

Supporting Information

NBS-Promoted Regioselective Thiocyanatothiolation of Alkenes with Free-thiols and NH₄SCN

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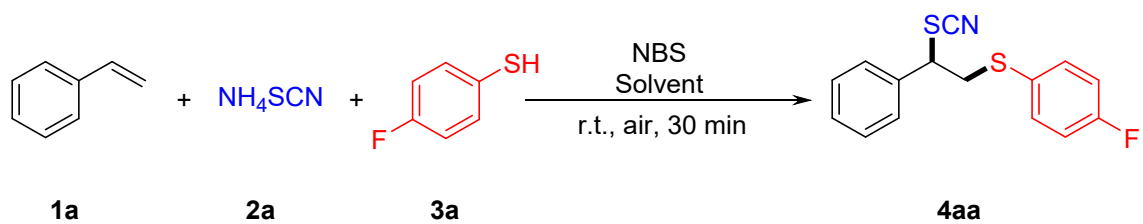
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1. General methods

Unless otherwise noted, materials were obtained from commercial suppliers and used without further purified. Reactions were monitored by thin layer chromatography (TLC). Yields refer to products isolated after purified by column chromatography. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded on a Bruker AV 400 MHz spectrometer using CDCl₃ or DMSO-*d*₆ as the solvent with TMS as the internal standard. Chemical shifts are reported in parts per million. Multiplicity was indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Coupling constants (*J*) were reported in Hz.

2. Optimization of reaction conditions

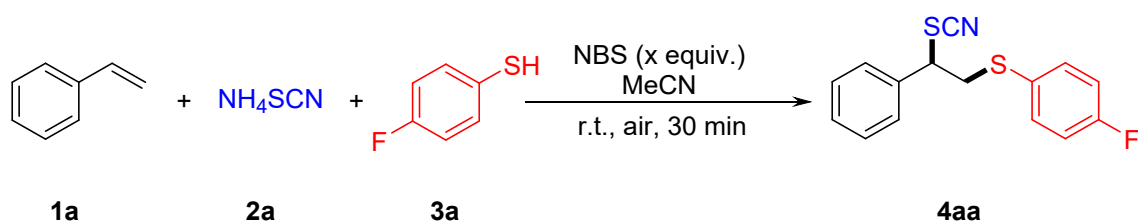
Table S1 Optimization of solvents^a



Entry	Solvent	Yield ^b (%)
1	MeCN	86
2	MeOH	23
3	THF	19
4	DCE	14
5	DCM	20
6	1,4-Dioxane	n.d.
7	EtOH	31
8	HFIP	34
9	H ₂ O	24
10	toluene	26
11	DMF	trace
12	MeCN/H ₂ O (4:1, v/v)	42
13	MeCN/H ₂ O (20:1, v/v)	64

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), **3a** (0.4 mmol, 2.0 equiv.), NBS (0.4 mmol, 2.0 equiv.), solvent (4.0 mL), under air, room temperature.

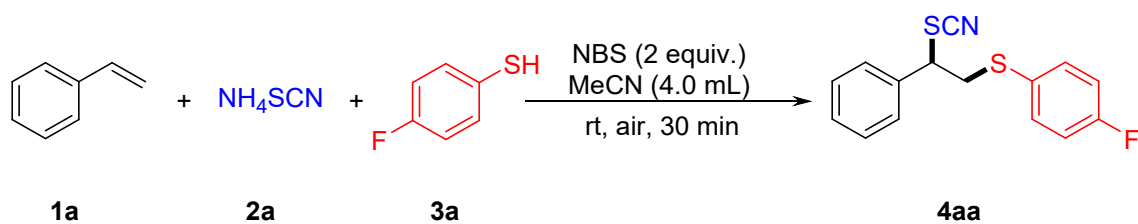
Table S2 Screening the amount of NBS



Entry	Oxidant	Equiv.	Yield ^b (%)
1	NBS	2	86
2	NBS	0.5	trace
3	NBS	1	19
4	NBS	1.5	57
5	NBS	4	32
6	NBS	6	38
7	NCS	2	n.d.
8	NIS	2	n.d.
9	-	-	n.d.

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), **3a** (0.4 mmol, 2.0 equiv.), NBS, MeCN (4.0 mL), under air, room temperature.

Table S3 Screening of the amount ratio



Entry	Ratio	Yield ^b (%)
1	1:1.2:2	86
2	1:1.2:1	68
3	1:1.2:1.5	65
2	1:1.2:3	45
3	1:1:2	72
4	1:1.5:2	74

5	1:2:2	71
6	1.5:1.2:1	63
7	2:1.2:1	67
8	2:1:2	64

^aReaction conditions: **1a**, **2a**, **3a**, NBS (0.4 mmol, 2.0 equiv.), NBS (0.4 mmol, 2.0 equiv.), MeCN (4.0 mL), under air, room temperature.

Table S4 Reaction time screening

1a	2a	3a	4aa
Entry	Time (min)	Yield ^b (%)	
1	15	75	
2	5	52	
3	30	86	
4	60	83	
5	240	82	

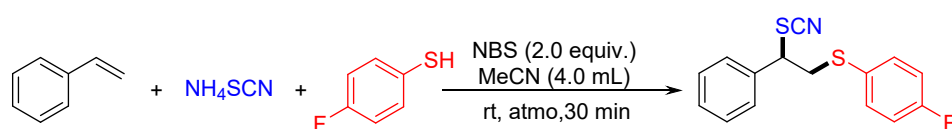
^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), **3a** (0.4 mmol, 1.0 equiv.), NBS (0.4 mmol, 2.0 equiv.), MeCN (4.0 mL), under air, room temperature, Time.

Table S5 Reaction temperature screening

1a	2a	3a	4aa
Entry	Temperature (°C)	Yield ^b (%)	
1	rt	86	
2	40	70	
3	60	56	
4	80	44	

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), **3a** (0.4 mmol, 2.0 equiv.), NBS (0.4 mmol, 2.0 equiv.), MeCN (4.0 mL), under air, temperature, 30 min.

Table S6 Atmosphere screening

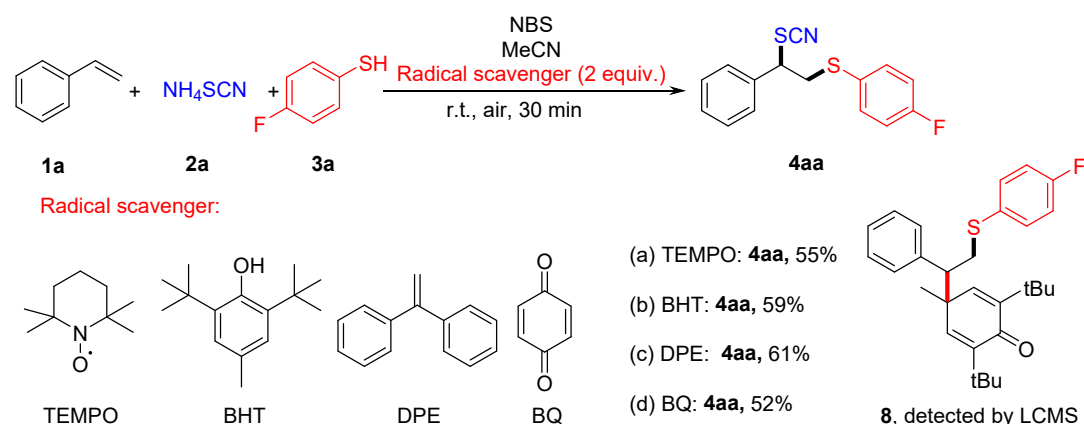


Entry	Atmosphere	Yield ^b (%)
1	Air	86
2	N ₂	68

^aReaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), **3a** (0.4 mmol, 1.0 equiv.), NBS (0.4 mmol, 2.0 equiv.), MeCN (4.0 mL), under air, room temperature.

3. Mechanistic investigation

Radical trapping experiment



In order to confirm whether the reaction undergoes a radical mechanism, commonly used radical scavengers such as 2,2,6,6-tetramethylpiperidinoxy (TEMPO), 1,1-diphenylethylene (DPE), butylated hydroxytoluene (BHT) and 1,4-Benzoquinone (BQ) was used respectively in radical capture and suppression experiments. Under the standard conditions, the radical scavenger (2.0 equiv. to **1a**) was added to the model reaction system at the beginning of the reaction. Additionally, after 30 min, a small amount of the reaction mixture added with TEMPO was used to measurement. The radical trapping product **9** can be observed by LC-MS.

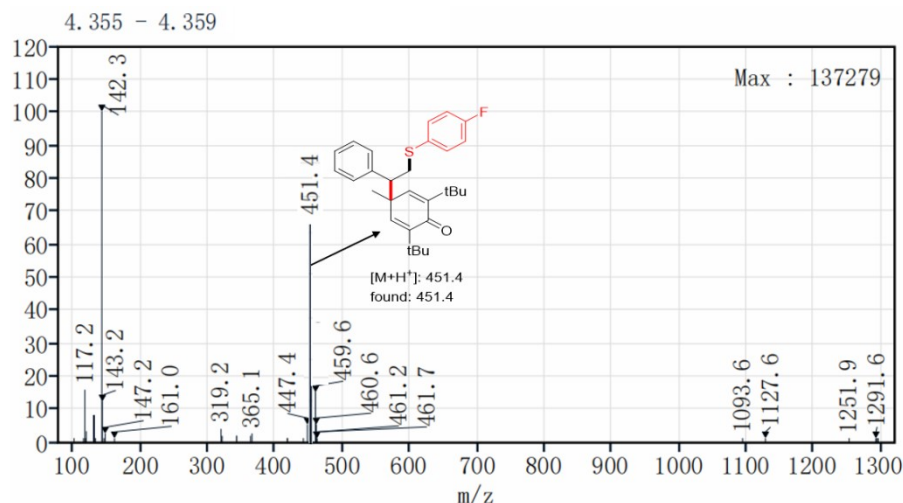
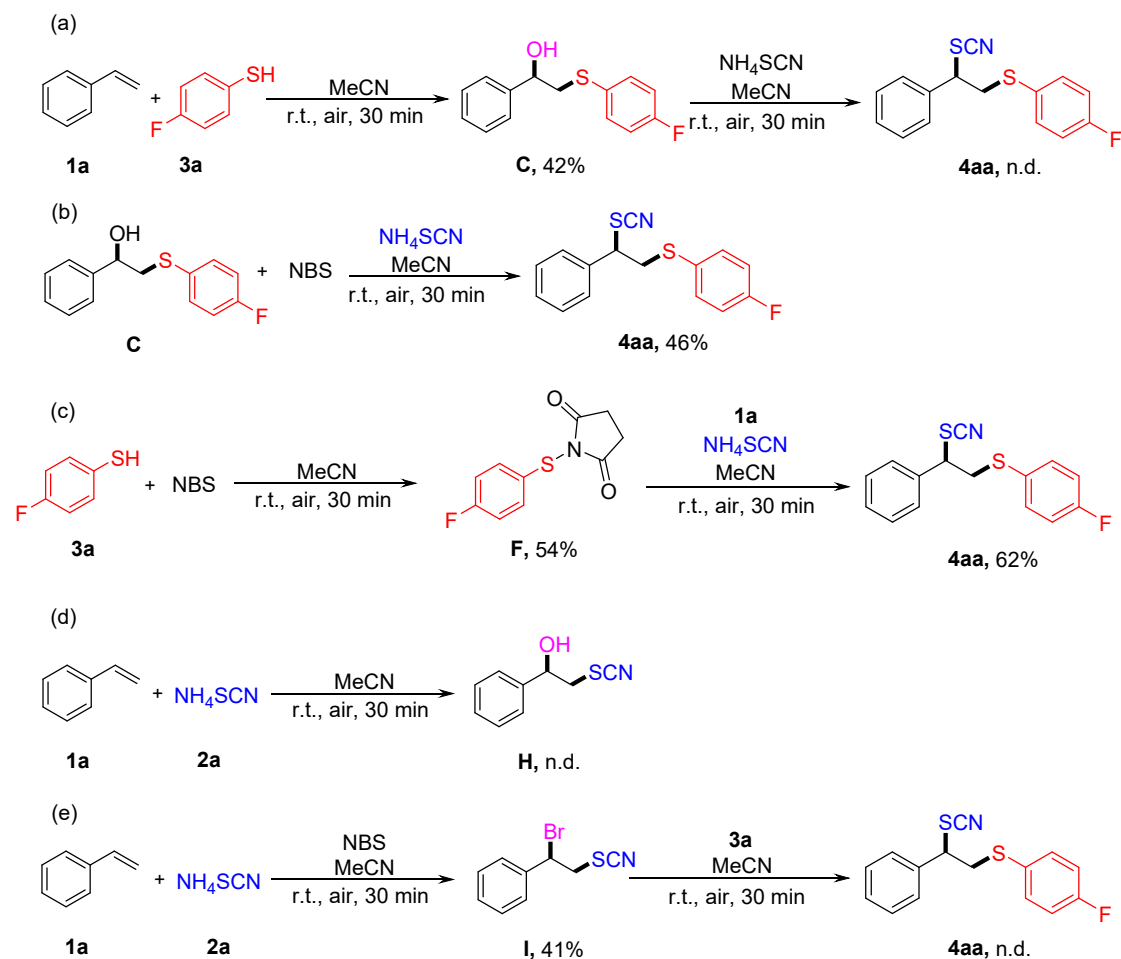


Figure S1. Mass spectrometry (LCMS) data of possible intermediate (with BHT).

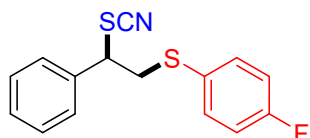
Control experiments



In an oven-dried round bottom flask (25.0 mL) equipped with a stir bar, styrene **1a** (0.2 mmol, 1.0 equiv.), **3a** (0.4 mmol, 2.0 equiv.), MeCN (4.0 mL). The reaction mixture was stirred under room temperature for 15 min. After reaction completion, the solvents were removed in vacuum,

the products **C** were obtained by silica gel column chromatography, then **C** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), MeCN(4.0 mL). The reaction mixture was stirred under room temperature for 15 min. After reaction completion, the solvents were removed in vacuum, the products **4aa** were obtained by silica gel column chromatography. Then proceed to the subsequent control steps with similar steps.

4. Characterization data of the products



(4-fluorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4aa).

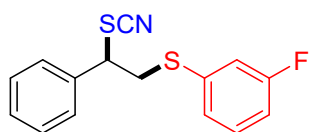
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4aa** as a colorless liquid (49.8 mg, 86% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.40 – 7.32 (m, 5H), 7.29 – 7.25 (m, 2H), 7.04 – 6.97 (m, 2H), 4.41 (dd, $J = 9.0, 6.6$ Hz, 1H), 3.65 – 3.50 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.68 (d, $^1J_{\text{FC}} = 248.7$ Hz), 136.24, 134.32 (d, $^3J_{\text{FC}} = 8.3$ Hz), 129.57, 129.32, 128.72 (d, $^4J_{\text{FC}} = 3.4$ Hz), 127.92, 116.61 (d, $^2J_{\text{FC}} = 22.0$ Hz), 111.21, 52.52, 41.11.

$^{19}\text{F NMR}$ (375 MHz, CDCl_3) δ -112.79.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{FNS}_2$ ($[\text{M}+\text{H}]^+$): 290.0473, Found: 290.0481.



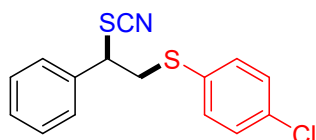
(3-fluorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ab).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ab** as a colorless liquid (41.7 mg, 72% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 (dd, $J = 4.9, 2.0$ Hz, 3H), 7.33 – 7.23 (m, 3H), 7.12 (ddd, $J = 7.8, 1.7, 1.0$ Hz, 1H), 7.02 (dt, $J = 9.1, 2.1$ Hz, 1H), 6.95 (tdd, $J = 8.4, 2.5, 0.9$ Hz, 1H), 4.47 (dd, $J = 9.1, 6.4$ Hz, 1H), 3.72 – 3.58 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.92 (d, $J = 249.5$ Hz), 136.25 (d, $J = 7.8$ Hz), 136.17, 130.79 (d, $J = 8.5$ Hz), 129.69, 129.38, 127.90, 126.18 (d, $J = 3.0$ Hz), 117.59 (d, $J = 22.8$ Hz), 114.60 (d, $J = 21.1$ Hz), 111.12, 52.39, 39.80.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{FNS}_2$ ($[\text{M}+\text{H}]^+$): 290.0473, Found: 290.0478.



(4-chlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ac).

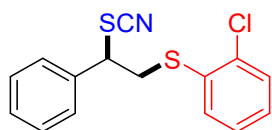
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ac** as a colorless liquid (49.5 mg, 81% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.40 – 7.35 (m, 3H), 7.27 (d, $J = 5.8$ Hz, 6H), 4.43 (dd, $J = 9.1,$

6.4 Hz, 1H), 3.72 – 3.52 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 136.03, 133.83, 132.56, 132.18, 129.52, 129.25, 127.79, 111.04, 52.31, 40.23.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{ClNS}_2$ ($[\text{M}+\text{H}]^+$): 306.0178, Found: 306.0198.



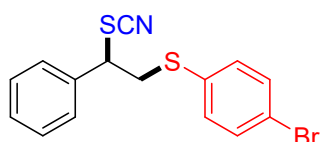
(2-chlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ad).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ad** as a colorless liquid (43.4 mg, 71% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.41 (dd, $J = 5.9, 3.5$ Hz, 1H), 7.39 – 7.34 (m, 4H), 7.33 – 7.27 (m, 2H), 7.21 (dd, $J = 5.9, 3.5$ Hz, 2H), 4.49 (t, $J = 7.7$ Hz, 1H), 3.68 (d, $J = 7.8$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 136.24, 136.16, 132.83, 132.20, 130.37, 129.61, 129.32, 128.81, 127.87, 127.52, 111.12, 52.54, 38.80.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{ClNS}_2$ ($[\text{M}+\text{H}]^+$): 306.0178, Found: 306.0180.



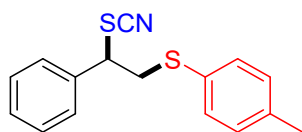
(4-bromophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ae).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ae** as a colorless liquid (58.8 mg, 84% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.40 (m, 2H), 7.37 (q, $J = 3.7$ Hz, 3H), 7.27 (dd, $J = 4.4, 2.8$ Hz, 2H), 7.23 – 7.18 (m, 2H), 4.43 (dd, $J = 9.2, 6.3$ Hz, 1H), 3.69 – 3.54 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 136.16, 133.03, 132.81, 132.59, 129.68, 129.40, 127.93, 121.90, 111.14, 52.43, 40.21.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{BrNS}_2$ ($[\text{M}+\text{H}]^+$): 349.9673, Found: 349.9678.



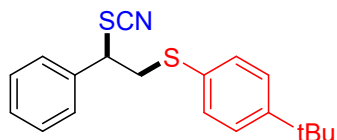
(2-phenyl-2-thiocyanatoethyl)(p-tolyl)sulfane (4af).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4af** as a colorless liquid (44.0 mg, 77% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.36 (m, 3H), 7.29 (dd, $J = 8.0, 2.2$ Hz, 4H), 7.14 (d, $J = 7.9$ Hz, 2H), 4.42 (dd, $J = 8.8, 6.7$ Hz, 1H), 3.64 – 3.51 (m, 2H), 2.35 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 138.08, 136.60, 132.00, 130.26, 129.90, 129.46, 129.27, 127.95, 111.35, 52.51, 40.61, 21.21.

HRMS m/z (ESI) calcd. for $\text{C}_{16}\text{H}_{16}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 286.0724, Found: 286.0745.



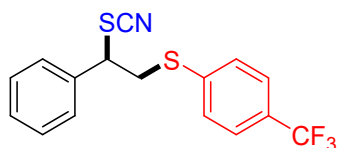
(2-(tert-butyl)phenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ag).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ag** as a colorless liquid (51.7 mg, 79% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.36 (td, $J = 4.8, 4.4, 1.8$ Hz, 3H), 7.34 – 7.28 (m, 6H), 4.46 (dd, $J = 8.8, 6.7$ Hz, 1H), 3.65 – 3.54 (m, 2H), 1.32 (s, 9H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 151.18, 136.61, 131.49, 130.10, 129.49, 129.28, 128.00, 126.56, 111.38, 52.64, 40.42, 34.75, 31.37.

HRMS m/z (ESI) calcd. for $\text{C}_{19}\text{H}_{22}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 328.1194, Found: 328.1198.



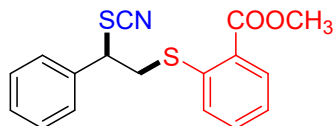
(2-phenyl-2-thiocyanatoethyl)(4-(trifluoromethyl)phenyl)sulfane (4ah).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ah** as a colorless liquid (44.1 mg, 65% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.55 (d, $J = 8.3$ Hz, 2H), 7.43 (d, $J = 8.2$ Hz, 2H), 7.37 (td, $J = 6.8, 6.3, 3.0$ Hz, 3H), 7.30 (dd, $J = 7.7, 1.6$ Hz, 2H), 4.90 (t, $J = 6.8$ Hz, 1H), 3.43 – 3.39 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 139.33, 135.89, 129.78, 129.65, 129.44, 129.05, 127.88, 126.23 (q, $J = 3.8$ Hz), 125.38, 122.68, 111.07, 52.27, 39.08.

HRMS m/z (ESI) calcd. for $\text{C}_{16}\text{H}_{13}\text{F}_3\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 340.0442, Found: 340.0451.



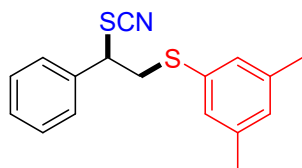
methyl 2-((2-phenyl-2-thiocyanatoethyl)thio)benzoate (4ai).

Purified by column chromatography on silica gel (10:1 petroleum ether/ethyl acetate) afforded **4ai** as a White solid (44.8 mg, 68% yield, mp $71^\circ\text{C}\sim 73^\circ\text{C}$).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.93 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.50 – 7.46 (m, 1H), 7.41 – 7.32 (m, 6H), 7.25 (td, $J = 7.8, 1.1$ Hz, 1H), 4.60 (dd, $J = 8.6, 6.6$ Hz, 1H), 3.87 (s, 3H), 3.77 – 3.65 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.91, 138.15, 136.44, 132.69, 131.48, 129.91, 129.62, 129.39, 127.86, 127.57, 125.67, 111.26, 52.39, 52.05, 38.33.

HRMS m/z (ESI) calcd. For $\text{C}_{17}\text{H}_{16}\text{NO}_2\text{S}_2$ ($[\text{M}+\text{H}]^+$): 330.0122, Found: 330.0134.



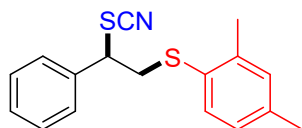
(3,5-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4aj).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4aj** as a colorless liquid (44.3 mg, 74% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 (dt, $J = 3.9, 2.2$ Hz, 3H), 7.33 – 7.28 (m, 2H), 6.97 (s, 2H), 6.90 (s, 1H), 4.47 (dd, $J = 9.0, 6.4$ Hz, 1H), 3.68 – 3.52 (m, 2H), 2.30 (s, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 139.14, 136.59, 133.18, 130.58, 129.51, 129.48, 129.24, 128.73, 127.98, 111.41, 52.58, 39.95, 21.29.

HRMS m/z (ESI) calcd. for $\text{C}_{17}\text{H}_{18}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 300.0881, Found: 300.0892.



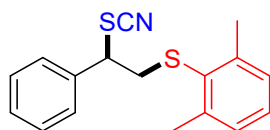
(2,4-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ak).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ak** as a colorless liquid (45.5 mg, 76% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.39 (td, $J = 5.4, 2.3$ Hz, 3H), 7.32 – 7.27 (m, 3H), 7.06 – 6.98 (m, 2H), 4.42 (dd, $J = 8.5, 7.0$ Hz, 1H), 3.55 (dd, $J = 7.8, 4.0$ Hz, 2H), 2.33 (d, $J = 3.4$ Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 140.11, 138.14, 136.70, 132.24, 131.75, 129.47, 129.27, 129.06, 127.95, 127.69, 111.34, 52.54, 39.71, 21.11, 20.62.

HRMS m/z (ESI) calcd. for $\text{C}_{17}\text{H}_{18}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 300.0881, Found: 300.0896.



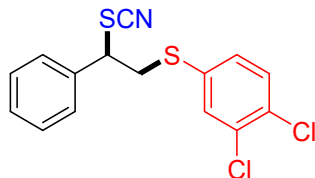
(2,6-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4al).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4al** as a colorless liquid (40.7 mg, 68% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.40 – 7.35 (m, 3H), 7.29 (dd, $J = 6.7, 2.9$ Hz, 2H), 7.14 (dd, $J = 8.7, 6.0$ Hz, 1H), 7.09 (d, $J = 6.9$ Hz, 2H), 4.38 (dd, $J = 9.0, 6.7$ Hz, 1H), 3.52 – 3.40 (m, 2H), 2.43 (s, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 142.96, 136.53, 131.92, 129.51, 129.24, 128.91, 128.53, 127.89, 111.19, 52.94, 39.72, 22.05.

HRMS m/z (ESI) calcd. for $\text{C}_{17}\text{H}_{18}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 300.0881, Found: 300.0894.



(3,4-dichlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4am).

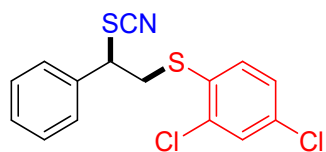
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4am** as a colorless liquid (47.0 mg, 69% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.41 (d, $J = 2.2$ Hz, 1H), 7.39 – 7.35 (m, 3H), 7.32 – 7.26 (m, 3H), 7.17 (dd, $J = 8.4, 2.2$ Hz, 1H), 4.48 (dd, $J = 8.3, 7.2$ Hz, 1H), 3.67 (dd, $J = 7.7, 1.7$ Hz, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 137.17, 135.97, 134.41, 133.35, 131.44, 130.23, 129.73, 129.40,

127.88, 127.82, 111.05, 52.54, 39.05.

HRMS m/z (ESI) calcd. for $C_{15}H_{12}Cl_2NS_2$ ($[M+H]^+$): 339.9788, Found: 339.9801.



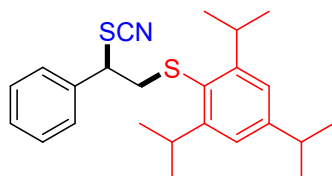
(2,4-dichlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4an).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4an** as a colorless liquid (42.9 mg, 63% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.36 (ddt, $J = 8.5, 5.0, 3.0$ Hz, 5H), 7.31 – 7.26 (m, 2H), 7.16 (dd, $J = 8.4, 2.2$ Hz, 1H), 4.47 (dd, $J = 9.3, 6.2$ Hz, 1H), 3.71 – 3.57 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 135.85, 134.14, 133.34, 132.59, 132.03, 131.10, 130.15, 129.78, 129.40, 127.89, 111.03, 52.43, 40.22.

HRMS m/z (ESI) calcd. for $C_{15}H_{12}Cl_2NS_2$ ($[M+H]^+$): 339.9788, Found: 339.9798.



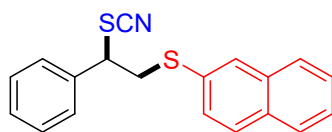
(2-phenyl-2-thiocyanatoethyl)(2,4,6-triisopropylphenyl)sulfane (4ao).

Purified by column chromatography on silica gel (10:1 petroleum ether/ethyl acetate) afforded **4ao** as a white solid (49.3 mg, 62% yield, mp 105°C~107 °C).

1H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.36 (m, 3H), 7.36 – 7.31 (m, 2H), 7.01 (s, 2H), 4.47 (dd, $J = 9.0, 6.6$ Hz, 1H), 3.73 (p, $J = 6.9$ Hz, 2H), 3.48 – 3.28 (m, 2H), 2.88 (p, $J = 6.9$ Hz, 1H), 1.25 (d, $J = 6.9$ Hz, 6H), 1.21 (dd, $J = 6.8, 4.0$ Hz, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.11, 150.43, 136.88, 129.50, 129.27, 127.81, 127.13, 122.24, 111.17, 52.72, 42.56, 34.41, 31.77, 24.68, 24.64, 24.01.

HRMS m/z (ESI) calcd. for $C_{24}H_{32}NS_2$ ($[M+H]^+$): 398.1976, Found: 398.1996.



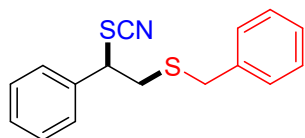
naphthalen-2-yl(2-phenyl-2-thiocyanatoethyl)sulfane (4ap).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ap** as a colorless liquid (45.6 mg, 71% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.75 (m, 4H), 7.54 – 7.47 (m, 2H), 7.43 (dd, $J = 8.5, 1.9$ Hz, 1H), 7.36 (dt, $J = 4.7, 2.6$ Hz, 3H), 7.32 – 7.27 (m, 2H), 4.50 (dd, $J = 9.2, 6.3$ Hz, 1H), 3.79 (dd, $J = 14.1, 6.3$ Hz, 1H), 3.68 (dd, $J = 14.1, 9.2$ Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.38, 133.80, 132.50, 130.98, 129.74, 129.58, 129.32, 129.23, 128.25, 127.96, 127.88, 127.52, 126.98, 126.59, 111.36, 52.41, 39.79.

HRMS m/z (ESI) calcd. for $C_{19}H_{16}NS_2$ ($[M+H]^+$): 322.0724, Found: 322.0745.



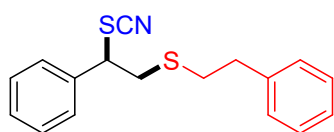
benzyl(2-phenyl-2-thiocyanatoethyl)sulfane (4aq).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4aq** as a colorless liquid (36.5 mg, 64% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.38 (ddd, *J* = 4.7, 2.8, 1.3 Hz, 3H), 7.36 – 7.32 (m, 2H), 7.29 (td, *J* = 7.1, 6.4, 1.5 Hz, 3H), 7.25 – 7.22 (m, 2H), 4.28 (t, *J* = 7.8 Hz, 1H), 3.65 (d, *J* = 2.9 Hz, 2H), 3.19 – 3.15 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 137.65, 136.75, 129.48, 129.28, 129.10, 128.92, 127.94, 127.65, 111.44, 53.22, 37.28, 36.99.

HRMS *m/z* (ESI) calcd. for C₁₆H₁₆NS₂ ([M+H]⁺): 286.0724, Found: 286.0730.



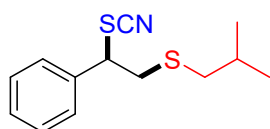
phenethyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ar).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ar** as a colorless liquid (39.5 mg, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.36 (m, 3H), 7.36 – 7.28 (m, 4H), 7.24 (d, *J* = 7.1 Hz, 1H), 7.18 – 7.13 (m, 2H), 4.48 (dd, *J* = 8.7, 6.7 Hz, 1H), 3.31 – 3.18 (m, 2H), 2.88 – 2.80 (m, 2H), 2.73 (dd, *J* = 8.1, 6.8 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 139.96, 136.83, 129.55, 129.34, 128.72, 128.66, 127.92, 126.73, 111.50, 53.60, 37.94, 36.31, 34.60.

HRMS *m/z* (ESI) calcd. for C₁₇H₁₈NS₂ ([M+H]⁺): 300.0881, Found: 300.0891.



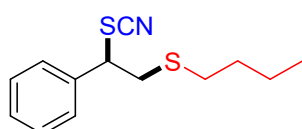
isobutyl(2-phenyl-2-thiocyanatoethyl)sulfane (4as).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4as** as a colorless liquid (29.7 mg, 59% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.32 (m, 5H), 4.55 (dd, *J* = 8.8, 6.6 Hz, 1H), 3.31 – 3.17 (m, 2H), 2.35 (dd, *J* = 6.9, 2.0 Hz, 2H), 1.75 (dp, *J* = 13.4, 6.7 Hz, 1H), 0.95 (dd, *J* = 6.6, 5.5 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 136.98, 129.50, 129.31, 127.94, 111.59, 53.75, 42.29, 38.28, 28.69, 22.02, 21.95.

HRMS *m/z* (ESI) calcd. for C₁₃H₁₈NS₂ ([M+H]⁺): 252.0881, Found: 252.0889.



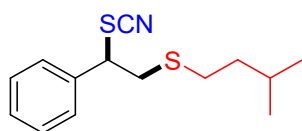
butyl(2-phenyl-2-thiocyanatoethyl)sulfane (4at).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4at** as a colorless liquid (31.2 mg, 62% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.43 – 7.30 (m, 5H), 4.88 (dd, $J = 7.6, 5.9$ Hz, 1H), 3.05 – 2.92 (m, 2H), 2.52 – 2.45 (m, 2H), 1.55 – 1.50 (m, 2H), 1.38 (q, $J = 7.5$ Hz, 2H), 0.90 (t, $J = 7.3$ Hz, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.94, 129.51, 129.31, 127.92, 111.59, 53.68, 37.69, 32.75, 31.64, 21.94, 13.71.

HRMS m/z (ESI) calcd. for $\text{C}_{13}\text{H}_{18}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 252.0881, Found: 252.0893.



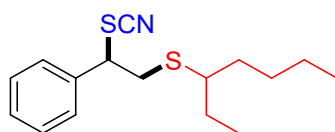
isopentyl(2-phenyl-2-thiocyanatoethyl)sulfane (4au).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4au** as a colorless liquid (31.8 mg, 60% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.47 – 7.30 (m, 5H), 4.88 (dd, $J = 7.5, 5.9$ Hz, 1H), 3.05 – 2.92 (m, 2H), 2.52 – 2.43 (m, 2H), 1.63 (dt, $J = 13.3, 6.7$ Hz, 1H), 1.48 – 1.40 (m, 2H), 0.88 (d, $J = 6.6$ Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.96, 129.50, 129.30, 127.92, 111.54, 53.71, 38.52, 37.70, 31.09, 27.45, 22.33.

HRMS m/z (ESI) calcd. for $\text{C}_{14}\text{H}_{20}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 266.1037, Found: 266.1049.



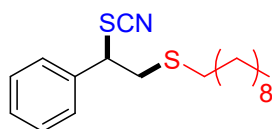
heptan-3-yl(2-phenyl-2-thiocyanatoethyl)sulfane (4av).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4av** as a colorless liquid (34.6 mg, 59% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.37 (dd, $J = 8.4, 4.7$ Hz, 5H), 4.60 – 4.49 (m, 1H), 3.32 – 3.18 (m, 2H), 2.51 – 2.39 (m, 2H), 1.40 – 1.15 (m, 9H), 0.93 – 0.81 (m, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 137.14, 129.48, 129.31, 127.97, 111.49, 53.90, 39.52, 38.54, 37.87, 32.45, 28.96, 25.67, 23.06, 14.15, 10.87.

HRMS m/z (ESI) calcd. for $\text{C}_{16}\text{H}_{24}\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 294.1350, Found: 294.1370.



decyl(2-phenyl-2-thiocyanatoethyl)sulfane (4aw).

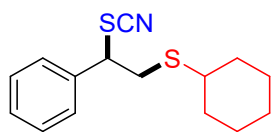
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4aw** as a colorless liquid (35.6 mg, 53% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.45 – 7.31 (m, 5H), 4.55 (dd, $J = 8.8, 6.7$ Hz, 1H), 3.33 – 3.18 (m, 2H), 2.50 – 2.41 (m, 2H), 1.52 (q, $J = 7.6$ Hz, 2H), 1.33 – 1.21 (m, 14H), 0.88 (t, $J = 6.9$ Hz, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.98, 129.52, 129.33, 127.94, 53.72, 37.73, 33.12, 32.03,

29.67, 29.62, 29.43, 29.27, 28.85, 22.82, 14.25.

HRMS m/z (ESI) calcd. for $C_{19}H_{30}NS_2$ ($[M+H]^+$): 336.1820, Found: 336.1841.



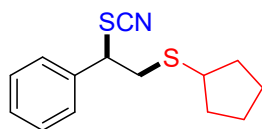
cyclohexyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ax).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ax** as a colorless liquid (28.9 mg, 52% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.29 (m, 5H), 4.55 (dd, $J = 8.9, 6.6$ Hz, 1H), 3.37 – 3.19 (m, 2H), 2.57 (ddt, $J = 10.5, 7.6, 3.6$ Hz, 1H), 1.97 – 1.83 (m, 2H), 1.78 – 1.71 (m, 2H), 1.63 – 1.55 (m, 1H), 1.26 (dt, $J = 19.0, 10.5$ Hz, 5H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.01, 129.49, 129.29, 127.94, 111.64, 54.01, 44.76, 35.70, 33.73, 33.65, 26.11, 25.77.

HRMS m/z (ESI) calcd. for $C_{15}H_{20}NS_2$ ($[M+H]^+$): 278.1037, Found: 278.1048.



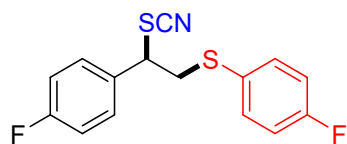
cyclopentyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ay).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ay** as a colorless liquid (27.9 mg, 53% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.38 (ddt, $J = 8.2, 6.1, 3.3$ Hz, 5H), 4.58 (dd, $J = 8.9, 6.6$ Hz, 1H), 3.38 – 3.21 (m, 2H), 3.05 (p, $J = 7.0$ Hz, 1H), 2.07 – 1.91 (m, 2H), 1.72 (tddd, $J = 11.4, 8.7, 5.5, 2.2$ Hz, 2H), 1.61 – 1.54 (m, 2H), 1.54 – 1.43 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.00, 129.51, 129.32, 127.93, 111.63, 53.74, 44.86, 37.48, 33.99, 33.85, 24.83.

HRMS m/z (ESI) calcd. for $C_{14}H_{18}NS_2$ ($[M+H]^+$): 264.0881, Found: 264.0894.



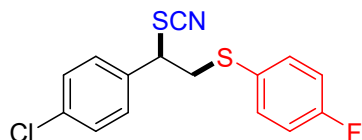
(4-fluorophenyl)(2-(4-fluorophenyl)-2-thiocyanatoethyl)sulfane (4ba).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ba** as a colorless liquid (51.6 mg, 84% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 2H), 7.21 – 7.15 (m, 2H), 7.03 – 6.90 (m, 4H), 4.33 (dd, $J = 9.2, 6.4$ Hz, 1H), 3.58 – 3.37 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 163.96 (d, $^1J_{FC} = 251.3$ Hz), 163.97 (d, $J = 251.6$ Hz), 134.40 (d, $^3J_{FC} = 8.3$ Hz), 132.17 (d, $J = 3.3$ Hz), 129.84 (d, $J = 8.5$ Hz), 128.58 (d, $^4J_{FC} = 3.4$ Hz), 116.72 (d, $J = 22.0$ Hz), 116.43 (d, $^2J_{FC} = 21.9$ Hz), 110.96, 51.83, 41.21.

HRMS m/z (ESI) calcd. for $C_{15}H_{12}F_2NS_2$ ($[M+H]^+$): 308.0379, Found: 308.0391.



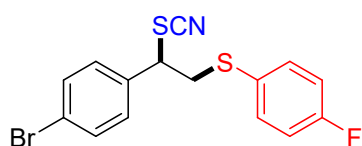
(2-(4-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ca).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ca** as a colorless liquid (53.1 mg, 82% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.34 (dd, $J = 8.4, 5.9$ Hz, 4H), 7.23 – 7.17 (m, 2H), 7.05 – 6.98 (m, 2H), 4.37 (dd, $J = 9.2, 6.4$ Hz, 1H), 3.61 – 3.45 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.82 (d, $^1J_{\text{FC}} = 249.2$ Hz), 135.62, 134.85, 134.45 (d, $^3J_{\text{FC}} = 8.3$ Hz), 129.58, 129.30, 128.47 (d, $^4J_{\text{FC}} = 3.4$ Hz), 116.74 (d, $^2J_{\text{FC}} = 22.0$ Hz), 110.82, 51.78, 41.01.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{12}\text{ClFNS}_2$ ($[\text{M}+\text{H}]^+$): 324.0084, Found: 324.0091.



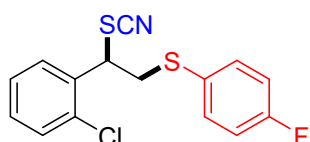
(2-(4-bromophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4da).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4da** as a colorless liquid (54.5 mg, 74% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.53 – 7.47 (m, 2H), 7.38 – 7.30 (m, 2H), 7.18 – 7.11 (m, 2H), 7.07 – 6.98 (m, 2H), 4.35 (dd, $J = 9.2, 6.4$ Hz, 1H), 3.60 – 3.45 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.82 (d, $^1J_{\text{FC}} = 249.1$ Hz), 135.36, 134.46 (d, $^3J_{\text{FC}} = 8.3$ Hz), 132.55, 129.56, 128.44 (d, $^4J_{\text{FC}} = 3.5$ Hz), 123.77, 116.75 (d, $^2J_{\text{FC}} = 22.0$ Hz), 110.80, 51.82, 40.94.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{12}\text{BrFNS}_2$ ($[\text{M}+\text{H}]^+$): 367.9579, Found: 367.9584.



