

# Supporting Information

**K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>-Mediated Direct C-H Functionalization to Synthesize**

**Quinoxalin-2(1*H*)-one Derivatives in Water**

Hui Qin, Guo-Liang Wei, Xiao-Wei Zheng, Mei-Hua Bao, Yi-Wen Zhang, Ping

Huang

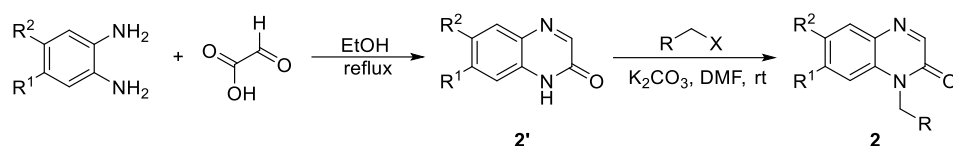
## List of Contents

1. General information	S2
2. General procedure for the synthesis of starting materials	S2
3. General procedure for the synthesis of <b>3a-3aq</b>	S2-3
4. The mechanistic studies	S3-6
5. X-ray Structure and Data of <b>3m</b>	S6-9
5. Characterization of products	S9-20
6. References	S20
7. Copies of NMR Spectra	S21-50

## 1. General information

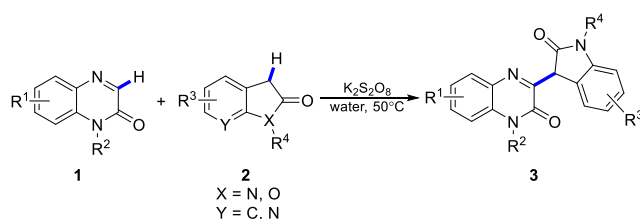
Unless stated otherwise, all reactions for preparing compound **3a-aj** were carried out under an air atmosphere. All reagents and solvents were of commercial quality and were used without further purification. Purification was carried out according to standard laboratory methods<sup>[1]</sup>. All reactions were monitored by TLC analysis with silica gel-coated plates with fluorescent indicator UV254. <sup>1</sup>H and <sup>13</sup>C NMR spectra were obtained on either a Bruker AV 400 at 400 MHz and 100 MHz, respectively. Chemical shifts are reported in ppm and coupling constants are reported in Hz with TMS at 0.0 ppm (<sup>1</sup>H and <sup>13</sup>C) and DMSO-*d*<sub>6</sub> referenced at 2.50 (<sup>1</sup>H) and 39.5 (<sup>13</sup>C). Mass spectra were measured with an Orbitrap Exploris™ 120 mass spectrometer using ESI ionization.

## 2. General procedure for the synthesis of starting materials<sup>[2]</sup>



Glyoxylic acid (1.1 equiv.) was added into a suspension of *o*-arylenediamine (1.0 equiv.) in ethanol (1 mol/L). The reaction mixture was stirred and heated at reflux in an oil bath for 2 h, then at room temperature for 1 h until the reaction completed. The precipitated solid was filtered and washed with ethanol, then dried to give quinoxalinone **2'**. For alkylation, the corresponding alkyl halide (1.6 equiv.) was added to a suspension of quinoxalinone **2'** (1 equiv.) and potassium carbonate (1.2 equiv.) in DMF. The reaction mixture was stirred at room temperature overnight. When TLC analysis indicated that the quinoxalinone disappeared, the reaction mixture was washed with saturated solution of ammonium chloride, ethyl acetate and water. The organic layer was separated and the aqueous layer was extracted twice with ethyl acetate. The combined organic layers were dried with anhydrous MgSO<sub>4</sub>, filtered and concentrated under reduced pressure. The resulting organic residue was purified by column chromatography over silica gel to afford the desired product **2**.

## 3. General procedure for the synthesis of target compounds

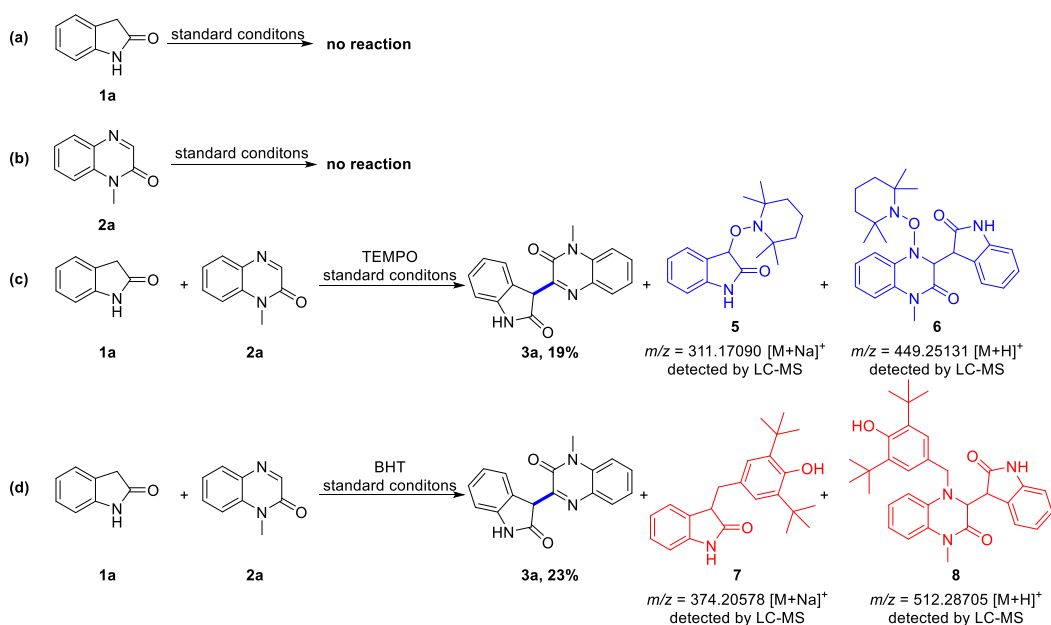


Different substituted quinoxalin-2-one **1** (0.2 mmol, 1.0 equiv.) and various oxindole **2** (0.24 mmol, 1.2 equiv.) was added into 2 ml water (H<sub>2</sub>O) and was treated with K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.2 mmol, 1.0 equiv.) at 50°C under air atmosphere in a 10 mL thick-walled ground test tube. Then mixture was stirred until the reaction completed. The progress of the reaction was monitored by TLC. After that, the reaction mixture was centrifuged with 75% ethanol for three time to obtain precipitate which was further purified through preparative TLC plate to obtain the desired product **3**.



## 4. The mechanistic studies

### 4.1 The control experiments about synthesis of **3a**

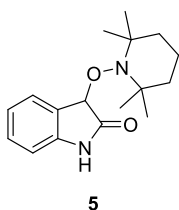


(a) Oxindole (**1a**, 0.2 mmol) and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 2.0 equiv.) were added to a 10 mL thick-walled ground test tube with a magnetic stirring bar, then the reaction solution was stirred at 50°C. No new product was detected via TLC plate.

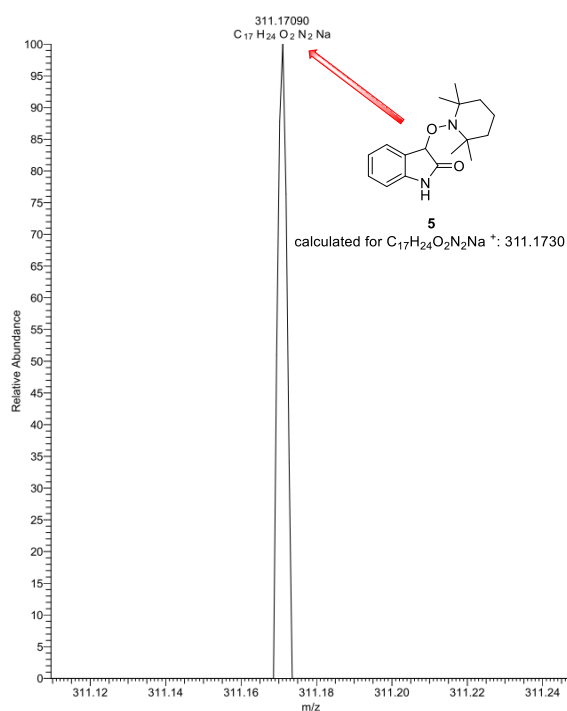
(b) 1-Methylquinoxalin-2-one (**2a**, 0.2 mmol, 1.2 equiv.) and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 2.0 equiv.) were added to a 10 mL thick-walled ground test tube with a magnetic stirring bar, then the reaction solution was stirred at 50°C until the reaction complete. No new product was detected via TLC plate.

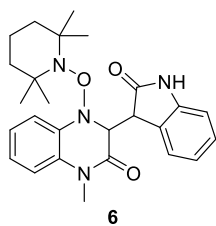
- (c) Oxindole (**1a**, 0.2 mmol), 1-methylquinoxalin-2-one (**2a**, 0.24 mmol, 1.2 equiv.),  $K_2S_2O_8$  (0.4 mmol, 2.0 equiv.) and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 0.4 mmol, 2.0 equiv.) were added to a 10 mL thick-walled ground test tube with a magnetic stirring bar, then the reaction mixture was stirred at rt until the reaction completed. the desired product **3a** was obtained in 19% yield. Furthermore, two free radical-trapping adducts **5** and **6** were detected by HRMS from the reaction solution, indicating that the reaction mechanism might be a radical reaction pathway.
- (d) Oxindole (**1a**, 0.2 mmol), 1-methylquinoxalin-2-one (**2a**, 0.24 mmol, 1.2 equiv.),  $K_2S_2O_8$  (0.4 mmol, 2.0 equiv.) and Butylated hydroxytoluene (BHT, 0.4 mmol, 2.0 equiv.) were added to a 10 mL thick-walled ground test tube with a magnetic stirring bar, then the reaction mixture was stirred at rt until the reaction completed. the desired product **3a** was obtained in 23% yield. Furthermore, two free radical-trapping adducts **7** and **8** were detected by HRMS from the reaction solution, indicating that the reaction mechanism might be a radical reaction pathway.

#### 4.2 The HRMS spectra of compounds 5-8 in first set of control experiments

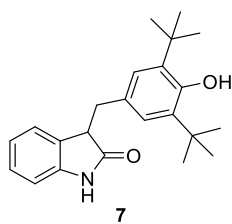
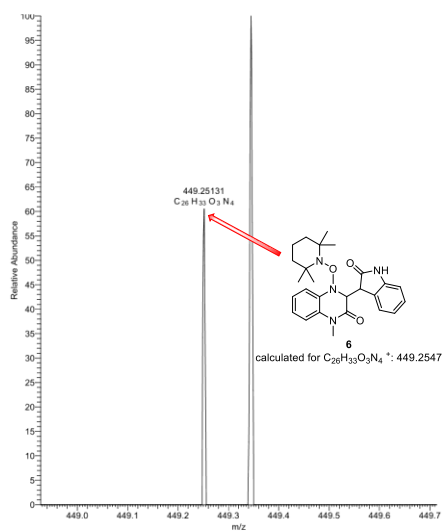


**Compound 6: HRMS (m/z) [ESI]:** calculated for  $C_{17}H_{24}O_2N_2Na^+$   $[M+Na]^+$ : 311.1730, found 311.17090.

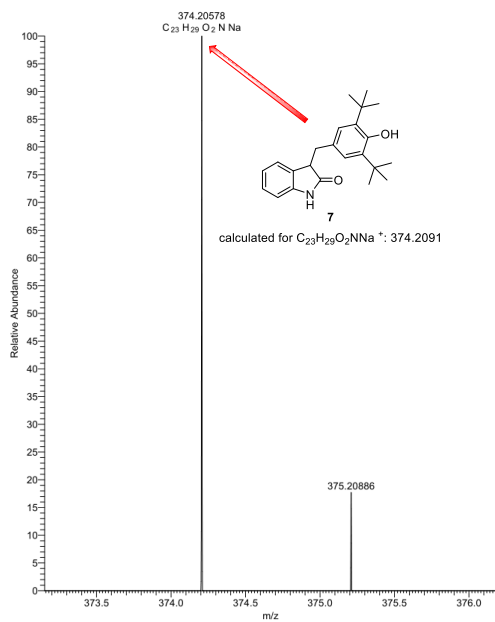


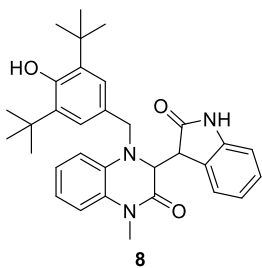


**Compound 6: HRMS (m/z) [ESI]:** calculated for  $C_{26}H_{33}O_3N_4^+$   $[M+H]^+$ : 449.2547, found 449.25131.

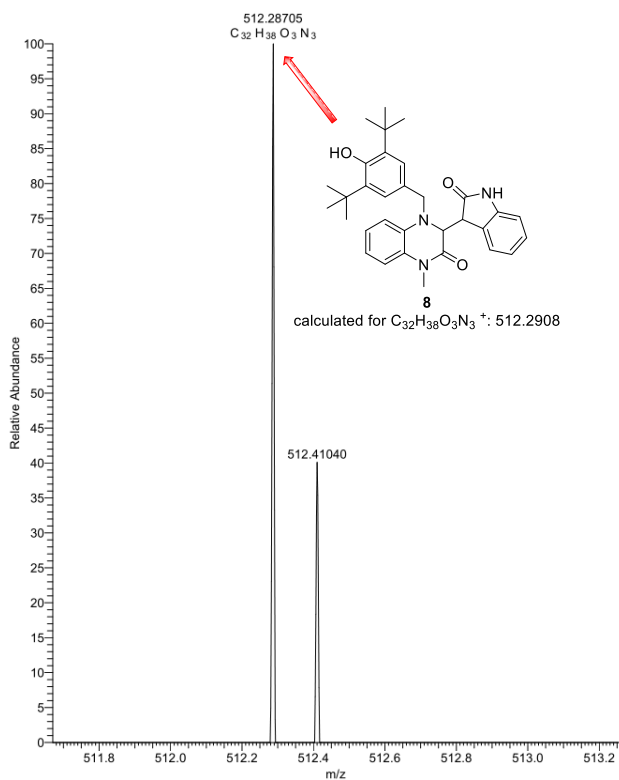


**Compound 7: HRMS (m/z) [ESI]:** calculated for  $C_{23}H_{29}O_2NNa^+$   $[M+Na]^+$ : 374.2091, found 374.20578.

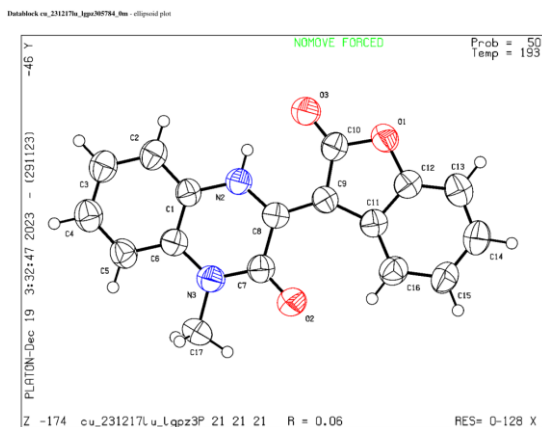




**Compound 8: HRMS (m/z) [ESI]:** calculated for  $C_{32}H_{38}O_3N_3^+$   $[M+H]^+$ : 512.2908, found 512.28750.



## 5. X-ray Structure and Data of 3m



## cu\_231217LU\_LGPZ305784\_0m

**Table 1 Crystal data and structure refinement for cu\_231217LU\_LGPZ305784\_0m.**

Identification code	cu_231217LU_LGPZ305784_0m
Empirical formula	C <sub>17</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub>
Formula weight	292.29
Temperature/K	193.00
Crystal system	orthorhombic
Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a/Å	4.7263(17)
b/Å	15.850(6)
c/Å	17.689(7)
α/°	90
β/°	90
γ/°	90
Volume/Å <sup>3</sup>	1325.1(9)
Z	4
ρ <sub>calc</sub> /cm <sup>3</sup>	1.465
μ/mm <sup>-1</sup>	0.843
F(000)	608.0
Crystal size/mm <sup>3</sup>	0.13 × 0.11 × 0.09
Radiation	CuKα (λ = 1.54178)
2θ range for data collection/°	7.488 to 158.524
Index ranges	-4 ≤ h ≤ 5, -16 ≤ k ≤ 18, -20 ≤ l ≤ 21
Reflections collected	12691
Independent reflections	2499 [R <sub>int</sub> = 0.0972, R <sub>sigma</sub> = 0.0727]
Data/restraints/parameters	2499/0/201
Goodness-of-fit on F <sup>2</sup>	1.038
Final R indexes [I >= 2σ (I)]	R <sub>1</sub> = 0.0619, wR <sub>2</sub> = 0.1488
Final R indexes [all data]	R <sub>1</sub> = 0.1231, wR <sub>2</sub> = 0.2033
Largest diff. peak/hole / e Å <sup>-3</sup>	0.28/-0.23
Flack parameter	-0.1(4)

**Table 2 Fractional Atomic Coordinates (×10<sup>4</sup>) and Equivalent Isotropic Displacement Parameters (Å<sup>2</sup>×10<sup>3</sup>) for cu\_231217LU\_LGPZ305784\_0m. U<sub>eq</sub> is defined as 1/3 of the trace of the orthogonalised U<sub>ij</sub> tensor.**

Atom	x	y	z	U(eq)
O(3)	4109 (9)	5521 (3)	7730 (2)	65.1 (12)
O(2)	3510 (9)	3557 (3)	5414 (2)	69.6 (14)
O(1)	798 (9)	4568 (3)	8037 (2)	63.7 (12)
N(2)	6304 (10)	5329 (3)	6382 (3)	54.0 (13)
N(3)	7077 (11)	4444 (4)	5078 (3)	59.6 (15)
C(1)	8224 (11)	5637 (4)	5856 (3)	50.2 (15)
C(2)	9720 (12)	6370 (4)	5995 (4)	59.2 (17)
C(3)	11653 (13)	6663 (5)	5469 (4)	66.3 (19)
C(4)	12097 (15)	6213 (5)	4812 (4)	67.5 (19)
C(5)	10616 (13)	5473 (5)	4671 (3)	60.9 (17)
C(6)	8649 (13)	5178 (4)	5192 (3)	53.1 (16)
C(7)	4998 (12)	4167 (4)	5565 (3)	56.4 (16)
C(8)	4657 (12)	4643 (4)	6285 (3)	49.9 (15)
C(9)	2795 (12)	4398 (4)	6856 (3)	50.5 (15)
C(10)	2738 (13)	4899 (4)	7542 (3)	55.1 (16)
C(11)	762 (12)	3720 (4)	6974 (3)	53.4 (15)
C(12)	-374 (13)	3860 (4)	7692 (4)	55.7 (16)
C(13)	-2390 (14)	3365 (5)	8027 (4)	66.0 (19)
C(14)	-3362 (14)	2681 (5)	7626 (4)	70 (2)
C(15)	-2338 (14)	2517 (4)	6911 (4)	68.2 (19)
C(16)	-323 (14)	3038 (4)	6577 (4)	63.1 (18)
C(17)	7530 (16)	3927 (4)	4396 (3)	75 (2)



**Table 3 Anisotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for cu\_231217LU\_LGPZ305784\_0m. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^2U_{11}+2hka^2b^2U_{12}+\dots]$ .**

Atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>23</sub>	U <sub>13</sub>	U <sub>12</sub>
O(3)	63 (3)	69 (3)	63 (2)	-7 (2)	2 (2)	-2 (3)
O(2)	65 (3)	72 (4)	72 (3)	-12 (3)	4 (3)	-11 (3)
O(1)	57 (2)	75 (3)	58 (2)	-6 (2)	8 (2)	-4 (3)
N(2)	46 (3)	60 (4)	55 (3)	-6 (3)	1 (2)	-3 (3)
N(3)	57 (3)	63 (4)	58 (3)	-8 (3)	2 (2)	-1 (3)
C(1)	38 (3)	58 (4)	55 (3)	2 (3)	-1 (2)	2 (3)
C(2)	45 (3)	62 (5)	71 (4)	3 (3)	-4 (3)	-1 (3)
C(3)	52 (4)	70 (5)	77 (4)	3 (4)	-1 (4)	-3 (3)
C(4)	56 (4)	79 (6)	68 (4)	7 (4)	1 (3)	0 (4)
C(5)	51 (3)	74 (5)	58 (3)	1 (4)	3 (3)	0 (4)
C(6)	46 (3)	59 (4)	55 (3)	-2 (3)	-4 (3)	3 (3)
C(7)	45 (3)	62 (5)	63 (4)	-3 (3)	-1 (3)	1 (3)
C(8)	45 (3)	53 (4)	51 (3)	-2 (3)	-4 (3)	5 (3)
C(9)	46 (3)	53 (4)	53 (3)	1 (3)	1 (3)	5 (3)
C(10)	49 (3)	57 (5)	60 (3)	4 (3)	-5 (3)	-1 (3)
C(11)	42 (3)	59 (4)	59 (3)	0 (3)	-6 (3)	4 (3)
C(12)	49 (3)	55 (4)	63 (3)	0 (3)	-4 (3)	4 (3)
C(13)	52 (4)	82 (6)	64 (4)	10 (4)	0 (3)	7 (4)
C(14)	54 (4)	76 (6)	82 (5)	11 (4)	1 (4)	-7 (4)
C(15)	58 (4)	64 (5)	83 (4)	1 (4)	1 (3)	-12 (4)
C(16)	56 (4)	64 (5)	69 (4)	-3 (4)	-3 (3)	-4 (4)
C(17)	81 (5)	84 (6)	58 (4)	-17 (4)	15 (4)	-8 (4)

**Table 4 Bond Lengths for cu\_231217LU\_LGPZ305784\_0m.**

Atom	Atom	Length/\AA	Atom	Atom	Length/\AA
O(3)	C(10)	1.226 (7)	C(4)	C(5)	1.389 (9)
O(2)	C(7)	1.225 (7)	C(5)	C(6)	1.390 (8)
O(1)	C(10)	1.373 (7)	C(7)	C(8)	1.489 (8)
O(1)	C(12)	1.393 (7)	C(8)	C(9)	1.395 (8)
N(2)	C(1)	1.387 (7)	C(9)	C(10)	1.450 (9)
N(2)	C(8)	1.348 (7)	C(9)	C(11)	1.457 (8)
N(3)	C(6)	1.395 (8)	C(11)	C(12)	1.397 (8)
N(3)	C(7)	1.379 (7)	C(11)	C(16)	1.387 (8)
N(3)	C(17)	1.474 (8)	C(12)	C(13)	1.369 (9)
C(1)	C(2)	1.382 (8)	C(13)	C(14)	1.374 (10)
C(1)	C(6)	1.397 (8)	C(14)	C(15)	1.380 (9)
C(2)	C(3)	1.384 (8)	C(15)	C(16)	1.392 (9)
C(3)	C(4)	1.380 (9)			

**Table 5 Bond Angles for cu\_231217LU\_LGPZ305784\_0m.**

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C(10)	O(1)	C(12)	107.1 (5)	N(2)	C(8)	C(9)	119.8 (5)
C(8)	N(2)	C(1)	125.1 (5)	C(9)	C(8)	C(7)	123.1 (5)
C(6)	N(3)	C(17)	120.3 (5)	C(8)	C(9)	C(10)	117.7 (5)
C(7)	N(3)	C(6)	123.7 (5)	C(8)	C(9)	C(11)	136.5 (6)
C(7)	N(3)	C(17)	116.0 (5)	C(10)	C(9)	C(11)	105.8 (5)
N(2)	C(1)	C(6)	118.4 (6)	O(3)	C(10)	O(1)	119.2 (6)
C(2)	C(1)	N(2)	120.8 (6)	O(3)	C(10)	C(9)	131.1 (6)
C(2)	C(1)	C(6)	120.9 (6)	O(1)	C(10)	C(9)	109.7 (5)
C(1)	C(2)	C(3)	120.1 (6)	C(12)	C(11)	C(9)	105.4 (5)
C(4)	C(3)	C(2)	119.5 (7)	C(16)	C(11)	C(9)	138.3 (6)
C(3)	C(4)	C(5)	120.8 (6)	C(16)	C(11)	C(12)	116.2 (6)
C(4)	C(5)	C(6)	120.1 (6)	O(1)	C(12)	C(11)	112.0 (5)
N(3)	C(6)	C(1)	118.6 (5)	C(13)	C(12)	O(1)	123.3 (6)
C(5)	C(6)	N(3)	122.8 (6)	C(13)	C(12)	C(11)	124.8 (6)
C(5)	C(6)	C(1)	118.6 (6)	C(12)	C(13)	C(14)	117.4 (6)
O(2)	C(7)	N(3)	121.6 (6)	C(13)	C(14)	C(15)	120.3 (7)
O(2)	C(7)	C(8)	121.6 (5)	C(14)	C(15)	C(16)	121.2 (7)
N(3)	C(7)	C(8)	116.8 (6)	C(11)	C(16)	C(15)	120.0 (6)
N(2)	C(8)	C(7)	117.1 (5)				

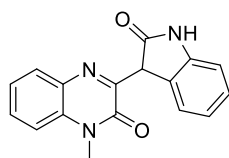
**Table 6 Torsion Angles for cu\_231217LU\_LGPZ305784\_0m.**

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O(2)C(7)	C(8)	N(2)		-176.9(5)	C(8)	N(2)	C(1)	C(6)	-4.4(8)
O(2)C(7)	C(8)	C(9)		4.8(9)	C(8)	C(9)	C(10)O(3)		-1.2(10)
O(1)C(12)C(13)C(14)				-179.7(6)	C(8)	C(9)	C(10)O(1)		-180.0(5)
N(2)C(1)	C(2)	C(3)		179.2(5)	C(8)	C(9)	C(11)C(12)		179.6(6)
N(2)C(1)	C(6)	N(3)		1.6(8)	C(8)	C(9)	C(11)C(16)		-2.9(13)
N(2)C(1)	C(6)	C(5)		-178.6(5)	C(9)	C(11)C(12)O(1)			-0.5(7)
N(2)C(8)	C(9)	C(10)		-0.5(8)	C(9)	C(11)C(12)C(13)			179.8(6)
N(2)C(8)	C(9)	C(11)		-179.2(6)	C(9)	C(11)C(16)C(15)			180.0(7)
N(3)C(7)	C(8)	N(2)		3.3(8)	C(10)O(1)	C(12)C(11)			-0.1(7)
N(3)C(7)	C(8)	C(9)		-175.0(5)	C(10)O(1)	C(12)C(13)			179.6(6)
C(1)N(2)	C(8)	C(7)		1.9(8)	C(10)C(9)	C(11)C(12)			0.8(6)
C(1)N(2)	C(8)	C(9)		-179.8(5)	C(10)C(9)	C(11)C(16)			178.4(7)
C(1)C(2)	C(3)	C(4)		-0.7(9)	C(11)C(9)	C(10)O(3)			177.8(6)
C(2)C(1)	C(6)	N(3)		-179.7(5)	C(11)C(9)	C(10)O(1)			-1.0(7)
C(2)C(1)	C(6)	C(5)		0.1(9)	C(11)C(12)C(13)C(14)				0.0(10)
C(2)C(3)	C(4)	C(5)		0.3(10)	C(12)O(1)	C(10)O(3)			-178.3(5)
C(3)C(4)	C(5)	C(6)		0.3(9)	C(12)O(1)	C(10)C(9)			0.7(7)
C(4)C(5)	C(6)	N(3)		179.3(6)	C(12)C(11)C(16)C(15)				-2.7(9)
C(4)C(5)	C(6)	C(1)		-0.5(9)	C(12)C(13)C(14)C(15)				-0.6(10)
C(6)N(3)	C(7)	O(2)		174.0(6)	C(13)C(14)C(15)C(16)				-0.4(10)
C(6)N(3)	C(7)	C(8)		-6.2(8)	C(14)C(15)C(16)C(11)				2.2(10)
C(6)C(1)	C(2)	C(3)		0.5(9)	C(16)C(11)C(12)O(1)				-178.6(5)
C(7)N(3)	C(6)	C(1)		3.8(9)	C(16)C(11)C(12)C(13)				1.7(9)
C(7)N(3)	C(6)	C(5)		-176.0(6)	C(17)N(3)	C(6)	C(1)		-177.9(6)
C(7)C(8)	C(9)	C(10)		177.7(5)	C(17)N(3)	C(6)	C(5)		2.3(9)
C(7)C(8)	C(9)	C(11)		-0.9(10)	C(17)N(3)	C(7)	O(2)		-4.3(9)
C(8)N(2)	C(1)	C(2)		176.8(5)	C(17)N(3)	C(7)	C(8)		175.5(5)

**Table 7 Hydrogen Atom Coordinates ( $\text{\AA} \times 10^4$ ) and Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for cu\_231217LU\_LGPZ305784\_0m.**

Atom	x	y	z	U(eq)
H(2)	6148.77	5603.91	6812.34	65
H(2A)	9419.99	6673.37	6450.79	71
H(3)	12667.65	7170.63	5559.89	80
H(4)	13430.96	6411.75	4451.87	81
H(5)	10948.45	5167.37	4217.22	73
H(13)	-3088.94	3489.68	8518.34	79
H(14)	-4746.92	2320.24	7842.88	85
H(15)	-3019.27	2039.79	6641.88	82
H(16)	309.58	2926.05	6077.03	76
H(17A)	6958.28	4247.13	3947.77	112
H(17B)	6396.42	3410.37	4431.28	112
H(17C)	9537.44	3778.06	4356.35	112

## 6. Characterization of products

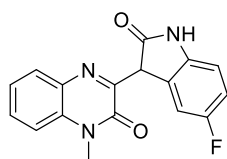


**3-(2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3a)**<sup>[3]</sup> orange solid, mp: 266-268 °C, 56.6 mg, 91%;

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  = 14.50 (s, 1H), 10.94 (s, 1H), 8.70 (dd, *J* = 1.16, 8.00 Hz, 1H), 7.35-7.37 (m, 1H), 7.26-7.29 (m, 1H), 7.16-7.20 (m, 2H), 7.05 (dt, *J* = 1.26, 7.50 Hz, 1H), 6.88-6.95 (m, 2H), 3.58 (s, 3H);

<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  = 172.20, 156.43, 140.23, 137.49, 128.75, 125.91, 125.63, 125.17, 124.54, 124.04, 123.01, 121.01, 116.47, 115.28, 109.52, 97.82, 30.03.

