

Supporting Information

Selective sulfonylation and diazotization of indoles

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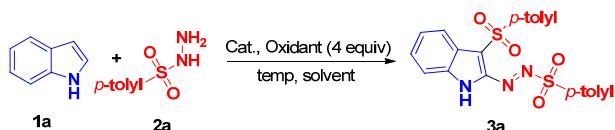
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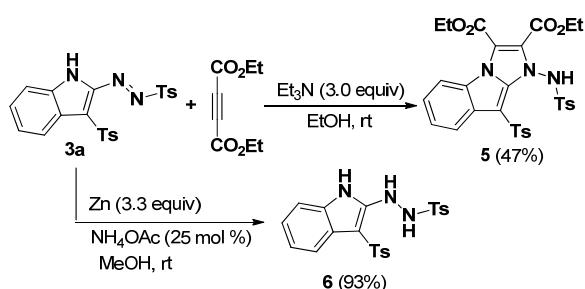
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Table 1 Optimization conditions for the synthesis of **3a**



Entry	Oxidant	Cat. (mol %)	Solvent	<i>t</i> (°C)	Yield (%) ^a
1	TBHP	TBAI (30)	CH ₃ CN	rt	81
2	TBHP	TBAI (30)	DMSO	rt	ND ^b
3	TBHP	TBAI (30)	EtOH	rt	37
4	TBHP	TBAI (30)	MeOH	rt	58
5	TBHP	TBAI (30)	DCE	rt	64
6	TBHP	TBAI (30)	toluene	rt	57
7	TBHP	TBAI (30)	CH ₃ CN	40	87
8	TBHP	TBAI (30)	CH ₃ CN	60	46
9	TBHP	TBAI (50)	CH ₃ CN	40	86
10	TBHP	TBAI (10)	CH ₃ CN	40	65
11	TBHP	I ₂ (30)	CH ₃ CN	40	84
12	TBHP	CuI (30)	CH ₃ CN	40	ND ^b
13	TBHP	CuBr (30)	CH ₃ CN	40	ND ^b
14	TBHP	-	CH ₃ CN	40	ND ^b
15	BPO	TBAI (30)	CH ₃ CN	40	60
16	TBPB	TBAI (30)	CH ₃ CN	40	42
17	H ₂ O ₂	TBAI (30)	CH ₃ CN	40	80
18	DTBP	TBAI (30)	CH ₃ CN	40	67
19	m-CPBA	TBAI (30)	CH ₃ CN	40	47

^a Isolated yield. ^b Not detected (ND).



Scheme 4 The application of 3-sulfonyl-2-sulfonyldiazenyl-1*H*-indoles **3a**

Experimental

General information

Melting points were determined in open capillaries. IR spectra were taken on a FT-IR-Tensor 27 spectrometer. ^1H NMR spectra were measured on a Bruker DPX 400 MHz spectrometer. HRMS (ESI) was determined by using microTOF-Q HRMS/MS instrument II (BRUKER).

General Procedure for the Synthesis of 3a-3u

Typical Procedure for the Preparation of 3a:

The substrate indole (**1a**, 1 mmol, 0.117 g), *p*-toluenesulfonyl hydrazide (**2a**, 2.2 mmol, 0.409 g), TBAI (0.3 mmol, 0.111 g, 30 mol%), and acetonitrile (2 ml) were added to a 10 mL Schlenk tube, followed by addition of TBHP (70% aq, 4 equiv, 4 mmol). The mixture was stirred at 40 °C as monitored by TLC, cooled to room temperature. The solution was then extracted with EtOAc, the combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (the eluent, petroleum ether/ethyl acetate) to afford the desired product **3a**.

General Procedure for the Synthesis of 4a-4e

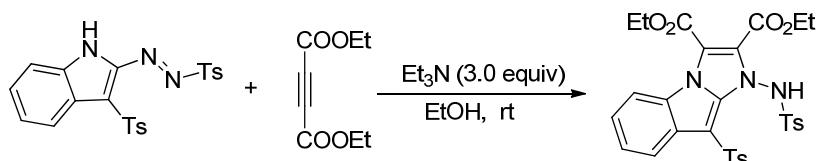
Typical Procedure for the Preparation of 4a:

The substrate 3-methylindole (**1l**, 1 mmol, 0.117 g), *p*-toluenesulfonyl hydrazide (**2a**, 2.2 mmol, 0.109 g), TBAI (0.3 mmol, 0.111 g, 30 mol%), and acetonitrile (2 ml) were added to a 10 mL Schlenk tube, followed by addition of TBHP (70% aq, 4 equiv, 4 mmol). The mixture was stirred at 40 °C as monitored by TLC, cooled to room temperature. The solution was then extracted with EtOAc, the combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (the eluent, petroleum ether/ethyl acetate) to afford the desired product **4a**.

General Procedure for the Synthesis of 1o¹

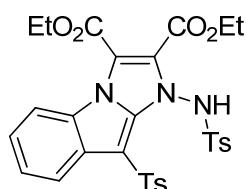
The substrate indole (1 mmol, 0.117 g), 4-bromobenzene-1-sulfonyl chloride (1.2 mmol, 0.3066 g), CuI (0.05 mmol, 0.0095 g, 5 mol%), and acetonitrile (2 ml) were added to a 10 mL Schlenk tube. The mixture was stirred and heated to reflux monitored by TLC, cooled to room temperature. The solution was then extracted with EtOAc, the combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (the eluent, petroleum ether/ethyl acetate) to afford the desired product **1o**.

General Procedure for the Synthesis of 5



The substrate 3-tosyl-2-(tosyldiazenyl)-1*H*-indole (**3a**, 1.0 mmol, 454 mg), Diethyl acetylenedicarboxylate (2.0 mmol, 340 mg) and ethanol (2 ml) were added to a 10 mL Schlenk tube, followed by addition of triethylamine (3.0 mmol, 304 mg). The mixture was stirred at room temperature as monitored by TLC, cooled to room temperature, and the solution was then quenched by diluted hydrochloric acid and extracted with EtOAc, the combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel, eluting with petroleum ether/ethyl acetate, to afford the desired product.

Diethyl 1-(4-methylphenylsulfonamido)-9-tosyl-1*H*-imidazo[1,2-*a*]indole-2,3-dicarboxylate



Red solid, mp: 185–186 °C.

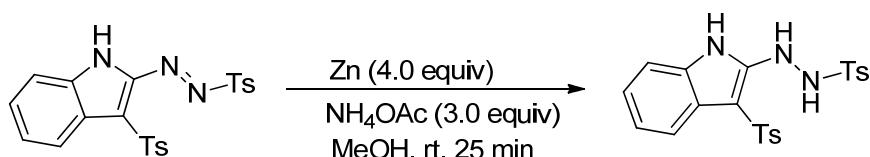
IR (KBr, ν , cm⁻¹): 3134, 2987, 1620, 1727, 1613, 1572, 1455, 1380, 1351, 1294, 1214, 1163, 1084, 1036, 1016, 950, 890, 844, 815.

¹H NMR (400 MHz, DMSO-*d*₆) δ 12.05 (s, 1H), 8.01–7.88 (m, 4H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.40–7.37 (m, 3H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.27–7.23 (m, 1H), 4.45–4.39 (m, 2H), 3.89–3.68 (m, 2H), 2.38 (s, 3H), 2.32 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.15 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 158.6, 156.7, 143.2, 141.7, 140.4, 130.3, 129.9, 129.8, 128.3, 126.9, 125.6, 124.9, 121.5, 119.5, 114.2, 90.3, 63.4, 62.8, 21.5, 21.4, 14.1, 13.8

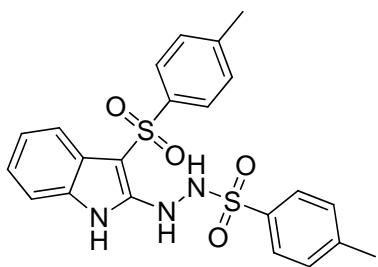
HRMS (ESI) *m/z*: calcd for C₃₀H₂₉N₃O₈S₂Na: 646.1288 [M+Na]⁺; found: 646.1299.

General Procedure for the Synthesis of **6²**



The substrate 3-tosyl-2-(tosyldiazenyl)-1*H*-indole (**3a**, 1.0 mmol, 454 mg), ammonium acetate (3.0 mmol, 231 mg) and methyl alcohol (2 ml) were added to a 10 mL Schlenk tube, followed by addition of Zn (4.0 mmol, 262 mg). The mixture was stirred at room temperature for 25 min as monitored by TLC. The solution was then evaporated under vacuum. The residue was purified by column chromatography on silica gel, eluting with petroleum ether/ethyl acetate, to afford the desired product.

4-Methyl-N'-(6-methyl-3-tosyl-1H-indol-2-yl)benzenesulfonohydrazide



Red solid, mp: 195–196 °C.

IR (KBr, ν , cm⁻¹): 3340, 3254, 1715, 1620, 1579, 1502, 1475, 1461, 1375, 1343, 1276, 1242, 1166, 1134, 1091, 1063, 1015, 813.

¹H NMR (400 MHz, DMSO-*d*₆) δ 11.75 (s, 1H), 9.99 (s, 1H), 7.94 (s, 1H), 7.81 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.26 (m, 1H), 7.06–6.97 (m, 2H), 2.45 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 148.7, 144.9, 143.4, 141.6, 134.4, 132.5, 130.4, 130.1, 128.4, 125.7, 125.1, 121.7, 121.2, 116.6, 111.8, 90.2, 21.6, 21.4

HRMS (ESI) *m/z*: calcd for C₂₂H₂₁N₃NaO₄S₂Na: 478.0865 [M+Na]⁺; found: 478.0884.

Reference

1. M. Rahman, M. Ghosh, A. Hajra and A. Majee, *J. Sulfur Chem.* 2013, **34**, 342.
2. (a) L. Hu, X. Cao, L. Chen, J. Zheng, J. Lu, X. Sun and H. Gu, *Chem. Commun.* 2012, **48**, 3445.
 (b) N. L. Dunn, M. Ha and A. T. Radosevich, *J. Am. Chem. Soc.* 2012, **134**, 11330. (c) W. M. Koppes, J. S. Moran, J. C. Oxley and J. L. Smith, *Tetrahedron Lett.* 2008, **49**, 3234. (d) F. A. Khan, J. Dash, C. Sudheer and R. K. Gupta, *Tetrahedron Lett.* 2003, **44**, 7783.

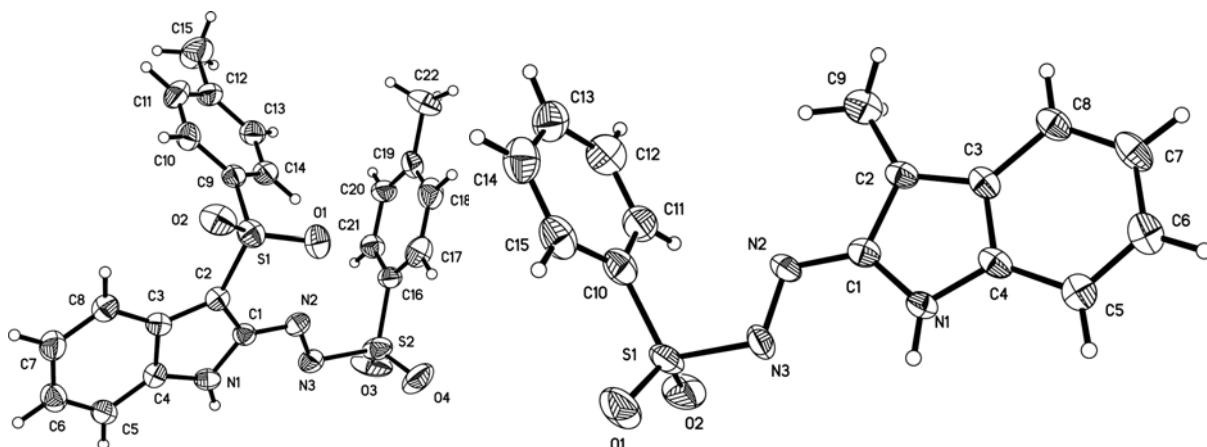
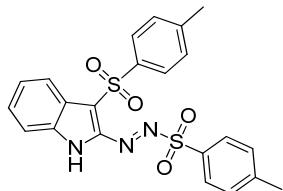


Fig 1, X-ray Structure of 3a

Fig 2, X-ray Structure of 4b

3-Tosyl-2-(tosyldiazenyl)-1H-indole (3a)



Red solid, mp: 176-177 °C.

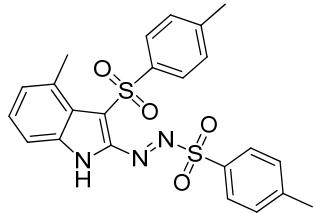
IR (KBr, ν , cm⁻¹): 3277, 2360, 1595, 1504, 1455, 1426, 1390, 1337, 1215, 1186, 1163, 1082, 944, 891, 818.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.34 (s, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.48–7.55 (m, 2H), 7.42–7.34 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 2H), 2.50 (s, 3H), 2.33 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.1, 144.7, 141.1, 139.9, 136.5, 130.9, 130.8, 130.5, 130.4, 129.5, 127.2, 125.4, 124.5, 123.8, 121.9, 114.6, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₉N₃O₄S₂: 452.0733 [M-H]⁻; found: 452.0725.

4-Methyl-3-tosyl-2-(tosyldiazenyl)-1H-indole (3b)



Red solid, mp: 170-171 °C.

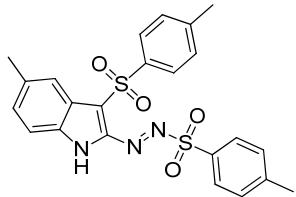
IR (KBr, ν , cm⁻¹): 3266, 2924, 2359, 1595, 1490, 1426, 1366, 1323, 1209, 1188, 1157, 1083, 931, 872, 814.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.32 (s, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.36–7.35 (m, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.07 (t, *J* = 7.2 Hz, 1H), 2.73 (s, 3H), 2.42 (s, 3H), 2.35 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 146.9, 144.3, 142.9, 140.3, 137.1, 133.0, 130.8, 130.7, 130.3, 129.9, 129.8, 127.6, 126.5, 124.5, 124.3, 112.4, 23.4, 21.7, 21.5

HRMS (ESI) *m/z*: calcd for C₂₃H₂₁N₃O₄S₂: 466.0890 [M-H]⁻; found: 466.0902.

5-Methyl-3-tosyl-2-(tosyldiazenyl)-1H-indole (3c)



Red solid, mp: 187-188 °C.

IR (KBr, ν , cm⁻¹): 3272, 2359, 1595, 1507, 1462, 1420, 1376, 1334, 1291, 1209, 1185, 1157, 1082, 899, 819, 806.

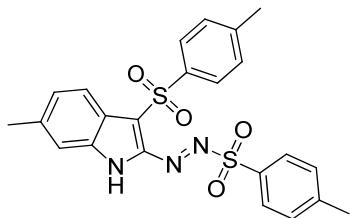
¹H NMR (400 MHz, DMSO-*d*₆) δ 13.26 (s, 1H), 8.02 (s, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.41–7.37 (m, 4H), 7.27 (d, *J* = 7.2 Hz, 2H), 2.50 (s, 3H), 2.44 (s, 3H), 2.33 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.0, 144.6, 141.0, 140.0, 135.1, 134.0, 132.8, 130.9, 130.7,

130.3, 129.7, 127.2, 125.7, 123.2, 120.6, 114.3, 21.9, 21.8, 21.5

HRMS (ESI) m/z : calcd for $C_{23}H_{21}N_3O_4S_2$: 466.0890 [M-H]⁻; found: 466.0910.

6-Methyl-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3d)



Red solid, mp: 180-181 °C.

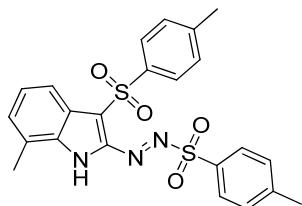
IR (KBr, ν , cm⁻¹): 3312, 2359, 1597, 1492, 1454, 1372, 1345, 1300, 1211, 1187, 1159, 1083, 932, 854, 805.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.20 (s, 1H), 8.12 (d, J = 8.8 Hz, 1H), 7.88 (d, J = 8.4 Hz, 2H), 7.66 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.0 Hz, 2H), 7.27–7.20 (m, 4H), 2.49 (s, 3H), 2.42 (s, 3H), 2.33 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 146.9, 144.7, 141.5, 140.8, 139.9, 137.2, 130.8, 130.7, 130.4, 129.9, 127.3, 126.9, 124.4, 123.8, 121.6, 113.5, 22.3, 21.8, 21.5

HRMS (ESI) m/z : calcd for $C_{23}H_{21}N_3O_4S_2$: 466.0890 [M-H]⁻; found: 466.0892.

7-Methyl-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3e)



Red solid, mp: 172-173 °C.

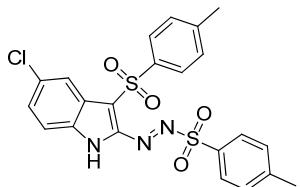
IR (KBr, ν , cm⁻¹): 3267, 2359, 1595, 1490, 1426, 1366, 1323, 1188, 1158, 1083, 932, 872, 814.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.32 (s, 1H), 7.88 (d, J = 8.0 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 4.4 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 7.08–7.06 (m, 1H), 2.73 (s, 3H), 2.43 (s, 3H), 2.36 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 146.9, 144.3, 142.9, 140.3, 137.1, 132.9, 130.8, 130.7, 130.3, 129.9, 129.8, 127.6, 126.5, 124.5, 124.3, 112.4, 23.3, 21.7, 21.5

HRMS (ESI) m/z : calcd for $C_{23}H_{21}N_3O_4S_2$: 466.0890 [M-H]⁻; found: 466.0892.

5-Chloro-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3f)



Red solid, mp: 192 -193 °C.

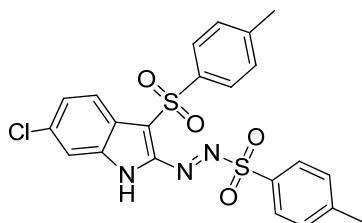
IR (KBr, ν , cm⁻¹): 3255, 2361, 1595, 1505, 1454, 1380, 1336, 1269, 1186, 1160, 1082, 966, 896, 838, 811.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.57 (s, 1H), 8.21–8.20 (m, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 1.6 Hz, 2H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.50 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.3, 144.9, 141.9, 139.6, 134.7, 130.9(3), 130.8(8), 130.4, 129.2, 129.2, 129.0, 127.4, 125.9, 122.7, 120.6, 116.7, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₈ClN₃O₄S₂: 486.0343 [M-H]⁻; found: 486.0345.

6-Chloro-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3g)



Orange solid, mp: 184 -185 °C.

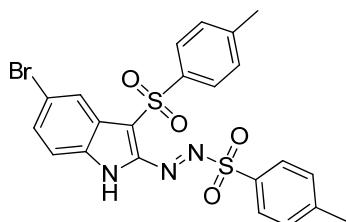
IR (KBr, *v*, cm⁻¹): 3303, 2359, 1612, 1596, 1494, 1418, 1376, 1351, 1296, 1186, 1162, 1084, 917, 847, 809.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.48 (s, 1H), 8.24 (d, *J* = 8.8 Hz, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 2.0Hz, 1H), 7.41–7.38 (m, 3H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.50 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.2, 145.0, 141.6, 139.6, 136.7, 134.6, 130.9(3), 130.8(6), 130.4, 129.3, 127.3, 125.2, 124.0, 123.9, 123.7, 113.9, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₈ClN₃O₄S₂: 486.0343 [M-H]⁻; found: 486.0343.

5-Bromo-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3h)



Orange solid, mp: 187-188 °C.

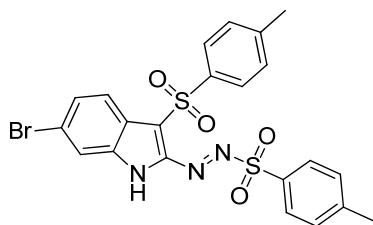
IR (KBr, *v*, cm⁻¹): 3279, 2360, 1595, 1504, 1452, 1423, 1370, 1341, 1266, 1186, 1161, 1084, 957, 893, 835, 805.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.57 (s, 1H), 8.37 (d, *J* = 1.6 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 2H), 7.69–7.63 (m, 3H), 7.47 (d, *J* = 9.2 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 2.51 (s, 3H), 2.35 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.3, 144.9, 141.7, 139.6, 134.9, 132.9, 130.9(4), 130.8(8), 130.4, 129.2, 127.3, 126.4, 123.6, 122.5, 117.2, 116.9, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₈BrN₃O₄S₂: 531.9819 [M-H]⁻; found: 531.9820.

6-Bromo-3-tosyl-2-(tosyldiazenyl)-1*H*-indole (3i)



Yellow solid, mp: 187-188 °C.

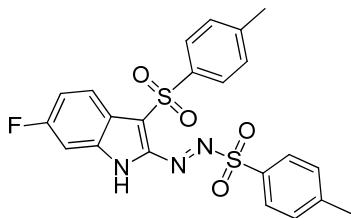
IR (KBr, ν , cm⁻¹): 3303, 2922, 2360, 1596, 1494, 1446, 1420, 1390, 1377, 1350, 1295, 1185, 1161, 1084, 908, 843, 811.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.47 (s, 1H), 8.18 (d, *J* = 8.8 Hz, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.69–7.65 (m, 3H), 7.53–7.51 (m, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.50 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.2, 145.0, 141.4, 139.6, 136.9, 130.9(2), 130.8(6), 130.4, 129.3, 127.7, 127.3, 124.2, 123.9, 123.8, 123.1, 116.9, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₈BrN₃O₄S₂: 531.9819 [M-H]⁻; found: 531.9830.

6-Fluoro-3-tosyl-2-(tosyldiazenyl)-1H-indole (3j)



Orange solid, mp: 189-190 °C.

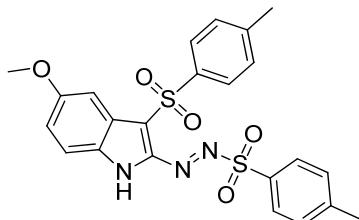
IR (KBr, ν , cm⁻¹): 3265, 2360, 1625, 1595, 1508, 1450, 1422, 1390, 1397, 1332, 1302, 1231, 1187, 1163, 1120, 1082, 934, 856, 818.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.46 (s, 1H), 7.30–8.27 (m, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.30–7.20 (m, 4H), 2.50 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 164.5, 147.1, 144.9, 141.7, 139.6, 137.2, 130.9, 130.8, 130.4, 129.5, 127.3, 124.5, 124.3, 122.3, 114.7, 100.1, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₂H₁₈FN₃O₄S₂: 470.0639 [M-H]⁻; found: 470.0645.

5-Methoxy-3-tosyl-2-(tosyldiazenyl)-1H-indole (3k)



Black solid, mp: 185-186 °C.

IR (KBr, ν , cm⁻¹): 3288, 2360, 1619, 1595, 1572, 1506, 1465, 1331, 1280, 1154, 1027, 981, 899, 840.

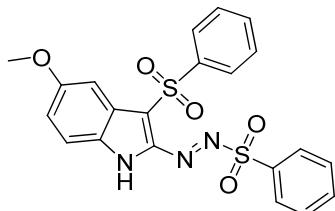
¹H NMR (400 MHz, DMSO-*d*₆) δ 13.32 (s, 1H), 7.88 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 2.4 Hz, 1H), 7.44–7.39 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.21–7.18 (m, 1H), 3.88 (s, 3H), 2.50 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 156.8, 146.9, 144.6, 141.0, 140.1, 132.3, 130.8, 130.7, 130.3,

129.9, 127.2, 126.5, 123.4, 122.8, 116.0, 100.4, 56.0, 21.8, 21.5

HRMS (ESI) m/z : calcd for $C_{23}H_{21}N_3O_5S_2$: 482.0838 [M-H]⁻; found: 482.0856.

5-Methoxy-3-(phenylsulfonyl)-2-((phenylsulfonyl)diazenyl)-1H-indole (3l)



Black solid, mp: 181–182 °C.

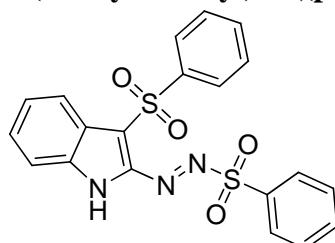
IR (KBr, ν , cm⁻¹): 3273, 2360, 1572, 1504, 1447, 1332, 1278, 1208, 1158, 1082, 897, 839, 814.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.40 (s, 1H), 8.02–7.95 (m, 3H), 7.87–7.83 (m, 2H), 7.63–7.58 (m, 2H), 7.54–7.46 (m, 4H), 7.42 (d, J = 8.8 Hz, 1H), 7.21 (dd, J = 2.4, 2.4 Hz, 1H), 3.88 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 156.9, 142.8, 141.0, 136.0, 133.9, 132.9, 132.4, 130.7, 130.4, 129.9, 127.1, 126.7, 123.5, 122.4, 116.1, 100.3, 56.0

HRMS (ESI) m/z : calcd for $C_{21}H_{17}N_3O_5S_2$: 456.0465 [M-H]⁻; found: 456.0457.

3-(Phenylsulfonyl)-2-((phenylsulfonyl)diazenyl)-1H-indole (3m)



Red solid, mp: 179–181 °C.

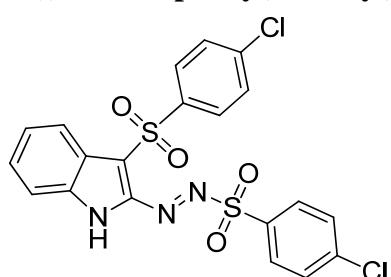
IR (KBr, ν , cm⁻¹): 3273, 2359, 1614, 1583, 1506, 1455, 1448, 1431, 1331, 1216, 1159, 1101, 1082, 999, 944, 892, 837.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.42 (s, 1H), 8.25 (d, J = 8.4 Hz, 1H), 8.04–8.02 (m, 2H), 8.00–7.96 (m, 1H), 7.88–7.85 (m, 2H), 7.62–7.59 (m, 1H), 7.52–7.46 (m, 6H), 7.39–7.35 (m, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 142.6, 141.2, 136.5, 136.1, 134.0, 132.6, 130.8, 130.5, 130.4, 130.0, 127.2, 125.5, 124.6, 123.5, 121.8, 114.7

HRMS (ESI) m/z : calcd for $C_{20}H_{15}N_3O_4S_2$: 424.0420 [M-H]⁻; found: 424.0416.

3-((4-Chlorophenyl)sulfonyl)-2-(((4-chlorophenyl)sulfonyl)diazenyl)-1H-indole (3n)



Orange solid, mp: 182–183 °C.

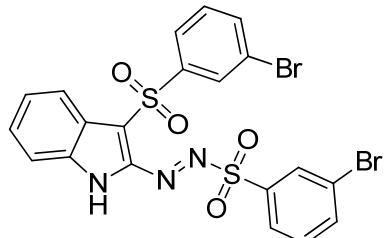
IR (KBr, ν , cm⁻¹): 3197, 2360, 1595, 1581, 1507, 1476, 1430, 1393, 1351, 1312, 1281, 1151, 1091, 1014, 945, 893, 835.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.52 (s, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 8.03–8.00 (m, 2H), 7.96–7.94 (m, 2H), 7.60 (d, *J* = 8.8 Hz, 2H), 7.55–7.50 (m, 4H), 7.41–7.37 (m, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 141.5, 141.4, 141.2, 139.2, 136.7, 132.8, 131.3, 130.7, 130.6, 130.1, 129.0, 125.6, 124.8, 122.8, 121.8, 114.8

HRMS (ESI) *m/z*: calcd for C₂₀H₁₃Cl₂N₃O₄S₂: 491.9640 [M-H]⁻; found: 491.9658.

3-((3-Bromophenyl)sulfonyl)-2-(((3-bromophenyl)sulfonyl)diazenyl)-1H-indole (3o)



Yellow solid, mp: 173–174 °C.

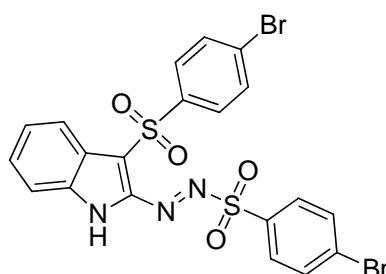
IR (KBr, *v*, cm⁻¹): 3200, 2362, 1619, 1568, 1508, 1459, 1435, 1392, 1344, 1221, 1148, 1069, 996, 945, 894, 838.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.55 (s, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 8.17–8.14 (m, 2H), 8.04 (d, *J* = 7.6 Hz, 1H), 7.88–7.84 (m, 2H), 7.78 (t, *J* = 8.0 Hz, 1H), 7.59–7.47 (m, 4H), 7.41–7.38 (m, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 144.5, 141.3, 139.0, 137.1, 136.8, 134.7, 132.8, 132.5, 132.3, 130.8, 129.7, 129.4, 126.2, 125.6, 124.9, 123.3, 122.9, 122.7, 121.8, 114.8

HRMS (ESI) *m/z*: calcd for C₂₀H₁₃Br₂N₃O₄S₂: 581.8610 [M-H]⁻; found: 581.8616.

3-((4-Bromophenyl)sulfonyl)-2-(((4-bromophenyl)sulfonyl)diazenyl)-1H-indole (3p)



Red solid, mp: 169–170 °C.

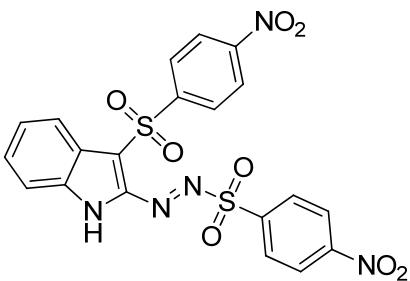
IR (KBr, *v*, cm⁻¹): 3198, 2360, 1614, 1572, 1505, 1431, 1391, 1351, 1310, 1216, 1149, 1097, 1069, 1009, 943, 892, 827.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.52 (s, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 8.09–8.07 (m, 2H), 7.93 (d, *J* = 8.8 Hz, 2H), 7.74–7.71 (m, 2H), 7.55–7.51 (m, 2H), 7.44–7.36 (m, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 141.8, 141.2, 136.7, 133.5, 133.1, 132.7, 131.7, 130.7, 129.0, 128.3, 125.6, 124.8, 122.8, 121.8, 114.8

HRMS (ESI) *m/z*: calcd for C₂₀H₁₃Br₂N₃O₄S₂: 581.8610 [M-H]⁻; found: 581.8619.

