Electronic Supplementary Information

Transformation of Arylbornoic Acids with Sodium Thiosulfate into Organodisulfides Catalyzed by Recyclable Polyoxometalate-based Cr (III) Catalyst

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[†] Electronic Supplementary Information (ESI) available: experimental conditions, supplementary table and NMR spectra. See DOI: 10.1039/x0xx00000x

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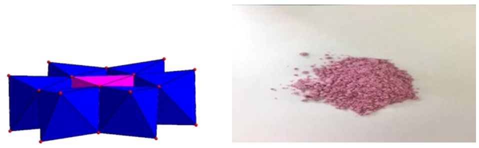
I. General information.

The catalyst was prepared according to published literature methods.[1] All reagents were purchased from Sigma-Aldrich and Adamas-beta, which were used without further purification. ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker AVANCE III 500 MHz (500 MHz for proton, 125MHz for carbon) spectrometer and Bruker AVANCE III 400MHz (400MHz for proton, 100MHz for carbon) with *tetra*-methylsilane as the internal reference using $CDCl_3$ or DMSO-d_6 as solvent in all cases, and chemical shifts were reported in parts per million (ppm, δ). FT-IR spectra were recorded on a Thermo Fisher Nicolet 6700. ESI-MS was performed on Analysis Center of Shanghai Institute of Technology. GC mass spectra were recorded on Shimadzu GCMS-QP2010 with RTX-5MS column (0.25 mm× 30 m). Column chromatography was performed using 300-400 mesh silica gel.

II. Synthesis and characterizations of catalyst.

$[NH_4]_3[CrMo_6O_{18}(OH)_6]$•$7H_2O$ was synthesized according to a published procedure with some modification. Firstly, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (5.0 g, 4 mmol) was dissolved in water (80 mL) and put in an oil bath under stirring. Then, $Cr(NO_3)_3\cdot 9H_2O$(2.3 g, 5.75 mmol) was dissolved in 20 mL water and added dropwise into the above solution, which needs to control the pH within 4 to 6.5 during this process. After the dropwise was completed, the mixed solution is further stirred at a constant temperature of 80 ^{o}C for 1 h. Following by, the obtained red wine-like solution was filtered immediately while it was still hot. After the solution was left at room temperature for 12 h, the purple crystals precipitated. While recrystallized, filtered and vacuum dried, the pink crystals (4.9 g) was collected (Figure S1). FTIR: 3202.28 (vas NH, m), 1649.16 (δ OH m), 1399.09 (δ NH, s), 936.78(ν Mo=O, vs), 881.87 (ν Mo=O, vs), 650.35 (ν Mo-O-Mo, vs), 576.14 (ν M-O-Mo, w) cm-1.



1: $(NH_4)_3[CrMo_6O_{18}(OH)_6]\cdot 7H_2O$

Figure S1. Appearance of $[NH_4]_3[CrMo_6O_{18}(OH)_6]$•$7H_2O$.

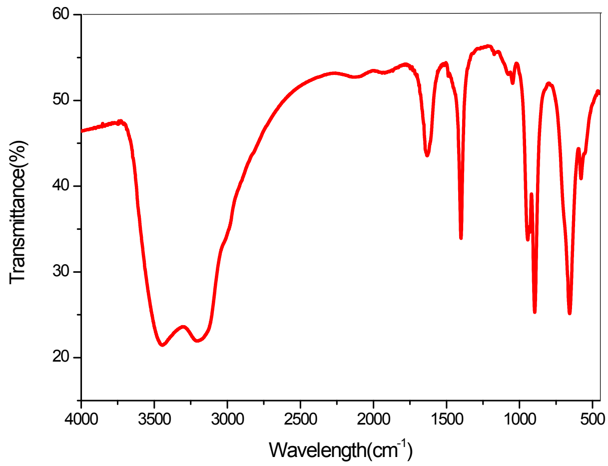
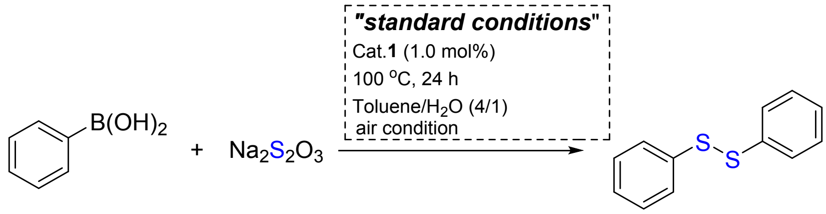


Figure S2. FT-IR spectra of $[NH_4]_3[CrMo_6O_{18}(OH)_6]$•$7H_2O$.

III. Reaction optimization.

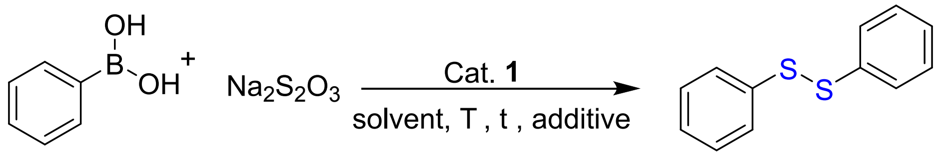
Table S1. Investigation the influence of hybrid metal center of Cat. **1** on the synthesis of symmetric disulfides[a]



Entry	Hybrid metal center	Conv. (%)[b]	Yield (%)[b]
1	CrPOM	99	99
2	NiPOM	79	64
3	FePOM	57	53
4	CuPOM	62	57

[a]Reaction conditions: Catalyst (1.0 mol%), phenylboronic (1.0 mmol), $Na_2S_2O_3$ (2.0 equiv), Toluene (1.6 mL), H_2O (0.4 mL) at 100 °C for 24 h. [b]Sustrate conversion and yield were determined by GC-Ms analysis.

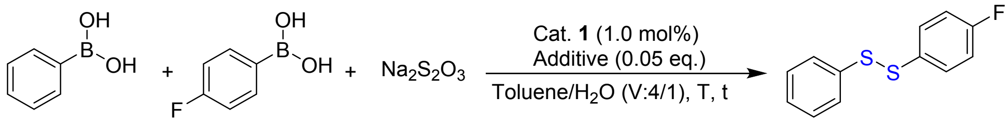
Table S2. Effect of solvent, temperature, time, additive on the synthesis of symmetric disulfides[a]



Entry	Catalyst (mol%)	T (°C)	Additive(0.05 equiv.)	Solvent (2.0 ml)	Yield (%)[f]
1	**1** (1.0)	60	K_3PO_4	Toluene/H_2O	22
2	**1** (1.0)	60	Na_2SO_3	Toluene/H_2O	34
3	**1** (1.0)	60	KF	Toluene/H_2O	11
4	**1** (1.0)	60	NaCl	Toluene/H_2O	27
5	**1** (1.0)	60	-	Toluene/H_2O	58
6	**1** (1.0)	60	-	CH_3CN	12
7	**1** (1.0)	60	-	benzene	trace
8	**1** (1.0)	60	-	Toluene	35
9	**1** (1.0)	60	-	DMSO	33
10	**1** (1.0)	60	-	1,4-oxidane	40
11	**1** (1.0)	60	-	DMF	51
12	**1** (1.0)	70	-	Toluene/H_2O	62
13	**1** (1.0)	80	-	Toluene/H_2O	73
14	**1** (1.0)	90	-	Toluene/H_2O	87
15	**1 (1.0)**	**100**	-	**Toluene/H_2O**	**99**
16	**1** (1.0)	105	-	Toluene/H_2O	75
17	**1** (1.0)	110	-	Toluene/H_2O	70
18	**1** (1.0)	120	-	Toluene/H_2O	64
19[b]	**1** (1.0)	100	-	Toluene/H_2O	12
20[c]	**1** (1.0)	100	-	Toluene/H_2O	28
21[d]	**1** (1.0)	100	-	Toluene/H_2O	67
22[e]	**1** (1.0)	100	-	Toluene/H_2O	36

[a]Reaction conditions: Cat. **1** (1.0 mol%), phenylboronic acid (1.0 mmol), $Na_2S_2O_3$ (2.0 equiv.), toluene/ H_2O (4:1), air condition, at 60 °C for 24 h. [b]8 h, [c]16 h, [d]32 h, [e]40 h , [f]Substrate yield was determined by GC-MS analysis.

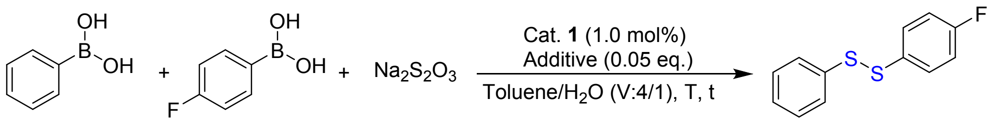
Table S3. Effect of temperature, additive on the synthesis of unsymmetric disulfides[a]



Entry	Catalyst (mol%)	T (°C)	Time (h)	Yield (%)[b]
1	**1** (1.0)	80	24	14
2	**1** (1.0)	90	24	21
3	**1** (1.0)	110	24	32
4	**1** (1.0)	120	24	27
5	**1** (1.0)	100	6	Trace
6	**1** (1.0)	100	12	Trace
7	**1** (1.0)	100	18	16
8	**1 (1.0)**	**100**	**24**	**39**
9	**1** (1.0)	100	30	12
10	**1** (1.0)	100	36	Trace

[a]Reaction conditions: Cat. **1** (1.0 mol%), phenylboronic acid (1.0 mmol), $Na_2S_2O_3$ (4.0 equiv.), toluene/ H_2O (4:1), air condition, at 100 °C for 24 h. [b]Substrate yield was determined by GC-MS analysis.

Table S4. Effect of additive on the synthesis of unsymmetric disulfides[a]



Entry	Catalyst (mol%)	Additive (equiv.)	Yield (%)[b]
1	**1** (1.0)	K_3PO_4	24
2	**1** (1.0)	Na_2SO_4	18
3	**1** (1.0)	KF	16
4	**1** (1.0)	NaCl	trace
5	**1** (1.0)	KCl	trace
6	**1** (1.0)	NaBr	trace
7	**1 (1.0)**	**Na_2SO_3**	**61**

[a]Reaction conditions: Cat. **1** (1.0 mol%), phenylboronic acid (1.0 mmol), $Na_2S_2O_3$ (4.0 eq.), toluene/ H_2O (4:1), air condition, at 100 °C for 24 h. [b]Substrate yield was determined by GC-MS analysis.

IV. Experimental section.

Reaction condition A: The Cat. **1** (1.0 mol%), phenylboronic acid (1.0 mmol), $Na_2S_2O_3$(2.0 equiv.) were added into toluene/ H_2O (2.0 mL) with air condition at 100 °C for 24 h. Afterwards, a small amount of ethyl acetate was added into the reaction mixture and the solution was quickly filtered. The filtered solid was washed, dried and then recycled. Reaction mixture was analyzed by GC-MS analysis. Finally, the solvent was removed in vacuo, and the corresponding disulfide was purified by washing through base-washed silica gel column. (Petroleum Ether: EtOAc = 20:1)

Reaction condition B: The Cat. **1** (1.0 mol%), boronic acid **1** (1.0 mmol), boronic acid **2** (1.0 mmol), $Na_2S_2O_3$ (4.0 equiv.) were added into toluene/ H_2O (4.0 mL) with air condition at 100 °C for 24 h. Afterwards, a small amount of ethyl acetate was added into the reaction mixture and the solution was quickly filtered. The filtered solid was washed, dried and then recycled. Reaction mixture was analyzed by GC-MS analysis. Finally, the solvent was removed in vacuo, and the corresponding disulfide was purified by washing through base-washed silica gel column. (Petroleum Ether: EtOAc = 20:1)

V. Recycling experiments.

The Cat. **1** was precipitated after the oxidative coupling experiment, and then recovered for reusing (Figure S4). The recovered catalyst was characterized by FT-IR and XRD (Figure S5, S6).

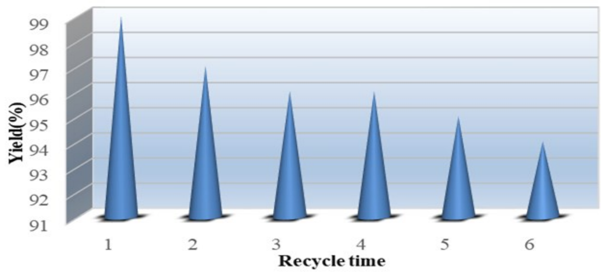


Figure S4. Recycling experiments for the Cat. **1**.

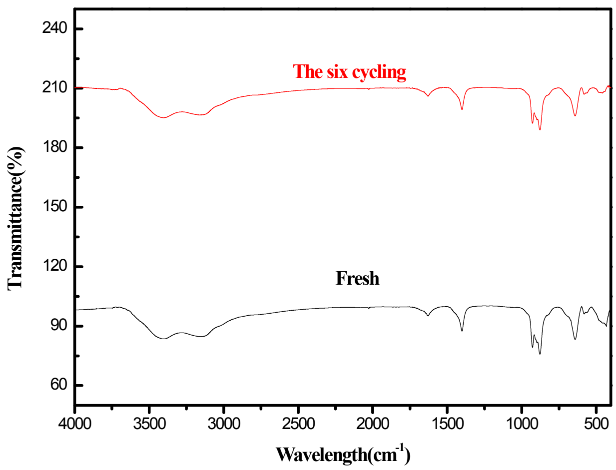


Figure S5. FT-IR spectra of Cat. **1** before and after reaction.

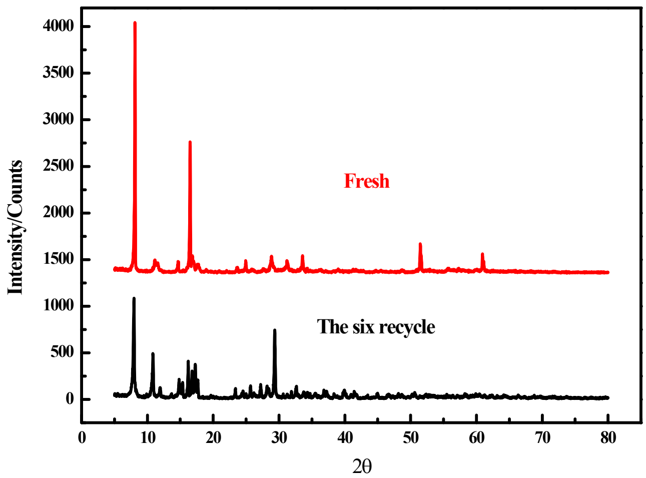
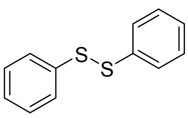
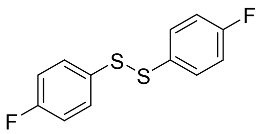


Figure S6. XRD spectra of Cat. **1** before and after reaction.

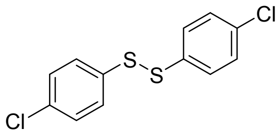
VI. NMR spectra.



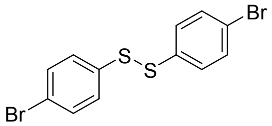
1,2-diphenyldisulfane (2)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.53 (d, *J* = 7.3 Hz, 4H), 7.39 (t, *J* = 7.7 Hz, 4H), 7.31 (d, *J* = 7.3 Hz, 2H). ^{13}C NMR (125 MHz, DMSO) δ 136.27 (s), 129.94 (s), 128.07 (s), 127.70 (s).



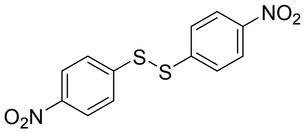
1,2-bis(4-fluorophenyl)disulfane (3)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.55 (dd, *J* = 10.4, 6.8 Hz, 4H), 7.23 (t, *J* = 10.5 Hz, 4H). ^{13}C NMR (125 MHz, DMSO) δ 161.47 (s), 131.90 (s), 131.33 (s), 117.13 (s).



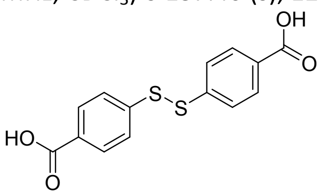
1,2-bis(4-chlorophenyl)disulfane (4)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.44 (d, *J* = 8.6 Hz, 4H), 7.35 (d, *J* = 8.6 Hz, 4H). ^{13}C NMR (125 MHz, DMSO) δ 134.99 (s), 133.04 (s), 129.91 (s), 129.67 (s).



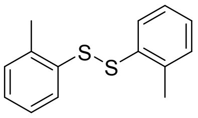
1,2-bis(4-bromophenyl)disulfane (5)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 4H), 7.36 (s, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.03 (s), 132.51 (s), 129.70 (s), 121.84 (s).



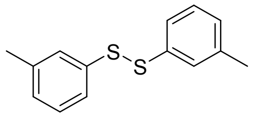
1,2-bis(4-nitrophenyl)disulfane (6)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.7 Hz, 4H), 7.28 (s, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.40 (s), 129.41 (s), 127.89 (s), 127.50 (s).



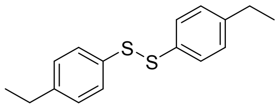
4,4'-disulfanediyldibenzoic acid (7)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 4H), 7.21 (s, 4H), 4.04 (s, 2H).^{13}C NMR (125 MHz, $CDCl_3$) δ 171.10 (s), 137.05 (s), 130.49 (s), 129.08 (s), 127.17 (s).



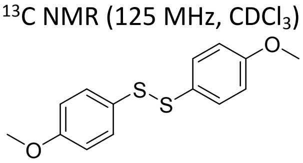
1,2-di-o-tolyldisulfane (8)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.48 (s, 2H), 7.21 (d, J = 15.5 Hz, 6H), 2.35 (s, 6H). 13C NMR (125 MHz, DMSO) δ 137.40 (s), 134.67 (s), 131.00 (s), 128.61 (s), 128.19 (s), 127.39 (s), 19.94 (s).



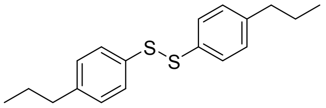
1,2-di-m-tolyldisulfane (9)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.36 – 7.24 (m, 6H), 7.10 (d, J = 7.2 Hz, 2H), 2.28 (s, 6H). ^{13}C NMR (125 MHz, DMSO) δ 139.41 (s), 136.16 (s), 129.77 (s), 128.81 (s), 128.07 (s), 124.76 (s), 21.34 (s).



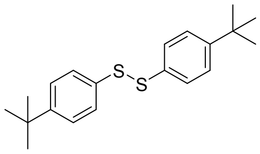
1,2-bis(4-ethylphenyl)disulfane (10)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (s, 4H), 7.16 (s, 4H), 2.60 (s, 4H), 0.97 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.87 (s), 138.10 (s), 134.07 (s), 133.19 (s), 32.89 (s), 20.58 (s).



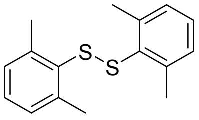
1,2-bis(4-methoxyphenyl)disulfane (11)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (s, 4H), 6.85 (s, 4H), 3.80 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.19 (s), 132.92 (s), 128.72 (s), 114.88 (s), 55.61 (s).



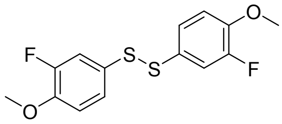
1,2-bis(4-propylphenyl)disulfane (12)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 4H), 7.12 (s, 4H), 2.57 (s, 4H), 1.64 (s, 4H), 0.95 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.19 (s), 134.21 (s), 129.22 (s), 128.34 (s), 37.61 (s), 24.45 (s), 13.81 (s)



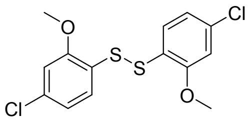
1,2-bis(4-(tert-butyl)phenyl)disulfane (13)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.44 (d, *J* = 8.5 Hz, 4H), 7.38 (d, *J* = 8.5 Hz, 4H), 1.23 (s, 18H). ^{13}C NMR (125 MHz, DMSO) δ 127.88 (s), 126.83 (s), 126.62 (s), 34.76 (s), 31.42 (s).



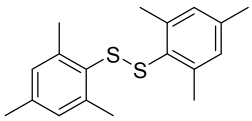
1,2-bis(2,6-dimethylphenyl)disulfane (14)[2-4]**:** 1H NMR (500 MHz, DMSO) δ 7.22 – 7.16 (m, 2H), 7.09 (d, J = 7.5 Hz, 4H), 2.17 (s, 12H). ^{13}C NMR (125 MHz, DMSO) δ 143.20 (s), 134.16 (s), 130.27 (s), 128.68 (s), 21.46 (s).



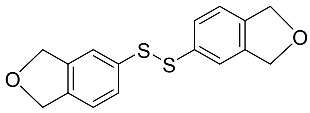
1,2-bis(3-fluoro-4-methoxyphenyl)disulfane (15)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.46 (d, *J* = 6.7 Hz, 2H), 7.00 (d, *J* = 11.0 Hz, 2H), 6.83 (d, *J* = 6.2 Hz, 2H), 3.84 (s, 6H). ^{13}C NMR (125 MHz, DMSO) δ 164.38 (s), 162.43 (s), 130.70 (s), 108.02 (s), 100.77 (s), 56.94 (s).



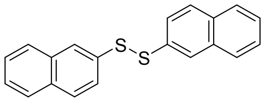
1,2-bis(4-chloro-2-methoxyphenyl)disulfane (16)[2-4]**:** ^{1}H NMR (500 MHz, CDCl3) δ 7.27 (d, J = 147.8 Hz, 3H), 6.85 (s, 3H), 3.71 (s, 6H). ^{13}C NMR (125 MHz, CDCl3) δ 160.63 (s), 135.17 (s), 130.46 (s), 121.12 (s), 114.58 (s), 112.85 (s), 55.71 (s).



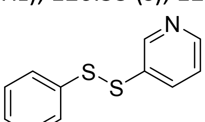
1,2-dimesityldisulfane (17)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 6.92 (s, 4H), 2.13 (s, 12H), 1.99 (s, 6H). ^{13}C NMR (125 MHz, DMSO) δ 143.27 (s), 140.15 (s), 131.25 (s), 129.64 (s), 21.69 (s), 21.39 (s).



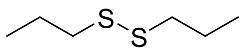
1,2-bis(benzo[d][1,3]dioxol-5-yl)disulfane (18)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.00 (s, 2H), 6.94 (d, *J* = 9.7 Hz, 2H), 6.72 (d, *J* = 8.0 Hz, 2H), 5.97 (s, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.44 (d, *J* = 6.2 Hz), 130.08 (s), 124.90 (s), 111.25 (s), 108.82 (s), 101.73 (s).



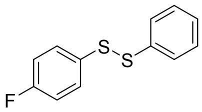
1,2-di(naphthalen-2-yl)disulfane (19)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (s, 2H), 7.92 (s, 1H), 7.79 (d, *J* = 28.0 Hz, 5H), 7.65 (s, 2H), 7.47 (s, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.80 (s), 129.26 (s), 128.05 (s), 127.75 (s), 126.94 (d, *J* = 19.5 Hz), 126.53 (s), 125.96 (s).



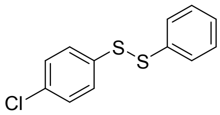
1,2-di(pyridin-3-yl)disulfane(20)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (s, 2H), 8.53 (s, 2H), 7.84 (s, 2H), 7.28 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.87 (s), 149.24 (s), 136.84 (s), 124.31 (s).



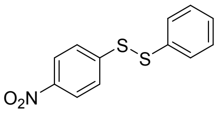
1,2-dipropyldisulfane(21)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.67 (s, 4H), 1.71 (s, 4H), 1.00 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 41.17 (s), 22.50 (s), 13.09 (s).



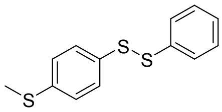
1-(4-fluorophenyl)-2-phenyldisulfane (22)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.52 (d, *J* = 20.9 Hz, 4H), 7.40 – 7.17 (m, 5H). ^{13}C NMR (125 MHz, DMSO) δ 163.30 (s), 136.30 (s), 130.93 (s), 129.87 (s), 128.05 (d, *J* = 18.4 Hz), 127.63 (s), 117.07 (s).



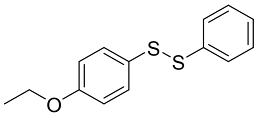
1-(4-chlorophenyl)-2-phenyldisulfane (23)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.50 (d, *J* = 8.5 Hz, 4H), 7.42 – 7.21 (m, 5H). ^{13}C NMR (125 MHz, DMSO) δ 136.32 (s), 133.06 (s), 132.81 (s), 129.90 (d, J = 7.7 Hz), 128.18 (s), 127.90 (d, J = 15.6 Hz).



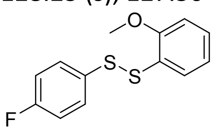
1-(4-nitrophenyl)-2-phenyldisulfane (24)[2-4]: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (s, 2H), 7.68 (s, 2H), 7.50 (d, *J* = 9.7 Hz, 2H), 7.32 (d, *J* = 33.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.45 (s), 144.35 (s), 135.63 (s), 129.68 (s), 128.10 (s), 127.45 (s), 126.45 (s), 124.44 (s).



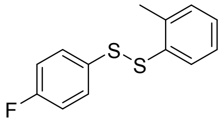
1-(4-(methylthio)phenyl)-2-phenyldisulfane(25)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 – 7.89 (m, 2H), 7.82 – 7.77 (m, 2H), 7.60 – 7.42 (m, 5H), 2.34 (d, *J* = 4.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.96 (s), 133.77 (s), 132.73 (s), 130.15 (s), 128.98 (s), 128.05 (s), 126.43 (s), 125.95 (s), 14.48 (s).



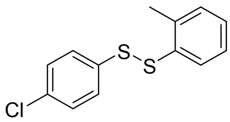
1-(4-ethoxyphenyl)-2-phenyldisulfane (26)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.4 Hz, 3H), 7.26 (d, *J* = 47.8 Hz, 5H), 7.13 (s, 1H), 3.91 (s, 2H), 1.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.21 (s), 131.79 (s), 129.00 (d, *J* = 9.7 Hz), 128.23 (s), 127.50 (s), 127.17 (s), 115.19 (s), 63.57 (s), 14.17 (s).



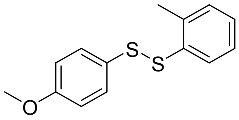
1-(4-fluorophenyl)-2-(2-methoxyphenyl)disulfane(27)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 20.5 Hz, 2H), 7.28 (s, 3H), 6.64 (s, 3H), 3.86 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.79 (s), 131.56 (s), 131.21 (s), 129.23 (s), 128.21 (s), 127.39 (s), 120.09 (s), 108.00 (s), 99.77 (s), 56.37 (s).



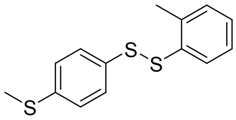
1-(4-fluorophenyl)-2-(o-tolyl)disulfane (28)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.36 (m, 4H), 7.12 (s, 1H), 6.94 (d, *J* = 8.6 Hz, 3H), 2.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.63 (s), 137.57 (s), 132.22 (s), 131.30 (d, *J* = 8.3 Hz), 130.53 (s), 127.68 (s), 126.73 (s), 116.40 (s), 20.07 (s).



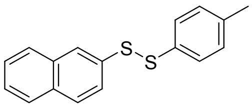
1-(4-chlorophenyl)-2-(o-tolyl)disulfane (29)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.4 Hz, 4H), 7.19 (s, 3H), 7.09 (s, 1H), 1.98 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.17 (s), 133.65 (s), 130.52 (s), 129.47 (s), 129.31 (s), 129.15 (s), 128.22 (s), 127.60 (s), 126.80 (s), 14.24 (s).