HRMS (ESI-TOF) calcd for C<sub>17</sub>H<sub>14</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 292.1081; found: 292.1076.

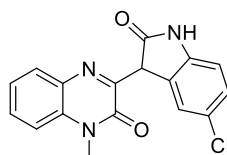


**3-(5-Fluoro-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3b)** orange solid, mp: 254-256 °C, 58.1 mg, 88% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.64 (s, 1H), 10.97 (s, 1H), 8.51 (dd,  $J$  = 2.18, 12.02 Hz, 1H), 7.40-7.42 (m, 1H), 7.32-7.34 (m, 1H), 7.20-7.23 (m, 2H), 6.83-6.86 (m, 2H), 3.60 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.25, 156.79-159.08 (d,  $J_{C-F}$  = 171.70 Hz), 156.33, 141.02, 133.63, 128.86, 125.34, 124.62, 124.47, 116.80, 115.34, 112.12-112.40 (d,  $J_{C-F}$  = 21.02 Hz), 111.01-111.25 (d,  $J_{C-F}$  = 18.12 Hz), 109.59-109.68 (d,  $J_{C-F}$  = 6.76 Hz), 97.32-97.35 (d,  $J_{C-F}$  = 2.22 Hz), 30.11.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>13</sub>FN<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 310.0986; found: 310.0982.

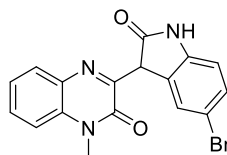


**3-(5-Chloro-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3c)** orange solid, mp: 276-278 °C, 51.4 mg, 74% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.63 (s, 1H), 11.08 (s, 1H), 8.75 (d,  $J$  = 2.15 Hz, 1H), 7.41-7.43 (m, 1H), 7.34-7.36 (m, 1H), 7.21-7.24 (m, 2H), 7.06 (dd,  $J$  = 2.27, 8.22 Hz, 1H), 6.88 (d,  $J$  = 8.25 Hz, 1H), 3.62 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.02, 156.32, 141.25, 135.91, 128.92, 125.27, 125.21, 125.04, 124.69, 124.64, 124.28, 116.90, 115.41, 110.59, 96.57, 30.19.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>13</sub>ClN<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 326.0691; found: 326.0694.

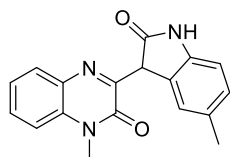


**3-(5-Bromo-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3d)** orange solid, mp: 267-269 °C, 56.4 mg, 71% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.62 (s, 1H), 11.08 (s, 1H), 8.88 (d,  $J$  = 2.04 Hz, 1H), 7.40-7.43 (m, 1H), 7.32-7.35 (m, 1H), 7.21-7.24 (m, 2H), 7.16-7.19 (m, 1H), 6.84 (d,  $J$  = 8.16 Hz, 1H), 3.58 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 176.38, 156.37, 141.33, 136.27, 130.53, 128.96, 127.81, 127.68, 127.08, 125.25, 124.71, 116.94, 115.46, 113.26, 111.18, 96.44, 30.23.

**HRMS (ESI-TOF)** calcd for  $C_{17}H_{13}BrN_3O_2$   $[M + H]^+$ : 370.0186; found: 370.0183.

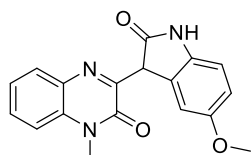


**3-(5-Methyl-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3e)** orange solid, mp: 271-273 °C, 45.5 mg, 70% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.54 (s, 1H), 10.85 (s, 1H), 8.55 (d,  $J$  = 1.76 Hz, 1H), 7.37-7.40 (m, 1H), 7.27-7.30 (m, 1H), 7.18-7.20 (m, 2H), 6.88 (dd,  $J$  = 1.76, 7.88 Hz, 1H), 6.78 (d,  $J$  = 7.80 Hz, 1H), 3.60 (s, 3H), 2.30 (s, 3H);

**$^{13}C$  NMR (100 MHz, DMSO- $d_6$ ):**  $\delta$  = 176.38, 156.37, 141.33, 136.27, 130.53, 128.96, 127.81, 127.68, 127.08, 125.25, 124.71, 116.94, 115.46, 113.26, 111.18, 96.44, 30.23.

**HRMS (ESI-TOF)** calcd for  $C_{18}H_{16}N_3O_2$   $[M + H]^+$ : 306.1237; found: 306.1233.

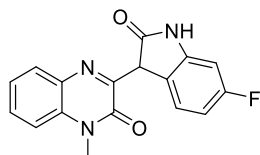


**3-(5-Methoxy-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3f)** orange solid, mp: 268-270 °C, 26.4 mg, 38% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.64 (s, 1H), 10.78 (s, 1H), 8.44 (d,  $J$  = 2.60 Hz, 1H), 7.39-7.41 (m, 1H), 7.30-7.32 (m, 1H), 7.19-7.21 (m, 2H), 6.72 (d,  $J$  = 2.45 Hz, 1H), 6.67-6.69 (m, 1H), 3.74 (s, 3H), 3.61 (s, 3H);

**$^{13}C$  NMR (100 MHz, DMSO- $d_6$ ):**  $\delta$  = 176.59, 156.54, 154.56, 140.50, 137.47, 131.65, 128.81, 127.54, 124.62, 124.12, 123.96, 115.37, 112.61, 111.91, 109.77, 98.18, 55.38, 30.14.

**HRMS (ESI-TOF)** calcd for  $C_{18}H_{16}N_3O_3$   $[M + H]^+$ : 322.1186; found: 322.1183.



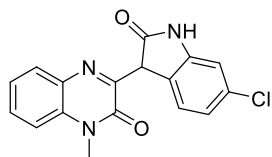
**3-(6-Fluoro-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3g)** orange solid, mp: 252-254 °C, 35.9 mg, 54% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.27 (s, 1H), 11.04 (s, 1H), 8.63-8.68 (m, 1H), 7.31-7.35 (m, 1H), 7.22-7.26 (m, 1H), 7.15-7.17 (m, 2H), 6.67-6.72 (m, 1H), 6.62-6.65 (m, 1H), 3.55 (s, 3H);

**$^{13}C$  NMR (100 MHz, DMSO- $d_6$ ):**  $\delta$  = 172.08, 159.39-161.78 (d,  $J_{C-F}$  = 179.07 Hz), 156.39, 139.83, 138.52-138.64 (d,  $J_{C-F}$  = 8.82 Hz), 128.63, 127.04, 127.12, 125.52, 124.53, 124.05, 119.39-119.42 (d,  $J_{C-F}$  = 1.80 Hz), 116.44, 115.24, 107.10-107.31 (d,  $J_{C-F}$  = 15.81 Hz), 97.09-97.15 (d,  $J_{C-F}$  = 4.65

Hz), 29.99.

**HRMS (ESI-TOF)** calcd for  $C_{17}H_{13}FN_3O_2$   $[M + H]^+$ : 310.0986; found: 310.0984.

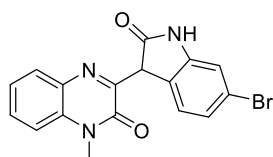


**3-(6-Chloro-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3h)** orange solid, mp: 251-253 °C, 61.3 mg, 89% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.44 (s, 1H), 11.06 (s, 1H), 8.65 (d,  $J$  = 8.70 Hz, 1H), 7.37-7.39 (m, 1H), 7.30-7.31 (m, 1H), 7.19-7.21 (m, 2H), 6.94 (dd,  $J$  = 2.20, 8.55 Hz, 1H), 6.86 (d,  $J$  = 2.15 Hz, 1H), 3.58 (s, 3H);

**$^{13}C$  NMR (100 MHz, DMSO- $d_6$ ):**  $\delta$  = 172.18, 156.33, 140.73, 138.43, 129.03, 128.86, 126.94, 125.38, 124.63, 124.44, 122.04, 120.63, 116.75, 115.38, 109.29, 96.71, 30.11.

**HRMS (ESI-TOF)** calcd for  $C_{17}H_{13}ClN_3O_2$   $[M + H]^+$ : 326.0691; found: 326.0692.

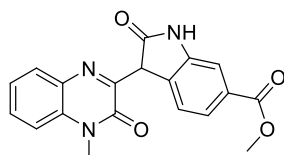


**3-(6-Bromo-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3i)** orange solid, mp: 248-250 °C, 51.3 mg, 65% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.50 (s, 1H), 11.08 (s, 1H), 8.63 (d,  $J$  = 8.65 Hz, 1H), 7.40-7.42 (m, 1H), 7.33-7.35 (m, 1H), 7.21-7.23 (m, 2H), 7.10 (dd,  $J$  = 2.05, 8.65 Hz, 1H), 7.02 (d,  $J$  = 2.00 Hz, 1H), 3.60 (s, 3H);

**$^{13}C$  NMR (125 MHz, DMSO- $d_6$ ):**  $\delta$  = 172.02, 156.34, 140.92, 138.65, 128.90, 127.31, 125.38, 124.66, 124.50, 123.49, 122.42, 117.25, 116.80, 115.41, 112.08, 96.69, 30.13.

**HRMS (ESI-TOF)** calcd for  $C_{17}H_{13}BrN_3O_2$   $[M + H]^+$ : 370.0186; found: 370.0184.

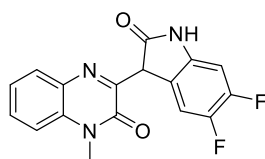


**3-(1-Methylquinoxalin-2-one)-2-oxoindoline-6-carboxylate (3j)** orange solid, mp: 284-286 °C, 53.6 mg, 73% yield;

**$^1H$  NMR (400 MHz, DMSO- $d_6$ ):**  $\delta$  = 14.78 (s, 1H), 11.13 (s, 1H), 8.72 (d,  $J$  = 8.36 Hz, 1H), 7.32-7.39 (m, 4H), 7.20-7.24 (m, 2H), 3.81 (s, 3H), 3.58 (s, 3H);

**$^{13}C$  NMR (100 MHz, DMSO- $d_6$ ):**  $\delta$  = 172.08, 166.87, 155.87, 141.85, 136.75, 129.04, 127.83, 125.03, 124.96, 124.93, 124.83, 124.56, 122.07, 117.12, 115.28, 109.66, 96.61, 52.21, 30.09.

**HRMS (ESI-TOF)** calcd for  $C_{19}H_{16}N_3O_4$   $[M + H]^+$ : 350.1135; found: 350.1138.

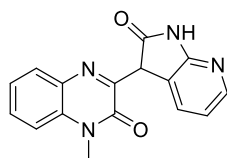


**3-(5,6-Difluoro-2-oxoindolin-3-yl)-1-methylquinoxalin-2-one (3k)** orange solid, mp: 261-263 °C, 52.4 mg, 74% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.44 (s, 1H), 11.07 (s, 1H), 8.65 (dd,  $J$  = 8.84, 13.92 Hz, 1H), 7.38-7.40 (m, 1H), 7.30-7.32 (m, 1H), 7.20-7.22 (m, 1H), 7.18-7.20 (m, 1H), 6.82 (dd,  $J$  = 7.36, 10.56 Hz, 1H), 3.59 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.31, 156.40, 140.69, 133.51, 128.78, 126.02, 125.28, 124.70, 124.56, 124.00, 123.67, 116.83, 115.85, 115.41-115.48 (d,  $J_{C-F}$  = 5.46 Hz), 113.81-114.05 (d,  $J_{C-F}$  = 17.49 Hz), 96.47, 30.11.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>12</sub>F<sub>2</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 328.0892; found: 328.0896.

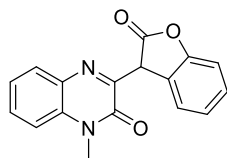


**3-(1H-pyrrolo [2,3-b] pyridine-2-one)-1-methylquinoxalin-2-one (3l)** orange solid, mp: 223-225 °C, 63.0 mg, 98% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.35 (s, 1H), 11.71 (s, 1H), 8.95 (d,  $J$  = 7.40 Hz, 1H), 7.99 (dd,  $J$  = 1.55, 5.10 Hz, 1H), 7.42-7.47 (m, 2H), 7.23-7.30 (m, 2H), 7.04-7.06 (m, 1H), 3.63 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 171.66, 156.14, 140.31, 136.69, 133.34, 129.04, 125.09, 124.95, 124.75, 117.50, 117.19, 115.52, 30.20.

**HRMS (ESI-TOF)** calcd for C<sub>16</sub>H<sub>13</sub>N<sub>4</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 293.1033; found: 293.1035.

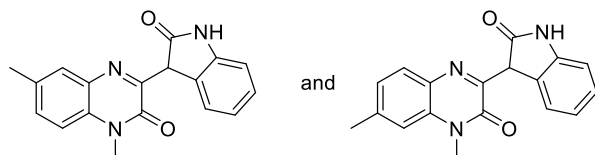


**3-(Benzofuran-2-one)-1-ethylquinoxalin-2-one (3m)** orange solid, mp: 124-126 °C, 40.9 mg, 66% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 12.98 (s, 1H), 8.68 (dd,  $J$  = 1.45, 7.80 Hz, 1H), 7.57 (dd,  $J$  = 1.47, 7.92 Hz, 1H), 7.50 (d,  $J$  = 8.15 Hz, 1H), 7.32 (dt,  $J$  = 1.50, 7.70 Hz, 1H), 7.27 (dt,  $J$  = 1.35, 7.62 Hz, 1H), 7.14-7.23 (m, 3H), 3.65 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 176.03, 155.34, 150.08, 149.80, 132.46, 129.03, 126.01, 125.75, 124.81, 123.86, 117.43, 115.47, 111.50, 110.05, 81.02, 30.25.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>13</sub>N<sub>2</sub>O<sub>3</sub> [M + H]<sup>+</sup>: 293.0921; found: 293.0924.



(regioisomeric mixture with 4:1 ratio)

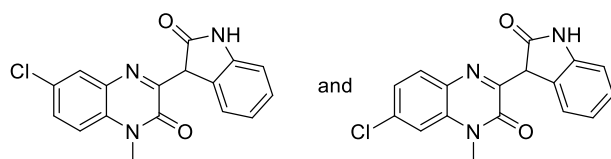
### 3-(2-oxoindolin-3-yl)-1, 6-dimethylquinoxalin-2-one

**3-(2-oxoindolin-3-yl)-1, 7-dimethylquinoxalin-2-one (3ab)** orange solid, mp: 239-241 °C, 53.2 mg, 82% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.57 (s, 0.25H), 14.52 (s, 1H), 10.95 (s, 0.93H), 10.92 (s, 0.31H), 8.72 (d, *J* = 7.80 Hz, 1.02H), 8.69 (s, 0.19H), 7.29 (d, *J* = 8.40 Hz, 1.04H), 7.24 (s, 0.22H), 7.22 (d, *J* = 8.10 Hz, 0.31H), 7.13 (d, *J* = 1.85 Hz, 1.02H), 7.01-7.07 (m, 2.59H), 6.89-6.95 (m, 2.59H), 3.60 (s, 0.65H), 3.59 (s, 2.85H), 2.37 (s, 0.64H), 2.33 (s, 2.93H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.20, 156.26, 140.41, 137.35, 134.26, 126.61, 125.86, 125.16, 125.02, 123.02, 121.06, 116.60, 115.21, 109.58, 97.62, 30.06, 21.36, 20.65.

**HRMS (ESI-TOF)** calcd for C<sub>18</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 306.1237; found: 306.1233.



(regioisomeric mixture with 2:1 ratio)

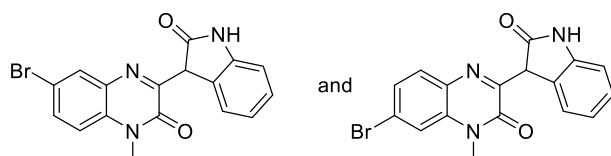
### 3-(2-oxoindolin-3-yl)-1-methyl-6-chloroquinoxalin-2-one

**3-(2-oxoindolin-3-yl)-1-methyl-7-chloroquinoxalin-2-one (3ac)** orange solid, mp: 255-257 °C, 38.2 mg, 54% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.48 (s, 0.58H), 14.39 (s, 1H), 10.99 and 10.98 (s, 1.64H), 8.70 and 8.67 (m, 1.63H), 7.47 (m, 1.63H), 7.33-7.38 (m, 1.69H), 7.19-7.22 (m, 1.69H), 7.06-7.10 (m, 1.64H), 6.89-6.96 (m, 3.36), 3.58 (s, 4.87H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.17, 156.47, 156.34, 139.70, 137.86, 137.66, 129.98, 128.47, 127.82, 126.18, 125.97, 125.67, 125.47, 124.81, 124.11, 123.30, 122.75, 121.15, 117.83, 116.78, 115.89, 115.09, 109.63, 98.96, 98.36, 30.24, 30.20.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>13</sub>ClN<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 326.0691; found: 326.0692.



(regioisomeric mixture with 4:1 ratio)

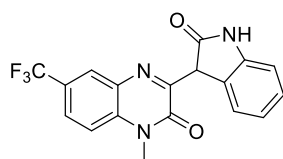
**3-(2-oxoindolin-3-yl)-1-methyl-6-bromoquinoxalin-2-one**

**3-(2-oxoindolin-3-yl)-1-methyl-7-bromoquinoxalin-2-one (3ad)** orange solid, mp: 268-270 °C, 68.6 mg, 89% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.47 (s, 0.25H), 14.38 (s, 1H), 11.00 (s, 1.24H), 8.69 (d, *J* = 8.05 Hz, 1.25H), 7.57 (s, 1.26H), 7.24-7.27 (m, 1.19H), 7.06-7.14 (m, 2.19H), 6.89-6.96 (m, 2.42H), 3.57 (s, 3.53H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.15, 156.34, 139.70, 137.86, 128.20, 127.27, 126.17, 126.13, 125.66, 125.15, 123.22, 122.75, 121.58, 121.14, 118.64, 117.08, 116.24, 109.62, 98.97, 30.15.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>13</sub>BrN<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 370.0186; found: 370.0182.

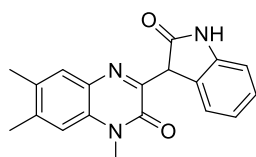


**3-(2-oxoindolin-3-yl)-1-methyl-6-trifluoromethylquinoxalin-2-one (3ae)** orange solid, mp: 291-293 °C, 38.4 mg, 53% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.43 (s, 1H), 11.02 (s, 1H), 8.69 (d, *J* = 8.00 Hz, 1H), 7.71 (d, *J* = 2.00 Hz, 1H), 7.54 (d, *J* = 8.60 Hz, 1H), 7.48 (dd, *J* = 1.95, 8.65 Hz, 1H), 7.09 (dt, *J* = 1.25, 7.52 Hz, 1H), 6.95 (dt, *J* = 1.30, 7.60 Hz, 1H), 6.91 (dd, *J* = 1.30, 7.60 Hz, 1H), 3.61 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.17, 156.74, 139.59, 137.98, 131.65, 126.22, 125.80-126.16 (d, *J*<sub>C-F</sub> = 36.15 Hz), 124.85, 124.52, 123.10, 122.63, 121.17, 120.06, 115.90, 113.34, 109.63, 99.21, 30.25.

**HRMS (ESI-TOF)** calcd for C<sub>18</sub>H<sub>12</sub>F<sub>3</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 360.0954; found: 360.0951.



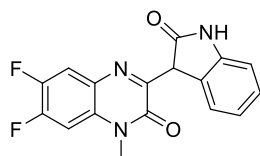
**3-(2-oxoindolin-3-yl)-1, 6, 7-trimethylquinoxalin-2-one (3af)** orange solid, mp: 244-246 °C, 45.9 mg, 63% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.58 (s, 1H), 10.93 (s, 1H), 7.22 (s, 1H), 7.16 (s, 1H), 6.81 (d, *J* = 7.75 Hz, 1H), 6.74-6.76 (m, 1H), 3.60 (s, 3H), 2.28 (s, 3H), 2.24 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 176.87, 161.03, 144.11, 136.50, 127.90, 126.22, 124.84, 121.61, 120.95, 116.08, 114.81, 109.56, 96.94, 31.42, 19.75, 19.11.

**HRMS (ESI-TOF)** calcd for C<sub>19</sub>H<sub>18</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 320.1394; found: 320.1395.



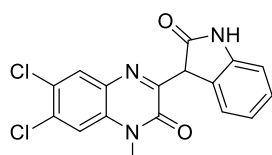


**3-(2-oxoindolin-3-yl)-1-methyl-6,7-difluoroquinoxalin-2-one (3ag)** orange solid, mp: 274-276 °C, 57.0 mg, 81% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.41 (s, 1H), 10.99 (s, 1H), 8.68 (d,  $J$  = 8.00 Hz, 1H), 7.63 (dd,  $J$  = 7.77, 10.92 Hz, 1H), 7.57 (dd,  $J$  = 7.55, 12.15 Hz, 1H), 7.07 (dt,  $J$  = 1.25, 7.60 Hz, 1H), 6.94 (dt,  $J$  = 1.30, 7.77 Hz, 1H), 6.90 (dd,  $J$  = 1.22, 7.67 Hz, 1H), 3.55 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 171.28, 156.31, 139.87, 137.69, 129.77, 126.00, 125.53, 122.81, 122.13, 109.62, 104.86-105.10 (d,  $J_{C-F}$  = 23.70 Hz), 30.67.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>12</sub>F<sub>2</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 328.0892; found: 328.0894.

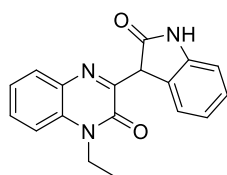


**3-(2-oxoindolin-3-yl)-1-methyl-6,7-dichloroquinoxalin-2-one (3ah)** orange solid, mp: 259-261 °C, 75.5 mg, 97% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.33 (s, 1H), 11.01 (s, 1H), 8.66 (d,  $J$  = 8.00 Hz, 1H), 7.67 (s, 1H), 7.59 (s, 1H), 7.08 (t,  $J$  = 7.55 Hz, 1H), 6.94 (t,  $J$  = 7.60 Hz, 1H), 6.90 (d,  $J$  = 7.65 Hz, 1H), 3.55 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 173.54, 156.43, 143.81, 138.03, 130.07, 128.92, 126.22, 125.93, 124.09, 121.57, 116.70, 110.05, 94.92, 30.42.

**HRMS (ESI-TOF)** calcd for C<sub>17</sub>H<sub>12</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 360.0301; found: 360.0304.

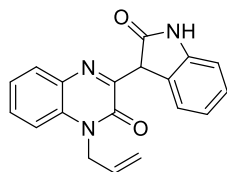


**3-(2-oxoindolin-3-yl)-1-ethylquinoxalin-2-one (3ai)** orange solid, mp: 259-261 °C, 58.7 mg, 90% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.51 (s, 1H), 10.94 (s, 1H), 8.71 (d,  $J$  = 7.96 Hz, 1H), 7.40-7.43 (m, 1H), 7.28-7.31 (m, 1H), 7.15-7.21 (m, 2H), 7.05 (dt,  $J$  = 1.24, 7.44 Hz, 1H), 6.88-6.95 (m, 2H), 4.21-4.26 (q,  $J$  = 7.07 Hz, 2H), 1.27 (t,  $J$  = 7.04 Hz, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.19, 156.00, 140.26, 137.48, 127.44, 125.92, 125.88, 125.17, 124.49, 124.21, 123.01, 121.00, 116.89, 115.03, 109.52, 97.74, 37.82, 12.53.

**HRMS (ESI-TOF)** calcd for C<sub>18</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 306.1237; found: 306.1231.

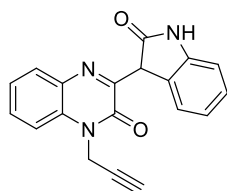


**3-((2-oxoindolin-3-yl)-1-allyl)quinoxalin-2-one (3aj)** orange solid, mp: 257-259 °C, 62.5 mg, 93% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.52 (s, 1H), 10.97 (s, 1H), 8.69 (d, *J* = 7.96 Hz, 1H), 7.28-7.32 (m, 2H), 7.14-7.18 (m, 2H), 7.06 (dt, *J* = 1.24, 7.48 Hz, 1H), 6.89-6.95 (m, 2H), 5.92-6.01 (m, 1H), 5.22-5.25 (m, 1H), 5.19-5.20 (m, 1H), 4.84-4.86 (m, 1H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.21, 156.29, 140.17, 137.55, 132.07, 127.75, 125.93, 125.28, 124.60, 123.97, 122.95, 121.04, 117.48, 116.72, 115.66, 109.56, 98.03, 44.86.

**HRMS (ESI-TOF)** calcd for C<sub>19</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 318.1237; found: 318.1239.

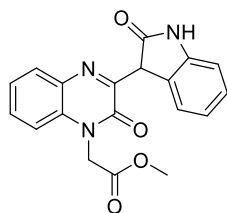


**3-((2-oxoindolin-3-yl)-1-(2-propynyl))quinoxalin-2-one (3ak)** orange solid, mp: 272-274 °C, 44.2 mg, 64% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.45 (s, 1H), 11.01 (s, 1H), 8.68 (d, *J* = 8.00 Hz, 1H), 7.45-7.46 (m, 1H), 7.34-7.36 (m, 1H), 7.23-7.24 (m, 2H), 7.08 (t, *J* = 7.47 Hz, 1H), 6.92 (t, *J* = 7.67 Hz, 1H), 5.08 (d, *J* = 2.50 Hz, 1H), 3.46 (m, 1H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.69, 156.04, 138.89, 137.69, 130.35, 127.40, 125.87, 125.63, 125.17, 124.08, 123.28, 121.19, 118.23, 115.57, 112.70, 109.74, 78.64, 75.45, 32.31.

**HRMS (ESI-TOF)** calcd for C<sub>19</sub>H<sub>14</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 316.1081; found: 316.1085.

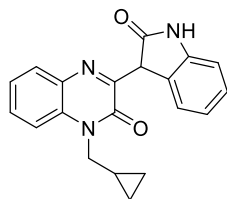


**3-(2-oxoindolin-3-yl)-1-methylacetatequinoxalin-2-one (3al)** orange solid, mp: 278-280 °C, 72.2 mg, 94% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.46 (s, 1H), 11.02 (s, 1H), 8.63 (d, *J* = 7.48 Hz, 1H), 7.35-7.39 (m, 2H), 7.15-7.24 (m, 2H), 7.09 (dt, *J* = 1.24, 7.52 Hz, 1H), 6.90-6.96 (m, 2H), 5.10 (s, 2H), 3.74 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.17, 168.67, 156.78, 139.27, 137.77, 127.87, 125.84, 125.67, 125.64, 124.97, 124.11, 122.63, 121.14, 116.87, 115.18, 109.67, 98.51, 52.94, 44.59.

**HRMS (ESI-TOF)** calcd for C<sub>19</sub>H<sub>16</sub>N<sub>3</sub>O<sub>4</sub> [M + H]<sup>+</sup>: 350.1135; found: 350.1132.

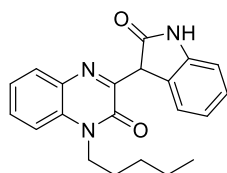


**3-(2-oxoindolin-3-yl)-1-cyclopropylmethylquinoxalin-2-one (3am)** orange solid, mp: 267-269 °C, 63.8 mg, 91% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.53 (s, 1H), 10.96 (s, 1H), 8.67 (d, *J* = 8.05 Hz, 1H), 7.52-7.54 (m, 1H), 7.32-7.34 (m, 1H), 7.19-7.21 (m, 2H), 7.06 (t, *J* = 7.55 Hz, 1H), 6.94 (t, *J* = 7.67 Hz, 1H), 6.91 (d, *J* = 7.60 Hz, 1H), 4.19 (d, *J* = 6.95 Hz, 1H), 1.26-1.34 (m, 2H), 0.51-0.52 (m, 4H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.19, 156.59, 140.24, 137.51, 127.90, 125.95, 125.77, 125.25, 124.62, 124.21, 122.97, 121.07, 116.92, 115.58, 109.57, 97.89, 46.25, 9.98, 4.27.

**HRMS (ESI-TOF)** calcd for C<sub>20</sub>H<sub>18</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 332.1394; found: 332.1392.

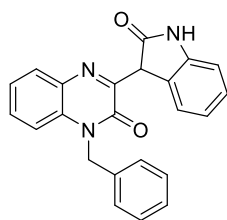


**3-(2-oxoindolin-3-yl)-1-pentylmethylquinoxalin-2-one (3an)** orange solid, mp: 231-233 °C, 57.7 mg, 77% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.51 (s, 1H), 10.94 (s, 1H), 8.69 (dd, *J* = 1.20, 8.04 Hz, 1H), 7.35-7.38 (m, 1H), 7.27-7.29 (m, 1H), 7.16-7.18 (m, 2H), 7.05 (dt, *J* = 1.24, 7.48 Hz, 1H), 6.88-6.95 (m, 2H), 4.15 (t, *J* = 7.76 Hz, 1H), 1.62-1.70 (m, 2H), 1.33-1.42 (m, 4H), 3.32 (t, *J* = 6.94 Hz, 1H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.19, 156.19, 140.18, 137.48, 127.66, 125.92, 125.83, 125.17, 124.48, 124.17, 123.00, 121.01, 116.86, 115.12, 109.52, 97.78, 42.58, 28.93, 26.70, 22.40, 14.36.

