Supporting Information

Visible-light Induced Decarboxylative Coupling of Phenoxyacetic Acid with Disulfides: Synthesis of α-Arylthioanisole Derivatives

Ning Li[a], Zhao-Nian Peng[a], Run Xiong[a], Ao-Cheng Wang[a], Zhi-Bing Dong*[a, b]

[a]School of Chemistry and Environmental Engineering, Wuhan Institute of Technology, Wuhan 430205, China.

[b]School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang 453007, China.

Email: dzb04982@wit.edu.cn

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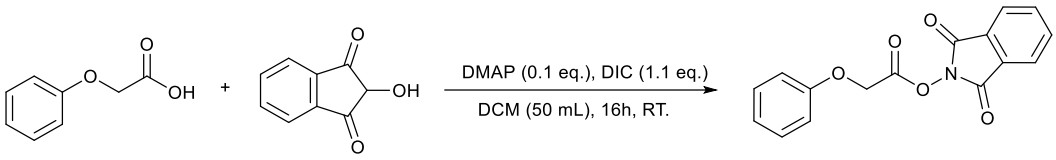
General Information.

All manipulations were carried out by using standard Schlenk techniques. Unless otherwise stated, analytical grade solvents and commercially available reagents were used to conduct the reactions. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum ether (bp. 60-90 ºC). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum ether to the ethyl acetate. All new compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. The known compounds were characterized by ^{1}H NMR and ^{13}C NMR. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz NMR spectrometer. The chemical shifts (δ) were given in part per million relative to internal tetramethyl silane (TMS, 0 ppm for ^{1}H), $CDCl_3$ (77.16 ppm for ^{13}C). High resolution mass spectra (HRMS) were measured with a Thermo Fisher Scientific LTQ FTICR-MS or a Bruker UltiMate3000 & Compact.

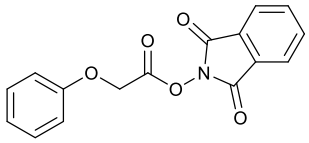
Experimental procedures.

1.General Procedure for Synthesis of redox-active esters

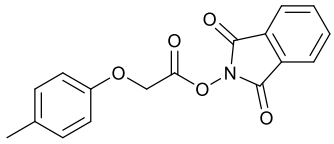


Phenoxyacetic acids (5.0 mmol), *N*-hydroxy-phthalimide (5.0 mmol), 4-dimethylaminopyridine (DMAP, 0.1 equiv) and dichloromethane (50 mL) were placed in a round bottom flask. After stirring the mixture vigorously, *N, N′*-diisopropylcarbodiimide (DIC, 1.1 equiv) was added dropwise to the mixture and the mixture was stirred for 16 hours. After completion of the reaction, the product was identified by TLC. The white solid was removed by filtration and the filtrate was concentrated. Finally, it was recrystallized with ethanol to obtain the corresponding product.

2. Characterization of redox-active esters

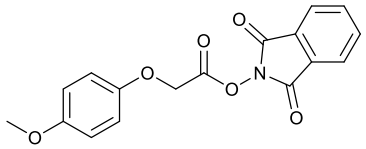


1,3-dioxoisoindolin-2-yl 2-phenoxyacetate (1a). According to General Procedure, a white solid was obtained in 79% isolated yield. Rf = 0.5 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.40–7.29 (m, 2H), 7.09–7.02 (m, 1H), 7.03–6.94 (m, 2H), 5.04 (s, 2H).

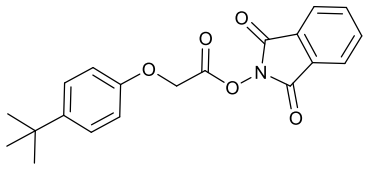


1,3-dioxoisoindolin-2-yl 2-(p-tolyloxy)acetate (1aa). According to General Procedure, a white solid was obtained in 80% isolated yield. Rf = 0.5 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5,

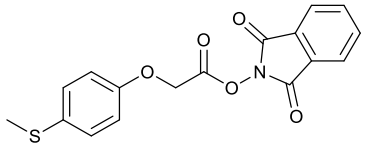
3.1 Hz, 2H), 7.12 (d, *J* = 8.2 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 5.00 (s, 2H), 2.29 (s, 3H).



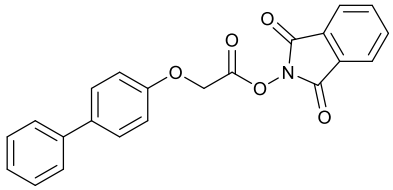
1,3-dioxoisoindolin-2-yl 2-(4-methoxyphenoxy)acetate (1ab). According to General Procedure, a white solid was obtained in 78% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.92 (d, *J* = 9.2 Hz, 2H), 6.83 (d, *J* = 9.1 Hz, 2H), 4.97 (s, 2H), 3.74 (s, 3H).



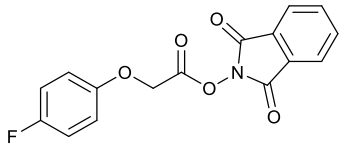
1,3-dioxoisoindolin-2-yl 2-(4-(tert-butyl)phenoxy)acetate (1ac). According to General Procedure, a white solid was obtained in 83% isolated yield. Rf = 0.5 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.35 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 5.02 (s, 2H), 1.30 (s, 9H).



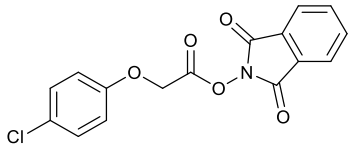
1,3-dioxoisoindolin-2-yl 2-(4-(methylthio)phenoxy)acetate (1ad). According to General Procedure, a yellow solid was obtained in 78% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 8.9 Hz, 2H), 5.01 (s, 2H), 2.43 (s, 3H).



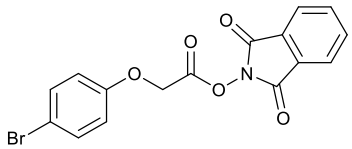
1,3-dioxoisoindolin-2-yl 2-([1,1'-biphenyl]-4-yloxy)acetate (1ae). According to General Procedure, a white solid was obtained in 74% isolated yield. Rf = 0.5 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.63–7.48 (m, 4H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 8.7 Hz, 2H), 5.07 (s, 2H).



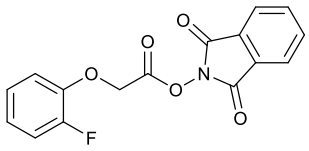
1,3-dioxoisoindolin-2-yl 2-(4-fluorophenoxy)acetate (1af). According to General Procedure, a white solid was obtained in 72% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.06 – 6.98 (m, 2H), 6.97 – 6.91 (m, 2H), 5.00 (s, 2H).



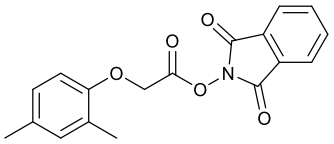
1,3-dioxoisoindolin-2-yl 2-(4-chlorophenoxy)acetate (1ag). According to General Procedure, a white solid was obtained in 77% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.29 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 9.0 Hz, 2H), 5.02 (s, 2H).



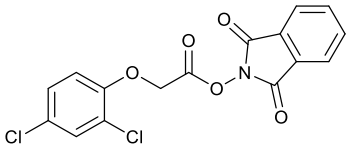
1,3-dioxoisoindolin-2-yl 2-(4-bromophenoxy)acetate (1ah). According to General Procedure, a white solid was obtained in 80% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.38 (d, *J* = 9.0 Hz, 2H), 6.85 (d, *J* = 9.0 Hz, 2H), 5.00 (s, 2H).



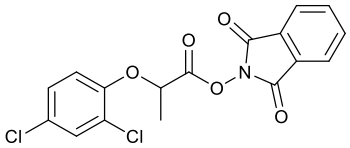
1,3-dioxoisoindolin-2-yl 2-(2-fluorophenoxy)acetate (1ai). According to General Procedure, a white solid was obtained in 70% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.13–7.01 (m, 3H), 7.02–6.92 (m, 1H), 5.09 (s, 2H).



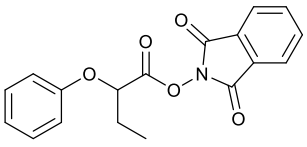
1,3-dioxoisoindolin-2-yl 2-(2,4-dimethylphenoxy)acetate (1aj). According to General Procedure, a white solid was obtained in 77% isolated yield. Rf = 0.5 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.98 (m, 2H), 6.76 (d, *J* = 8.2 Hz, 1H), 5.02 (s, 2H), 2.29 (s, 3H) 2.22 (s, 3H).



1,3-dioxoisoindolin-2-yl 2-(2,4-dichlorophenoxy)acetate (1ak). According to General Procedure, a white solid was obtained in 75% isolated yield. Rf = 0.4 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.39 (d, *J* = 2.5 Hz, 1H), 7.23 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 1H), 5.09 (s, 2H).



1,3-dioxoisoindolin-2-yl 2-(2,4-dichlorophenoxy)propanoate (1al). According to General Procedure, a white solid was obtained in 75% isolated yield. Rf = 0.6 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.34 (d, *J* = 2.5 Hz, 1H), 7.21 (dd, *J* = 8.8, 2.6 Hz, 1H), 7.01 (d, *J* = 8.8 Hz, 1H), 5.05 (q, *J* = 6.9 Hz, 1H), 1.90 (d, *J* = 6.9 Hz, 3H).



1,3-dioxoisoindolin-2-yl 2-phenoxybutanoate (1am). According to General Procedure, a white solid was obtained in 67% isolated yield. Rf = 0.6 (petroleum ether : EtOAc = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.34 (t, 2H), 7.03 (t, 3H), 4.95 (t, *J* = 6.0 Hz, 1H), 2.28–2.20 (m, 2H), 1.25 (t, *J* = 7.5 Hz, 3H).

3. Characterization of products

Typical procedure (TP) for visible-light induced decarboxylative coupling of aryl methyl ether derivatives with disulfides: To a Schlenk tube filled with nitrogen, a solution of the phenylmethoxy redox-active ester derivatives **1a** (0.2 mmol), diphenyl disulfide derivative **2a** (0.2 mmol) and $Ru(bpy)_3(PF_6)_2$ (1 mol%) in DMAC (2 mL) with DIPEA (0.05 mmol) was added, the mixture was stirred with the irradiation of 24 W blue LEDs for 12 h. When the reaction was finished, the mixture was then quenched with saturated NH_4Cl solution and extracted with ethyl acetate. The stock solution was dried with anhydrous Na_2SO_4 and the solvent was evaporated in vacuo. The pure product **3a** was purified by flash column chromatography (PE/EA) on silica gel.

(Phenoxymethyl)(phenyl)sulfane (3a). According to TP, a yellow liquid was obtained in 80% isolated yield (34.6 mg). Rf = 0.5 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.45 (d, 2H), 7.33–7.20 (m, 5H), 7.01 (t, 1H), 6.98–6.93 (d, 2H), 5.44 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 135.3, 130.6, 129.6, 129.1, 127.3, 122.1, 116.2, 73.1. HRMS (ESI) calculated for $C_{13}H_{13}OS^+$ $[M+H]^+$ 217.0682, found 217.0675.

(Phenoxymethyl)(p-tolyl)sulfane (3b). According to TP, a yellow liquid was obtained in 76% isolated yield (35.0 mg). Rf = 0.5 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.37 (m, 2H), 7.34–7.28 (m, 2H), 7.14–7.09 (m, 2H), 7.04–6.98 (m, 1H), 6.98–6.94 (m, 2H), 5.41 (s, 2H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 137.6, 131.5, 131.4, 129.9, 129.6, 122.0, 116.2, 73.7, 21.2. HRMS (ESI) calculated for $C_{14}H_{15}OS^+$ $[M+H]^+$ 231.0838, found 231.0830.

(4-Methoxyphenyl)(phenoxymethyl)sulfane (3c). According to TP, a yellow liquid was obtained in 82% isolated yield (40.6 mg). Rf = 0.4 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47–7.42 (m, 2H), 7.30 (dd, *J* = 8.6, 7.3 Hz, 2H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.98–6.91 (m, 2H), 6.87–6.78 (m, 2H), 5.34 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 156.9, 134.3, 129.6, 125.2, 122.0, 116.2, 114.7, 74.5, 55.4. HRMS (ESI) calculated for $C_{14}H_{15}O_2S^+$ $[M+H]^+$ 247.0787, found 247.0781.

(4-(Tert-butyl)phenyl)(phenoxymethyl)sulfane (3d). According to TP, a yellow liquid was obtained in 74% isolated yield (40.3 mg). Rf = 0.5 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.41 (m, 2H), 7.37–7.28 (m, 4H), 7.06–6.94 (m, 3H), 5.44 (s, 2H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 150.7, 131.6, 131.0, 129.6, 126.2, 122.0, 116.2, 73.5, 34.7, 31.4. HRMS (ESI) calculated for $C_{17}H_{21}OS^+$ $[M+H]^+$ 273.1308, found 273.1300.

(4-Fluorophenyl)(phenoxymethyl)sulfane (3e). According to TP, a yellow liquid was obtained in 69% isolated yield (32.3 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.45 (m, 2H), 7.35–7.29 (m, 2H), 7.07–6.91 (m, 5H), 5.39 (s, 2H). ^{13}C NMR (100 MHz, CDCl3) δ 162.6 (d, *J* = 247.4 Hz), 156.7, 133.8 (d, *J* = 8.1 Hz), 130.0 (d, *J* = 3.4 Hz), 129.7, 122.2, 116.4, 116.2, 73.8. ^{19}F NMR (377 MHz, $CDCl_3$) δ -114.0. HRMS (ESI) calculated for $C_{13}H_{12}FOS^+$ $[M+H]^+$ 235.0587, found 235.0579.

(4-Chlorophenyl)(phenoxymethyl)sulfane (3f). According to TP, a yellow liquid was obtained in 87% isolated yield (43.5 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.39 (m, 2H), 7.36–7.21 (m, 4H), 7.07–7.00 (m, 1H), 6.99–6.92 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 133.7, 133.5, 132.1, 129.7, 129.3, 122.3, 116.2, 73.1. HRMS (ESI) calculated for $C_{13}H_{12}ClOS^+$ $[M+H]^+$ 251.0292, found 251.0286.

(4-Bromophenyl)(phenoxymethyl)sulfane (3g). According to TP, a yellow liquid was obtained in 79% isolated yield (46.5 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.39 (m, 2H), 7.38–7.29 (m, 4H), 7.09–6.99 (m, 1H), 6.96 (t, *J* = 8, 1.1 Hz, 2H), 5.43 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 134.4,

132.2, 132.2, 129.7, 122.3, 121.5, 116.2, 72.9. HRMS (ESI) calculated for $C_{13}H_{12}BrOS^+$ $[M+H]^+$ 294.9787, found 294.9780.

(Phenoxymethyl)(4-(trifluoromethyl)phenyl)sulfane (3h). According to TP, a yellow liquid was obtained in 71% isolated yield (40.3 mg). Rf = 0.5 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61–7.50 (m, 4H), 7.40–7.29 (m, 2H), 7.09–7.02 (m, 1H), 7.00–6.94 (m, 2H), 5.52 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 140.7, 129.8, 129.2, δ 128.8 (q, J = 32.7 Hz), 125.9 (q, J = 4 Hz), δ 124.2 (q, J = 272.7 Hz). 122.5, 116.2, 71.9. HRMS (ESI) calculated for $C_{14}H_{12}F_3OS^+$ $[M+H]^+$ 285.0555, found 285.0547.

2-Fluorophenyl)(phenoxymethyl)sulfane (3i). According to TP, a yellow liquid was obtained in 78% isolated yield (36.5 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 1.8 Hz, 1H), 7.37–7.22 (m, 3H), 7.15–6.99 (m, 3H), 6.96 (dd, J = 8.7, 1.1 Hz, 2H), 5.45 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4 (d, J = 246.0 Hz), 156.7, 133.5, 133.5, 129.7, 129.6 (d, J = 7.7 Hz), 124.8 (d, J = 3.8 Hz), 122.3, 121.7 (d, J = 17.6 Hz), 116.2, 115.9 (d, J = 22.4 Hz), 72.2, 72.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ -109.1. HRMS (ESI) calculated for $C_{13}H_{12}FOS^+$ $[M+H]^+$ 235.0587, found 235.0580.

(2-Chlorophenyl)(phenoxymethyl)sulfane (3j). According to TP, a yellow liquid was obtained in 83% isolated yield (41.5 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, J = 7.9, 1.7 Hz, 1H), 7.40–7.29 (m, 3H), 7.17 (m, 1H), 7.07–6.95 (m, 3H), 5.52 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 134.7, 134.0, 130.3, 129.8, 129.7, 127.9, 127.6, 122.3, 116.1, 71.3. HRMS (ESI) calculated for $C_{13}H_{12}ClOS^+$ $[M+H]^+$ 251.0292, found 251.0284.

(3-Bromophenyl)(phenoxymethyl)sulfane (3k). According to TP, a yellow liquid was obtained in 87% isolated yield (51.2 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (t, J = 1.9 Hz, 1H), 7.38 (m, 2H), 7.35–7.29 (m, 2H), 7.15 (t, J = 7.9 Hz, 1H), 7.06–7.00 (m, 1H), 6.99–6.94 (m, 2H), 5.45 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 137.6, 132.8, 130.4, 130.3, 129.7, 128.8, 122.9, 122.4,

116.2, 72.8. HRMS (ESI) calculated for $C_{13}H_{12}BrOS^+$ $[M+H]^+$ 294.9787, found 294.9780.

(2,4-Dimethylphenyl)(phenoxymethyl)sulfane (3l). According to TP, a yellow liquid was obtained in 81% isolated yield (39.5 mg). Rf = 0.5 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.8 Hz, 1H), 7.30 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.05–6.90 (m, 5H), 5.39 (s, 2H), 2.33 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 139.2, 137.6, 131.8, 131.2, 130.8, 129.6, 127.6, 121.9, 116.0, 73.1, 21.1, 20.9. HRMS (ESI) calculated for $C_{15}H_{17}OS^+$ $[M+H]^+$ 245.0995, found 245.0987.

(3,4-Dichlorophenyl)(phenoxymethyl)sulfane (3m). According to TP, a yellow liquid was obtained in 70% isolated yield (39.8 mg). Rf = 0.4 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 2.1 Hz, 1H), 7.43–7.33 (m, 4H), 7.13–7.07 (m, 1H), 7.04–6.97 (m, 2H), 5.50 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 135.4, 133.0, 131.9, 131.6, 130.8, 129.8, 129.7, 122.5, 116.2, 72.8. HRMS (ESI) calculated for $C_{13}H_{11}Cl_2OS^+$ $[M+H]^+$ 284.9902, found 284.9895.

4-((Phenoxymethyl)thio)pyridine (3n). According to TP, a yellow liquid was obtained in 56% isolated yield. (24.3 mg). Rf = 0.6 (petroleum ether : EtOAc = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 – 8.44 (m, 2H), 7.37–7.30 (m, 4H), 7.09–7.03 (m, 1H), 7.01–6.95 (m, 2H), 5.57 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 149.6, 147.4, 129.8, 122.7, 121.8, 116.2, 70.0. HRMS (ESI) calculated for $C_{12}H_{12}NOS^+$ $[M+H]^+$ 218.0634, found 218.0626.

2-((Phenoxymethyl)thio)pyridine (3o). According to TP, a yellow liquid was obtained in 70% isolated yield (30.4 mg). Rf = 0.5 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (m, 1H), 7.52 (m, 1H), 7.35–7.23 (m, 3H), 7.09–6.95 (m, 4H), 5.90 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 156.7, 149.8, 136.7, 129.6, 123.0, 122.0, 120.7, 116.0, 68.4. HRMS (ESI) calculated for $C_{12}H_{12}NOS^+$ $[M+H]^+$ 218.0634, found 218.0626.

2-(3-Phenylpropyl)thiophene (3p). According to TP, a colorless transparent liquid was obtained in 84% isolated yield (37.3 mg). Rf = 0.4 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 5.3, 1.3 Hz, 1H), 7.32 (dd, *J* = 8.7, 7.2 Hz,

2H), 7.18 (dd, *J* = 3.6, 1.3 Hz, 1H), 7.06–6.94 (m, 4H), 5.29 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 134.8, 131.7, 130.4, 129.7, 127.7, 122.3, 116.4, 75.8. HRMS (ESI) calculated for $C_{11}H_{11}OS_2^+$ $[M+H]^+$ 223.0246, found 223.0237.

(2,4-Dimethylphenyl)(phenoxymethyl)sulfane (3q). According to TP, a yellow liquid was obtained in 31% isolated yield (16.9 mg). Rf = 0.5 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (m, 1H), 7.79 (m, 1H), 7.45 (m, 1H), 7.34 (m, 3H), 7.11–6.99 (m, 3H), 5.95 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 156.4, 153.1, 135.8, 129.8, 126.4, 124.8, 122.8, 122.2, 121.2, 116.3, 71.1. HRMS (ESI) calculated for $C_{14}H_{12}NOS_2^+$ $[M+H]^+$ 274.0355, found 274.0347.

Phenyl((p-tolyloxy)methyl)sulfane (4a). According to TP, a yellow liquid was obtained in 80% isolated yield (36.8 mg). Rf = 0.5 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.46 (m, 2H), 7.33–7.22 (m, 2H), 7.13–7.08 (m, 1H), 6.90–6.83 (m, 2H), 5.44 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 135.4, 131.5, 130.6, 130.1, 129.1, 127.2, 116.2, 73.4, 20.7. HRMS (ESI) calculated for $C_{14}H_{15}OS^+$ $[M+H]^+$ 231.0838, found 231.0832.

((4-Methoxyphenoxy)methyl)(phenyl)sulfane (4b). According to TP, a yellow liquid was obtained in 82% isolated yield (40.4 mg). Rf = 0.5 (petroleum ether : EtOAc = 6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53–7.47 (m, 2H), 7.34–7.22 (m, 3H), 6.95–6.82 (m, 4H), 5.42 (s, 2H), 3.77 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.0, 150.7, 135.4, 130.5, 129.1, 127.2, 117.7, 114.8, 74.3, 55.8. HRMS (ESI) calculated for $C_{14}H_{15}O_2S^+$ $[M+H]^+$ 247.0787, found 247.0780.

((4-(Tert-butyl)phenoxy)methyl)(phenyl)sulfane (4c). According to TP, a yellow liquid was obtained in 86% isolated yield (46.8 mg). Rf = 0.5 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.46 (m, 2H), 7.35–7.26 (m, 4H), 7.26–7.20 (m, 1H), 6.93–6.88 (m, 2H), 5.44 (s, 2H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 144.9, 135.5, 130.6, 129.1, 127.2, 126.5, 115.7, 73.3, 34.3, 31.6. HRMS (ESI) calculated for $C_{17}H_{21}OS^+$ $[M+H]^+$ 273.1308, found 273.1302.

Methyl(4-((phenylthio)methoxy)phenyl)sulfane (4d). According to TP, a yellow liquid was obtained in 89% isolated yield (46.6 mg). Rf = 0.5 (petroleum ether : EtOAc =

6:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.45 (m, 2H), 7.34–7.21 (m, 5H), 6.94–6.86 (m, 2H), 5.43 (s, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.0, 135.1, 130.7, 130.7, 129.6, 129.2, 127.3, 116.9, 73.4, 17.6. HRMS (ESI) calculated for $C_{14}H_{15}OS_2^+$ $[M+H]^+$ 263.0559, found 263.0553.

([1,1'-Biphenyl]-4-yloxy)methyl)(phenyl)sulfane (4e). According to TP, a yellow solid was obtained in 54% isolated yield (31.5 mg). Rf = 0.5 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.48 (m, 6H), 7.41 (dd, *J* = 8.5, 6.9 Hz, 2H), 7.35–7.23 (m, 4H), 7.06–7.00 (m, 2H), 5.49 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 140.7, 135.2, 130.7, 129.2, 128.9, 128.4, 127.4, 127.0, 126.9, 116.4, 73.3. HRMS (ESI) calculated for $C_{19}H_{17}OS^+$ $[M+H]^+$ 293.0995, found 293.0987.

((4-Fluorophenoxy)methyl)(phenyl)sulfane (4f). According to TP, a yellow liquid was obtained in 82% isolated yield (38.4 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.46 (m, 2H), 7.35–7.20 (m, 3H), 7.00 (dd, *J* = 9.2, 8.1 Hz, 2H), 6.96 – 6.88 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2 (d, *J* = 240.2 Hz),152.8, 152.8, 135.0, 130.7, 129.2, 127.4, 117.7 (d, *J* = 8.1 Hz), 116.1 (d, *J* = 23.1 Hz).74.1. ^{19}F NMR (377 MHz, $CDCl_3$) δ -122.0. HRMS (ESI) calculated for $C_{13}H_{12}FOS^+$ $[M+H]^+$ 235.0587, found 235.0580.

((4-Chlorophenoxy)methyl)(phenyl)sulfane (4g). According to TP, a yellow liquid was obtained in 78% isolated yield (39.0 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.44 (m, 2H), 7.36–7.20 (m, 5H), 6.92–6.84 (m, 2H), 5.42 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.3, 134.8, 130.8, 129.5, 129.2, 127.5, 127.1, 117.6, 73.5. HRMS (ESI) calculated for $C_{13}H_{12}ClOS^+$ $[M+H]^+$ 251.0292, found 251.0286.

((4-Bromophenoxy)methyl)(phenyl)sulfane (4h). According to TP, a yellow liquid was obtained in 75% isolated yield (44.1 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.44 (m, 2H), 7.43–7.37 (m, 2H), 7.34–7.21 (m, 3H), 6.87–6.79 (m, 2H), 5.42 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 134.8, 132.5, 130.8, 129.2, 127.5, 118.0, 114.5, 73.4. HRMS (ESI) calculated for $C_{13}H_{12}BrOS^+$ $[M+H]^+$ 294.9787, found 294.9780.

((2-Fluorophenoxy)methyl)(phenyl)sulfane (4i). According to TP, a yellow liquid was obtained in 77% isolated yield (36.0 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–7.49 (m, 2H), 7.35–7.22 (m, 3H), 7.13–6.95 (m, 4H), 5.53 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9 (d, *J* = 246.5 Hz), 144.4 (d, *J* = 10.6 Hz), 135.0, 130.8, 129.2, 127.5, 127.4, 124.3 (d, *J* = 3.8 Hz), 123.2 (d, *J* = 7.0 Hz), 118.6 (d, *J* = 1.8 Hz), 116.8 (d, *J* = 18.4 Hz). 75.3, 75.2. ^{19}F NMR (377 MHz, $CDCl_3$) δ -132.5. HRMS (ESI) calculated for $C_{13}H_{12}FOS^+$ $[M+H]^+$ 235.0587, found 235.0582.

((2,4-Dimethylphenoxy)methyl)(phenyl)sulfane (4j). According to TP, a yellow liquid was obtained in 81% isolated yield (39.5 mg). Rf = 0.5 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61–7.55 (m, 2H), 7.37 (dd, *J* = 8.3, 6.5 Hz, 2H), 7.34–7.29 (m, 1H), 7.04 (d, *J* = 8.3 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 1H), 5.53 (s, 2H), 2.34 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 135.6, 132.0, 131.2, 130.5, 129.1, 128.0, 127.1, 127.0, 113.4, 73.3, 20.7, 16.5. HRMS (ESI) calculated for $C_{15}H_{17}OS^+$ $[M+H]^+$ 245.0995, found 245.0990.

((2,4-Dichlorophenoxy)methyl)(phenyl)sulfane (4k). According to TP, a yellow liquid was obtained in 53% isolated yield (30.1 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–7.49 (m, 2H), 7.39 (d, *J* = 2.5 Hz, 1H), 7.31 (m, 3H), 7.19 (dd, *J* = 8.8, 2.6 Hz, 1H), 6.91 (d, *J* = 8.8 Hz,1H), 5.50 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 134.5, 131.1, 130.4, 129.3, 127.7, 127.5, 127.5, 125.4, 117.1, 74.7. HRMS (ESI) calculated for $C_{13}H_{11}Cl_2OS^+$ $[M+H]^+$ 284.9902, found 284.9898.