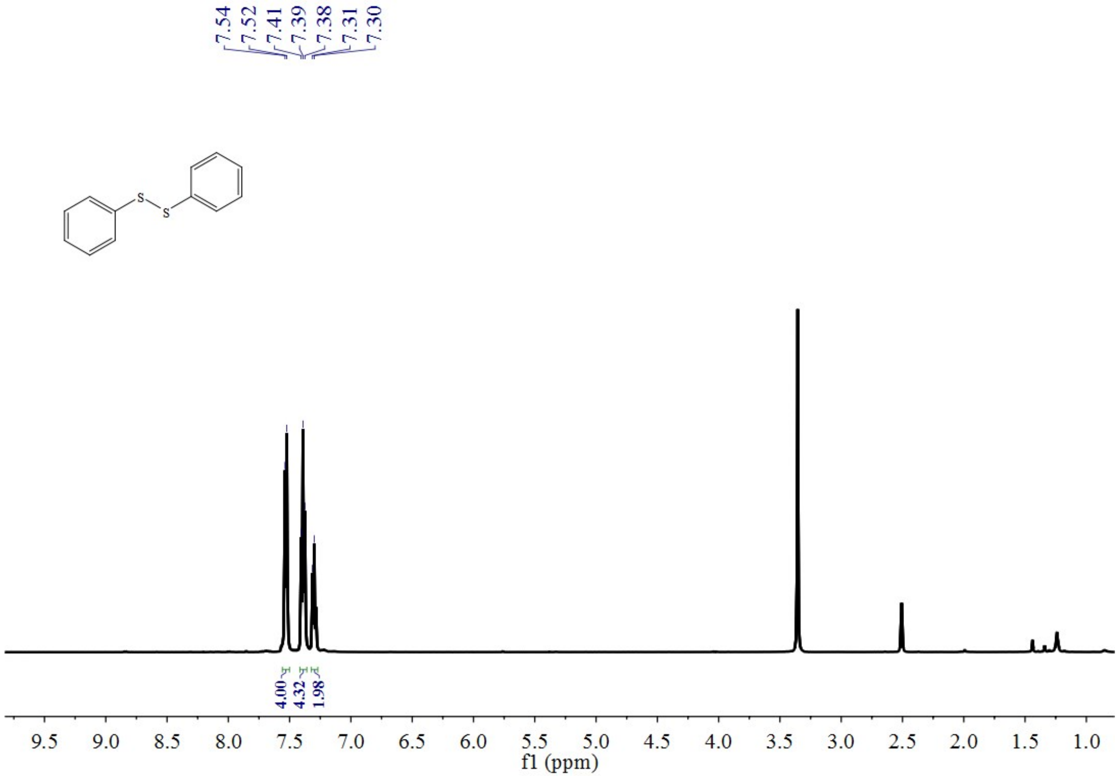
1-(4-methoxyphenyl)-2-(m-tolyl)disulfane (30)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 2H), 7.31 (d, *J* = 33.0 Hz, 2H), 7.22 (s, 1H), 7.08 (s, 1H), 6.86 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.08 (s), 139.16 (s), 137.51 (s), 132.94 (s), 132.00 (s), 129.08 (d, J = 11.8 Hz), 128.40 (s), 125.55 (s), 114.98 (s), 55.64 (s), 21.63 (s).



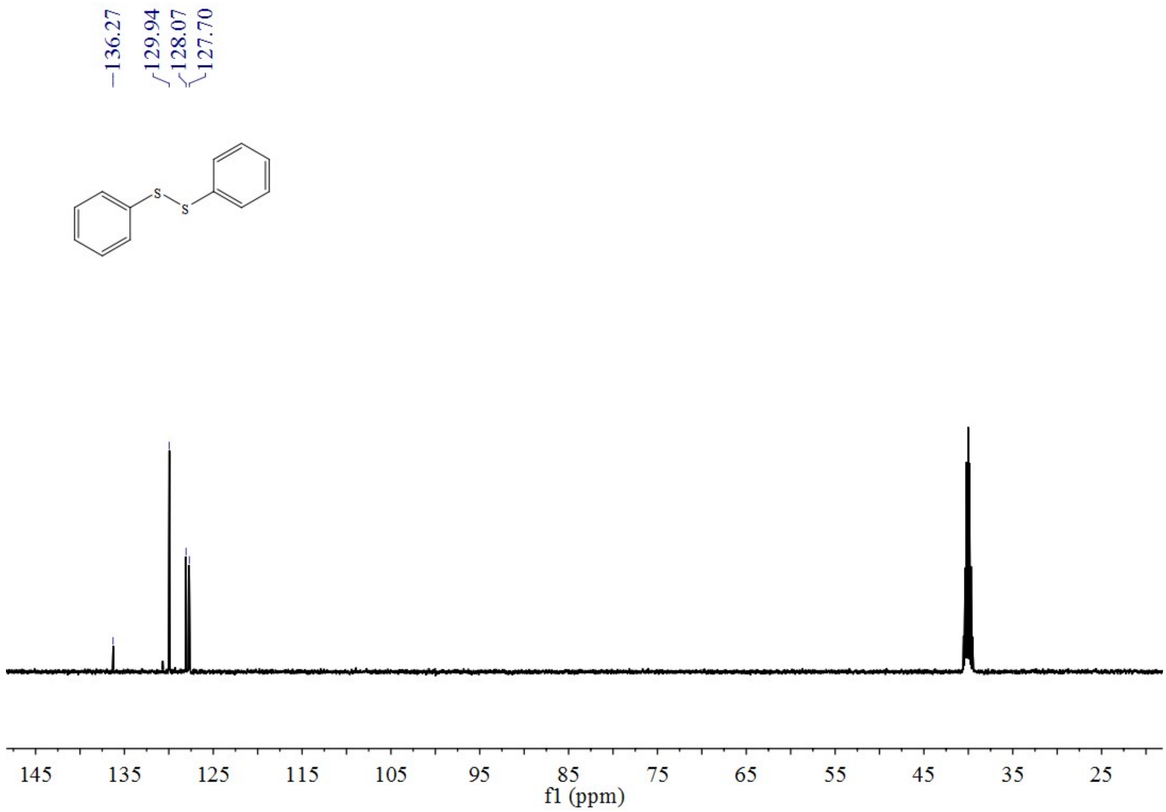
1-(4-(methylthio)phenyl)-2-(o-tolyl)disulfane(31)[2-4]**:** ^{1}H NMR (500 MHz, DMSO) δ 7.36 (d, J = 1.6 Hz, 1H), 7.34 – 7.23 (m, 4H), 7.21 – 7.14 (m, 3H), 2.44 (d, J = 5.0 Hz, 3H), 2.36 – 2.20 (m, 3H). ^{13}C NMR (125 MHz, DMSO) δ 140.48 (s), 137.80 (s), 137.13 (s), 130.31 (s), 129.35 (s), 128.65 (s), 125.76 (s), 125.47 (s), 124.10 (s), 123.91 (s), 21.21 (s), 14.54 (s).



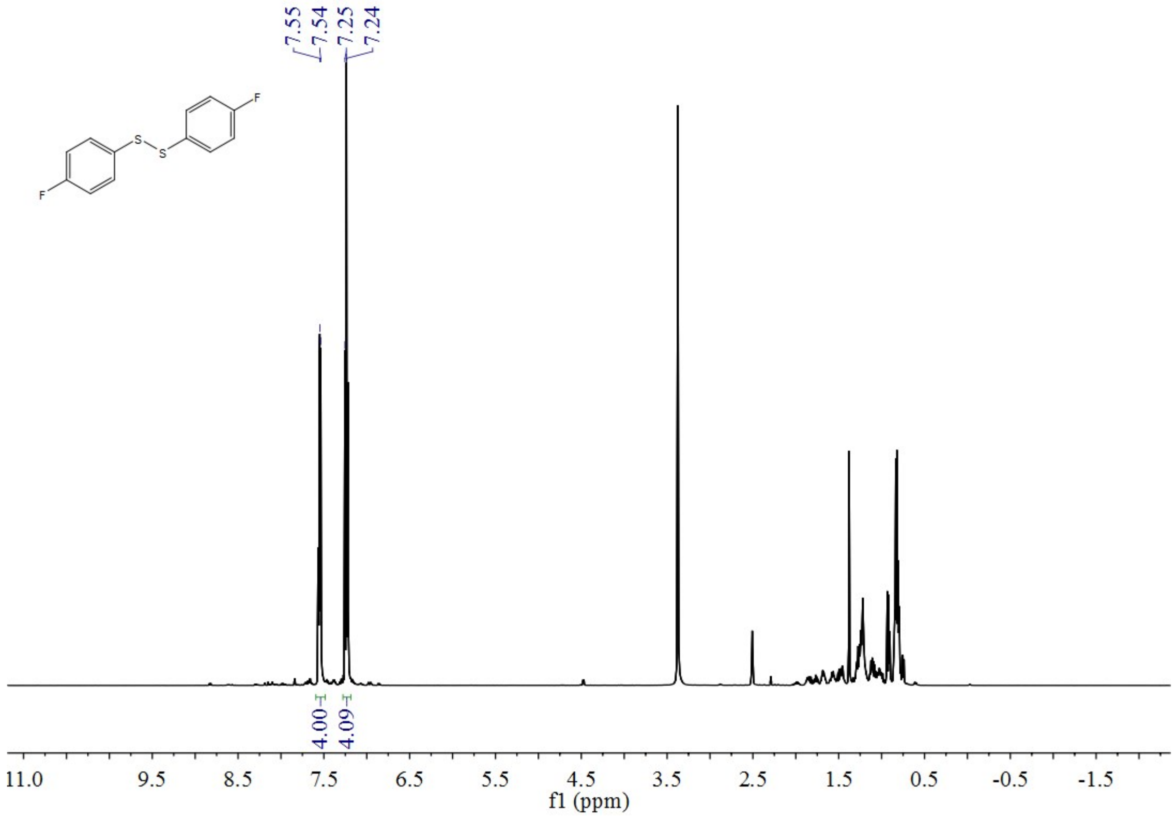
1-(naphthalen-2-yl)-2-(p-tolyl)disulfane(32)[2-4]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, J = 77.8 Hz, 4H), 7.64 (s, 1H), 7.35 (d, J = 71.5 Hz, 6H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.68 (s), 133.49 (s), 129.86 (s), 128.86 (s), 128.23 (s), 127.76 (s), 127.45 (s), 126.69 (s), 126.40 (s), 126.15 (s), 125.68 (s), 21.05 (s).



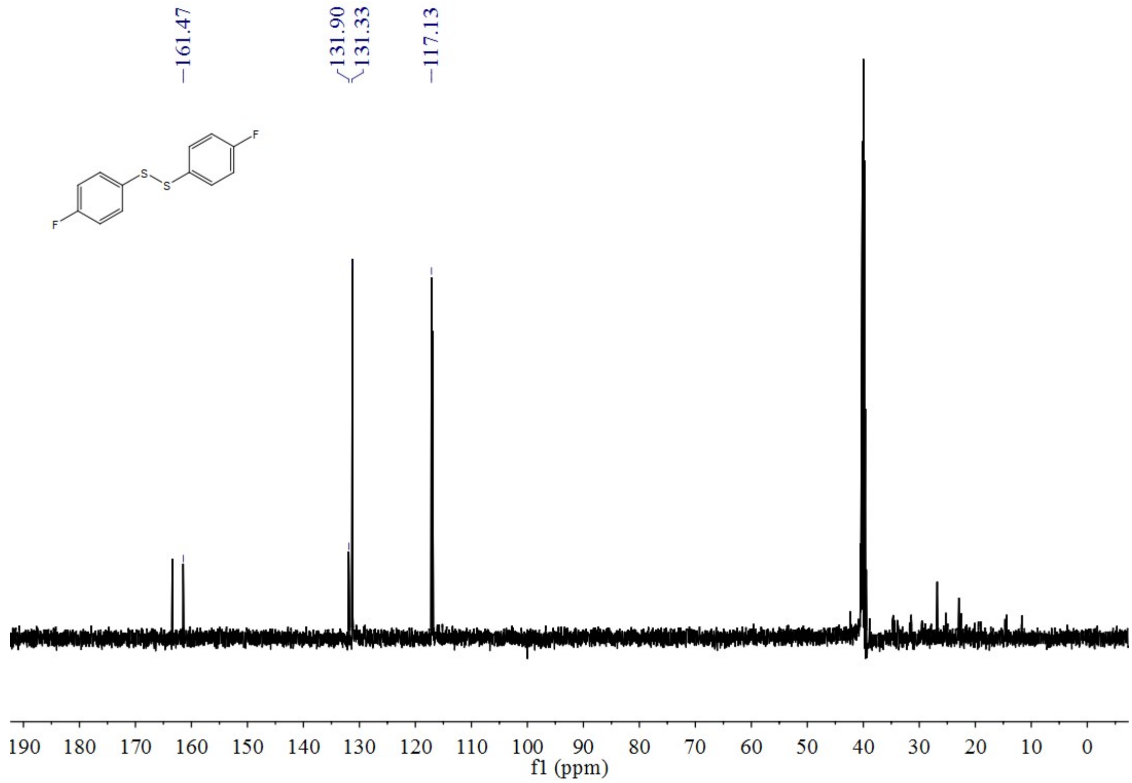
^{1}H NMR spectra of 2 (500 MHz, DMSO)



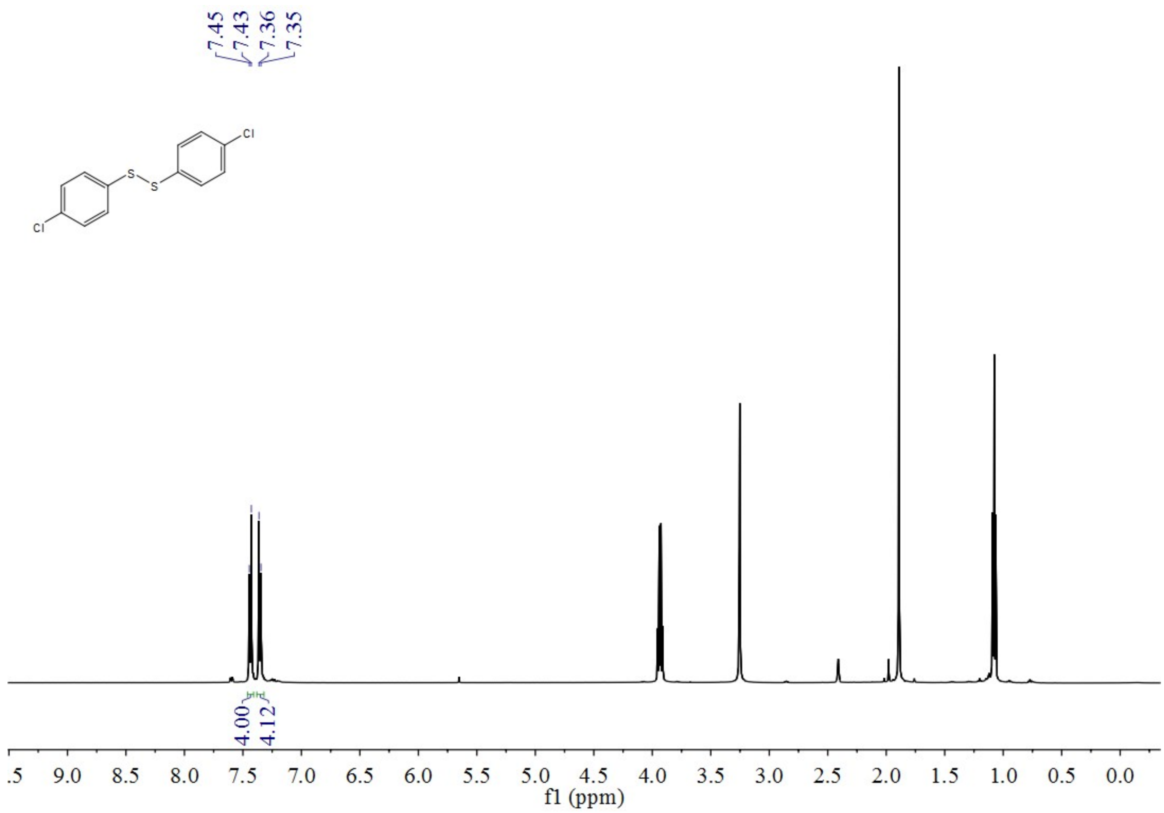
^{13}C NMR spectra of 2 (125 MHz, DMSO)



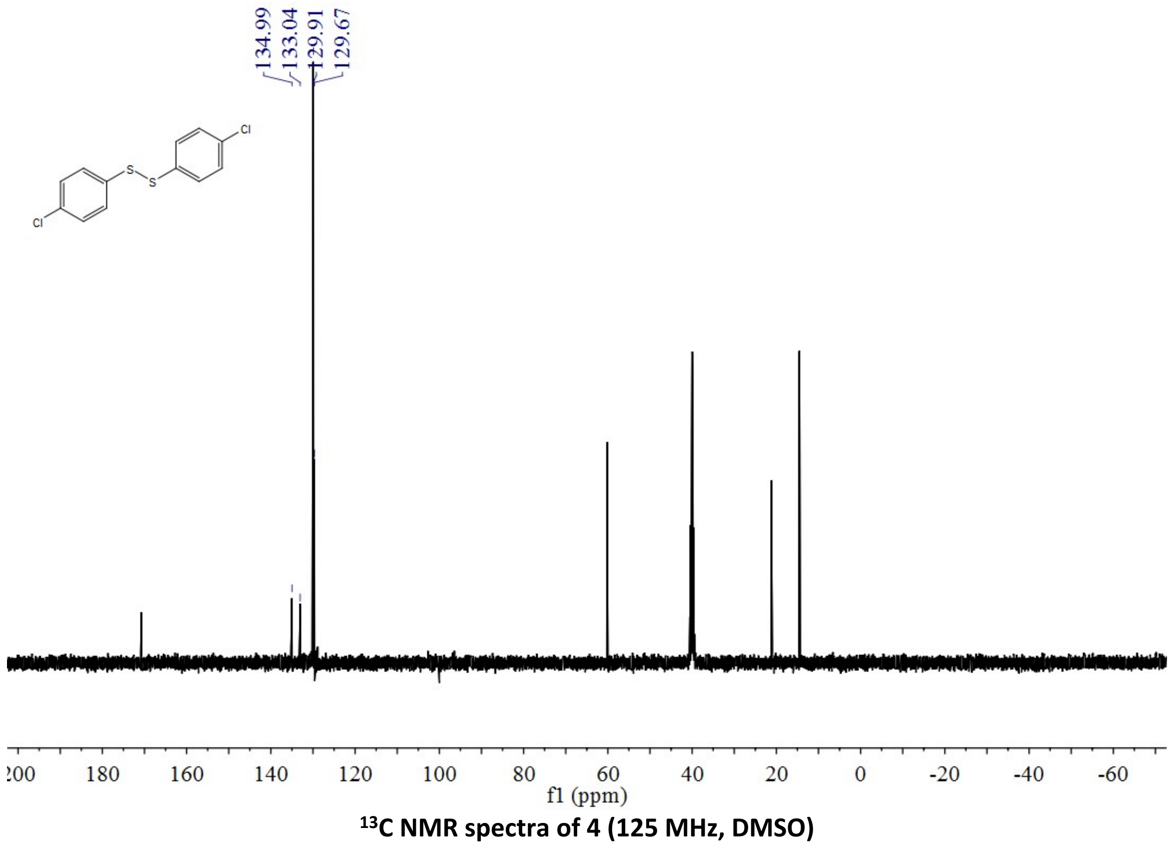
[1]H NMR spectra of 3 (500 MHz, DMSO)



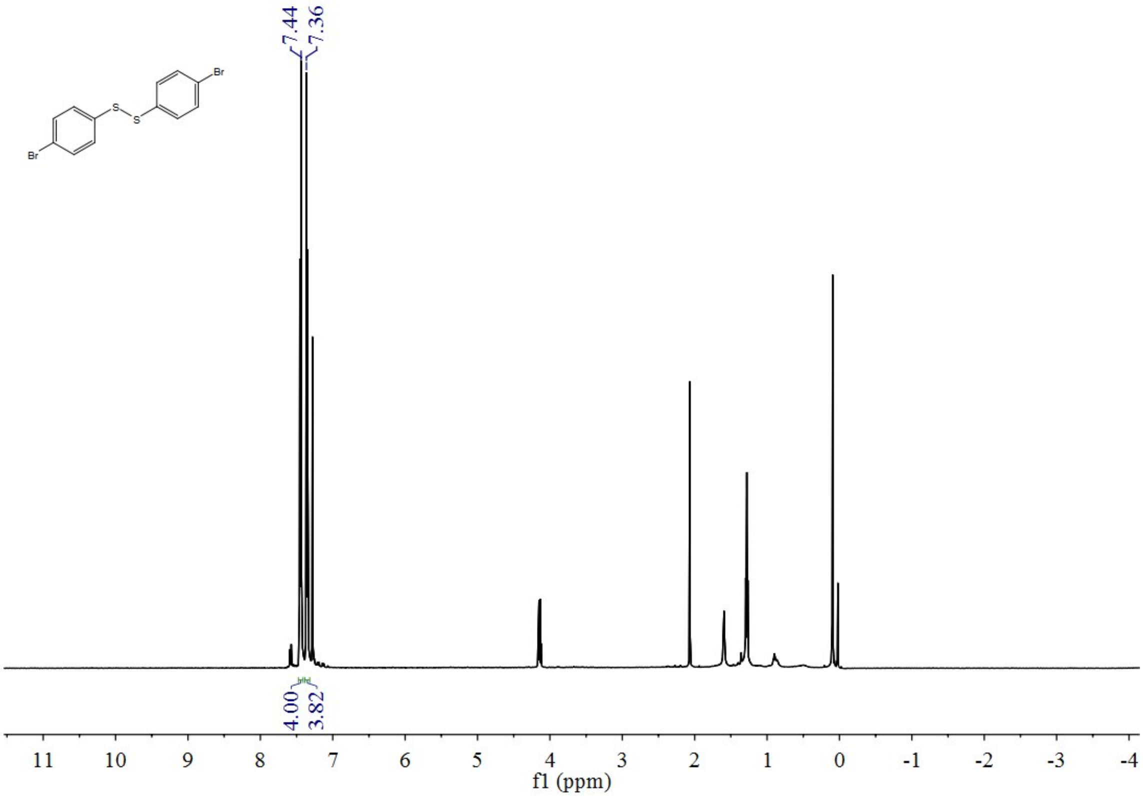
[13]C NMR spectra of 3 (125 MHz, DMSO)



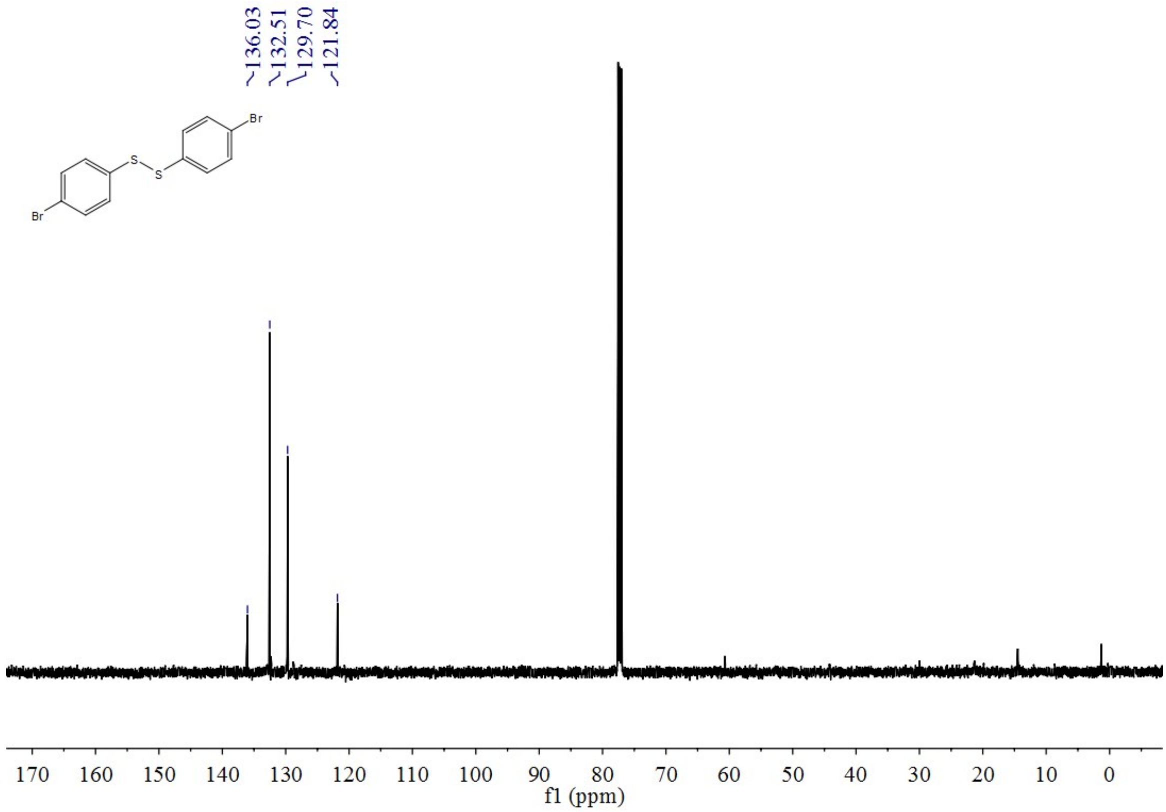
[1]H NMR spectra of 4 (500 MHz, DMSO)



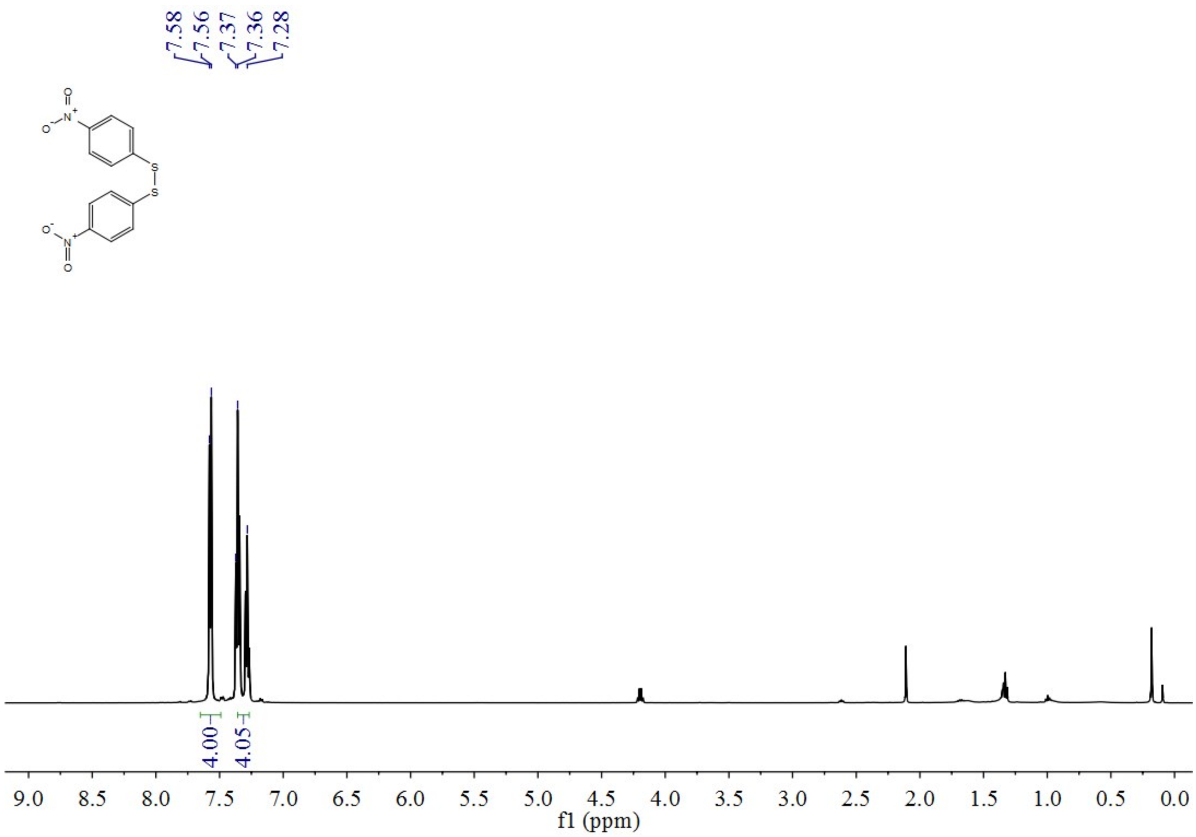
[13]C NMR spectra of 4 (125 MHz, DMSO)



