Supporting Information

Q. Zhang, V. Massé, C. Darcel*

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1. General Method

All reagents were obtained from commercial sources and used as received. THF and toluene were dried using a MBraun Solvent Purification System and stored under an argon atmosphere. All reactions were carried out under argon atmosphere. Technical grade petroleum ether (40-60 °C bp.) and ethyl acetate were used for chromatography column. ^{1}H NMR spectra were recorded in $CDCl_3$ at ambient temperature on Bruker AVANCE 400 and 500 spectrometers at 400 MHz or 500 MHz, respectively, using the solvent as internal standard (7.26 ppm). ^{13}C NMR spectra were obtained at 101 MHz or 126 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm). Chemical shift (δ) and coupling constants (*J*) are given in ppm and in Hz, respectively. The peak patterns are indicated as follows: (s, singlet; d, doublet; t, triplet; q, quartet; pent, pentuplet; m, multiplet, and br. for broad). GC analyses were performed with GC-2014 (Shimadzu) 2010 equipped with a 30-m capillary column (Supelco, SPBTM-20, fused silica capillary column, 30 M×0.25 mm×0.25 mm film thickness), was used with N_2/air as vector gas. GCMS were measured by GCMS-QP2010S (Shimadzu) with GC-2010 equipped with a 30-m capillary column (Supelco, SLBTM-5ms, fused silica capillary column, 30 M×0.25 mm×0,25 mm film thickness), was used with helium as vector gas. The following GC conditions were used: initial temperature 80 °C, for 2 minutes, then rate 10 °C/min. until 225 °C and 225 °C for 15 minutes.

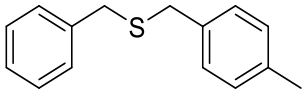
2. Sulfane synthesis

2.1. Typical procedure for thioester deoxygenation (Procedure 1)

In a dry Schlenk tube, with a magnetic bar, $Fe_2(CO)_9$ (11.0 mg, 0.015 mmol, 10 mol%), thioester **1** (0.3 mmol, 1 equiv.), phenylsilane (97.2 mg, 0.9 mmol, 3 equiv.) and dry toluene (0.2 mL) were added successively under an argon atmosphere. Then, the mixture was stirred upon Blue LED-light irradiation (2×24 W, 450-460 nm) at ambient temperature. After 24 h, the reaction was quenched by adding NaOH (1M, 1 mL) and MeOH (1 mL) and stirred at ambient temperature for 2 h. Then, the mixture was extracted with dichloromethane and the organic layer was collected, dried with anhydrous Na_2SO_4, filtered and concentrated in vacuum. The residue was distilled through using a Kugel Rohr bulb-to-bulb apparatus under vacuum, and then the pure thioether **2** was obtained after flash chromatography on silica eluting with petroleum ether and ethyl acetate (20:1 to 10:1).

2.2. Characterizations of the sulfanes

Benzyl(4-methylbenzyl)sulfane 2a[1]



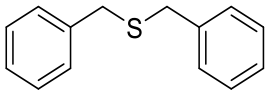
The compound **2a** was prepared as described in the general procedure **1** (colorless oil, 63 mg, 93 % isolated yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.20 (m, 5H), 7.16-7.05 (m, 4H), 3.56 (s, 2H), 3.53 (s, 2H), 2.30 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.25, 136.57, 135.01, 129.14, 128.98, 128.87, 128.43, 126.90, 35.57, 35.31, 21.07.

MS (EI): m/z (%) = 228 (M^+, 6), 105 (16), 91 (100).

Dibenzylsulfane 2b[1,6]



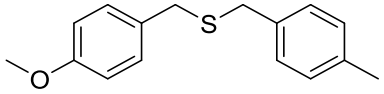
The compound **2b** was prepared as described in the general procedure **1** (colorless oil, 59 mg, 93 % isolated yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.31 (m, 5H), 7.29-7.25 (m, 5H), 3.63 (s, 4H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 137.38, 129.41, 128.48, 127.42, 43.31.

MS (EI): m/z (%) = 214 (M^+, 6), 91 (100), 65 (23).

(4-Methoxybenzyl)(4-methylbenzyl)sulfane 2c[2]



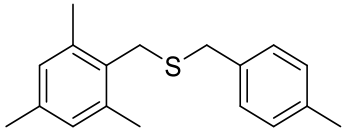
The compound **2c** was prepared as described in the general procedure **1** (colorless oil, 67 mg, 87 % isolated yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.15 (m, 4H), 7.12 (d, *J* = 7.8 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.57 (s, 2H), 3.56 (s, 2H), 2.34 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.58, 136.52, 135.13, 130.18, 130.04, 129.13, 128.86, 113.86, 55.27, 35.25, 34.96, 21.07.

MS (EI): m/z (%) = 258 (M^+, 6), 91 (100), 77 (23).

(4-Methylbenzyl)(2,4,6-trimethylbenzyl)sulfane 2d

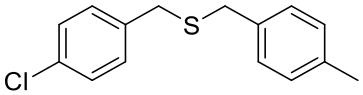


The compound **2d** was prepared as described in the general procedure **1** (colorless oil, 67 mg, 83 % isolated yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 6.79 (s, 2H), 3.74 (s, 2H), 3.62 (s, 2H), 2.35 (s, 3H), 2.25 (s, 6H), 2.23 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.97, 136.51, 136.37, 135.36, 130.89, 129.07, 128.89, 128.74, 37.22, 30.56, 21.08, 20.87, 19.38.
MS (EI): m/z (%) = 270 (M^+, 6), 105(13), 91 (100), 77 (23).

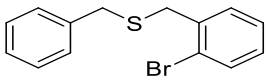
HR-MS HRMS (ACPI): m/z $[M+Me_3C_6H_2\text{-}CH_2]^+$ calculated for $C_{28}H_{35}S$: 403.2466, found: 403.2466.

(4-Chlorobenzyl)(4-methylbenzyl)sulfane 2e[2]



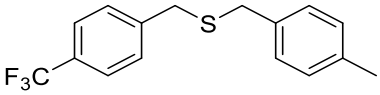
The compound **2e** was prepared as described in the general procedure **1** (colorless oil, 69 mg, 88 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.26 (m, 2H), 7.24-7.19 (m, 2H), 7.19-7.11 (m, 4H), 3.56 (s, 2H), 3.55 (s, 2H), 2.34 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.78, 136.70, 134.70, 132.66, 130.30, 129.18, 128.84, 128.56, 35.30, 34.83, 21.07.
MS (EI): m/z (%) = 264 (M^+, 6), 262 (M^+, 11), 105 (100), 91 (16).

Benzyl(2-bromobenzyl)sulfane 2f[3]



The compound **2f** was prepared as described in the general procedure **1** (colorless oil, 83 mg, 95 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.9 Hz, 1H), 7.39-7.31 (m, 5H), 7.30-7.26 (m, 2H), 7.13 (td, *J* = 7.9, 1.7 Hz, 1H), 3.77 (s, 2H), 3.73 (s, 2H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 137.90, 137.55, 133.12, 130.72, 128.98, 128.57, 128.51, 127.36, 127.05, 124.65, 36.22, 36.07.
MS (EI): m/z (%) = 293 (M^+, 9), 291 (M^+, 9), 105 (23), 91 (100).

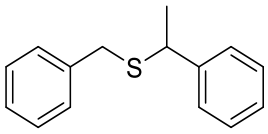
(4-Methylbenzyl)(4-(trifluoromethyl)benzyl)sulfane 2g



The compound **2g** was prepared as described in the general procedure **1** (colorless oil, 82 mg, 93 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.16 d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 3.62 (s, 2H), 3.58 (s, 2H), 2.34 (s, 2H).

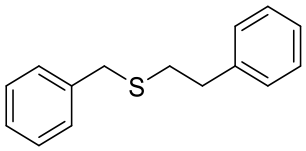
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 142.52, 136.81, 134.53, 129.26, 129.22, 129.16, 128.85, 125.38, 125.34, 35.44, 35.08, 21.06.
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.44.
MS (EI): m/z (%) = 296 (M^+, 12), 105(100), 91 (60).
HRMS (ACPI): m/z $[M+MeC_6H_4\text{-}CH_2]^+$ calculated for $C_{24}H_{24}F_3S$: 401.1551, found: 401.1552.

Benzyl(1-phenylethyl)sulfane 2h[4]



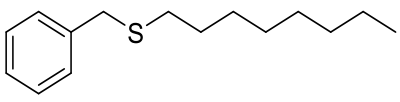
The compound **2h** was prepared as described in the general procedure **1** (colorless oil, 60 mg, 89 % isolated yield)
^1H NMR (400 MHz, $CDCl_3$) δ 7.45-7.21 (m, 10H), 3.85 (q, *J* = 7.1 Hz, 1H), 3.59 (d, *J* = 13.5 Hz, 1H), 3.49 (d, *J* = 13.5 Hz, 1H), 1.58 (d, *J* = 7.1 Hz, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 143.81, 138.43, 128.87, 128.48, 128.38, 127.45, 127.06, 126.82, 43.57, 35.73, 22.54.
MS (EI): m/z (%) = 228 (M^+, 10), 105(100), 91 (55).

Benzyl(2-phenethyl)sulfane 2i[5]



The compound **2i** was prepared as described in the general procedure **1** (colorless oil, 58 mg, 85 % isolated yield)
^1H NMR (400 MHz, $CDCl_3$) δ 7.28-7.16 (m, 8H), 7.12-7.07 (m, 2H), 3.67 (s, 2H), 2.85-2.71 (m, 2H), 2.66-2.58 (m, 2H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 140.54, 138.41, 128.87, 128.49, 128.43, 126.98, 126.31, 36.46, 36.04, 32.79.
MS (EI): m/z (%) = 228 (M^+, 7), 105(55), 91 (100), 77(12).

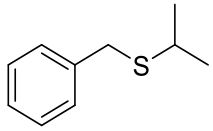
Benzyl(octyl)sulfane 2j[6]



The compound **2j** was prepared as described in the general procedure **1** (colorless oil, 66 mg, 93 % isolated yield)
^1H NMR (400 MHz, $CDCl_3$) δ 7.28-7.21 (m, 4H), 7.20-7.17 (m, 1H), 3.66 (s, 2H), 2.37 (t, *J* = 7.4 Hz, 2H), 1.54-1.48 (m, 2H), 1.24-1.21 (m, 10H), 0.84 (t, *J* = 6.8 Hz, 3H).

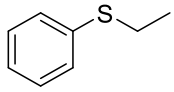
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 138.69, 128.80, 128.41, 126.82, 36.31, 31.79, 31.42, 29.23, 29.16, 29.14, 28.87, 22.63, 14.06.
MS (EI): m/z (%) = 236 (M^+, 7), 91 (100), 77(16).

Benzyl(isopropyl)sulfane 2k[7]



The compound **2k** was prepared as described in the general procedure **1** (colorless oil, 48 mg, 97 % isolated yield)
^1H NMR (400 MHz, $CDCl_3$) δ 7.32-7.24 (m, 4H), 7.21-7.16 (m, 1H), 3.71 (s, 2H), 2.77 (hept, *J* = 6.6 Hz, ^1H), 1.22 (d, *J* = 6.6 Hz, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 138.78, 128.75, 128.43, 126.79, 35.16, 34.27, 23.12.
MS (EI): m/z (%) = 166 (M^+, 9), 91 (100), 77(23).

Ethyl(phenyl)sulfane 2l[8]



The compound **2l** was prepared as described in the general procedure **1** (colorless oil, 37 mg, 91 % isolated yield)
^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 4H), 7.18-7.12 (m, 1H), 2.92 (q, *J* = 7.3 Hz, 2H), 1.29 (t, *J* = 7.3 Hz, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 136.62, 129.00, 128.78, 125.72, 27.62, 14.32.
MS (EI): m/z (%) = 138 (M^+, 100), 123(16), 77(23).

3. Ether oxide synthesis

3.1. Typical procedure for thioester desulfurization (Procedure 2)

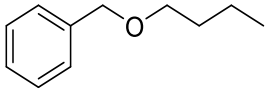
In a dry Schlenk tube, with a magnetic bar, $Fe_2(CO)_9$ (11.0 mg, 0.015 mmol, 10 mol%), thioester **4** (0.3 mmol, 1 equiv.), phenylsilane (97.2 mg, 0.9 mmol, 3 equiv.) and dry toluene (0.2 mL) were added successively under an argon atmosphere. Then, the mixture was stirred upon Blue LED-light irradiation (2×24 W, 450-460 nm) at ambient temperature. After 24 h, the reaction was quenched by adding NaOH (1M, 1 mL) and MeOH (1 mL) and stirred at ambient temperature for 2 h. Then, the mixture was extracted with dichloromethane and the organic layer was collected, dried with anhydrous Na_2SO_4, filtered and concentrated in vacuo. The residue was distillated using a Kugel Rohr bulb-to-bulb apparatus under vacuum, and then the pure ether product **5** was obtained after flash chromatography on silica eluting with petroleum ether and ethyl acetate (20:1 to 10:1).

3.2. Typical procedure for carboxylic ester deoxygenation (Procedure 3)

In a dry Schlenk tube, with a magnetic bar, $Fe_2(CO)_9$ (5.5 mg, 0.015 mmol, 5 mol%), carboxylic ester **6** (0.3 mmol, 1 equiv.), phenylsilane (97.2 mg, 0.9 mmol, 3 equiv.) and dry toluene (0.2 mL) were added successively under an argon atmosphere. Then, the mixture was stirred upon Blue LED-light irradiation (2×24 W, 450-460 nm) at ambient temperature. After 24 h, the reaction was quenched by adding NaOH (1M, 1 mL) and MeOH (1 mL) and stirred at ambient temperature for 2 h. Then, the mixture was extracted with dichloromethane and the organic layer was collected, dried with anhydrous Na_2SO_4, filtered and concentrated in vacuo. The residue was distillated using a Kugel Rohr bulb-to-bulb apparatus under vacuum, and then the pure ether product **5** was obtained after flash chromatography on silica eluting with petroleum ether and ethyl acetate (20:1 to 10:1).

3.3. Characterization of the ether oxides

(Butoxymethyl)benzene 5a[9]



The compound **5a** was prepared as described in the general procedure **2** starting from S-*n*-butyl benzothioate (colorless oil, 44 mg, 91 % isolated yield)

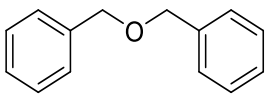
The compound **5a** was also prepared as described in the general procedure **3** starting from *n*-butyl benzoate (colorless oil, 48 mg, 99 % isolated yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.26 (m, 4H), 7.24-7.20 (m, 1H), 4.45 (s, 2H), 3.42 (t, *J* = Hz, 2H), 1.59-1.51 (m, 2H), 1.35 (sext, *J* = Hz, 2H), 0.87 (t, *J* = Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.76, 128.32, 127.59, 127.43, 72.85, 70.22, 31.85, 19.38, 13.91.

MS (EI): m/z (%) = 164 (M^+, 12), 91 (100), 77(19).

Dibenzylether 5b[10]

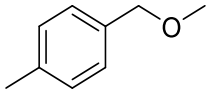


The compound **5b** was prepared as described in the general procedure **2** starting from S-benzyl benzothioate **1b** (colorless oil, 55 mg, 93 % isolated yield).

The compound **5b** was also prepared as described in the general procedure **3** starting from benzyl benzoate (colorless oil, 57 mg, 96 % isolated yield).

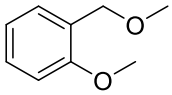
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.34 (m, 8H), 7.32-7.20 (m, 2H), 4.58 (s, 4H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 138.29, 128.39, 127.77, 127.62, 72.12.
MS (EI): m/z (%) = 198 (M^+, 10), 91 (100), 77(25).

1-(Methoxymethyl)-4-methylbenzene 5c[11]



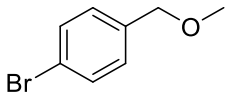
The compound **5c** was prepared as described in the general procedure **2** from S-methyl *p*-methylbenzothioate. (colorless oil, 36 mg, 90 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.7 Hz, 2H), 7.21 (d, *J* = 7.7 Hz, 2H), 4.51 (s, 2H), 3.47-3.42 (m, 3H), 2.44 (s, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 137.21, 135.10, 128.98, 127.76, 74.50, 57.81, 21.06.
MS (EI): m/z (%) = 136 (M^+, 13), 91 (100), 77(25).

1-Methoxy-2-(methoxymethyl)benzene 5d[12]



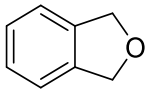
The compound **5d** was prepared as described in the general procedure **2** from S-methyl 2-methoxybenzothioate. (colorless oil, 37 mg, 83 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.34 (m, 1H), 7.29-7.25 (m, 1H), 6.96 (t, *J* = 7.4 Hz, ^{1}H), 6.88 (d, *J* = 8.2 Hz, ^{1}H), 4.51 (s, 2H), 3.84 (s, 3H), 3.42 (s, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 157.22, 129.10, 128.73, 126.52, 120.40, 110.26, 69.55, 58.31, 55.38.
MS (EI): m/z (%) = 152 (M^+, 13), 91 (100), 77(25).

1-Bromo-4-(methoxymethyl)benzene 5e[11]



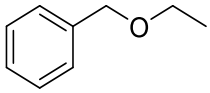
The compound **5e** was prepared as described in the general procedure **2** from S-methyl *p*-bromobenzothiate (colorless oil, 52 mg, 87 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.4 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 4.40 (s, 1H), 3.38 (s, 2H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 137.22, 131.44, 129.23, 121.42, 73.84, 58.12.
MS (EI): m/z (%) = 201 (M^+, 9), 199 (M^+, 9), 91 (100), 31(25).

1,3-Dihydroisobenzofuran 5f[13]



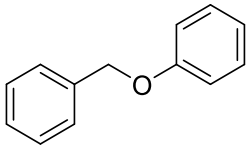
The compound **5f** was prepared as described in the general procedure **2** from isobenzofuran-1(3H)-thione (colorless oil, 33 mg, 93 % isolated yield)
The compound **5f** was prepared as described in the general procedure **3** from isobenzofuran-1(3H)-one (colorless oil, 33 mg, 93 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 4H), 5.13 (s, 4H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 139.04, 127.20, 120.92, 73.55.
MS (EI): m/z (%) = 120 (M^+, 16), 91 (100), 65(12).

(Ethoxymethyl)benzene 5g[11]



The compound **5g** was prepared as described in the general procedure **3** from S-ethyl benzothioate. (colorless oil, 39 mg, 99 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.34 (m, 4H), 7.32-7.27 (m, 1H), 4.54 (s, 2H), 3.58 (d, *J* = 7.0 Hz, 2H), 1.29 (d, *J* = 7.0 Hz, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.58, 128.28, 127.59, 127.42, 72.64, 65.64, 15.16.
MS (EI): m/z (%) = 136 (M^+, 13), 91 (100), 65(12).

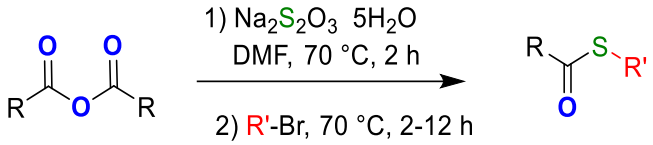
(Benzyloxy)benzene 5h[14]



The compound **5h** was prepared as described in the general procedure **3** from S-phenyl benzothioate. (colorless oil, 45 mg, 83 % isolated yield)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.43 (m, 4H), 7.43-7.34 (m, 3H), 7.12-6.98 (m, 3H), 5.15 (s, 2H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.81, 137.10, 129.47, 128.57, 127.92, 127.46, 120.93, 114.87, 69.93.
MS (EI): m/z (%) = 184 (M^+, 12), 91 (100), 77(25).

4. Preparation of the starting thioamides (Procedure 4)

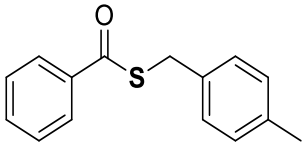
Before being able to study different experimental conditions for the reduction of thioester derivatives, it was first necessary to synthesize them. The chosen preparation method consisted of the reaction of acyl anhydrides with sodium thiosulfate pentahydrate, in the presence of halogenated derivatives.[15] (Scheme S1)



Scheme S1: Preparation of thioesters from acyl anhydrides

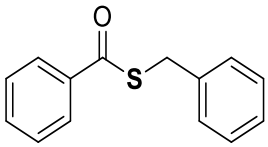
In a dry Schlenk tube, with a magnetic bar, to a solution of the organic anhydride (1.0 equiv.) in DMF (0.1 M) was added $Na_2S_2O_3 \cdot 5H_2O$ (1.1 equiv.) and stirred at 70 °C under an argon atmosphere for several hours. Then, the organic halide (1.5 or 3.0 equiv.) in DMF was added to the reaction mixture (overall, 0.067 M) and stirred at 70 °C under an argon atmosphere. The resulting reaction mixture was stirred for several hours until the reaction was completed as indicated by TLC. The mixture was extracted with ether three times, and the combined organic layer was washed with brine, dried over MgSO4, filtered and concentrated. The crude products were purified by flash column chromatography to afford the desired product. (petroleum ether /CH_2Cl_2, 2:1 to 20:1)

S-(4-Methylbenzyl) benzothioate 1a[16]



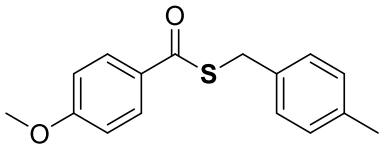
The compound **1a** was prepared as described in the general procedure **4**. (72 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (m, 2H), 7.57 (m, 1H), 7.47-7.40 (m, 2H), 7.29 (d, *J* = 7.4 Hz, 2H), 7.13 (d, *J* = 7.4 Hz, 2H), 4.31 (s, 2H), 2.34 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 191.40, 137.03, 136.88, 134.35, 133.35, 129.33, 128.86, 128.58, 127.27, 33.12, 21.09.
MS (EI): m/z (%) = 242 (M^+, 10), 227 (7), 105 (43), 91 (100).

S-Benzyl benzothioate 1b[16]



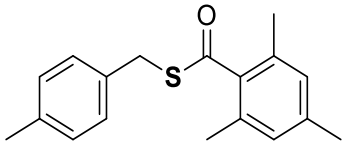
The compound **1b** was prepared as described in the general procedure **4**. (65 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 2H) 7.59 (m, 1H), 7.48-7.27 (m, 7H), 4.35 (s, 2H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 191.31, 137.46, 136.79, 133.44, 129.97, 128.65, 128.62, 127.32, 127.29. 33.33.
MS (EI): m/z (%) = 228 (M^+, 8), 137 (30), 105 (55), 91 (100).

S-(4-Methylbenzyl) 4-methoxybenzothioate 1c NEW



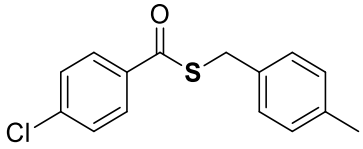
The compound **1c** was prepared as described in the general procedure **4**. (53 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17-8.07 (m, 2H), 7.33-7.27 (m, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.99-6.82 (m, 2H), 4.59 (s, 2H), 3.88 (s, 3H), 2.36 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 189.92, 163.79, 136.95, 134.65, 129.77, 129.47, 129.31, 128.86, 113.77, 55.50, 32.98, 21.09.
MS (EI): m/z (%) = 272 (M^+, 12), 257 (6), 105 (55), 91 (100).

S-(4-Methylbenzyl) 2,4,6-trimethylbenzothioate 1d NEW



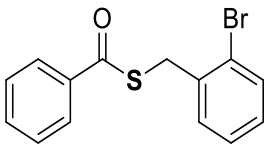
The compound **1d** was prepared as described in the general procedure **4**. (43 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.9 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 2H), 6.96 (s, 2H), 4.41 (s, 2H), 2.47 (s, 3H), 2.41 (s, 3H), 2.39 (s, 6H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 197.32, 139.27, 137.20, 136.93, 134.51, 133.75, 129.31, 128.73, 128.37, 33.66, 21.12, 21.10, 18.94.
MS (EI): m/z (%) = 284 (M^+, 12), 147 (100), 65 (13).

S-(4-Methylbenzyl) 4-chlorobenzothioate 1e NEW



The compound **1e** was prepared as described in the general procedure **4**. (60 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.90 (m, 2H), 7.47-7.40 (m, 2H), 7.31-7.25 (m, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 4.29 (s, 2H), 2.33 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 190.24, 139.76, 137.17, 135.22, 134.06, 129.37, 128.90, 128.85, 128.61, 33.25, 21.10.
MS (EI): m/z (%) = 278 (M^+, 5), 276 (M^+, 12), 139 (100), 105 (16).

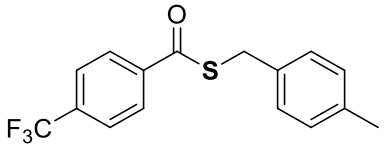
S-(2-Bromobenzyl) benzothioate 1f[16]



The compound **1f** was prepared as described in the general procedure **4**. (40 % isolated yield).

