 346.1545; **HPLC analysis:** Chiral NQ(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 0.5 mL/min, 25 ºC, 254 nm), t_r (major) =27.47 min, t_r (minor) = 9.96 min; **[α]$_D^{20}$** = 14.8 (c = 0.3, $CHCl_3$).



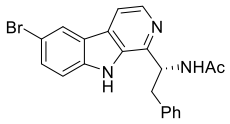


Entry	Retention Time	Height	Area	Area%
1	9.927	301.058	6110.340	49.962
2	25.884	101.368	6119.585	50.038



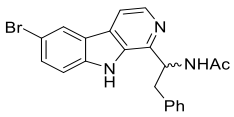


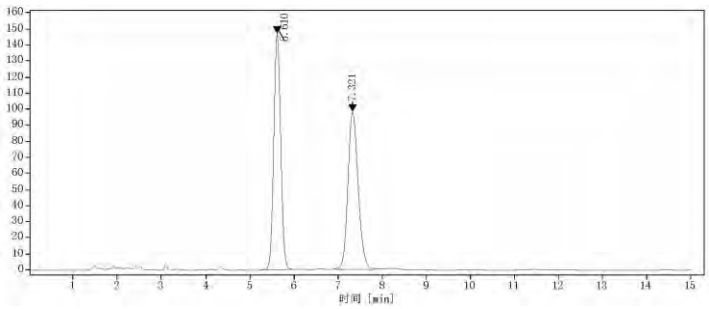
Entry	Retention Time	Height	Area	Area%
1	9.961	210.819	4185.877	8.944
2	27.465	479.163	42616.374	91.056



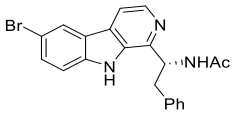
3p

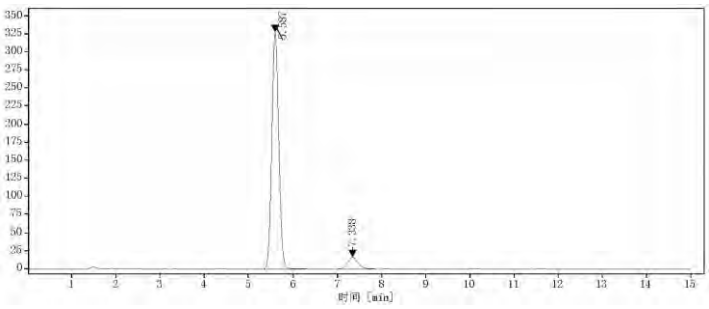
(*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3p**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 65.4 mg, 80% yield; 86% ee). **Mp**: 212−214 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.78 (s, 1H), 8.58 (d, J = 8.3 Hz, 1H), 8.53 – 8.42 (m, 1H), 8.35 (d, J = 5.3 Hz, 1H), 8.07 (d, J = 5.2 Hz, 1H), 7.74 – 7.61 (m, 1H), 7.57 (d, J = 8.7 Hz, 1H), 7.25 (d, J = 7.5 Hz, 2H), 7.19 (t, J = 7.4 Hz, 2H), 7.11 (t, J = 7.2 Hz, 1H), 5.78 (q, J = 8.0 Hz, 1H), 3.19 (q, J = 8.3 Hz, 2H), 1.78 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.5, 139.2, 138.3, 137.7, 133.4, 130.5, 129.4, 127.9, 127.0, 126.1, 124.3, 122.7, 114.1, 111.2, 51.2, 39.6, 22.5.; **FTIR** (ν_{max}, cm^{-1}): 3165, 3035, 2984, 2387, 1651, 1539, 1488, 1273, 1232, 1072, 823, 804, 741, 698, 603; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{19}BrN_3O^+$ [M + H]$^+$, 408.0706; found, 408.0702; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 5.59 min, t_r (minor) = 7.34 min; **$[\alpha]_D^{20}$** = 32.7 (c = 0.3, $CHCl_3$).



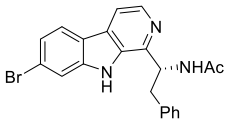


Entry	Retention Time	Height	Area	Area%
1	5.610	146.865	1602.415	50.198
2	7.321	98.056	1589.768	49.802



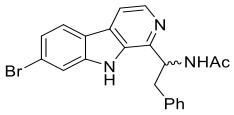


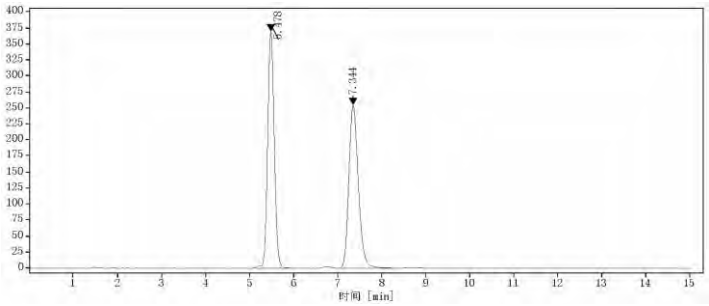
Entry	Retention Time	Height	Area	Area%
1	5.587	327.484	3615.801	93.051
2	7.338	16.421	270.038	6.949



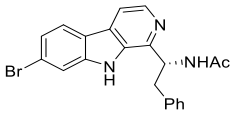
3q

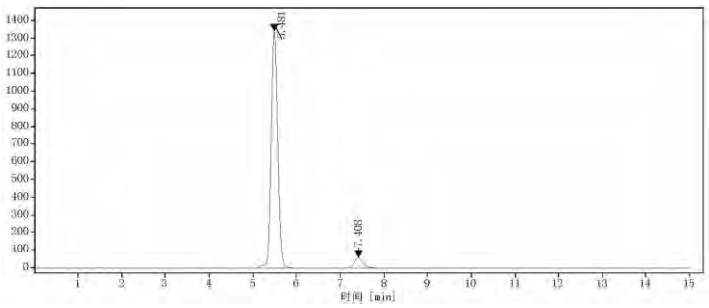
N-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**lmp-6-26**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 63.7 mg, 78% yield; 88% ee). **Mp**: 245−247 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.74 (s, 1H), 8.61 (d, J = 8.3 Hz, 1H), 8.36 (d, J = 4.9 Hz, 1H), 8.18 (d, J = 8.3 Hz, 1H), 8.04 (d, J = 5.2 Hz, 1H), 7.77 (s, 1H), 7.37 (d, J = 8.1 Hz, 1H), 7.32 – 7.15 (m, 4H), 7.11 (d, J = 7.2 Hz, 1H), 5.77 (q, J = 7.7 Hz, 1H), 3.19 (q, J = 7.7 Hz, 2H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 145.4, 141.3, 138.3, 138.0, 133.2, 129.4, 127.9, 127.5, 126.1, 123.6, 122.3, 120.9, 120.0, 114.6, 113.9, 51.2, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3223, 3166, 3022, 2853, 2359, 1648, 1624, 1588, 1541, 1495, 1312, 1232, 1048, 920, 845, 794, 716, 574; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{19}BrN_3O^+$ [M + H]$^+$, 408.0706; found, 408.0704; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 5.48 min, t_r (minor) = 7.41 min; **$[\alpha]_D^{20}$** = 15.0 (c = 0.3, $CHCl_3$).



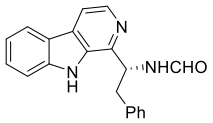


Entry	Retention Time	Height	Area	Area%
1	5.478	369.694	3839.386	50.186
2	7.344	254.36	3810.968	49.814



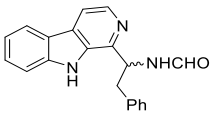


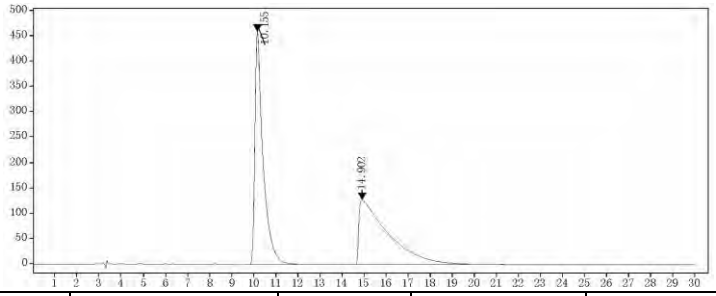
Entry	Retention Time	Height	Area	Area%
1	5.481	1339.625	13961.656	93.976
2	7.408	58.145	895.009	6.024



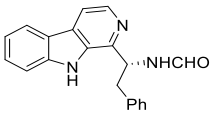
3r

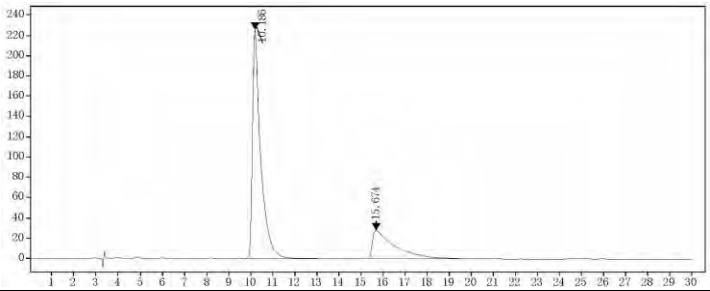
(*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3r**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 49.2 mg, 78% yield; 50% ee). **Mp**: 217−219 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.72 (s, 1H), 8.80 (d, *J* = 8.5 Hz, 1H), 8.33 (d, *J* = 5.2 Hz, 1H), 8.22 (d, *J* = 8.0 Hz, 1H), 8.04 (d, *J* = 5.2 Hz, 1H), 8.00 (s, 1H), 7.62 (d, *J* = 8.3 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.29 – 7.16 (m, 5H), 7.12 (t, *J* = 7.0 Hz, 1H), 5.92 (td, *J* = 8.7, 5.7 Hz, 1H), 3.19 (qd, *J* = 13.7, 7.2 Hz, 2H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 160.7, 144.3, 140.6, 138.0, 137.4, 132.9, 129.5, 128.2, 128.1, 127.9, 126.2, 121.7, 120.8, 119.4, 113.9, 112.0, 49.8; **FTIR** (ν_{max}, cm^{-1}): 3165, 2986, 2872, 2359, 1655, 1544, 1505, 482, 1454, 1375, 1237, 1050, 823, 779, 731, 700, 571; **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{18}N_3O^+$ [M + H]$^+$, 316.1444; found, 316.1441; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 85:15, 0.5 mL/min, 25 ºC, 254 nm), t_r (major) = 10.19 min, t_r (minor) = 15.67 min; **[α]$_D^{20}$** = 7.5 (c = 0.3, $CHCl_3$).



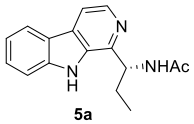


Entry	Retention Time	Height	Area	Area%
1	10.155	458.171	11772.060	50.288
2	14.902	126.169	11637.382	49.712



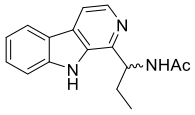


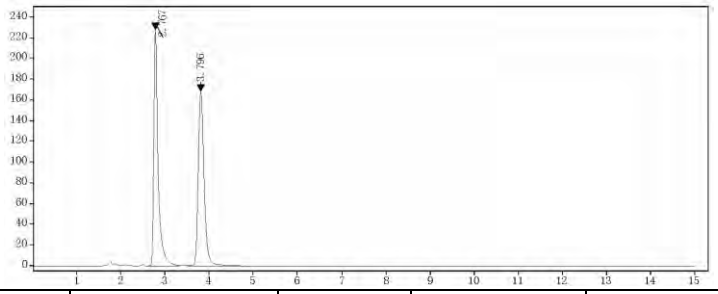
Entry	Retention Time	Height	Area	Area%
1	10.186	225.628	5817.220	75.177
2	15.674	27.441	1920.782	24.823



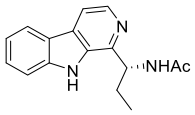
5a

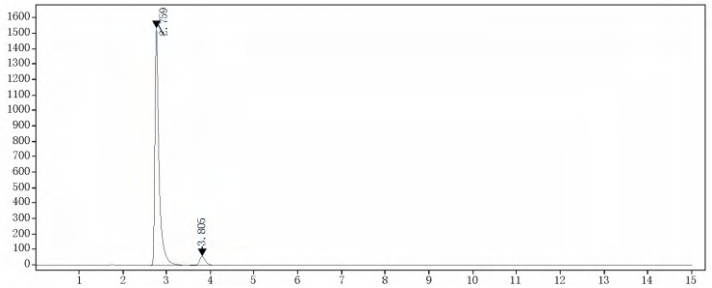
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5a**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 43.3 mg, 81% yield; 90% ee). **Mp**: 218−220 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.64 (s, 1H), 8.40 (d, J = 8.2 Hz, 1H), 8.31 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.8 Hz, 1H), 8.00 (d, J = 5.2 Hz, 1H), 7.61 (d, J = 8.2 Hz, 1H), 7.58 – 7.48 (m, 1H), 7.23 (t, J = 7.4 Hz, 1H), 5.49 (q, J = 7.5 Hz, 1H), 1.99 – 1.78 (m, 2H), 1.90 (s, 3H), 0.86 (t, J = 7.3 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 145.5, 140.5, 137.3, 133.1, 128.1, 127.8, 121.7, 120.9, 119.3, 113.5, 112.0, 51.3, 27.5, 22.6, 10.5; **FTIR** (ν_{max}, cm^{-1}): 3649, 3175, 2993, 2899, 2357, 2345, 1655, 1539, 1503, 1459, 1314, 1239, 1058, 874, 738, 564; **HRMS** (ESI-TOF) m/z: calcd for $C_{16}H_{18}N_3O^+$ [M + H]$^+$, 268.1444; found, 268.1441; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 2.76 min, t_r (minor) = 3.81 min; **$[\alpha]_D^{20}$** = -41.7 (c = 0.3, $CHCl_3$).



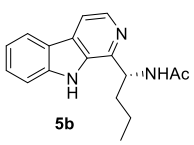


Entry	Retention Time	Height	Area	Area%
1	2.767	227.484	1548.524	49.799
2	3.796	167.978	1561.037	50.201

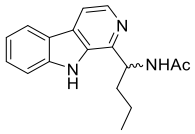


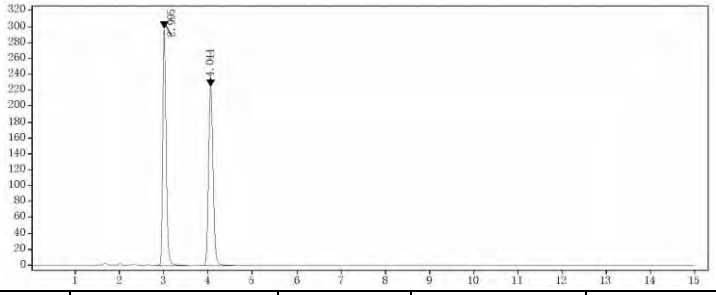


Entry	Retention Time	Height	Area	Area%
1	2.759	1534.351	10026.186	94.948
2	3.805	57.601	533.504	5.052

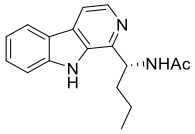


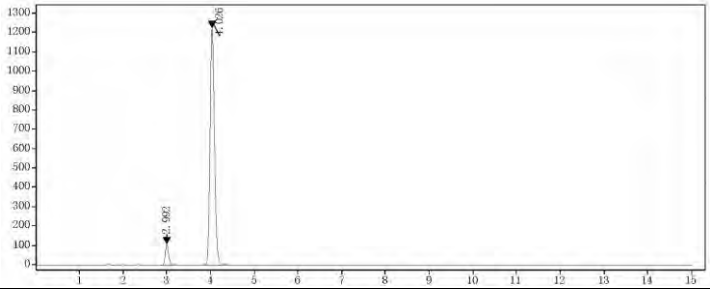
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5b**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 41.1 mg, 73% yield; 87% ee). **Mp**: 182−184 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.60 (s, 1H), 8.40 (d, J = 8.2 Hz, 1H), 8.30 (d, J = 5.2 Hz, 1H), 8.20 (d, J = 7.9 Hz, 1H), 8.00 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.23 (t, J = 7.4 Hz, 1H), 5.55 (q, J = 7.5 Hz, 1H), 1.92 – 1.78 (m, 2H), 1.88, (s, 3H), 1.46 – 1.31 (m, 1H), 1.30 – 1.16 (m, 1H), 0.85 (t, J = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.7, 140.5, 137.4, 133.0, 128.0, 127.8, 121.6, 120.9, 119.3, 113.5, 112.0, 49.9, 36.6, 22.6, 18.9, 13.9; **FTIR** (ν_{max}, cm^{-1}): 3654, 3228, 3160, 2988, 2911, 2382, 2342, 1701, 1650, 1638, 1544, 1505, 1457, 1398, 1237, 1075, 1053, 874, 738, 622; **HRMS** (ESI-TOF) m/z: calcd for $C_{17}H_{20}N_3O^+$ $[M + H]^+$, 282.1601; found, 282.1598; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 4.03 min, t_r (minor) = 3.00 min; **$[\alpha]_D^{20}$** = 69.2 (c = 0.3, $CHCl_3$).



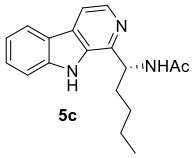


Entry	Retention Time	Height	Area	Area%
1	2.995	297.181	1570.258	49.628
2	4.044	224.072	1593.789	50.372



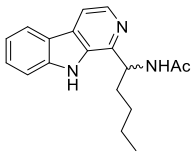


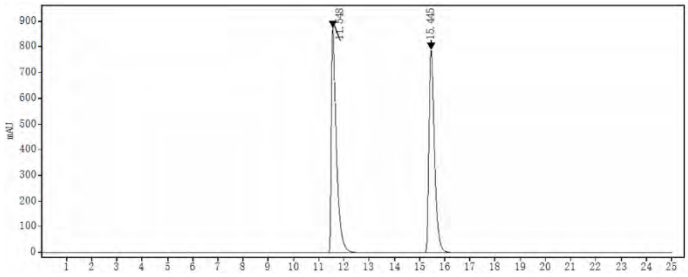
Entry	Retention Time	Height	Area	Area%
1	2.992	106.732	587.763	6.385
2	4.026	1223.335	8617.555	93.615



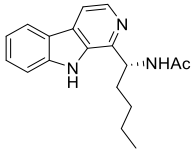
5c

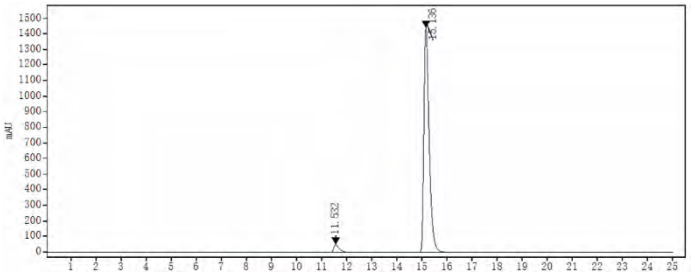
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)pentyl)acetamide (**5c**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 15% acetone in hexanes, 50.2 mg, 85% yield; 93% ee). **Mp**: 183−185 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.59 (s, 1H), 8.39 (d, J = 8.2 Hz, 1H), 8.30 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.8 Hz, 1H), 7.99 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.23 (ddd, J = 8.0, 7.0, 1.0 Hz, 1H), 5.61 – 5.45 (m, 1H), 1.90 – 1.83 (m, 2H), 1.88 (s, 3H), 1.38 – 1.12 (m, 4H), 0.80 (t, J = 7.0 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.7, 140.5, 137.4, 133.0, 128.0, 127.8, 121.6, 120.9, 119.3, 113.5, 112.0, 50.0, 34.1, 27.8, 22.6, 22.1, 13.9; **FTIR** (ν_{max}, cm^{-1}): 3218, 3090, 2930, 2879, 2392, 2323, 1652, 1627, 1554, 1505, 1432, 1370, 1321, 1243, 1071, 821, 738, 626, 577; **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{22}N_3O^+$ $[M+H]^+$, 296.1757; found, 296.1754; **HPLC analysis:** Chiral AD-3 (250 x 4.6 mm, 3 μm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 15.14 min, t_r (minor) = 11.53 min; **$[\alpha]_D^{20}$** = 53.3 (c = 0.3, $CHCl_3$).



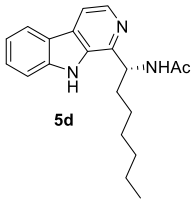


Entry	Retention Time	Height	Area	Area%
1	11.548	875.677	12070.048	50.146
2	15.445	789.868	11999.669	49.854

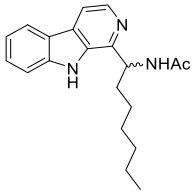


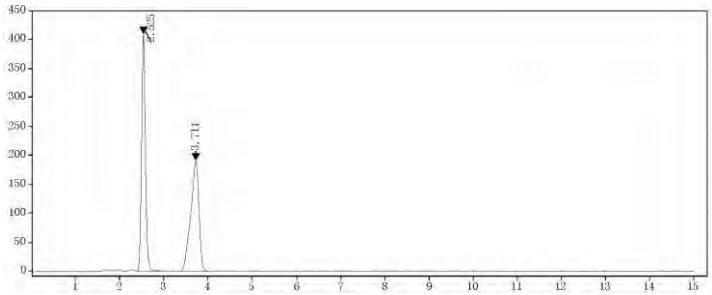


Entry	Retention Time	Height	Area	Area%
1	11.532	49.907	798.127	3.646
2	15.136	1439.368	21091.327	96.354

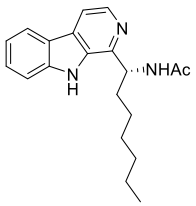


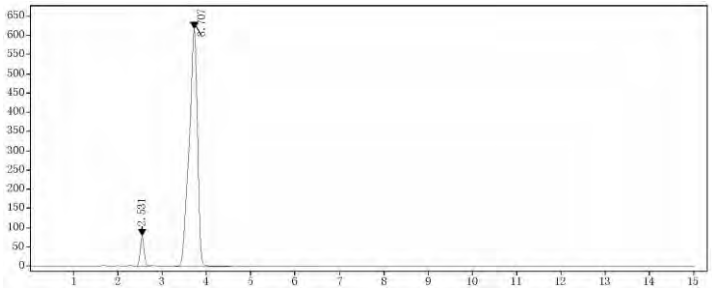
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)heptyl)acetamide (**5d**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 15% acetone in hexanes, 51.7 mg, 80% yield; 89% ee). **Mp**: 136−138 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.59 (s, 1H), 8.39 (d, J = 8.2 Hz, 1H), 8.30 (d, J = 5.2 Hz, 1H), 8.20 (d, J = 7.9 Hz, 1H), 7.99 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.23 (t, J = 7.4 Hz, 1H), 5.53 (q, J = 7.5 Hz, 1H), 1.92 – 1.81 (m, 2H), 1.88 (s, 3H), 1.43 – 1.28 (m, 1H), 1.28 – 1.11 (m, 7H), 0.79 (t, J = 6.7 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 145.7, 140.5, 137.3, 133.0, 128.0, 127.8, 121.6, 120.9, 119.2, 113.4, 112.0, 50.1, 34.4, 31.2, 28.6, 25.5, 22.6, 22.0, 13.9; **FTIR** (ν_{max}, cm^{-1}): 3223, 3012, 2920, 2855, 2384, 2325, 2180, 2141, 1972, 1718, 1650, 1561, 1459, 1377, 1239, 1055, 818, 733, 627,569; **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{26}N_3O^+$ [M + H]$^+$, 324.2070; found, 324.2067; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.71 min, t_r (minor) = 2.53 min; **[α]$_D^{20}$** = 46.4 (c = 0.3, $CHCl_3$).



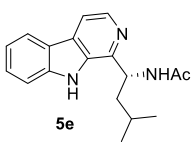


Entry	Retention Time	Height	Area	Area%
1	2.525	409.483	2471.375	49.883
2	3.711	191.070	2482.952	50.117



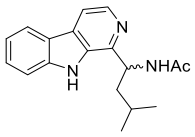


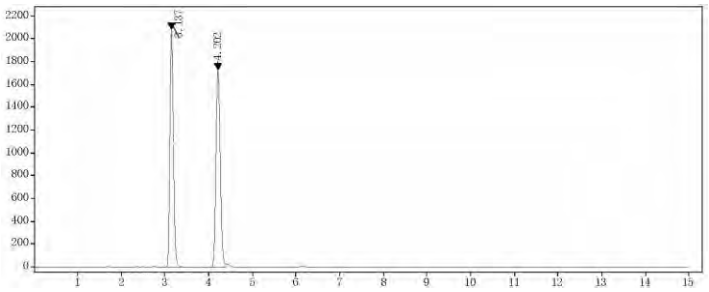
Entry	Retention Time	Height	Area	Area%
1	2.531	77.445	479.984	5.641
2	3.707	616.447	8029.514	94.359



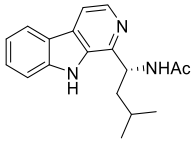
5e

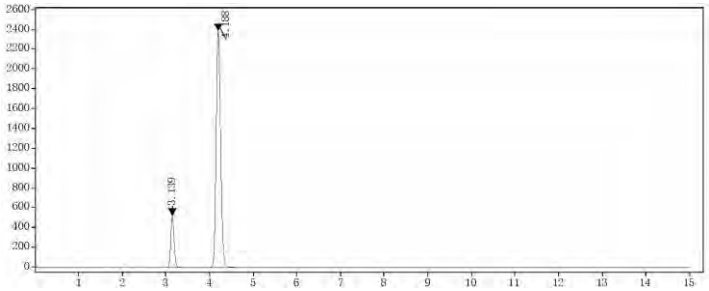
(*R*)-*N*-(3-methyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5e**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 43.7 mg, 74% yield; 72% ee). **Mp:** 199−201 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.49 (s, 1H), 8.41 (d, J = 8.3 Hz, 1H), 8.30 (d, J = 5.2 Hz, 1H), 8.20 (d, J = 7.9 Hz, 1H), 7.99 (d, J = 5.2 Hz, 1H), 7.64 (d, J = 8.2 Hz, 1H), 7.54 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.29 – 7.16 (m, 1H), 5.58 (td, J = 8.5, 6.2 Hz, 1H), 1.90 – 1.83 (m, 1H), 1.86 (s, 3H), 1.81 – 1.68 (m, 1H), 1.58 (hept, J = 6.6 Hz, 1H), 0.90 (dd, J = 6.6, 2.8 Hz, 6H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 146.0, 140.5, 137.5, 133.0, 128.1, 128.0, 121.6, 120.9, 119.3, 113.5, 112.1, 48.5, 42.8, 24.6, 23.0, 22.6, 22.3; **FTIR** (ν_{max}, cm^{-1}): 3218, 3175, 2981, 2923, 2382, 1648, 1560, 1542, 1504, 1428, 1375, 1320, 1242, 1128, 1055, 823, 742, 705, 654, 601, 566; **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{22}N_3O^+$ $[M + H]^+$, 296.1757; found, 296.1754; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 4.19 min, t_r (minor) = 3.14 min; **[α]$_D^{20}$** = 51.1 (c = 0.3, $CHCl_3$).



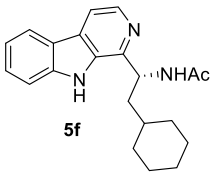


Entry	Retention Time	Height	Area	Area%
1	3.137	2079.841	11701.923	49.800
2	4.202	1723.98	11795.814	50.200



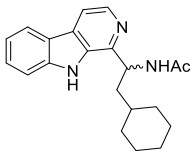


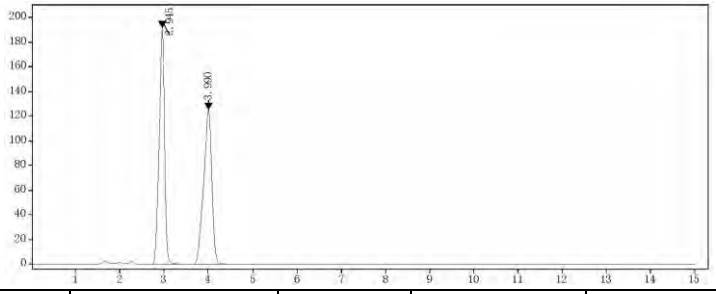
Entry	Retention Time	Height	Area	Area%
1	3.139	520.937	2726.653	13.757
2	4.188	2387.235	17093.874	86.243



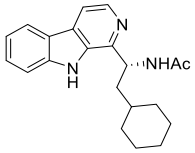
5f

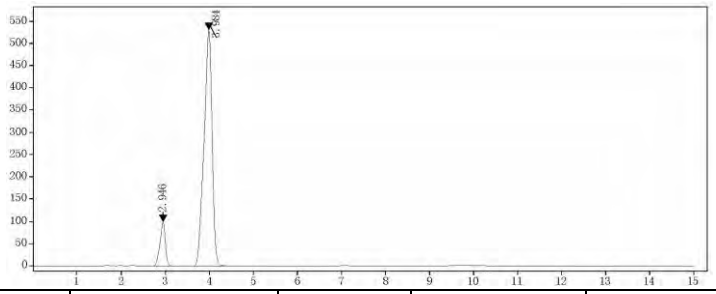
(*R*)-*N*-(2-cyclohexyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5f**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 15% acetone in hexanes, 56.3 mg, 84% yield; 78% ee). **Mp**: 218−220 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.45 (s, 1H), 8.40 (d, *J* = 8.3 Hz, 1H), 8.29 (d, *J* = 5.2 Hz, 1H), 8.20 (d, *J* = 7.9 Hz, 1H), 7.99 (d, *J* = 5.2 Hz, 1H), 7.63 (d, *J* = 8.2 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.23 (t, *J* = 7.4 Hz, 1H), 5.60 (td, *J* = 8.7, 6.1 Hz, 1H), 1.87 – 1.77 (m, 2H), 1.85 (s, 3H), 1.73 (dd, *J* = 13.4, 6.1 Hz, 2H), 1.67 – 1.51 (m, 3H), 1.31 (d, *J* = 12.6 Hz, 1H), 1.10 (d, *J* = 7.7 Hz, 3H), 0.93 (q, *J* = 10.6 Hz, 2H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 146.1, 140.5, 137.4, 132.9, 128.0, 128.0, 121.6, 120.9, 119.3, 113.5, 112.1, 47.8, 41.4, 33.9, 33.2, 32.4, 26.1, 25.8, 25.7, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3205, 2971, 2923, 2364, 2342, 1783, 1750, 1647, 1541, 1500, 1487, 1420, 1248, 1056, 880, 735, 670, 569; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{26}N_3O^+$ $[M + H]^+$, 336.2070; found, 336.2065; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.98 min, t_r (minor) = 2.95 min; **$[\alpha]_D^{20}$** = 20.8 (c = 0.3, $CHCl_3$).



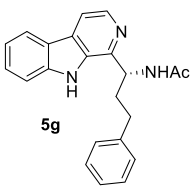


Entry	Retention Time	Height	Area	Area%
1	2.945	191.716	1613.873	50.081
2	3.990	125.088	1608.657	49.919

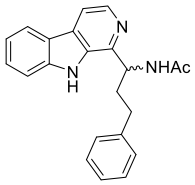


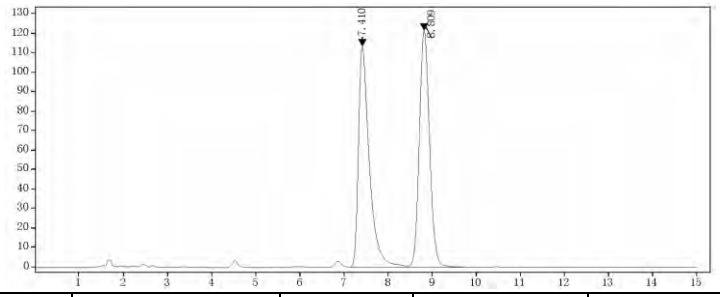


Entry	Retention Time	Height	Area	Area%
1	2.946	98.728	842.512	11.090
2	3.984	529.756	6754.530	88.910

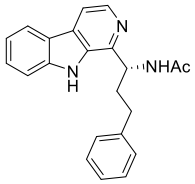


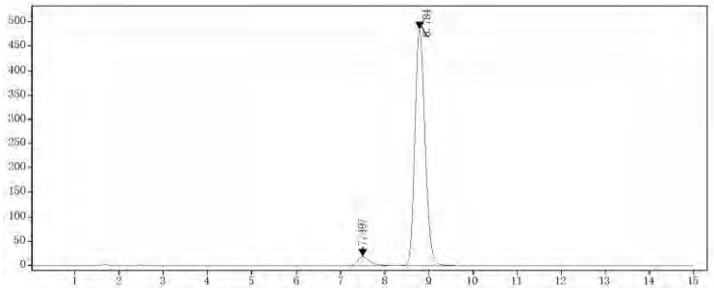
(*R*)-*N*-(3-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5g**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 15% acetone in hexanes, 48.8 mg, 71% yield; 91% ee). **Mp**: 184−186 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.60 (s, 1H), 8.54 (d, J = 8.3 Hz, 1H), 8.32 (d, J = 5.2 Hz, 1H), 8.22 (d, J = 7.8 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.55 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.29 – 7.18 (m, 3H), 7.18 – 7.08 (m, 3H), 5.62 (td, J = 8.0, 6.0 Hz, 1H), 2.71 (ddd, J = 13.6, 10.8, 5.6 Hz, 1H), 2.56 (ddd, J = 13.7, 10.9, 5.7 Hz, 1H), 2.26 – 2.09 (m, 2H), 1.92 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.2, 145.2, 141.7, 140.5, 137.4, 133.0, 128.3, 128.1, 128.0, 125.7, 121.7, 120.9, 119.3, 113.6, 112.0, 50.1, 36.1, 31.9, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3216, 3165, 2965, 2905, 2887, 2380, 2325, 2308, 2138, 1922, 1845, 1653, 1558, 15441, 1507, 1473, 1458, 1372, 1323, 1235, 1074, 737, 624; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{22}N_3O^+$ [M + H]$^+$, 344.1757; found, 344.1753; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 8.78 min, t_r (minor) = 7.50 min; **[α]$_D^{20}$** = 58.0 (c = 0.3, $CHCl_3$).



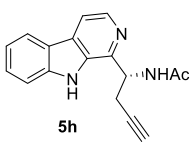


Entry	Retention Time	Height	Area	Area%
1	7.410	113.256	1950.212	49.970
2	8.809	121.369	1952.572	50.030



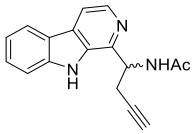


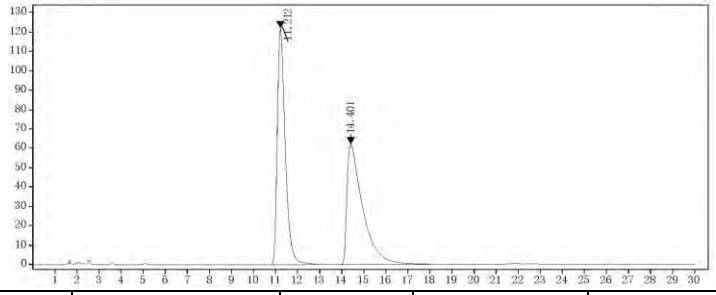
Entry	Retention Time	Height	Area	Area%
1	7.497	18.642	362.428	4.478
2	8.784	485.549	7731.853	95.522



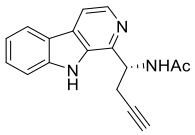
5h

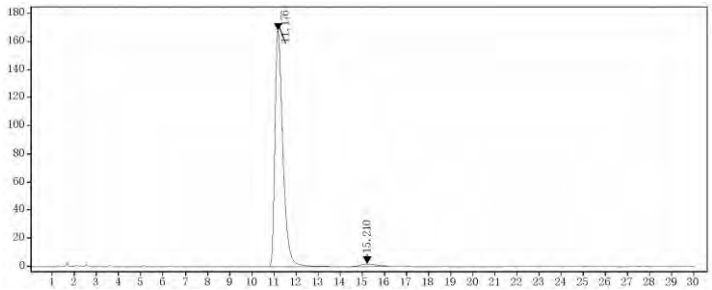
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-yn-1-yl)acetamide (**5h**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 45.5 mg, 82% yield; 96% ee). **Mp**: 151−153 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.64 (s, 1H), 8.53 (d, J = 7.9 Hz, 1H), 8.34 (d, J = 5.2 Hz, 1H), 8.22 (d, J = 7.9 Hz, 1H), 8.05 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.59 – 7.45 (m, 1H), 7.24 (t, J = 7.4 Hz, 1H), 5.70 (q, J = 7.3 Hz, 1H), 2.93 – 2.77 (m, 2H), 2.67 (d, J = 2.6 Hz, 1H), 1.89 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.3, 143.3, 140.6, 137.4, 133.3, 128.2, 128.1, 121.7, 120.8, 119.4, 114.0, 112.1, 81.4, 72.5, 49.2, 23.5, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3332, 3305, 3225, 2974, 2908, 2384, 1750, 1699, 1648, 1508, 1241, 1070, 840, 731, 685, 657, 600, 554; **HRMS** (ESI-TOF) m/z: calcd for $C_{17}H_{16}N_3O^+$ [M + H]$^+$, 278.1288; found, 278.1285; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 90:10, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 11.18 min, t_r (minor) = 15.21 min; **[α]$_D^{20}$** = 58.3 (c = 0.3, $CHCl_3$).



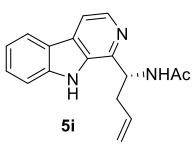


Entry	Retention Time	Height	Area	Area%
1	11.212	121.989	3059.897	49.968
2	14.401	61.990	3063.777	50.030



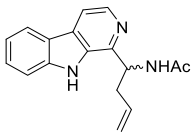


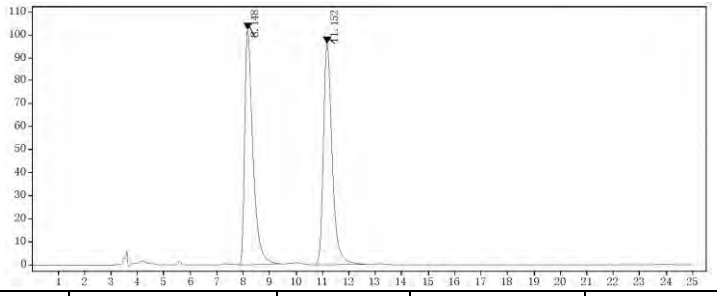
Entry	Retention Time	Height	Area	Area%
1	11.176	167.925	4204.174	97.766
2	15.210	1.820	96.087	2.234



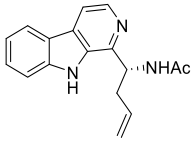
5i

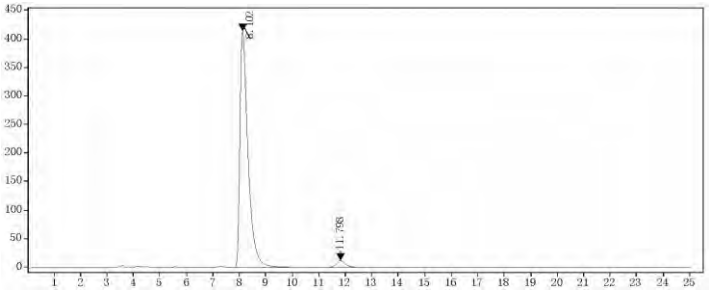
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-en-1-yl)acetamide (**5i**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 40.2 mg, 72% yield; 94% ee). **Mp**: 157−159 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.61 (s, 1H), 8.43 (d, J = 8.3 Hz, 1H), 8.31 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.01 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.24 (t, J = 7.5 Hz, 1H), 5.78 (ddt, J = 17.1, 10.9, 6.9 Hz, 1H), 5.63 (q, J = 7.5 Hz, 1H), 5.01 (dd, J = 17.2, 2.1 Hz, 1H), 4.93 (dd, J = 10.3, 2.2 Hz, 1H), 2.65 (dp, J = 14.0, 7.3 Hz, 2H), 1.88 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 144.8, 140.5, 137.3, 134.8, 133.0, 128.1, 127.9, 121.6, 120.9, 119.3, 117.3, 113.6, 112.0, 49.6, 38.5, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3223, 2964, 2848, 2357, 2163, 1994, 1650, 1544, 1459, 1237, 1120, 808, 741, 582; **HRMS** (ESI-TOF) m/z: calcd for $C_{17}H_{18}N_3O^+$ $[M + H]^+$, 280.1444; found, 280.1441; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 8.10 min, t_r (minor) = 11.80 min; **$[\alpha]_D^{20}$** = 37.1 (c = 0.3, $CHCl_3$).



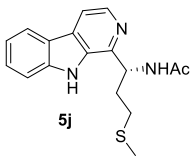


Entry	Retention Time	Height	Area	Area%
1	8.148	101.883	2216.014	49.853
2	11.152	95.825	2229.081	50.147



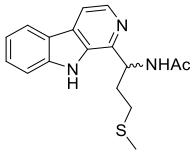


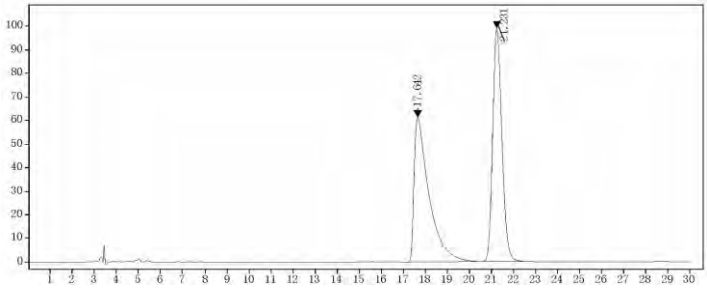
Entry	Retention Time	Height	Area	Area%
1	8.102	413.72	8839.74	96.786
2	11.798	11.746	293.503	3.214



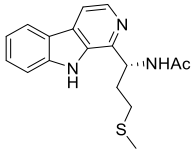
5j

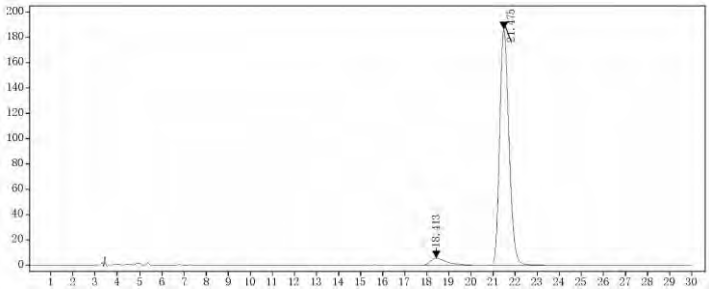
(*R*)-*N*-(3-(methylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5j**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 40.7 mg, 68%yield; 90% ee). **Mp**: 198−200 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.57 (s, 1H), 8.49 (d, J = 8.2 Hz, 1H), 8.31 (d, J = 5.2 Hz, 1H), 8.22 (d, J = 7.9 Hz, 1H), 8.03 (d, J = 5.2 Hz, 1H), 7.63 (d, J = 8.1 Hz, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.24 (t, J = 7.4 Hz, 1H), 5.62 (q, J = 7.4 Hz, 1H), 2.54-2.41 (m, 2H), 2.16 (tp, J = 16.4, 5.7 Hz, 2H), 2.00 (s, 3H), 1.89 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.2, 144.7, 140.5, 137.3, 133.0, 128.2, 128.0, 121.7, 120.9, 119.4, 113.7, 112.1, 49.5, 34.0, 29.9, 22.6, 14.7; **FTIR** (ν_{max}, cm^{-1}): 3223, 3181, 3100, 3025, 2950, 2911, 2854, 2360, 2165, 2010, 1771, 1717, 1650, 1541,1425, 1238, 821, 730, 624, 556; **HRMS** (ESI-TOF) m/z: calcd for $C_{17}H_{20}N_3OS^+$ [M + H]$^+$, 314.1322; found, 314.1317; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 21.48 min, t_r (minor) = 18.41 min; **[α]$_D^{20}$** = 25.2 (c = 0.3, $CHCl_3$).



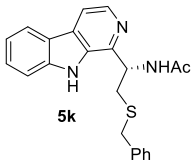


Entry	Retention Time	Height	Area	Area%
1	17.642	61.267	2878.65	49.962
2	21.231	98.961	2882.992	50.038



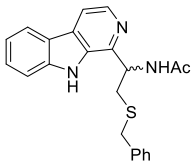


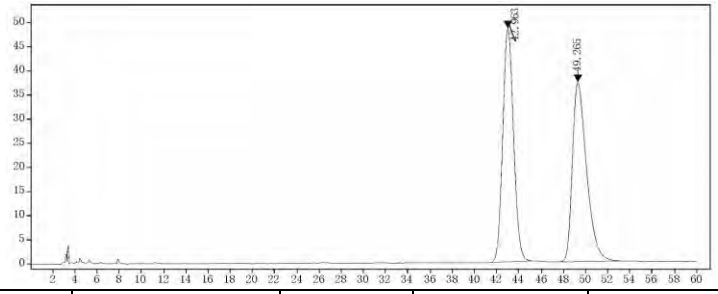
Entry	Retention Time	Height	Area	Area%
1	18.413	5.640	295.755	5.107
2	21.475	186.544	5494.876	94.893



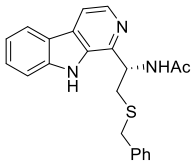
5k

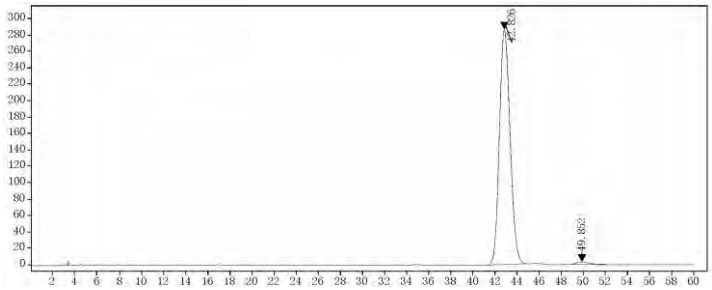
(*R*)-*N*-(2-(benzylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5k**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 51.8 mg, 69% yield; 98% ee). **Mp:** 152−154 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.70 (s, 1H), 8.50 (d, J = 8.1 Hz, 1H), 8.34 (d, J = 5.2 Hz, 1H), 8.23 (d, J = 7.9 Hz, 1H), 8.06 (d, J = 5.2 Hz, 1H), 7.65 (d, J = 8.2 Hz, 1H), 7.56 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.30 – 7.12 (m, 6H), 5.84 – 5.72 (m, 1H), 3.65 (q, J = 13.0 Hz, 2H), 3.07 – 2.90 (m, 2H), 1.89 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.2, 143.8, 140.6, 138.5, 137.4, 133.7, 128.9, 128.3, 128.2, 128.1, 126.7, 121.7, 120.8, 119.4, 114.0, 112.1, 49.7, 38.9, 35.2, 34.7, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3212, 2970, 2897, 2365, 2340, 1651, 1542, 1457, 1385, 1230, 1087, 828, 743, 703, 619, 570; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{22}N_3OS^+$ $[M + H]^+$, 376.1478; found, 376.1473; **HPLC analysis:** Chiral NX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 42.83 min, t_r (minor) = 49.85 min; **$[\alpha]_D^{20}$** = 29.0 (c = 0.3, $CHCl_3$).



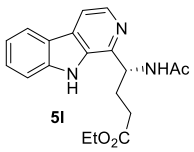


Entry	Retention Time	Height	Area	Area%
1	42.963	48.481	3282.055	49.827
2	49.265	37.252	3304.780	50.173

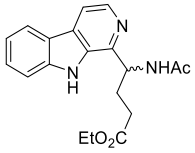


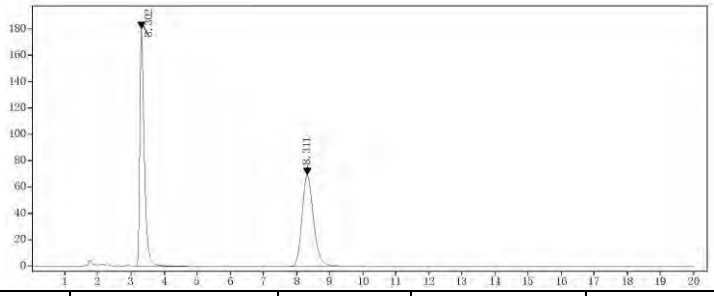


Entry	Retention Time	Height	Area	Area%
1	42.826	286.037	19164.32	98.755
2	49.852	2.95	241.625	1.245

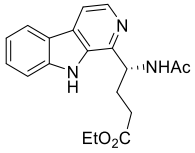


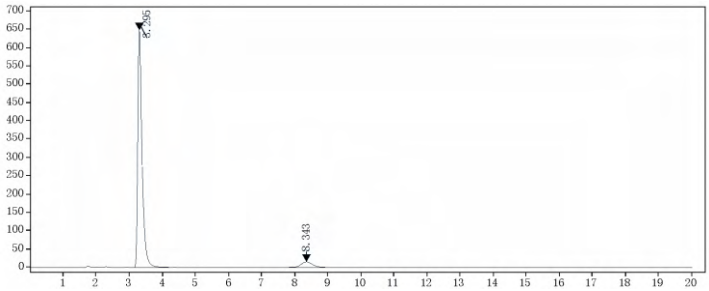
(*R*)-Ethyl 4-acetamido-4-(9*H*-pyrido[3,4-*b*]indol-1-yl)butanoate (**5l**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 30% acetone in hexanes, 50.2 mg, 74% yield; 88% ee). **Mp**: 142−144 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.59 (s, 1H), 8.47 (d, J = 8.2 Hz, 1H), 8.31 (d, J = 5.2 Hz, 1H), 8.22 (d, J = 7.9 Hz, 1H), 8.03 (d, J = 5.2 Hz, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.59 – 7.48 (m, 1H), 7.24 (t, J = 7.5 Hz, 1H), 5.58 (q, J = 7.3 Hz, 1H), 3.95 (qq, J = 6.8, 3.7 Hz, 2H), 2.44 – 2.23 (m, 2H), 2.23 – 2.05 (m, 2H), 1.90 (s, 3H), 1.09 (t, J = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 172.4, 169.2, 144.5, 140.5, 137.4, 133.0, 128.2, 128.0, 121.7, 120.9, 119.4, 113.8, 112.0, 59.8, 49.3, 30.4, 29.4, 22.6, 14.0; **FTIR** (ν_{max}, cm^{-1}): 3351, 3199, 2983, 2905, 2359, 2337, 2161, 1723, 1648, 1541, 1500, 1450, 1398, 1244, 1055, 741, 670, 593; **HRMS** (ESI-TOF) m/z: calcd for $C_{19}H_{22}N_3O_3^+$ $[M + H]^+$, 340.1656; found, 340.1651; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 80:20, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.30 min, t_r (minor) = 8.34 min; **$[\alpha]_D^{20}$** = -43.0 (c = 0.3, $CHCl_3$).



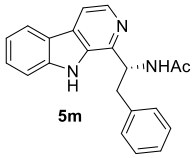


Entry	Retention Time	Height	Area	Area%
1	3.302	179.628	1681.764	50.251
2	8.311	68.876	1664.970	49.749



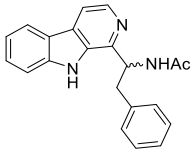


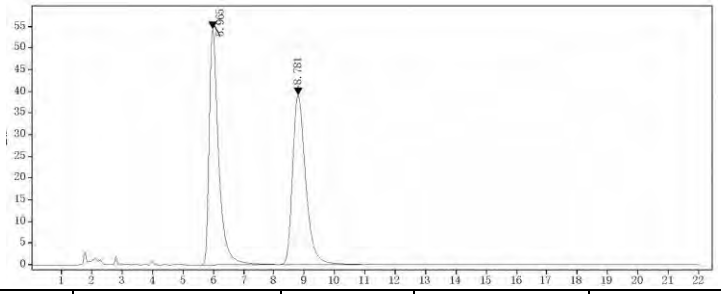
Entry	Retention Time	Height	Area	Area%
1	3.295	648.15	5832.035	93.778
2	8.343	15.38	386.955	6.222



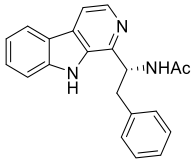
5m

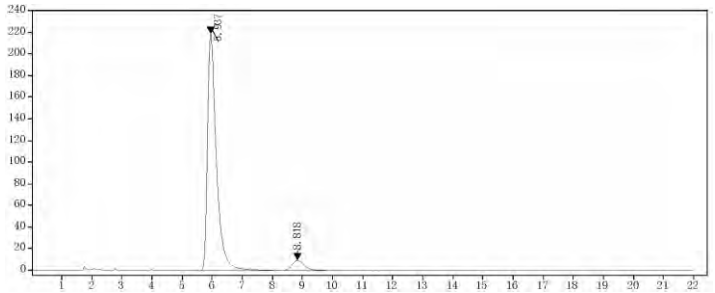
(*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5m**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 52.7 mg, 80% yield; 89% ee). **Mp**: 158−160 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.58 (s, 1H), 8.54 (d, J = 8.4 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.20 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.61 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.37 – 7.15 (m, 5H), 7.11 (t, J = 7.2 Hz, 1H), 5.81 (q, J = 7.8 Hz, 1H), 3.16 (d, J = 13.5 Hz, 2H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.0, 140.5, 138.4, 137.3, 133.0, 129.4, 128.1, 128.0, 127.8, 126.0, 121.6, 120.8, 119.3, 113.7, 112.0, 51.2, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3215, 3178, 3025, 2991, 2825, 2361, 2341, 1649, 15556, 15041, 1429, 1375, 1320, 1241, 1065, 741, 705, 656, 602, 566; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{20}N_3O^+$ $[M + H]^+$, 330.1601; found, 330.1595; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 5.94 min, t_r (minor) = 8.82 min; **$[\alpha]_D^{20}$** = 63.2 (c = 0.3, $CHCl_3$).



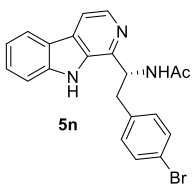


Entry	Retention Time	Height	Area	Area%
1	5.965	54.398	1169.110	49.940
2	8.781	39.063	1171.900	50.060



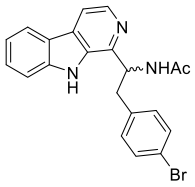


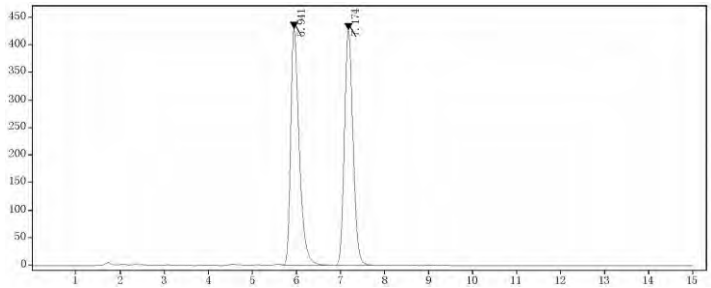
Entry	Retention Time	Height	Area	Area%
1	5.937	218.604	4476.203	94.570
2	8.818	256.992	1096.370	5.430



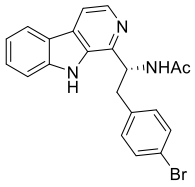
5n

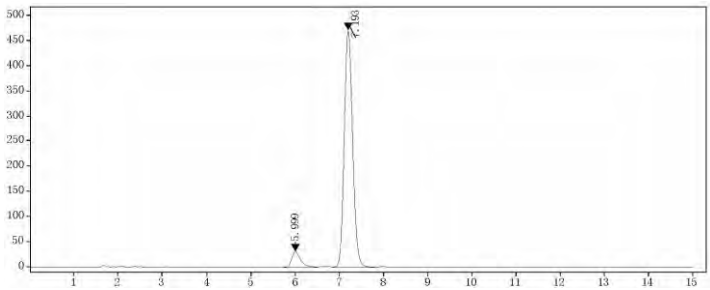
(*R*)-*N*-(2-(4-bromophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5n**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 54.7 mg, 67% yield; 87% ee). **Mp**: 190−192 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.63 (s, 1H), 8.56 (d, J = 8.4 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.03 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.55 (ddd, J = 8.2, 6.9, 1.2 Hz, 1H), 7.45 – 7.34 (m, 2H), 7.24 (dq, J = 7.8, 1.7 Hz, 3H), 5.80 (td, J = 8.7, 5.6 Hz, 1H), 3.20 (dd, J = 13.7, 5.7 Hz, 1H), 3.12 (dd, J = 13.7, 8.9 Hz, 1H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 144.7, 140.5, 137.9, 137.3, 132.9, 131.7, 130.7, 128.1, 128.0, 121.7, 120.8, 119.3, 119.3, 113.8, 112.0, 50.9, 39.0, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3172, 2978, 2938, 2359, 2338, 1793, 1748, 1717, 1688, 1648, 1557, 1542, 1507, 1456, 1431, 1374, 1318, 1238, 1071, 1012, 880, 735, 647, 562; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{19}BrN_3O^+$ [M + H]$^+$, 408.0706; found, 408.0702; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 7.19 min, t_r (minor) = 6.00 min; **[α]$_D^{20}$** = 80.3 (c = 0.3, $CHCl_3$).



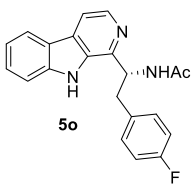


Entry	Retention Time	Height	Area	Area%
1	5.941	428.610	5785.245	50.016
2	7.174	426.428	5781.487	49.984



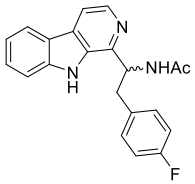


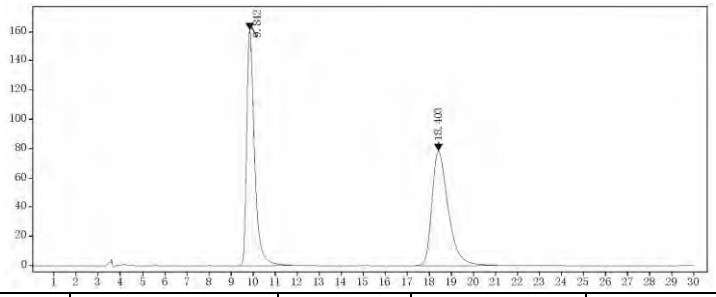
Entry	Retention Time	Height	Area	Area%
1	5.999	31.007	445.513	6.680
2	7.193	470.564	6224.020	93.320



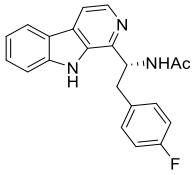
5o

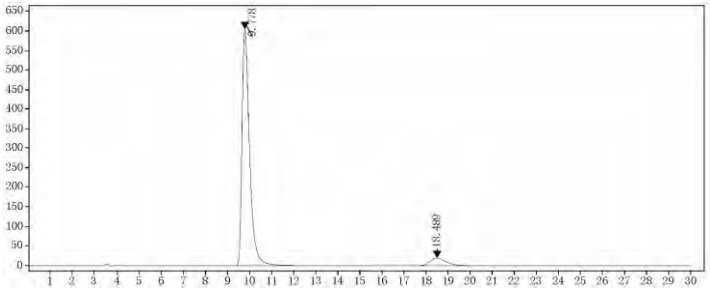
(*R*)-*N*-(2-(4-fluorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5o**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 61.8 mg, 89% yield; 87% ee). **Mp**: 184−186 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.69 (s, 1H), 8.62 (d, J = 8.4 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.62 (dt, J = 8.2, 1.0 Hz, 1H), 7.54 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.36 – 7.27 (m, 2H), 7.23 (ddd, J = 7.9, 7.0, 1.1 Hz, 1H), 7.08 – 6.97 (m, 2H), 5.77 (td, J = 8.5, 6.0 Hz, 1H), 3.26 – 3.08 (m, 2H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 144.9, 140.5, 137.3, 134.6, 132.9, 131.3, 131.2, 128.1, 128.0, 121.6, 120.8, 119.3, 114.6, 114.4, 113.7, 112.0, 51.5, 38.7, 22.5; **^{19}F NMR** (376 MHz, DMSO-d_6) δ -117.14 (septet); **FTIR** (ν_{max}, cm^{-1}): 3218, 2974, 2911, 2366, 2335, 1754, 1701, 1650, 1556, 1505, 1457, 1396, 1237, 1072, 818, 738, 670, 533; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{19}FN_3O^+$ [M + H]$^+$, 348.1507; found, 348.1503; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 9.78 min, t_r (minor) = 18.49 min; **[α]$_D^{20}$** = 67.4 (c = 0.3, $CHCl_3$).



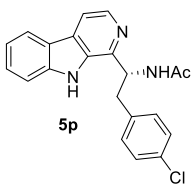


Entry	Retention Time	Height	Area	Area%
1	9.842	160.977	3979.740	49.753
2	18.403	78.109	4019.250	50.247



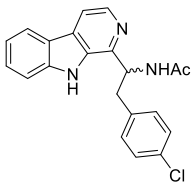


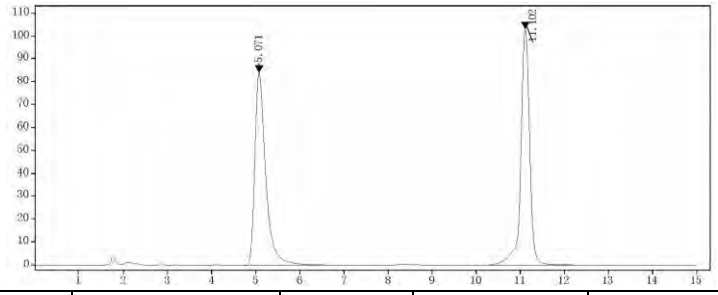
Entry	Retention Time	Height	Area	Area%
1	9.778	605.152	14325.842	93.309
2	18.489	19.349	1027.214	6.691



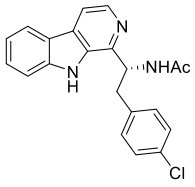
5p

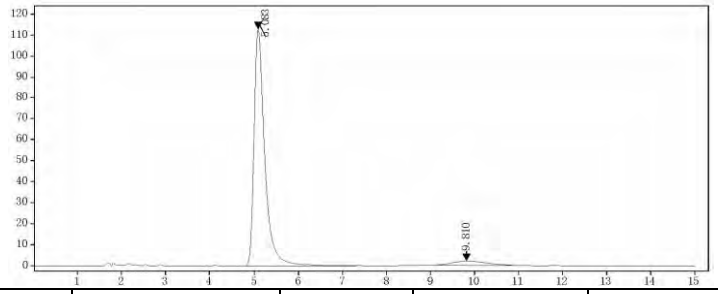
(*R*)-*N*-(2-(4-chlorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5p**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 52.4 mg, 72% yield; 89% ee). **Mp**: 191−193 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.65 (s, 1H), 8.59 (d, J = 8.4 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.54 (ddd, J = 8.2, 6.9, 1.2 Hz, 1H), 7.35 – 7.19 (m, 5H), 5.82 (td, J = 8.7, 5.6 Hz, 1H), 3.29 – 3.08 (m, 2H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 144.7, 140.5, 137.5, 137.3, 133.0, 131.3, 130.8, 128.2, 128.0, 127.8, 121.7, 120.8, 119.3, 113.8, 112.0, 51.0, 39.0, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3218, 3172, 2988, 2923, 2357, 1655, 1563, 1499, 1319, 1241, 1094, 1036, 1019, 738, 576; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{19}ClN_3O^+$ [M + H]$^+$, 364.1211; found, 364.1207; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 5.08 min, t_r (minor) = 9.81 min; **$[\alpha]_D^{20}$** = 82.3 (c = 0.3, $CHCl_3$).



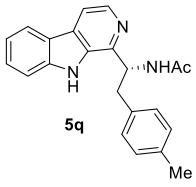


Entry	Retention Time	Height	Area	Area%
1	5.071	83.961	1446.461	50.002
2	11.102	102.979	1446.362	49.998

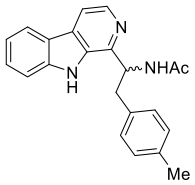


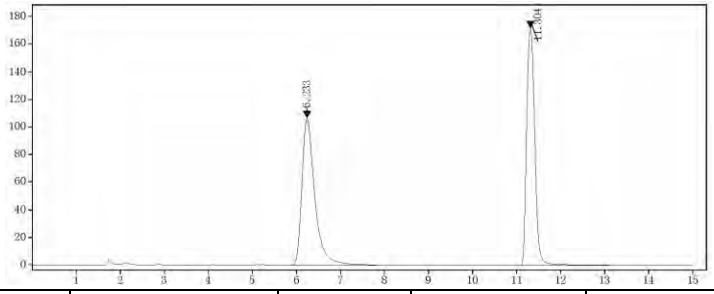


Entry	Retention Time	Height	Area	Area%
1	5.083	112.808	1909.267	94.619
2	9.810	1.944	108.574	5.381

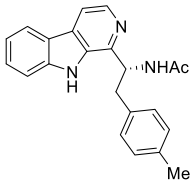


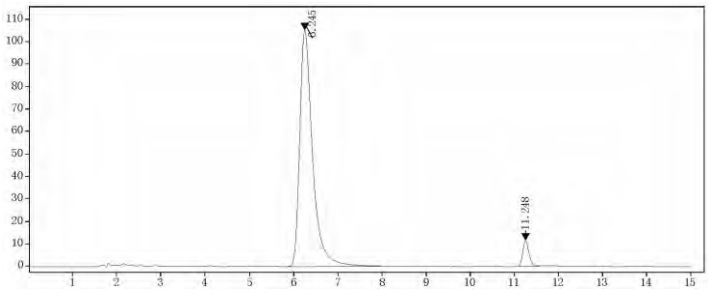
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)-2-(p-tolyl)ethyl)acetamide (**5q**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 50.1 mg, 73% yield; 90% ee). **Mp**: 166−168 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.58 (s, 1H), 8.52 (d, J = 8.3 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.20 (d, J = 7.8 Hz, 1H), 8.01 (d, J = 5.2 Hz, 1H), 7.62 (dt, J = 8.3, 1.0 Hz, 1H), 7.54 (ddd, J = 8.3, 7.0, 1.2 Hz, 1H), 7.23 (ddd, J = 8.0, 7.0, 1.0 Hz, 1H), 7.15 (d, J = 7.9 Hz, 2H), 7.00 (d, J = 7.8 Hz, 2H), 5.78 (td, J = 8.5, 5.9 Hz, 1H), 3.25 – 3.08 (m, 2H), 2.20 (s, 3H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.1, 140.5, 137.3, 135.3, 134.9, 133.1, 129.3, 128.5, 128.1, 128.0, 121.6, 120.8, 119.3, 113.7, 112.0, 51.3, 39.1, 22.5, 20.6; **FTIR** (ν_{max}, cm^{-1}): 3218, 3179, 2981, 2904, 2381, 2340, 1859, 1771, 1749, 1698, 1653, 1647, 1576, 1541, 1521, 1507, 1457, 1398, 1320, 1235, 883, 748, 705, 670, 518; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{22}N_3O^+$ [M + H]$^+$, 344.1757; found, 344.1753; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 6.25 min, t_r (minor) = 11.25 min; **[α]$_D^{20}$** = 67.2 (c = 0.3, $CHCl_3$).



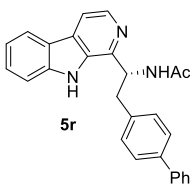


Entry	Retention Time	Height	Area	Area%
1	6.233	106.469	2174.898	49.702
2	11.304	171.717	2200.952	50.298



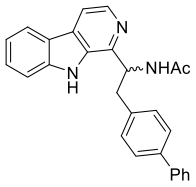


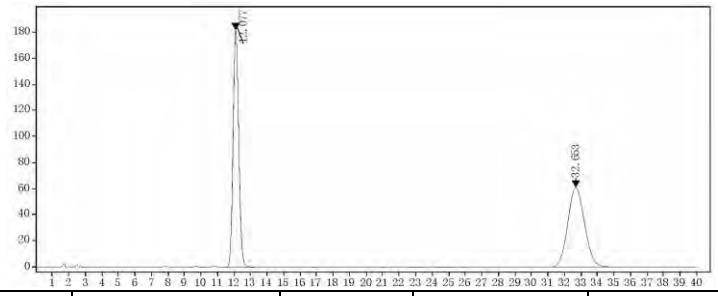
Entry	Retention Time	Height	Area	Area%
1	6.245	105.064	2130.711	95.027
2	11.248	11.342	111.506	4.973



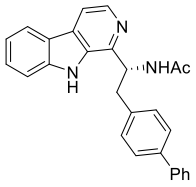
5r

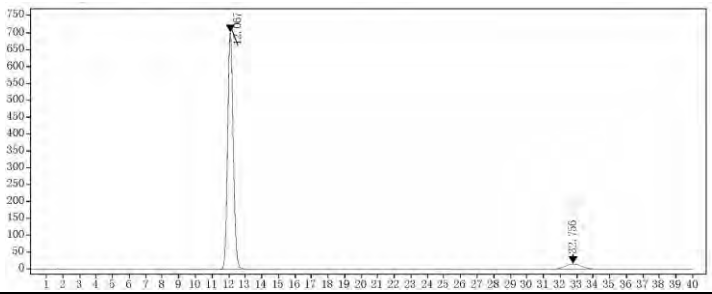
(*R*)-*N*-(2-([1,1'-bipheyl]-4-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5r**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 58.4 mg, 72% yield; 88% ee). **Mp**: 180−182 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.64 (s, 1H), 8.61 (d, J = 8.4 Hz, 1H), 8.35 (d, J = 5.2 Hz, 1H), 8.22 (d, J = 7.9 Hz, 1H), 8.04 (d, J = 5.2 Hz, 1H), 7.67 – 7.58 (m, 3H), 7.58 – 7.49 (m, 3H), 7.42 (dd, J = 8.4, 6.9 Hz, 2H), 7.40 – 7.35 (m, 2H), 7.35 – 7.28 (m, 1H), 7.24 (ddd, J = 8.0, 7.0, 1.1 Hz, 1H), 5.86 (td, J = 8.6, 5.7 Hz, 1H), 3.25 (qd, J = 13.8, 7.3 Hz, 2H), 1.82 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 145.0, 140.5, 140.0, 137.8, 137.8, 137.3, 133.0, 130.0, 128.8, 128.1, 128.0, 127.2, 126.5, 126.1, 121.6, 120.8, 119.3, 113.8, 112.0, 51.2, 39.2, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3218, 3179, 2975, 2925, 2364, 2340, 1772, 1748, 1648, 1455, 1419, 1398, 1375, 1318, 1240, 1055, 744, 690, 669, 570; **HRMS** (ESI-TOF) m/z: calcd for $C_{27}H_{24}N_3O^+$ [M + H]$^+$, 406.1914; found, 406.1909; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 12.07 min, t_r (minor) = 32.76 min; **[α]$_D^{20}$** = 61.9r (c = 0.3, $CHCl_3$).



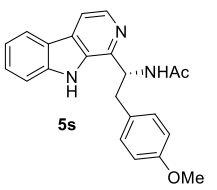


Entry	Retention Time	Height	Area	Area%
1	12.077	181.569	4310.033	50.052
2	32.653	60.766	4300.094	49.948



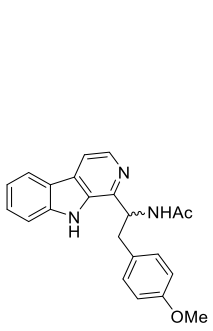


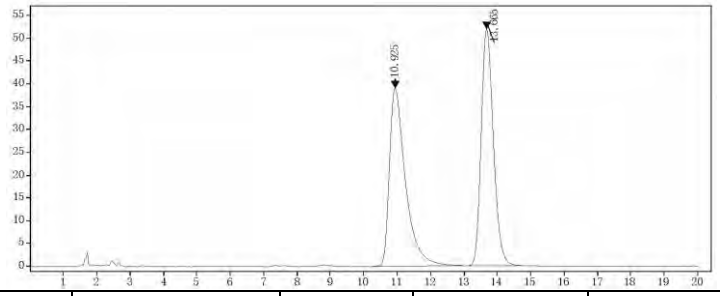
Entry	Retention Time	Height	Area	Area%
1	12.067	701.21	16468.797	93.891
2	32.756	15.547	1071.504	6.109



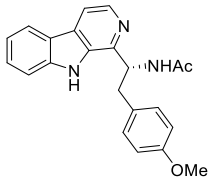
5s

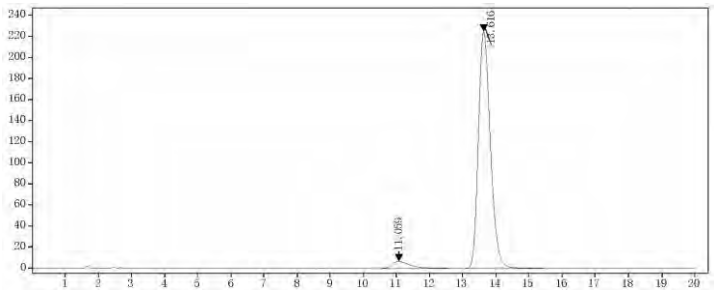
(*R*)-*N*-(2-(4-methoxyphenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5s**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 63.3 mg, 88% yield; 91% ee). **Mp**: 188−190 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.59 (s, 1H), 8.52 (d, *J* = 8.4 Hz, 1H), 8.33 (d, *J* = 5.2 Hz, 1H), 8.21 (d, *J* = 7.8 Hz, 1H), 8.01 (d, *J* = 5.2 Hz, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.54 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 7.23 (ddd, *J* = 8.0, 6.9, 1.1 Hz, 1H), 7.20 – 7.13 (m, 2H), 6.81 – 6.70 (m, 2H), 5.81 – 5.70 (m, 1H), 3.67 (s, 3H), 3.25 – 3.02 (m, 2H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 157.6, 145.1, 140.5, 137.3, 133.0, 130.4, 130.3, 128.1, 127.9, 121.6, 120.8, 119.3, 113.7, 113.3, 112.0, 54.9, 51.5, 38.7, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3216, 3175, 2985, 2920, 2901, 2359, 2341, 1748, 1701, 1650, 1637, 1558, 1505, 1462, 1432, 1378, 1319, 1183, 1083, 880, 814, 775, 738, 668, 629, 557; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{22}N_3O_2^+$ [M + H]$^+$, 360.1707; found, 360.1701; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 13.62 min, t_r (minor) = 11.06 min; **[α]$_D^{20}$** = 69.6 (c = 0.3, $CHCl_3$).



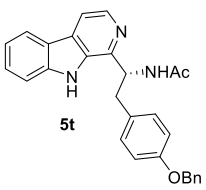


Entry	Retention Time	Height	Area	Area%
1	10.925	38.976	1311.686	49.761
2	13.665	51.831	1324.299	50.239



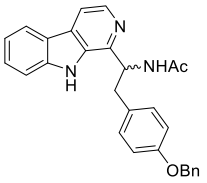


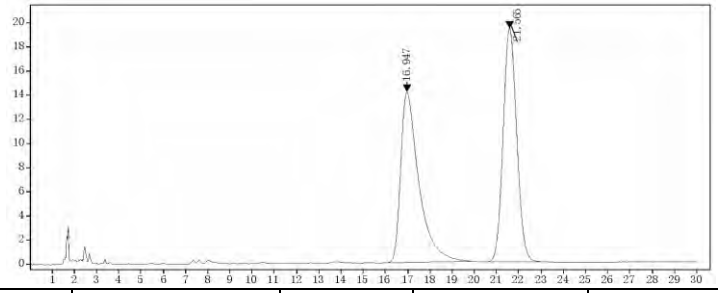
Entry	Retention Time	Height	Area	Area%
1	11.059	6.717	259.156	4.393
2	13.616	224.838	5640.629	95.607



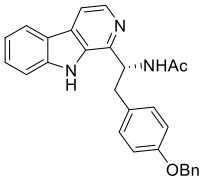
5t

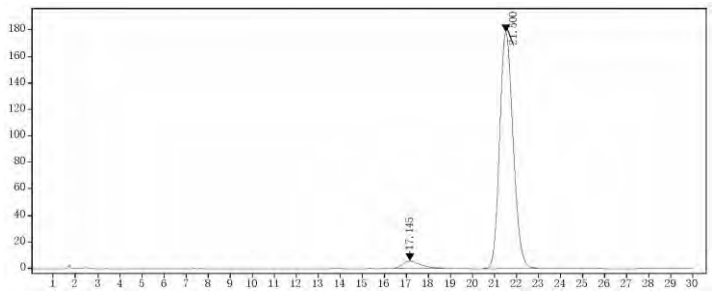
(*R*)-*N*-(2-(4-(benzyloxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5t**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 15% acetone in hexanes, 66.2 mg, 76% yield; 92% ee). **Mp**: 176−178 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.60 (s, 1H), 8.53 (d, J = 8.3 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.59 – 7.48 (m, 1H), 7.43 – 7.33 (m, 4H), 7.33 – 7.27 (m, 1H), 7.24 (t, J = 7.2 Hz, 1H), 7.19 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 5.76 (td, J = 8.5, 5.8 Hz, 1H), 5.00 (s, 2H), 3.13 (qd, J = 13.8, 7.3 Hz, 2H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 156.7, 145.1, 140.5, 137.3, 137.2, 133.0, 130.6, 130.4, 128.4, 128.1, 127.9, 127.7, 127.7, 121.6, 120.8, 119.3, 114.2, 113.7, 112.0, 69.1, 51.4, 38.8, 22.6; **FTIR** (ν_{max}, cm^{-1}): 3217, 3180, 2987, 2392, 2340, 1739, 1716, 1659, 1648, 1542, 1508, 1487, 1378, 1241, 1058, 1044, 737, 669, 571; **HRMS** (ESI-TOF) m/z: calcd for $C_{28}H_{26}N_3O_2^+$ $[M + H]^+$, 436.2020; found, 436.2014; **HPLC analysis:** Chiral ND(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 ºC, 254 nm), t_r (major) = 21.50 min, t_r (minor) = 17.15 min; **$[\alpha]_D^{20}$** = 44.3 (c = 0.3, $CHCl_3$).



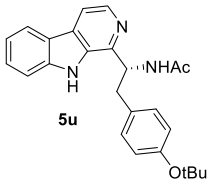


Entry	Retention Time	Height	Area	Area%
1	16.947	14.125	800.937	49.392
2	21.565	19.335	820.645	50.608

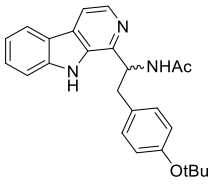


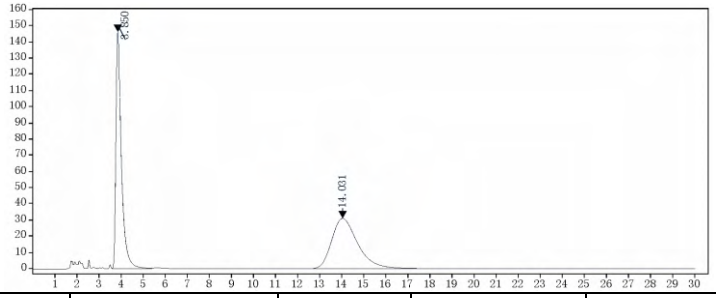


Entry	Retention Time	Height	Area	Area%
1	17.145	5.599	330.441	4.187
2	21.500	178.457	7562.007	95.813

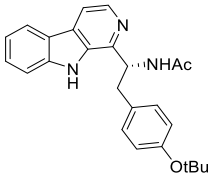


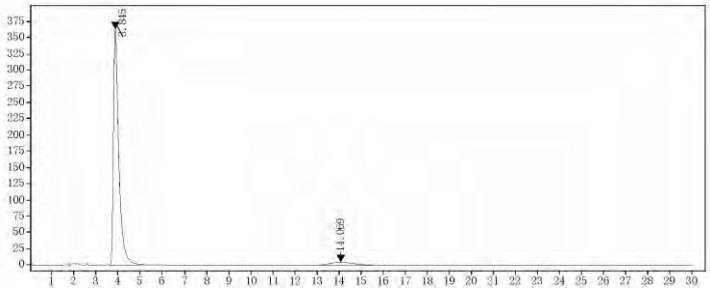
(*R*)-*N*-(2-(4-(tert-butoxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5u**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 76.2 mg, 95% yield; 88% ee). **Mp**: 186−188 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.51 (s, 1H), 8.54 (d, J = 8.3 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.18 (d, J = 7.9 Hz, 1H), 8.00 (d, J = 5.3 Hz, 1H), 7.66 – 7.40 (m, 2H), 7.21 (t, J = 7.4 Hz, 1H), 7.09 (d, J = 8.0 Hz, 2H), 6.74 (d, J = 7.9 Hz, 2H), 5.76 (q, J = 7.7 Hz, 1H), 3.21 – 2.98 (m, 2H), 1.80 (s, 3H), 1.14 (s, 9H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 168.9, 153.1, 145.0, 140.5, 137.2, 133.0, 129.8, 129.0, 128.0, 123.6, 123.2, 121.6, 120.7, 119.2, 113.6, 112.0, 77.5, 51.4, 34.5, 28.4, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3216, 3118, 2974, 2908, 2376, 2342, 1774, 1752, 1648, 1581, 1544, 1508, 1482, 1385, 1244, 1070, 900, 820, 738, 666, 559; **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{28}N_3O_2^+$ [M + H]$^+$, 402.2176; found, 402.2171; **HPLC analysis:** Chiral ND (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 3.85 min, t_r (minor) = 14.07 min; **[α]$_D^{20}$** = 47.8 (c = 0.3, $CHCl_3$).



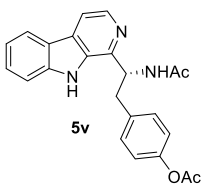


Entry	Retention Time	Height	Area	Area%
1	3.850	146.141	2536.158	49.809
2	14.031	31.117	2555.613	50.191



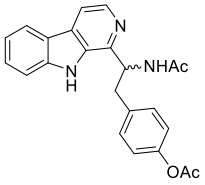


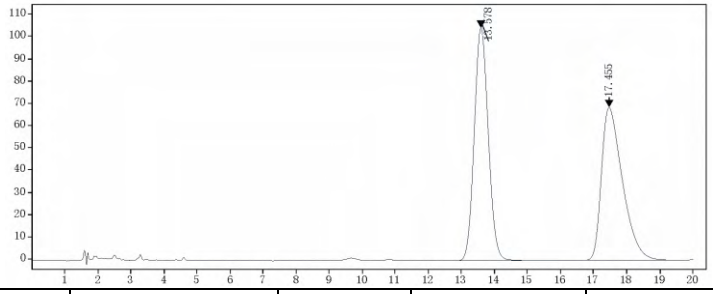
Entry	Retention Time	Height	Area	Area%
1	3.845	362.751	6135.983	94.051
2	14.069	4.763	388.109	5.949



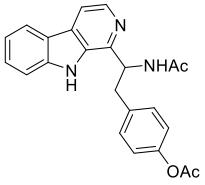
5v

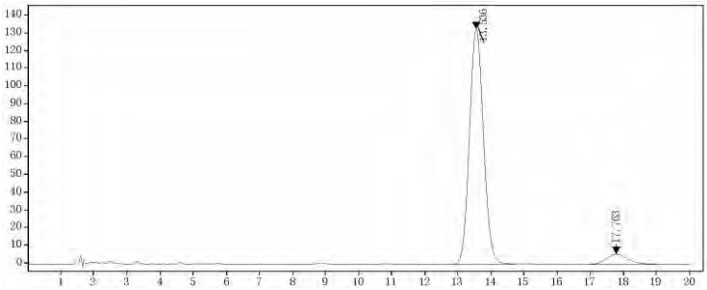
(*R*)-4-(2-acetamido-2-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)phenyl acetate (**5v**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 53.4 mg, 69% yield, 88% ee). **Mp:** 204−206 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.62 (s, 1H), 8.57 (d, J = 8.4 Hz, 1H), 8.33 (d, J = 5.2 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.02 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.40 – 7.12 (m, 3H), 6.97 (d, J = 8.0 Hz, 2H), 5.81 (td, J = 8.6, 5.6 Hz, 1H), 3.20 (qd, J = 14.0, 7.2 Hz, 2H), 2.22 (s, 3H), 1.80 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 169.1, 148.8, 145.0, 140.5, 137.3, 135.9, 133.0, 130.3, 128.1, 128.0, 121.6, 121.1, 120.8, 119.3, 113.7, 112.0, 51.2, 38.9, 22.5, 20.8; **FTIR** (ν_{max}, cm^{-1}): 3216, 3177, 2983, 2928, 2382, 2335, 1760, 1647, 1558, 1542, 1500, 1457, 1431, 1374, 1228, 1071, 914, 822, 738, 668, 623, 570; **HRMS** (ESI-TOF) m/z: calcd for $C_{23}H_{22}N_3O_3^+$ [M + H]$^+$, 388.1656; found, 388.1649; **HPLC analysis:** Chiral NX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 13.54 min, t_r (minor) = 17.76 min; **[α]$_D^{20}$** = 44.0 (c = 0.3, $CHCl_3$).



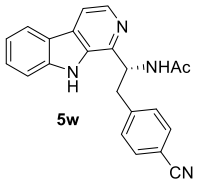


Entry	Retention Time	Height	Area	Area%
1	13.578	104.487	3082.700	50.503
2	17.455	68.646	3021.239	49.497

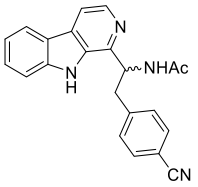


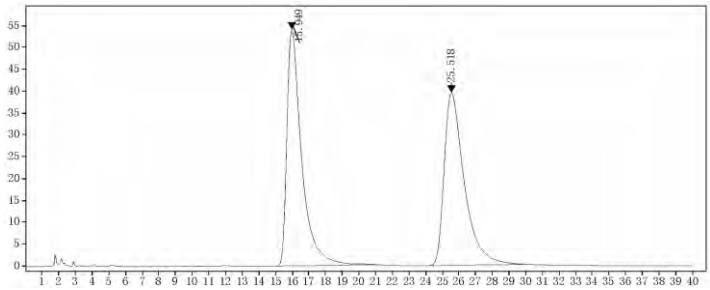


Entry	Retention Time	Height	Area	Area%
1	13.536	132.664	3900.318	93.998
2	17.763	5.649	249.039	6.002

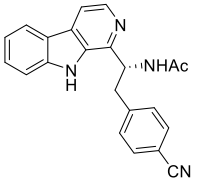


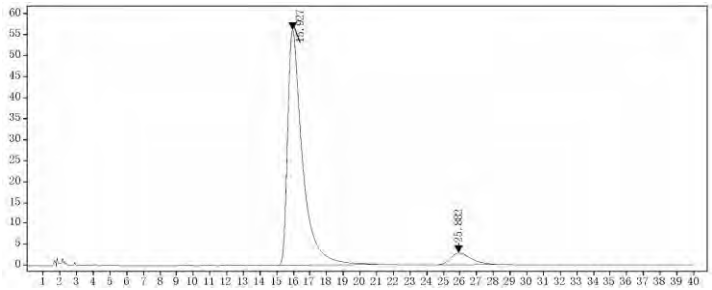
(*R*)-*N*-(2-(4-cyanophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5w**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 25% acetone in hexanes, 56.7 mg, 80% yield; 87% ee). **Mp**: 206−208 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.66 (s, 1H), 8.62 (d, J = 8.5 Hz, 1H), 8.34 (d, J = 5.1 Hz, 1H), 8.22 (d, J = 7.9 Hz, 1H), 8.04 (d, J = 5.2 Hz, 1H), 7.71 (d, J = 8.0 Hz, 2H), 7.62 (d, J = 8.2 Hz, 1H), 7.59 – 7.52 (m, 1H), 7.50 (d, J = 8.0 Hz, 2H), 7.31 – 7.17 (m, 1H), 5.86 (td, J = 8.8, 5.5 Hz, 1H), 3.32 – 3.18 (m, 2H), 1.79 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.1, 144.6, 144.4, 140.5, 137.4, 132.9, 131.7, 130.6, 128.2, 128.1, 121.7, 120.8, 119.4, 119.0, 113.9, 112.0, 109.0, 50.7, 39.7, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3230, 3177, 2979, 2942, 2383, 2338, 2226, 1748, 1715, 1698, 1658, 1648, 1558, 1537, 1455, 1430, 1377, 1318, 1238, 1059, 884, 818, 738, 625, 554; **HRMS** (ESI-TOF) m/z: calcd for $C_{22}H_{19}N_4O^+$ [M + H]$^+$, 355.1553; found, 355.1549; **HPLC analysis:** Chiral MX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 15.93 min, t_r (minor) = 25.88 min; **[α]$_D^{20}$** = 106.9 (c = 0.3, $CHCl_3$).



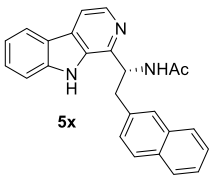


Entry	Retention Time	Height	Area	Area%
1	15.949	54.015	3316.629	50.033
2	25.518	39.45	3312.215	49.967

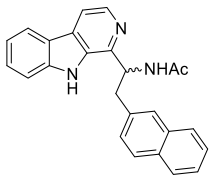


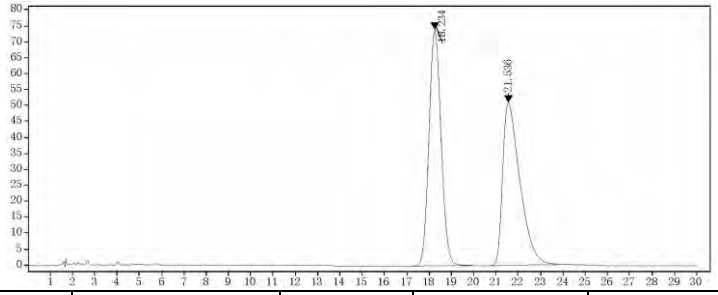


Entry	Retention Time	Height	Area	Area%
1	15.927	56.221	3458.233	93.448
2	25.882	2.803	242.459	6.552

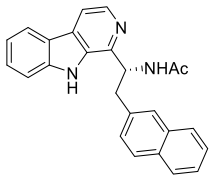


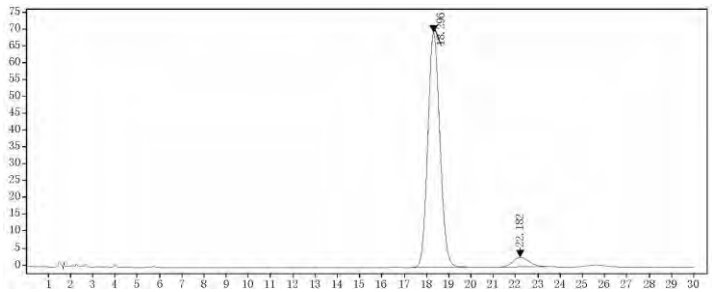
(*R*)-*N*-(2-(naphthalen-2-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5x**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 50.8 mg, 67% yield; 90% ee). **Mp**: 210−212 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.66 (s, 1H), 8.61 (d, J = 8.4 Hz, 1H), 8.35 (d, J = 5.3 Hz, 1H), 8.21 (d, J = 7.9 Hz, 1H), 8.03 (d, J = 5.2 Hz, 1H), 7.78 (td, J = 11.9, 9.0 Hz, 4H), 7.62 (d, J = 8.2 Hz, 1H), 7.54 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.50 – 7.36 (m, 3H), 7.29 – 7.17 (m, 1H), 5.95 (td, J = 8.6, 5.8 Hz, 1H), 3.42 (dd, J = 13.7, 5.8 Hz, 1H), 3.34 (dd, J = 13.7, 5.8 Hz, 1H), 1.78 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.0, 145.0, 140.5, 137.4, 136.2, 133.0, 132.9, 131.7, 128.2, 128.1, 128.0, 127.6, 127.4, 127.3, 127.1, 125.9, 125.3, 121.6, 120.8, 119.3, 113.8, 112.0, 51.2, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3546, 3526, 3503, 2987, 2971, 2901, 2891, 2780, 2741, 1749, 1715, 1698, 1647, 1558, 1542, 1522, 1507, 1458, 1397, 1055, 737, 669; **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{22}N_3O^+$ [M + H]$^+$, 380.1757; found, 380.1753; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 18.30 min, t_r (minor) = 22.18 min; **[α]$_D^{20}$** = –15.0 (c = 0.3, $CHCl_3$).



