General

Chemicals and Solvents. Commercial chemicals were used as obtained from Sigma-Aldrich or Fisher. Solvents were used without further purification. DMSO was dried over molecular sieves (certified <0.005% water content, Sigma-Aldrich).

Analytical thin-layer chromatography. TLC was performed using aluminium plates with silica gel and fluorescent indicator (Merck, 60F254). Thin layer chromatography plates were visualized by exposure to UV light.

Column chromatography. Flash column chromatography with silica gel 60 Å (220-240 mesh) from *Acros*. Pentane or mixtures thereof with ethyl acetate were used as eluents. Product yields were determined as isolated by column chromatography.

Gas chromatography with mass-selective detector. *Agilent* 6890N Network GC-System, mass detector 5975 MS. Column: BPX5 (30m x 0.25 mm x 0.25, from *SGE*, carrier gas: H_2. Standard heating procedure: 50°C (2 min), 25°C/min -> 300°C (5 min).

Gas chromatography with FID. *Agilent* 7820A GC-Systems. Column: HP 5 19091J 413 (30 m x 0.32 mm x 0.25 μm) from *Agilent*, carrier gas: N_2. GC-FID was used for reaction optimization screening (Calibration with internal standard *n*-pentadecane or dodecanenitrile and analytically pure samples).

NMR. ^{1}H and ^{13}C nuclear magnetic resonance spectra were recorded on a *Bruker* Avance 300 (300 MHz ^{1}H; 75 MHz ^{13}C) and *Bruker* Avance 400 (400 MHz ^{1}H, 101 MHz ^{13}C) spectrometers. Chemicals shifts are reported in ppm (δ) relative to solvent residual peak as internal reference. Coupling constants (*J*) are reported in Hertz (Hz). Following abbreviations are used for spin multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet, dt = doublet of triplet, dq = doublet of quartet, ddt = doublet of doublet of triplet.

IR spectroscopy. Infrared spectra were recorded on a Cary 630 FTIR Spectrometer equipped with a ATR unit. Wavenumbers are indicated in cm^{-1}. Intensive absorption bands are indicated with „s“ (strong), medium bands with „m“ (medium), and weak bands with „w“ (weak).

High resolution mass spectrometry (HRMS). The spectra were recorded by the Central Analytics Lab at the Department of Chemistry, University of Regensburg, on a MAT SSQ 710 A from *Finnigan*.

Superscripts behind compound names are literature references.

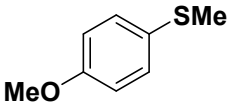
General procedure for the synthesis of arenediazonium salts

The parent aniline (10 mmol) was dissolved in glacial acetic acid (6 mL) and 32% aqueous tetrafluoroboric acid (1.6 mL) at room temperature. Then, a solution of *tert*-butyl nitrite (1.2 mL) in glacial acetic acid (2 mL) was slowly added at room temperature over 5 min. Diethylether (15 mL) was added, and the reaction mixture was cooled to -30 °C in order to induce crystallization of the ionic product. The crystals were filtered off, washed with cold diethylether (2 x 10 mL) and dried on air to give analytically pure arenediazonium tetrafluoroborates.

General procedure for base-induced thiolation, selenylation and telluration

A vial (5 mL) was charged with a magnetic stir bar, the arene diazonium salt (0.5 mmol), disulfide (0.5 mmol) and sodium acetate (0.5 mmol) and capped with a rubber septum. The vial was purged with N_2 (5 min). Dry DMSO (2.5 mL) was added. After 8 h of stirring water (5 mL) was added to give an emulsion, which was extracted with diethylether (3 x 5 mL). The organic phases were washed with brine (5 mL) and dried over MgSO4. The solvent was evaporated *in vacuo*, and the residue was purified by flash column chromatography (silica gel) using pentane/ethyl acetate mixtures (from 100/0 to 100/20) as eluent to obtain pure product.