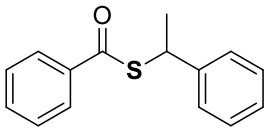
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.95 (m, 2H), 7.60-7.53 (m, 3H), 7.44 (t, *J* = 7.8 Hz, 2H), 7.29-7.24 (m, 1H), 7.13 (td, *J* = 7.8, 1.7 Hz, 1H), 4.47 (s, 2H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 191.12, 137.11, 136.70, 133.50, 132.88, 131.37, 129.06, 128.63, 127.68, 127.31, 124.65, 33.85.
MS (EI): m/z (%) = 307 (M^+, 9), 305 (M^+, 10), 105 (60), 91 (100).

S-(4-Methylbenzyl) 4-(trifluoromethyl)benzothioate 1g NEW



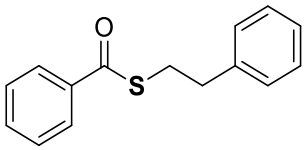
The compound **1g** was prepared as described in the general procedure **4**. (57 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.2 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 7.7 Hz, 2H), 7.14 (d, *J* = 7.7 Hz, 2H), 4.32 (s, 2H), 2.34 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 190.50, 139.63, 137.30, 134.66 (q, *J* = 32.7 Hz), 133.78, 129.41, 128.87, 127.61, 125.67 (q, *J* = 3.8 Hz), 123.51 (q, *J* = 272.8 Hz), 33.42, 21.10.
^{19}F NMR (376 MHz, $CDCl_3$) δ -63.13.
MS (EI): m/z (%) = 310 (M^+, 12), 105 (25), 91 (100).

S-(1-Phenylethyl) benzothioate 1h[17]



The compound **1h** was prepared as described in the general procedure **4**. (69 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.91 (m, 2H) 7.59-7.51 (m, 1H), 7.48-7.22 (m, 7H), 4.96 (q, *J* = 7.1 Hz, 1H), 1.77 (d, *J* = 7.1 Hz, 3H)
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 191.17, 142.65, 136.97, 133.31, 128.62, 128.56, 127.36, 127.23, 43.04, 22.37.
MS (EI): m/z (%) = 242 (M^+, 9), 227 (18), 105 (100), 77 (22).

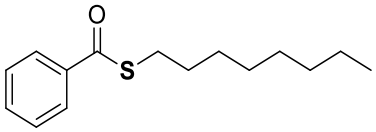
S-Phenethyl benzothioate 1i[16,18]



The compound **1i** was prepared as described in the general procedure **4**. (47 % isolated yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01-7.94 (m, 2H), 7.62-7.53 (m, 1H), 7.50-7.41 (m, 2H), 7.38-7.20 (m, 5H), 3.37-3.29 (m, 2H), 3.02-2.94 (m, 2H).

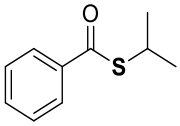
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 191.83, 140.07, 137.14, 133.33, 128.64, 128.59, 128.53, 127.21, 126.53, 35.93, 30.43.
MS (EI): m/z (%) = 242 (M^+, 10), 227 (6), 105 (100).

S-Octyl benzothioate 1j[18]



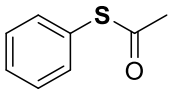
The compound **1j** was prepared as described in the general procedure **4**. (59 % isolated yield).
^1H NMR (400 MHz, $CDCl_3$) δ 8.05-7.96 (m, 2H), 7.61-7.41 (m, 1H), 7.48-7.41 (m, 2H), 3.09 (t, *J* = 7.0 Hz, 2H), 1.7 (m, 2H), 1.51-1.25 (m, 10H), 0.91 (t, *J* = 7.0 Hz, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 191.93, 137.27 133.07, 128.45, 127.11, 31.75, 29.53, 29.08, 28.99, 29.0, 28.90, 22.58, 14.01.
MS (EI): m/z (%) = 250 (M^+, 6), 121 (100), 77(16).

S-Isopropyl benzothioate 1k[18]



The compound **1k** was prepared as described in the general procedure **4**. (48 % isolated yield).
^1H NMR (400 MHz, $CDCl_3$) δ 7.98-7.91 (m, 2H), 7.59-5.51 (m, 1H), 7.48-7.39 (m, 2H), 3.86 (sept, *J* = 6.8 Hz, 1H), 1.41 (d, *J* = 6.8 Hz, 6H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 192.12, 137.38, 133.14, 128.52, 127.11, 34.90, 33.13.
MS (EI): m/z (%) = 180 (M^+, 6), 121 (100), 77(13).

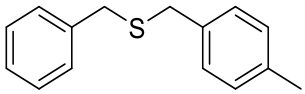
S-Phenyl ethanethioate 1l[19]



The compound **1l** was prepared as described in the general procedure **4**. (62 % isolated yield).
^1H NMR (400 MHz, $CDCl_3$) δ 7.29-7.23 (m, 5H), 2.26 (s, 3H).
$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 193.91, 134.39, 129.37, 129.14, 127.93, 30.12.
MS (EI): m/z (%) = 152 (M^+, 12), 110 (100), 77(20).

5. *^{1}H NMR $^{13}C\{^{1}H\}$ NMR and ^{19}F NMR spectra of the compounds*

Benzyl(4-methylbenzyl)sulfane 2a



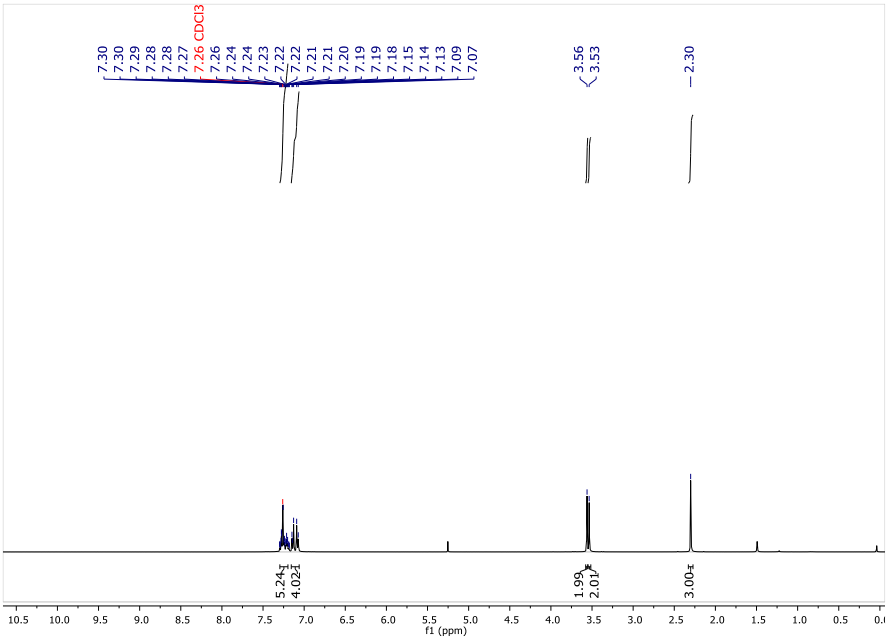


Figure S1 – ^{1}H-NMR of **2a**

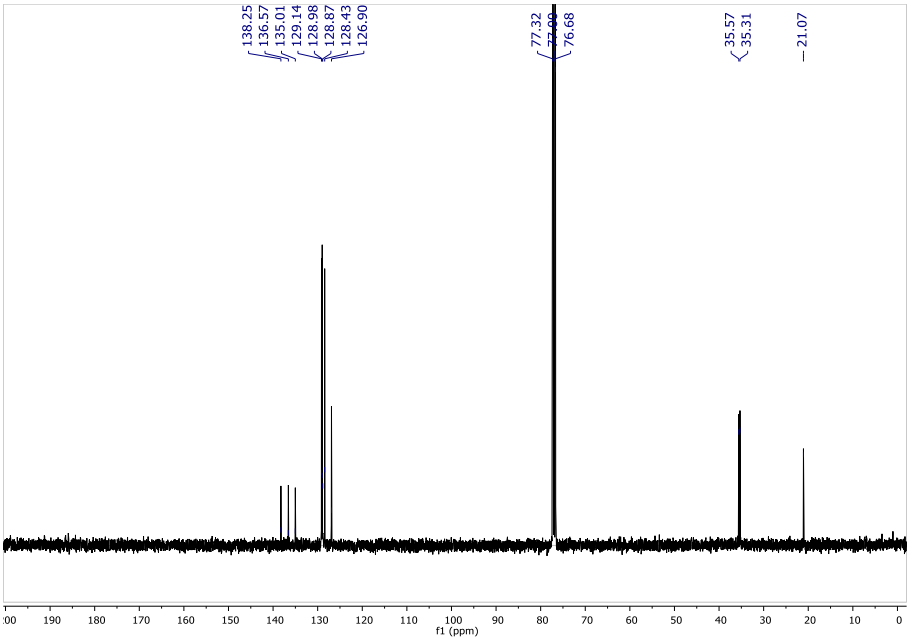
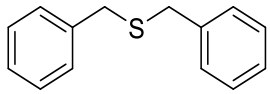


Figure S2 – $^{13}C\{^{1}H\}$-NMR of **2a**

Dibenzylsulfane 2b



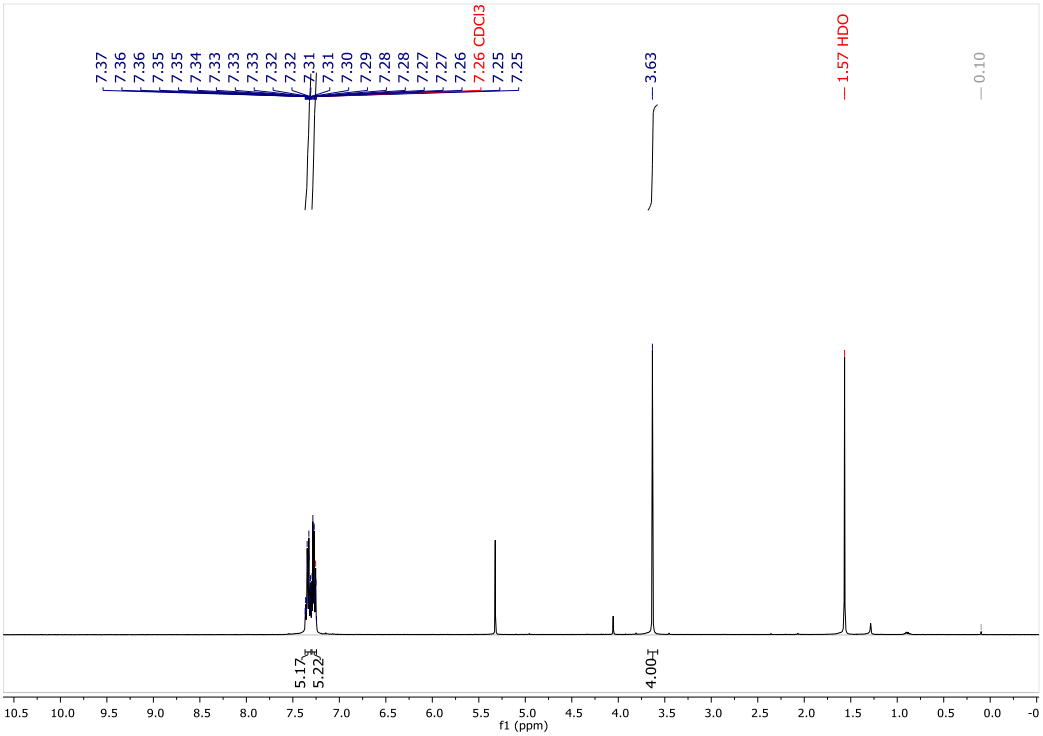


Figure S3 – ^{1}H-NMR of **2b**

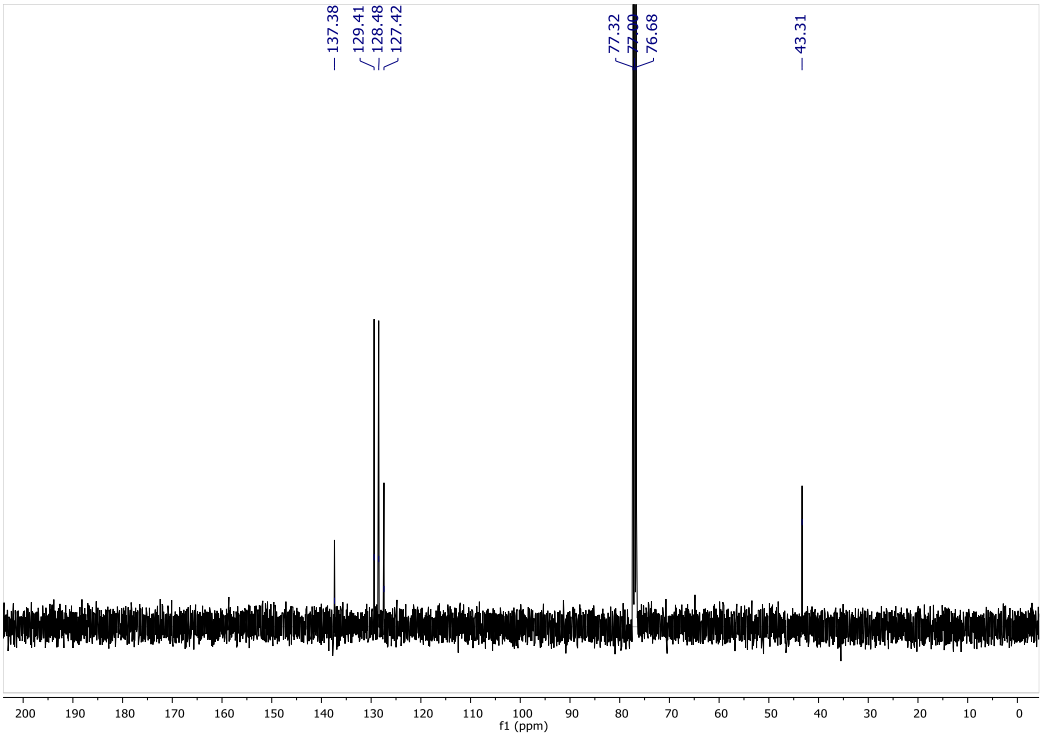
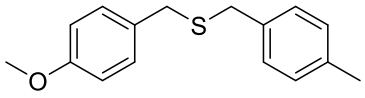


Figure S4 – ^{13}C{^{1}H}-NMR of **2b**

(4-Methoxybenzyl)(4-methylbenzyl)sulfane 2c



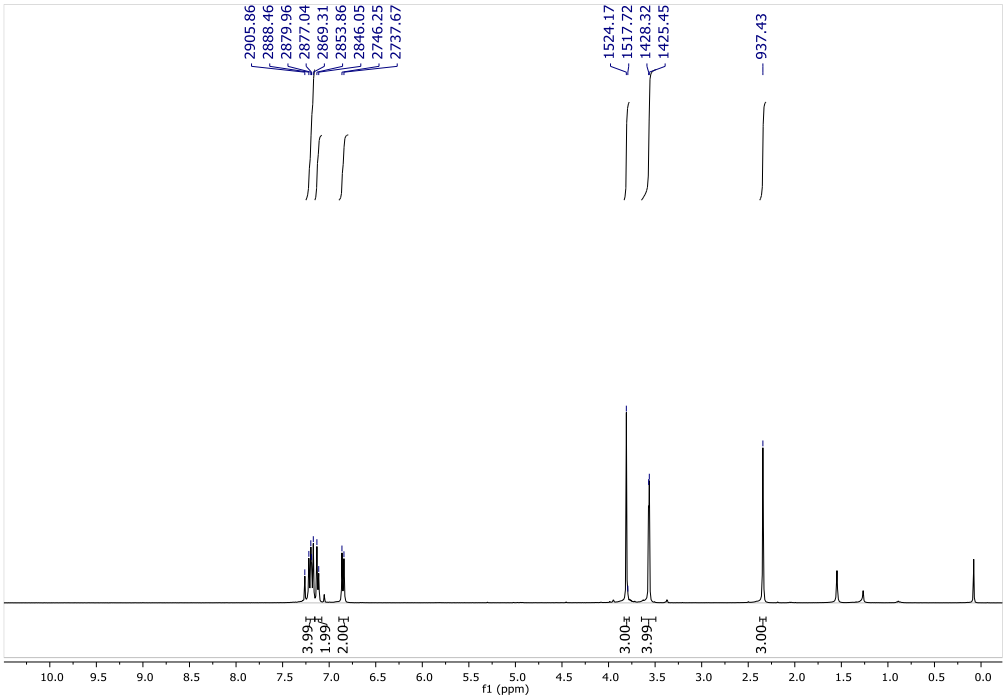


Figure S5 – ^{1}H-NMR of **2c**

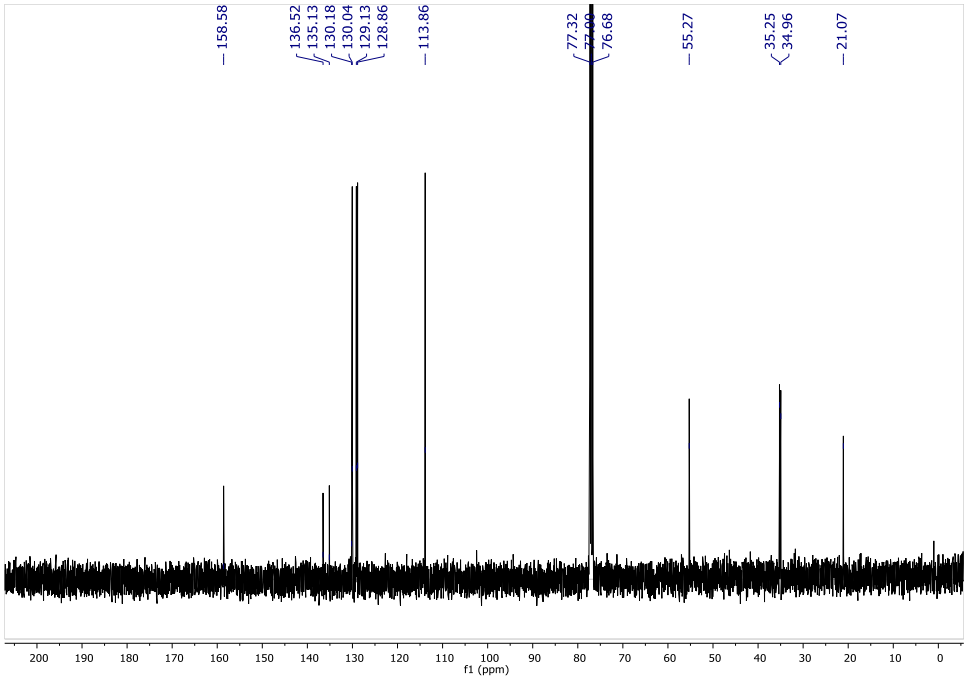
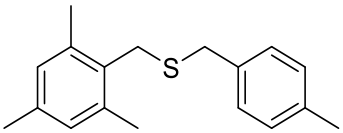


Figure S6 – ^{13}C{^{1}H}-NMR of **2c**

(4-Methylbenzyl)(2,4,6-trimethylbenzyl)sulfane 2d



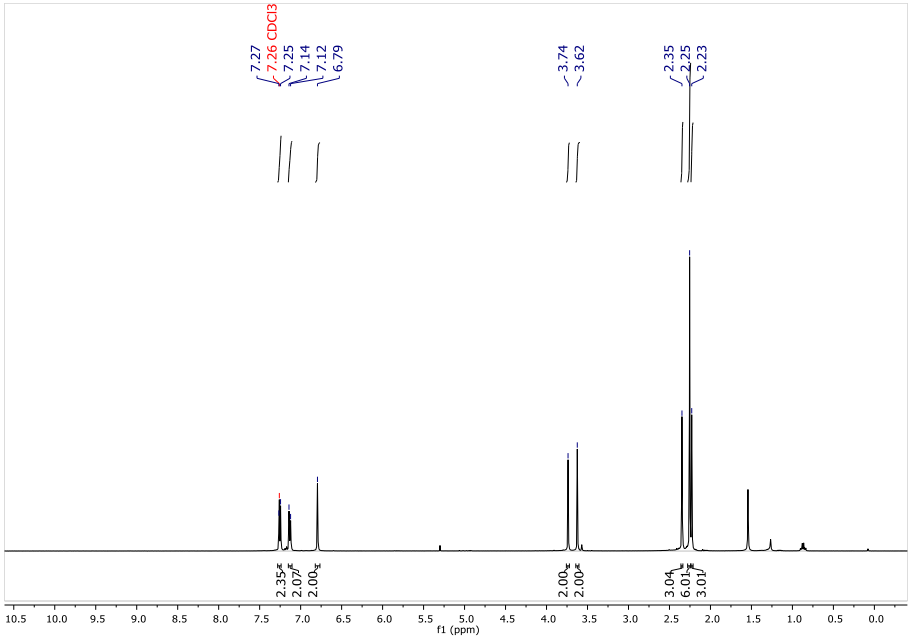


Figure S7 – ^{1}H-NMR of **2d**

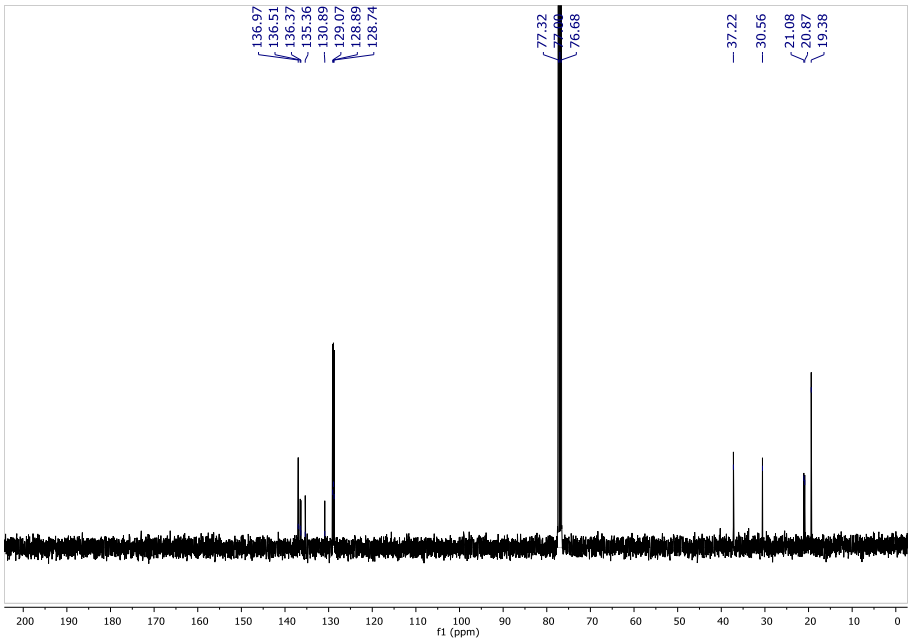
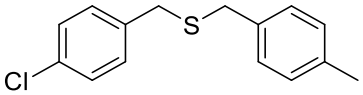


