

Supporting Information

Synthesis of unsymmetrical disulfides *via* PPh₃-mediated reductive coupling of thiophenols with sulfonyl chlorides

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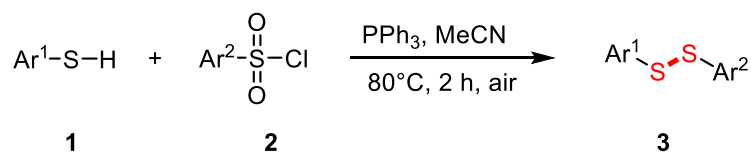
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1. General Information

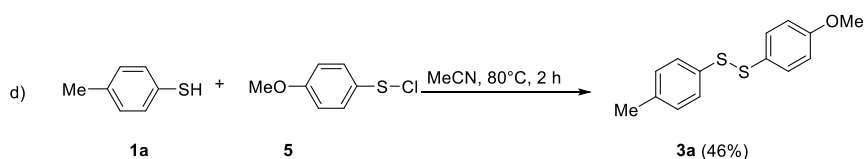
All the solvents and reagents were available from commercial sources and used without purification unless stated otherwise. ^1H and ^{13}C NMR spectra were gathered on Bruker Avance 400 MHz NMR spectrometers using CDCl_3 or $\text{DMSO-}d_6$. Chemical shifts are reported in parts per million (ppm). Reference peaks for CDCl_3 in ^1H NMR and ^{13}C NMR spectra were set at 7.26 and 77.0 ppm. For $\text{DMSO-}d_6$, the reference peaks were set as follows: ^1H NMR: DMSO at 2.50 ppm; ^{13}C NMR: DMSO at 40.0 ppm. Data are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. High-resolution mass spectras (HRMS) were obtained on Waters GCT premier of EI. X-ray was obtained on Gemini A Ultra with Atlas CCD.

2. General procedure



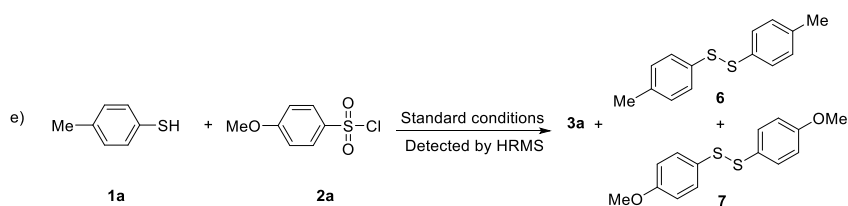
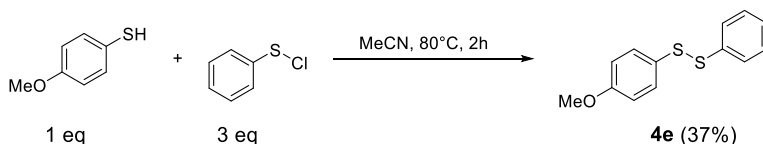
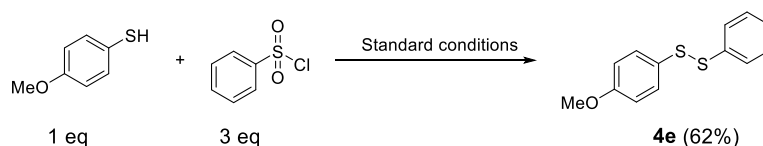
Add PPh_3 (3 mmol, 6 equiv) to the solution of aryl thiol **1** (0.5 mmol, 1 equiv) and aryl sulfonyl chloride **2** (1.5 mmol, 3 equiv) in MeCN (2.0 mL). The reaction was stirred at 80°C for 2 h. After the reaction is completed, remove the solvent and purified by column chromatography (DCM/hexane = 1/20 – 1/10) to offer the product **3**.

3. Control Experiments d and e



Under an atmosphere of N₂, *N*-chlorosuccinimide (1.46 g, 11 mmol) was placed in a 50-mL reaction flask and dissolved in dichloromethane (25 mL). 4-methoxybenzenethiol (1.2 mL, 10.0 mmol) was added slowly at 0 °C and the reaction mixture was stirred at 0 °C for 15 min. After the solvent was removed, hexane (15 mL) was added to the residue. The resulting white precipitate of succinimide was filtrated. The hexane was removed by distillation under reduced pressure to obtain the crude material **5** for the experiment d without further purification.¹

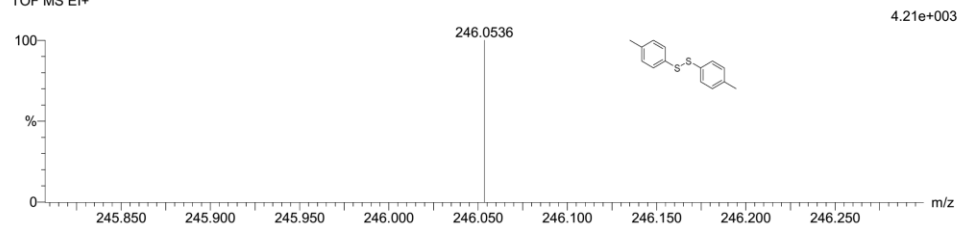
In order to confirm the credibility of the results of experiment f, we conducted following experiments using commercial materials phenylsulfenylchloride.



Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 70 best isotopic matches for each mass)

Elements Used:
C: 0-100 H: 0-200 S: 0-2
WDG1064 737 (4.884)
TOF MS EI+

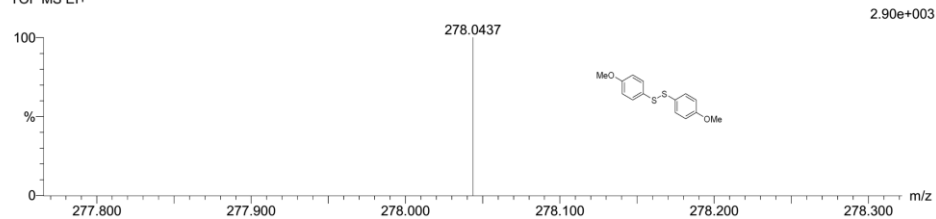


Minimum:				-1.5		
Maximum:	1.0	10.0		50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
246.0536	246.0537	-0.1	-0.4	8.0	5548111.0	C14 H14 S2

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

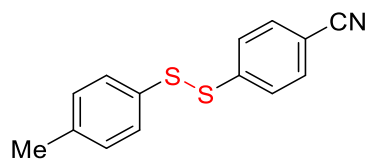
Monoisotopic Mass, Odd and Even Electron Ions
43 formula(e) evaluated with 1 results within limits (up to 70 best isotopic matches for each mass)

Elements Used:
C: 0-100 H: 0-200 O: 0-3 S: 0-2
WDG1064 888 (5.689)
TOF MS EI+

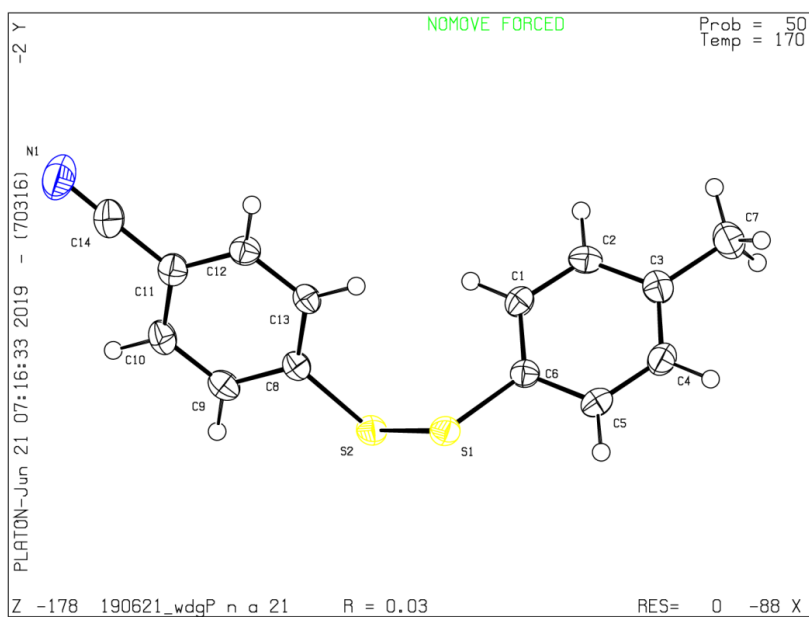


Minimum:				-1.5		
Maximum:	1.0	10.0		50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
278.0437	278.0435	0.2	0.7	8.0	5547455.5	C14 H14 O2 S2

4. X-ray of compound 3t



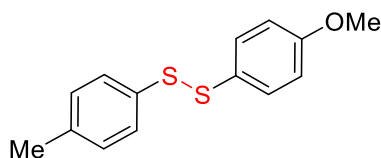
The structure of Compound 3t



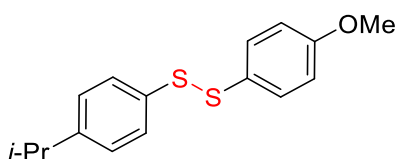
X-ray of compound 3t

CCDC: 1938235

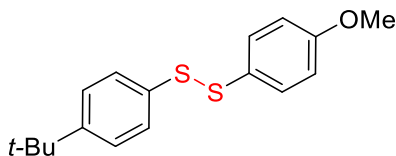
5. Spectra Data



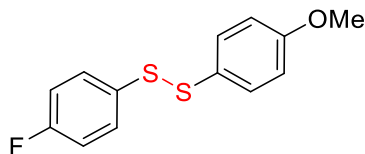
1-(4-methoxyphenyl)-2-(p-tolyl)disulfane (3a)², Yellow oil. 97 mg, 74%. ¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.38 (m, 4H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 3.79 (s, 3H), 2.34 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.8, 137.6, 134.0, 131.9, 129.8, 129.2, 128.3, 114.6, 55.3, 21.1. HRMS (EI) *m/z* calcd for C₁₄H₁₄OS₂⁺ 262.0486 [M⁺], found 262.0484.



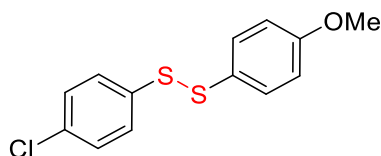
1-(4-isopropylphenyl)-2-(4-methoxyphenyl)disulfane (3b), Yellow oil. 91 mg, 63%. ¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.39 (m, 4H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 3.80 (d, *J* = 4.0 Hz, 3H), 2.93 – 2.85 (m, 1H), 1.24 (d, *J* = 2.4 Hz, 3H), 1.22 (d, *J* = 2.4 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.9 159.7, 148.5 148.3, 134.4 134.2, 132.6 131.7, 128.9 128.2, 128.4 128.3, 127.2 127.2, 114.6 114.6, 55.4, 33.7 33.7, 23.9. HRMS (EI) *m/z* calcd for C₁₆H₁₈OS₂⁺ 290.0799 [M⁺], found 290.0798.



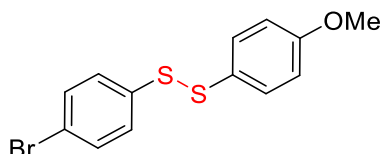
1-(4-(tert-butyl)phenyl)-2-(4-methoxyphenyl)disulfane (3c), Yellow oil. 104 mg, 68%. ¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.42 (m, 4H), 7.34 (d, *J* = 12.0 Hz, 2H), 6.85 (d, *J* = 8.0 Hz, 2H), 3.80 (s, 3H), 1.31 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 159.7, 150.7, 134.1, 131.6, 128.5, 128.3, 126.1, 114.6, 55.4, 34.6, 31.3. HRMS (EI) *m/z* calcd for C₁₇H₂₀OS₂⁺ 304.0956 [M⁺], found 304.0954.



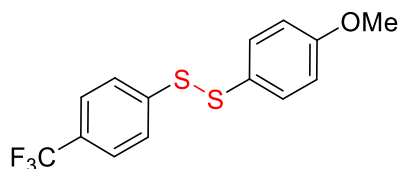
1-(4-fluorophenyl)-2-(4-methoxyphenyl)disulfane (3d), Yellow oil. 93 mg, 70%. ¹H NMR (400 MHz, CDCl₃) δ 7.48 – 7.44 (m, 4H), 7.41 (d, *J* = 12.0 Hz, 2H), 7.01 (t, *J* = 12.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 3.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 162.5 (d, *J* = 246 Hz), 160.0, 132.7 (d, *J* = 3.0 Hz), 132.4, 131.5 (d, *J* = 8.0 Hz), 127.8, 116.1 (d, *J* = 22.0 Hz), 114.7, 55.3. HRMS (EI) *m/z* calcd for C₁₃H₁₁FOS₂⁺ 266.0235 [M⁺], found 266.0236.



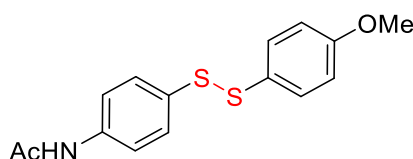
1-(4-chlorophenyl)-2-(4-methoxyphenyl)disulfane (3e)³, Yellow oil. 99 mg, 70%. ¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.38 (m, 4H), 7.28 – 7.25 (m, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 3.78 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 160.0, 136.0, 133.3, 132.1, 129.8, 129.1, 127.5, 114.8, 55.4. HRMS (EI) *m/z* calcd for C₁₃H₁₁ClOS₂⁺ 281.9940 [M⁺], found 281.9940.



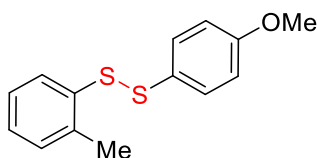
1-(4-bromophenyl)-2-(4-methoxyphenyl)disulfane (3f), Yellow oil. 102 mg, 63%. ¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.36 (m, 6H), 6.84 (d, *J* = 12.0 Hz, 2H), 3.79 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 160.0, 136.7, 132.0, 132.0, 129.8, 127.4, 121.2, 114.7, 55.3. HRMS (EI) *m/z* calcd for C₁₃H₁₁BrOS₂⁺ 325.9435 [M⁺], found 325.9433.



1-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)disulfane (3g)⁴, Yellow oil. 100 mg, 63%. ¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, *J* = 8.0 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 12.0 Hz, 2H), 3.79 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 160.1, 142.3, 131.8, 128.9 (d, *J* = 32.0 Hz), 127.0, 126.9, 125.8 (q, *J* = 4.0 Hz), 124.0 (q, *J* = 270.0 Hz), 114.9, 55.4. HRMS (EI) *m/z* calcd for C₁₄H₁₁F₃OS₂⁺ 316.0203 [M⁺], found 316.0202.

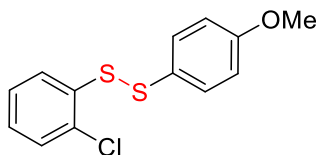


N-(4-((4-methoxyphenyl)disulfanyl)phenyl)acetamide (3h), Yellow oil. 91 mg, 62%. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.08 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 4H), 6.94 (d, *J* = 8.0 Hz, 2H), 3.74 (s, 3H), 2.04 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.5, 159.7, 139.4, 132.1, 130.1, 129.5, 126.8, 119.7, 115.0, 55.3, 24.0. HRMS (EI) *m/z* calcd for C₁₅H₁₅NO₂S₂⁺ 305.0544 [M⁺], found 305.0543.

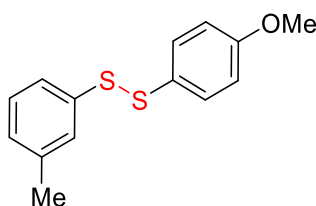


1-(4-methoxyphenyl)-2-(*o*-tolyl)disulfane (3i), Yellow oil. 86 mg, 66%. ¹H NMR (400 MHz, CDCl₃) δ 7.63 – 7.62 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 4.0 Hz, 3H), 6.83 (d, *J* = 8.0 Hz, 2H), 3.79 (s, 3H), 2.40 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.8,

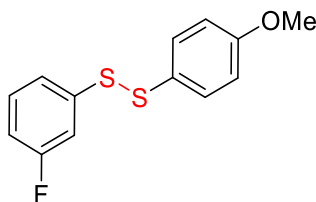
137.6, 135.9, 132.1, 130.3, 129.2, 127.8, 127.4, 126.6, 114.6, 55.3, 20.1. HRMS (EI) m/z calcd for $C_{14}H_{14}OS_2^+$ 262.0486 [M^+], found 262.0485.



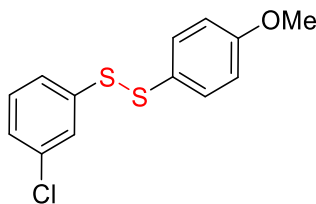
1-(2-chlorophenyl)-2-(4-methoxyphenyl)disulfane (3j), Yellow oil. 86 mg, 61%. 1H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, $J = 8.0$ Hz, 1H), 7.44 (d, $J = 8.0$ Hz, 2H), 7.35 (d, $J = 12.0$ Hz, 1H), 7.29 – 7.24 (m, 1H), 7.18 – 7.14 (m, 1H), 6.84 (d, $J = 12.0$ Hz, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 135.8, 131.9, 131.5, 129.7, 127.8, 127.6, 127.3, 126.9, 114.8, 55.3. HRMS (EI) m/z calcd for $C_{13}H_{11}ClOS_2^+$ 281.9940 [M^+], found 281.9940.



1-(4-methoxyphenyl)-2-(m-tolyl)disulfane (3k), Yellow oil. 90 mg, 69%. 1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.21 (t, $J = 8.0$ Hz, 1H), 7.05 (d, $J = 8.0$ Hz, 1H), 6.84 (d, $J = 8.0$ Hz, 2H), 3.79 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 138.8, 137.2, 131.7, 128.8, 128.7, 128.1, 128.1, 125.2, 114.6, 55.3, 21.4. HRMS (EI) m/z calcd for $C_{14}H_{14}OS_2^+$ 262.0486 [M^+], found 262.0486.

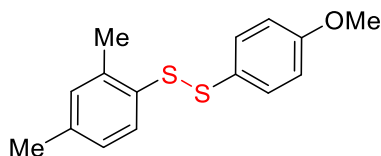


1-(3-fluorophenyl)-2-(4-methoxyphenyl)disulfane (3l), Yellow oil. 85 mg, 64%. 1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, $J = 8.0$ Hz, 2H), 7.30 – 7.25 (m, 3H), 6.94 – 6.90 (m, 1H), 6.84 (d, $J = 8.0$ Hz, 2H), 3.79 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (d, $J = 247.0$ Hz), 160.0, 139.9 (d, $J = 7.0$ Hz), 131.9, 130.3 (d, $J = 8.0$ Hz), 127.4, 123.1 (d, $J = 3.0$ Hz), 114.8, 114.4 (d, $J = 24.0$ Hz), 114.0 (d, $J = 21.0$ Hz), 55.4. HRMS (EI) m/z calcd for $C_{13}H_{11}FOS_2^+$ 266.0235 [M^+], found 266.0236.

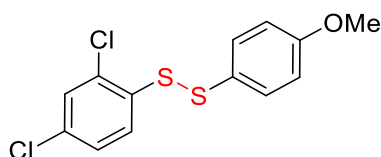


1-(3-chlorophenyl)-2-(4-methoxyphenyl)disulfane (3m), Yellow oil. 94 mg, 67%. 1H NMR (400 MHz, $CDCl_3$) δ 7.53 (s, 1H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.38 (d, $J = 8.0$ Hz,

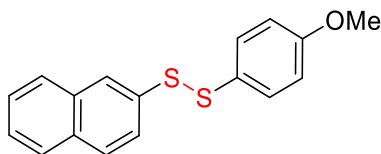
1H), 7.26 – 7.18 (m, 2H), 6.85 (d, $J = 12.0$ Hz, 2H), 3.79 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.0, 139.5, 134.9, 132.0, 130.0, 127.4, 127.3, 127.2, 125.7, 114.8, 55.3. HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{11}\text{ClOS}_2^+$ 281.9940 [M^+], found 281.9942.



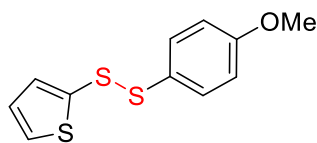
1-(2,4-dimethylphenyl)-2-(4-methoxyphenyl)disulfane (3n), Yellow oil. 92 mg, 67%. ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.40 (m, 3H), 7.02 – 6.96 (m, 2H), 6.83 (d, $J = 8.0$ Hz, 2H), 3.80 (s, 3H), 2.37 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.8, 138.3, 138.0, 132.5, 132.4, 131.3, 130.7, 128.1, 127.3, 114.5, 55.3, 21.0, 20.2. HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{16}\text{OS}_2^+$ 276.0643 [M^+], found 276.0644.



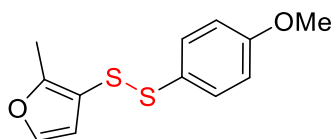
1-(2,4-dichlorophenyl)-2-(4-methoxyphenyl)disulfane (3o), Yellow oil. 106 mg, 67%. ^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 8.0$ Hz, 1H), 7.40 (d, $J = 8.0$ Hz, 2H), 7.35 (d, $J = 4.0$ Hz, 1H), 7.25 – 7.21 (m, 1H), 6.82 (d, $J = 8.0$ Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.0, 134.6, 133.0, 132.7, 131.8, 129.4, 129.0, 127.6, 126.5, 114.8, 55.4. HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{10}\text{ClOS}_2^+$ 315.9550, [M^+], found 315.9553.



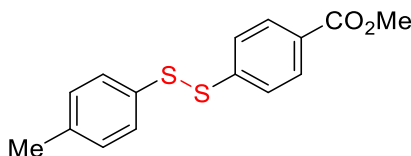
1-(4-methoxyphenyl)-2-(naphthalen-2-yl)disulfane (3p)³, Yellow oil. 98 mg, 66%. ^1H NMR (400 MHz, CDCl_3) δ 7.96 (s, 1H), 7.83 – 7.75 (m, 3H), 7.63 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.50 – 7.44 (m, 4H), 6.82 (d, $J = 8.0$ Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.9, 134.7, 133.4, 132.5, 132.0, 128.8, 127.9, 127.7, 127.5, 127.0, 126.7, 126.2, 126.2, 114.7, 55.3. HRMS (EI) m/z calcd for $\text{C}_{17}\text{H}_{14}\text{OS}_2^+$ 298.0486, [M^+], found 298.0488.



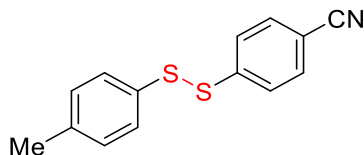
2-((4-methoxyphenyl)disulfaneyl)thiophene (3q), Yellow oil. 89 mg, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.42 (m, 3H), 7.10 (dd, $J = 3.6, 1.2$ Hz, 1H), 6.97 (dd, $J = 5.3, 3.6$ Hz, 1H), 6.88 (d, $J = 8.0$ Hz, 2H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.4, 136.5, 134.5, 134.3, 131.3, 127.9, 127.5, 114.6, 55.3. HRMS (EI) m/z calcd for $\text{C}_{11}\text{H}_{10}\text{OS}_3^+$ 253.9894, [M^+], found 253.9892.



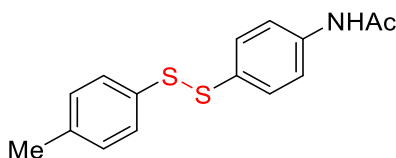
3-((4-methoxyphenyl)disulfaneyl)-2-methylfuran (3r), Yellow oil. 78 mg, 62%. ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, *J* = 8.0 Hz, 2H), 7.26 (s, 1H), 6.84 (d, *J* = 8.0 Hz, 2H), 6.35 (d, *J* = 2.0 Hz, 1H), 3.81 (s, 3H), 2.08 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 160.3, 156.5, 140.8, 134.4, 128.1, 114.5, 114.4, 113.1, 55.4, 11.6. HRMS (EI) *m/z* calcd for C₁₂H₁₂O₂S₂⁺ 252.0279, [M⁺], found 252.0277.



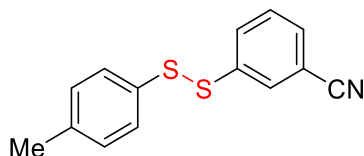
methyl 4-(p-tolyldisulfaneyl)benzoate (3s), Yellow oil. 85 mg, 59%. ¹H NMR (400 MHz, CDCl₃) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 12.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 3.90 (s, 3H), 2.32 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.5, 143.4, 137.9, 132.7, 130.1, 130.0, 128.5, 128.4, 126.0, 52.1, 21.0. HRMS (EI) *m/z* calcd for C₁₅H₁₄O₂S₂⁺ 290.0435, [M⁺], found 290.0433.



