Supporting Information

for

Base-promoted anaerobic intramolecular cyclization synthesis of 4,5-disubstituted 1,2,3-thiadiazoles

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1. General Information

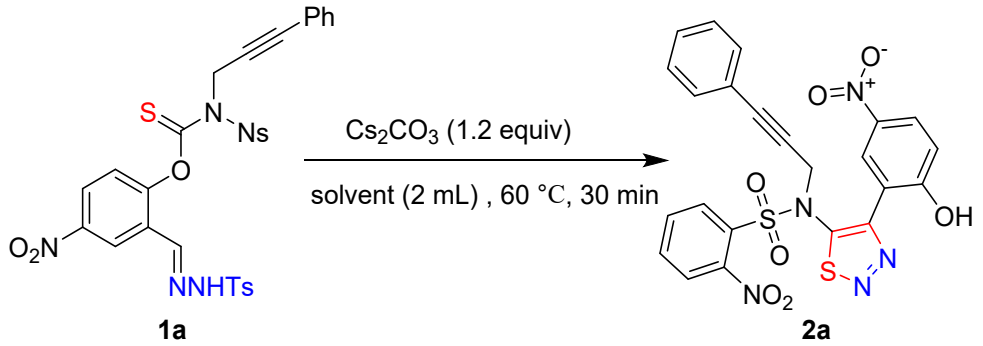
^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of compounds were recorded at rt in $CDCl_3$ on 400 MHz and 500MHz instrument with tetramethylsilane (TMS) as internal standard. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; m, multiplet), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). Chemical shifts were reported in ppm on the scale relative to $CDCl_3$ (δ = 7.26 for ^{1}H-NMR, δ = 77.00 for ^{13}C-NMR) or DMSO-d_6 (δ = 2.50 for ^{1}H-NMR, δ = 39.60 for ^{13}C-NMR) as an internal reference. High resolution mass spectra (HRMS) were recorded on a LC-TOF spectrometer (Micromass). ESI-HRMS data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source and controlled by Xcalibur software. Oil bath was used as heating source.

Aryl iodides, aryl bromides and propargylamine were obtained from the commercial sources or synthesized following literature procedures[1]. THF was dried by Na prior to use. Unless otherwise stated, all experiments were conducted in a 25 mL schlenk tube under N_2 atmosphere. Other chemicals were purchased from TCI, Energy, Adamas, Meryer, Acros, and Alfa Aesar, and used as received. Reactions were monitored by TLC or GC-MS analysis. Flash column chromatography was performed over silica gel (200-300 mesh).

2. Experimental Section

2.1 Optimization of experiment conditions

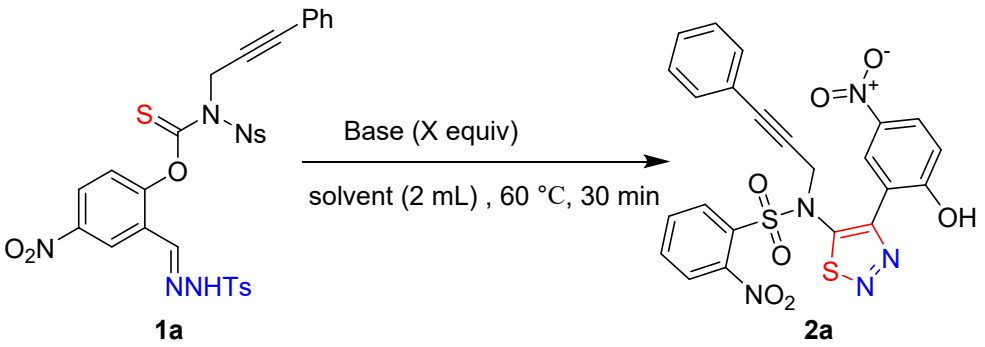
Table S1. Optimization of the reaction conditions[a]



Entry	T(°C)	Solvent	Yield (%)[b]
1	60	PhCl	ND
2	60	DCM	-
3	60	1,4-dioxane	-
4	60	DMF	-
5	60	toluene	-
6	60	acetone	trace
7	60	EtOH	-
8	60	DCE	trace
9	60	MeCN	-
10	60	THF	52%

[a] Reaction conditions: 1a (0.2 mmol), base (1.2 mmol), and solvent (2 mL) at 60 °C under air for 0.5 h under air, a 25 mL schlenk tube. [b] Isolated yield.

Table S2. Optimization of the reaction conditions[a]



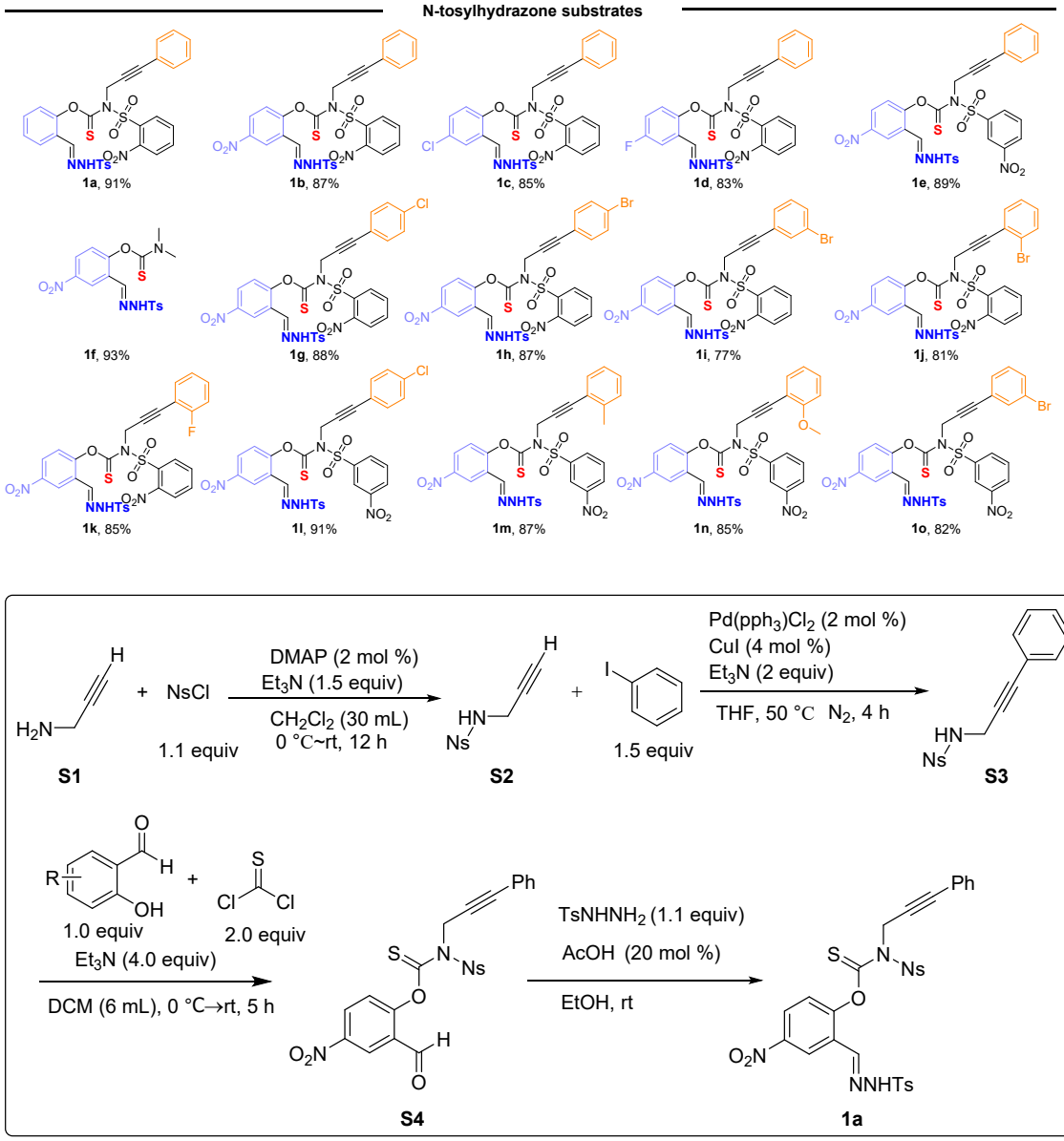
1a **2a**

Entry	Base	T(°C)	Solvent	Yield (%)[b]
1	NaO^tBu (1.2 equiv)	60	THF	trace
2	NaOH (1.2 equiv)	60	THF	NR
3	Na_2CO_3 (1.2 equiv)	60	THF	34
4	$NaHCO_3$ (1.2 equiv)	60	THF	27
5	Cs_2CO_3 (1.2 equiv)	60	THF	52
6	CsF (1.2 equiv)	60	THF	30
7	DBU (1.2 equiv)	60	THF	26
8	Et_3N (1.2 equiv)	60	THF	19
9	DABCO (1.2 equiv)	60	THF	47
10	CH_3ONa (1.2 equiv)	60	THF	15
11	LiO^tBu (1.2 equiv)	60	THF	44
12	Cs_2CO_3 (2.5 equiv)	80	THF	71
13	Cs_2CO_3 (5.0 equiv)	100	THF	67
14	Cs_2CO_3 (1.2 equiv)	80	THF	60
15	Cs_2CO_3 (1.2 equiv)	100	THF	62
16[c]	Cs_2CO_3 (1.2 equiv)	60	THF	45
17[d]	Cs_2CO_3 (1.2 equiv)	60	THF	90

[a] Reaction conditions: 1a (0.2 mmol), base (1.2 mmol), and solvent (2 mL) at 60 °C under air for 0.5 h, a 25 mL schlenk tube. [b] Isolated yield. [c] Under a N_2 atmosphere. [d] Under a N_2 atmosphere, THF (dry).

2.2 Materials and Methods

The *N*-tosylhydrazones were synthesized as detailed below.



Scheme S1. Synthetic route to *N*-tosylhydrazones 1

To a solution of the corresponding prop-2-yn-1-amine **S1** (100 mmol, 1.0 equiv) in CH_2Cl_2 (100 ml) at ice bath was added triethylamine (150 mmol, 1.5 equiv), and DMAP (2 mmol, 2 mol%) successively. After 10 min, added NsCl (110 mmol, 1.1 equiv) to the reaction mixture. The mixture was stirred for 0.5 h at ice bath, then remove the ice bath and stirred at room temperature overnight. After the reaction was completed (monitoring by TLC), brine was added (30 mL) and the mixture was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried with $MgSO_4$, filtered, and evaporated in vacuo. A portion of the crude product **S2** was directly subjected for

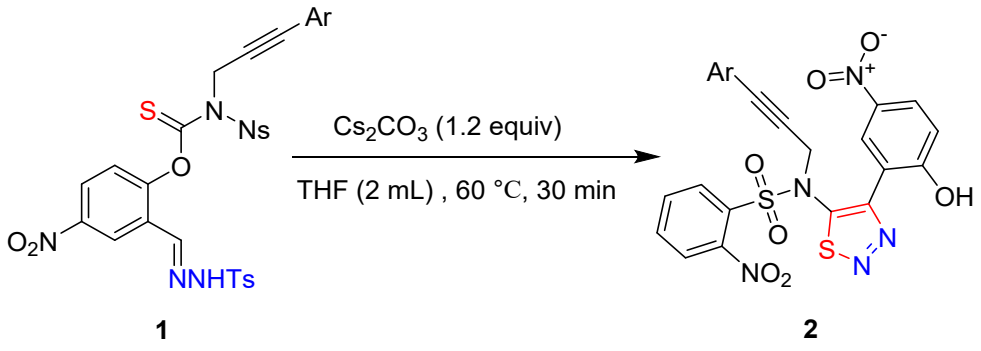
the next step. The starting materials were synthetized according to methods reported by previous literatures[1].

To a mixture of the above crude product (100 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (2 mmol, 2 mol%), and copper(I) iodide (4 mmol, 4 mol%), a solution of triethylamine (200 mmol, 2.0 equiv) was added followed by 50 ml THF under a N_2 atmosphere. The reaction mixture was stirred at room temperature for 3 h under argon atmosphere. The reaction mixture was then concentrated, and purified by column chromatography on silica gel (petroleum ether/EtOAc: 6/1, v/v) to afford the corresponding silyl alkynes **S3** (80 – 95% yields). The starting materials were synthetized according to methods reported by previous literatures[2].

To a solution of the corresponding propargylamine (80 mmol, 1.0 equiv) in DCM (100 ml) at ice bath was added thiophosgene (160 mmol, 2.0 equiv). After 5 min, added triethylamine (320 mmol, 4.0 equiv) to the reaction mixture. Then added salicylaldehyde (80 mmol, 1.0 equiv) 5 min later and stirred at the ice bath for 4 h. Once the reaction was completed (monitoring by TLC), water was added (30 mL) and the mixture was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic phases were washed with 5% HCl (20 mL), 1 M NaOH (20 mL), and brine (20 mL), dried over Na_2SO_4, and evaporated in vacuo. The residue was purified by column chromatography (petroleum ether/EtOAc: 6/1-4/1, v/v), giving the expected product **S4** (45 – 64% yields). This experiment was synthetized according to methods reported by previous literatures[3].

To a solution of aryl benzaldehyde (10 mmol, 1.0 equiv) and *p*-toluene sulfonylhydrazide (11 mmol, 1.1 equiv) in methanol, a catalytic amount of acetic acid was added. The mixture was stirred at 60 °C for 3h and the reaction was monitored by TLC. After the completion of the reaction, methanol was evaporated under reduced pressure, then added water and extracted with CH_2Cl_2 (3 × 30 mL). The organic phases were combined and dried over anhydrous Na_2SO_4. The solvent was removed at reduced pressure and afforded as a white solid **1a** in 80 – 95% yields. This experiment was synthetized according to methods reported by previous literatures[4].

2.3 General procedure for synthesis of target product 2

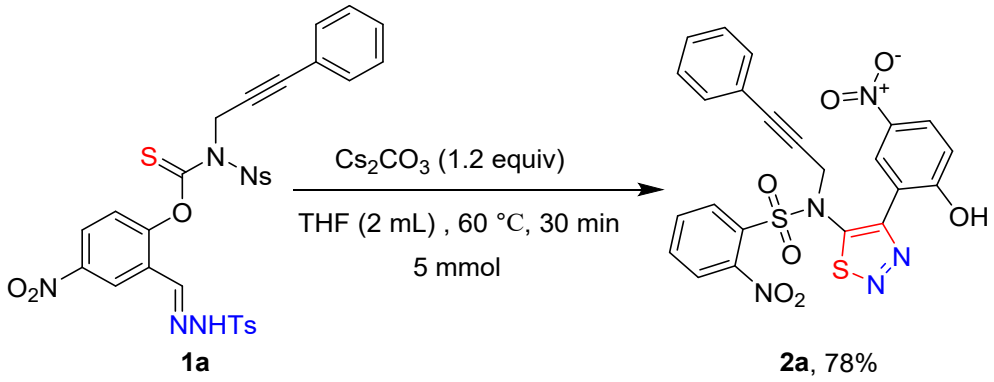


1 **2**

Scheme S2. General procedure for synthesis of target product 2.

Unless otherwise noted, an oven-dried 25 mL resealable Schlenk tube equipped with a magnetic stir bar was charged with *N*-tosylhydrazones **1** (0.2 mmol, 1.0 equiv), Cs_2CO_3 (2.4 mmol, 1.2 equiv). After that, argon was pumped three times, and anhydrous THF (2 mL) was added under N_2 atmosphere. Then the reaction mixture was stirred at 60 ℃ for 0.5 h. Upon completion (monitored by TLC), the filtrate was evaporated and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc: 4/1-2/1, v/v) to afford the desired product **2**.

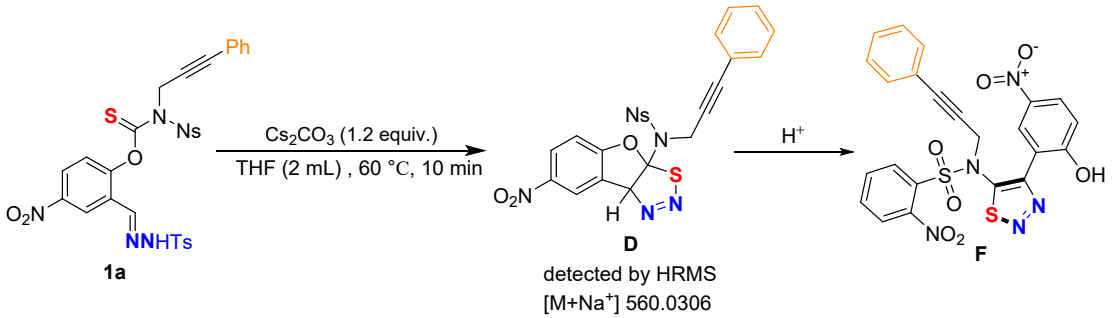
2.4 Gram scale synthesis



Scheme S3. Gram-scale synthesis of products 2a.

In an oven-dried 100 mL resealable Schlenk tube equipped with a magnetic stir bar was charged with *N*-tosylhydrazones **1a** (5.0 mmol, 1.0 equiv), Cs_2CO_3 (6.0 mmol, 1.2 equiv). After that, argon was pumped three times, and anhydrous THF (2 mL) was added under N_2 atmosphere. Then the reaction mixture was stirred at 60 ℃ for 0.5 h. Upon completion (monitored by TLC), the filtrate was evaporated and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc: 6/1-4/1, v/v) to afford the desired product **2a** in 78% yield.

2.5 Mechanistic studies

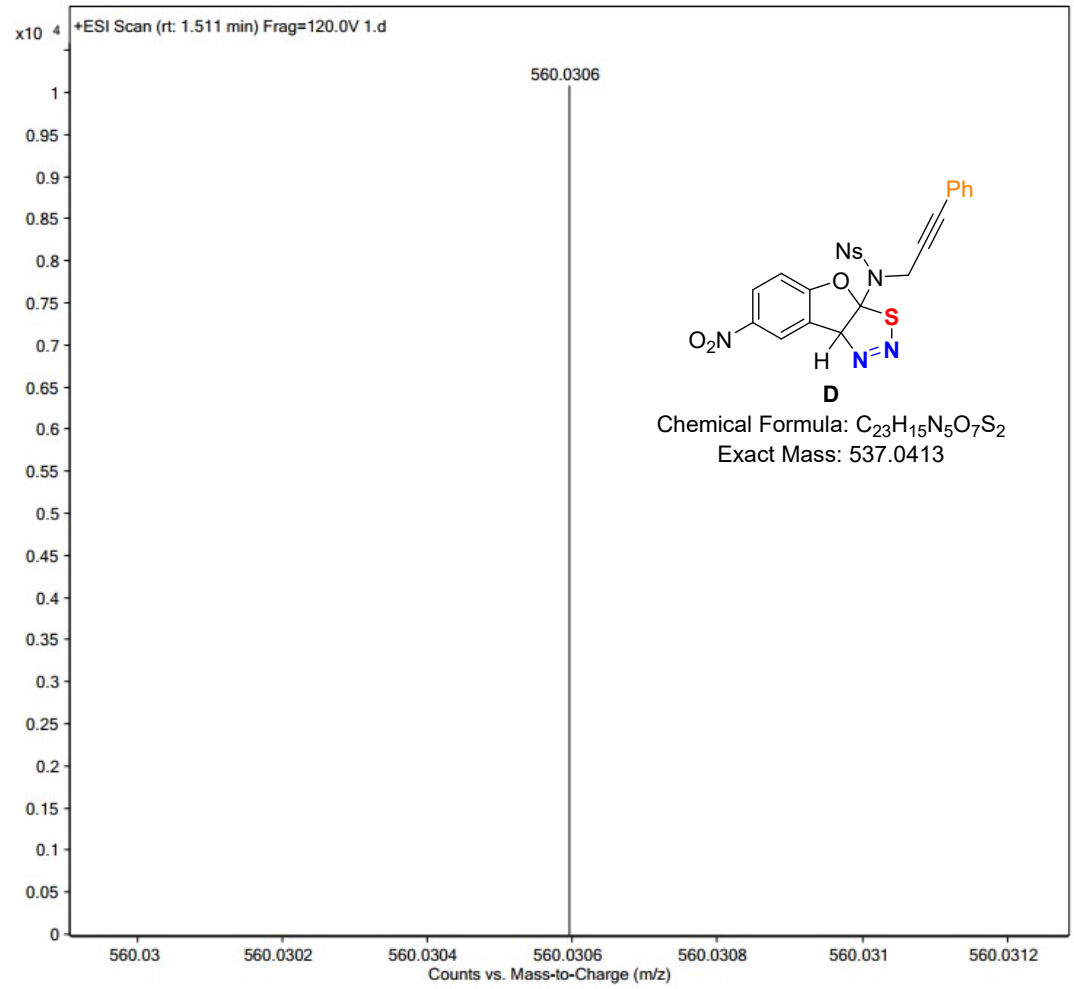


In an oven-dried 25 mL resealable Schlenk tube equipped with a magnetic stir bar was charged with *N*-tosylhydrazones **1a** (0.2 mmol, 1.0 equiv), Cs_2CO_3 (2.4 mmol, 1.2 equiv). After that, argon was pumped three times, and anhydrous THF (2 mL) was added under N_2 atmosphere. Then the reaction mixture was stirred at 60 ℃ for 10 mins.

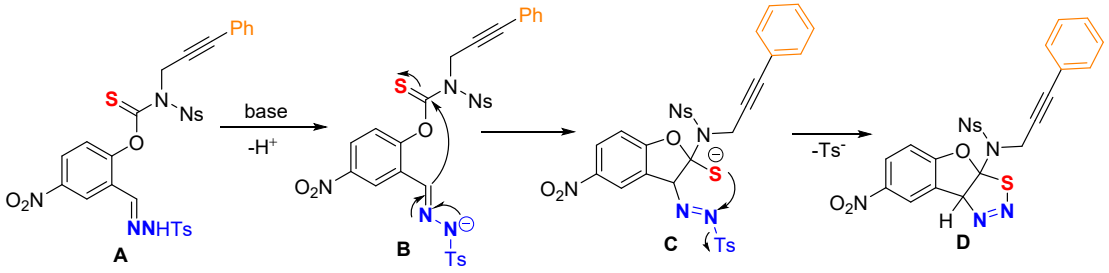
And the possible intermediate D was detected by HRMS.

Via HRMS

Species **D** was detected by HRMS:



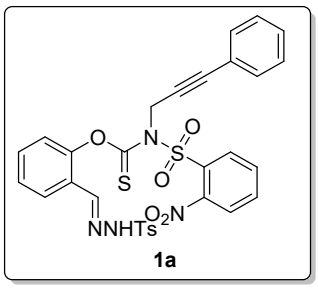
Proposed mechanism for the formation of **D:**



3. Characterization data for substrate 1 and products

Characterization data for substrate 1

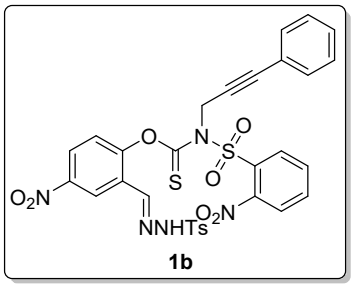
(E)-O-(2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1a)



1a

The title compound **1a** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (118 mg, 91% yield), m.p. = 153–155 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.41 (d, *J* = 10.0 Hz, 1H), 8.22 (d, *J* = 10.0 Hz, 1H), 7.90 (d, *J* = 5.0 Hz, 1H), 7.86 – 7.79 (m, 4H), 7.66 (t, *J* = 5.0 Hz, 1H), 7.51 (d, *J* = 5.0 Hz, 3H), 7.37 – 7.26 (m, 8H), 6.81 (d, *J* = 10.0 Hz, 1H), 5.31 (s, 2H), 2.42 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 185.1, 150.8, 147.8, 144.3, 140.5, 135.4, 135.2, 132.8, 132.5, 132.0, 131.2, 129.9, 129.7, 128.8, 128.4, 128.2, 127.8, 127.4, 127.0, 126.2, 125.2, 122.6, 122.1, 84.8, 82.7, 42.4, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{25}N_4O_7S_3^+$, 649.0880, found: 649.0882;

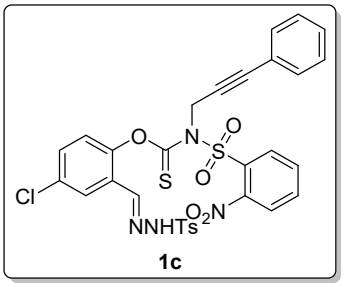
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1b)



1b

The title compound **1b** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (120 mg, 87% yield), m.p. = 157–158 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.64 (s, 1H), 8.44 (s, 1H), 8.24 (d, *J* = 5.0 Hz, 1H), 8.17 (d, *J* = 5.0 Hz, 1H), 7.92 (d, *J* = 5.0 Hz, 1H), 7.87 (d, *J* = 10.0 Hz, 3H), 7.22 (t, *J* = 10.0 Hz, 1H), 7.57 (s, 1H), 7.49 (d, *J* = 10.0 Hz, 1H), 7.40 – 7.28 (m, 5H), 7.03 (d, *J* = 10.0 Hz, 1H), 5.30 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.7, 154.1, 147.8, 146.5, 144.8, 137.9, 135.7, 134.7, 132.7, 132.5, 131.9, 129.8, 129.1, 128.5, 128.1, 128.0, 125.5, 125.3, 124.3, 122.7, 121.8, 85.2, 82.2, 42.5, 21.6.; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{24}N_5O_9S_3^+$, 694.0731, found: 694.0735;

(E)-O-(4-chloro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1c)

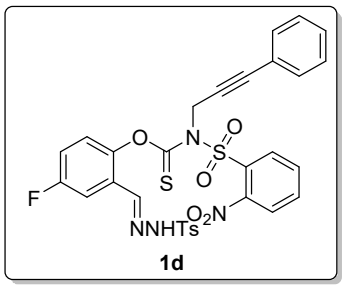


1c

The title compound **1c** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (116 mg, 85% yield), m.p. = 159–162 ºC; **^{1}H NMR (500 MHz,**

Chloroform-*d*) δ 8.69 (s, 1H), 8.20 (d, *J* = 5.0 Hz, 1H), 7.89 (d, *J* = 5.0 Hz, 1H), 7.83 – 7.78 (m, 4H), 7.67 (t, *J* = 10.0 Hz, 1H), 7.47 (d, *J* = 10.0 Hz, 3H), 7.35 – 7.30 (m, 6H), 7.29 (dd, *J* = 10.0, 5.0 Hz, 1H), 6.77 (d, *J* = 5.0 Hz, 1H), 5.29 (s, 2H), 2.41 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.8, 171.5, 149.1, 147.8, 144.5, 139.1, 135.6, 135.0, 133.1, 132.6, 132.6, 131.9, 131.0, 129.8, 128.9, 128.4, 127.9, 127.8, 126.6, 125.4, 124.1, 122.0, 84.9, 82.7, 42.5, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}ClN_4O_7S_3^+$, 683.0490, found: 683.0491;

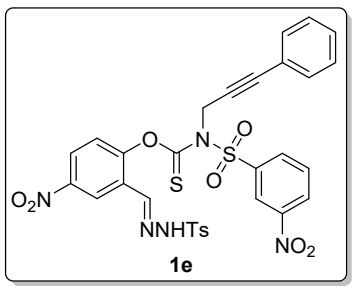
(E)-O-(4-fluoro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1d)



1d

The title compound **1d** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (110 mg, 83% yield), m.p. = 154–155 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.90 (d, *J* = 10.0 Hz, 1H), 8.21 (d, *J* = 10.0 Hz, 1H), 7.89 (d, *J* = 10.0 Hz, 1H), 7.85 – 7.77 (m, 3H), 7.66 (t, *J* = 10.0 Hz, 1H), 7.51 (d, *J* = 10.0 Hz, 1H), 7.48 – 7.44 (m, 3H), 7.35 – 7.28 (m, 6H), 7.03 (t, *J* = 10.0 Hz, 1H), 6.79 (dd, *J* = 10.0, 5.0 Hz, 1H), 5.30 (s, 2H), 2.40 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 185.2, 171.6, 161.7, 159.7, 147.7, 146.7, 146.7, 144.5, 139.4, 135.7, 135.1, 132.6, 132.5, 131.9, 129.8, 128.9, 128.4, 127.8, 125.4, 124.4, 124.4, 122.0, 118.3, 118.1, 113.1, 112.9, 84.9, 82.8, 42.5, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{24}FN_4O_7S_3^+$, 667.0786, found: 667.0789;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((3-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1e)

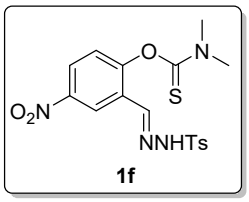


1e

The title compound **1e** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (123 mg, 89% yield), m.p. = 159–162 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.07 (s, 1H), 8.93 (d, *J* = 5.0 Hz, 1H), 8.64 (s, 1H), 8.52 (s, 1H), 8.52 (dd, *J* = 10.0, 5.0 Hz, 1H), 8.42 (dd, *J* = 10.0, 5.0 Hz, 1H), 8.17 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.86 (d, *J* = 10.0 Hz, 3H), 7.76 (t, *J* = 10.0 Hz, 1H), 7.67 (s, 1H), 7.49 (d, *J* = 10.0 Hz, 2H), 7.43 – 7.33 (m, 6H), 6.92 (d, *J* = 10.0 Hz, 1H), 5.48 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.1, 154.0, 148.2, 146.5, 144.8, 140.5, 138.2, 134.8, 134.0, 131.9, 130.6, 129.8, 129.3, 128.9, 128.5, 128.2, 127.9, 125.4, 124.3, 123.6, 123.1, 121.4, 85.8, 81.9, 42.2, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{24}N_5O_9S_3^+$, 694.0731, found: 694.0730;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl)