((2,3-Dichlorophenoxy)methyl)(phenyl)sulfane (4l). According to TP, a yellow liquid was obtained in 82% isolated yield (48.9 mg). Rf = 0.6 (petroleum ether : EtOAc = 7:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47–7.42 (m, 2H), 7.37 (d, *J* = 2.5 Hz, 1H), 7.32–7.26 (m, 3H), 7.15 (dd, *J* = 8.8, 2.6 Hz, 1H), 6.97 (d, *J* = 8.8 Hz, 1H), 5.59 (q, *J* = 6.3 Hz, 1H), 1.69 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.3, 134.7, 130.9, 130.3, 129.0, 128.5, 127.5, 125.7, 118.7, 83.3, 22.3. HRMS (ESI) calculated for $C_{14}H_{13}Cl_2OS^+$ $[M+H]^+$ 299.0059, found 299.0053.

(1-Phenoxypropyl)(phenyl)sulfane (4m). According to TP, a yellow liquid was obtained in 87% isolated yield (42.5 mg). Rf = 0.6 (petroleum ether : EtOAc = 7:1). ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.46–7.41 (m, 2H), 7.32–7.21 (m, 5H), 7.03–6.95 (m, 3H), 5.32 (t, *J* = 6.5 Hz, 1H), 2.04–1.80 (m, 2H), 1.07 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 134.6, 131.8, 129.6, 128.8, 128.2, 122.0, 117.1, 87.3, 29.1, 10.9. HRMS (ESI) calculated for $C_{15}H_{17}OS^+$ $[M+H]^+$ 245.0995, found 245.0988.

(4-Methoxyphenoxy)methyl morpholine-4-carbodithioate (5a) According to TP, a white solid was obtained in 60% isolated yield (35.9 mg). Rf = 0.6 (petroleum ether : EtOAc = 3:1). ^1H NMR (400 MHz, $CDCl_3$) δ 6.97–6.93 (m, 2H), 6.85–6.81 (m, 2H), 6.01 (s, 2H), 4.35 (s, 2H), 3.93 (s, 2H), 3.78–3.72 (m, 7H). ^{13}C NMR (100 MHz, CDCl3) δ 194.6, 155.10, 151.1, 117.4, 114.7, 75.1, 66.4, 66.2, 55.8, 51.9, 51.0. HRMS (ESI) calculated for $C_{13}H_{18}NO_3S_2^+$ $[M+H]^+$ 300.0723, found 300.0717.

(p-Tolyloxy)methyl diethylcarbamodithioate (5b) According to TP, a yellow liquid was obtained in 64% isolated yield (34.4 mg). Rf = 0.6 (petroleum ether : EtOAc = 5:1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.10–7.08 (m, 2H), 6.93–6.89 (m, 2H), 6.02 (s, 2H), 4.04 (q, *J* = 7.1 Hz, 2H), 3.73 (q, *J* = 7.2 Hz, 2H), 2.29 (s, 3H), 1.27 (q, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 155.1, 131.5, 130.0, 115.9, 74.5, 49.8, 47.1, 20.6, 12.8, 11.6. HRMS (ESI) calculated for $C_{13}H_{20}NOS_2^+$ $[M+H]^+$ 270.0981, found 270.0974.

The failed substrates in this protocol

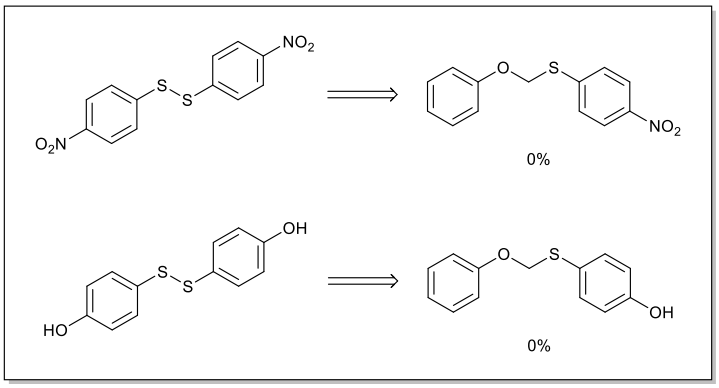


Figure S1 The failed substrates in this protocol.

Cyclic voltammetry experiment

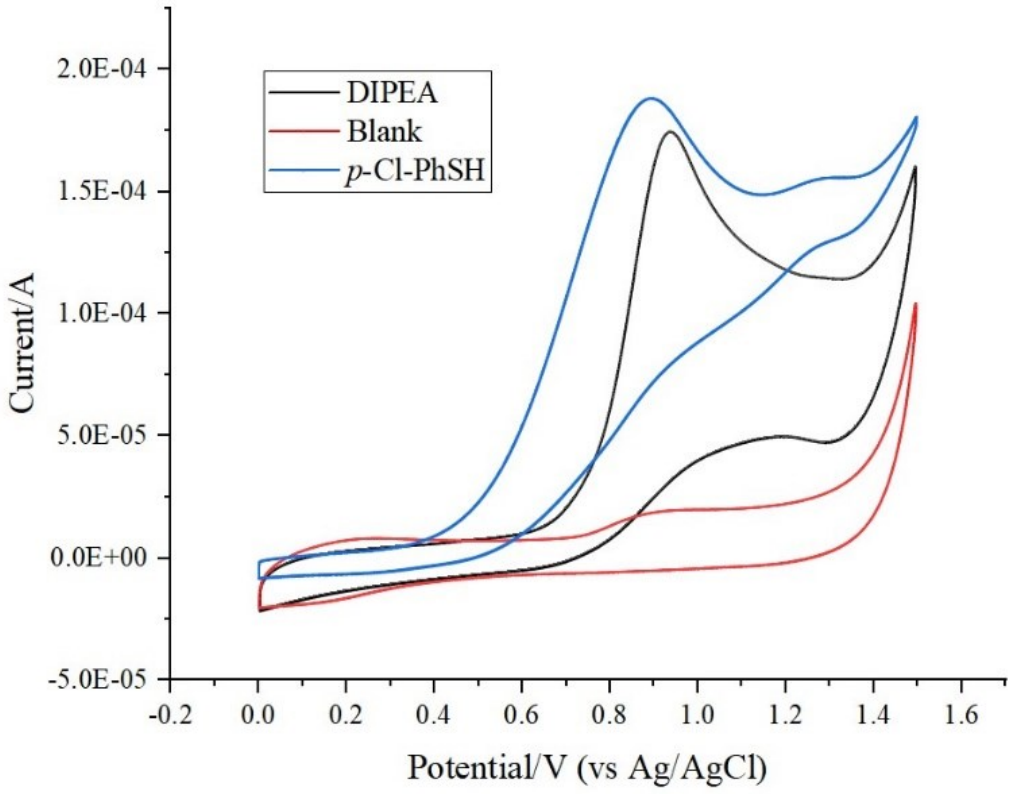
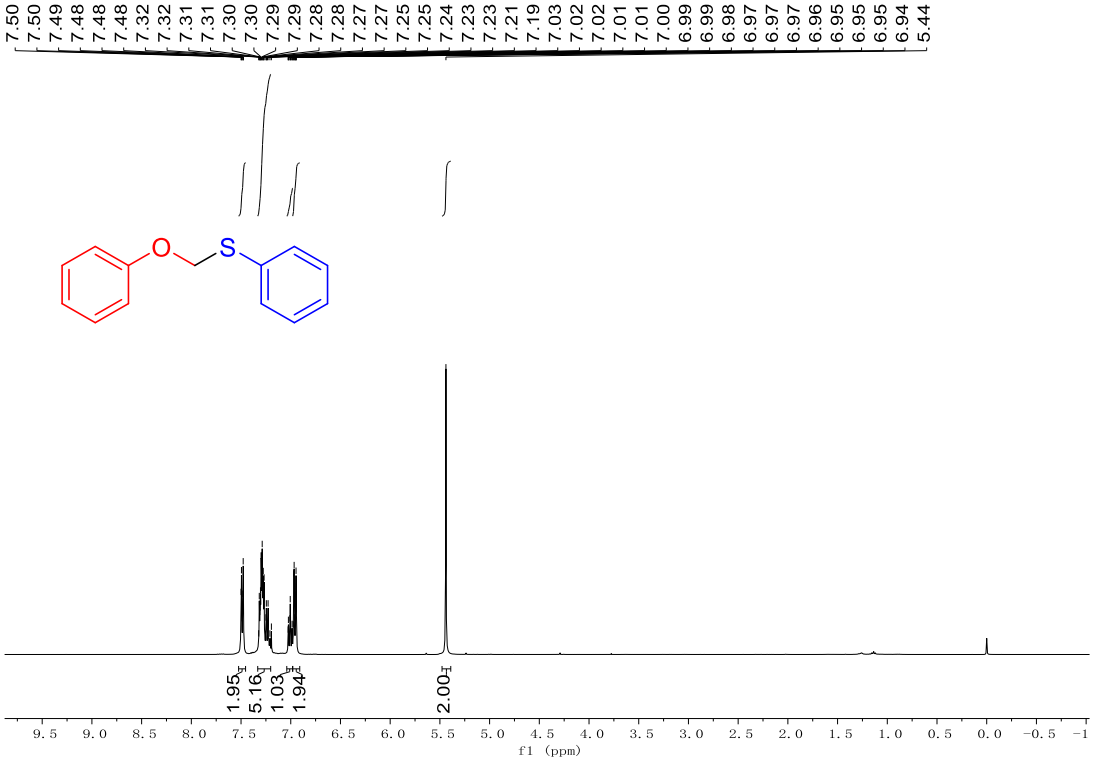


Figure S2 Cyclic voltammetry experiment of DIPEA and p-Cl-PhSH.

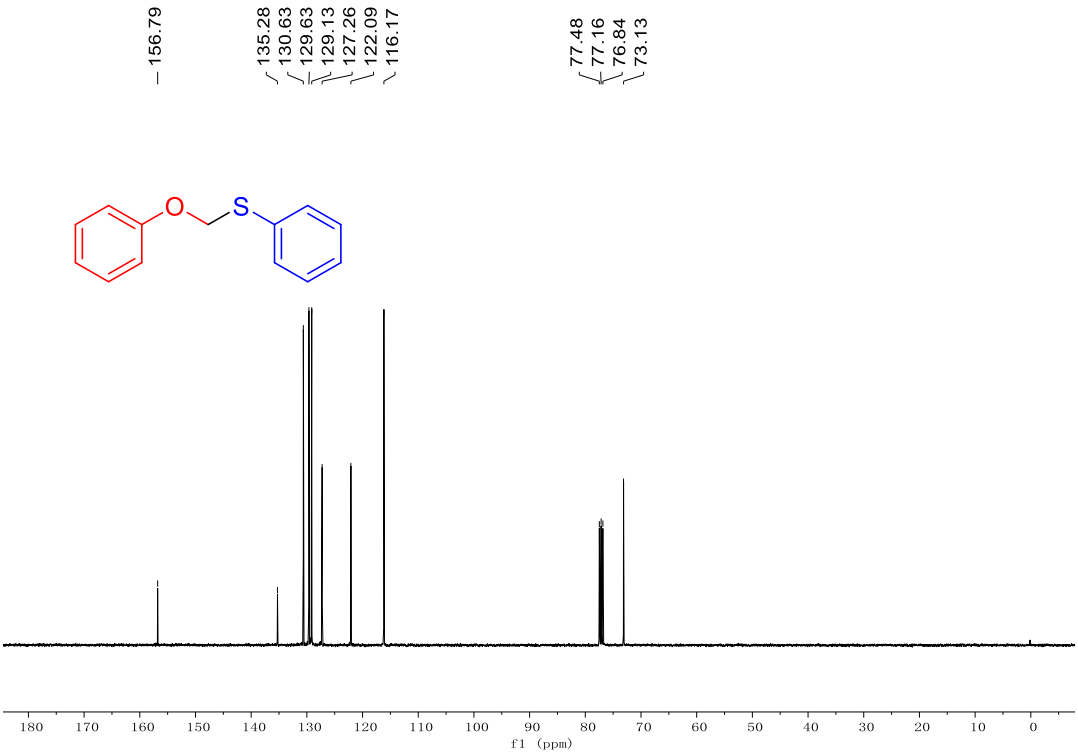
Cyclic voltammetry experiment was performed in a three-electrode cell connected to a Schlenk line under air at room temperature. The working electrode was a glass carbon electrode, the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in 10 ml DMAC. Corresponding test substance (0.1 mmol), ^{n}-Bu_4NBF_4 (0.5 mmol) was added each time when the experiment was performed. The scan rate is 0.1 V/s.

^{1}H-NMR, ^{13}C-NMR and ^{19}F-NMR spectrum of 3a-3q, 4a-4m.

(Phenoxymethyl)(phenyl)sulfane (3a).

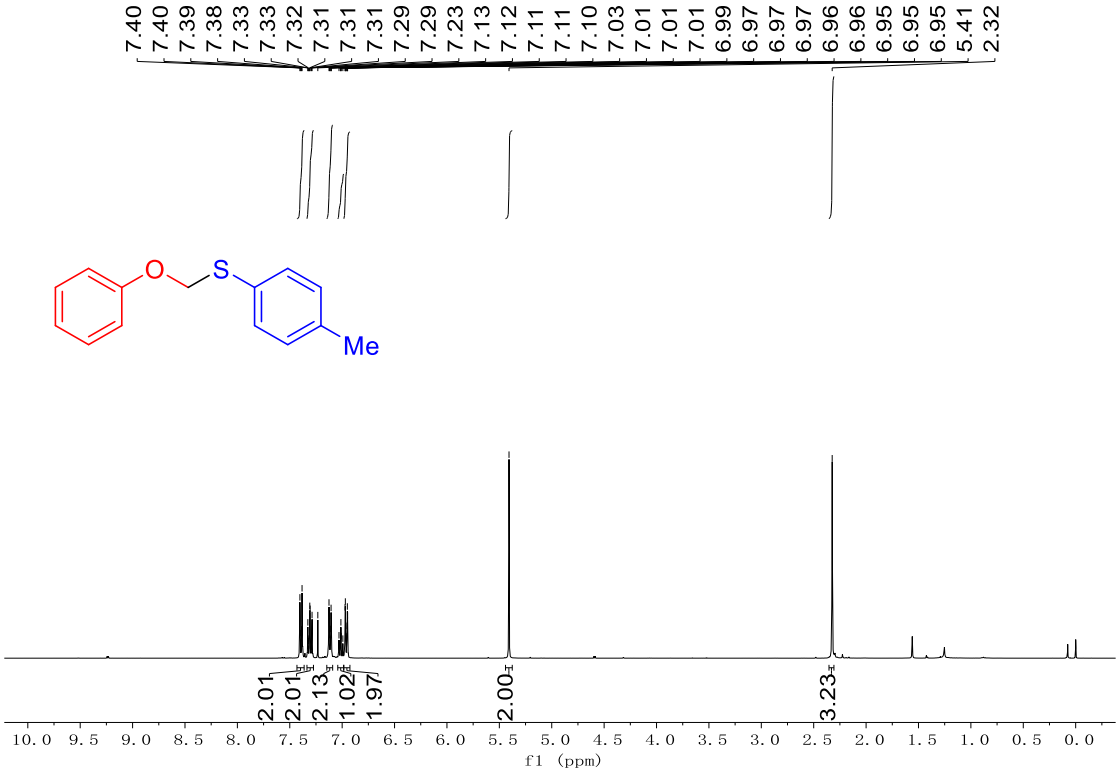


^{1}H-NMR spectrum of **3a** (400 MHz, $CDCl_3$)

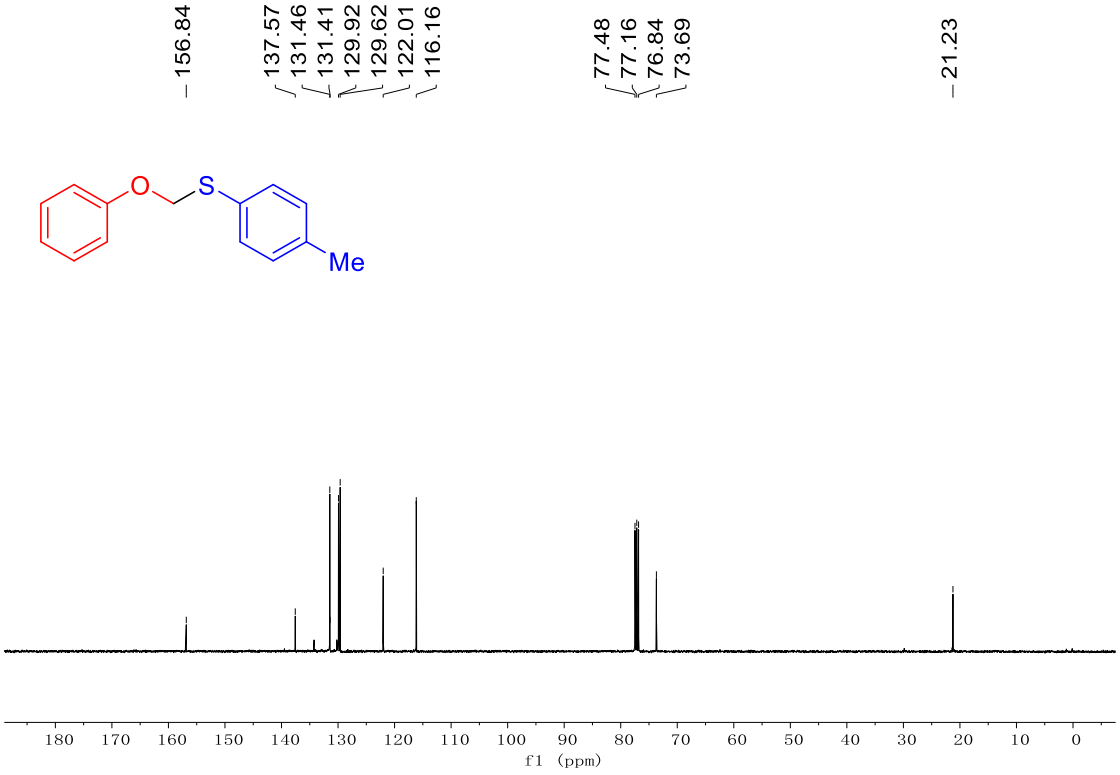


^{13}C-NMR spectrum of **3a** (100 MHz, $CDCl_3$)

(Phenoxymethyl)(p-tolyl)sulfane (3b).

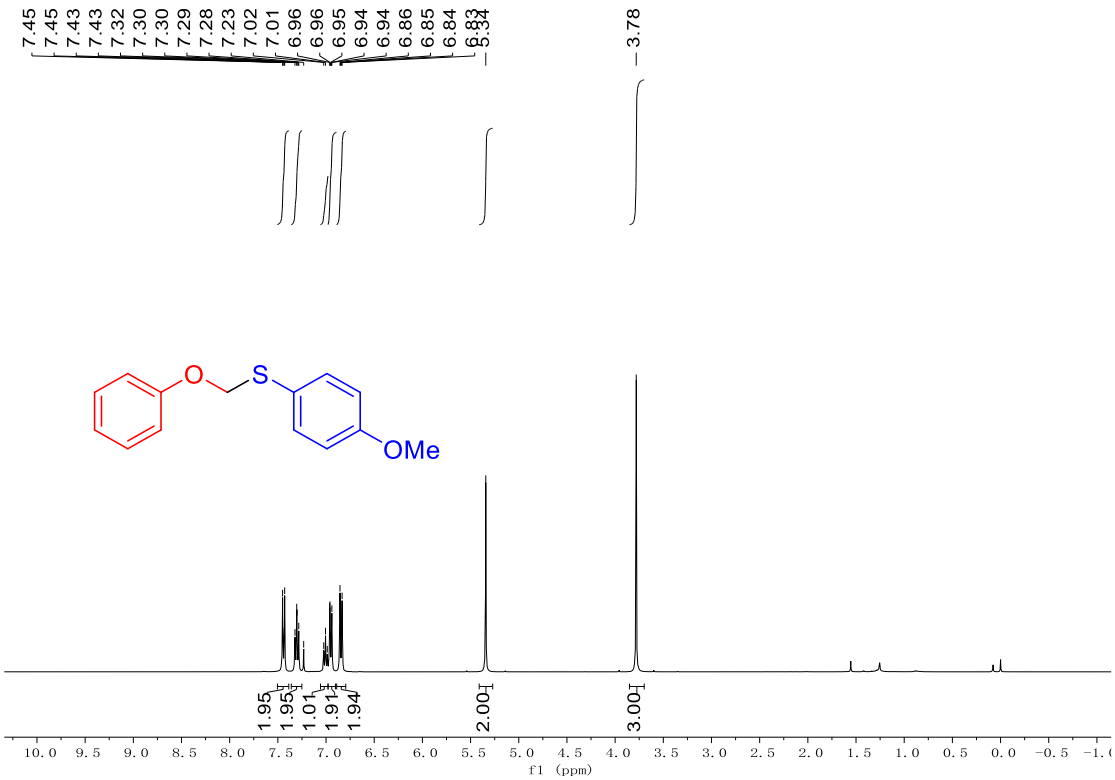


^{1}H-NMR spectrum of **3b** (400 MHz, $CDCl_3$)

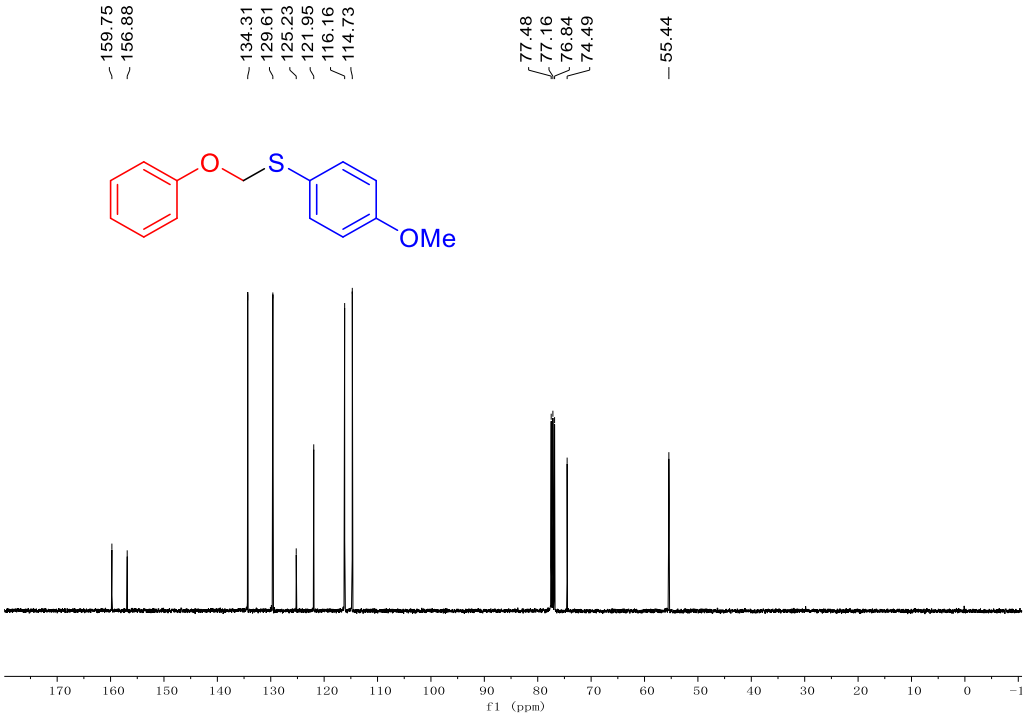


^{13}C-NMR spectrum of **3b** (100 MHz, $CDCl_3$)

(4-Methoxyphenyl)(phenoxymethyl)sulfane (3c).

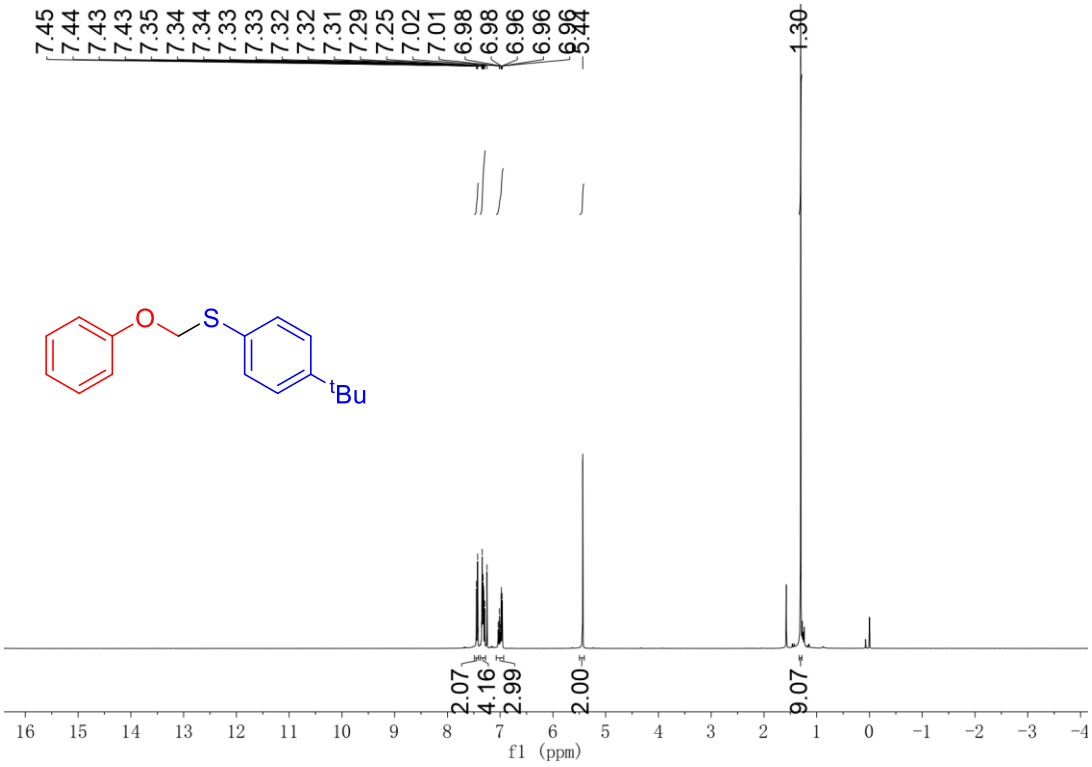


^{1}H-NMR spectrum of **3c** (400 MHz, $CDCl_3$)

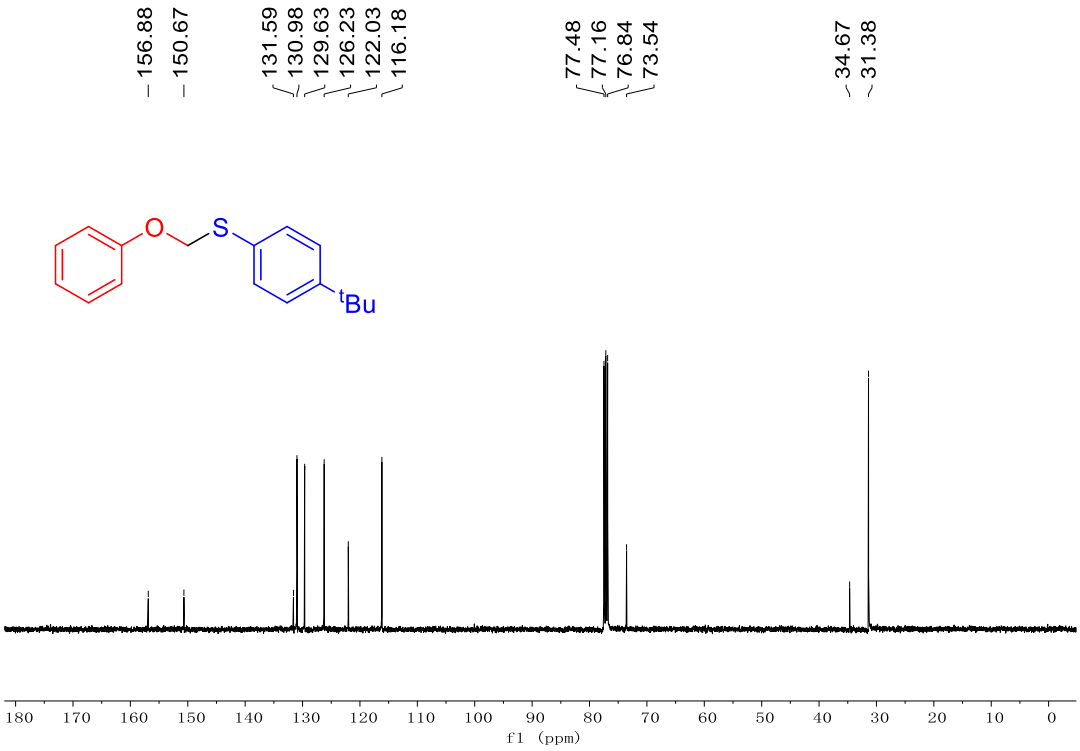


^{13}C-NMR spectrum of **3c** (100 MHz, $CDCl_3$)

(4-(Tert-butyl)phenyl)(phenoxymethyl)sulfane (3d).