Figure S8 – ^{13}C{^{1}H}-NMR of **2d**

(4-Chlorobenzyl)(4-methylbenzyl)sulfane 2e



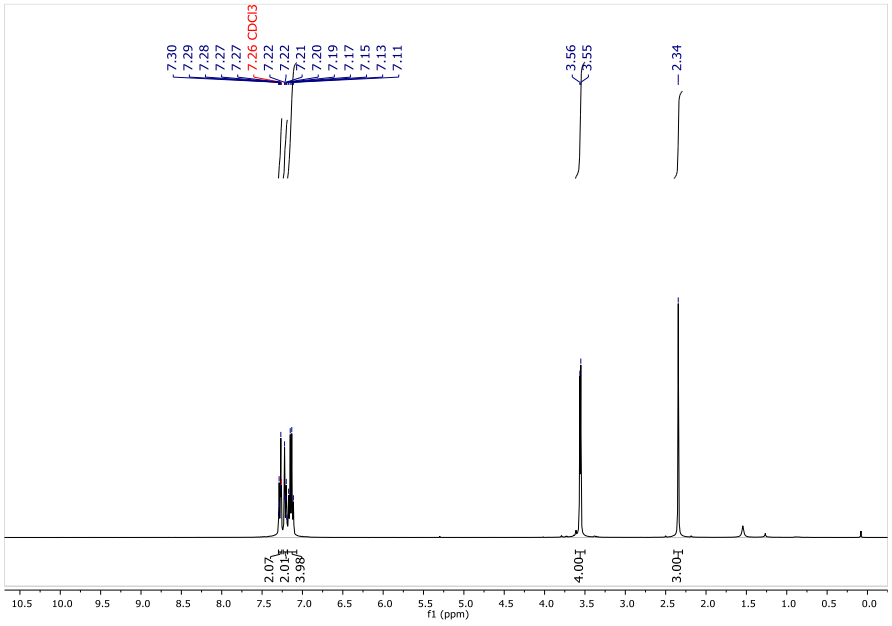


Figure S9 – ^{1}H-NMR of **2e**

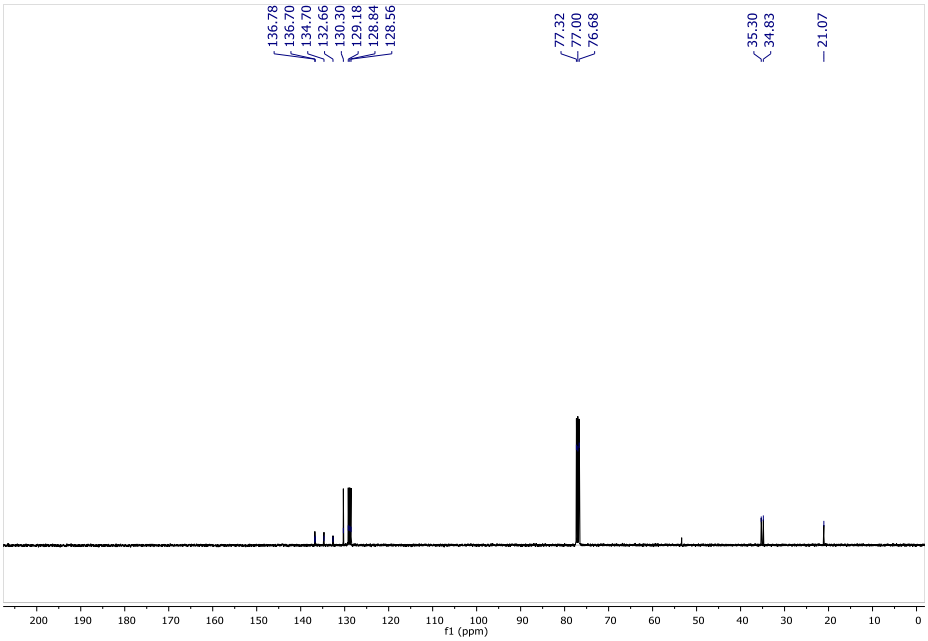
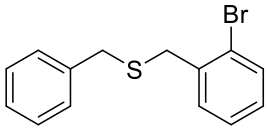


Figure S10 – ^{13}C{^{1}H}-NMR of **2e**

benzyl(2-bromobenzyl)sulfane 2f



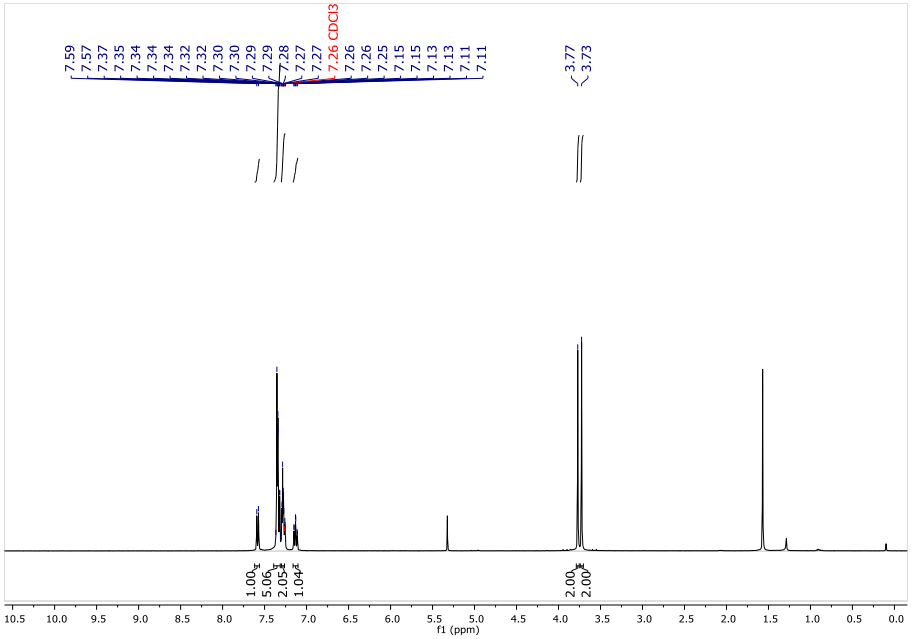


Figure S11 – ^{1}H-NMR of **2f**

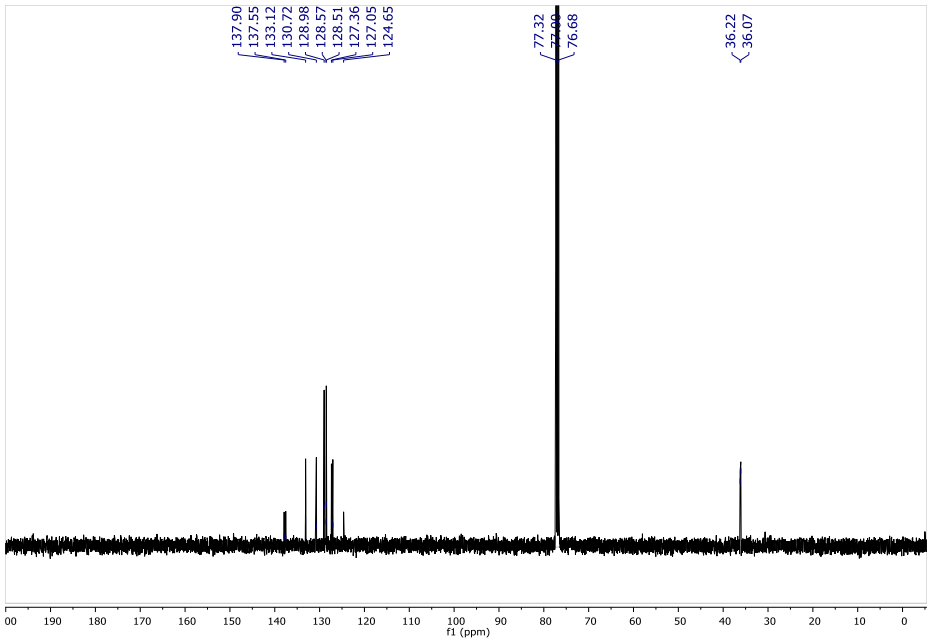
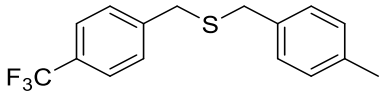


Figure S12 – ^{13}C{^{1}H}-NMR of **2f**

(4-Methylbenzyl)(4-(trifluoromethyl)benzyl)sulfane 2g



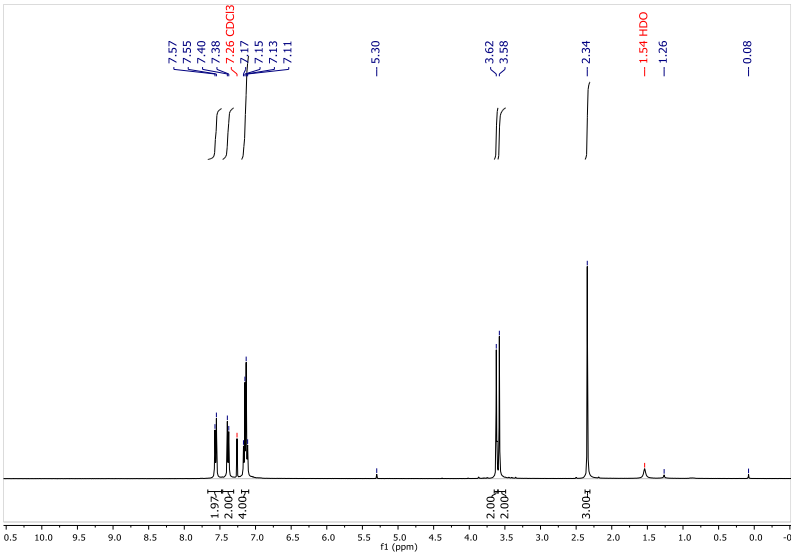


Figure S13 – ^{1}H-NMR of **2g**

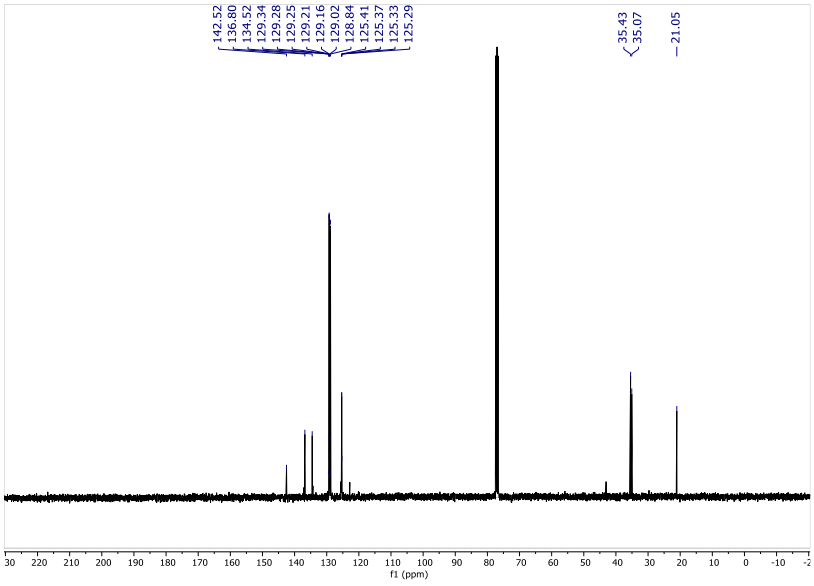


Figure S14 – ^{13}C{^{1}H}-NMR of **2g**

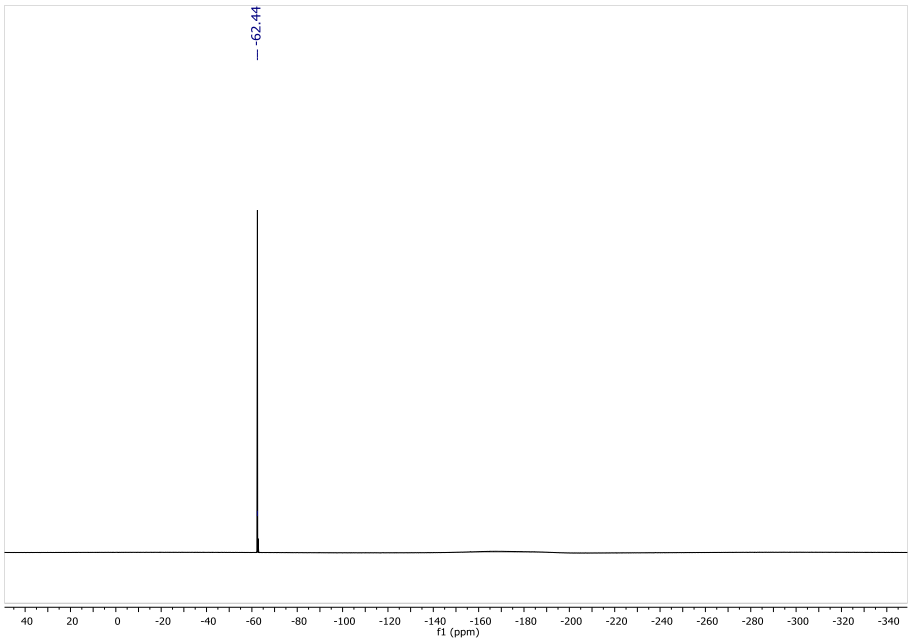
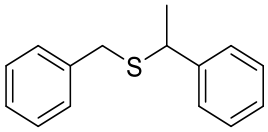


Figure S15 – ^{19}F-NMR of **2g**

Benzyl(1-phenylethyl)sulfane 2h



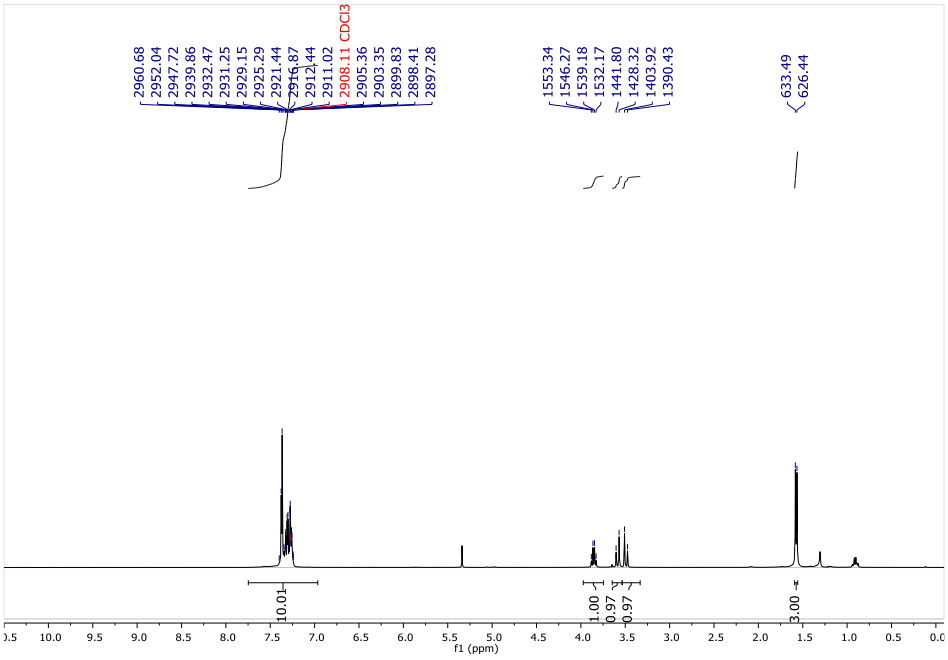


Figure S16 – ^{1}H-NMR of **2h**

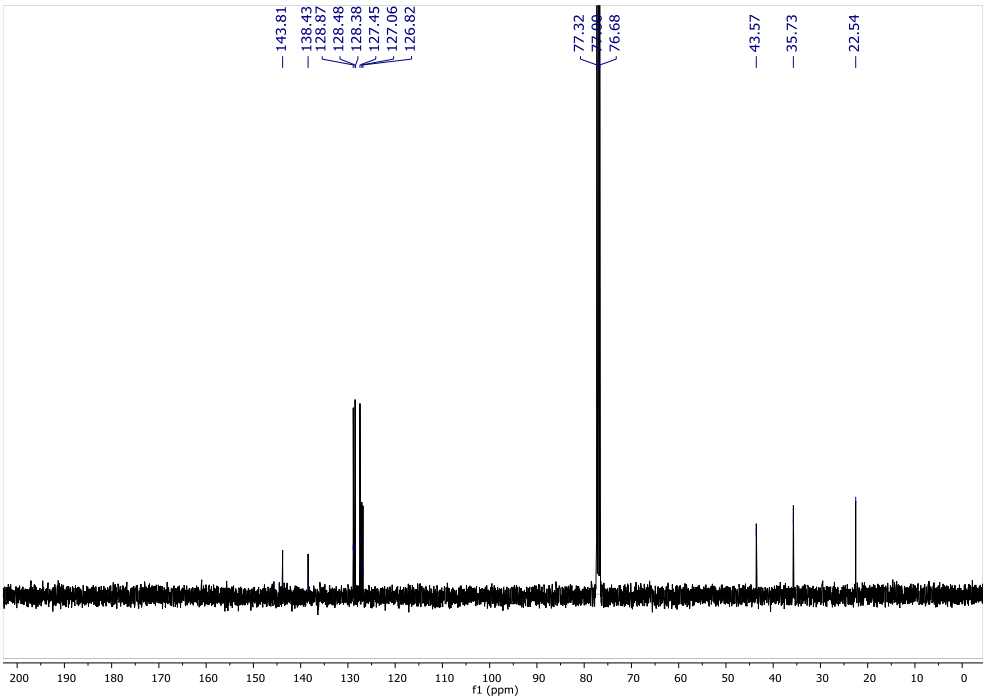
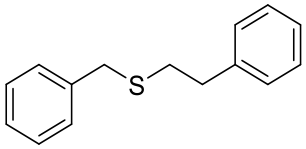


Figure S17 – $^{13}C\{^{1}H\}$-NMR of **2h**

Benzyl(phenethyl)sulfane 2i



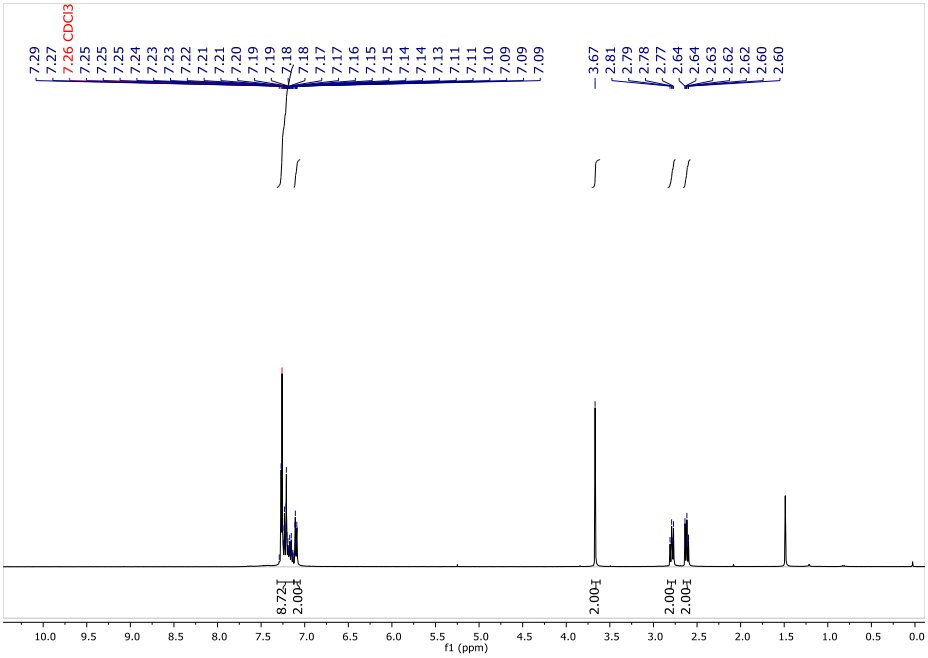


Figure S18 – ^{1}H-NMR of **2i**

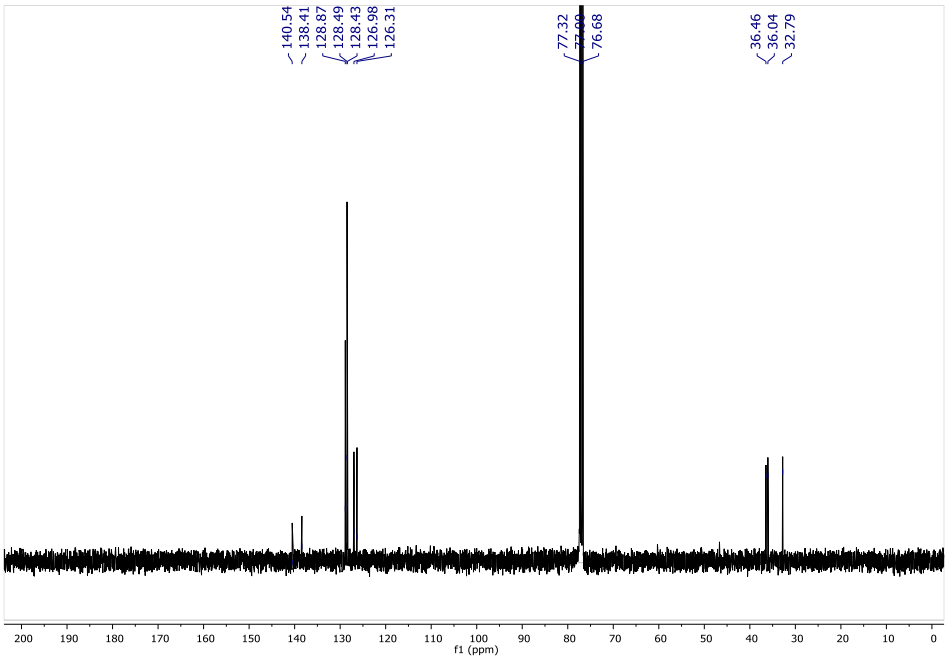
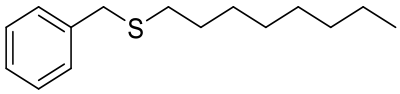


Figure S19 – $^{13}C\{^{1}H\}$-NMR of **2i**

benzyl(octyl)sulfane 2j



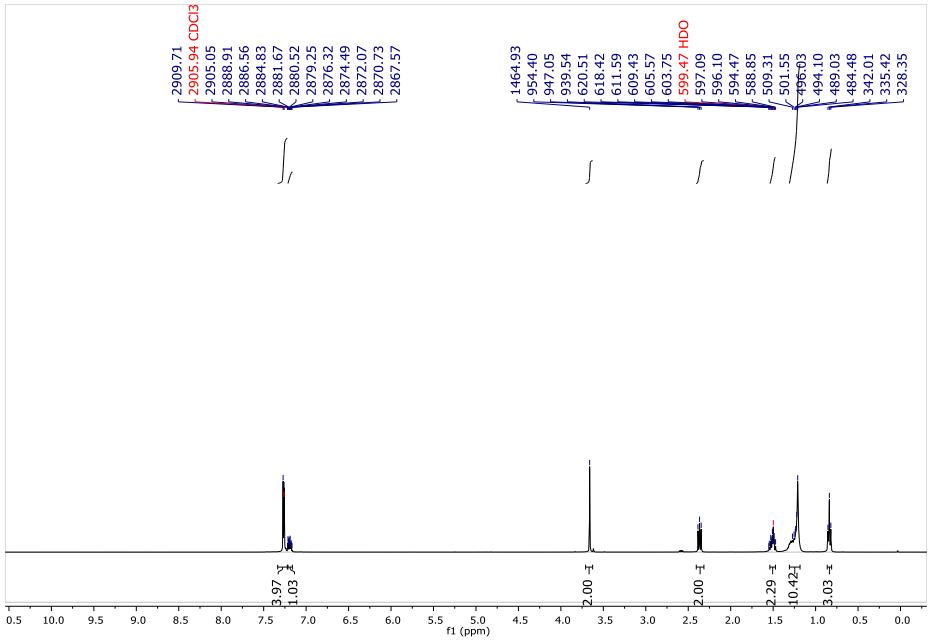


Figure S20 – ^{1}H-NMR of **2j**

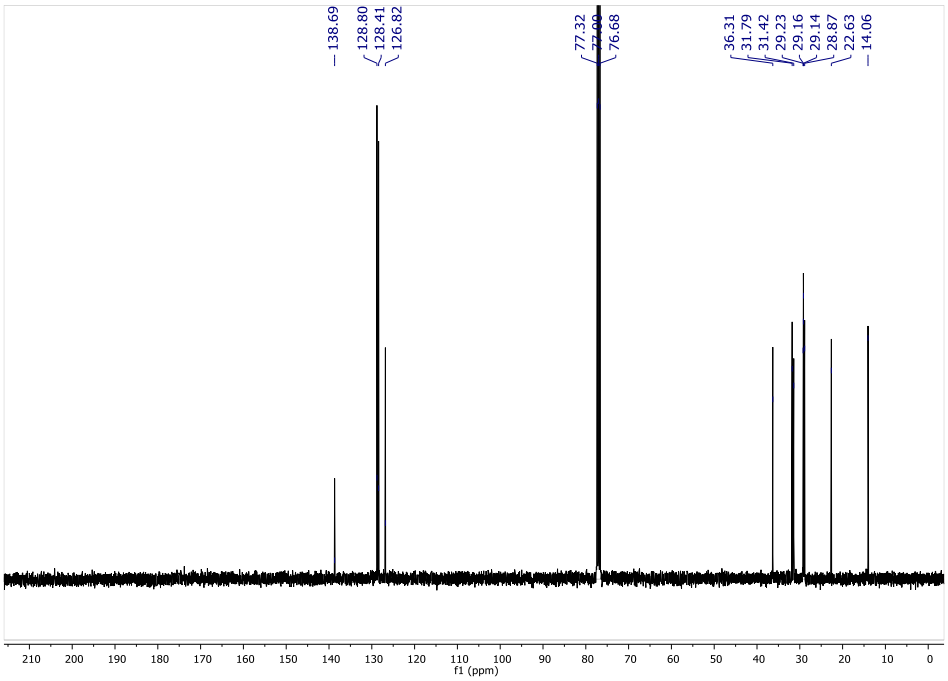
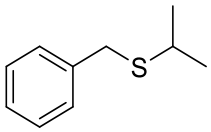