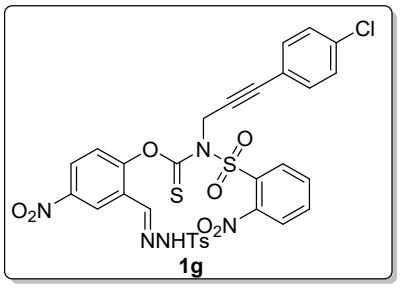
dimethylcarbamothioate (1f)



1f

The title compound **1f** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (78 mg, 93% yield), m.p. = 133–135 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.86 (s, 1H), 8.42 (d, *J* = 5.0 Hz, 1H), 8.26 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.93 (s, 1H), 7.77 (d, *J* = 5.0 Hz, 2H), 7.43 (t, *J* = 5.0 Hz, 3H), 3.36 (s, 6H), 2.37 (s, 3H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 184.9, 156.3, 145.8, 144.2, 140.1, 136.4, 130.3, 128.6, 127.5, 126.7, 125.5, 121.3, 43.5, 21.4; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{17}H_{19}N_4O_5S_2^+$, 423.0791, found: 423.0790;

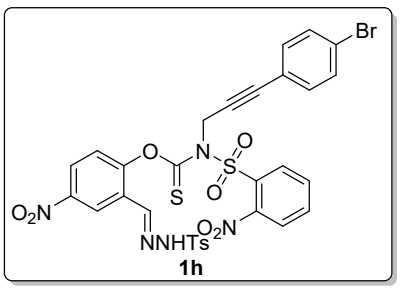
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(4-chlorophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1g)



1g

The title compound **1g** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (128 mg, 88% yield), m.p. = 133–139 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.08 (s, 1H), 8.63 (d, *J* = 10.0 Hz, 1H), 8.24 – 8.16 (m, 2H), 7.95 (d, *J* = 10.0 Hz, 1H), 7.86 (d, *J* = 10.0 Hz, 3H), 7.72 (t, *J* = 10.0 Hz, 1H), 7.61 (s, 1H), 7.53 (s, 1H), 7.51 (d, *J* = 5.0 Hz, 1H), 7.42 (d, *J* = 10.0 Hz, 1H), 7.37 (d, *J* = 10.0 Hz, 2H), 7.23 (t, *J* = 5.0 Hz, 1H), 7.06 (d, *J* = 10.0 Hz, 1H), 5.29 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.7, 154.0, 147.8, 146.5, 144.8, 137.9, 135.8, 134.7, 134.6, 132.7, 132.6, 132.5, 132.1, 130.6, 129.9, 128.1, 127.9, 125.5, 124.3, 123.9, 122.7, 122.1, 83.6, 83.5, 42.5, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}ClN_5O_9S_3^+$, 728.0341, found: 728.0343;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(4-bromophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1h)

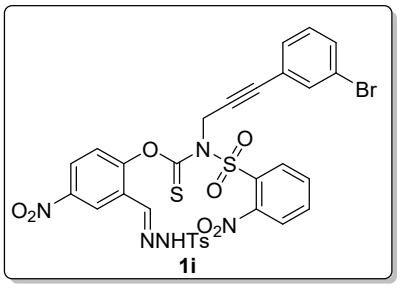


1h

The title compound **1h** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (134 mg, 87% yield), m.p. = 135–138 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.60 (s, 1H), 8.21 (d, *J* =

5.0 Hz, 1H), 8.15 (d, *J* = 5.0 Hz, 1H), 7.93 (d, *J* = 5.0 Hz, 1H), 7.86 – 7.82 (m, 3H), 7.71 – 7.67 (m, 1H), 7.56 (s, 1H), 7.44 (d, *J* = 5.0 Hz, 2H), 7.34 – 7.28 (m, 4H), 7.03 (d, *J* = 5.0 Hz, 1H), 5.27 (s, 2H), 2.41 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.8, 171.4, 154.1, 147.7, 146.4, 144.8, 138.1, 135.9, 134.8, 133.4, 132.7, 132.6, 132.4, 131.6, 129.8, 128.2, 127.9, 125.5, 125.4, 124.3, 123.2, 122.6, 120.9, 84.1, 83.5, 42.6, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}BrN_5O_9S_3^+$, 771.9836, found: 771.9839;

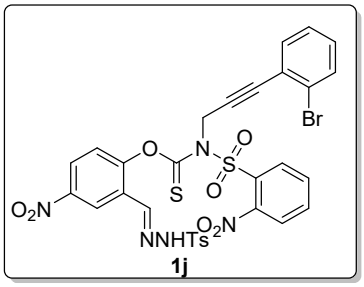
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(3-bromophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1i)



1i

The title compound **1i** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (119 mg, 77% yield), m.p. = 143–144 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.08 (s, 1H), 8.66 (s, 1H), 8.64 (d, *J* = 5.0 Hz, 1H), 8.21 (d, *J* = 10.0 Hz, 1H), 8.18 (d, *J* = 10.0 Hz, 1H), 7.94 (t, *J* = 10.0 Hz, 1H), 7.86 (d, *J* = 10.0 Hz, 3H), 7.71 (t, *J* = 5.0 Hz, 1H), 7.54 (s, 1H), 7.42 – 7.28 (m, 7H), 7.05 (d, *J* = 10.0 Hz, 1H), 5.28 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.7, 162.6, 154.1, 147.8, 146.5, 144.8, 144.8, 143.7, 138.4, 138.0, 135.8, 135.1, 134.7, 133.2, 132.6, 132.6, 132.5, 129.9, 128.7, 128.1, 128.0, 127.9, 125.5, 124.3, 123.7, 122.7, 121.4, 120.4, 84.0, 83.2, 42.6, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}BrN_5O_9S_3^+$, 771.9836, found: 771.9838;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(2-bromophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1j)

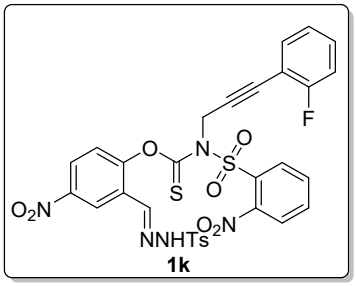


1j

The title compound **1j** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (125 mg, 81% yield), m.p. = 141–142 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.08 (s, 1H), 8.66 (s, 1H), 8.64 (d, *J* = 5.0 Hz, 1H), 8.21 (d, *J* = 10.0 Hz, 1H), 8.18 (d, *J* = 10.0 Hz, 1H), 7.94 (t, *J* = 10.0 Hz, 1H), 7.86 (d, *J* = 10.0 Hz, 3H), 7.71 (t, *J* = 5.0 Hz, 1H), 7.54 (s, 1H), 7.42 – 7.28 (m, 7H), 7.05 (d, *J* = 10.0 Hz, 1H), 5.28 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.7, 162.6, 154.1, 147.8, 146.5, 144.8, 144.8, 143.7, 138.4, 138.0, 135.8, 135.1, 134.7, 133.2, 132.6, 132.6, 132.5, 129.9,

128.7, 128.1, 128.0, 127.9, 125.5, 124.3, 123.7, 122.7, 121.4, 120.4, 84.0, 83.2, 42.6, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}BrN_5O_9S_3^+$, 771.9836, found: 771.9838;

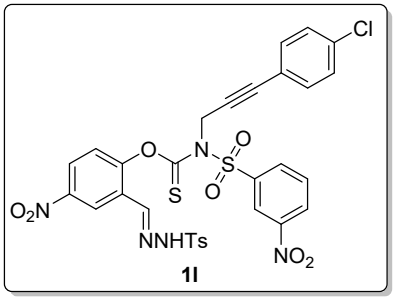
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(2-fluorophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1k)



1k

The title compound **1k** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (121 mg, 85% yield), m.p. = 163–165 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.06 (s, 1H), 8.69 (d, *J* = 5.0 Hz, 1H), 8.53 (s, 1H), 8.27 (d, *J* = 5.0 Hz, 1H), 8.17 (d, *J* = 10.0 Hz, 1H), 7.91 – 7.85 (m, 4H), 7.76 (t, *J* = 10.0 Hz, 1H), 7.58 (s, 1H), 7.51 (t, *J* = 10.0 Hz, 1H), 7.41 – 7.35 (m, 3H), 7.18 (d, *J* = 10.0 Hz, 1H), 6.96 (d, *J* = 10.0 Hz, 1H), 5.35 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 183.6, 162.0, 154.1, 147.9, 146.5, 144.7, 137.8, 135.7, 134.9, 133.6, 132.6, 132.5, 132.4, 130.8, 129.8, 128.2, 127.9, 125.4, 125.3, 124.2, 124.2, 122.6, 115.7, 115.5, 87.5, 78.8, 42.4, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}FN_5O_9S_3^+$, 712.0636, found: 712.0634;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(4-chlorophenyl)prop-2-yn-1-yl)((3-nitrophenyl)sulfonyl)carbamothioate (1l)

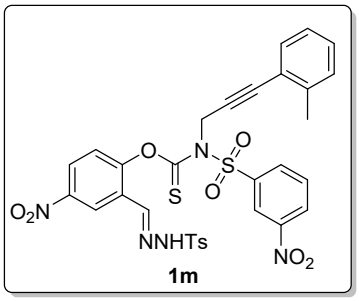


1l

The title compound **1l** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (132 mg, 91% yield), m.p. = 128–129 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.06 (s, 1H), 8.92 (d, *J* = 5.0 Hz, 1H), 8.63 (d, *J* = 5.0 Hz, 2H), 8.53 (d, *J* = 10.0 Hz, 1H), 8.17 (d, *J* = 10.0 Hz, 2H), 8.02 (s, 1H), 7.87 (t, *J* = 10.0 Hz, 3H), 7.77 (t, *J* = 10.0 Hz, 1H), 7.44 (d, *J* = 10.0 Hz, 2H), 7.39 – 7.33 (m, 6H), 7.04 (d, *J* = 10.0 Hz, 1H), 6.92 (d, *J* = 10.0 Hz, 1H), 5.47 (s, 2H), 2.44 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.2, 163.0, 154.0, 148.5, 148.2, 146.5, 145.2, 144.8, 140.5, 140.4, 138.5, 135.3, 134.7, 134.1, 133.2, 130.6, 130.2, 130.0, 129.9, 128.9, 128.8, 128.6, 128.2, 127.9, 127.8, 127.1, 126.8, 125.5, 124.3, 123.5, 123.1, 119.9, 117.8, 116.9, 84.6, 82.9, 42.2, 21.6, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}ClN_5O_9S_3^+$, 728.0341, found: 728.0343;

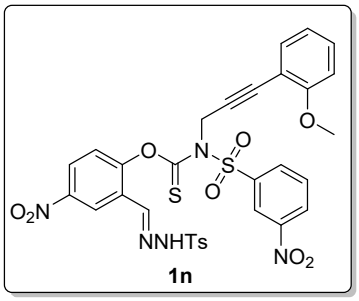
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((3-

nitrophenyl)sulfonyl)(3-(o-tolyl)prop-2-yn-1-yl)carbamothioate (1m)



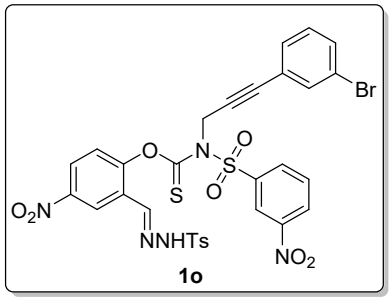
The title compound **1m** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (123 mg, 87% yield), m.p. = 155–157 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.20 (s, 1H), 8.90 (s, 1H), 8.65 (d, *J* = 15.0 Hz, 2H), 8.50 (d, *J* = 5.0 Hz, 1H), 8.40 (d *J* = 5.0 Hz, 1H), 8.17 (q, *J* = 5.0 Hz, 1H), 7.86 (d, *J* = 5.0 Hz, 2H), 7.75 (t, *J* = 10.0 Hz, 1H), 7.67 (s, 1H), 7.45 (d, *J* = 10.0 Hz, 1H), 7.37 (d, *J* = 10.0 Hz, 1H), 7.32 (t, *J* = 10.0 Hz, 1H), 7.24 (d, *J* = 5.0 Hz, 1H), 7.21 (t, *J* = 10.0 Hz, 1H), 6.88 (d, *J* = 10.0 Hz, 1H), 5.50 (s, 2H), 2.44 (s, 3H), 2.40 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.1, 154.0, 148.2, 146.5, 144.8, 140.8, 140.6, 138.2, 134.8, 133.9, 132.2, 130.6, 129.9, 129.7, 129.3, 128.9, 128.2, 127.9, 125.8, 125.4, 124.3, 123.6, 123.1, 121.2, 85.7, 84.7, 42.5, 21.6, 20.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{31}H_{26}N_5O_9S_3^+$, 708.0887, found: 708.0889;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(2-methoxyphenyl)prop-2-yn-1-yl)((3-nitrophenyl)sulfonyl)carbamothioate (1n)



The title compound **1n** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid (123 mg, 85% yield), m.p. = 165–169 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.11 (s, 1H), 8.92 (s, 1H), 8.76 (s, 1H), 8.67 (s, 1H), 8.48 (s, 2H), 8.17 (d, *J* = 10.0 Hz, 1H), 7.82 (d, *J* = 10.0 Hz, 2H), 7.73 (t, *J* = 5.0 Hz, 1H), 7.61 (s, 1H), 7.42 – 7.29 (m, 4H), 7.00 (d, *J* = 5.0 Hz, 3H), 5.50 (s, 2H), 3.83 (s, 3H), 2.43 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.1, 160.2, 154.0, 148.1, 146.5, 144.6, 140.3, 137.9, 134.9, 134.1, 133.8, 131.0, 130.4, 129.8, 128.8, 128.3, 127.9, 125.4, 124.3, 123.9, 122.8, 121.2, 112.0, 110.9, 86.1, 82.3, 56.4, 42.2, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{31}H_{26}N_5O_{10}S_3^+$, 724.0836, found: 724.0838;

(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(3-bromophenyl)prop-2-yn-1-yl)((3-nitrophenyl)sulfonyl)carbamothioate (1o)

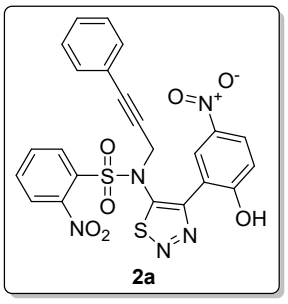


The title compound **1o** was purified by flash chromatography in PE/EA = 6/1-2/1 as yellow solid

(126 mg, 82% yield), m.p. = 157–158 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 11.12 (s, 1H), 8.90 (s, 1H), 8.60 (d, *J* = 5.0 Hz, 1H), 8.52 (d, *J* = 10.0 Hz, 1H), 8.39 (d, *J* = 10.0 Hz, 1H), 8.16 – 8.11 (m, 2H), 8.07 (d, *J* = 5.0 Hz, 1H), 8.02 (d, *J* = 5.0 Hz, 1H), 7.91 – 7.80 (m, 4H), 7.77 (t, *J* = 5.0 Hz, 1H), 7.70 (d, *J* = 5.0 Hz, 1H), 7.59 (d, *J* = 5.0 Hz, 1H), 7.50 (d, *J* = 5.0 Hz, 1H), 7.43 (q, *J* = 10.0 Hz, 1H), 7.38 – 7.32 (m, 4H), 7.24 (d, *J* = 5.0 Hz, 2H), 6.92 (d, *J* = 10.0 Hz, 1H), 5.46 (s, 2H), 2.42 (s, 3H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 184.2, 163.0, 153.9, 148.5, 148.2, 146.5, 145.1, 144.8, 140.4, 140.3, 138.6, 138.6, 134.8, 134.7, 134.6, 134.2, 134.1, 132.3, 130.7, 130.5, 130.1, 129.9, 129.9, 129.9, 129.7, 129.0, 128.2, 127.9, 127.8, 127.0, 126.8, 125.5, 124.4, 123.6, 123.5, 123.2, 122.2, 117.8, 117.0, 84.0, 83.4, 83.3, 42.2, 21.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{30}H_{23}BrN_5O_9S_3^+$, 771.9836, found: 771.9833;

Characterization of Products

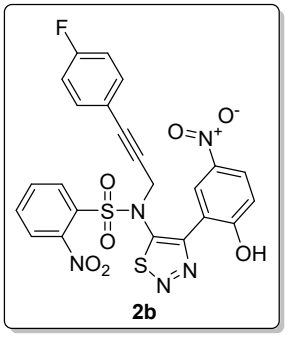
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitro-N-(3-phenylprop-2-yn-1-yl)benzenesulfonamide (2a)



2a

The title compound **2a** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (98 mg, 91% yield); **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 7.95 (d, *J* = 10.0 Hz, 1H), 7.72 (d, *J* = 10.0 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.63 (t, *J* = 5.0 Hz, 1H), 7.54 (t, *J* = 5.0 Hz, 1H), 7.28 – 7.25 (m, 5H), 6.88 (d, *J* = 10.0 Hz, 1H), 4.73 (s, 2H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 162.2, 154.7, 150.6, 147.4, 139.6, 136.5, 133.2, 131.9, 131.8, 129.9, 129.2, 129.1, 127.6, 127.4, 125.2, 121.3, 117.3, 116.8, 88.0, 83.4, 43.0; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{16}N_5O_7S_2^+$, 538.0486, found: 538.0487;

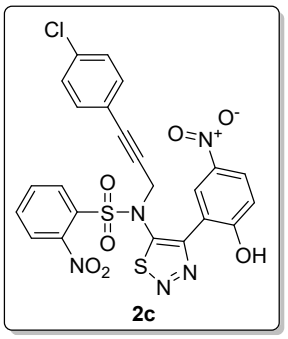
N-(3-(4-fluorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2b)



2b

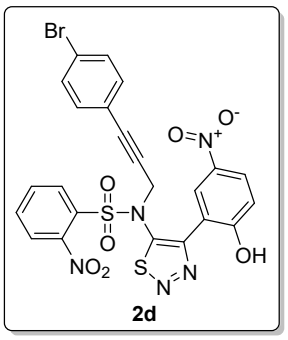
The title compound **2b** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (98 mg, 88% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.78 (s, 1H), 8.14 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.91 (d, *J* = 5.0 Hz, 1H), 7.88 – 7.84 (m, 2H), 7.81 (t, *J* = 5.0 Hz, 1H), 7.71 (t, *J* = 5.0 Hz, 1H), 7.34 (d, *J* = 5.0 Hz, 2H), 7.25 (d, *J* = 5.0 Hz, 2H), 7.06 (d, *J* = 10.0 Hz, 1H), 4.90 (s, 2H), 2.35 (s, 3H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 162.2, 154.7, 150.6, 147.4, 139.8, 139.6, 136.5, 133.2, 131.8, 129.8, 129.1, 127.6, 127.4, 125.2, 118.3, 117.3, 116.8, 88.2, 82.8, 43.0, 21.5; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}FN_5O_7S_2^+$, 556.0391, found: 556.0390;

N-(3-(4-chlorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2c)



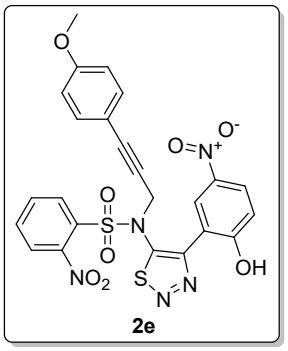
The title compound **2c** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (99 mg, 87% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.66 (s, 1H), 8.43 (dd, *J* = 8.4, 2.3 Hz, 1H), 8.34 (t, *J* = 2.1 Hz, 1H), 8.19 (d, *J* = 2.9 Hz, 1H), 8.17 (dd, *J* = 9.0, 2.9 Hz, 1H), 8.09 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.79 (t, *J* = 8.1 Hz, 1H), 7.50 – 7.44 (m, 2H), 7.35 – 7.30 (m, 2H), 7.04 (d, *J* = 9.0 Hz, 1H), 4.99 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 154.7, 151.3, 148.1, 139.6, 138.4, 134.7, 134.4, 133.4, 131.9, 129.4, 129.2, 127.8, 127.5, 123.0, 120.0, 117.8, 117.0, 86.9, 84.3, 43.2; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}ClN_5O_7S_2^+$, 572.0096, found: 572.0098;

N-(3-(4-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2d)



The title compound **2d** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (105 mg, 85% yield), m.p. = 193–195 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.79 (s, 1H), 8.13 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.89 – 7.84 (m, 3H), 7.82 (t, *J* = 5.0 Hz, 1H), 7.72 (t, *J* = 5.0 Hz, 1H), 7.65 (t, *J* = 10.0 Hz, 2H), 7.40 (d, *J* = 10.0 Hz, 2H), 7.06 (d, *J* = 10.0 Hz, 1H), 4.91 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 162.3, 154.7, 150.6, 147.4, 139.5, 136.6, 133.8, 133.2, 132.3, 131.8, 129.0, 127.6, 127.4, 125.2, 123.4, 120.5, 117.2, 116.9, 86.8, 84.7, 43.0; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}BrN_5O_7S_2^+$, 615.9591, found: 615.9590;

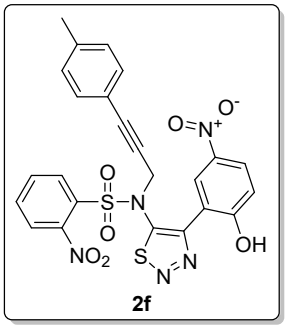
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-N-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-2-nitrobenzenesulfonamide (2e)



The title compound **2e** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (96 mg, 85% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.90 (s, 1H), 8.14 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.91 (d, *J* = 5.0 Hz, 1H), 7.86 (t, *J* = 10.0 Hz, 2H), 7.82 (t, *J* = 10.0 Hz, 1H), 7.73 (t, *J* = 10.0 Hz, 1H), 7.39 (d, *J* = 5.0 Hz, 2H), 7.05 (d, *J* = 10.0 Hz, 1H), 7.00 (d, *J* =

10.0 Hz, 2H), 4.89 (s, 2H), 3.80 (s, 3H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 162.3, 160.5, 154.7, 150.6, 147.5, 139.5, 136.5, 133.6, 133.2, 131.8, 129.1, 127.6, 127.4, 125.1, 117.3, 116.8, 114.9, 113.1, 88.3, 81.9, 74.4, 66.7, 56.3, 55.8, 43.1; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{24}H_{18}N_5O_8S_2^+$, 568.0591, found: 568.0590;

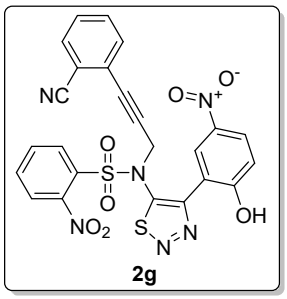
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitro-N-(3-(p-tolyl)prop-2-yn-1-yl)benzenesulfonamide (2f)



2f

The title compound **2f** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (91 mg, 83% yield), m.p. = 187–188 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.86 (s, 1H), 8.13 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.90 – 7.84 (m, 3H), 7.81 (t, *J* = 5.0 Hz, 1H), 7.71 (t, *J* = 5.0 Hz, 1H), 7.53 – 7.50 (m, 2H), 7.30 (t, *J* = 5.0 Hz, 2H), 7.05 (d, *J* = 10.0 Hz, 1H), 4.91 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 163.8, 162.3, 161.9, 154.7, 150.6, 147.4, 139.5, 136.6, 134.4, 134.3, 133.2, 131.8, 129.1, 127.6, 127.4, 125.2, 117.7, 117.3, 116.9, 116.6, 116.5, 86.9, 83.3, 42.9; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{24}H_{18}N_5O_7S_2^+$, 552.0642, found: 552.0645;

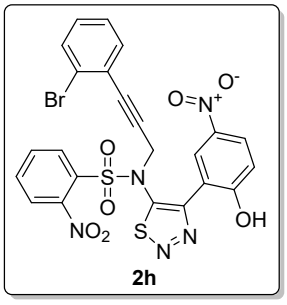
N-(3-(2-cyanophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2g)



2g

The title compound **2g** was purified by flash chromatography in PE/EA = 2/1-1/1 as yellow oil (96 mg, 85% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.82 (s, 1H), 8.12 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.91 – 7.82 (m, 3H), 7.80 (td, *J* = 7.7, 1.4 Hz, 1H), 7.70 (td, *J* = 7.7, 1.3 Hz, 1H), 7.55 – 7.51 (m, 2H), 7.39 – 7.31 (m, 1H), 7.28 (td, *J* = 7.6, 1.1 Hz, 1H), 7.04 (d, *J* = 9.2 Hz, 1H), 4.96 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 154.7, 151.3, 148.1, 138.4, 134.7, 134.4, 133.4, 131.9, 129.4, 129.2, 127.8, 127.5, 123.0, 120.0, 117.8, 117.0, 86.9, 84.3, 43.2; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{24}H_{15}N_6O_7S_2^+$, 563.0438, found: 563.0441;

N-(3-(2-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2h)

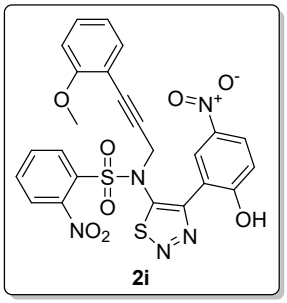


2h

The title compound **2h** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (95 mg, 77% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.79 (s, 1H), 8.13 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.91 (d, *J* = 5.0 Hz, 1H), 7.89 – 7.85 (m,

2H), 7.82 (t, *J* = 5.0 Hz, 1H), 7.74 – 7.71 (m, 2H), 7.56 (d, *J* = 5.0 Hz, 1H), 7.47 (t, *J* = 5.0 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.06 (d, *J* = 10.0 Hz, 1H), 4.97 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d$_6$)** δ 172.4, 162.2, 154.5, 153.6, 147.4, 139.6, 136.5, 134.3, 133.3, 133.0, 131.8, 131.6, 129.3, 128.3, 127.5, 125.3, 125.0, 123.3, 117.2, 116.8, 87.7, 86.0, 43.0, 21.4; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}BrN_5O_7S_2^+$, 615.9591, found: 615.9593;

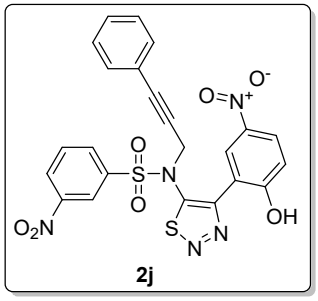
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-N-(3-(2-methoxyphenyl)prop-2-yn-1-yl)-2-nitrobenzenesulfonamide (2i)



2i

The title compound **2i** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (82 mg, 72% yield), m.p. = 189–191 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d$_6$)** δ 11.99 (s, 1H), 8.39 (d, *J* = 5.0 Hz, 1H), 8.29 (d, *J* = 10.0 Hz, 1H), 8.24 (q, *J* = 5.0 Hz, 1H), 8.20 (d, *J* = 10.0 Hz, 1H), 8.01 (t, *J* = 10.0 Hz, 1H), 7.89 (t, *J* = 10.0 Hz, 1H), 7.75 (d, *J* = 10.0 Hz, 2H), 7.52 (d, *J* = 5.0 Hz, 1H), 7.44 (d, *J* = 5.0 Hz, 2H), 7.33 – 7.24 (m, 4H), 7.11 (d, *J* = 10.0 Hz, 1H), 5.34 (s, 2H), 3.35 (s, 3H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d$_6$)** δ 184.7, 162.2, 154.1, 147.7, 146.7, 144.3, 138.6, 137.7, 137.2, 136.3, 133.9, 132.5, 132.0, 131.1, 130.4, 130.3, 129.2, 129.1, 128.4, 127.6, 126.3, 125.2, 121.6, 121.4, 84.9, 83.2, 42.7, 21.4, 21.1; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{24}H_{18}N_5O_8S_2^+$, 568.0591, found: 568.0590;

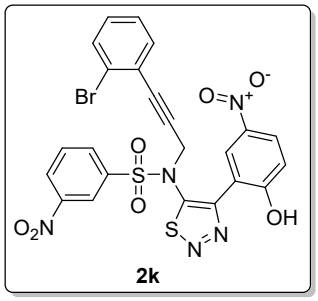
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitro-N-(3-phenylprop-2-yn-1-yl)benzenesulfonamide (2j)



2j

The title compound **2j** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (89 mg, 83% yield), m.p. = 173–175 ºC; **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.38 (d, *J* = 5.0 Hz, 1H), 8.11 (dd, *J* = 10.0, 5.0 Hz, 1H), 7.92 (d, *J* = 10.0 Hz, 1H), 7.81 (d, J = 10.0 Hz, 1H), 7.77 (t, *J* = 10.0 Hz, 1H), 7.63 (t, *J* = 10.0 Hz, 1H), 7.41 – 7.32 (m, 5H), 7.14 (d, *J* = 10.0 Hz, 1H), 4.92 (s, 2H); **^{13}C NMR (126 MHz, Chloroform-*d*)** δ 161.7, 152.2, 150.2, 147.8, 140.4, 135.5, 132.5, 131.8, 131.3, 131.2, 129.4, 128.4, 127.2, 125.5, 122.8, 121.0, 118.9, 114.0, 89.2, 80.9, 44.2; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{16}N_5O_7S_2^+$, 538.0486, found: 538.0482;