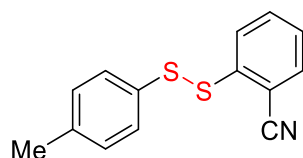
4-(p-tolyldisulfaneyl)benzonitrile (3t), White solid. 103 mg, 80%, mp: 74.7-76.5°C. ¹H NMR (400 MHz, CDCl₃) δ 7.61 – 7.55 (m, 4H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 144.1, 138.3, 132.4, 132.1, 130.1, 128.5, 126.4, 118.5, 109.9, 21.0. HRMS (EI) *m/z* calcd for C₁₄H₁₁NS₂⁺ 257.0333, [M⁺], found 257.0336.



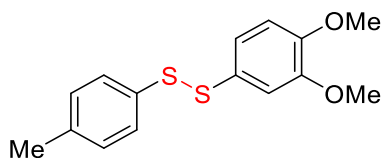
N-(4-(p-tolyldisulfaneyl)phenyl)acetamide (3u), Colorless oil. 65 mg, 50%. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.07 (s, 1H), 7.60 – 7.57 (m, 2H), 7.44 – 7.37 (m, 4H), 7.18 (d, *J* = 4.0 Hz, 2H), 2.27 (s, 3H), 2.03 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.5, 139.5, 139.4, 137.6, 137.5, 132.7, 132.6, 130.1, 130.0, 129.8, 129.3, 129.3, 128.5, 128.1, 119.7, 119.7, 24.0, 20.6. HRMS (EI) *m/z* calcd for C₁₅H₁₅NOS₂⁺ 289.0595, [M⁺], found 289.0596.



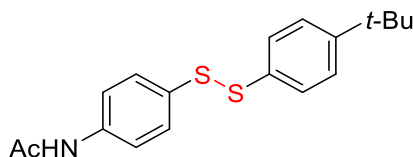
3-(p-tolyldisulfaneyl)benzonitrile (3v), Yellow oil. 87 mg, 68%. ¹H NMR (400 MHz, CDCl₃) δ 7.78 (t, *J* = 4.0 Hz, 1H), 7.72 – 7.69 (m, 1H), 7.50 – 7.48 (m, 1H), 7.42 – 7.37 (m, 3H), 7.13 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.6, 138.3, 132.2, 131.2, 130.3, 130.1, 129.6, 128.7, 118.2, 113.3, 21.1. HRMS (EI) *m/z* calcd for C₁₄H₁₁NS₂⁺ 257.0333, [M⁺], found 257.0333.



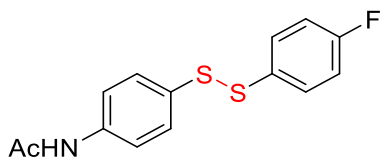
2-(p-tolyldisulfaneyl)benzonitrile (3w), Yellow oil. 104 mg, 81%. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.81 – 7.78 (m, 1H), 7.61 (dd, $J = 8.0, 1.0$ Hz, 1H), 7.54 (td, $J = 8.0, 1.4$ Hz, 1H), 7.38 (d, $J = 8.0$ Hz, 2H), 7.31 (td, $J = 8.0, 1.0$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 2.33 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.8, 138.4, 135.5, 133.1, 132.0, 130.0, 129.0, 128.4, 127.2, 116.3, 111.9, 21.1. HRMS (EI) m/z calcd for $\text{C}_{14}\text{H}_{11}\text{NS}_2^+$ 257.0333, $[\text{M}^+]$, found 257.0332.



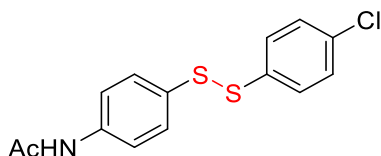
1-(3,4-dimethoxyphenyl)-2-(p-tolyl)disulfane (3x), Yellow oil. 103 mg, 71%. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.39 (d, $J = 8.0$ Hz, 2H), 7.12 (d, $J = 8.0$ Hz, 2H), 7.05 (dd, $J = 8.4, 2.0$ Hz, 1H), 7.00 (d, $J = 2.0$ Hz, 1H), 6.78 (d, $J = 8.0$ Hz, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 2.33 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 149.2, 149.1, 137.8, 134.0, 129.8, 129.5, 128.5, 122.8, 113.2, 111.3, 55.9, 55.9, 21.1. HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{16}\text{O}_2\text{S}_2^+$ 292.0592, $[\text{M}^+]$, found 292.0589.



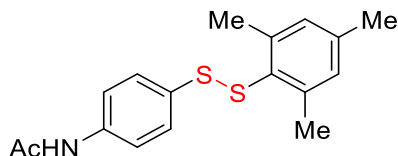
N-(4-((4-tert-butyl)phenyl)disulfaneyl)phenylacetamide (3y), White solid. 76 mg, 46%, mp: 163.7-165.3°C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.09 (s, 1H), 7.60 (d, $J = 8.0$ Hz, 2H), 7.46 – 7.37 (m, 6H), 2.04 (s, 3H), 1.23 (s, 10H). $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 168.5, 150.5, 139.3, 132.8, 129.4, 129.2, 127.9, 126.3, 119.7, 34.3, 31.0, 24.0. HRMS (EI) m/z calcd for $\text{C}_{18}\text{H}_{21}\text{NOS}_2^+$ 331.1065, $[\text{M}^+]$, found 331.1063.



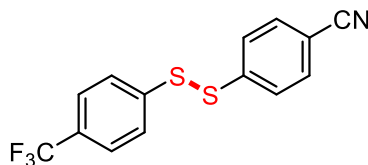
N-(4-((4-fluorophenyl)disulfaneyl)phenyl)acetamide (3z), White solid. 72 mg, 49%, mp: 90.5-92.1°C. $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.10 (s, 1H), 7.61 – 7.58 (m, 2H), 7.55 – 7.52 (m, 2H), 7.45 – 7.42 (m, 2H), 7.25 – 7.21 (m, 2H), 2.04 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 168.5, 161.9 (d, $J = 244.0$ Hz), 139.6, 131.9 (d, $J = 3.0$ Hz), 131.0 (d, $J = 9.0$ Hz), 130.1, 128.9, 119.8, 116.5 (d, $J = 22.0$ Hz), 24.0. HRMS (EI) m/z calcd for $\text{C}_{14}\text{H}_{12}\text{FNOS}_2^+$ 293.0344, $[\text{M}^+]$, found 293.0343.



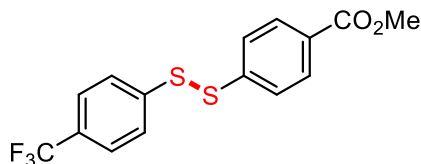
***N*-((4-(4-chlorophenyl)disulfaneyl)phenyl)acetamide (4a)**, White solid. 52 mg, 34%, mp: 116.3-118.0°C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.10 (s, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.54 – 7.51 (m, 2H), 7.46 – 7.43 (m, 4H), 2.03 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.5, 139.7, 135.2, 132.3, 130.0, 129.4, 129.3, 128.6, 119.8, 24.0. HRMS (EI) *m/z* calcd for C₁₄H₁₂ClNOS₂⁺ 309.0049, [M⁺], found 309.0045.



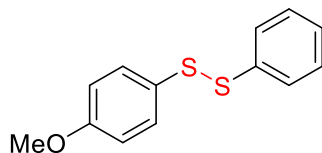
***N*-((4-(mesityldisulfaneyl)phenyl)acetamide (4b)**, White solid. 70 mg, 44%, mp: 150.2-152.1°C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.11 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.92 (s, 2H), 2.21 (s, 3H), 2.19 (s, 6H), 2.05 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.6, 142.2, 140.3, 139.4, 133.5, 131.1, 129.2, 129.3, 119.2, 24.1, 21.4, 20.7. HRMS (EI) *m/z* calcd for C₁₇H₁₉NOS₂⁺ 317.0908, [M⁺], found 317.0909.



4-((4-(trifluoromethyl)phenyl)disulfaneyl)benzonitrile (4c), White solid. 100 mg, 64%. mp: 65.7-67.3°C. ¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, *J* = 2.9 Hz, 4H), 7.57 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 142.6, 140.2, 132.7, 129.6 (q, *J* = 32.8 Hz), 126.6, 126.4, 126.2 (q, *J* = 3.8 Hz), 123.7 (q, *J* = 271 Hz), 118.2, 110.6. HRMS (EI) *m/z* calcd for C₁₄H₈F₃NS₂⁺ 311.0050, [M⁺], found 311.0049.



methyl 4-((4-(trifluoromethyl)phenyl)disulfaneyl)benzoate (4d), White solid. 104 mg, 60%. mp: 77.3-79.2°C. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, *J* = 8.6 Hz, 2H), 7.67–7.43 (m, 6H), 3.90 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.3, 141.9, 140.9, 130.3, 129.3 (d, *J* = 32.8 Hz), 128.9, 126.5, 126.03 (q, *J* = 3.8 Hz), 125.9, 123.8 (q, *J* = 270 Hz), 52.2. HRMS (EI) *m/z* calcd for C₁₅H₁₁F₃O₂S₂⁺ 344.0153, [M⁺], found 344.0155.

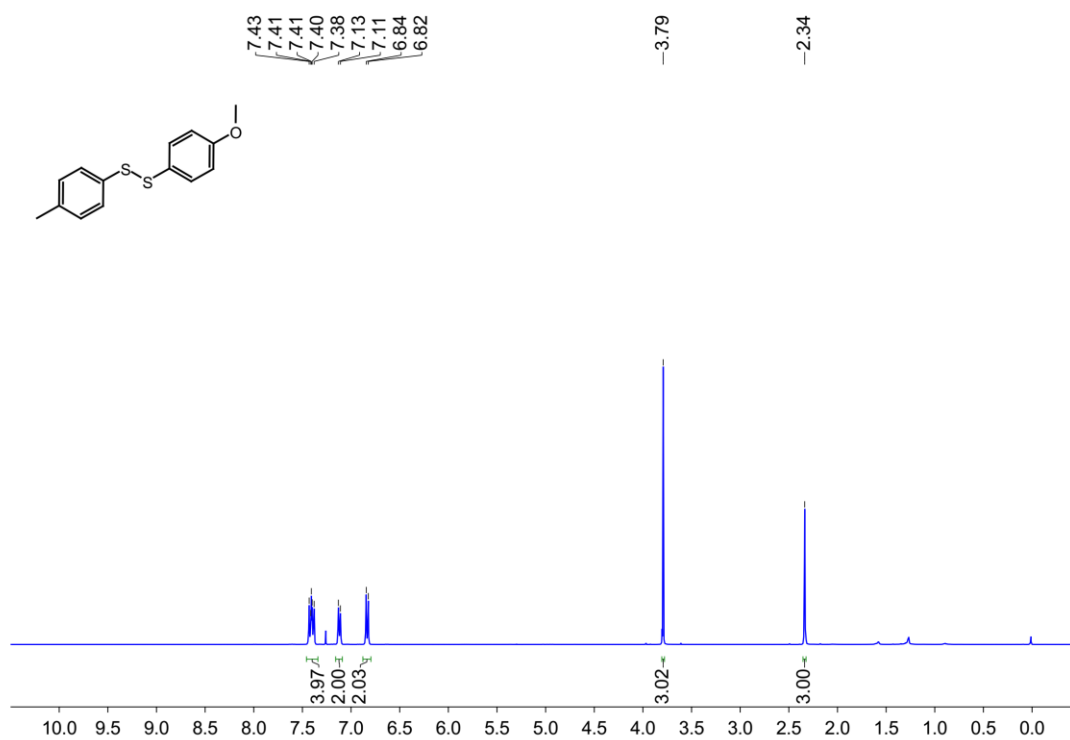


1-(4-methoxyphenyl)-2-phenyldisulfane (4e)⁵, Yellow oil. 77 mg, 62%. ¹H NMR (400 MHz, CDCl₃) δ 7.51-7.37 (m, 2H), 7.43-7.40 (m, 2H), 7.32-7.28 (m, 2H), 7.24-7.20 (m, 1H), 6.83-6.81 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 159.8, 137.4, 131.7, 129.0, 128.2, 128.0, 127.2, 114.7, 55.3.

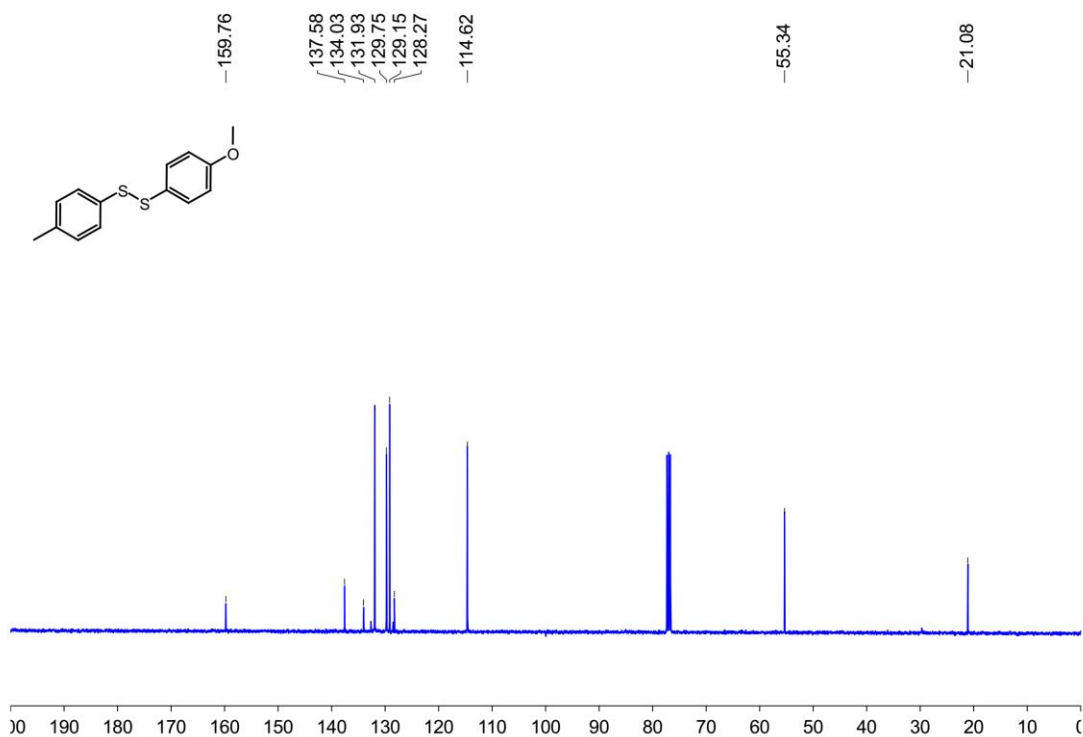
6. Reference

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2. Taniguchi, N. *Tetrahedron.* **2017**, *73*, 2030.
3. Dethe, D. H.; Srivastava, A.; Dherange, B. D.; Kumar, B. V. *Adv. Synth. Catal.* **2018**, *360*, 3020.
4. Xiao, X.; Feng, M.; Jiang, X. *Chem. Commun.* **2015**, *51*, 4208.
5. Ruan, H.; Meng, L -G.; Zhu, L.; Wang, L. *Adv. Synth. Catal.* **2019**, *361*, 3217.

