

Supporting information

Solid-State Molecular Oxygen Activation Using Ball Milling and Piezoelectric Material for Aerobic Oxidation of Thiols

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General Information

All starting materials were purchased from commercial sources. ^1H NMR and ^{13}C NMR were recorded on a 400 MHz Bruker spectrometer (400 MHz for ^1H NMR and 100 MHz for ^{13}C NMR). Chemical shifts (δ) of ^1H NMR and ^{13}C NMR are reported in ppm relative to TMS and the residual solvent peak were converted to the TMS scale (CDCl_3 : $\delta\text{H} = 7.26$ ppm, $\delta\text{C} = 77.16$ ppm, H_2O : δH : 7.26; DMSO-d_6 : $\delta\text{H} = 2.50$ ppm, $\delta\text{C} = 39.52$ ppm, H_2O : δH : 3.33). The coupling constants (J) are in Hertz (Hz). The used abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), ddd (doublet of doublet of doublets), dt (doublet of triplets), td (triplet of doublets), tt (triplet of triplets), qd (a quartet of doublets), m (multiplet). The high-resolution mass spectra (HRMS) data were measured on a UHPLC Q-TOF HR-MS using the ESI technique, with accurate masses reported for the molecular ion ($[\text{M}]^+$). The low-resolution mass spectra (LRMS) data were measured on the SHIMADZU GCMS-QP 2010 SE mass spectrometer (Kyoto, Japan) by means of the EI technique. The melting points of these compounds were determined by an X-4A micro-melting point apparatus (Shanghai, China). All chemical reactions were carried out in an MSK-SFM-12M-22V ball mill (produced by HE FEI KE JING materials technology company) The reaction was monitored by thin-layer chromatography (TLC) and GCMS. Gram-scale aerobic oxidative coupling of **1a** was conducted in an AM100S mixer mill (Ants Scientific Instruments). GCMS basic information: The temperature of oven: 70°C , Inlet temperature: 310°C , Ion source temperature: 200°C , Interface temperature: 250°C , Sample way: Shunt, Injector pressure: 100 kPa . All SEM images were taken using a Zeiss Sigma 500 SEM. X-ray diffraction (XRD) patterns were obtained using an X-ray diffractometer (PANalytical, X'Pert PRO).

Typical Procedure for the Cross-coupling reaction of all thiols

A 2 mL PE milling jar equipped with five 4 mm stainless steel balls was charged with **1** (0.2 mmol) and BaTiO_3 ($< 3\ \mu\text{m}$) (450 mg, 1.93 mmol). After closing the jar tightly, the mechanochemical reaction was carried out for 6 mins at 3800 rpm. After the reaction was completed, the powdery mixture was washed out with dichloromethane, centrifuged and concentrated under reduced pressure to get the pure product (**2a-s**, **2w-z**). In the case of compounds **2t-v**, the concentrated filtrate was further purified by silica gel chromatography.

Procedure for the recycling of the piezoelectric catalyst BaTiO_3

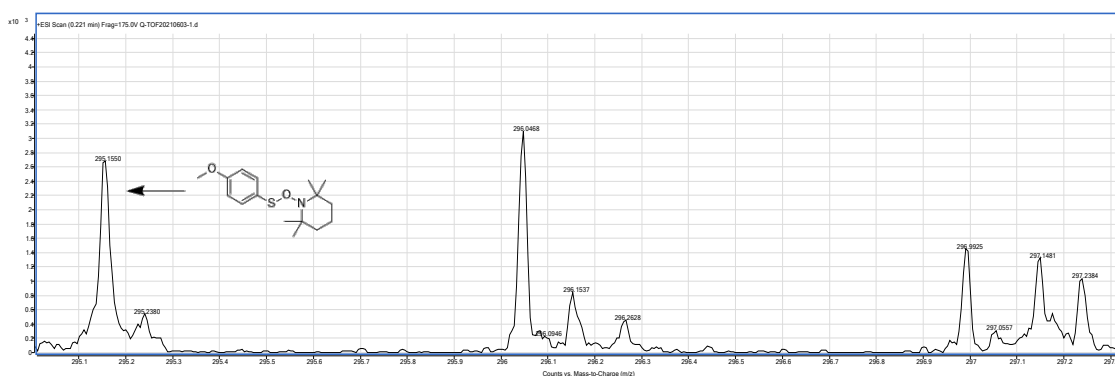
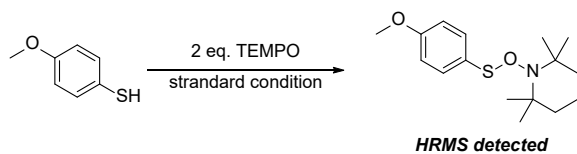
A 2 mL PE steel milling jar equipped with 5 stainless steel balls of 4 mm in diameter was charged with 4-methoxybenzenethiol **1a** (0.2 mmol, 1.0 equiv.), and BaTiO_3 ($< 3\ \mu\text{m}$) (450 mg, 1.93 mmol). The mechanochemical reaction was carried out for 6 min at 3800 rpm. After the reaction completed, the reaction mixture was washed with dichloromethane and filtered. The filtrate was concentrated under reduced pressure to get the pure product. The BaTiO_3 was collected and dried in the oven at 80°C for 1 h. The recycled BaTiO_3 was used for the next run by adding a fresh batch of **1a** under the same reaction condition. The BaTiO_3 was used for the 30 runs. The product yields of 30 runs are as following: $>99\%$, 97% , 93% , 90% , 82% , 73% , 65% , 60% , 56% , 50% .

Free radical trapping experiments

1. Free radical trapping of **1a** with TEMPO

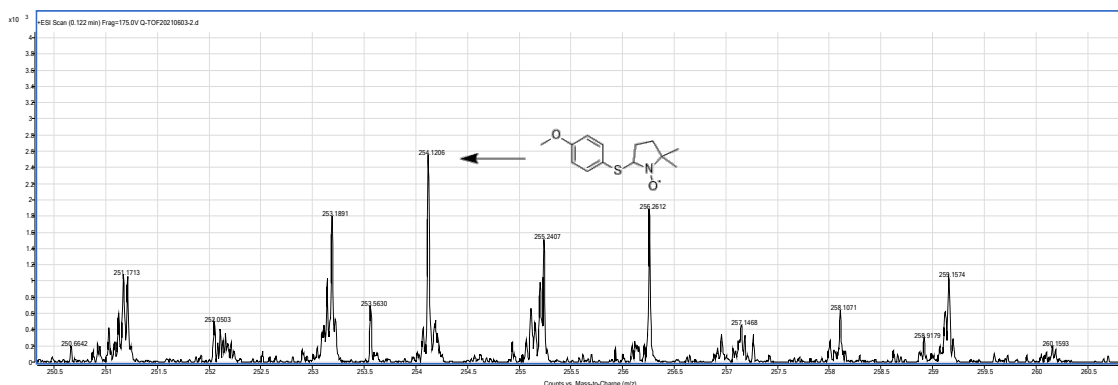
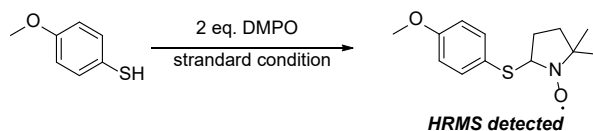
To a 2 mL PE milling jar was added **1a** (0.2 mmol), BaTiO_3 (450 mg, 1.93 mmol), TEMPO (62.4

mg, 0.4 mmol). The reaction was carried out at 3800 rpm for 6 min. The reaction mixture was extracted with dichloromethane (2 mL), and analyzed by HRMS analysis. HRMS (ESI) m/z calcd for $C_{16}H_{25}NO_2S$ ($[M]^+$): 295.1601; found, 295.1550. The HRMS result suggested that a thiyl radical might be involved in this transformation.



2. Free radical trapping of **1a** with DMPO

To a 2 mL PE milling jar was added **1a** (0.2 mmol), $BaTiO_3$ (450 mg, 1.93 mmol), DMPO (45.2mg, 0.4 mmol). The reaction was carried out at 3800 rpm for 6 min. The reaction mixture was extracted with dichloromethane (2 mL) and analyzed by HRMS analysis. HRMS (ESI) m/z calcd for $C_{13}H_{18}NO_2S$ ($[M]^+$): 254.1209; found, 254.1206. The HRMS result suggested that a thiyl radical might be involved in this transformation.



Gram-scale aerobic oxidative coupling of **1a**

A 10 mL PE steel milling jar equipped with five 5 mm stainless steel balls, 4-methoxybenzenethiol **1a** (3 mmol), and $BaTiO_3$ ($< 3 \mu m$) (6.6 g, 28.5 mmol). After closing the jar tightly, the mechanochemical reaction was carried out for 20 mins at 2100 rpm. During the reaction, we opened the mill jar every five minutes to ensure there was enough molecular oxygen for the reaction. After the reaction was completed, the reaction mixture was washed with dichloromethane (10×5 mL) and filtered. The filtrate was

concentrated under reduced pressure to get the product.

SEM analysis of the piezoelectric catalyst

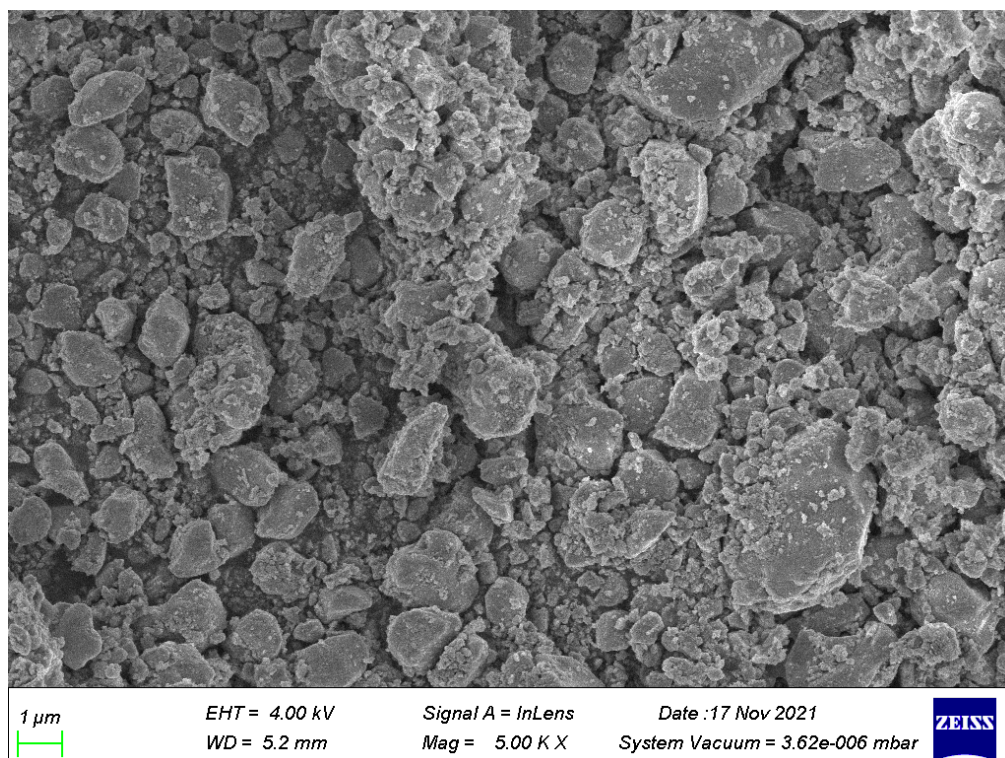


Figure S1. SEM image of commercially available BaTiO_3 (< 3 μm) before use.

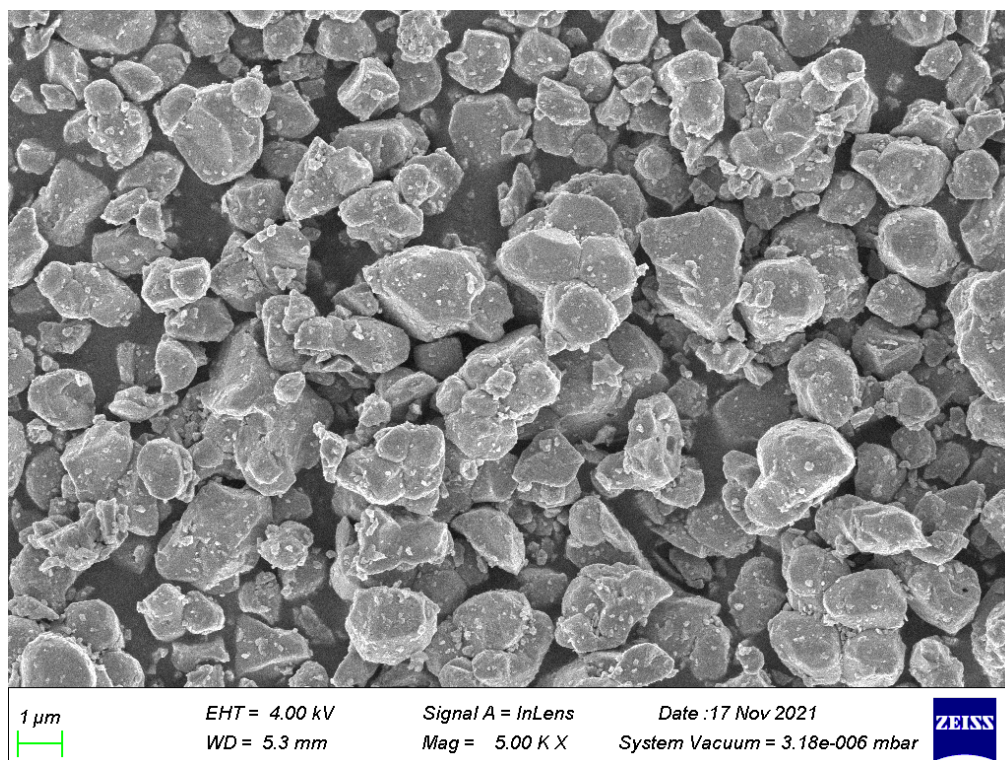


Figure S2. SEM image of BaTiO_3 (< 3 μm) under ball milling for aerobic oxidative coupling of **1a** recovered from the 15th cycle.

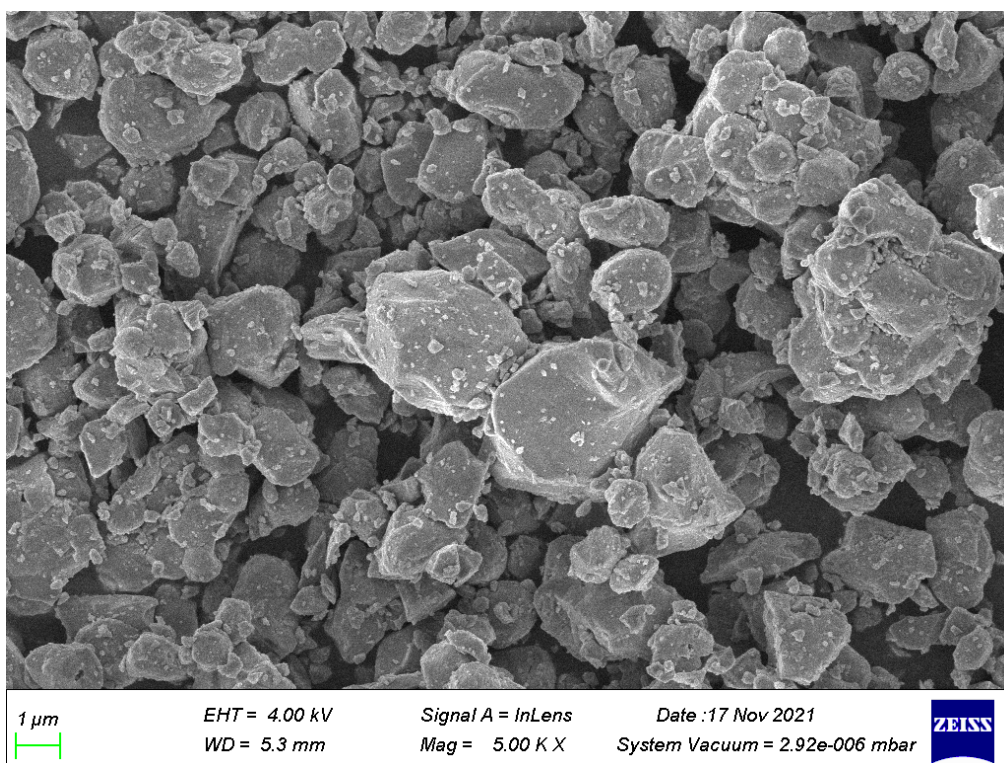


Figure S3. SEM image of BaTiO₃ (< 3 μm) under ball milling for aerobic oxidative coupling of **1a** recovered from the 24th cycle.

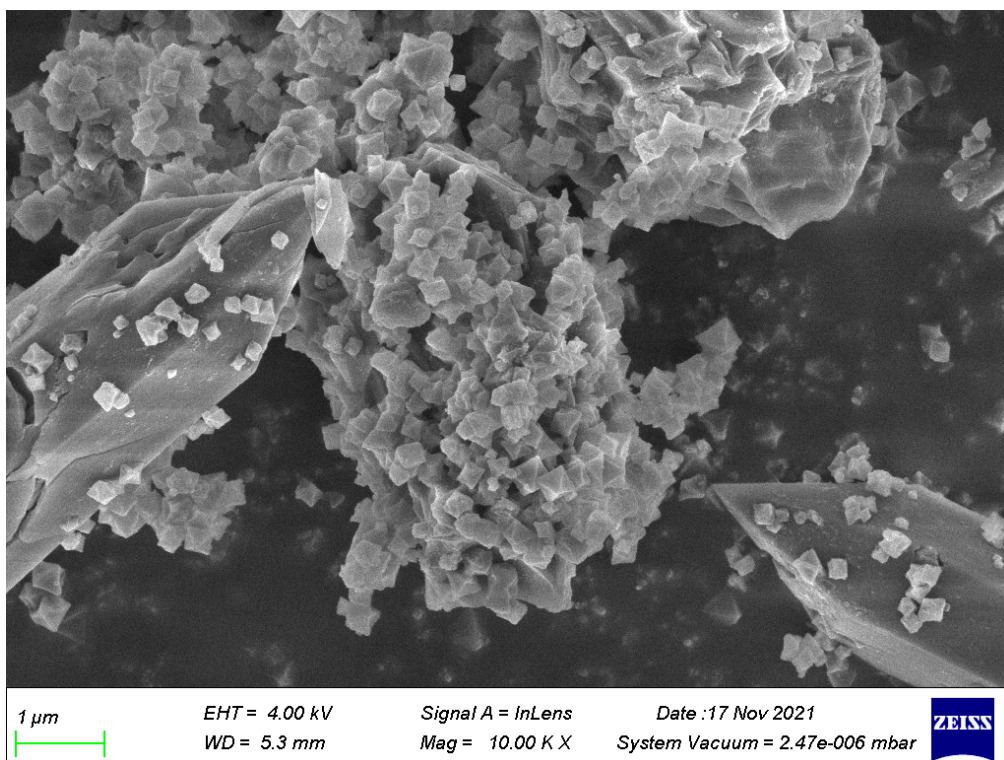


Figure S4. SEM image of BaTiO₃ (< 3 μm) under ball milling for aerobic oxidative coupling of **1a** recovered from the 30th cycle.

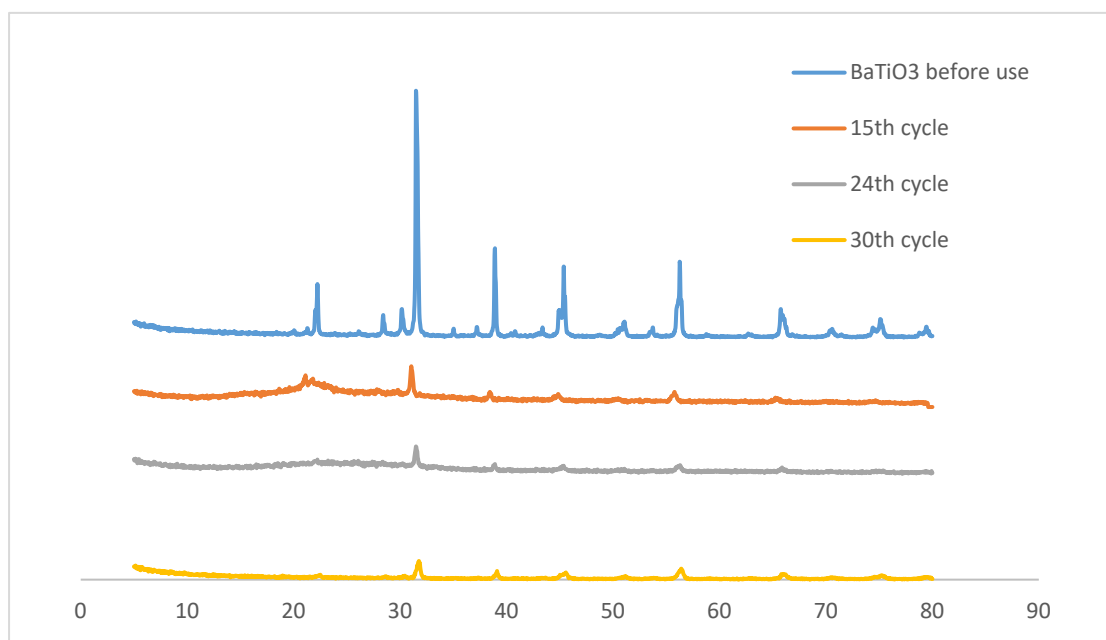
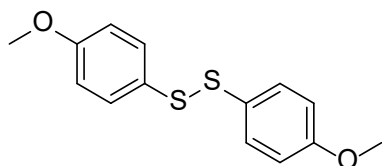
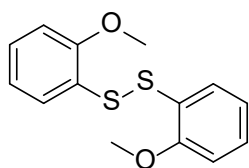


Figure S5. XRD analysis of BaTiO₃ (< 3 um) under ball milling for aerobic oxidative coupling of **1a** before use, recovered from the 15th cycle, the 24th cycle, and the 30th cycle.

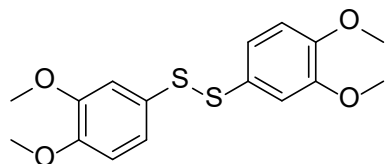
Characterization Data of all products



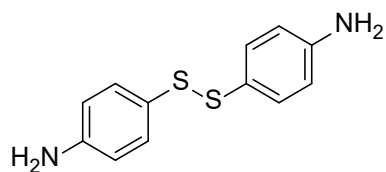
1,2-bis(4-methoxyphenyl)disulfane (2a)¹ Yellow oil (27.8 mg, > 99%), (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.39 (s, 4H), 6.83 (d, *J* = 8.9 Hz, 4H), 3.80 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 159.93, 132.67, 128.46, 114.63, 55.38. LRMS (EI): *m/z* calcd for C₁₄H₁₄O₂S₂([M]⁺): 278.31; found 277.90.



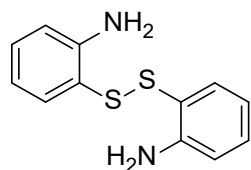
1,2-bis(2-methoxyphenyl)disulfane (2b)¹ White solid (27.8 mg, >99%), (hexane as TLC eluent). mp: 115-116 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.53 (dd, *J* = 7.8, 1.6 Hz, 2H), 7.18 (ddd, *J* = 8.1, 7.4, 1.6 Hz, 2H), 6.91 (td, *J* = 7.6, 1.2 Hz, 2H), 6.85 (dd, *J* = 8.2, 1.2 Hz, 2H), 3.90 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 156.62, 127.76, 127.63, 124.63, 121.36, 110.52, 55.91. LRMS (EI): *m/z* calcd for C₁₄H₁₄O₂S₂([M]⁺): 278.04; found 278.15.



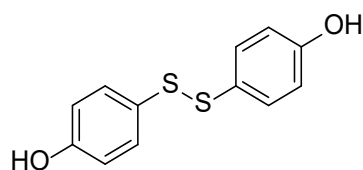
1,2-bis(3,4-dimethoxyphenyl)disulfane (2c)² Light yellow solid (33.8 mg, >99%), (hexane as TLC eluent). mp: 79–80 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.09–6.98 (m, 4H), 6.79 (d, *J* = 8.3 Hz, 2H), 3.87 (s, 6H), 3.83 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 149.5, 149.1, 128.7, 123.9, 114.1, 111.3, 55.9 (d, *J* = 5.9 Hz). LRMS (EI) *m/z* calcd for C₁₆H₁₈O₄S₂ ([M]⁺): 338.06, found 338.10.