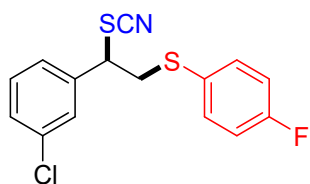
(2-(2-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ea).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ea** as a colorless liquid (48.5 mg, 75% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 – 7.28 (m, 4H), 7.24 (t, $J = 1.7$ Hz, 1H), 7.19 – 7.14 (m, 1H), 7.05 – 6.98 (m, 2H), 4.35 (dd, $J = 9.0, 6.6$ Hz, 1H), 3.59 – 3.47 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.83 (d, $^1J_{\text{FC}} = 249.2$ Hz), 138.34, 135.26, 134.49 (d, $^3J_{\text{FC}} = 8.3$ Hz), 130.59, 129.81, 128.45 (d, $^4J_{\text{FC}} = 3.5$ Hz), 128.15, 126.16, 116.74 (d, $^2J_{\text{FC}} = 22.0$ Hz), 110.70, 51.91, 40.99.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{12}\text{ClFNS}_2$ ($[\text{M}+\text{H}]^+$): 324.0084, Found: 324.0089.



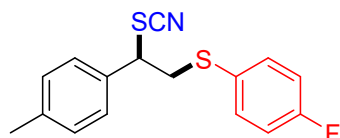
(2-(3-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4fa).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4fa** as a colorless liquid (51.2 mg, 79% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.40 (m, 1H), 7.40 – 7.35 (m, 3H), 7.33 – 7.29 (m, 2H), 7.04 – 6.98 (m, 2H), 4.86 (dd, *J* = 8.7, 7.0 Hz, 1H), 3.67 – 3.53 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 162.82 (d, ¹*J*_{FC} = 249.2 Hz), 138.31, 135.25, 134.50 (d, ³*J*_{FC} = 8.3 Hz), 130.59, 129.81, 128.27 (d, ⁴*J*_{FC} = 3.5 Hz), 126.16, 116.75 (d, ²*J*_{FC} = 22.0 Hz), 110.73, 51.89, 40.97.

HRMS *m/z* (ESI) calcd. for C₁₅H₁₂ClFNS₂ ([M+H]⁺): 324.0084, Found: 324.0097.



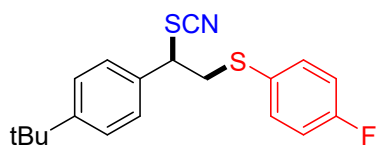
(4-fluorophenyl)(2-thiocyanato-2-(*p*-tolyl)ethyl)sulfane (4ga).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ga** as a colorless liquid (49.7 mg, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.37 – 7.32 (m, 2H), 7.19 – 7.14 (m, 4H), 7.03 – 6.98 (m, 2H), 4.40 (dd, *J* = 9.1, 6.5 Hz, 1H), 3.62 – 3.49 (m, 2H), 2.36 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 162.38 (d, ¹*J*_{FC} = 248.7 Hz), 139.41, 133.97 (d, ³*J*_{FC} = 8.3 Hz), 132.83, 129.71, 128.48 (d, ⁴*J*_{FC} = 3.5 Hz), 127.52, 116.29 (d, ²*J*_{FC} = 22.0 Hz), 111.09, 52.11, 40.84, 21.05.

HRMS *m/z* (ESI) calcd. for C₁₆H₁₅FNS₂ ([M+H]⁺): 304.0630, Found: 304.0654.



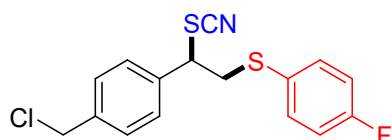
(2-(4-(tert-butyl)phenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ha).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ha** as a colorless liquid (58.0 mg, 84% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.37 – 7.30 (m, 4H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.01 – 6.94 (m, 2H), 4.43 (dd, *J* = 8.8, 6.7 Hz, 1H), 3.63 – 3.52 (m, 2H), 1.31 (s, 9H).

¹³C NMR (100 MHz, CDCl₃) δ 162.61 (d, ¹*J*_{FC} = 248.5 Hz), 152.76, 134.32 (d, ³*J*_{FC} = 8.2 Hz), 132.99, 128.91 (d, ⁴*J*_{FC} = 3.4 Hz), 127.62, 126.23, 116.53 (d, ²*J*_{FC} = 22.0 Hz), 111.49, 52.57, 41.21, 34.85, 31.34.

HRMS *m/z* (ESI) calcd. for C₁₉H₂₁FNS₂ ([M+H]⁺): 346.1099, Found: 346.1104.

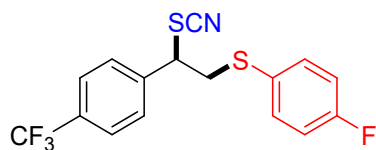


(2-(4-(chloromethyl)phenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ia). Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ia** as a colorless liquid (55.4 mg, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, *J* = 8.2 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 7.04 – 6.97 (m, 2H), 4.58 (s, 2H), 4.42 (dd, *J* = 9.0, 6.6 Hz, 1H), 3.64 – 3.48 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 163.02 (d, $^1J_{\text{FC}} = 248.9$ Hz), 139.21, 136.78, 134.71 (d, $^3J_{\text{FC}} = 8.3$ Hz), 129.75, 128.89 (d, $^4J_{\text{FC}} = 3.4$ Hz), 128.64, 116.98 (d, $^2J_{\text{FC}} = 22.0$ Hz), 111.30, 52.44, 45.80, 41.34.

HRMS m/z (ESI) calcd. for $\text{C}_{16}\text{H}_{14}\text{ClFNS}_2$ ($[\text{M}+\text{H}]^+$): 338.0248, Found: 338.0251.



(4-fluorophenyl)(2-thiocyanato-2-(4-(trifluoromethyl)phenyl)ethyl)sulfane (4ja).

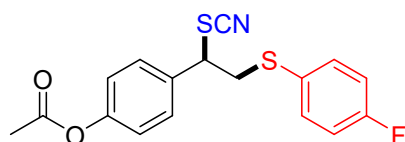
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ja** as a colorless liquid (47.9 mg, 67% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.63 (d, $J = 8.2$ Hz, 2H), 7.40 (d, $J = 8.1$ Hz, 2H), 7.33 (dd, $J = 8.8, 5.2$ Hz, 2H), 7.00 (t, $J = 8.6$ Hz, 2H), 4.43 (dd, $J = 9.0, 6.5$ Hz, 1H), 3.63 – 3.49 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 162.64 (d, $^1J_{\text{FC}} = 249.4$ Hz), 140.15, 134.34 (d, $^3J_{\text{FC}} = 8.3$ Hz), 131.48 (q, $J = 32.9$ Hz), 128.22, 128.07 (d, $^4J_{\text{FC}} = 3.4$ Hz), 126.11 (q, $J = 3.8$ Hz), 124.44, 122.64, 120.84, 116.57 (d, $^2J_{\text{FC}} = 22.1$ Hz), 110.31, 51.48, 40.66.

^{19}F NMR (375 MHz, CDCl_3) δ -62.86, -112.46.

HRMS m/z (ESI) calcd. for $\text{C}_{16}\text{H}_{12}\text{F}_4\text{NS}_2$ ($[\text{M}+\text{H}]^+$): 358.0374, Found: 358.0376.



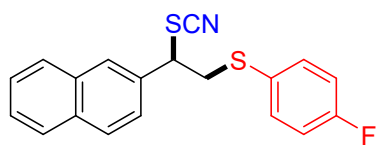
(2-((4-fluorophenyl)thio)-1-thiocyanatoethyl)phenyl acetate (4ka).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ka** as a colorless liquid (51.4 mg, 74% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.32 (dd, $J = 8.5, 5.2$ Hz, 2H), 7.28 (s, 2H), 7.13 – 7.07 (m, 2H), 7.00 (t, $J = 8.5$ Hz, 2H), 4.41 (dd, $J = 8.9, 6.5$ Hz, 1H), 3.63 – 3.46 (m, 2H), 2.29 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 169.03, 162.78 (d, $^1J_{\text{FC}} = 248.9$ Hz), 151.51, 134.39 (d, $^3J_{\text{FC}} = 8.3$ Hz), 133.77, 129.08, 128.76 (d, $^4J_{\text{FC}} = 3.5$ Hz), 122.48, 116.67 (d, $^2J_{\text{FC}} = 22.0$ Hz), 110.93, 52.08, 41.28, 21.18.

HRMS m/z (ESI) calcd. for $\text{C}_{17}\text{H}_{15}\text{FNO}_2\text{S}_2$ ($[\text{M}+\text{H}]^+$): 348.0548, Found: 348.0554.



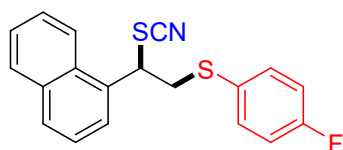
(4-fluorophenyl)(2-(naphthalen-2-yl)-2-thiocyanatoethyl)sulfane (4la).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4la** as a colorless liquid (44.1 mg, 65% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.84 (dd, $J = 7.9, 3.1$ Hz, 3H), 7.71 (s, 1H), 7.56 – 7.48 (m, 2H), 7.47 – 7.40 (m, 2H), 7.33 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.05 – 6.98 (m, 2H), 4.96 (dd, $J = 7.8, 5.9$ Hz, 1H), 3.41 – 3.31 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 162.69 (d, $^1J_{\text{FC}} = 248.8$ Hz), 134.37 (d, $^3J_{\text{FC}} = 8.3$ Hz), 133.67, 133.39, 133.16, 129.49, 128.73 (d, $^4J_{\text{FC}} = 3.3$ Hz), 128.25, 127.93, 127.82, 127.23, 127.03, 124.60, 116.59 (d, $^2J_{\text{FC}} = 22.0$ Hz), 111.21, 52.94, 41.07.

HRMS m/z (ESI) calcd. for $C_{19}H_{15}FNS_2$ ($[M+H]^+$): 340.0630, Found: 340.0641.



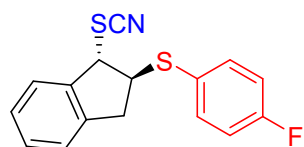
(4-fluorophenyl)(2-(naphthalen-1-yl)-2-thiocyanatoethyl)sulfane (4ma).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ma** as a colorless liquid (42.1 mg, 62% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.85 (m, 2H), 7.82 (d, $J = 8.3$ Hz, 1H), 7.60 – 7.51 (m, 3H), 7.47 (t, $J = 7.7$ Hz, 1H), 7.29 (dd, $J = 8.5, 5.3$ Hz, 2H), 6.96 (t, $J = 8.6$ Hz, 2H), 5.26 (t, $J = 7.6$ Hz, 1H), 3.82 (dd, $J = 7.7, 2.2$ Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 163.84, 161.37, 134.29, 134.21, 134.08, 131.31, 130.79, 130.19, 129.43, 128.82, 127.13, 126.30, 125.24, 121.91, 116.51, 116.29, 111.09, 77.31, 77.00, 76.68, 47.87, 41.17.

HRMS m/z (ESI) calcd. for $C_{19}H_{15}FNS_2$ ($[M+H]^+$): 340.0630, Found: 340.0635.



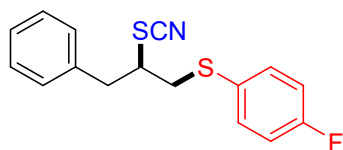
(4-fluorophenyl)(1-thiocyanato-2,3-dihydro-1H-inden-2-yl)sulfane (4na).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4na** as a colorless liquid (37.4 mg, 62% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.45 (m, 2H), 7.40 – 7.27 (m, 5H), 7.07 (t, $J = 8.6$ Hz, 2H), 4.71 (d, $J = 2.2$ Hz, 1H), 4.17 (dt, $J = 7.1, 2.5$ Hz, 1H), 3.67 (dd, $J = 17.1, 7.1$ Hz, 1H), 3.01 (dd, $J = 17.1, 2.5$ Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.86 (d, $^1J_{FC} = 249.1$ Hz), 141.92, 137.01, 135.02 (d, $^3J_{FC} = 8.3$ Hz), 130.17, 128.20 (d, $^4J_{FC} = 3.4$ Hz), 128.10, 126.00, 125.54, 116.61 (d, $^2J_{FC} = 22.0$ Hz), 111.20, 58.15, 54.13, 37.35.

HRMS m/z (ESI) calcd. for $C_{16}H_{13}FNS_2$ ($[M+H]^+$): 302.0473, Found: 302.0493.



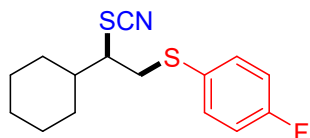
(4-fluorophenyl)(3-phenyl-2-thiocyanatopropyl)sulfane (4oa).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4oa** as a colorless liquid (39.4 mg, 65% yield).

1H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.39 (m, 2H), 7.38 – 7.31 (m, 2H), 7.31 – 7.26 (m, 1H), 7.25 – 7.20 (m, 2H), 7.07 – 7.00 (m, 2H), 3.50 (qd, $J = 7.0, 5.7$ Hz, 1H), 3.19 – 2.96 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 163.46 (d, $^1J_{FC} = 249.6$ Hz), 137.48, 136.37 (d, $^3J_{FC} = 8.4$ Hz), 129.64, 129.26, 127.70, 117.03 (d, $^2J_{FC} = 21.9$ Hz), 112.47, 51.23, 39.55, 38.92.

HRMS m/z (ESI) calcd. for $C_{16}H_{15}FNS_2$ ($[M+H]^+$): 304.0630, Found: 304.0637.



(2-cyclohexyl-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4pa).

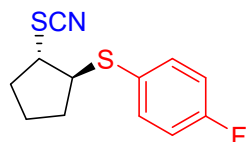
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4pa** as a colorless liquid (34.3 mg, 58% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48 – 7.42 (m, 2H), 7.07 – 7.00 (m, 2H), 3.29 – 3.17 (m, 2H), 3.13 – 3.06 (m, 1H), 1.80 (dtd, $J = 16.5, 8.9, 7.5, 4.3$ Hz, 5H), 1.48 – 1.07 (m, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.85 (d, $^1J_{\text{FC}} = 248.9$ Hz), 135.42 (d, $^3J_{\text{FC}} = 8.3$ Hz), 128.98 (d, $^4J_{\text{FC}} = 3.5$ Hz), 116.65 (d, $^2J_{\text{FC}} = 21.9$ Hz), 112.41, 56.91, 39.92, 37.67, 31.11, 28.99, 26.30, 26.22.

$^{19}\text{F NMR}$ (375 MHz, CDCl_3) δ -112.84.

HRMS m/z (ESI) calcd. for $\text{C}_{15}\text{H}_{19}\text{FNS}_2$ ($[\text{M}+\text{H}]^+$): 296.0943, Found: 296.0956.



(4-fluorophenyl)(2-thiocyanatocyclopentyl)sulfane (4qa).

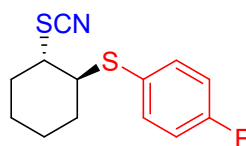
Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4qa** as a colorless liquid (27.9 mg, 55% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 – 7.42 (m, 2H), 7.08 – 7.00 (m, 2H), 3.56 (dt, $J = 7.4, 4.9$ Hz, 1H), 3.47 (dt, $J = 7.0, 4.6$ Hz, 1H), 2.45 – 2.36 (m, 1H), 2.30 (dq, $J = 14.6, 7.4$ Hz, 1H), 1.86 (pt, $J = 6.9, 3.0$ Hz, 3H), 1.81 – 1.70 (m, 1H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.90 (d, $^1J_{\text{FC}} = 248.8$ Hz), 135.24 (d, $^3J_{\text{FC}} = 8.3$ Hz), 128.59 (d, $^4J_{\text{FC}} = 3.4$ Hz), 116.57 (d, $^2J_{\text{FC}} = 21.9$ Hz), 111.46, 54.38, 53.08, 32.18, 31.53, 23.09.

$^{19}\text{F NMR}$ (375 MHz, CDCl_3) δ -112.81.

HRMS m/z (ESI) calcd. for $\text{C}_{12}\text{H}_{13}\text{FNS}_2$ ($[\text{M}+\text{H}]^+$): 254.0473, Found: 254.0479.



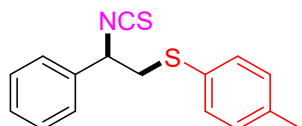
(4-fluorophenyl)(2-thiocyanatocyclohexyl)sulfane (4ra).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **4ra** as a colorless liquid (33.1 mg, 62% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.54 – 7.46 (m, 2H), 7.08 – 7.00 (m, 2H), 3.15 (td, $J = 10.2, 3.9$ Hz, 1H), 2.91 (td, $J = 10.2, 3.9$ Hz, 1H), 2.49 – 2.38 (m, 1H), 2.22 – 2.10 (m, 1H), 1.76 (ddt, $J = 10.7, 7.4, 2.4$ Hz, 3H), 1.49 – 1.27 (m, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 163.28 (d, $^1J_{\text{FC}} = 249.5$ Hz), 136.98 (d, $^3J_{\text{FC}} = 8.4$ Hz), 126.81 (d, $^4J_{\text{FC}} = 3.2$ Hz), 116.47 (d, $^2J_{\text{FC}} = 21.8$ Hz), 111.52, 52.26, 52.14, 33.90, 33.69, 25.26.

HRMS m/z (ESI) calcd. for $\text{C}_{13}\text{H}_{15}\text{FNS}_2$ ($[\text{M}+\text{H}]^+$): 268.0630, Found: 268.0643.

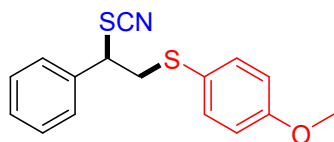


(4-fluorophenyl)(2-isothiocyanato-2-phenylpropyl)sulfane (5)¹.

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **5** as a colorless liquid (54.7 mg, 96% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.32 (m, 5H), 7.30 – 7.25 (m, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 4.80 (dd, *J* = 7.9, 5.9 Hz, 1H), 3.34 – 3.22 (m, 2H), 2.36 (s, 3H).

HRMS *m/z* (ESI) calcd. for C₁₆H₁₆NS₂ ([M+H]⁺): 286.0724, Found: 286.0734.



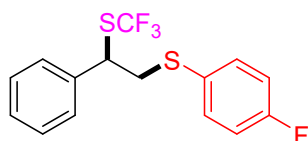
(4-methoxyphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (6).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **6** as a colorless liquid (45.8 mg, 76% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.32 (m, *J* = 16.3, 6.3, 5.1, 3.2 Hz, 7H), 7.01 – 6.93 (m, 2H), 4.25 (dd, *J* = 8.1, 5.0 Hz, 1H), 3.30 – 3.25 (m, 1H), 3.24 (d, *J* = 2.2 Hz, 3H), 3.07 (dd, *J* = 13.3, 5.0 Hz, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 159.90, 136.54, 134.68, 129.37, 129.18, 127.90, 123.65, 115.02, 111.34, 55.42, 52.50, 41.45.

HRMS *m/z* (ESI) calcd. for C₁₆H₁₆NOS₂ ([M+H]⁺): 302.0673, Found: 302.0678.



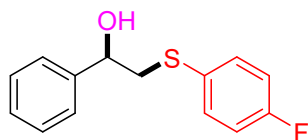
(4-fluorophenyl)(2-phenyl-2-((trifluoromethyl)thio)ethyl)sulfane (7).

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **7** as a colorless liquid (47.8 mg, 72% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.26 (m, 3H), 7.22 (ddd, *J* = 11.3, 5.7, 2.9 Hz, 2H), 7.12 – 7.07 (m, 1H), 7.06 – 7.00 (m, 1H), 6.95 (t, *J* = 8.6 Hz, 2H), 3.59 – 3.42 (m, 2H), 3.28 – 3.19 (m, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 162.95 (d, ⁴*J*_{C-F} = 2.1 Hz), 161.31 (d, ⁴*J*_{C-F} = 2.1 Hz), 138.40 (d, ³*J*_{C-F} = 13.1 Hz), 133.36 (q, ⁴*J*_{C-F} = 8.1, 4.8 Hz), 130.29 (q, ¹*J*_{C-F} = 210.3 Hz), 128.76 (d, ⁴*J*_{C-F} = 4.9 Hz), 128.34 (d, ³*J*_{C-F} = 14.6 Hz), 128.31 (d, ³*J*_{C-F} = 11.9 Hz), 116.17 (dd, ²*J*_{C-F} = 21.7 Hz), 54.18 (d, ⁴*J*_{C-F} = 15.7 Hz), 39.97 (d, ³*J*_{C-F} = 21.6 Hz).

HRMS *m/z* (ESI) calcd. for C₁₅H₁₃F₄S₂ ([M+H]⁺): 333.0395, Found: 333.0402.



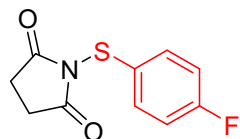
2-((4-fluorophenyl)thio)-1-phenylethan-1-ol (C)².

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **C** as a colorless liquid (20.9 mg, 42% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.39 (m, 2H), 7.36 – 7.27 (m, 5H), 7.08 – 6.97 (m, 2H), 4.69 (dt, *J* = 9.3, 3.2 Hz, 1H), 3.25 (dd, *J* = 13.8, 3.6 Hz, 1H), 3.07 (dd, *J* = 13.8, 9.3 Hz, 1H),

2.81 (d, $J = 2.5$ Hz, 1H), 1.57 (s, 1H).

HRMS m/z (ESI) calcd. for $C_{14}H_{14}FOS$ ($[M+H]^+$): 249.0749, Found: 249.0754.

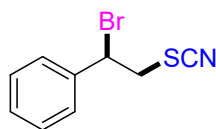


1-((4-fluorophenyl)thio)pyrrolidine-2,5-dione (F)³.

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **F** as a colorless liquid (24.3 mg, 54% yield).

¹H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.67 (m, 2H), 7.05 – 6.96 (m, 2H), 2.78 (s, 4H).

HRMS m/z (ESI) calcd. for $C_{10}H_9FNO_2S$ ($[M+H]^+$): 226.0338, Found: 226.0354.



(1-bromo-2-thiocyanatoethyl)benzene (I)⁴.

Purified by column chromatography on silica gel (20:1 petroleum ether/ethyl acetate) afforded **I** as a colorless liquid (19.8 mg, 41% yield).

¹H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.33 (m, 5H), 5.15 (dd, $J = 10.6, 5.5$ Hz, 1H), 4.17 – 3.96 (m, 2H).

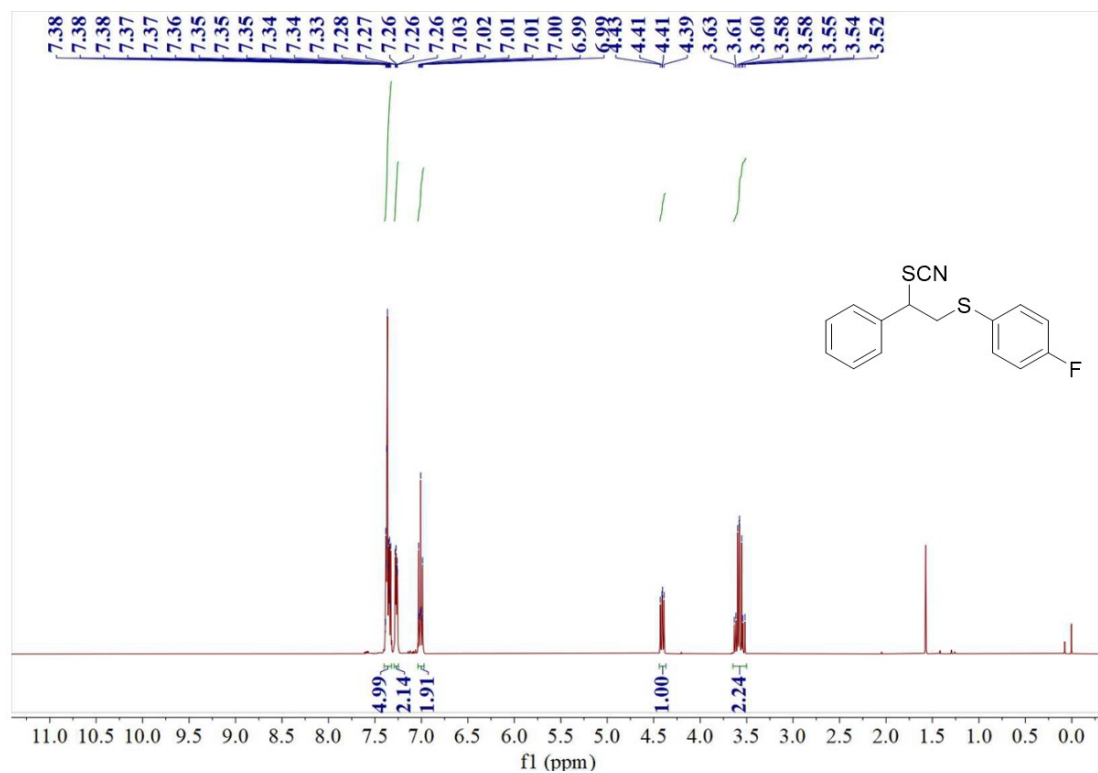
HRMS m/z (ESI) calcd. for C_9H_9BrNS ($[M+H]^+$): 241.9639, Found: 241.9645.

References

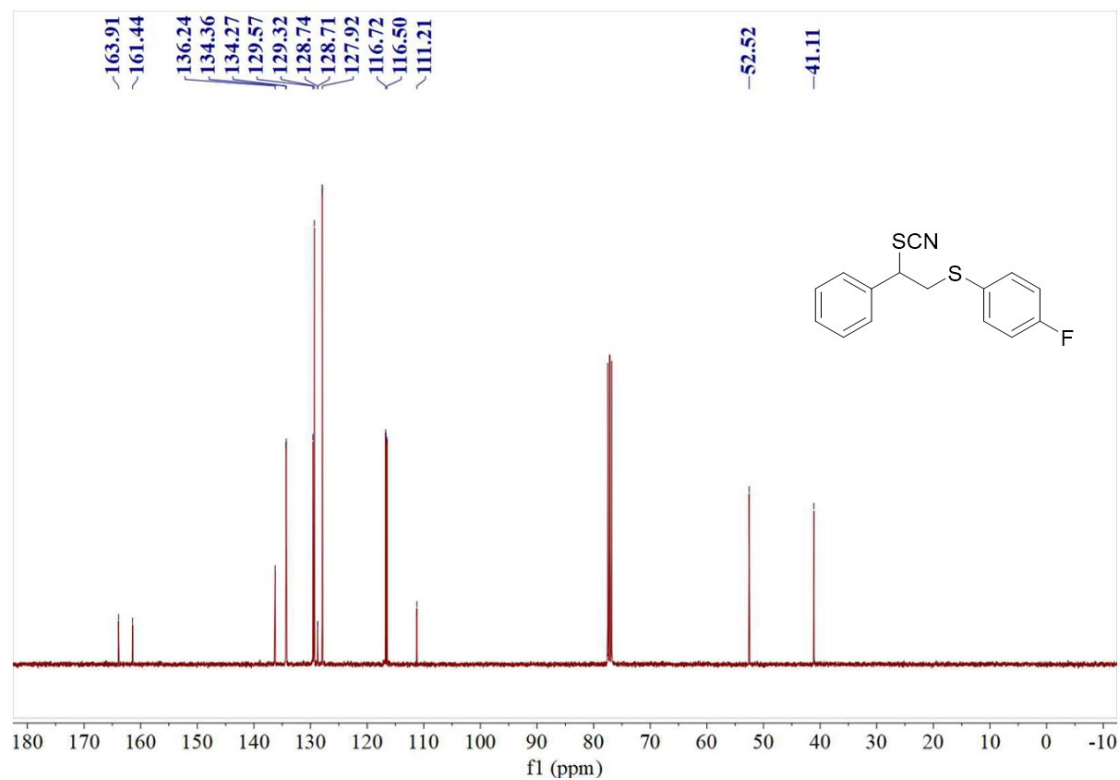
1. Chao Xu, Ze He, Xiaokang Kang and Qingle Zeng, *Green Chemistry*, **2021**, 23, 7544–7548.
2. Jiawei Huang, Xiaoman Li, Liang Xu, and Yu Wei, *The Journal of Organic Chemistry*, **2023**, 88, 3054–3067.
3. Hua Tian, Jipan Yu, Haijun Yang, Changjin Zhu, and Hua Fua, *Advanced Synthesis & Catalysis* **2016**, 358, 1794–1800.
4. Cambie, Richard C, Larsen, David S, Rutledge, Peter S, Woodgate, Paul D, *Organic and Bio-Organic Chemistry*, **1981**, 1, 58-63.

5. NMR Spectra of Products

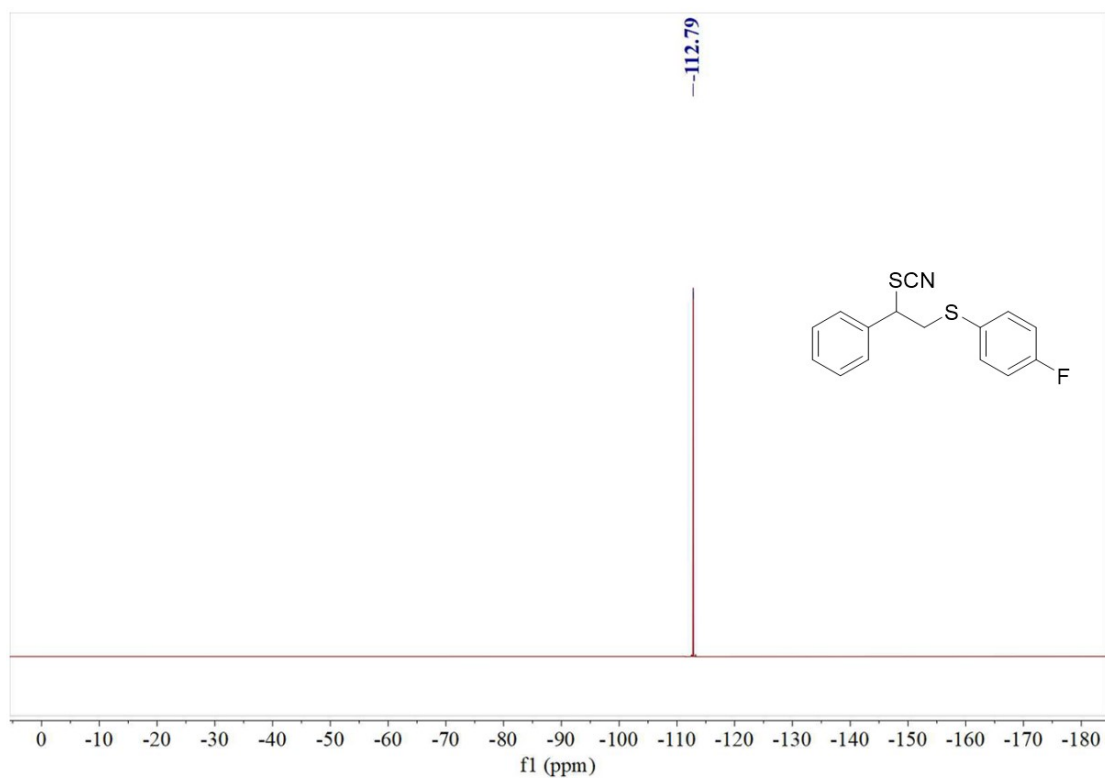
(4-fluorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4aa)



¹H NMR (400 MHz, CDCl₃) spectrum of 4aa

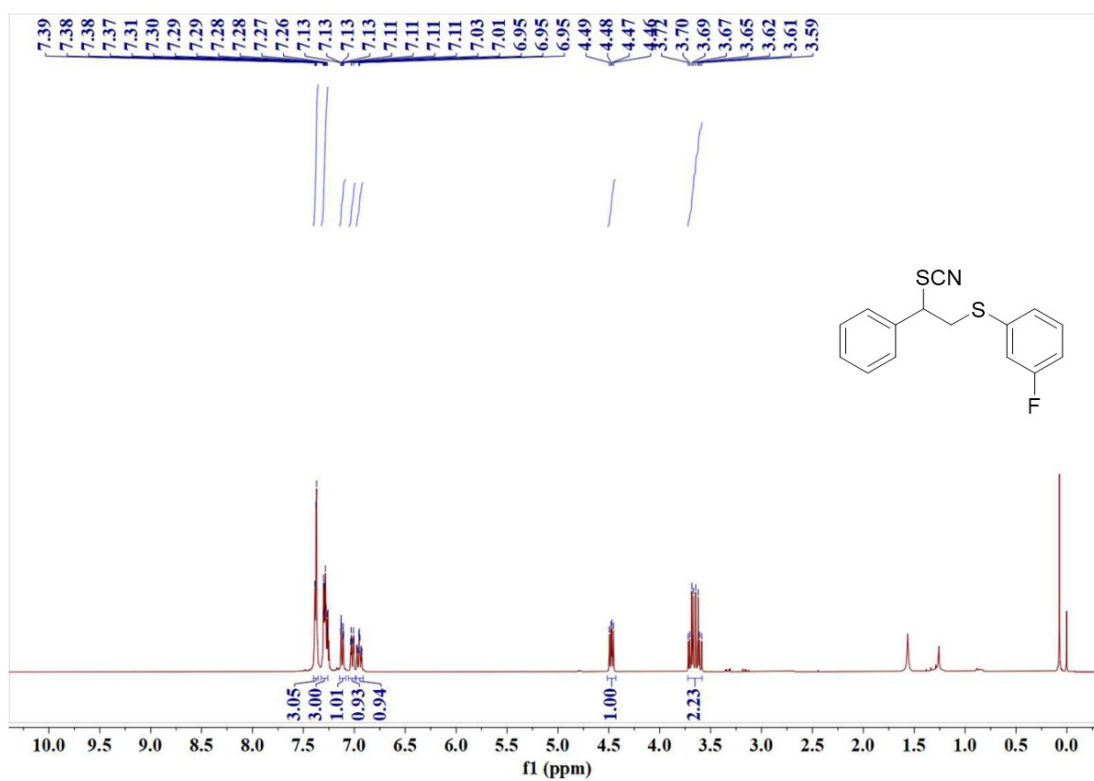


¹³C NMR (100 MHz, CDCl₃) spectrum of 4aa

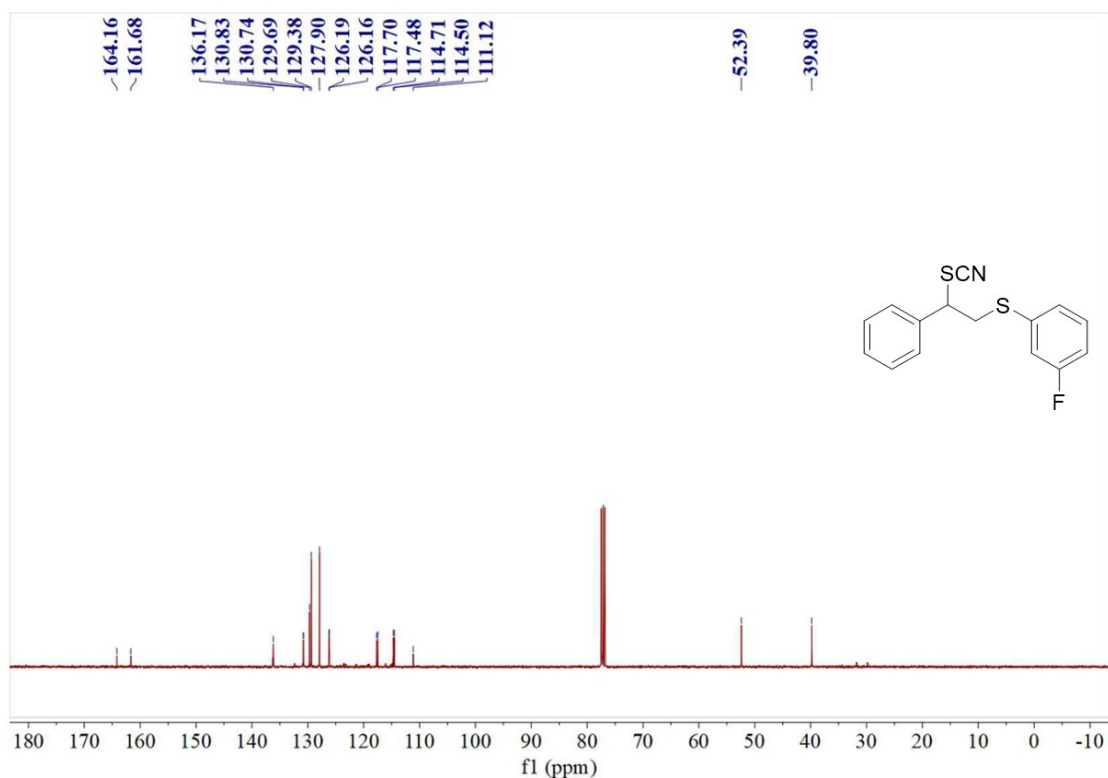


¹⁹F NMR (375 MHz, CDCl₃) spectrum of 4aa

(3-fluorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane(4ab)

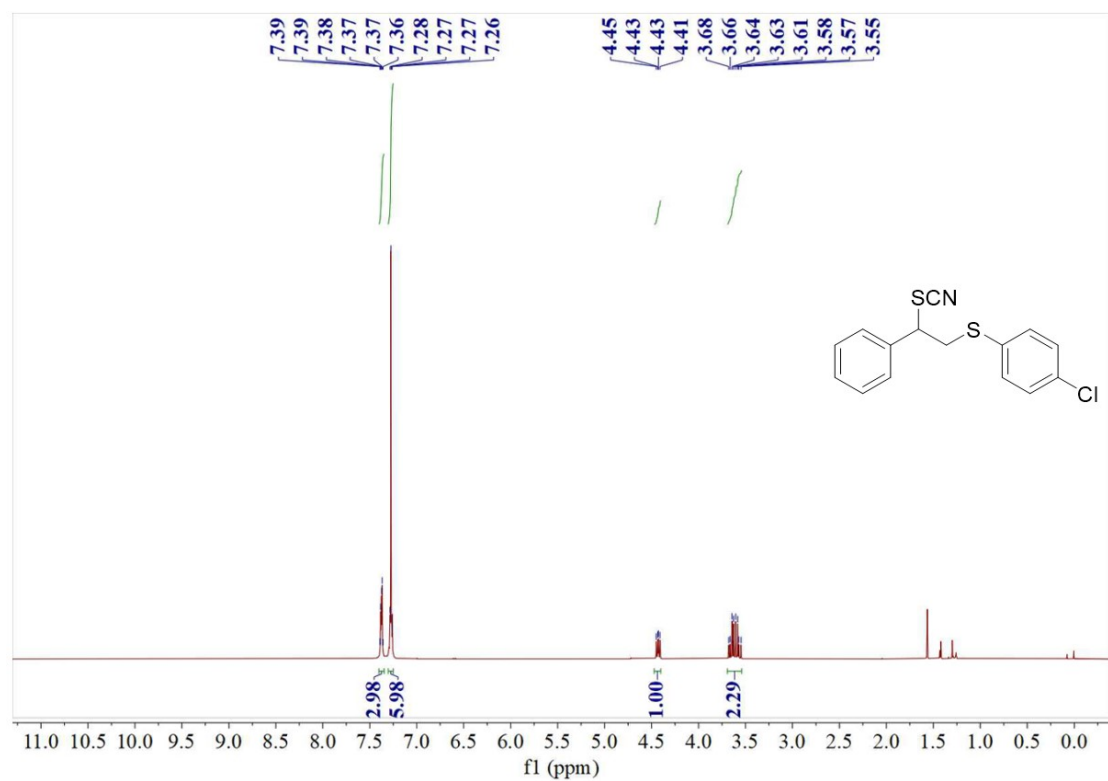


¹H NMR (400 MHz, CDCl₃) spectrum of 4ab

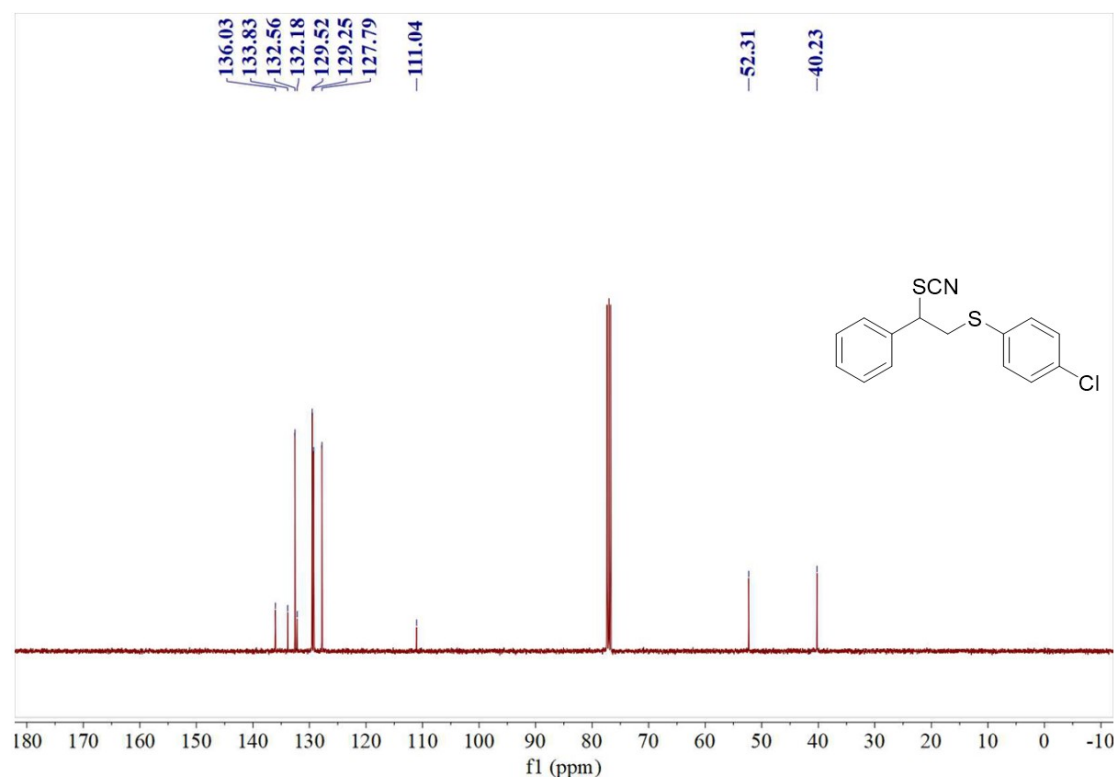


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ab

(4-fluorophenyl)(2-(4-fluorophenyl)-2-thiocyanatoethyl)sulfane (4ac)