**HRMS (ESI-TOF)** calcd for C<sub>21</sub>H<sub>22</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 348.1707; found: 348.1702.

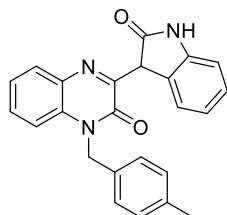


**3-(2-oxoindolin-3-yl)-1-benzylquinoxalin-2-one (3ao)** orange solid, mp: 293-295 °C, 60.2 mg, 76% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.57 (s, 1H), 11.01 (s, 1H), 8.69 (d, *J* = 7.72 Hz, 1H), 7.34-7.36 (m, 4H), 7.22-7.30 (m, 3H), 7.16 (t, *J* = 6.98 Hz, 1H), 7.06-7.11 (m, 2H), 6.91-6.95 (m, 3H), 5.50 (s, 2H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.27, 157.00, 140.21, 137.63, 136.36, 129.18, 128.92, 127.86, 127.72, 127.13, 126.00, 125.40, 124.75, 123.99, 122.96, 121.09, 116.83, 115.74, 109.62, 98.28, 45.93.

**HRMS (ESI-TOF)** calcd for C<sub>23</sub>H<sub>18</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 368.1394; found: 368.1392.

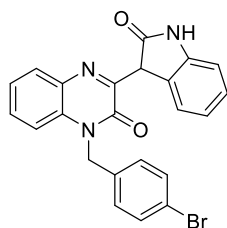


**3-(2-oxoindolin-3-yl)-1-*p*-Methylphenylquinoxalin-2-one (3ap)** orange solid, mp: 285-287 °C, 38.6 mg, 47% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.57 (s, 1H), 10.99 (s, 1H), 8.70 (d, *J* = 7.95 Hz, 1H), 7.35 (dd, *J* = 1.37, 7.92 Hz, 1H), 7.24-7.26 (m, 3H), 7.14-7.16 (m, 3H), 7.06-7.10 (m, 2H), 6.91-6.95 (m, 2H), 5.46 (s, 2H), 2.26 (s, 3H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 170.95, 156.99, 140.41, 138.33, 131.28, 129.73, 127.82, 127.15, 124.74, 117.15, 110.18, 97.69, 15.66, 21.10.

**HRMS (ESI-TOF)** calcd for C<sub>24</sub>H<sub>20</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 382.1550; found: 382.1552.

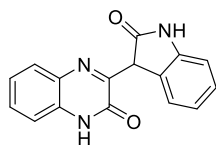


**3-(2-oxoindolin-3-yl)-1-*p*-bromophenylquinoxalin-2-one (3aq)** orange solid, mp: 300-301 °C, 49.6 mg, 52% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.55 (s, 1H), 11.00 (s, 1H), 8.68 (d, *J* = 7.88 Hz, 1H), 7.54 (d, *J* = 8.32 Hz, 2H), 7.32-7.35 (m, 3H), 7.14-7.22 (m, 2H), 7.05-7.10 (m, 2H), 6.90-6.94 (m, 2H), 5.45 (s, 2H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.26, 157.04, 140.18, 137.63, 135.90, 132.02, 129.52, 127.75, 126.05, 125.98, 125.40, 124.80, 123.98, 122.93, 121.08, 120.80, 116.84, 115.61, 109.61, 98.30, 45.43.

**HRMS (ESI-TOF)** calcd for C<sub>23</sub>H<sub>17</sub>BrN<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 446.0499; found: 446.0451.



**3-(2-oxoindolin-3-yl)-quinoxalin-2(1H)-one (3ar)**<sup>[3]</sup> orange solid, mp: 408-410 °C, 38.0 mg, 59% yield;

**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 14.40 (s, 1H), 11.98 (s, 1H), 10.94 (s, 1H), 8.71 (dd, *J* = 1.12, 8.04 Hz, 1H), 7.27-7.30 (m, 1H), 7.10-7.16 (m, 3H), 7.06 (dt, *J* = 1.28, 7.56 Hz, 1H), 6.89-6.96 (m, 2H);

**<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>):**  $\delta$  = 172.11, 157.10, 141.48, 137.48, 127.89, 127.33, 125.96, 125.19, 124.27, 124.01, 123.07, 121.03, 116.20, 115.42, 109.52, 97.96.

**HRMS (ESI-TOF)** calcd for C<sub>16</sub>H<sub>12</sub>N<sub>3</sub>O<sub>2</sub> [M + H]<sup>+</sup>: 278.0924; found: 278.0919.

## 7. Reference

- [1] W. L. F. Armarego, C. Chai, in *Purification of Laboratory Chemicals (Seventh Edition)*, Butterworth-Heinemann, Boston, **2013**, pp. 103-554.
- [2] J. Zhou, Q. Ren, N. Xu, C. Wang, S. Song, Z. Chen, J. Li, *Green Chem.* **2021**, *23*, 5753-5758.
- [3] Y.-Y. Han, Z.-J. Wu, X.-M. Zhang, W.-C. Yuan, *Tetrahedron Lett.* **2010**, *51*, 2023-2028.

## 8. Copies of $^1\text{H}$ NMR and $^{13}\text{C}$ NMR Spectra

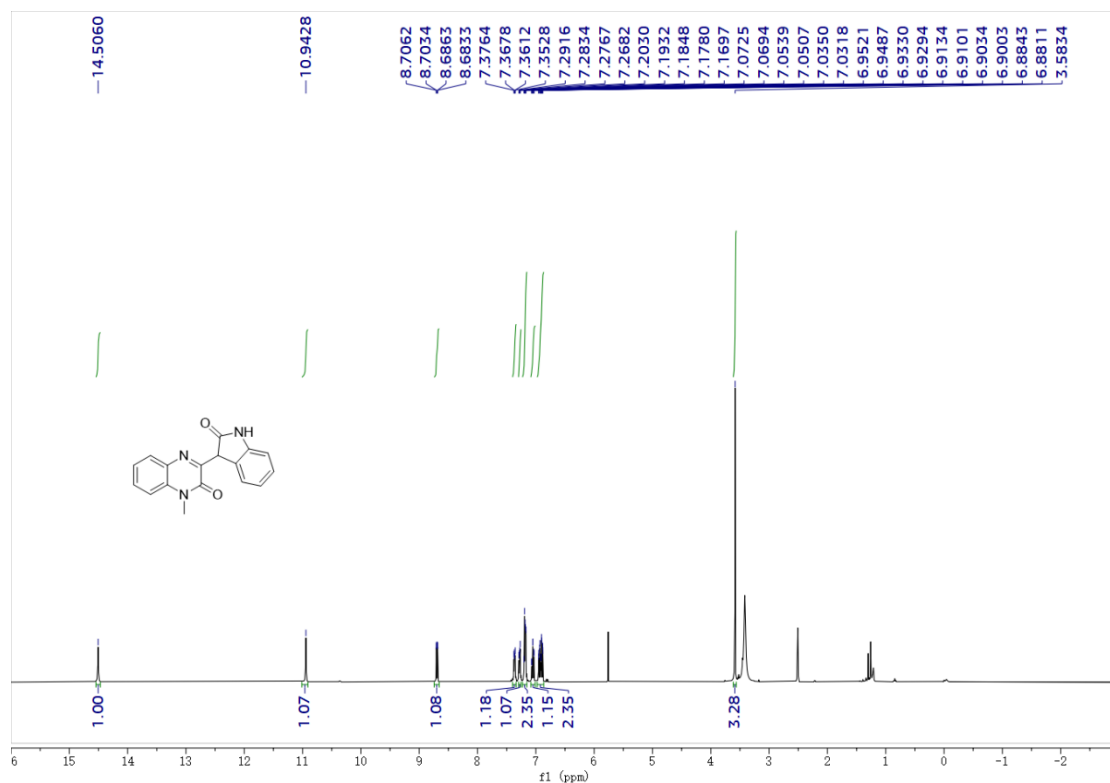


Figure S1 The  $^1\text{H}$  NMR Spectrum of Compound 3a in  $\text{DMSO-}d_6$

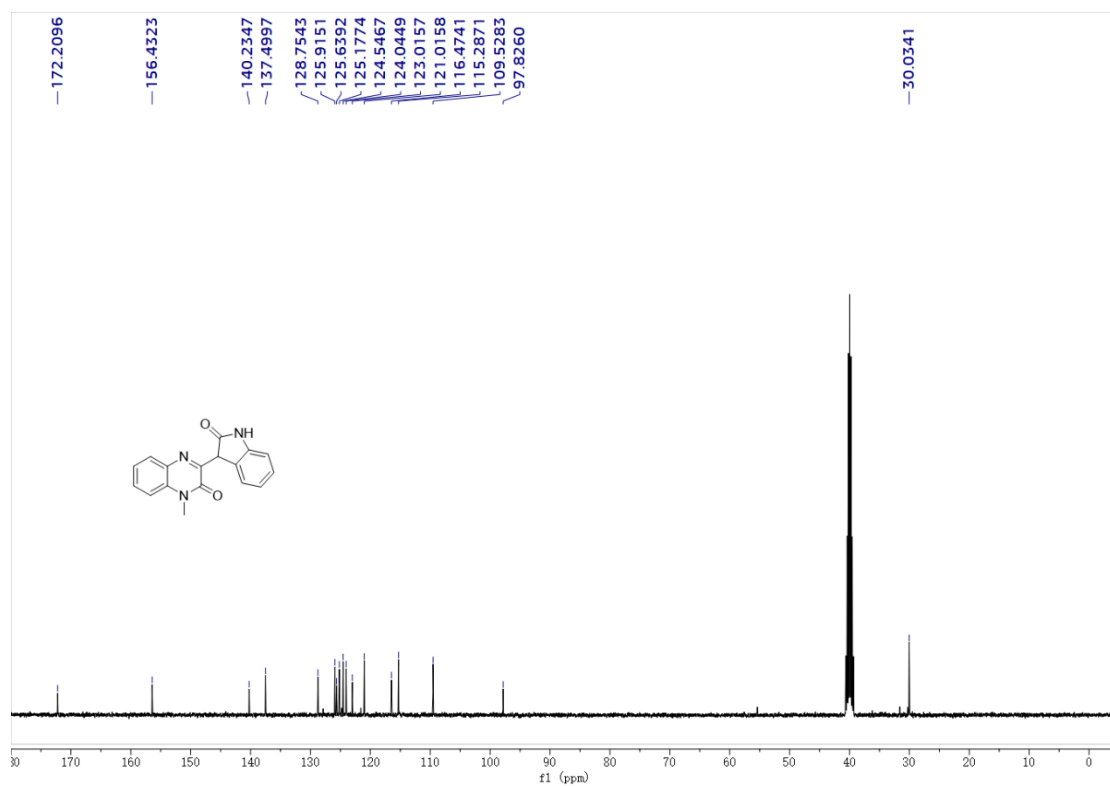


Figure S2 The  $^{13}\text{C}$  NMR Spectrum of Compound 3a in  $\text{DMSO-}d_6$

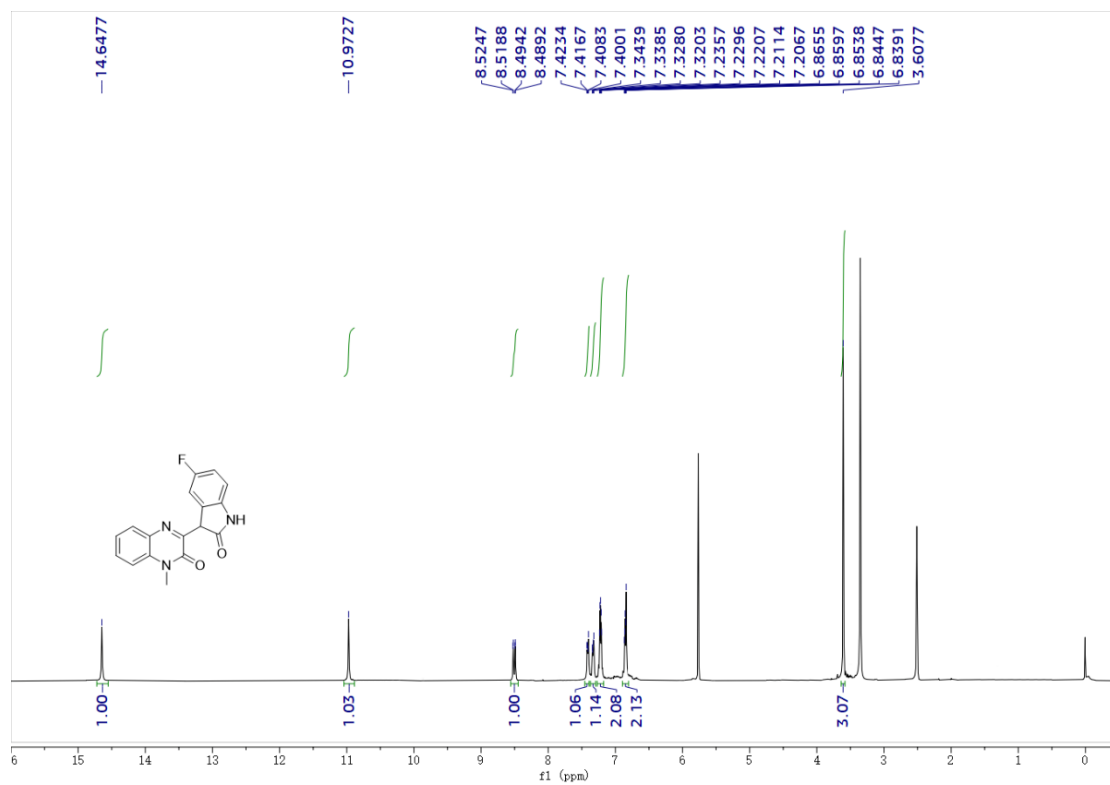


Figure S3 The <sup>1</sup>H NMR Spectrum of Compound 3b in DMSO-*d*<sub>6</sub>

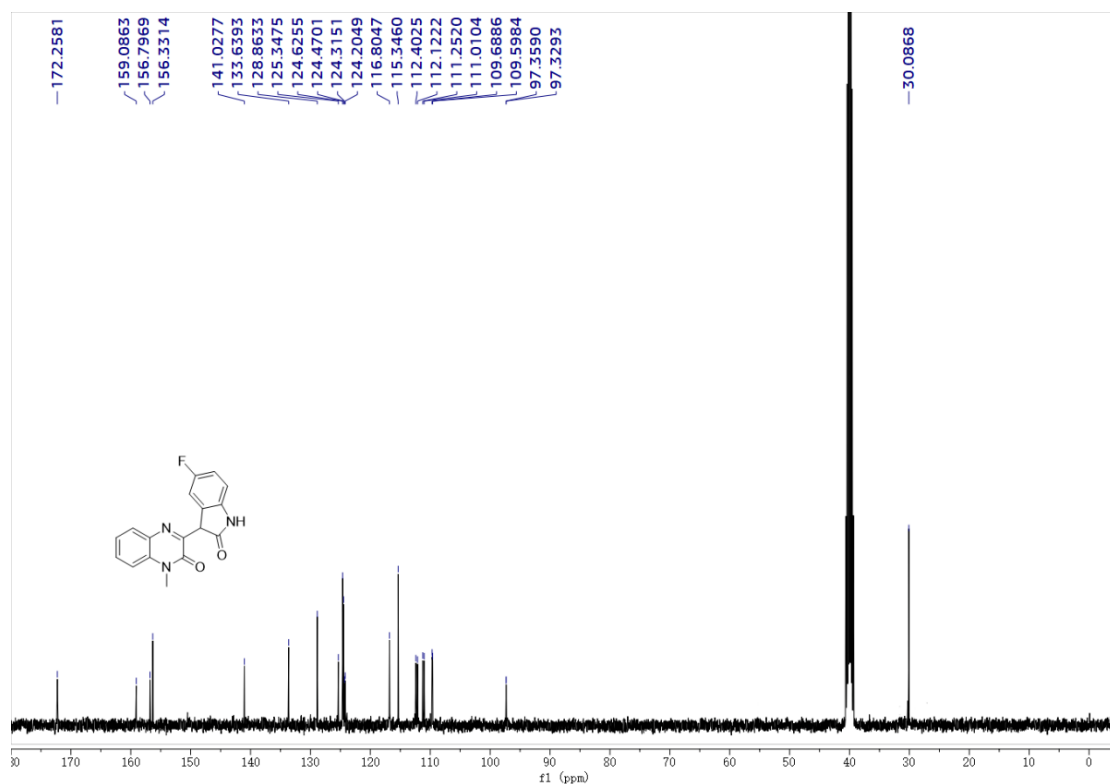


Figure S4 The <sup>13</sup>C NMR Spectrum of Compound 3b in DMSO-*d*<sub>6</sub>

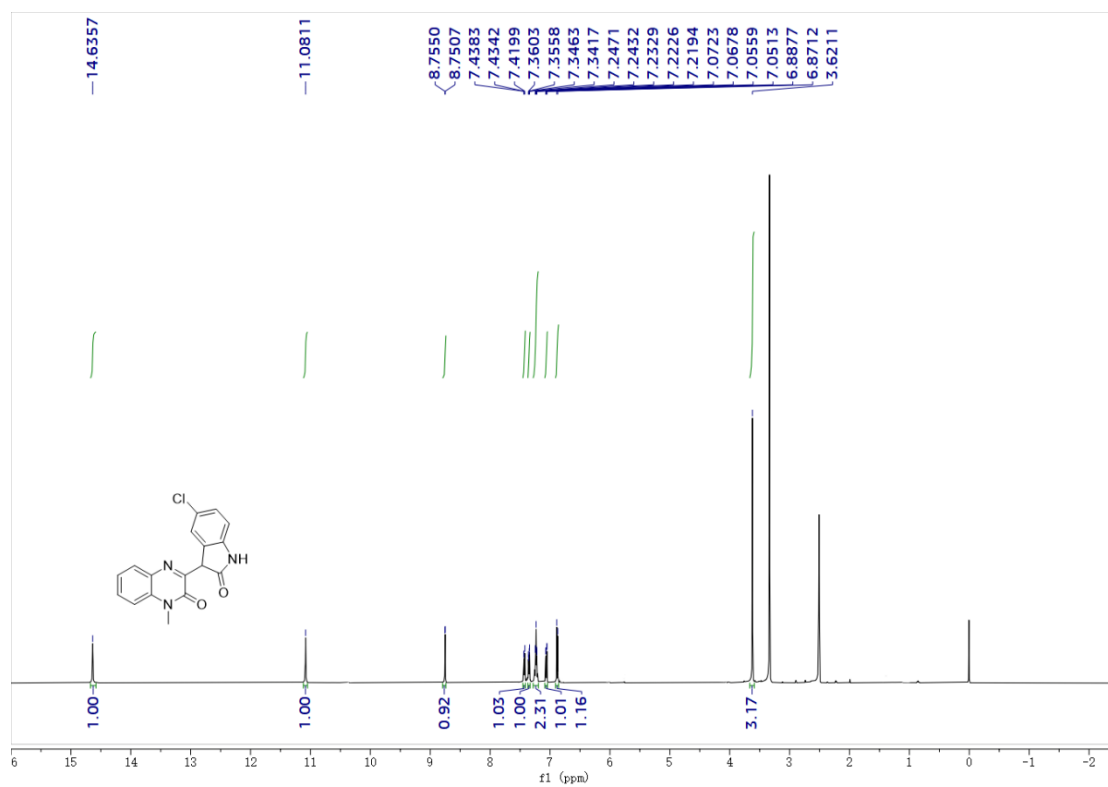


Figure S5 The  $^1\text{H}$  NMR Spectrum of Compound 3c in  $\text{DMSO-}d_6$

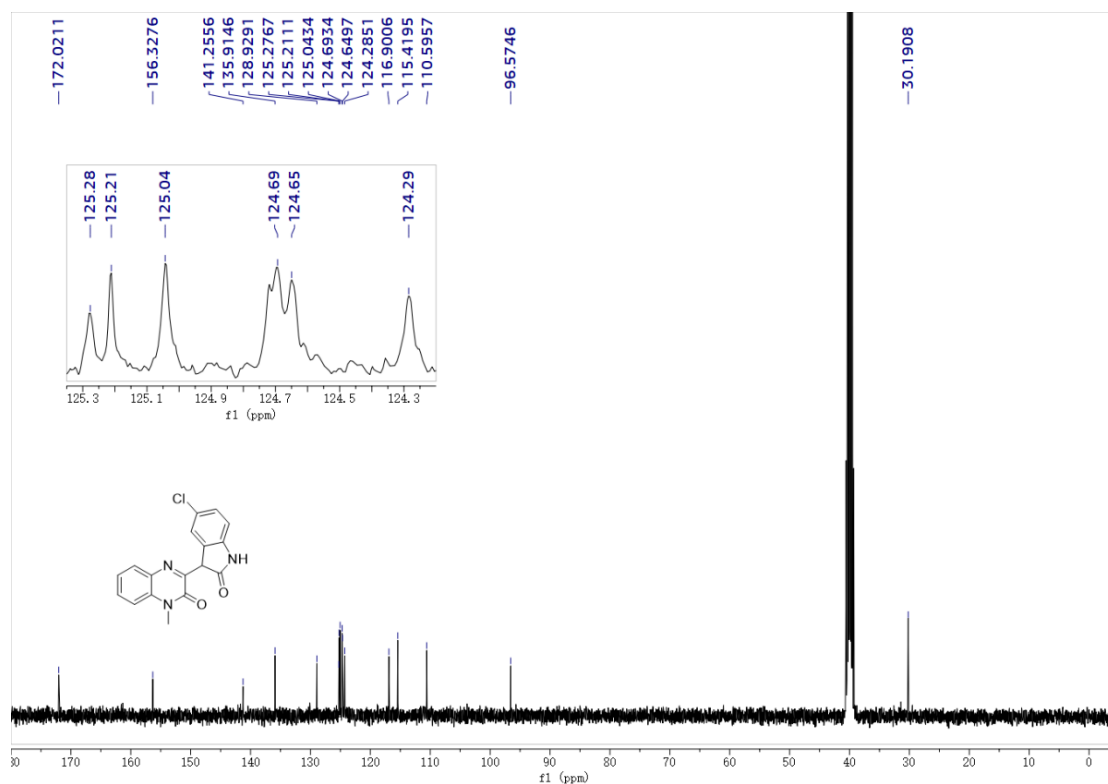


Figure S6 The  $^{13}\text{C}$  NMR Spectrum of Compound 3c in  $\text{DMSO-}d_6$



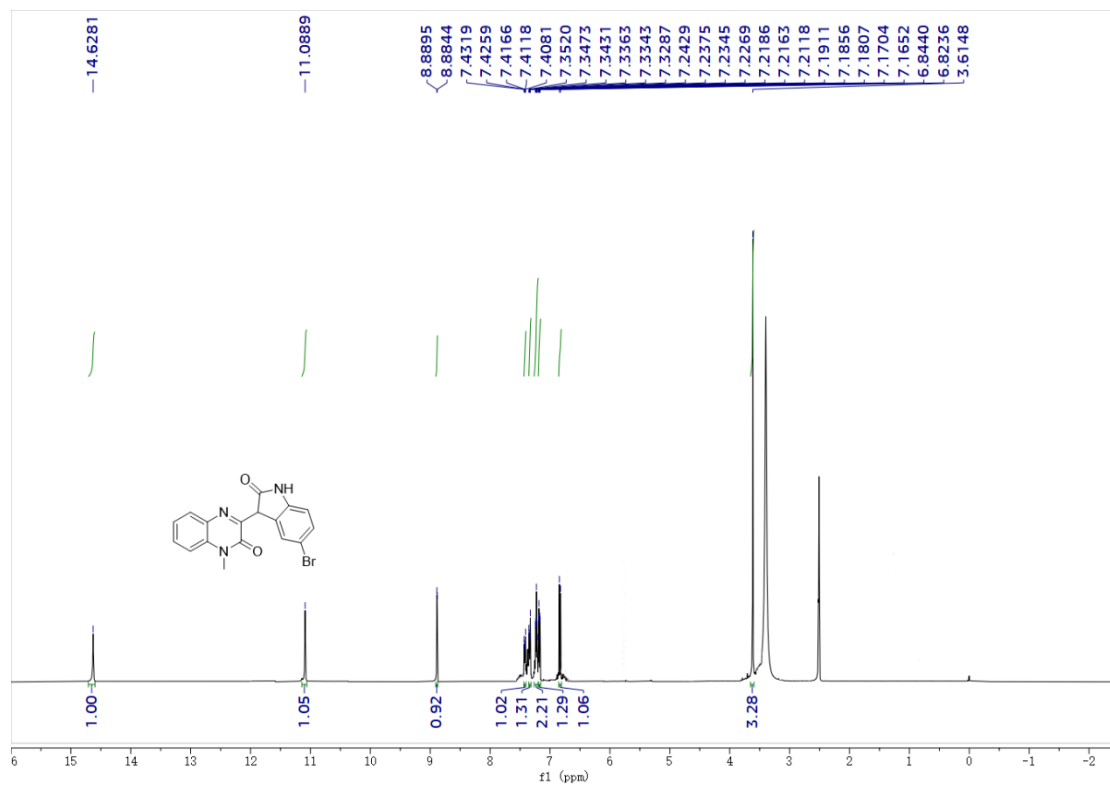


Figure S7 The <sup>1</sup>H NMR Spectrum of Compound 3d in DMSO-*d*<sub>6</sub>

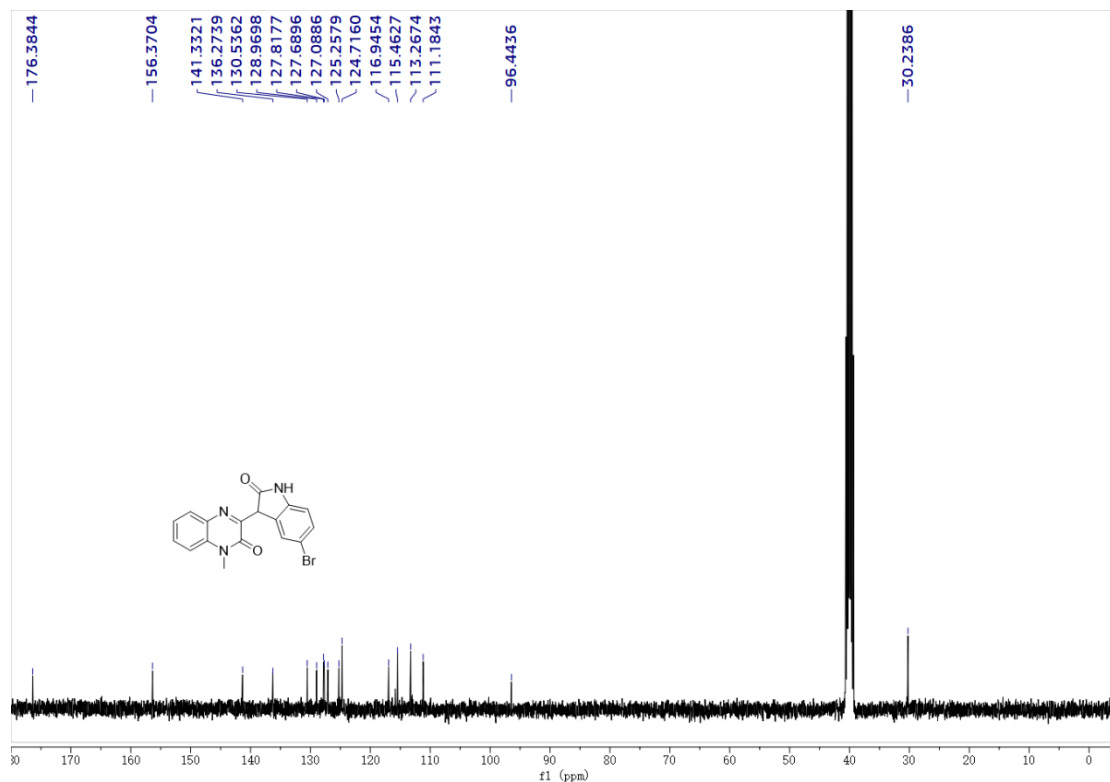


Figure S8 The <sup>13</sup>C NMR Spectrum of Compound 3d in DMSO-*d*<sub>6</sub>

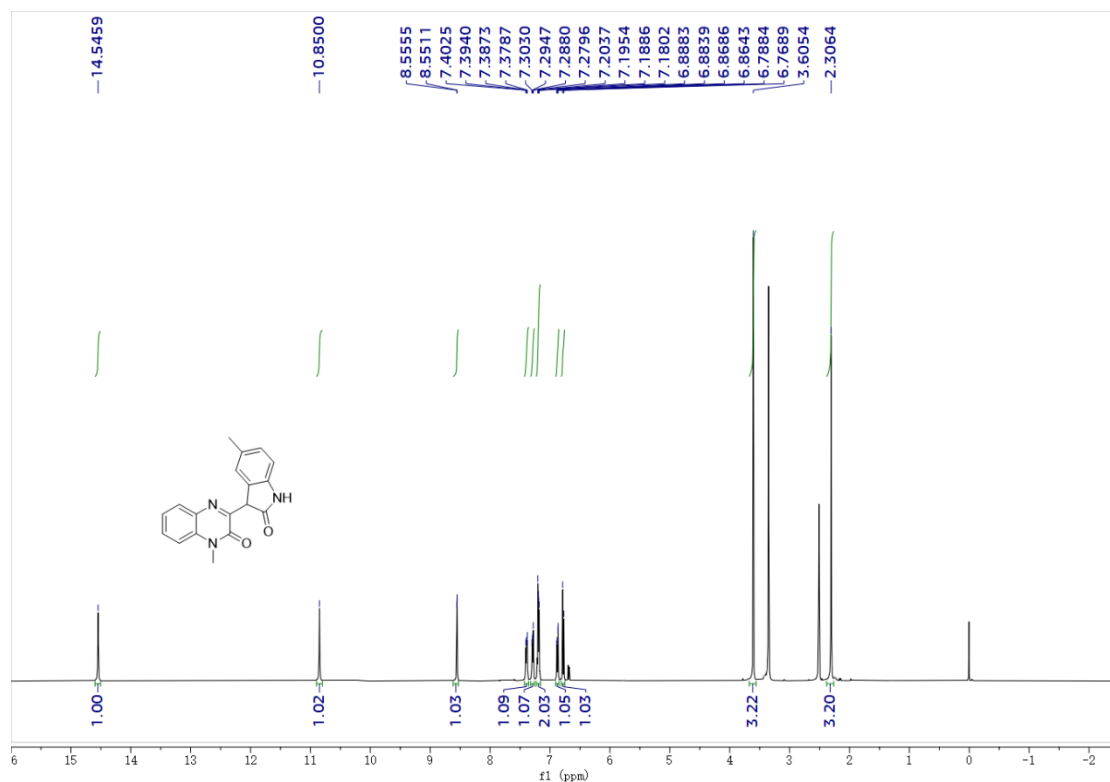


Figure S9 The  $^1\text{H}$  NMR Spectrum of Compound 3e in  $\text{DMSO-}d_6$

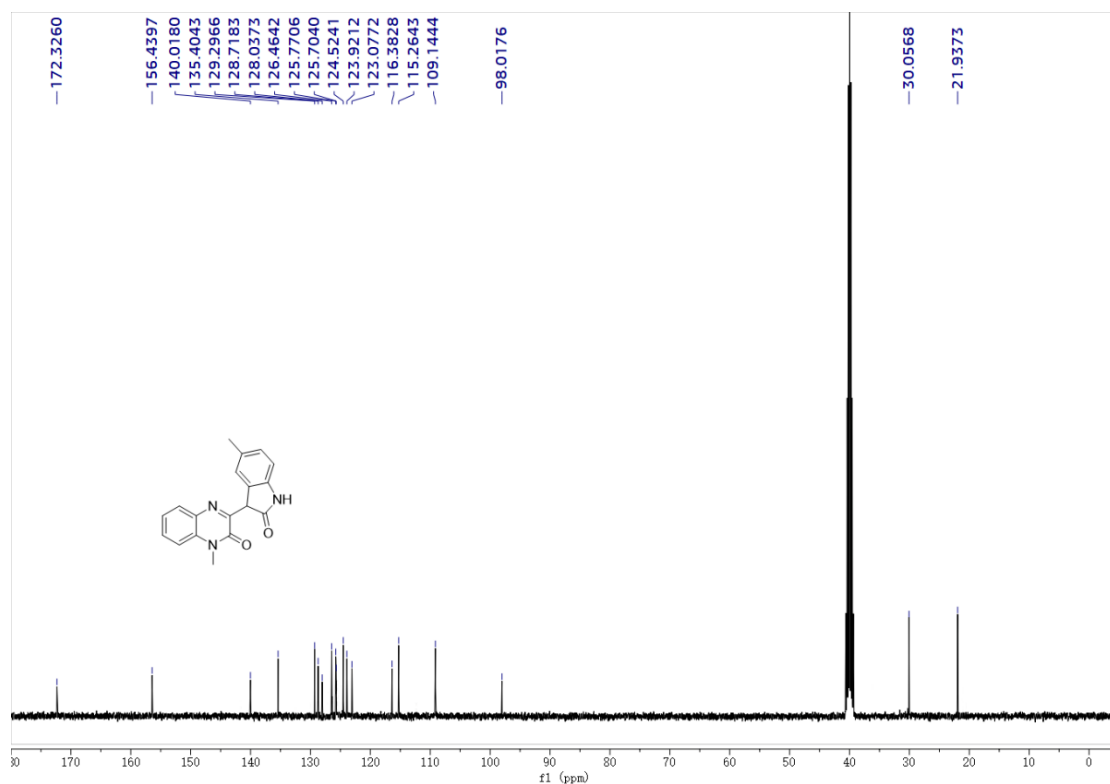


Figure S10 The  $^{13}\text{C}$  NMR Spectrum of Compound 3e in  $\text{DMSO-}d_6$

