

Supporting information for

**Sustainable synthesis of thiosulfonates and disulfides by
molybdenum-catalyzed selective oxidation of thiols**

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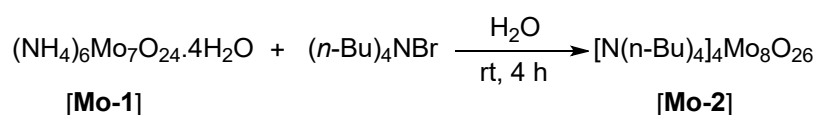
Experimental procedures and analytical data

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1. General information

The thiols, H₂O₂, (NH₄)₆Mo₇O₂₄·4H₂O, (*n*-Bu)₄NBr, all commercial reagents and metal catalyst were obtained from Energy Chemical, Sinopharm Chemical Reagent Co., Ltd and TCI, and used without further purification. ¹H, ¹³C and ¹⁹F NMR spectra were recorded on Varian 400 MHz NMR spectrometer using CDCl₃ as solvent and TMS as an internal standard. Chemical shifts are reported in parts per million (ppm, δ). Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF 254), and were performed under UV light (254 nm). Liquid chromatography was performed using forced flow (flash chromatography) on silica gel (SiO₂, 200 × 300 Mesh) purchased from Huanghai Chemical Co., Ltd. All the new products were further characterized by high resolution mass spectra (ESI-QTOF). IR spectra were recorded on a Nicolet iS10 FT-IR spectrophotometer (KBr pellets) over the region of 400-4000 cm⁻¹. The Raman spectra were obtained on ThermoFisher DXR2xi with an excitation wavelength of 633 nm. The UV/vis was tested on SHIMADZU UV2550 over the region of 800-200 nm. The cyclic voltammetry (CV) were tested on Metnohm AUT128N.V. Gas Chromatography (GC) was tested on Agilent 7890B.

2. General procedure for preparation of molybdenum catalyst ^[1]



To a round-bottomed flask (500 mL) was added (NH₄)₆Mo₇O₂₄·4H₂O (18.5g, 15mmol) and H₂O (200 mL), and then a solution of (*n*-Bu)₄NBr (17g, 53 mmol) in 100 mL H₂O was added dropwise within 1 hour. The reaction mixture was stirred at 25 °C for 3 h and the formed precipitate was filter and dried under vacuum to afford the crude product. The crude compound was recrystallized from acetonitrile to obtain analytically pure [Mo-2] product (20 g, 72%, white solid). [N(*n*-Bu)₄]₄Mo₈O₂₆ (2168): calcd. C 35.95, H 6.74, N 2.62; found C 35.76, H 6.81, N 2.49. IR (KBr disks): 2979 (v CH, s), 2948 (v CH, s), 1633 (CN, m), 1491 (δ CH, m), 1380 (δ CH, m), 1353 (w), 1157 (w), 1069 (w), 1037 (w), 951 (v MoO, s), 925 (v MoO, s), 906 (v MoO, s), 812

(w), 800 (w), 733 (w), 664 (v MoOMo, vs), 560 (m), 522 (w) cm^{-1} . Spectral data were in accordance with the literature.^[1]

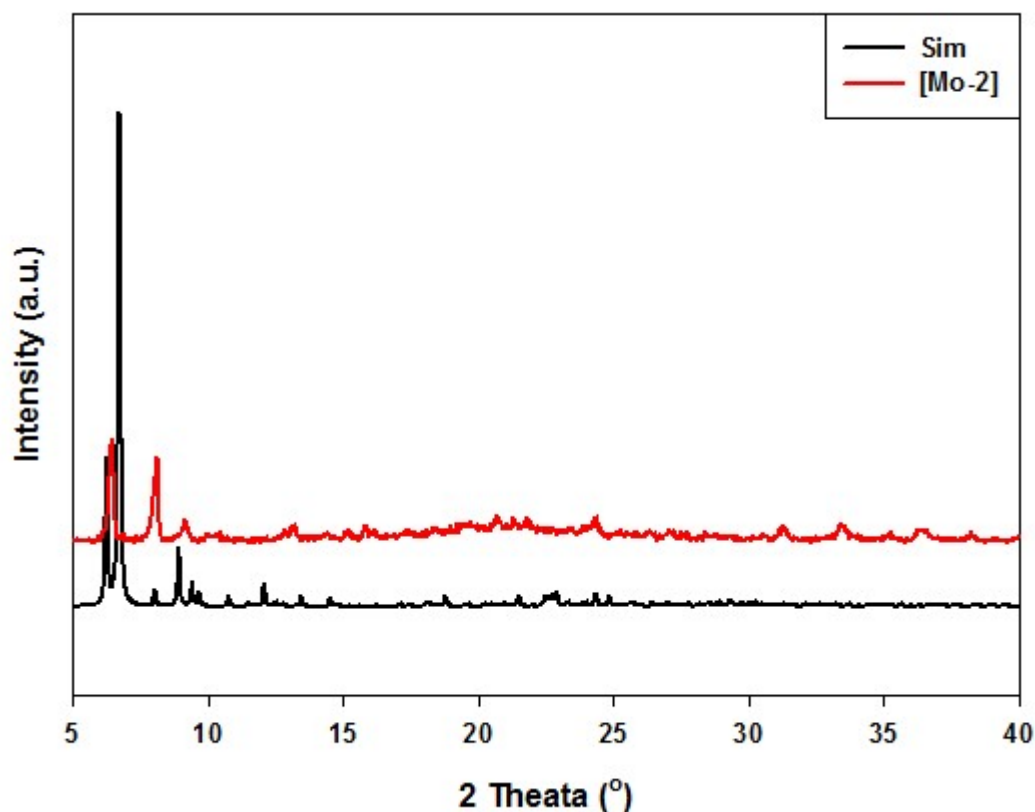
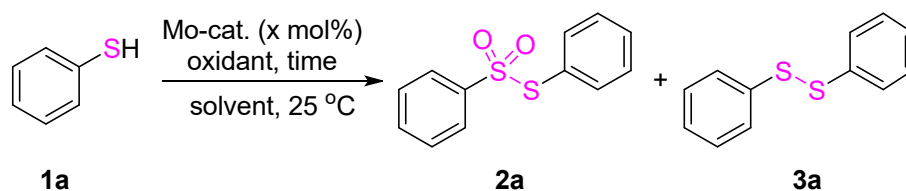


Figure S1. The experimental (red) and simulated (black) PXRD patterns for [Mo-2]. The main diffraction peaks of experimental patterns basically match with the simulated ones, confirming the crystal phase purity of compound [Mo-2].

3. Optimization of reaction conditions

Table S1. Optimization of reaction conditions for **2a**^a

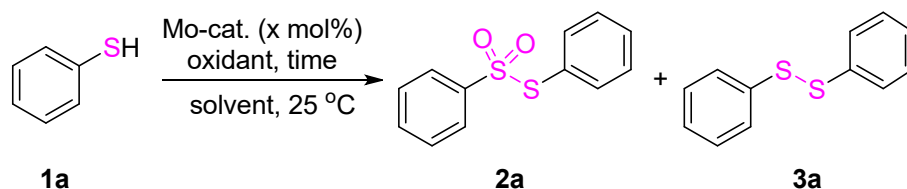


entry	Mo-cat. (x mol%)	oxidant (y equiv.)	solvent	time (h)	2a (%) ^b	3a (%) ^b
1	[Mo-1](0.5%)	H ₂ O ₂ (4)	EtOH	6	8	20
2	[Mo-2](0.5%)	H ₂ O ₂ (4)	EtOH	6	95	3

3	Na ₂ MoO ₄ (0.5%)	H ₂ O ₂ (4)	EtOH	6	17	32
4	MoO ₃ (0.5%)	H ₂ O ₂ (4)	EtOH	6	78	14
5	[Mo-2] (0.5%)	H ₂ O ₂ (3)	EtOH	6	90	6
6	[Mo-2] (0.5%)	H ₂ O ₂ (4)	MeCN	6	96	2
7	[Mo-2] (0.5%)	H ₂ O ₂ (4)	THF	6	80	9
8	[Mo-2] (0.5%)	H ₂ O ₂ (4)	MeOH	6	87	12
9	[Mo-2] (0.5%)	H ₂ O ₂ (4)	H ₂ O	6	45	23
10	[Mo-2] (0.5%)	H ₂ O ₂ (4)	CH ₃ COCH ₃	6	71	15
11	[Mo-2] (0.5%)	H ₂ O ₂ (4)	EtOH	5	86	12
12	[Mo-2] (0.5%)	H ₂ O ₂ (3)	EtOH	8	95	2
13	MoO ₃ (0.5%)	H ₂ O ₂ (4)	EtOH	24	91	5

^aReaction conditions: **1a** (0.4 mmol), catalyst (x mol%), oxidant (y equiv.), Solvent (2 mL), 25 °C. ^bIsolated yield.

Table S2. Optimization of reaction conditions for **3a**^a

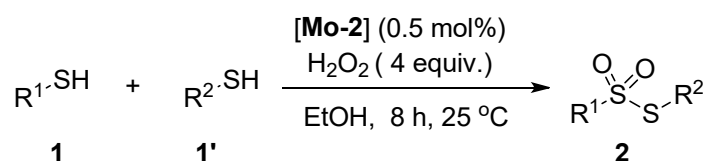


entry	Mo-cat. (x mol%)	oxidant	solvent	time (h)	2a (%) ^b	3a (%) ^b
1	[Mo-1](0.5%)	Air	EtOH	24	-	38
2	[Mo-2](0.5%)	Air	EtOH	24	-	65
3	Na ₂ MoO ₄ (0.5%)	Air	EtOH	24	-	47
4	MoO ₃ (0.5%)	Air	EtOH	24	-	54
5	[Mo-2] (1%)	Air	EtOH	24	2	88
6	[Mo-2] (1%)	O ₂	EtOH	12	7	85
7	[Mo-2] (1%)	Air	THF	24	-	52
8	[Mo-2] (1%)	Air	MeOH	24	-	80
9	[Mo-2] (1%)	Air	H ₂ O	24	-	36

10	[Mo-2] (1%)	Air	CH ₃ COCH ₃	24	-	72
11	[Mo-2] (1%)	Air	EtOH	18	-	81
12	[Mo-2] (1%)	Air	EtOH	36	4	86

^aReaction conditions: **1a** (0.4 mmol), catalyst (x mol%), oxidant, solvent (2 mL), 25 °C. ^bIsolated yield.

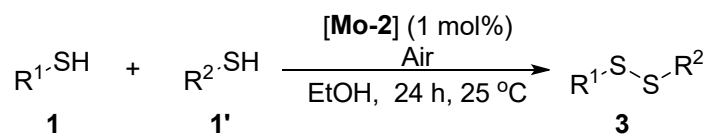
4. General procedure for synthesis of thiosulfonates



0.2 mmol scale: In a 10 mL tube was added **1** (0.2 mmol), **1'** (0.2 mmol), [**Mo-2**] (2.2 mg), H₂O₂ (91 mg), EtOH (2 mL). The reaction mixture was stirred at 25 °C for 8 h. Then the resulting mixture was evaporated under reduced pressure and purified by flash chromatography to afford the desired product.

5 mmol scale: In a 10 mL tube was added **1a** (0.2 mmol), [**Mo-2**] (28 mg), H₂O₂ (1.14g), EtOH (5 mL). The reaction mixture was stirred at 25 °C for 8 h. Then the resulting mixture was evaporated under reduced pressure and purified by flash chromatography to afford the desired product.

5. General procedure for synthesis of disulfides

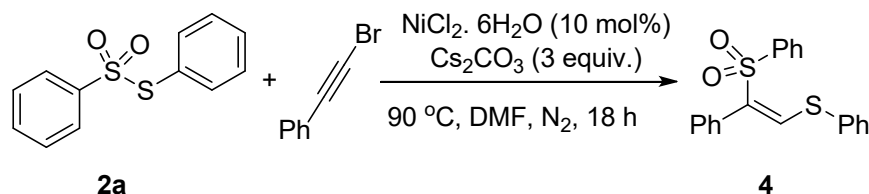


0.2 mmol scale: In a 10 mL tube was added **1** (0.2 mmol), **1'** (0.2 mmol), [**Mo-2**] (4.4 mg) and EtOH (2 mL). The reaction mixture was stirred at 25 °C for 24 h under air atmosphere. Then the resulting mixture was evaporated under reduced pressure and purified by flash chromatography to afford the desired product.

5 mmol scale: In a 10 mL tube was added **1a** (5 mmol), [**Mo-2**] (56 mg) and EtOH (5 mL). The reaction mixture was stirred at 25 °C for 24 h under air atmosphere. Then the resulting mixture was evaporated under reduced pressure and purified by

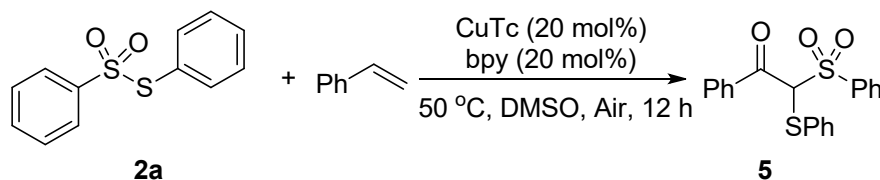
flash chromatography to afford the desired product.

6. General procedure for synthesis of compound 4^[2]



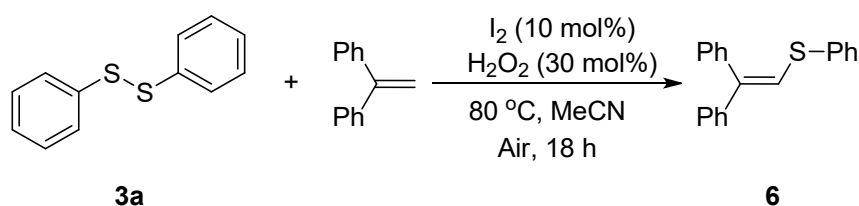
A 10 mL tube was charged with (bromoethynyl)benzene **1a** (0.2 mmol), thiosulfonate **2a** (0.3 mmol), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.02 mmol) and Cs_2CO_3 (0.6 mmol) in DMF (1.0 mL). The reaction mixture was stirred at 90 °C for 18 h under N_2 atmosphere. Then the mixture was quenched by the addition of H_2O (1 mL) followed by extraction with EtOAc (3×3 mL). The combined organic layers were washed with brine (2×3 mL), dried over anhydrous MgSO_4 , and the solvent was removed under reduced pressure. The resulting residue was purified by flash chromatography to give the corresponding product.

7. General procedure for synthesis of compound 5^[3]



To a mixture of copper thiophene-2-carboxylate (20 mol %), 2,2'-bipyridine (20 mol %) in solvent (1 mL DMSO + 36 mg water), styrene (0.2 mmol), and thiosulfonate **2a** (0.6 mmol) were added. The reaction system was stirred at 50 °C under air atmosphere for 12 h. Then, the resulting mixture was evaporated under reduced pressure and purified by flash chromatography to afford the desired product.

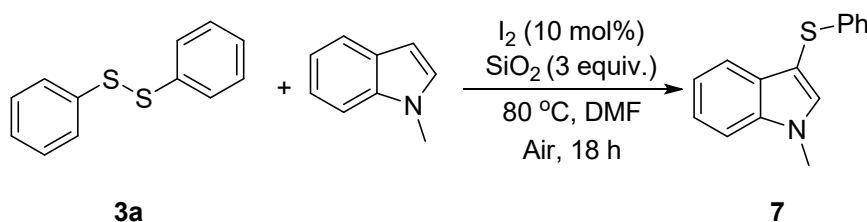
8. General procedure for synthesis of compound 6^[4]



A 10 mL tube was charged with ethene-1,1-diyldibenzene (0.2 mmol), disulfide

3a (0.2 mmol), I₂ (0.02 mmol) and H₂O₂ (0.06 mmol) in MeCN (2 mL). The reaction mixture was stirred at 80 °C for 18 h under air atmosphere. Then the mixture was quenched by the addition of H₂O (1 mL) followed by extraction with EtOAc (3×3 mL). The combined organic layers were washed with brine (2×3 mL), dried over anhydrous MgSO₄, and the solvent was removed under reduced pressure. The resulting residue was purified by flash chromatography to give the corresponding product.

9. General procedure for synthesis of compound **7**^[5]

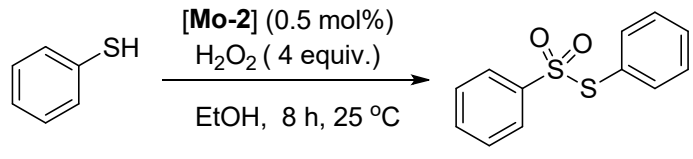


A 10 mL tube was charged with 1-methyl-1*H*-indole (0.22 mmol), disulfide **3a** (0.2 mmol), I₂ (0.02 mmol) and SiO₂ (0.6 mmol) in DMF (2 mL). The reaction mixture was stirred at 80 °C for 18 h under air atmosphere. Then the mixture was quenched by the addition of H₂O (1 mL) followed by extraction with EtOAc (3×3 mL). The combined organic layers were washed with brine (2×3 mL), dried over anhydrous MgSO₄, and the solvent was removed under reduced pressure. The resulting residue was purified by flash chromatography to give the corresponding product.

10. Recycling and reuse of catalyst

A 10 mL tube was charged with **1a** (5 mmol), [**Mo-2**] (28 mg), H₂O₂ (1.14 g), EtOH (5 mL). The reaction mixture was stirred at 25 °C for 8 h. In each run, the resulting mixture was evaporated under reduced pressure, and then washed thoroughly with ethyl acetate, and dried under vacuum. Subsequently, the dried catalyst was used further, without any purification or reactivation. The washing liquid was evaporated under vacuum, and the residue was purified by column chromatography.

Table S3. Recycling of [Mo-2] catalyst

					
	1a			2a	
Run	1st	2nd	3rd	4th	5th
Yield (%)	86	85	82	83	80

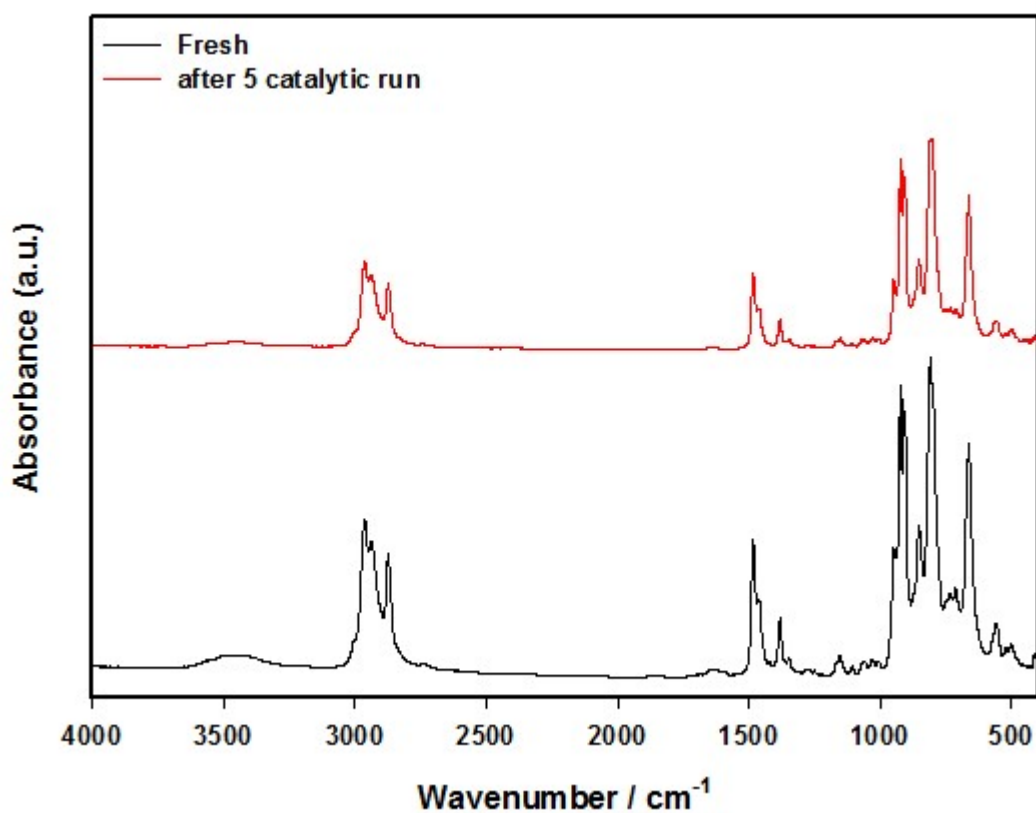
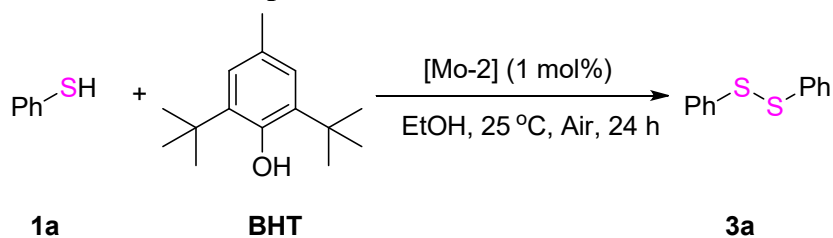


Figure S2. IR spectrums of the [Mo-2] catalyst

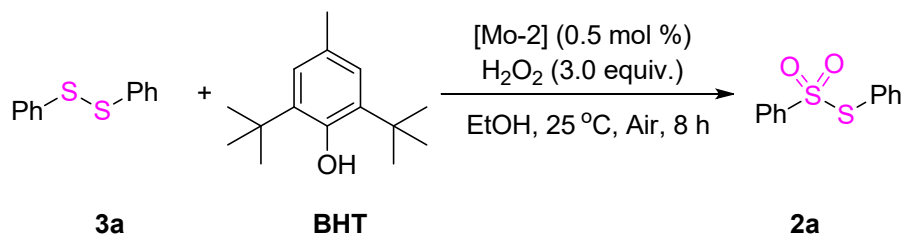
11. Mechanistic investigations

11.1 Radical intermediate trap reaction

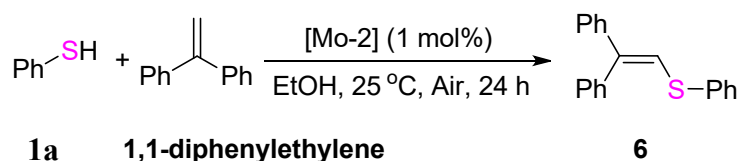


1a (0.4 mmol), [Mo-2] (1 mol%), BHT (0.2 or 2 equiv.), and EtOH (2.0 mL) were

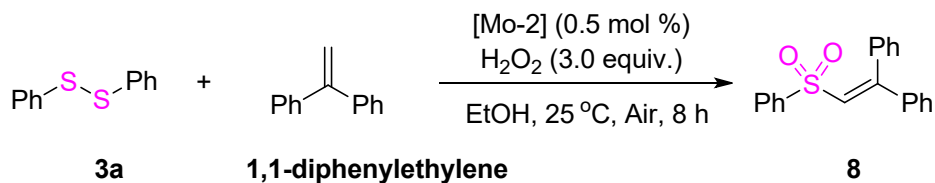
added to a tube (10 mL) under air atmosphere. The resulting mixture was stirred at 25 °C for 24 h. After completion, the solvent was removed under reduced pressure and the resulting crude compound was tested by ¹H NMR.



1a (0.2 mmol), [Mo-2] (0.5 mol%), H₂O₂ (3.0 equiv.), BHT (0.2 or 2 equiv.), and EtOH (2.0 mL) were added to a tube (10 mL) under air atmosphere. The resulting mixture was stirred at 25 °C for 8 h. After completion, the solvent was removed under reduced pressure and the resulting crude compound was tested by ¹H NMR.



1a (0.2 mmol), [Mo-2] (1 mol%), 1,1-Diphenylethylene (0.3 mmol), and EtOH (2.0 mL) were added to a tube (10 mL) under air atmosphere. The resulting mixture was stirred at 25 °C for 24 h. After completion, the solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography with petroleum ether (30~60 °C b.p.) and ethyl acetate as the eluent (PE/EA=10:1, V/V) to afford the compound **6** in 17% yield (Colorless oil, 9.8 mg).



3a (0.2 mmol), [Mo-2] (0.5 mol%), H₂O₂ (3.0 equiv.), 1,1-Diphenylethylene (0.3 mmol), and EtOH (2.0 mL) were added to a tube (10 mL) under air atmosphere. The resulting mixture was stirred at 25 °C for 24 h. After completion, the solvent was removed under reduced pressure and the residue was purified by silica gel column

chromatography with petroleum ether (30~60 °C b.p.) and ethyl acetate as the eluent (PE/EA=5:1, V/V) to afford the compound **8** in 23% yield (Colorless oil, 14.7 mg).

11.2 Kinetic profile of **1a** with H₂O₂ as an oxidant for oxidation

In a 10 mL tube was added **1** (0.4 mmol), [**Mo-2**] (2.2 mg), H₂O₂ (91 mg), EtOH (2 mL). The reaction mixture was stirred at 25 °C. Then the resulting mixture was monitored by GC spectrum.

11.3 Kinetic profile of **1a** with Air as an oxidant for oxidation

In a 10 mL tube was added **1** (0.4 mmol), [**Mo-2**] (4.4 mg), H₂O₂ (91 mg), EtOH (2 mL). The reaction mixture was stirred at 25 °C. Then the resulting mixture was monitored by GC spectrum.

11.4 General procedure for UV-vis studies

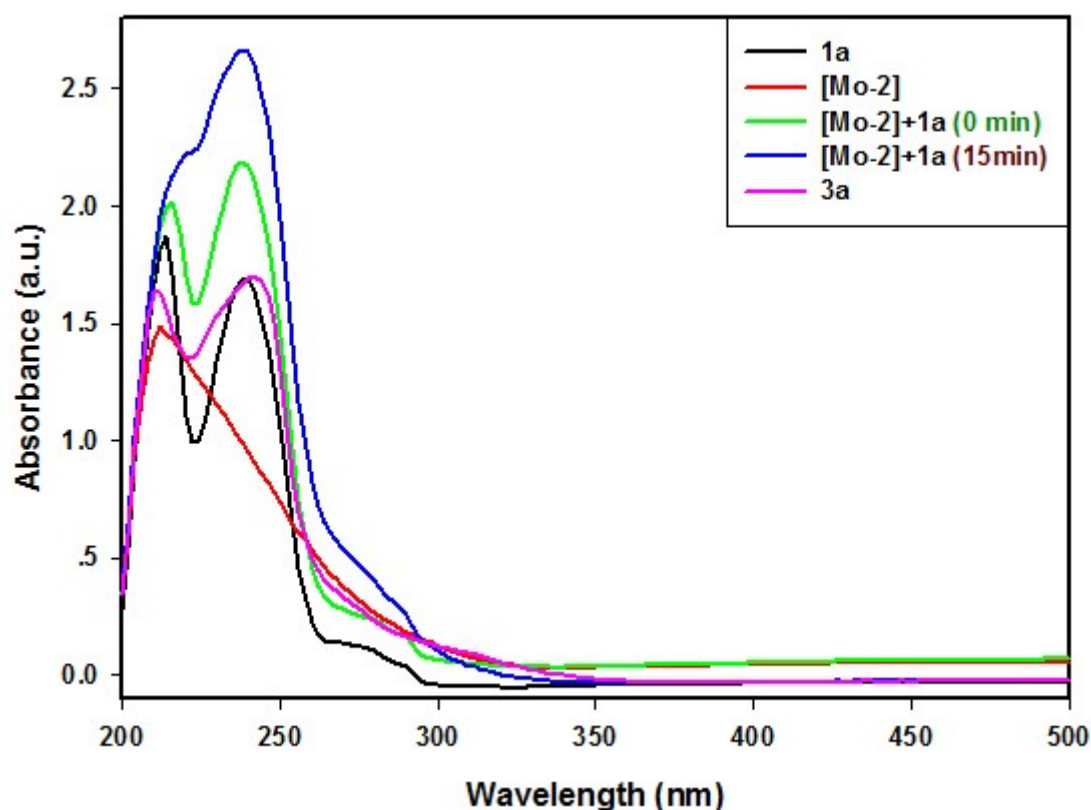


Figure S3. UV-visible spectra of compounds in CH₃CH₂OH solution: **1a**=0.00013 mmol/mL (black line); [**Mo-2**]=0.00012 mmol/mL (red line); **3a**=0.00013 mmol/mL (pink line); the volume ratio of [**Mo-2**]/**1a**=1:1 (green line and blue line). All the test conditions are in air atmosphere.

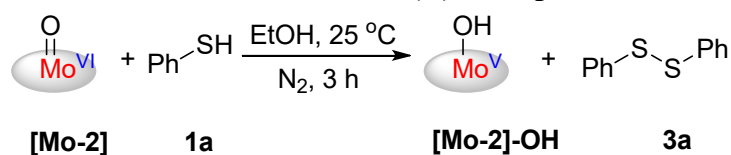
11.5 General procedure for cyclic voltammetry studies

The compound (0.2 mmol) was added in MeCN solution (with 0.1 M *n*-Bu₄NBF₄ as electrolyte) at room temperature. The voltammogram was obtained with a Pt wire as counter electrode and Ag/AgCl as a reference electrode. The scan rate was 0.1 V/s on a glassy carbon electrode (d = 1 mm).

11.6 General procedure for Raman spectra studies

The Raman spectra were obtained on ThermoFisher DXR2xi with an excitation wavelength of 633 nm. Firstly, 10 mg of [Mo-2] powder were placed on a glass slide and then tested in an instrument to obtain data. Secondly, these samples were treated with 30% H₂O₂ (1.2 mg) for 10 min. Thirdly, the resulting solid was washing with ethanol and drying under flow N₂ atmosphere for 10 min, then it was used to test again.

12. Preparation and characterization of Mo(V)-OH species



1a (0.08 mmol), [Mo-2] (0.01 mmol), and EtOH (2.0 mL) were added to a tube (10 mL) under N₂ atmosphere. The resulting mixture was stirred at 25 °C for 3 h. After completion, the resulting mixture was evaporated under reduced pressure, and then the solid was washed thoroughly with *n*-hexane, and dried under vacuum. Subsequently, the dried solid was tested by IR. The washing liquid was evaporated under vacuum, and the residue was purified by column chromatography to give **3a** in 86% yield.

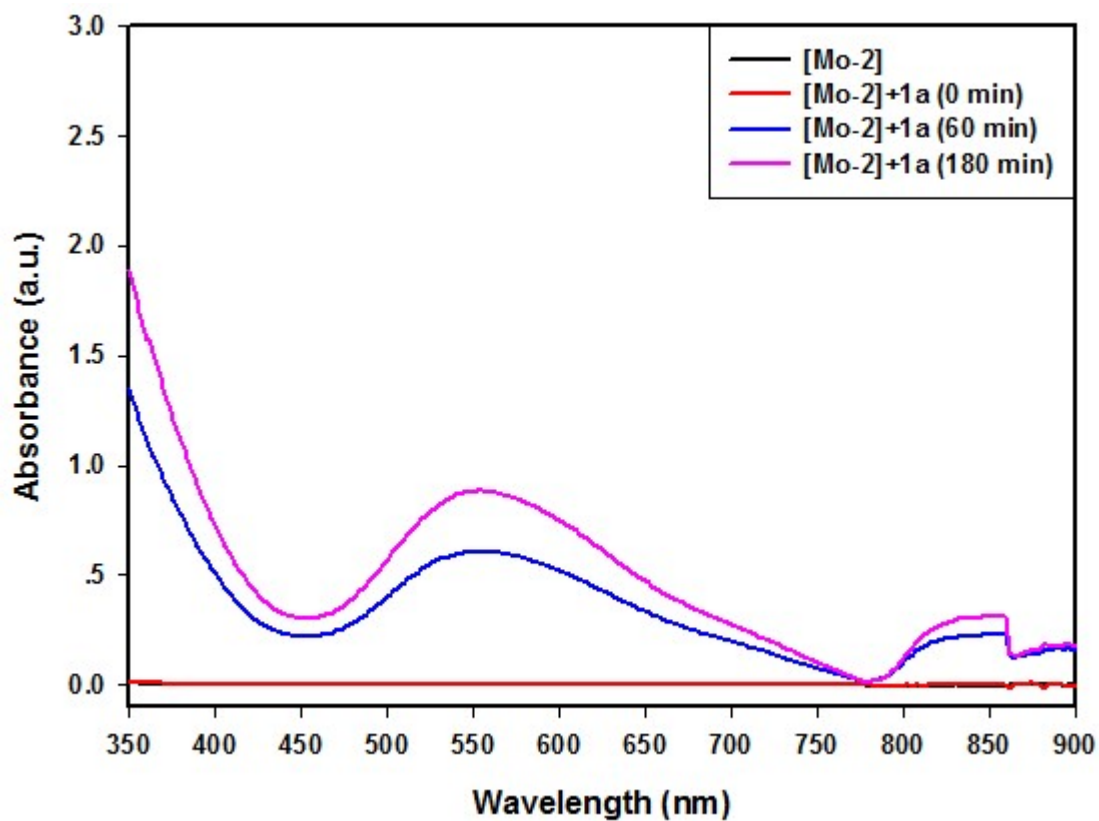


Figure S4. UV-visible spectra of the complexes in CH₃CH₂OH solution, and the concentration is about 0.001 mmol/mL (based on [Mo-2]). All the test conditions are in N₂ atmosphere.

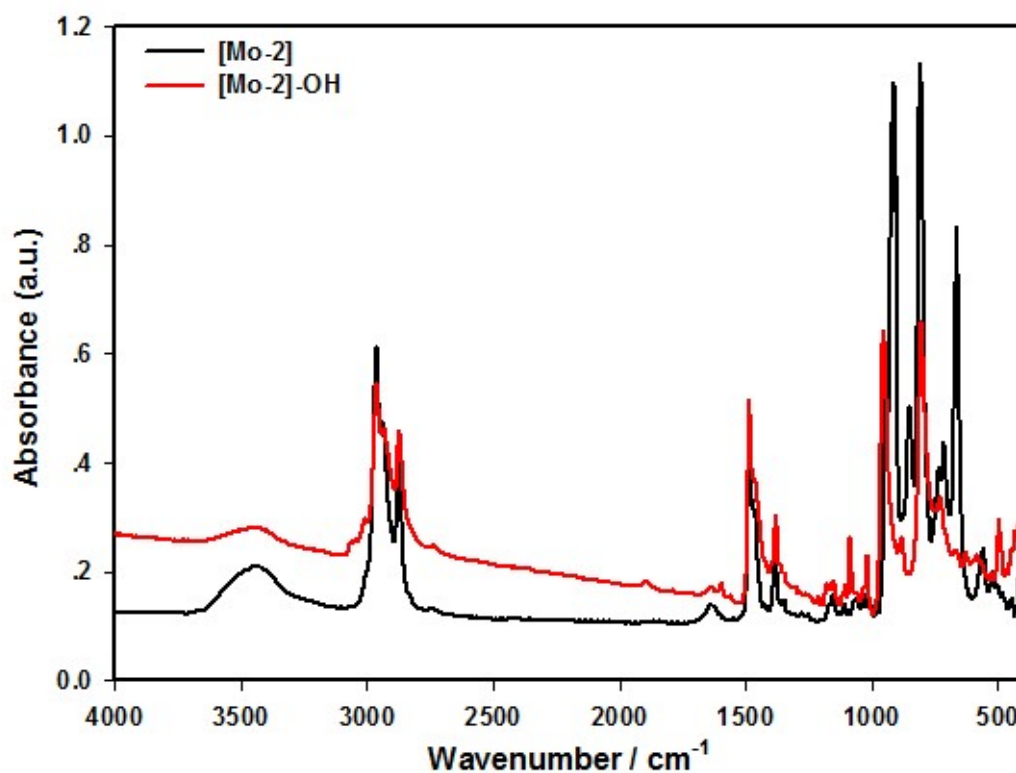
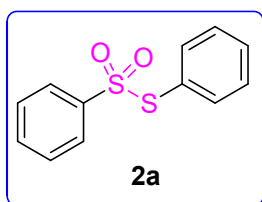
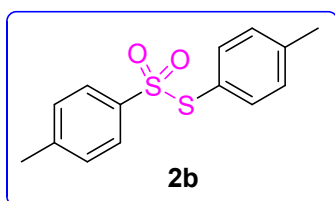


Figure S5. IR spectra of the [Mo-2] and resulting solid [Mo-2]-OH (KBr pellet)

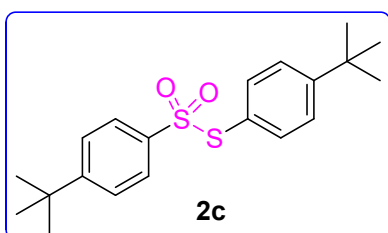
13. Analytical data



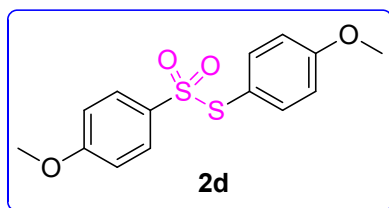
S-phenyl benzenesulfonylthioate (**2a**). White solid, yield 47.5 mg (95%); TLC (petroleum ether: AcOEt=5:1), $R_f = 0.52$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.55-7.59 (m, 3H), 7.47 (t, $J=6.4$ Hz, 1H), 7.39-7.43 (m, 2H), 7.31-7.36 (m, 4H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 142.9, 136.6, 133.7, 131.4, 129.4, 128.8, 127.8, 127.5. Spectral data were in accordance with the literature.^[6]



S-*p*-tolyl 4-methylbenzenesulfonylthioate (**2b**). White solid, yield 52.3 mg (94%); TLC (petroleum ether: AcOEt=5:1), $R_f = 0.56$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.46 (d, $J=8.4$ Hz, 2H), 7.21-7.27 (m, 4H), 7.14 (d, $J=7.6$ Hz, 2H), 2.43 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 144.6, 142.0, 140.5, 136.5, 130.2, 129.3, 127.6, 124.6, 21.7, 21.5. Spectral data were in accordance with the literature.^[6]

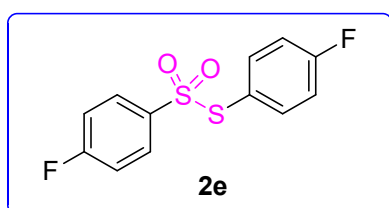


S-(4-(*tert*-butyl)phenyl) 4-(*tert*-butyl)benzenesulfonylthioate (**2c**). White solid, yield 66.6 mg (92%); TLC (petroleum ether: AcOEt=10:1), $R_f = 0.62$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.34 (d, $J=8.0$ Hz, 2H), 7.26 (d, $J=7.6$ Hz, 2H), 7.19 (d, $J=8.0$ Hz, 2H), 7.13 (d, $J=6.8$ Hz, 2H), 1.88 (s, 9H), 1.79 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 157.4, 154.9, 140.0, 136.2, 127.3, 126.3, 125.5, 124.5, 35.1, 34.8, 31.0, 30.9. Spectral data were in accordance with the literature.^[6]



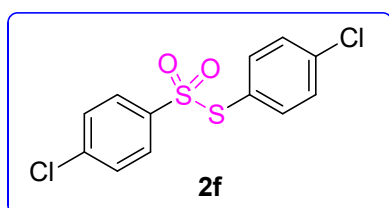
S-(4-methoxyphenyl) 4-methoxybenzenesulfonylthioate

(**2d**). White solid, yield 58.9 mg (95%); TLC (petroleum ether: AcOEt=5:1), $R_f = 0.32$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.48 (d, $J=8.8$ Hz, 2H), 7.24 (d, $J=7.2$ Hz, 2H), 6.82-6.87 (m, 4H), 3.85 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 163.5, 162.2, 138.2, 134.8, 129.9, 118.9, 114.9, 113.8, 55.7, 55.5. Spectral data were in accordance with the literature.^[6]



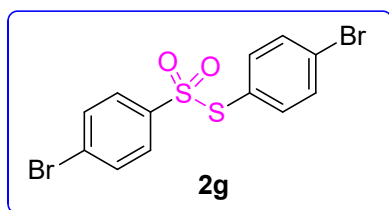
S-(4-fluorophenyl) 4-fluorobenzenesulfonylthioate (**2e**).

White solid, yield 50.4 mg (88%); TLC (petroleum ether: AcOEt=10:1), $R_f = 0.60$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.57 (dd, $J=4.8$ Hz, 2H), 7.33-7.36 (m, 2H), 7.11 (t, $J=8.0$ Hz, 2H), 7.05 (t, $J=8.0$ Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 165.0 (d, $J=256$ Hz), 164.7 (d, $J=253$ Hz), 138.8, 138.7, 130.3 (d, $J=10$ Hz), 123.1 (d, $J=4$ Hz), 116.8 (d, $J=23$ Hz), 116.1 (d, $J=23$ Hz); ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -102.6, -106.9. Spectral data were in accordance with the literature.^[6]



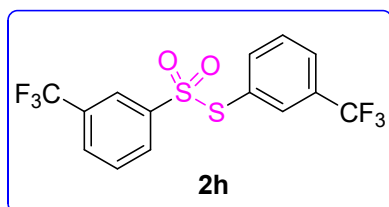
S-(4-chlorophenyl) 4-chlorobenzenesulfonylthioate

(**2f**). White solid, yield 58.5 mg (92%); TLC (petroleum ether: AcOEt=10:1), $R_f = 0.56$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.52 (d, $J=8.4$ Hz, 2H), 7.43 (d, $J=8.0$ Hz, 2H), 7.30-7.36 (m, 4H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 141.3, 140.6, 138.6, 137.7, 129.9, 129.3, 128.9, 126.0. Spectral data were in accordance with the literature.^[6]



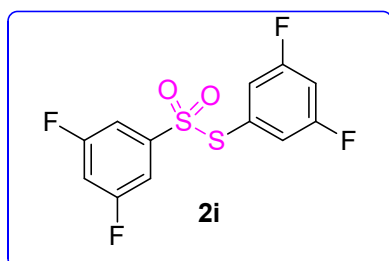
S-(4-bromophenyl) 4-bromobenzenesulfonylthioate

(2g). White solid, yield 60.9 mg (75%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.50. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.57-7.62 (m, 2H), 7.41-7.50 (m, 4H), 7.31 (d, $J=7.6$ Hz, 1H), 7.22 (d, $J=7.6$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 137.8, 136.9, 132.9, 132.5, 132.3, 132.2, 128.9, 125.9. Spectral data were in accordance with the literature.^[6]



S-(3-(trifluoromethyl)phenyl) 3-(trifluoromethyl)benzenesulfonylthioate (**2h**)

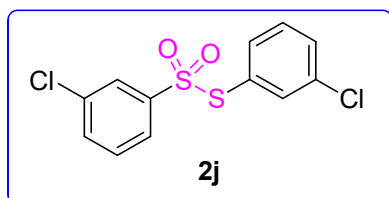
(2h). Yellow oil, yield 61.8 mg (80%); TLC (petroleum ether: AcOEt=10:1), R_f = 0.52. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.87 (d, $J=7.6$ Hz, 1H), 7.77-7.83 (m, 2H), 7.63-7.71 (m, 3H), 7.44-7.59 (m, 1H), 7.26 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 143.3, 139.9, 133.0, 132.98, 130.57, 130.54, 130.49, 130.48, 130.34, 130.04, 128.67, 128.45 (q, $J=3$ Hz), 124.7; ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -63.10, -63.12. Spectral data were in accordance with the literature.^[7]



S-(3,5-difluorophenyl) 3,5-difluorobenzenesulfonylthioate (**2i**)

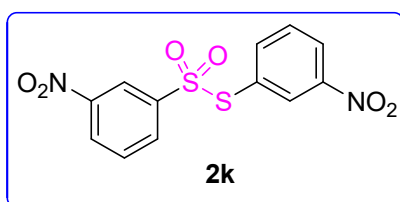
(2i). Yellow solid, yield 41.9 mg (65%); TLC (petroleum ether: AcOEt=10:1), R_f = 0.50. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.19 (s, 2H), 7.10 (t, $J=6.8$ Hz, 1H), 7.01 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ

(ppm) = 163.8 (d, $J=13$ Hz), 161.3 (d, $J=13$ Hz), 119.4, 119.3, 119.2, 119.1, 111.3, 111.0, 109.9, 107.9; ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -104.5, -106.5. Spectral data were in accordance with the literature.^[8]



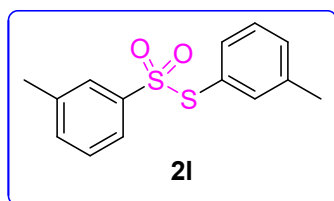
S-(3-chlorophenyl) 3-chlorobenzenesulfonylthioate

(2j). Colorless oil, yield 57.2 mg (90%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.45. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.56 (d, $J=6.4$ Hz, 1H), 7.52 (s, 1H), 7.38-7.48 (m, 3H), 7.28-7.38 (m, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 143.9, 136.1, 135.2, 135.1, 134.6, 134.1, 131.9, 130.6, 130.2, 128.9, 127.6, 125.6. Spectral data were in accordance with the literature.^[9]



S-(3-nitrophenyl) 3-nitrobenzenesulfonylthioate (**2k**).

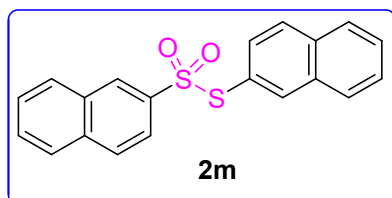
Yellow solid, yield 38.8 mg (57%); TLC (petroleum ether: AcOEt=2:1), R_f = 0.47. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.49 (d, $J=8.8$ Hz, 1H), 8.39 (d, $J=8.0$ Hz, 1H), 8.35 (s, 1H), 8.14 (s, 1H), 7.93 (d, $J=7.6$ Hz, 1H), 7.83 (d, $J=7.6$ Hz, 1H), 7.76 (d, $J=8.0$ Hz, 1H), 7.68 (d, $J=8.0$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 148.3, 148.0, 144.1, 142.1, 132.5, 130.8, 130.7, 129.1, 128.5, 126.6, 122.5. Spectral data were in accordance with the literature.^[10]



S-*m*-tolyl 3-methylbenzenesulfonylthioate (**2l**). Colorless

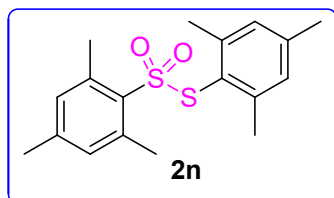
oil, yield 51.2 mg (92%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.54. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.24-7.37 (m, 5H), 7.17-7.21 (m, 1H), 7.11 (s, 2H); ^{13}C

NMR (101 MHz, CDCl₃): δ (ppm) = 142.7, 139.4, 139.0, 137.1, 134.4, 133.6, 132.2, 129.2, 128.6, 128.0, 127.5, 124.7. Spectral data were in accordance with the literature.^[11]



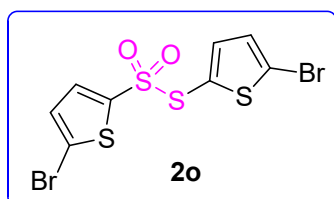
S-naphthalen-2-yl naphthalene-2-sulfonylthioate (**2m**).

White solid, yield 62.3 mg (89%); TLC (petroleum ether: AcOEt=10:1), R_f = 0.54. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.94 (s, 2H), 7.88 (d, J =8.4 Hz, 2H), 7.83 (s, 2H), 7.73 (d, J =8.4 Hz, 1H), 7.46-7.68 (m, 7H), 7.35 (d, J =8.4 Hz, 1H); ¹³C NMR (101 MHz, CDCl₃): δ (ppm) = 139.7, 137.7, 135.1, 134.1, 133.2, 131.8, 131.6, 129.4, 129.3, 129.1, 128.4, 128.3, 127.9, 127.7, 127.6, 126.9, 125.1, 122.4. Spectral data were in accordance with the literature.^[6]



S-mesityl 2,4,6-trimethylbenzenesulfonylthioate (**2n**).

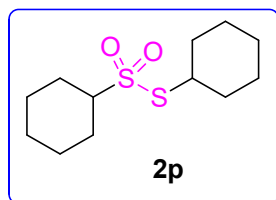
White solid, yield 56.8 mg (85%); TLC (petroleum ether: AcOEt=10:1), R_f = 0.60. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 6.88-6.89 (m, 4H), 2.35 (s, 6H), 2.29 (s, 3H), 2.27 (s, 3H), 2.15 (s, 6H); ¹³C NMR (101 MHz, CDCl₃): δ (ppm) = 145.4, 143.5, 141.8, 139.7, 139.4, 131.9, 129.5, 123.3, 22.7, 21.5, 21.2, 21.1. Spectral data were in accordance with the literature.^[6]



S-(5-bromothiophen-2-yl) 5-bromothiophene-2-

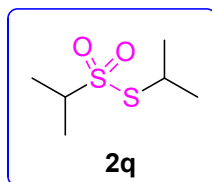
sulfonylthioate (**2o**). White solid, yield 58.5 mg (70%); TLC (petroleum ether:

AcOEt=20:1), $R_f = 0.48$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.20-7.21 (m, 1H), 7.05-7.11 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 142.8, 140.1, 134.6, 131.5, 130.3, 125.8, 123.3, 122.1, 22.7, 21.5, 21.2, 21.1. HRMS (ESI-QTOF) Calcd for $\text{C}_8\text{H}_4\text{Br}_2\text{O}_2\text{S}_4\text{Na}$, $[\text{M}+\text{Na}]^+$ 440.7353; Found 440.7348.



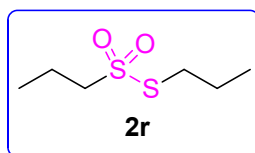
S-cyclohexyl cyclohexanesulfonylthioate (**2p**). Colorless oil,

yield 47.7 mg (91%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.38$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 3.47 (s, 1H), 3.02-3.08 (m, 1H), 2.30 (d, $J=12.4$ Hz, 2H), 2.10 (d, $J=12.0$ Hz, 2H), 1.94 (d, $J=12.8$ Hz, 2H), 1.73 (s, 3H), 1.52-1.58 (m, 5H), 1.39-1.47 (m, 2H), 1.19-1.35 (m, 5H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 71.5, 50.4, 34.3, 26.2, 25.9, 25.2, 25.1, 25.0. Spectral data were in accordance with the literature.^[11]



S-isopropyl propane-2-sulfonylthioate (**2q**). Colorless oil, yield 32.8

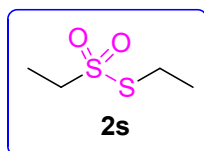
mg (90%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.48$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 3.63 (q, $J=6.8$ Hz, 1H), 3.32 (q, $J=6.8$ Hz, 1H), 1.42-1.43 (m, 12H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 63.5, 42.9, 24.2, 16.3. Spectral data were in accordance with the literature.^[9]



S-propyl propane-1-sulfonylthioate (**2r**). Colorless oil, yield 30.9

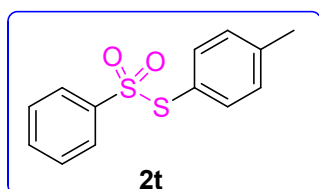
mg (85%); TLC (petroleum ether: AcOEt=10:1), $R_f = 0.65$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 3.27 (t, $J=7.6$ Hz, 2H), 3.10 (q, $J=6.8$ Hz, 2H), 1.93 (q, $J=6.8$ Hz, 2H), 1.76 (q, $J=6.8$ Hz, 2H), 1.05-1.09 (m, 3H), 1.00-1.04 (m, 3H); $^{13}\text{C NMR}$ (101

MHz, CDCl₃): δ (ppm) = 64.3, 38.1, 23.2, 17.3, 13.1, 12.7. Spectral data were in accordance with the literature.^[12]



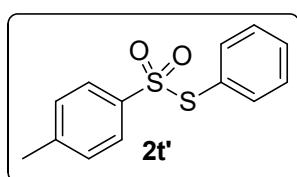
S-ethyl ethanesulfonylthioate (**2s**). Colorless oil, yield 25.3 mg (82%);

TLC (petroleum ether: AcOEt=6:1), R_f = 0.42. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 3.31 (q, J =7.6 Hz, 2H), 3.15 (q, J =7.2 Hz, 2H), 1.40-1.48 (m, 6H), 1.00-1.04 (m, 3H); ¹³C NMR (101 MHz, CDCl₃): δ (ppm) = 57.1, 30.7, 15.2, 8.4. Spectral data were in accordance with the literature.^[6]



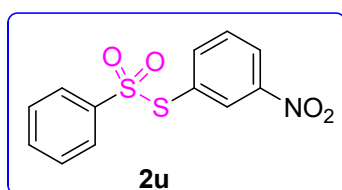
S-p-tolyl benzenesulfonylthioate (**2t**). Colorless oil, yield

15.9 mg (30%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.52. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.53-7.55 (m, 3H), 7.39 (t, J =6.4 Hz, 1H), 7.18 (d, J =5.6 Hz, 2H), 7.10 (d, J =7.2 Hz, 2H), 2.34 (s, 3H); ¹³C NMR (101 MHz, CDCl₃): δ (ppm) = 142.90, 142.2, 136.4, 133.6, 130.3, 128.8, 127.5, 124.3, 21.5. Spectral data were in accordance with the literature.^[13]



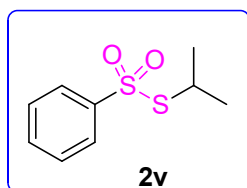
S-phenyl 4-methylbenzenesulfonylthioate (**2t'**)^[14]. ¹H NMR

(400 MHz, CDCl₃) δ (ppm) = 7.49-7.45 (m, 3H), 7.40-7.34 (m, 4H), 7.22 (d, J = 8.1 Hz, 2H), 2.44 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 144.7, 140.3, 136.6, 131.3, 129.4, 129.4, 128.0, 127.6, 21.7.