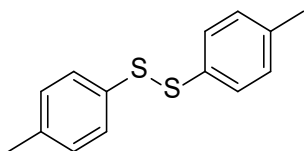
4,4'-disulfanediyldianiline (2d)^{1,3} Yellow solid (24.8 mg, >99%), (hexane as TLC eluent). mp: 74–75 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.26 (dd, *J* = 6.3, 2.2 Hz, 4H), 6.59 (d, *J* = 8.5 Hz, 4H), 3.78 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 147.0, 133.9, 125.8, 115.3. LRMS (EI) *m/z* calcd for C₁₂H₁₂N₂S₂ ([M]⁺): 248.04, found 247.85.



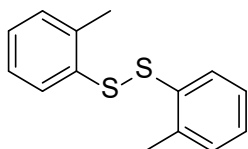
2,2'-disulfanediyldianiline (2e)³ Yellow solid (24.8 mg, >99%), (hexane as TLC eluent). mp: 90–91 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.22–7.09 (m, 4H), 6.71 (dd, *J* = 8.4, 1.3 Hz, 2H), 6.58 (td, *J* = 7.5, 1.3 Hz, 2H), 4.32 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 148.6, 136.8, 131.6, 118.7, 118.2, 115.2. LRMS (EI) calcd for C₁₂H₁₂N₂S₂ ([M]⁺): 248.04, found 247.95.



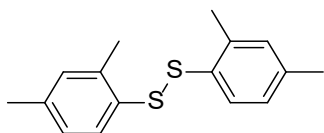
4,4'-disulfanediyldiphenol (2f)⁴ White solid (25mg, >99%), (hexane as TLC eluent). mp: 144–146 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.83 (s, 2H), 7.27 (d, *J* = 8.6 Hz, 4H), 6.76 (d, *J* = 8.7 Hz, 4H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 158.3, 133.0, 125.1, 116.3. LRMS (EI) *m/z* calcd for C₁₂H₁₀O₂S₂ ([M]⁺): 250.01, found 250.34.



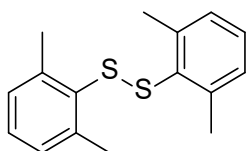
1,2-di-p-tolyldisulfane (2g)⁵ Colorless solid (24.6 mg, >99%), (hexane as TLC eluent). mp: 44-46 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.30 (d, *J* = 8.2 Hz, 4H), 7.07–6.92 (m, 4H), 2.24 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 137.4, 133.9, 129.8, 128.5, 21.1. LRMS (EI) *m/z* calcd for C₁₄H₁₄S₂ ([M]⁺): 246.05, found 246.15.



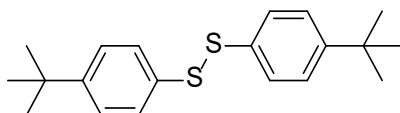
1,2-di-o-tolyldisulfane (2h)³ White solid (24.6 mg, >99%), (hexane as TLC eluent). mp: 38-39 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.57–7.45 (m, 2H), 7.20–7.08 (m, 6H), 2.43 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 137.3, 135.4, 130.3, 128.5, 127.3, 126.7, 20.0. LRMS (EI) *m/z* calcd for C₁₄H₁₄S₂ ([M]⁺): 246.05, found 246.05.



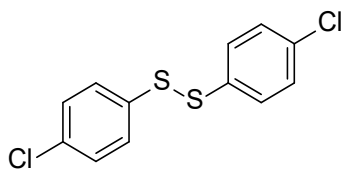
1,2-bis(2,4-dimethylphenyl)disulfane (2i)^{6, 7} Yellow oil (27.4 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, *J* = 7.9 Hz, 2H), 7.04–6.98 (m, 2H), 6.93 (dd, *J* = 7.9, 2.0 Hz, 2H), 2.37 (s, 6H), 2.29 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 138.3, 137.8, 132.3, 131.2, 130.5, 127.3, 20.9, 20.2. LRMS (EI): *m/z* calcd for C₁₆H₁₈S₂ ([M]⁺): 274.08, found 274.10.



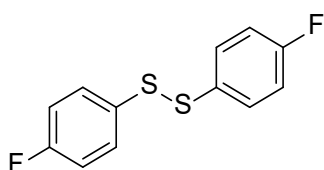
1,2-bis(2,6-dimethylphenyl)disulfane (2j)⁸ Colorless oil (27.4 mg, >99%), (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.10 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.01 (d, *J* = 7.2 Hz, 4H), 2.23 (s, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 143.5, 134.8, 129.4, 128.1, 21.5. LRMS (EI) *m/z* calcd for C₁₆H₁₈S₂ ([M]⁺): 274.08, found 274.20.



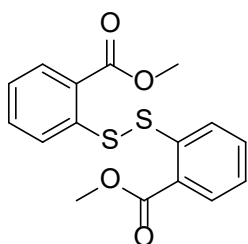
1,2-bis(4-tert-butylphenyl)disulfane (2k)⁹ White solid (33 mg, >99%), (hexane as TLC eluent). mp: 70–72 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.52–7.40 (m, 4H), 7.37–7.28 (m, 4H), 1.29 (s, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 150.5, 134.0, 127.7, 126.1, 34.5, 31.3. LRMS (EI) *m/z* calcd for C₂₀H₂₆S₂ ([M]⁺): 330.15, found 330.25.



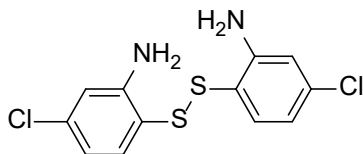
1,2-bis(4-chlorophenyl)disulfane (2l)⁵ White solid, (28.5 mg, >99%), (hexane as TLC eluent). mp: 60-63 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.51–7.42 (m, 4H), 7.37–7.29 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 135.15, 133.67, 129.36, 129.32. LRMS (EI): m/z calcd for C₁₂H₈S₂Cl₂ ([M]⁺): 285.94, found 286.05.



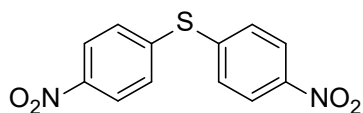
1,2-bis(4-fluorophenyl)disulfane(2m)¹² Colorless oil (25.4 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.55 (dd, J = 10.4, 6.8 Hz, 4H), 7.23 (t, J = 10.5 Hz, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 161.40, 132.17, 131.35, 116.40. LRMS (EI) m/z calcd for C₁₂H₈F₂S₂ ([M]⁺): 254.00, found 253.90.



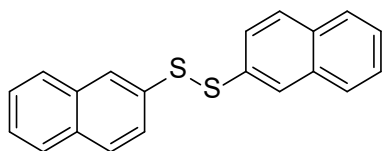
dimethyl 2,2'-disulfanediyl dibenzoate(2n)¹³ White solid (22.7 mg, 68% yield), (hexane : dichloromethane = 5:1 as eluent). mp:126-128 °C. ¹H NMR (400 MHz, DMSO-d₆) δ 8.05 (dd, J = 7.8, 1.4 Hz, 2H), 7.69 – 7.54 (m, 4H), 7.38 (m, 2H), 3.93 (s, 6H). ¹³C NMR (100 MHz, DMSO-d₆) δ 166.0, 138.6, 133.7, 131.4, 126.9, 126.3, 125.2, 52.5. LRMS (EI) m/z calcd for C₁₆H₁₄O₄S₂ ([M]⁺): 334.03, found 334.10.



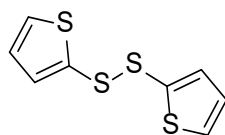
6,6'-disulfanediylbis(3-chloroaniline)(2o)¹⁴ Yellow solid (31.7 mg, >99%), (hexane as TLC eluent). mp: 76-78 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.03 (d, J = 8.2 Hz, 2H), 6.71 (d, J = 2.2 Hz, 2H), 6.56 (dd, J = 8.2, 2.2 Hz, 2H), 4.41 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 149.4, 137.8, 118.3, 116.6, 114.7. LRMS (EI) calcd for C₁₂H₁₂Cl₂N₂S₂ ([M+2H]⁺): 317.97, found 318.10.



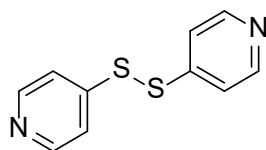
bis(4-nitrophenyl)sulfane(2p)^{10, 11} White solid (21.5 mg, 78%), (hexane as eluent). mp: 161-163 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 8.9 Hz, 4H), 7.62 (d, *J* = 8.9 Hz, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 147.0, 144.0, 126.4, 124.4. LRMS (EI) *m/z* calcd for C₁₂H₈N₂O₄S([M]⁺): 276.02, found 276.10.



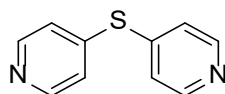
1,2-di(naphthalen-2-yl)disulfane(2q)¹⁵ Pale yellow solid (31.8 mg, >99%), (hexane as TLC eluent). mp: 141-142 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, *J* = 1.9 Hz, 2H), 7.79 (dd, *J* = 9.0, 2.8 Hz, 4H), 7.76-7.70 (m, 2H), 7.62 (dd, *J* = 8.7, 1.9 Hz, 2H), 7.52-7.35 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 134.3, 133.5, 132.5, 129.0, 127.7, 127.4, 126.7, 126.6, 126.2, 125.7. LRMS (EI) *m/z* calcd for C₂₀H₁₄S₂([M]⁺): 318.05, found 318.46.



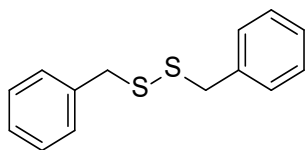
1,2-di(thiophen-2-yl)disulfane (2r)¹⁶ Yellow oil (22.9 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.51 (s, 2H), 7.16 (dd, *J* = 3.6, 1.3 Hz, 2H), 7.02 (dd, *J* = 5.3, 3.6 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 135.75, 135.67, 132.30, 127.78. LRMS (EI) *m/z* calcd for C₈H₆S₄ ([M]⁺): 230.94, found 230.00.



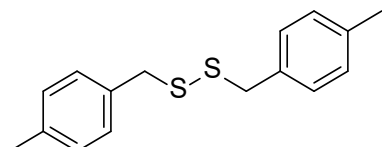
1,2-di(pyridin-4-yl)disulfane (2s)^{3, 17} Yellow oil (39 % yield). (hexane : ethyl acetate =3:1 as eluent) ¹H NMR (400 MHz, CDCl₃) δ 8.56-8.53 (m, 4H), 7.26-7.23 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 149.9, 144.0, 124.8. LRMS (EI) *m/z* calcd for C₁₀H₈N₂S₂ ([M]⁺): 220.01, found 219.95.



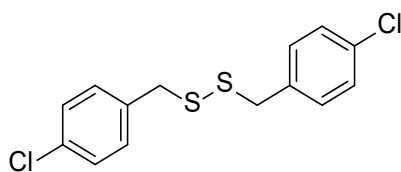
di(pyridin-4-yl)sulfane(2s') Yellow oil (61% yield). (hexane : ethyl acetate =3:1 as eluent). ¹H NMR (400 MHz, CDCl₃) δ 8.51-8.47 (m, 4H), 7.39-7.34 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 149.9, 146.5, 120.1. LRMS (EI) *m/z* calcd for C₁₀H₈N₂S ([M]⁺): 188.04, found 188.10.



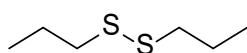
1,2-dibenzyl disulfane(2t)³ Yellow oil (17.2 mg, 70% yield), (hexane : dichloromethane = 5:1 as eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.38–7.16 (m, 10H), 3.60 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 137.3, 129.4, 128.5, 127.4, 43.3. LRMS (EI) m/z calcd for C₁₄H₁₄S₂ ([M]⁺): 246.05, found 246.05.



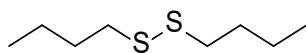
1,2-bis(4-methylbenzyl) disulfane(2u)¹⁸ Colorless solid, isolated yield (17mg, 70% yield), (hexane : dichloromethane = 5:1 as eluent). ¹H NMR (400 MHz, CDCl₃) δ 7.14 (s, 8H), 3.61 (s, 4H), 2.34 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 137.1, 134.2, 129.2, 43.0, 21.1. LRMS (EI) m/z calcd for C₁₆H₁₈S₂ ([M]⁺): 274.08, found 273.95.



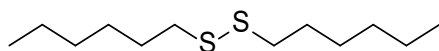
Bis(4-chlorobenzyl) disulfide(2v)⁷ white solid, isolated yield (22.2 mg, 71% yield), (hexane : dichloromethane = 5:1 as eluent). mp: 62-63 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.30–7.28 (m, 4H), 7.16–7.14 (m, 4H), 3.57 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 135.8, 133.4, 130.6, 128.7, 42.5. LRMS (EI) calcd for C₁₄H₁₂S₂Cl₂ ([M]⁺): 313.98, found 314.00.



1,2-dipropyl disulfane(2w)¹⁸ Colorless oil (15 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 2.67 (dd, J = 7.7, 6.8 Hz, 4H), 1.71 (h, J = 7.3 Hz, 4H), 1.00 (t, J = 7.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 41.2, 22.5, 13.1. LRMS (EI) calcd for C₆H₁₄S₂ ([M]⁺): 150.05, found 150.05.

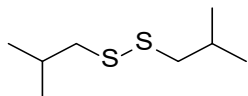


1,2-dibutyl disulfane(2x)¹⁹ Colorless oil (17.8 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 2.85 – 2.54 (m, 4H), 1.66 (tt, J = 7.6, 6.3 Hz, 8H), 1.42 (q, J = 7.5 Hz, 6H), 0.92 (t, J = 7.3 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 38.9, 31.3, 21.6, 13.6. LRMS (EI) calcd for C₈H₁₈S₂ ([M]⁺): 178.08, found 178.05.



1,2-dihexyl disulfane(2y)²⁰ Colorless oil (23.4 mg, >99%). (hexane as TLC eluent). ¹H NMR (400

MHz, CDCl₃) δ 2.76–2.51 (m, 4H), 1.76–1.61 (m, 4H), 1.43–1.21 (m, 12H), 0.95–0.85 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 39.2, 31.4, 29.2, 28.2, 22.5, 14.0. LRMS (EI) calcd for C₁₂H₂₆S₂([M]⁺): 234.15, found 234.10.



1,2-diisobutyldisulfane(2z)²¹ Colorless oil (17.8 mg, >99%). (hexane as TLC eluent). ¹H NMR (400 MHz, CDCl₃) δ 2.59 (d, *J* = 6.9 Hz, 4H), 1.94 (dp, *J* = 13.4, 6.7 Hz, 2H), 1.00 (d, *J* = 6.7 Hz, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 48.5, 28.1, 21.7. LRMS (EI) *m/z* calcd for C₈H₁₈S₂ ([M]⁺): 178.08, found 178.05.

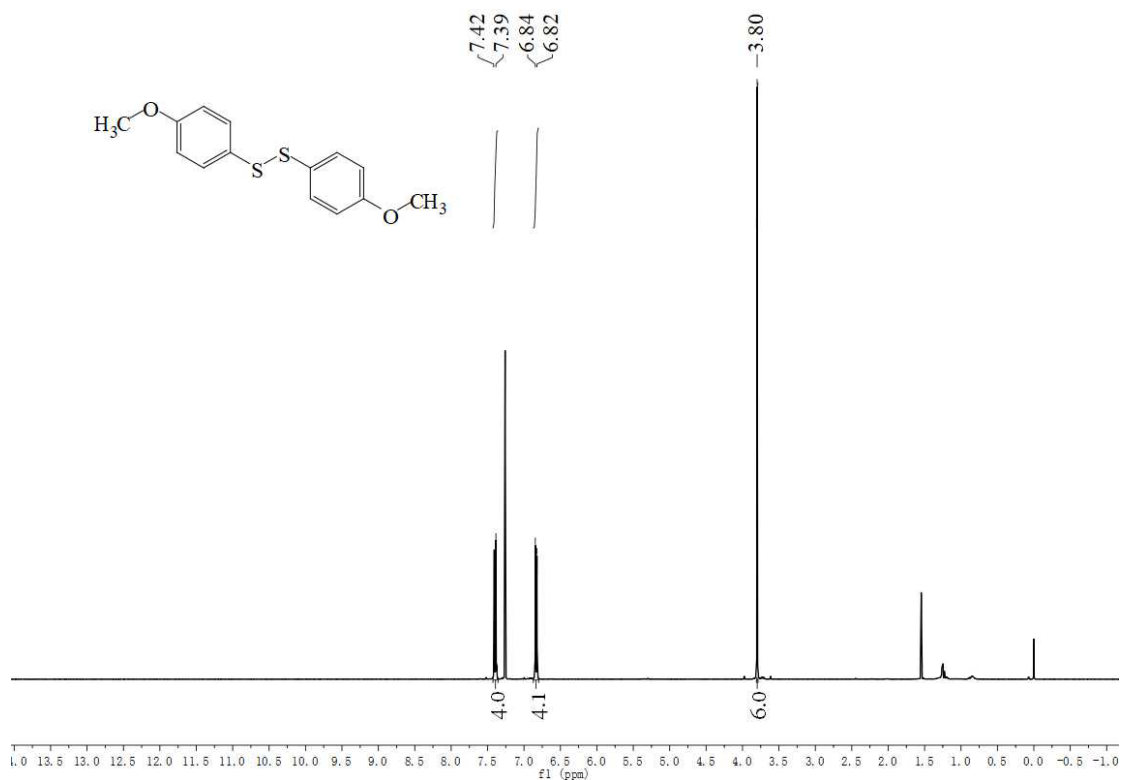
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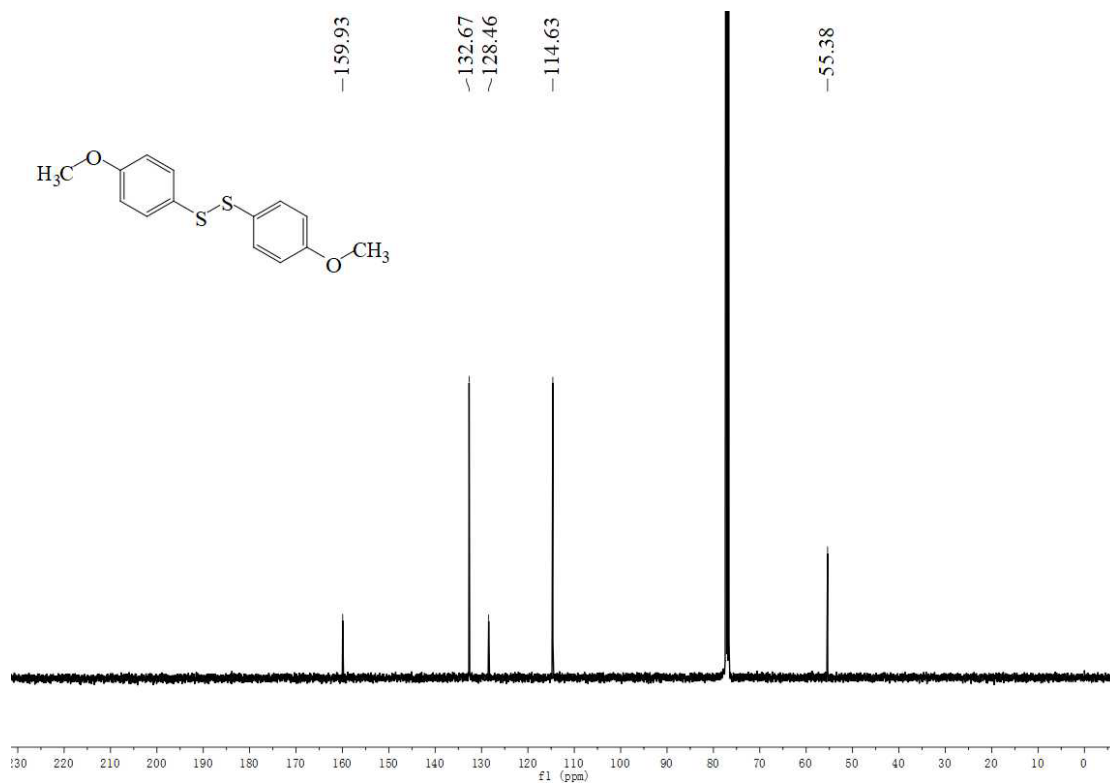
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The NMR Spectra of all products

1,2-bis(4-methoxyphenyl)disulfane (**2a**)

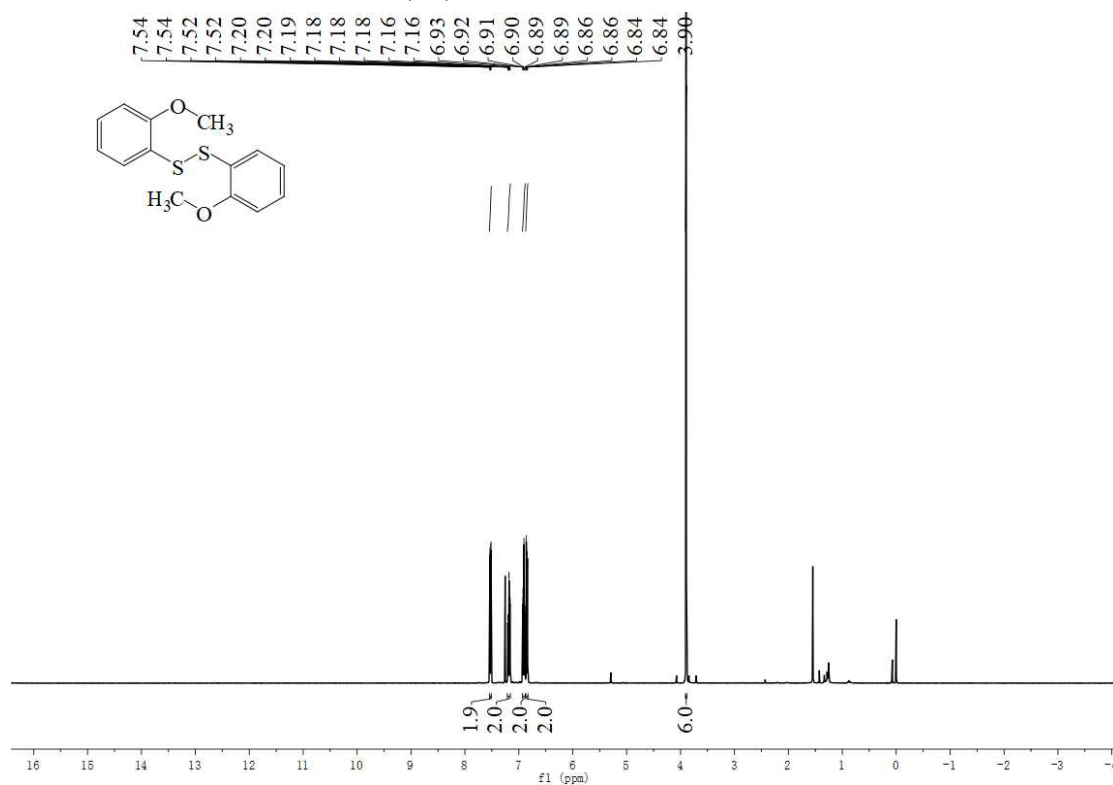


¹H NMR (CDCl₃, 400 MHz) of **2a**

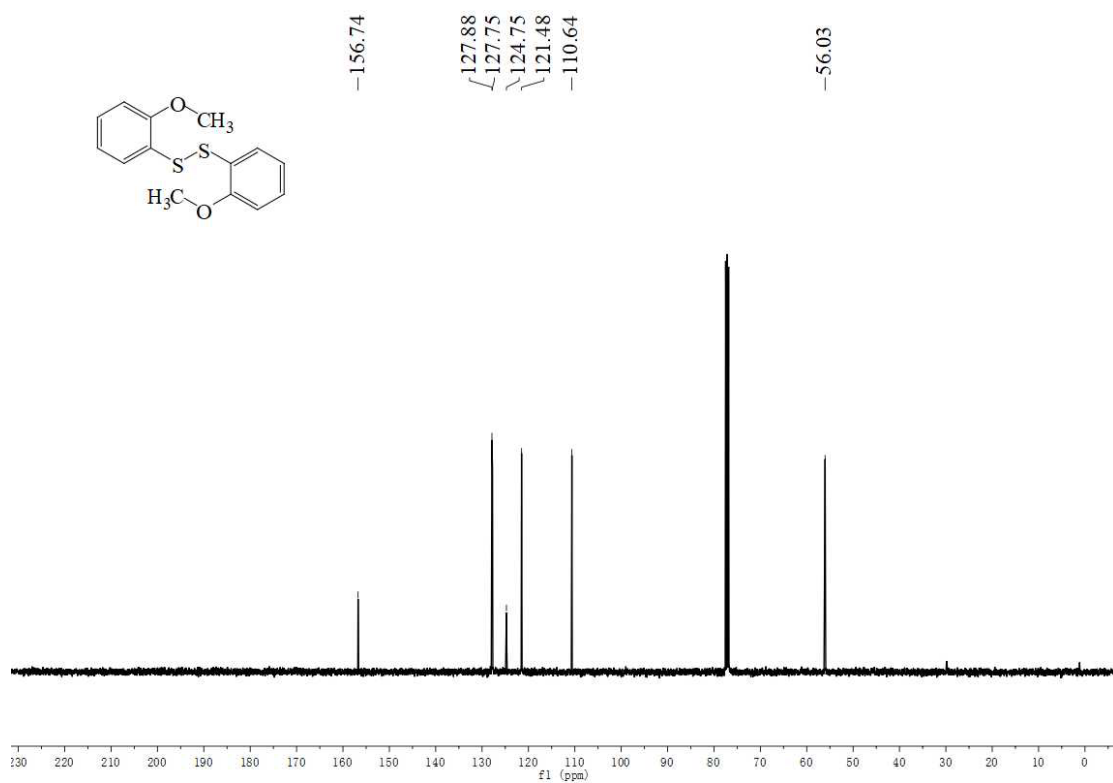


¹³C NMR (CDCl₃, 100 MHz) of **2a**

1,2-bis(2-methoxyphenyl)disulfane (2b)