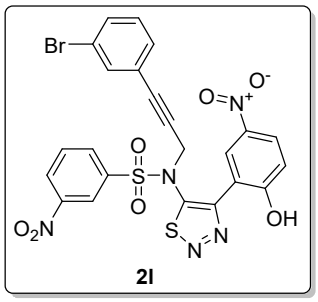
N-(3-(2-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2k)



2k

The title compound **2k** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (96 mg, 78% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 7.95 (d, *J* = 10.0 Hz, 1H), 7.72 (d, *J* = 10.0 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.63 (t, *J* = 5.0 Hz, 1H), 7.54 (t, *J* = 5.0 Hz, 1H), 7.28 – 7.25 (m, 5H), 6.88 (d, *J* = 10.0 Hz, 1H), 4.73 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 163.0, 162.8, 154.7, 151.2, 148.1, 144.9, 139.4, 138.5, 134.4, 133.8, 132.9, 131.9, 131.2, 130.7, 130.0, 129.1, 128.8, 128.6, 127.8, 127.5, 126.4, 123.2, 123.0, 122.1, 117.8, 117.1, 116.7, 115.9, 86.3, 84.6, 43.1; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}BrN_5O_7S_2^+$, 615.9591, found: 615.9594;

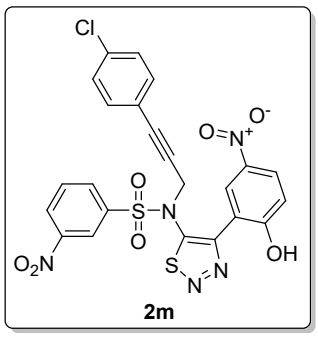
N-(3-(3-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2l)



2l

The title compound **2l** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (93 mg, 76% yield), m.p. = 187–188 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.82 (s, 1H), 8.11 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.95 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.90 (d, *J* = 2.9 Hz, 1H), 7.87 (td, *J* = 8.0, 1.3 Hz, 2H), 7.81 – 7.76 (m, 2H), 7.73 – 7.63 (m, 3H), 7.04 (d, *J* = 9.1 Hz, 1H), 5.03 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 166.9, 159.4, 155.4, 152.1, 144.3, 141.3, 138.7, 138.2, 138.1, 136.6, 135.4, 133.9, 132.4, 132.2, 130.1, 129.2, 122.3, 121.9, 121.5, 119.1, 94.1, 88.5, 47.6; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}BrN_5O_7S_2^+$, 615.9591, found: 615.9592;

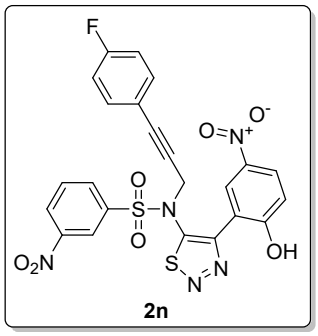
N-(3-(4-chlorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2m)



2m

The title compound **2m** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow solid (90 mg, 79% yield), m.p. = 162–165 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 10.05 (s, 1H), 8.12 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.89 (d, *J* = 2.9 Hz, 1H), 7.85 (td, *J* = 7.9, 1.3 Hz, 2H), 7.79 (td, *J* = 7.7, 1.4 Hz, 1H), 7.71 (td, *J* = 7.7, 1.3 Hz, 1H), 7.49 – 7.39 (m, 5H), 7.04 (d, *J* = 9.1 Hz, 1H), 4.92 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 162.4, 154.8, 150.6, 147.4, 139.4, 136.5, 133.2, 131.9, 131.9, 129.9, 129.3, 129.1, 127.6, 127.4, 125.2, 121.3, 117.3, 116.8, 88.0, 83.4, 42.9; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}ClN_5O_7S_2^+$, 572.0096, found: 572.0097;

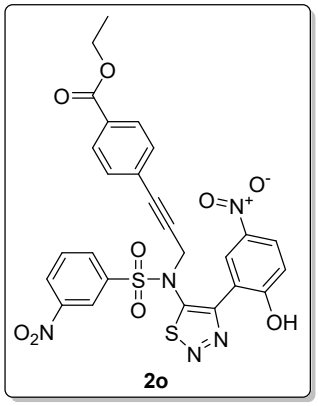
N-(3-(4-fluorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2n)



2n

The title compound **2n** was purified by flash chromatography in PE/EA = 4/1-2/1 as yellow oil (91 mg, 82% yield); **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.70 (s, 1H), 8.43 (d, *J* = 10.0 Hz, 1H), 8.35 (d, *J* = 5.0 Hz, 1H), 8.21 (s, 1H), 8.19 (d, *J* = 10.0 Hz, 1H), 8.10 (d, *J* = 10.0 Hz, 1H), 8.04 (d, *J* = 5.0 Hz, 1H), 7.80 (t, *J* = 5.0 Hz, 1H), 7.58 (d, *J* = 5.0 Hz, 1H), 7.39 – 7.35 (m, 3H), 7.26 (t, *J* = 10.0 Hz, 2H), 7.08 (d, *J* = 10.0 Hz, 1H), 6.91 (d, *J* = 10.0 Hz, 1H), 4.97 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 163.8, 162.9, 162.2, 161.8, 161.4, 154.6, 151.3, 148.0, 144.9, 139.8, 139.5, 138.4, 136.1, 134.4, 134.1, 134.0, 131.9, 130.5, 130.0, 129.2, 128.8, 128.6, 128.0, 127.7, 127.5, 126.4, 123.0, 117.8, 117.5, 117.0, 116.7, 116.6, 116.4, 115.9, 87.1, 82.9, 43.2; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}FN_5O_7S_2^+$, 556.0391, found: 556.0390;

ethyl 4-(3-((N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrophenyl)sulfonamido)prop-1-yn-1-yl)benzoate (2o)



2o

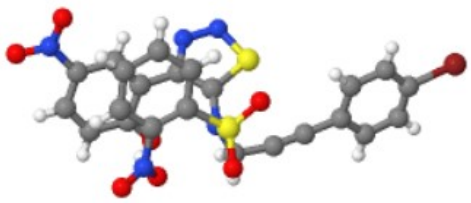
The title compound **2o** was purified by flash chromatography in PE/EA = 3/1-1/1 as yellow solid (91 mg, 75% yield), m.p. = 176–178 ºC; **^{1}H NMR (500 MHz, Dimethyl Sulfoxide-d_6)** δ 11.80 (s, 1H), 8.13 (dd, *J* = 10.0, 5.0 Hz, 1H), 8.00 (d, *J* = 5.0 Hz, 2H), 7.91 (d, *J* = 5.0 Hz, 1H), 7.89 – 7.85 (m, 2H), 7.82 (t, *J* = 5.0 Hz, 1H), 7.73 – 7.69 (m, 3H), 7.58 (d, *J* = 5.0 Hz, 2H), 7.41 (d, *J* = 10.0 Hz, 1H), 7.06 (d, *J* = 10.0 Hz, 1H), 4.95 (s, 2H); **^{13}C NMR (126 MHz, Dimethyl Sulfoxide-d_6)** δ 165.4, 162.2, 154.7, 150.7, 147.4, 139.5, 136.7, 133.2, 132.2, 131.9, 130.7, 129.9, 129.0, 127.6, 127.5, 125.9, 125.2, 117.2, 116.8, 86.9, 86.5, 61.5, 42.9, 14.5; **HRMS (ESI)** calcd for $(M+H)^+$ $C_{23}H_{15}ClN_5O_7S_2^+$, 610.0697, found: 610.0699;

4. **Crystal Structure of 2d and 2j**

Method for single crystals cultivation: The single crystal for compound 2a and 2j were prepared from a mixture solvent of Dichloromethane/ Ethyl acetate and Petroleum ether (v/v = 1:1). a pure solid sample (10–20 mg) was dissolved in dichloromethane/ethyl acetate (2 mL) in a vial at room temperature, and petroleum ether/hexane (2-3 mL) was added into the above solution slowly while keeping the sample completely dissolved. The vial was properly sealed with parafilm and kept at room temperature to allow the slow evaporation of the solvents until a single crystal was obtained.

The data were collected on a Agilent Gemini E diffractometer（Mo，50kV 40mA）instrument using Mo-Kαradiation (λ = 0.71073 Å) at 296 K and reducted by CrysAlisPro (Rigaku). The crystal structures were solved and refined using the SHELXTL software package. Refinements were performed with SHELXL-2013 using fullmatrix least-squares calculations on F2, with anisotropic displacement parameters for all the nonhydrogen atoms. The crystallographic data have already been deposited at the Cambridge Crystallographic Data Centre.

Crystallographic data for compound **2d** (CCDC-2085419) has been deposited with the Cambridge Crystallographic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email:deposit@ccdc.cam.ac.uk). Thermal ellipsoids are drawn at 50% probability level.



Bond precision: C-C = 0.0076 A Wavelength=1.54178

Cell: a=23.7139(19) b=7.3466(6) c=14.3578(11)
alpha=90 beta=92.434(4) gamma=90

Temperature: 298 K

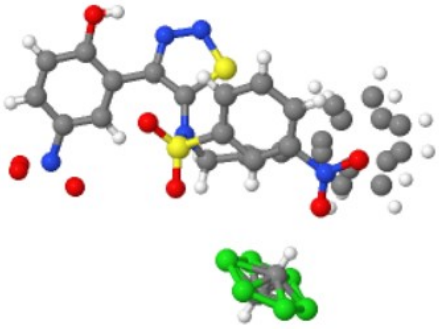
	Calculated	Reported
Volume	2499.1(3)	2499.1(3)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C23 H14 Br N5 O7 S2	C23 H14 Br N5 O7 S2
Sum formula	C23 H14 Br N5 O7 S2	C23 H14 Br N5 O7 S2
Mr	616.41	616.42
Dx,g cm-3	1.638	1.638
Z	4	4
Mu (mm-1)	4.291	4.291
F000	1240.0	1240.0
F000'	1243.45	

h,k,lmax	28,8,17	28,8,17
Nref	4602	4581
Tmin,Tmax	0.411,0.709	0.546,0.753
Tmin'	0.310	

Correction method= # Reported T Limits: Tmin=0.546 Tmax=0.753
AbsCorr = MULTI-SCAN

Data completeness= 0.995 Theta(max)= 68.607
R(reflections)= 0.0681(4228) wR2(reflections)= 0.1682(4581)
S = 1.148 Npar= 344

Crystallographic data for compound **2j** (CCDC-1963640) has been deposited with the Cambridge Crystallographic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email:deposit@ccdc.cam.ac.uk). Thermal ellipsoids are drawn at 50% probability level.



Bond precision: C-C = 0.0041 A Wavelength=0.71073
Cell: a=10.6408(8) b=10.8920(7) c=12.6948(11)
alpha=71.578(7) beta=68.187(7) gamma=89.832(5)
Temperature: 300 K

	Calculated	Reported
Volume	1284.78(19)	1284.78(18)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	2(C23 H15 N5 O7 S2), C H Cl3	0.5 (C H Cl3), C23 H15 N5 O7 S2
Sum formula	C47 H31 Cl3 N10 O14 S4	C23.50 H15.50 N5 O7 S2
Mr	1194.41	597.20
Dx,g cm-3	1.544	1.544
Z	1	2
Mu (mm-1)	0.418	0.418
F000	610.0	610.0
F000'	611.21	
h,k,lmax	14,15,17	14,14,16
Nref	7098	5744
Tmin,Tmax	0.951,0.959	0.765,1.000
Tmin'	0.951	

Correction method= # Reported T Limits: Tmin=0.765 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.809 Theta(max)= 29.439
R(reflections)= 0.0597(4023) wR2(reflections)= 0.2062(3280)
S = 1.050 Npar= 421

5. References

[1] a) Jepsen, T. H.; Glibstrup, E.; Crestey, F.; Jensen, A. A.; and Kristensen J. L. A strategic approach to [6,6]-bicyclic lactones: application towards the CD fragment of DhβE. *Beilstein J. Org. Chem.,* **2017,** *13,* 988–994; b) Gao, M.; Gao, Q. Q.; Hao, X. B.; Wu, Y.; Zhang, Q. M.; Liu, G. H.; and Liu. R. Ruthenium Carbene-Mediated Construction of Strained Allenes via the Enyne Cross-Metathesis/Cyclopropanation of 1,6-Enynes. *Org. Lett.,* **2020**, *22*, 1139-1143; c) Sui, X. L.; Zhang, T. Q.; Pabarue, A. B.; Fu, L. B. and Gutekunst W. R. Alternating Cascade Metathesis Polymerization of Enynes and Cyclic Enol Ethers with Active Ruthenium Fischer Carbenes. *J. Am. Chem. Soc*., **2020**, *142*, 12942-12947; d) Kaushik, C. P.; Pahwa, A.; Kumar, A.; Singh, D. & Kumar, K. Facile synthesis, characterization, and antimicrobial studies of some disubstituted 1,2,3-triazoles with sulfonamide functionality. *Synthetic Communications*, **2017**, *47*:16, 1485-1494

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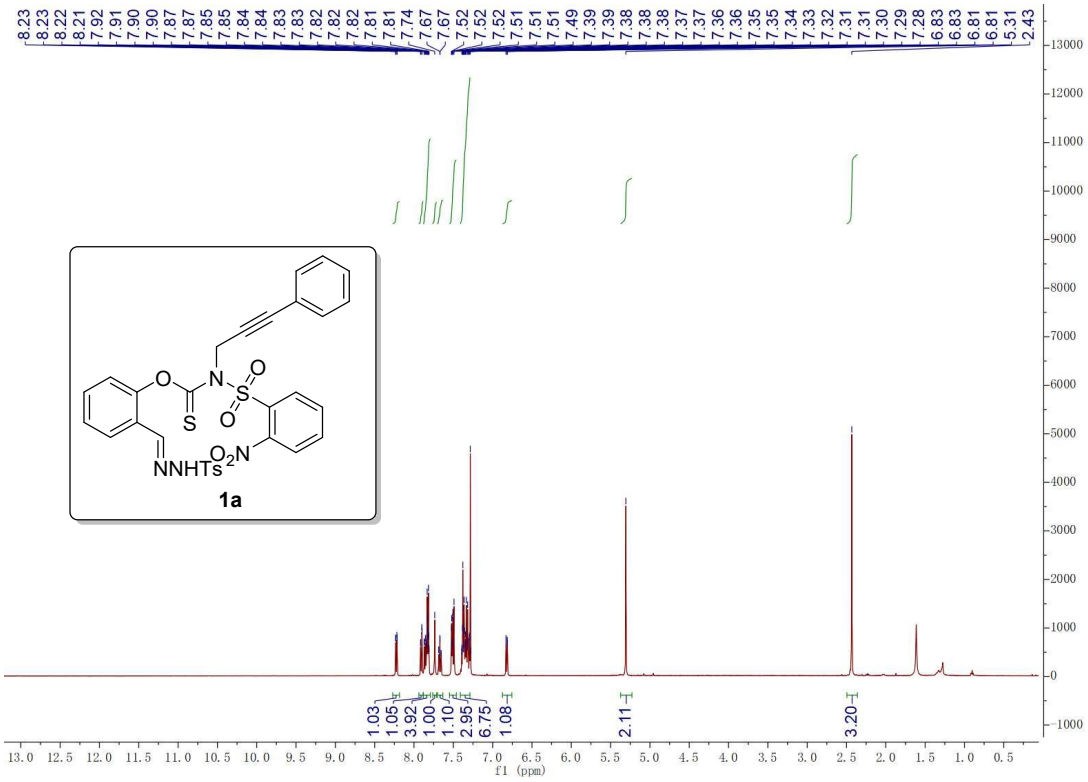
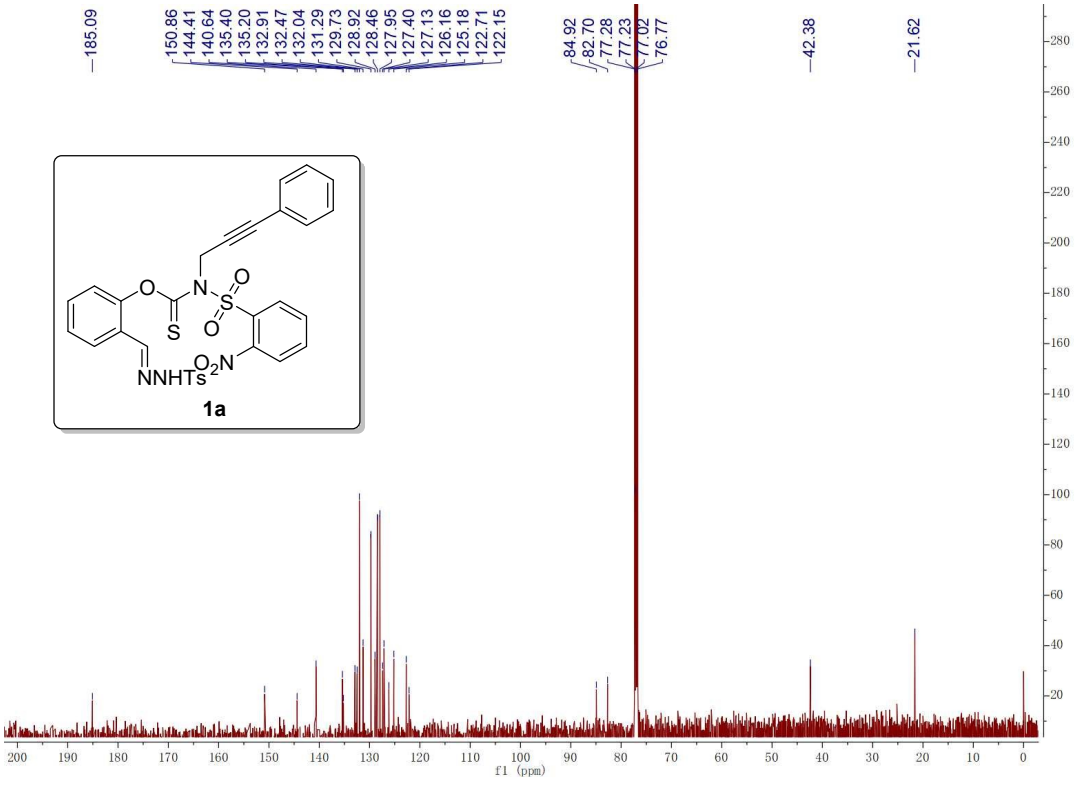
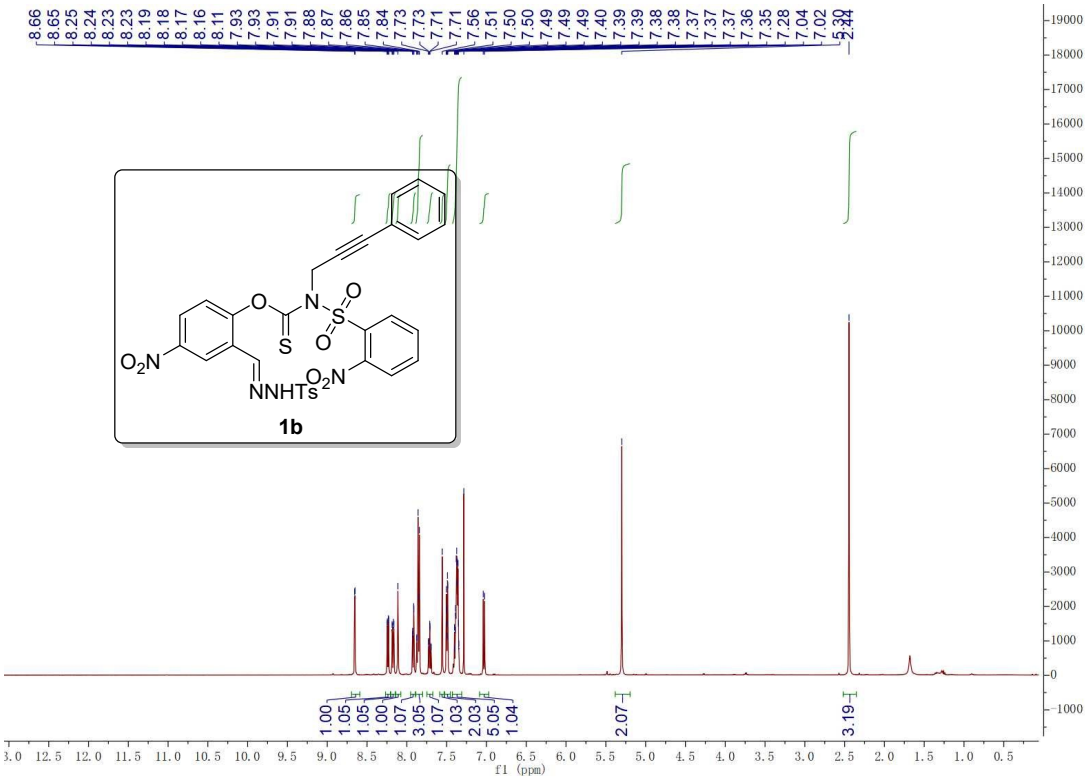
[4] a) Wang, Y.; Lin, J. B.; Xie, J. K.; Lu, H.; Hu, X. Q. and Xu, P. F. Dearomative Dienolate-Mediated Catalysis: A Remote Activation Strategy for Asymmetric Functionalization of Benzylic C-H Bonds of Heteroaryl Aldehydes. *Org. Lett*., **2018**, *20*, 18, 5835–5839; b) Yu, Y. H.; Chakraborty, P.; Song, J. S.; Zhu, L.; Li C. S. and Huang, X. L. Easy access to medium-sized lactones through metal carbene migratory insertion enabled 1,4-palladium shift. *Nat. Commun*., **2020**, *11*, 461; c) Kurma, S. H.; Sridhar, B. and Bhimapaka, C. R. Direct Access for the Regio- and Stereoselective Synthesis of *N*‑Alkenylpyrazoles and Chromenopyrazoles. *J. Org. Chem*., **2021**, *86*, 3, 2271–2282.

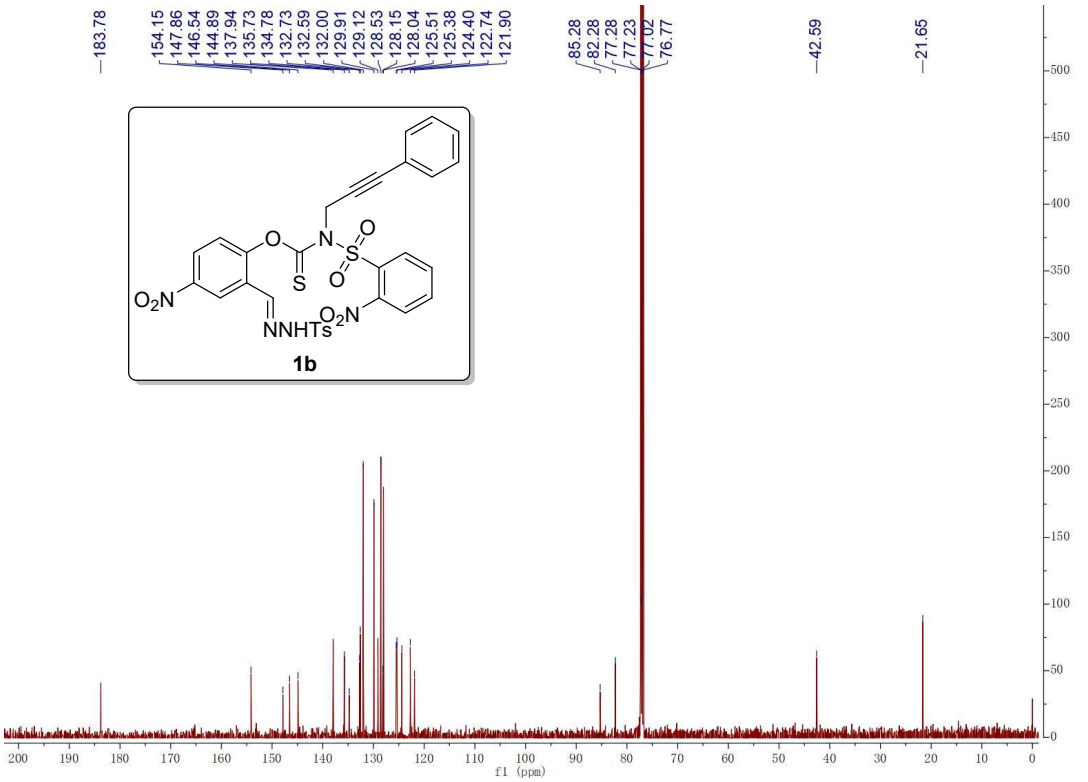
6. NMR Spectra of substrate 1 and product



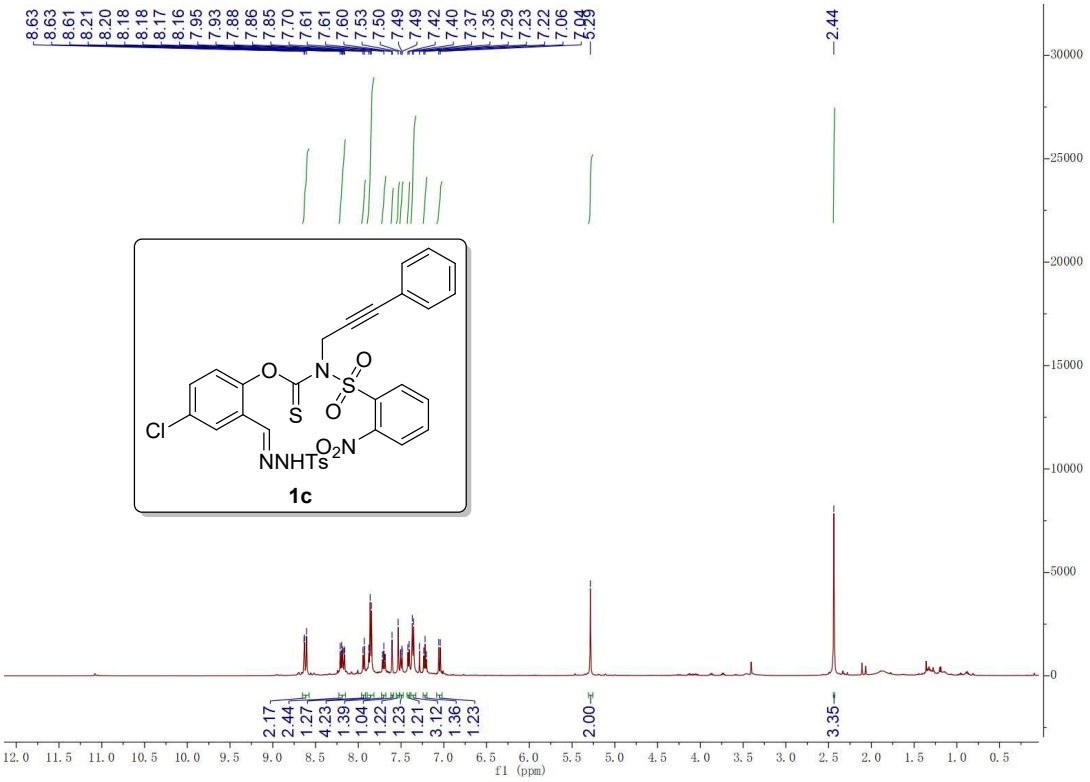


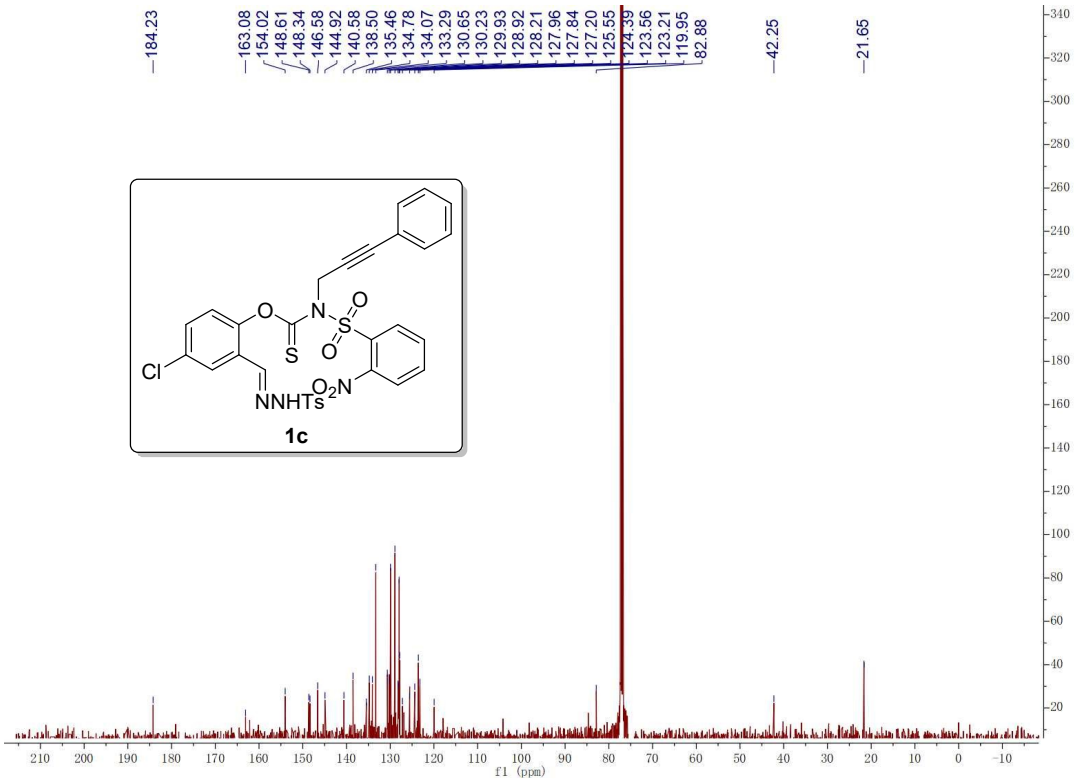
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1b)