3-((4-Nitrophenyl)sulfonyl)-2-(((4-nitrophenyl)sulfonyl)diazenyl)-1H-indole (3q)



Yellow solid, mp: 176-177 °C.

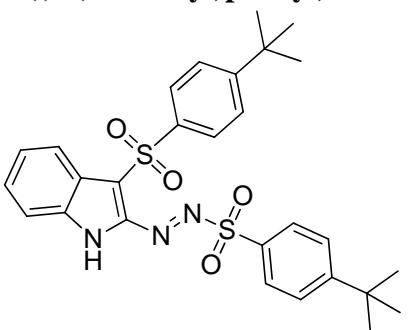
IR (KBr, ν , cm⁻¹): 3214, 2360, 1733, 1607, 1539, 1459, 1439, 1391, 1309, 1219, 1143, 1081, 1013, 945, 894, 852, 834.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.71 (s, 1H), 8.59 (d, *J* = 8.8 Hz, 2H), 8.34–8.30 (m, 4H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.8 Hz, 2H), 7.60–7.53 (m, 2H), 7.40 – 7.43 (m, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 152.0, 150.6, 147.4, 141.5, 138.3, 136.9, 132.7, 131.1, 128.6, 125.8, 125.4, 125.3, 125.2, 121.8, 121.7, 114.9

HRMS (ESI) *m/z*: calcd for C₂₀H₁₃N₅O₈S₂: 514.0121 [M-H]⁻; found: 514.0111.

3-((4-(*tert*-Butyl)phenyl)sulfonyl)-2-(((4-(*tert*-butyl)phenyl)sulfonyl)diazenyl)-1H-indole (3r)



Yellow solid, mp: 193-194 °C.

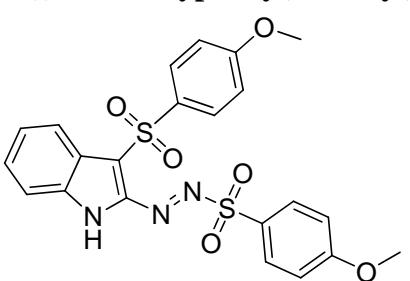
IR (KBr, ν , cm⁻¹): 3266, 2964, 2359, 1593, 1435, 1426, 1388, 1332, 1314, 1205, 1172, 1109, 1083, 892, 833.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.37 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 7.99–7.96 (m, 2H), 7.89–7.87 (m, 2H), 7.54–7.41 (m, 6H), 7.38–7.34 (m, 1H), 1.30 (s, 9H), 1.21 (s, 9H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 159.4, 157.1, 141.1, 139.8, 136.5, 130.8, 130.5, 129.5, 127.4, 127.2, 126.8, 125.3, 124.5, 123.9, 121.8, 114.6, 35.7, 35.4, 31.1

HRMS (ESI) *m/z*: calcd for C₂₈H₃₁N₃O₄S₂: 536.1672 [M-H]⁻; found: 536.1661.

3-((4-Methoxyphenyl)sulfonyl)-2-(((4-methoxyphenyl)sulfonyl)diazenyl)-1H-indole (3s)

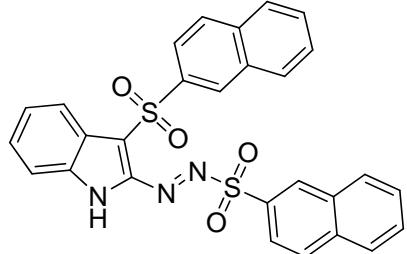


Red solid, mp: 181-183 °C.

IR (KBr, ν , cm⁻¹): 3265, 2360, 1593, 1497, 1459, 1425, 1392, 1315, 1266, 1183, 1158, 1085, 1022, 945, 891, 837, 802.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.29 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 9.2 Hz, 2H), 7.54–7.48 (m, 4H), 7.39–7.33 (m, 3H), 6.98 (d, *J* = 9.2 Hz, 2H), 3.92 (s, 3H), 3.81 (s, 3H).
¹³C NMR (100 MHz, DMSO-*d*₆) δ 165.3, 163.5, 141.0, 136.4, 134.3, 133.2, 130.3, 129.6, 125.2, 124.4, 124.2, 123.4, 121.9, 115.8, 115.1, 114.5, 56.6, 56.2
HRMS (ESI) *m/z*: calcd for C₂₂H₁₉N₃O₆S₂: 484.0631 [M-H]⁻; found: 484.0641.

3-(Naphthalen-2-ylsulfonyl)-2-((naphthalen-2-ylsulfonyl)diazenyl)-1H-indole (3t)



Orange solid, mp: 181–1823 °C.

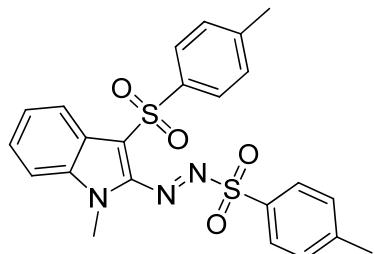
IR (KBr, *v*, cm⁻¹): 3331, 3053, 2359, 1621, 1589, 1508, 1460, 1392, 1330, 1242, 1206, 1150, 1097, 1069, 1019, 948, 893, 858, 828.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.42 (s, 1H), 8.84 (s, 1H), 8.41 (s, 1H), 8.35–8.29 (m, 3H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.97–7.94 (m, 1H), 7.91–7.88 (m, 2H), 7.85–7.77 (m, 2H), 7.69–7.65 (m, 1H), 7.62–7.58 (m, 2H), 7.54–7.47 (m, 2H), 7.40–7.35 (m, 1H), 7.24–7.21 (m, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 141.4, 139.5, 136.6, 136.0, 134.8, 133.2, 132.4, 132.0, 130.8, 130.3, 129.9, 129.8(4), 129.8(1), 128.5(9), 128.5(5), 128.2(2), 128.1(9), 125.5, 124.6, 124.5(7), 123.6, 122.1, 121.9, 114.7

HRMS (ESI) *m/z*: calcd for C₂₈H₁₉N₃O₄S₂: 524.0733 [M-H]⁻; found: 524.0736.

1-Methyl-3-tosyl-2-(tosyldiazenyl)-1H-indole (3u)



Orange solid, mp: 177–178 °C.

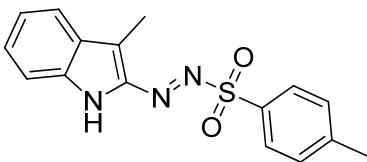
IR (KBr, *v*, cm⁻¹): 2360, 1595, 1471, 1455, 1411, 1345, 1317, 1185, 1164, 1083, 966, 917, 872, 816.

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.37 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.62–7.56 (m, 5H), 7.47–7.43 (m, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 3.87 (s, 3H), 2.48 (s, 3H), 2.34 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 147.0, 144.6, 139.6, 139.5, 138.4, 130.8, 130.7, 130.1, 129.7, 129.4, 127.5, 125.1, 124.7, 122.2, 118.0, 113.1, 33.4, 21.8, 21.5

HRMS (ESI) *m/z*: calcd for C₂₃H₂₁N₃O₄S₂: 466.0890 [M-H]⁻; found: 466.0890.

3-Methyl-2-(tosyldiazenyl)-1H-indole (4a)



Red solid, mp: 167–168 °C.

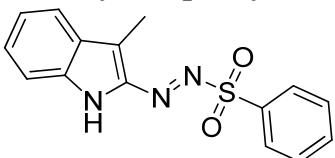
IR (KBr, ν , cm⁻¹): 3378, 2360, 1620, 1595, 1536, 1468, 1391, 1319, 1212, 1181, 1152, 1081, 925, 888, 807.

¹H NMR (400 MHz, DMSO-*d*₆) δ 11.96 (s, 1H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.46–7.42 (m, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.12–7.08 (m, 1H), 2.46 (s, 3H), 2.44 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 145.8, 143.0, 140.0, 132.0, 131.5, 130.5, 129.8, 128.0, 122.8, 121.2, 113.6, 21.7, 9.3

HRMS (ESI) *m/z*: calcd for C₁₆H₁₅N₃O₂S: 336.0777 [M+Na]⁺; found: 336.0775.

3-Methyl-2-((phenylsulfonyl)diazenyl)-1H-indole (4b)



Red solid, mp: 165–166 °C.

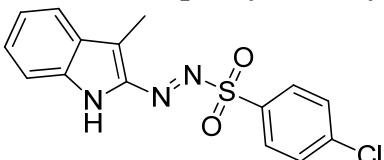
IR (KBr, ν , cm⁻¹): 3389, 3065, 2359, 1620, 1536, 1467, 1441, 1365, 1321, 1210, 1153, 1080, 925, 887, 809.

¹H NMR (400 MHz, DMSO-*d*₆) δ 11.99 (s, 1H), 7.94–7.92 (m, 2H), 7.85–7.81 (m, 1H), 7.74–7.70 (m, 3H), 7.46–7.42 (m, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.11–7.01 (m, 1H), 2.44 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 143.0, 140.1, 135.0, 134.9(5), 131.6, 130.0, 129.8, 128.1, 122.8, 121.2, 113.6, 9.3

HRMS (ESI) *m/z*: calcd for C₁₅H₁₃N₃O₂S: 322.0621 [M+Na]⁺; found: 322.0602.

2-(((4-Chlorophenyl)sulfonyl)diazenyl)-3-methyl-1H-indole (4c)



Red solid, mp: 172–173 °C.

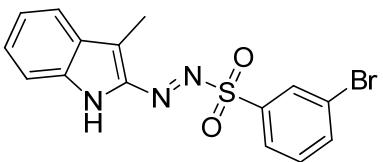
IR (KBr, ν , cm⁻¹): 3374, 2361, 1618, 1580, 1529, 1471, 1434, 1391, 1329, 1153, 1079, 1012, 926, 887, 808, 754, 710, 625, 586, 563.

¹H NMR (400 MHz, DMSO-*d*₆) δ 12.02 (s, 1H), 7.97–7.94 (m, 2H), 7.81–7.79 (m, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.48–7.44 (m, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.12–7.09 (m, 1H), 2.46 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 143.1, 140.3, 140.1, 133.9, 131.9, 131.7, 130.2, 128.1, 122.9, 121.3, 113.7, 9.4

HRMS (ESI) *m/z*: calcd for C₁₅H₁₂ClN₃O₂S: 356.0231 [M+Na]⁺; found: 356.0232.

2-(((3-Bromophenyl)sulfonyl)diazenyl)-3-methyl-1H-indole (4d)



Yellow solid, mp: 173-174 °C.

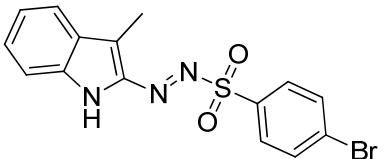
IR (KBr, ν , cm⁻¹): 3353, 3085, 2360, 1620, 1570, 1534, 1462, 1391, 1323, 1214, 1155, 924, 887, 813.

¹H NMR (400 MHz, DMSO-*d*₆) δ 12.03 (s, 1H), 8.05 (d, *J* = 7.6 Hz, 2H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 7.2 Hz, 1H), 7.70–7.66 (m, 1H), 7.48–7.44 (m, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.12–7.09 (m, 1H), 2.46 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 143.1, 140.5, 137.8, 137.1, 132.2, 132.1, 131.9, 128.9, 128.1, 123.0, 122.8, 121.4, 113.7, 9.4

HRMS (ESI) *m/z*: calcd for C₁₅H₁₂BrN₃O₂S: 401.9706 [M+Na]⁺; found: 401.9699.

2-((4-Bromophenyl)sulfonyl)diazenyI-3-methyl-1H-indole (4e)



Red solid, mp: 174-175 °C.

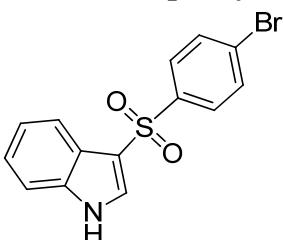
IR (KBr, ν , cm⁻¹): 3374, 2361, 1620, 1571, 1527, 1469, 1432, 1388, 1295, 1150, 1079, 1008, 926, 887, 808.

¹H NMR (400 MHz, DMSO-*d*₆) δ 12.02 (s, 1H), 7.94 (d, *J* = 8.8 Hz, 2H), 7.89–7.85 (m, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.47–7.44 (m, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.12–7.08 (m, 1H), 2.46 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 143.1, 140.4, 134.3, 133.1, 132.0, 131.7, 129.3, 128.1, 122.9, 121.3, 113.7, 9.4

HRMS (ESI) *m/z*: calcd for C₁₅H₁₂BrN₃O₂S: 401.9706 [M+Na]⁺; found: 401.9690.

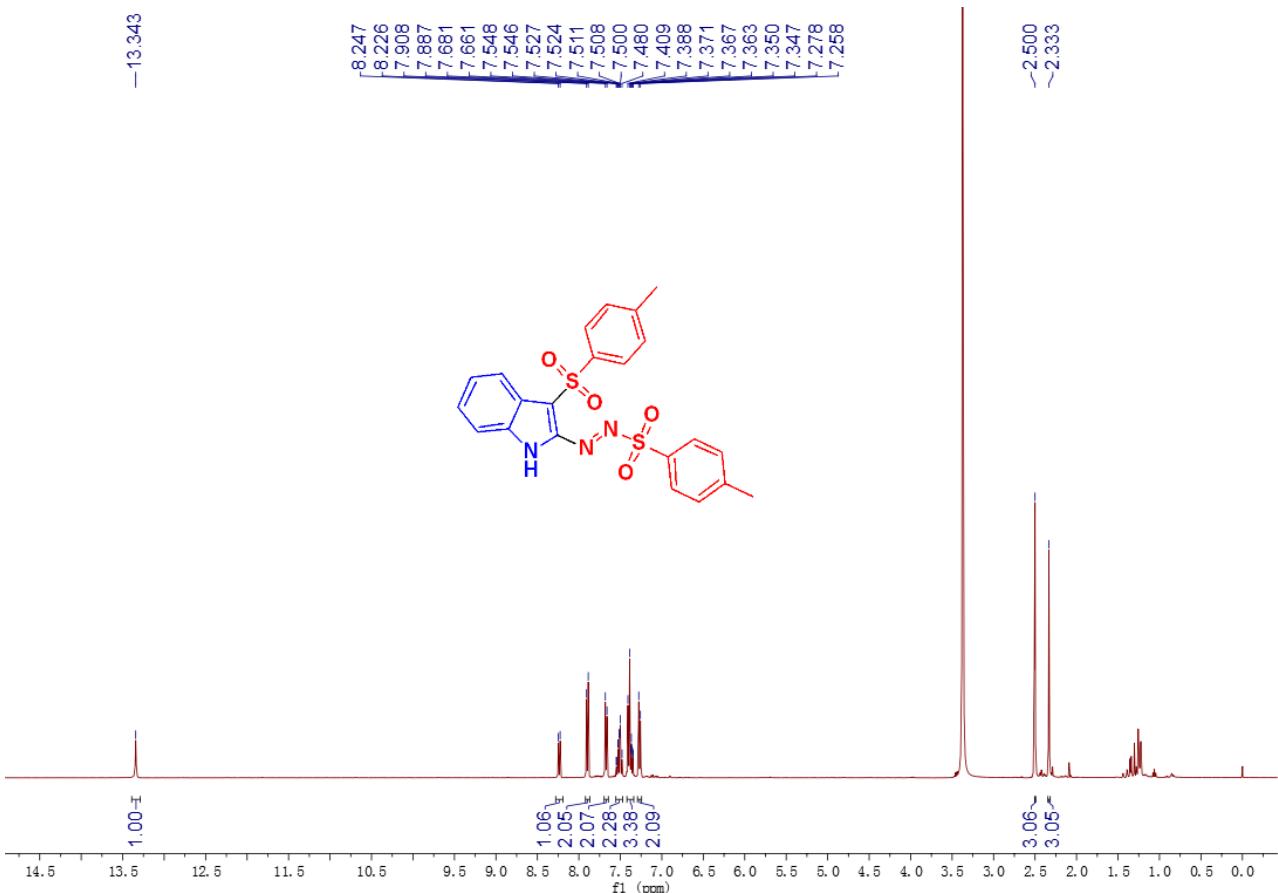
3-((4-Bromophenyl)sulfonyl)-1H-indole (1o)



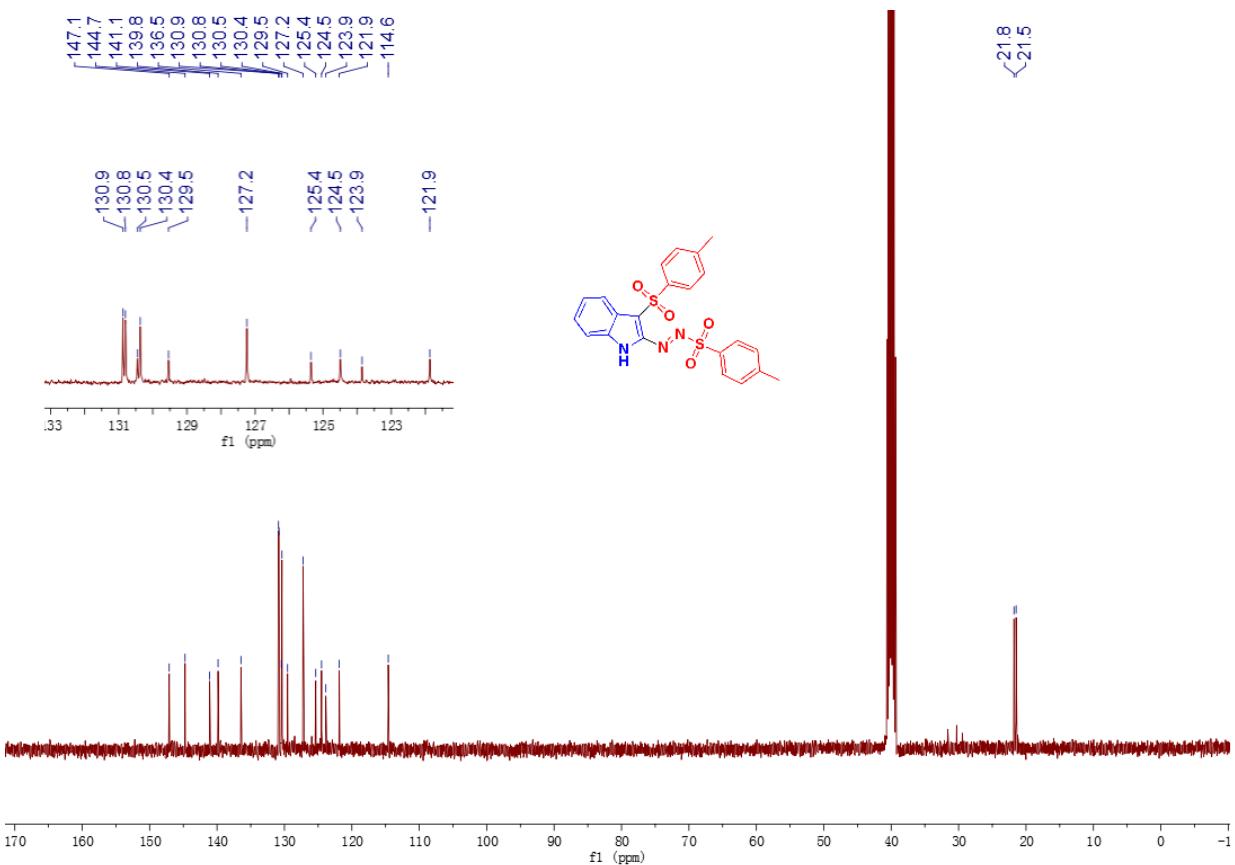
White solid, mp: 153-154 °C.

IR (KBr, ν , cm⁻¹): 3388, 2360, 1610, 1501, 1471, 1454, 1409, 1386, 1338, 1280, 1236, 1080, 1004, 810.

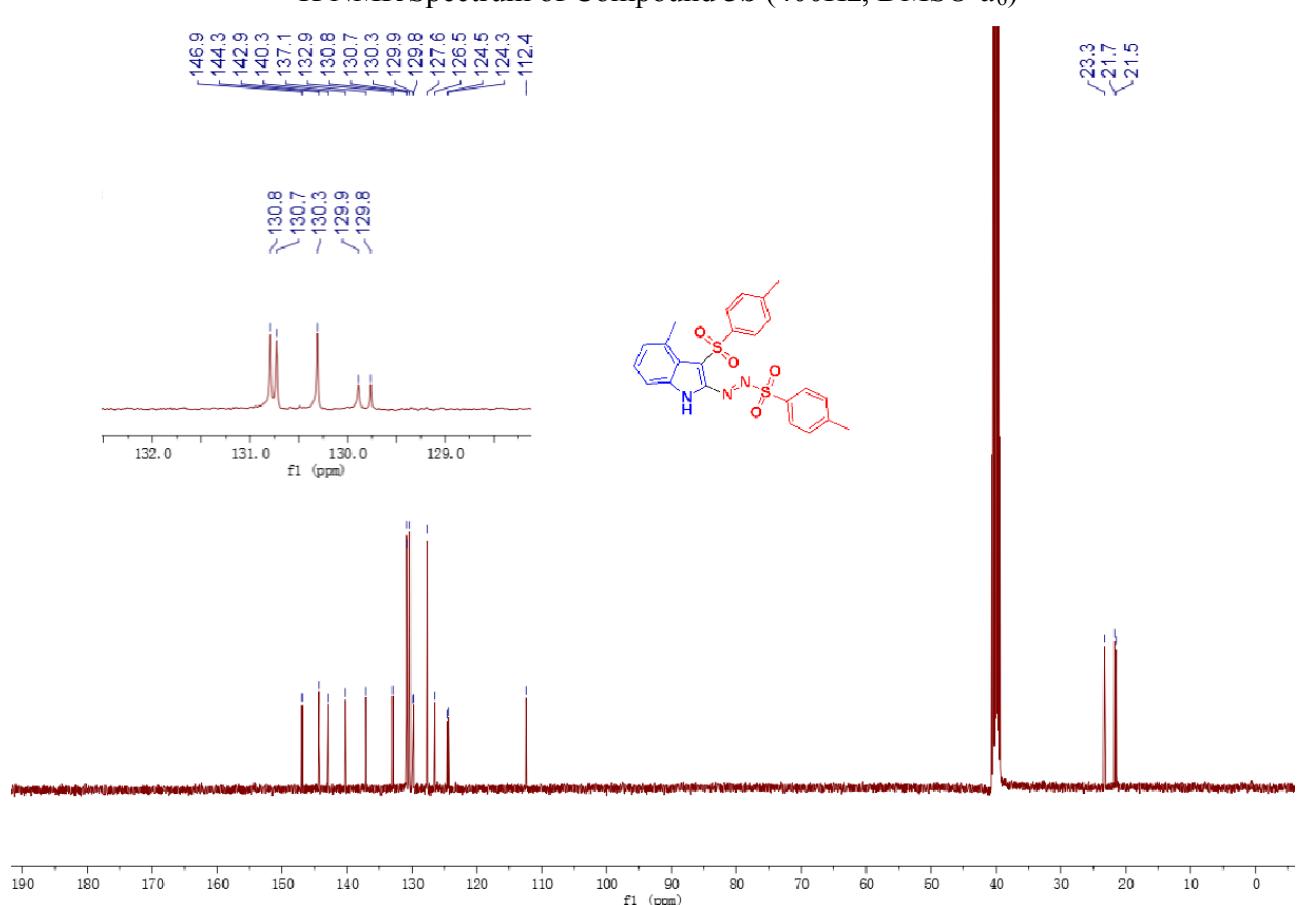
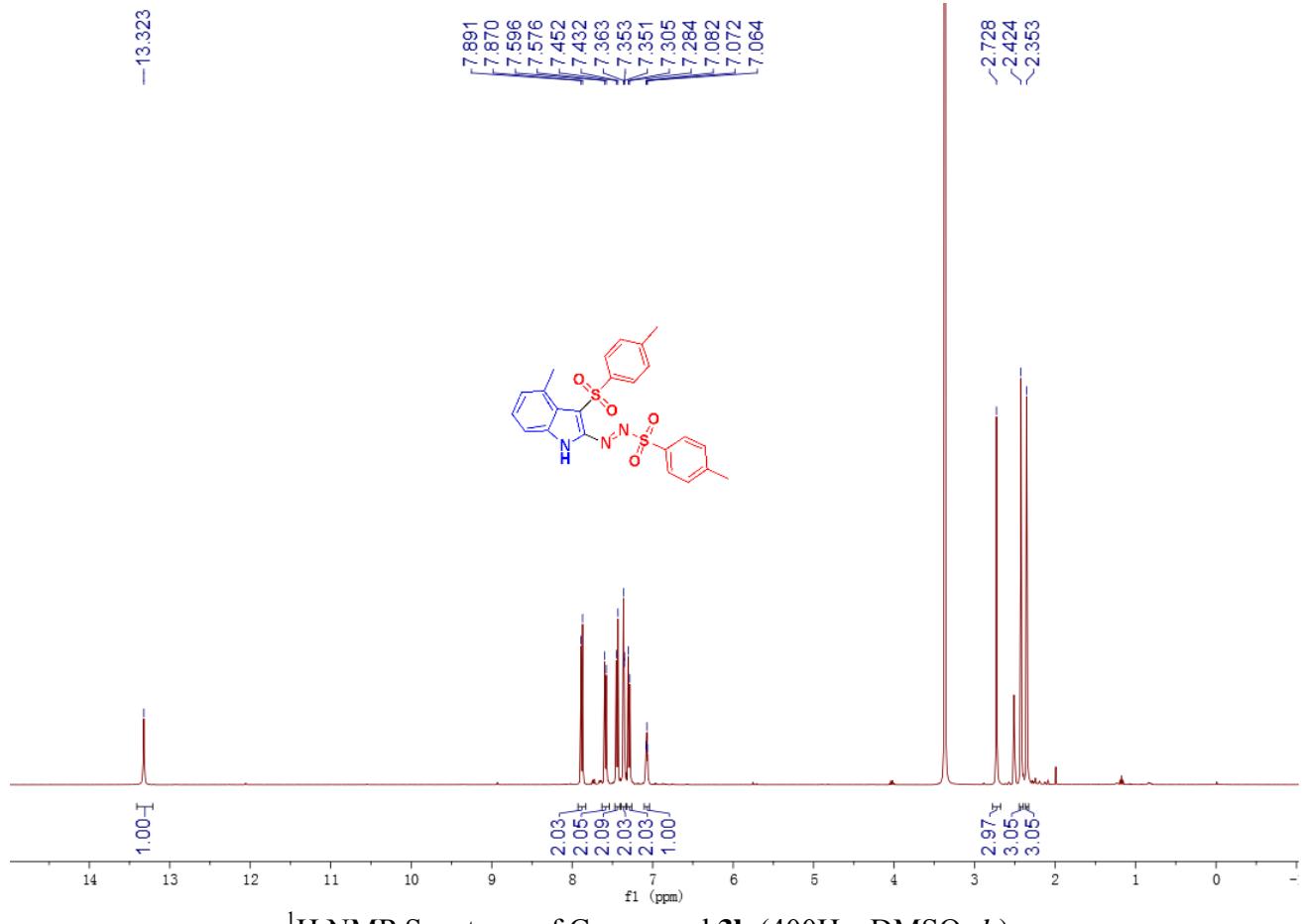
¹H NMR (400 MHz, DMSO-*d*₆) δ 11.76 (s, 1H), 7.80 (d, *J* = 2.4 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.40–7.37 (m, 3H), 7.22–7.18 (m, 1H), 7.10–7.06 (m, 1H), 6.95 (d, *J* = 8.4 Hz, 2H).

