Supporting Information

A simple Iodine-DMSO-promoted multicomponent reaction for the synthesis of 2,4-disubstituted dihydrotriazole-3-ones

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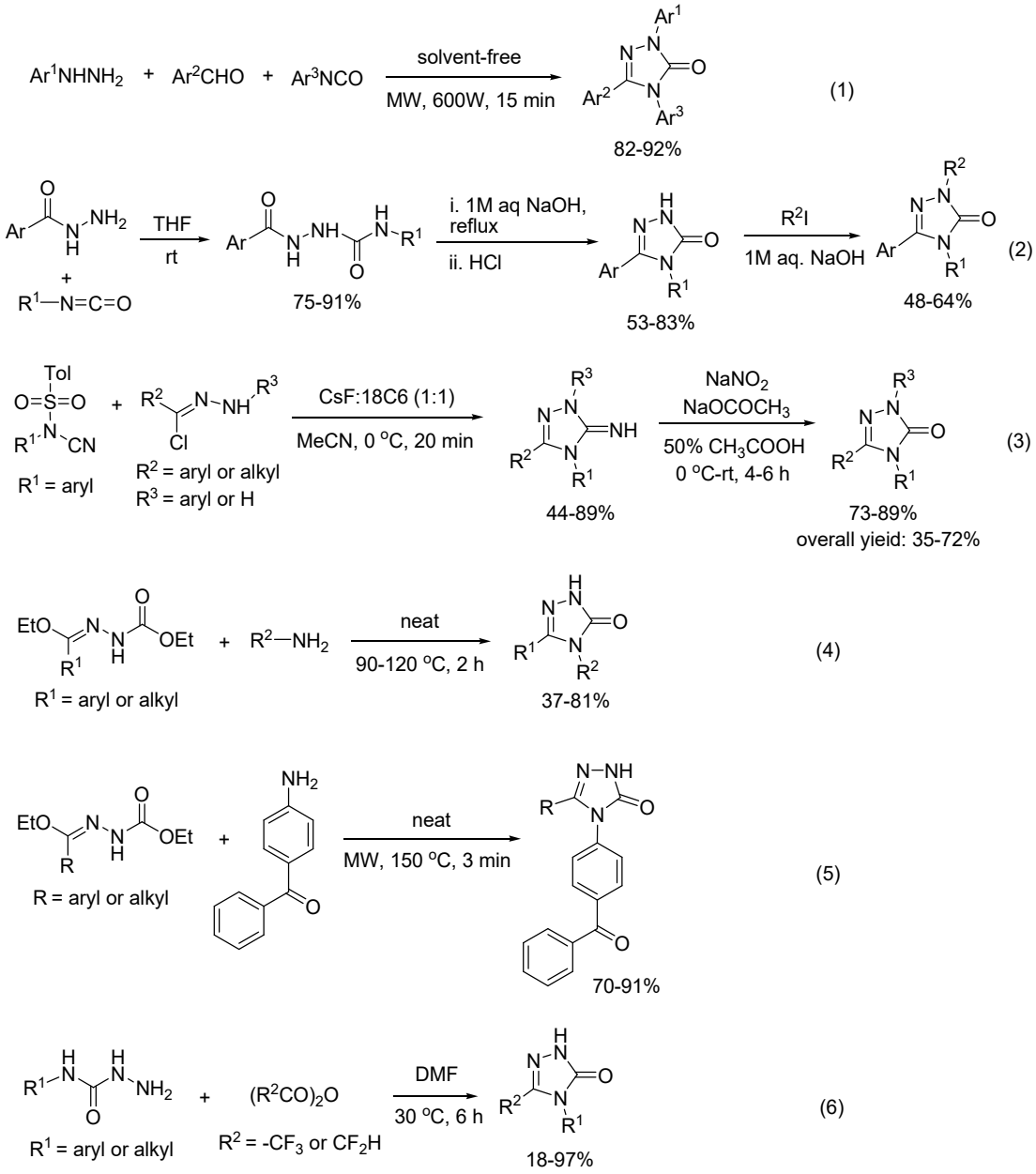


Figure S1. Synthesis of 2,4,5-trisubstitutied or 4,5-disubstituted 1,2,4-triazole-3-ones[1] [2] [3] [4] [5] [6]

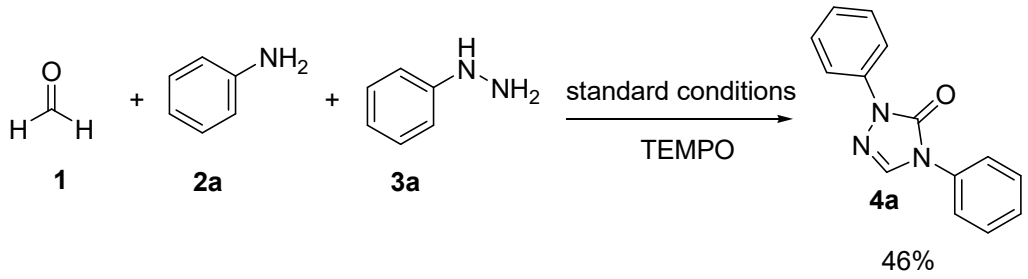
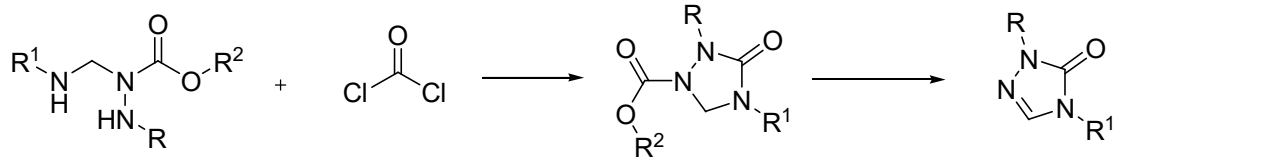


Figure S2. Control experiment



4g: R=Ph; R^1= 4-ClPh

4s: R=4-ClPh; R^1= Ph

Figure S3 synthesis of 4g and 4s in patent[7]

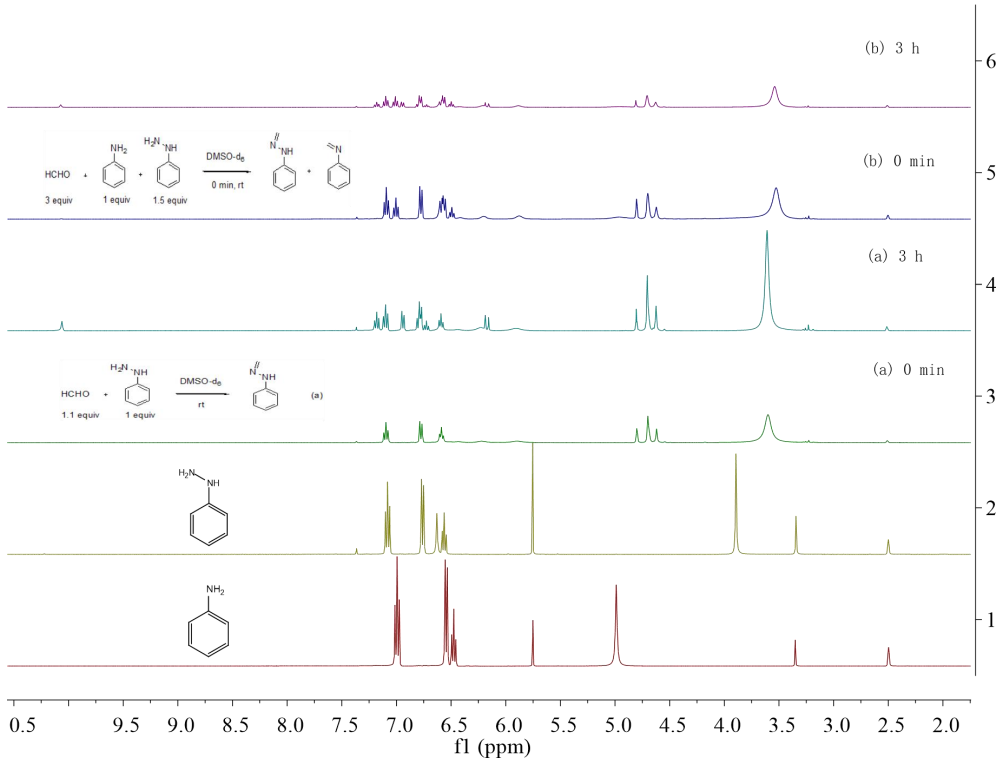
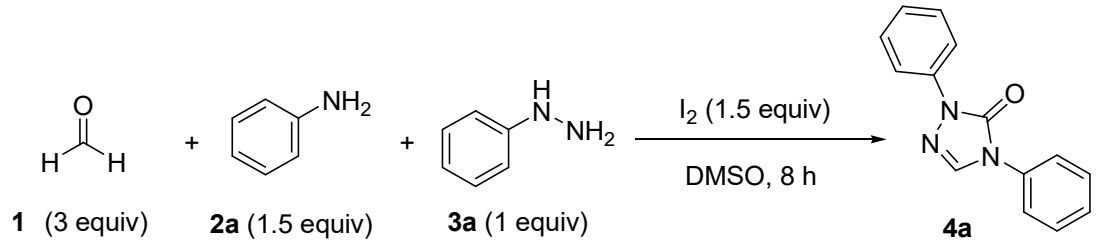


Figure S4. 1H NMR spectra monitoring formation of intermediate 5 and 6

1H NMR spectra of aniline (1), phenylhydrazine (2), the reaction (a) of aldehyde and phenylhydrazine in DMSO-d6 at room temperature for 0 min and 3 h, respectively (3 and 4, respectively), the reaction (b) of aldehyde, aniline and phenylhydrazine in DMSO-d6 at room temperature for 0 min and 3 h, respectively (5 and 6, respectively)

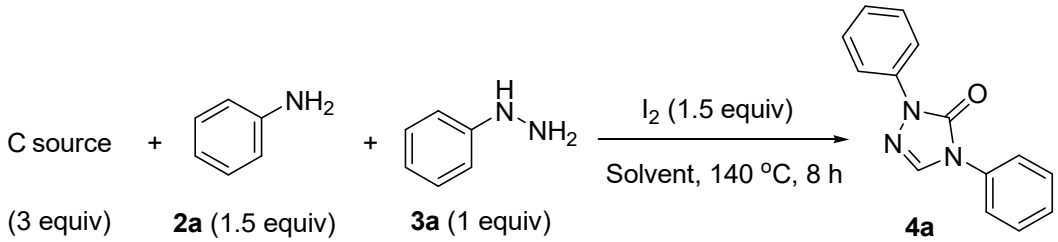
Table S1. Influence of temperature on the MCR for the synthesis of 4a[a]



1 (3 equiv) **2a** (1.5 equiv) **3a** (1 equiv) **4a**

entry	*T* (°C)	Yield (%)
1	100	0
2	120	14
3	140	28
4	160	28

Table S2. Optimization of C sources, iodine-containing activators and solvents for the MCR synthesis of 4a[a]



(3 equiv) **2a** (1.5 equiv) **3a** (1 equiv) **4a**

Entry	C source	[I]	Solvent	Yield[b] (%)
1	HCHO	I_2	DMSO	28
2	$(CH_2O)_n$	I_2	DMSO	0
3	DMSO	I_2	DMSO	0
4	DMF	I_2	DMSO	0
5	MeOH	I_2	DMSO	0
6	HCHO	NIS	DMSO	13
7	HCHO	CuI	DMSO	5
8	HCHO	KI	DMSO	trace
9	HCHO	TBAI	DMSO	trace
10	HCHO	NH_4I	DMSO	0
11	HCHO	I_2	DMF	0
12	HCHO	I_2	HMPA	0
13	HCHO	I_2	1,4-Dioxane	0
14	HCHO	I_2	*n*BuOH	0
15	HCHO	I_2	H_2O	0
16	HCHO	I_2	Toluene	0

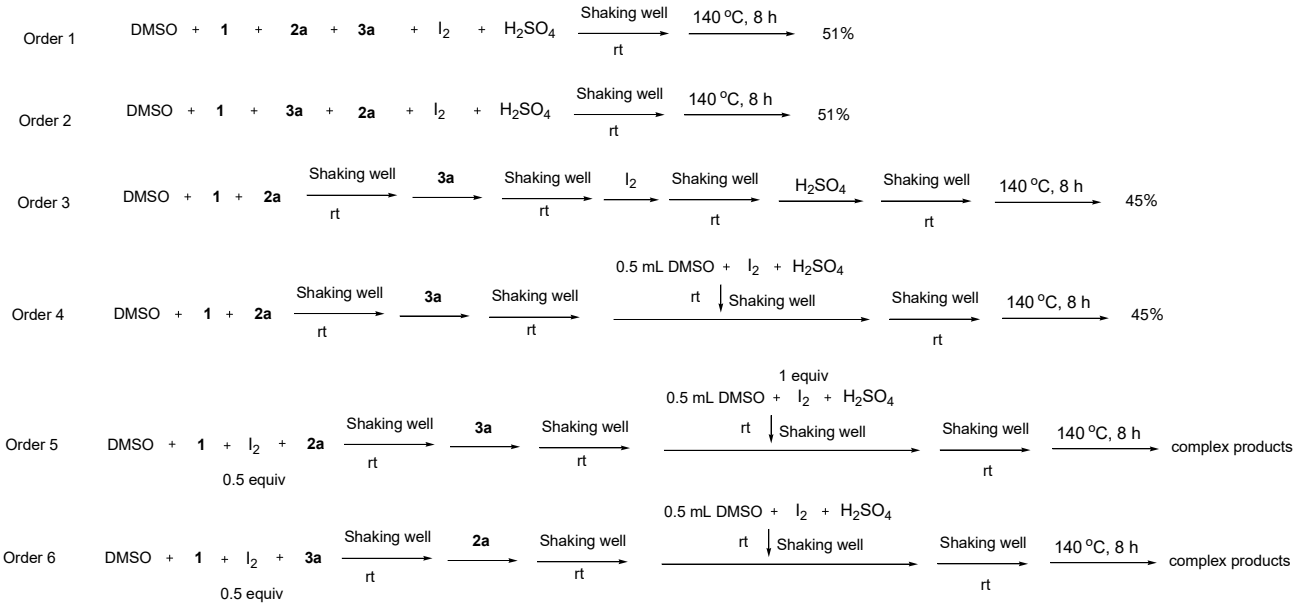
[a] Reaction was carried out with C source (0.9 mmol), **2a** (0.45 mmol), **3a** (0.3 mmol) and iodine-containing catalyst (0.45 mmol) in 2 mL solvent at 140 °C for 8 h. [b] Isolated yield.

Table S3. Crystallographic information of 4a

T^a [K]	293
crystal system	Monoclinic
space group	*P*121/*c*1
a [Å]	13.7128 (11)
b [Å]	7.4784 (7)
c [Å]	11.7095 (9)
α [Å]	90
β [deg]	96.628 (7)
γ [deg]	90
V [Å^3]	1192.78 (17)
Z	4
D_{calcu} [mg/m^3]	1.321
R	0.0458

wR_2	0.1213

Scheme S1. Screen of the adding orders of reactants



Materials and Instruments

All the starting materials were purchased from commercial suppliers and used without further purification. All melting points were measured using a X-5 micro melting point apparatus and were uncorrected. ^{1}H NMR (400 MHz) and ^{13}C NMR (101 MHz) spectra were recorded on a Bruker Avance 400 MHz NMR spectrometer with DMSO-d_6 or $CDCl_3$ as the solvent. ^{1}H NMR chemical shifts were referenced to DMSO-d_6 at 2.50 ppm or referenced to TMS at 0.00 ppm. ^{13}C NMR chemical shifts were referenced to DMSO-d_6 at 39.50 ppm. IR spectra were obtained as potassium bromide pellets or as liquid films on potassium bromide pellets with a Bruker Vector 22 spectrometer. High resolution mass spectra (HRMS) were recorded on an Thermo Q Exactive Plus or Agilent 6210 ESI/TOF mass spectrometer. Single-crystal X-ray diffraction data were measured by Bruker D8 Venture. The reactions were monitored by thin-layer chromatography (TLC) using 100−400 mesh silica gel plates (GF254) and were visualized using UV lamp (254 and 365 nm).

General procedure for the one-pot synthesis of 4a−4w

The reactions were run with the following steps: Add 0.9 mmol formaldehyde **1**, 0.3 mmol amine **2**, 0.45 mmol hydrazine **3** (If **3** is hydrazine hydrochloride, adding **3** and equivalent of $NaHCO_3$ into a 25 ml sealed tube containing 2 ml DMSO before other reagents, stirring until no carbon dioxide gas emission and then adding other reagents.), 0.45 mmol iodine and 0.15 mmol H_2SO_4 to a 25 ml sealed tube containing 2 ml DMSO, and stir in a 140 ℃ oil bath for 8 hours. Then, take out the sealed tube and cool it to room temperature; extract the reaction solution with saturated sodium thiosulfate aqueous solution and ethyl acetate for three times; collect the organic layer; dry anhydrous sodium sulfate, filter, and remove the solvent to obtain the concentrated solution via a rotary evaporation instrument; The concentrated solution was separated by thin layer chromatography to obtain the target products **4a−w** with petroleum ether: ethyl acetate = 6:1−2:1 as eluent.

Structure Characteristics of 4a–4w

2,4-Diphenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4a): Yellow brown solid, 51% yield, mp=107.5-108.5 °C; IR (KBr) ν_{max} = 3133, 3062, 2929, 2851, 1696, 1588, 1490, 1388, 1235, 932, 750, 685 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.74 (s, 1H), 8.07–7.86 (m, 2H), 7.83–7.66 (m, 2H), 7.65–7.37 (m, 5H), 7.29 (t, J = 7.4 Hz, 1H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.70, 137.51, 136.70, 133.54, 129.38, 129.13, 127.55, 125.50, 122.40, 118.39 ppm. HRMS (ESI) calculated for $C_{14}H_{12}N_3O$ $[M+H]^+$: 238.0975; found: 238.0971.

2-Phenyl-4-(*p*-tolyl)-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4b): Yellow brown solid, 55% yield, mp=134.4-135.1 °C; IR (KBr) ν_{max} = 3132, 3067, 2923, 2849, 1692, 1579, 1504, 1382, 1230, 940, 892, 743 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.66 (s, 1H), 7.94 (d, J = 7.8 Hz, 2H), 7.59 (d, J = 8.3 Hz, 2H), 7.49 (t, J = 8.0 Hz, 2H), 7.35 (d, J = 8.2 Hz, 2H), 7.27 (t, J = 7.4 Hz, 1H), 2.36 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.75, 137.55, 137.09, 136.75, 131.05, 129.75, 129.11, 125.45, 122.33, 118.35, 20.57 ppm. HRMS (ESI) calculated for $C_{15}H_{13}N_3NaO$ $[M+Na]^+$: 274.0951; found: 274.0945.

4-(4-(*tert*-Butyl)phenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4c): White solid, 49% yield, mp=154.5-155.2 °C; IR (KBr) ν_{max} = 3136, 3074, 2925, 2860, 1692, 1600, 1506, 1458, 1382, 1228, 833, 749 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.66 (s, 1H), 7.94 (d, J = 8.0 Hz, 2H), 7.62 (d, J = 8.6 Hz, 2H), 7.56 (d, J = 8.6 Hz, 2H), 7.50 (t, J = 7.9 Hz, 2H), 7.28 (t, J = 7.4 Hz, 1H), 1.32 (s, 9H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 150.24, 149.81, 137.55, 136.86, 130.98, 129.14, 126.13, 125.47, 122.31, 118.35, 34.41, 31.03 ppm. HRMS (ESI) calculated for $C_{18}H_{19}N_3NaO$ $[M+Na]^+$: 316.1420; found: 316.1417.

4-(4-Methoxyphenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4d): Yellow brown solid, 57% yield, mp=161.1-161.8 °C; IR (KBr) ν_{max} = 3071, 2923, 2847, 1688, 1453, 1240, 1029, 826, 757 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.63 (s, 1H), 7.95 (d, J = 7.7 Hz, 2H), 7.61 (d, J = 9.0 Hz, 2H), 7.55–7.44 (m, 2H), 7.28 (t, J = 7.4 Hz, 1H), 7.11 (d, J = 9.9 Hz, 2H), 3.82 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 158.54, 149.98, 137.61, 137.10, 129.13, 126.33, 125.43, 124.53, 118.30, 114.51, 55.49 ppm. HRMS (ESI) calculated for $C_{15}H_{13}N_3NaO_2$ $[M+Na]^+$: 290.0900; found: 290.0898.

4-(4-Ethoxyphenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4e): White solid, 56% yield, mp=141.5-142.1 °C; IR (KBr) ν_{max} = 3145, 3077, 2922, 2858, 1692, 1581, 1509, 1456, 1375, 1242, 1048, 937, 822, 745 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.60 (s, 1H), 7.94 (d, J = 7.7 Hz, 2H), 7.58 (d, J = 8.9 Hz, 2H), 7.49 (t, J = 8.0 Hz, 2H), 7.27 (t, J = 7.4 Hz, 1H), 7.08 (d, J = 9.0 Hz, 2H), 4.08 (q, J = 6.9 Hz, 2H), 1.35 (t, J = 7.0 Hz, 3H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 157.80, 149.97, 137.61, 137.08, 129.12, 126.19, 125.41, 124.50, 118.29, 114.95, 63.44, 14.56 ppm. HRMS (ESI) calculated for $C_{16}H_{16}N_3O_2$ $[M+H]^+$: 282.1237; found: 282.1234.