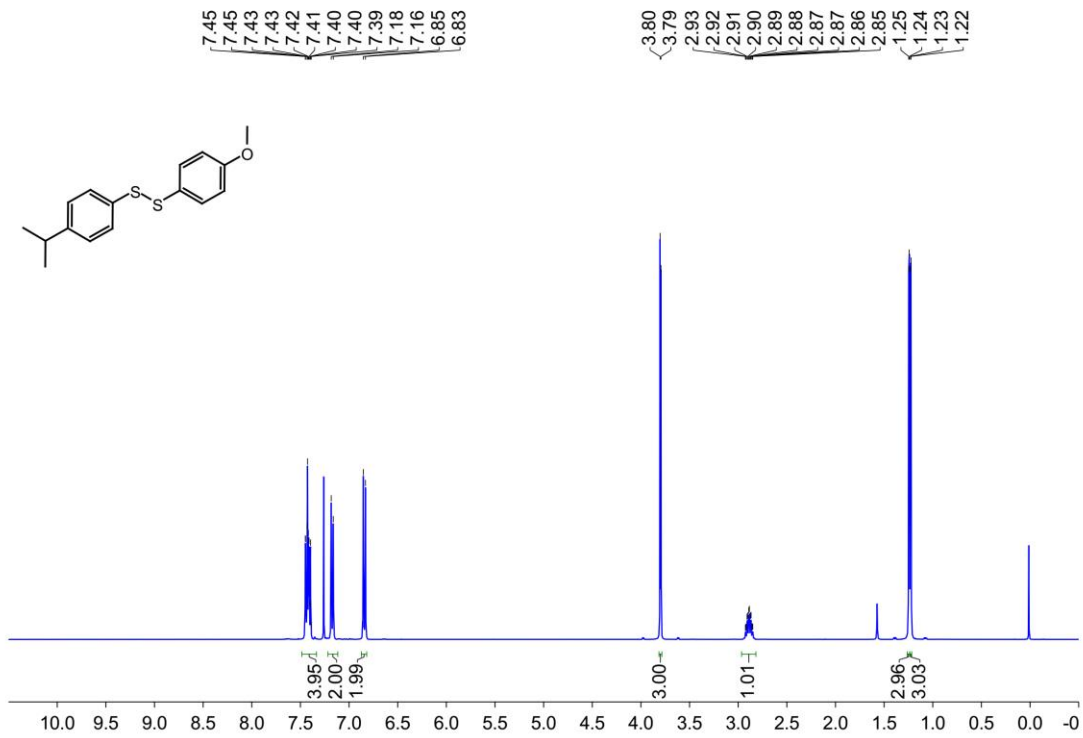
7. NMR Spectra for the Compounds



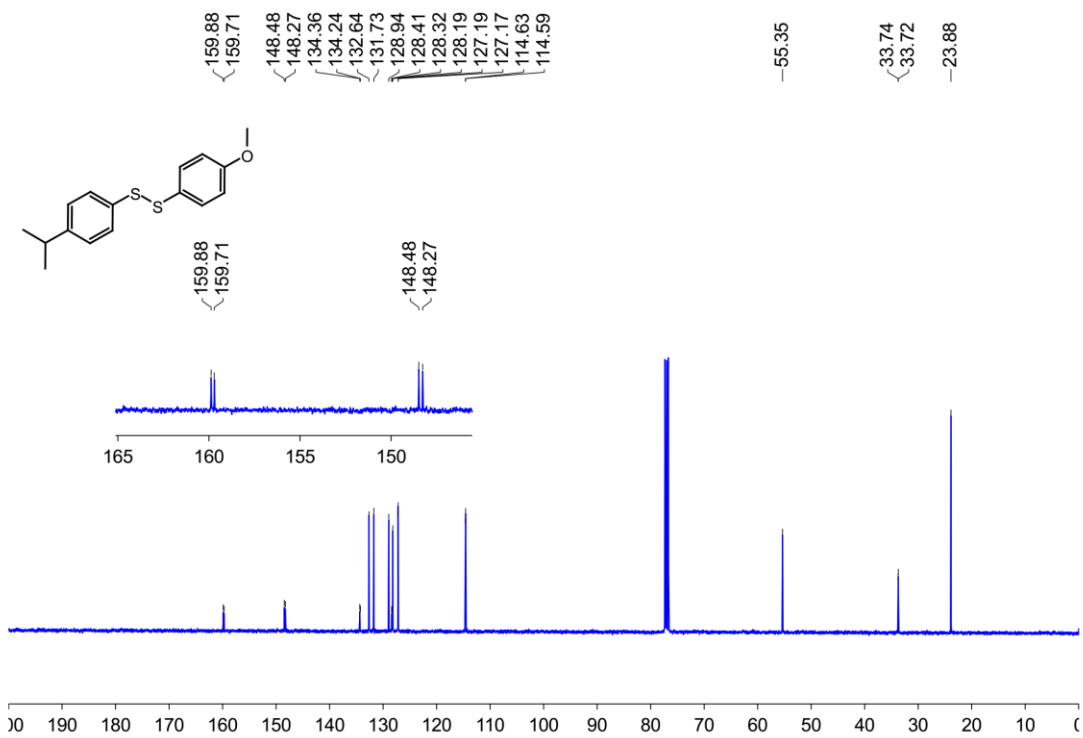
¹H NMR spectrum of compound 3a



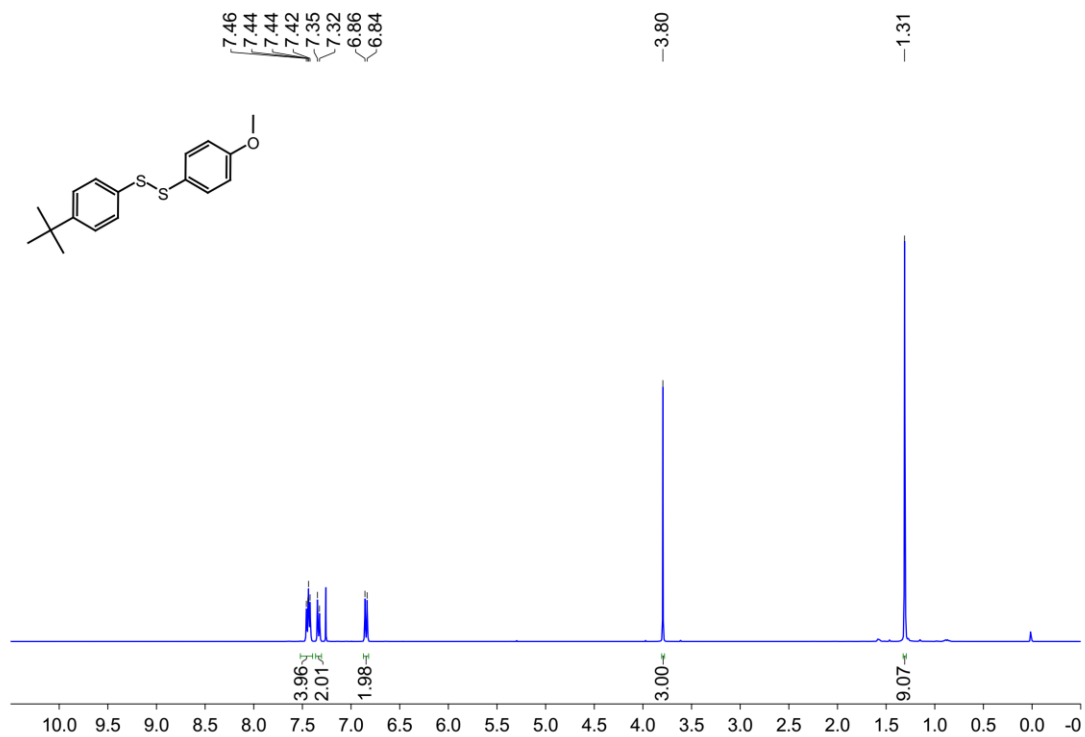
¹³C NMR spectrum of compound 3a



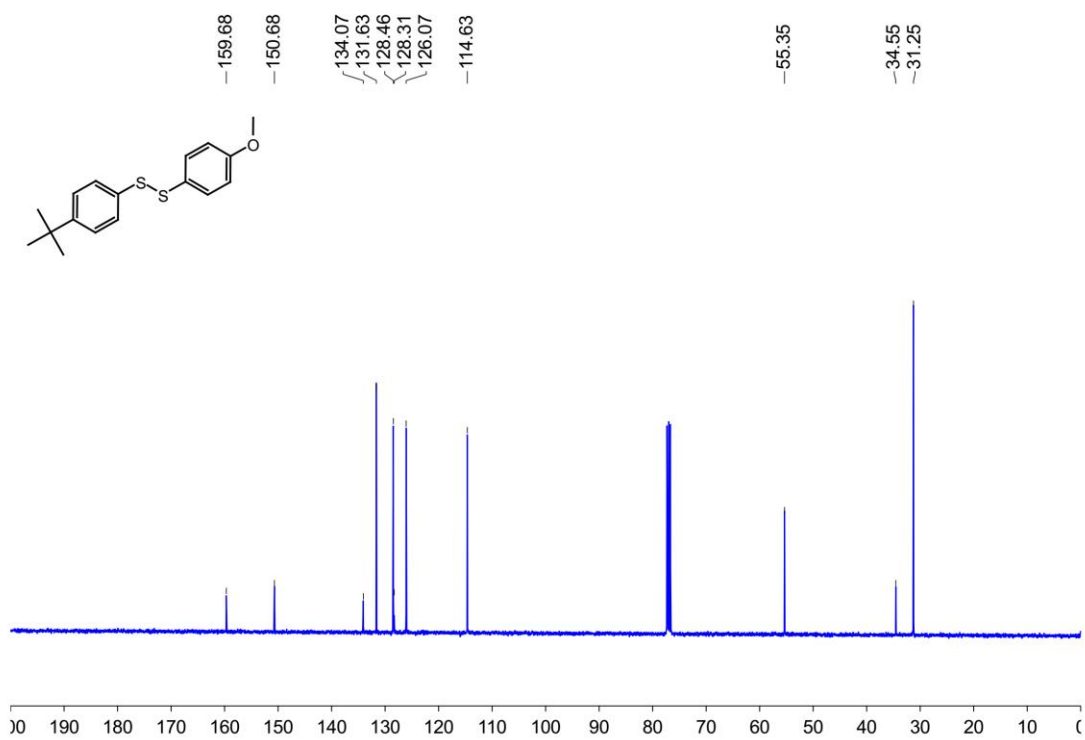
¹H NMR spectrum of compound **3b**



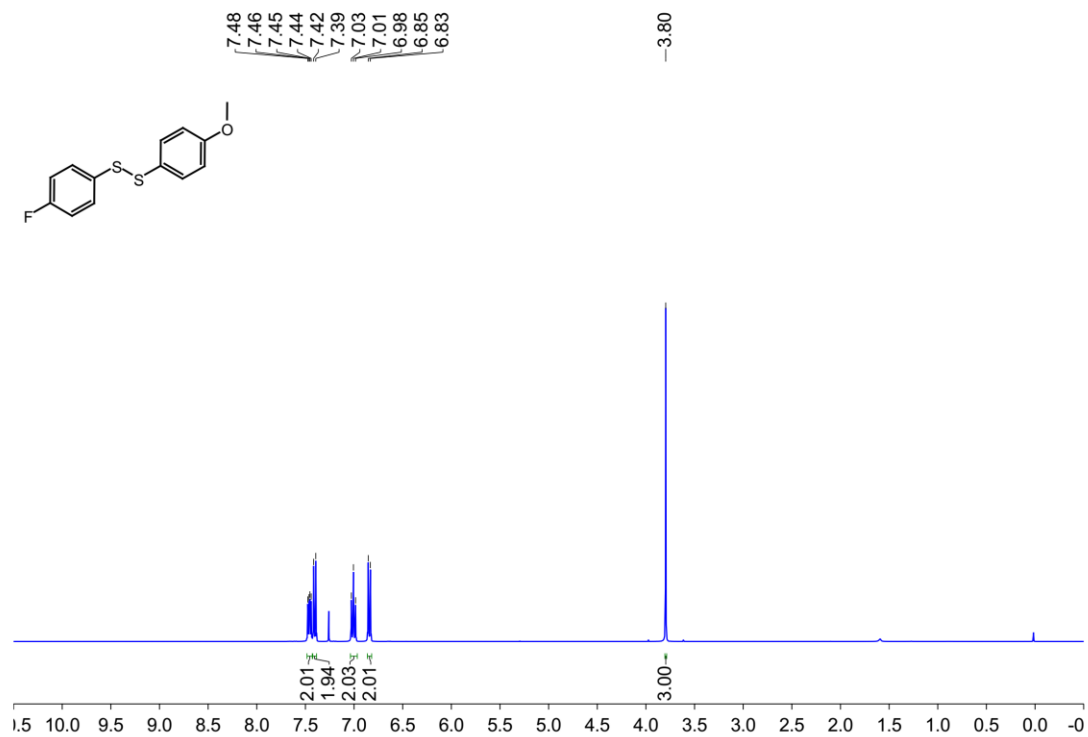
¹³C NMR spectrum of compound **3b**



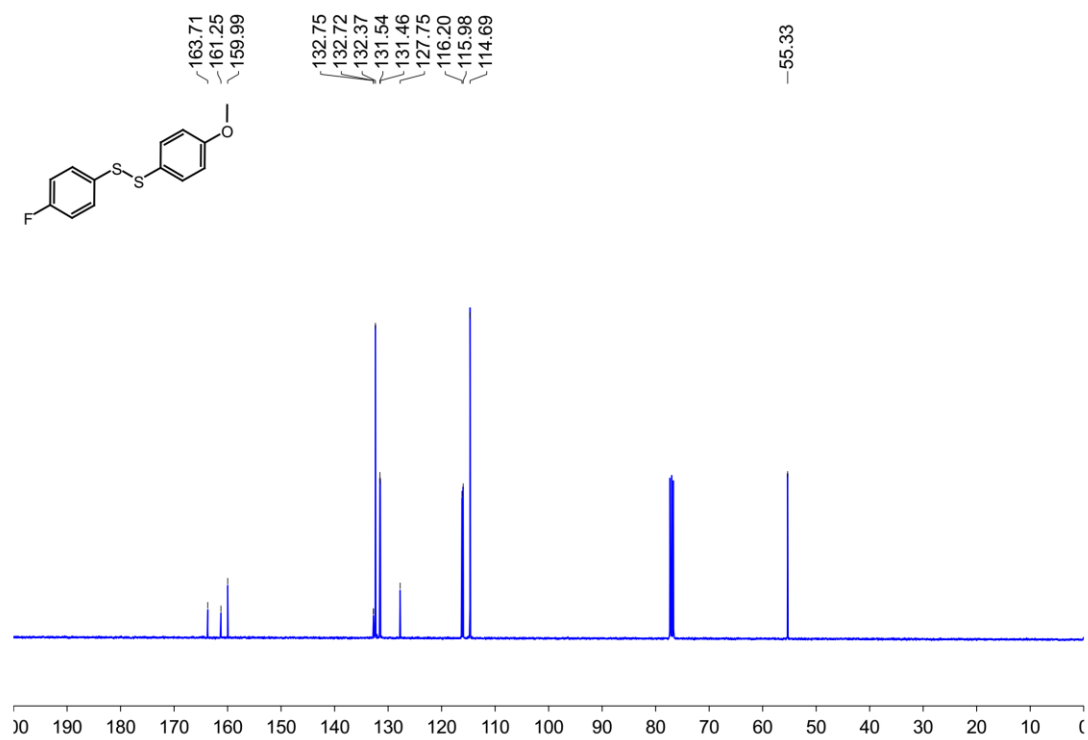
^1H NMR spectrum of compound **3c**



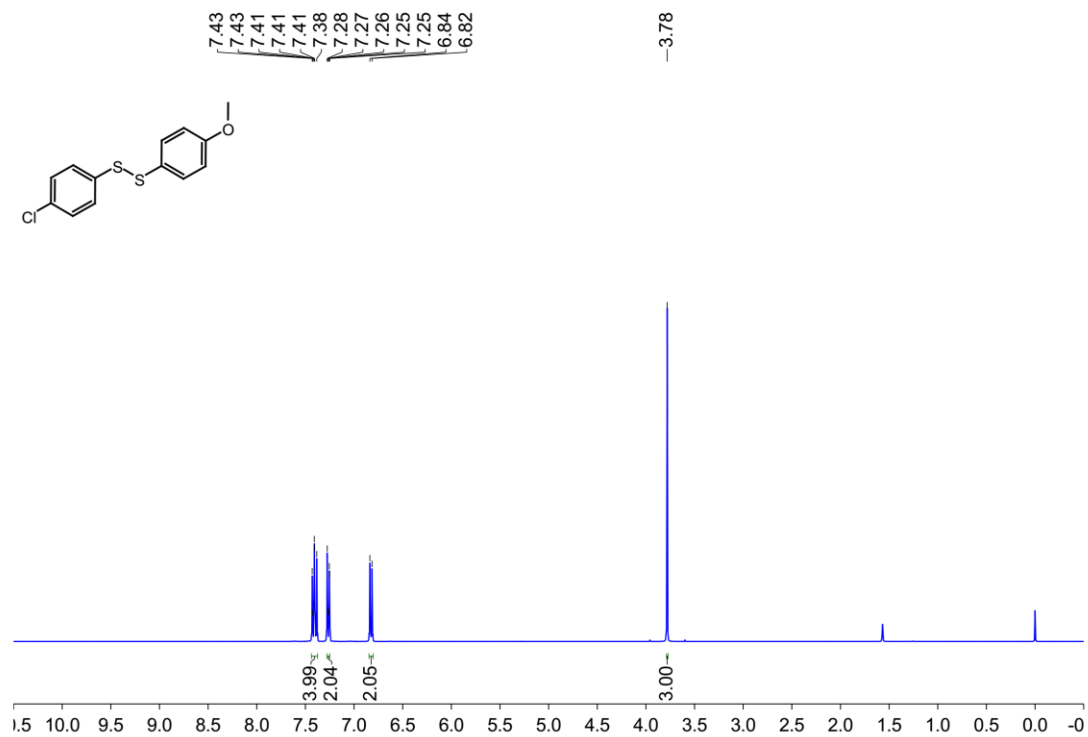
^{13}C NMR spectrum of compound **3c**



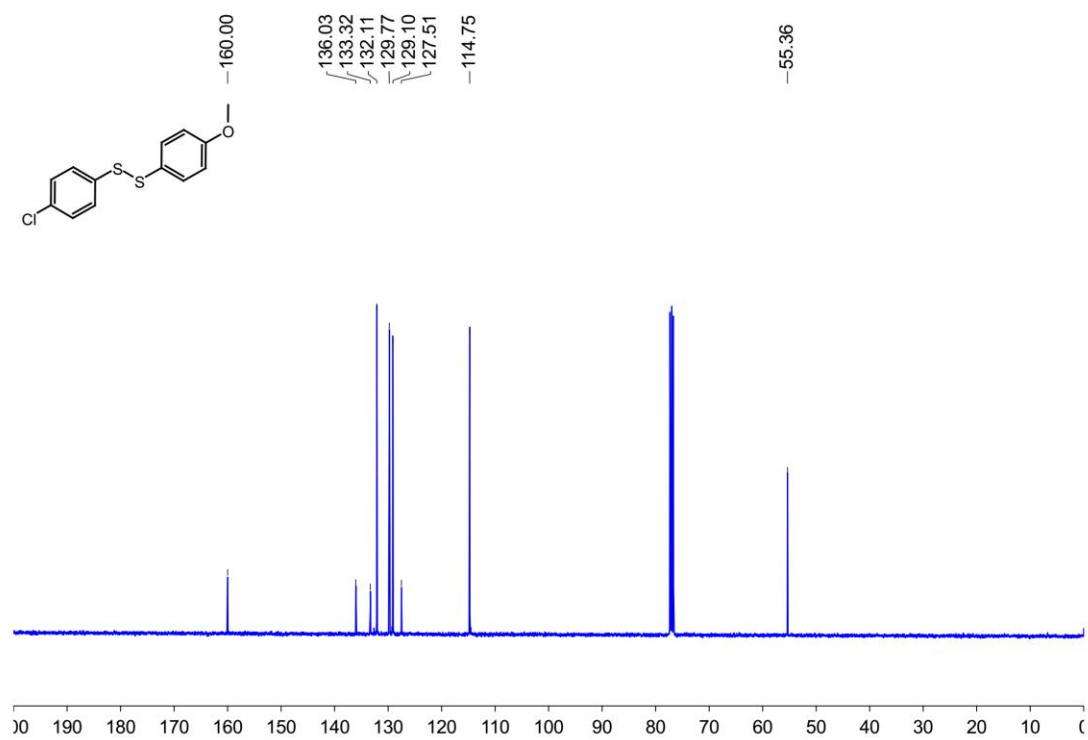
^1H NMR spectrum of compound **3d**



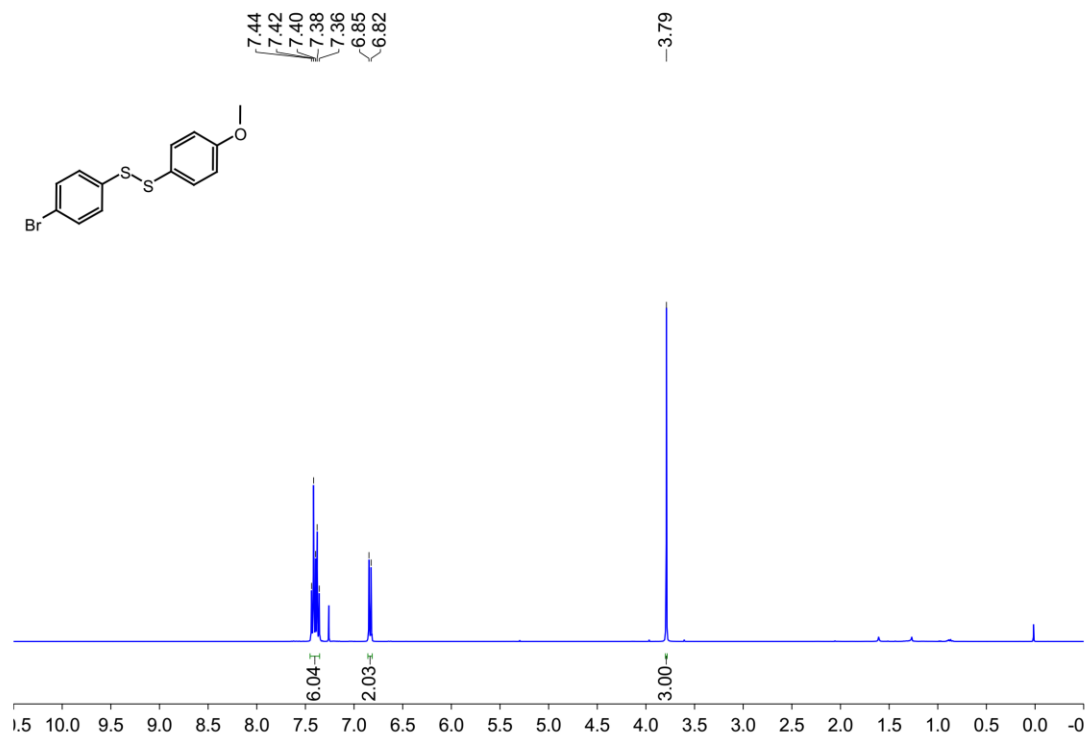
^{13}C NMR spectrum of compound **3d**



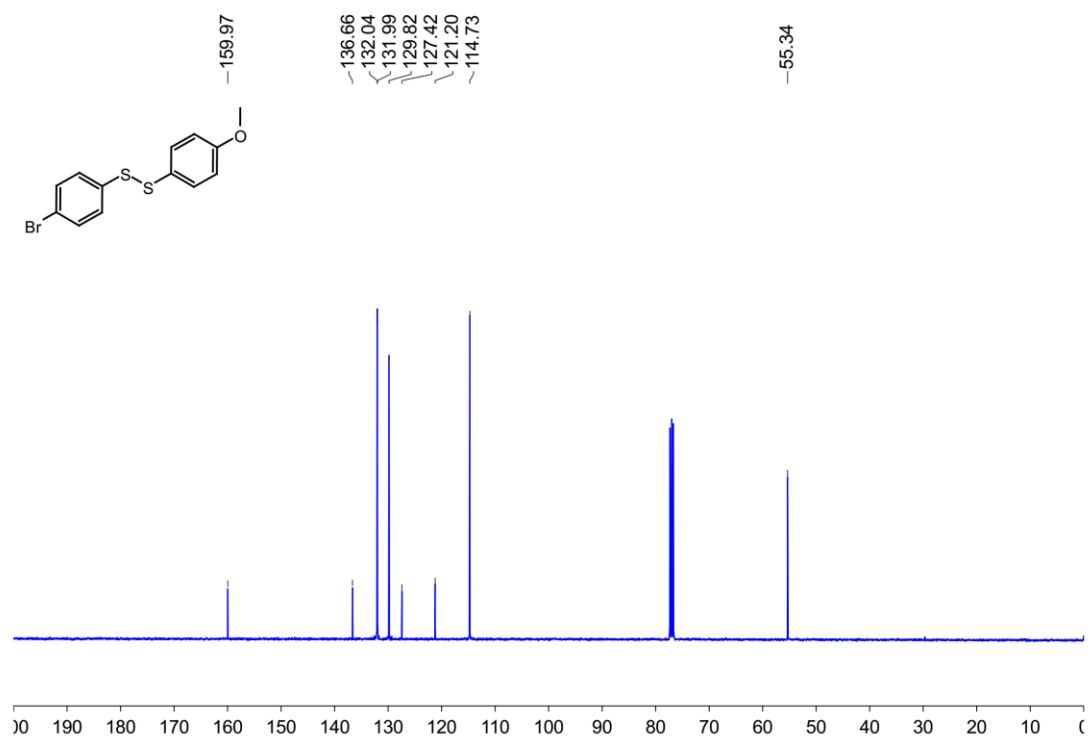
^1H NMR spectrum of compound **3e**



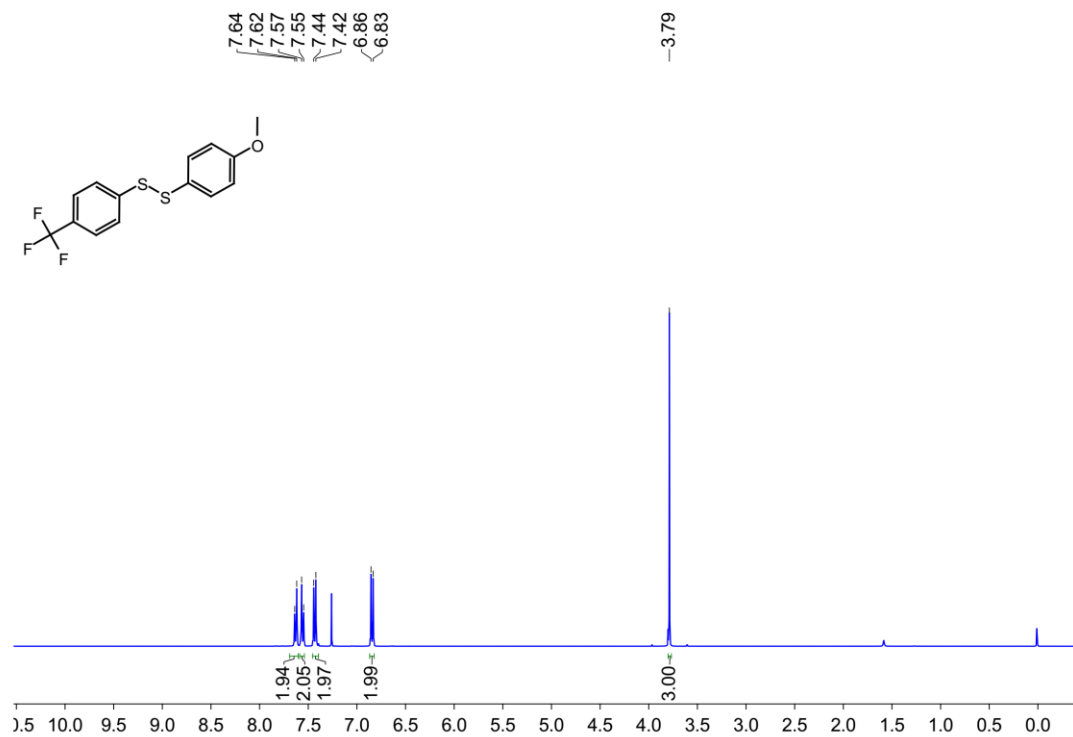
^{13}C NMR spectrum of compound **3e**



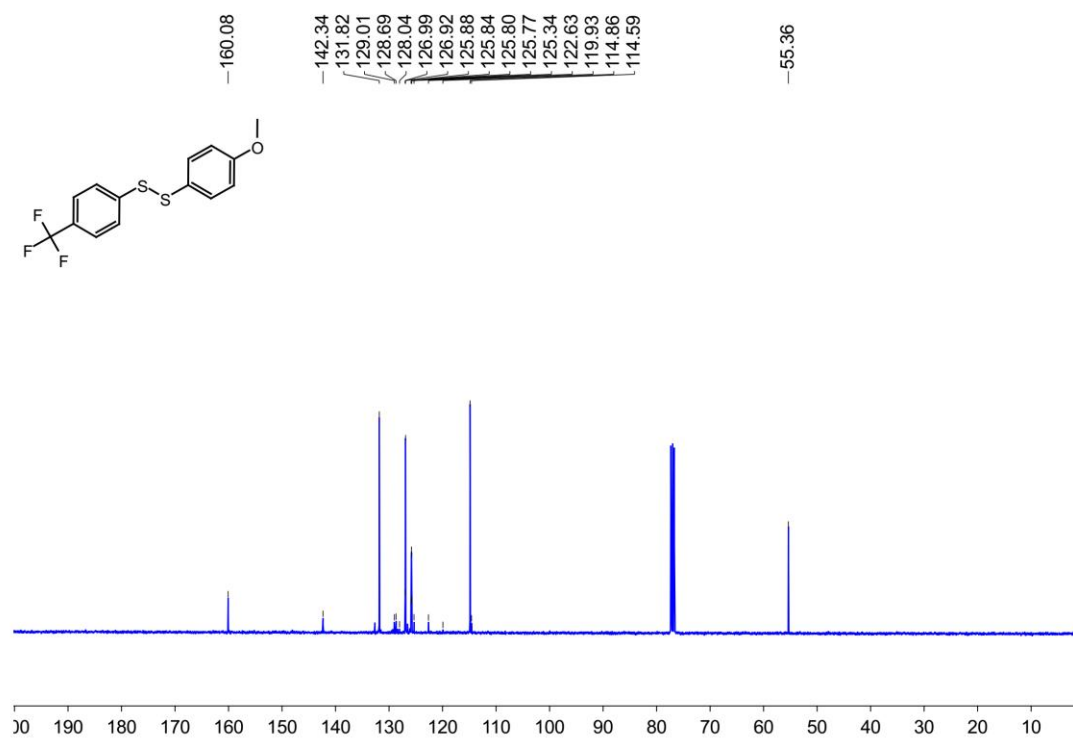
$^1\text{H NMR}$ spectrum of compound **3f**



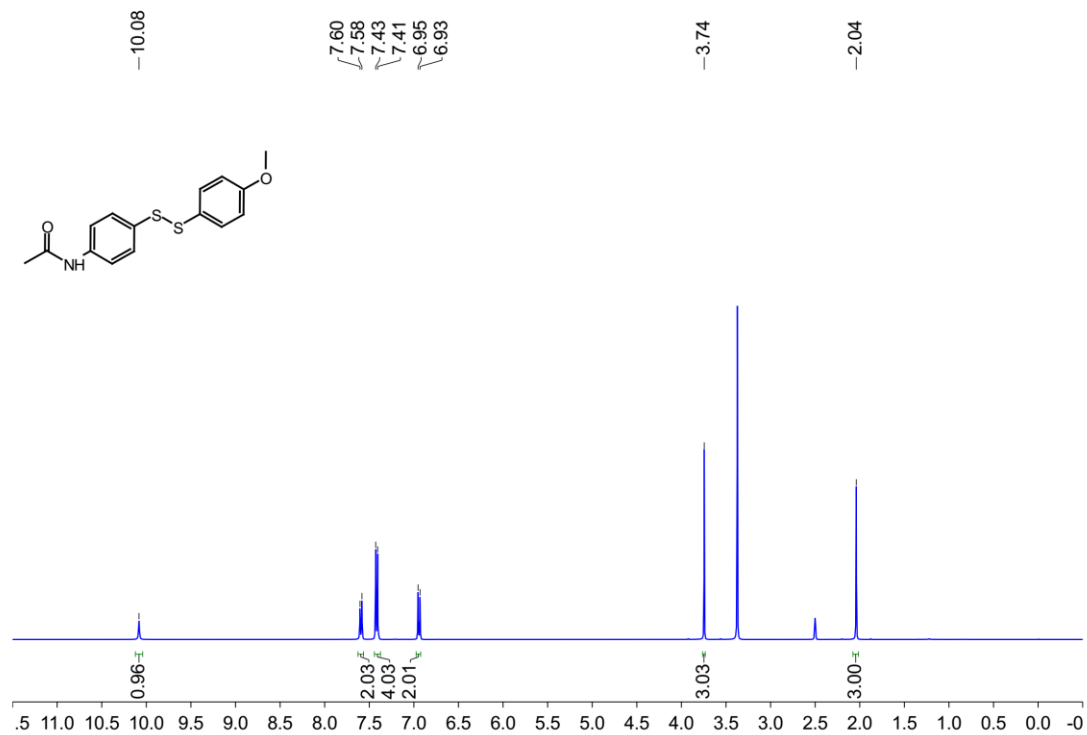
$^{13}\text{C NMR}$ spectrum of compound **3f**