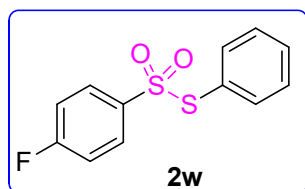


S-(3-nitrophenyl) benzenesulfonylthioate (**2u**). White solid,

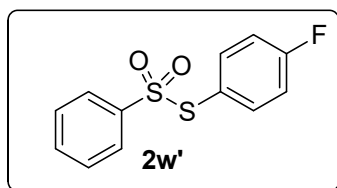
yield 23.5 mg (40%); TLC (petroleum ether: AcOEt=6:1), $R_f = 0.45$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.32 (d, $J=8.0$ Hz, 1H), 8.09 (s, 1H), 7.76 (s, 1H), 7.57-7.63 (m, 4H), 7.45-7.48 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 148.2, 142.3, 136.4, 134.4, 130.8, 130.4, 129.2, 127.5, 126.0. Spectral data were in accordance with the literature.^[15]



S-isopropyl benzenesulfonylthioate (**2v**). Colorless oil, yield 19.4 mg (45%); TLC (petroleum ether: AcOEt=10:1), $R_f = 0.50$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.87 (d, $J=7.6$ Hz, 2H), 7.47-7.56 (m, 3H), 3.42 (q, $J=6.8$ Hz, 2H), 1.23 (d, $J=6.8$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 136.5, 133.7, 129.3, 126.8, 42.7, 23.5. HRMS (ESI-QTOF) Calcd for $\text{C}_9\text{H}_{12}\text{O}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 239.0171; Found 239.0165.

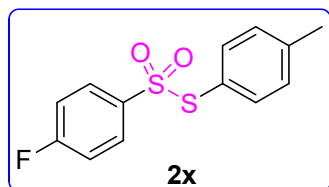


S-phenyl 4-fluorobenzenesulfonylthioate (**2w**). White solid, yield 22.0 mg (41%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.38$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.54-7.57 (m, 2H), 7.47-7.48 (m, 1H), 7.34-7.35 (m, 4H), 7.05-7.10 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 165.0 (d, $J=255$ Hz), 138.9, 136.6, 131.6, 130.4 (d, $J=10$ Hz), 129.6, 127.6, 116.0 (d, $J=22$ Hz); ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -102.8. HRMS (ESI-QTOF) Calcd for $\text{C}_{12}\text{H}_9\text{FO}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 290.9920; Found 290.9918. Spectral data were in accordance with the literature.^[16]

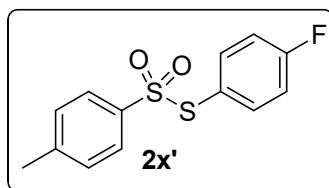


S-(4-fluorophenyl) benzenesulfonylthioate (**2w'**)^[17]. ^1H NMR (400 MHz, CDCl_3) δ = 7.62-7.55 (m, 3H), 7.47-7.41(m, 2H), 7.37-7.29 (m, 2H),

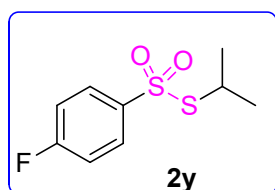
7.07-6.98 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ = 164.8 (d, J = 253.9 Hz), 142.8, 138.9 (d, J = 9.0 Hz), 133.8, 128.9, 127.6, 123.5 (d, J = 3.3 Hz), 116.8 (d, J = 22.2 Hz).



S-*p*-tolyl 4-fluorobenzenesulfonothioate (**2x**). White solid, yield 21.4 mg (38%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.40. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.55 (d, J =6.8 Hz, 2H), 7.21 (d, J =6.8 Hz, 2H), 7.13 (d, J =7.6 Hz, 2H), 7.05-7.09 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 165.1 (d, J =255 Hz), 142.4, 139.0, 136.4, 130.3 (d, J =10 Hz), 124.2, 116.1, 115.9, 21.5; ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -103.1. Spectral data were in accordance with the literature.^[13]

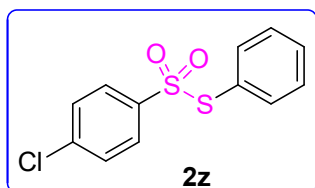


S-(4-fluorophenyl) 4-methylbenzenesulfonothioate (**2x'**)^[14]. ^1H NMR (400 MHz, CDCl_3) δ = (ppm) 7.47 (d, J = 8.3 Hz, 2H), 7.36 (dd, J = 8.7, 5.2 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.05 (t, J = 8.6 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ = (ppm) 164.8 (d, J = 253.7 Hz), 145.0, 140.0, 138.8 (d, J = 9.1 Hz), 129.5, 127.6, 123.6 (d, J = 3.3 Hz), 116.8 (d, J = 22.3 Hz), 21.7.

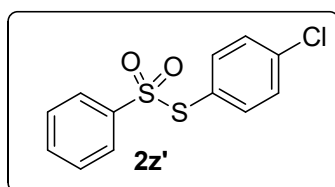


S-isopropyl 4-fluorobenzenesulfonothioate (**2y**). Colorless oil, yield 19.7 mg (42%); TLC (petroleum ether: AcOEt=20:1), R_f = 0.38. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.87-7.88 (m, 2H), 7.15 (d, J =8.0 Hz, 2H), 3.42 (q, J =7.2 Hz, 1H), 1.24-1.25 (m, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 165.3 (d, J =255 Hz), 138.9, 129.7, 116.4 (d, J =23 Hz), 42.9, 23.5; ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -103.4. HRMS (ESI-QTOF) Calcd for $\text{C}_9\text{H}_{11}\text{FO}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 257.0077;

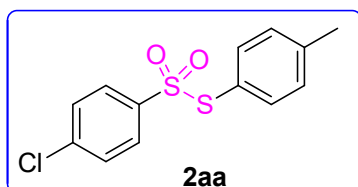
Found 257.0072.



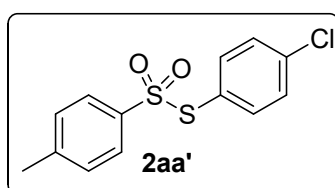
S-phenyl 4-chlorobenzenesulfonylthioate (**2z**). White solid, yield 21.3 mg (43%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.45$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.46-7.48 (m, 3H), 7.36-7.38 (m, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 141.3, 140.3, 136.5, 131.7, 129.6, 129.1, 128.9, 127.5. Spectral data were in accordance with the literature.^[11]



S-(4-chlorophenyl) benzenesulfonylthioate (**2z'**)^[17]. ^1H NMR (400 MHz, CDCl_3) $\delta = 7.64$ -7.56 (m, 3H), 7.48-7.43 (m, 2H), 7.34-7.26 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) $\delta = 142.8$, 138.3, 137.7, 133.8, 129.7, 128.9, 127.6, 126.3.

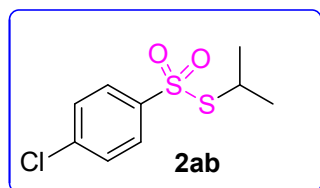


S-*p*-tolyl 4-chlorobenzenesulfonylthioate (**2aa**). White solid, yield 22.1 mg (37%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.45$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.49 (d, $J=8.8$ Hz, 2H), 7.38 (d, $J=8.8$ Hz, 2H), 7.24 (d, $J=7.6$ Hz, 2H), 7.15 (d, $J=7.6$ Hz, 2H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 142.5, 141.5, 140.2, 136.4, 130.4, 129.1, 128.9, 124.1, 21.5. Spectral data were in accordance with the literature.^[13]

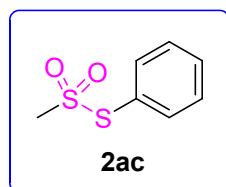


S-(4-chlorophenyl) 4-methylbenzenesulfonylthioate (**2aa'**)^[14]. ^1H NMR (400 MHz, CDCl_3) $\delta =$ (ppm) 7.48 (d, $J = 8.3$ Hz, 2H), 7.35-7.30 (m, 4H), 7.25 (d, $J = 8.2$ Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) $\delta =$

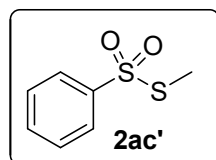
(ppm) 145.1, 140.1, 138.2, 137.7, 129.7, 129.6, 127.6, 126.5, 21.7.



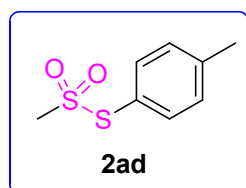
S-isopropyl 4-chlorobenzenesulfonothioate (**2ab**). White solid, yield 21.0 mg (42%); TLC (petroleum ether: AcOEt=20:1), $R_f = 0.54$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.84 (d, $J=8.0$ Hz, 2H), 7.48 (d, $J=8.4$ Hz, 2H), 3.46 (q, $J=6.8$ Hz, 1H), 1.28 (d, $J=6.8$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 143.9, 140.0, 129.5, 128.3, 42.9, 23.5. HRMS (ESI-QTOF) Calcd for $\text{C}_9\text{H}_{11}\text{ClO}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 272.9781; Found 272.9774.



S-phenyl methanesulfonothioate (**2ac**). White solid, yield 14.7 mg (39%); TLC (petroleum ether: AcOEt=15:1), $R_f = 0.41$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.69-7.71 (m, 2H), 7.53-7.56 (m, 1H), 7.46-7.49 (m, 2H), 3.17 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 136.2, 131.7, 129.9, 127.9, 47.5. Spectral data were in accordance with the literature.^[6]

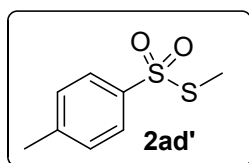


S-methyl benzenesulfonothioate (**2ac'**)^[18]. ^1H NMR (400 MHz, CDCl_3) δ = 7.93 (dd, $J = 8.4, 1.3$ Hz, 2H), 7.70 – 7.61 (m, 1H), 7.57 (dd, $J = 8.3, 6.9$ Hz, 2H), 2.51 (s, 3H).

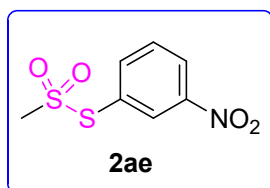


S-phenyl methanesulfonothioate (**2ad**). Colorless oil, yield 18.6 mg (46%); TLC (petroleum ether: AcOEt=15:1), $R_f = 0.45$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.57 (d, $J=8.0$ Hz, 2H), 7.27 (d, $J=8.0$ Hz, 1H), 3.15 (s, 3H), 2.40

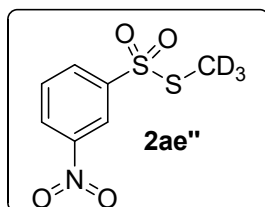
(s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 142.5, 136.2, 130.7, 124.5, 47.2, 21.5. Spectral data were in accordance with the literature.^[6]



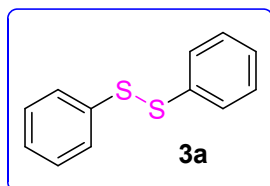
S-methyl 4-methylbenzenesulfonothioate (**2ad'**)^[19]. ^1H NMR (500 MHz, CDCl_3) δ = 7.80 (d, 2H, J = 8.3 Hz), 7.36 (d, 2H, J = 8.3 Hz), 2.50 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ = 144.9, 140.9, 129.9, 127.2, 21.7, 18.1.



S-(3-nitrophenyl) methanesulfonothioate (**2ae**). Colorless oil, yield 14.0 mg (30%); TLC (petroleum ether: AcOEt=2:1), R_f = 0.32. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.53 (s, 1H), 8.37 (d, J =6.8 Hz, 1H), 8.05 (d, J =6.0 Hz, 1H), 7.69-7.75 (m, 1H), 3.26 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 148.6, 142.0, 130.9, 130.5, 130.0, 126.3, 48.4. Spectral data were in accordance with the literature.^[20]

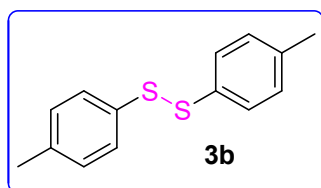


(**2ae''**)^[21]: ^1H NMR (400 MHz, CDCl_3) δ = 8.67 (1H, s), 8.48 (1H, d, J = 8.2 Hz), 8.23 (1H, d, J = 7.8 Hz), 7.87-7.82 (1H, m); ^{13}C NMR (101 MHz, CDCl_3) δ = 148.2, 145.3, 132.4, 131.0, 128.2, 122.1, 18.2-17.3 (m, C-D₃).

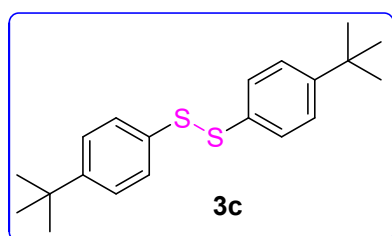


1,2-diphenyldisulfane (**3a**). White solid, yield 38.4 mg (88%); TLC (petroleum ether), R_f = 0.81. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.53 (d, J =6.8 Hz, 4H), 7.32 (t, J =7.6 Hz, 4H), 7.22-7.26 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 137.1, 129.1, 127.5, 127.2. Spectral data were in accordance with

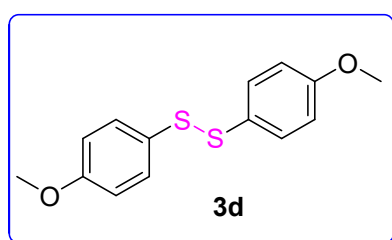
the literature.^[6]



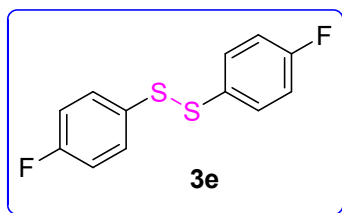
1,2-di-p-tolyl disulfane (3b). White solid, yield 46.3 mg (94%); TLC (petroleum ether), $R_f = 0.84$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.40 (d, $J=7.6$ Hz, 4H), 7.20 (d, $J=7.6$ Hz, 4H), 2.34 (s, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 137.4, 133.9, 129.8, 128.5, 21.1. Spectral data were in accordance with the literature.^[6]



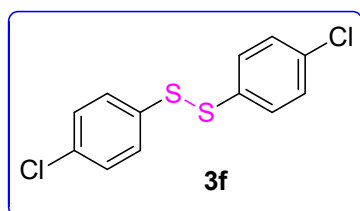
1,2-bis(4-(tert-butyl)phenyl) disulfane (3c). White solid, yield 60.1 mg (91%); TLC (petroleum ether), $R_f = 0.92$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.44 (s, 4H), 7.33 (s, 4H), 1.29 (s, 18H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 150.5, 133.9, 127.7, 126.1, 34.5, 31.3. Spectral data were in accordance with the literature.^[6]



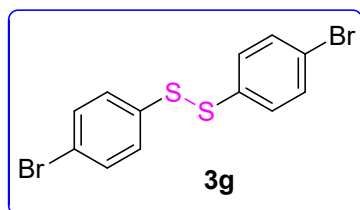
1,2-bis(4-methoxyphenyl) disulfane (3d). White solid, yield 50.0 mg (90%); TLC (petroleum ether:AcOEt=20:1), $R_f = 0.25$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.41 (d, $J=7.6$ Hz, 4H), 6.84 (d, $J=7.6$ Hz, 4H), 3.80 (s, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 159.9, 132.7, 128.4, 114.6, 55.4. Spectral data were in accordance with the literature.^[6]



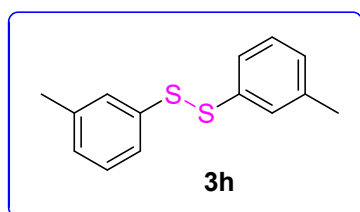
1,2-bis(4-fluorophenyl)disulfane (3e). Colorless oil, yield 46.7 mg (92%); TLC (petroleum ether), $R_f = 0.85$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.43-7.44 (m, 4H), 6.99-7.02 (m, 4H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 156.4, 131.3, 131.2, 116.2 (d, $J=22$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3): δ (ppm) = -113.5. Spectral data were in accordance with the literature.^[6]



1,2-bis(4-chlorophenyl)disulfane (3f). White solid, yield 50.9 mg (89%); TLC (petroleum ether), $R_f = 0.90$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.39 (s, 4H), 7.26 (s, 4H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 135.1, 133.6, 129.3, 129.2. Spectral data were in accordance with the literature.^[6]

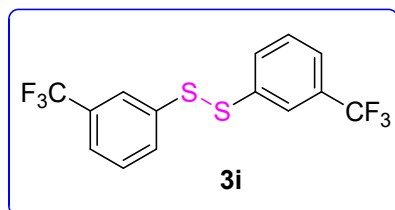


1,2-bis(4-bromophenyl)disulfane (3g). White solid, yield 44.9 mg (60%); TLC (petroleum ether), $R_f = 0.91$. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.42 (d, $J=8.0$ Hz, 4H), 7.34 (d, $J=7.6$ Hz, 4H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ (ppm) = 135.7, 132.2, 129.4, 121.6. Spectral data were in accordance with the literature.^[6]



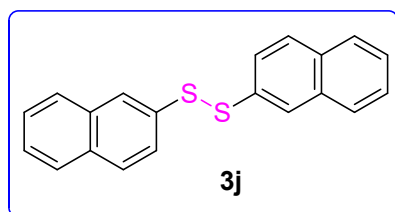
1,2-di-m-tolyldisulfane (3h). Colorless oil, yield 44.7 mg

(91%); TLC (petroleum ether), $R_f = 0.90$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.34-7.36 (m, 4H), 7.22 (t, $J=7.2$ Hz, 2H), 7.06 (d, $J=7.6$ Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 138.9, 136.9, 128.9, 128.04, 128.03, 124.6, 21.4. Spectral data were in accordance with the literature.^[22]



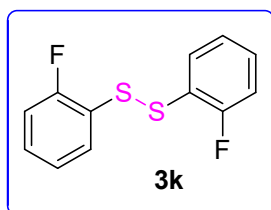
1,2-bis(3-(trifluoromethyl)phenyl)disulfane (**3i**).

Colorless oil, yield 63.7 mg (90%); TLC (petroleum ether), $R_f = 0.81$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.76 (s, 2H), 7.67 (d, $J=7.6$ Hz, 2H), 7.51 (d, $J=7.6$ Hz, 2H), 7.45 (t, $J=7.6$ Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 137.7, 131.6 (d, $J=32$ Hz), 130.6, 129.7, 124.9, 124.2 (q, $J=2$ Hz), 122.2; ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -62.9. Spectral data were in accordance with the literature.^[23]



1,2-di(naphthalen-2-yl)disulfane (**3j**). White solid,

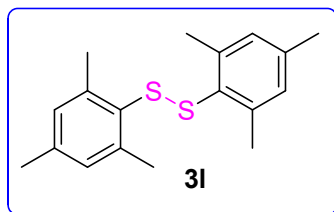
yield 55.3 mg (87%); TLC (petroleum ether), $R_f = 0.75$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.99 (s, 2H), 7.75-7.80 (m, 6H), 7.64 (s, 2H), 7.47 (s, 4H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 134.2, 133.4, 132.5, 128.9, 127.7, 127.4, 126.7, 126.5, 126.2, 125.6. Spectral data were in accordance with the literature.^[6]



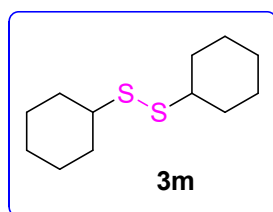
1,2-bis(2-fluorophenyl)disulfane (**3k**). Colorless oil, yield 45.7

mg (90%); TLC (petroleum ether), $R_f = 0.80$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.57 (t, $J=7.6$ Hz, 2H), 7.23-7.28 (m, 2H), 7.03-7.12 (m, 4H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 160.0 (d, $J=246$ Hz), 131.2, 129.7 (d, $J=8$ Hz), 124.7 (d, $J=4$ Hz),

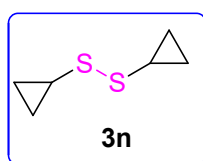
123.5 (d, $J=17$ Hz), 115.7 (d, $J=24$ Hz); ^{19}F NMR (376 MHz, CDCl_3): δ (ppm) = -109.9. Spectral data were in accordance with the literature.^[24]



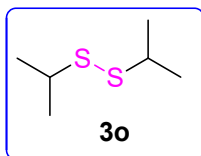
1,2-dimesityldisulfane (31). White solid, yield 49.5 mg (82%); TLC (petroleum ether), $R_f = 0.95$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 6.84 (s, 4H), 2.21 (s, 18H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 143.2, 139.2, 131.5, 128.8, 21.4, 21.1. Spectral data were in accordance with the literature.^[6]



1,2-dicyclohexyldisulfane (3m). Colorless oil, yield 42.3 mg (92%); TLC (petroleum ether), $R_f = 0.86$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 2.68 (s, 2H), 2.03 (s, 4H), 1.79 (s, 4H), 1.56-1.63 (m, 4H), 1.25-1.31 (m, 8H). Spectral data were in accordance with the literature.^[25]



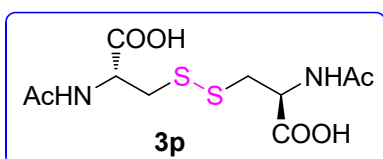
1,2-dicyclopropyldisulfane (3n). Colorless oil, yield 24.8 mg (85%); TLC (petroleum ether), $R_f = 0.90$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 3.25-3.29 (m, 2H), 1.59 (s, 4H), 1.35 (d, $J=7.6$ Hz, 4H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 43.2, 9.2. HRMS (ESI-QTOF) Calcd for $\text{C}_6\text{H}_{10}\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 169.0116; Found 169.0113.



1,2-diisopropyldisulfane (3o). Colorless oil, yield 27.6 mg (92%);

TLC (petroleum ether), $R_f = 0.90$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 2.93-2.96 (m, 2H), 1.27 (d, $J=5.2$ Hz, 12H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 41.4, 22.6.

Spectral data were in accordance with the literature.^[26]

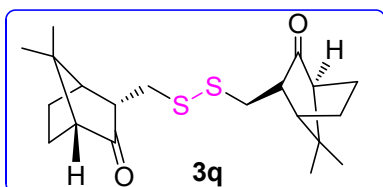


(S)-2-acetamido-3-(((R)-2-acetamido-2-

carboxyethyl)disulfanyl)propanoic acid (3p). White solid, yield 39.5 mg (61%); TLC

(AcOEt:DCM=1:1), $R_f = 0.21$. ^1H NMR (400 MHz, $\text{D}_6\text{-DMSO}$): δ (ppm) = 8.27 (s, 2H), 2.71-2.77 (m, 4H), 2.19 (s, 2H), 1.84 (s, 6H); ^{13}C NMR (101 MHz, $\text{D}_6\text{-DMSO}$):

δ (ppm) = 172.3, 171.6, 54.8, 25.7, 22.5. HRMS (ESI-QTOF) Calcd for $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_6\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 347.0342; Found 347.0338.



(1S,3S)-3-((((1R,2R,4R)-7,7-dimethyl-3-

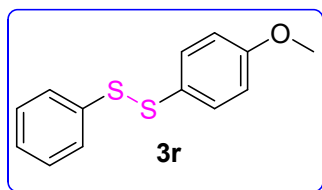
oxobicyclo[2.2.1]heptan-2-yl)methyl)disulfanyl)methyl)-7,7-

dimethylbicyclo[2.2.1]heptan-2-one (3q). White solid, yield 36.6 mg (50%); TLC

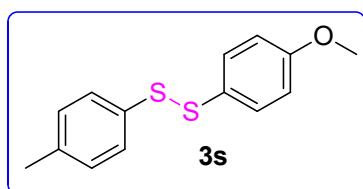
(petroleum ether:AcOEt=10:1), $R_f = 0.48$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 4.23 (d, $J=14.8$ Hz, 2H), 3.69 (d, $J=14.8$ Hz, 2H), 2.35-2.40 (m, 4H), 2.12 (s, 2H),

2.04 (s, 2H), 1.91-1.96 (m, 2H), 1.71-1.73 (m, 2H), 1.41-1.46 (m, 2H), 1.07 (s, 6H), 0.86 (s, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 212.8, 64.3, 59.6, 48.2, 42.7,

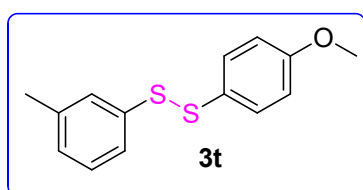
42.3, 26.8, 25.2, 19.6, 19.5. HRMS (ESI-QTOF) Calcd for $\text{C}_{20}\text{H}_{30}\text{O}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$ 389.1579; Found 389.1572.



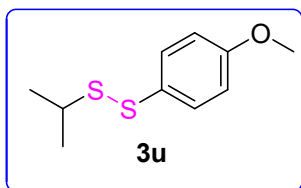
1-(4-methoxyphenyl)-2-phenyldisulfane (3r). Colorless oil, yield 23.8 mg (48%); TLC (petroleum ether:AcOEt=15:1), $R_f = 0.35$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.51 (d, $J=7.2$ Hz, 2H), 7.42 (d, $J=7.2$ Hz, 2H), 7.29-7.33 (m, 2H), 7.23-7.25 (m, 1H), 6.82-6.84 (m 2H), 3.78 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 159.8, 137.5, 132.7, 131.8, 129.0, 128.2, 127.2, 114.7, 55.4. Spectral data were in accordance with the literature.^[27]



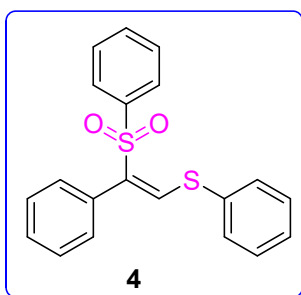
1-(4-methoxyphenyl)-2-phenyldisulfane (3s). Colorless oil, yield 26.2 mg (50%); TLC (petroleum ether:AcOEt=15:1), $R_f = 0.37$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.39-7.44 (m, 4H), 7.12 (d, $J=8.0$ Hz, 2H), 6.84 (d, $J=8.8$ Hz, 2H), 3.79 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 159.8, 137.6, 134.1, 132.0, 129.8, 129.2, 114.6, 55.4, 21.1. Spectral data were in accordance with the literature.^[27]



1-(4-methoxyphenyl)-2-(m-tolyl)disulfane (3t). Colorless oil, yield 23.6 mg (45%); TLC (petroleum ether:AcOEt=40:1), $R_f = 0.31$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.43 (d, $J=9.2$ Hz, 2H), 7.33 (s, 2H), 7.21 (t, $J=8.0$ Hz, 1H), 7.08 (d, $J=7.2$ Hz, 1H), 6.84 (d, $J=8.8$ Hz, 2H), 3.79 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 159.7, 138.9, 137.2, 131.7, 128.9, 128.7, 128.2, 128.1, 125.2, 114.7, 55.4, 21.4. Spectral data were in accordance with the literature.^[28]

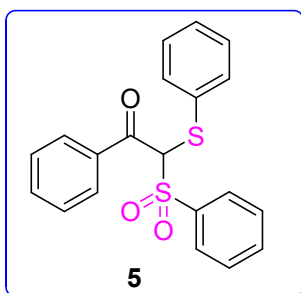


1-isopropyl-2-(4-methoxyphenyl)disulfane (3u). Colorless oil, yield 16.7 mg (39%); TLC (petroleum ether:AcOEt=20:1), $R_f = 0.56$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.48 (d, $J=7.2$ Hz, 2H), 6.85 (d, $J=8.8$ Hz, 2H), 3.80 (s, 3H), 3.05 (q, $J=6.8$ Hz, 1H), 1.29 (d, $J=6.8$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 159.2, 130.9, 129.1, 114.5, 55.4, 41.3, 22.4. Spectral data were in accordance with the literature.^[29]



(Z)-phenyl(2-phenyl-2-(phenylsulfonyl)vinyl)sulfane (4).

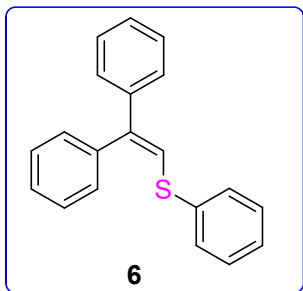
Yellow solid, yield 63.4 mg (90%); TLC (petroleum ether:AcOEt=5:1), $R_f = 0.56$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.15 (s, 1H), 7.62 (d, $J=7.6$ Hz, 2H), 7.47-7.54 (m, 3H), 7.33-7.41 (m, 8H), 7.21 (d, $J=6.0$ Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 144.0, 139.2, 135.9, 133.1, 132.7, 131.2, 130.5, 130.2, 129.6, 129.4, 128.8, 128.7, 128.2. Spectral data were in accordance with the literature.^[2]



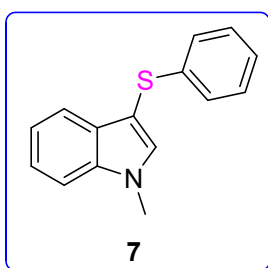
1-phenyl-2-(phenylsulfonyl)-2-(phenylthio)ethanone (5).

White solid, yield 63.3 mg (86%); TLC (petroleum ether:AcOEt=4:1), $R_f = 0.34$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.00 (d, $J=8.0$ Hz, 2H), 7.86 (d, $J=8.0$ Hz, 2H), 7.42-7.65 (m, 8H), 7.24-7.29 (m, 3H), 5.83 (s, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 189.2, 136.3, 135.0, 134.5, 134.4, 133.6, 131.9, 130.7, 129.5, 129.4, 129.2,

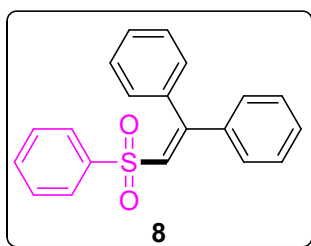
128.9, 128.7, 75.4. Spectral data were in accordance with the literature.^[3]



(2,2-diphenylvinyl)(phenyl)sulfane (**6**). Colorless oil, yield 54.7 mg (95%); TLC (petroleum ether), $R_f = 0.68$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.49-7.53 (m, 4H), 7.40-7.46 (m, 3H), 7.32-7.38 (m, 8H), 6.96 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 141.5, 141.1, 139.2, 136.6, 129.8, 129.6, 129.2, 128.5, 128.4, 127.9, 127.4, 127.3, 126.8, 124.2. Spectral data were in accordance with the literature.^[4]



1-methyl-3-(phenylthio)-1H-indole (**7**). White solid, yield 40.2 mg 84%); TLC (petroleum ether:AcOEt=20:1), $R_f = 0.45$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.66 (d, $J=8.0$ Hz, 1H), 7.41 (d, $J=8.0$ Hz, 1H), 7.32-7.35 (m, 2H), 7.14-7.23 (m, 5H), 7.08 (t, $J=6.8$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 139.7, 137.6, 135.1, 129.8, 128.7, 125.7, 124.7, 122.6, 120.5, 119.7, 100.5, 33.1. Spectral data were in accordance with the literature.^[5]



(2-(phenylsulfonyl)ethene-1,1-diyl)dibenzene (**8**). Colorless oil, yield 14.7 mg (23%); TLC (petroleum ether: AcOEt=5:1), $R_f = 0.32$. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.58 (d, $J= 8.0$ Hz, 2H), 7.46-7.50 (m, 1H), 7.27-7.38

(m, 8H), 7.21 (d, $J = 7.2$ Hz, 2H), 7.07-7.09 (m, 2H), 6.87 (s, 1H), 7.04 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) = 155.2, 141.4, 139.1, 135.5, 132.9, 130.4, 129.8, 128.9, 128.73, 128.70, 128.6, 128.2, 127.9, 127.6. Spectral data were in accordance with the literature.^[30]

14. References

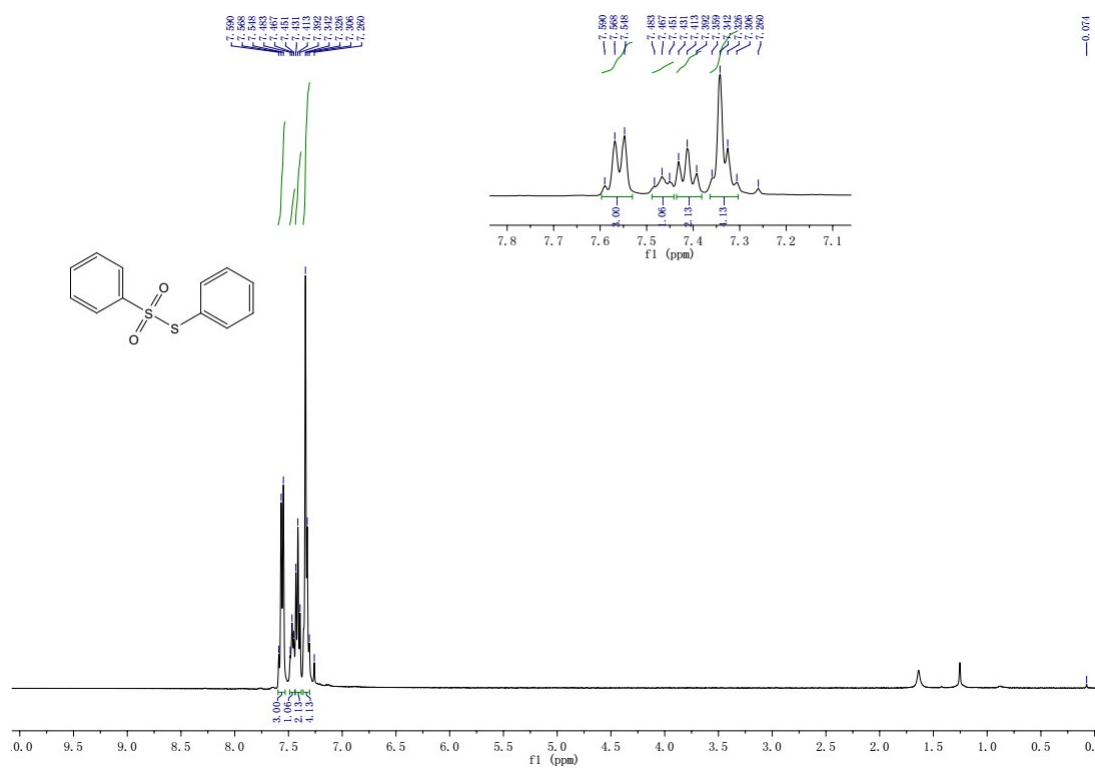
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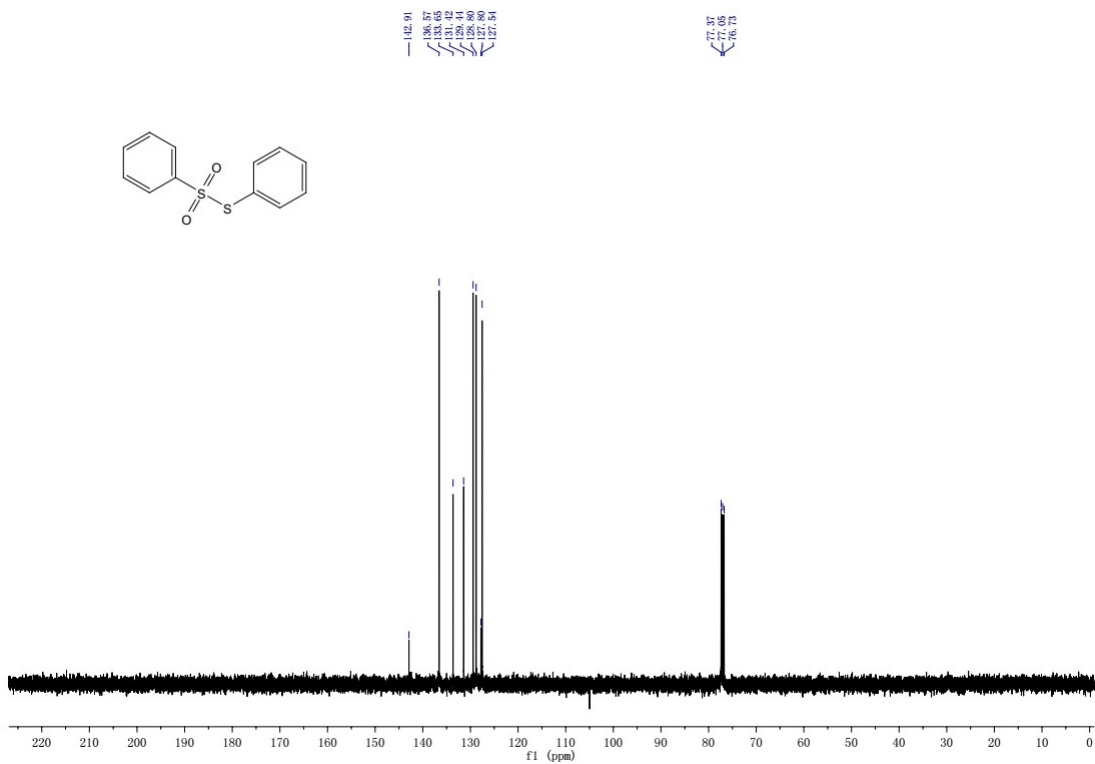
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15. Copies of NMR Spectra for Compounds

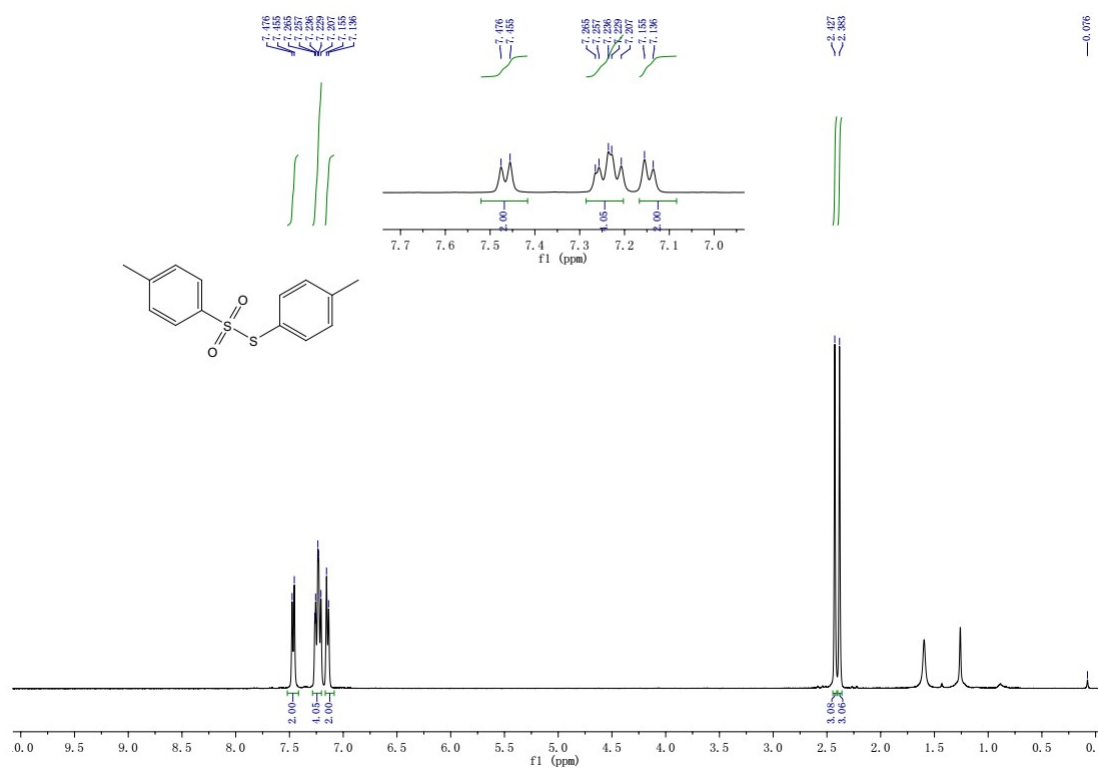
¹H NMR spectrum for compound **2a** (CDCl₃, 400 MHz)



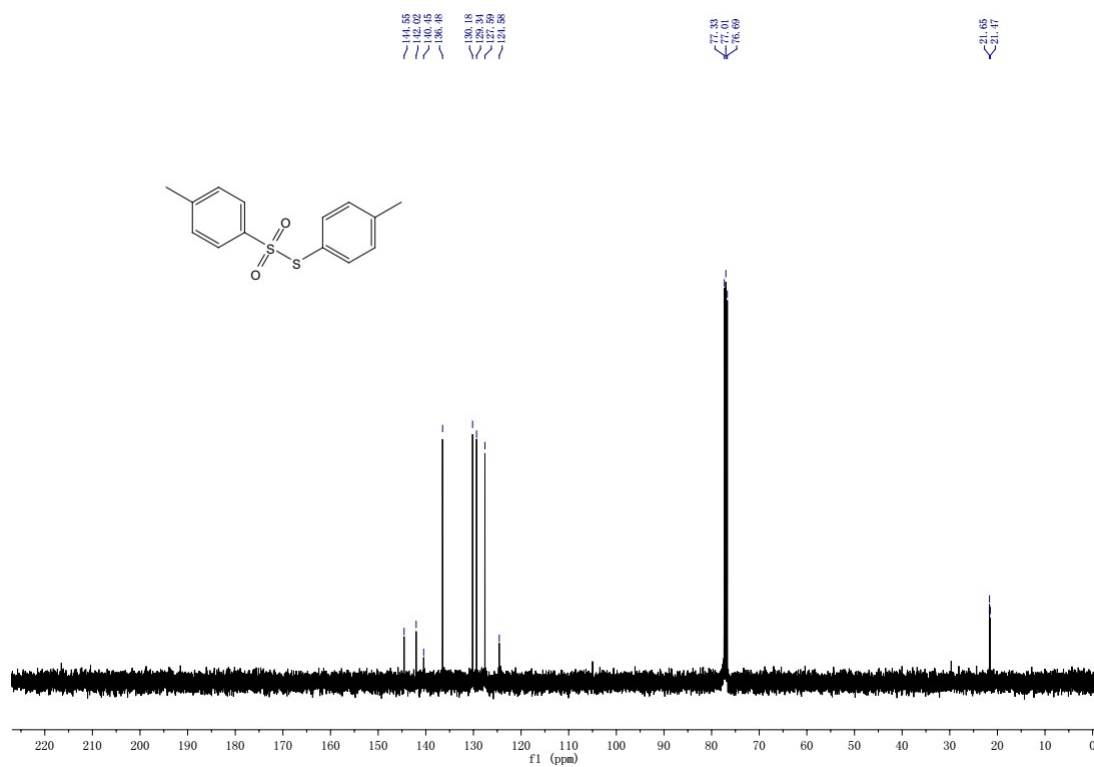
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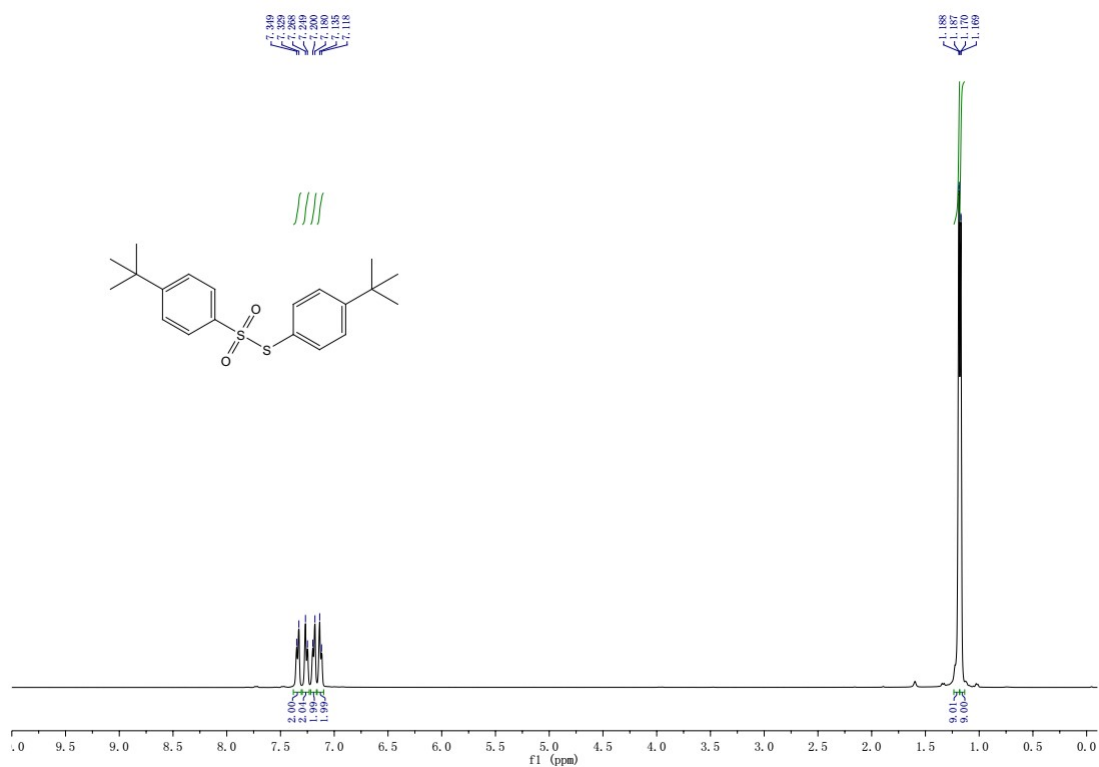
¹H NMR spectrum for compound **2b** (CDCl₃, 400 MHz)



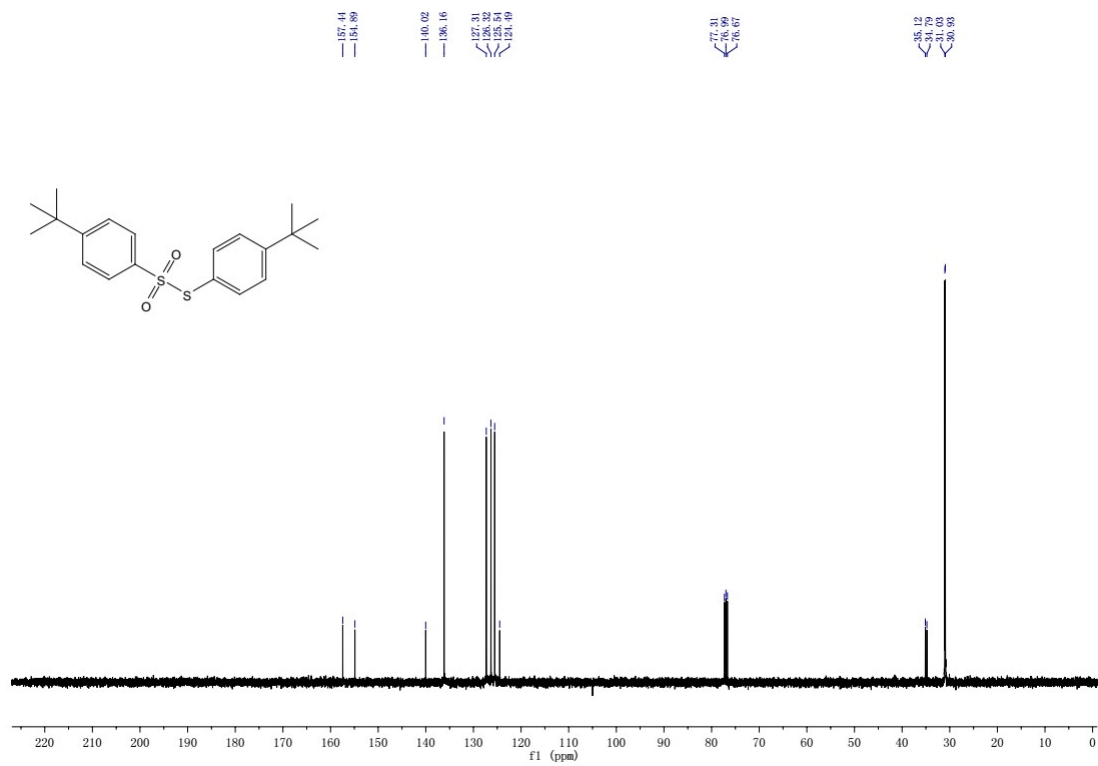
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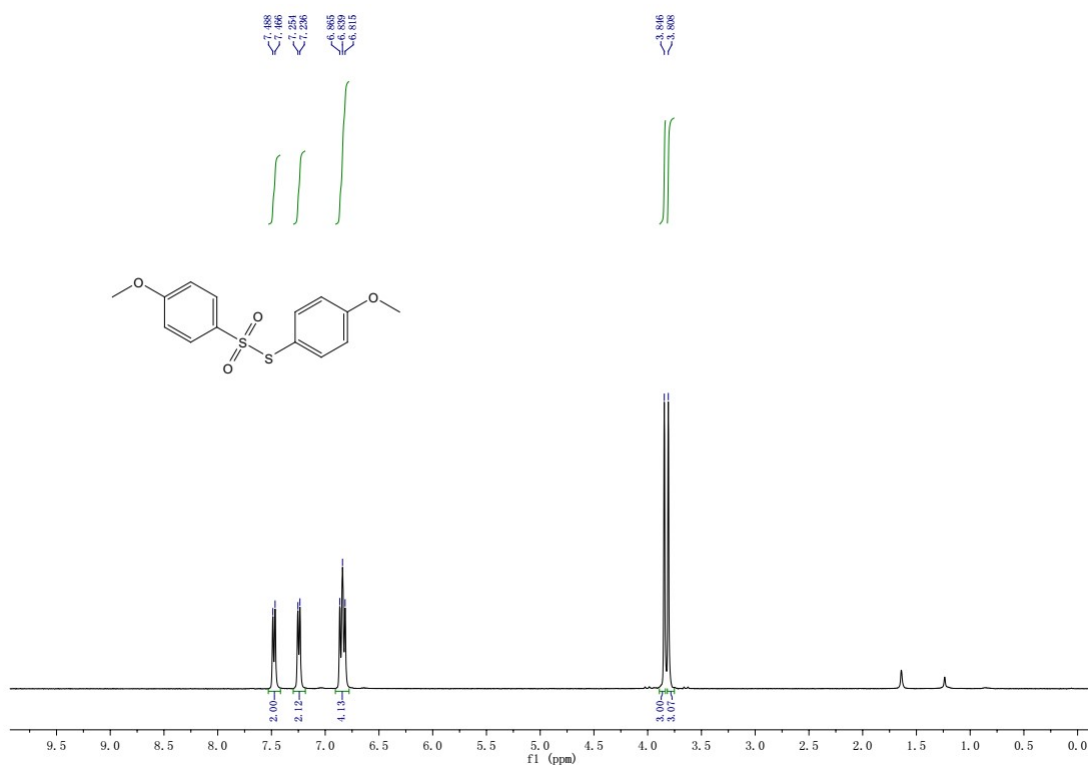
¹H NMR spectrum for compound 2c (CDCl₃, 400 MHz)