4-(4-Fluorophenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4f): Yellow brown solid, 45% yield, mp=161.5-162.4 °C; IR (KBr) ν_{max} = 3128, 3066, 2922, 2855, 1689, 1457, 1233, 833, 741 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.71 (s, 1H), 7.95 (d, J = 8.2 Hz, 2H), 7.82–7.70 (m, 2H), 7.56-7.35 (m, 4H), 7.29 (t, J = 7.4 Hz, 1H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 162.13, 159.70, 149.76, 137.49, 136.80, 129.90, 129.87, 129.39, 129.14, 125.52, 125.02, 124.93, 122.40, 118.40, 118.36, 116.31, 116.08 ppm. HRMS (ESI) calculated for $C_{14}H_{10}FN_3NaO$ $[M+Na]^+$: 278.0700; found: 278.0701.

4-(4-Chlorophenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4g): Light yellow solid, 33% yield, mp=187.2-188.1 °C; IR (KBr) ν_{max} = 3130, 3065, 2923, 2855, 1689, 1458, 1230, 826, 744 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.76 (s, 1H), 8.02–7.87 (m, 2H), 7.87–7.73 (m, 2H), 7.70–7.57 (m, 2H), 7.57–7.43 (m, 2H), 7.30 (t, J = 7.4 Hz, 1H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.52, 137.41, 136.45, 132.48, 131.77, 129.32, 129.14, 125.57, 123.97, 118.41 ppm. HRMS (ESI) calculated for $C_{14}H_{10}ClN_3NaO$ $[M+Na]^+$: 294.0405; found: 294.0438.

4-(4-Bromophenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4h): White solid, 32% yield, mp=201.3-202.2 °C; IR (KBr) ν_{max} = 3068, 2928, 2855, 1728, 1673, 1593, 1509, 1252, 930, 835, 750 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ = 8.74 (s, 1H), 7.93 (d, J = 7.8 Hz, 2H), 7.80–7.70 (m, 4H), 7.50 (t, J = 8.0 Hz, 2H), 7.28 (t, J = 7.4

Hz, 1H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.47, 137.39, 136.37, 132.90, 132.25, 129.13, 125.57, 124.19, 120.07, 118.41 ppm. HRMS (ESI) calculated for $C_{14}H_{11}BrN_3O$ $[M+H]^+$: 316.0080; found: 316.0073.

4-(3,4-Dimethylphenyl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4i): Orange red solid, 52% yield, mp=107.5-108.2 °C; IR (KBr) ν_{max} = 3144, 3078, 2925, 2858, 1706, 1573, 1497, 1458, 1378, 1232, 954, 753 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.64 (s, 1H), 7.98–7.90 (m, 2H), 7.49 (t, J = 7.9 Hz, 3H), 7.42 (d, J = 8.1 Hz, 1H), 7.32–7.24 (m, 2H), 2.27 (d, J = 7.7 Hz, 6H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.77, 137.57, 137.49, 136.81, 135.89, 131.19, 130.13, 129.12, 125.46, 123.35, 119.81, 118.34, 19.43, 18.94 ppm. HRMS (ESI) calculated for $C_{16}H_{15}N_3NaO$ $[M+Na]^+$: 288.1107; found: 288.1105.

2-Phenyl-4-(3,4,5-trimethoxyphenyl)-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4j): Yellow brown solid, 50% yield, mp=144.7-145.5 °C; IR (KBr) ν_{max} = 3146, 3078, 2930, 2850, 1707, 1587, 1490, 1367, 1242, 1118, 1006, 847, 757 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.69 (s, 1H), 7.94 (d, J = 8.3 Hz, 2H), 7.50 (t, J = 7.8 Hz, 2H), 7.28 (t, J = 7.3 Hz, 1H), 7.07 (s, 2H), 3.83 (s, 6H), 3.70 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 153.17, 149.71, 137.50, 136.96, 136.68, 129.26, 129.14, 125.54, 118.44, 100.70, 60.16, 56.20 ppm. HRMS (ESI) calculated for $C_{17}H_{17}N_3NaO_4$ $[M+Na]^+$: 350.1111; found: 350.1106.

4-(Naphthalen-2-yl)-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4k): Yellow brown solid, 39% yield, mp=172.4-173.5 °C; IR (KBr) ν_{max} = 3137, 3070, 2922, 2852, 1690, 1454, 1374, 1234, 858, 743 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.84 (s, 1H), 8.31 (d, J = 1.6 Hz, 1H), 8.11 (d, J = 8.8 Hz, 1H), 8.05–7.94 (m, 4H), 7.91–7.84 (m, 1H), 7.65–7.56 (m, 2H), 7.52 (t, J = 7.9 Hz, 2H), 7.30 (t, J = 7.4 Hz, 1H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.85, 137.54, 136.84, 132.79, 131.66, 131.09, 129.26, 129.18, 127.92, 127.76, 127.14, 126.67, 125.58, 120.88, 120.29, 118.45 ppm. HRMS (ESI) calculated for $C_{18}H_{14}N_3O$ $[M+H]^+$: 288.1131; found: 288.1104.

4-Benzyl-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4l): Reddish brown oil, 37% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.38 (s, 1H), 7.91 (d, J = 8.4 Hz, 2H), 7.54–7.16 (m, 8H), 4.89 (s, 2H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 151.61, 137.83, 135.60, 134.88, 129.16, 128.95, 128.58, 128.10, 125.54, 118.65, 46.21 ppm. HRMS (ESI) calculated for $C_{15}H_{13}N_3NaO$ $[M+Na]^+$: 274.0951; found: 274.0946.

4-Butyl-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4m): Reddish brown oil, 34% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.29 (s, 1H), 8.04–7.77 (m, 2H), 7.47 (t, J = 7.8 Hz, 2H), 7.24 (t, J = 7.4 Hz, 1H), 3.66 (t, J = 7.2 Hz, 2H), 1.75-1.60 (m, 2H), 1.43–1.16 (m, 2H), 0.92 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 151.65, 137.90, 135.82, 128.91, 125.43, 118.61, 42.31, 31.12, 19.70, 13.47 ppm. HRMS (ESI) calculated for $C_{12}H_{15}N_3NaO$ $[M+Na]^+$: 240.1107; found: 240.1104.

4-Cyclopropyl-2-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4n): White solid, 32% yield, mp=120.5-121.4 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.20 (s, 1H), 7.88 (d, J = 7.9 Hz, 2H), 7.60–7.36 (m, 2H), 7.33–7.15 (m, 1H), 3.12 – 2.97 (m, 1H), 1.05-0.88 (m, 4H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 152.28, 137.84, 136.19, 128.94, 125.49, 118.70, 24.23, 5.65 ppm. HRMS (ESI) calculated for $C_{11}H_{11}N_3NaO$ $[M+Na]^+$: 224.0794; found: 224.0792.

4-Phenyl-2-(*p*-tolyl)-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4o): Light yellow solid, 48% yield, mp=156.2-157.1 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.69 (s, 1H), 7.81 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 7.8 Hz, 2H), 7.55 (t, J = 7.7 Hz, 2H), 7.43 (d, J = 7.4 Hz, 1H), 7.30 (d, J = 8.1 Hz, 2H), 2.33 (s, 3H) ppm; ^{13}C NMR (101 MHz, DMSO-d_6) δ = 149.66, 138.96, 137.51, 136.69, 133.45, 129.17, 129.10, 128.14, 125.46, 122.78, 119.43, 118.34, 20.91 ppm. HRMS (ESI) calculated for $C_{15}H_{13}N_3NaO$ $[M+Na]^+$: 274.0951; found: 274.0945.

2-(4-Ethylphenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4p): Yellow brown solid, 56% yield, mp=111.0-112.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89 (d, J = 8.3 Hz, 2H), 7.84 (s, 1H), 7.60 (d, J = 7.8 Hz, 2H), 7.51 (t, J = 7.7 Hz, 2H), 7.40 (t, J = 7.4 Hz, 1H), 7.28 (d, J = 8.3 Hz, 2H), 2.68 (q, J = 7.6 Hz, 2H), 1.26 (t, J = 7.6 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.20, 141.98, 135.40, 134.46, 133.63, 129.69, 128.39, 127.86, 122.37, 119.19, 28.39, 15.57 ppm. HRMS (ESI) calculated for $C_{16}H_{15}N_3NaO$ $[M+Na]^+$: 288.1107; found: 288.1105.

2-(4-(*tert*-Butyl)phenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4q): Yellow brown solid, 55% yield, mp=152.4-153.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.90 (d, J = 8.7 Hz, 2H), 7.84 (s, 1H), 7.60 (d, J = 7.9 Hz, 2H), 7.55–7.44 (m, 4H), 7.39 (t, J = 7.4 Hz, 1H), 1.35 (s, 9H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.22, 148.86, 135.10, 134.49, 133.62, 129.68, 127.84, 125.89, 122.35, 118.87, 34.50, 31.33 ppm. HRMS (ESI) calculated for $C_{18}H_{19}N_3NaO$ $[M+Na]^+$: 316.1420; found: 316.1418.

2-(4-Fluorophenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4r): Yellow brown solid, 46% yield, mp=171.3-172.1 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.74 (s, 1H), 8.02–7.89 (m, 2H), 7.82–7.66 (m, 2H), 7.56 (t, J = 7.7 Hz, 2H), 7.49–7.29 (m, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 161.65, 159.21, 150.15, 134.69, 133.45, 129.73, 128.01, 122.38, 120.82, 120.74, 115.88, 115.66 ppm. HRMS (ESI) calculated for $C_{14}H_{10}FN_3NaO$ $[M+Na]^+$: 278.0700; found: 278.0698.

2-(4-Chlorophenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4s): Yellow brown solid, 37% yield, mp=206.3-207.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.99 (d, J = 8.9 Hz, 2H), 7.85 (s, 1H), 7.58 (d, J = 7.9 Hz, 2H), 7.52 (t, J = 7.8 Hz, 2H), 7.42 (d, J = 8.9 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.08, 136.27, 134.90, 133.36, 131.07, 129.76, 129.10, 128.08, 122.42, 120.02 ppm. HRMS (ESI) calculated for $C_{14}H_{10}ClN_3NaO$ $[M+Na]^+$: 294.0405; found: 294.0403.

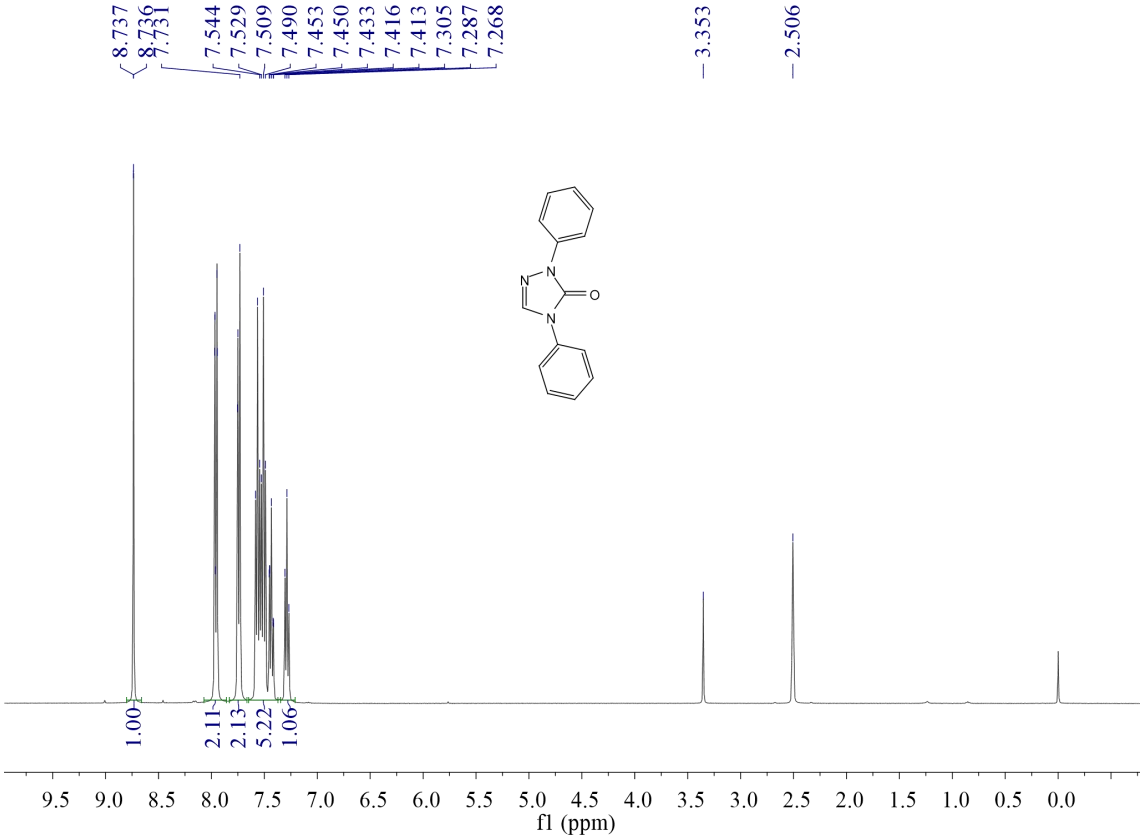
2-(4-Bromophenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4t): Yellow brown solid, 39% yield, mp=213.2-214.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.94 (d, J = 8.9 Hz, 2H), 7.85 (s, 1H), 7.62–7.49 (m, 6H), 7.41 (t, J = 7.3 Hz, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.07, 136.78, 134.94, 133.34, 132.05, 129.76, 128.09, 122.42, 120.30, 118.85 ppm. HRMS (ESI) calculated for $C_{14}H_{10}BrN_3NaO$ $[M+Na]^+$: 337.9899; found: 337.9898.

2-(3,4-Dimethylphenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4u): Reddish brown solid, 46% yield, mp=140.2-141.0 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 8.70 (s, 1H), 7.78-7.69 (m, 3H), 7.68-7.62 (m, 1H), 7.56 (t, J = 7.7 Hz, 2H), 7.43 (t, J = 7.4 Hz, 1H), 7.25 (d, J = 8.2 Hz, 1H), 2.32-2.18 (m, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.17, 137.38, 135.44, 134.35, 134.33, 133.66, 130.04, 129.67, 127.82, 122.32, 120.32, 116.66, 19.98, 19.30 ppm. HRMS (ESI) calculated for $C_{16}H_{15}N_3NaO$ $[M+Na]^+$: 288.1107; found: 288.1105.

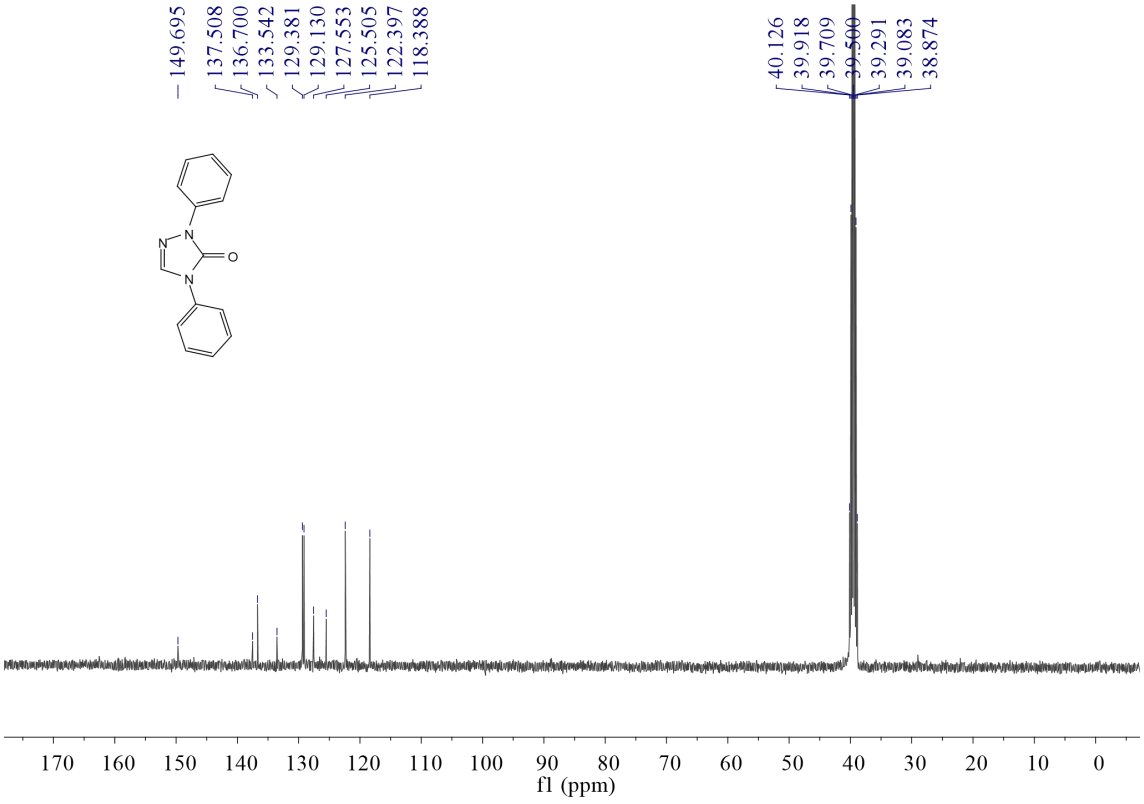
2-(3,5-Dimethylphenyl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4v): Yellow solid, 52% yield, mp=95.4-96.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.84 (s, 1H), 7.68–7.56 (m, 4H), 7.51 (t, J = 7.6 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H), 6.91 (s, 1H), 2.38 (s, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.20, 138.81, 137.49, 134.43, 133.62, 129.67, 127.85, 127.59, 122.30, 116.87, 21.46 ppm. HRMS (ESI) calculated for $C_{16}H_{15}N_3NaO$ $[M+Na]^+$: 288.1107; found: 288.1105.

2-(Naphthalen-2-yl)-4-phenyl-2,4-dihydro-3*H*-1,2,4-triazol-3-one (4w): White solid, 47% yield, mp=147.5-148.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.54 (s, 1H), 8.19 (d, J = 9.3 Hz, 1H), 7.95–7.84 (m, 4H), 7.63 (d, J = 7.9 Hz, 2H), 7.56–7.39 (m, 5H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 150.35, 134.75, 133.43, 131.34, 129.72, 128.98, 128.16, 127.96, 127.65, 126.63, 125.71, 122.39, 118.17, 116.33 ppm. HRMS (ESI) calculated for $C_{18}H_{14}N_3O$ $[M+H]^+$: 288.1131; found: 288.1124.