4-Methoxyphenylmethylsulfane



$C_8H_{10}OS$, 154.23 g/mol

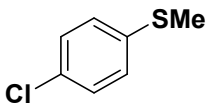
Yield 68.5 mg, 0.44 mmol, 89% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.27 (d, *J* = 8.9 Hz, 2H), 6.85 (d, *J* = 8.9 Hz, 2H), 3.79 (s, 3H), 2.44 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 158.1, 130.1, 128.7, 114.5, 55.3, 18.0.

LR MS (EI, 70 eV, m/z): 154 [M^+]

4-Chlorophenylmethylsulfane



C_7H_7ClS, 158.64 g/mol

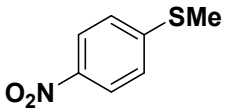
Yield 55.3 mg, 0.35 mmol, 70% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.25 (d, *J* = 8.7 Hz, 2H), 7.17 (d, *J* = 8.7 Hz, 2H), 2.47 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 136.9, 130.8, 128.9, 127.8, 16.0.

LR MS (EI, 70 eV, m/z): 158 [M^+]

4-Nitrophenylmethylsulfane



$C_7H_7NO_2S$, 169.20 g/mol

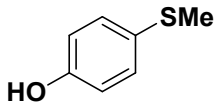
Yield 67.7 mg, 0.40 mmol, 85% (isolated)

^{1}H-NMR (400 MHz, $CDCl_3$): δ_H [ppm] = 8.15 (d, *J* = 9.0 Hz, 2H), 7.29 (d, *J* = 9.0 Hz, 2H), 2.55 (s, 3H).

^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 148.8, 144.8, 125.0, 123.9, 14.8.

LR MS (EI, 70 eV, m/z): 169 [M^+]

4-Methylthiophenol



C_7H_8OS, 140.20 g/mol

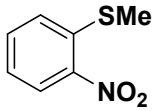
Yield 61.0 mg, 0.44 mmol, 87% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.19 (d, *J* = 8.7 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 2.42 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 154.8, 130.5, 128.0, 116.1, 18.2.

LR MS (EI, 70 eV, m/z): 140 [M^+]

2-Nitrophenylmethylsulfane



$C_7H_7NO_2S$, 169.20 g/mol

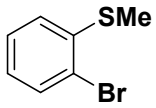
Yield 40.8 mg, 0.24 mmol, 80% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.26 (dd, *J* = 8.3 Hz, *J* = 1.2 Hz, 1H), 7.59 (ddd, *J* = 8.6 Hz, *J* = 7.3 Hz, *J* = 1.3 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.26 (ddd, *J* = 8.2 Hz, *J* = 7.2 Hz, *J* = 1.2 Hz, 1H), 2.50 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 145.4, 139.3, 133.7, 126.2, 125.6, 124.1, 15.9.

LR MS (EI, 70 eV, m/z): 169 [M^+]

2-Bromophenylmethylsulfane



C_7H_7BrS, 203.10 g/mol

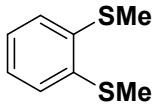
Yield 40.1 mg, 0.20 mmol, 66% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.52 (dd, *J* = 7.9 Hz, *J* = 1.4 Hz, 1H), 7.30 (ddd, *J* = 7.9 Hz, *J* = 7.4 Hz, *J* = 1.3 Hz, 1H), 7.13 (dd, *J* = 8.0 Hz, *J* = 1.5 Hz, 1H), 7.0 (ddd, *J* = 7.8 Hz, *J* = 7.6 Hz, *J* = 1.6 Hz, 1H), 2.47 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 139.6, 132.6, 127.8, 125.6, 125.3, 121.6, 15.7.

LR MS (EI, 70 eV, m/z): 203 [M^+]

1,2-*Bis*(methylthio)benzene



$C_8H_{10}S_2$, 170.29 g/mol

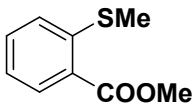
Yield 37.2 mg, 0.22 mmol, 73% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.24-7.13 (m, 4H), 2.48 (s, 6H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 137.3 (s), 126.6 (s), 125.8 (s), 16.2 (s).