Figure S21 – $^{13}C\{^{1}H\}$-NMR of **2j**

Benzyl(isopropyl)sulfane 2k



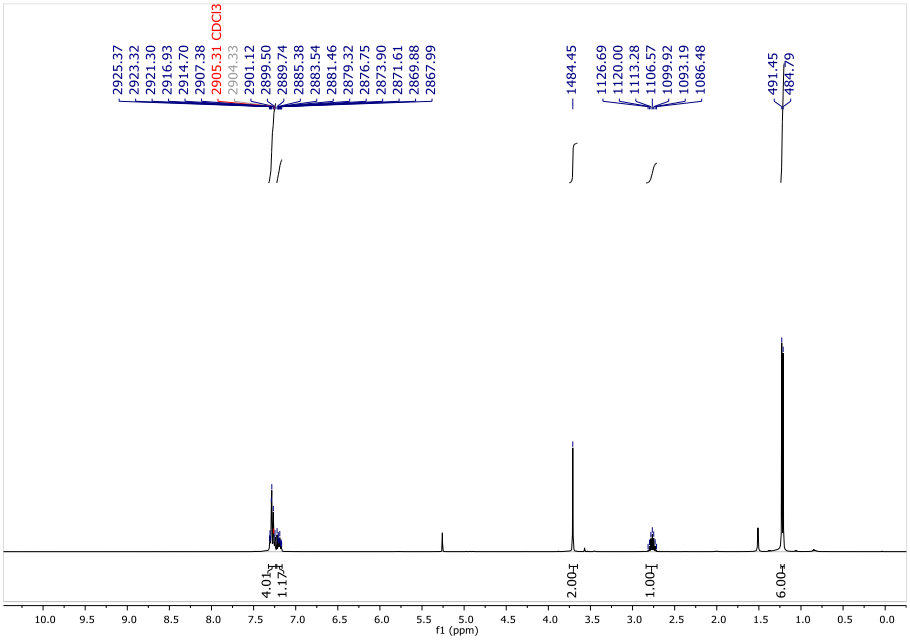


Figure S22 – ^{1}H-NMR of **2k**

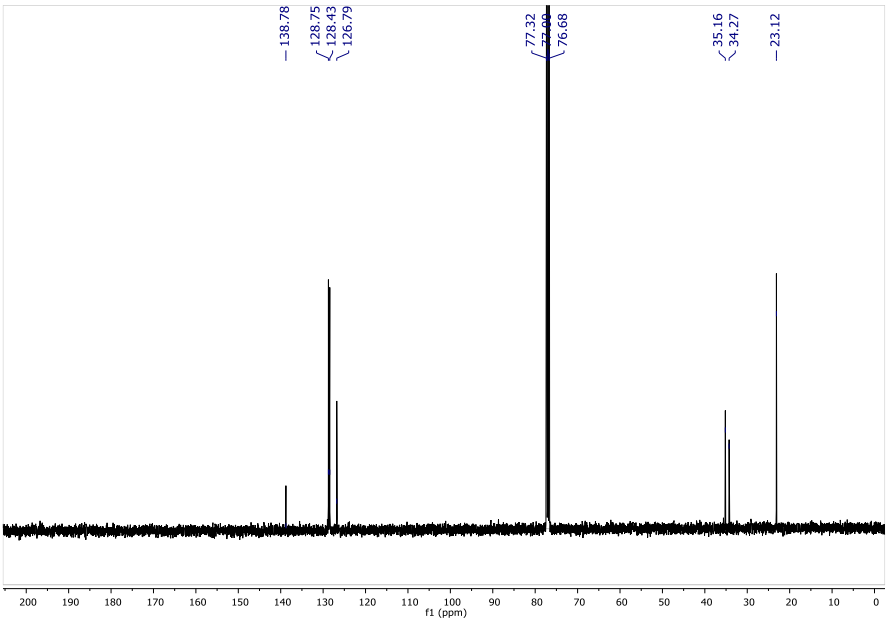
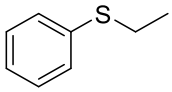


Figure S23 – $^{13}C\{^{1}H\}$-NMR of **2k**

Ethyl(phenyl)sulfane 2l



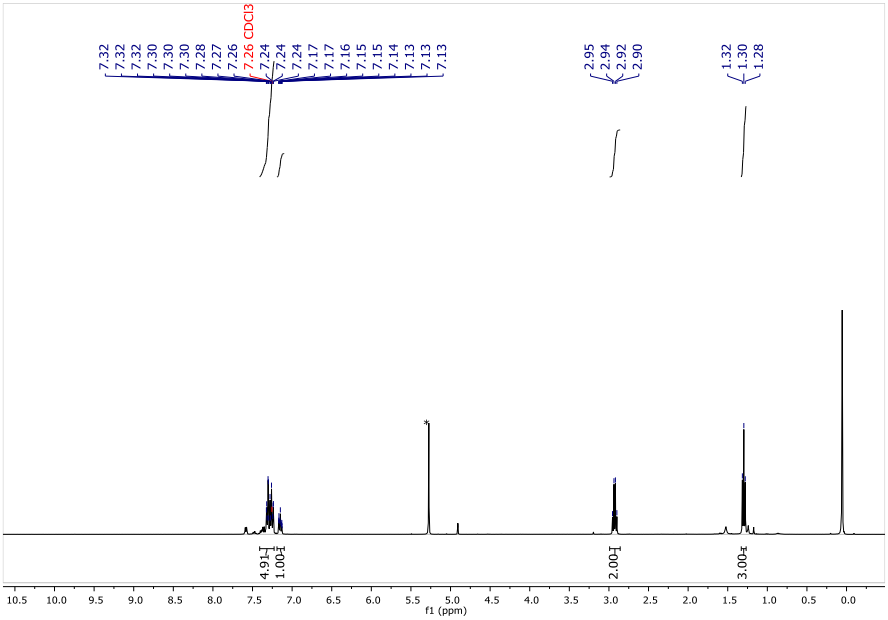


Figure S24 – ^{1}H-NMR of **2l.** * CH_2Cl_2 (difficult to eliminate due to volatility of **2l**).

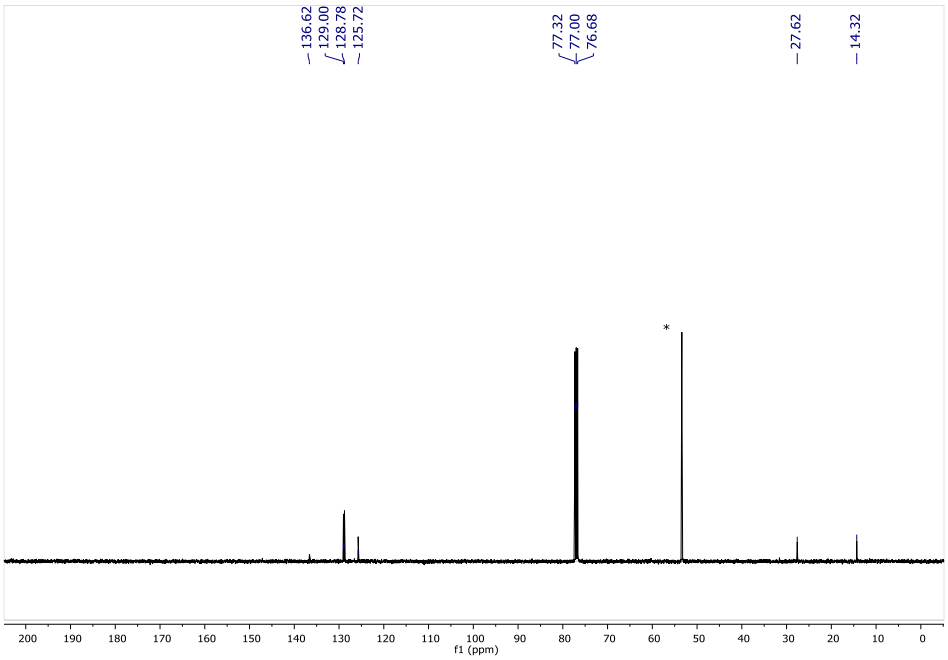
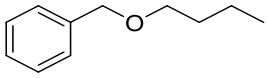


Figure S25 – $^{13}C\{^{1}H\}$-NMR of **2l.** * CH_2Cl_2 difficult to eliminate due to volatility of **2l**).

*

(Butoxymethyl)benzene 5a



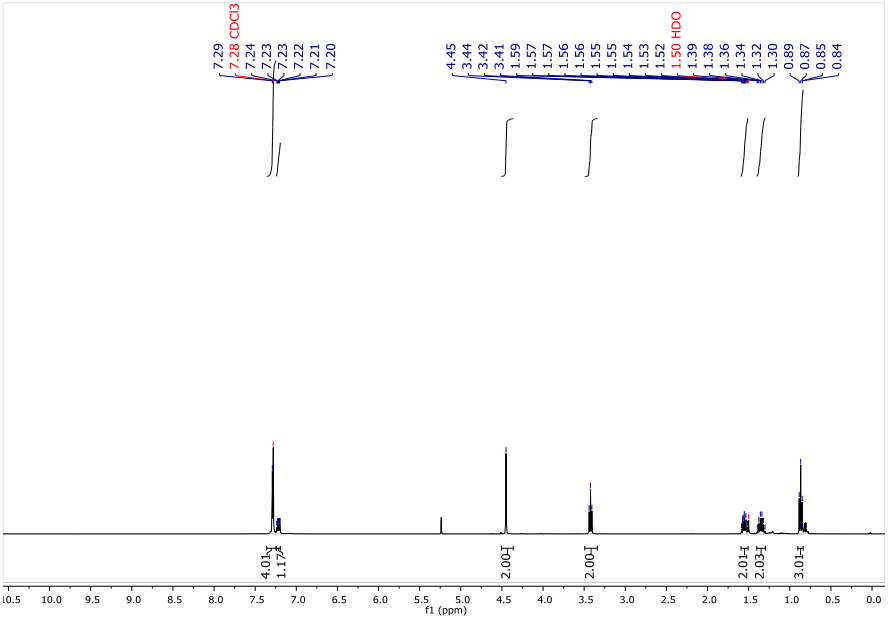


Figure S26 – ^{1}H-NMR of **5a**

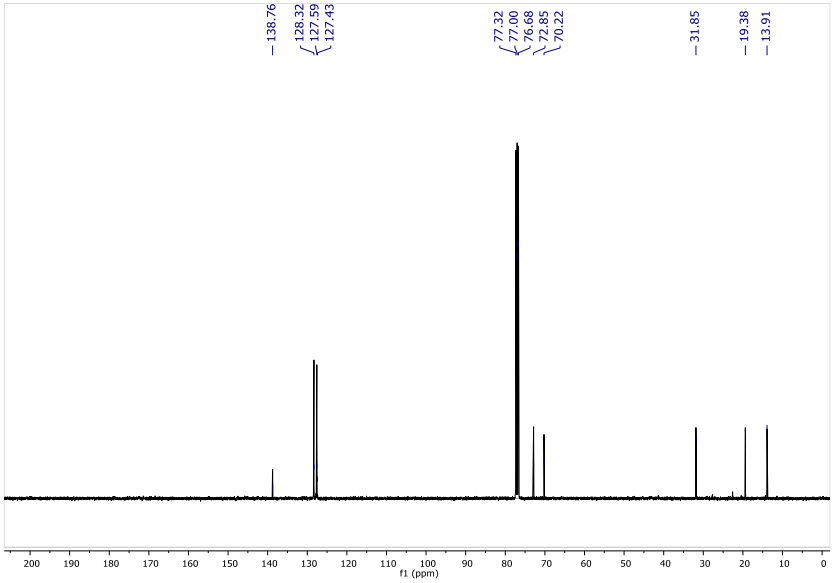
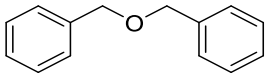


Figure S27 – $^{13}C\{^{1}H\}$-NMR of **5a**

Dibenzyl ether 5a



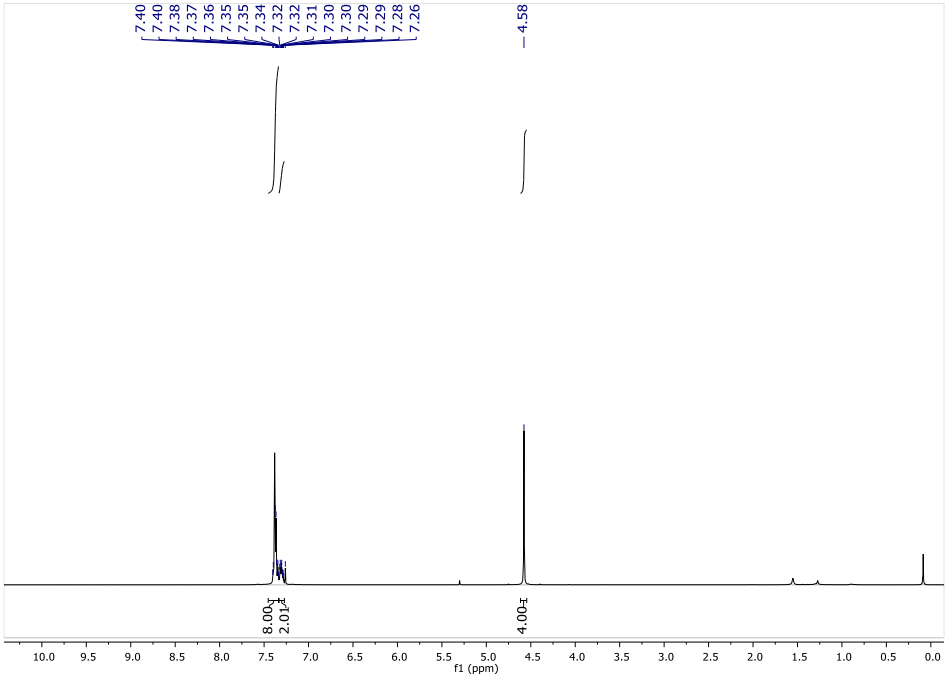


Figure S28 – ^{1}H-NMR of **5b**

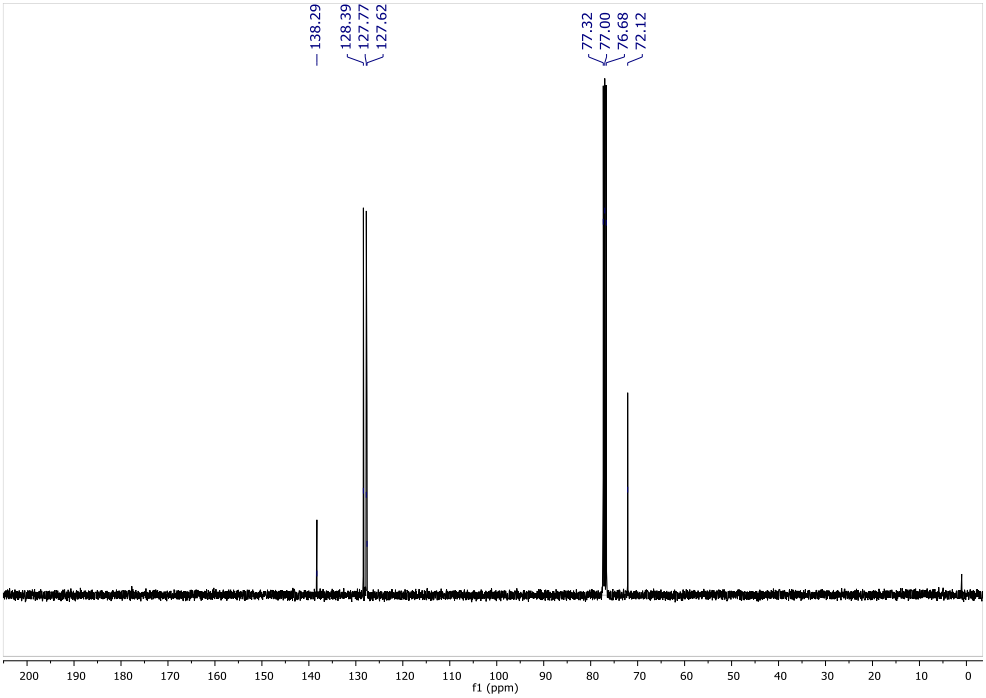
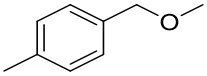


Figure S29 – $^{13}C\{^{1}H\}$-NMR of **5b**

1-(Methoxymethyl)-4-methylbenzene 5c



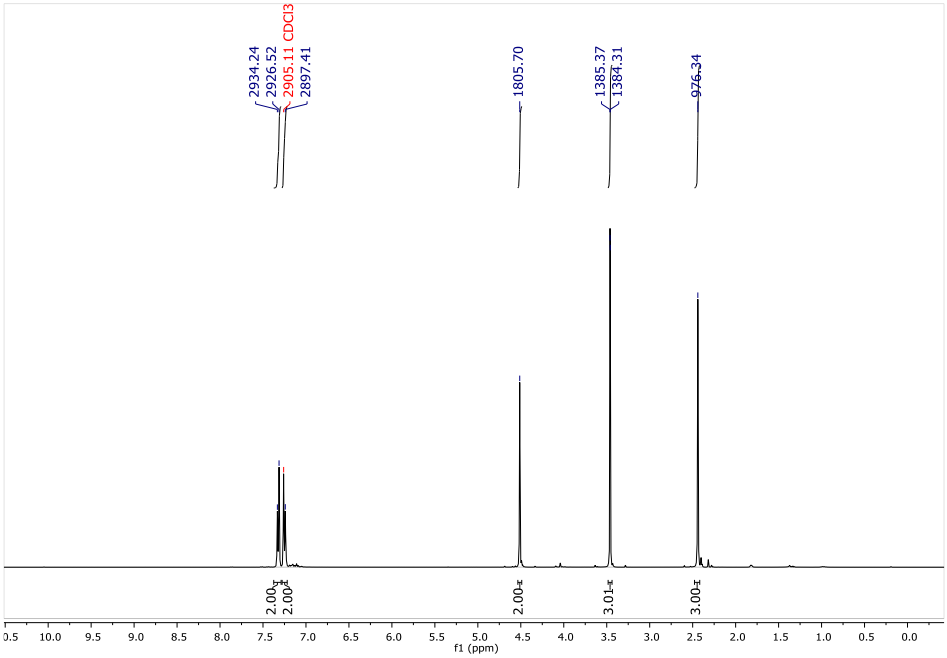


Figure S30 – ^{1}H-NMR of **5c**

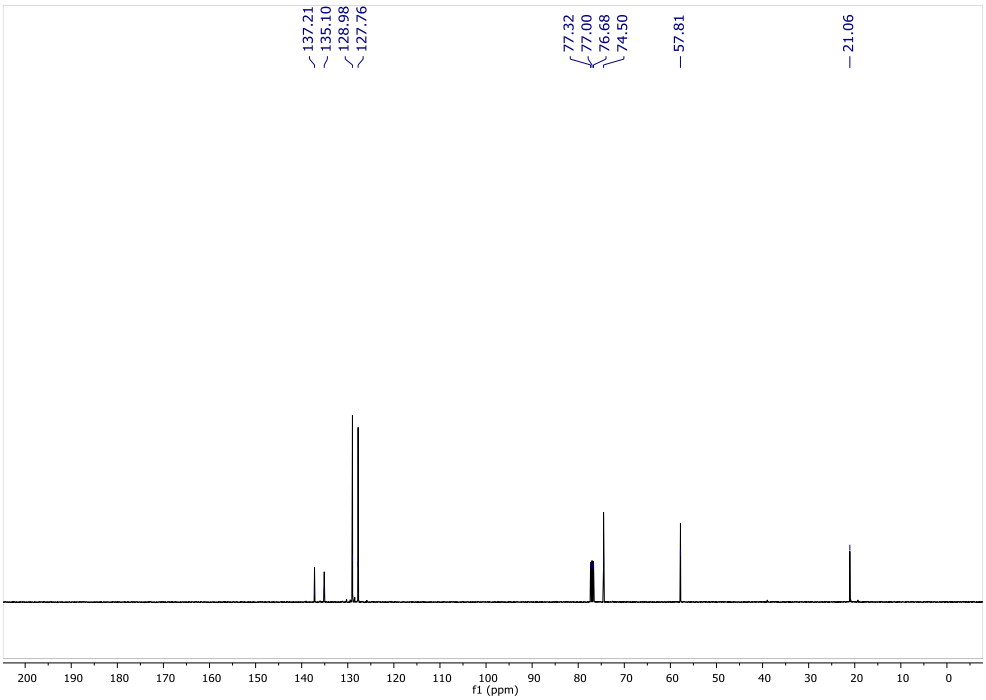
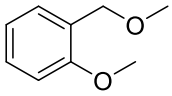


Figure S31 – $^{13}C\{^{1}H\}$-NMR of **5c**

1-Methoxy-2-(methoxymethyl)benzene 5d



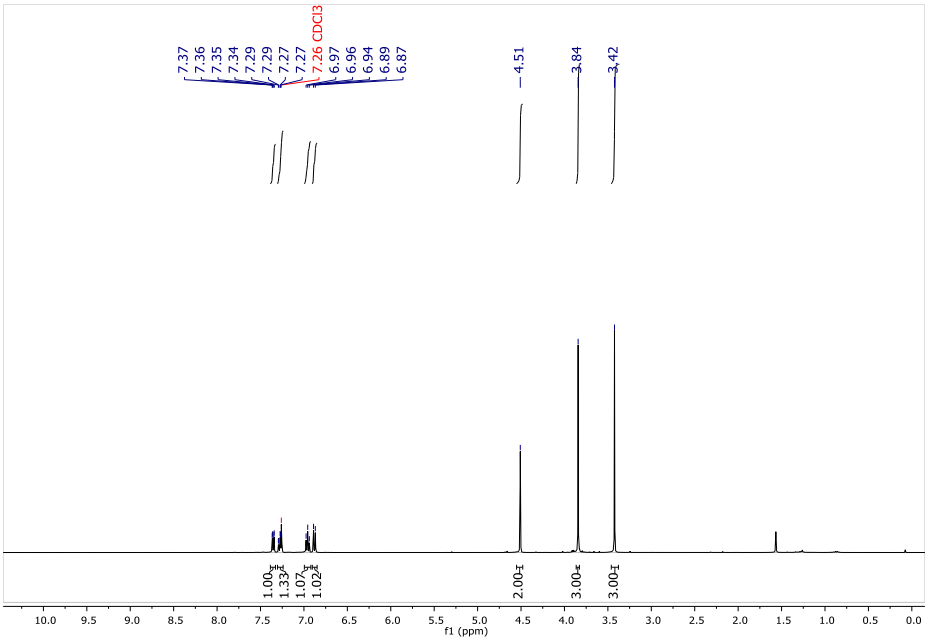


Figure S32 – ^{1}H-NMR of **5d**

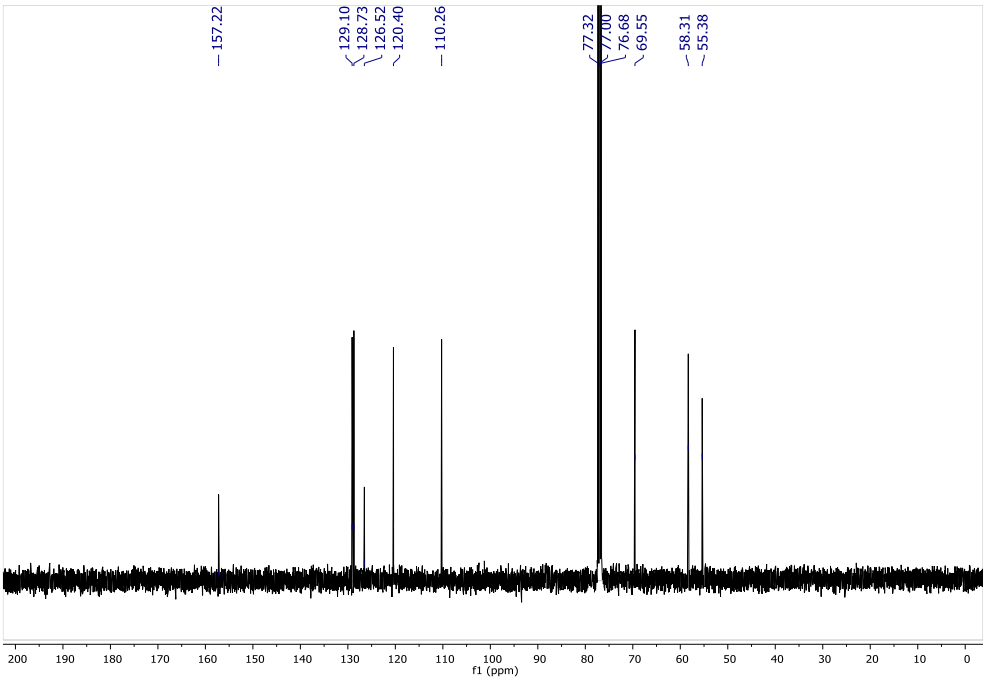
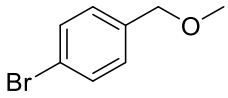