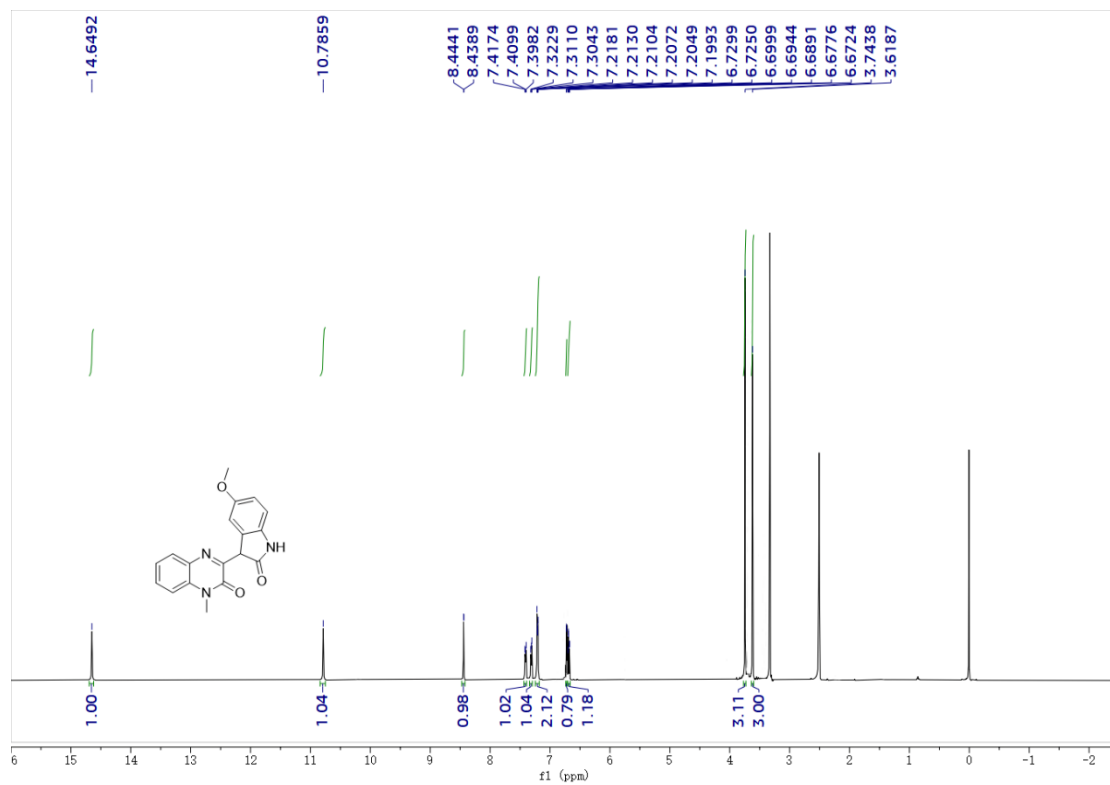


Figure S11 The  $^1\text{H}$  NMR Spectrum of Compound 3f in  $\text{DMSO-}d_6$

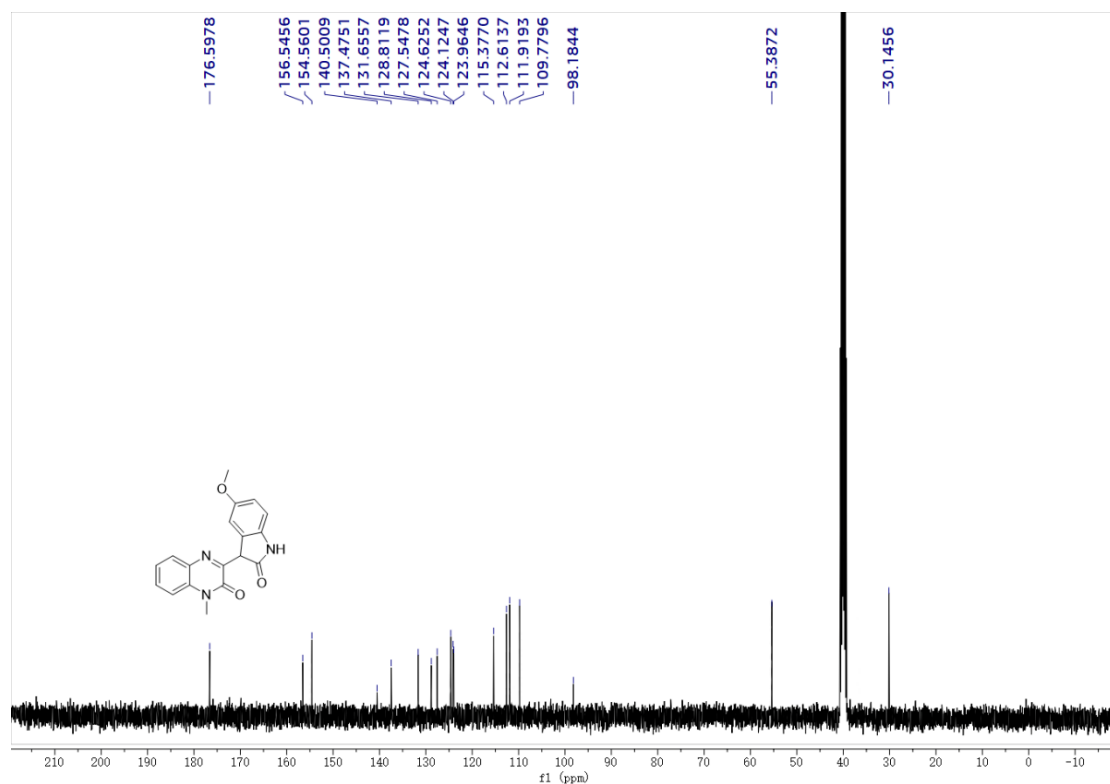


Figure S12 The  $^{13}\text{C}$  NMR Spectrum of Compound 3f in  $\text{DMSO-}d_6$

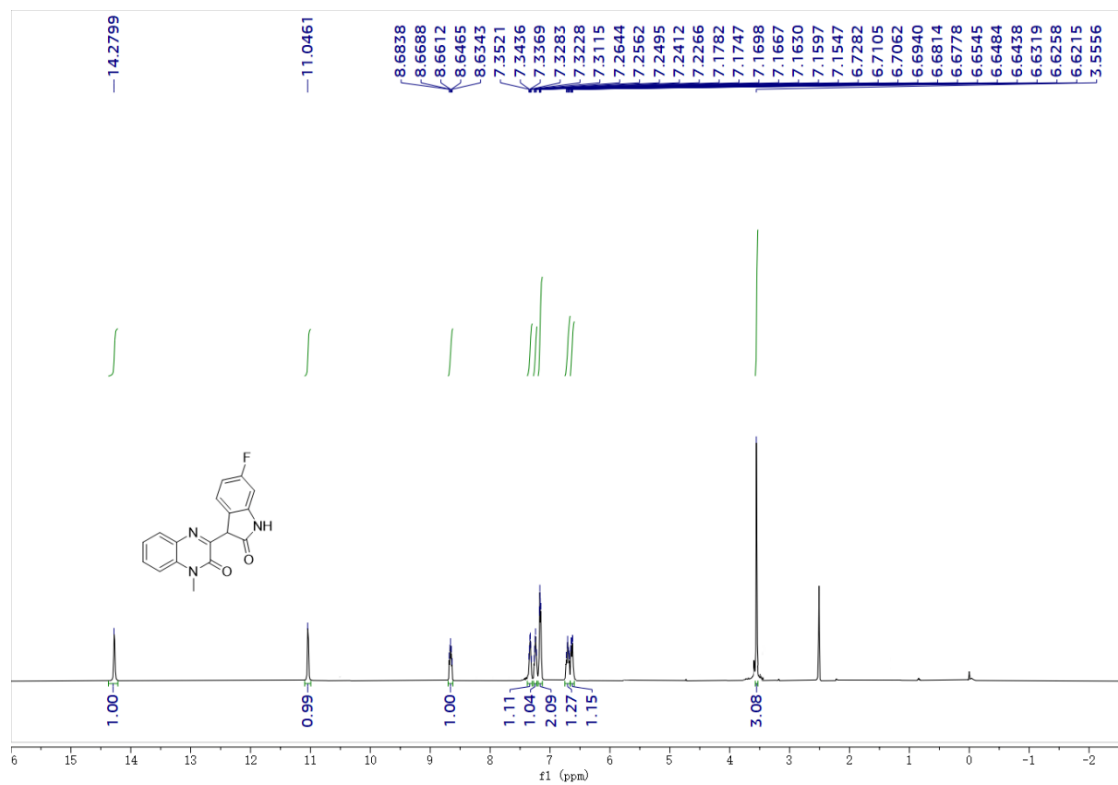


Figure S13 The  $^1\text{H}$  NMR Spectrum of Compound **3g** in  $\text{DMSO-}d_6$

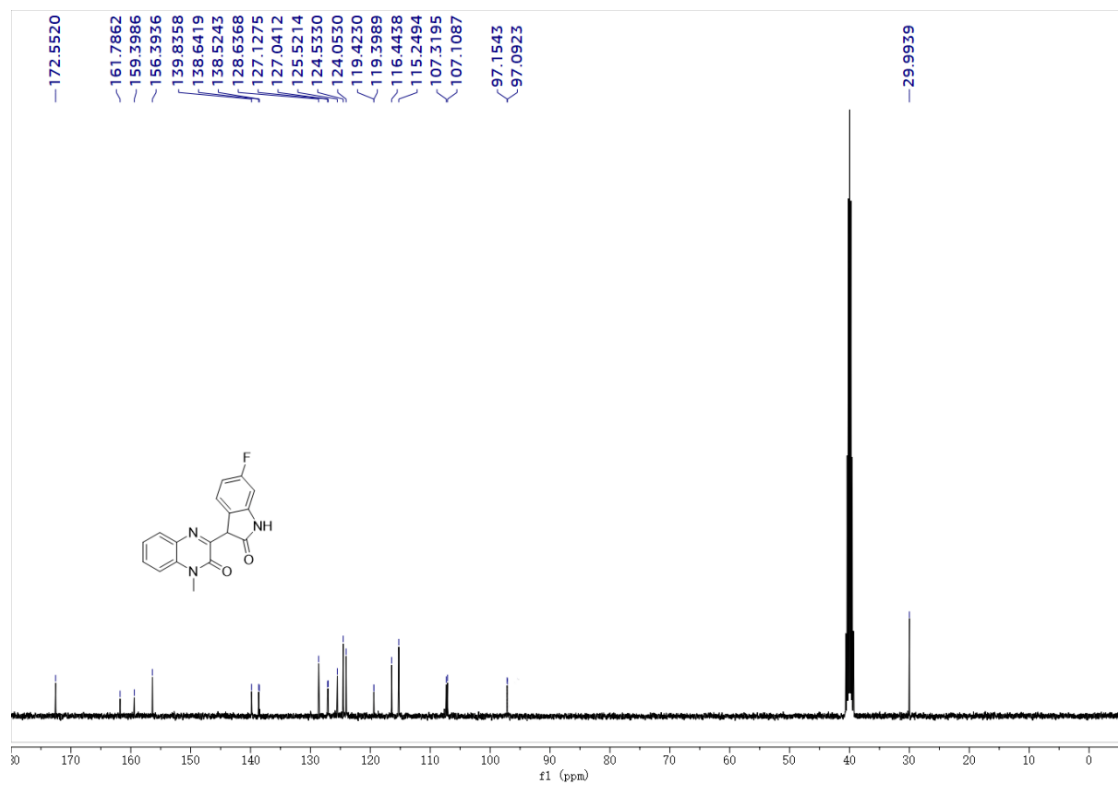


Figure S14 The  $^{13}\text{C}$  NMR Spectrum of Compound **3g** in  $\text{DMSO-}d_6$

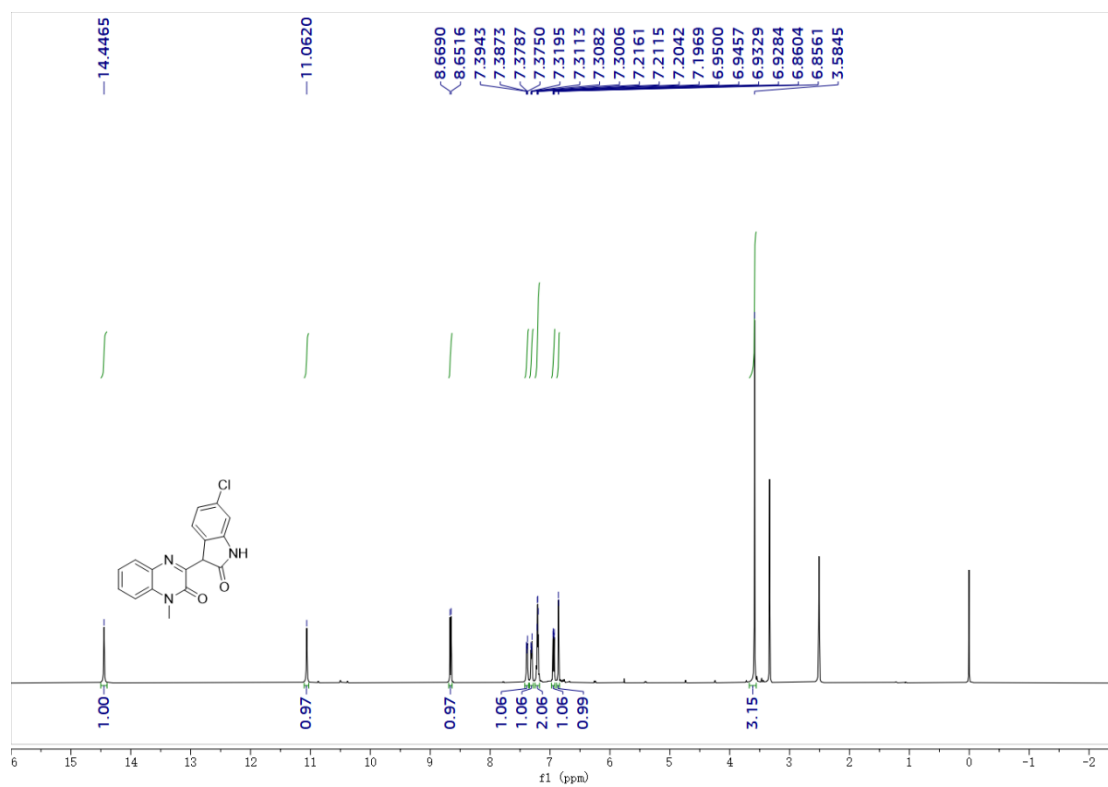


Figure S15 The  $^1\text{H}$  NMR Spectrum of Compound 3h in  $\text{DMSO-}d_6$

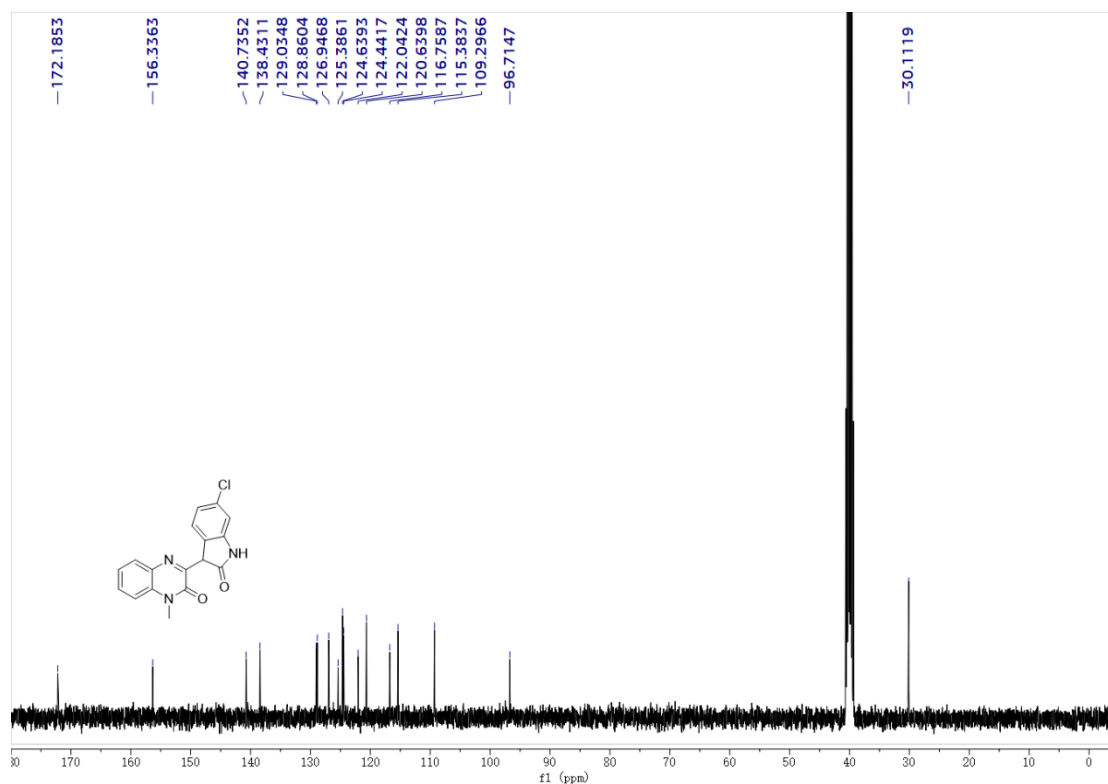


Figure S16 The  $^{13}\text{C}$  NMR Spectrum of Compound 3h in  $\text{DMSO-}d_6$

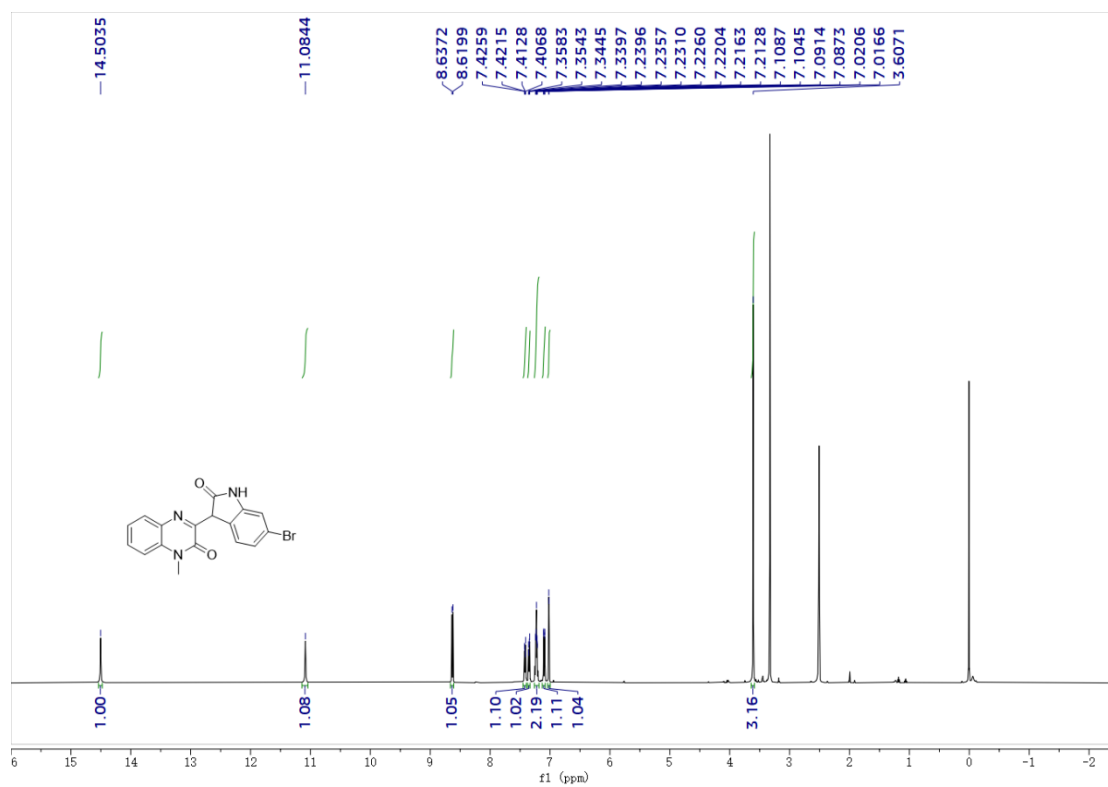


Figure S17 The  $^1\text{H}$  NMR Spectrum of Compound 3i in  $\text{DMSO}-d_6$

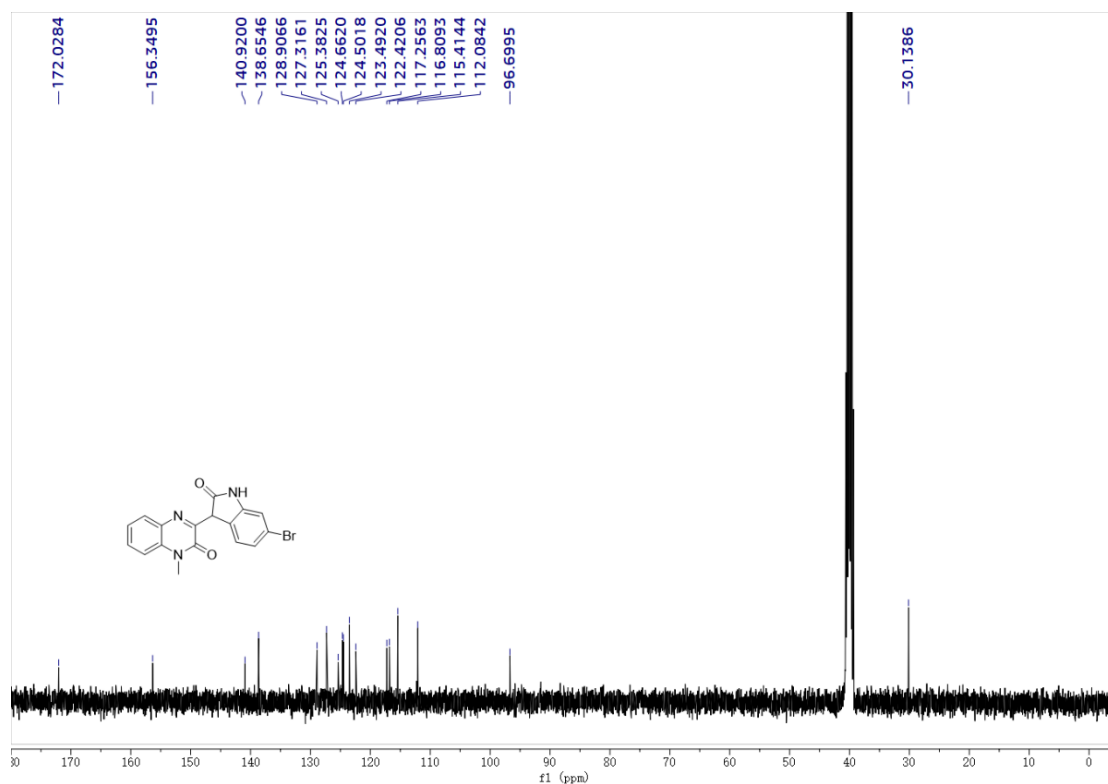


Figure S18 The  $^{13}\text{C}$  NMR Spectrum of Compound 3i in  $\text{DMSO}-d_6$