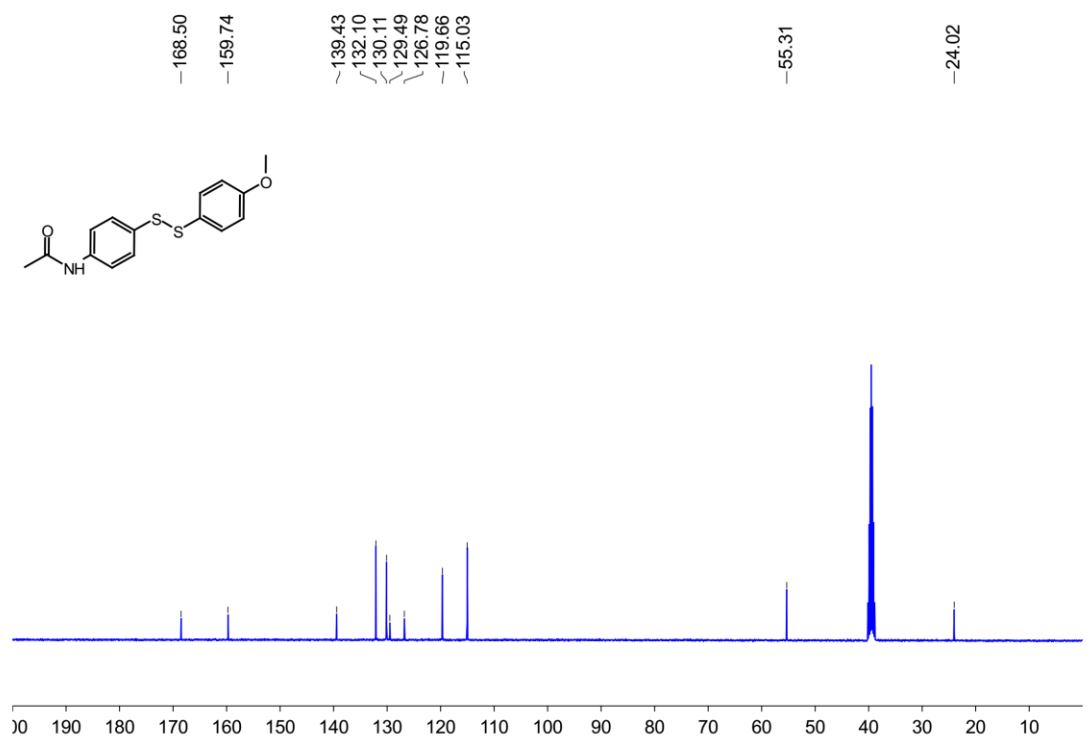
¹H NMR spectrum of compound **3g**



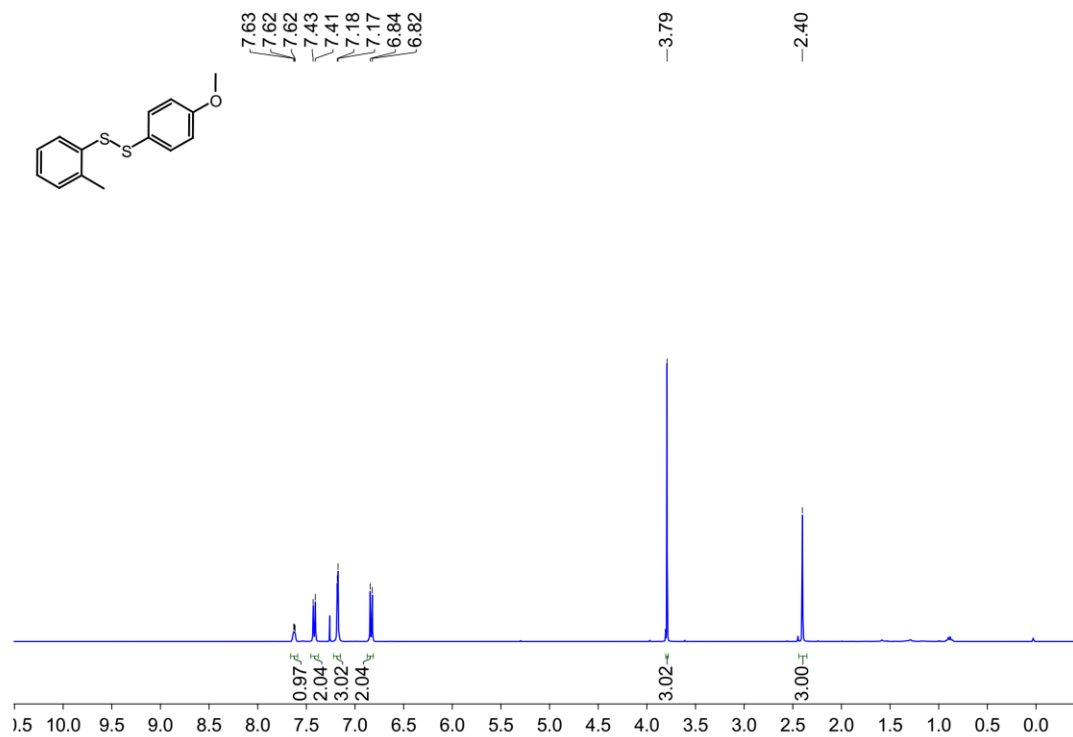
¹³C NMR spectrum of compound **3g**



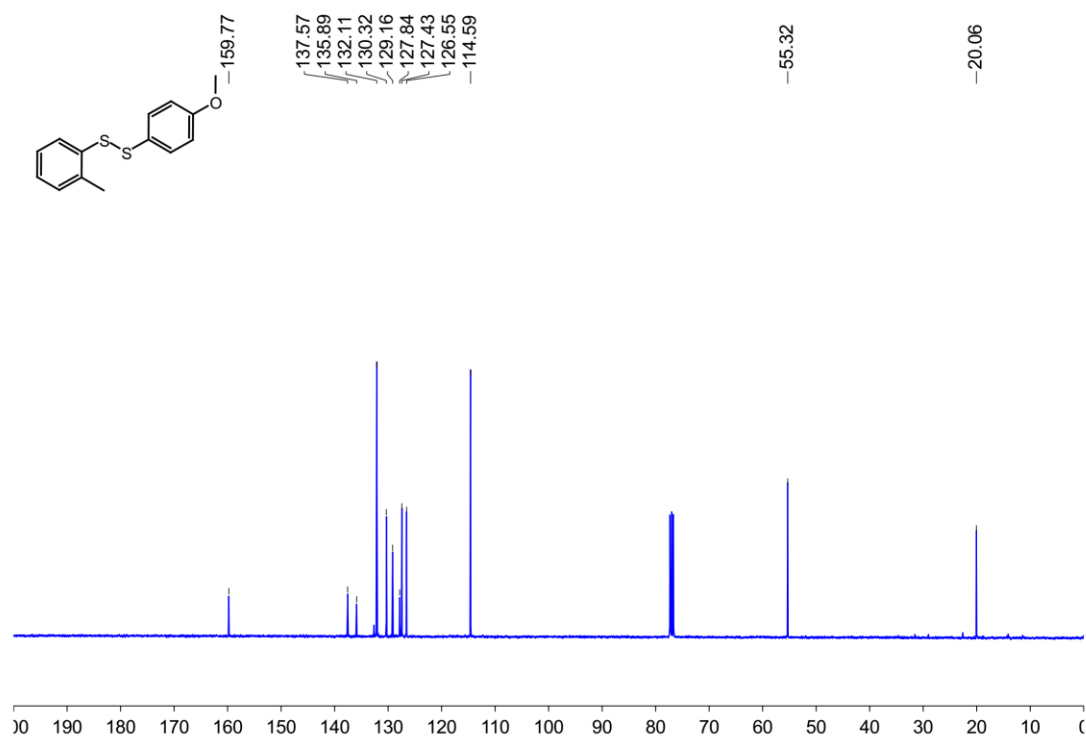
^1H NMR spectrum of compound **3h**



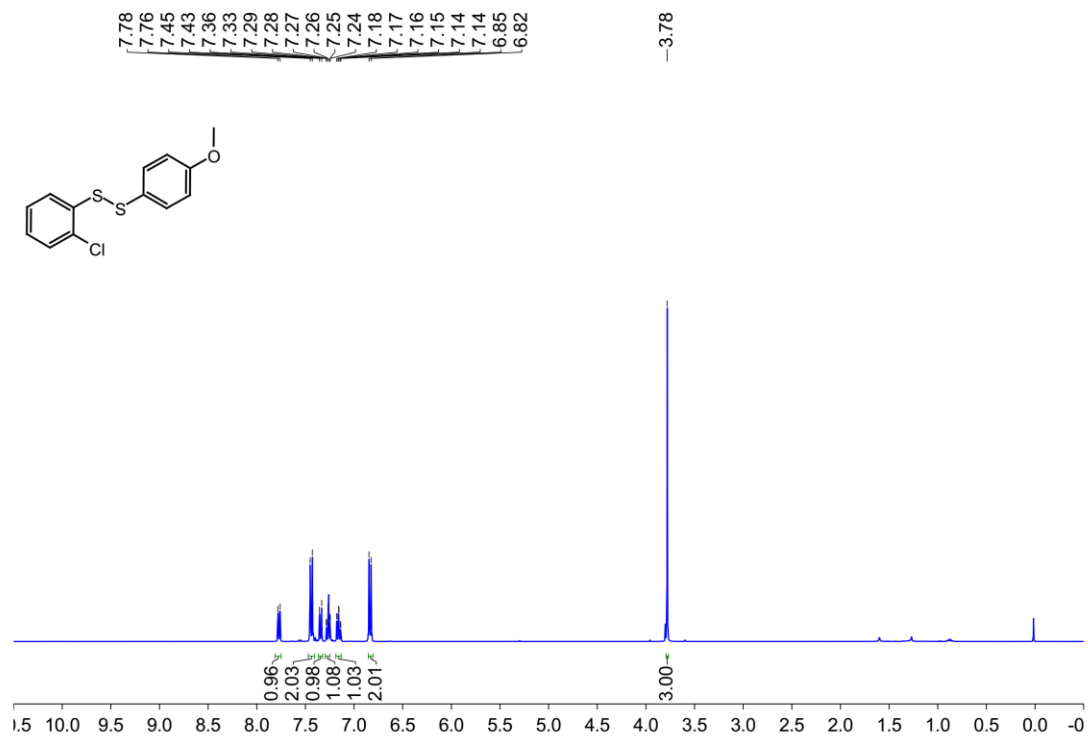
^{13}C NMR spectrum of compound **3h**



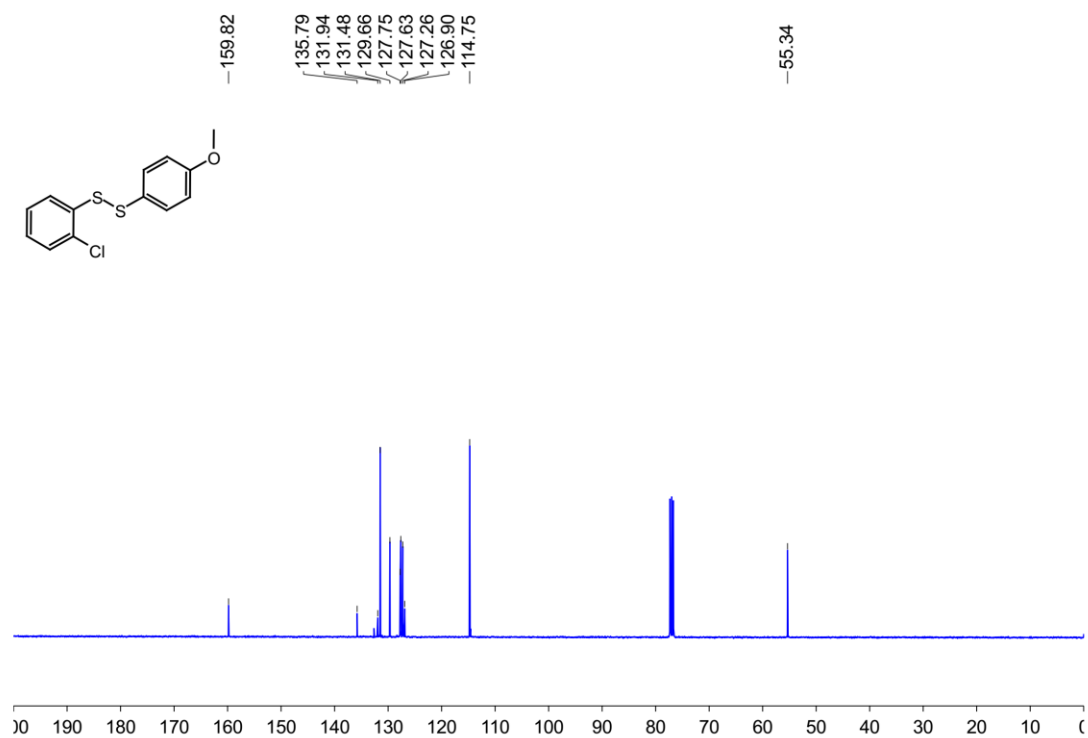
¹H NMR spectrum of compound **3i**



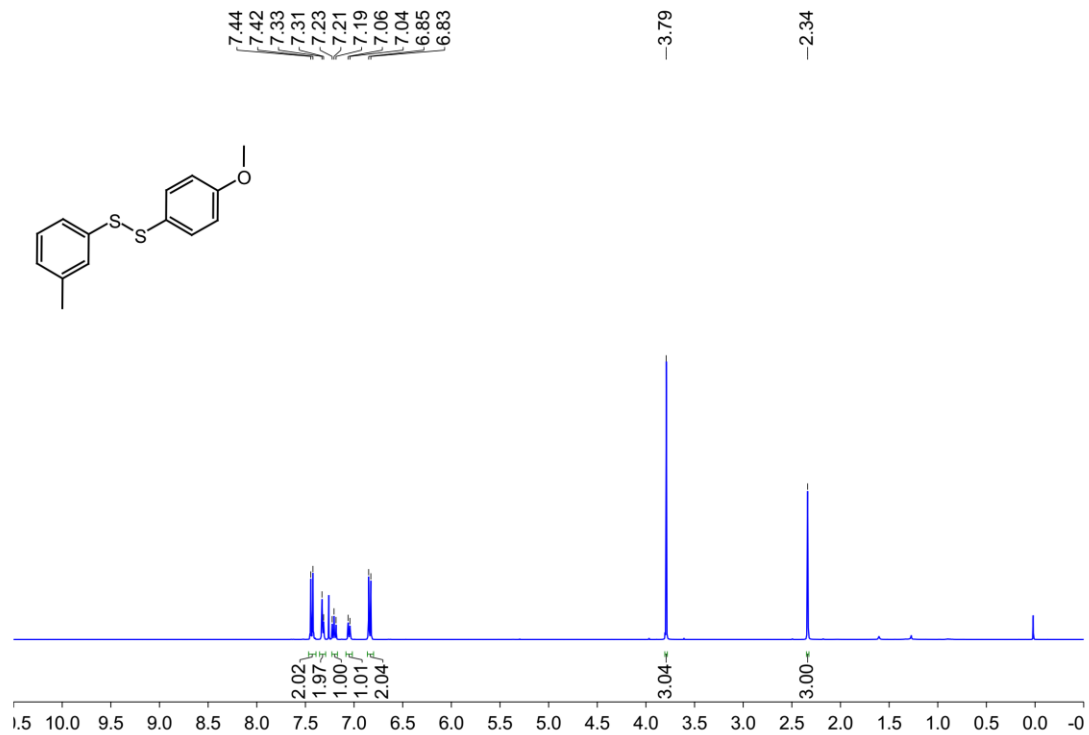
¹³C NMR spectrum of compound **3i**



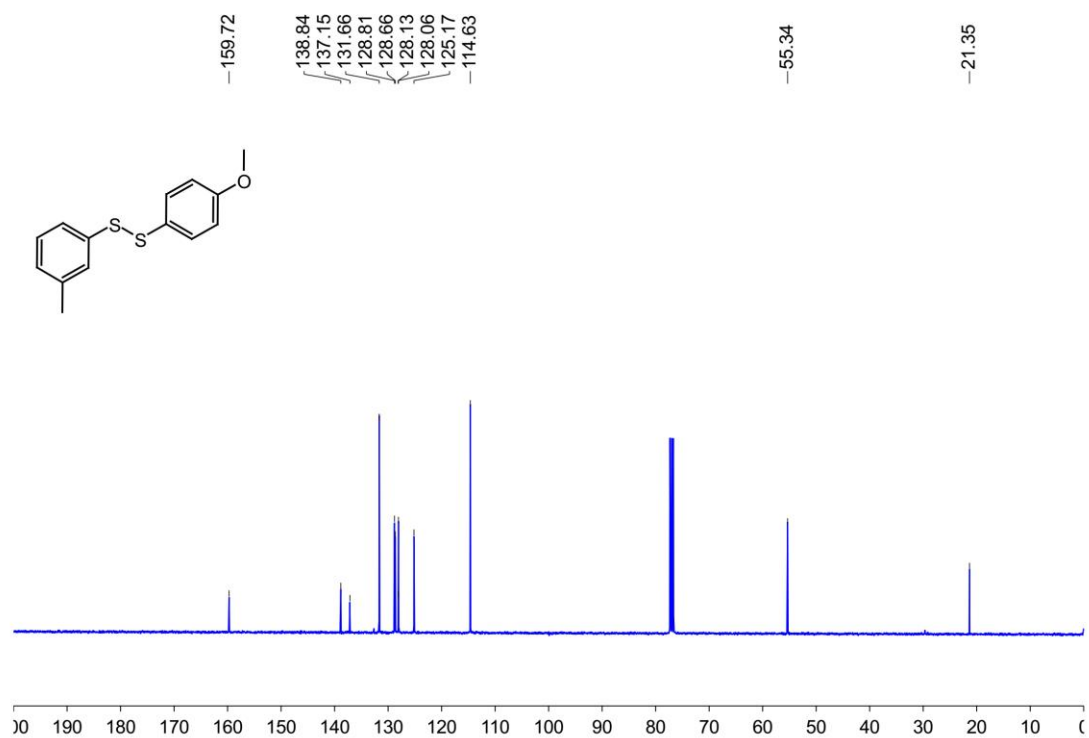
$^1\text{H NMR}$ spectrum of compound **3j**



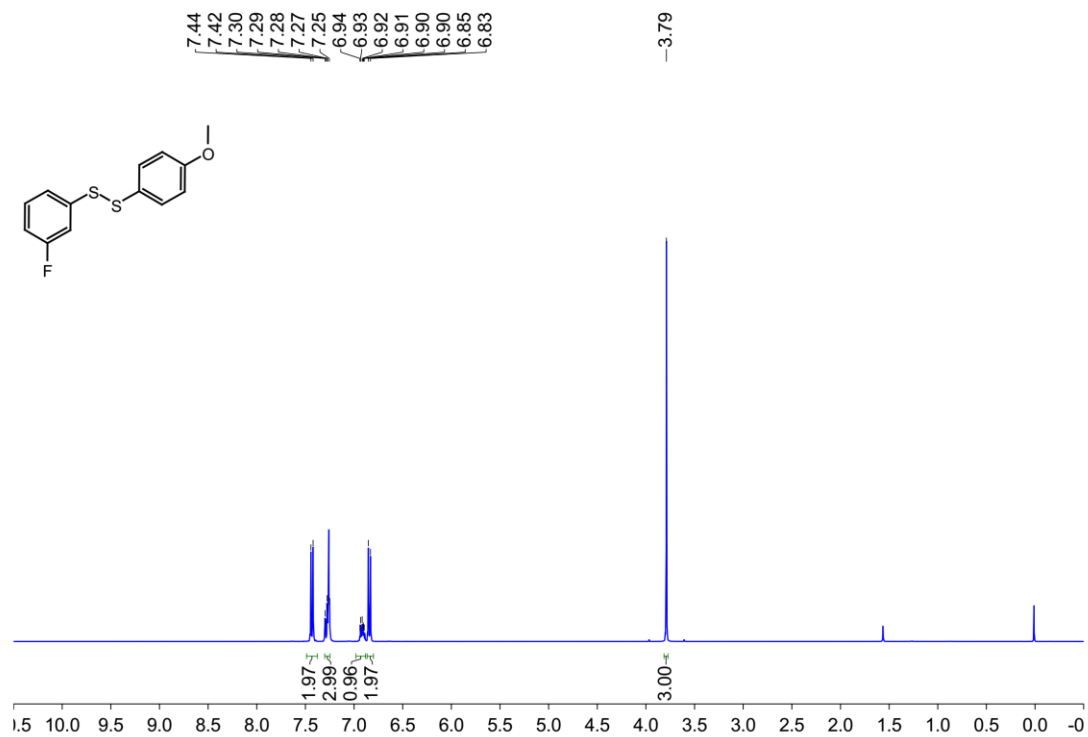
$^{13}\text{C NMR}$ spectrum of compound **3j**