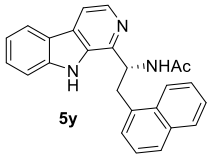


Entry	Retention Time	Height	Area	Area%
1	18.234	73.971	2798.119	50.479
2	21.536	50.903	2744.987	49.521

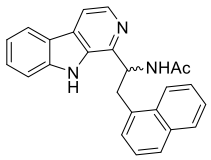


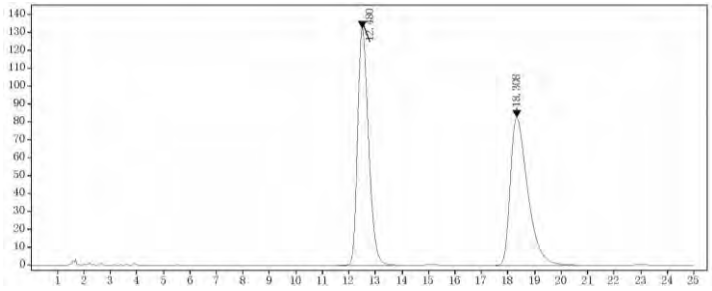


Entry	Retention Time	Height	Area	Area%
1	18.296	69.611	2627.988	94.831
2	22.182	2.824	143.258	5.169

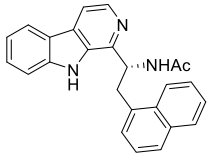


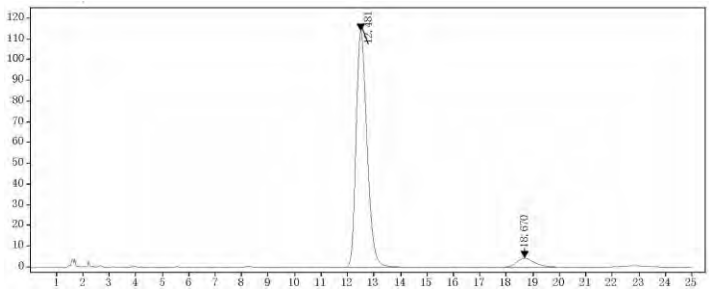
(*R*)-*N*-(2-(naphthalen-1-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5y**) was prepared as a white solid according to the General Procedure I (purification by flash column chromatography: 20% acetone in hexanes, 52.4 mg, 69% yield; 88% ee). **Mp**: 220−222 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.41 (s, 1H), 8.57 (d, *J* = 8.0 Hz, 1H), 8.36 (d, *J* = 5.2 Hz, 1H), 8.26 (d, *J* = 8.1 Hz, 1H), 8.19 (d, *J* = 7.9 Hz, 1H), 8.03 (d, *J* = 5.2 Hz, 1H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.49 (dt, *J* = 14.2, 7.0 Hz, 3H), 7.38 – 7.25 (m, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 5.95 (q, *J* = 7.4 Hz, 1H), 3.91 – 3.64 (m, 2H), 1.81 (s, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.3, 144.4, 140.5, 137.4, 134.4, 133.3, 133.3, 132.0, 128.4, 128.2, 128.1, 127.2, 126.7, 126.0, 125.4, 125.2, 124.0, 121.6, 120.8, 119.3, 113.9, 112.1, 51.0, 35.8, 22.5; **FTIR** (ν_{max}, cm^{-1}): 3221, 3179, 2981, 2908, 2382, 2337, 1698, 1648, 1542, 1506, 1474, 1373, 1319, 1234, 1089, 874, 749, 664, 561; **HRMS** (ESI-TOF) m/z: calcd for $C_{25}H_{22}N_3O^+$ [M + H]$^+$, 380.1757; found, 380.1753; **HPLC analysis:** Chiral MX (2) (150 x 4.6 mm, 3 µm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 12.48 min, t_r (minor) = 18.67 min; **[α]$_D^{20}$** = 15.7 (c = 0.3, $CHCl_3$).



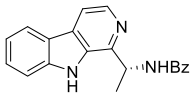


Entry	Retention Time	Height	Area	Area%
1	12.480	132.453	3806.458	50.130
2	18.308	82.661	3786.686	49.870



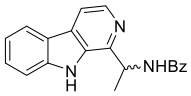


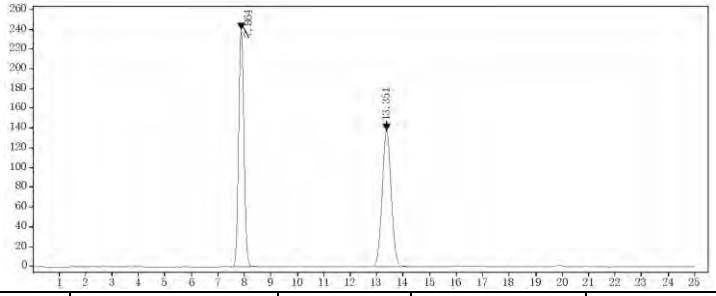
Entry	Retention Time	Height	Area	Area%
1	12.481	113.828	3257.848	93.949
2	18.670	4.317	209.814	6.051



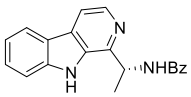
3ab

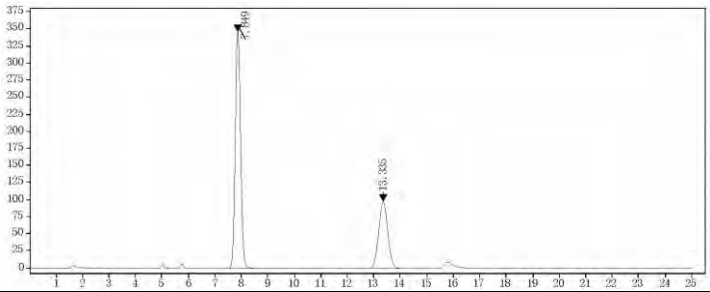
(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)benzamide (**3ab**) was prepared as a white solid (37.8 mg, 60% yield; 34% ee) according to the following procedure: sequentially, β-Carbolines (0.20 mmol, 1.0 equiv.), redox-active ester (RAEs) (0.30 mmol, 1.5 equiv.), $Ir[dF(CF_3)ppy)_2(dtbpy)]PF_6$ (2.2 mg, 0.002 mmol, 1 mol%), and (*R*)-STRIP (14.4 mg, 0.02 mmol, 10 mol%) were added to a 10 mL Schlenk tube containing a stirrer bar. After evacuated and refilled with argon three times, anhydrous, freshly argon-sparged THF (4.0 mL) was then added via syringe. The reaction mixture was stirred under an argon atmosphere at 25 °C, then irradiated by 3 W blue LED (λ = 455−465 nm) for another 48 h The reaction was monitored by TLC. After completion of the reaction, the solvent was removed in vacuo and the crude residue was purification by flash column chromatography (10% acetone in hexanes). **Mp**: 168−170 °C; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.70 (s, 1H), 8.94 (d, *J* = 7.4 Hz, 1H), 8.30 (d, *J* = 5.3 Hz, 1H), 8.23 (d, *J* = 7.9 Hz, 1H), 8.03 (d, *J* = 5.2 Hz, 1H), 7.98 – 7.86 (m, 2H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.54 (q, *J* = 7.4 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.25 (t, *J* = 7.5 Hz, 1H), 5.82 (p, *J* = 6.9 Hz, 1H), 1.64 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 165.8, 146.2, 140.5, 137.3, 131.3, 128.3, 128.1, 128.0, 127.5, 123.0, 121.7, 120.9, 119.4, 113.8, 112.1, 47.0, 20.2; **FTIR** (ν_{max}, cm^{-1}): 3312, 3199, 3061, 2918, 2382, 1752, 1631, 1522, 1389, 1307, 1091, 1050, 716, 646, 537; **HRMS** (ESI-TOF) m/z: calcd for $C_{20}H_{18}N_3O^+$ $[M + H]^+$, 316.1444; found, 316.1441; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 µm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 7.85 min, t_r (minor) = 13.33 min.



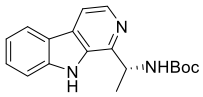


Entry	Retention Time	Height	Area	Area%
1	7.864	239.693	3292.599	49.911
2	13.351	137.443	3304.353	50.089



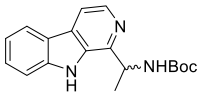


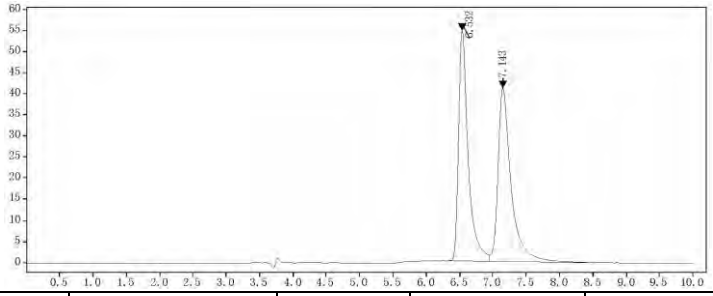
Entry	Retention Time	Height	Area	Area%
1	7.849	346.657	4781.334	67.099
2	13.335	97.718	2344.499	32.901



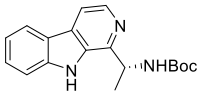
3ac

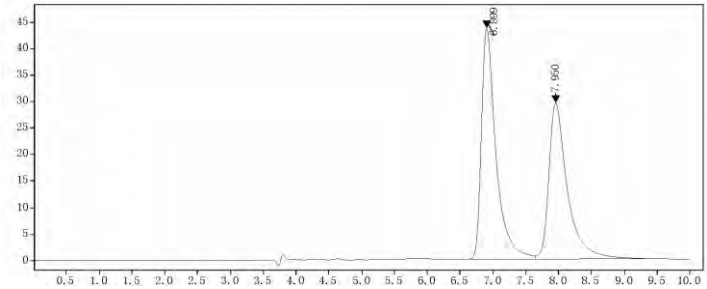
(*R*)-tert-butyl (1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ac**) was prepared as a white solid (40.5 mg, 65% yield; 7% ee) according to the following procedure: sequentially, β-Carbolines (0.20 mmol, 1.0 equiv.), redox-active ester (RAEs) (0.30 mmol, 1.5 equiv.), Ir[dF(CF_3)ppy)$_2$(dtbpy)]PF_6 (2.2 mg, 0.002 mmol, 1 mol%), and (*R*)-STRIP (14.4 mg, 0.02 mmol, 10 mol%) were added to a 10 mL Schlenk tube containing a stirrer bar. After evacuated and refilled with argon three times, anhydrous, freshly argon-sparged THF (4.0 mL) was then added via syringe. The reaction mixture was stirred under an argon atmosphere at 25 °C, then irradiated by 3 W blue LED (λ = 455−465 nm) for another 48 h The reaction was monitored by TLC. After completion of the reaction, the solvent was removed in vacuo and the crude residue was purification by flash column chromatography (10% acetone in hexanes). **Mp**: 133−135 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.58 (s, 1H), 8.27 (d, *J* = 5.2 Hz, 1H), 8.22 (d, *J* = 7.9 Hz, 1H), 8.00 (d, *J* = 5.3 Hz, 1H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.23 (q, *J* = 7.3 Hz, 1H), 5.30 (p, *J* = 6.9 Hz, 1H), 1.46 (d, *J* = 6.8 Hz, 3H), 1.36 (s, 9H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 169.3, 146.4, 140.5, 137.2, 132.6, 128.1, 128.0, 121.7, 120.9, 119.4, 113.7, 112.0, 78.0, 47.8, 28.2, 20.7; **FTIR** (ν_{max}, cm^{-1}): 2971, 2905, 2359, 1698, 1544, 1505, 1459, 1398, 1165, 1070, 879, 716, 545; **HRMS** (ESI-TOF) m/z: calcd for $C_{18}H_{22}N_3O_2^+$ $[M + H]^+$, 312.1707; found, 312.1702; **HPLC analysis:** Chiral INC(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 99:1, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 6.90 min, t_r (minor) = 7.95 min.



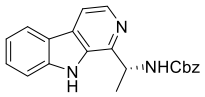


Entry	Retention Time	Height	Area	Area%
1	6.532	54.515	545.059	49.147
2	7.143	41.027	563.970	50.853



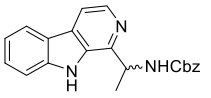


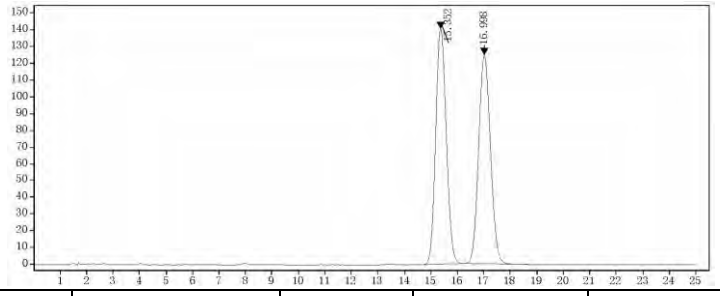
Entry	Retention Time	Height	Area	Area%
1	6.899	43.731	672.689	53.721
2	7.950	29.537	579.512	46.279



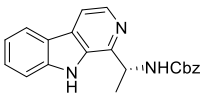
3ad

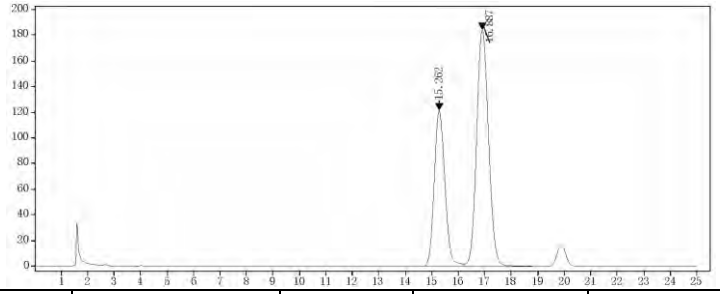
(*R*)-benzyl(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ad**) was prepared as a white solid (56.8 mg, 82% yield; 26% ee) according to the following procedure: sequentially, β-Carbolines (0.20 mmol, 1.0 equiv.), redox-active ester (RAEs) (0.30 mmol, 1.5 equiv.), Ir[dF(CF_3)ppy)$_2$(dtbpy)]PF_6 (2.2 mg, 0.002 mmol, 1 mol%), and (*R*)-STRIP (14.4 mg, 0.02 mmol, 10 mol%) were added to a 10 mL Schlenk tube containing a stirrer bar. After evacuated and refilled with argon three times, anhydrous, freshly argon-sparged THF (4.0 mL) was then added via syringe. The reaction mixture was stirred under an argon atmosphere at 25 °C, then irradiated by 3 W blue LED (λ = 455−465 nm) for another 48 h The reaction was monitored by TLC. After completion of the reaction, the solvent was removed in vacuo and the crude residue was purification by flash column chromatography (10% acetone in hexanes). **Mp**: 155−157 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.61 (s, 1H), 8.29 (d, *J* = 5.1 Hz, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 8.01 (d, *J* = 5.2 Hz, 1H), 7.76 (d, *J* = 7.7 Hz, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.44 – 7.18 (m, 6H), 5.38 (q, *J* = 7.1 Hz, 1H), 5.12 – 4.89 (m, 2H), 1.50 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 155.6, 146.1, 140.5, 137.3, 137.2, 132.3, 128.3, 128.1, 128.0, 127.7, 127.6, 121.7, 120.8, 119.3, 113.7, 112.0, 65.3, 48.3, 20.5; **FTIR** (ν_{max}, cm^{-1}): 3121, 2991, 2895, 2381, 1701, 1544, 1505, 1455, 1288, 1057, 1014, 733, 568; **HRMS** (ESI-TOF) m/z: calcd for $C_{21}H_{20}N_3O_2^+$ $[M + H]^+$, 346.1550; found, 346.1546; **HPLC analysis:** Chiral NX(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 95:5, 1.0 mL/min, 25 °C, 254 nm), t_r (major) = 16.89 min, t_r (minor) = 15.26 min.



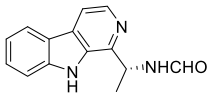


Entry	Retention Time	Height	Area	Area%
1	15.352	140.291	3873.916	49.997
2	16.998	124.446	3874.448	50.003



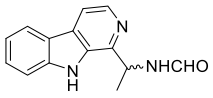


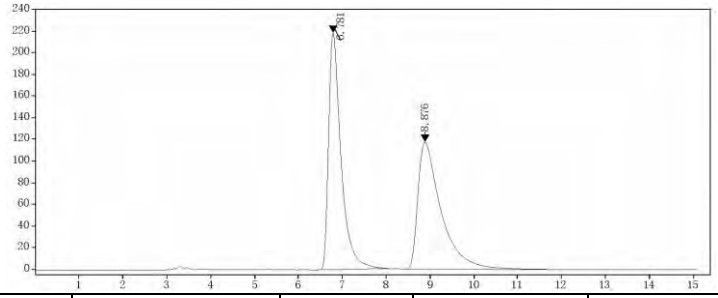
Entry	Retention Time	Height	Area	Area%
1	15.262	121.309	3386.441	37.071
2	16.887	184.152	5748.528	62.929



3ae

(*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3ae**) was prepared as a white solid (38.7 mg, 81% yield; 52% ee) according to the following procedure: sequentially, β-Carbolines (0.20 mmol, 1.0 equiv.), redox-active ester (RAEs) (0.30 mmol, 1.5 equiv.), Ir[dF(CF_3)ppy)$_2$(dtbpy)]PF_6 (2.2 mg, 0.002 mmol, 1 mol%), and (*R*)-STRIP (14.4 mg, 0.02 mmol, 10 mol%) were added to a 10 mL Schlenk tube containing a stirrer bar. After evacuated and refilled with argon three times, anhydrous, freshly argon-sparged THF (4.0 mL) was then added via syringe. The reaction mixture was stirred under an argon atmosphere at 25 °C, then irradiated by 3 W blue LED (λ = 455−465 nm) for another 48 h The reaction was monitored by TLC. After completion of the reaction, the solvent was removed in vacuo and the crude residue was purification by flash column chromatography (25% acetone in hexanes). **Mp**: 148−150 ℃ ; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.71 (s, 1H), 8.77 (d, *J* = 7.9 Hz, 1H), 8.31 (d, *J* = 5.2 Hz, 1H), 8.23 (d, *J* = 7.9 Hz, 1H), 8.09 (s, 1H), 8.04 (d, *J* = 5.2 Hz, 1H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.4 Hz, 1H), 5.69 (p, *J* = 6.9 Hz, 1H), 1.48 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (101 MHz, DMSO-d_6) δ 160.7, 145.3, 140.6, 137.3, 132.3, 128.2, 128.1, 121.7, 120.9, 119.4, 113.9, 112.1, 44.7, 20.9; **FTIR** (ν_{max}, cm^{-1}): 2990, 2896, 2359, 1754, 1672, 1587, 1543, 1505, 1459, 1243, 1052, 730, 619, 571; **HRMS** (ESI-TOF) m/z: calcd for $C_{14}H_{14}N_3O^+$ [M + H]$^+$, 240.1131; found, 240.1128; **HPLC analysis:** Chiral NY(2) (150 x 4.6 mm, 3 μm, hexane/i-PrOH = 90:10, 0.5 mL/min, 25 °C, 254 nm), t_r (major) = 6.77 min, t_r (minor) = 8.94 min; **[α]$_D^{20}$** = 52.8 (c = 0.3, $CHCl_3$).





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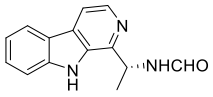
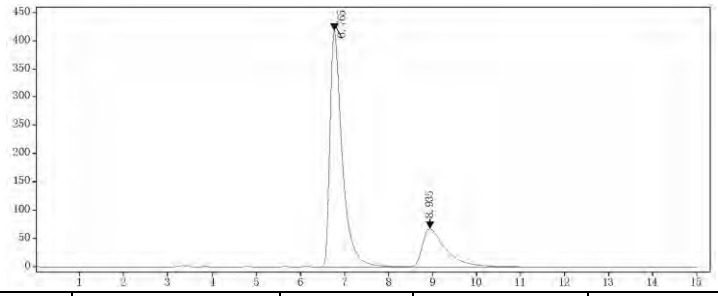
2	8.876	117.262	4305.648	50.010

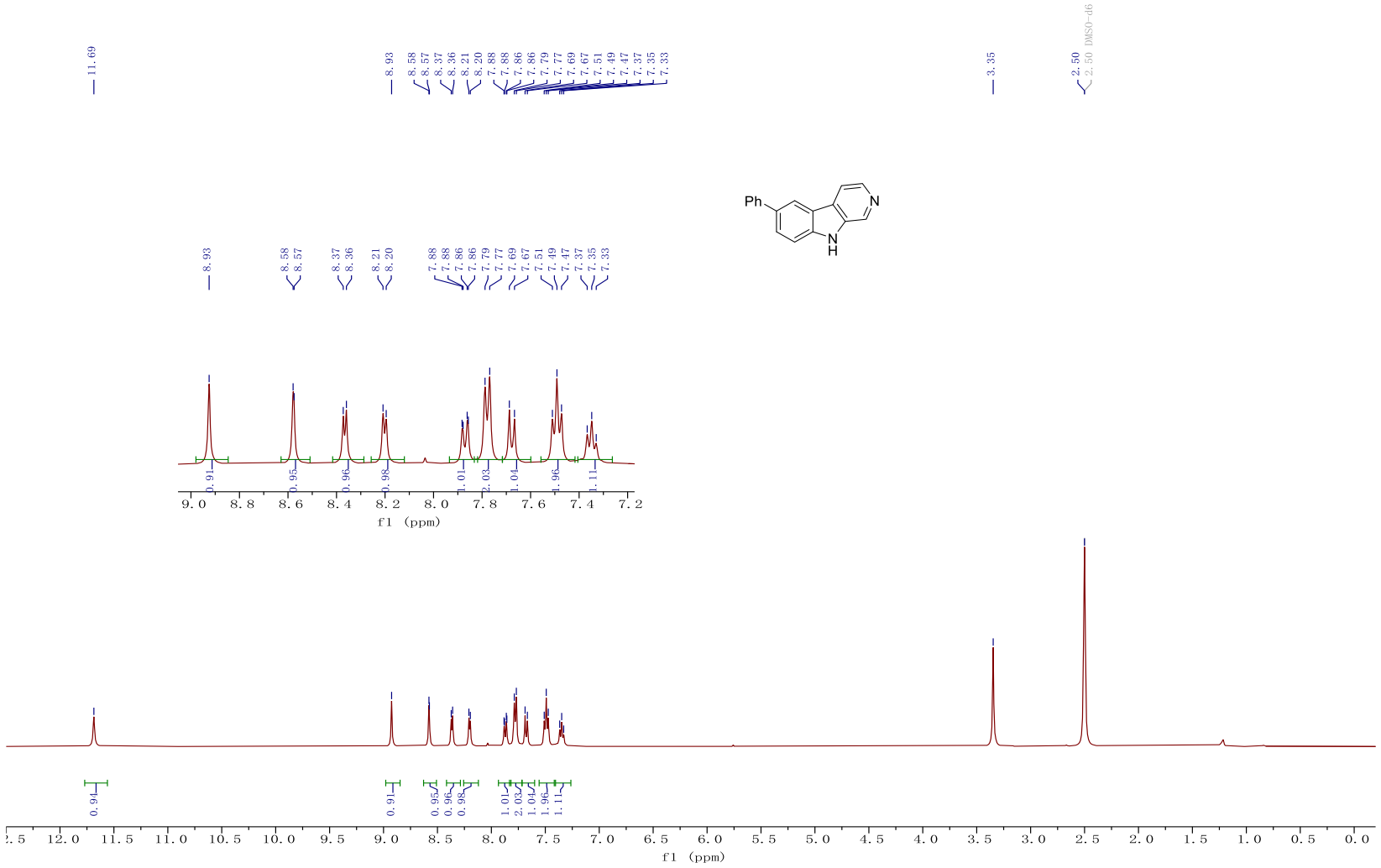




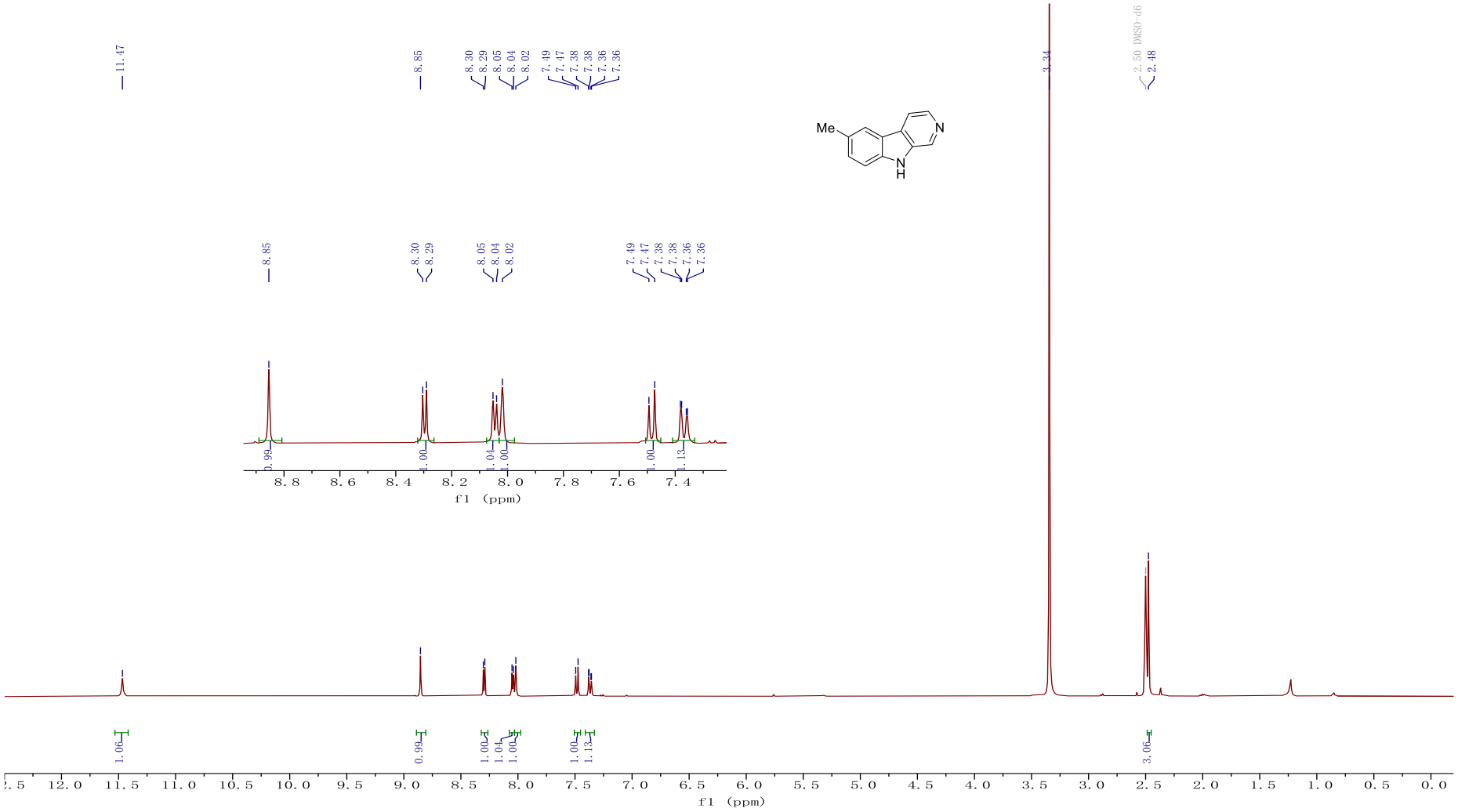
Entry	Retention Time	Height	Area	Area%
1	6.765	417.411	8118.536	76.061
2	8.935	67.548	2555.128	23.939

11 Copies of NMR spectra

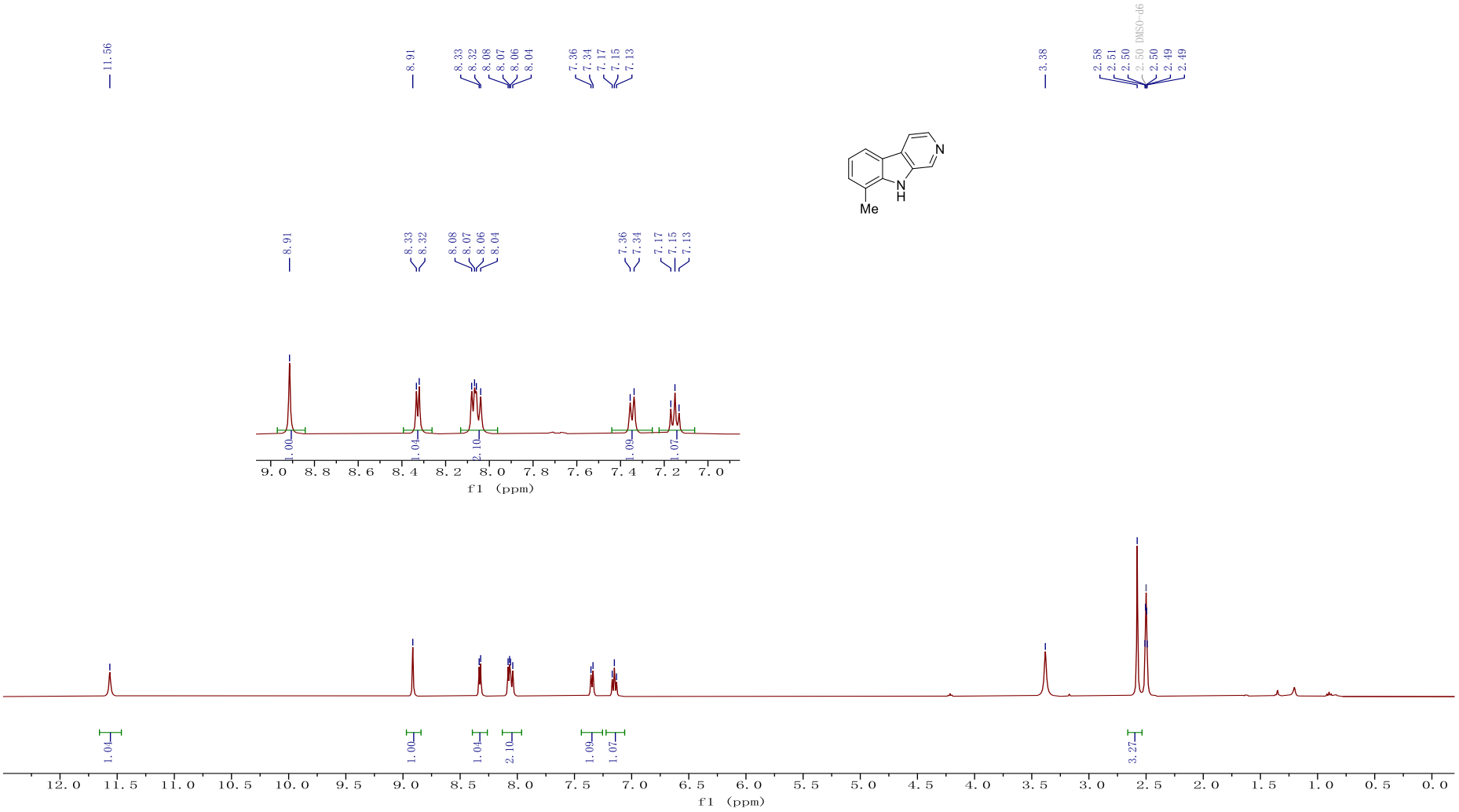
^{1}H NMR (400 MHz, DMSO-d_6) 6-phenyl-9*H*-pyrido[3,4-*b*]indole (**1b**)



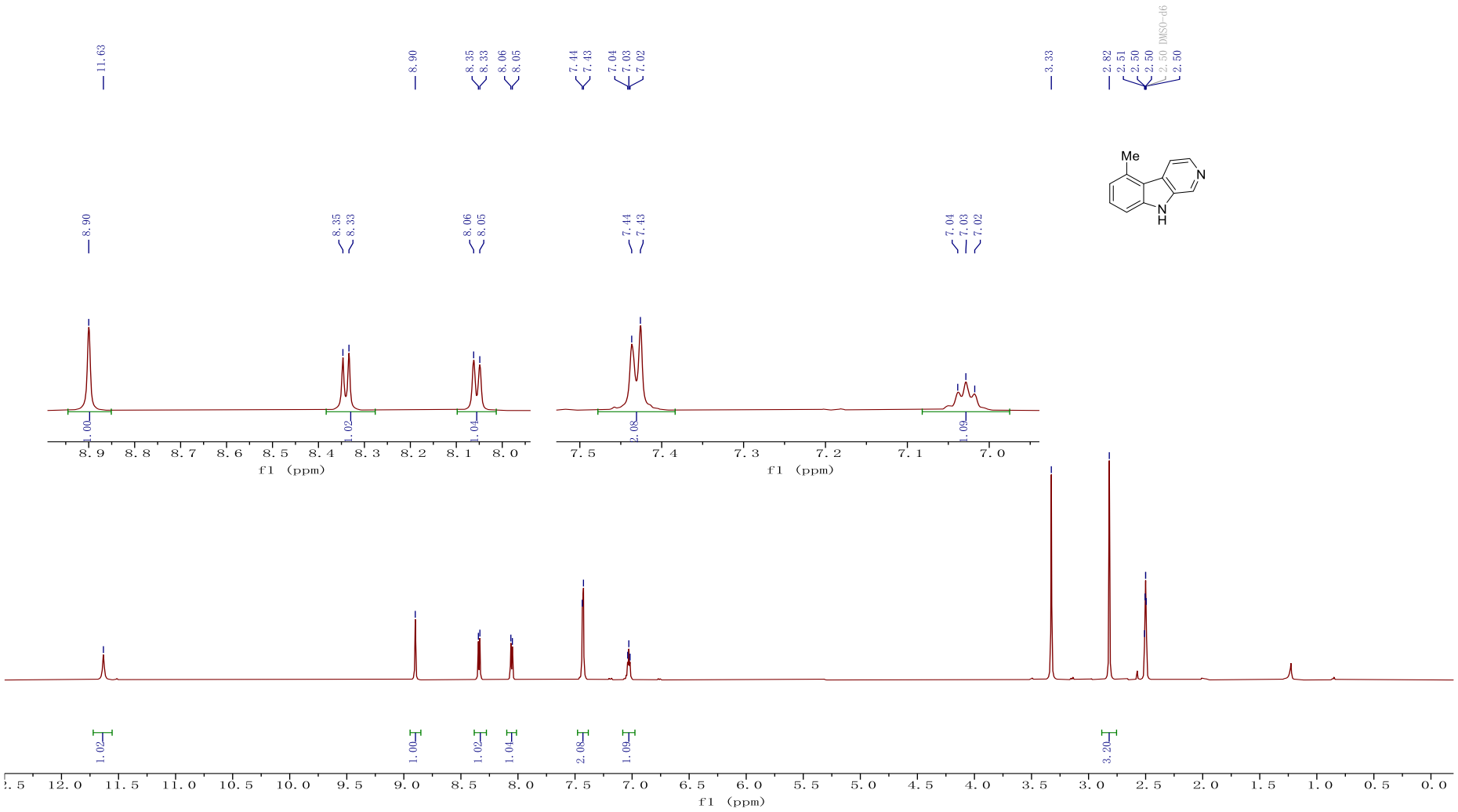
^{1}H NMR (400 MHz, DMSO-d_6) 6-methyl-9*H*-pyrido[3,4-*b*]indole (**1c**)



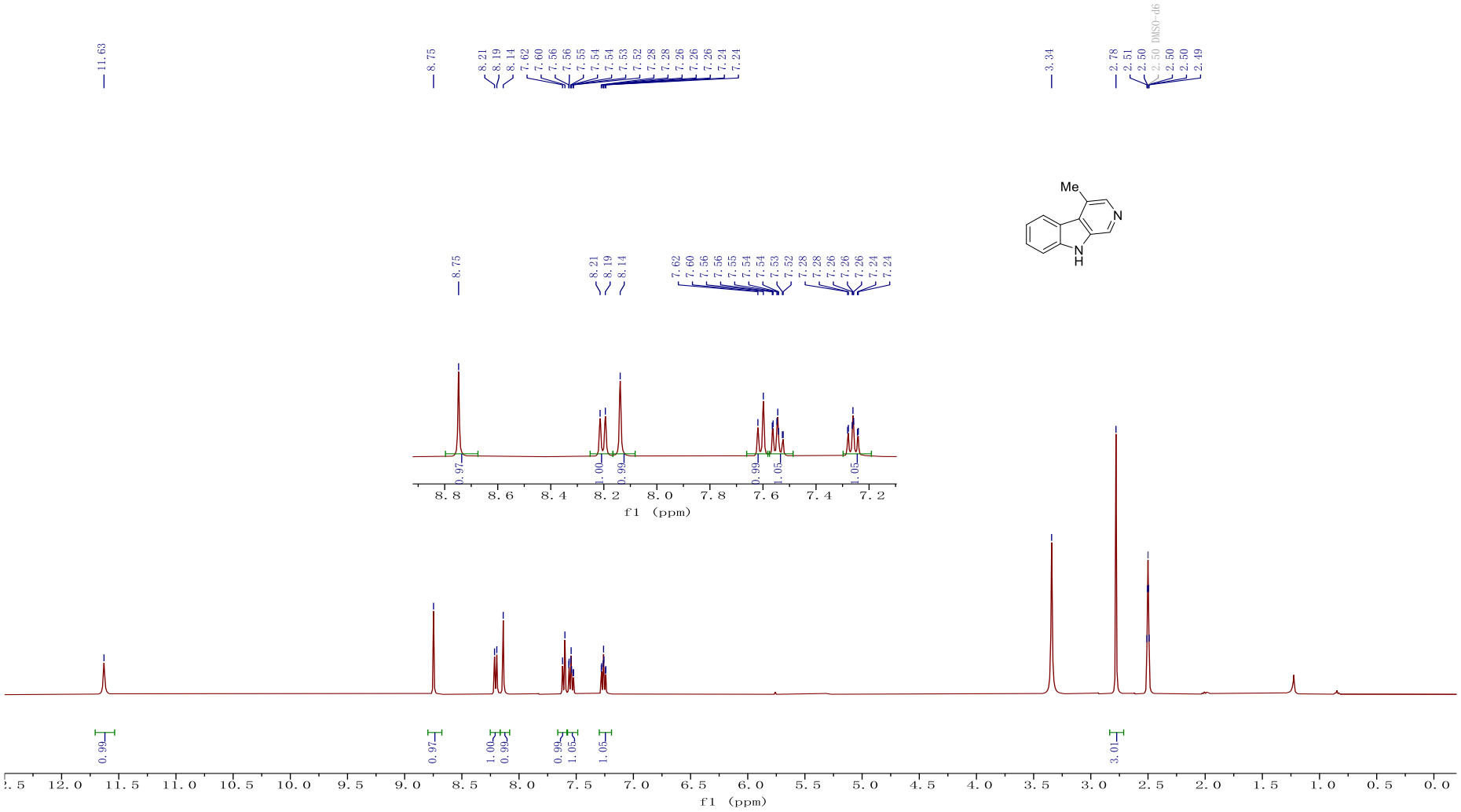
^{1}H NMR (400 MHz, DMSO-d_6) 8-methyl-9*H*-pyrido[3,4-*b*]indole (**1d**)



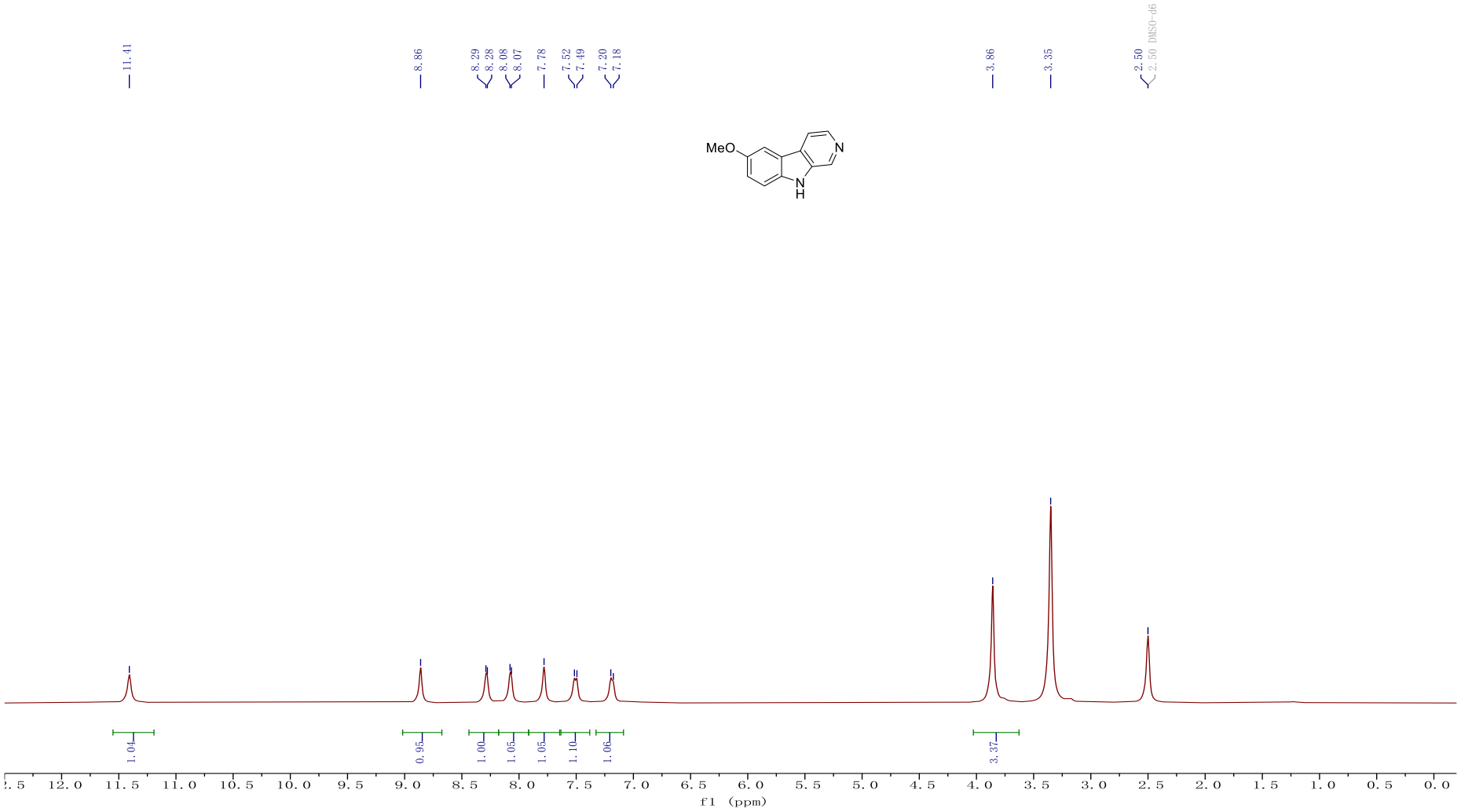
^{1}H NMR (400 MHz, DMSO-d_6) 5-methyl-9*H*-pyrido[3,4-*b*]indole (**1e**)



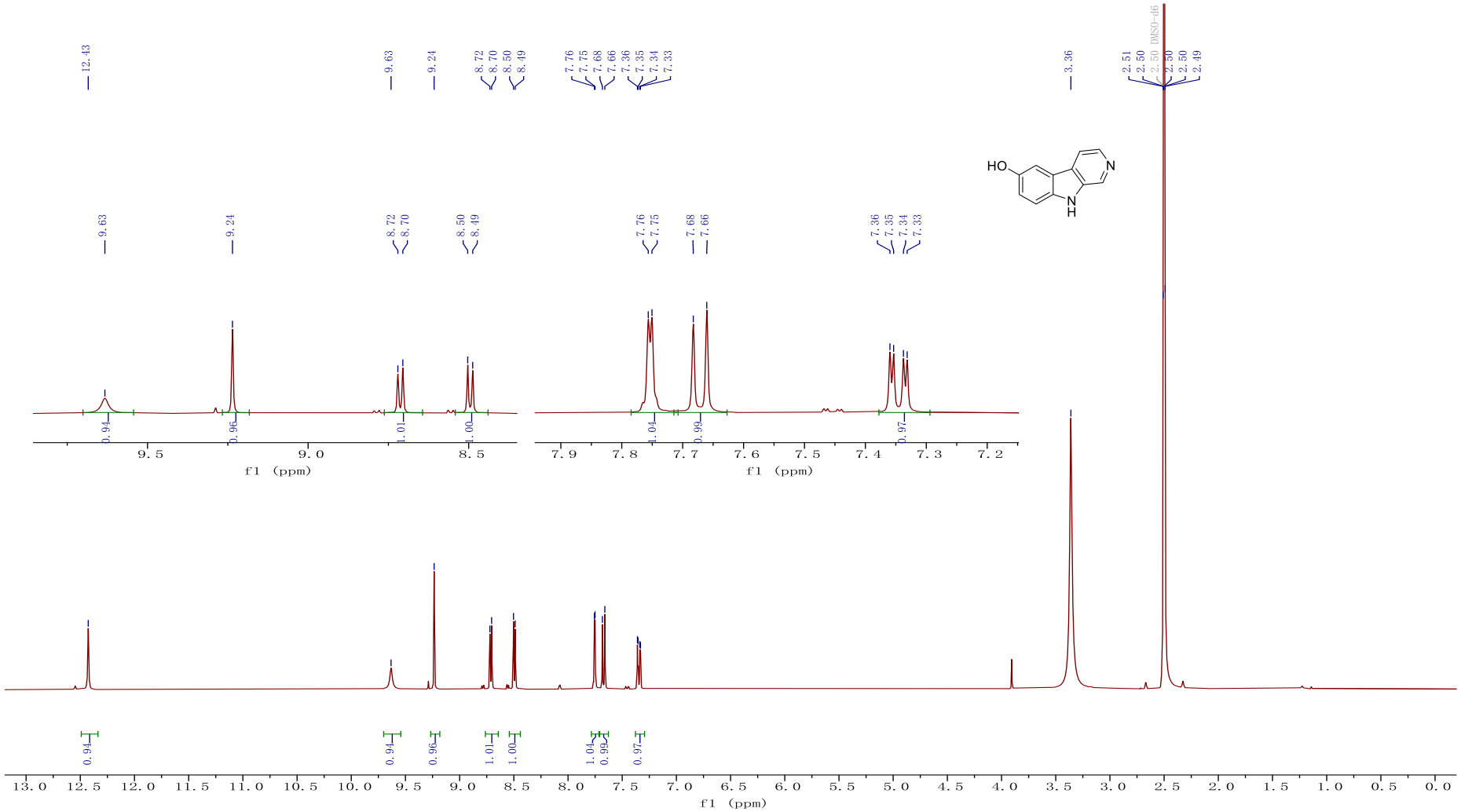
^{1}H NMR (400 MHz, DMSO-d_6) 4-methyl-9*H*-pyrido[3,4-*b*]indole (**1f**)



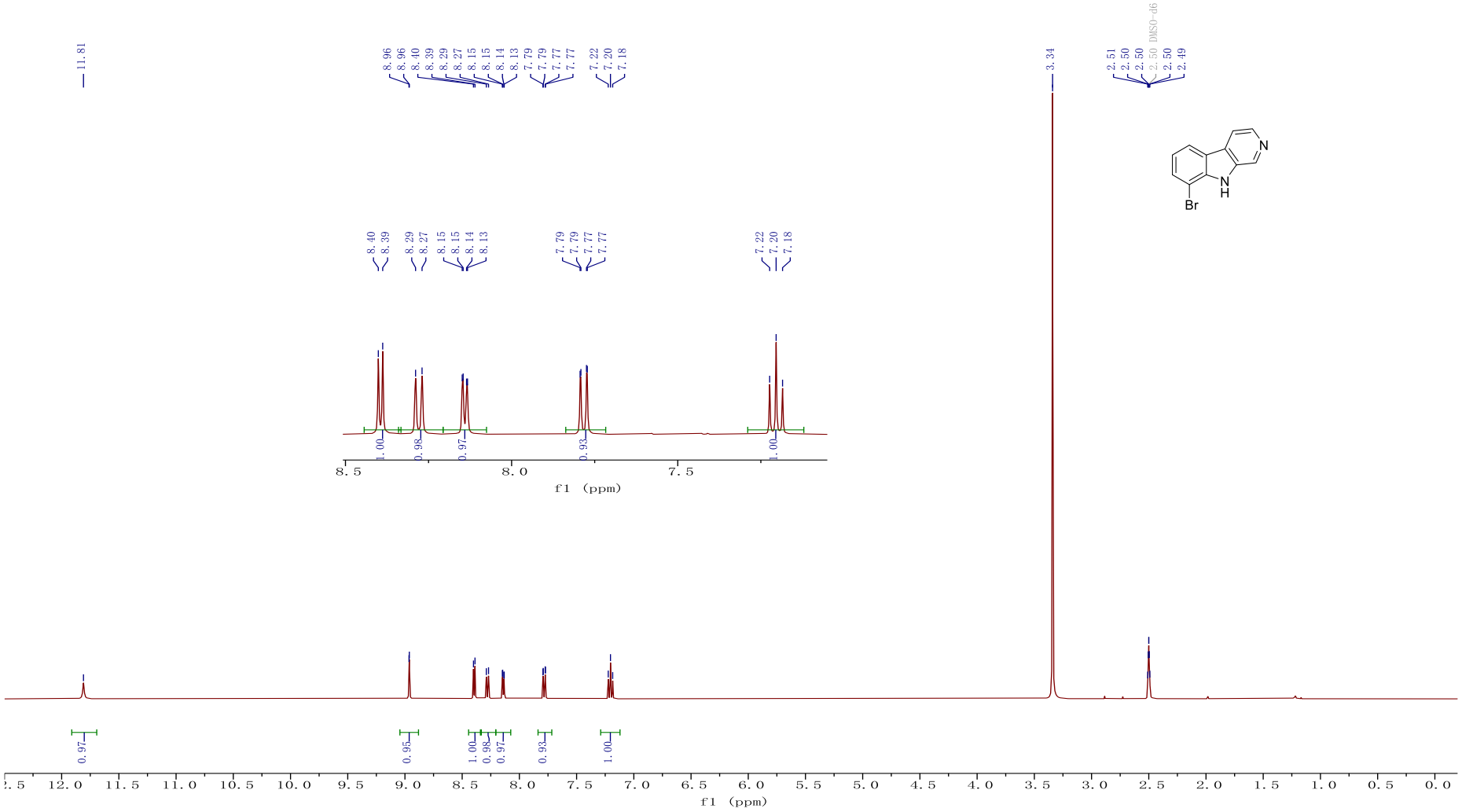
^{1}H NMR (400 MHz, DMSO-d_6) 6-methoxy-9*H*-pyrido[3,4-*b*]indole (**1g**)



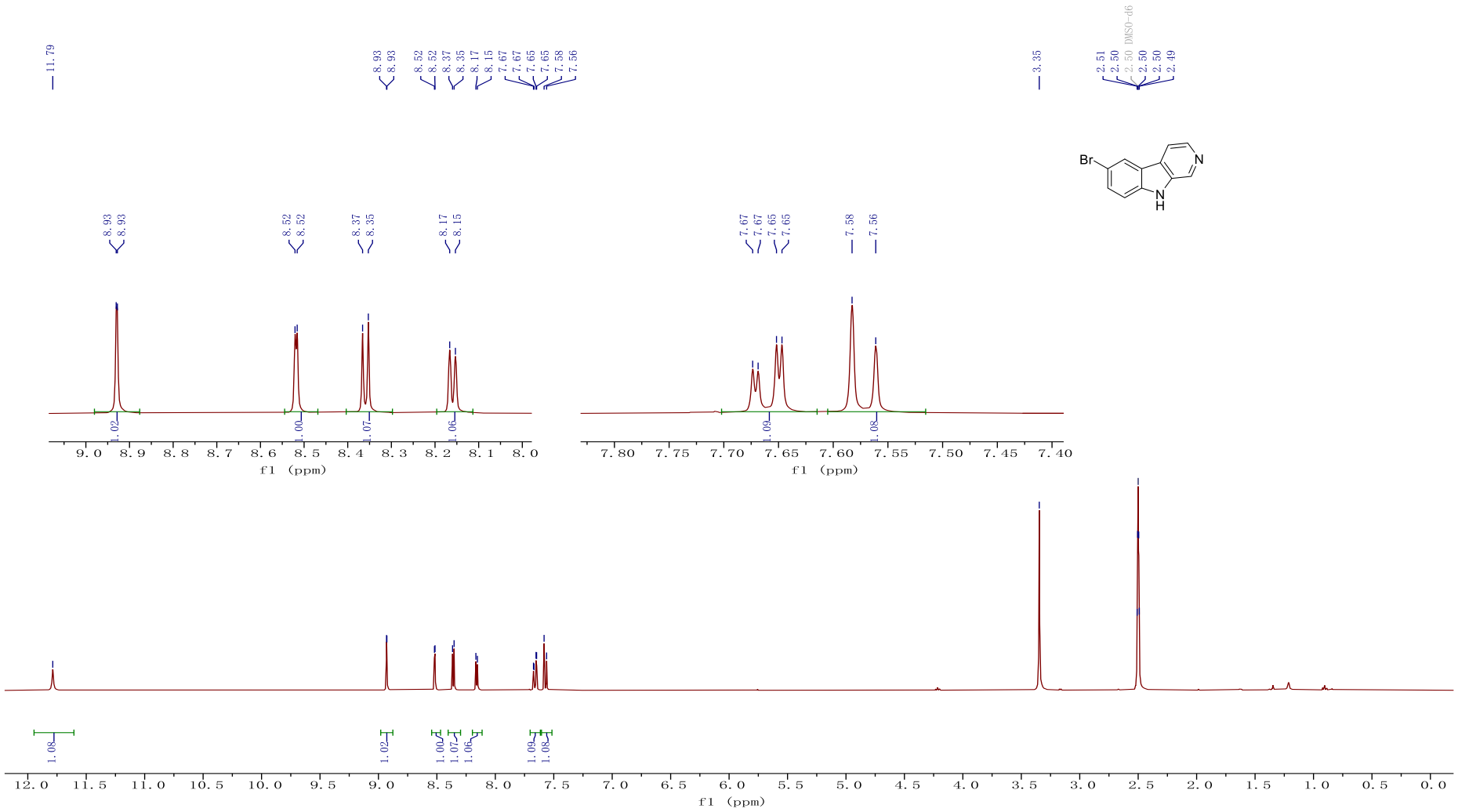
^{1}H NMR (400 MHz, DMSO-d_6) 9*H*-pyrido[3,4-*b*]indol-6-ol (**1h**)



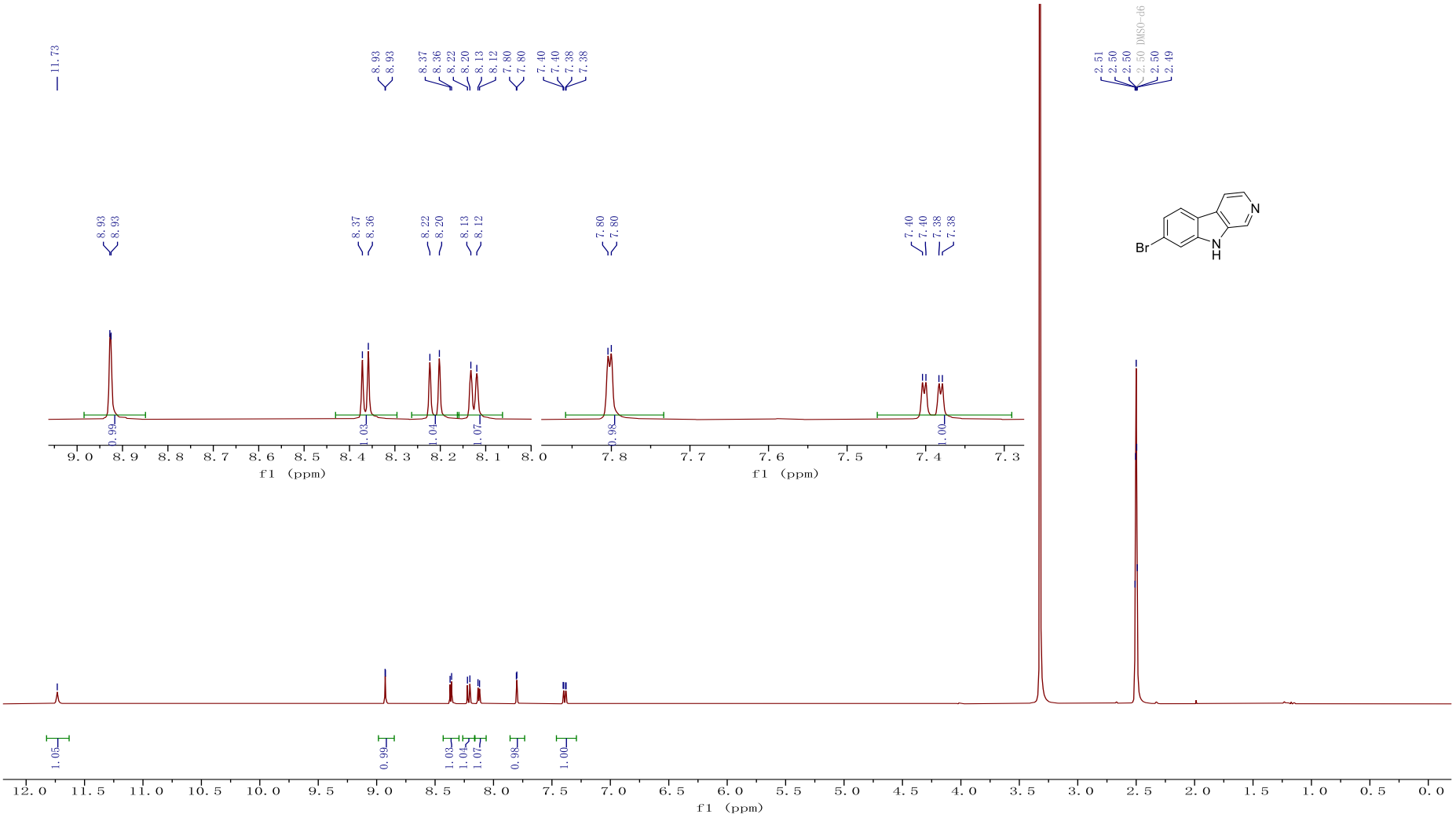
^{1}H NMR (400 MHz, DMSO-d_6) 8-bromo-9*H*-pyrido[3,4-*b*]indole (**1i**)



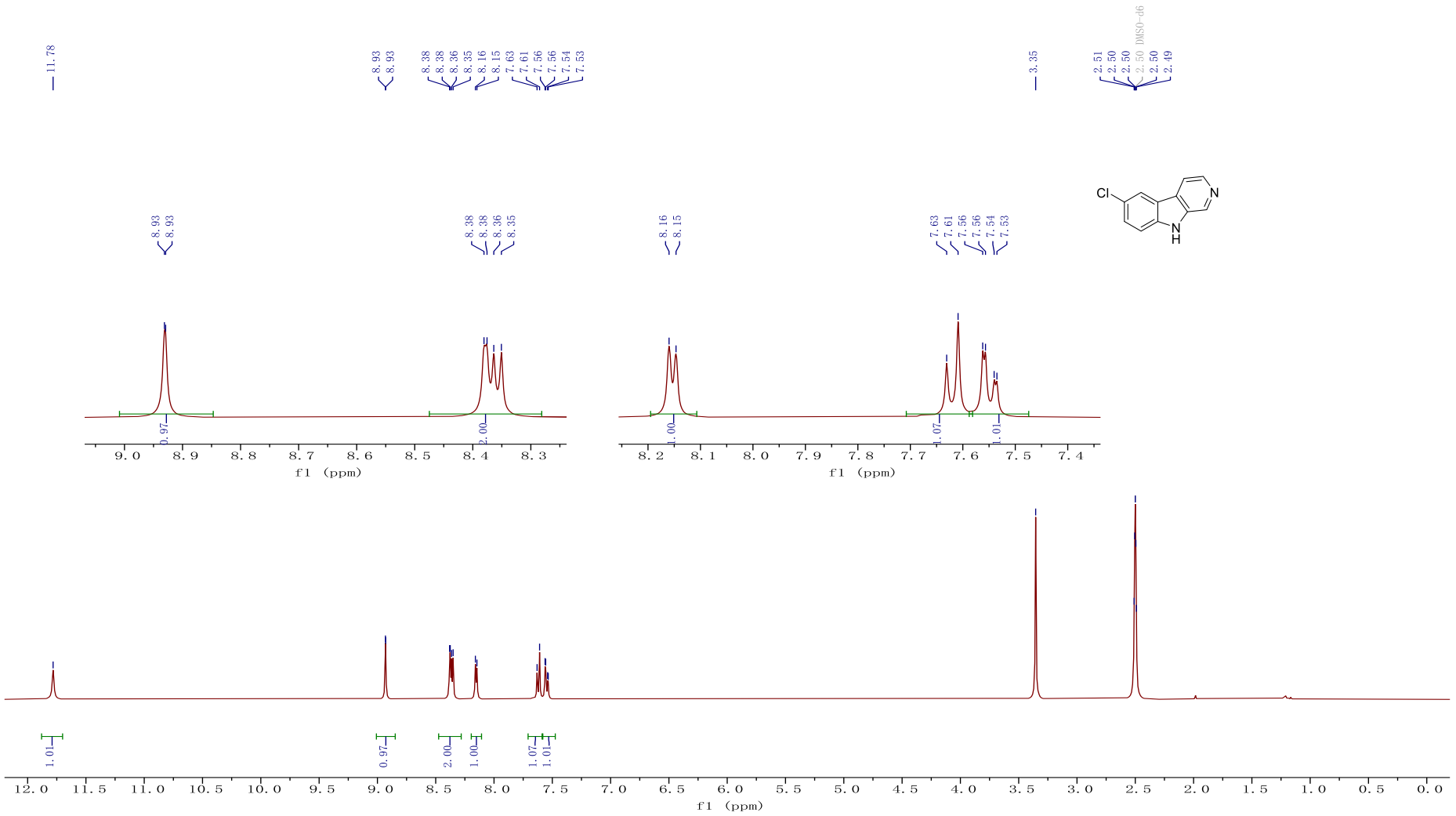
^{1}H NMR (400 MHz, DMSO-d_6) 6-bromo-9*H*-pyrido[3,4-*b*]indole (**1j**)



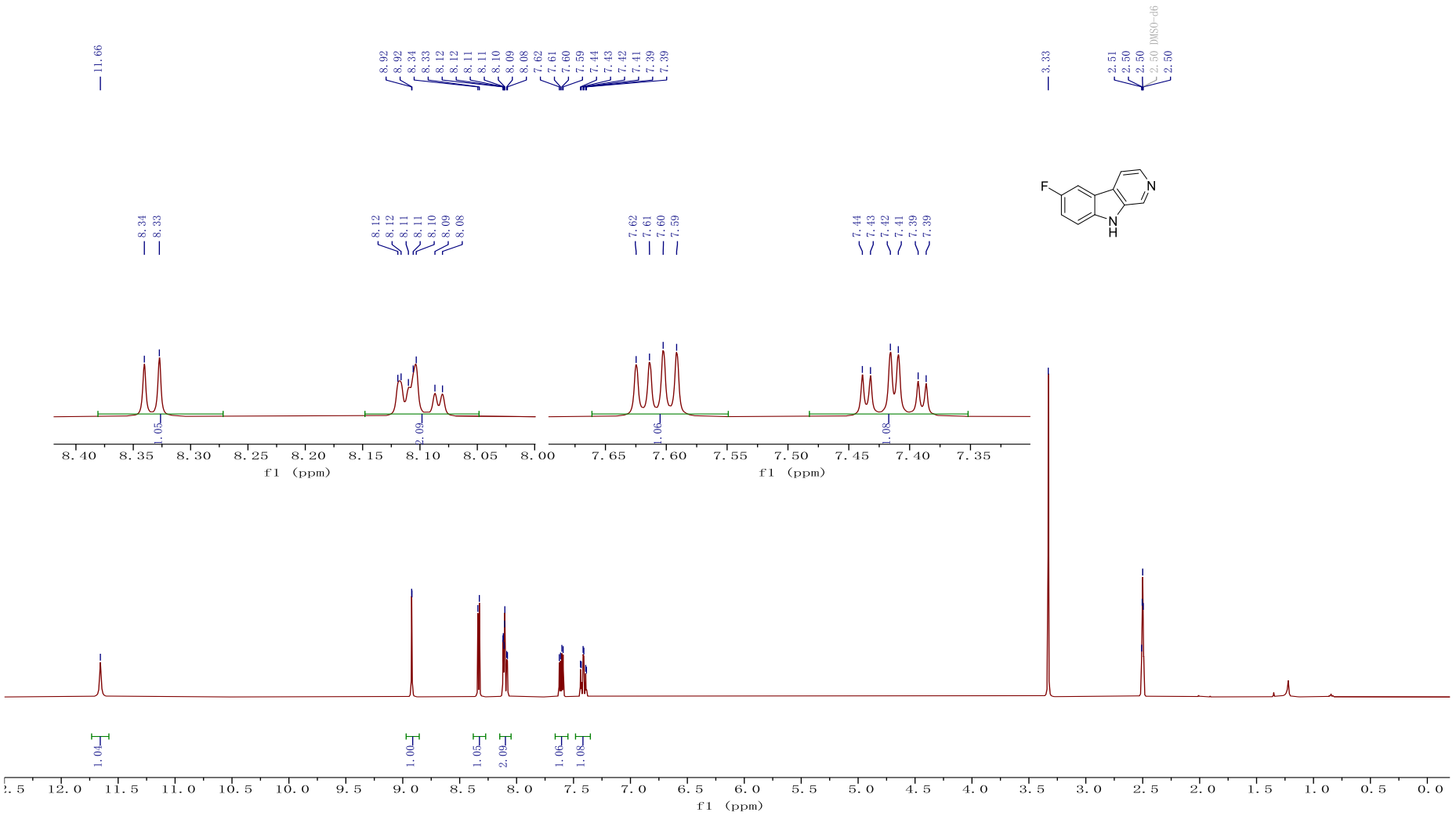
^{1}H NMR (400 MHz, DMSO-d_6) 7-bromo-9*H*-pyrido[3,4-*b*]indole (**1k**)



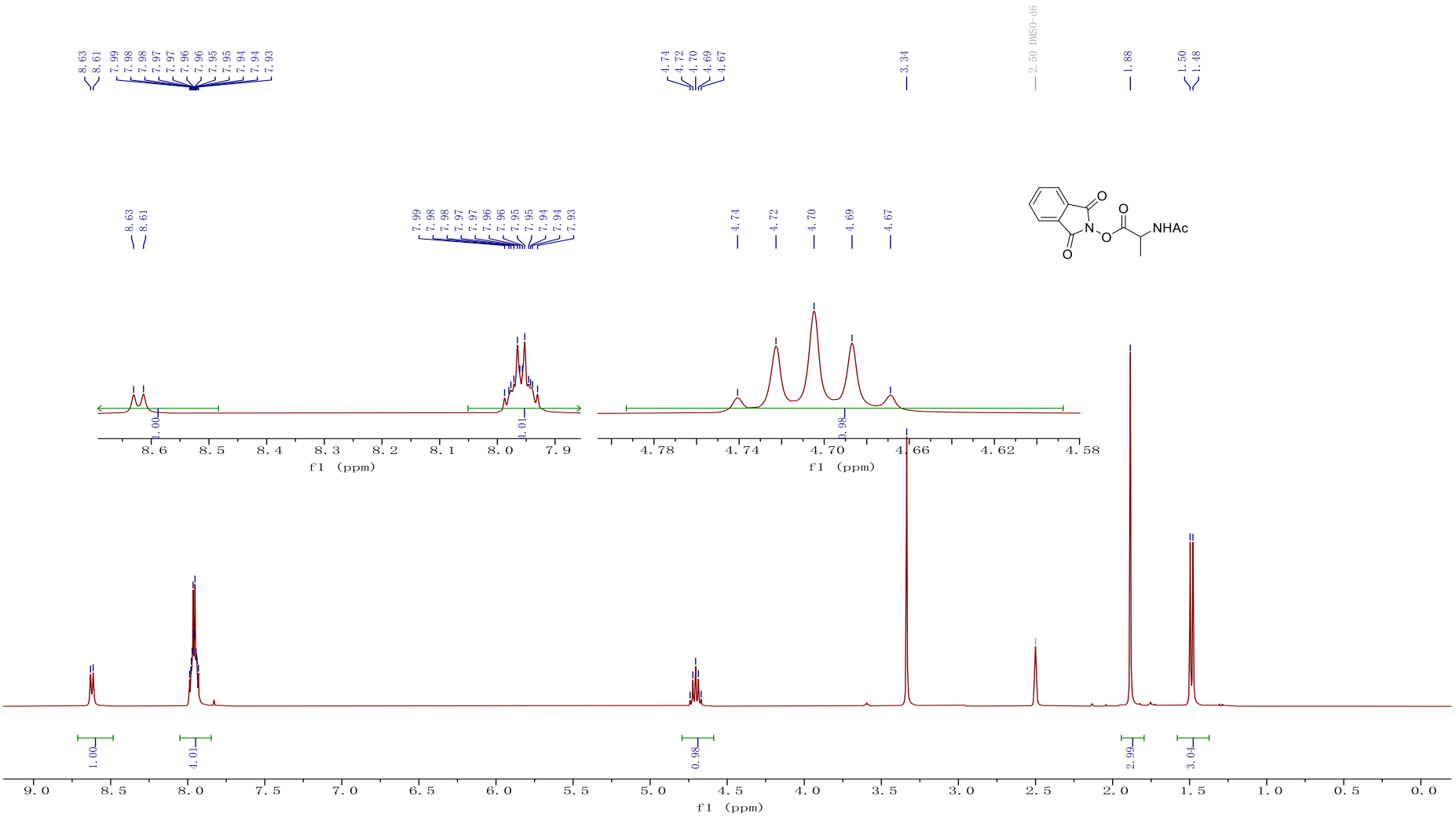
^{1}H NMR (400 MHz, DMSO-d_6) 6-chloro-9*H*-pyrido[3,4-*b*]indole (**11**)



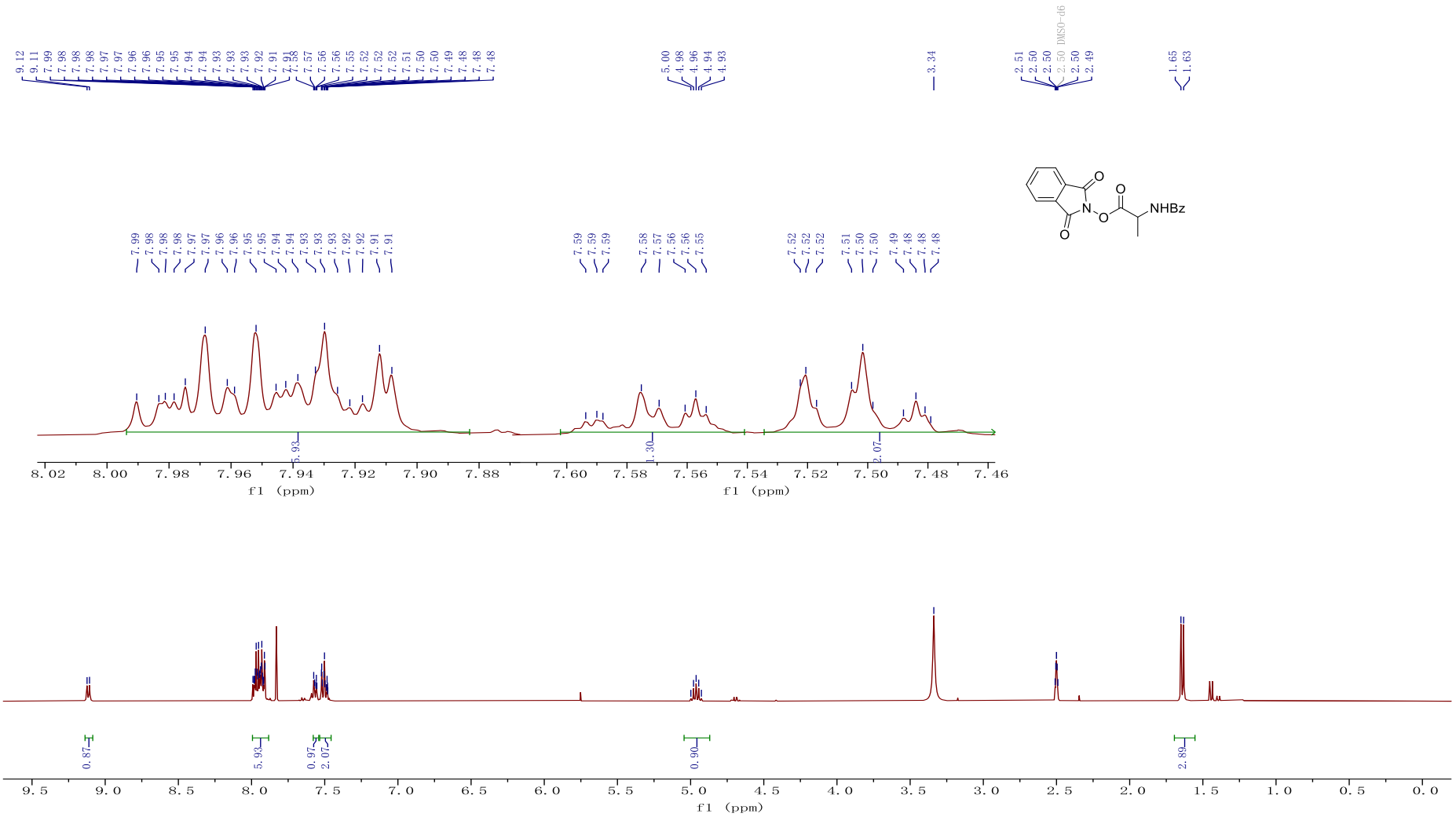
^{1}H NMR (400 MHz, DMSO-d_6) 6-fluoro-9*H*-pyrido[3,4-*b*]indole (**1m**)



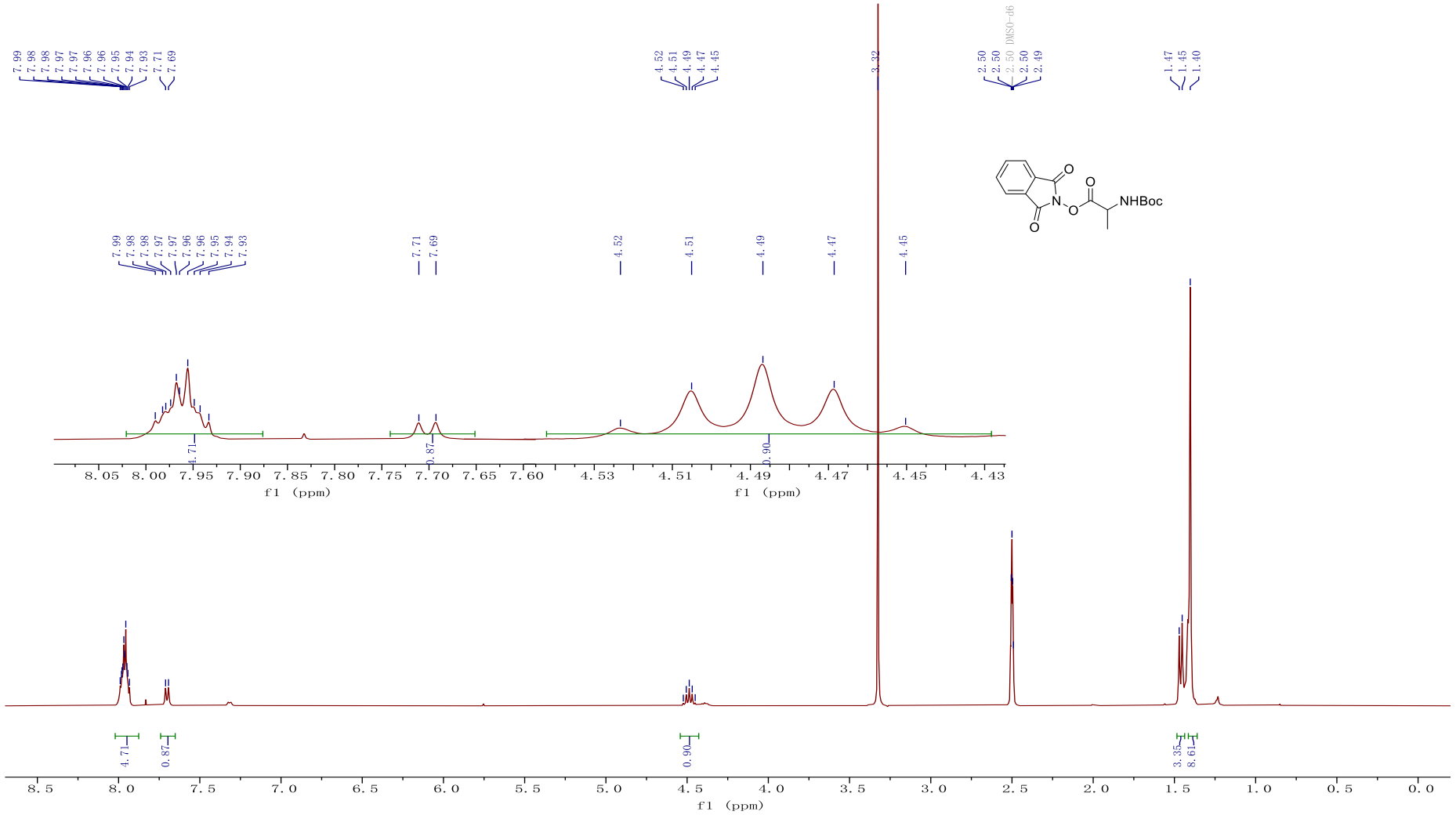
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl acetylalaninate (**2a**)



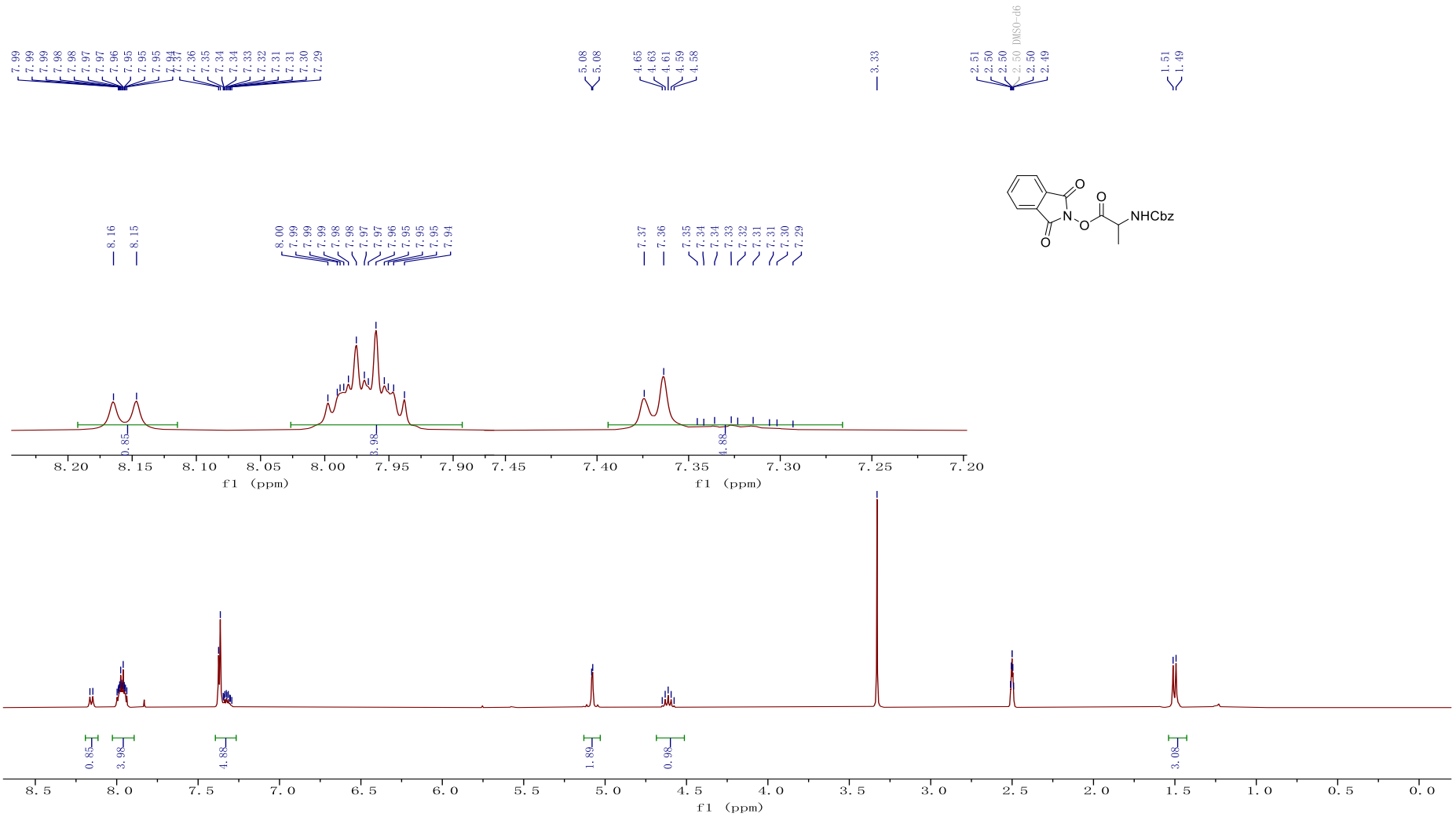
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl benzoylalaninate (**2ab**)



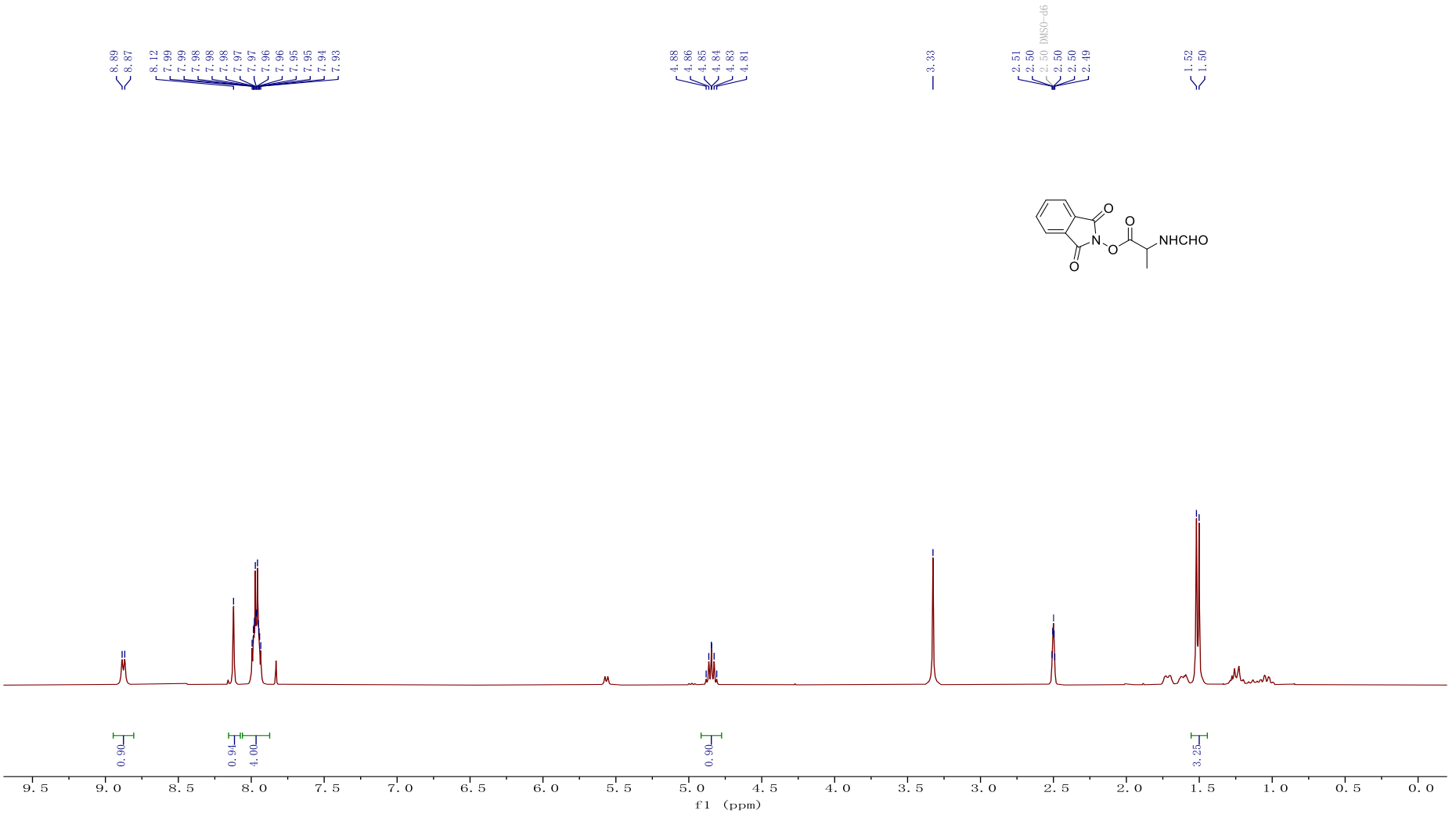
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl (tert-butoxycarbonyl)alaninate (**2ac**)



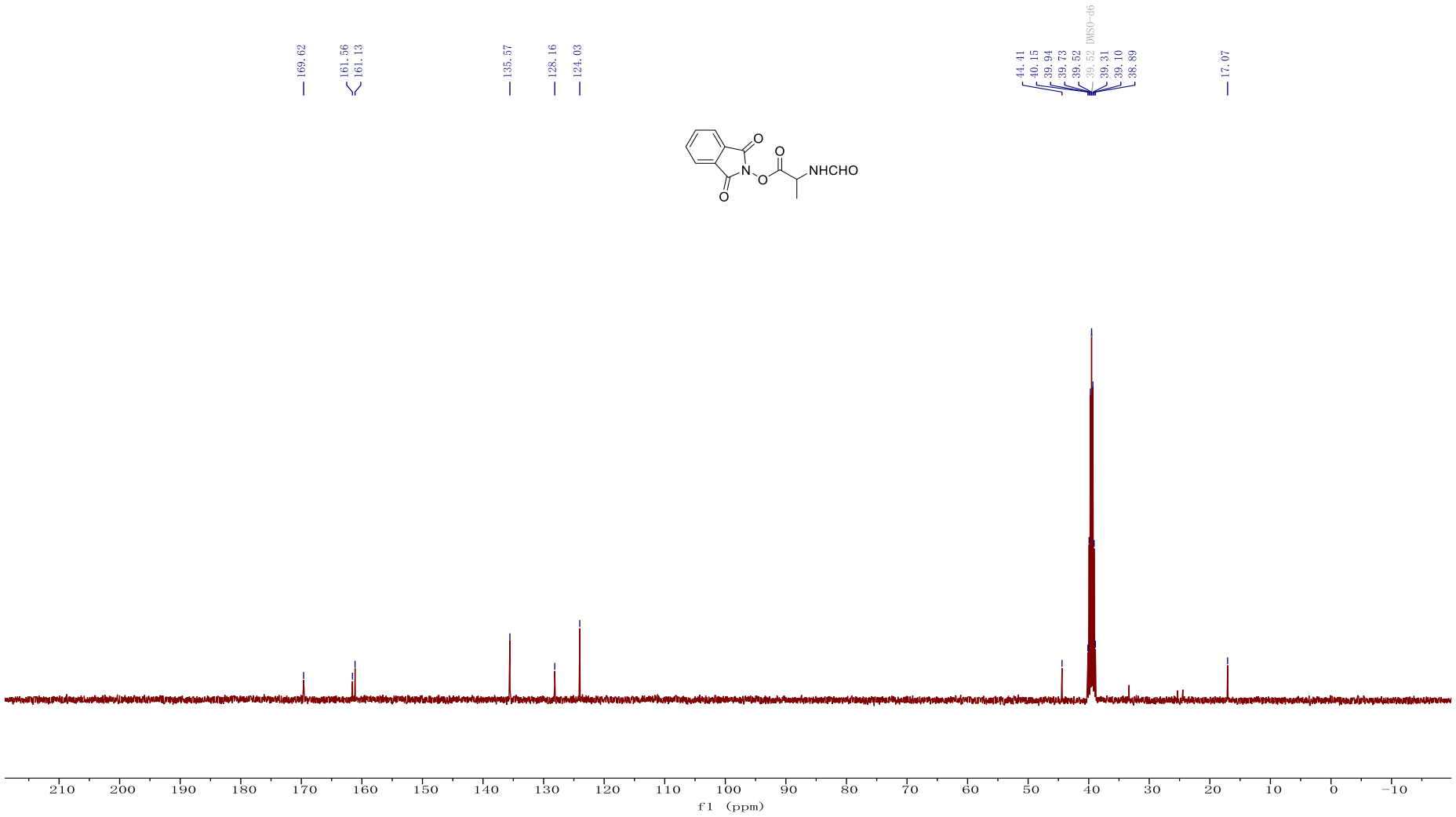
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl ((benzyloxy)carbonyl)alaninate (**2ad**)



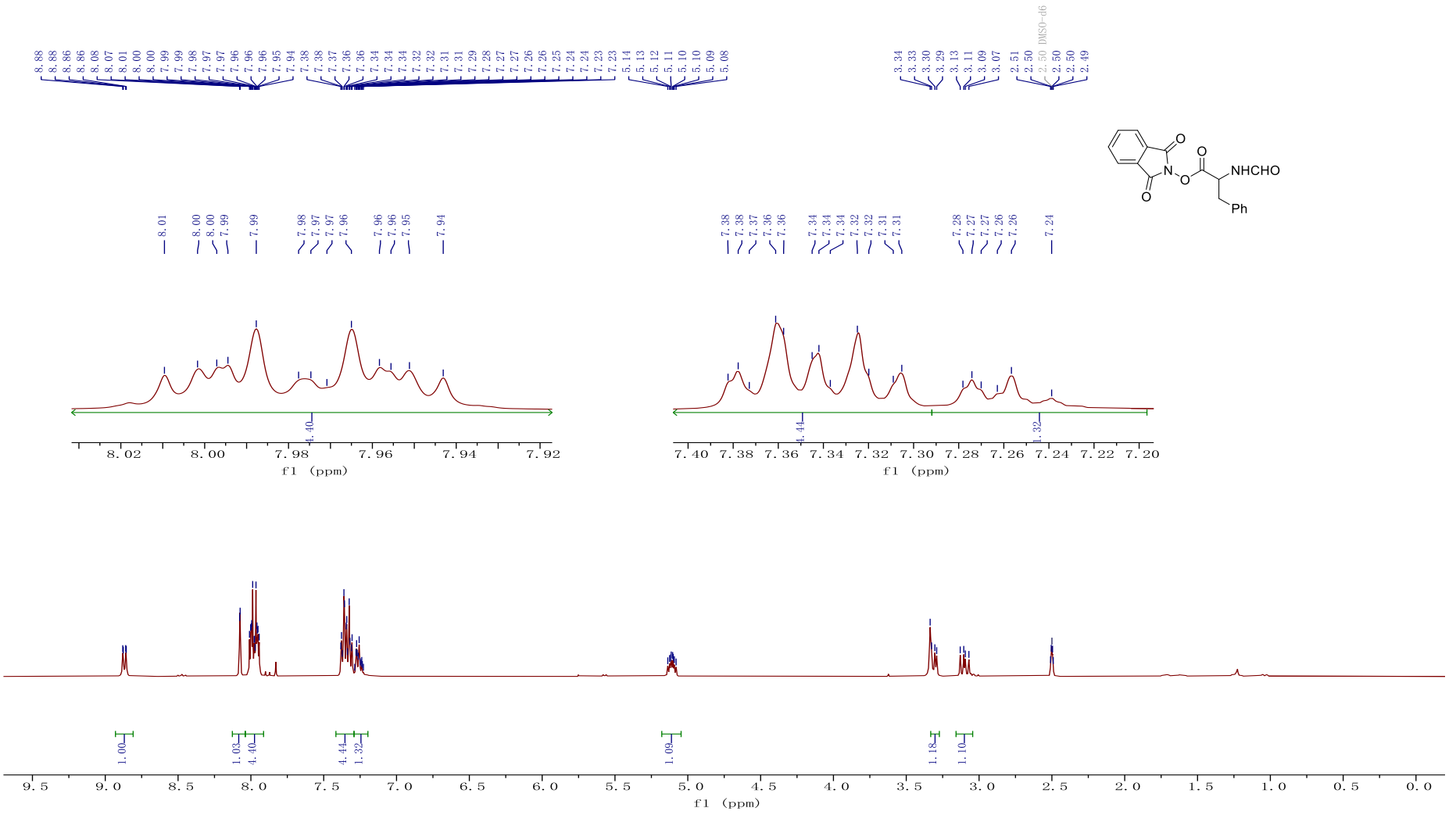
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl formylalaninate (**2ae**)