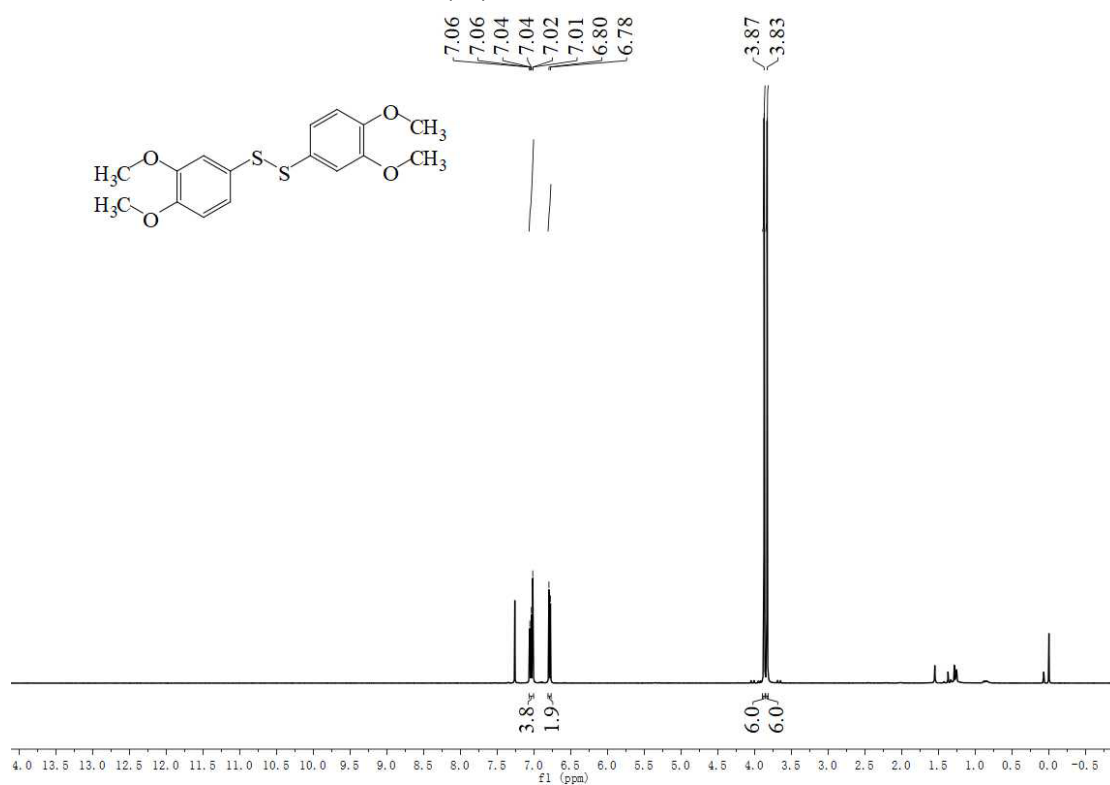


¹H NMR (CDCl₃, 400 MHz) of **2b**

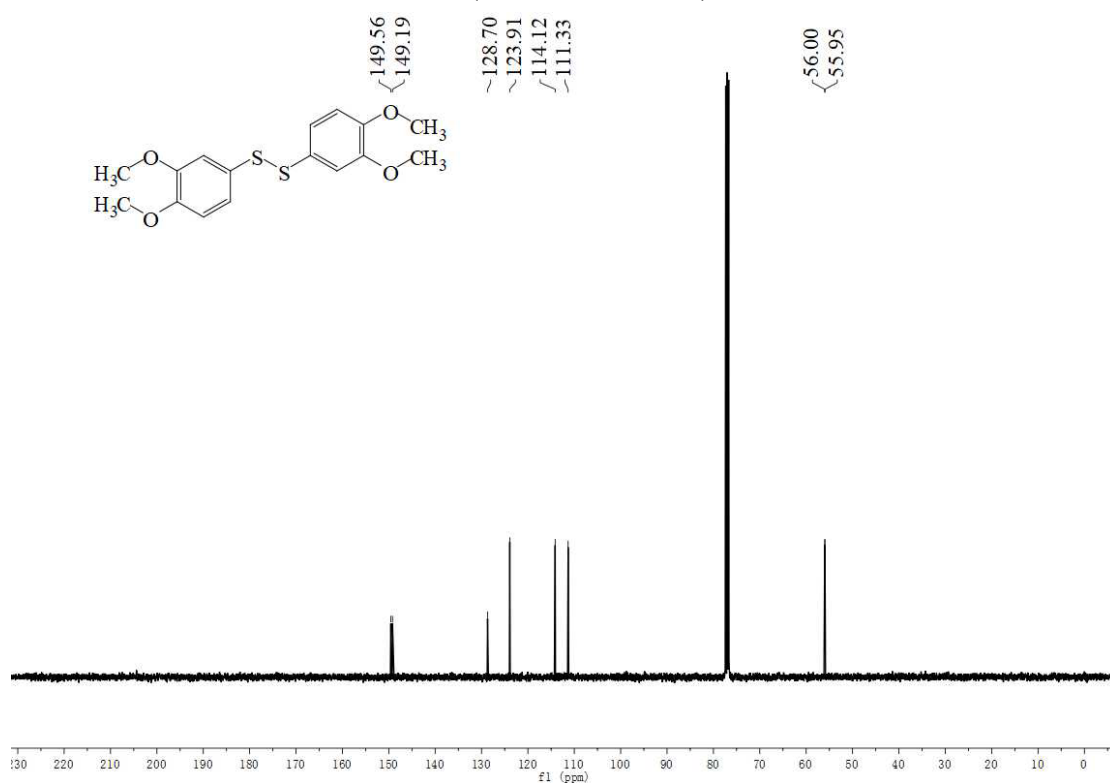


¹³C NMR (CDCl₃, 100 MHz) of **2b**

1,2-bis(3,4-dimethoxyphenyl)disulfane (2c)

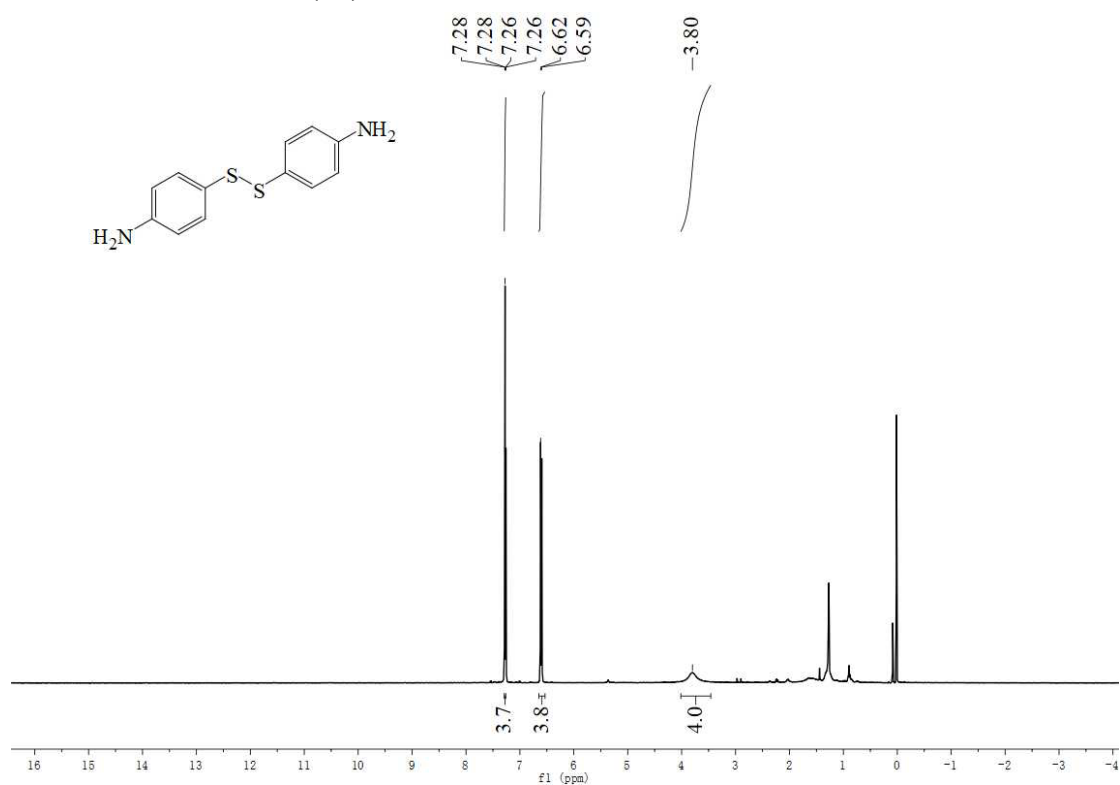


¹H NMR (CDCl₃, 400 MHz) of 2c

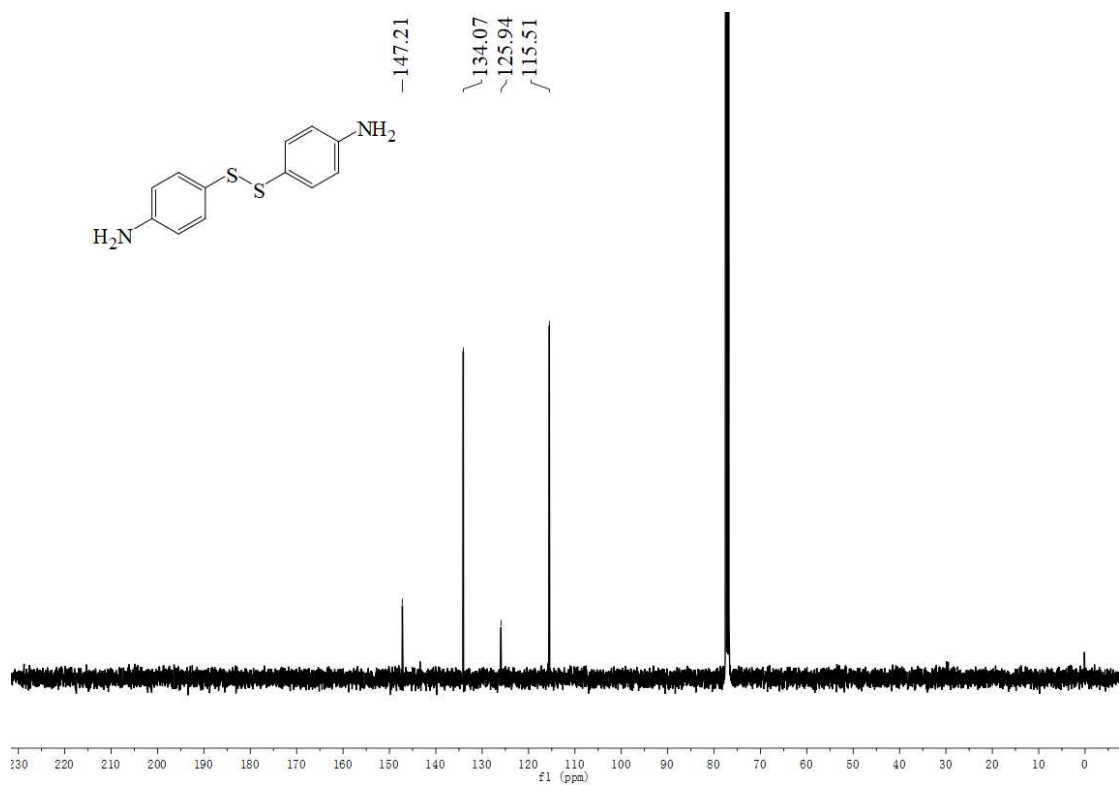


¹³C NMR (CDCl₃, 100 MHz) of 2c

4,4'-disulfaneyldianiline (2d)

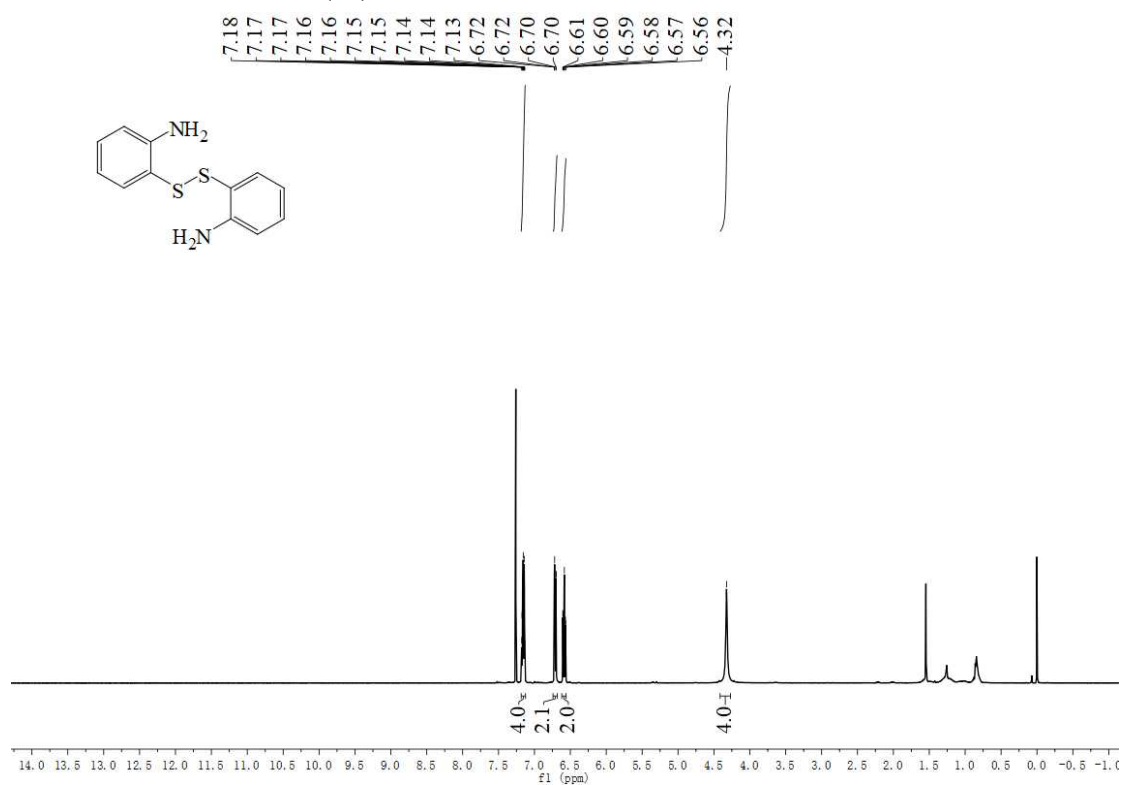


¹H NMR (CDCl₃, 400 MHz) of 2d

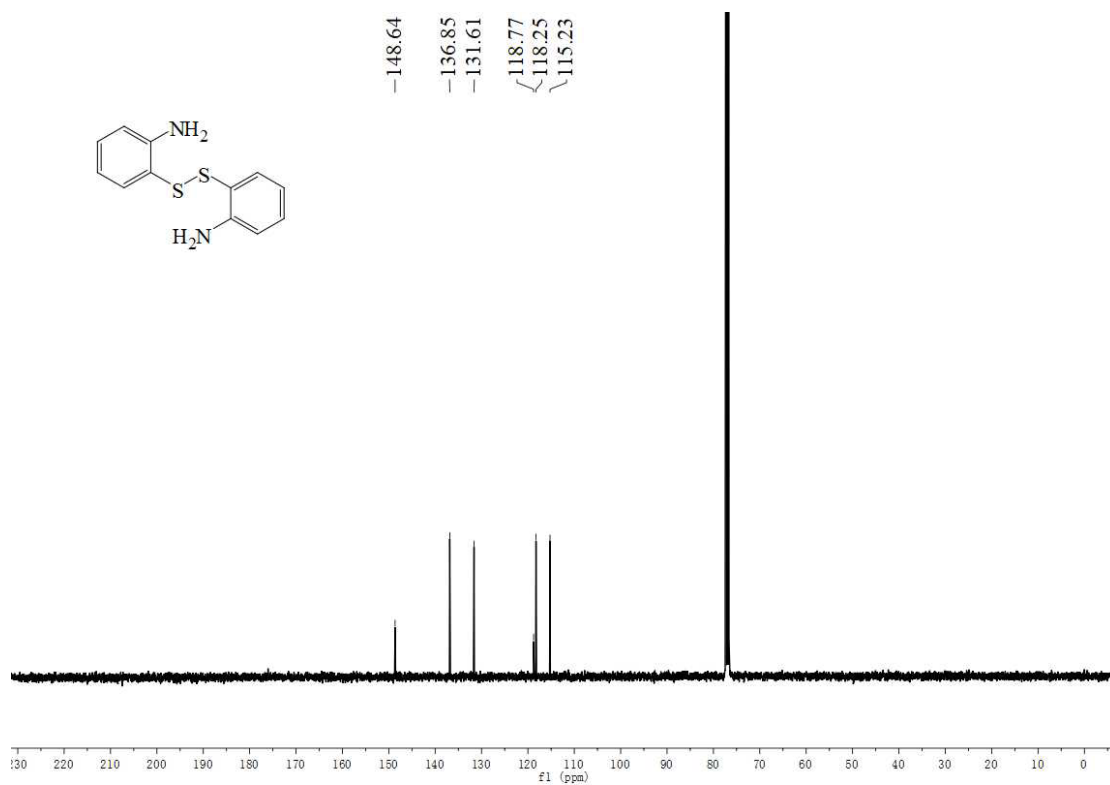


¹³C NMR (CDCl₃, 400 MHz) of 2d

2,2'-disulfanediyldianiline (2e)

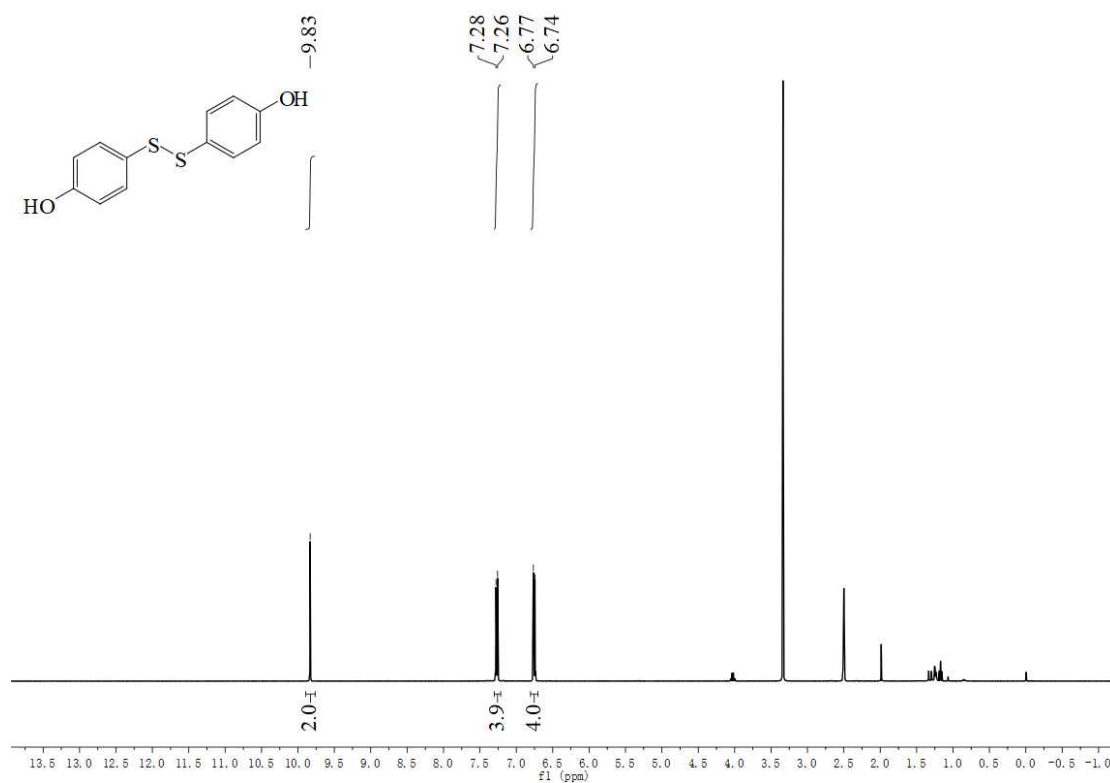


¹H NMR (CDCl₃, 400 MHz) of 2e

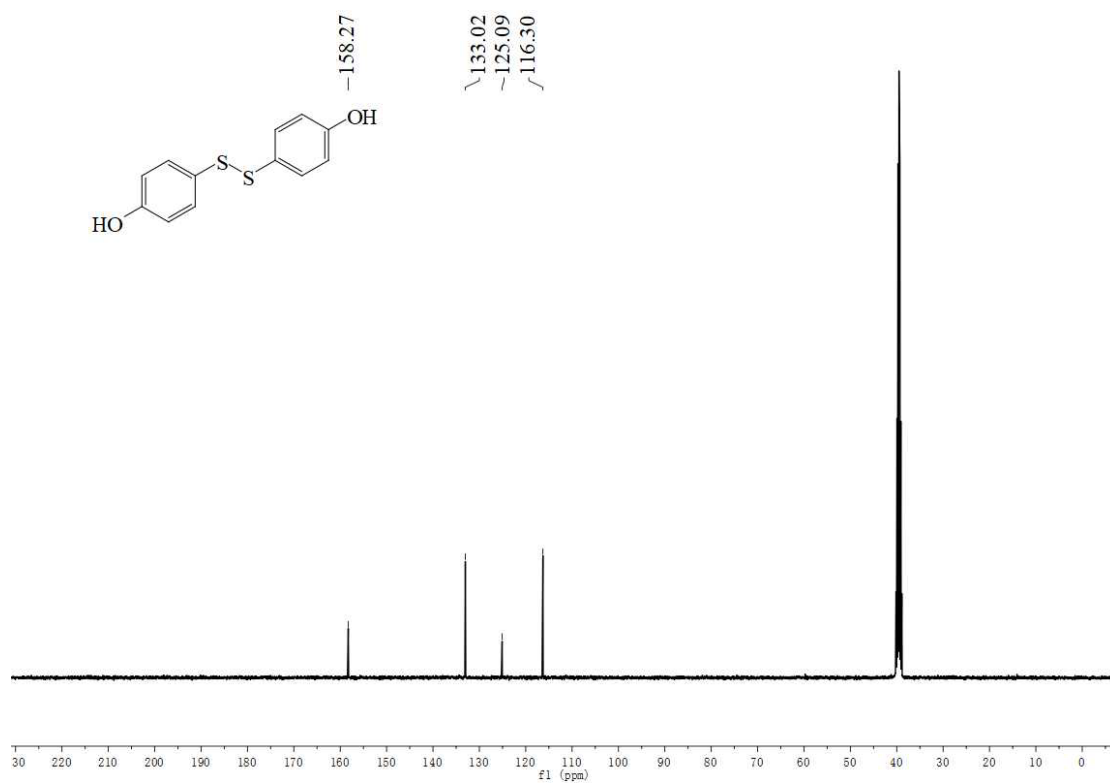


¹³C NMR (CDCl₃, 100 MHz) of 2e

4,4'-disulfanediylidiphenol (2f)