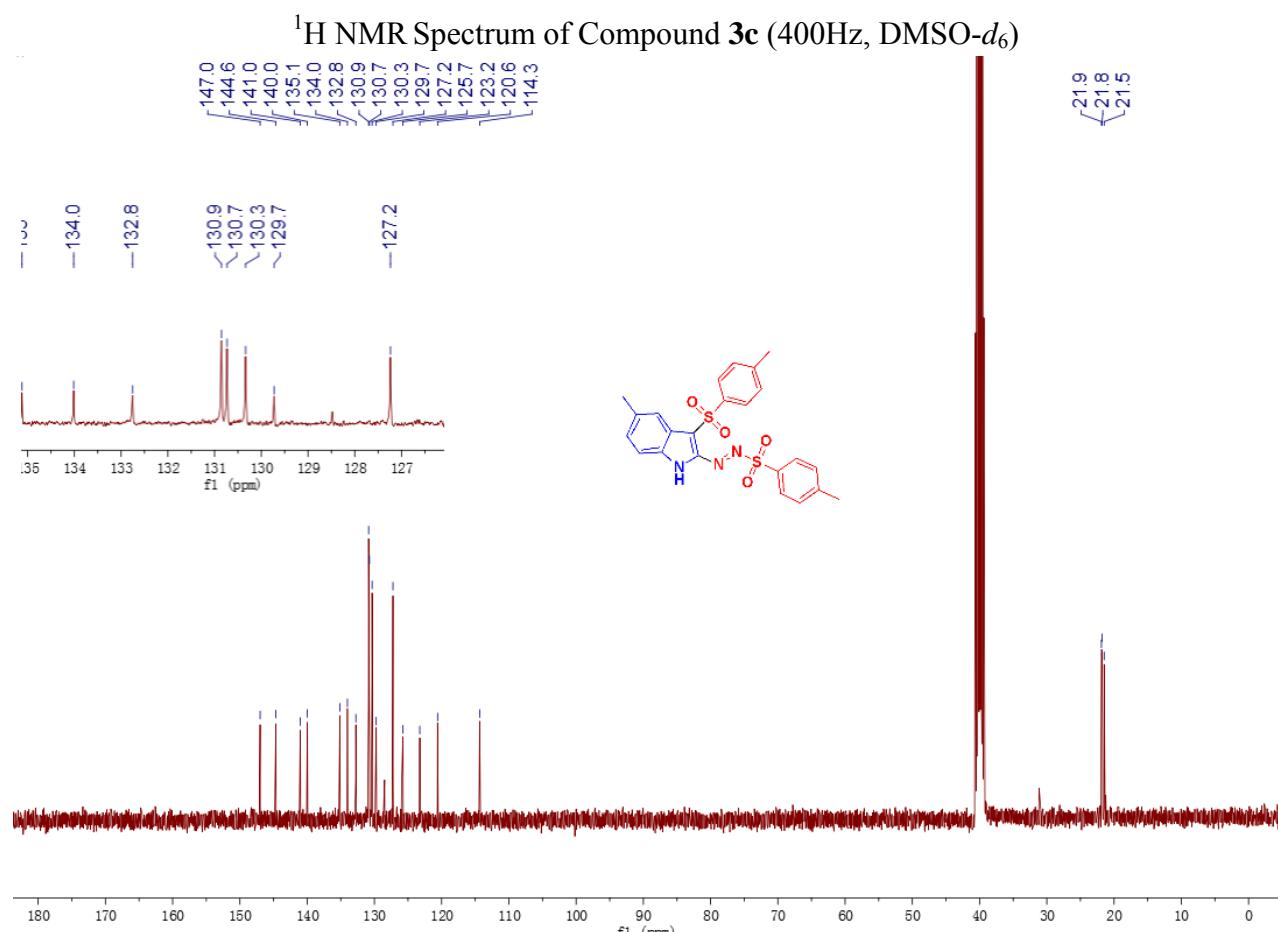
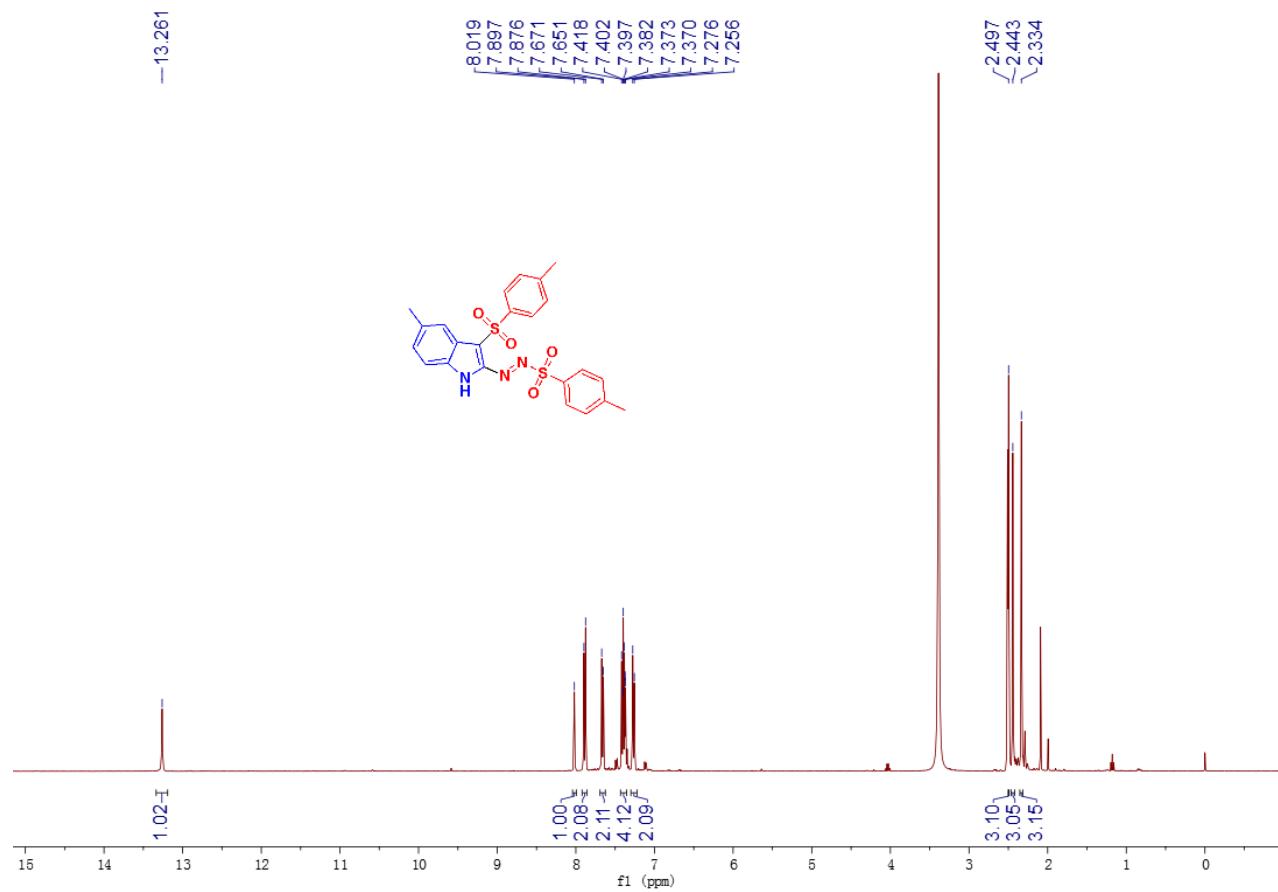


¹H NMR Spectrum of Compound **3a** (400Hz, DMSO-*d*₆)



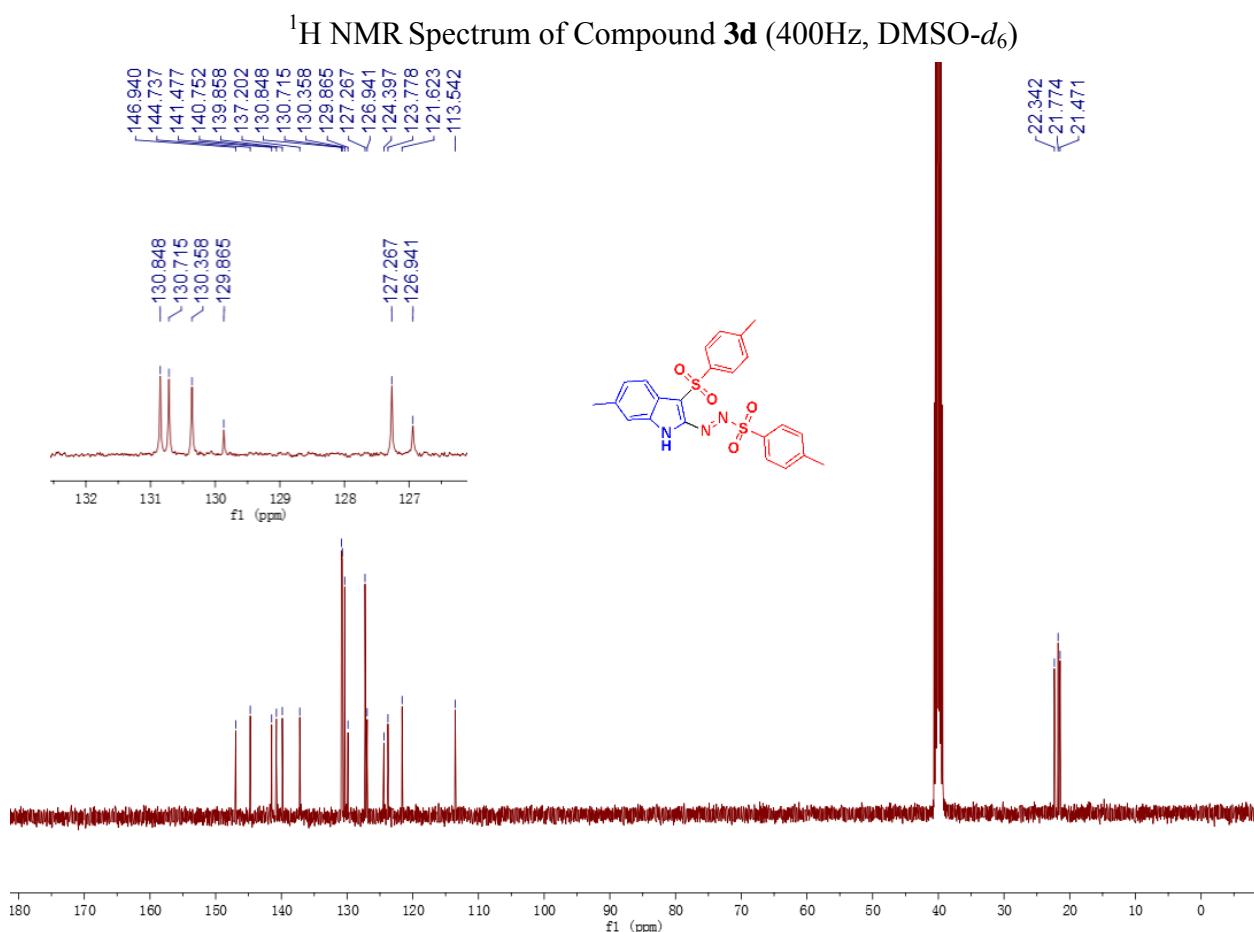
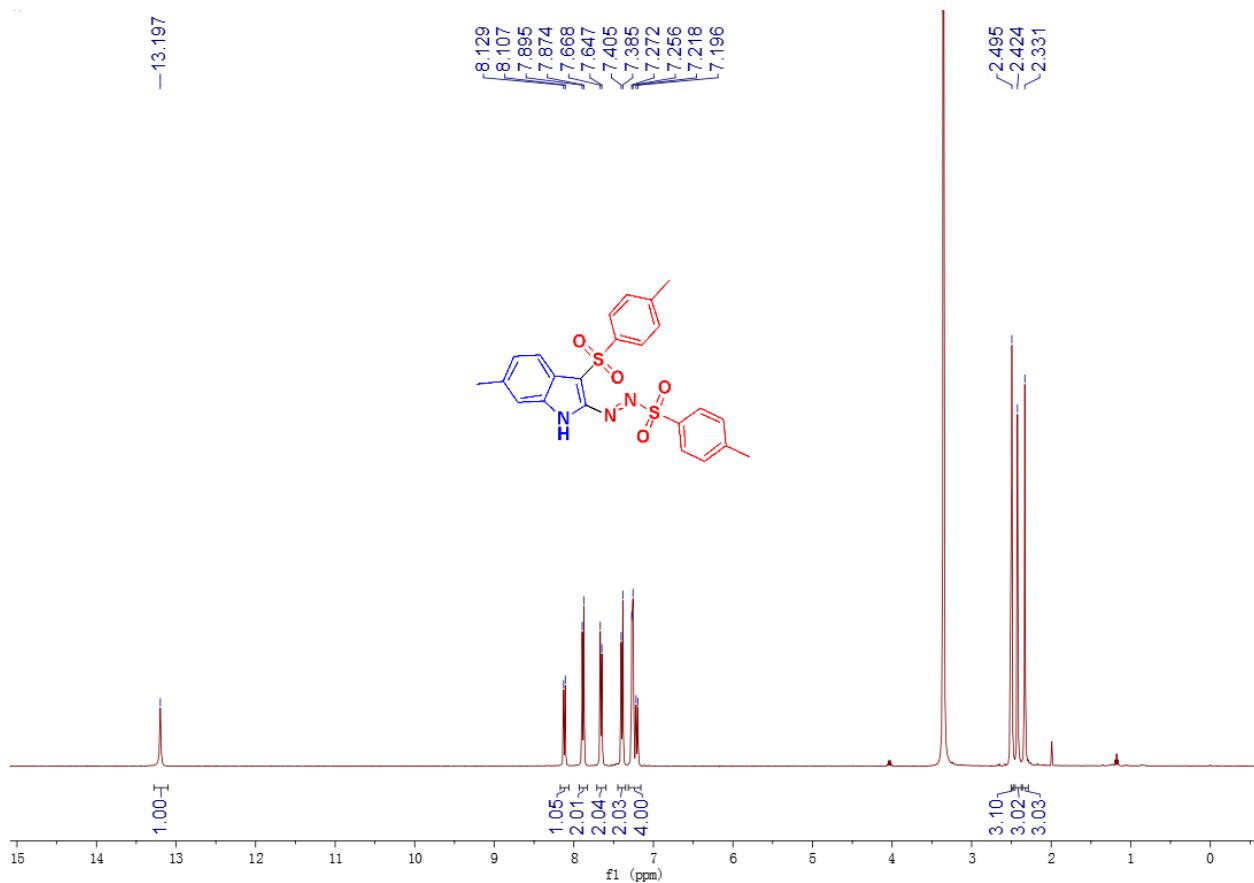
¹³C NMR Spectrum of Compound **3a** (100Hz, DMSO-*d*₆)
S16



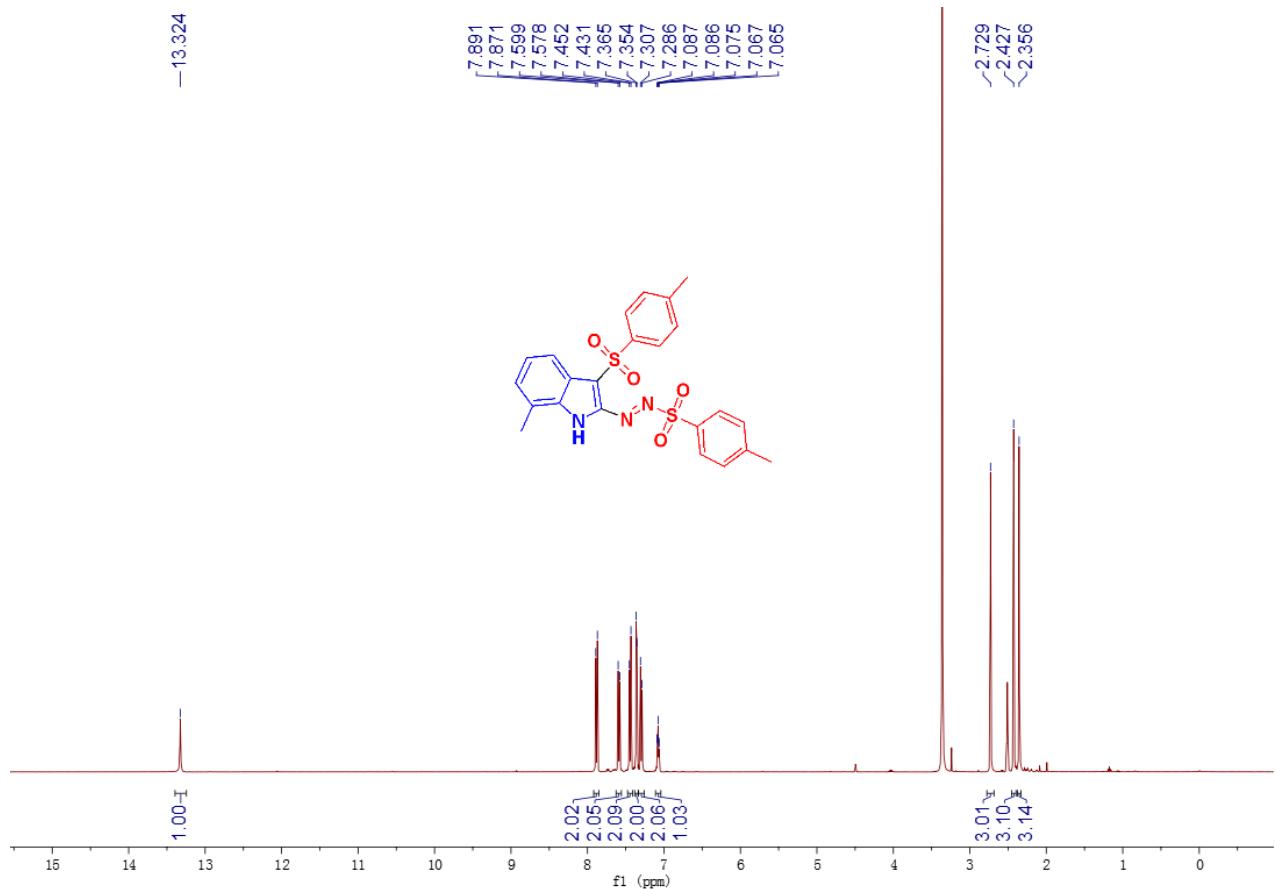


¹³C NMR Spectrum of Compound 3c (100Hz, DMSO-*d*₆)

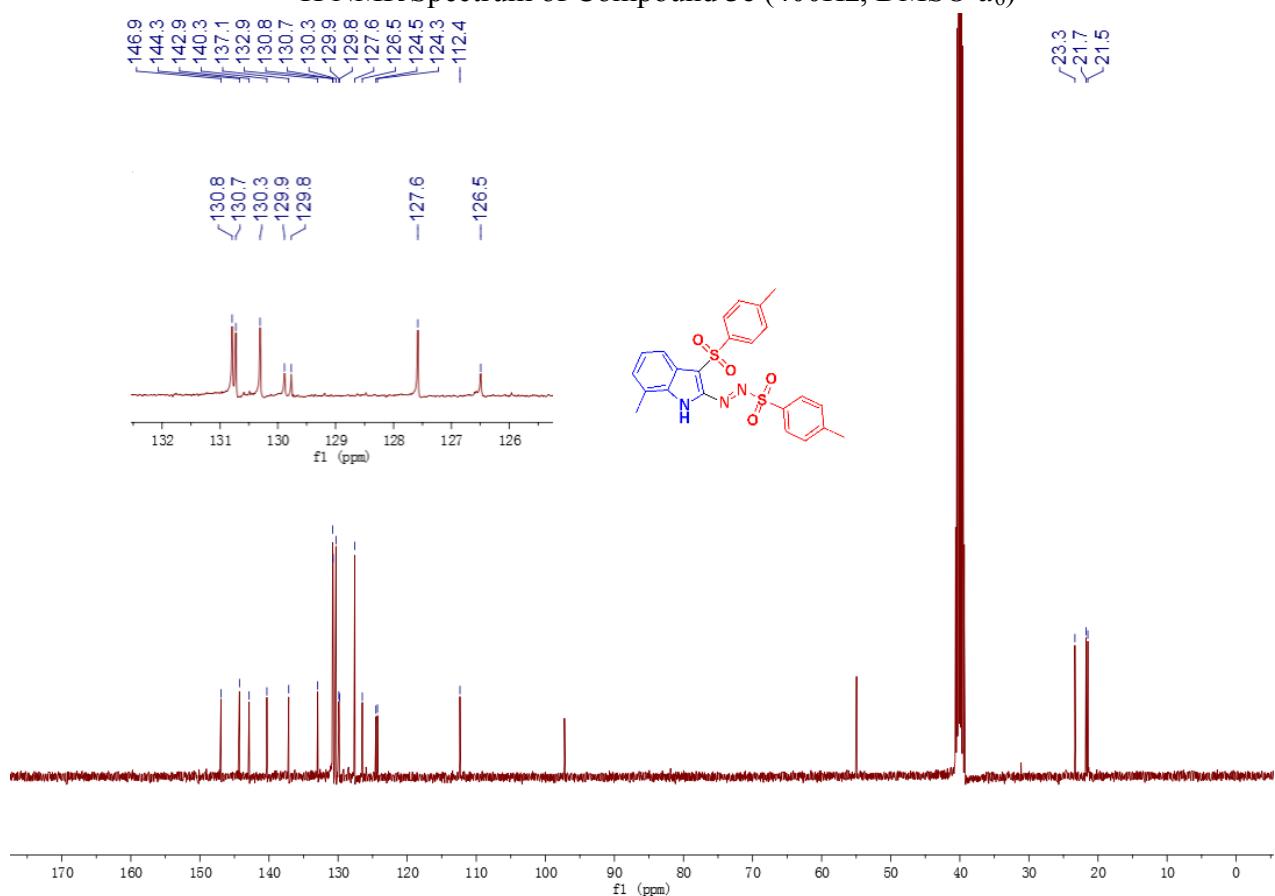
S18



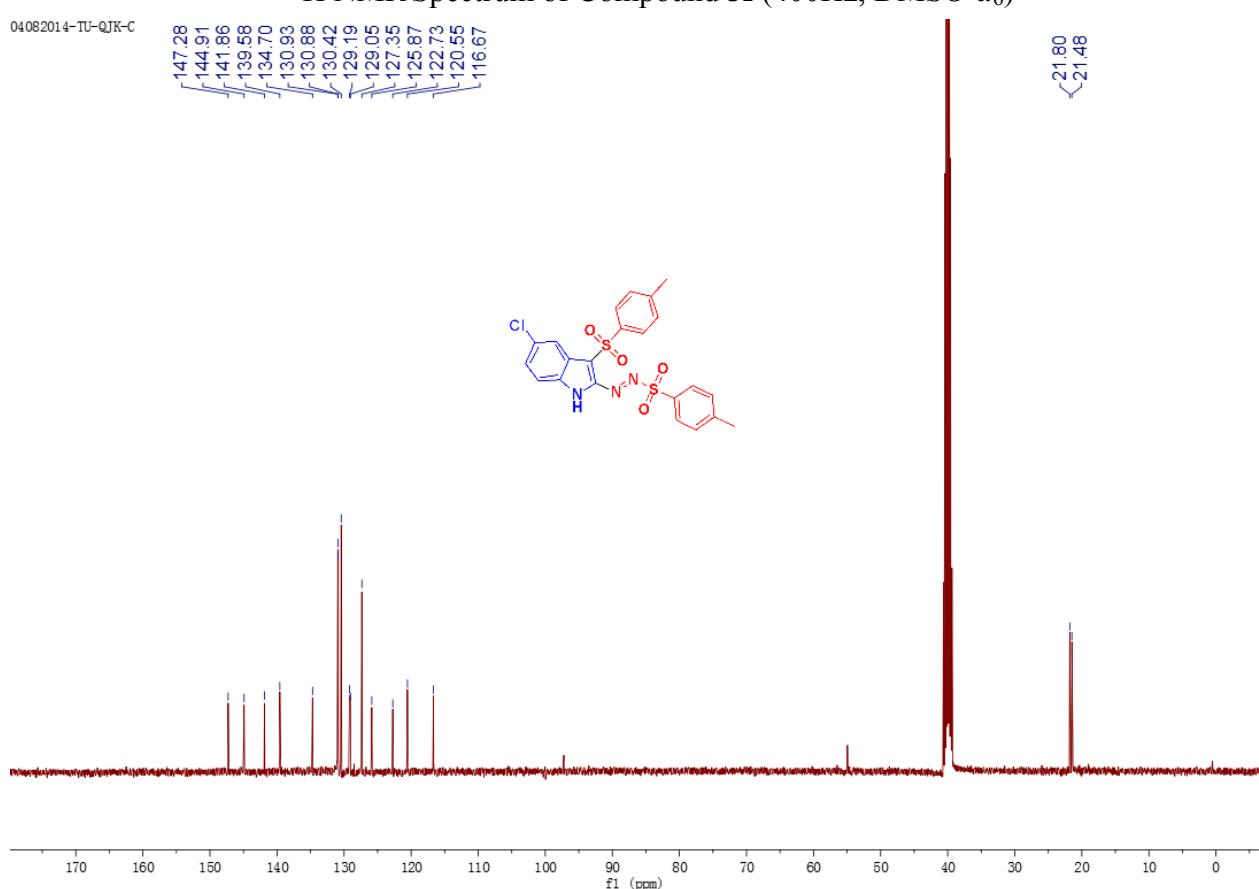
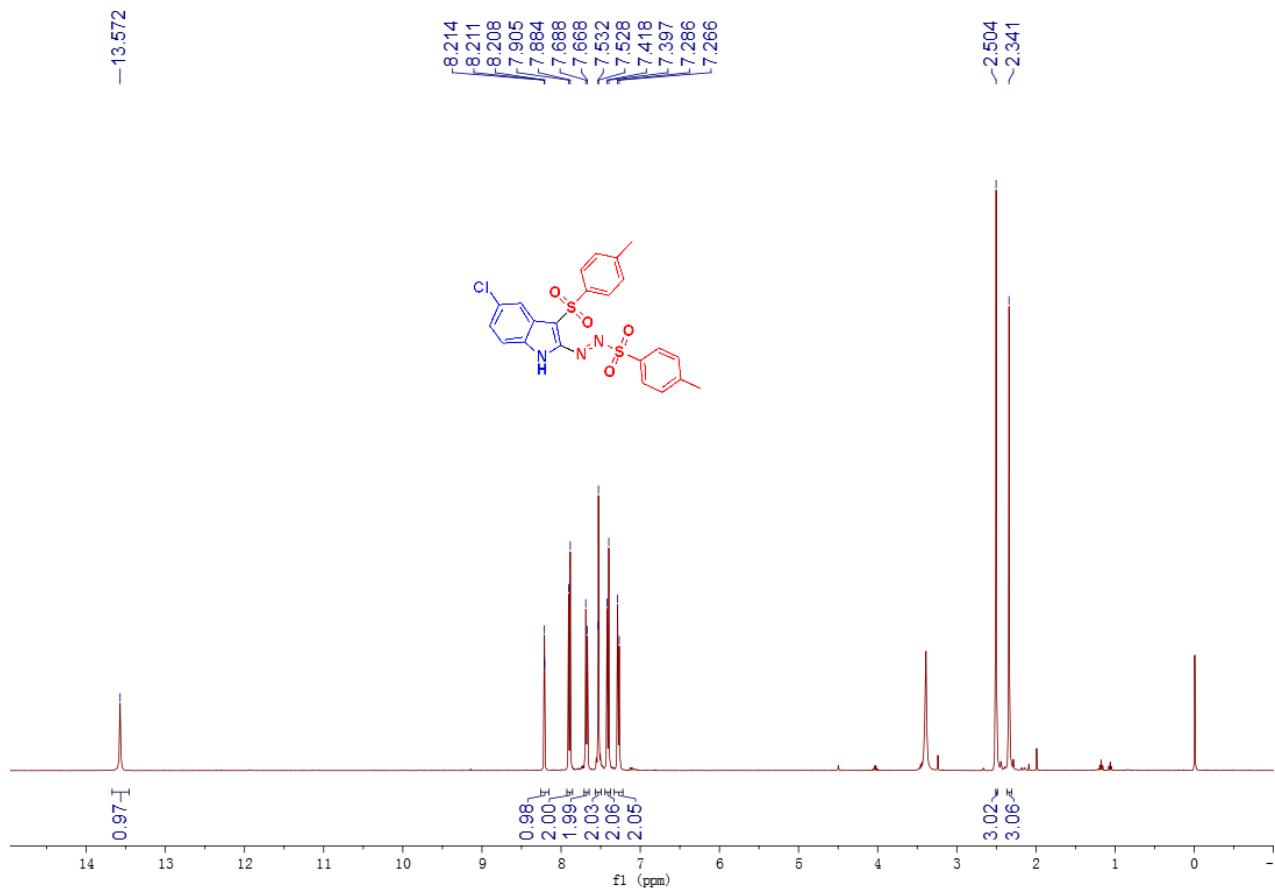
¹³C NMR Spectrum of Compound 3d (100Hz, DMSO-*d*₆)
S19

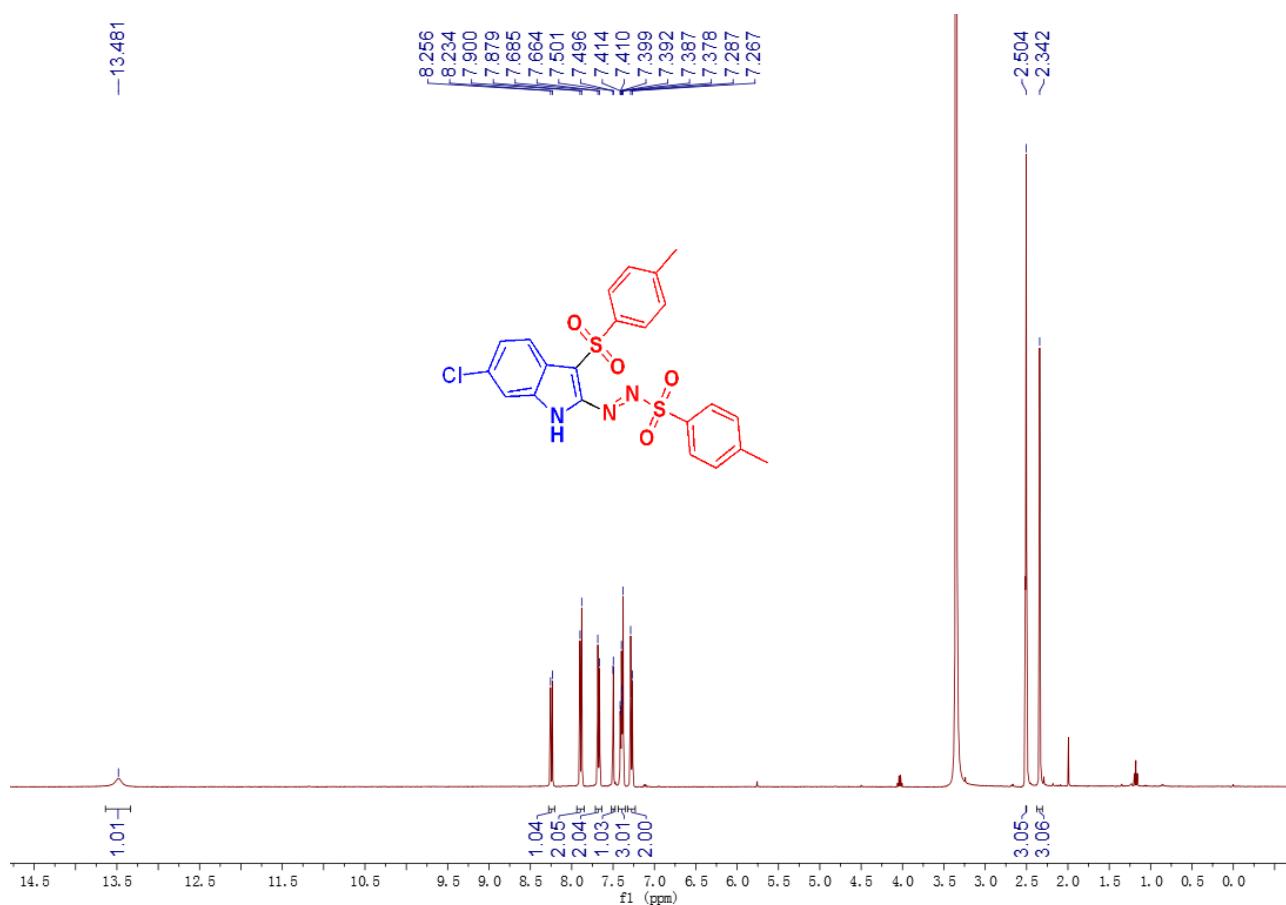


¹H NMR Spectrum of Compound **3e** (400Hz, DMSO-*d*₆)