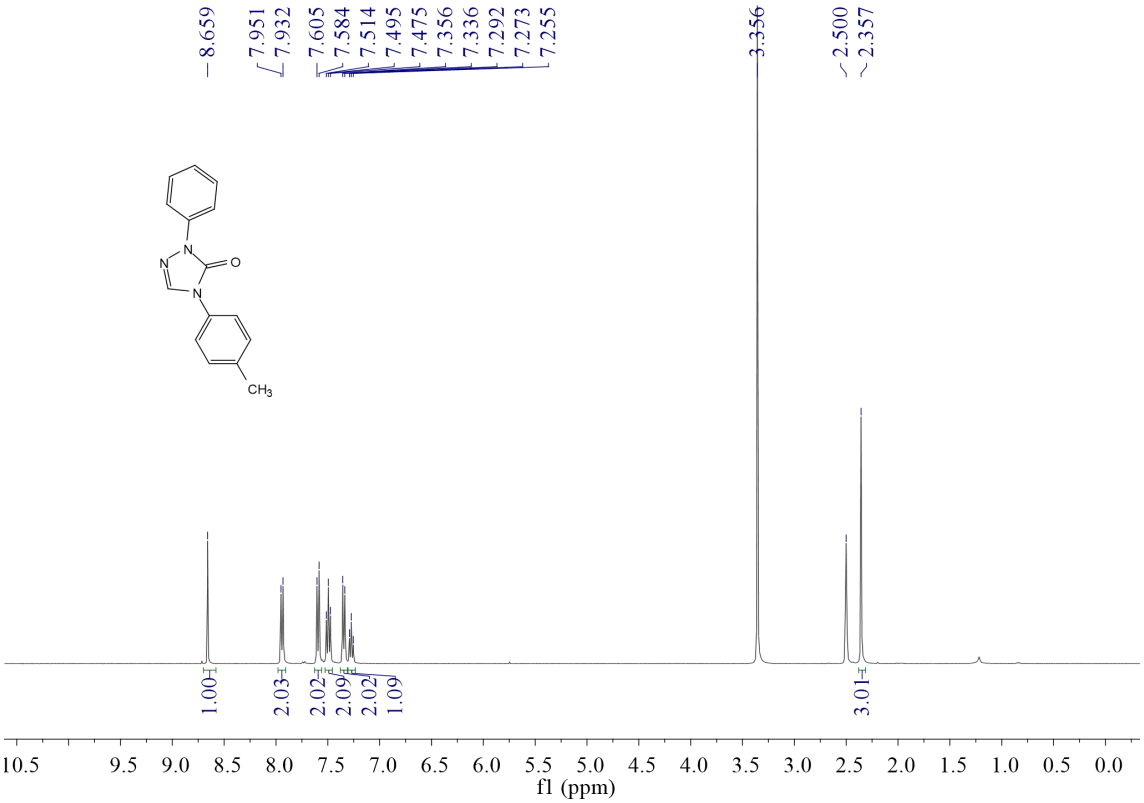
^{1}H and ^{13}CNMR spectrum copies of 4a–4w in Table 2