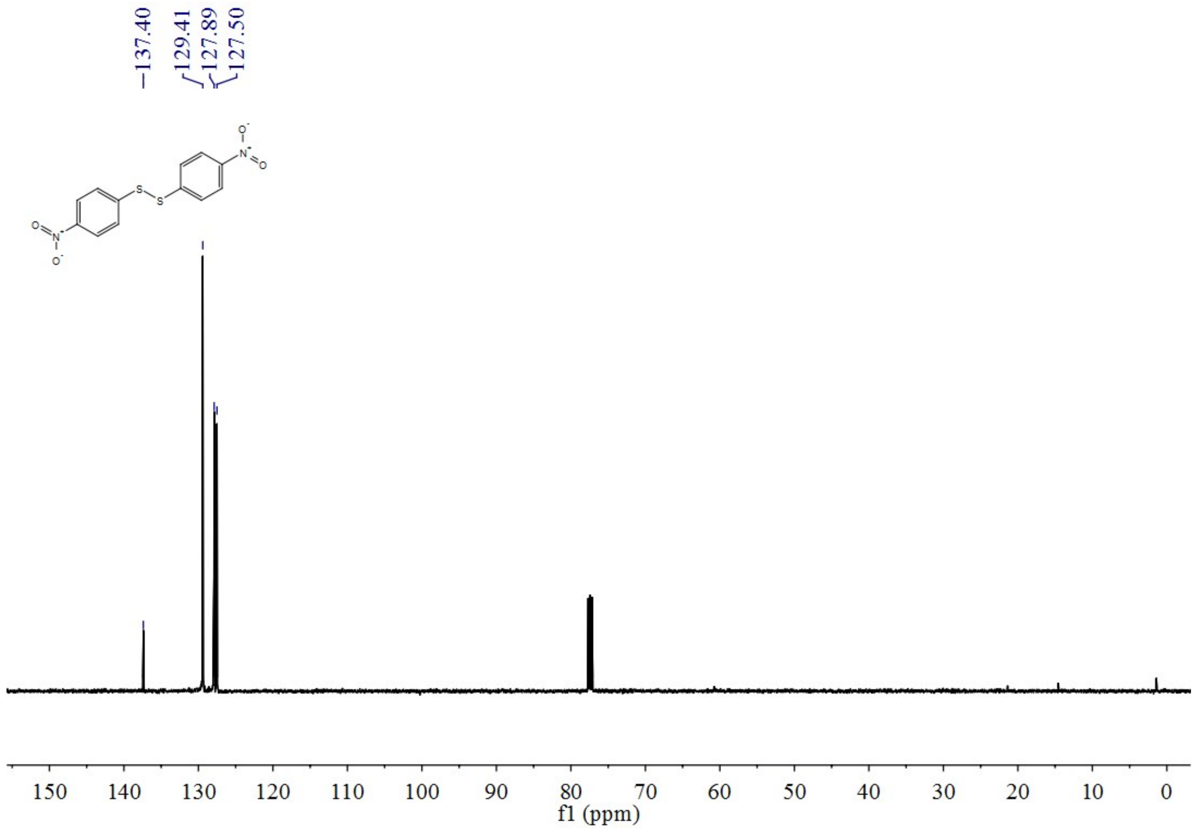
[1]H NMR spectra of 5 (500 MHz, $CDCl_3$)



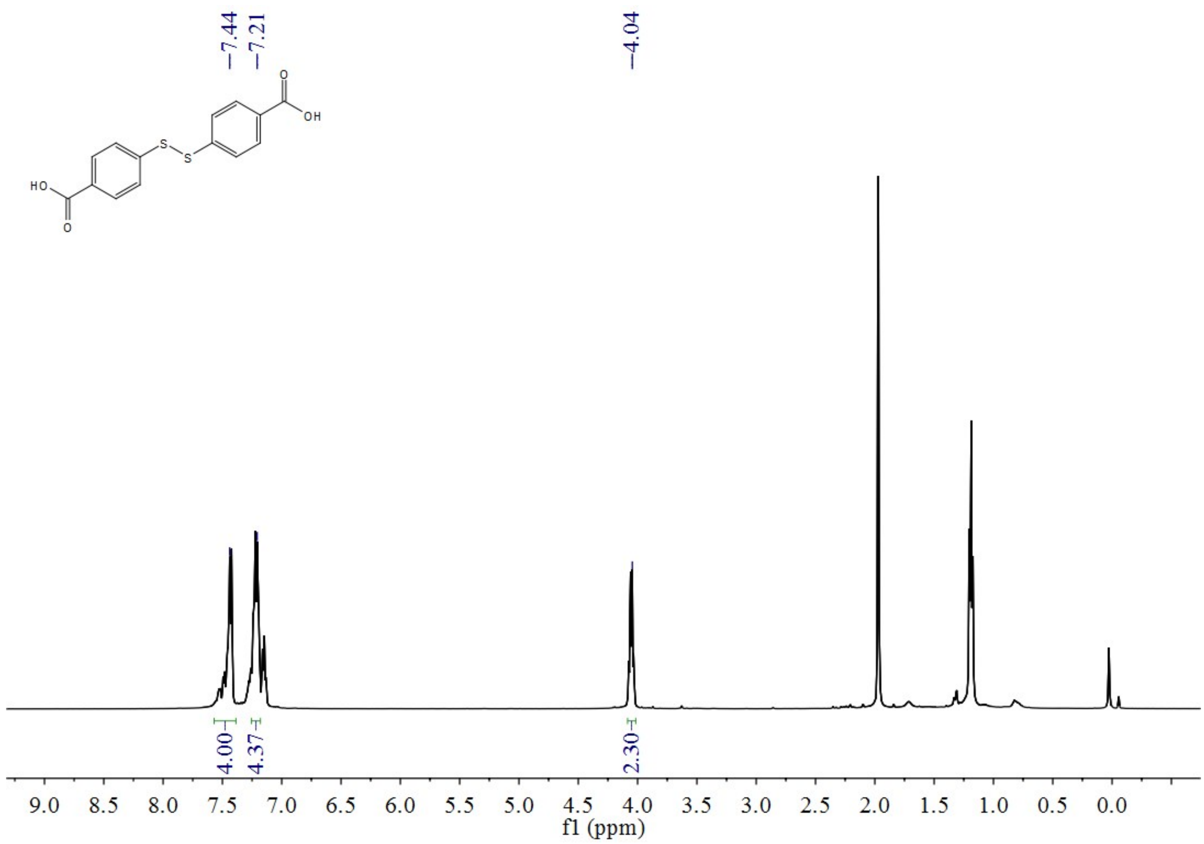
[13]C NMR spectra of 5 (125 MHz, $CDCl_3$)



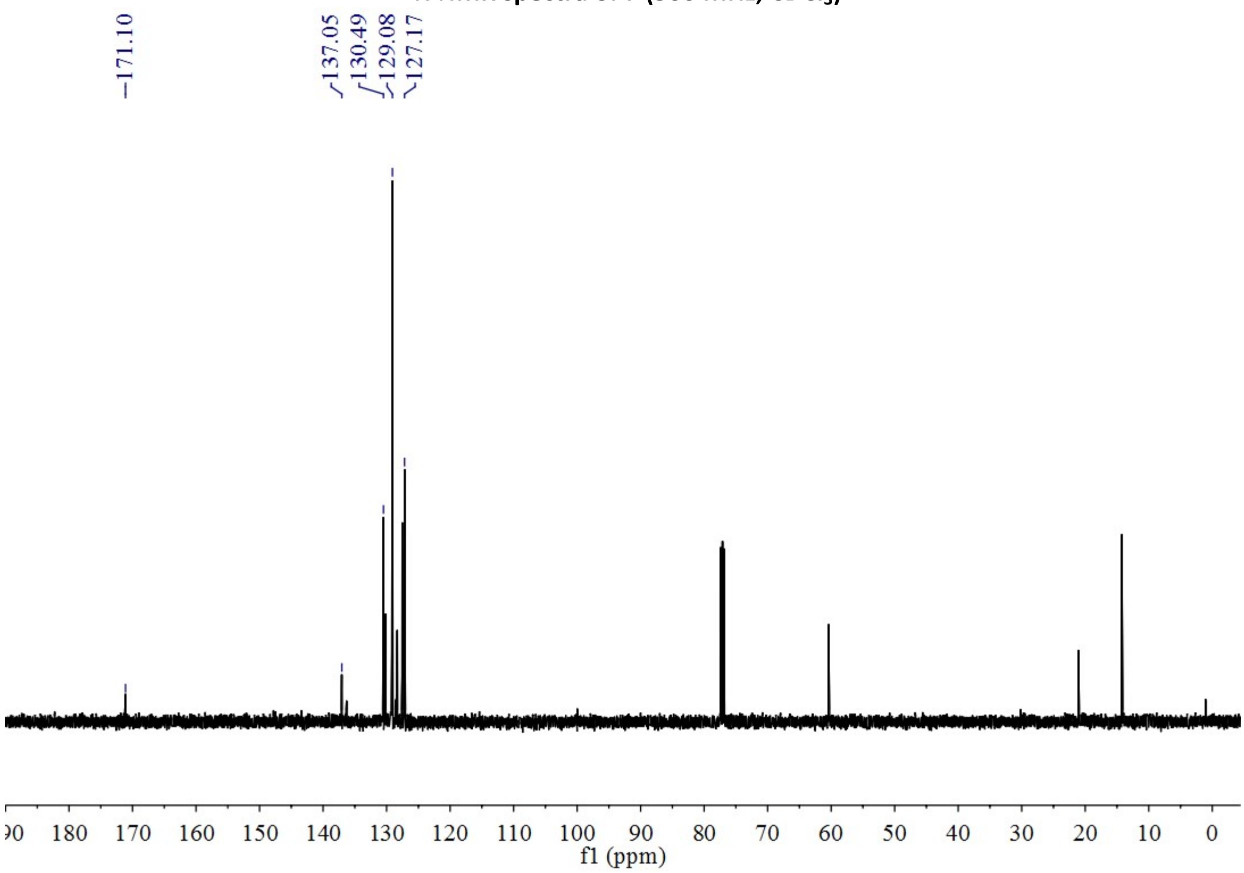
^{1}H NMR spectra of 6 (500 MHz, $CDCl_3$)



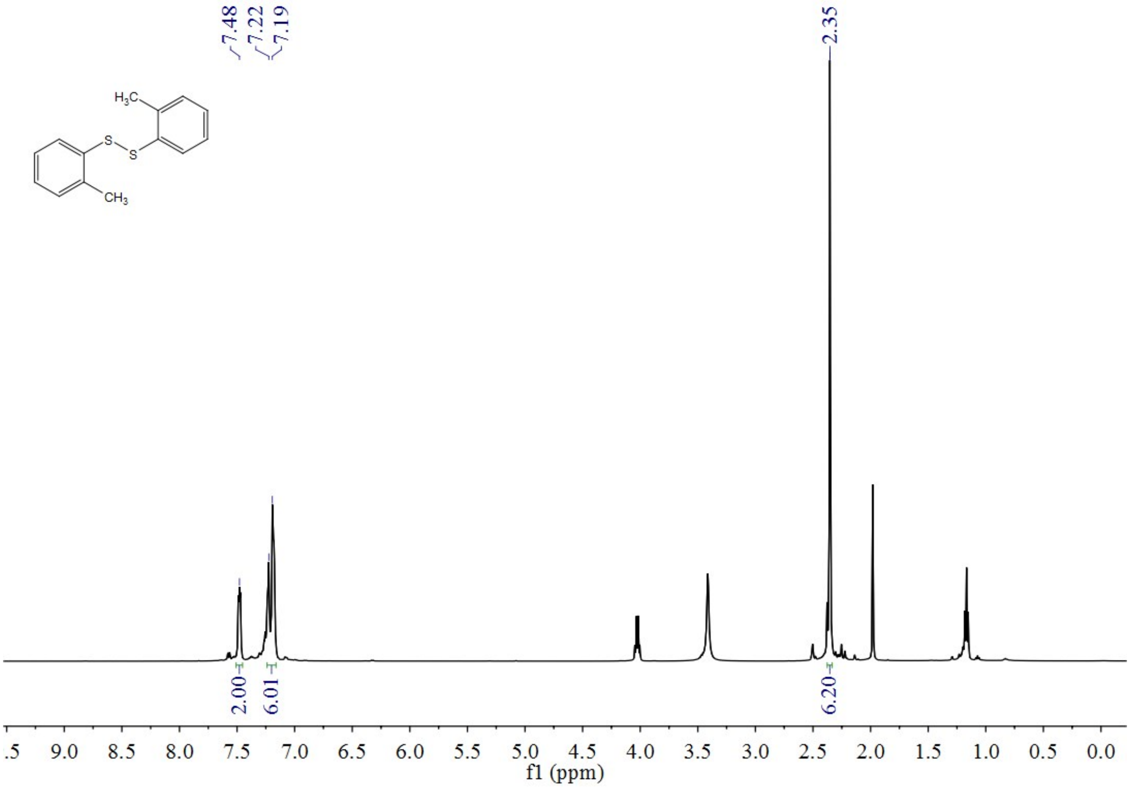
^{13}C NMR spectra of 6 (125 MHz, $CDCl_3$)



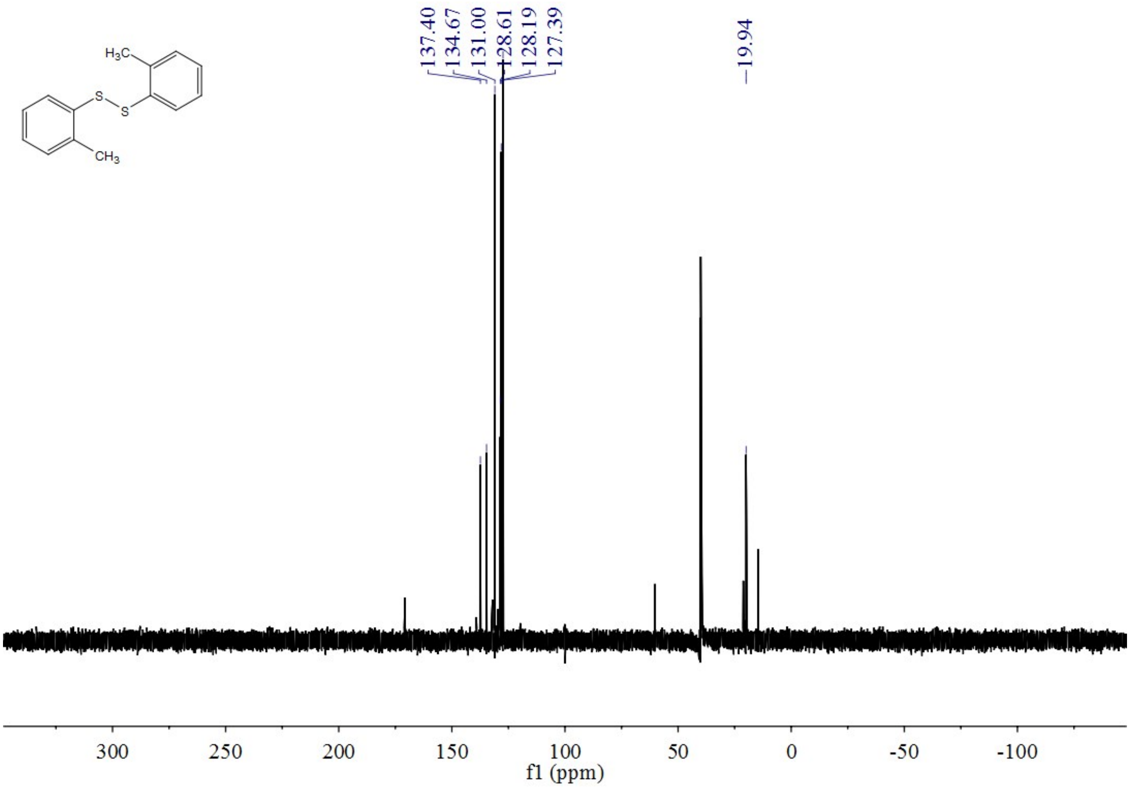
[1]H NMR spectra of 7 (500 MHz, $CDCl_3$)



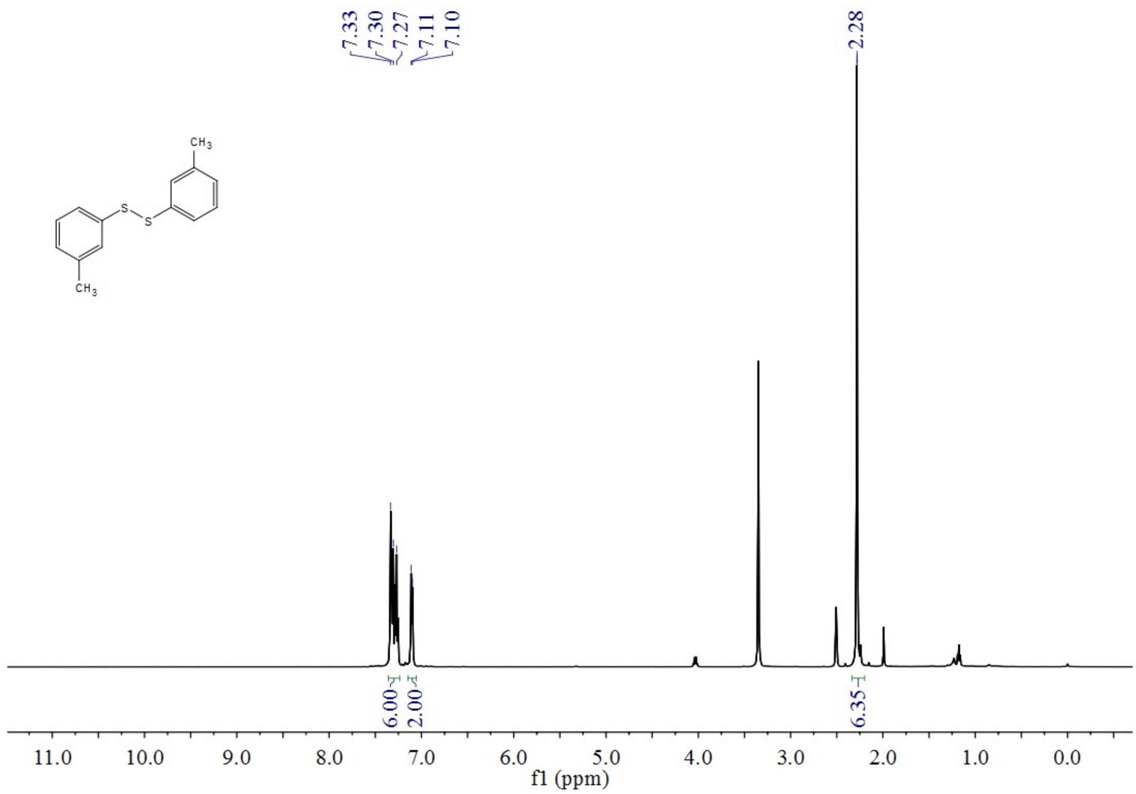
[13]C NMR spectra of 7 (125 MHz, $CDCl_3$)



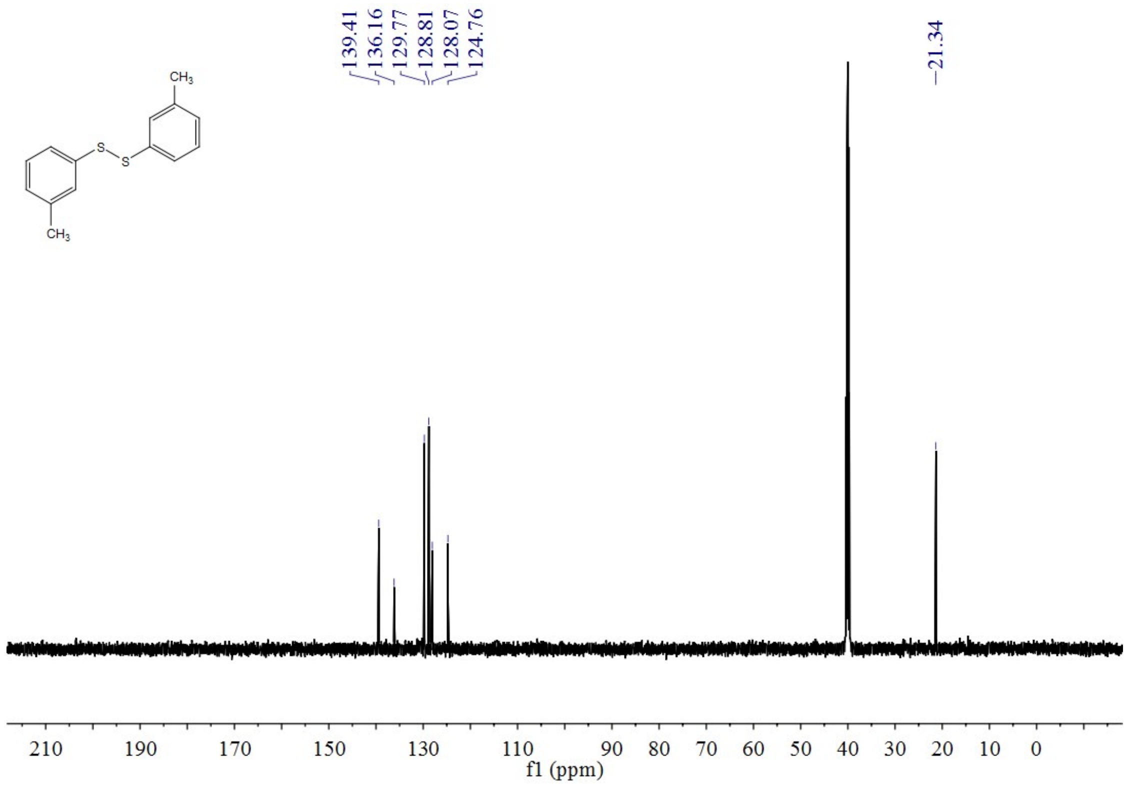
¹H NMR spectra of 8 (500 MHz, DMSO)



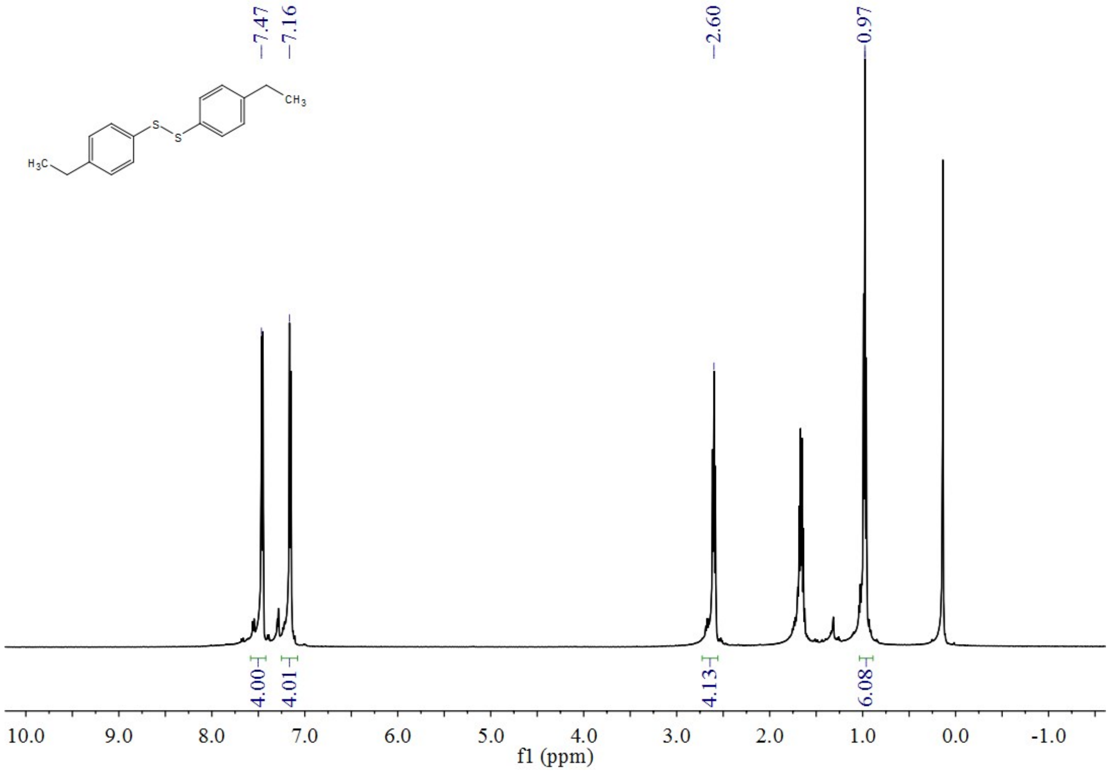
¹³C NMR spectra of 8 (125 MHz, DMSO)



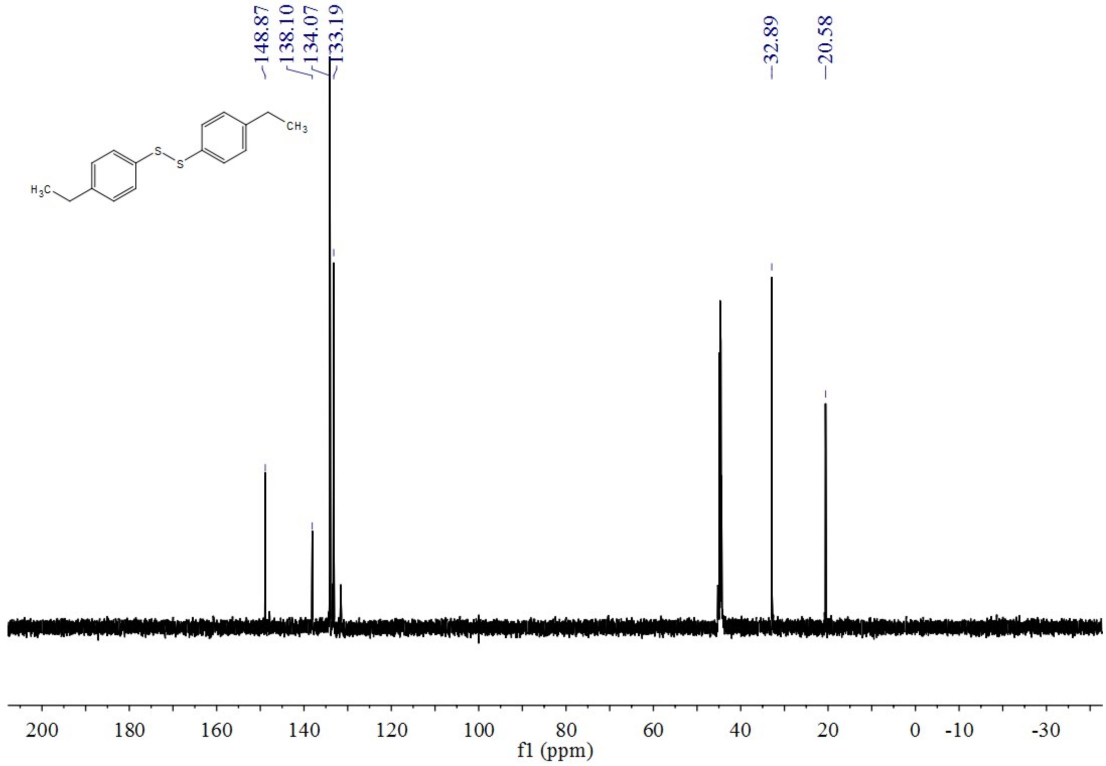
^{1}H NMR spectra of 9 (500 MHz, DMSO)