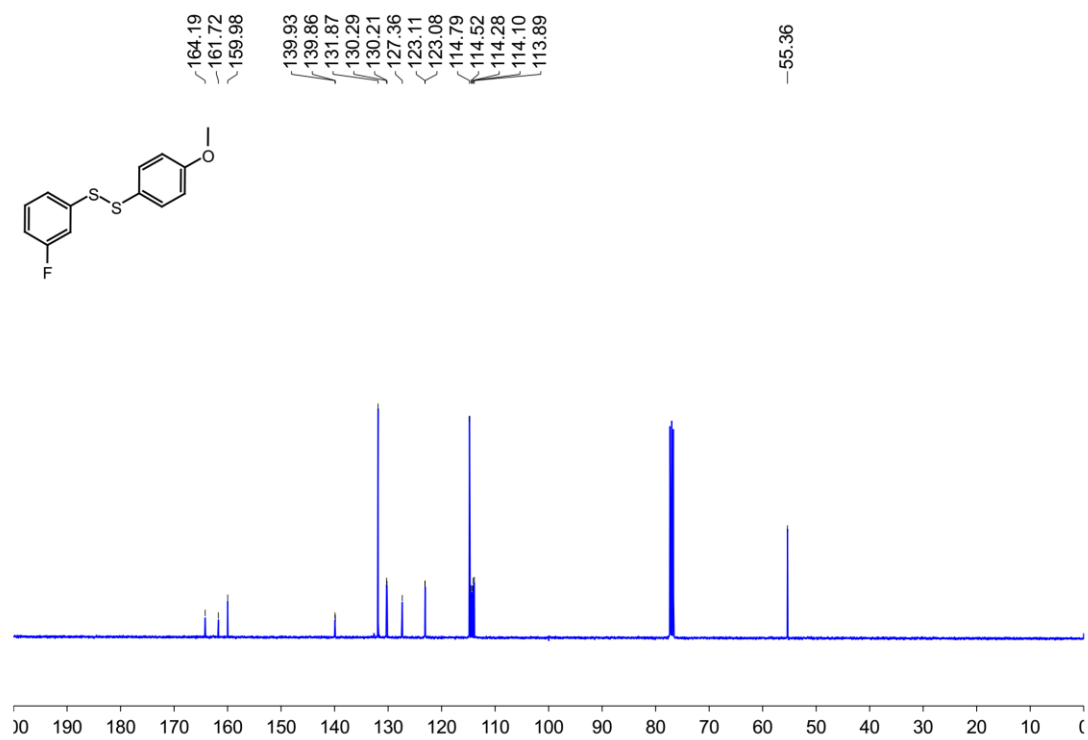
¹H NMR spectrum of compound **3k**



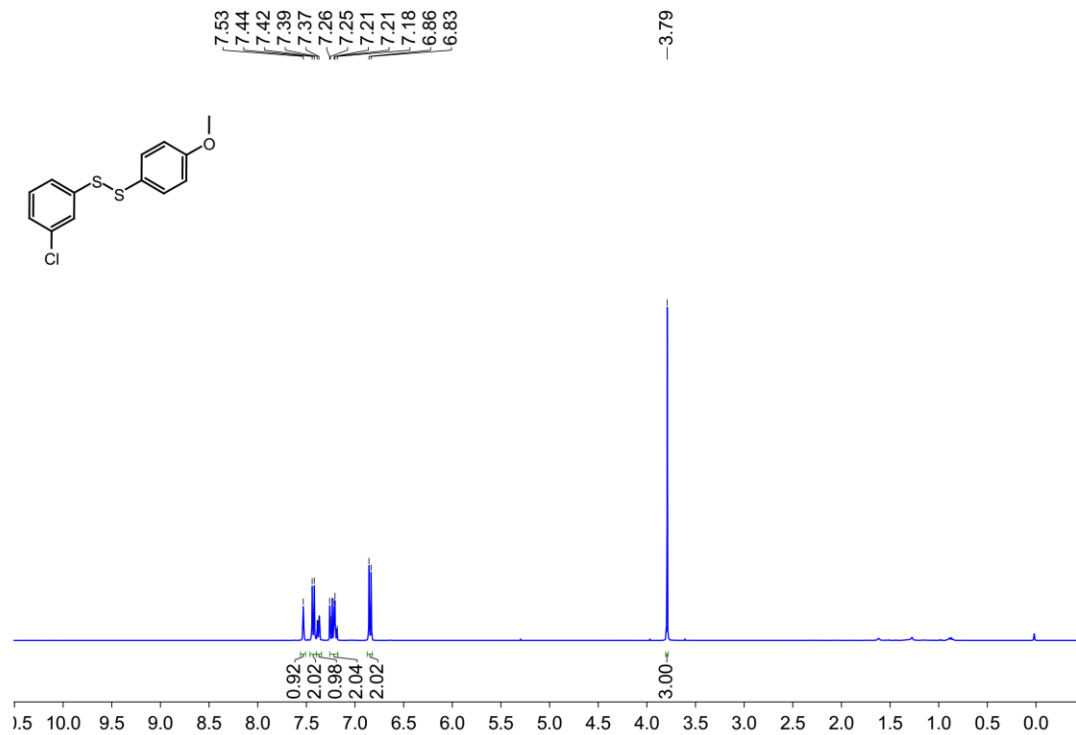
¹³C NMR spectrum of compound **3k**



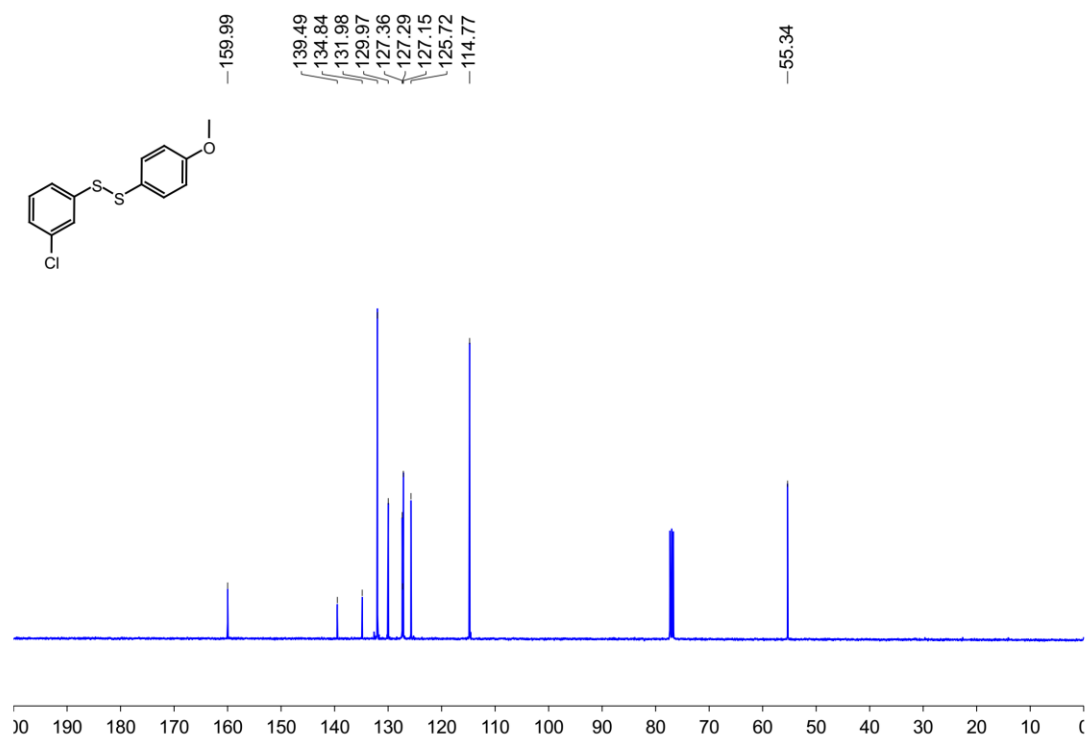
¹H NMR spectrum of compound **31**



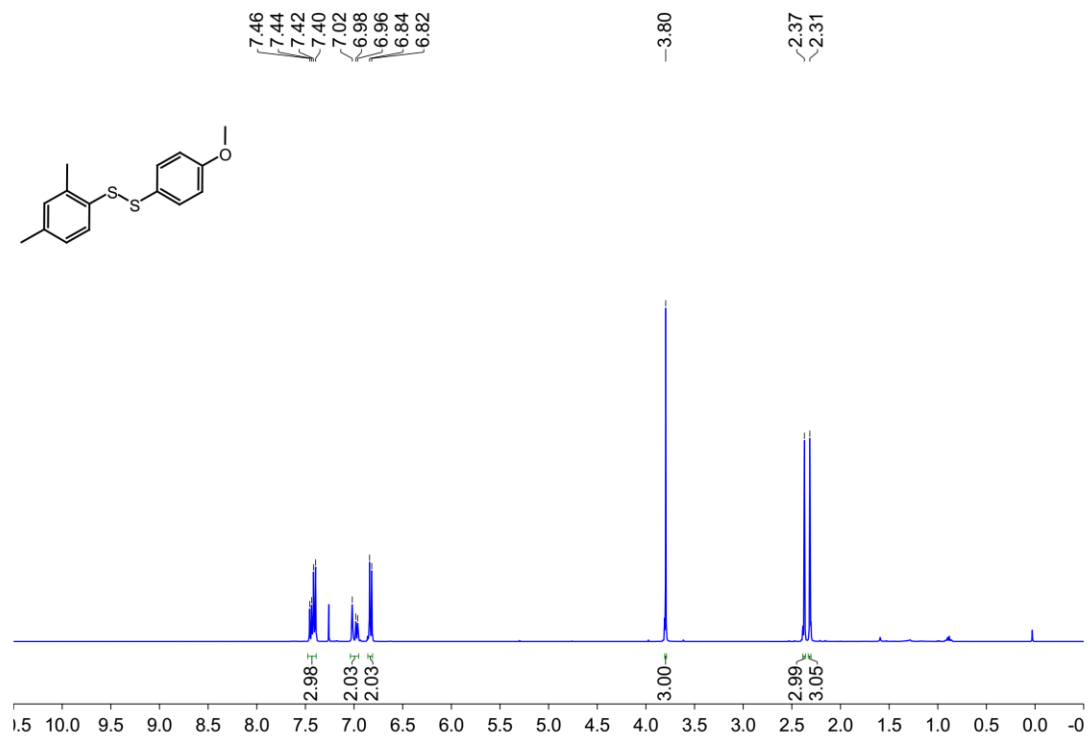
¹³C NMR spectrum of compound **31**



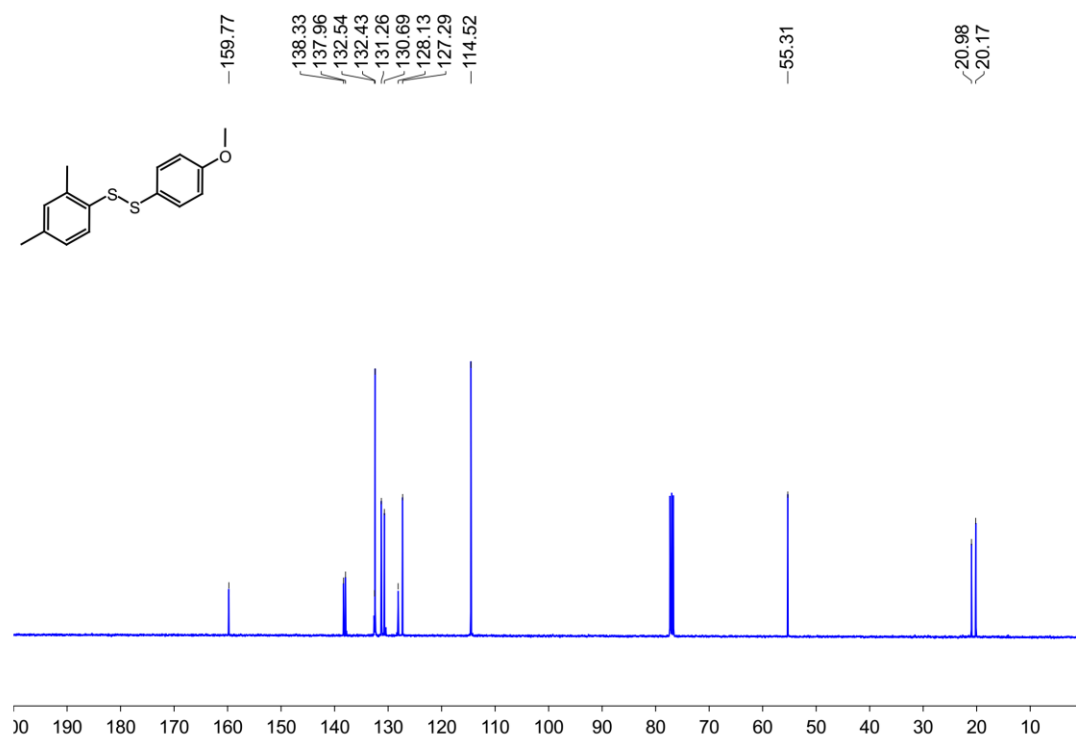
^1H NMR spectrum of compound **3m**



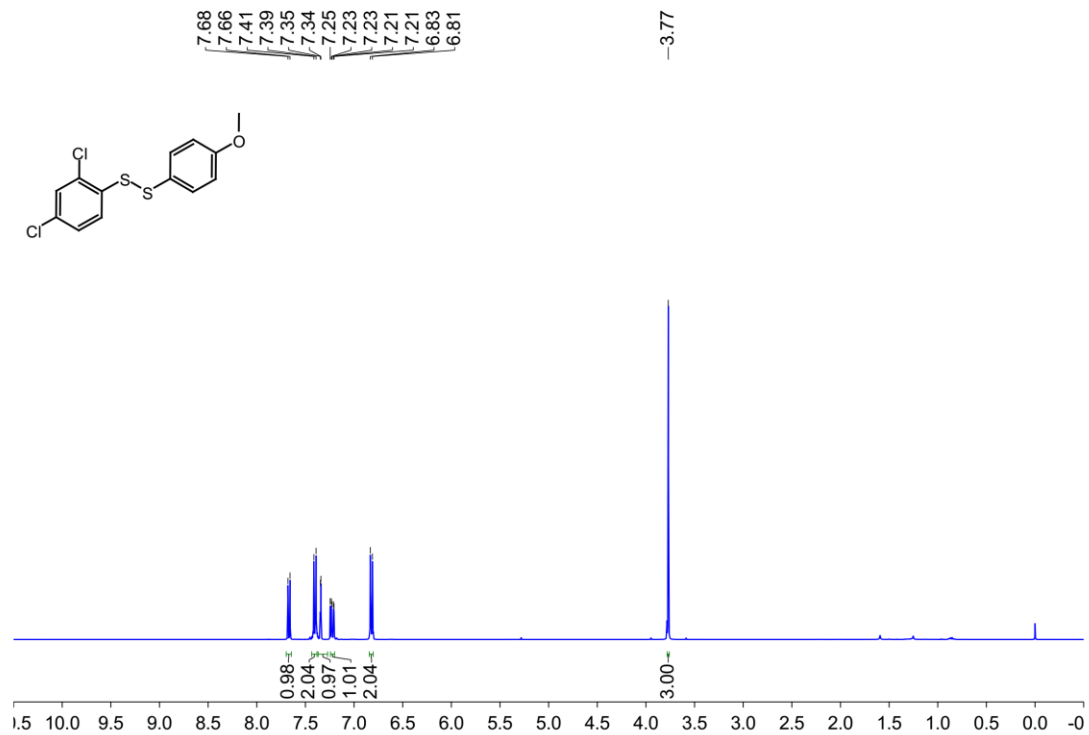
^{13}C NMR spectrum of compound **3m**



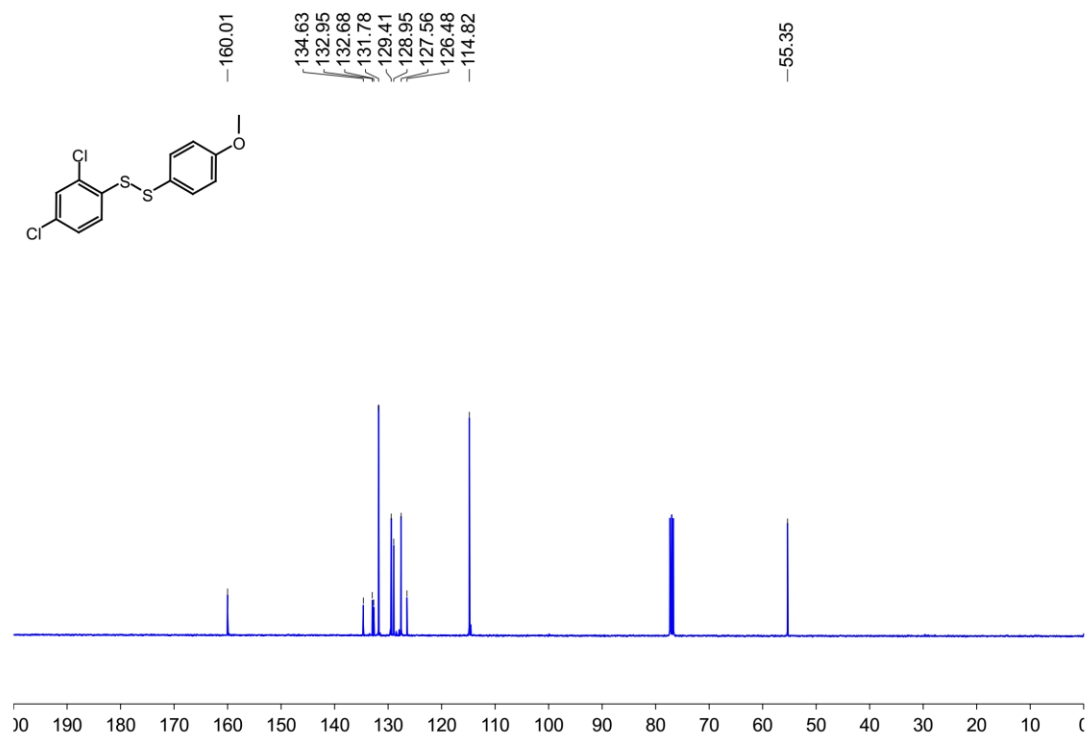
^1H NMR spectrum of compound **3n**



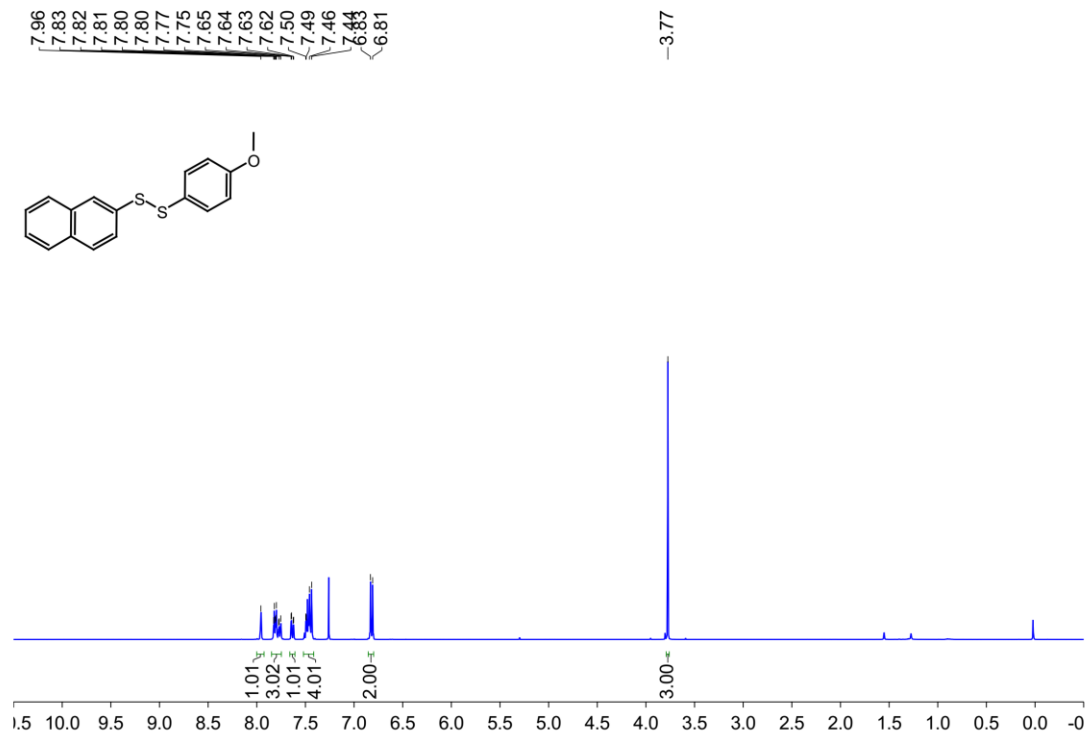
^{13}C NMR spectrum of compound **3n**



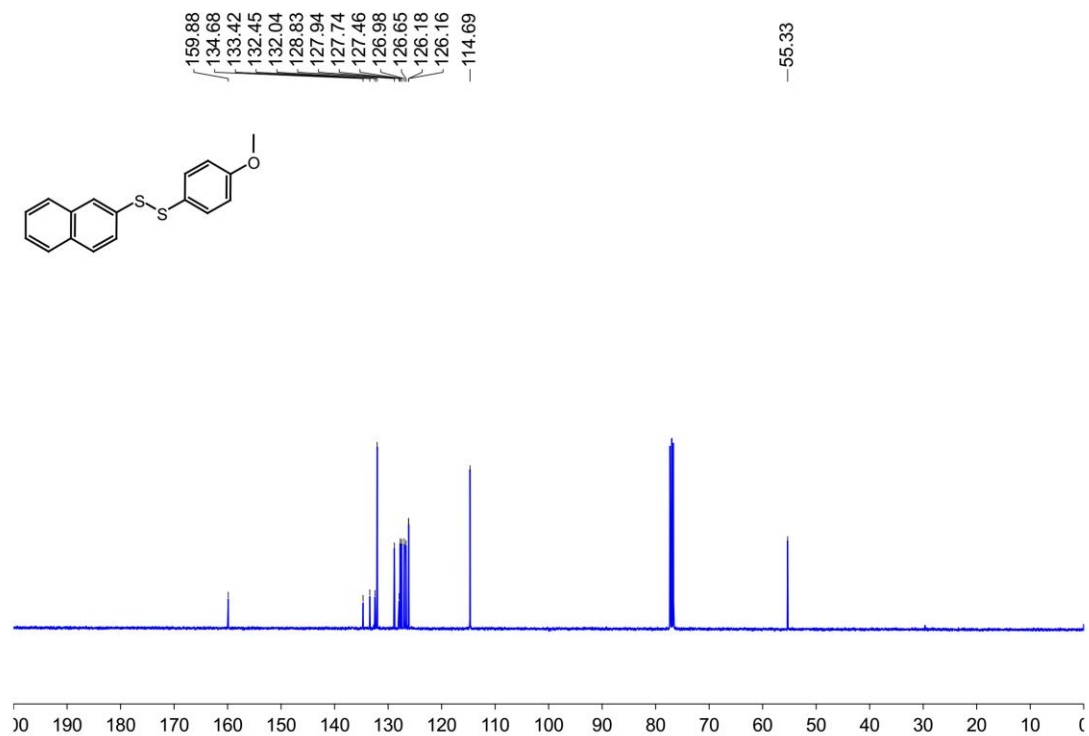
¹H NMR spectrum of compound **3o**



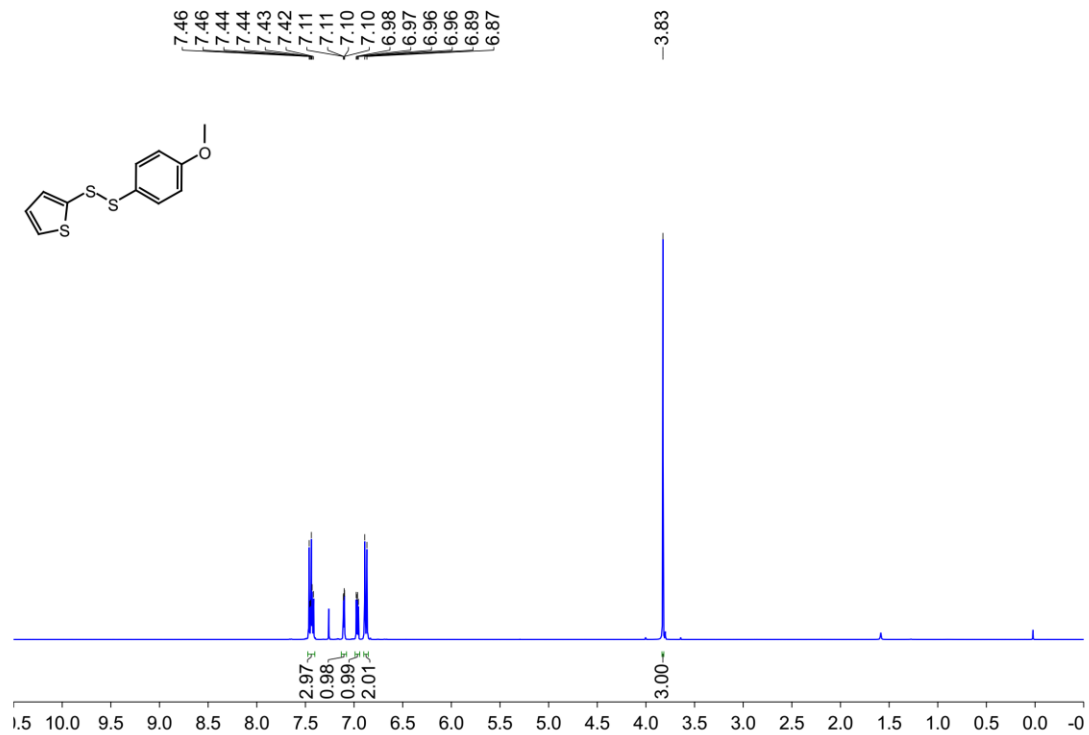
¹³C NMR spectrum of compound **3o**