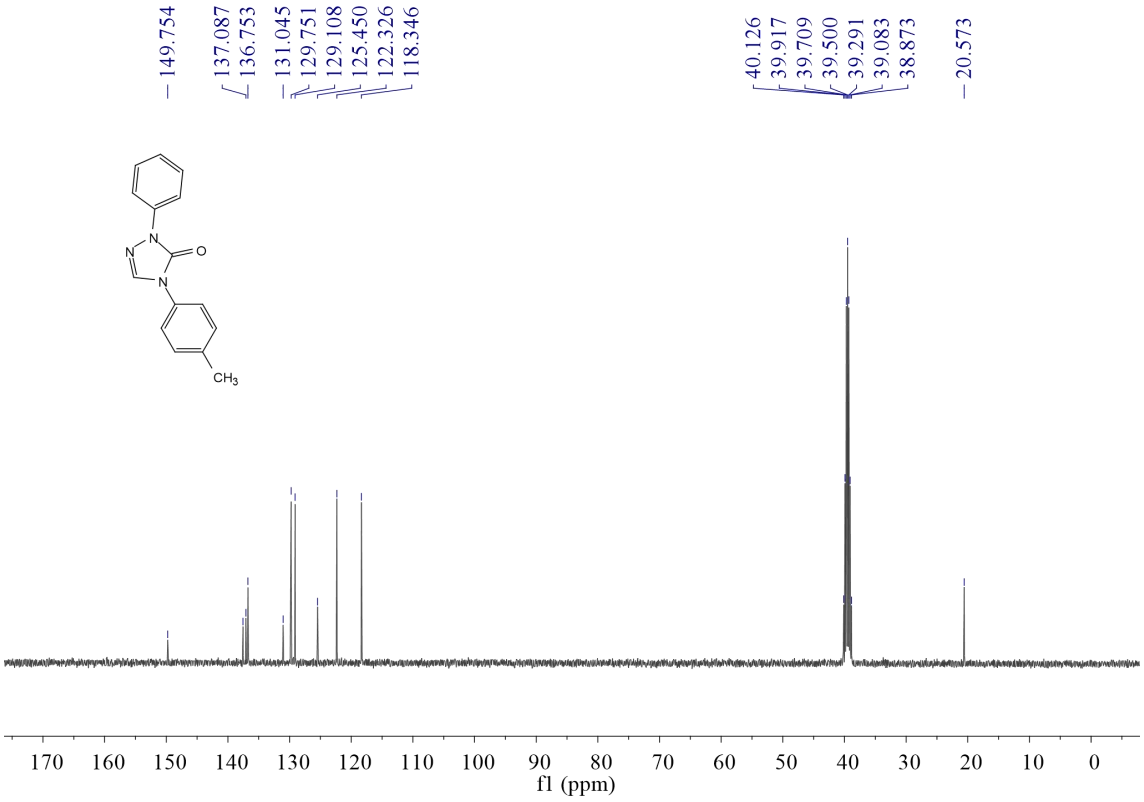
^{1}H NMR spectra of compound **4a**



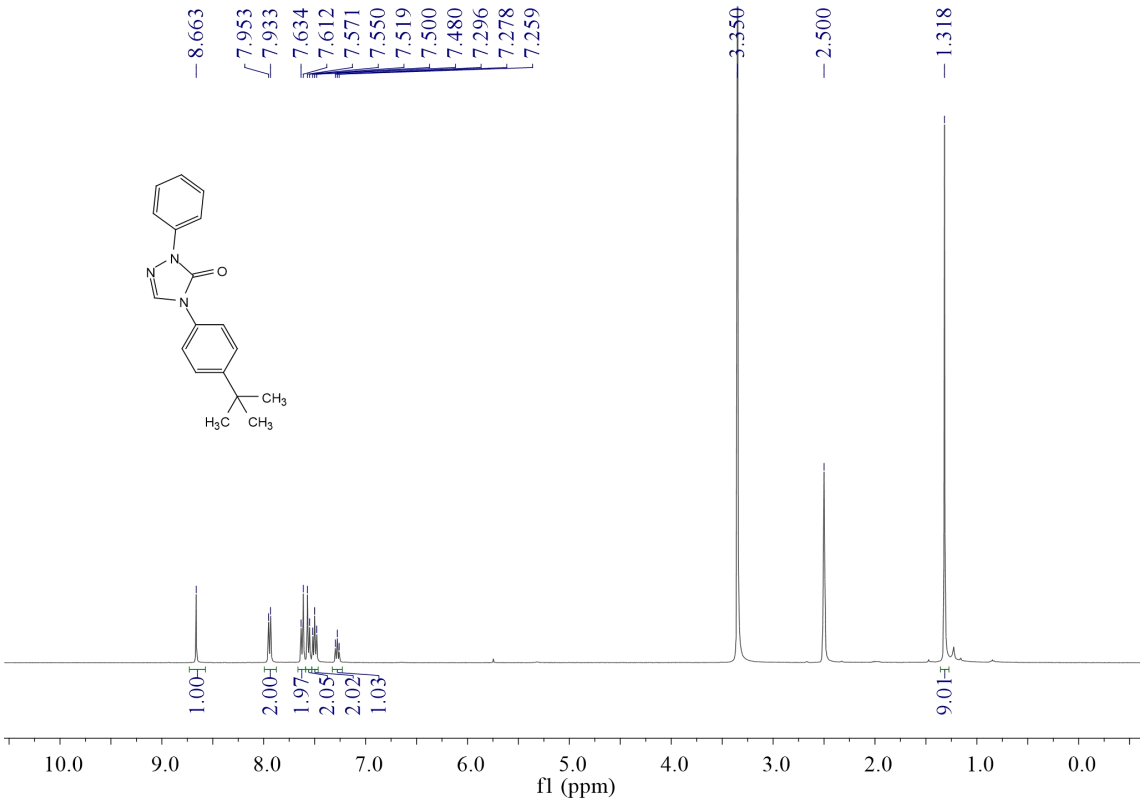
^{13}C NMR spectra of compound **4a**



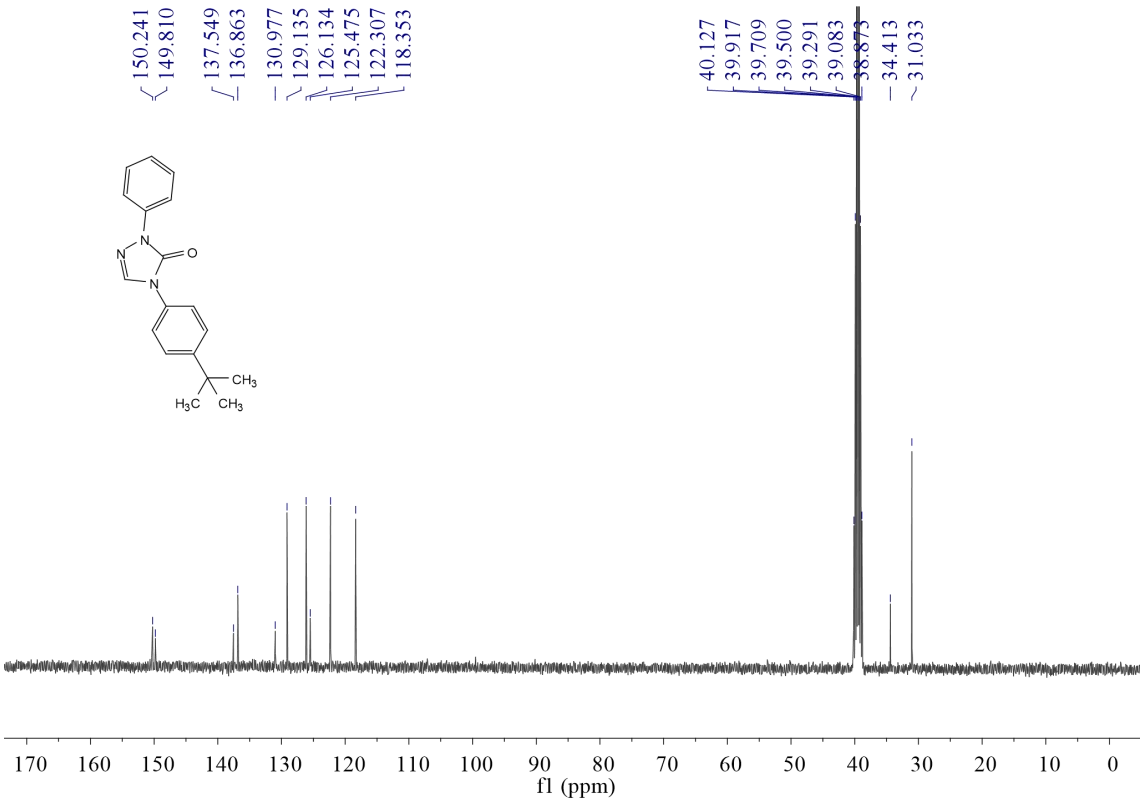
^{1}H NMR spectra of compound **4b**



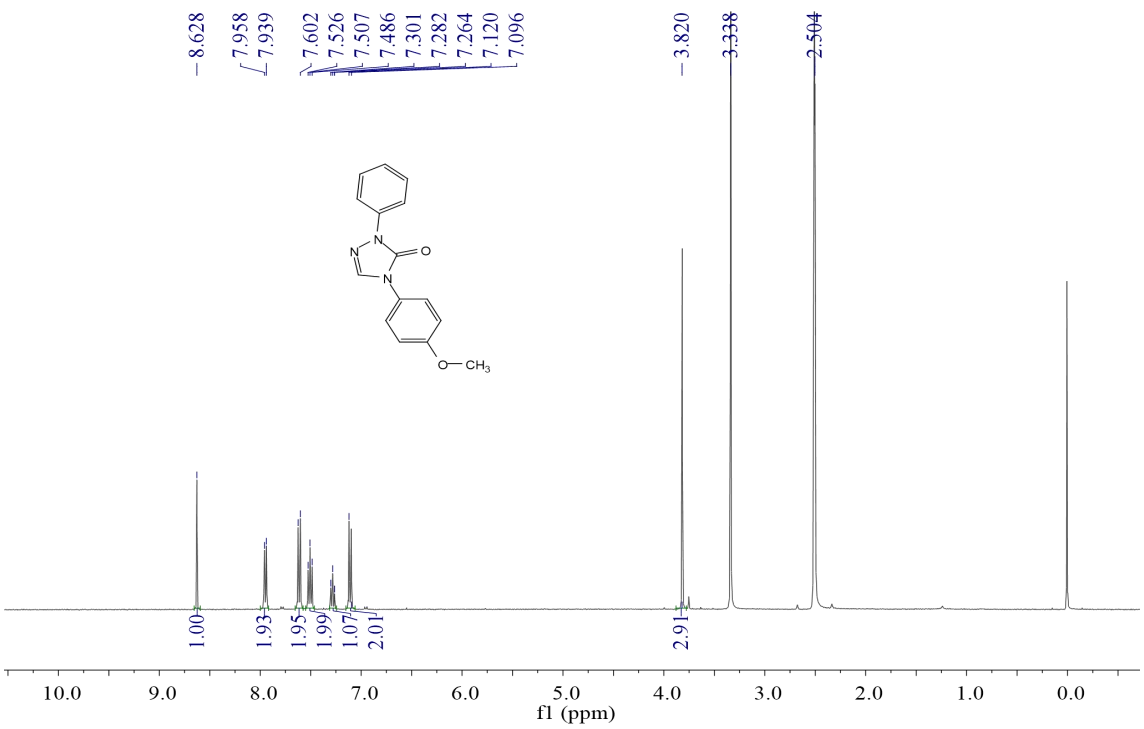
^{13}C NMR spectra of compound **4b**



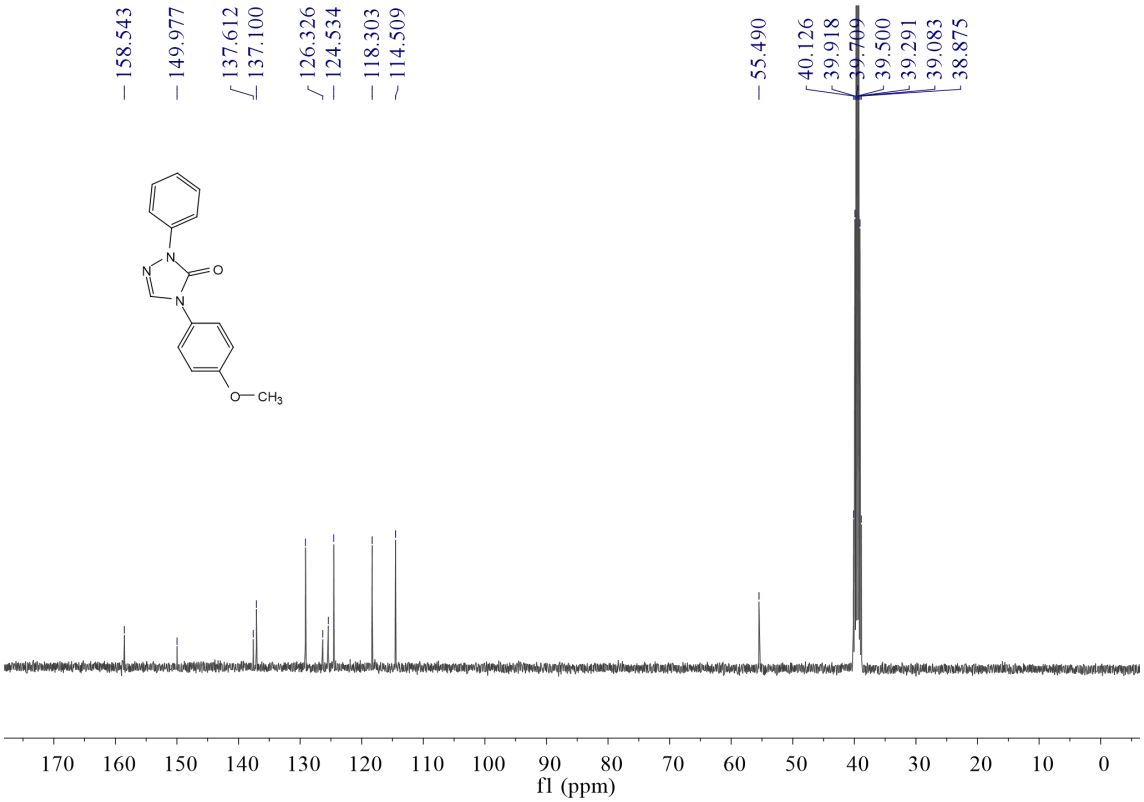
^{1}H NMR spectra of compound **4c**



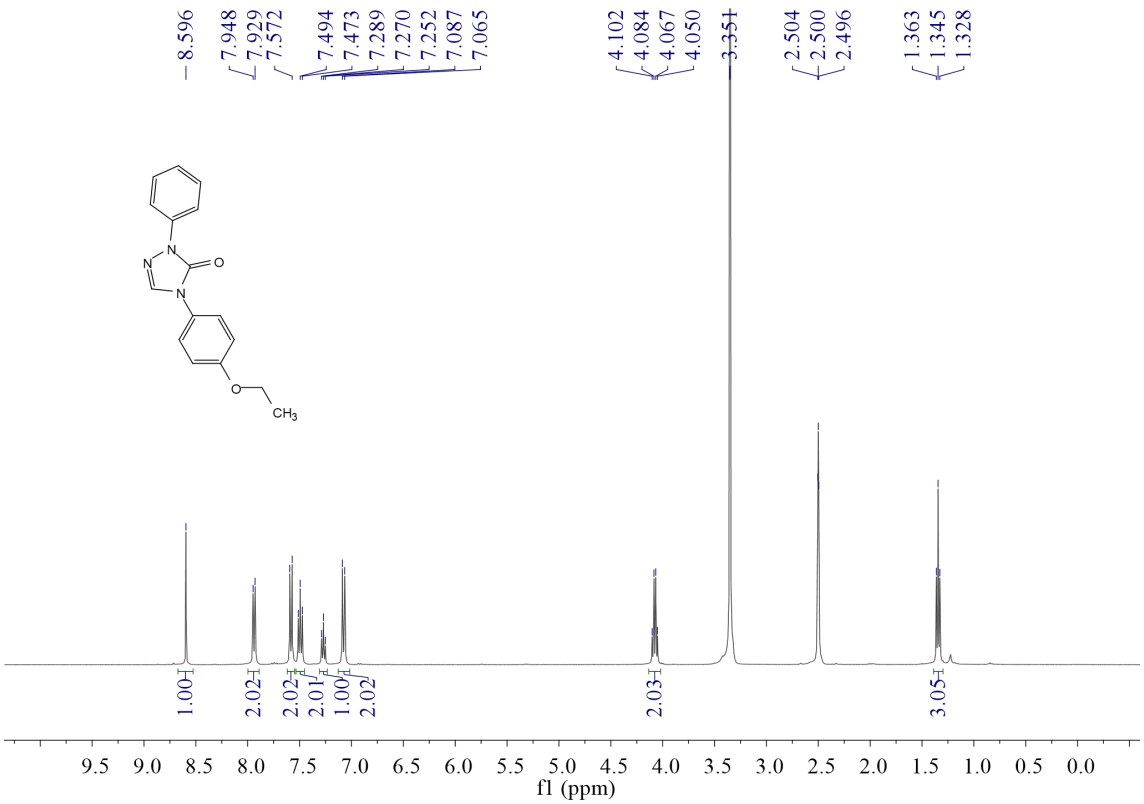
^{13}C NMR spectra of compound **4c**



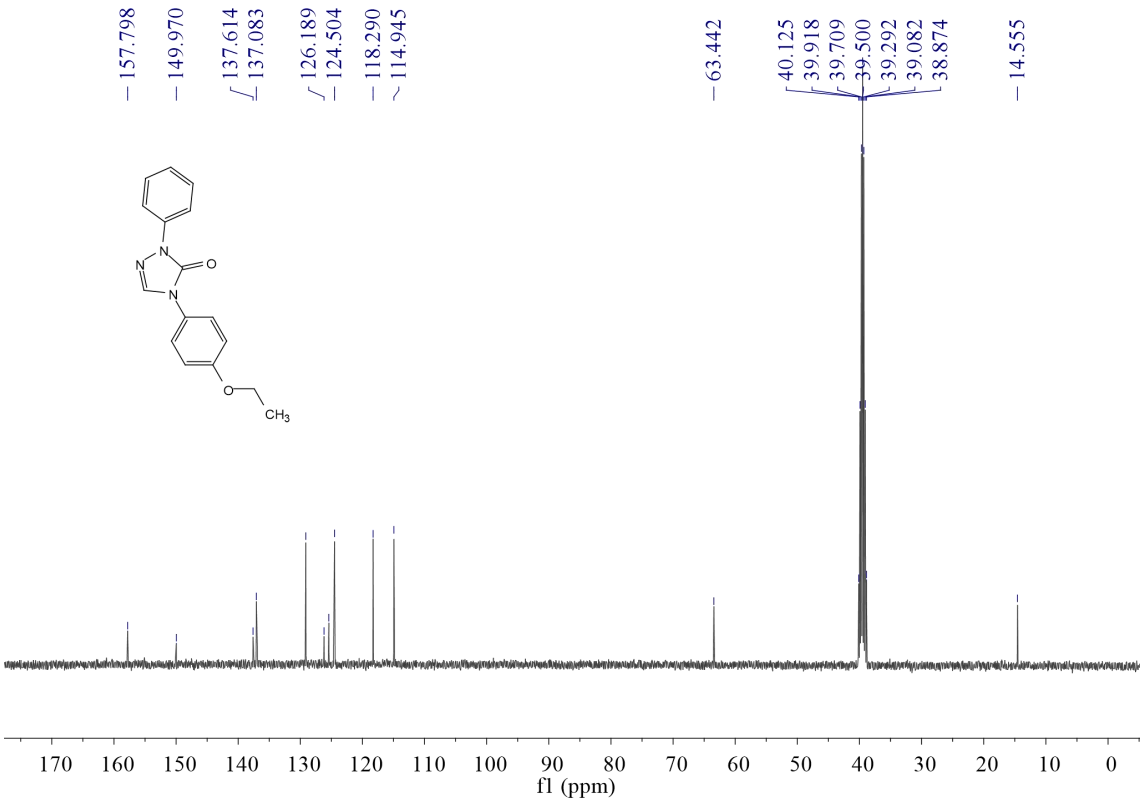
^{1}H NMR spectra of compound **4d**



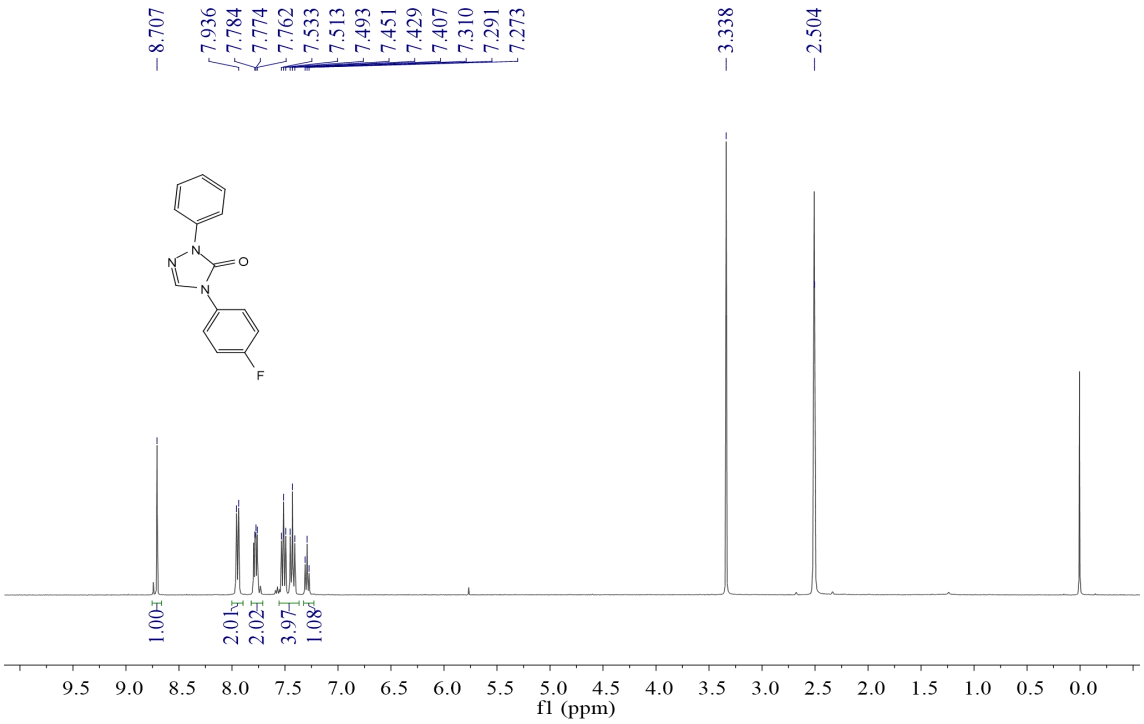
^{13}C NMR spectra of compound **4d**



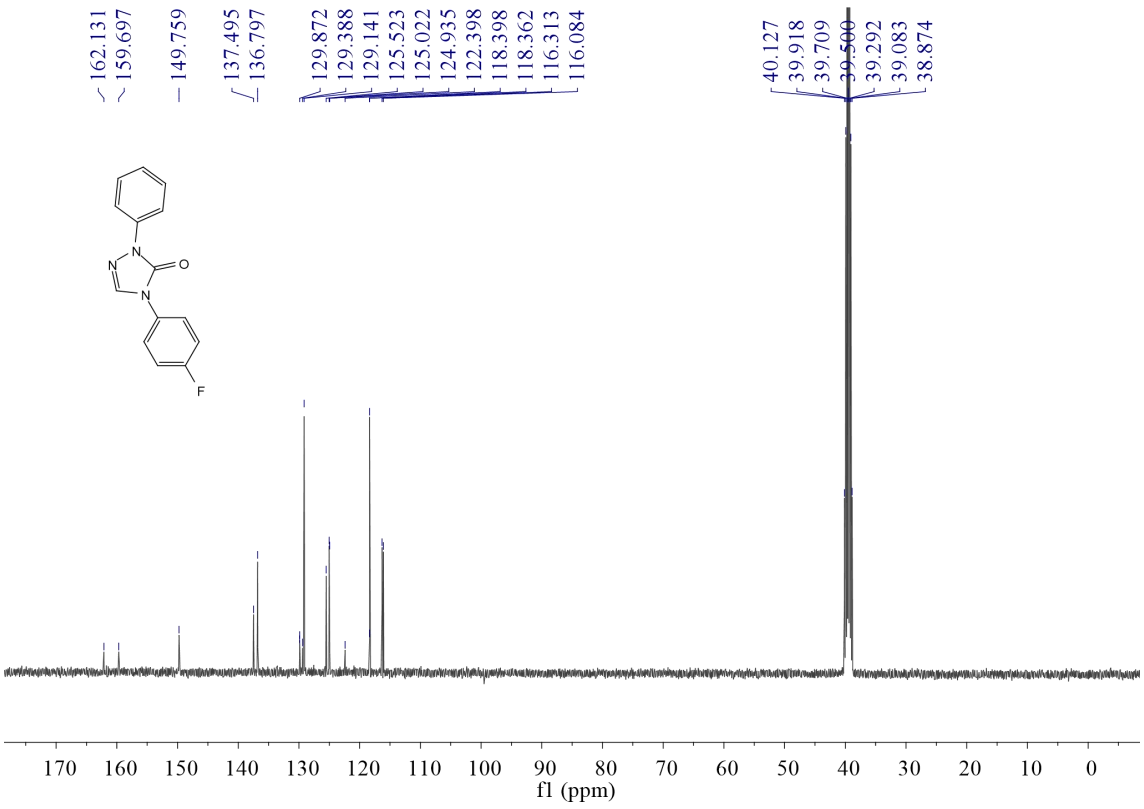
^{1}H NMR spectra of compound **4e**