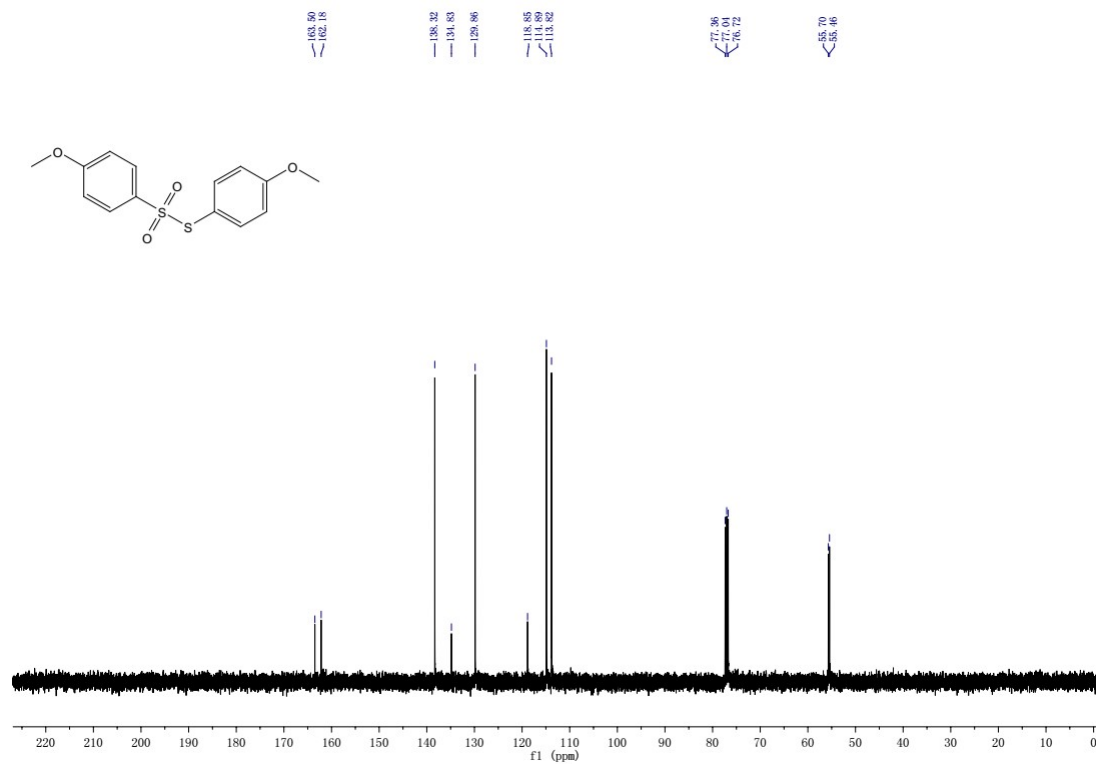
¹³C NMR spectrum for compound **2c** (CDCl₃, 101 MHz)



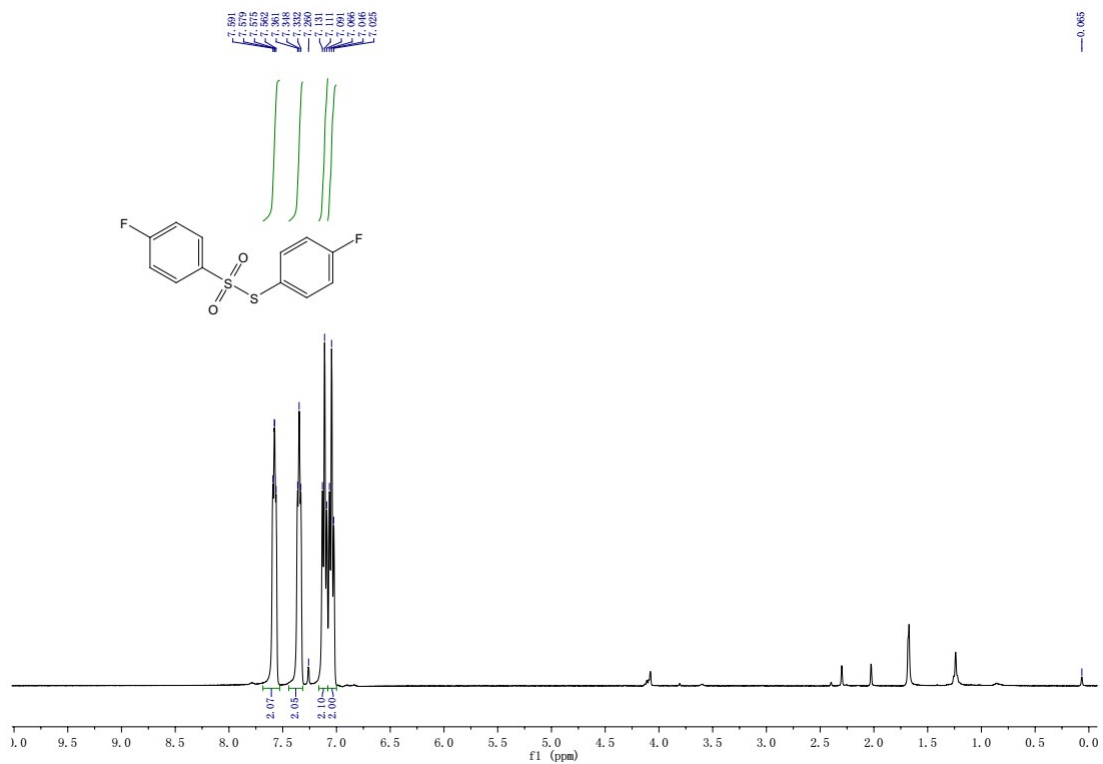
¹H NMR spectrum for compound **2d** (CDCl₃, 400 MHz)



^{13}C NMR spectrum for compound **2d** (CDCl_3 , 101 MHz)

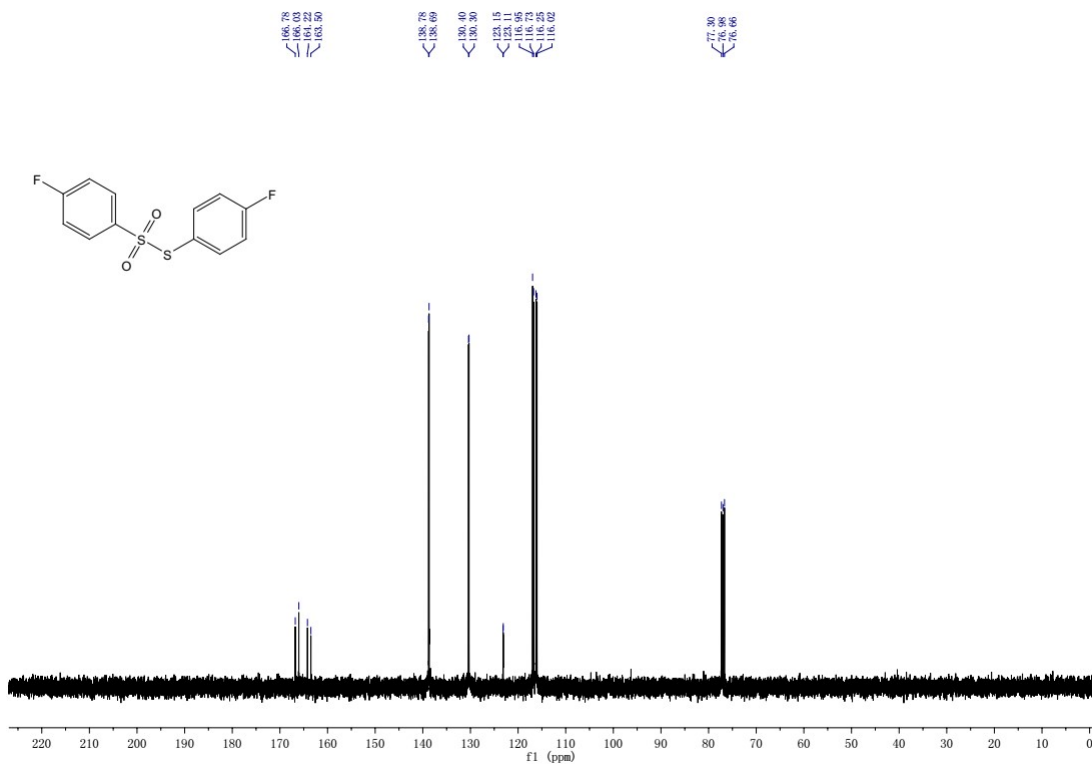


^1H NMR spectrum for compound **2e** (CDCl_3 , 400 MHz)

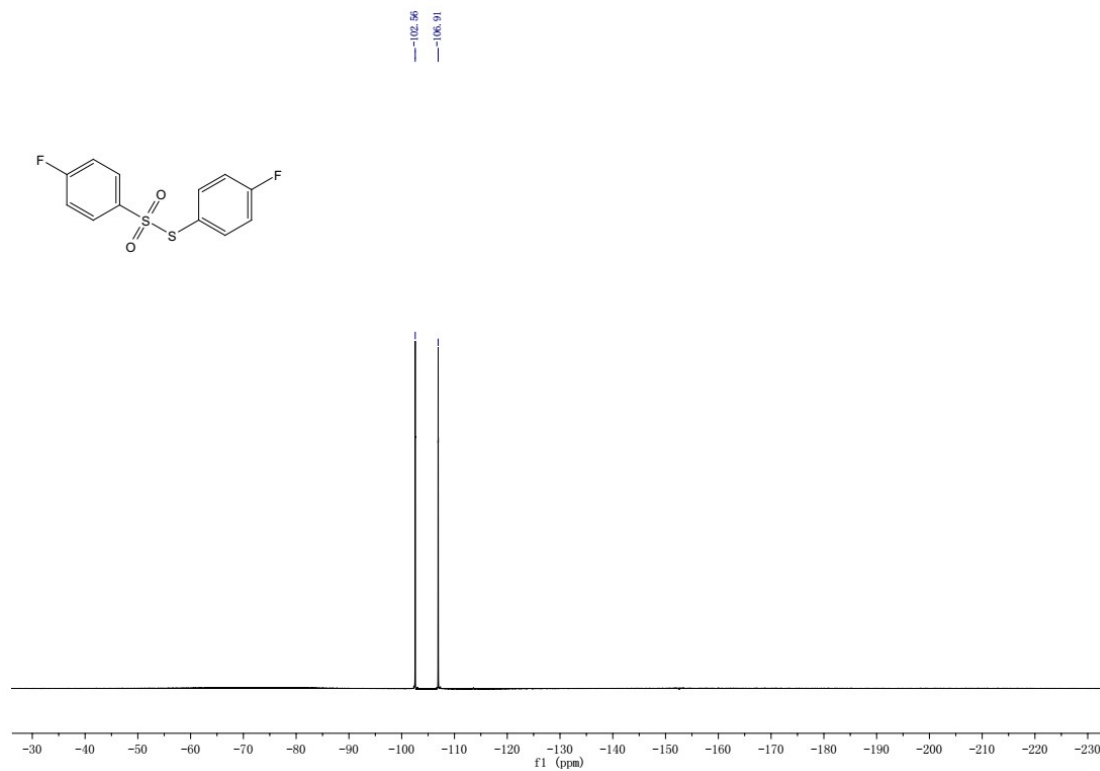


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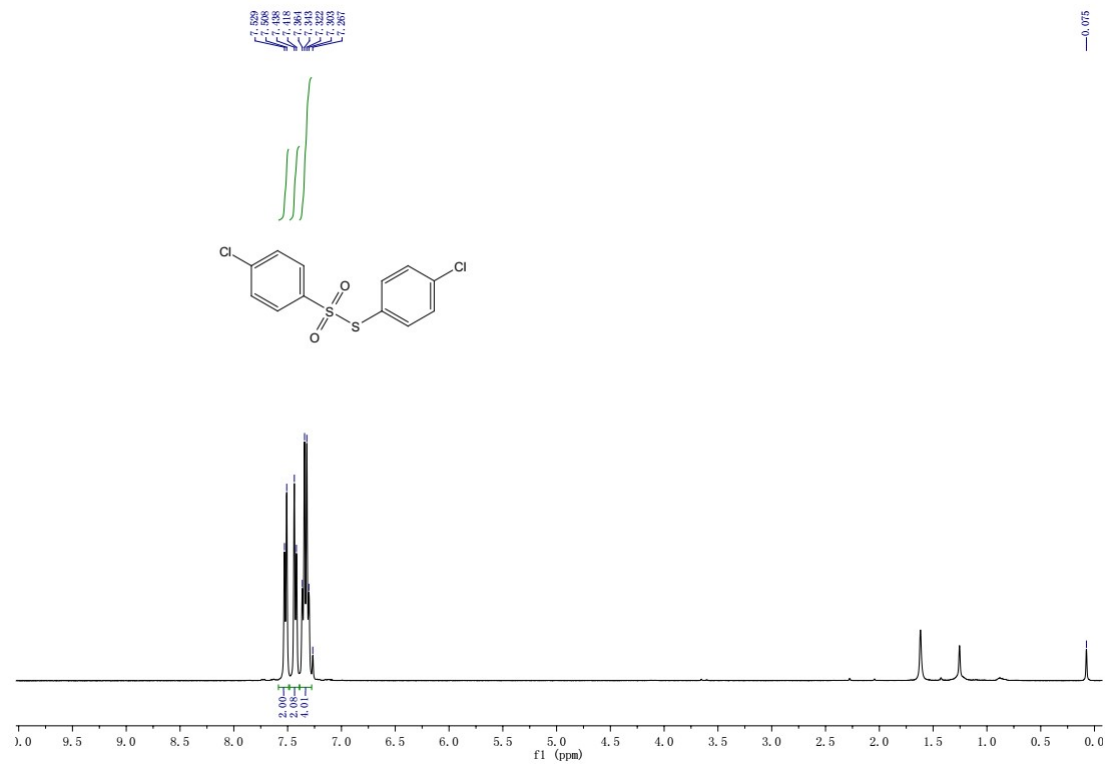
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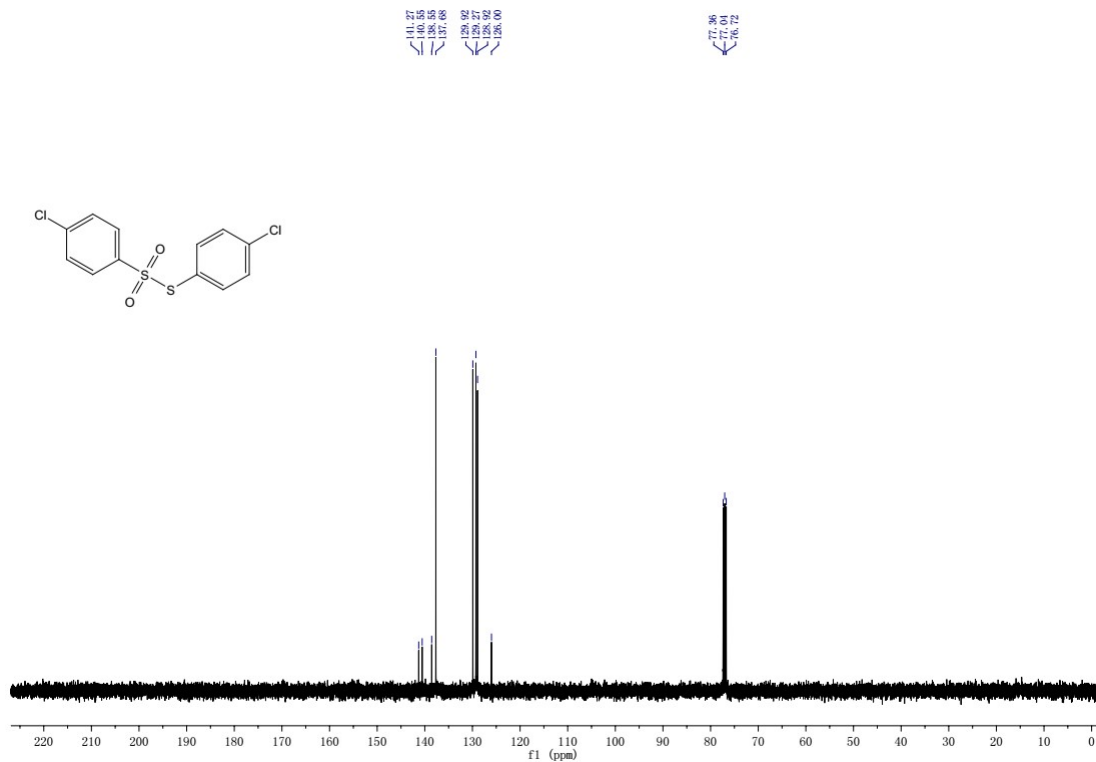
¹⁹F NMR spectrum for compound **2e** (CDCl₃, 376 MHz)



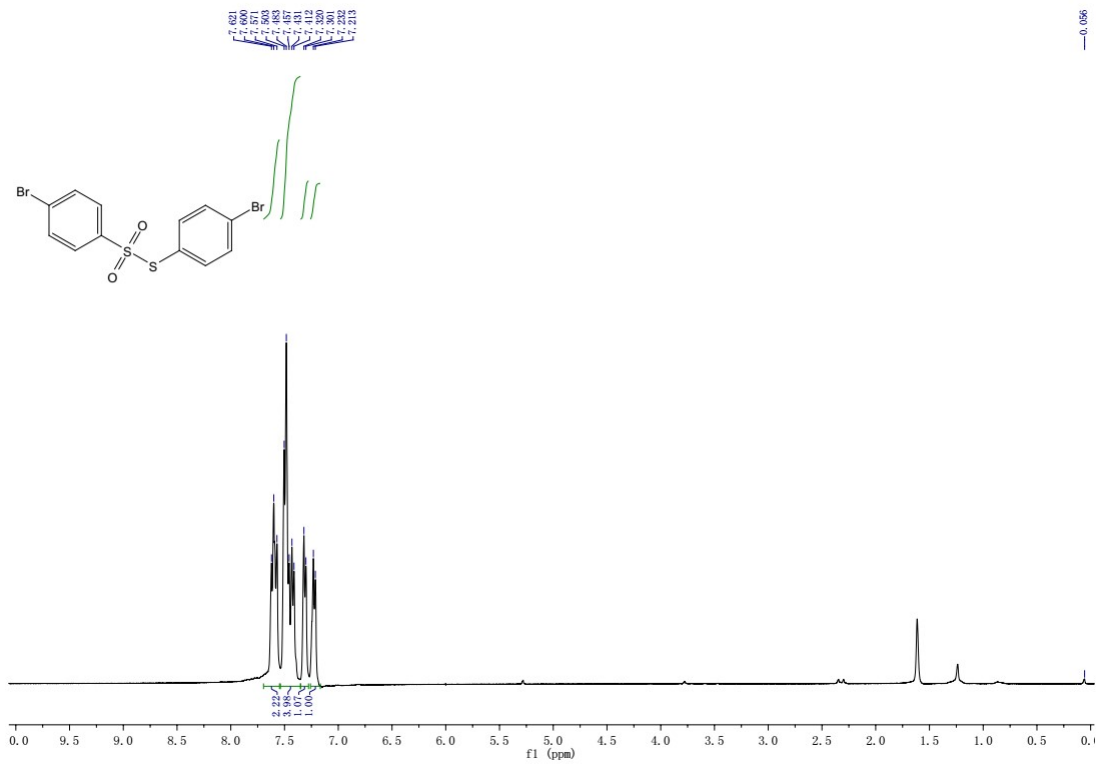
^1H NMR spectrum for compound **2f** (CDCl_3 , 400 MHz)



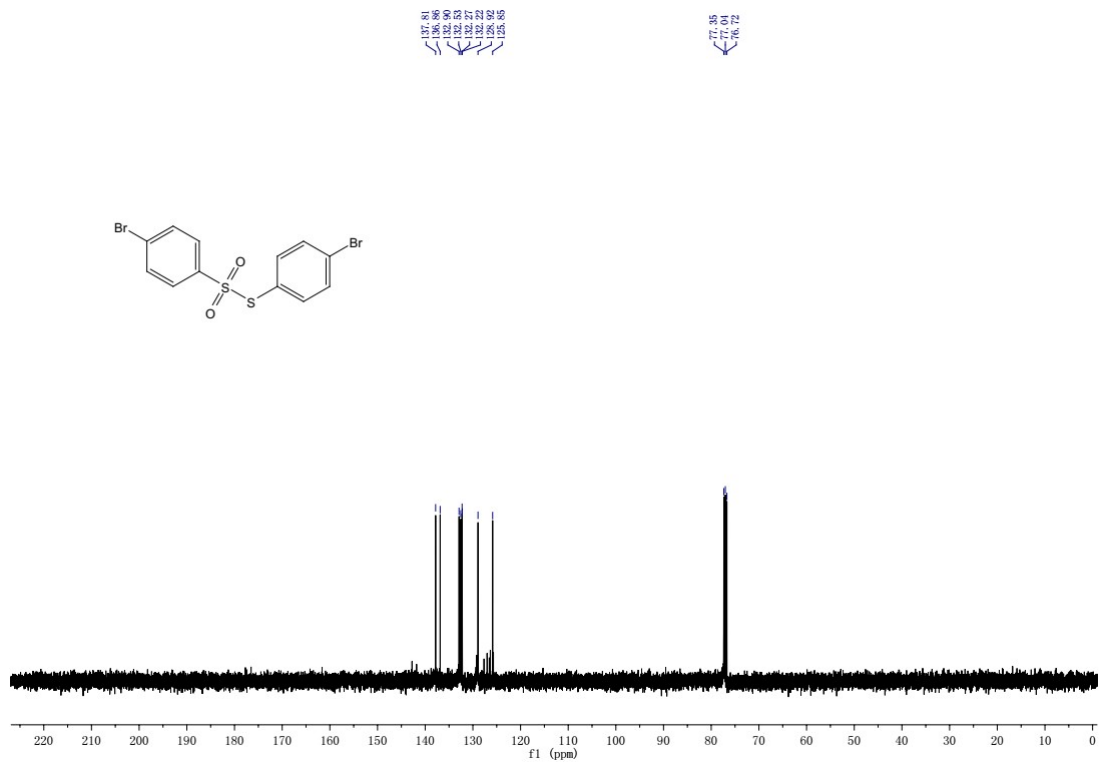
^{13}C NMR spectrum for compound **2f** (CDCl_3 , 101 MHz)



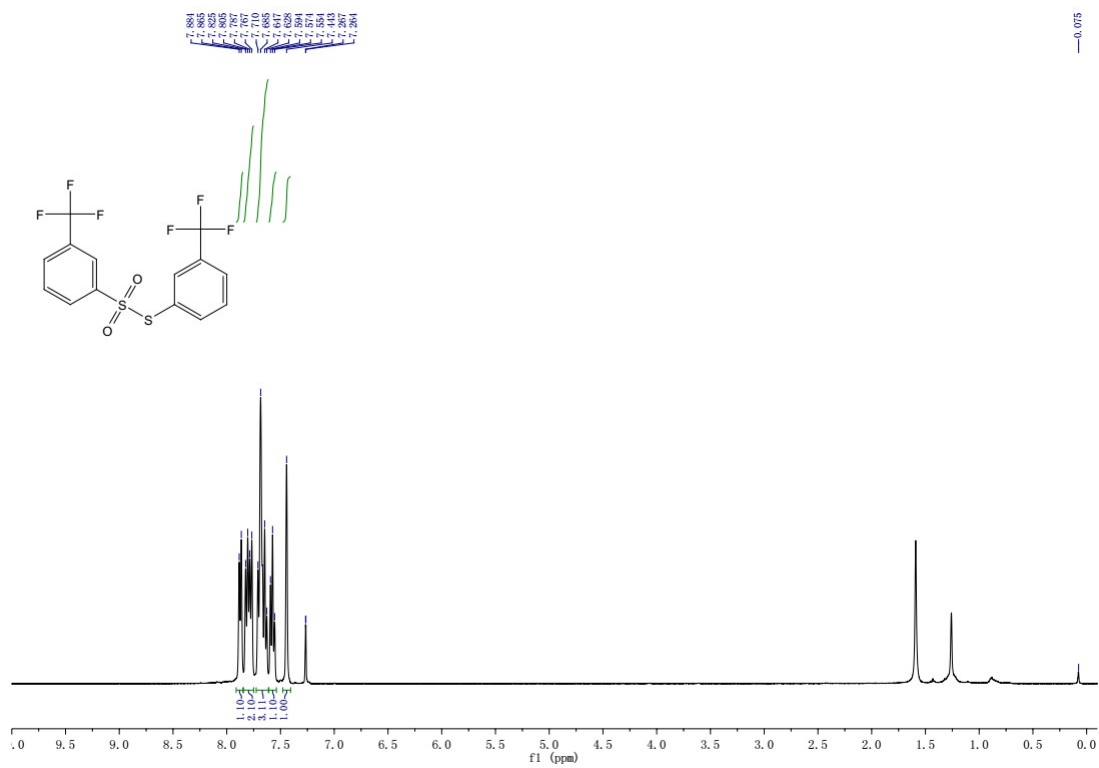
¹H NMR spectrum for compound **2g** (CDCl₃, 400 MHz)



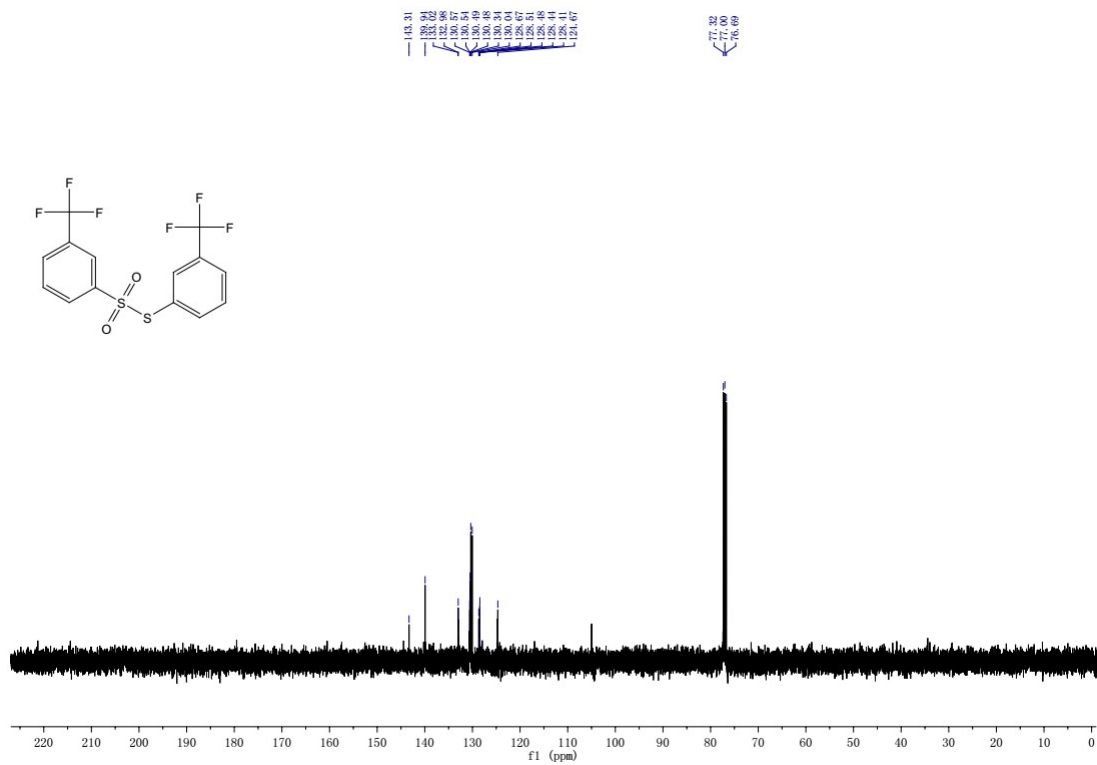
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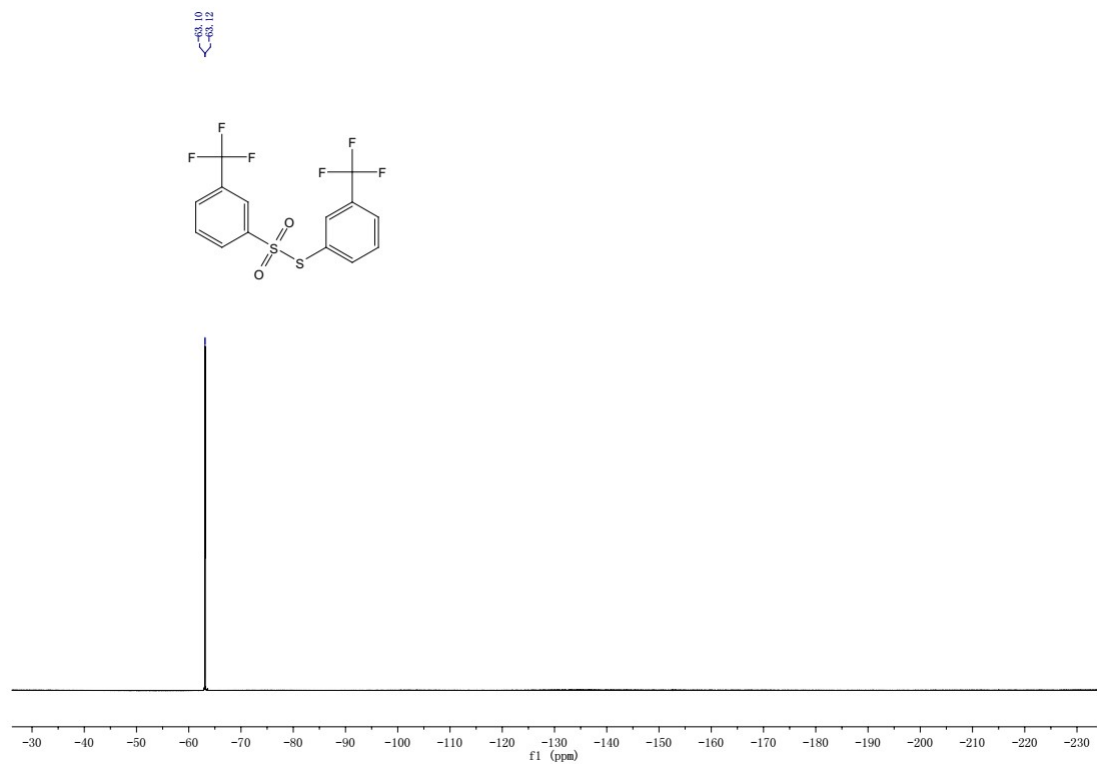
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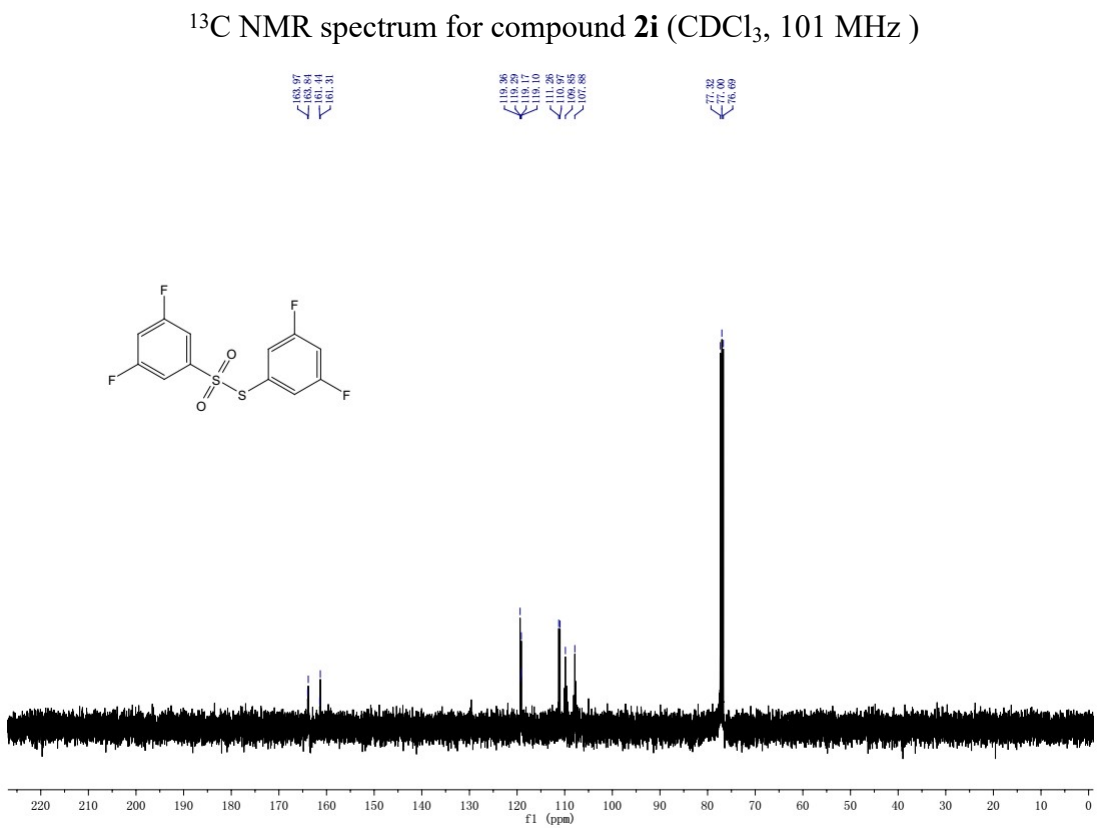
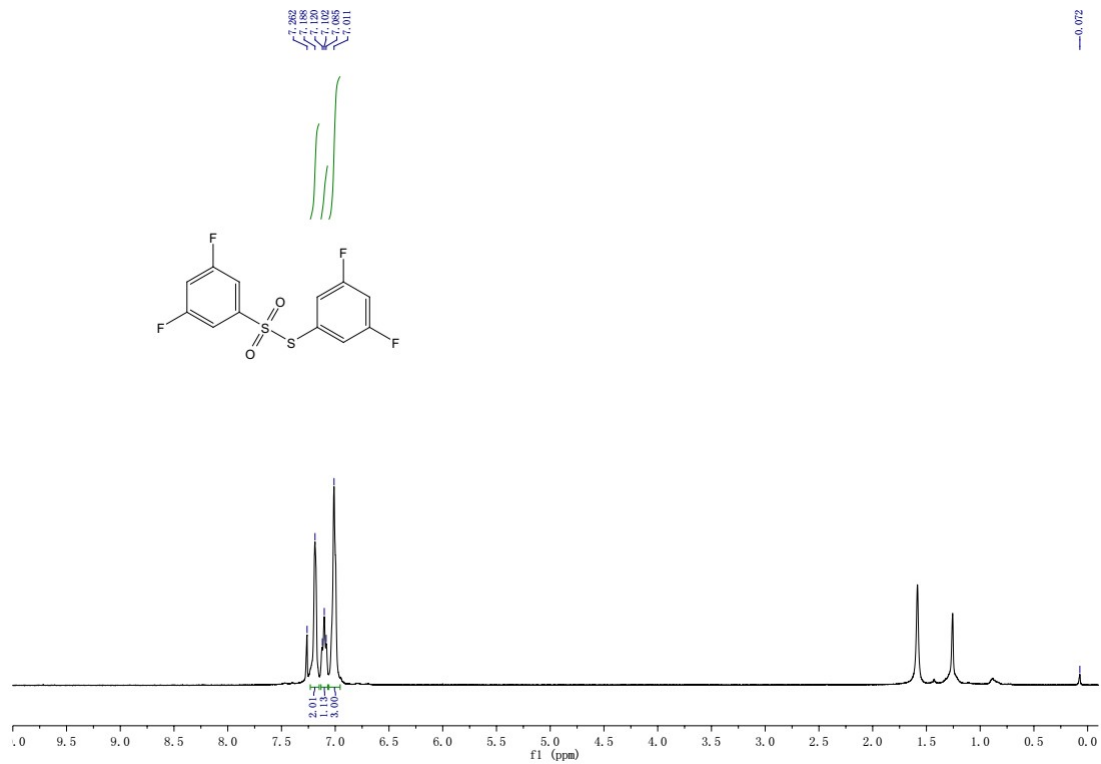
¹³C NMR spectrum for compound **2h** (CDCl₃, 101 MHz)



¹⁹F NMR spectrum for compound **2h** (CDCl₃, 376 MHz)



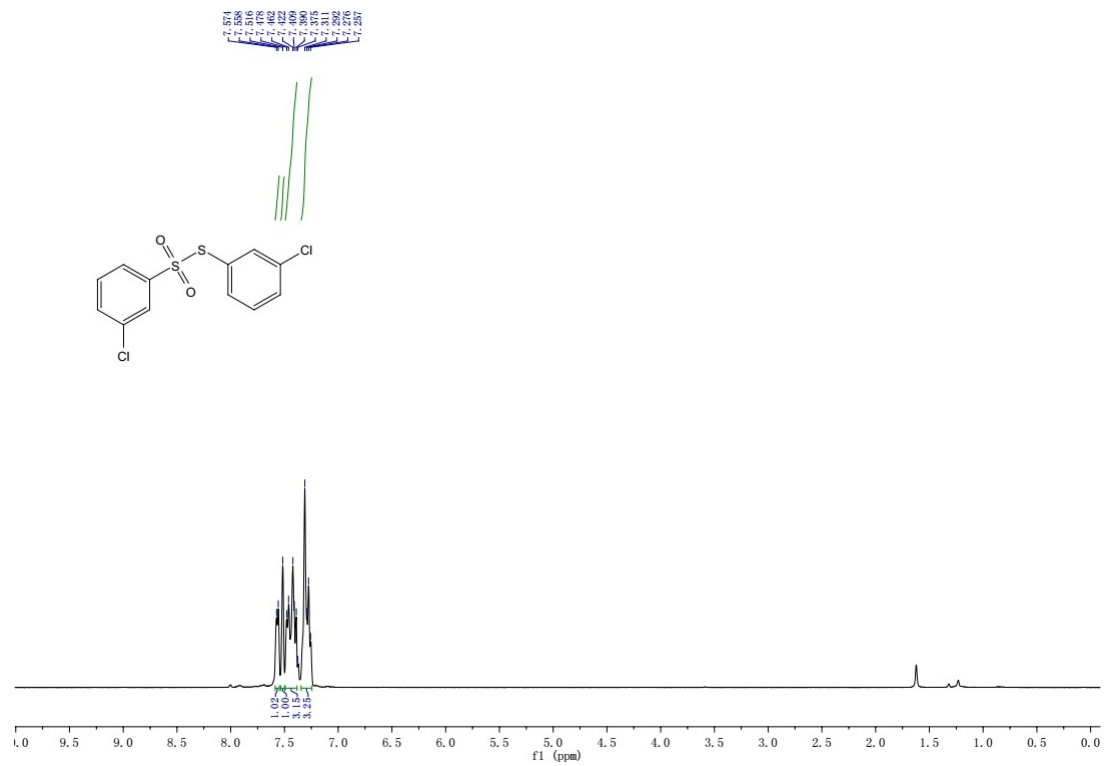
¹H NMR spectrum for compound **2i** (CDCl₃, 400 MHz)



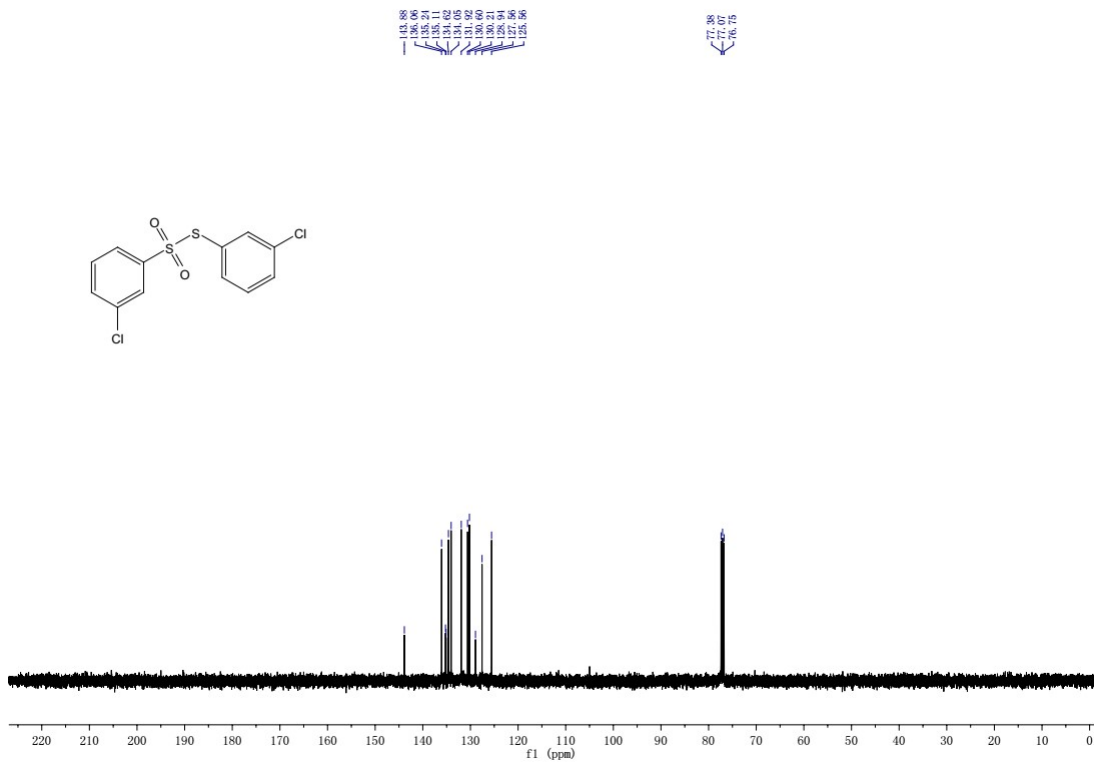
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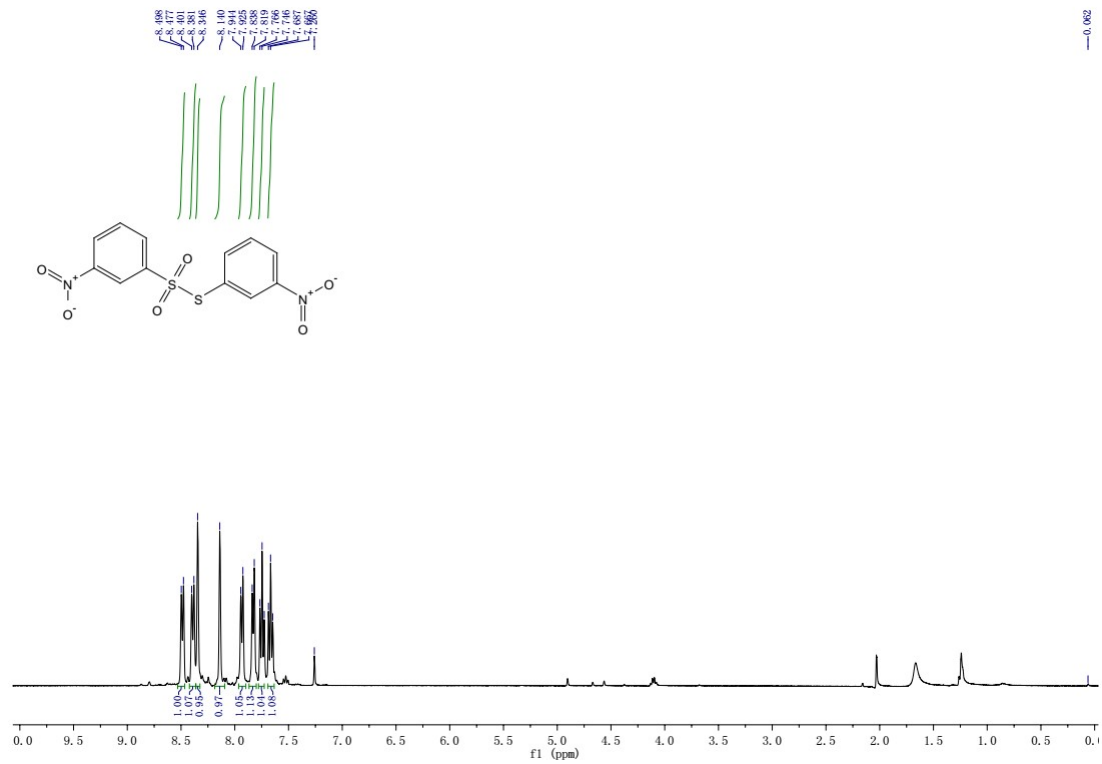
^1H NMR spectrum for compound **2j** (CDCl_3 , 400 MHz)



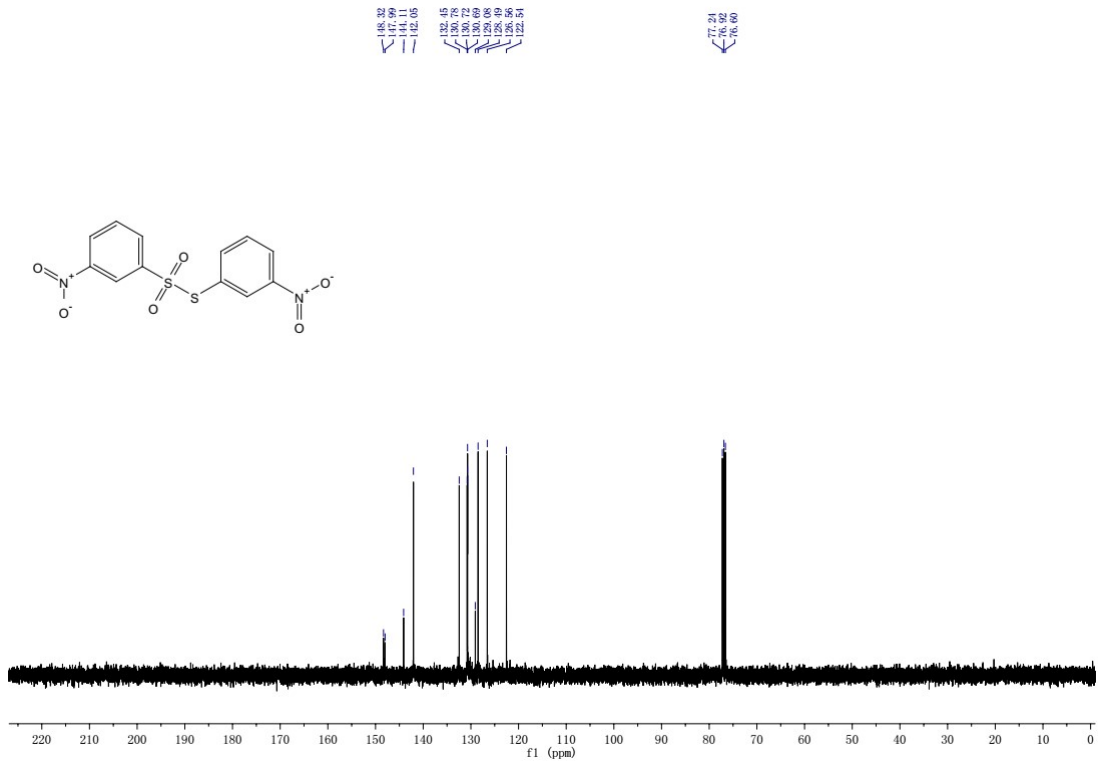
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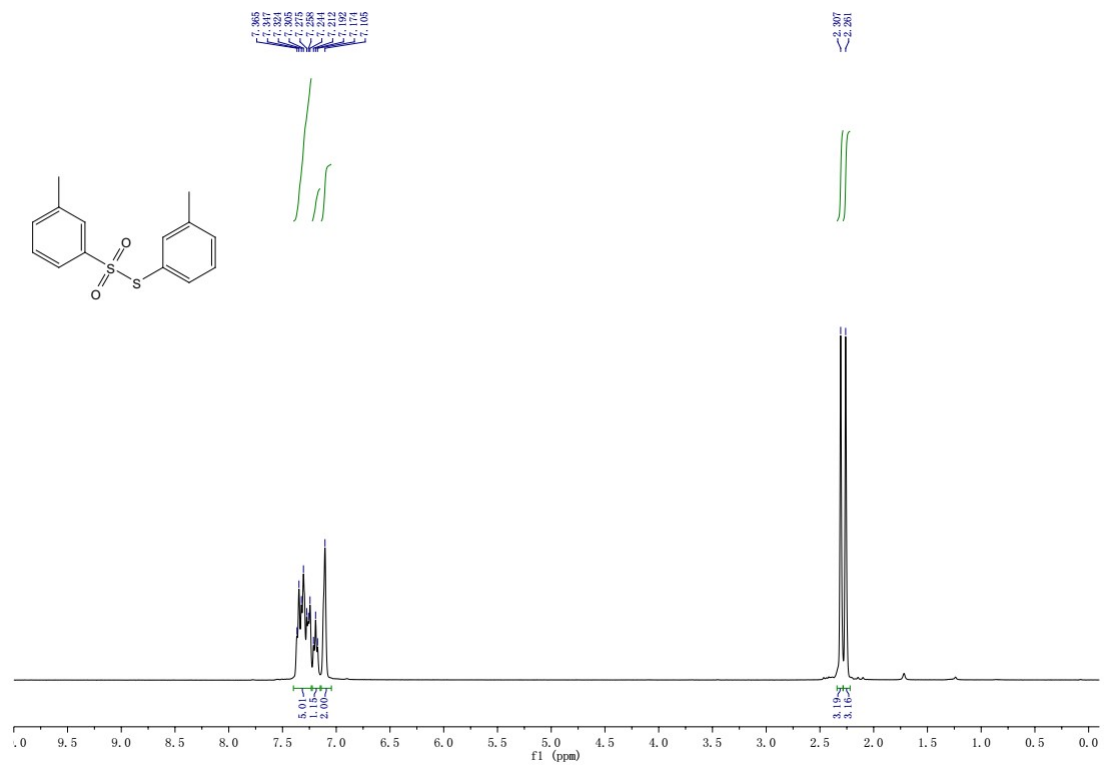
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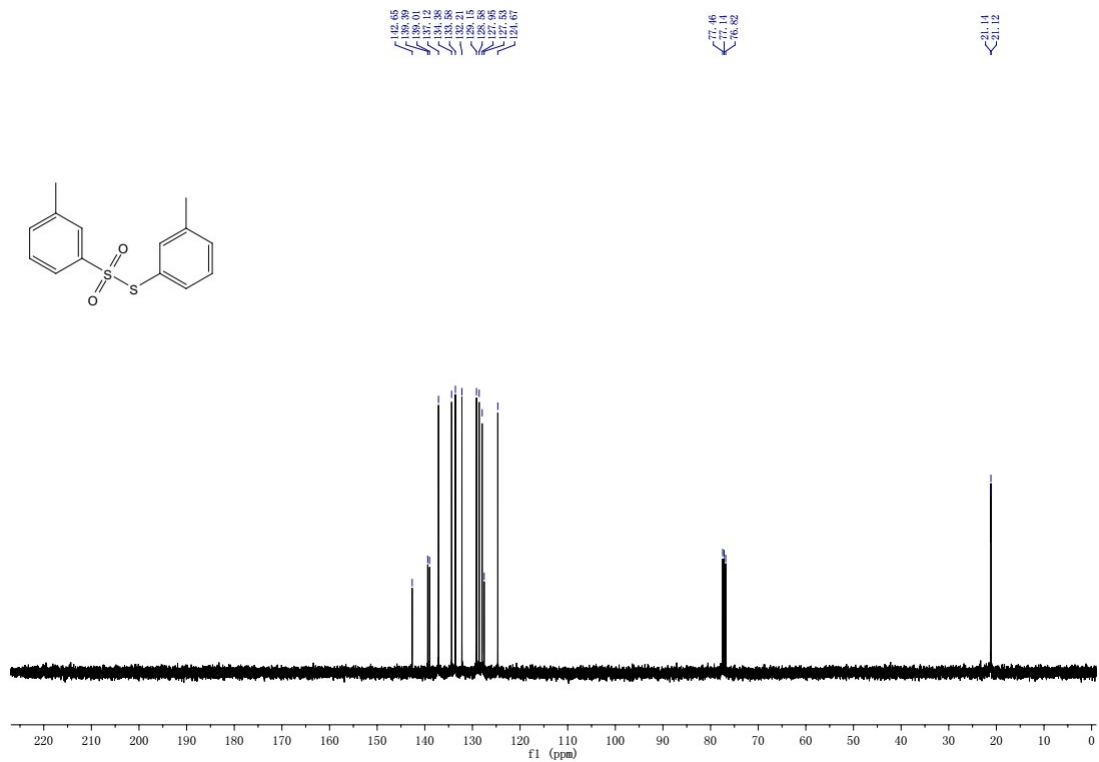
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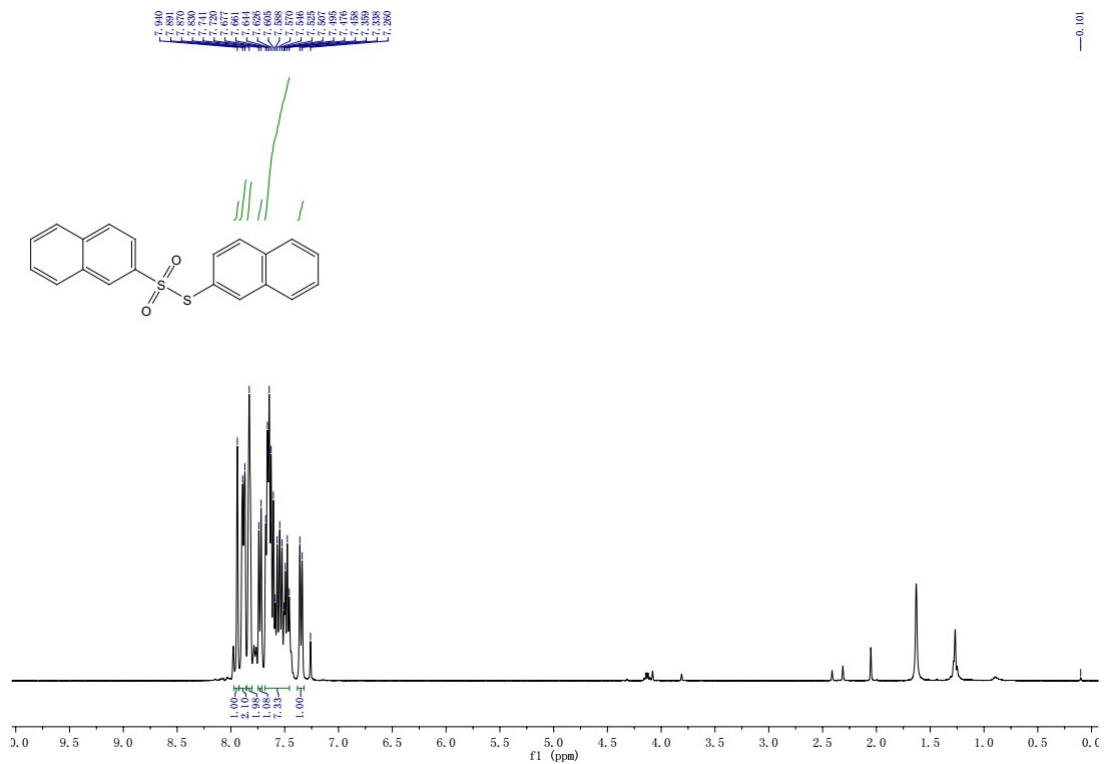
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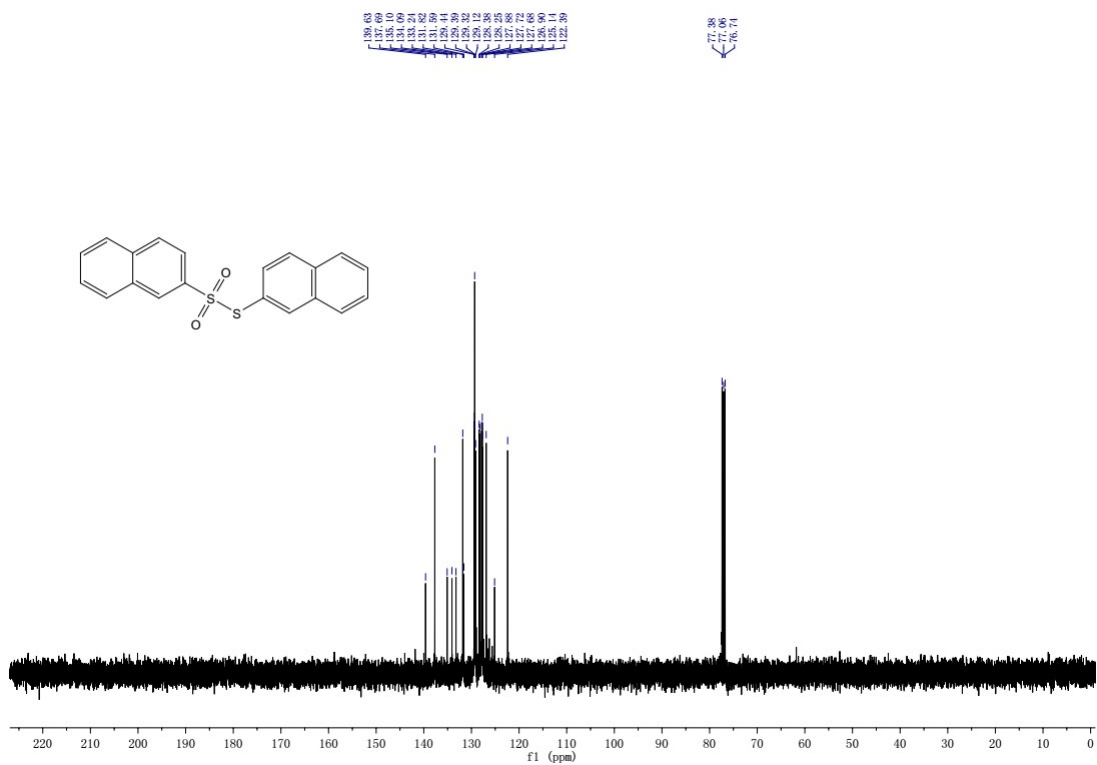
¹³C NMR spectrum for compound **2I** (CDCl₃, 101 MHz)