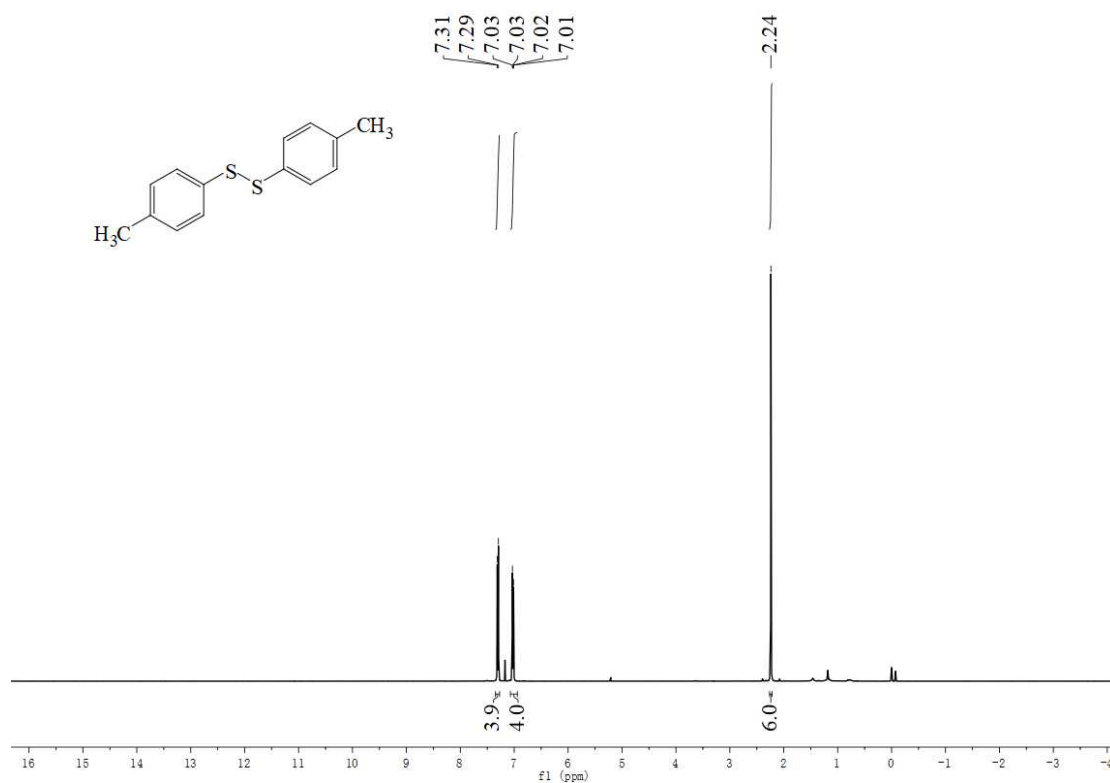


¹H NMR (DMSO, 400 MHz) of **2f**

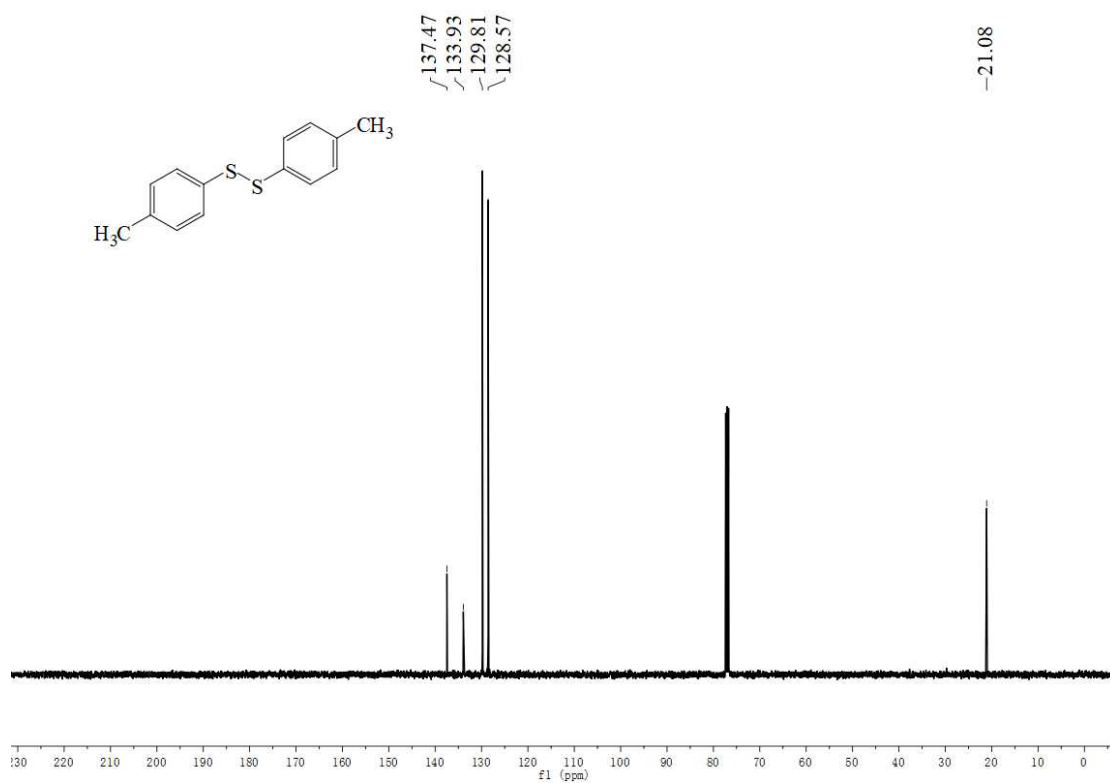


¹³C NMR (DMSO, 100 MHz) of **2f**

1,2-di-p-tolyldisulfane (2g)

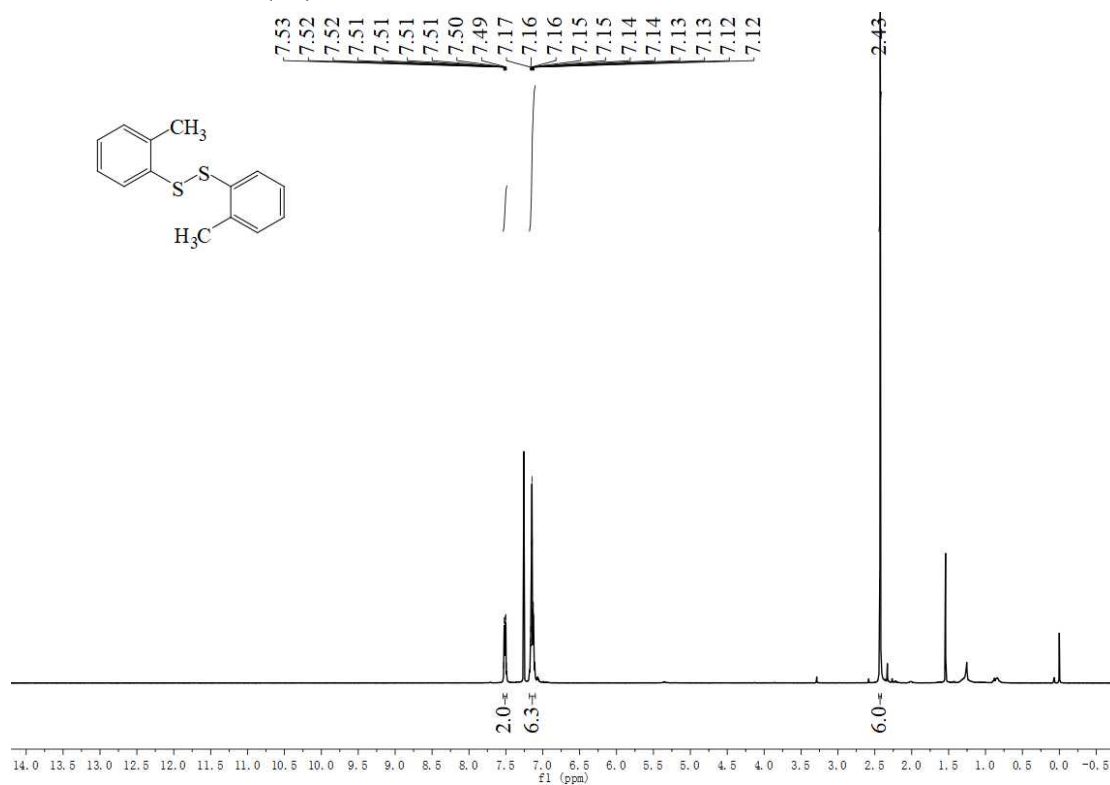


¹H NMR (CDCl₃, 400 MHz) of **2g**

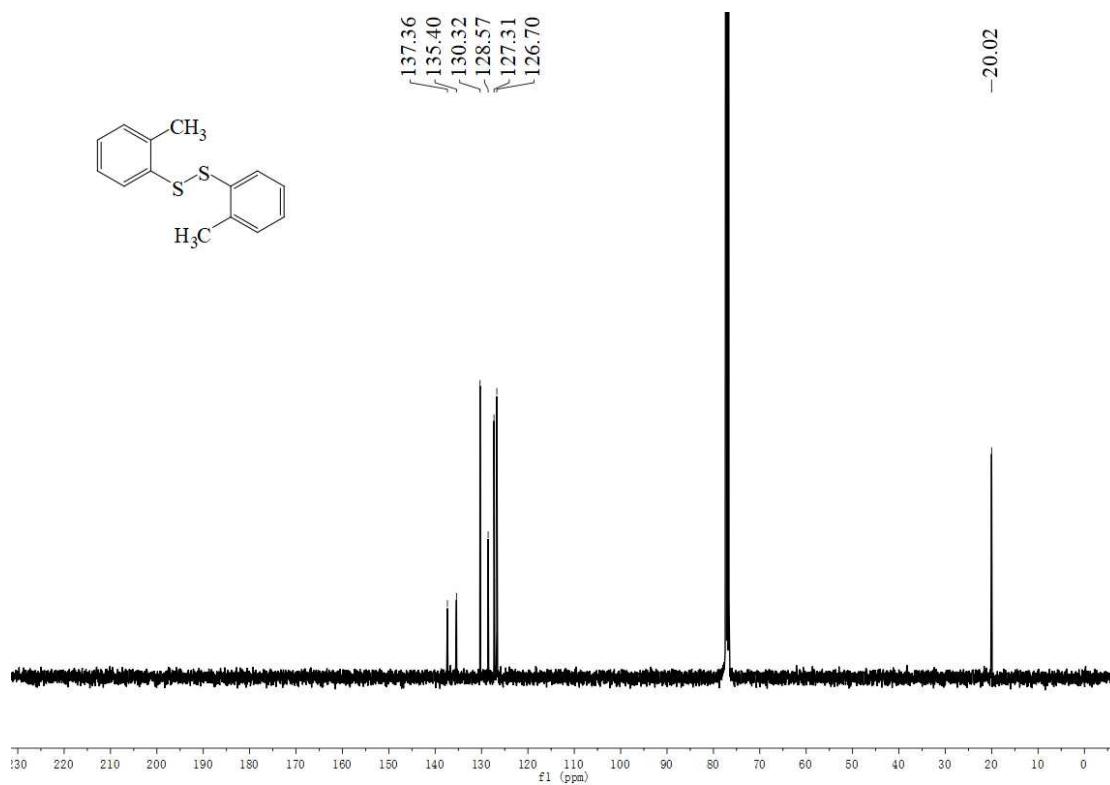


¹³C NMR (CDCl₃, 100 MHz) of **2g**

1,2-di-*o*-tolylsulfane (2h)

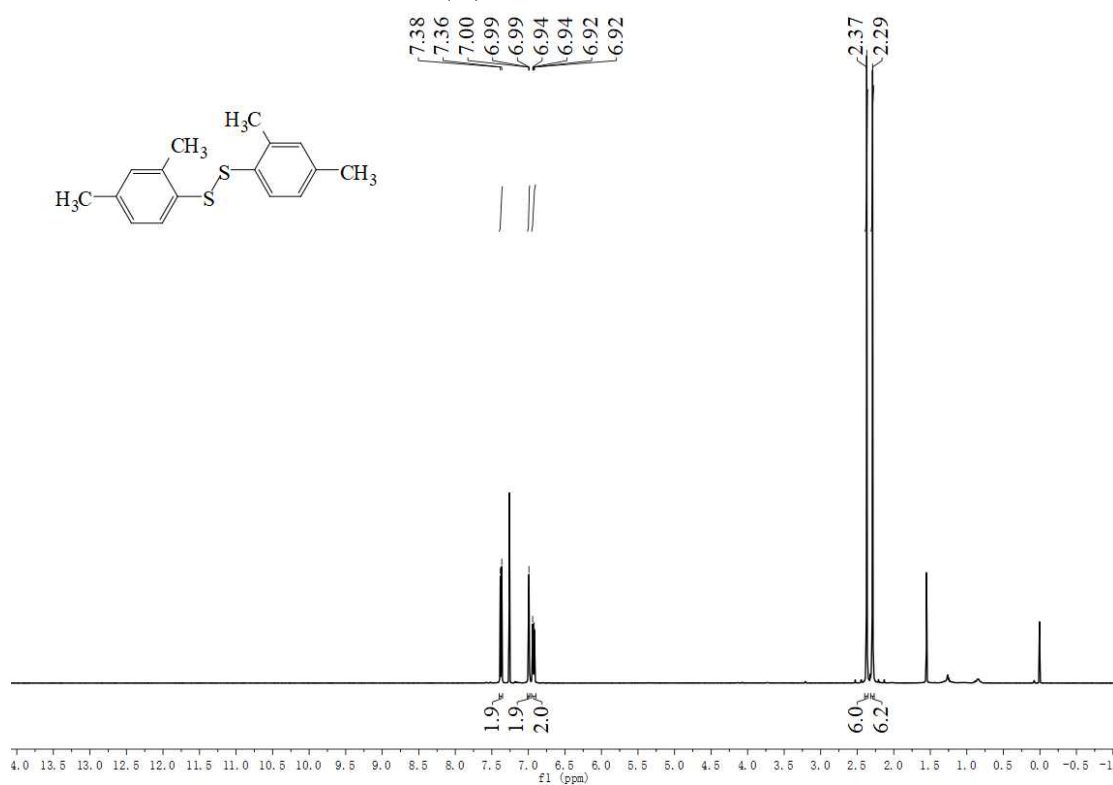


¹H NMR (CDCl₃, 400 MHz) of **2h**

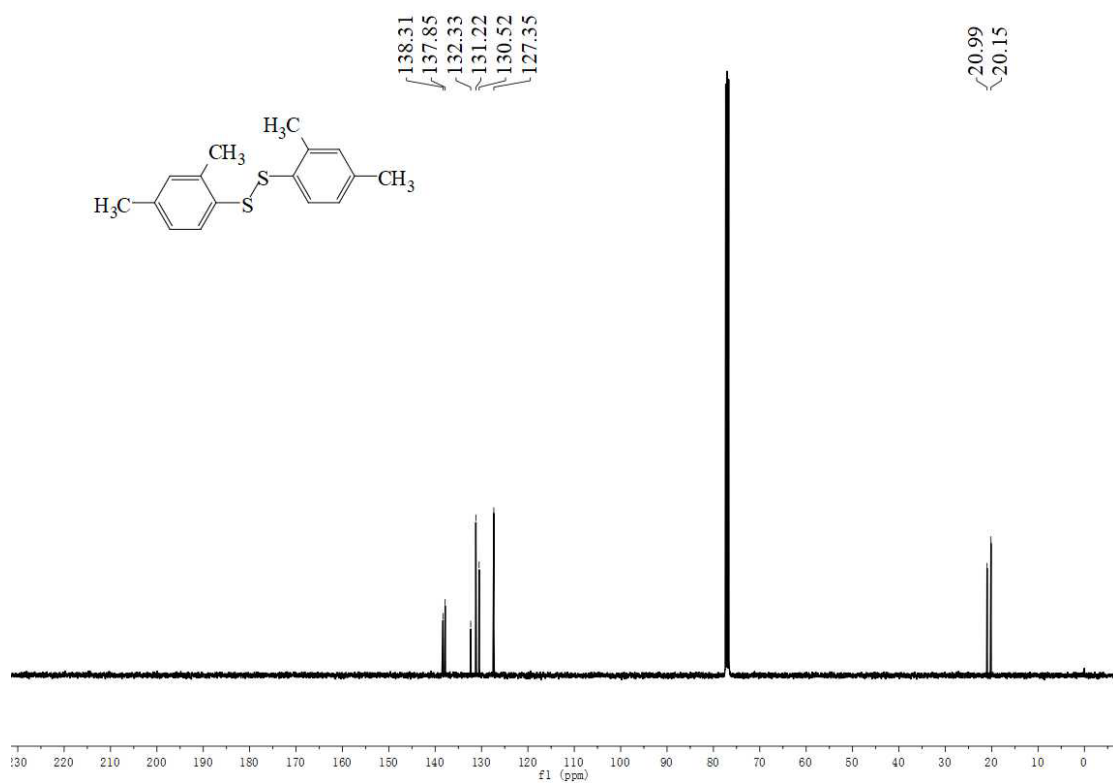


¹³C NMR (CDCl₃, 100 MHz) of **2h**

1,2-bis(2,4-dimethylphenyl)disulfane (2i)

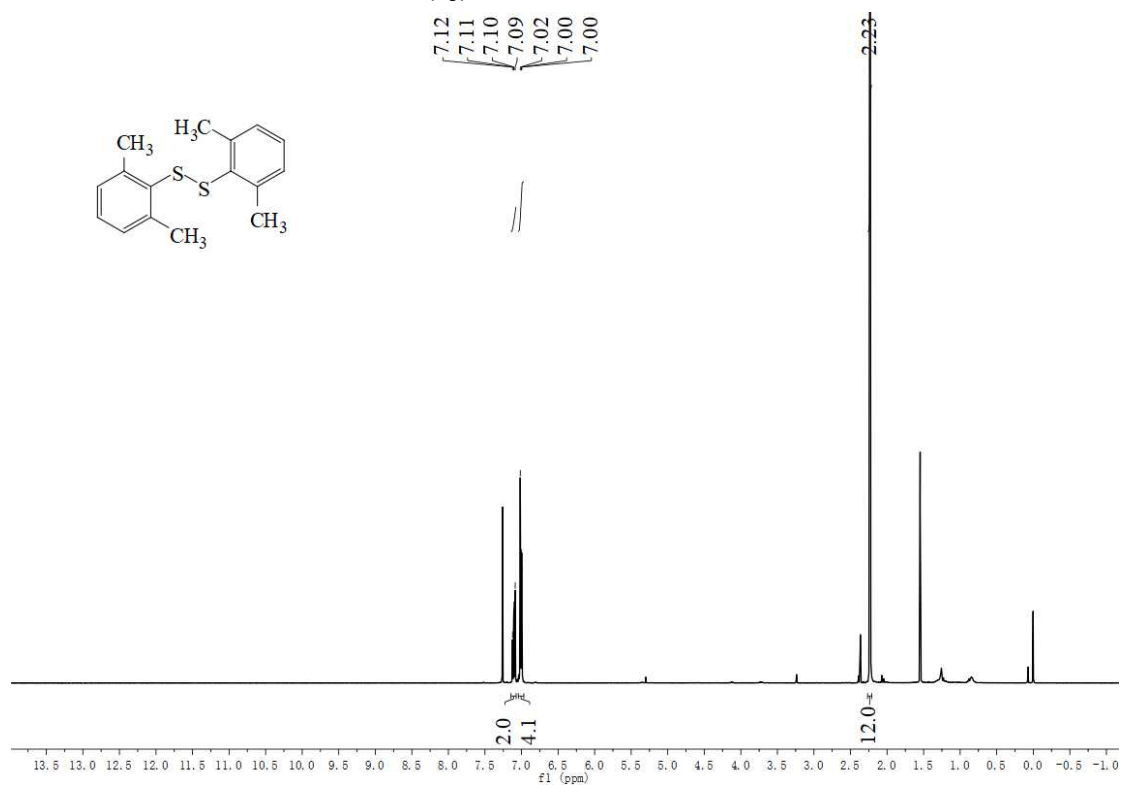


¹H NMR (CDCl₃, 400 MHz) of **2i**

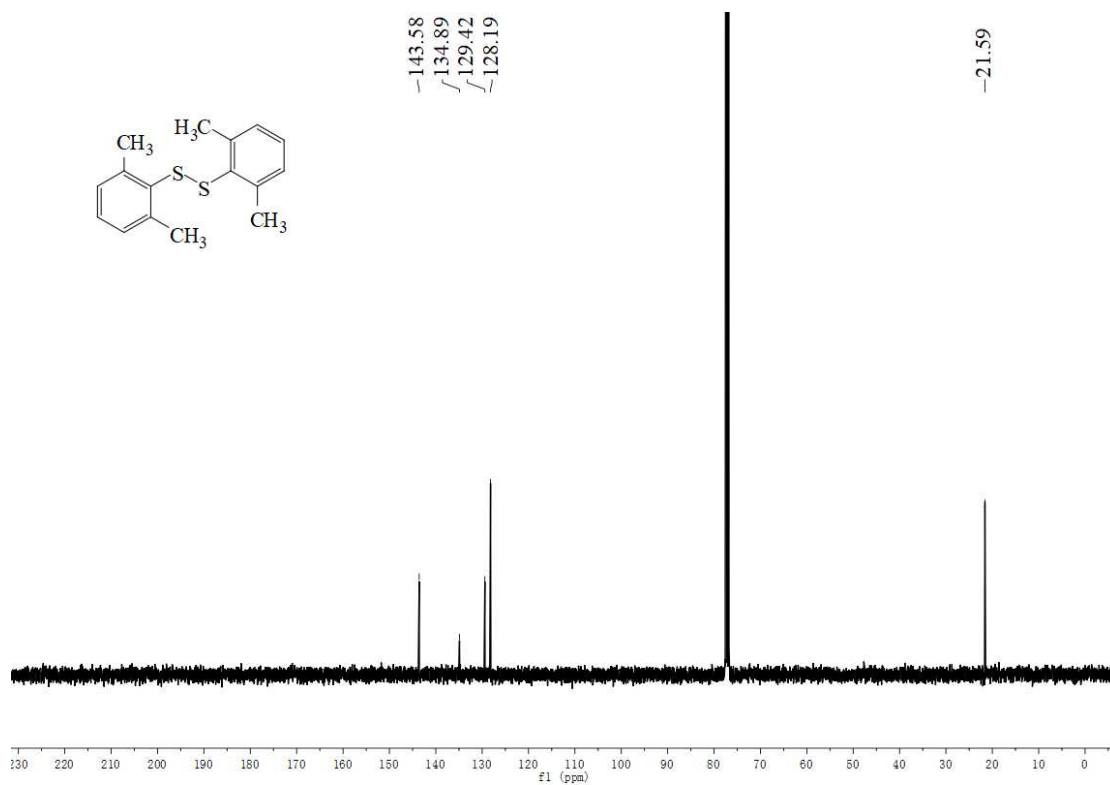


¹³C NMR (CDCl₃, 100 MHz) of **2i**

1,2-bis(2,6-dimethylphenyl)disulfane (2j)