(E)-O-(4-chloro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((2-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1c)





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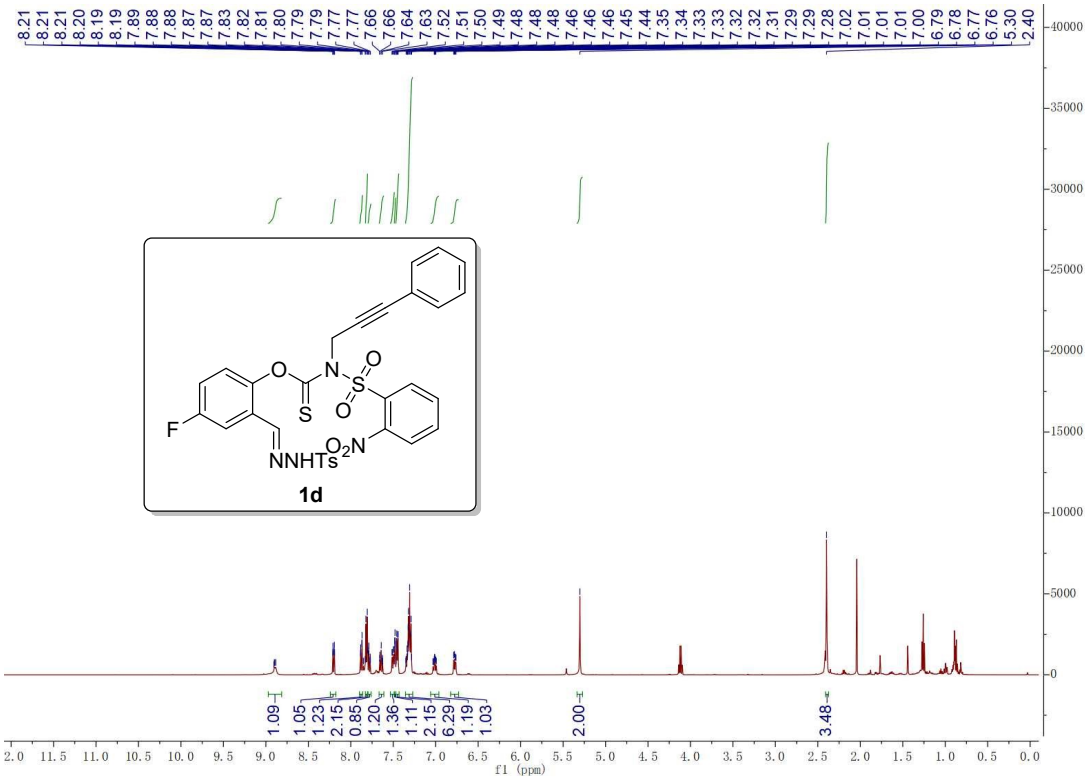
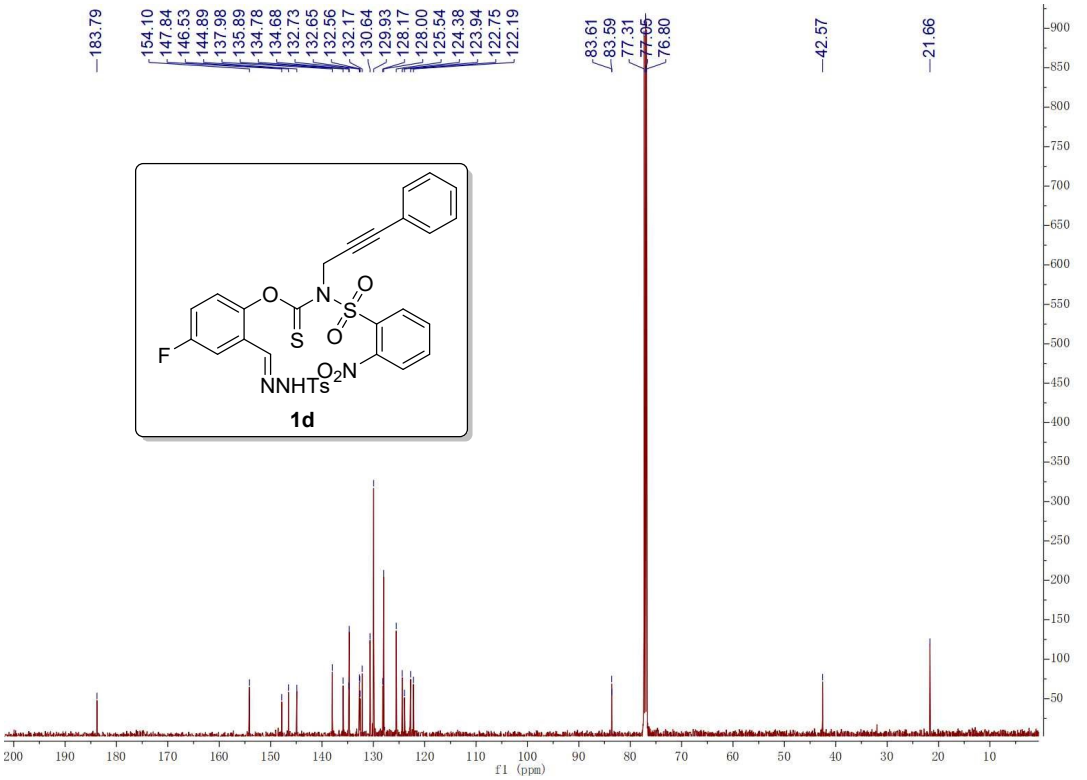
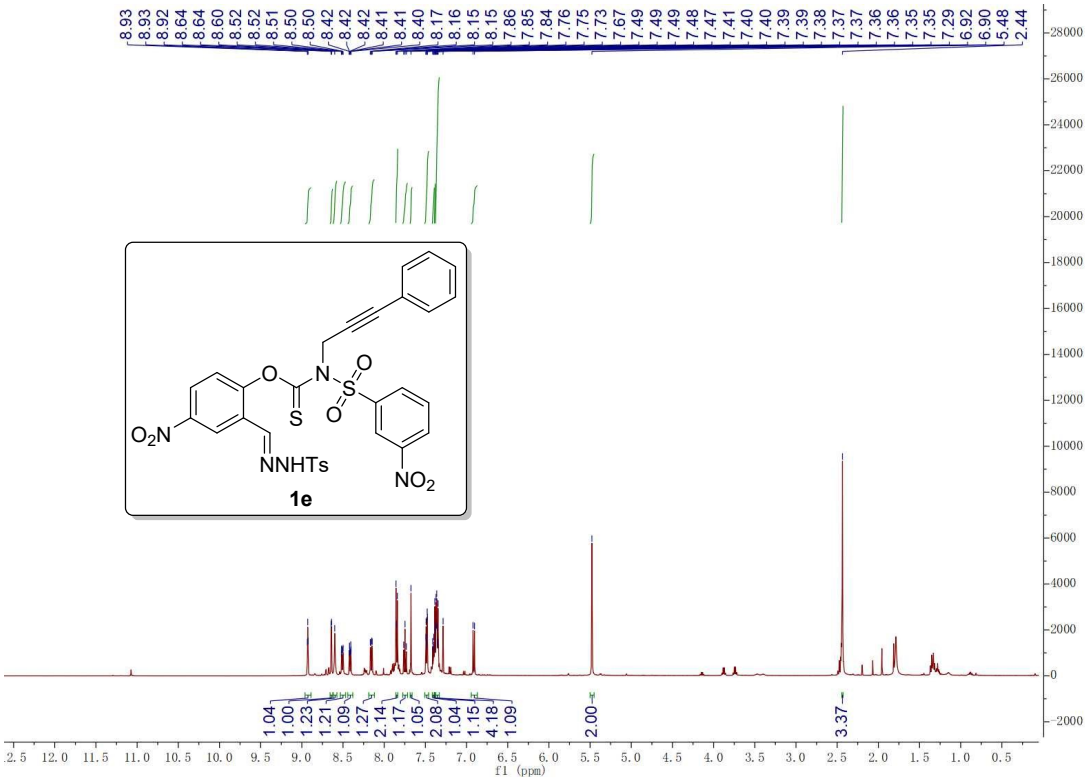
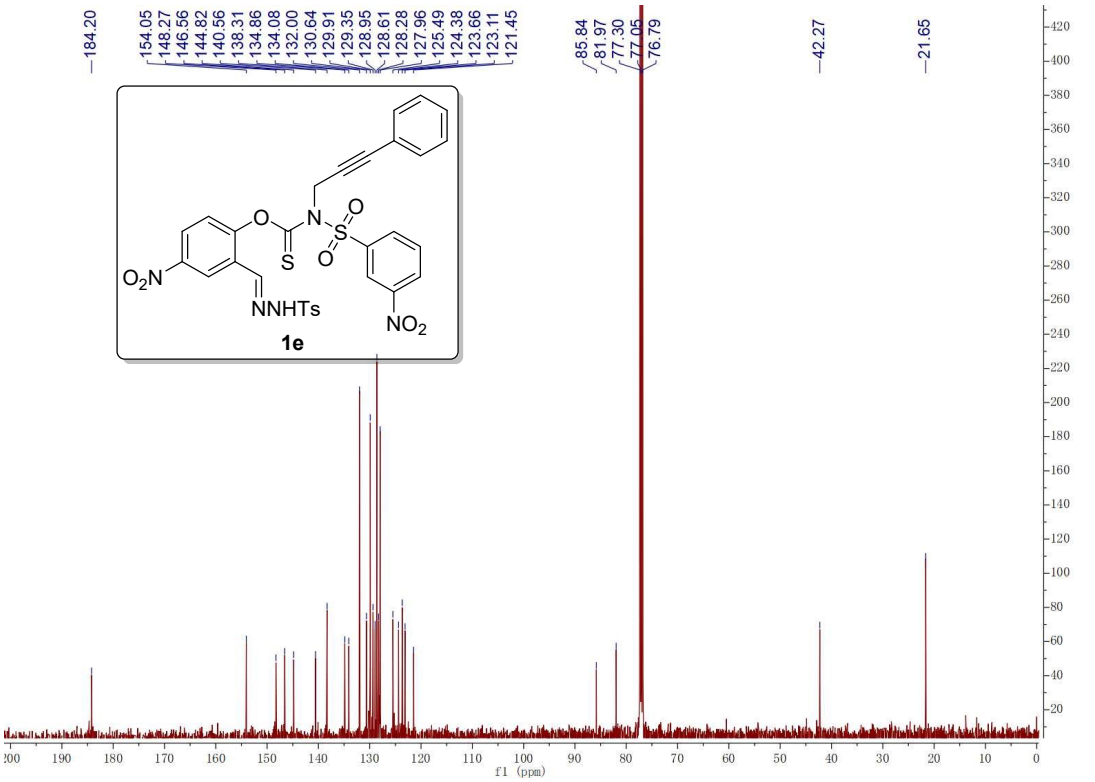


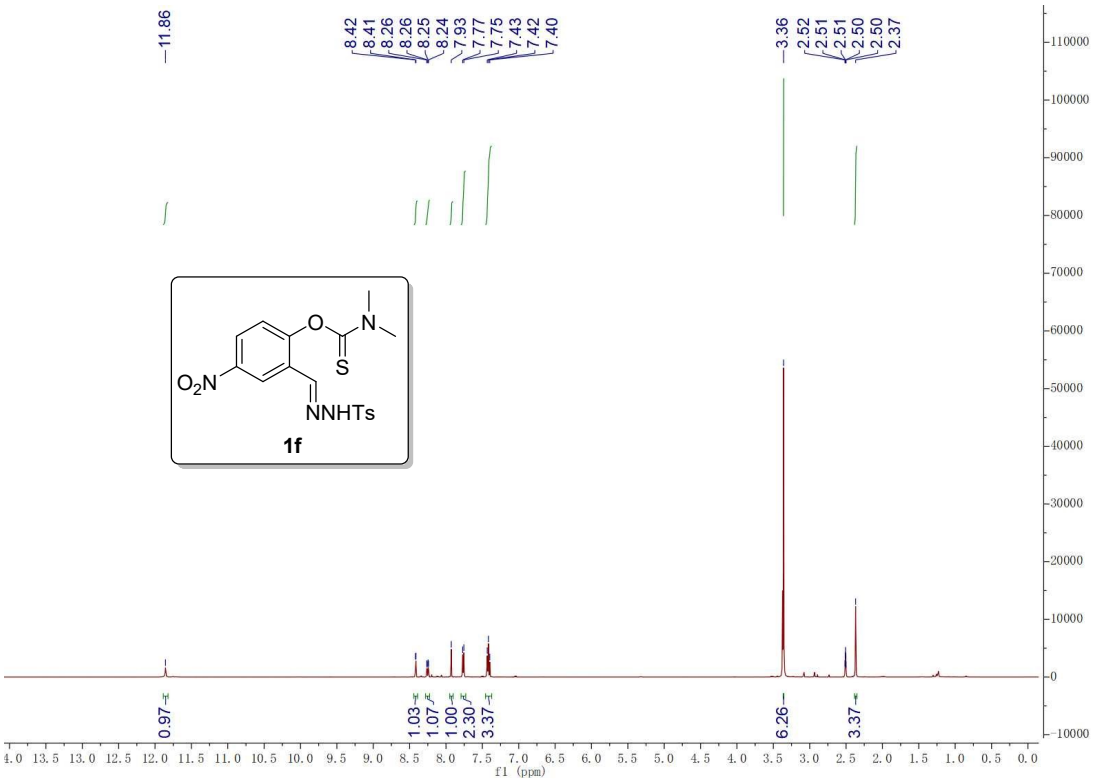


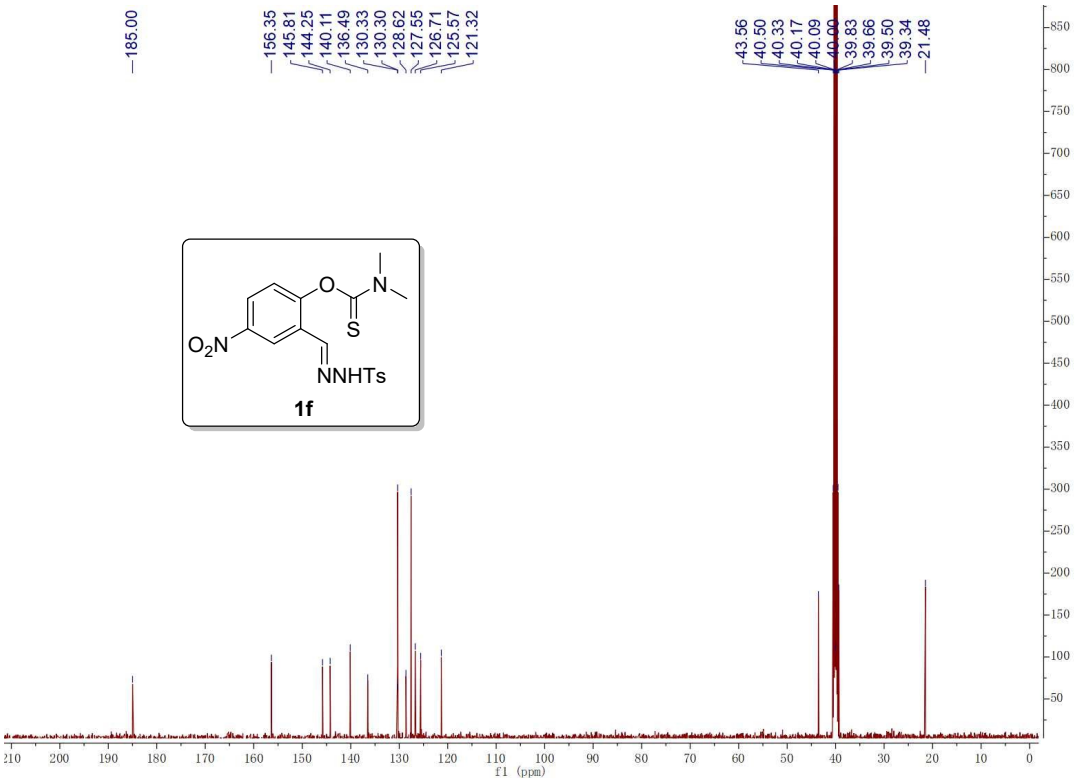
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) ((3-nitrophenyl)sulfonyl)(3-phenylprop-2-yn-1-yl)carbamothioate (1e)



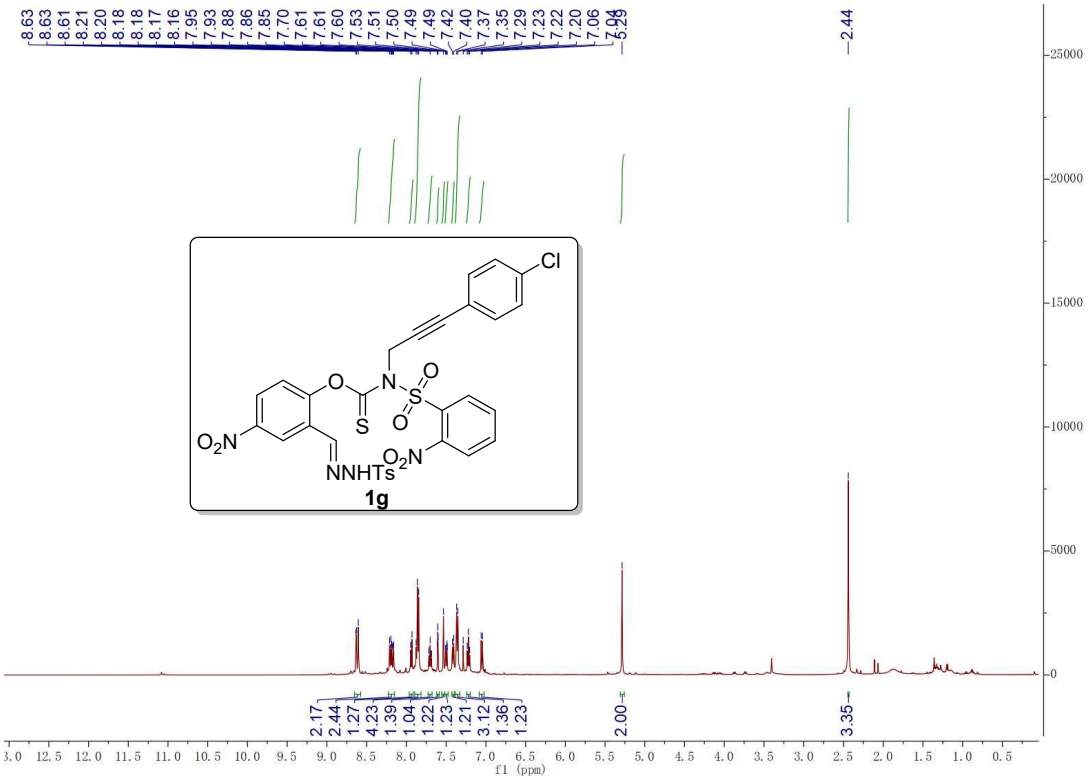


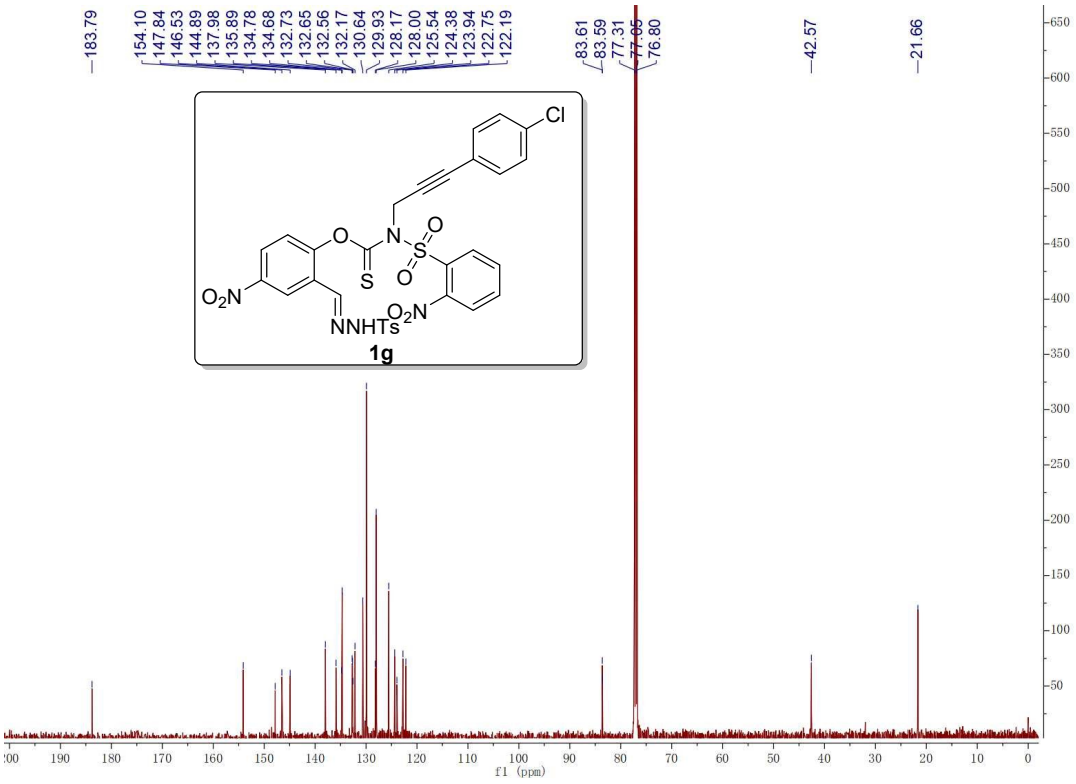
(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) dimethylcarbamothioate (1f)





(E)-O-(4-nitro-2-((2-tosylhydrazineylidene)methyl)phenyl) (3-(4-chlorophenyl)prop-2-yn-1-yl)((2-nitrophenyl)sulfonyl)carbamothioate (1g)





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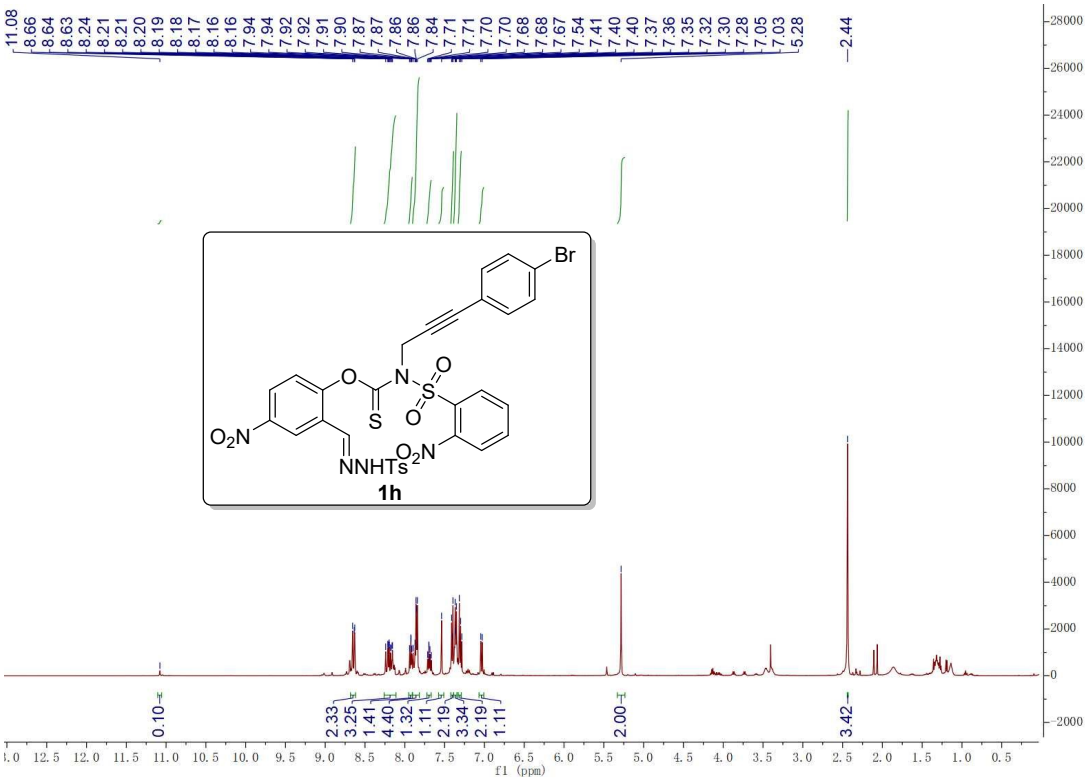