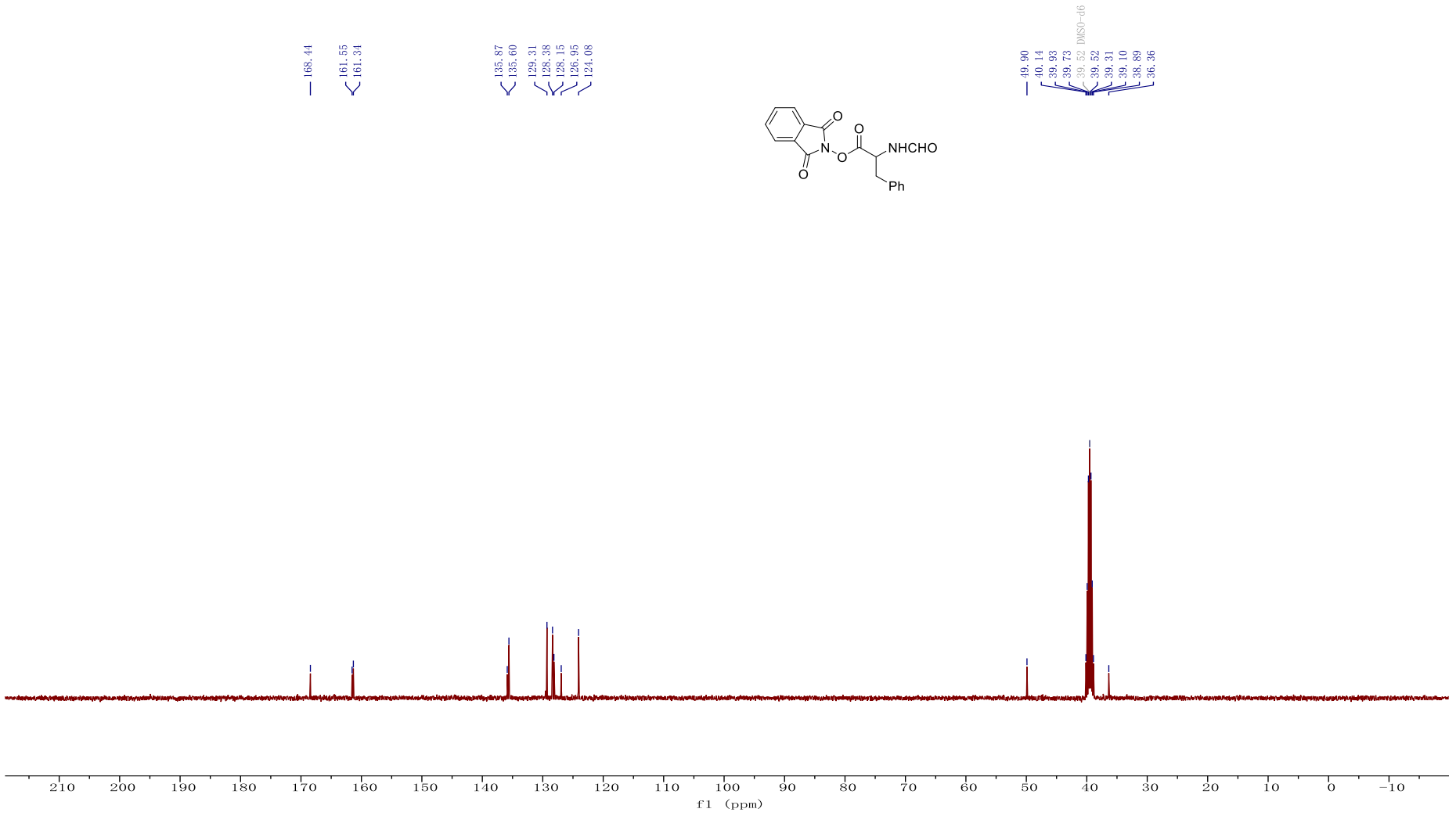
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl formylalaninate (**2ae**)



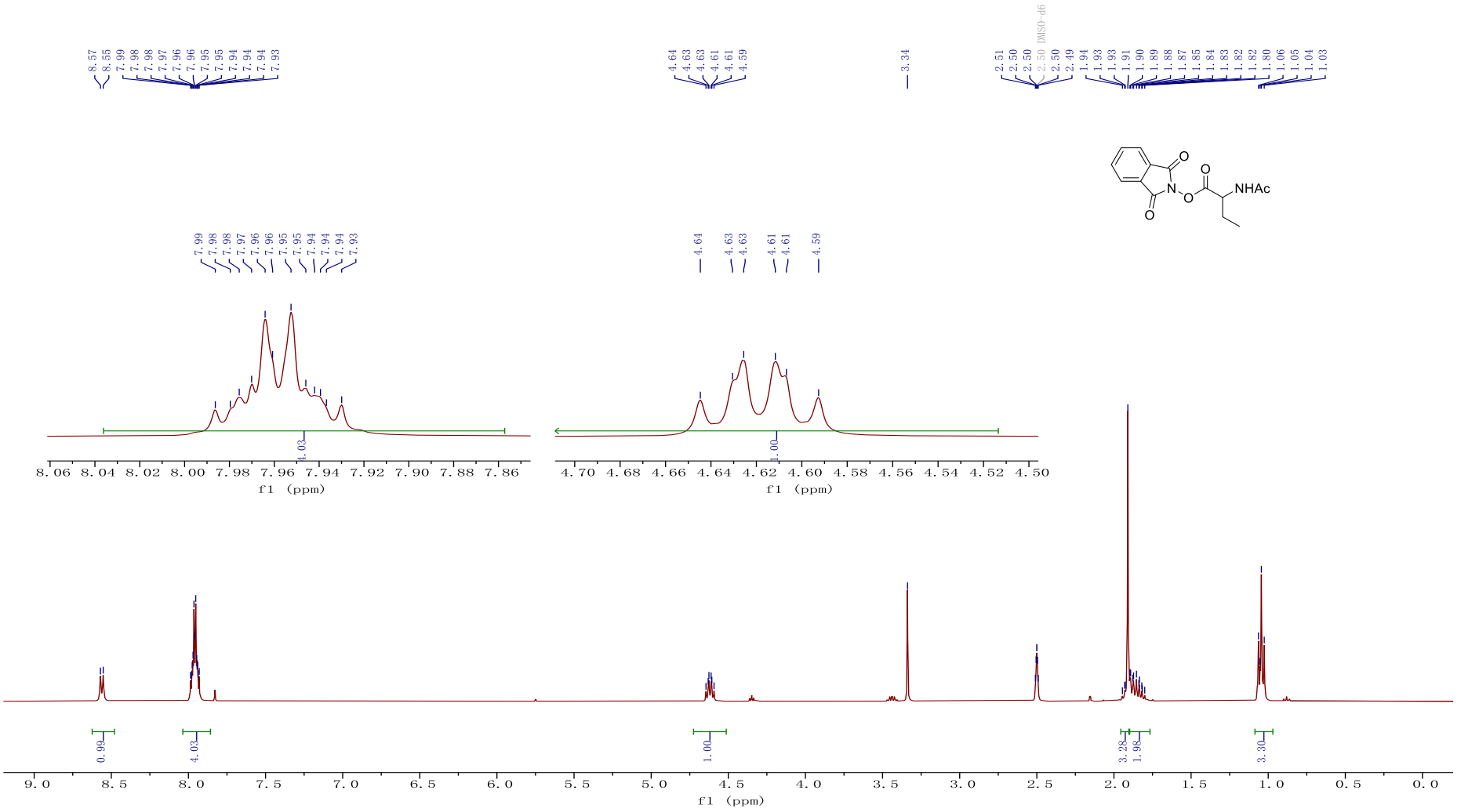
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl formylphenylalaninate (**2b**)



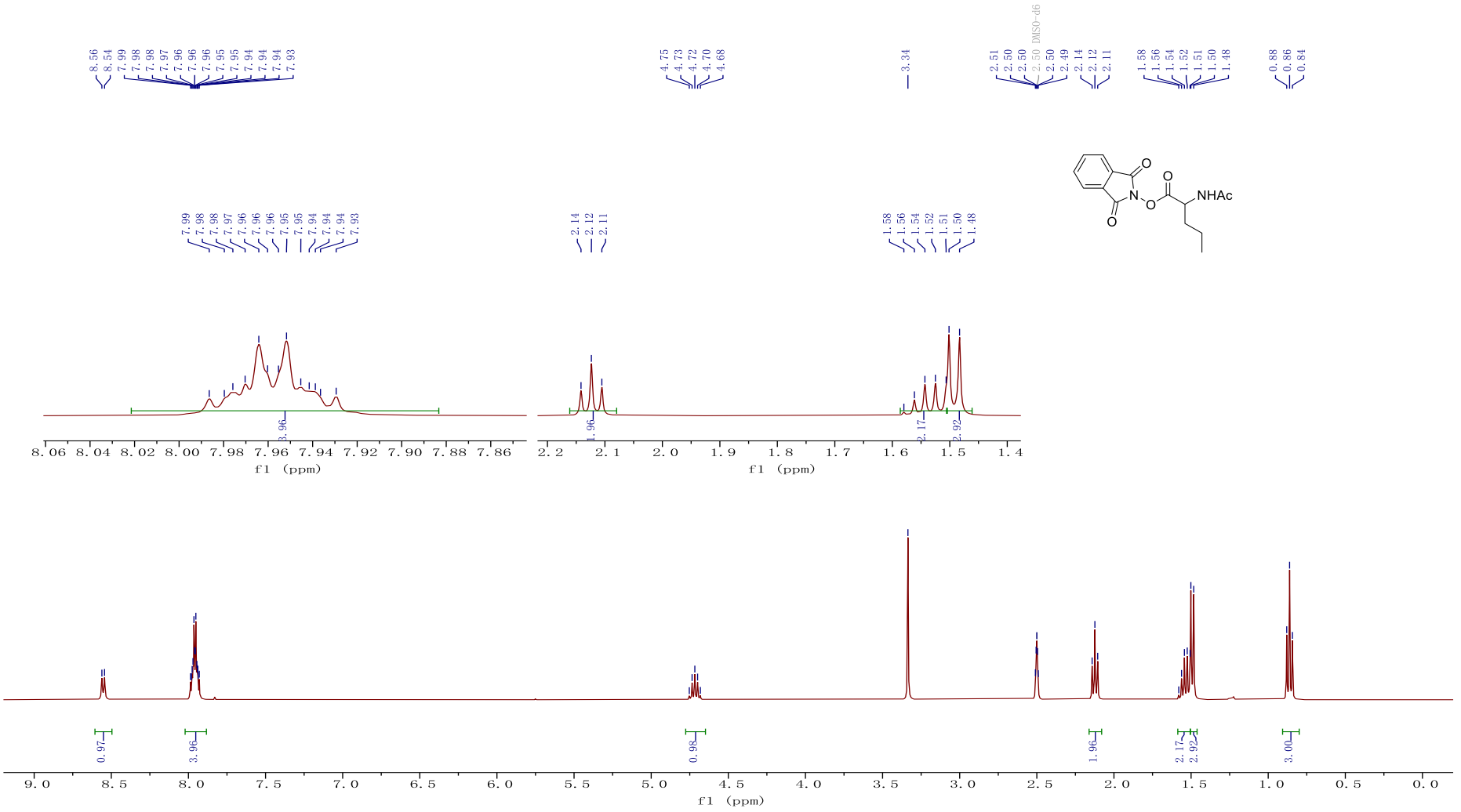
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl formylphenylalaninate (**2b**)



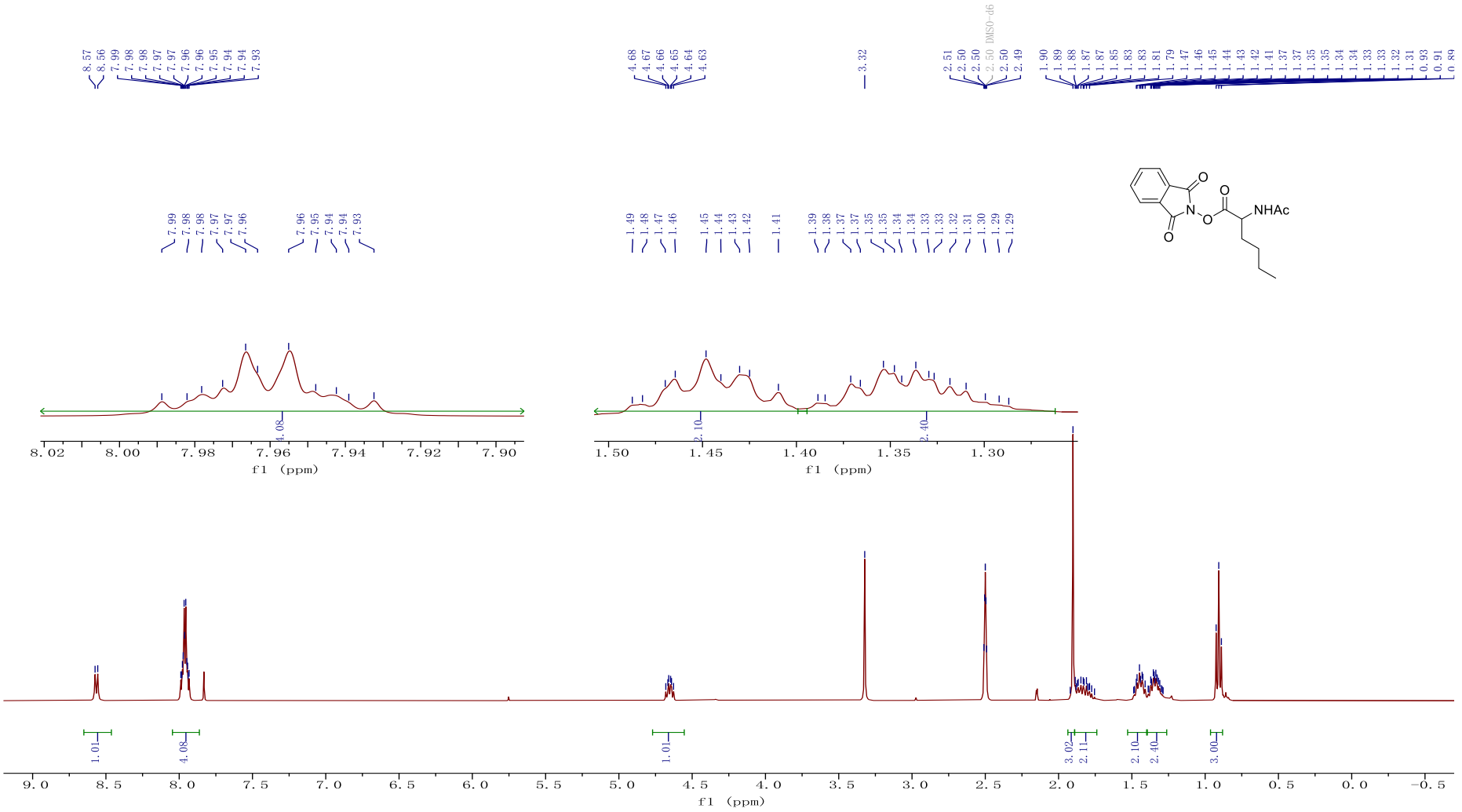
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidobutanoate (**4a**)



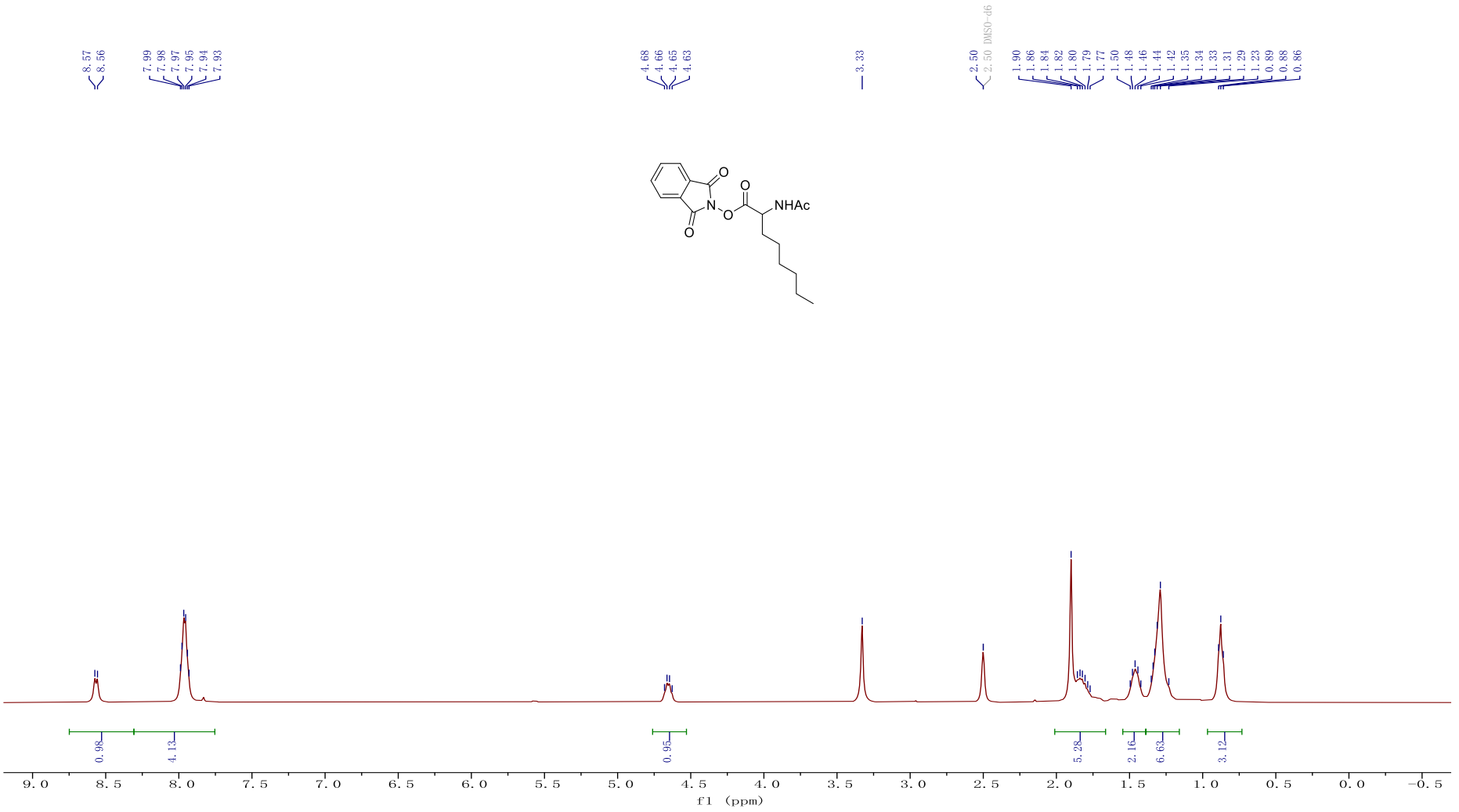
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidopentanoate (**4b**)



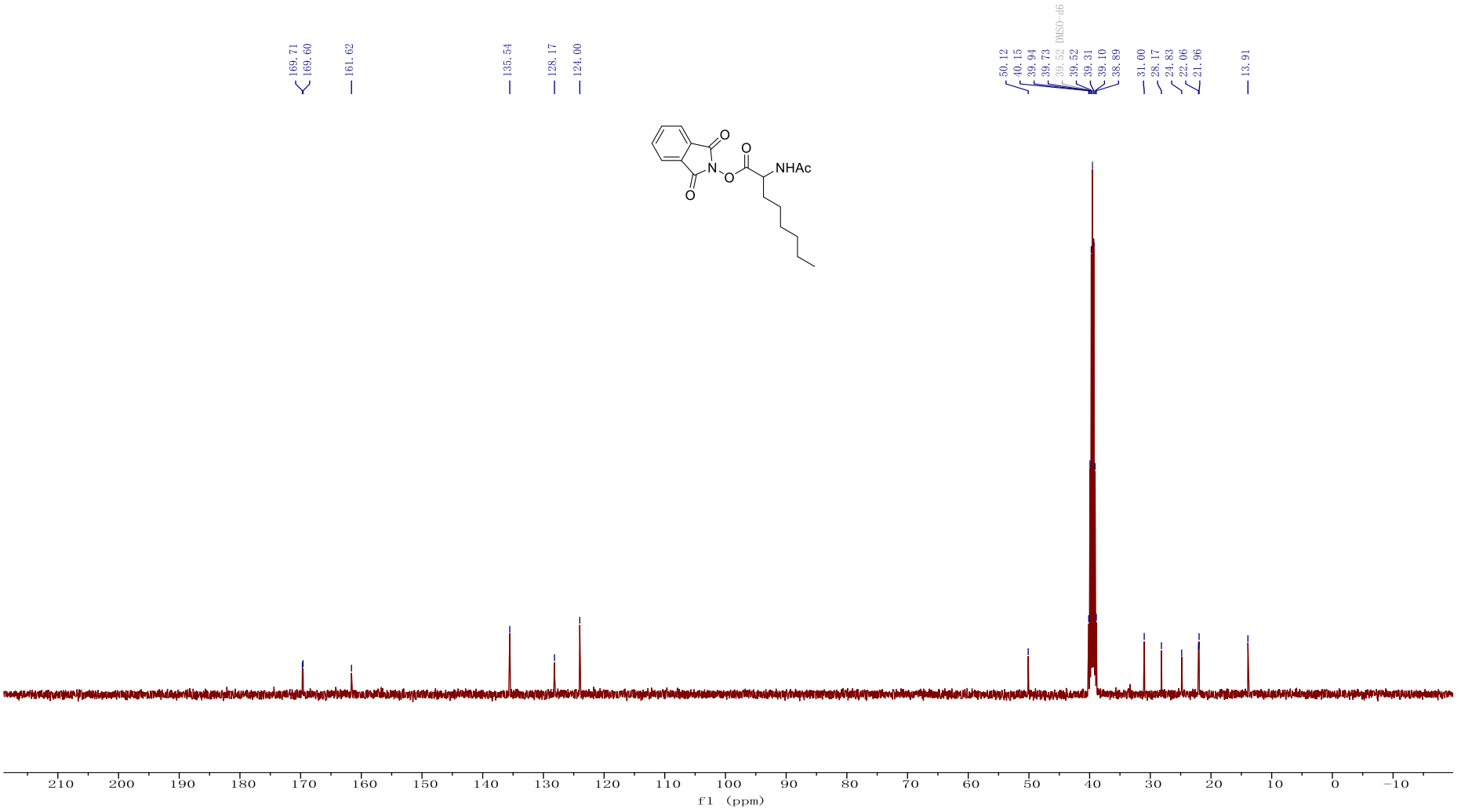
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidohexanoate (**4c**)



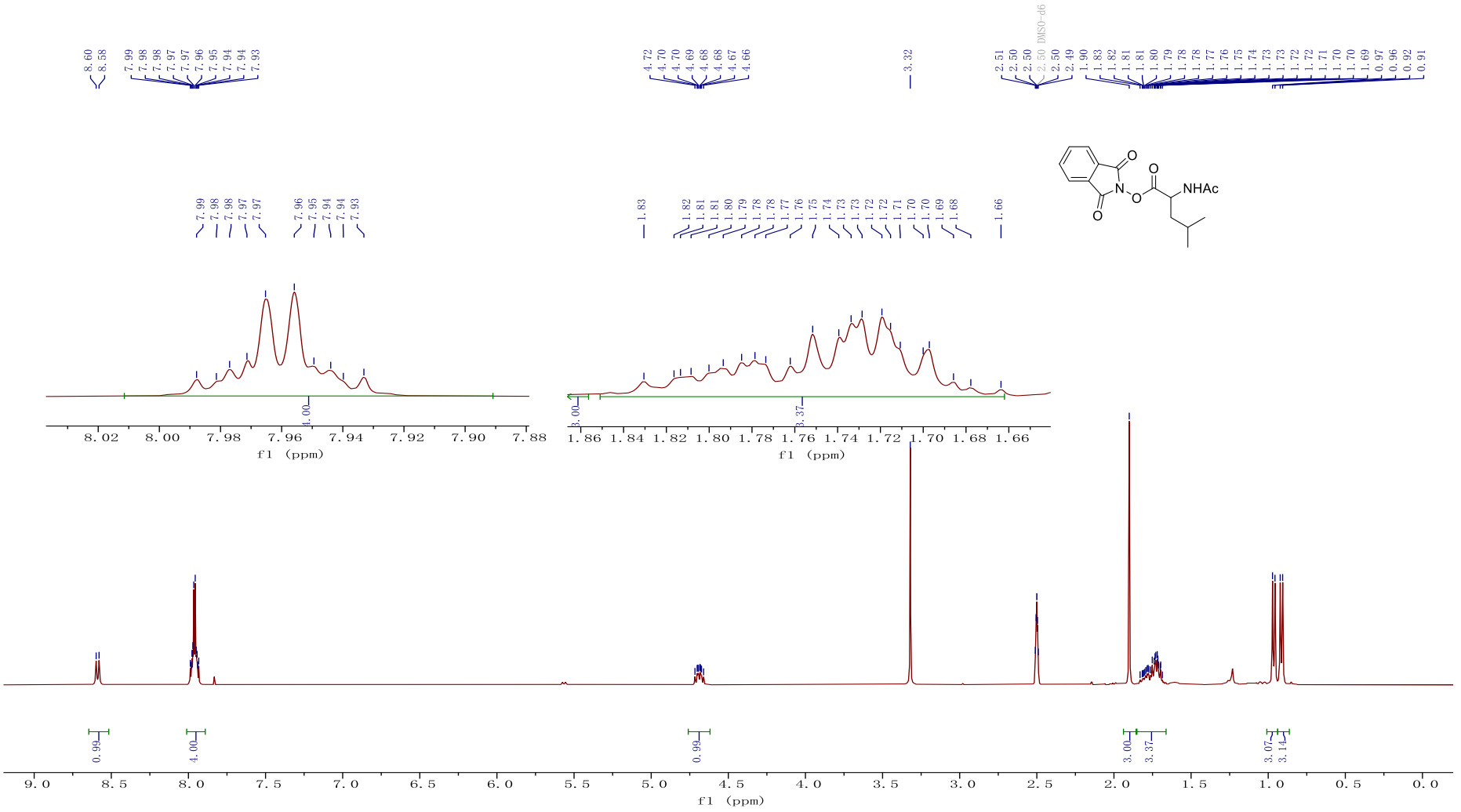
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidooctanoate (**4d**)



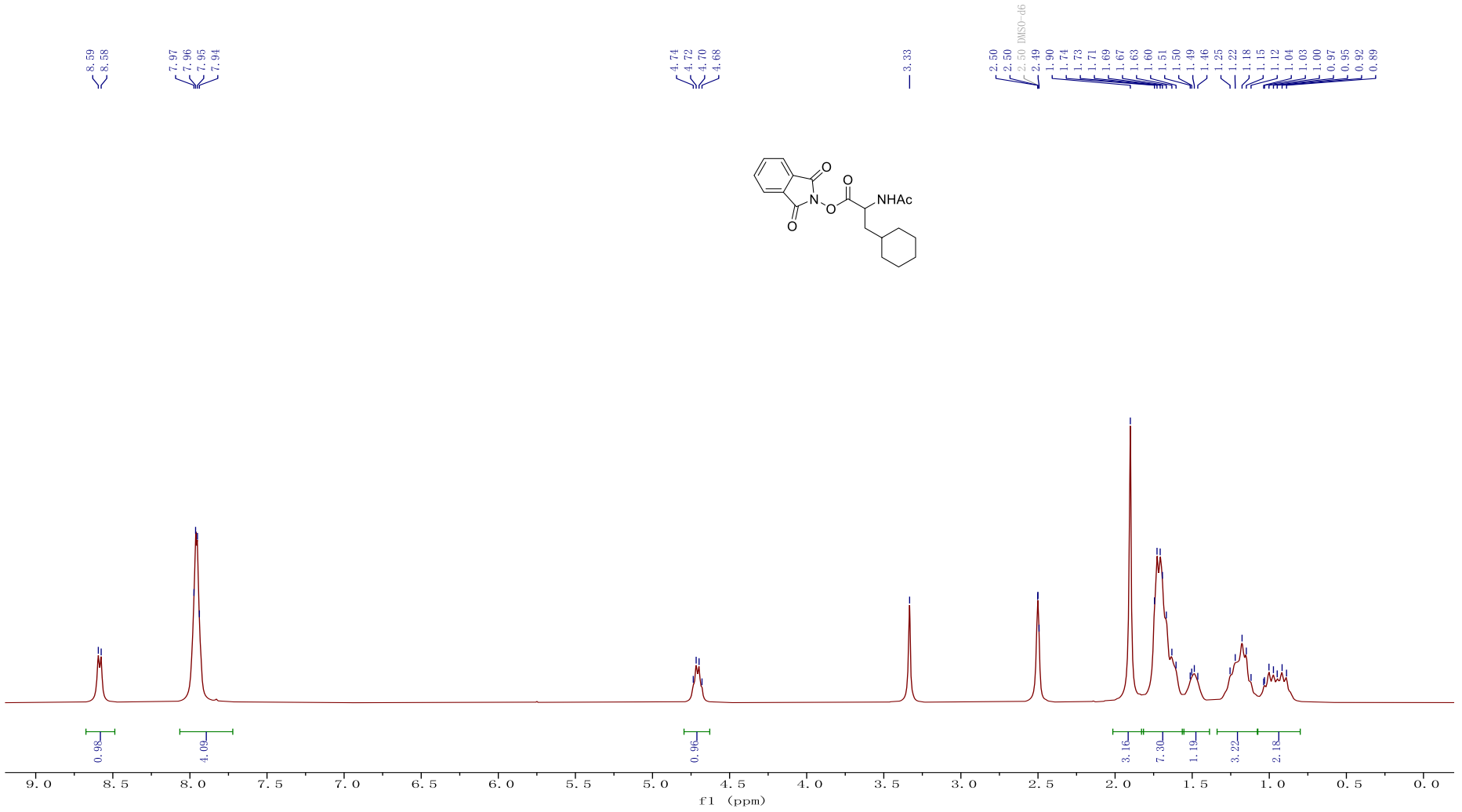
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidooctanoate (**4d**)



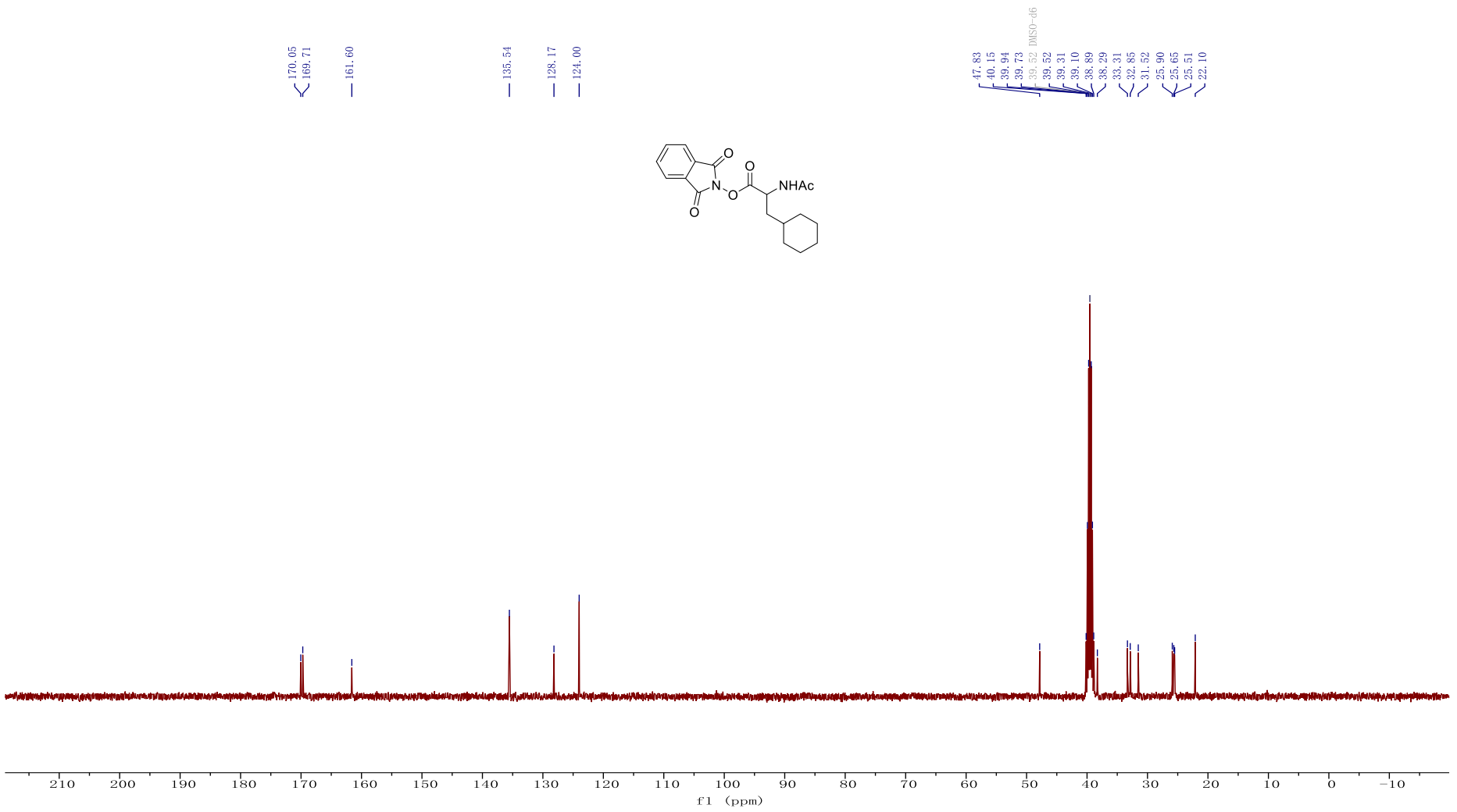
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl acetylleucinate (**4e**)



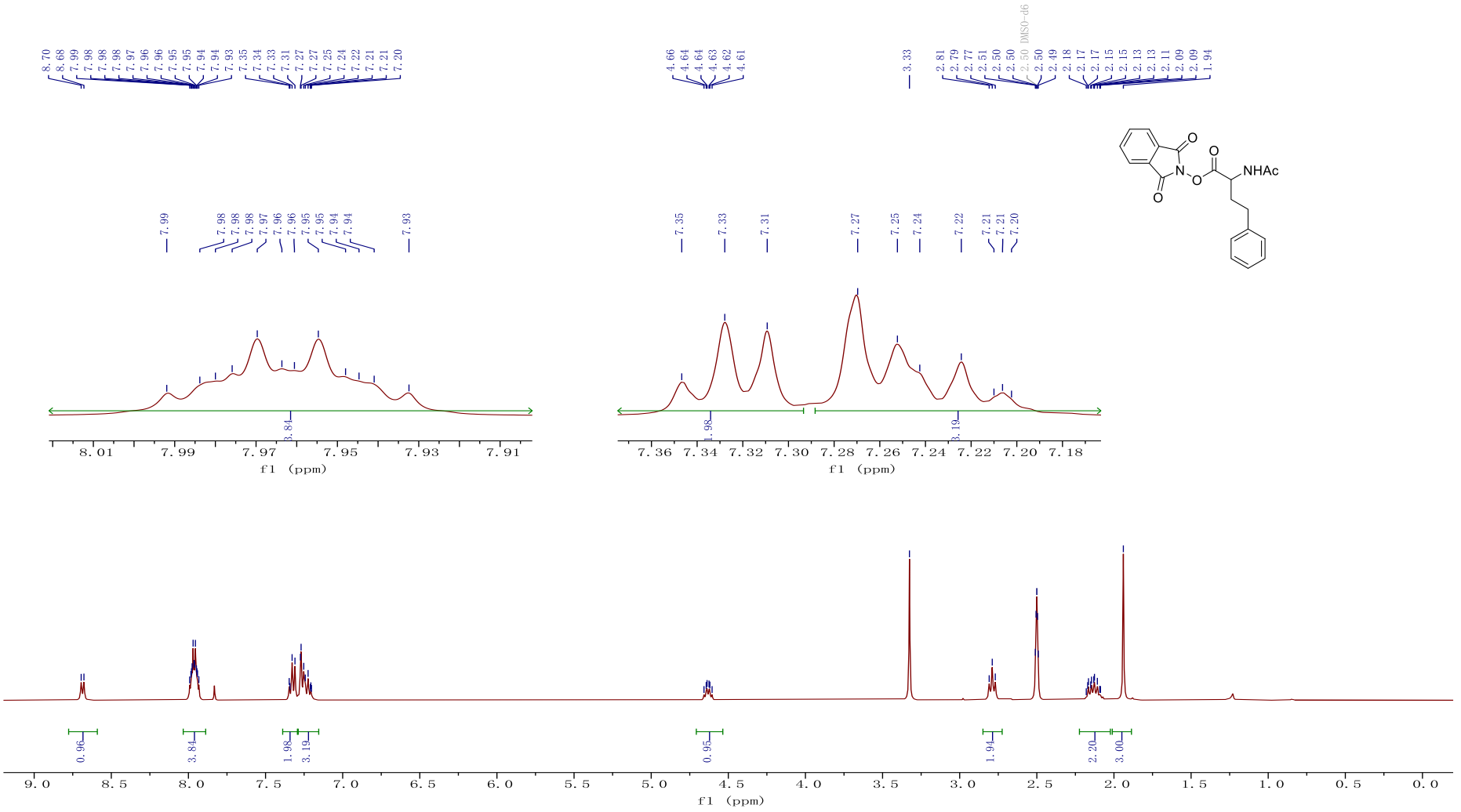
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-cyclohexylpropanoate (**4f**)



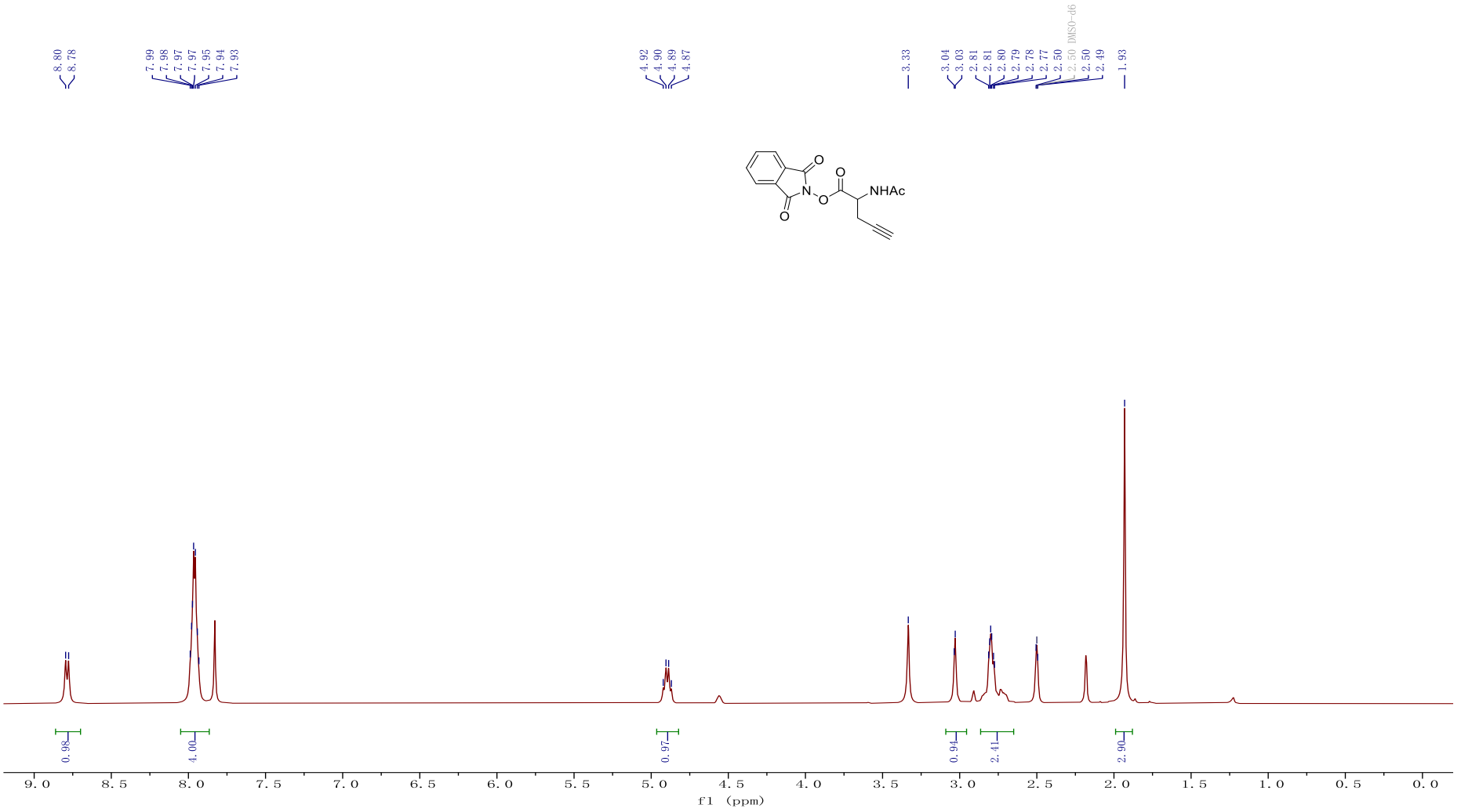
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-cyclohexylpropanoate (**4f**)



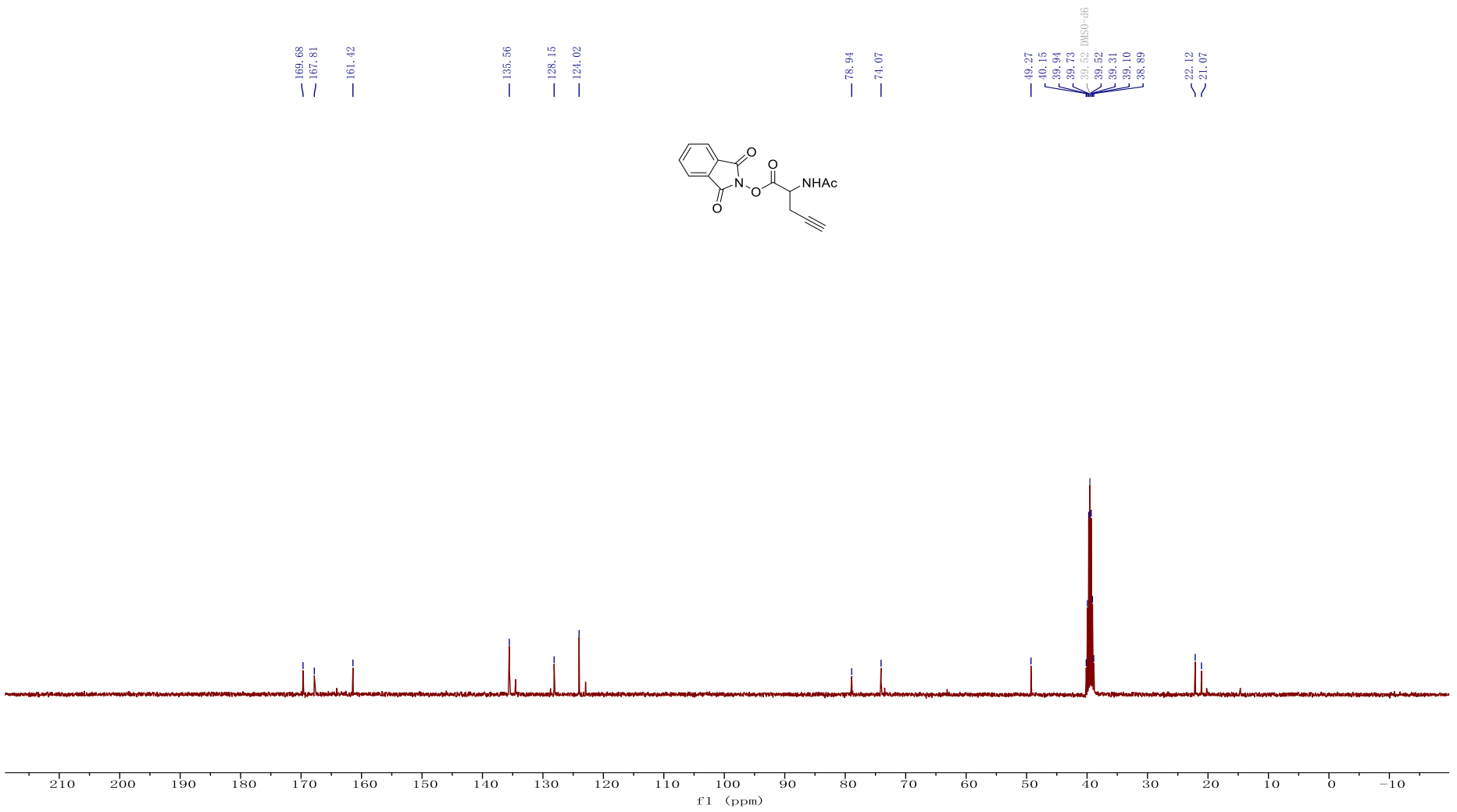
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-4-phenylbutanoate (**4g**)



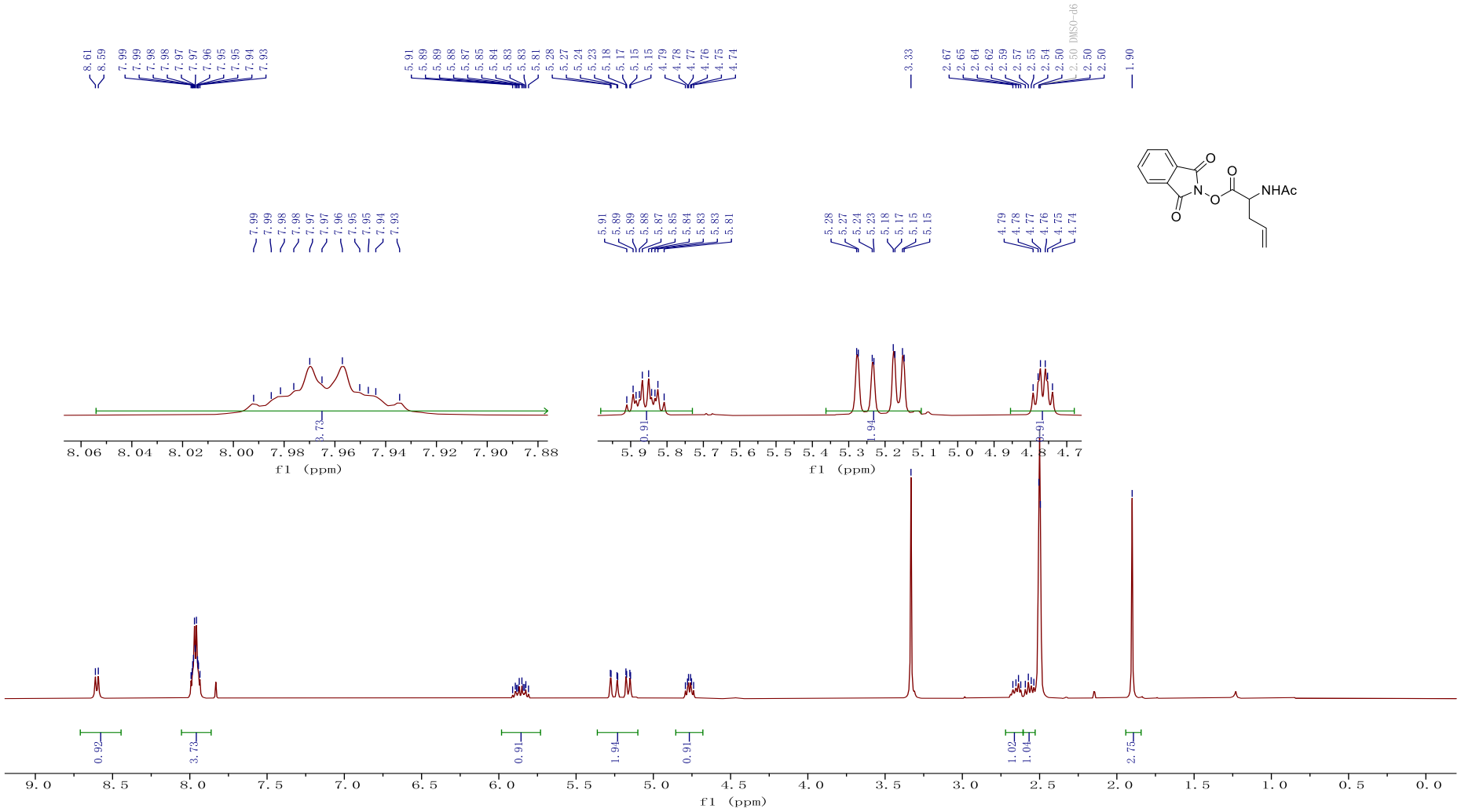
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-ynoate (**4h**)



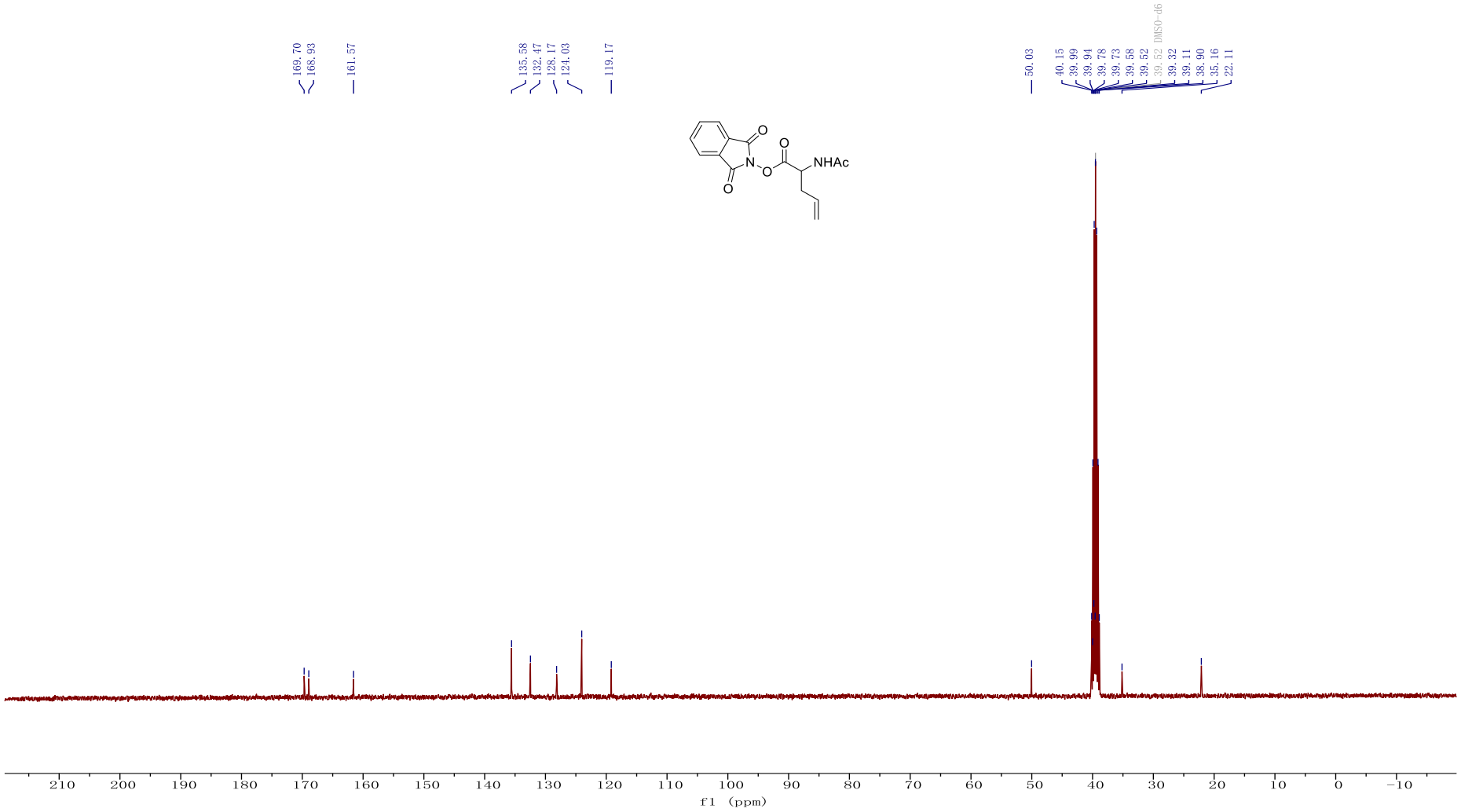
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-ynoate (**4h**)



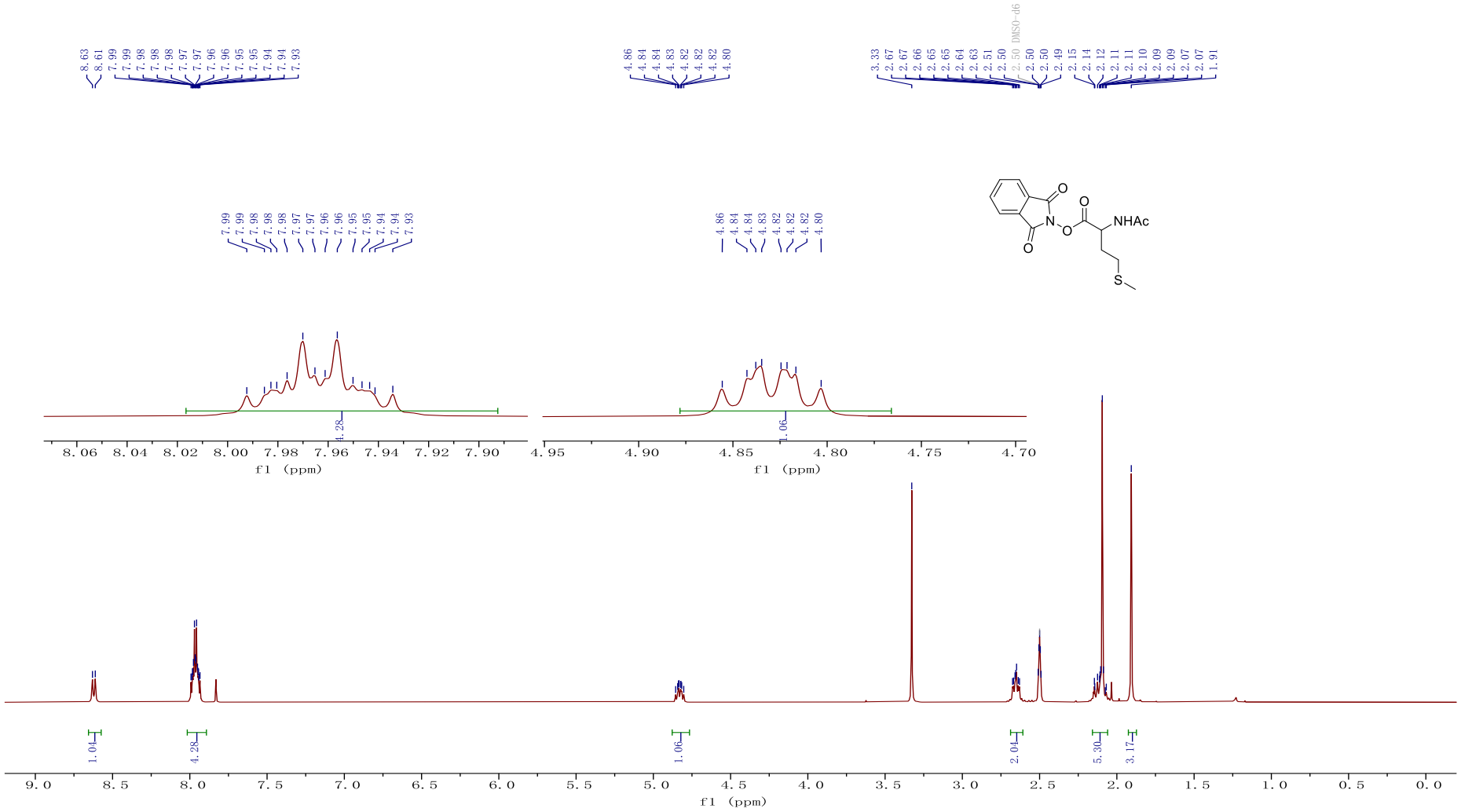
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-enoate (**4i**)



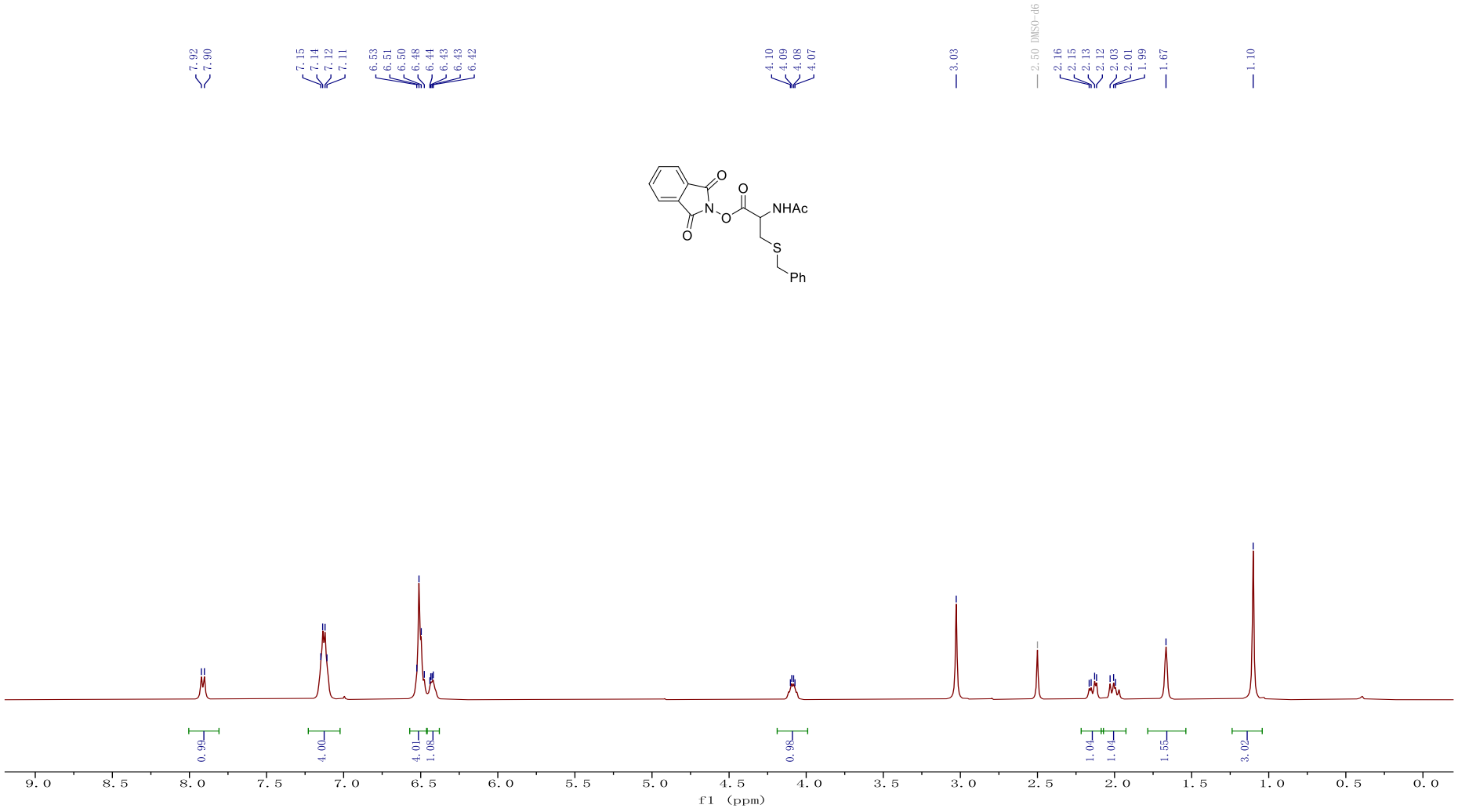
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamidopent-4-enoate (**4i**)



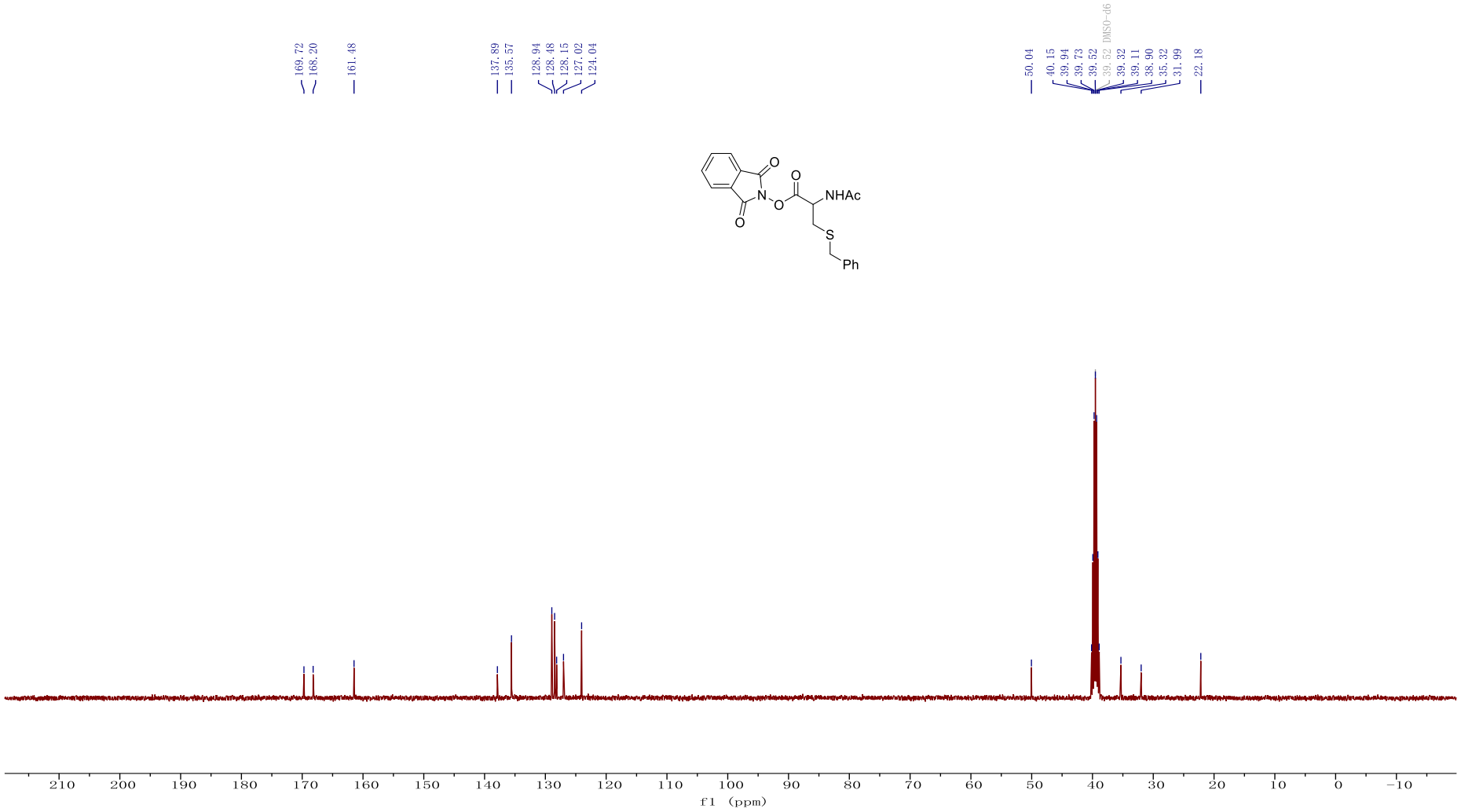
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl acetylmethioninate (**4j**)



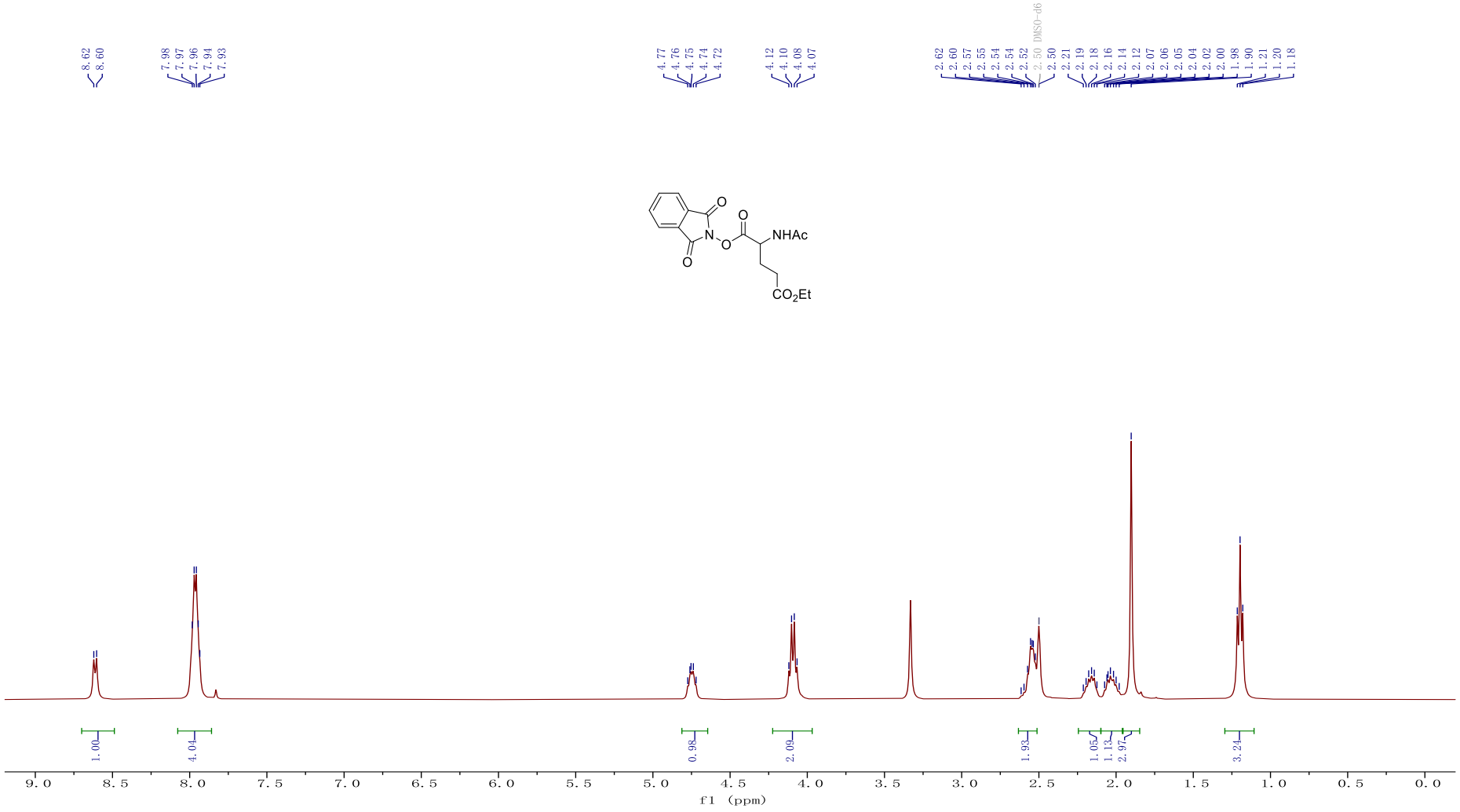
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl *N*-acetyl-*S*-benzylcysteinate (**4k**)



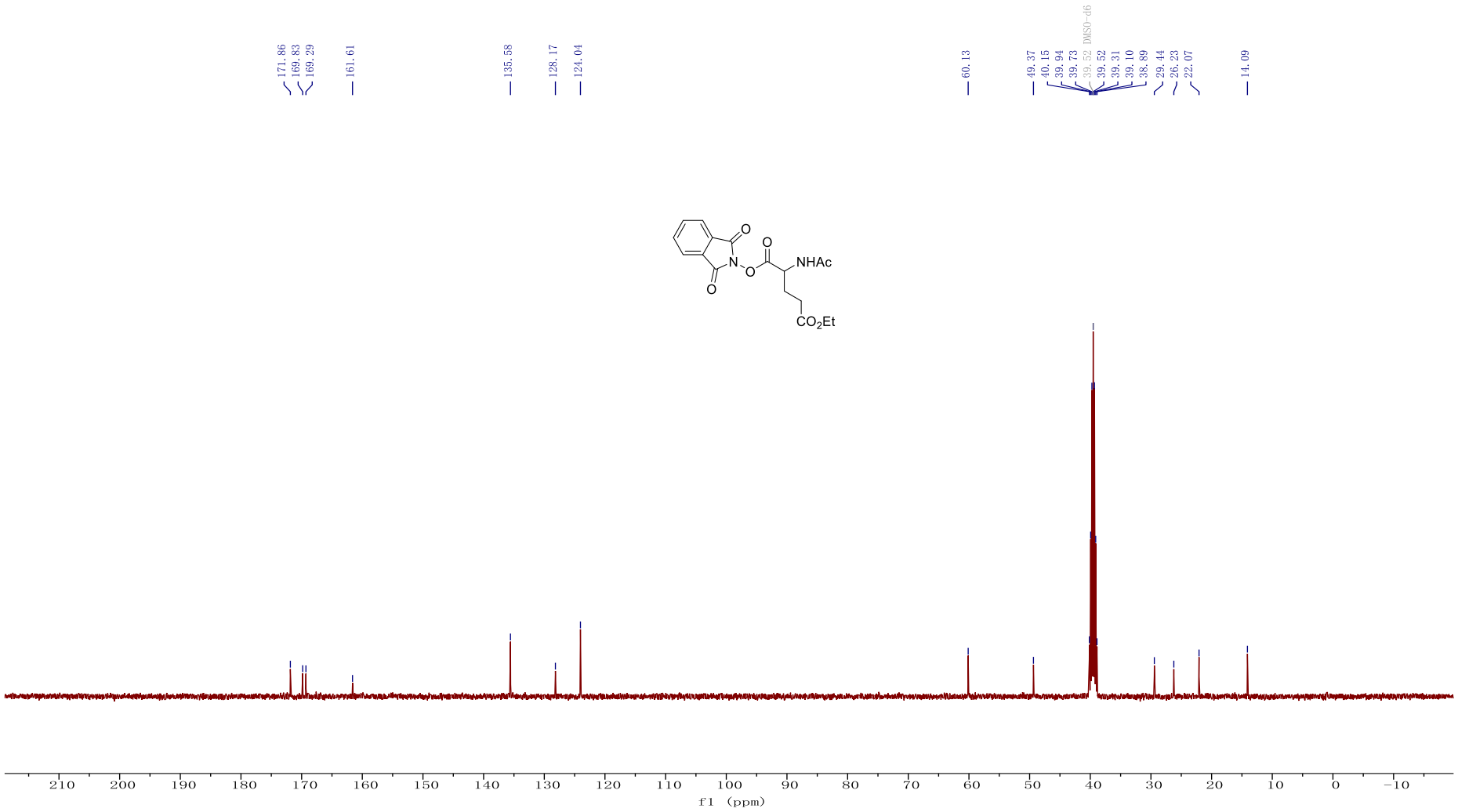
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl *N*-acetyl-*S*-benzylcysteinate (**4k**)



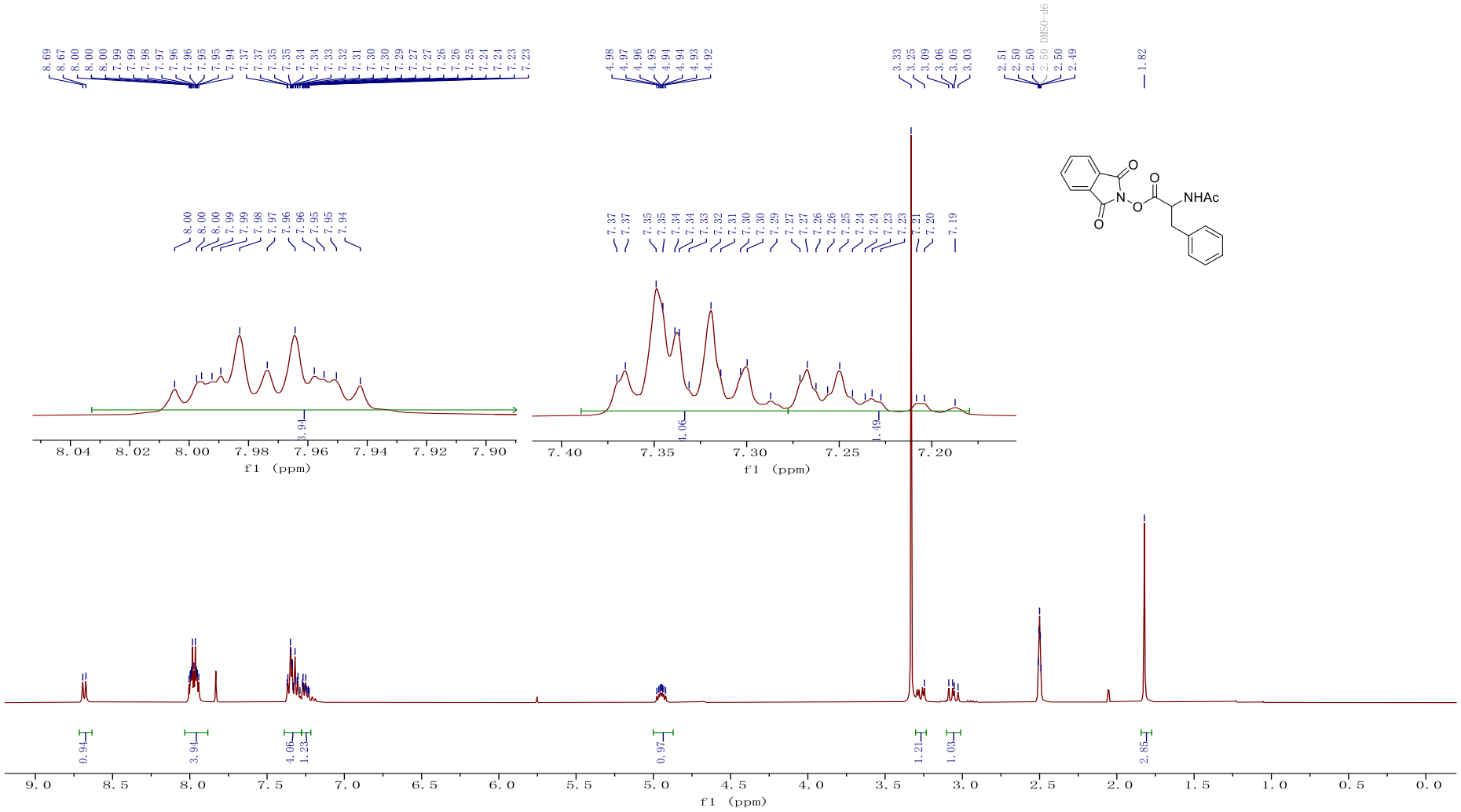
^{1}H NMR (400 MHz, DMSO-d_6) 1-(1,3-dioxoisoindolin-2-yl) 5-ethyl acetylglutamate (**4l**)



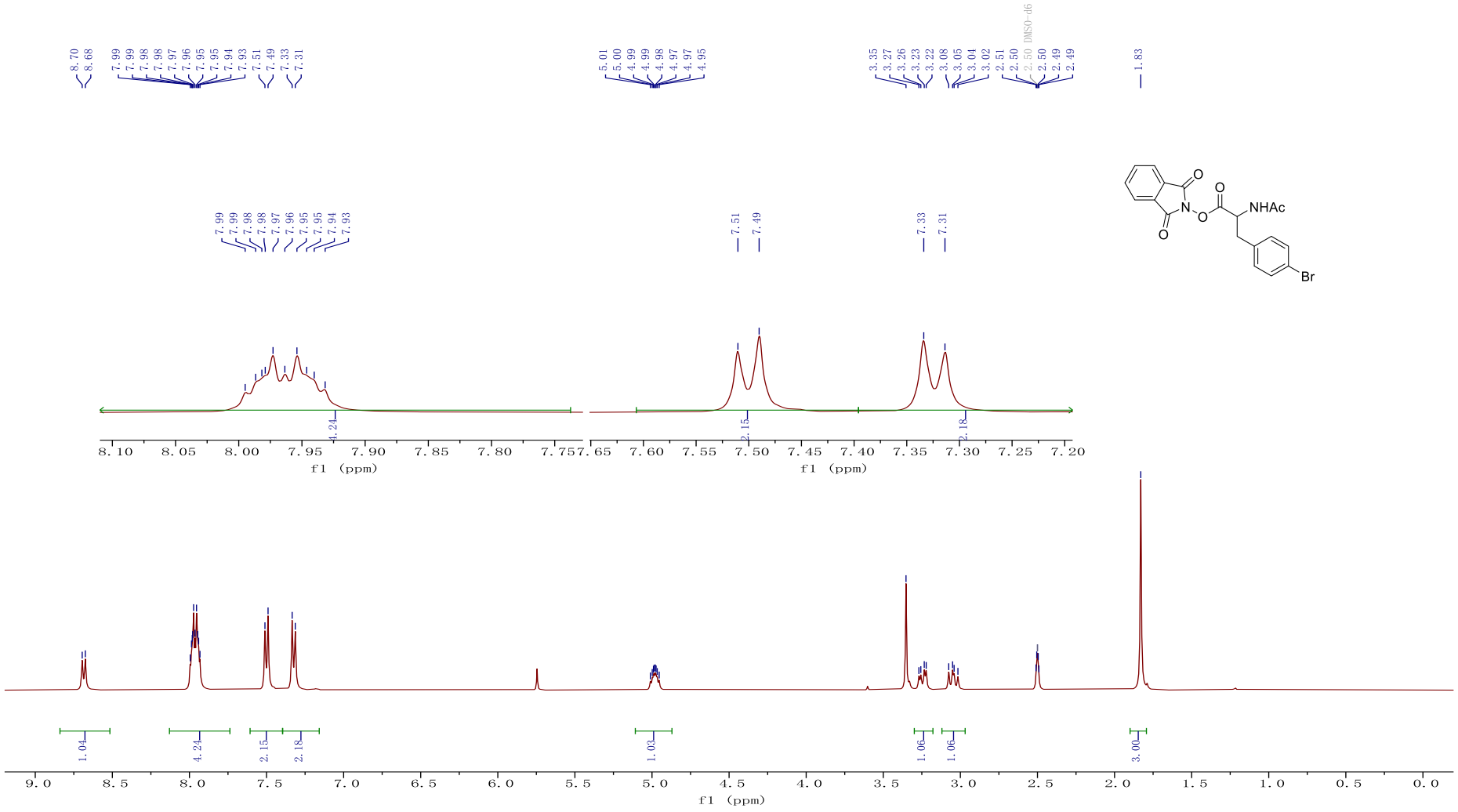
^{13}C NMR (101 MHz, DMSO-d_6) 1-(1,3-dioxoisoindolin-2-yl) 5-ethyl acetylglutamate (**4l**)



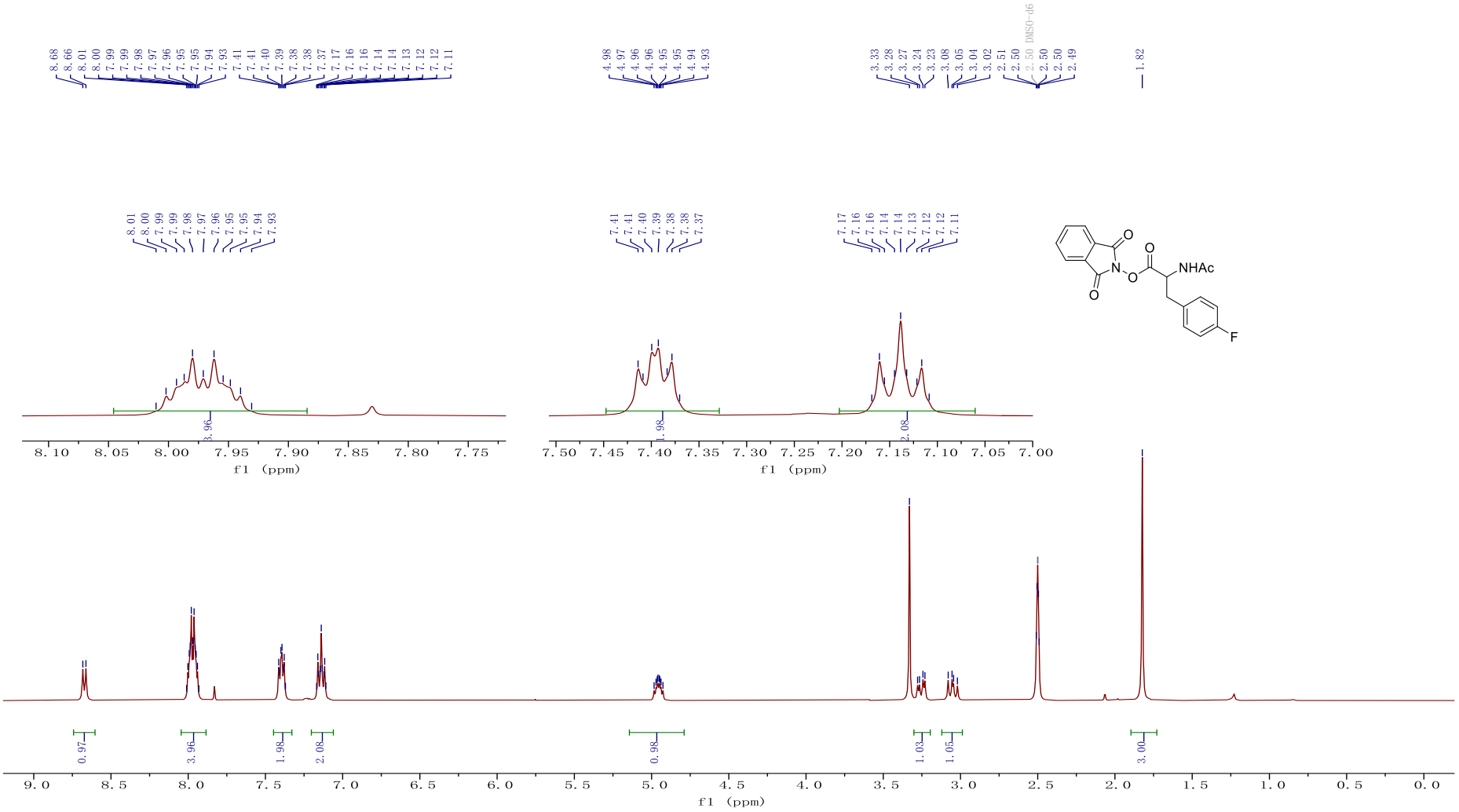
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl acetylphenylalaninate (**4m**)



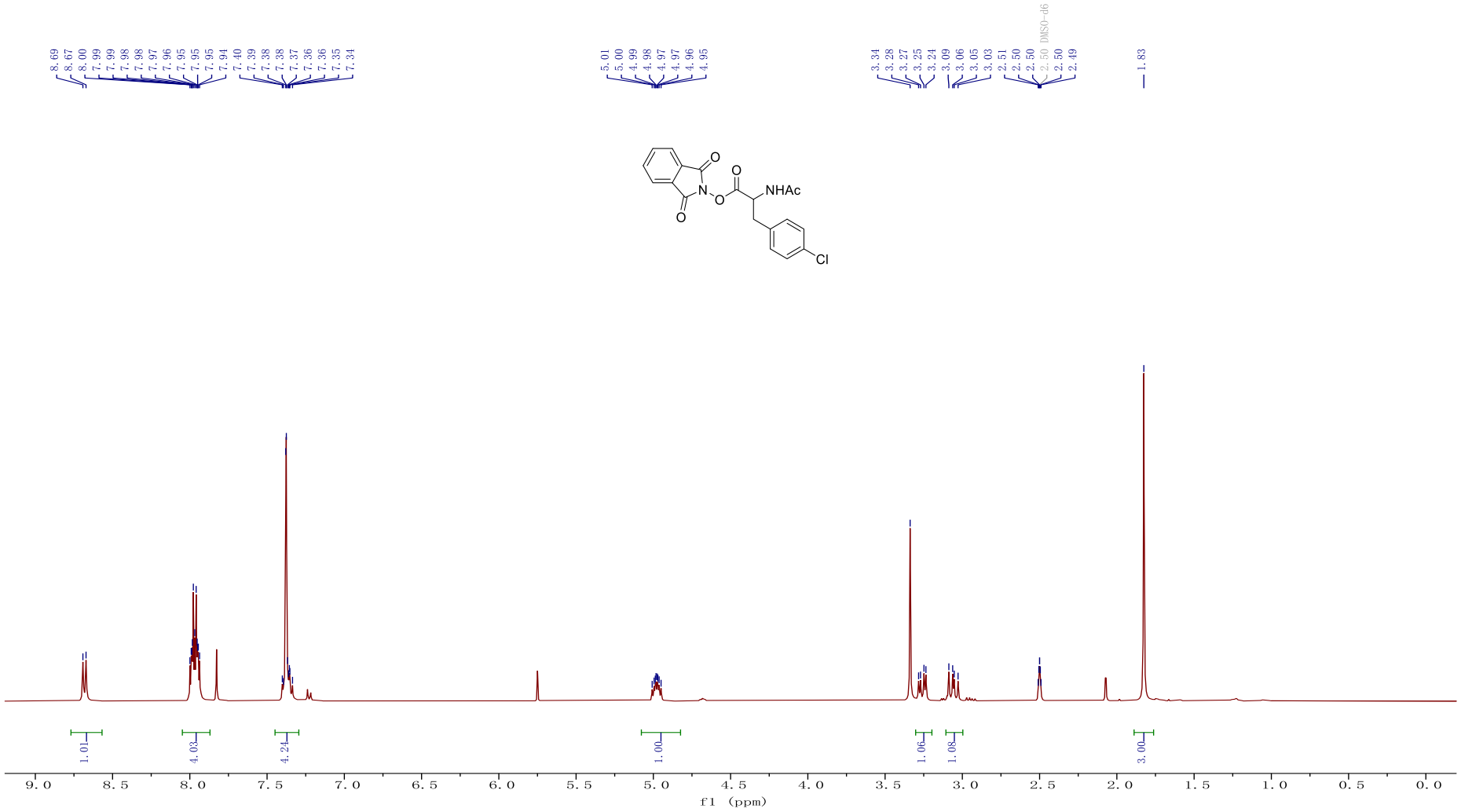
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-bromophenyl)propanoate (**4n**)



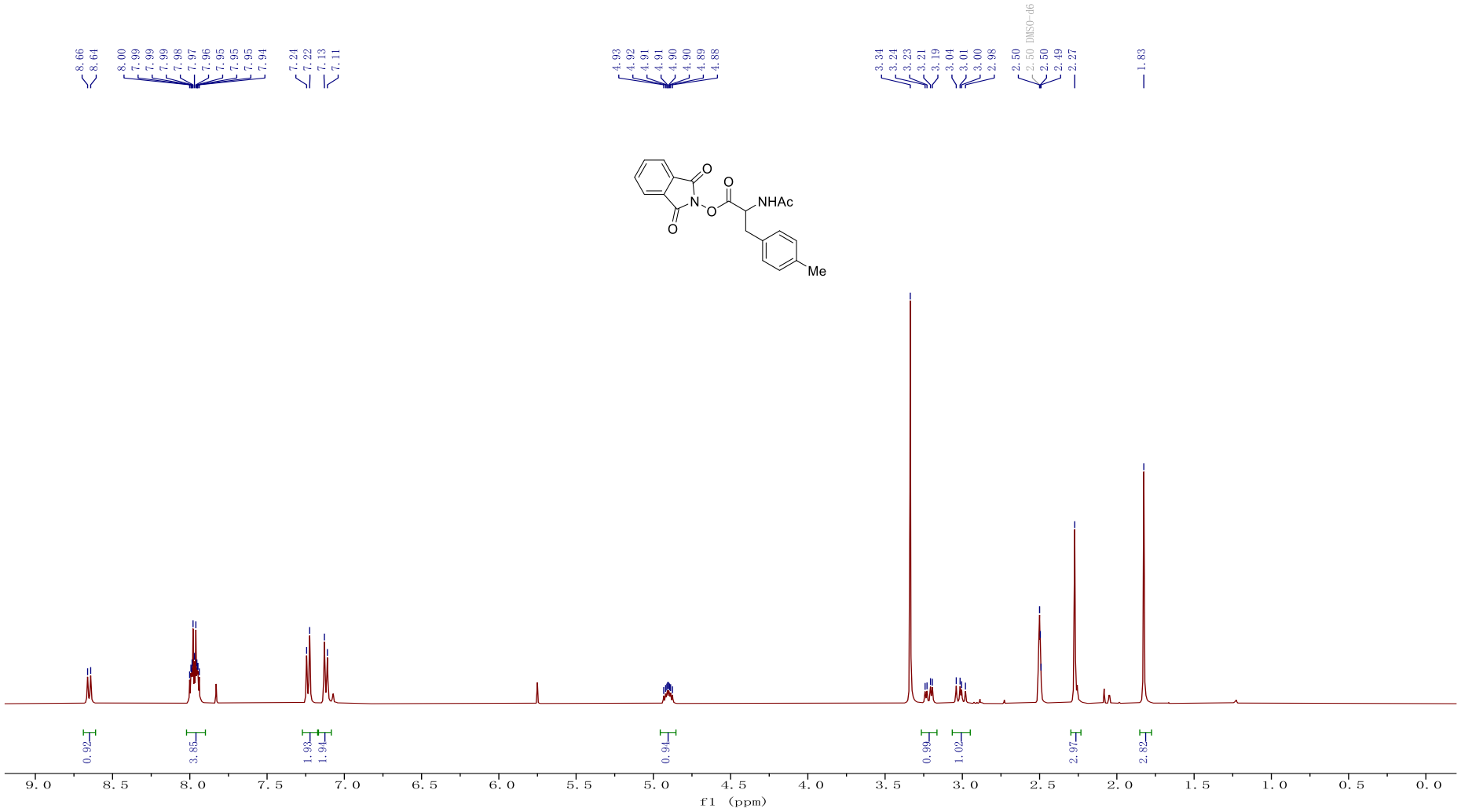
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-fluorophenyl)propanoate (**4o**)



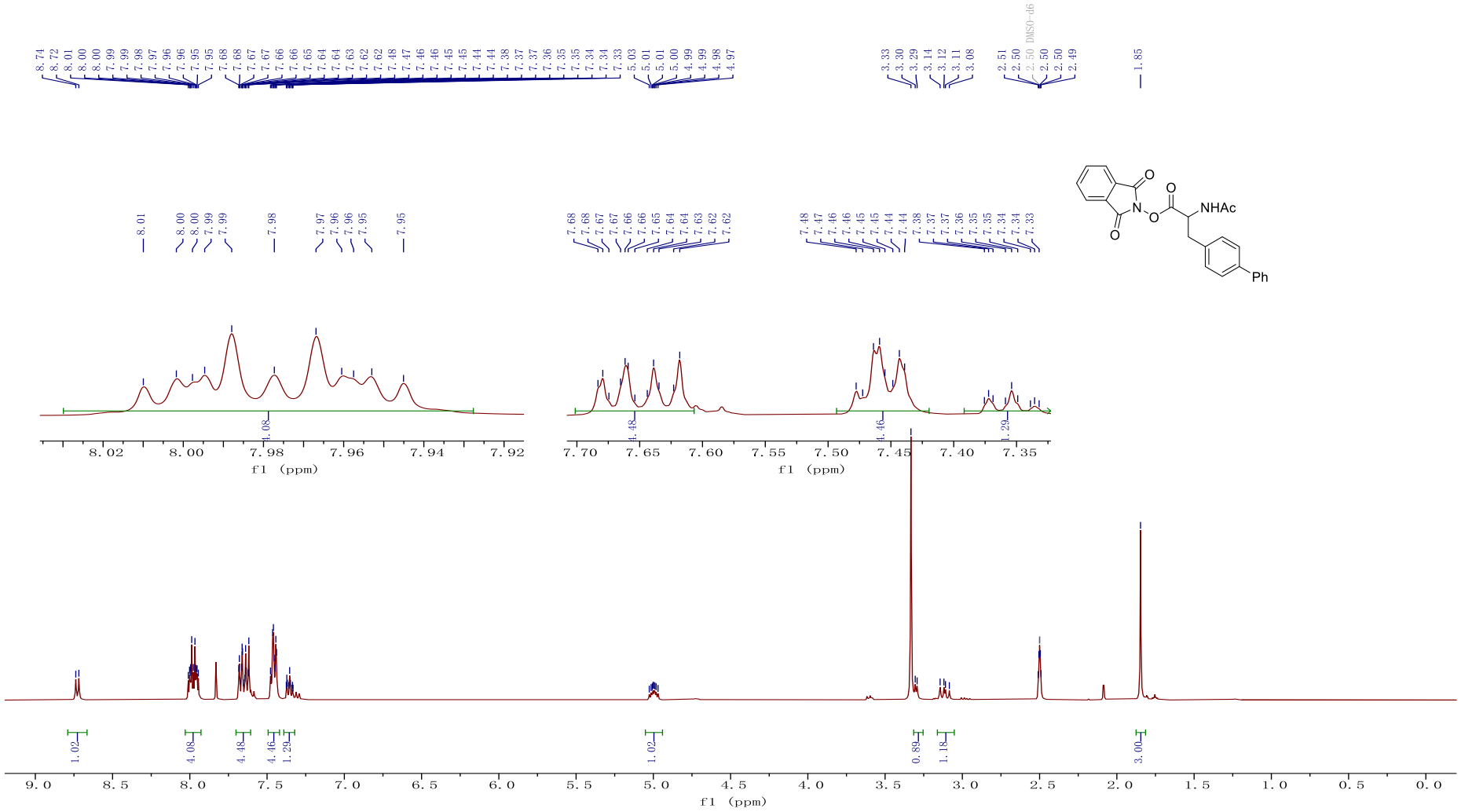
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-chlorophenyl)propanoate (**4p**)