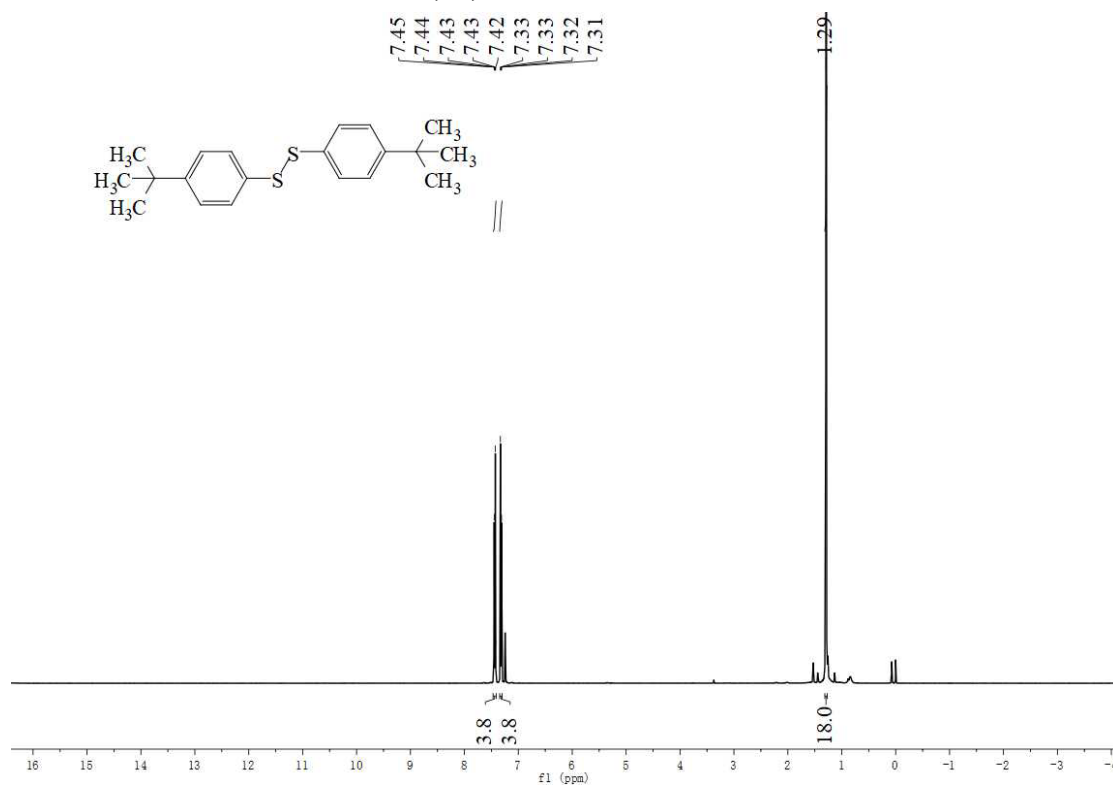


¹H NMR (DMSO, 400 MHz) of **2j**

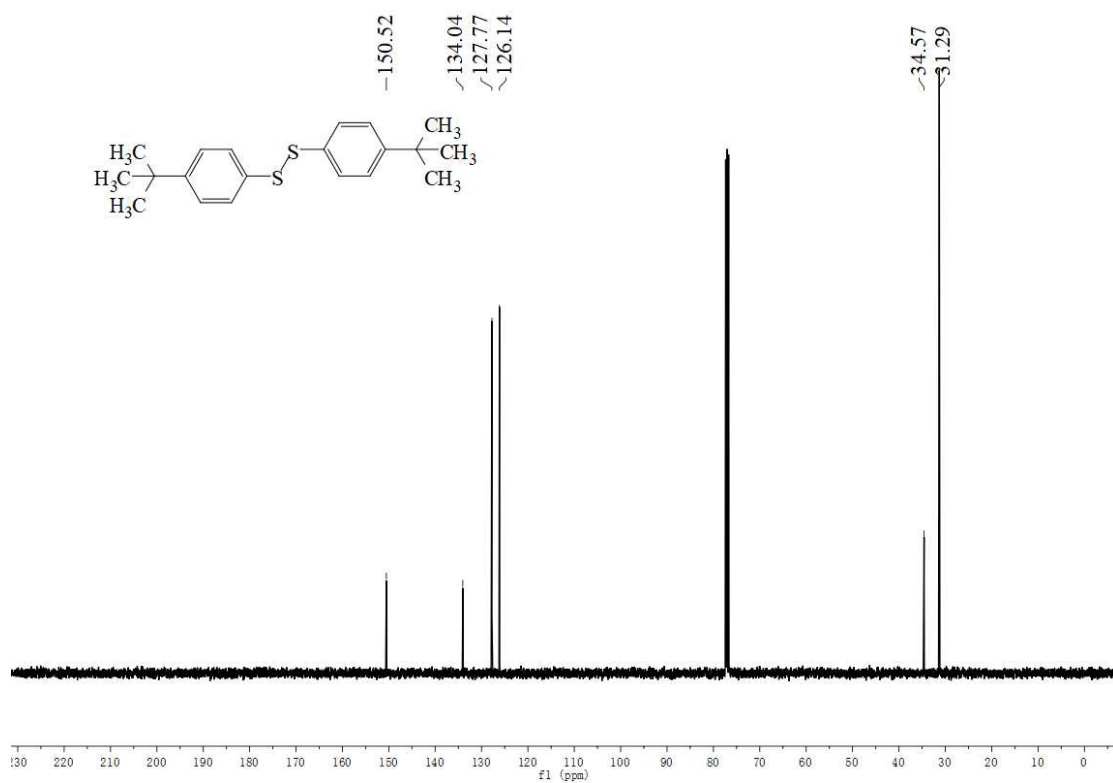


¹³C NMR (DMSO, 100 MHz) of **2j**

1,2-bis(4-(tert-butyl)phenyl)disulfane (2k)

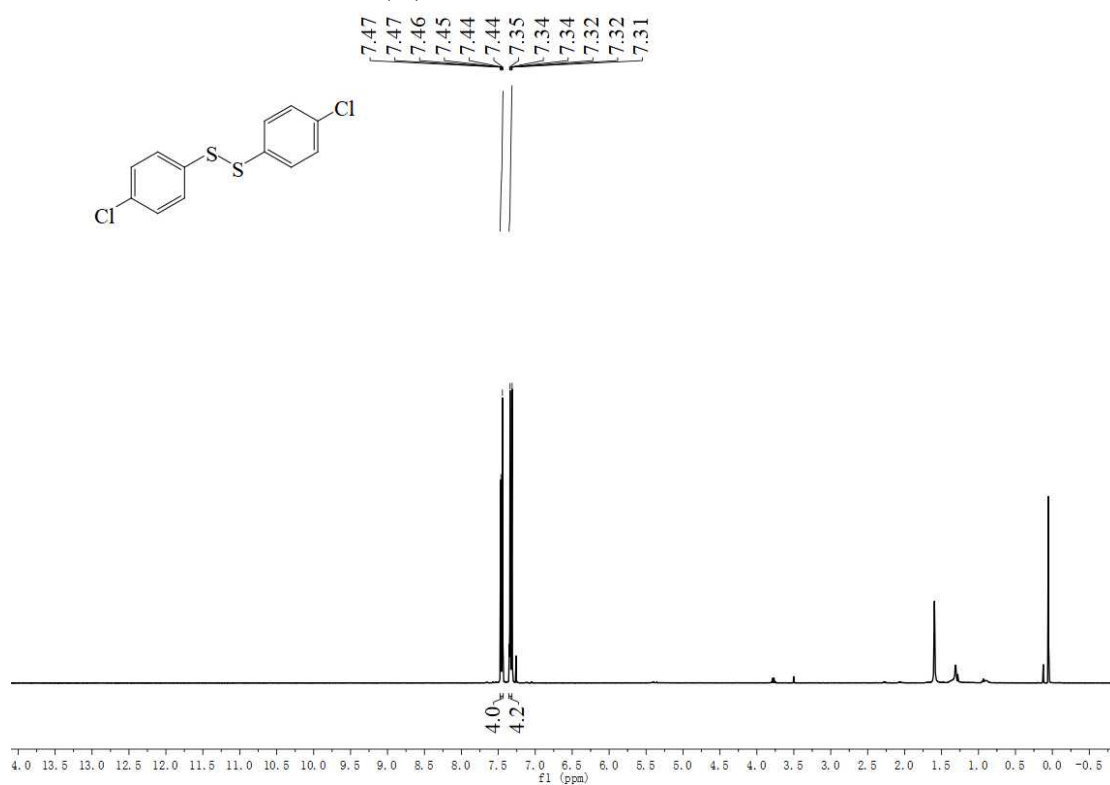


¹H NMR (CDCl₃, 400 MHz) of **2k**

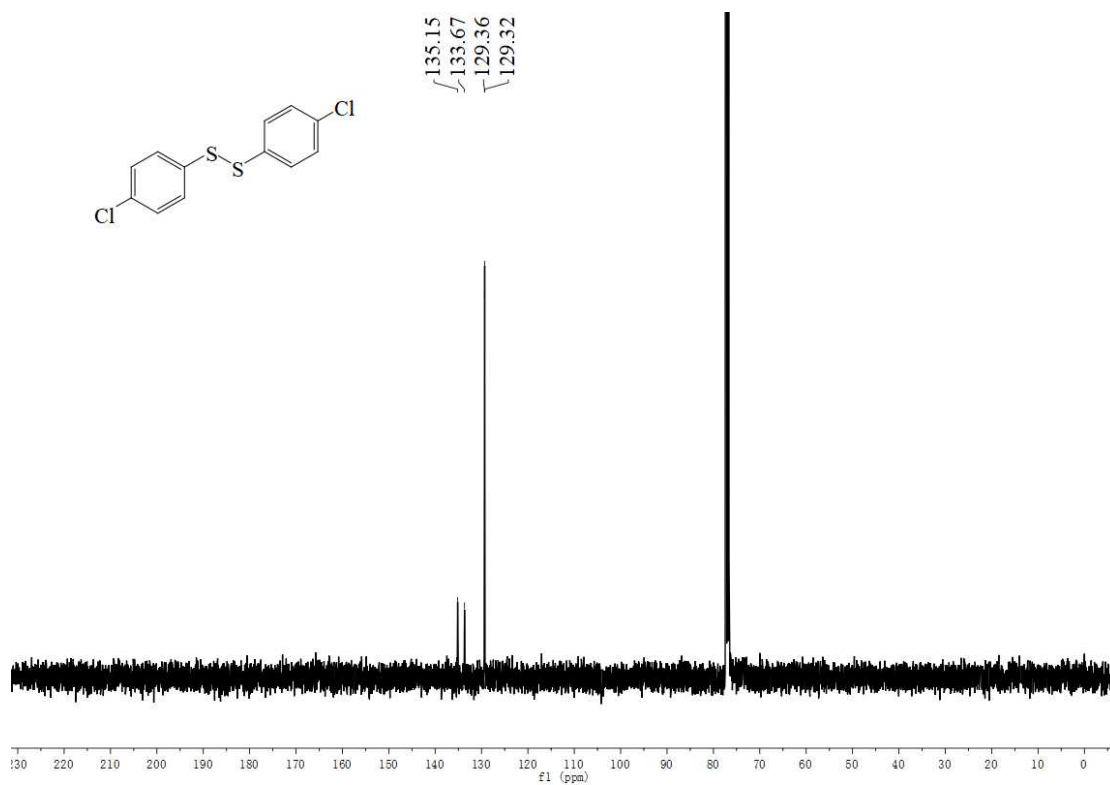


¹³C NMR (CDCl₃, 100 MHz) of **2k**

1,2-bis(4-chlorophenyl)disulfane (2l)

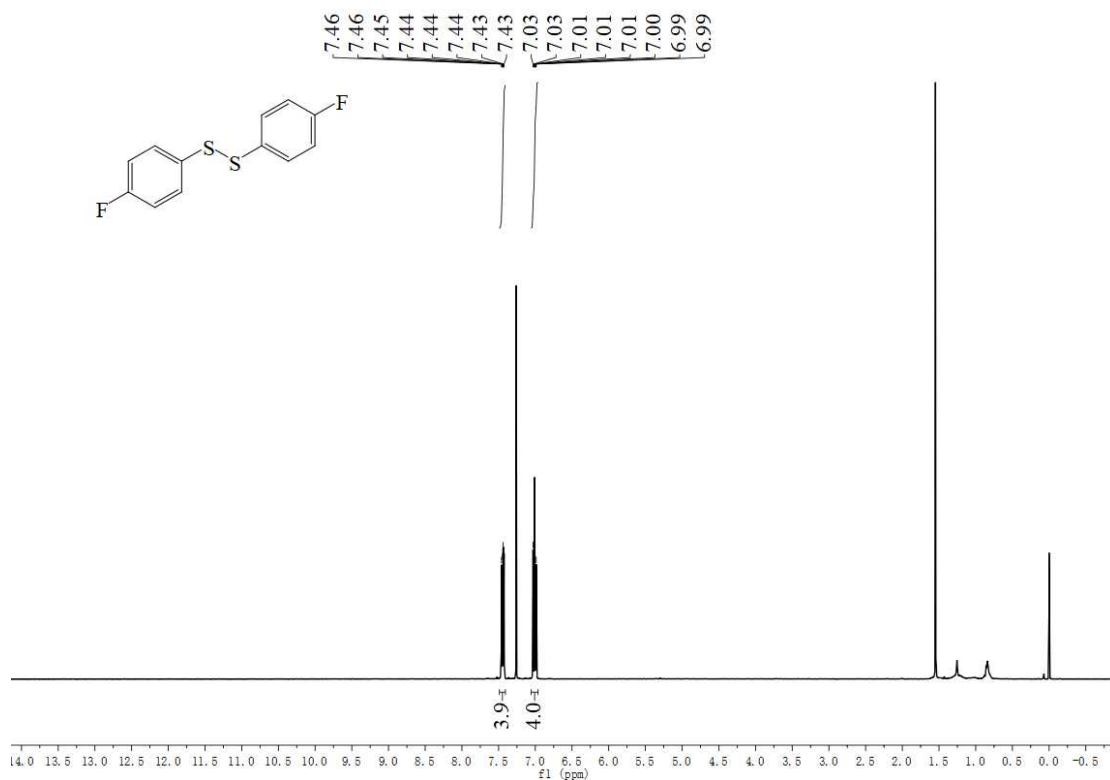


¹H NMR (CDCl₃, 400 MHz) of **2l**

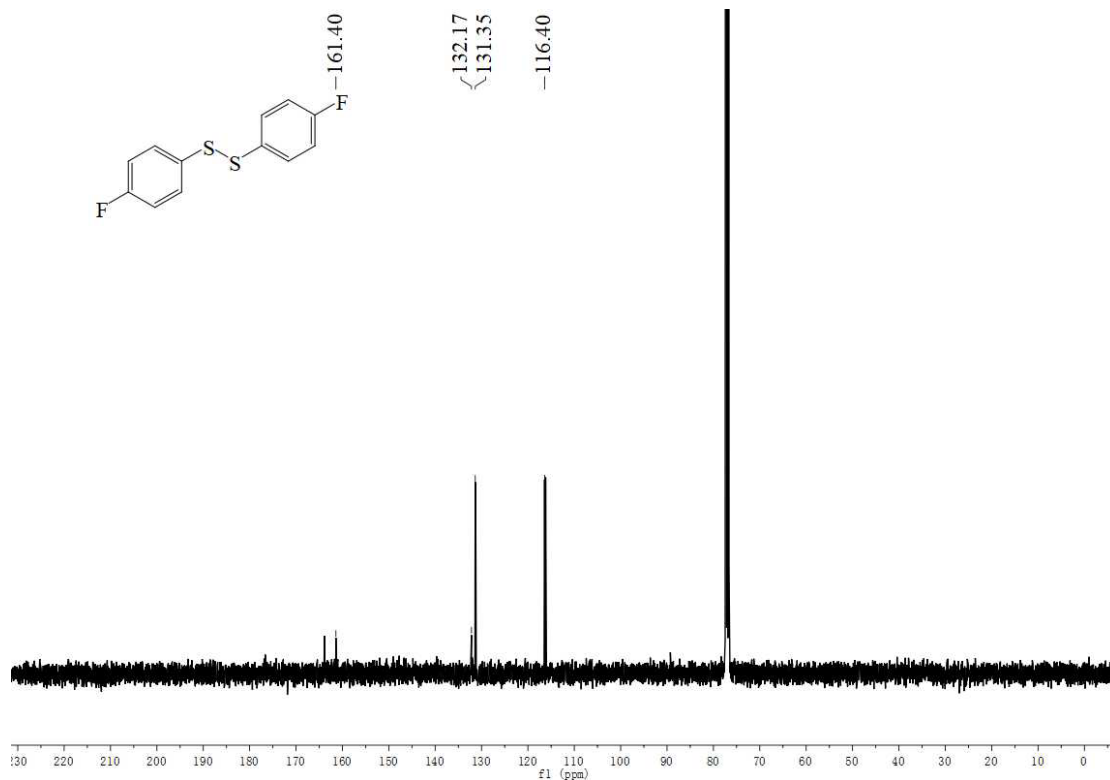


¹³C NMR (CDCl₃, 100 MHz) of **2l**

1,2-bis(4-fluorophenyl)disulfane (2m)

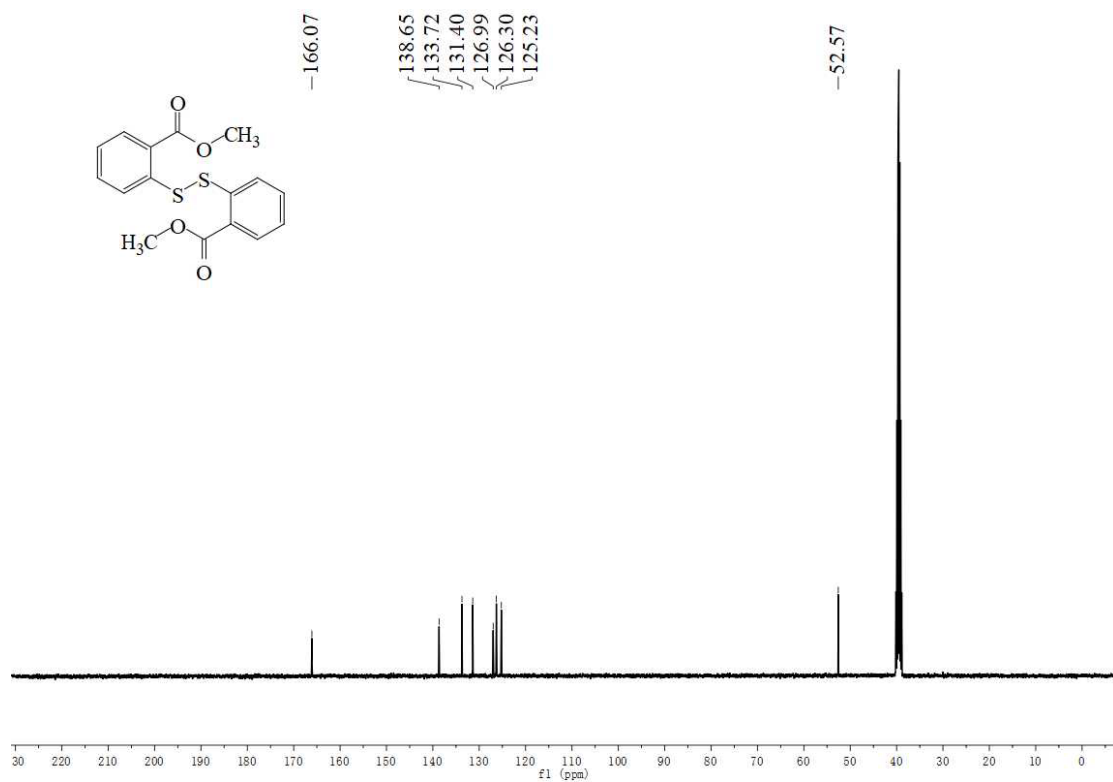
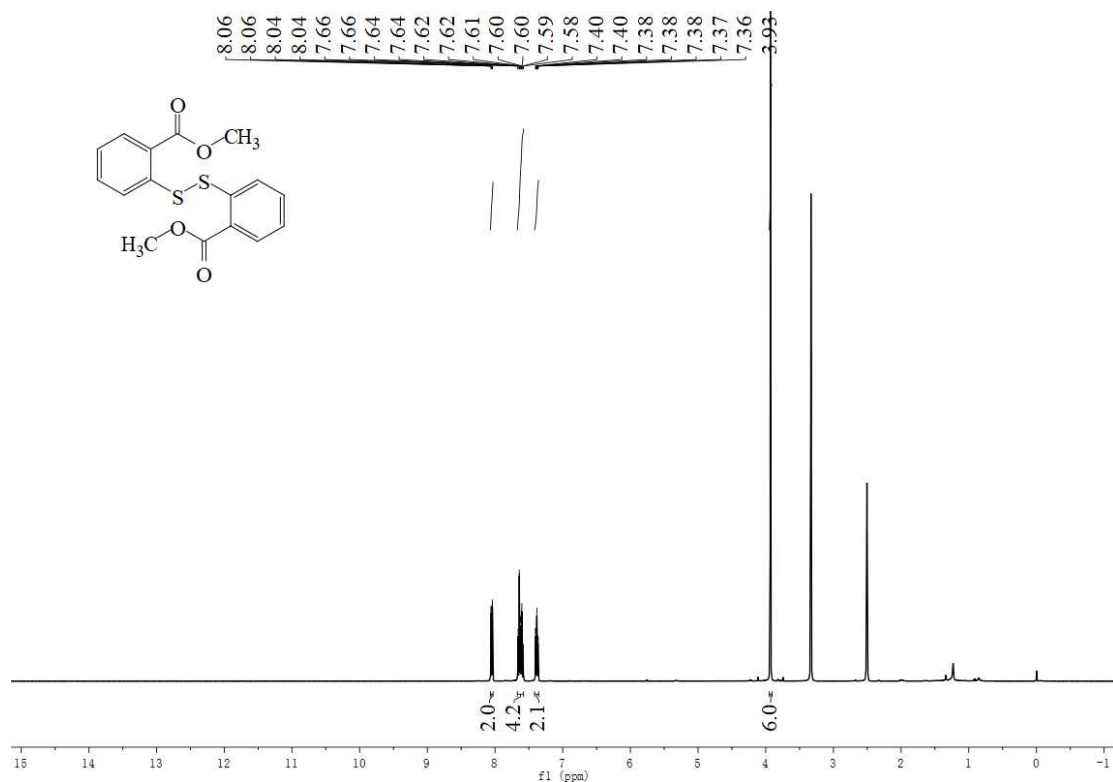