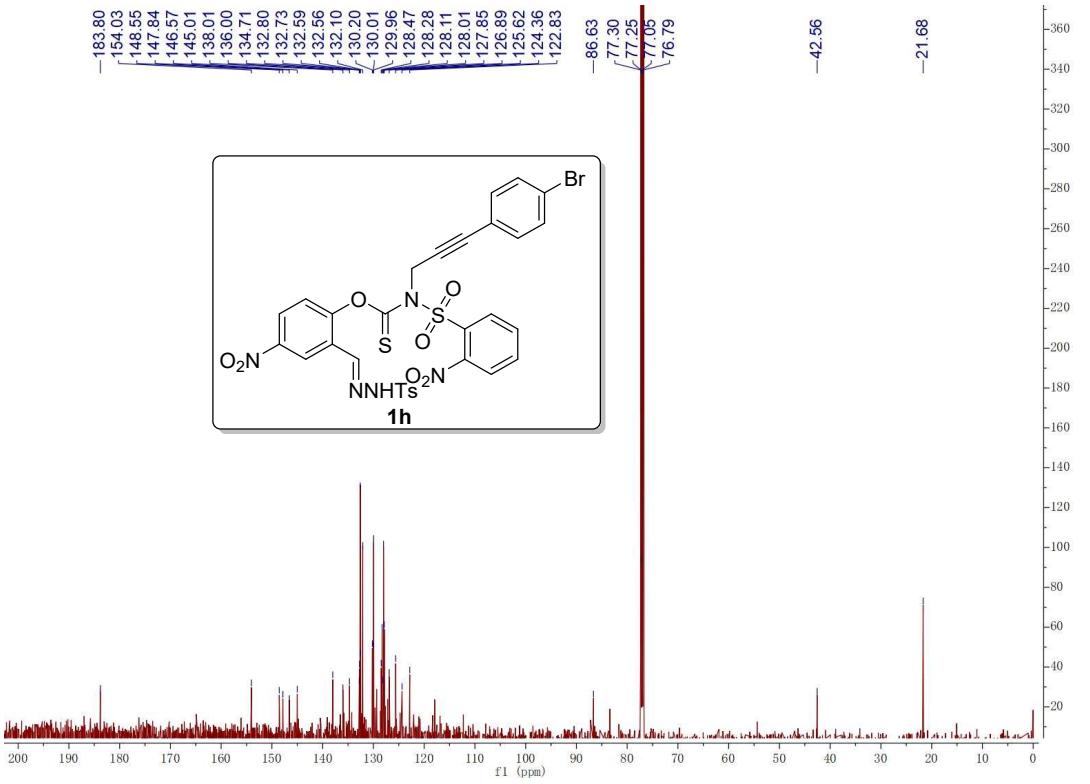

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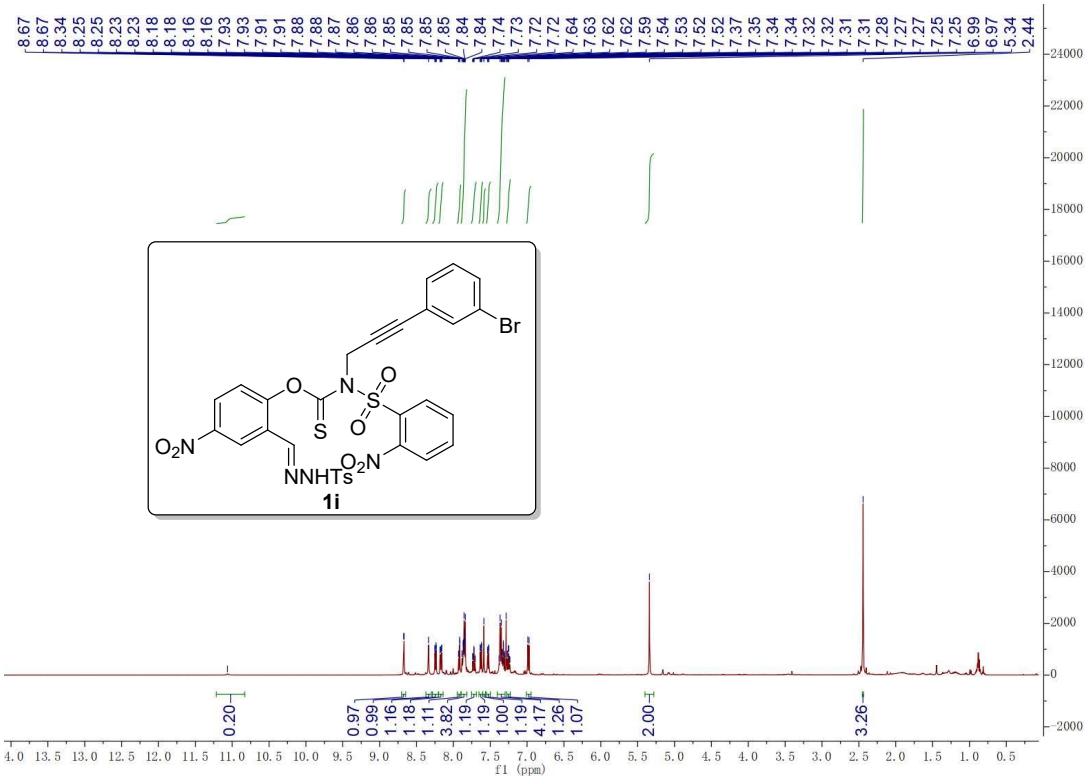
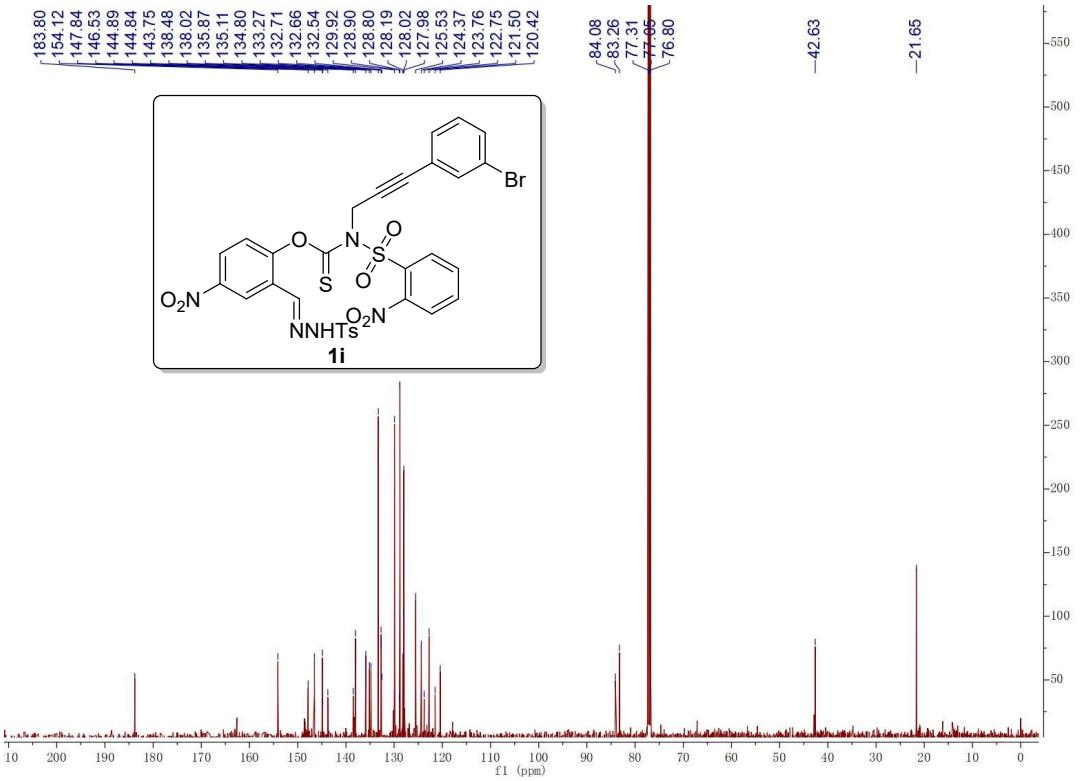
yl)((2-nitrophenyl)sulfonyl)carbamothioate (1i)





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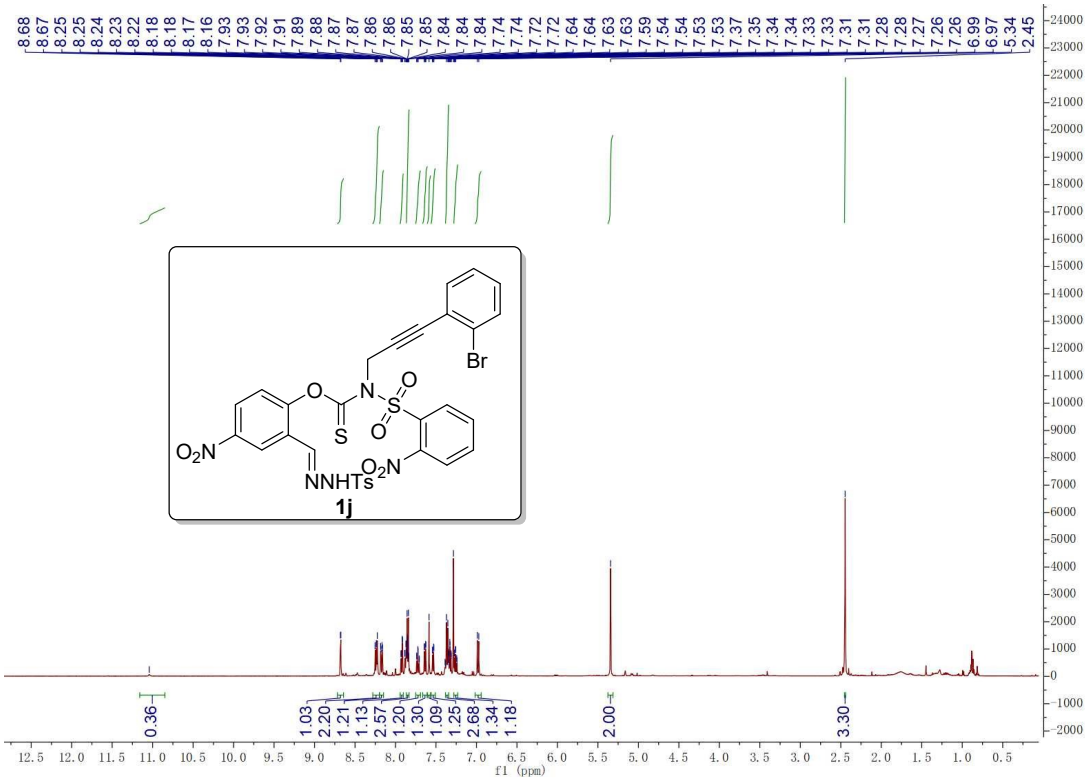

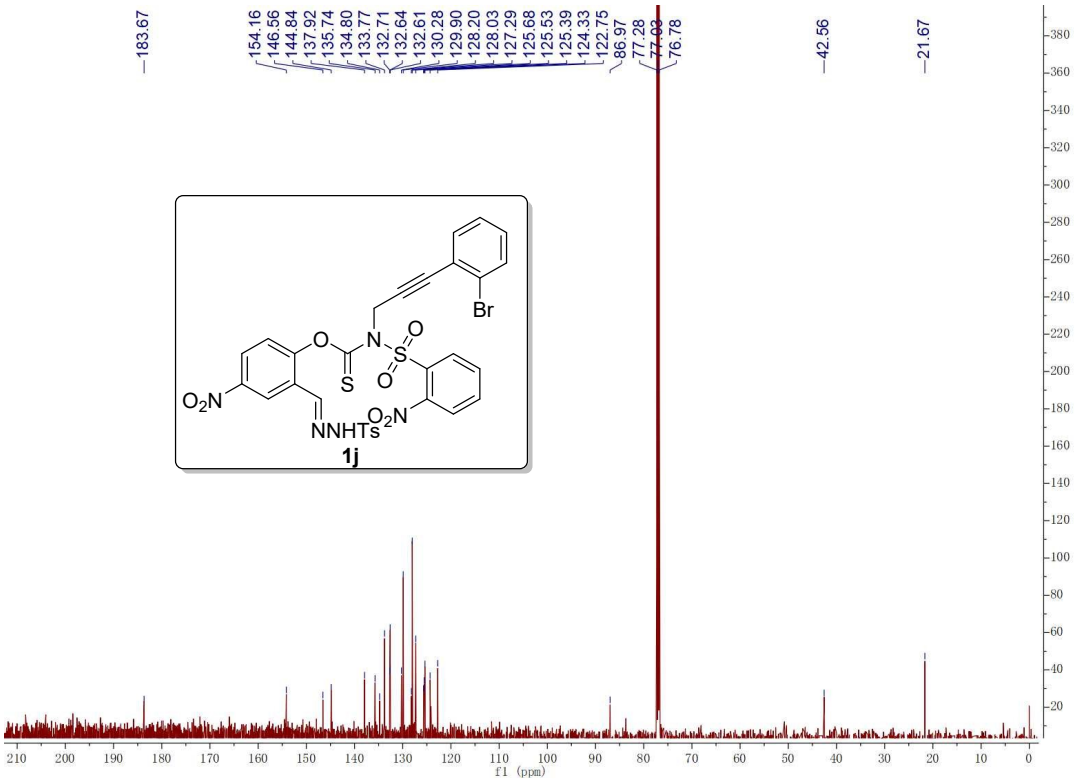

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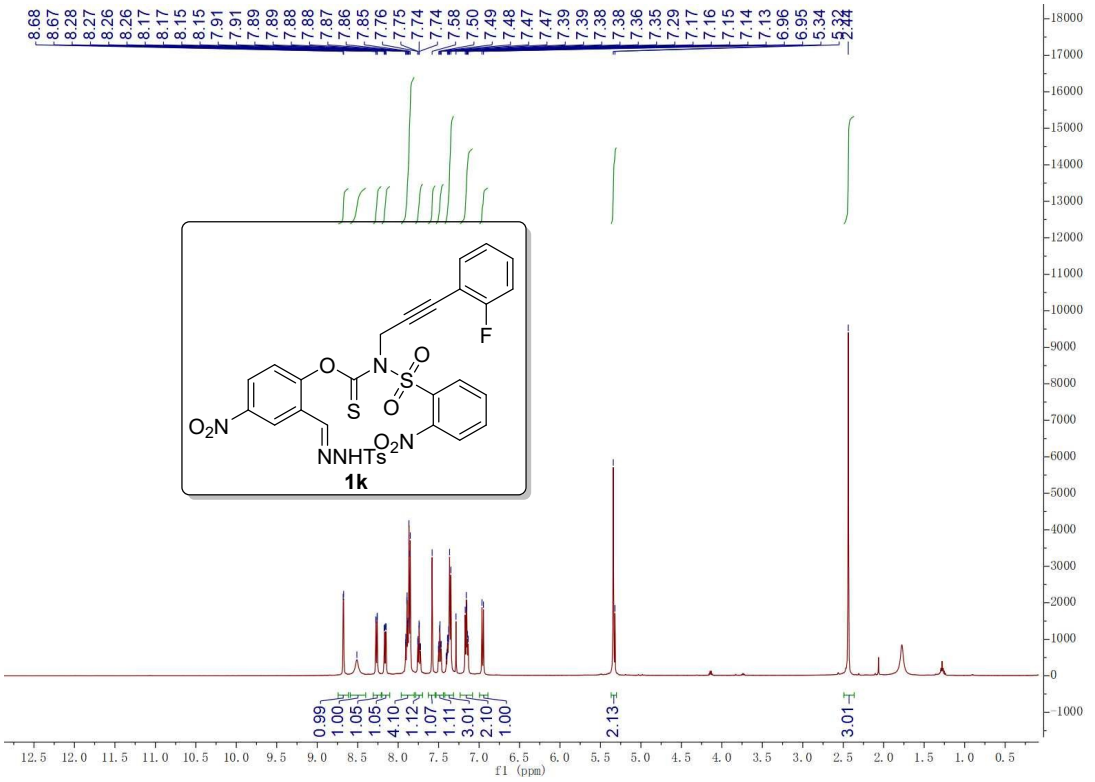

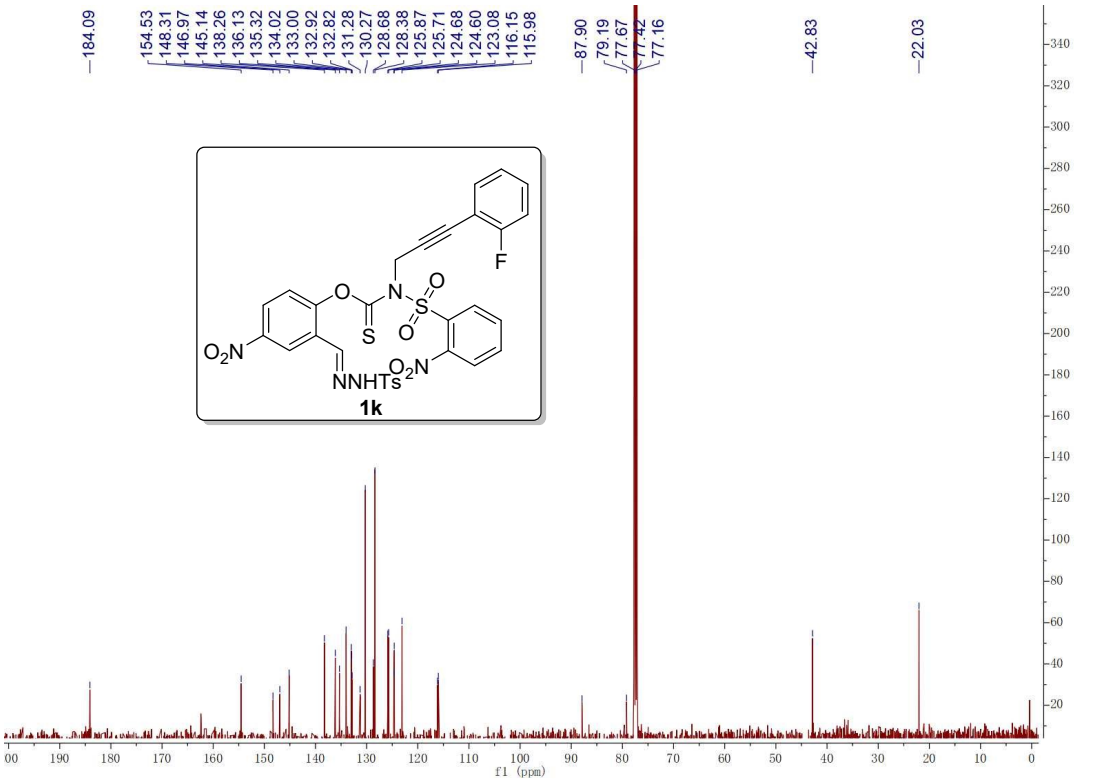

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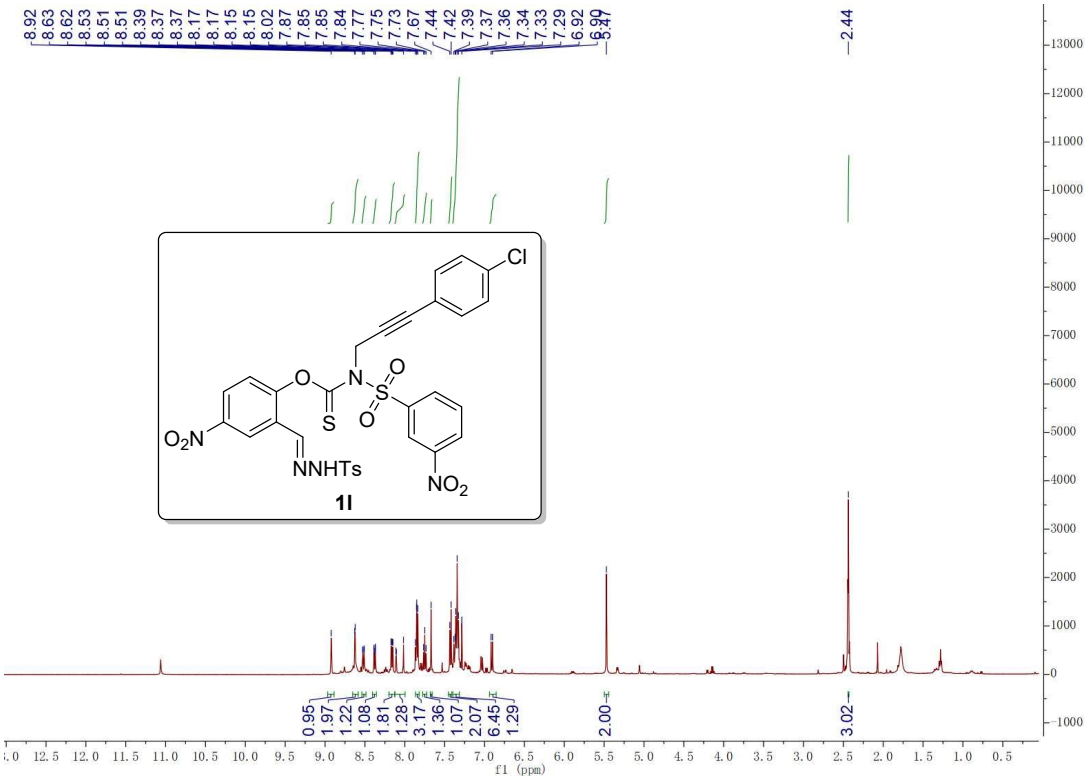

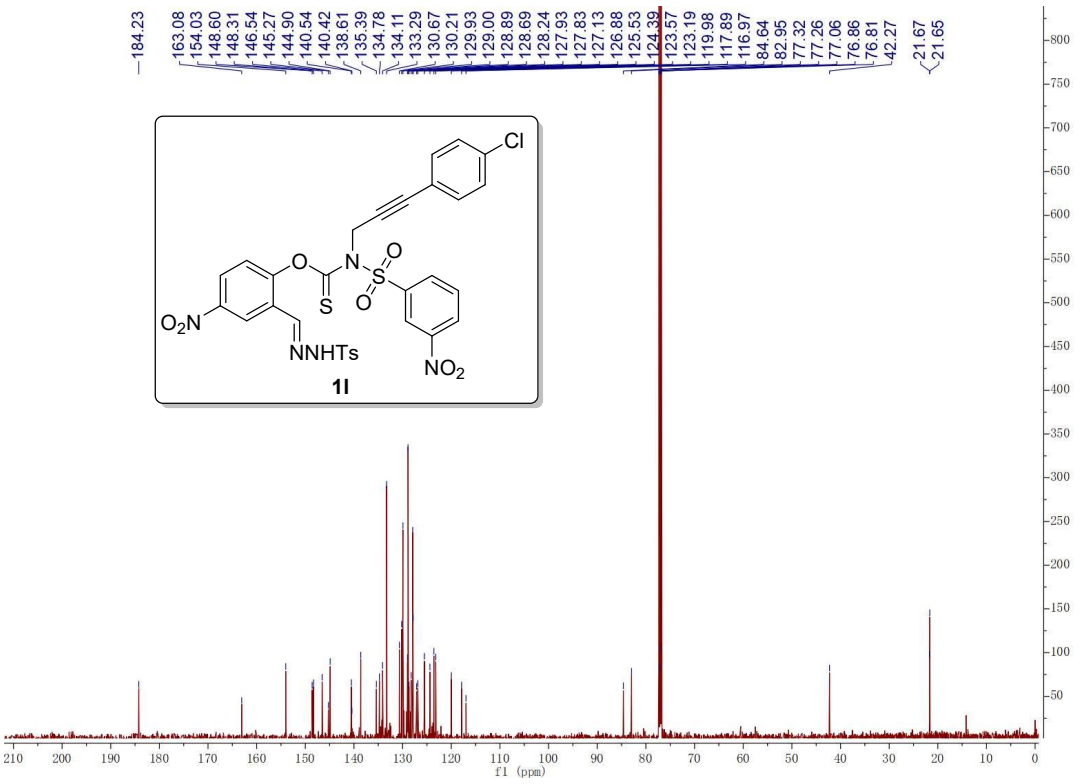

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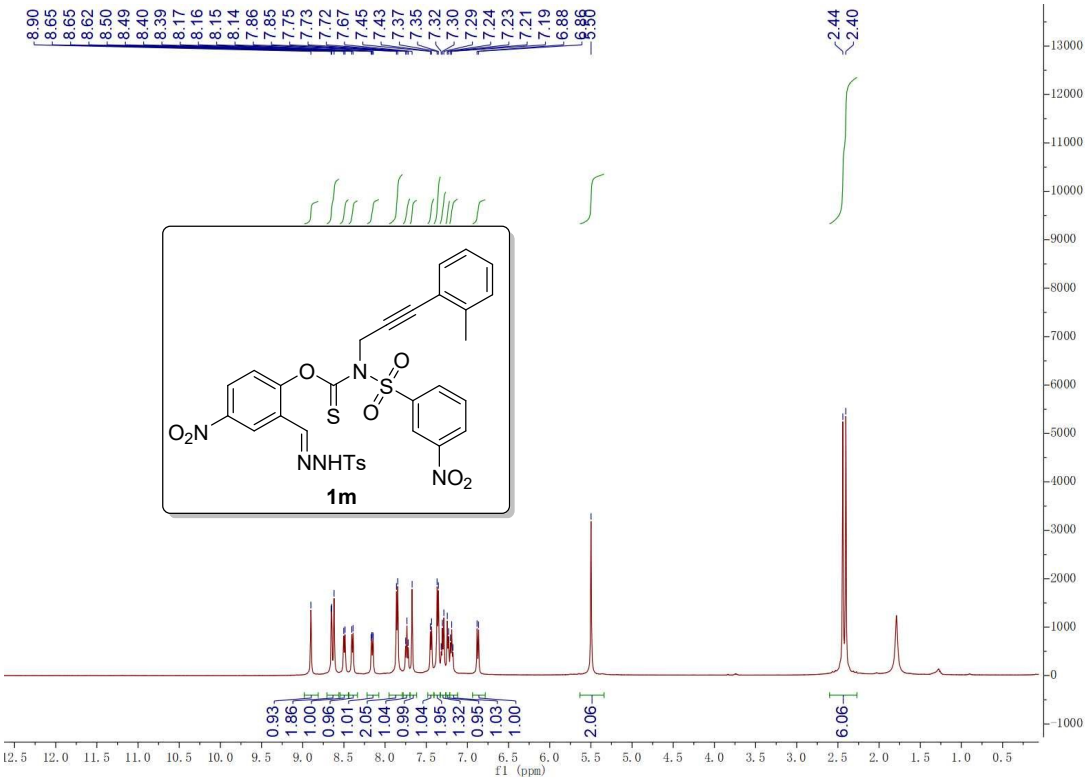

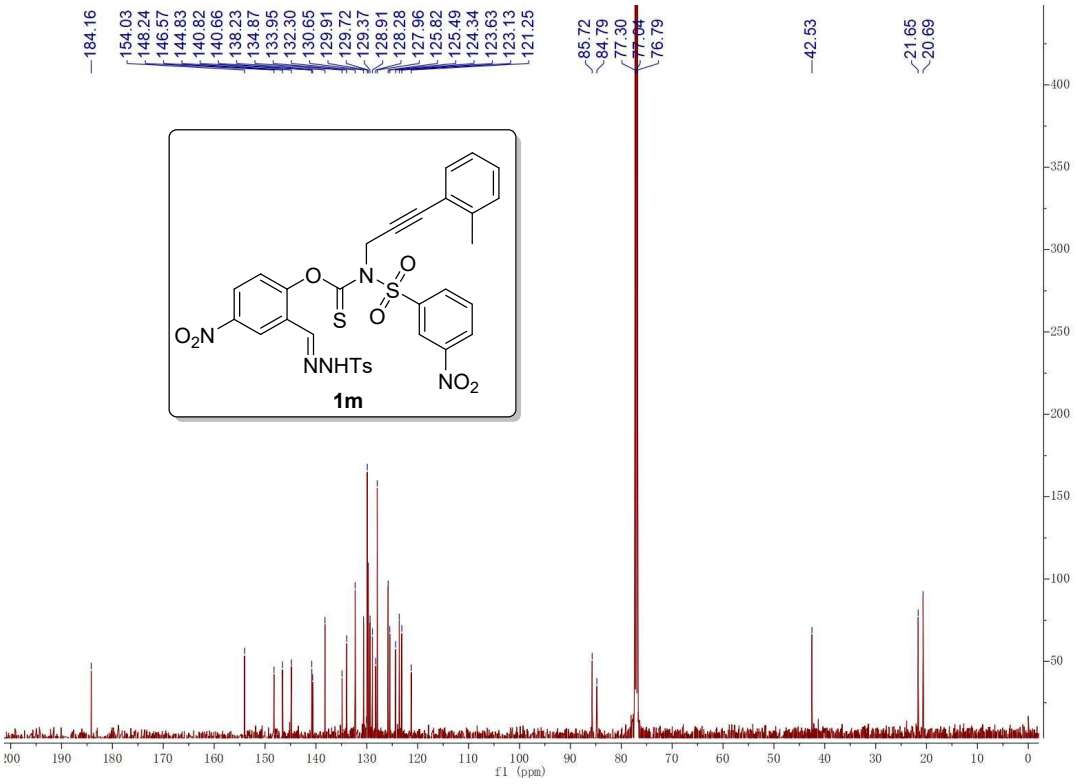

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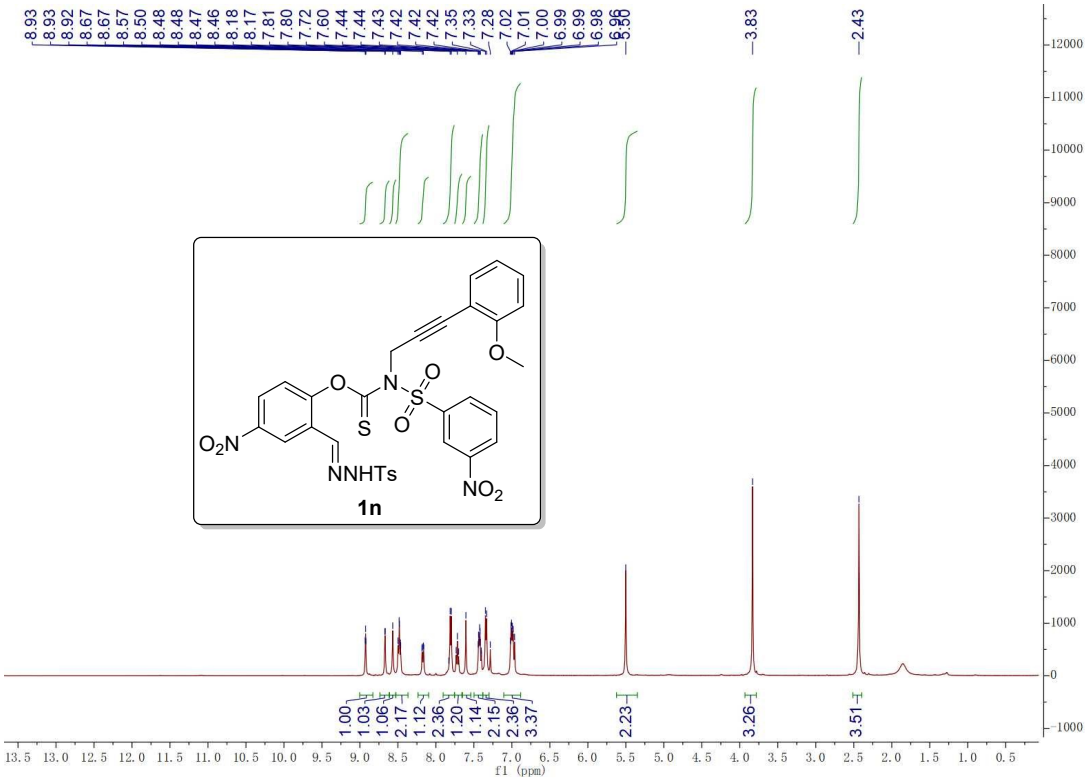