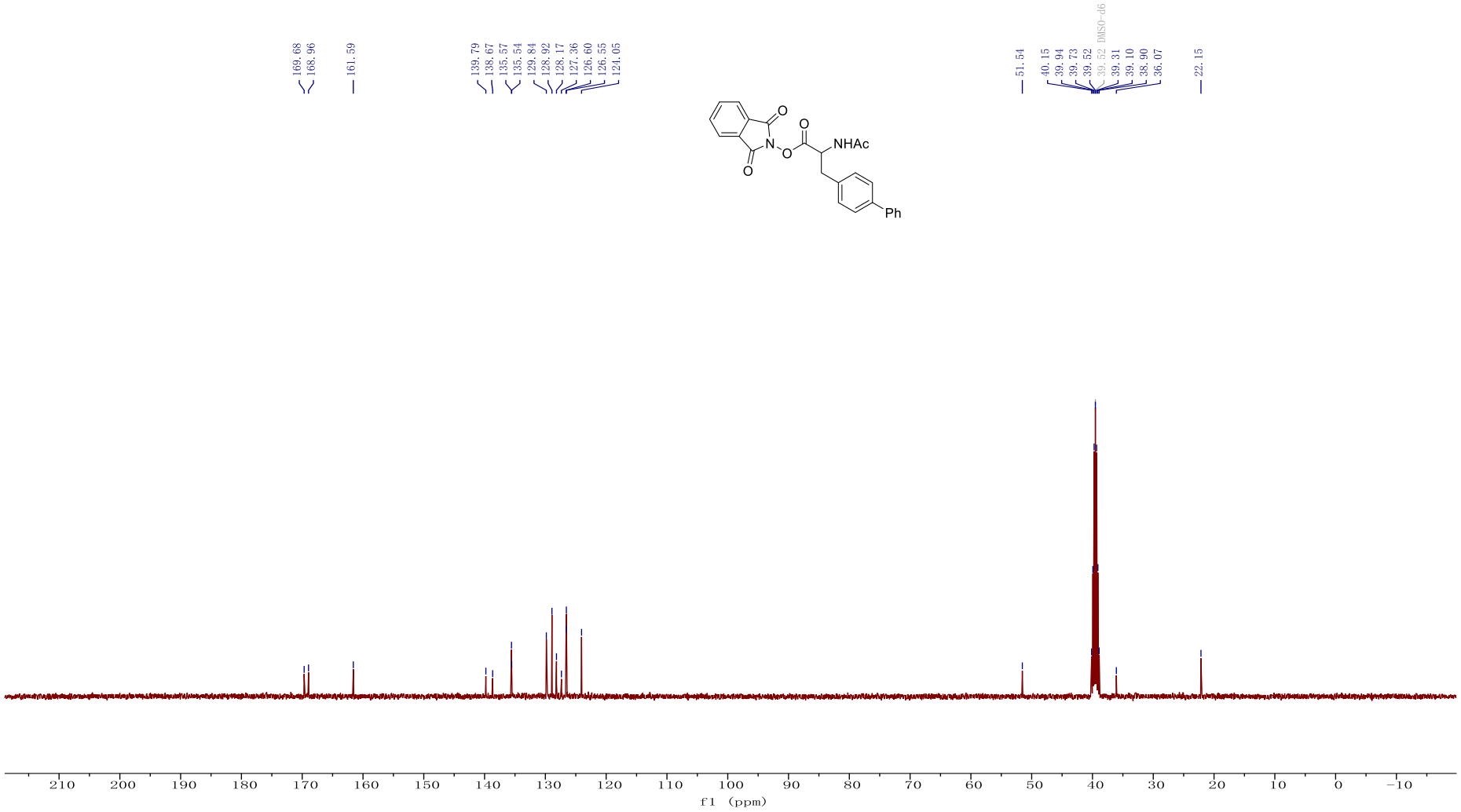
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(p-tolyl)propanoate (**4q**)



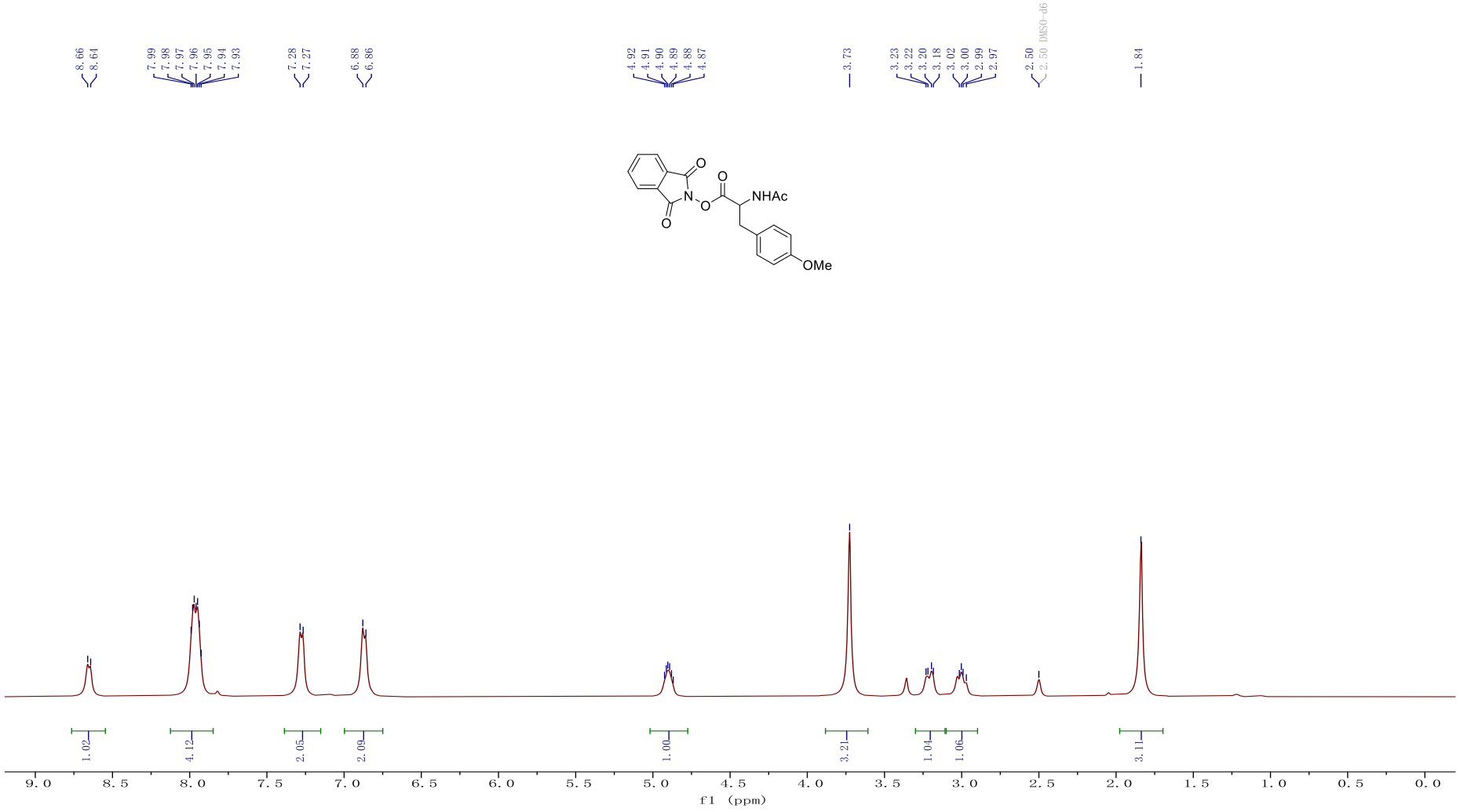
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 3-([1,1'-biphenyl]-4-yl)-2-acetamidopropanoate (**4r**)



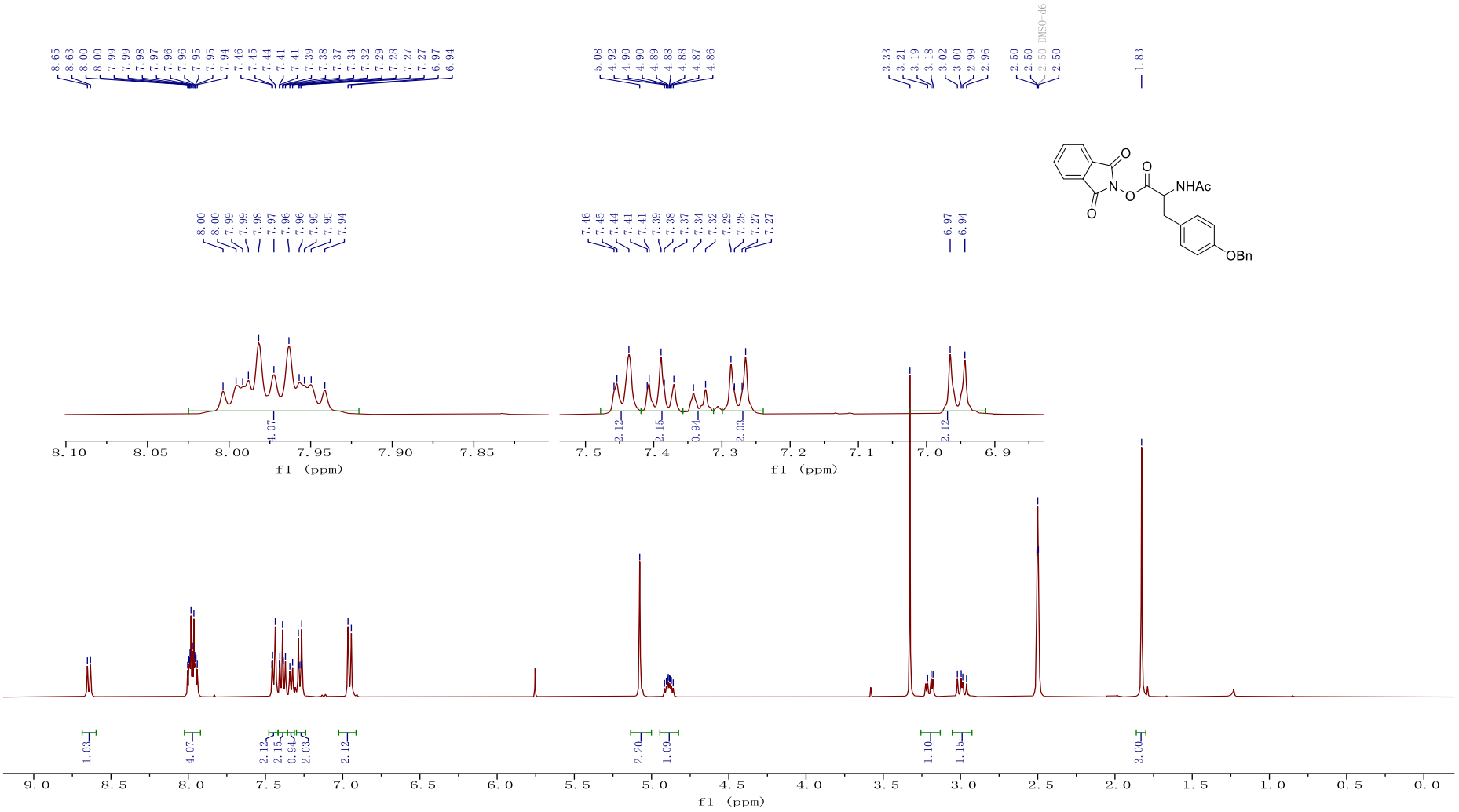
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 3-([1,1'-biphenyl]-4-yl)-2-acetamidopropanoate (**4r**)



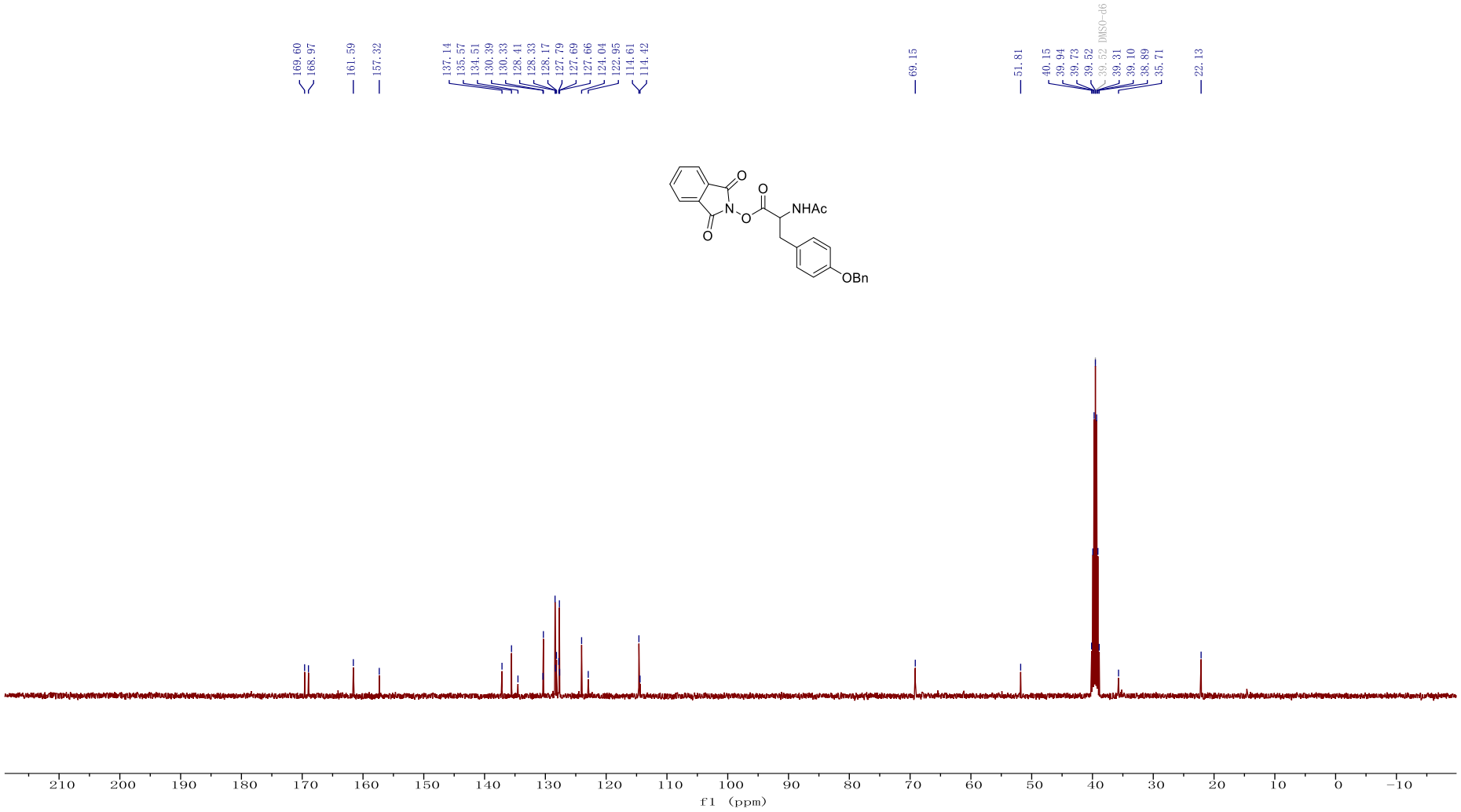
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-methoxyphenyl)propanoate (**4s**)



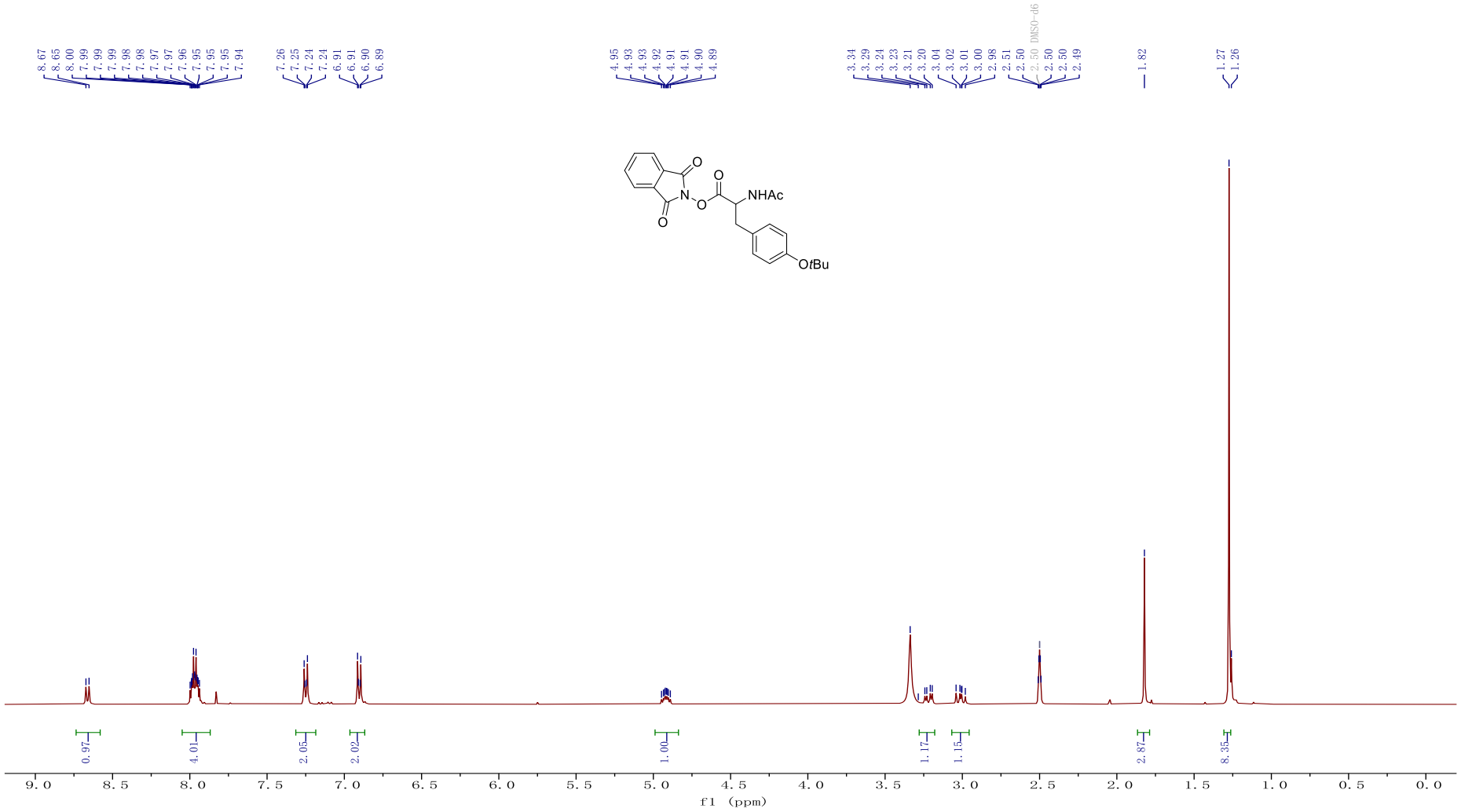
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(benzyloxy)phenyl)propanoate (**4t**)



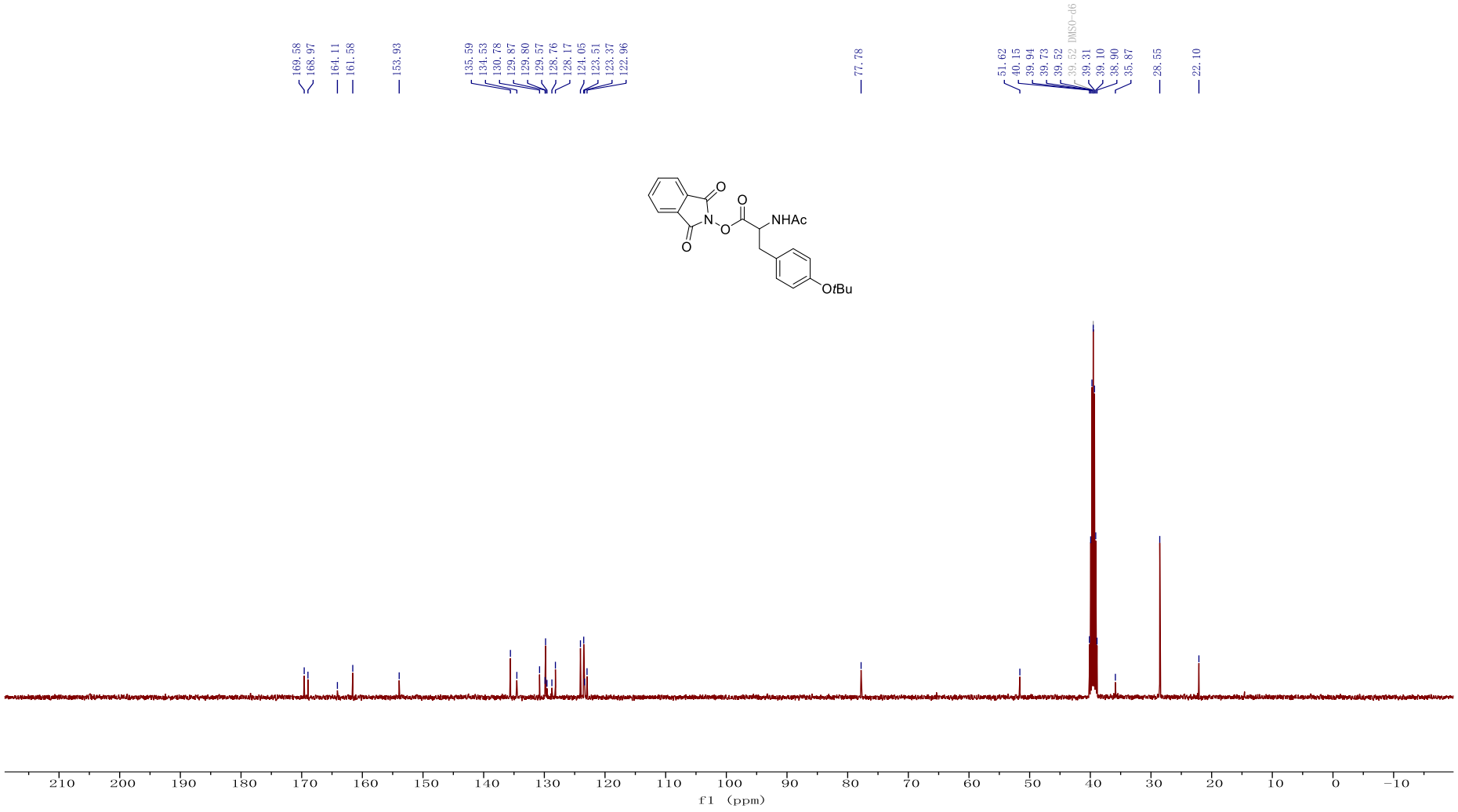
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(benzyloxy)phenyl)propanoate (**4t**)



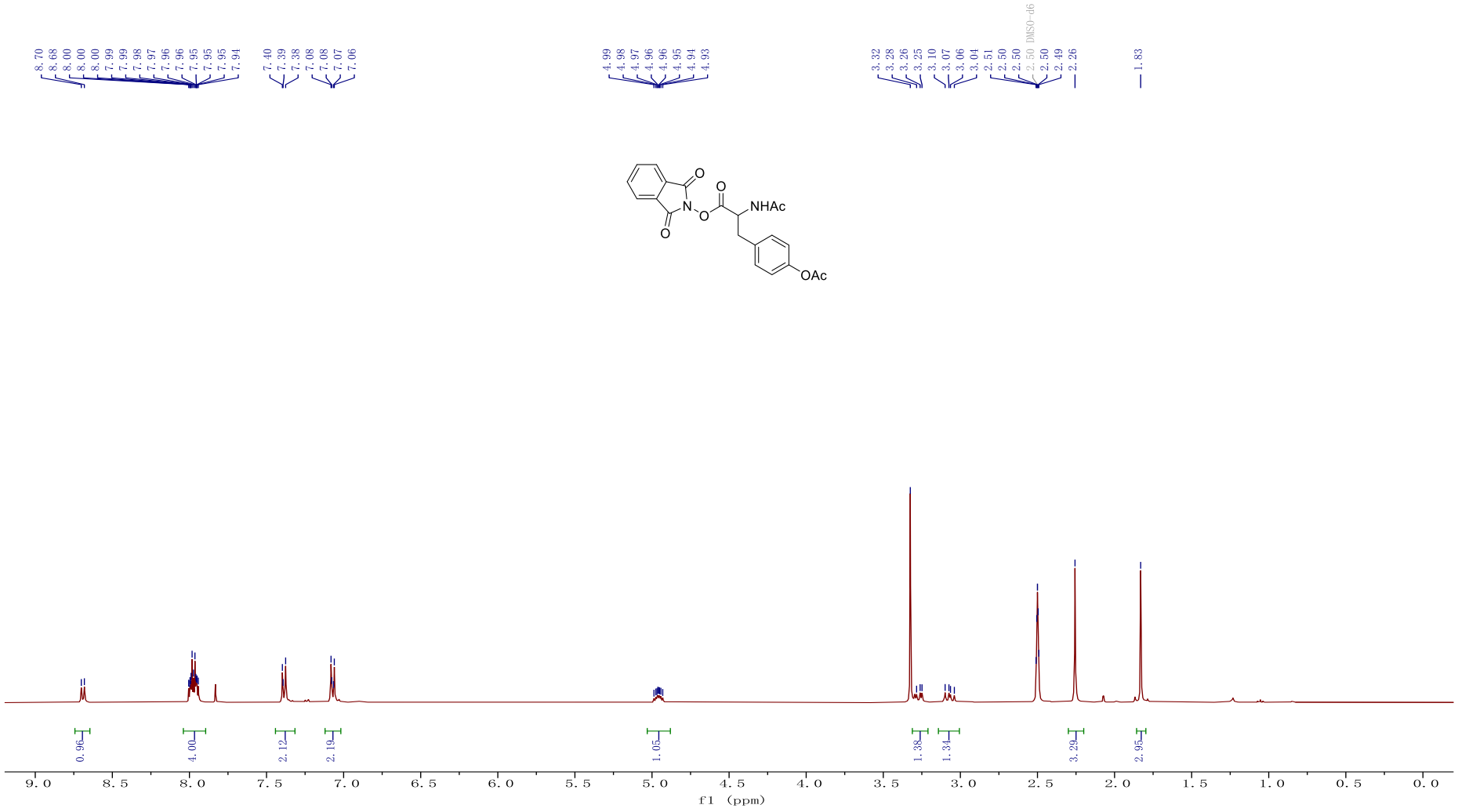
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(tert-butoxy)phenyl)propanoate (**4u**)



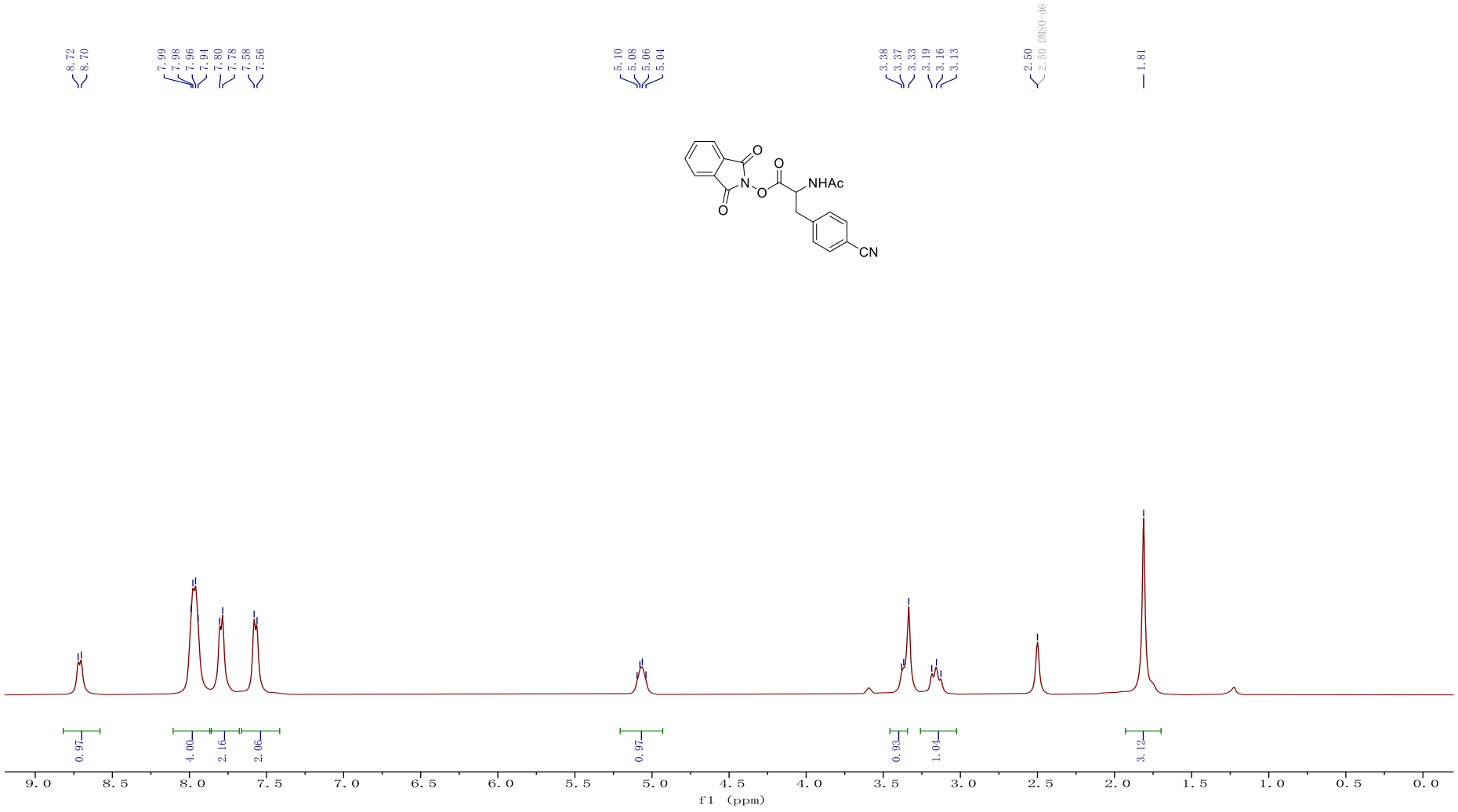
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-(tert-butoxy)phenyl)propanoate (**4u**)



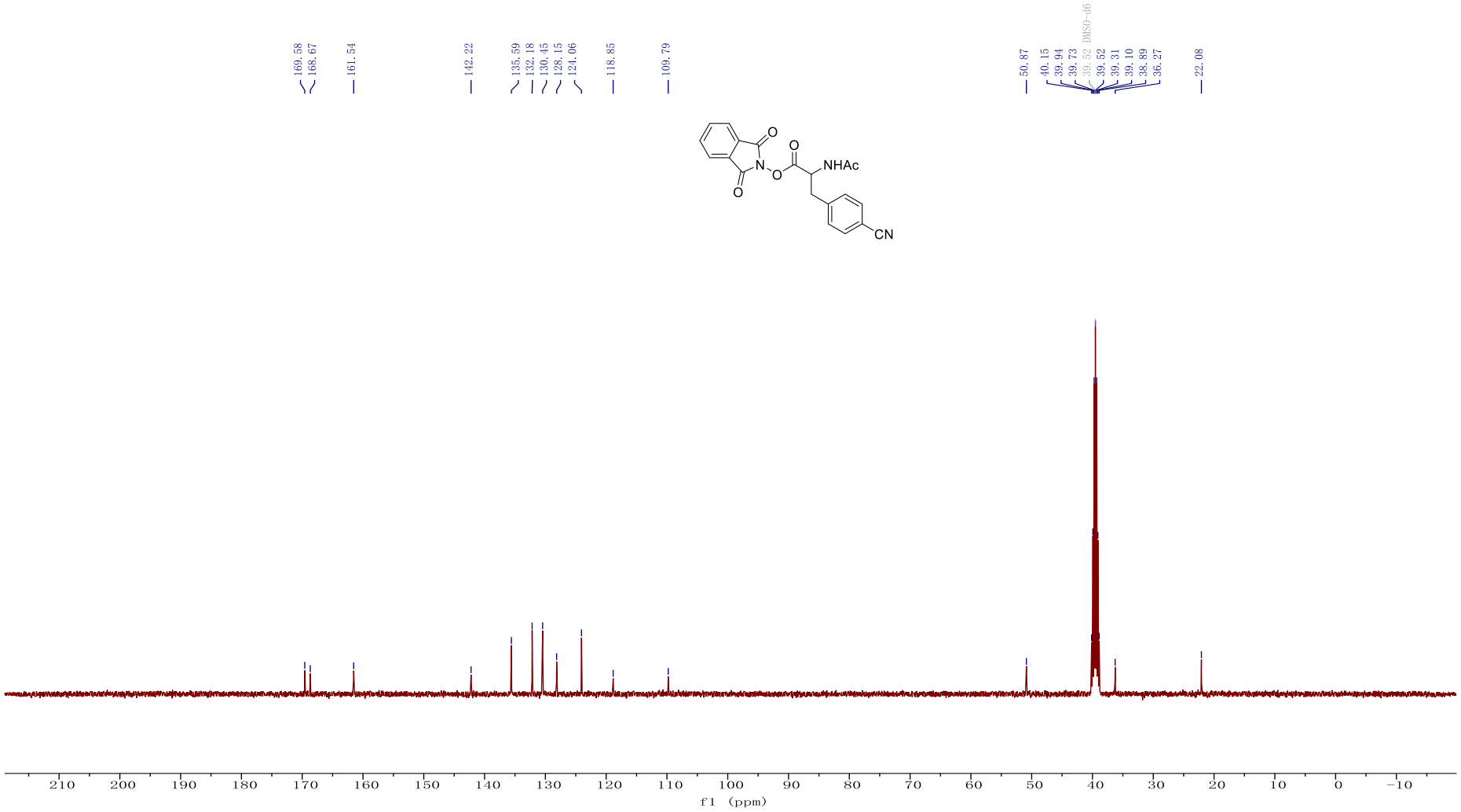
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-acetoxyphenyl)propanoate (**4v**)



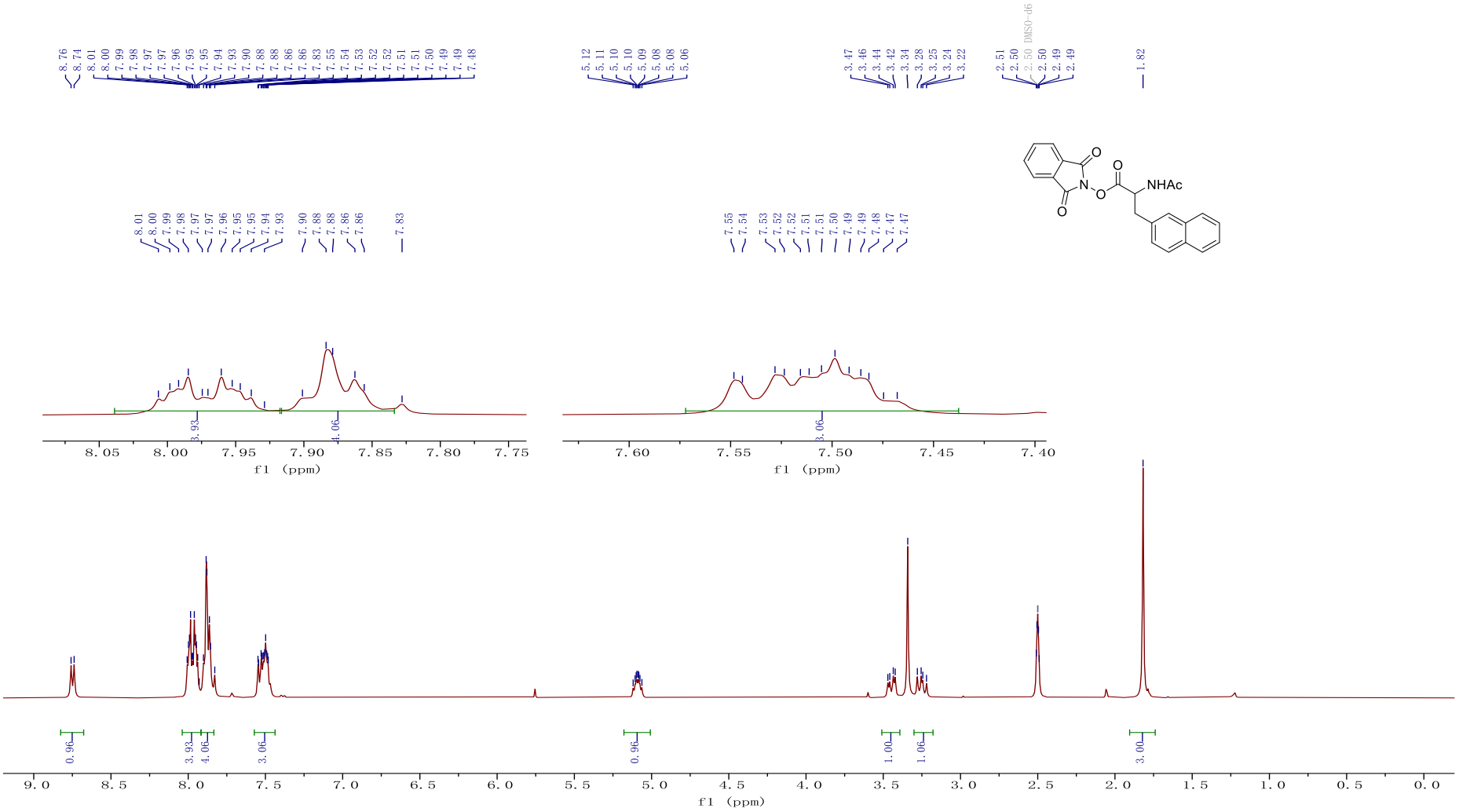
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-cyanophenyl)propanoate (**4w**)



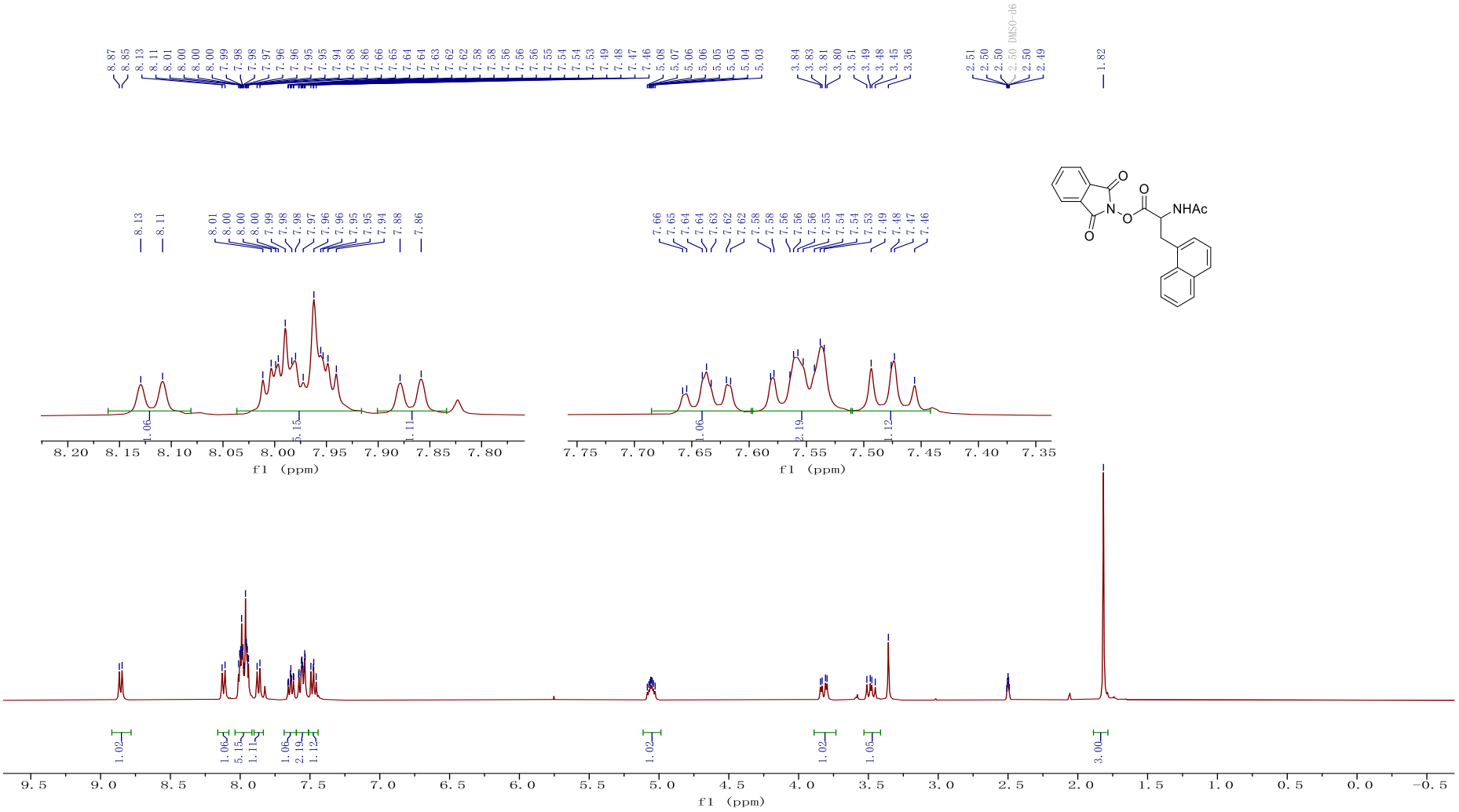
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(4-cyanophenyl)propanoate (**4w**)



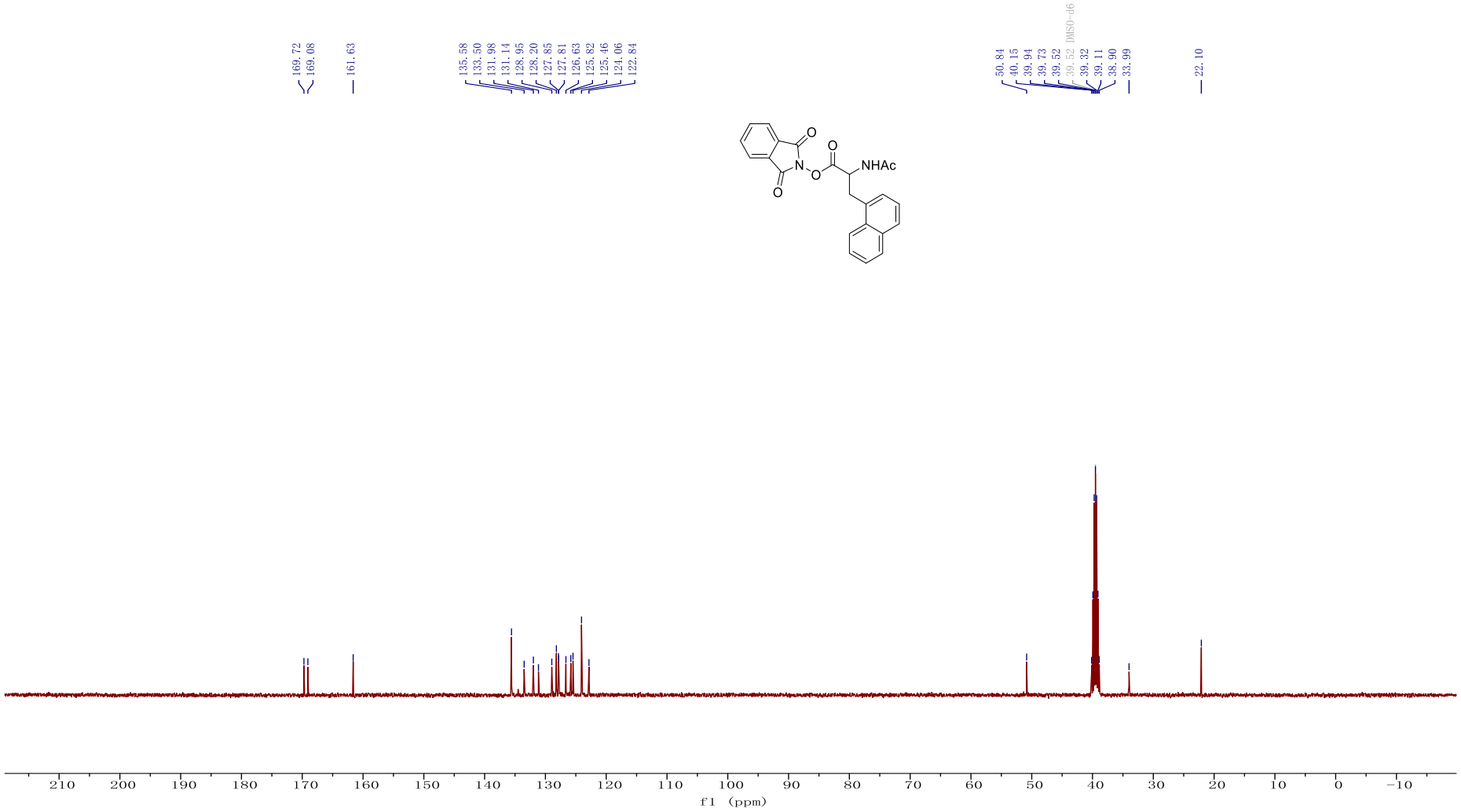
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(naphthalen-2-yl)propanoate (**4x**)



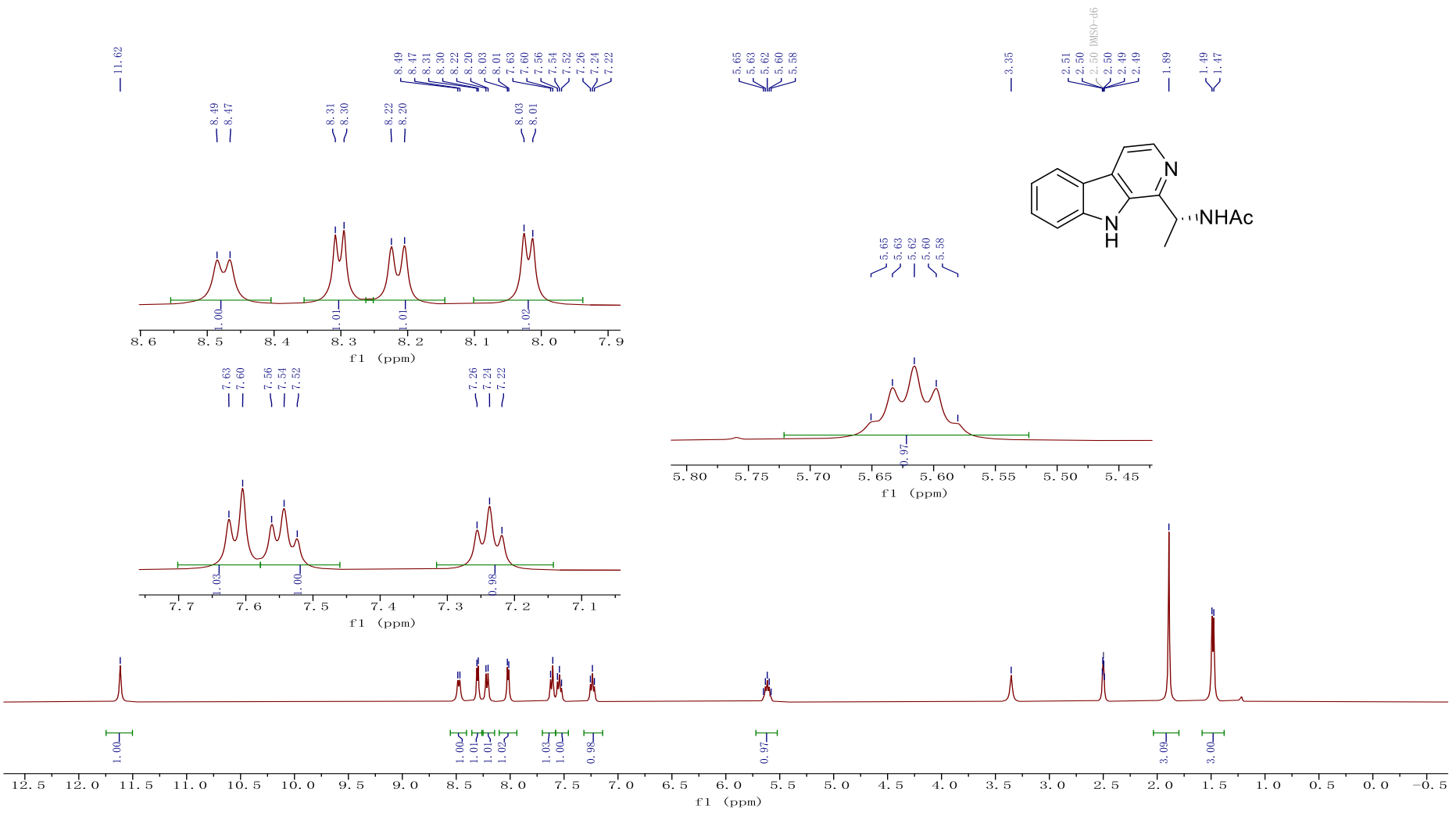
^{1}H NMR (400 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(naphthalen-1-yl)propanoate (**4y**)



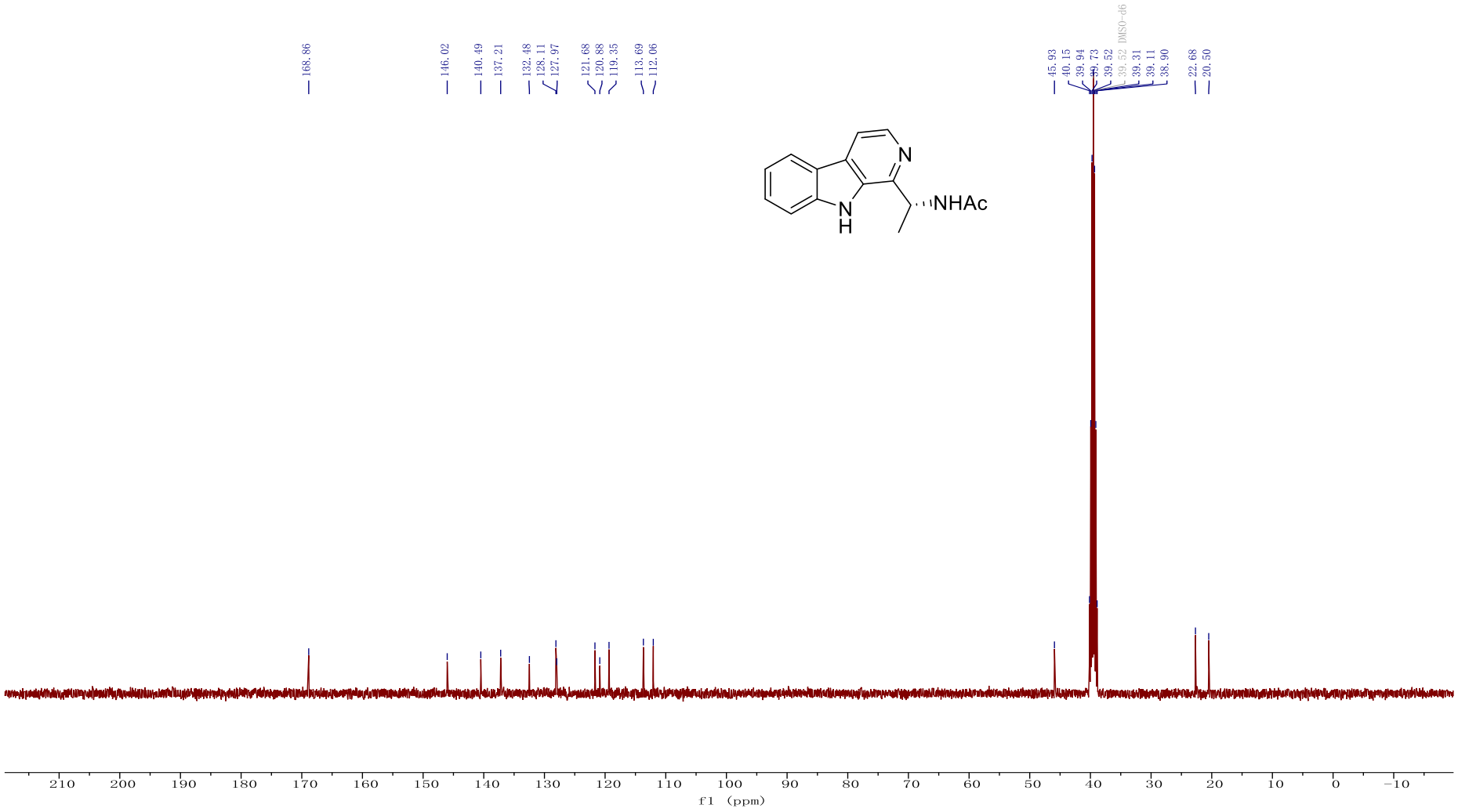
^{13}C NMR (101 MHz, DMSO-d_6) 1,3-dioxoisoindolin-2-yl 2-acetamido-3-(naphthalen-1-yl)propanoate (**4y**)



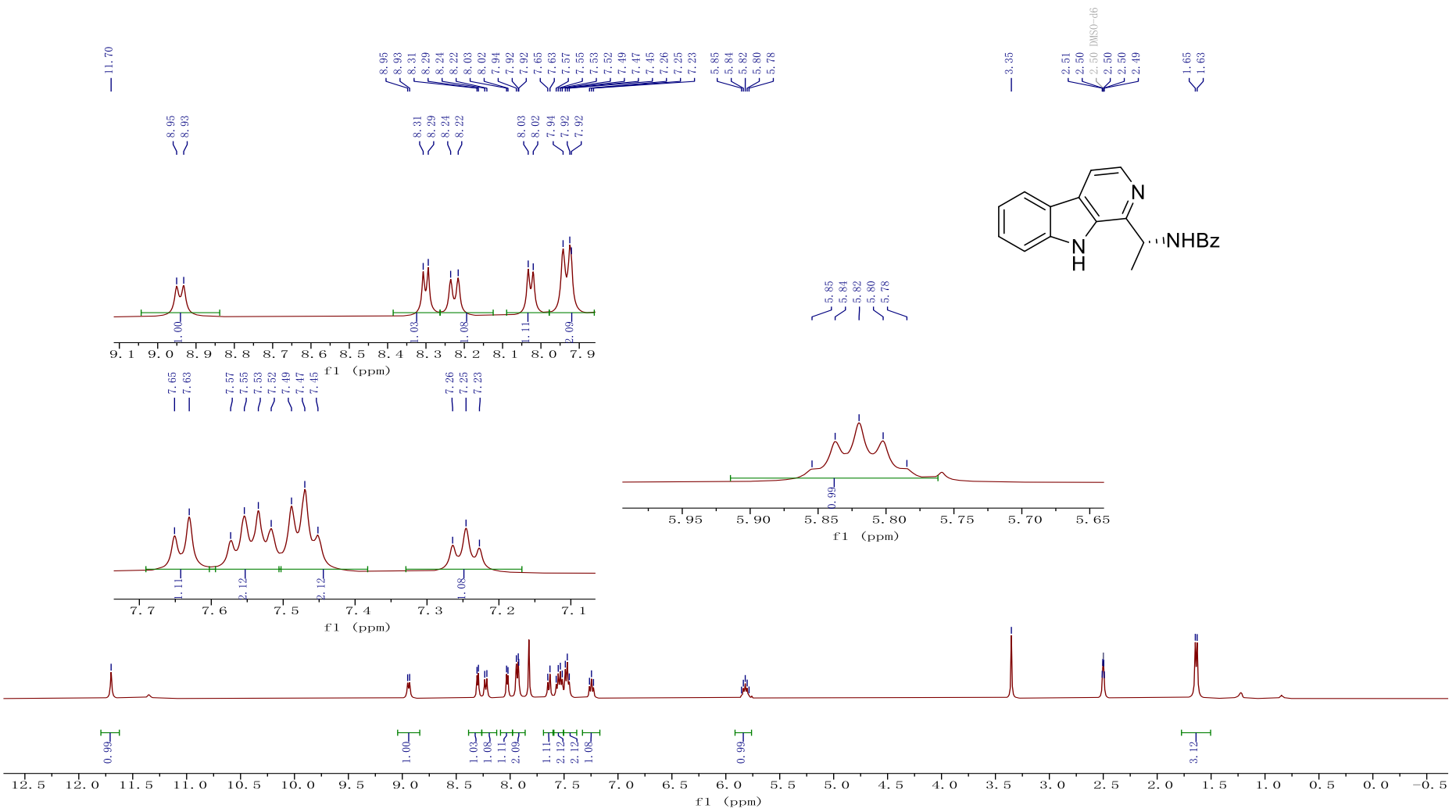
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3a**)



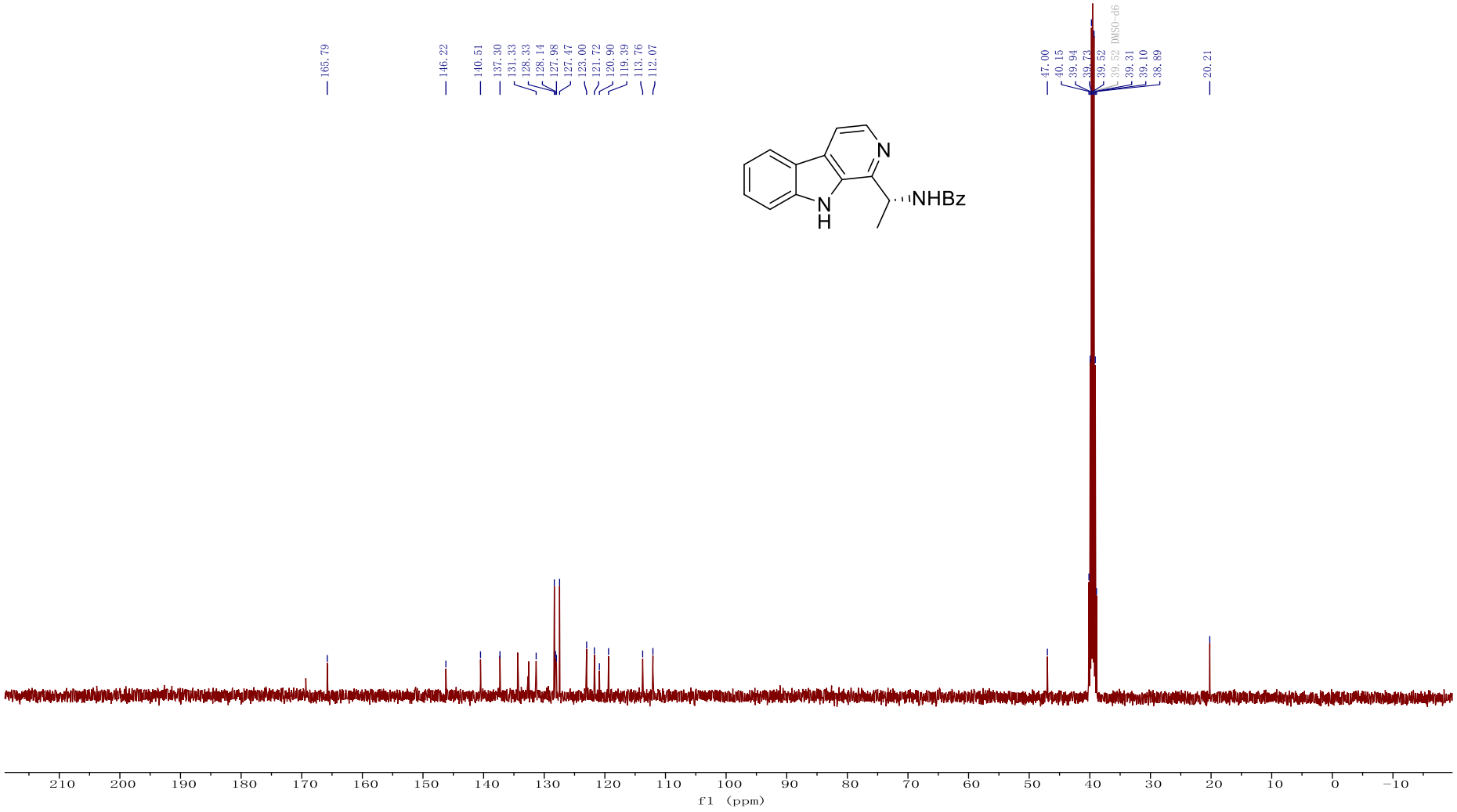
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3a**)



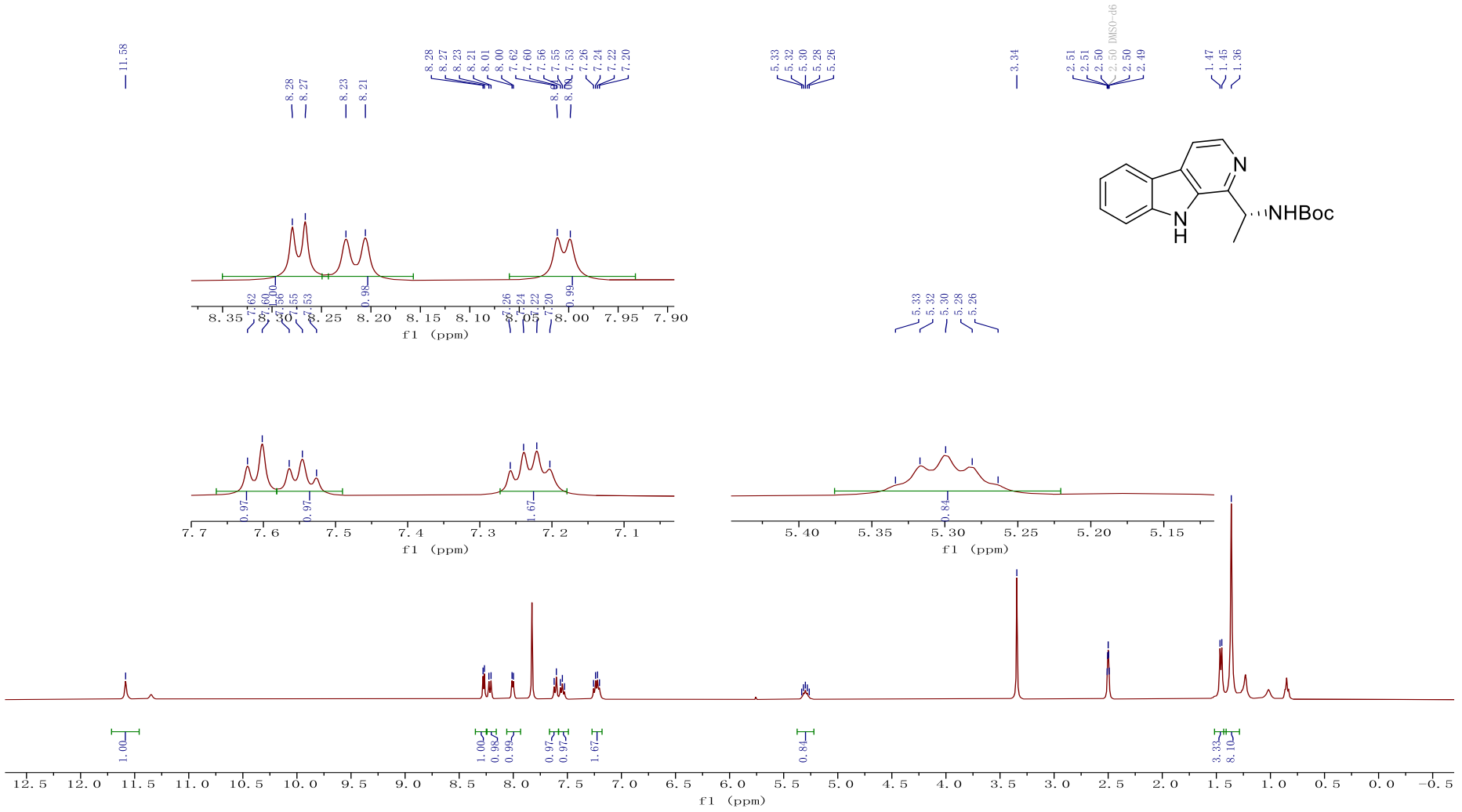
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)benzamide (**3ab**)



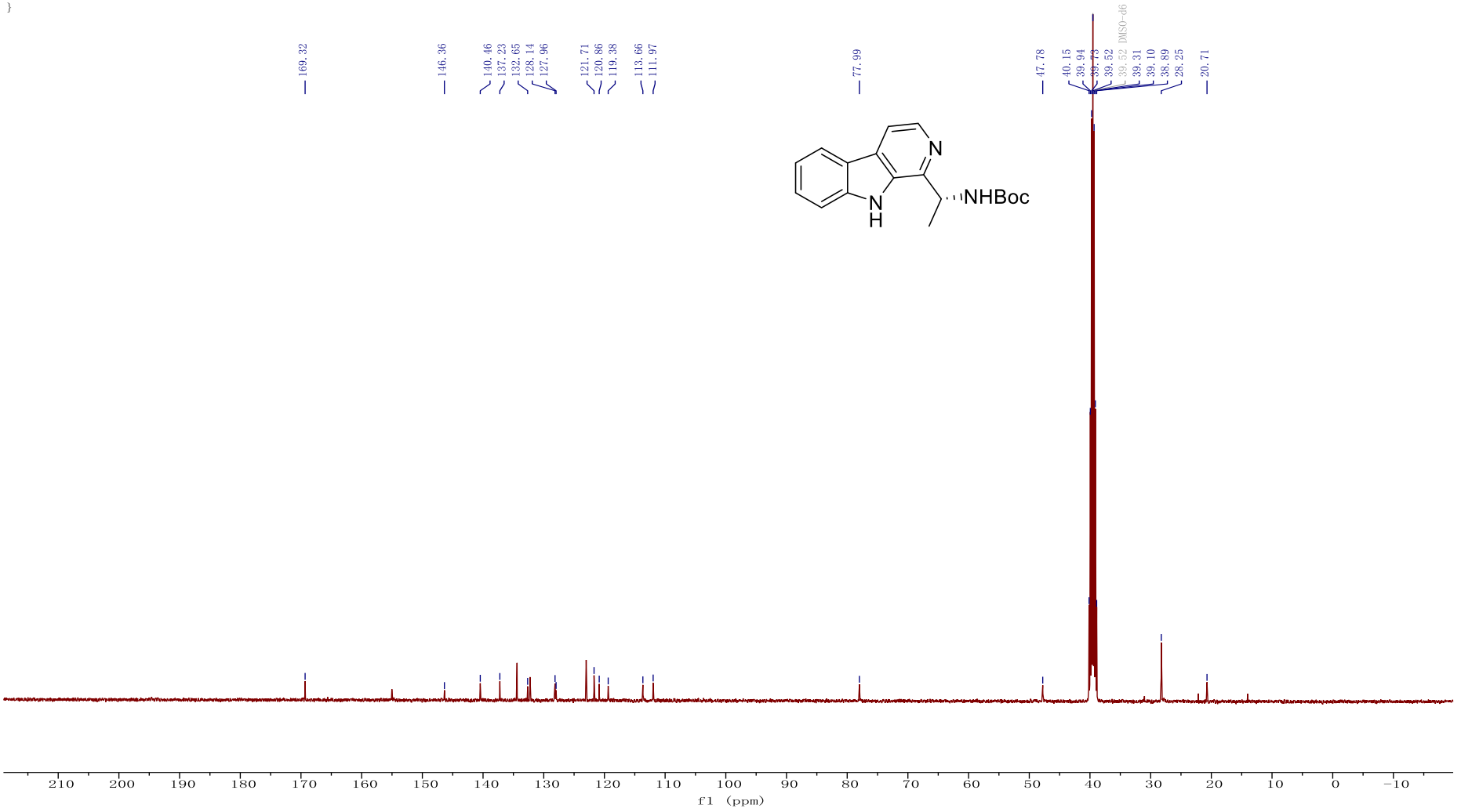
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)benzamide (**3ab**)



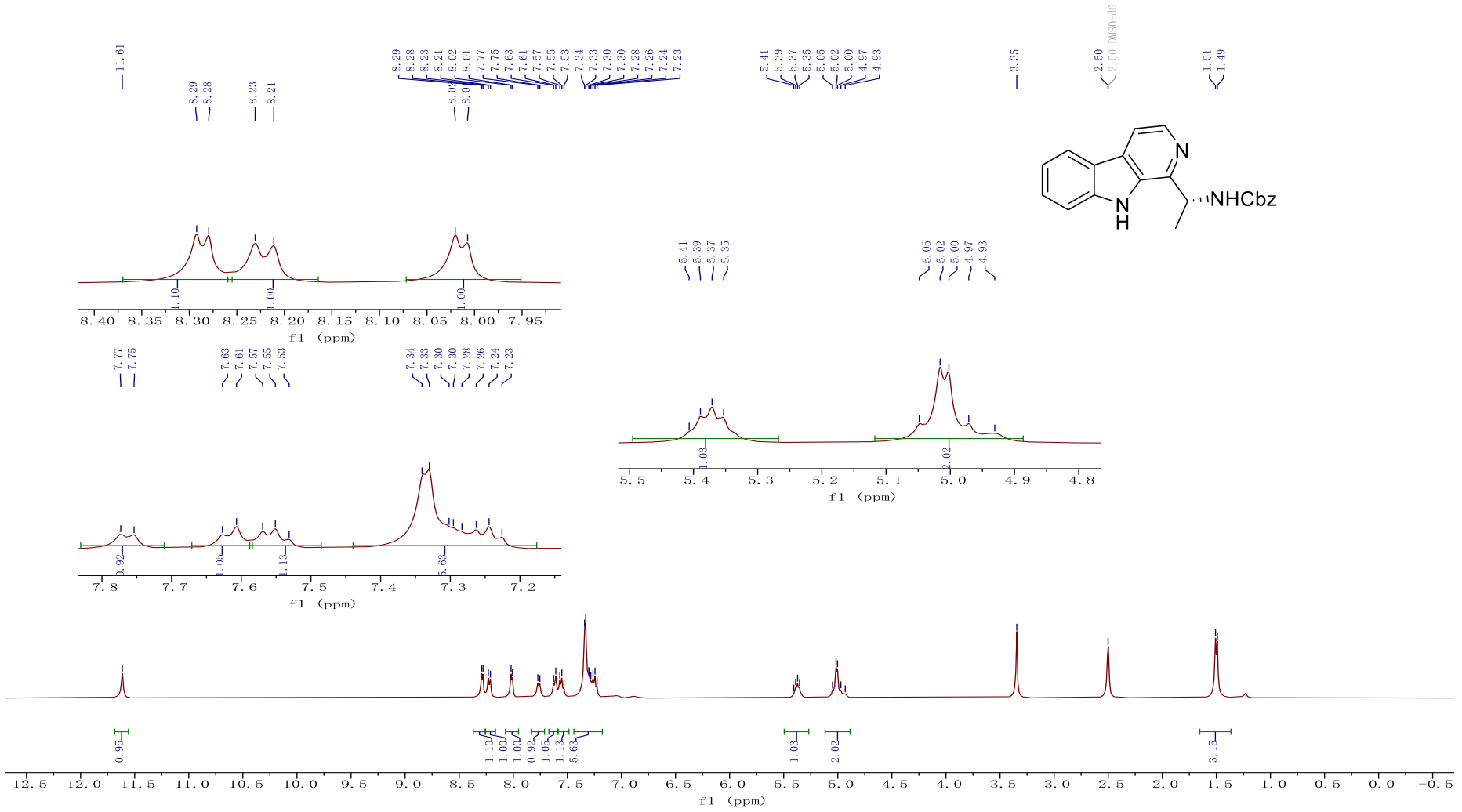
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-tert-butyl (1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ac**)



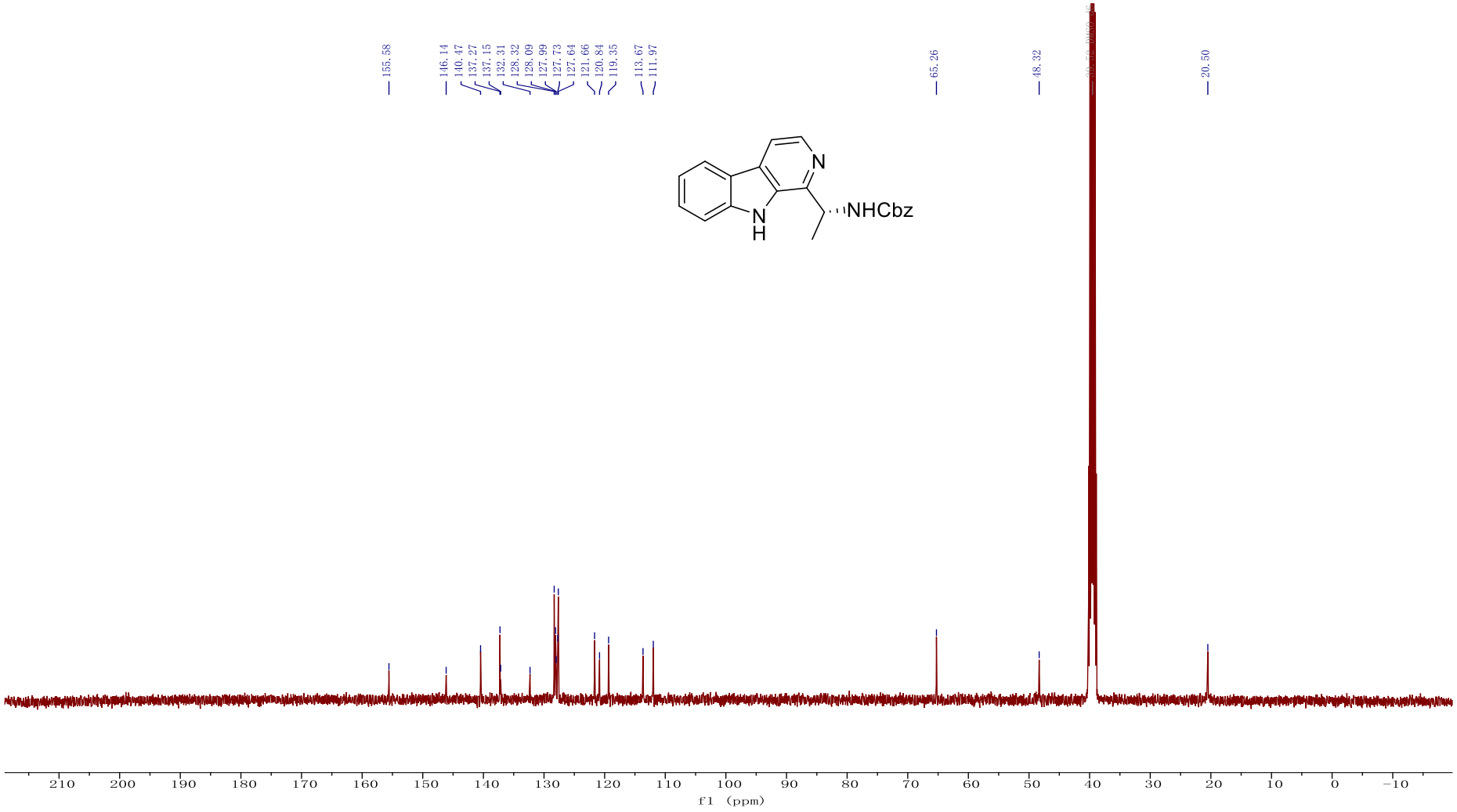
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-tert-butyl (1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ac**)



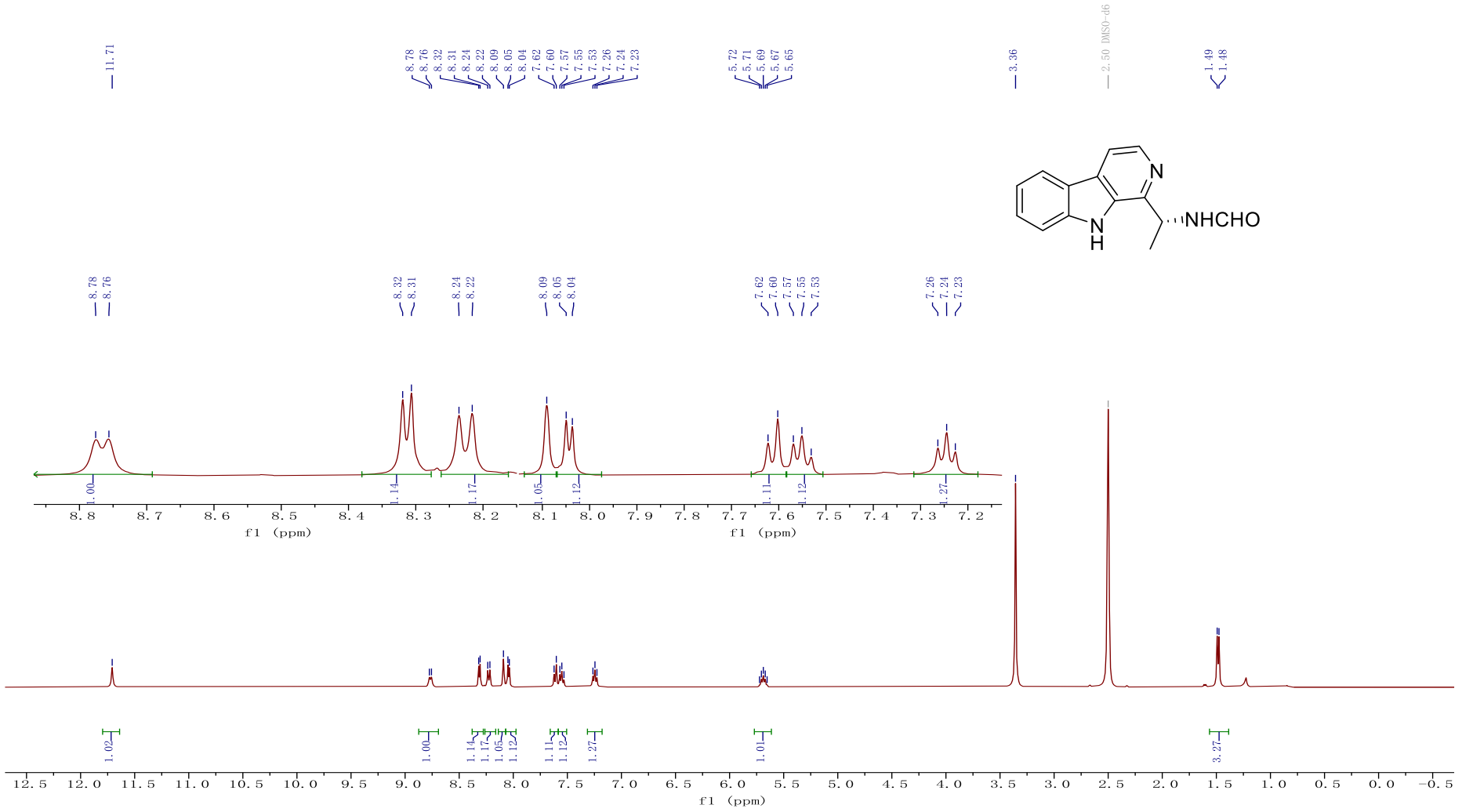
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-benzyl (1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ad**)



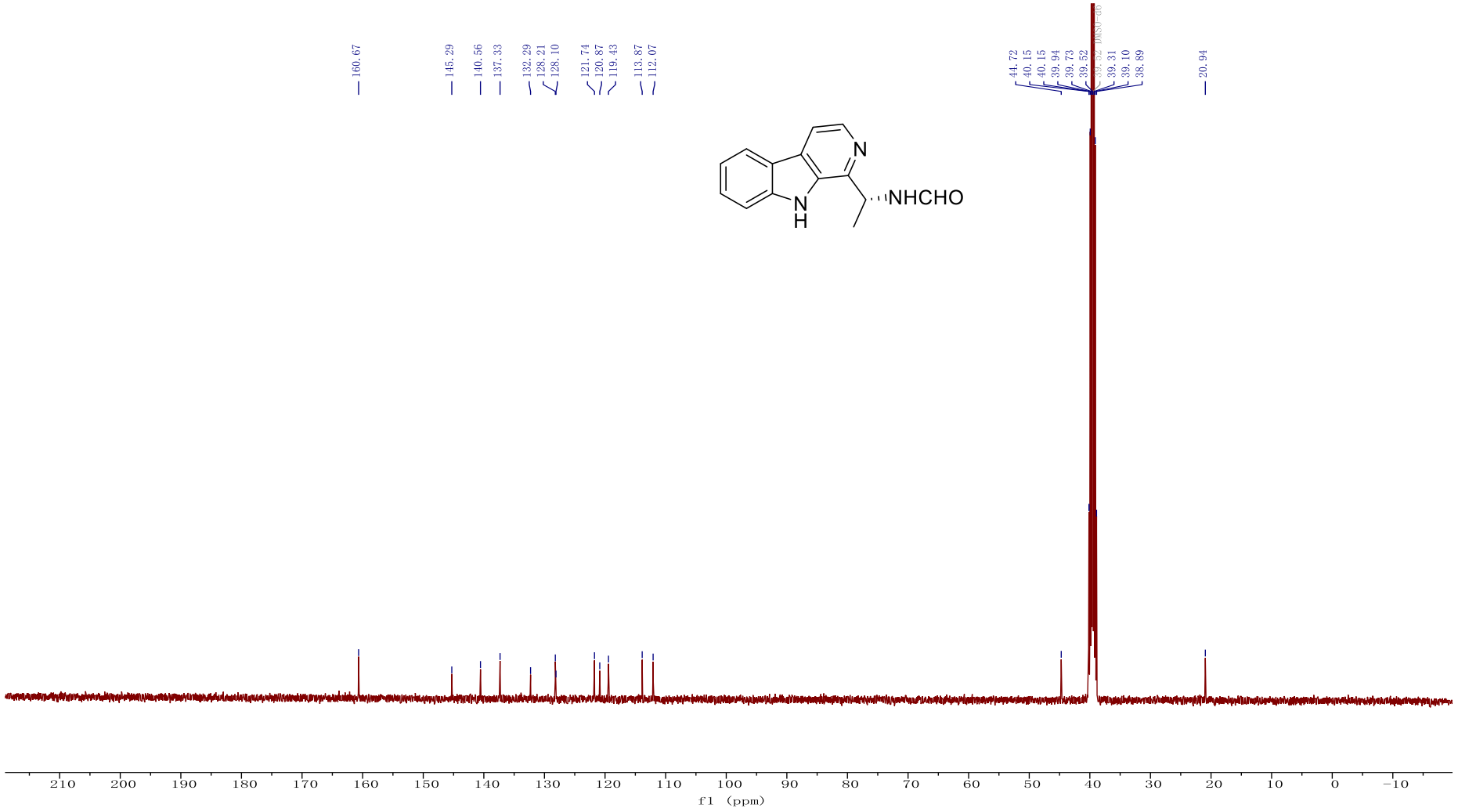
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-benzyl (1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)carbamate (**3ad**)



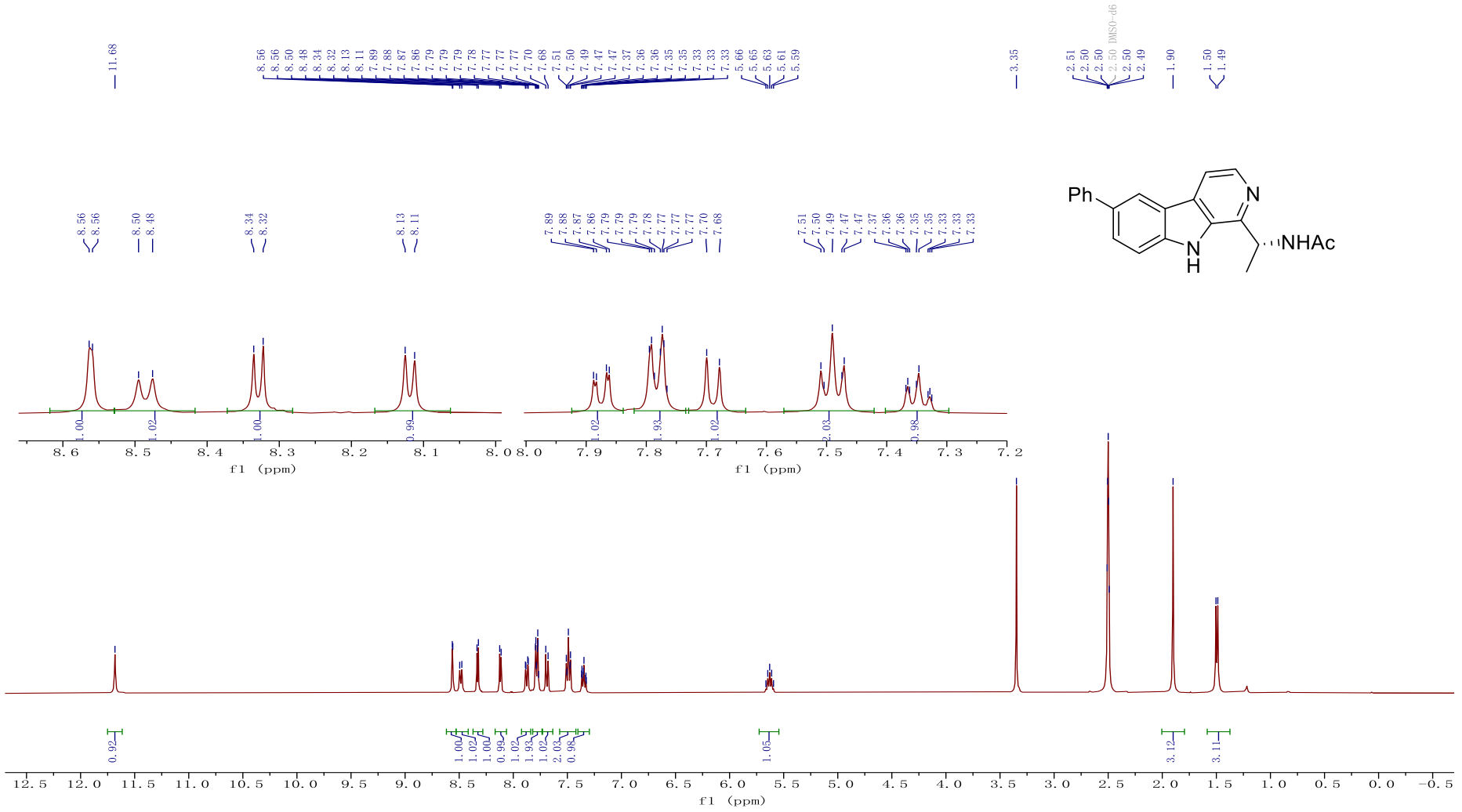
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3ae**)



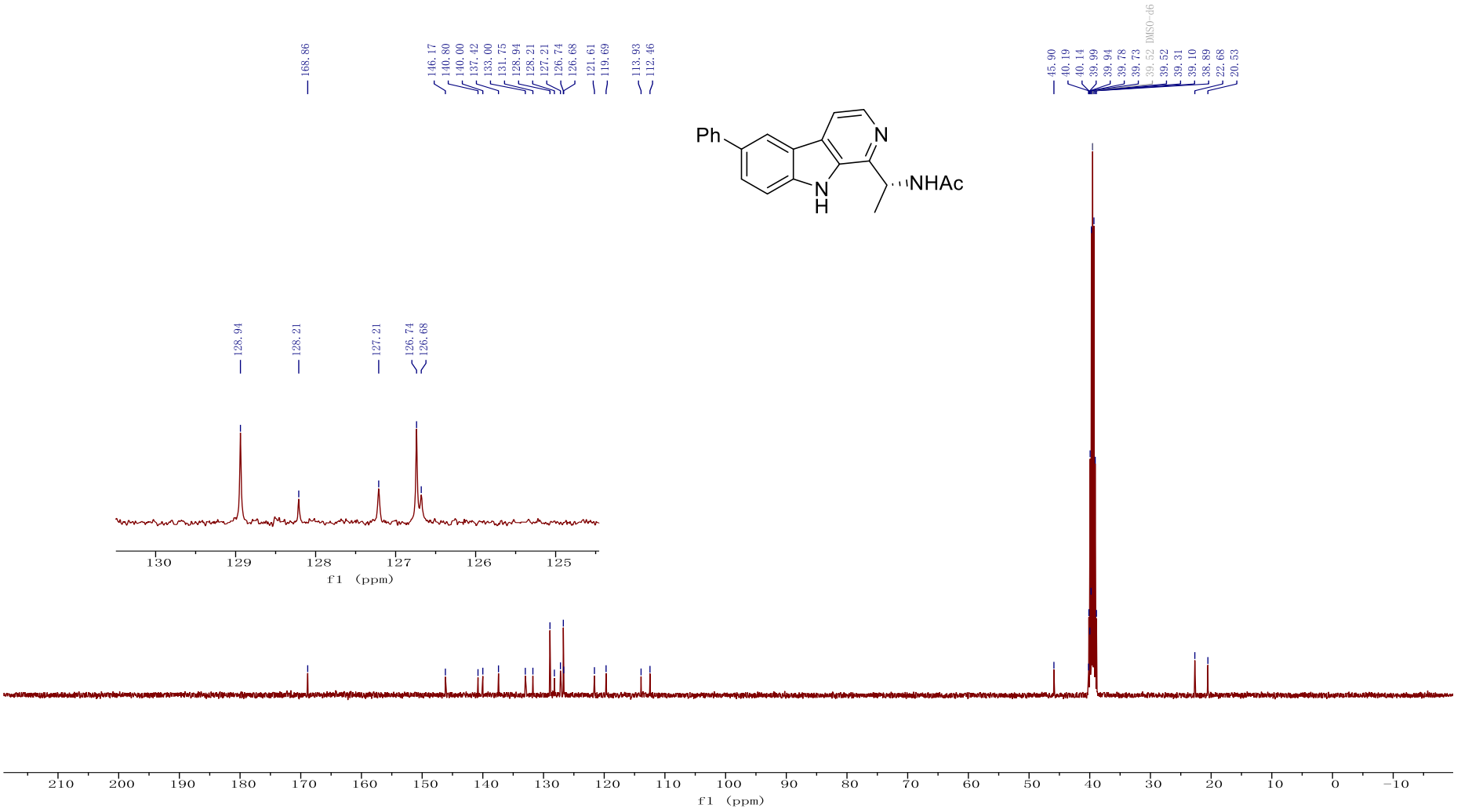
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3ae**)