LR MS (EI, 70 eV, m/z): 170 [M^+]

Methyl-2-(methylthio)benzoate



$C_9H_{10}O_2S$, 182.24 g/mol

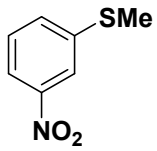
Yield 64.7 mg, 0.36 mmol, 71% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.98 (dd, *J* = 7.9 Hz, *J* = 1.5 Hz, 1H), 7.30 (ddd, *J* = 8.2 Hz, *J* = 7.3 Hz, *J* = 1.5 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 7.14 (ddd, *J* = 8.0 Hz, *J* = 7.8 Hz, *J* = 1.1 Hz, 1H), 3.90 (s, 3H), 2.44 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 166.7, 143.2, 132.4, 131.2, 126.6, 124.2, 123.3, 52.0, 15.5.

LR MS (EI, 70 eV, m/z): 182 [M^+]

3-nitrophenylmethylsulfane



$C_7H_7NO_2S$, 169.20 g/mol

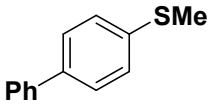
Yield 36.2 mg, 0.36 mmol, 77% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.04 (t, *J* = 2.0 Hz, 1H), 7.95 (ddd, *J* = 8.1 Hz, *J* = 2.2 Hz, *J* = 1.1 Hz, 1H), 7.52 (ddd, *J* = 7.9 Hz, *J* = 1.8 Hz, *J* = 1.1 Hz, 1H), 7.43 (t, *J* = 8.0 Hz, 1H), 2.55 (s, 3H).

^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 141.6, 131.9, 129.4, 120.2, 119.7, 119.0, 15.4.

LR MS (EI, 70 eV, m/z): 169 [M^+]

[1,1'-Biphenyl]-4-yl(methyl)sulfane



$C_{13}H_{12}S$, 200.30 g/mol

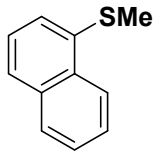
Yield 35.6 mg, 0.18 mmol, 58% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.60-7.50 (m, 4H), 7.47-7.40 (m, 2H), 7.37-7.30 (m, 3H), 2.53 (s, 3H).

^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 140.5, 138.0, 137.5, 128.8, 127.5, 127.2, 126.9, 126.8, 15.9.

LR MS (EI, 70 eV, m/z): 200 [M^+]

Naphthalen-1-ylmethylsulfane



$C_{11}H_{10}S$, 174.26 g/mol

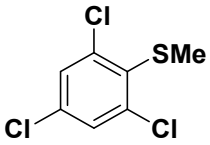
Yield 46.2 mg, 0.27 mmol, 53% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.30 (d, *J* = 7.2 Hz, 1H), 7.85 (d, *J* = 7.4 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.57-7.51 (m, 2H), 7.57-7.37 (m, 2H), 2.59 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 135.7, 133.5, 131.5, 128.5, 126.2, 126.1, 125.7, 125.6, 124.2, 123.5, 16.1.

LR MS (EI, 70 eV, m/z): 174 [M^+]

2,4,6-Trichlorophenylmethylsulfane



$C_7H_5Cl_3S$, 227.53 g/mol

Yield 78.3 mg, 0.34 mmol, 55% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.40 (s, 2H), 2.42 (s, 3H).

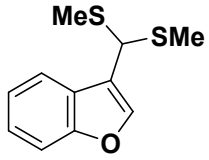
^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 141.4 (s), 134.9 (s), 133.3 (s), 128.5 (s), 18.3 (s).

LR MS (EI, 70 eV, m/z): 226 [M^+]

HR MS (m/z): found: 225.91721 [M^+] (calculated: 225.91721)

FT-IR: 794(s), 828(s), 857(s), 1118(m), 1178(m), 1364(s), 1532(s), 1562(m), 2922(w), 3068(w).