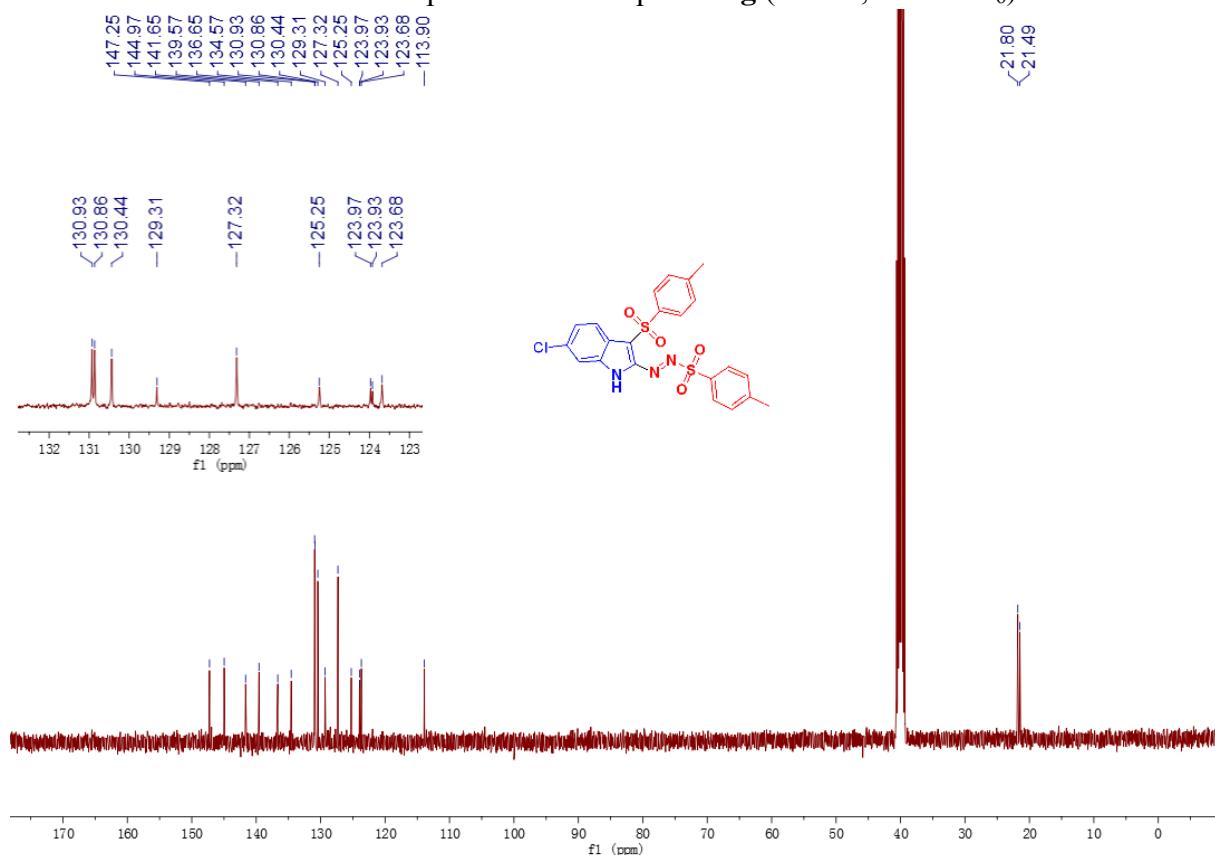


¹³C NMR Spectrum of Compound **3e** (100Hz, DMSO-*d*₆)

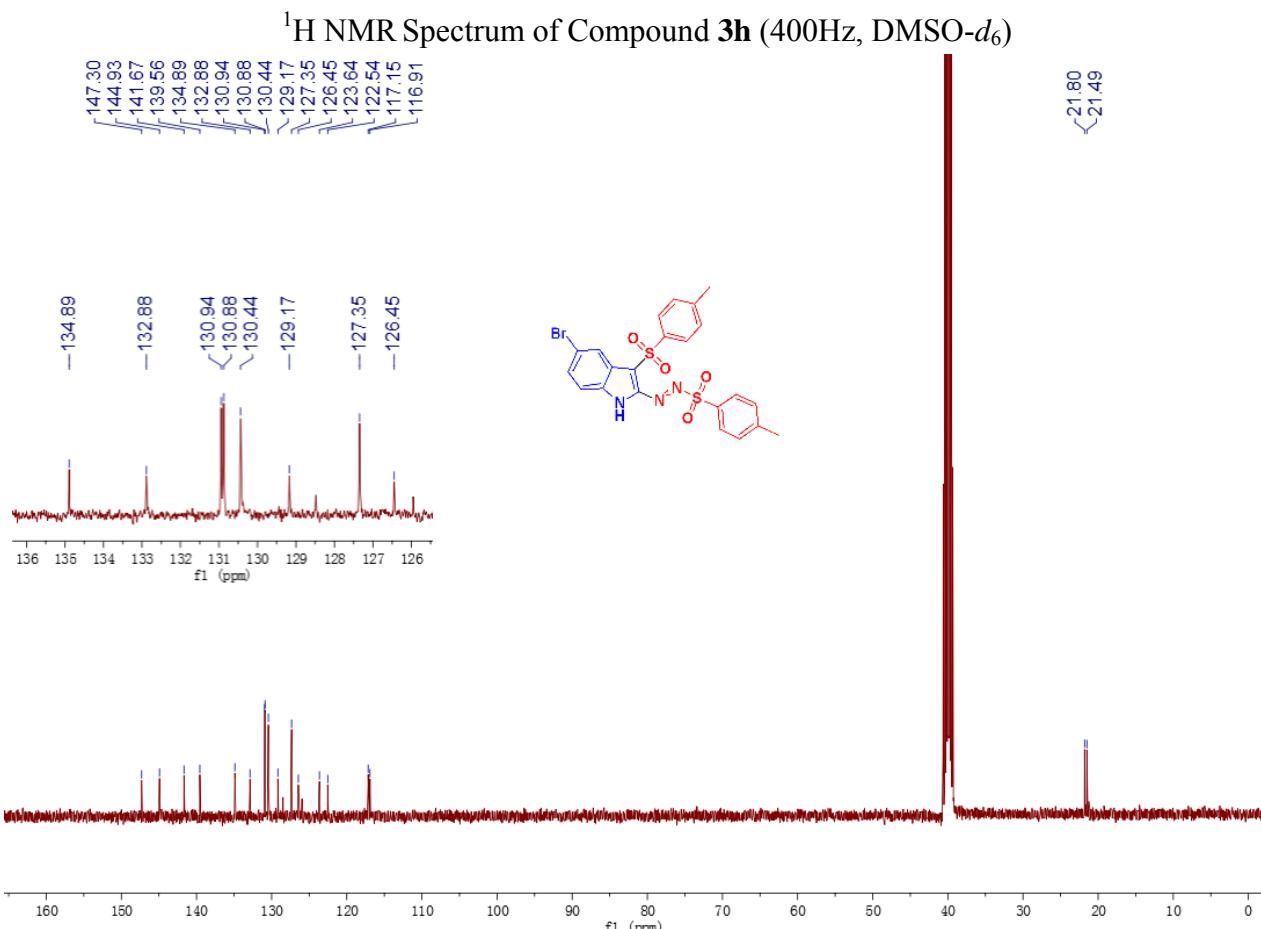
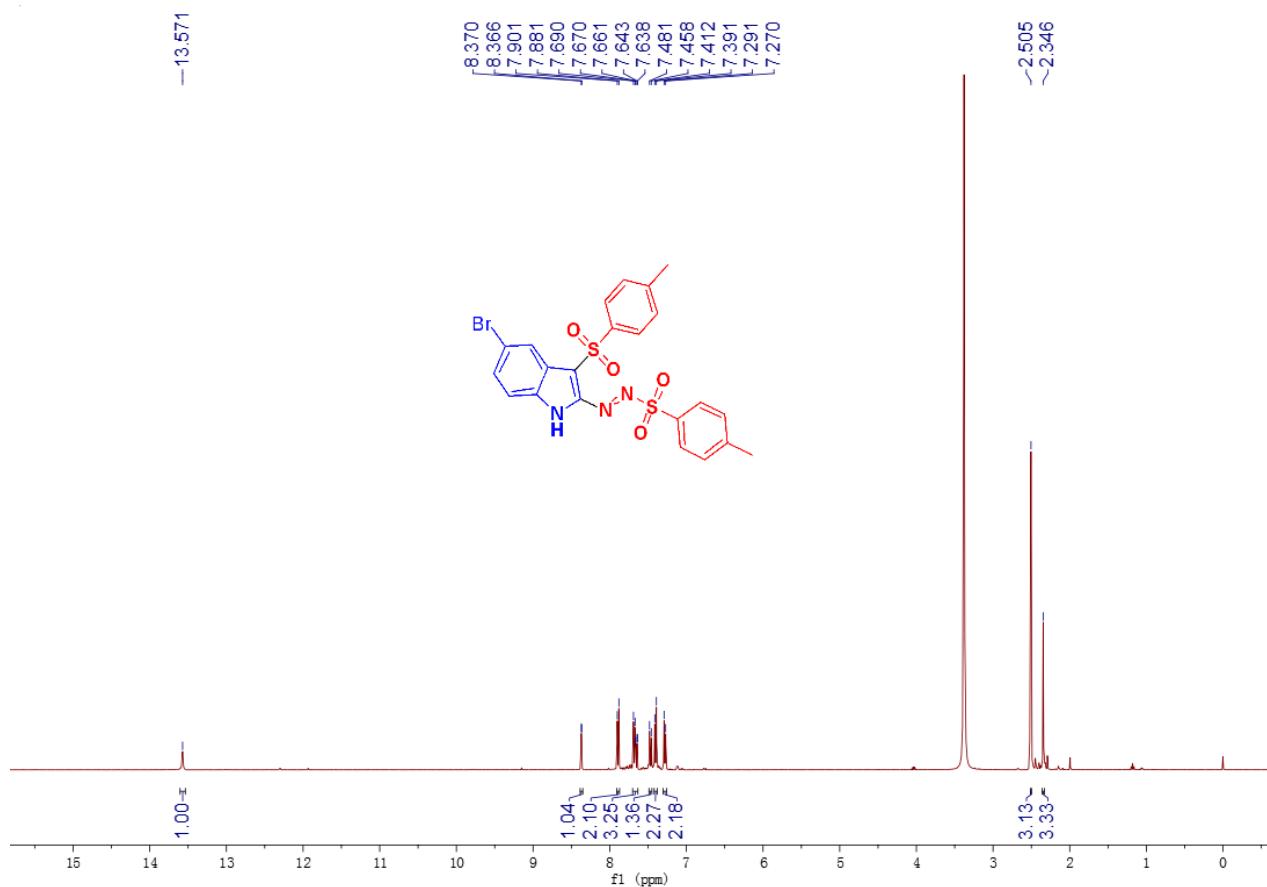


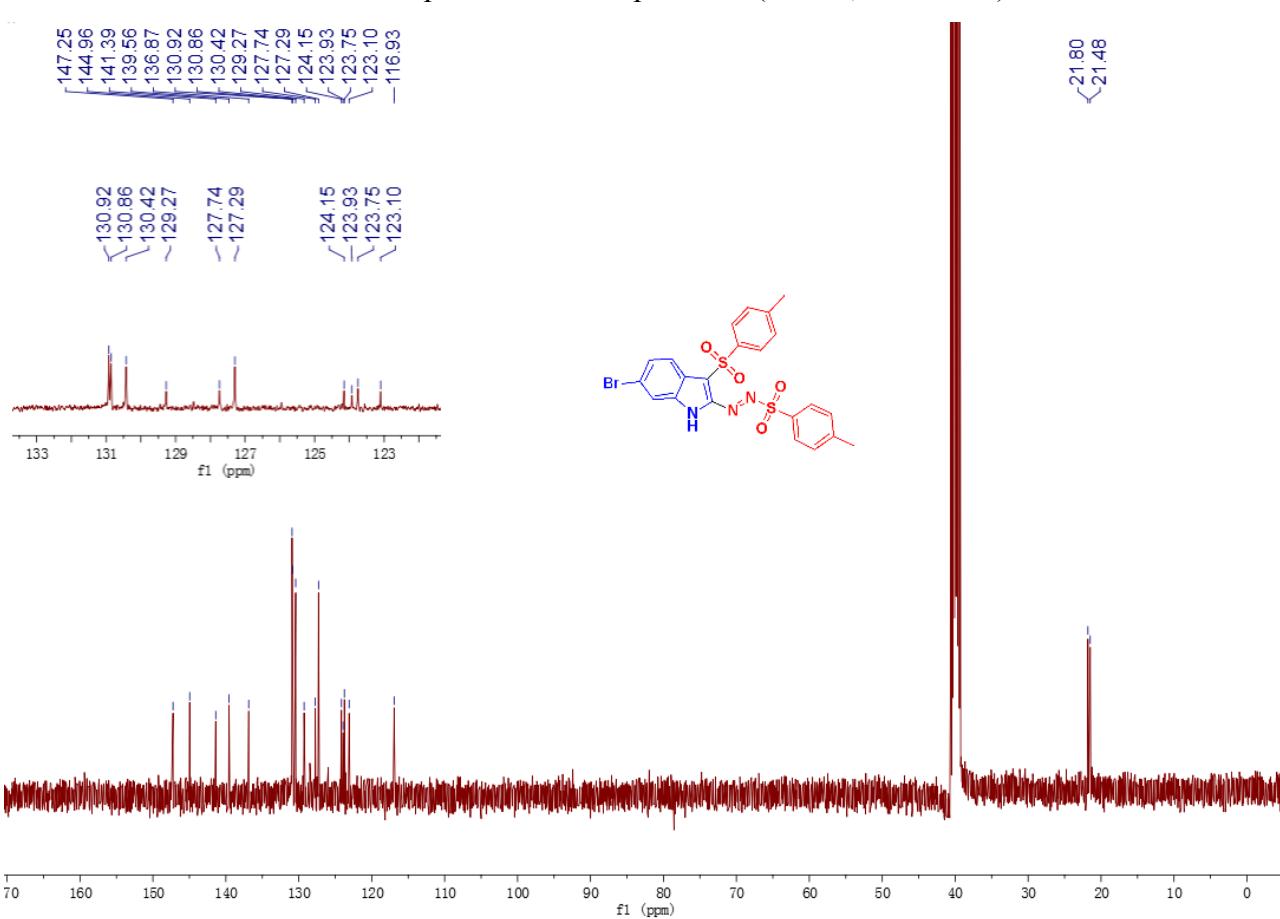
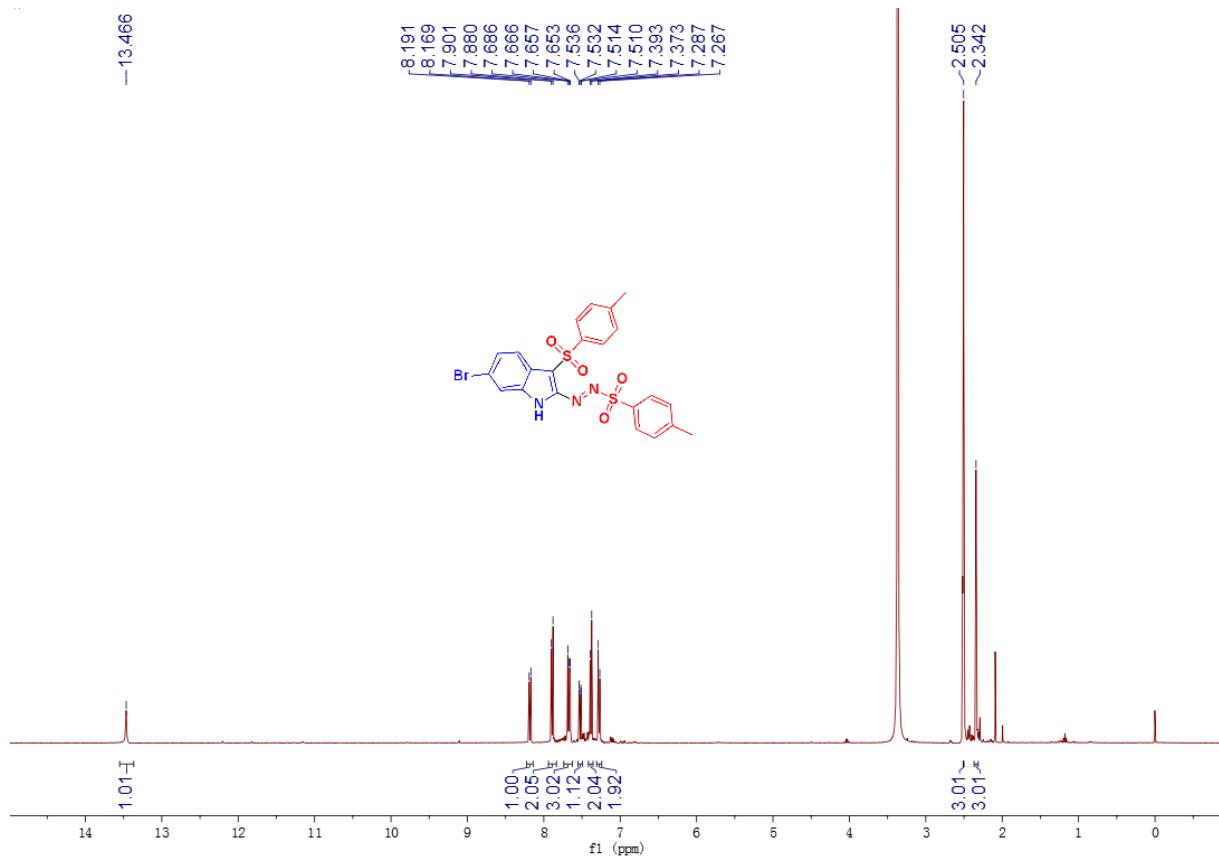


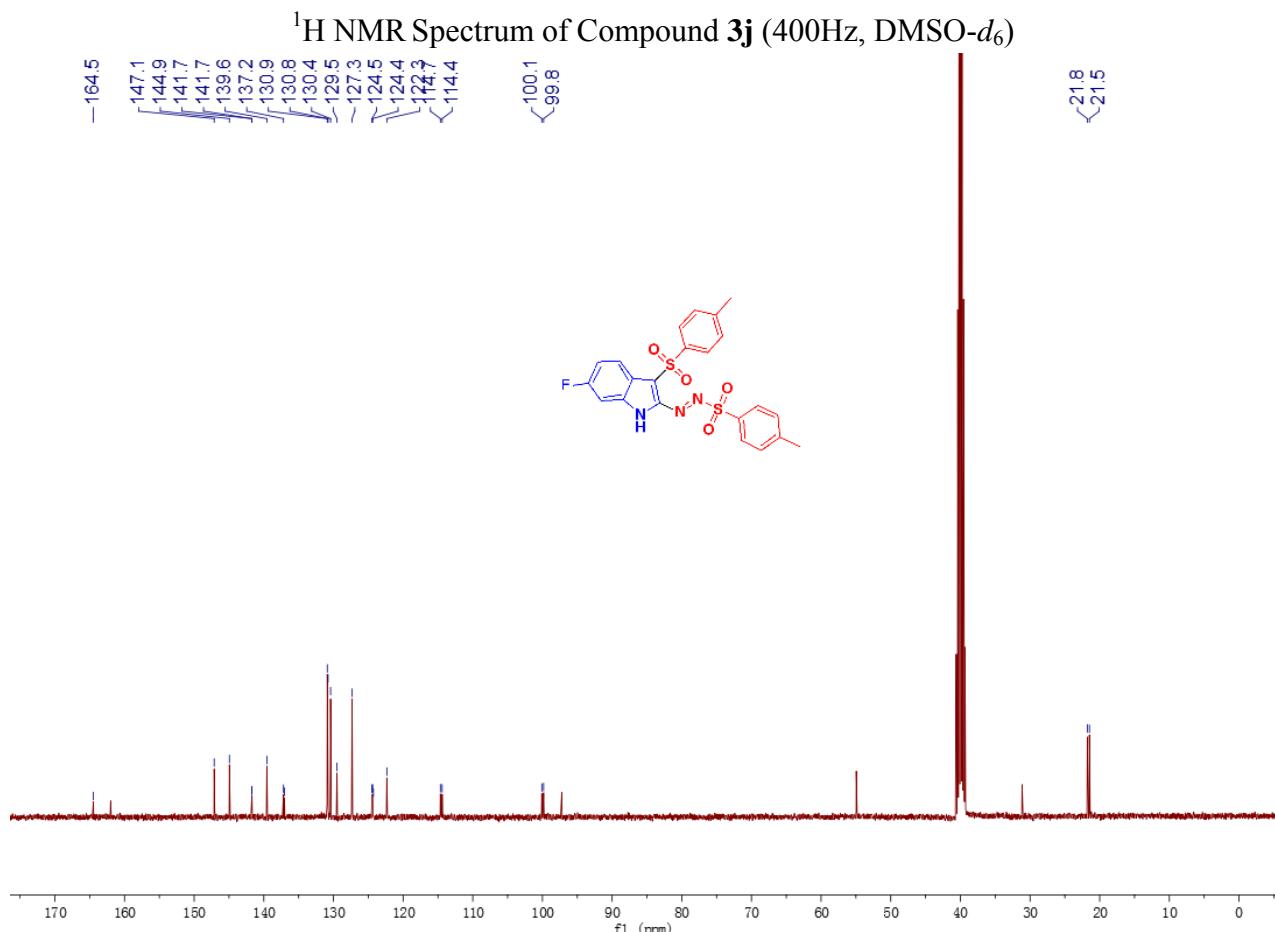
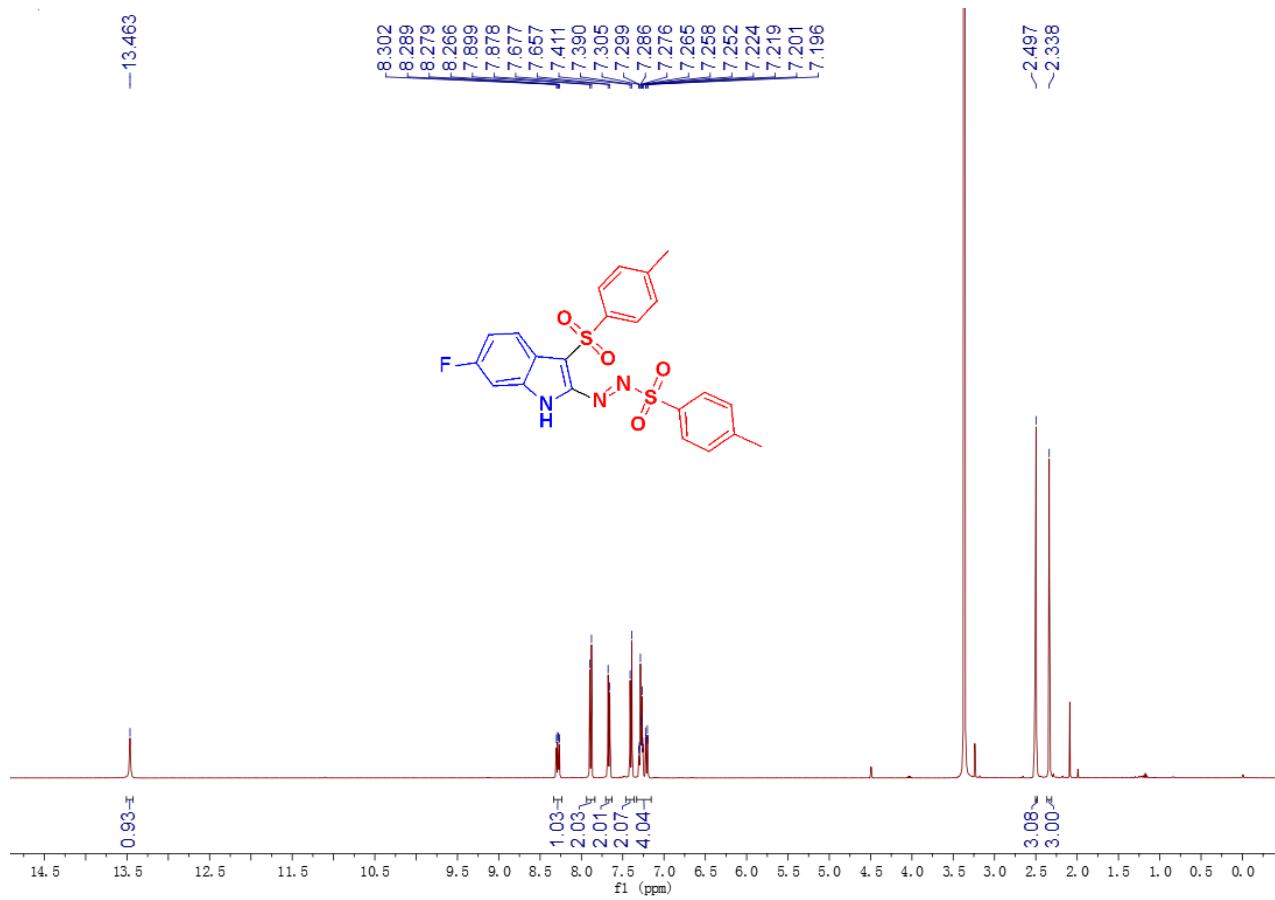
¹H NMR Spectrum of Compound 3g (400Hz, DMSO-*d*₆)



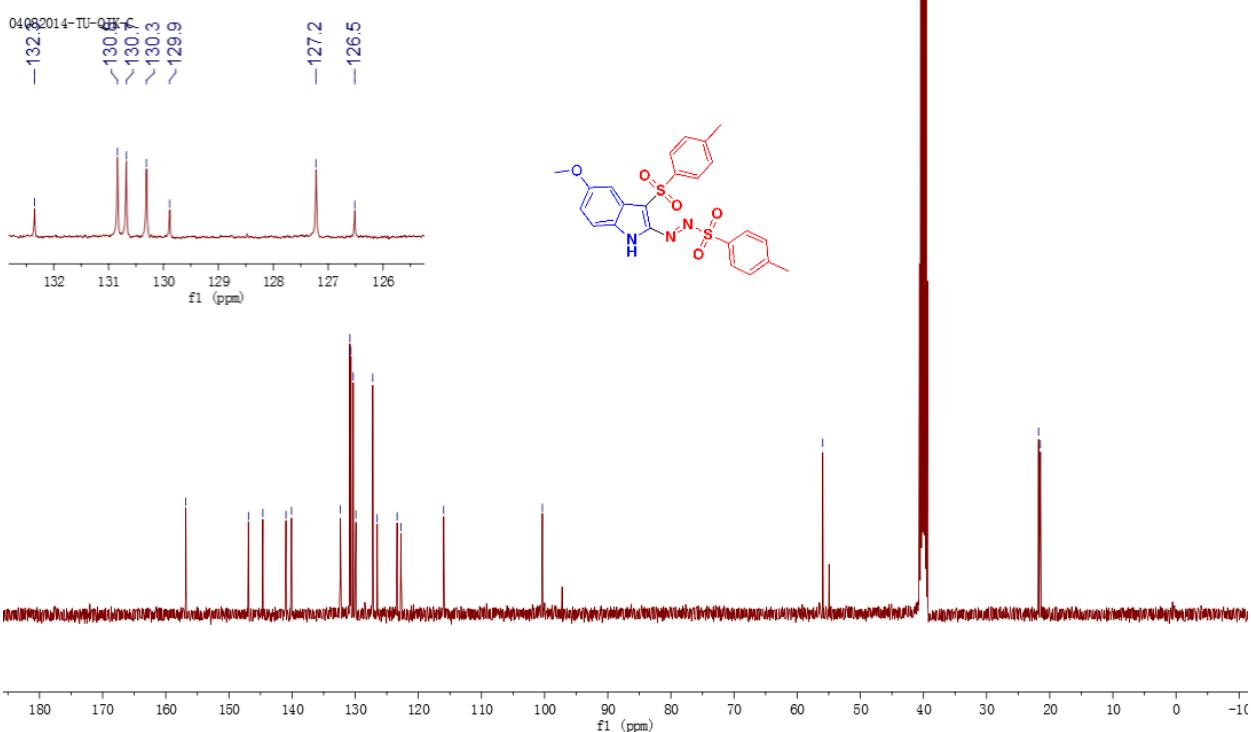
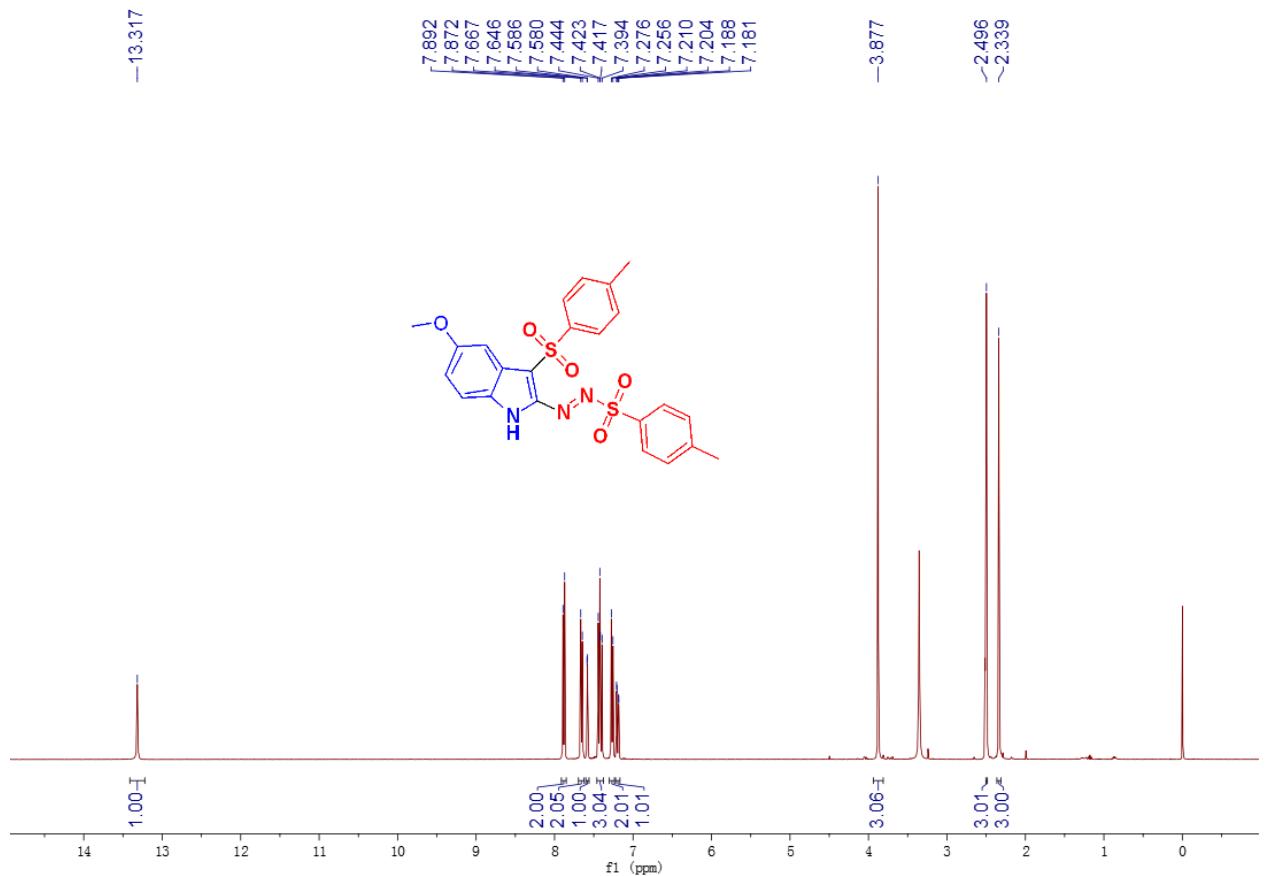
¹³C NMR Spectrum of Compound 3g (100Hz, DMSO-*d*₆)
S22