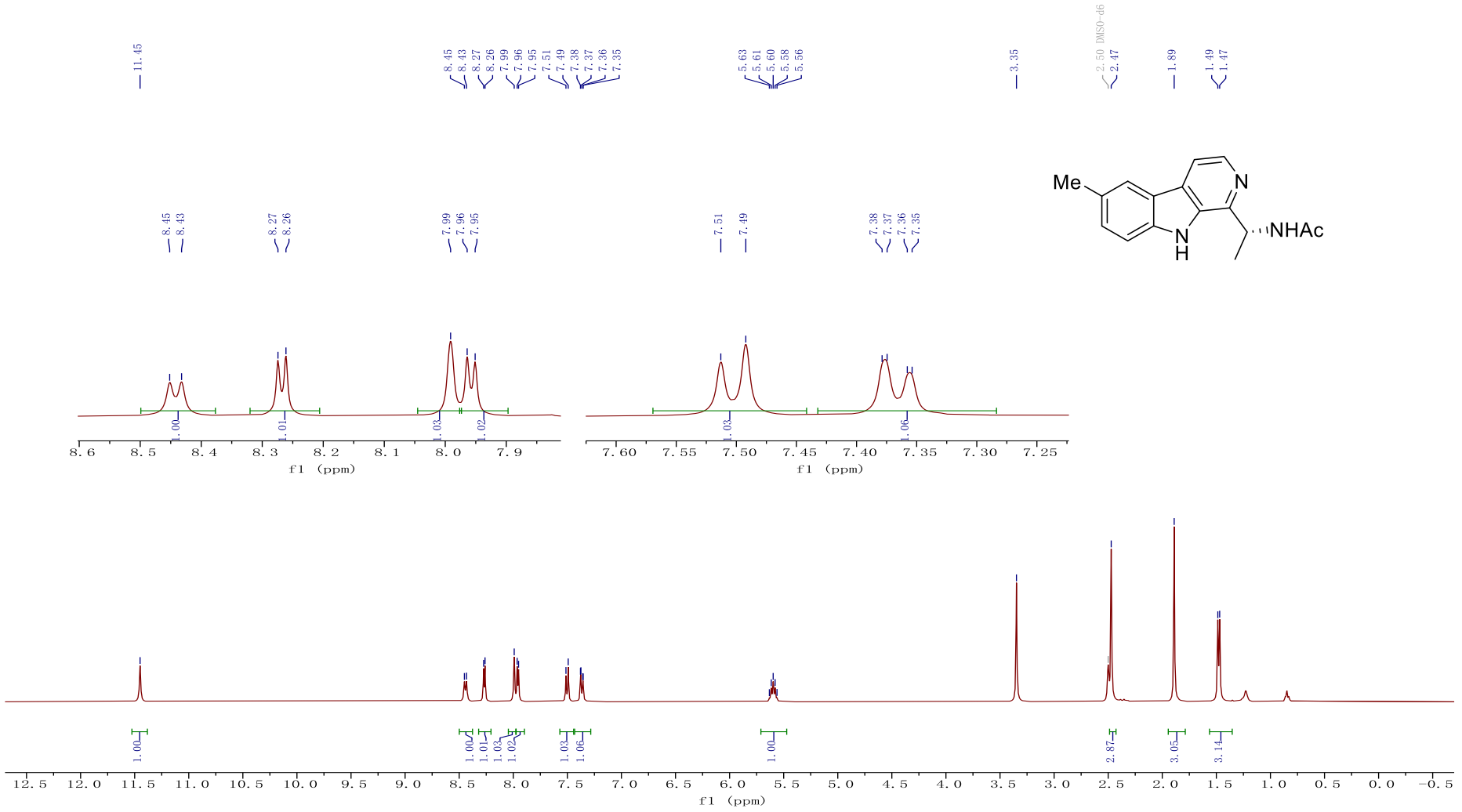
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-phenyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3b**)



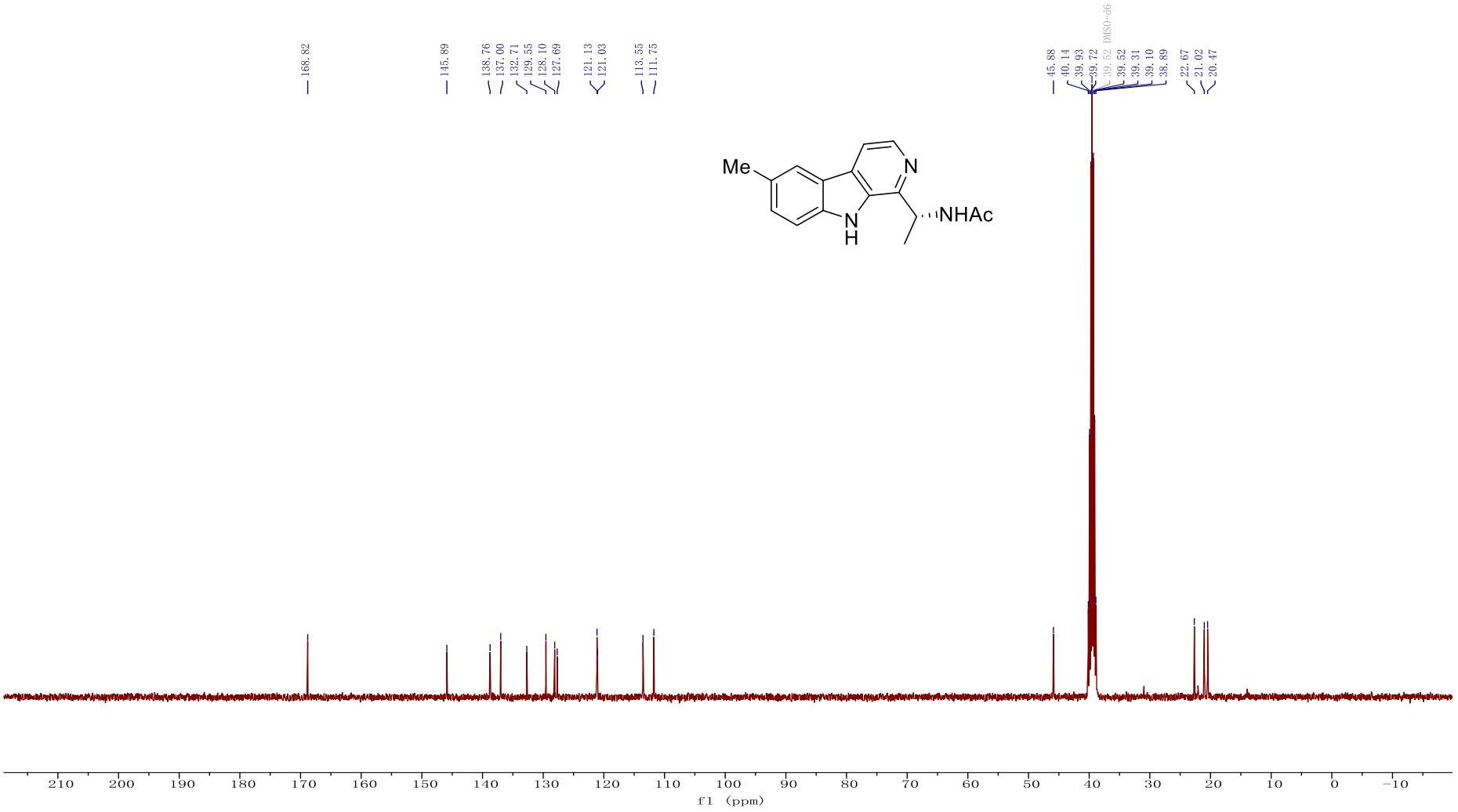
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-phenyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3b**)



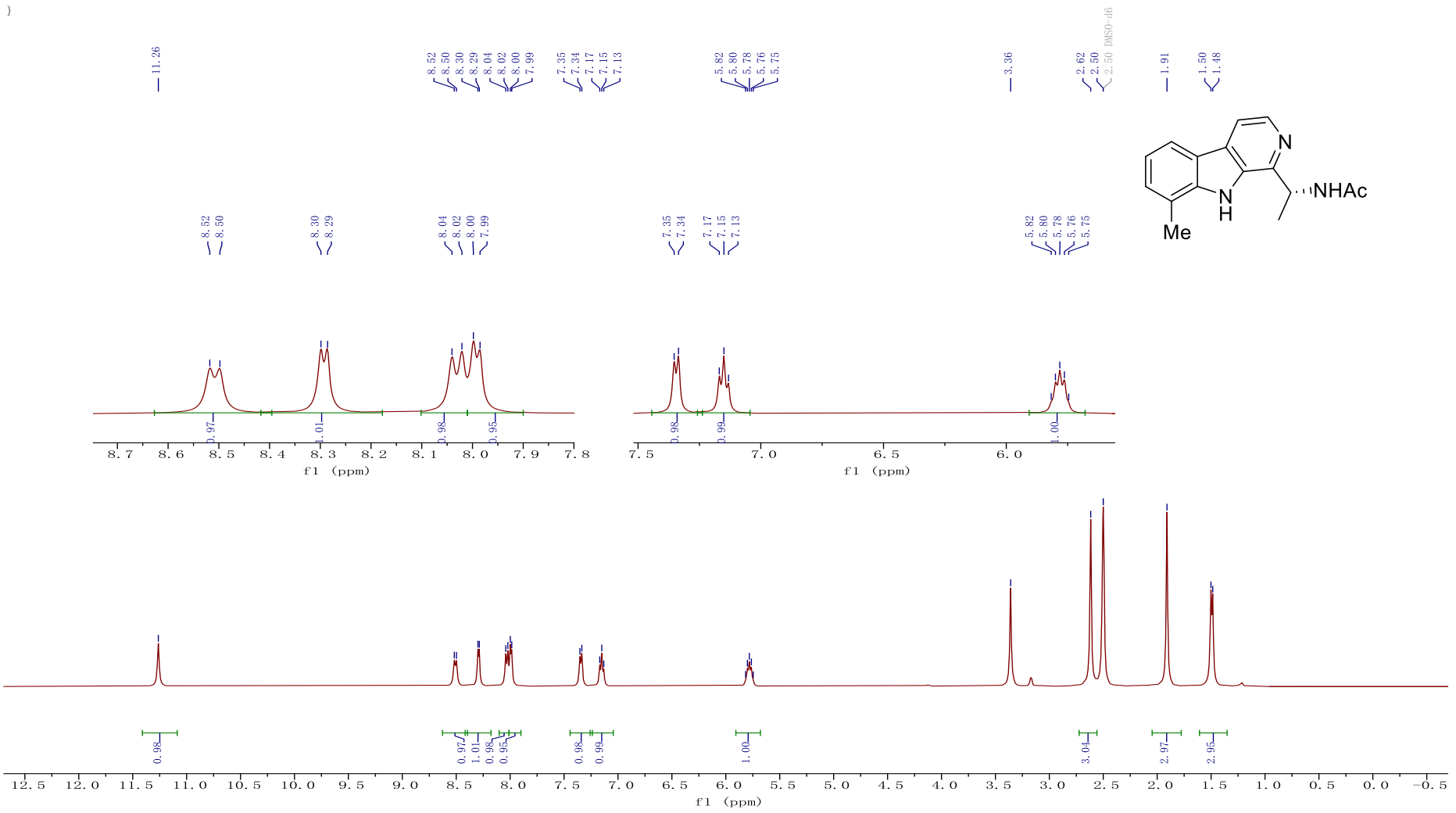
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3c**)



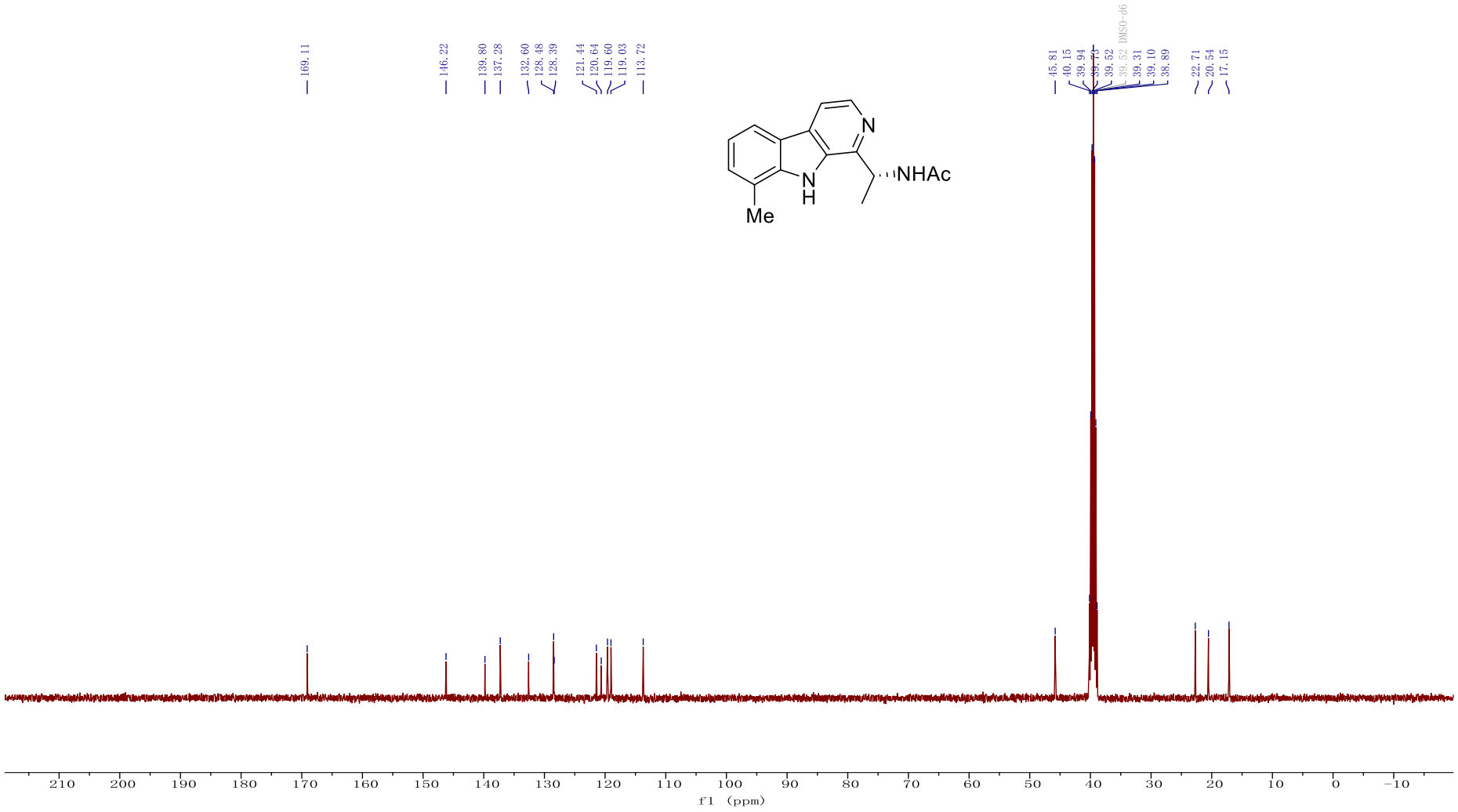
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3c**)



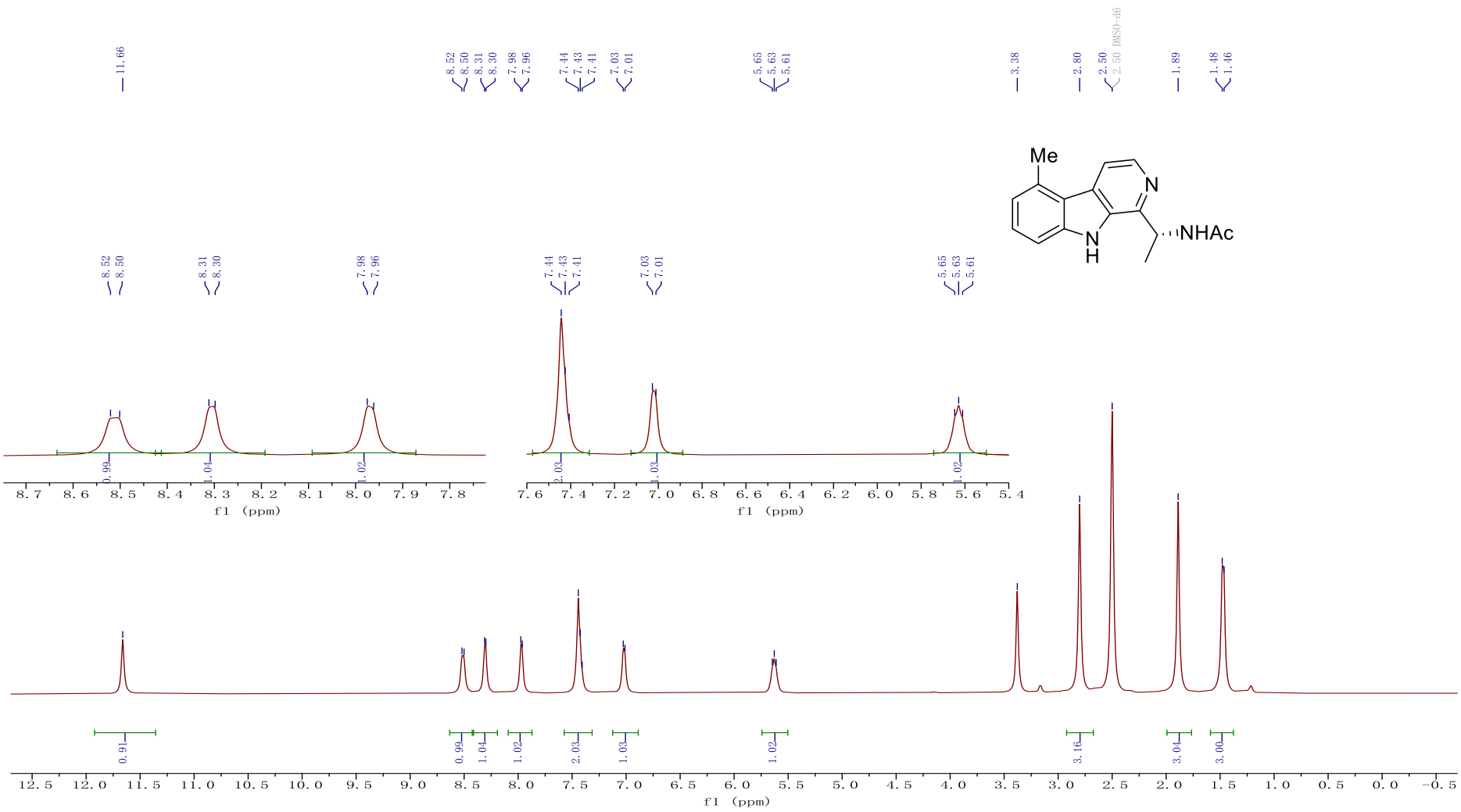
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(8-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3d**)



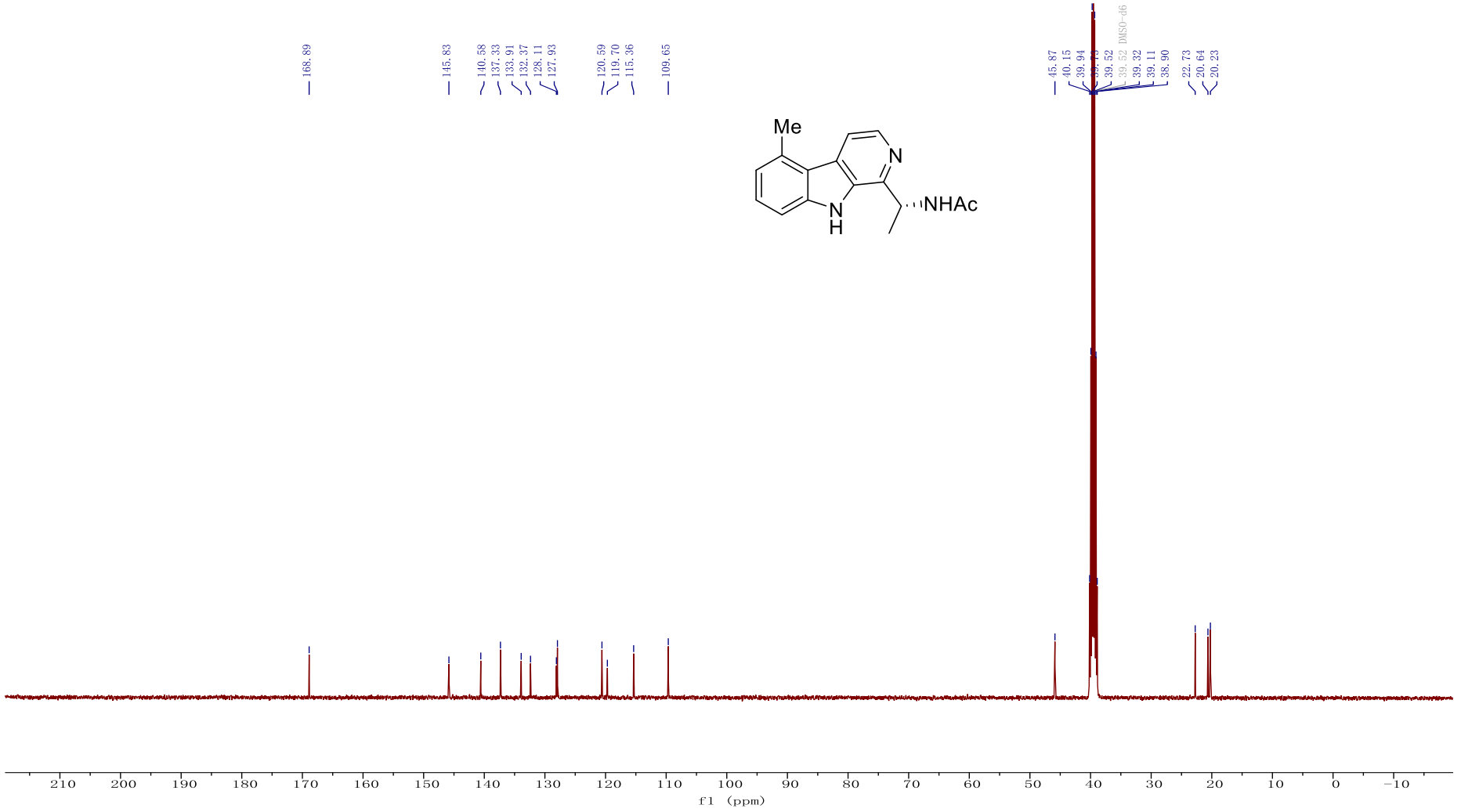
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(8-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3d**)



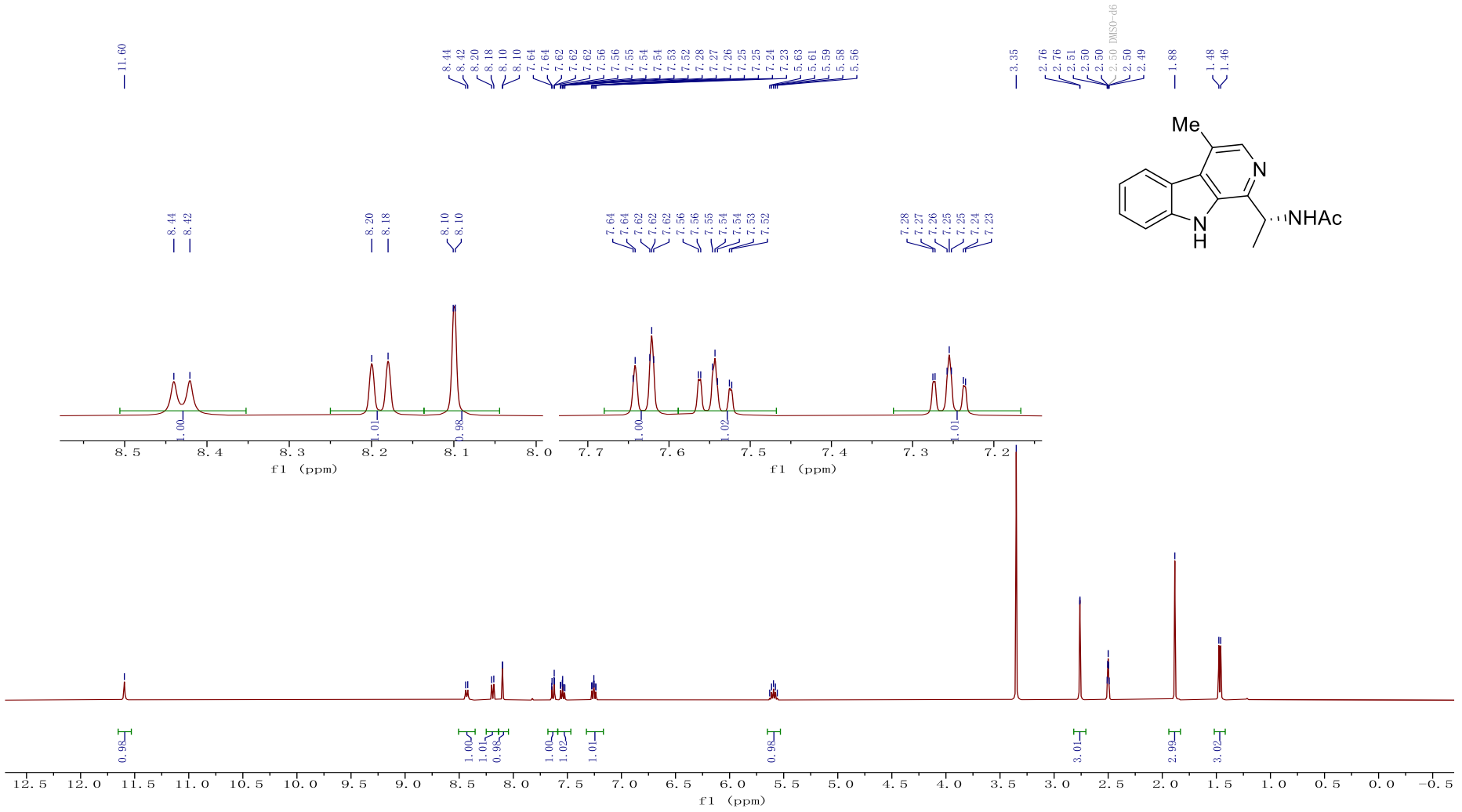
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(5-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3e**)



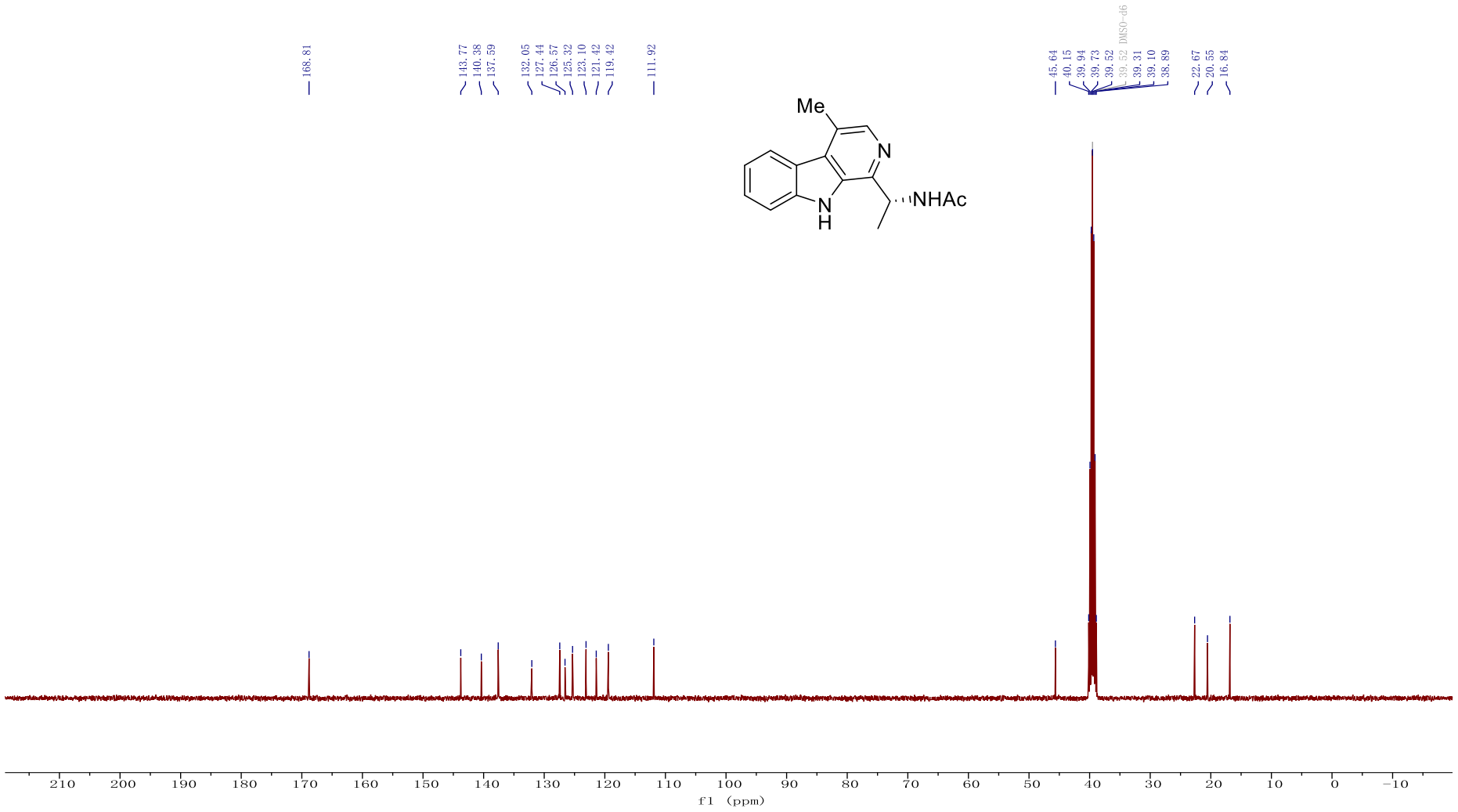
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(5-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3e**)



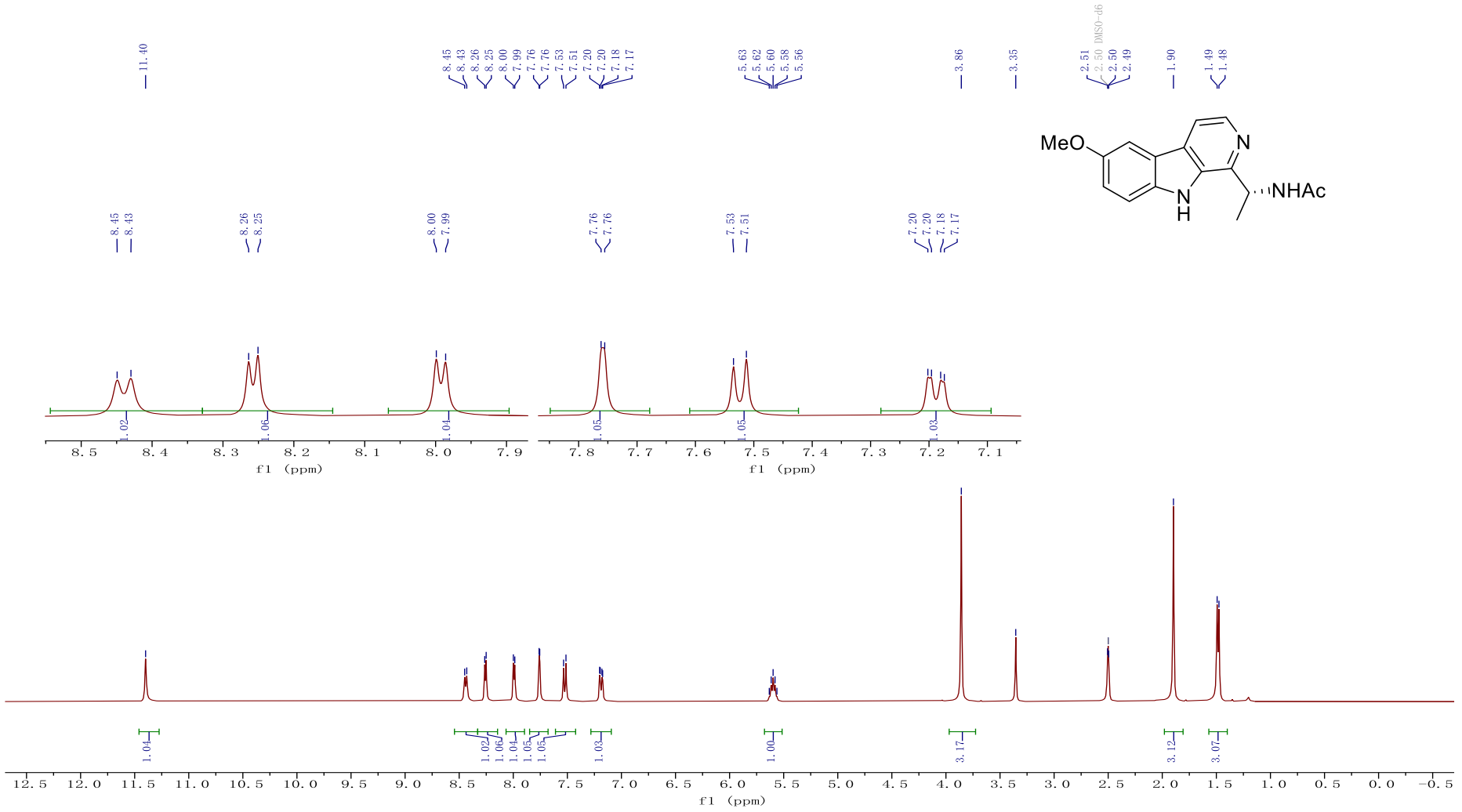
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(4-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3f**)



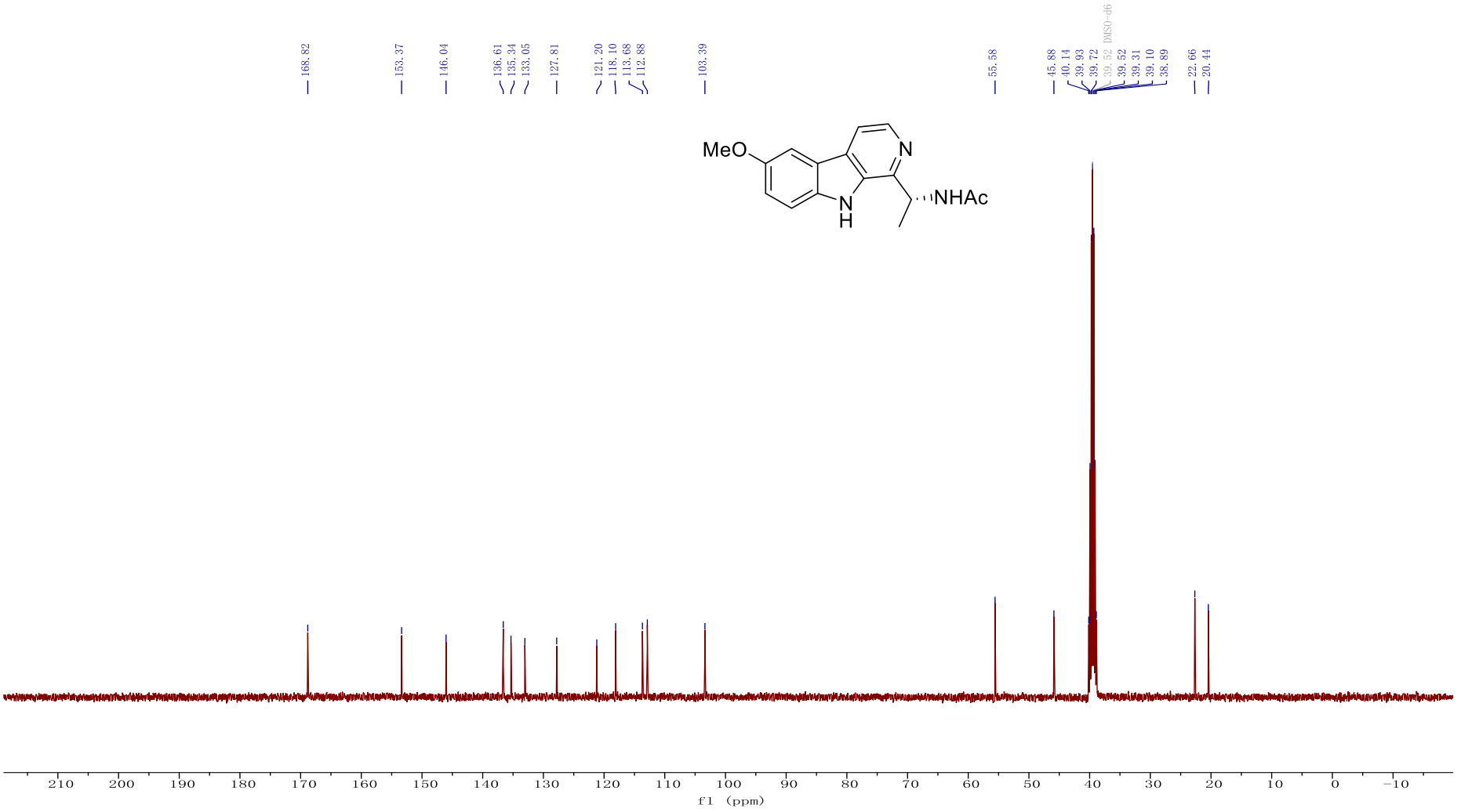
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(4-methyl-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3f**)



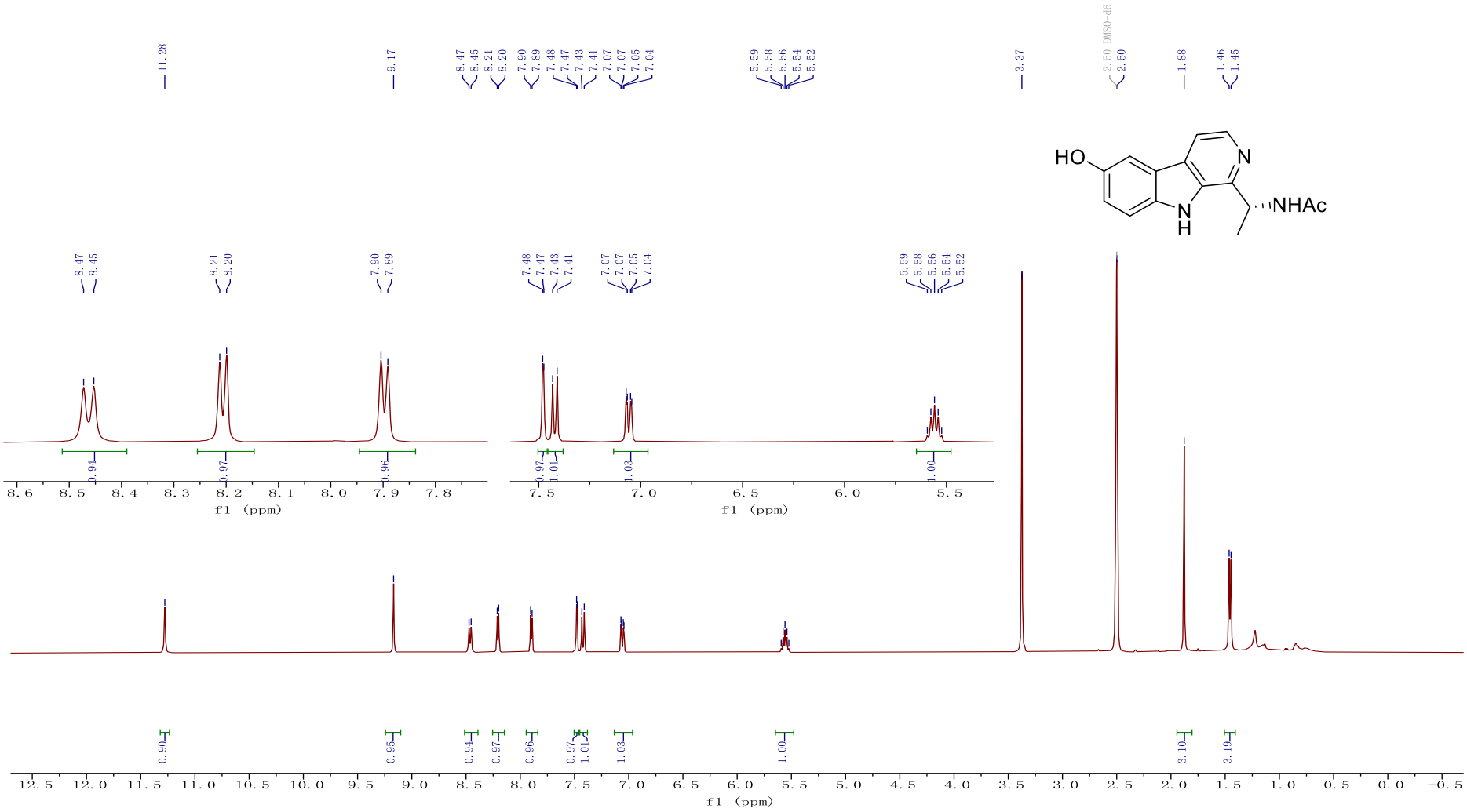
[1]H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3g**)



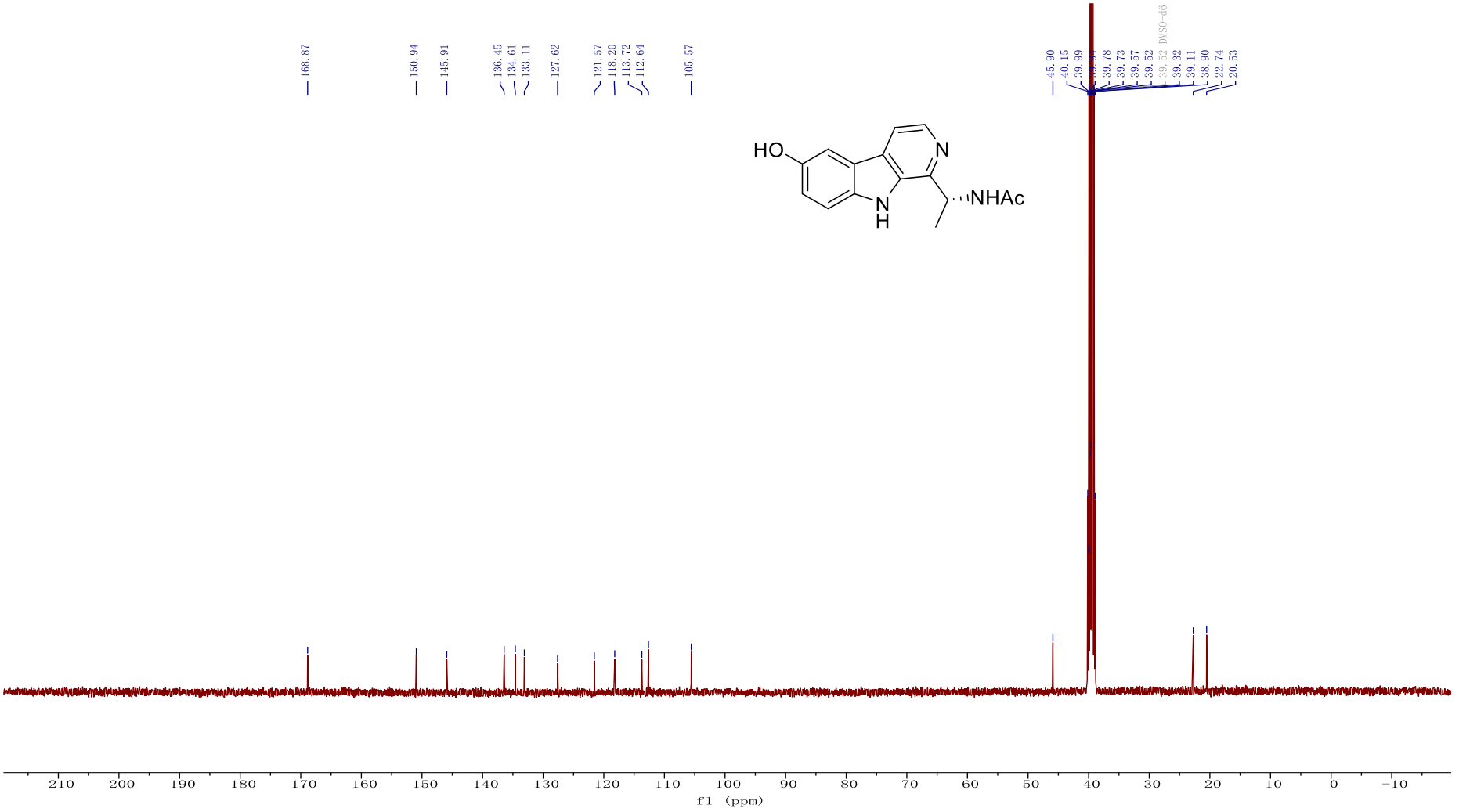
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3g**)



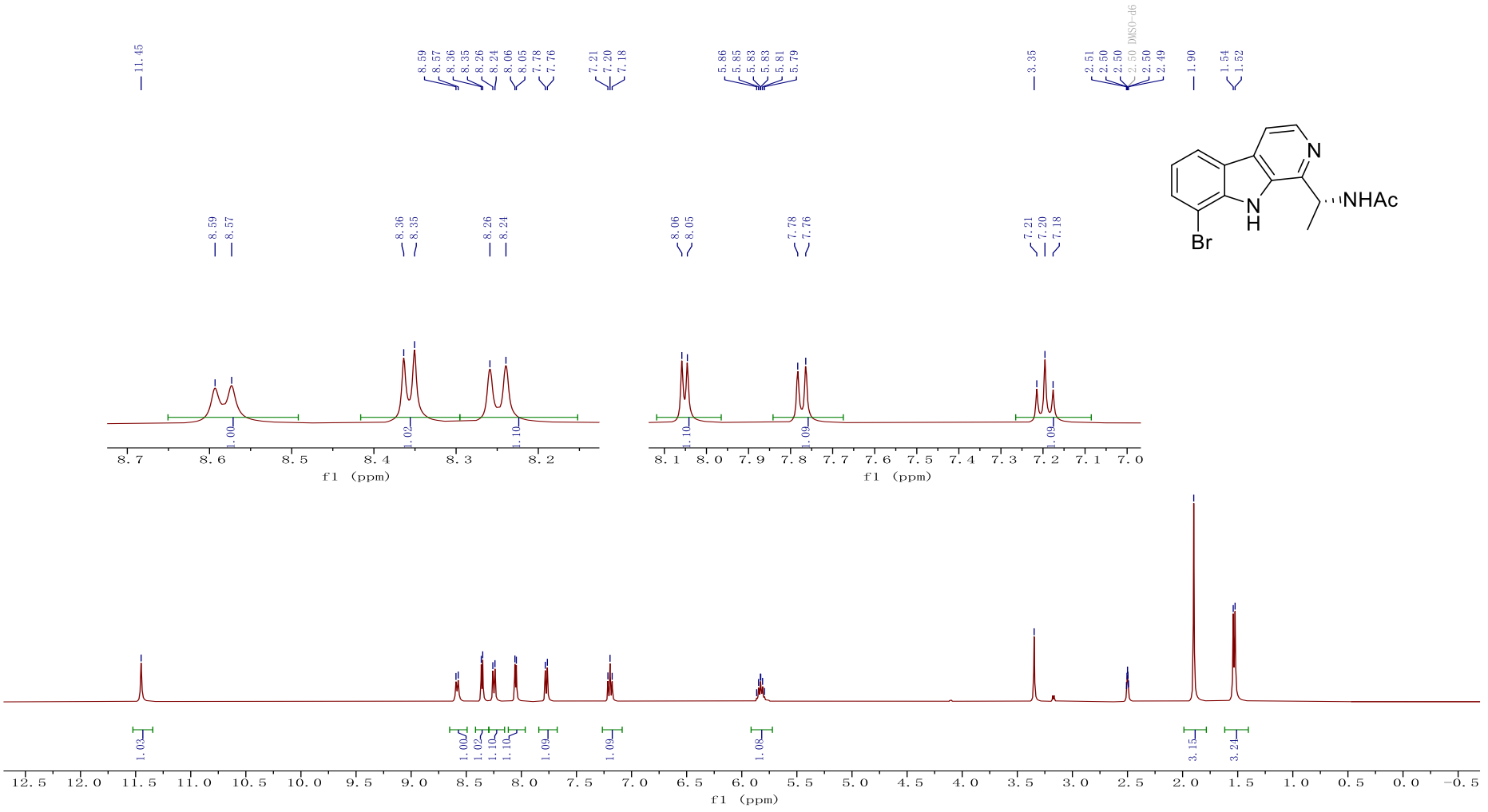
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3h**)



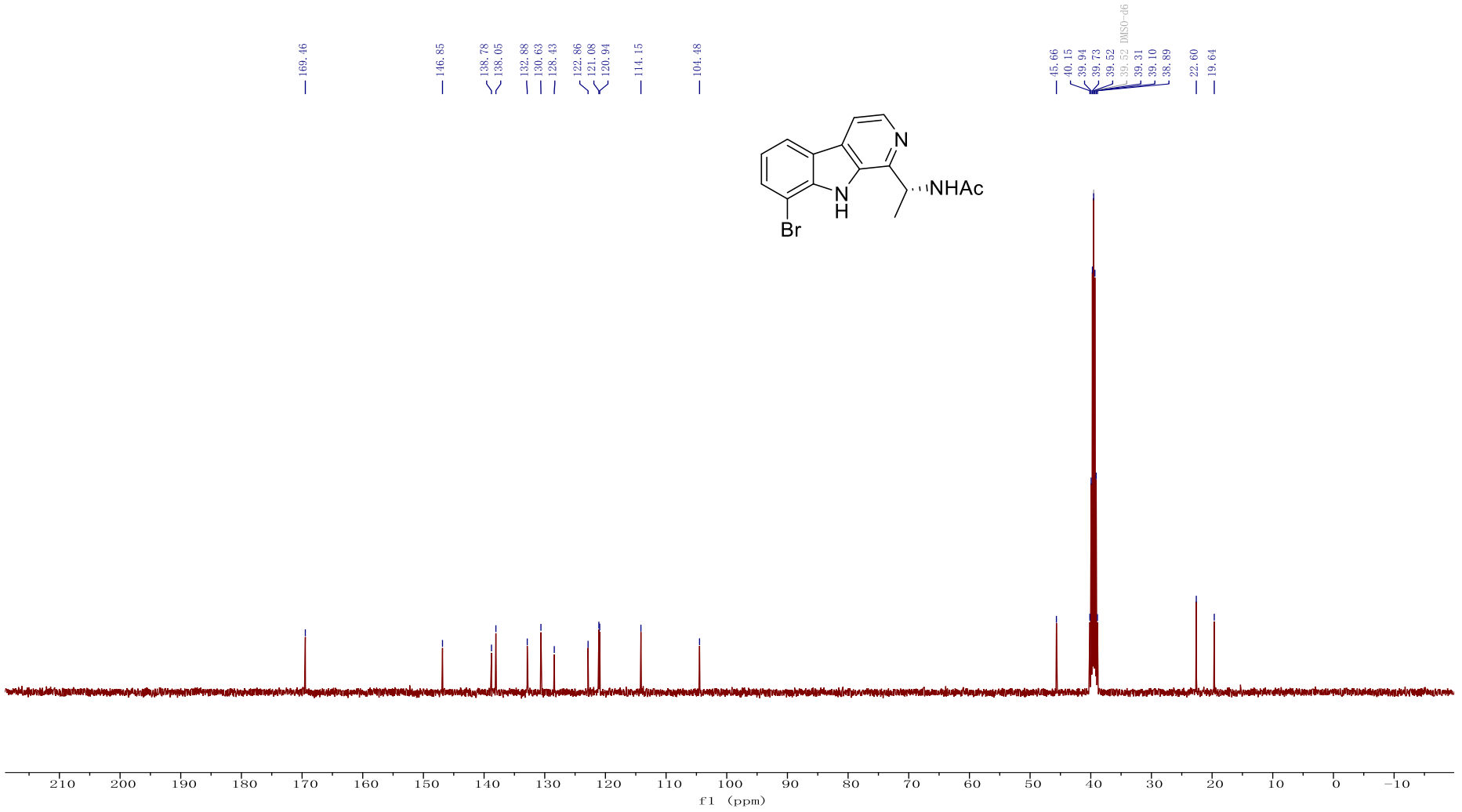
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3h**)



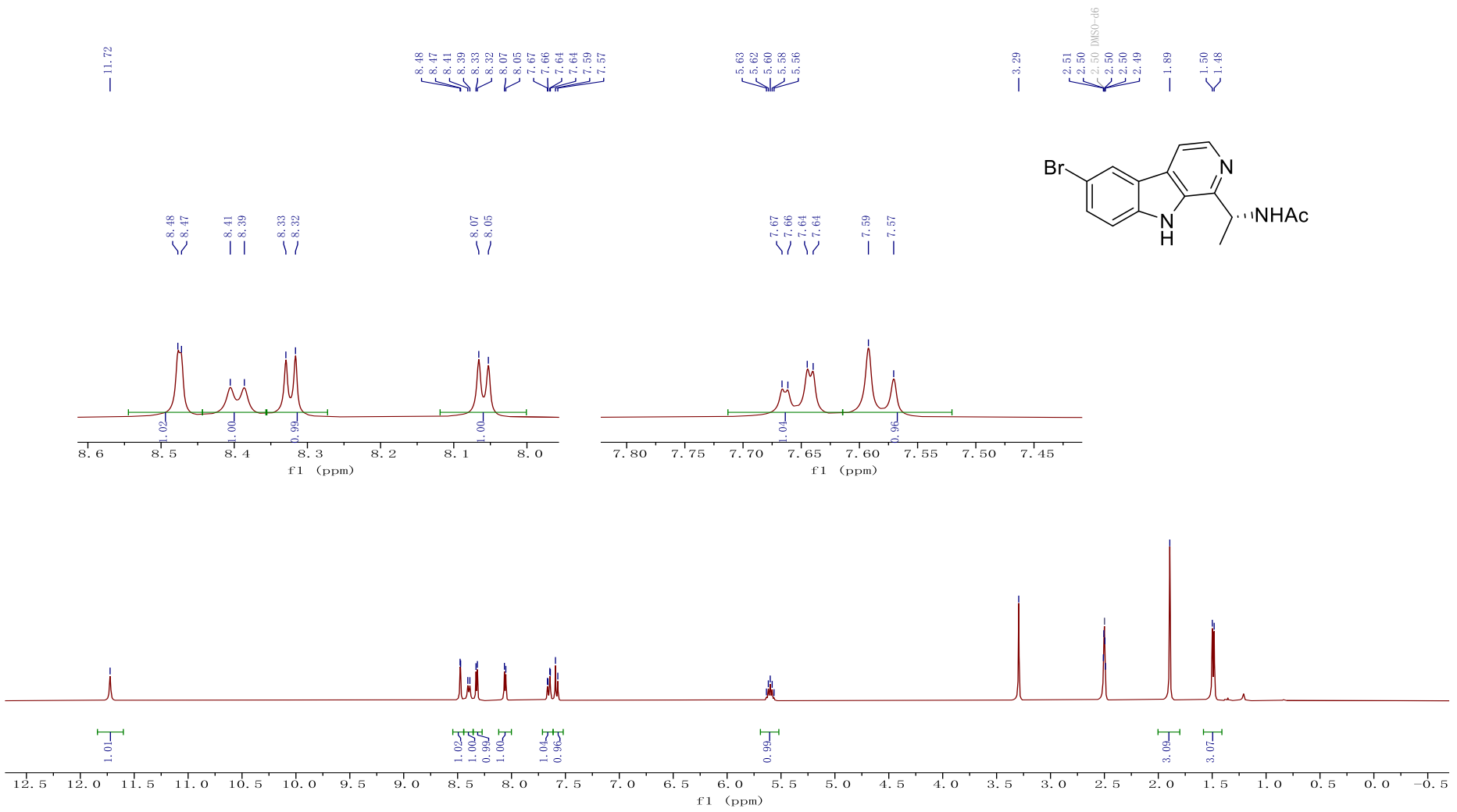
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(8-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3i**)



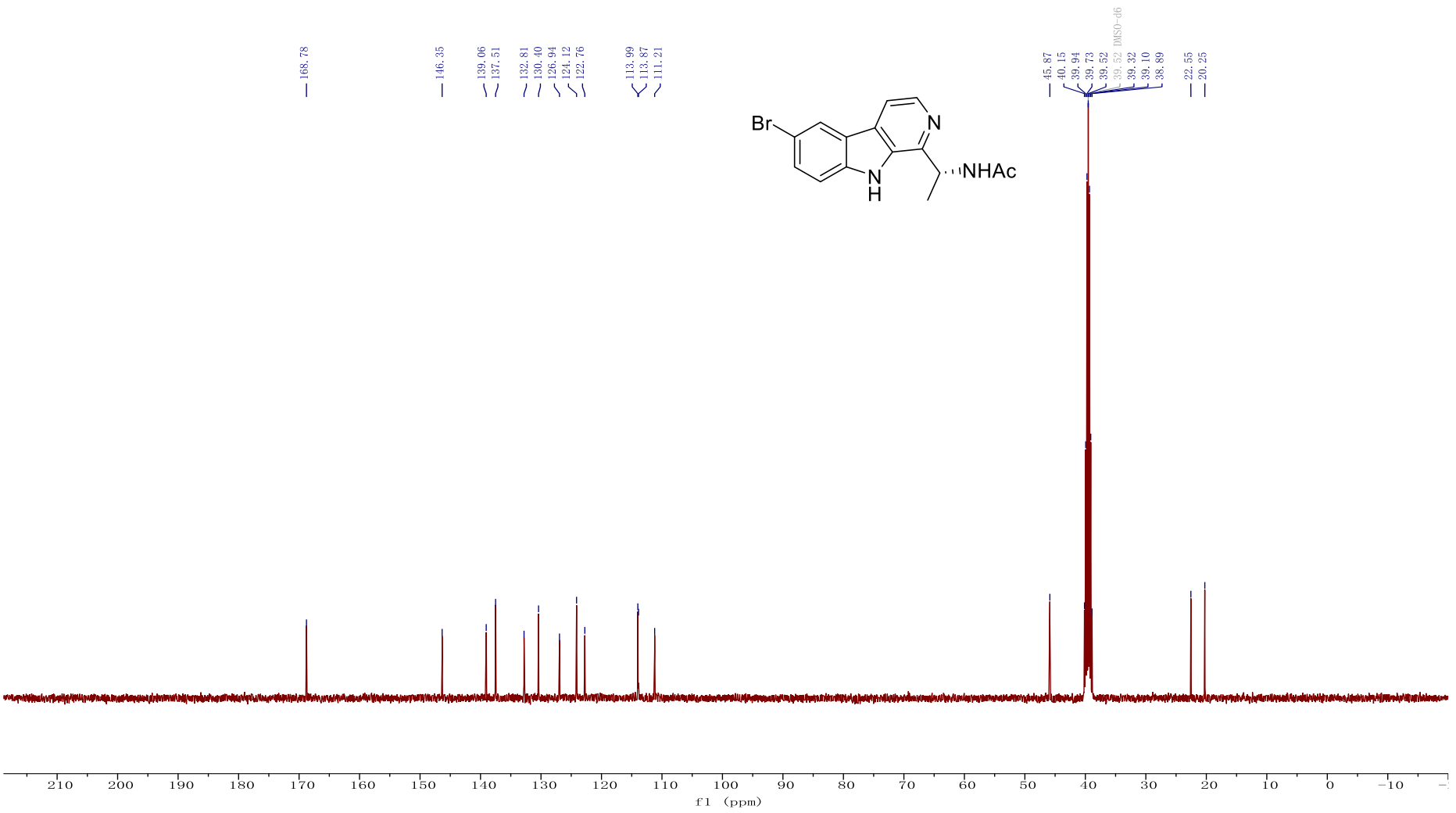
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(8-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3i**)



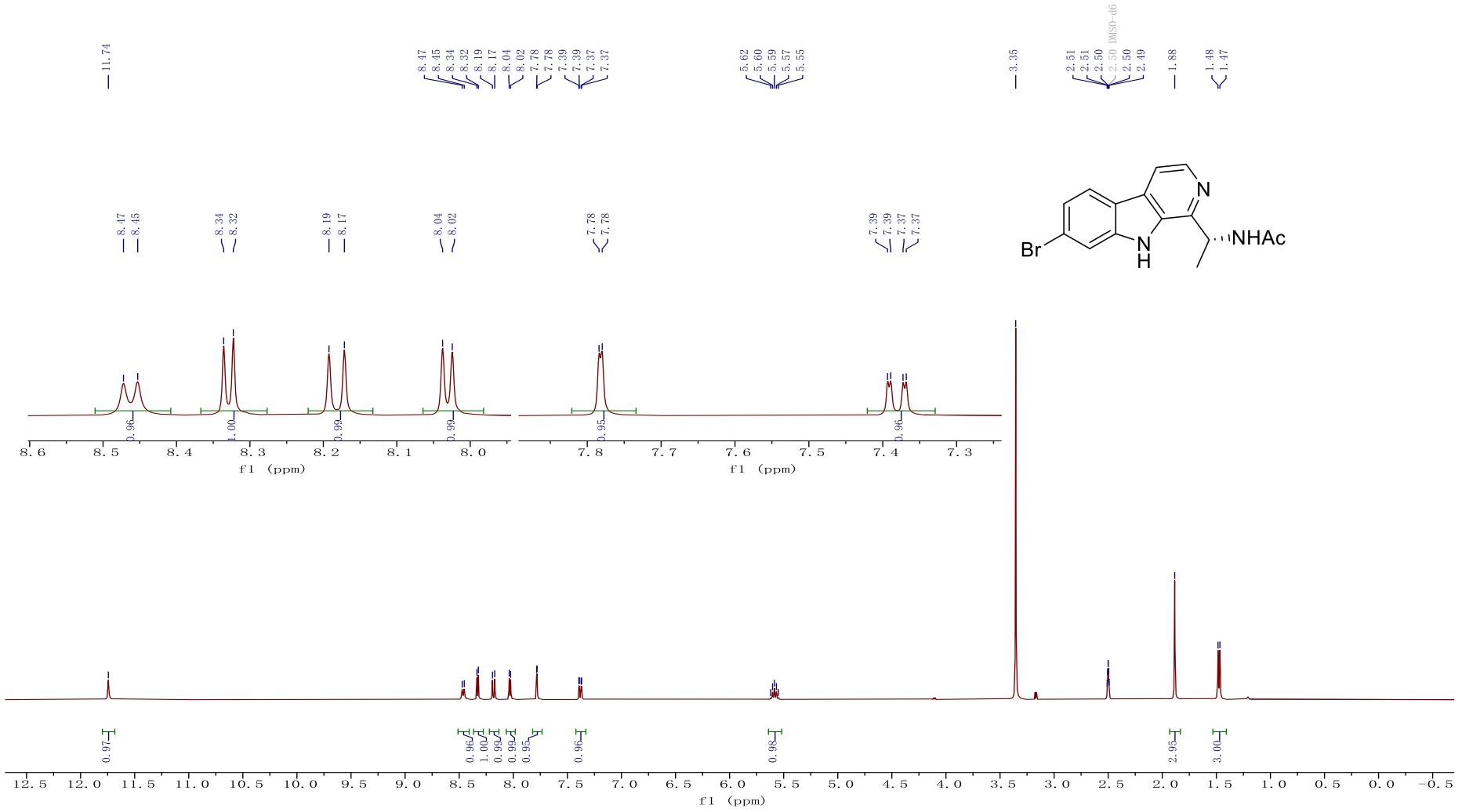
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3j**)



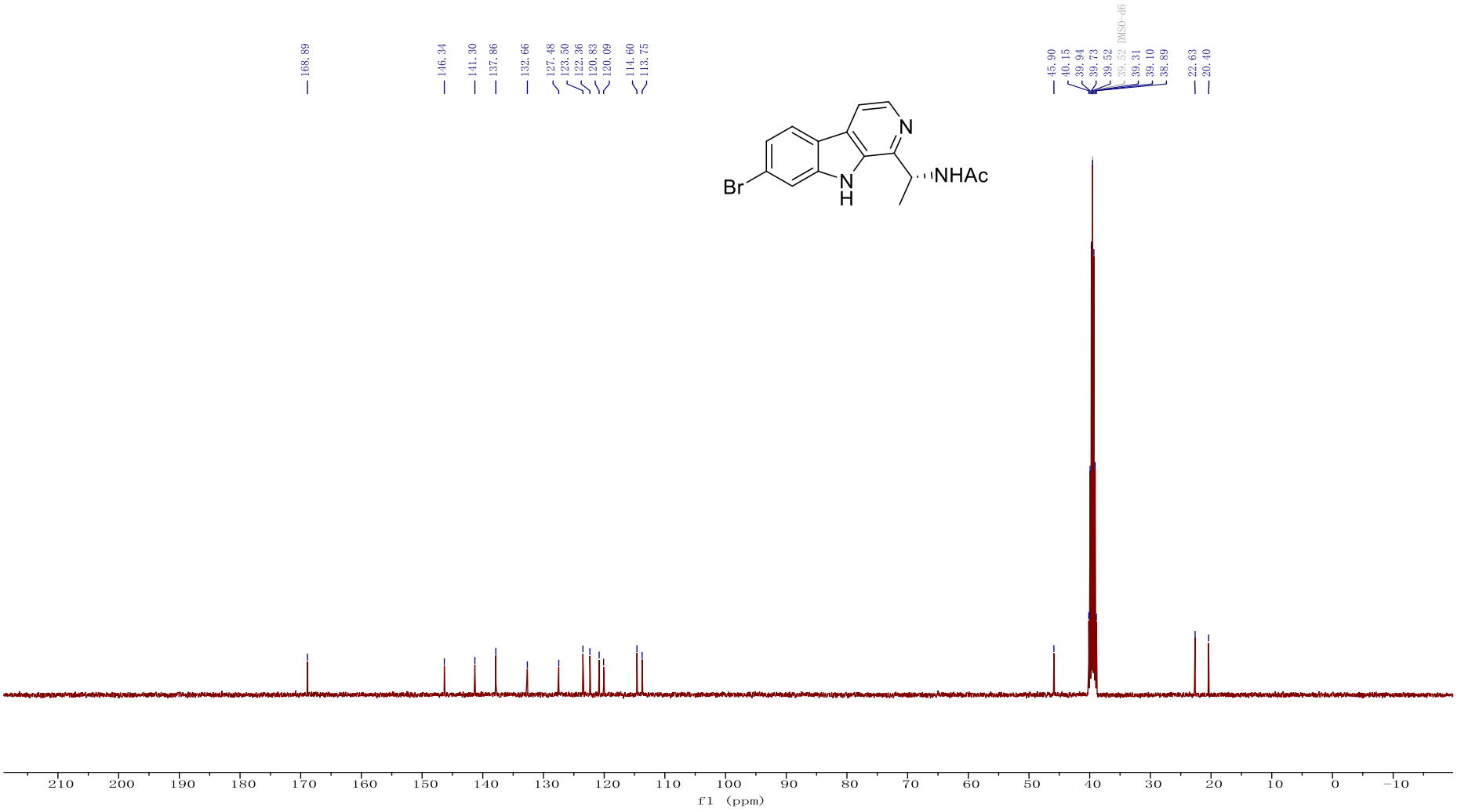
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3j**)



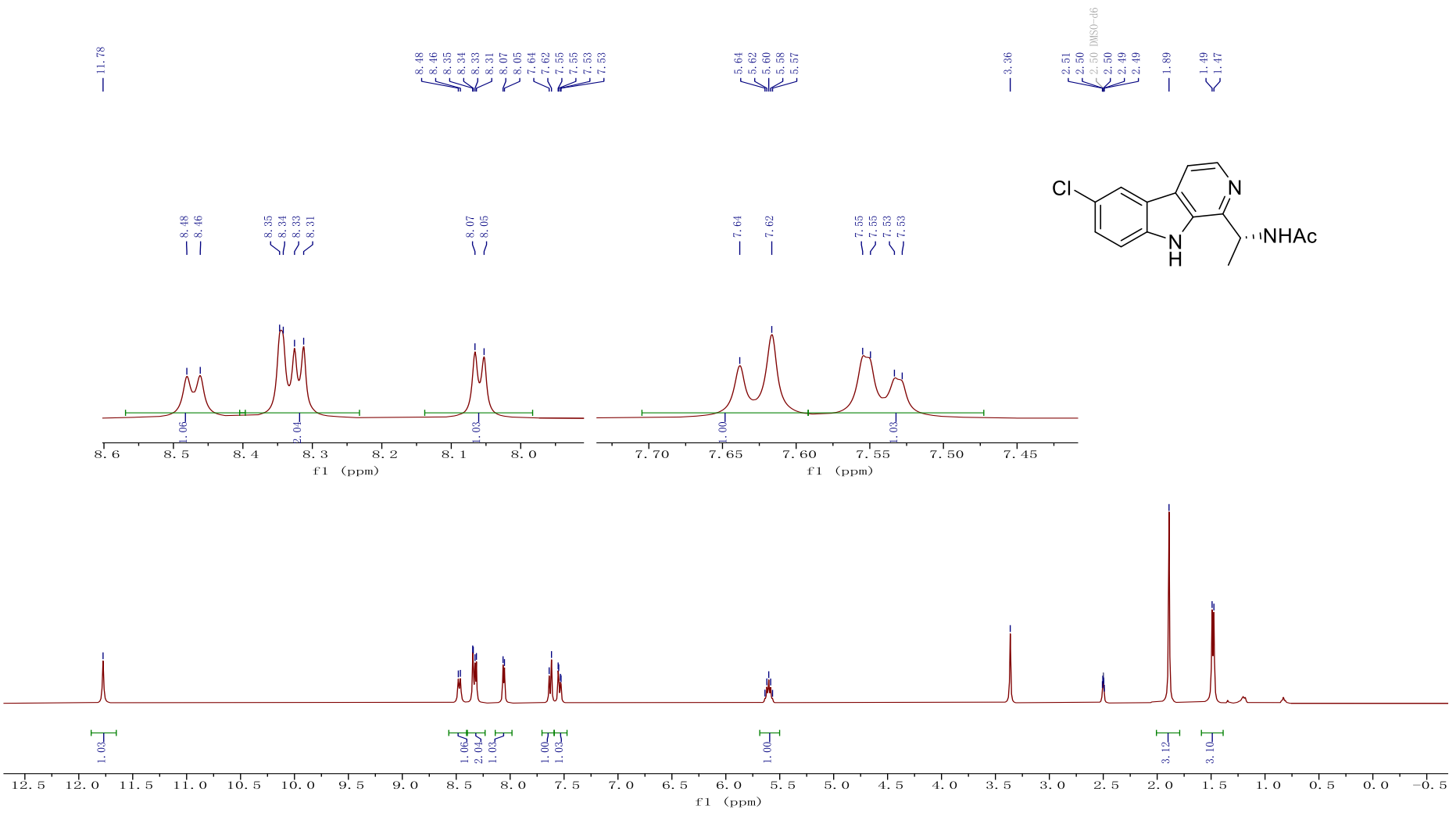
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3k**)



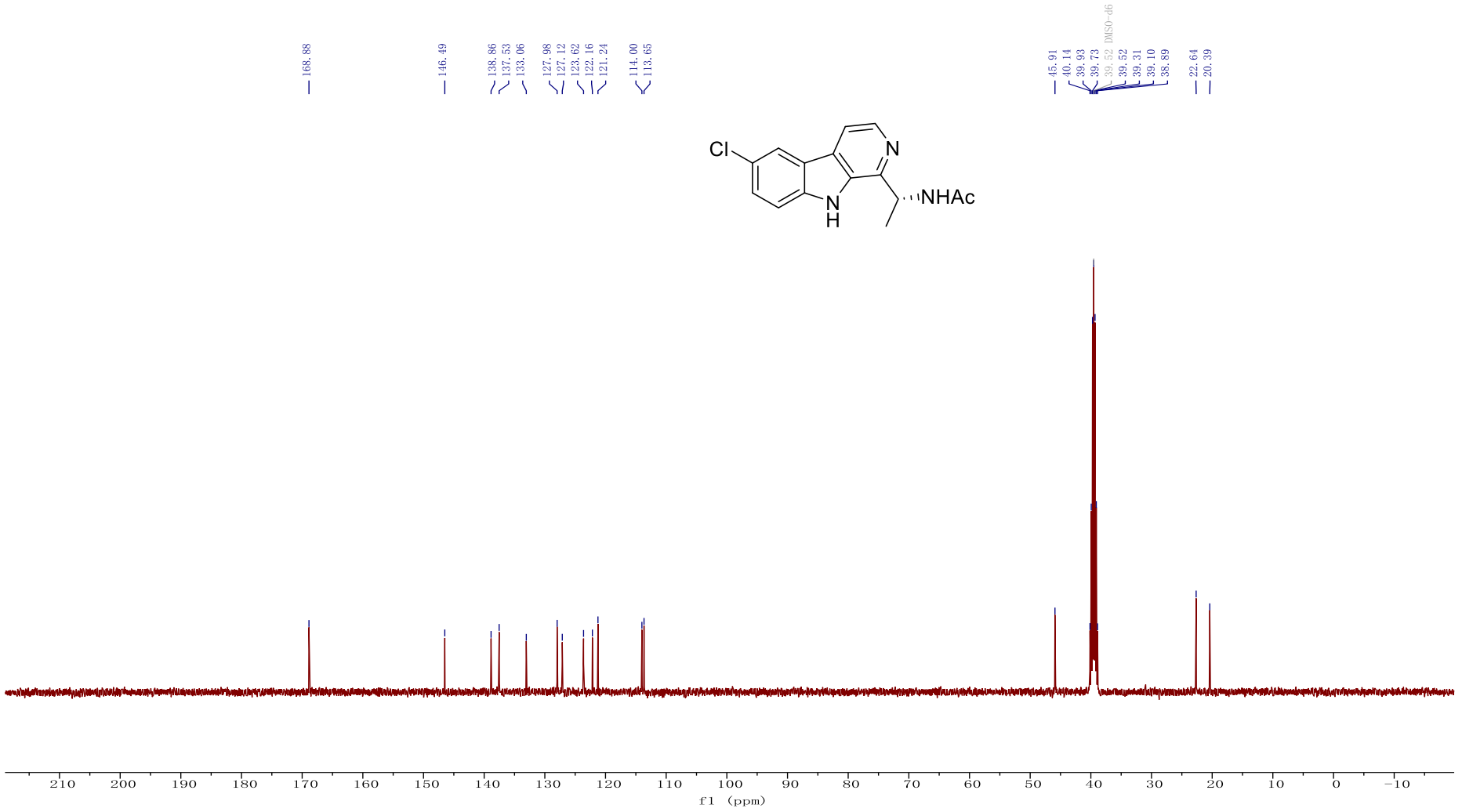
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3k**)



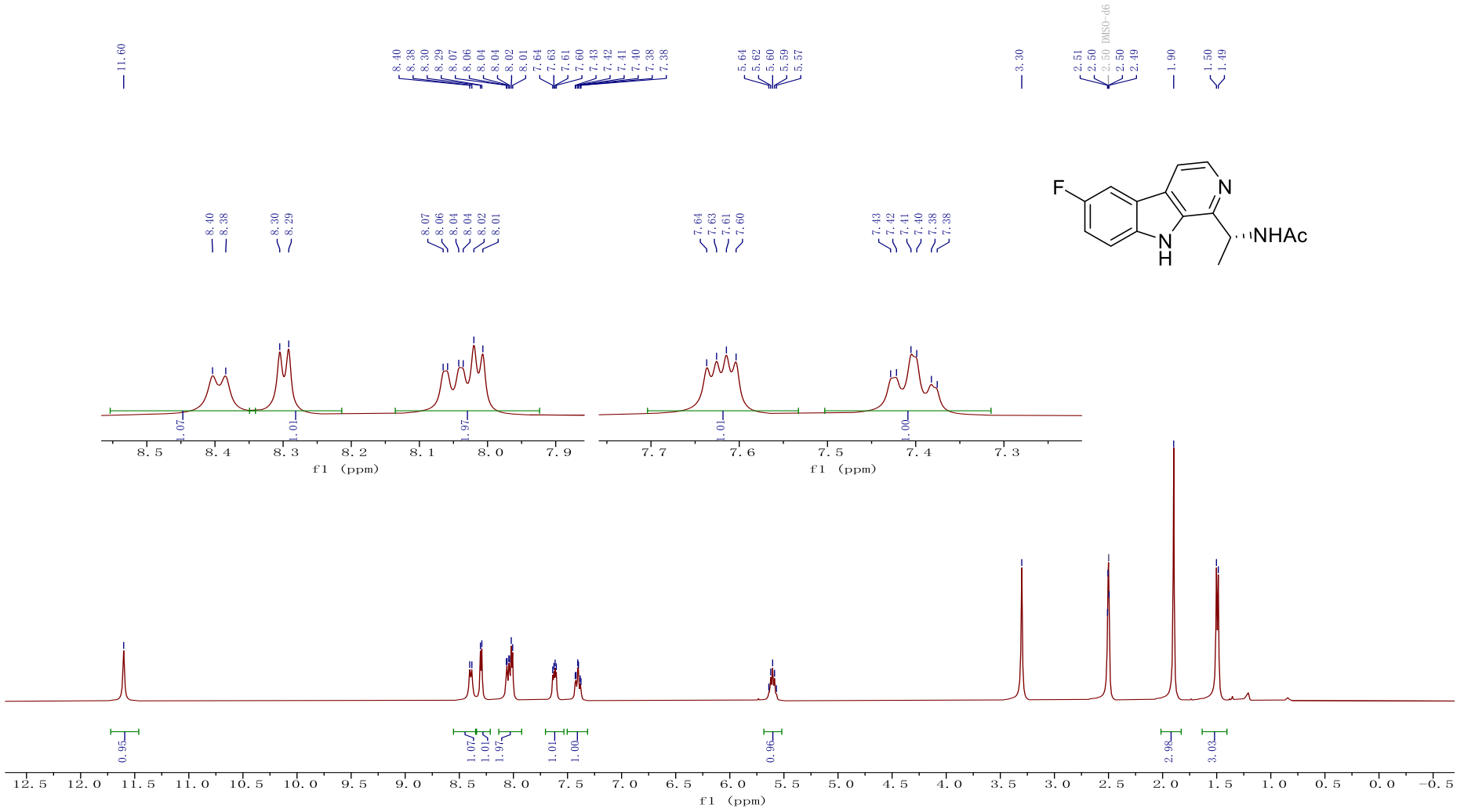
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-chloro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3l**)



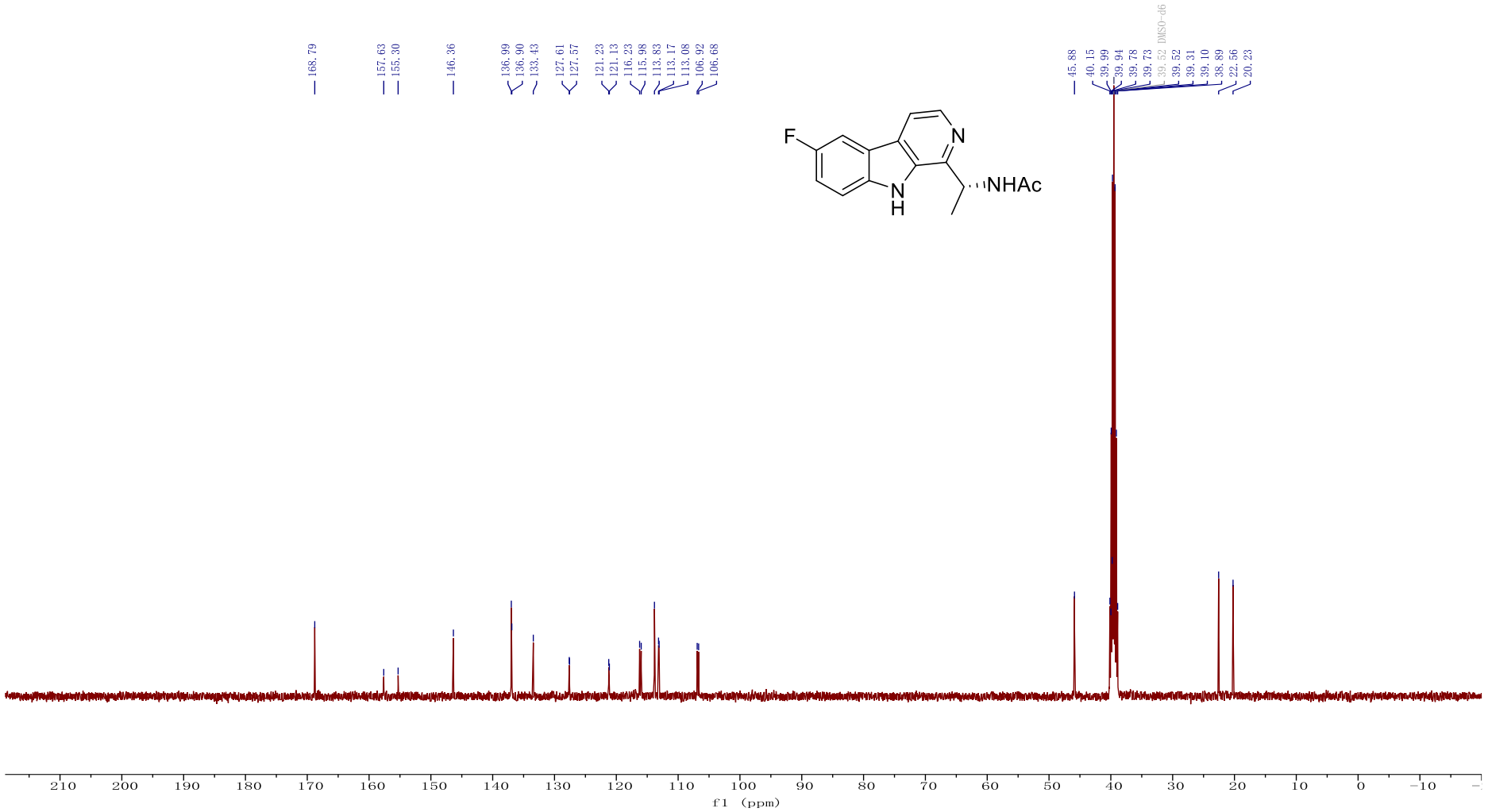
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-chloro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3l**)



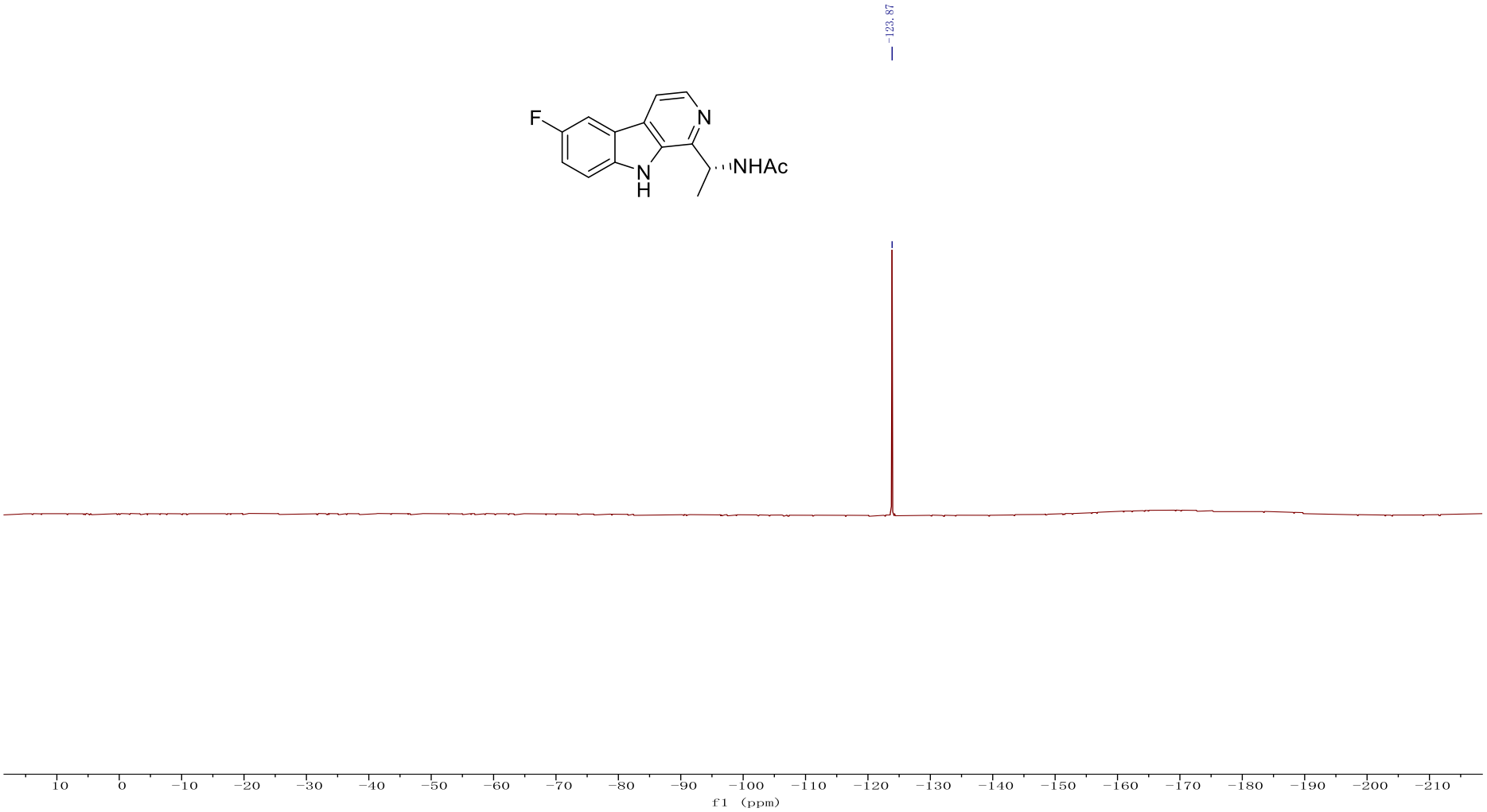
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-fluoro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3m**)



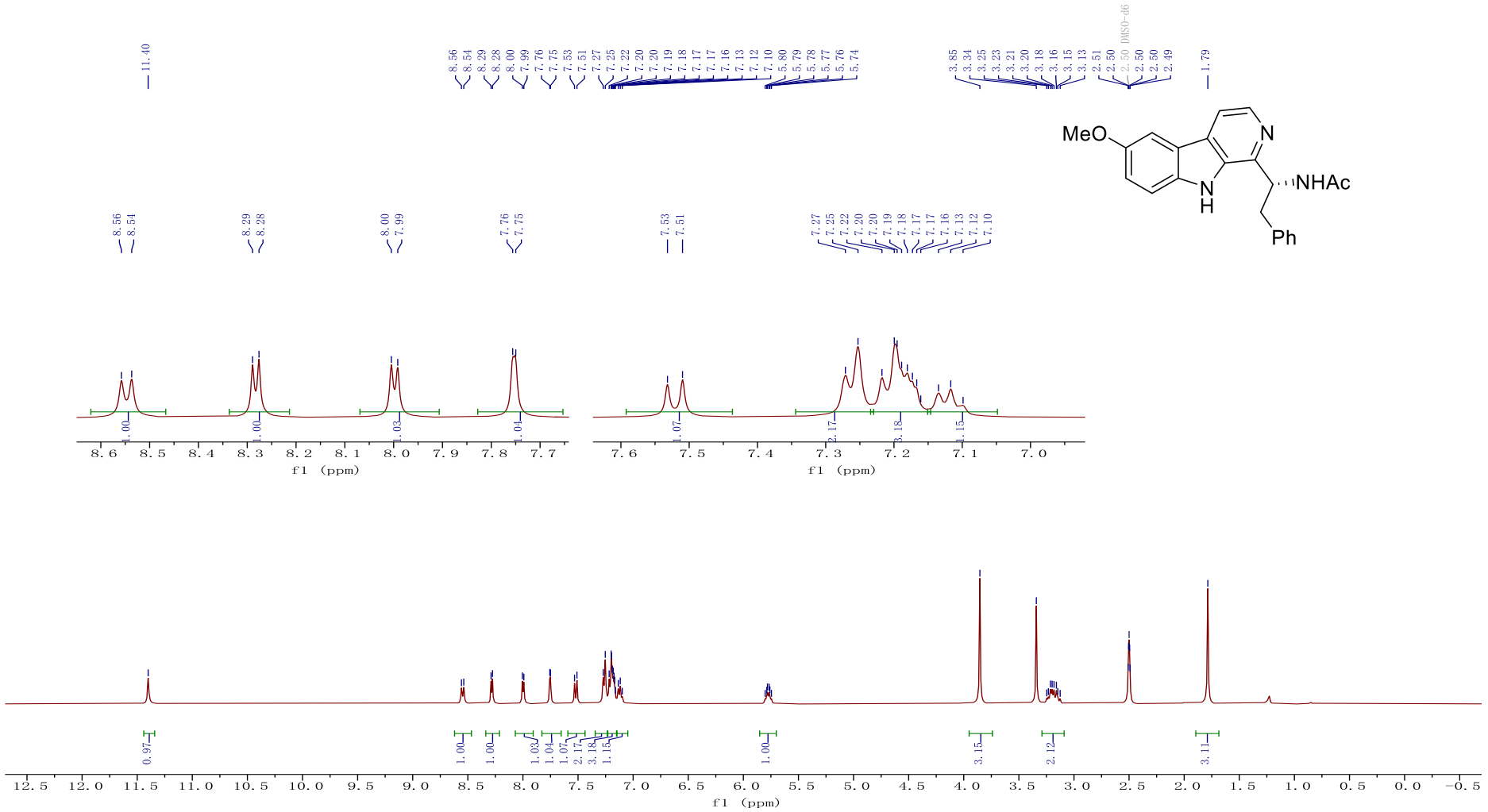
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-fluoro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3m**)



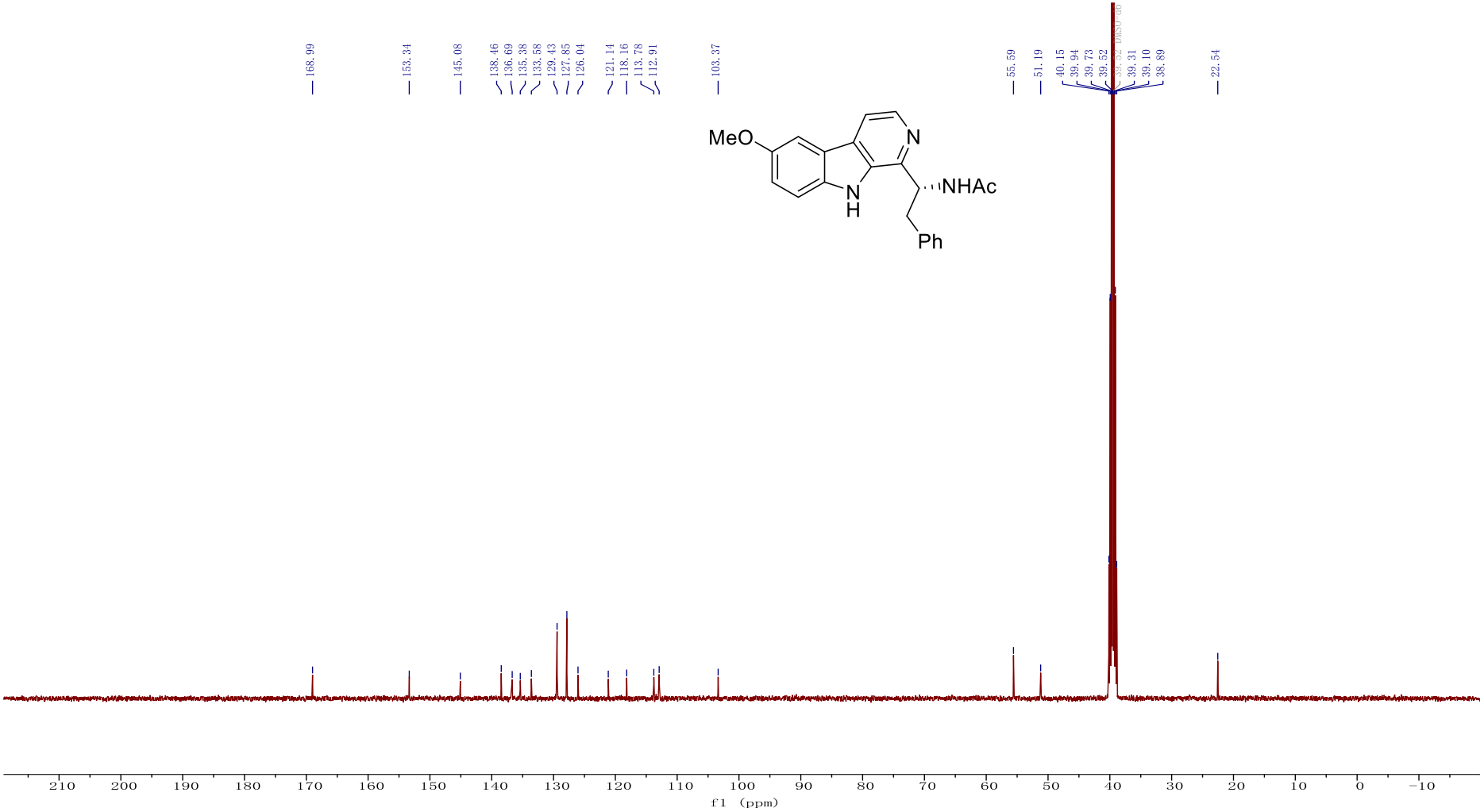
^{19}F NMR (376 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-fluoro-9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**3m**)