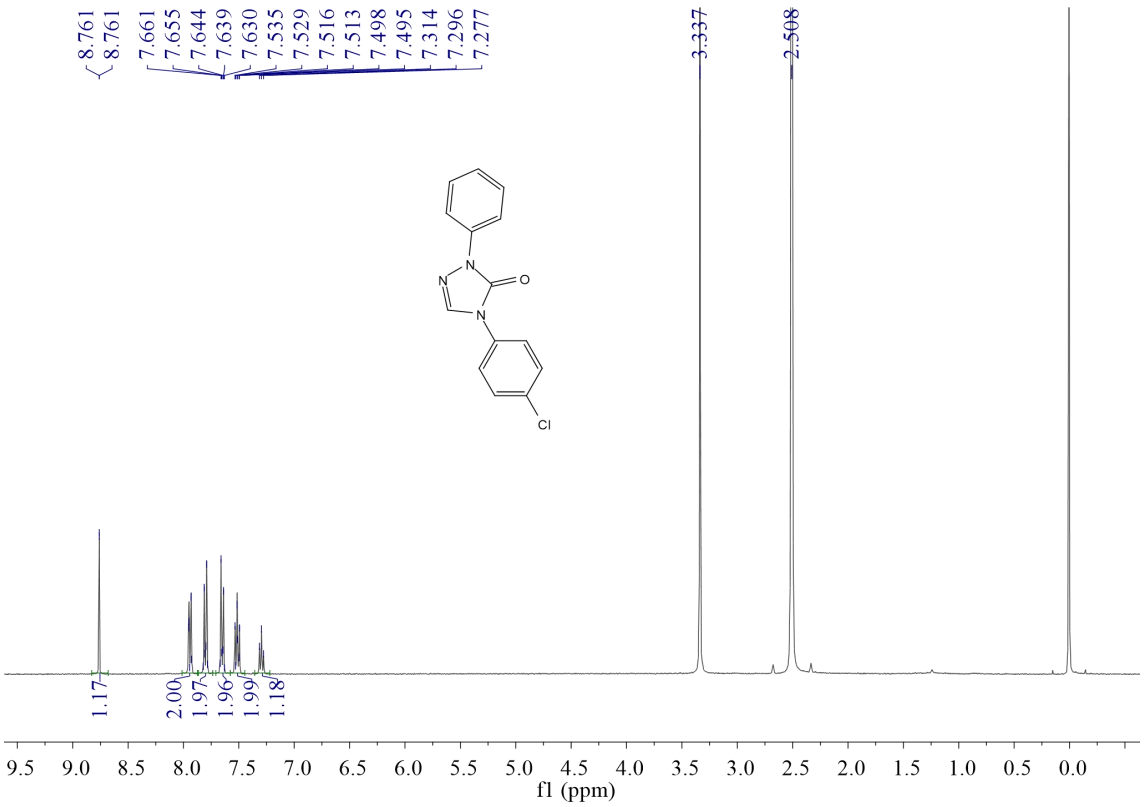
^{13}C NMR spectra of compound **4e**



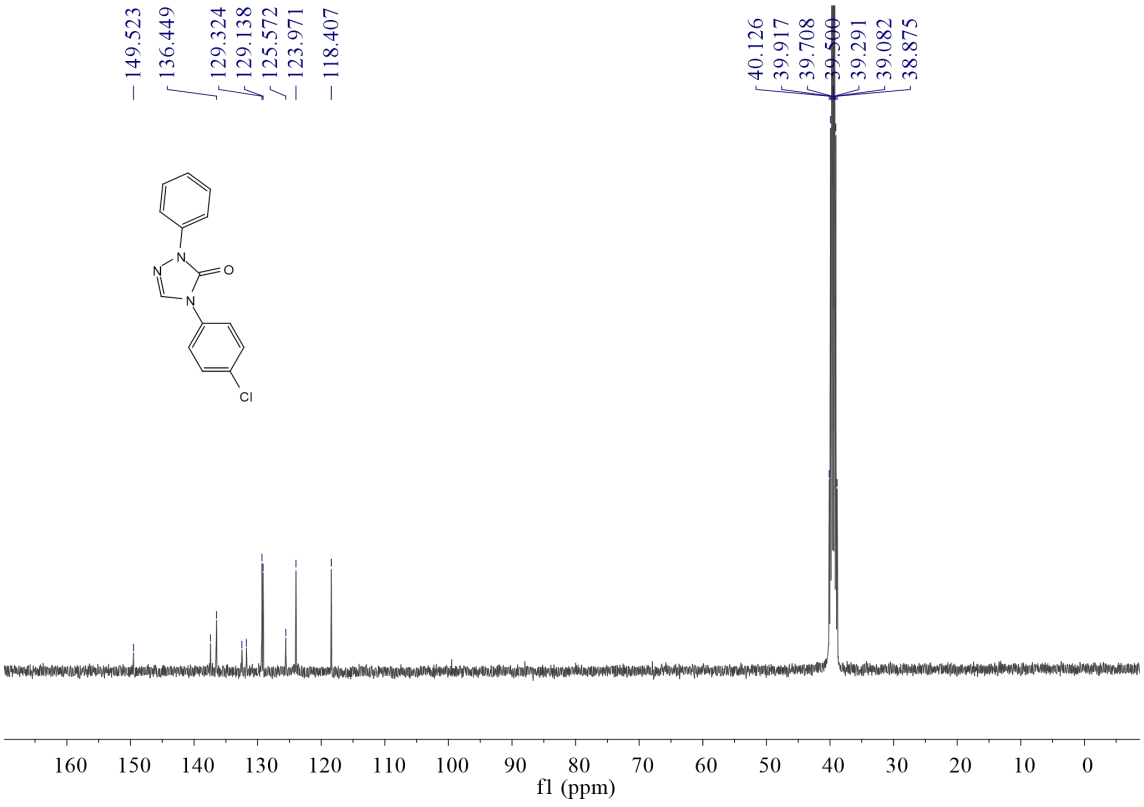
^{1}H NMR spectra of compound **4f**



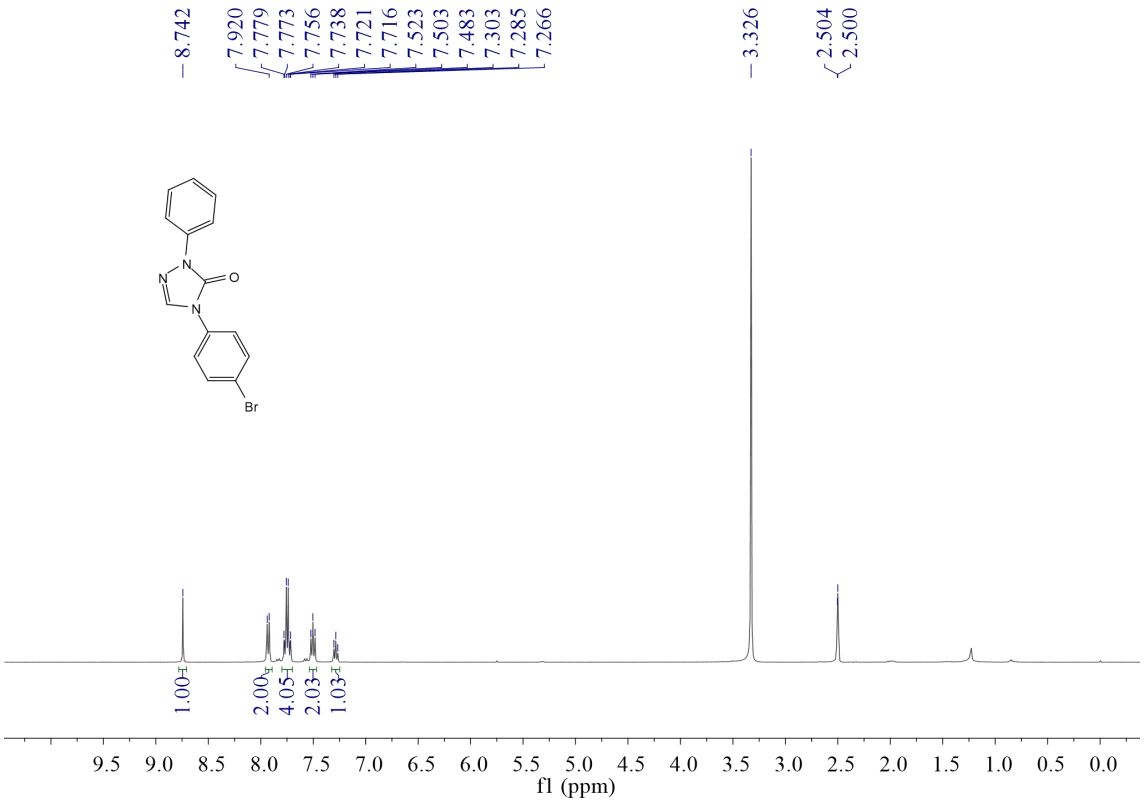
^{13}C NMR spectra of compound **4f**



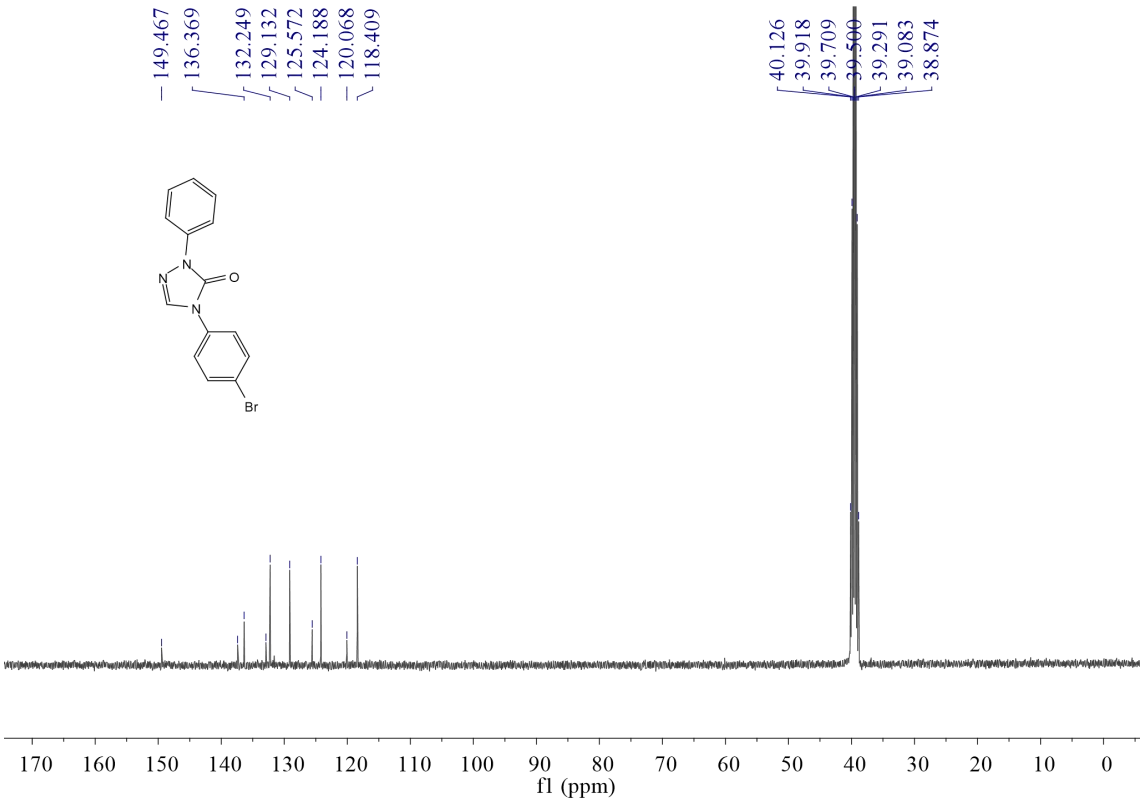
^{1}H NMR spectra of compound **4g**



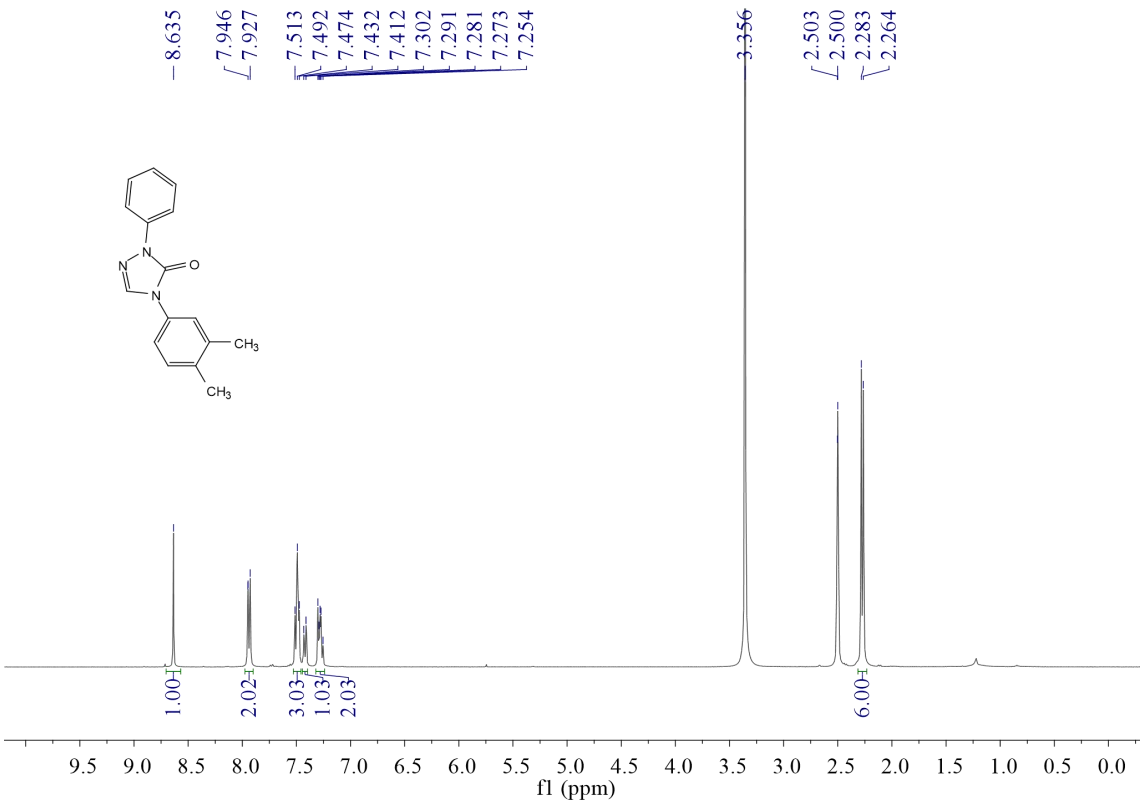
^{13}C NMR spectra of compound **4g**



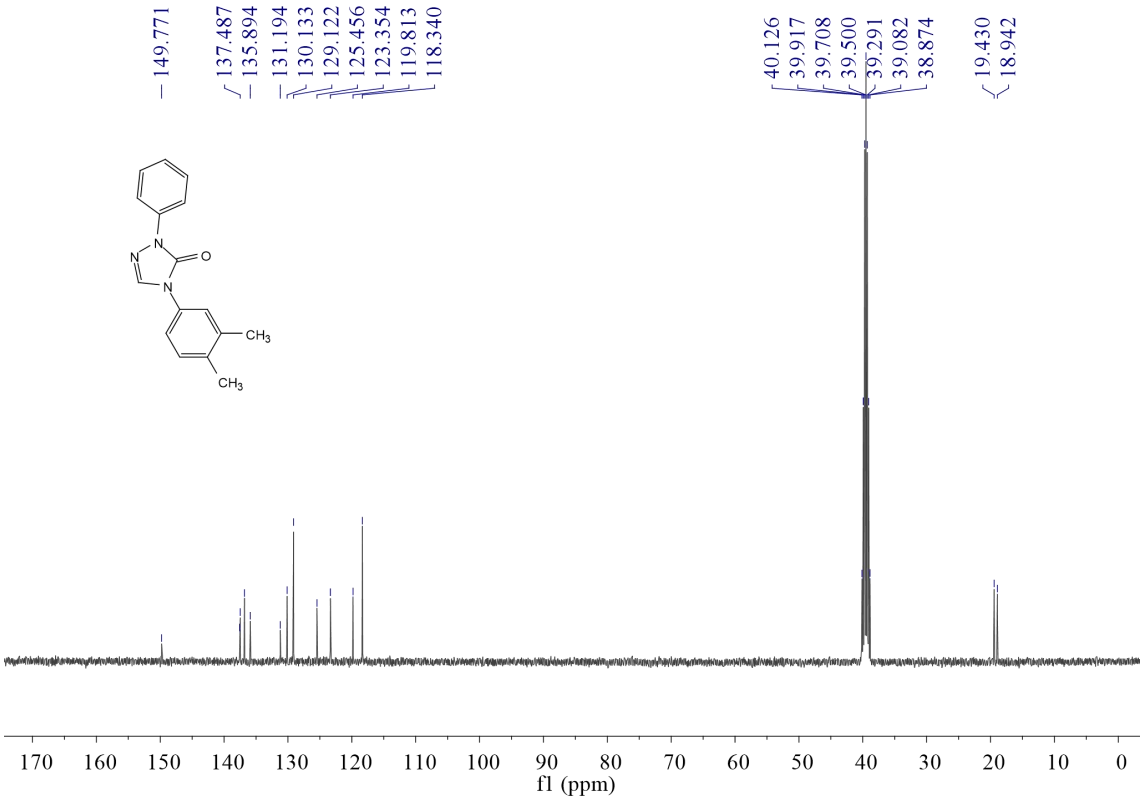
^{1}H NMR spectra of compound **4h**



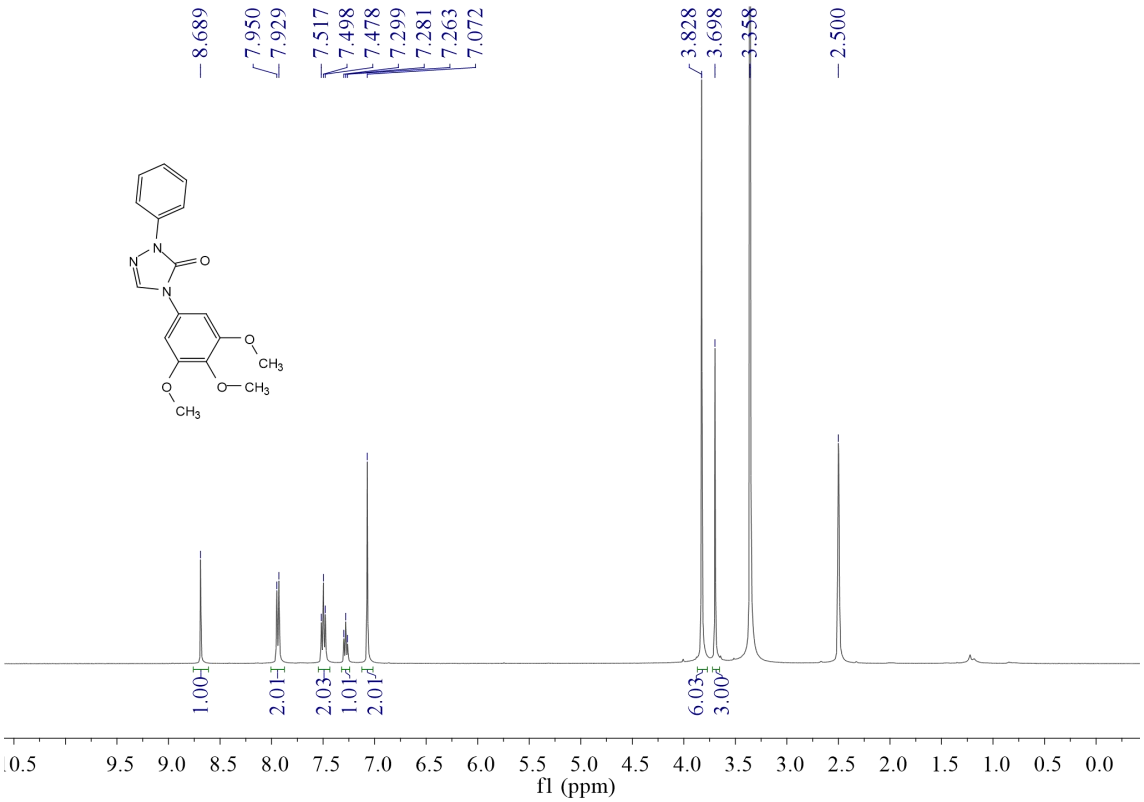
^{13}C NMR spectra of compound **4h**



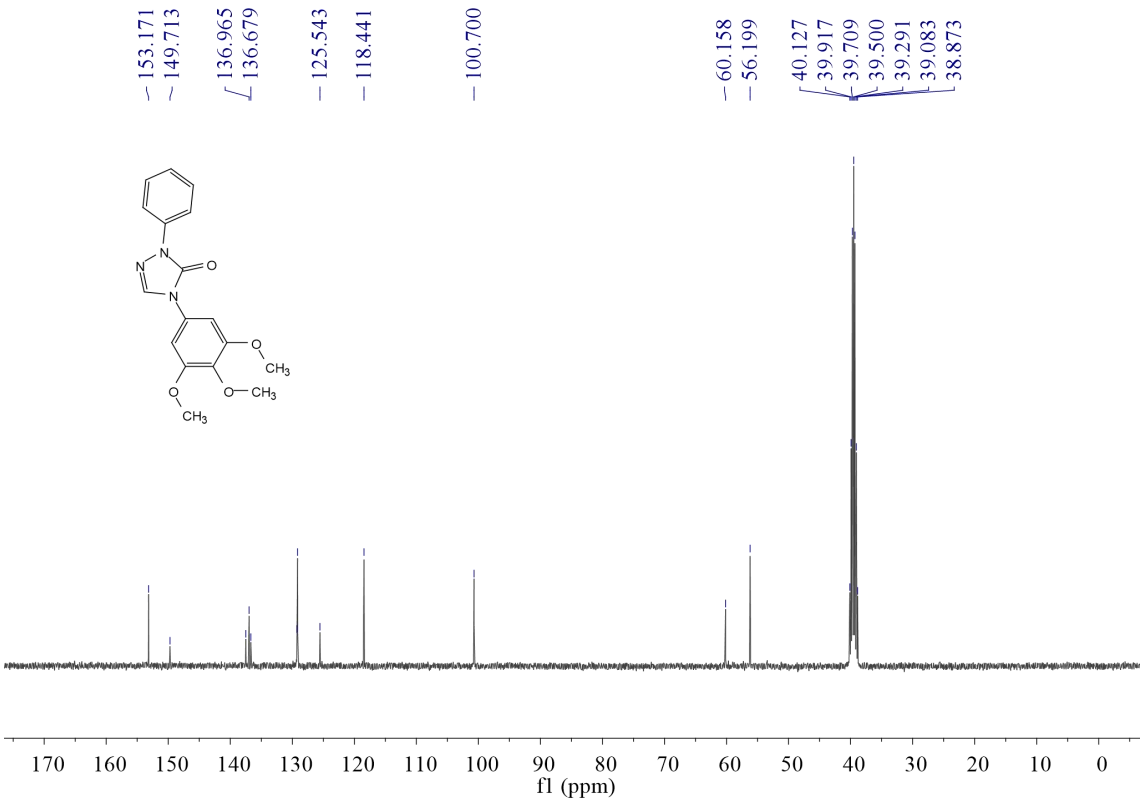
^{1}H NMR spectra of compound **4i**



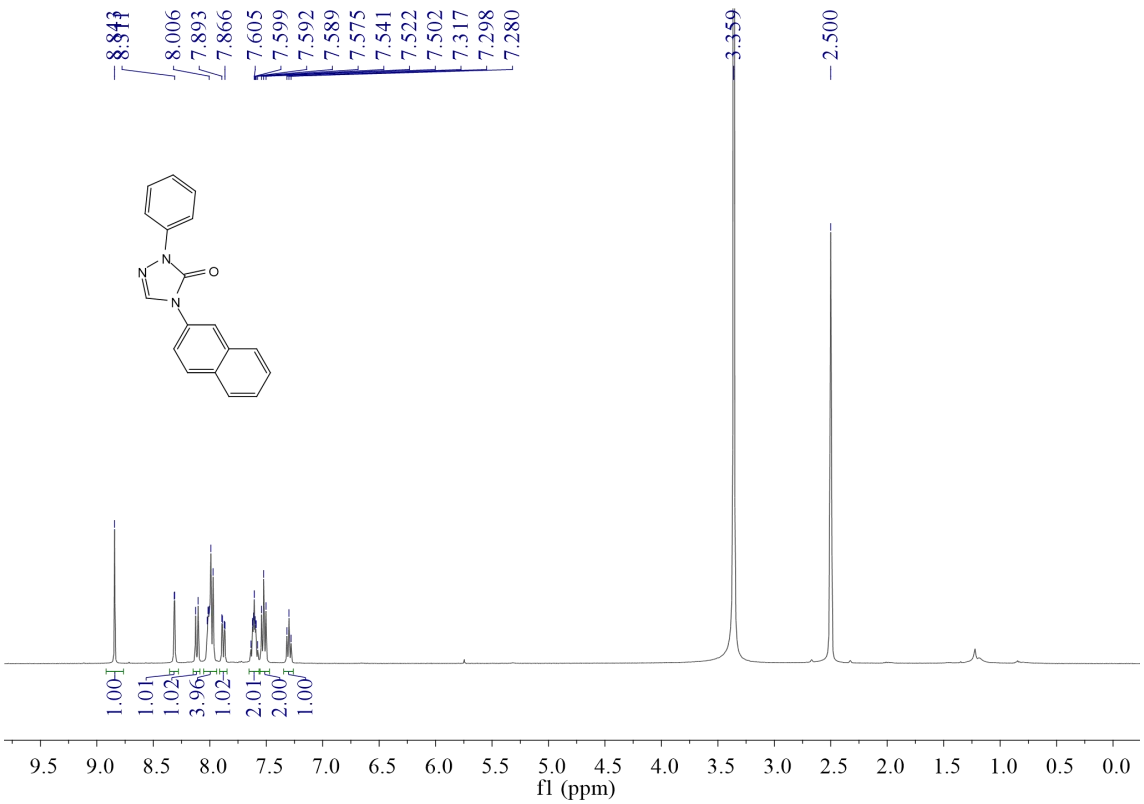
^{13}C NMR spectra of compound **4i**