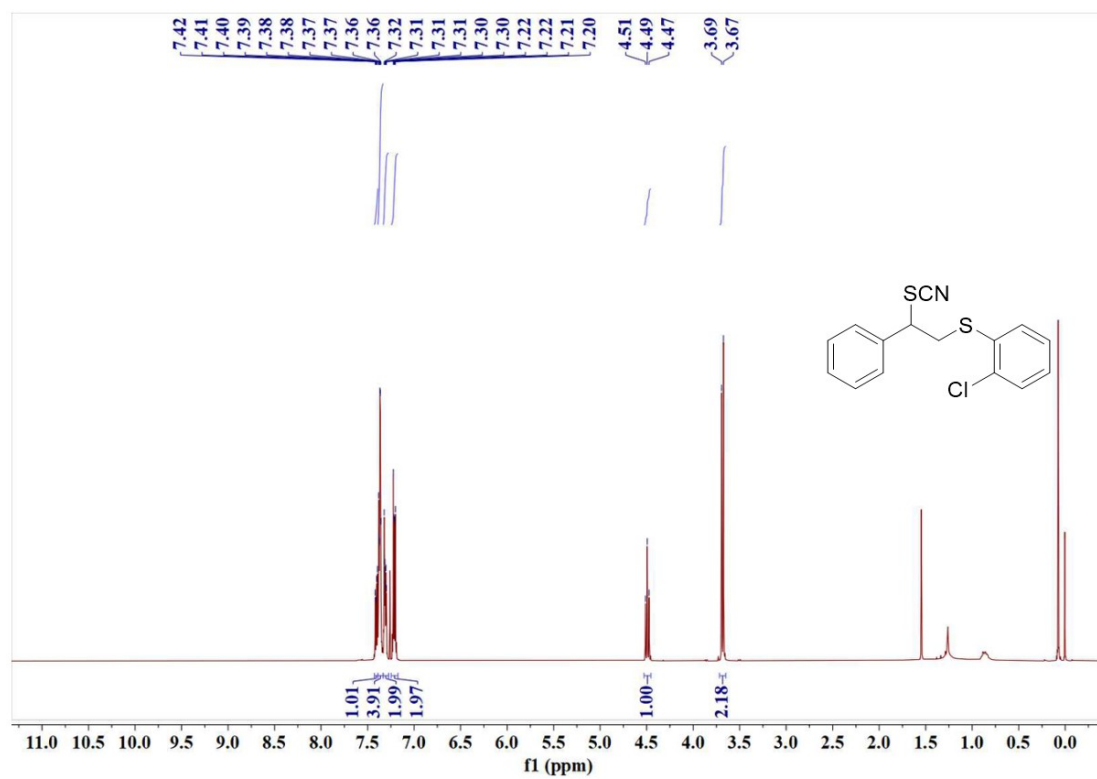


¹H NMR (400 MHz, CDCl₃) spectrum of 4ac

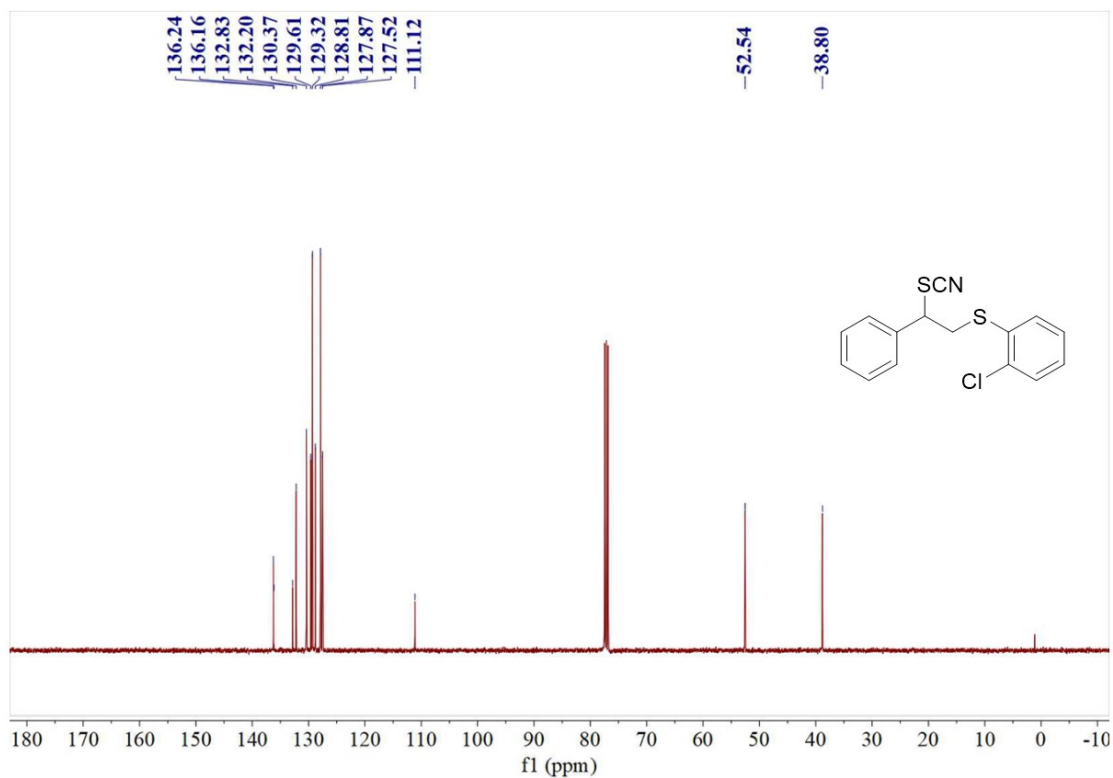


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ac

(2-chlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ad)

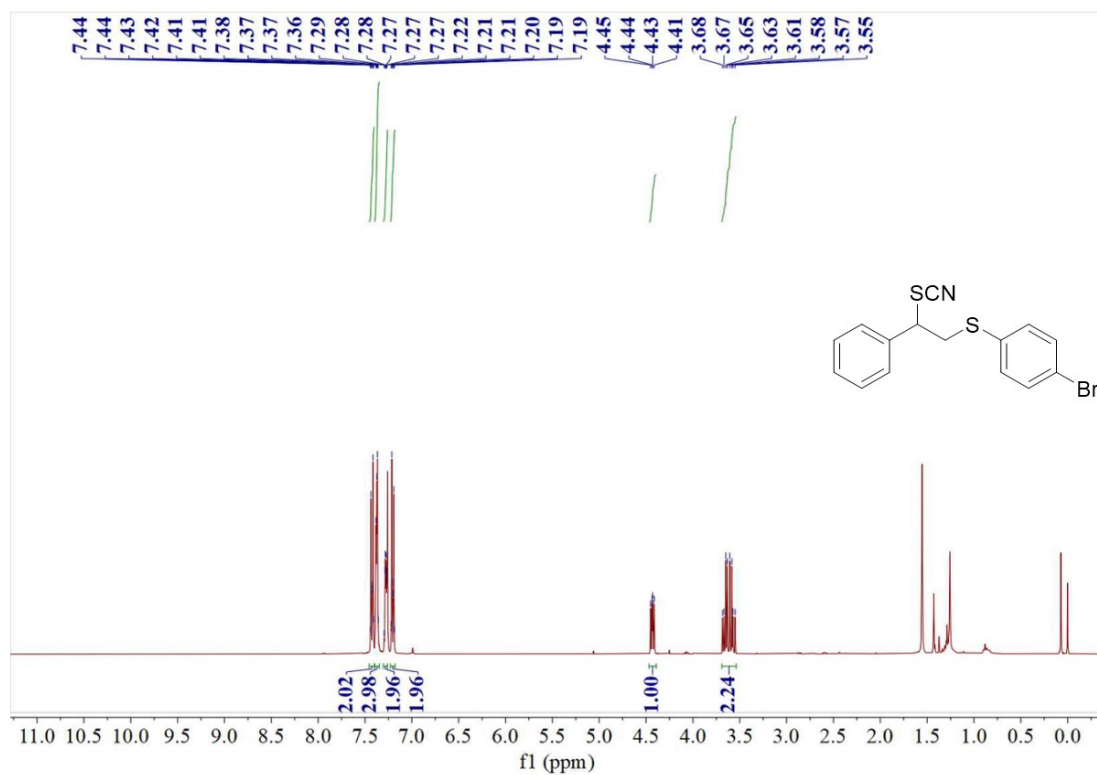


¹H NMR (400 MHz, CDCl₃) spectrum of 4ad

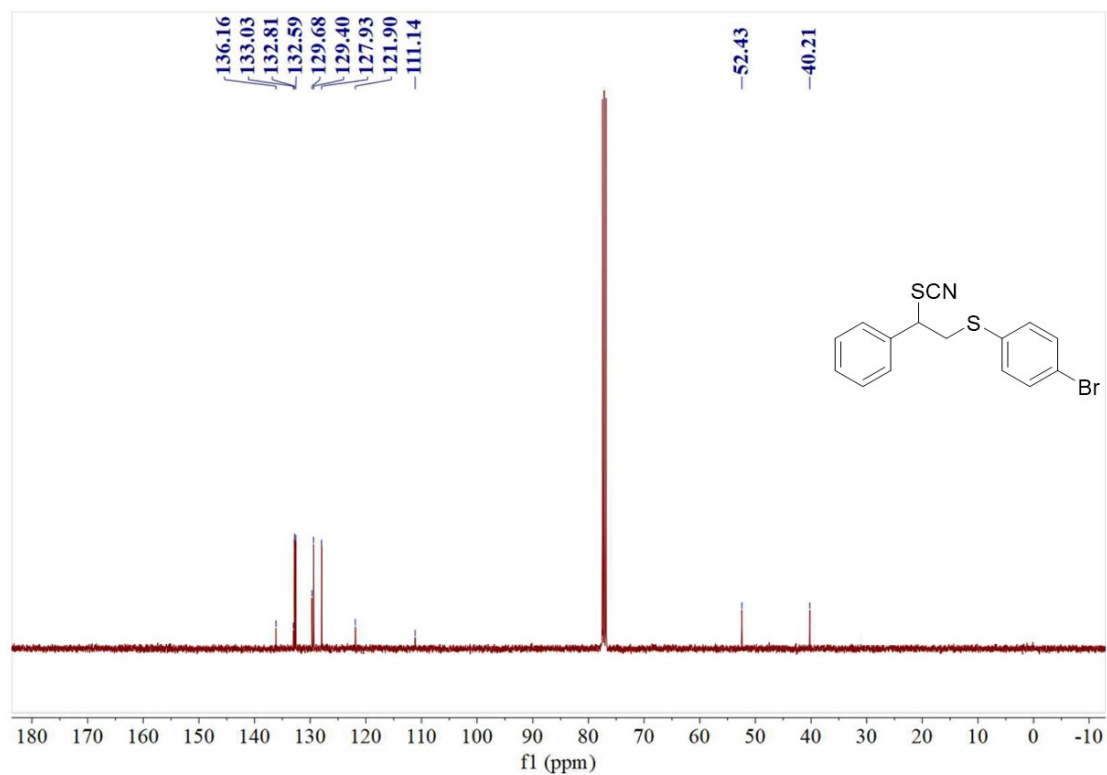


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ad

(4-bromophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ae)

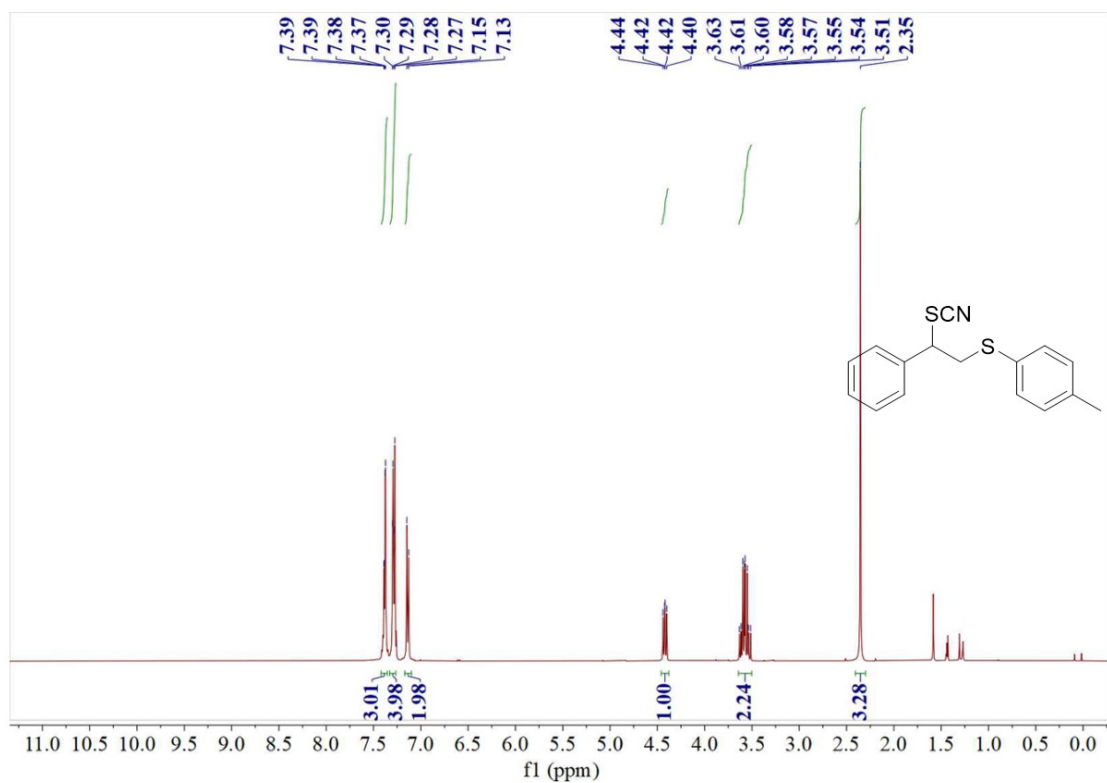


¹H NMR (400 MHz, CDCl₃) spectrum of 4ae

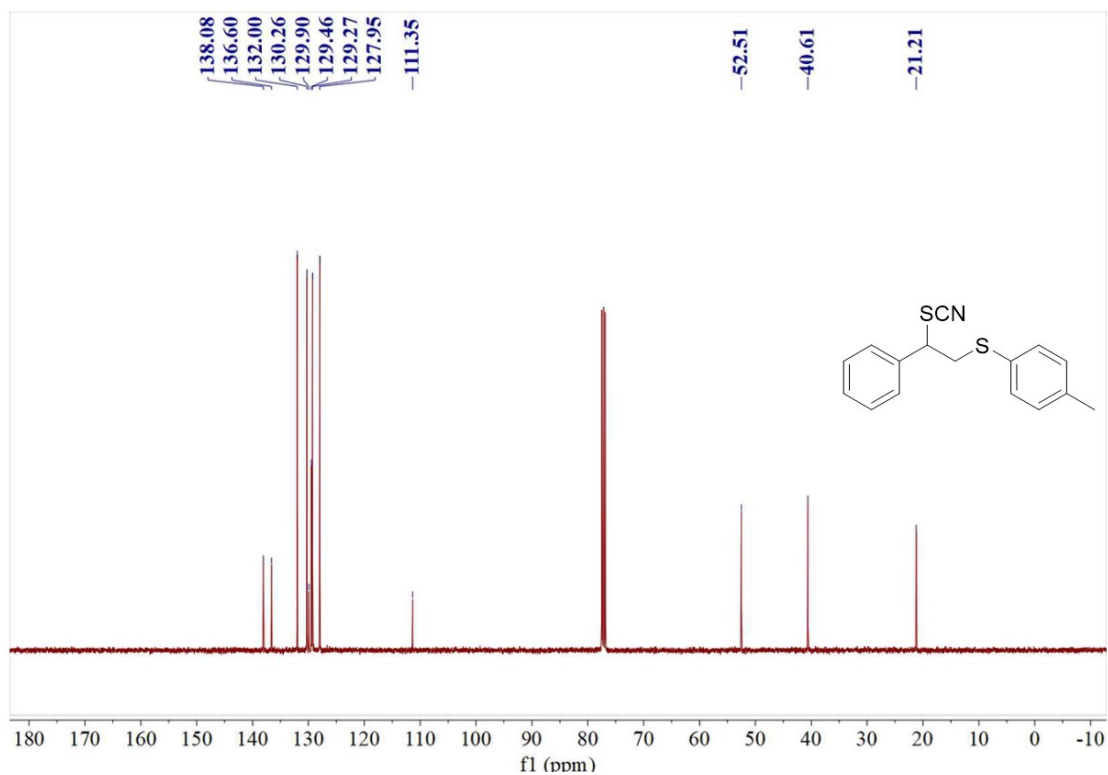


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ae

(2-phenyl-2-thiocyanatoethyl)(p-tolyl)sulfane (4af)

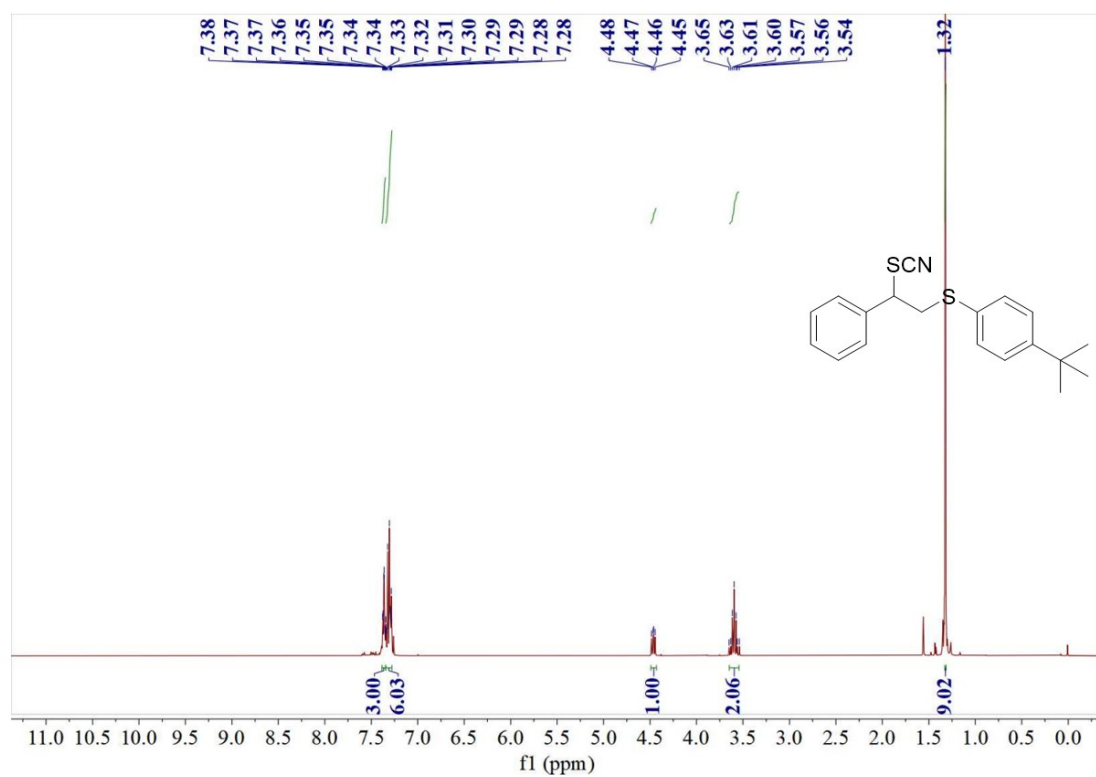


¹H NMR (400 MHz, CDCl₃) spectrum of 4af

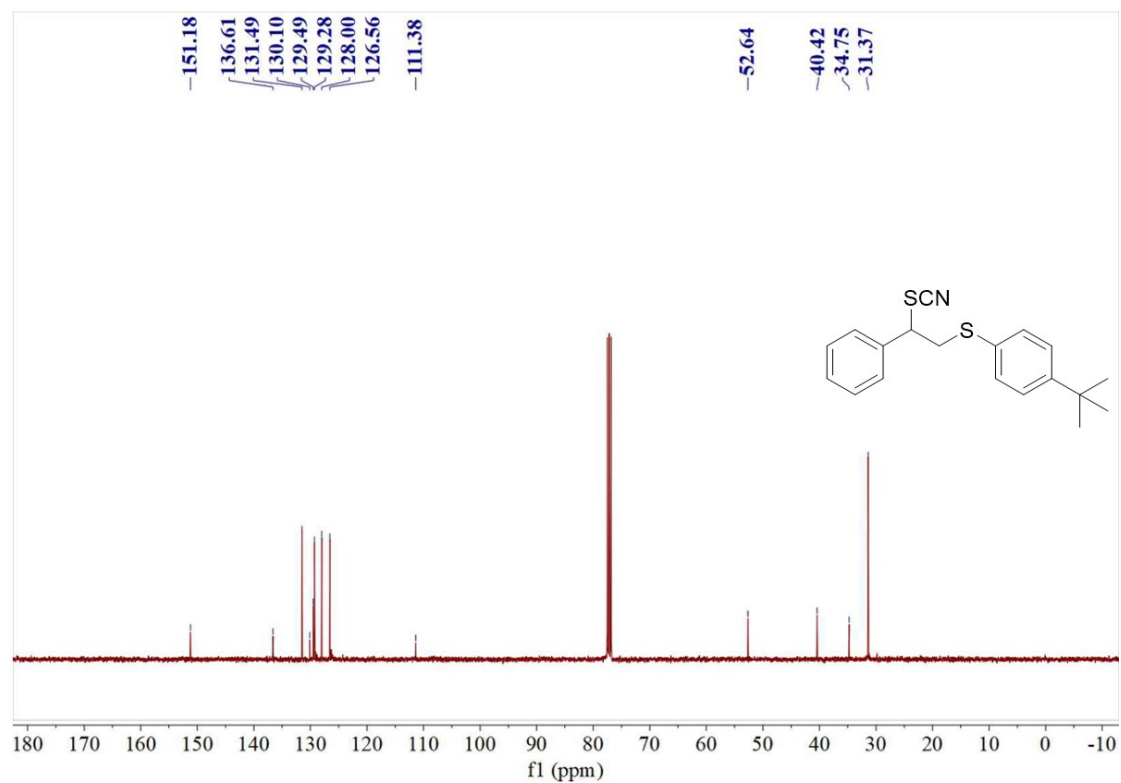


¹³C NMR (100 MHz, CDCl₃) spectrum of 4af

(2-(tert-butyl)phenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ag)

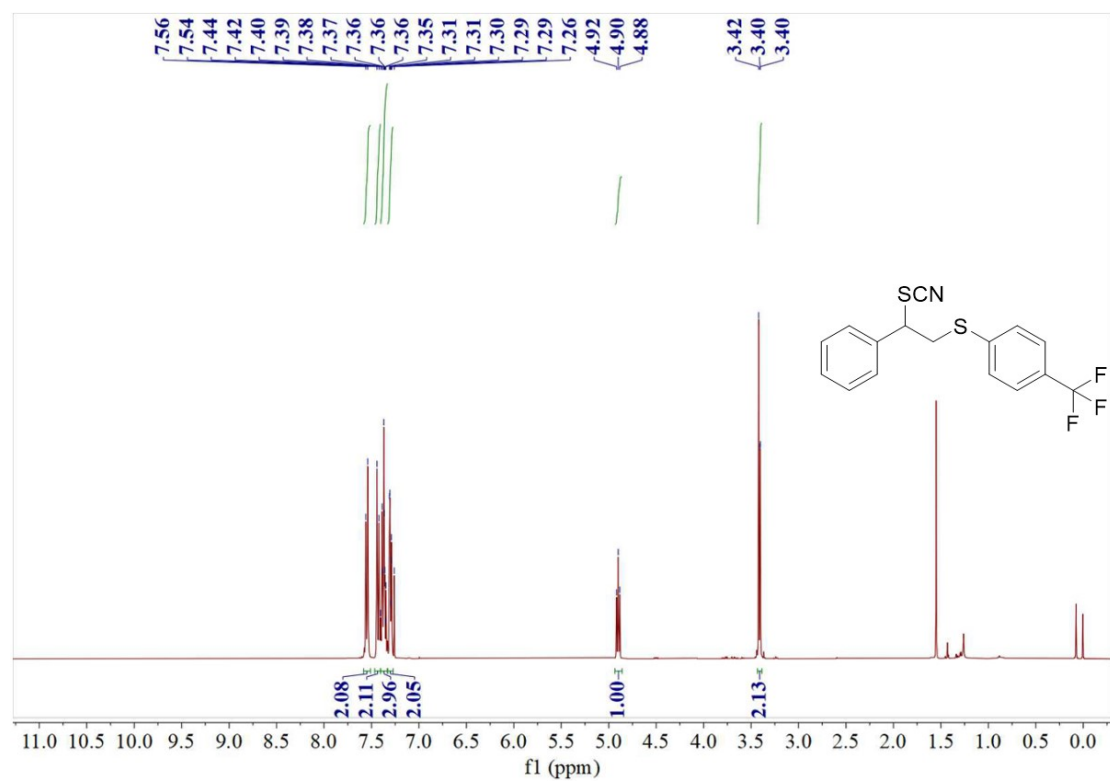


¹H NMR (400 MHz, CDCl₃) spectrum of 4ag

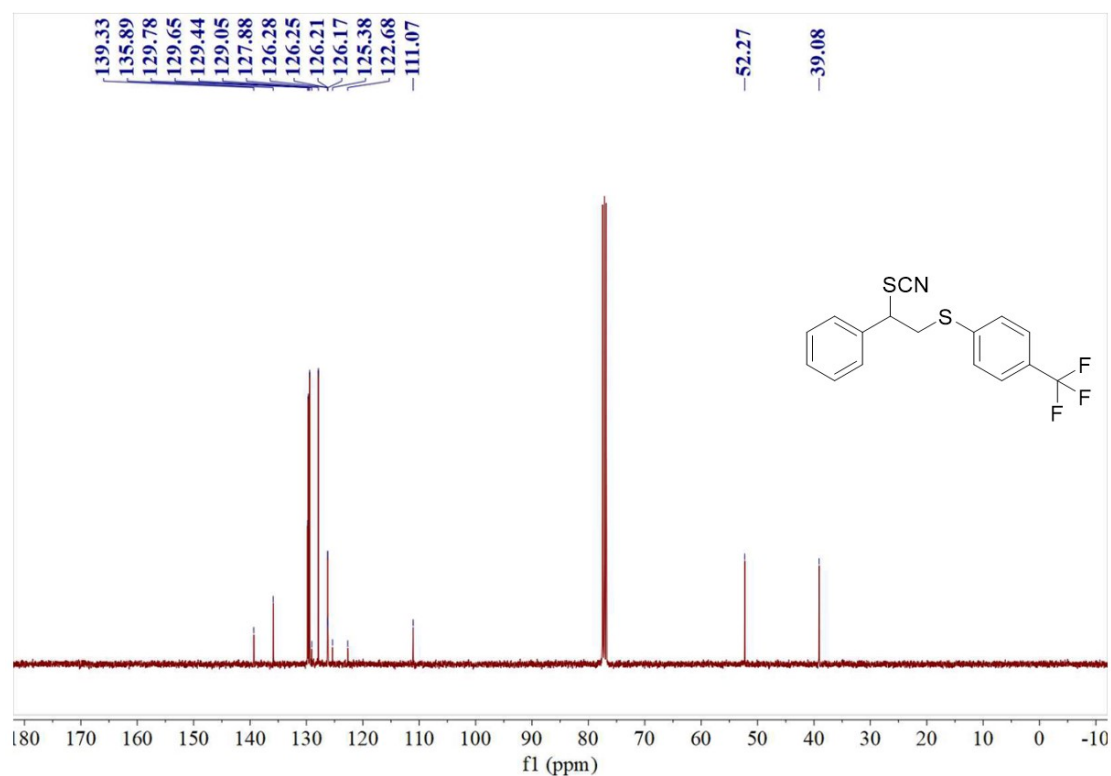


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ag

(2-phenyl-2-thiocyanatoethyl)(4-(trifluoromethyl)phenyl)sulfane (4ah)

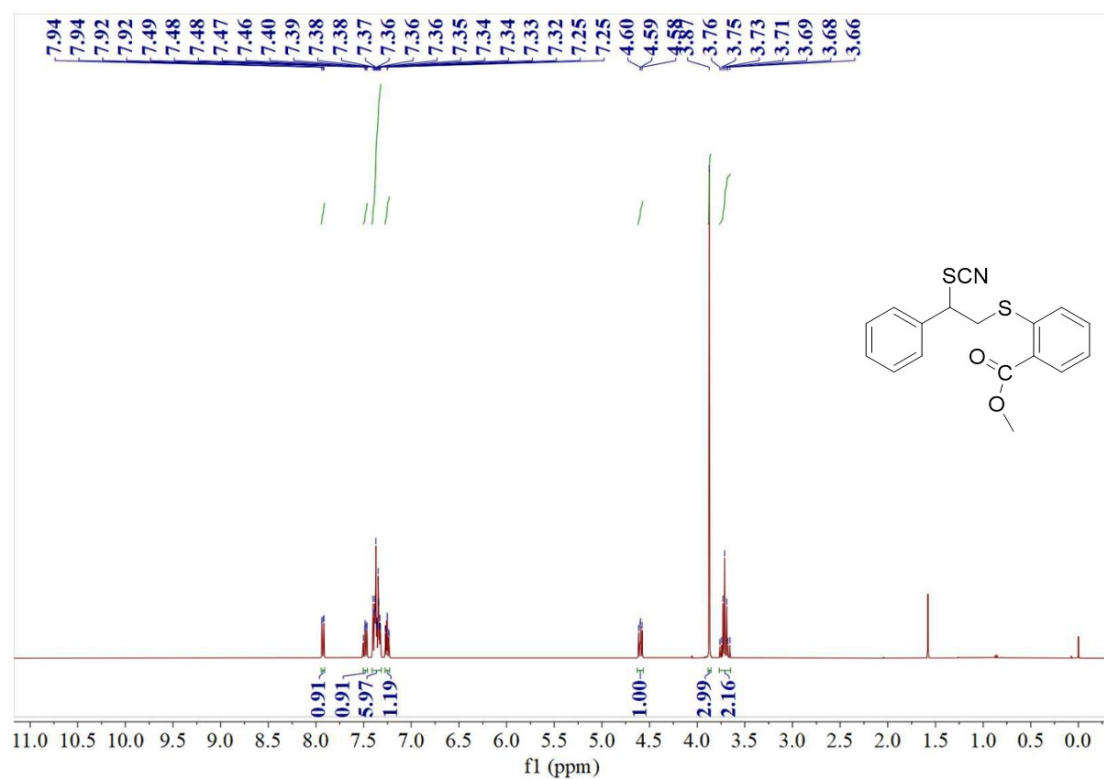


¹H NMR (400 MHz, CDCl₃) spectrum of 4ah

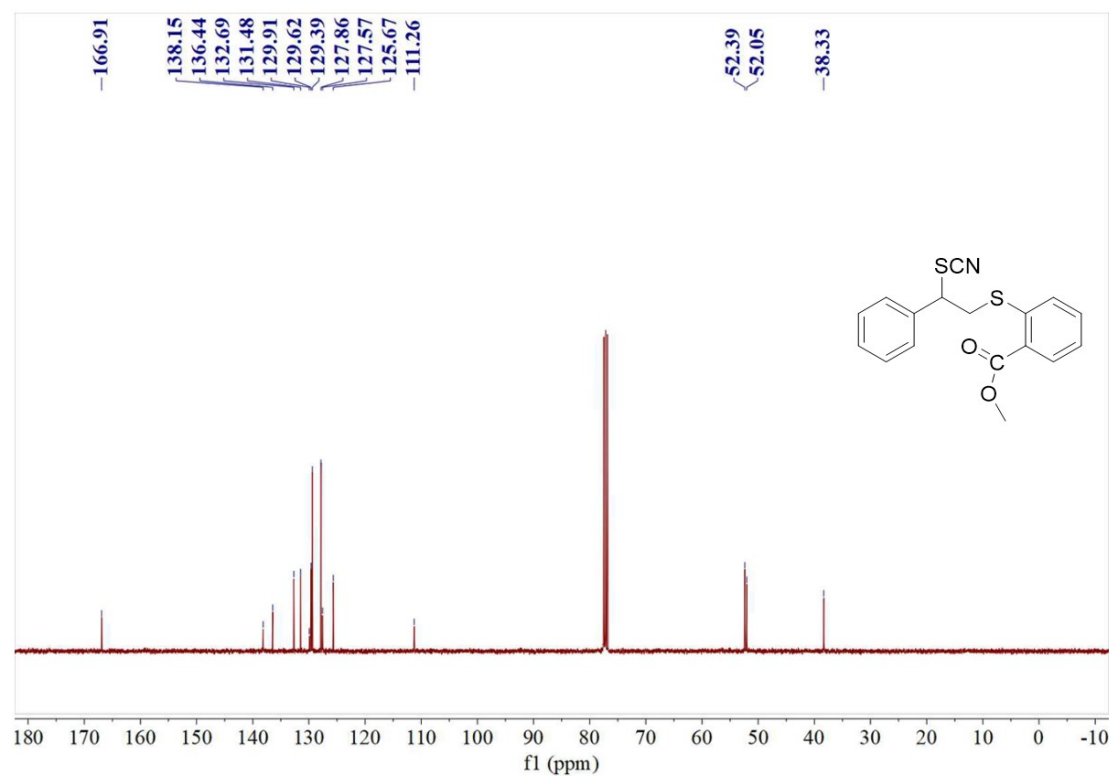


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ah

methyl 2-((2-phenyl-2-thiocyanatoethyl)thio)benzoate (4ai)

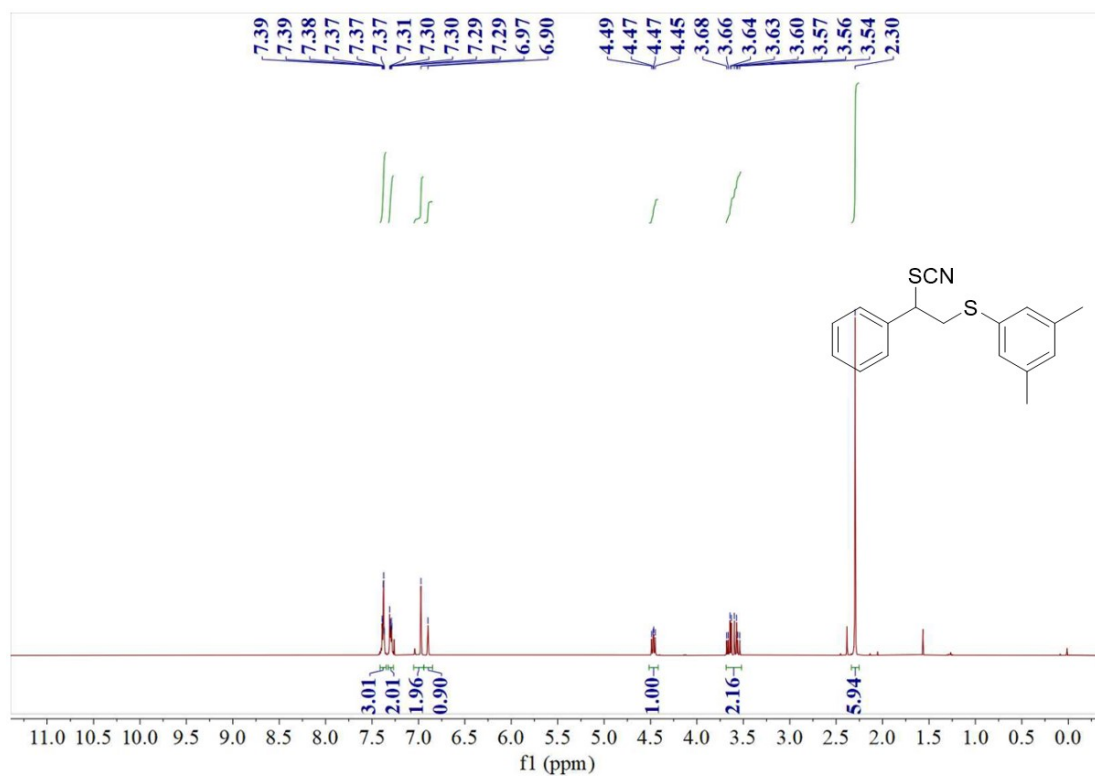


¹H NMR (400 MHz, CDCl₃) spectrum of 4ai

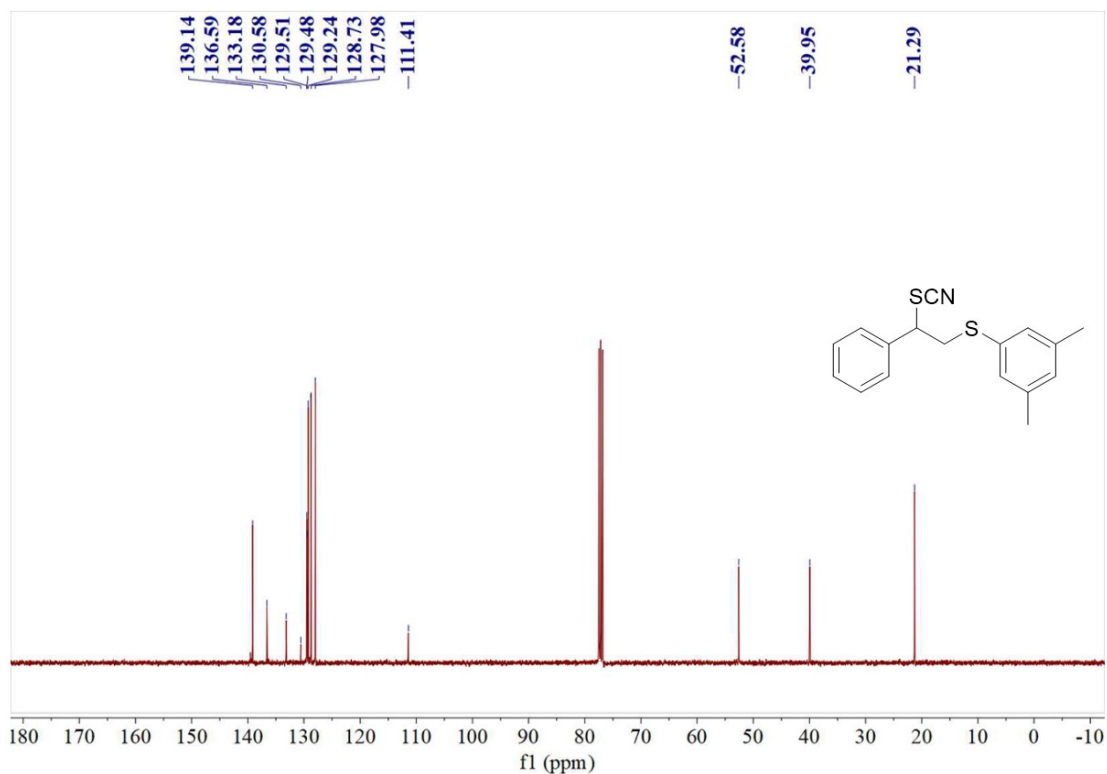


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ai

(3,5-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4aj)