Figure S33 – $^{13}C\{^{1}H\}$-NMR of **5d**

1-Bromo-4-(methoxymethyl)benzene 5e



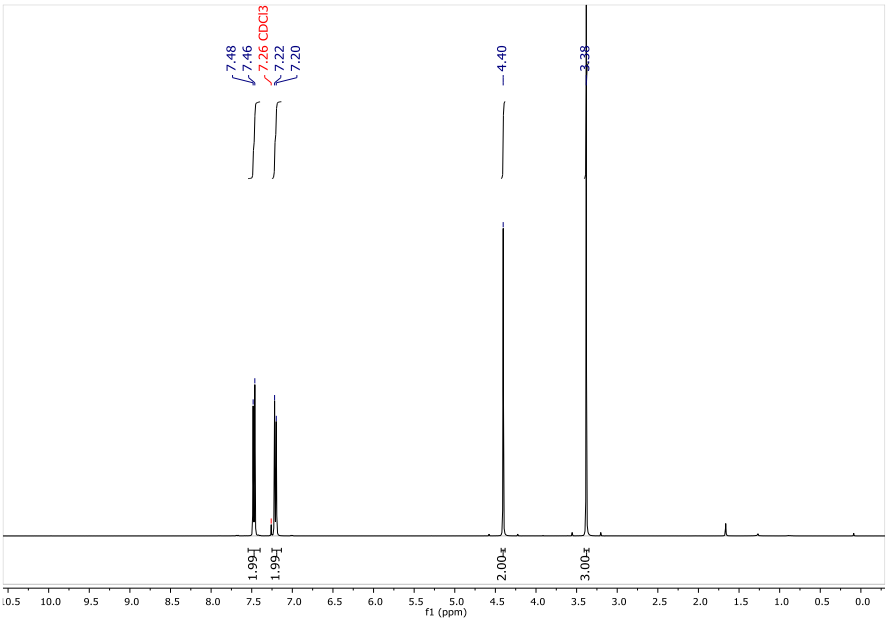


Figure S34 – ^{1}H-NMR of **5e**

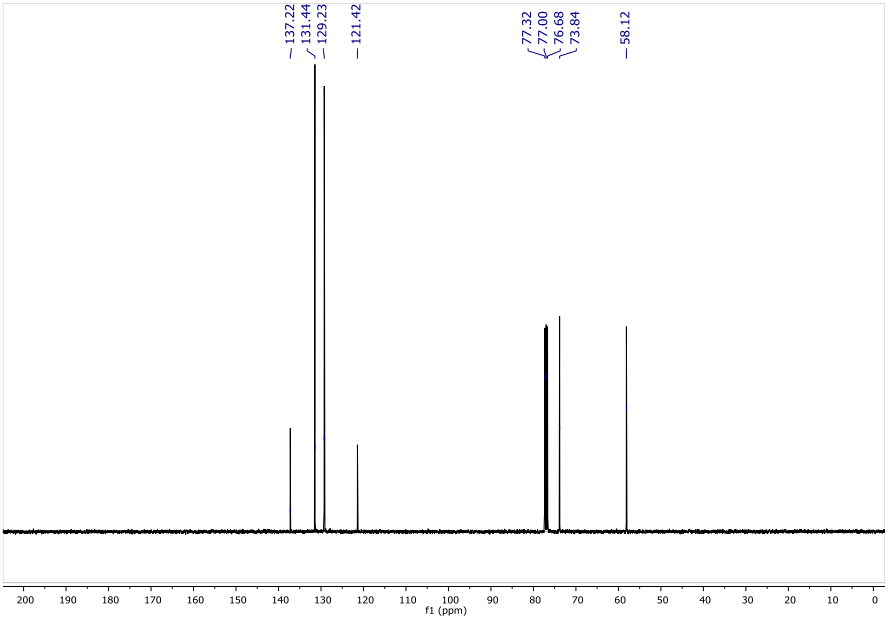
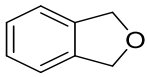


Figure S35 – $^{13}C\{^{1}H\}$-NMR of **5e**

1,3-dihydroisobenzofuran 5f



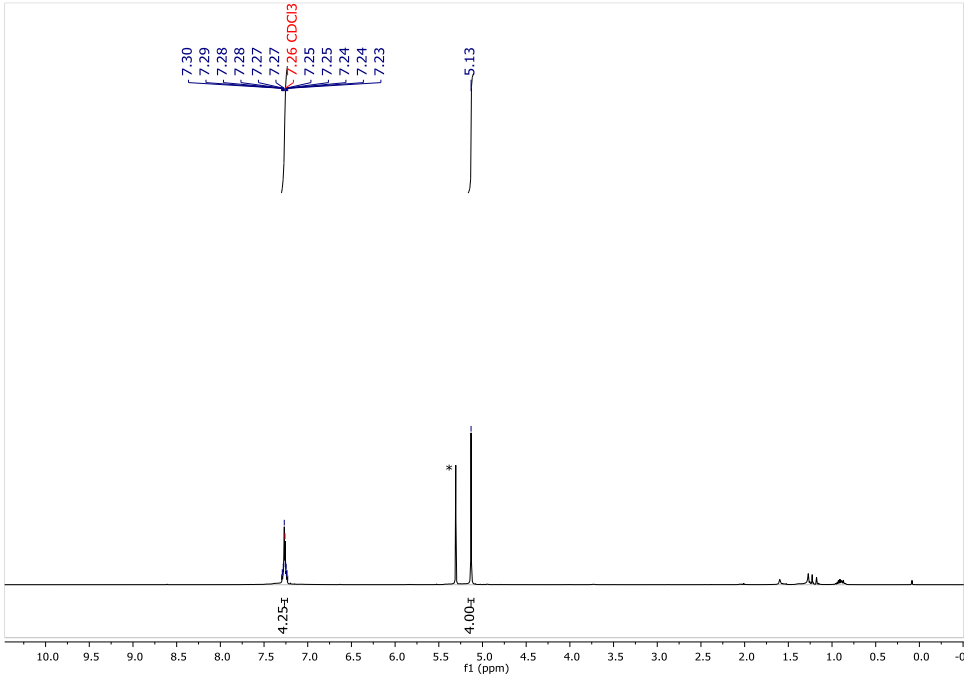


Figure S36 – ^{1}H-NMR of **5f**. * Peak corresponding to CH_2Cl_2.

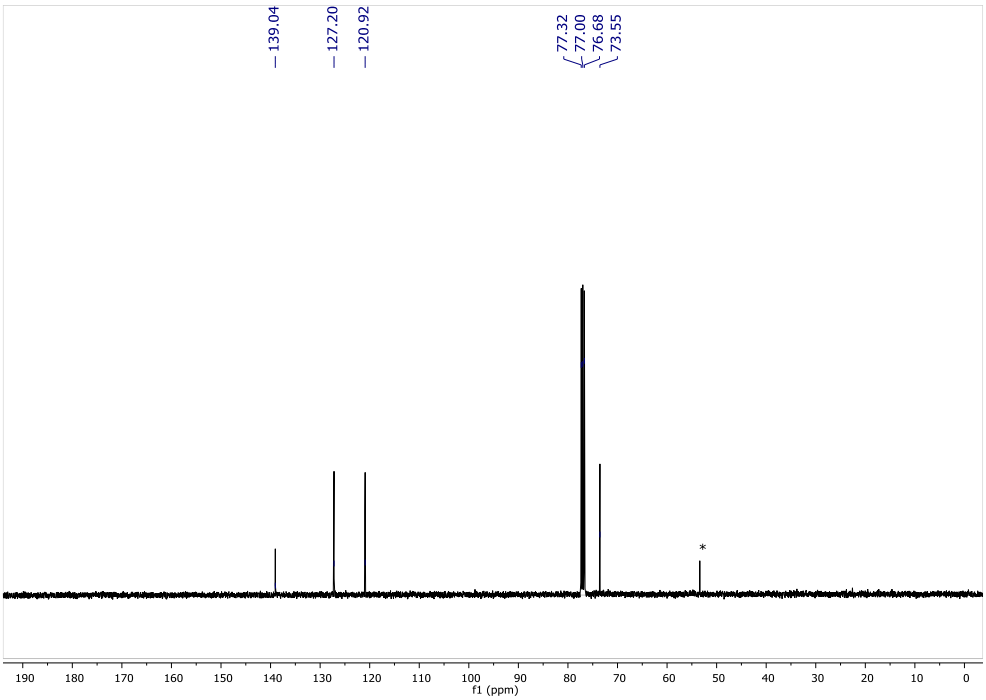
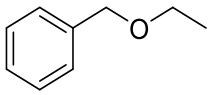


Figure S37 – $^{13}C\{^{1}H\}$-NMR of **5f.** * Peak corresponding to CH_2Cl_2.

(Ethoxymethyl)benzene 5g



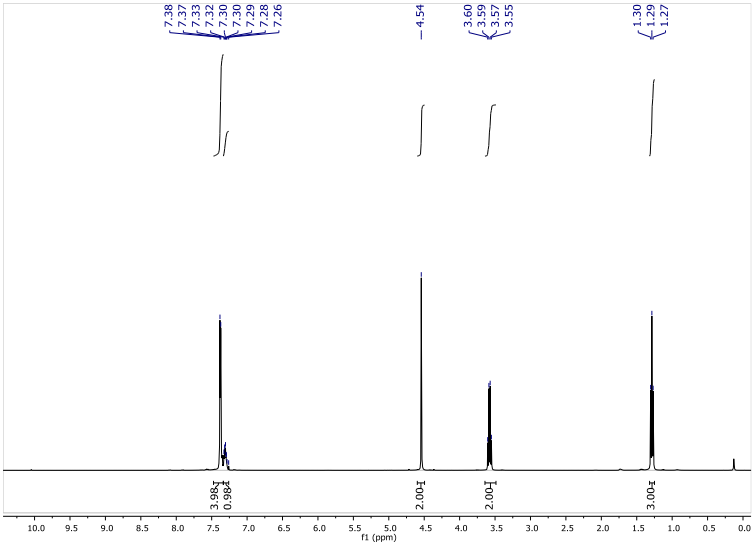


Figure S38 – ^{1}H-NMR of **5g**

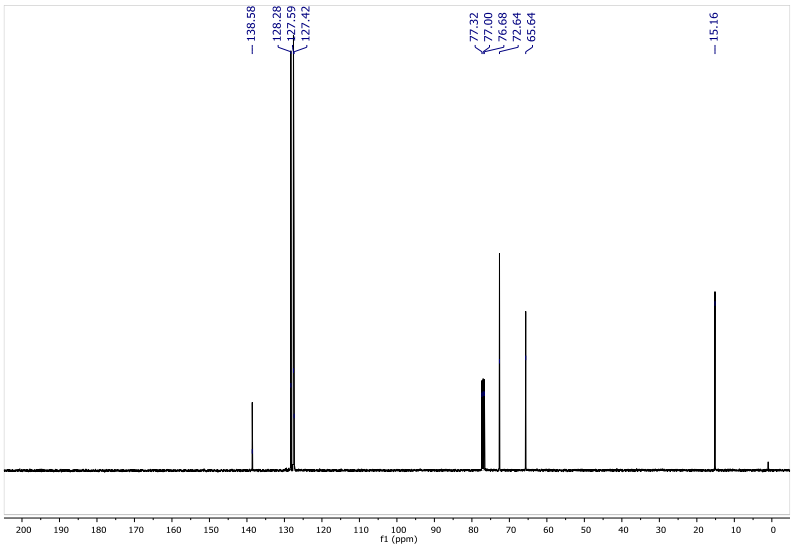
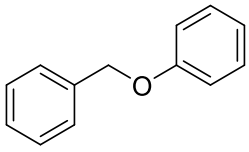


Figure S39 – $^{13}C\{^{1}H\}$-NMR of **5g**

(Benzyloxy)benzene 5h



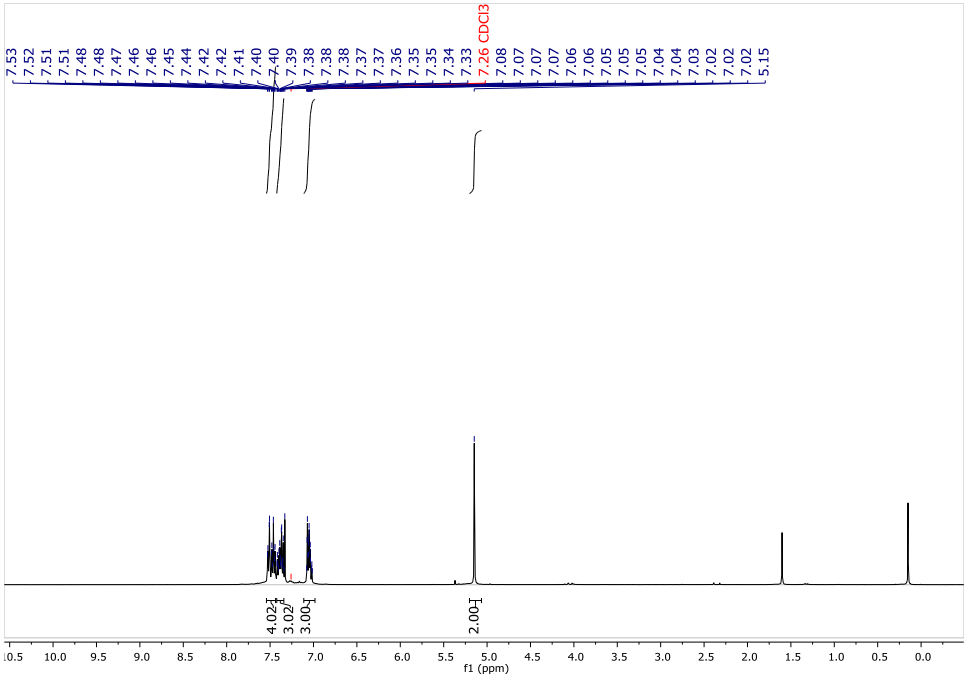


Figure S40 – ^{1}H-NMR of **7e**

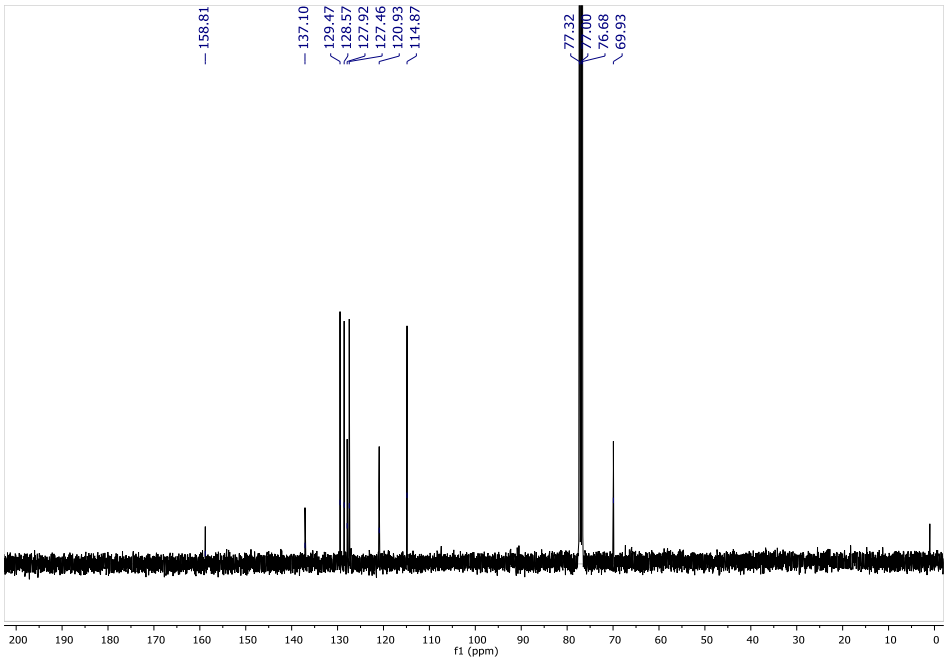
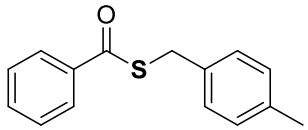


Figure S41 – $^{13}C\{^{1}H\}$-NMR of **7e**

S-(4-Methylbenzyl) benzothioate 1a



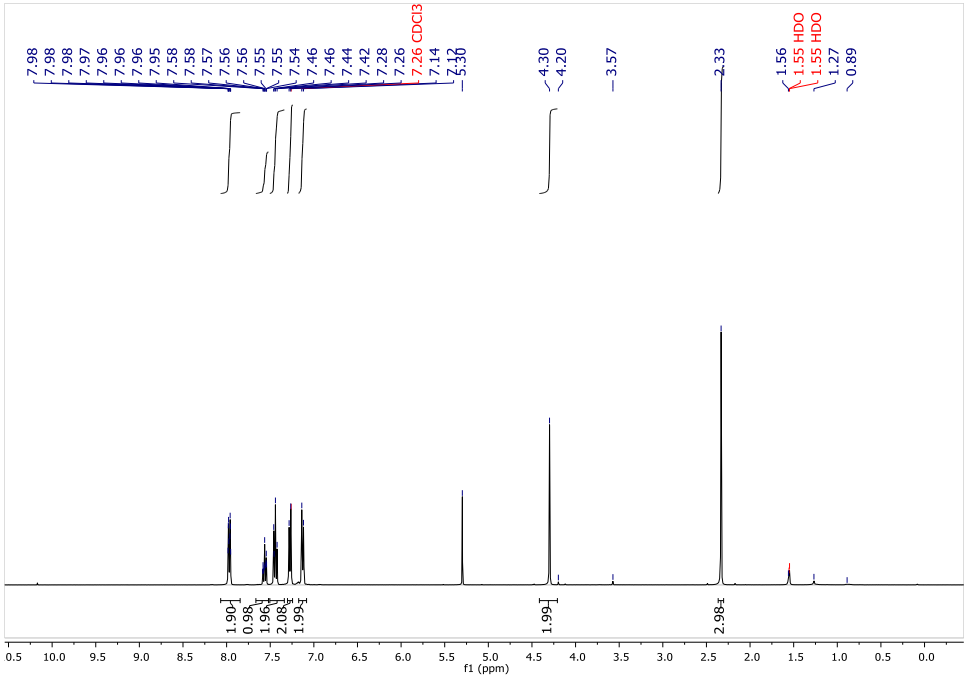


Figure S42 – ^{1}H-NMR of **1a**

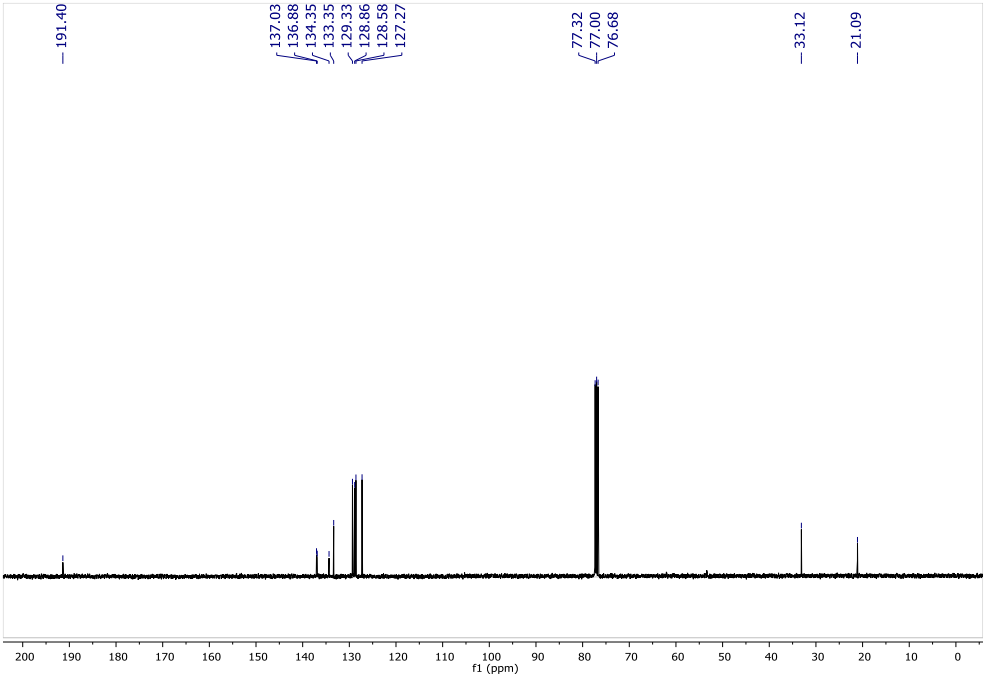
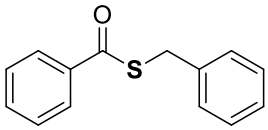


Figure S43 – ^{13}C{^{1}H}-NMR of **1a**

S-Benzyl benzothioate 1b



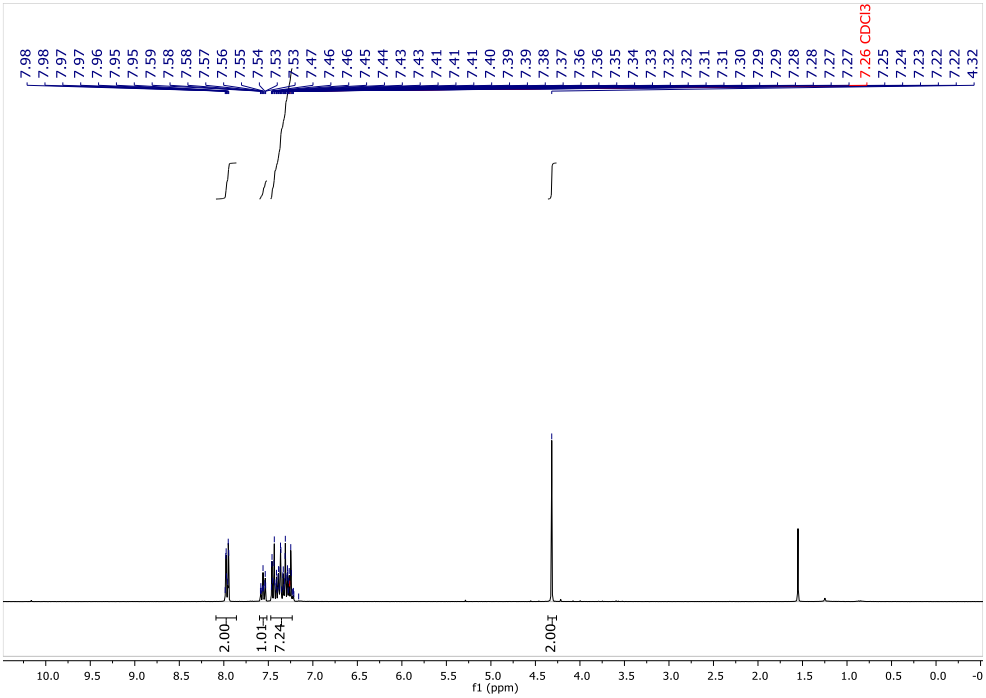


Figure S44 – ^{1}H-NMR of **1b**

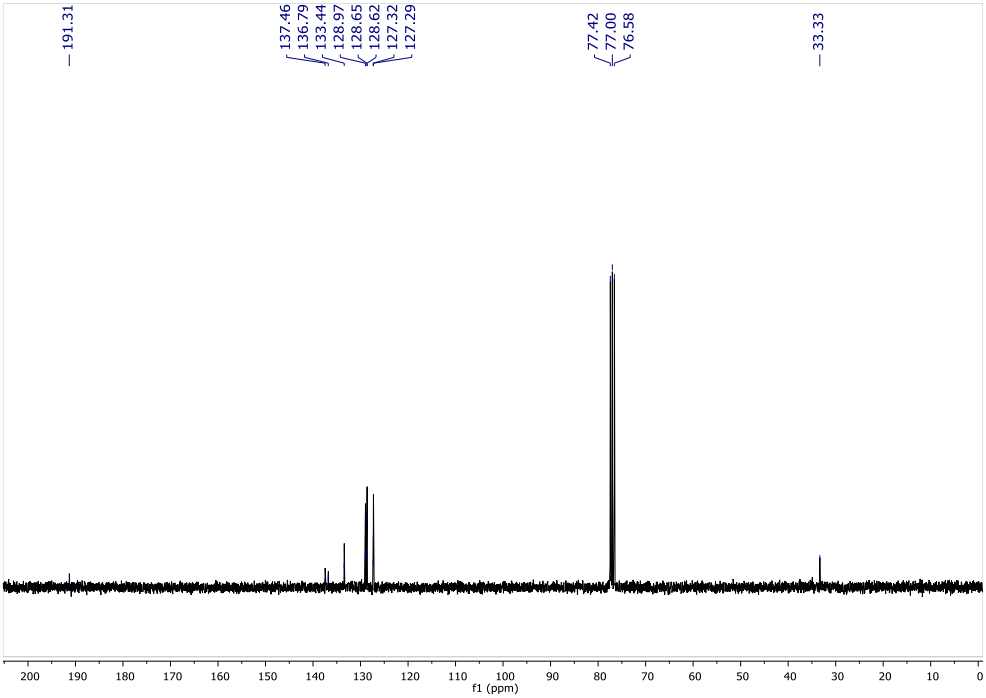
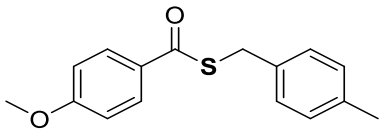