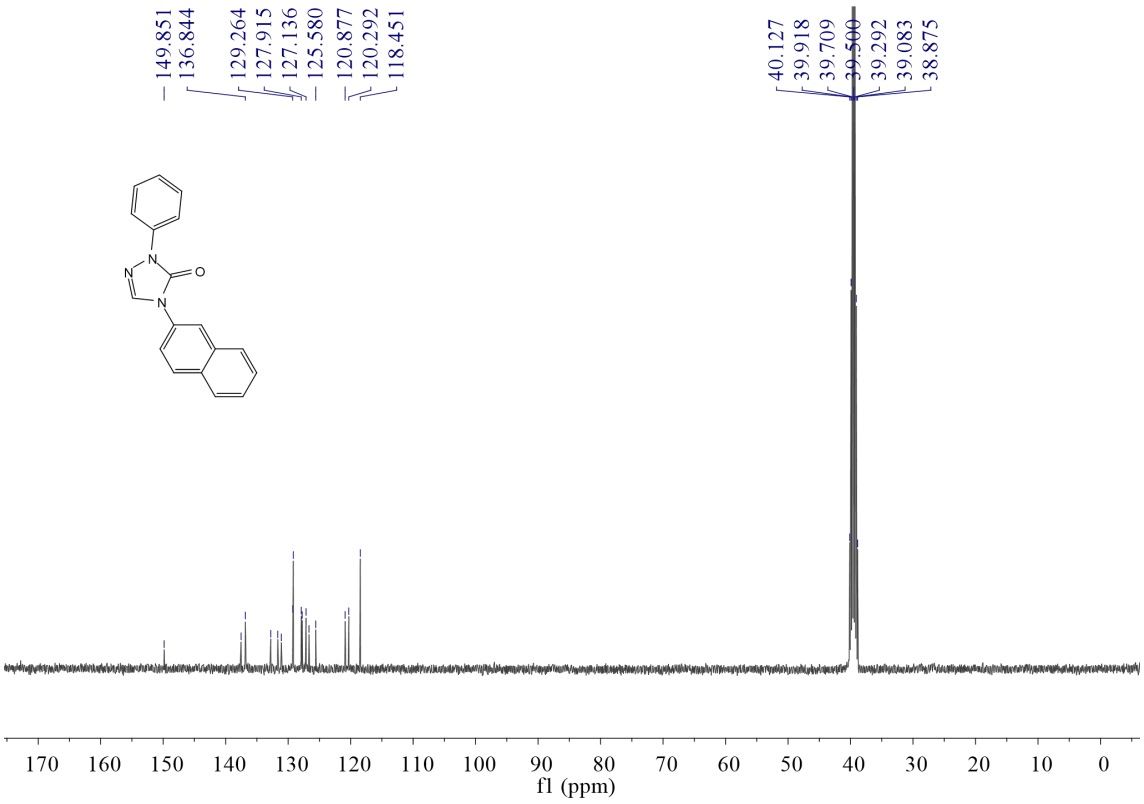
^{1}H NMR spectra of compound **4j**



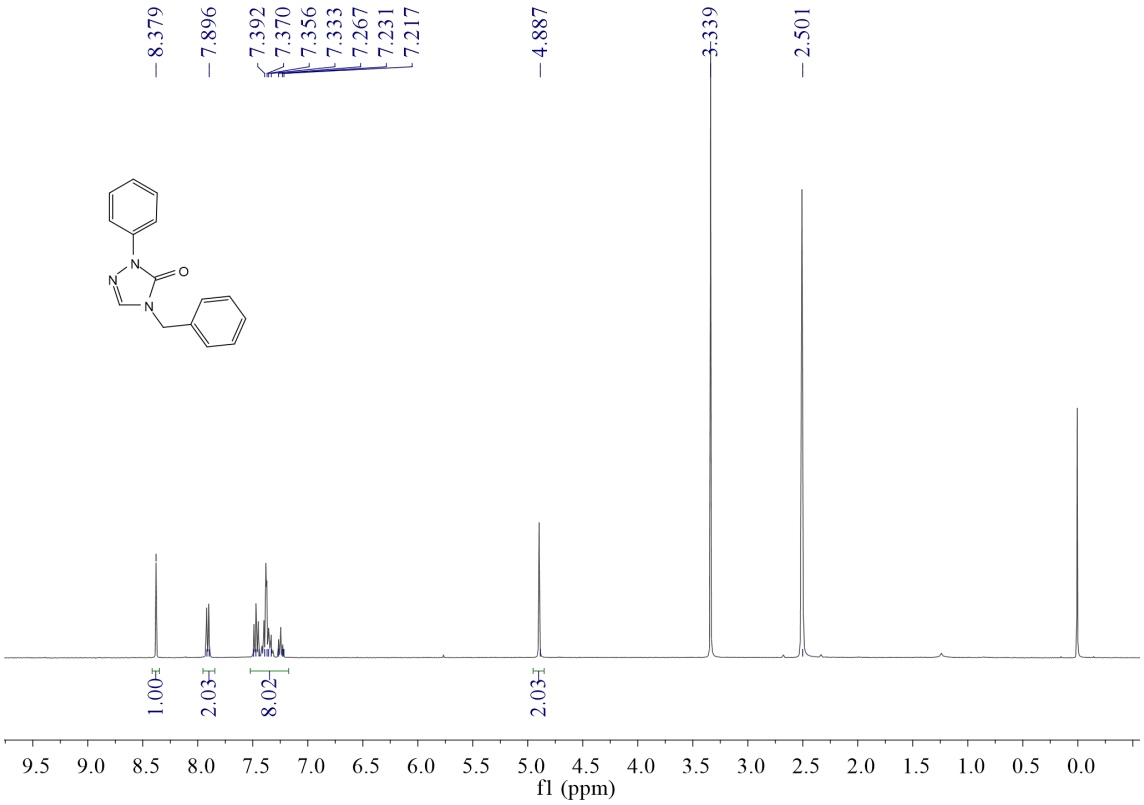
^{13}C NMR spectra of compound **4j**



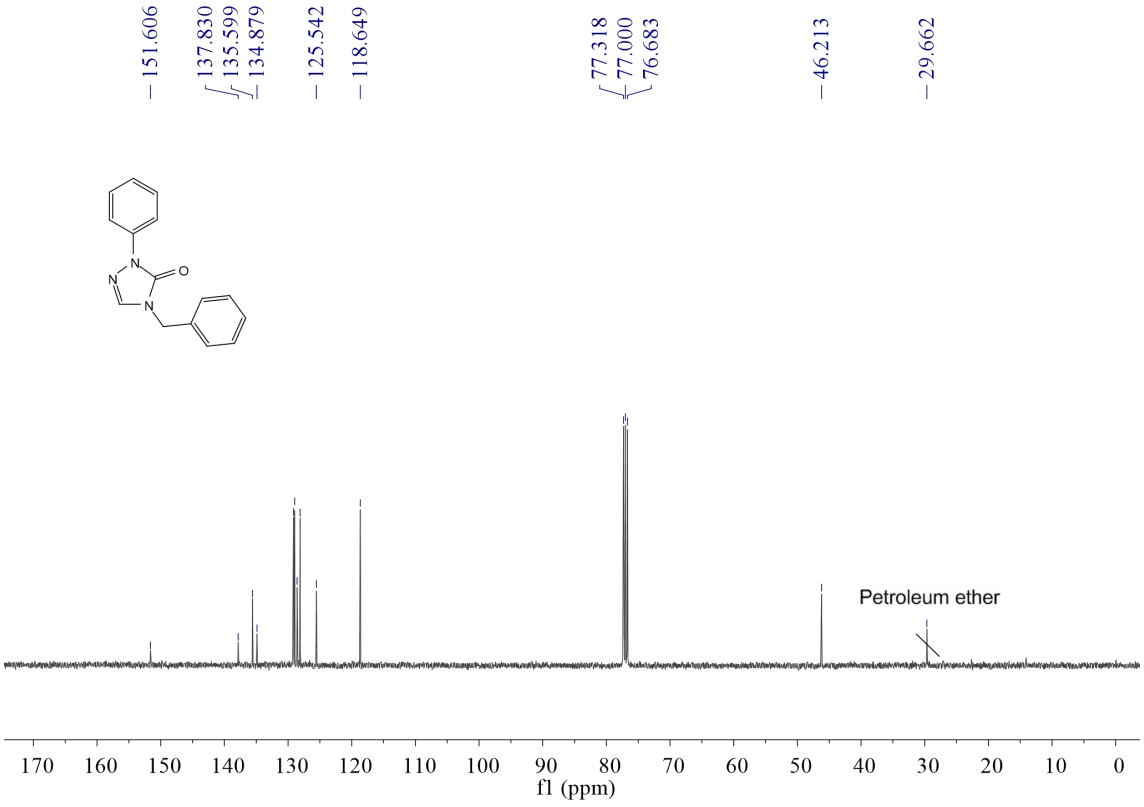
^{1}H NMR spectra of compound **4k**



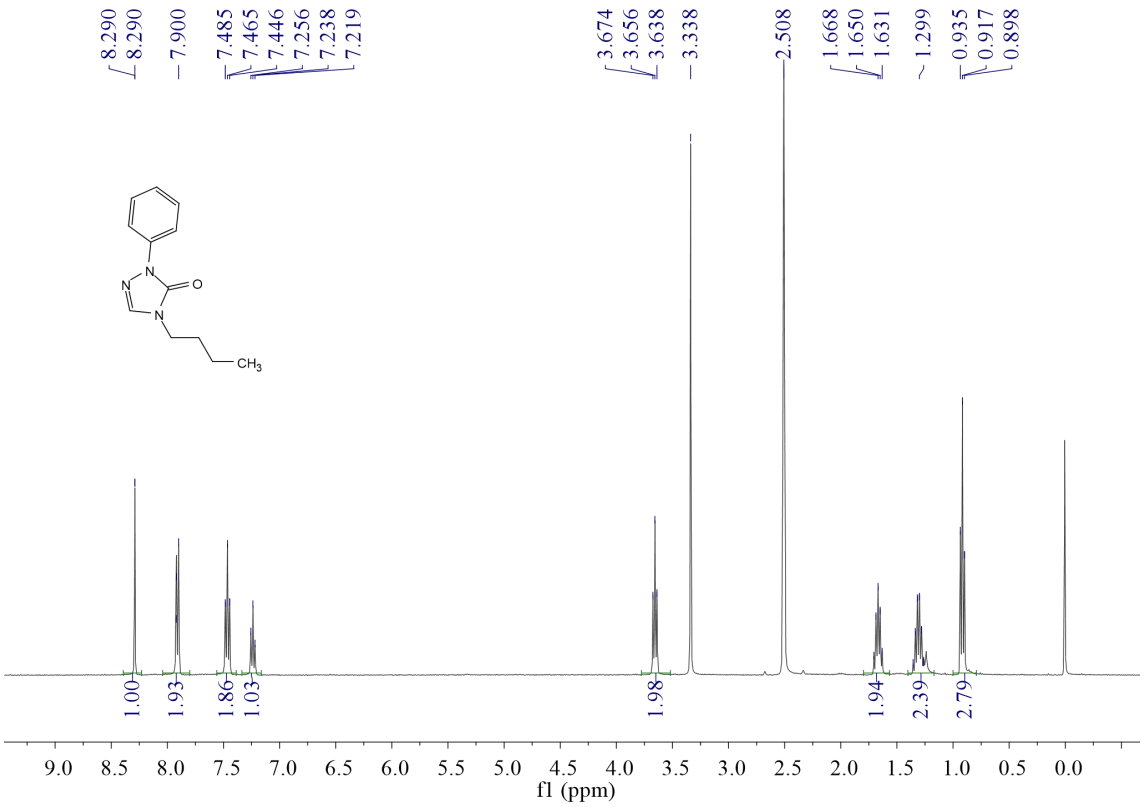
^{13}C NMR spectra of compound **4k**



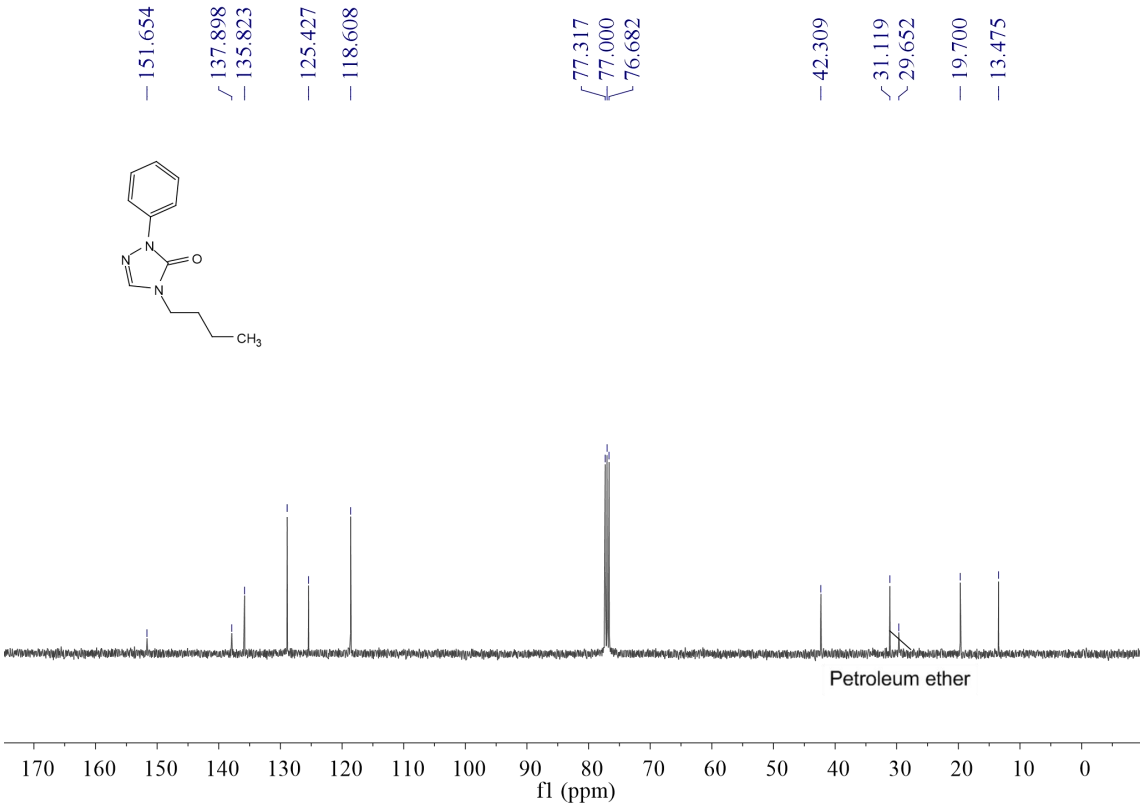
^{1}H NMR spectra of compound **4l**



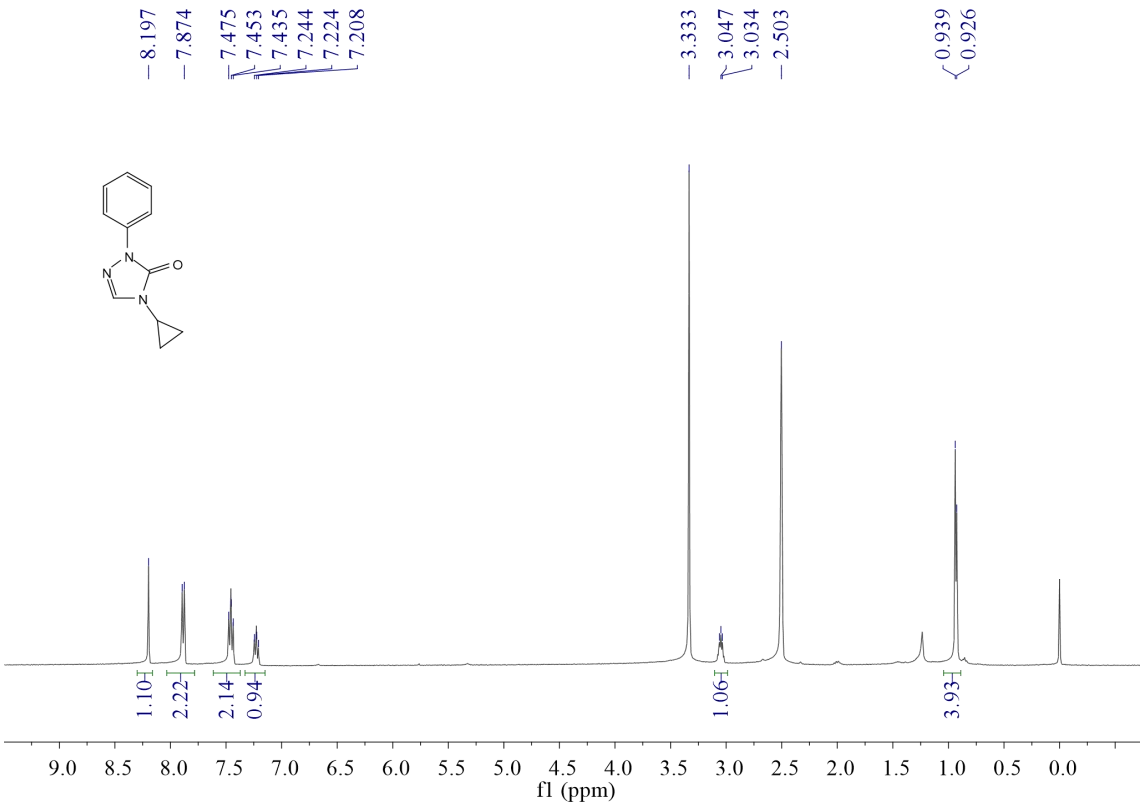
^{13}C NMR spectra of compound **4l**



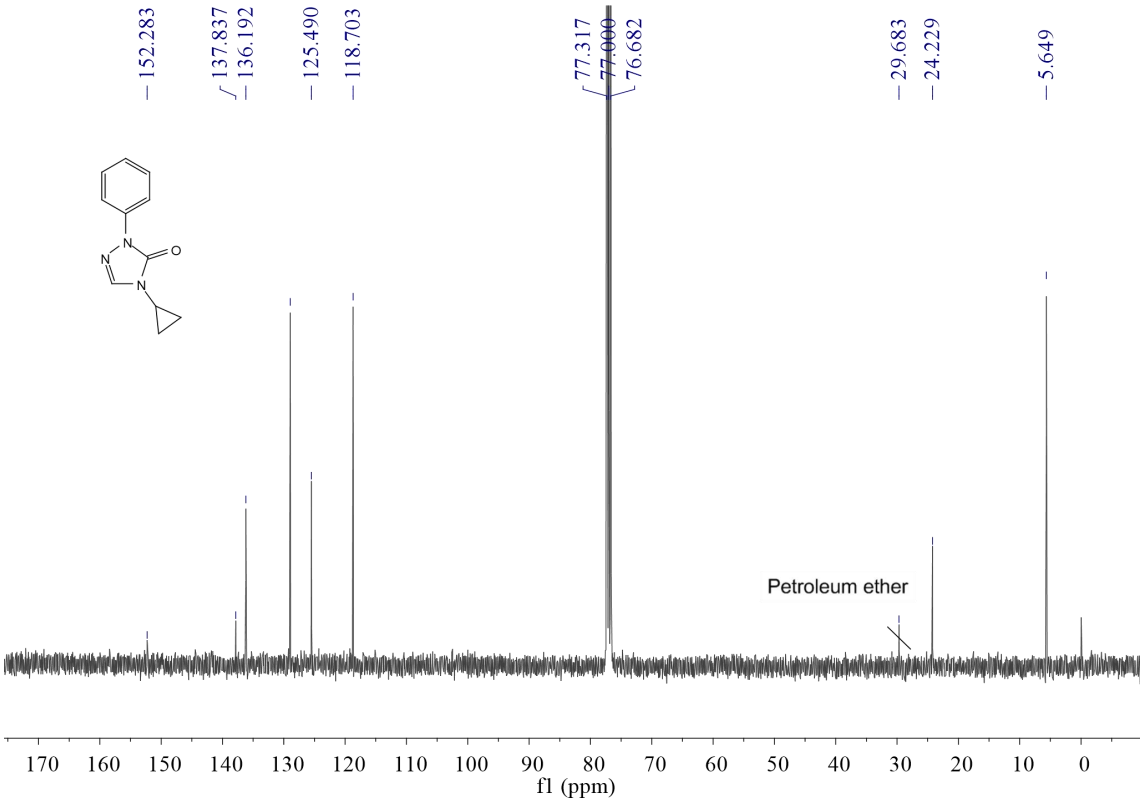
^{1}H NMR spectra of compound **4m**



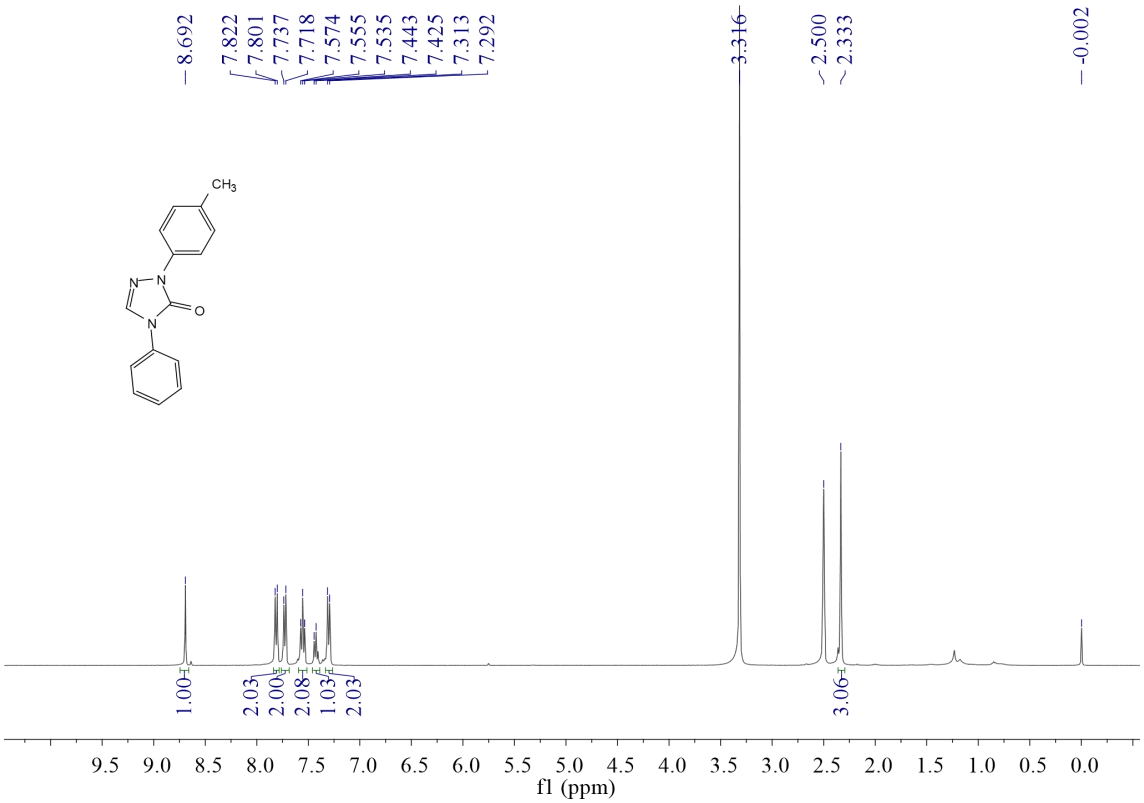
^{13}C NMR spectra of compound **4m**



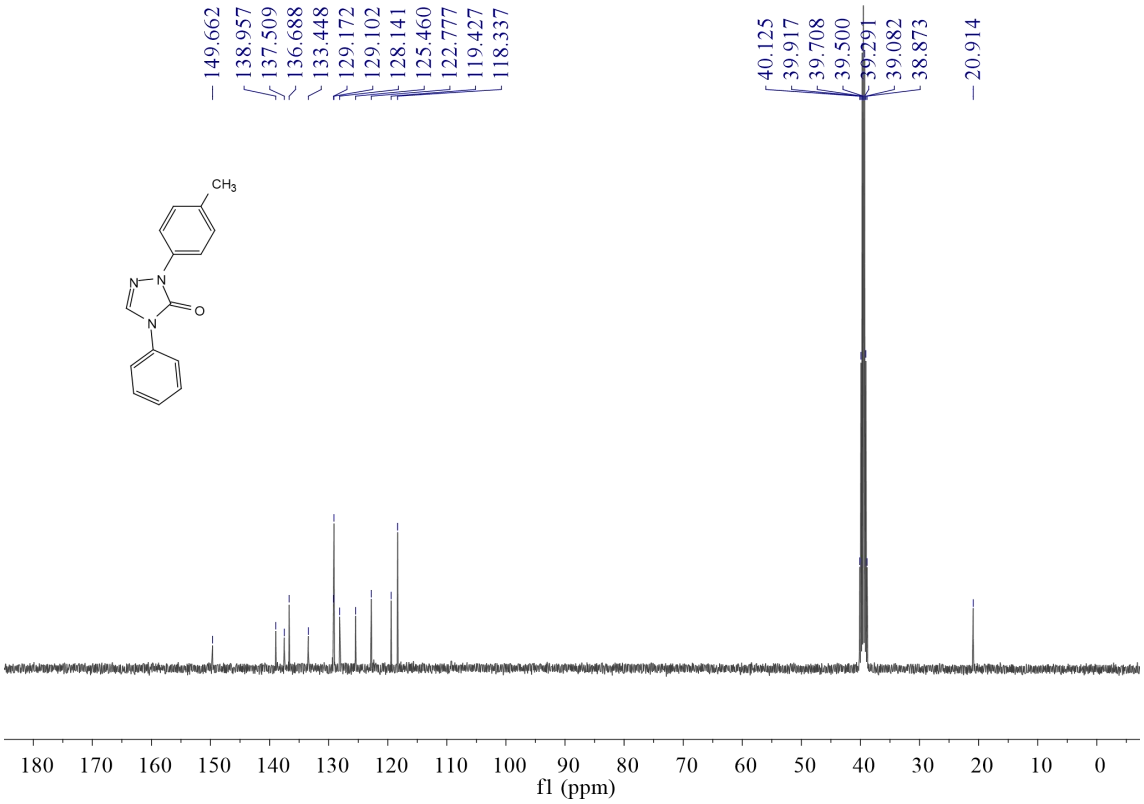
^{1}H NMR spectra of compound **4n**