3-(*Bis*(methylthio)methyl)benzofuran



$C_{11}H_{12}OS_2$, 224.34 g/mol

Yield 62.6 mg, 0.28 mmol, 58% (isolated)

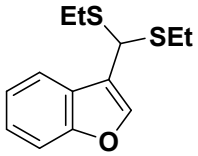
^{1}H-NMR (400 MHz, $CDCl_3$): δ_H [ppm] = 7.79 (d, *J* = 7.4 Hz, 1H), 7.67 (d, *J* = 0.6 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.35-7.24 (m, 2H), 5.05 (d, *J* = 0.9 Hz, 1H), 2.14 (s, 6H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 155.6, 142.8, 125.9, 124.8, 122.6, 120.7, 119.0, 111.6, 47.2, 14.2.

LR MS (EI, 70 eV, m/z): 224 [M^+]

HR MS (m/z): found: 224.03292 [M^+] (calculated: 224.03241)

3-(*Bis*(ethylthio)methyl)benzofuran



$C_{13}H_{16}OS_2$, 252,39 g/mol

Yield 54.3 mg, 0.22 mmol, 56% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.85-7.81 (m, 1H), 7.69 (d, *J* = 0.5 Hz, 1H), 7.50-7.45 (m, 1H), 7.35-7.23 (m, 2H), 5.18 (d, *J* = 0.7 Hz, 1H), 2.85-2.45 (m, 4H), 1.26 (t, *J* = 7.4 Hz, 6H).

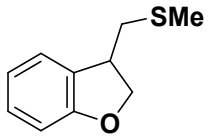
^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 155.7 (s), 142.7 (s), 125.9 (s), 124.7 (s), 122.6 (s), 121.0 (s), 119.8 (s), 111.6 (s), 43.4 (s), 25.7 (s), 14.2 (s).

LR MS (EI, 70 eV, m/z): 252 [M^+]

HR MS (m/z): found: 252.06298 [M^+] (calculated: 252.06371)

FT-IR: 742(s), 857(m), 1178(m), 1264(m), 1450(s), 1573(w), 2870(w), 2926(w), 2967(w).

3-((methylthio)methyl)-2,3-dihydrobenzofuran



$C_{10}H_{12}OS$, 180,06 g/mol

Yield 70.2 mg, 0.39 mmol, 20% (isolated)

^{1}H-NMR (400 MHz, $CDCl_3$): δ_H [ppm] = 7.26 (d, *J* = 7.4 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 6.88 (t, *J* = 7.1 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 4.66 (t, *J* = 9.0 Hz, 1H), 4.42 (dd, *J* = 9.1 Hz, *J* = 5.7 Hz, 1H), 3.66 (tt, *J* = 9.0 Hz, *J* = 4.8 Hz, 1H), 2.78 (q, *J* = 7.3 Hz, 1H), 2.77 (q, *J* = 7.3 Hz, 1H), 2.15 (s, 3H).

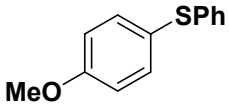
^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 160.0 (s), 129.3 (s), 128.7 (s), 124.4 (s), 120.4 (s), 109.7 (s), 76.1 (s), 41.4 (s), 39.2 (s), 15.8 (s).

LR MS (EI, 70 eV, m/z): 180 [M^+]

HR MS (m/z): found: 180.06031 [M^+] (calculated: 180.06034)

FT-IR: 746(s), 958(m), 1230(s), 1480(s), 1595(m), 2915(w).

(4-Methoxyphenyl)(phenyl)sulfane



$C_{13}H_{12}OS$, 216.30 g/mol

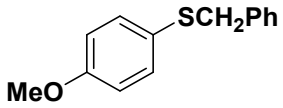
Yield 32.8 mg, 0.15 mmol, 51% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.43 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 7.30-7.10 (m, 5H), 3.83 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 159.8 (s), 138.6 (s), 135.4 (s), 128.9 (s), 128.1 (s), 125.7 (s), 124.2 (s), 114.9 (s), 55.3 (s).