Figure S45 – ^{13}C{^{1}H}-NMR of **1b**

S-(4-Methylbenzyl) 4-methoxybenzothioate 1c



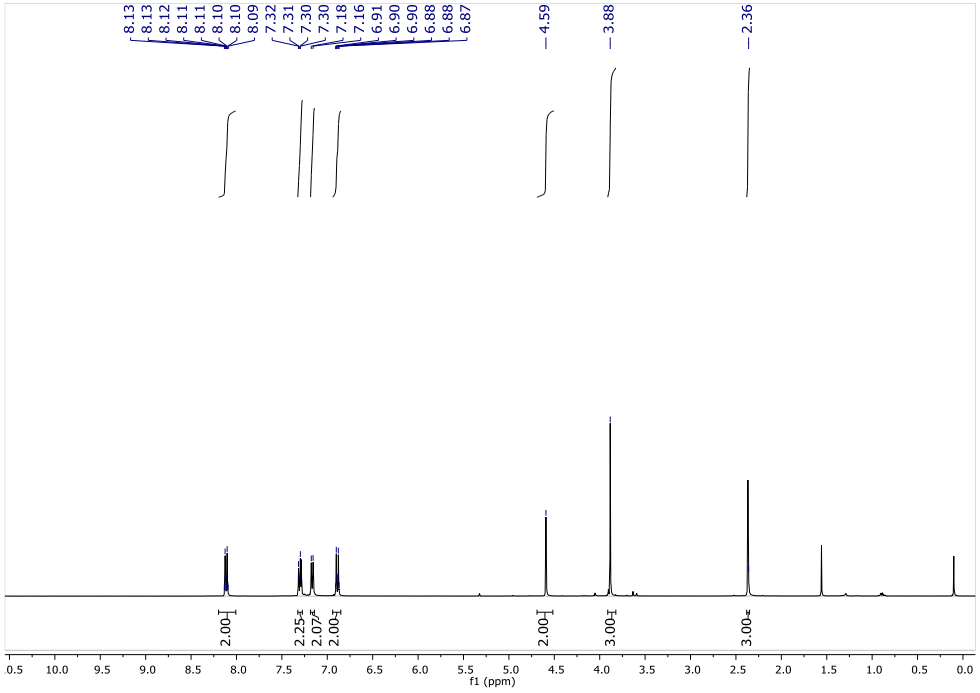


Figure S46 – ^{1}H-NMR of **1c**

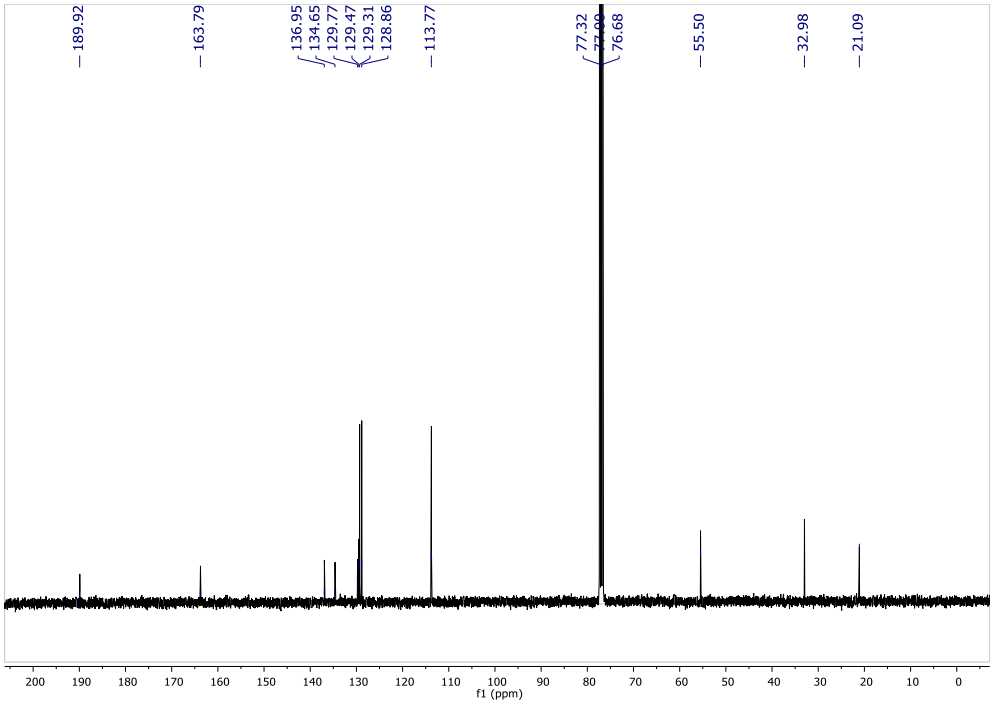
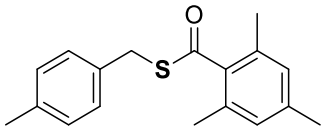


Figure S47 – ^{13}C{^{1}H}-NMR of **1c**

S-(4-Methylbenzyl) 2,4,6-trimethylbenzothioate 1d



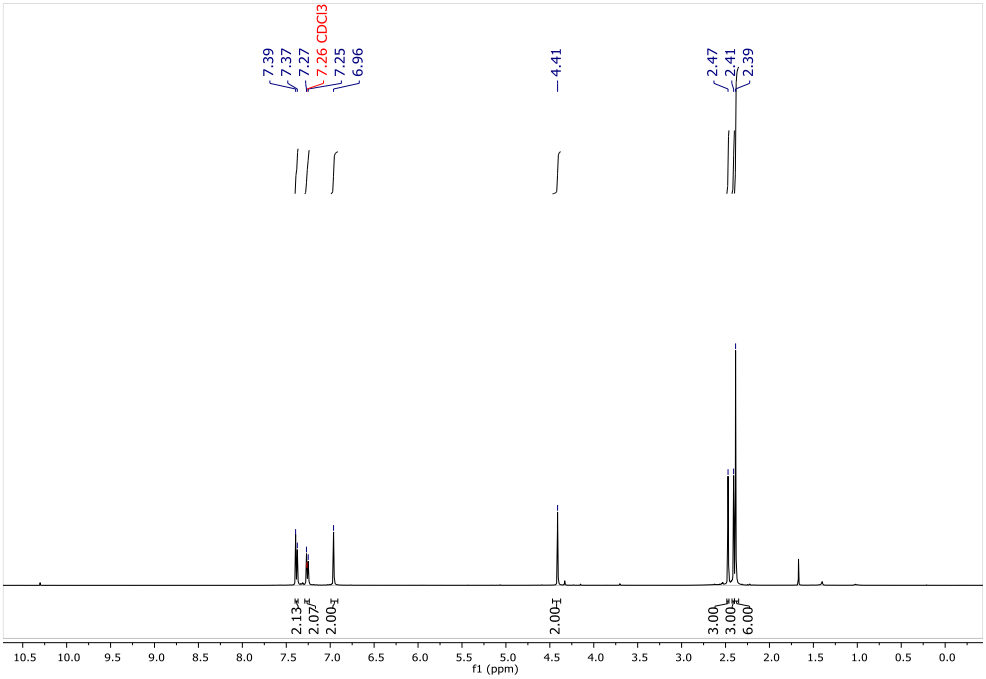


Figure S48 – ^{1}H-NMR of **1d**

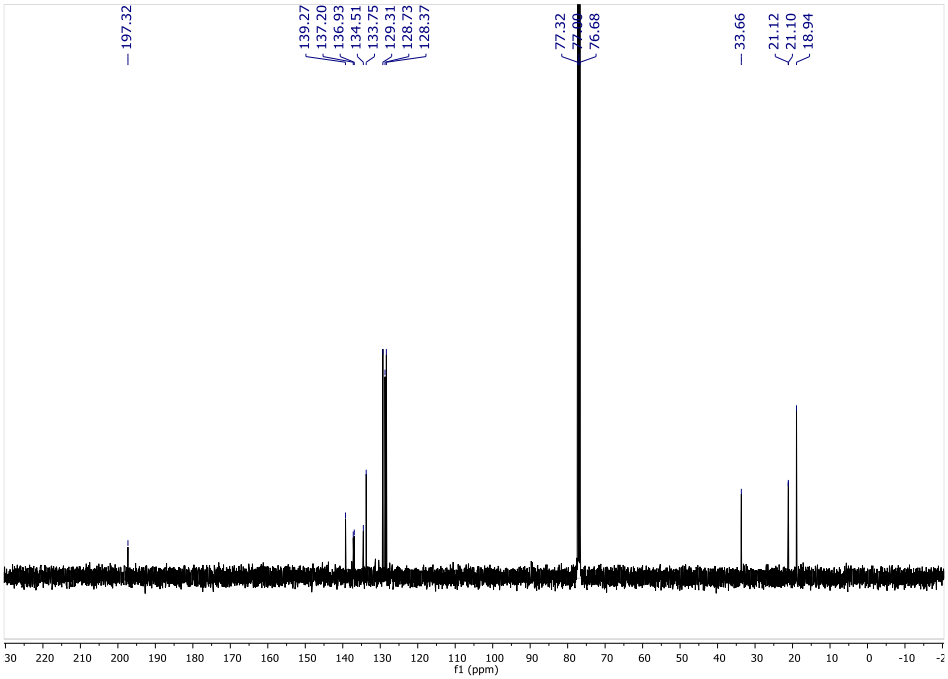
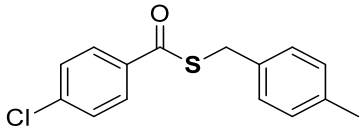


Figure S49 – ^{13}C{^{1}H}-NMR of **1d**

S-(4-Methylbenzyl) 4-chlorobenzothioate 1e



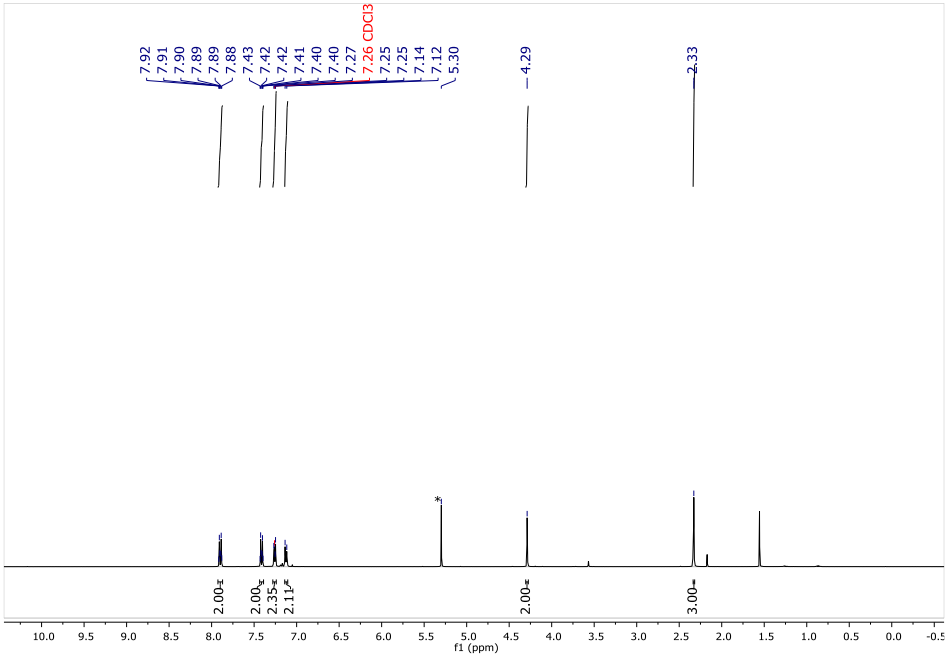


Figure S50 – ^{1}H-NMR of **1e.** * Trace of CH_2Cl_2.

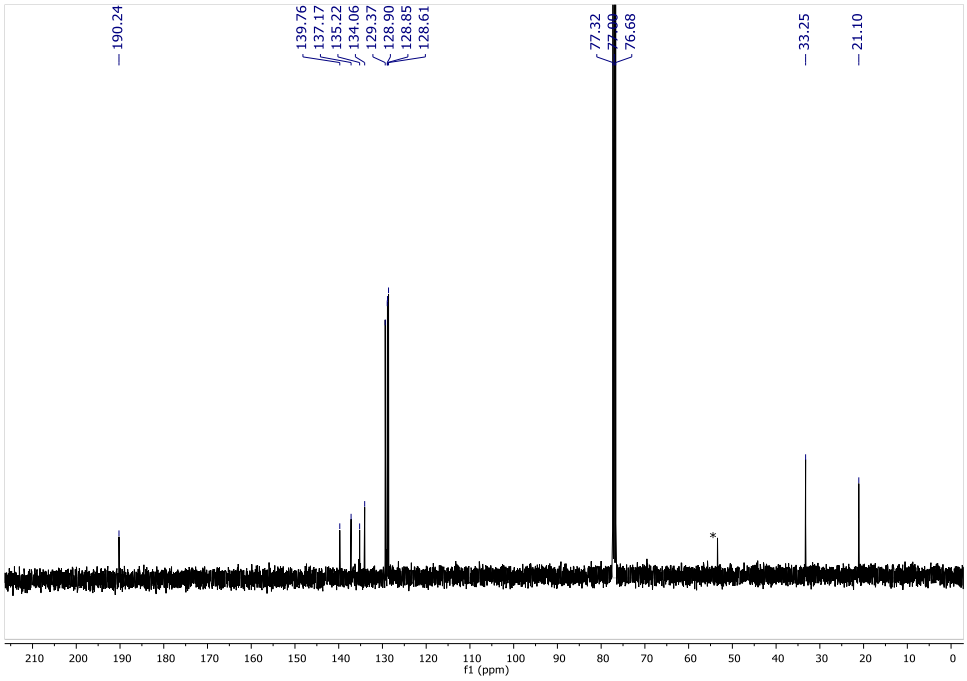
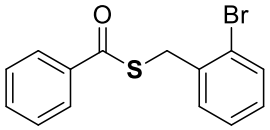


Figure S51 – ^{13}C{^{1}H}-NMR of **1e.** * Trace of CH_2Cl_2.

S-(2-Bromobenzyl) benzothioate 1f



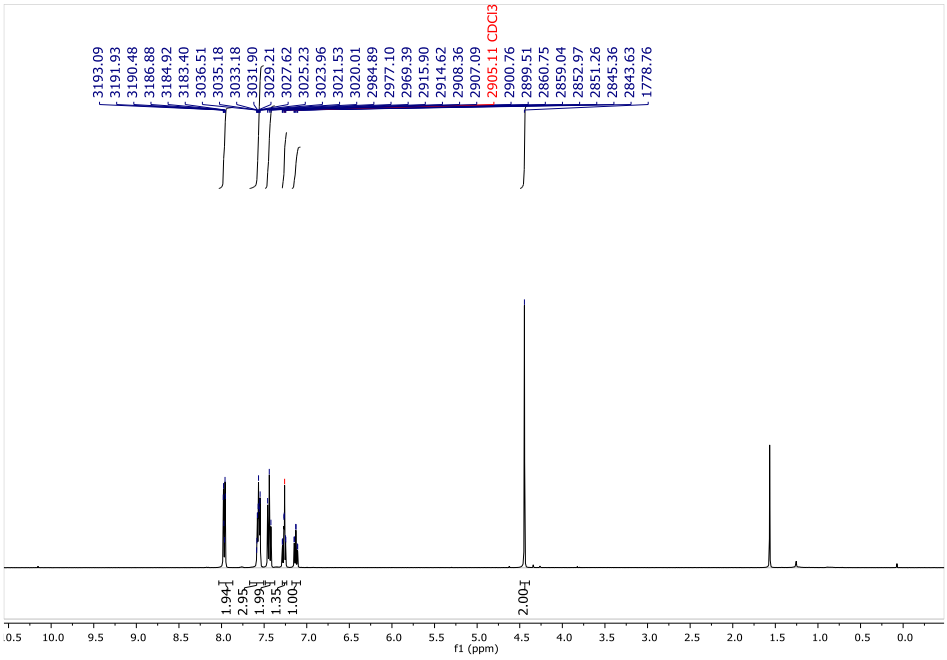


Figure S52 – ^{1}H-NMR of **1f**

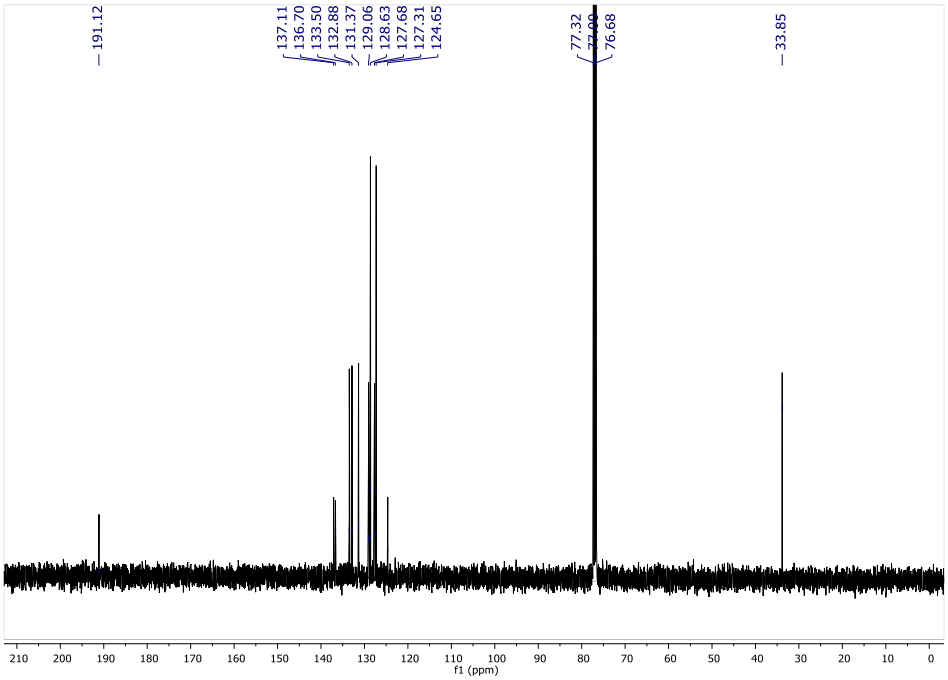
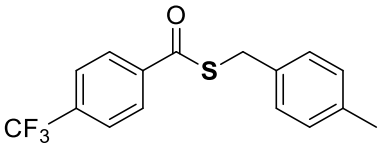


Figure S53 – ^{13}C{^{1}H}-NMR of **1f**

S-(4-Methylbenzyl) 4-(trifluoromethyl)benzothioate 1g



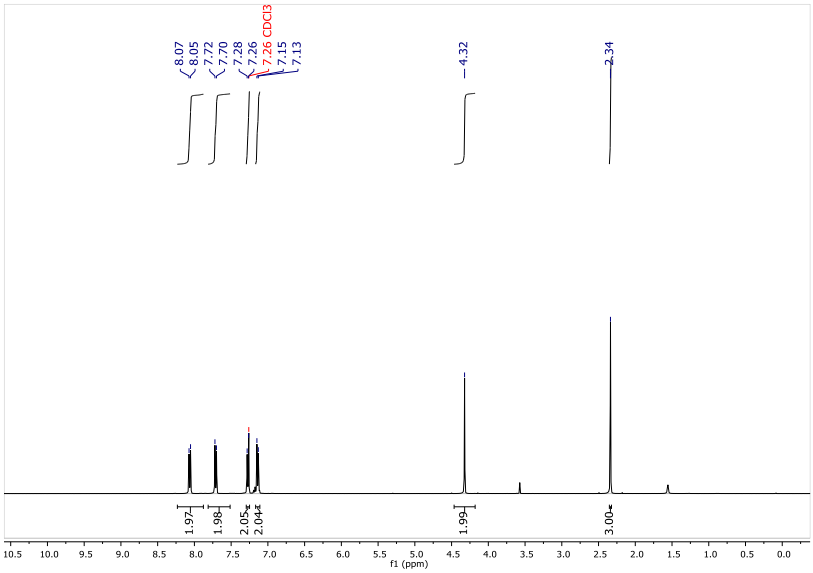


Figure S54 – ^{1}H-NMR of **1g**

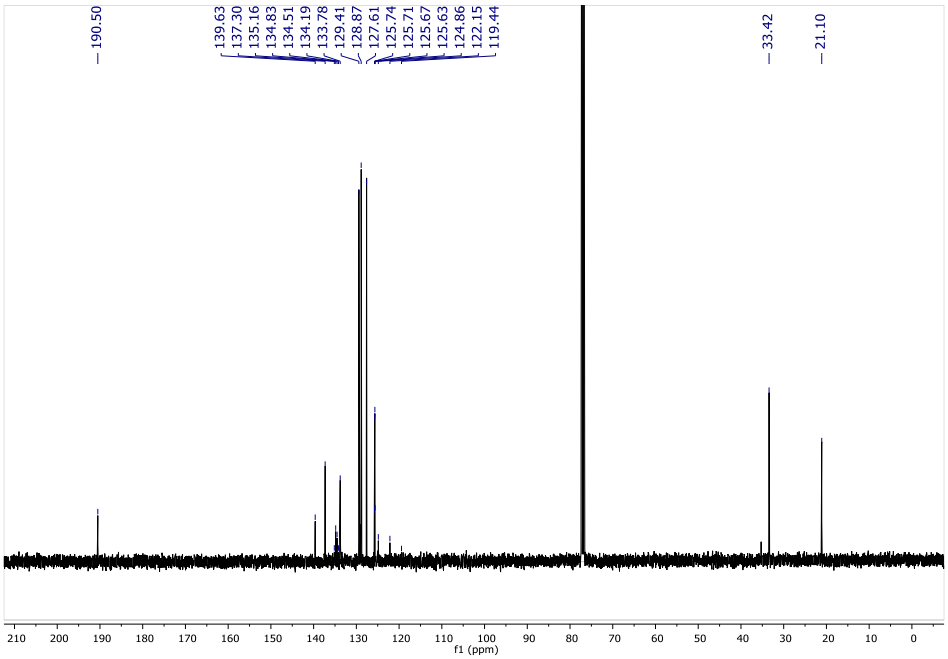


Figure S55 – ^{13}C{^{1}H}-NMR of **1g**

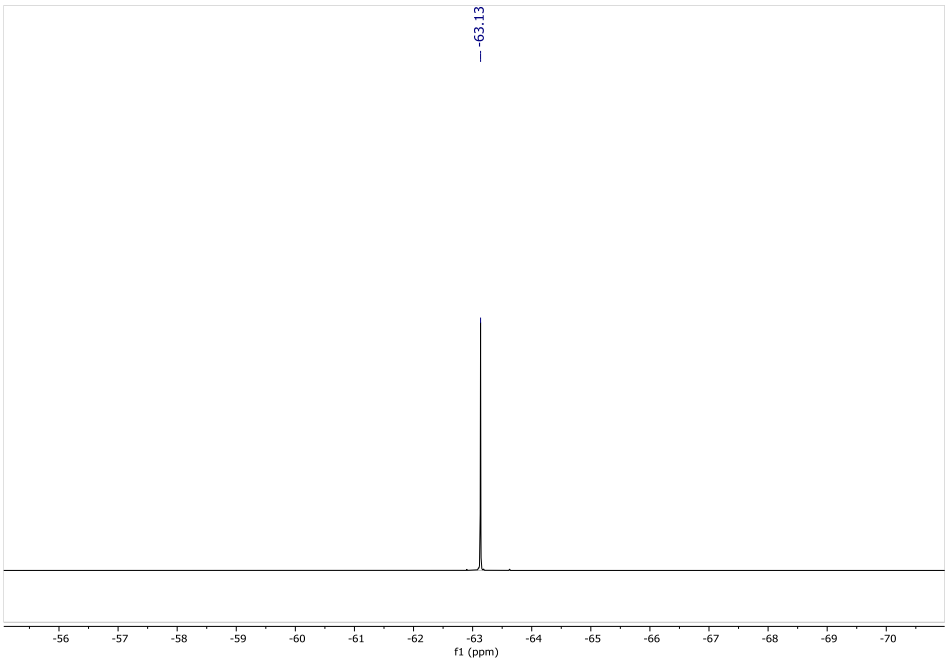
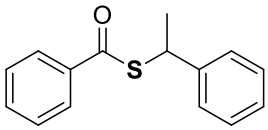