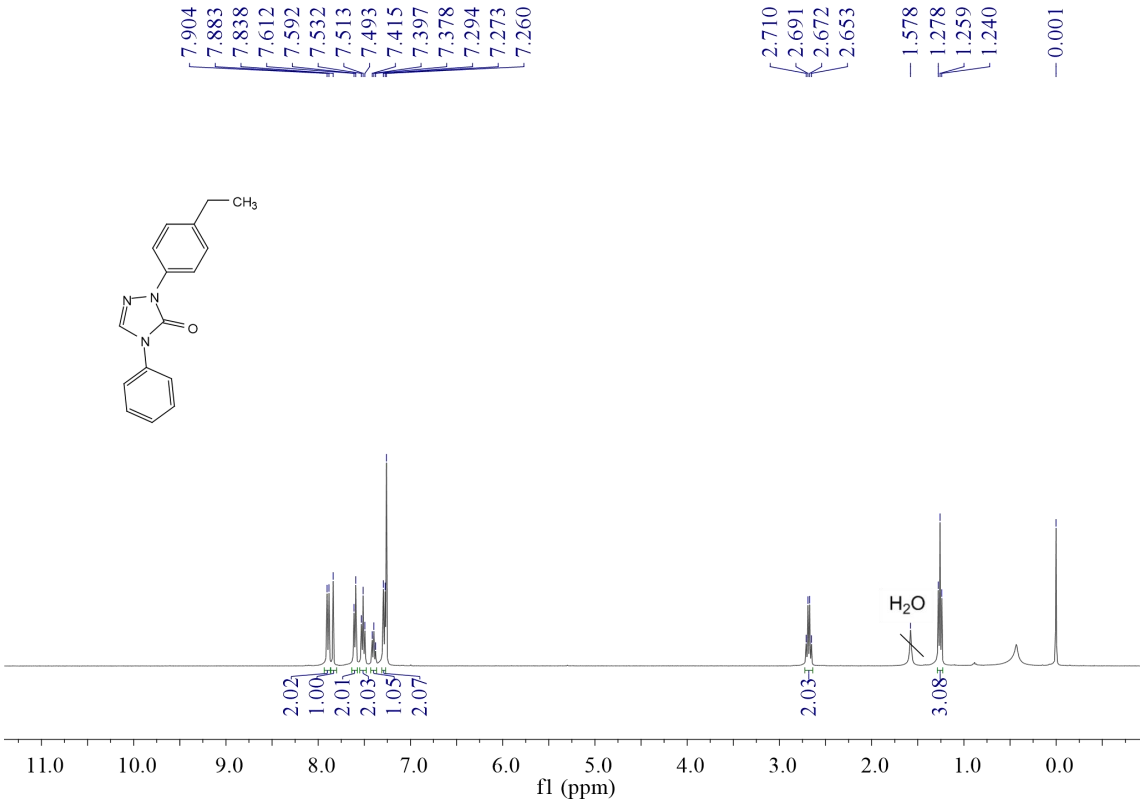
^{13}C NMR spectra of compound **4n**



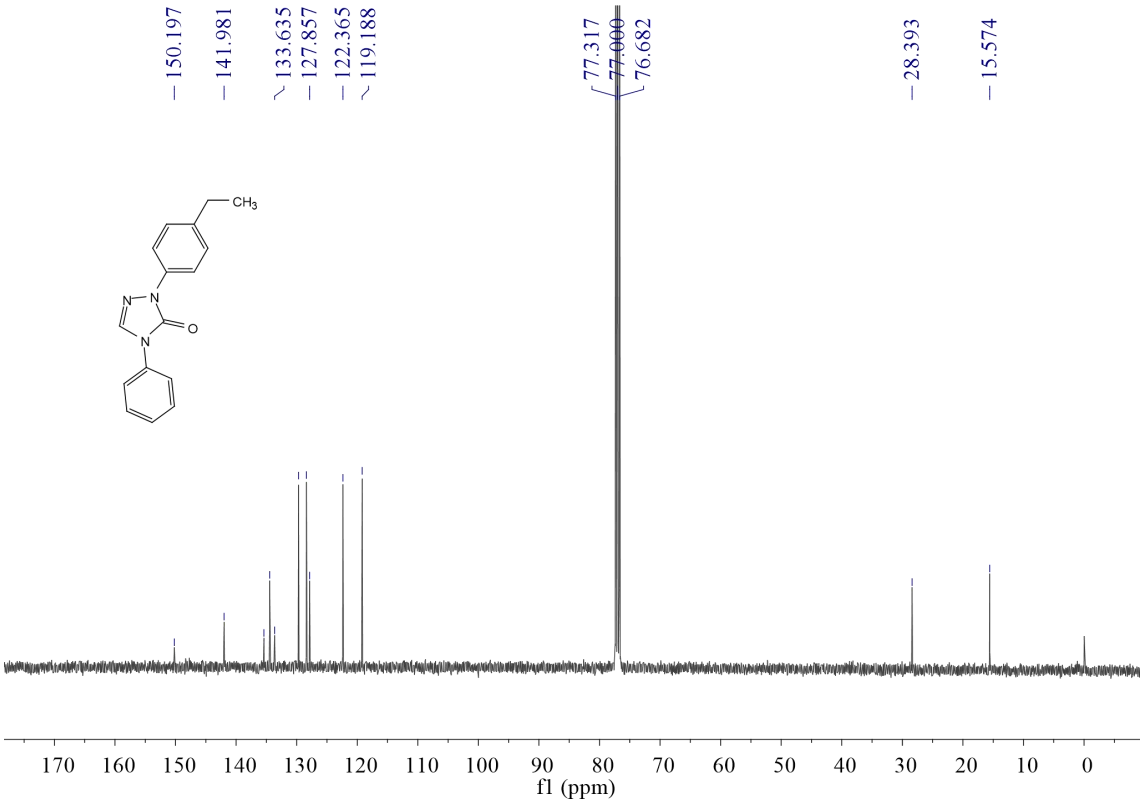
^{1}H NMR spectra of compound **4o**



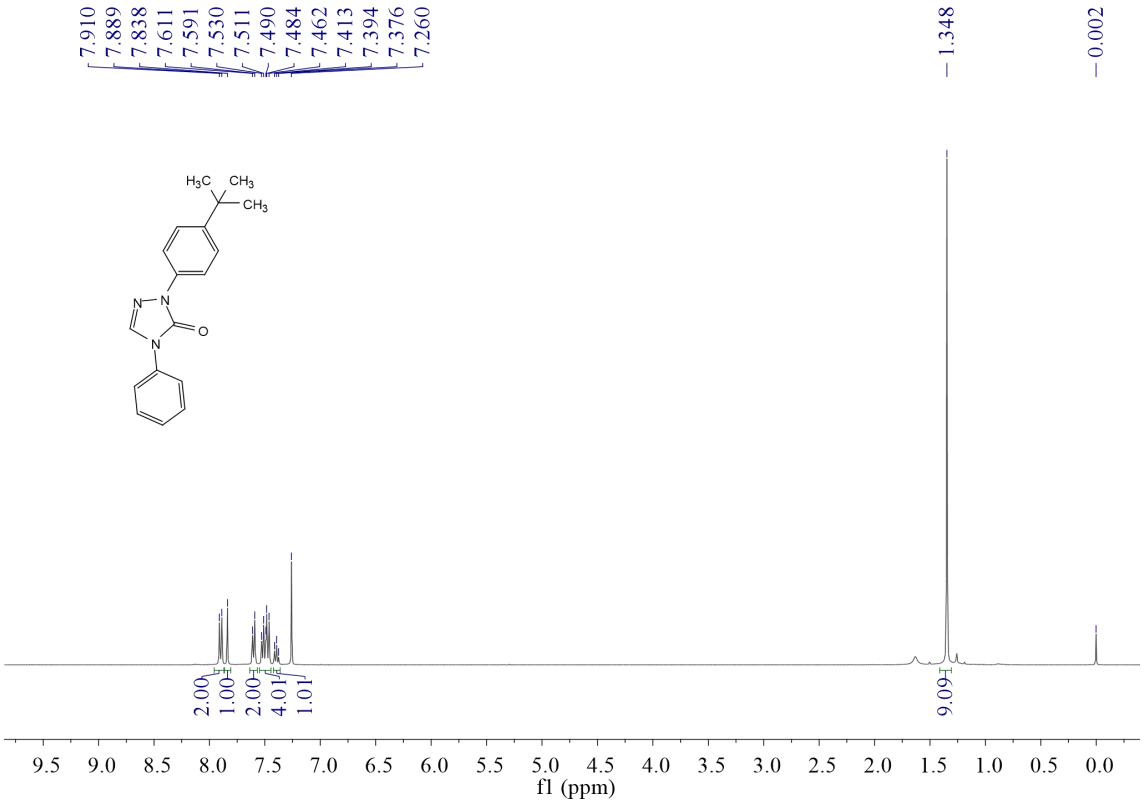
^{13}C NMR spectra of compound **4o**



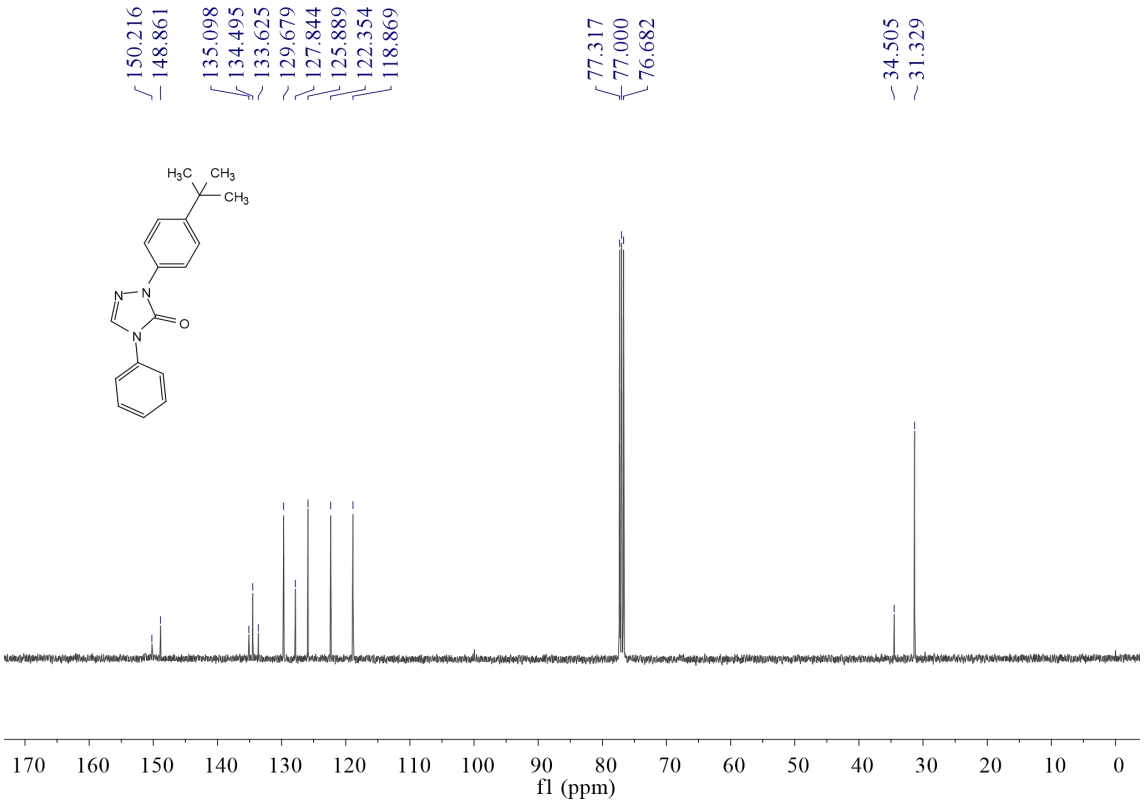
^{1}H NMR spectra of compound **4p**



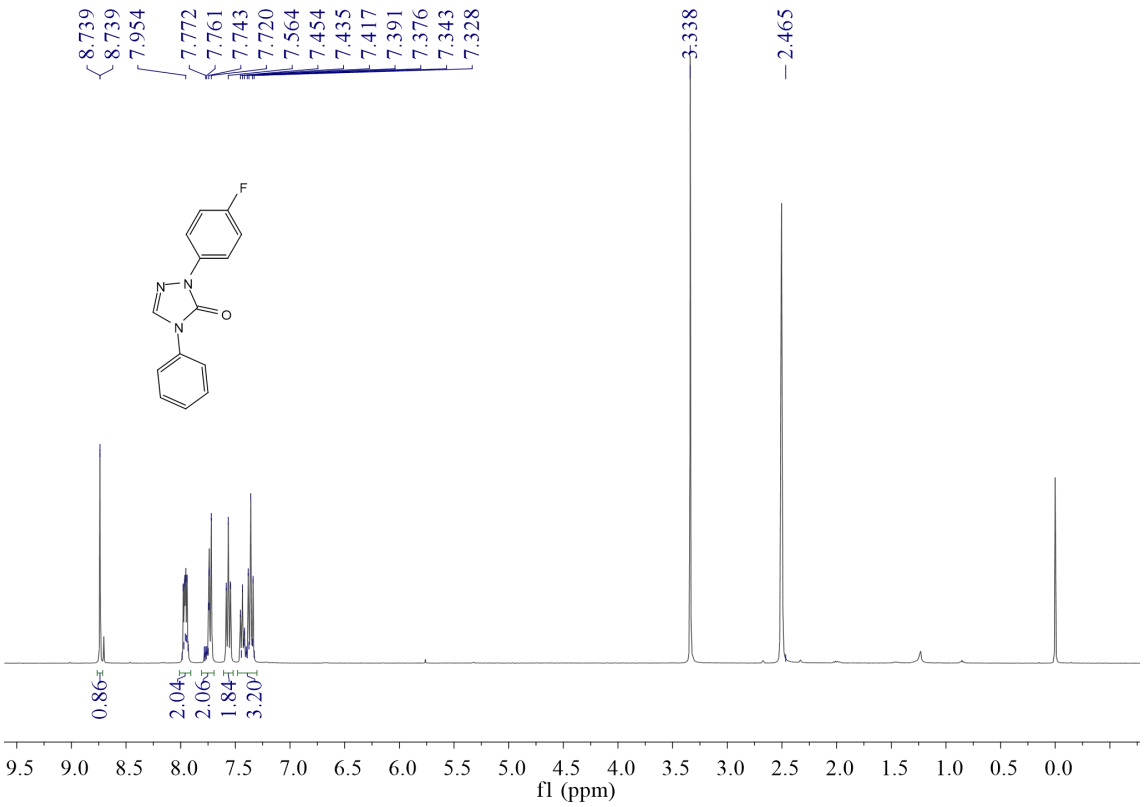
^{13}C NMR spectra of compound **4p**



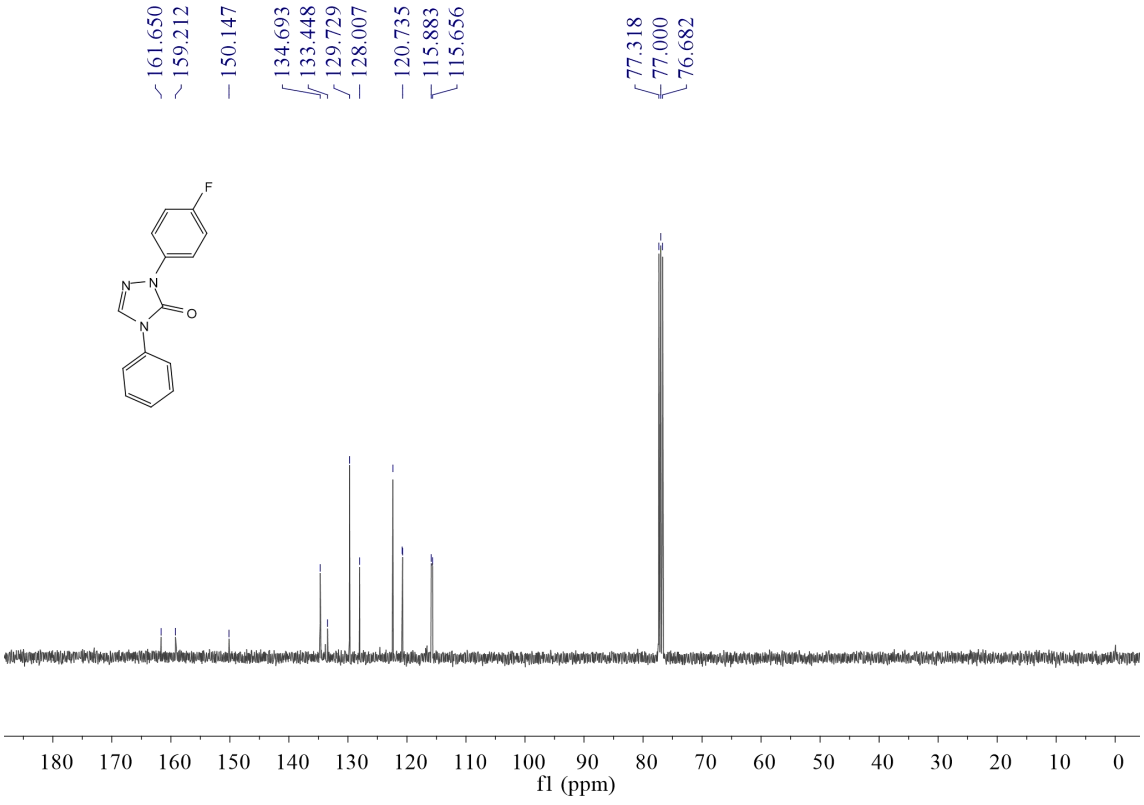
^{1}H NMR spectra of compound **4q**



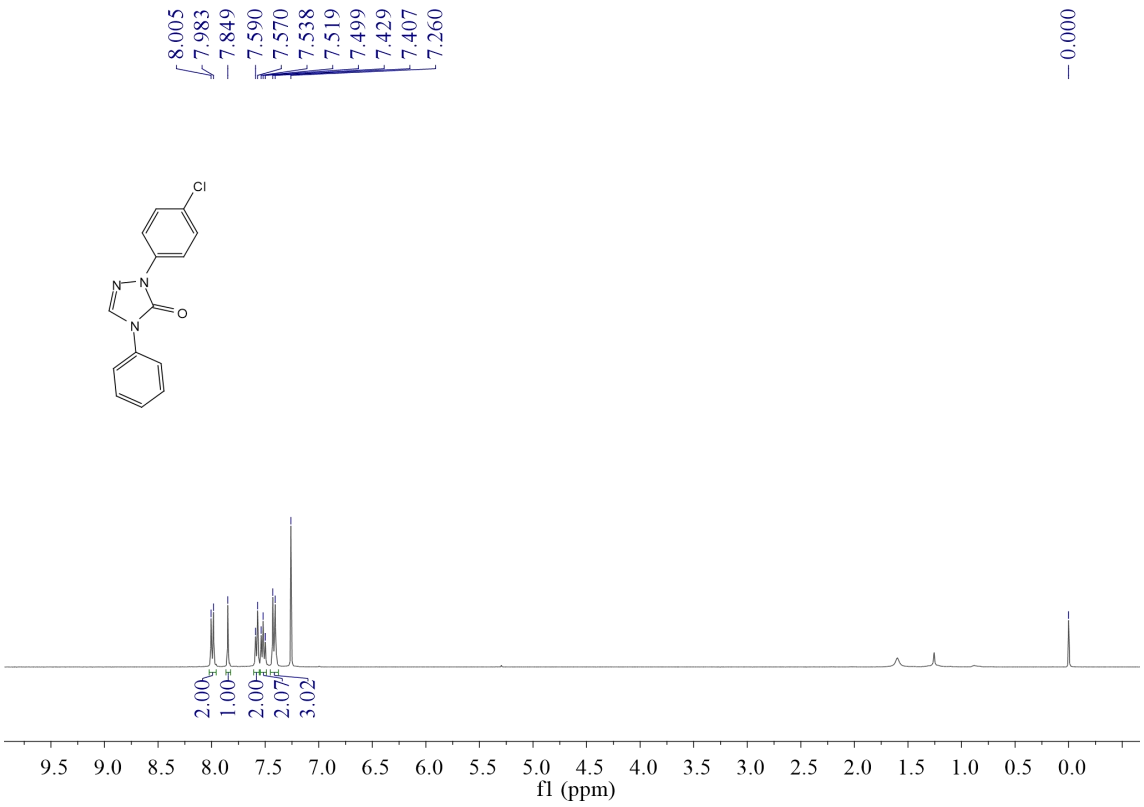
^{13}C NMR spectra of compound **4q**



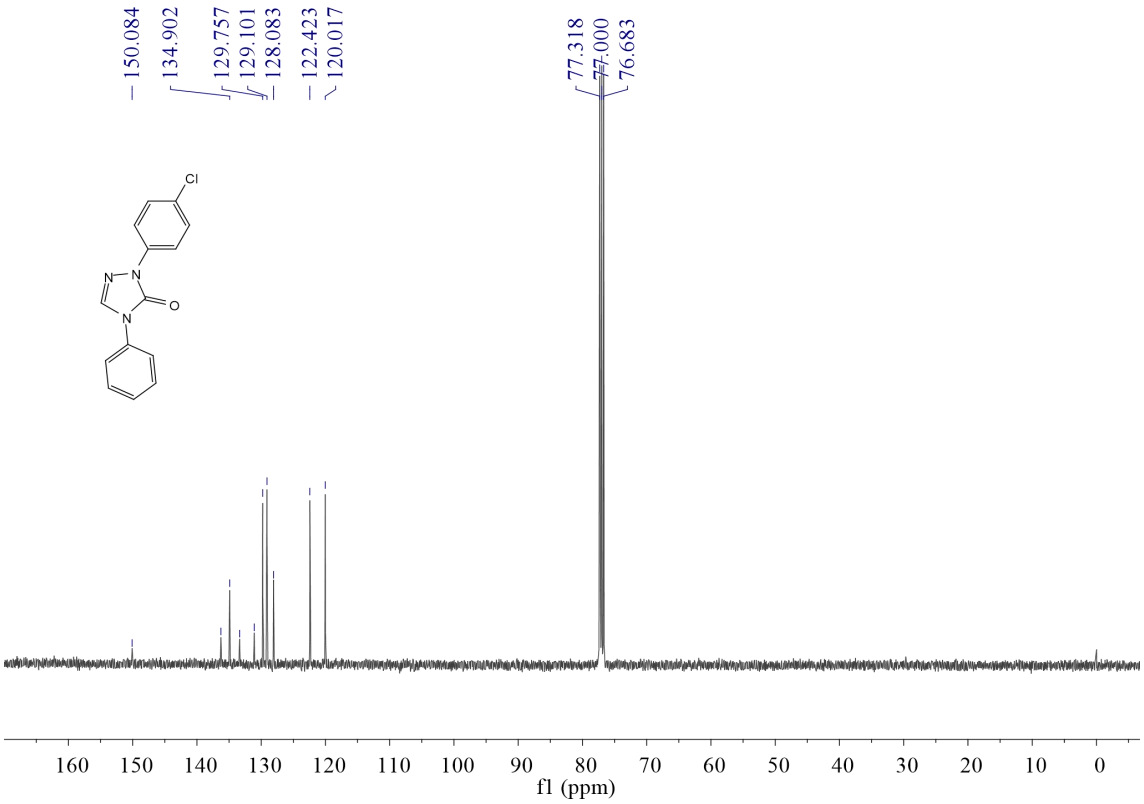
^{1}H NMR spectra of compound **4r**



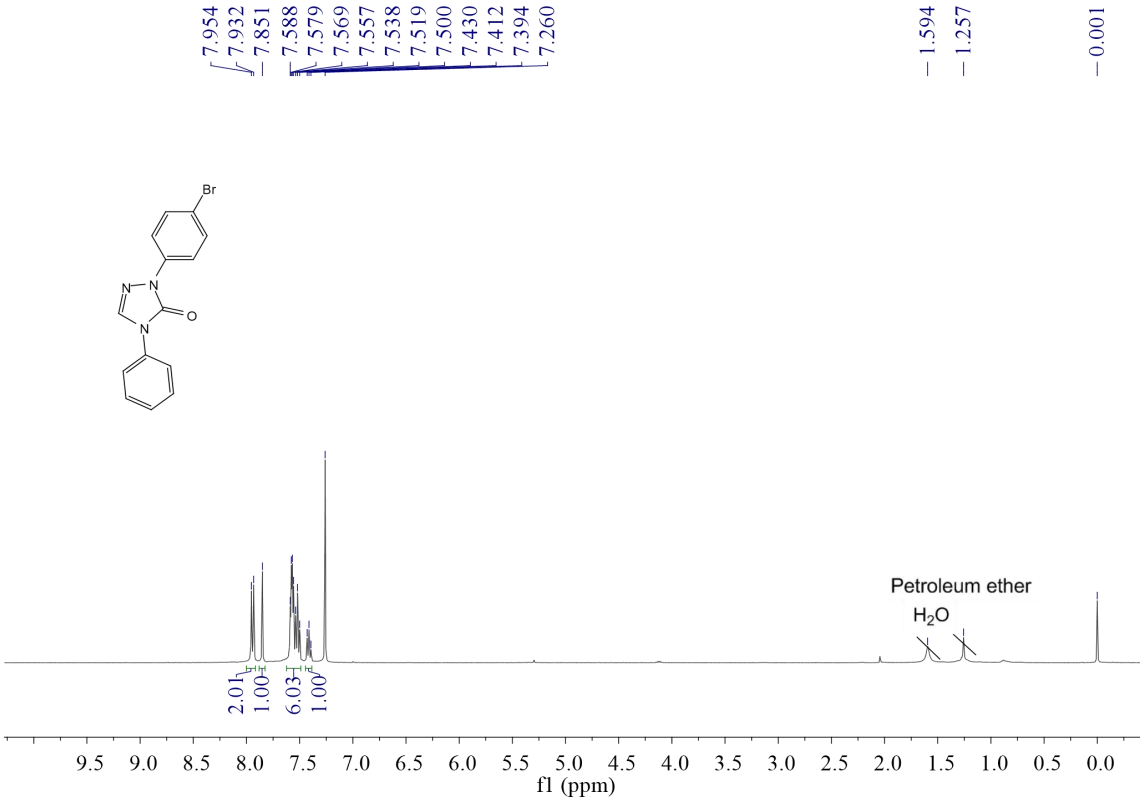
^{13}C NMR spectra of compound **4r**