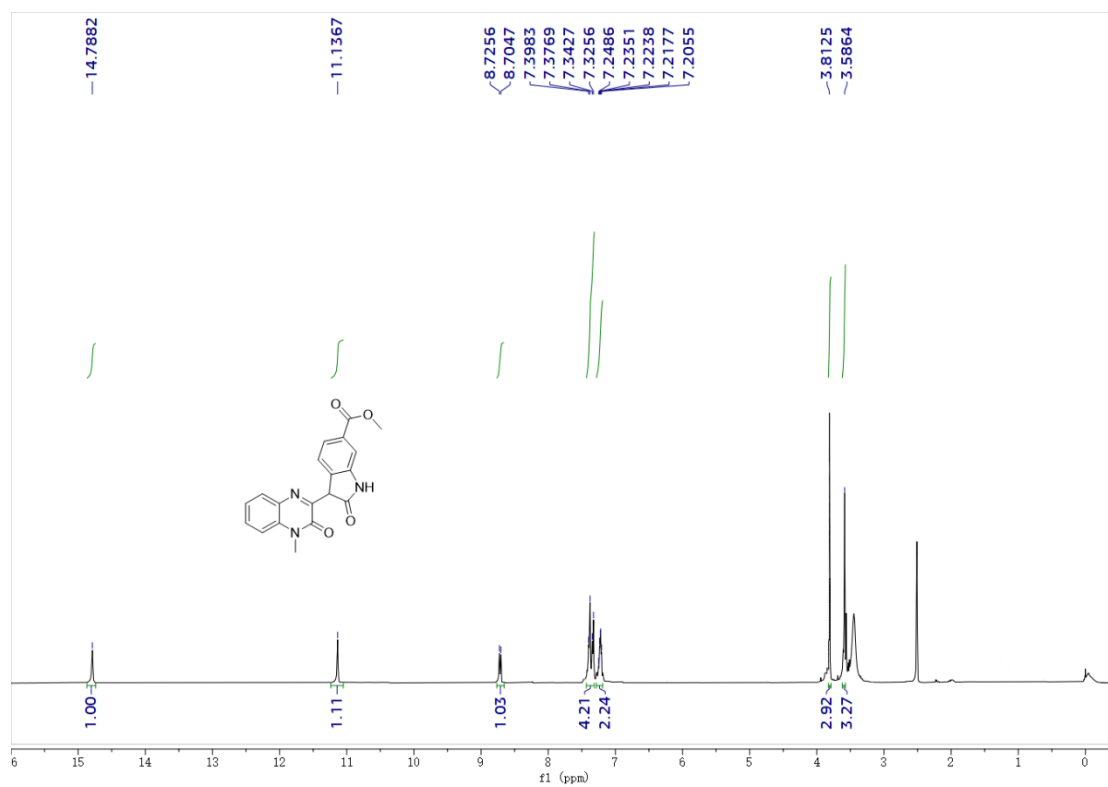


Figure S19 The  $^1\text{H}$  NMR Spectrum of Compound 3j in  $\text{DMSO-}d_6$

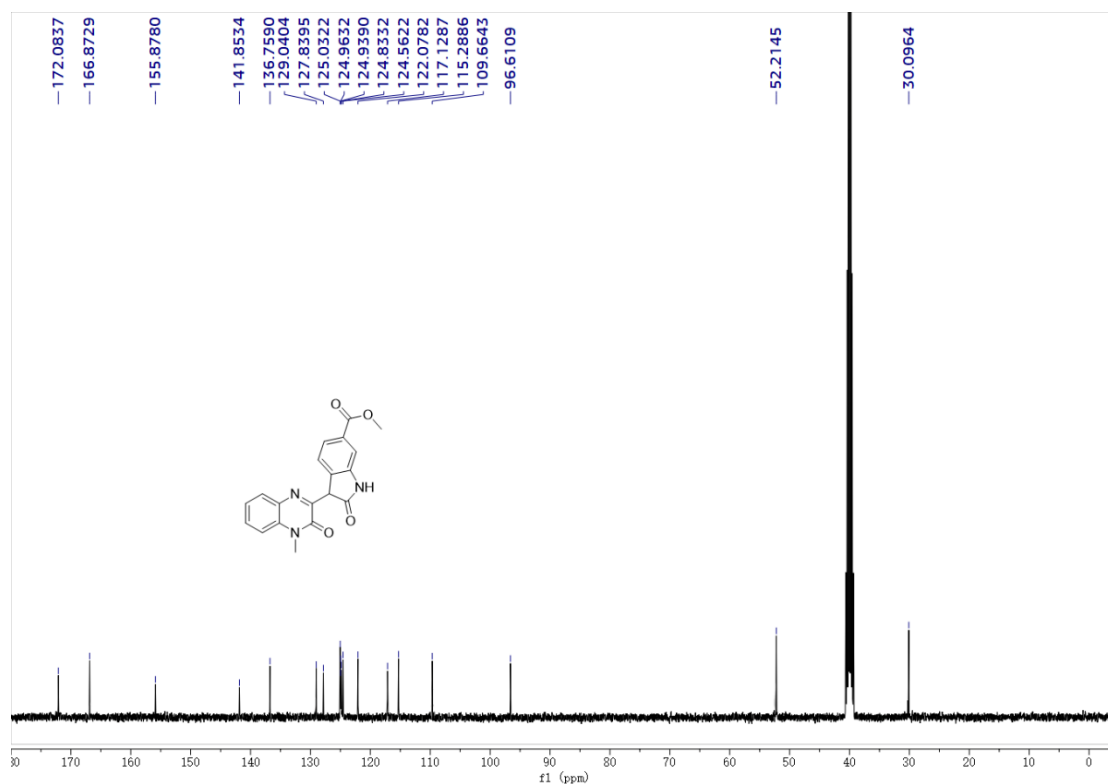


Figure S20 The  $^{13}\text{C}$  NMR Spectrum of Compound 3j in  $\text{DMSO-}d_6$

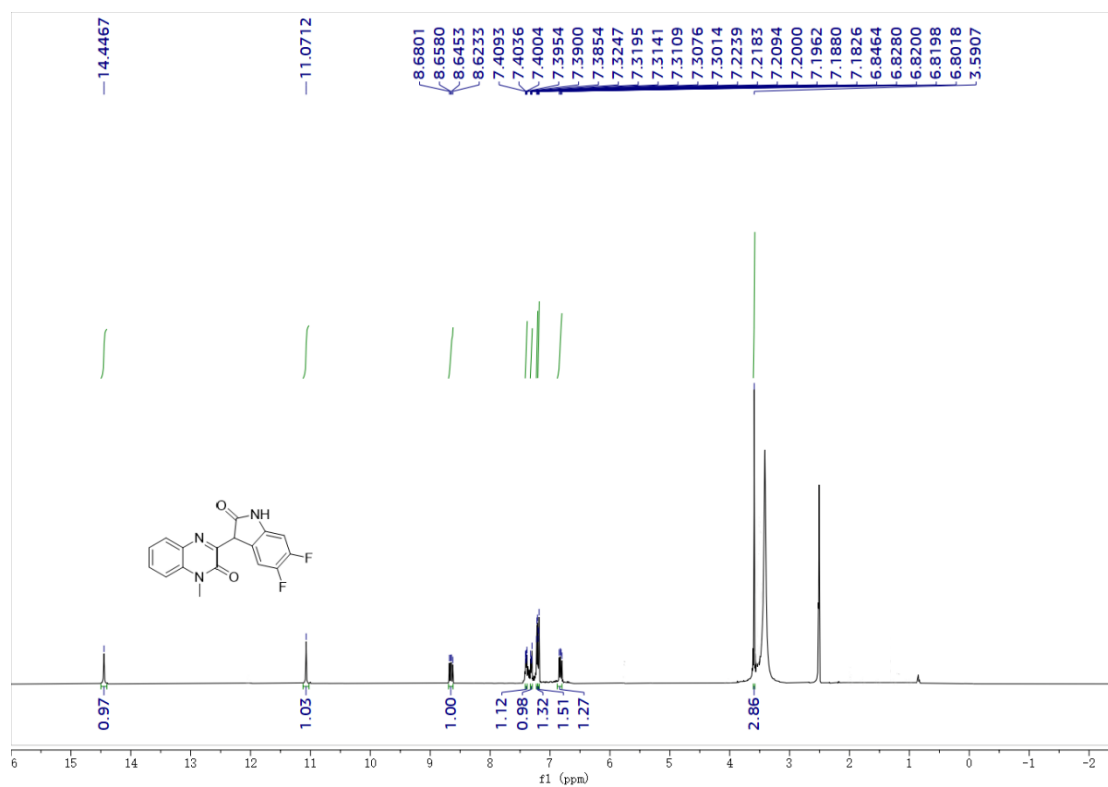


Figure S21 The  $^1\text{H}$  NMR Spectrum of Compound 3k in  $\text{DMSO-}d_6$

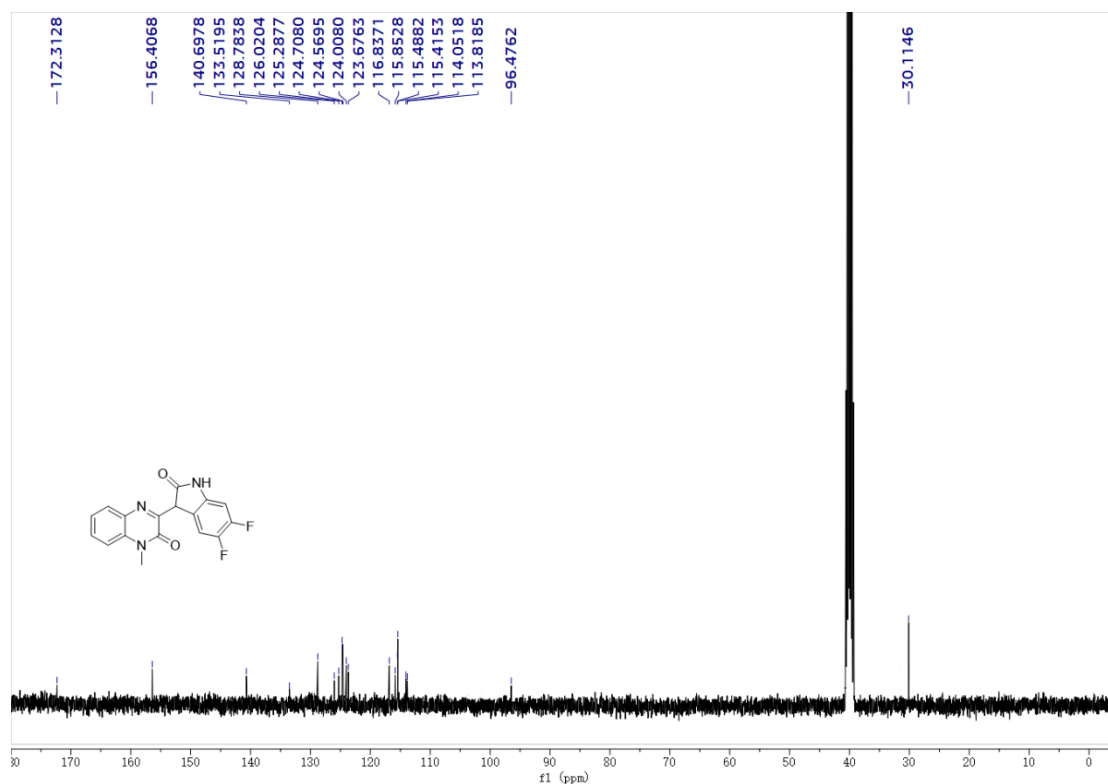


Figure S22 The  $^{13}\text{C}$  NMR Spectrum of Compound 3k in  $\text{DMSO-}d_6$



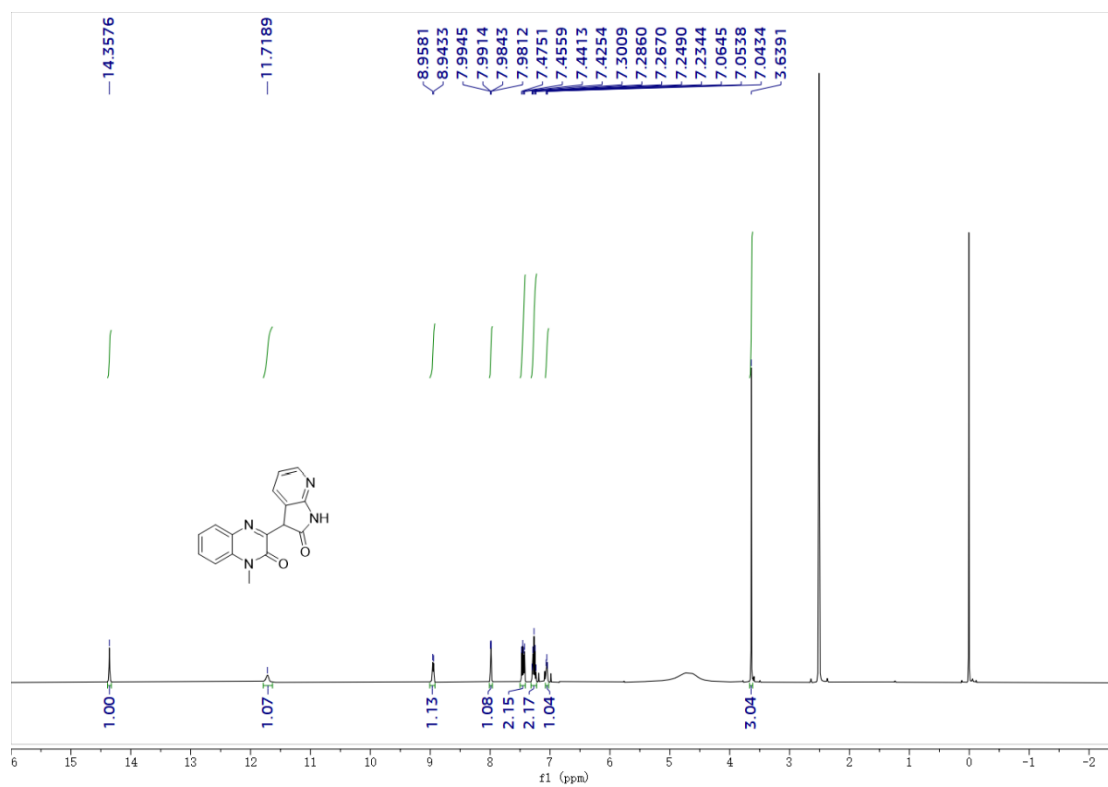


Figure S23 The  $^1\text{H}$  NMR Spectrum of Compound 31 in  $\text{DMSO}-d_6$

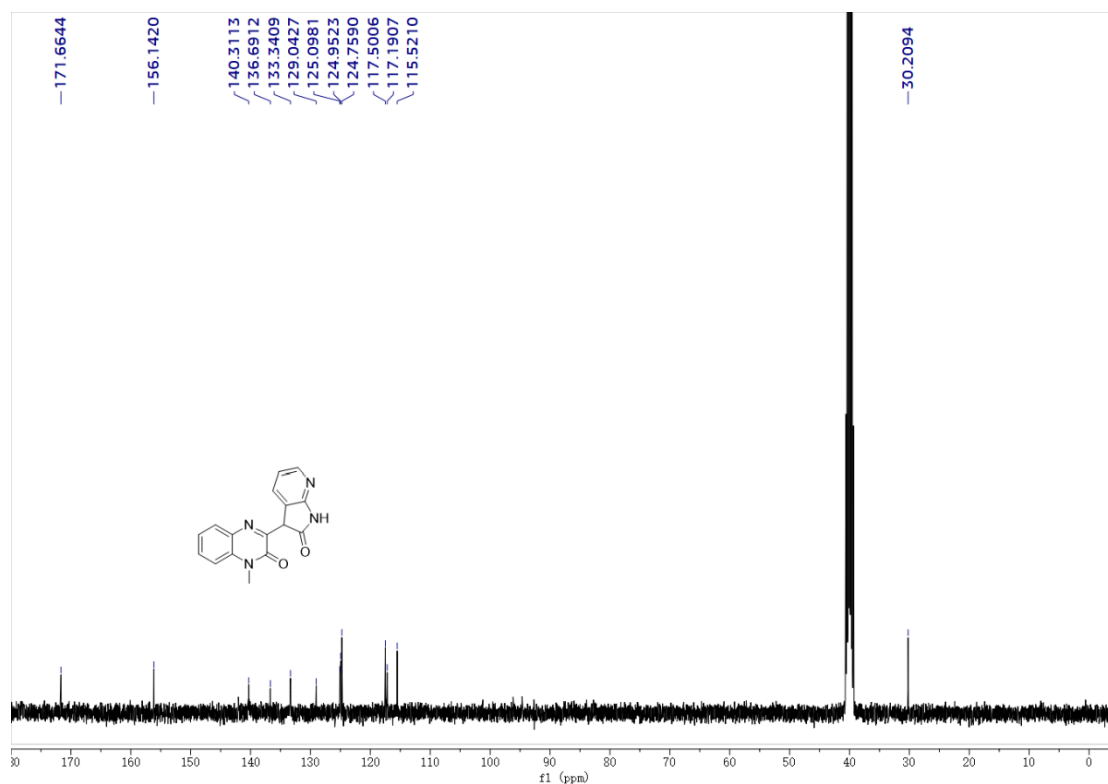


Figure S24 The  $^{13}\text{C}$  NMR Spectrum of Compound 31 in  $\text{DMSO}-d_6$

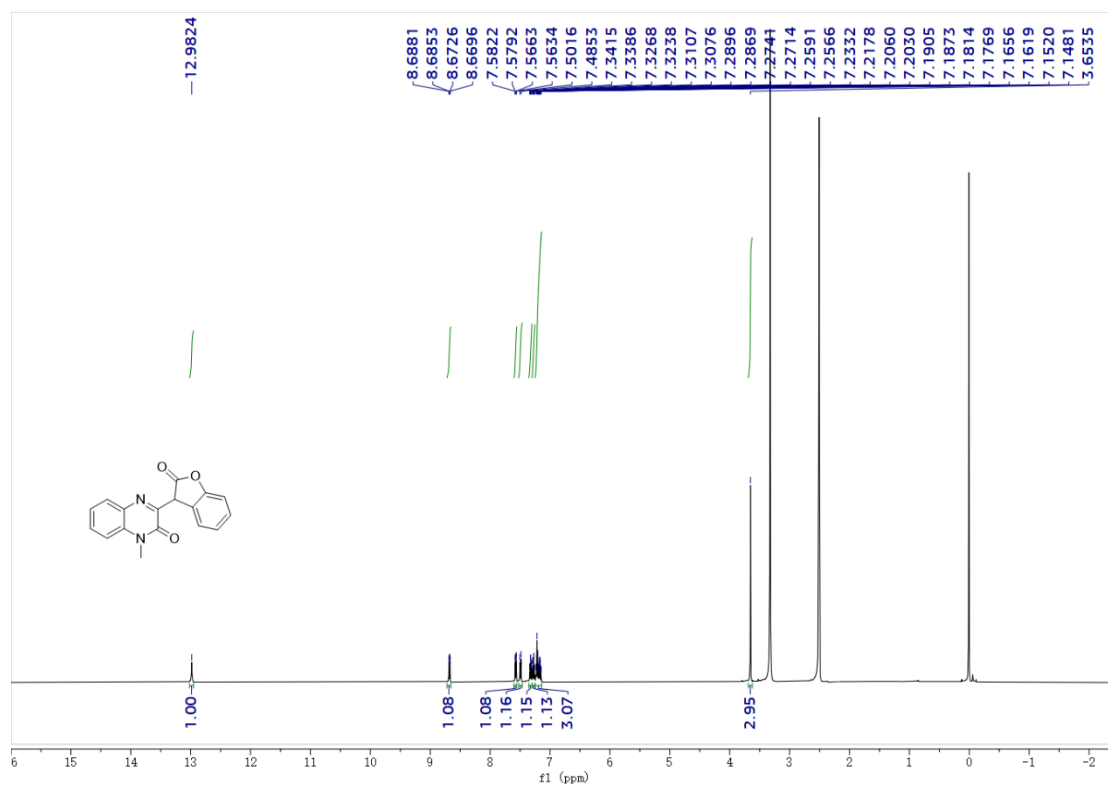


Figure S25 The  $^1\text{H}$  NMR Spectrum of Compound **3m** in  $\text{DMSO-}d_6$

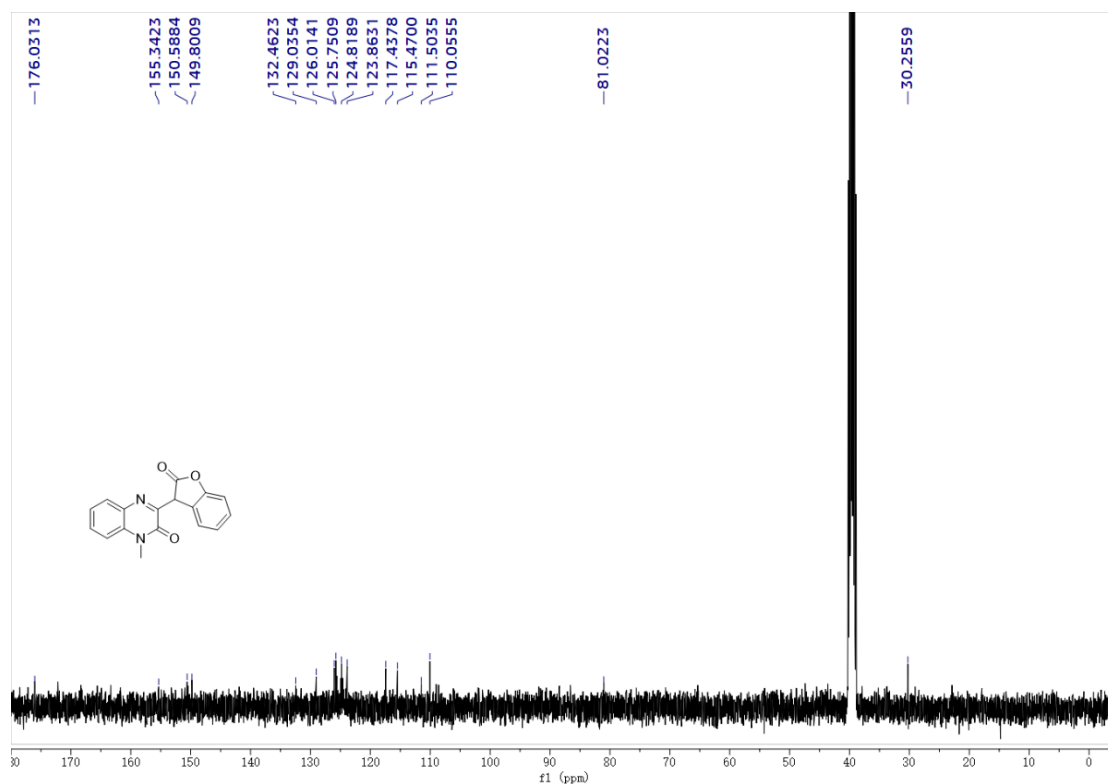


Figure S26 The  $^{13}\text{C}$  NMR Spectrum of Compound **3m** in  $\text{DMSO-}d_6$

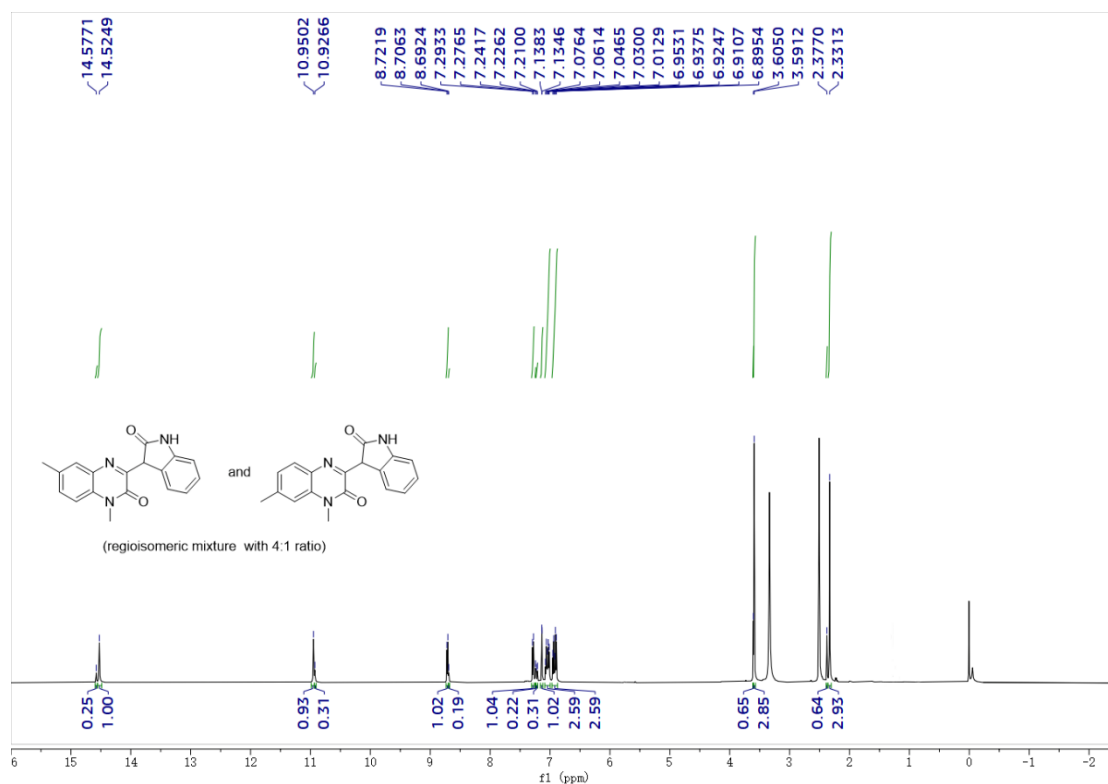


Figure S27 The <sup>1</sup>H NMR Spectrum of Compound **3ab** in DMSO-*d*<sub>6</sub>

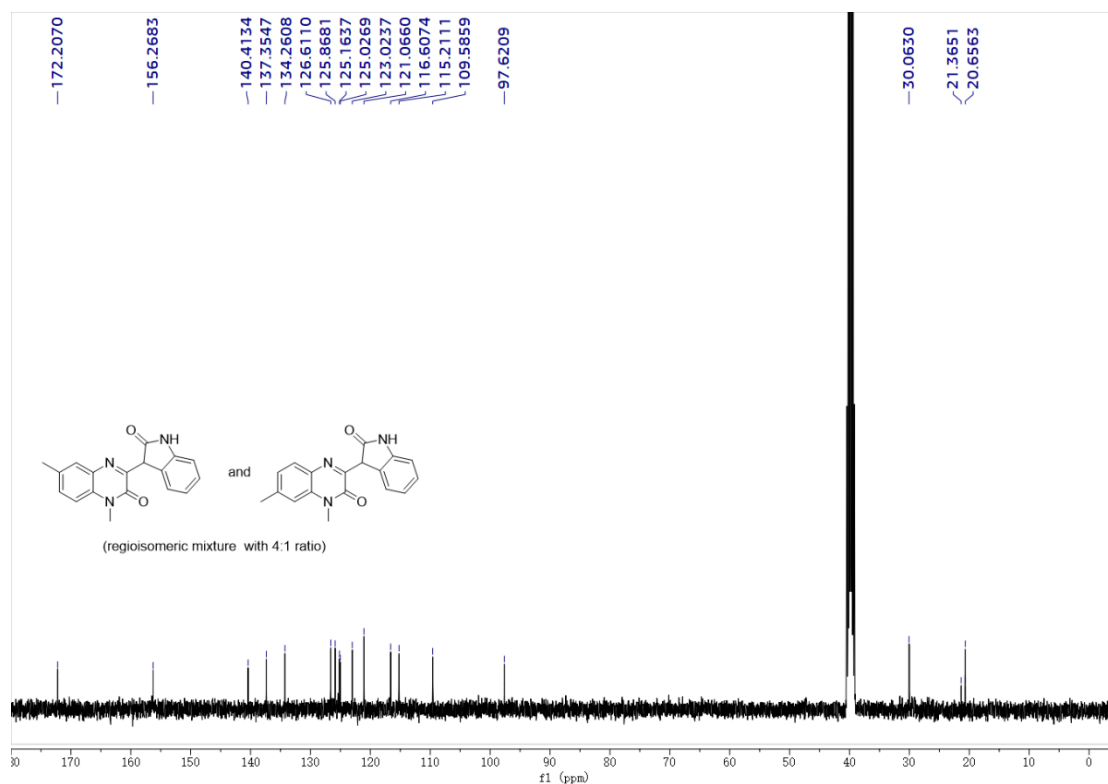


Figure S28 The <sup>13</sup>C NMR Spectrum of Compound **3ab** in DMSO-*d*<sub>6</sub>

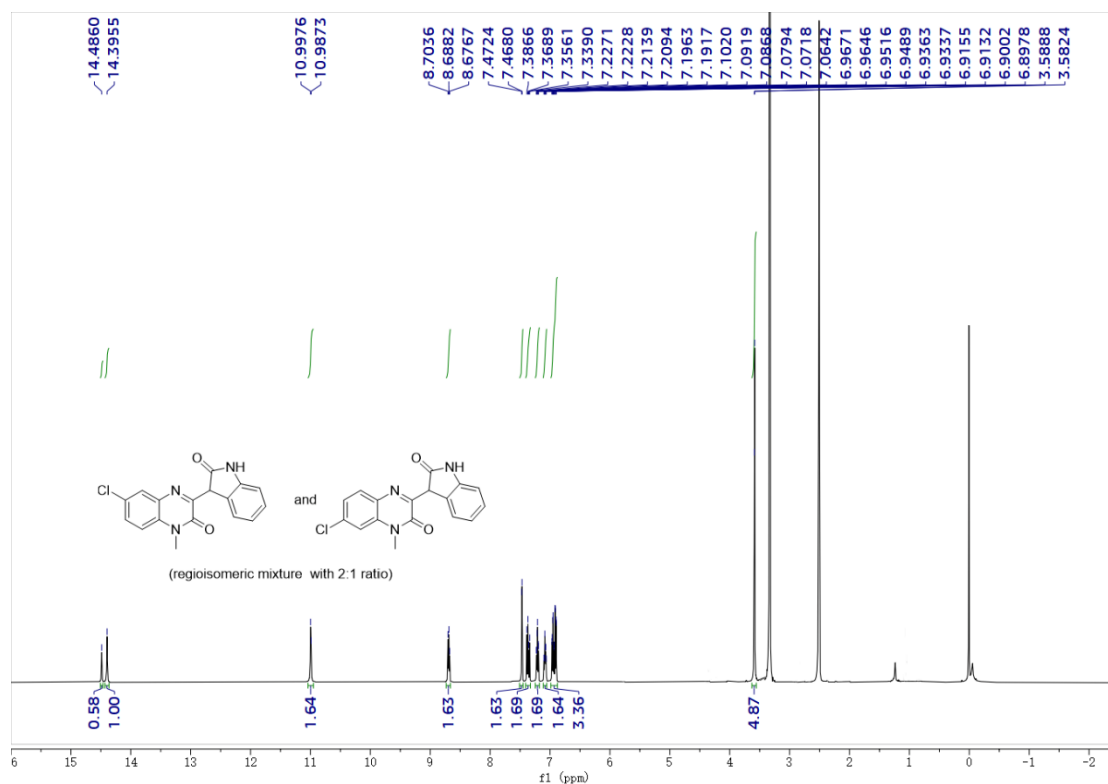


Figure S29 The <sup>1</sup>H NMR Spectrum of Compound 3ac in DMSO-*d*<sub>6</sub>