$^1\text{H NMR}$ (CDCl₃, 400 MHz) of **2m**

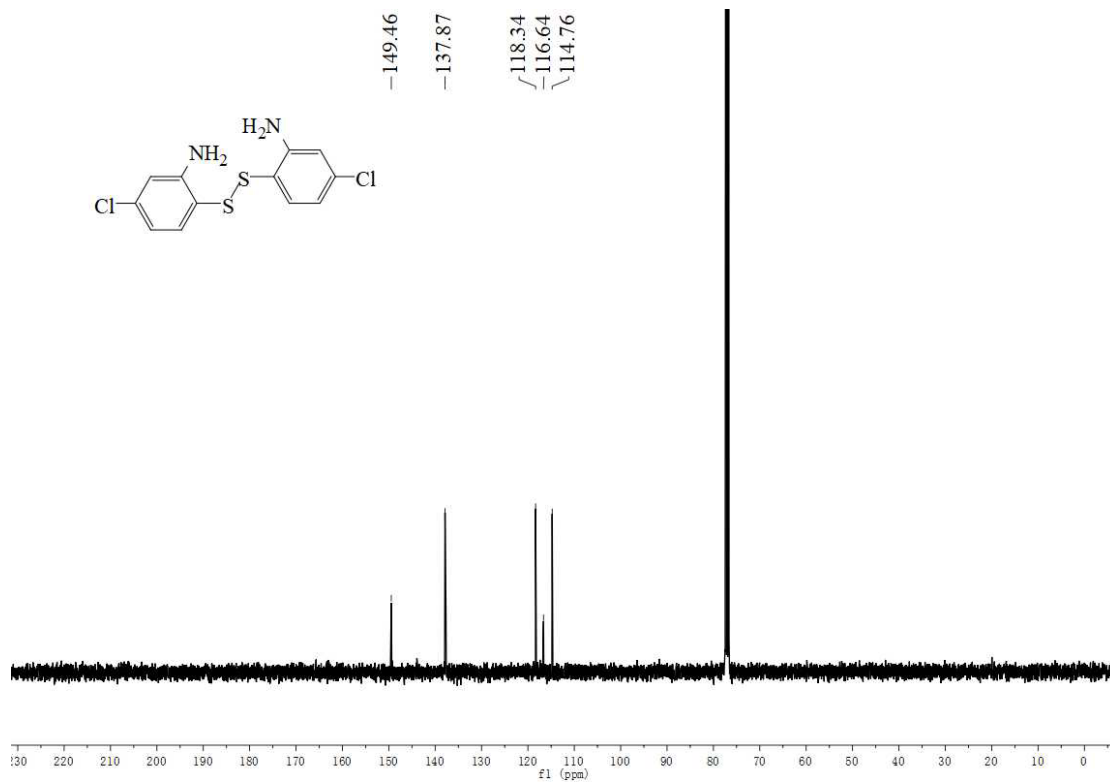
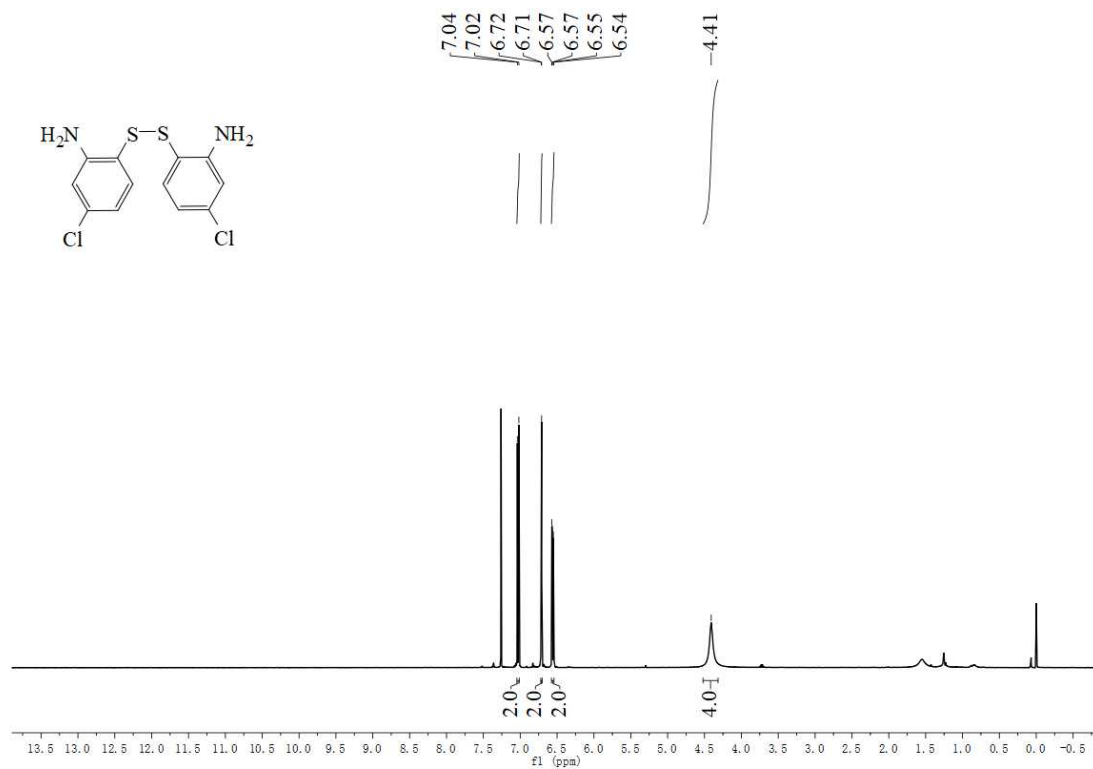


$^{13}\text{C NMR}$ (CDCl₃, 100 MHz) of **2m**

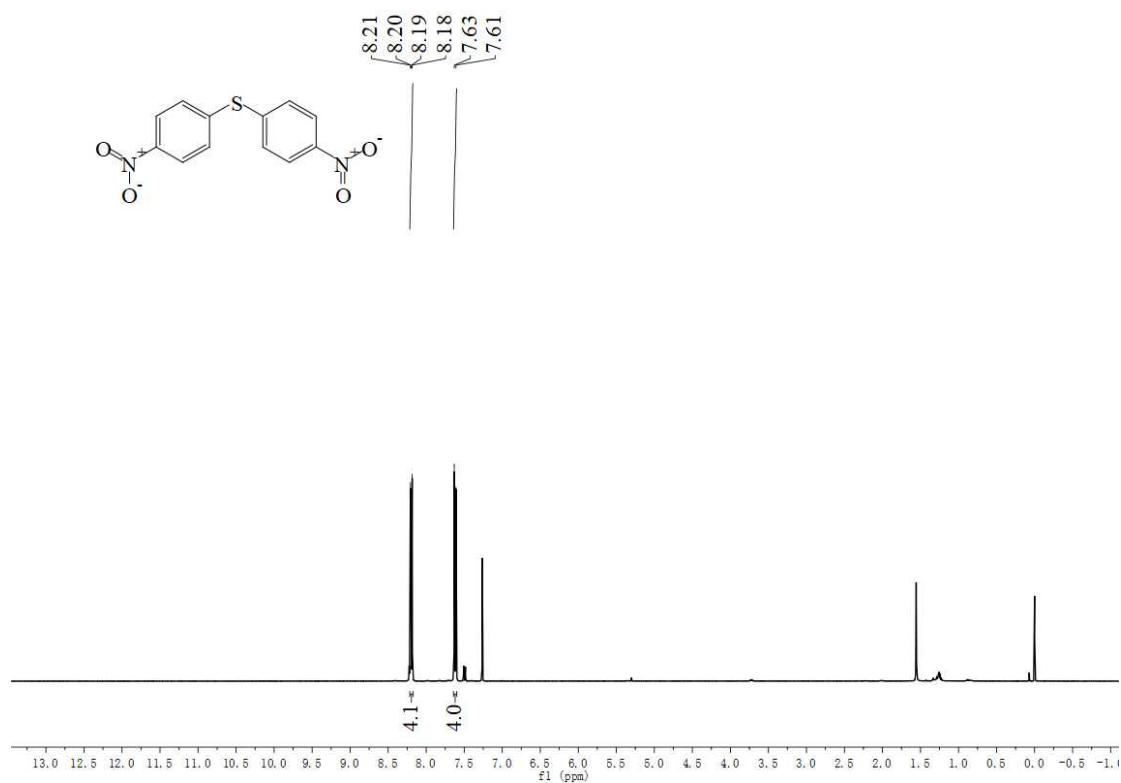
dimethyl 2,2'-disulfaneyldibenzoate (2n)



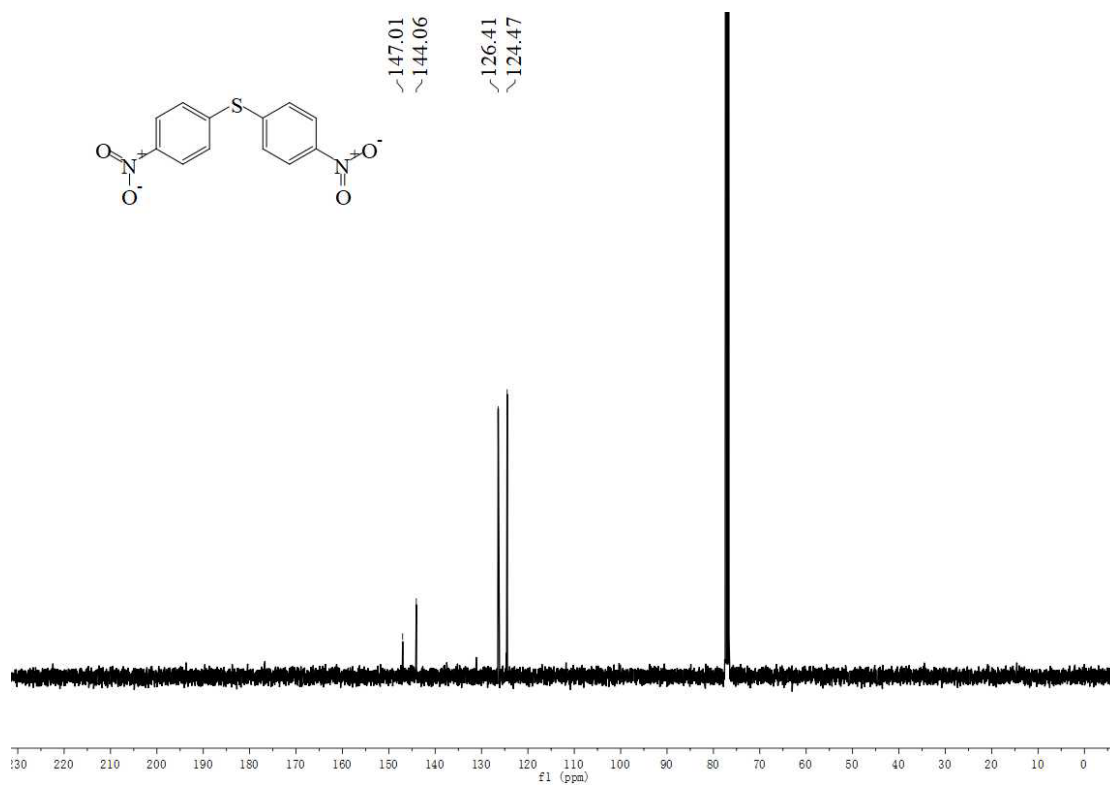
6,6'-disulfaneylbis(3-chloroaniline) (2o)



bis(4-nitrophenyl)sulfane (2p)

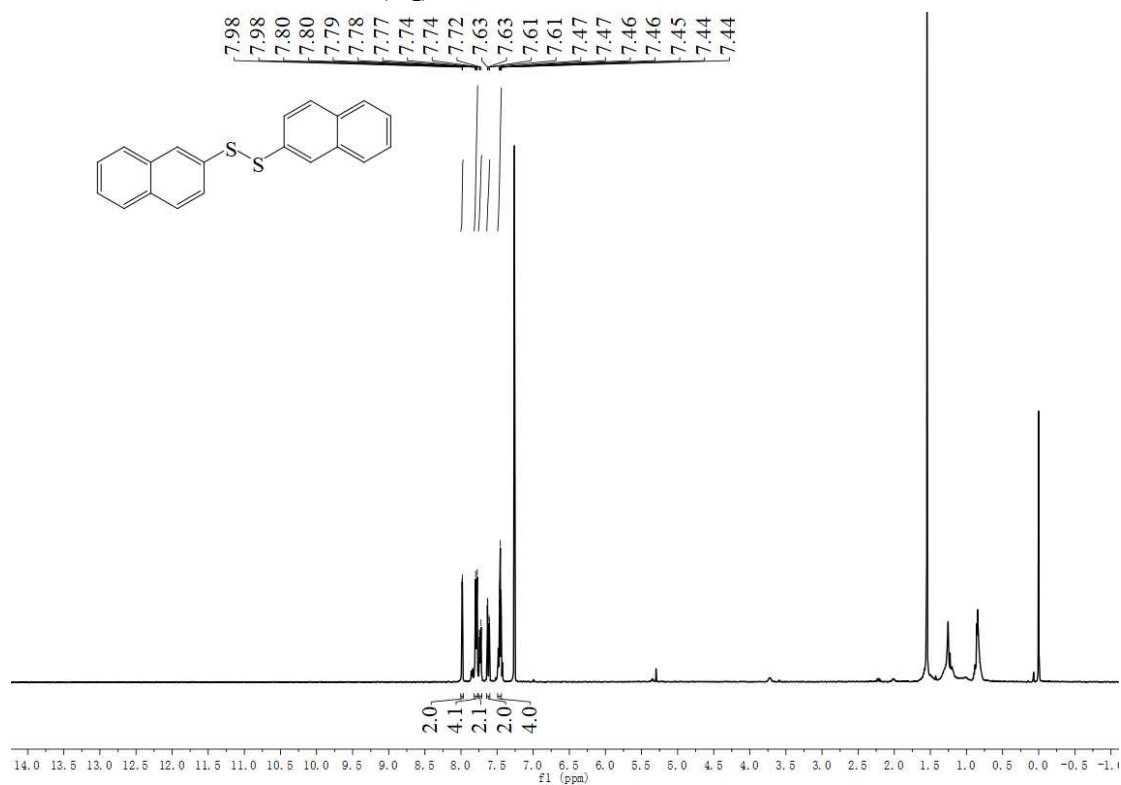


¹H NMR (CDCl₃, 400 MHz) of **2p**

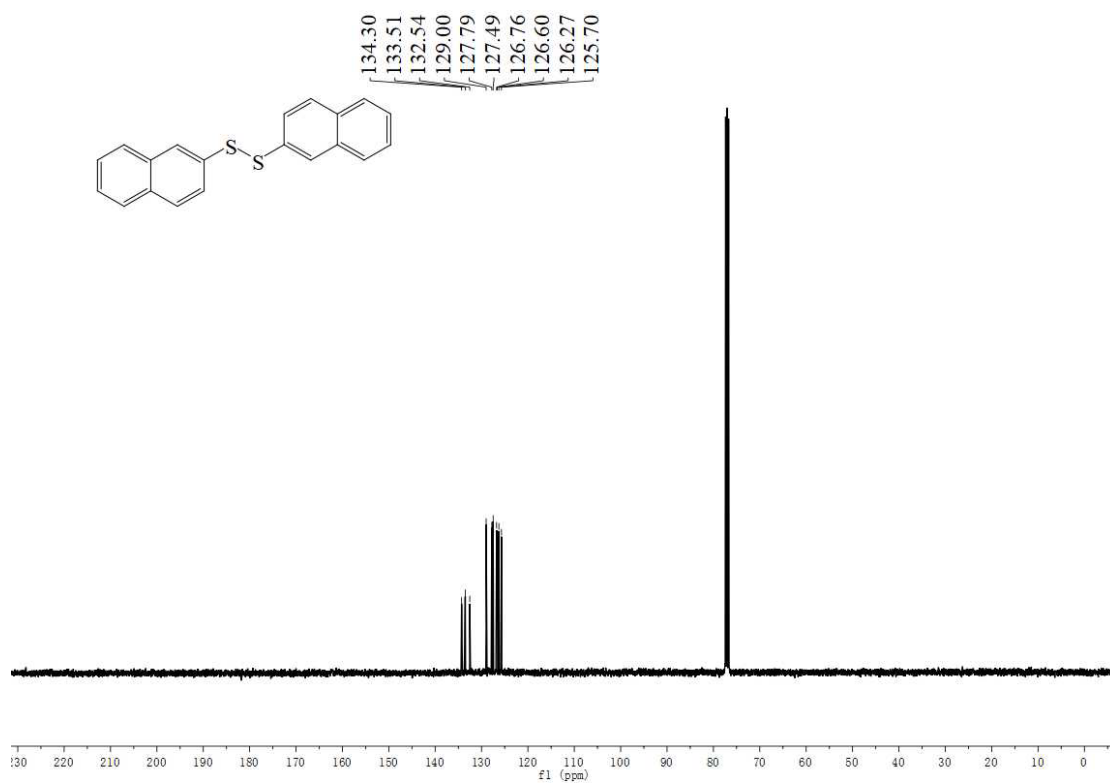


¹³C NMR (CDCl₃, 100 MHz) of **2p**

1,2-di(naphthalen-2-yl)disulfane (2q)

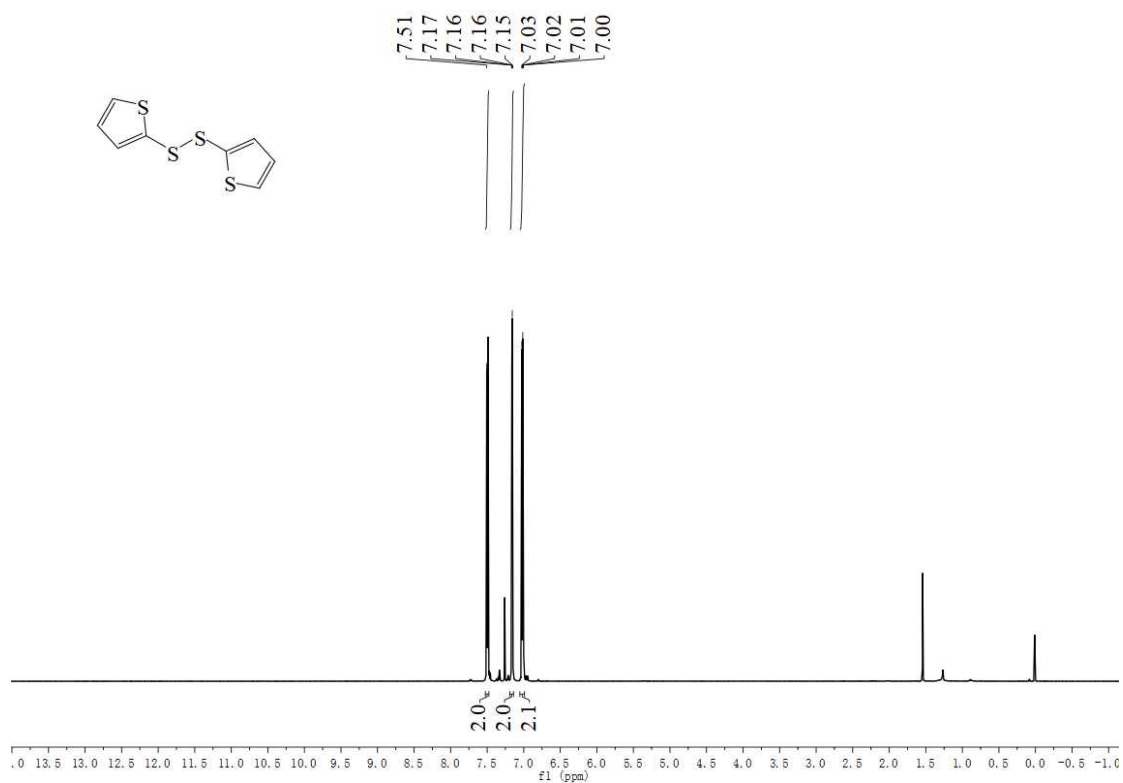


$^1\text{H NMR}$ (CDCl₃, 400 MHz) of **2q**

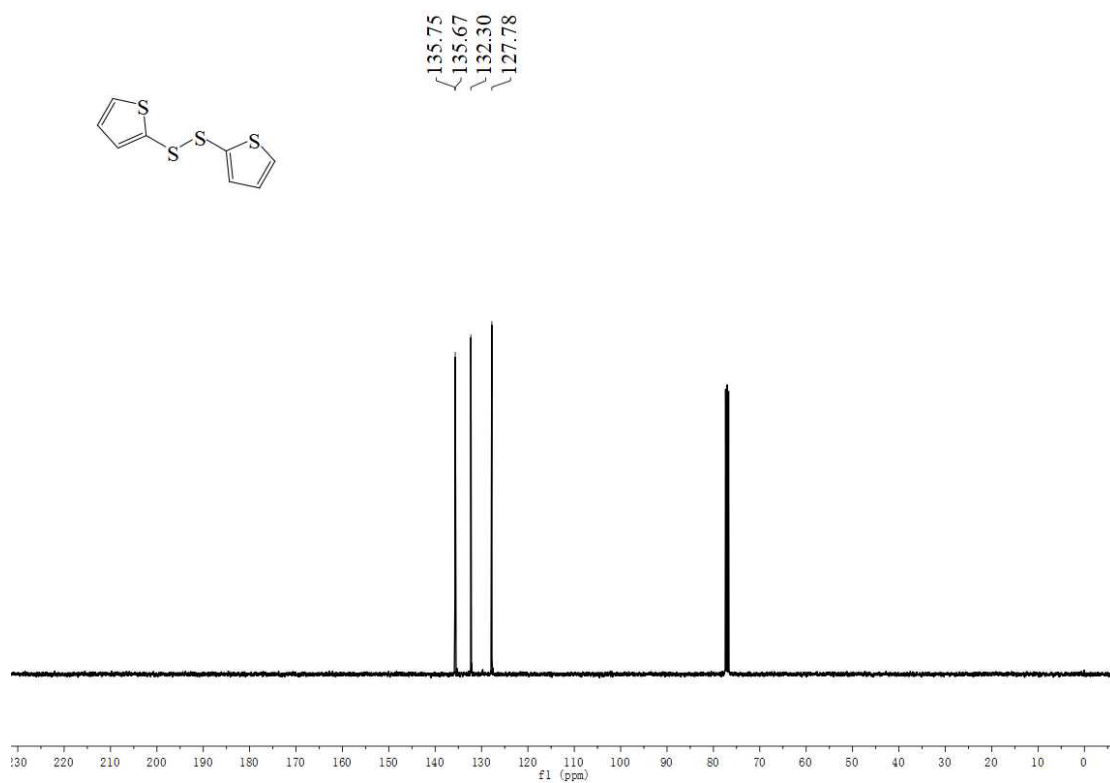


$^{13}\text{C NMR}$ (CDCl₃, 100 MHz) of **2q**

1,2-di(thiophen-2-yl)disulfane (2r)

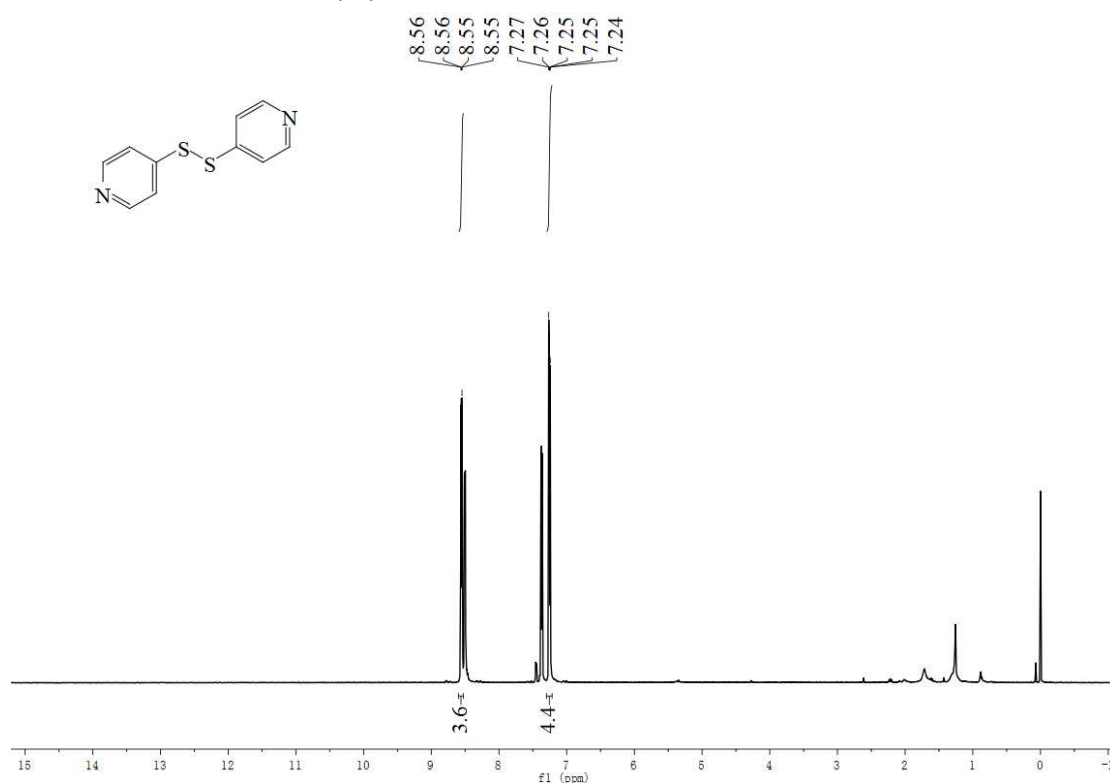


¹H NMR (CDCl₃, 400 MHz) of **2r**

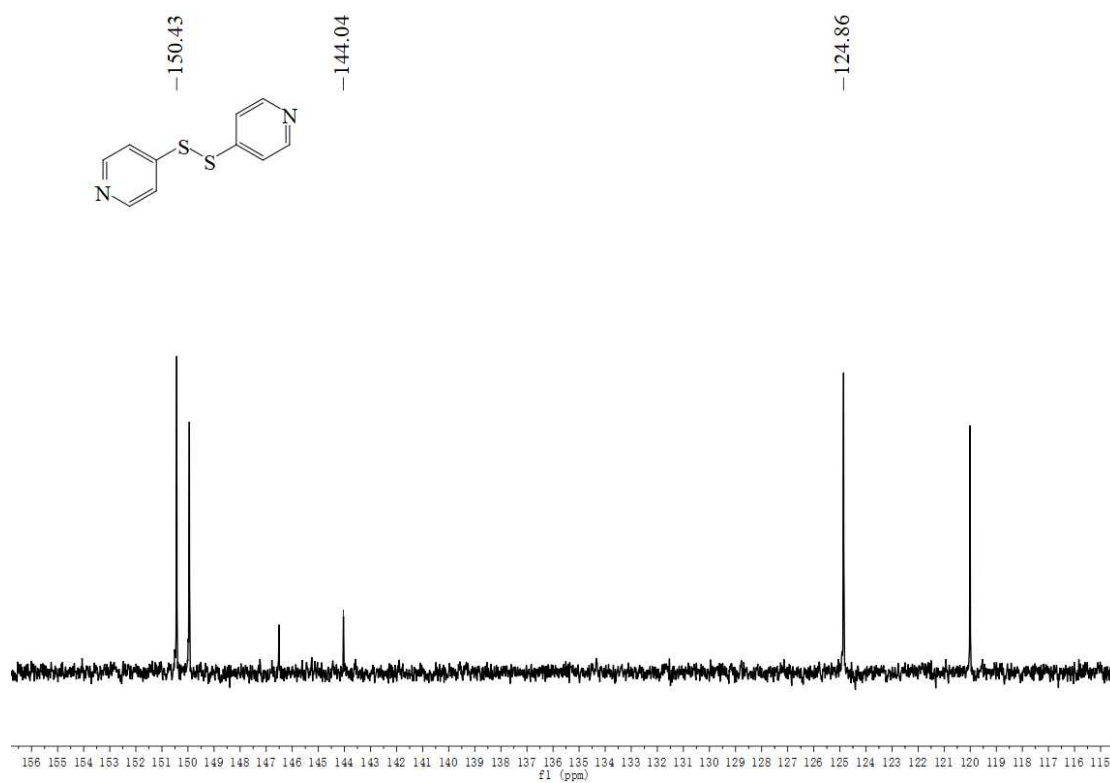


¹³C NMR (CDCl₃, 100 MHz) of **2r**

1,2-di(pyridin-4-yl)disulfane (2s)