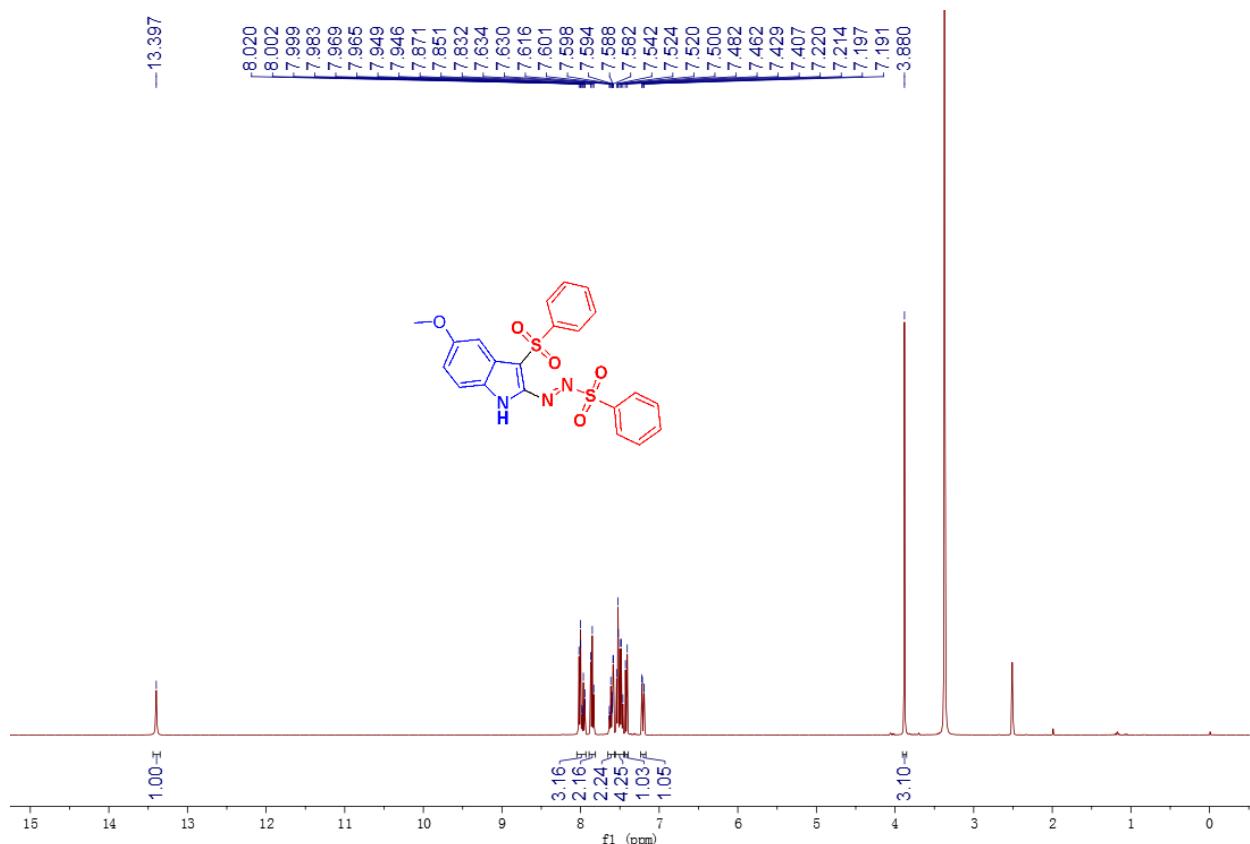




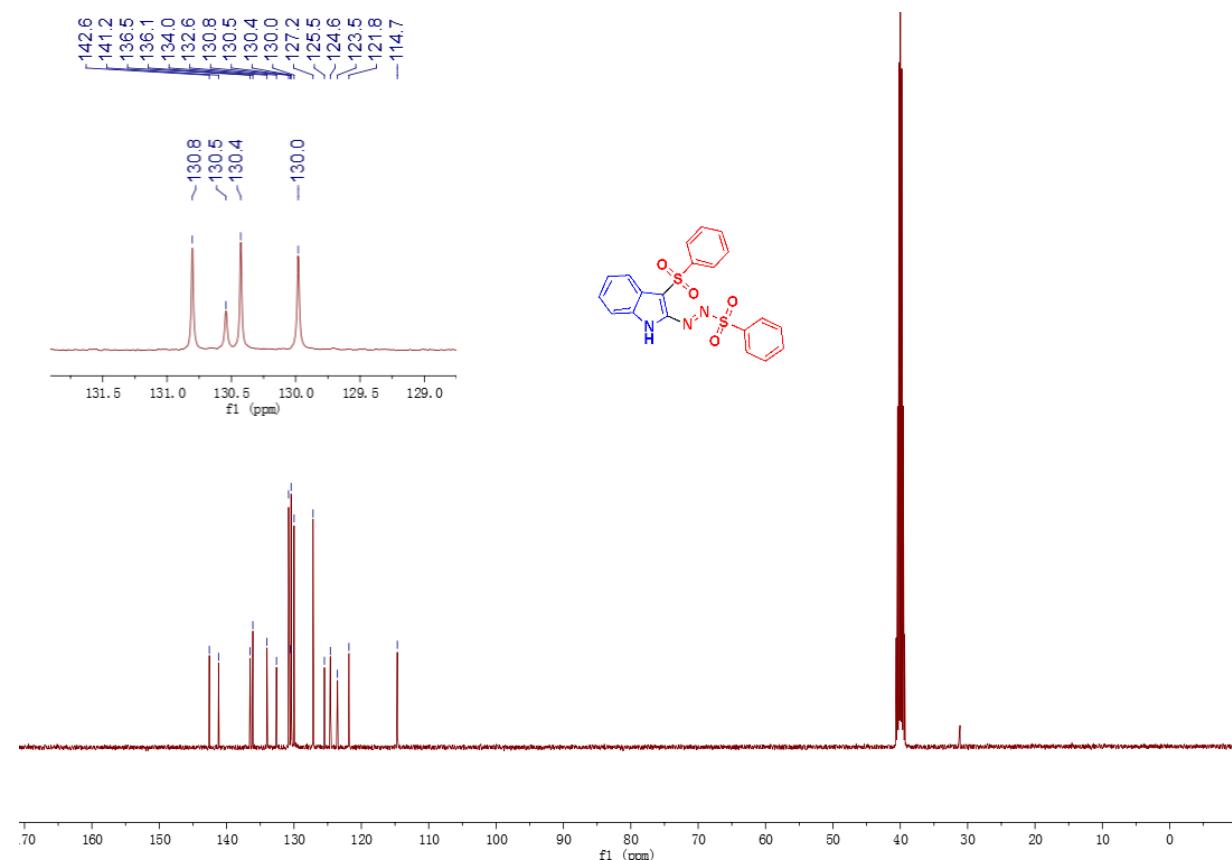
¹³C NMR Spectrum of Compound 3j (100Hz, DMSO-*d*₆)



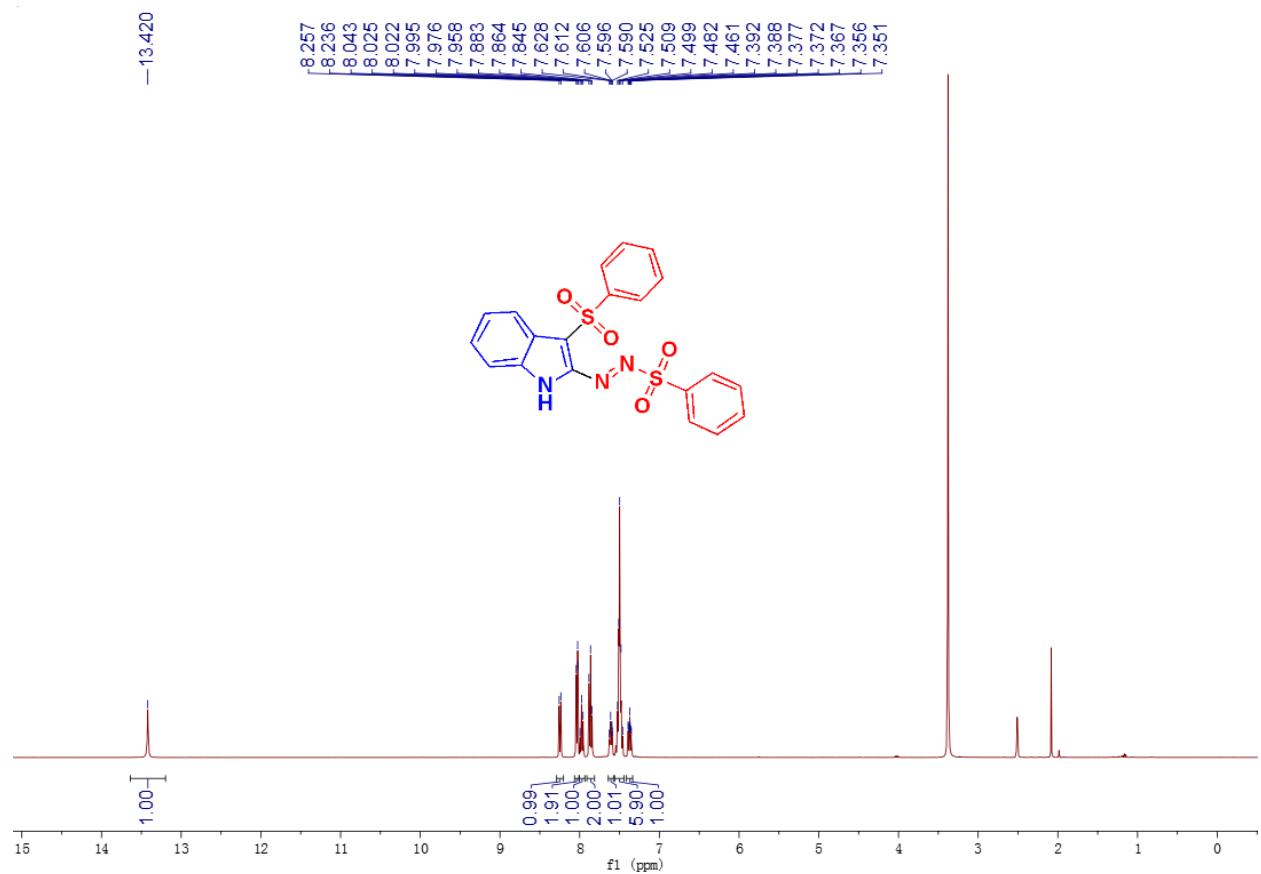
¹³C NMR Spectrum of Compound **3k** (100Hz, DMSO-*d*₆)
S26



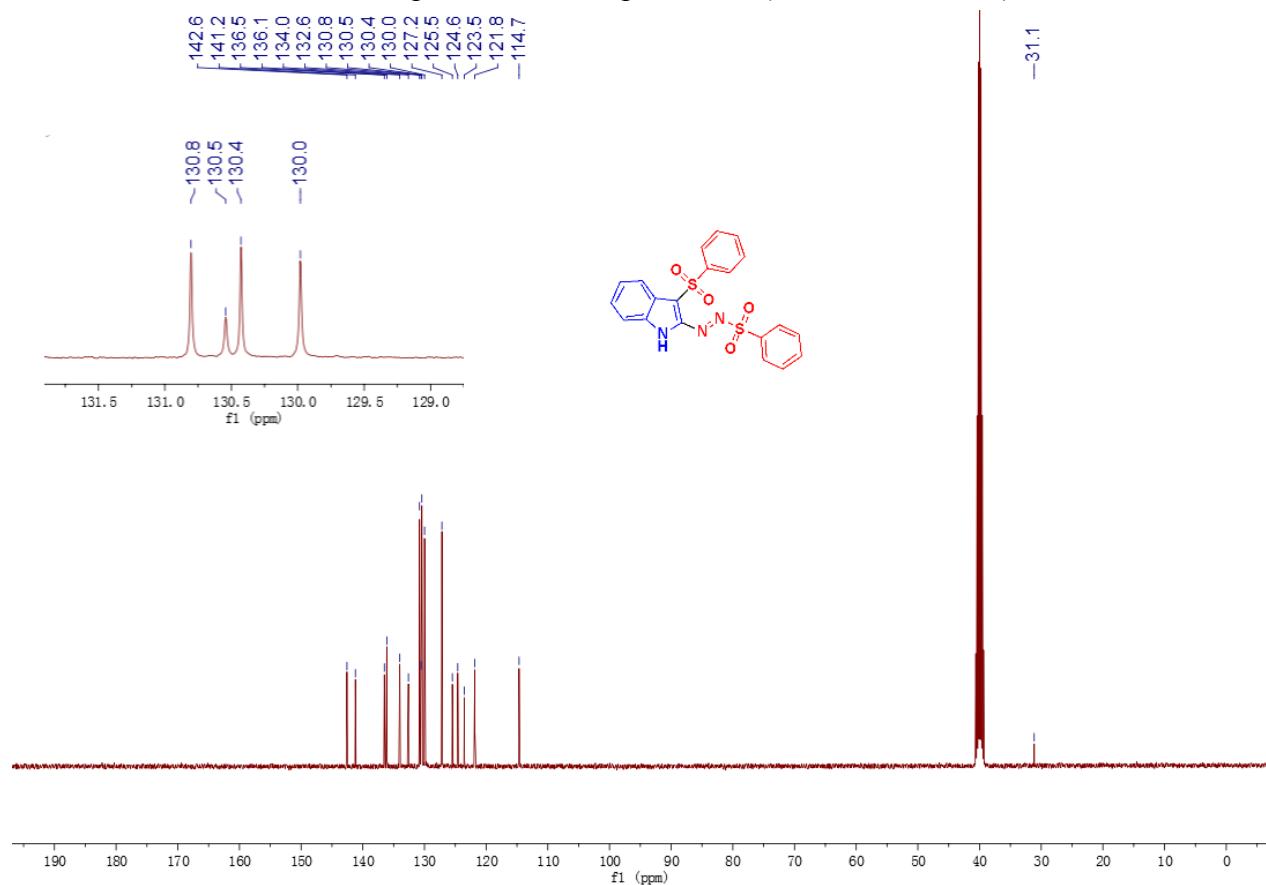
¹H NMR Spectrum of Compound 3l (400Hz, DMSO-*d*₆)



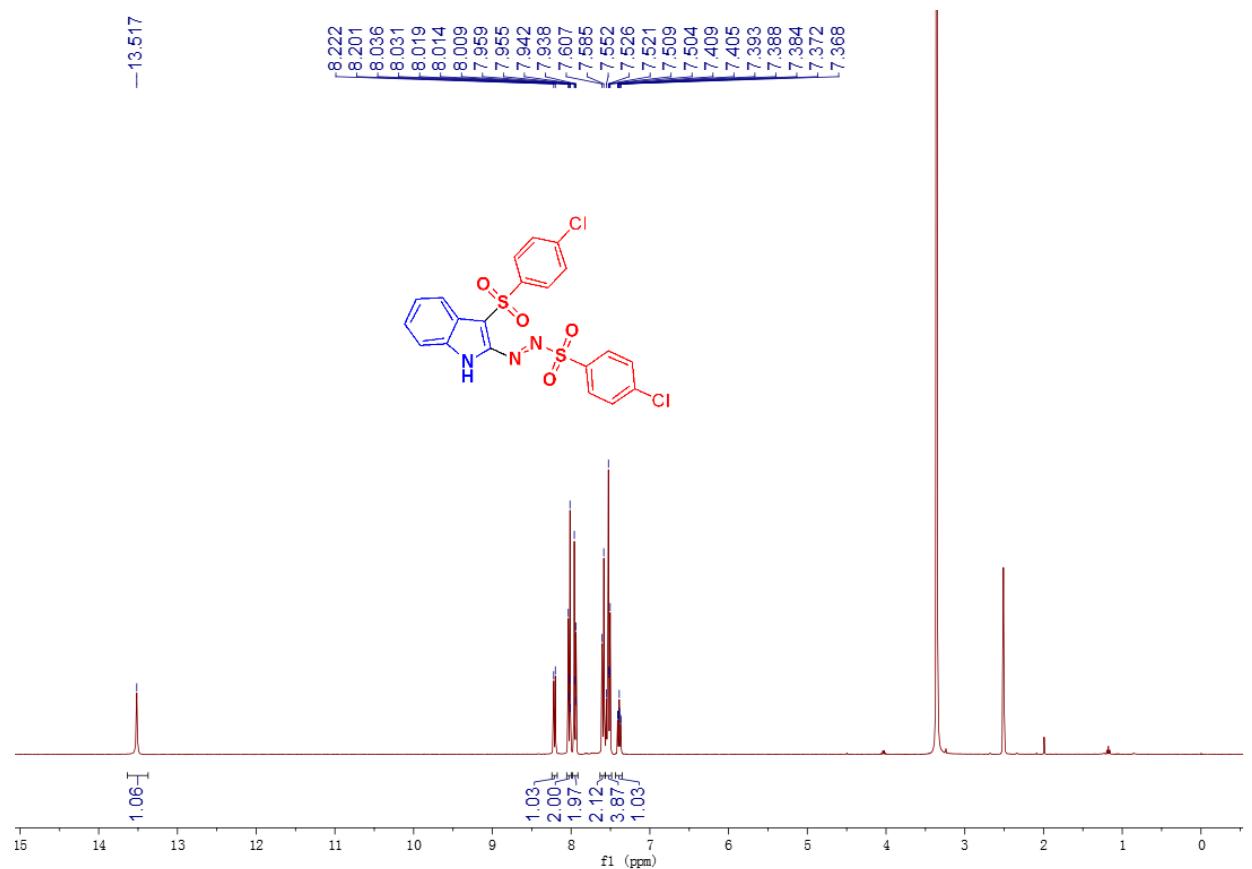
¹³C NMR Spectrum of Compound 3l (100Hz, DMSO-*d*₆)



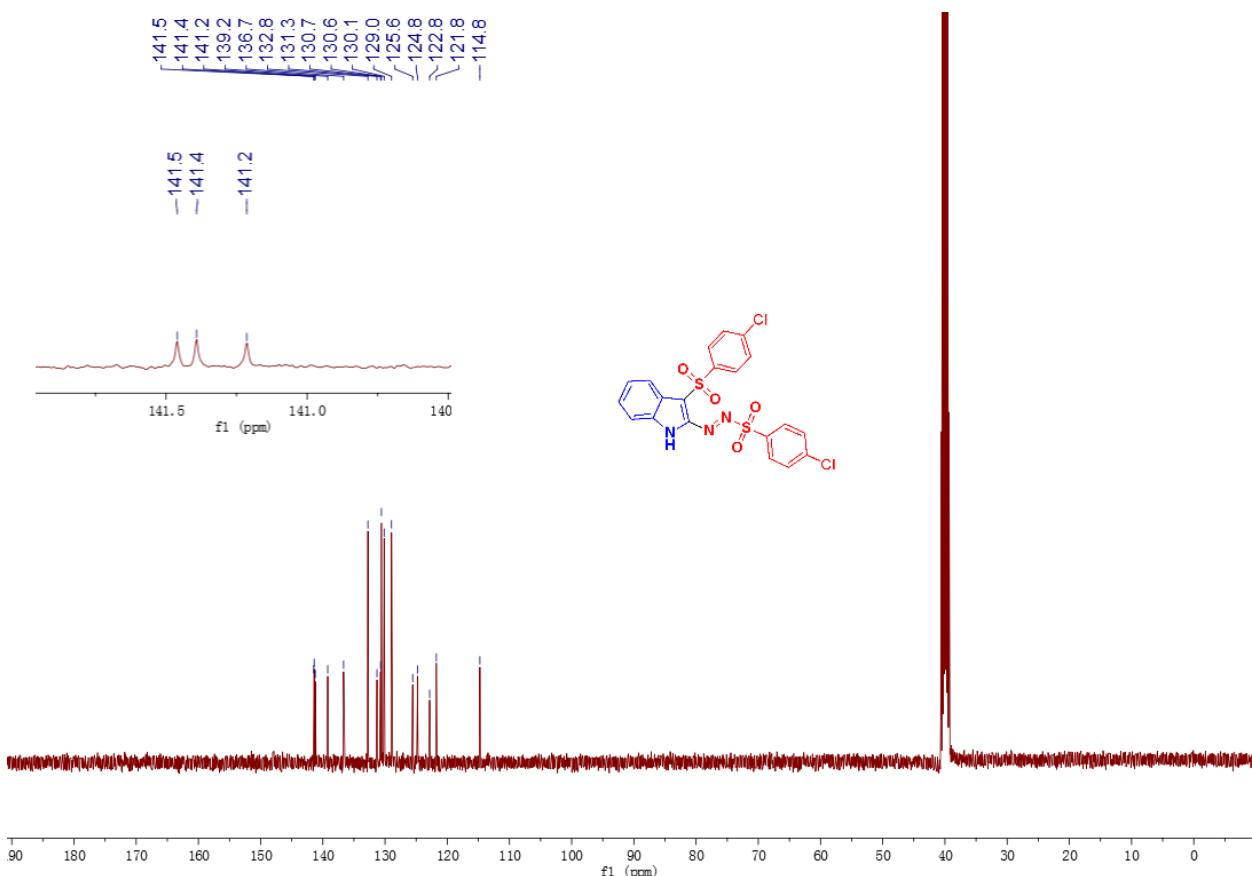
^1H NMR Spectrum of Compound **3m** (400Hz, $\text{DMSO}-d_6$)



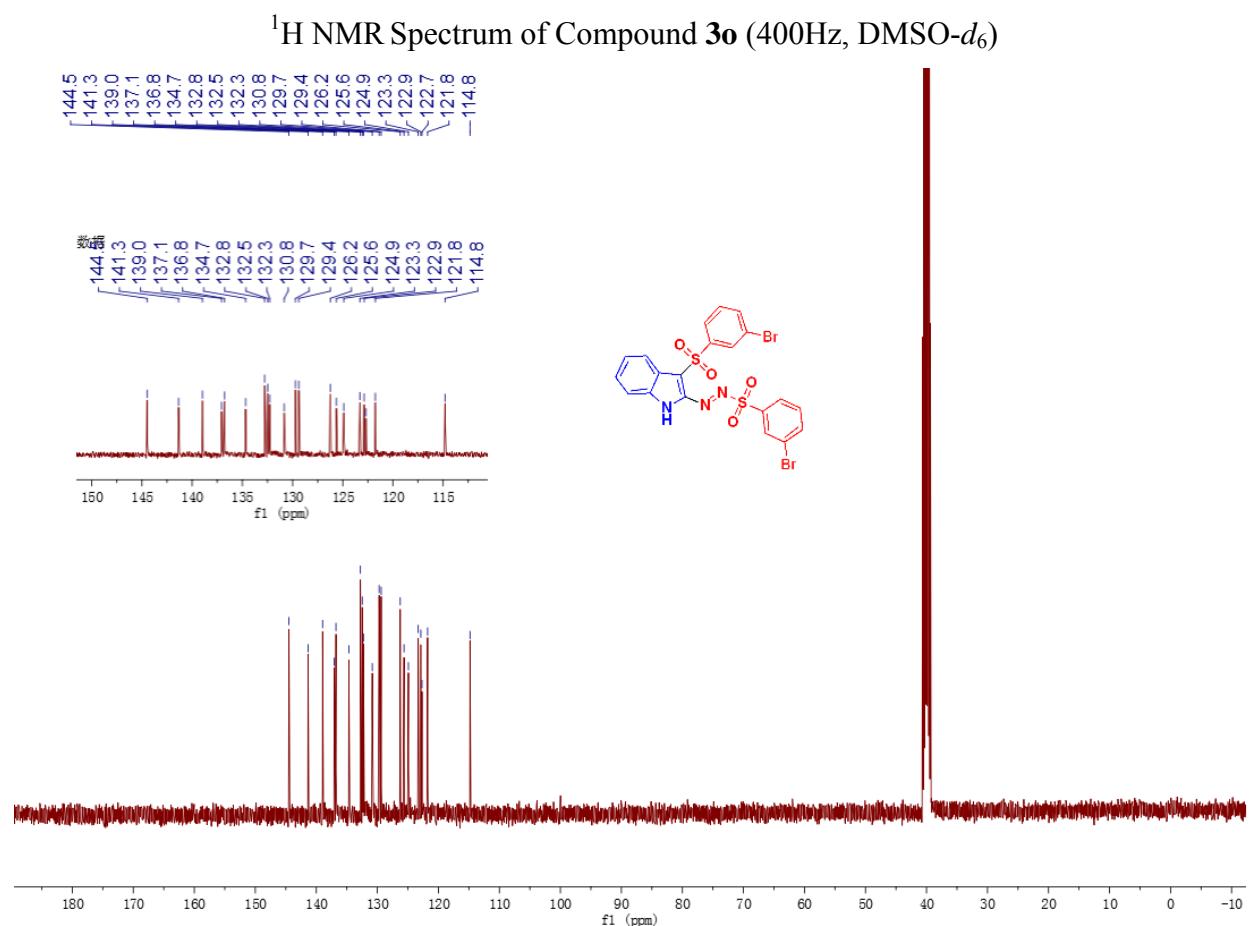
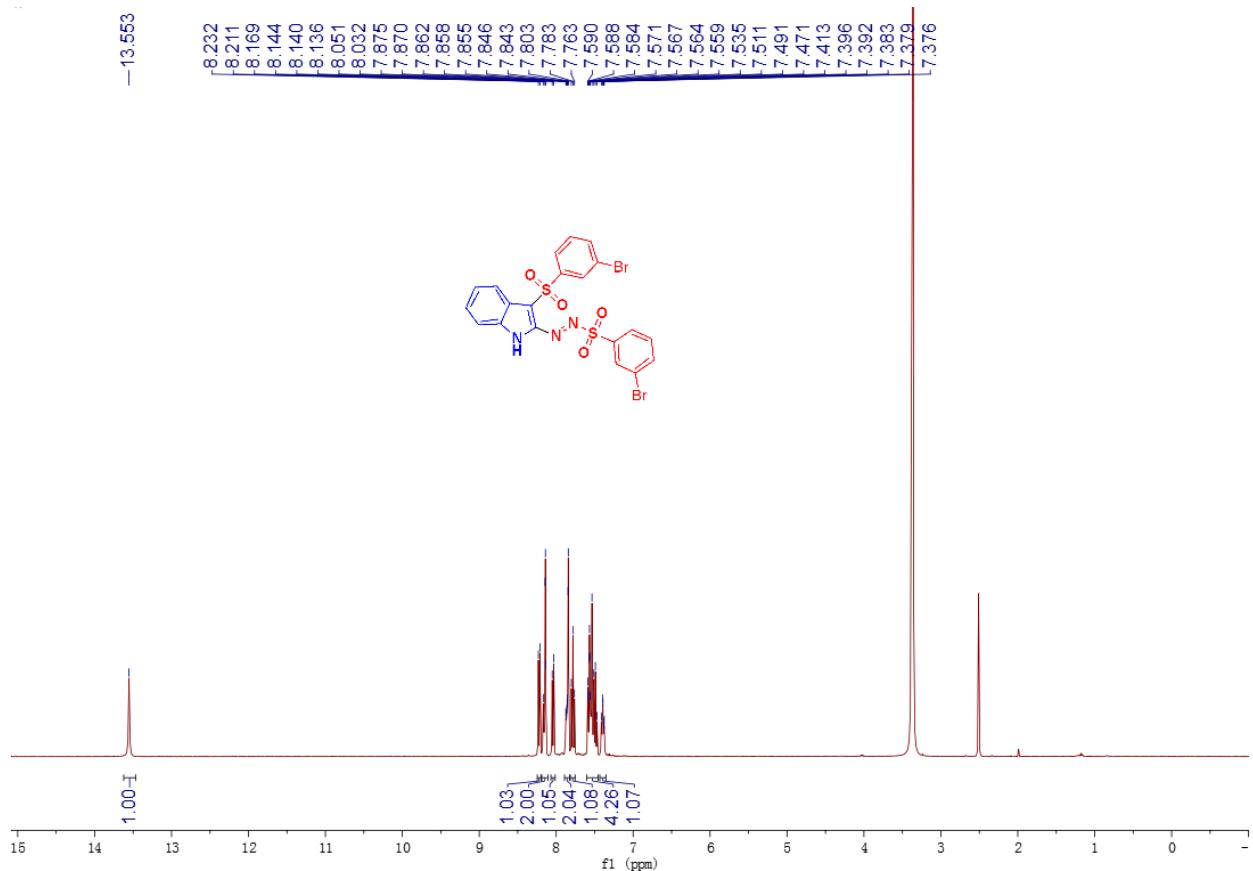
^{13}C NMR Spectrum of Compound **3m** (100Hz, $\text{DMSO}-d_6$)