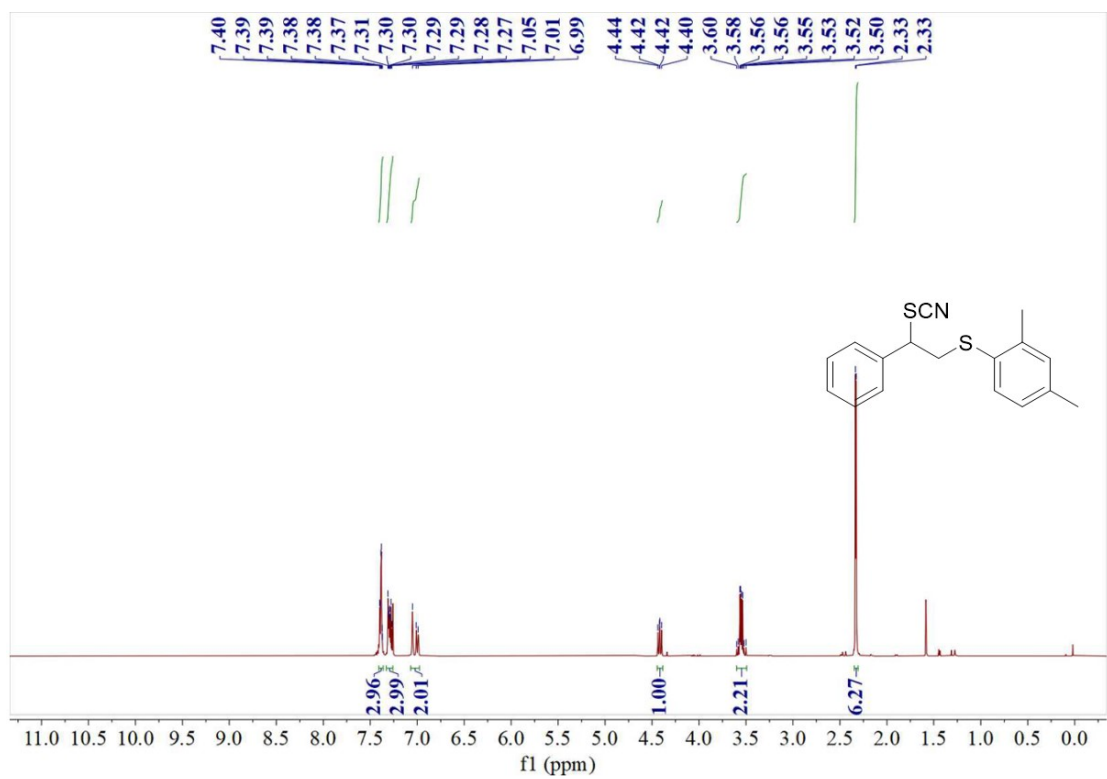


¹H NMR (400 MHz, CDCl₃) spectrum of 4aj

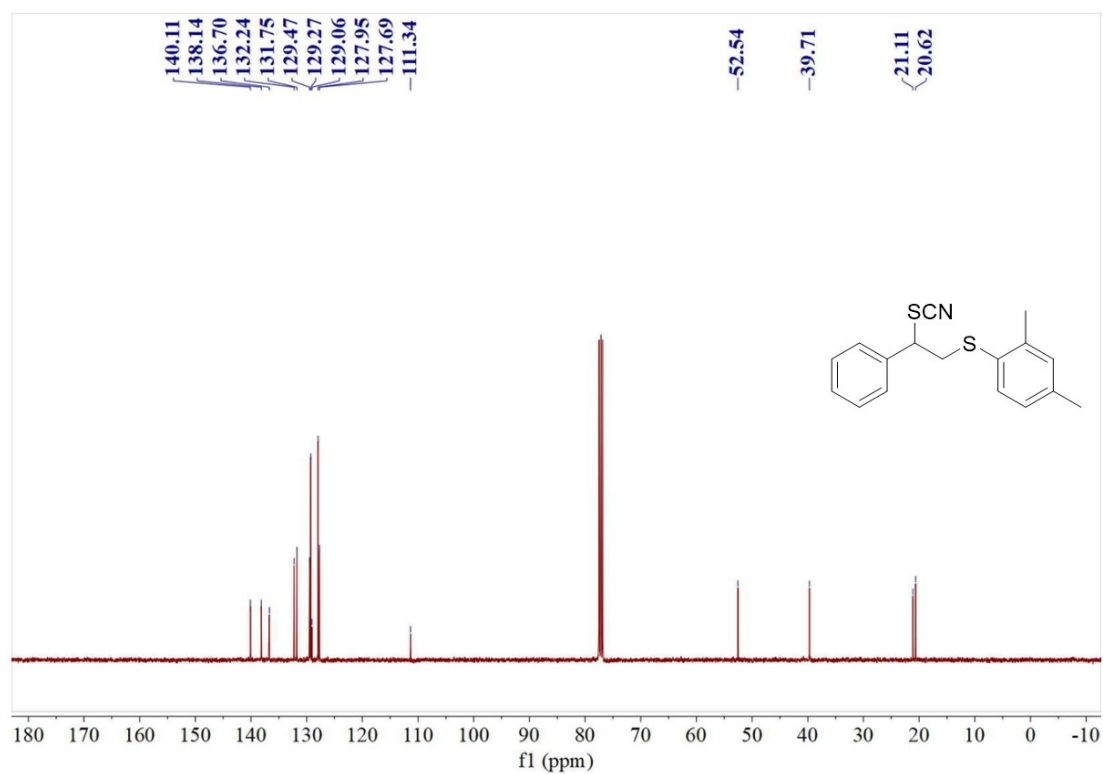


¹³C NMR (100 MHz, CDCl₃) spectrum of 4aj

(2,4-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4ak)

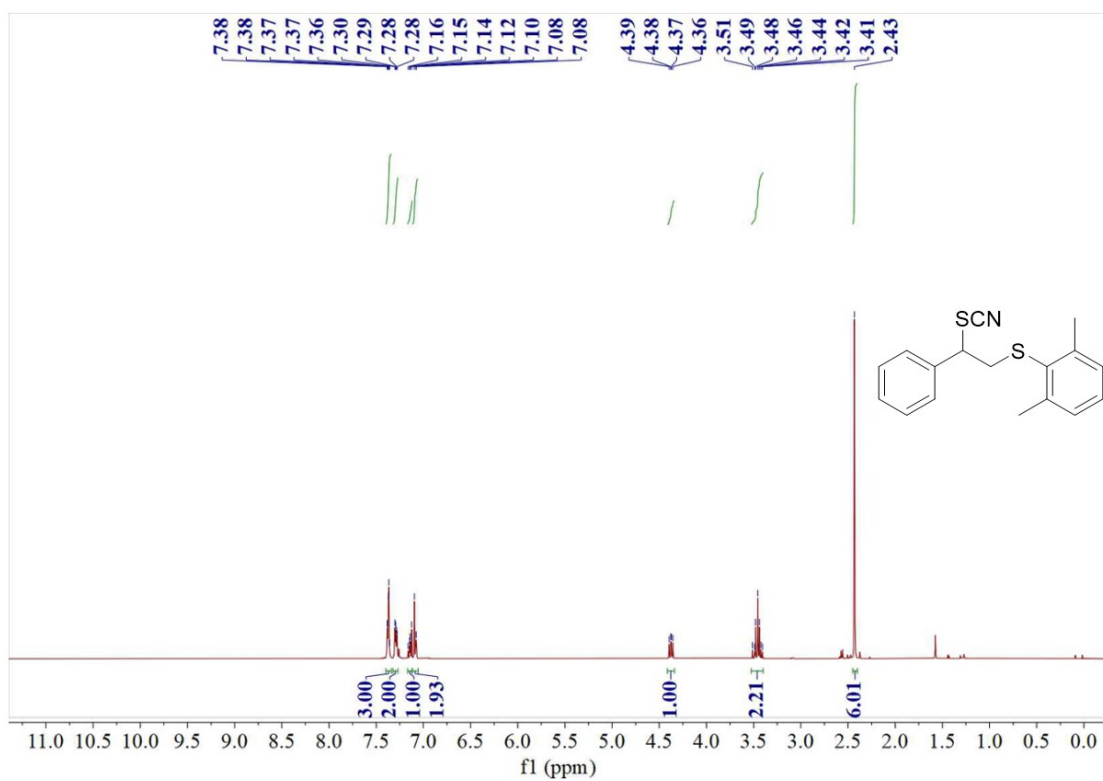


¹H NMR (400 MHz, CDCl₃) spectrum of 4ak

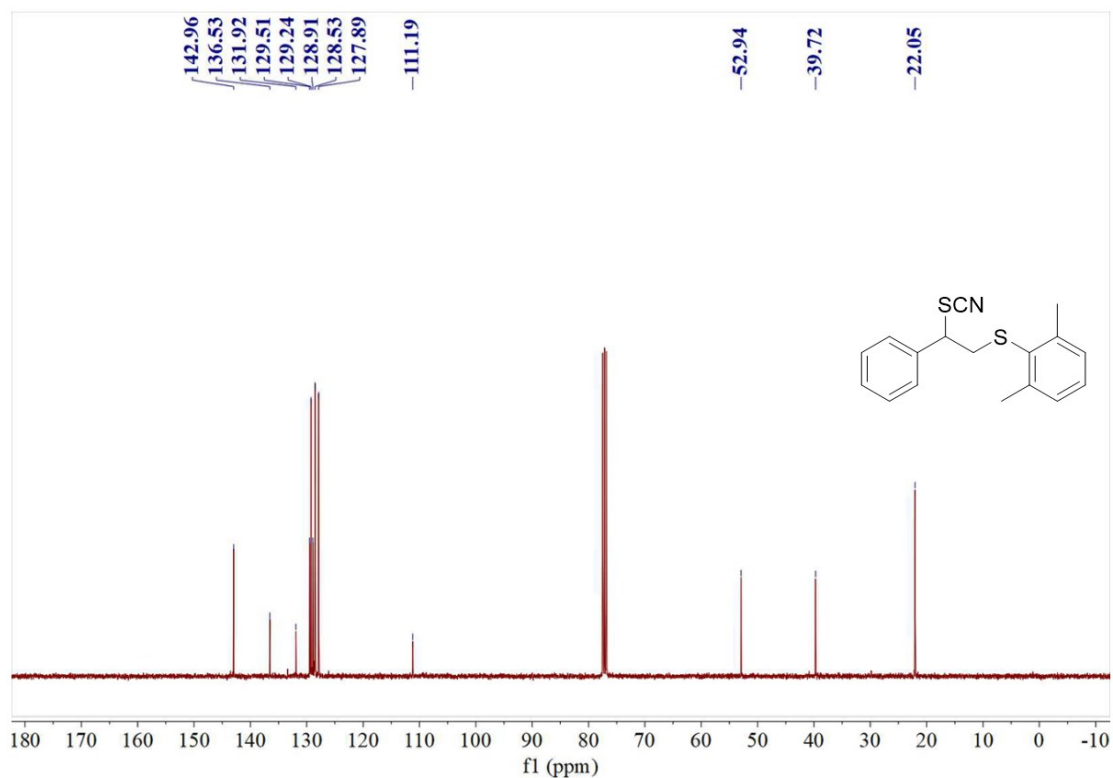


¹³C NMR (100 MHz, CDCl₃) spectrum of 4a

(2,6-dimethylphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4a)

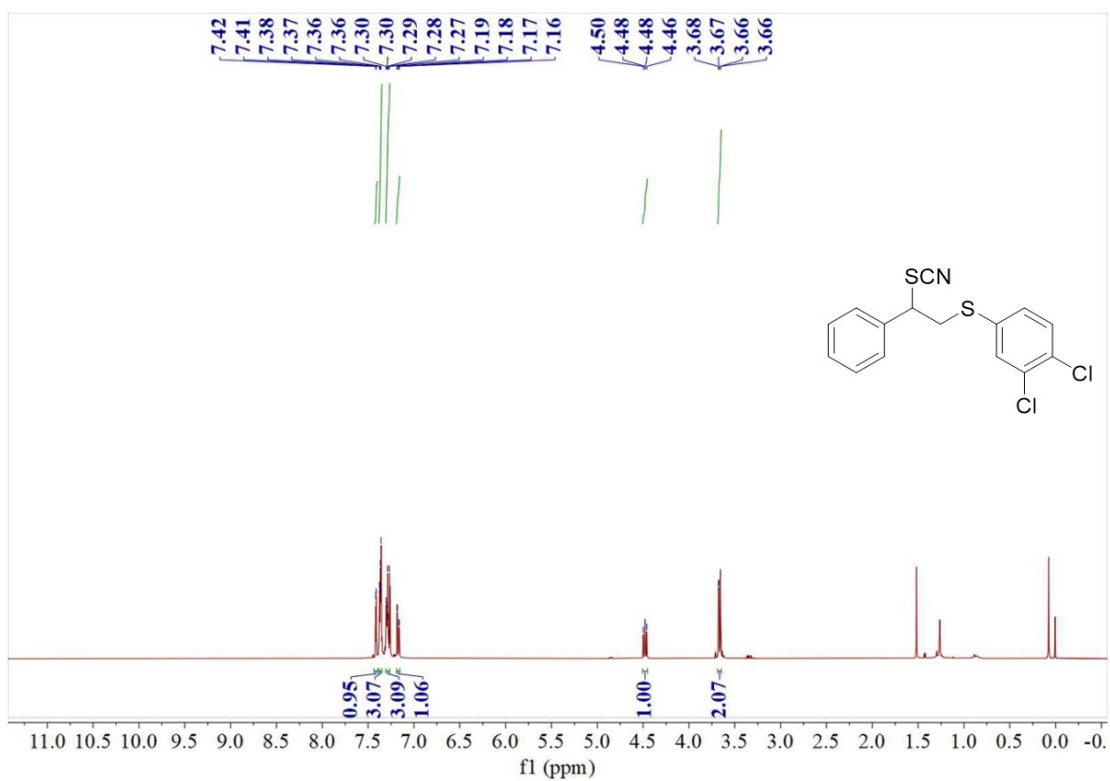


¹H NMR (400 MHz, CDCl₃) spectrum of 4a

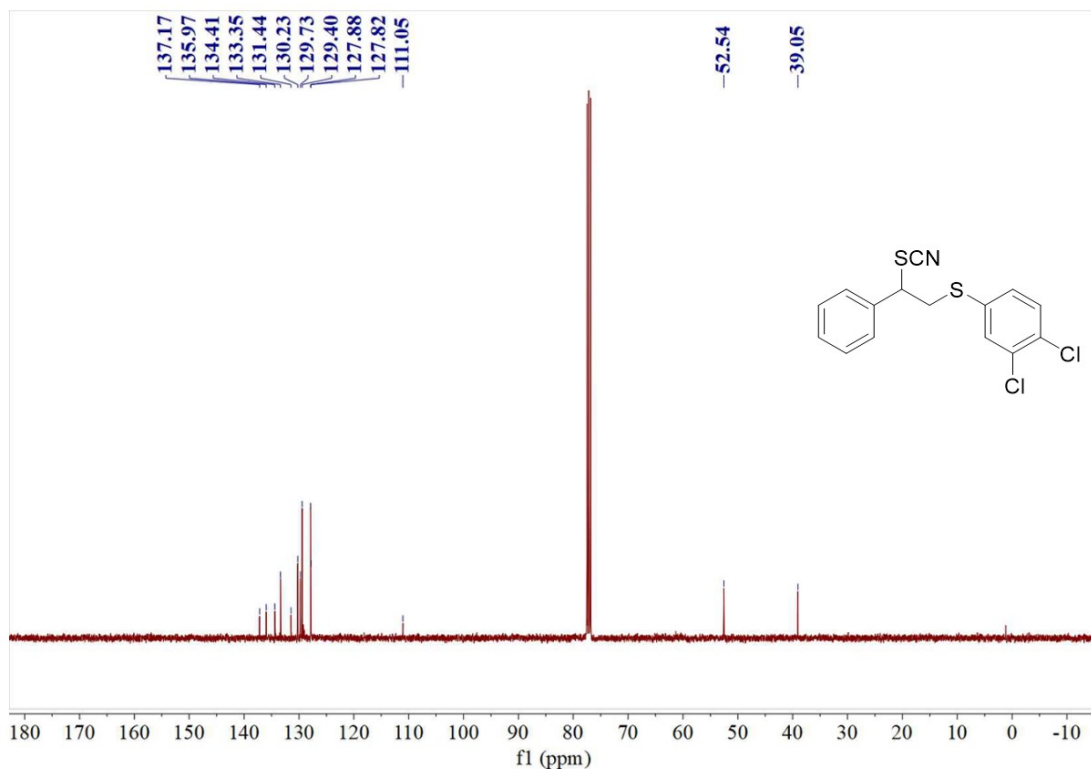


¹³C NMR (100 MHz, CDCl₃) spectrum of 4a

(3,4-dichlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4am)

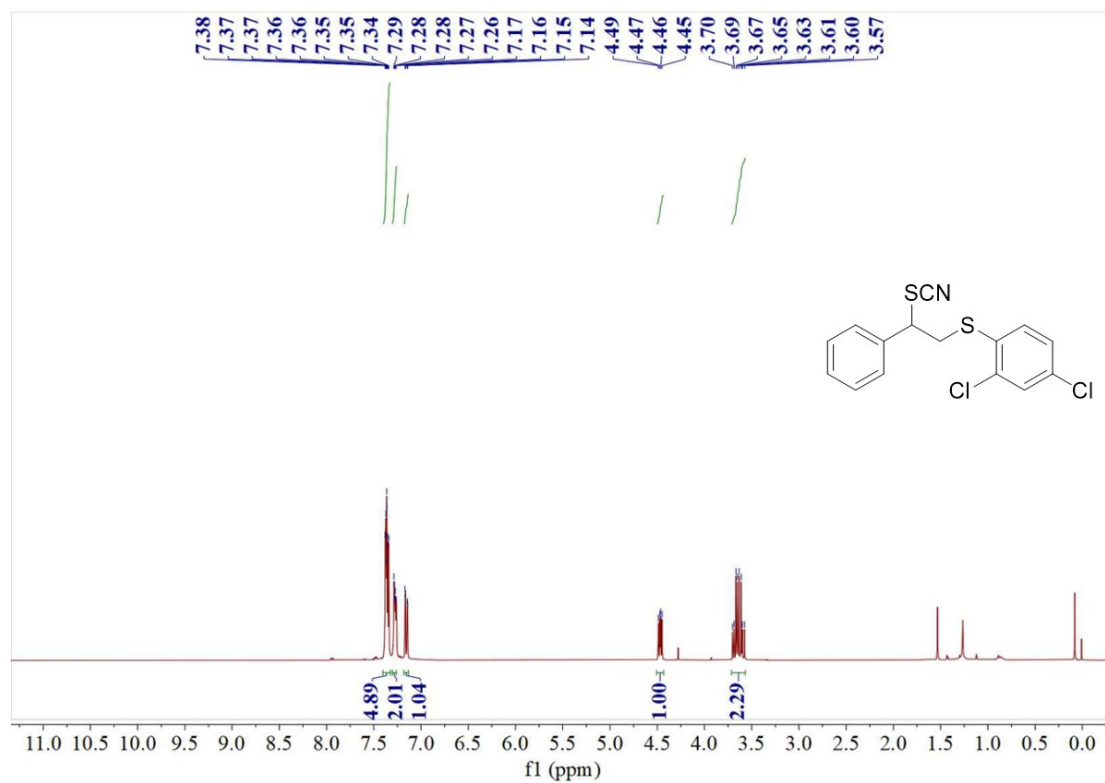


¹H NMR (400 MHz, CDCl₃) spectrum of 4am

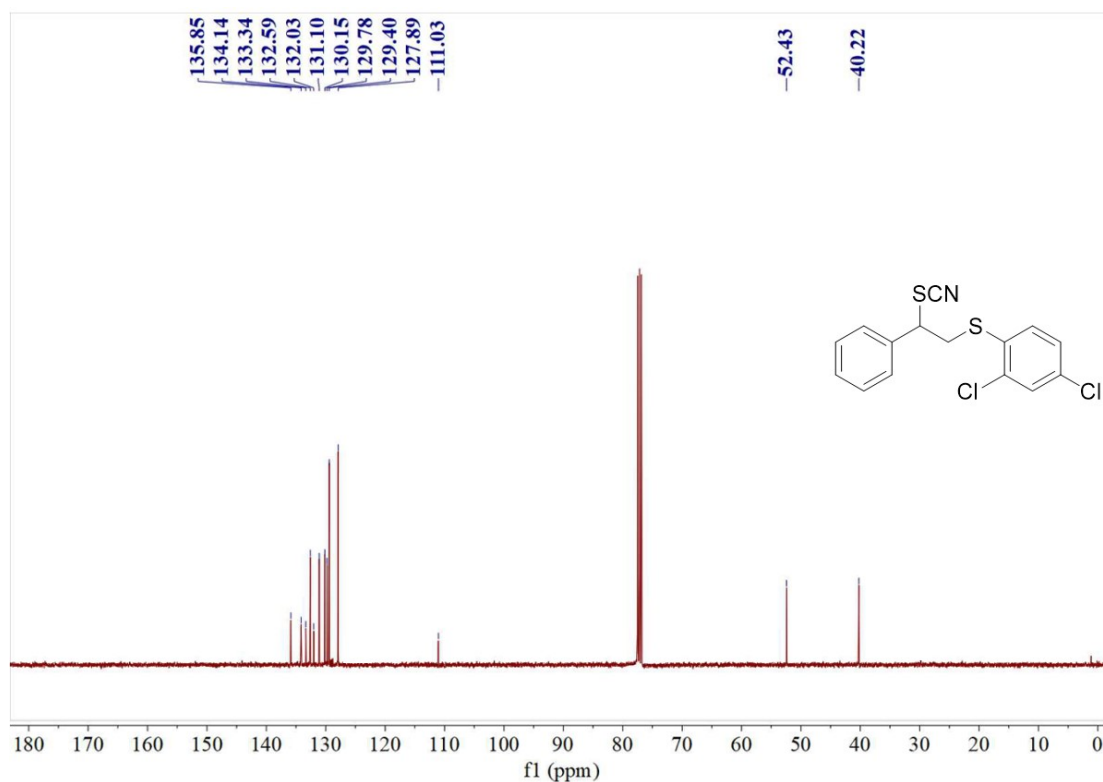


¹³C NMR (100 MHz, CDCl₃) spectrum of 4am

(2,4-dichlorophenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (4an)

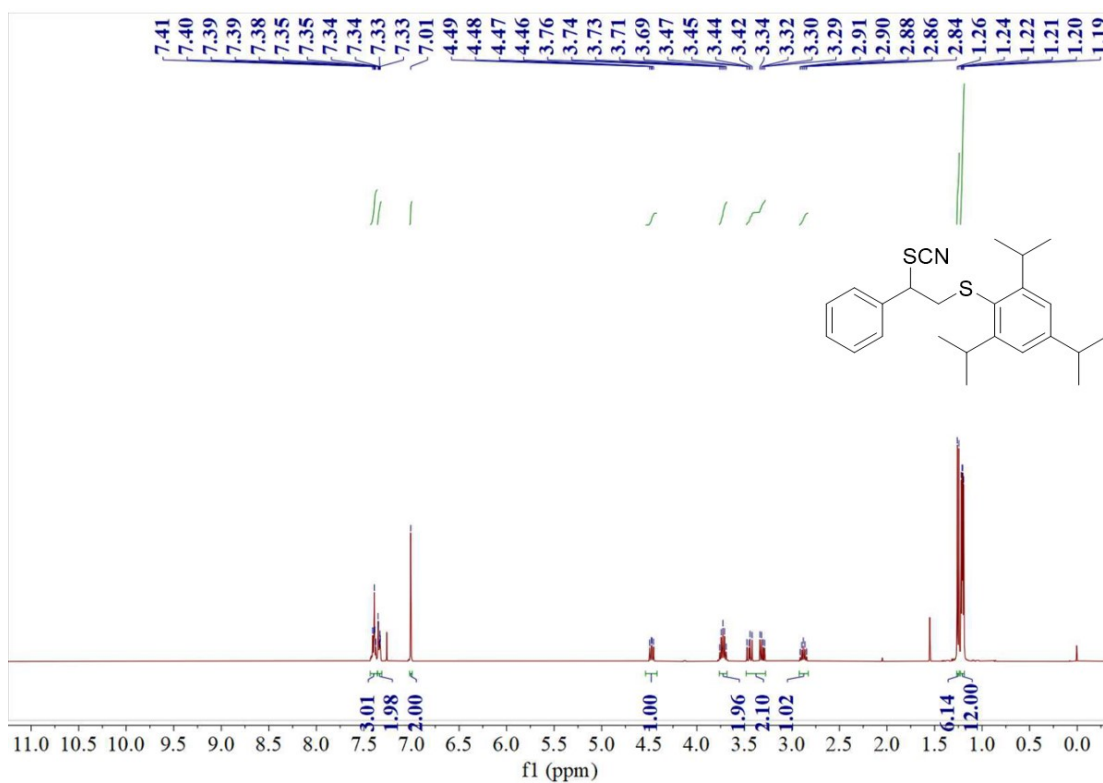


¹H NMR (400 MHz, CDCl₃) spectrum of 4an

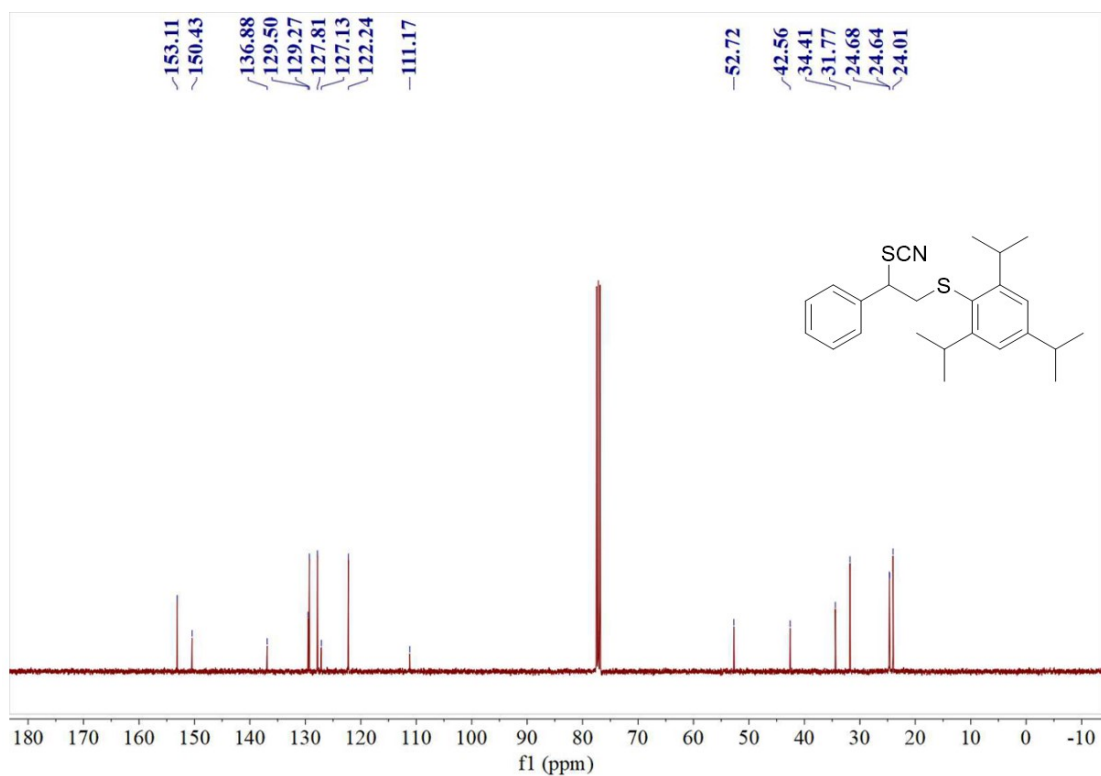


¹³C NMR (100 MHz, CDCl₃) spectrum of 4an

(2-phenyl-2-thiocyanatoethyl)(2,4,6-triisopropylphenyl)sulfane (4ao)

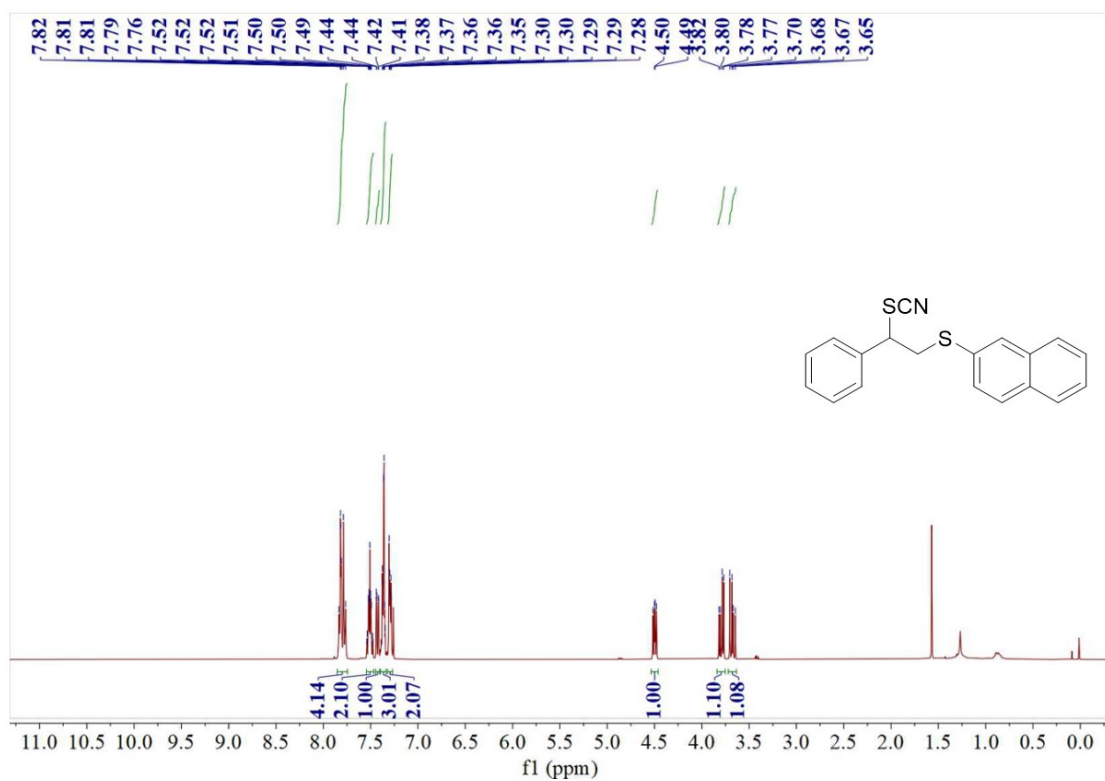


¹H NMR (400 MHz, CDCl₃) spectrum of 4ao

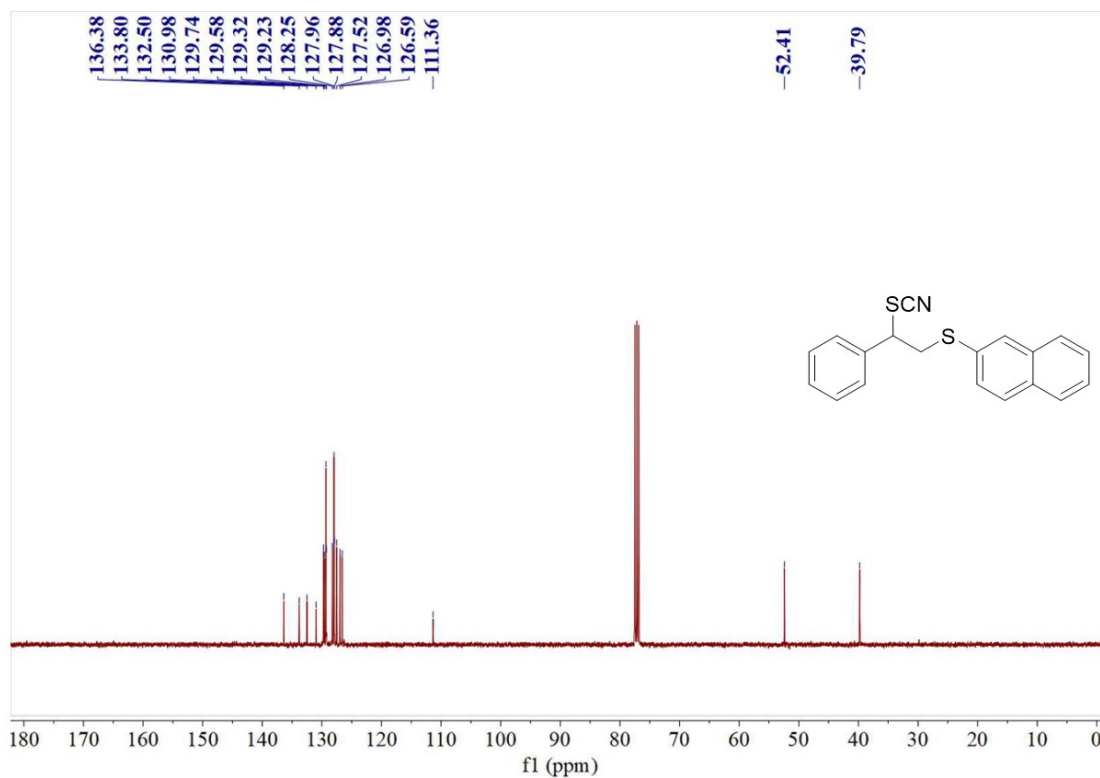


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ao

naphthalen-2-yl(2-phenyl-2-thiocyanatoethyl)sulfane (4ap)

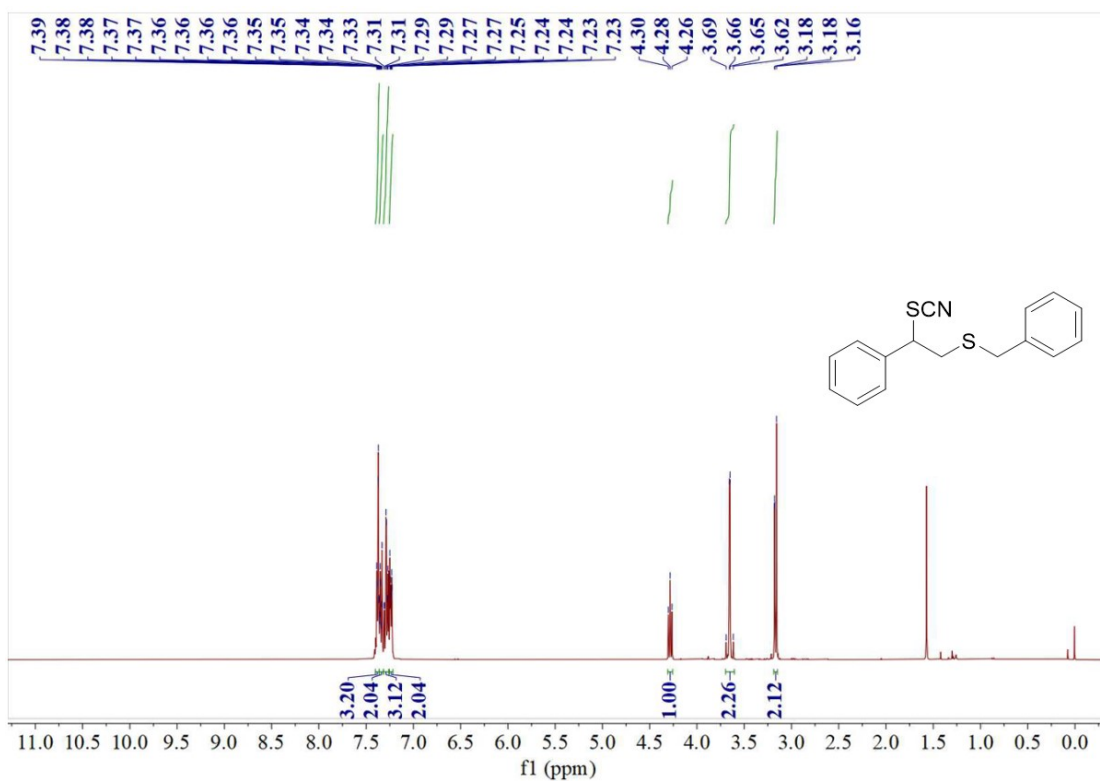


¹H NMR (400 MHz, CDCl₃) spectrum of 4ap

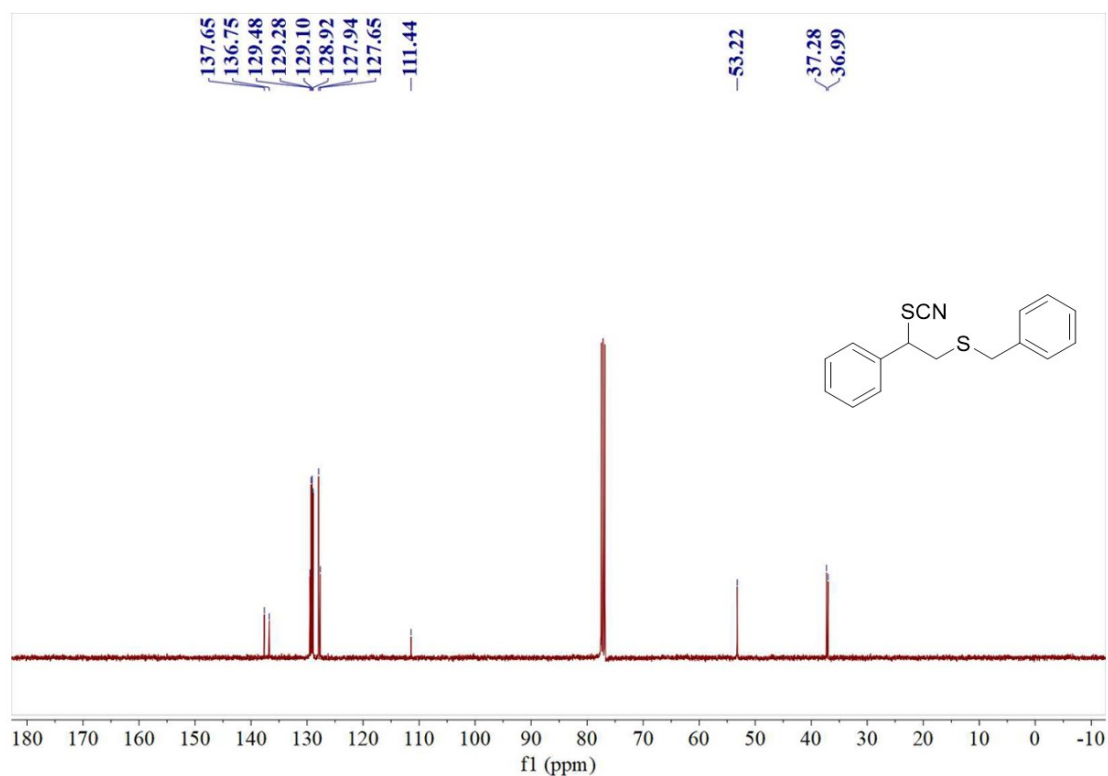


¹³C NMR (100 MHz, CDCl₃) spectrum of 4p

benzyl(2-phenyl-2-thiocyanatoethyl)sulfane (4aq)

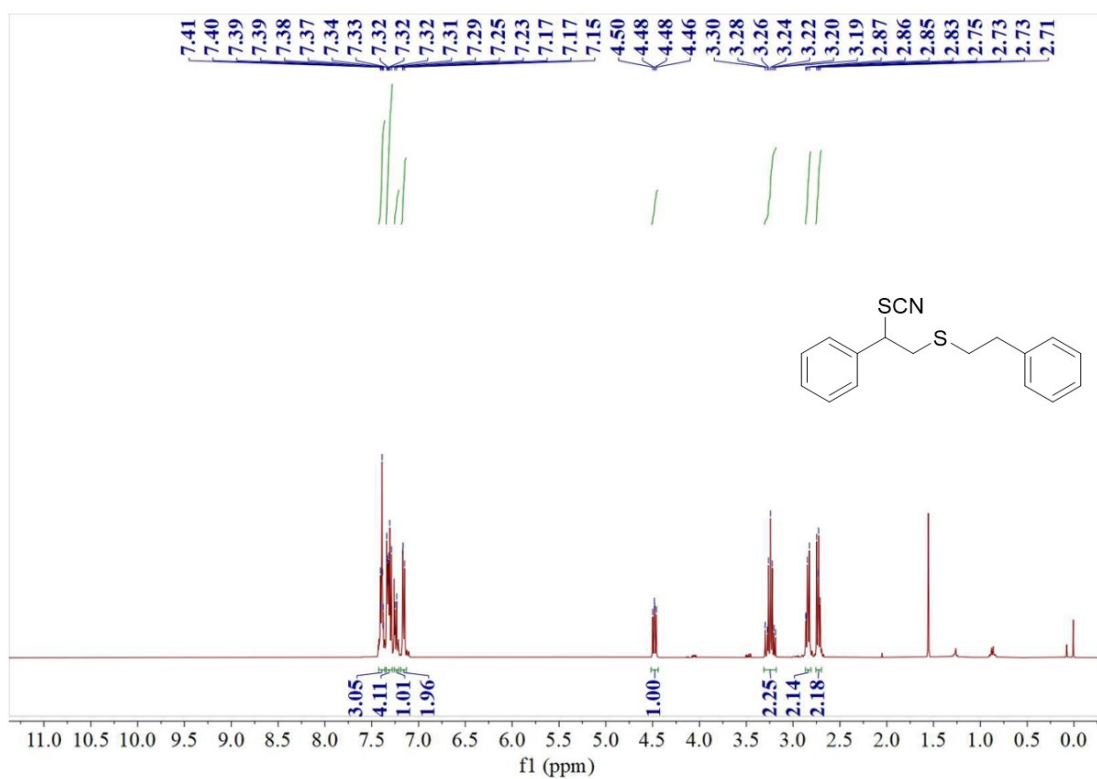


¹H NMR (400 MHz, CDCl₃) spectrum of 4aq

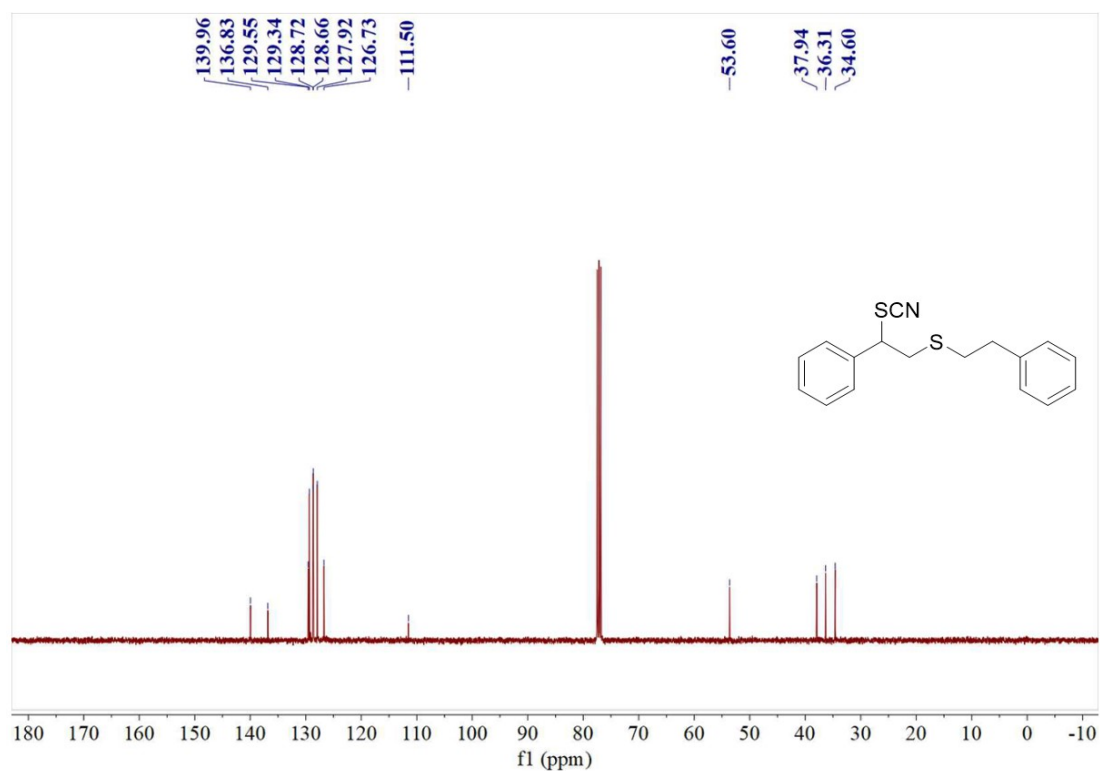


¹³C NMR (100 MHz, CDCl₃) spectrum of 4aq

phenethyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ar)