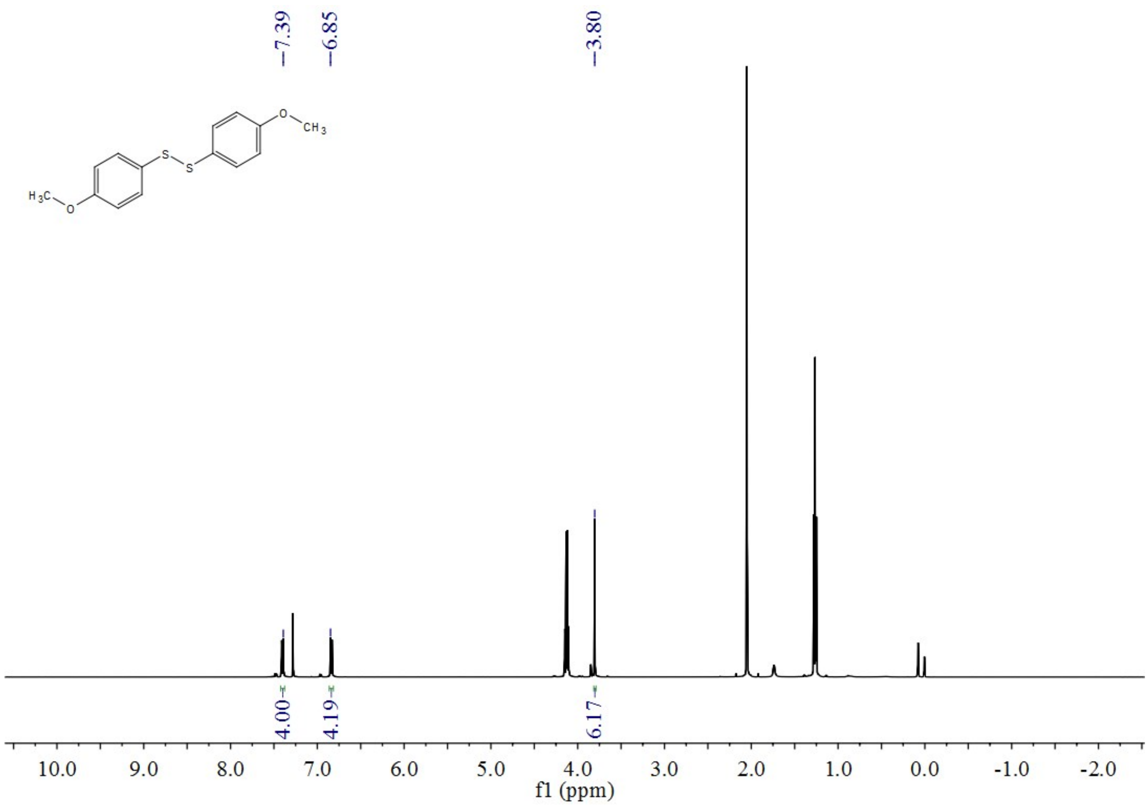
^{13}C NMR spectra of 9 (125 MHz, DMSO)



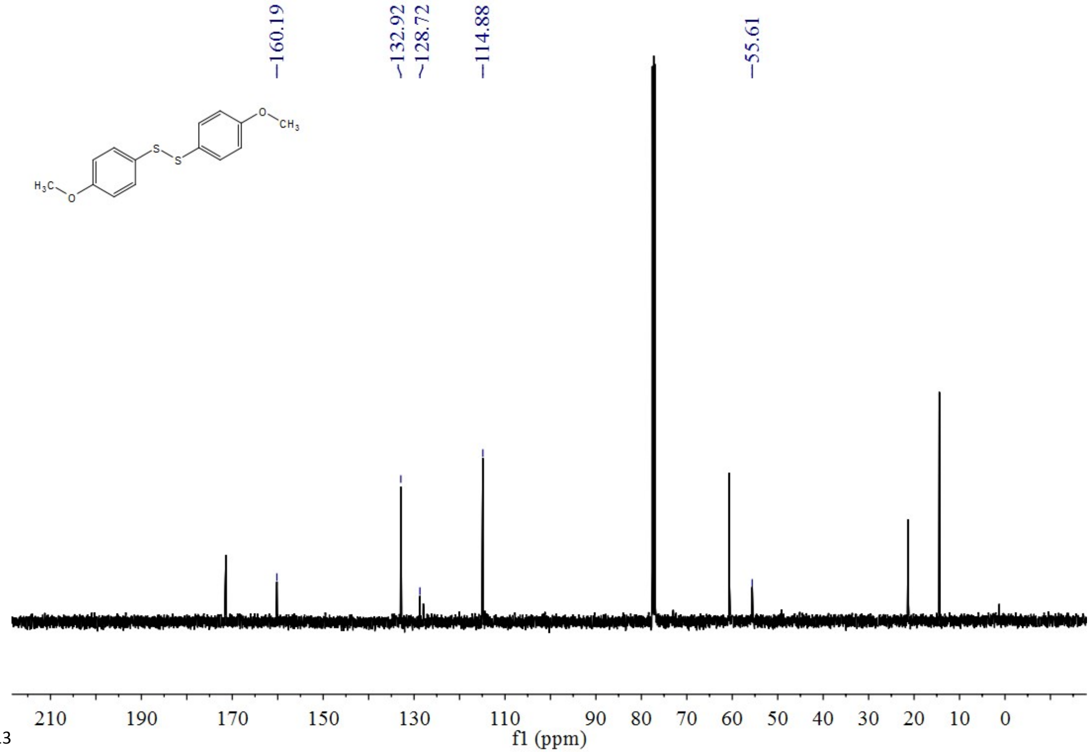
^{1}H NMR spectra of 10 (500 MHz, DMSO)



^{13}C NMR spectra of 10 (125 MHz, DMSO)

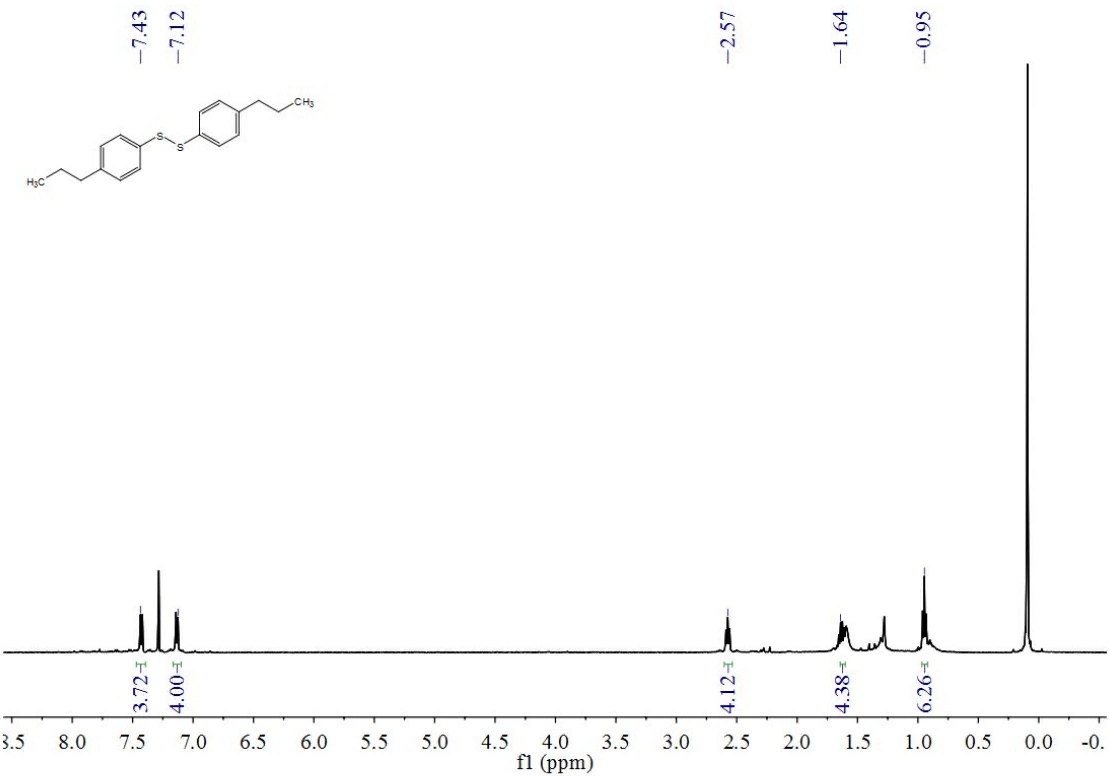


^{1}H NMR spectra of 11 (500 MHz, $CDCl_3$)

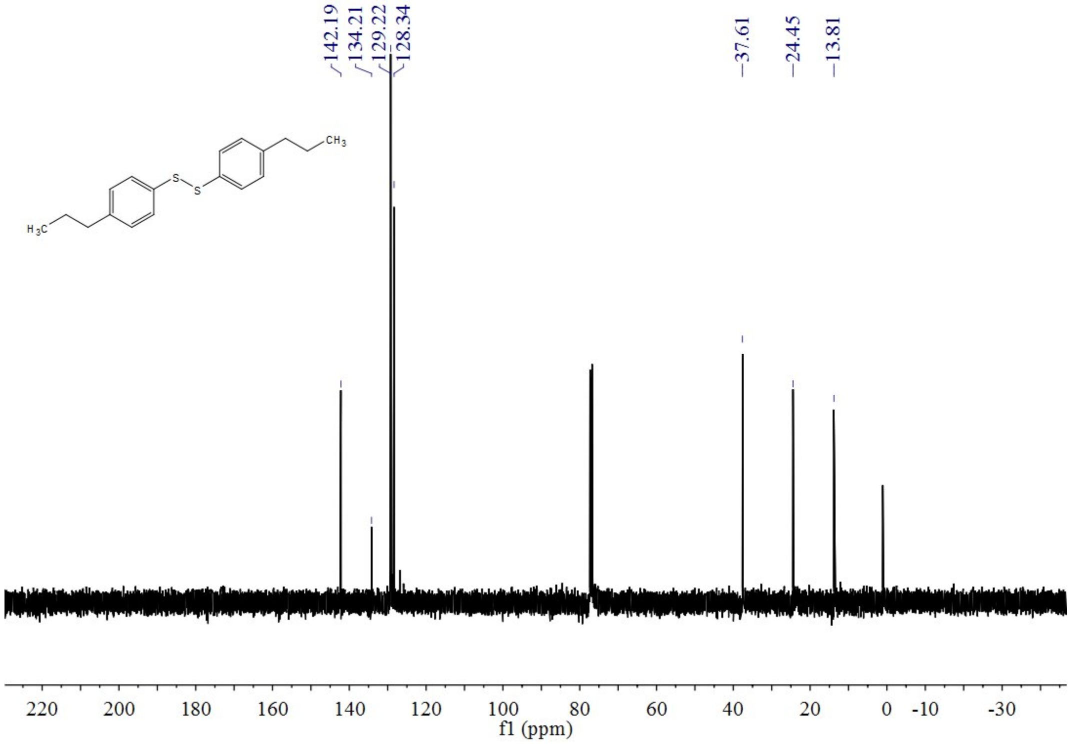


13

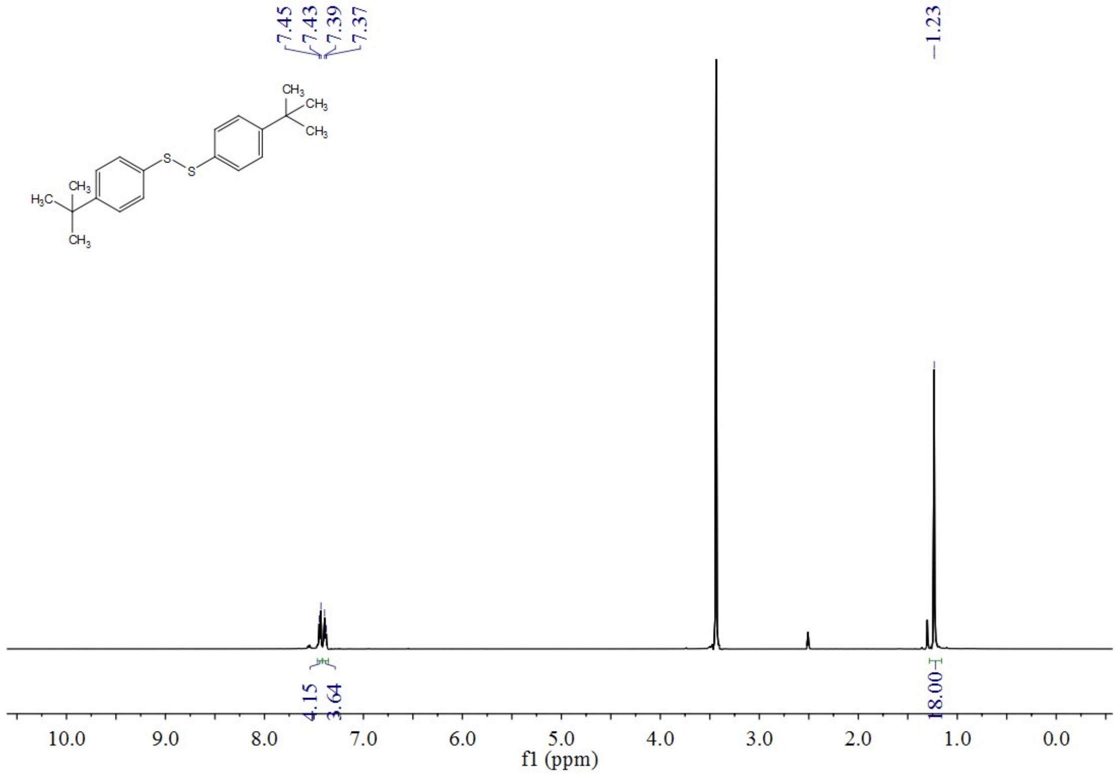
^{13}C NMR spectra of 11 (125 MHz, $CDCl_3$)



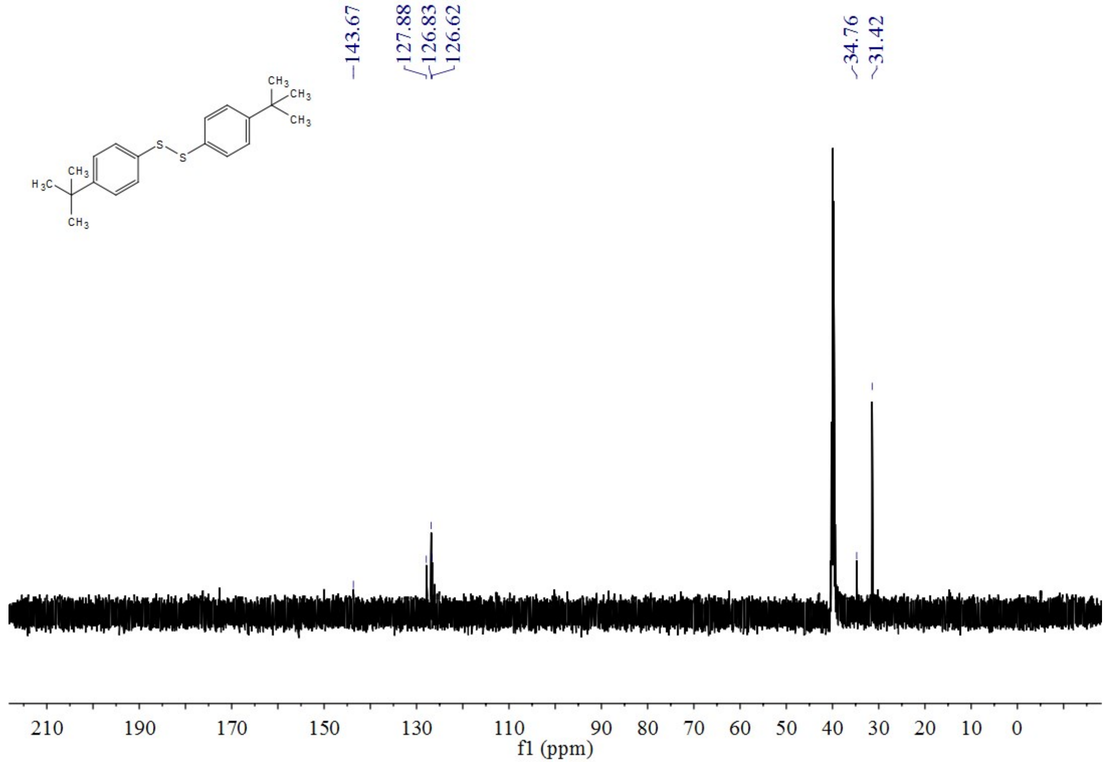
^{1}H NMR spectra of 12 (500 MHz, CDCl$_3$)



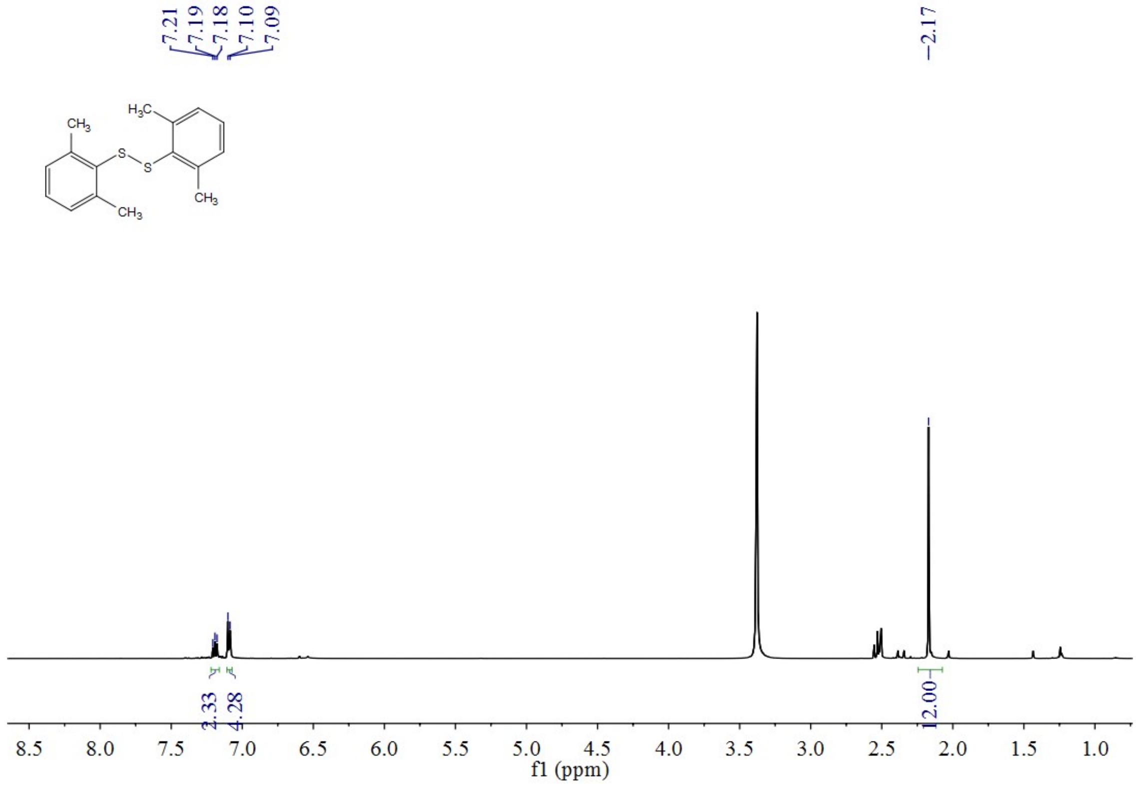
^{13}C NMR spectra of 12 (125 MHz, CDCl$_3$)



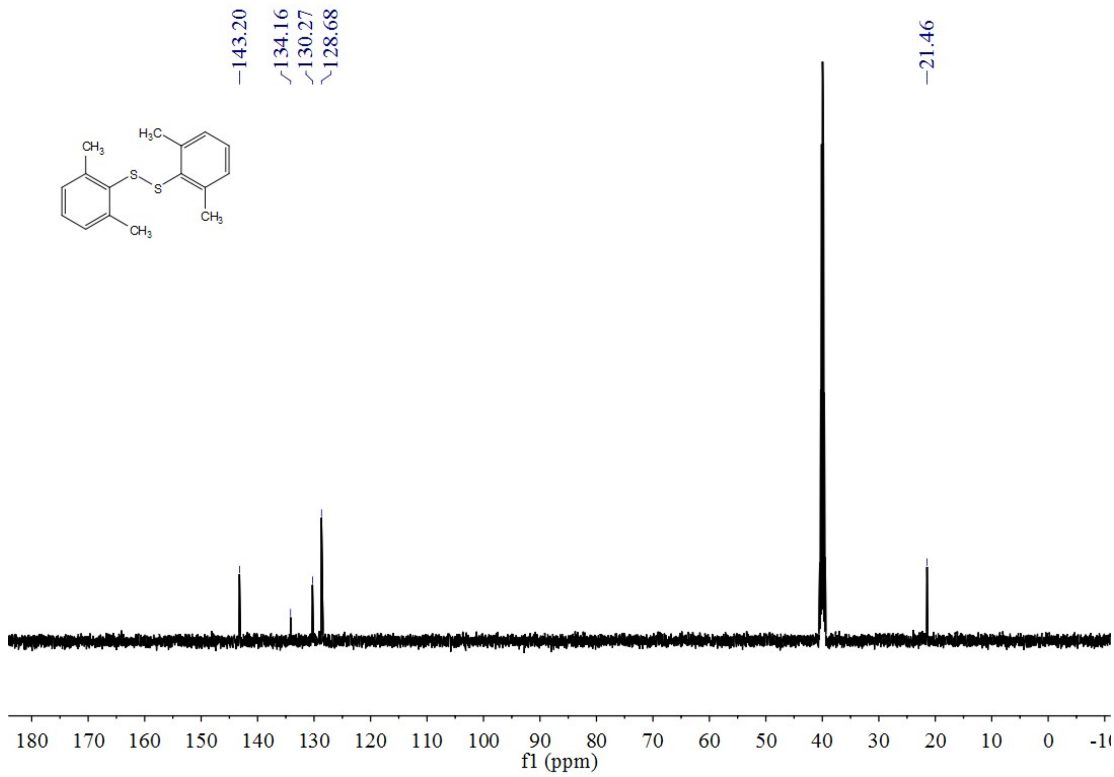
^{1}H NMR spectra of 13 (500 MHz, DMSO)



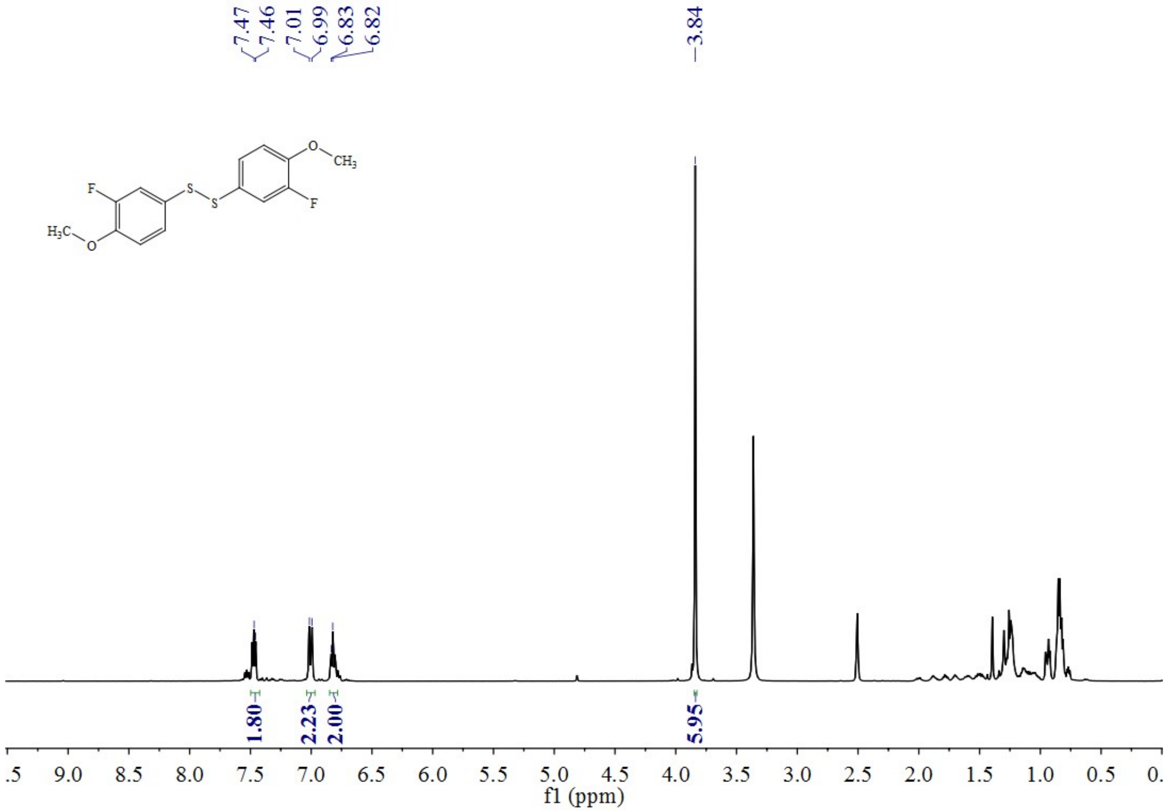
^{13}C NMR spectra of 13 (125 MHz, DMSO)