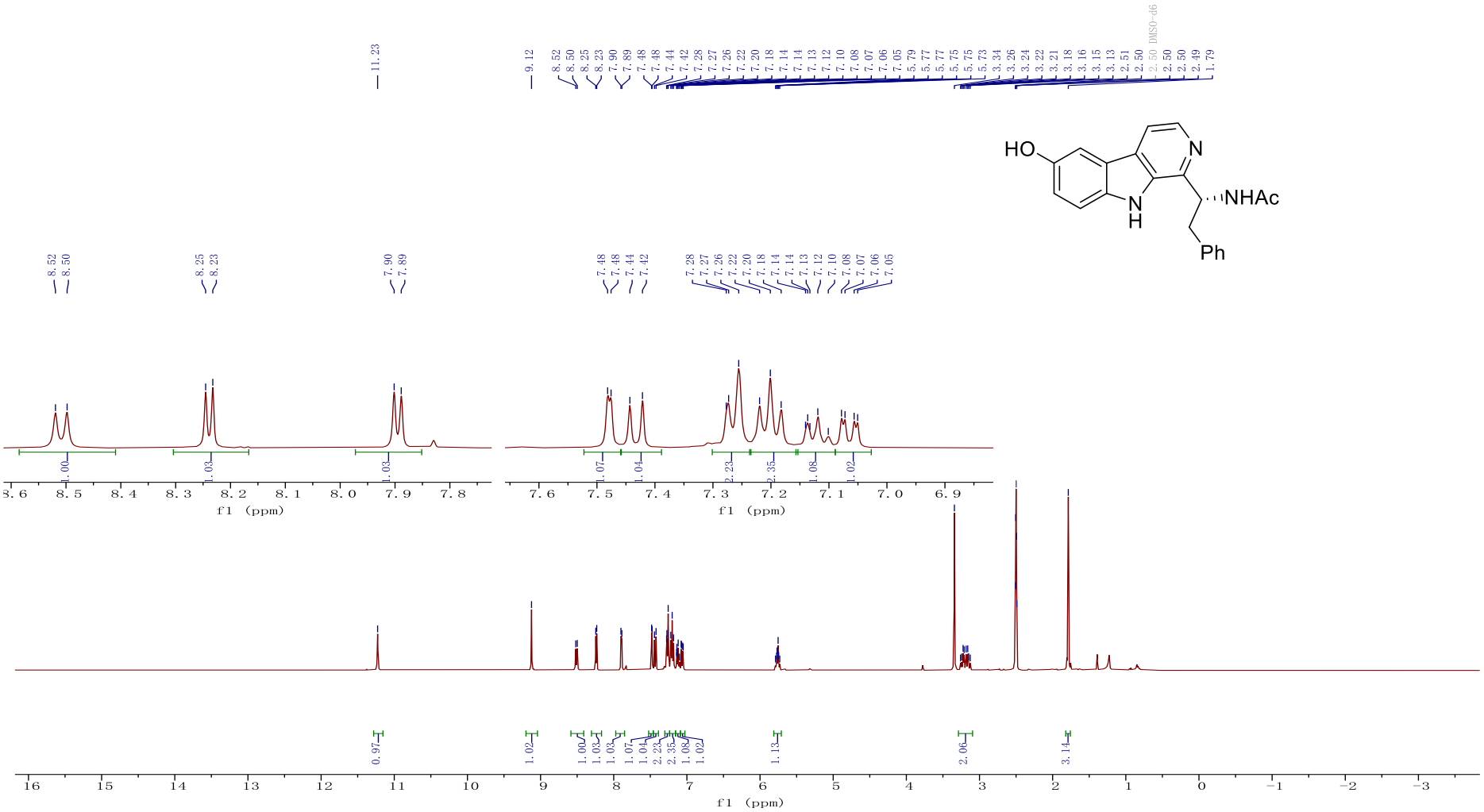
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3n**)



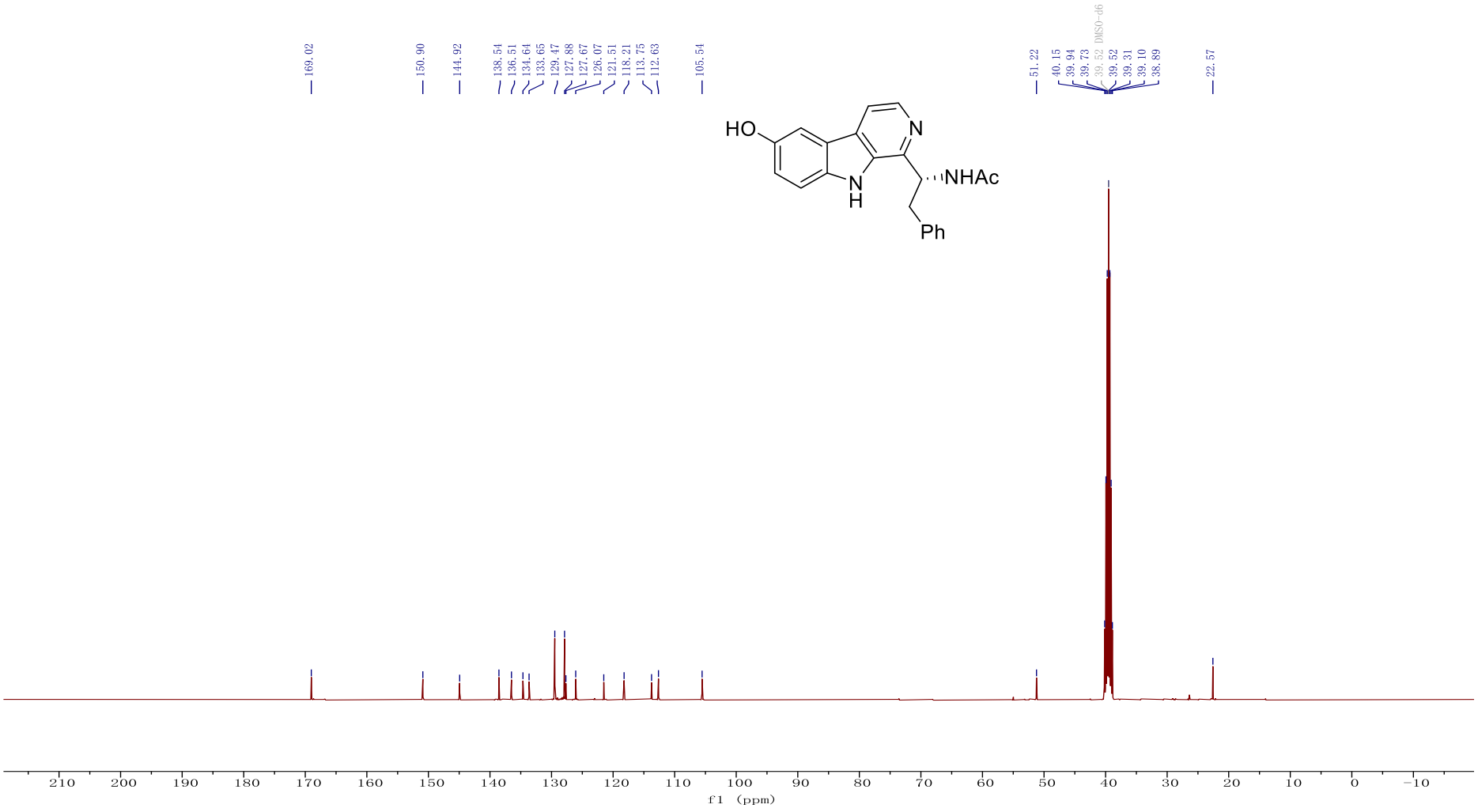
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3n**)



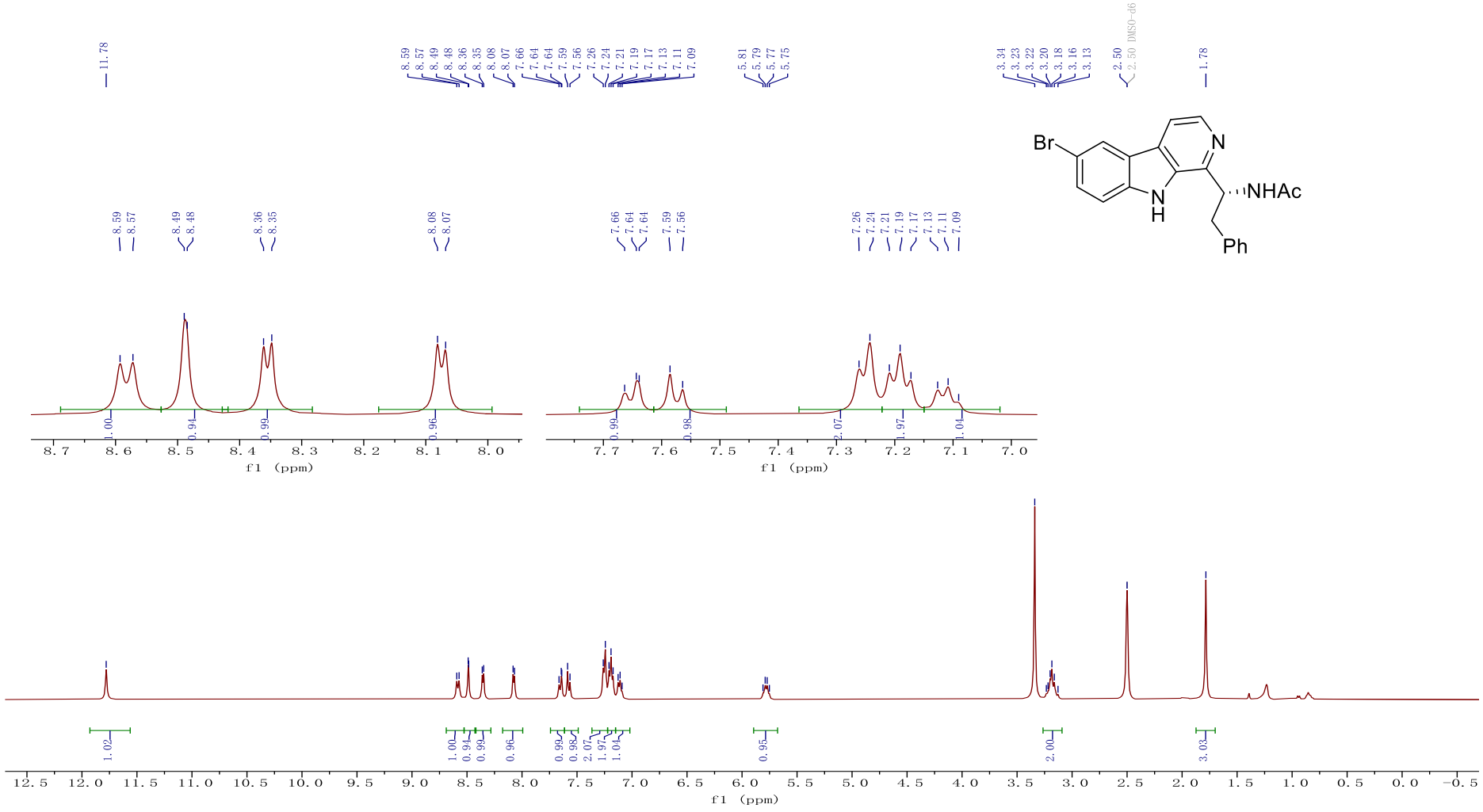
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3o**)



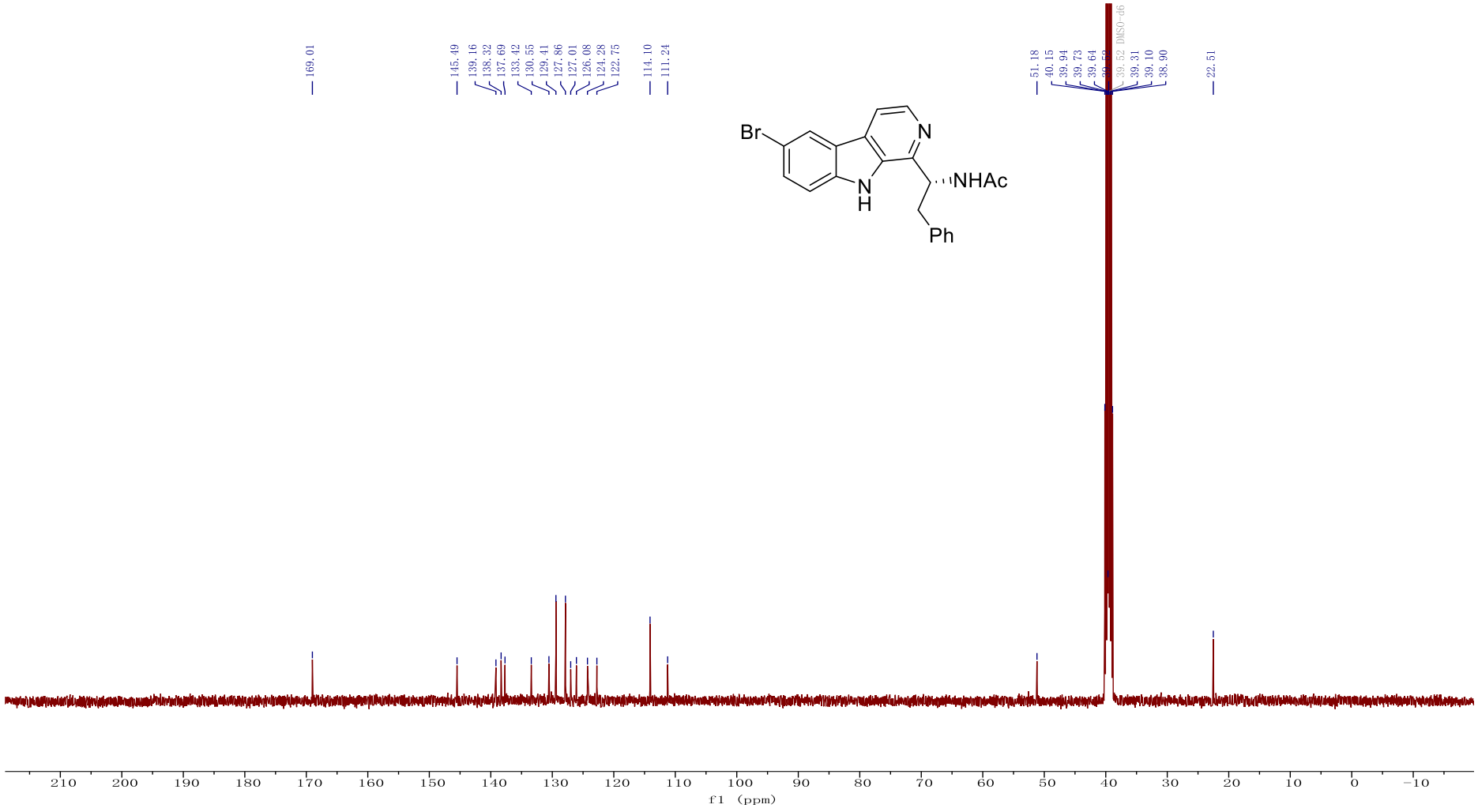
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-hydroxy-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3o**)



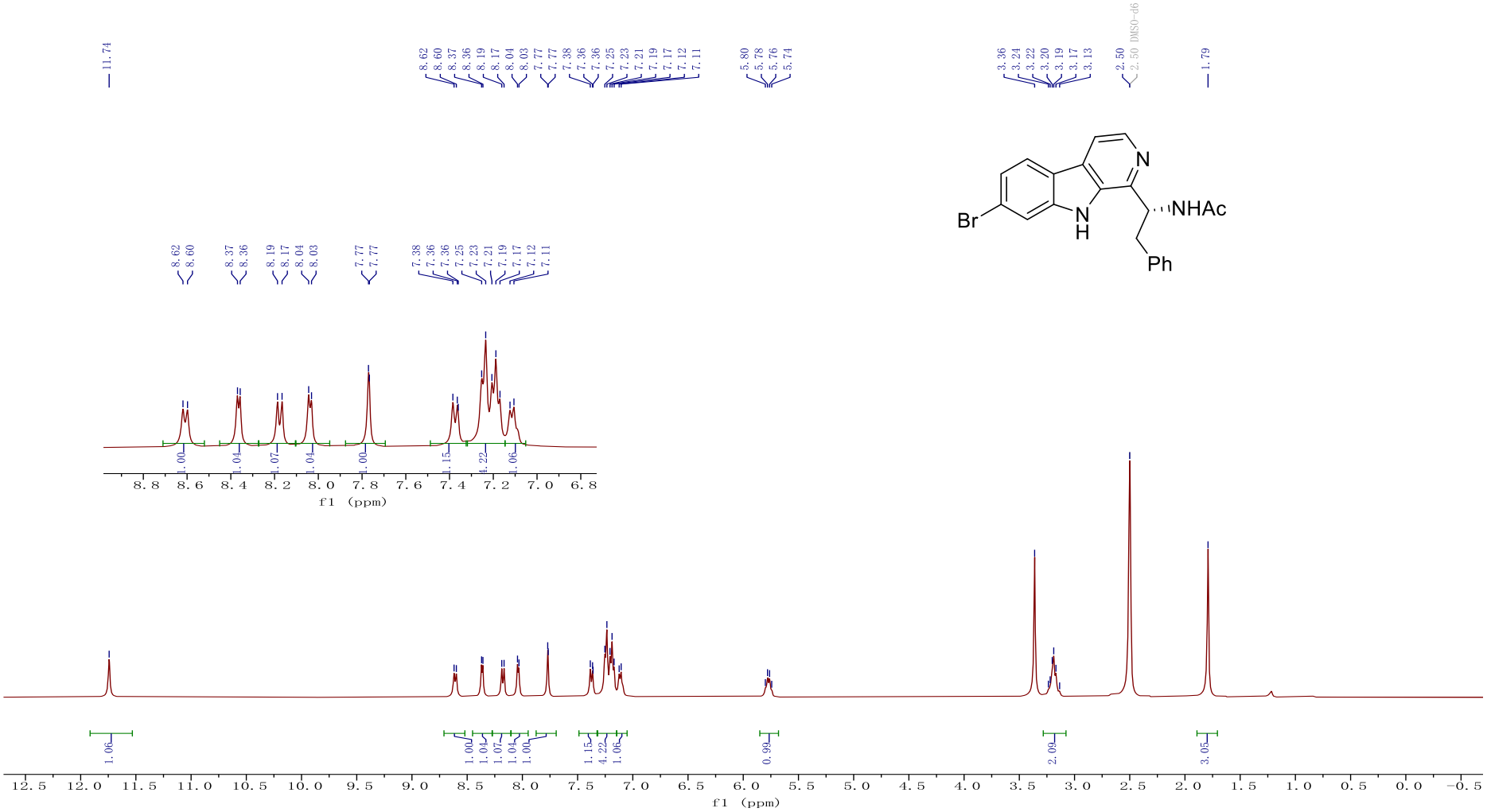
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3p**)



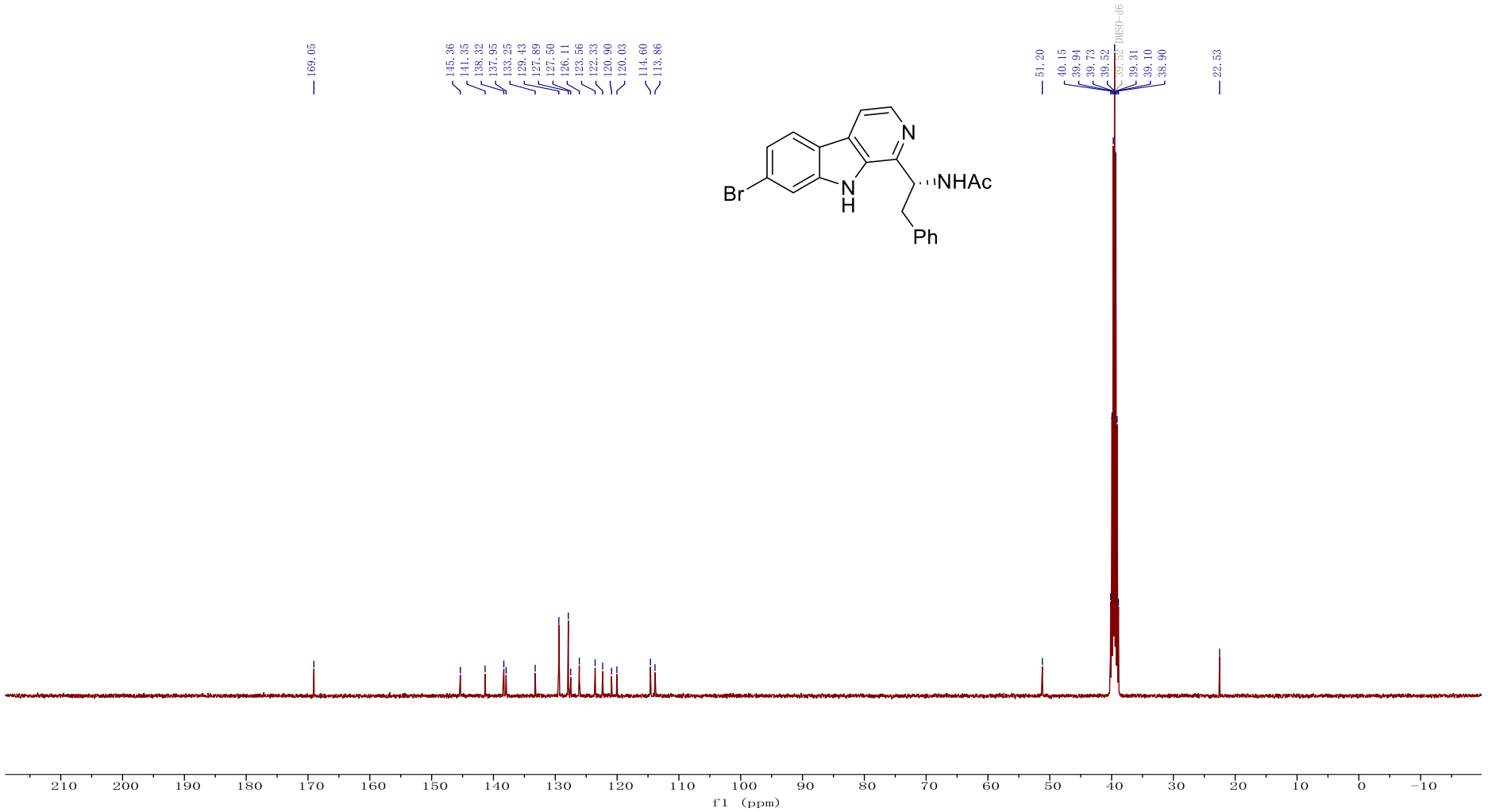
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3p**)



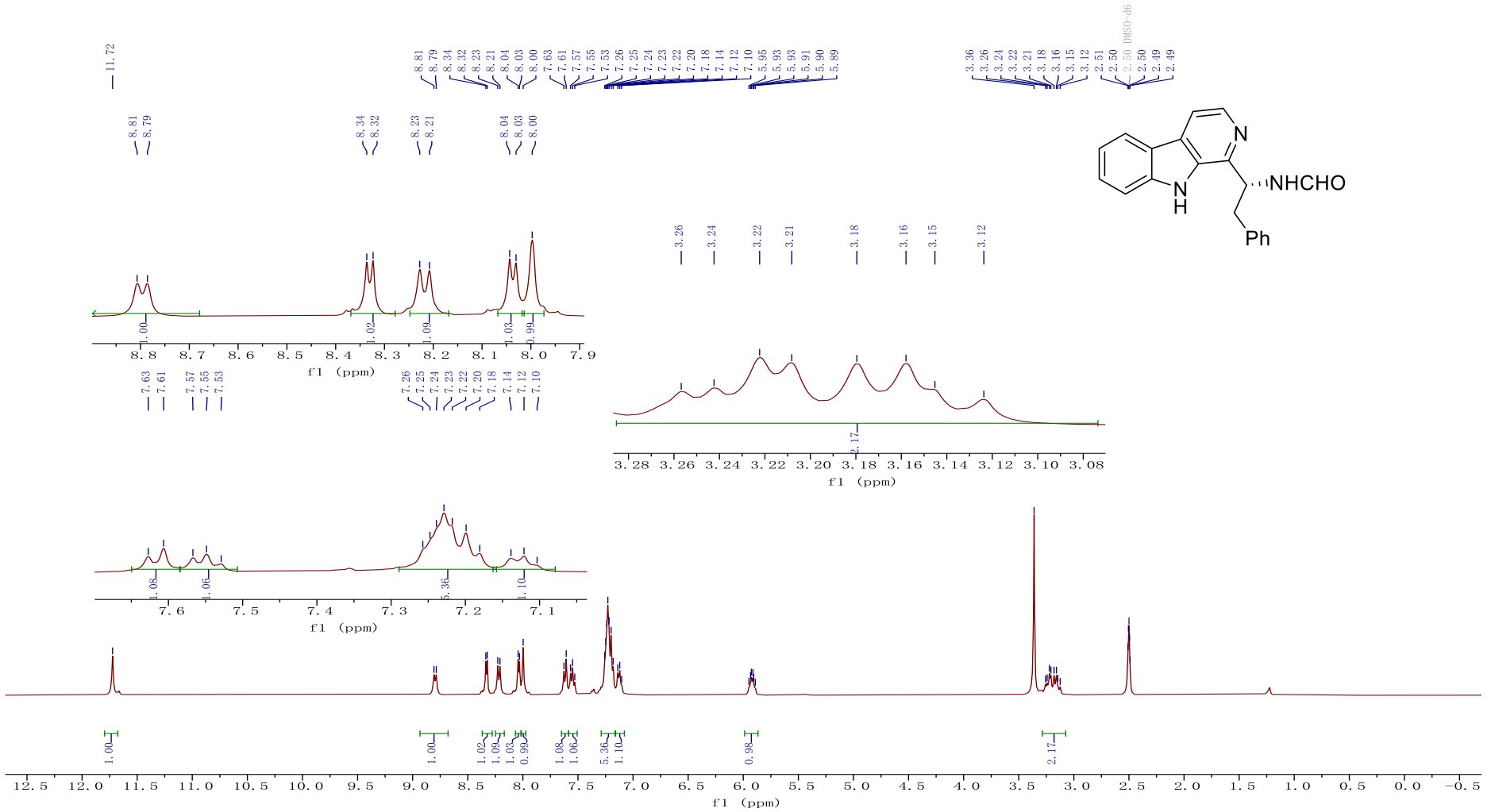
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3q**)



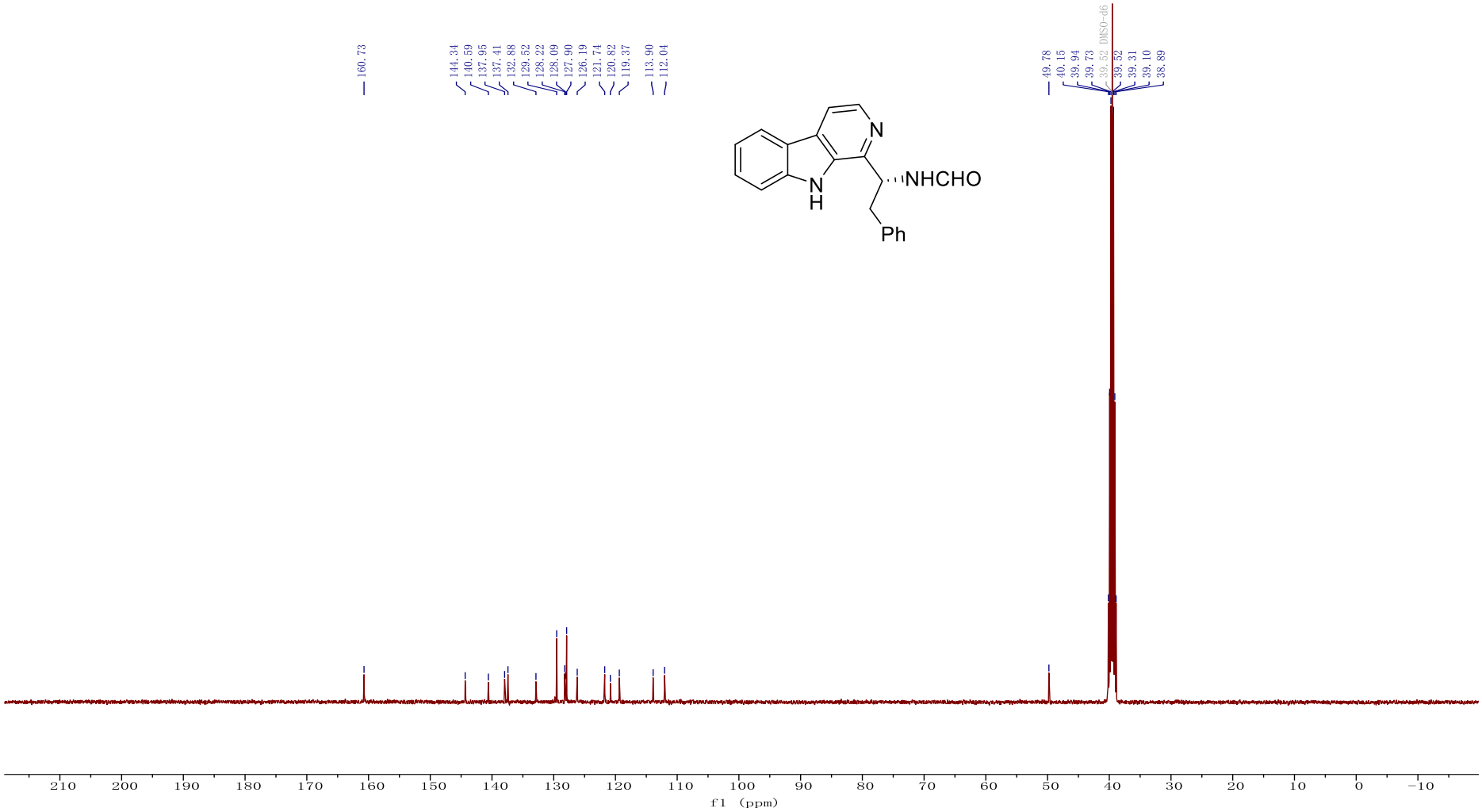
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(7-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)acetamide (**3q**)



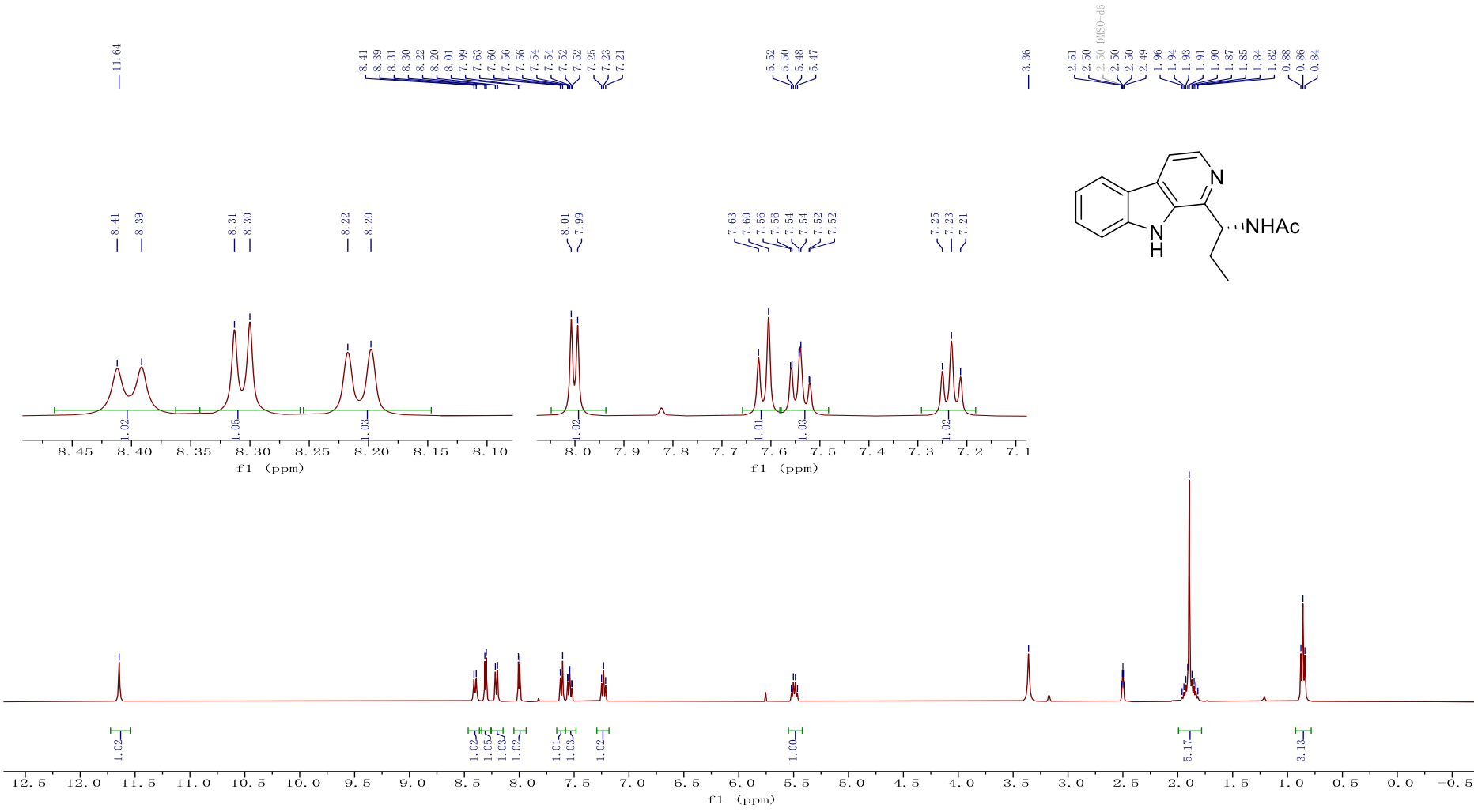
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3r**)



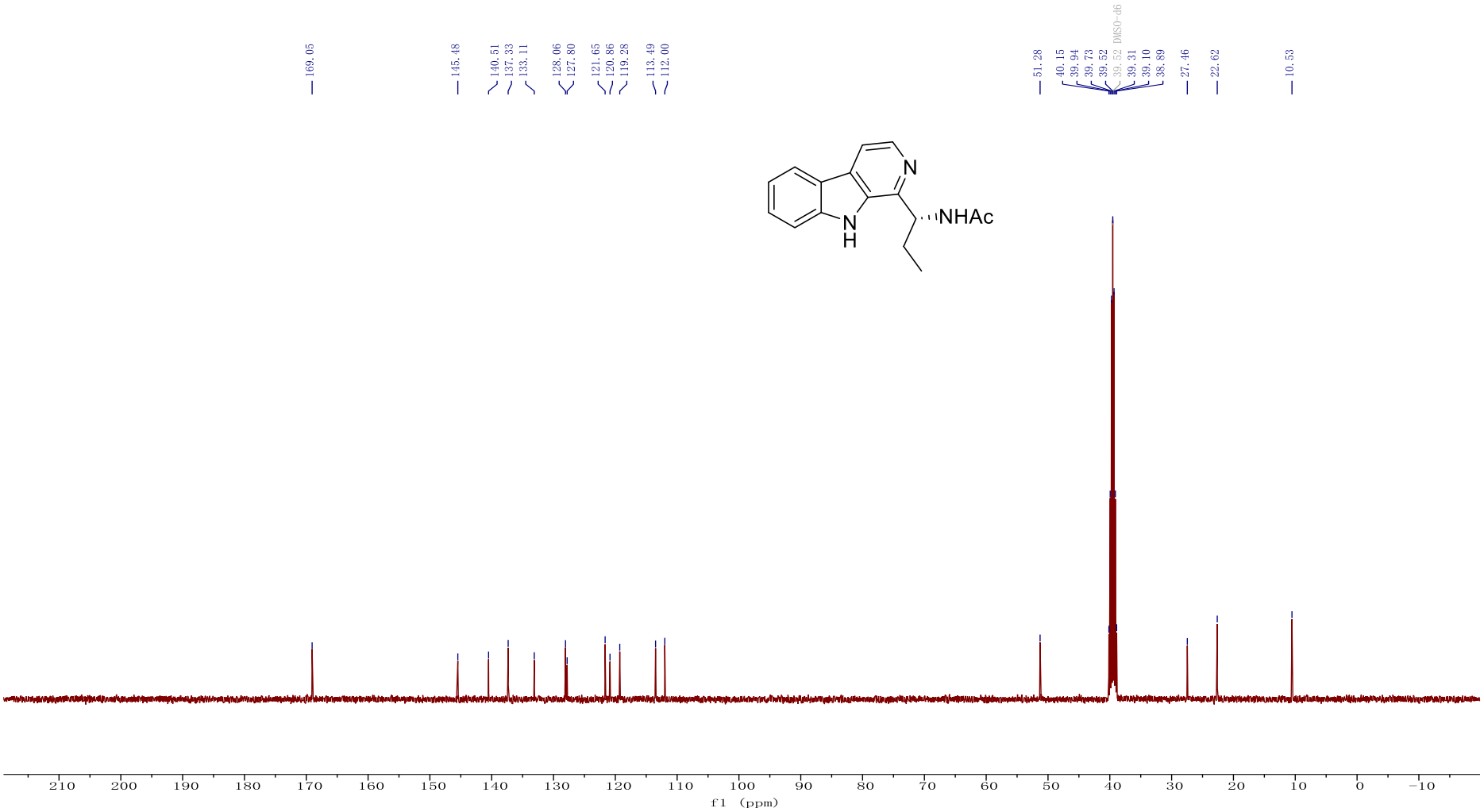
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)formamide (**3r**)



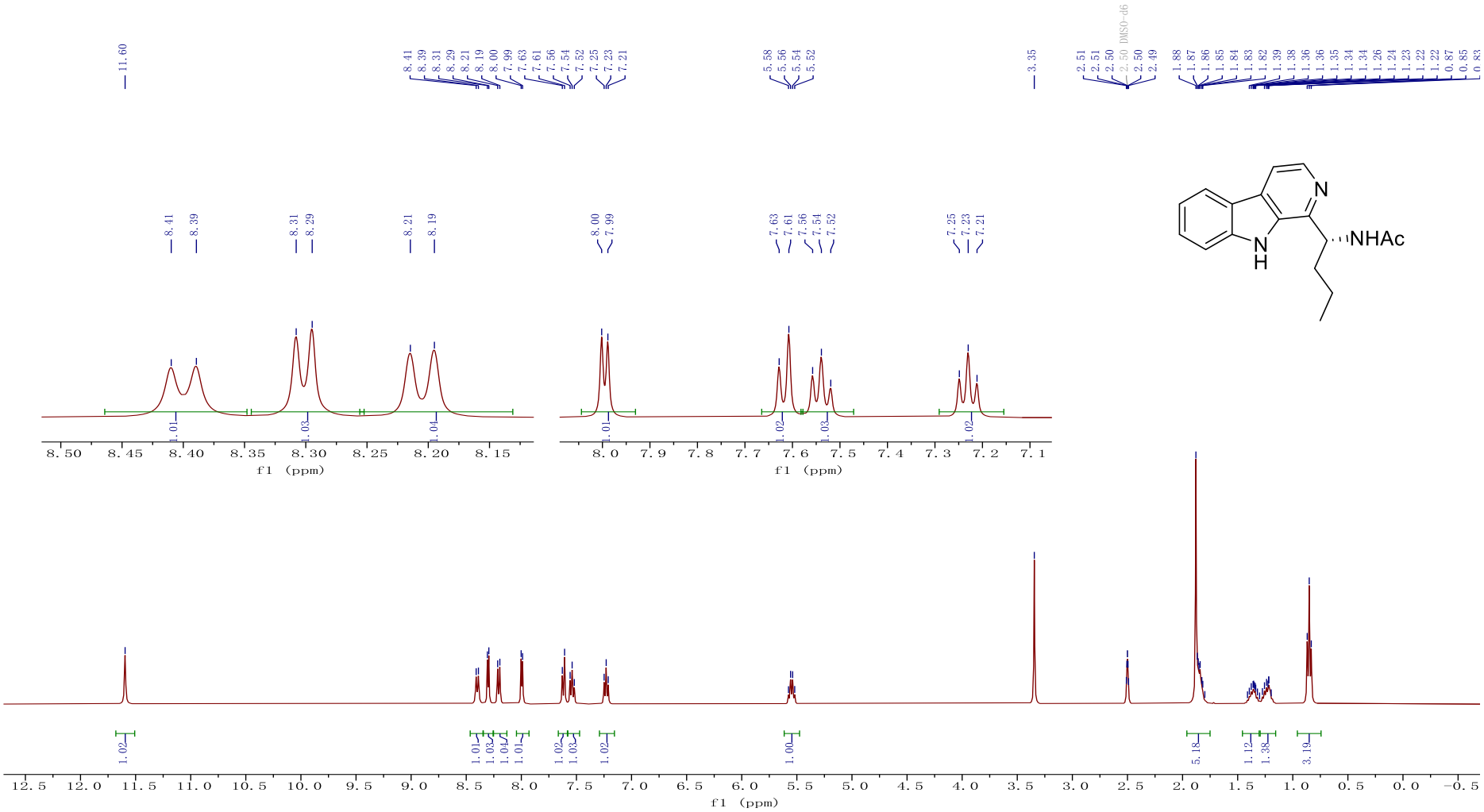
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5a**)



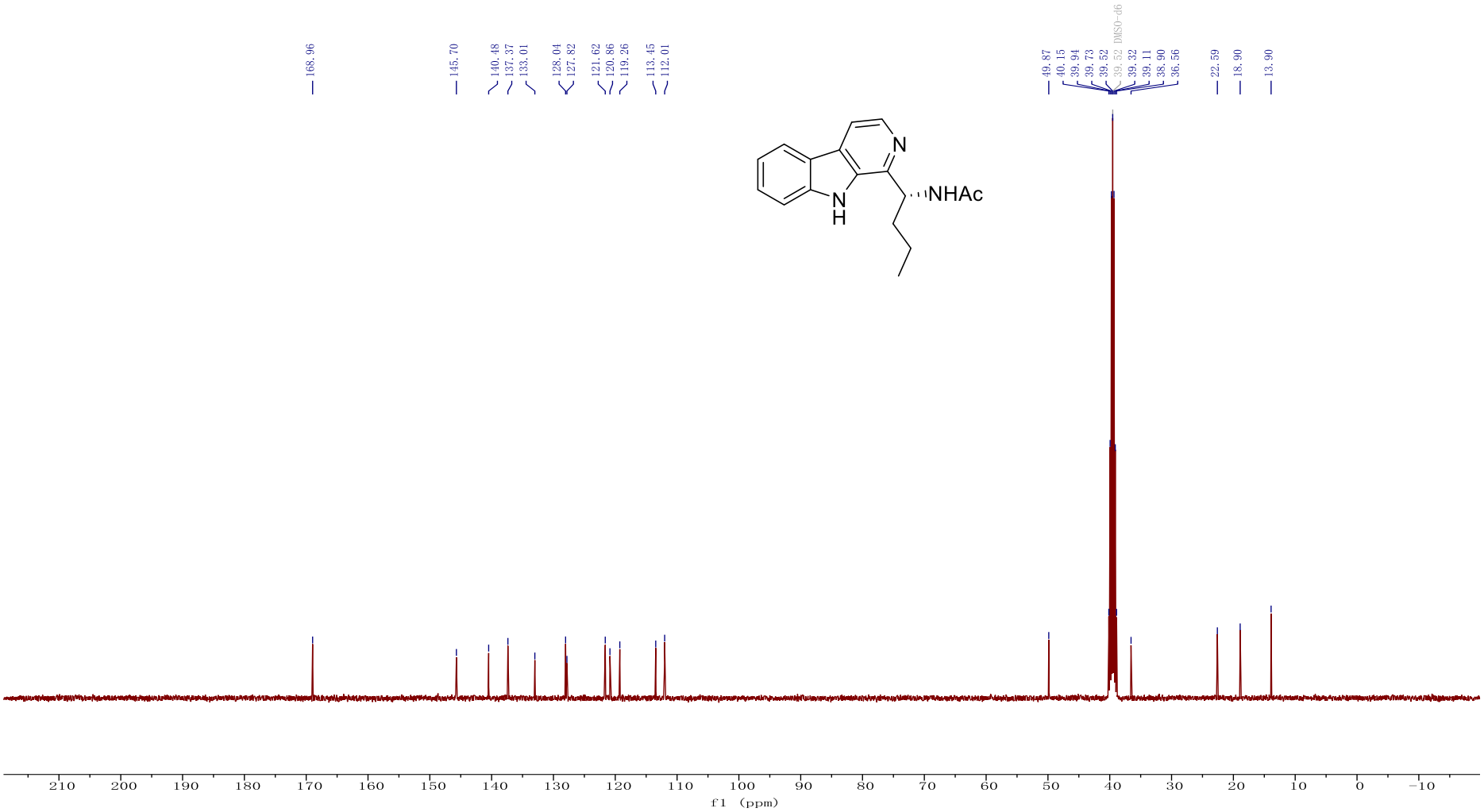
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5a**)



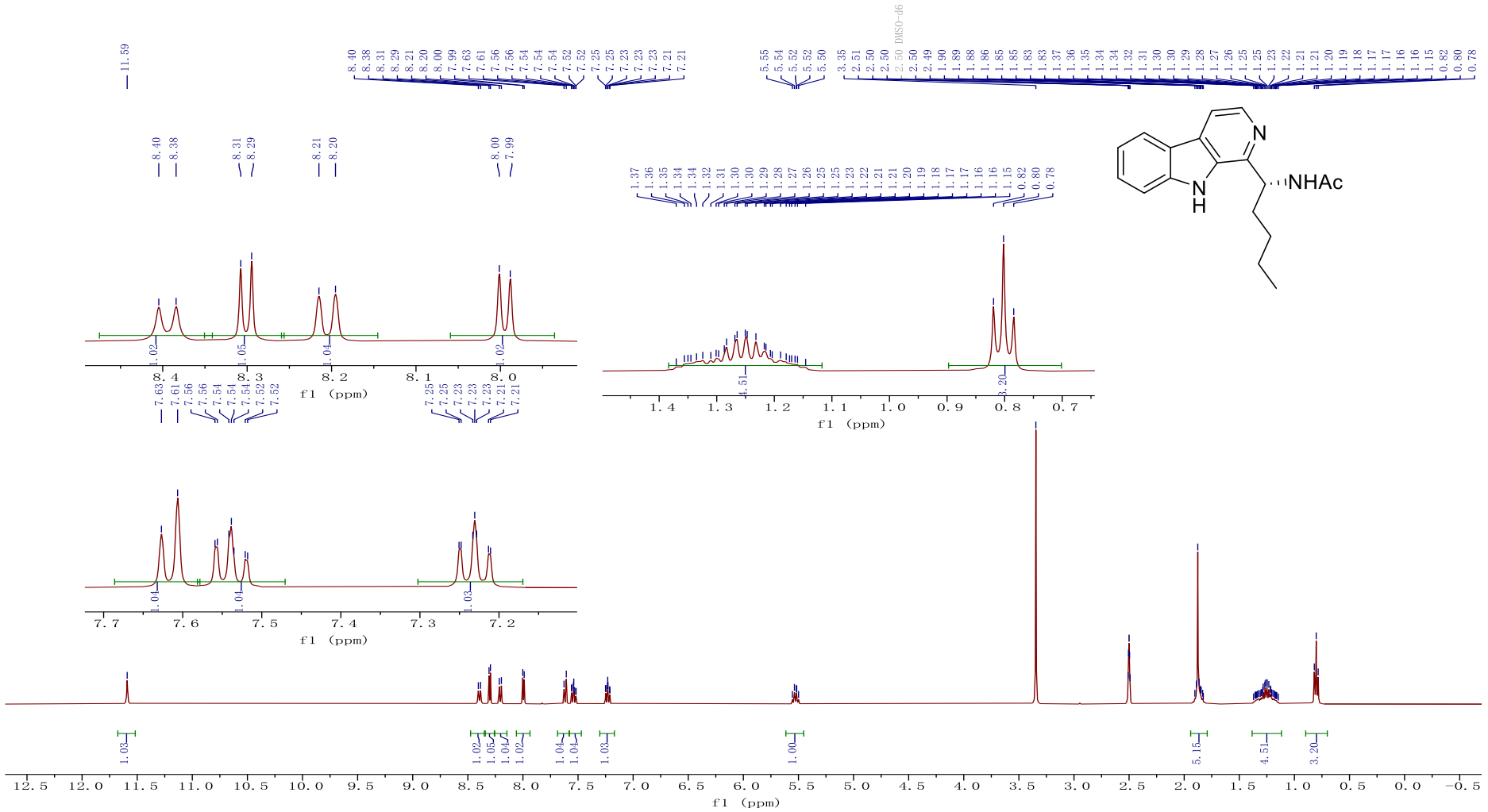
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5b**)



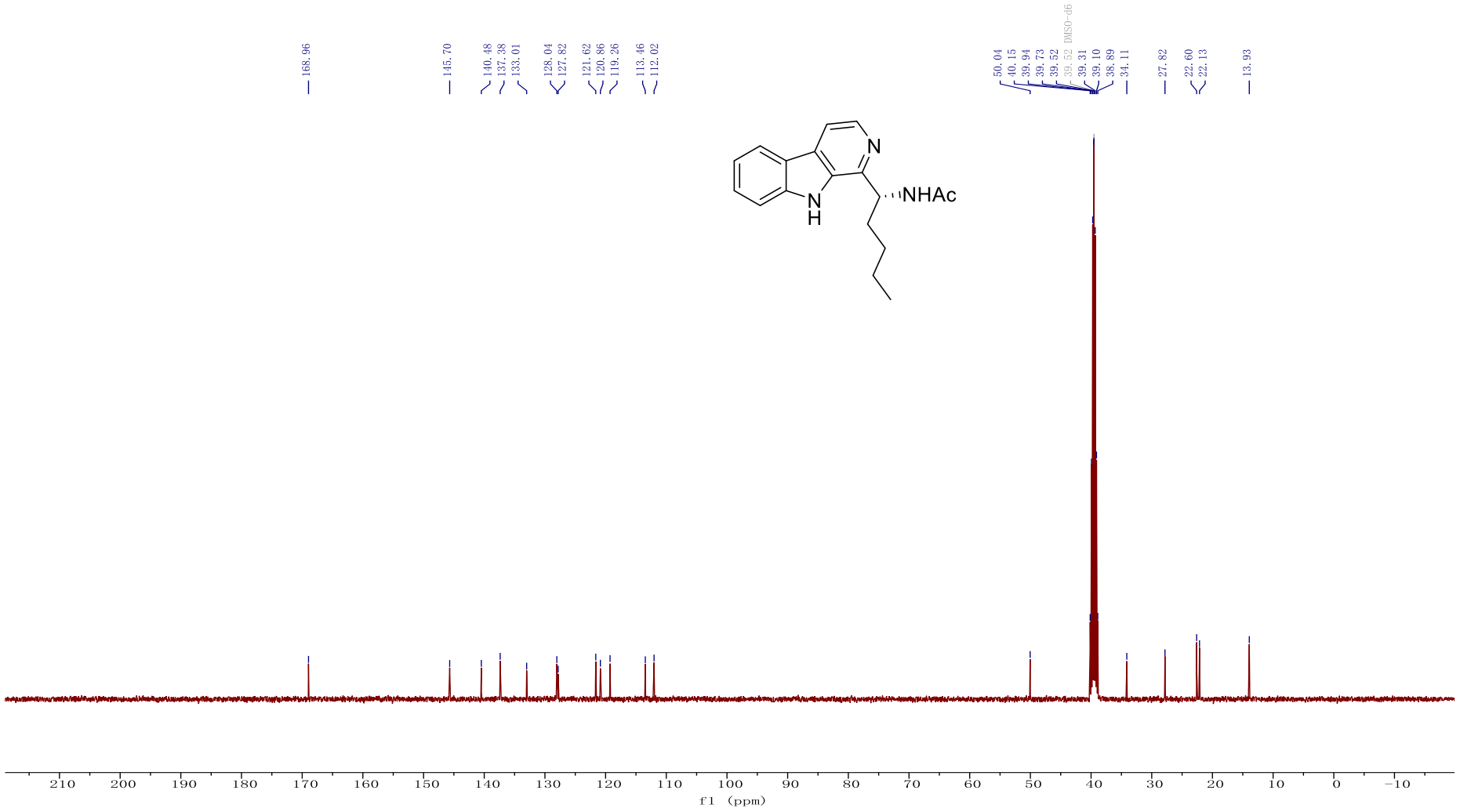
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5b**)



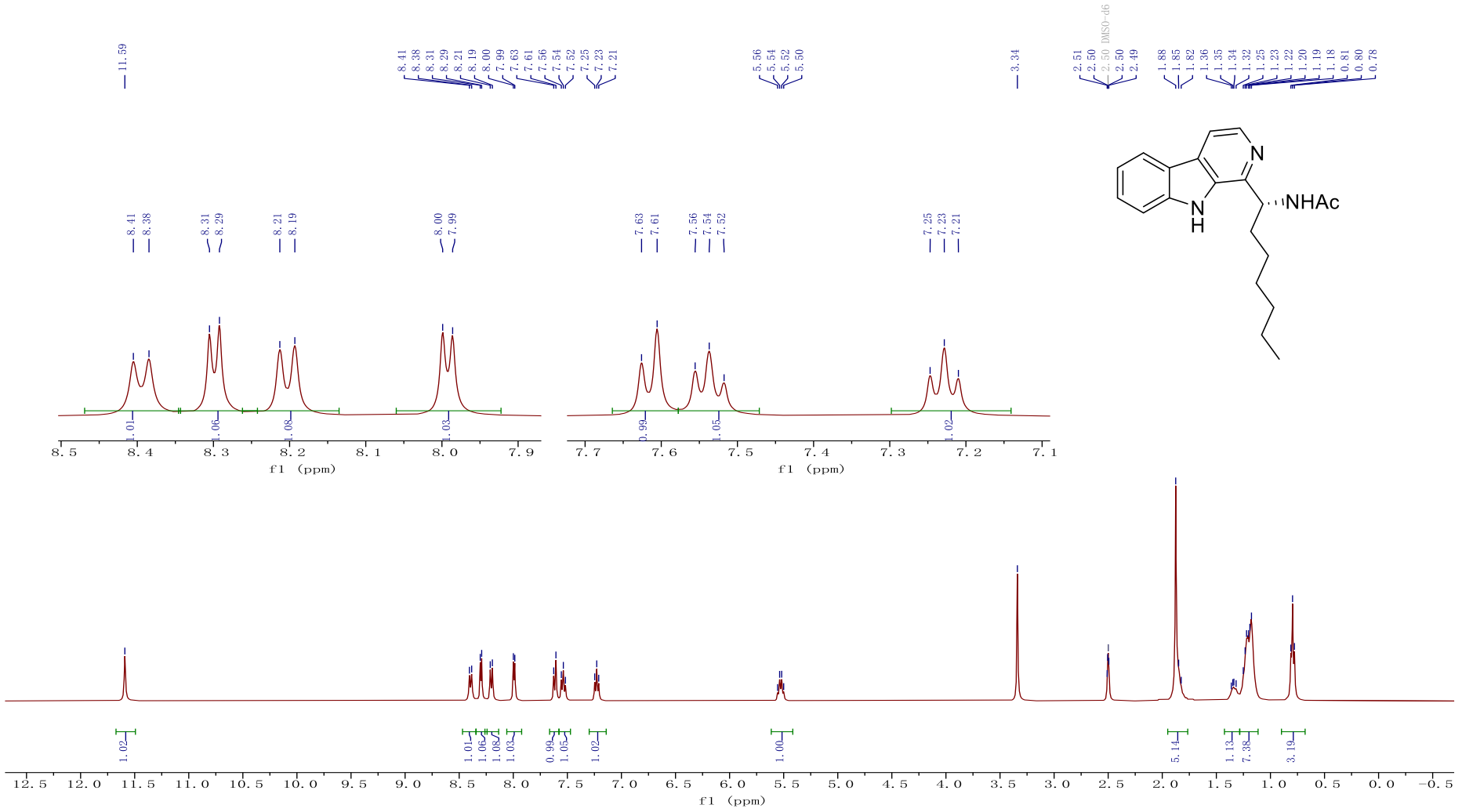
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)pentyl)acetamide (**5c**)



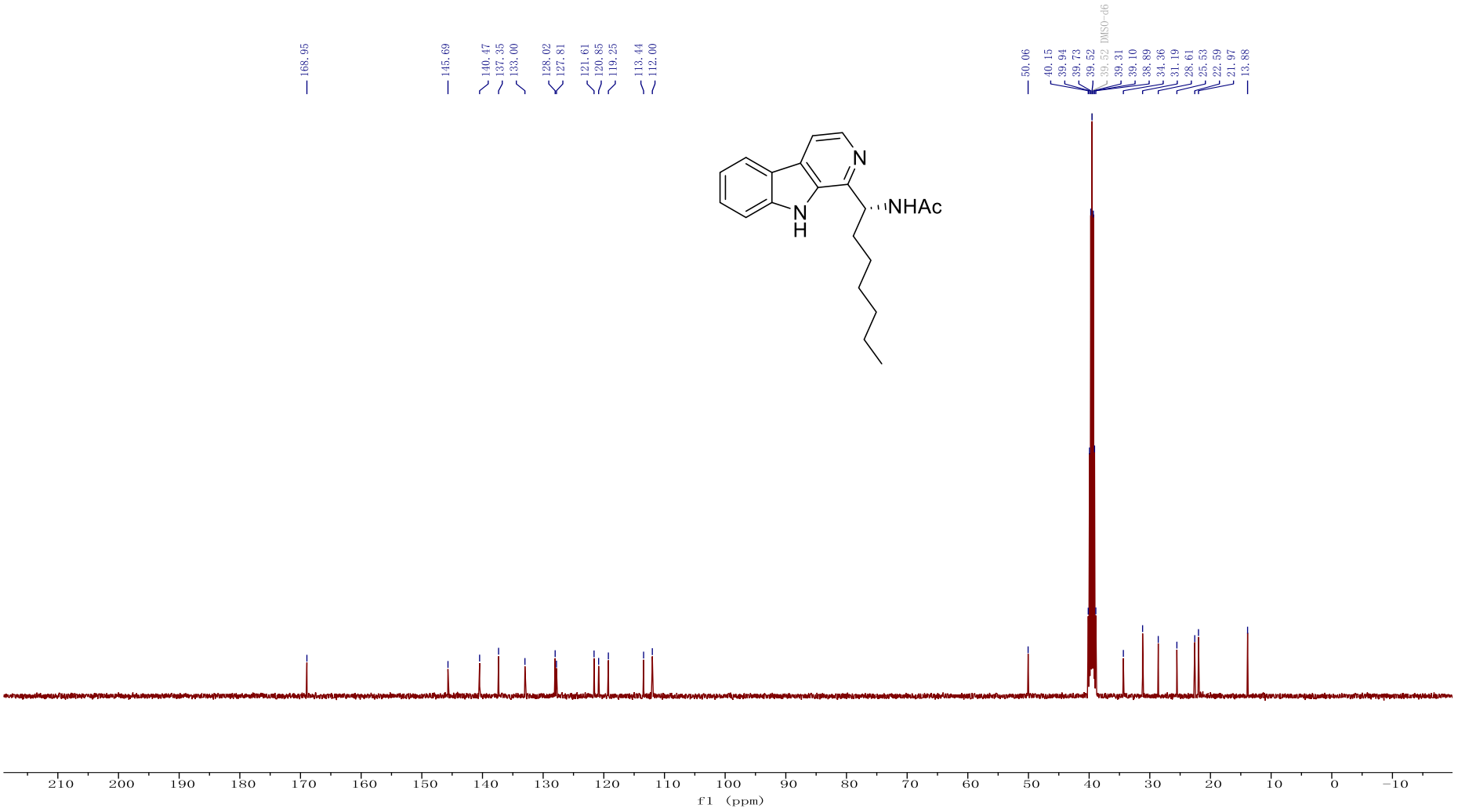
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)pentyl)acetamide (**5c**)



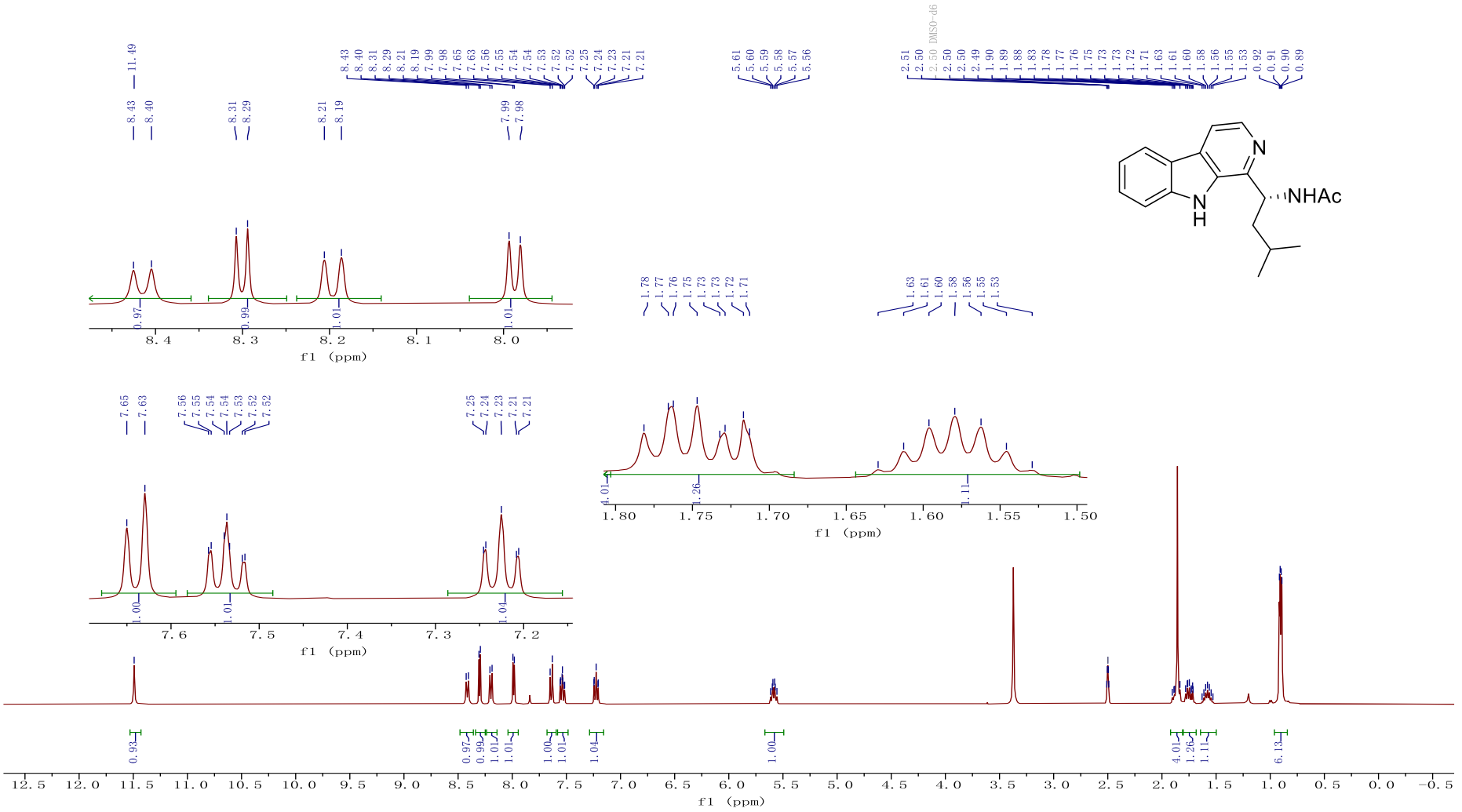
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)heptyl)acetamide (**5d**)



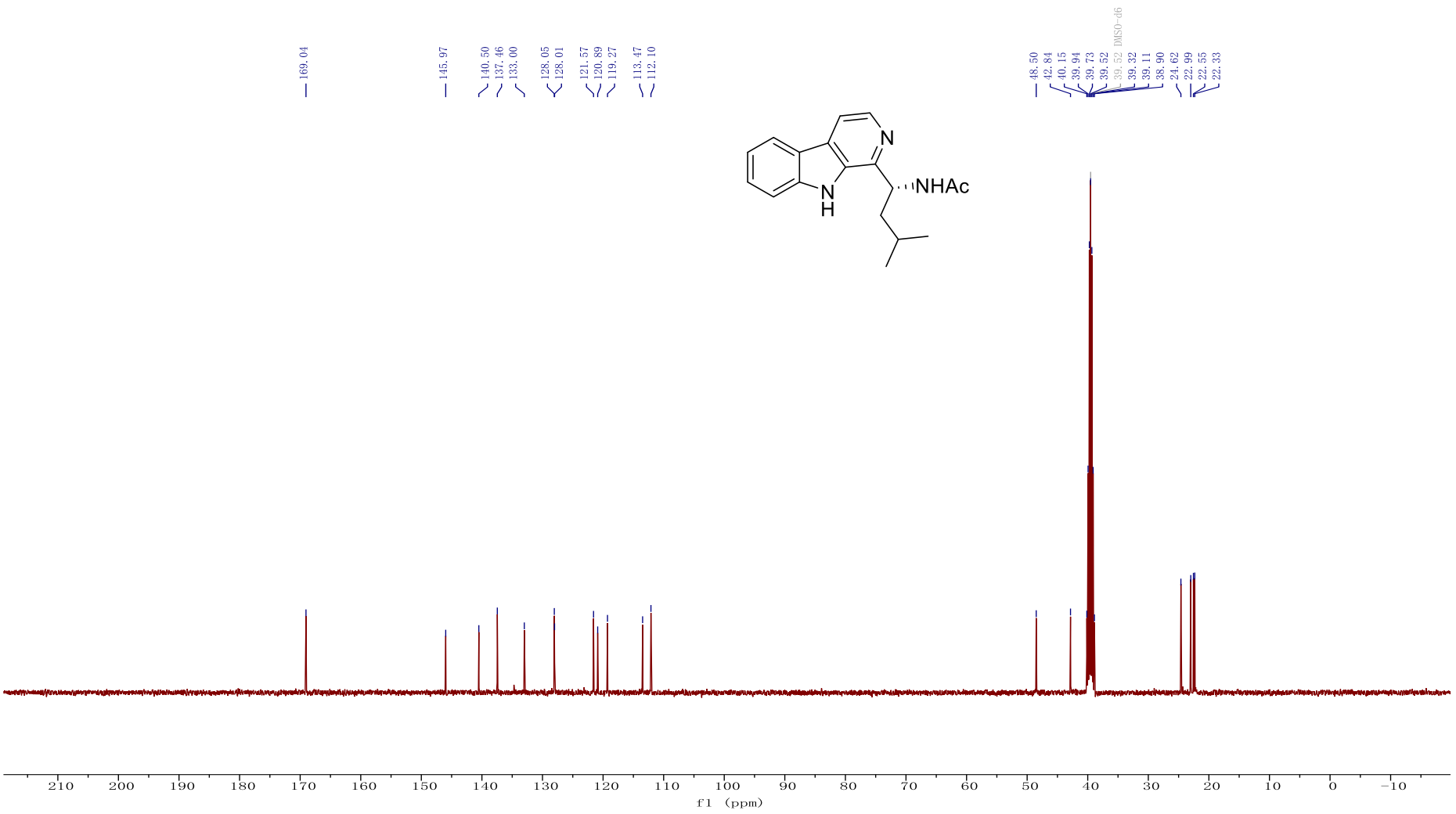
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)heptyl)acetamide (**5d**)



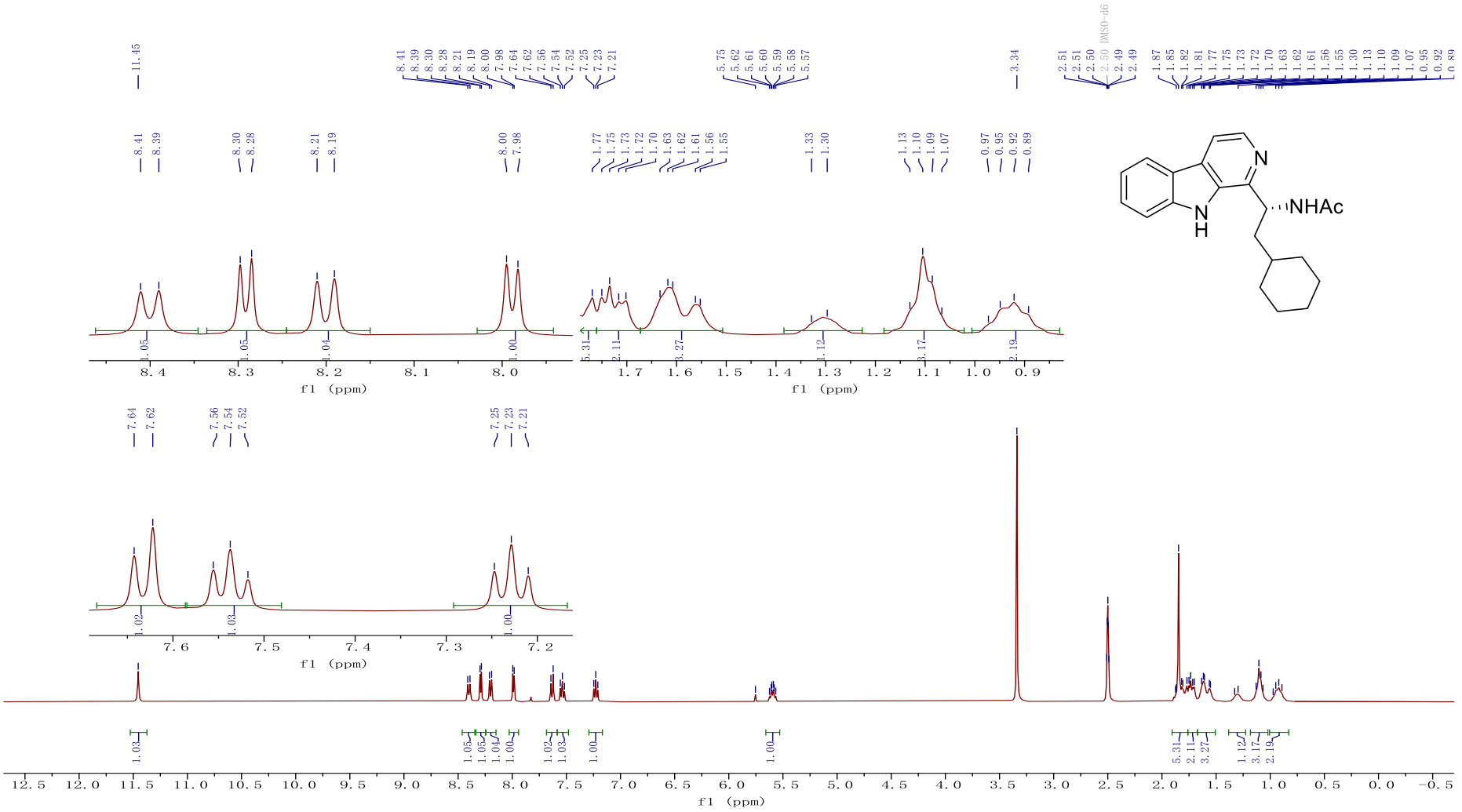
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(3-methyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5e**)



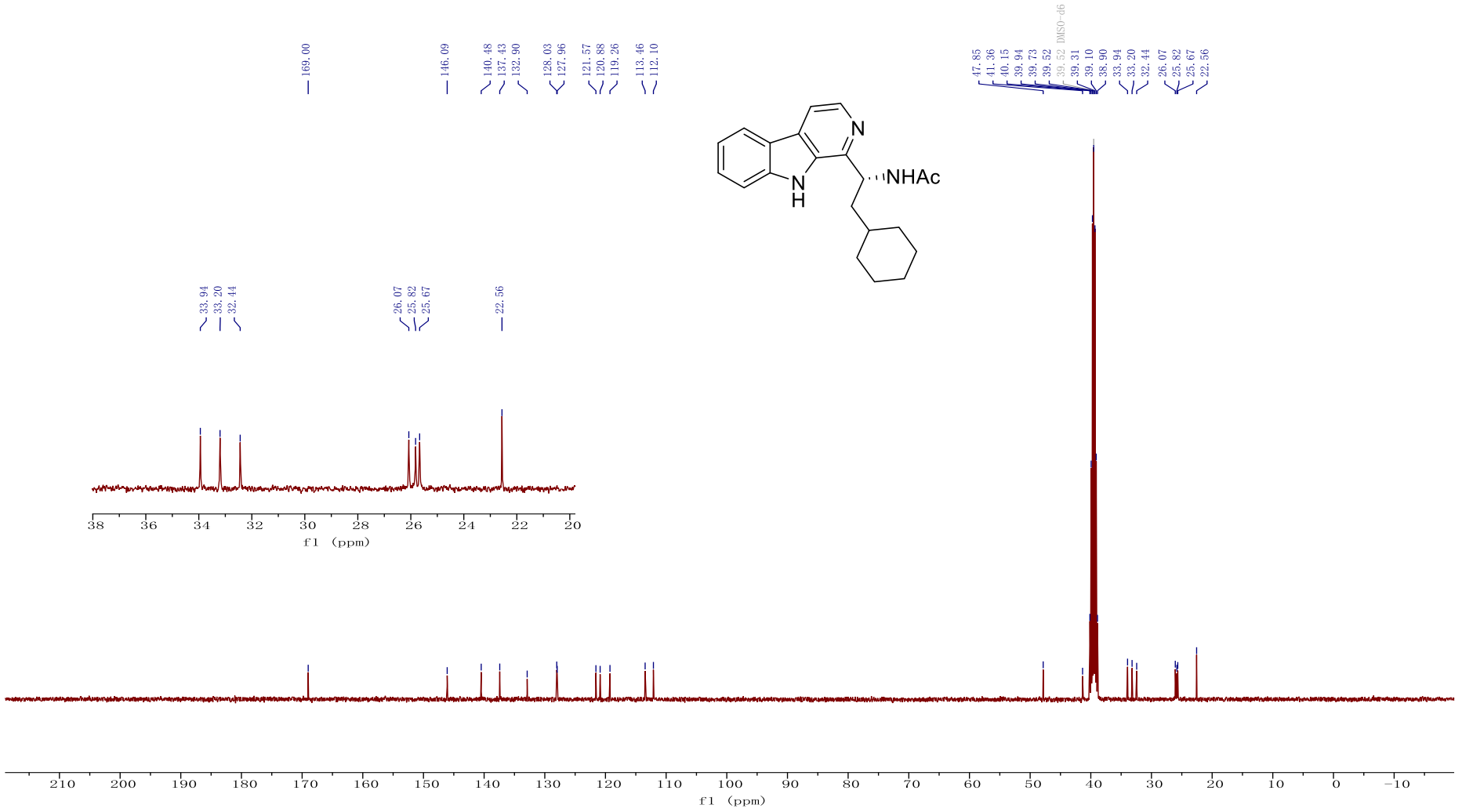
^{13}C NMR (100 MHz, DMSO-d_6) (*R*)-*N*-(3-methyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)butyl)acetamide (**5e**)



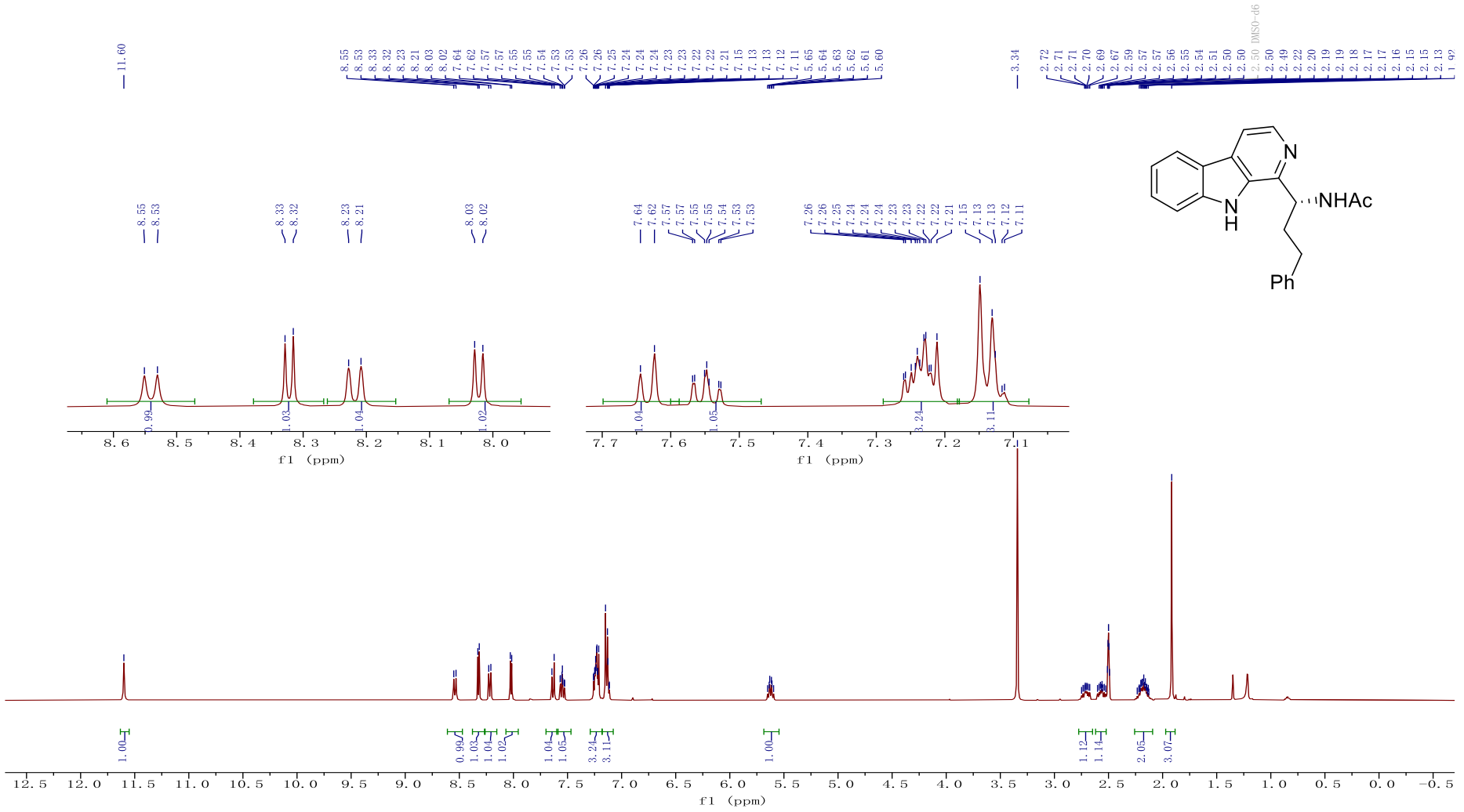
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-cyclohexyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5f**)



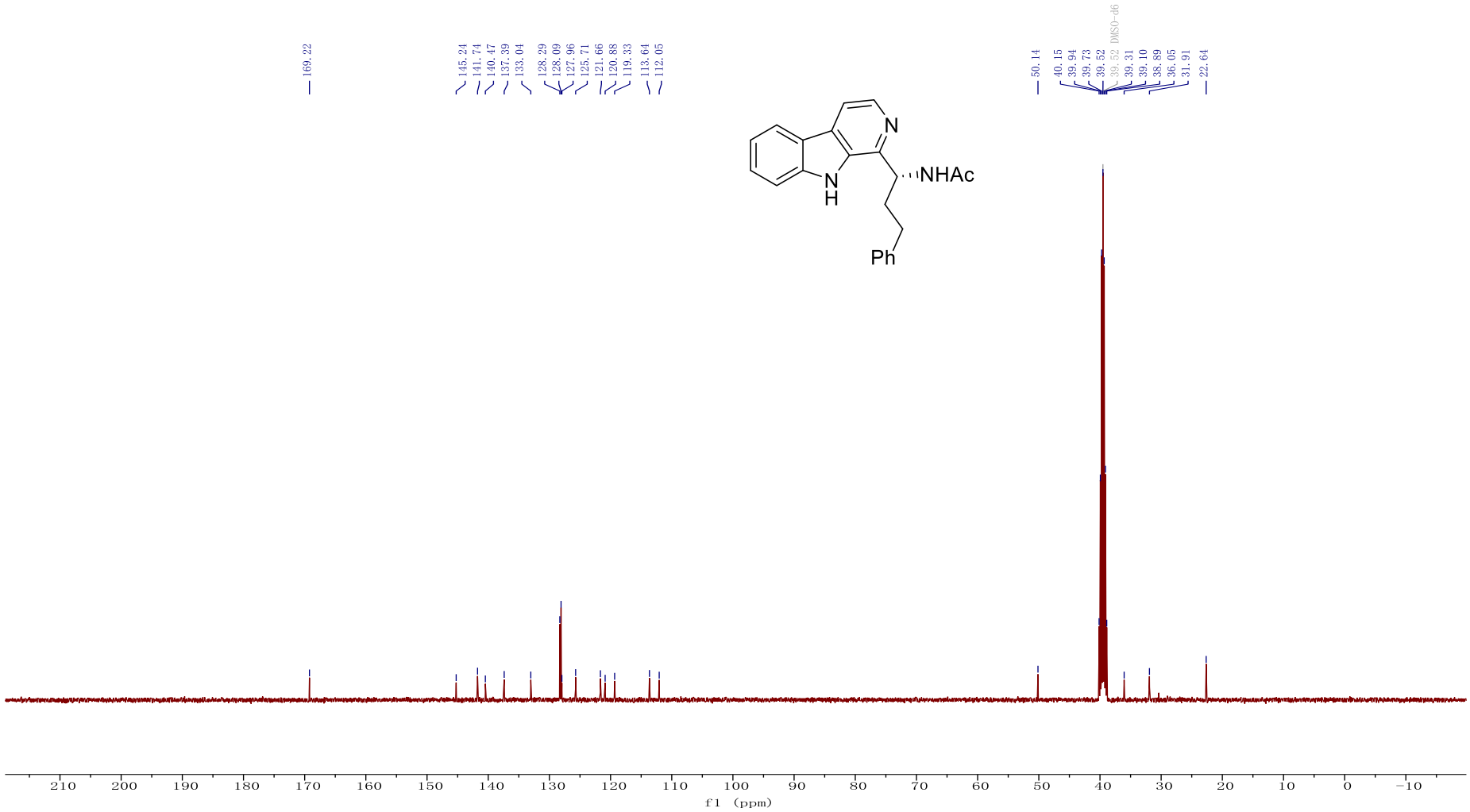
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-cyclohexyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5f**)



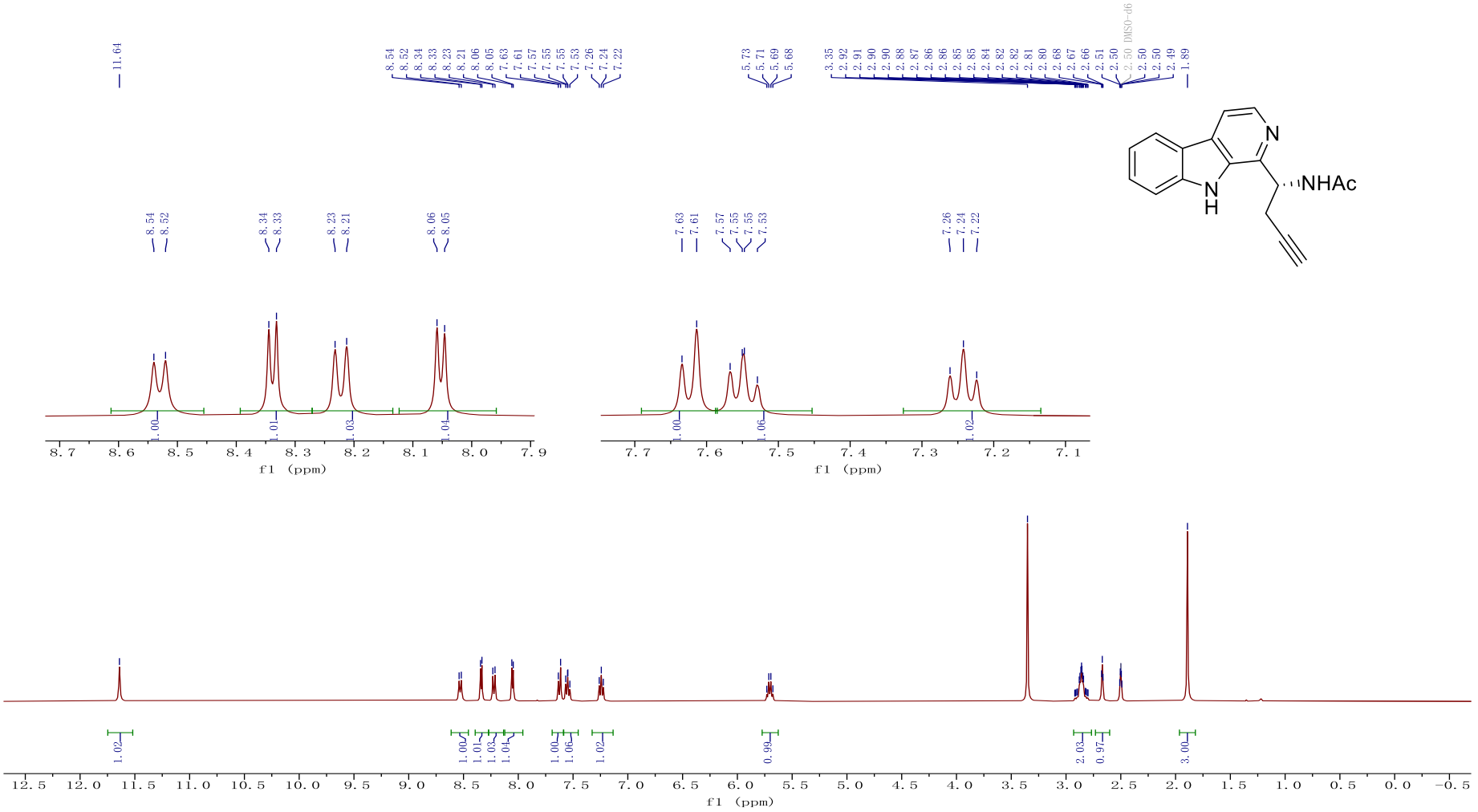
[1]H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(3-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5g**)



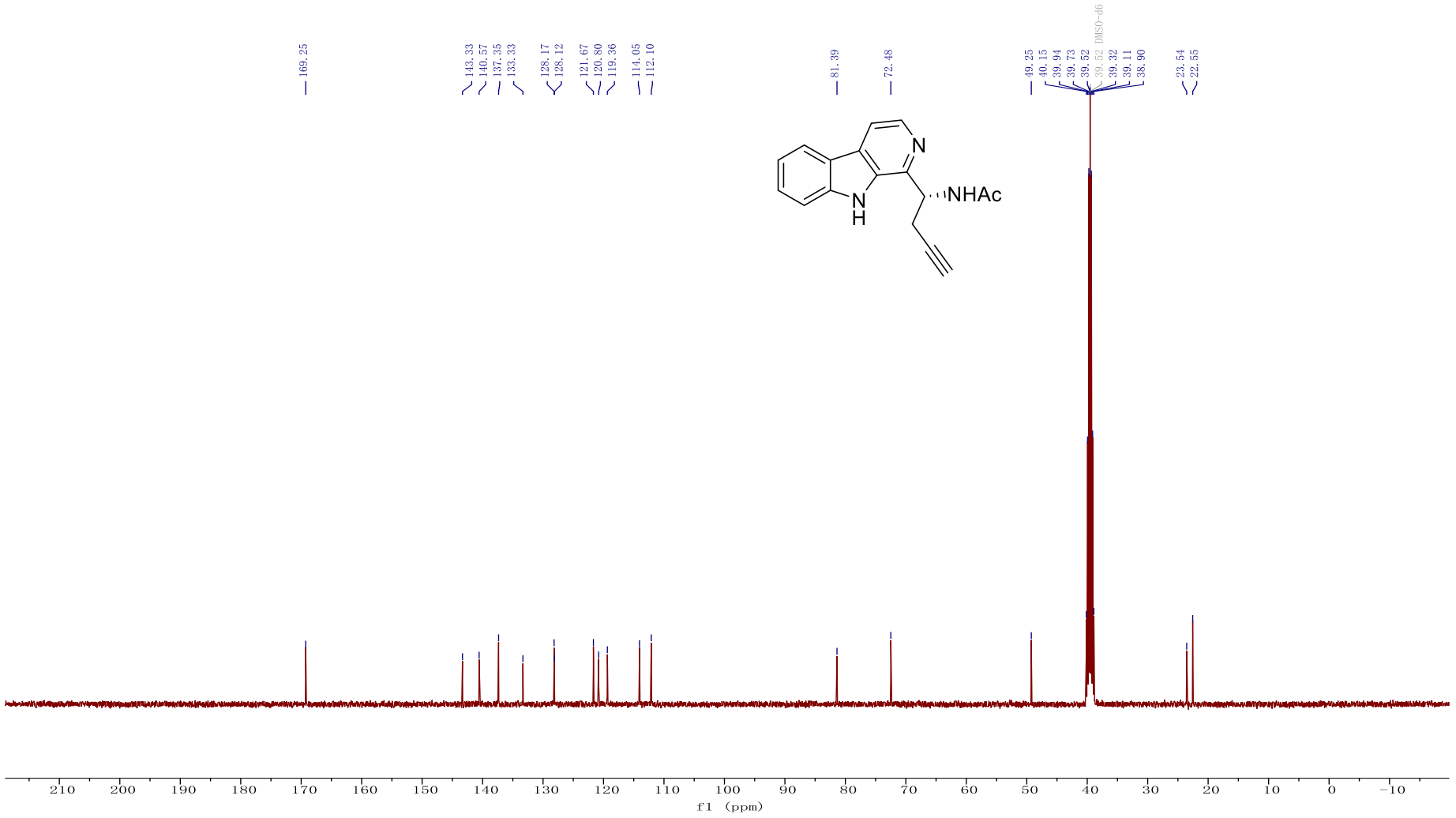
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(3-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)propyl)acetamide (**5g**)



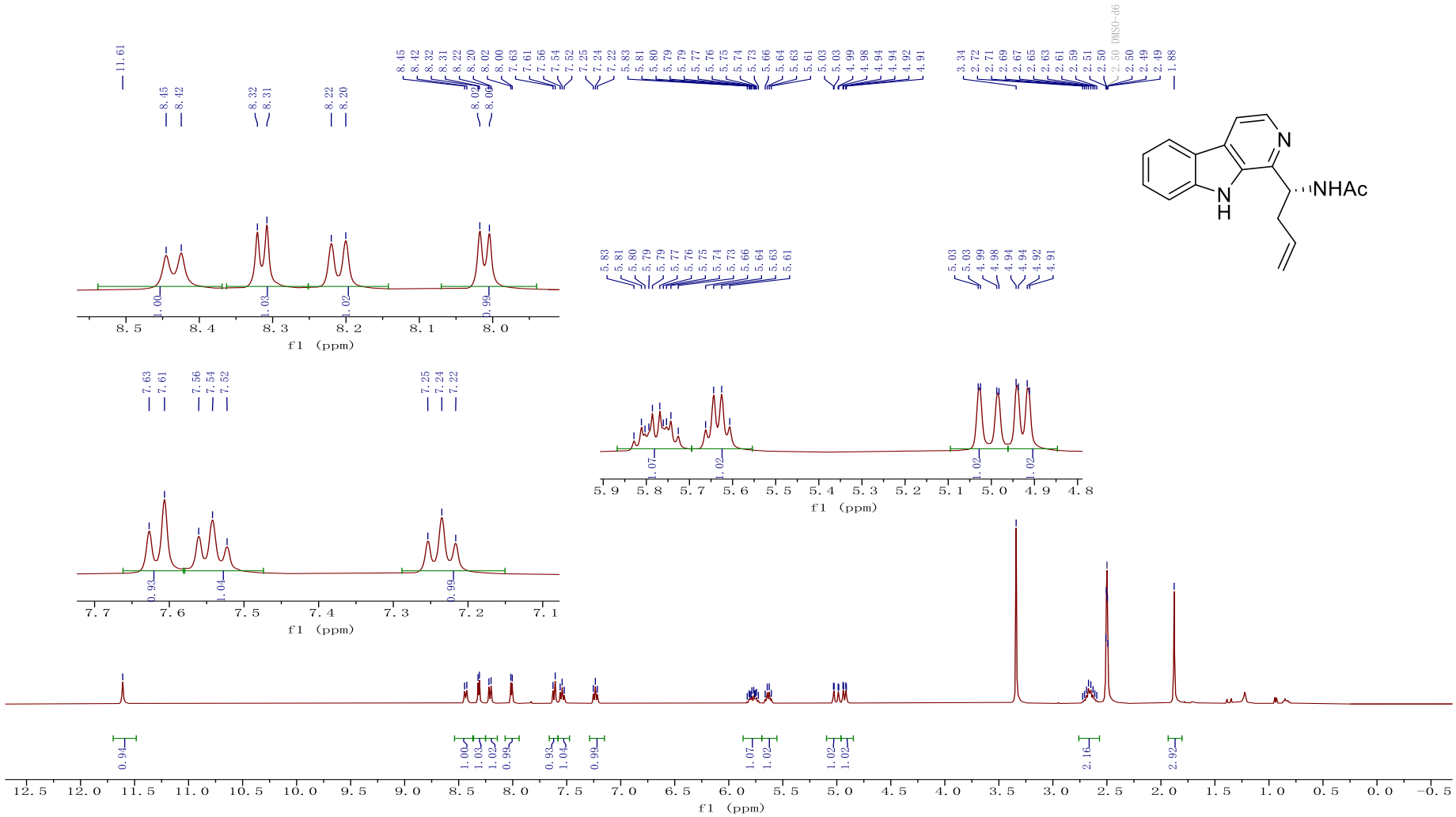
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-yn-1-yl)acetamide (**5h**)