Figure S56 – ^{19}F-NMR of **1g**

S-(1-Phenylethyl) benzothioate 1h



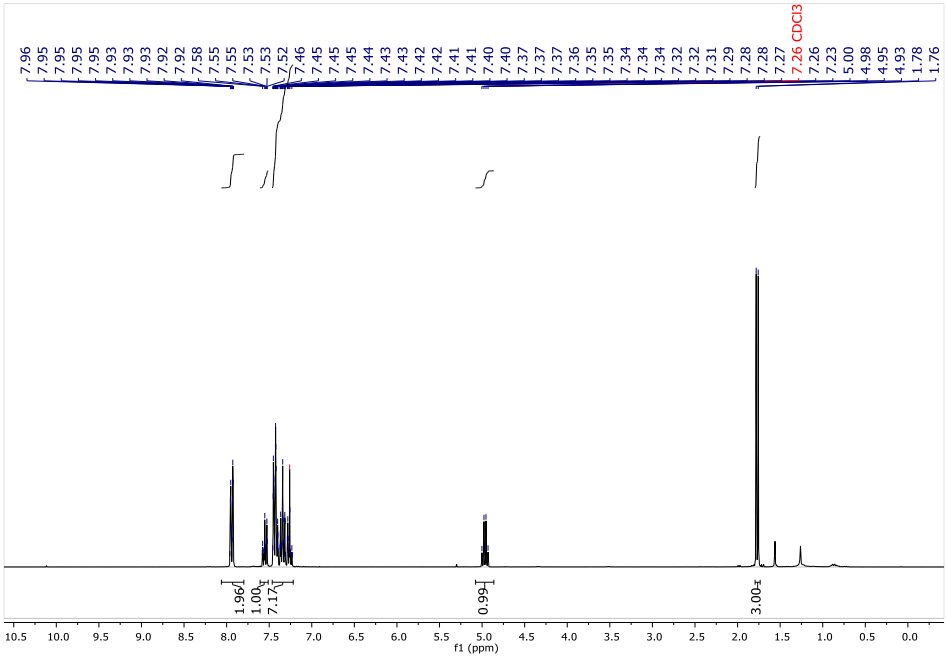


Figure S57 – ^{1}H-NMR of **1h**

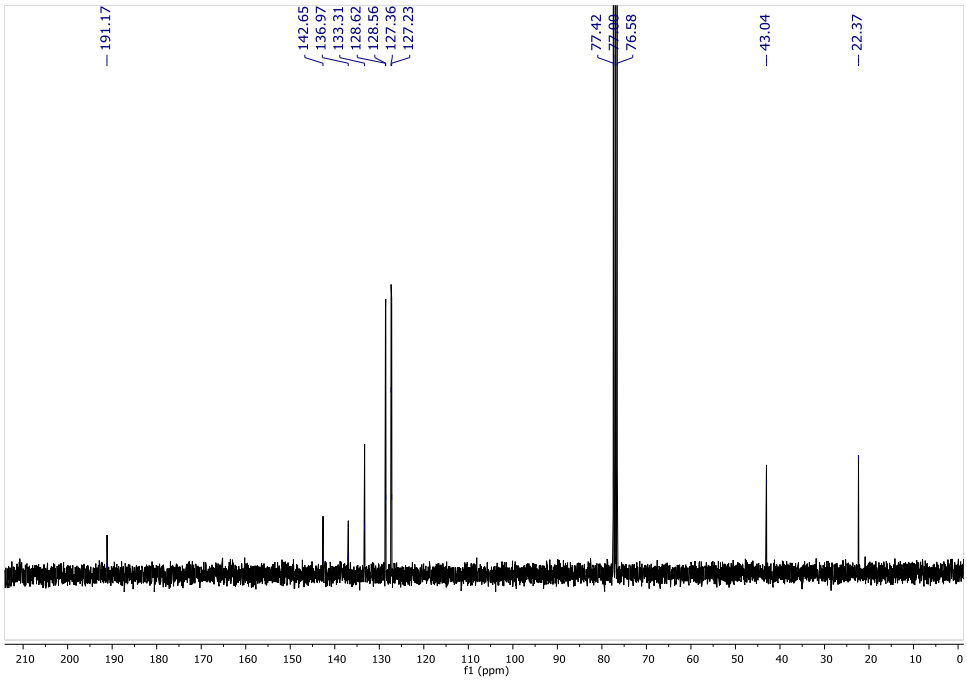
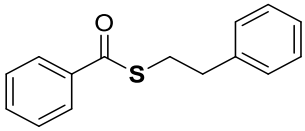


Figure S58 – $^{13}C\{^{1}H\}$-NMR of **1h**

S-Phenethyl benzothioate 1i



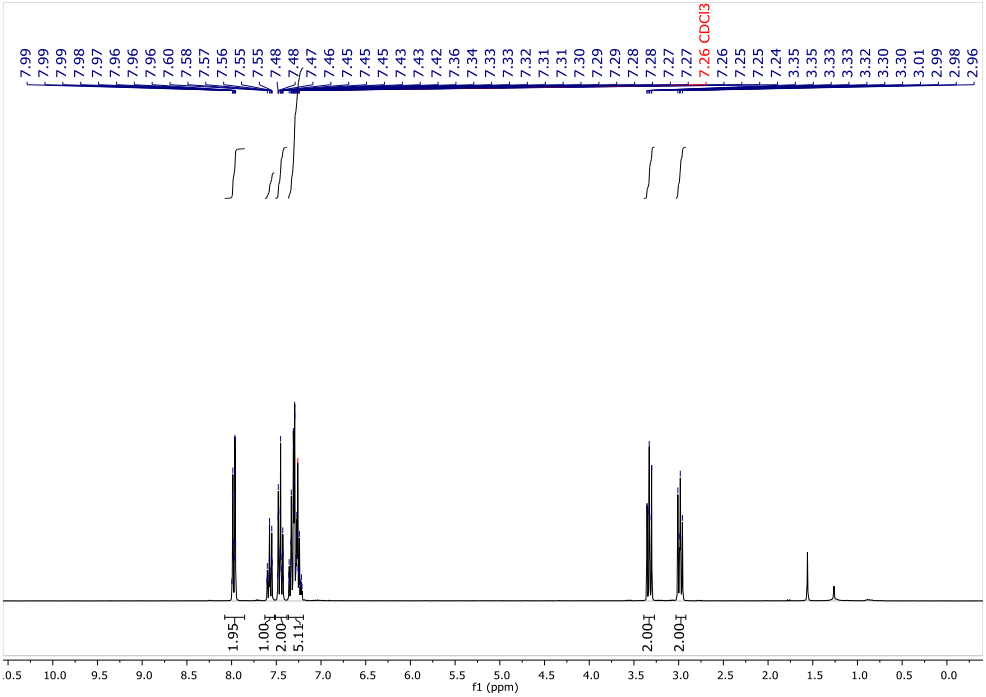


Figure S59 – ^{1}H-NMR of **1i**

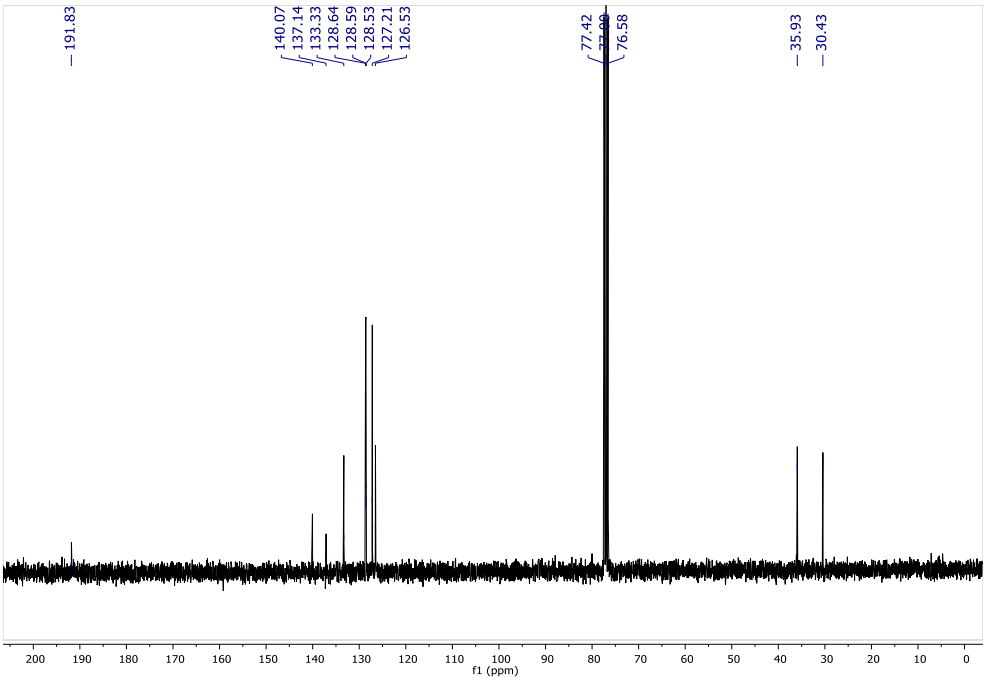
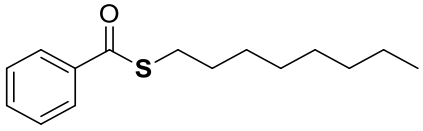


Figure S60 – $^{13}C\{^{1}H\}$-NMR of **1i**

S-Octyl benzothioate 1j



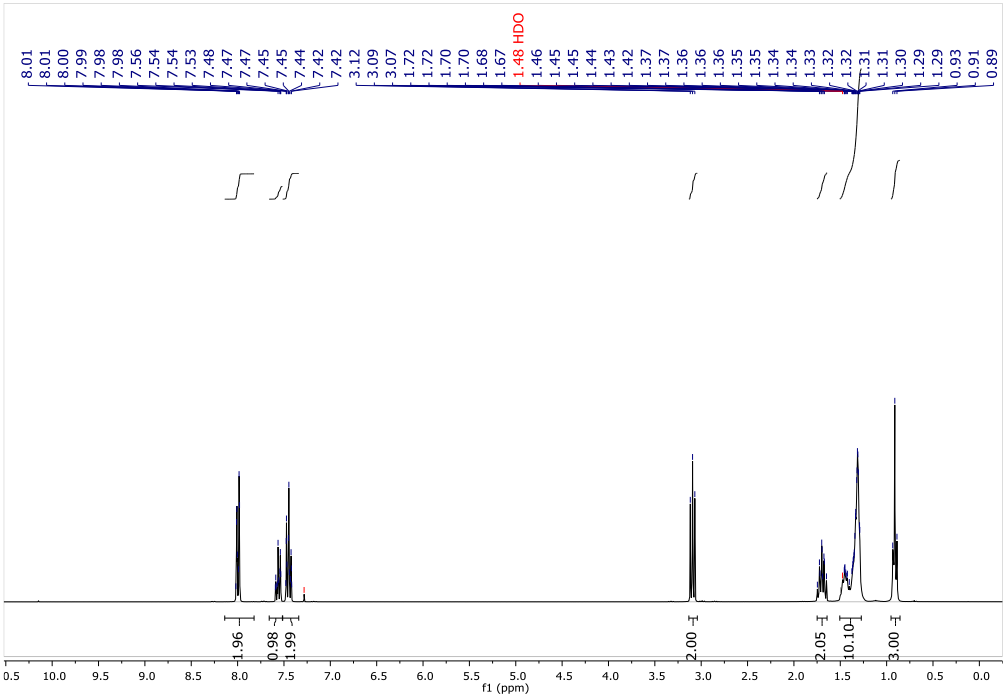


Figure S61 – ^{1}H-NMR of **1j**

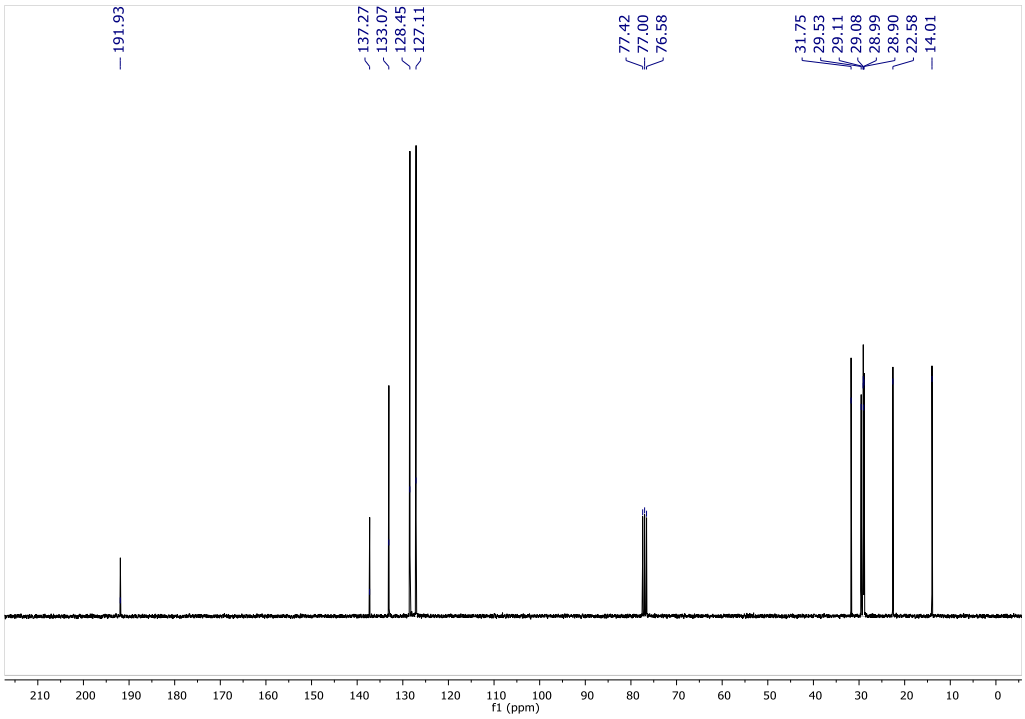
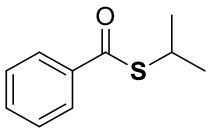


Figure S62 – $^{13}C\{^{1}H\}$-NMR of **1j**

S-Isopropyl benzothioate 1k



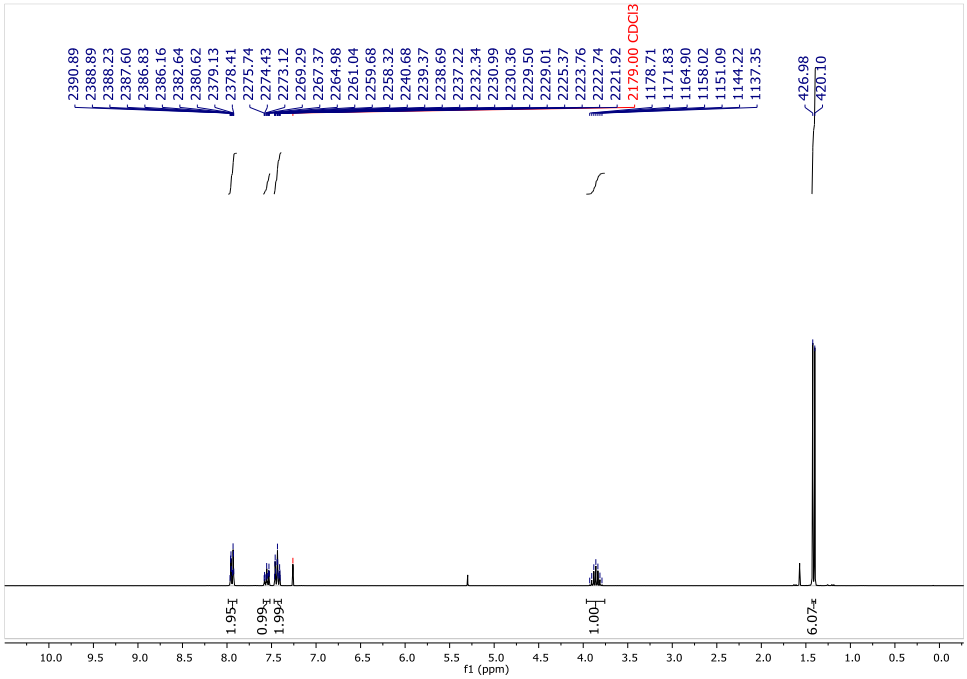


Figure S63 – ^{1}H-NMR of **1k**

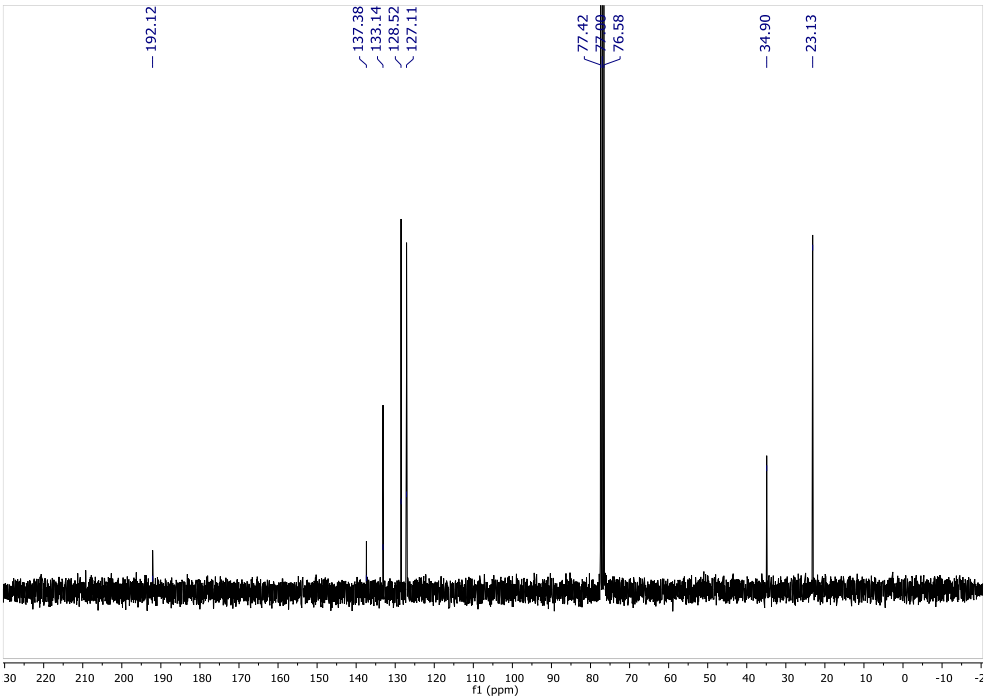
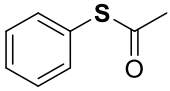


Figure S64 – $^{13}C\{^{1}H\}$-NMR of **1k**

S-Phenyl ethanethioate 1l



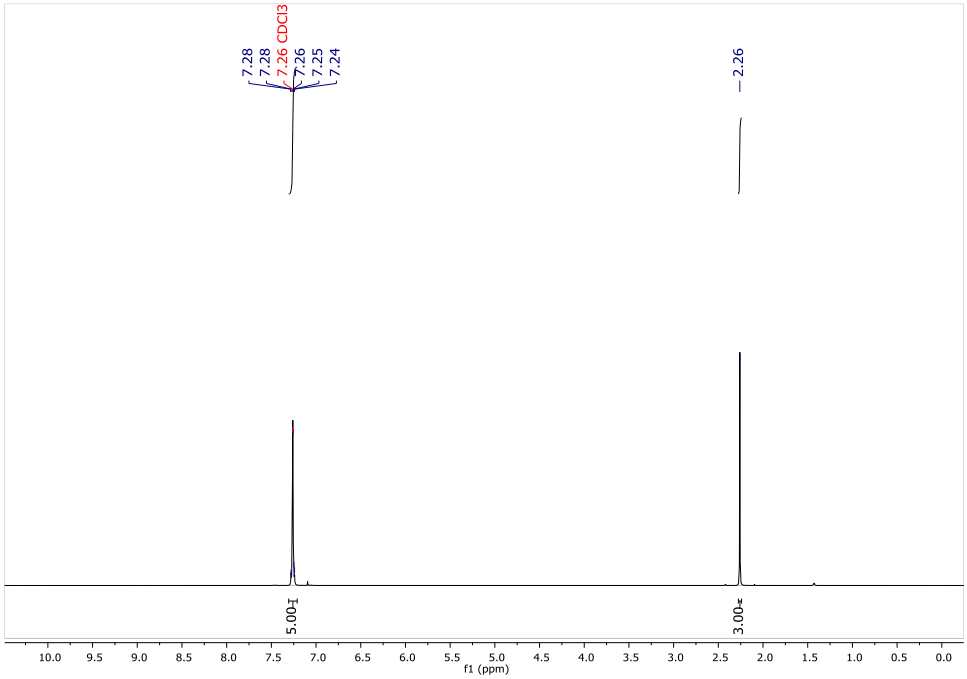


Figure S65 – ^{1}H-NMR of **1l**

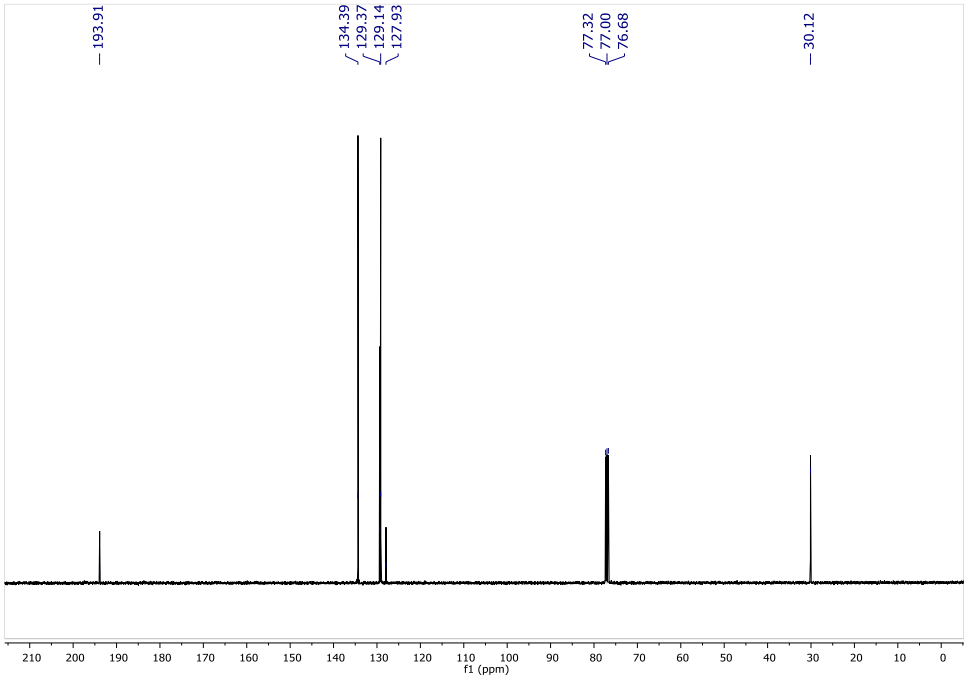
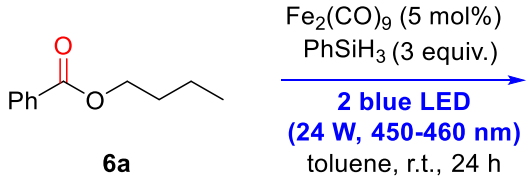


Figure S66 – ^{13}C{^{1}H}-NMR of **1l**

6. Experimental data for mechanism discussion

6.1. Radical scavenger action

Since iron is easily capable of single electron transfer (SET) reactions under blue light irradiation, the hydrosilylation reactions were conducted using radical-trapping agents such as TEMPO ((2,2,6,6-tetramethylpiperidin-1-yl)oxyl), diphenylethene or Galvinoxyl. Under the optimized conditions, the hydrosilylation reaction was not inhibited, which excluded any radical process (Scheme S1**).**



Additive	Yield
None,	99%
TEMPO (1 equiv.),	95%
$Ph_2C=CH_2$ (1 equiv.),	99%
Galvinoxyl (1 equiv.),	93%

Scheme S2. Influence of radical scavengers on the deoxygenation

6.2 Stoichiometric experiments

6.2.1.- Stoichiometric reaction between $Fe_2(CO)_9$ and phenylsilane

Stoichiometric reaction between $Fe_2(CO)_9$ and $PhSiH_3$ in deuterated toluene (C_7D_8) were performed at room temperature under blue light irradiation in order to detect the formation of Fe-H species. In Figure S67, in a classical glassware NMR tube. In Figure S68, in quartz NMR tube.