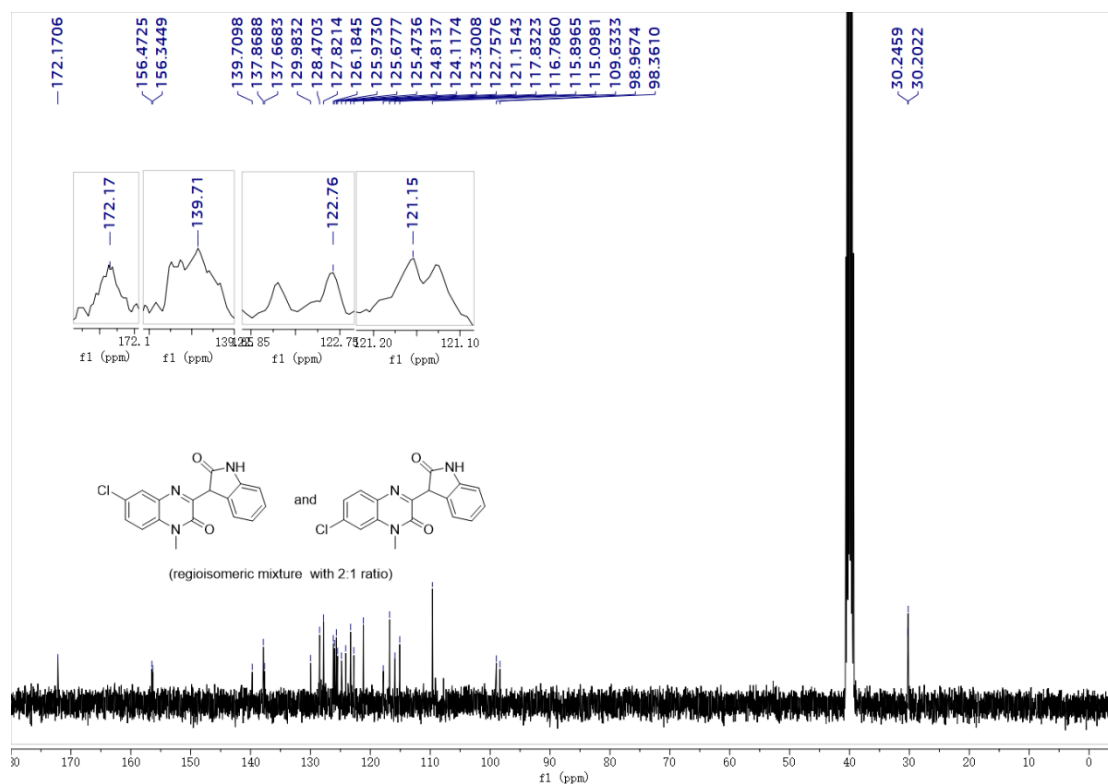


Figure S30 The <sup>13</sup>C NMR Spectrum of Compound 3ac in DMSO-*d*<sub>6</sub>

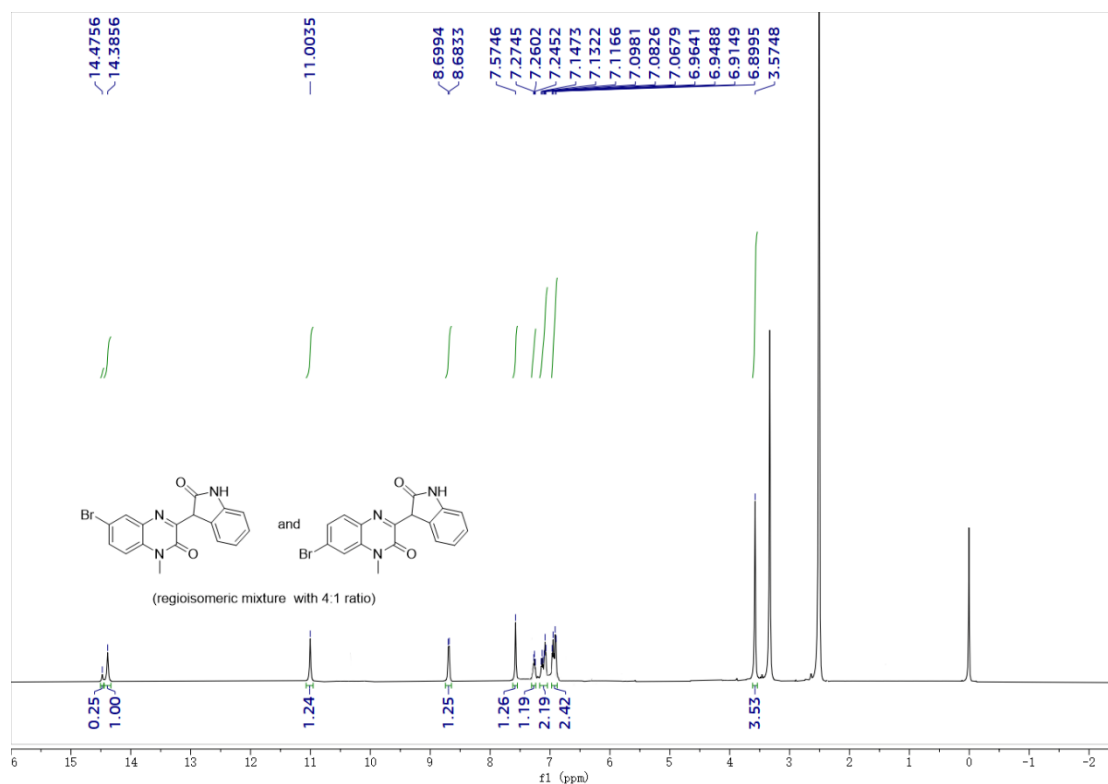


Figure S31 The <sup>1</sup>H NMR Spectrum of Compound 3ad in DMSO-*d*<sub>6</sub>

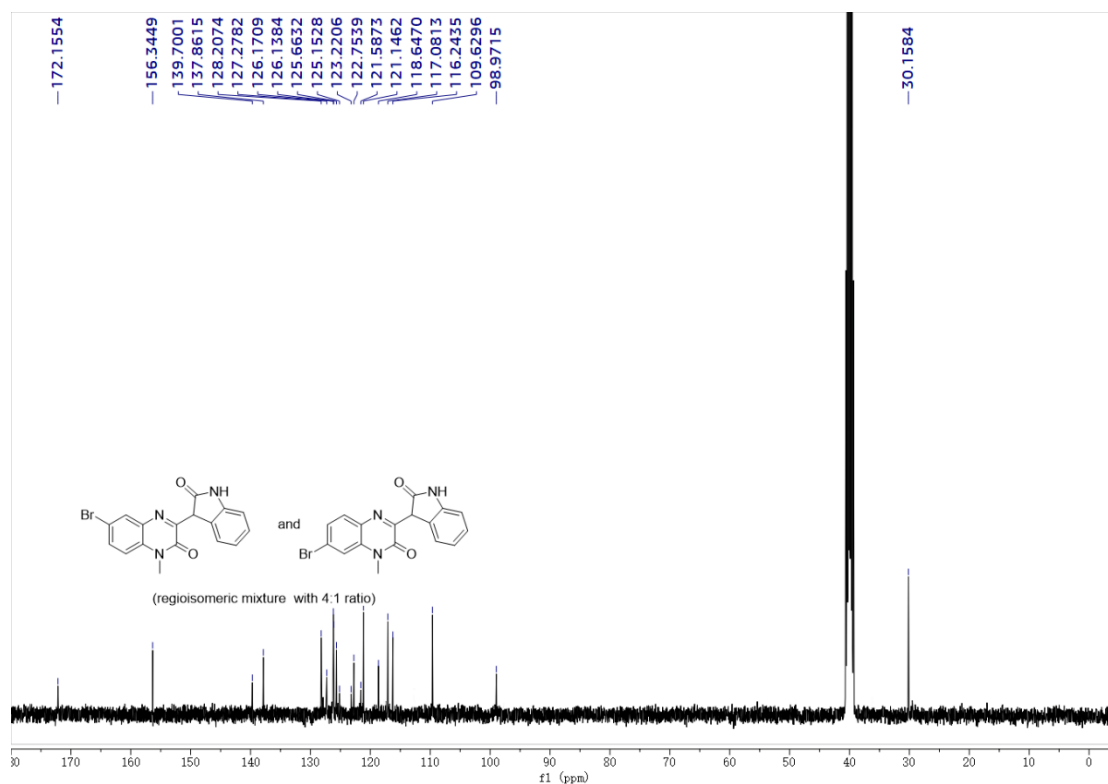


Figure S32 The <sup>13</sup>C NMR Spectrum of Compound 3ad in DMSO-*d*<sub>6</sub>

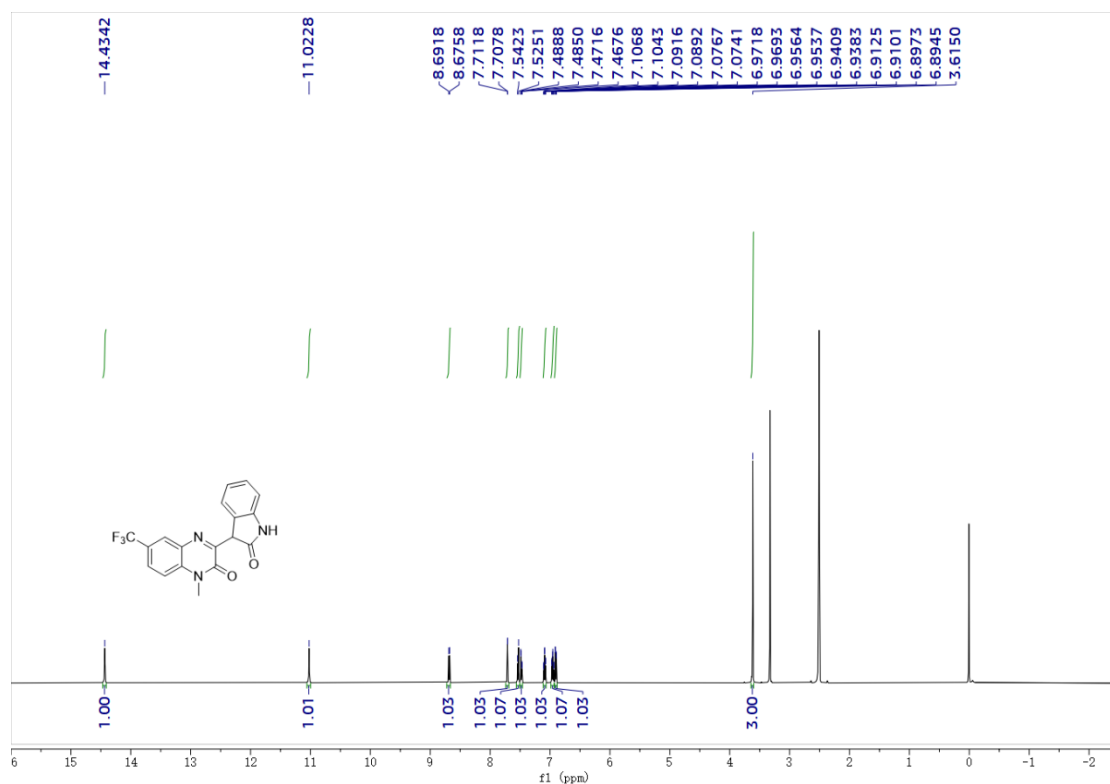


Figure S33 The  $^1\text{H}$  NMR Spectrum of Compound 3ae in  $\text{DMSO-}d_6$

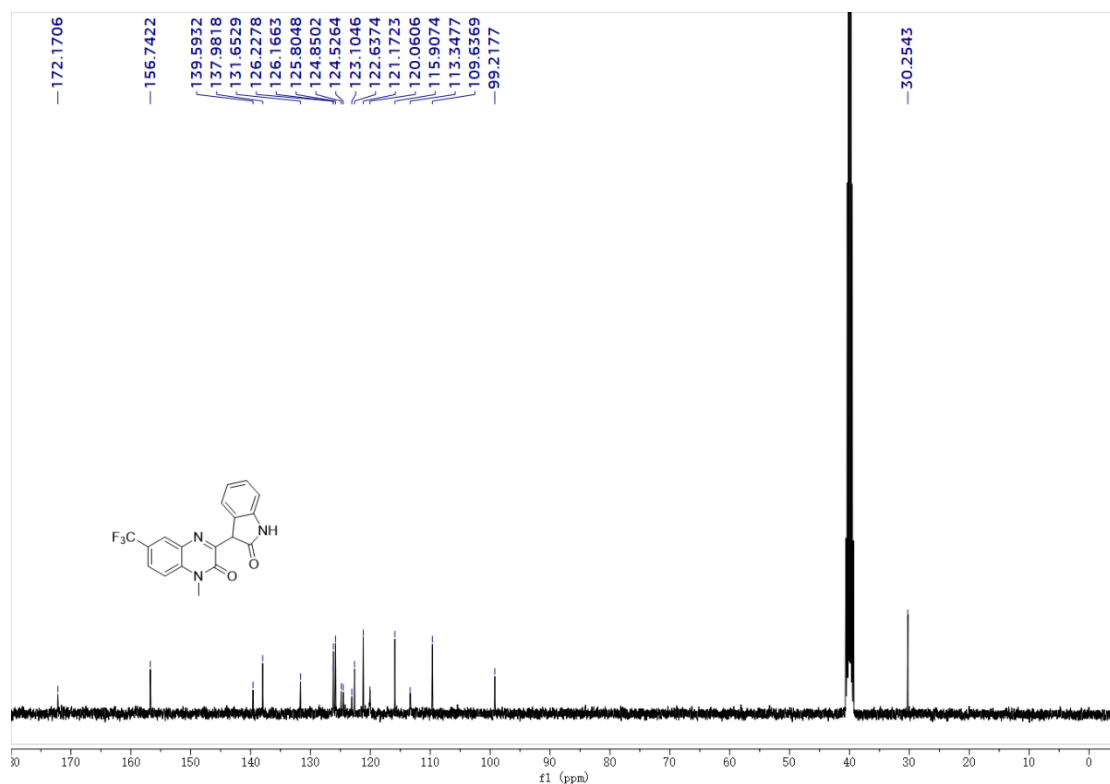


Figure S34 The  $^{13}\text{C}$  NMR Spectrum of Compound 3ae in  $\text{DMSO-}d_6$

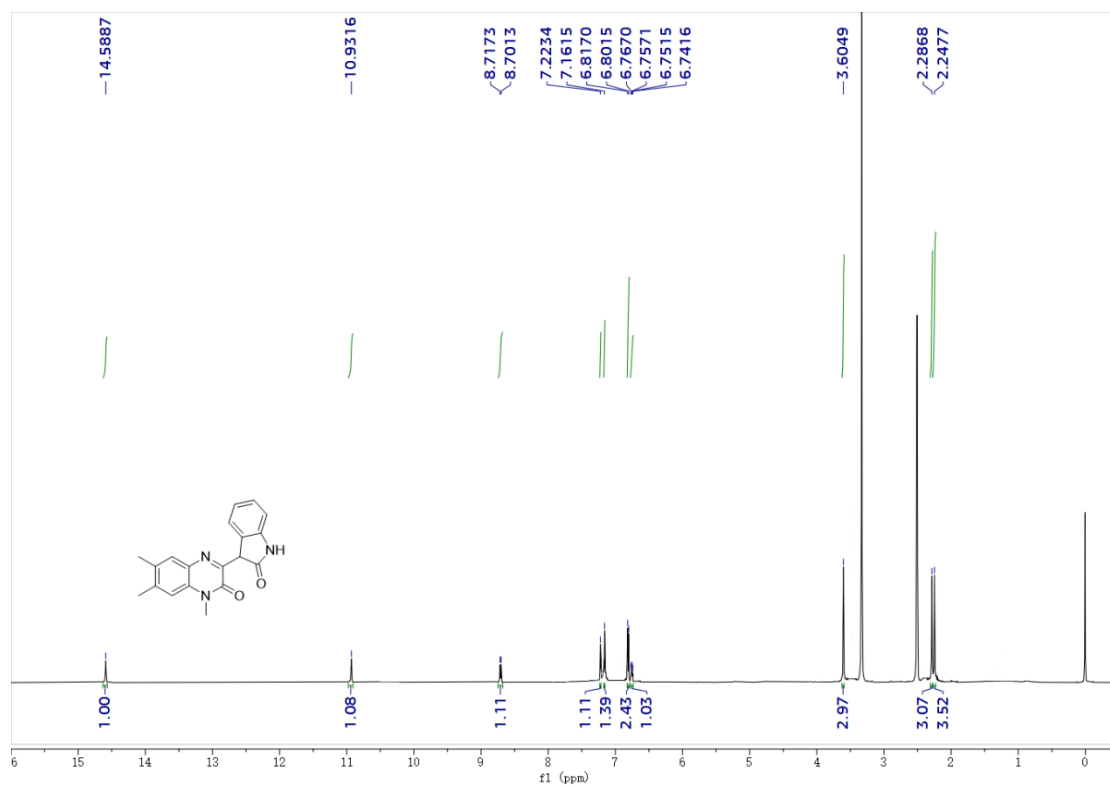


Figure S35 The  $^1\text{H}$  NMR Spectrum of Compound 3af in  $\text{DMSO-}d_6$

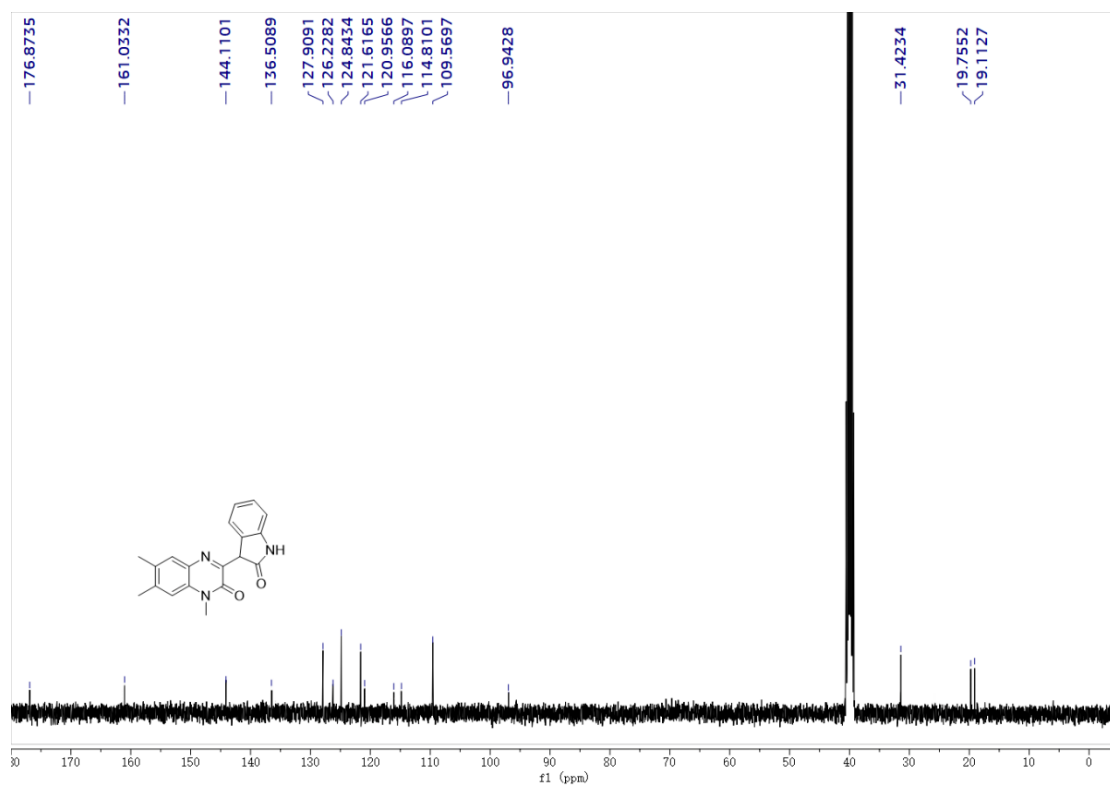


Figure S36 The  $^{13}\text{C}$  NMR Spectrum of Compound 3af in  $\text{DMSO-}d_6$

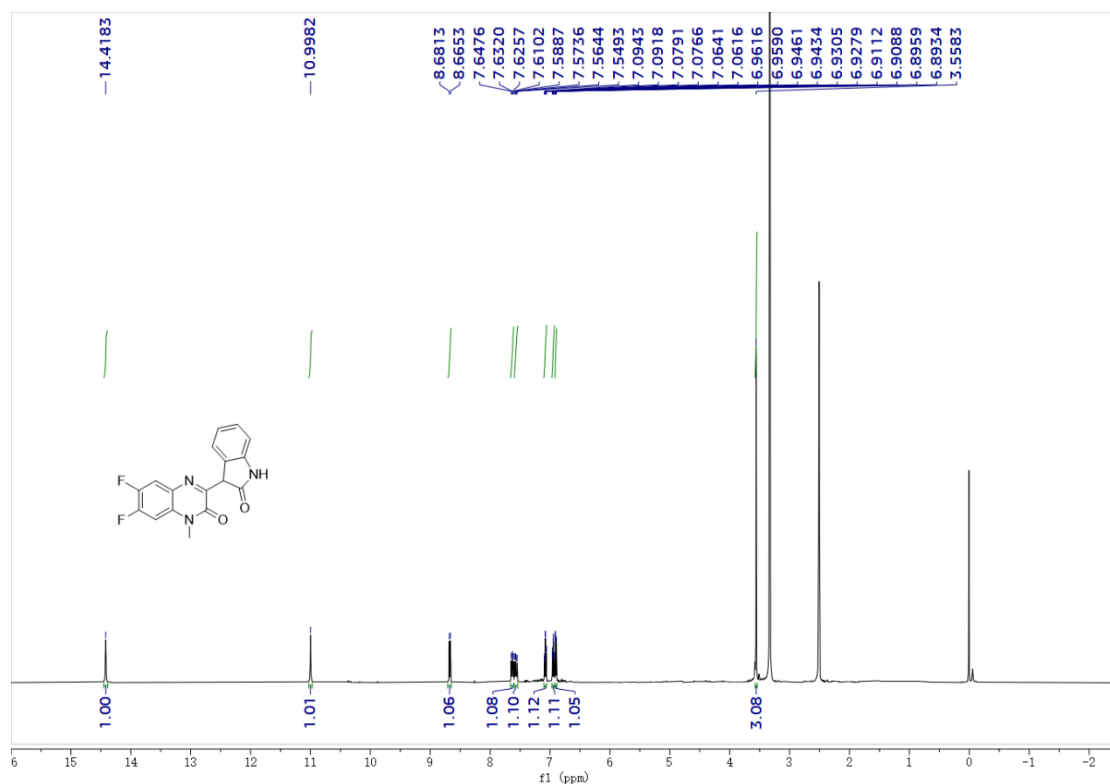


Figure S37 The  $^1\text{H}$  NMR Spectrum of Compound **3ag** in  $\text{DMSO-}d_6$

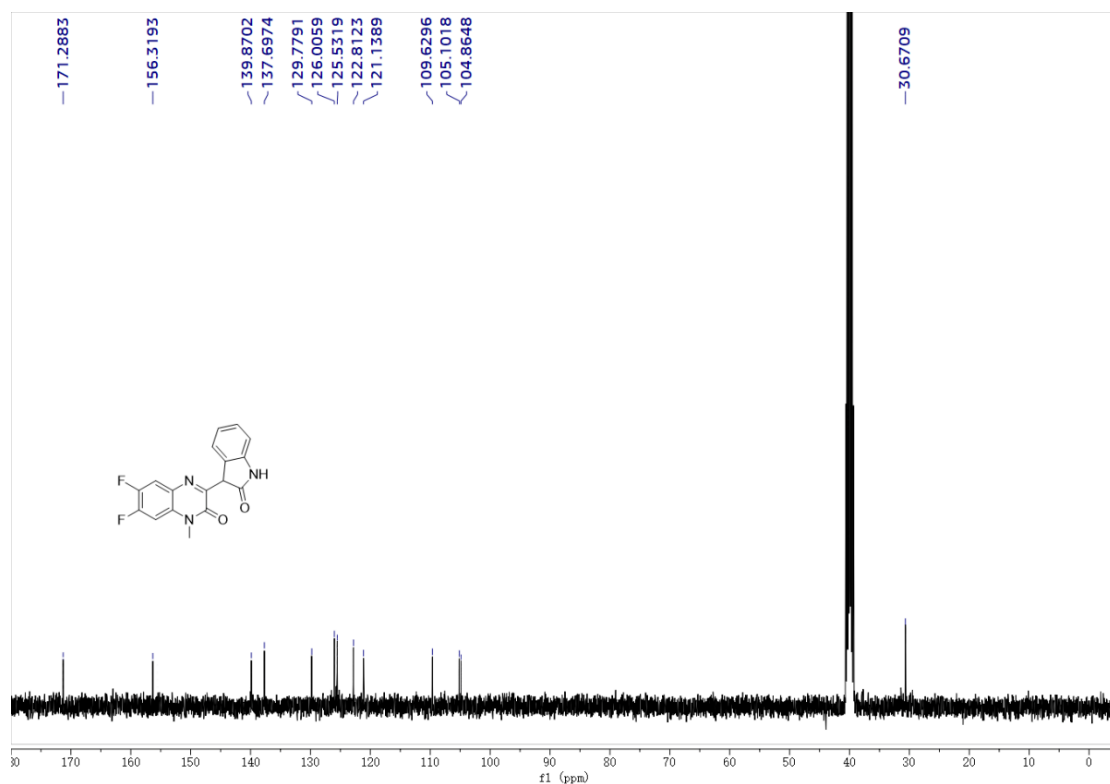


Figure S38 The  $^{13}\text{C}$  NMR Spectrum of Compound **3ag** in  $\text{DMSO-}d_6$



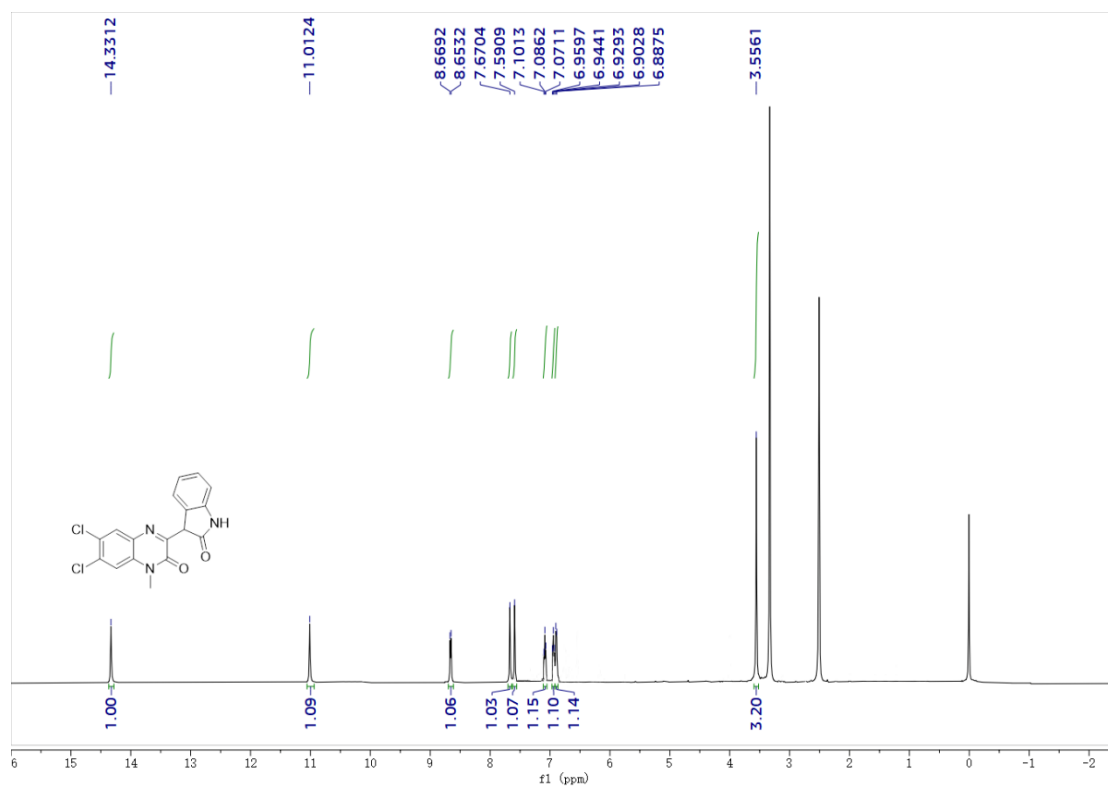


Figure S39 The  $^1\text{H}$  NMR Spectrum of Compound **3ah** in  $\text{DMSO-}d_6$