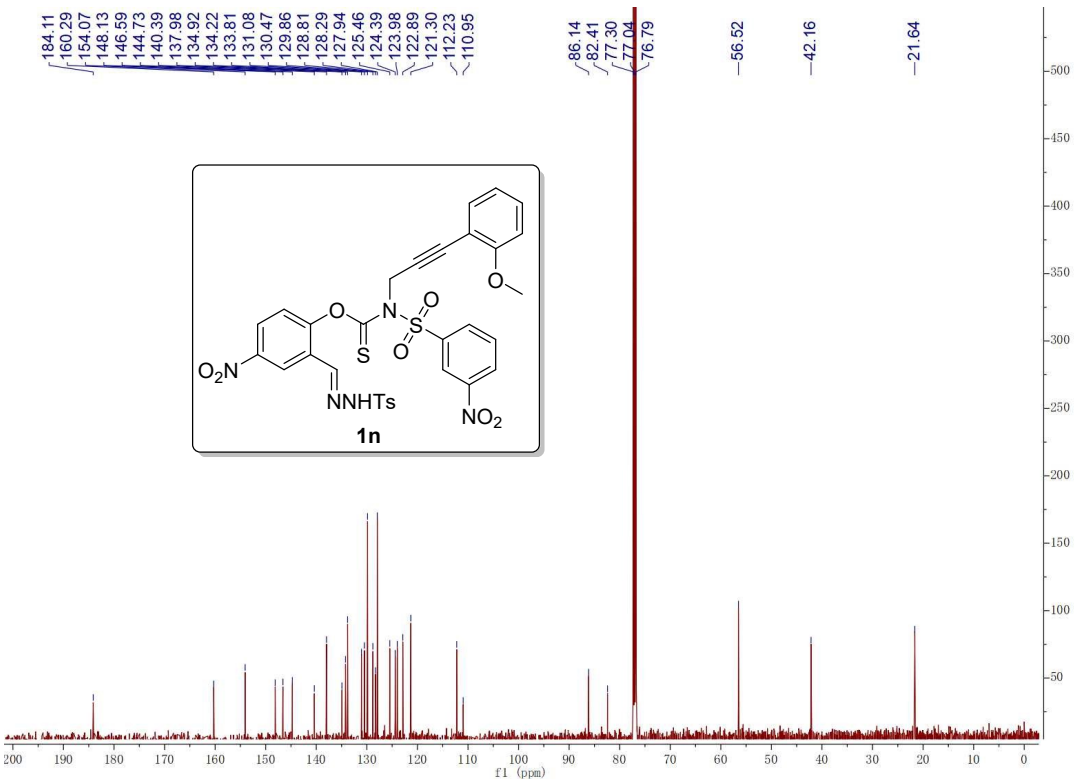

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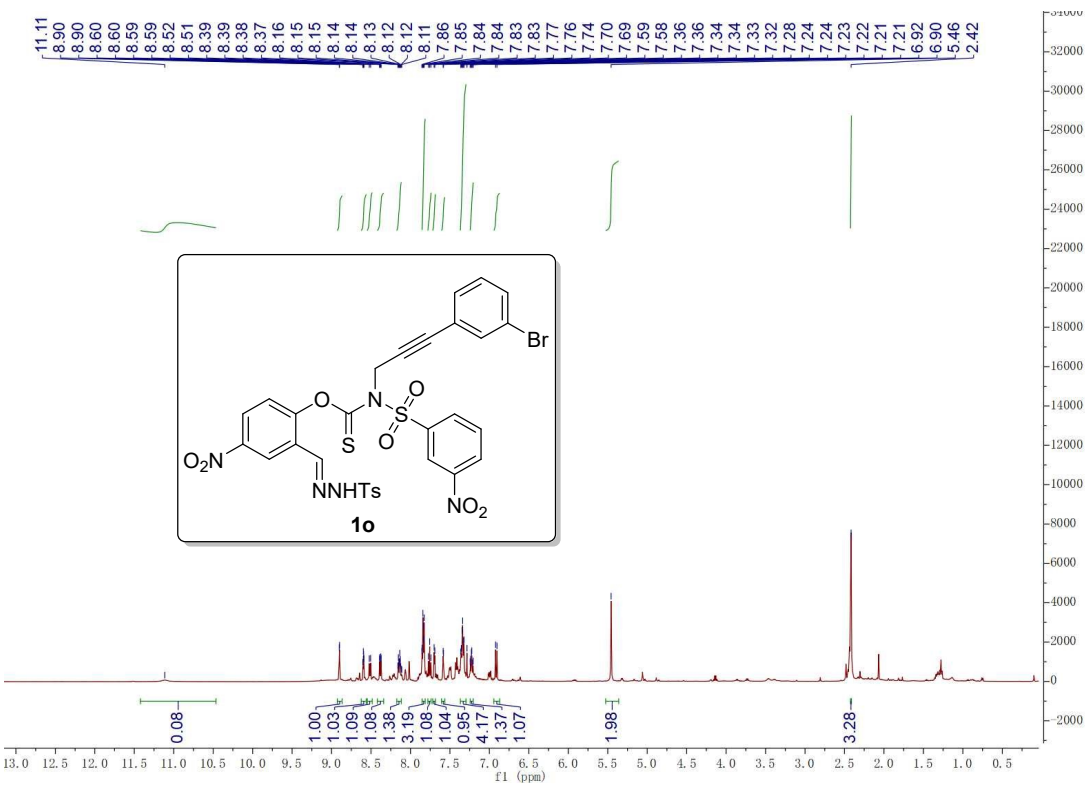

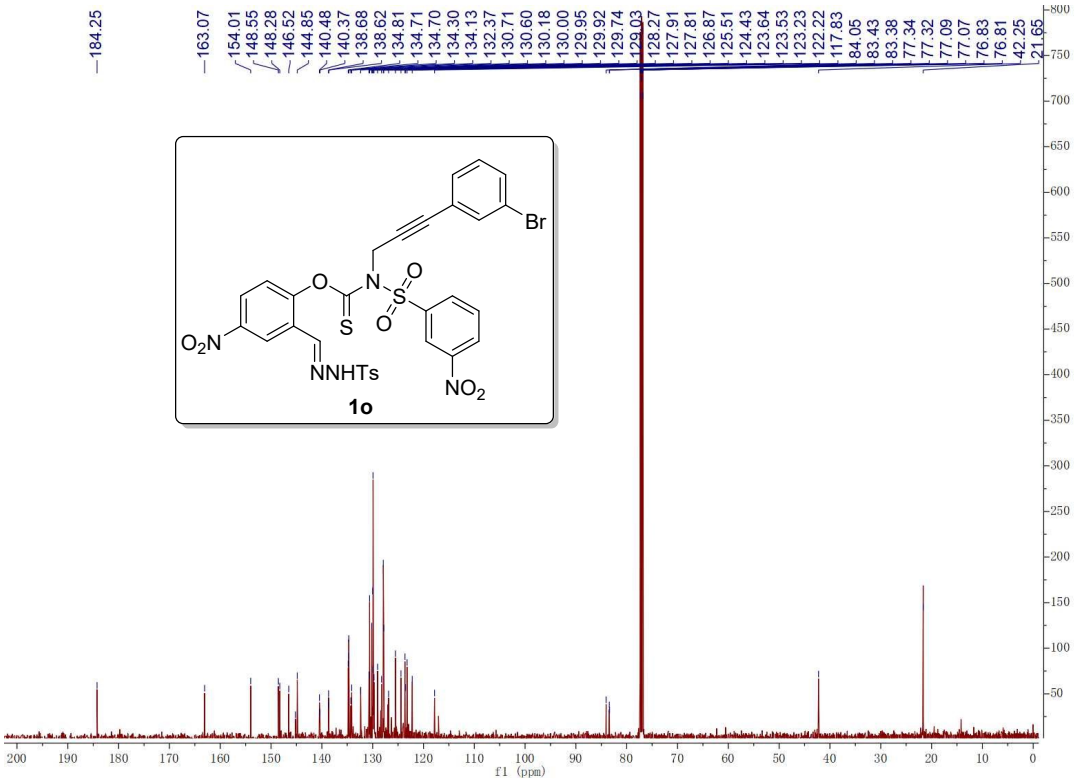

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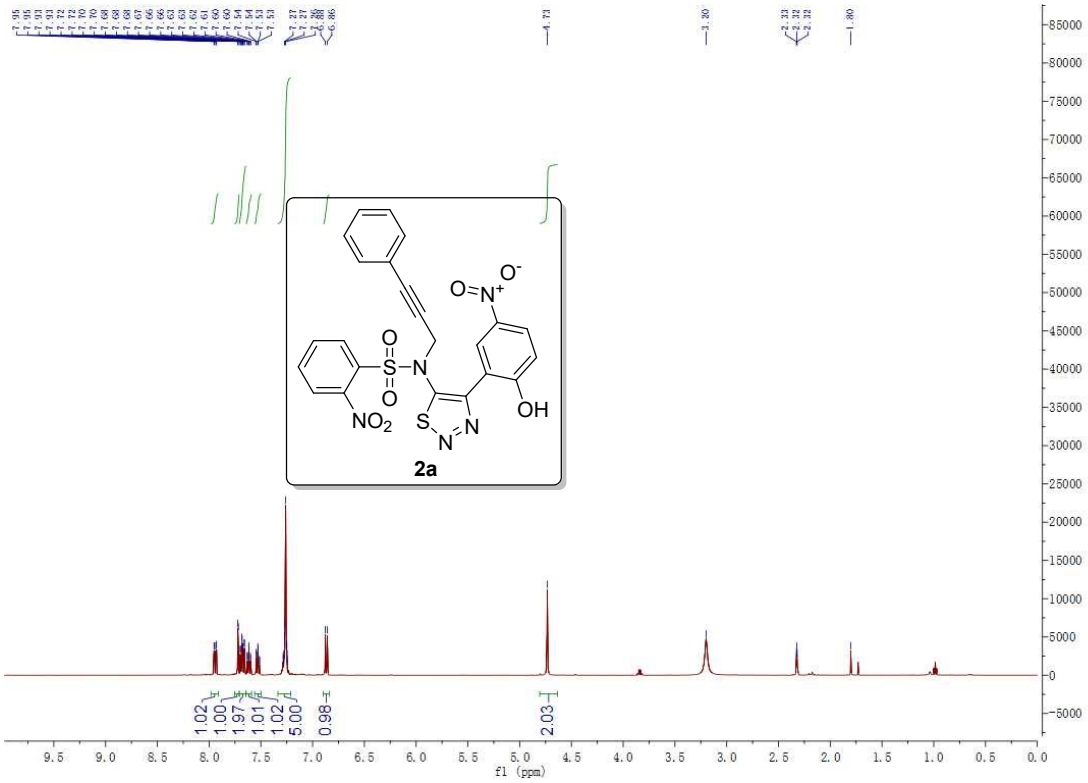

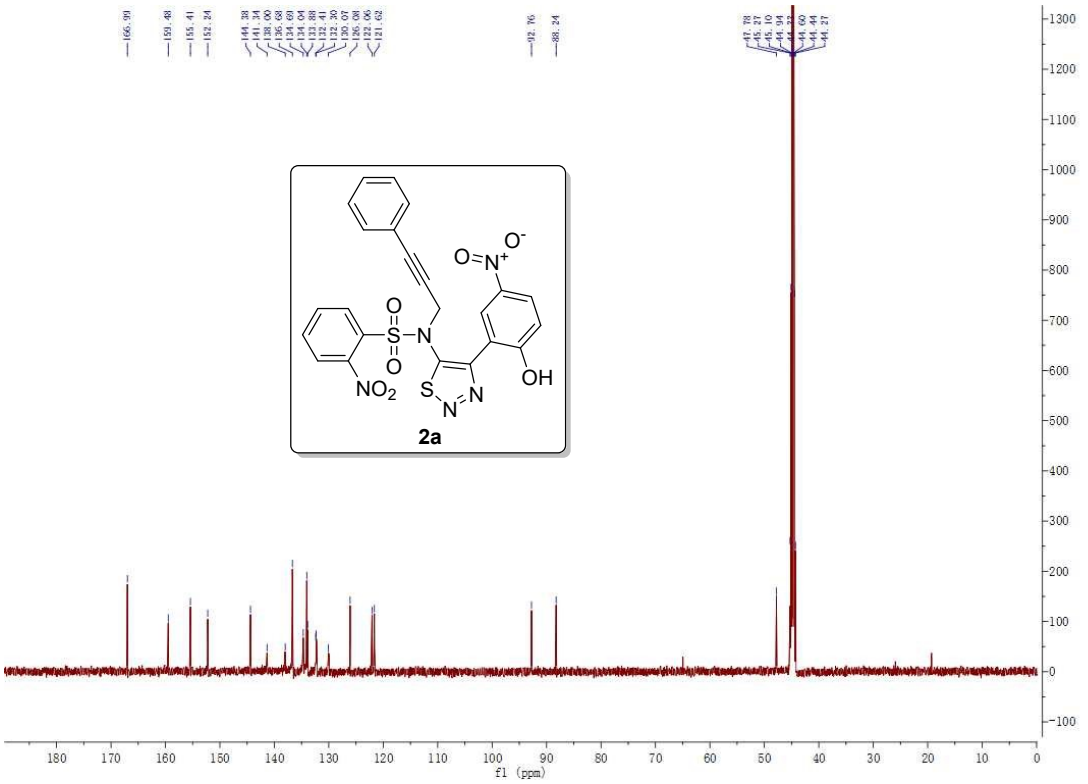

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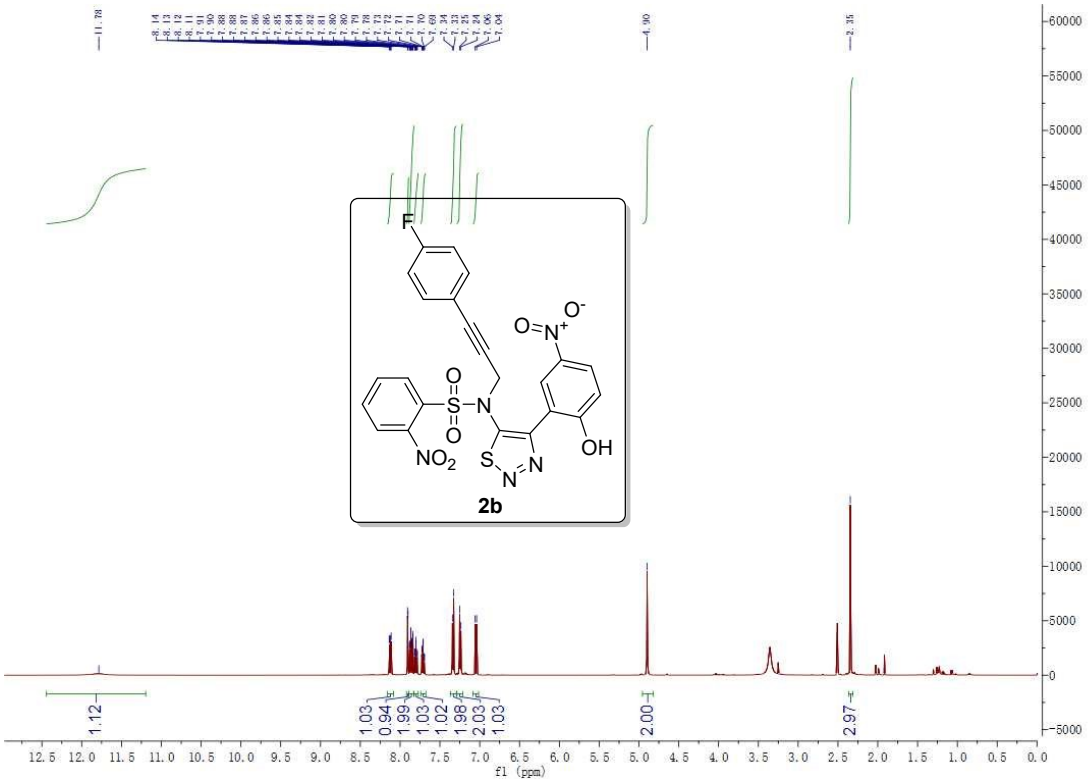
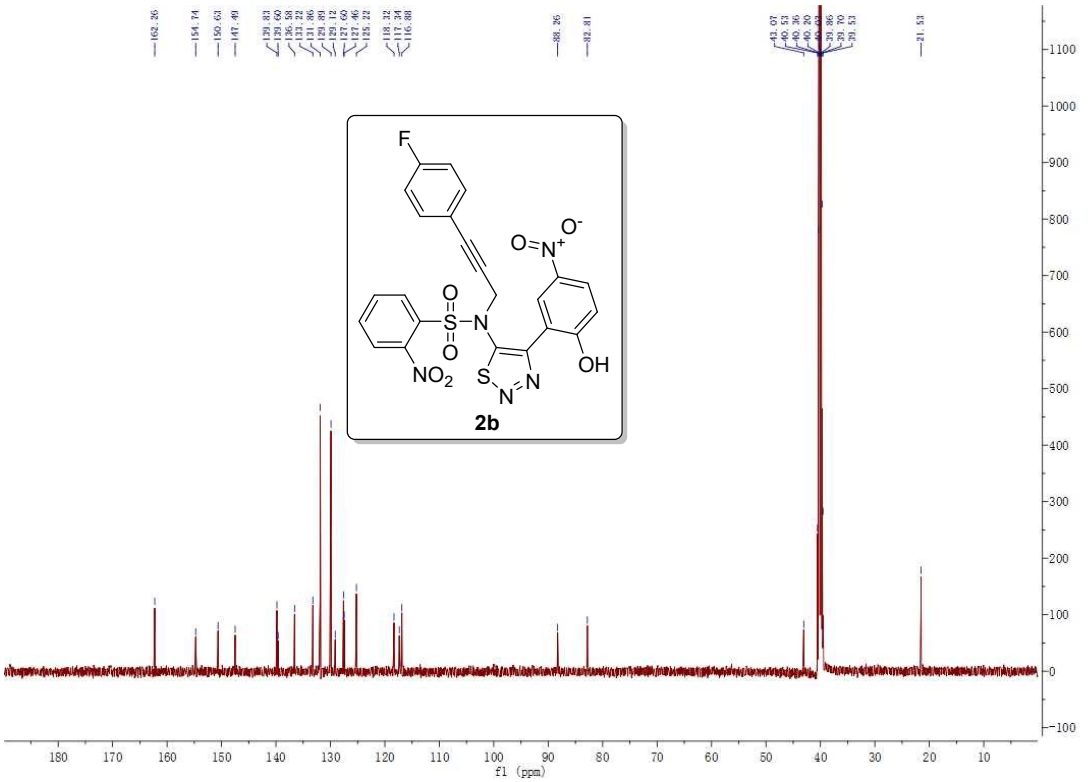
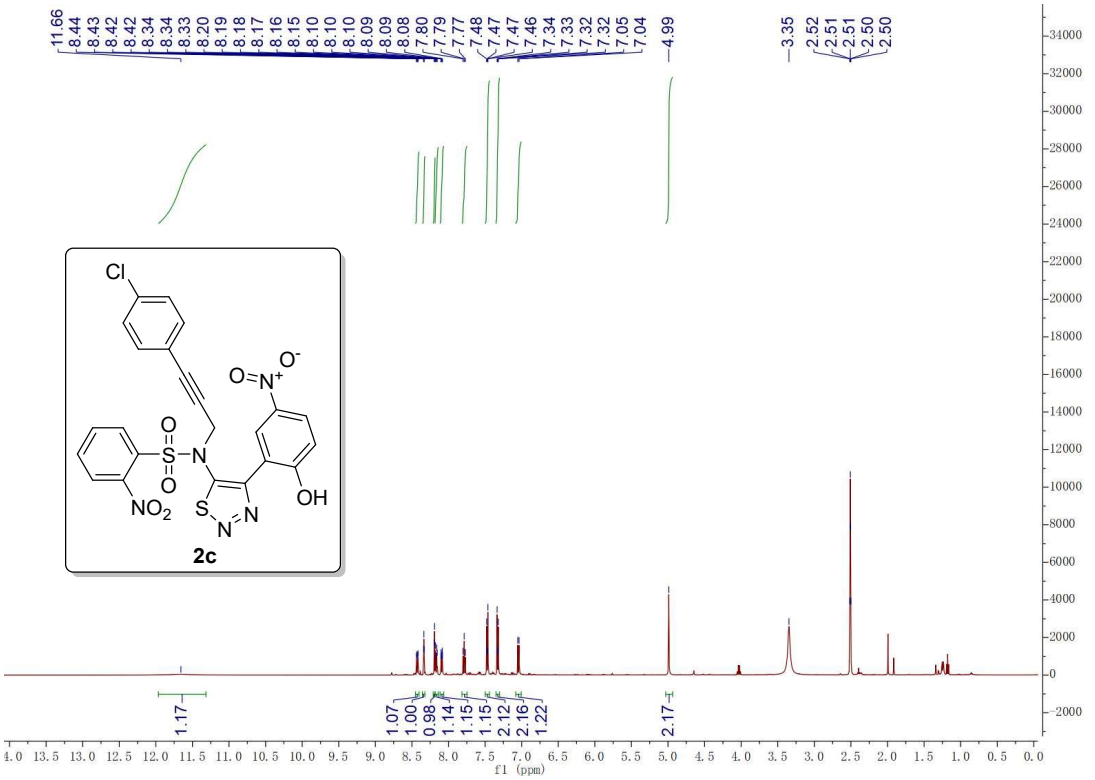
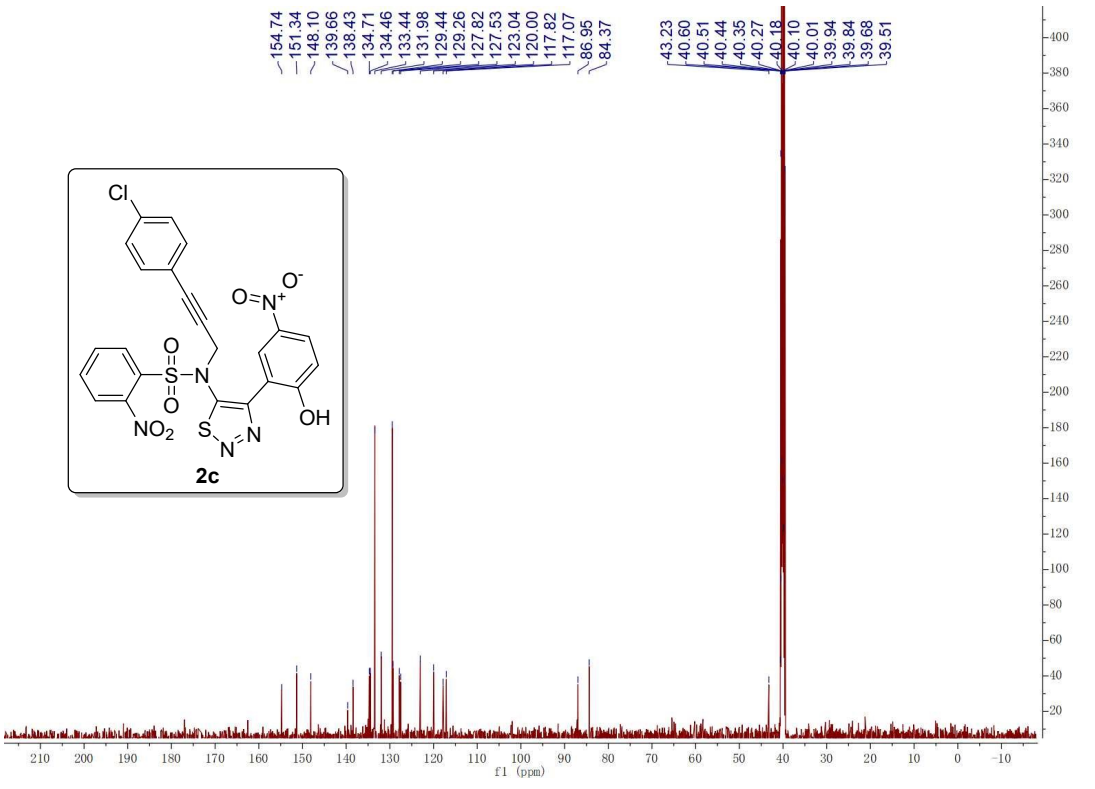
2-nitrobenzenesulfonamide (2b)



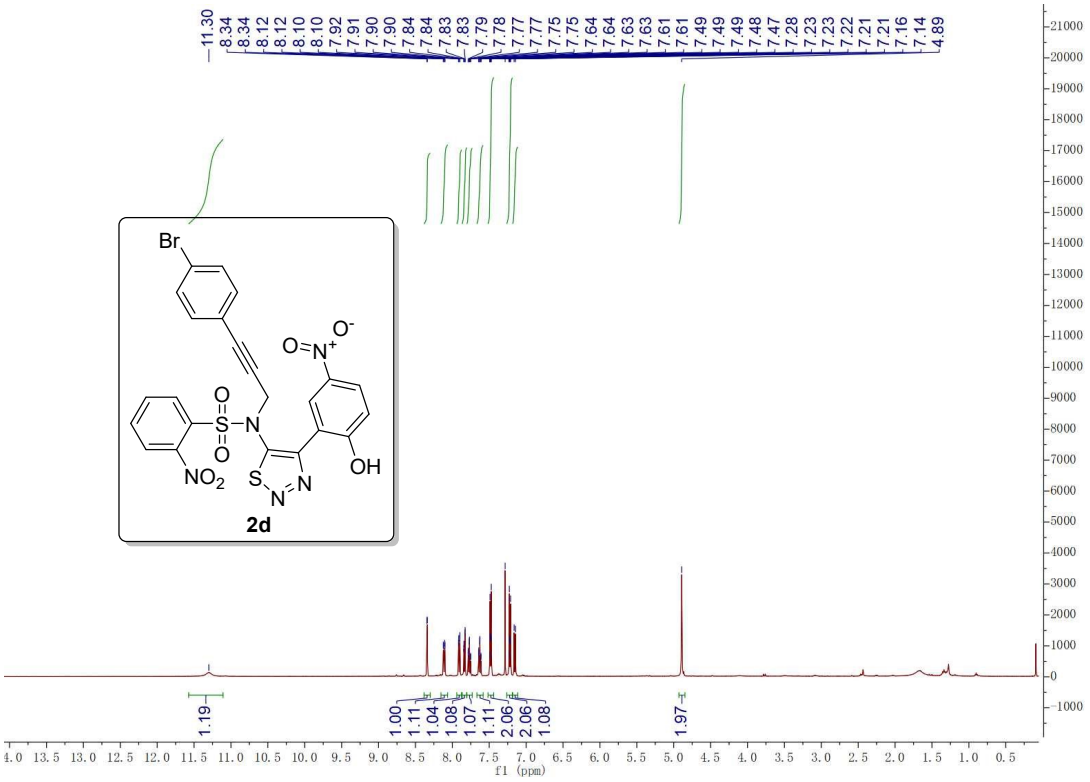


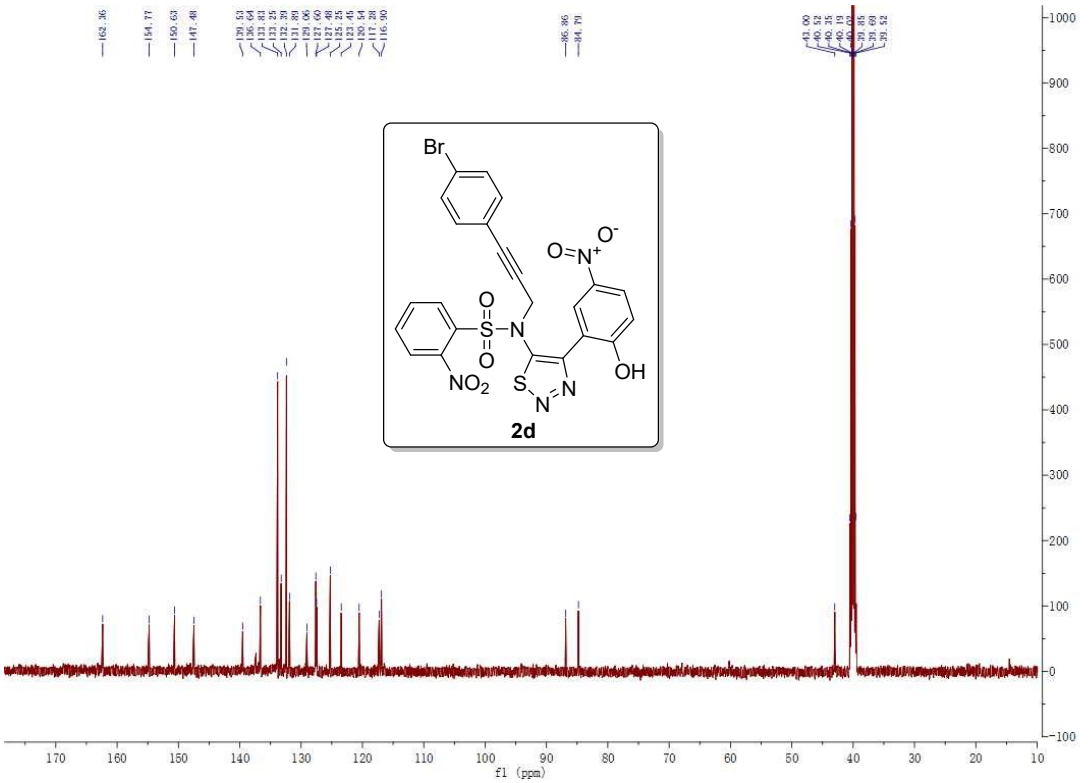
N-(3-(4-chlorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2c)