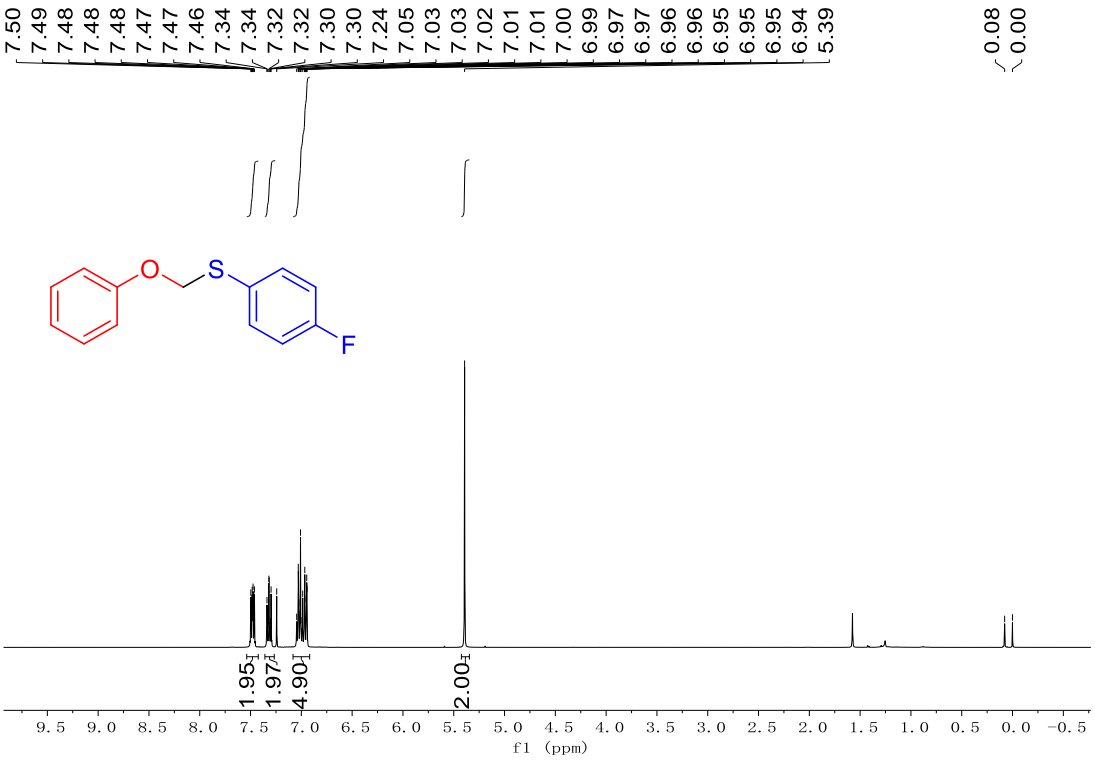


^{1}H-NMR spectrum of **3d** (400 MHz, $CDCl_3$)

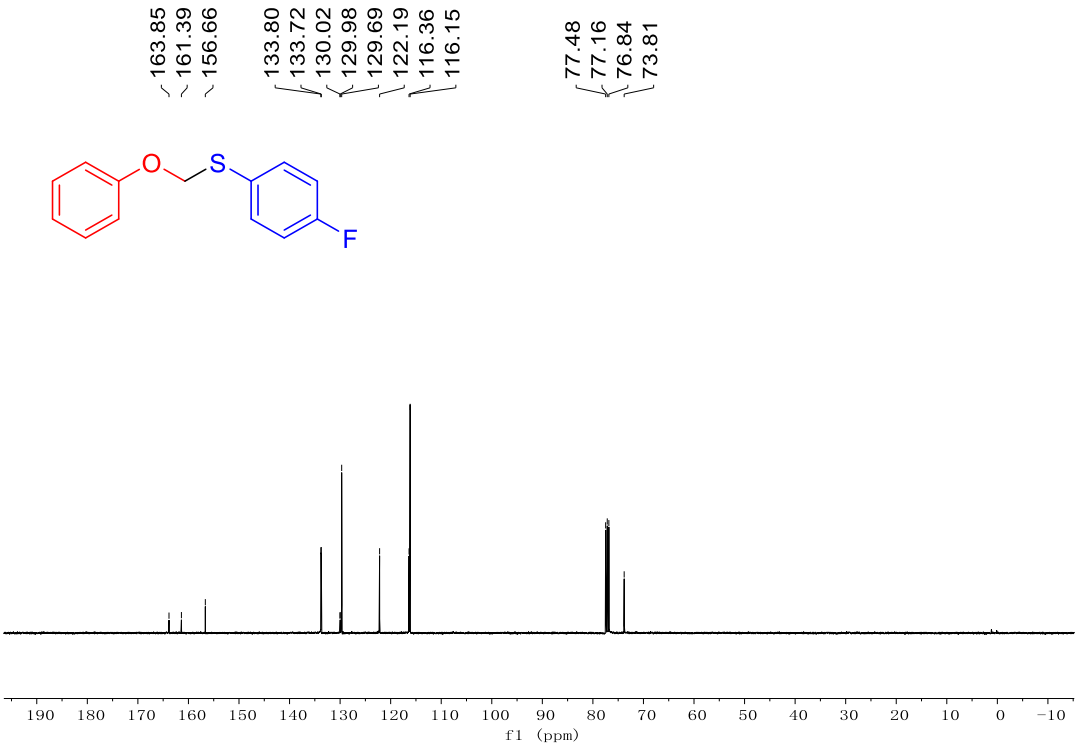


^{13}C-NMR spectrum of **3d** (100 MHz, $CDCl_3$)

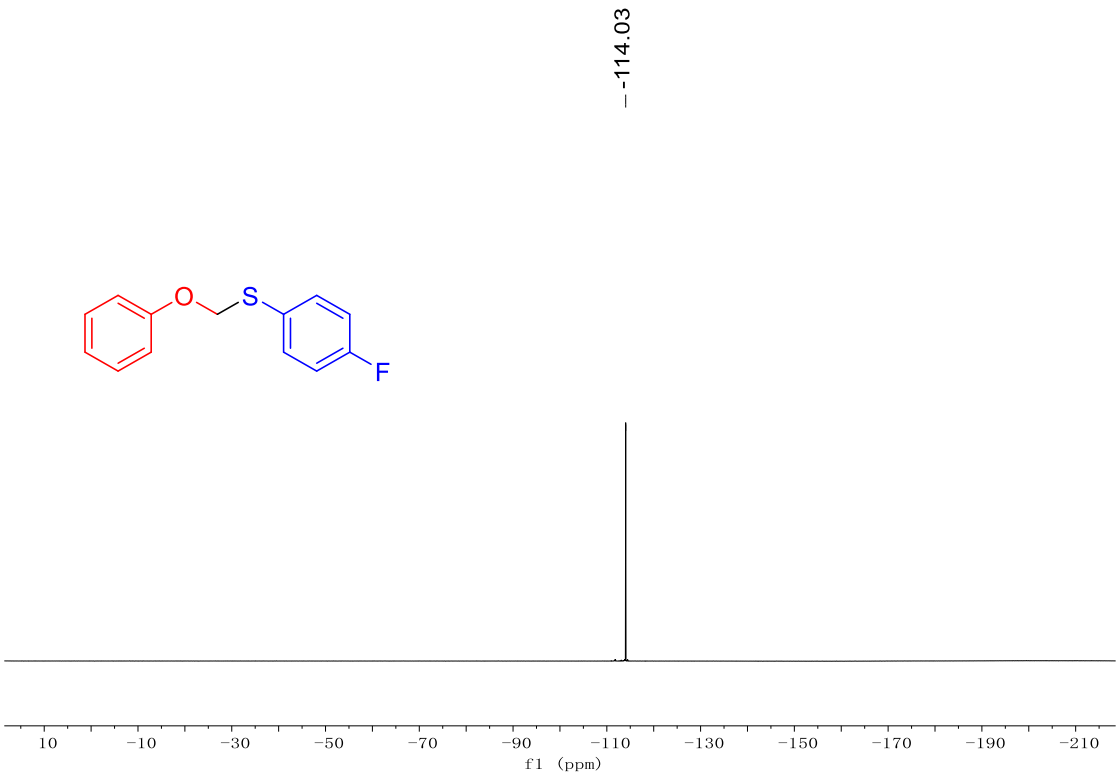
(4-Fluorophenyl)(phenoxymethyl)sulfane (3e).



^{1}H-NMR spectrum of **3e** (400 MHz, $CDCl_3$)

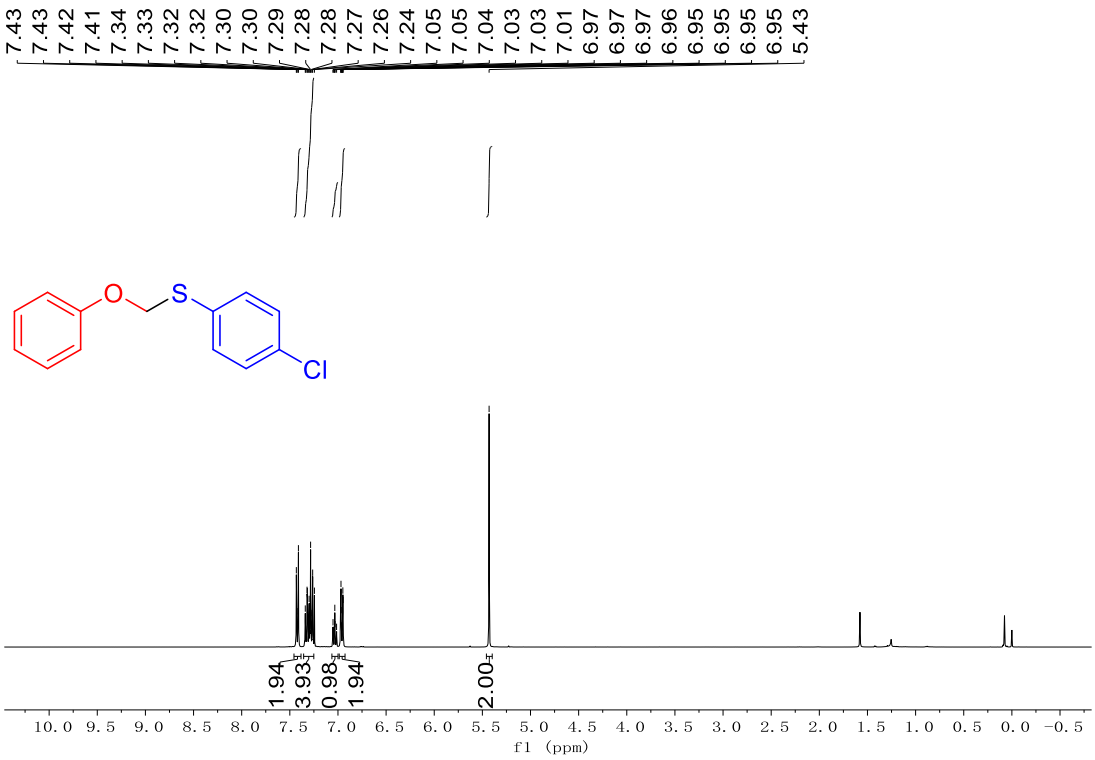


^{13}C-NMR spectrum of **3e** (100 MHz, $CDCl_3$)

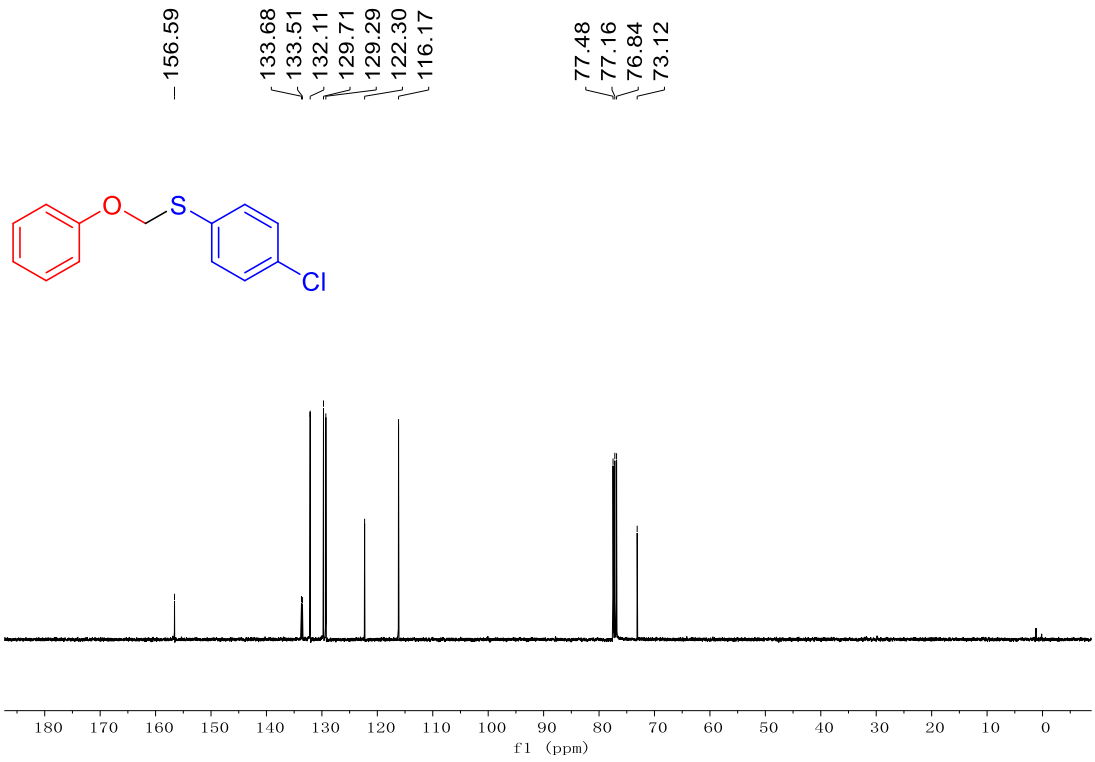


^{19}F-NMR spectrum of **3e** (377 MHz, $CDCl_3$)

(4-Chlorophenyl)(phenoxymethyl)sulfane (3f).

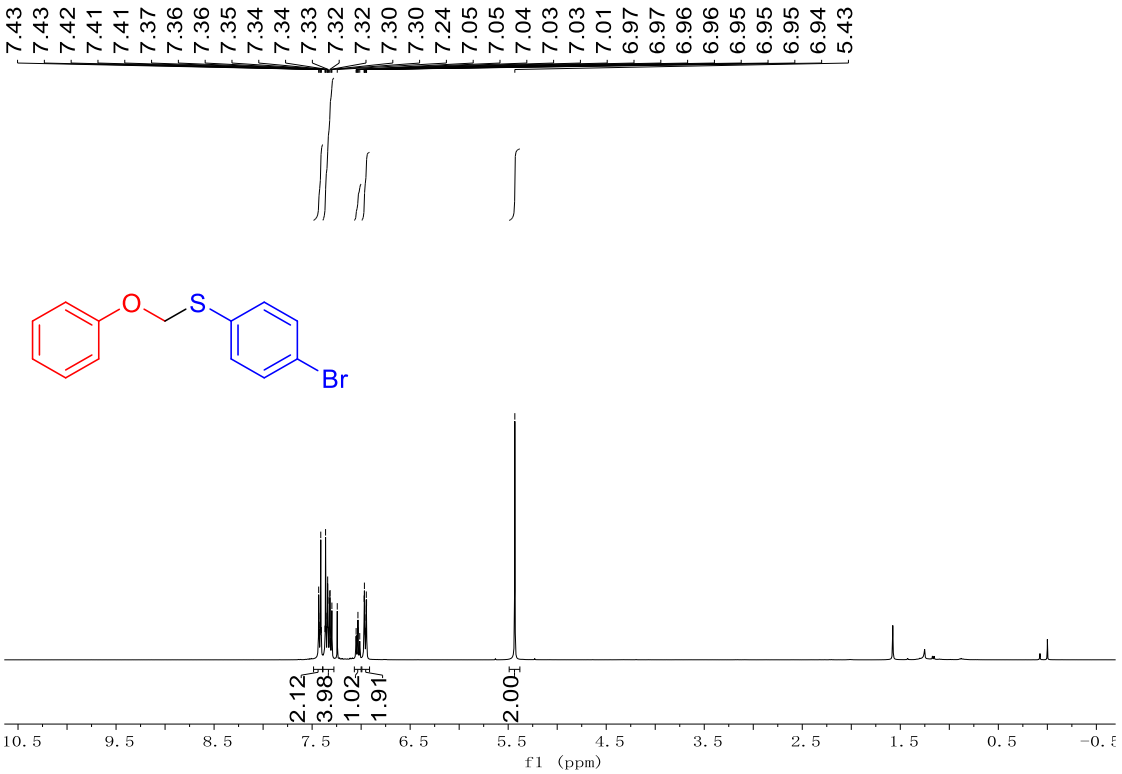


^{1}H-NMR spectrum of **3f** (400 MHz, $CDCl_3$)

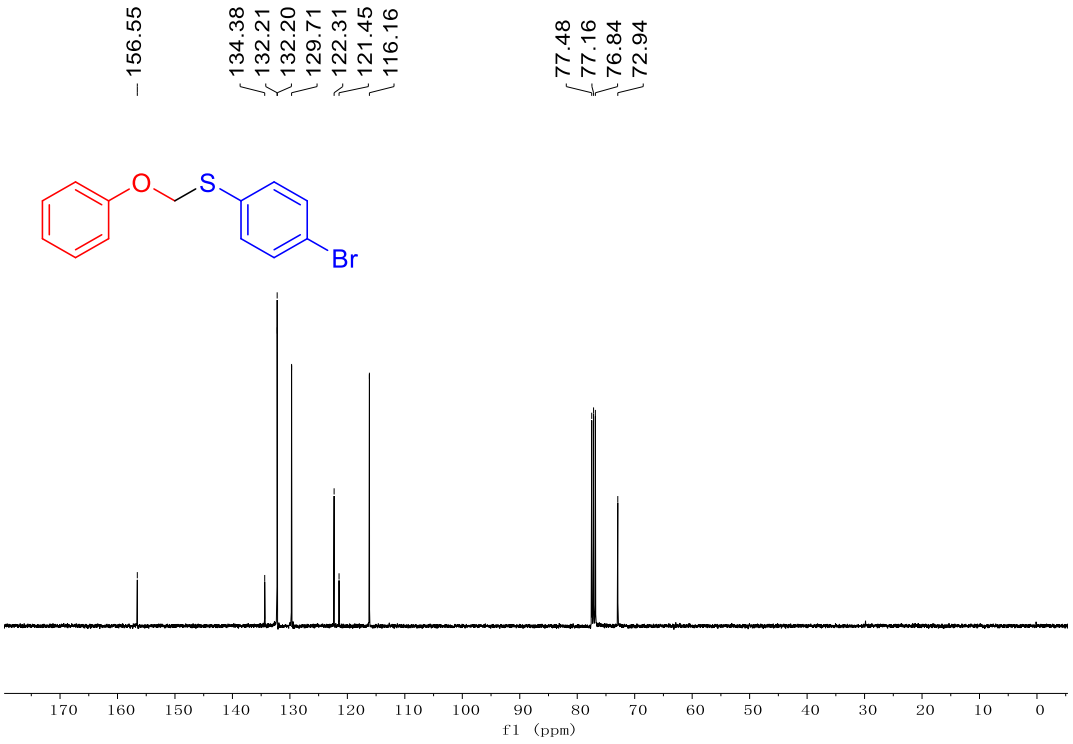


^{13}C-NMR spectrum of **3f** (100 MHz, $CDCl_3$)

(4-Bromophenyl)(phenoxymethyl)sulfane (3g).

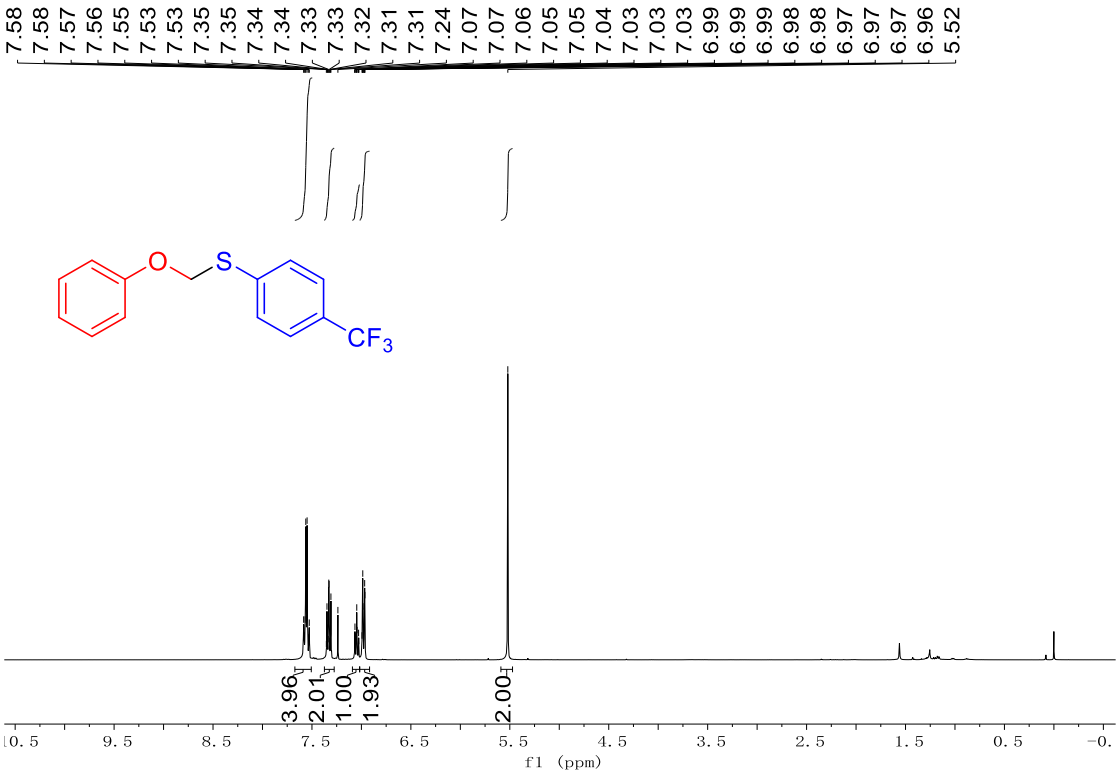


^{1}H-NMR spectrum of **3g** (400 MHz, $CDCl_3$)

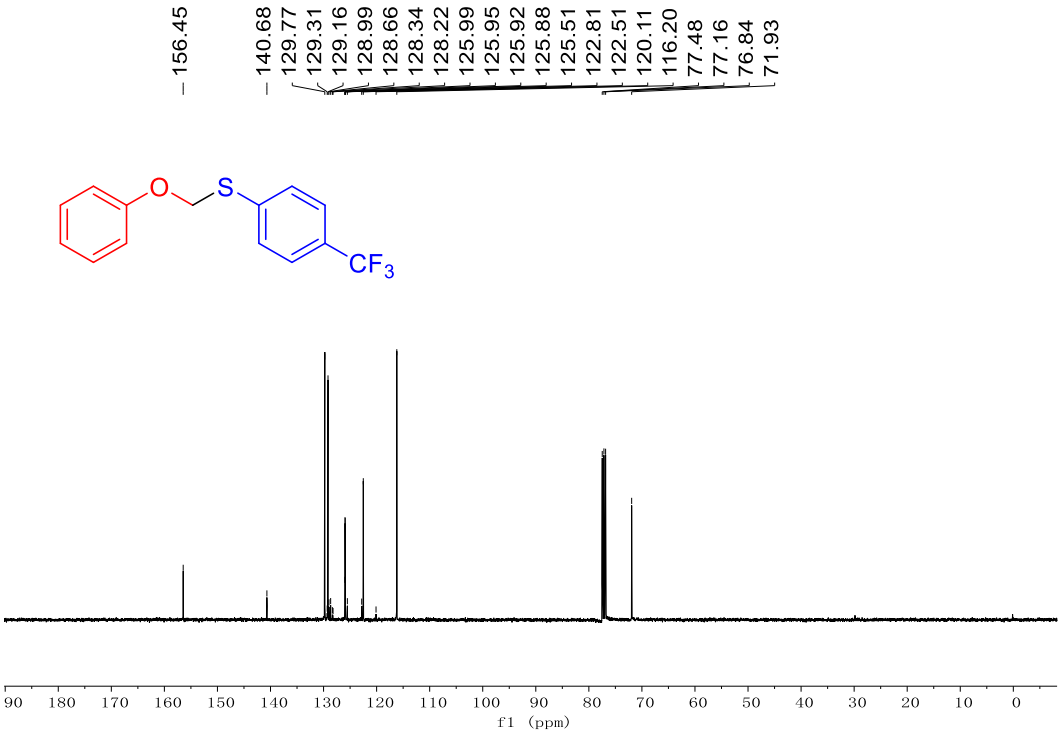


^{13}C-NMR spectrum of **3g** (100 MHz, $CDCl_3$)

(Phenoxymethyl)(4-(trifluoromethyl)phenyl)sulfane (3h).