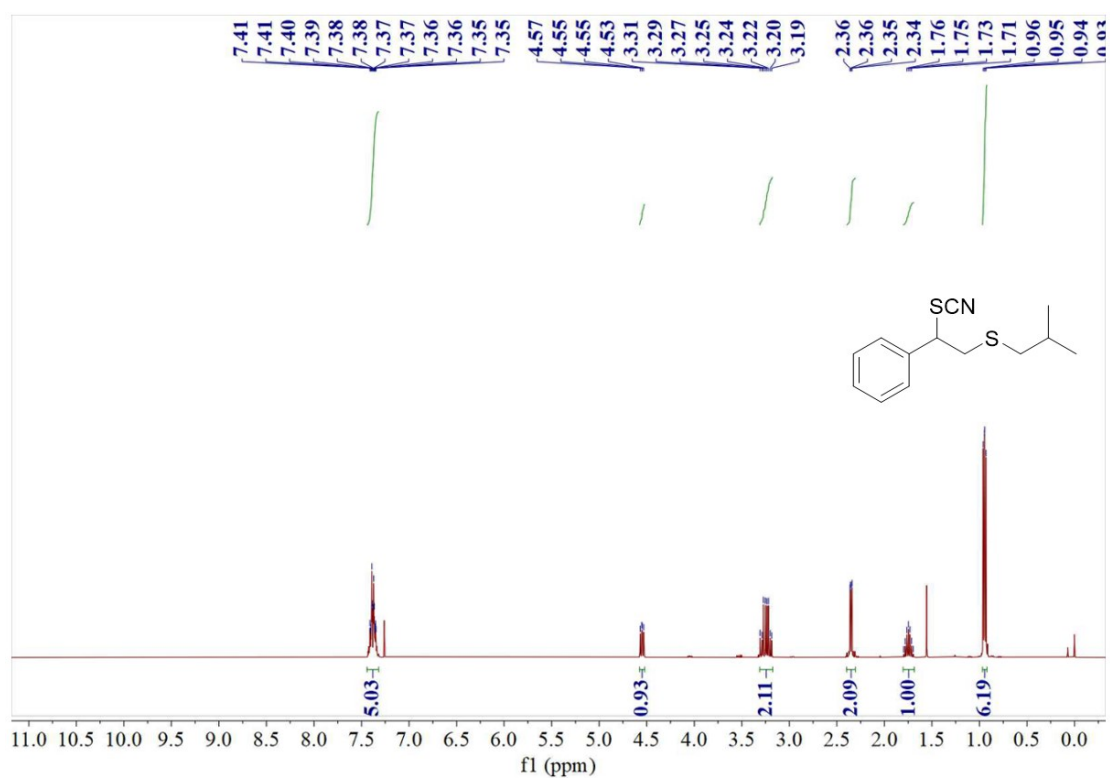


¹H NMR (400 MHz, CDCl₃) spectrum of 4ar

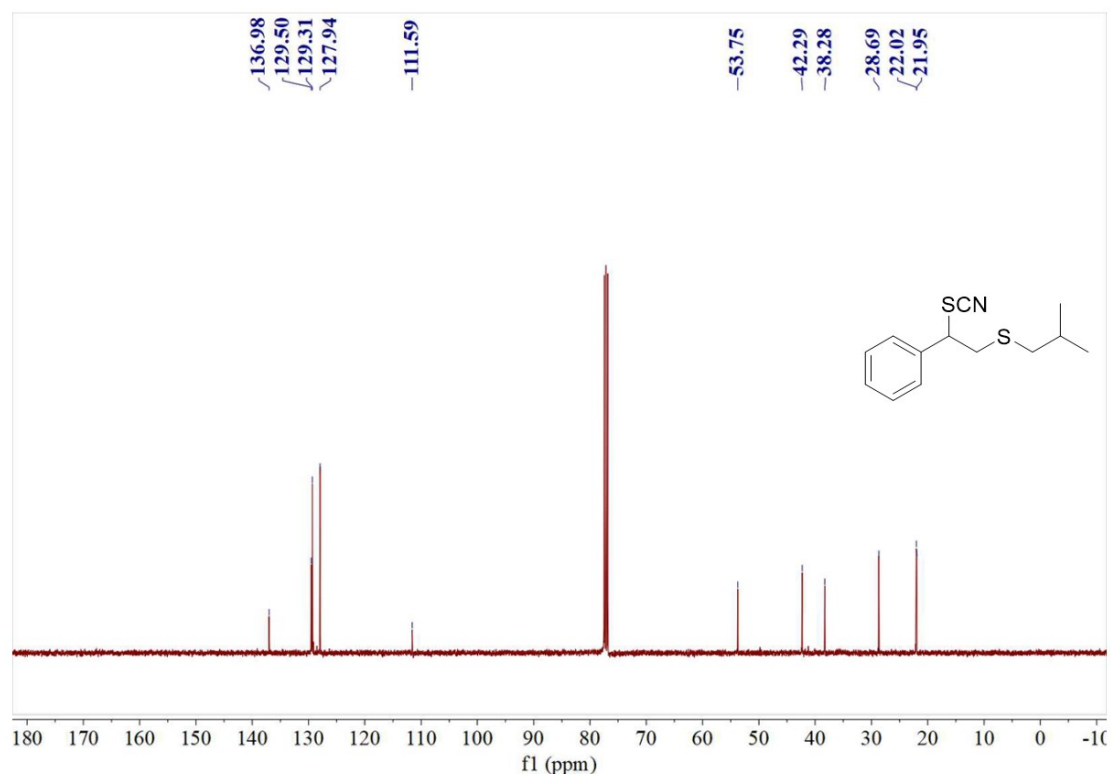


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ar

isobutyl(2-phenyl-2-thiocyanatoethyl)sulfane (4as)

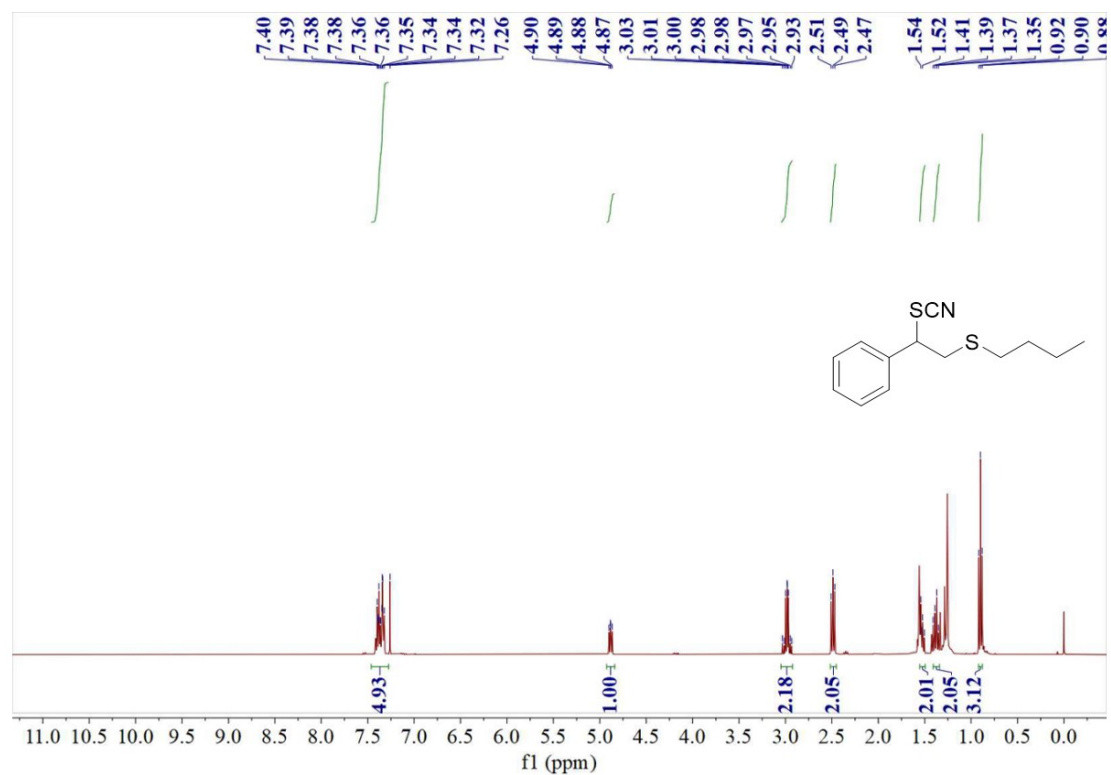


¹H NMR (400 MHz, CDCl₃) spectrum of 4as

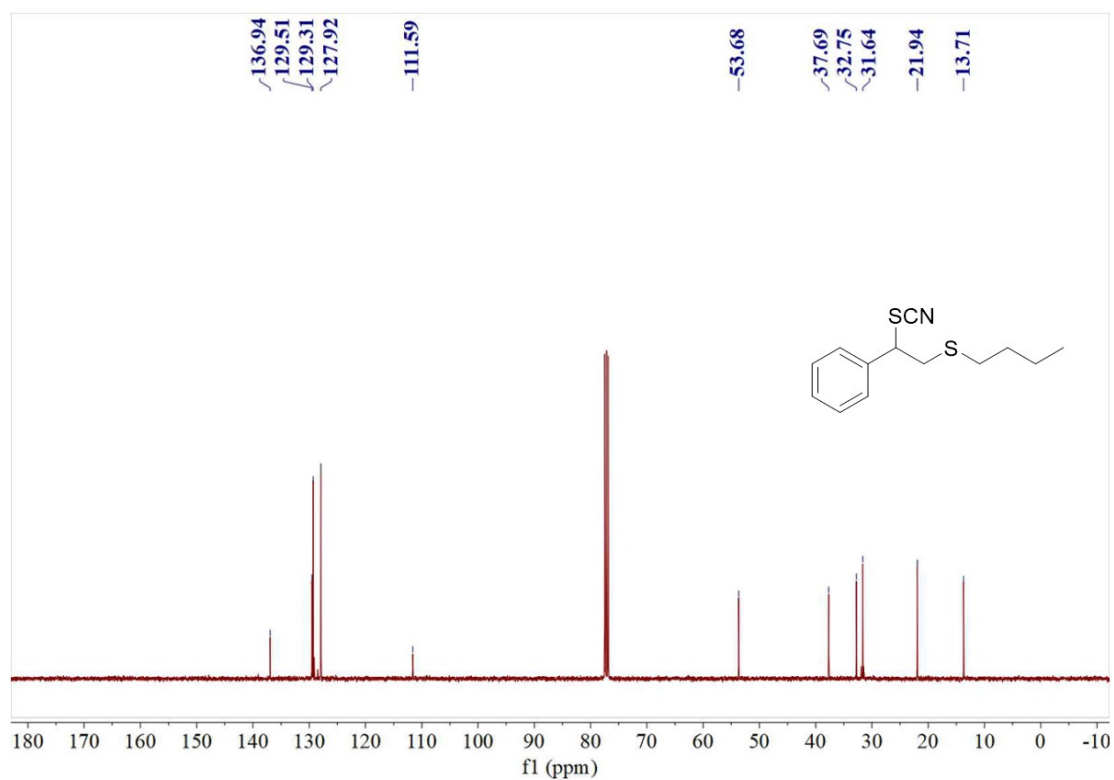


¹³C NMR (100 MHz, CDCl₃) spectrum of 4as

butyl(2-phenyl-2-thiocyanatoethyl)sulfane (4at)

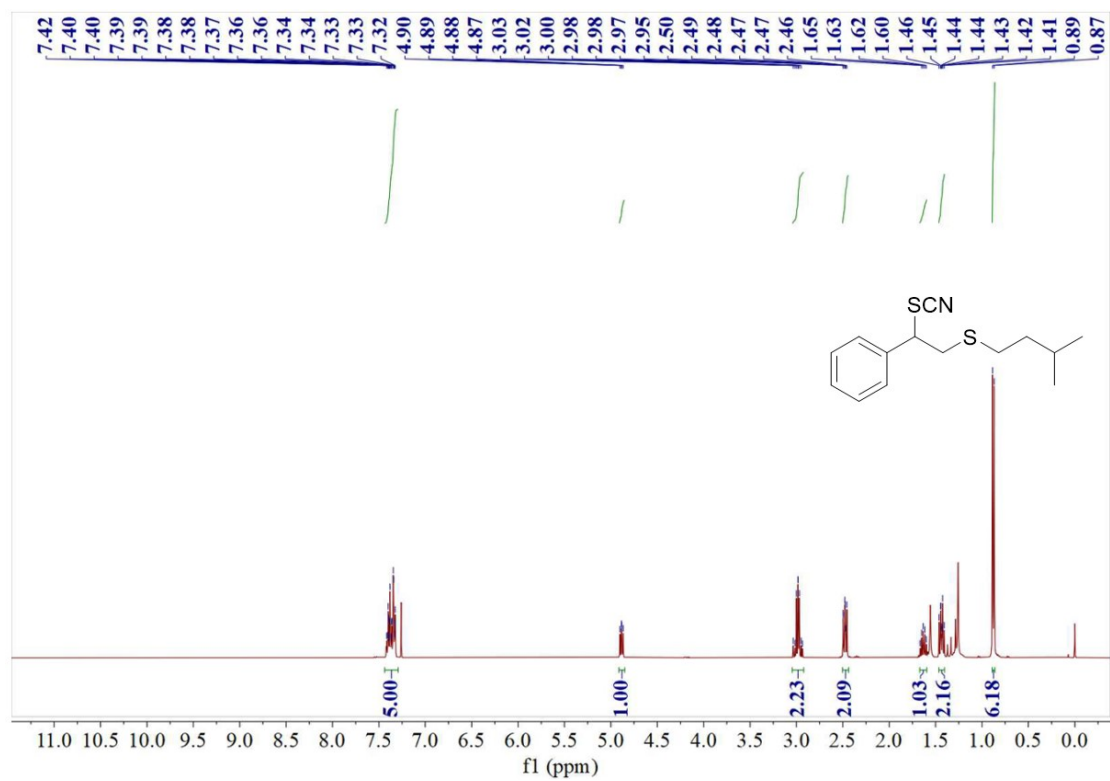


¹H NMR (400 MHz, CDCl₃) spectrum of 4at

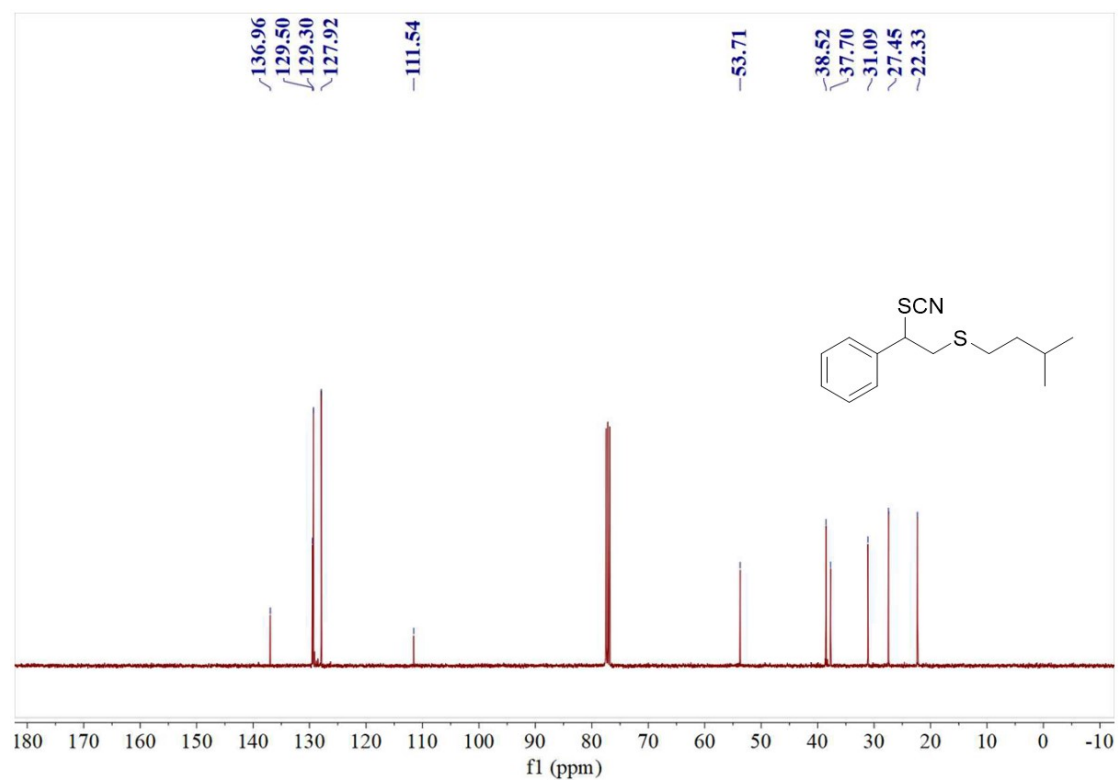


¹³C NMR (100 MHz, CDCl₃) spectrum of 4t

isopentyl(2-phenyl-2-thiocyanatoethyl)sulfane (4au)

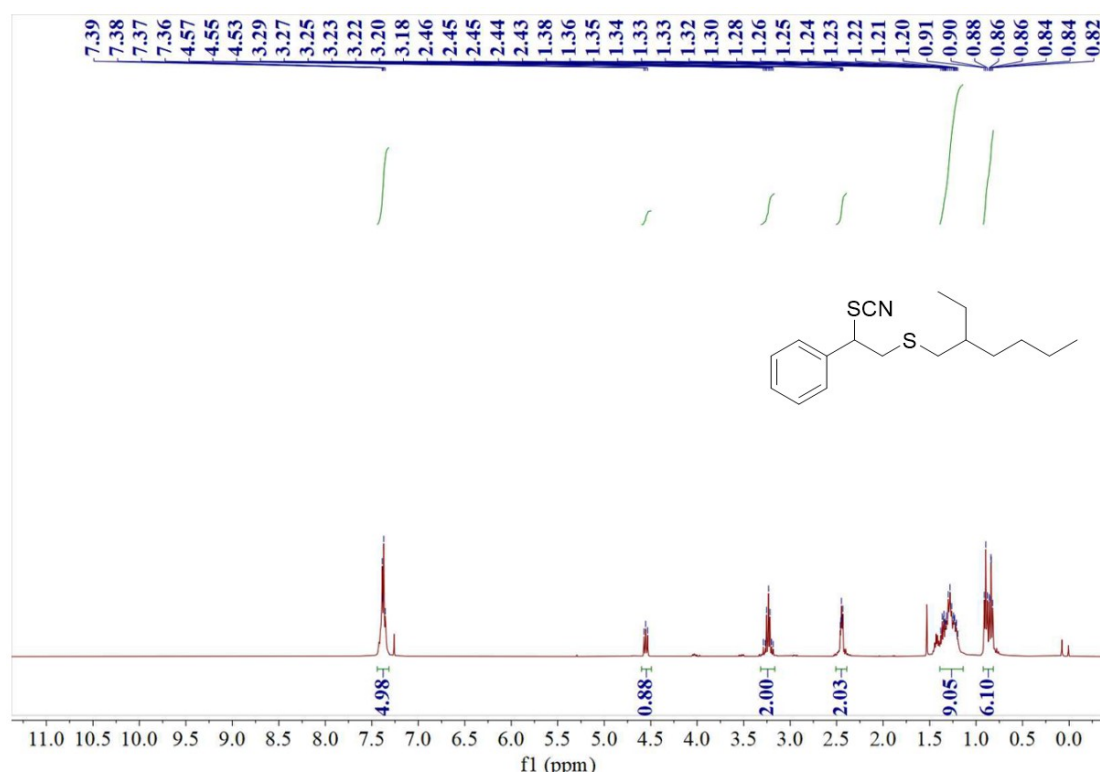


¹H NMR (400 MHz, CDCl₃) spectrum of 4au

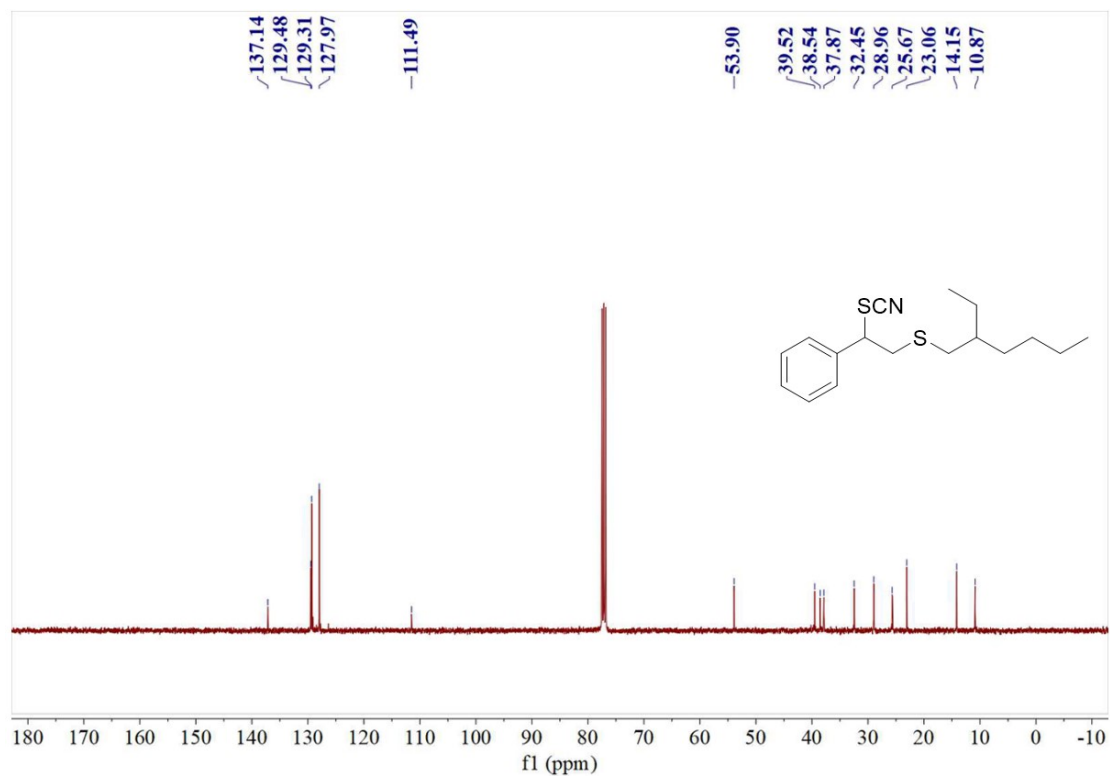


¹³C NMR (100 MHz, CDCl₃) spectrum of 4au

heptan-3-yl(2-phenyl-2-thiocyanatoethyl)sulfane (4av)

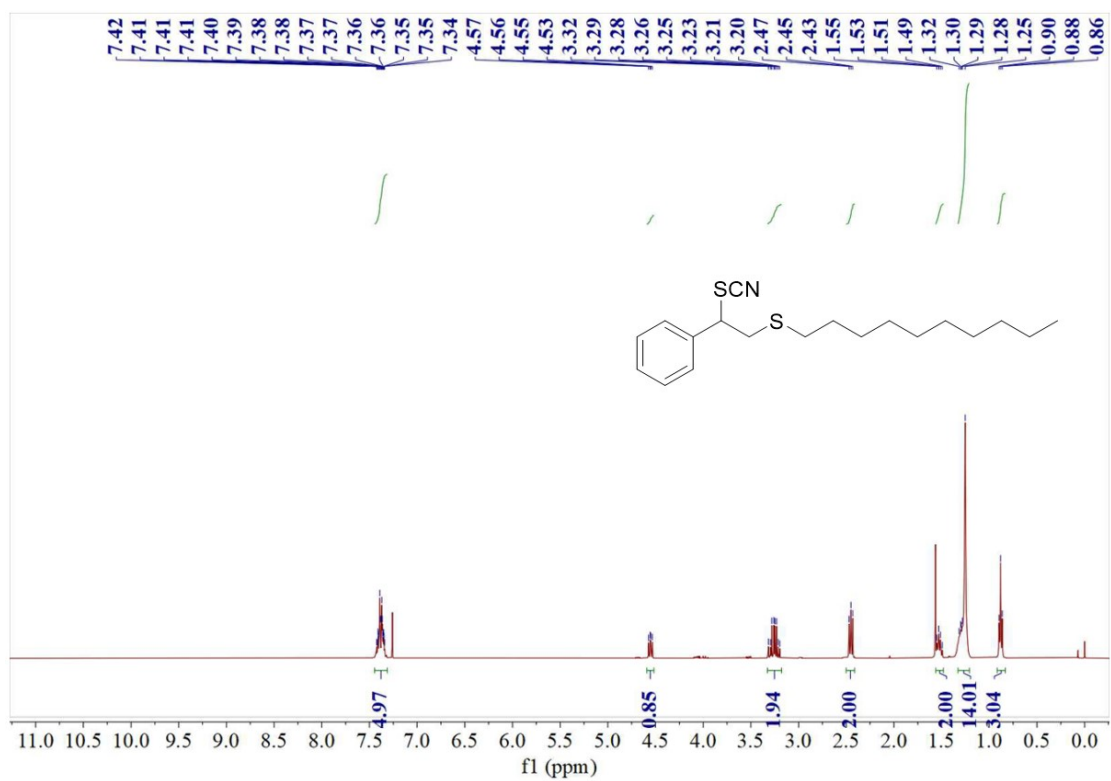


¹H NMR (400 MHz, CDCl₃) spectrum of 4av

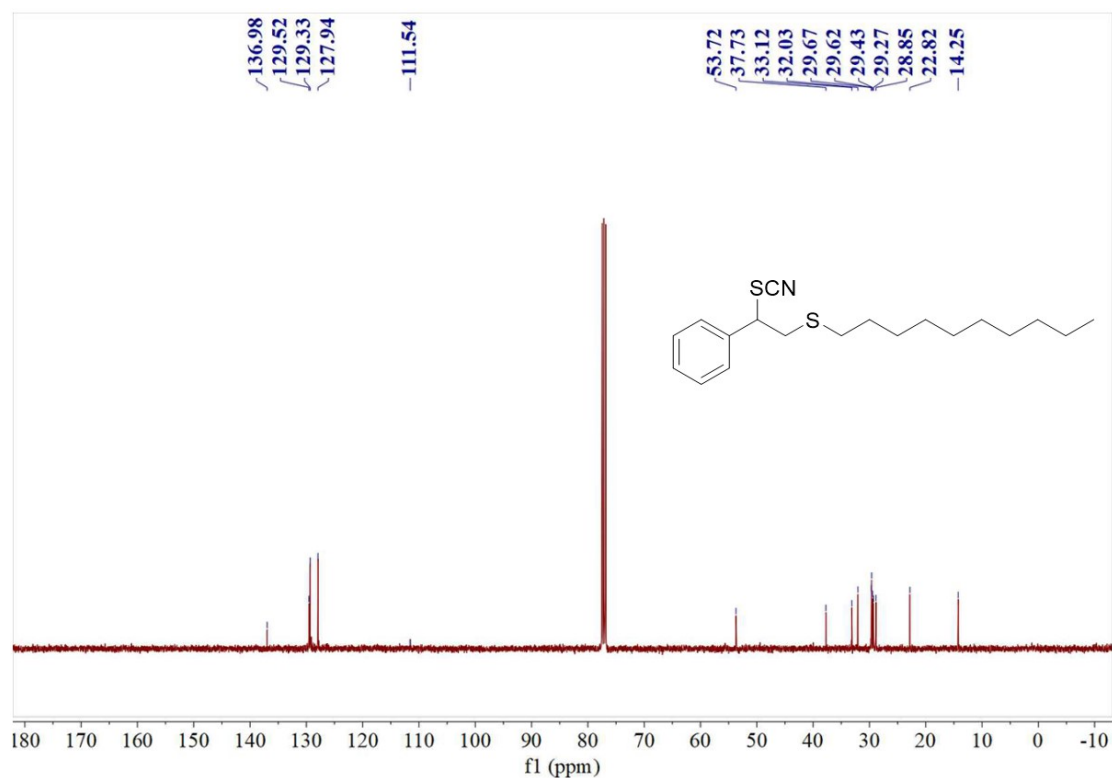


¹³C NMR (100 MHz, CDCl₃) spectrum of 4av

decyl(2-phenyl-2-thiocyanatoethyl)sulfane (4aw)

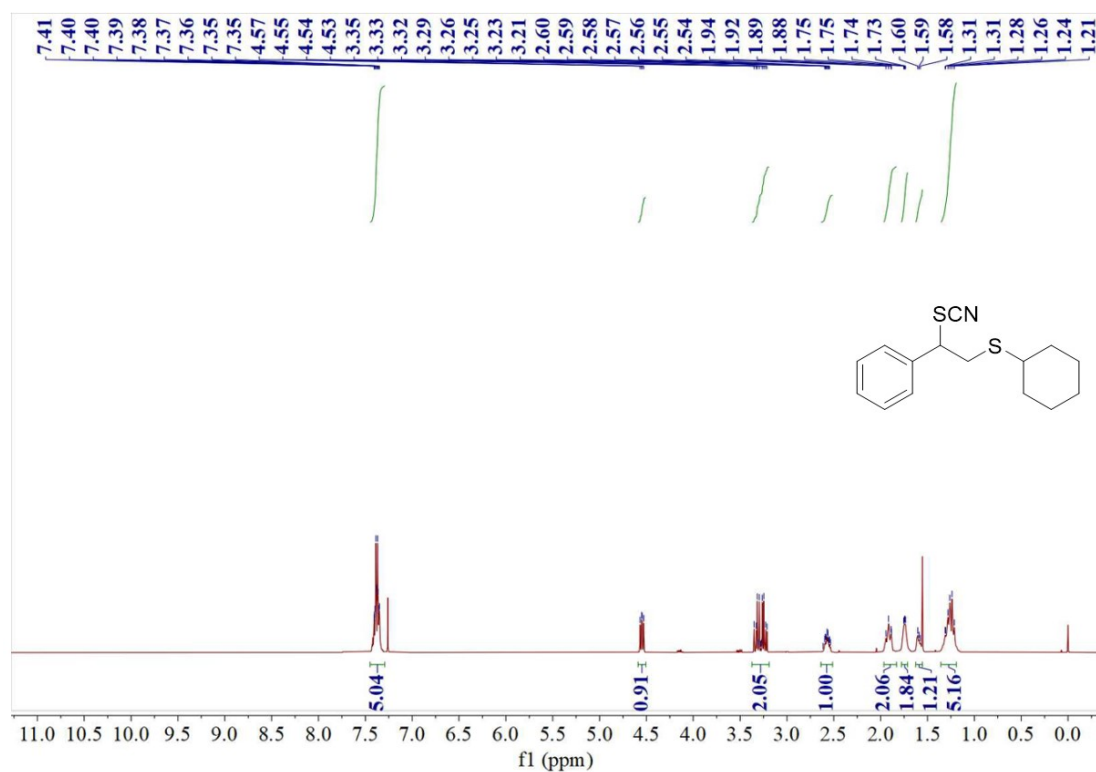


¹H NMR (400 MHz, CDCl₃) spectrum of 4aw

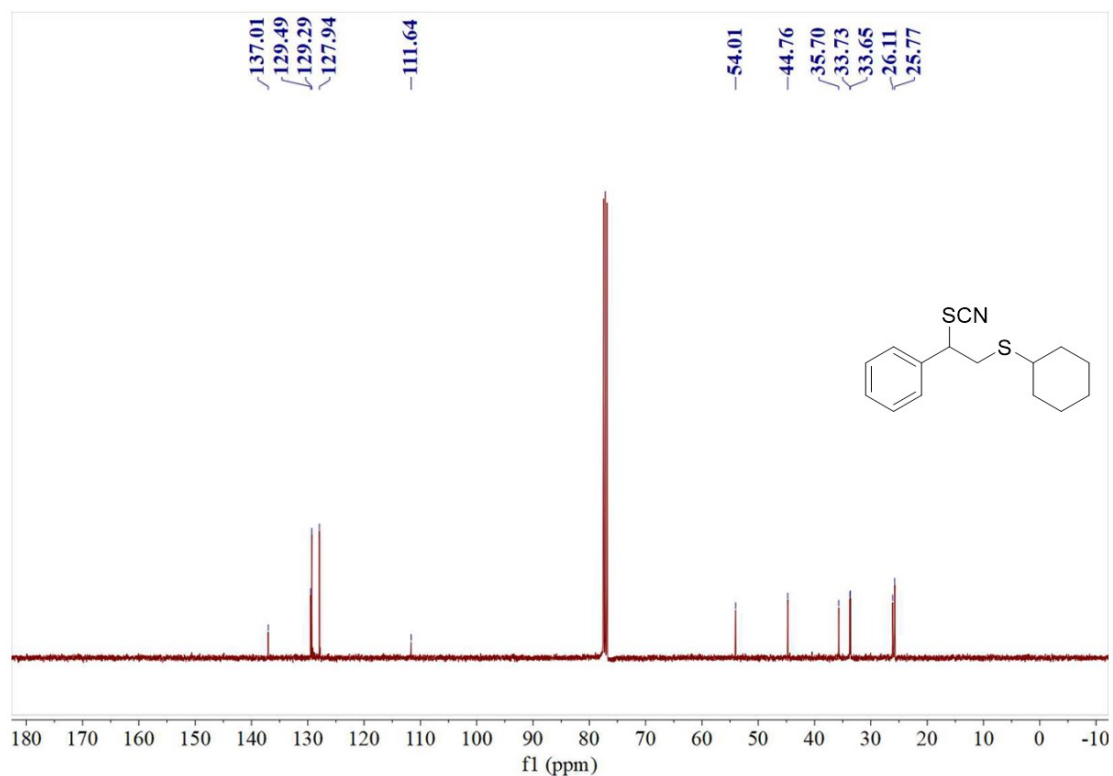


¹³C NMR (100 MHz, CDCl₃) spectrum of 4w

cyclohexyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ax)

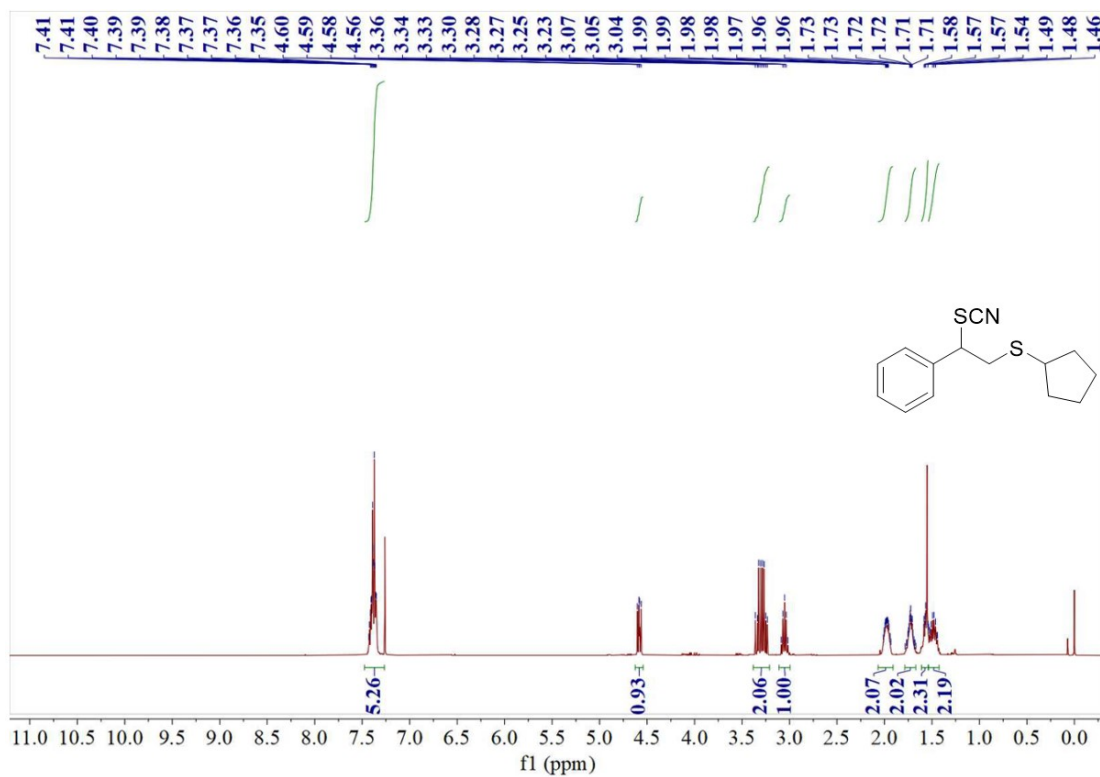


¹H NMR (400 MHz, CDCl₃) spectrum of 4ax

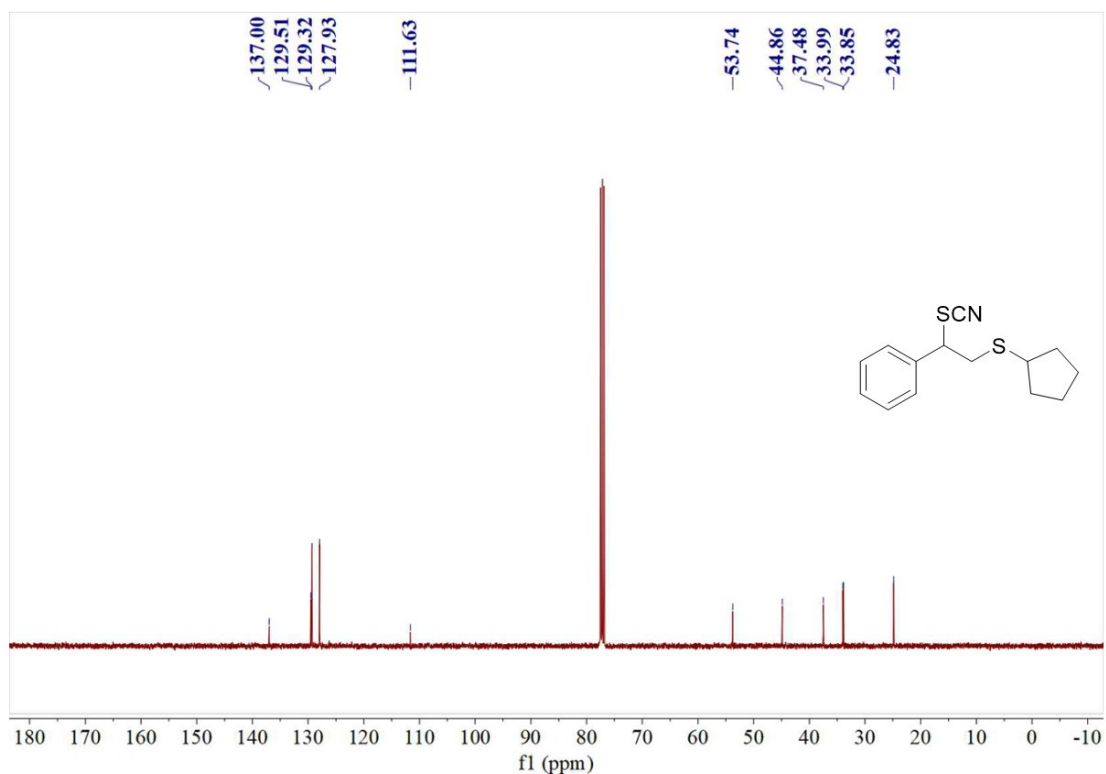


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ax

cyclopentyl(2-phenyl-2-thiocyanatoethyl)sulfane (4ay)