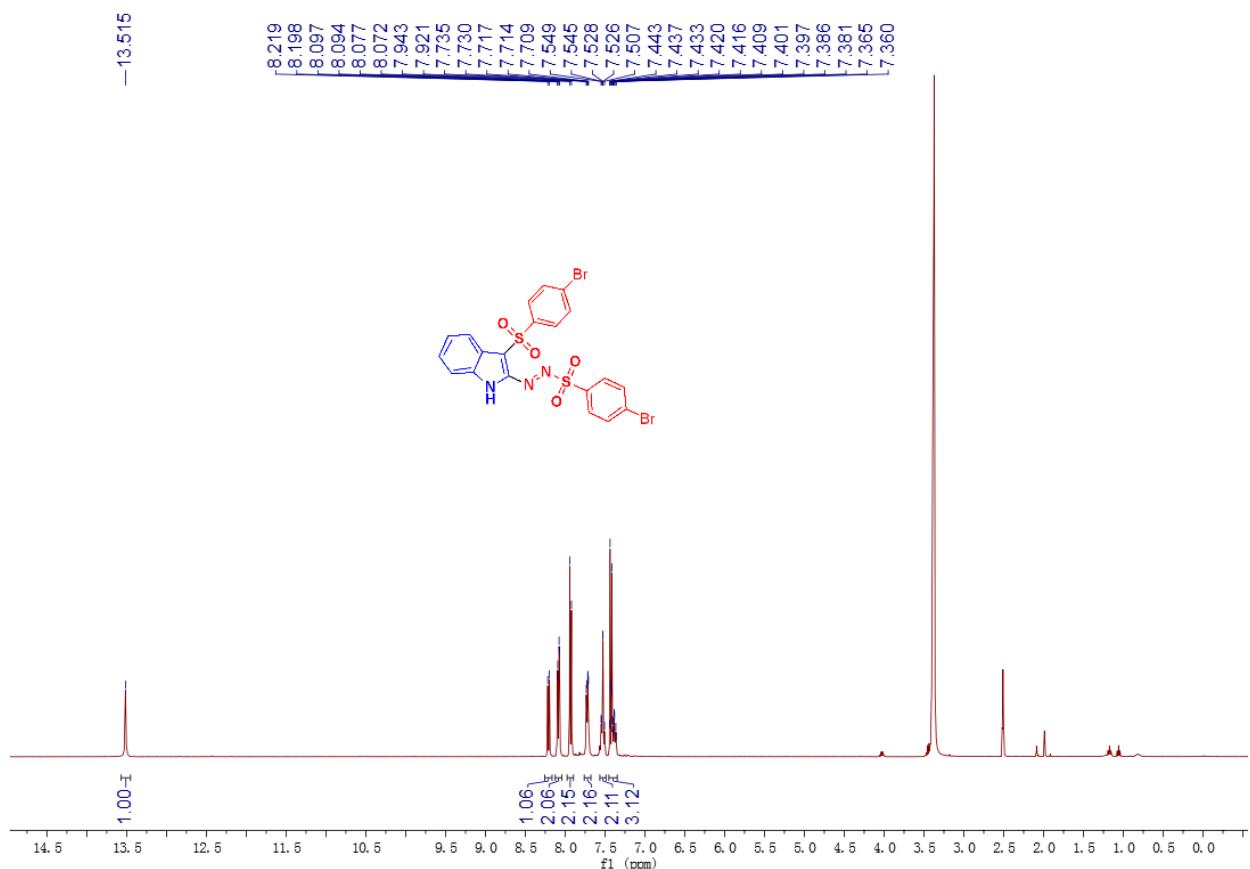


¹H NMR Spectrum of Compound 3n (400Hz, DMSO-*d*₆)

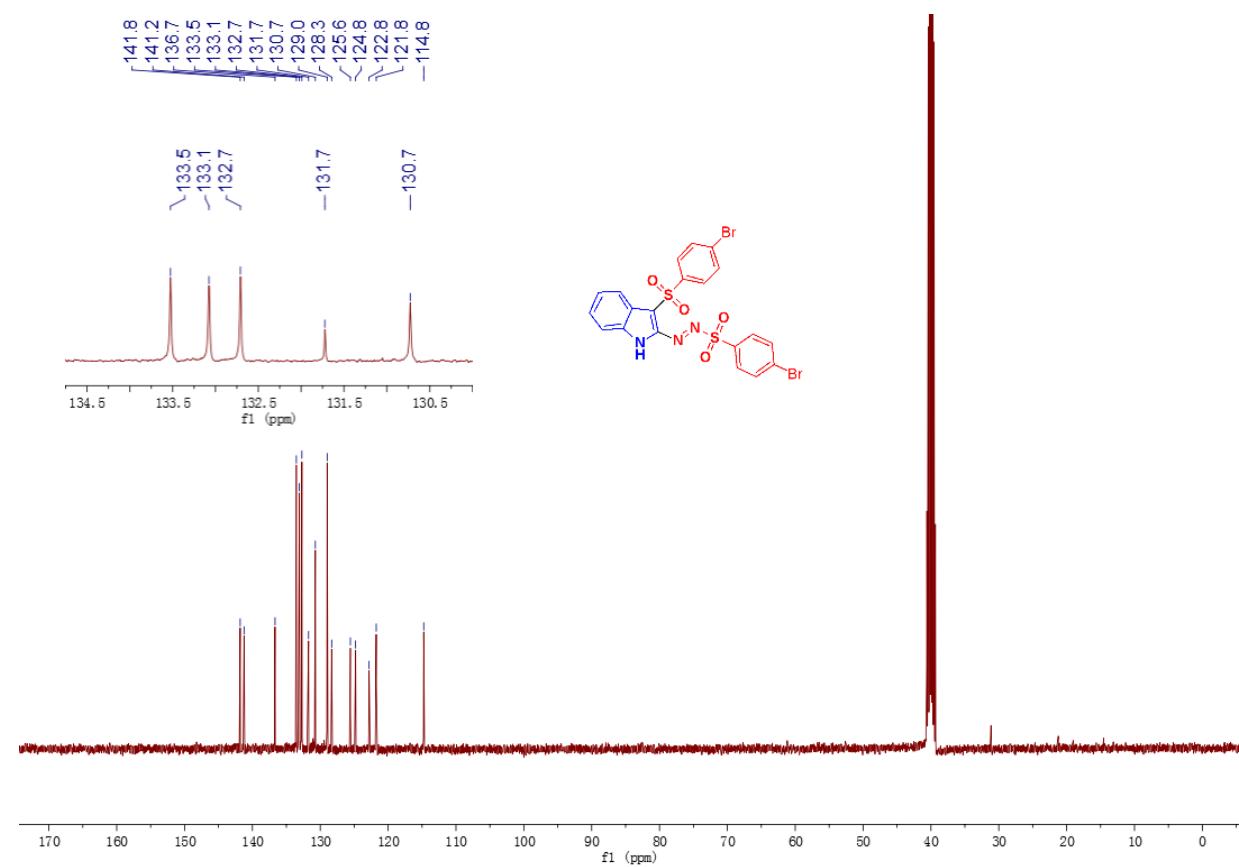


¹³C NMR Spectrum of Compound 3n (100Hz, DMSO-*d*₆)

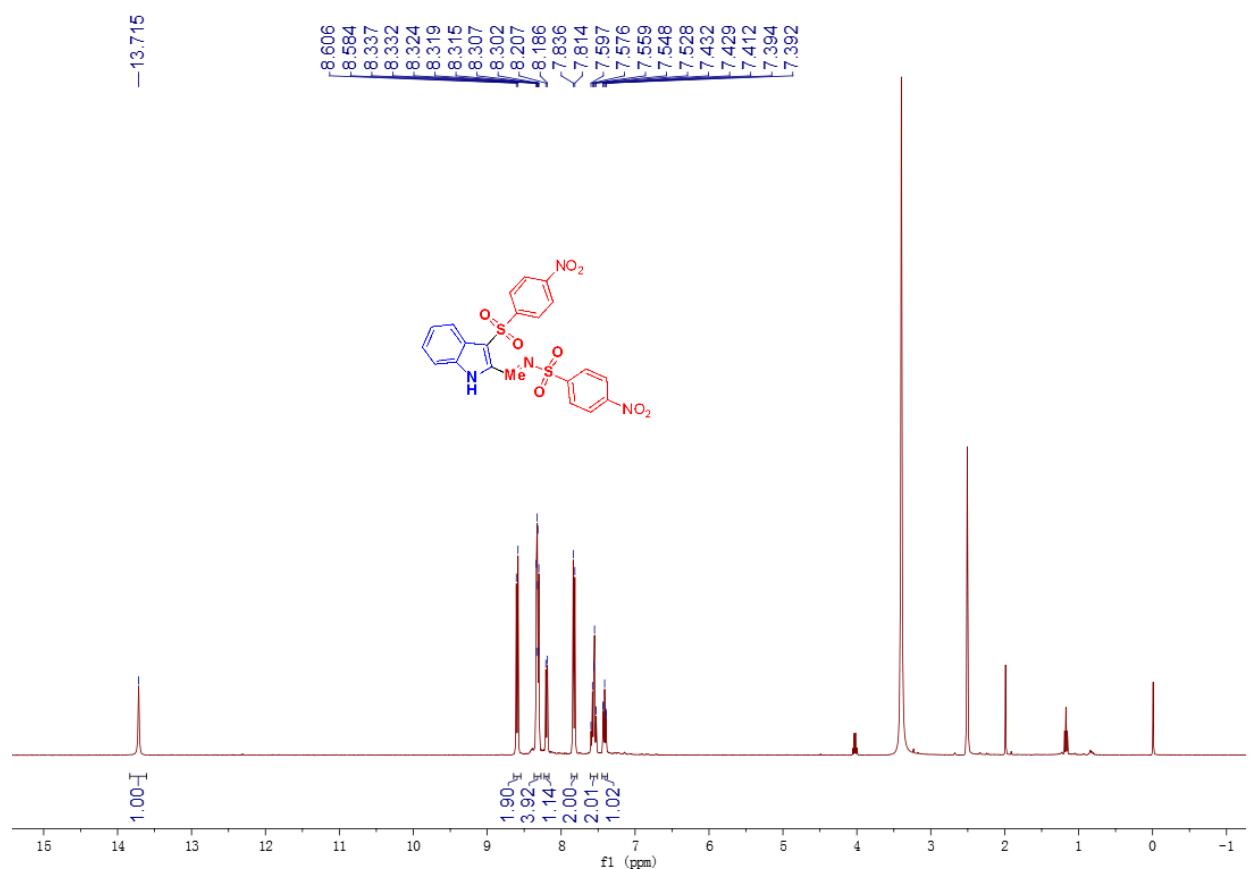




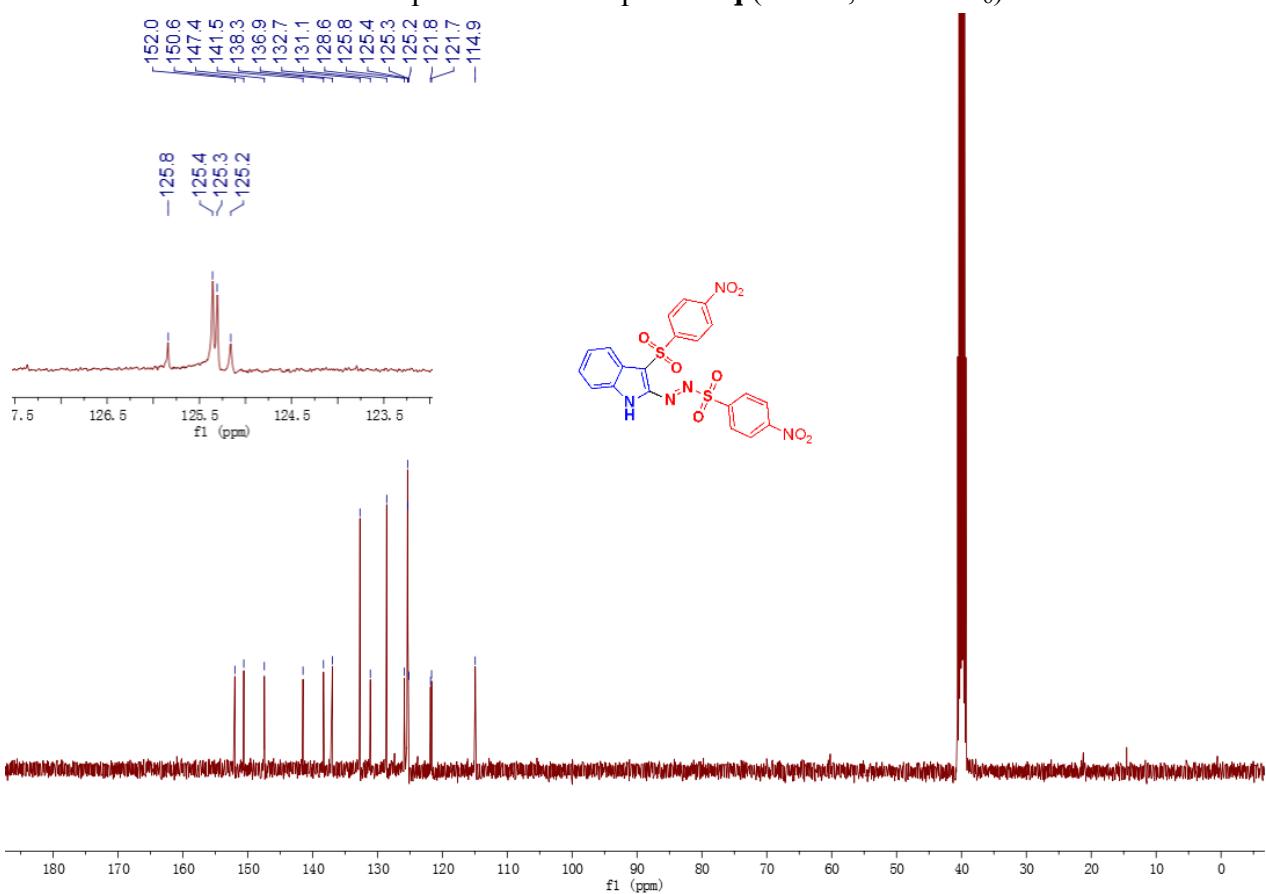
^1H NMR Spectrum of Compound **3p** (400Hz, $\text{DMSO}-d_6$)



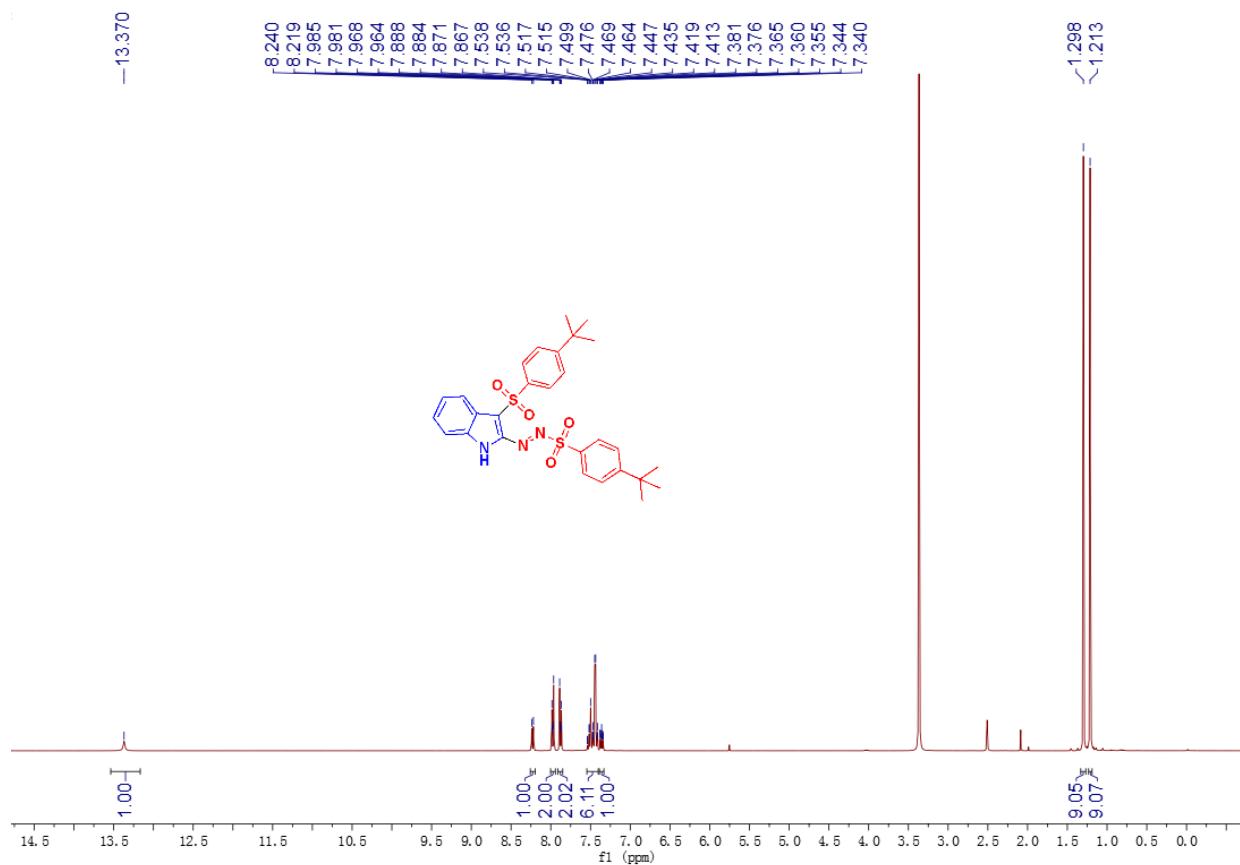
^{13}C NMR Spectrum of Compound **3p** (100Hz, $\text{DMSO}-d_6$)
S31



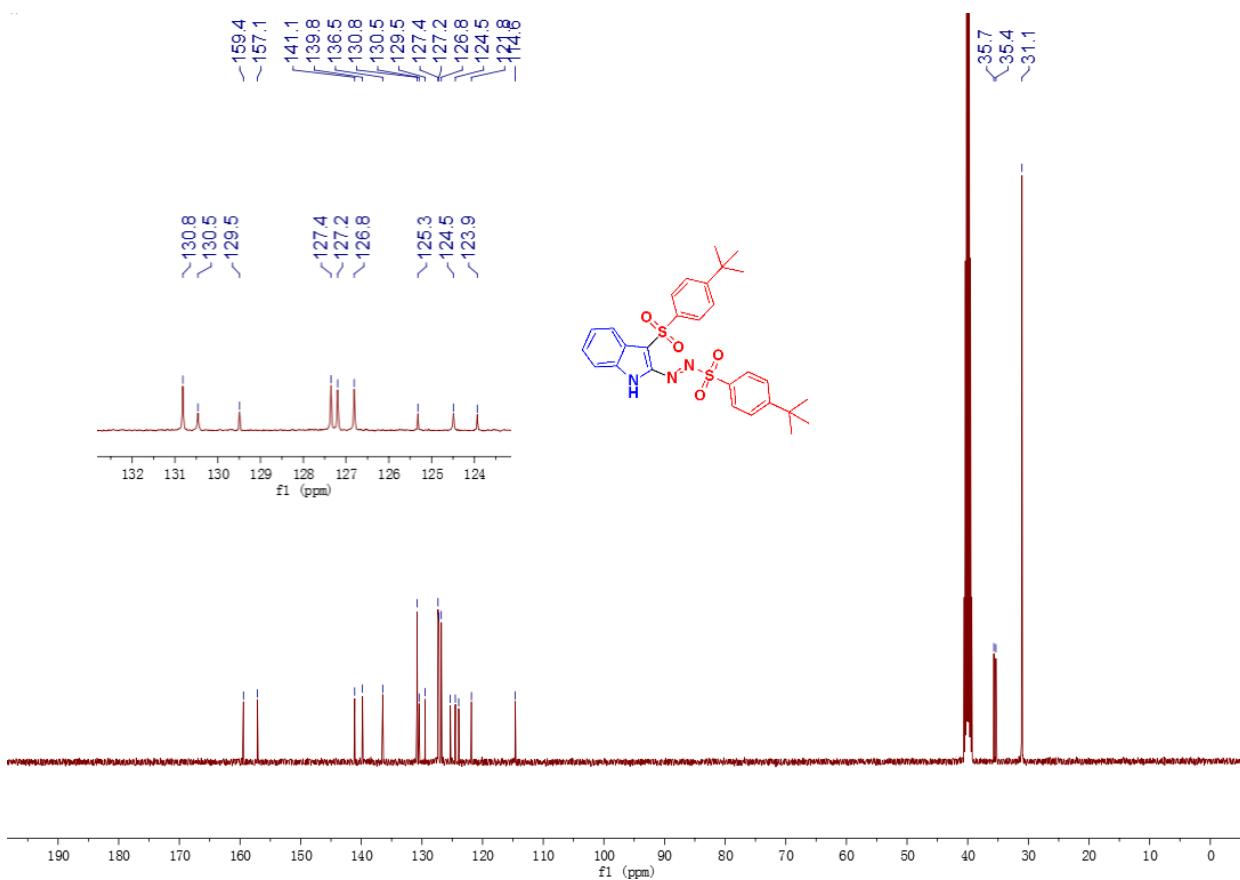
¹H NMR Spectrum of Compound 3q (400Hz, DMSO-*d*₆)



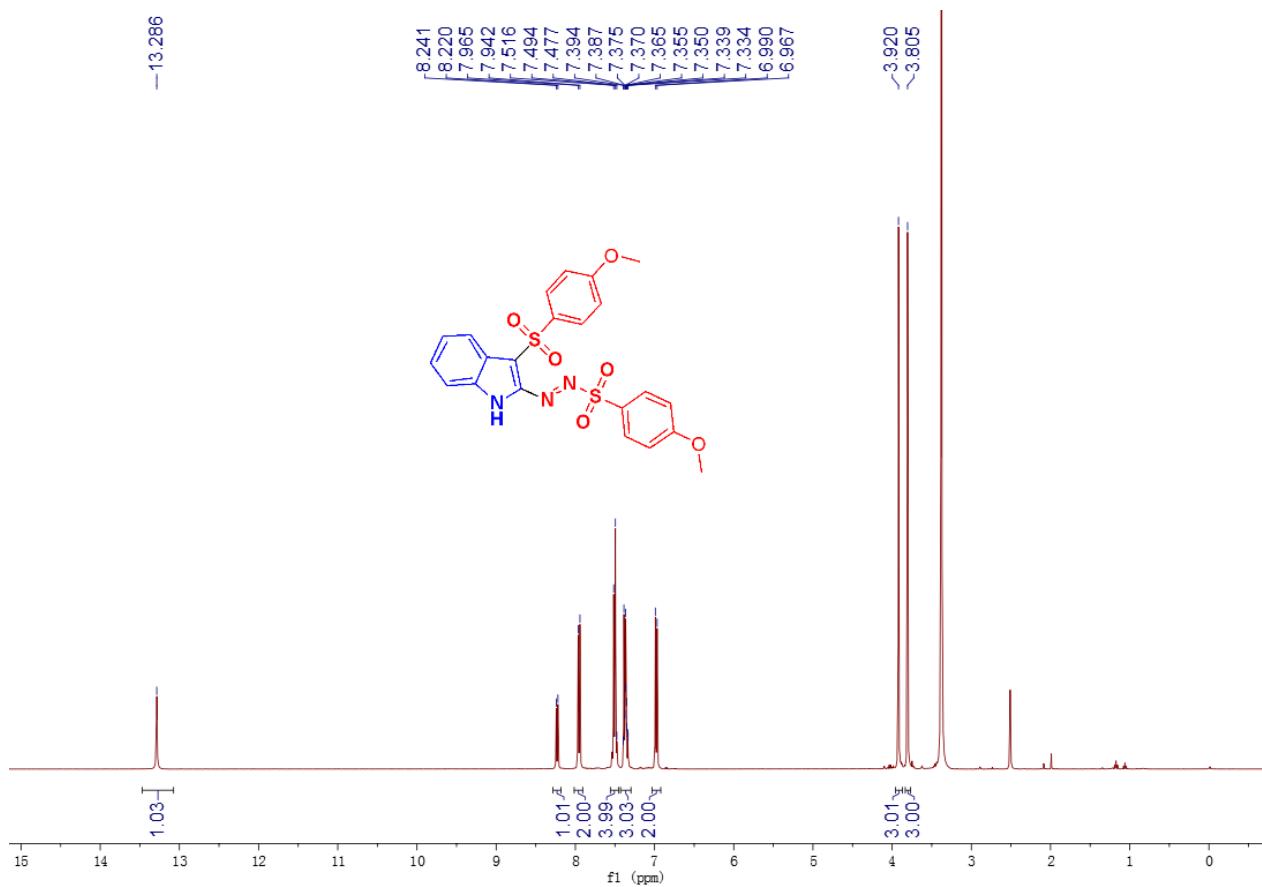
¹³C NMR Spectrum of Compound **3q** (100Hz, DMSO-*d*₆)



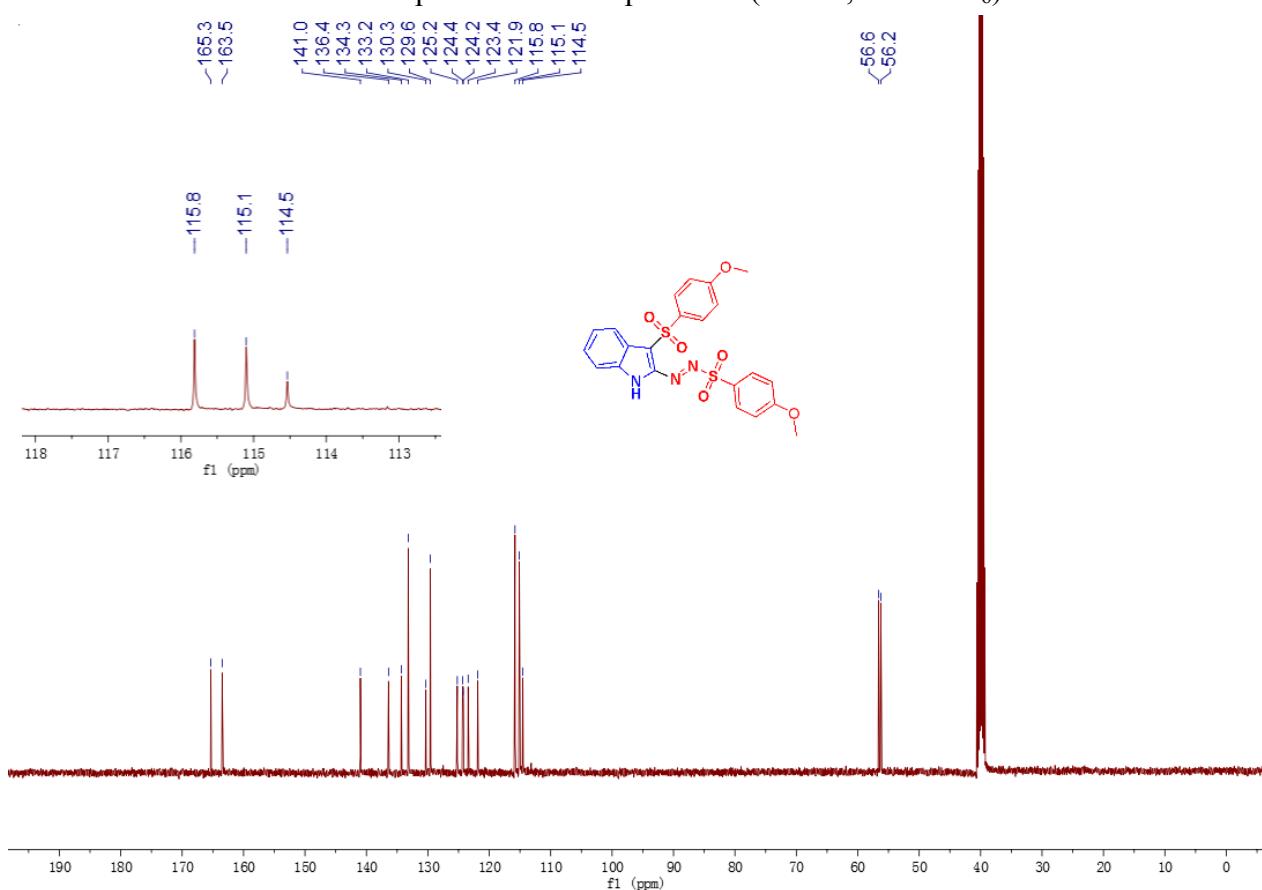
¹H NMR Spectrum of Compound **3r** (400Hz, DMSO-*d*₆)