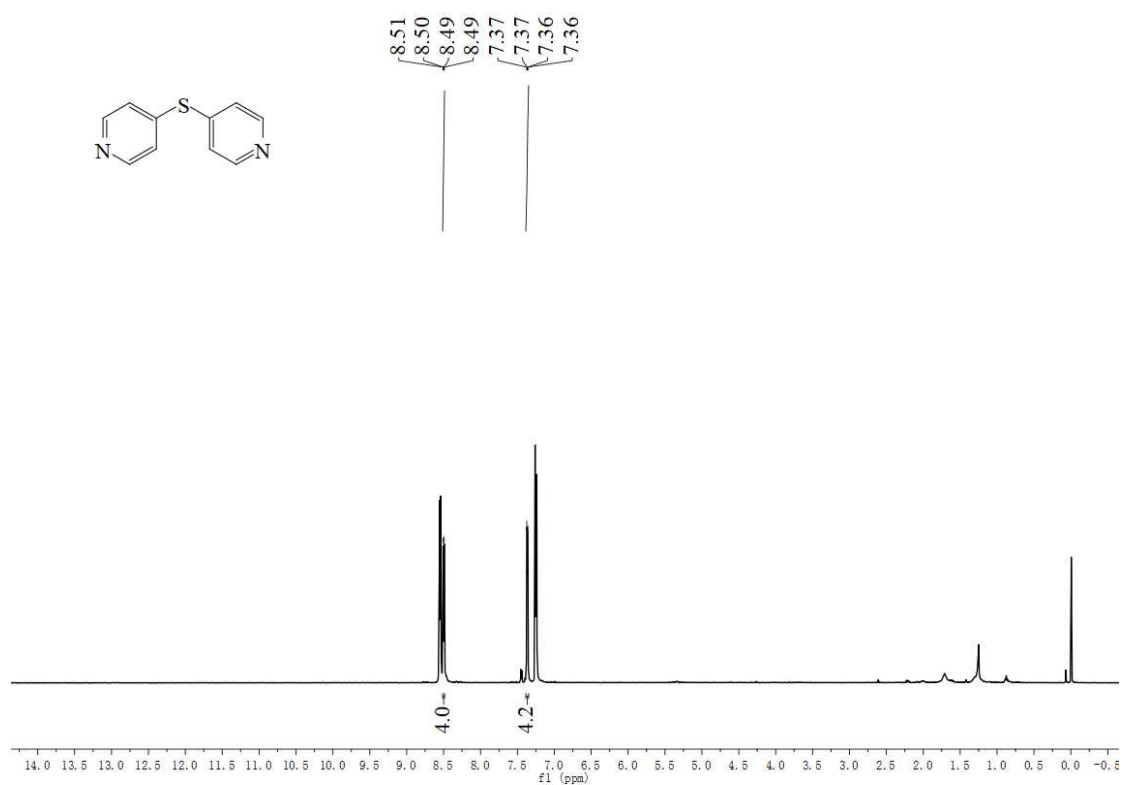


¹H NMR (CDCl₃, 400 MHz) of **2s**

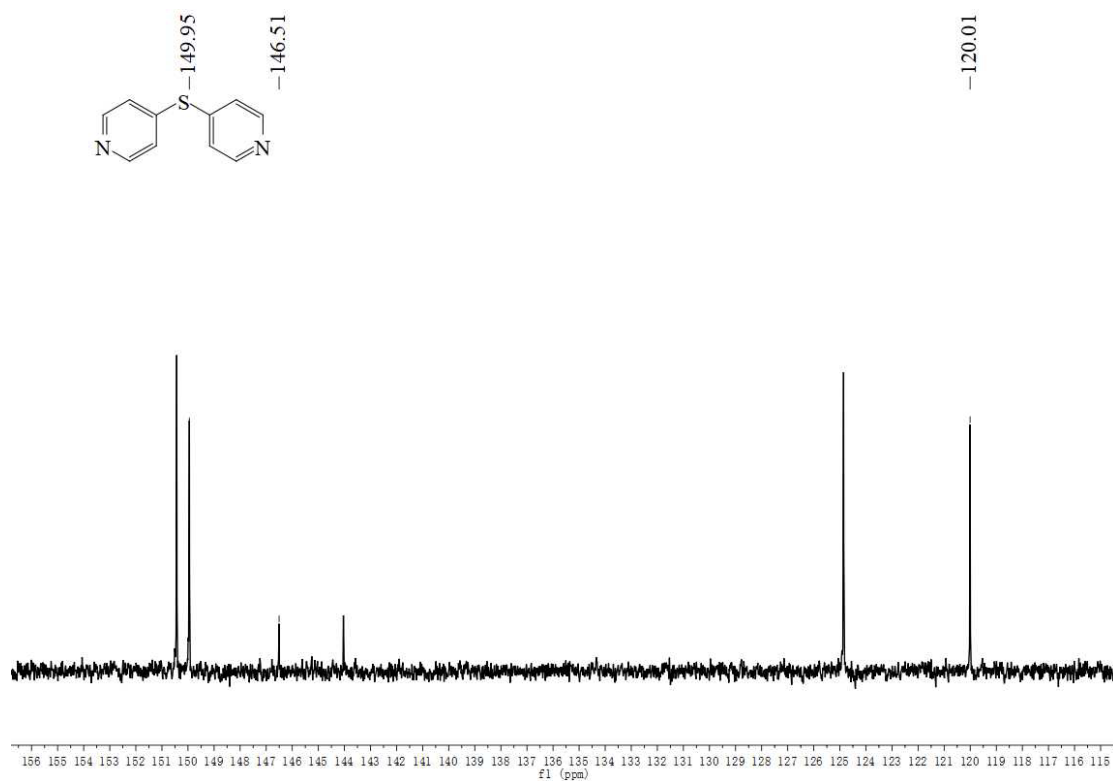


¹³C NMR (CDCl₃, 400 MHz) of **2s**

di(pyridine-4-yl)sulfane (2s')

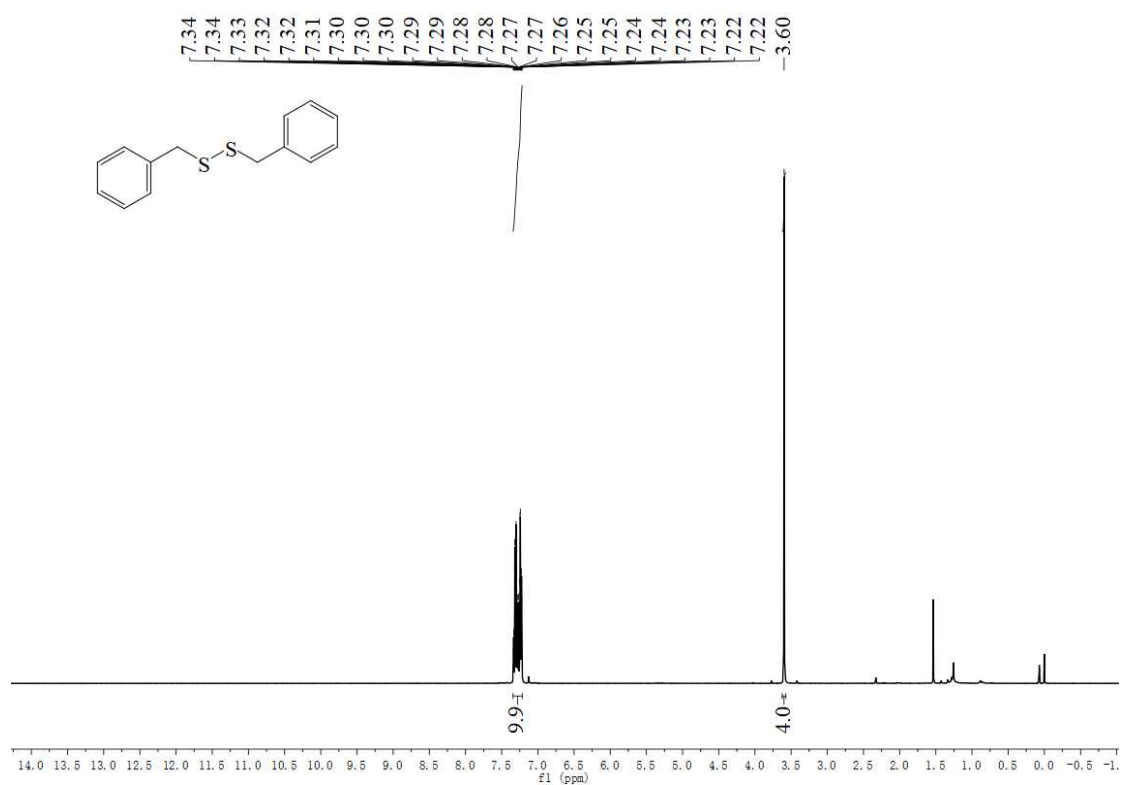


¹H NMR (CDCl₃, 400 MHz) of **2s'**

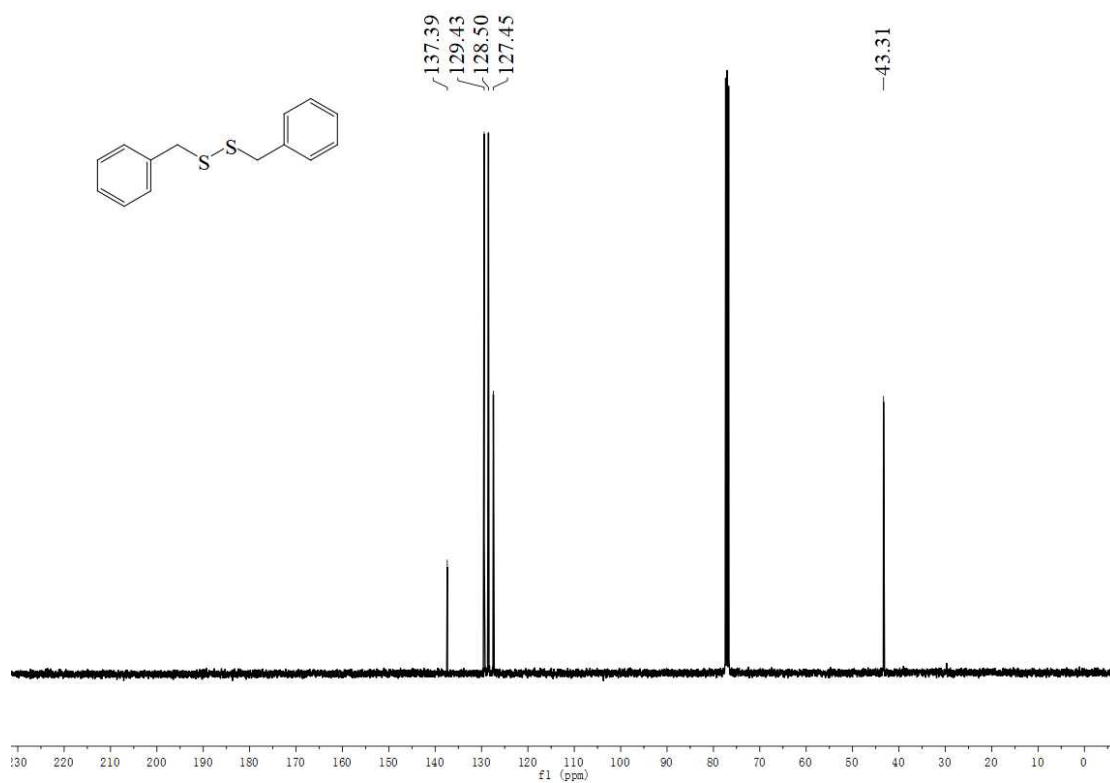


¹³C NMR (CDCl₃, 100 MHz) of **2s'**

1,2-dibenzylidysulfane (2t)

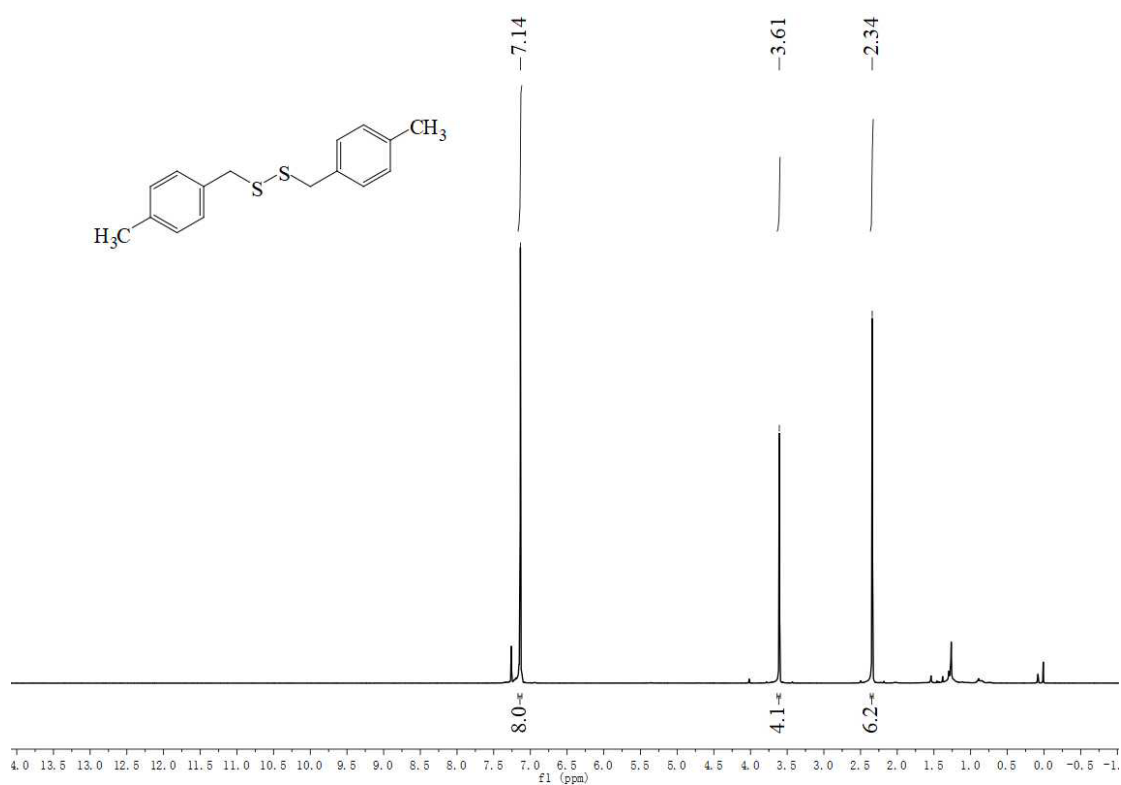


¹H NMR (CDCl₃, 400 MHz) of 2t

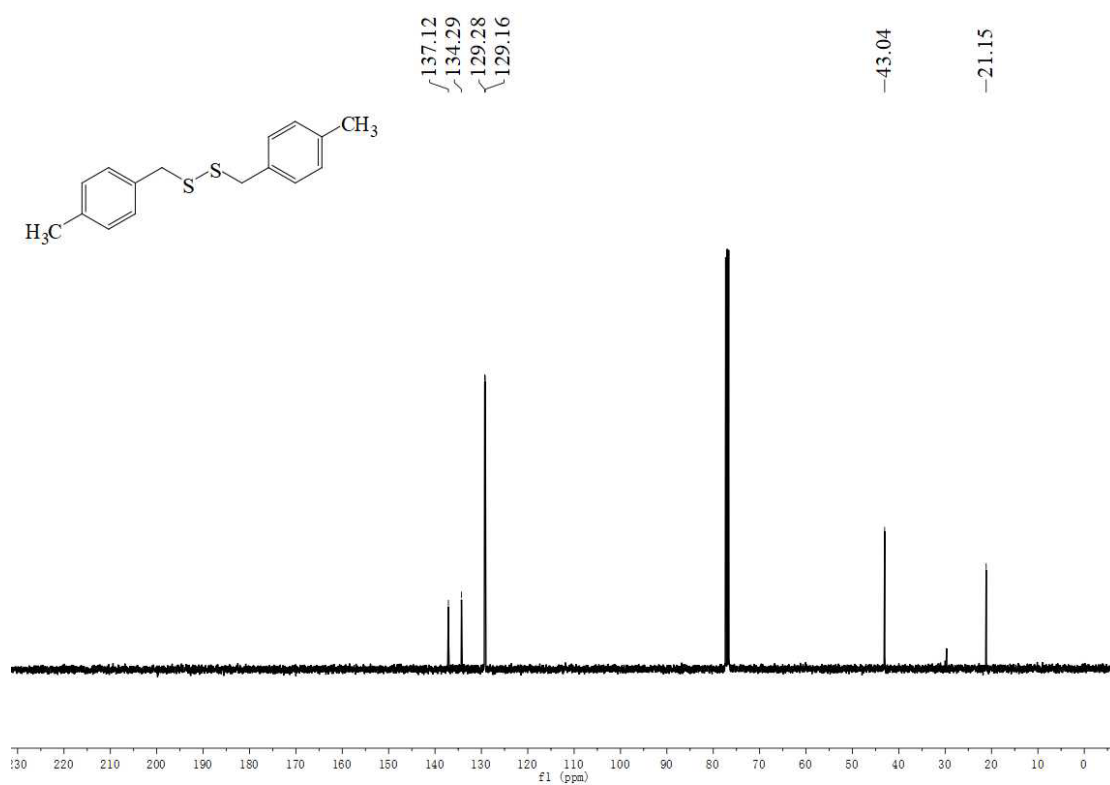


¹³C NMR (CDCl₃, 100 MHz) of 2t

1,2-bis(4-methylbenzyl)disulfane (2u)

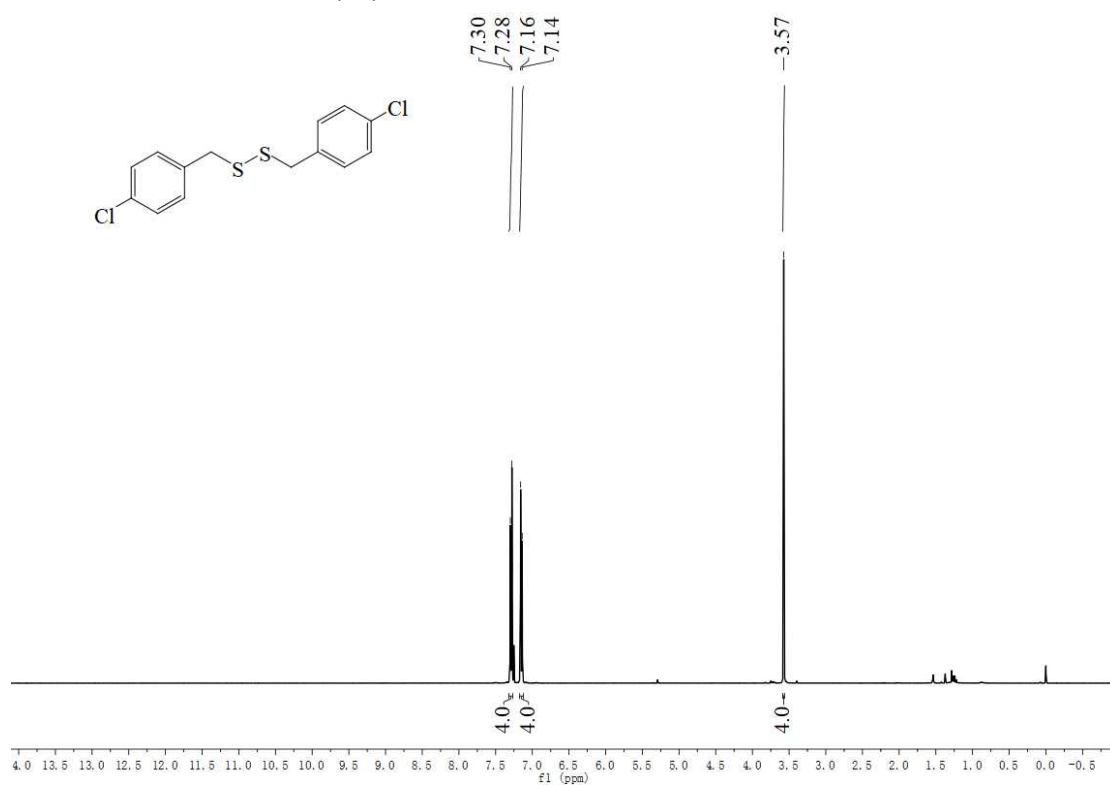


¹H NMR (CDCl₃, 400 MHz) of 2u

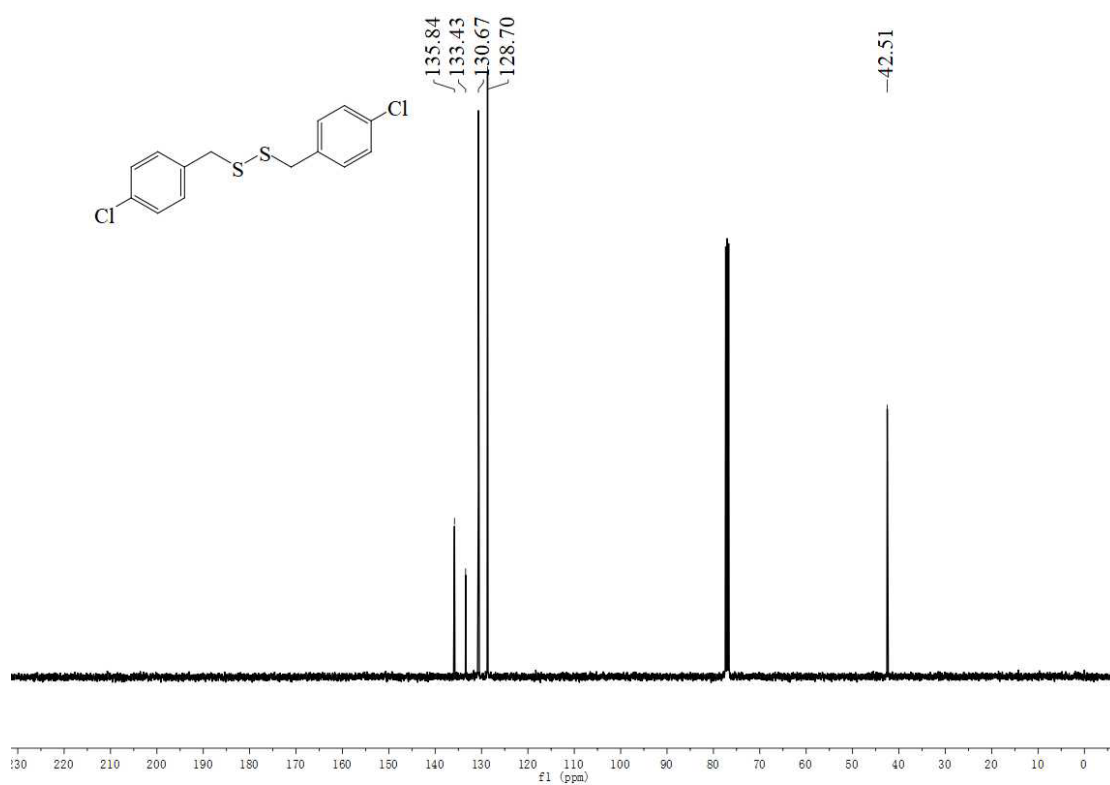


¹³C NMR (CDCl₃, 100 MHz) of 2u

Bis(4-chlorobenzyl)disulfide (2v)