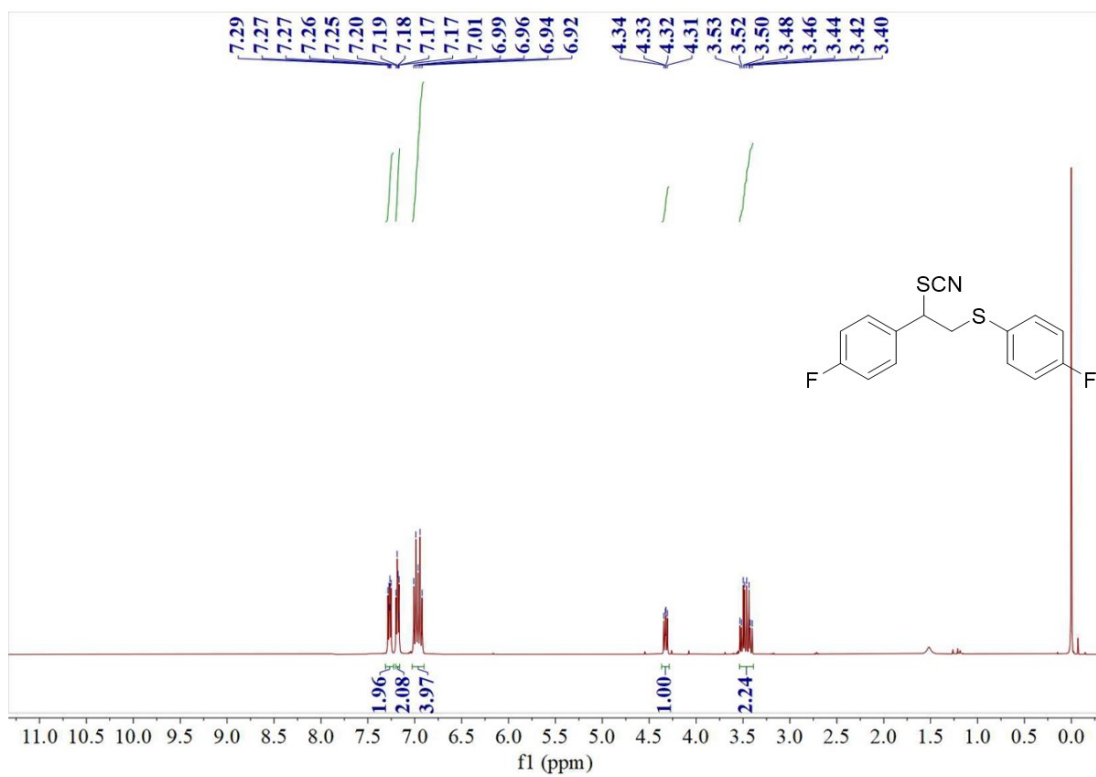


¹H NMR (400 MHz, CDCl₃) spectrum of 4ay

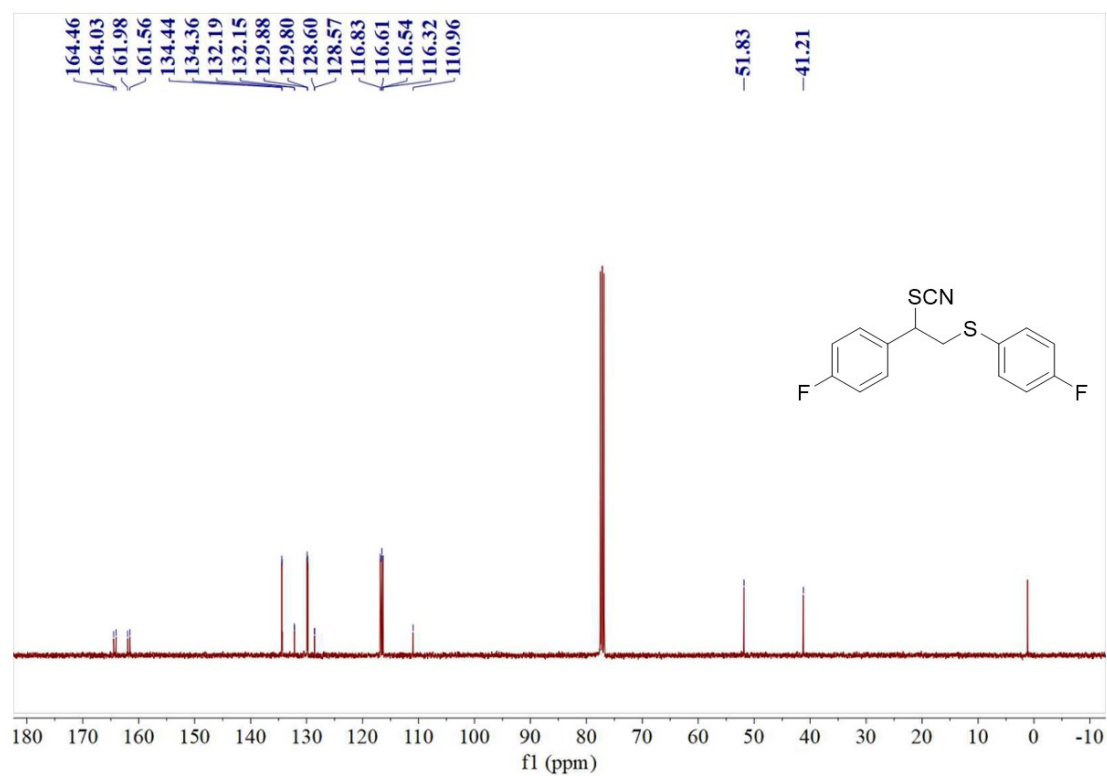


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ay

(4-fluorophenyl)(2-(4-fluorophenyl)-2-thiocyanatoethyl)sulfane (4ba)

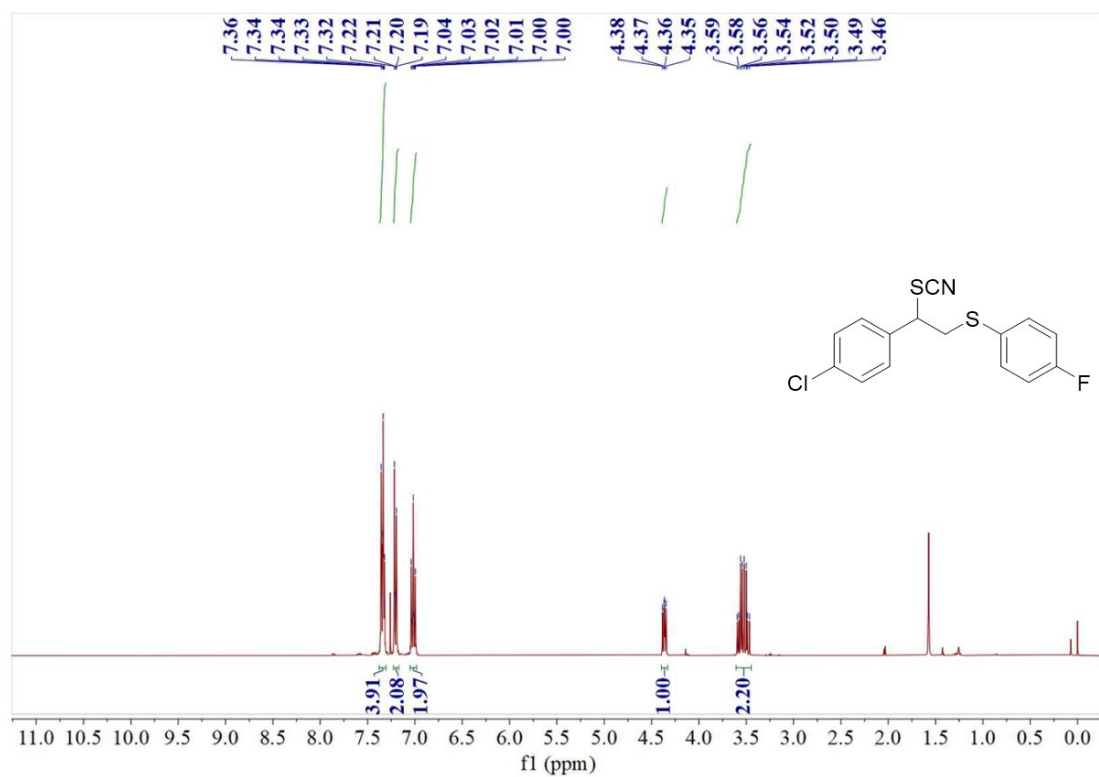


¹H NMR (400 MHz, CDCl₃) spectrum of 4ba

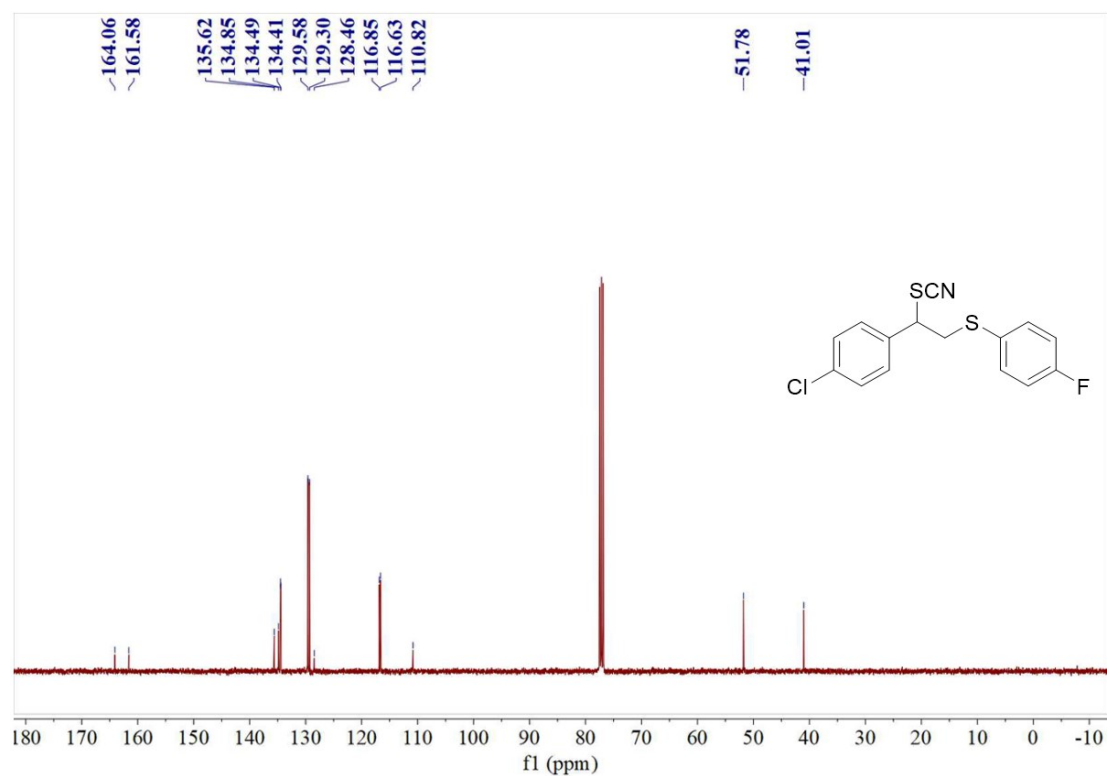


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ba

(2-(4-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ca)

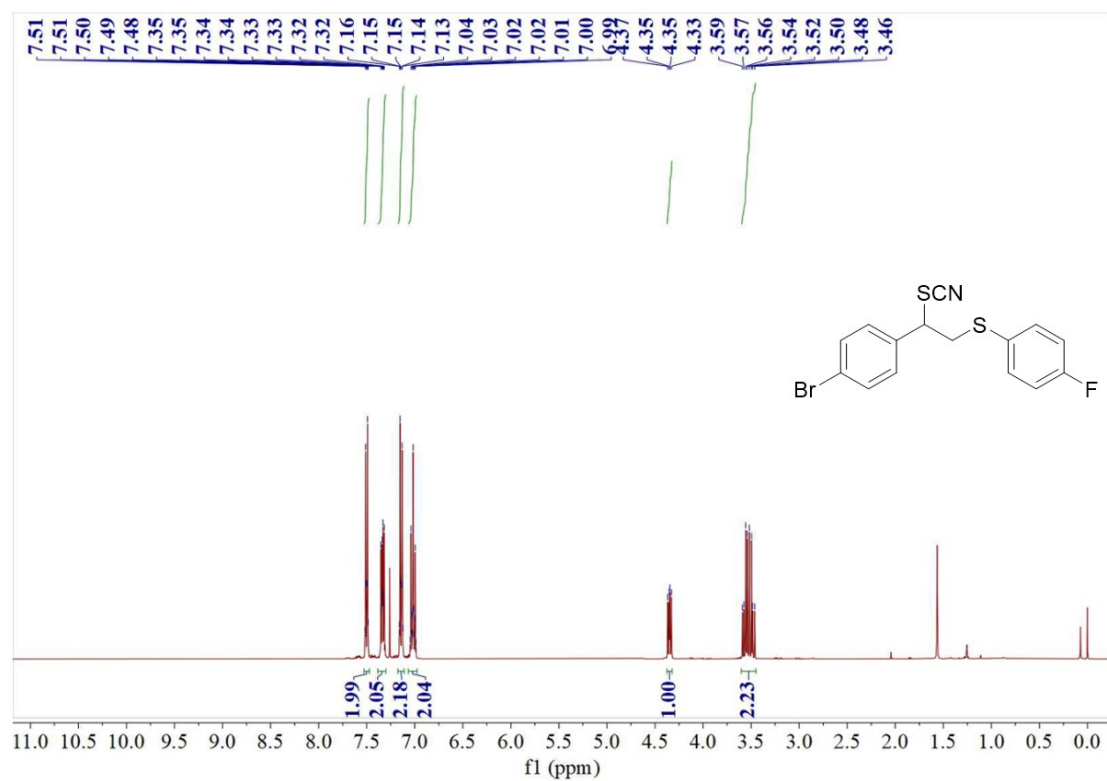


¹H NMR (400 MHz, CDCl₃) spectrum of 4ca

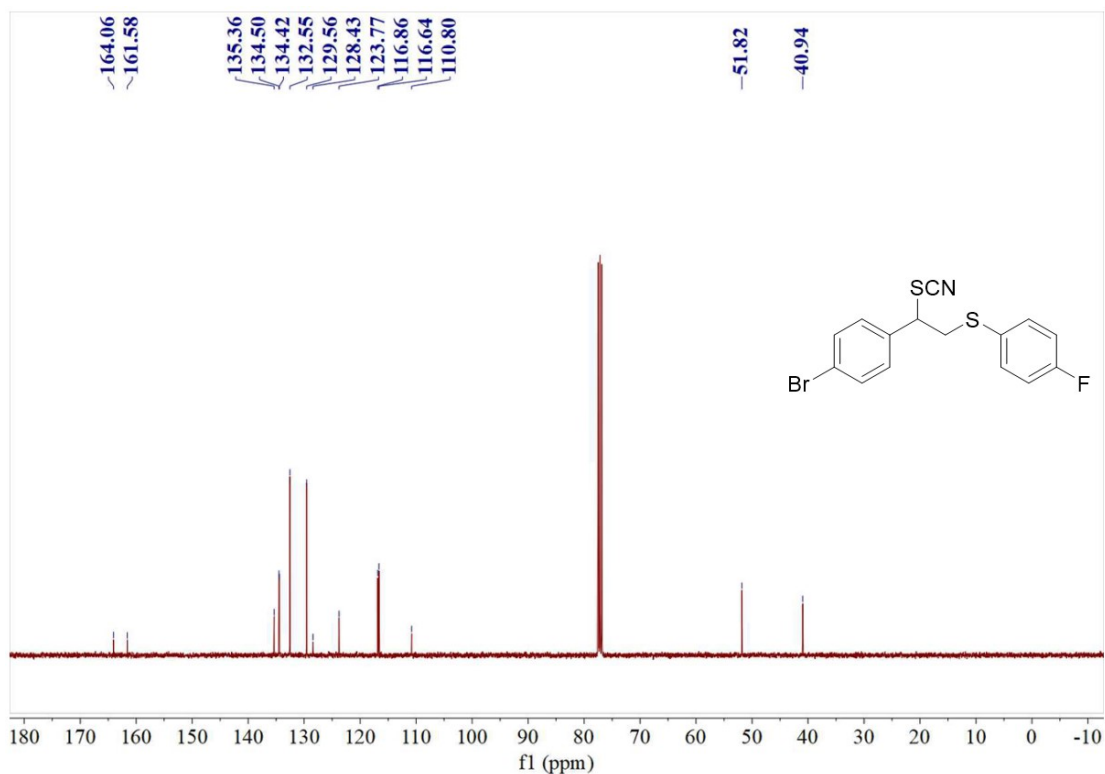


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ca

(2-(4-bromophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4da)

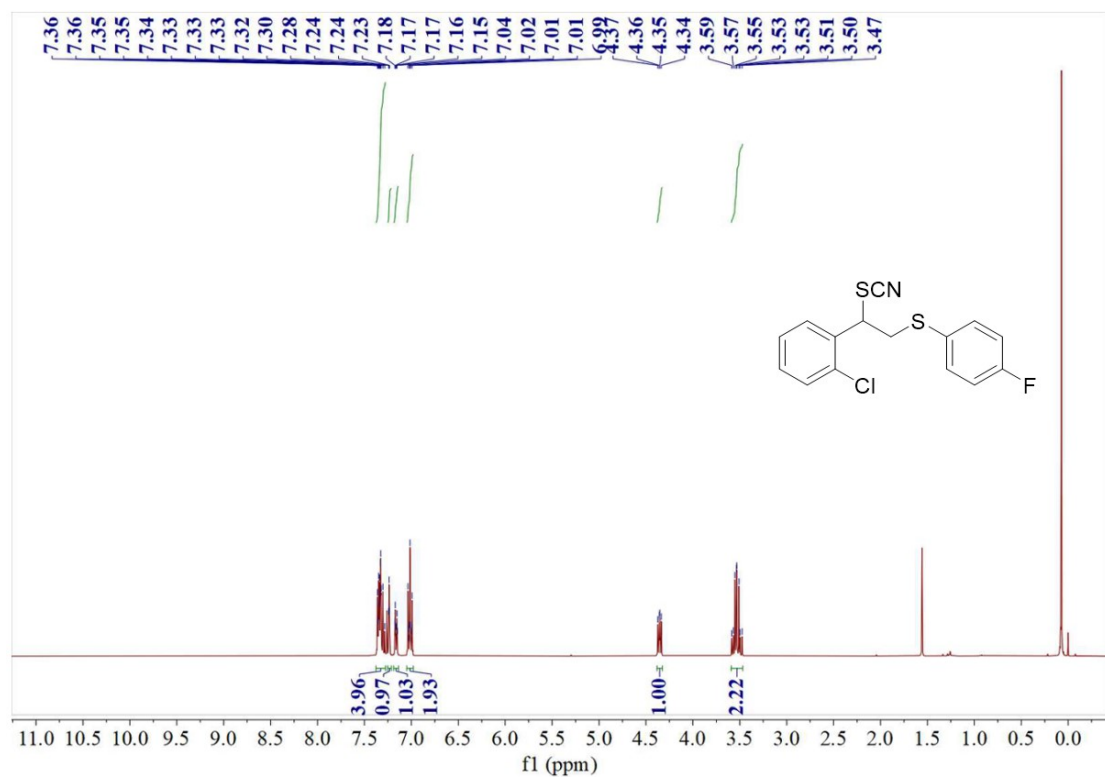


¹H NMR (400 MHz, CDCl₃) spectrum of 4da

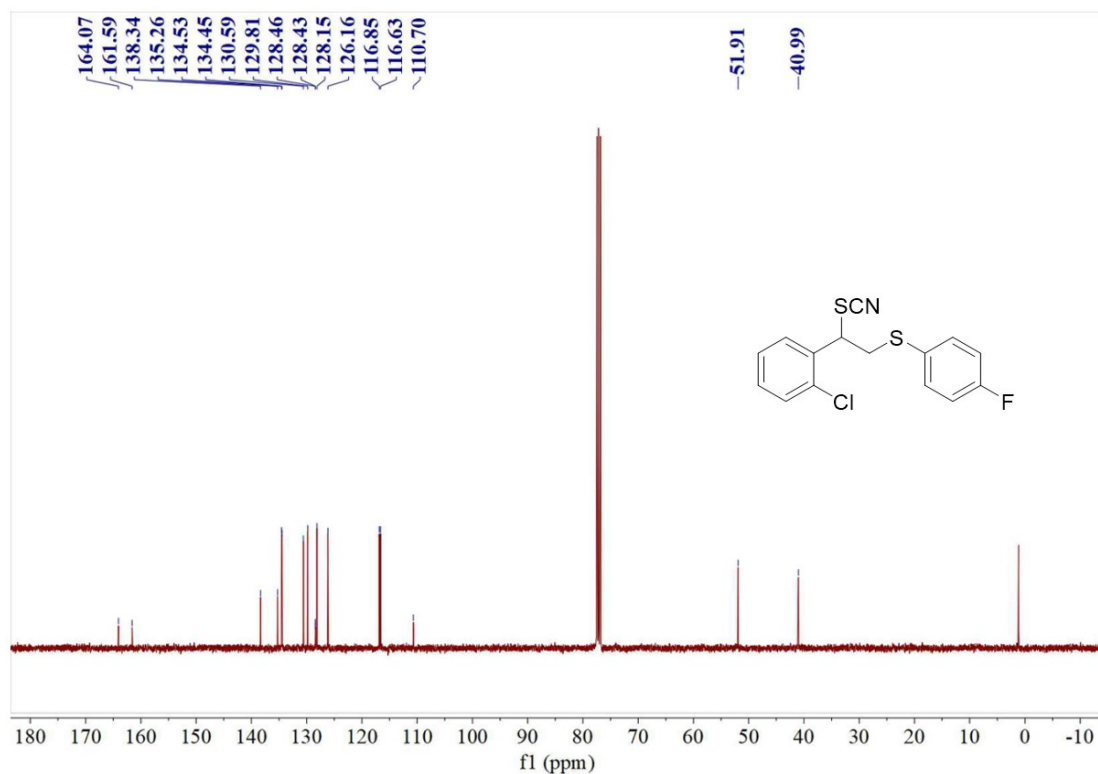


¹³C NMR (100 MHz, CDCl₃) spectrum of 4da

(2-(2-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ea)

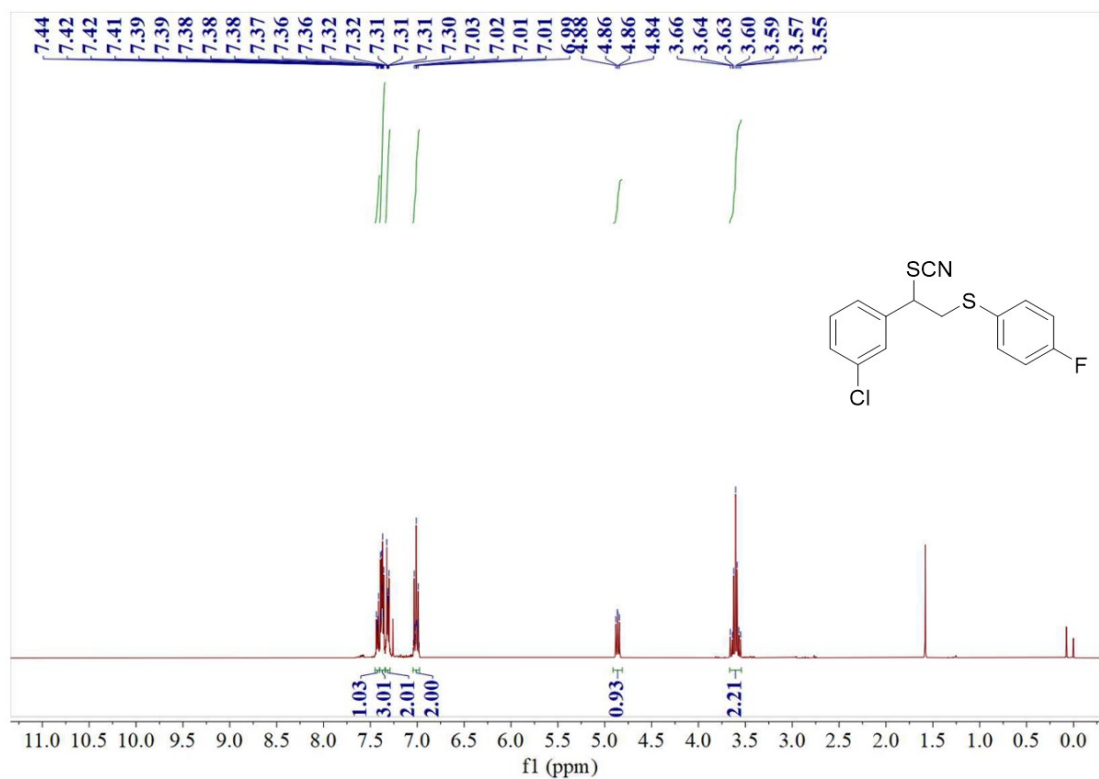


¹H NMR (400 MHz, CDCl₃) spectrum of 4ea

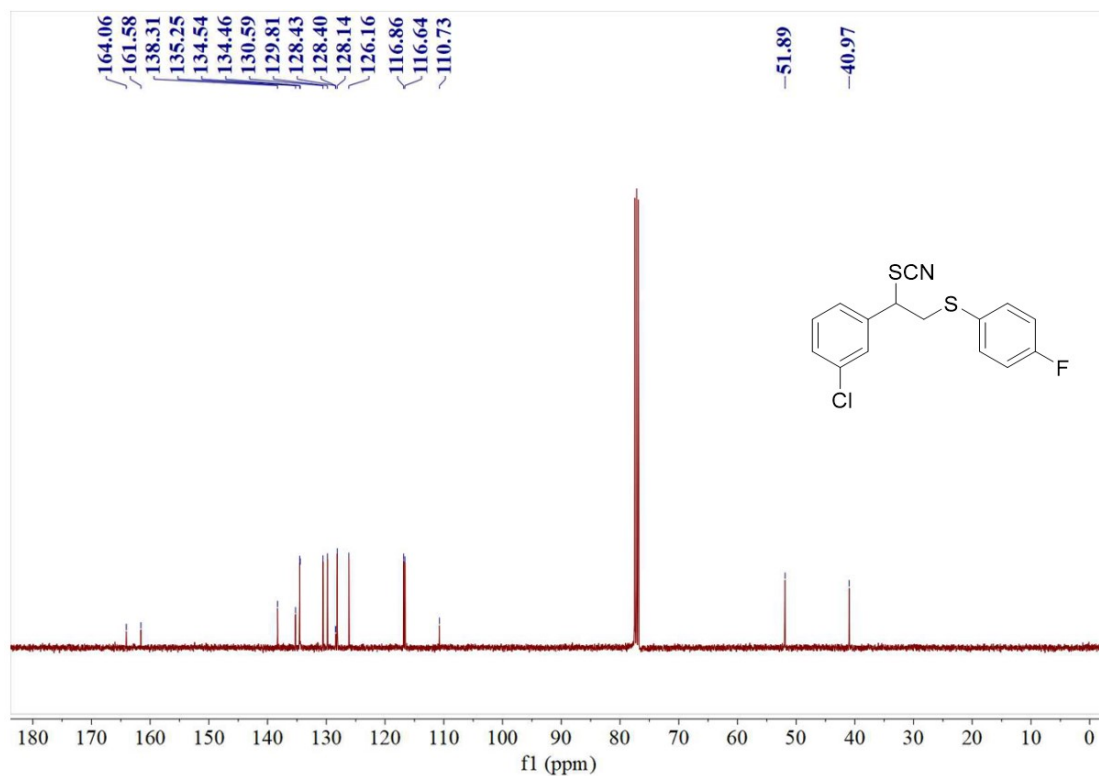


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ea

(2-(3-chlorophenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4fa)

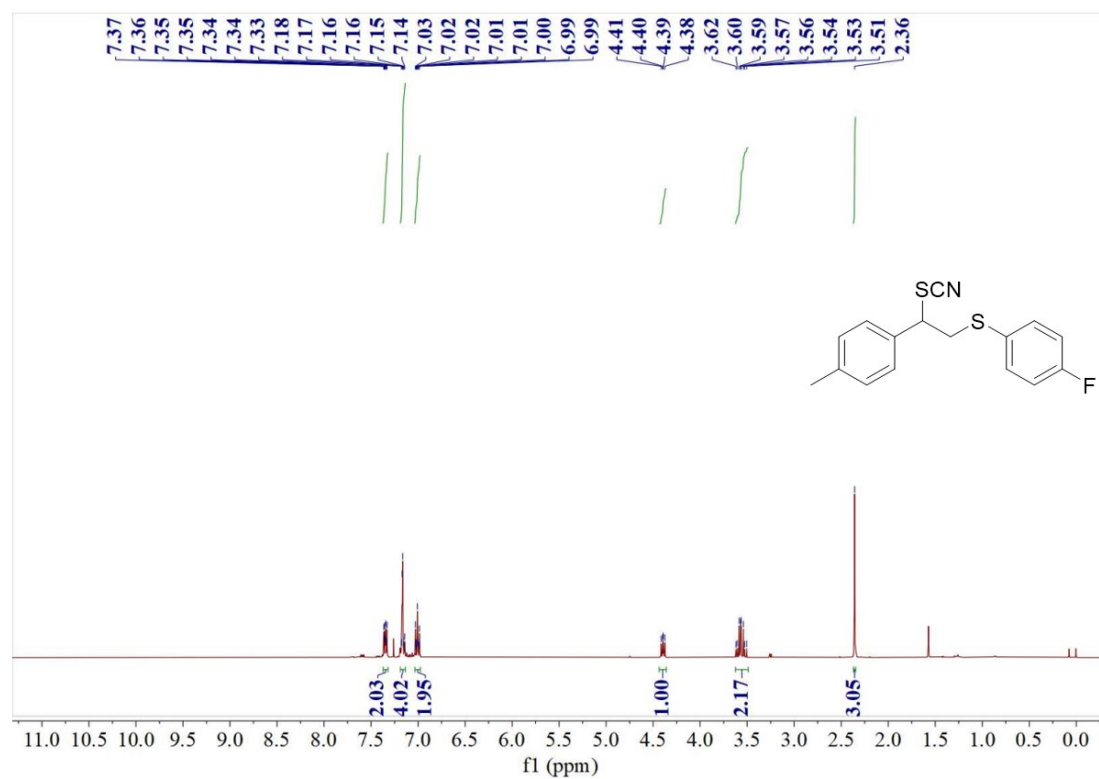


¹H NMR (400 MHz, CDCl₃) spectrum of 4fa

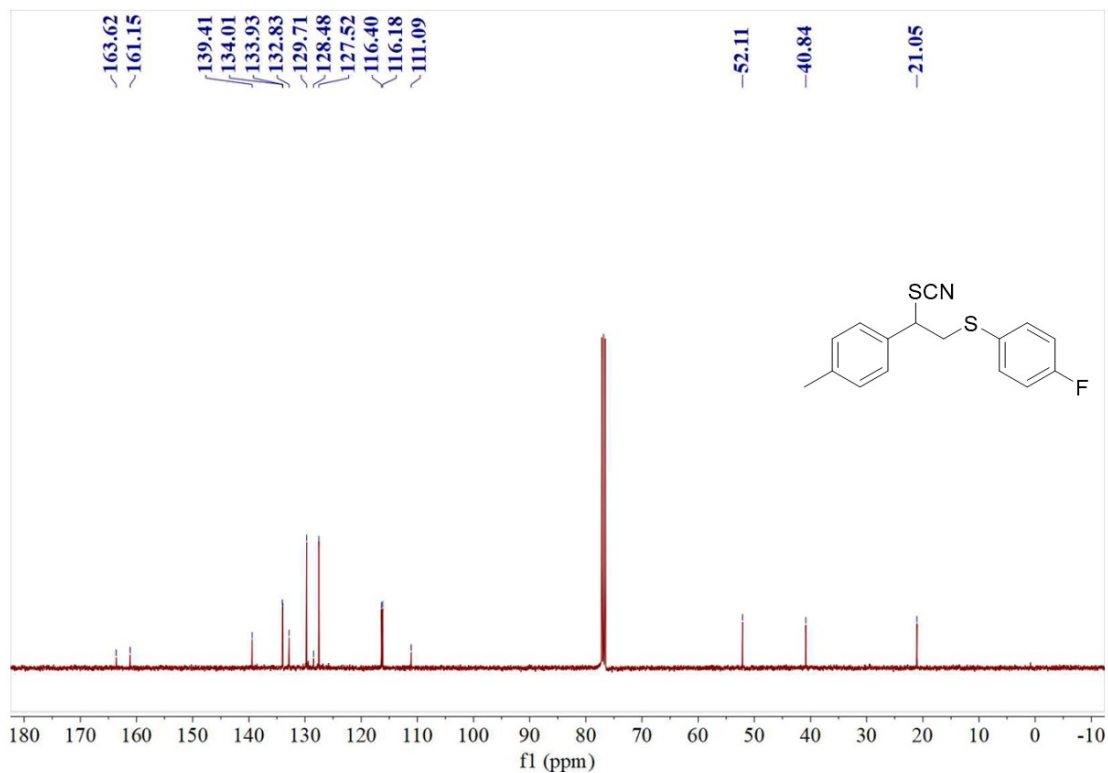


¹³C NMR (100 MHz, CDCl₃) spectrum of 4fa

(4-fluorophenyl)(2-thiocyanato-2-(p-tolyl)ethyl)sulfane (4ga)

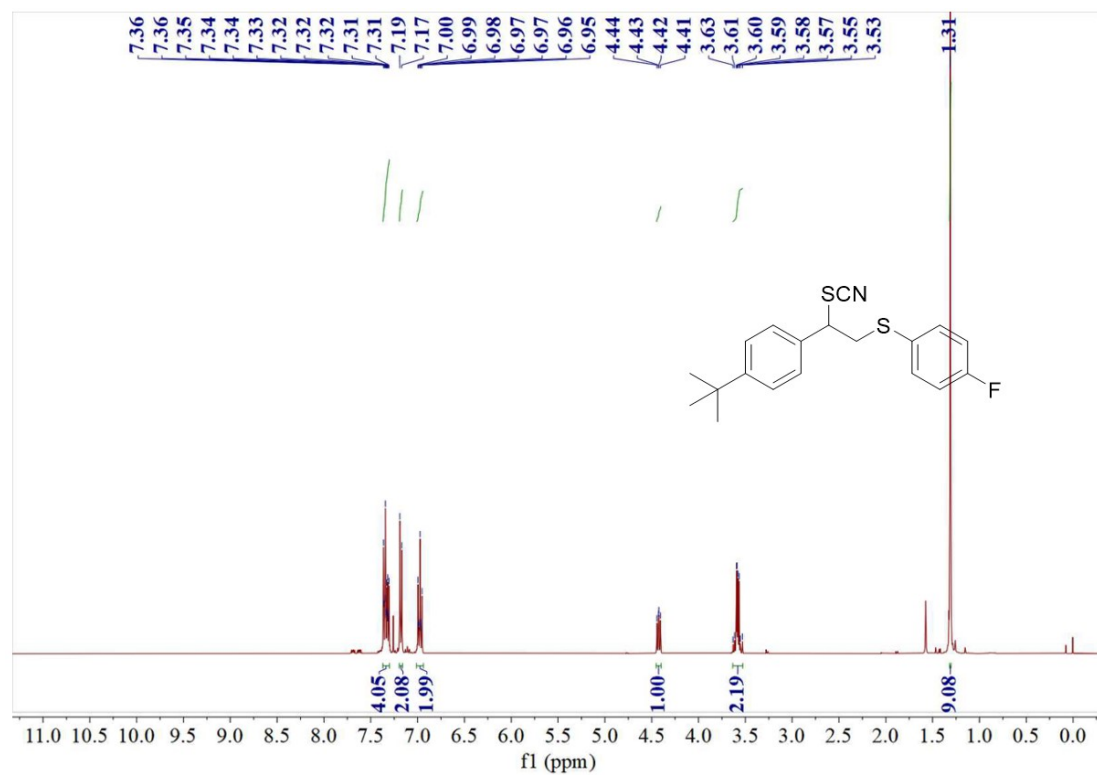


¹H NMR (400 MHz, CDCl₃) spectrum of 4ga

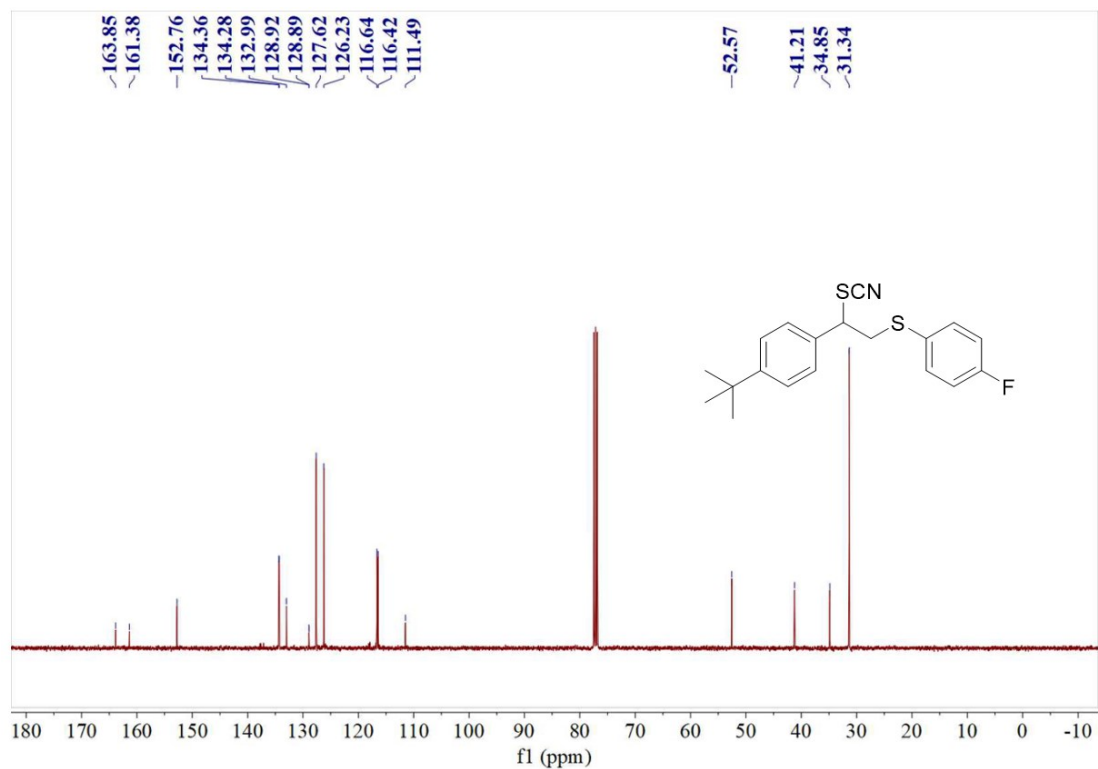


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ga

(2-(4-(tert-butyl)phenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ha)

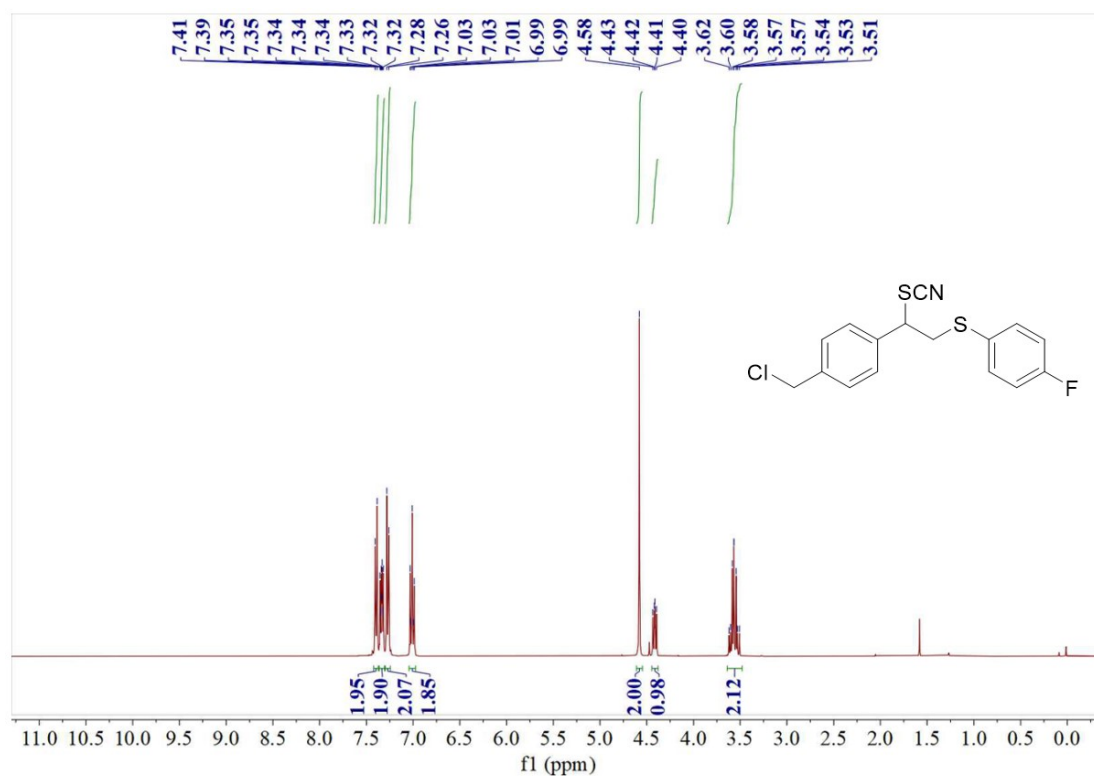


¹H NMR (400 MHz, CDCl₃) spectrum of 4ha

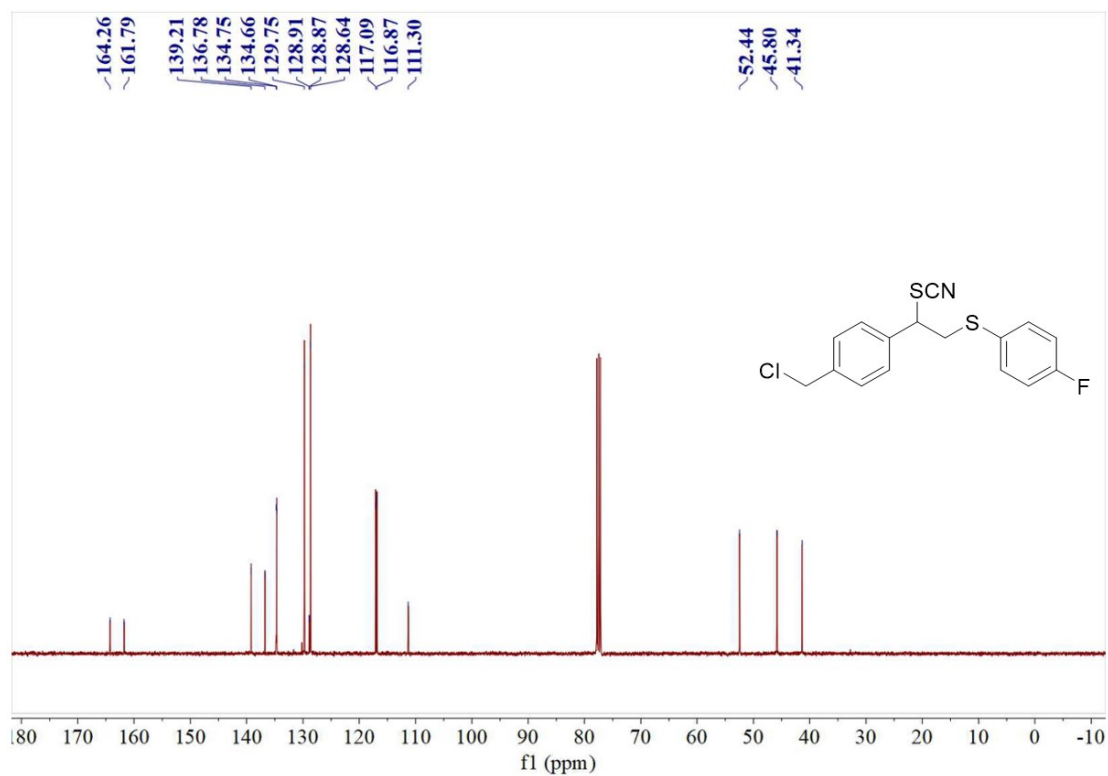


¹³C NMR (100 MHz, CDCl₃) spectrum of 4a

(2-(4-(chloromethyl)phenyl)-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4ia)