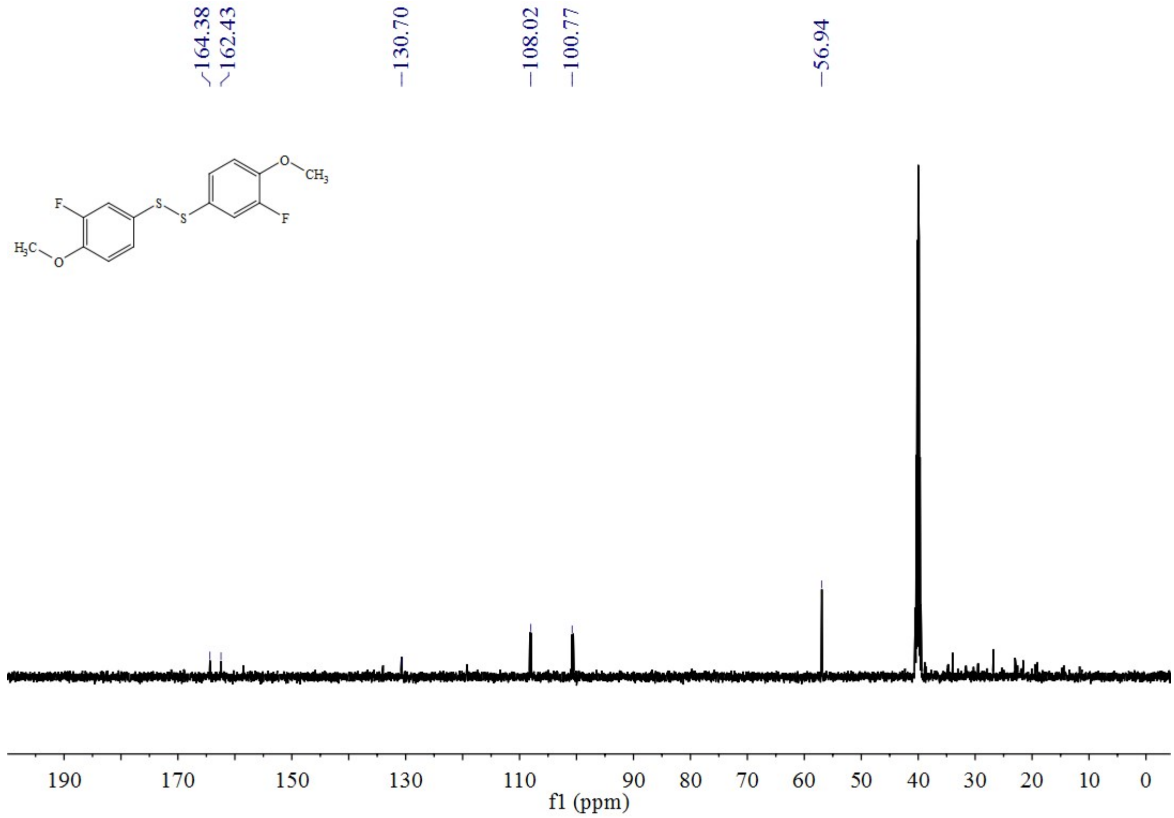
^{1}H NMR spectra of 14 (500 MHz, DMSO)



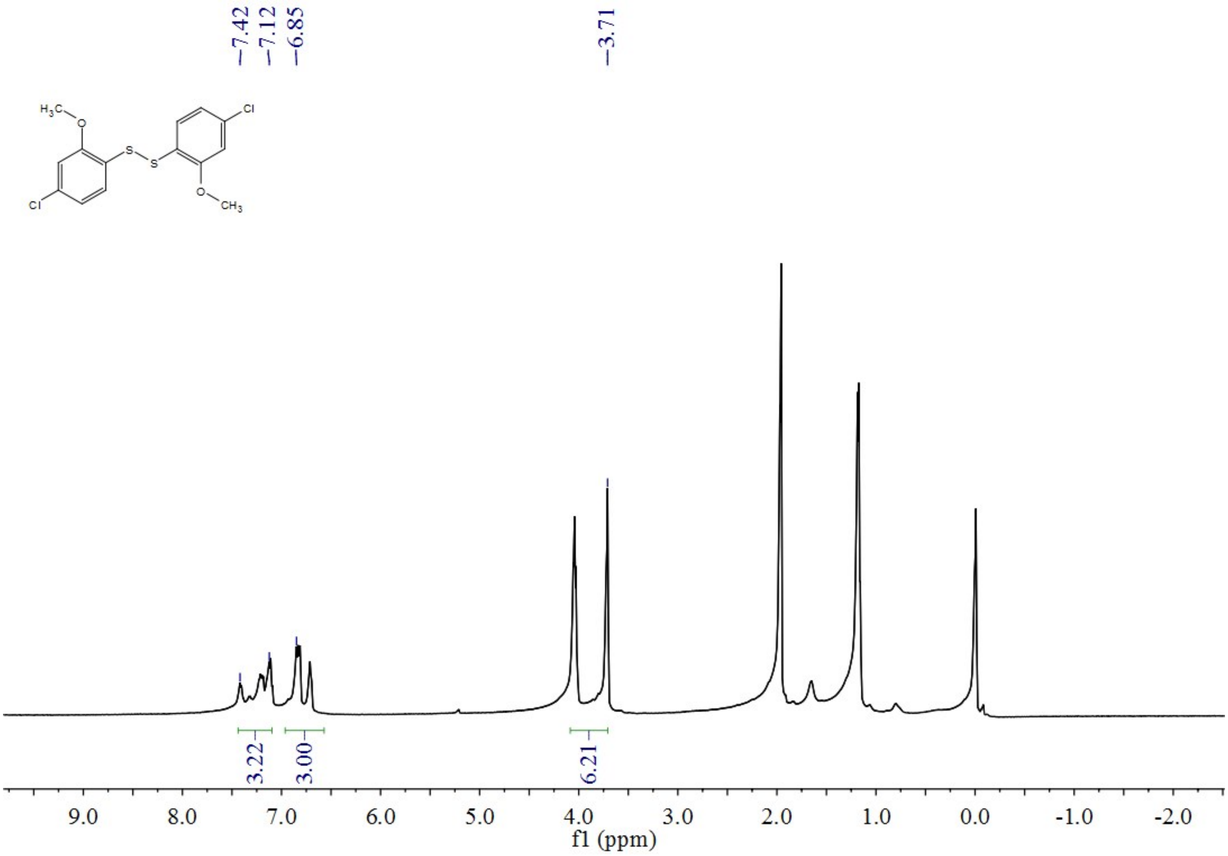
^{13}C NMR spectra of 14 (125 MHz, DMSO)



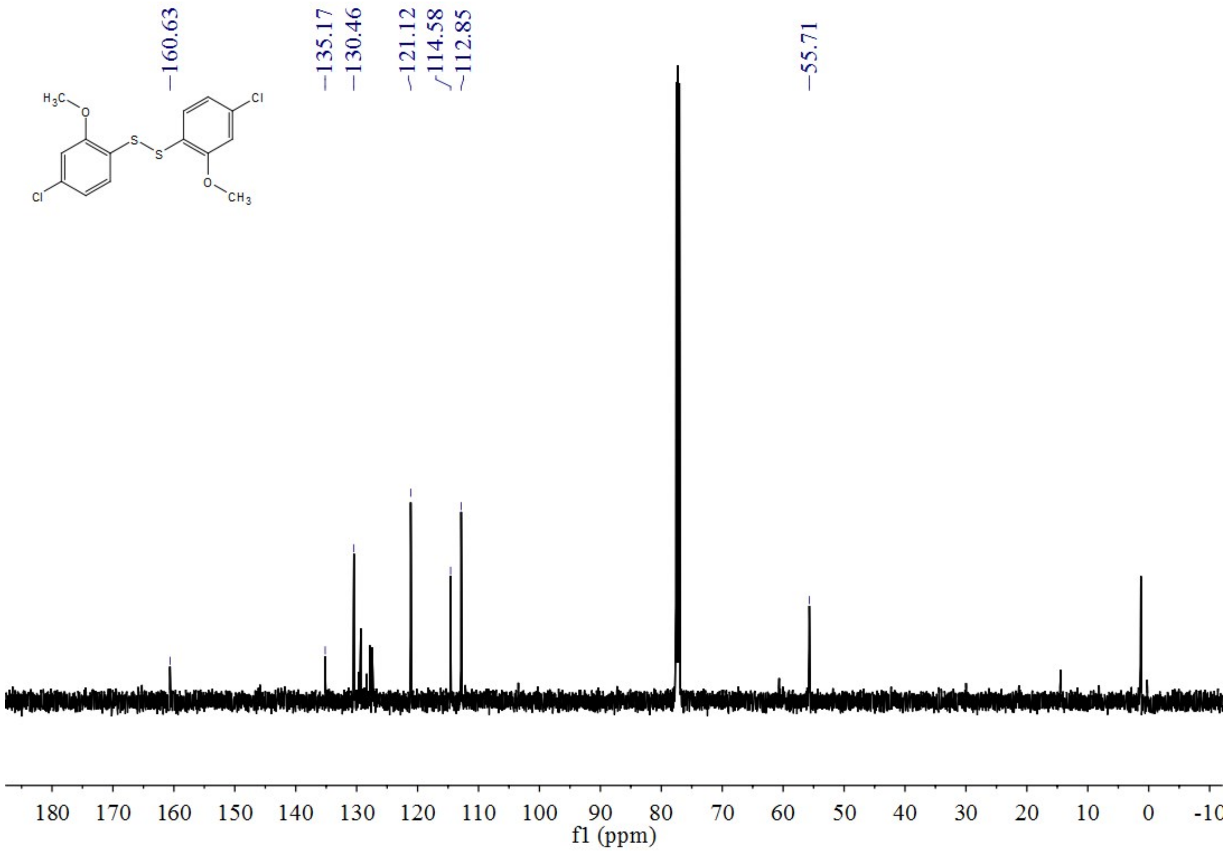
^{1}H NMR spectra of 15 (500 MHz, DMSO)



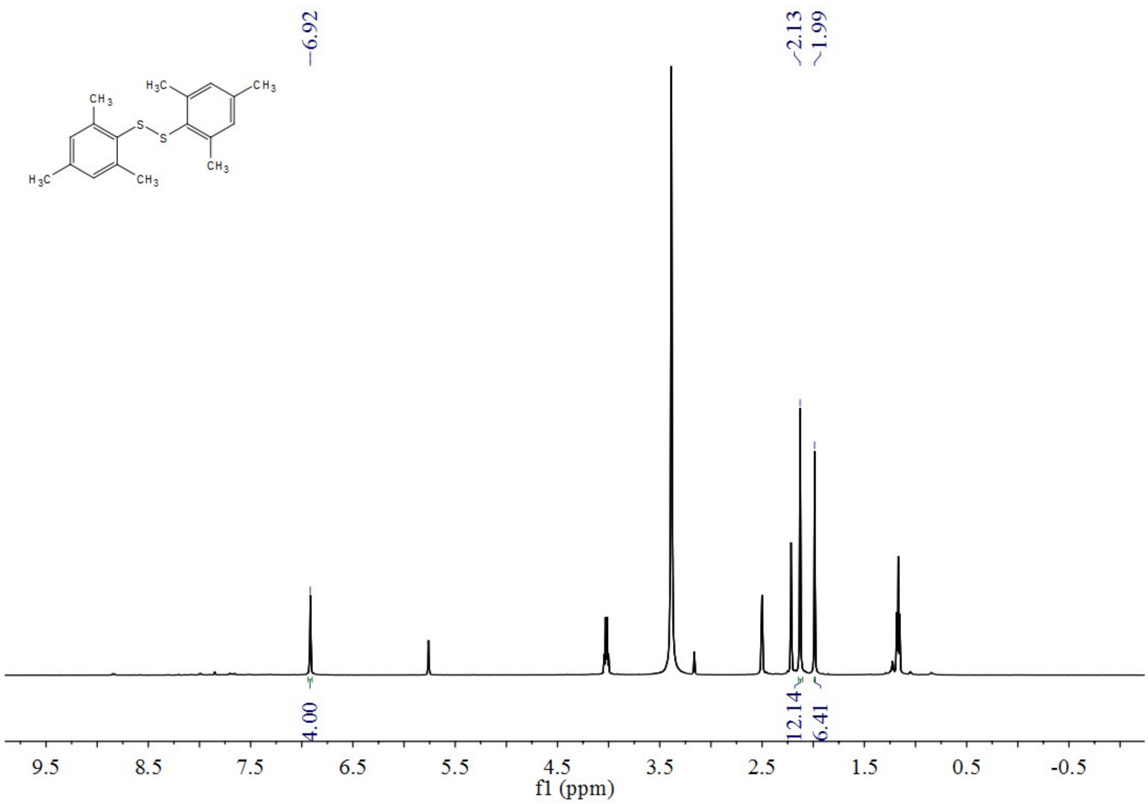
^{13}C NMR spectra of 15 (125 MHz, DMSO)



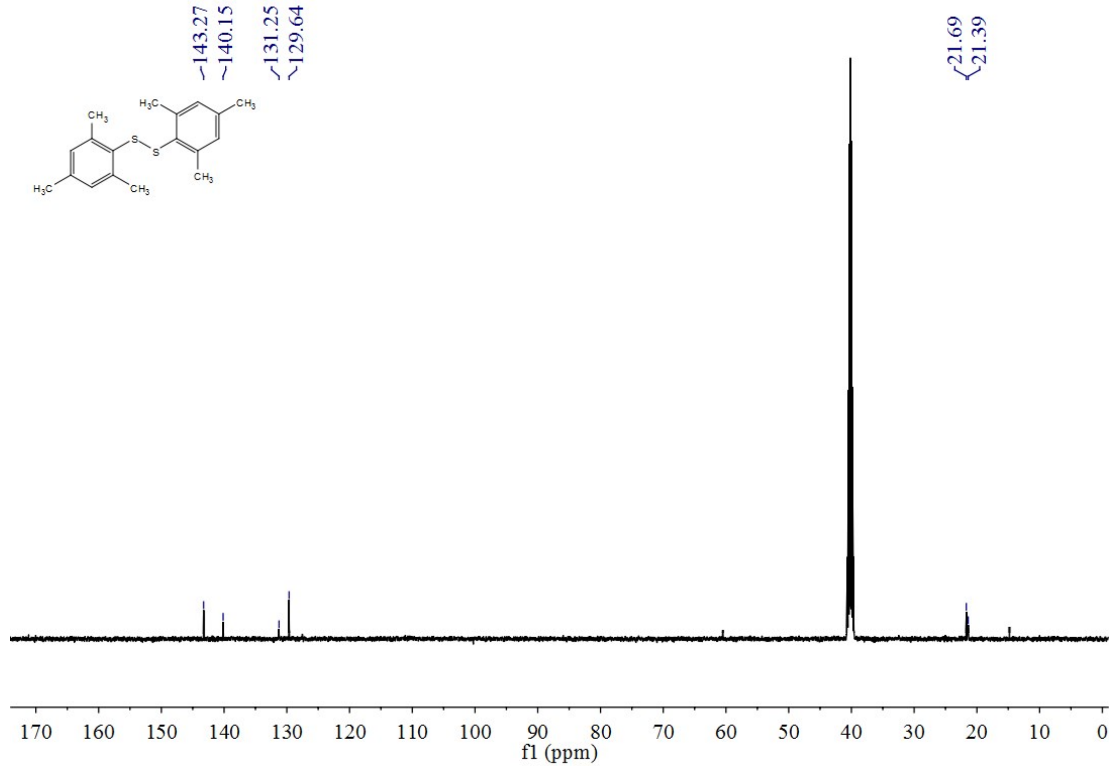
^{1}H NMR spectra of 16 (500 MHz, $CDCl_3$)



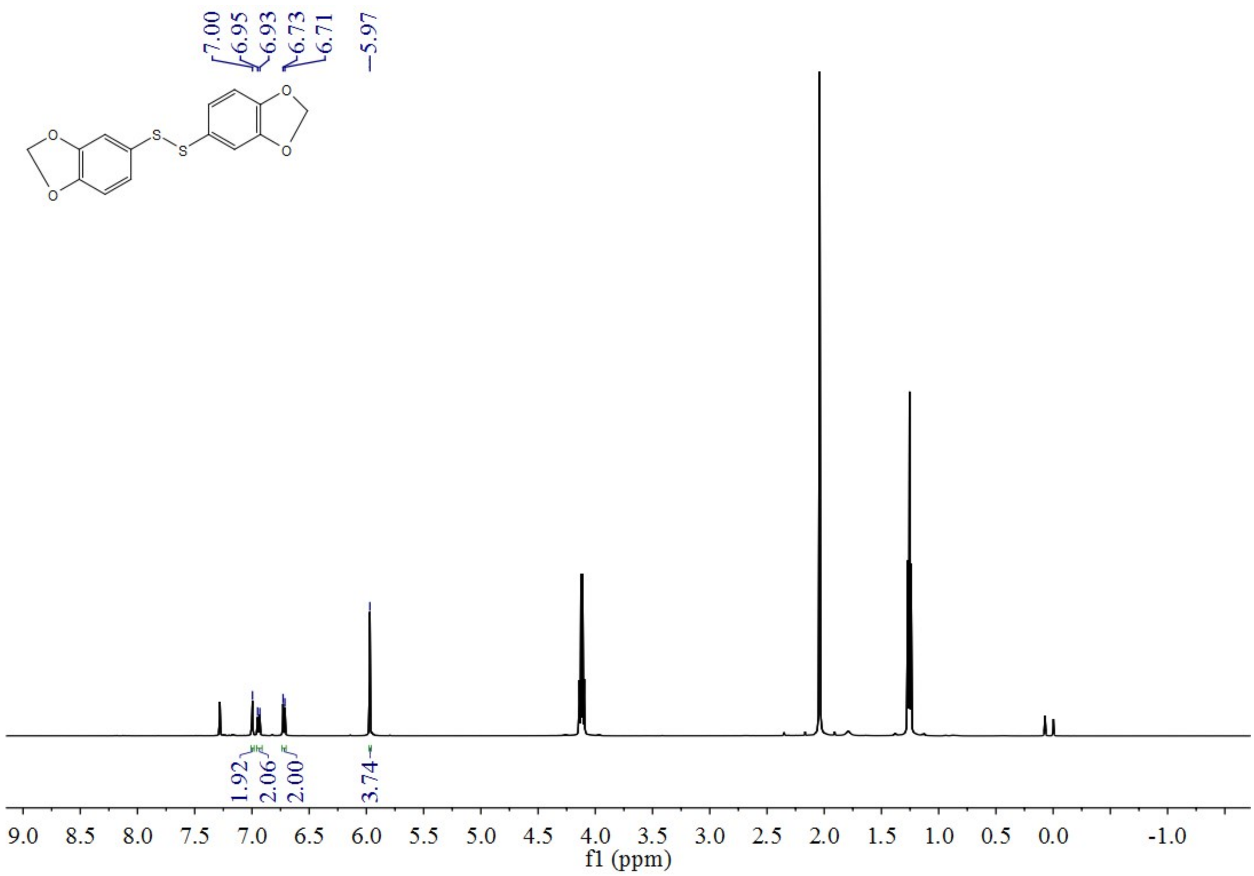
^{13}C NMR spectra of 16 (125 MHz, $CDCl_3$)



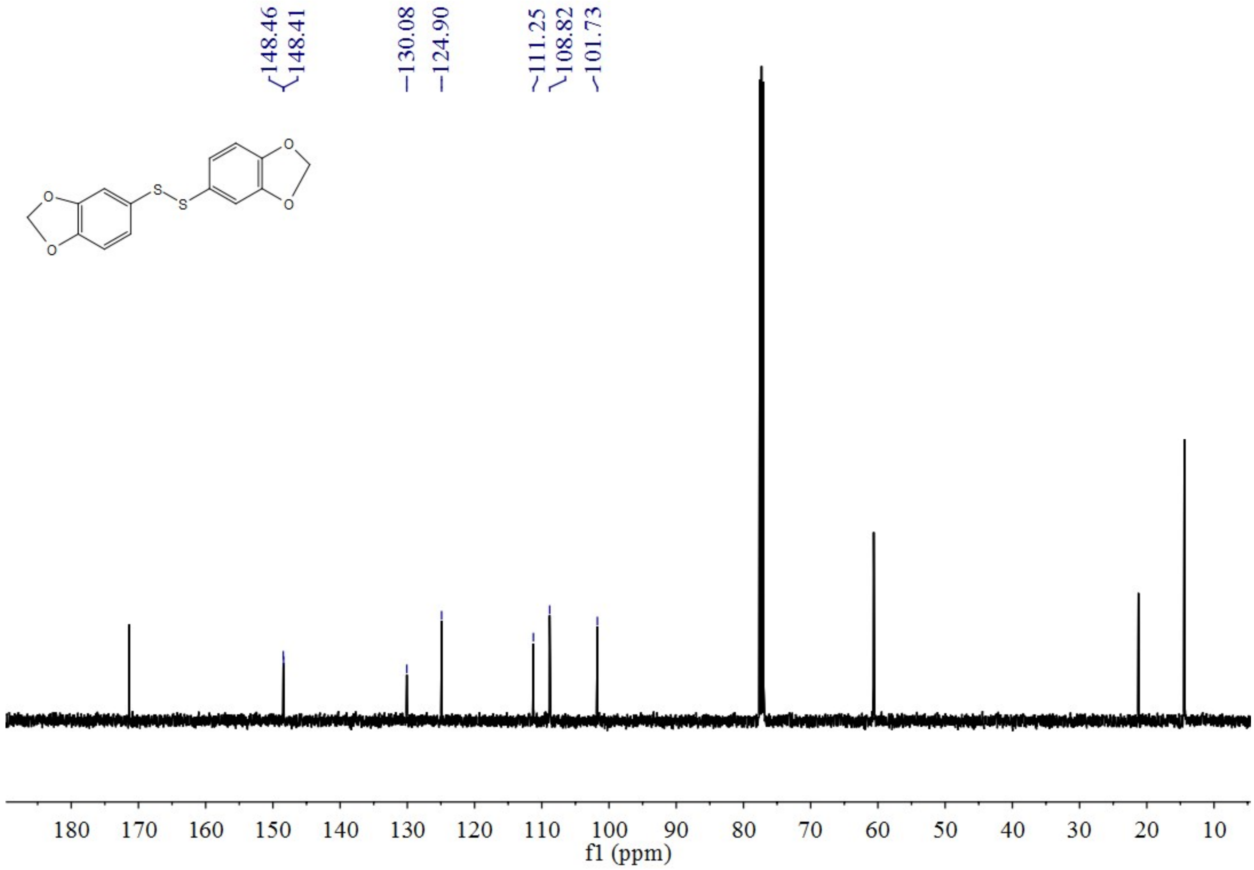
[1]H NMR spectra of 17 (500 MHz, DMSO)



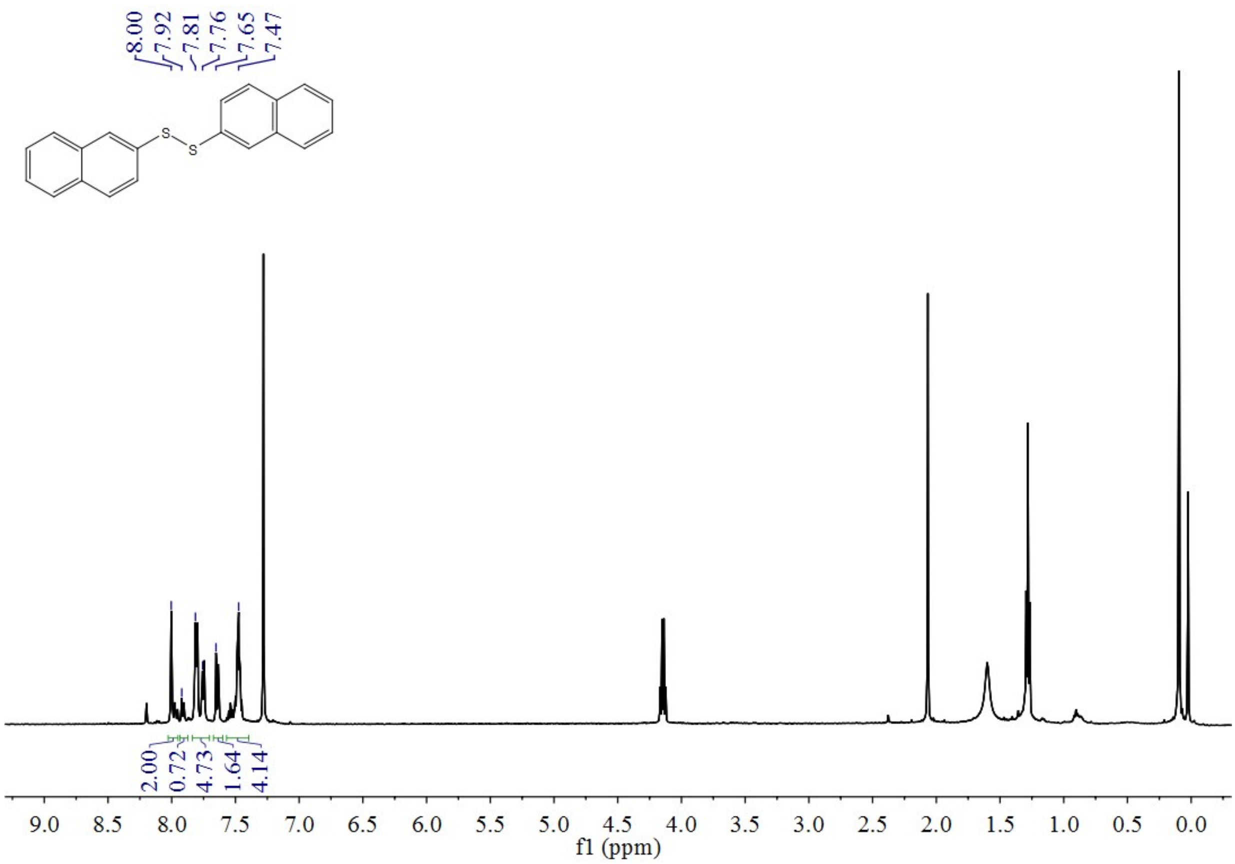
[13]C NMR spectra of 17 (125 MHz, DMSO)



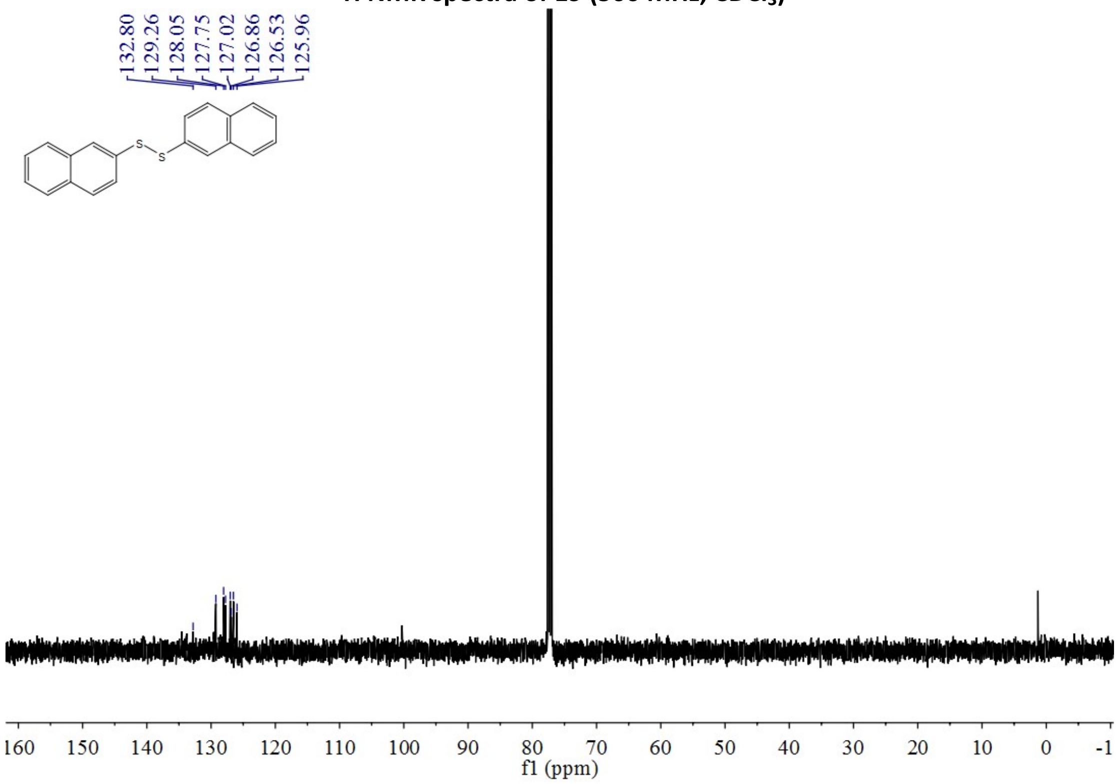
^{1}H NMR spectra of 18 (500 MHz, $CDCl_3$)