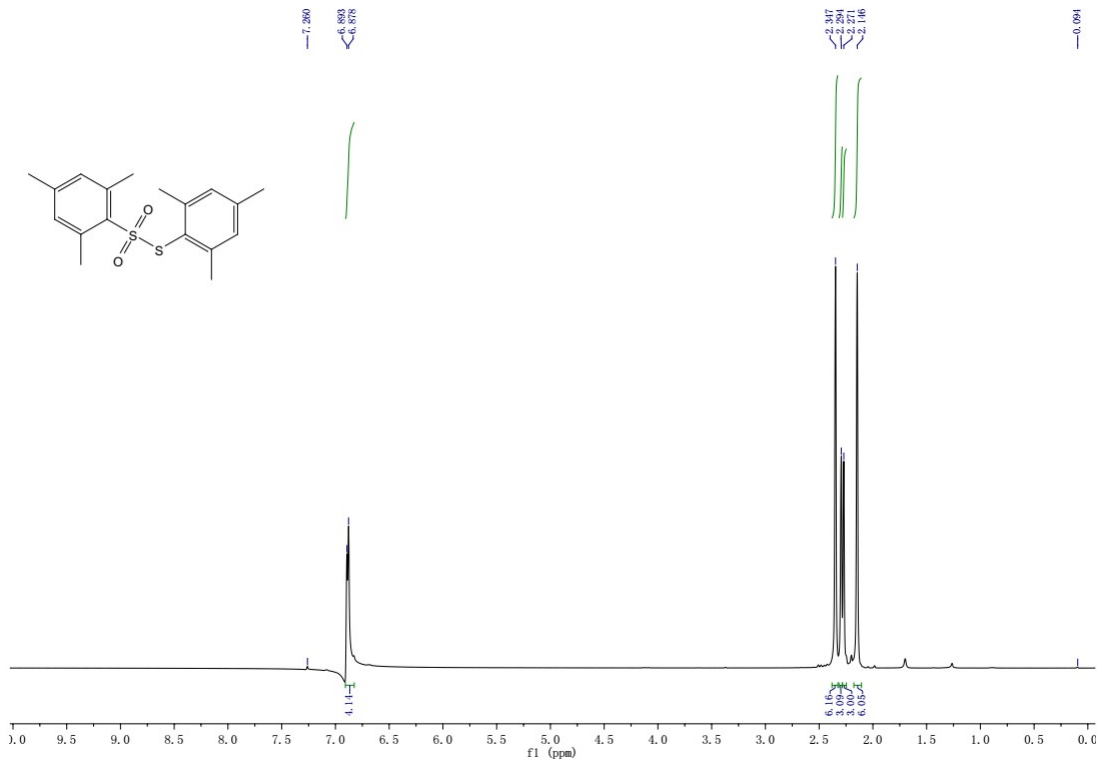
¹H NMR spectrum for compound **2m** (CDCl₃, 400 MHz)



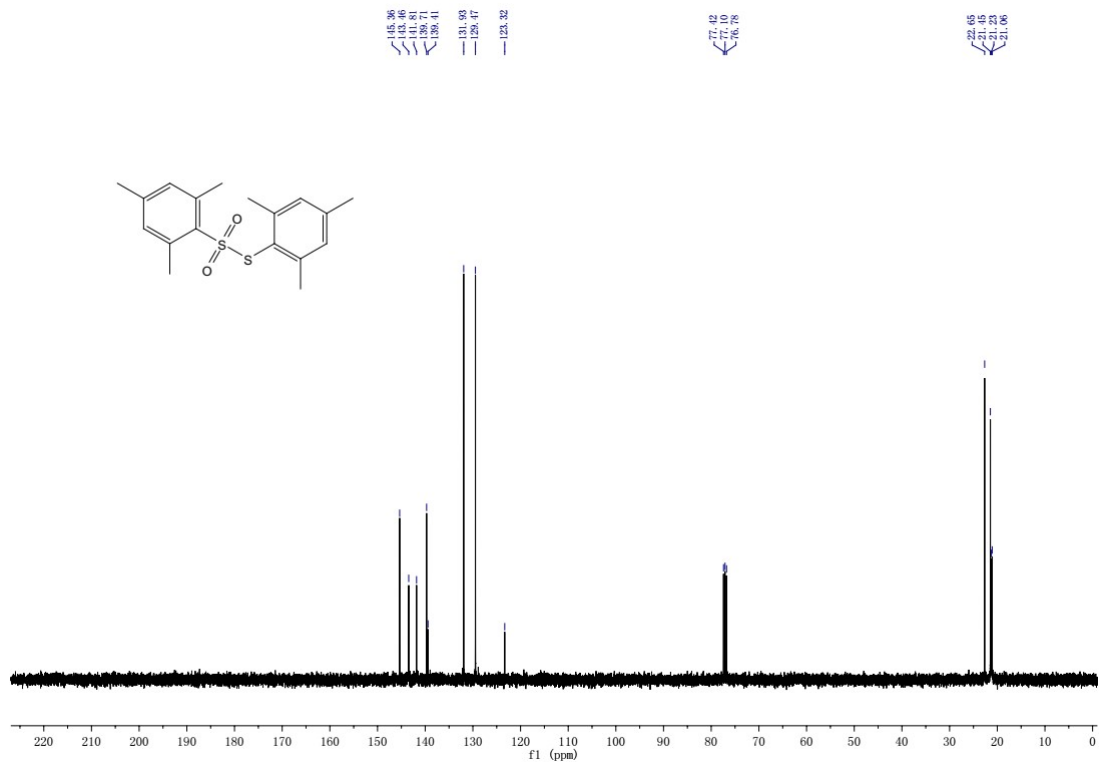
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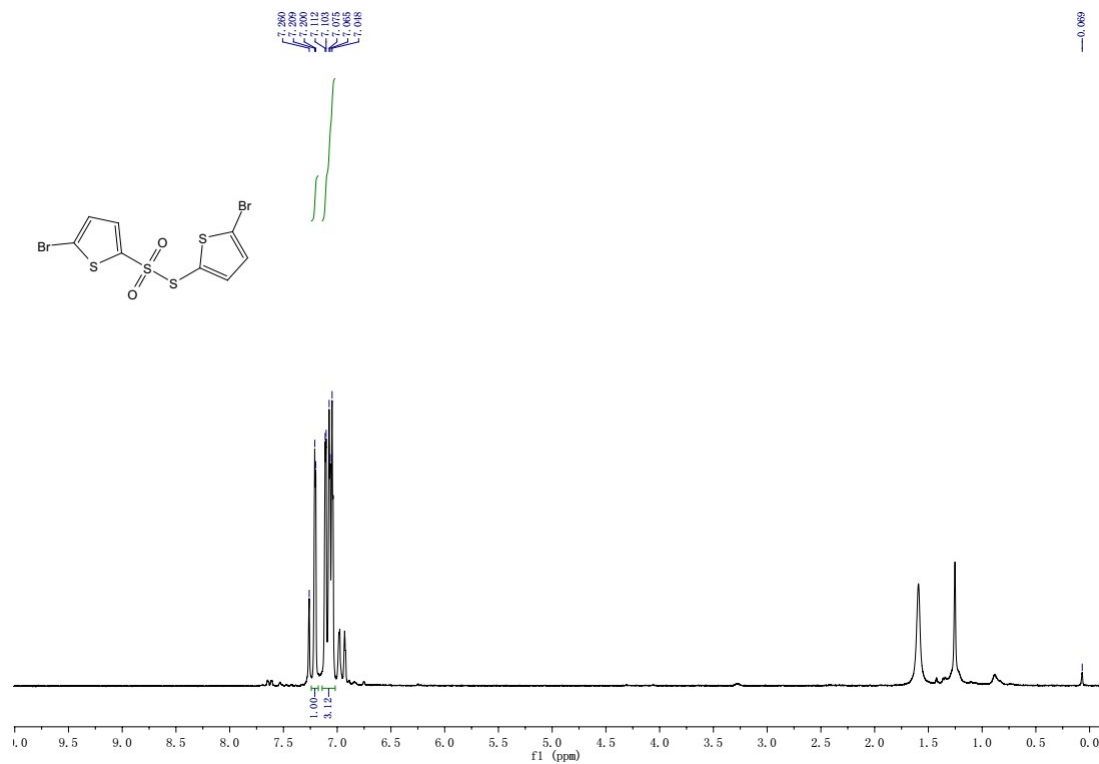
¹H NMR spectrum for compound **2n** (CDCl₃, 400 MHz)



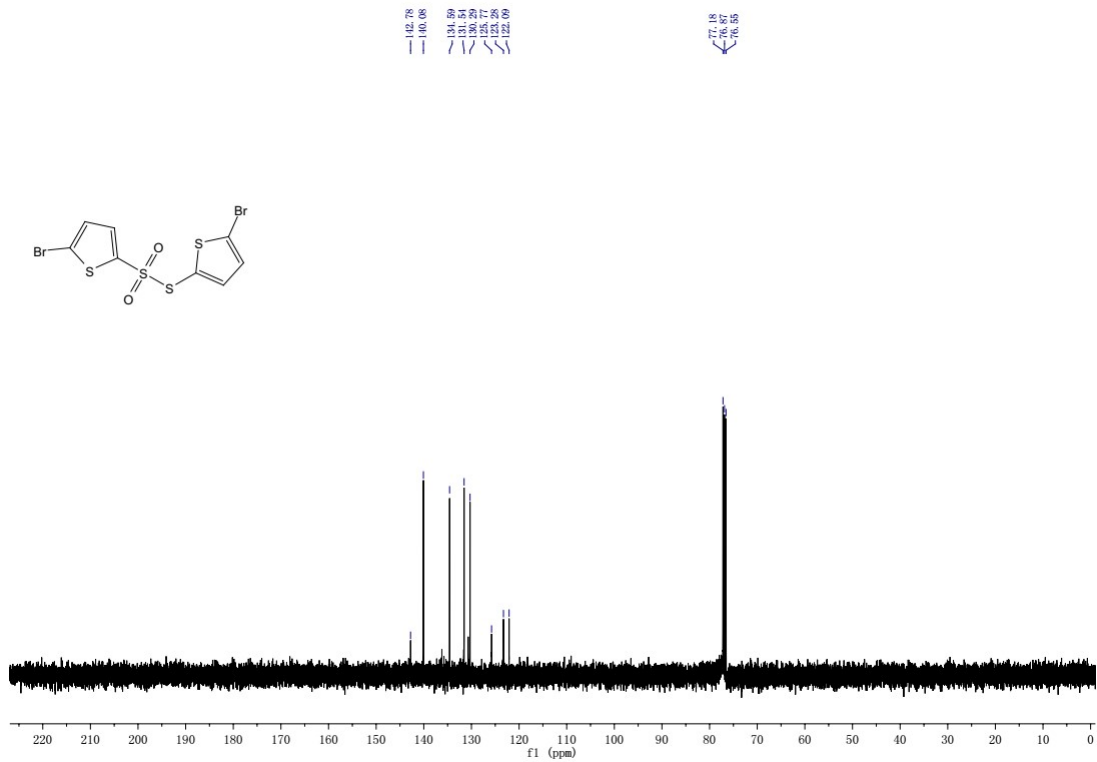
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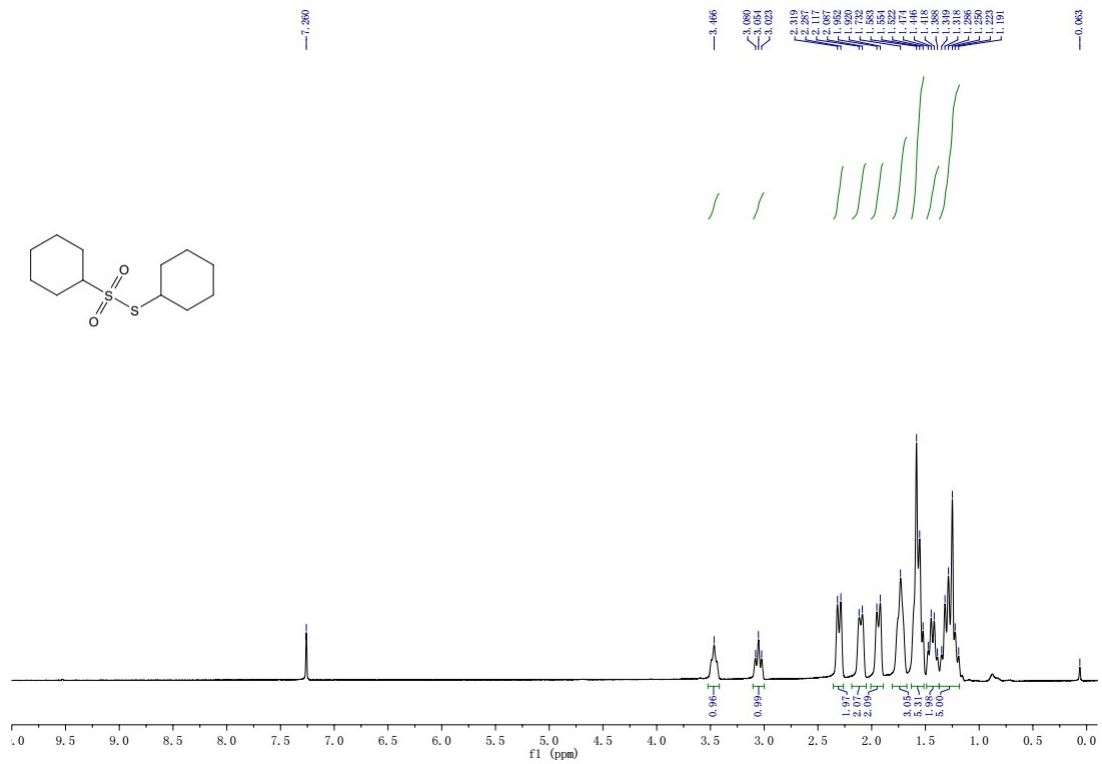
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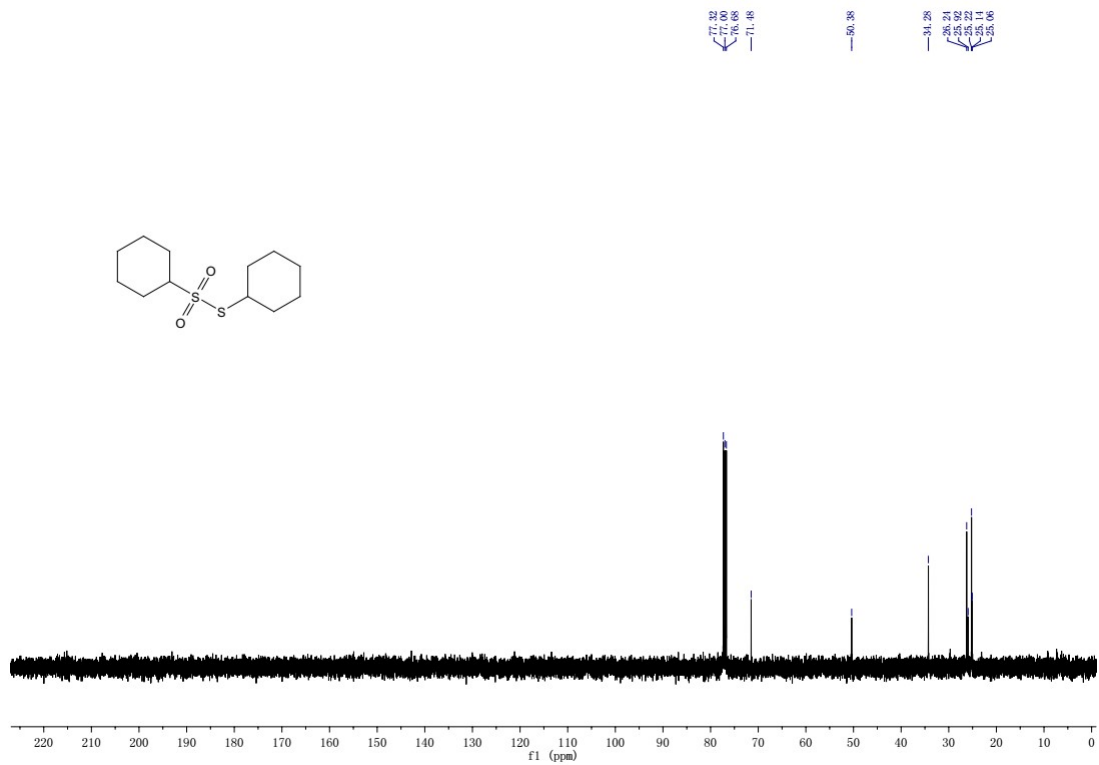
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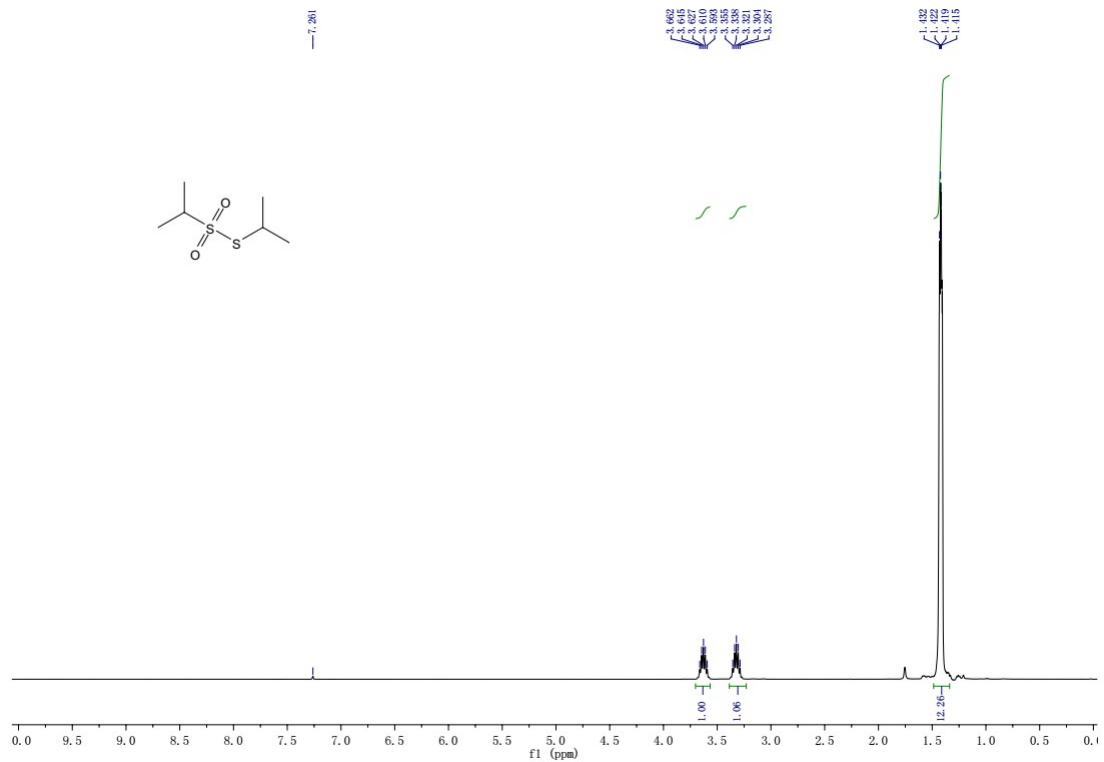
^1H NMR spectrum for compound **2p** (CDCl_3 , 400 MHz)



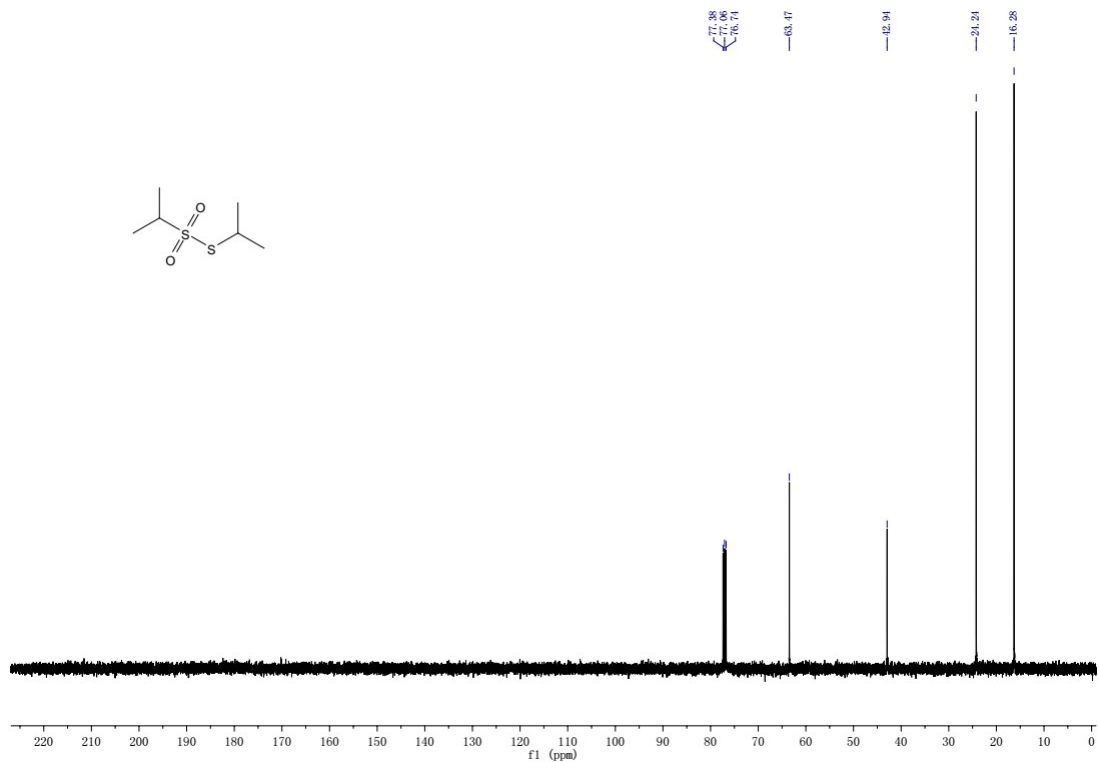
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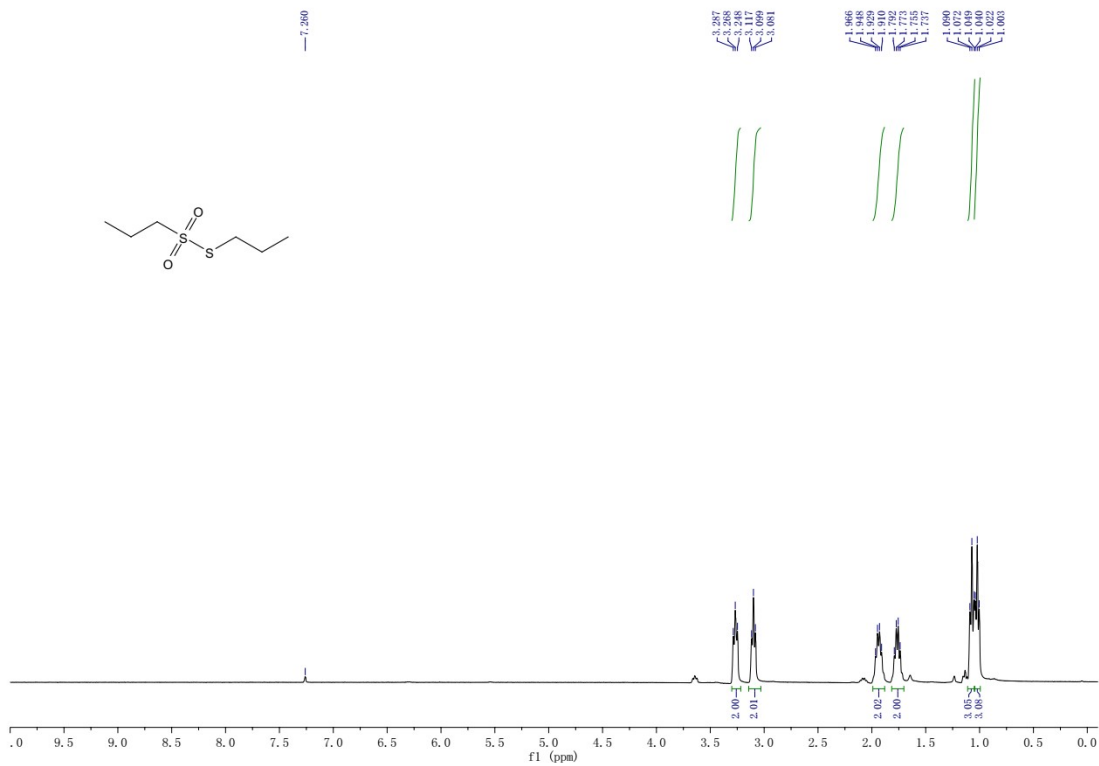
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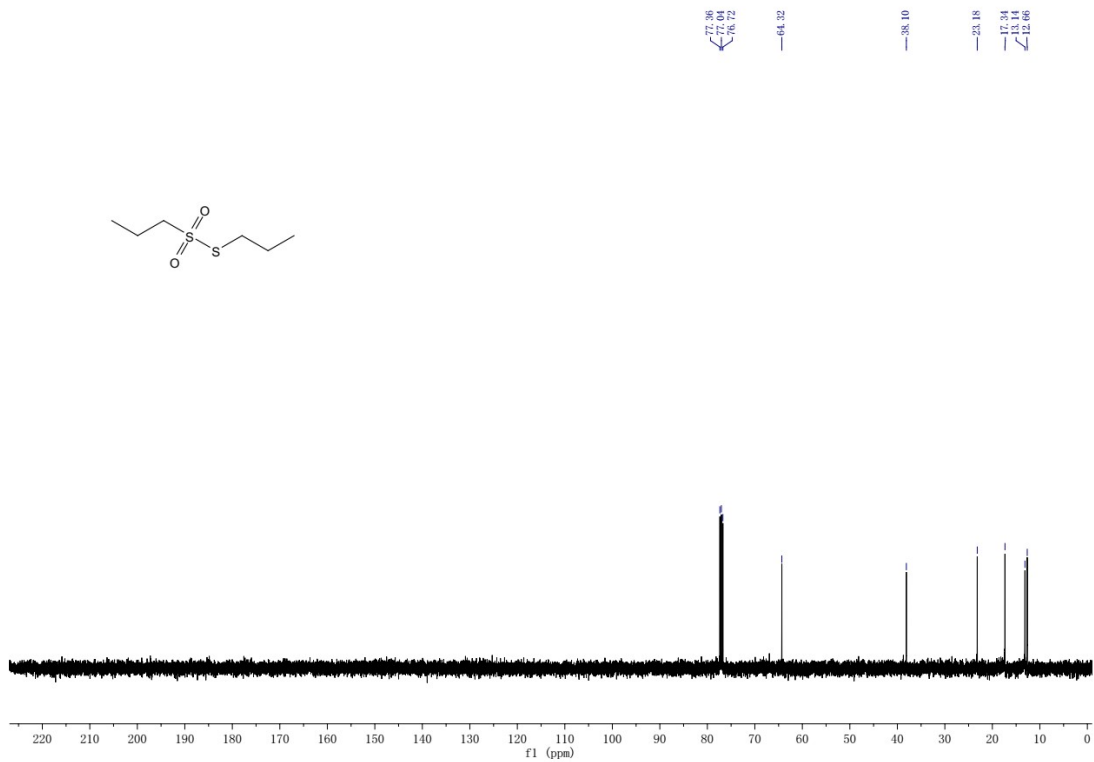
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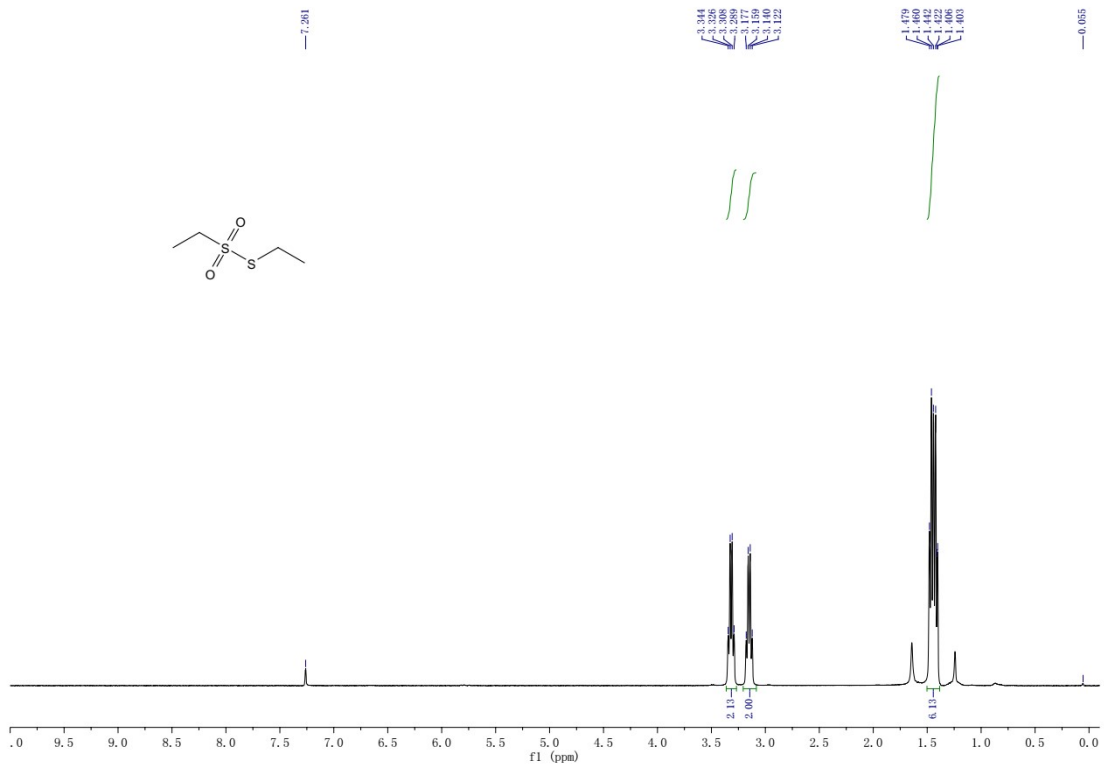
¹H NMR spectrum for compound **2r** (CDCl₃, 400 MHz)



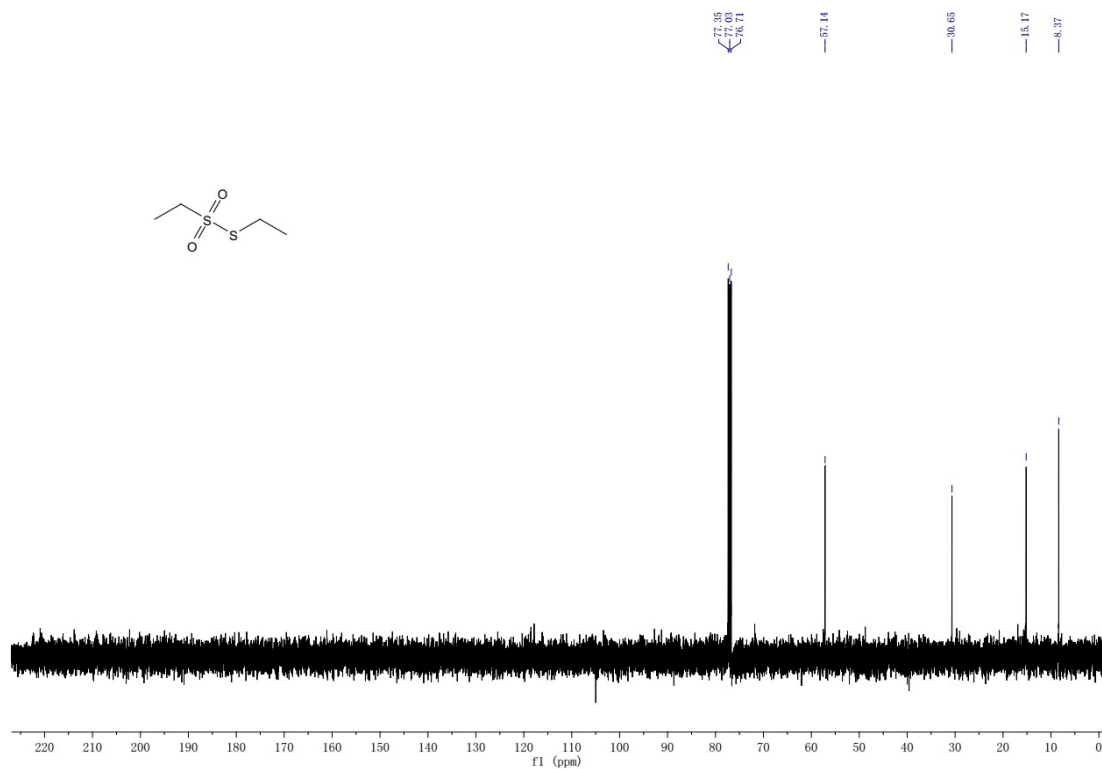
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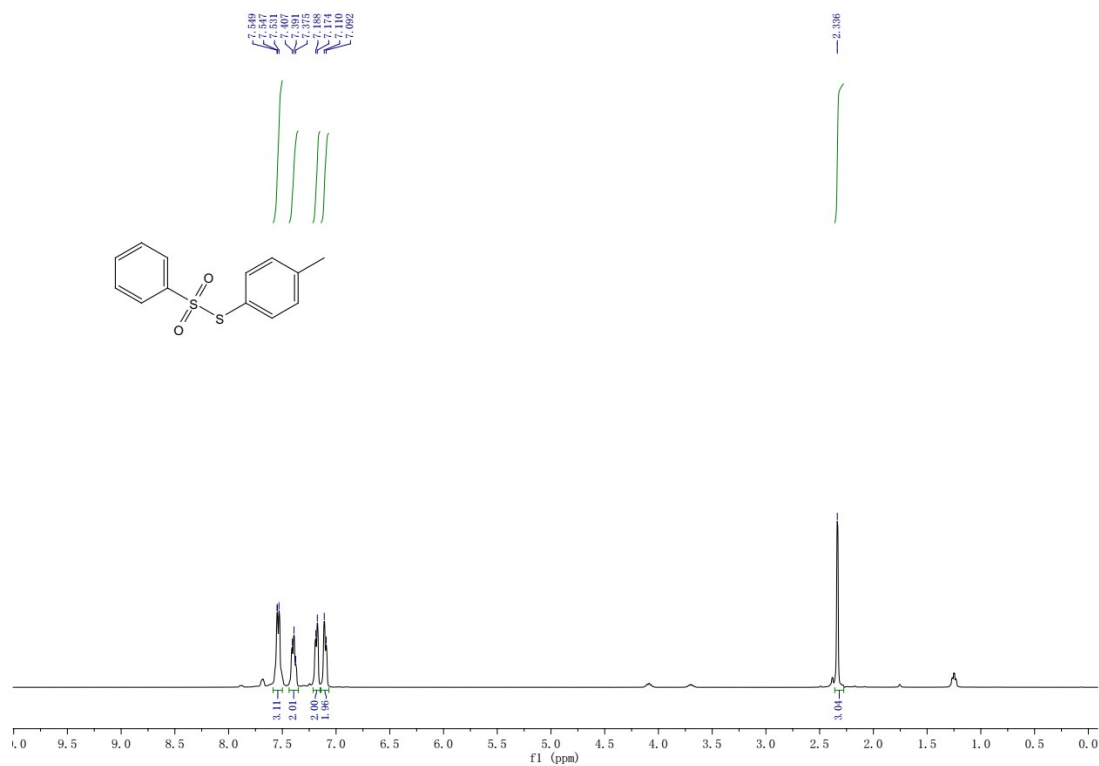
^1H NMR spectrum for compound 2s (CDCl₃, 400 MHz)



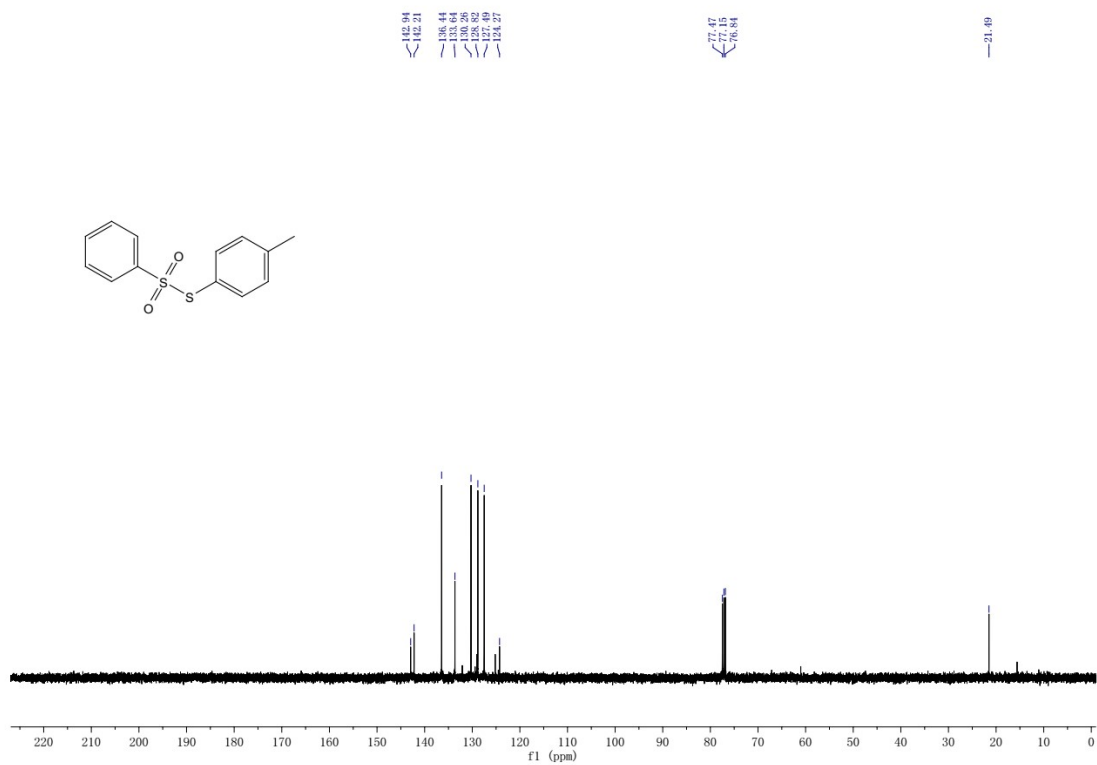
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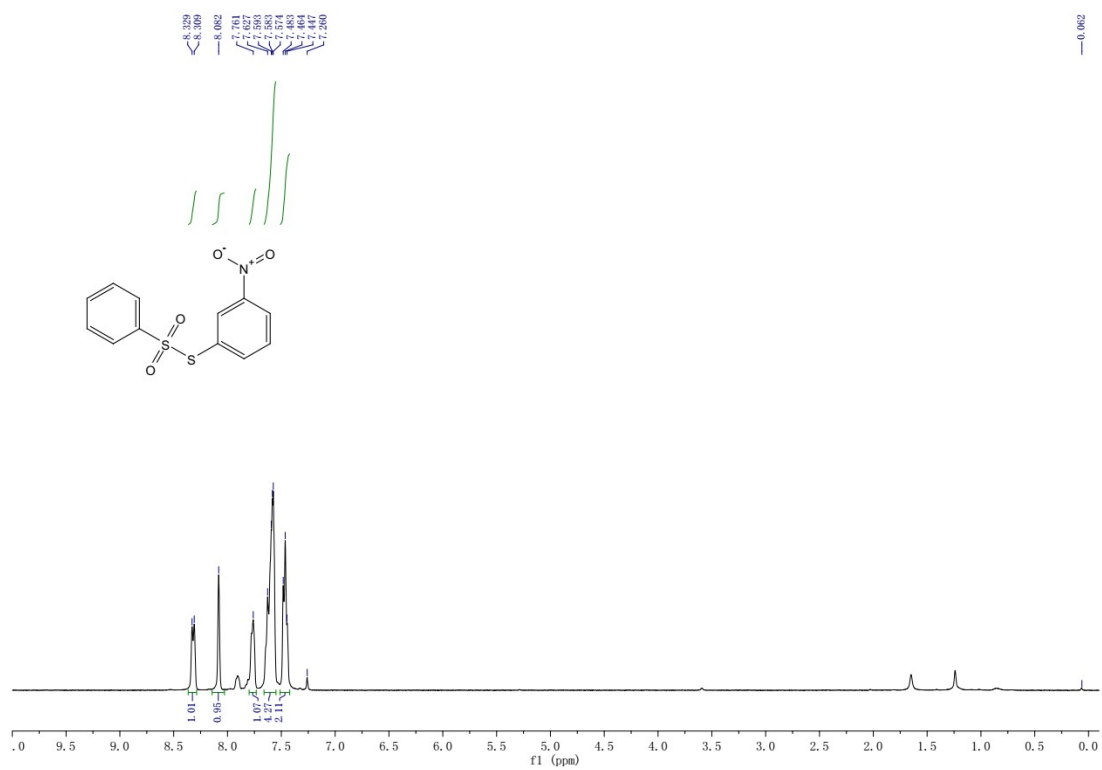
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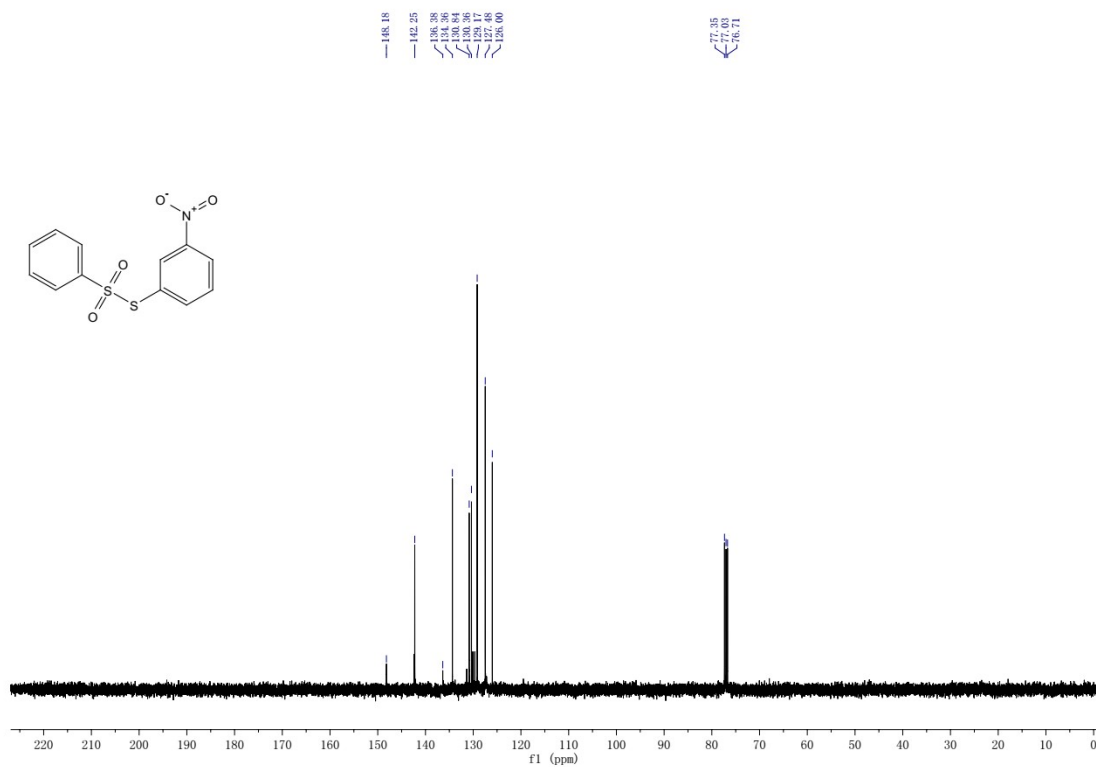
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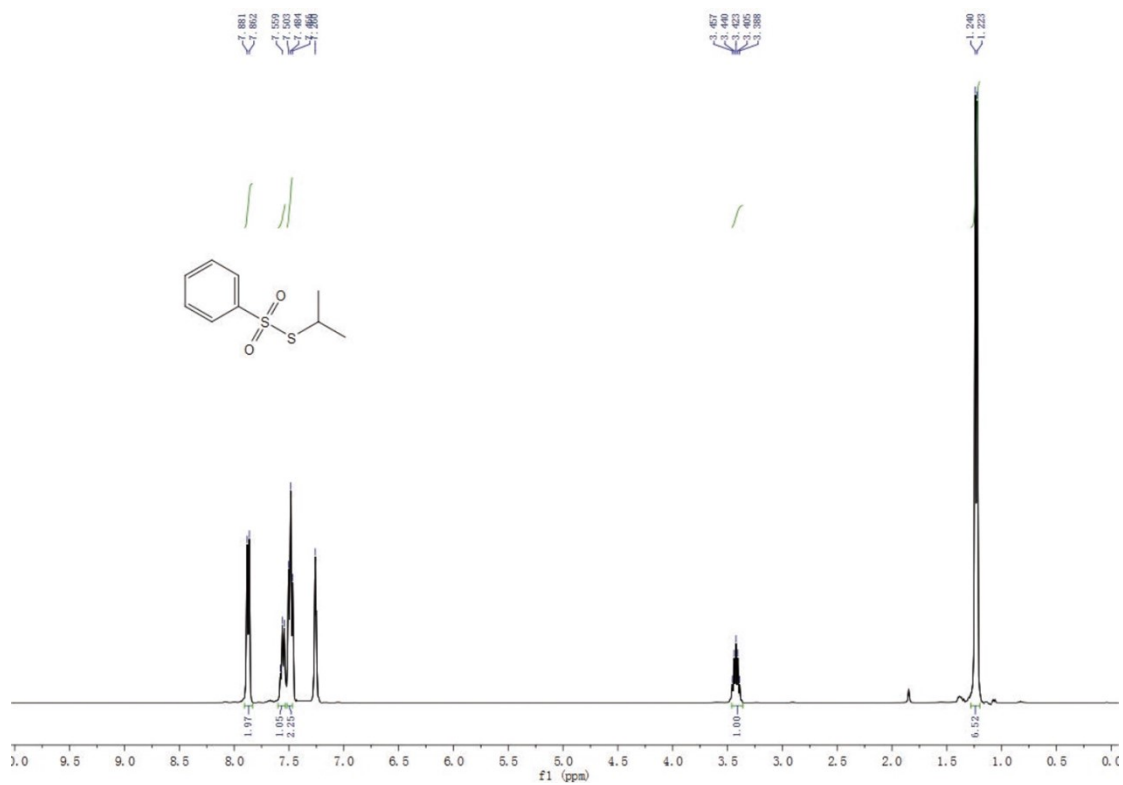
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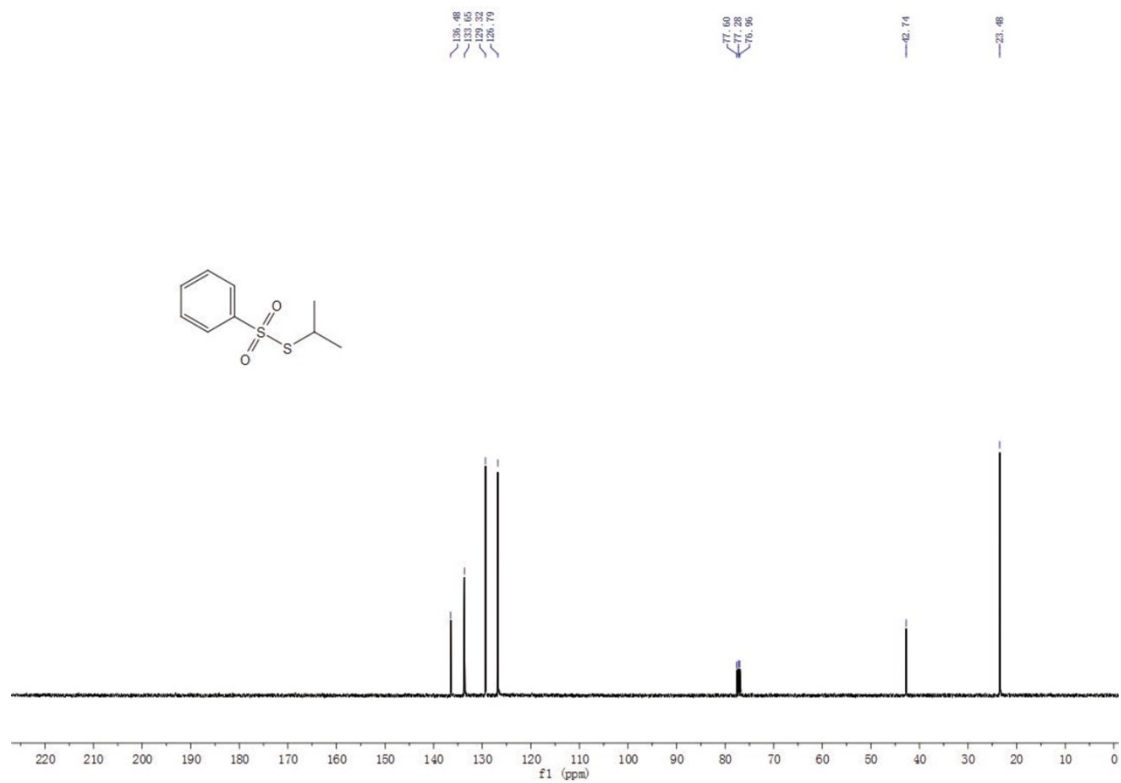
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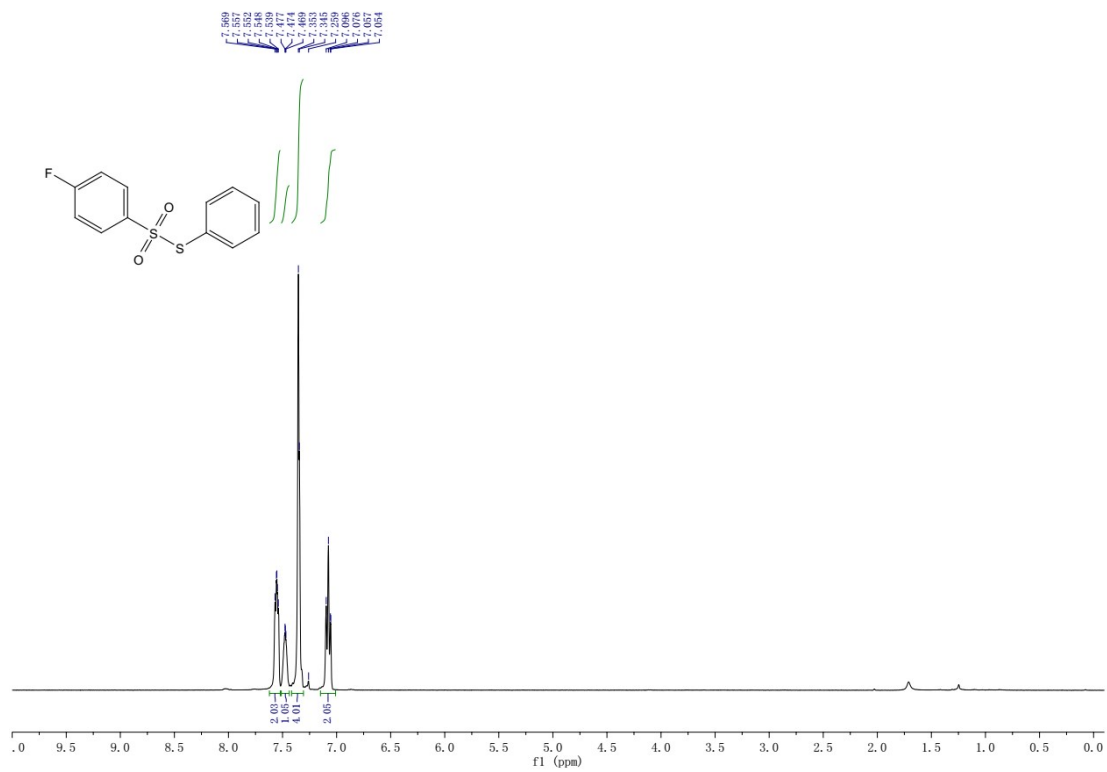
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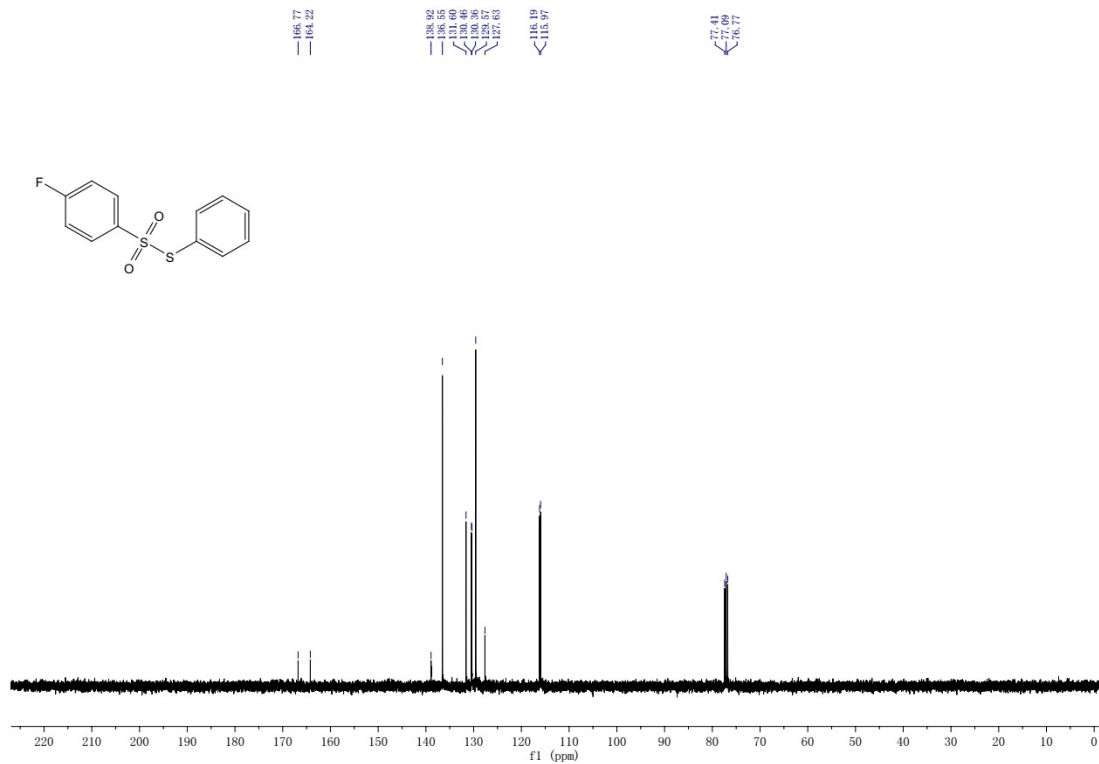
¹³C NMR spectrum for compound **2v** (CDCl₃, 101 MHz)