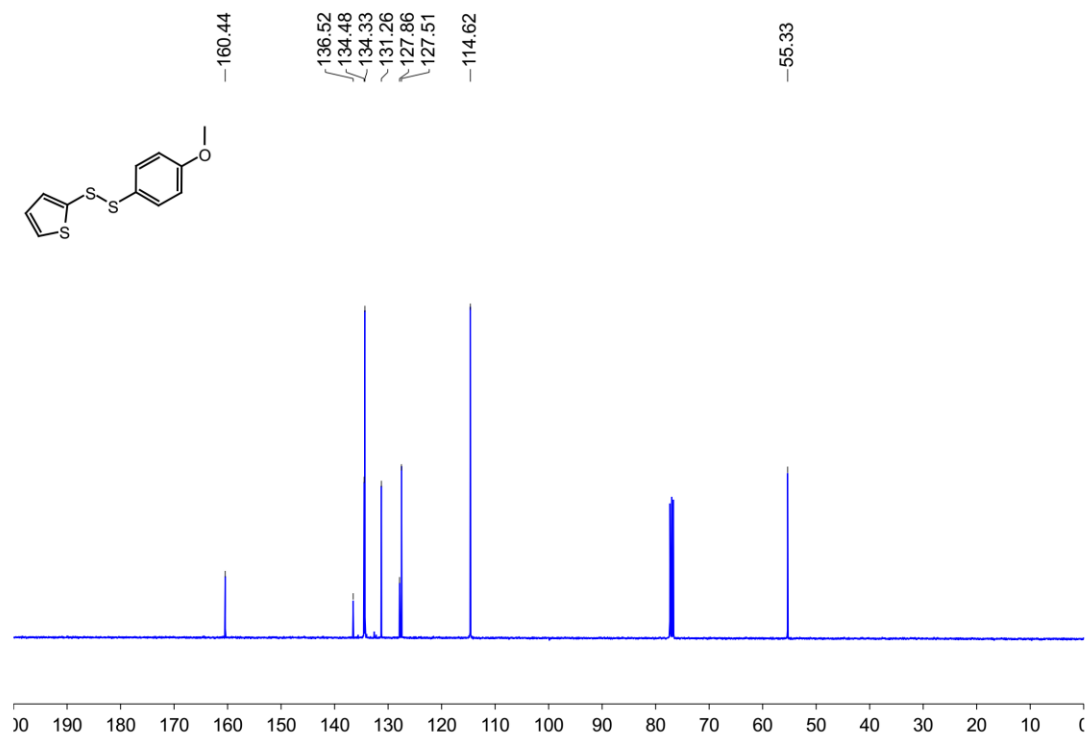
^1H NMR spectrum of compound **3p**



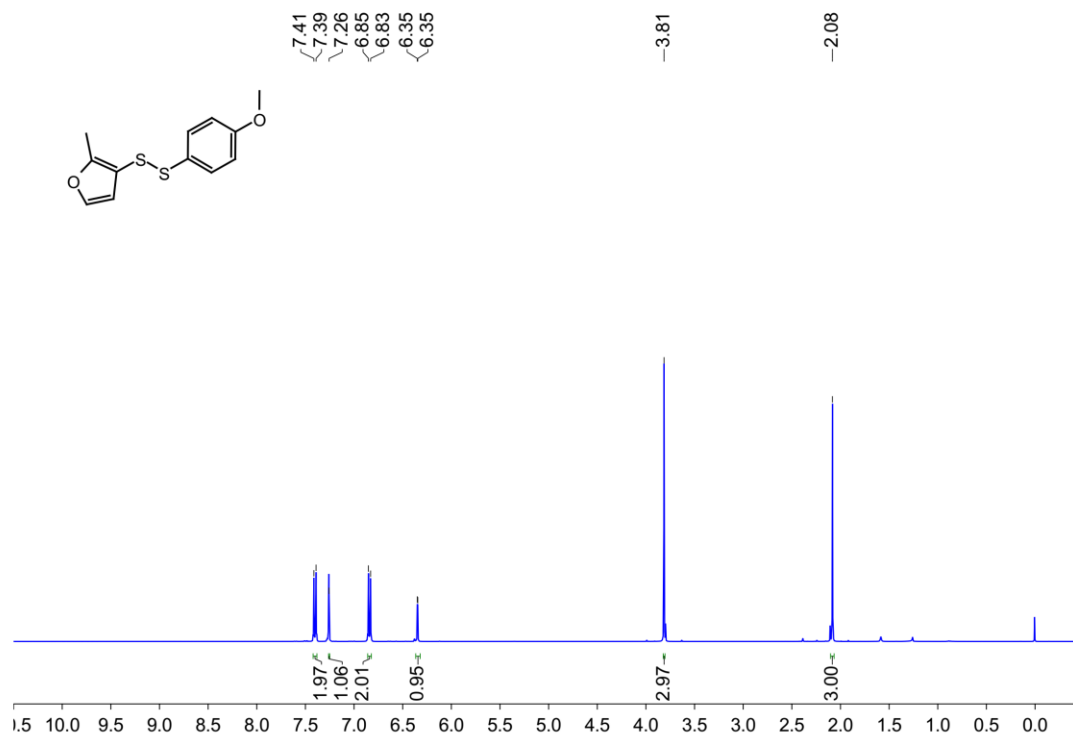
^{13}C NMR spectrum of compound **3p**



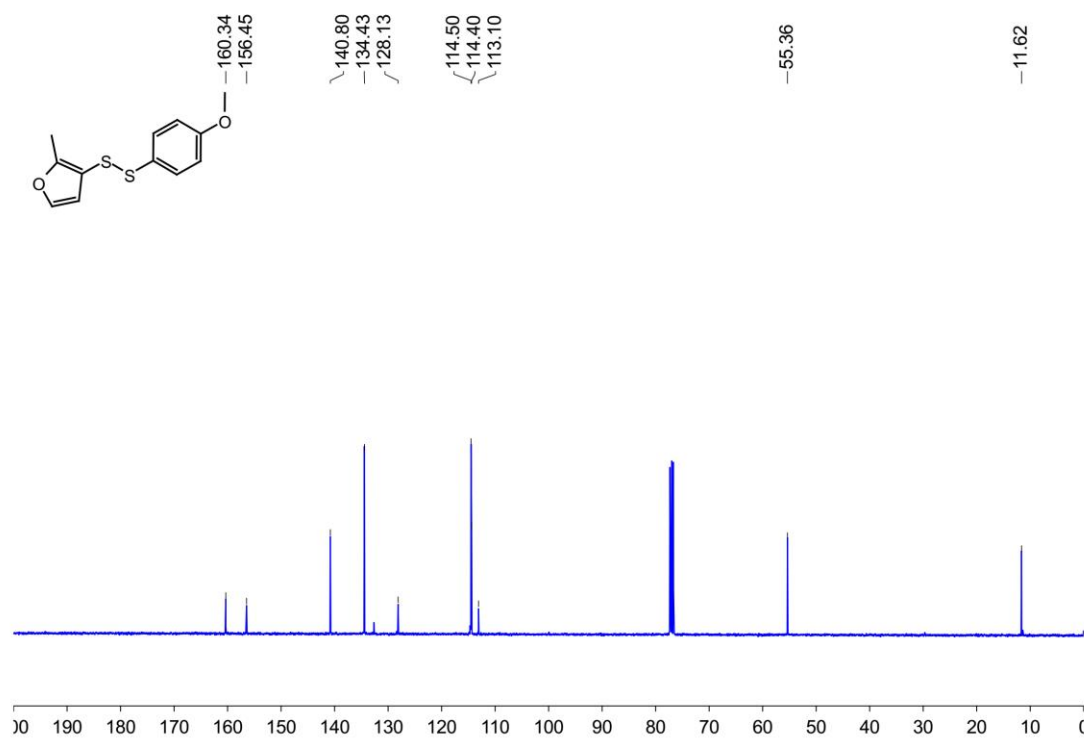
^1H NMR spectrum of compound **3q**



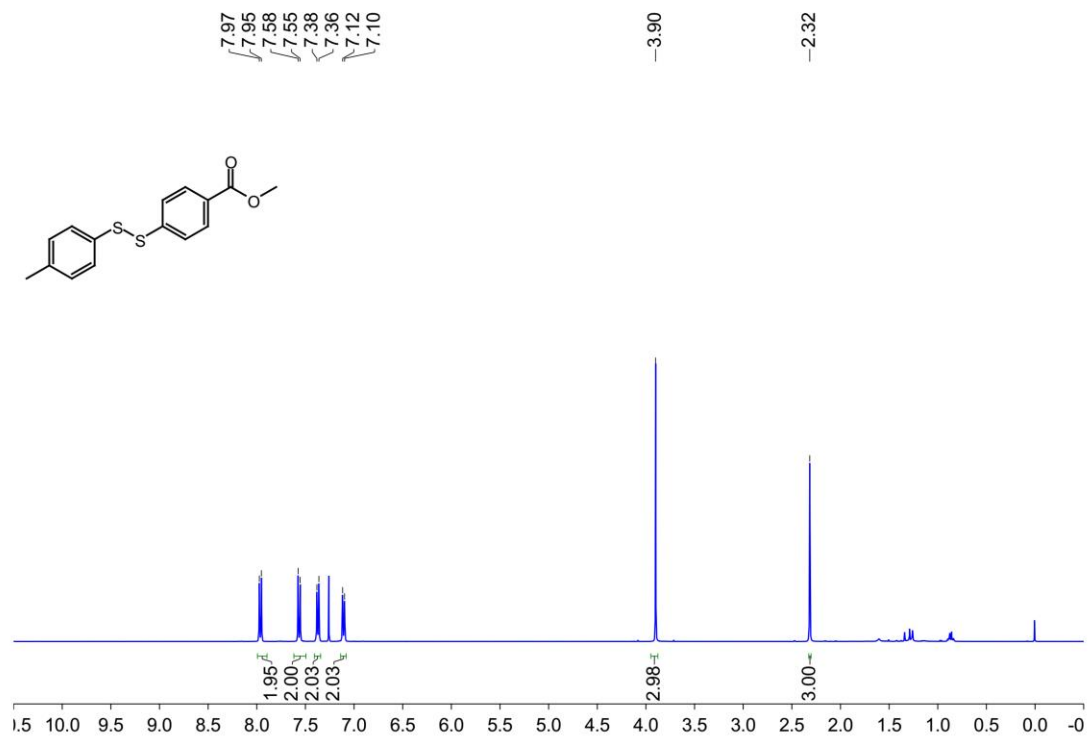
^{13}C NMR spectrum of compound **3q**



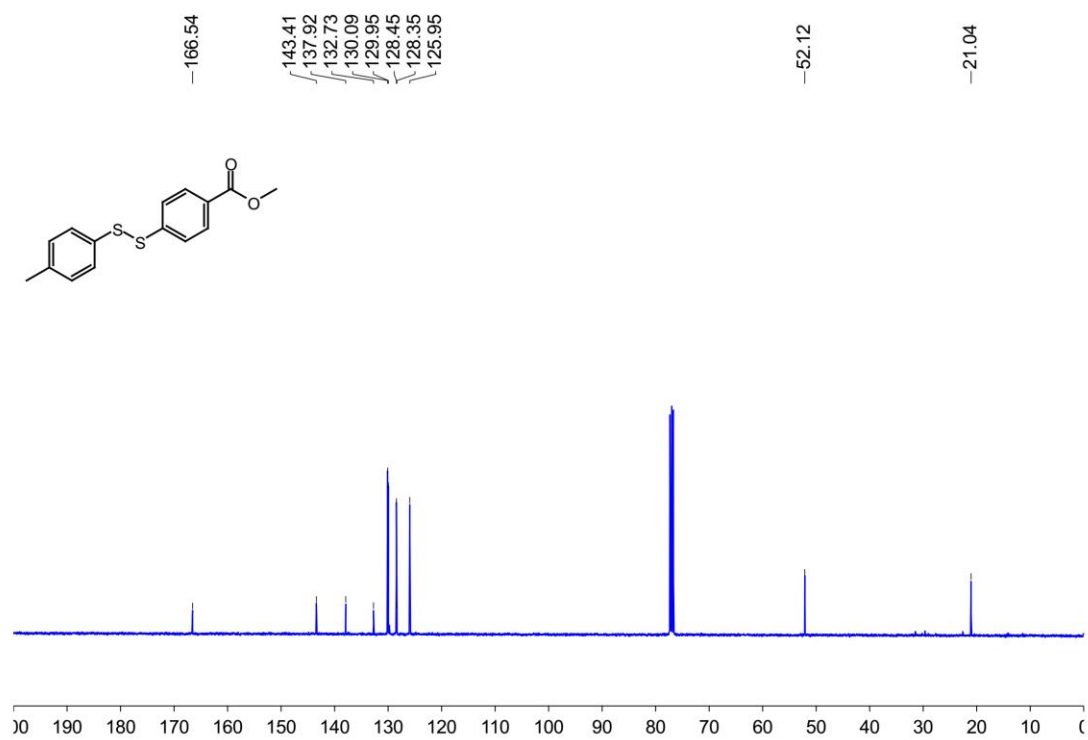
^1H NMR spectrum of compound **3r**



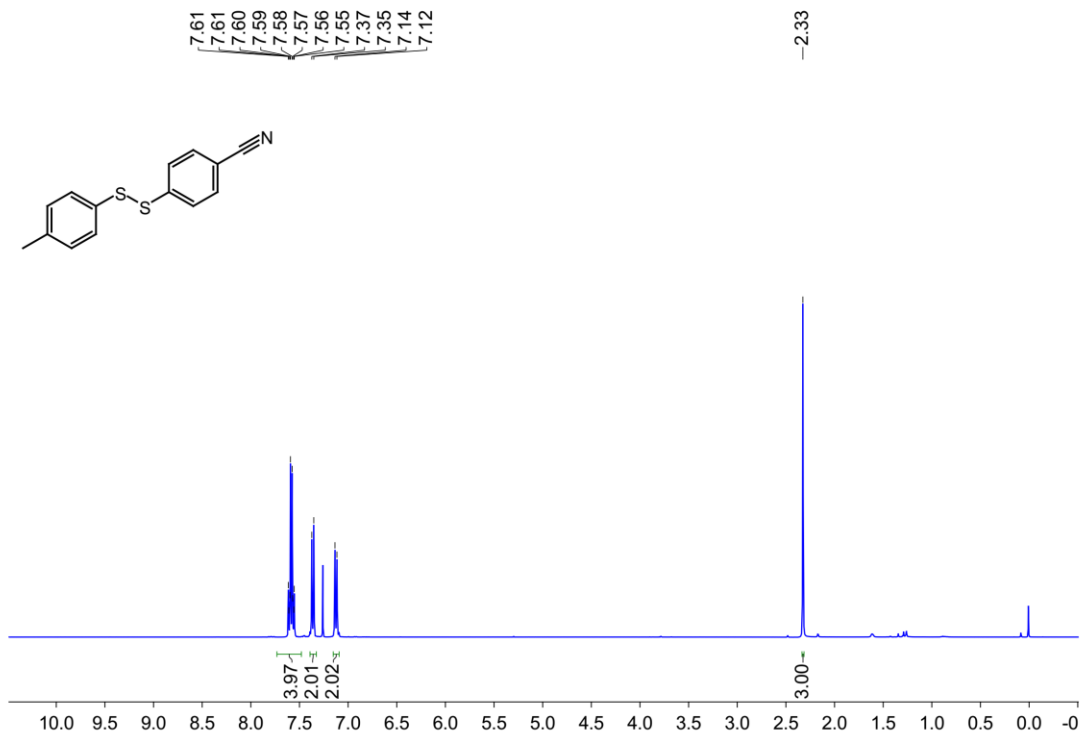
^{13}C NMR spectrum of compound **3r**



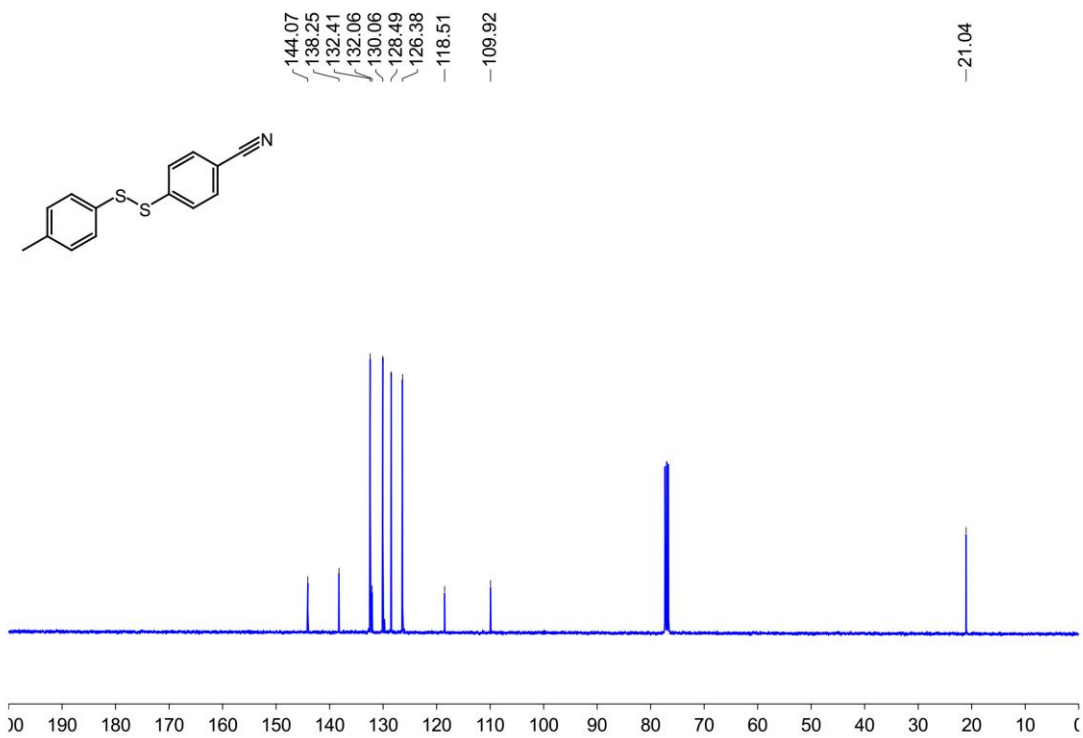
^1H NMR spectrum of compound **3s**



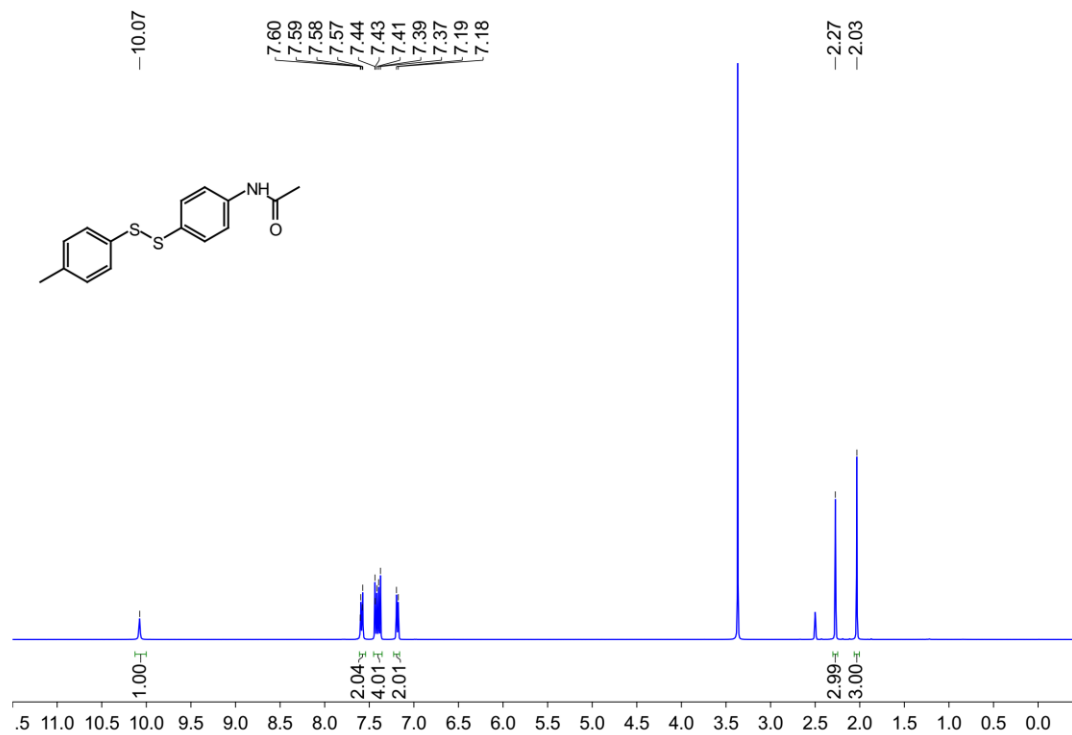
^{13}C NMR spectrum of compound **3s**



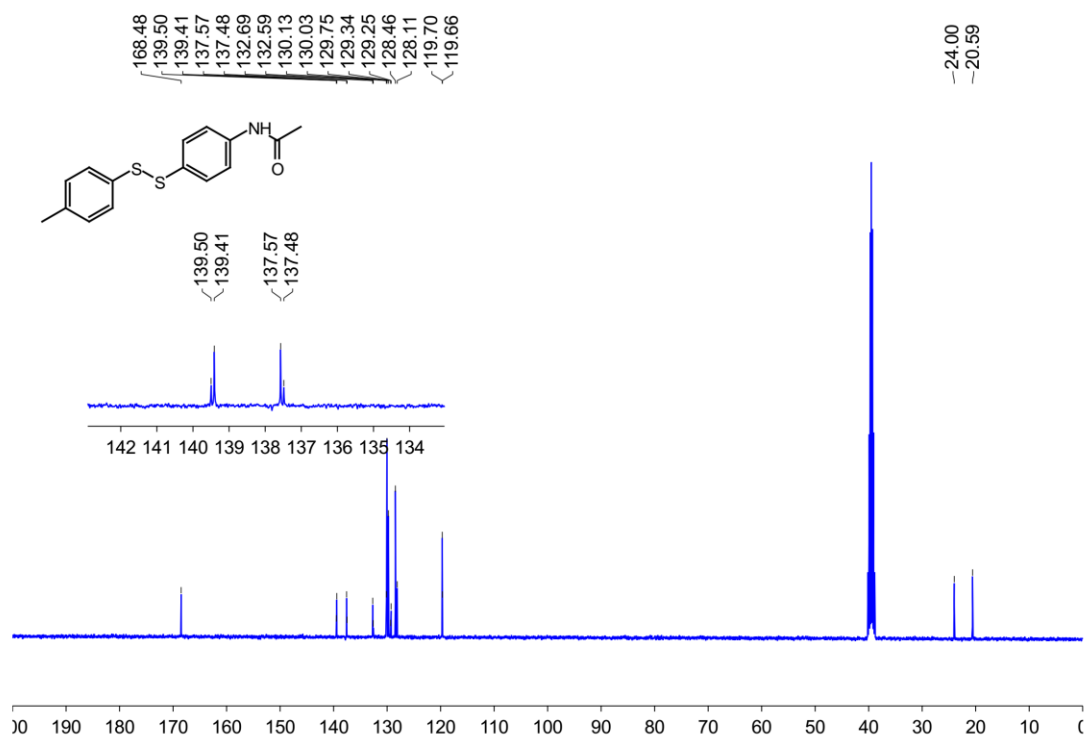
$^1\text{H NMR}$ spectrum of compound **3t**



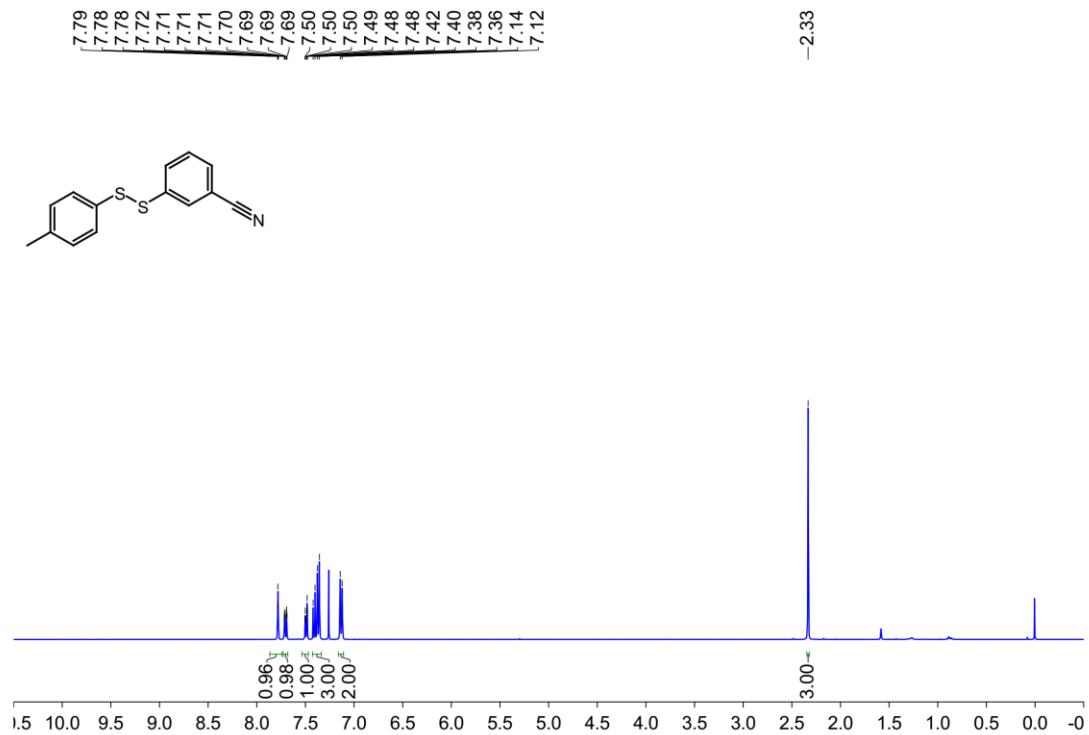
$^{13}\text{C NMR}$ spectrum of compound **3t**