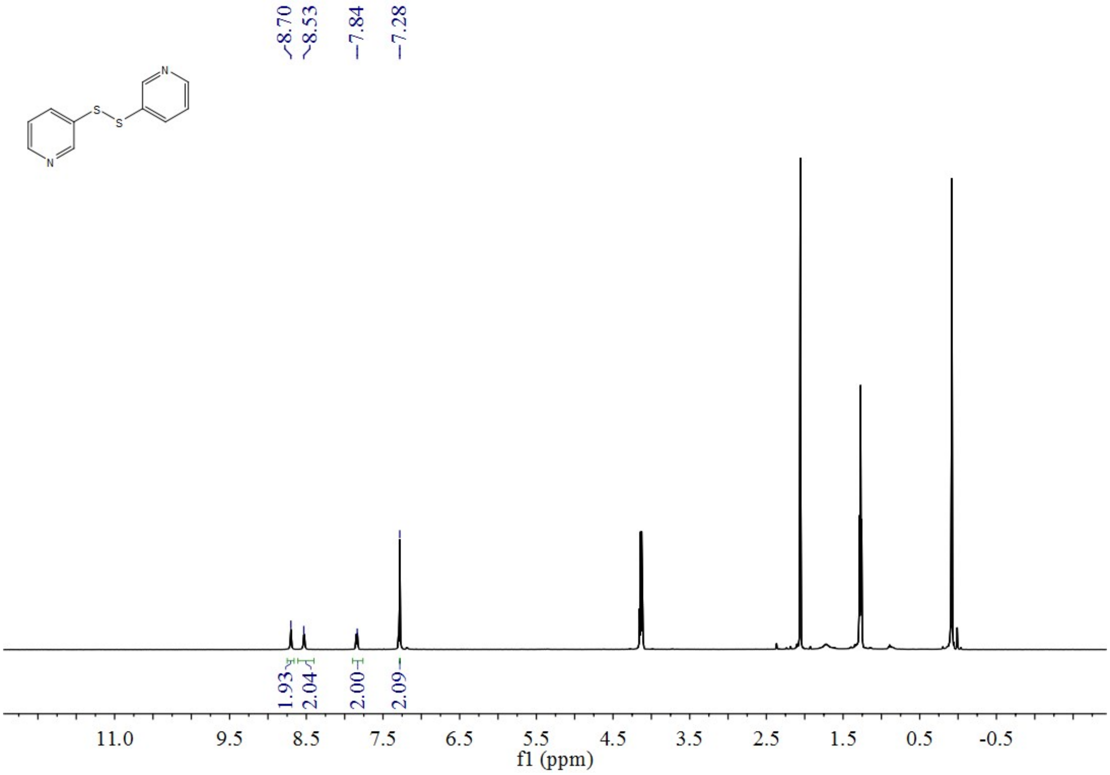
^{13}C NMR spectra of 18 (125 MHz, $CDCl_3$)



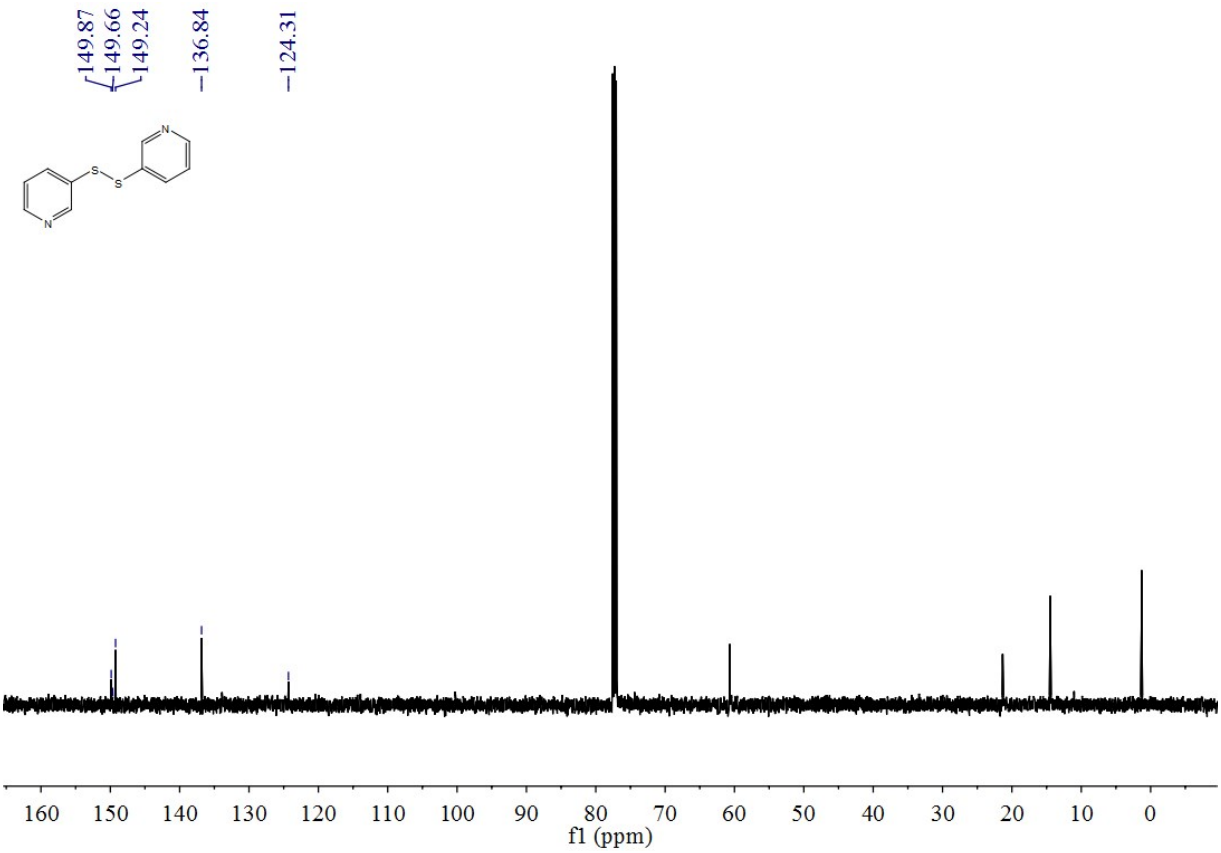
[1]H NMR spectra of 19 (500 MHz, $CDCl_3$)



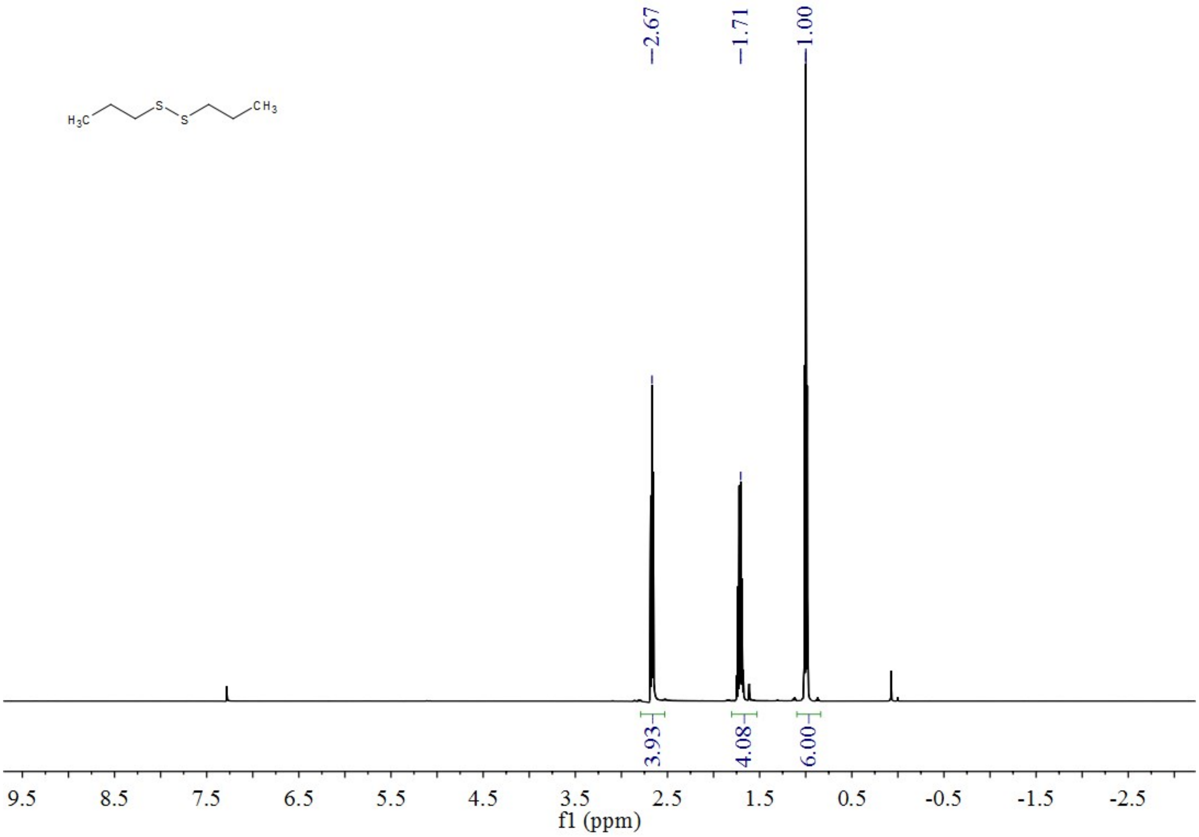
[13]C NMR spectra of 19 (125 MHz, $CDCl_3$)



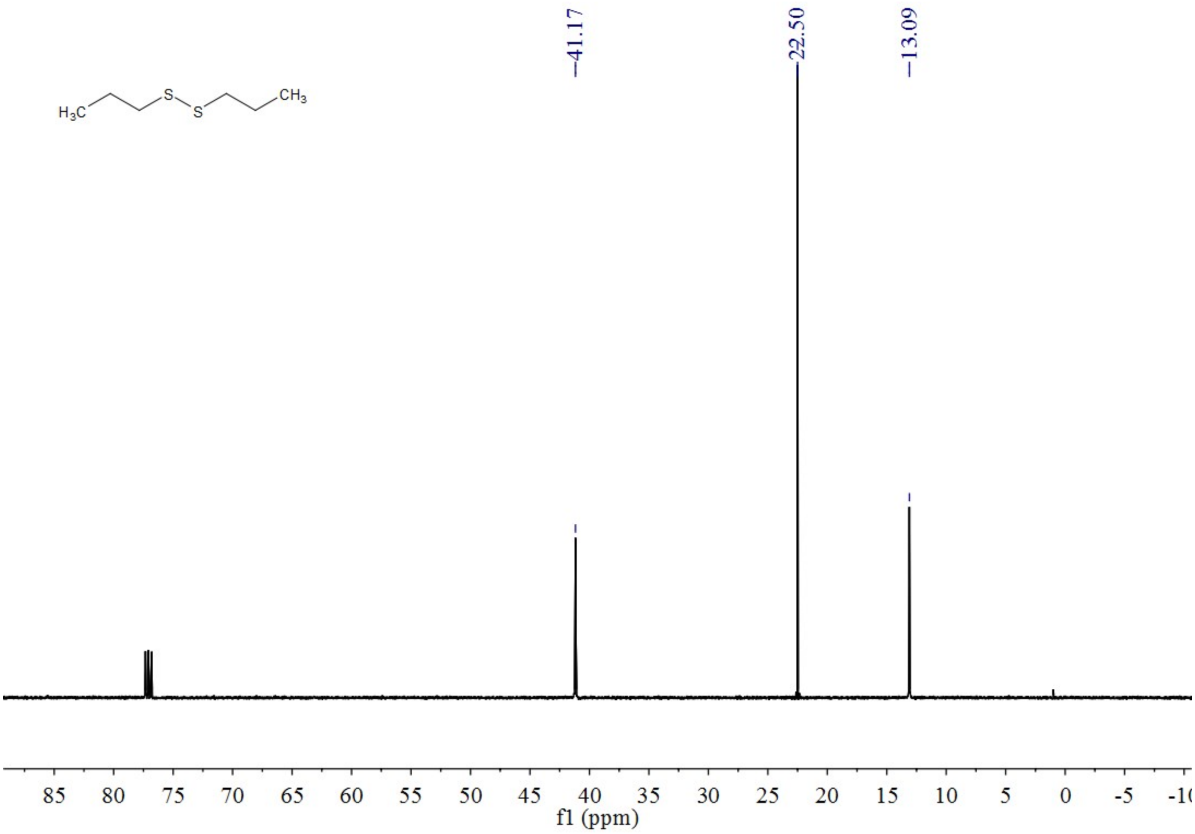
^{1}H NMR spectra of 20 (500 MHz, $CDCl_3$)



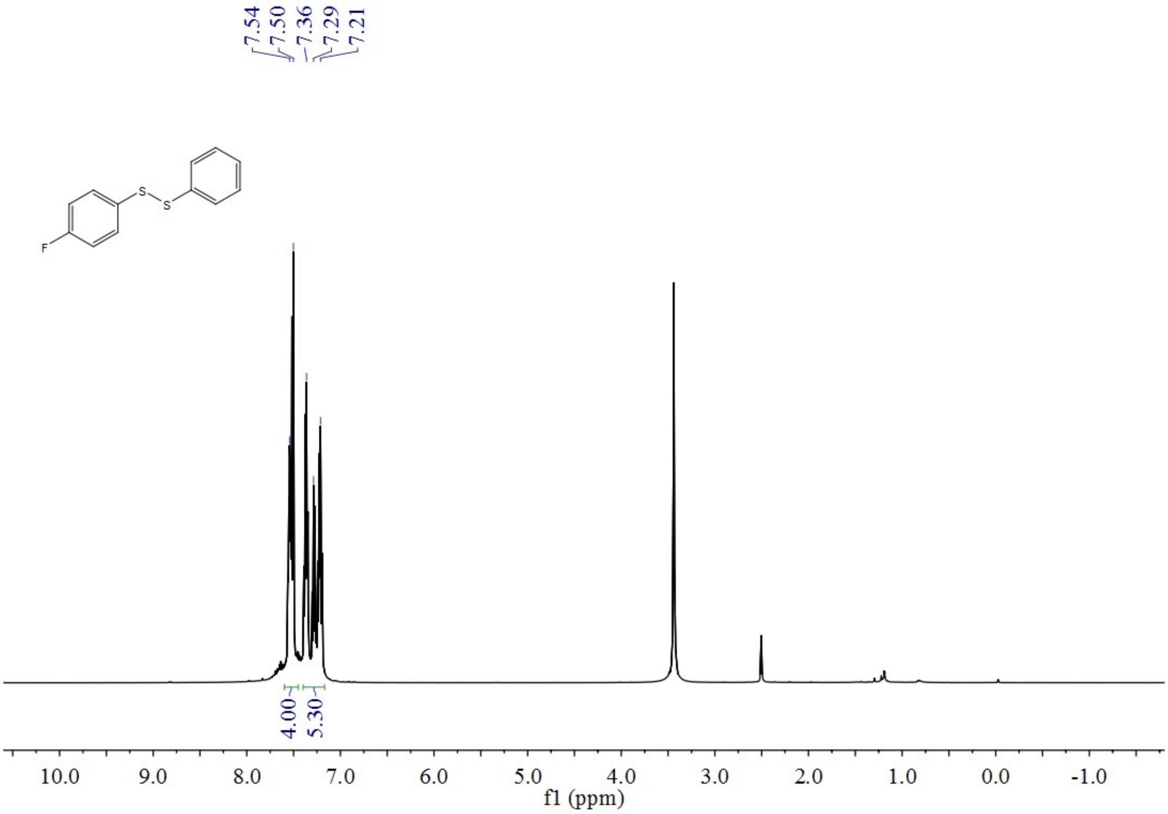
^{13}C NMR spectra of 20 (125 MHz, $CDCl_3$)



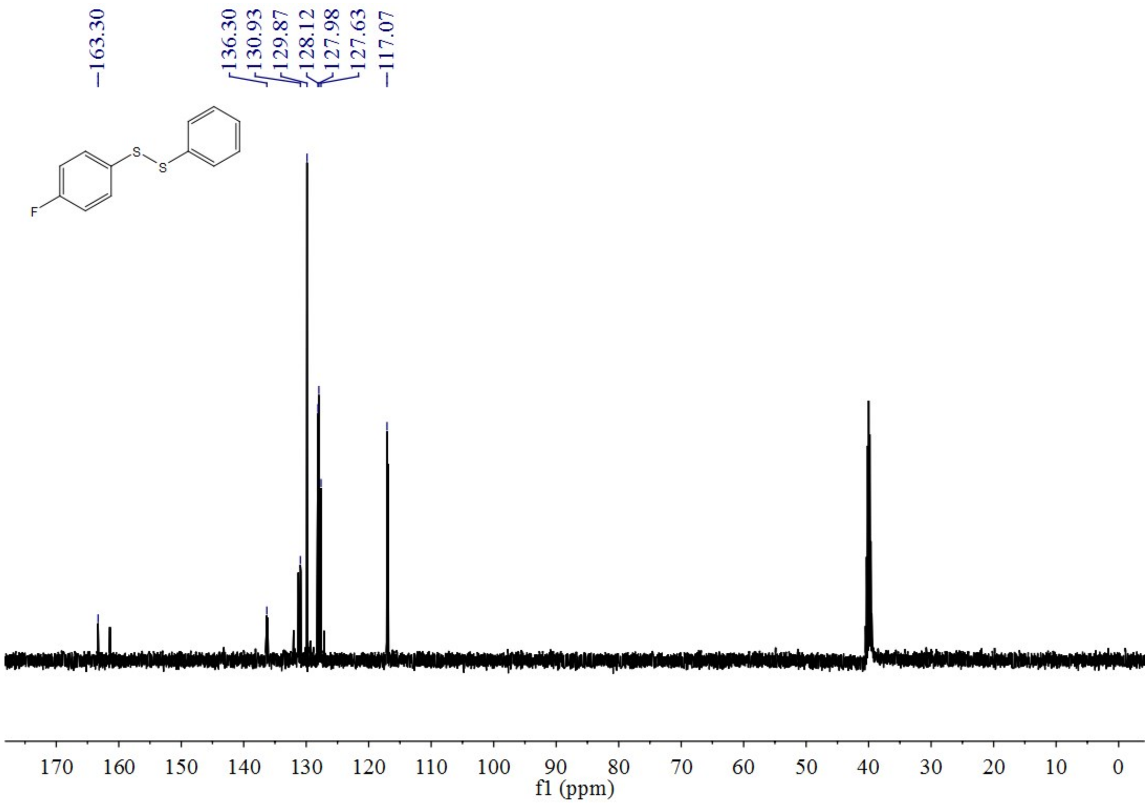
^{1}H NMR spectra of 21 (500 MHz, $CDCl_3$)



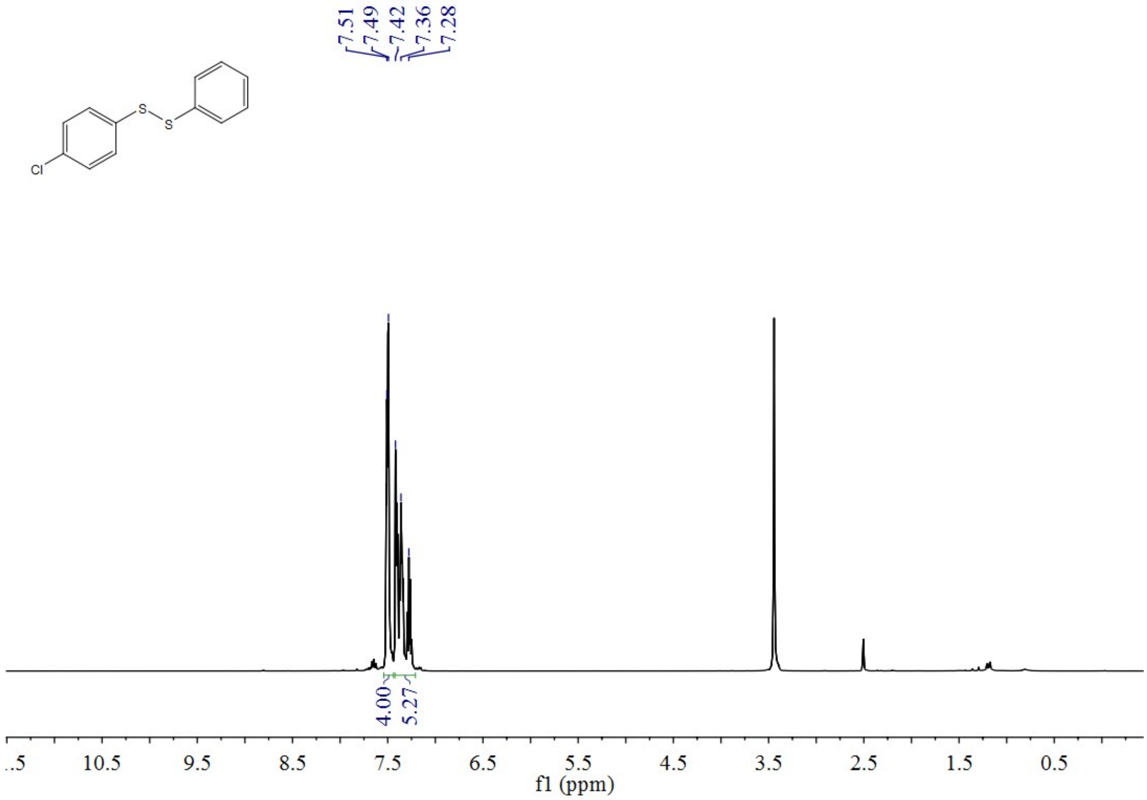
^{13}C NMR spectra of 21 (125 MHz, $CDCl_3$)



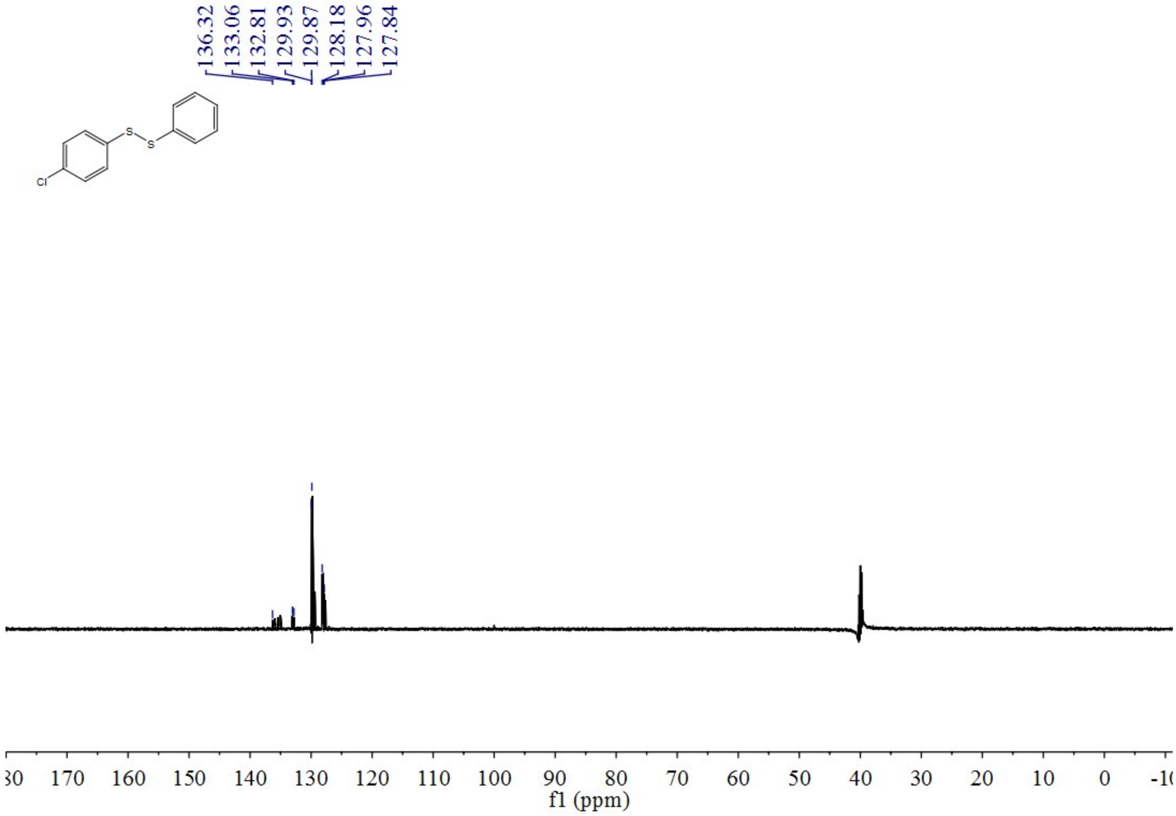
[1]H NMR spectra of 22 (500 MHz, DMSO)



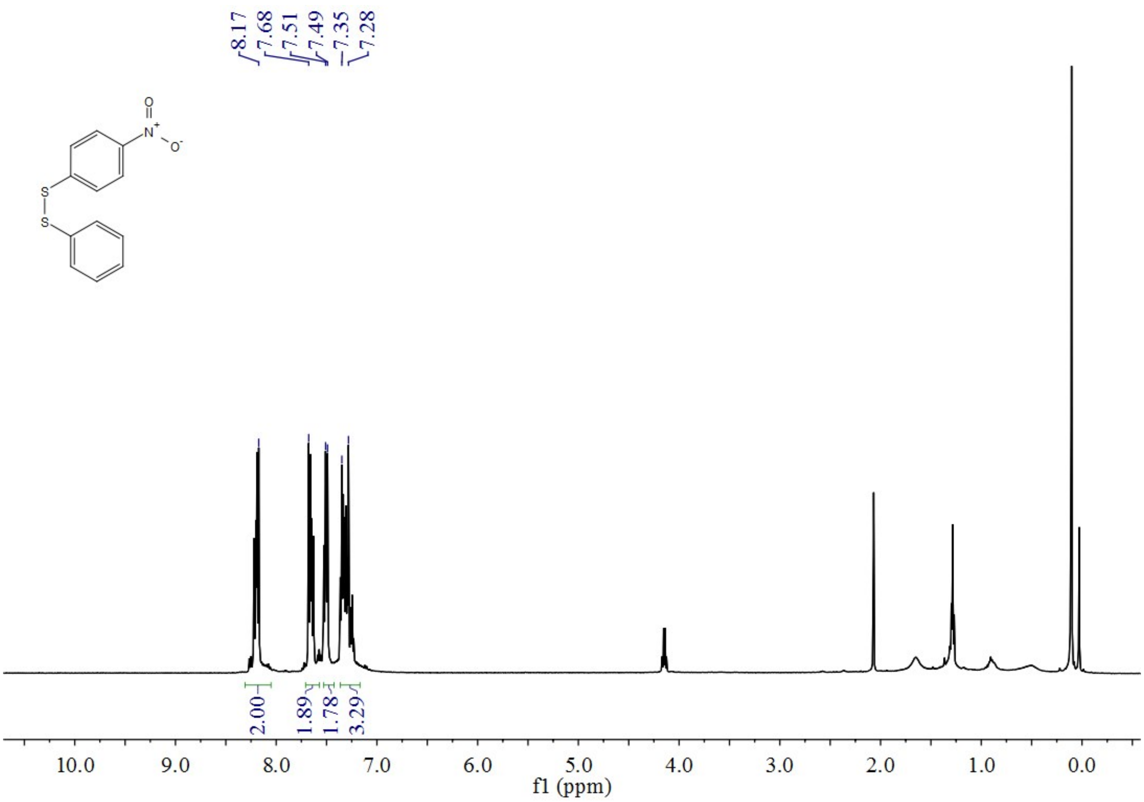
[13]C NMR spectra of 22 (125 MHz, DMSO)