¹H NMR spectrum for compound **2w** (CDCl₃, 400 MHz)



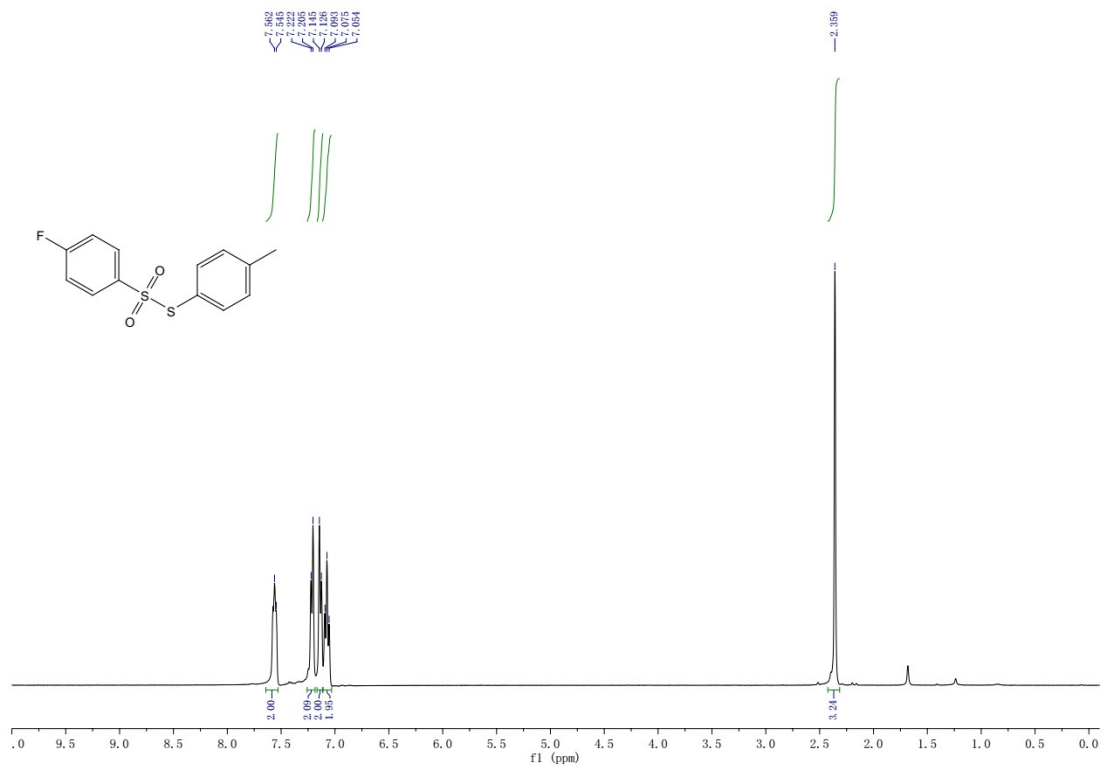
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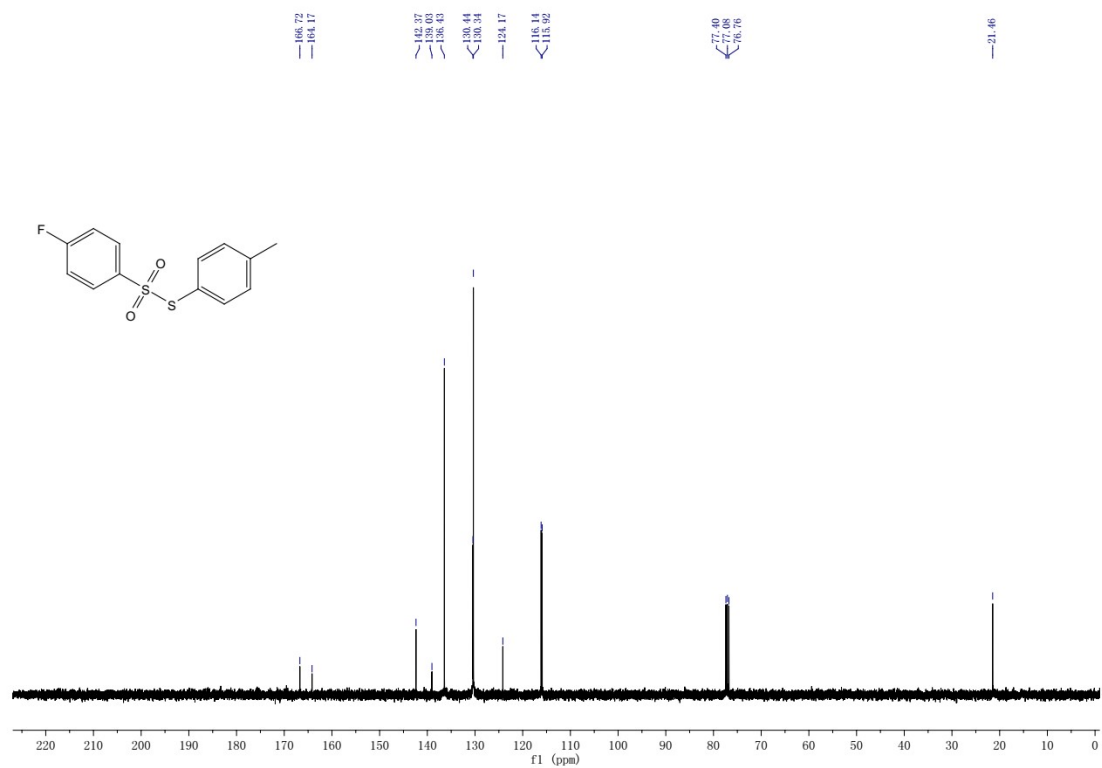
^{13}C NMR spectrum for compound **2w** (CDCl_3 , 376 MHz)



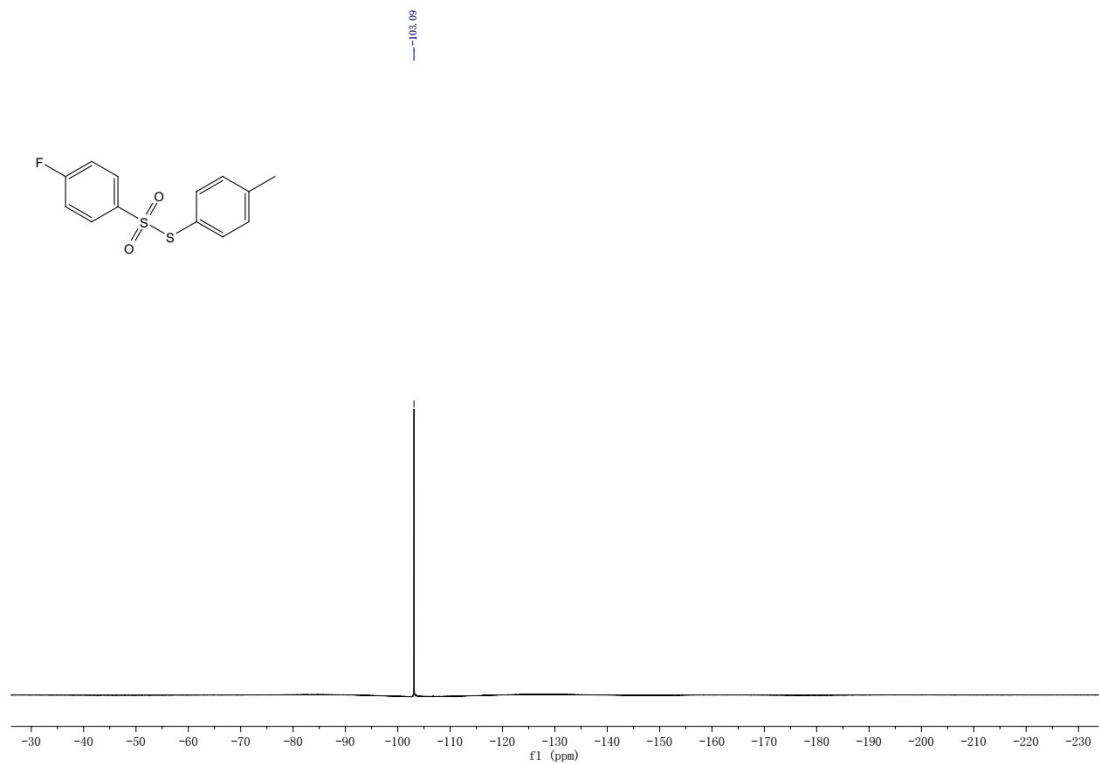
^{19}F NMR spectrum for compound **2x** (CDCl_3 , 400 MHz)



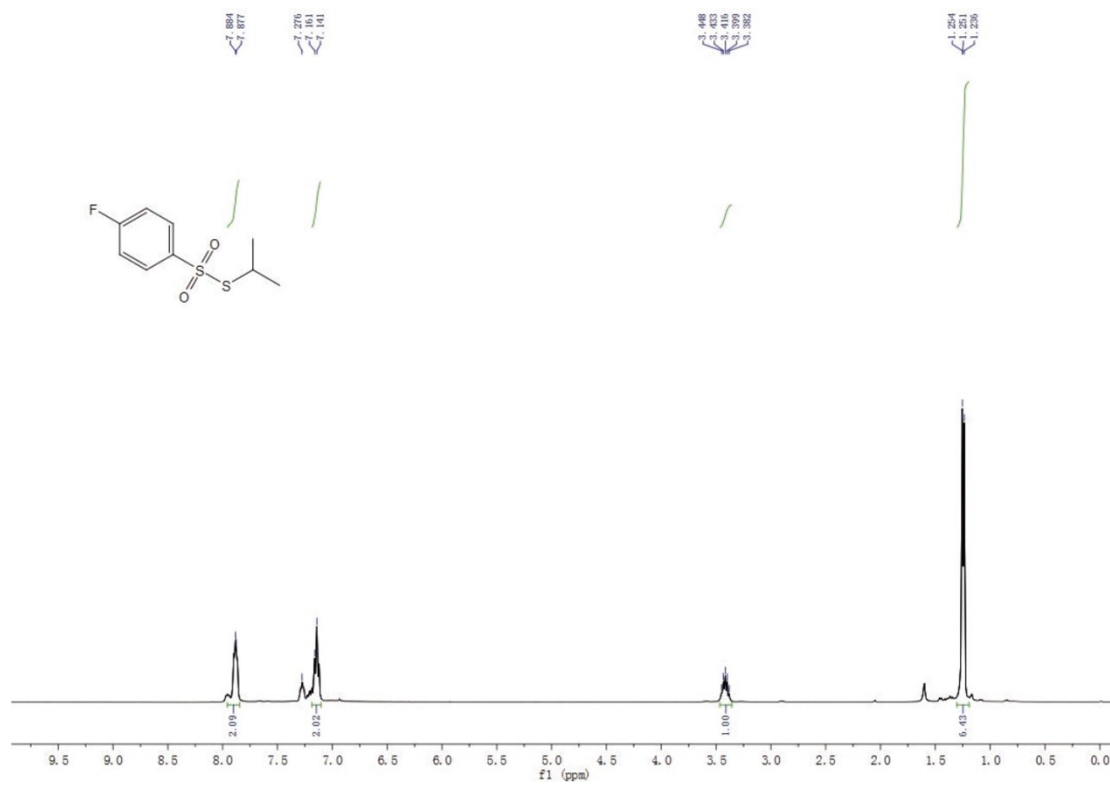
¹³C NMR spectrum for compound **2x** (CDCl₃, 101 MHz)



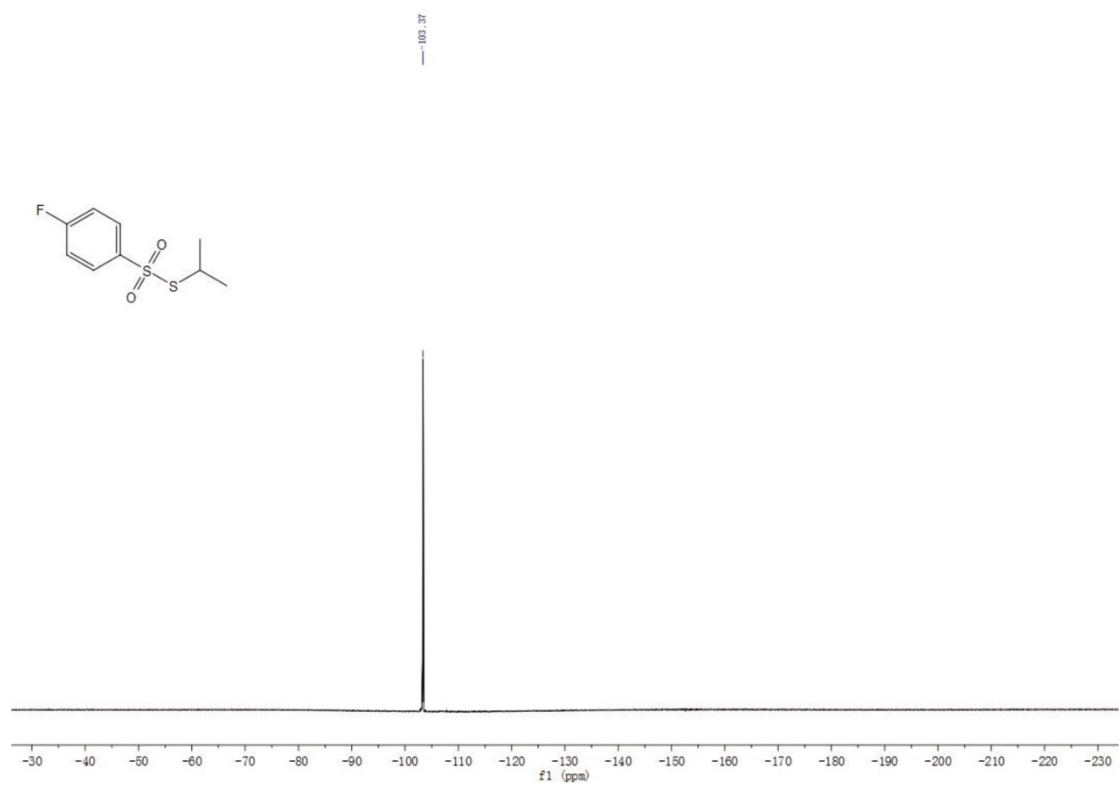
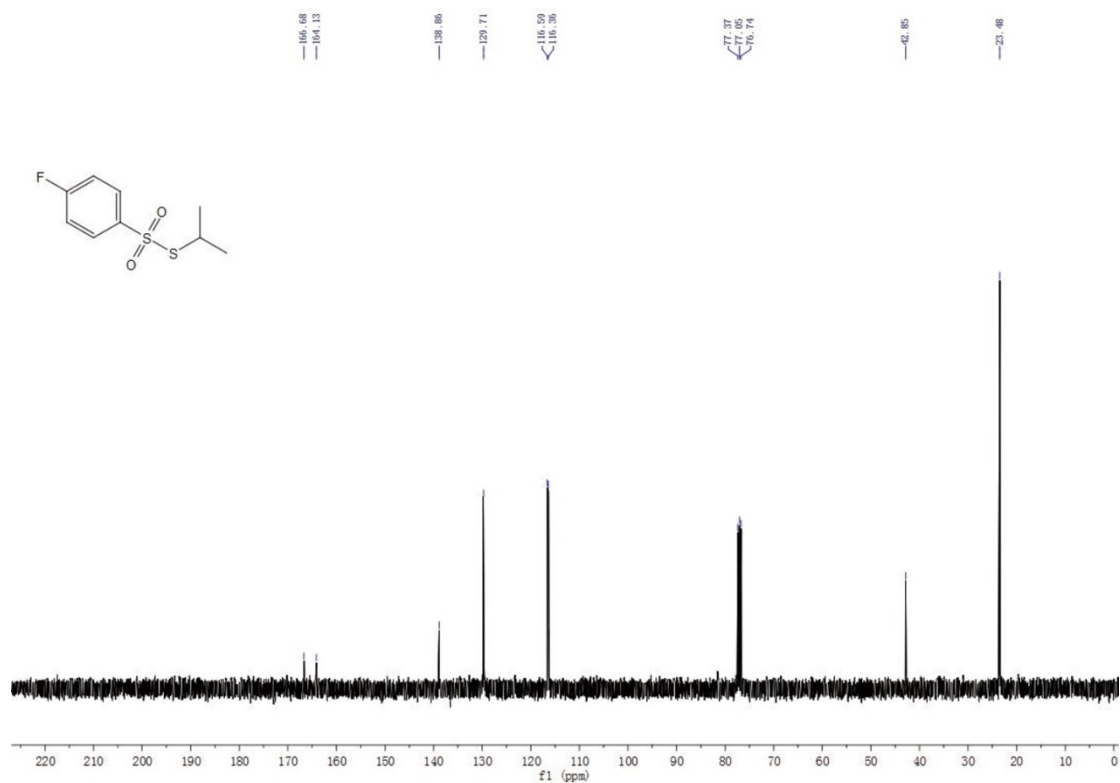
¹⁹F NMR spectrum for compound **2x** (CDCl₃, 376 MHz)

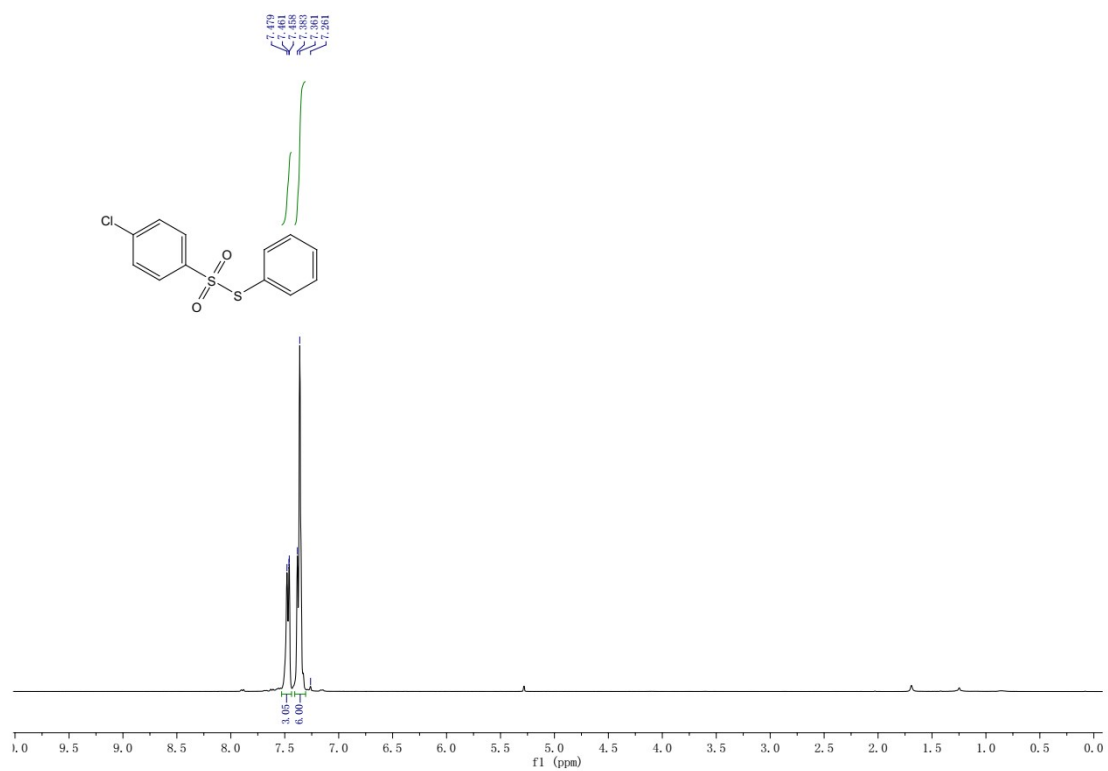


¹H NMR spectrum for compound **2y** (CDCl₃, 400 MHz)

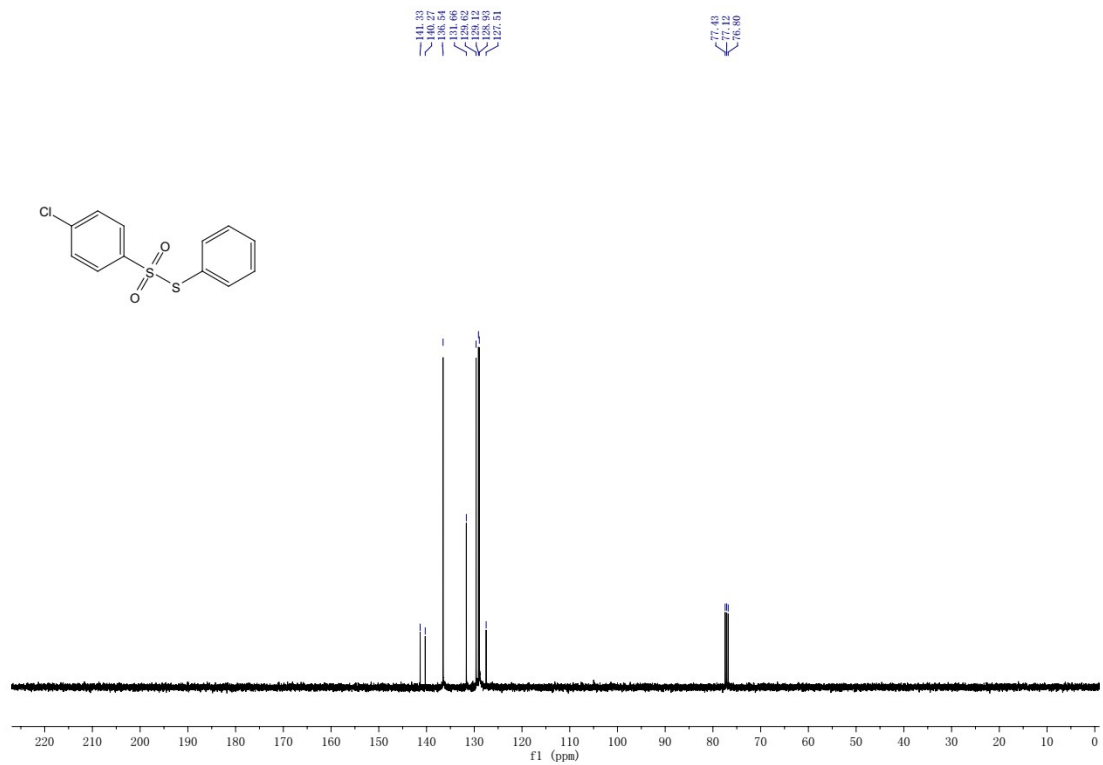


¹³C NMR spectrum for compound **2y** (CDCl₃, 101 MHz)

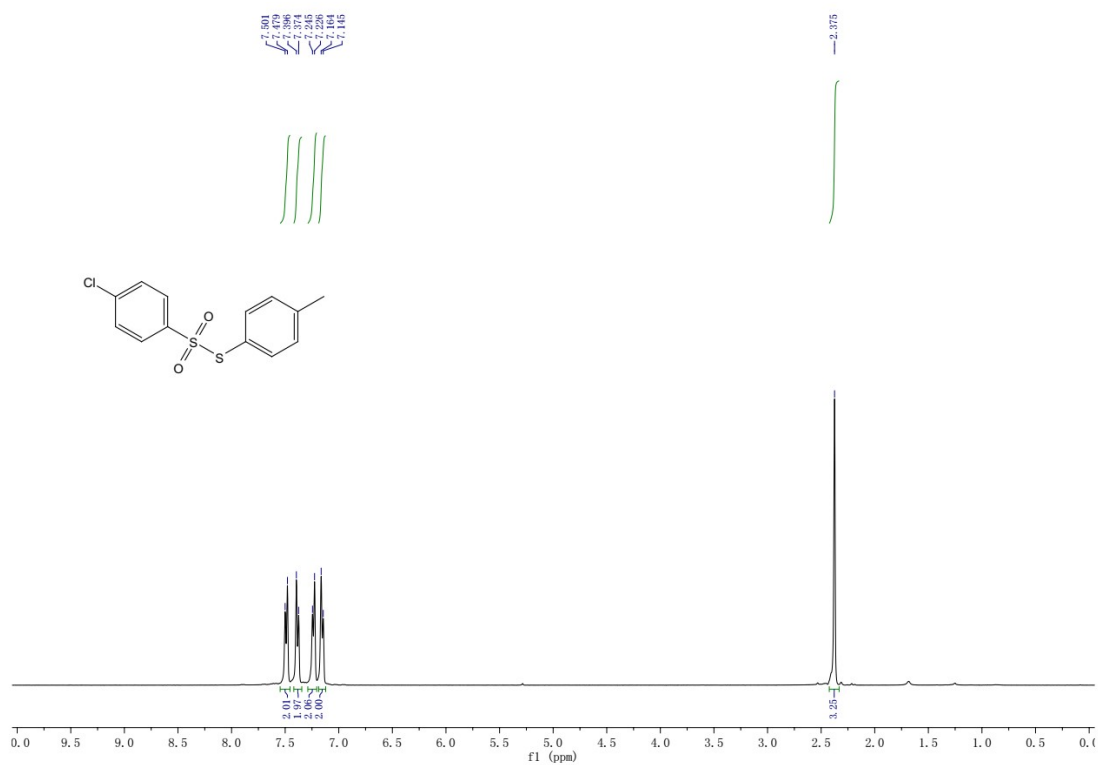




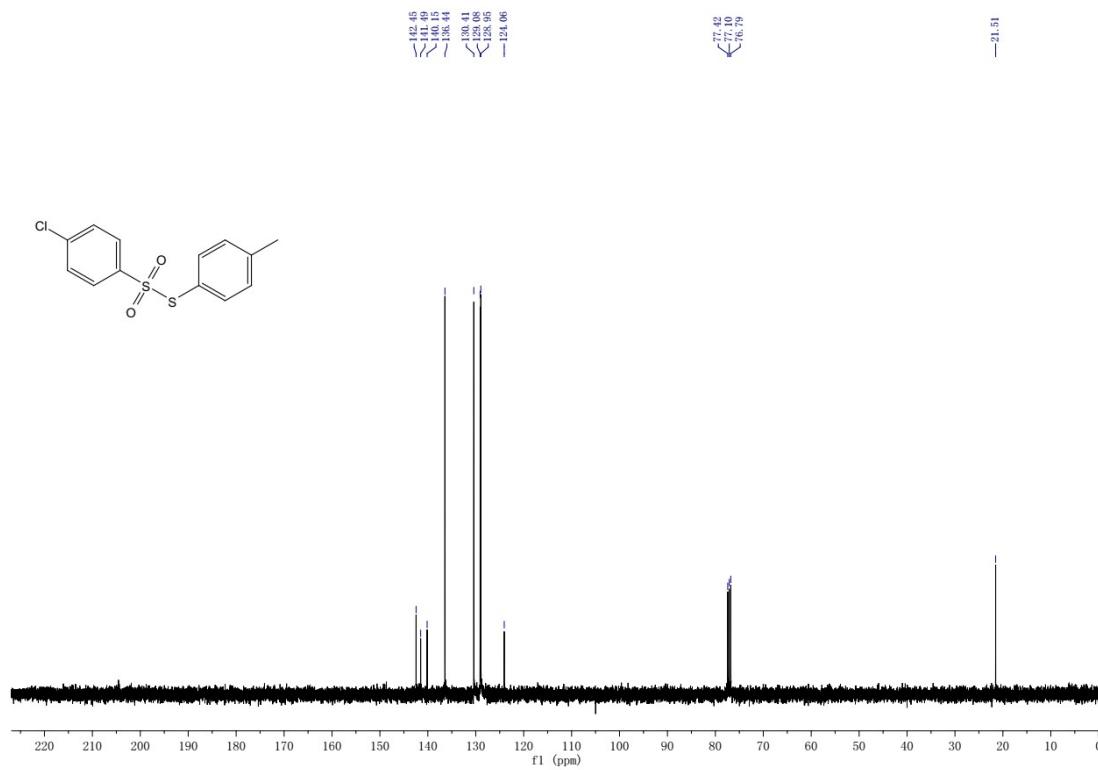
^{13}C NMR spectrum for compound **2x** (CDCl_3 , 101 MHz)



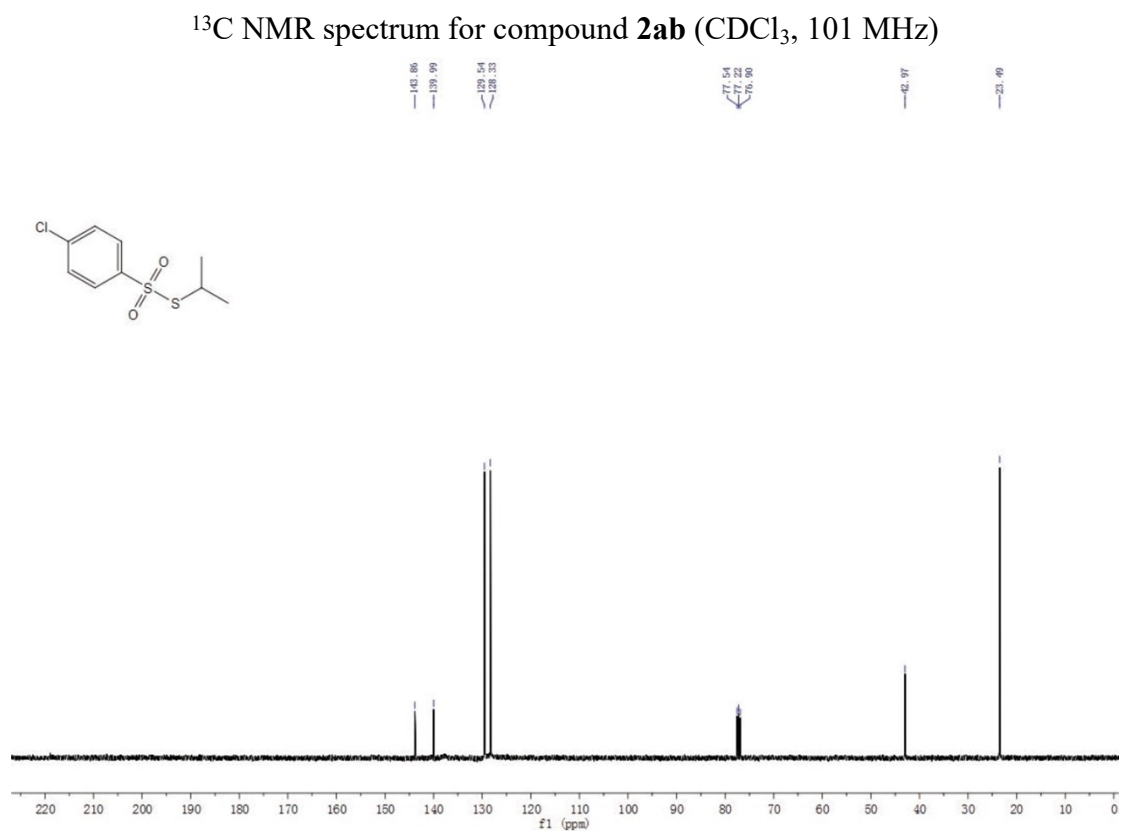
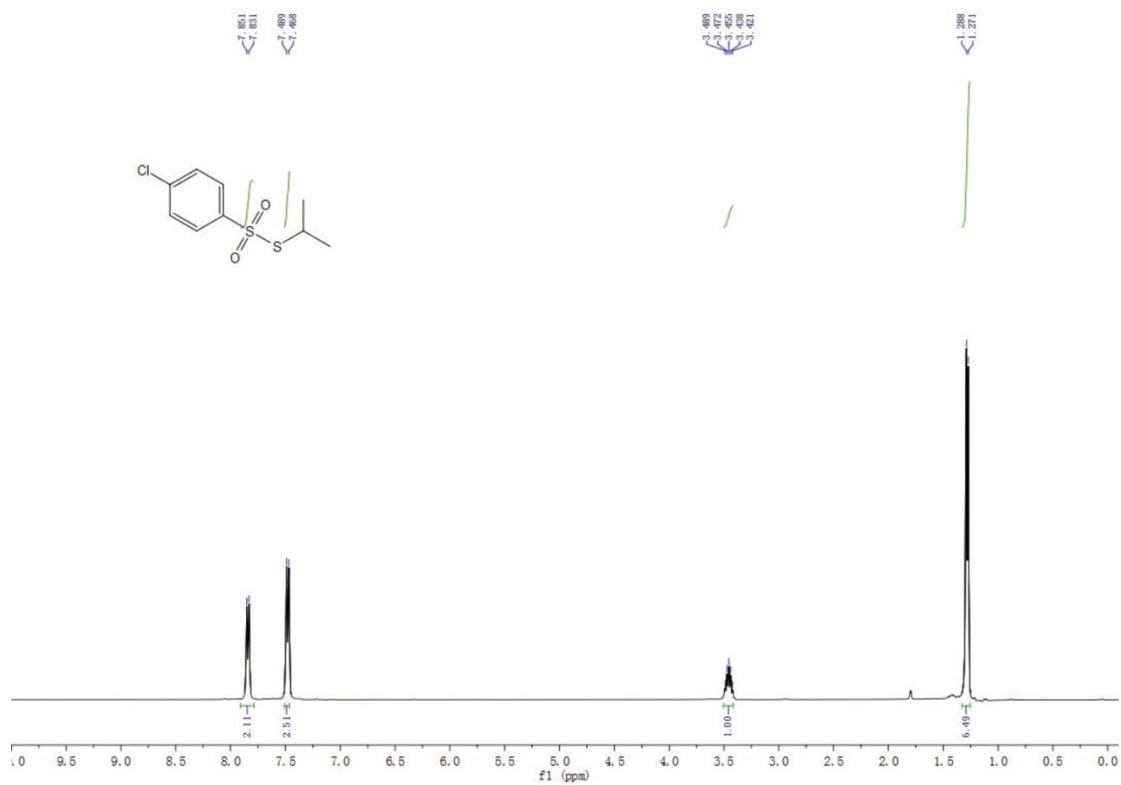
^1H NMR spectrum for compound **2aa** (CDCl_3 , 400 MHz)



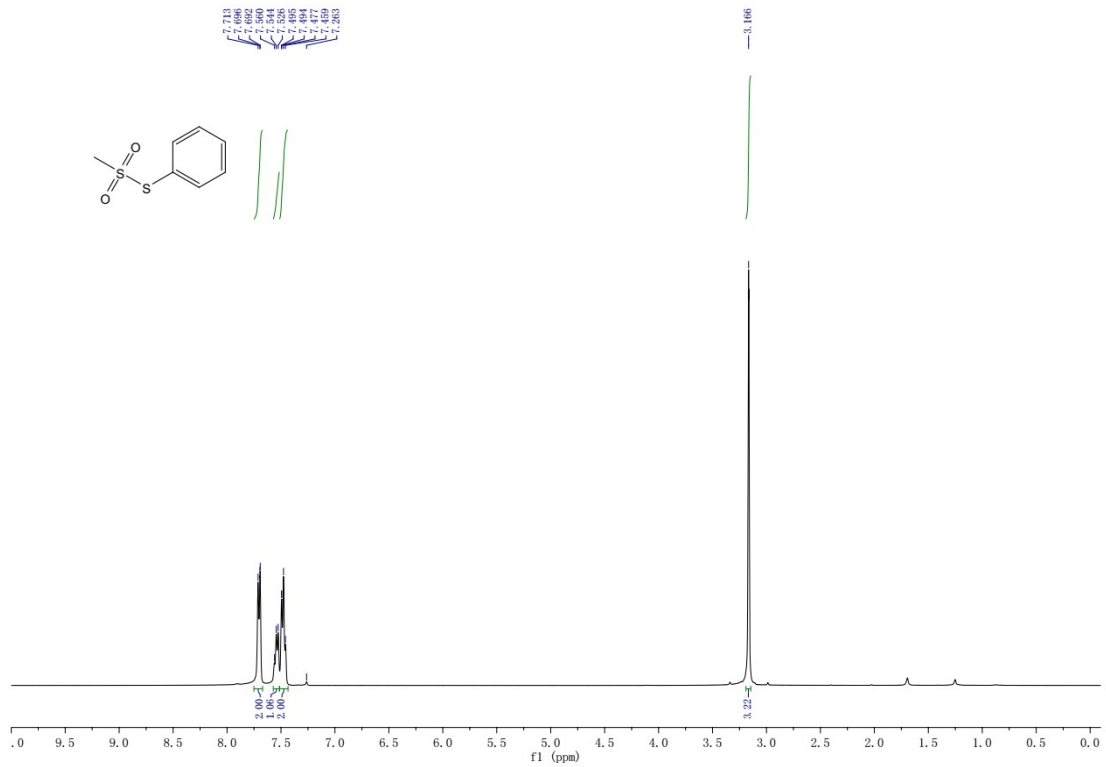
¹³C NMR spectrum for compound **2aa** (CDCl₃, 101 MHz)



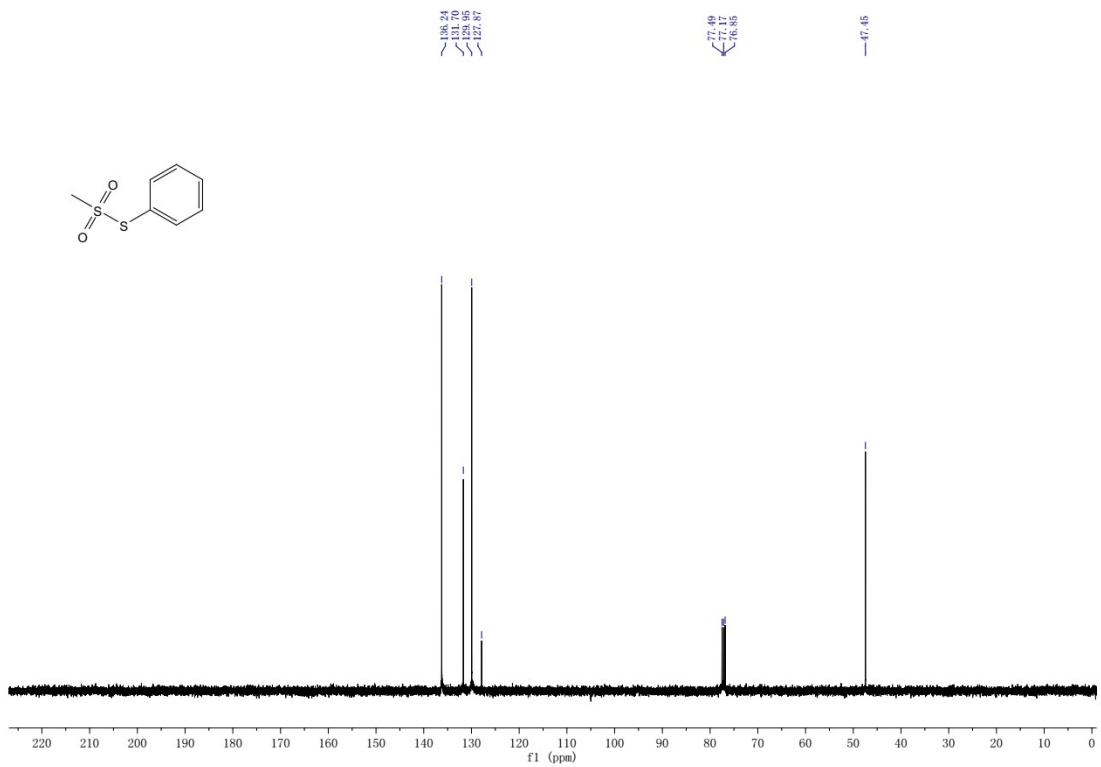
¹H NMR spectrum for compound **2ab** (CDCl₃, 400 MHz)



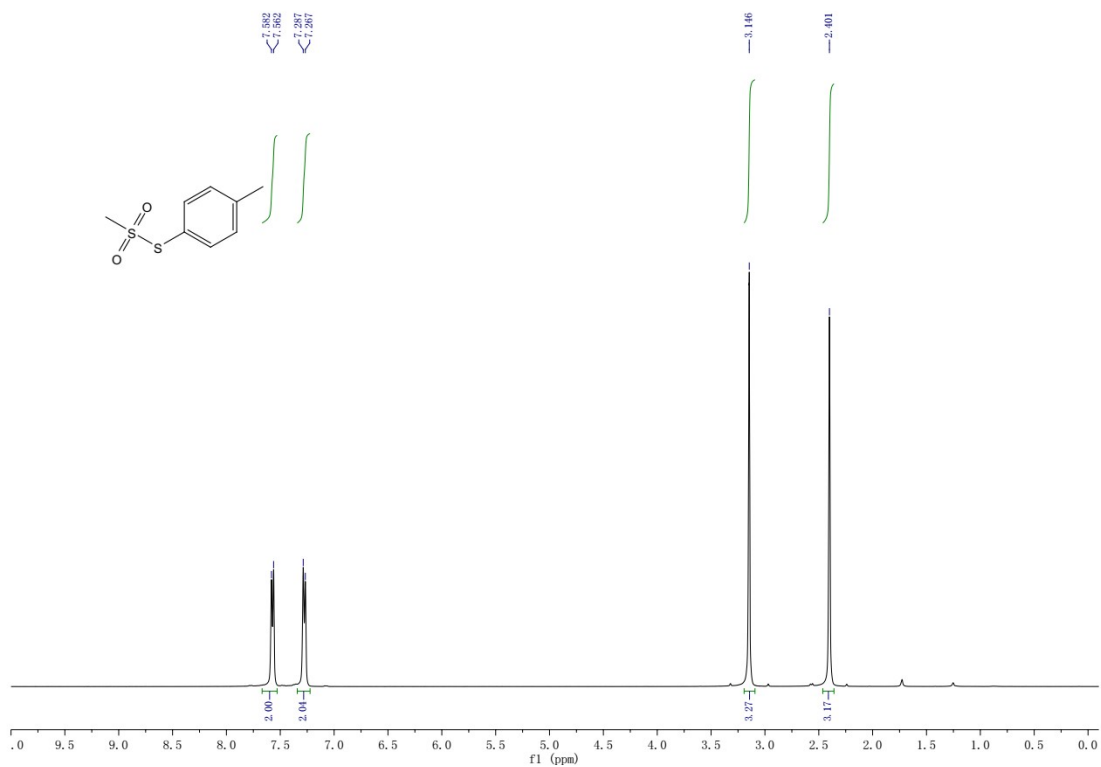
¹H NMR spectrum for compound **2ac** (CDCl₃, 400 MHz)