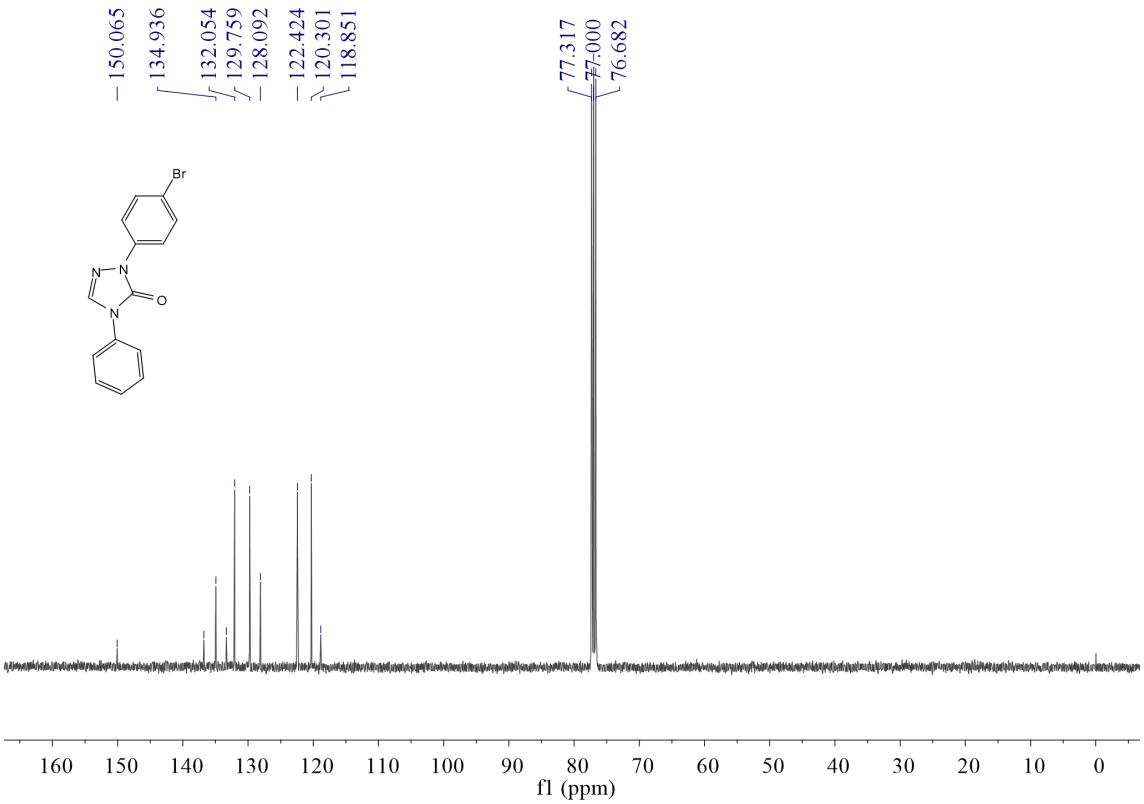
^{1}H NMR spectra of compound **4s**



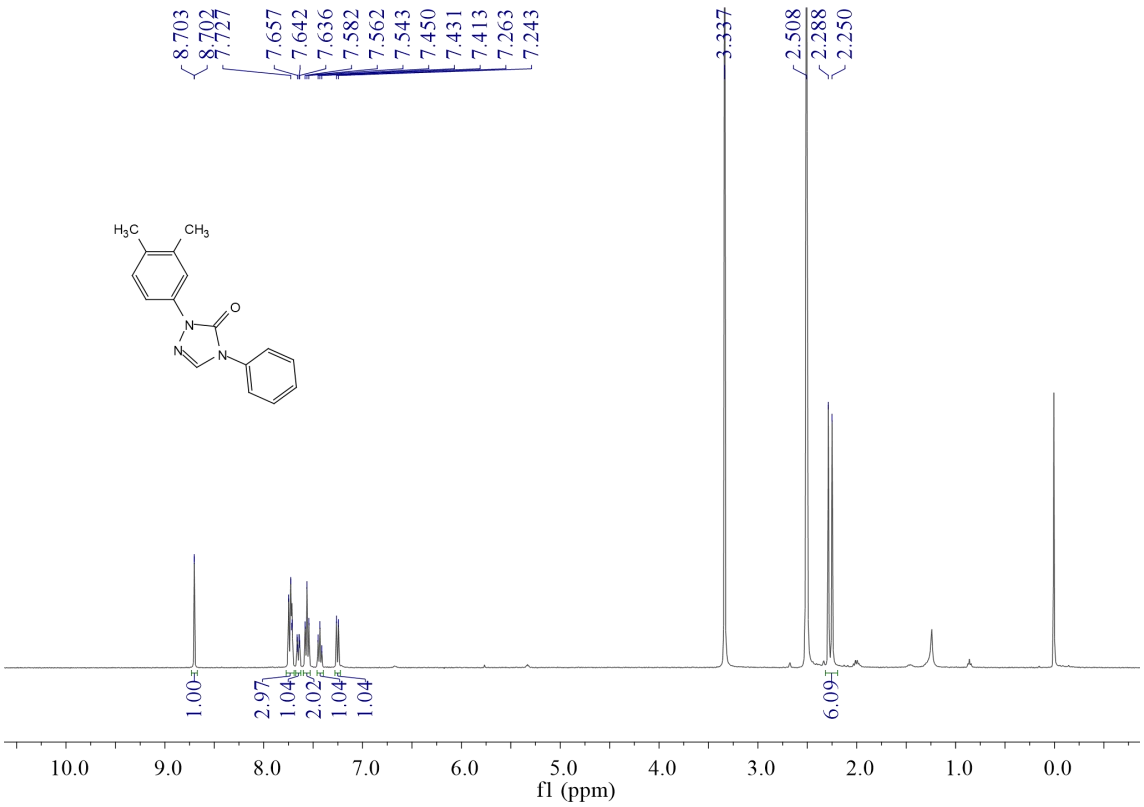
^{13}C NMR spectra of compound **4s**



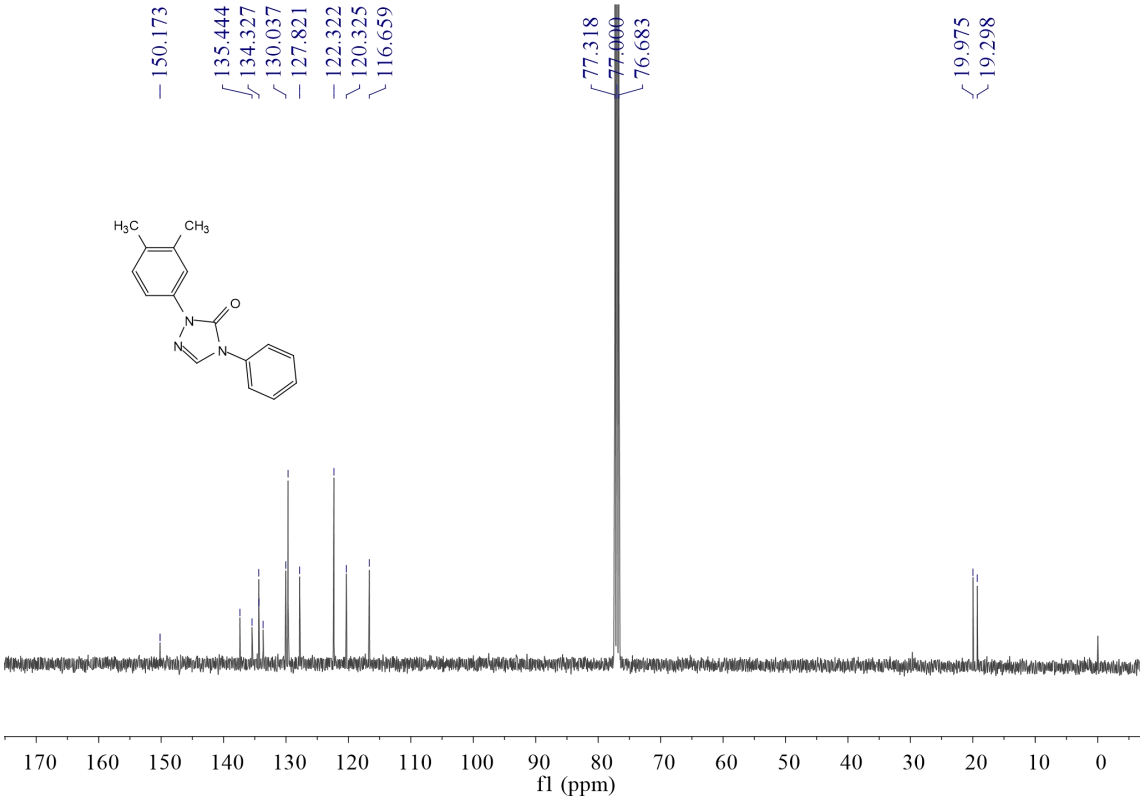
^{1}H NMR spectra of compound **4t**



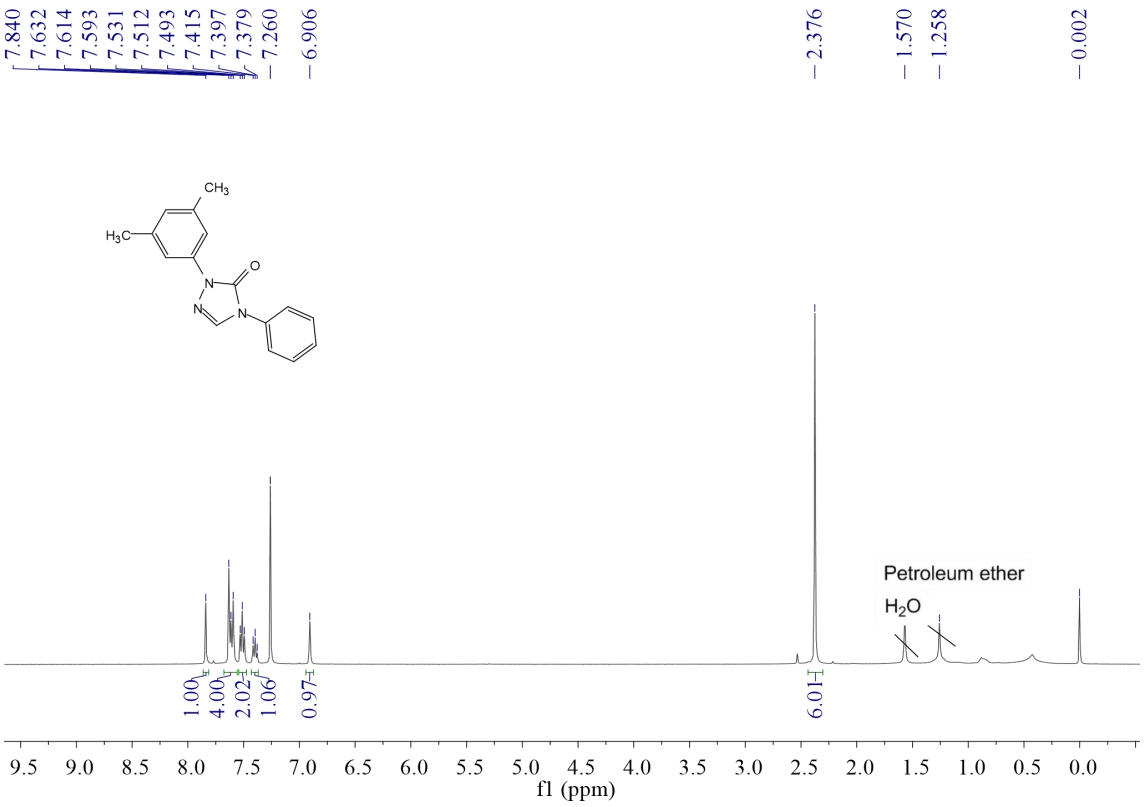
^{13}C NMR spectra of compound **4t**



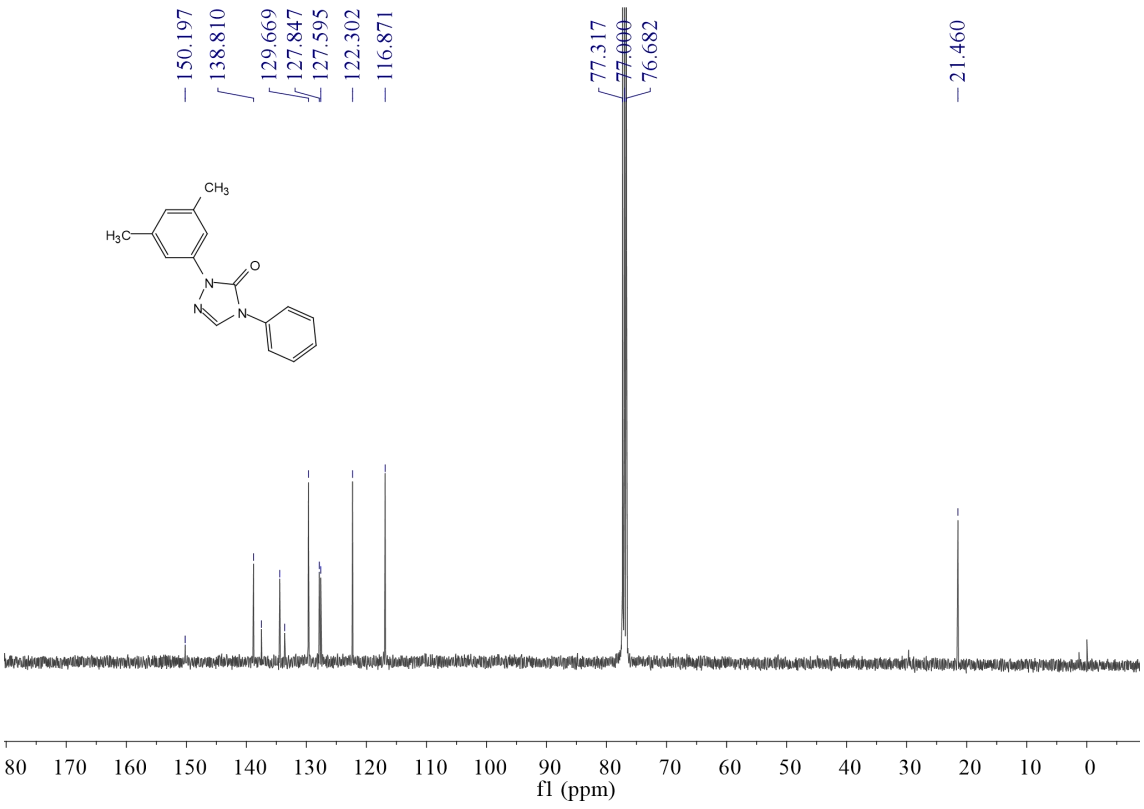
^{1}H NMR spectra of compound **4u**



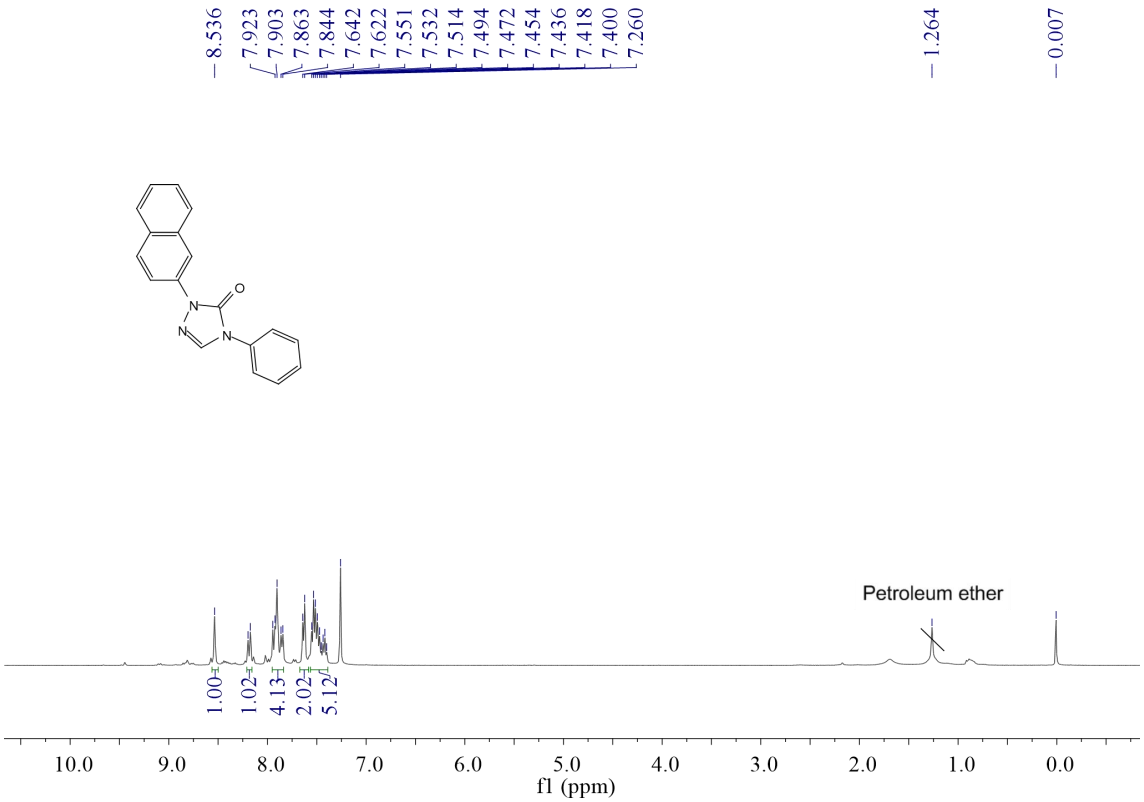
^{13}C NMR spectra of compound **4u**



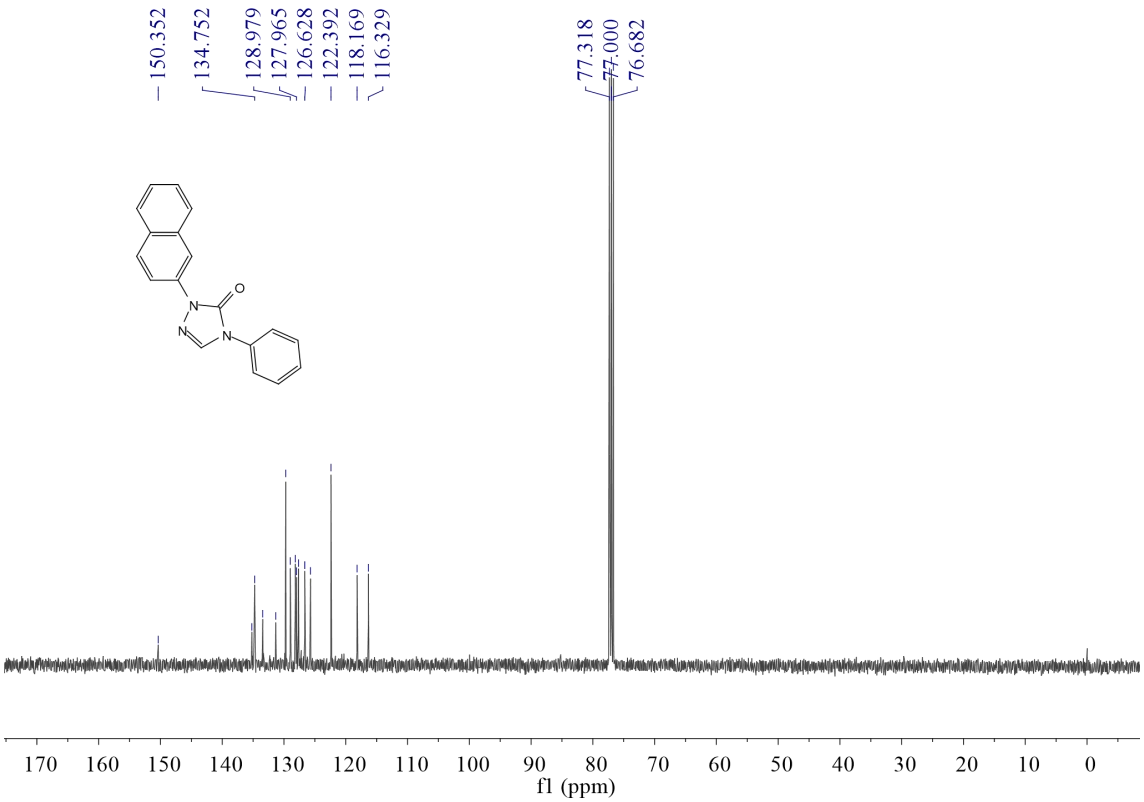
^{1}H NMR spectra of compound **4v**



^{13}C NMR spectra of compound **4v**



^{1}H NMR spectra of compound **4w**



^{13}C NMR spectra of compound **4w**

References

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