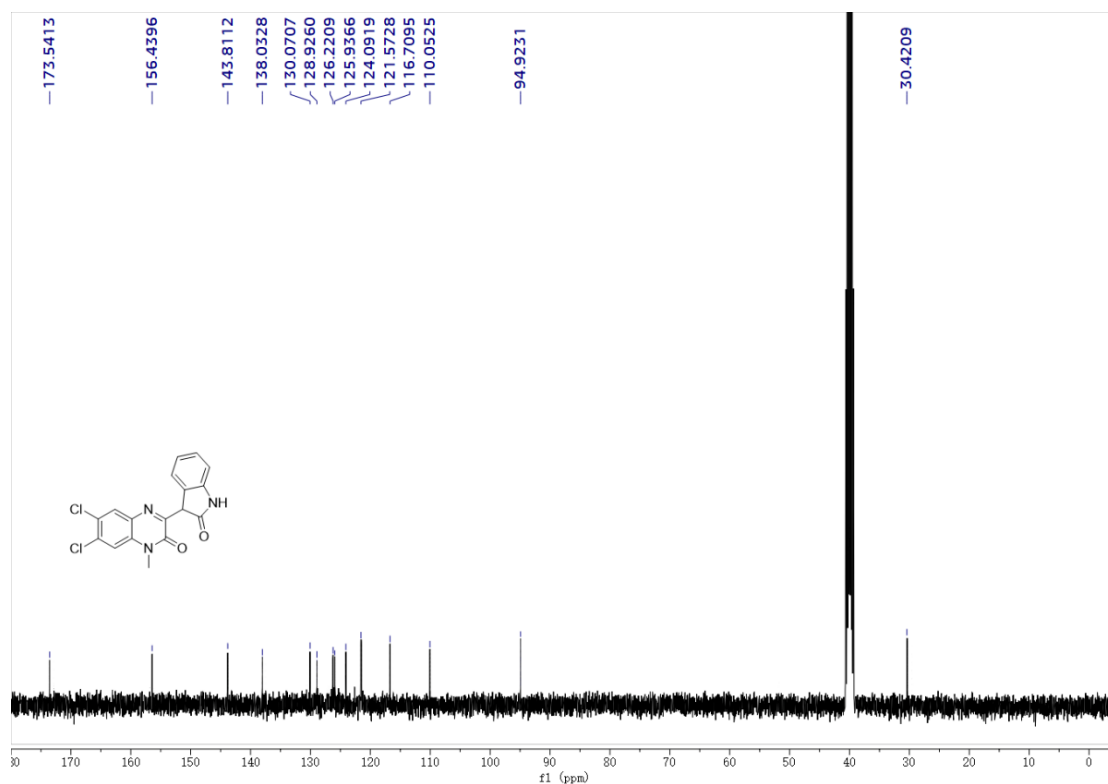


Figure S40 The  $^{13}\text{C}$  NMR Spectrum of Compound **3ah** in  $\text{DMSO-}d_6$

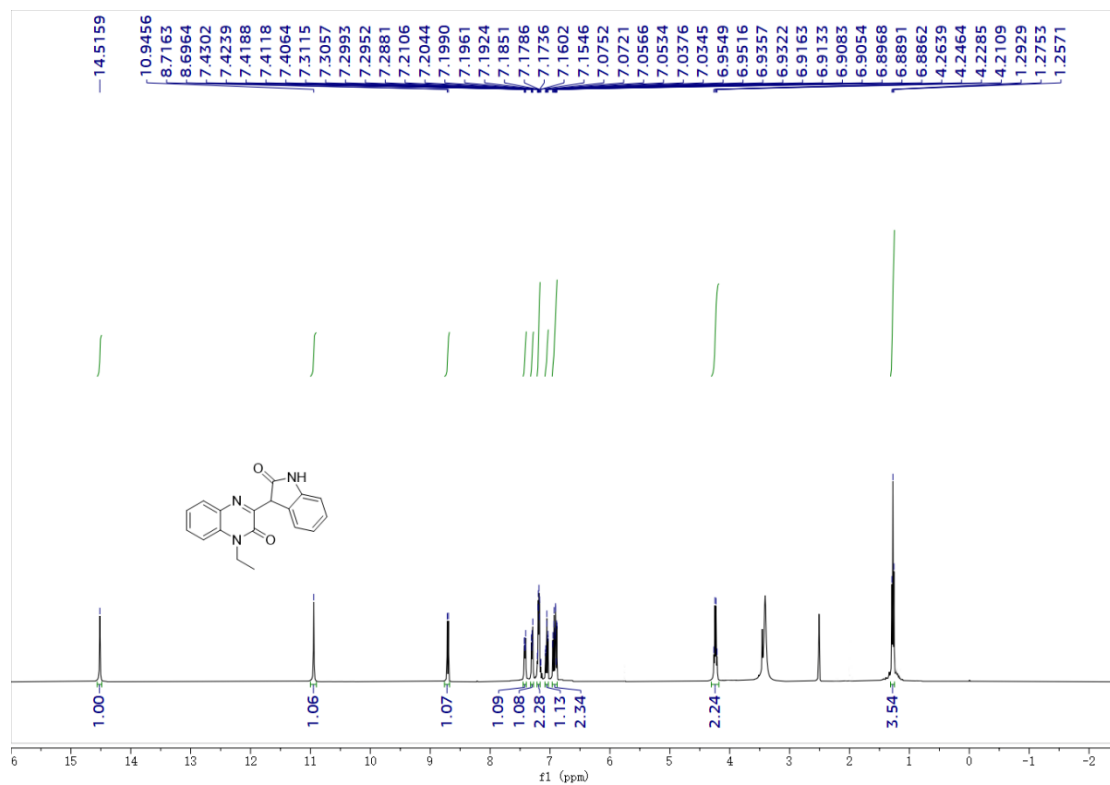


Figure S41 The  $^1\text{H}$  NMR Spectrum of Compound 3ai in  $\text{DMSO-}d_6$

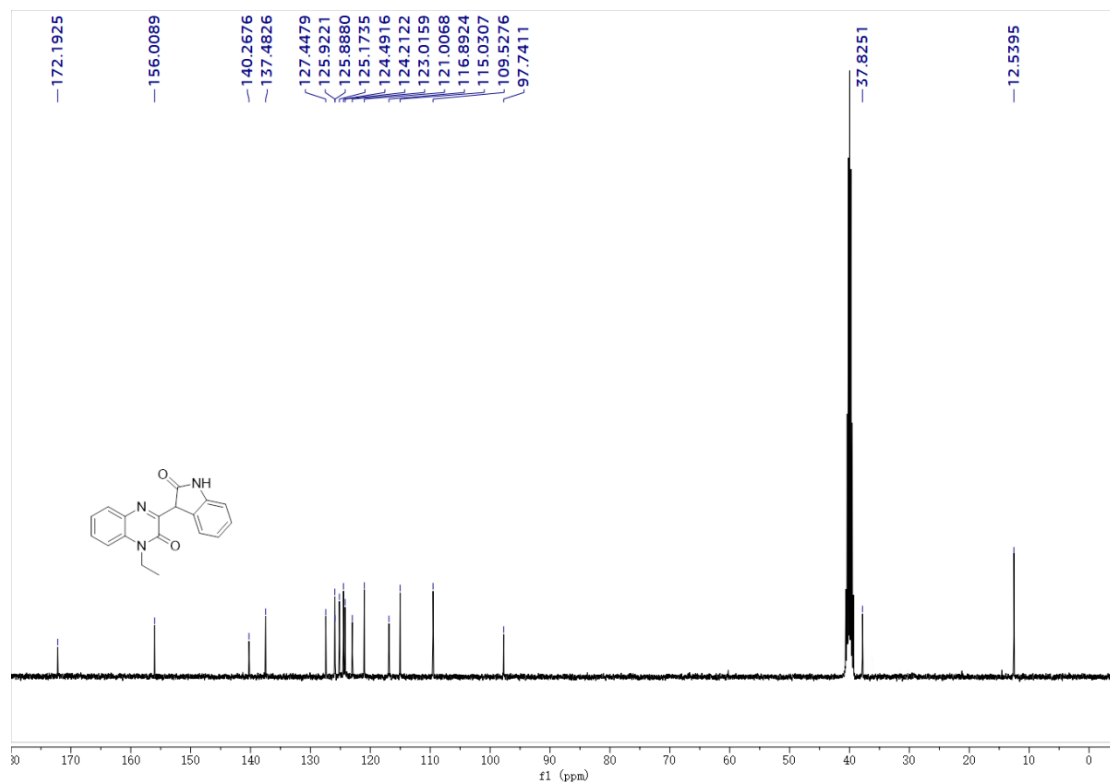


Figure S42 The  $^{13}\text{C}$  NMR Spectrum of Compound 3ai in  $\text{DMSO-}d_6$

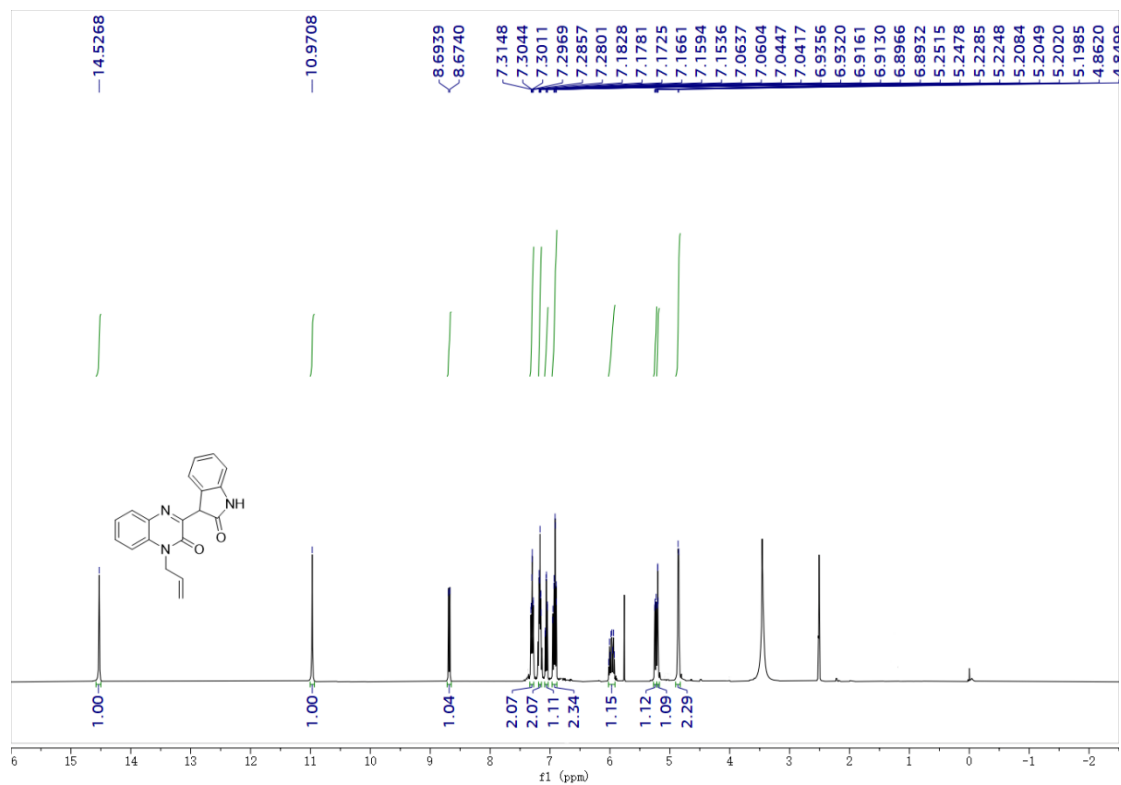


Figure S43 The  $^1\text{H}$  NMR Spectrum of Compound 3aj in  $\text{DMSO-}d_6$

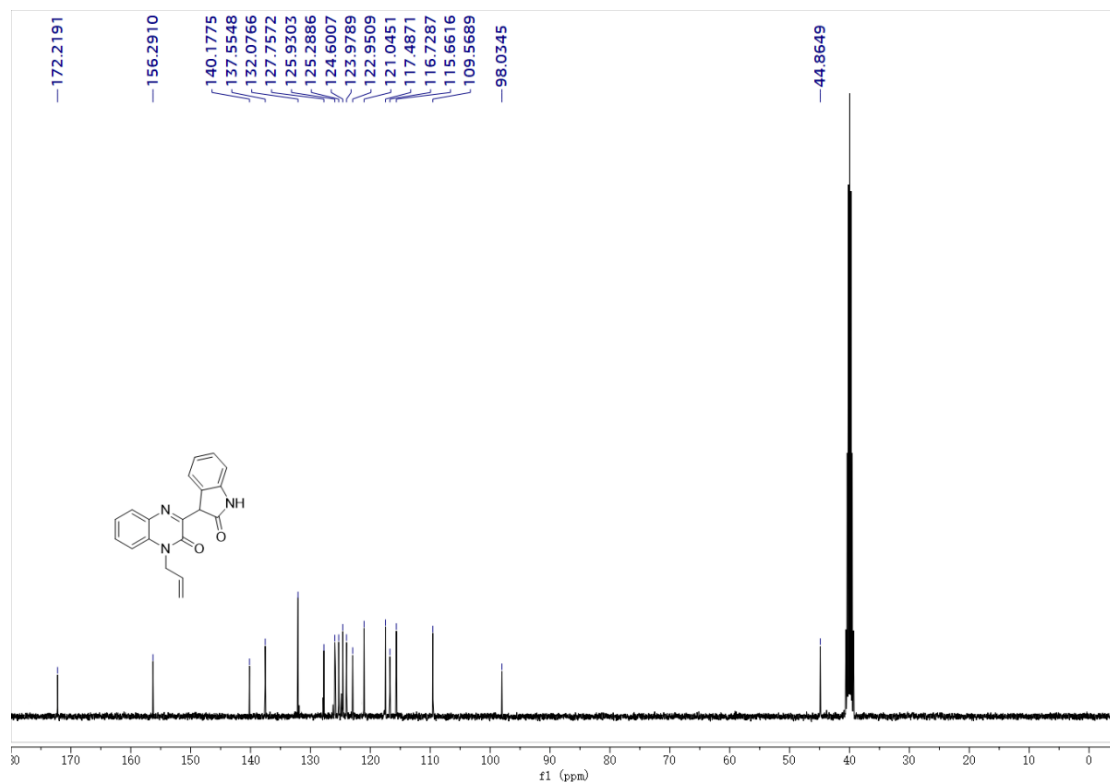


Figure S44 The  $^{13}\text{C}$  NMR Spectrum of Compound 3aj in  $\text{DMSO-}d_6$

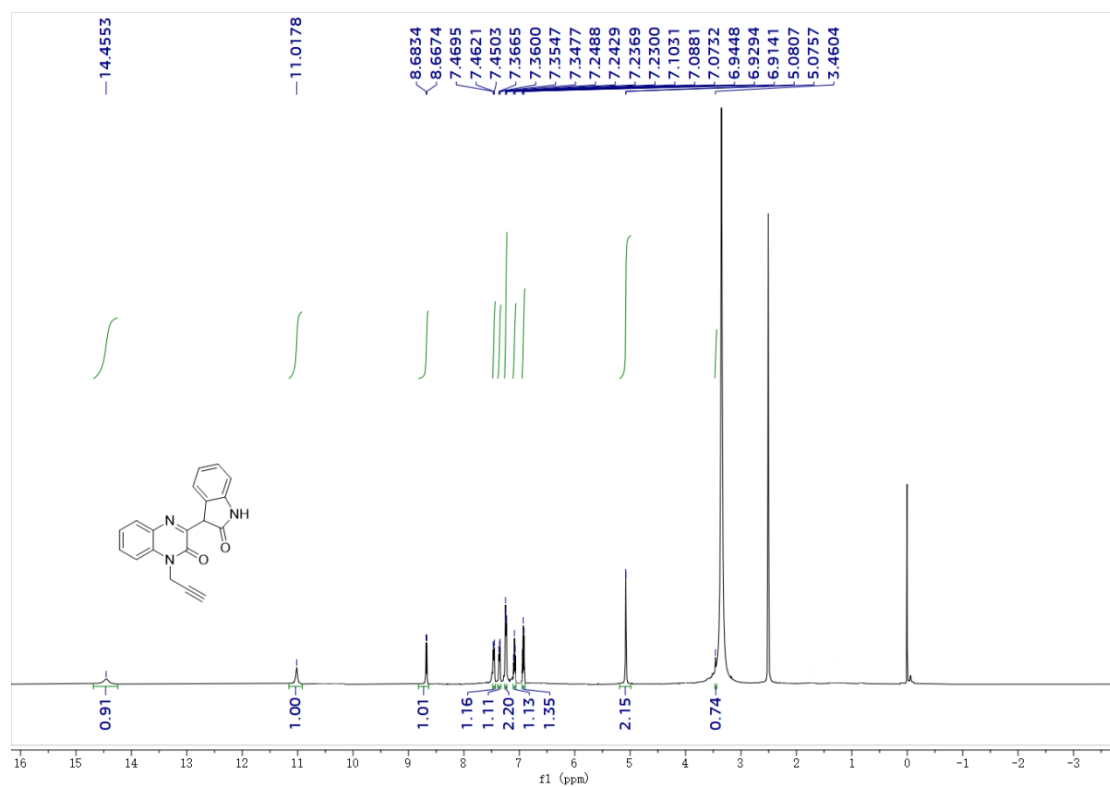


Figure S45 The  $^1\text{H}$  NMR Spectrum of Compound 3ak in  $\text{DMSO-}d_6$

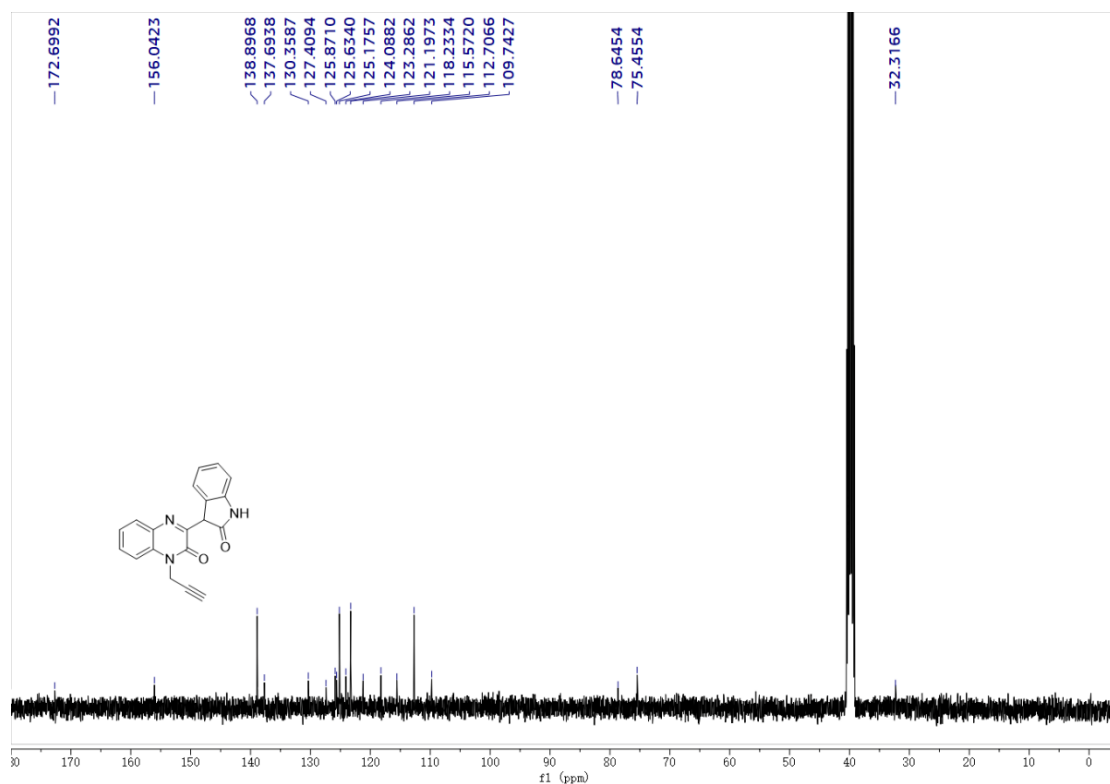


Figure S46 The  $^{13}\text{C}$  NMR Spectrum of Compound 3ak in  $\text{DMSO-}d_6$

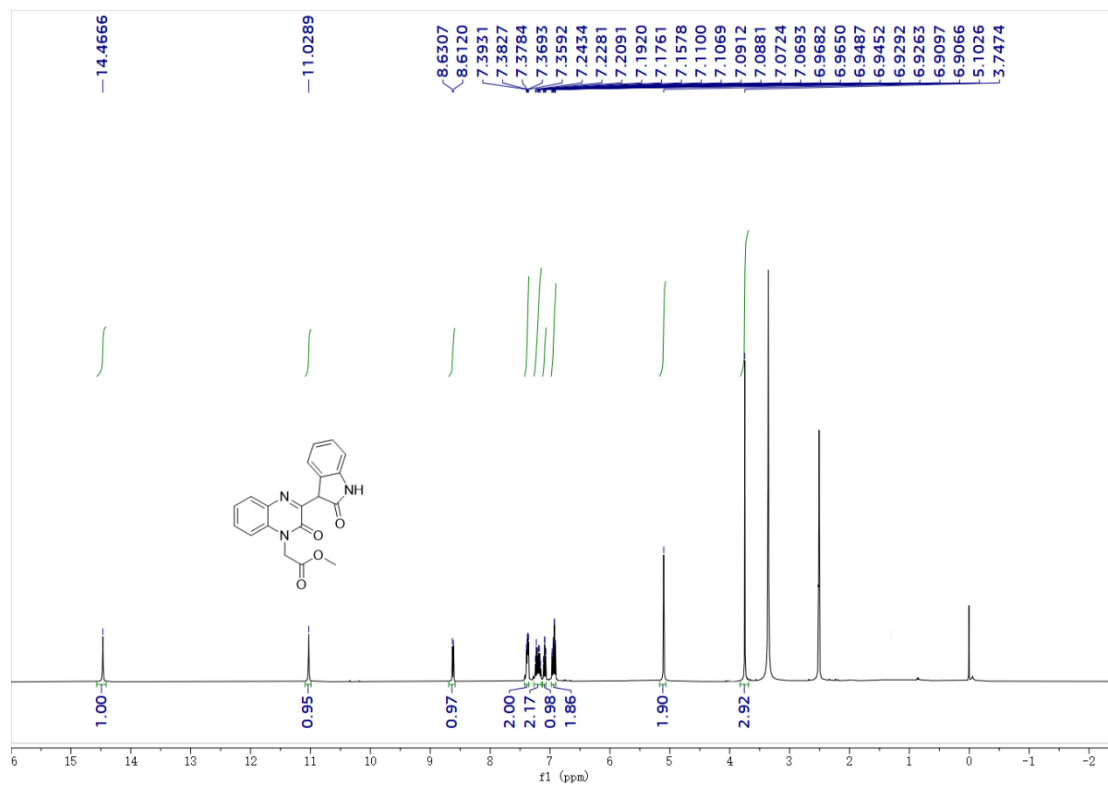


Figure S47 The  $^1\text{H}$  NMR Spectrum of Compound 3al in  $\text{DMSO-}d_6$

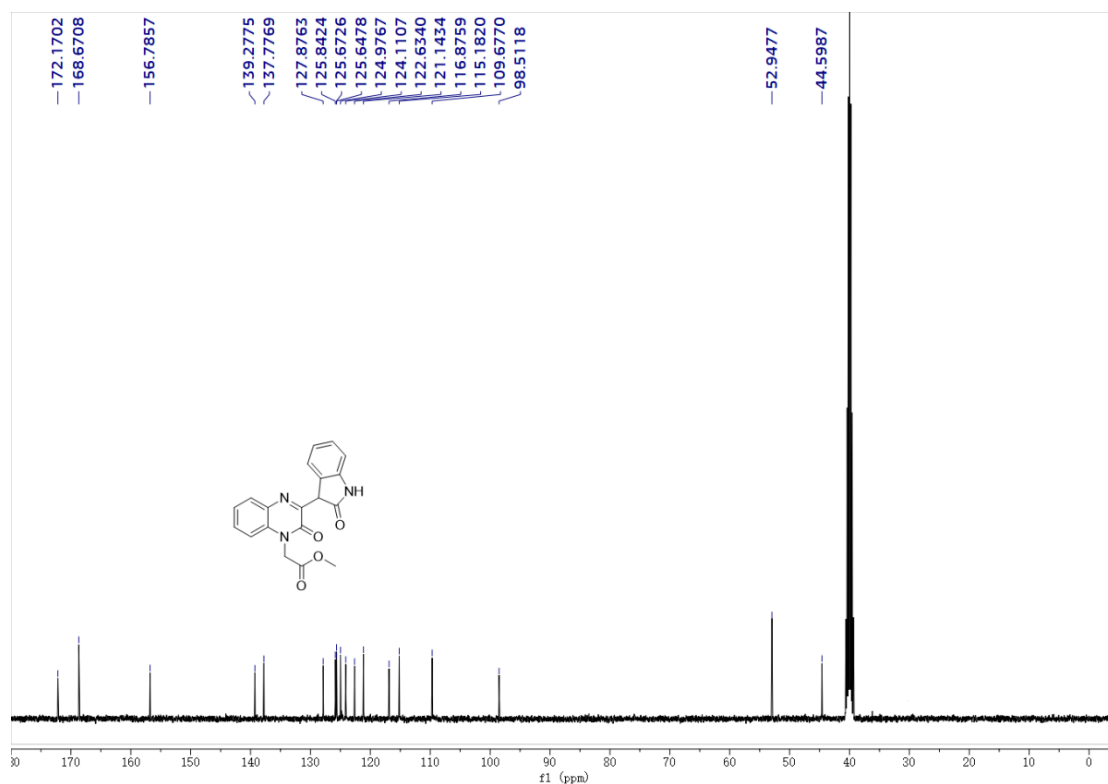


Figure S48 The  $^{13}\text{C}$  NMR Spectrum of Compound 3al in  $\text{DMSO-}d_6$

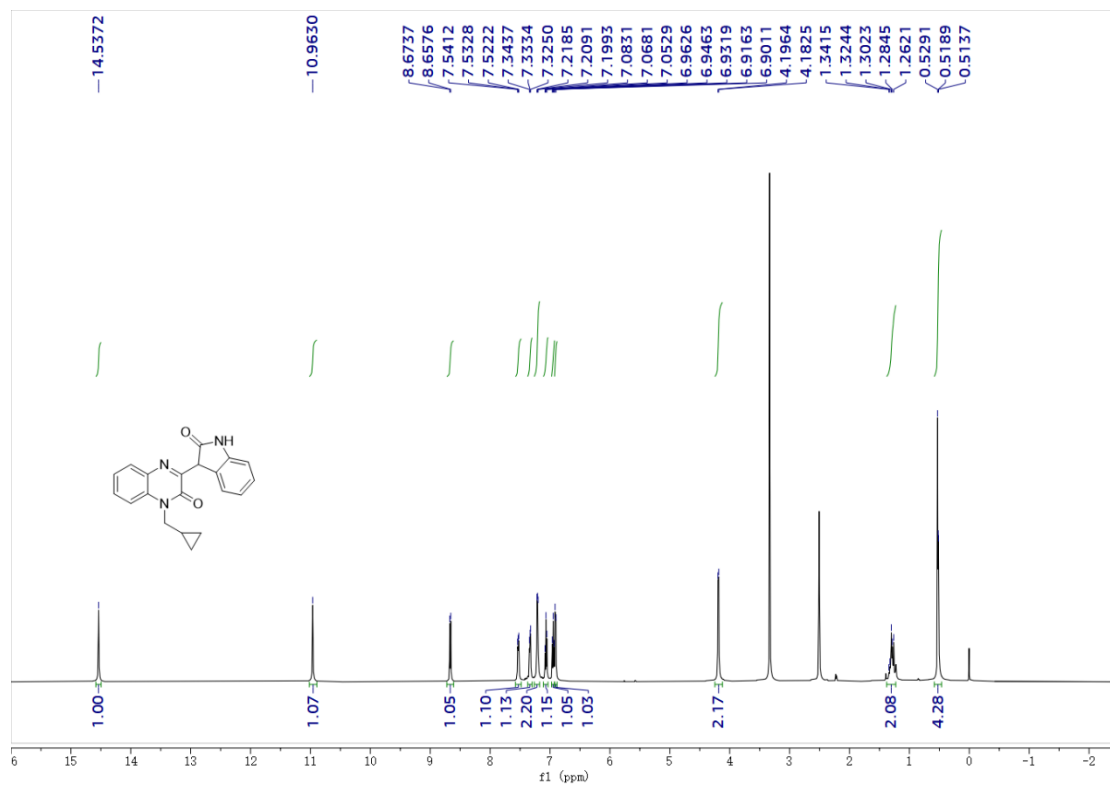


Figure S49 The <sup>1</sup>H NMR Spectrum of Compound 3am in DMSO-*d*<sub>6</sub>

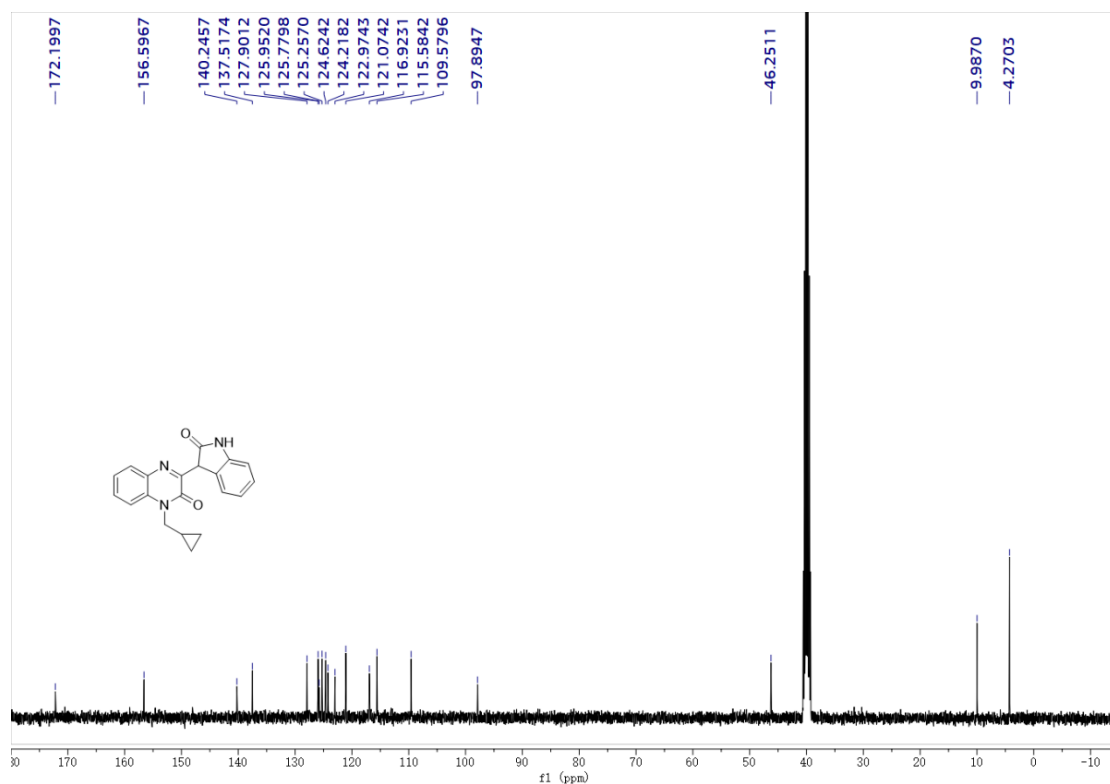


Figure S50 The <sup>13</sup>C NMR Spectrum of Compound 3am in DMSO-*d*<sub>6</sub>