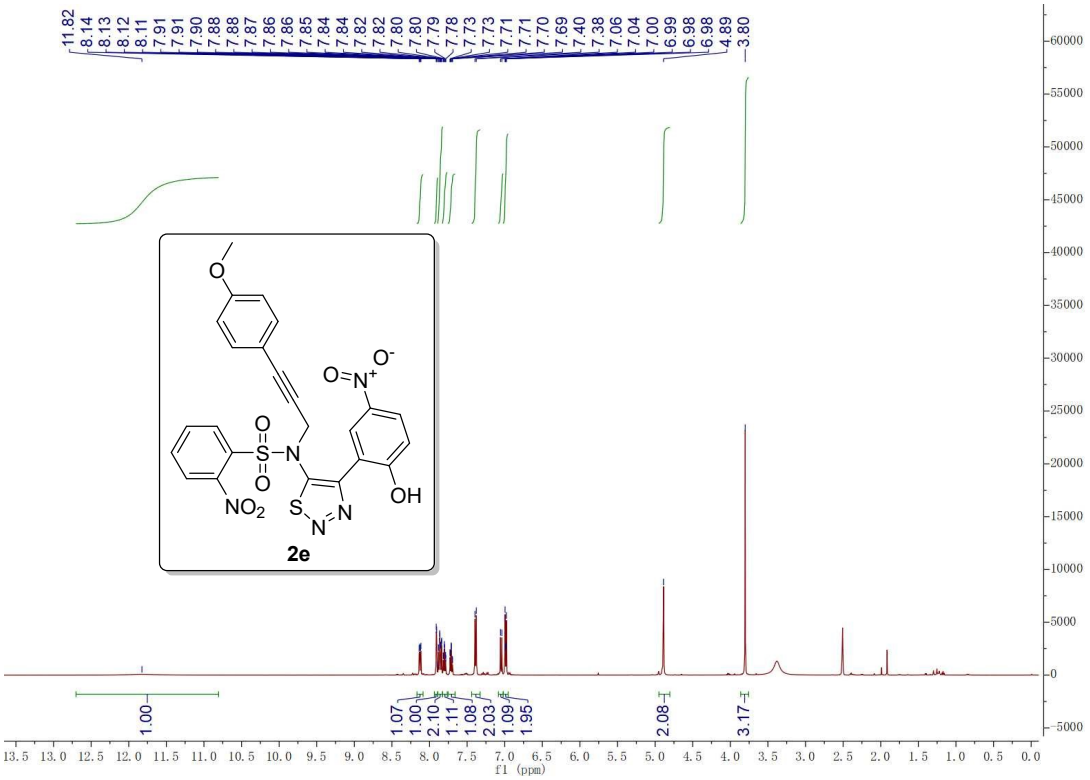


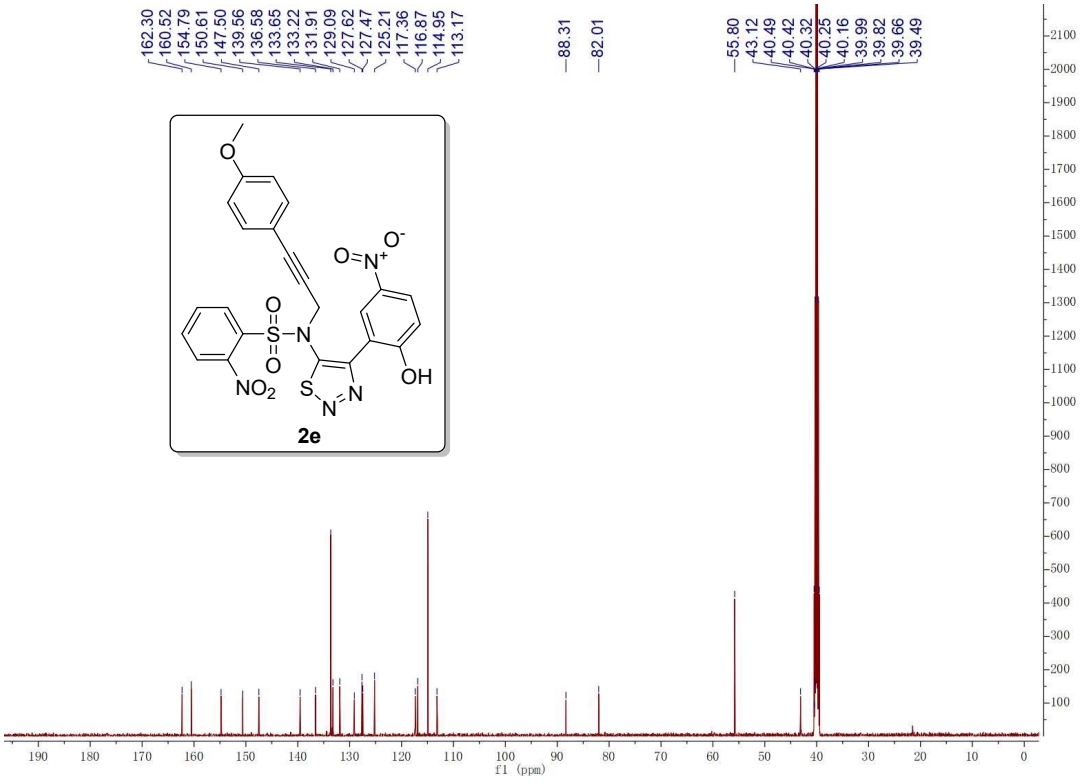
N-(3-(4-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2d)



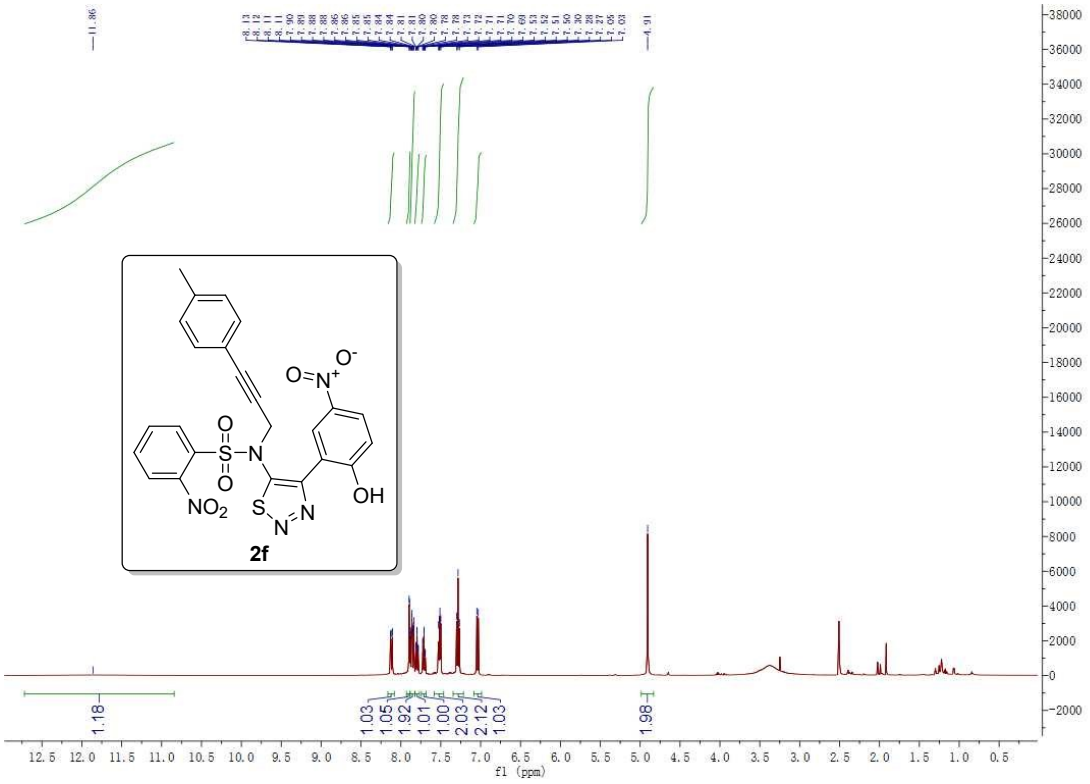


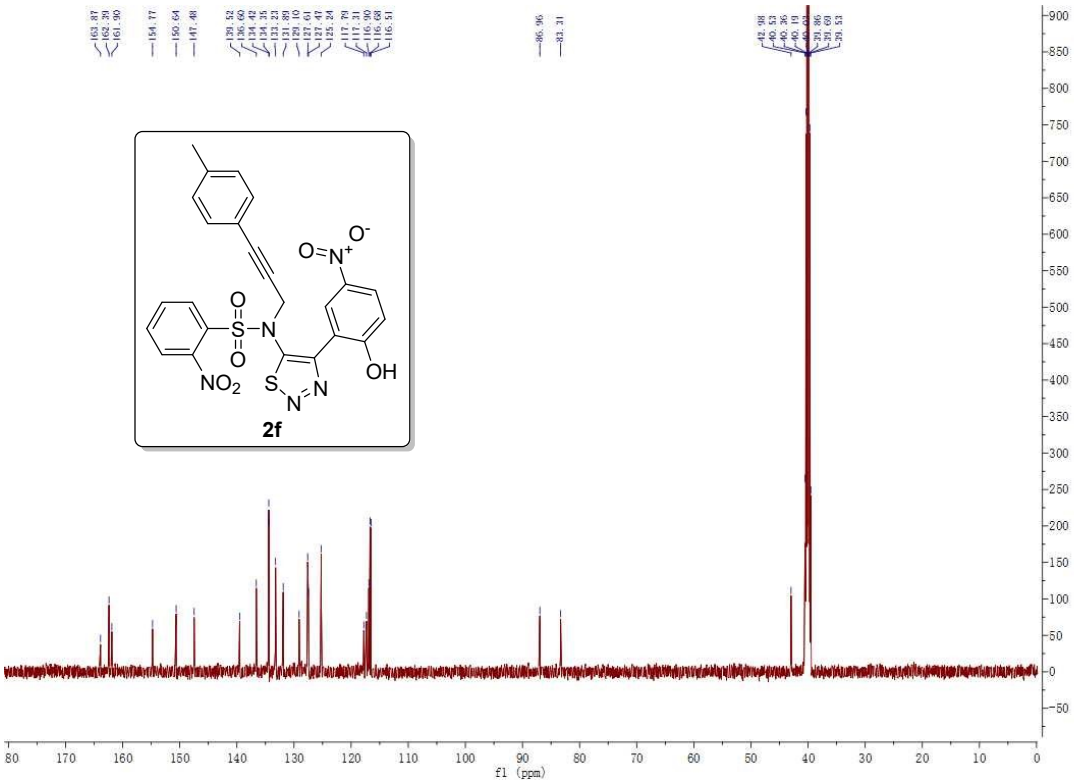
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-N-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-2-nitrobenzenesulfonamide (2e)



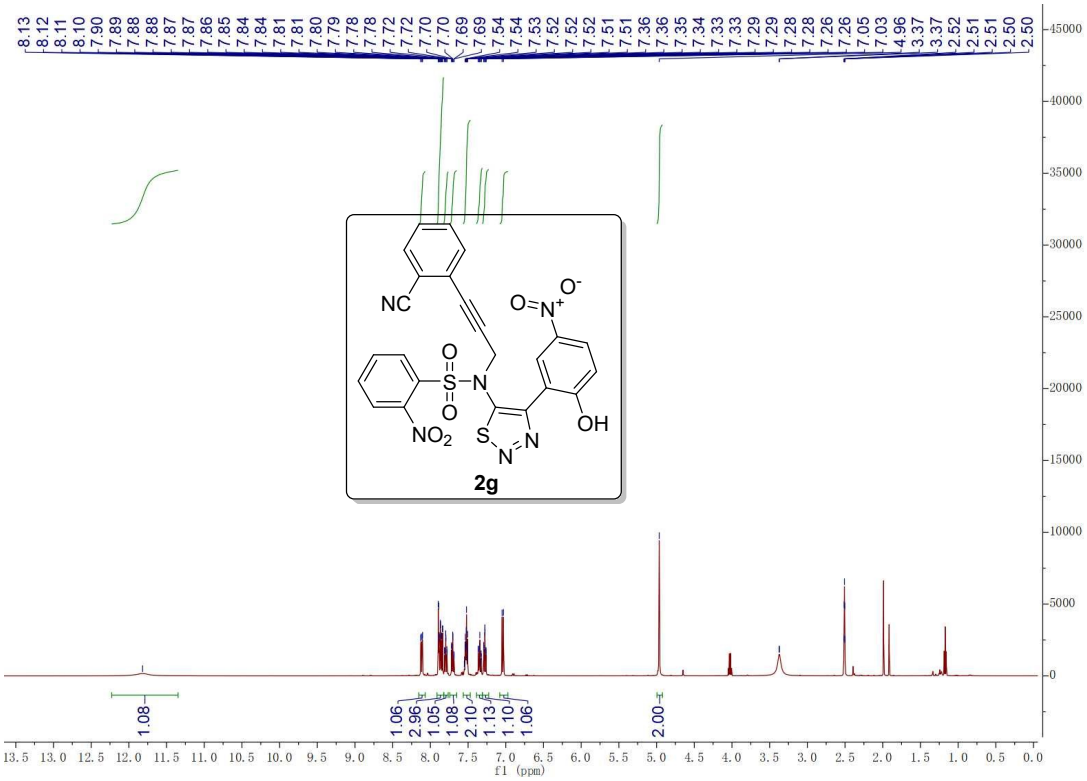


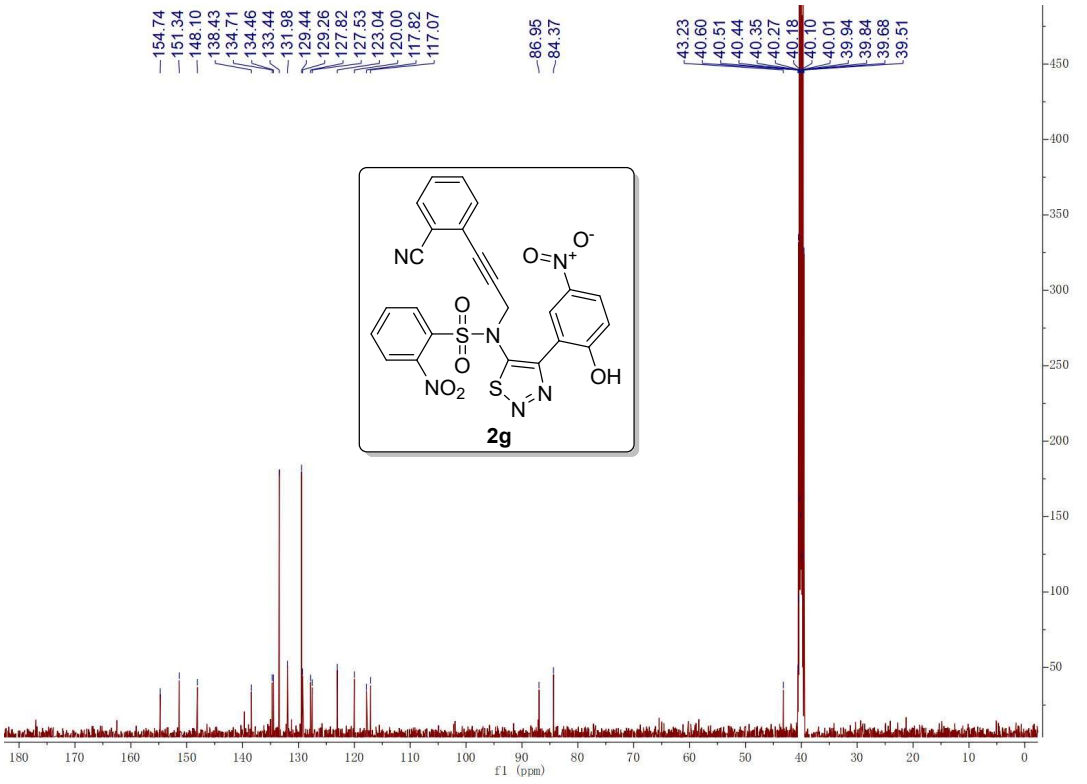
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitro-N-(3-(p-tolyl)prop-2-yn-1-yl)benzenesulfonamide (2f)



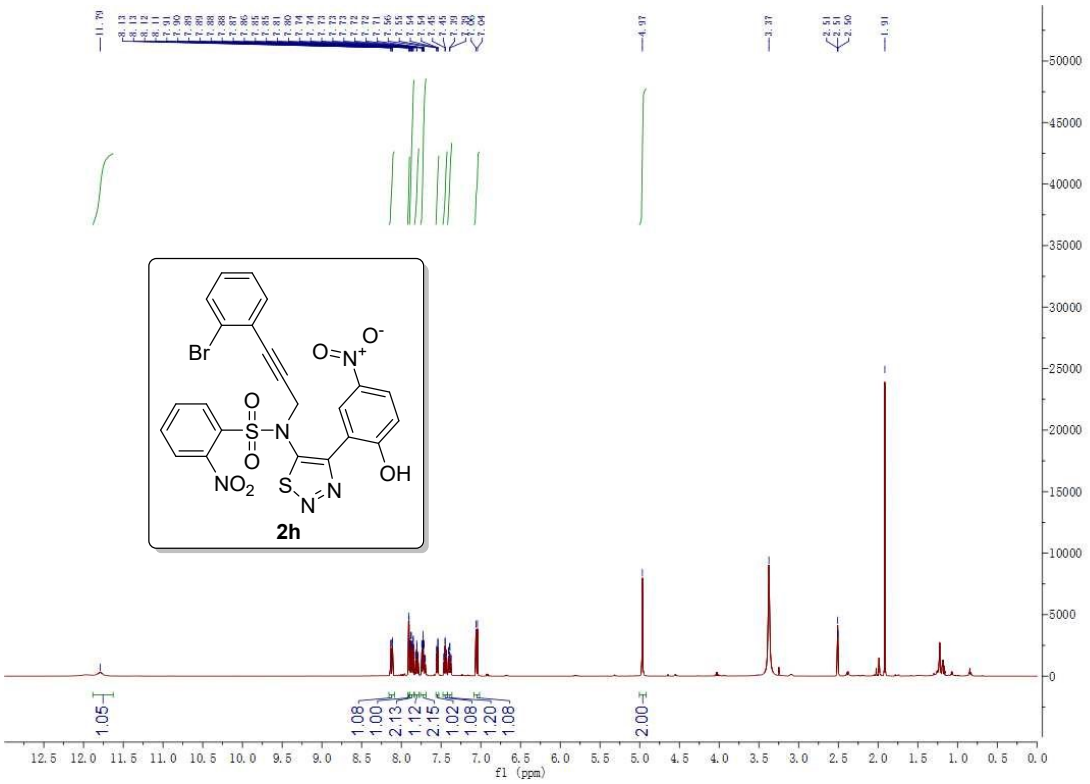


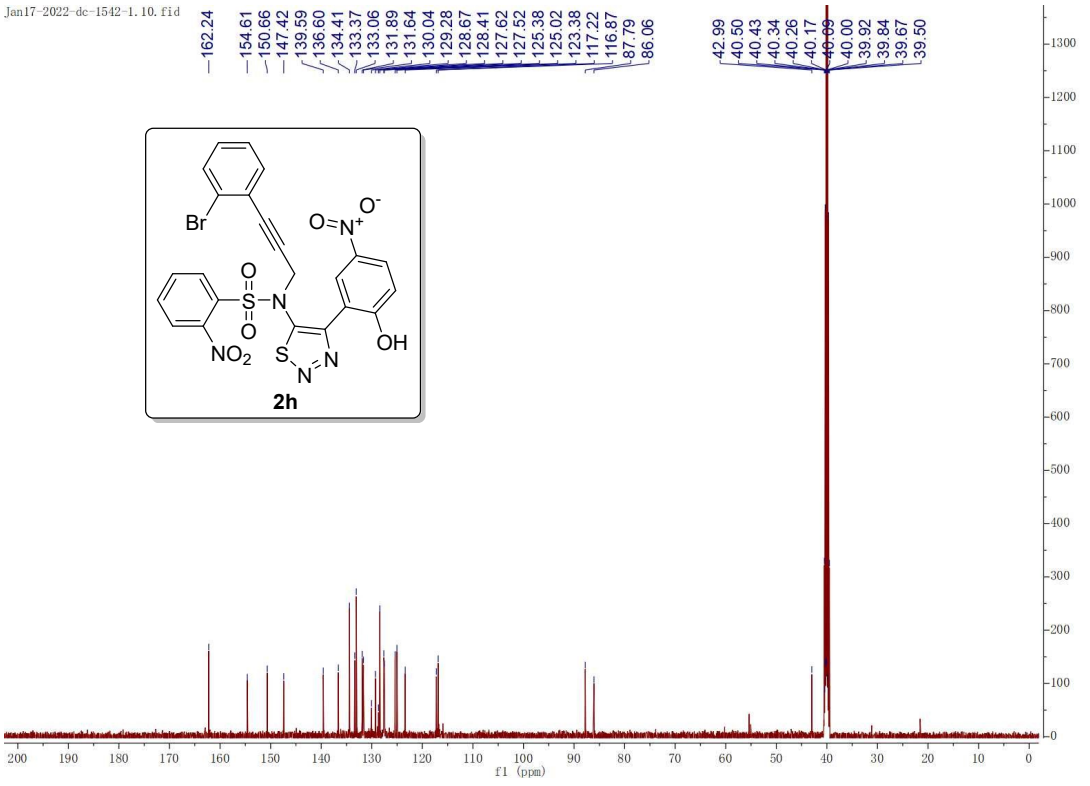
N-(3-(2-cyanophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2g)





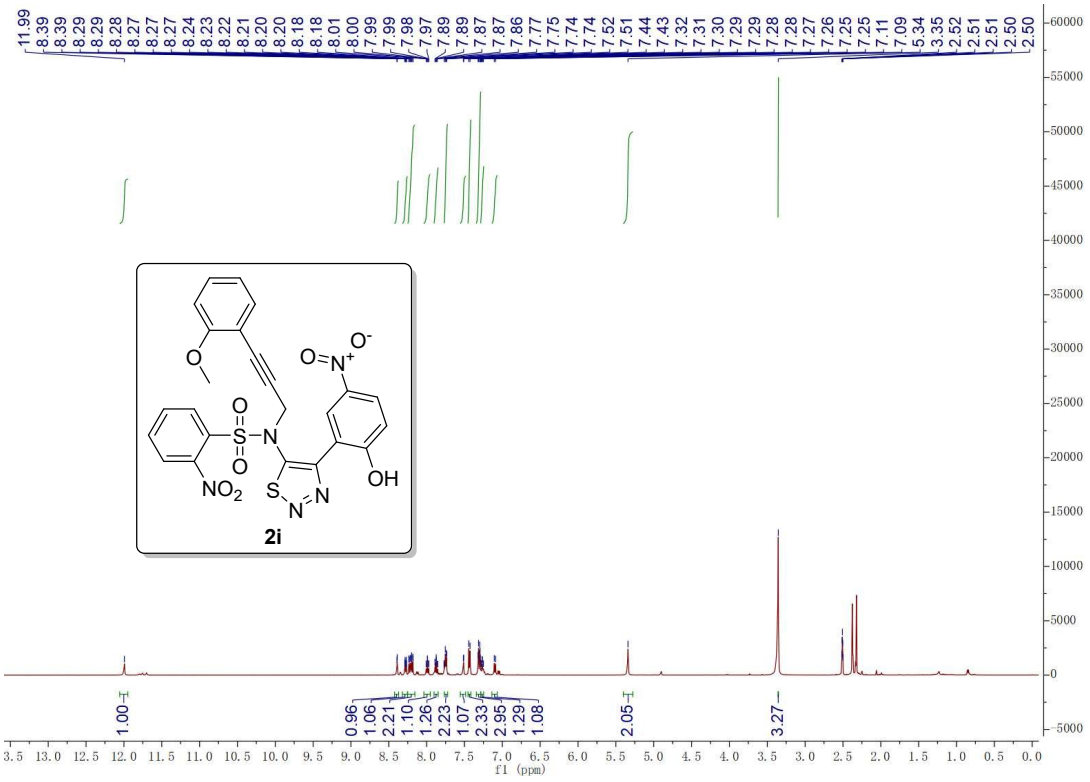
N-(3-(2-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-2-nitrobenzenesulfonamide (2h)

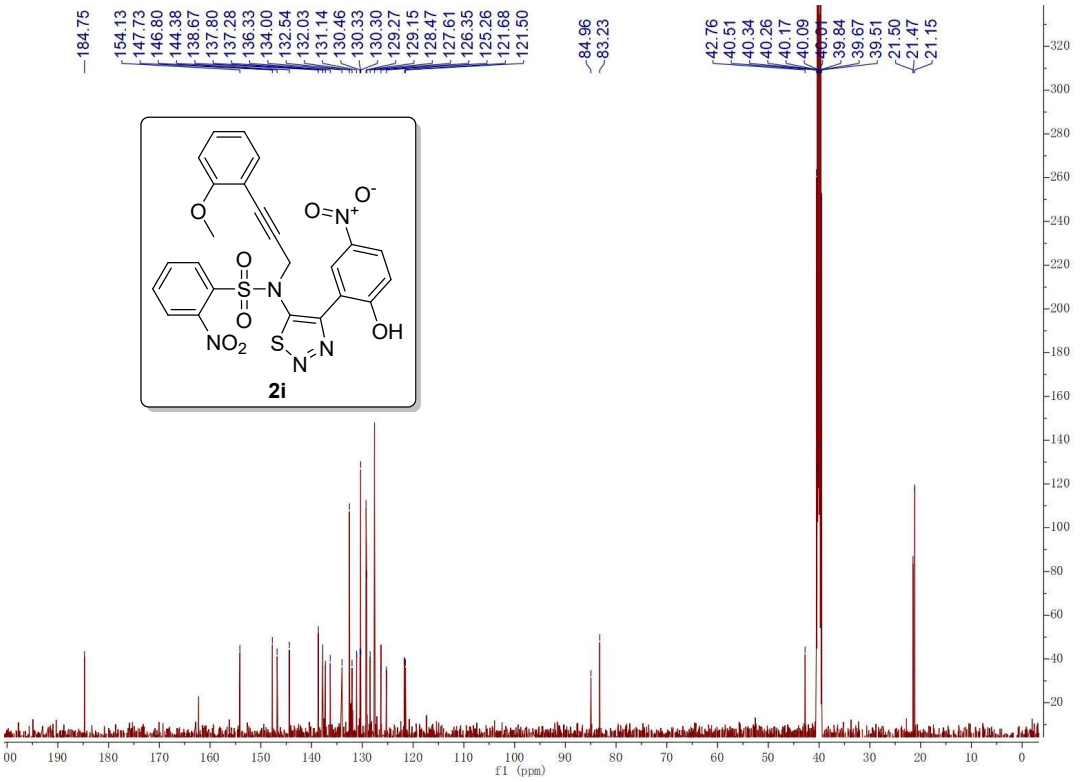




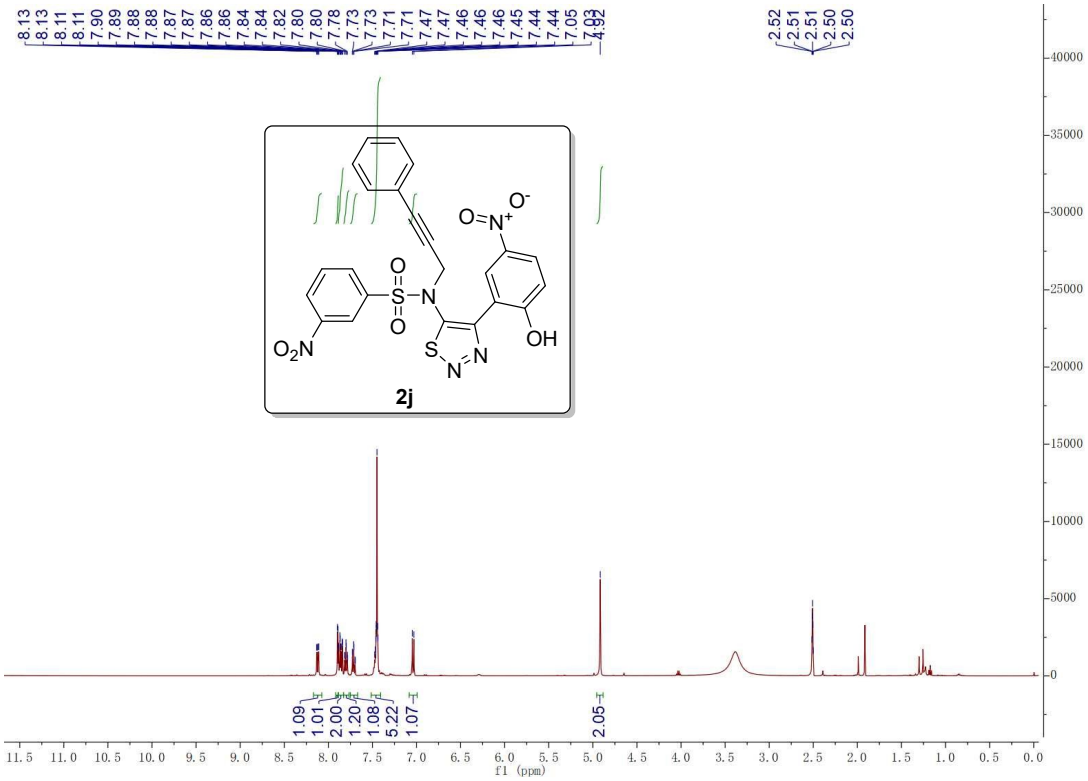
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-N-(3-(2-methoxyphenyl)prop-2-yn-1-yl)-