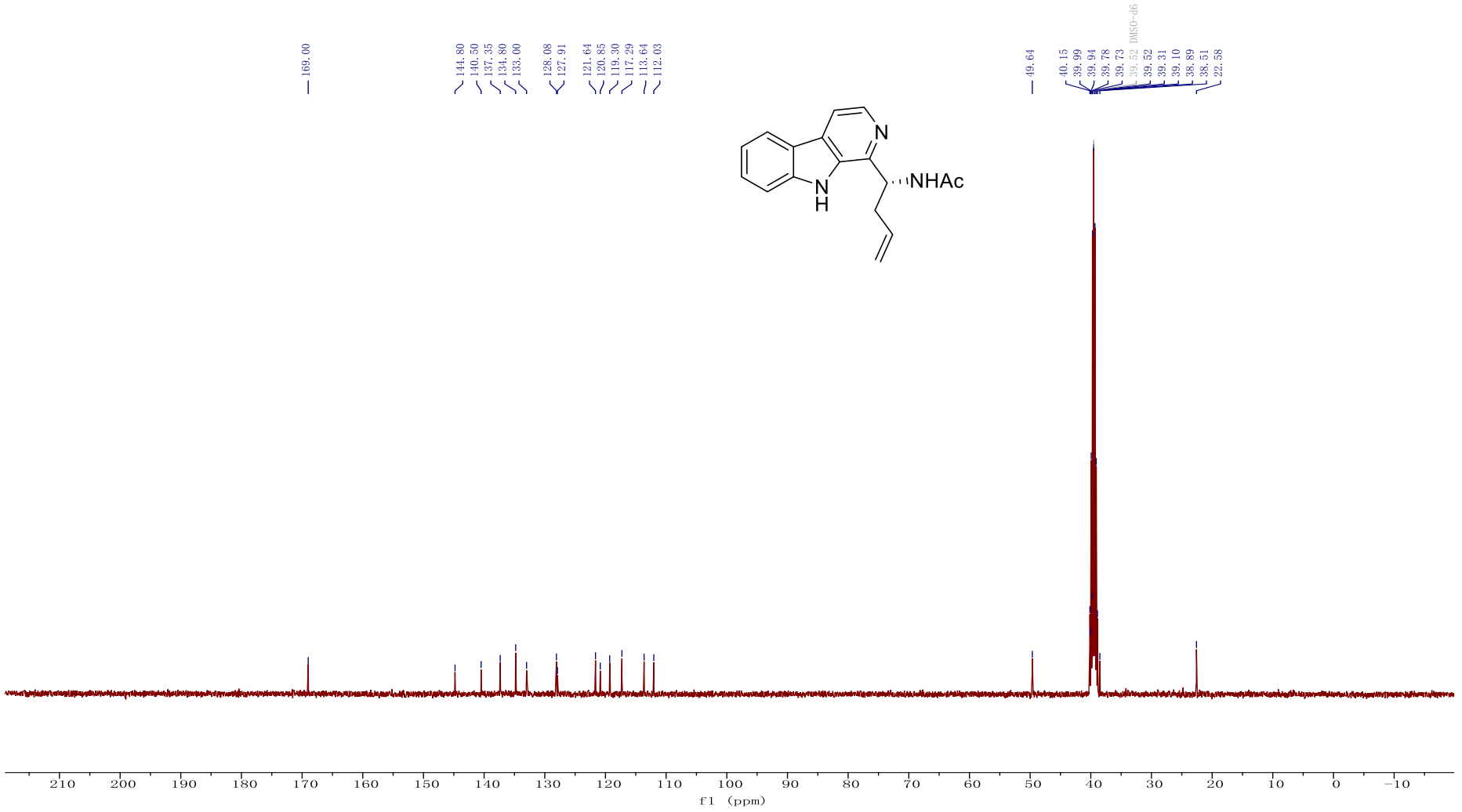
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-yn-1-yl)acetamide (**5h**)



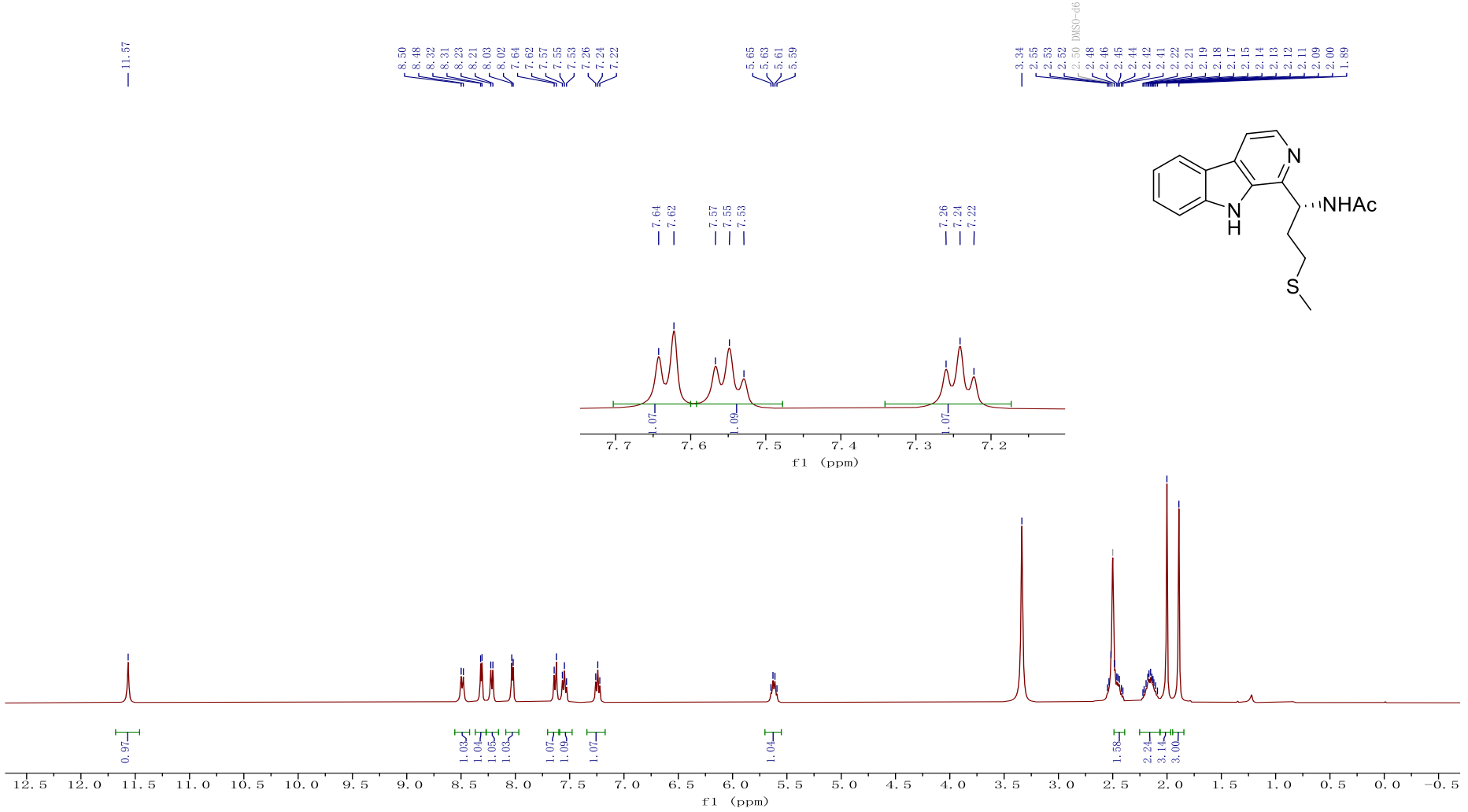
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-en-1-yl)acetamide (**5i**)



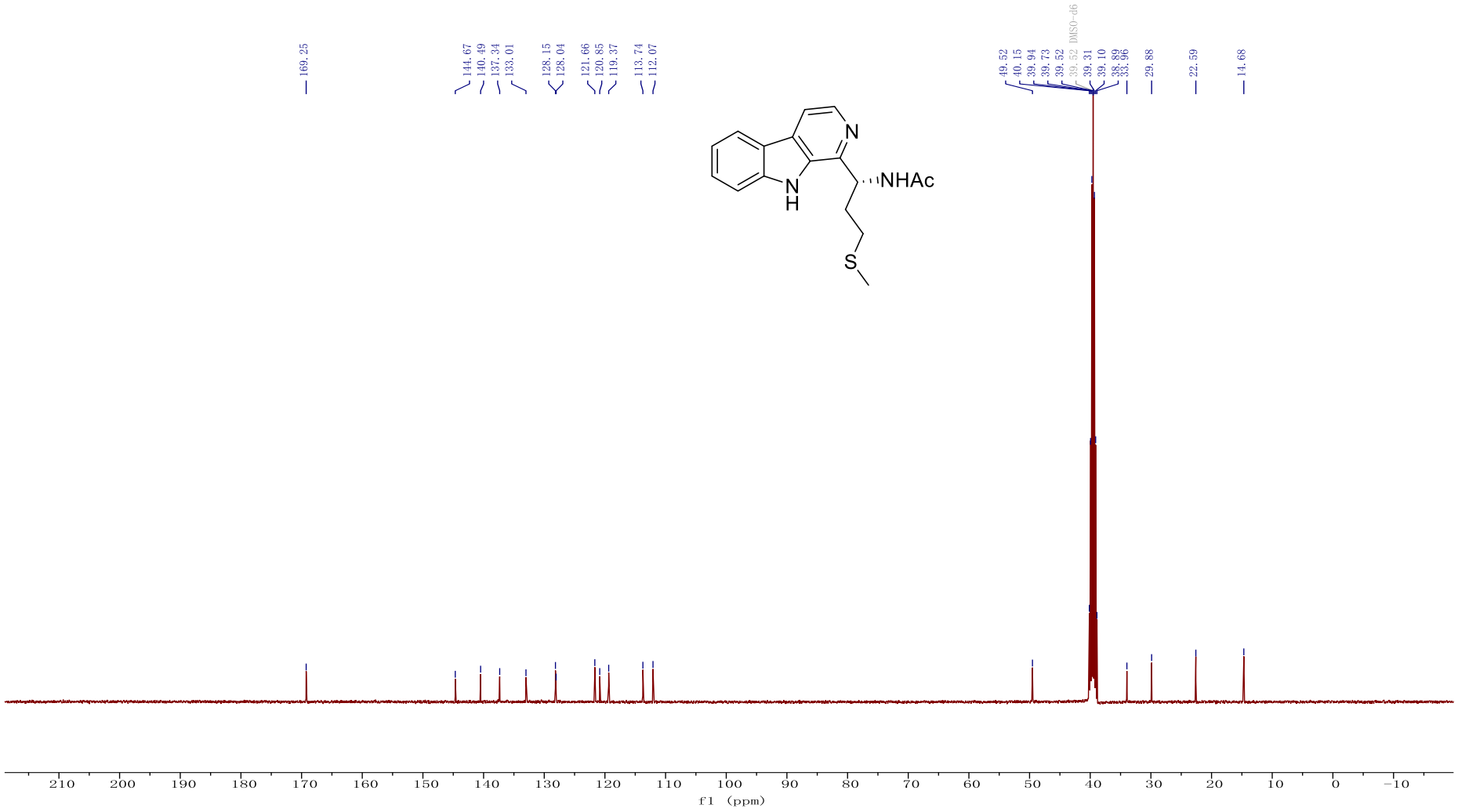
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)but-3-en-1-yl)acetamide (**5i**)



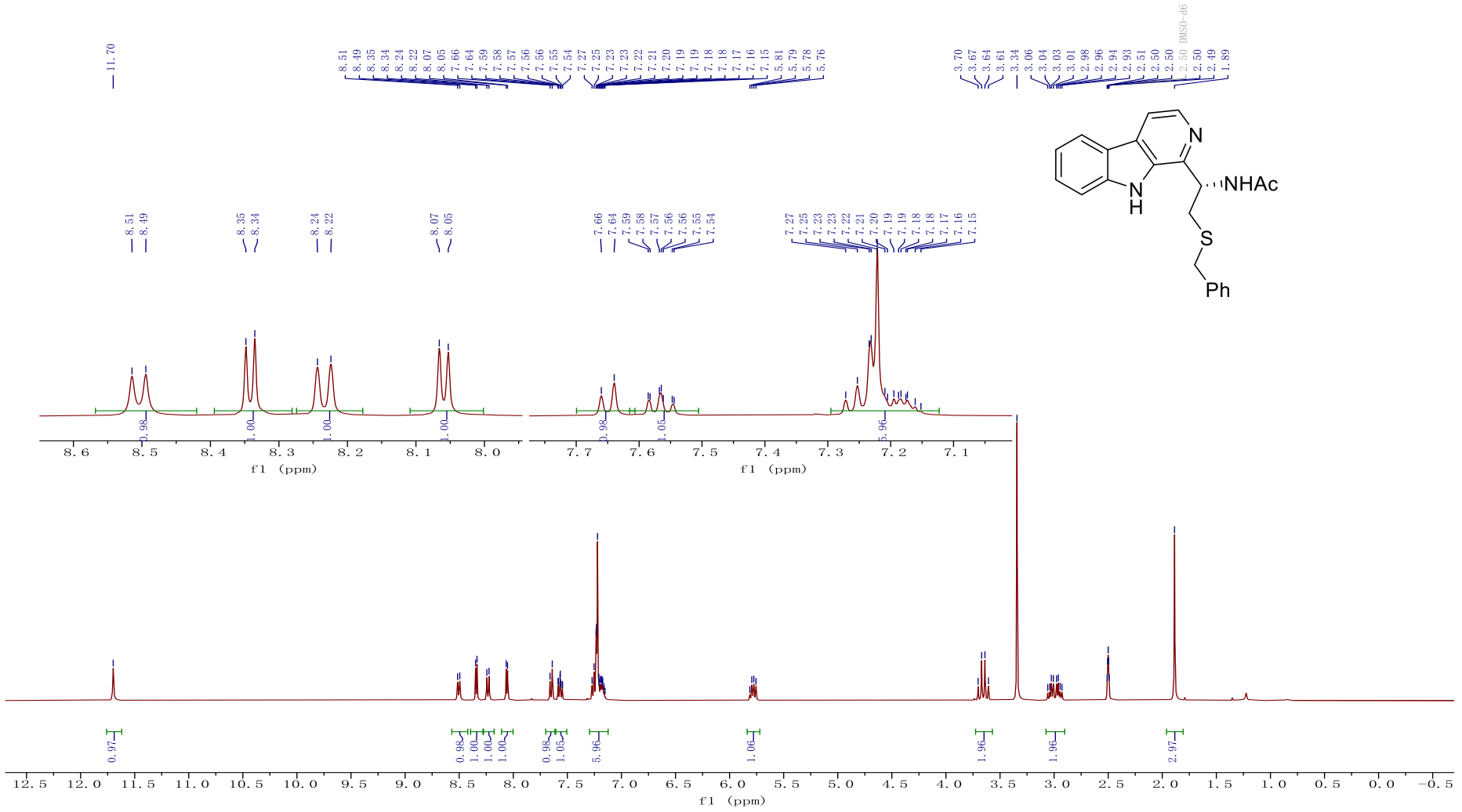
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(methylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5j**)



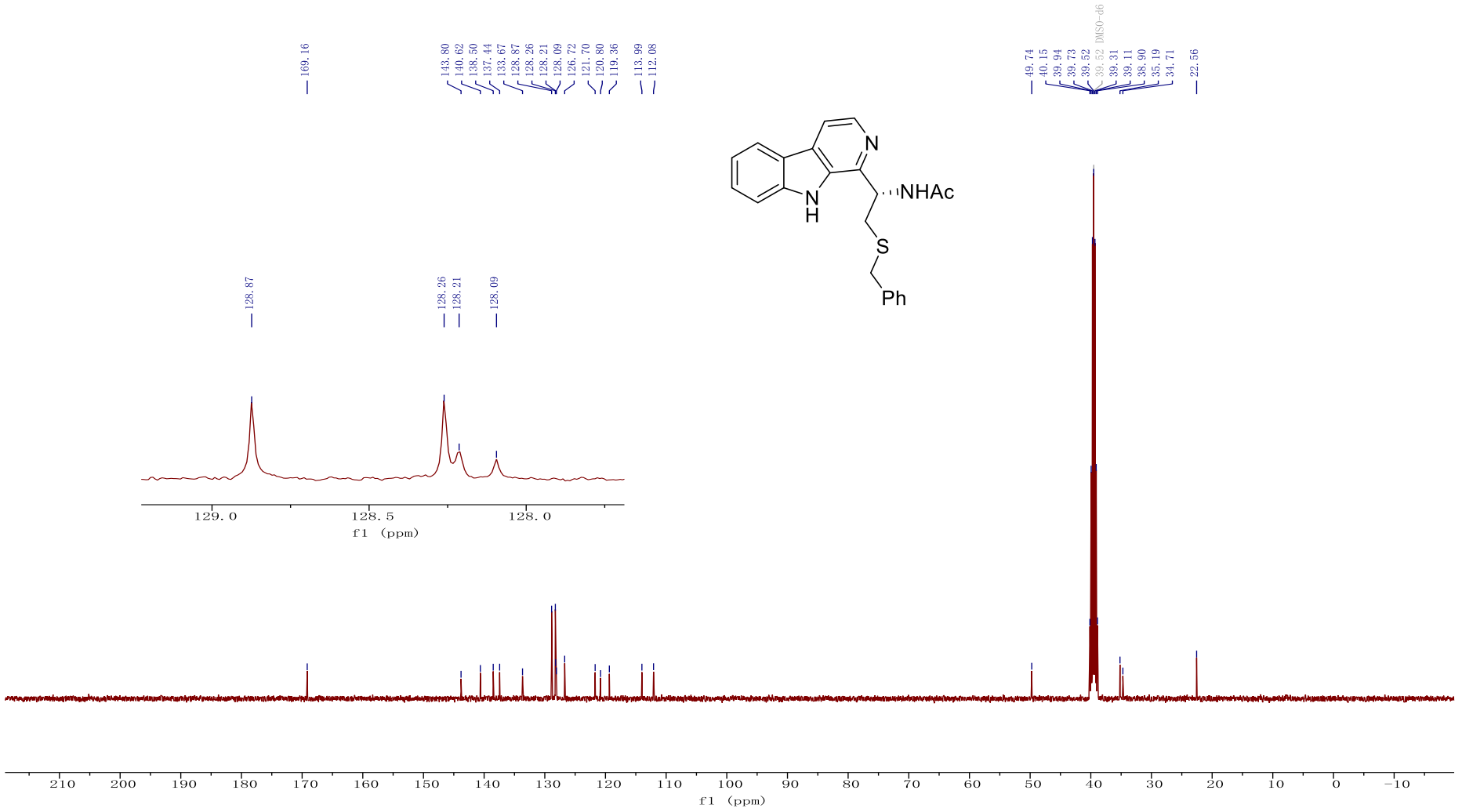
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(methylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5j**)



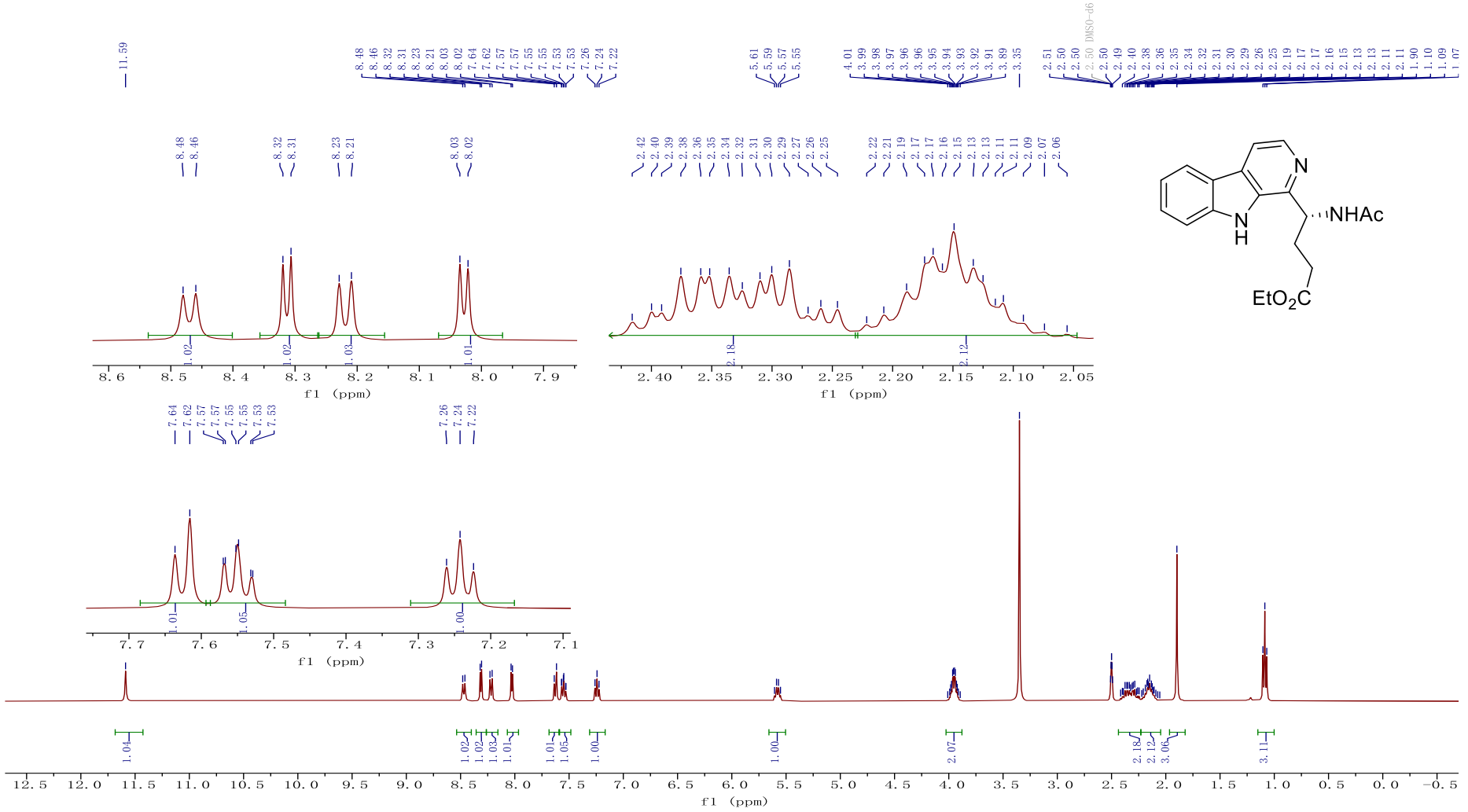
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(benzylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5k**)



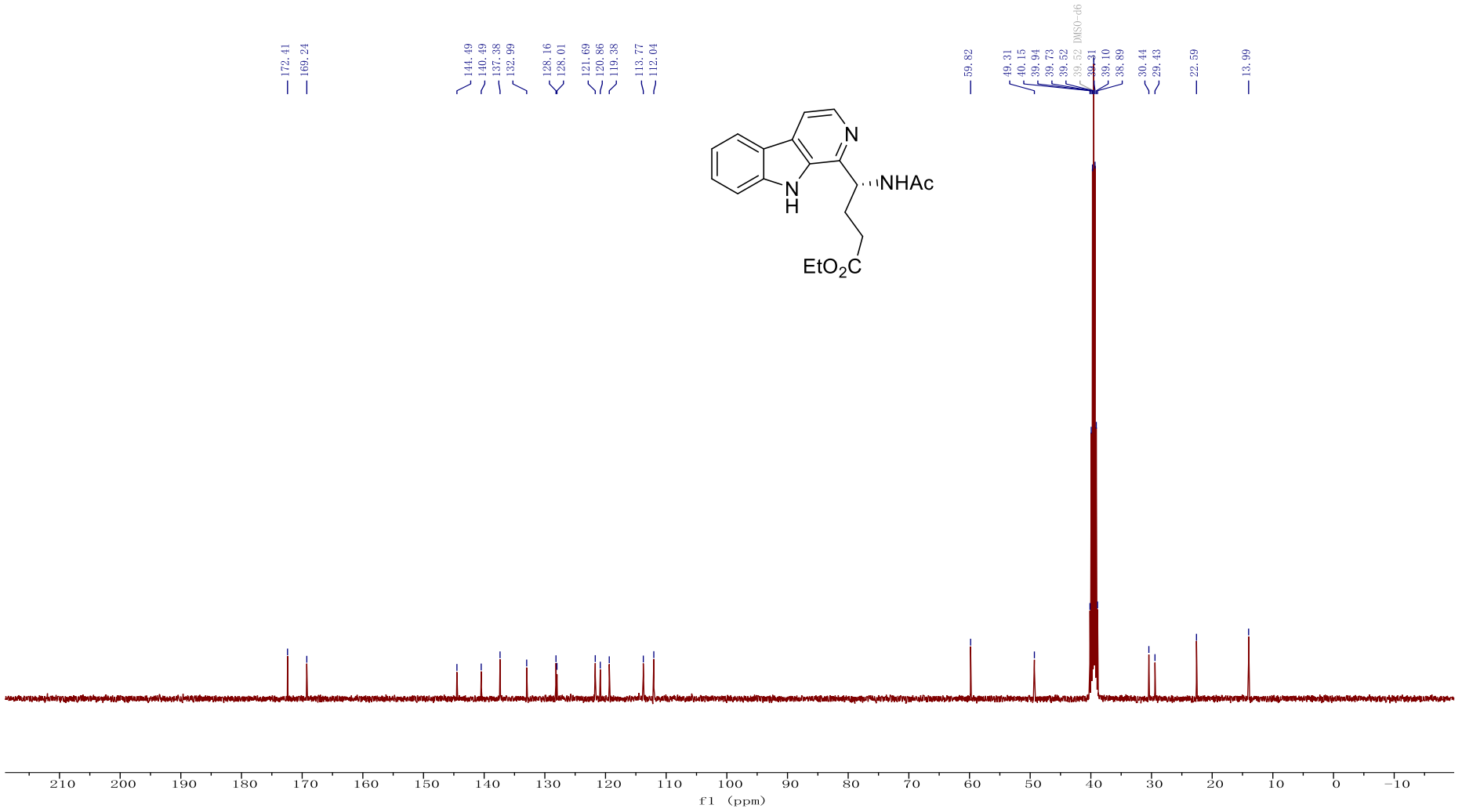
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(benzylthio)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5k**)



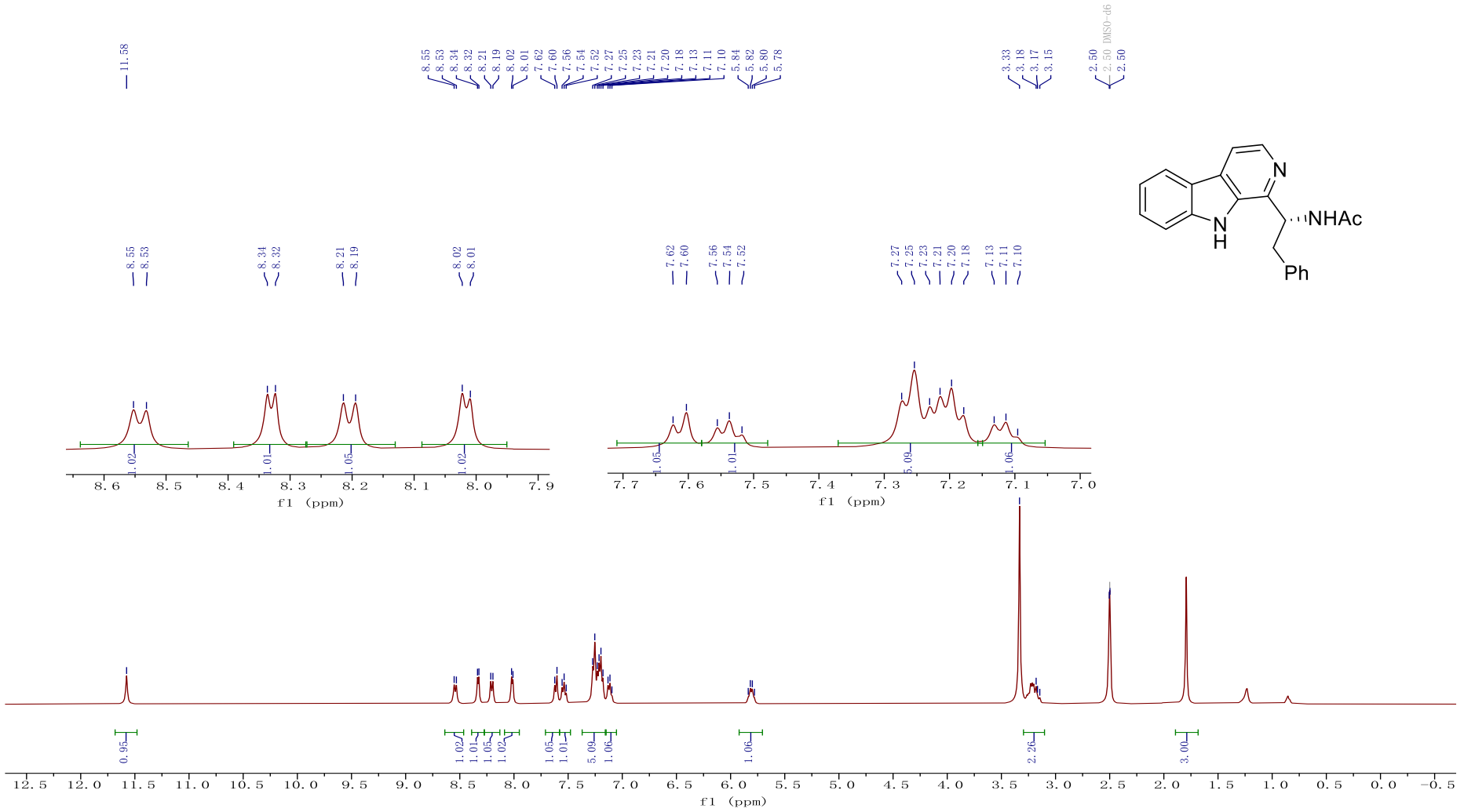
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-Ethyl 4-acetamido-4-(9*H*-pyrido[3,4-*b*]indol-1-yl)butanoate (**5l**)



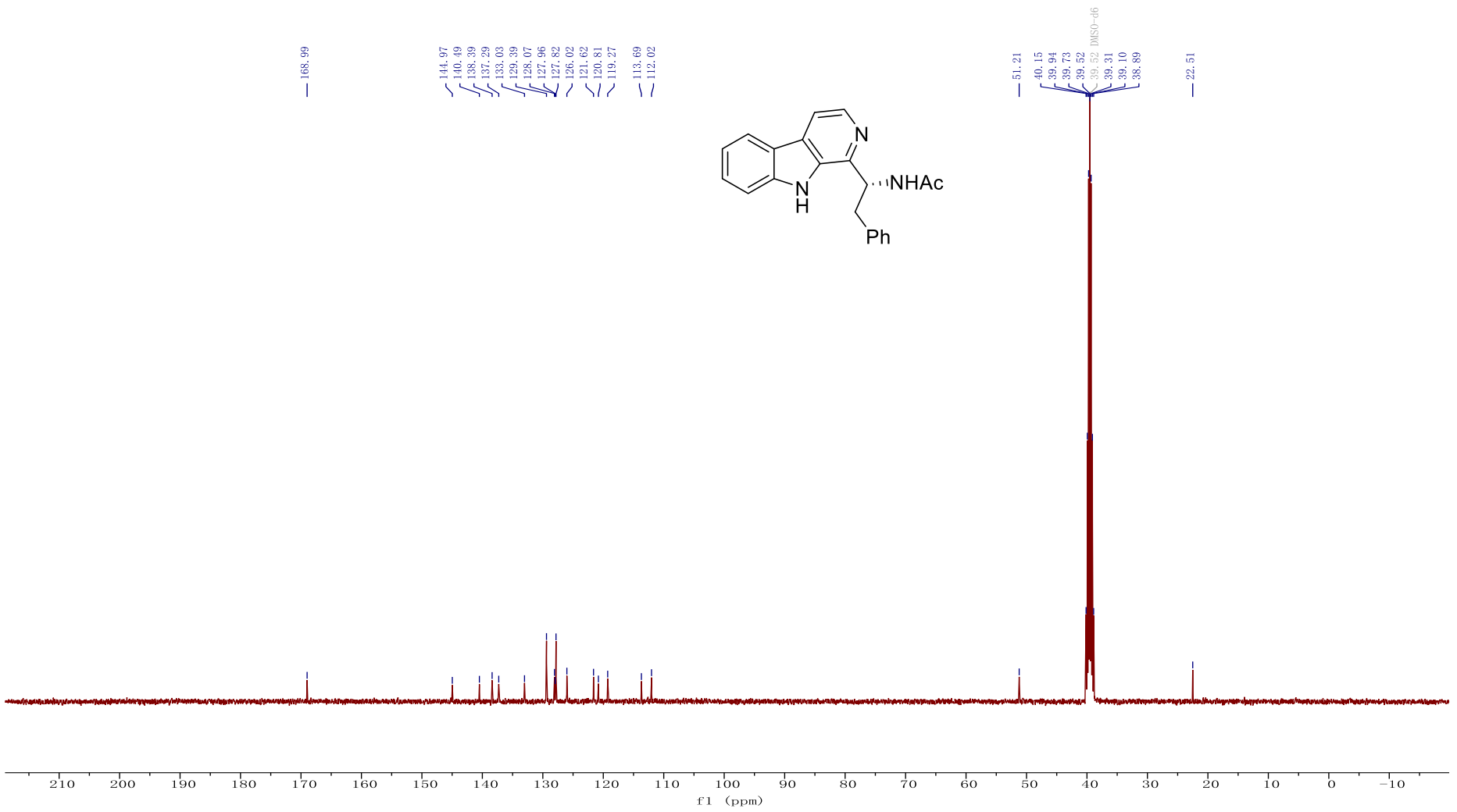
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-Ethyl 4-acetamido-4-(9*H*-pyrido[3,4-*b*]indol-1-yl)butanoate (**5l**)



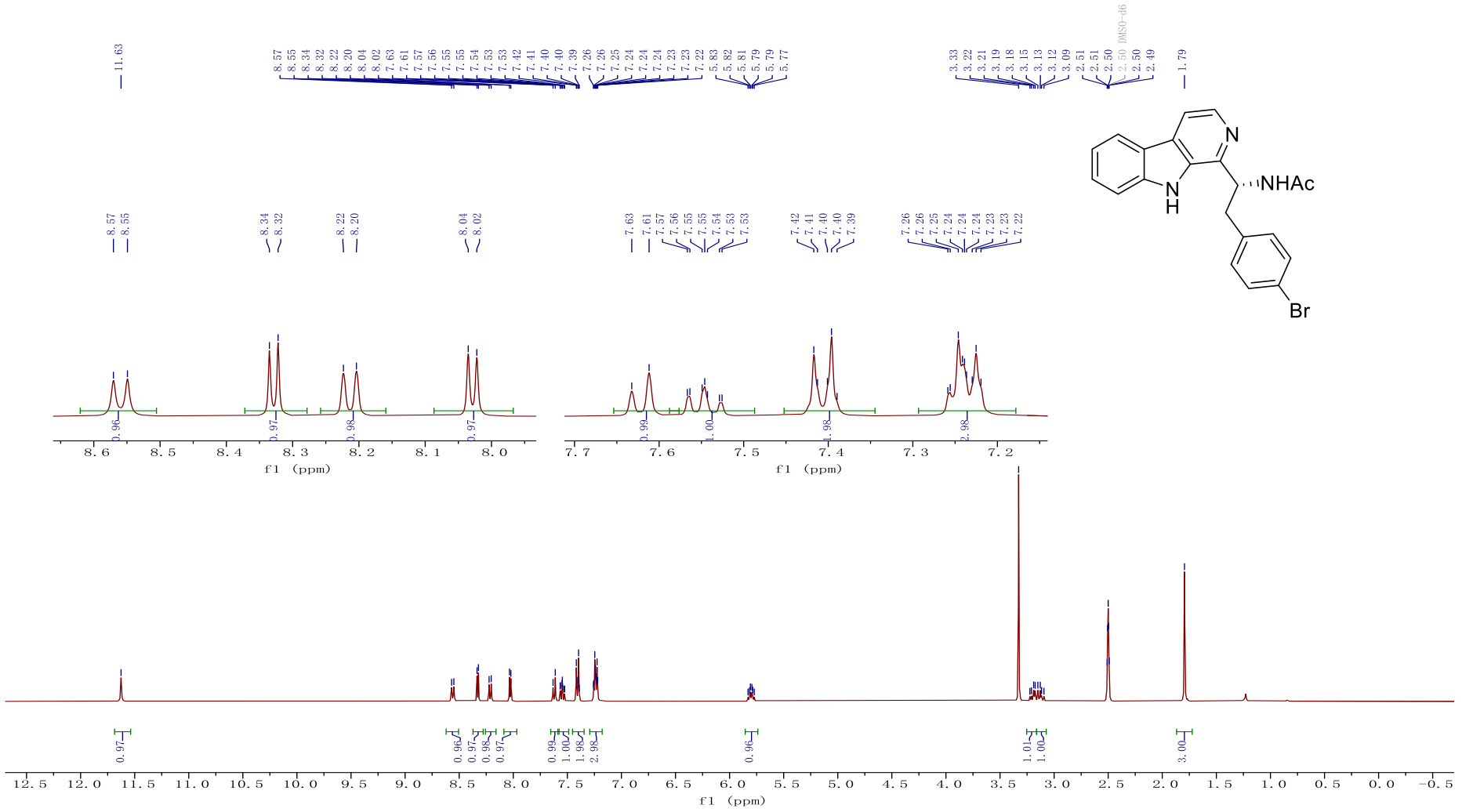
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5m**)



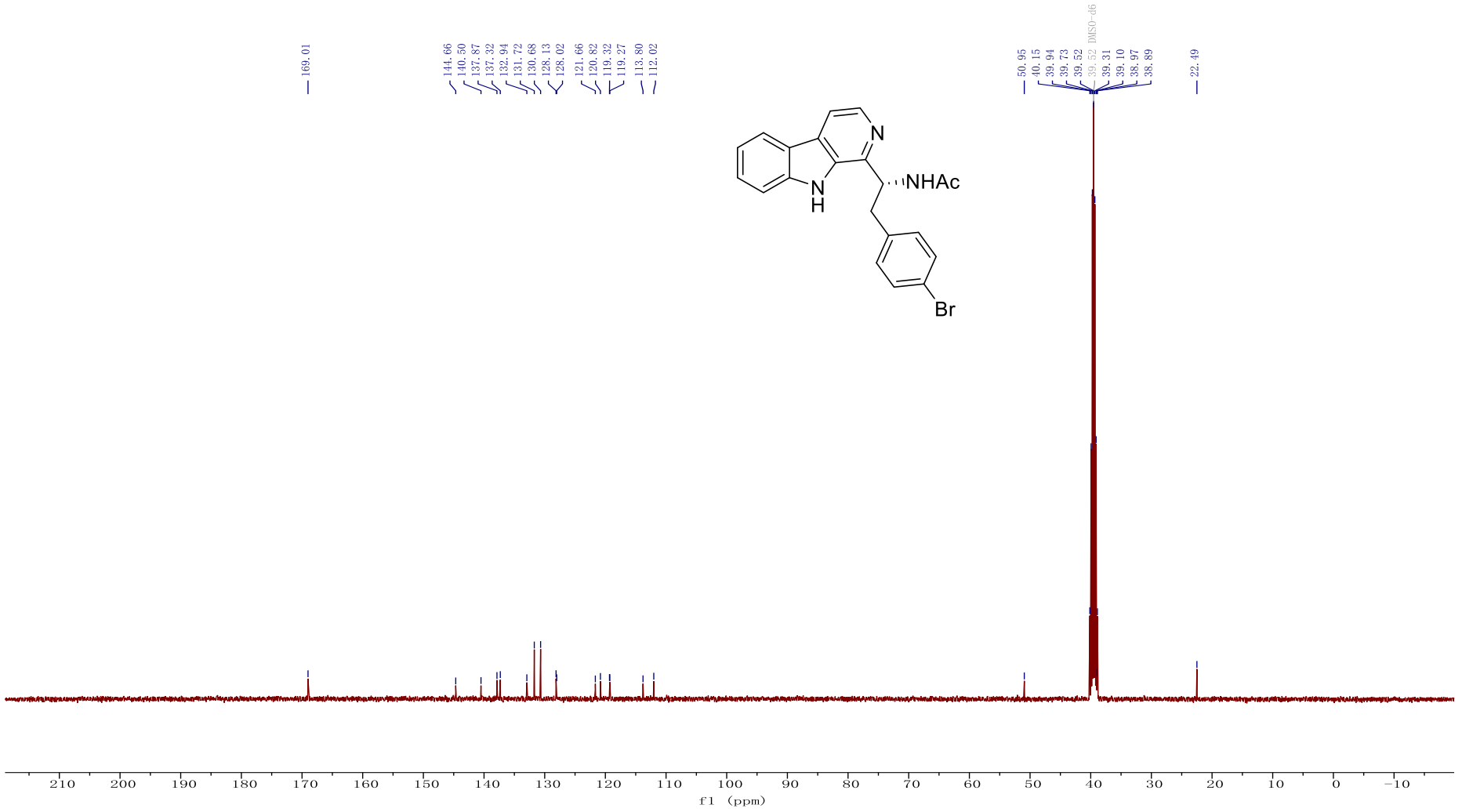
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-phenyl-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5m**)



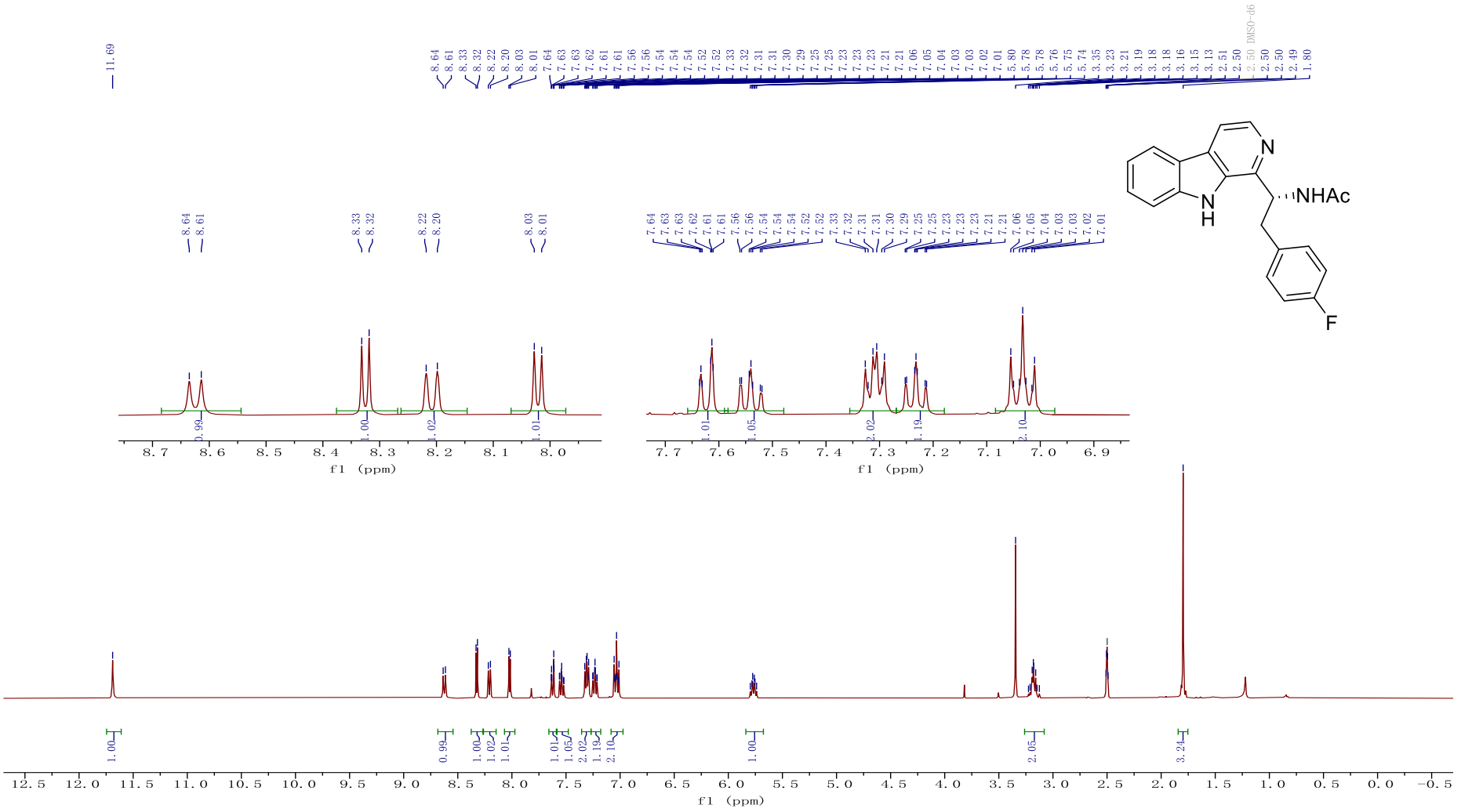
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-bromophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5n**)



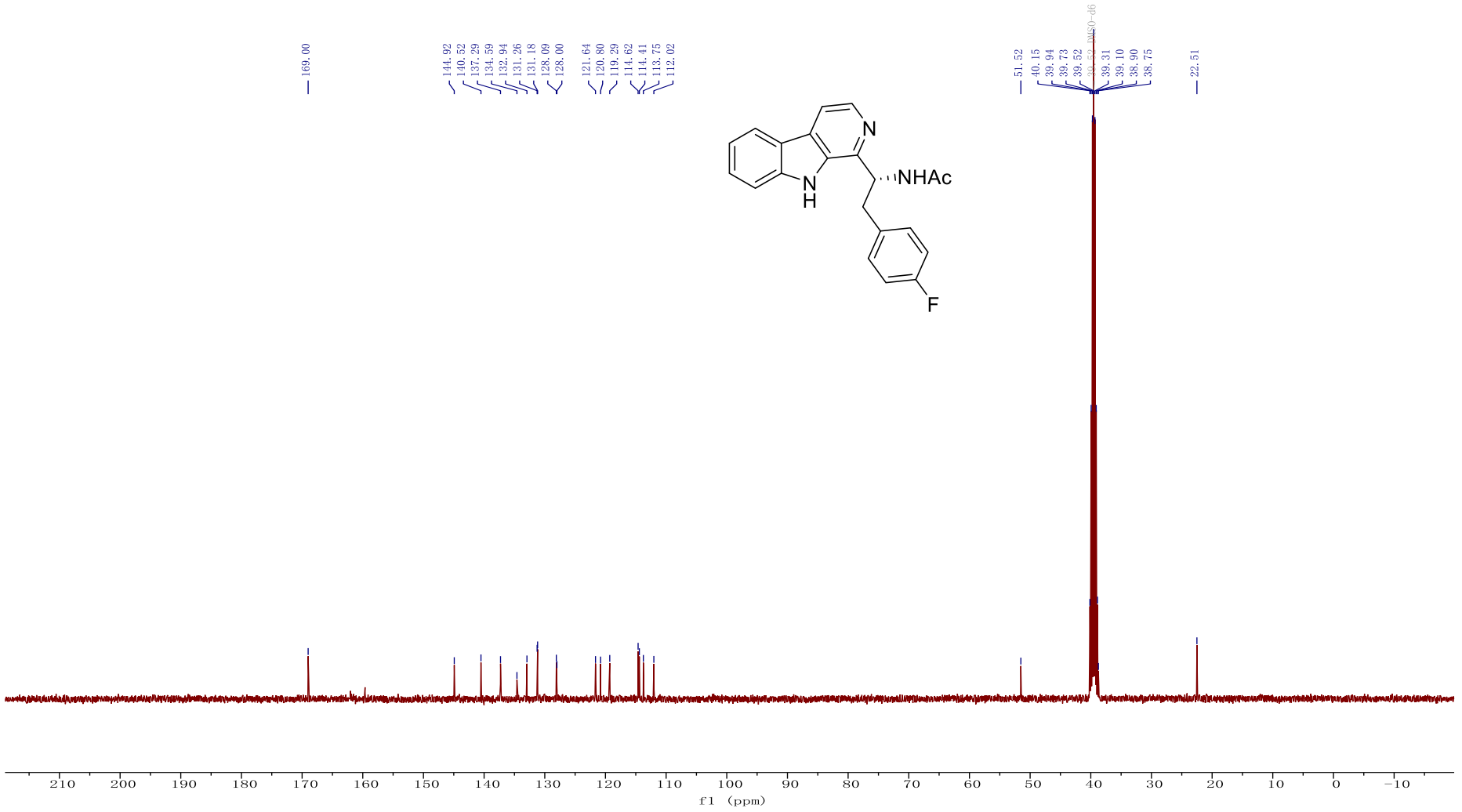
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-bromophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5n**)



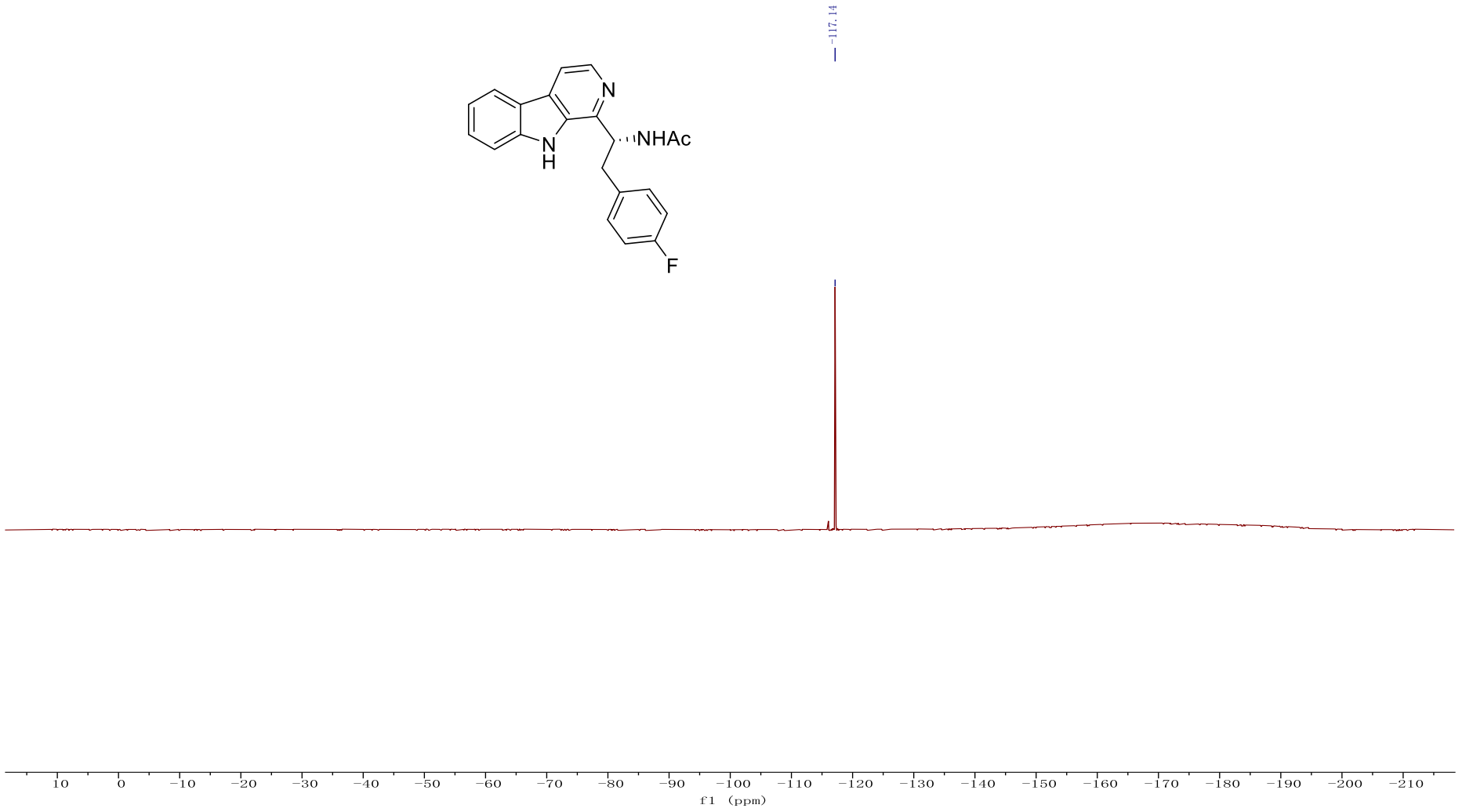
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-fluorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5o**)



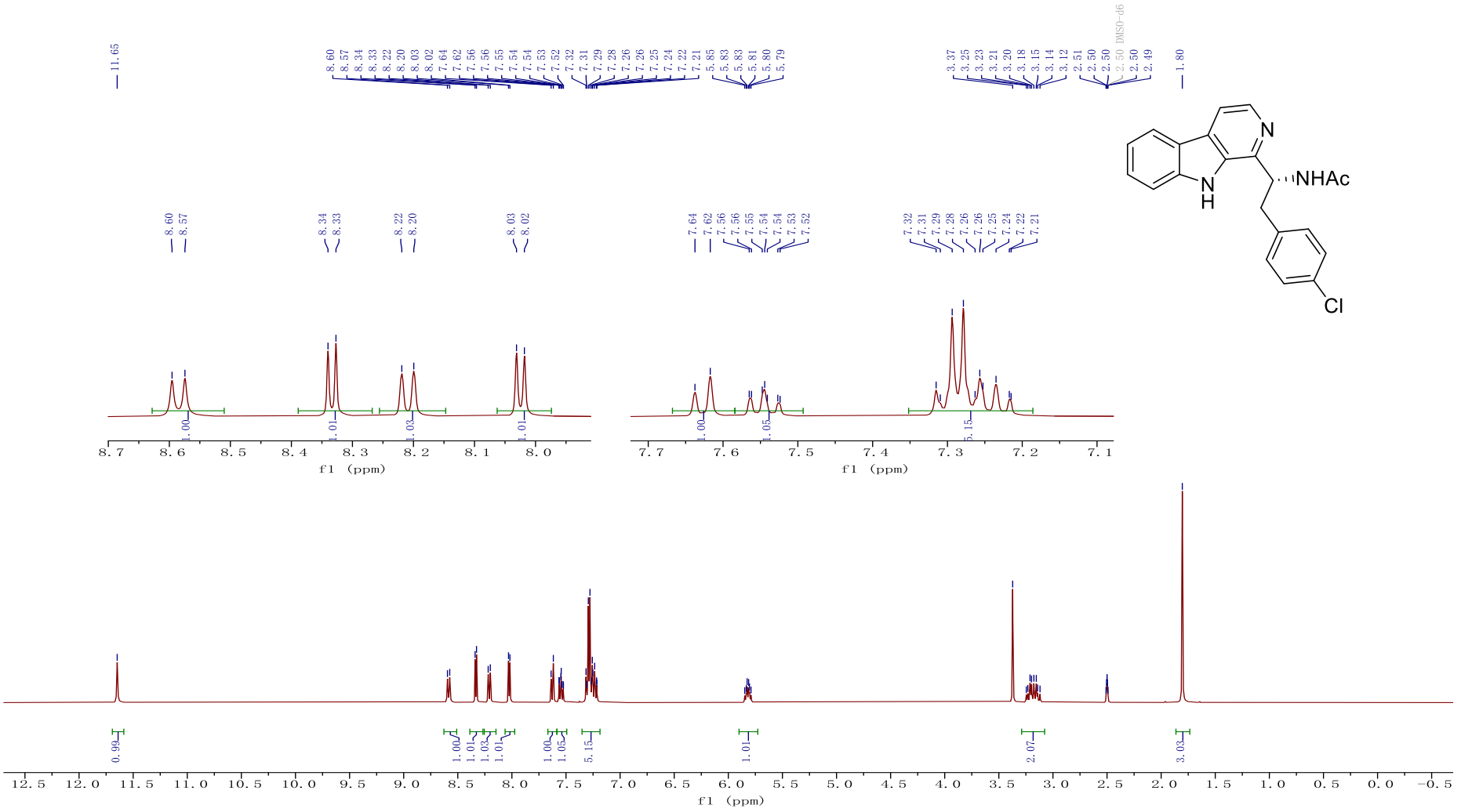
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-fluorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5o**)



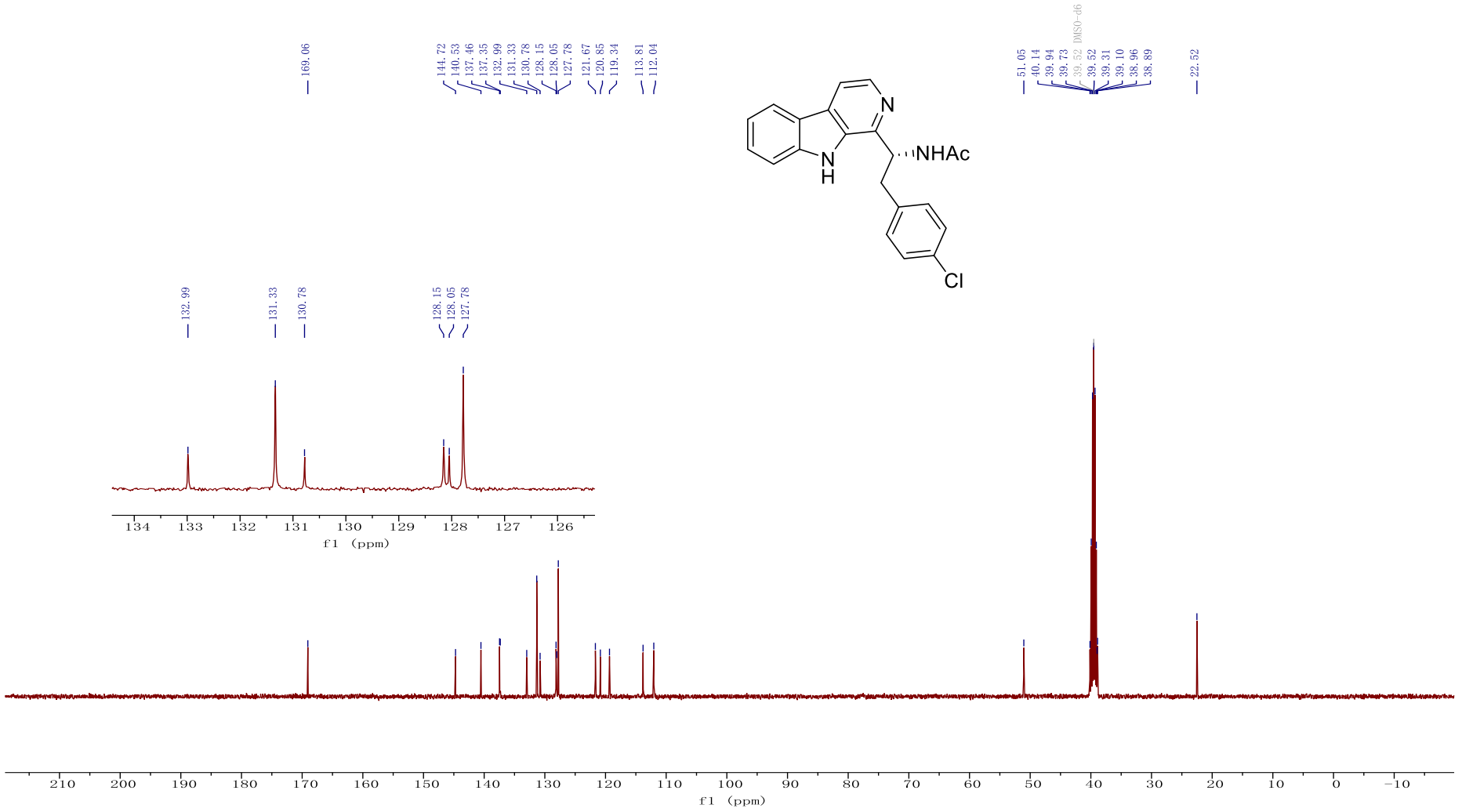
^{19}F NMR (376 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-fluorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5o**)



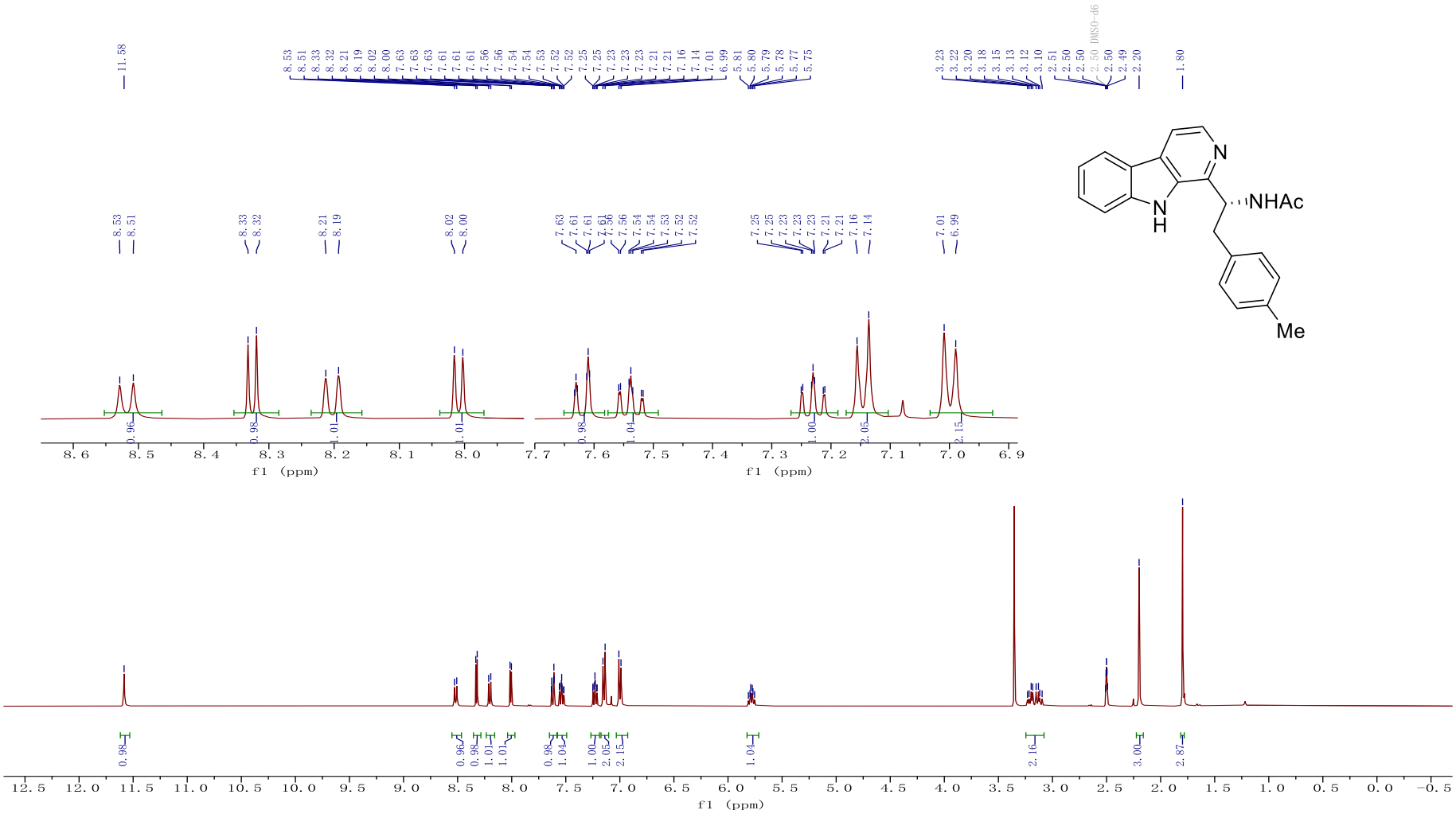
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-chlorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5p**)



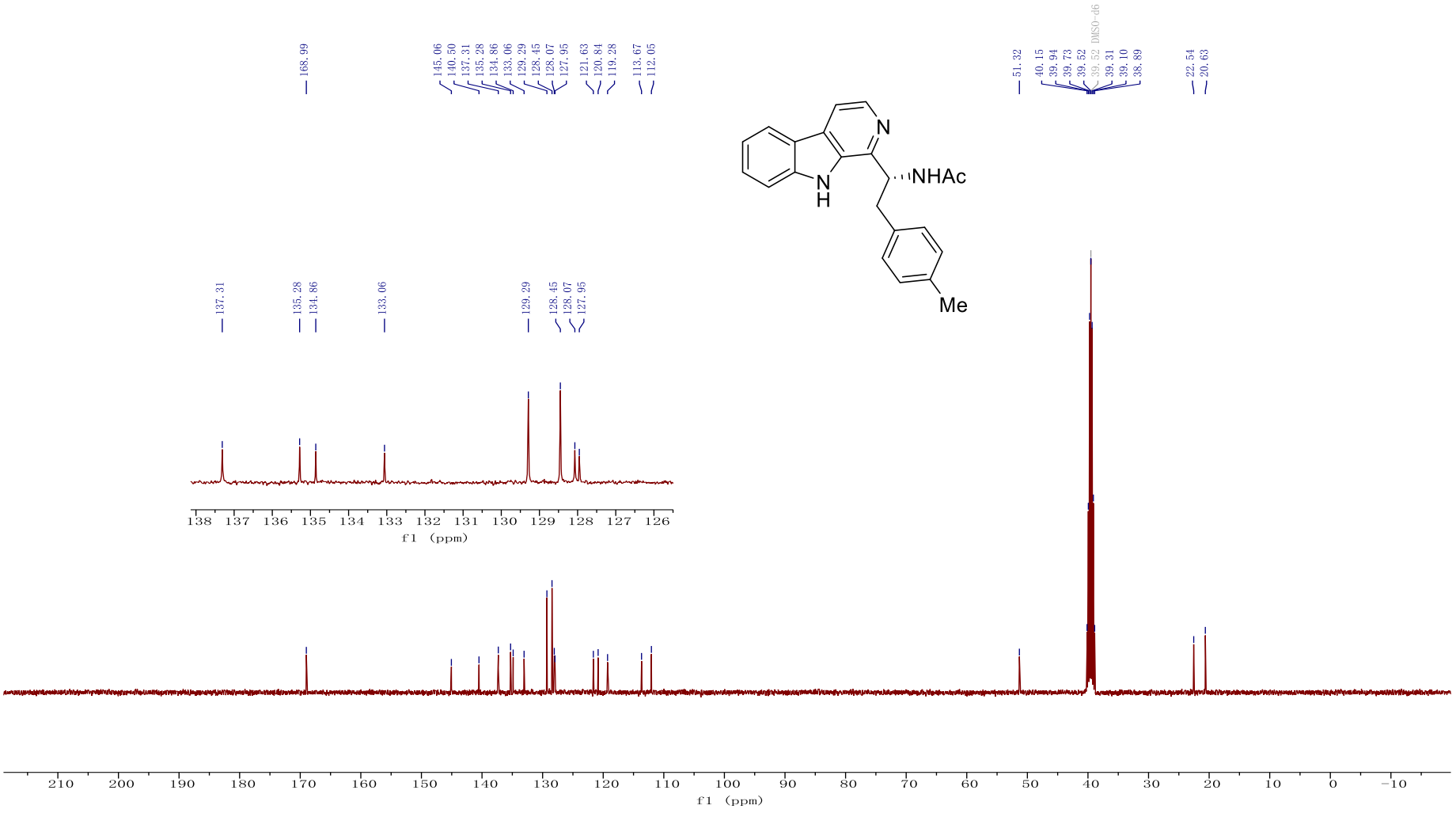
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-chlorophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5p**)



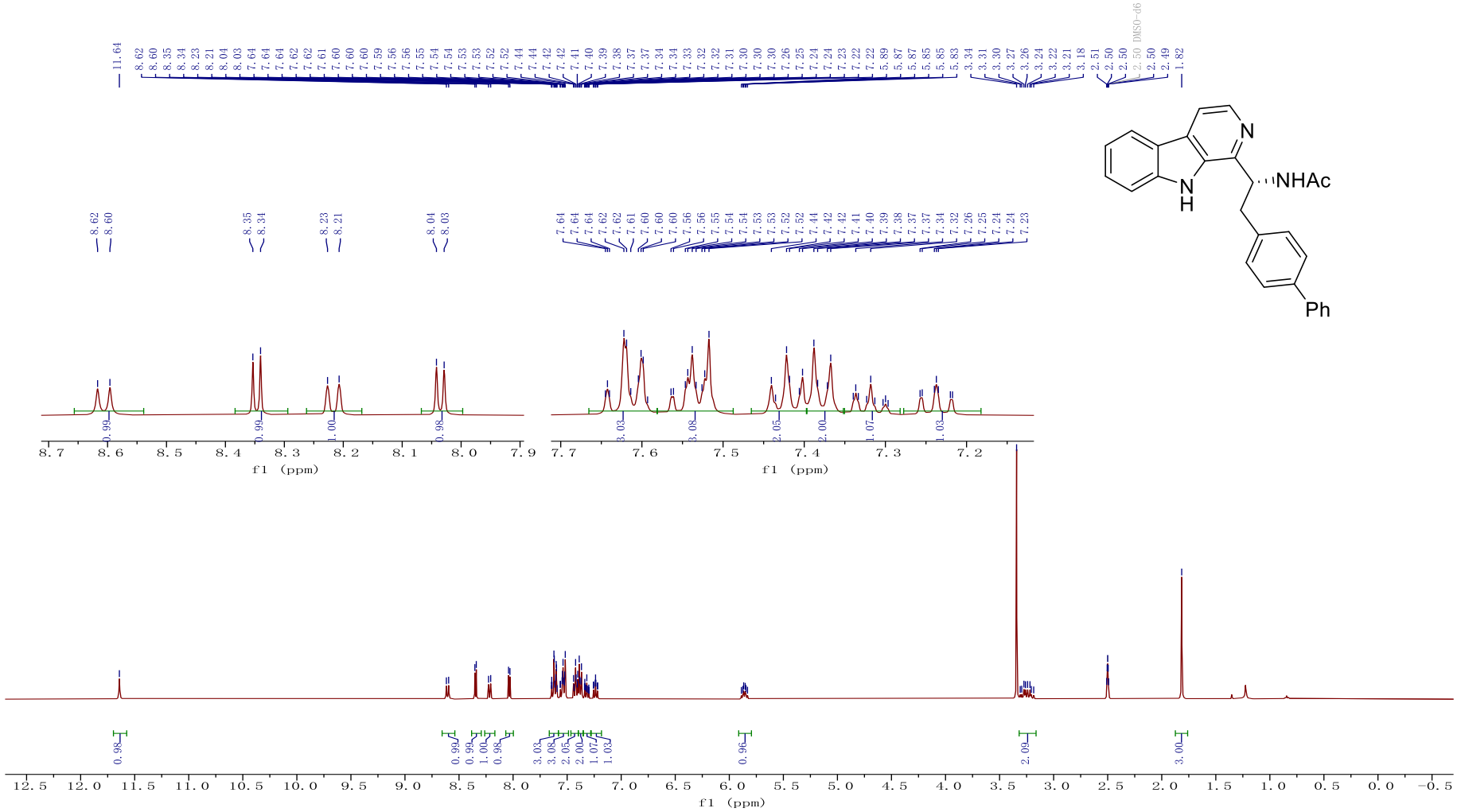
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)-2-(p-tolyl)ethyl)acetamide (**5q**)



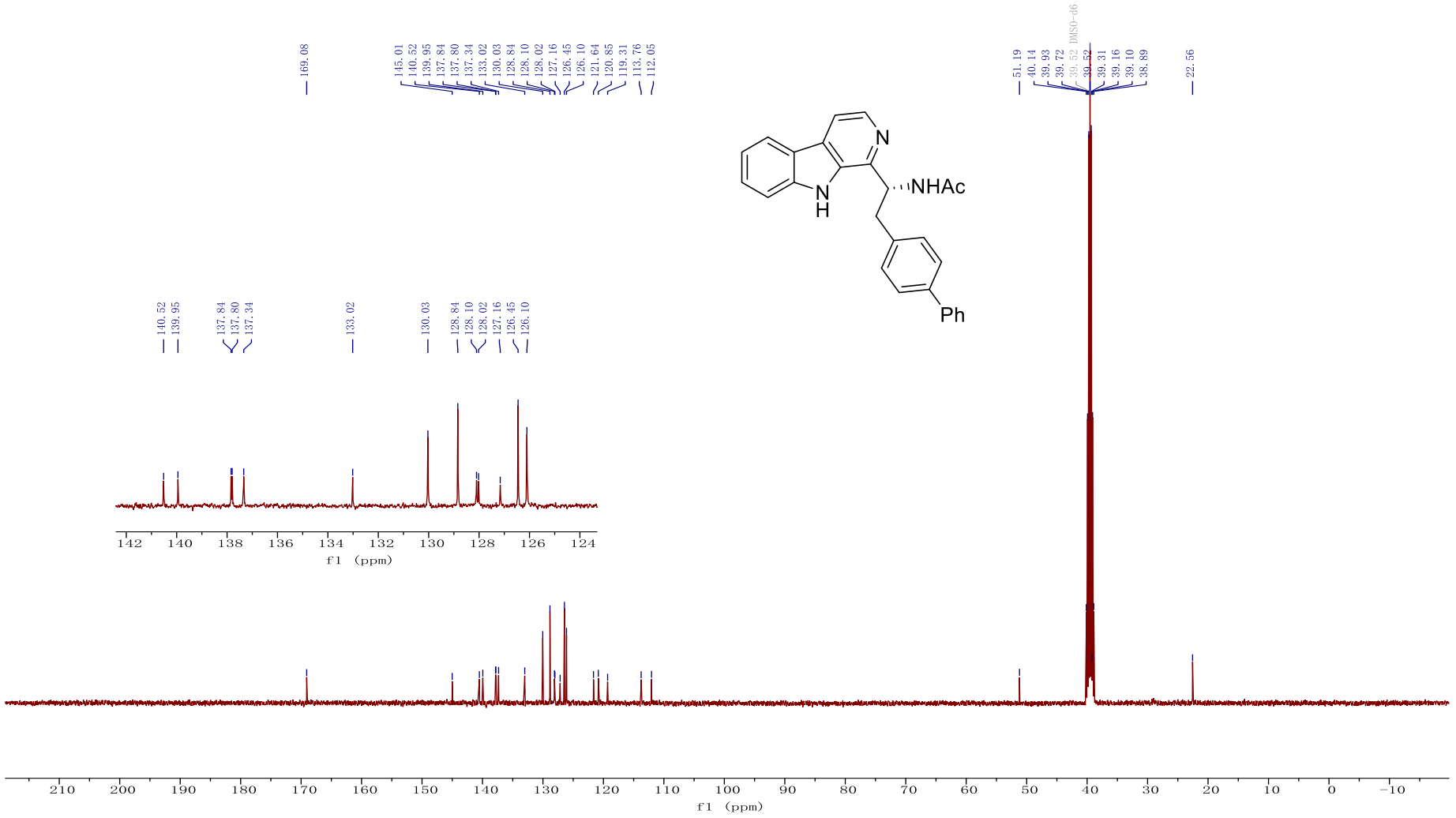
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(1-(9*H*-pyrido[3,4-*b*]indol-1-yl)-2-(p-tolyl)ethyl)acetamide (**5q**)



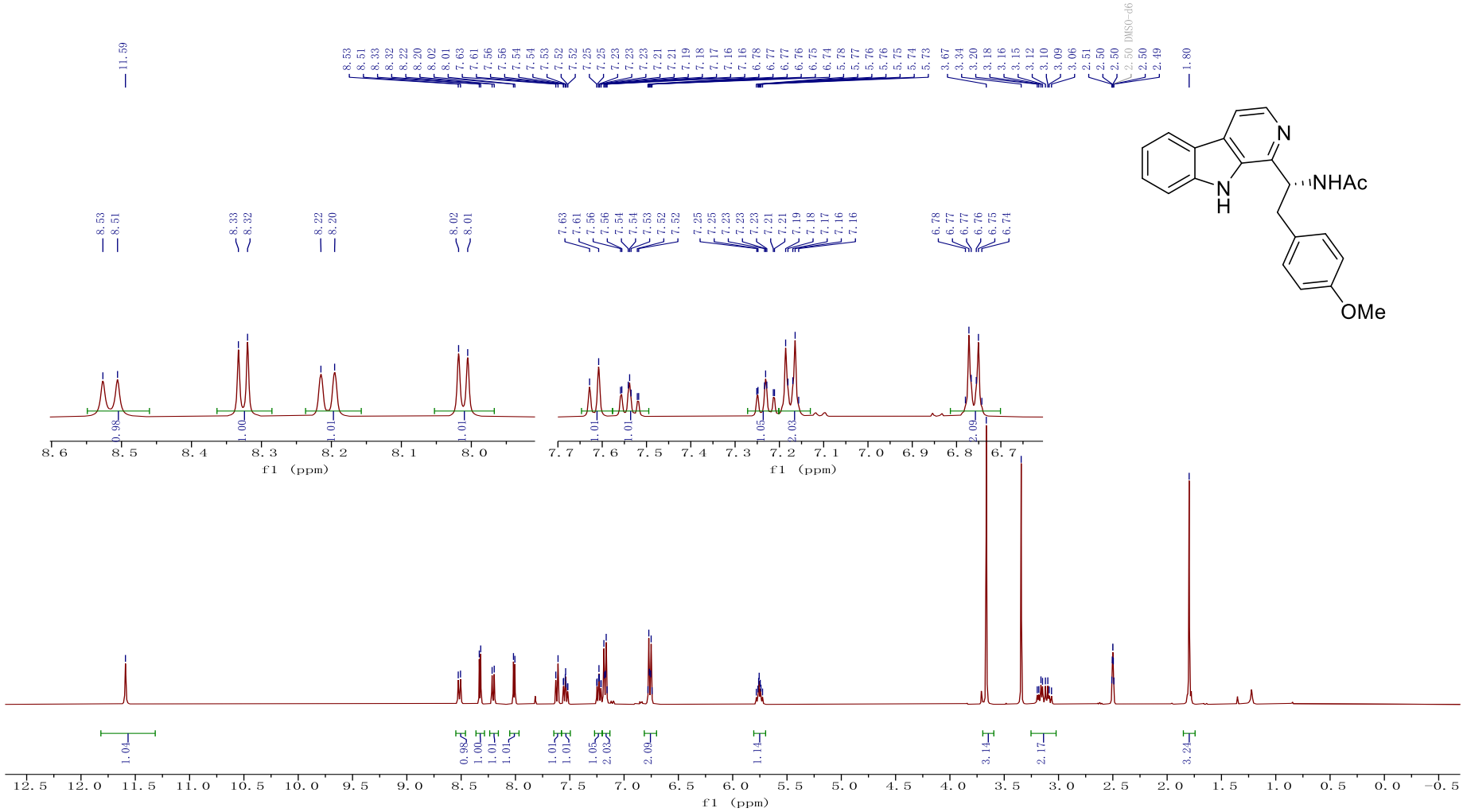
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-([1,1'-biphenyl]-4-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5r**)



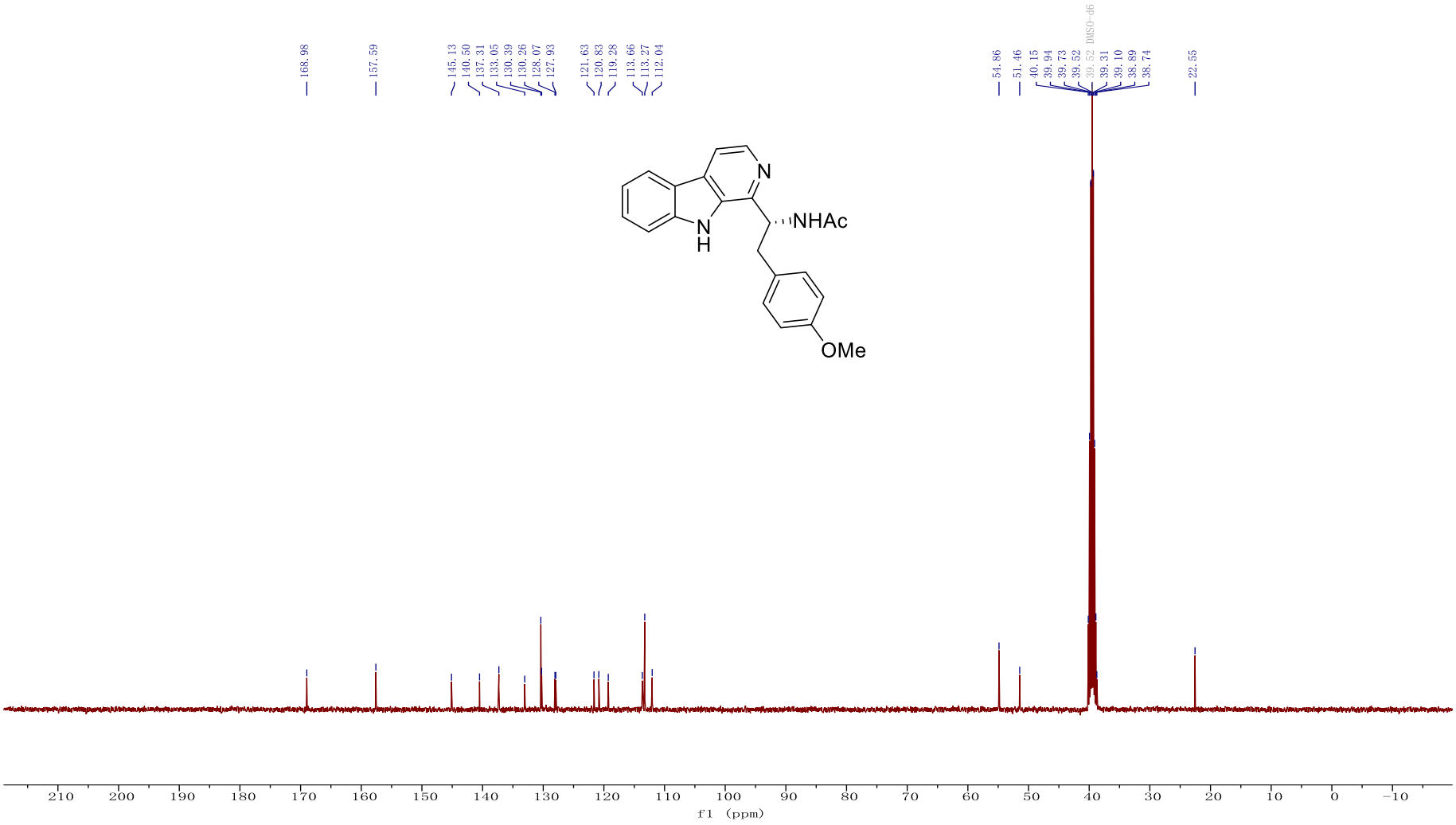
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-([1,1'-biphenyl]-4-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5r**)



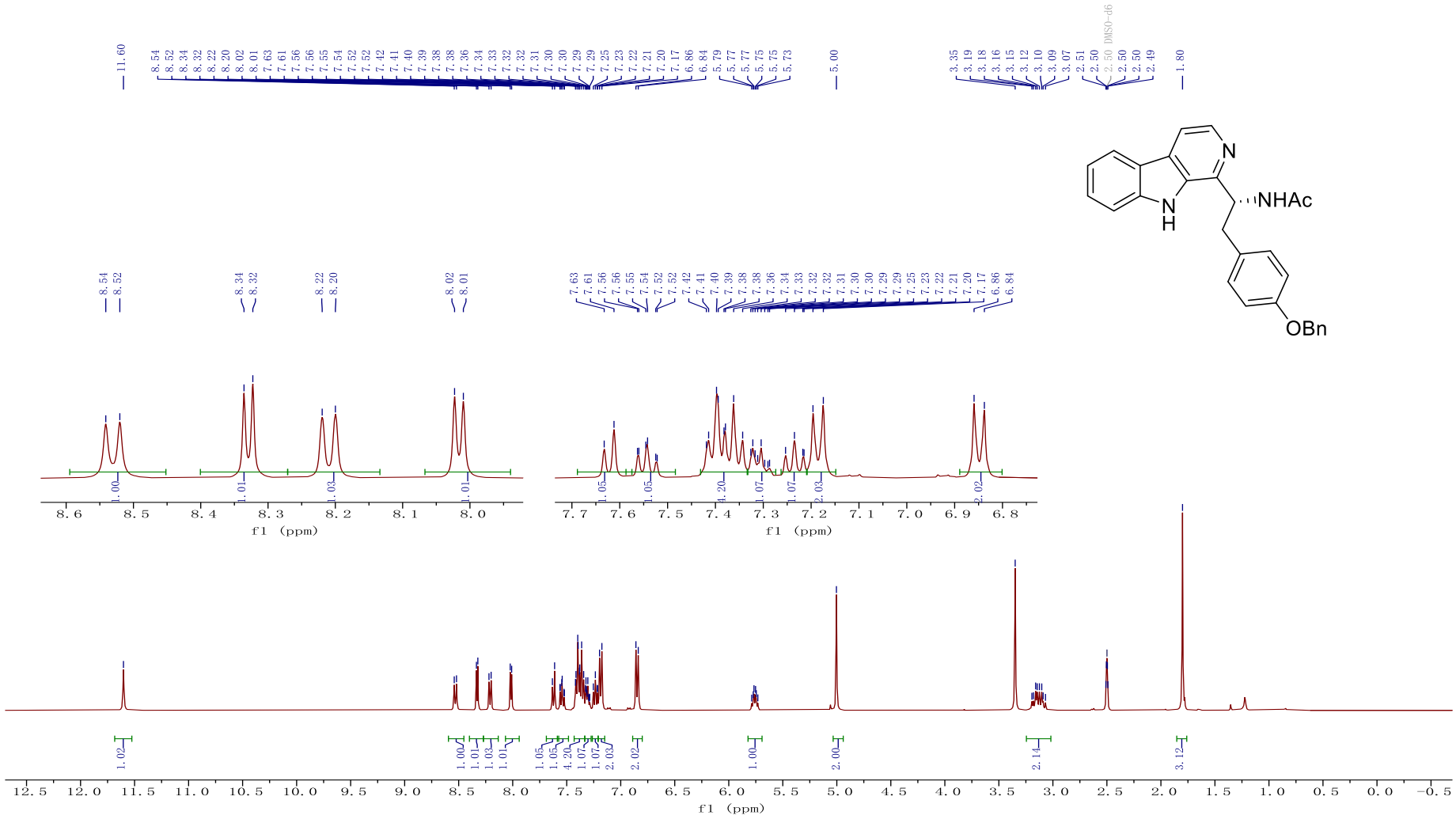
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-methoxyphenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5s**)



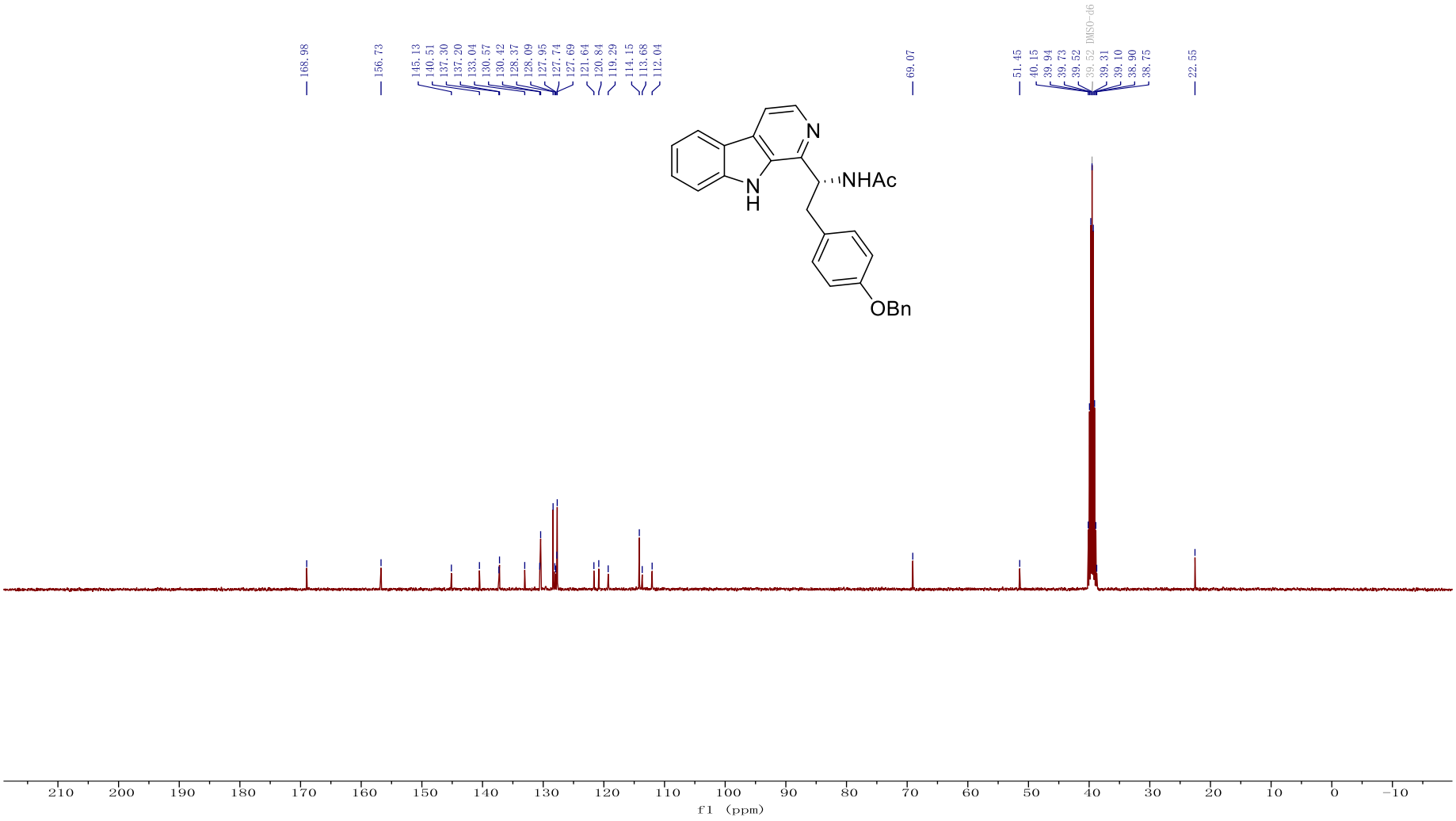
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-methoxyphenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5s**)