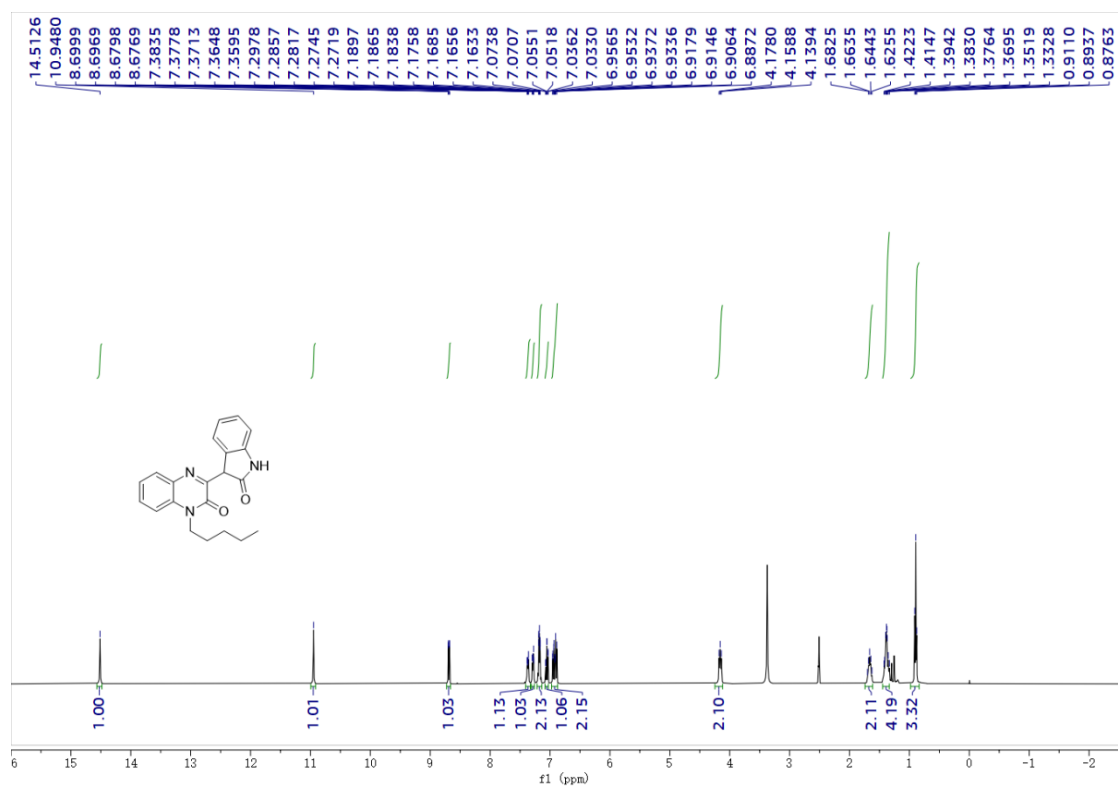


Figure S51 The  $^1\text{H}$  NMR Spectrum of Compound 3an in  $\text{DMSO-}d_6$

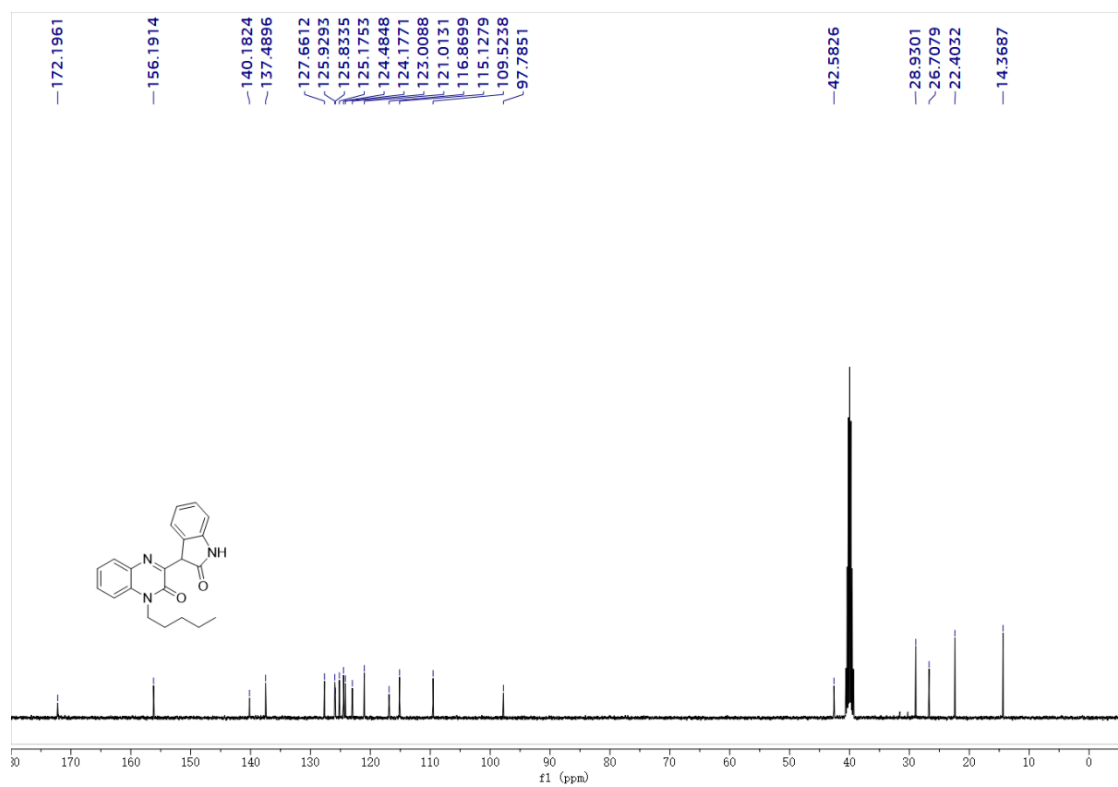


Figure S52 The  $^{13}\text{C}$  NMR Spectrum of Compound 3an in  $\text{DMSO-}d_6$

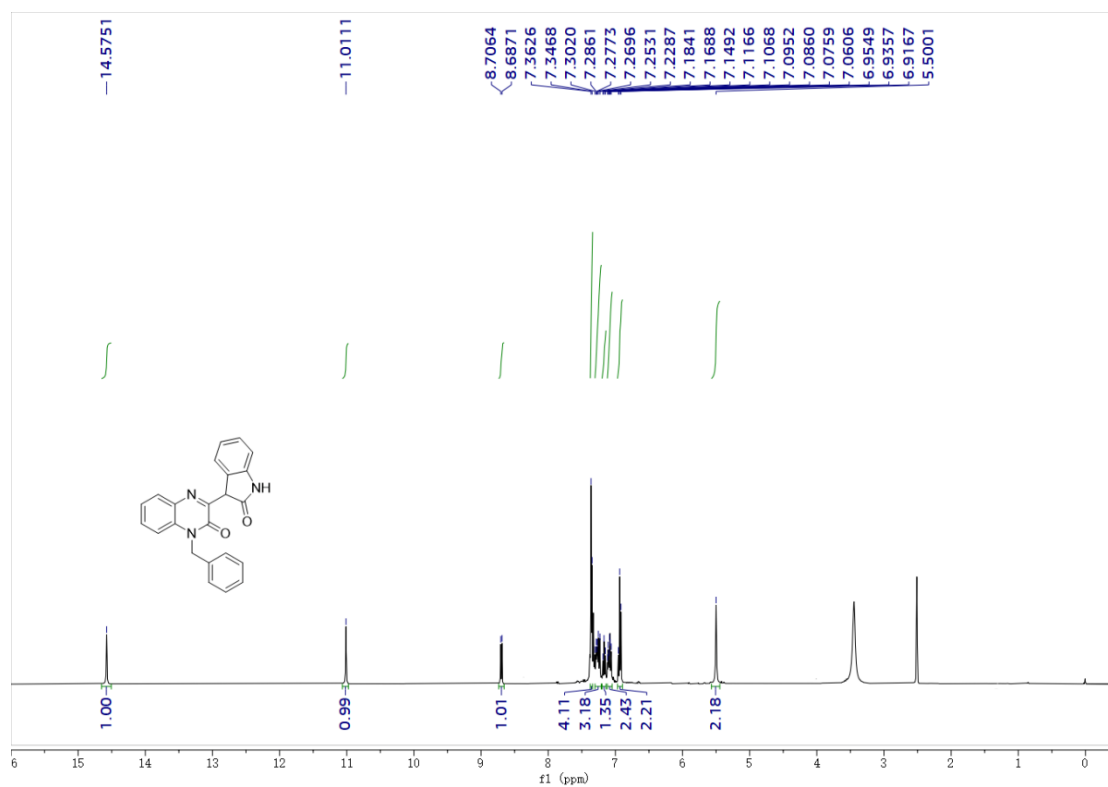


Figure S53 The  $^1\text{H}$  NMR Spectrum of Compound 3ao in  $\text{DMSO-}d_6$

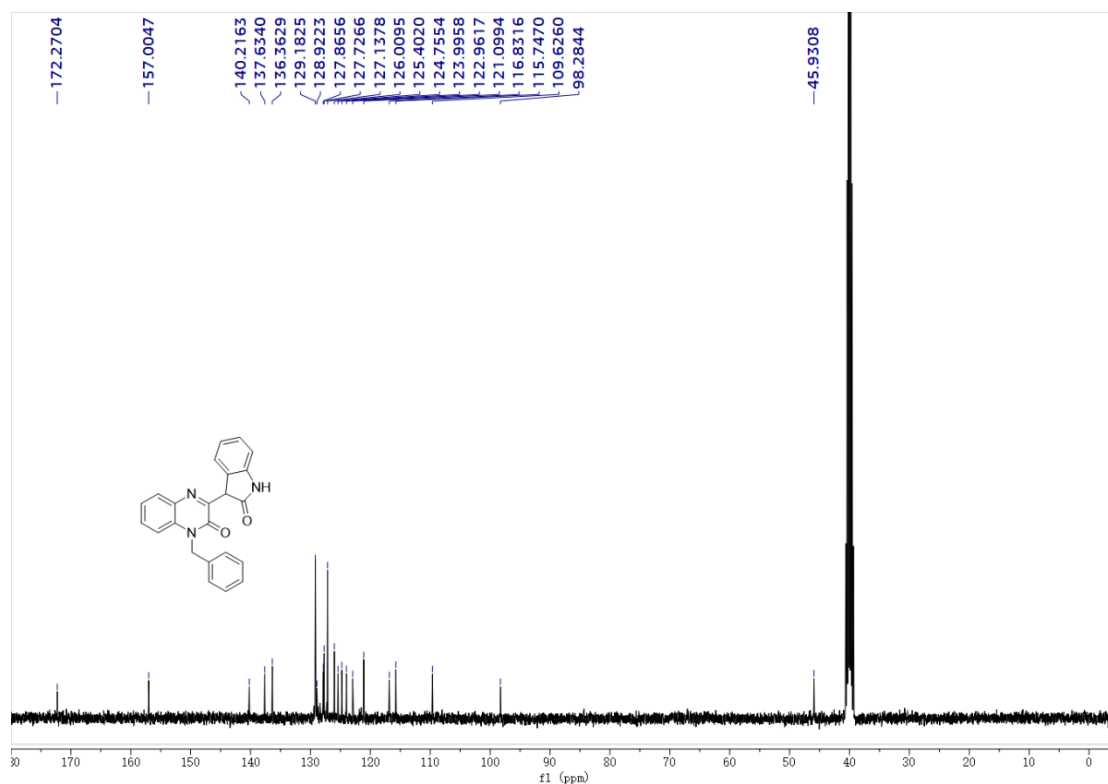


Figure S54 The  $^{13}\text{C}$  NMR Spectrum of Compound 3ao in  $\text{DMSO-}d_6$



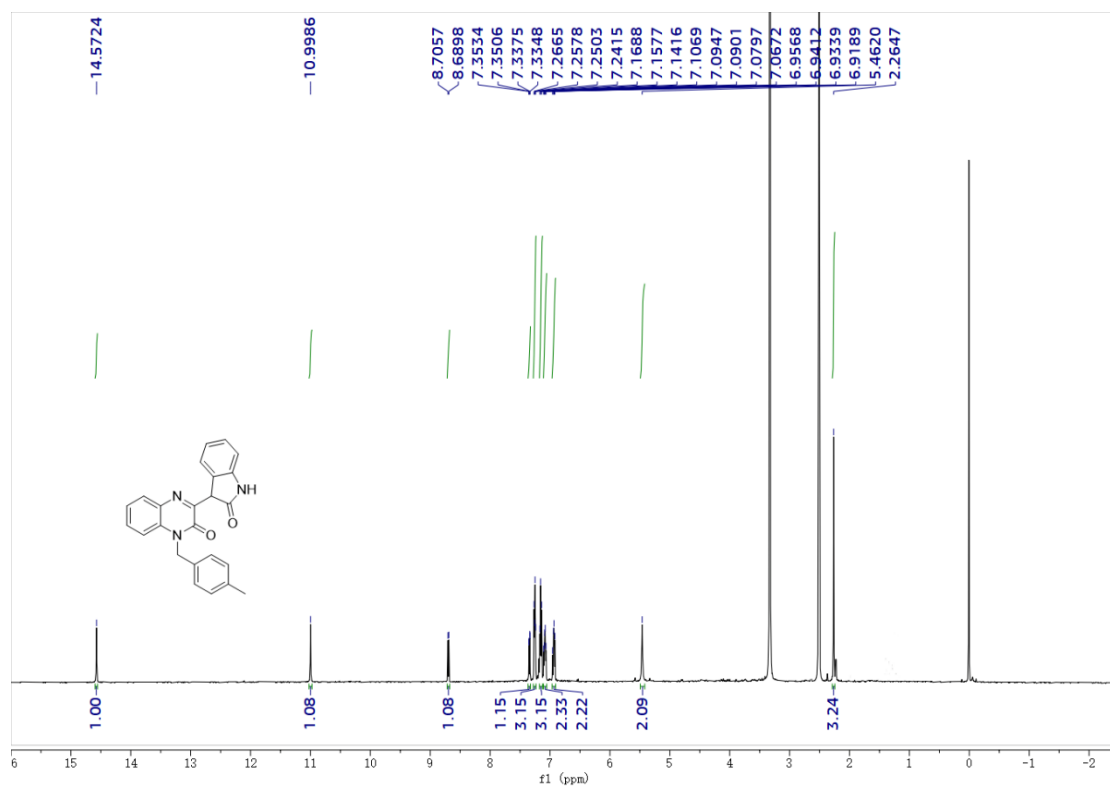


Figure S55 The  $^1\text{H}$  NMR Spectrum of Compound **3ap** in  $\text{DMSO-}d_6$

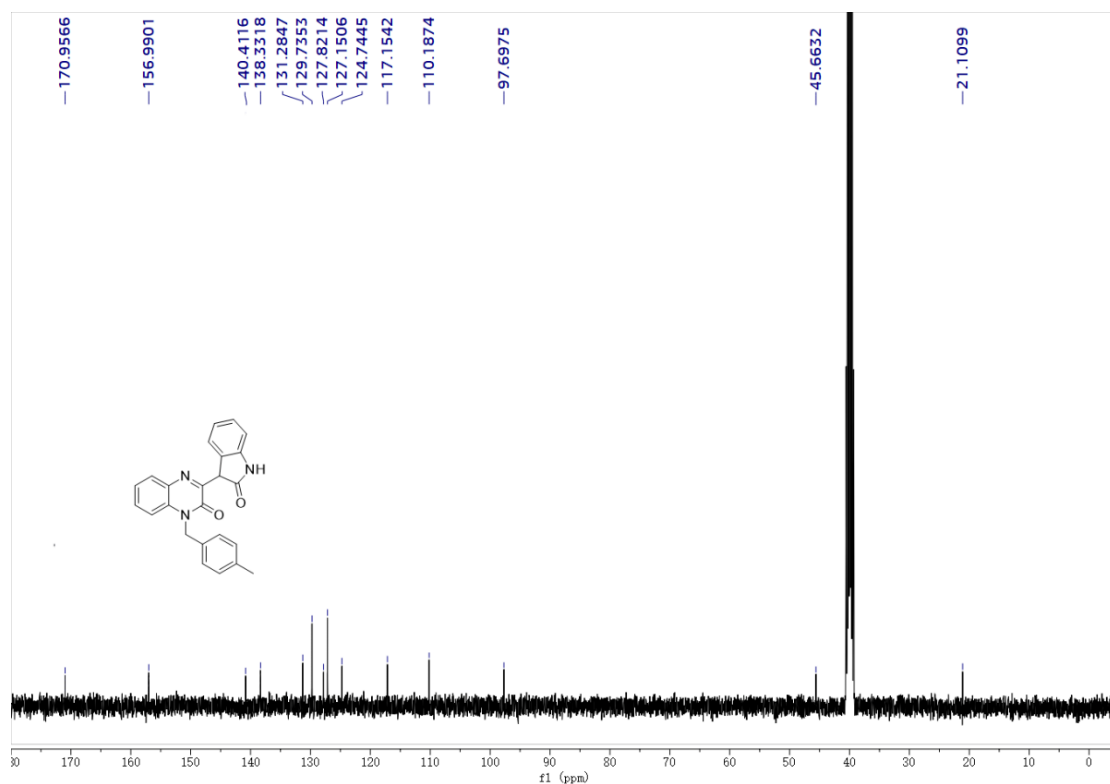


Figure S56 The  $^{13}\text{C}$  NMR Spectrum of Compound **3ap** in  $\text{DMSO-}d_6$

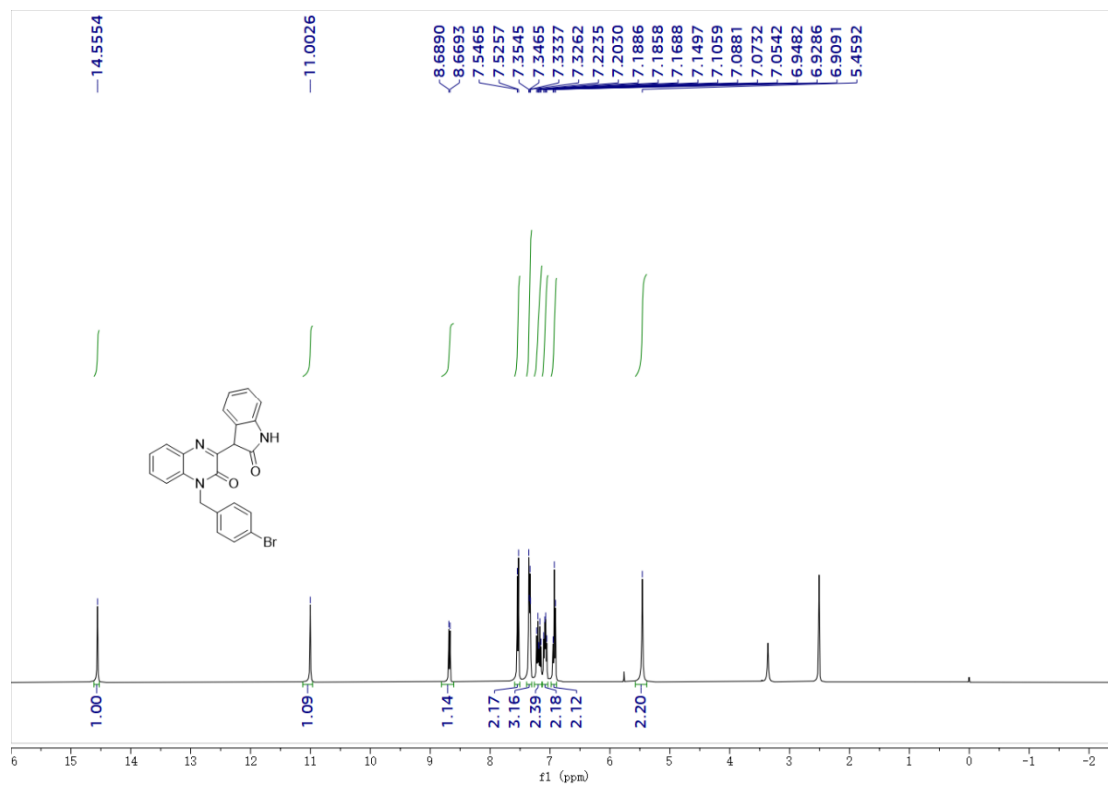


Figure S57 The  $^1\text{H}$  NMR Spectrum of Compound **3aq** in  $\text{DMSO-}d_6$

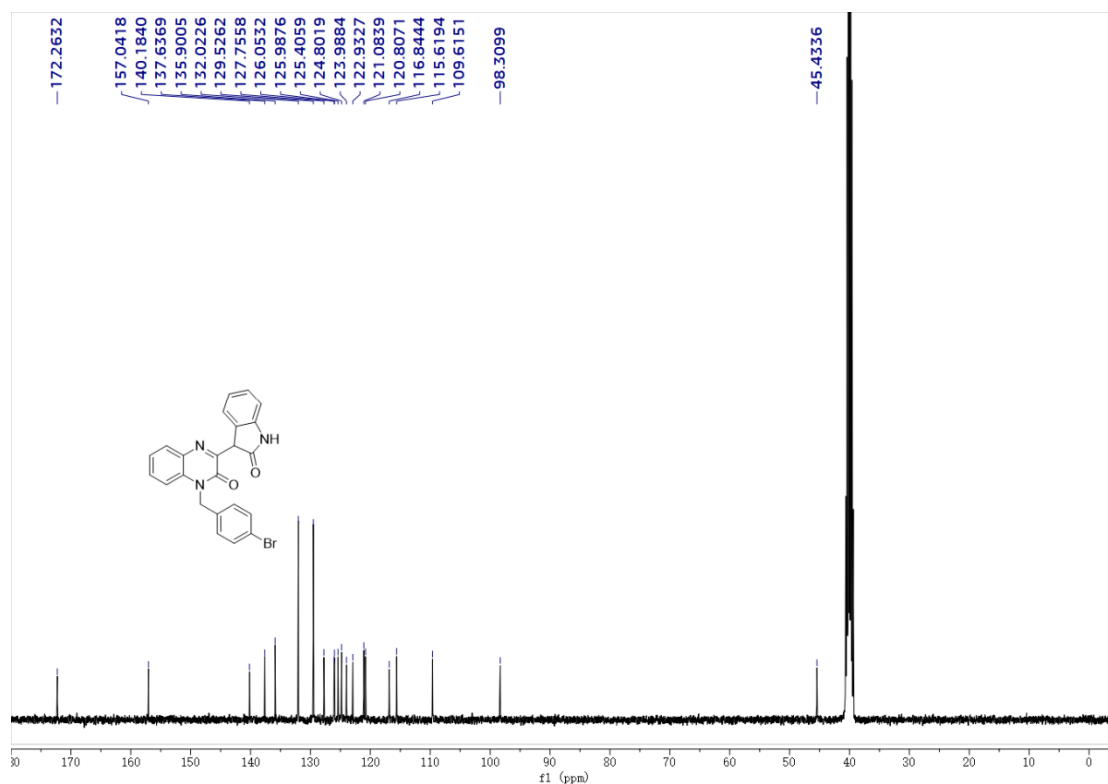


Figure S58 The  $^{13}\text{C}$  NMR Spectrum of Compound **3aq** in  $\text{DMSO-}d_6$

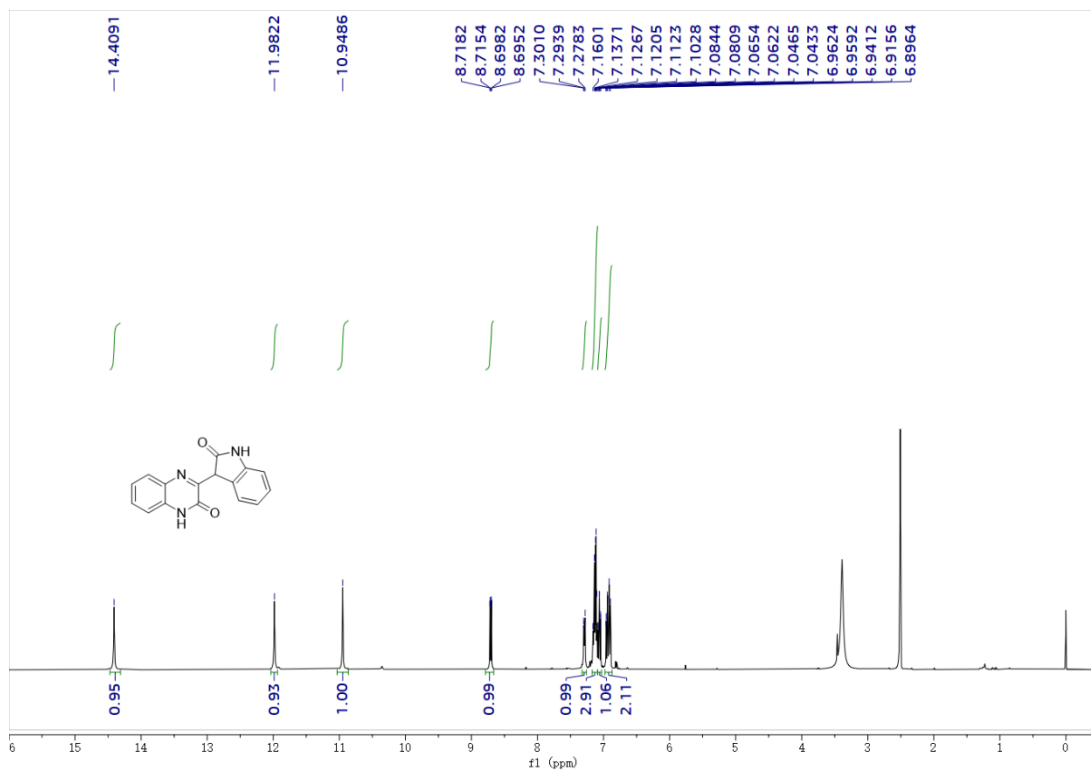


Figure S59 The  $^1\text{H}$  NMR Spectrum of Compound 3ar in  $\text{DMSO-}d_6$

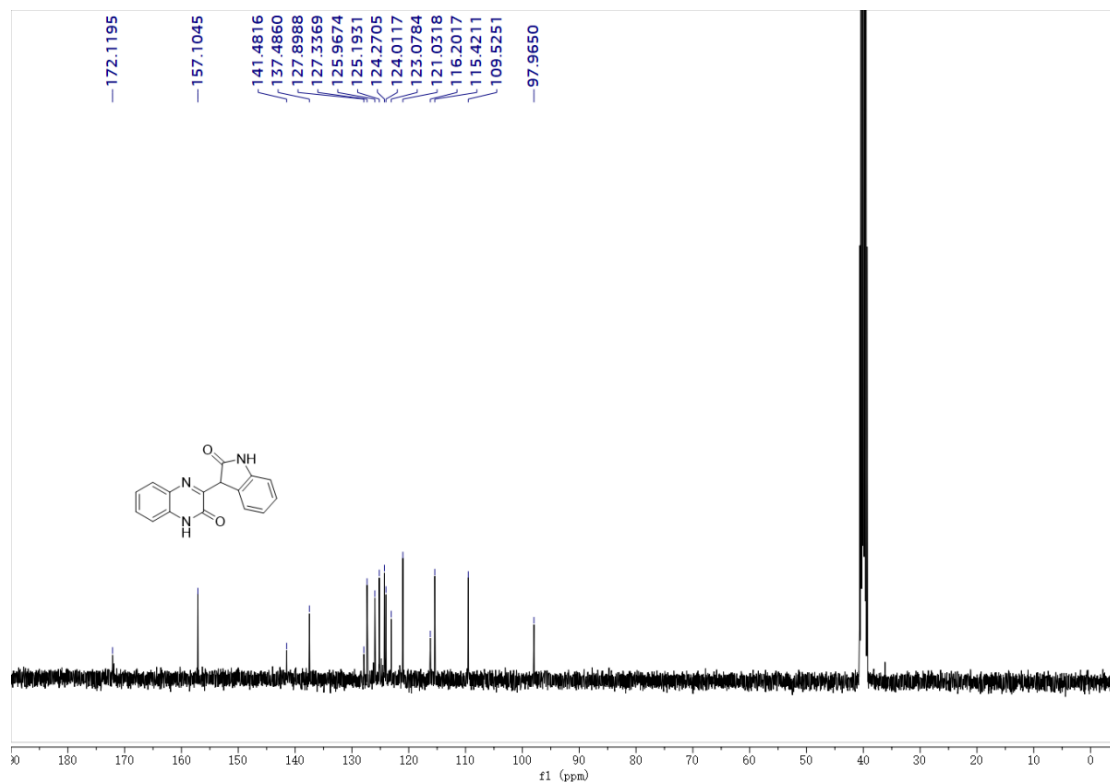


Figure S60 The  $^{13}\text{C}$  NMR Spectrum of Compound 3ar in  $\text{DMSO-}d_6$