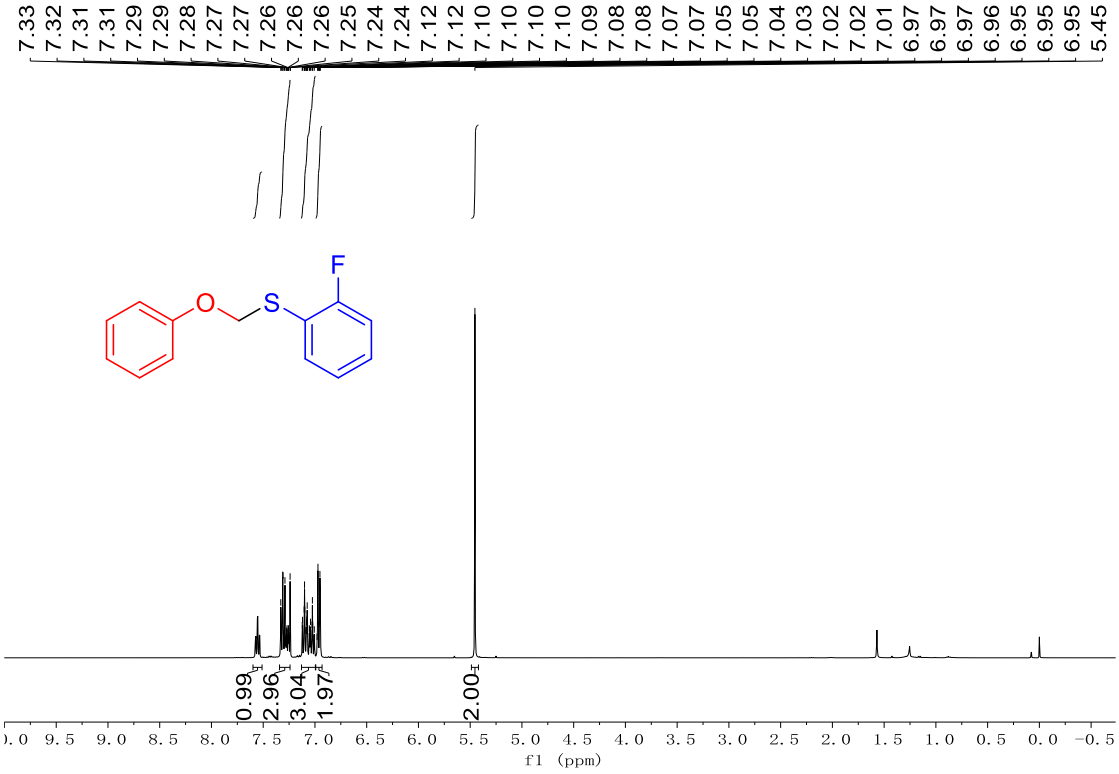


^{1}H-NMR spectrum of **3h** (400 MHz, $CDCl_3$)

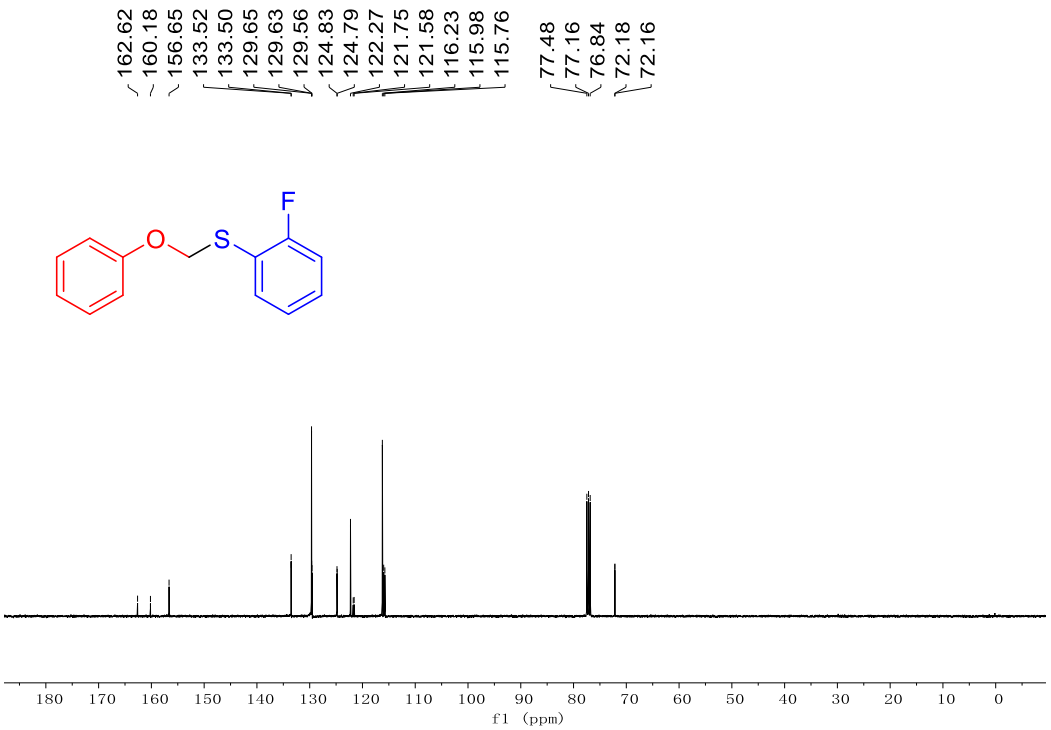


^{13}C-NMR spectrum of **3h** (100 MHz, $CDCl_3$)

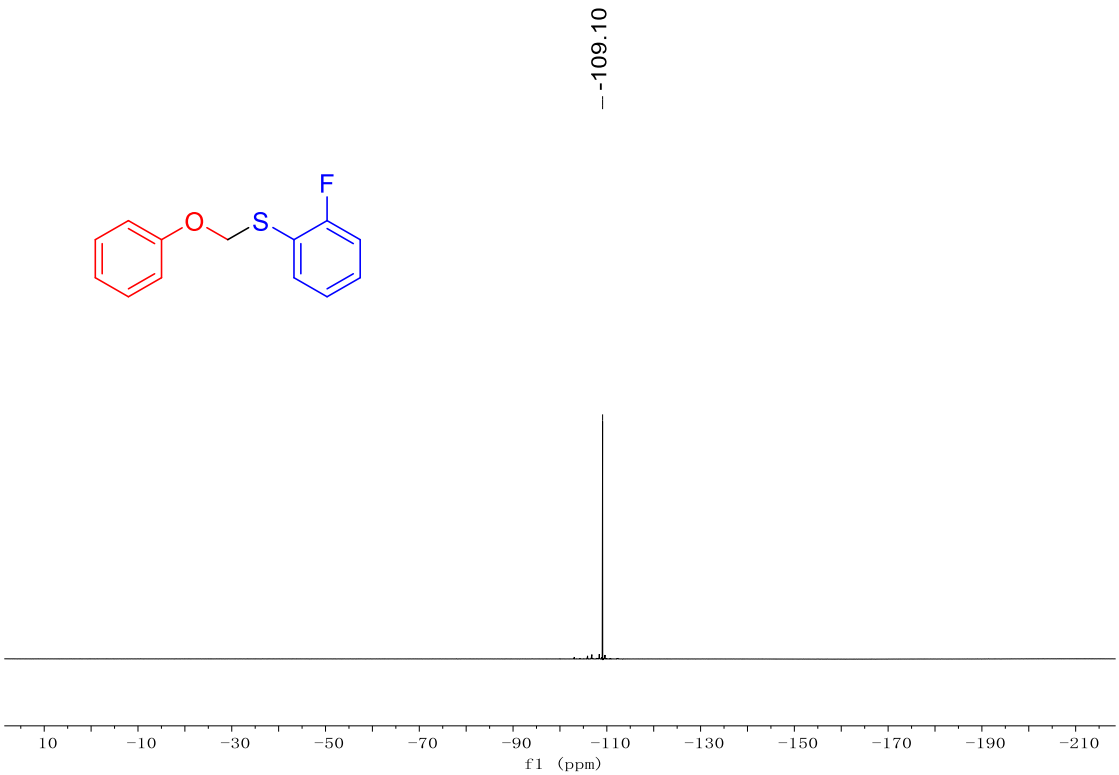
2-Fluorophenyl)(phenoxymethyl)sulfane (3i).



^{1}H-NMR spectrum of **3i** (400 MHz, $CDCl_3$)

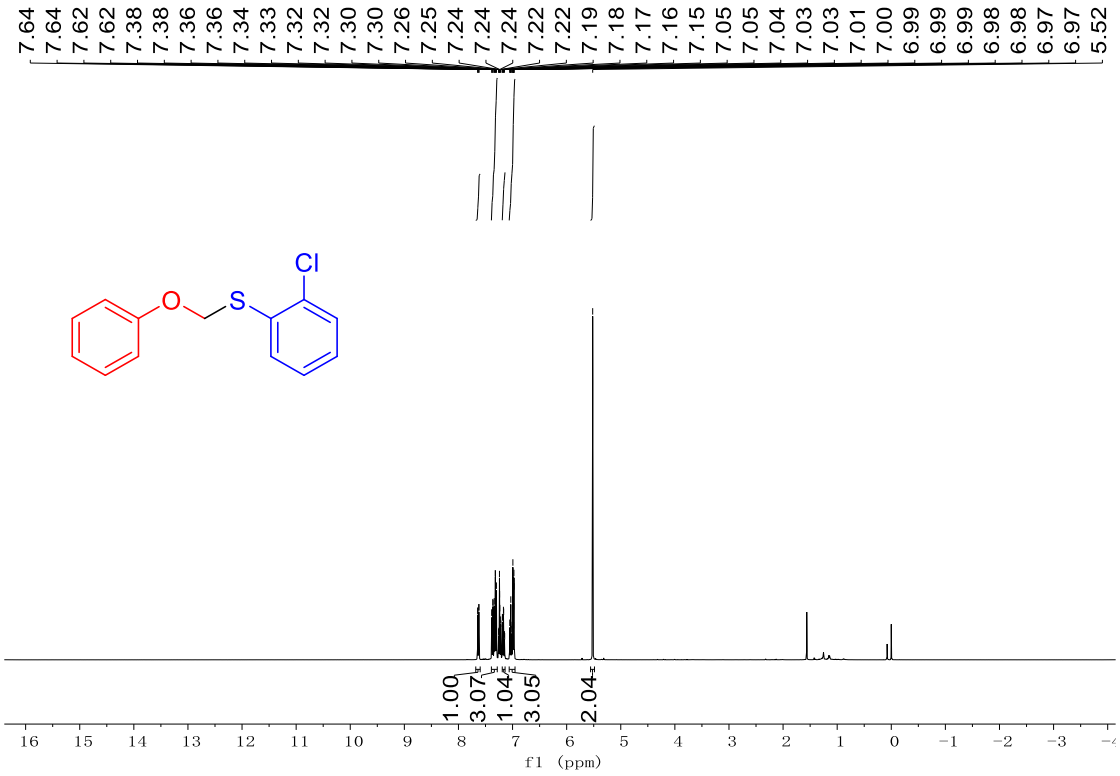


^{13}C-NMR spectrum of **3i** (100 MHz, $CDCl_3$)

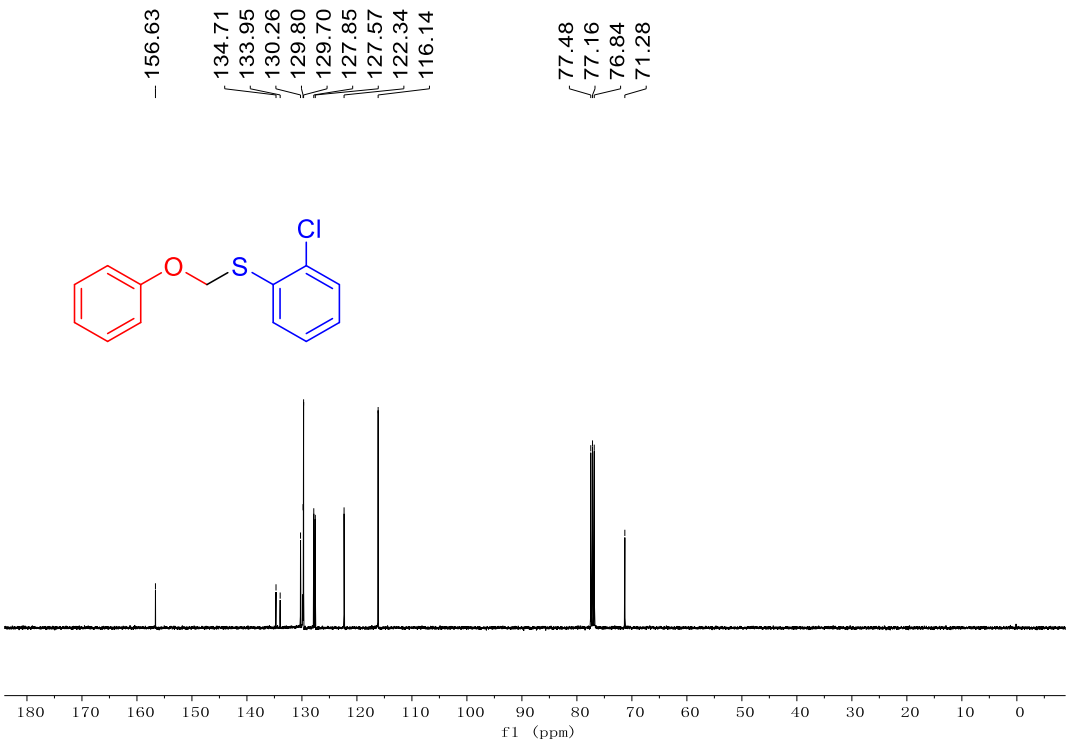


^{19}F-NMR spectrum of **3i** (377 MHz, $CDCl_3$)

(2-Chlorophenyl)(phenoxymethyl)sulfane (3j).

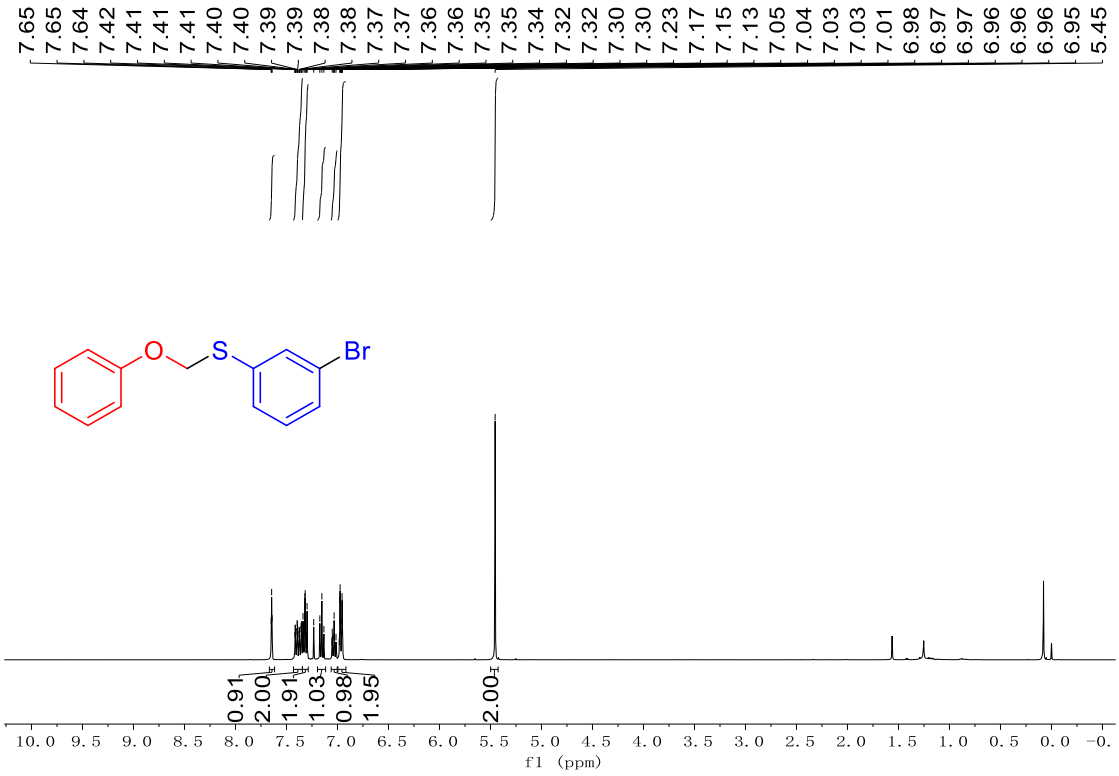


^{1}H-NMR spectrum of **3j** (400 MHz, $CDCl_3$)

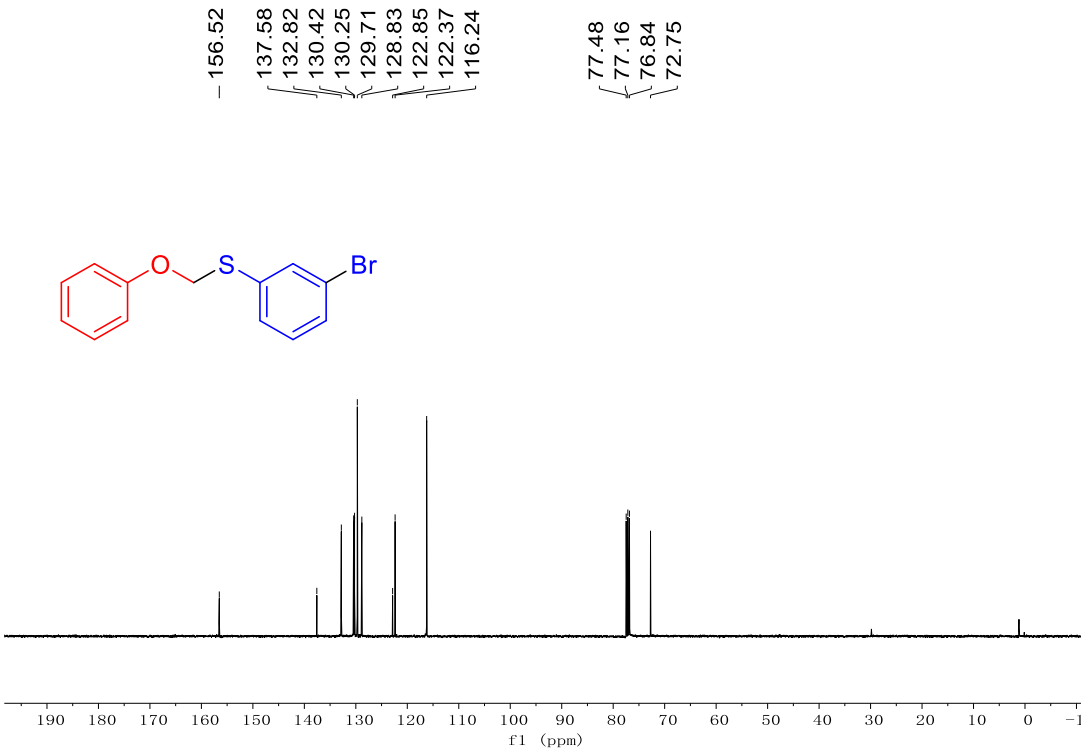


^{13}C-NMR spectrum of **3j** (100 MHz, $CDCl_3$)

(3-Bromophenyl)(phenoxymethyl)sulfane (3k).

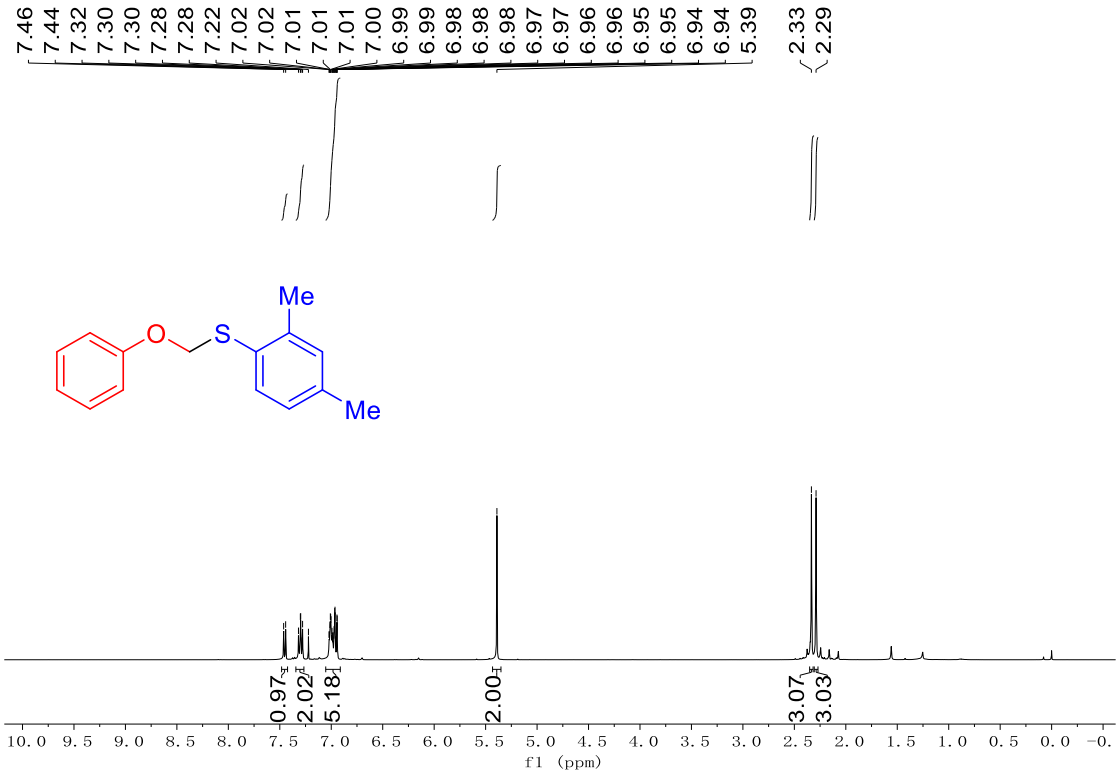


^{1}H-NMR spectrum of **3k** (400 MHz, $CDCl_3$)

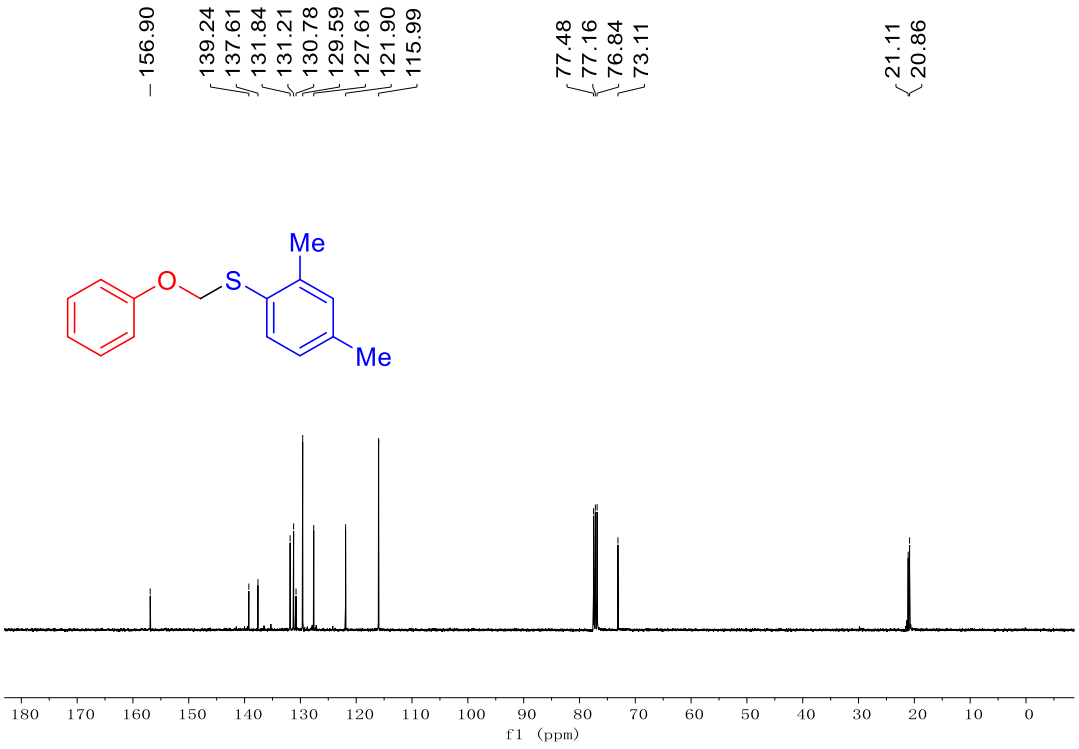


^{13}C-NMR spectrum of **3k** (100 MHz, $CDCl_3$)

(2,4-Dimethylphenyl)(phenoxymethyl)sulfane (3l).

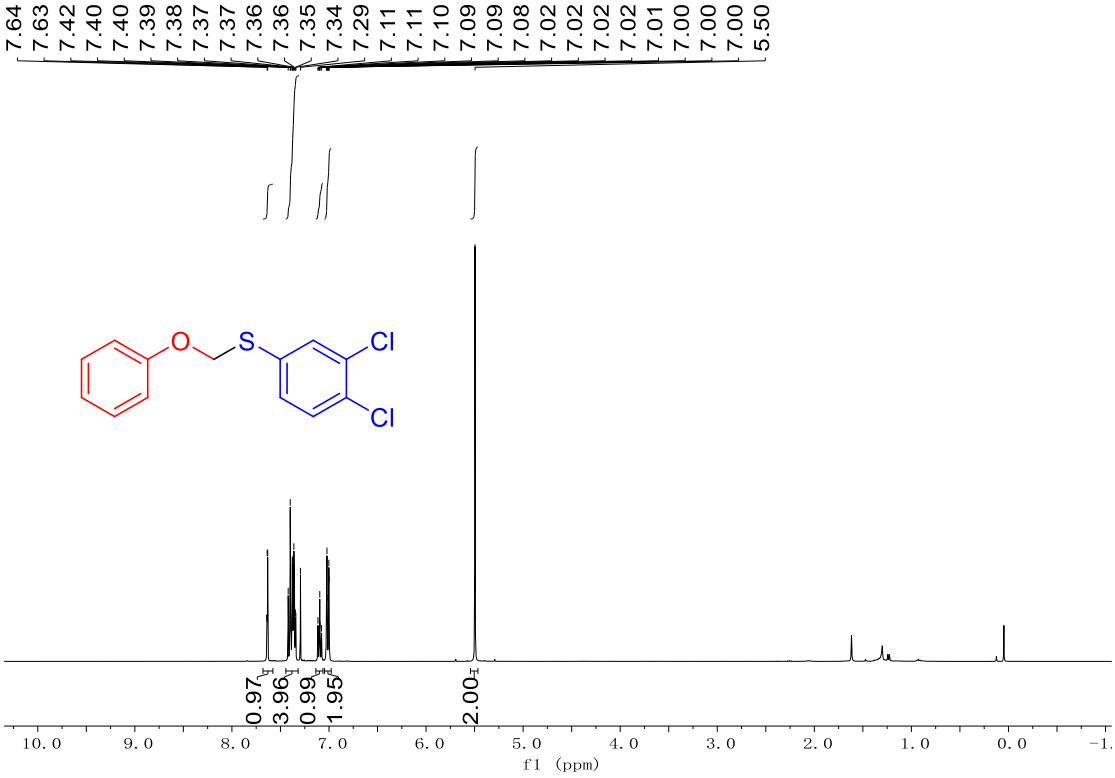


^{1}H-NMR spectrum of **3l** (400 MHz, $CDCl_3$)

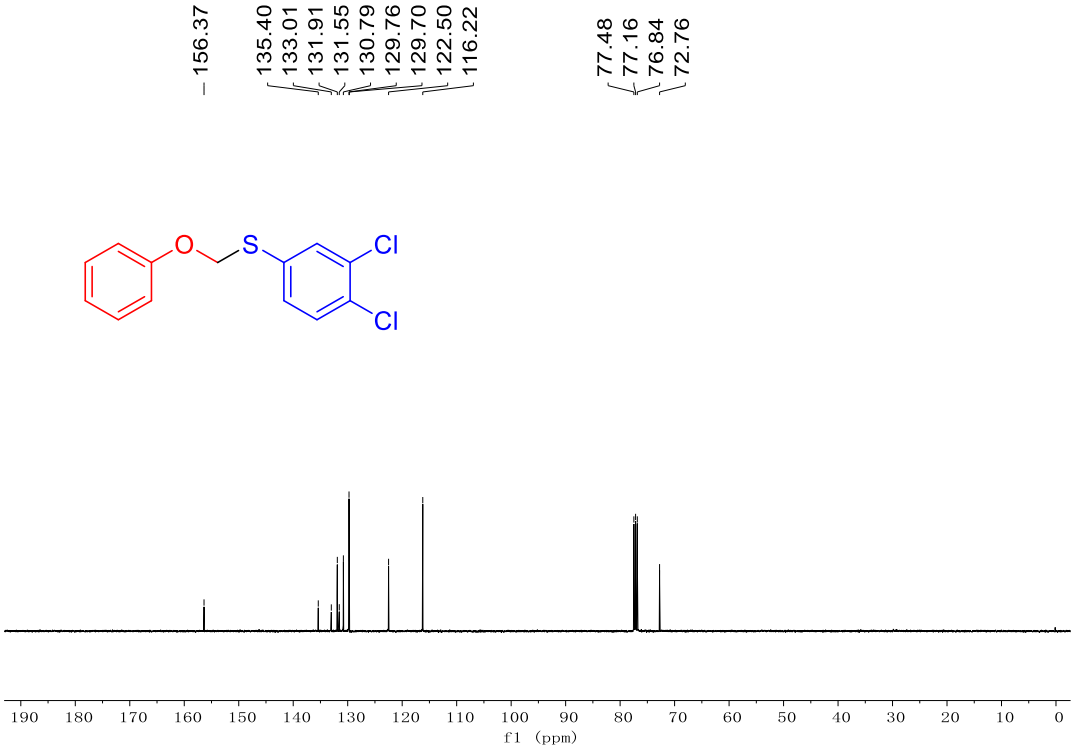


^{13}C-NMR spectrum of **3l** (100 MHz, $CDCl_3$)

(3,4-Dichlorophenyl)(phenoxymethyl)sulfane (3m).