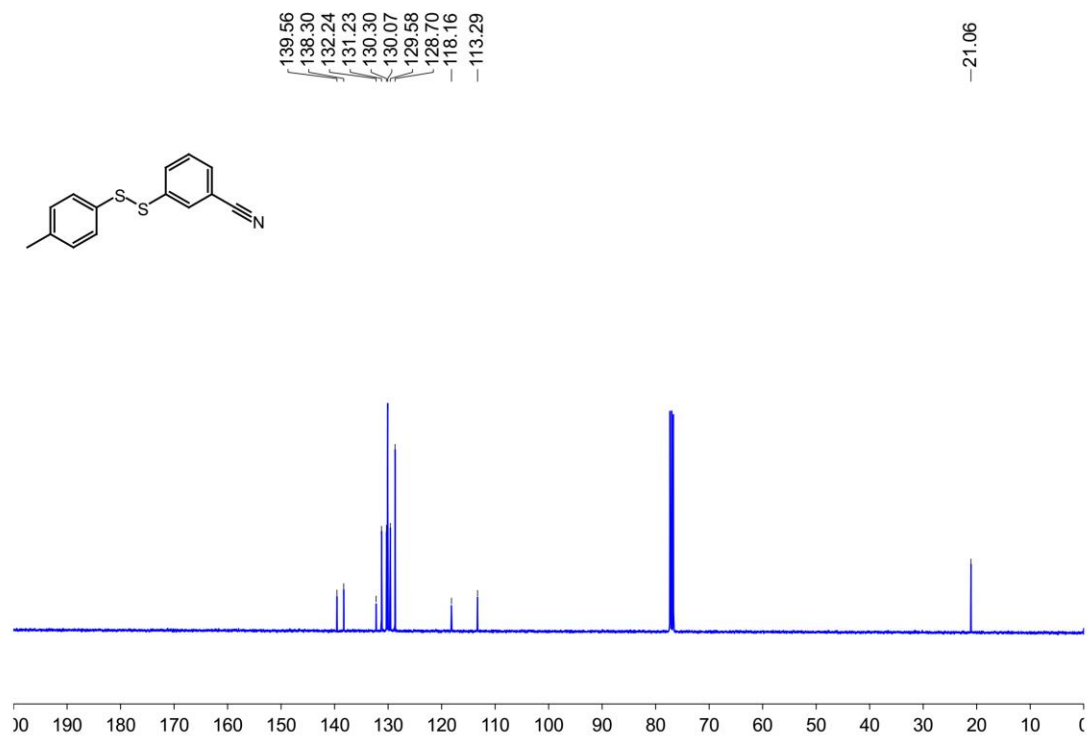
^1H NMR spectrum of compound **3u**



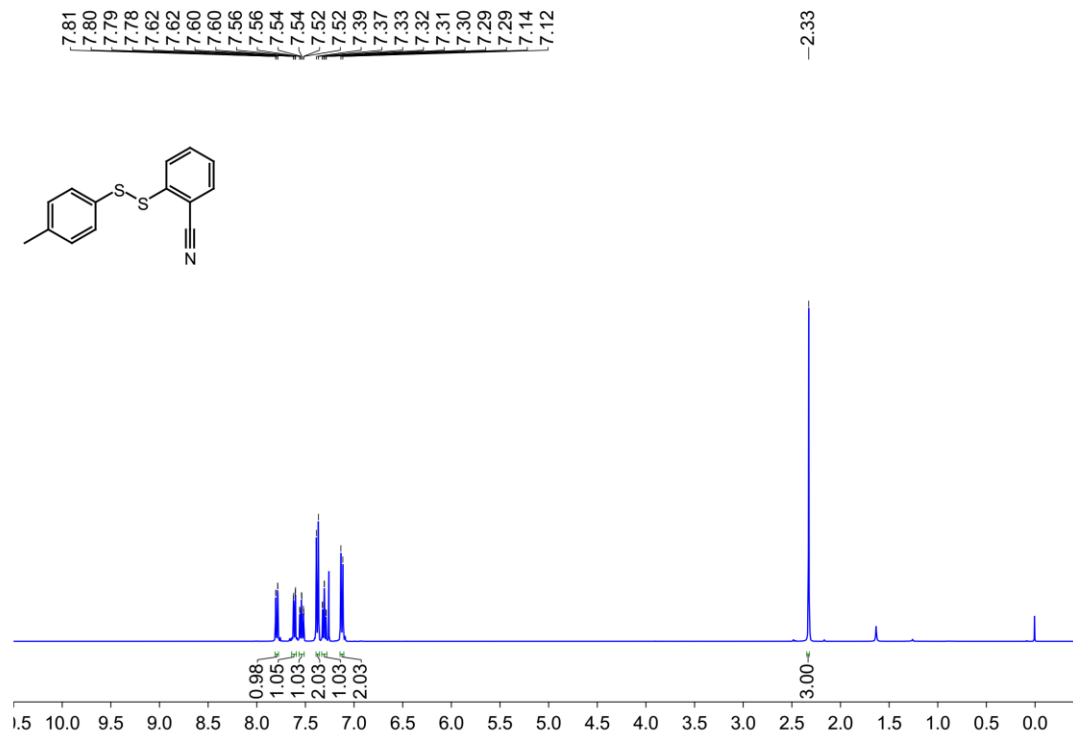
^{13}C NMR spectrum of compound **3u**



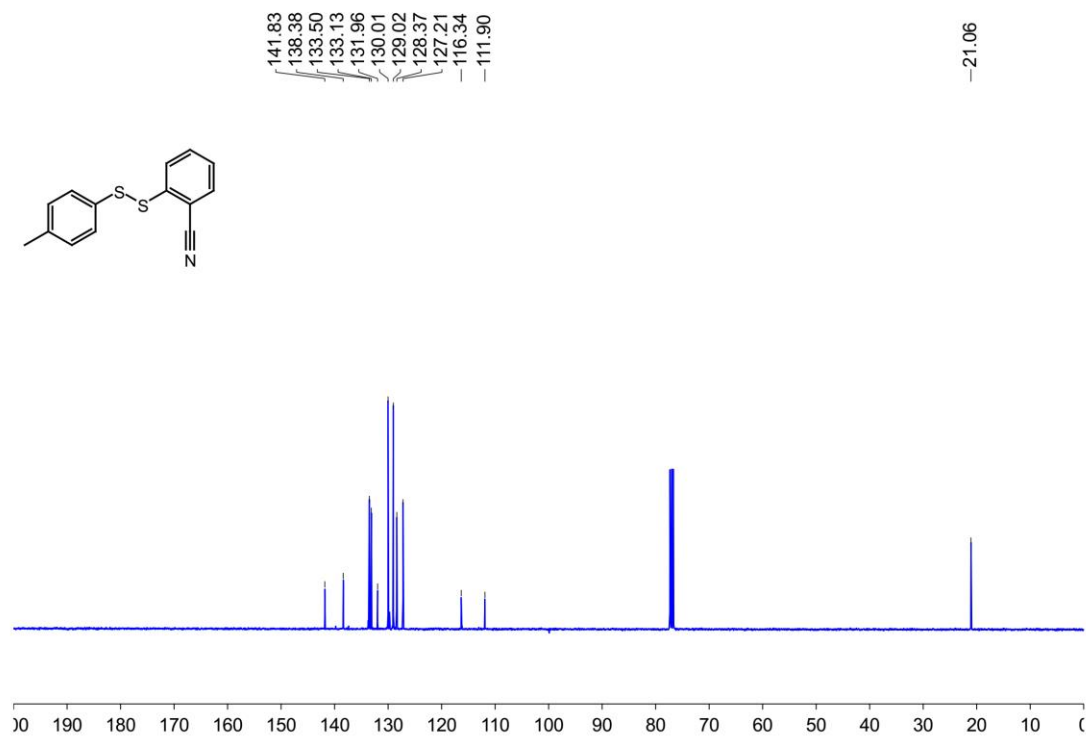
¹H NMR spectrum of compound **3v**



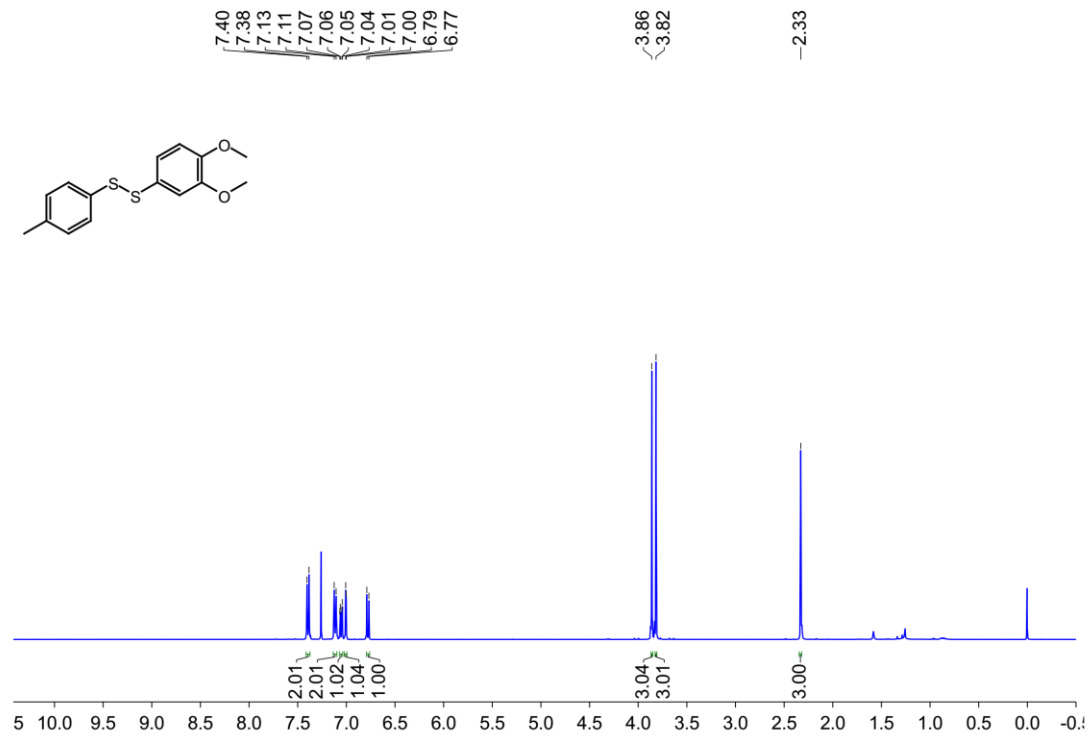
¹³C NMR spectrum of compound **3v**



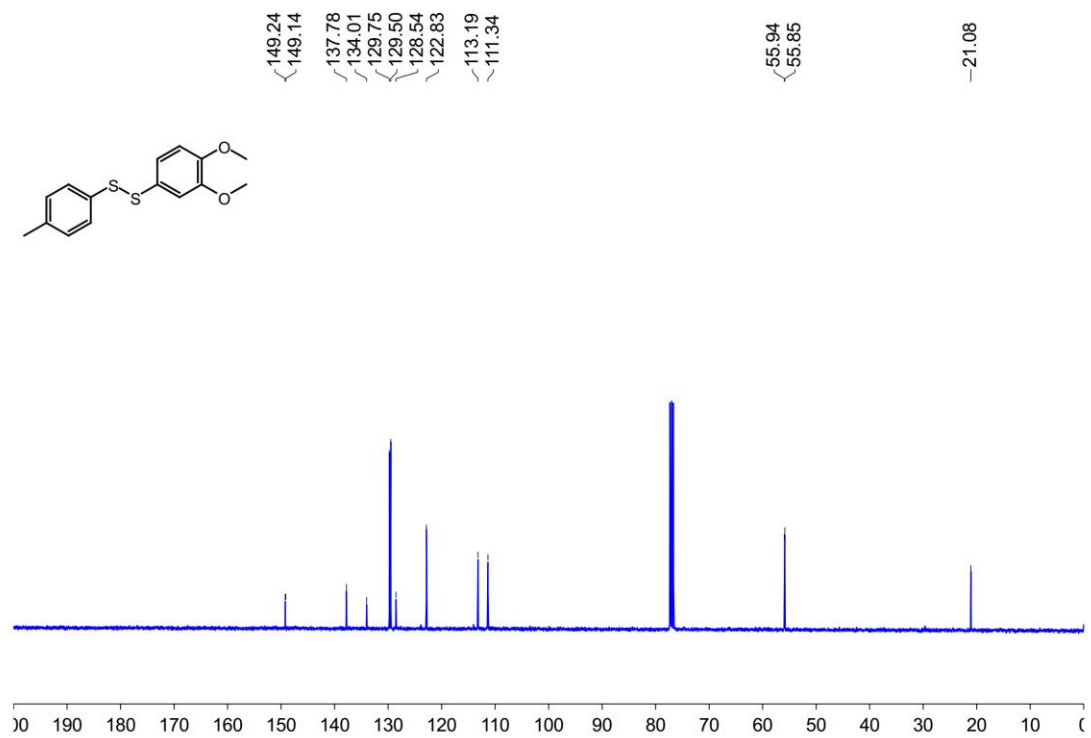
^1H NMR spectrum of compound **3w**



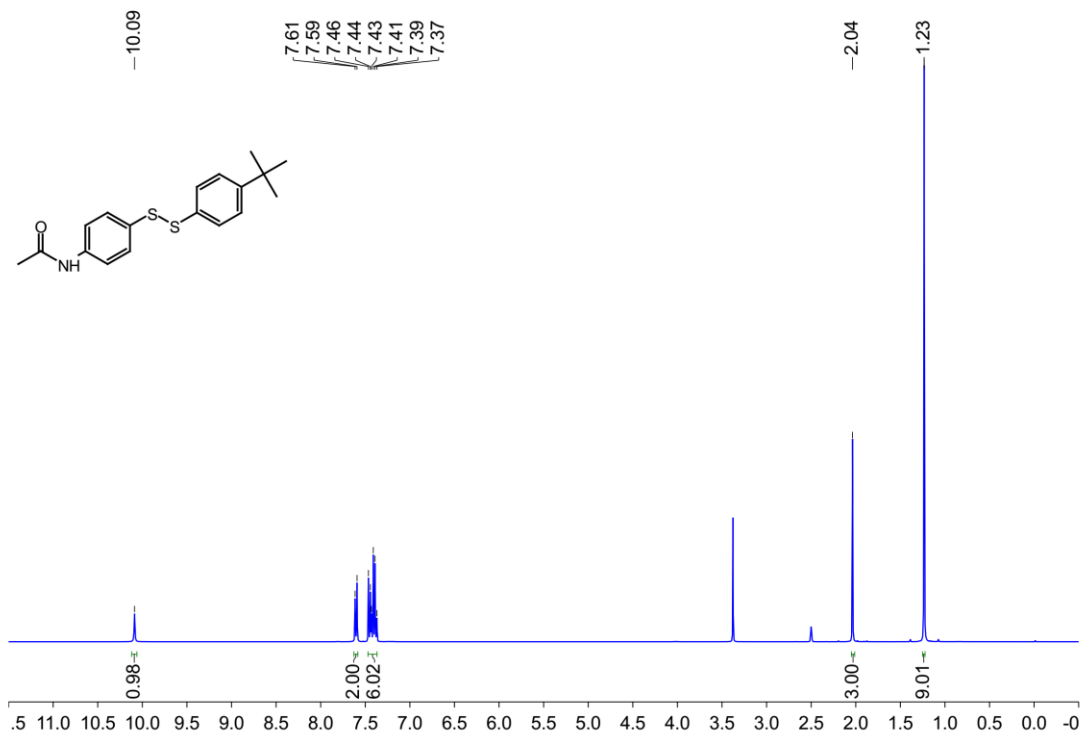
^{13}C NMR spectrum of compound **3w**



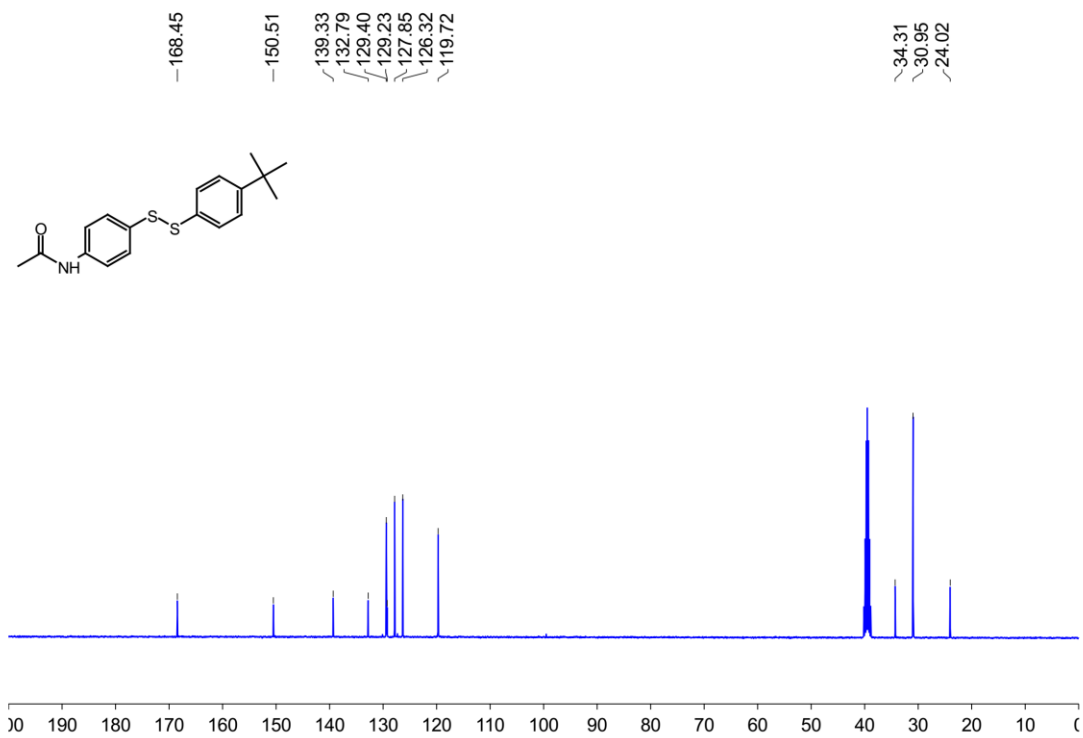
^1H NMR spectrum of compound **3x**



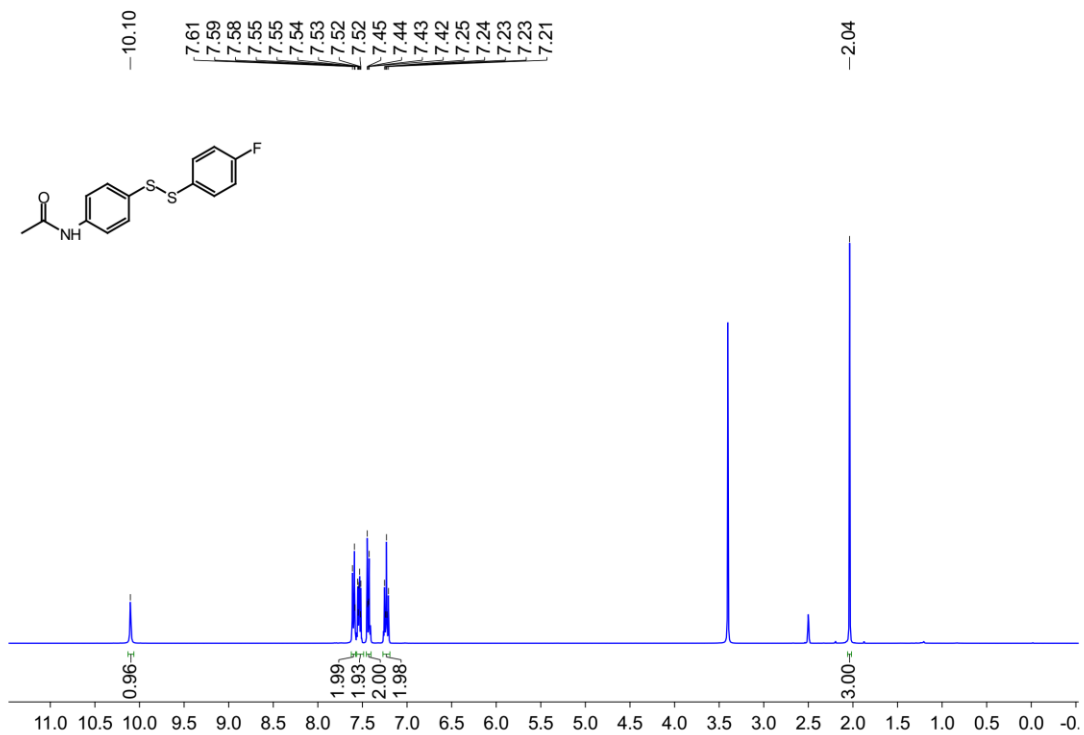
^{13}C NMR spectrum of compound **3x**



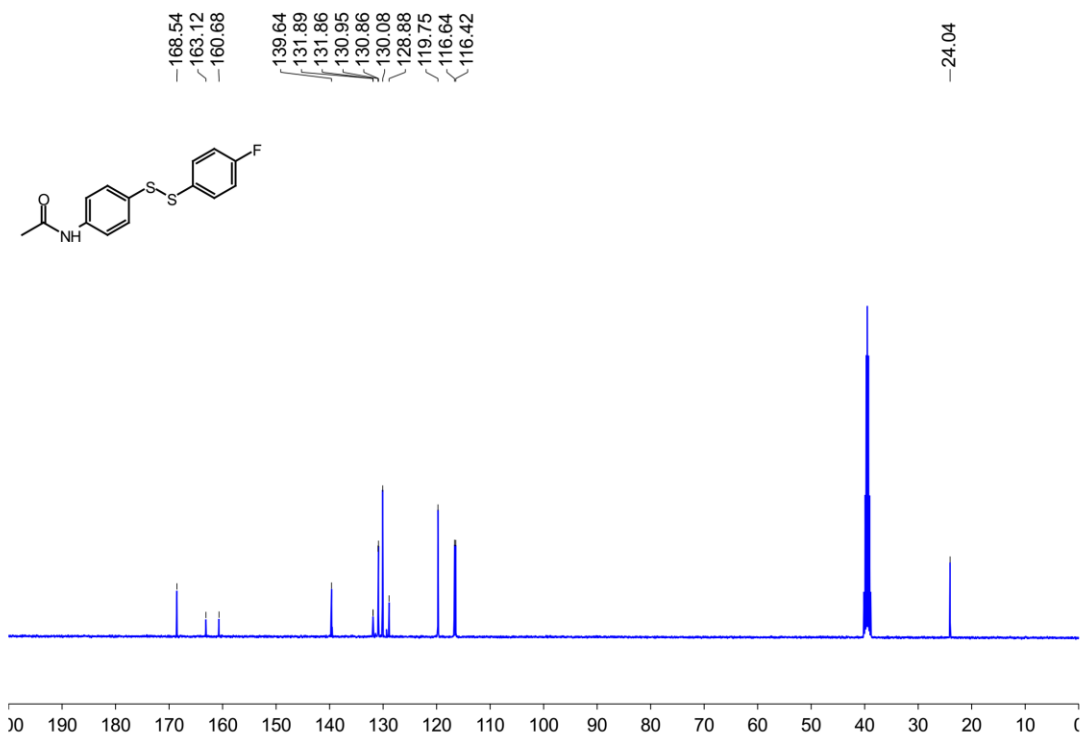
^1H NMR spectrum of compound **3y**



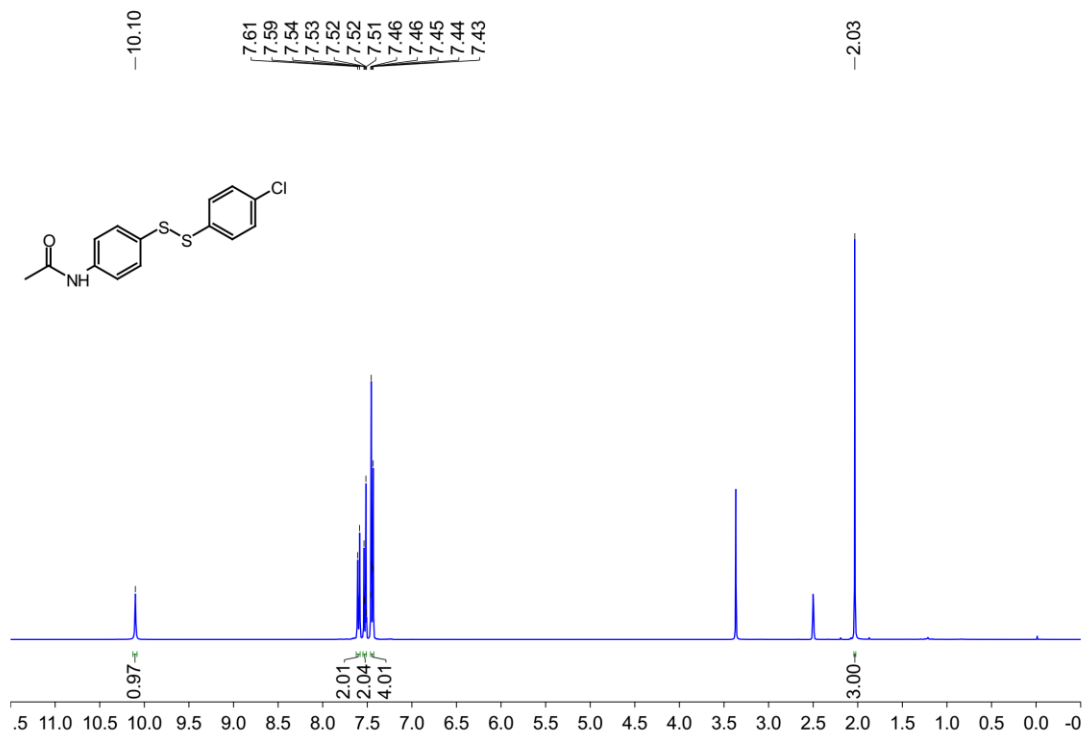
^{13}C NMR spectrum of compound **3y**



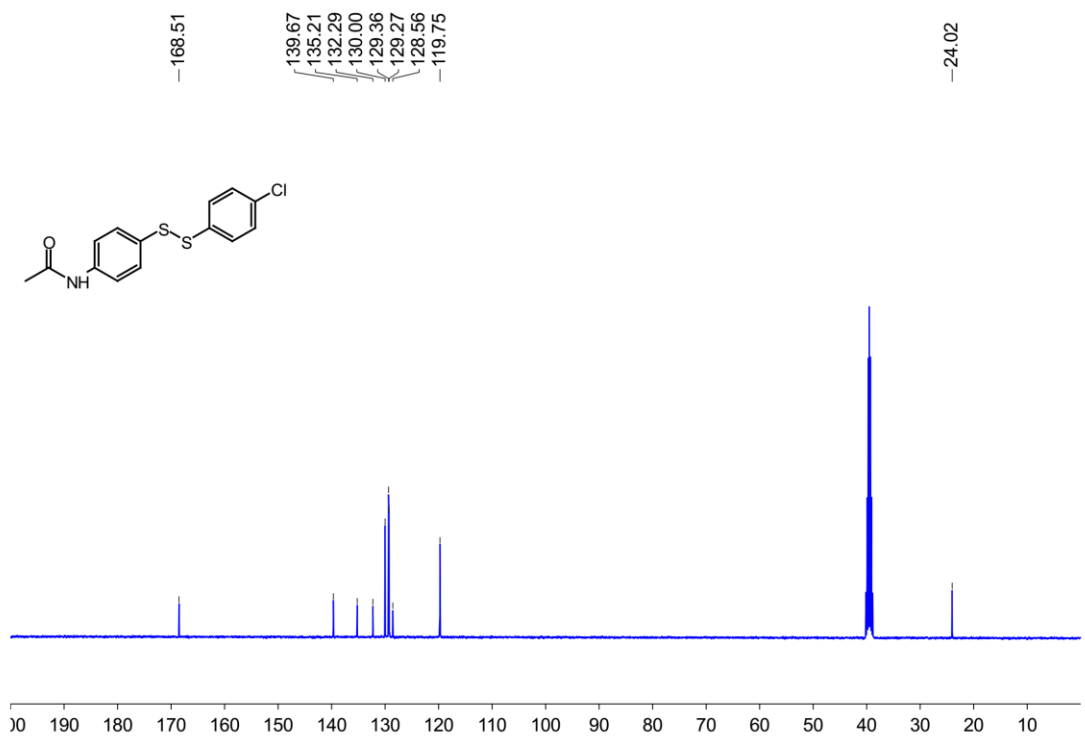