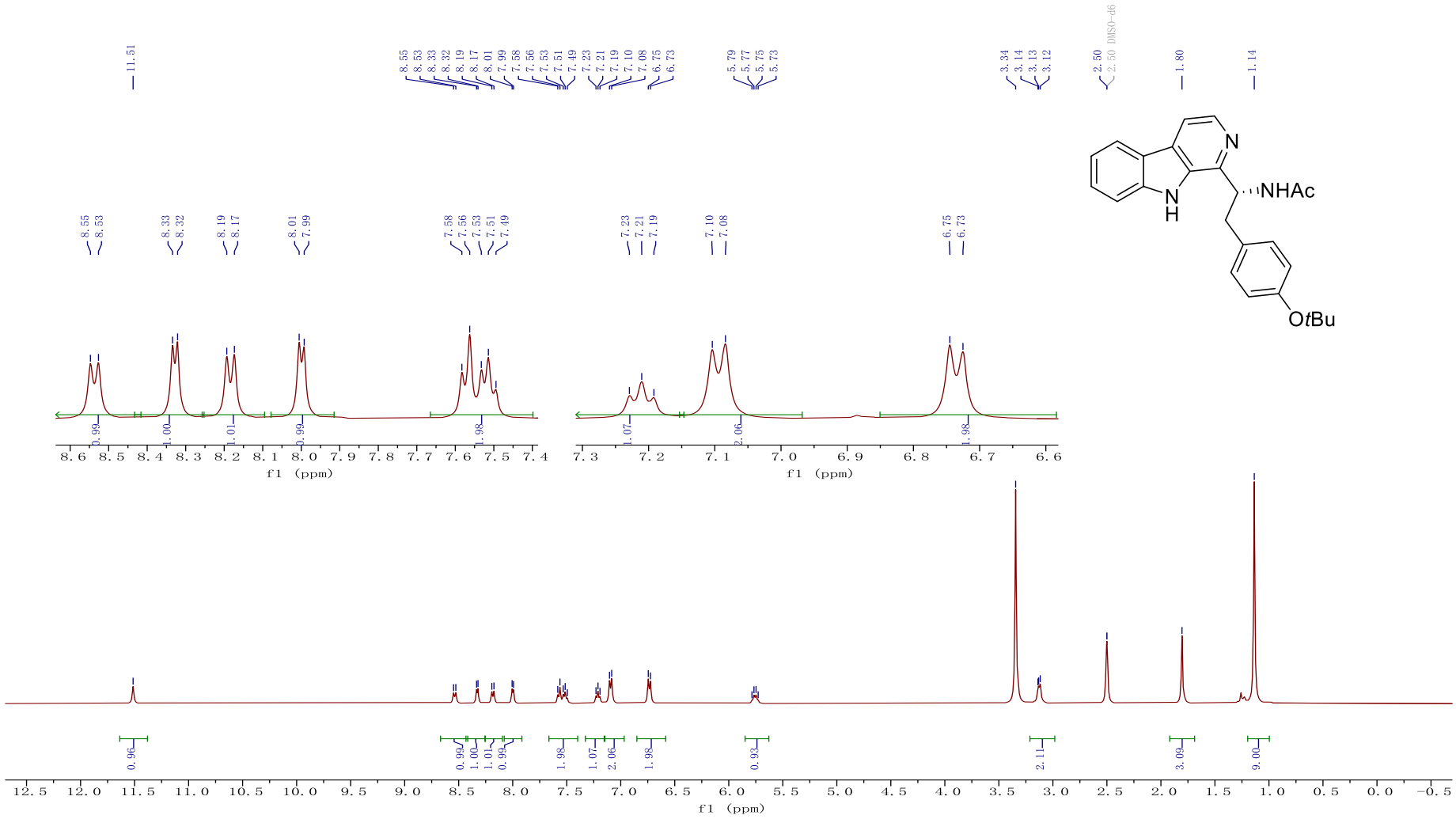
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-(benzyloxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5t**)



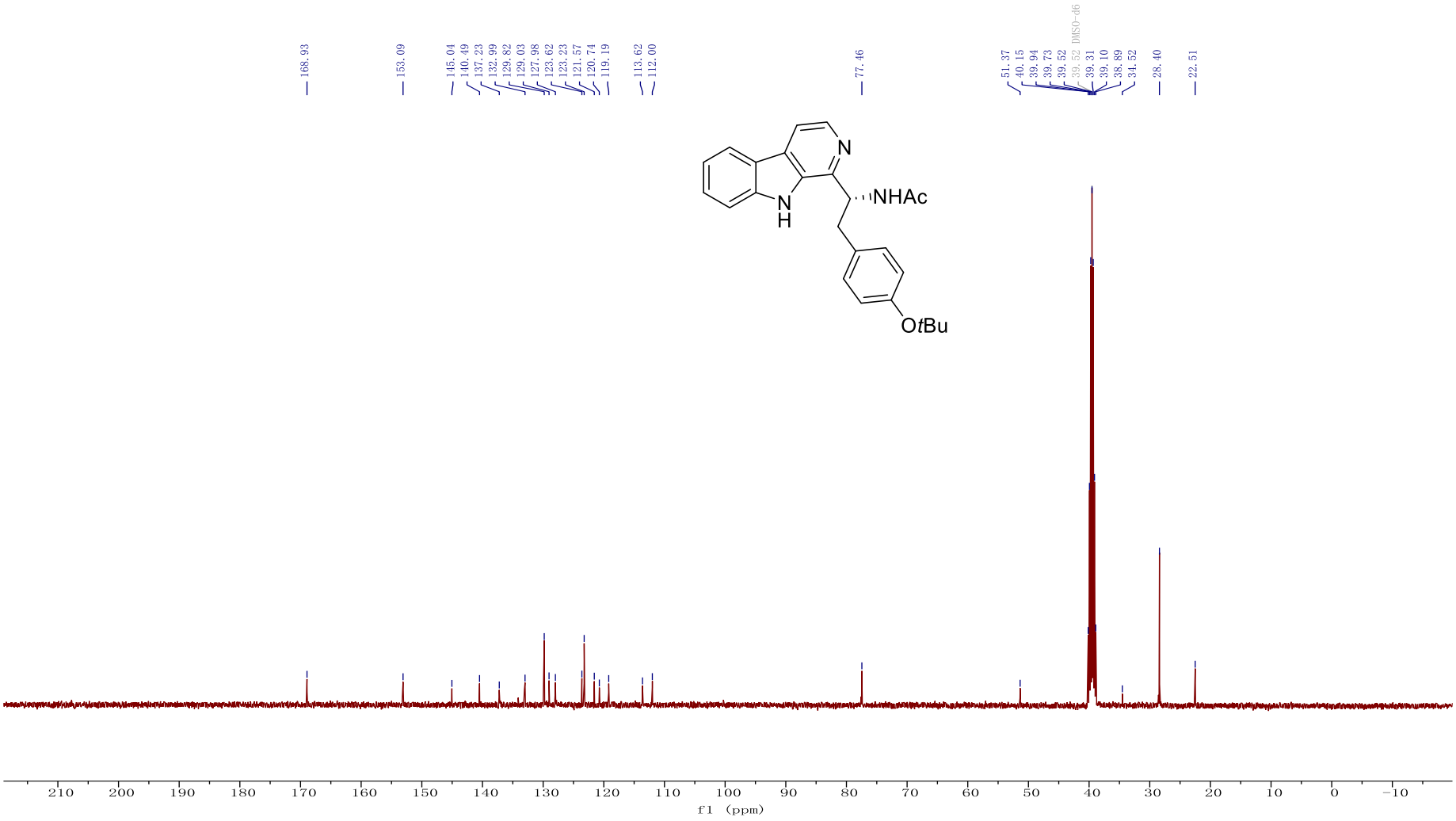
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-(benzyloxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5t**)



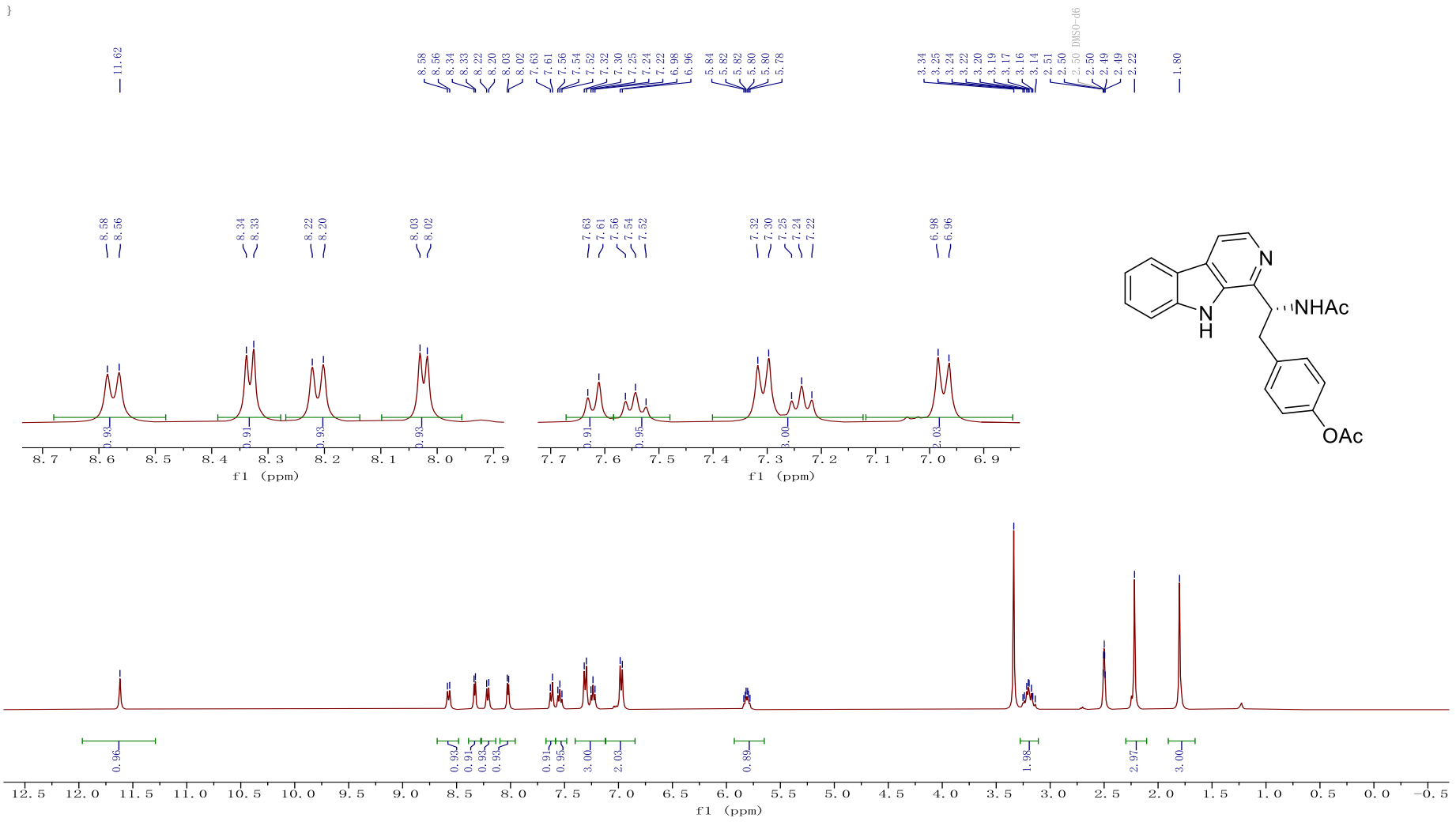
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-(tert-butoxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5u**)



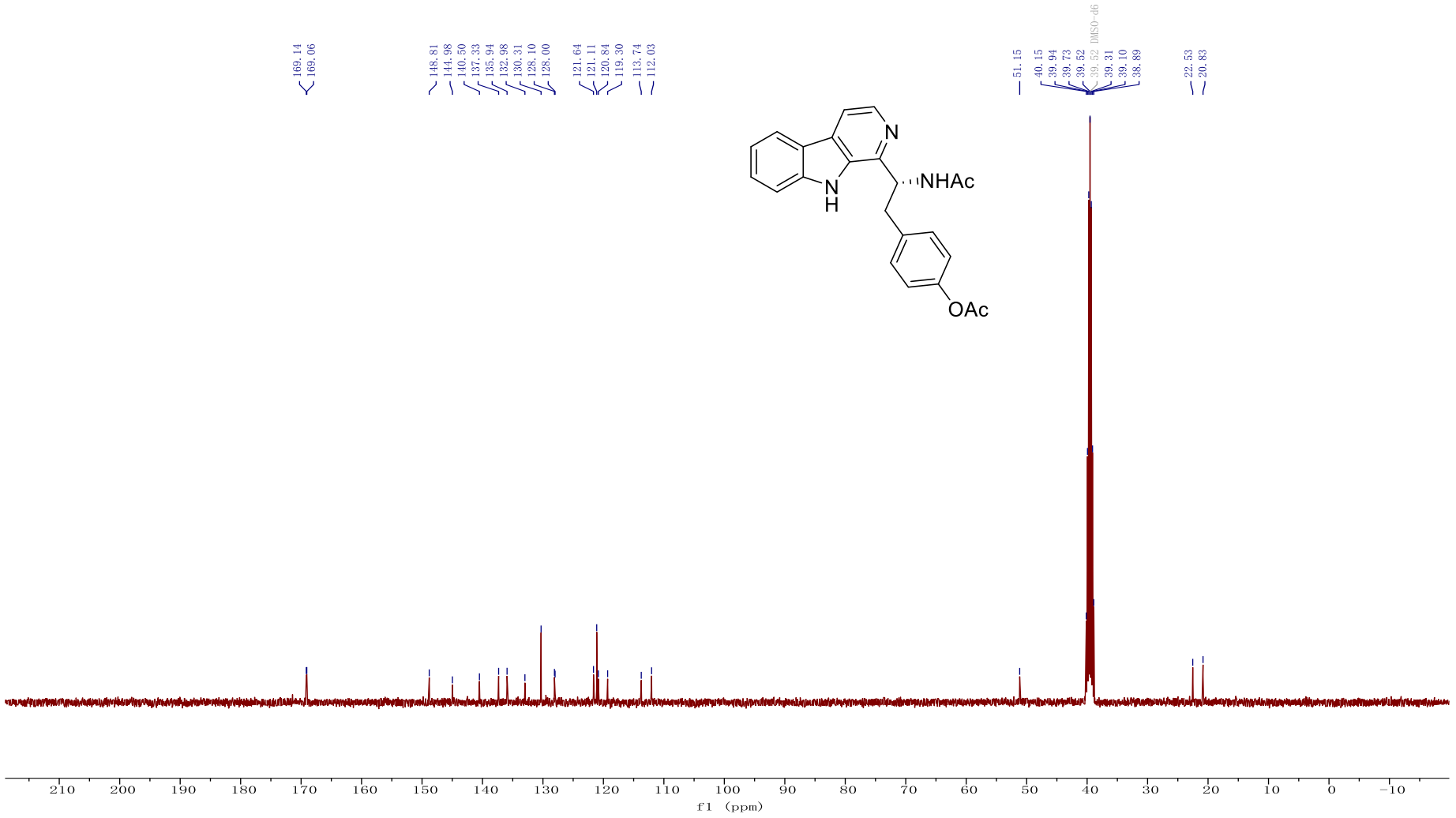
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-(tert-butoxy)phenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5u**)



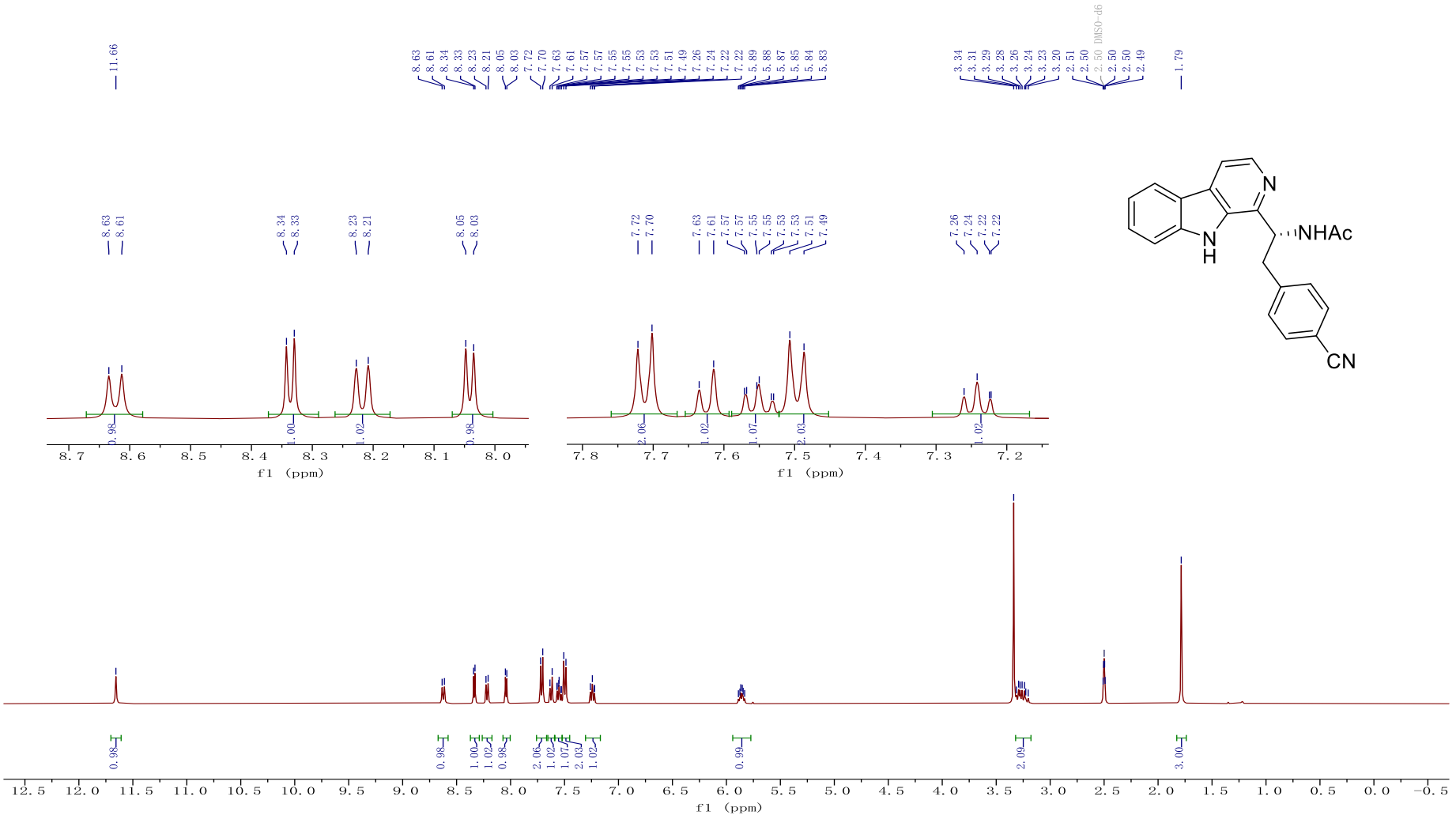
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-4-(2-acetamido-2-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)phenyl acetate (**5v**)



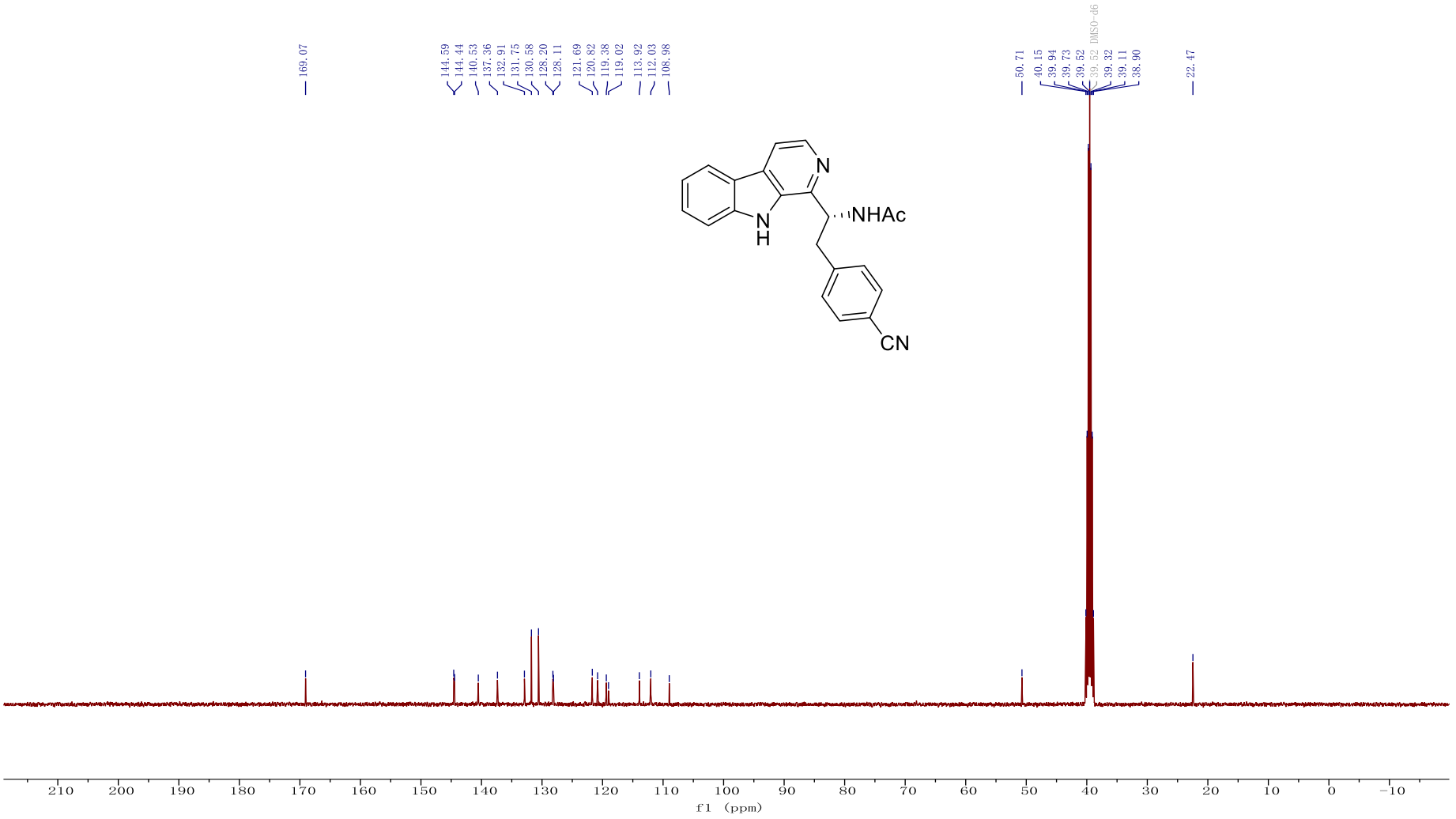
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-4-(2-acetamido-2-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)phenyl acetate (**5v**)



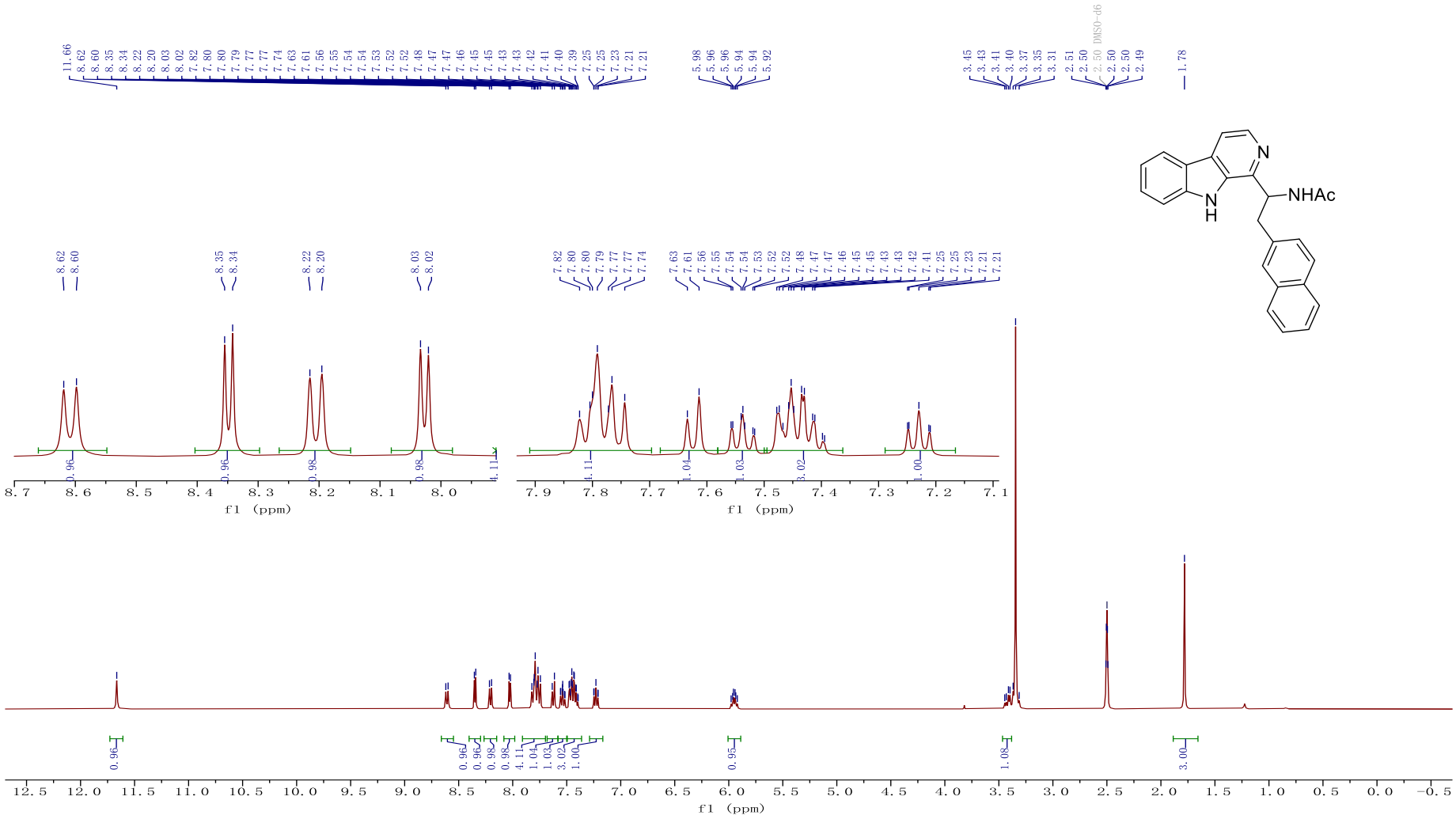
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-cyanophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5w**)



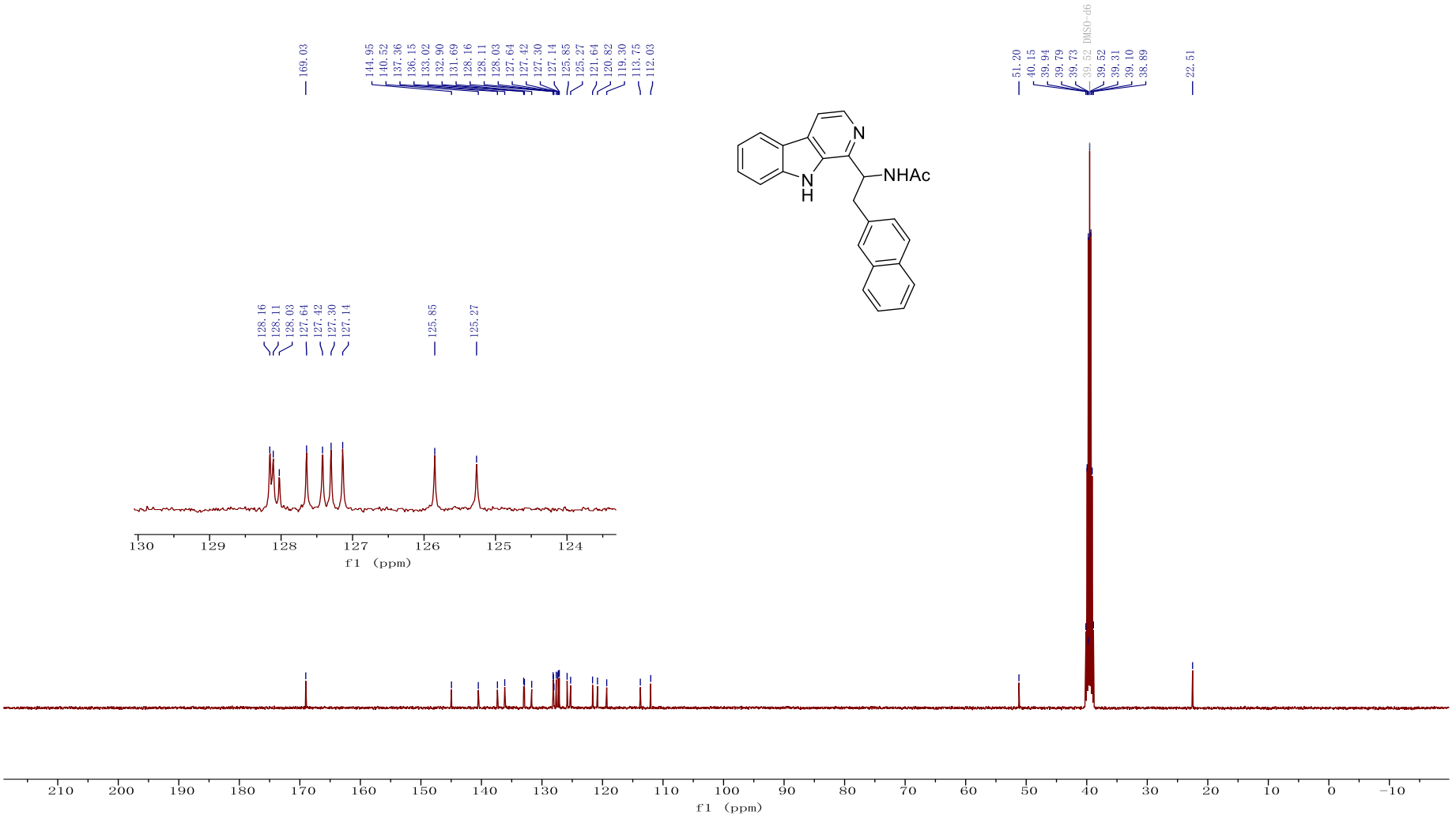
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(4-cyanophenyl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5w**)



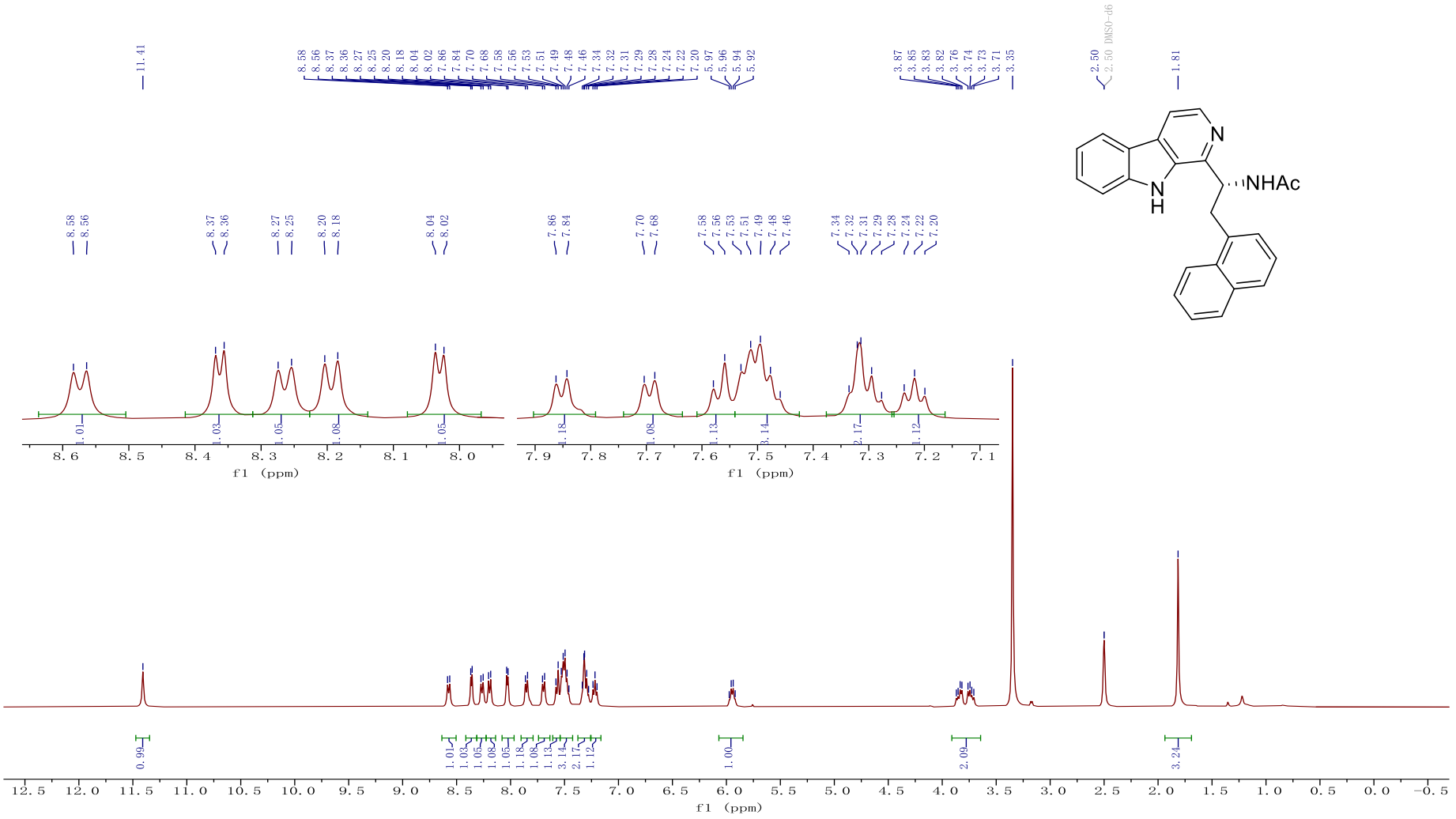
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(naphthalen-1-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5x**)



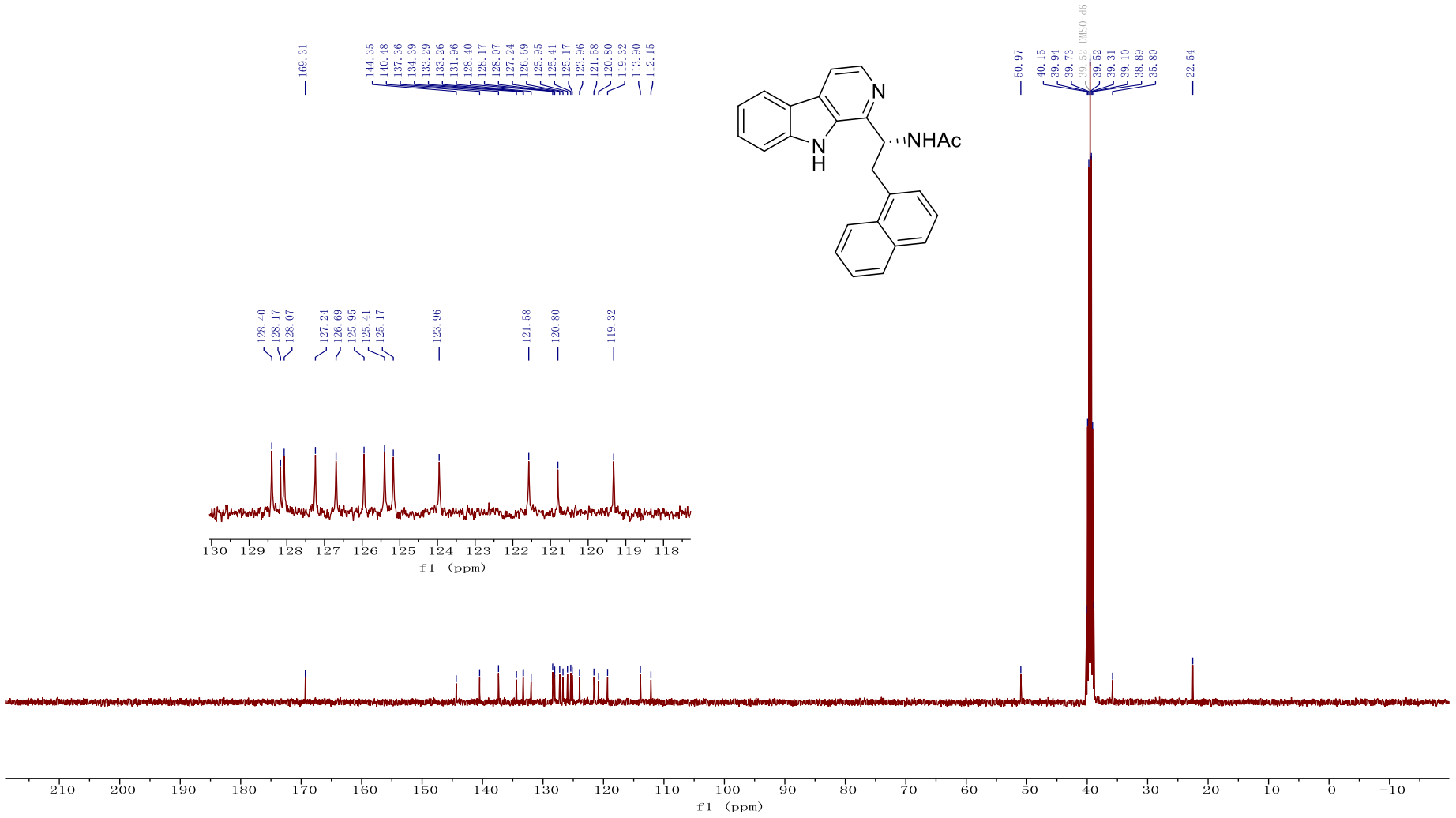
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(naphthalen-1-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5x**)



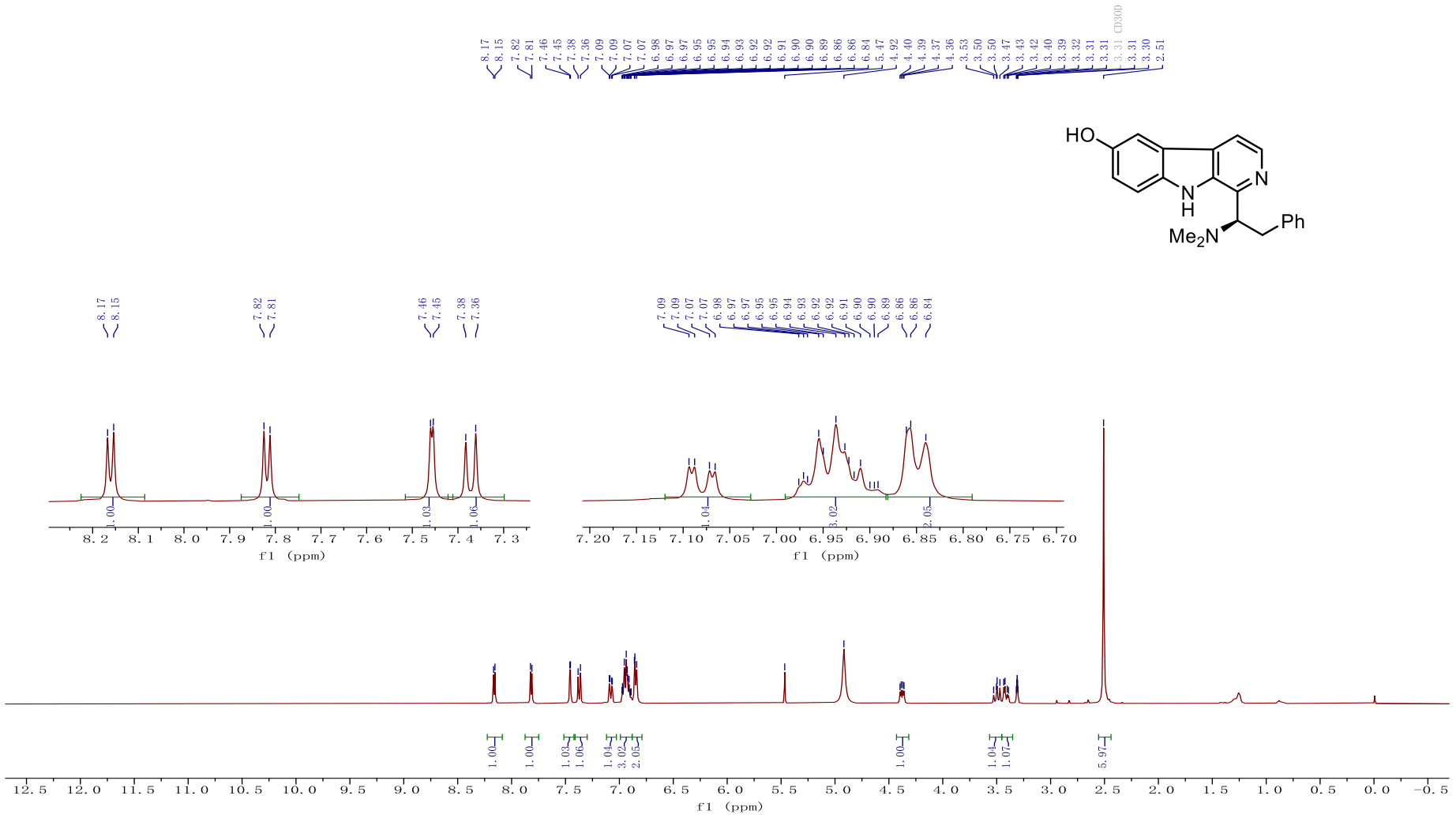
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-*N*-(2-(naphthalen-1-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5y**)



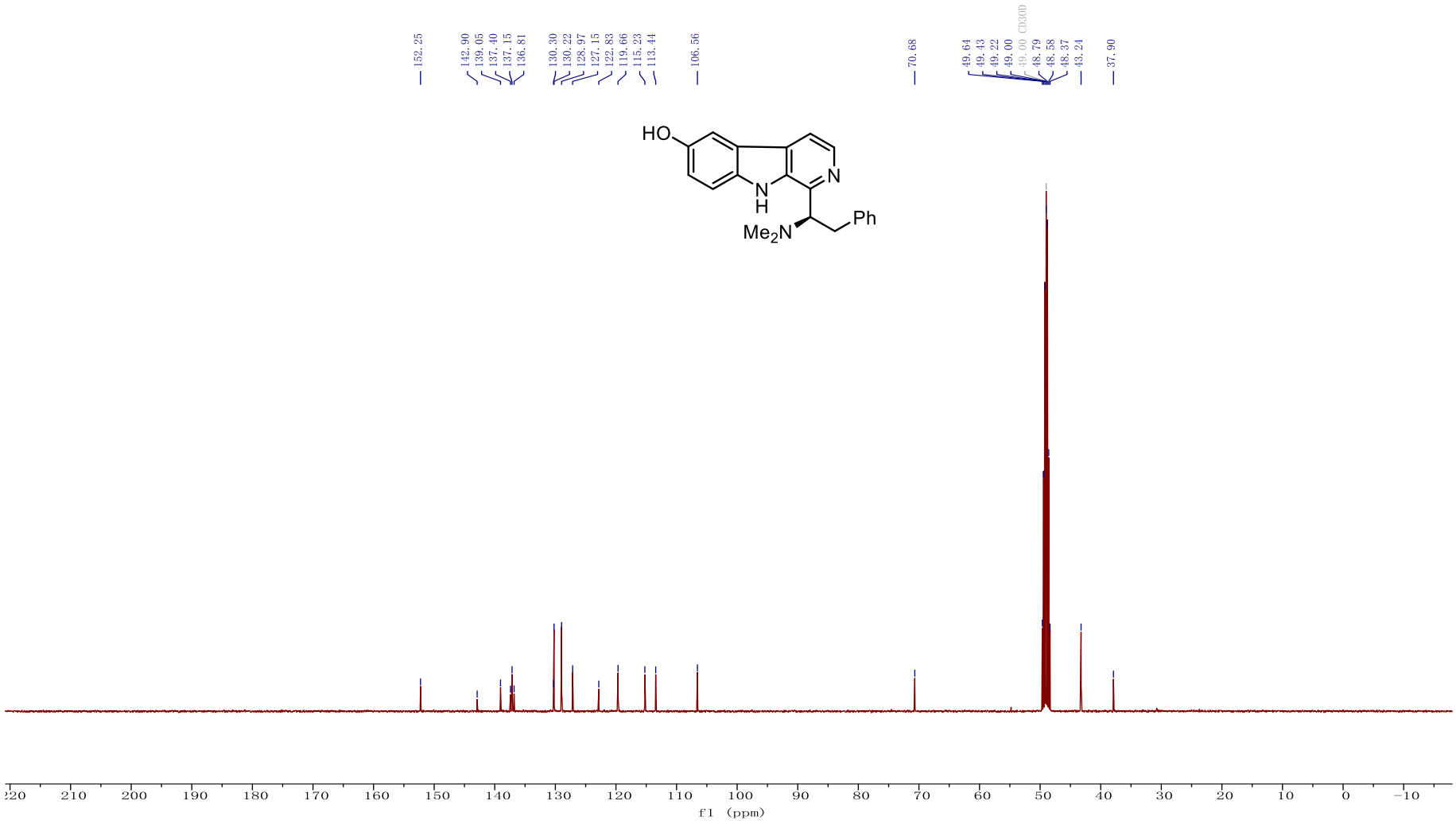
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-*N*-(2-(naphthalen-1-yl)-1-(9*H*-pyrido[3,4-*b*]indol-1-yl)ethyl)acetamide (**5y**)



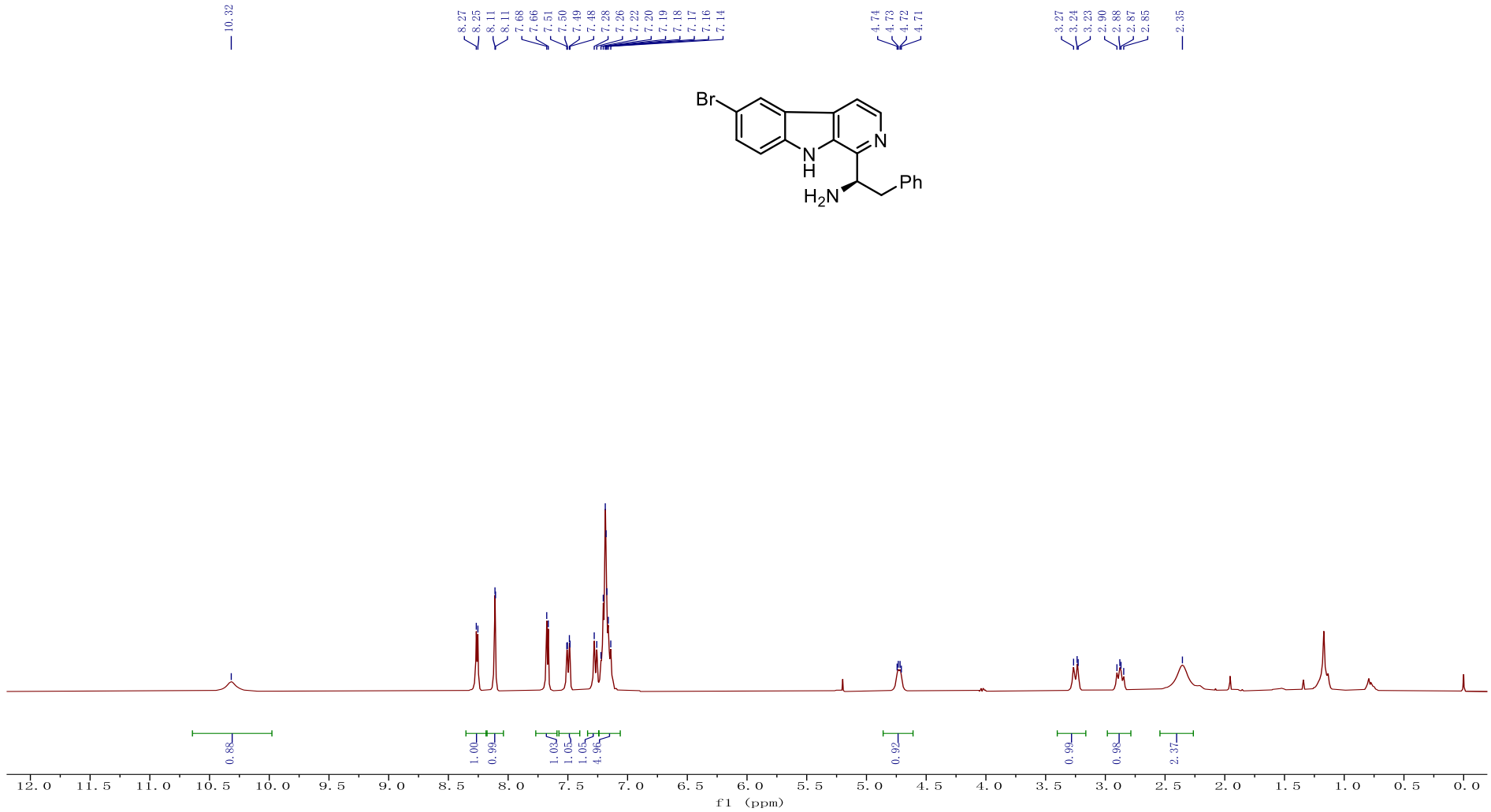
^{1}H NMR (400 MHz, CD_3OD) (*R*)-1-(1-(dimethylamino)-2-phenylethyl)-9*H*-pyrido[3,4-*b*]indol-6-ol (**7**, **eudistomin X**)



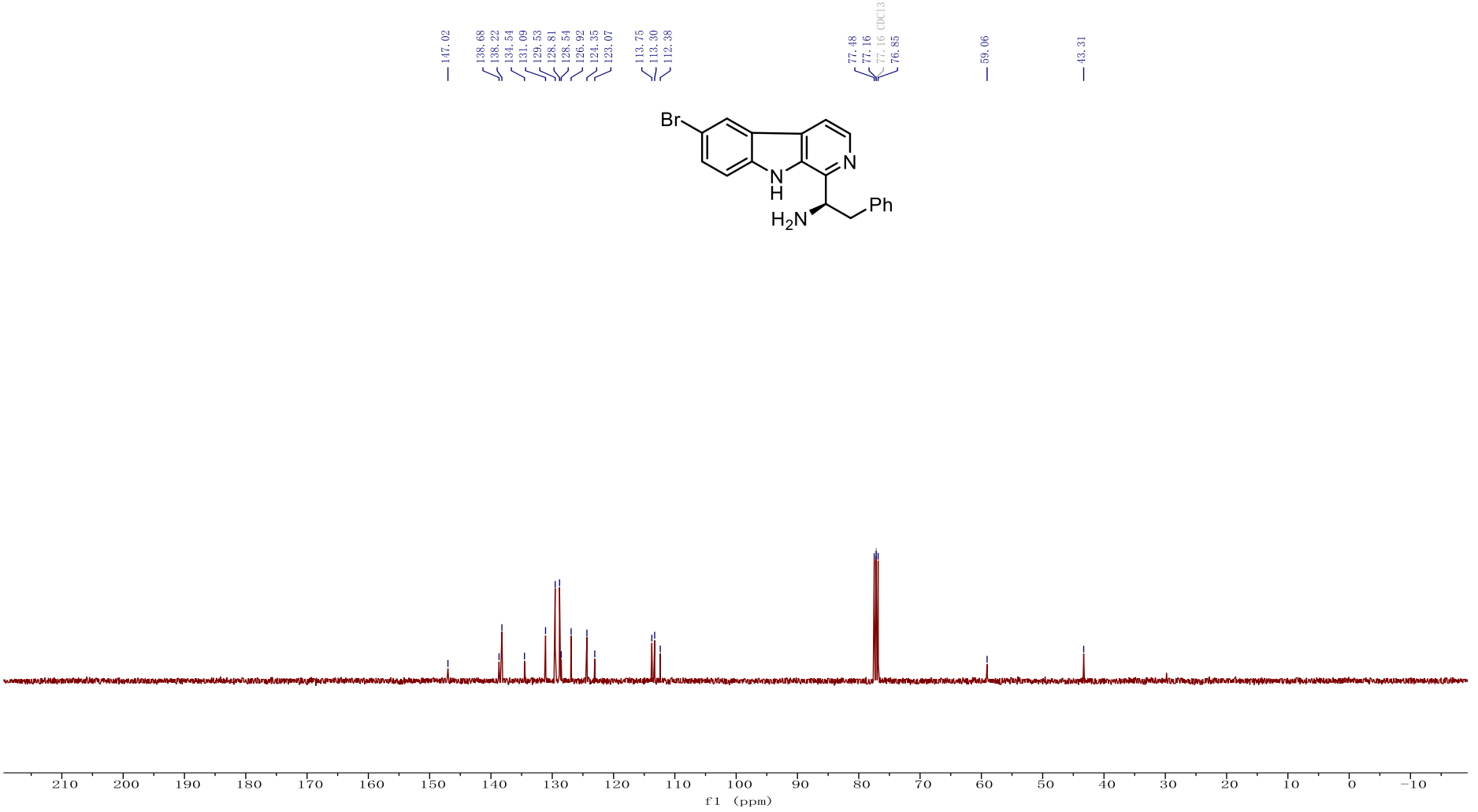
^{13}C NMR (101 MHz, CD_3OD) (*R*)-1-(1-(dimethylamino)-2-phenylethyl)-9*H*-pyrido[3,4-*b*]indol-6-ol (**7**, **eudistomin X**)



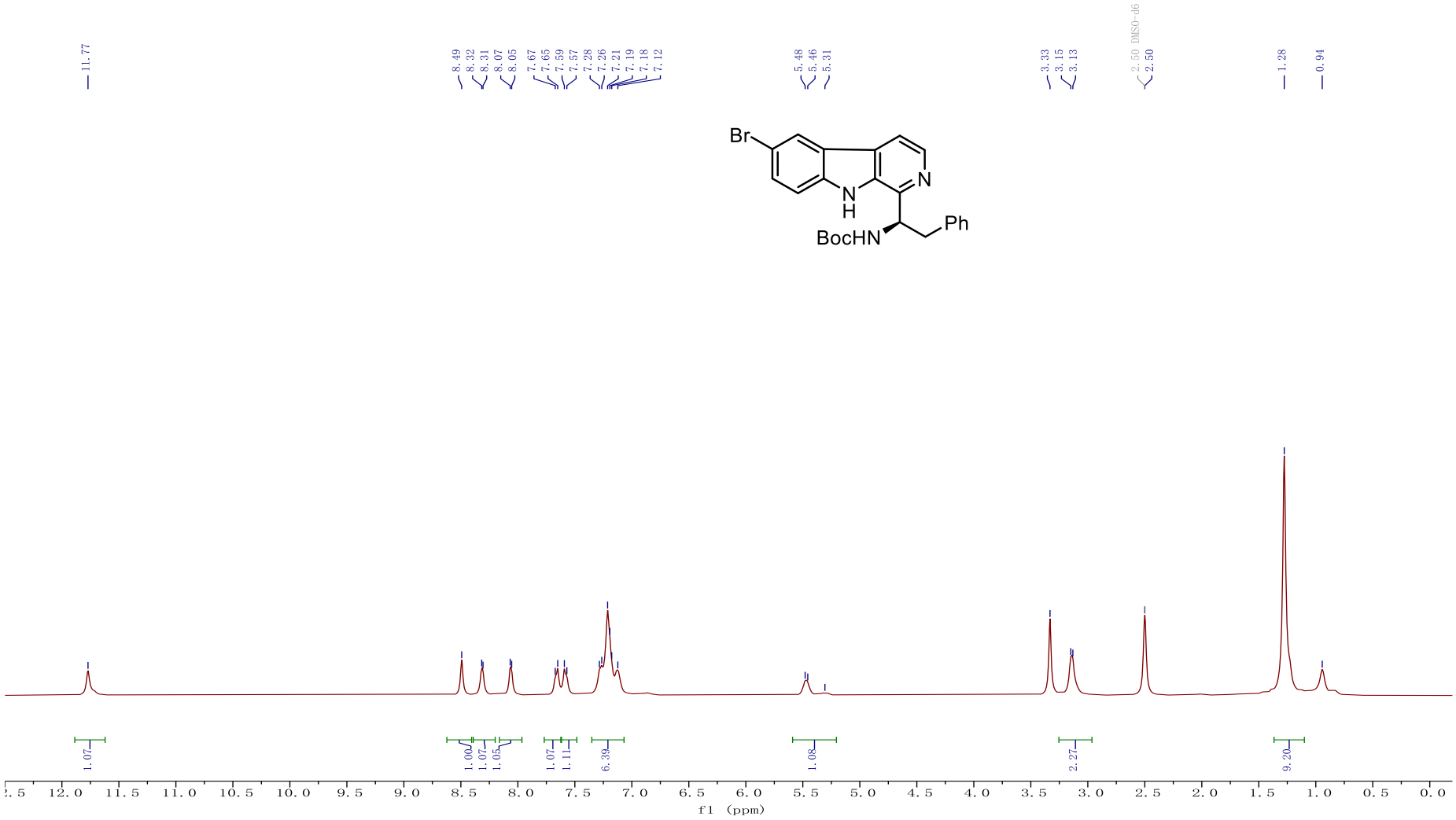
^{1}H NMR (400 MHz, $CDCl_3$) (*R*)-1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethan-1-amine (**8**)



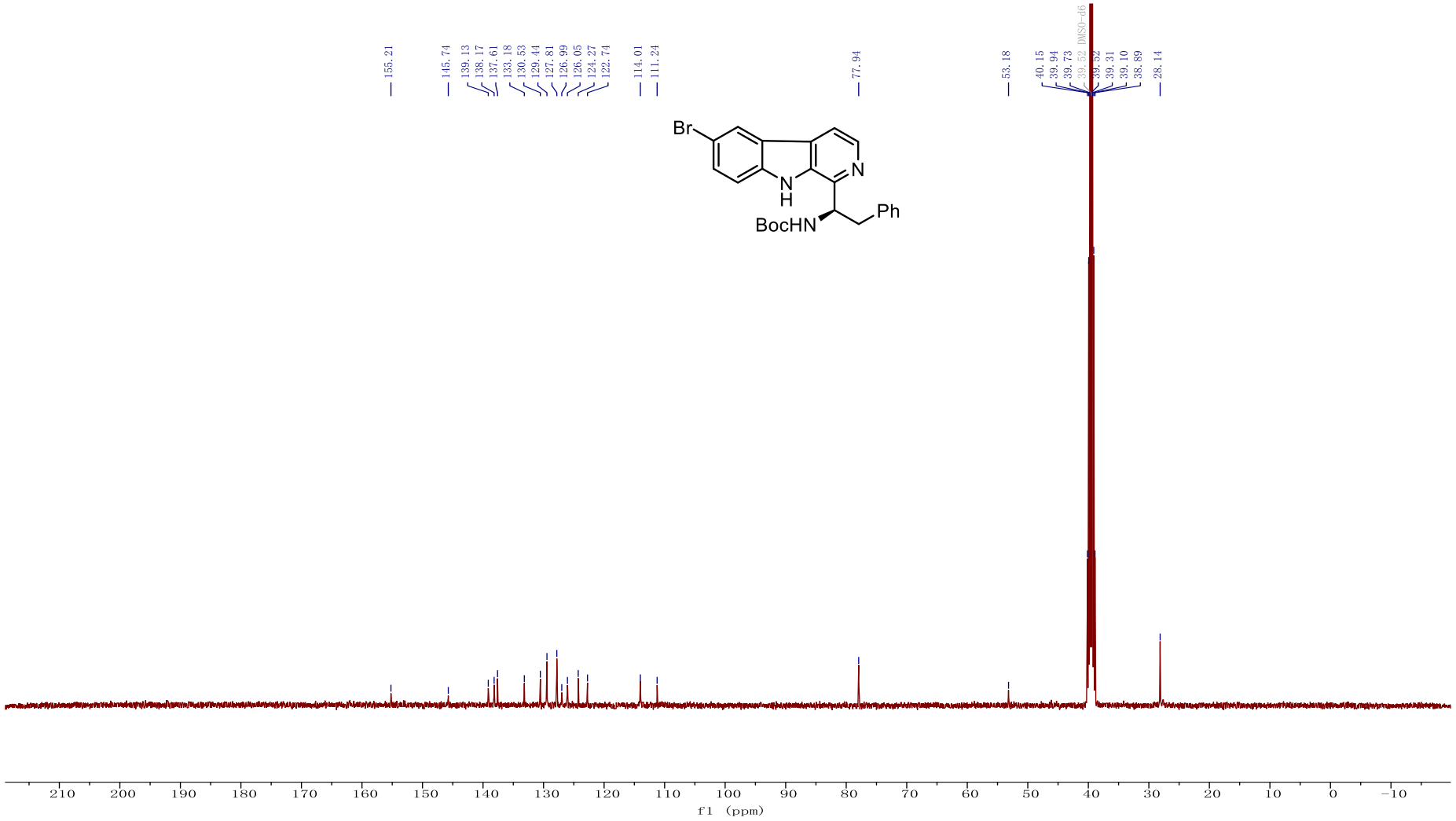
^{13}C NMR (400 MHz, $CDCl_3$) (*R*)-1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethan-1-amine (**8**)



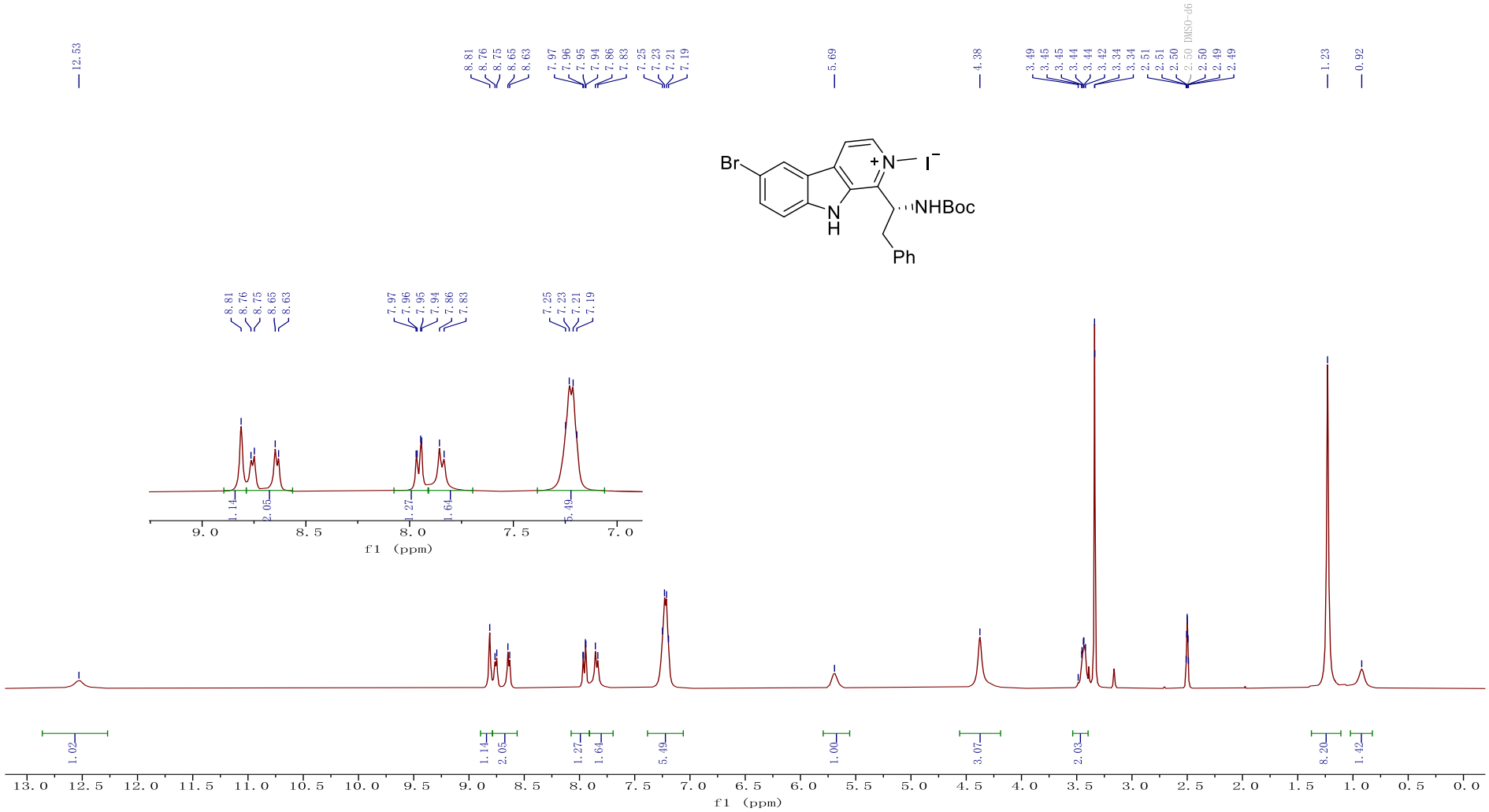
^{1}H NMR (400 MHz, DMSO-d_6) tert-butyl (*R*)-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)carbamate (**9**)



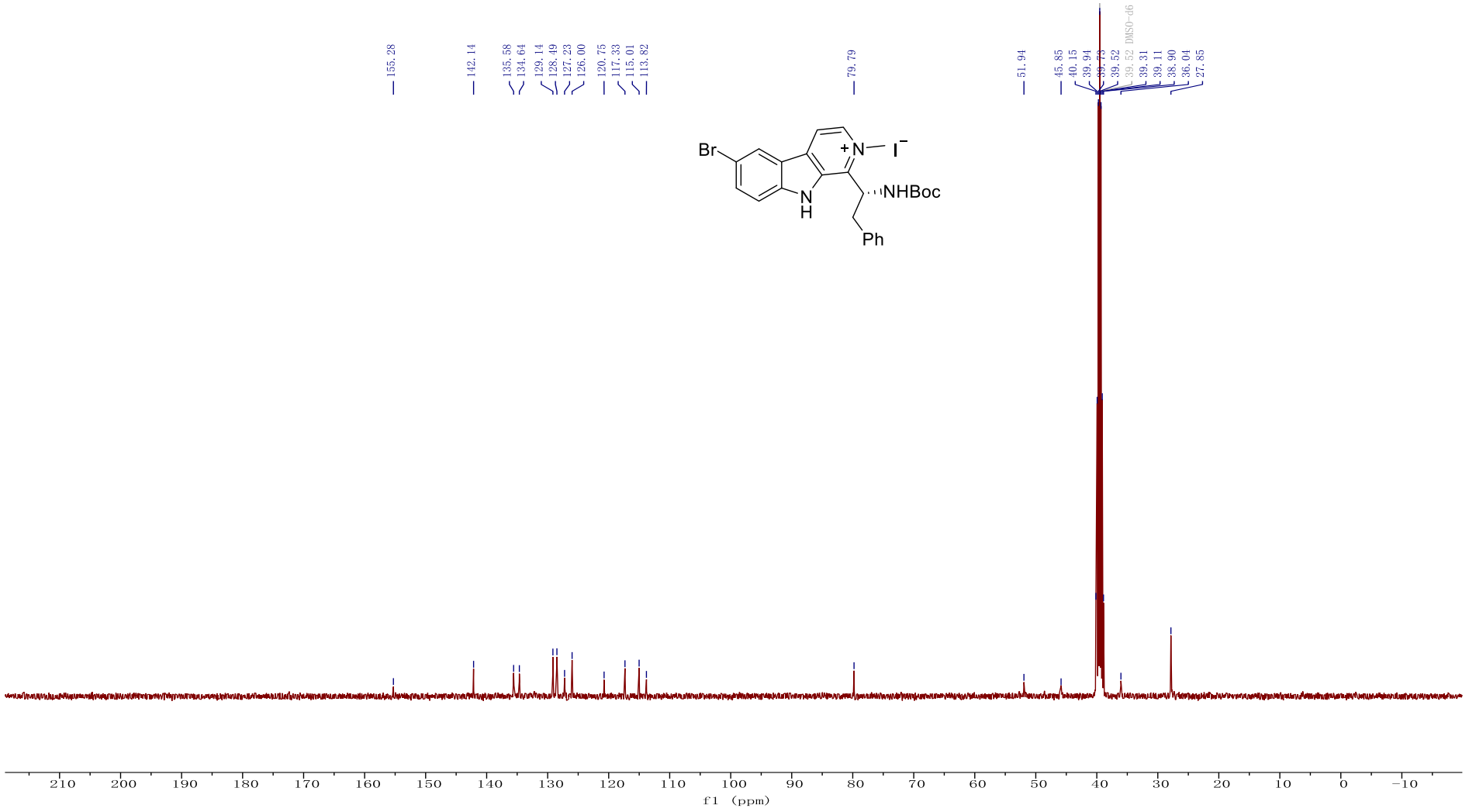
^{13}C NMR (100 MHz, DMSO-d_6) tert-butyl (*R*)-(1-(6-bromo-9*H*-pyrido[3,4-*b*]indol-1-yl)-2-phenylethyl)carbamate (**9**)



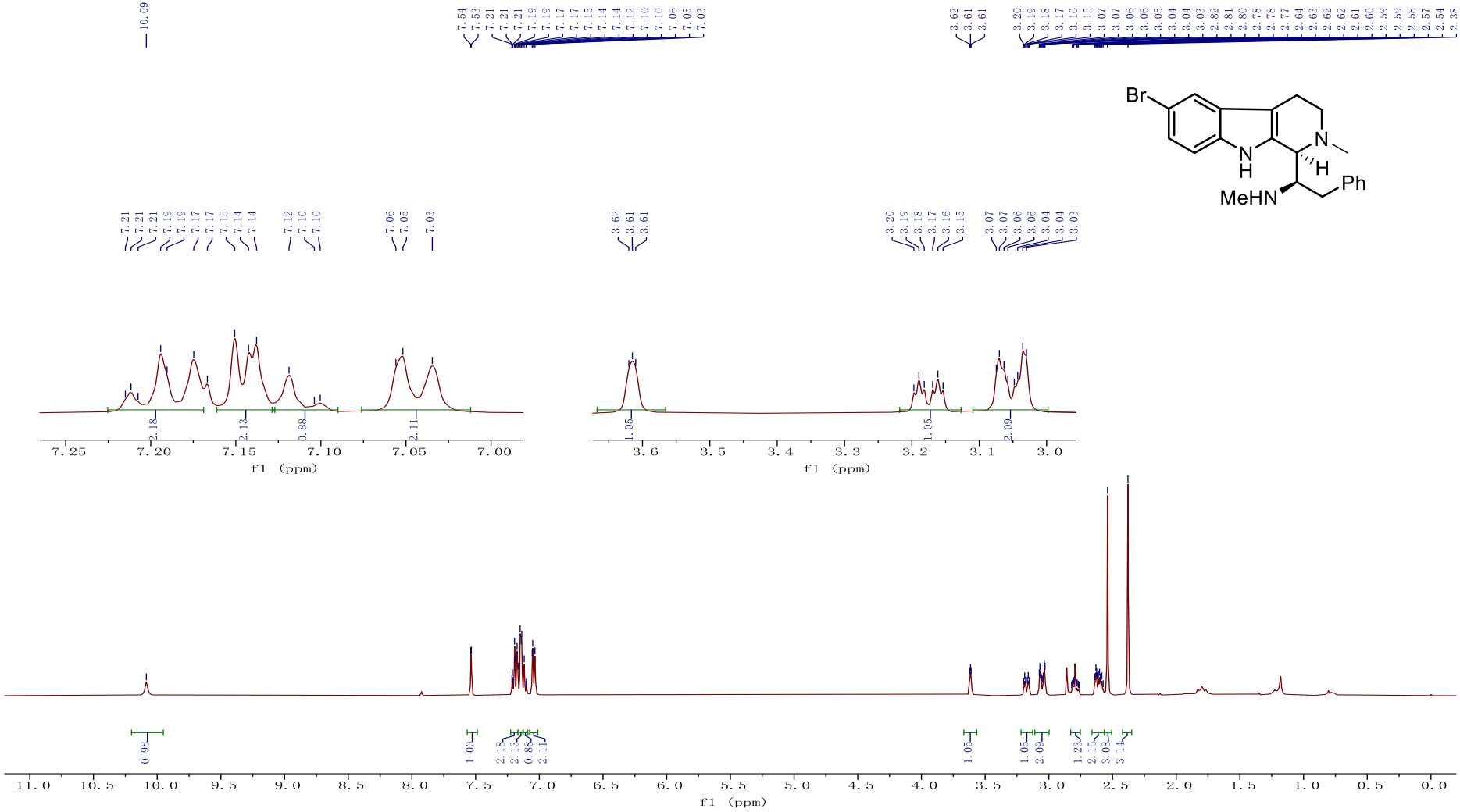
^{1}H NMR (400 MHz, DMSO-d_6) (*R*)-6-bromo-1-(1-((tert-butoxycarbonyl)amino)-2-phenylethyl)-2-methyl-9*H*-pyrido[3,4-*b*]indol-2-ium iodide (**10**)



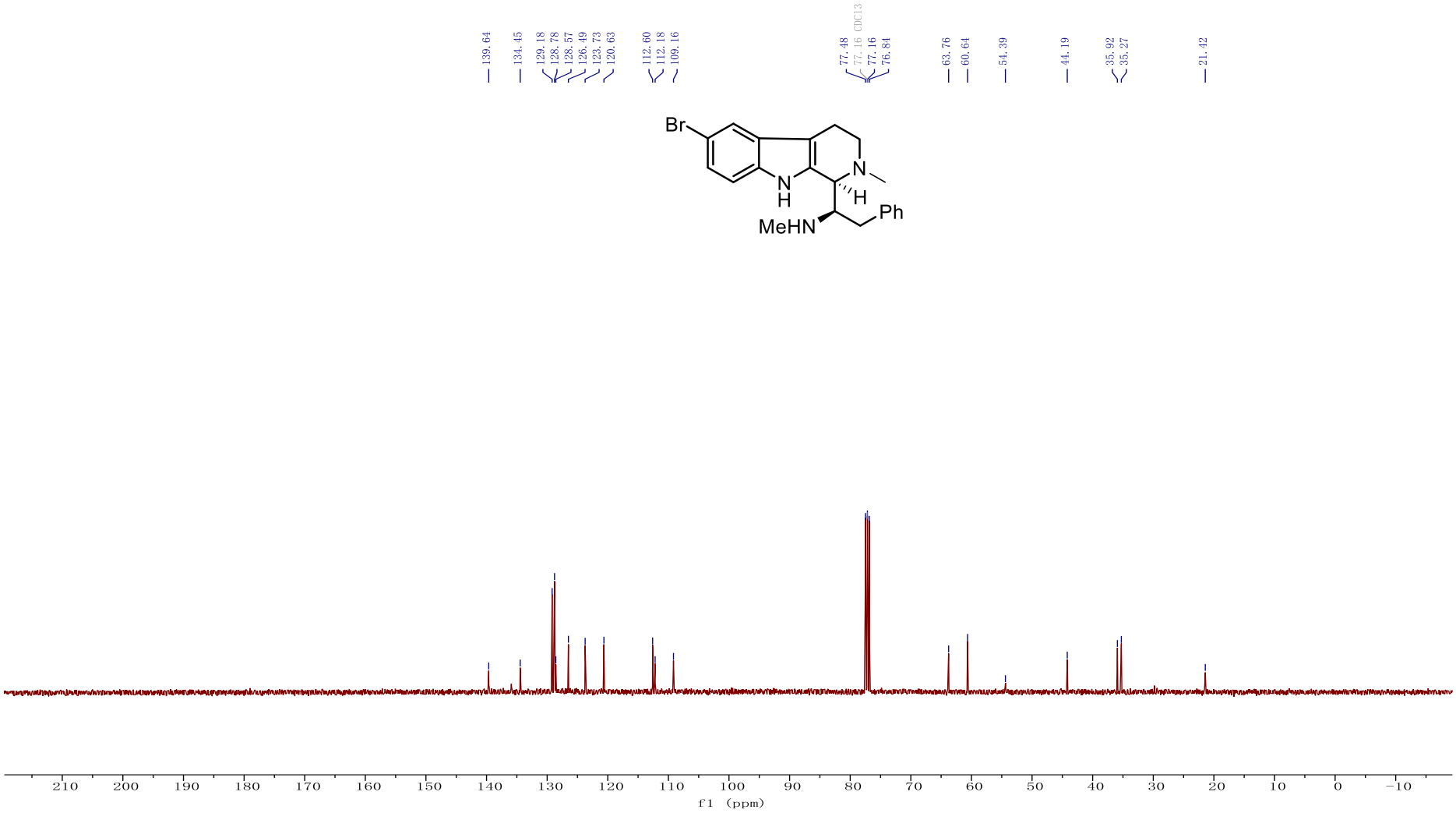
^{13}C NMR (101 MHz, DMSO-d_6) (*R*)-6-bromo-1-(1-((tert-butoxycarbonyl)amino)-2-phenylethyl)-2-methyl-9*H*-pyrido[3,4-*b*]indol-2-ium iodide (**10**)



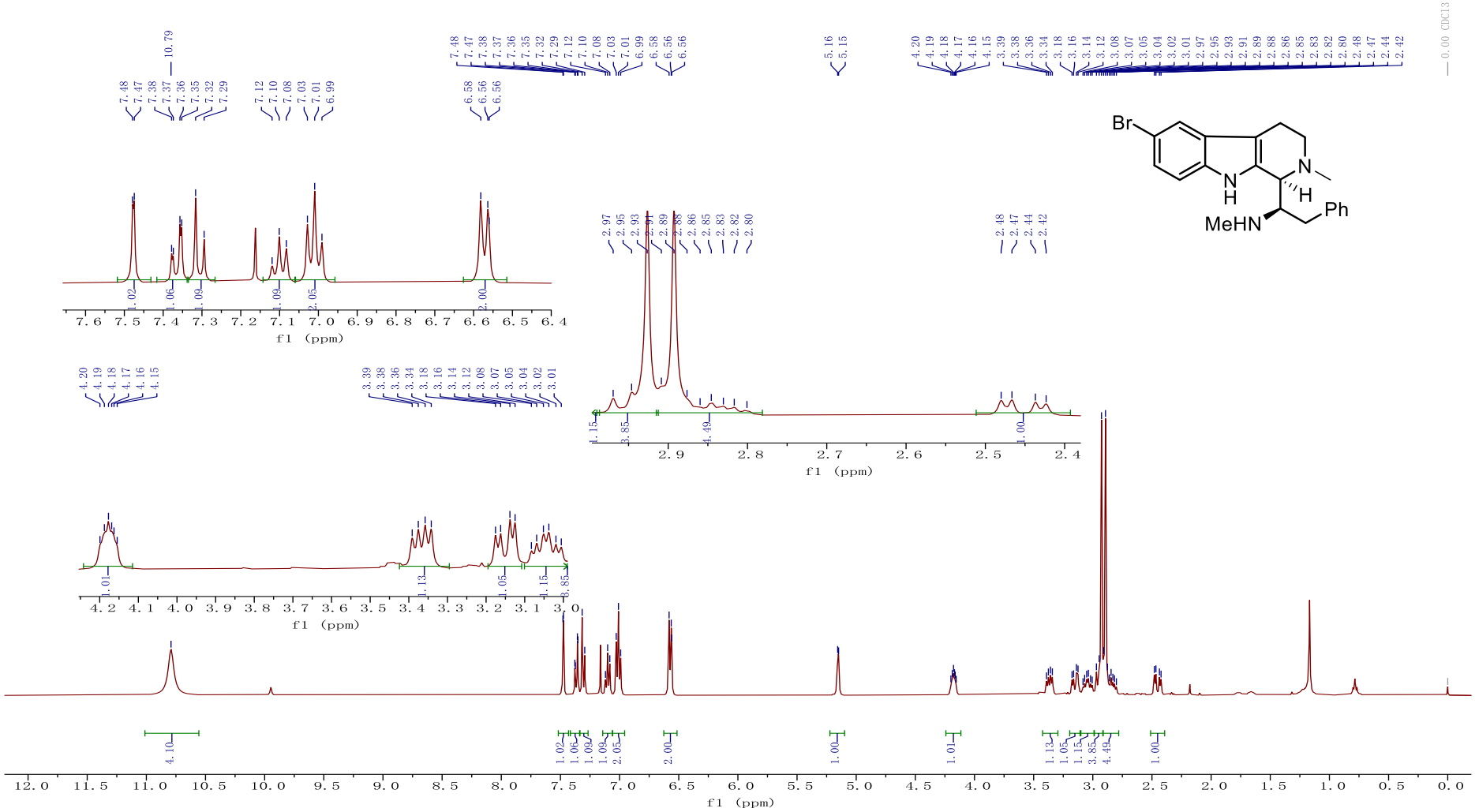
^{1}H NMR (400 MHz, $CDCl_3$) (*R*)-1-((*S*)-6-bromo-2-methyl-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)-N-methyl-2-phenylethan-1-amine (**12**)



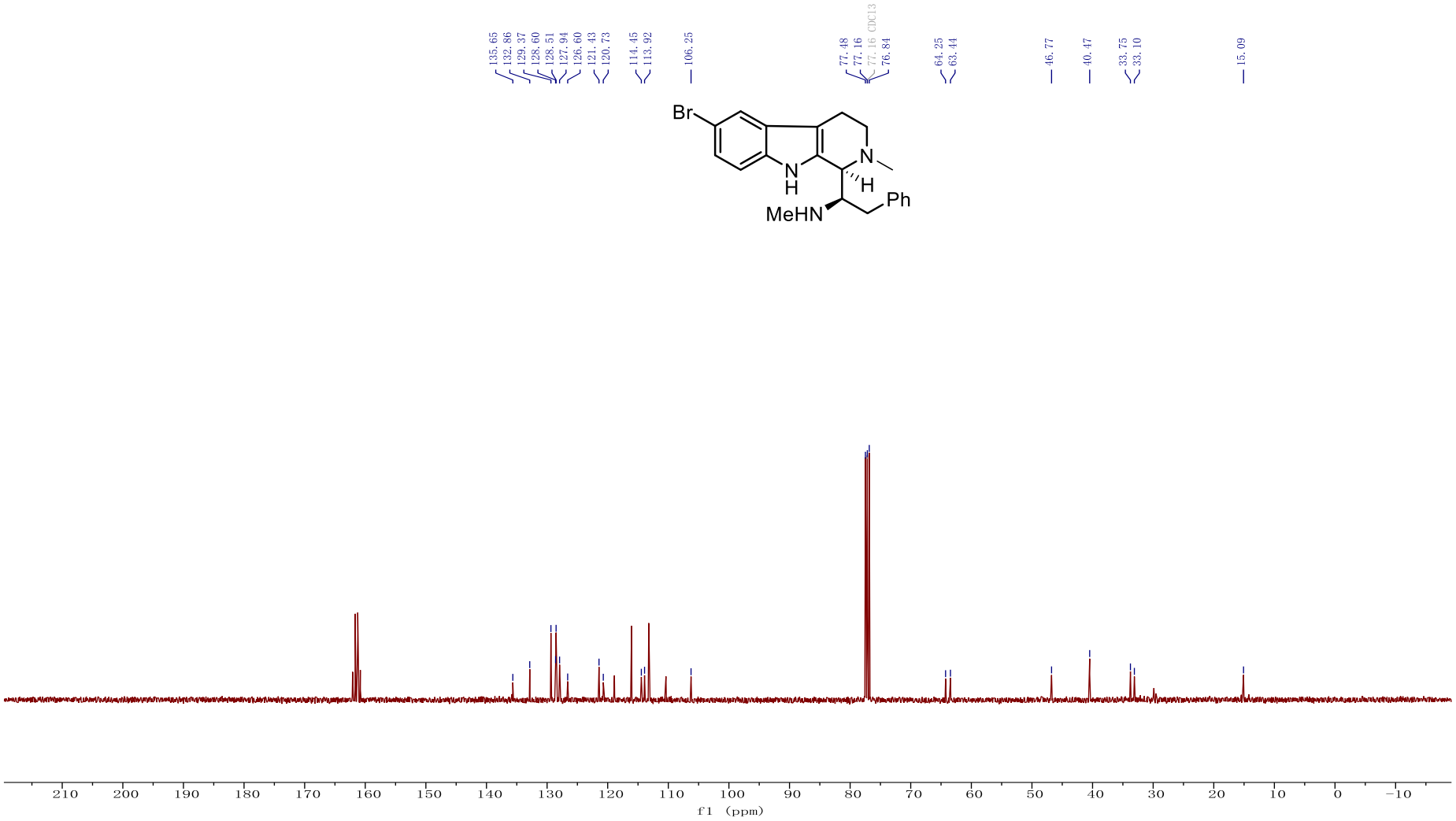
^{13}C NMR (101 MHz, $CDCl_3$) (*R*)-1-((*S*)-6-bromo-2-methyl-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)-N-methyl-2-phenylethan-1-amine (**12**)



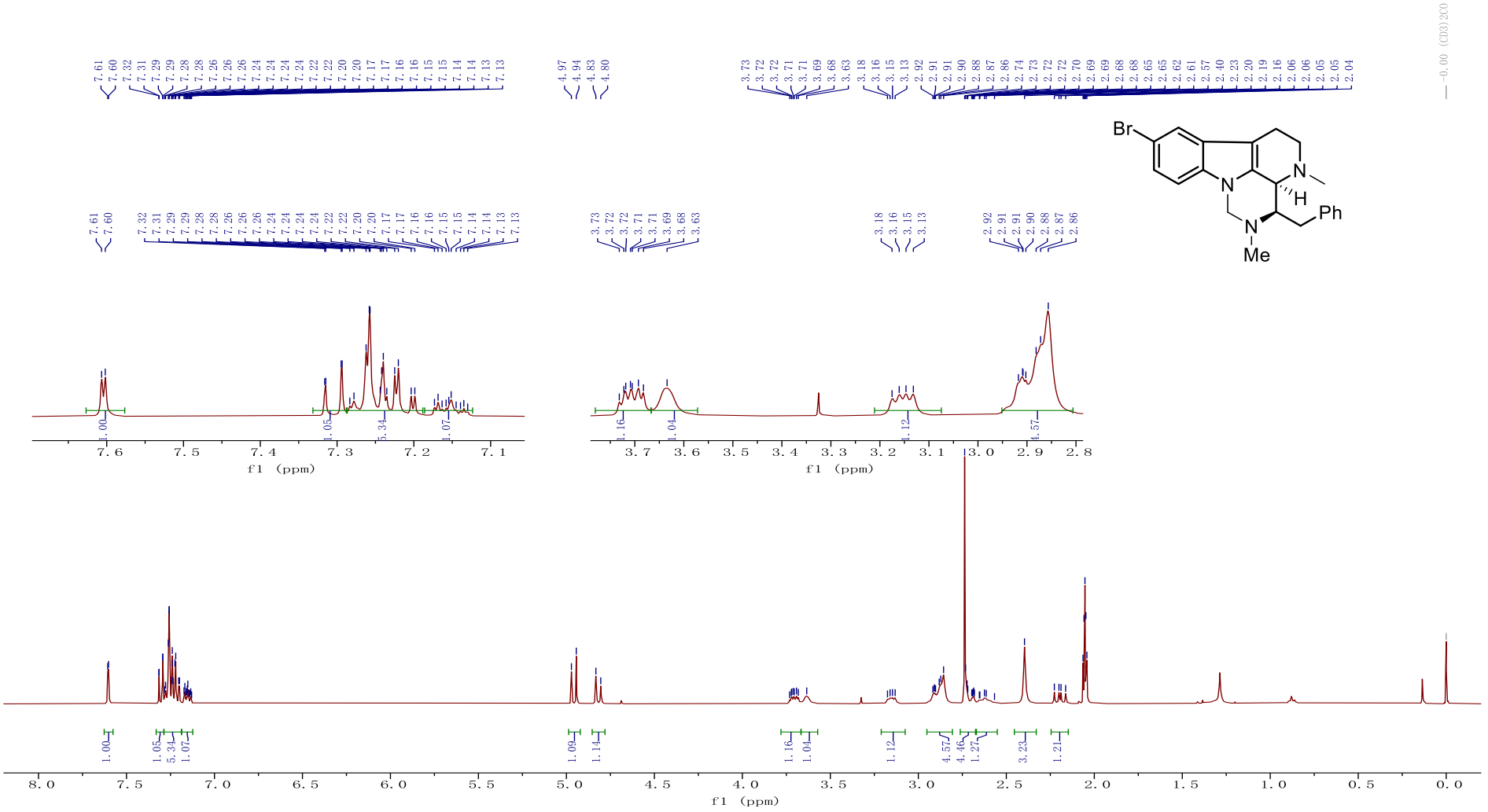
^{1}H NMR (400 MHz, $CDCl_3$: CF_3COOD = 10:1) (*R*)-1-((*S*)-6-bromo-2-methyl-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)-N-methyl-2-phenylethan-1-amine (**12**)



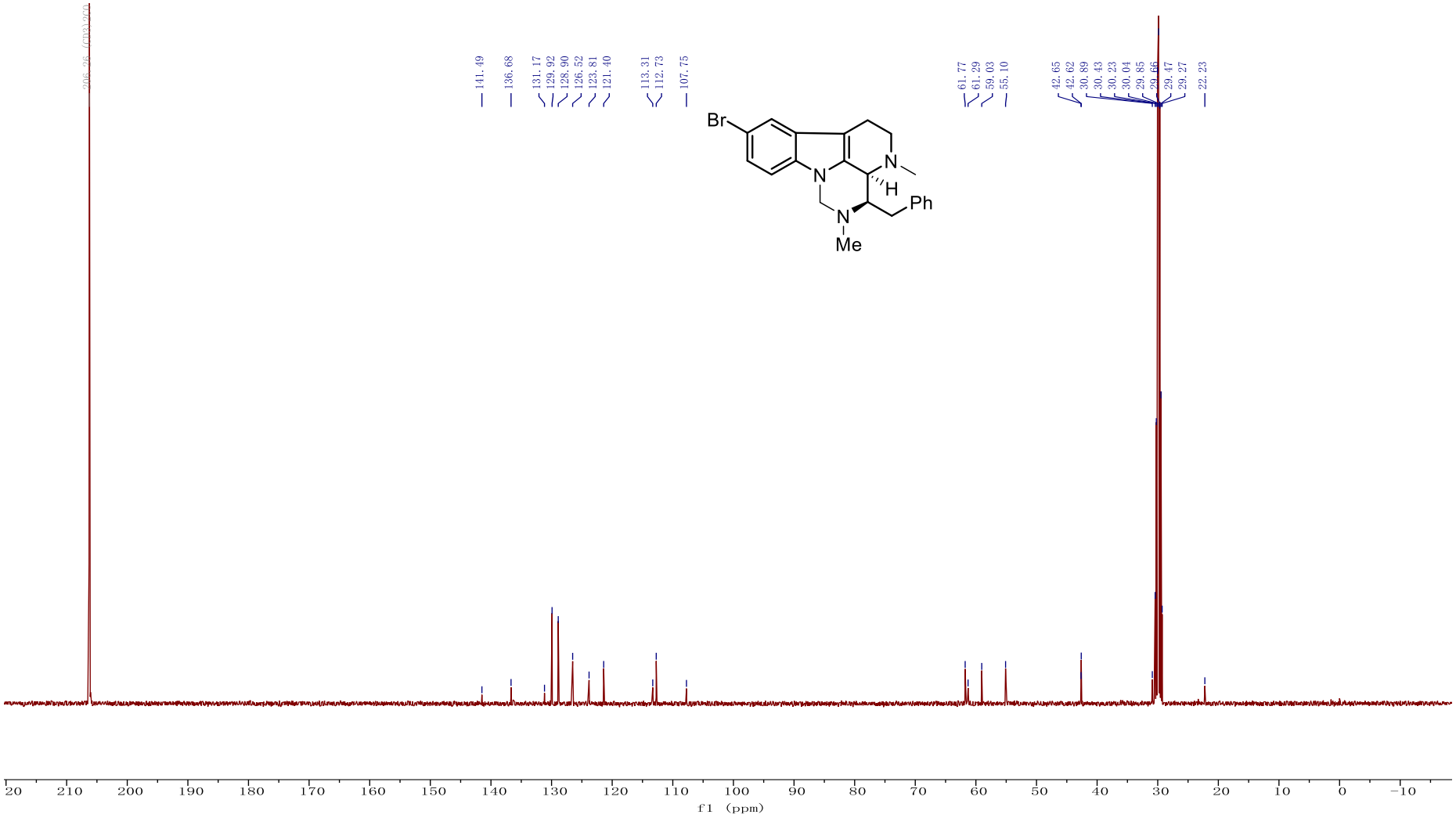
^{13}C NMR (101 MHz, $CDCl_3$: CF_3COOD = 10:1) (*R*)-1-((*S*)-6-bromo-2-methyl-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)-N-methyl-2-phenylethan-1-amine (**12**)



^{1}H NMR (400 MHz, Acetone-d_6) (3*aS*,4*R*)-4-benzyl-9-bromo-3,5-dimethyl-2,3,3*a*,4,5,6-hexahydro-1*H*-3,5,6*a*-triazafluoranthene (**13**)



^{13}C NMR (101 MHz, Acetone-d_6) (3*aS*,4*R*)-4-benzyl-9-bromo-3,5-dimethyl-2,3,3*a*,4,5,6-hexahydro-1*H*-3,5,6*a*-triazafluoranthene (**13**)



NOESY (400 MHz, Acetone-d_6) (3*aS*,4*R*)-4-benzyl-9-bromo-3,5-dimethyl-2,3,3*a*,4,5,6-hexahydro-1*H*-3,5,6*a*-triazafluoranthene (**13**)