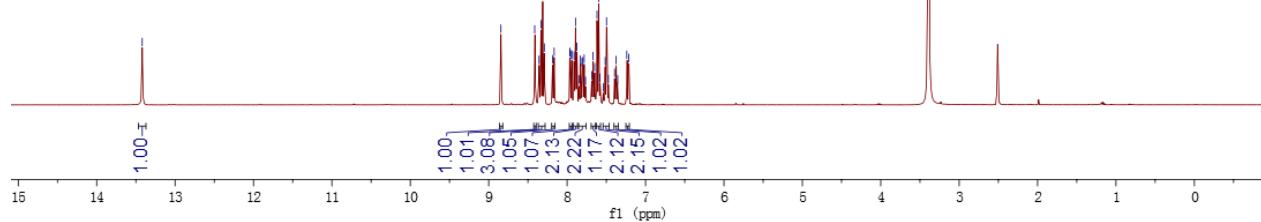
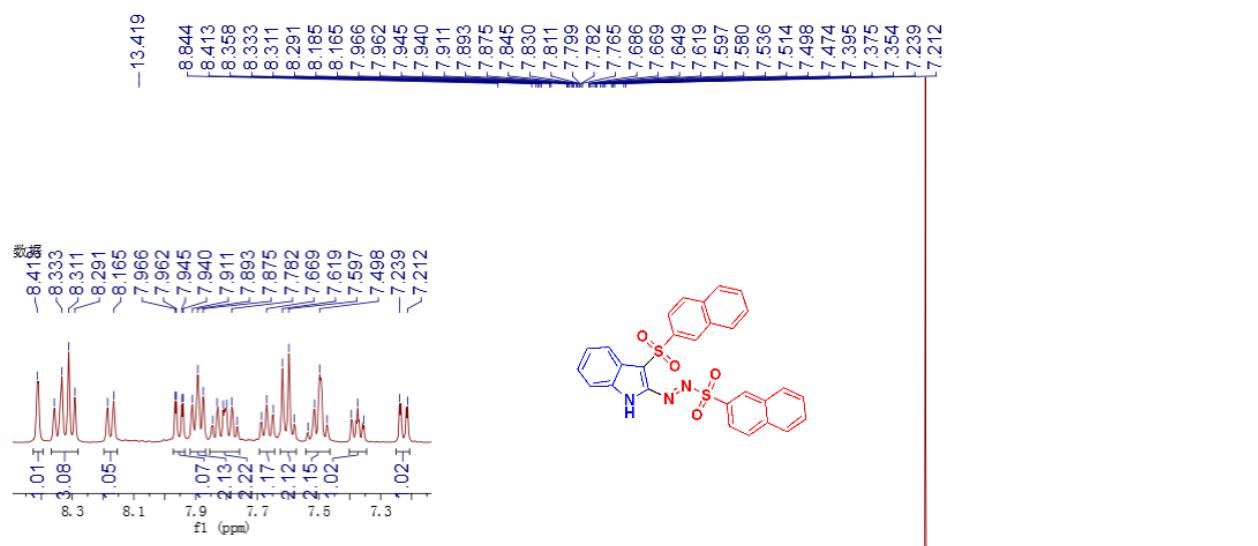
¹³C NMR Spectrum of Compound **3r** (100Hz, DMSO-*d*₆)
S33



^1H NMR Spectrum of Compound 3s (400Hz, $\text{DMSO}-d_6$)

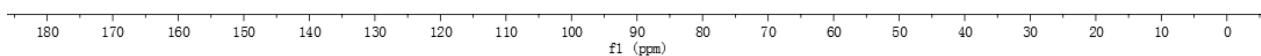
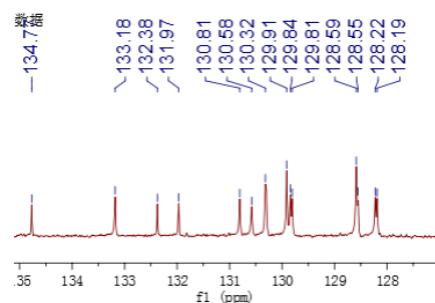


^{13}C NMR Spectrum of Compound 3s (100Hz, $\text{DMSO}-d_6$)

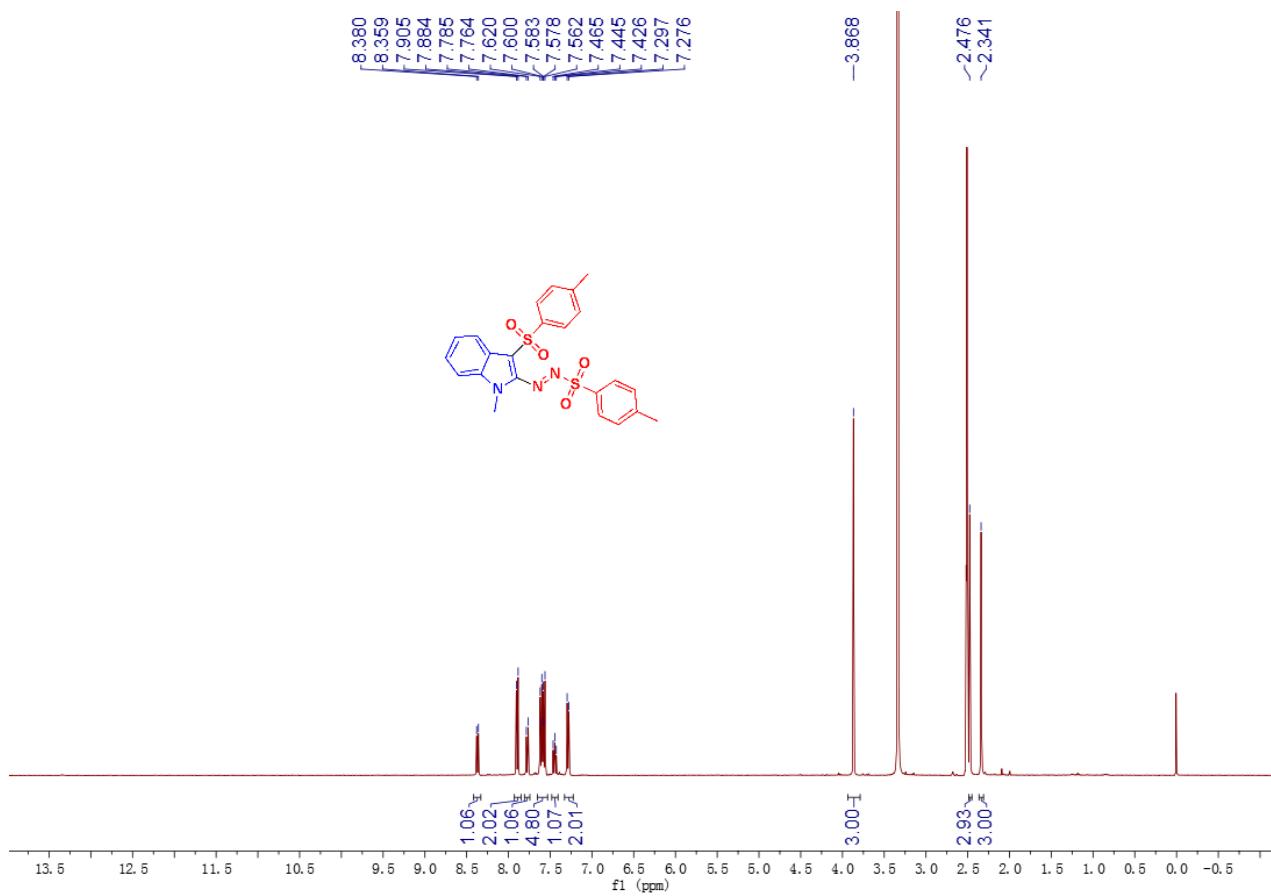


^1H NMR Spectrum of Compound **3t** (400Hz, $\text{DMSO}-d_6$)

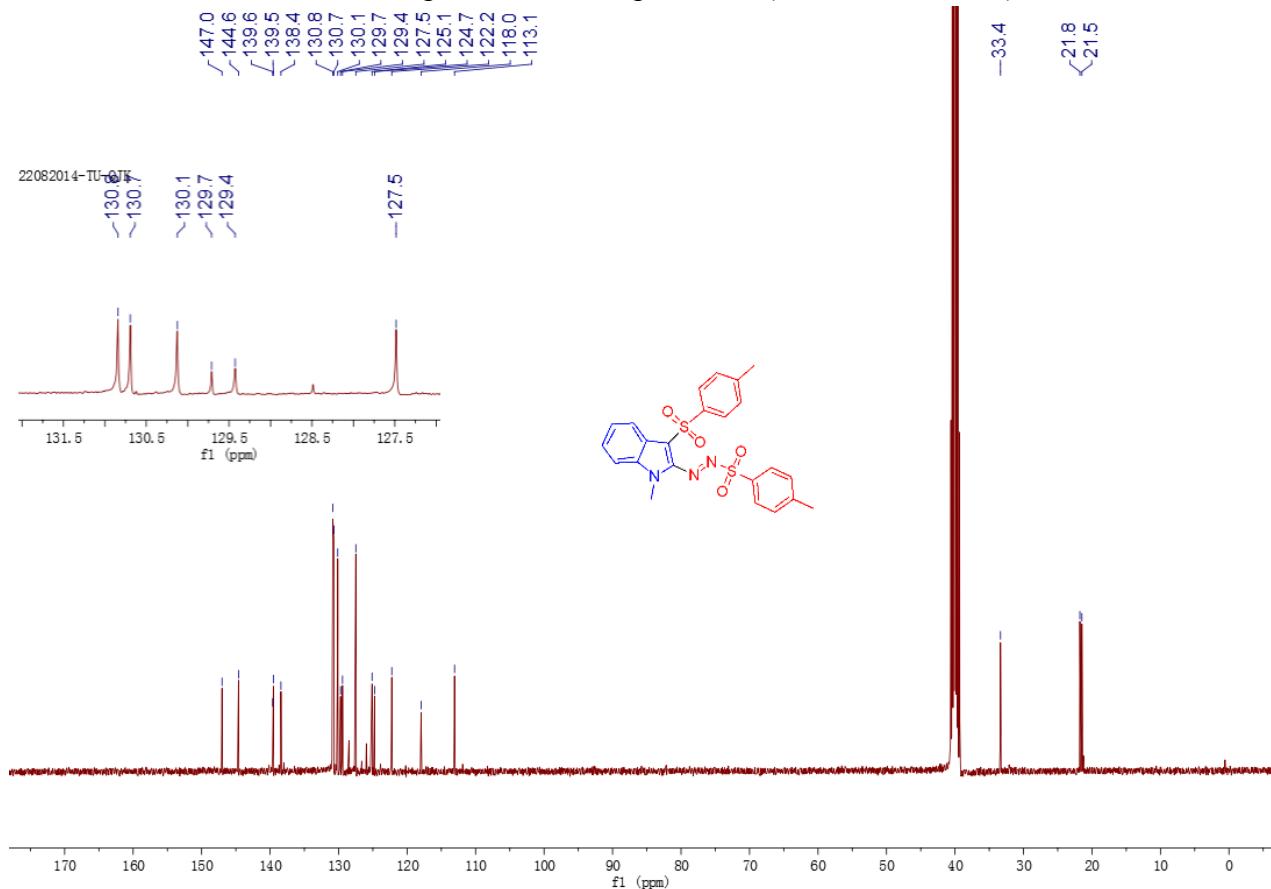
数据
 141.4
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 136.05
 134.77
 133.18
 132.38
 131.97
 131.97
 131.97
 130.81
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 129.91
 129.84
 129.81
 129.84
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 128.22
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 124.60
 124.57
 124.66
 124.66



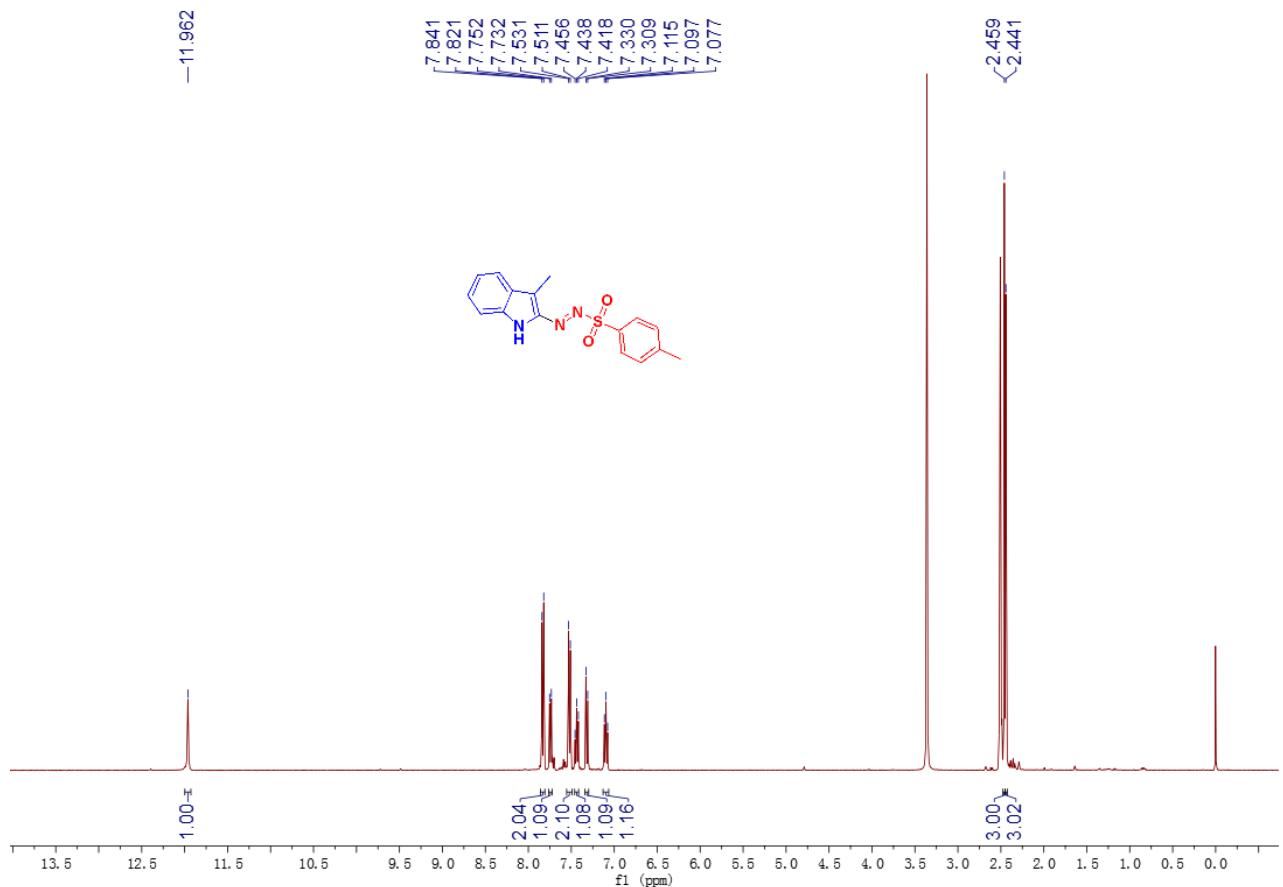
^{13}C NMR Spectrum of Compound **3t** (100Hz, $\text{DMSO}-d_6$)
 S35



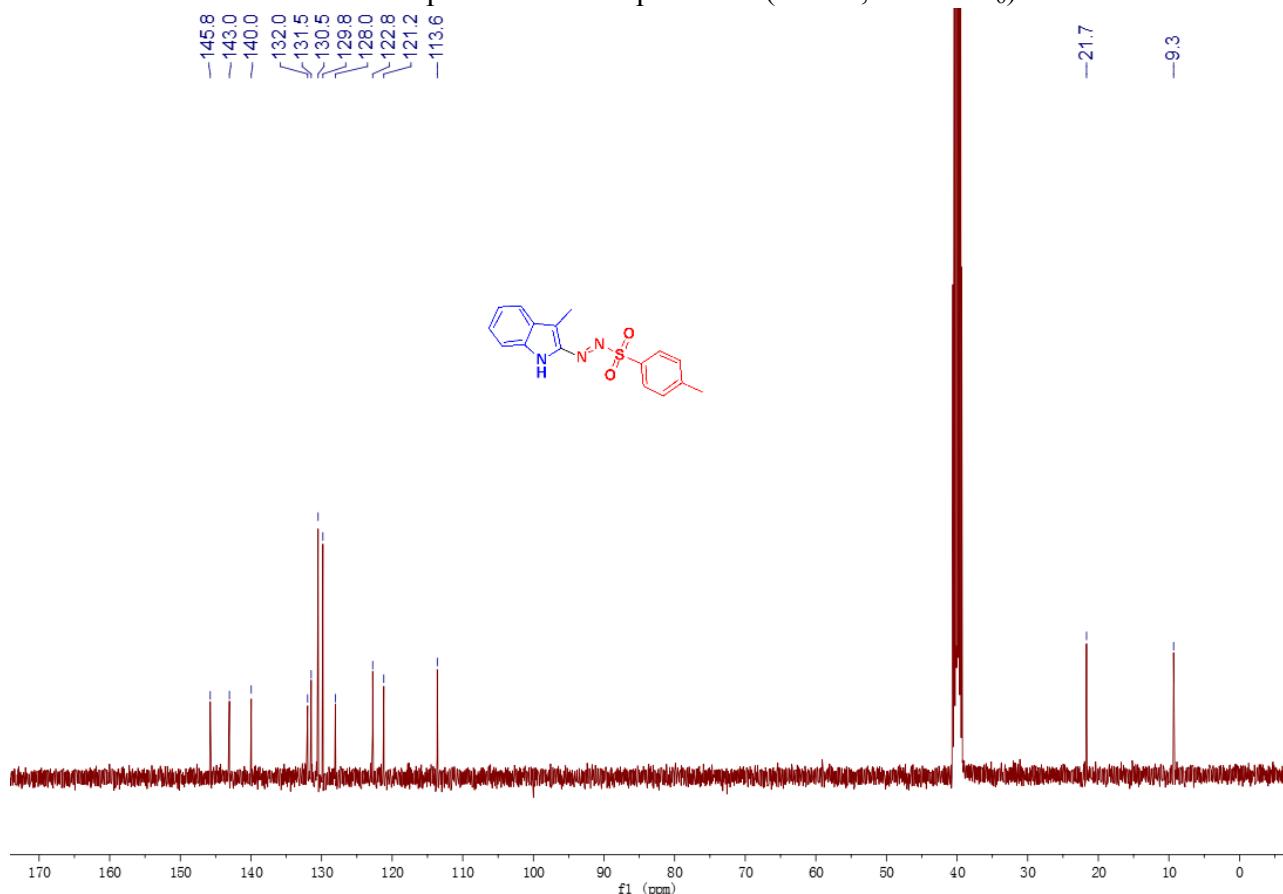
¹H NMR Spectrum of Compound 3u (400Hz, DMSO-*d*₆)



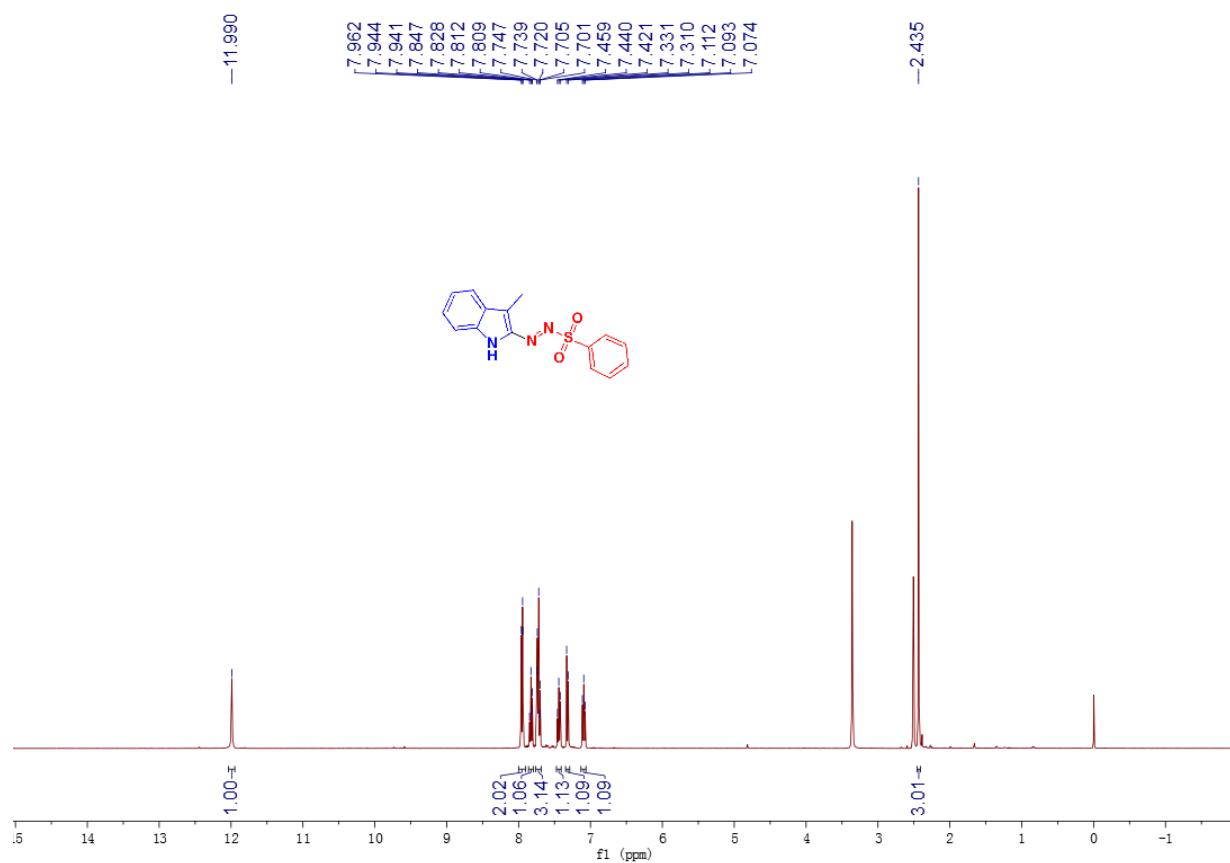
¹³C NMR Spectrum of Compound 3u (100Hz, DMSO-*d*₆)
S36



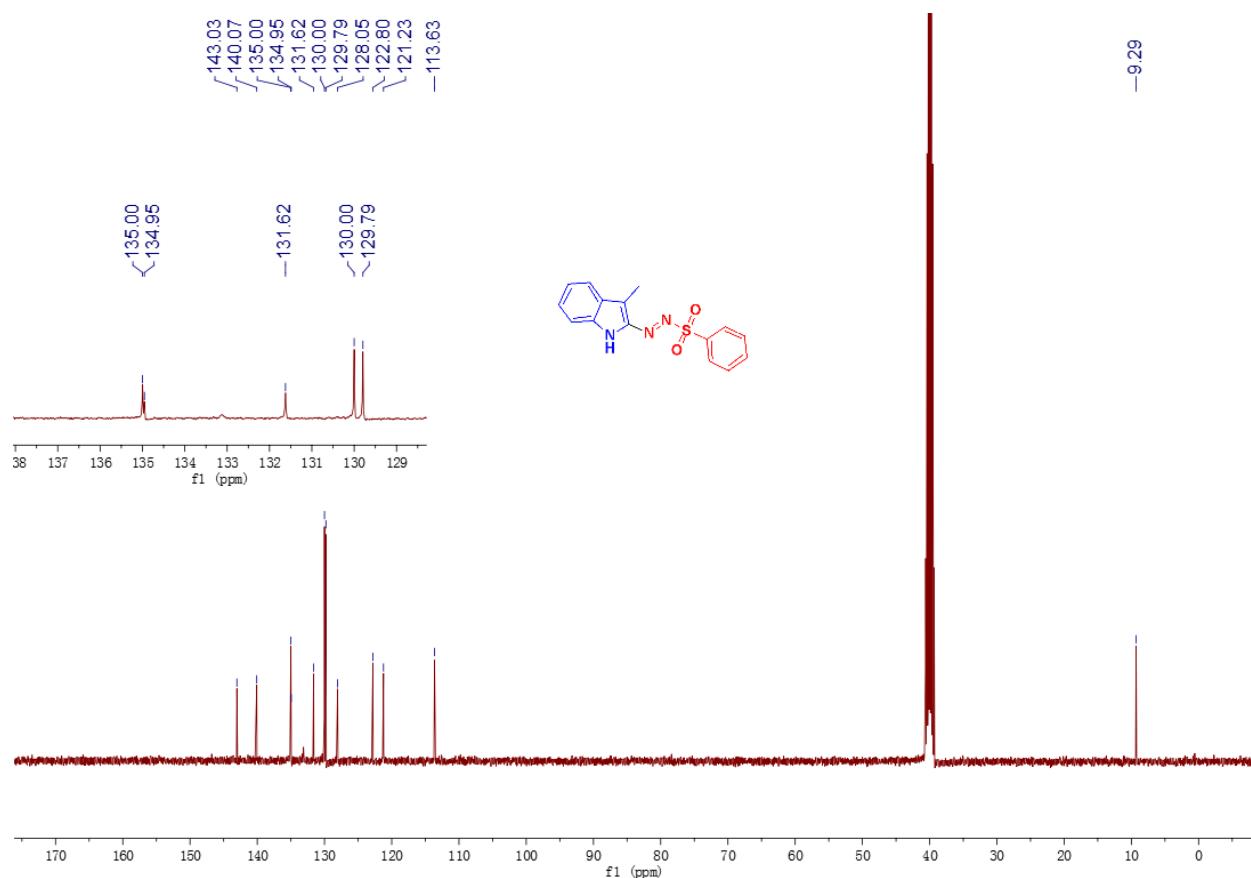
¹H NMR Spectrum of Compound 4a (400Hz, DMSO-*d*₆)



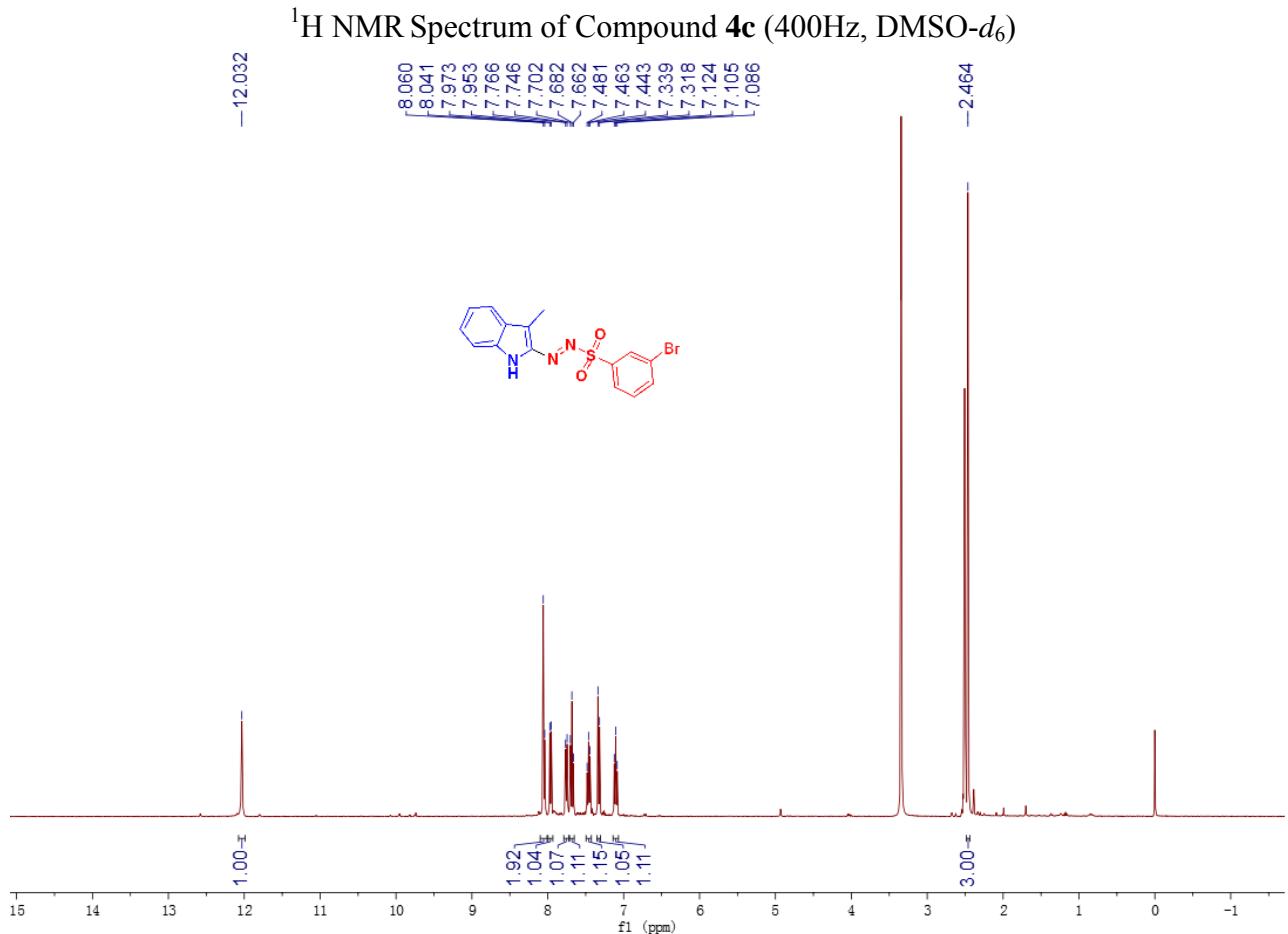
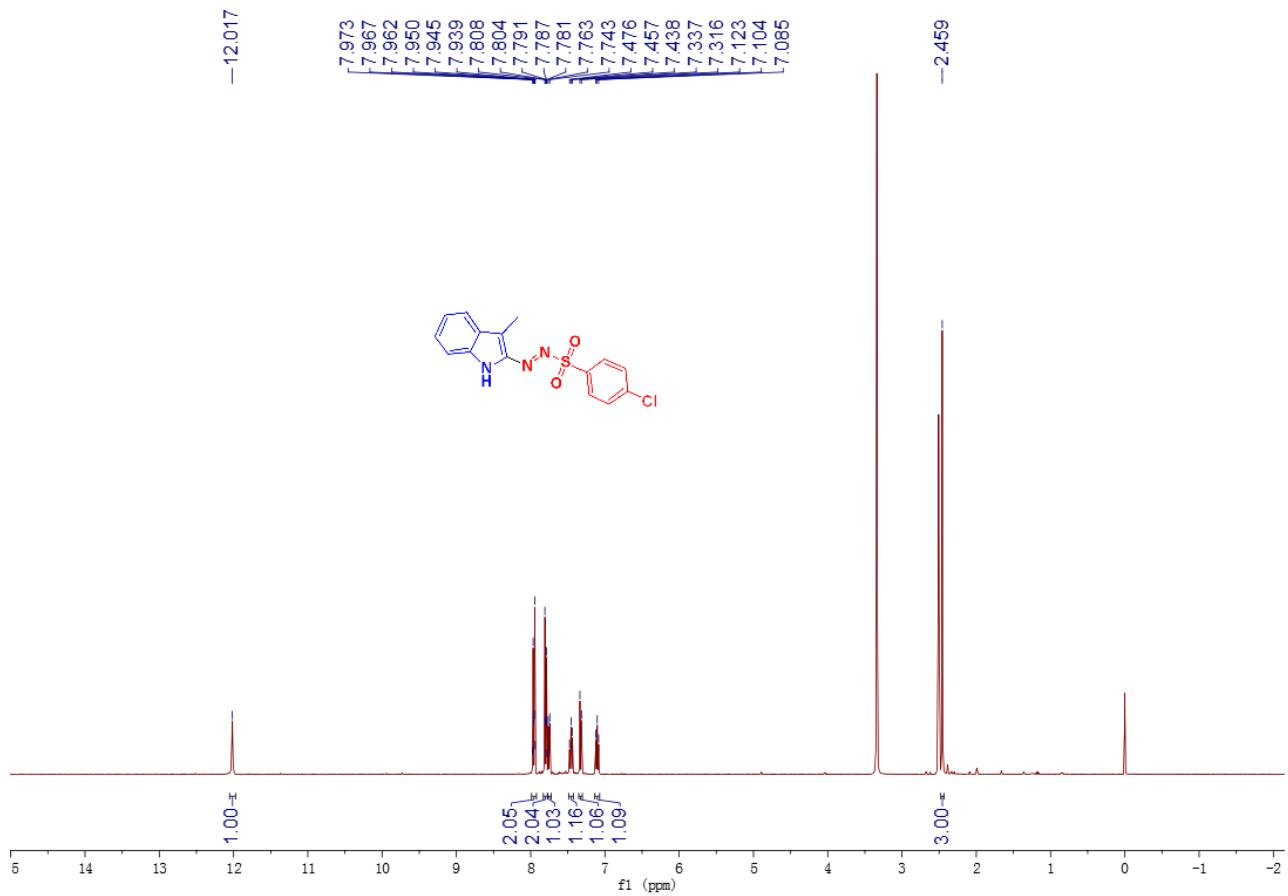
¹³C NMR Spectrum of Compound 4a (100Hz, DMSO-*d*₆)