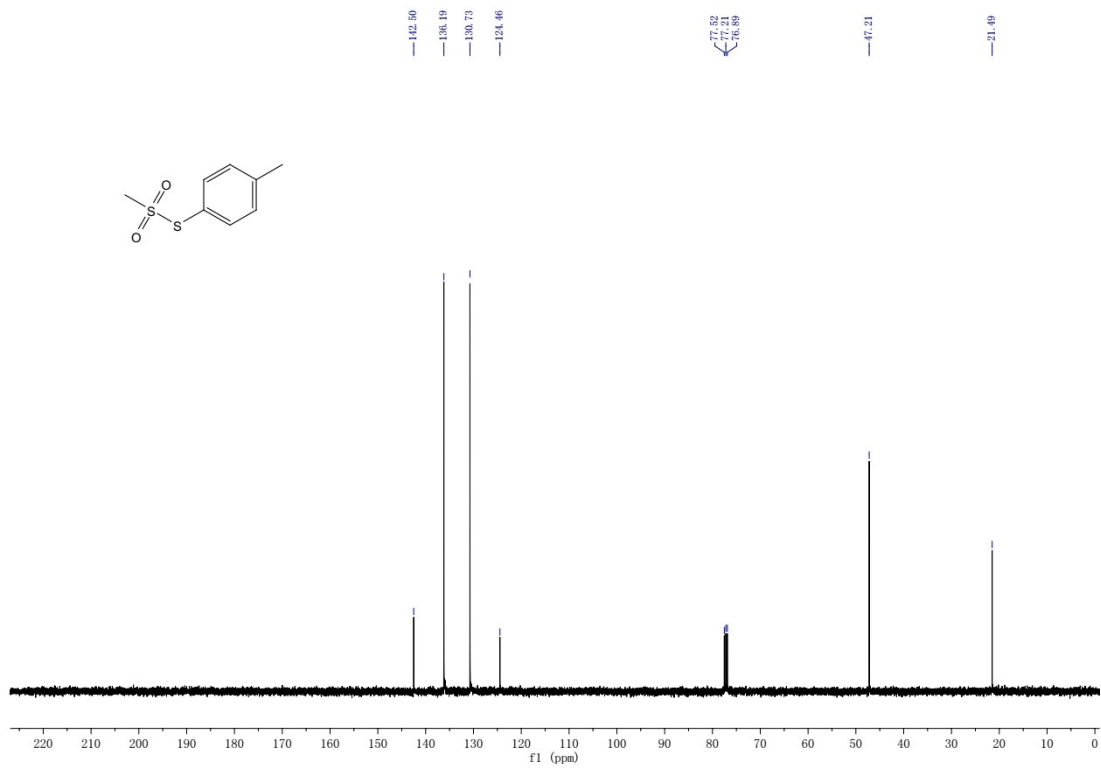
^{13}C NMR spectrum for compound **2ac** (CDCl_3 , 101 MHz)



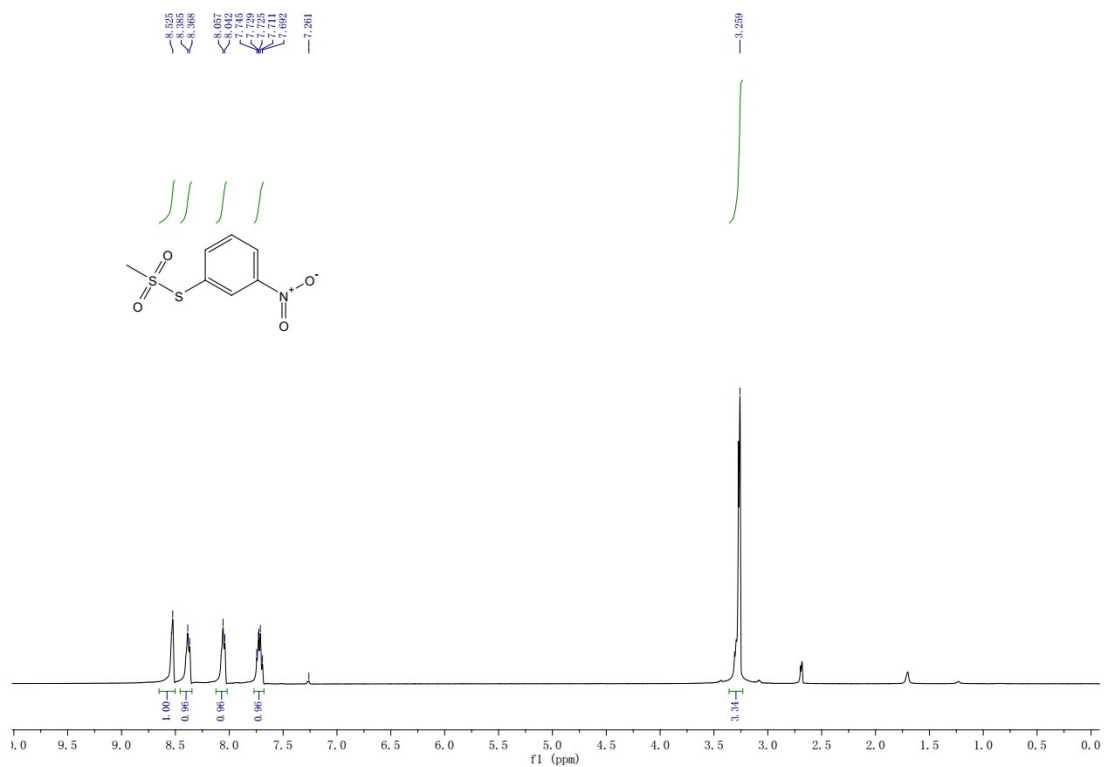
^1H NMR spectrum for compound **2ad** (CDCl_3 , 400 MHz)



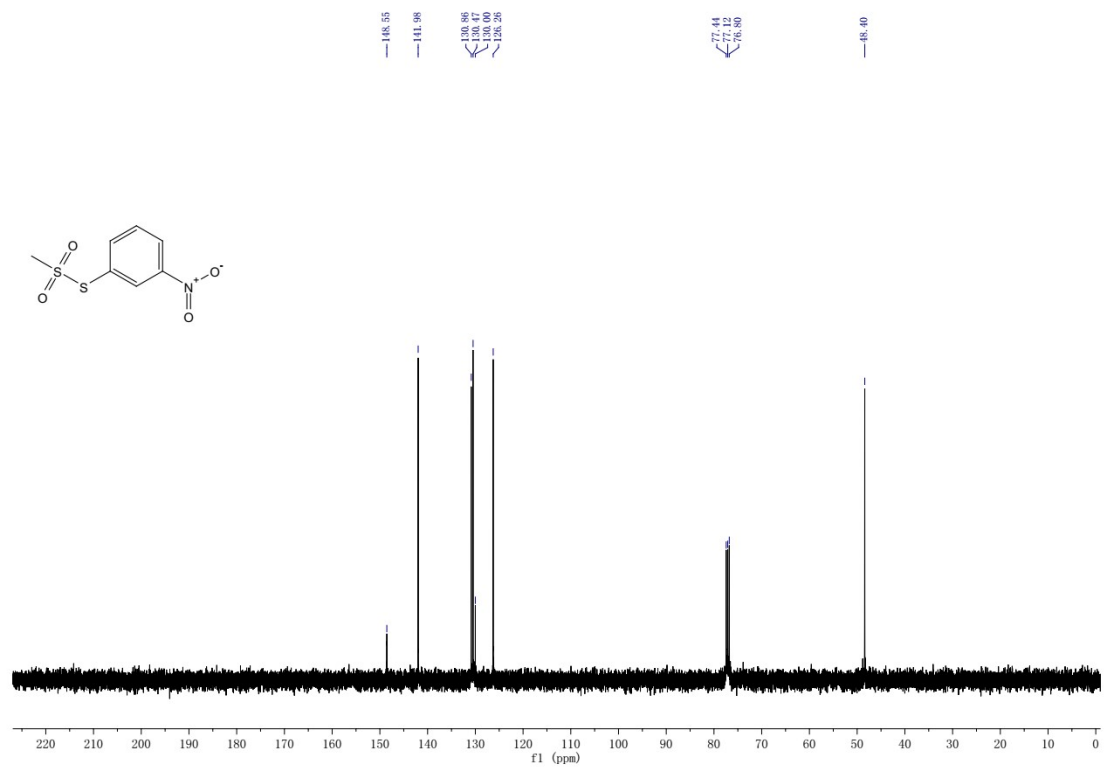
¹³C NMR spectrum for compound **2ad** (CDCl₃, 101 MHz)



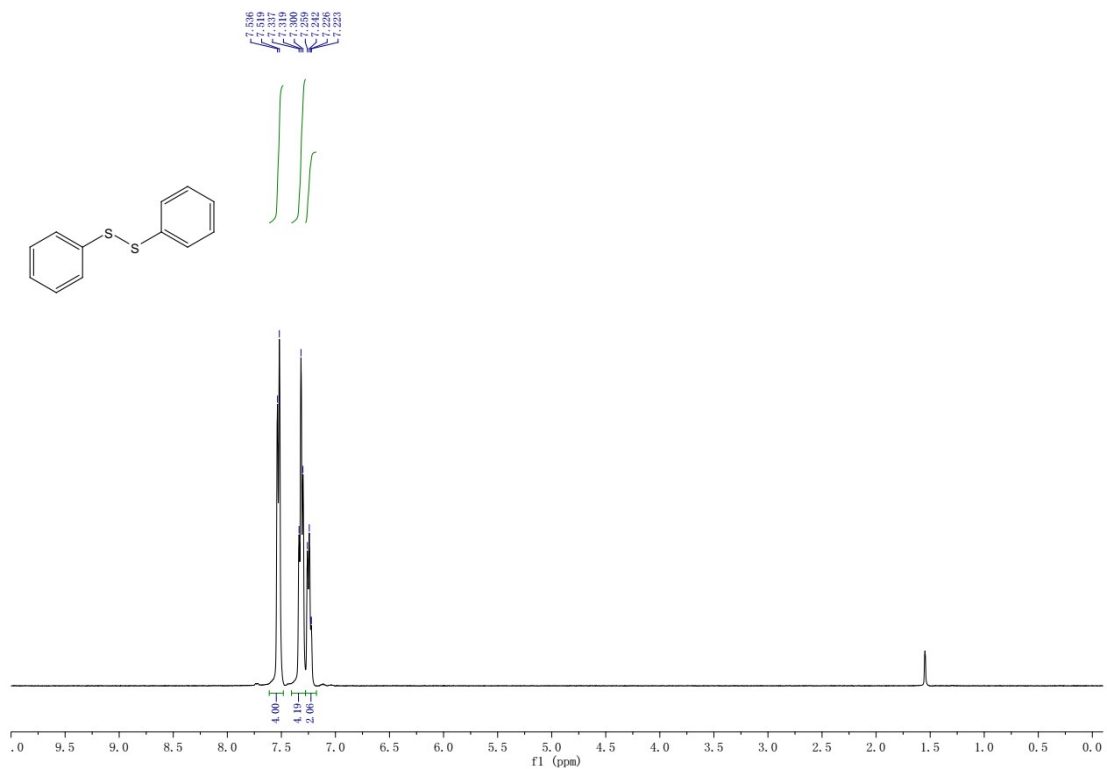
¹H NMR spectrum for compound **2ae** (CDCl₃, 400 MHz)



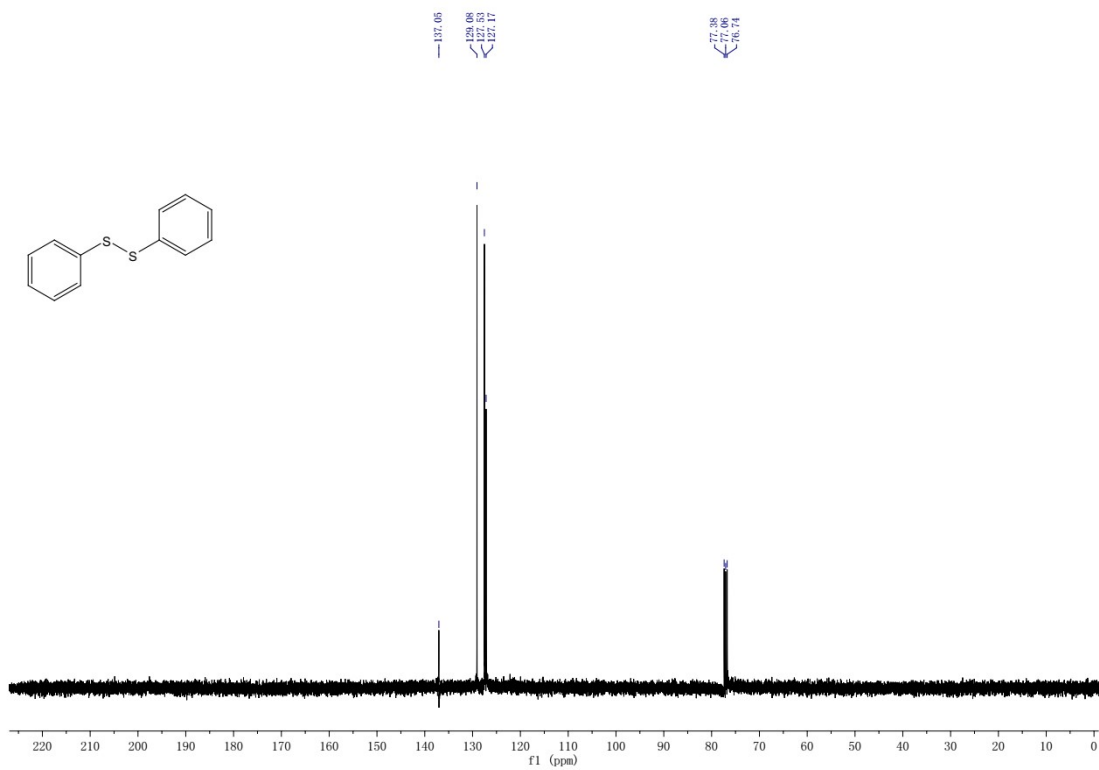
¹³C NMR spectrum for compound **2ae** (CDCl₃, 101 MHz)



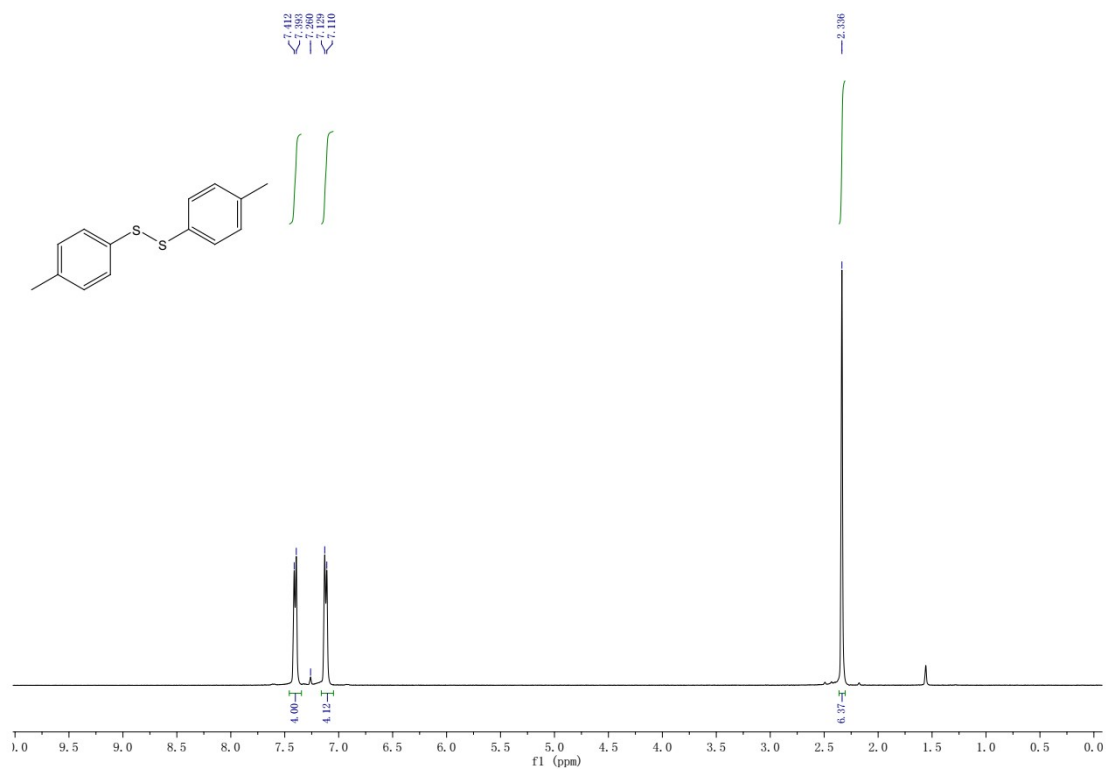
¹H NMR spectrum for compound **3a** (CDCl₃, 400 MHz)



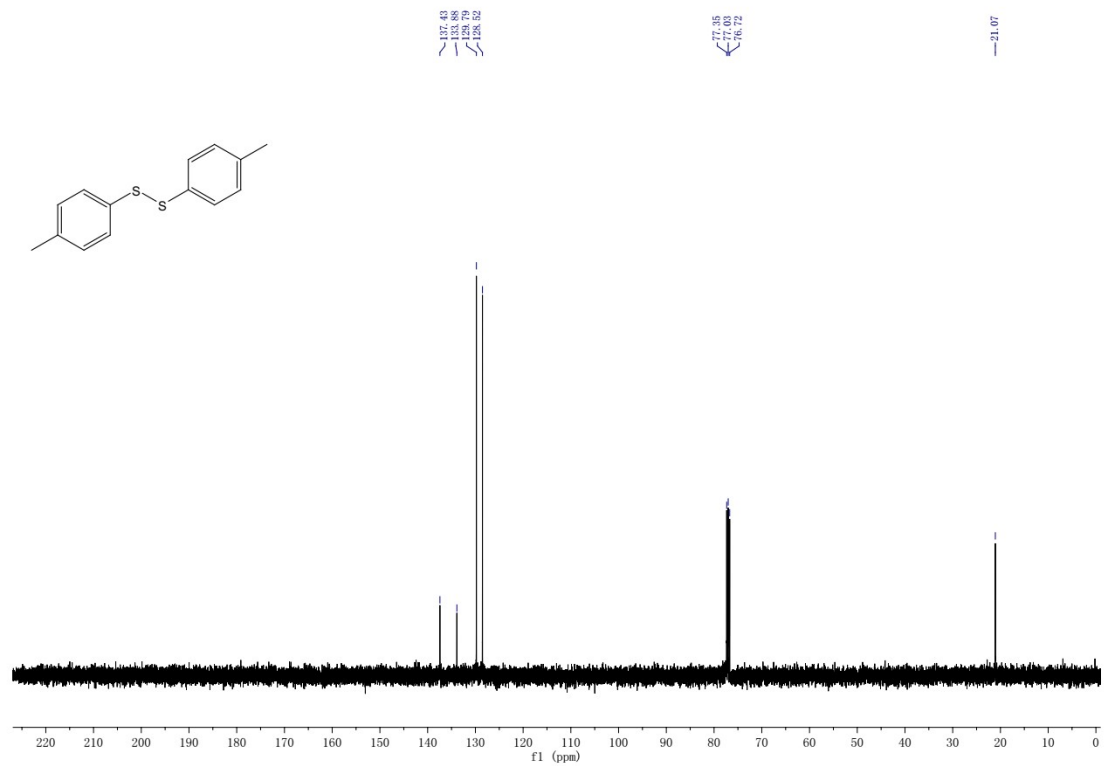
^{13}C NMR spectrum for compound **3a** (CDCl_3 , 101 MHz)



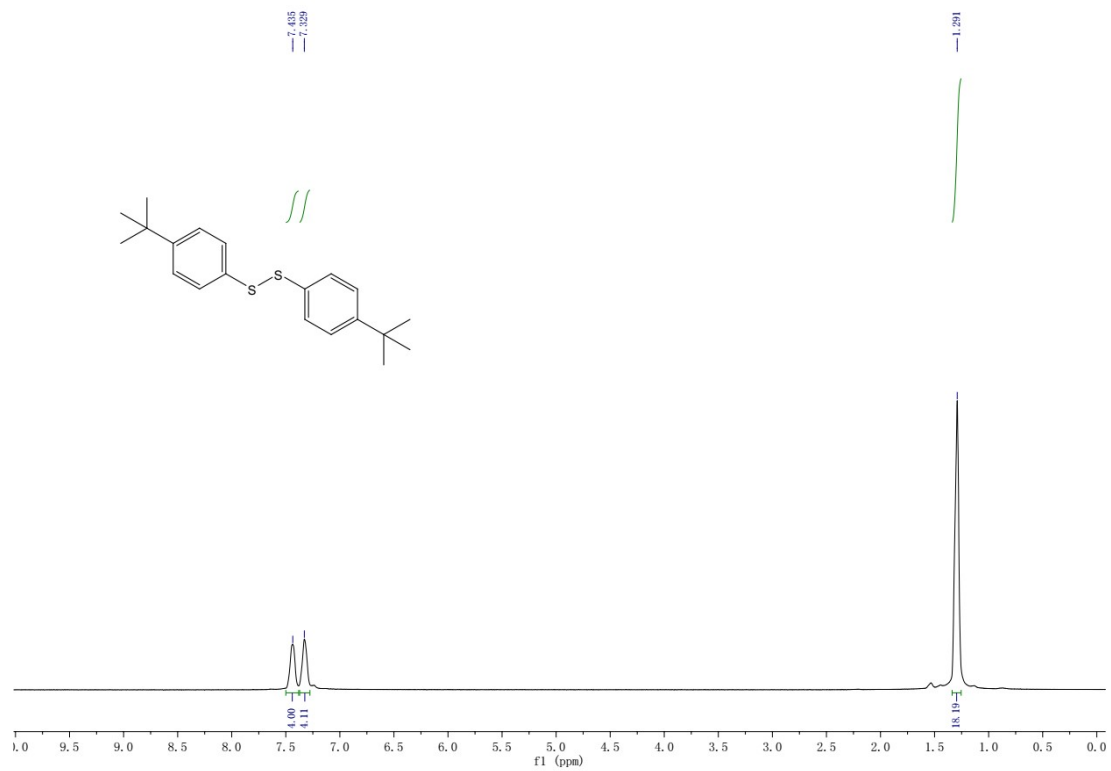
^1H NMR spectrum for compound **3b** (CDCl_3 , 400 MHz)



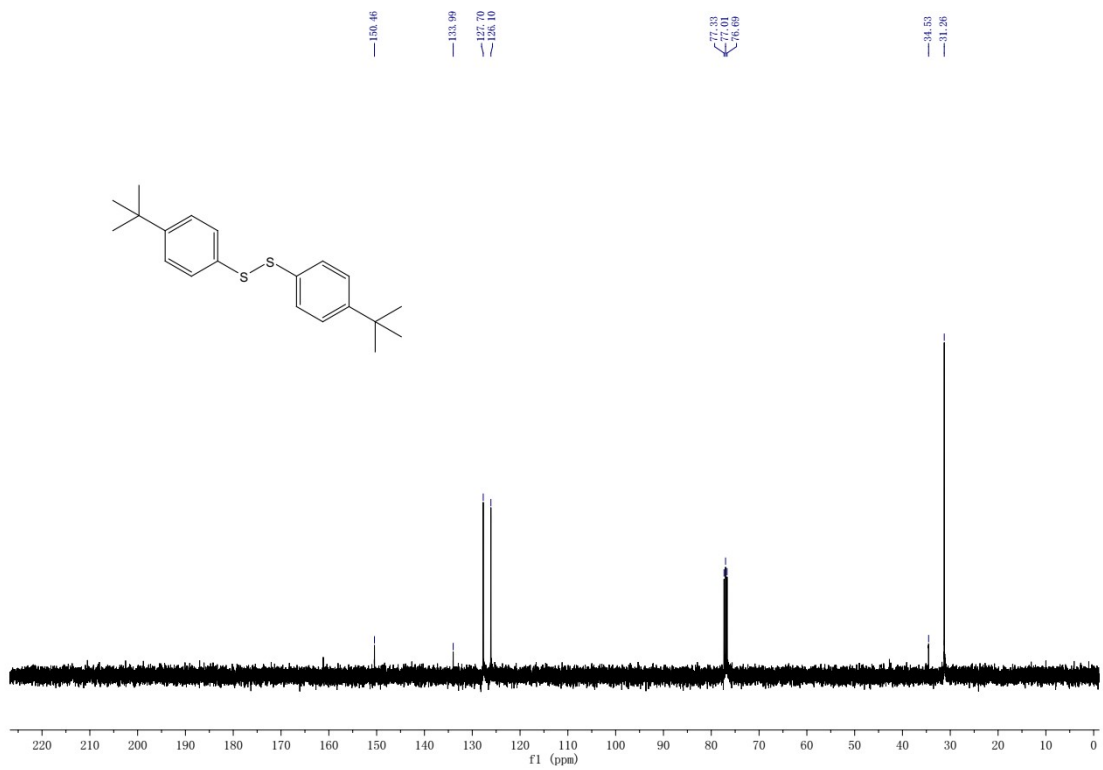
¹³C NMR spectrum for compound **3b** (CDCl₃, 101 MHz)



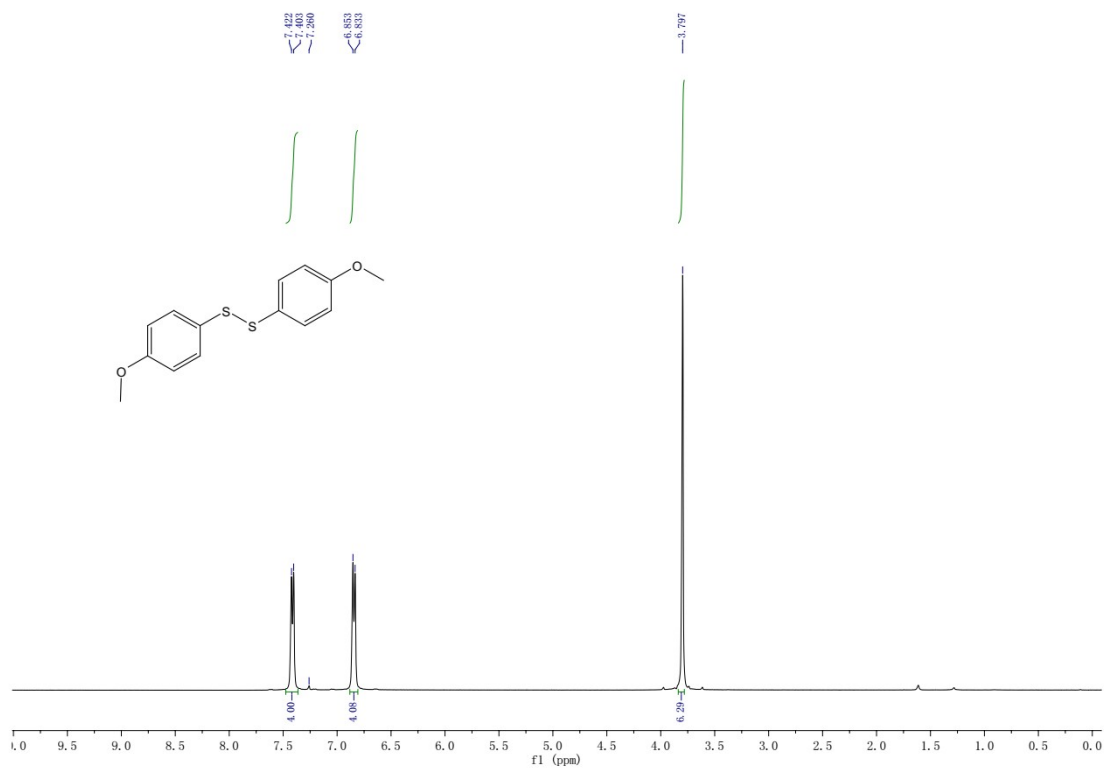
¹H NMR spectrum for compound **3c** (CDCl₃, 400 MHz)



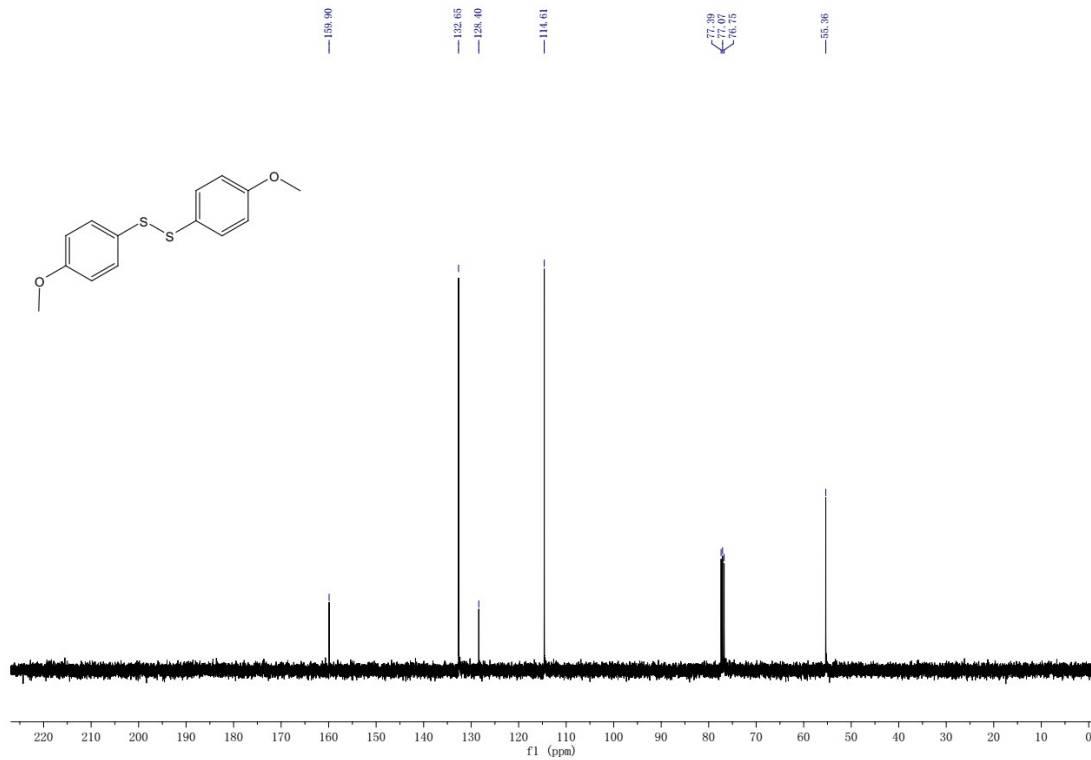
^{13}C NMR spectrum for compound **3c** (CDCl₃, 101 MHz)



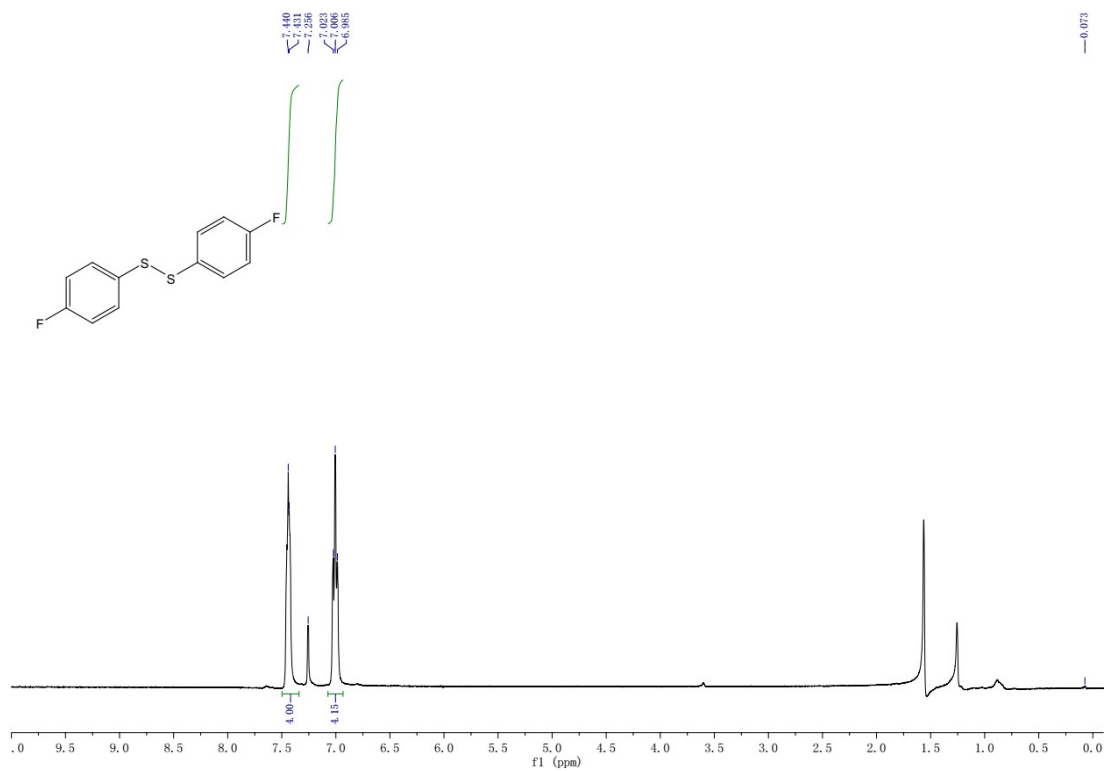
^1H NMR spectrum for compound **3d** (CDCl₃, 400 MHz)



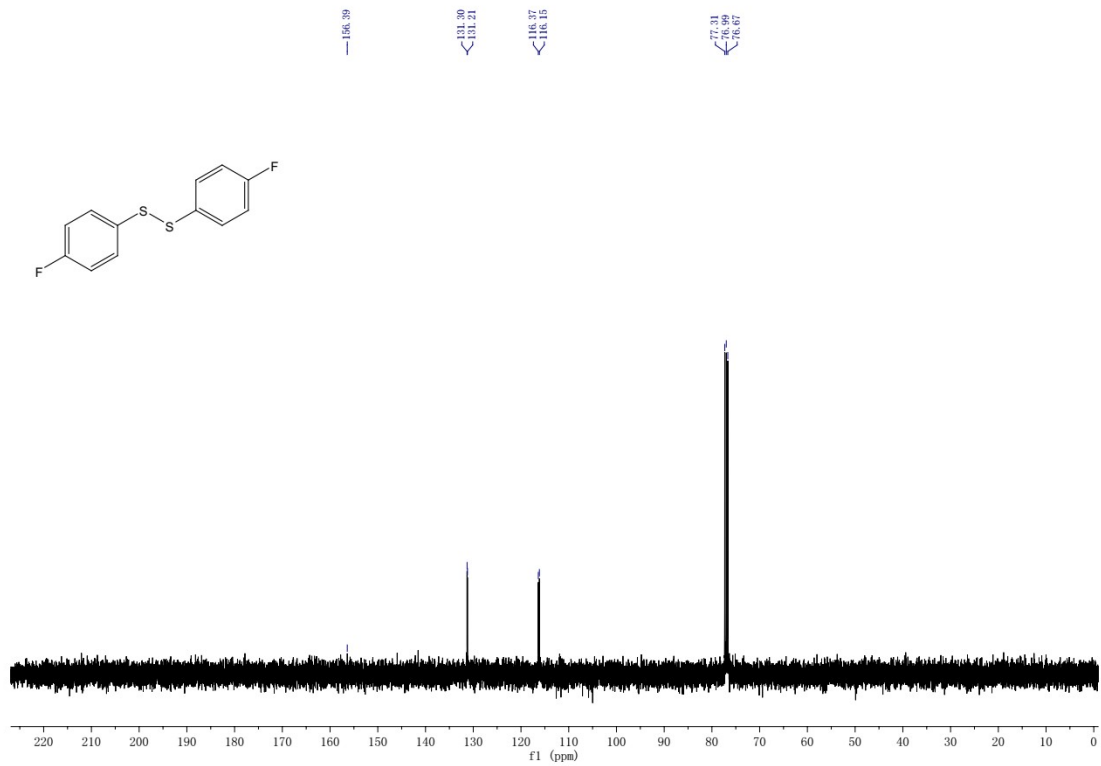
¹³C NMR spectrum for compound **3d** (CDCl₃, 101 MHz)



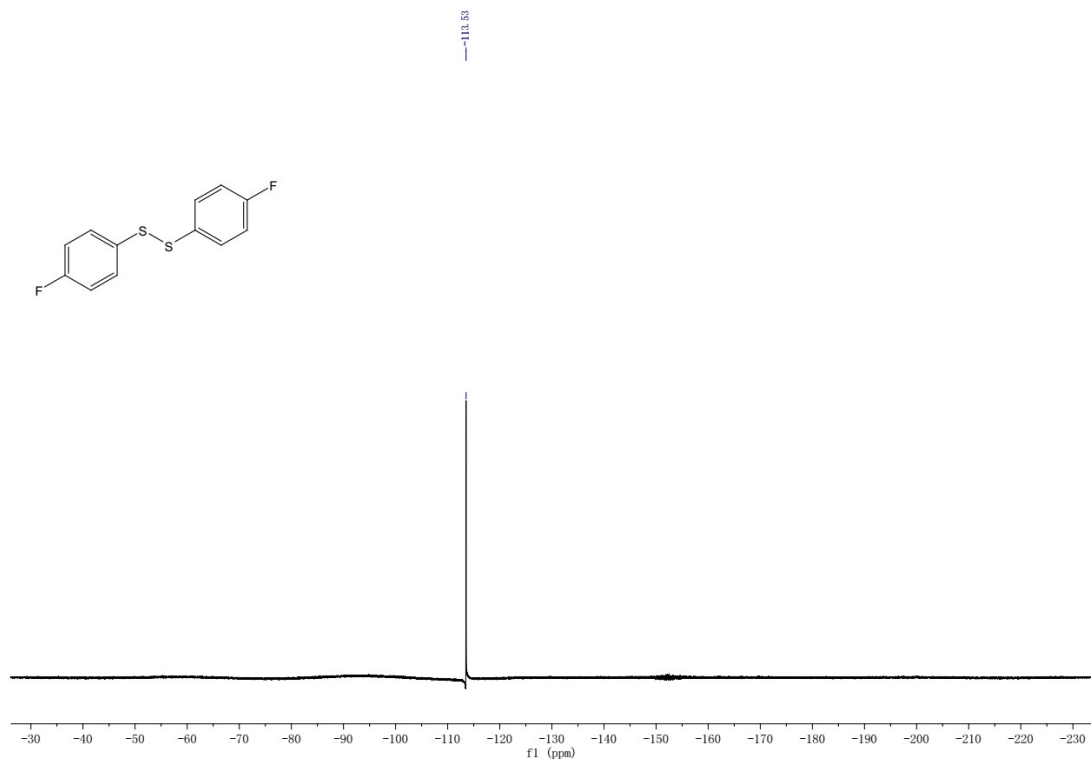
¹H NMR spectrum for compound **3e** (CDCl₃, 400 MHz)



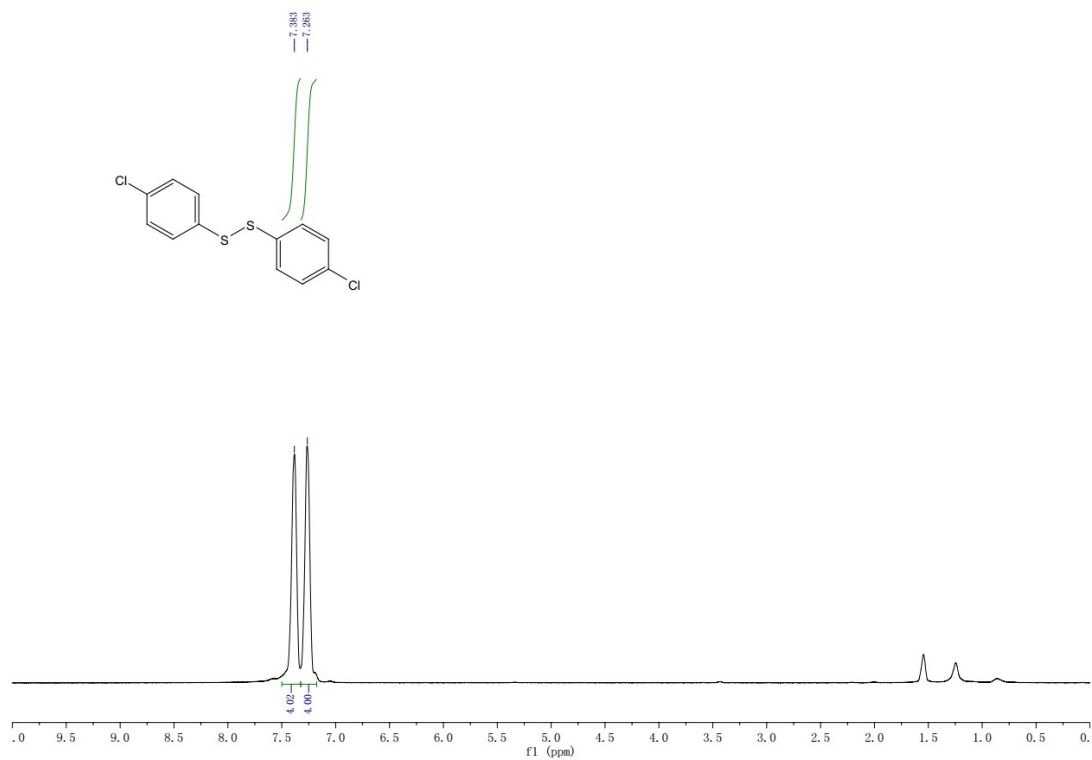
¹³C NMR spectrum for compound **3e** (CDCl₃, 101 MHz)



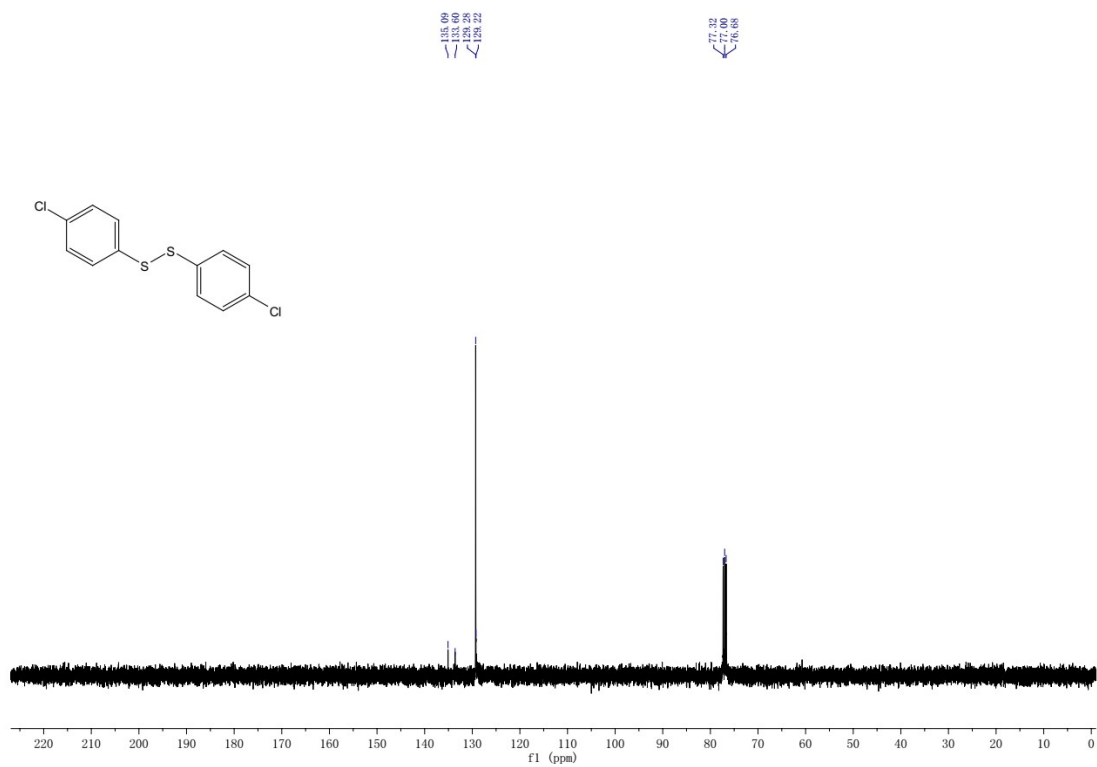
¹⁹F NMR spectrum for compound **3e** (CDCl₃, 376 MHz)



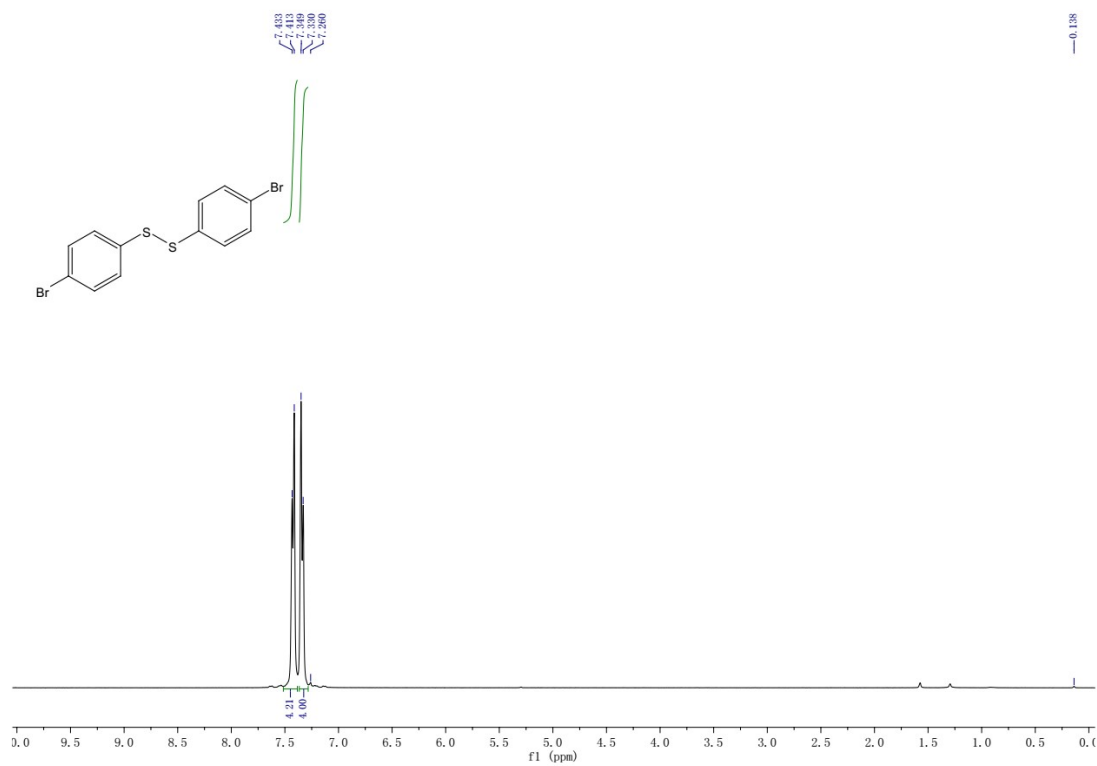
¹H NMR spectrum for compound 3f (CDCl₃, 400 MHz)



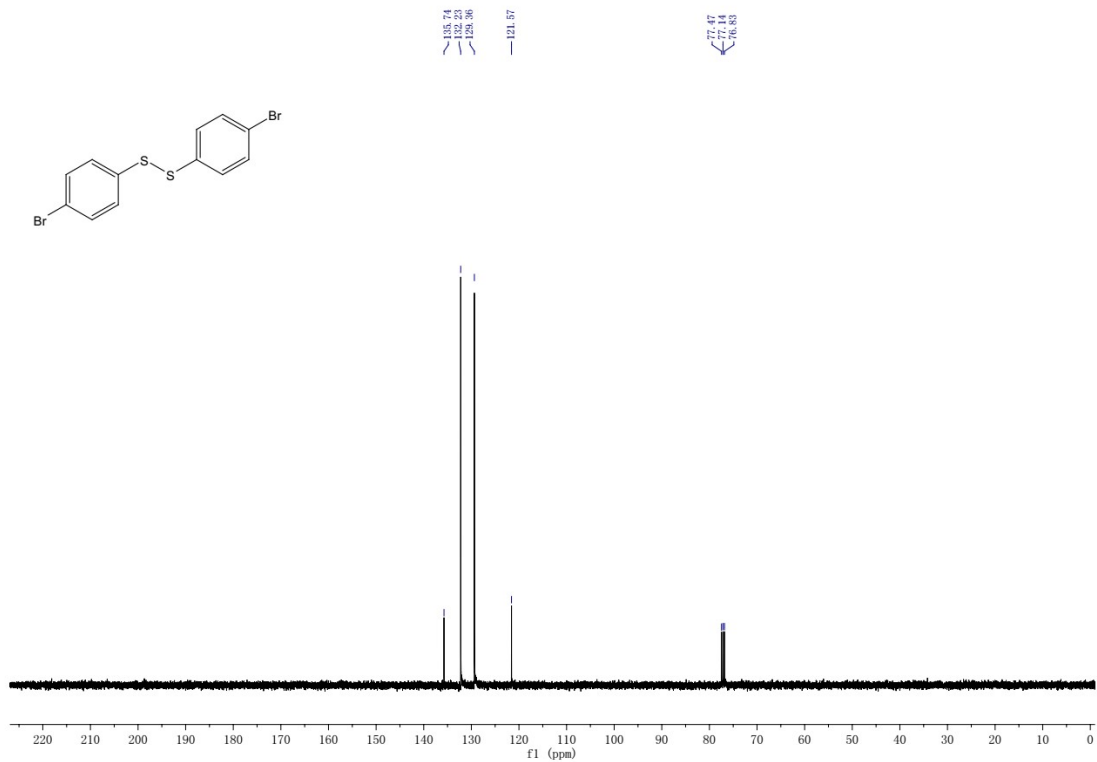
¹³C NMR spectrum for compound 3f (CDCl₃, 101 MHz)



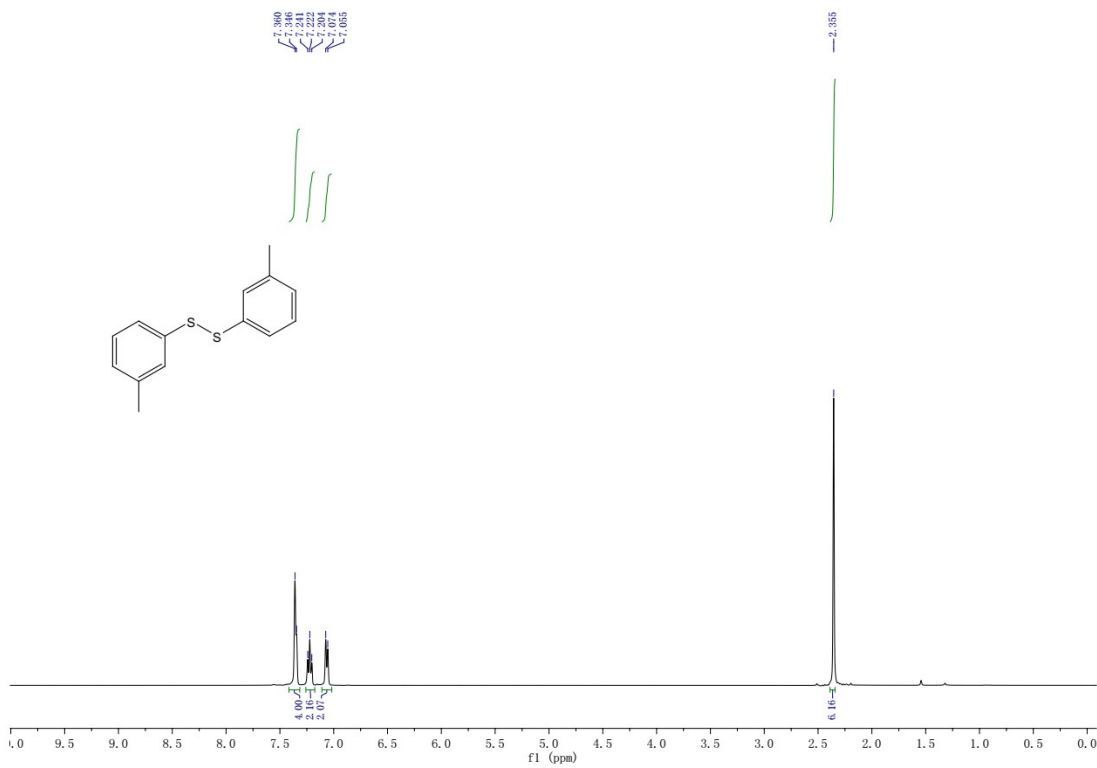
¹H NMR spectrum for compound **3g** (CDCl₃, 400 MHz)