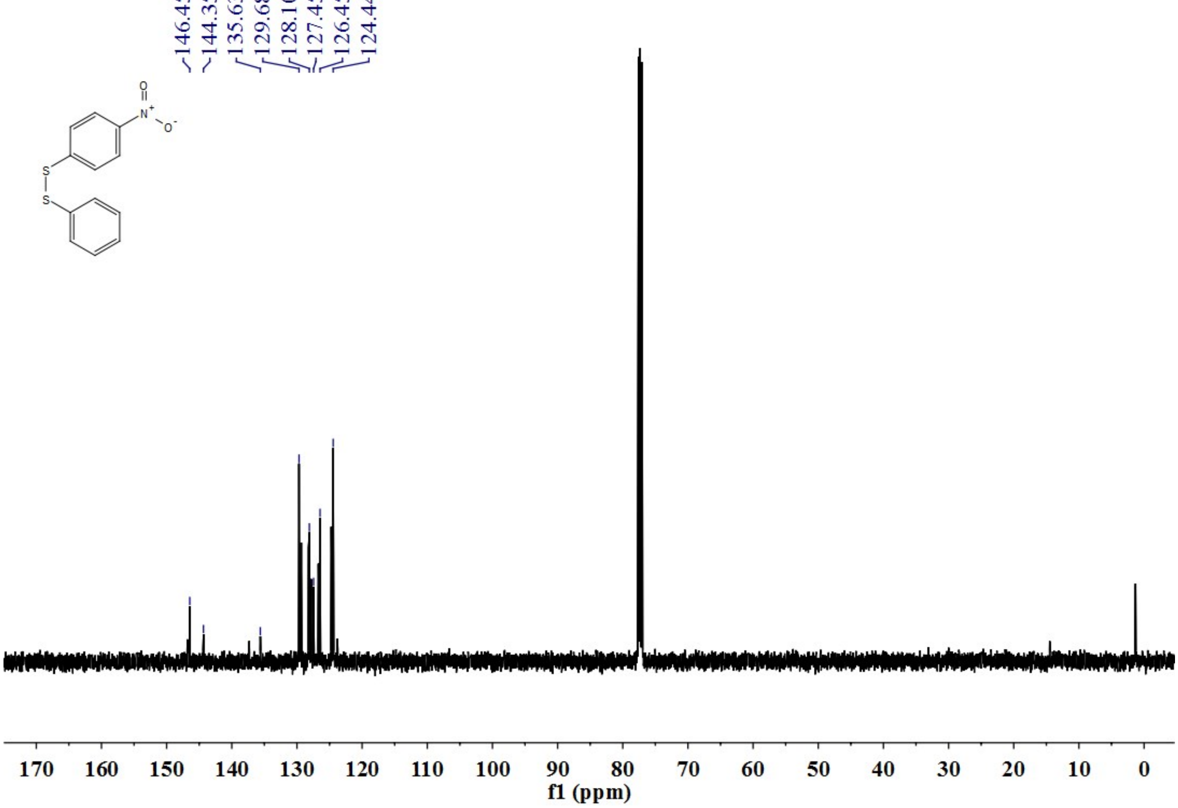
^{1}H NMR spectra of 23 (500 MHz, DMSO)



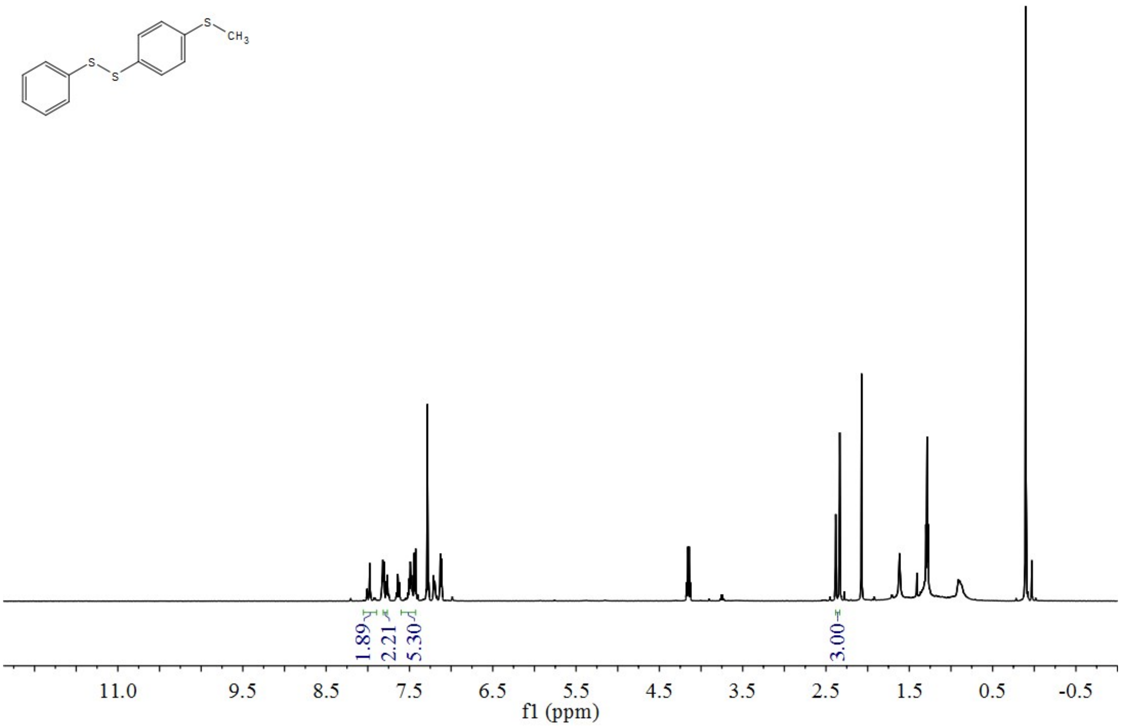
^{13}C NMR spectra of 23 (125 MHz, DMSO)



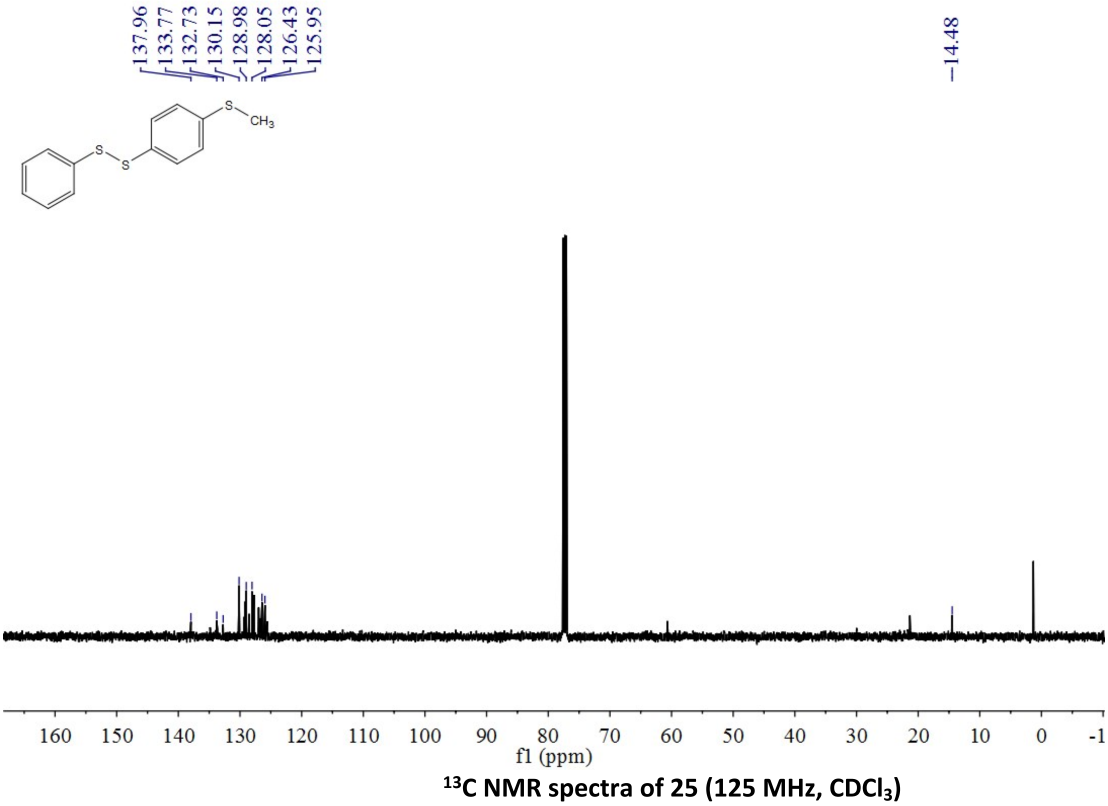
^{1}H NMR spectra of 24 (500 MHz, $CDCl_3$)



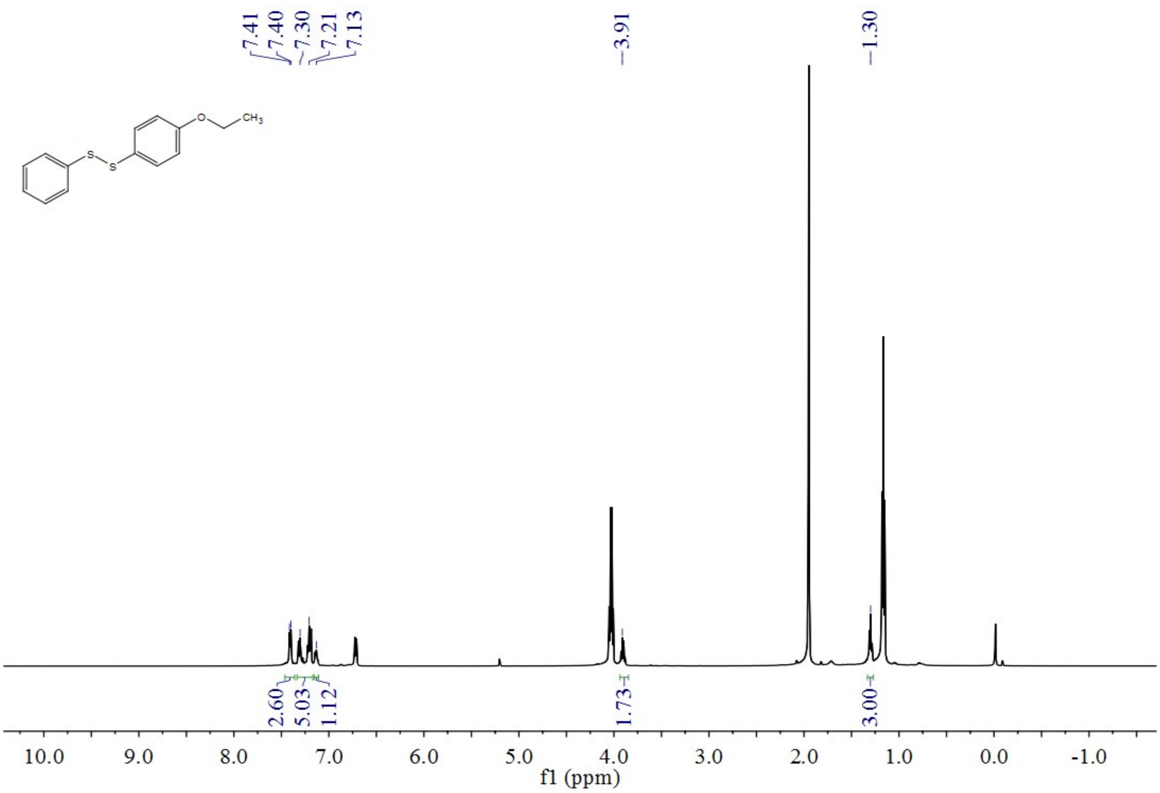
^{13}C NMR spectra of 24 (125 MHz, $CDCl_3$)



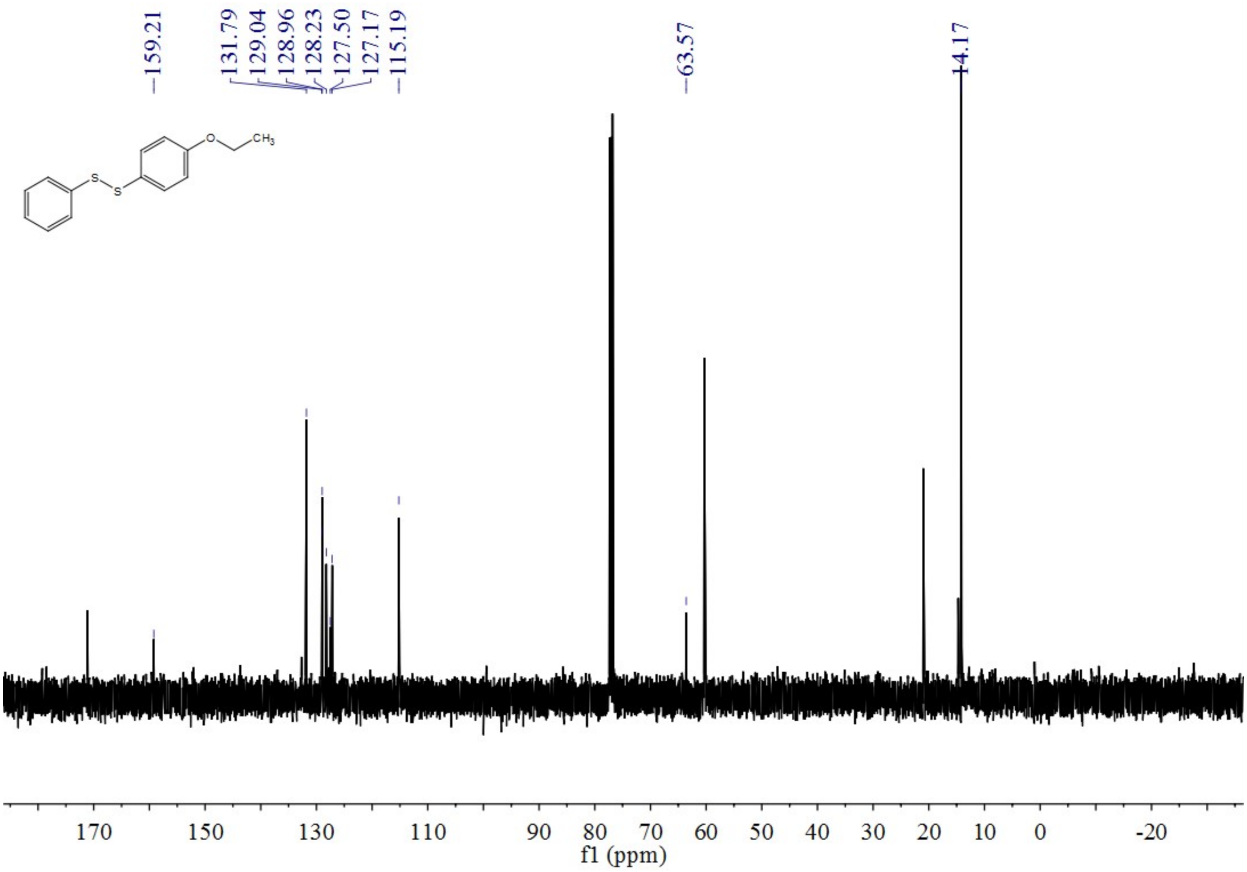
^{1}H NMR spectra of 25 (500 MHz, $CDCl_3$)



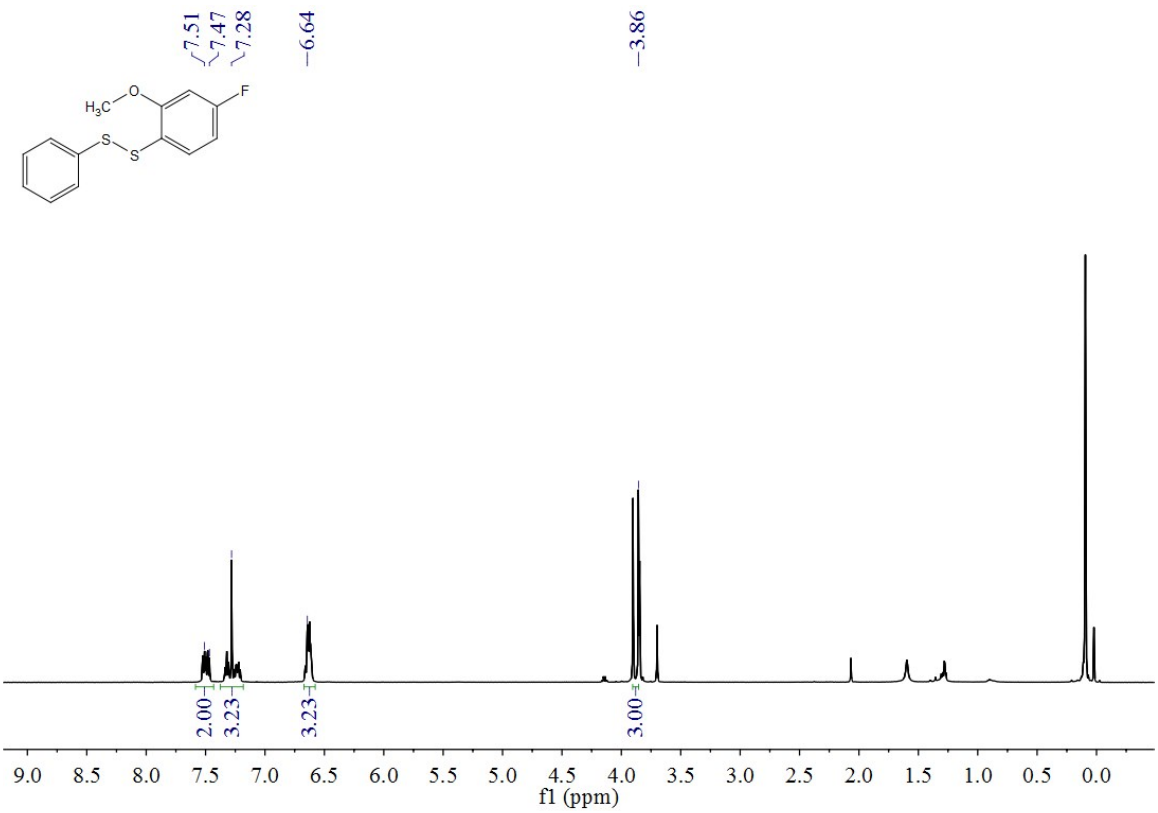
^{13}C NMR spectra of 25 (125 MHz, $CDCl_3$)



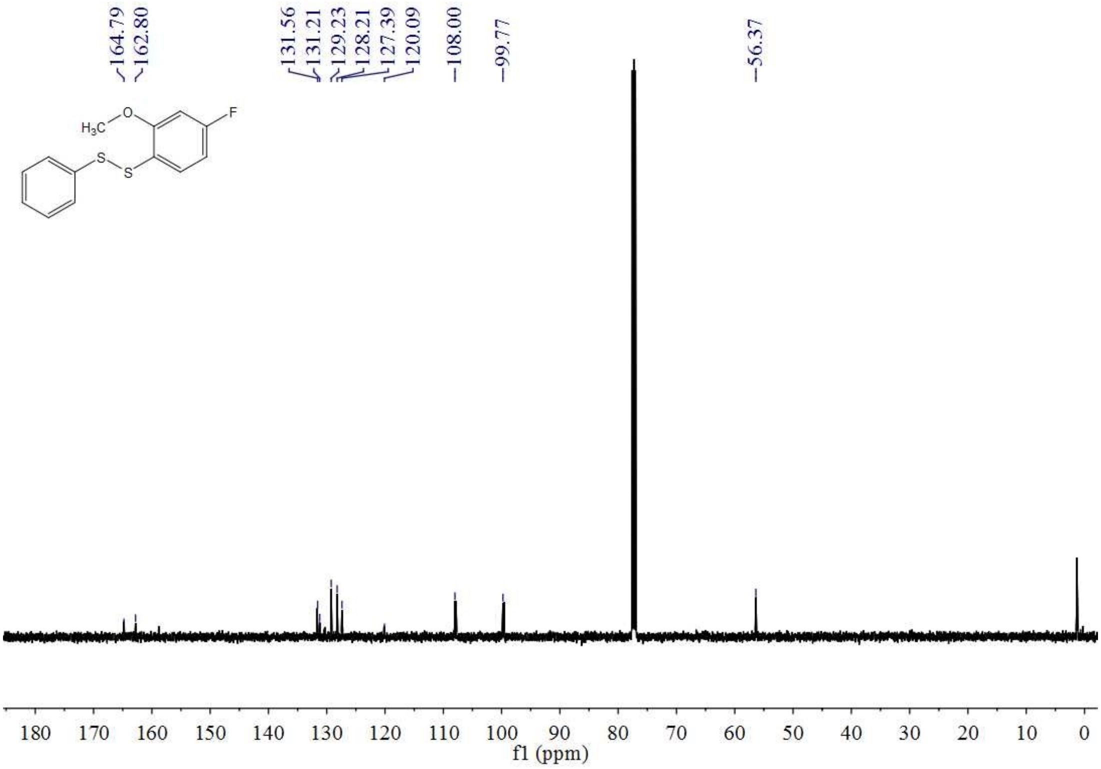
^{1}H NMR spectra of 26 (500 MHz, $CDCl_3$)



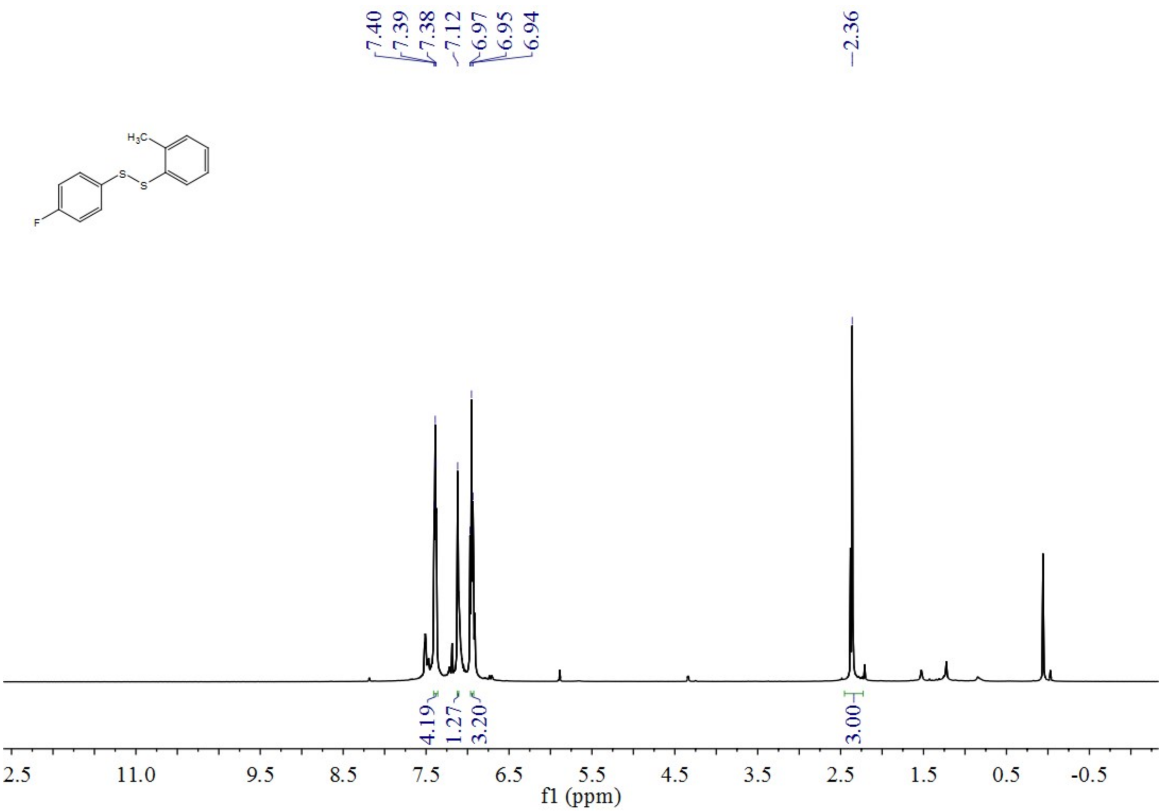
^{13}C NMR spectra of 26 (125 MHz, $CDCl_3$)



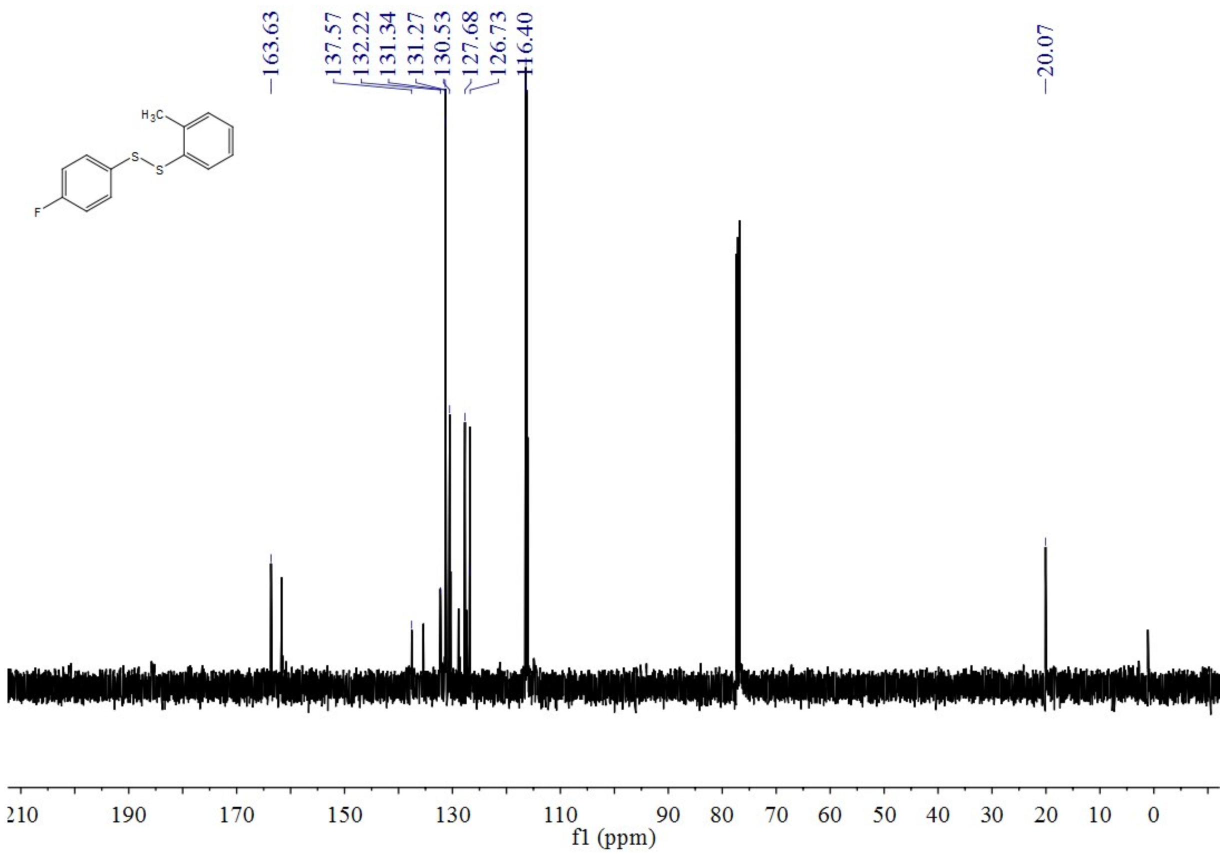
[1]H NMR spectra of 27 (500 MHz, $CDCl_3$)