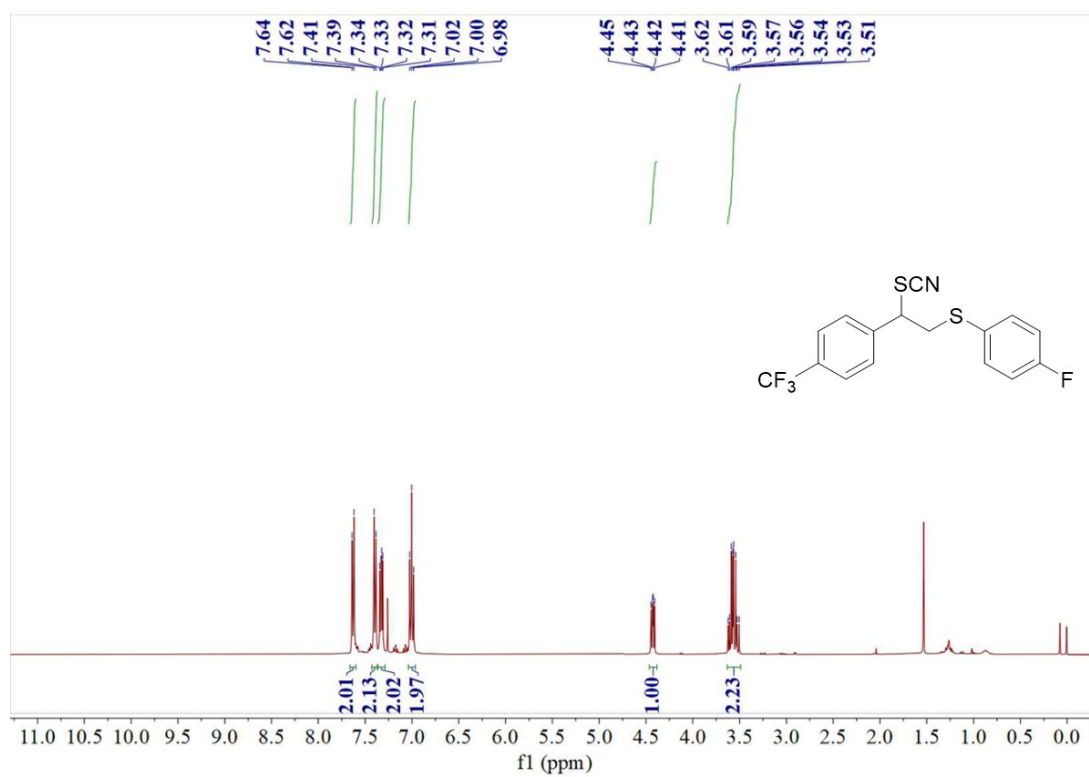


¹H NMR (400 MHz, CDCl₃) spectrum of 4ia

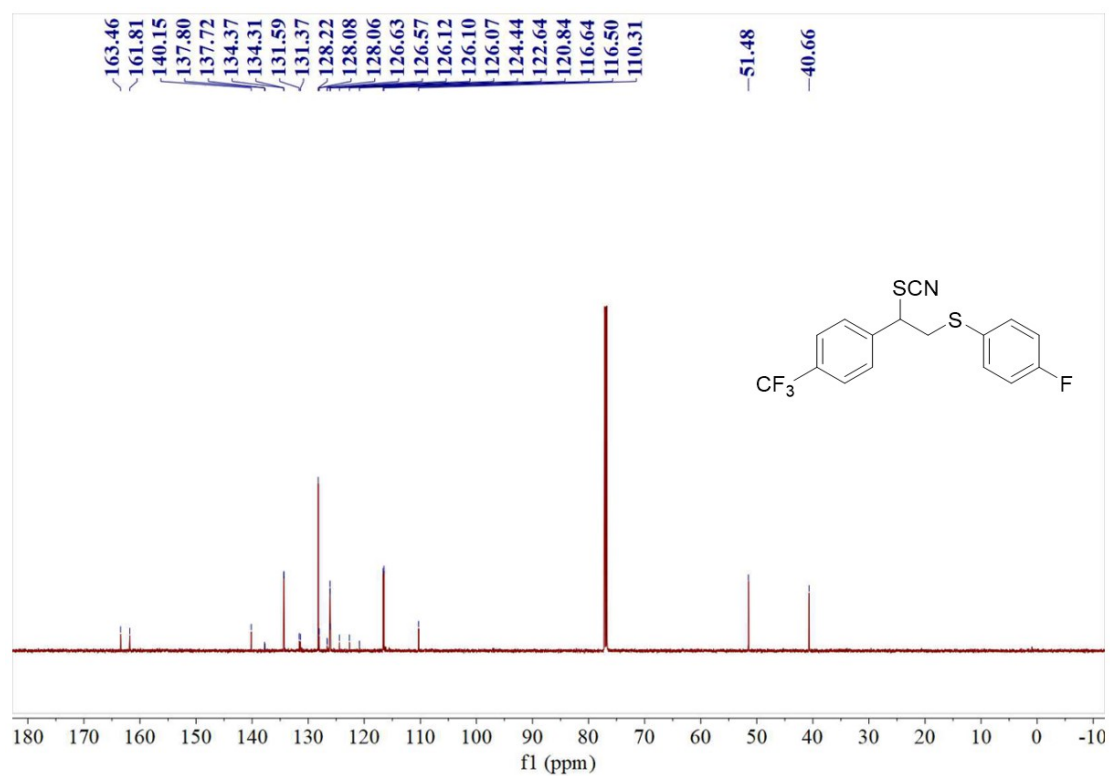


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ia

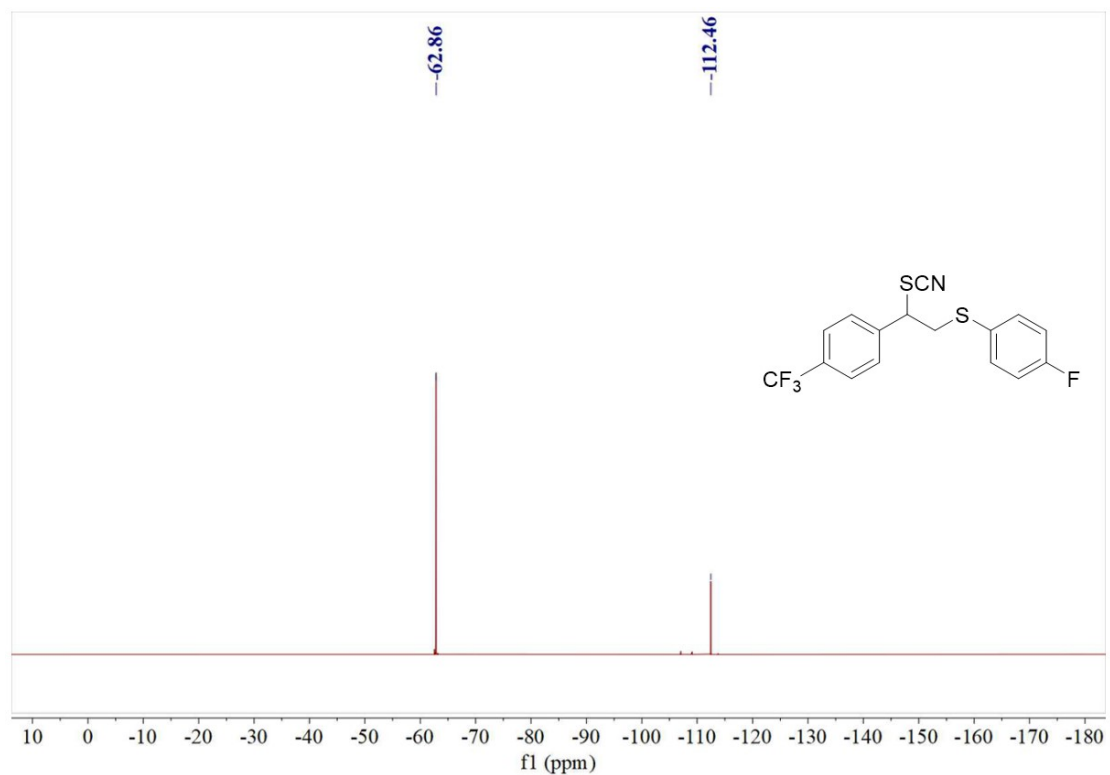
(4-fluorophenyl)(2-thiocyanato-2-(4-(trifluoromethyl)phenyl)ethyl)sulfane (4ja)



¹H NMR (400 MHz, CDCl₃) spectrum of 4ja

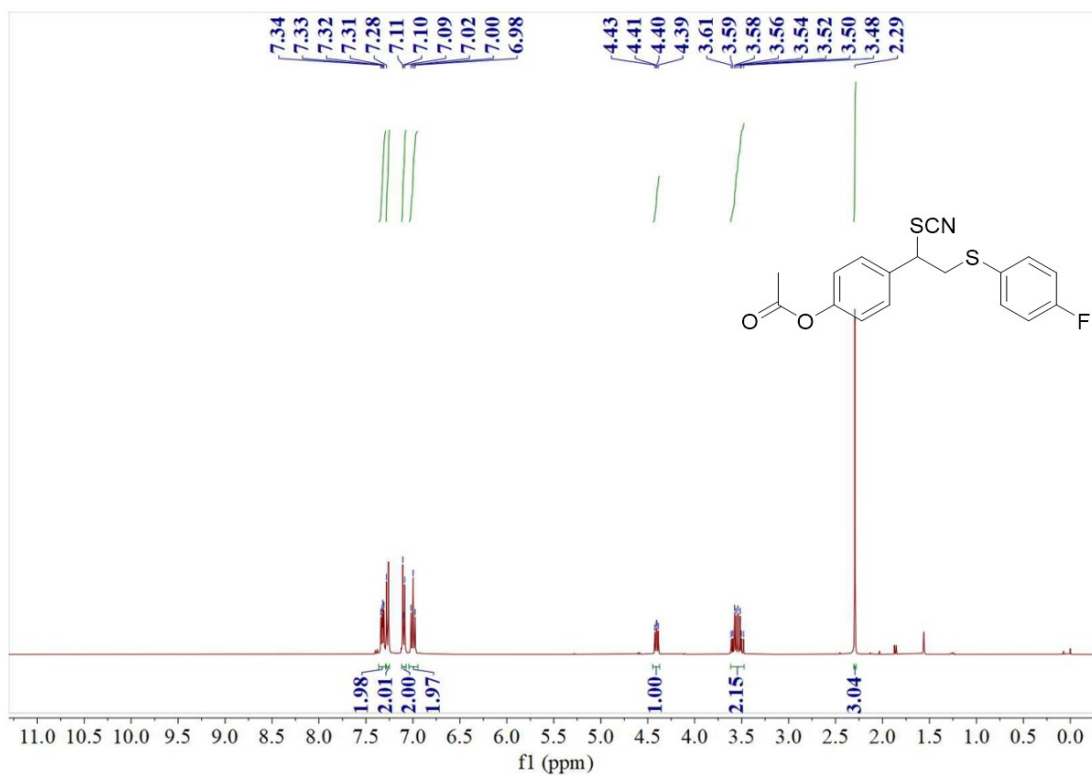


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ja

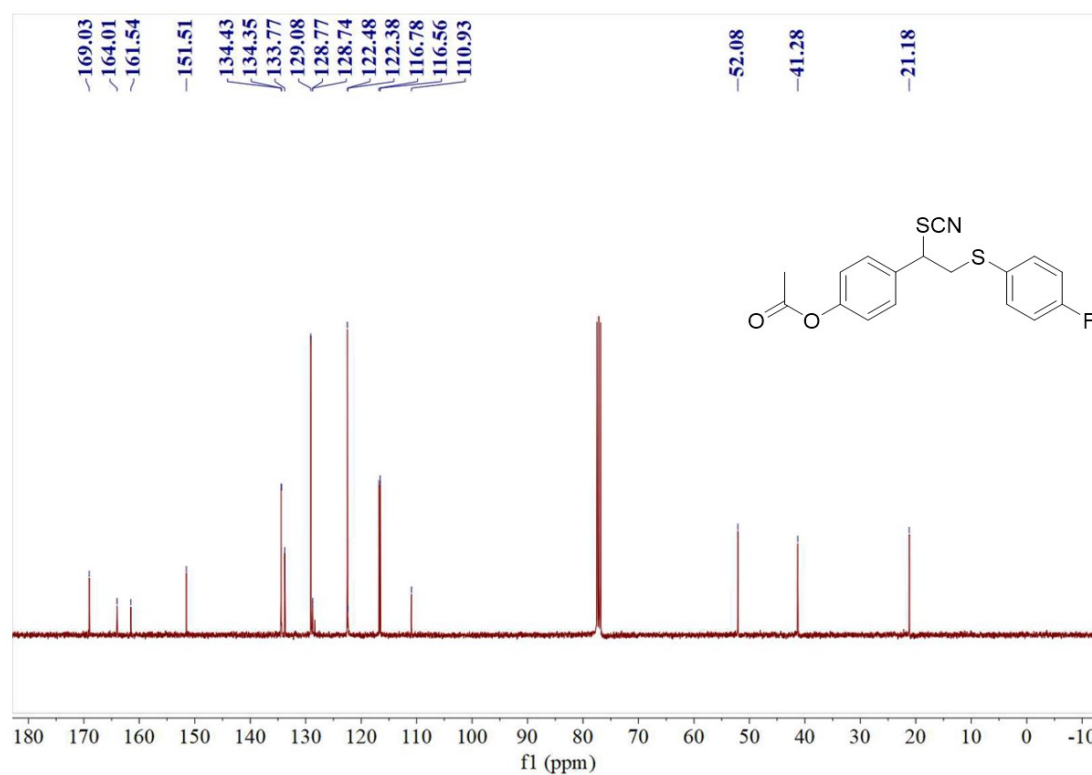


¹⁹F NMR (375 MHz, CDCl₃) spectrum of 4ja

(2-((4-fluorophenyl)thio)-1-thiocyanatoethyl)phenyl acetate (4ka)

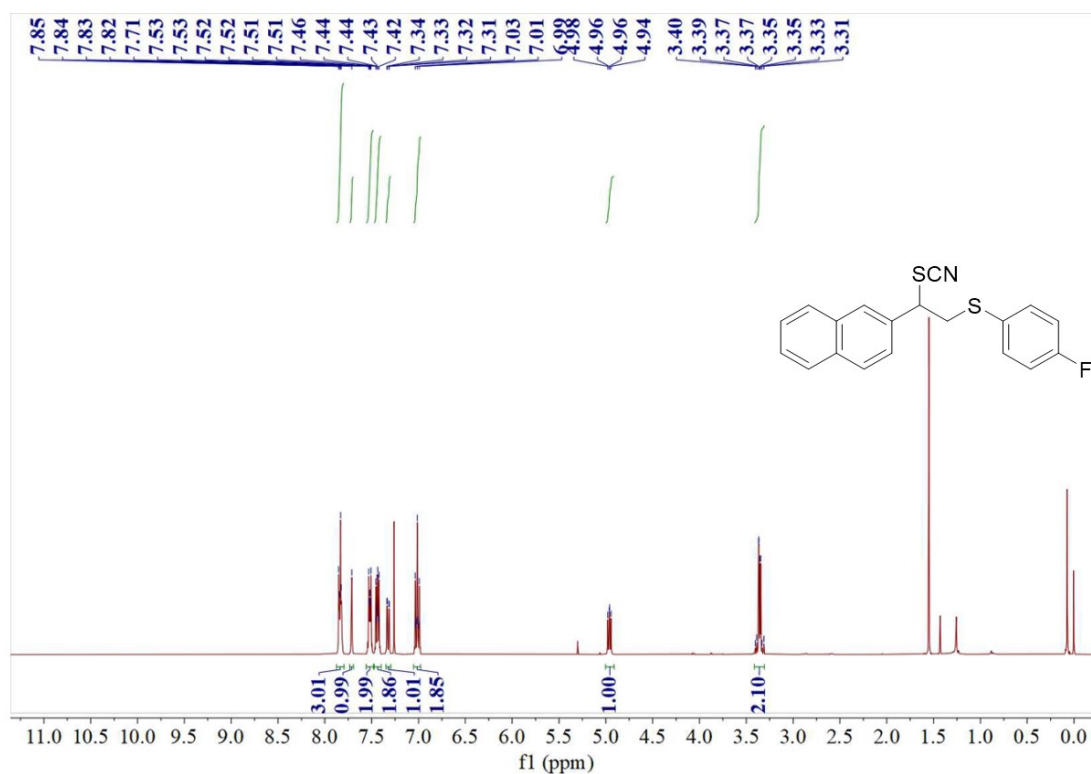


¹H NMR (400 MHz, CDCl₃) spectrum of 4ka

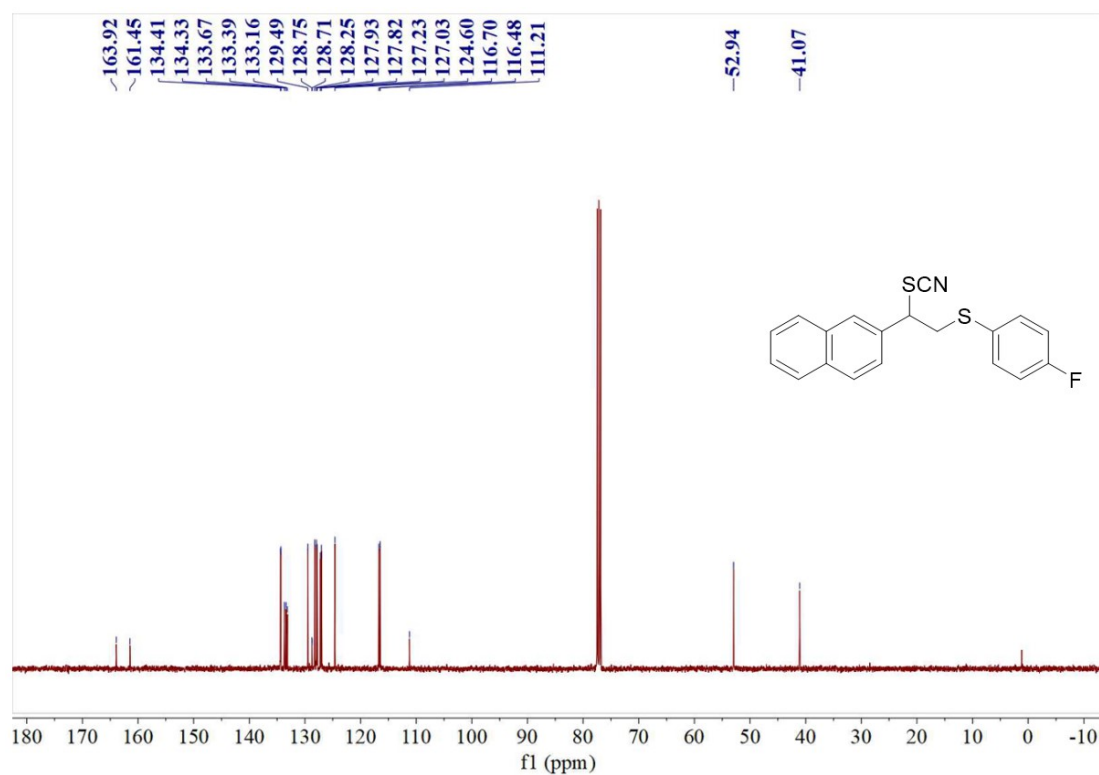


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ka

(4-fluorophenyl)(2-(naphthalen-2-yl)-2-thiocyanatoethyl)sulfane (4la)

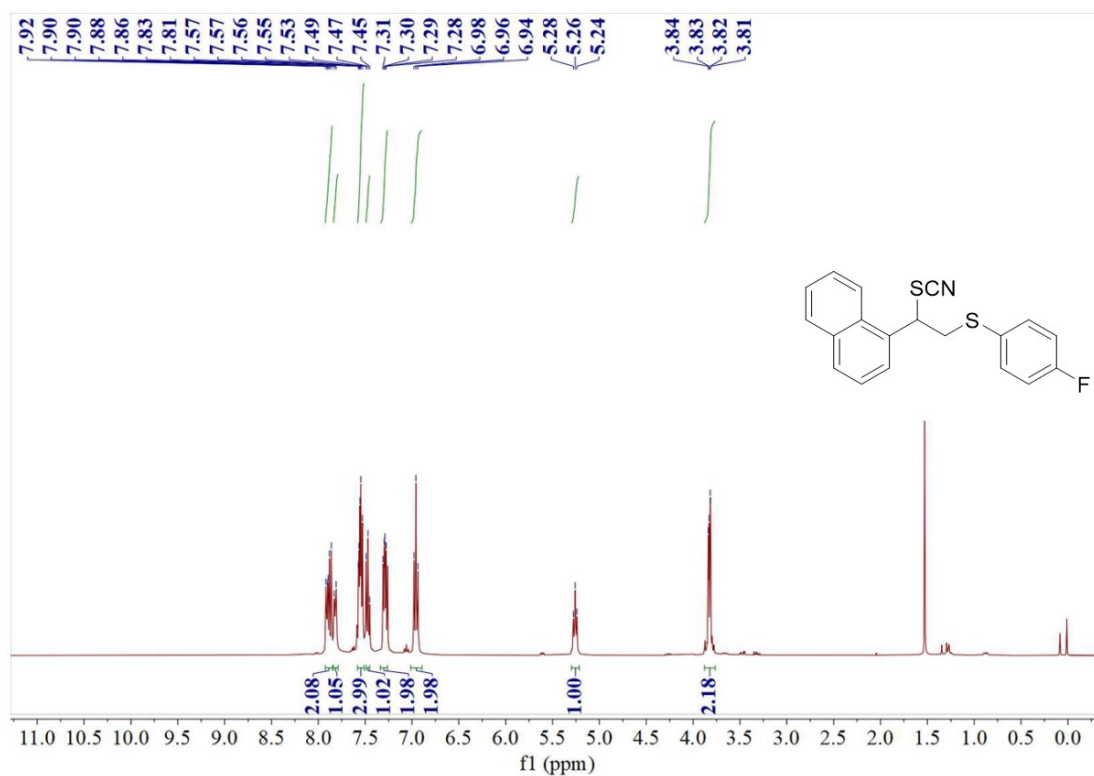


¹H NMR (400 MHz, CDCl₃) spectrum of 4a

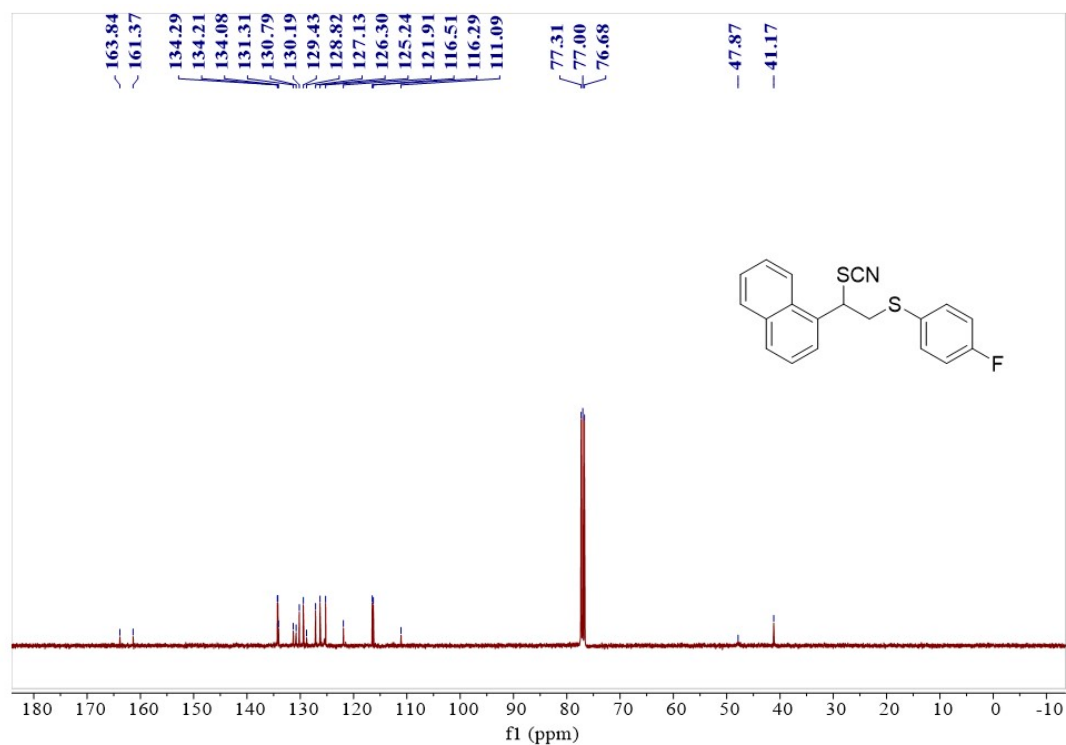


¹³C NMR (100 MHz, CDCl₃) spectrum of 4a

(4-fluorophenyl)(2-(naphthalen-1-yl)-2-thiocyanatoethyl)sulfane (4a)

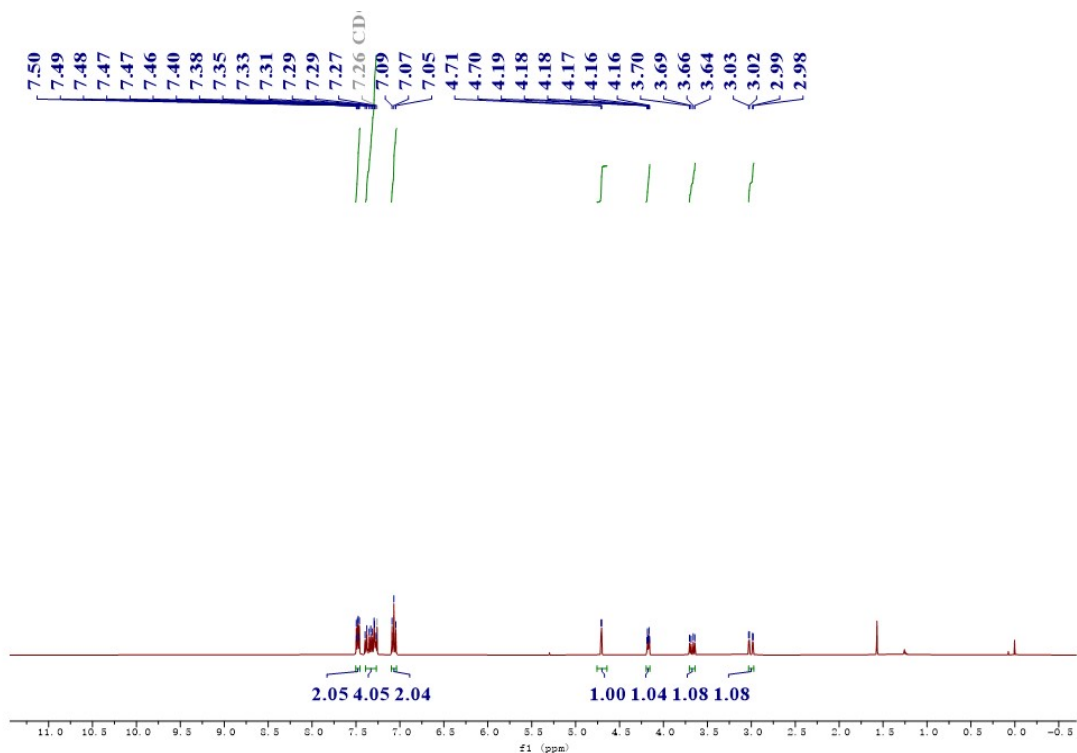


¹H NMR (400 MHz, CDCl₃) spectrum of 4ma

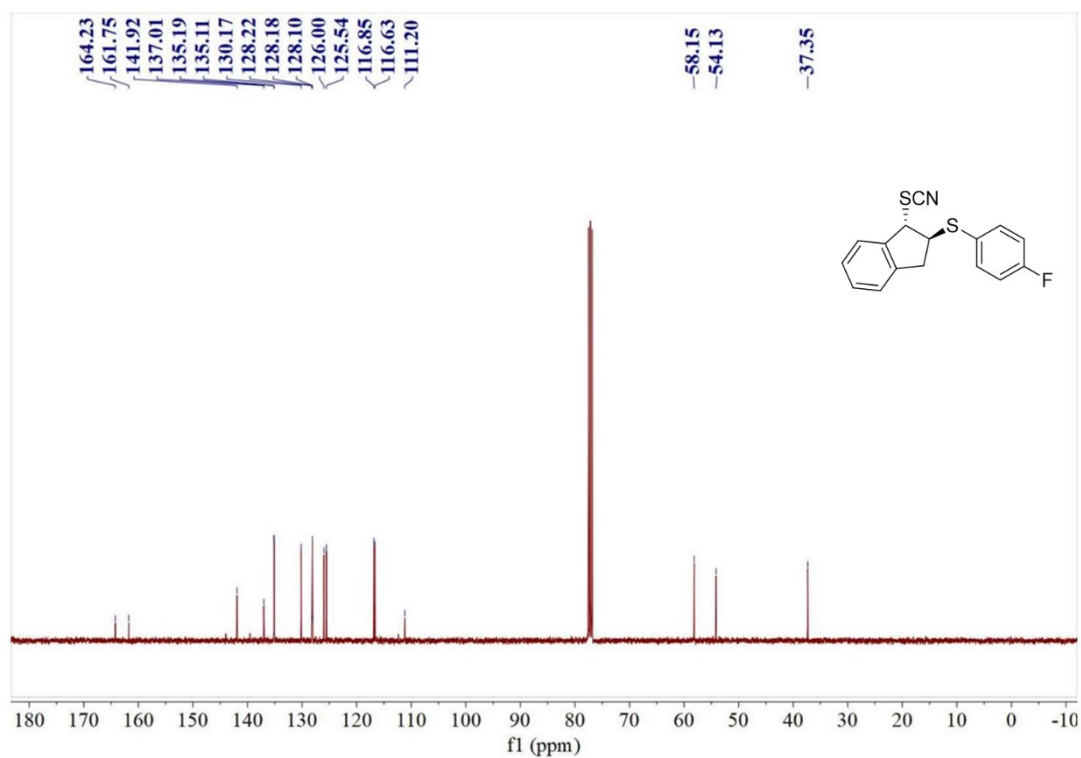


¹³C NMR (100 MHz, CDCl₃) spectrum of 4ma

(4-fluorophenyl)(1-thiocyanato-2,3-dihydro-1H-inden-2-yl)sulfane (4na)

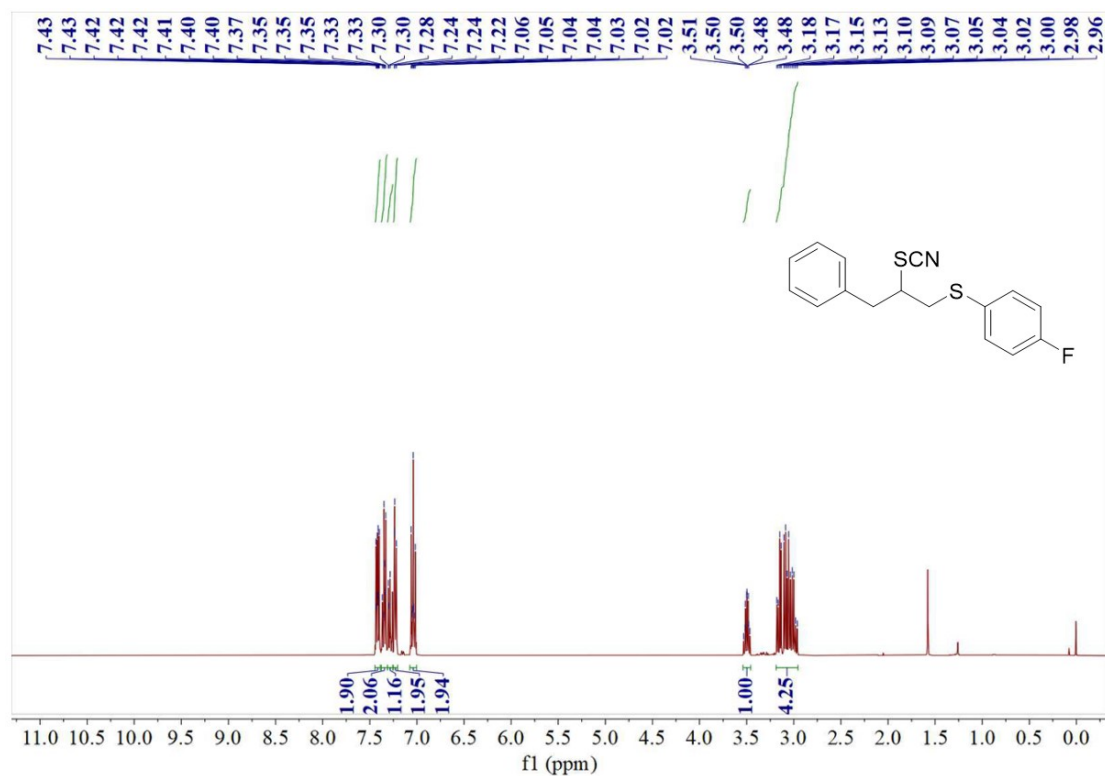


¹H NMR (400 MHz, CDCl₃) spectrum of 4na

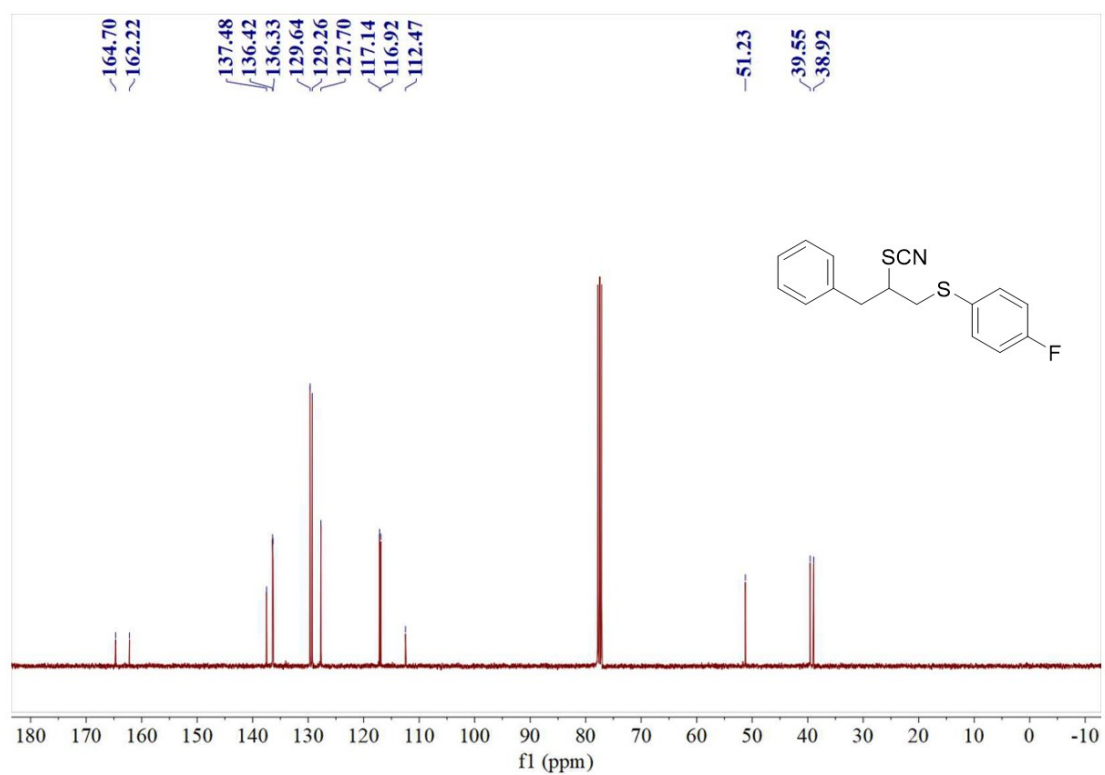


¹³C NMR (100 MHz, CDCl₃) spectrum of 4na

(4-fluorophenyl)(3-phenyl-2-thiocyanatopropyl)sulfane (40a)