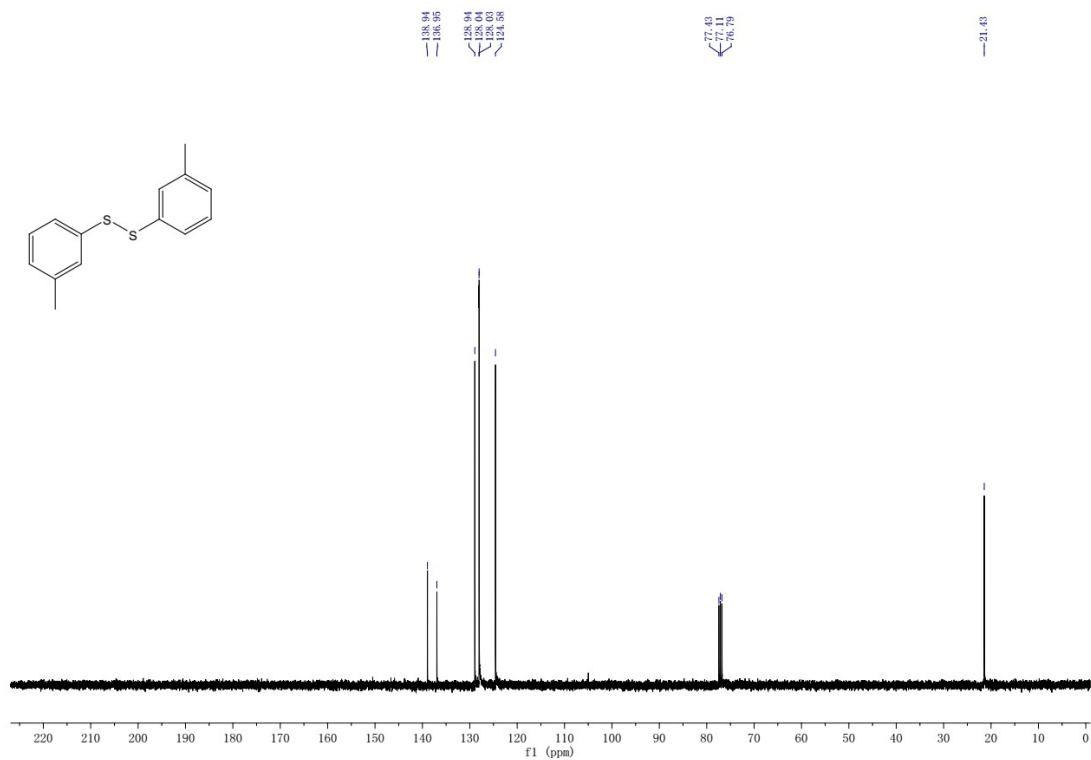
¹³C NMR spectrum for compound **3g** (CDCl₃, 101 MHz)



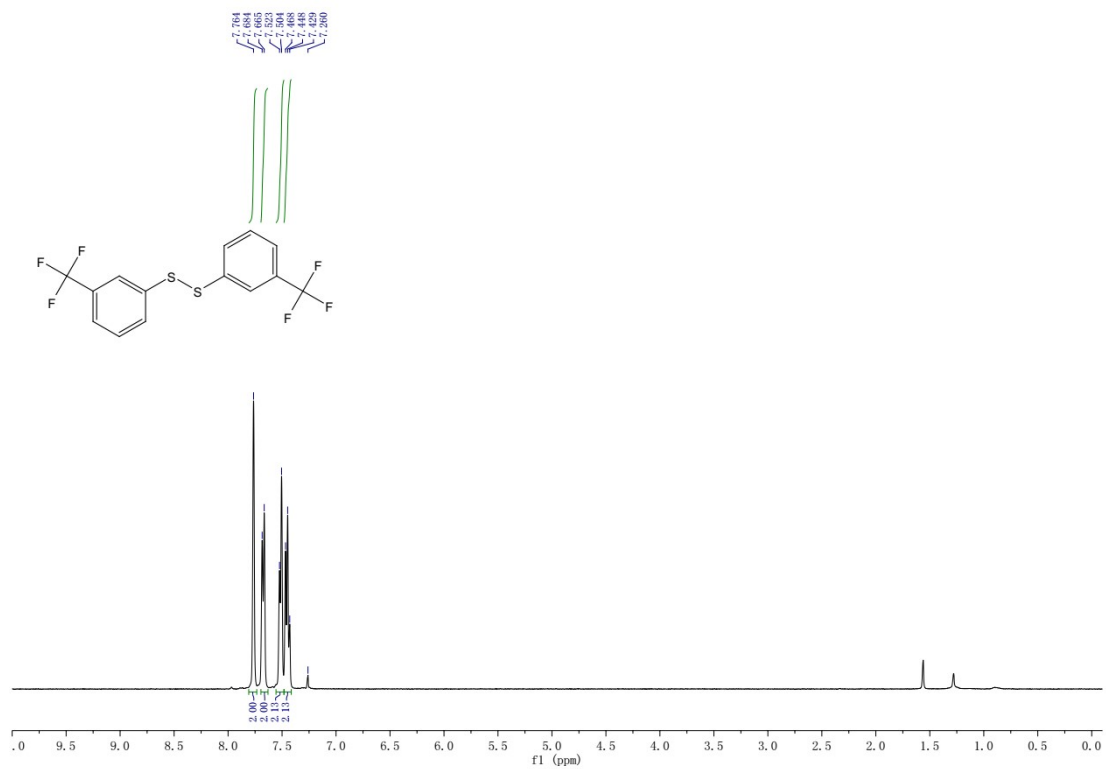
¹H NMR spectrum for compound **3h** (CDCl₃, 400 MHz)



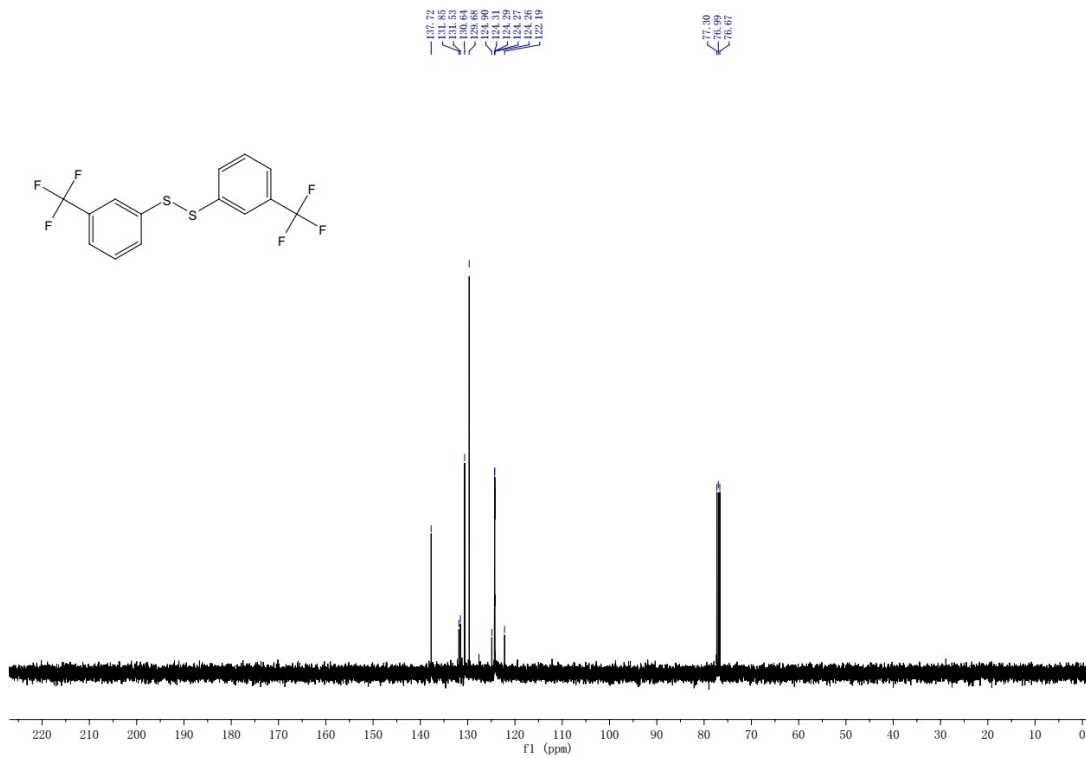
¹³C NMR spectrum for compound **3h** (CDCl₃, 101 MHz)



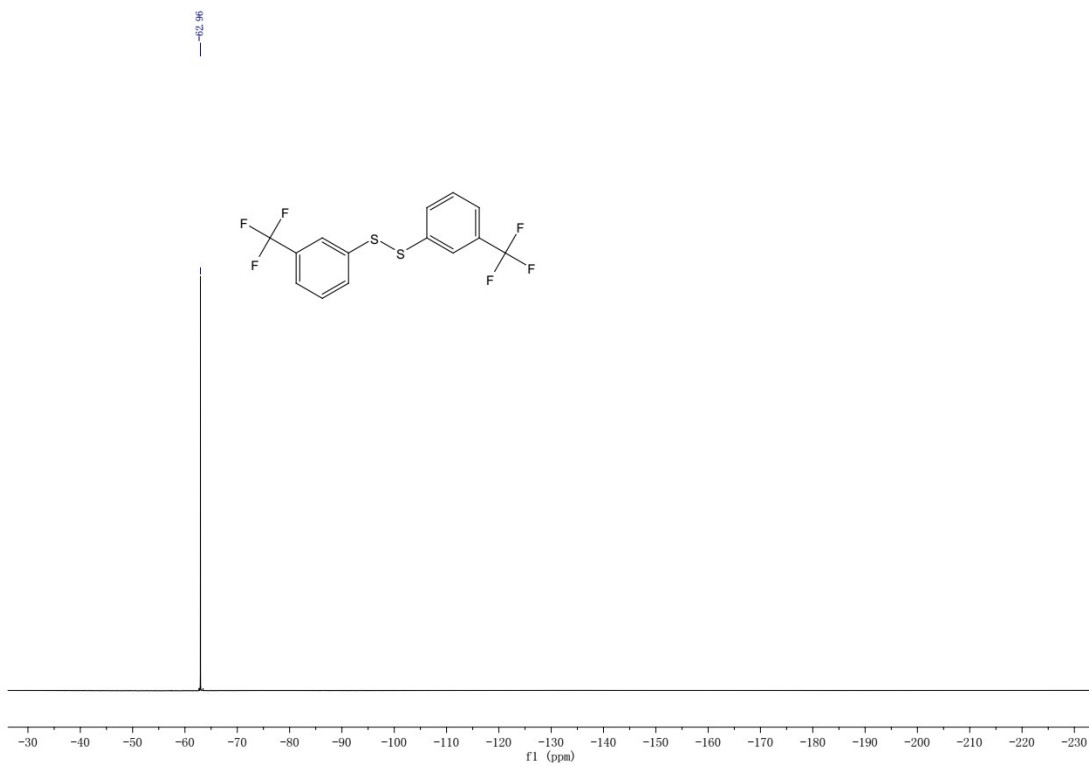
^1H NMR spectrum for compound **3i** (CDCl_3 , 400 MHz)



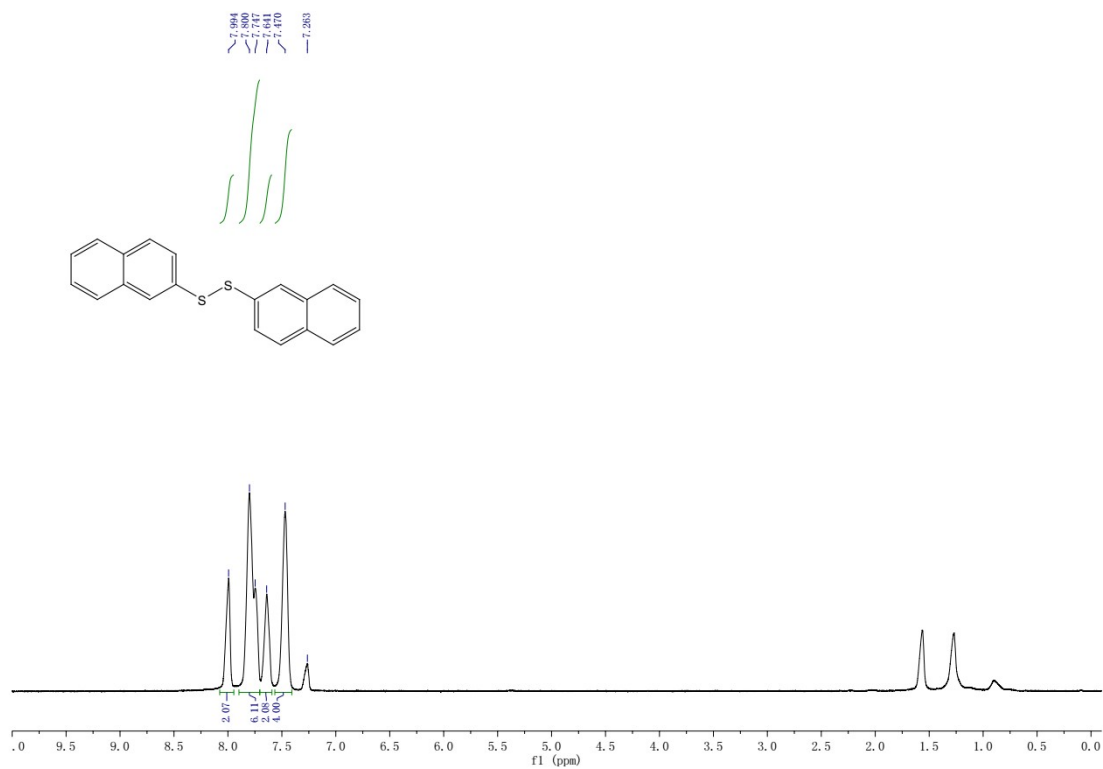
^{13}C NMR spectrum for compound **3i** (CDCl_3 , 101 MHz)



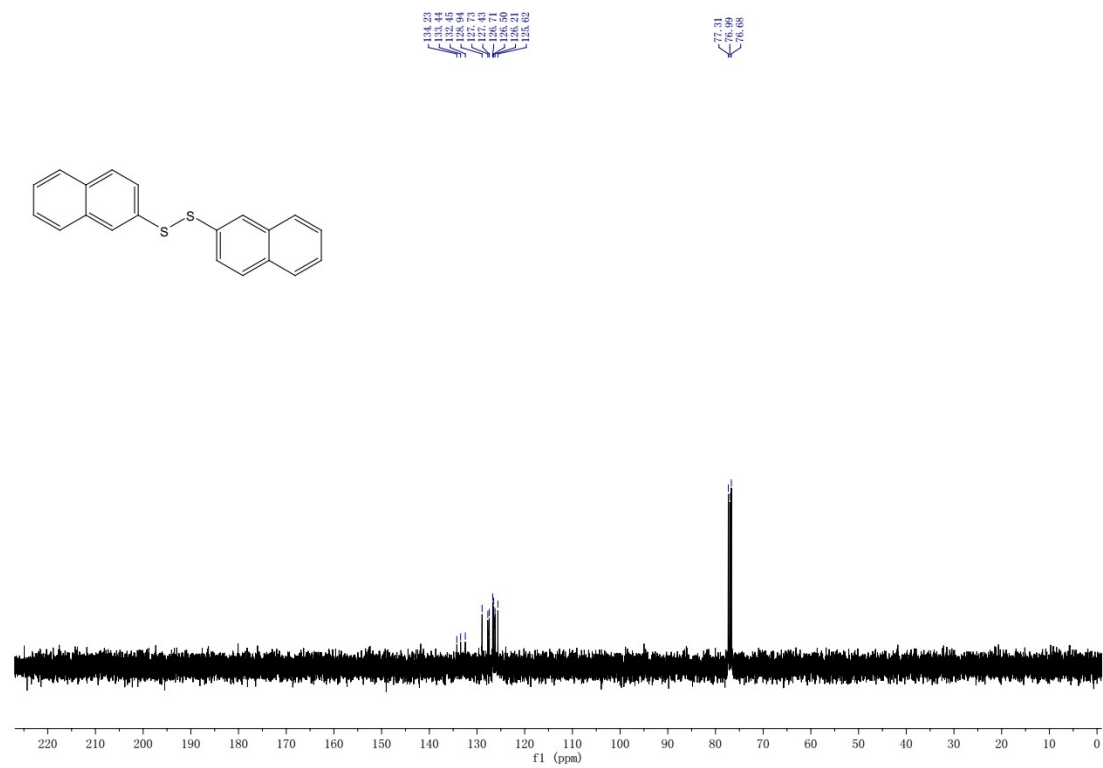
^{19}F NMR spectrum for compound **3i** (CDCl_3 , 376 MHz)



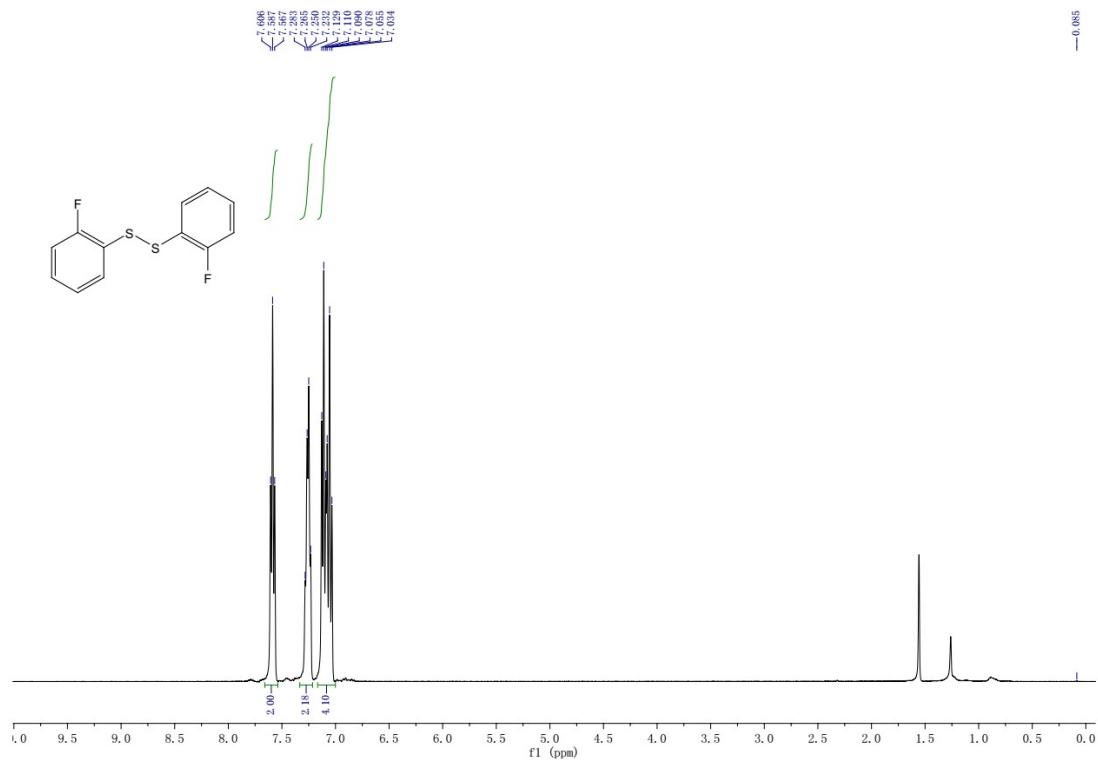
^1H NMR spectrum for compound **3j** (CDCl_3 , 400 MHz)



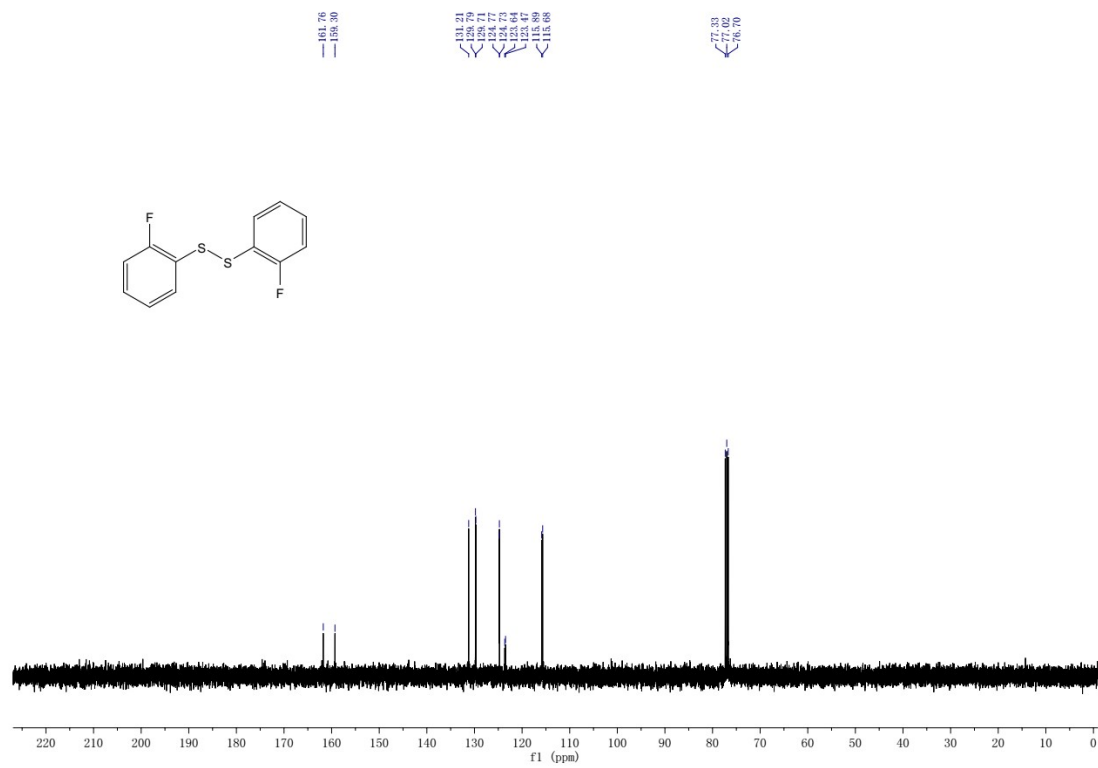
¹³C NMR spectrum for compound **3j** (CDCl₃, 101 MHz)



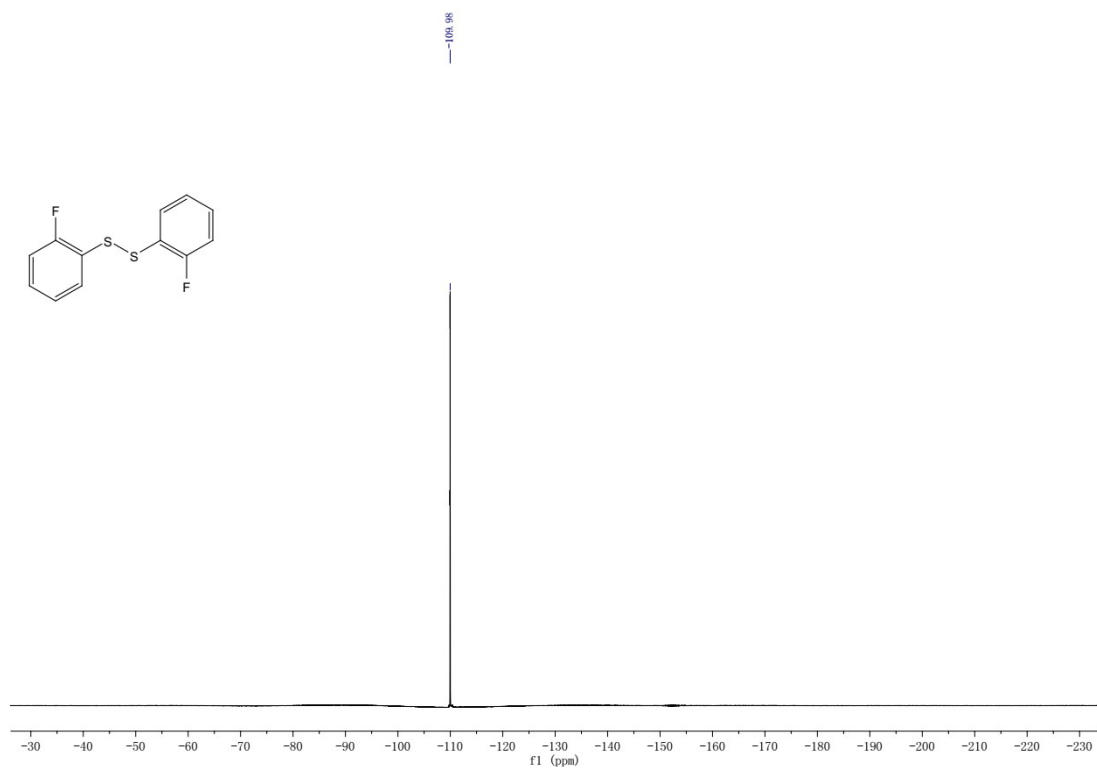
¹H NMR spectrum for compound **3k** (CDCl₃, 400 MHz)



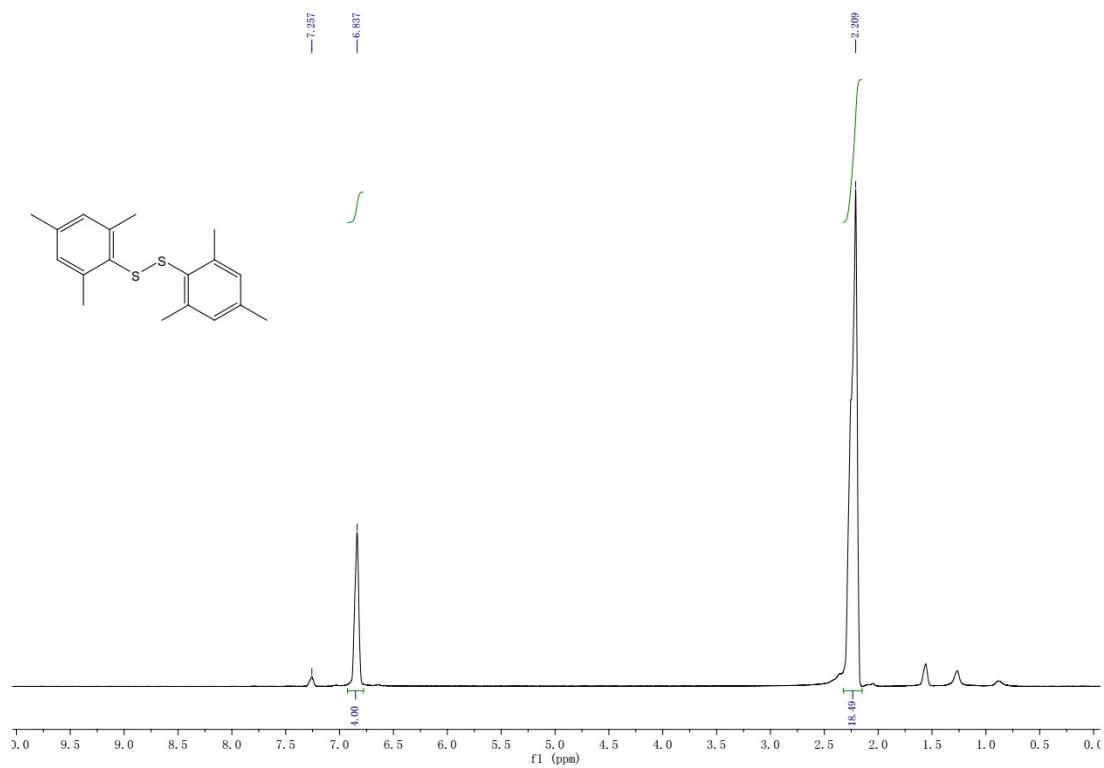
^{13}C NMR spectrum for compound **3k** (CDCl_3 , 101 MHz)



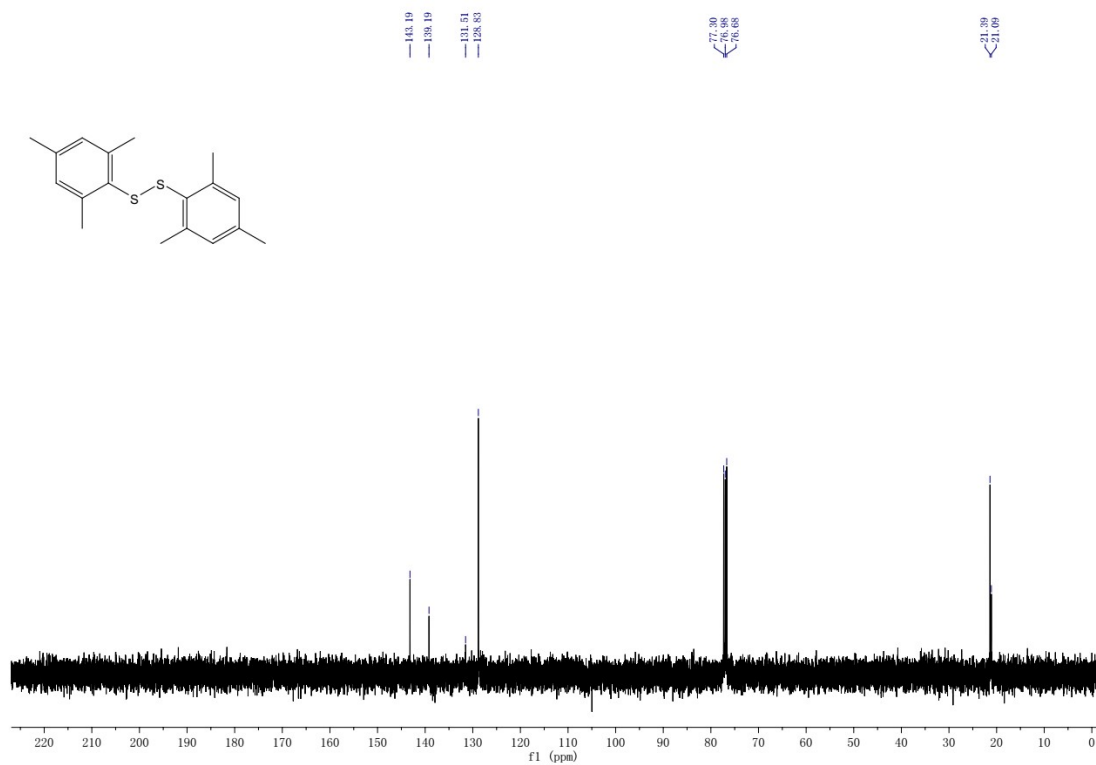
^{19}F NMR spectrum for compound **3k** (CDCl_3 , 376 MHz)



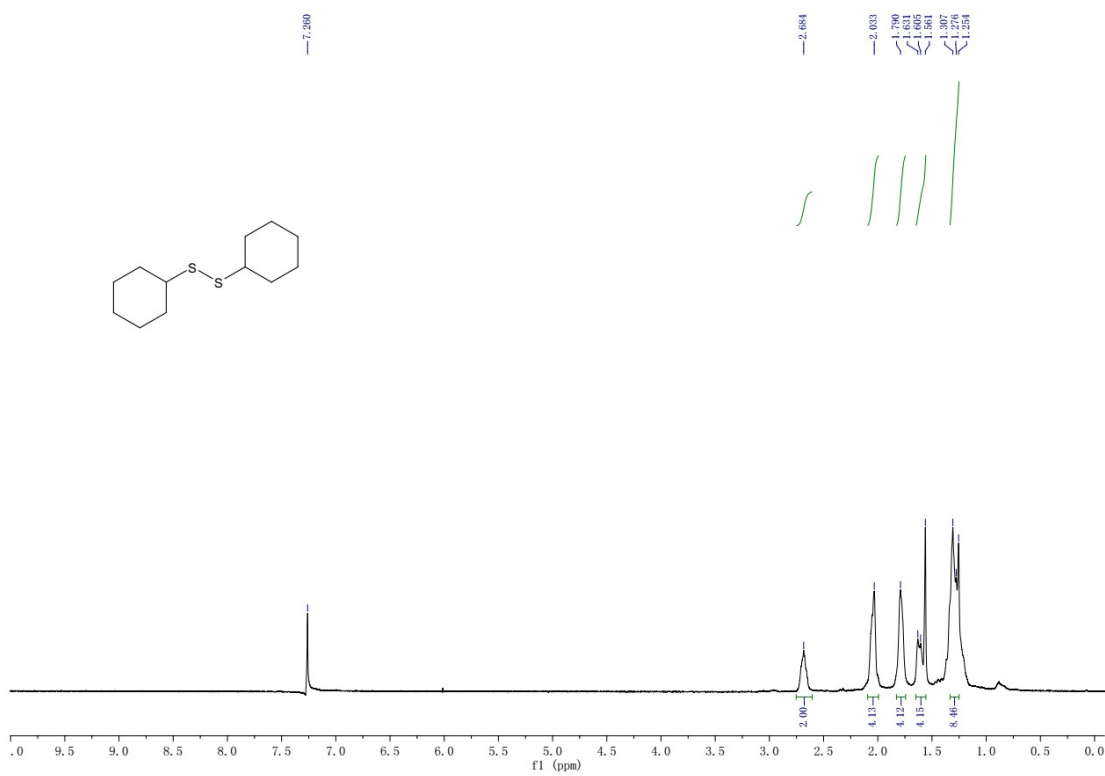
^1H NMR spectrum for compound **31** (CDCl_3 , 400 MHz)



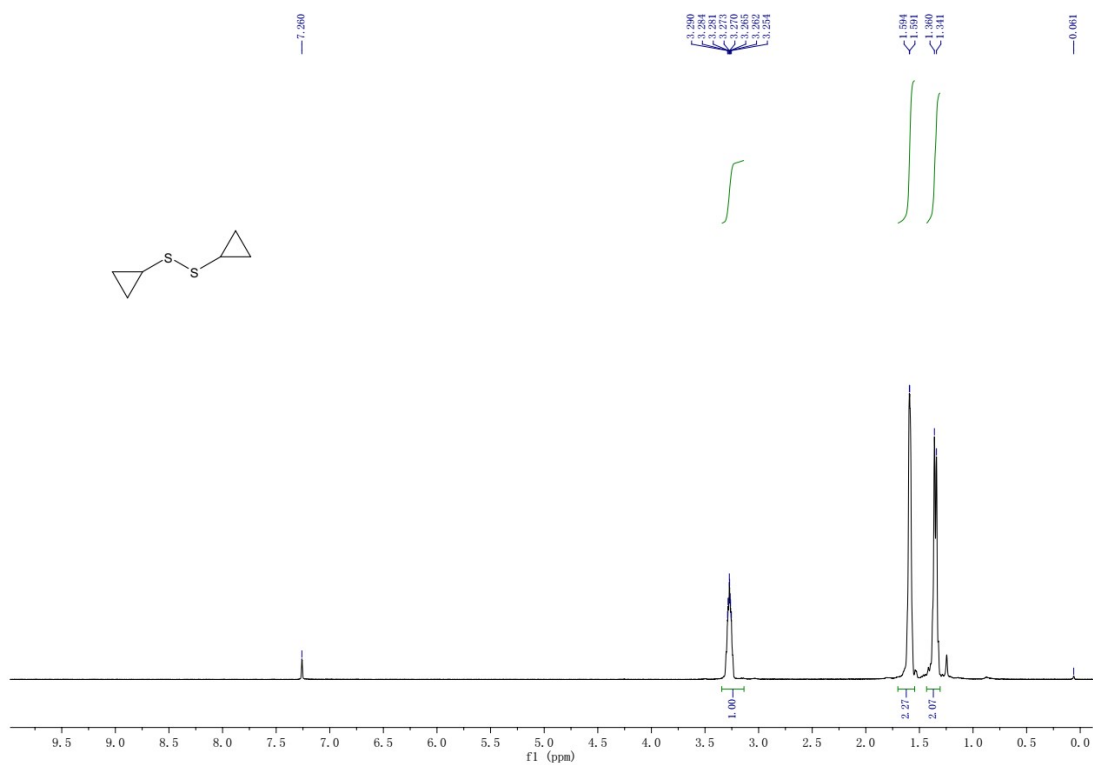
^{13}C NMR spectrum for compound **31** (CDCl_3 , 101 MHz)



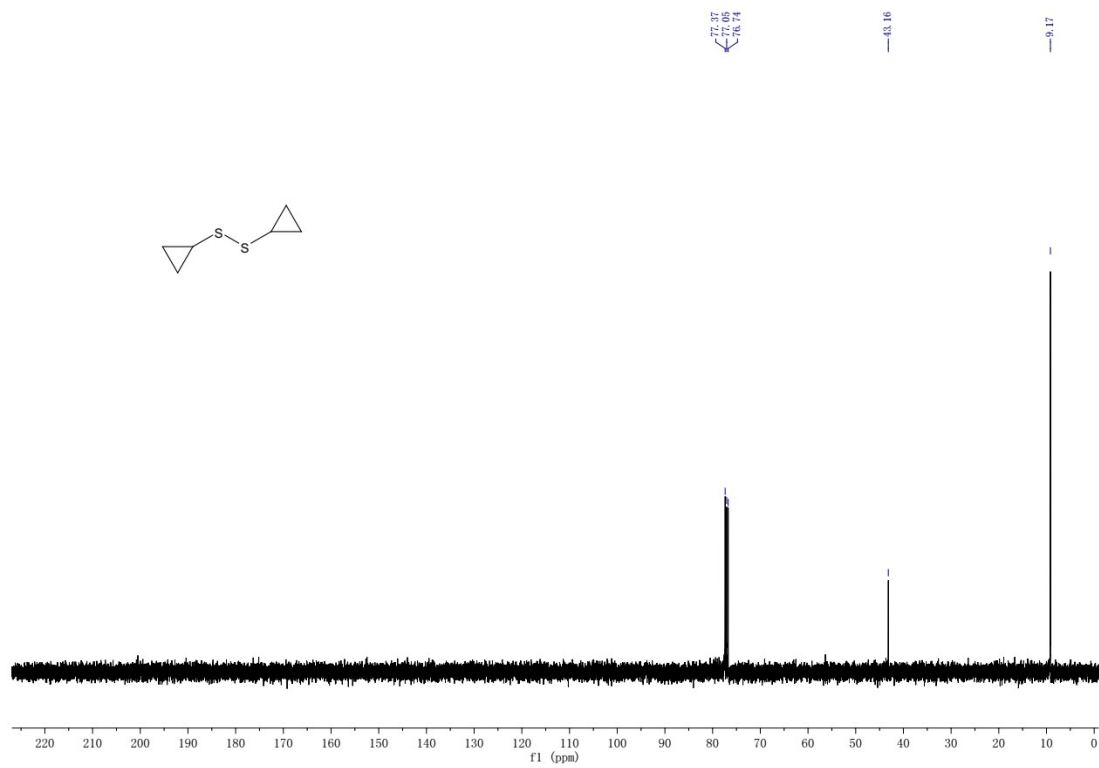
¹H NMR spectrum for compound **3m** (CDCl₃, 400 MHz)



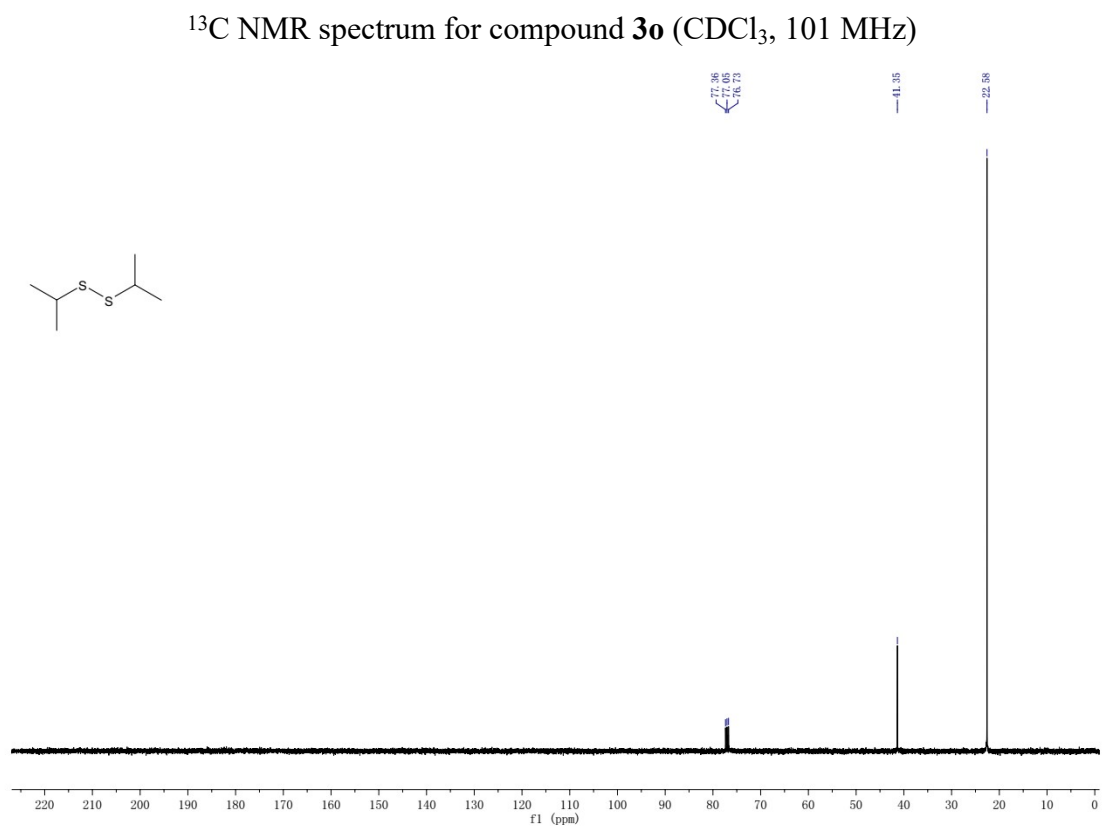
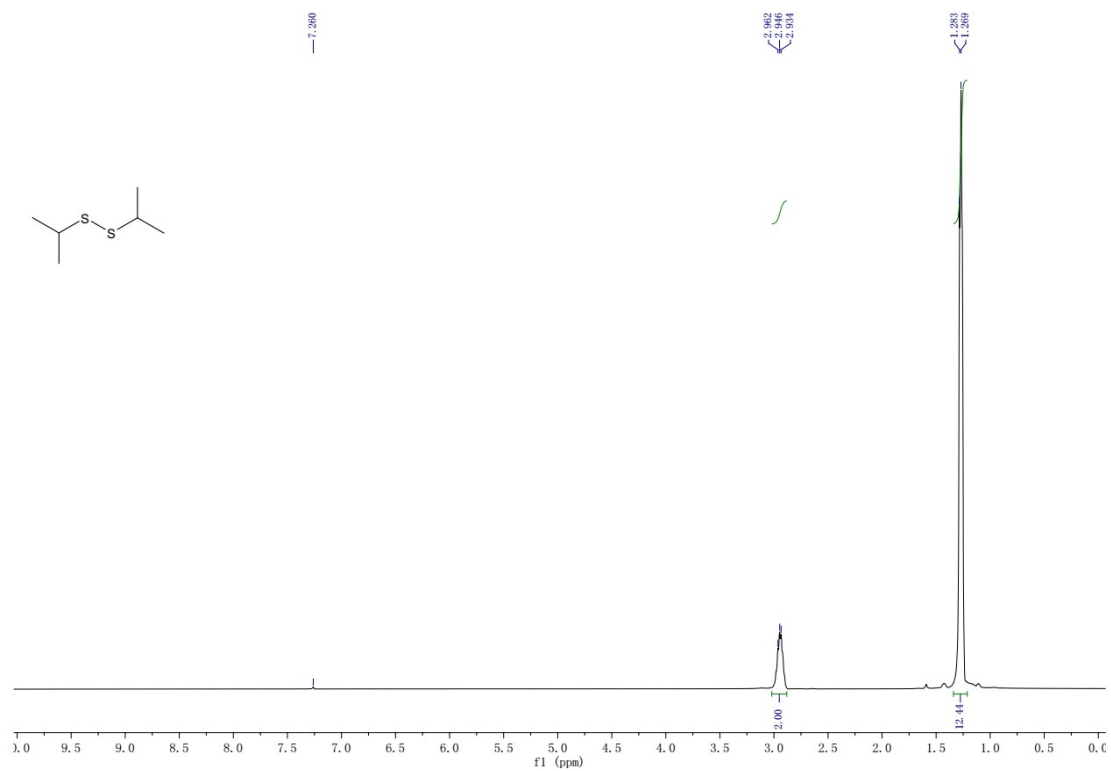
¹H NMR spectrum for compound **3n** (CDCl₃, 400 MHz)



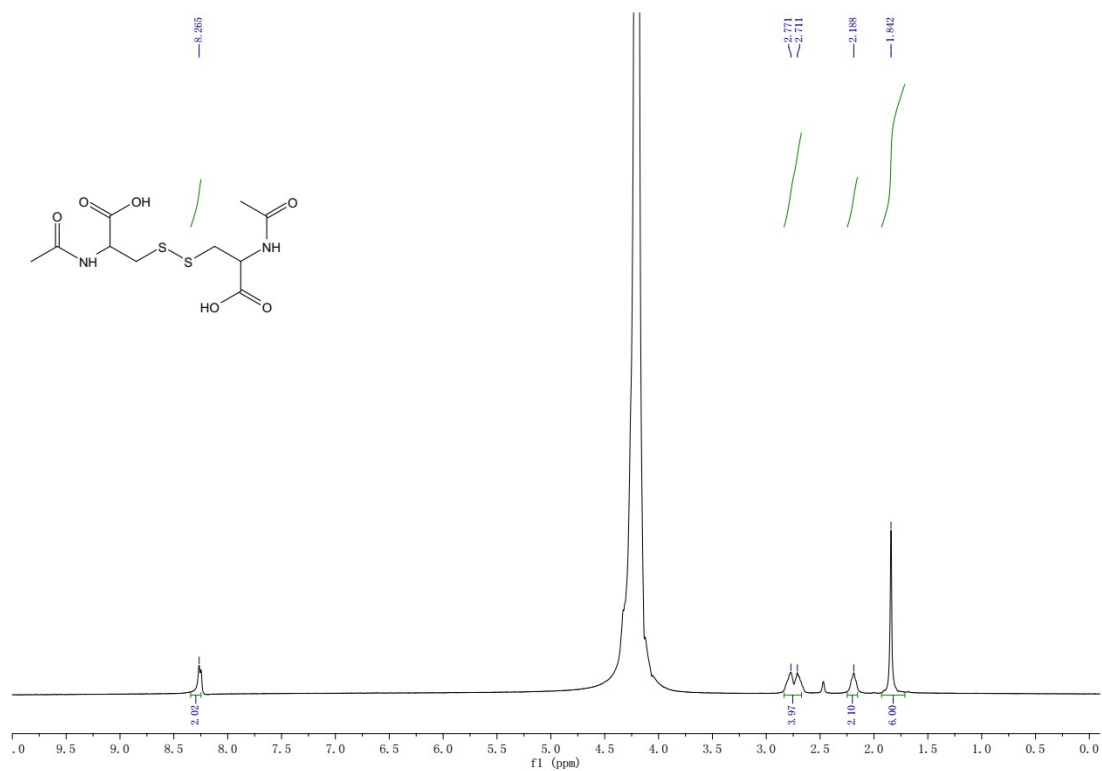
^{13}C NMR spectrum for compound **3n** (CDCl_3 , 101 MHz)



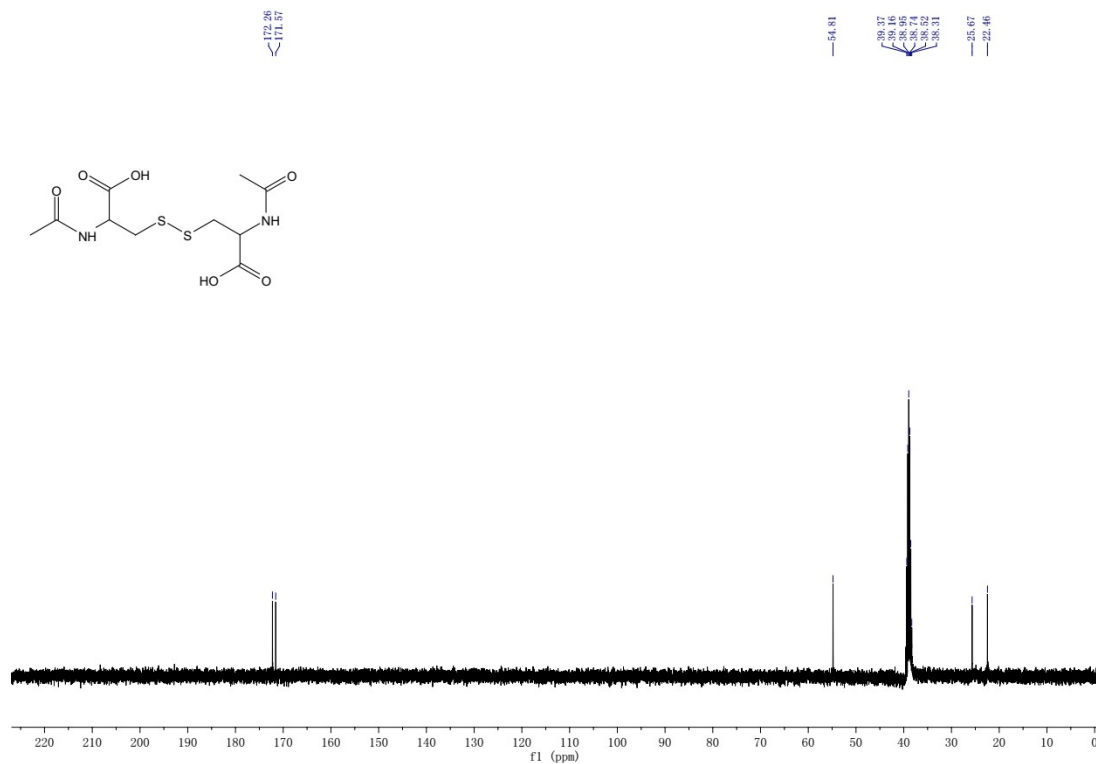
^1H NMR spectrum for compound **3o** (CDCl_3 , 400 MHz)