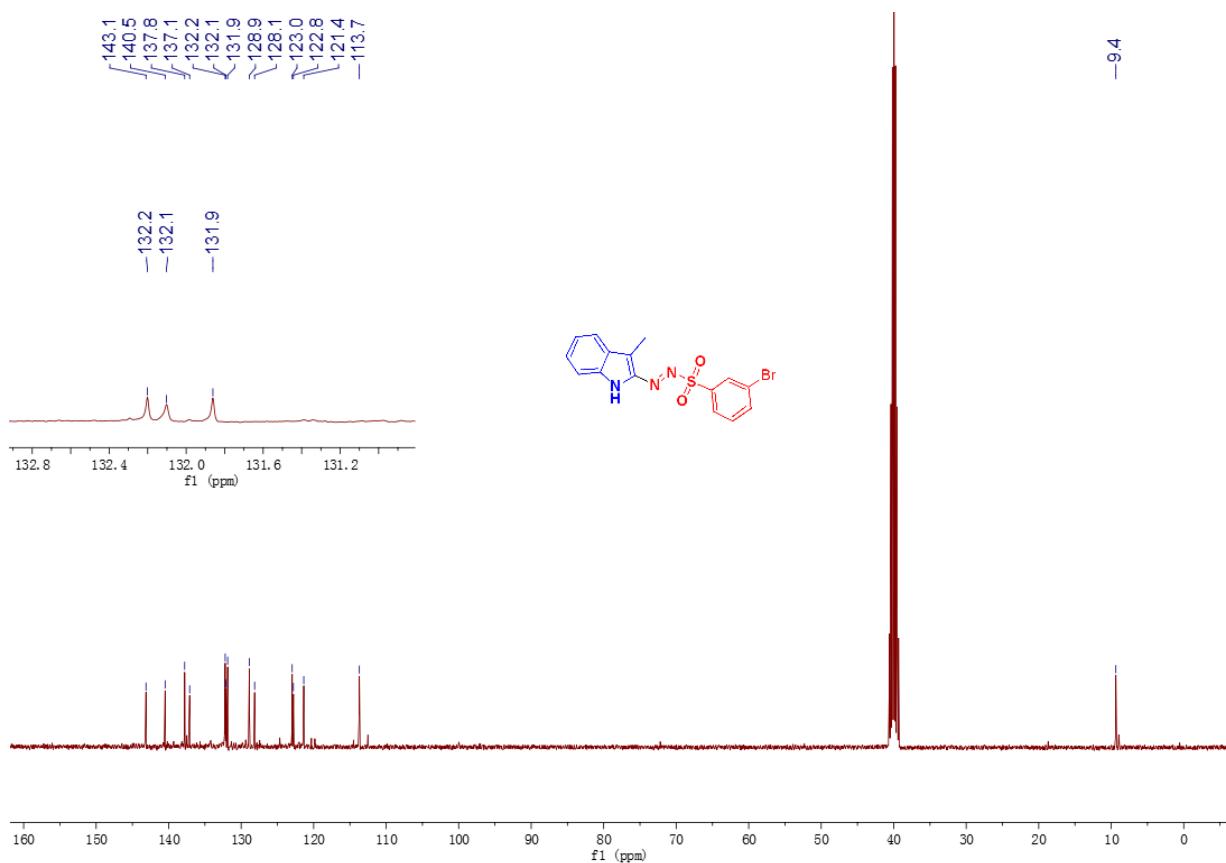
¹H NMR Spectrum of Compound **4b** (400Hz, DMSO-*d*₆)



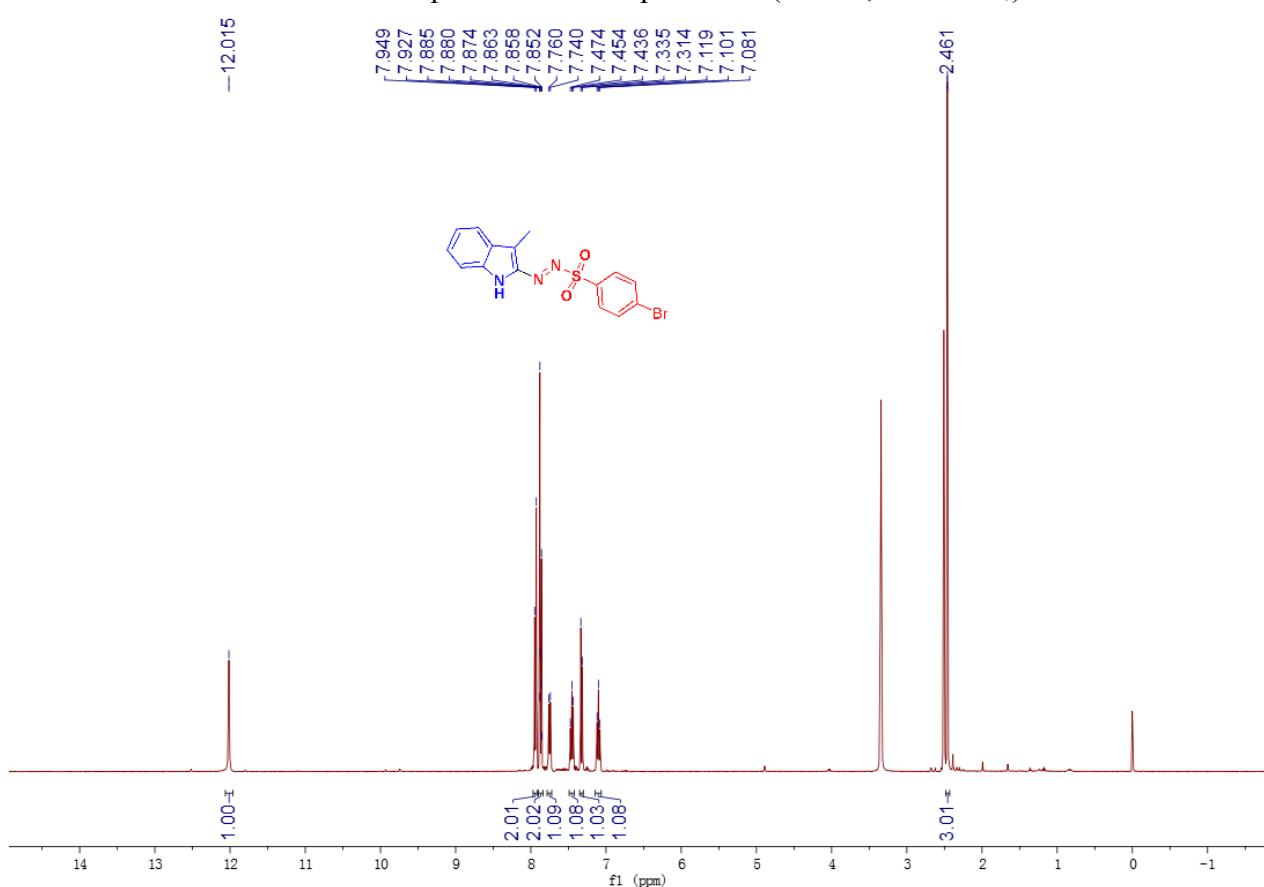
¹³C NMR Spectrum of Compound **4b** (100Hz, DMSO-*d*₆)
S38



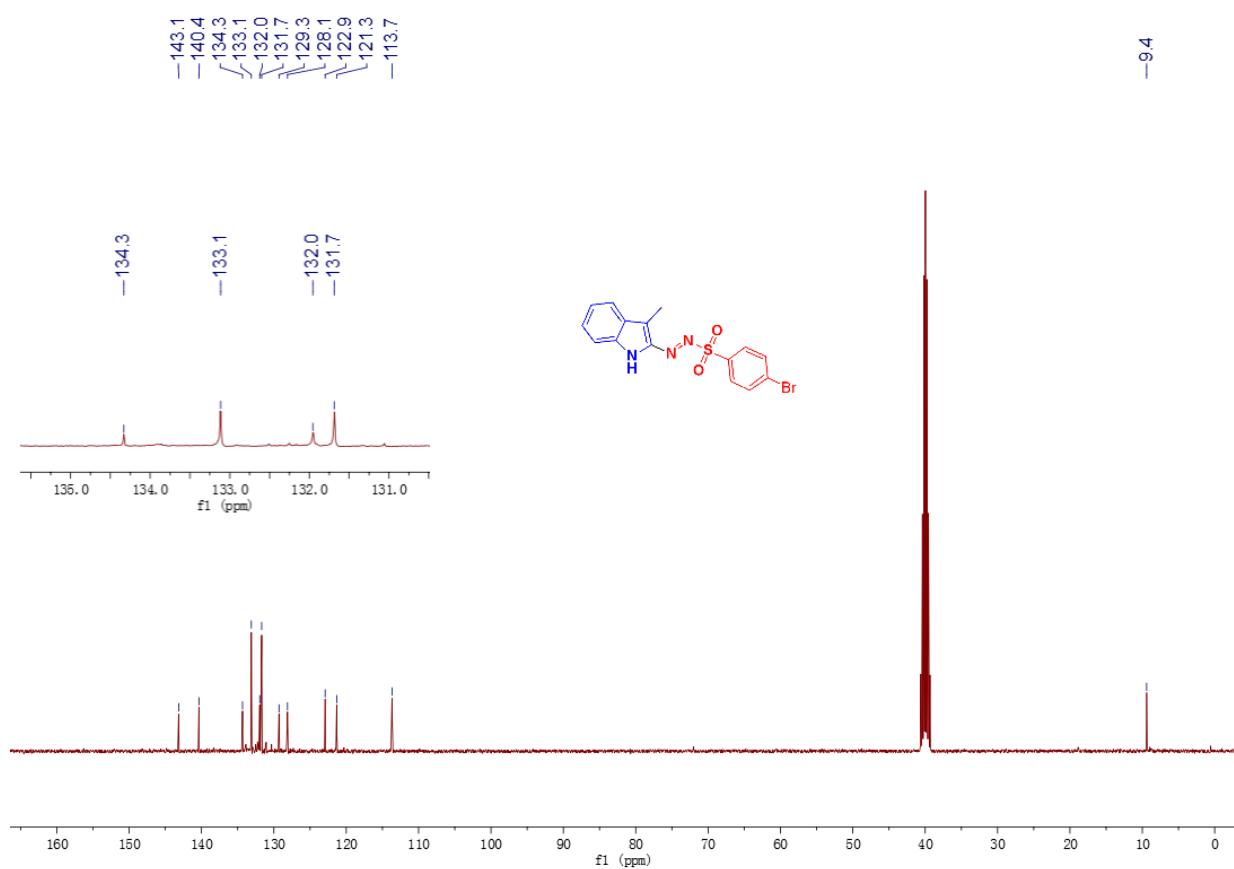
¹H NMR Spectrum of Compound **4d** (400Hz, DMSO-*d*₆)
S39



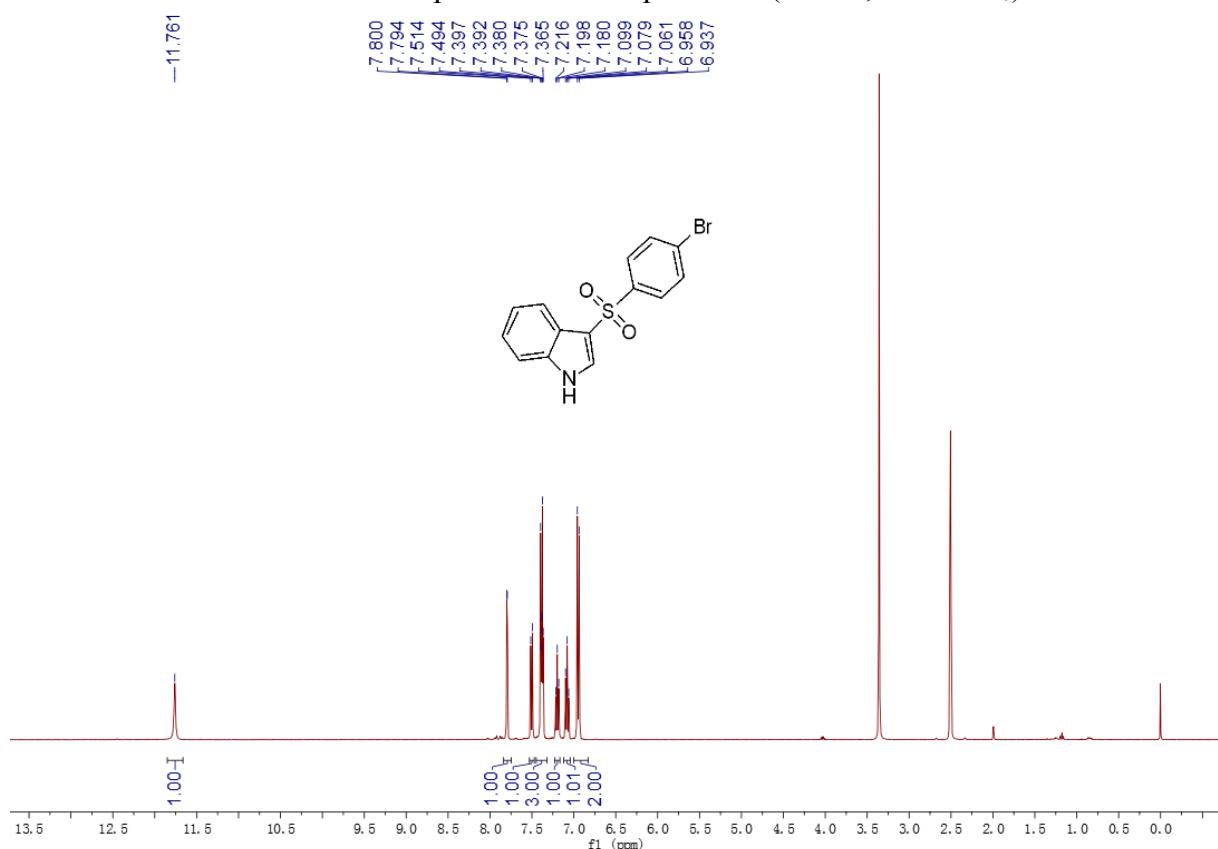
^{13}C NMR Spectrum of Compound **4d** (100Hz, $\text{DMSO}-d_6$)



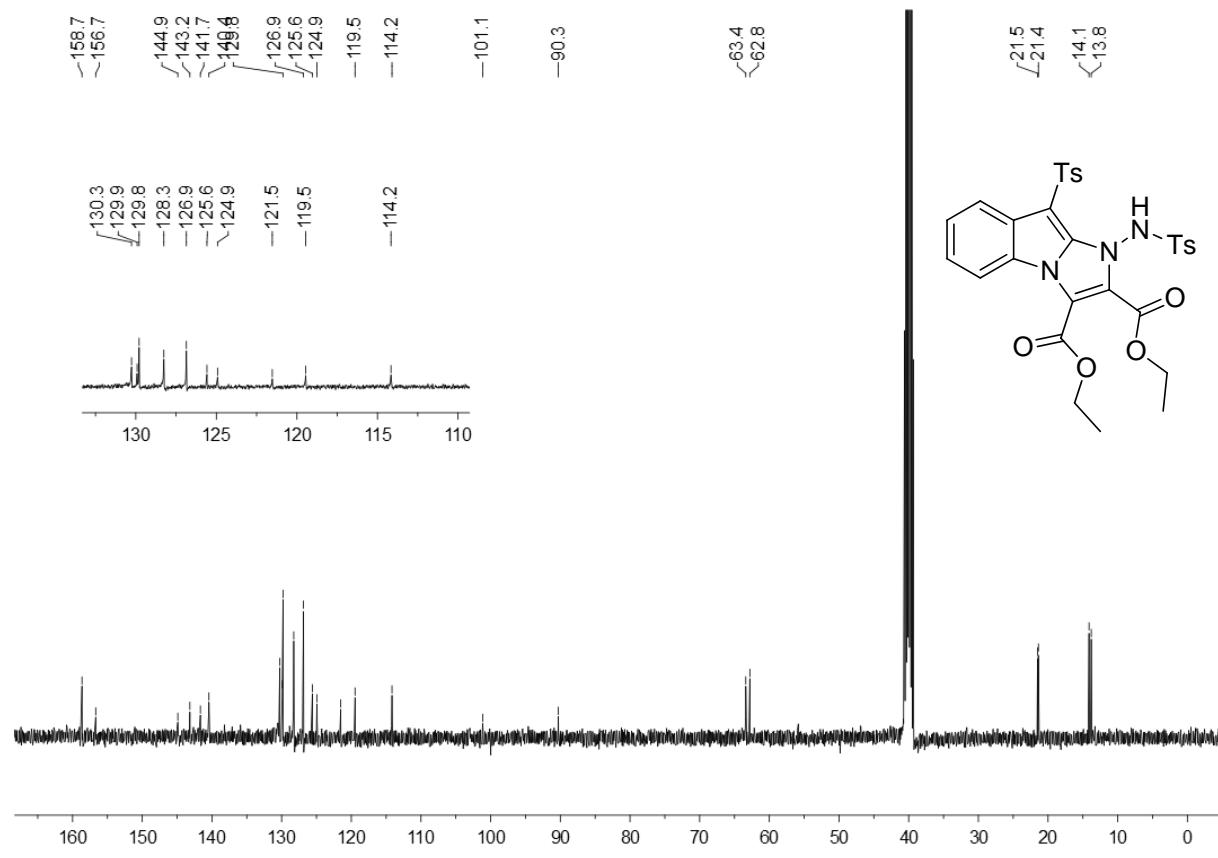
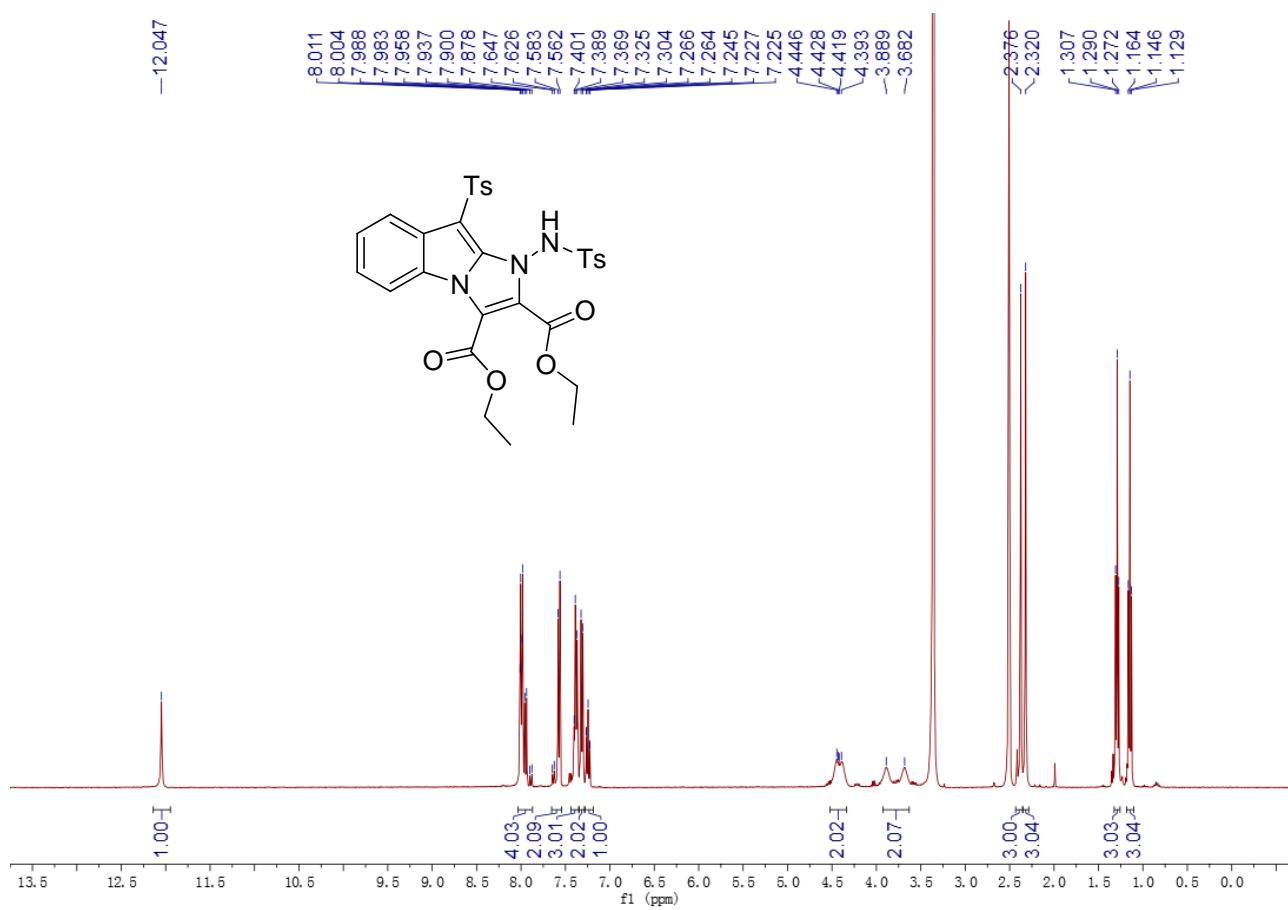
^1H NMR Spectrum of Compound **4e** (400Hz, $\text{DMSO}-d_6$)
S40



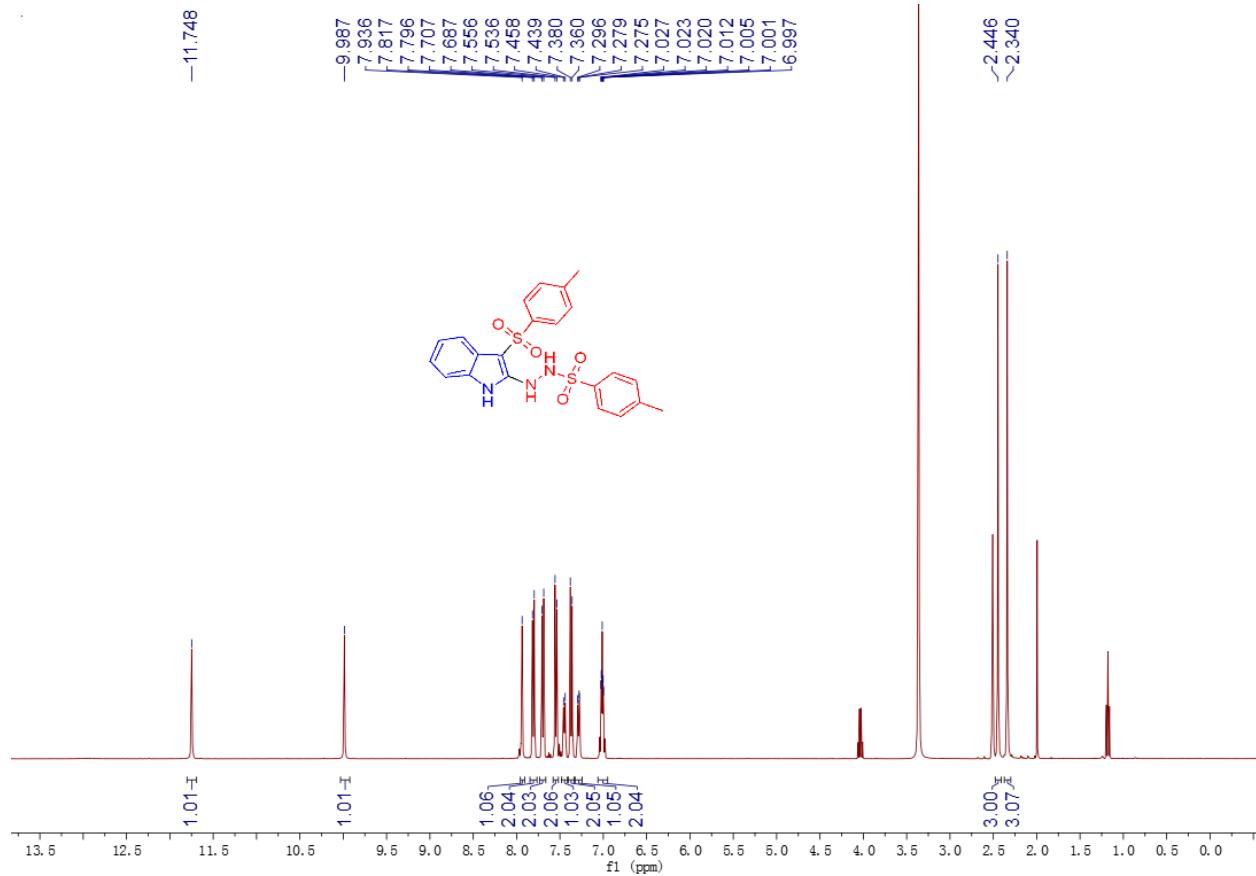
^{13}C NMR Spectrum of Compound **4e** (100Hz, $\text{DMSO}-d_6$)



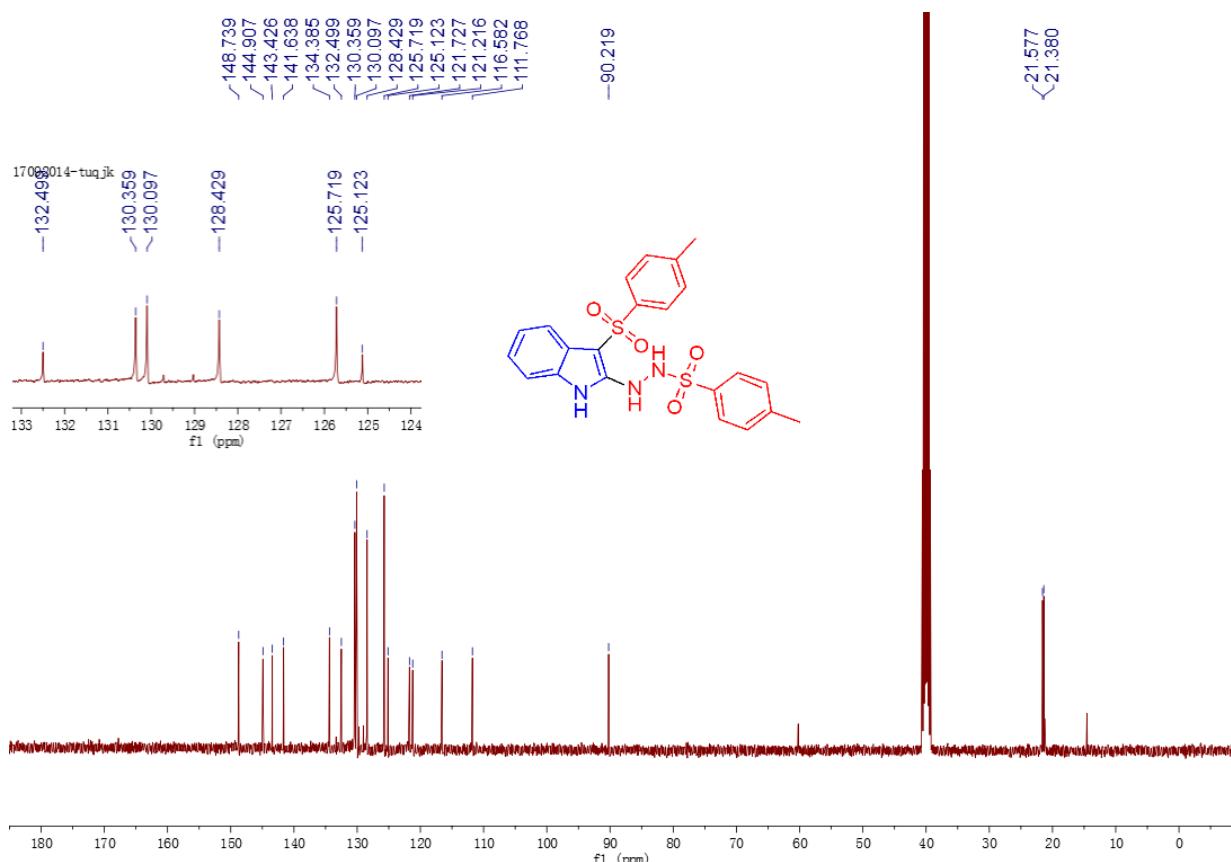
^1H NMR Spectrum of Compound **1o** (400Hz, $\text{DMSO}-d_6$)



¹³C NMR Spectrum of Compound 5 (100Hz, DMSO-*d*₆)



¹H NMR Spectrum of Compound 6 (400Hz, DMSO-*d*₆)



¹³C NMR Spectrum of Compound 6 (100Hz, DMSO-*d*₆)