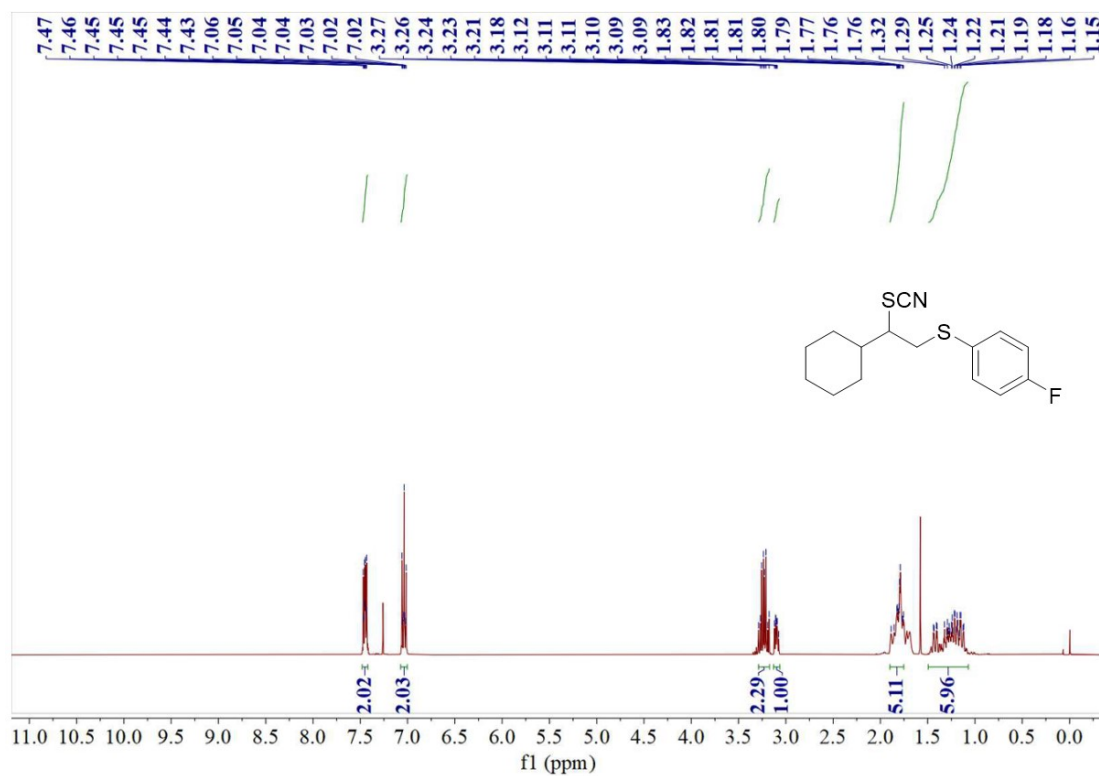


¹H NMR (400 MHz, CDCl₃) spectrum of 40a

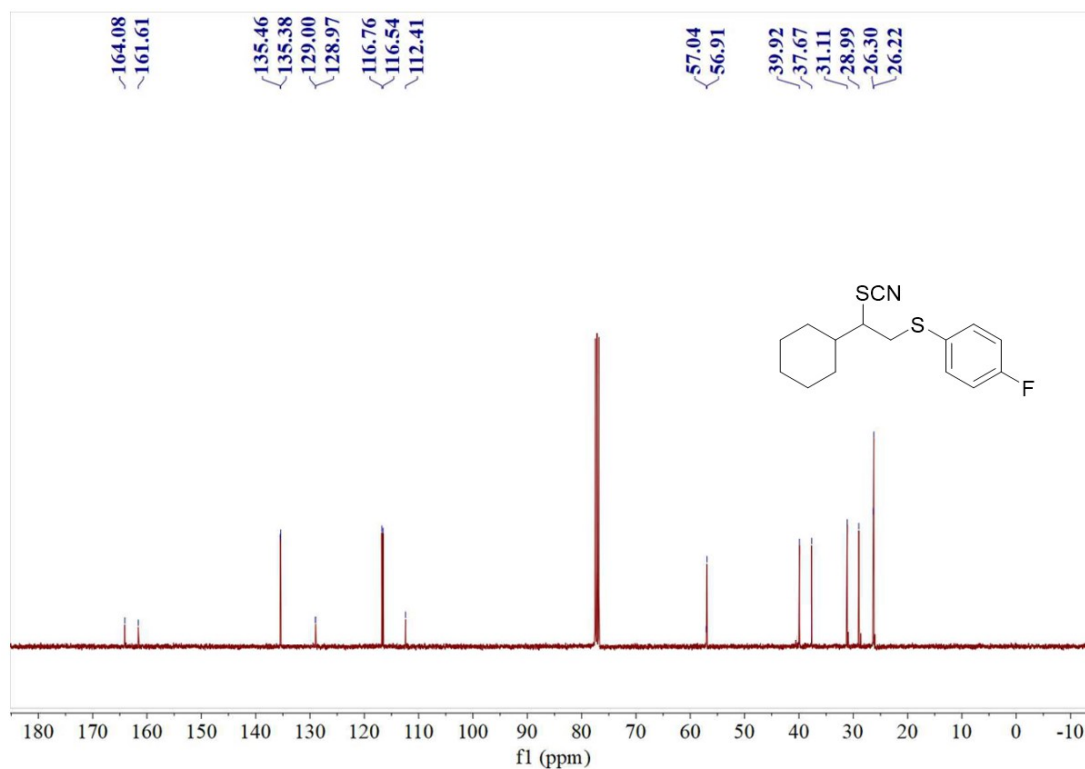


¹³C NMR (100 MHz, CDCl₃) spectrum of 40a

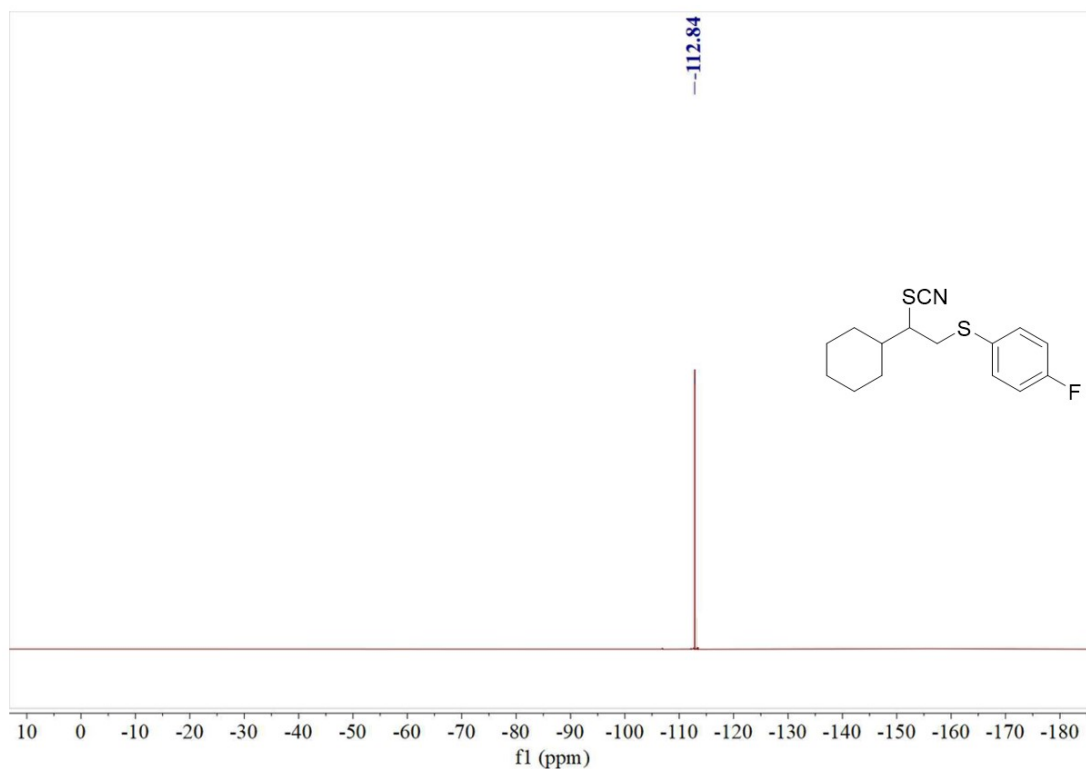
(2-cyclohexyl-2-thiocyanatoethyl)(4-fluorophenyl)sulfane (4pa)



¹H NMR (400 MHz, CDCl₃) spectrum of 4pa

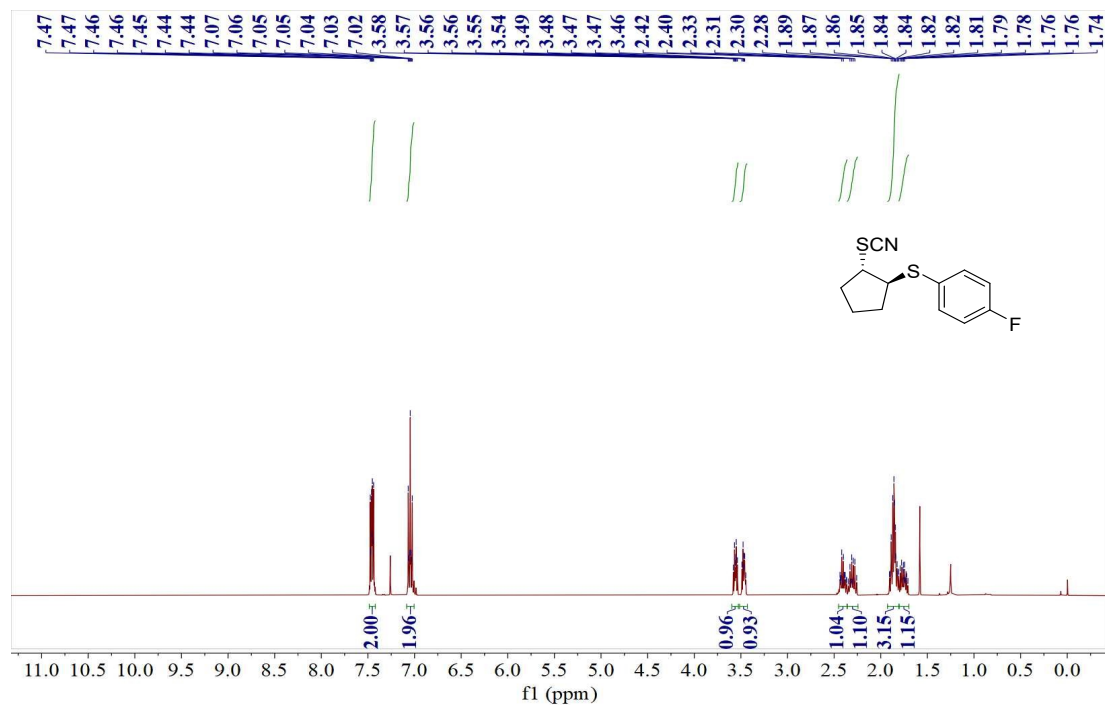


¹³C NMR (100 MHz, CDCl₃) spectrum of 4pa

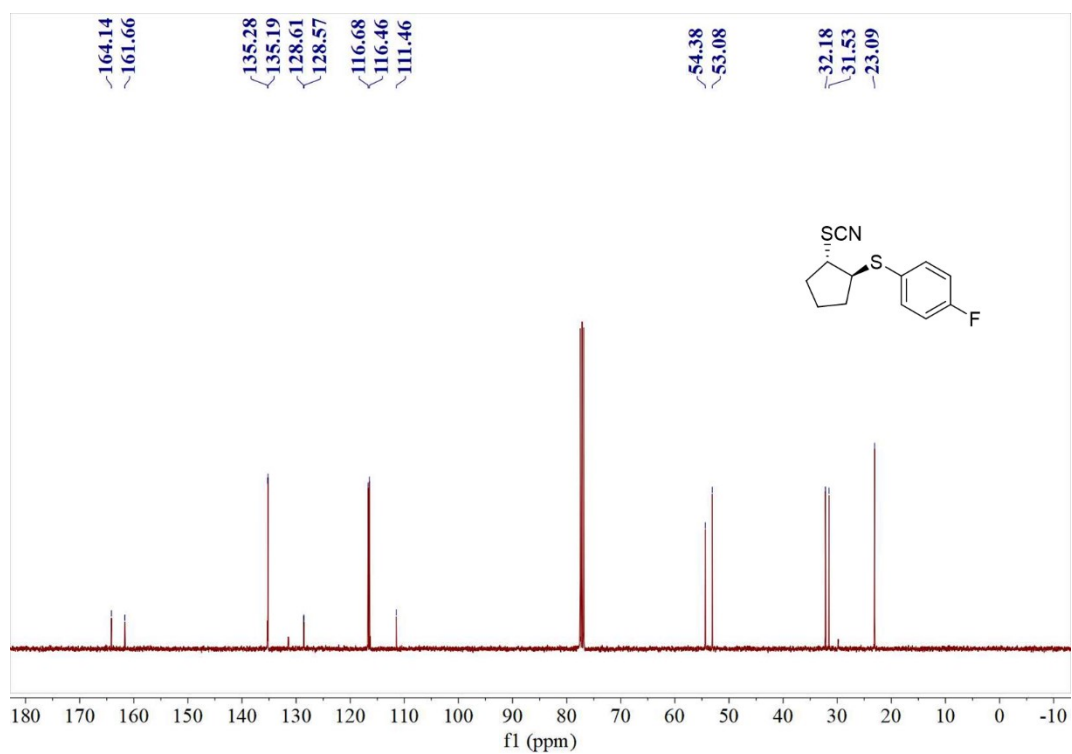


¹⁹F NMR (375 MHz, CDCl₃) spectrum of 4pa

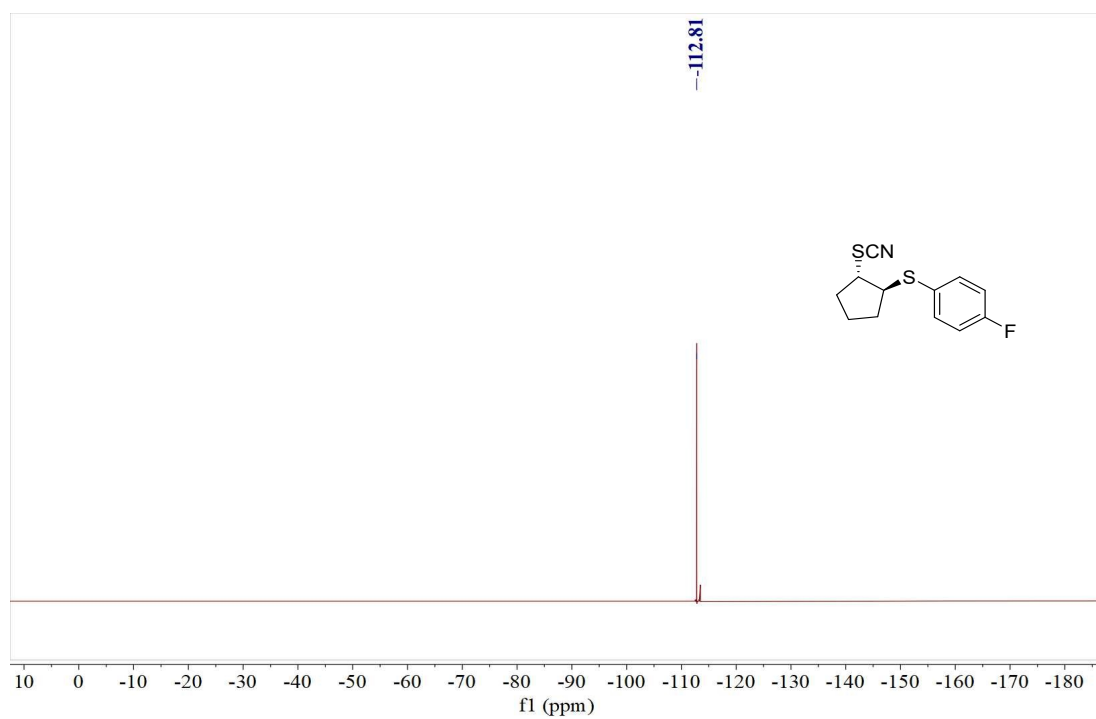
(4-fluorophenyl)(2-thiocyanatocyclopentyl)sulfane (4qa)



¹H NMR (400 MHz, CDCl₃) spectrum of 4qa

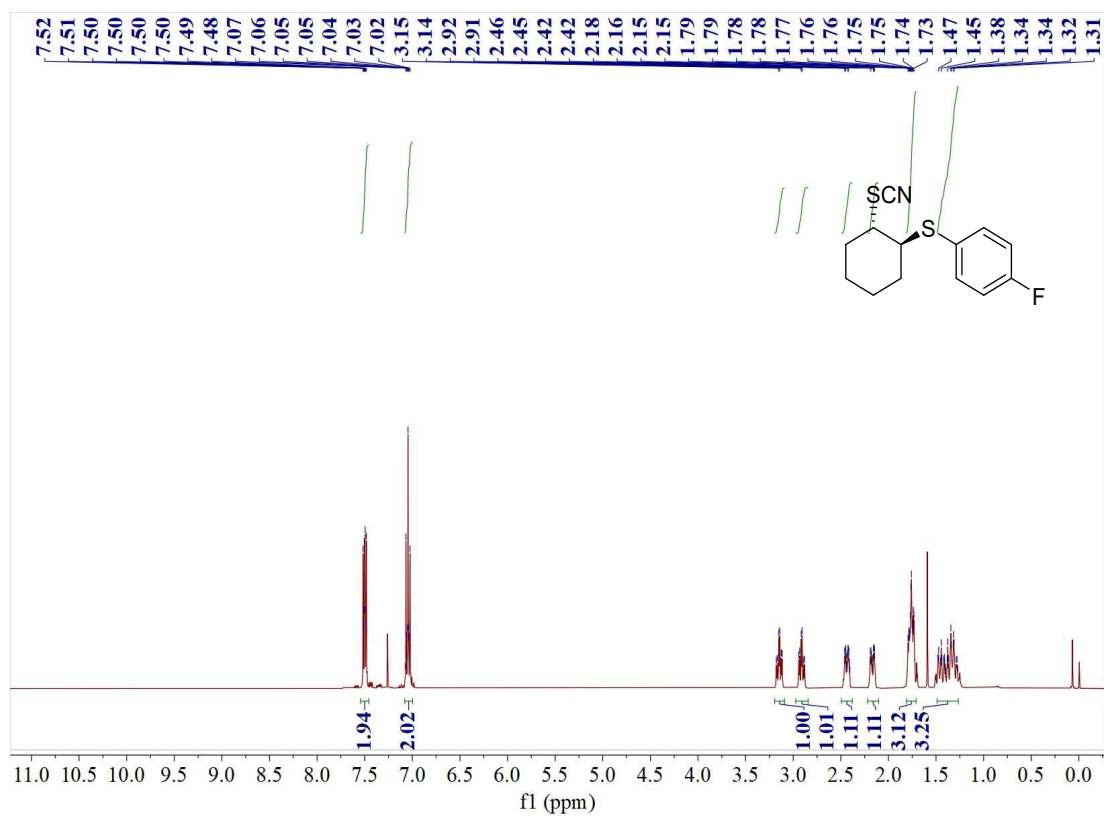


¹³C NMR (100 MHz, CDCl₃) spectrum of 4qa

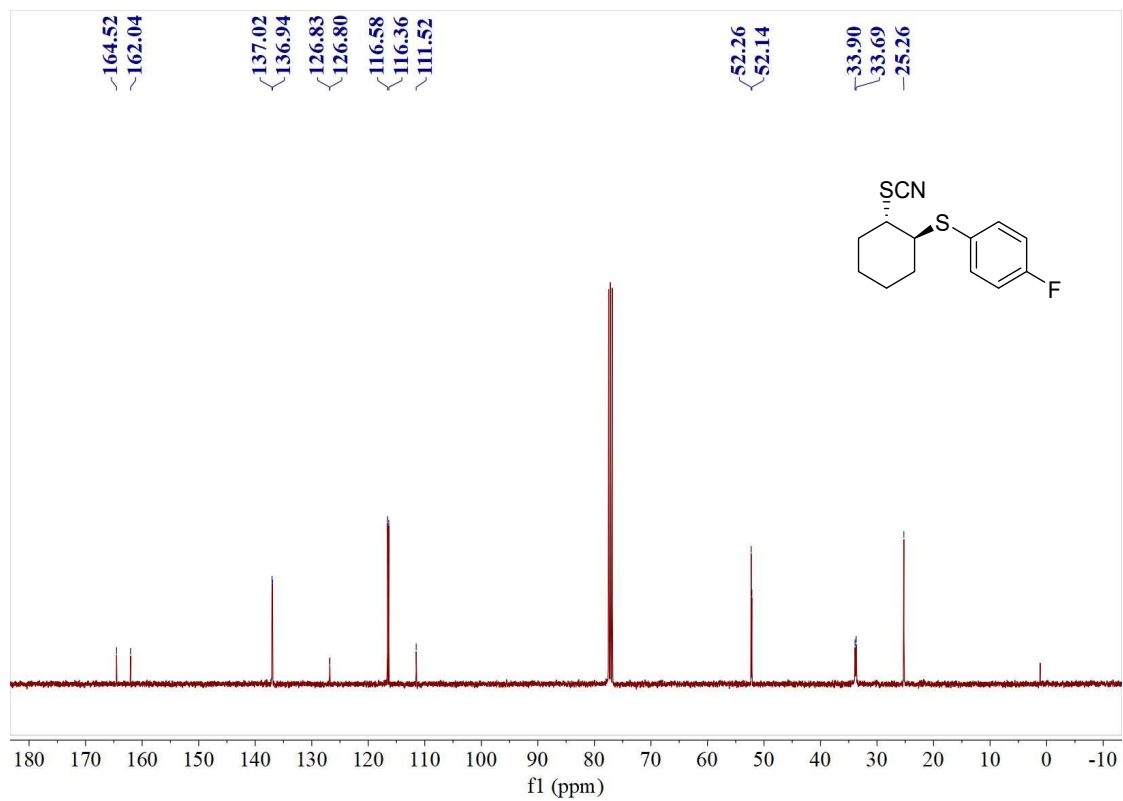


¹⁹F NMR (375 MHz, CDCl₃) spectrum of 4qa

(4-fluorophenyl)(2-thiocyanatocyclohexyl)sulfane (4ra)

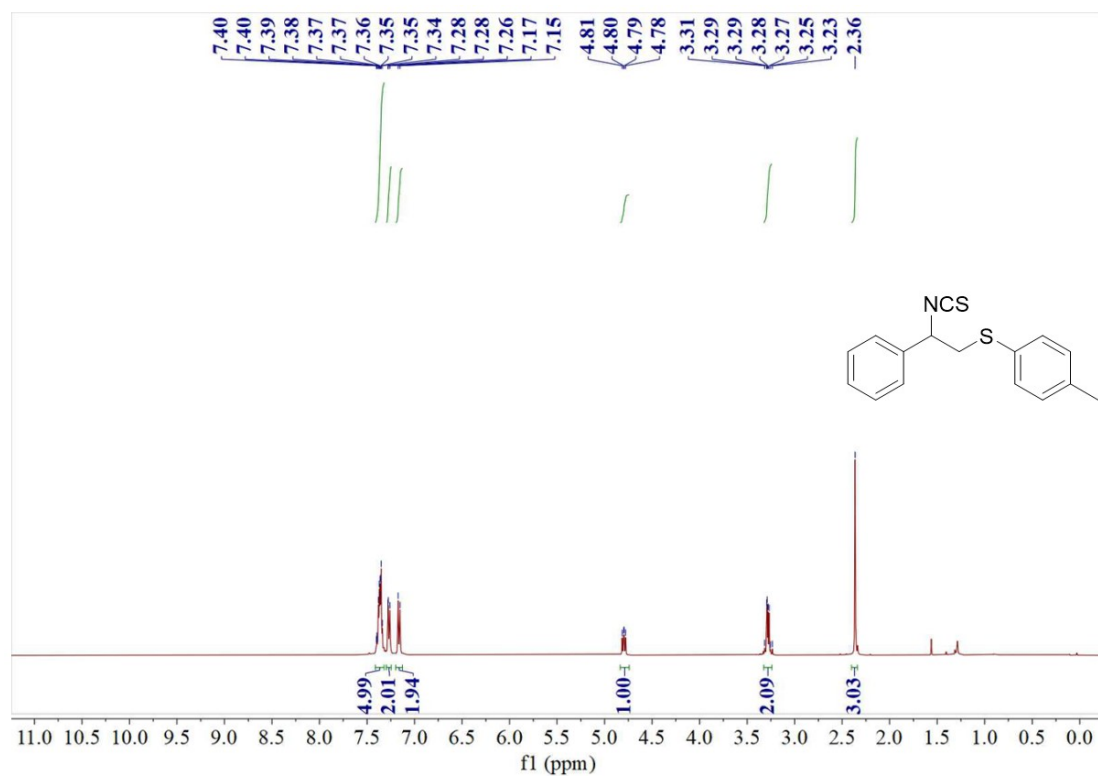


¹H NMR (400 MHz, CDCl₃) spectrum of 4ra



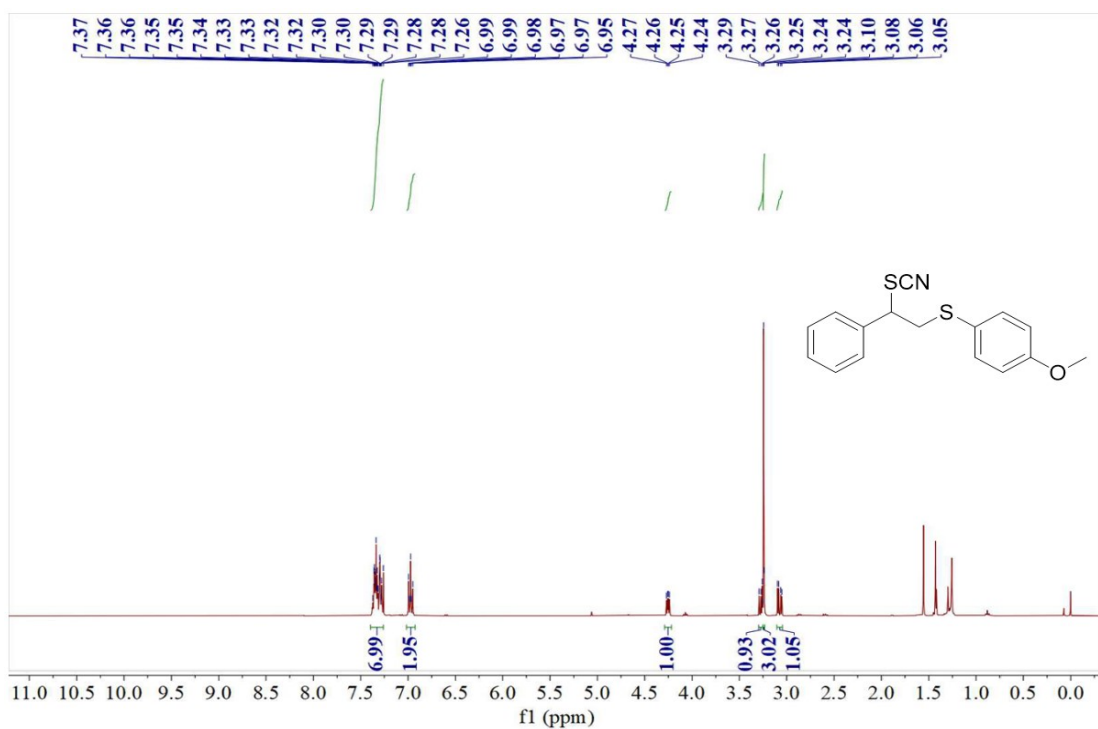
¹³C NMR (100 MHz, CDCl₃) spectrum of 4ra

(4-fluorophenyl)(2-isothiocyanato-2-phenylpropyl)sulfane (5)

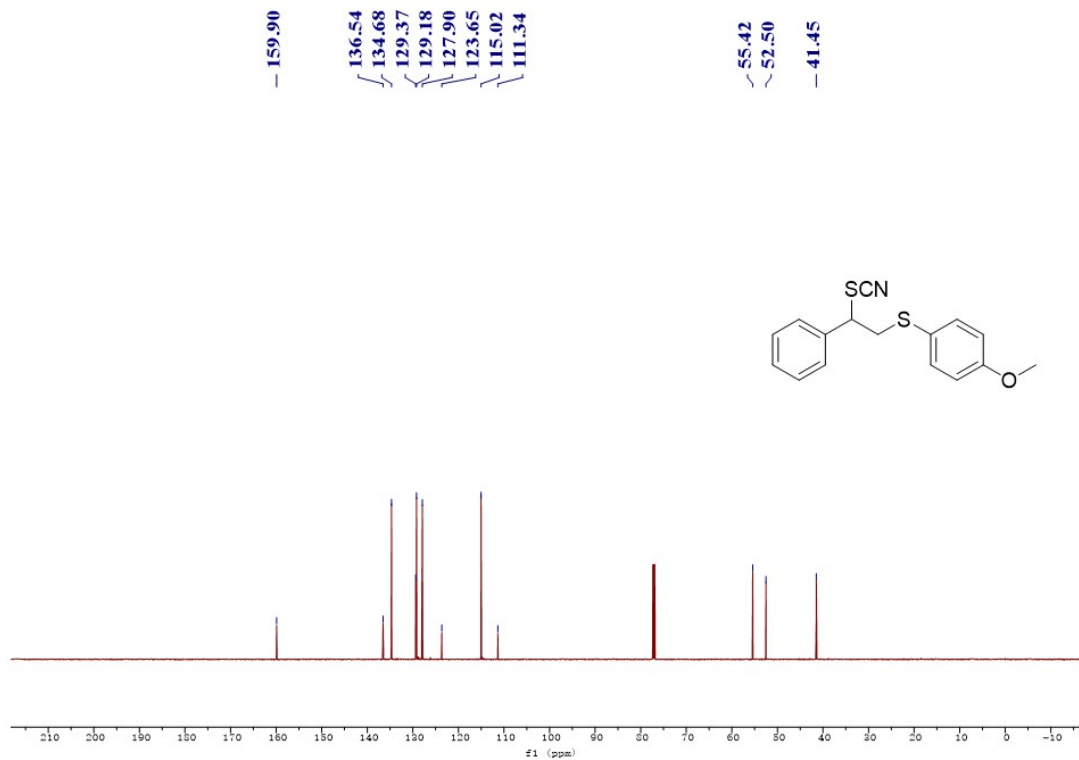


¹H NMR (400 MHz, CDCl₃) spectrum of 5

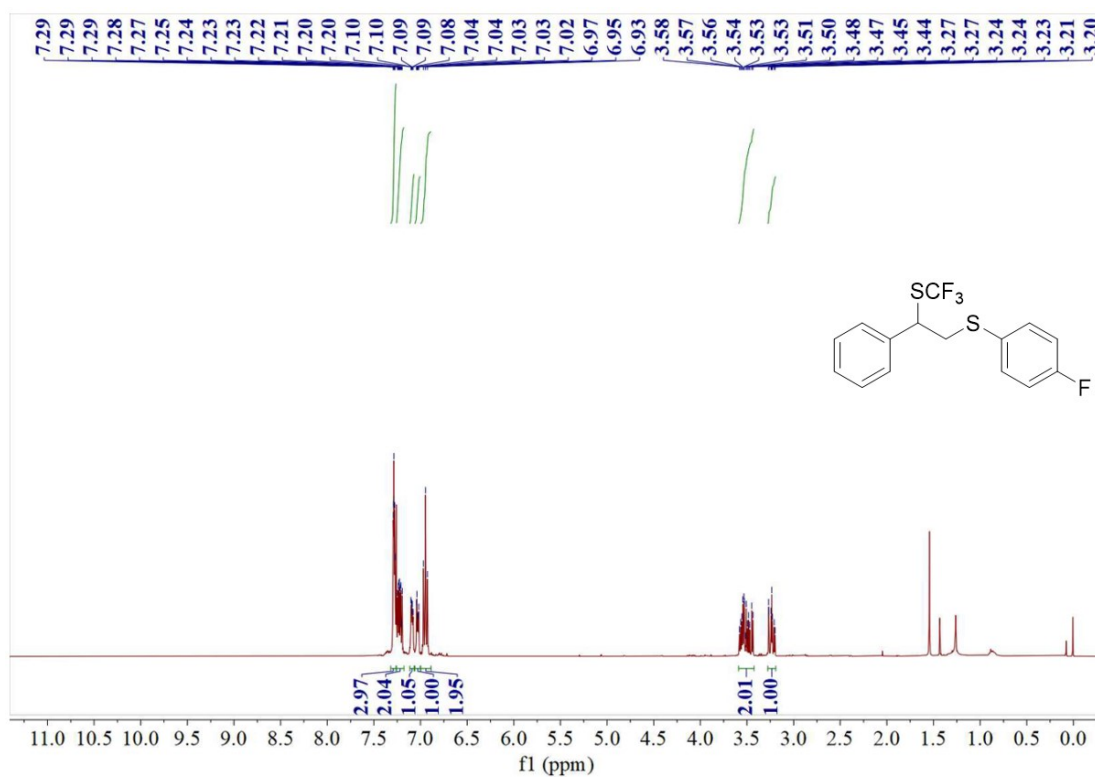
(5-methoxyphenyl)(2-phenyl-2-thiocyanatoethyl)sulfane (6)



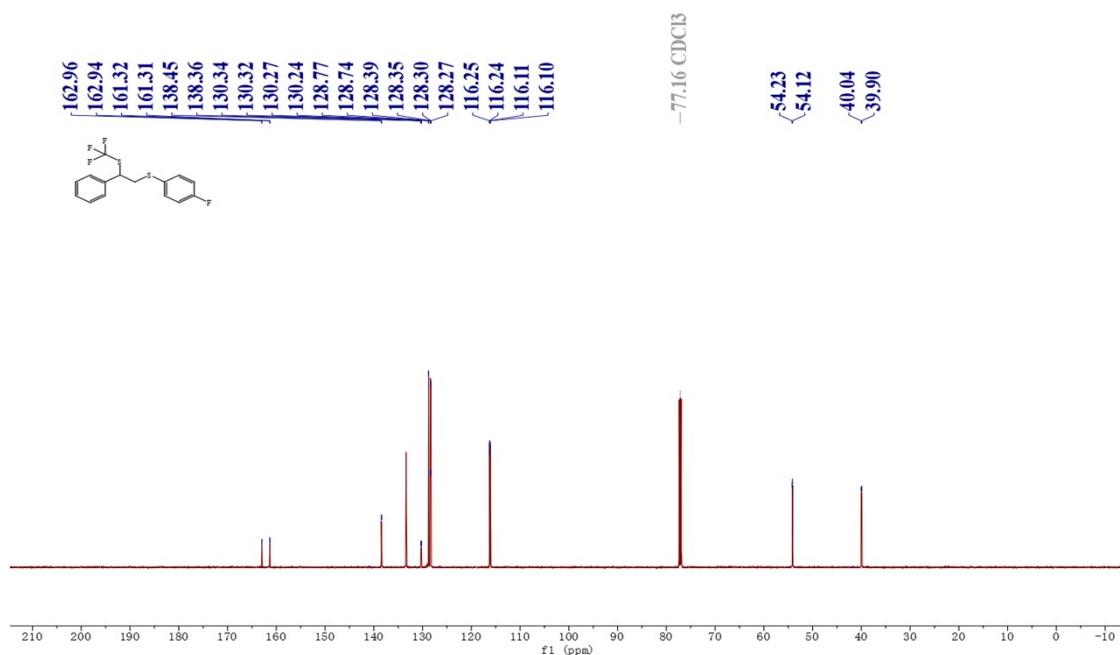
¹H NMR (400 MHz, CDCl₃) spectrum of 6



¹³C NMR (100 MHz, CDCl₃) spectrum of 6
(4-fluorophenyl)(2-phenyl-2-((trifluoromethyl)thio)ethyl)sulfane (7)

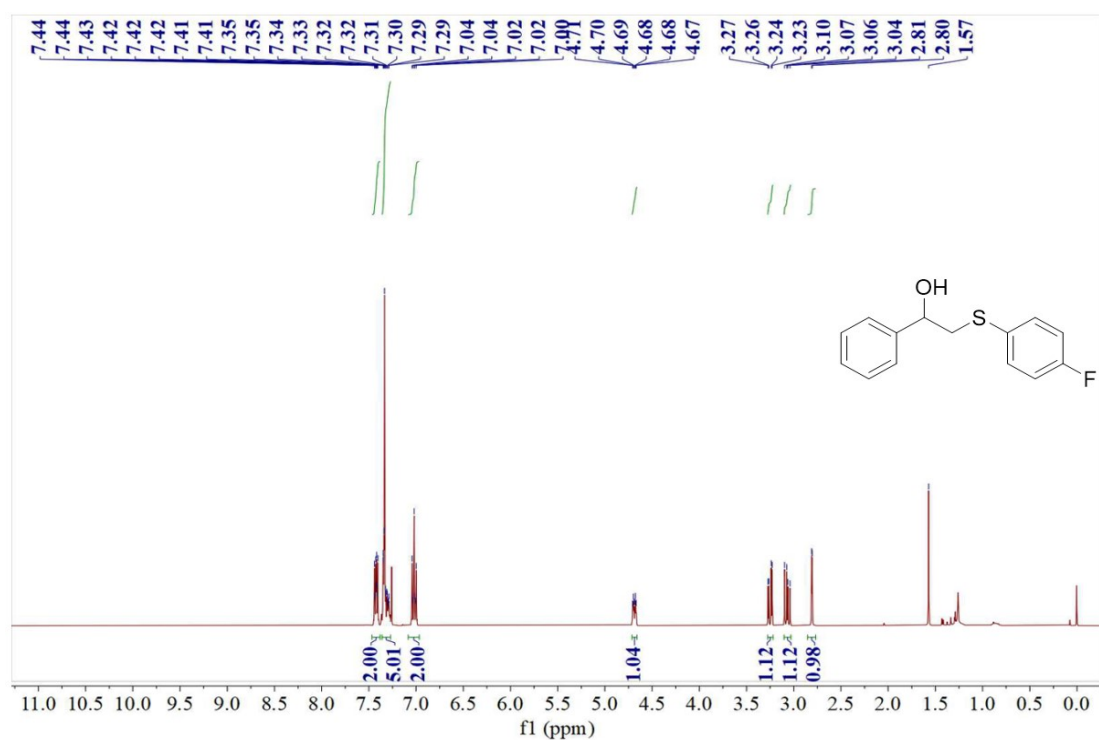


¹H NMR (400 MHz, CDCl₃) spectrum of 7



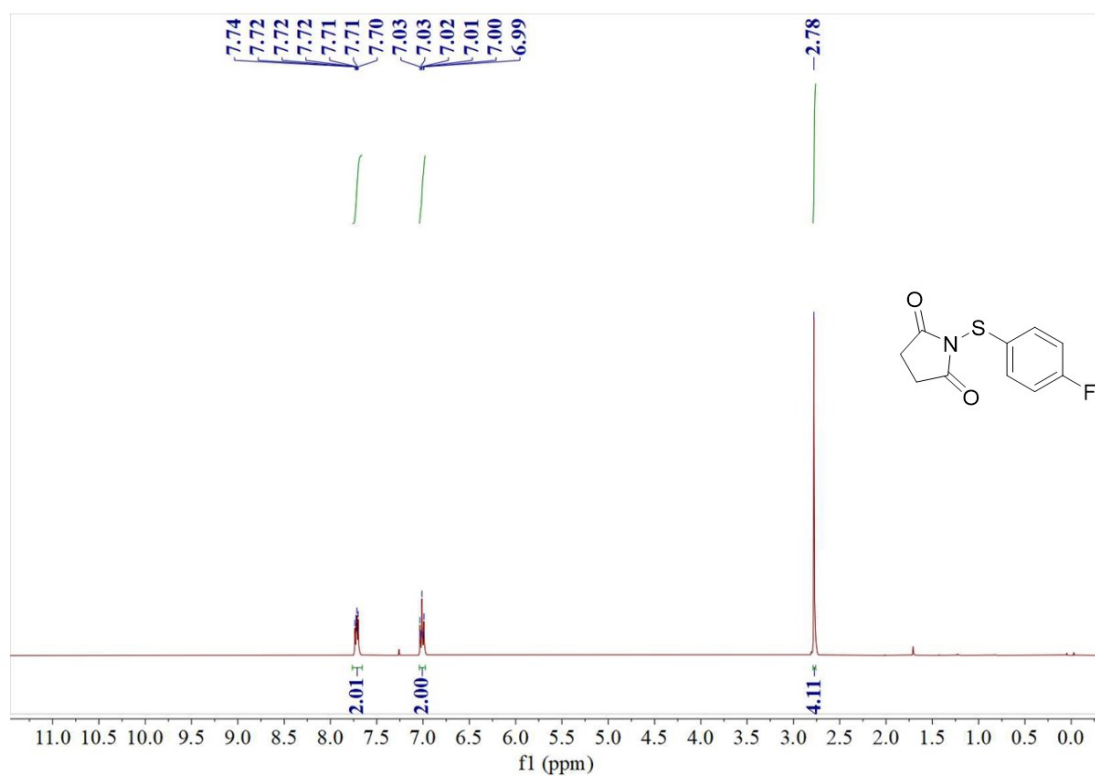
¹³C NMR (400 MHz, CDCl₃) spectrum of 7

2-((4-fluorophenyl)thio)-1-phenylethan-1-ol (C)



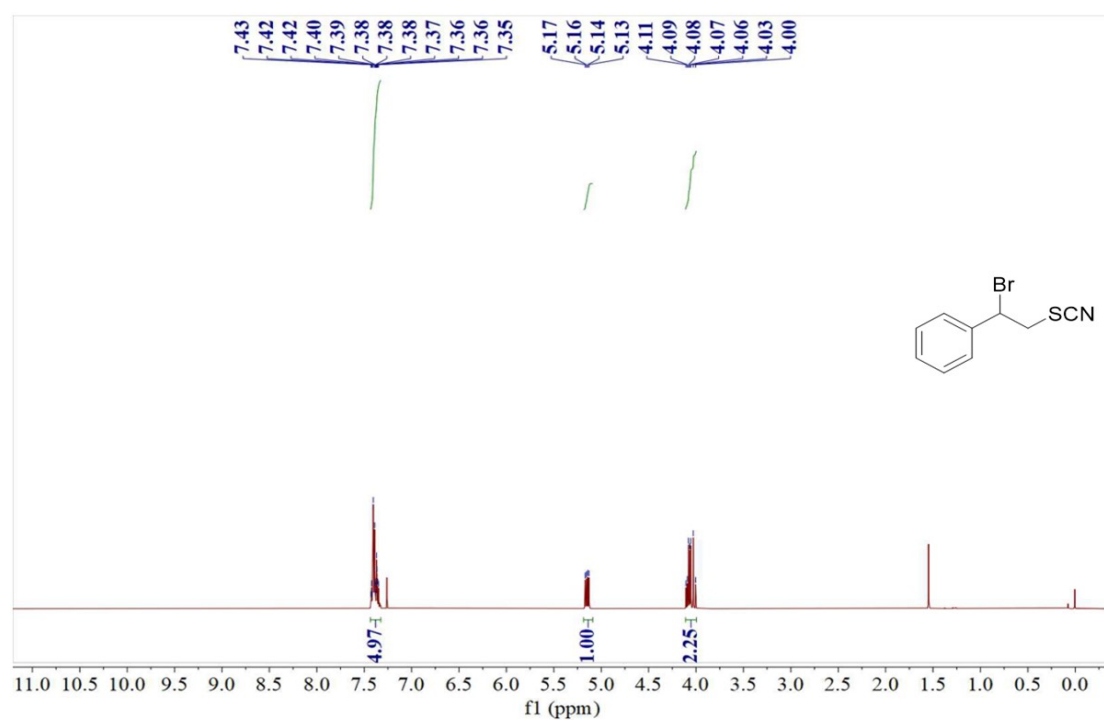
¹H NMR (400 MHz, CDCl₃) spectrum of C

1-((4-fluorophenyl)thio)pyrrolidine-2,5-dione (F)



¹H NMR (400 MHz, CDCl₃) spectrum of F

(1-bromo-2-thiocyanatoethyl)benzene (I)



¹H NMR (400 MHz, CDCl₃) spectrum of I