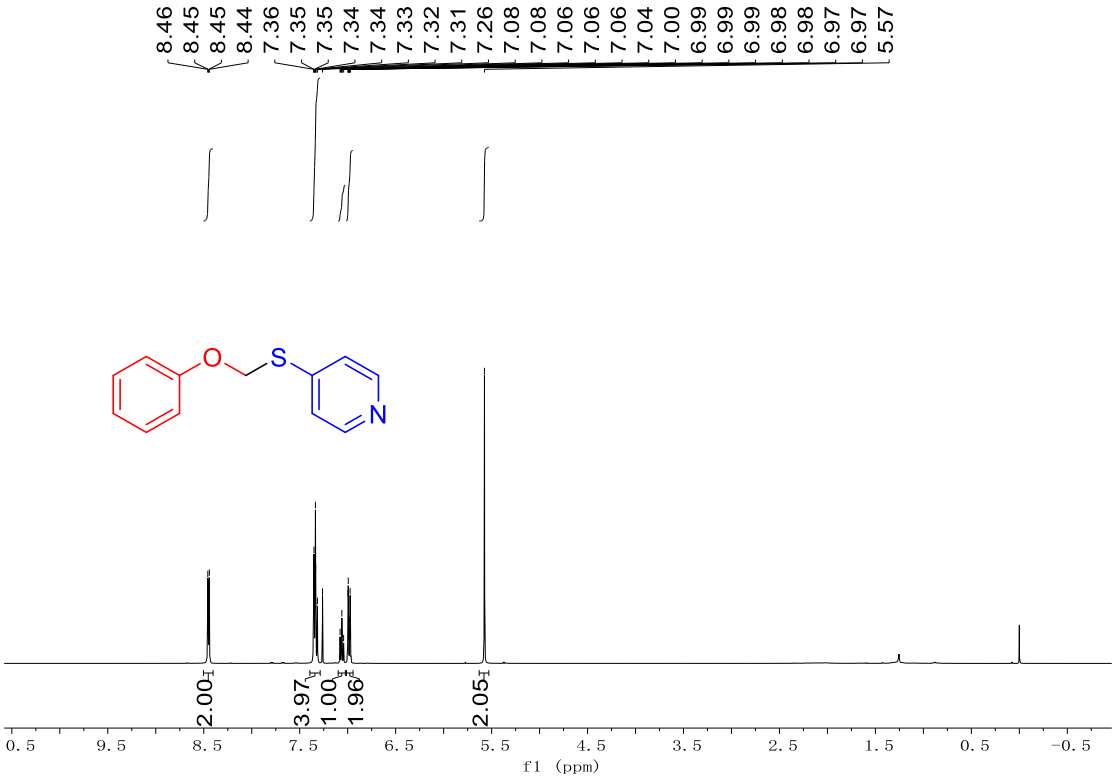


^{1}H-NMR spectrum of **3m** (400 MHz, $CDCl_3$)

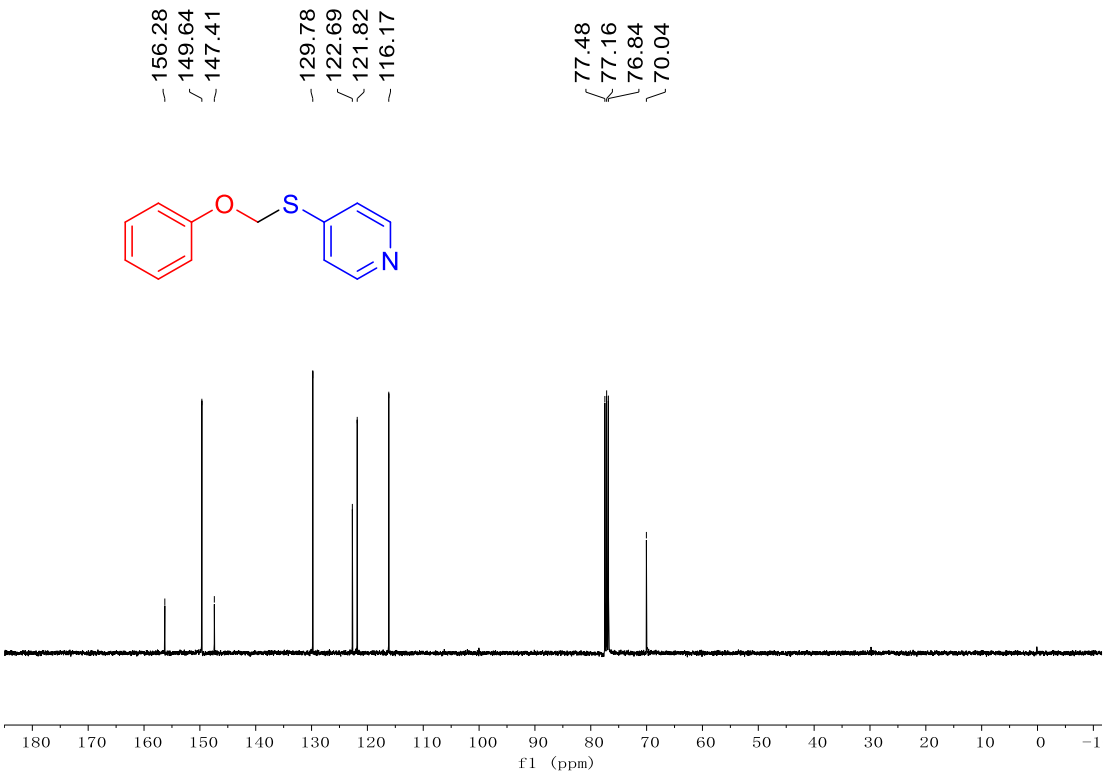


^{13}C-NMR spectrum of **3m** (100 MHz, $CDCl_3$)

4-((Phenoxymethyl)thio)pyridine (3n).

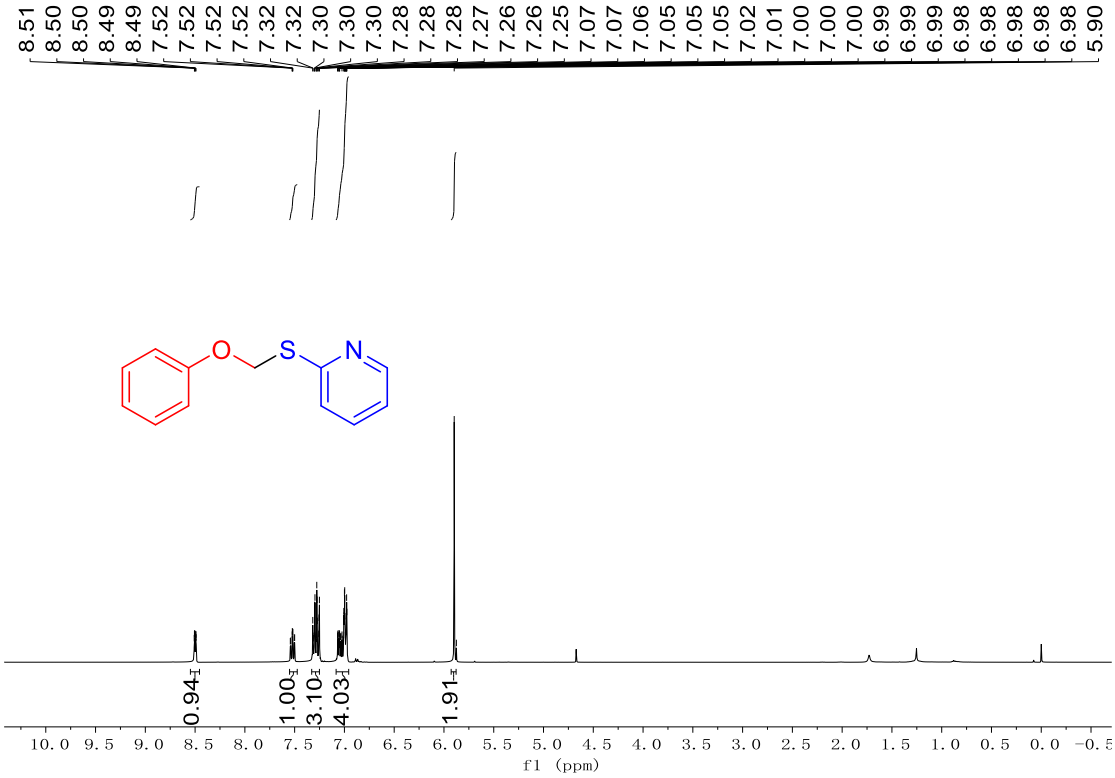


^{1}H-NMR spectrum of **3n** (400 MHz, $CDCl_3$)

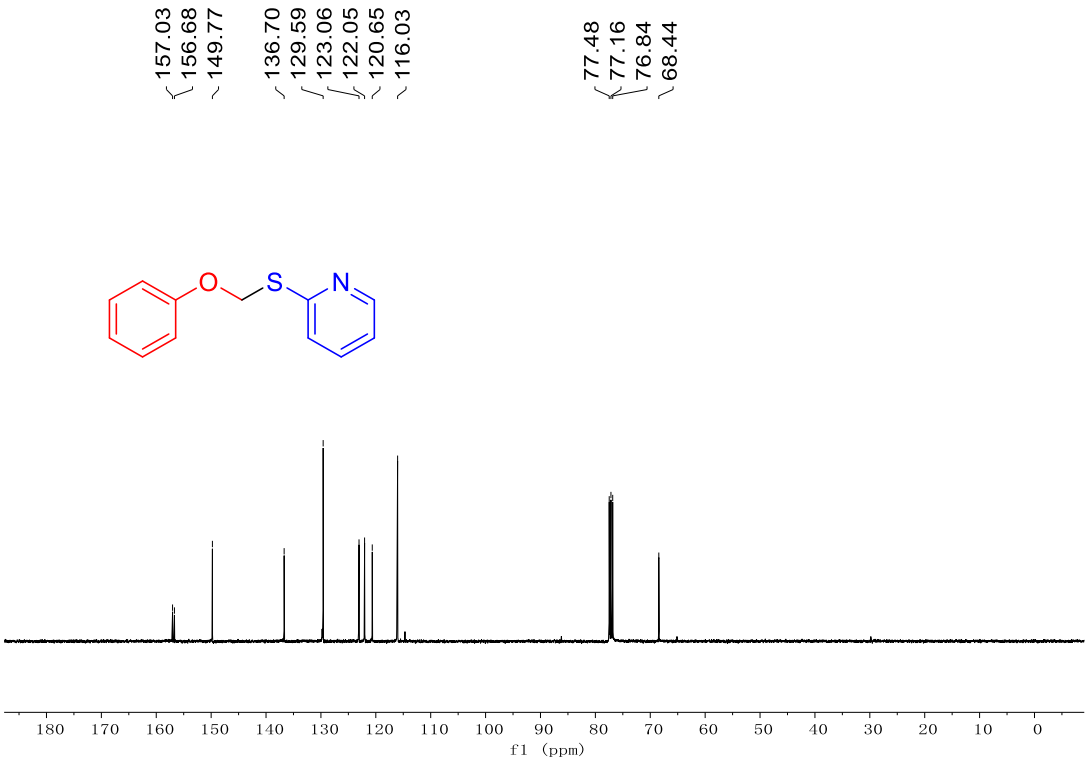


^{13}C-NMR spectrum of **3n** (100 MHz, $CDCl_3$)

2-((Phenoxymethyl)thio)pyridine (3o).

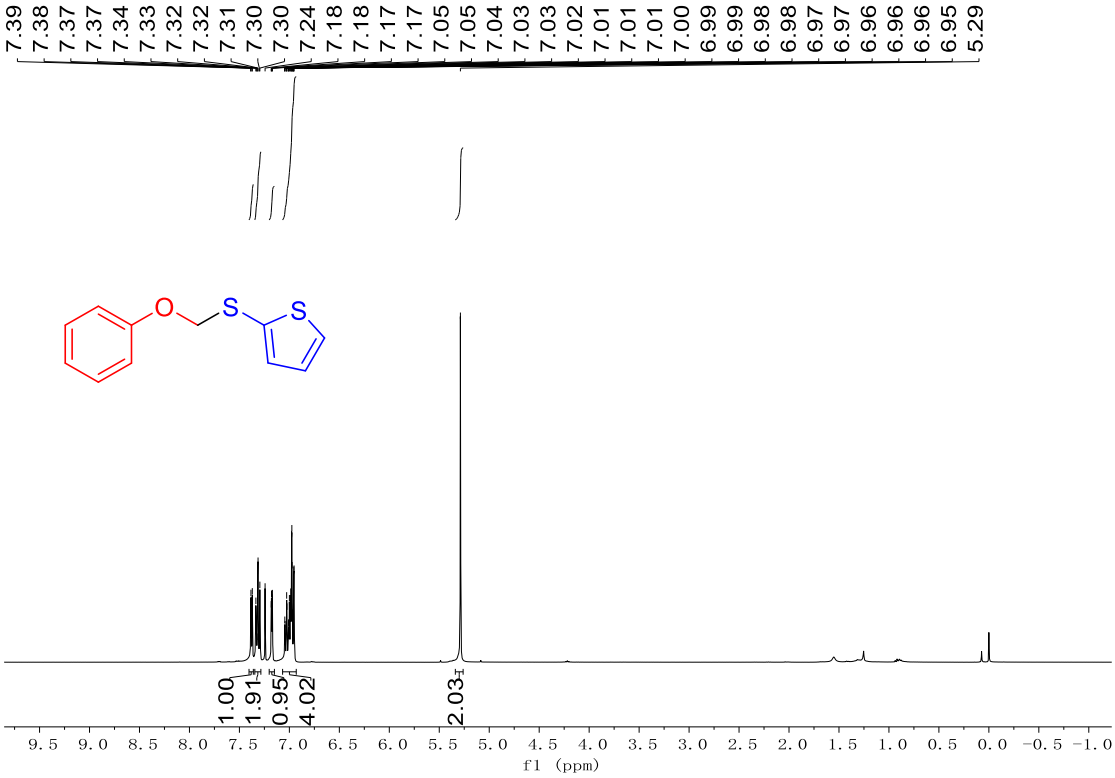


^{1}H-NMR spectrum of **3o** (400 MHz, $CDCl_3$)

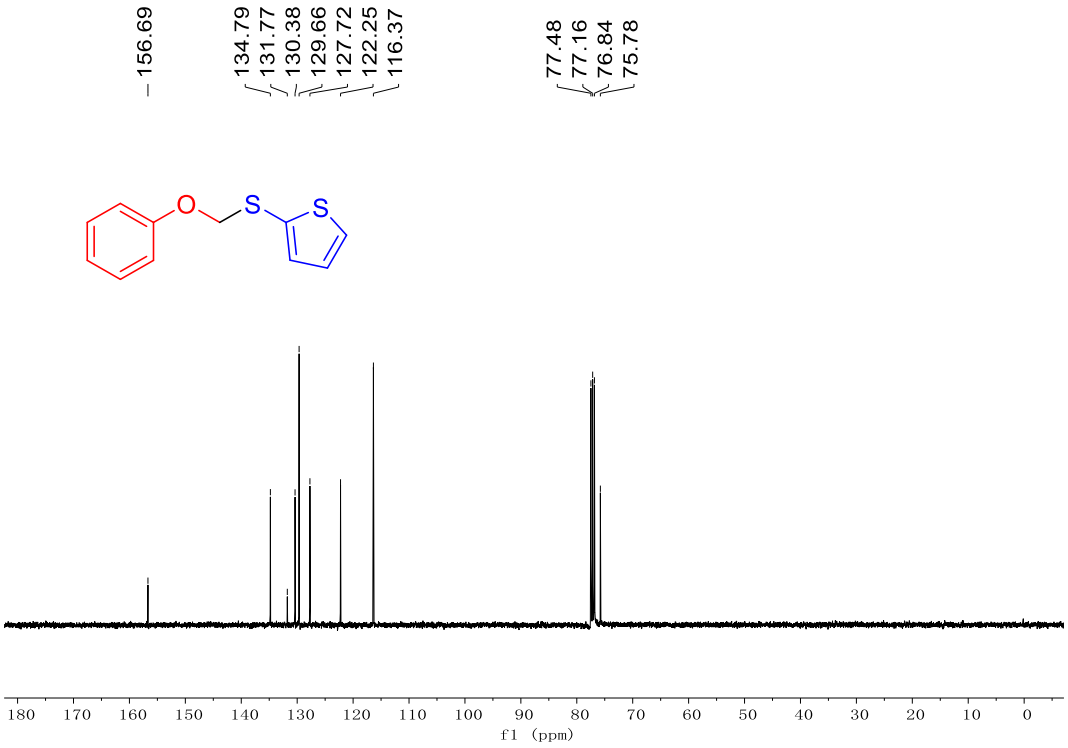


^{13}C-NMR spectrum of **3o** (100 MHz, $CDCl_3$)

2-(3-Phenylpropyl)thiophene (3p).

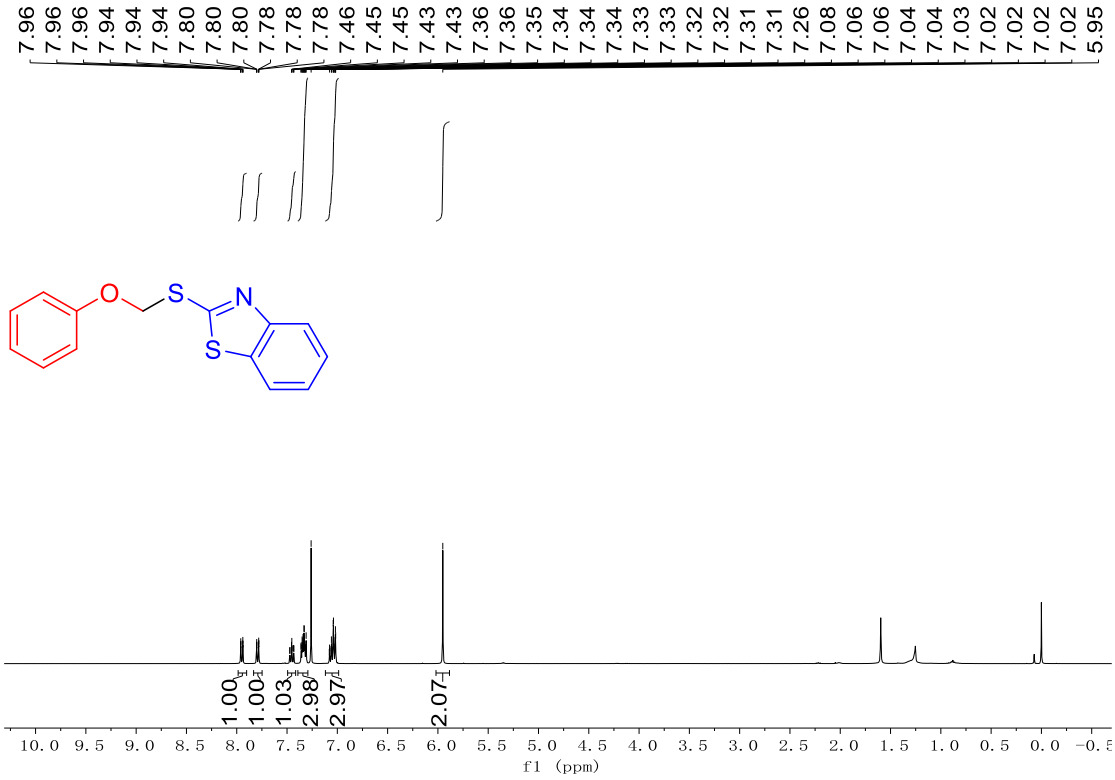


^{1}H-NMR spectrum of **3p** (400 MHz, $CDCl_3$)

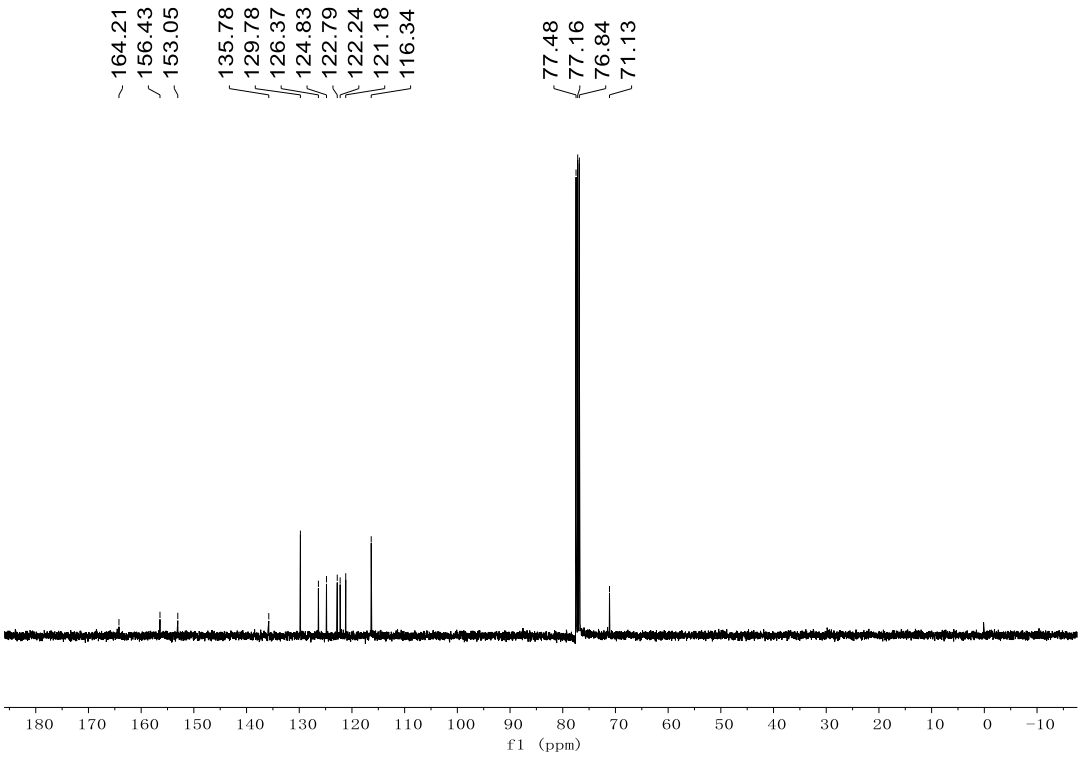


^{13}C-NMR spectrum of **3p** (100 MHz, $CDCl_3$)

(2,4-Dimethylphenyl)(phenoxymethyl)sulfane (3q).

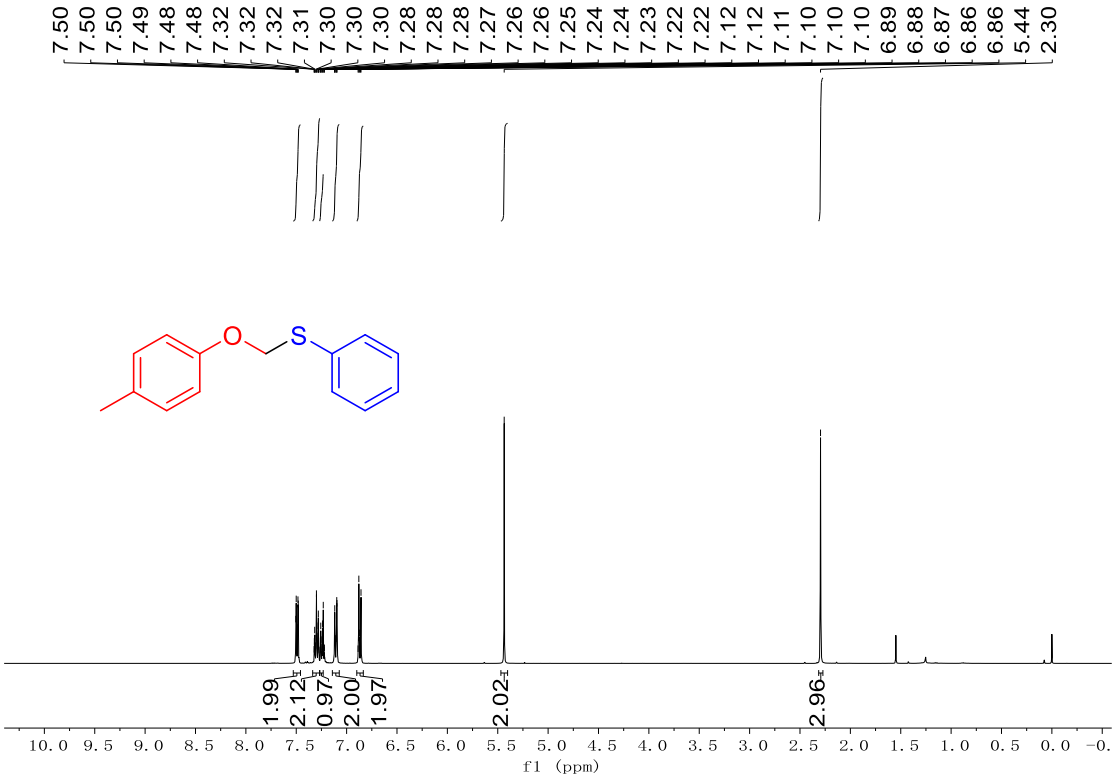


^{1}H-NMR spectrum of **3q** (400 MHz, $CDCl_3$)

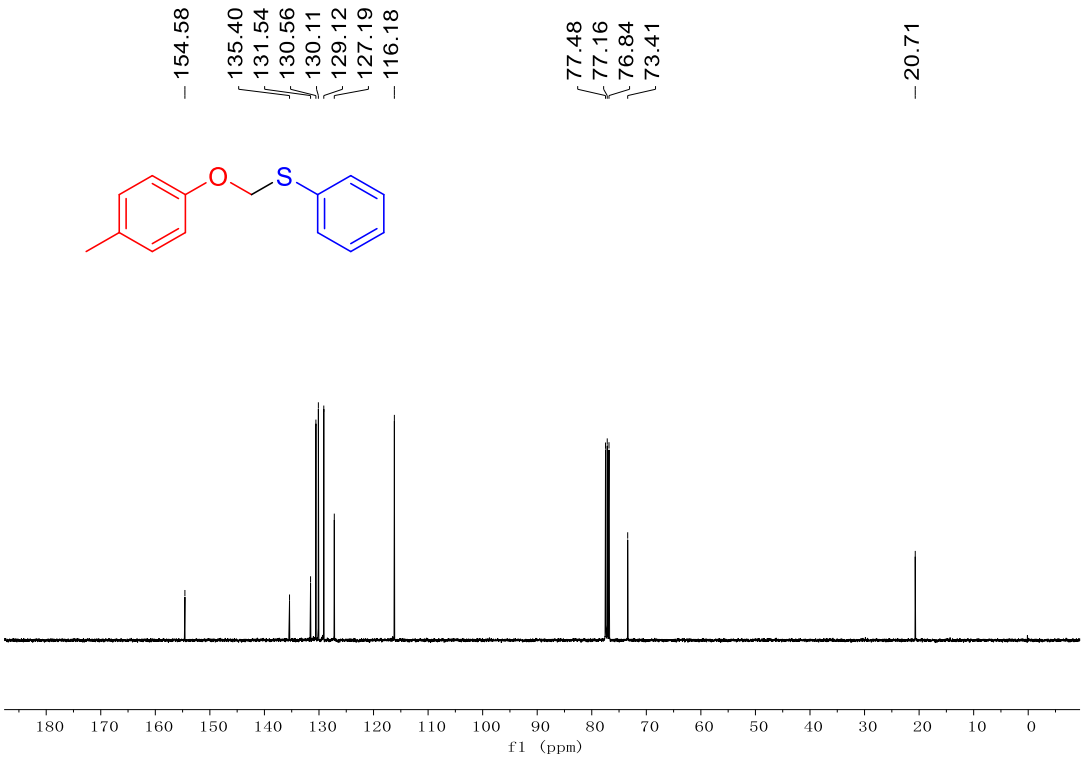


^{13}C-NMR spectrum of **3q** (100 MHz, $CDCl_3$)

Phenyl((p-tolyloxy)methyl)sulfane (4a).