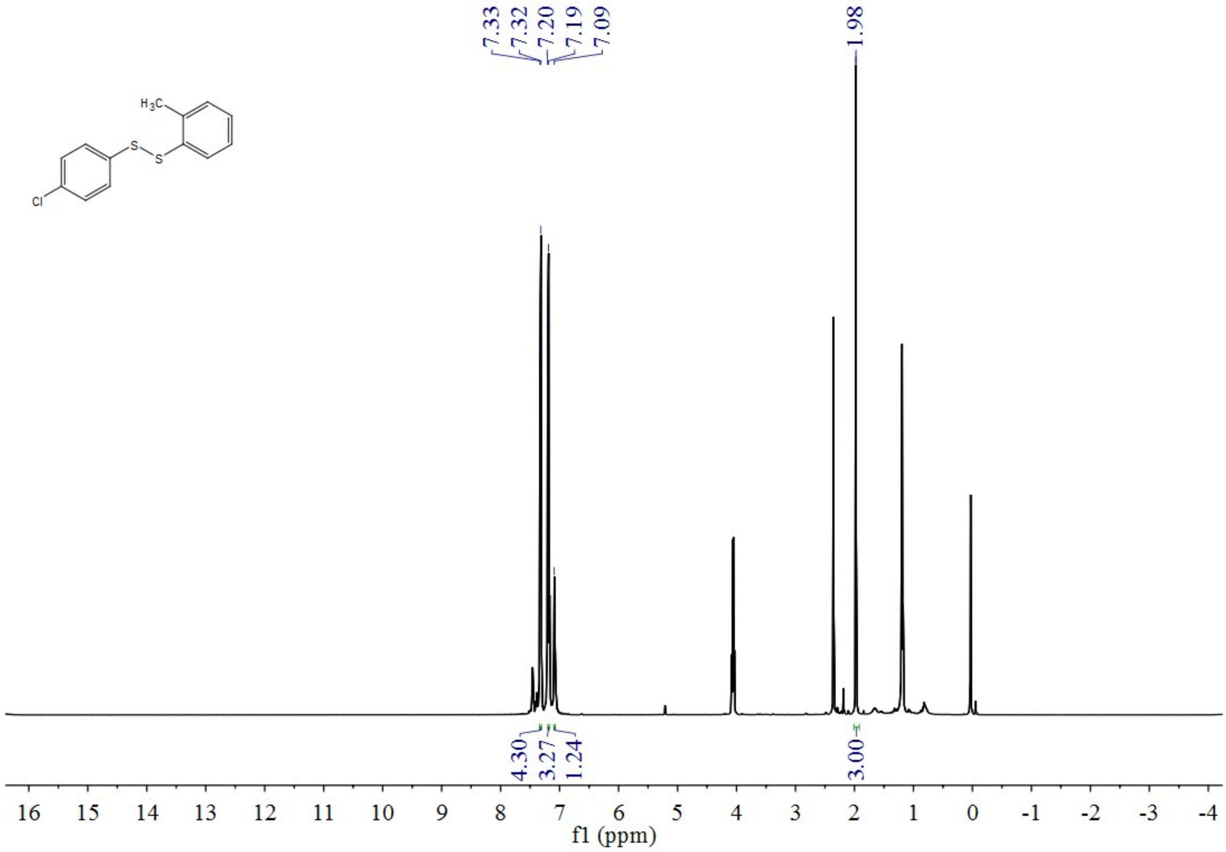
[13]C NMR spectra of 27 (125 MHz, $CDCl_3$)



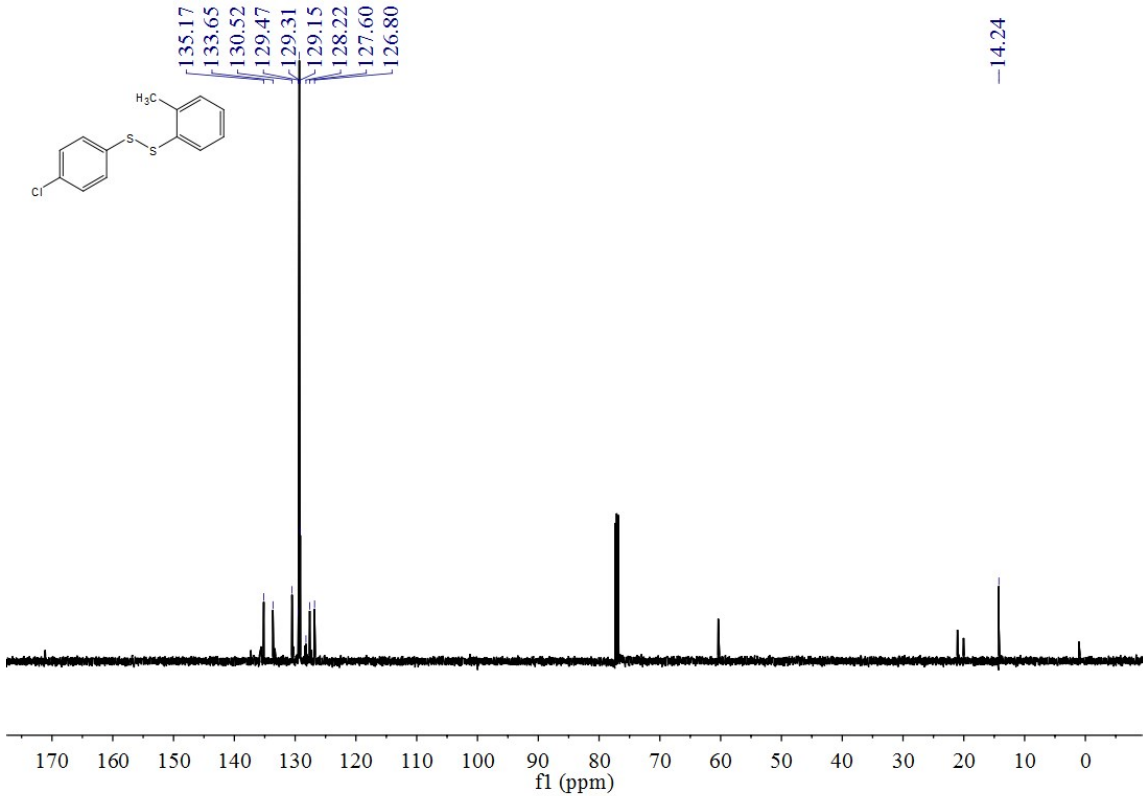
[1]H NMR spectra of 28 (500 MHz, $CDCl_3$)



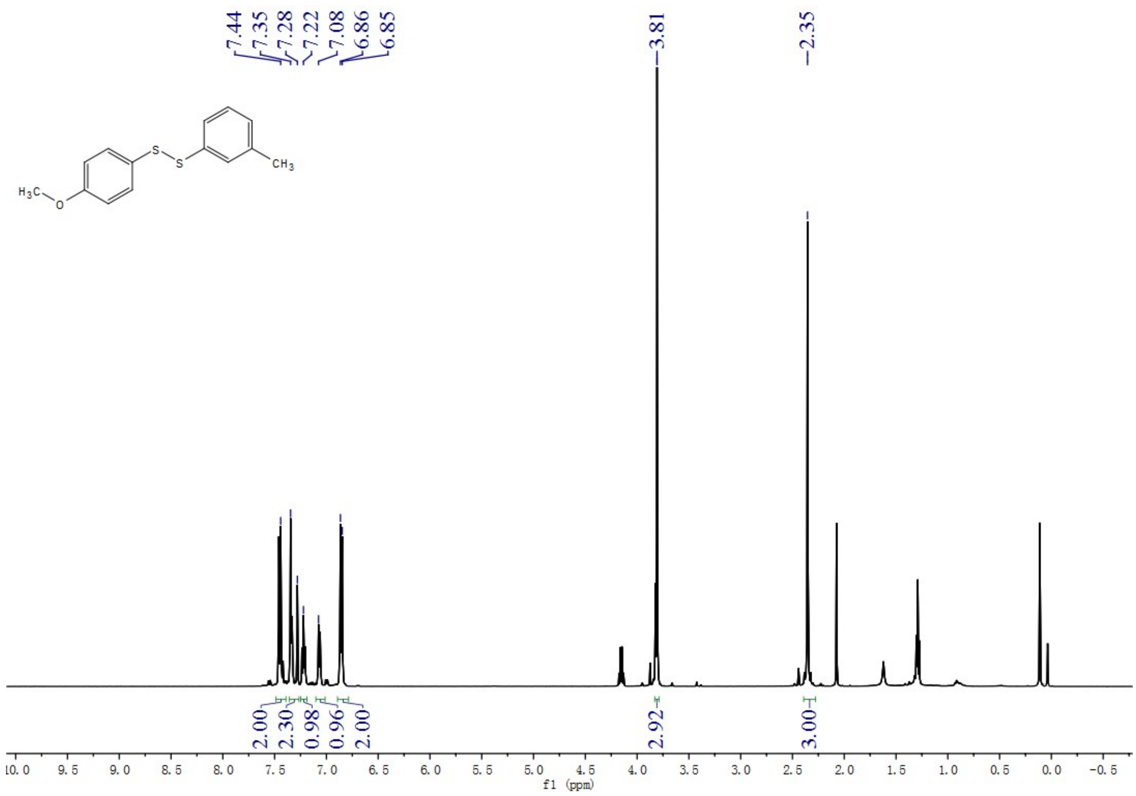
[13]C NMR spectra of 28 (125 MHz, $CDCl_3$)



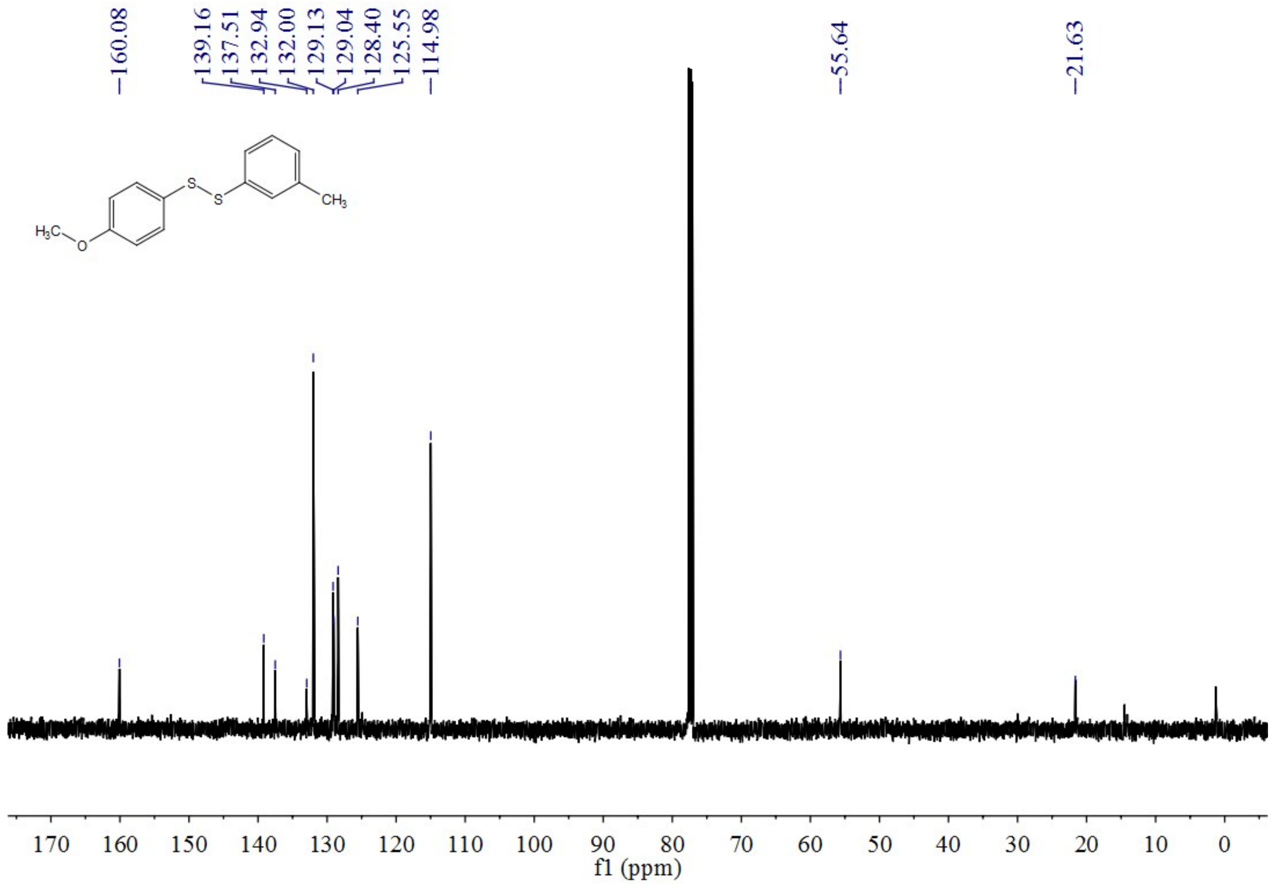
[1]H NMR spectra of 29 (500 MHz, $CDCl_3$)



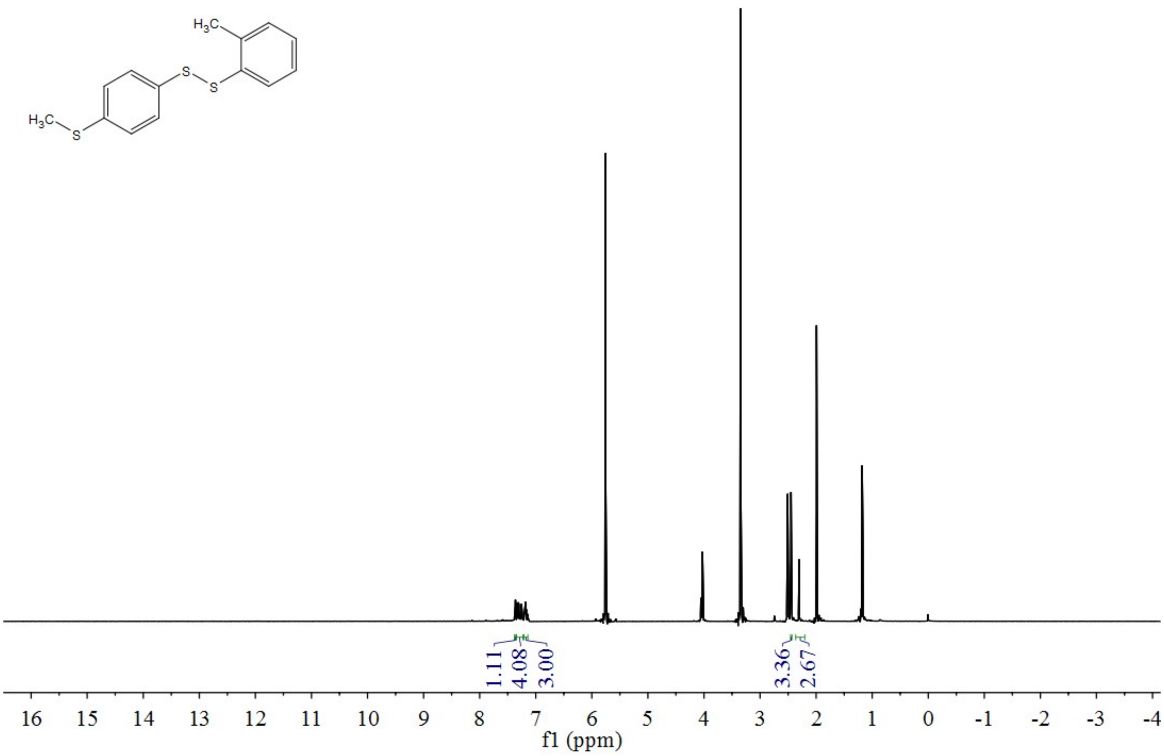
[13]C NMR spectra of 29 (125 MHz, $CDCl_3$)



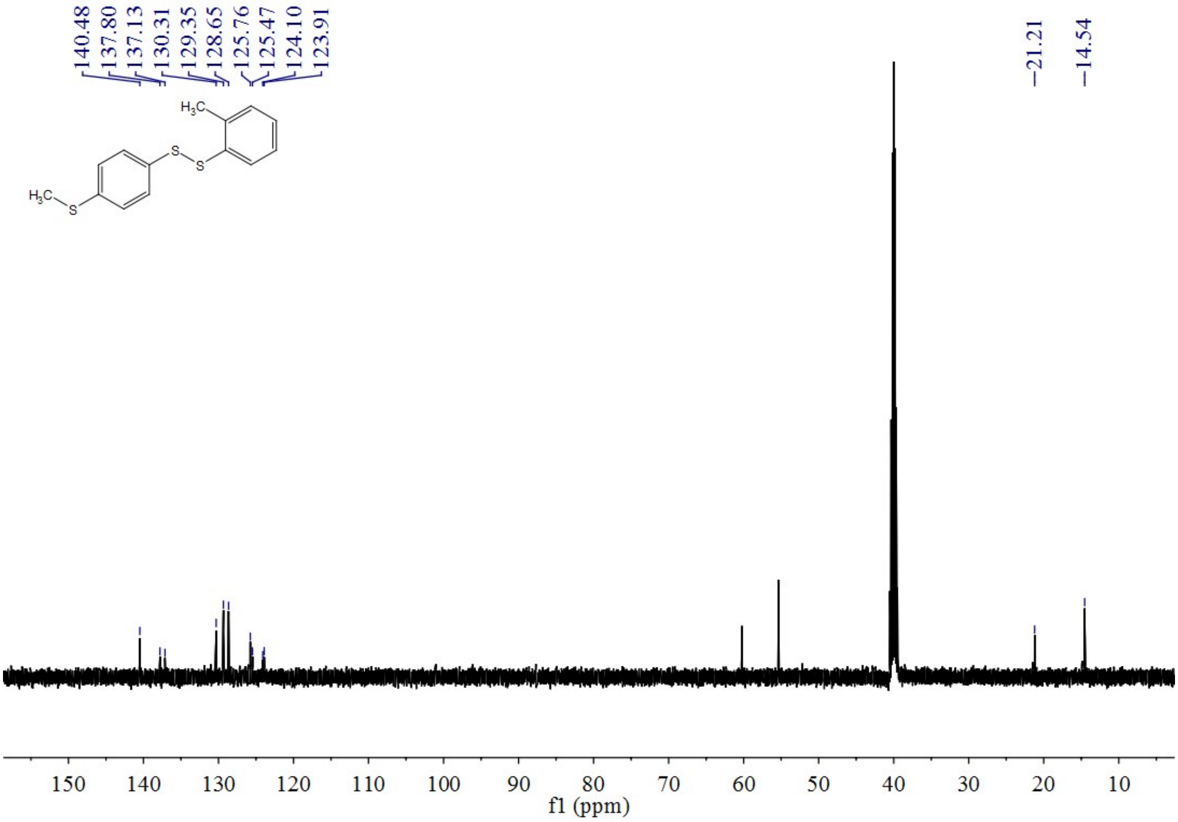
^{1}H NMR spectra of 30 (500 MHz, $CDCl_3$)



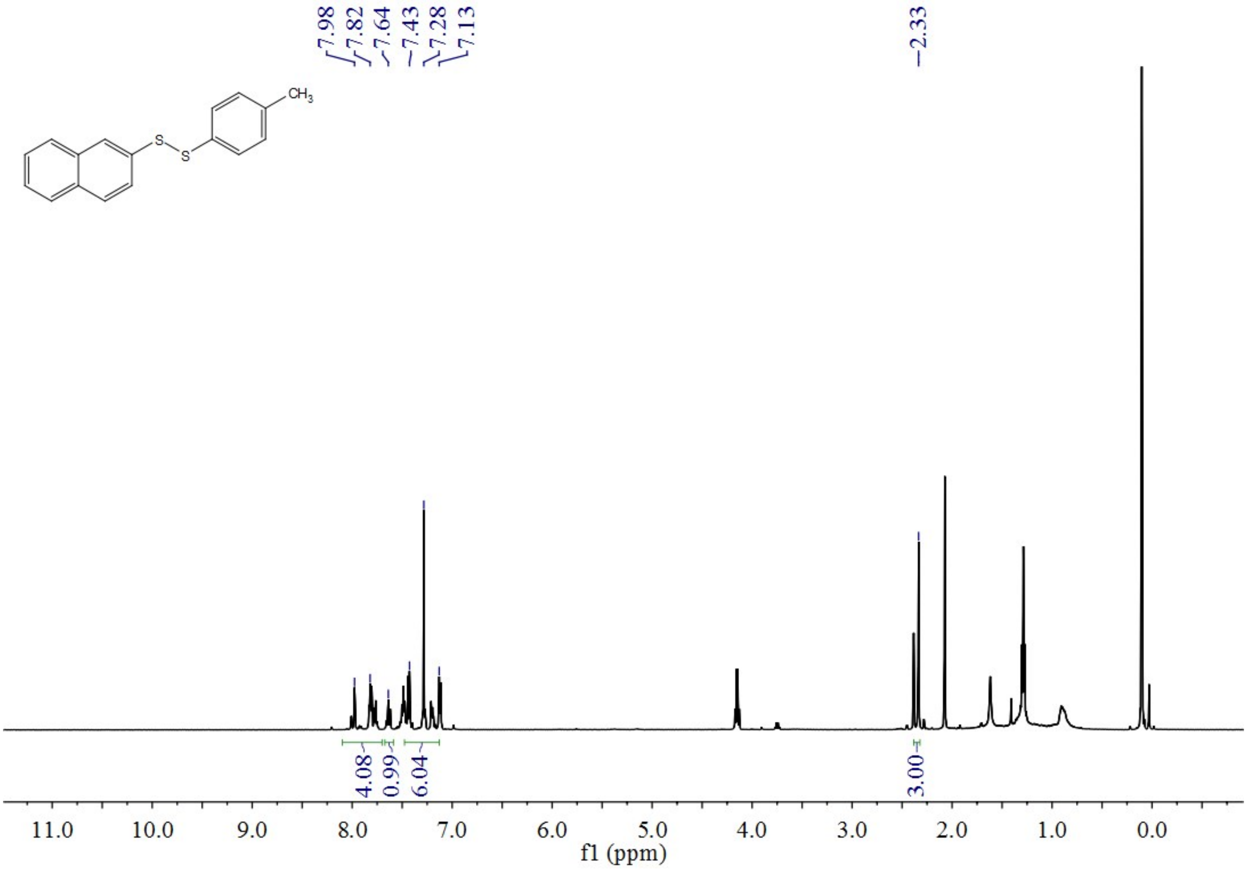
^{13}C NMR spectra of 30 (125 MHz, $CDCl_3$)



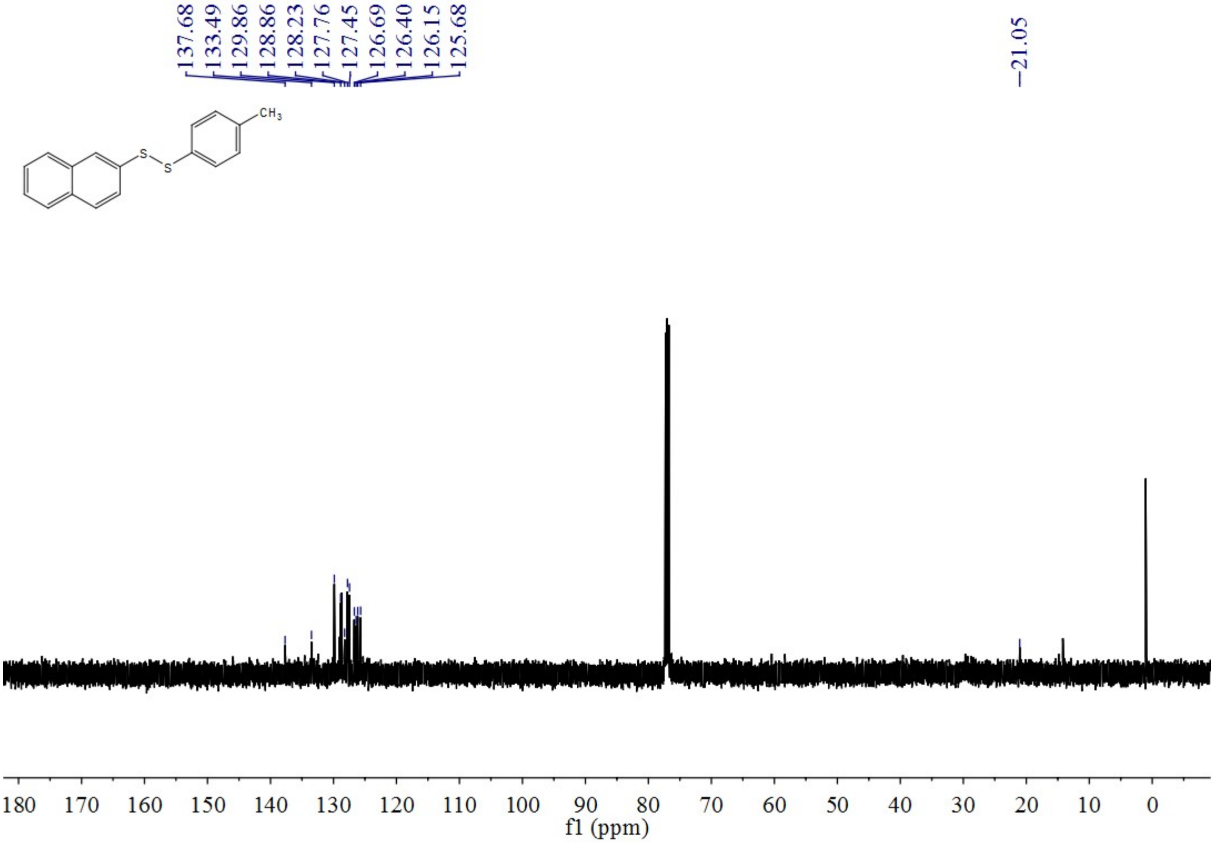
^{1}H NMR spectra of 31 (500 MHz,DMSO)



^{13}C NMR spectra of 31 (125 MHz, DMSO)



^{1}H NMR spectra of 32 (500 MHz, $CDCl_3$)



^{13}C NMR spectra of 32 (125 MHz, $CDCl_3$)

VII. Reference

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