^1H NMR spectrum of compound **3z**



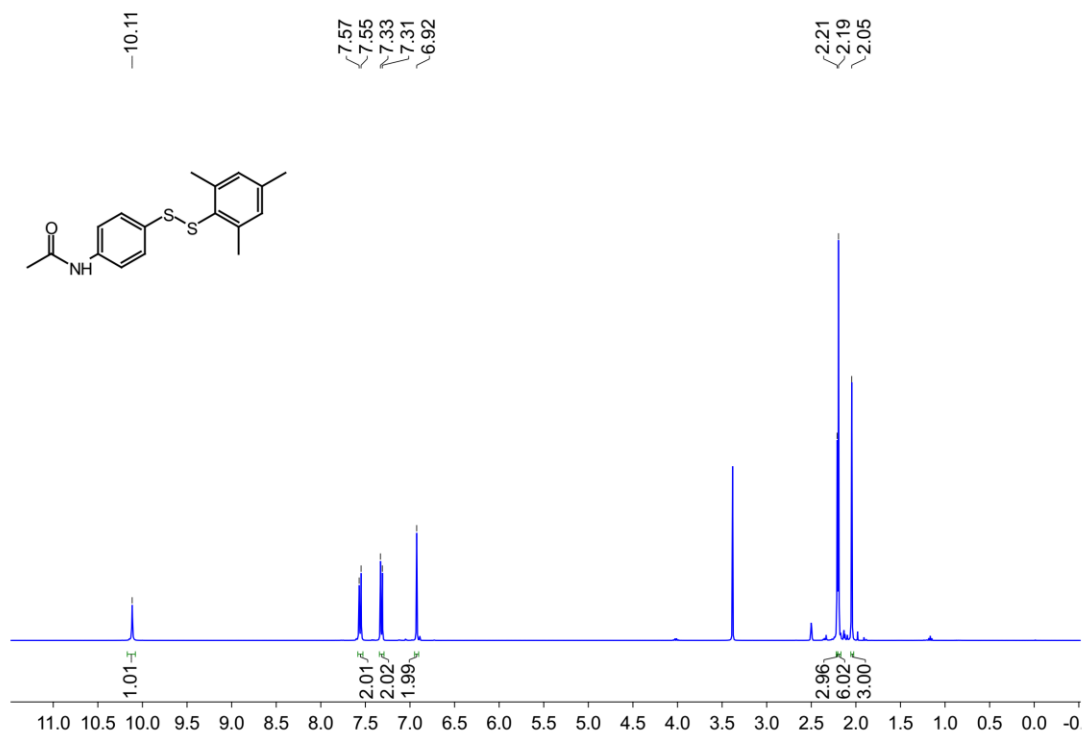
^{13}C NMR spectrum of compound **3z**



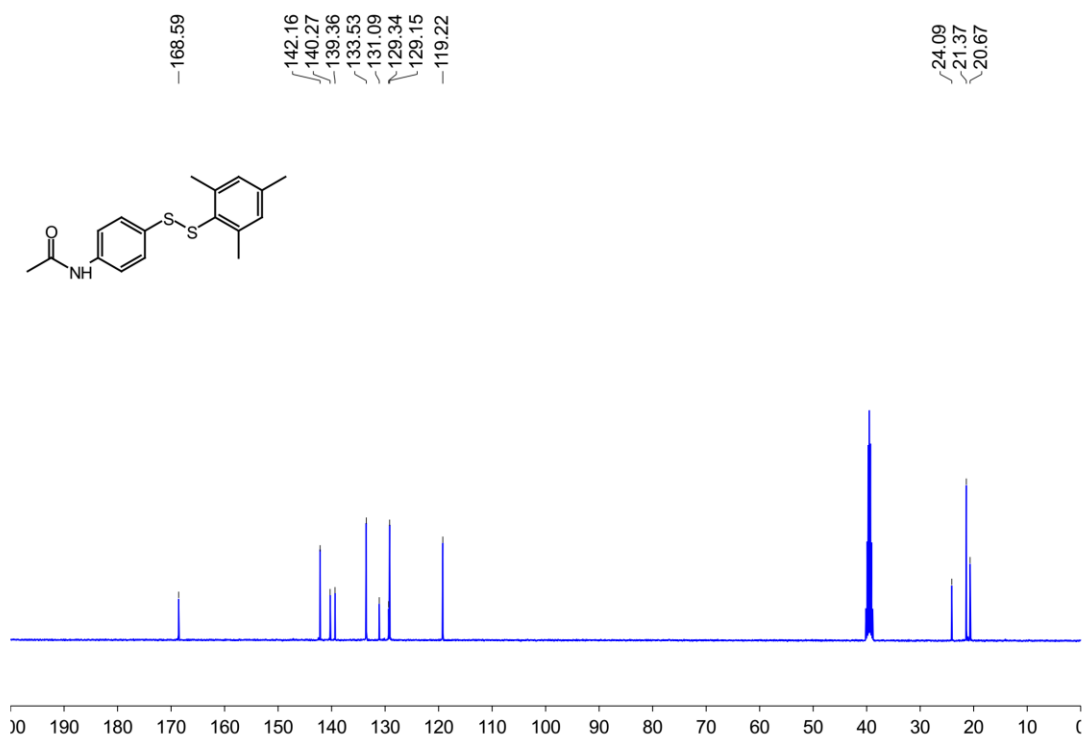
^1H NMR spectrum of compound **4a**



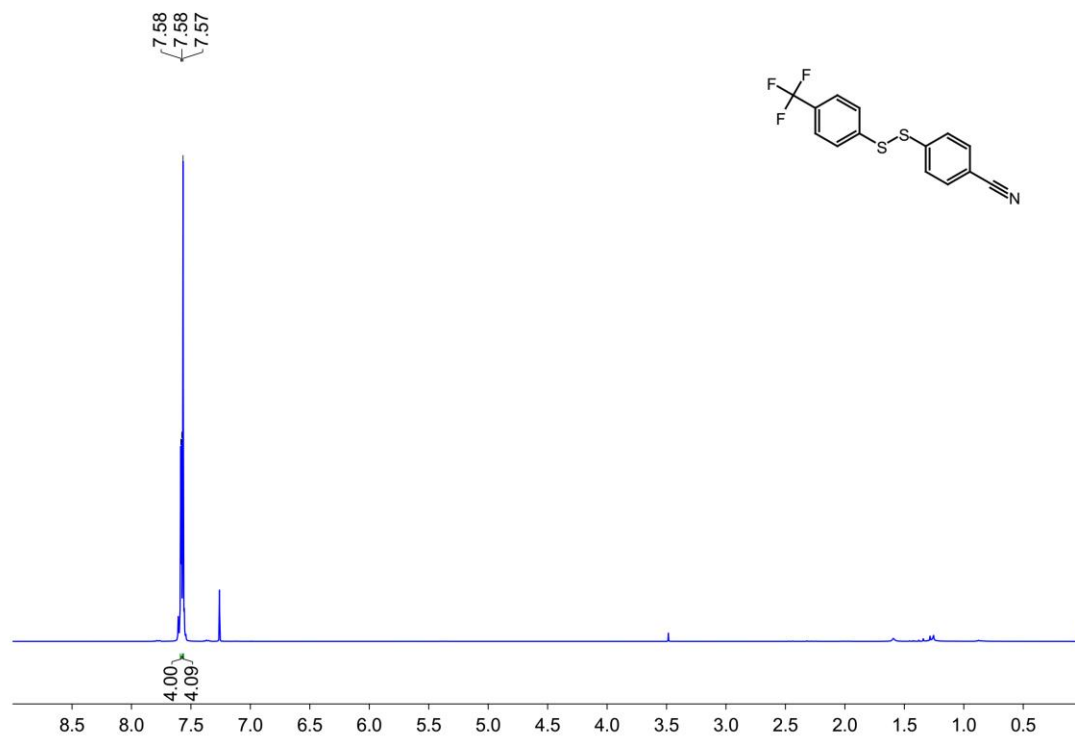
^{13}C NMR spectrum of compound **4a**



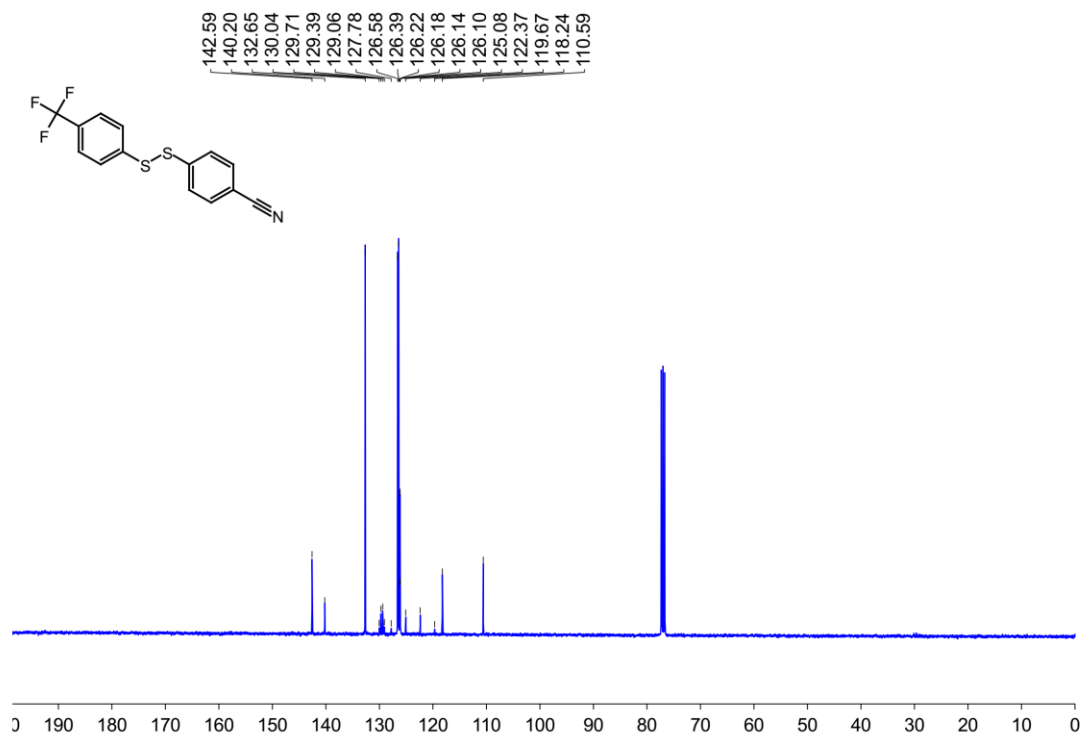
^1H NMR spectrum of compound **4b**



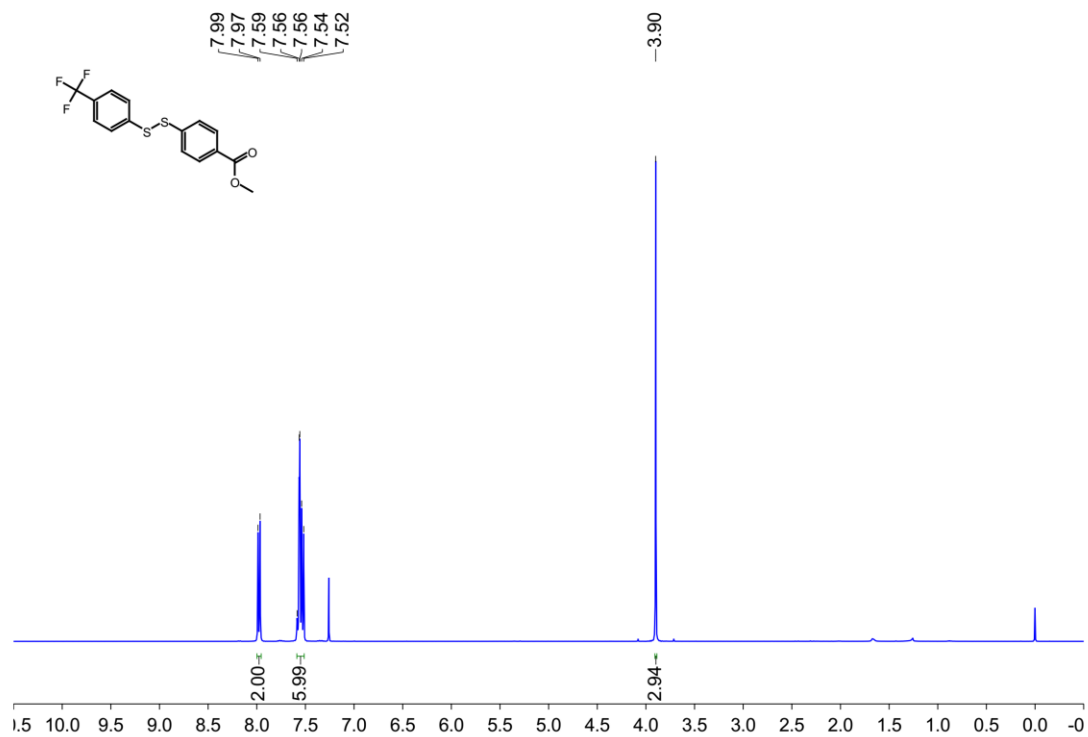
^{13}C NMR spectrum of compound **4b**



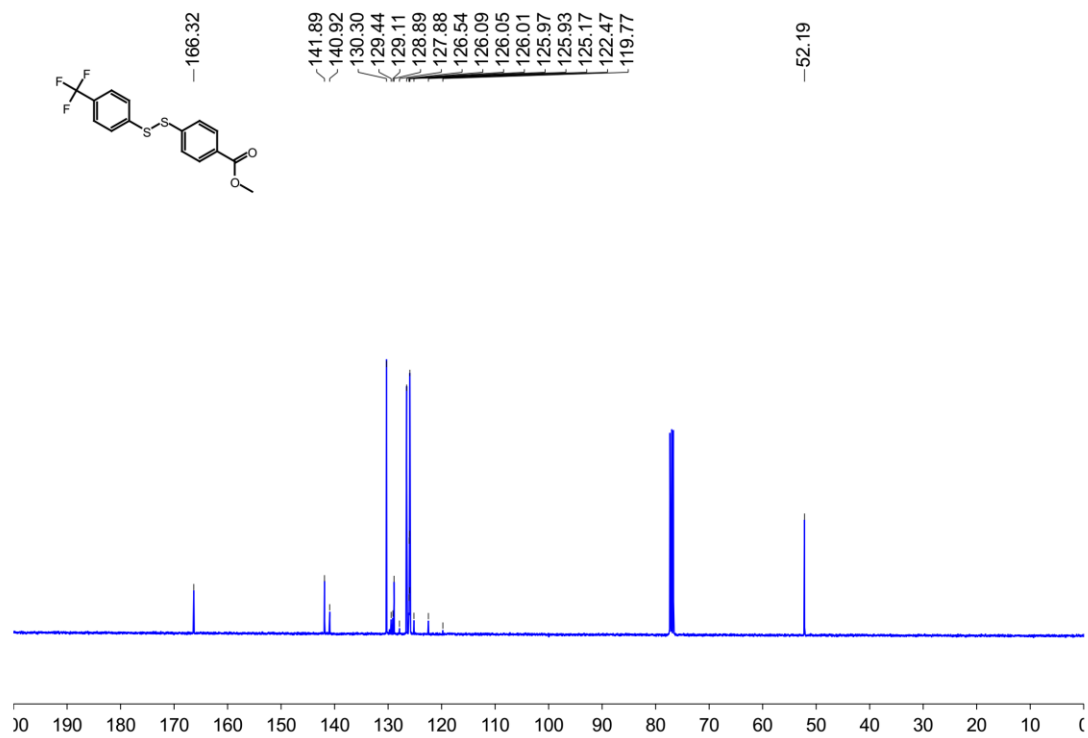
^1H NMR spectrum of compound **4c**



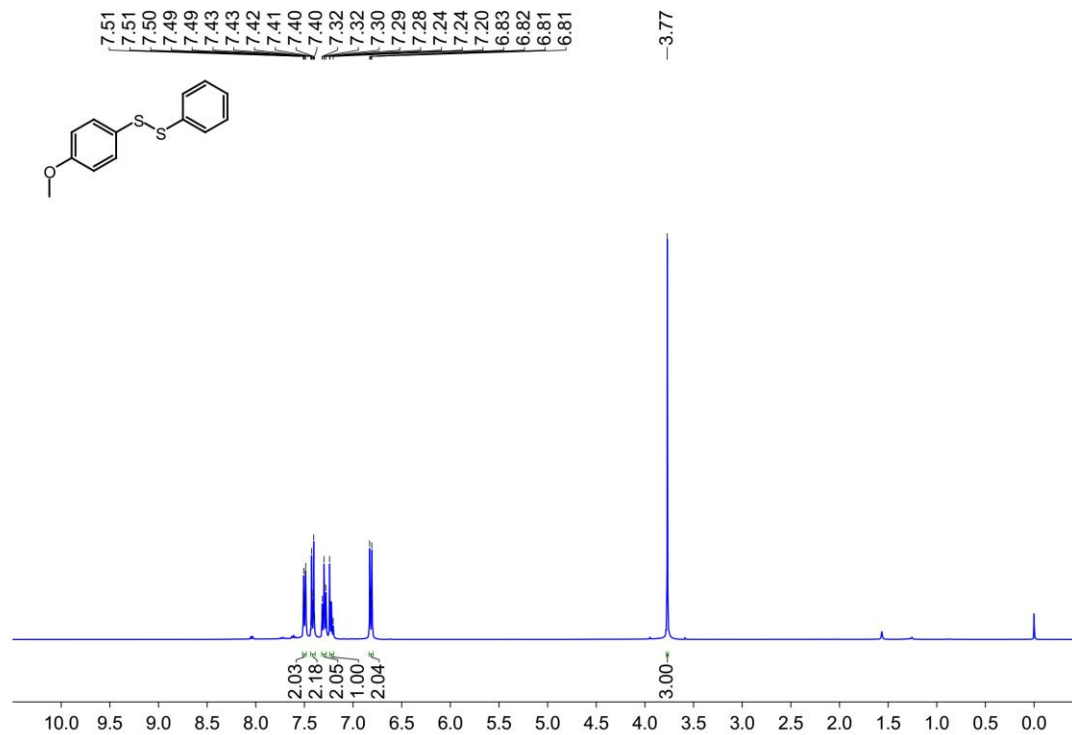
^{13}C NMR spectrum of compound **4c**



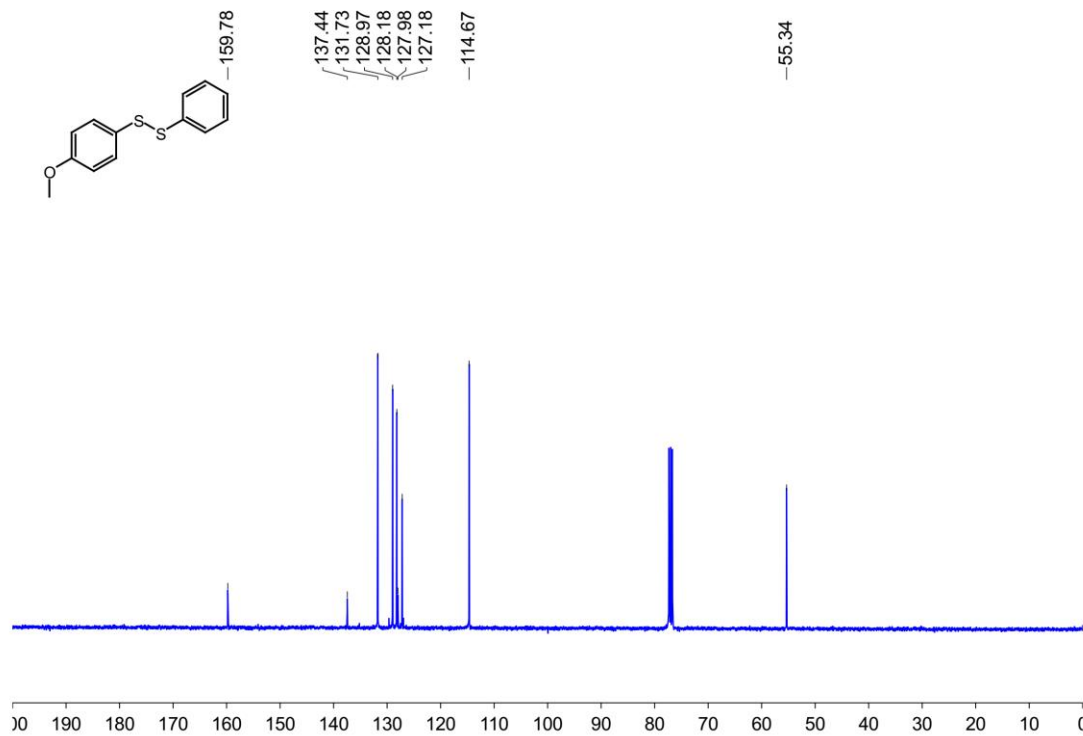
^1H NMR spectrum of compound **4d**



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



^1H NMR spectrum of compound **4e**



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