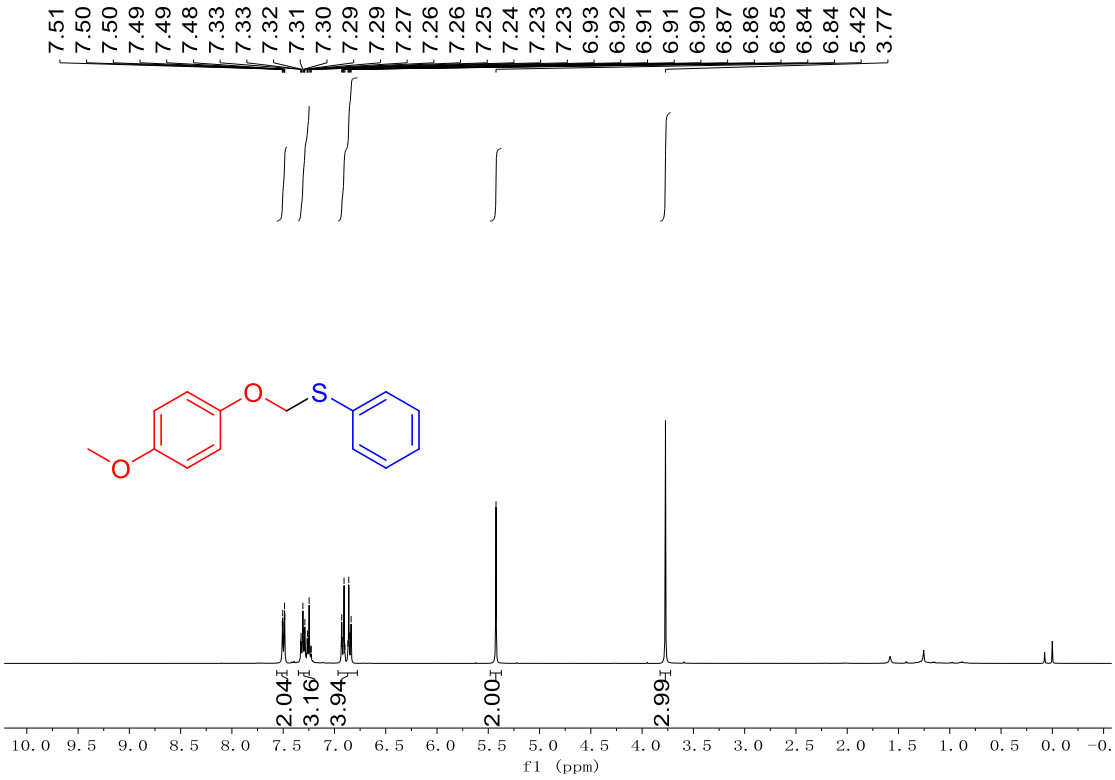


^{1}H-NMR spectrum of **4a** (400 MHz, $CDCl_3$)

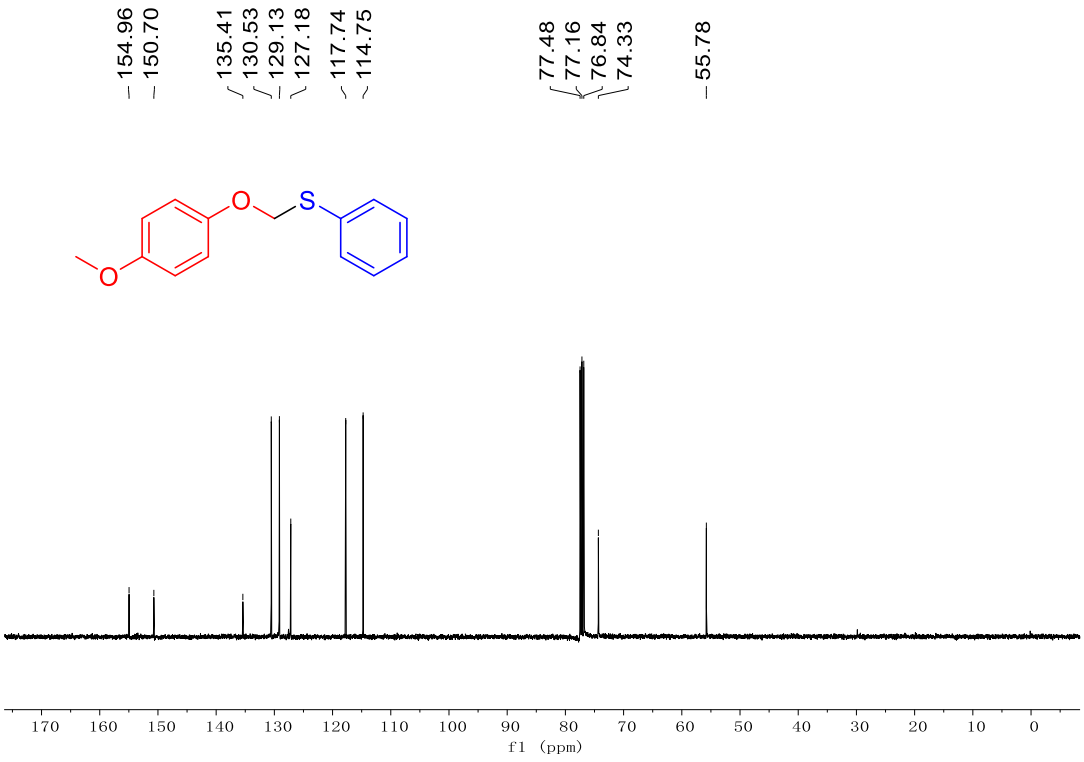


^{13}C-NMR spectrum of **4a** (100 MHz, $CDCl_3$)

((4-Methoxyphenoxy)methyl)(phenyl)sulfane (4b).

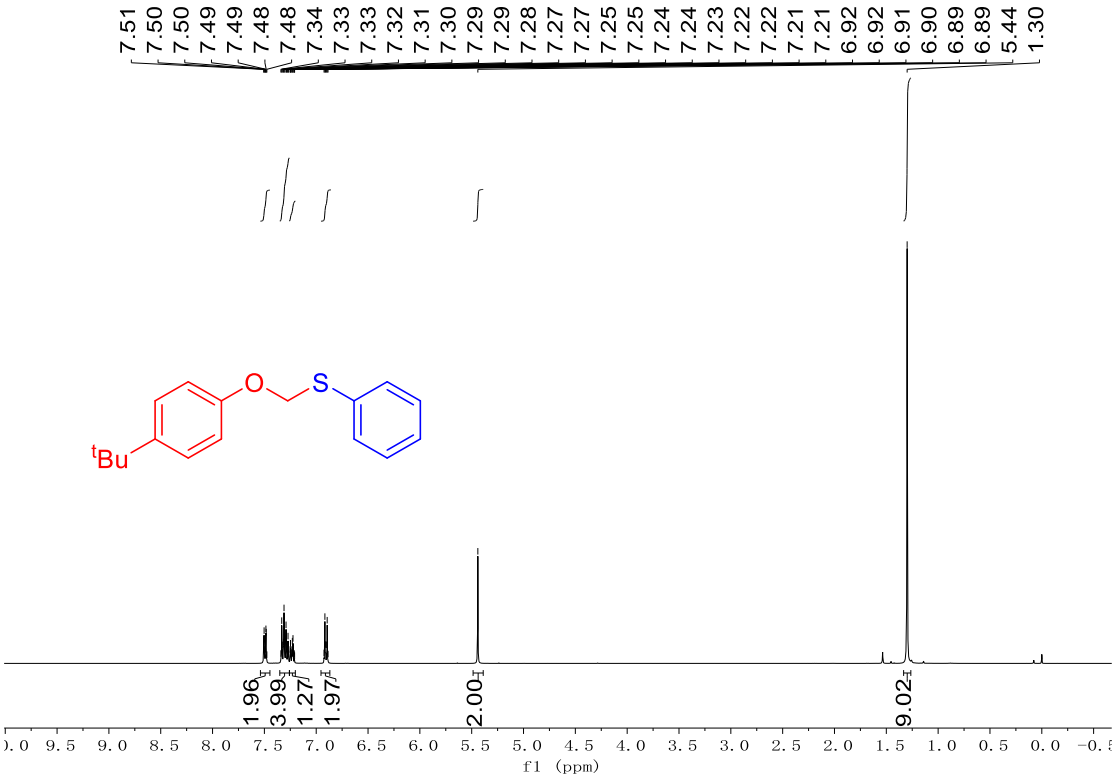


^{1}H-NMR spectrum of **4b** (400 MHz, $CDCl_3$)

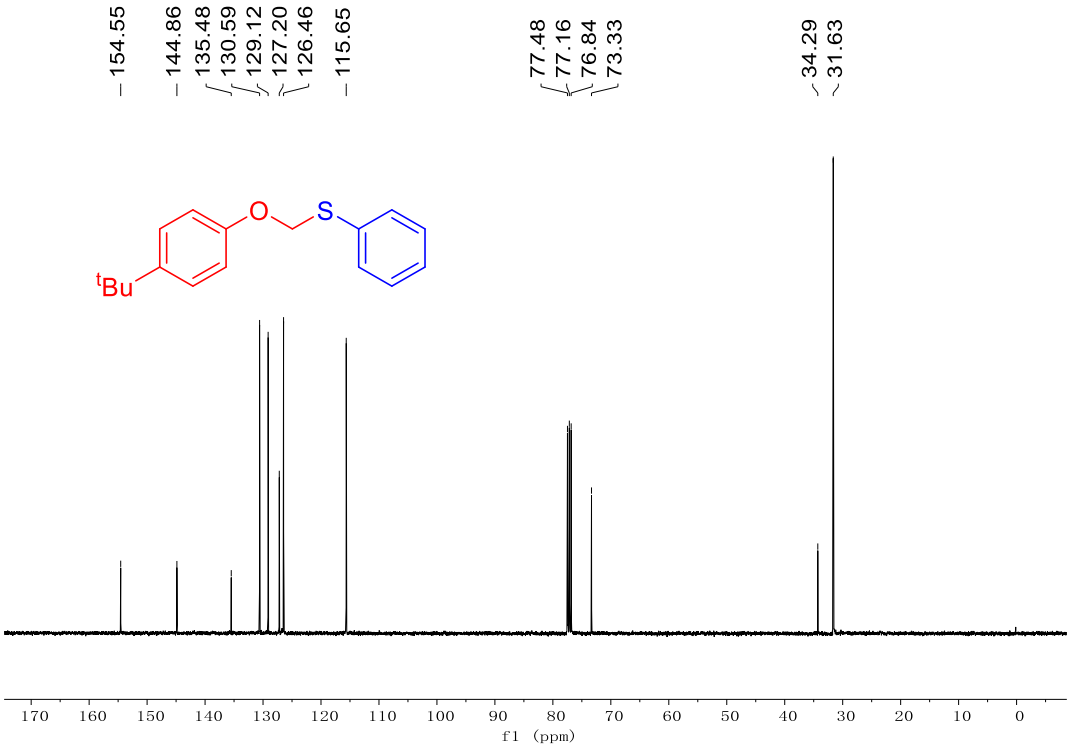


^{13}C-NMR spectrum of **4b** (100 MHz, $CDCl_3$)

((4-(Tert-butyl)phenoxy)methyl)(phenyl)sulfane (4c).

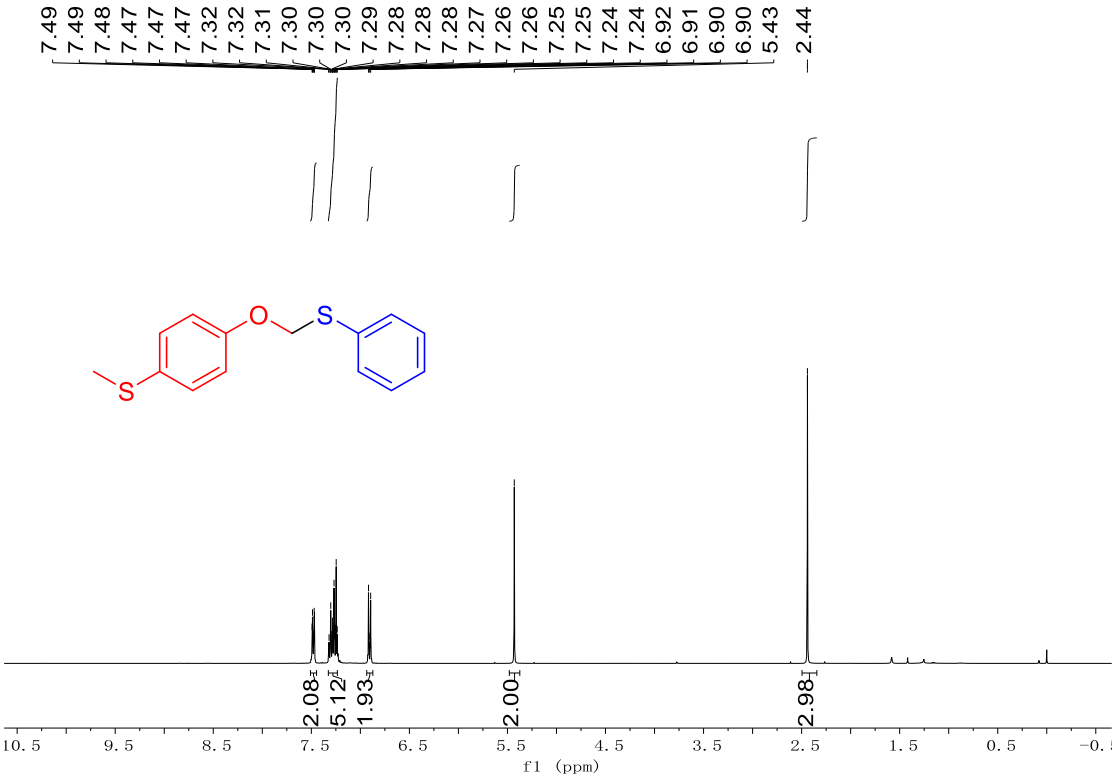


^{1}H-NMR spectrum of **4c** (400 MHz, $CDCl_3$)

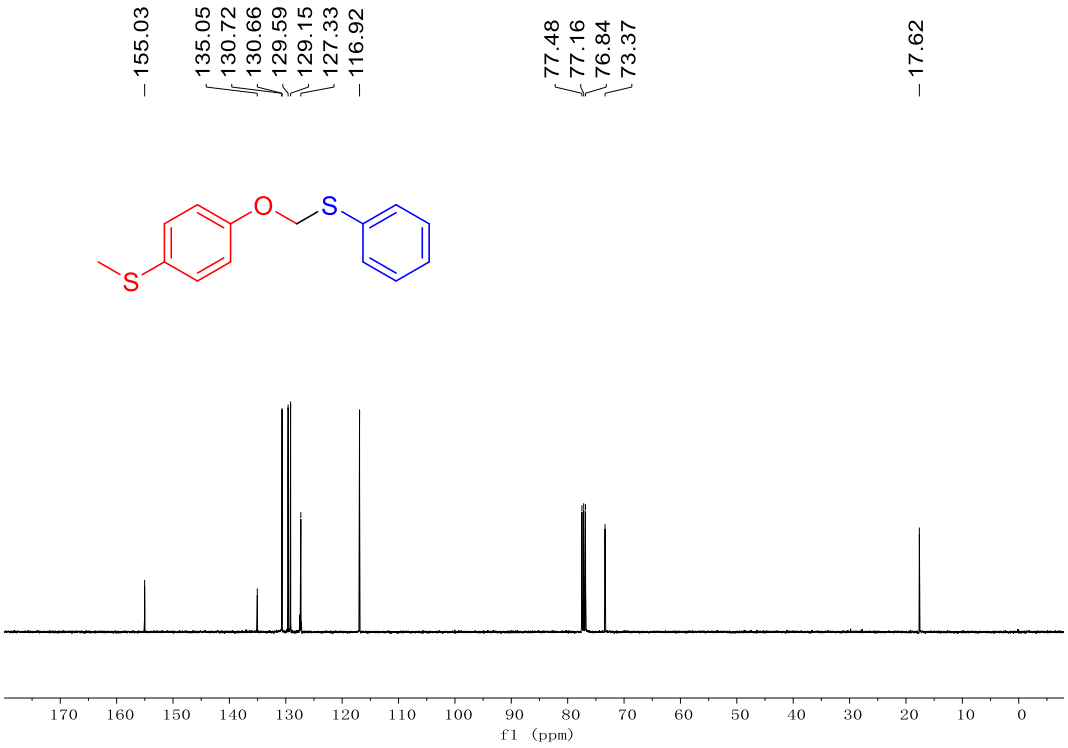


^{13}C-NMR spectrum of **4c** (100 MHz, $CDCl_3$)

Methyl(4-((phenylthio)methoxy)phenyl)sulfane (4d).

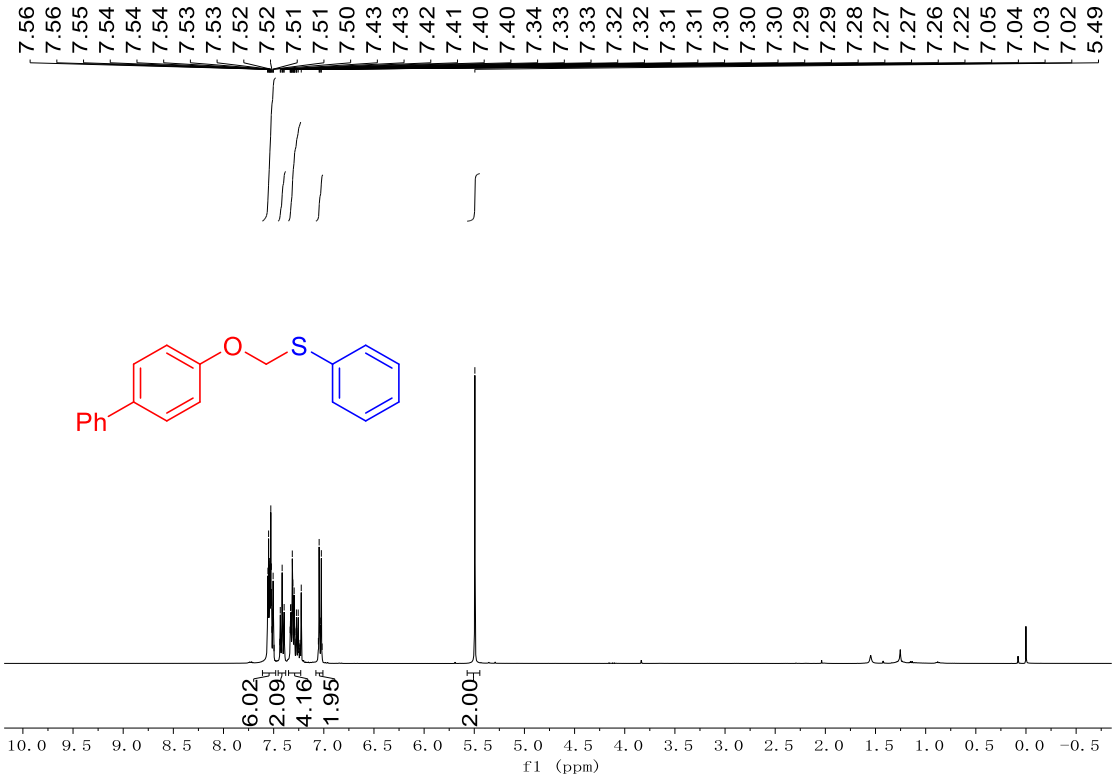


^{1}H-NMR spectrum of **4d** (400 MHz, $CDCl_3$)

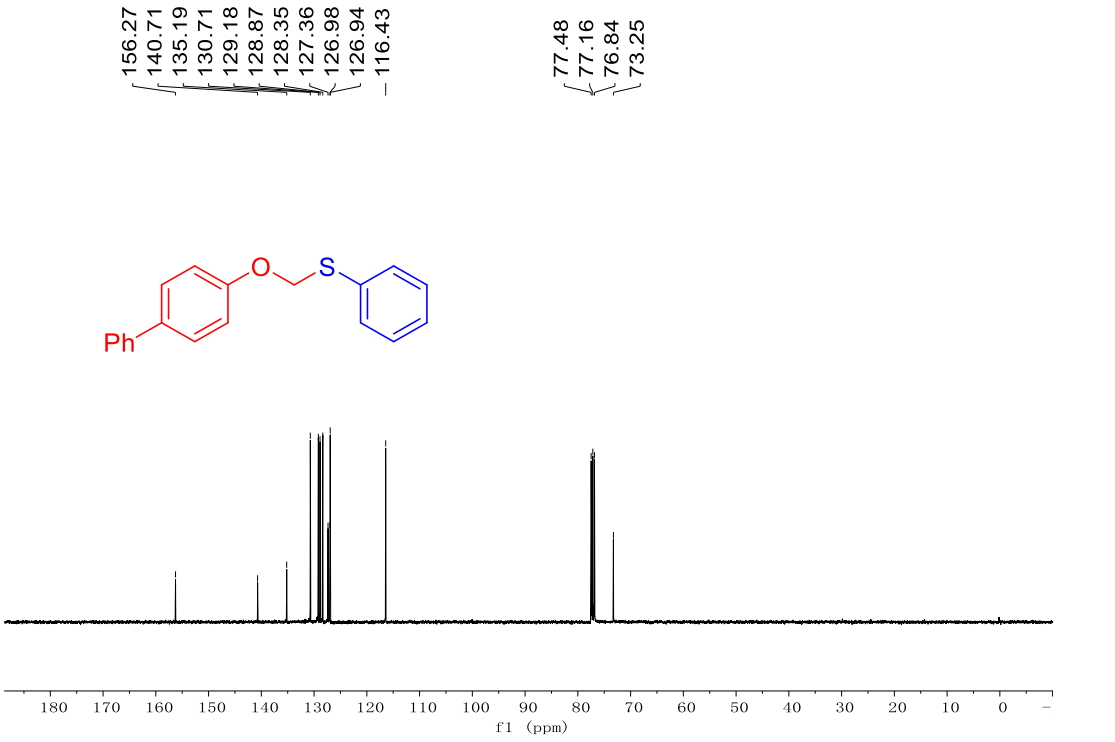


^{13}C-NMR spectrum of **4d** (100 MHz, $CDCl_3$)

(([1,1'-Biphenyl]-4-yloxy)methyl)(phenyl)sulfane (4e).

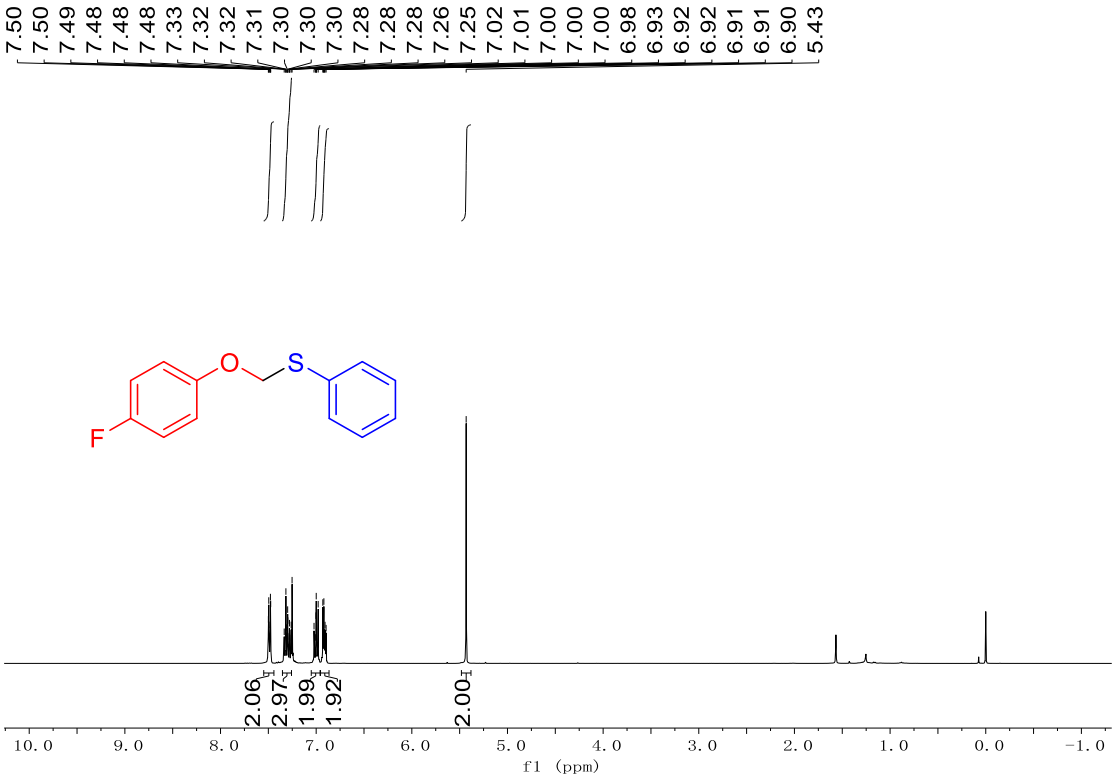


^{1}H-NMR spectrum of **4e** (400 MHz, $CDCl_3$)

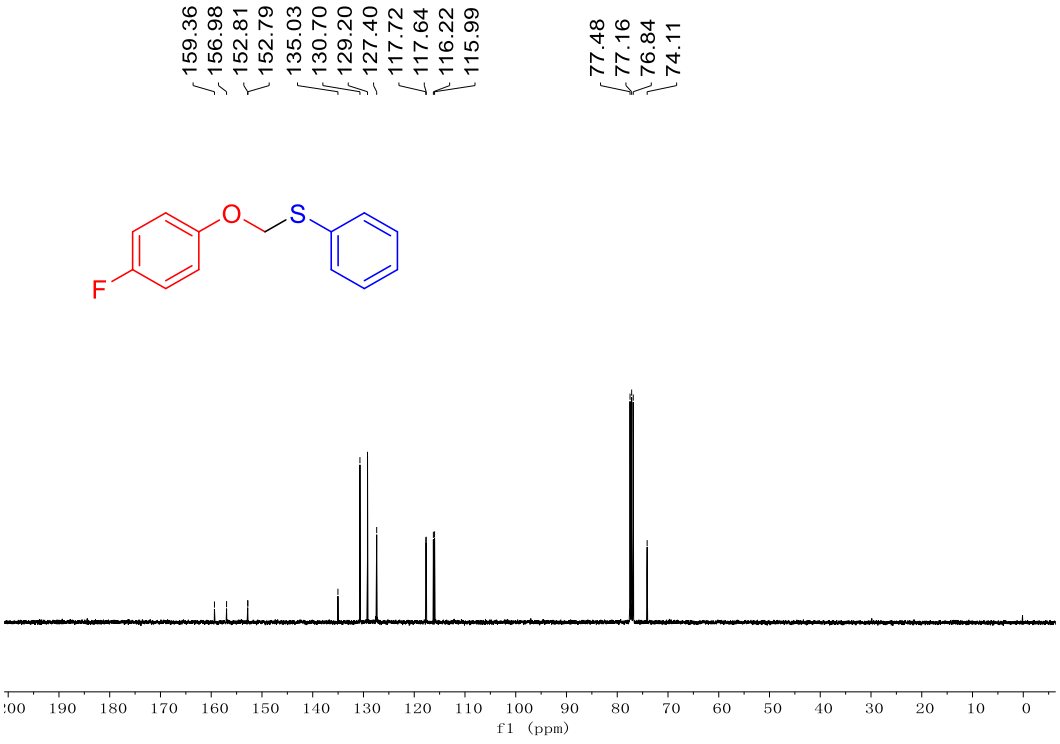


^{13}C-NMR spectrum of **4e** (100 MHz, $CDCl_3$)

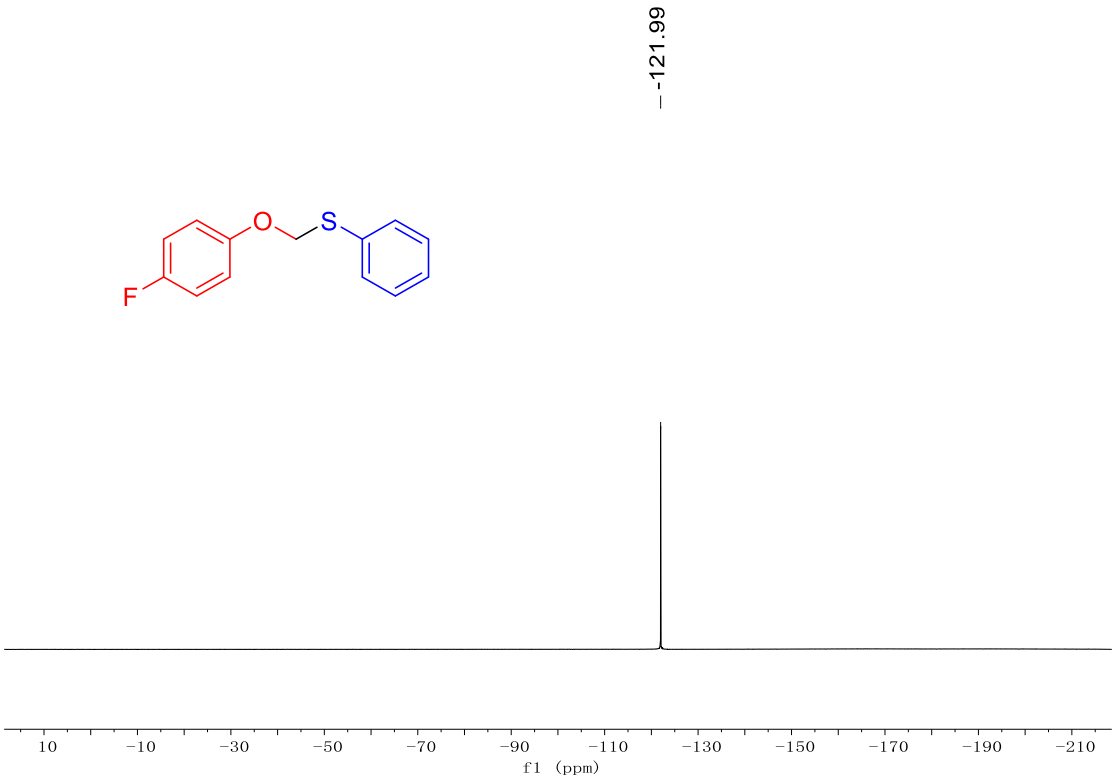
((4-Fluorophenoxy)methyl)(phenyl)sulfane (4f).