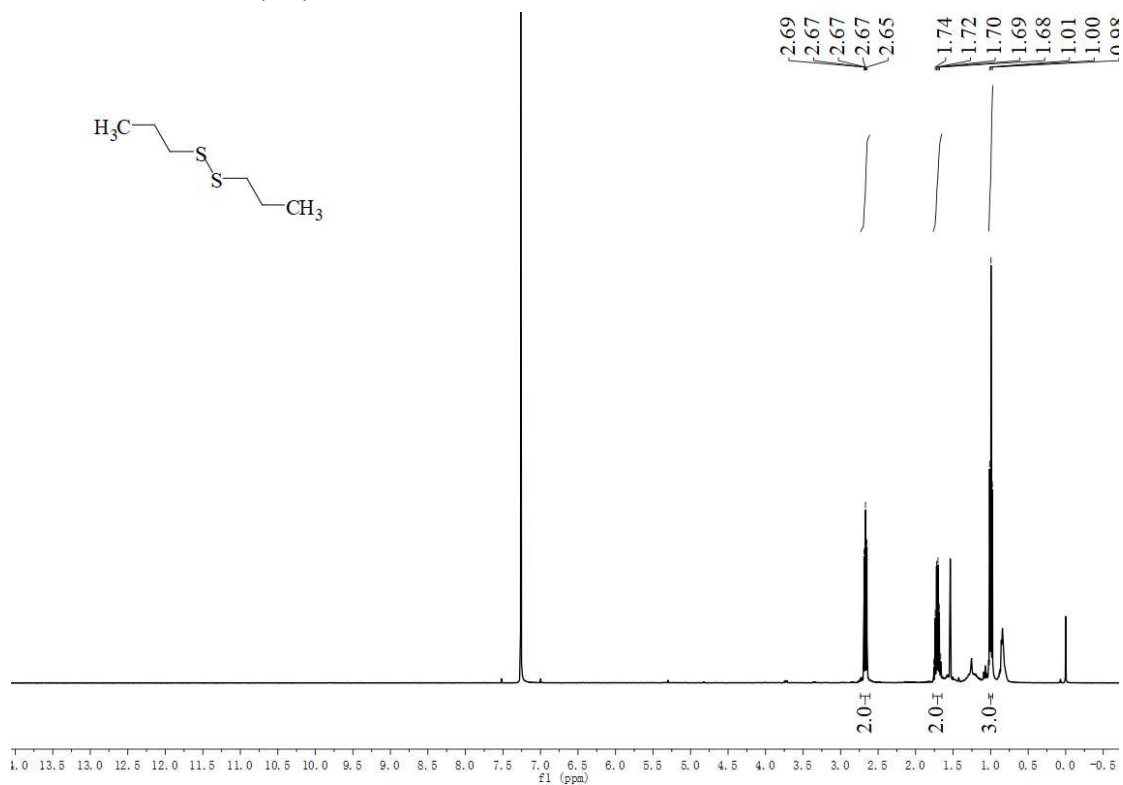


¹H NMR (CDCl₃, 400 MHz) of **2v**

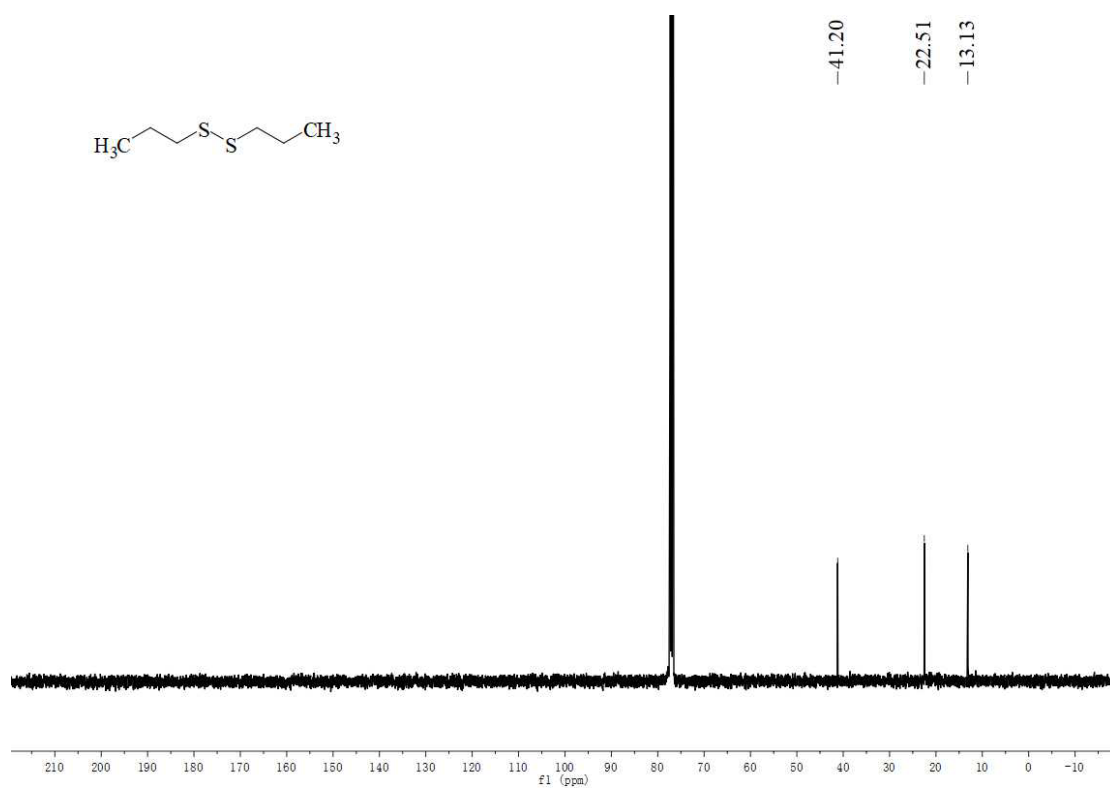


¹³C NMR (CDCl₃, 100 MHz) of **2v**

1,2-dipropyldisulfane (2w)

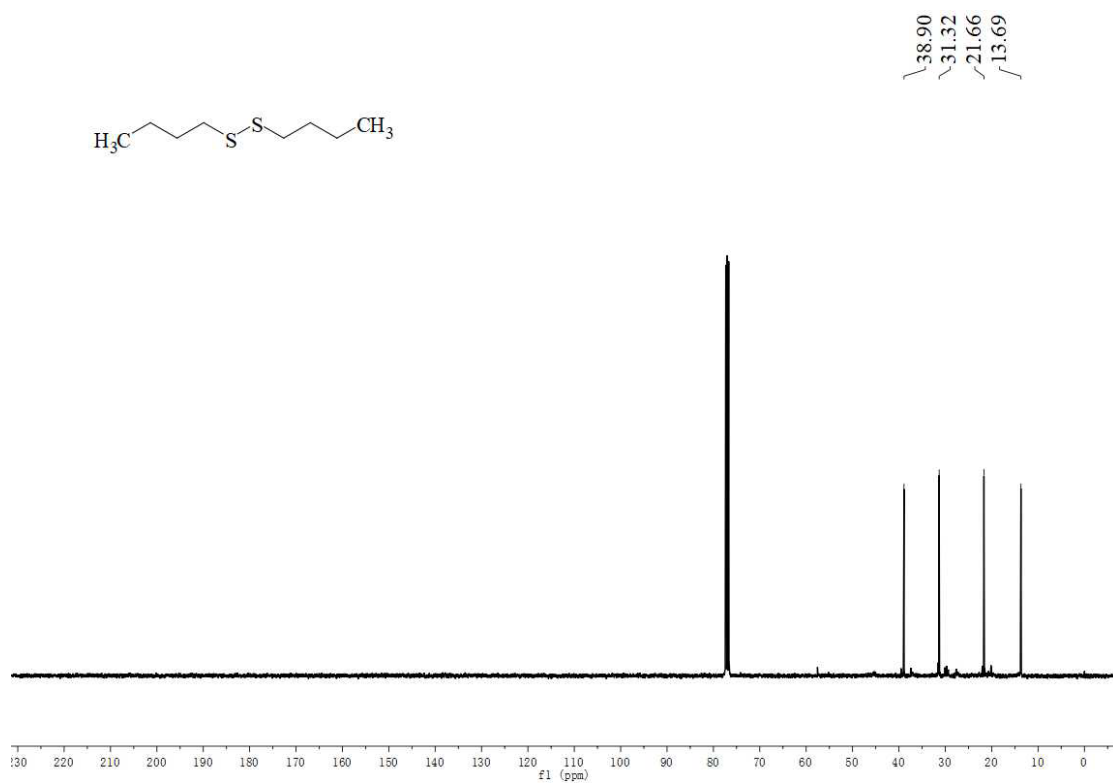
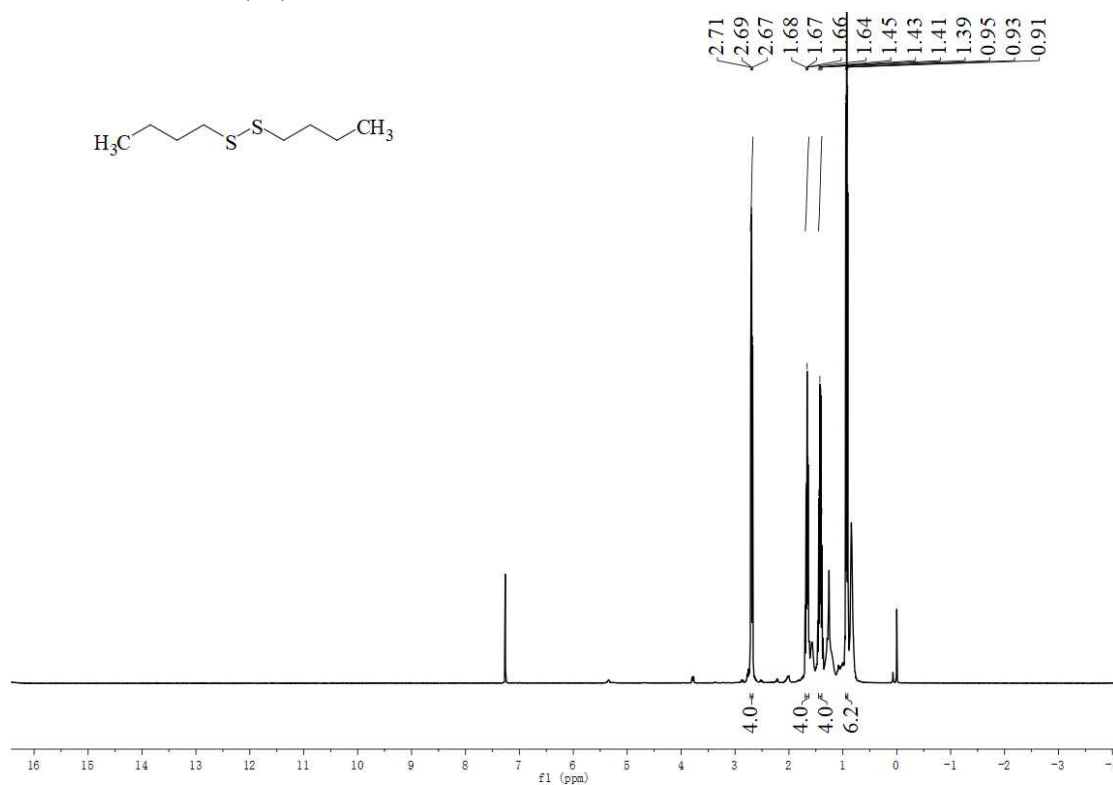


¹H NMR (CDCl₃, 400 MHz) of **2w**

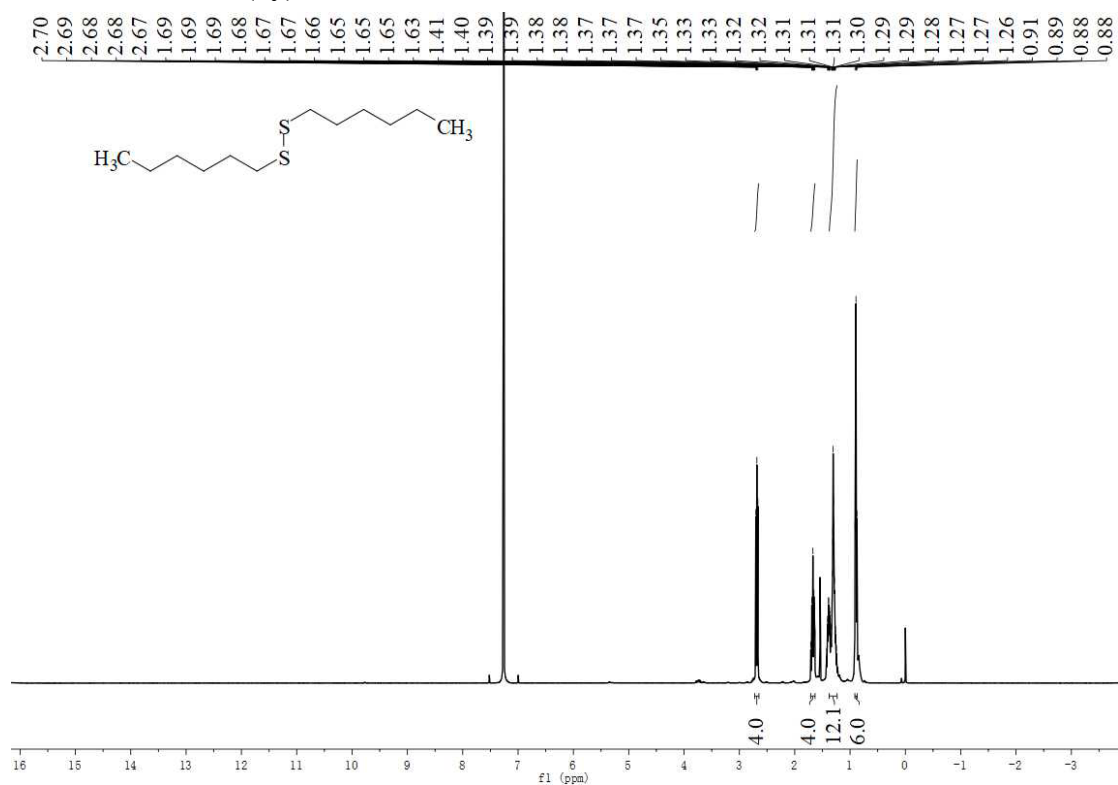


¹³C NMR (CDCl₃, 100 MHz) of **2w**

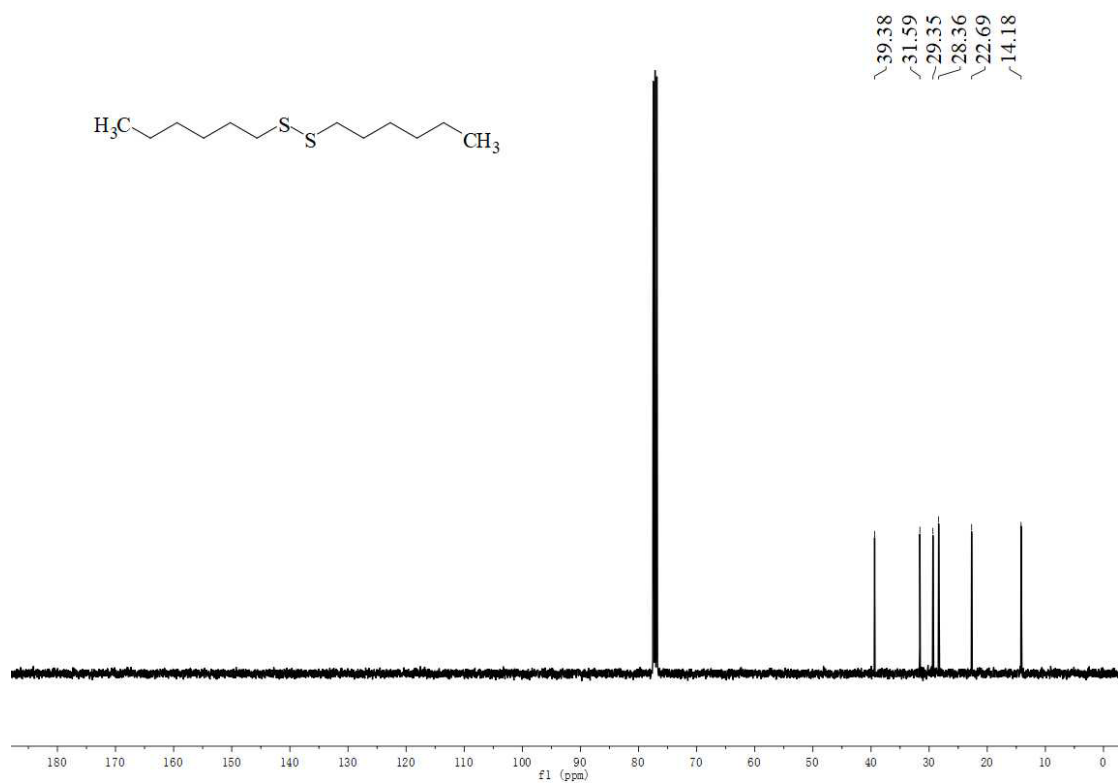
1,2-dibutylidisulfane (2x)



1,2-dihexyldisulfane (2y)

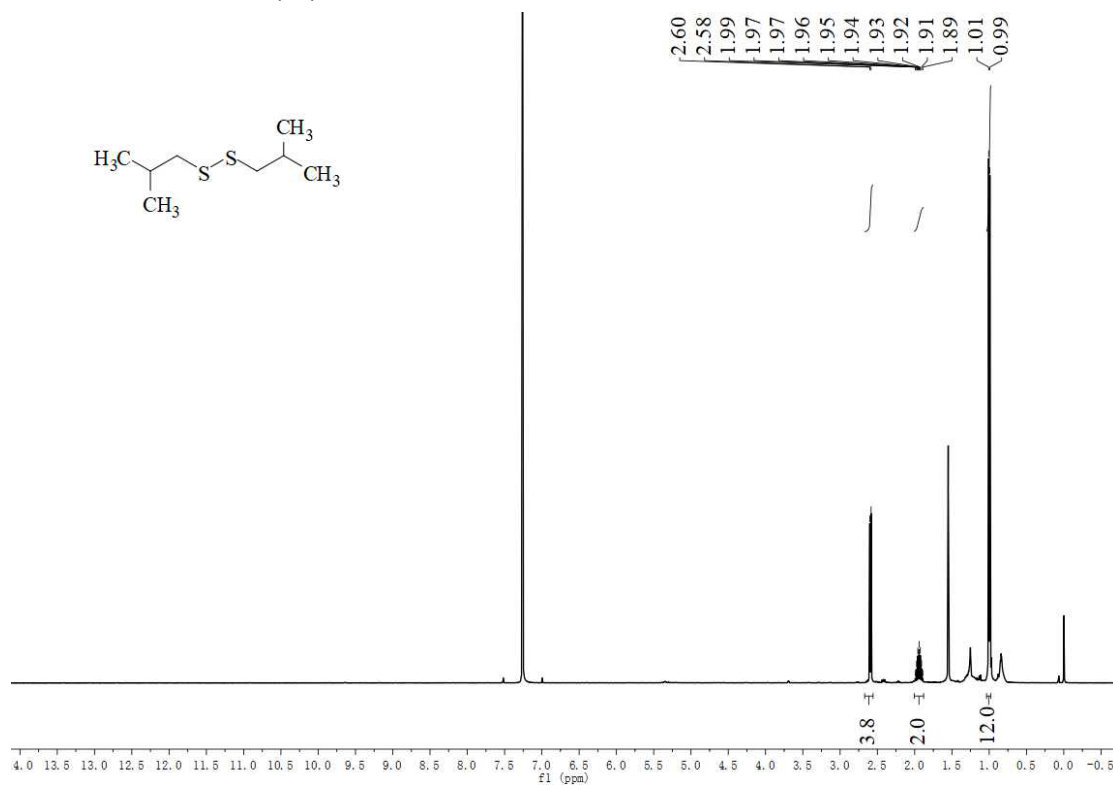


$^1\text{H NMR}$ (CDCl₃, 400 MHz) of **2y**

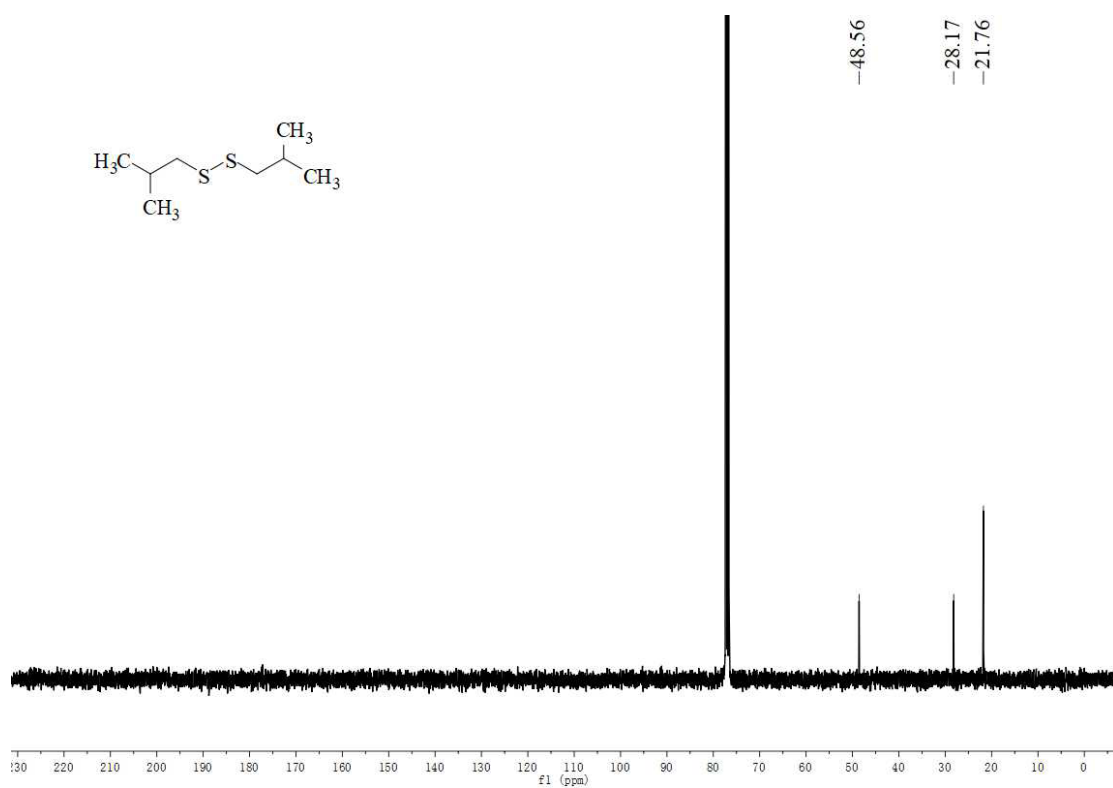


$^{13}\text{C NMR}$ (CDCl₃, 100 MHz) of **2y**

1,2-diisobutyldisulfane (2z)



¹H NMR (CDCl₃, 400 MHz) of **2z**



¹³C NMR (CDCl₃, 100 MHz) of **2z**