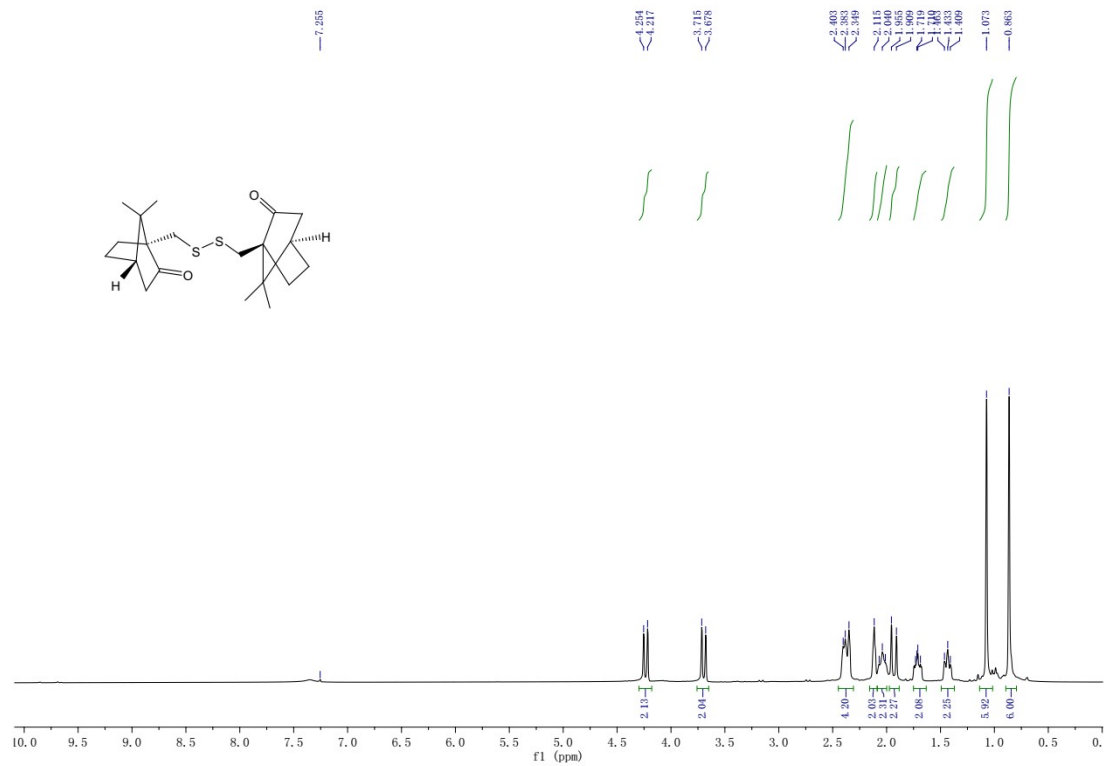
¹H NMR spectrum for compound **3p** (D₆-DMSO, 400 MHz)



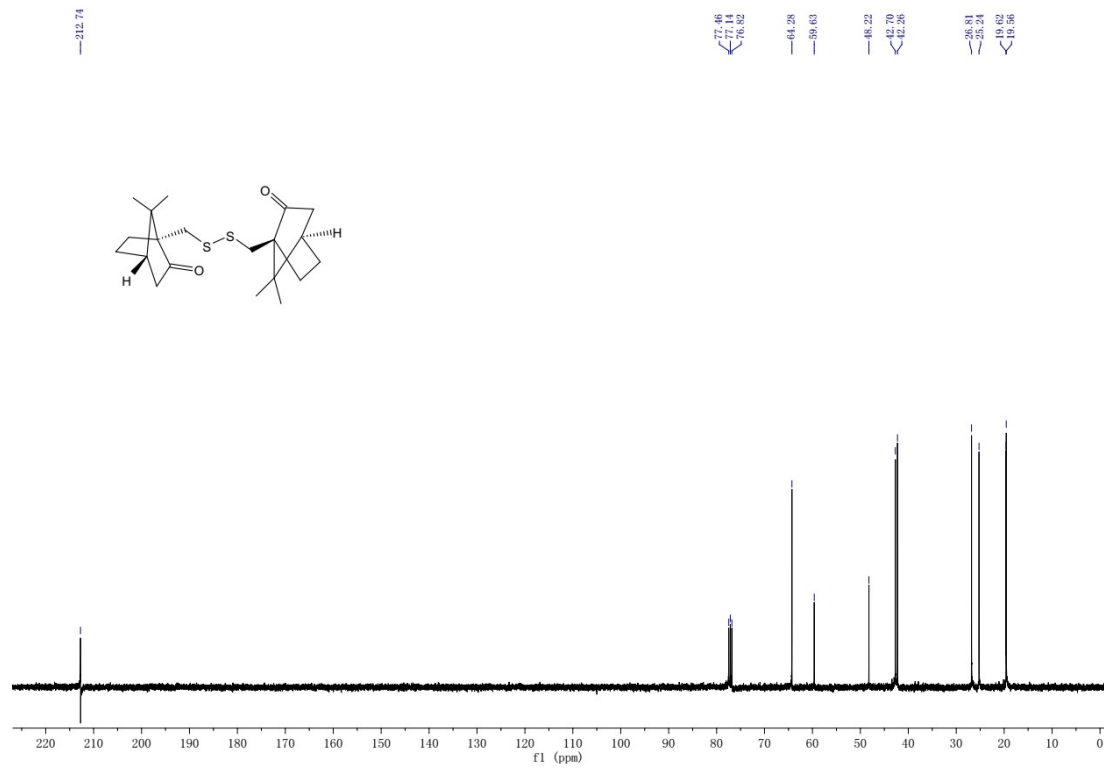
¹³C NMR spectrum for compound **3p** (D₆-DMSO, 101 MHz)



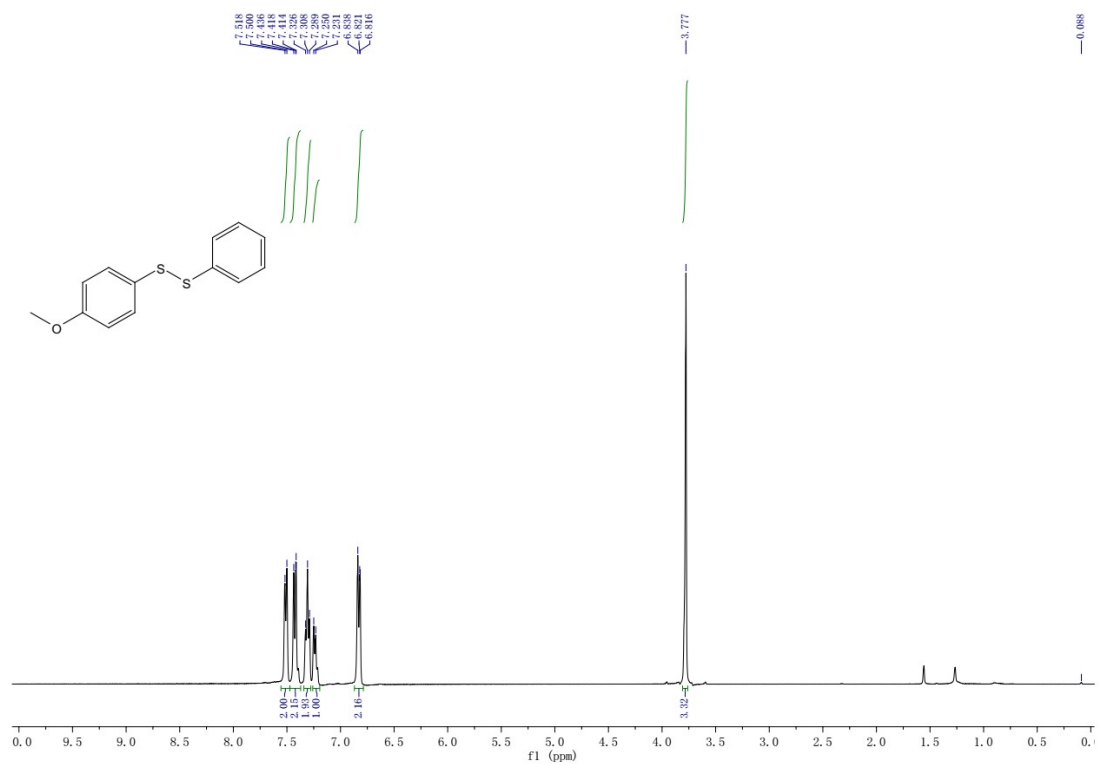
¹H NMR spectrum for compound **3q** (CDCl₃, 400 MHz)



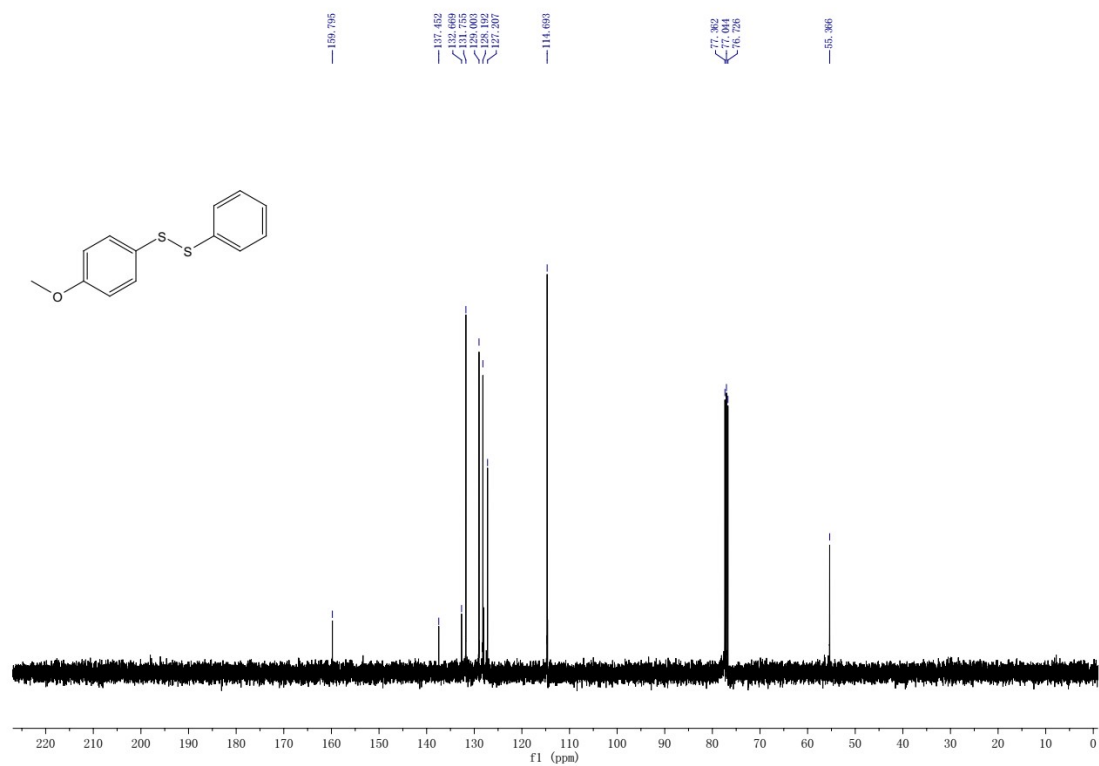
¹³C NMR spectrum for compound **3q** (CDCl₃, 101 MHz)



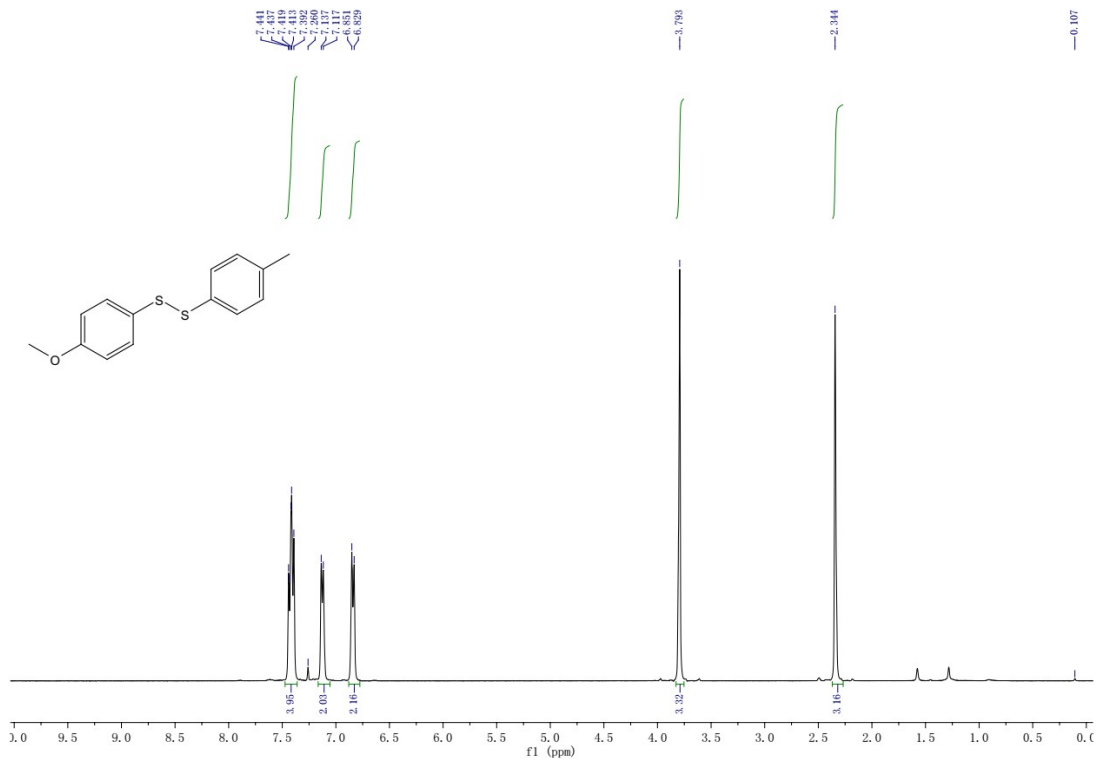
¹H NMR spectrum for compound **3r** (CDCl₃, 400 MHz)



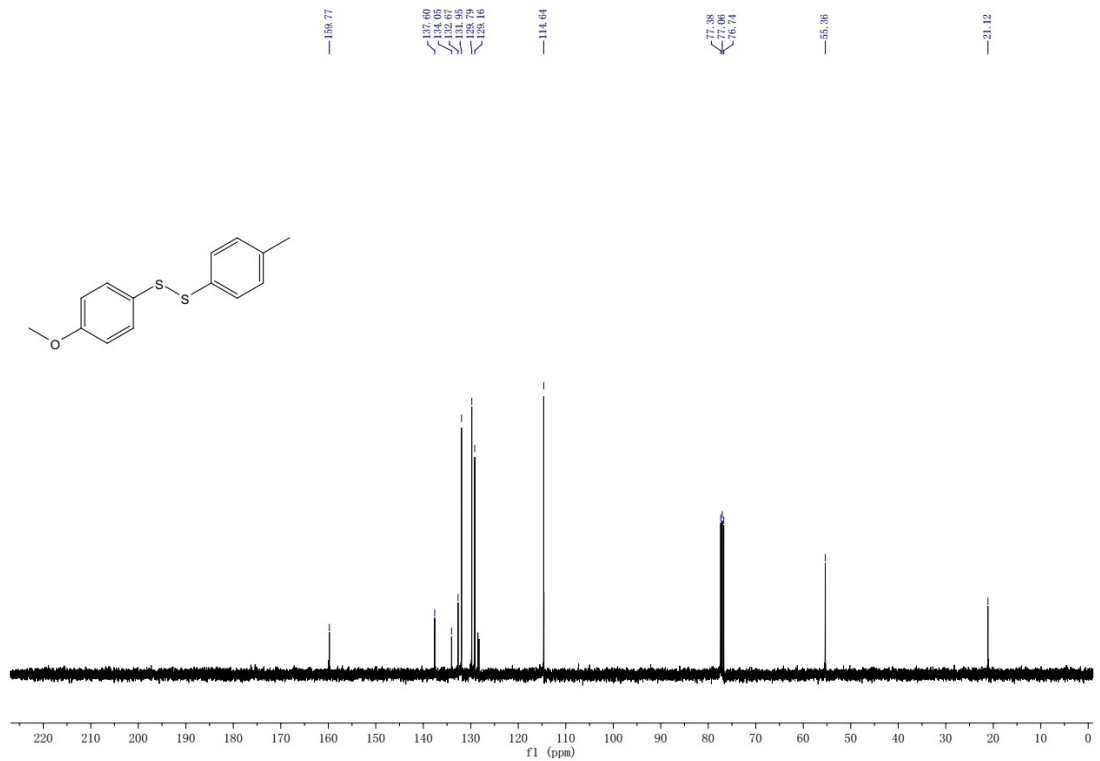
¹³C NMR spectrum for compound **3r** (CDCl₃, 101 MHz)



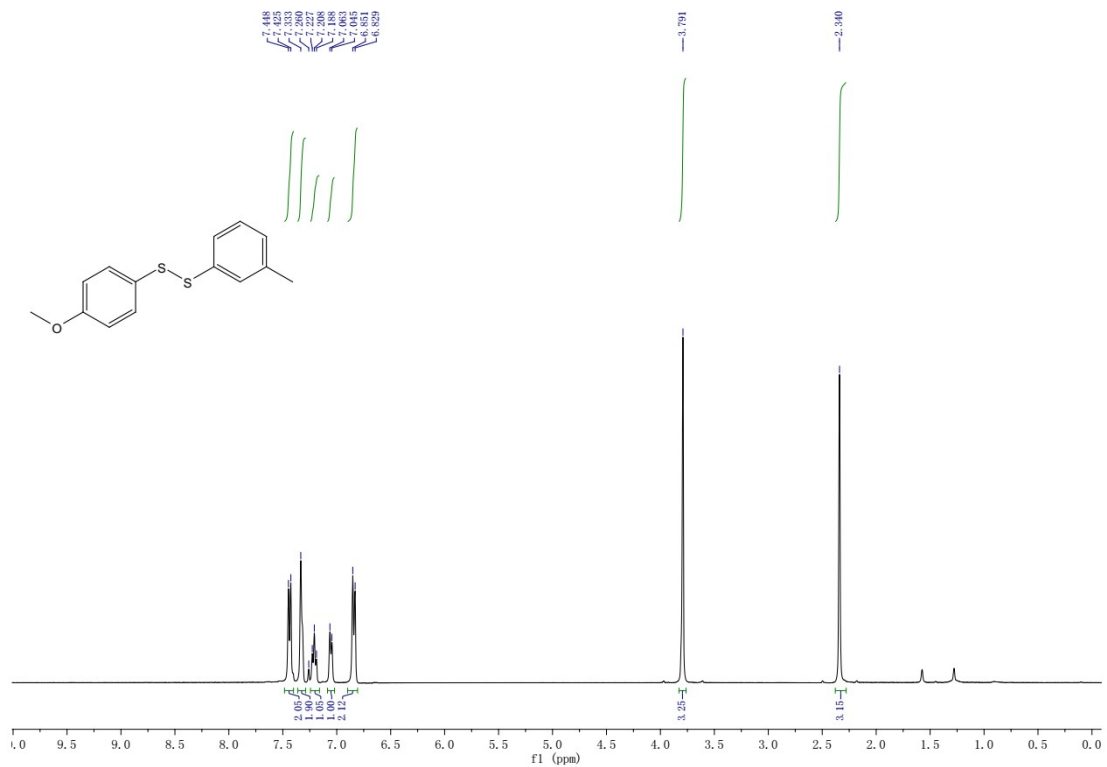
¹H NMR spectrum for compound **3s** (CDCl₃, 400 MHz)



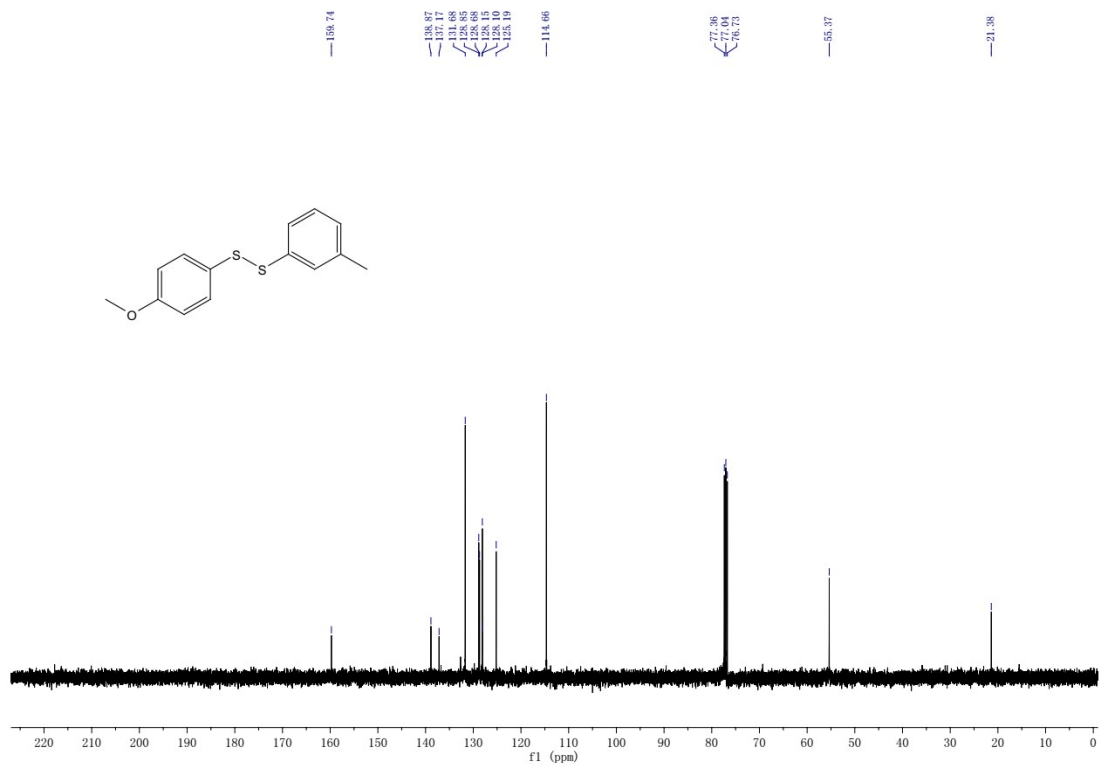
^{13}C NMR spectrum for compound **3s** (CDCl_3 , 101 MHz)



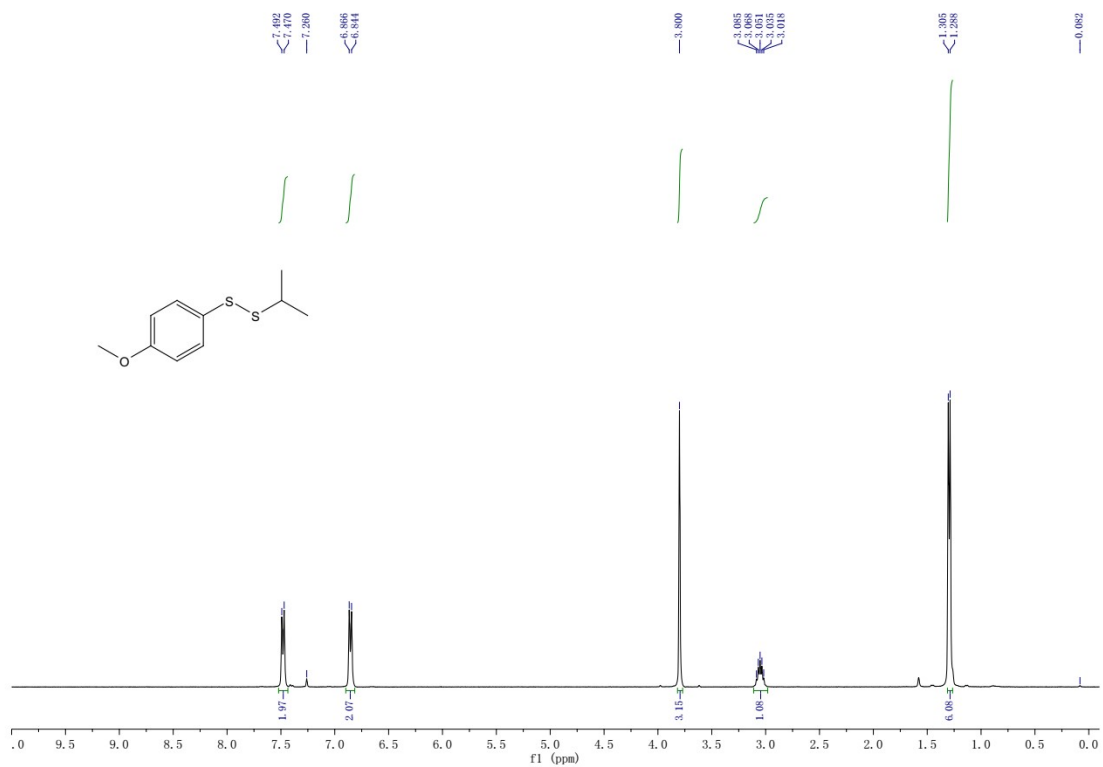
^1H NMR spectrum for compound **3t** (CDCl_3 , 400 MHz)



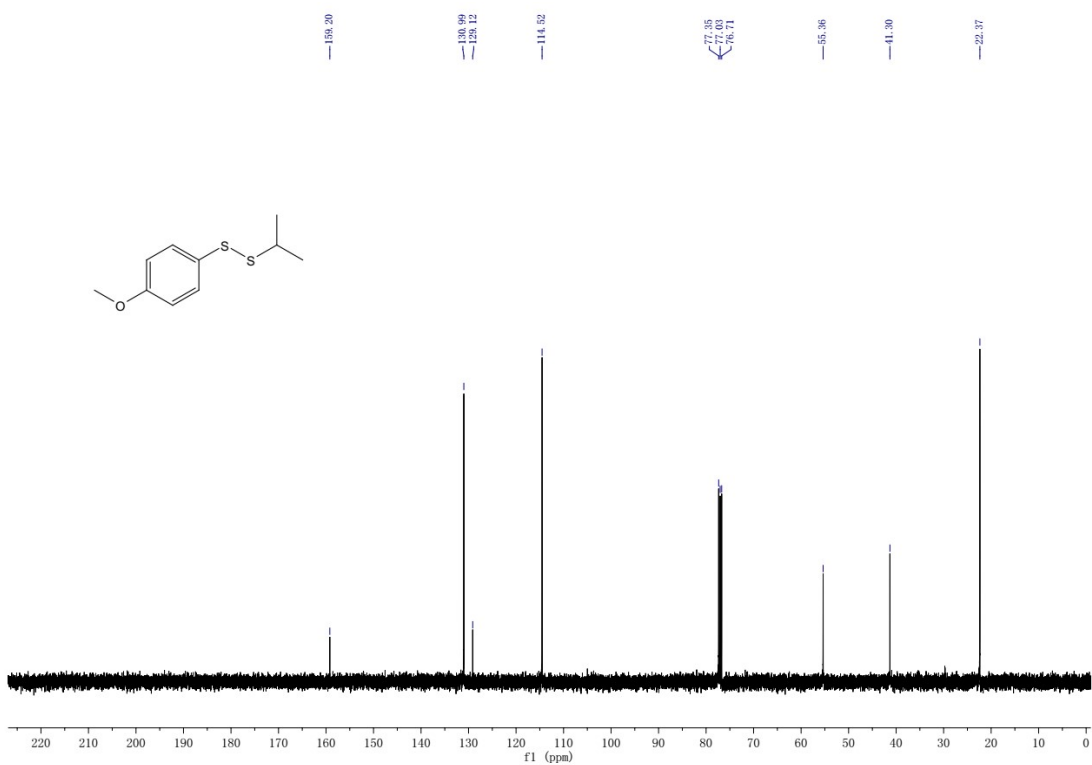
¹³C NMR spectrum for compound 3t (CDCl₃, 101 MHz)



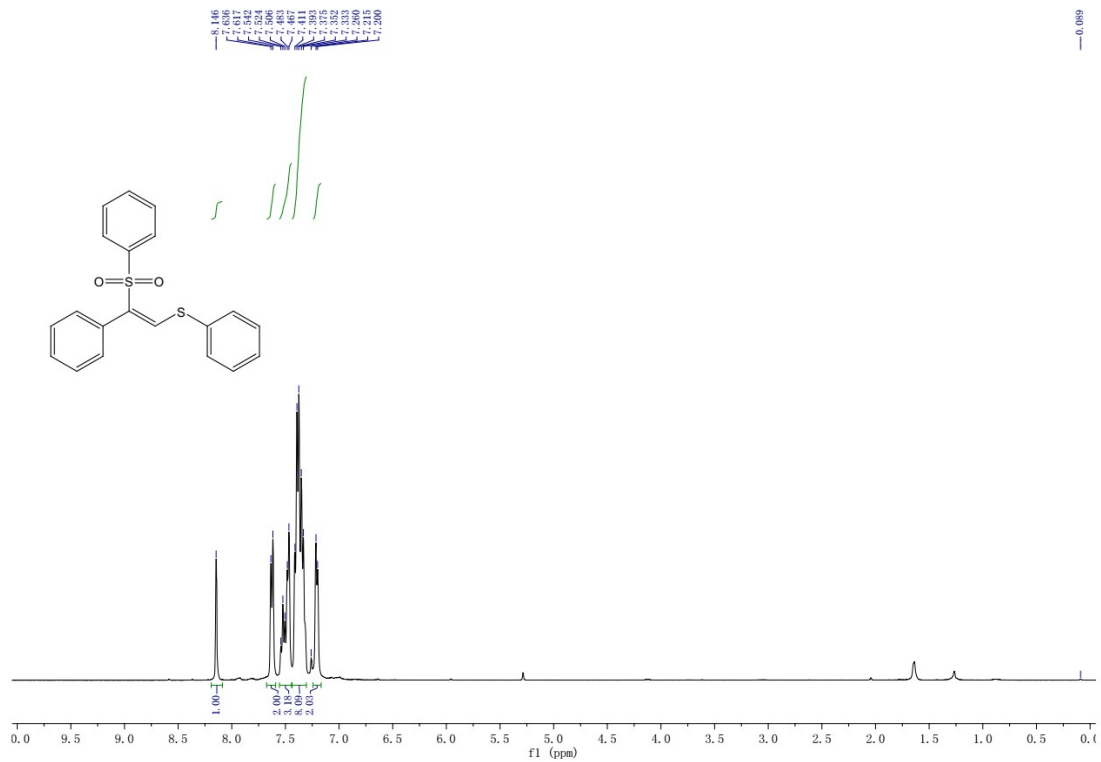
¹H NMR spectrum for compound 3u (CDCl₃, 400 MHz)



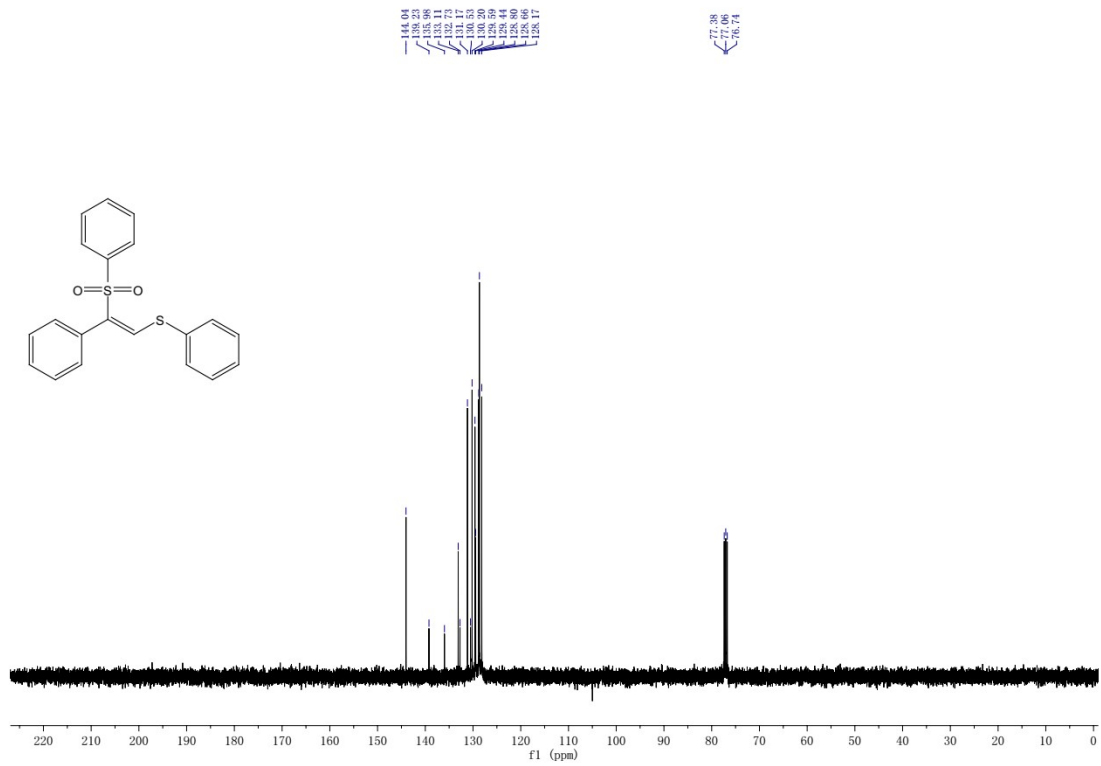
^{13}C NMR spectrum for compound **3u** (CDCl_3 , 101 MHz)



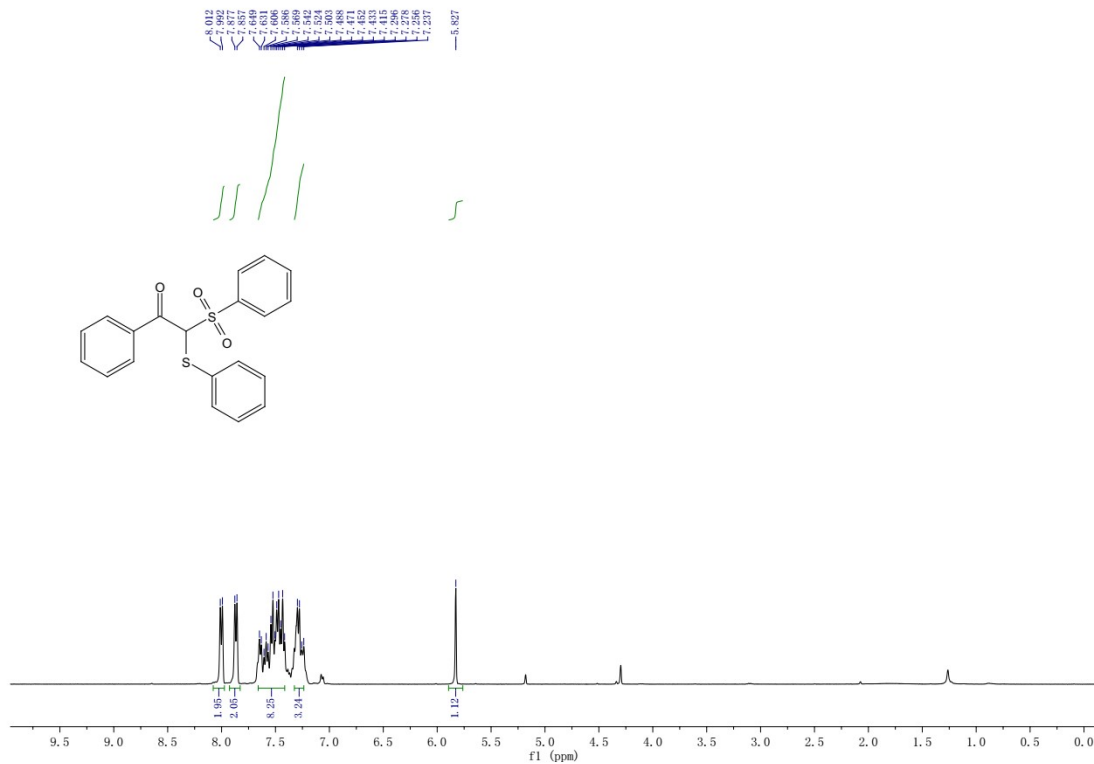
^1H NMR spectrum for compound **4** (CDCl_3 , 400 MHz)



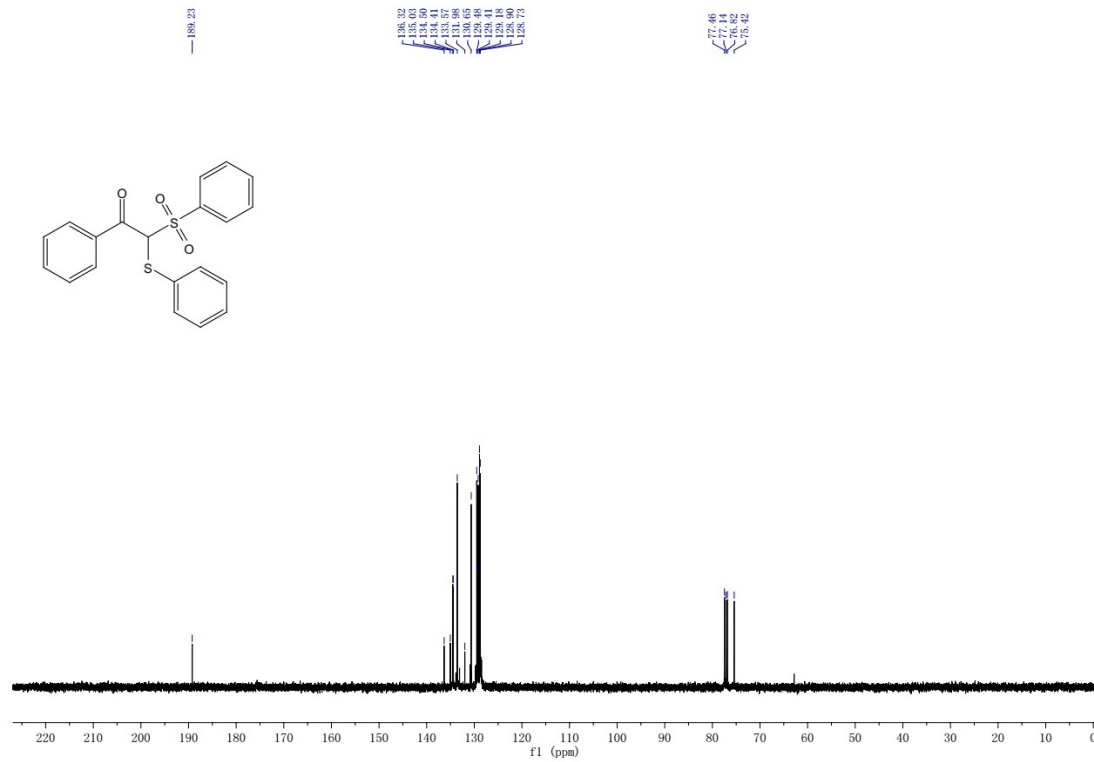
^{13}C NMR spectrum for compound 4 (CDCl_3 , 101 MHz)



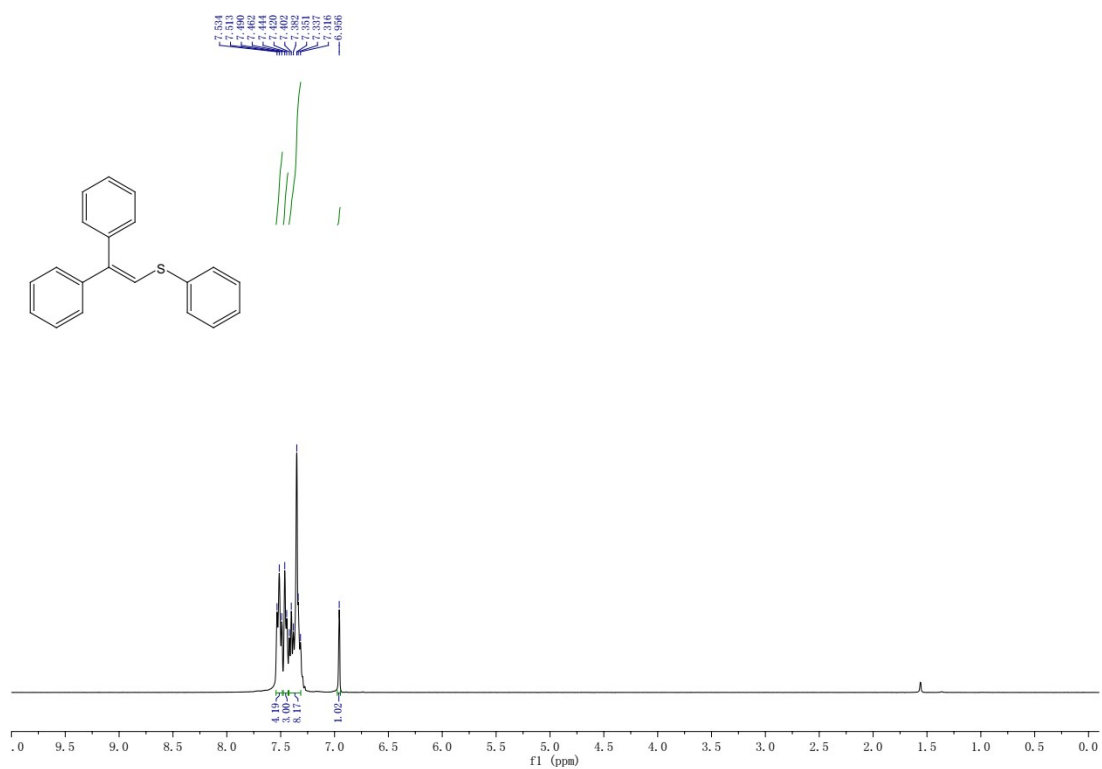
^1H NMR spectrum for compound 5 (CDCl_3 , 400 MHz)



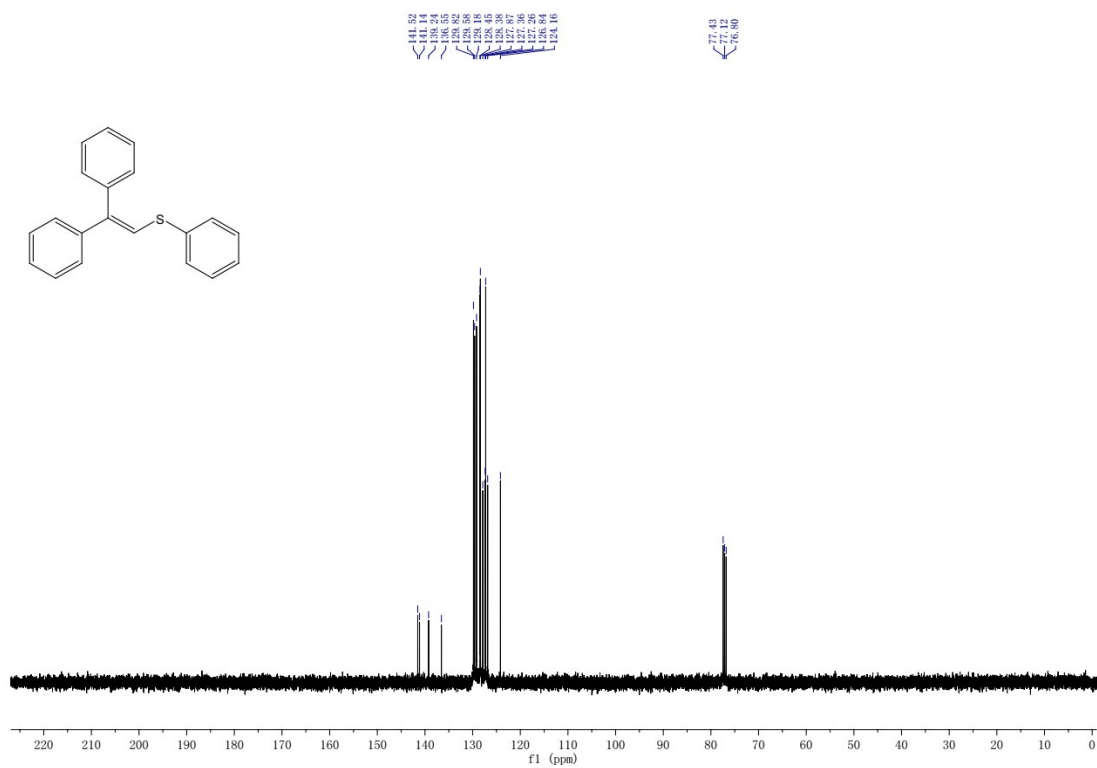
¹³C NMR spectrum for compound 5 (CDCl₃, 101 MHz)



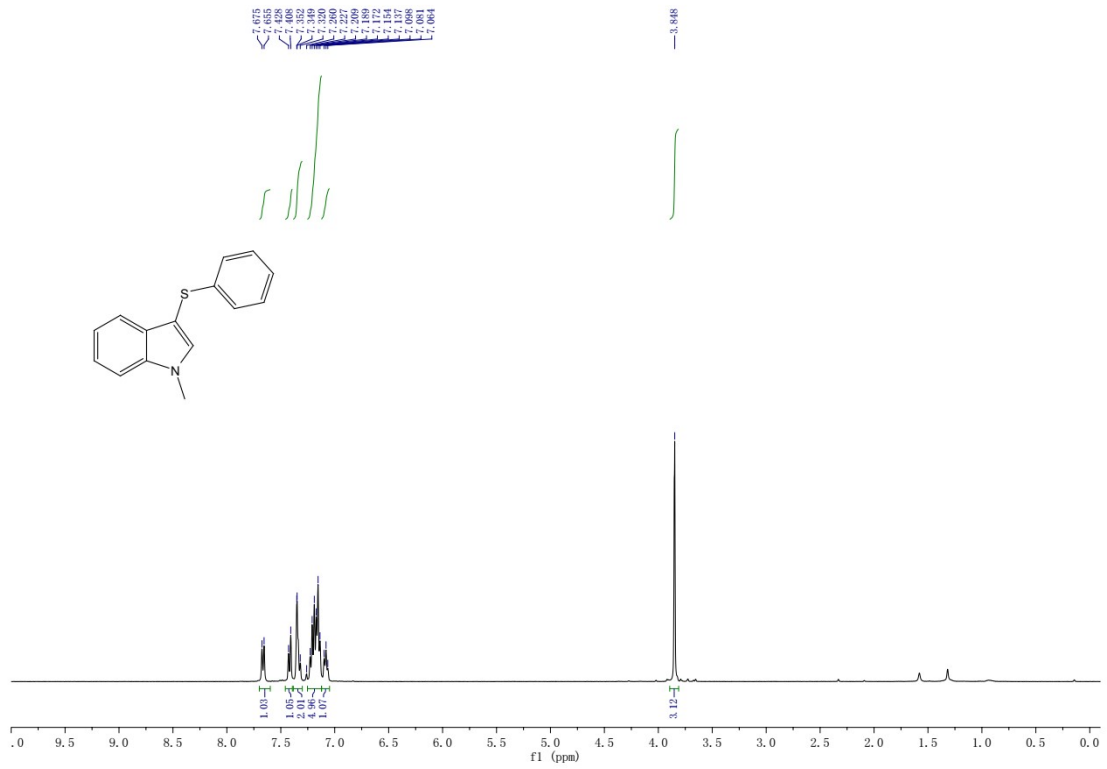
¹H NMR spectrum for compound 6 (CDCl₃, 400 MHz)



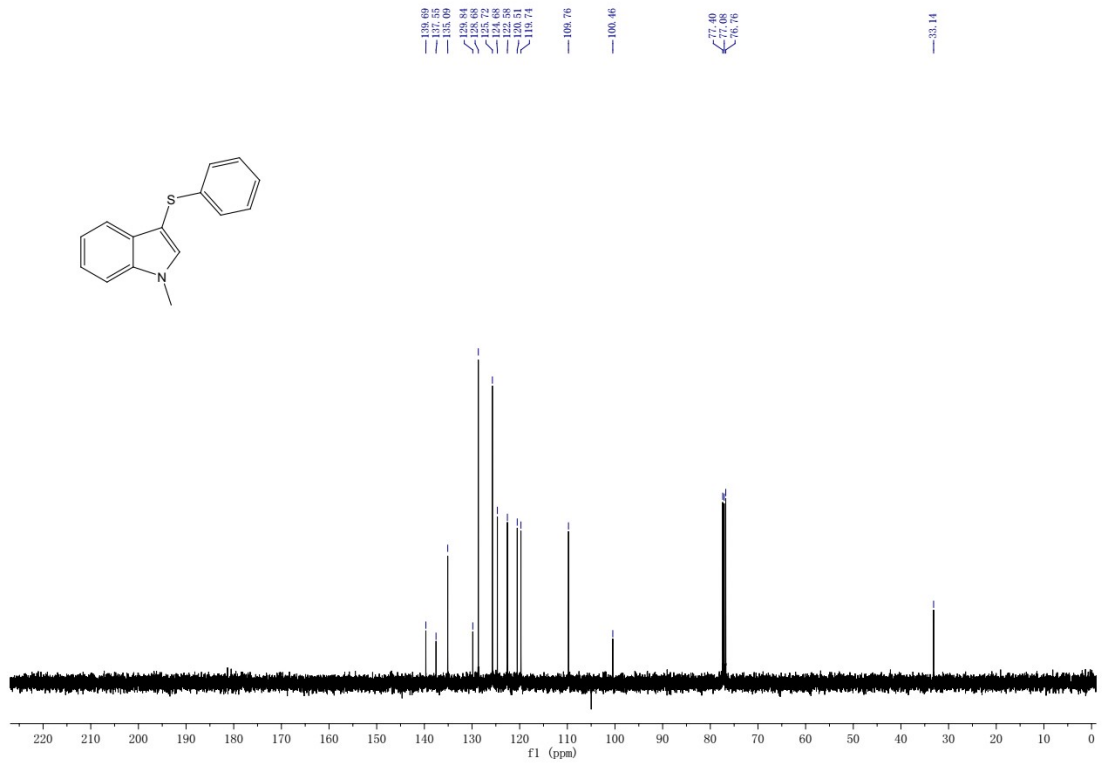
¹³C NMR spectrum for compound 6 (CDCl₃, 101 MHz)



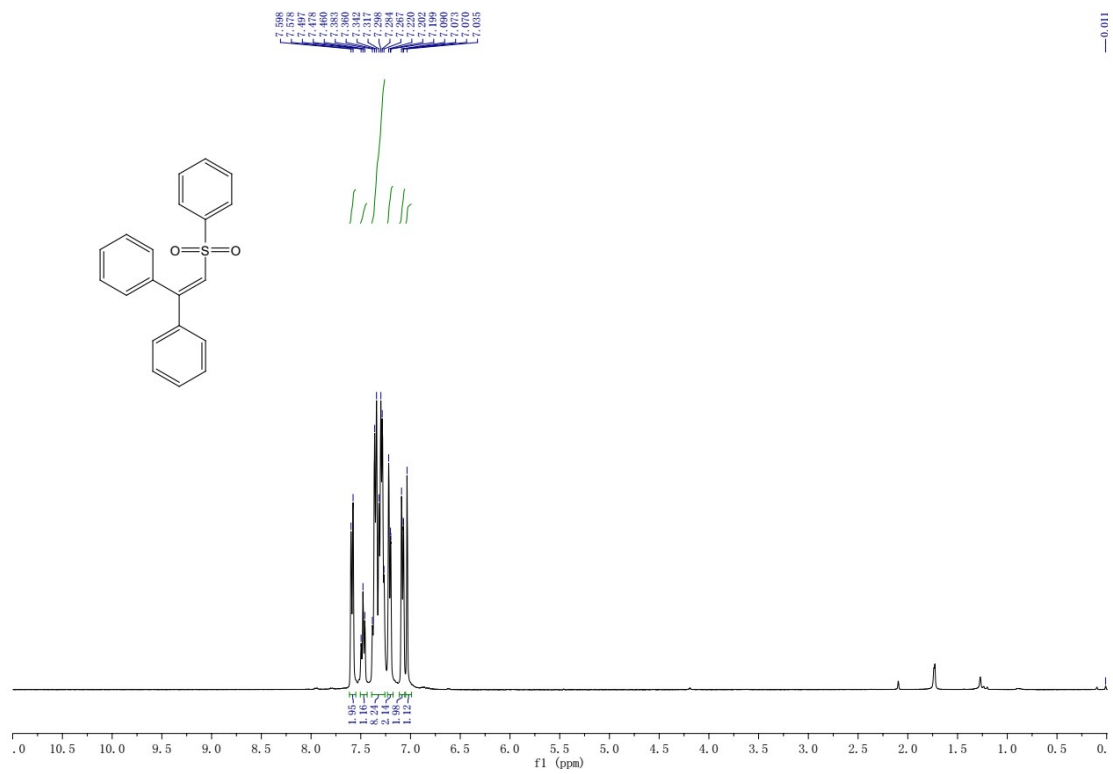
¹H NMR spectrum for compound 7 (CDCl₃, 400 MHz)



¹³C NMR spectrum for compound **7** (CDCl₃, 101 MHz)



¹H NMR(400 MHz, CDCl₃) for compound **8**



¹³C NMR(101 MHz, CDCl₃) for compound 8