^{1}H-NMR spectrum of **4f** (400 MHz, $CDCl_3$)

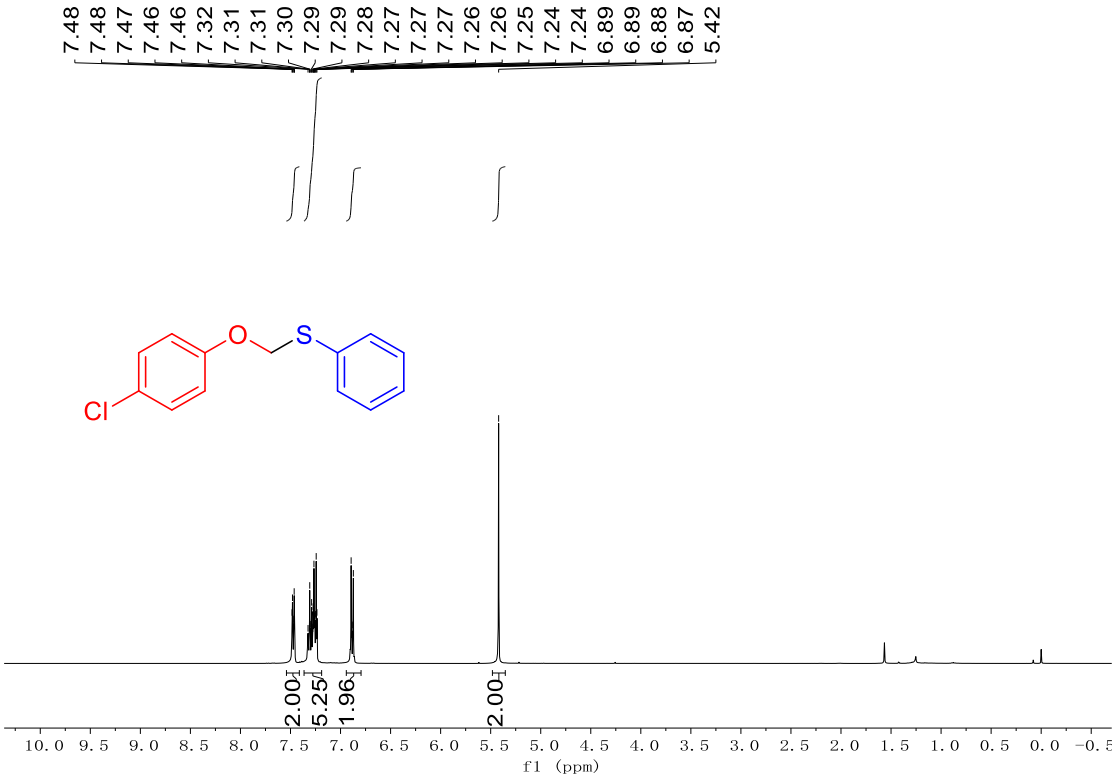


^{13}C-NMR spectrum of **4f** (100 MHz, $CDCl_3$)

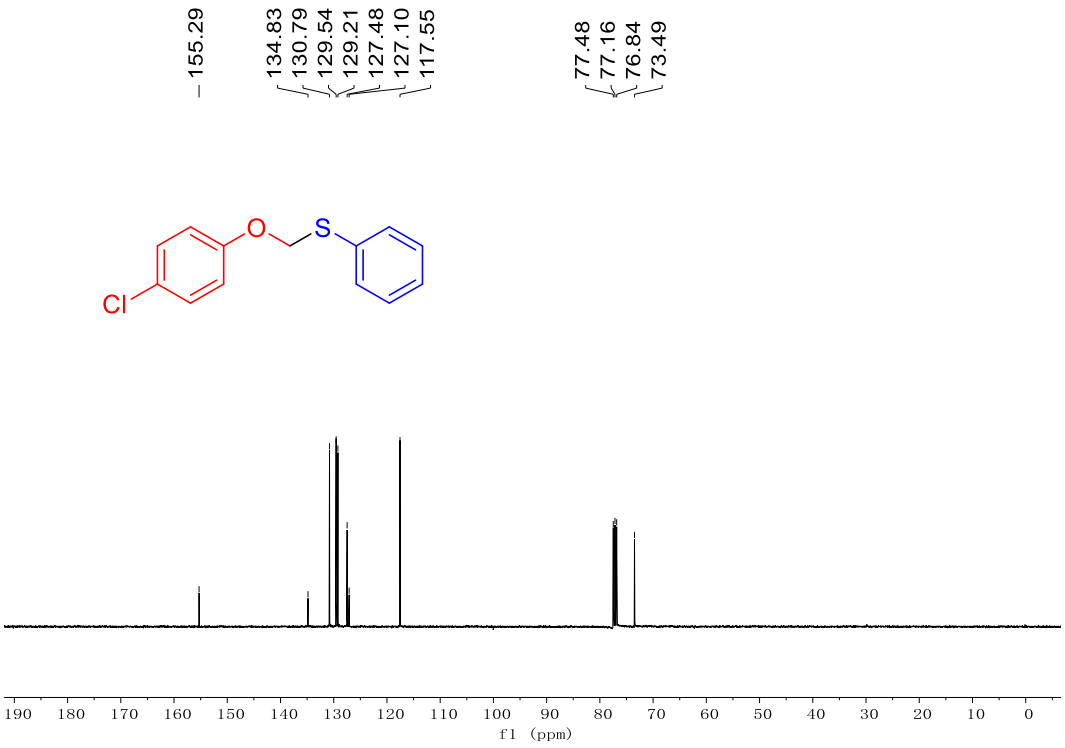


^{19}F-NMR spectrum of **4f** (377 MHz, $CDCl_3$)

((4-Chlorophenoxy)methyl)(phenyl)sulfane (4g).

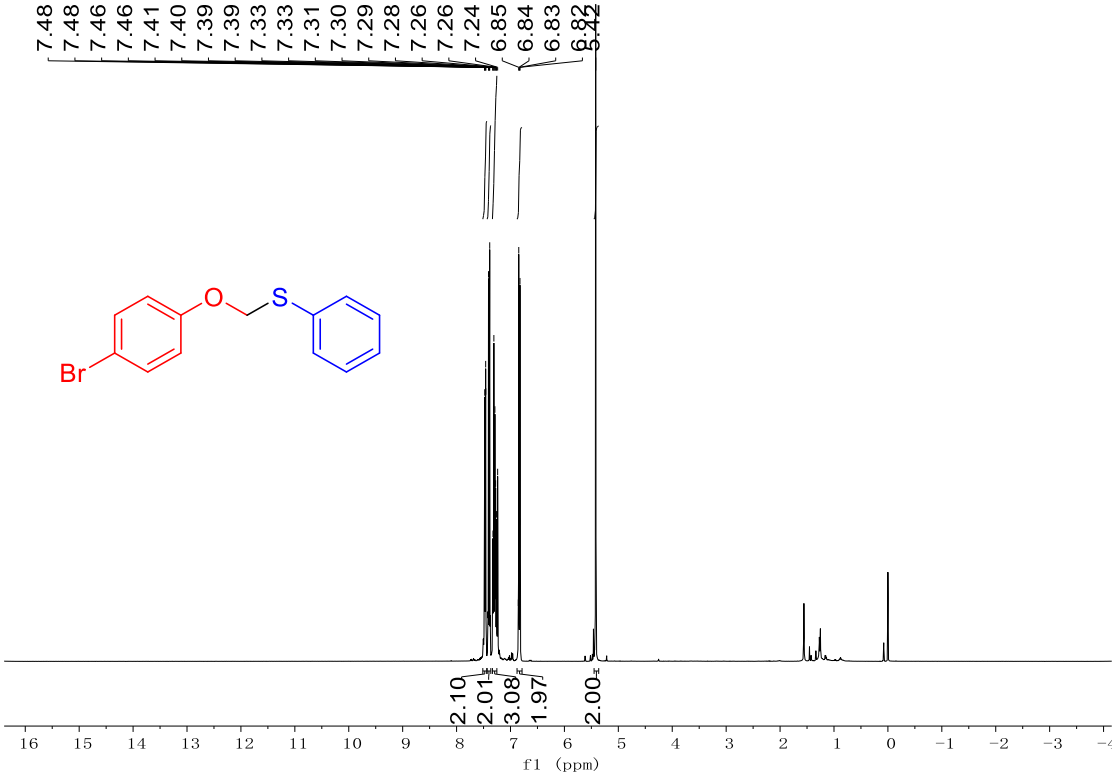


^{1}H-NMR spectrum of **4g** (400 MHz, $CDCl_3$)

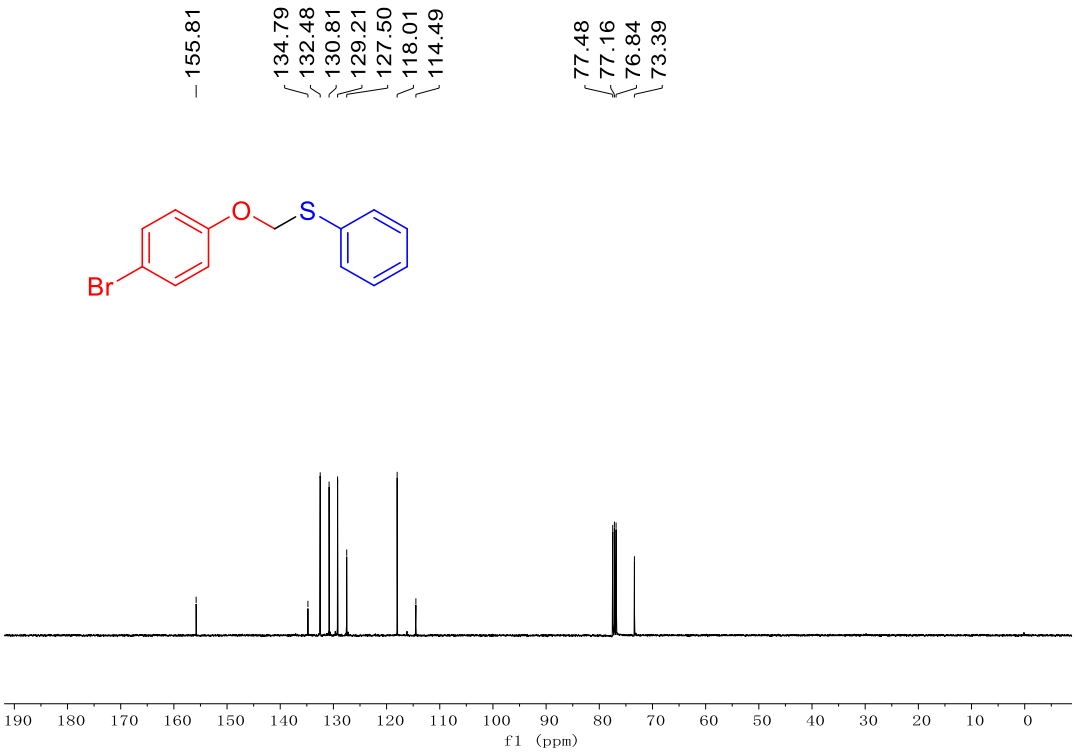


^{13}C-NMR spectrum of **4g** (100 MHz, $CDCl_3$)

((4-Bromophenoxy)methyl)(phenyl)sulfane (4h).

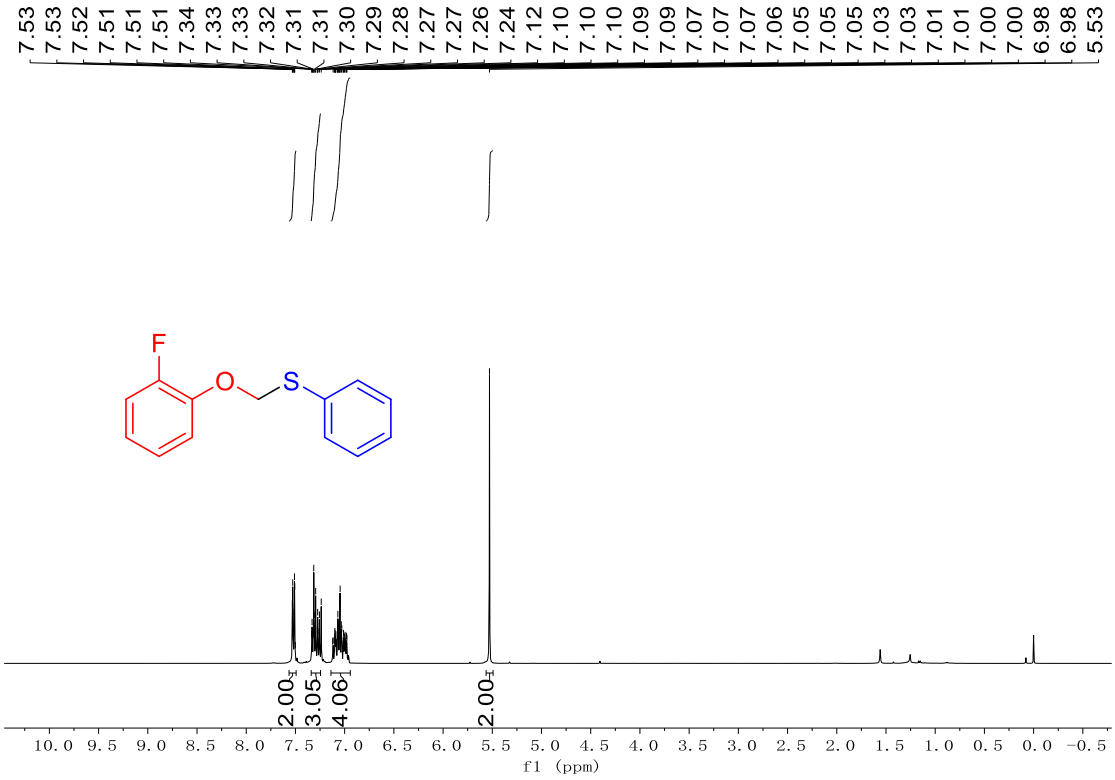


^{1}H-NMR spectrum of **4h** (400 MHz, $CDCl_3$)

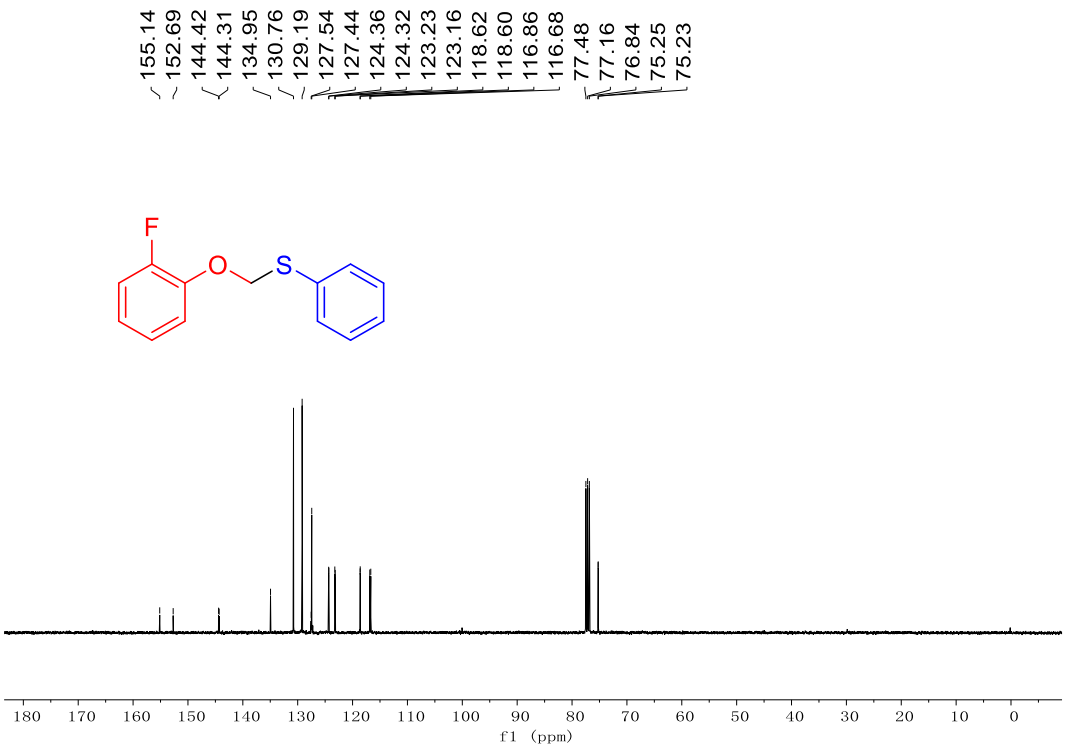


^{13}C-NMR spectrum of **4h** (100 MHz, $CDCl_3$)

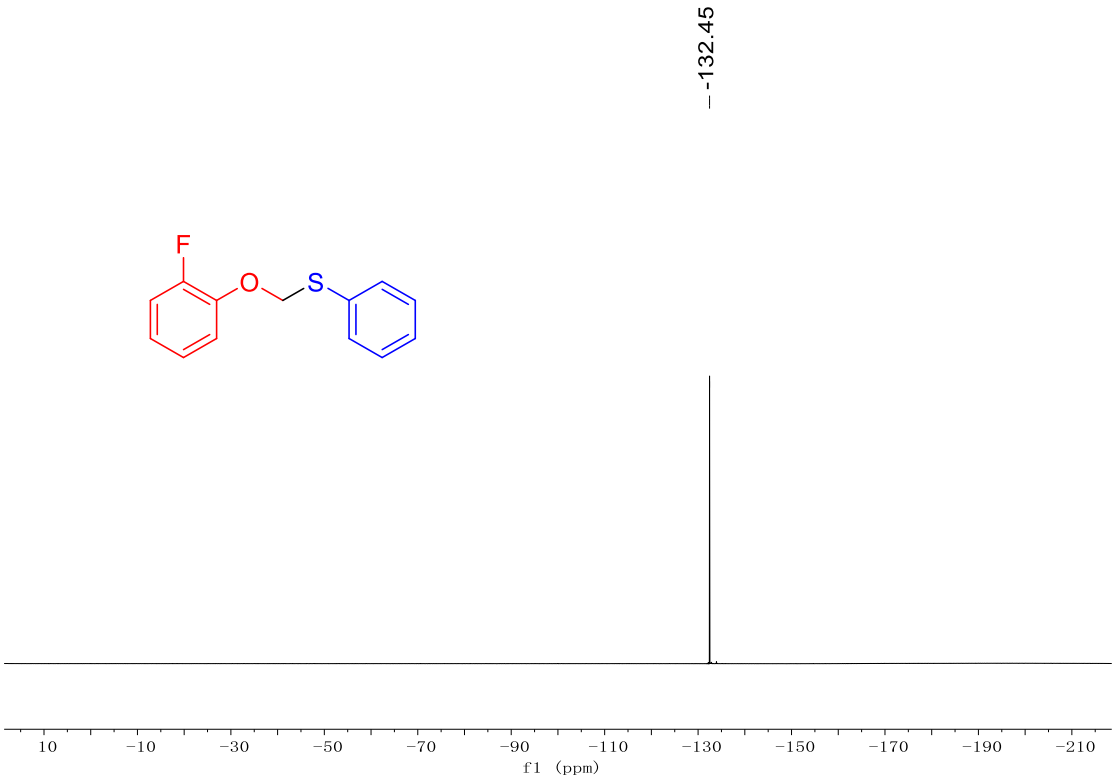
((2-Fluorophenoxy)methyl)(phenyl)sulfane (4i).



^{1}H-NMR spectrum of **4i** (400 MHz, $CDCl_3$)

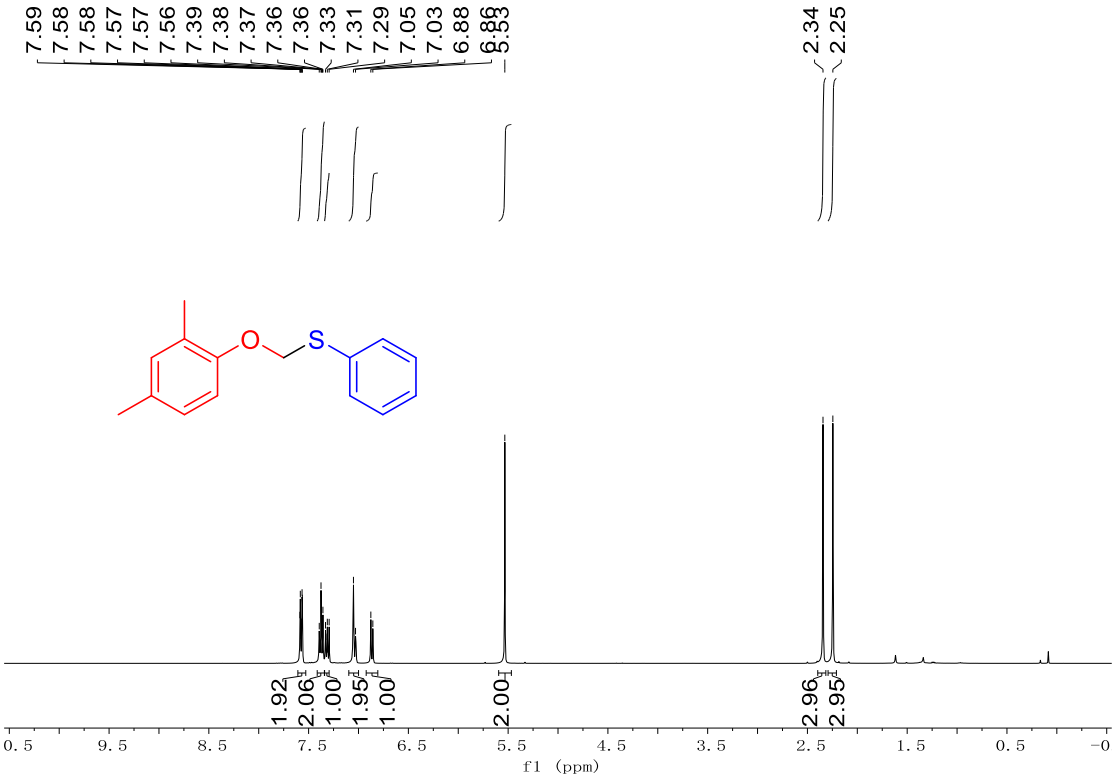


^{13}C-NMR spectrum of **4i** (100 MHz, $CDCl_3$)

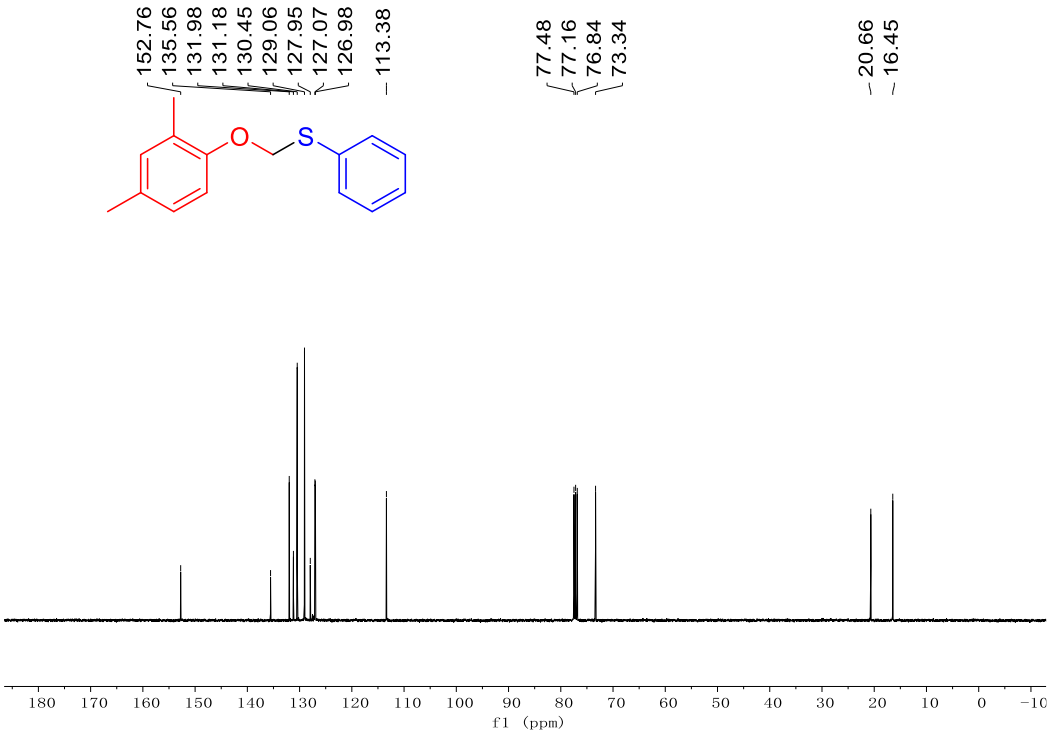


^{19}F-NMR spectrum of **4i** (377 MHz, $CDCl_3$)

((2,4-Dimethylphenoxy)methyl)(phenyl)sulfane (4j).