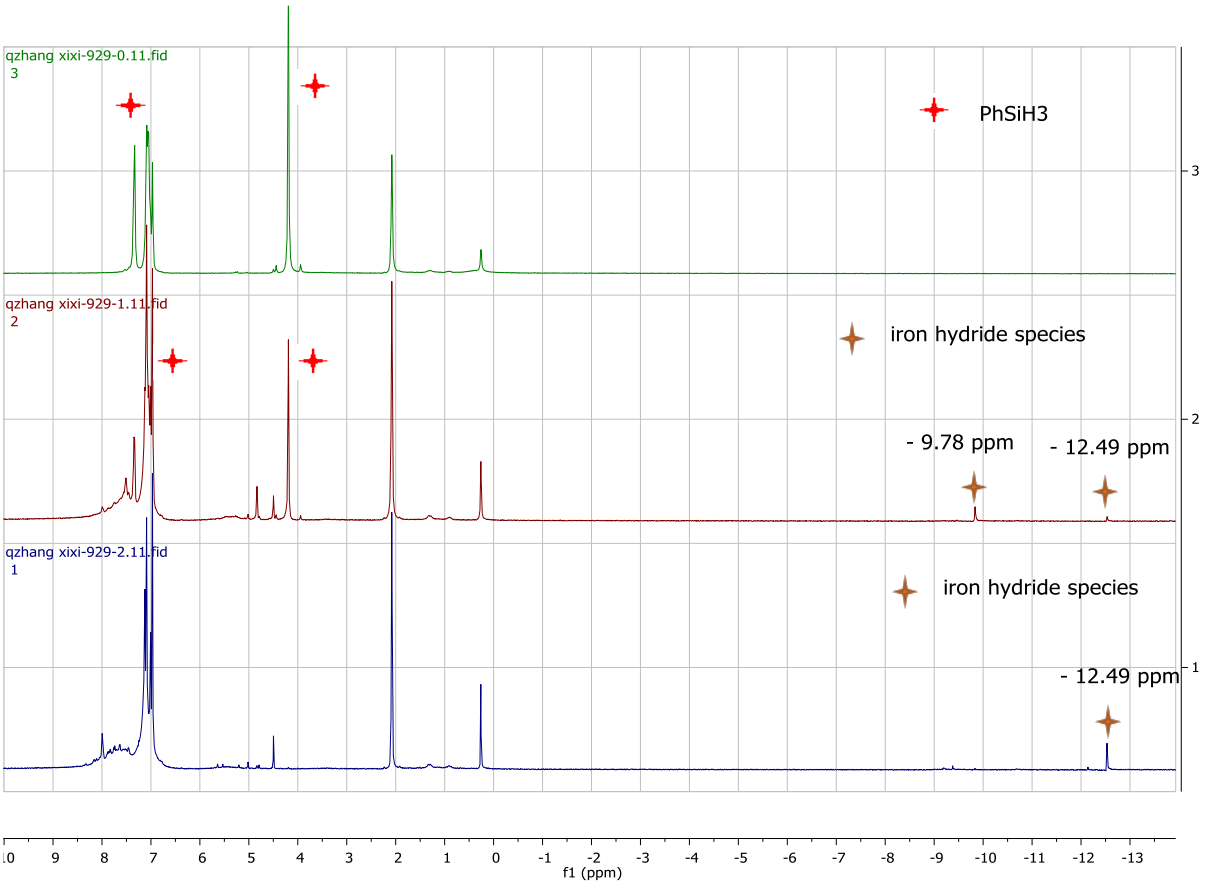


Figure 67. In a classical NMR tube, under argon atmosphere, 22.0 mg (0.03 mmol, 1 equiv.) of $Fe_2(CO)_9$ and 13 mg (0.06 mmol, 2 equiv.) of $PhSiH_3$ were dissolved in 0.5 mL of deuterated toluene (C_7D_8). The mixture was then irradiated for 6 h under blue light (2×24 W, 450-460 nm), (^{1}H NMR spectrum in Brown) and then 18 additional hours (^{1}H NMR spectrum in blue). For reference in green, ^{1}H NMR spectrum of phenylsilane.

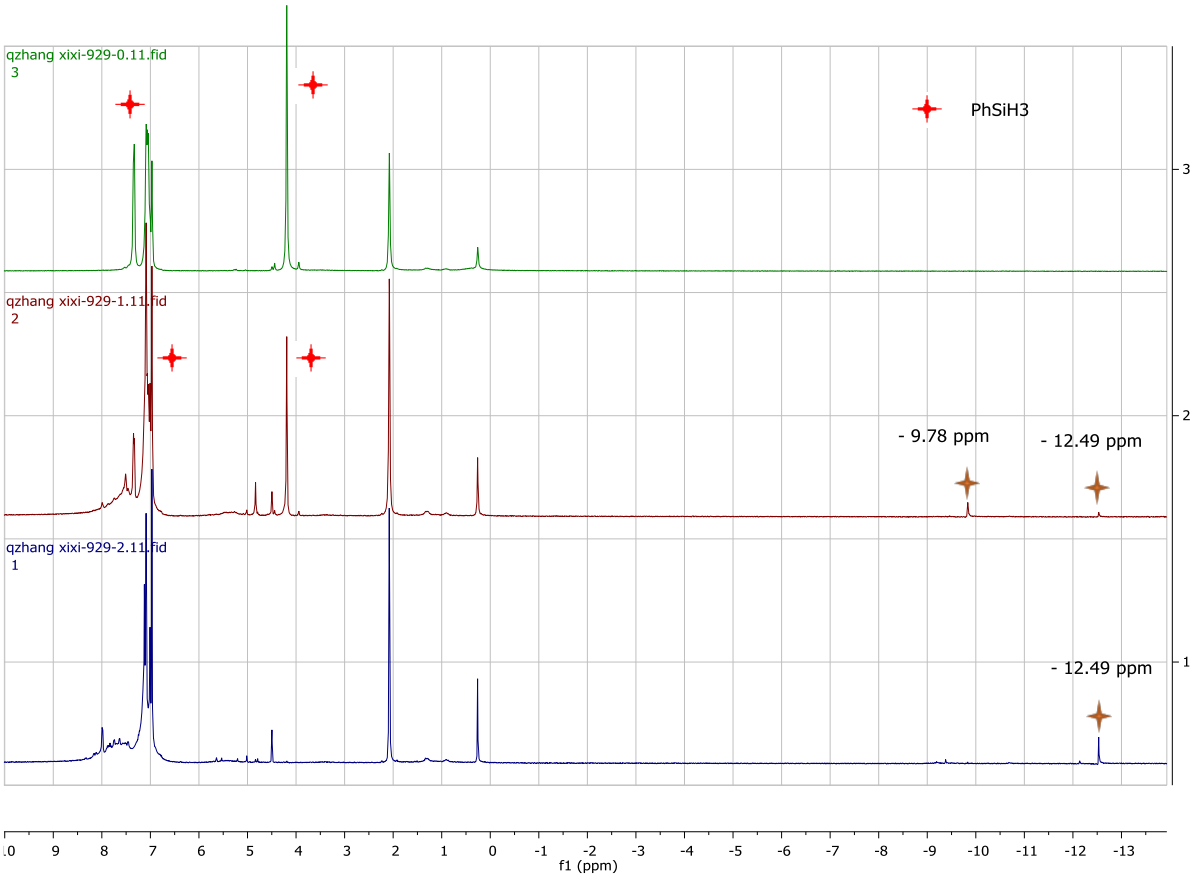


Figure S68. In a quartz tube, under argon atmosphere, 22.0 mg (0.03 mmol, 1 equiv.) of $Fe_2(CO)_9$ and 13 mg (0.06 mmol, 2 equiv. $PhSiH_3$ were dissolved in 0.5 mL of deuterated toluene (C_7D_8). The mixture was then irradiated for 6 h under blue light (2×24 W, 450-460 nm) (^{1}H NMR spectrum in Brown), and then 18 additional hours (^{1}H NMR spectrum in blue). For reference in green, ^{1}H NMR spectrum of phenylsilane.

6.2.2. Stoichiometric reaction between the in-situ generated species from $Fe_2(CO)_9$ and phenylsilane with thioester 1g

1- In a Young tube, under argon atmosphere, 11.0 mg (0.03 mmol, 1 equiv.) of $Fe_2(CO)_9$ and 6.5 mg (0.06 mmol, 2 equiv. $PhSiH_3$ were dissolved in 0.5 mL of deuterated toluene (C_7D_8). Figure S69 showed the ^{1}H-NMR of the mixture before light activation.

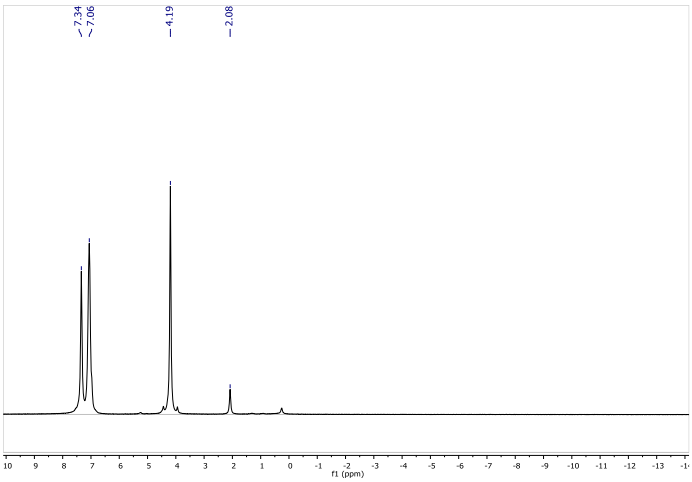


Figure S69 - ^{1}H-NMR of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ before light activation.

2. The resulting mixture was then irradiated for 12 h under blue light (2×24 W, 450-460 nm), and 9.3 mg (0.03 mmol, 1 equiv.) of S-(4-methylbenzyl) 4-(trifluoromethyl)benzothioate **1g** was added to the reaction mixture. The reaction was kept in dark for 16 h.
The ^{1}H (Figure S70) and ^{19}F-NMR (Figure S71) spectra showed that (*i*) iron hydride species were formed (signals at - 9.62 and -12.51 ppm) and that (*ii*) the starting thioester **1g** was not consumed.

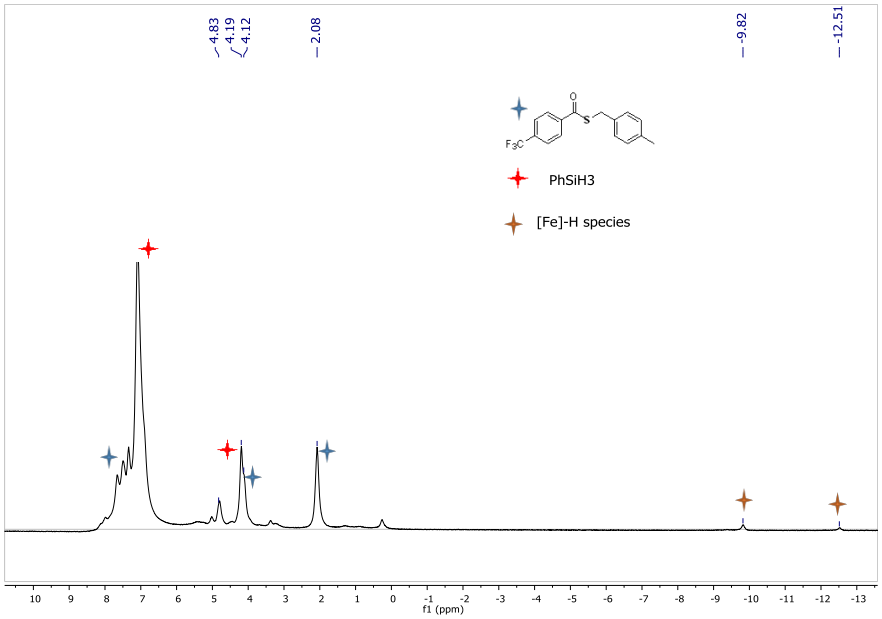


Figure S70 - ^{1}H-NMR of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ irradiated by blue light for 12 h to which thioester **1g** was added, and keep without irradiation for 16 h.

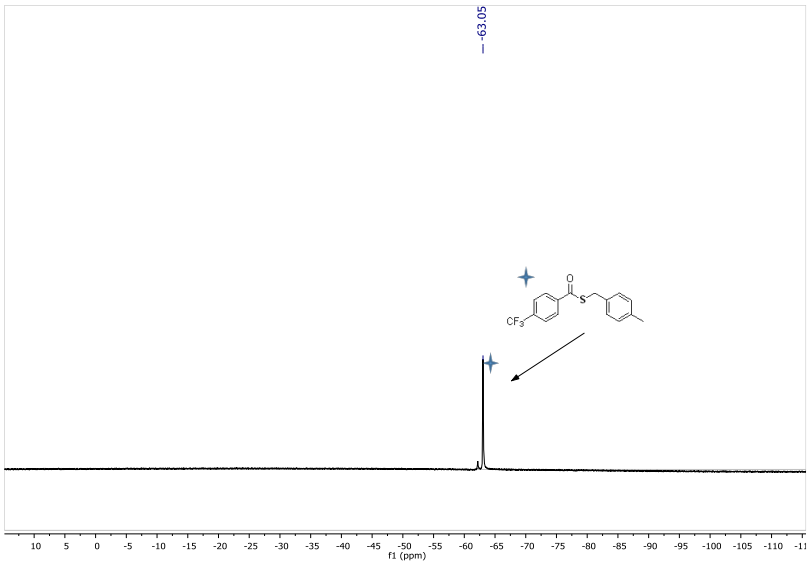


Figure S71 - ^{19}F-NMR of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ irradiated by blue light for 12 h to which thioester **1g** was added, and keep without irradiation for 16 h.

3. Then, the reaction mixture was irradiated under blue light (2×24 W, 450-460 nm) for 6 h. The ^{1}H and ^{19}F-NMR spectra showed that (*i*) an iron hydride species was formed (signal at -12.51 ppm) and that (*ii*) the starting thioamide **1g** was fully consumed and the thioester **2g** was formed. (Figures S72 and S73) Noticeably, an unidentified by-product was also produced (Signals at 2.11 or 2.13 ppm).

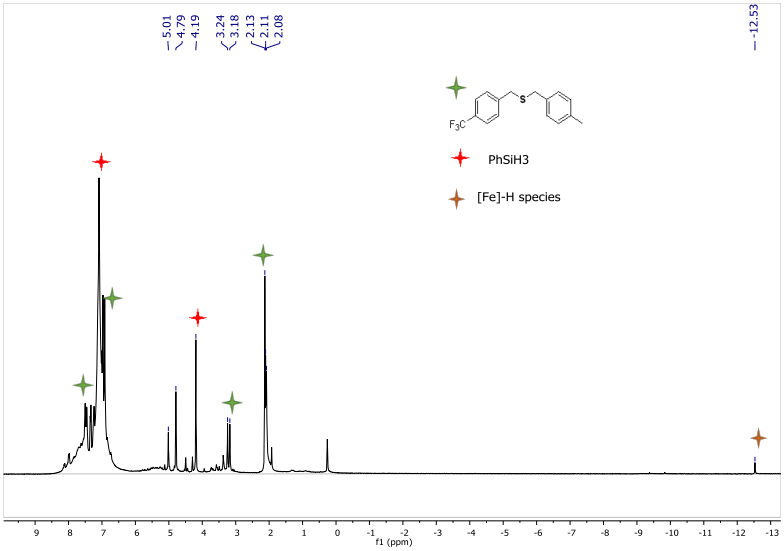


Figure **S72** - ^{1}H-NMR of the of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ irradiated by blue light for 12 h to which thioester **1g** was added, and the resulting mixture was irradiated by blue light for 6 h.

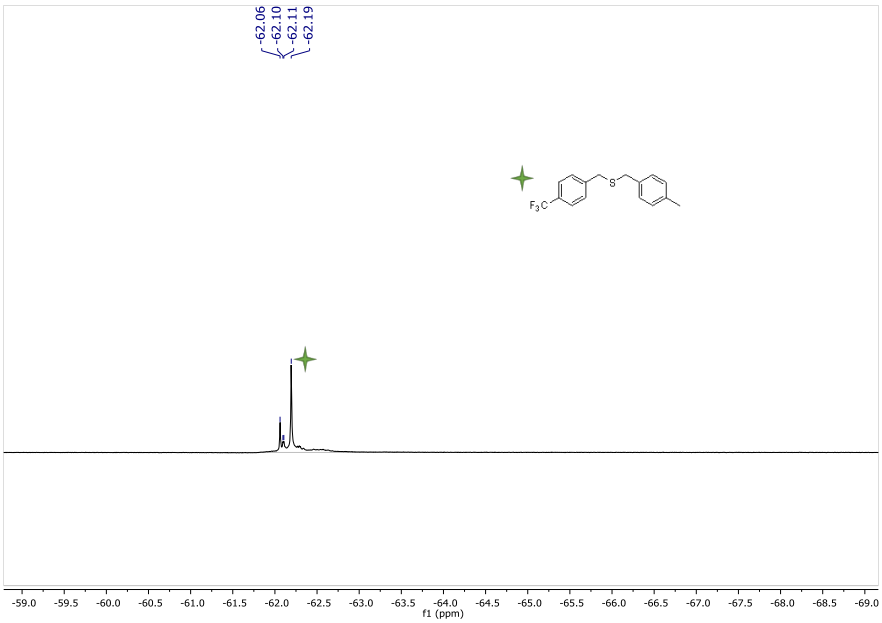


Figure **S73** – ^{19}F-NMR of the of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ irradiated by blue light for 12 h to which thioester **1g** was added, and the resulting mixture was irradiated by blue light for 6 h

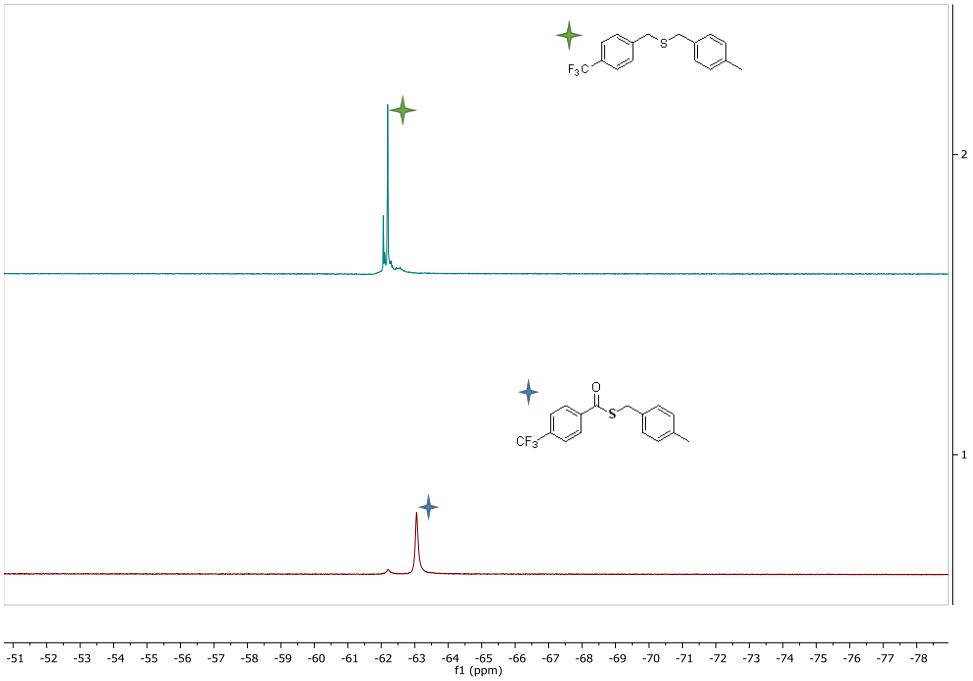


Figure S74 – ^{19}F-NMR of the of the mixture $Fe_2(CO)_9$ + $PhSiH_3$ irradiated by blue light for 12 h to which thioester **1g** was added, and the resulting mixture was irradiated by blue light for 6 h (Green spectrum). Brown spectrum, starting thioester **1g**, for reference.

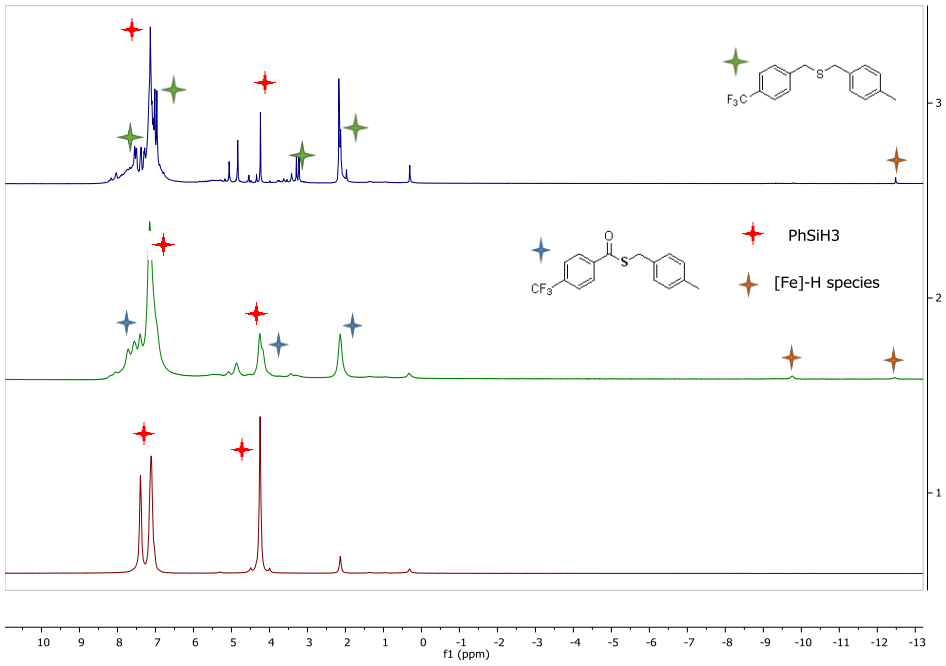


Figure S75 – Stacked ^{1}H NMR (figures S69, brown), S70 (green) and S72 (blue) for comparison.

6.2.3. Stoichiometric reaction between the in-situ generated Fe-H species from $Fe_2(CO)_9$ and phenylsilane with thioester 1g

To identify which iron hydride species (δ = –12.5 ppm or δ = –9.8 ppm) participated to the reduction of the thioester, an additional experiment was conducted. In a Young's tube under argon, $Fe_2(CO)_9$ (11.0 mg, 0.03 mmol, 1.0 equiv.) and $PhSiH_3$ (6.5 mg, 0.06 mmol, 2.0 equiv.) were dissolved in 0.5 mL of C_7D_8 and irradiated under blue light (2 × 24 W, 450–460 nm) for 24 h. After confirming that only the characteristic hydride resonance at –12.49 ppm remained, S-(4-methylbenzyl)-4-(trifluoromethyl)phenylthioester (**1g**, 9.3 mg, 0.03 mmol, 1.0 equiv.) was added. The reaction mixture was subsequently irradiated for 6, 12, and 72 h. ^{19}F NMR analysis showed no formation of thioether **2g**, thereby indicating that the hydride species corresponding to the signal at –9.78 ppm should correspond to the active species responsible for thioester reduction. (Figure S76)

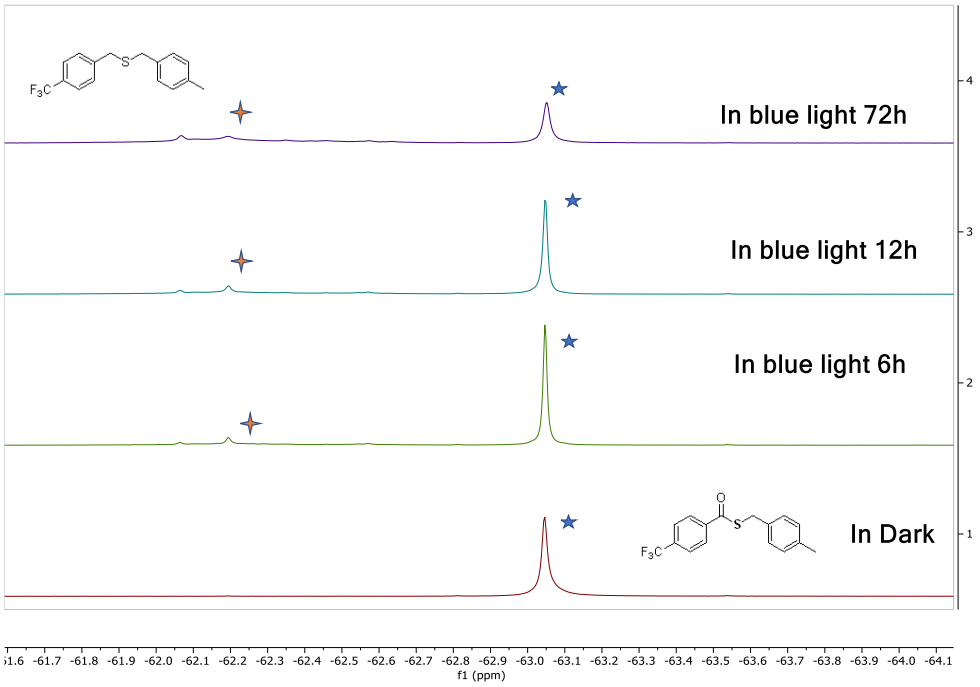


Figure S76. ^{19}F-NMR spectra of the of the mixture after 6, 12, 72h under blue light irradiation.

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