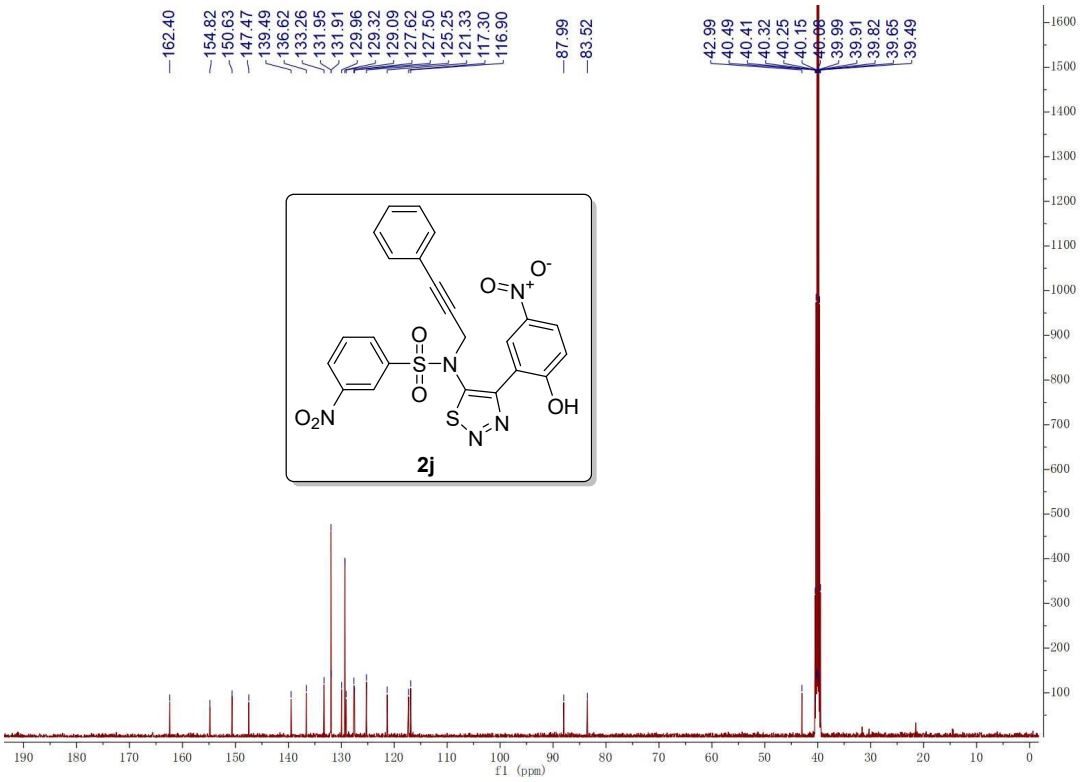
2-nitrobenzenesulfonamide (2i)



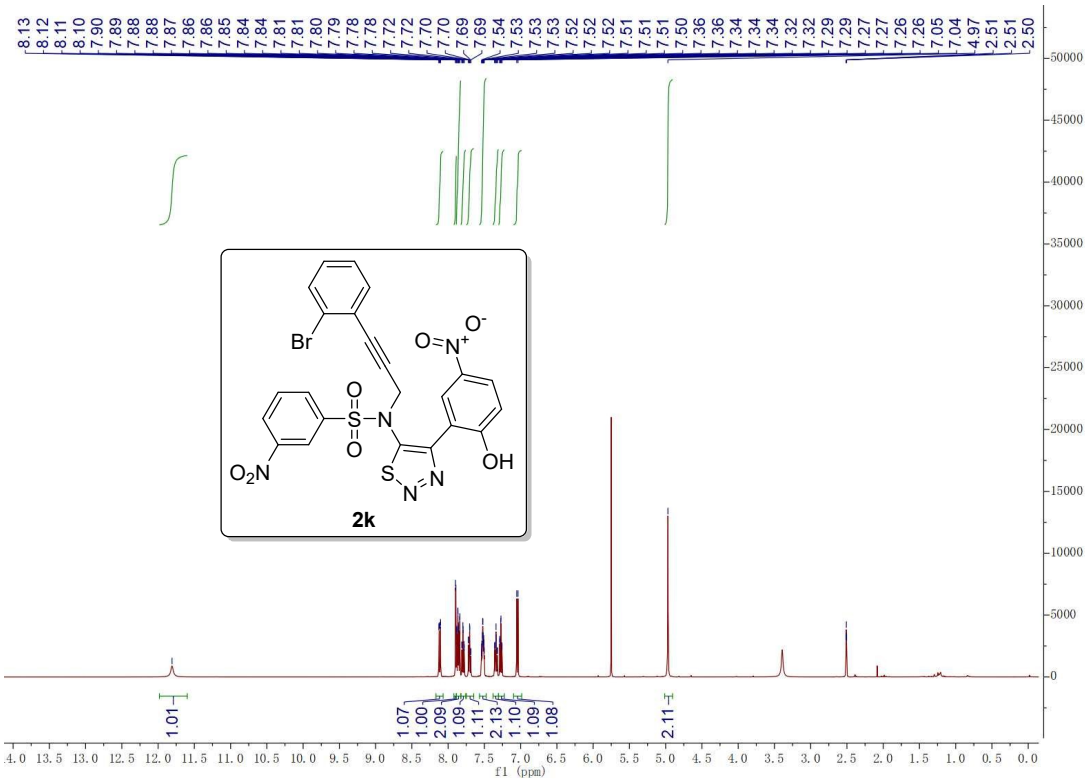


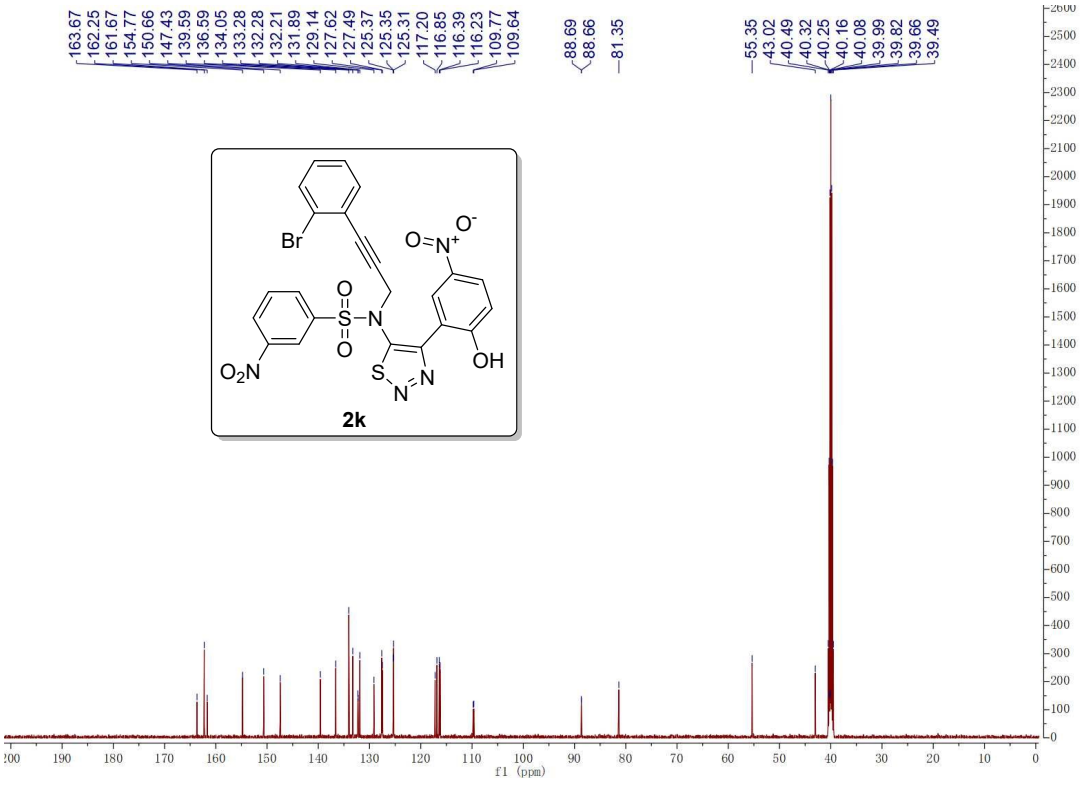
N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitro-N-(3-phenylprop-2-yn-1-yl)benzenesulfonamide (2j)



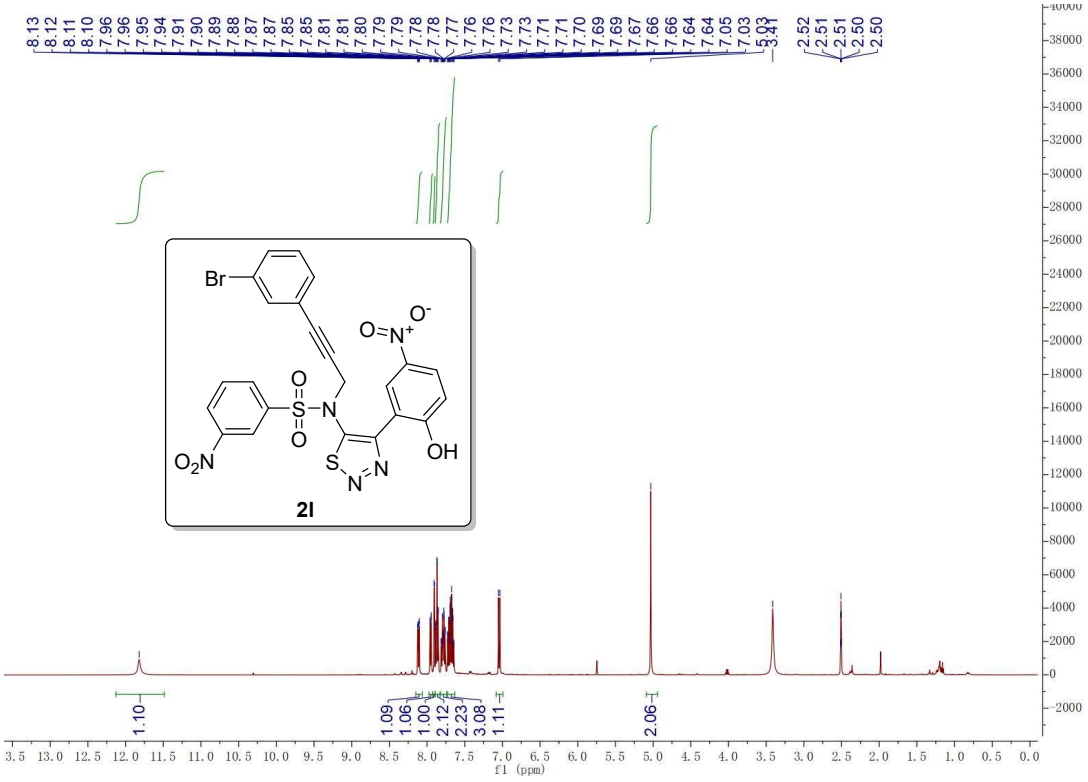


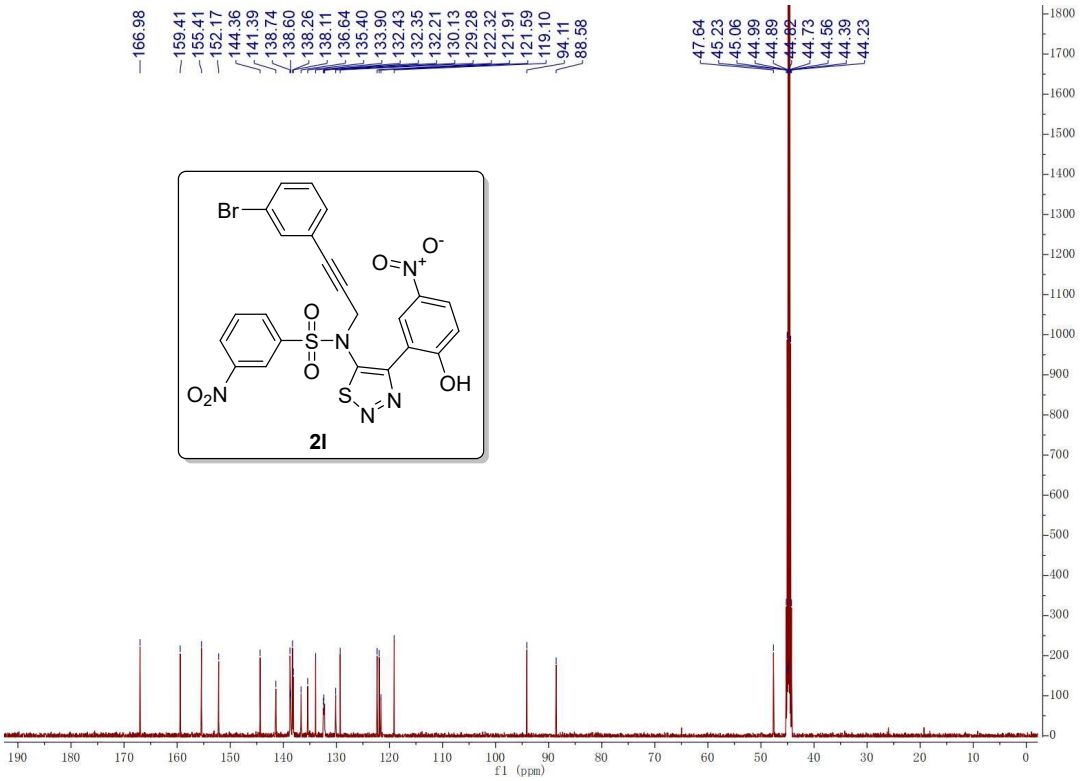
N-(3-(2-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2k)



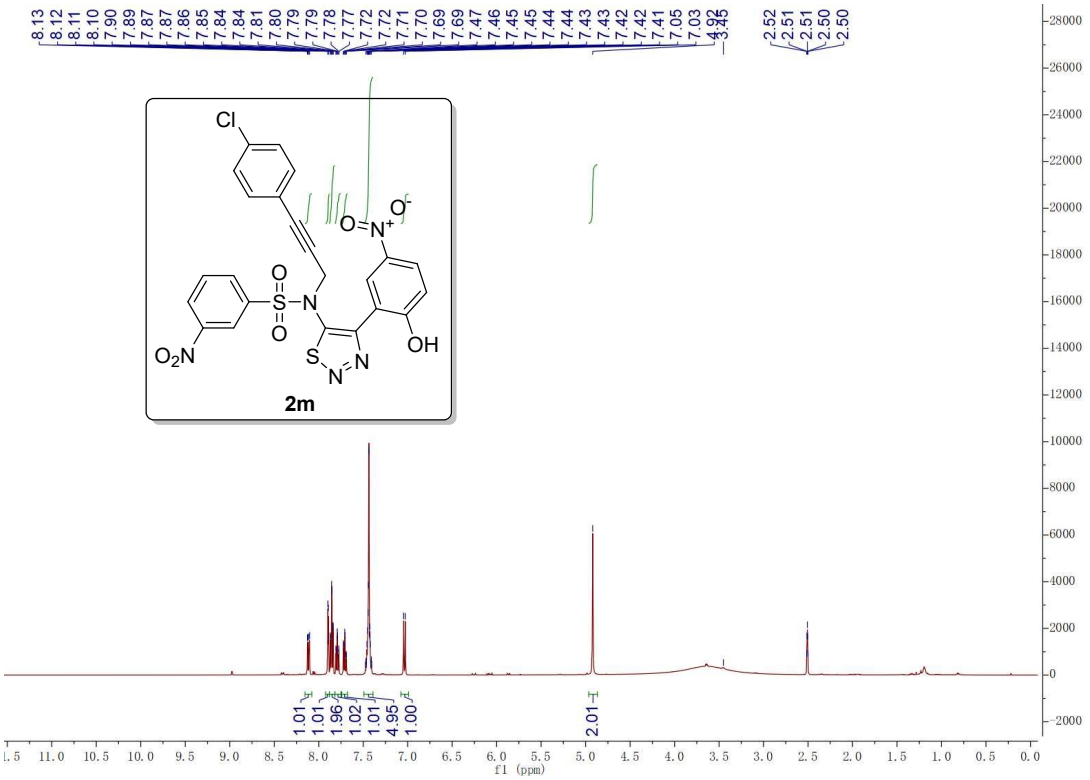


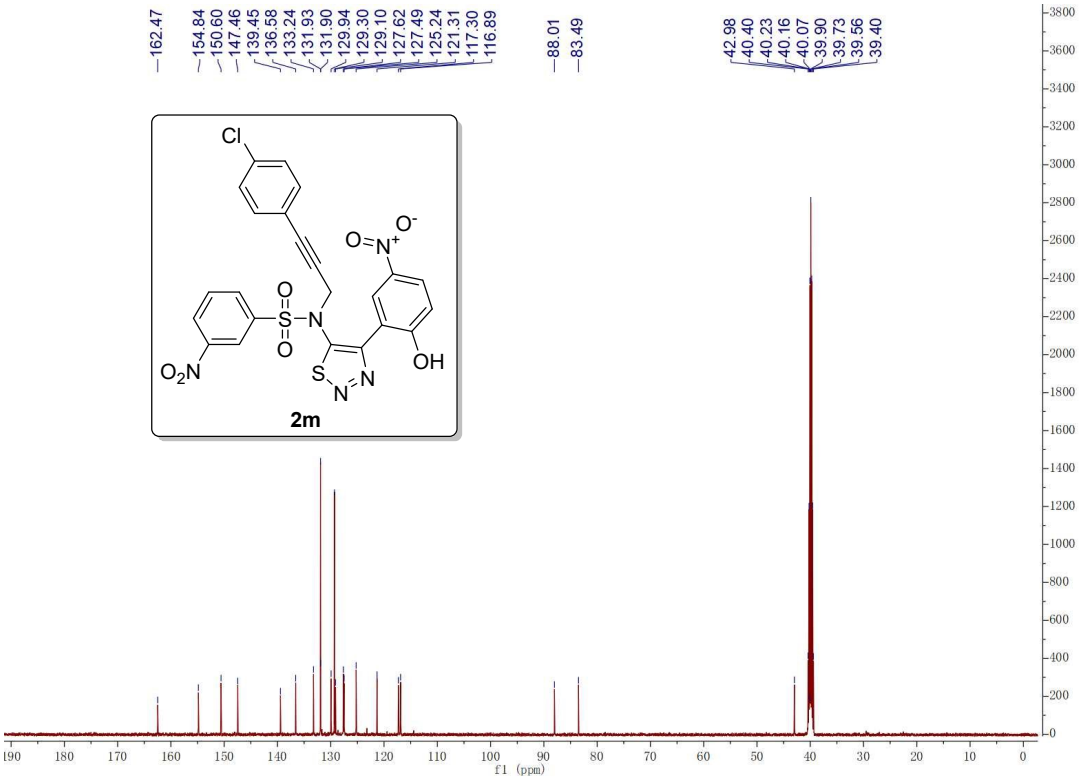
N-(3-(3-bromophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2l)



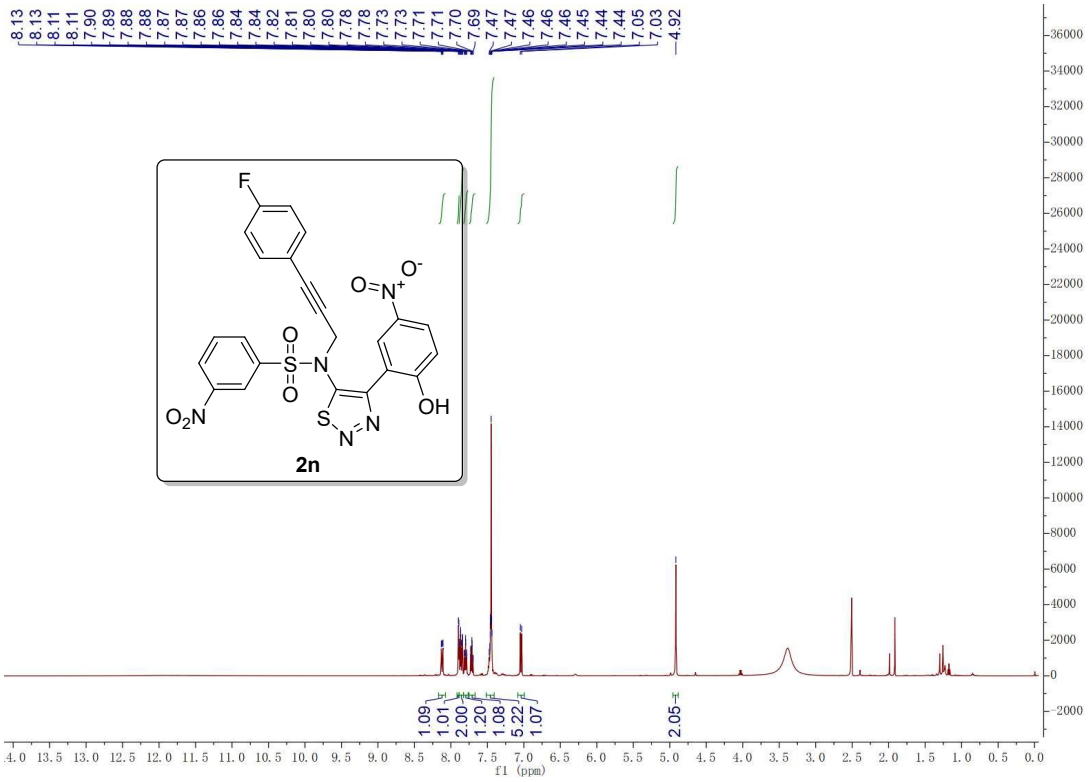


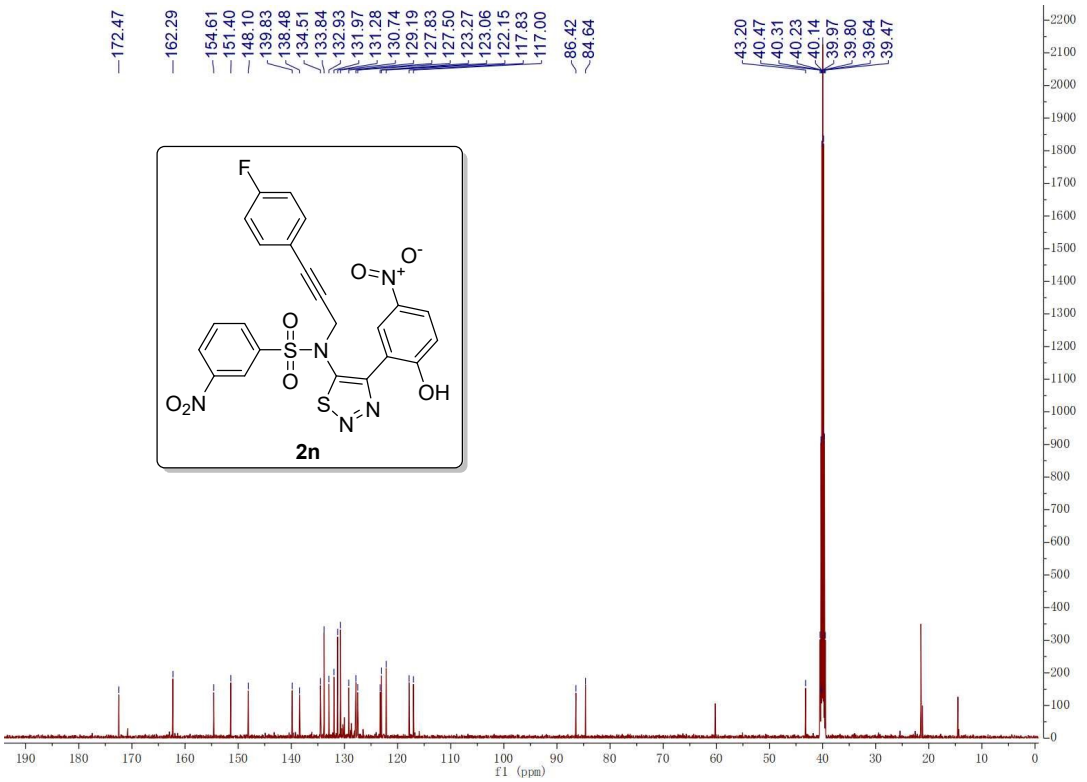
N-(3-(4-chlorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2m)



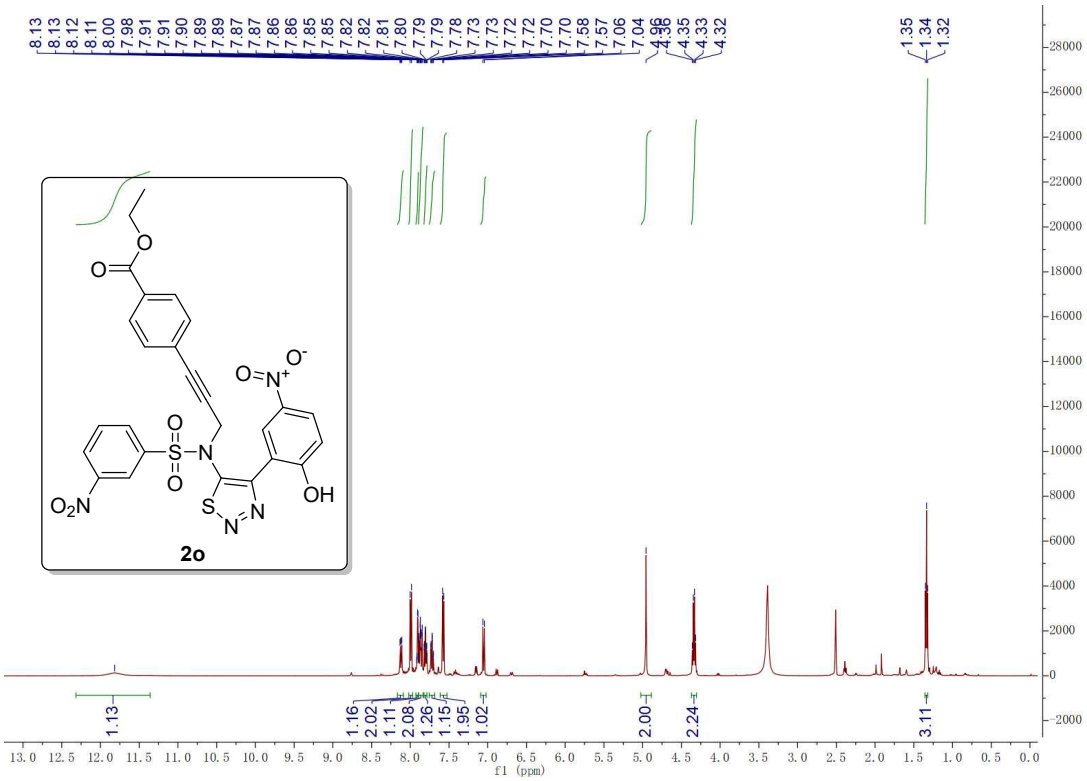


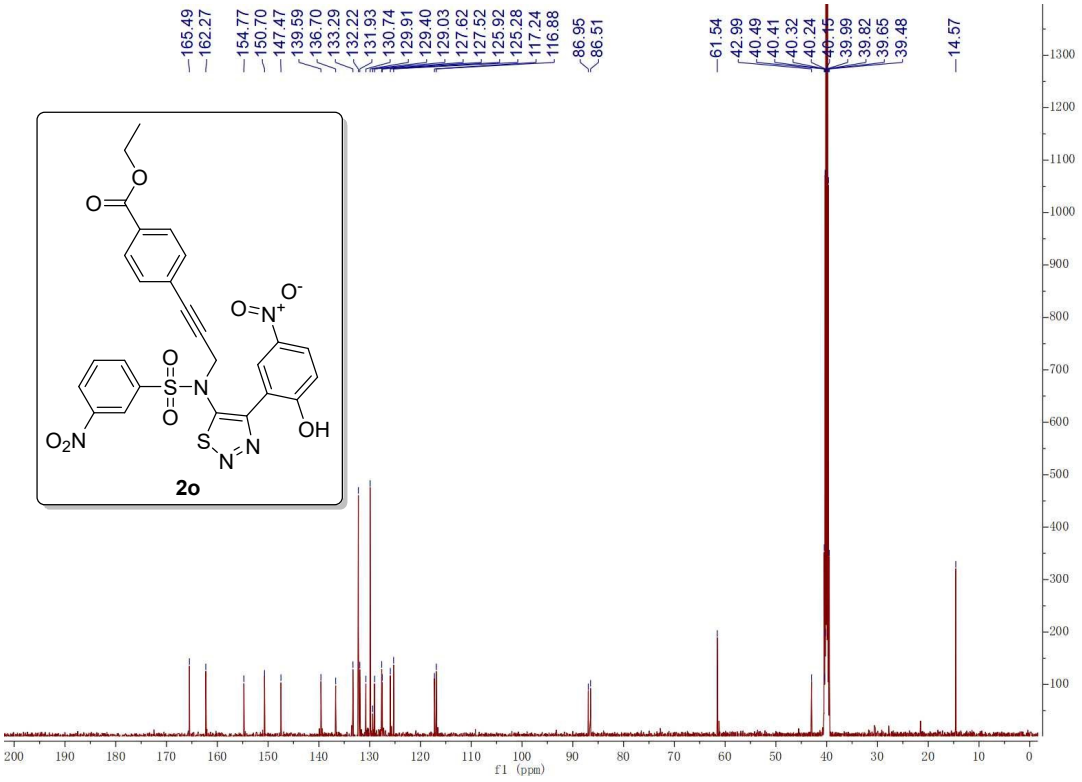
N-(3-(4-fluorophenyl)prop-2-yn-1-yl)-N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrobenzenesulfonamide (2n)





Ethyl 4-(3-((N-(4-(2-hydroxy-5-nitrophenyl)-1,2,3-thiadiazol-5-yl)-3-nitrophenyl)sulfonamido)prop-1-yn-1-yl)benzoate (2o)

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N
OH
2o
f1 (ppm)