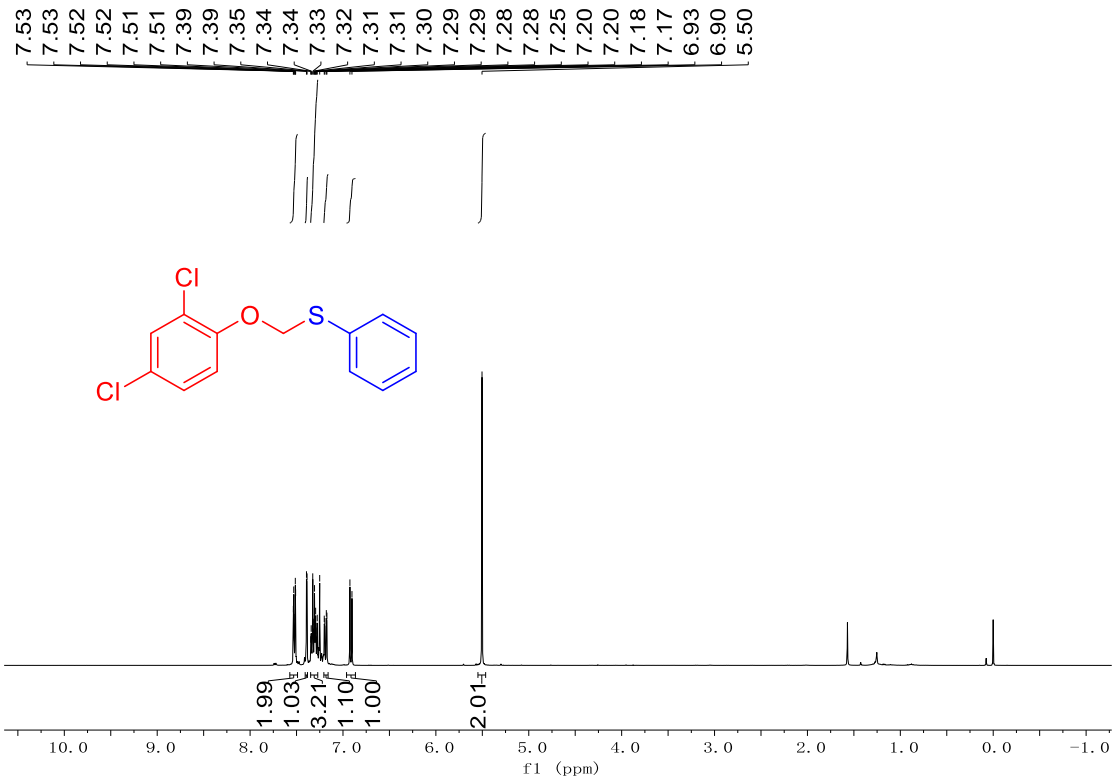


^{1}H-NMR spectrum of **4j** (400 MHz, $CDCl_3$)

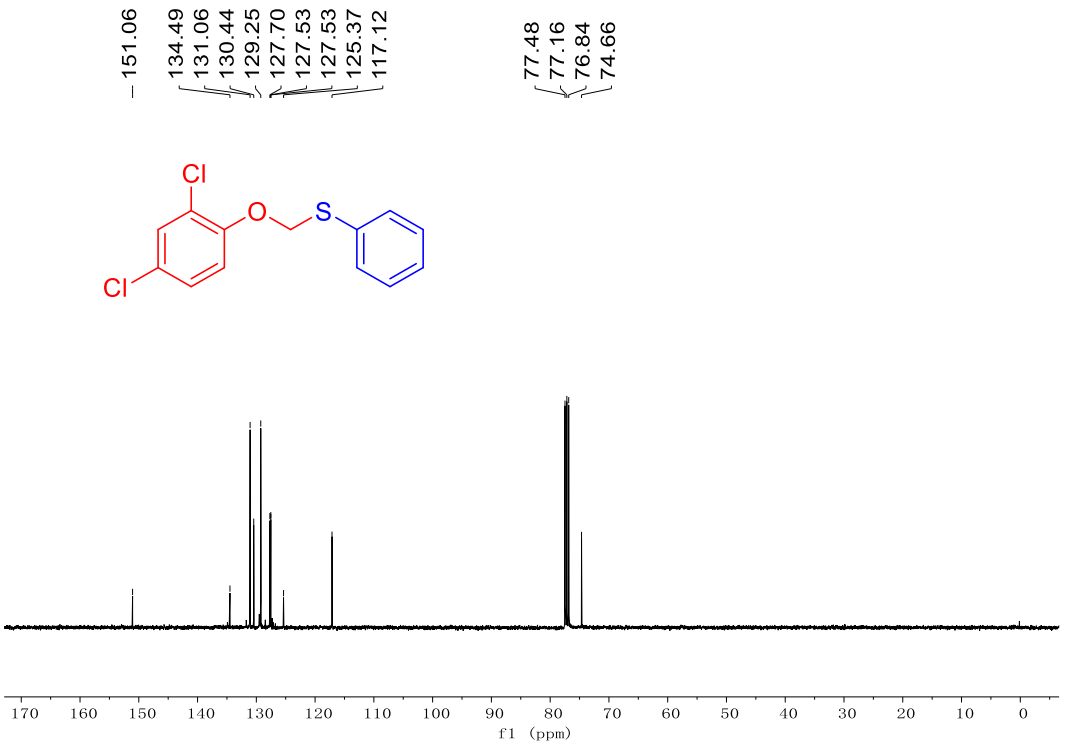


^{13}C-NMR spectrum of **4j** (100 MHz, $CDCl_3$)

((2,4-Dichlorophenoxy)methyl)(phenyl)sulfane (4k).

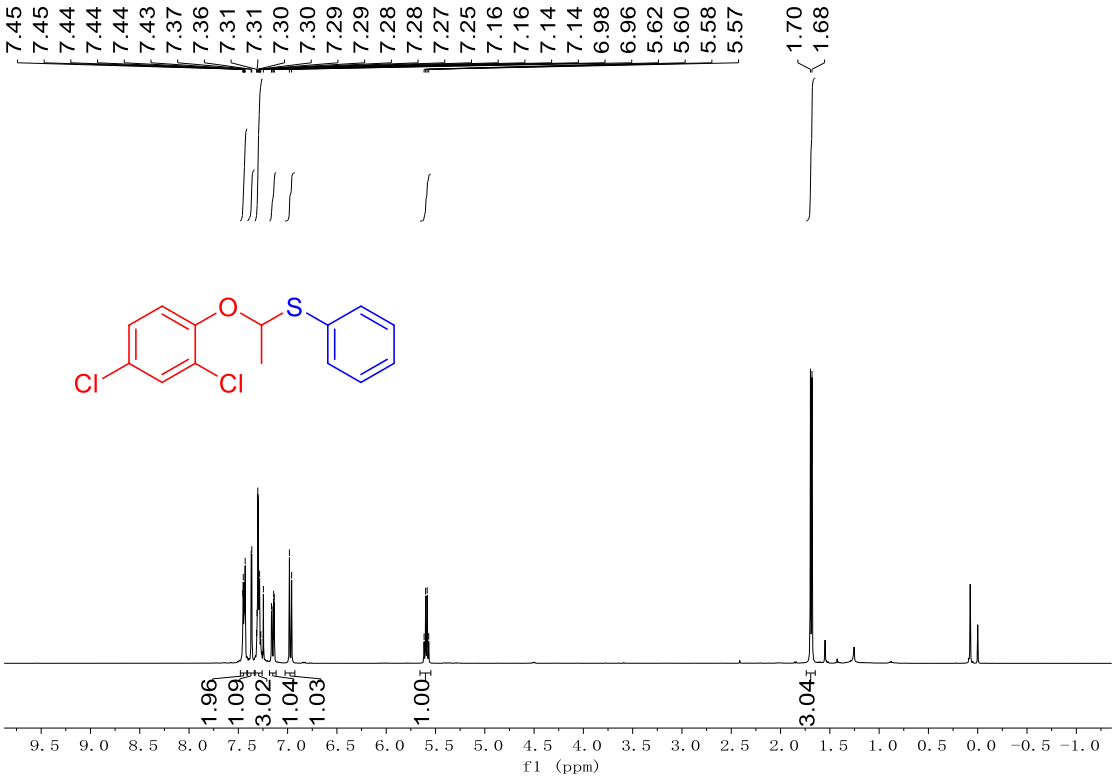


^{1}H-NMR spectrum of **4k** (400 MHz, $CDCl_3$)

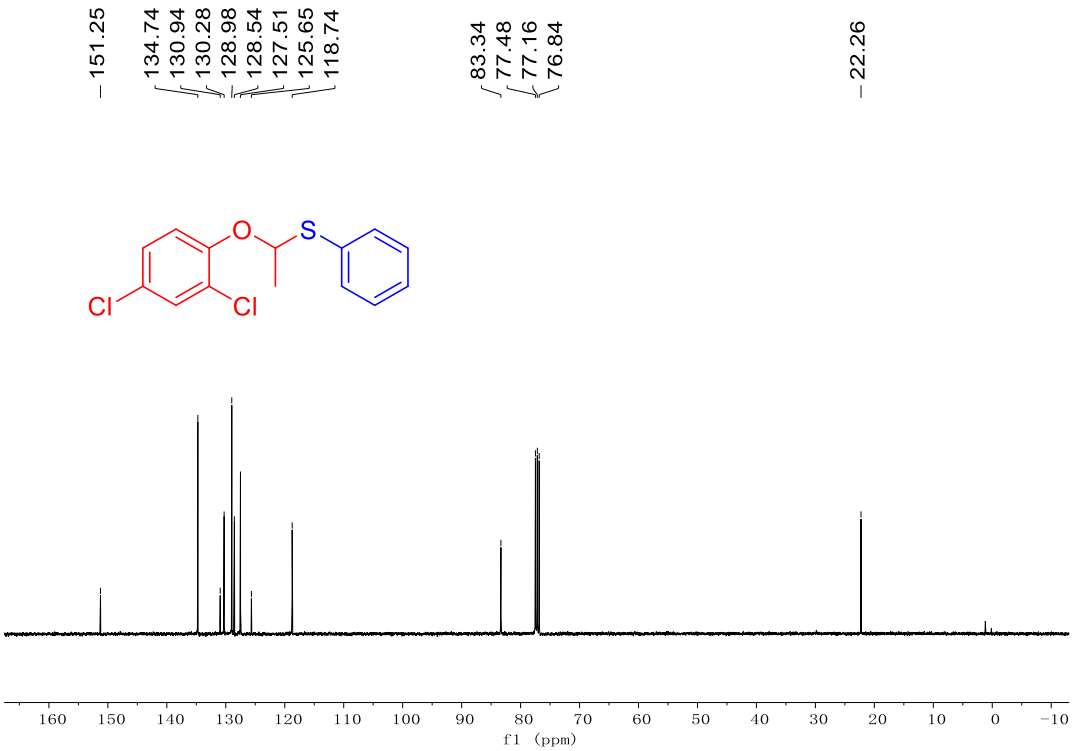


^{13}C-NMR spectrum of **4k** (100 MHz, $CDCl_3$)

((2,3-Dichlorophenoxy)methyl)(phenyl)sulfane (4l).

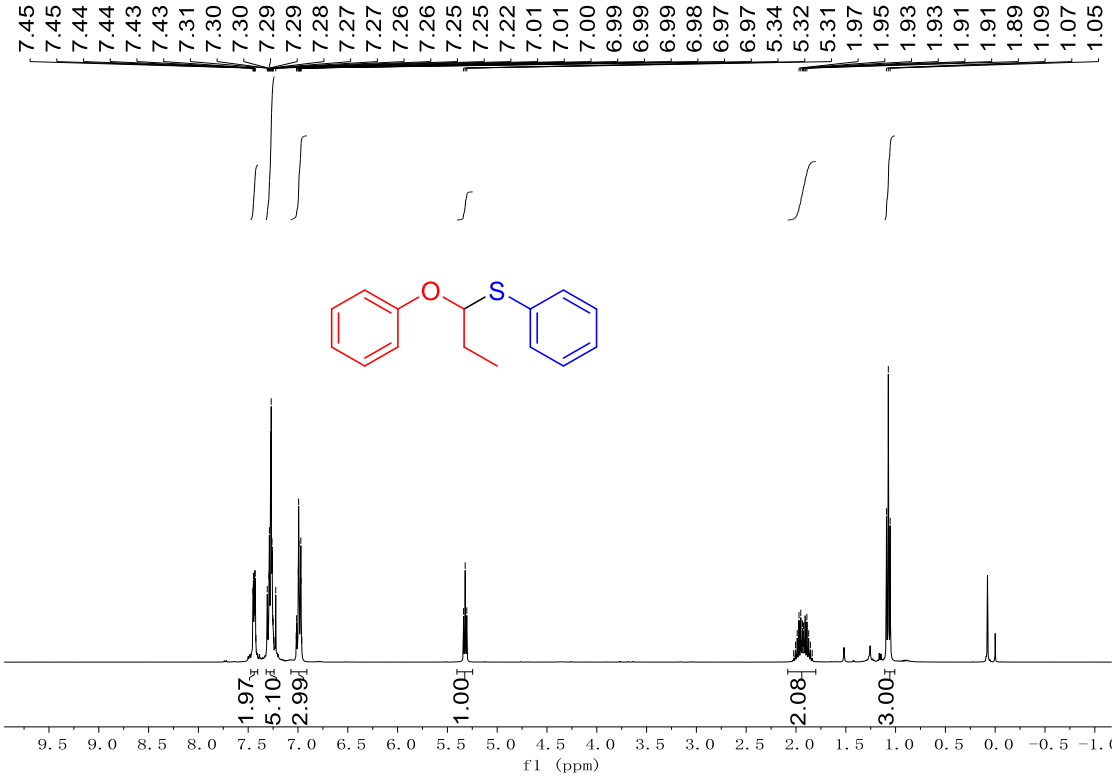


^{1}H-NMR spectrum of **4l** (400 MHz, $CDCl_3$)

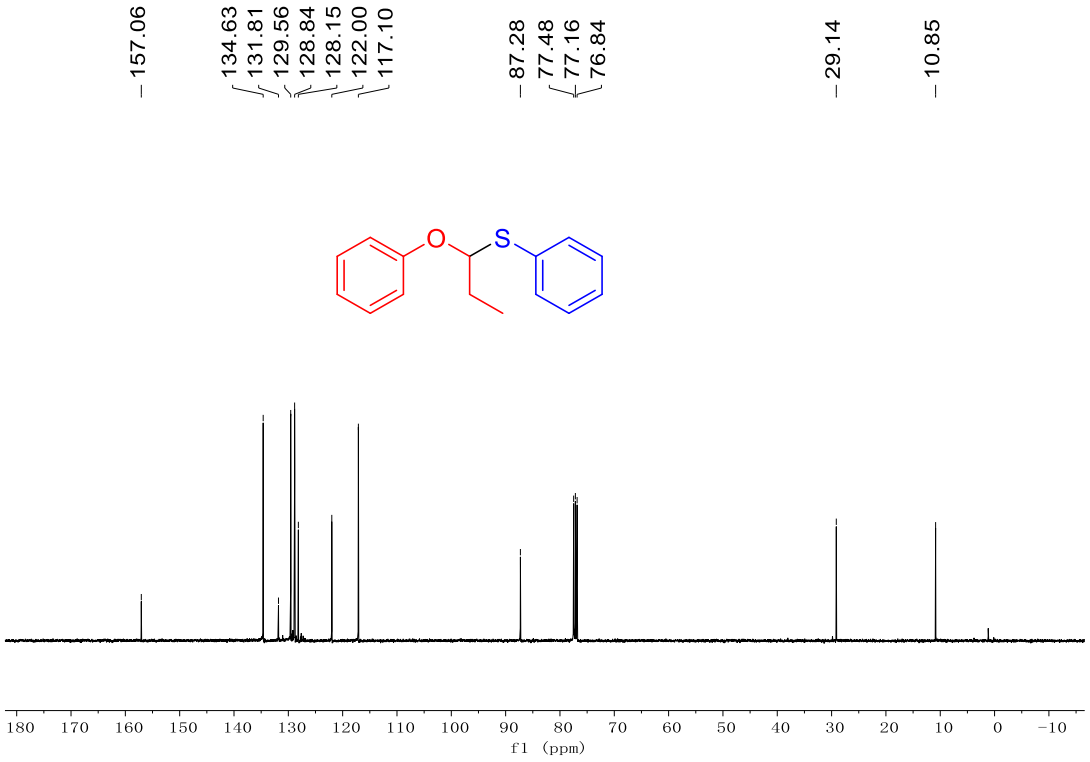


^{13}C-NMR spectrum of **4l** (100 MHz, $CDCl_3$)

(1- Phenoxypropyl)(phenyl)sulfane (4m).

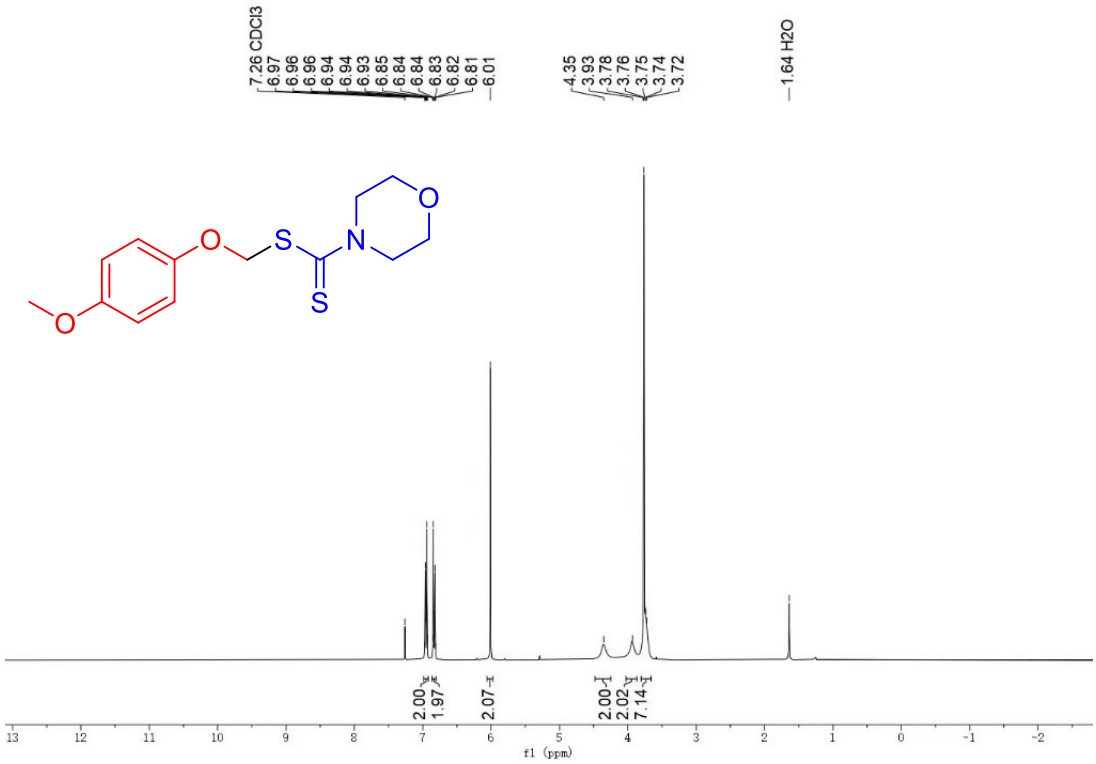


^{1}H-NMR spectrum of **4m** (400 MHz, $CDCl_3$)

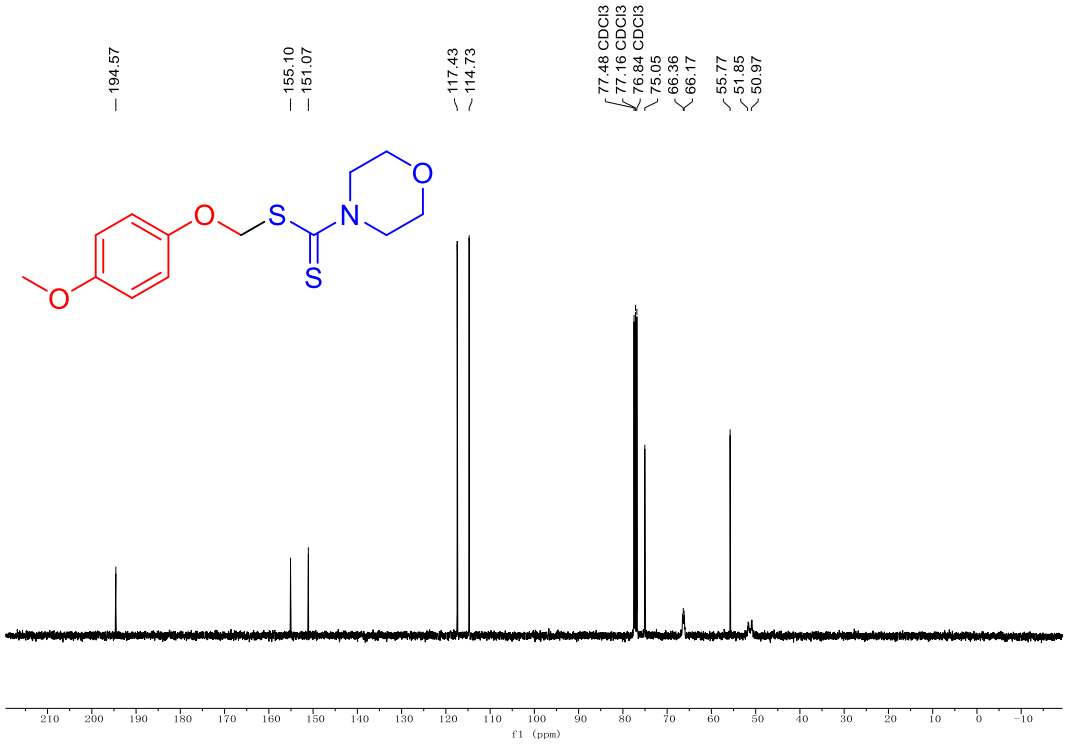


^{13}C-NMR spectrum of **4m** (100 MHz, $CDCl_3$)

(4-Methoxyphenoxy)methyl morpholine-4-carbodithioate (5a).

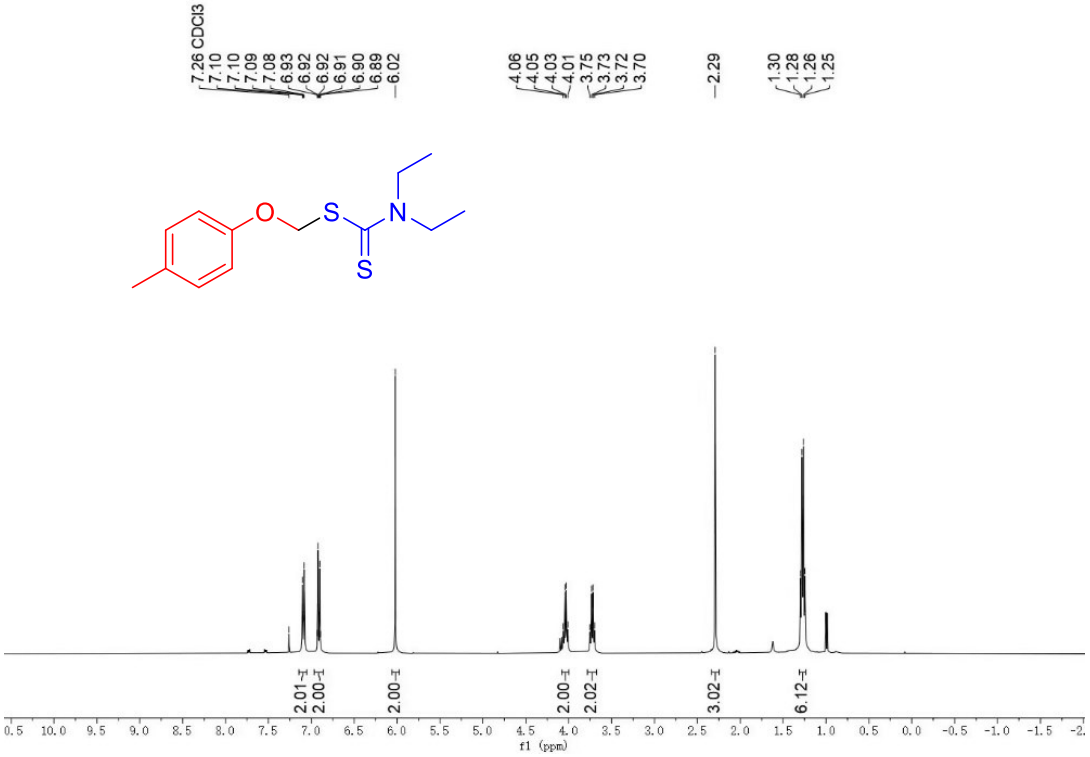


^{1}H-NMR spectrum of **5a** (400 MHz, $CDCl_3$)

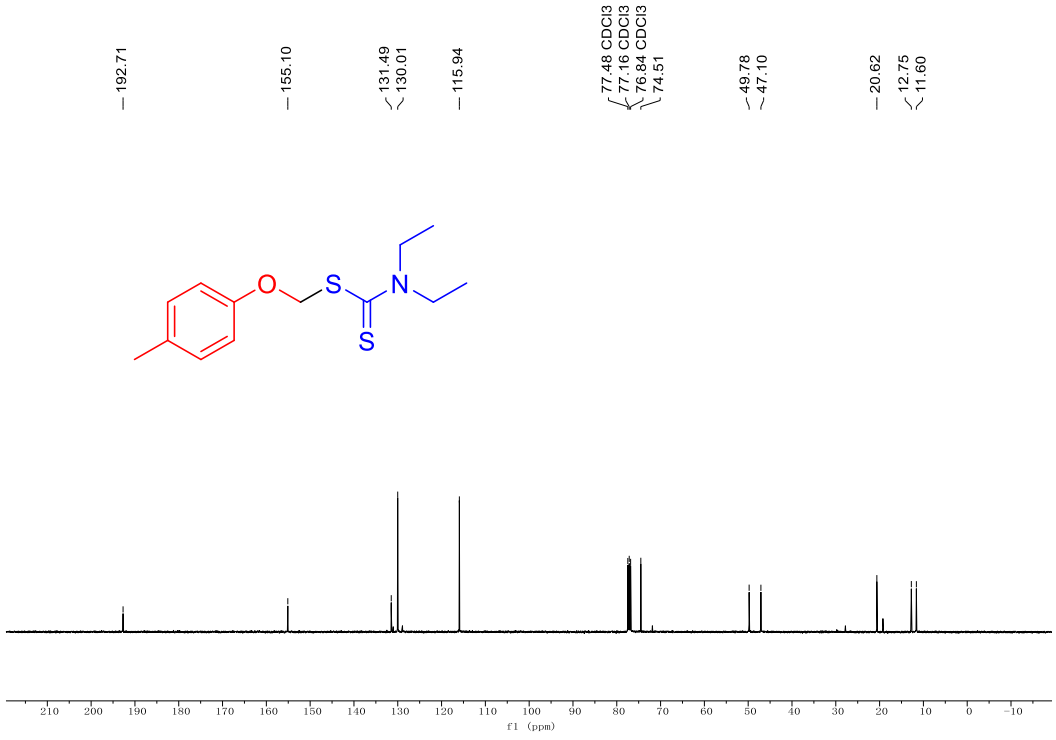


^{13}C-NMR spectrum of **5a** (100 MHz, $CDCl_3$)

(p-Tolyloxy)methyl diethylcarbamodithioate (5b).



^{1}H-NMR spectrum of **5b** (400 MHz, $CDCl_3$)



^{13}C-NMR spectrum of **5b** (100 MHz, $CDCl_3$)