LR MS (EI, 70 eV, m/z): 216 [M^+]

Benzyl(4-methoxyphenyl)sulfane



$C_{14}H_{14}OS$, 230.33 g/mol

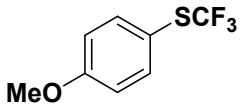
Yield 37.4 mg, 0.16 mmol, 54% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.28-7.16 (m, 2H), 6.83-6.76 (m, 2H), 3.99 (s, 2H), 3.78 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 159.2, 138.1, 134.0, 128.8, 128.3, 126.9, 126.0, 114.4, 55.3, 41.2.

LR MS (EI, 70 eV, m/z): 230 [M^+]

(4-Methoxyphenyl)(trifluoromethyl)sulfane



$C_8H_7F_3OS$, 208.20 g/mol

Yield 84.2 mg, 0.40 mmol, 81% (isolated)

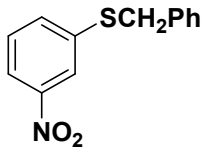
^{1}H-NMR (400 MHz, $CDCl_3$): δ_H [ppm] = 7.28-7.16 (m, 2H), 6.83-6.76 (m, 2H), 3.99 (s, 2H), 3.78 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 161.9, 138.3, 134.8, 129.6 (q, J = 308.1 Hz), 115.0, 55.4.

^{19}F-NMR (282 MHz, $CDCl_3$): δ_F [ppm] = -44.43.

LR MS (EI, 70 eV, m/z): 208 [M^+]

Benzyl(3-nitrophenyl)sulfane



$C_{13}H_{11}NO_2S$, 245.30 g/mol

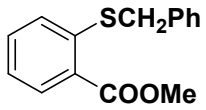
Yield 19.2 mg, 0.08 mmol, 39% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.13 (t, *J* = 2.0 Hz, 1H), 7.99 (ddd, *J* = 8.2 Hz, *J* = 2.2 Hz, *J* = 1.1 Hz, 1H), 7.55 (ddd, *J* = 7.9 Hz, *J* = 1.7 Hz, *J* = 1.0 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.36-7.26 (m, 5H), 4.21 (s, 2H).

^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 148.4, 139.4, 136.0, 134.6, 129.4, 128.8, 128.7, 127.6, 123.2, 120.8, 38.3.

LR MS (EI, 70 eV, m/z): 245 [M^+]

Methyl 2-(benzylthio)benzoate



$C_{15}H_{14}O_2S$, 258.34 g/mol

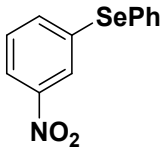
Yield 11.5 mg, 0.04 mmol, 22% (isolated)

^{1}H-NMR (400 MHz, $CDCl_3$): δ_H [ppm] = 7.96 (dd, *J* = 7.8 Hz, *J* = 1.4 Hz, 1H), 7.44-7.26 (m, 7H), 7.16 (td, *J* = 7.5 Hz, *J* = 1.1 Hz, 1H), 4.17 (s, 2H), 3.90 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 166.9, 141.9, 136.1, 132.4, 131.2, 129.1, 128.6, 127.5, 127.3, 126.0, 124.1, 52.1, 37.3.

LR MS (EI, 70 eV, m/z): 258 [M^+]

(3-Nitrophenyl)(phenyl)selane



$C_{12}H_9NO_2Se$, 278.17 g/mol

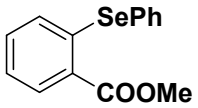
Yield 51.6 mg, 0.19 mmol, 55% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.20 (t, *J* = 1.9 Hz, 1H), 8.05 (ddd, *J* = 8.2 Hz, *J* = 2.2 Hz, *J* = 1.0 Hz, 1H), 7.64 (ddd, *J* = 7.7 Hz, *J* = 1.6 Hz, *J* = 1.1 Hz, 1H), 7.61-7.56 (m, 2H), 7.42-7.35 (m, 4H).

^{13}C-NMR (101 MHz, $CDCl_3$): δ_C [ppm] = 148.5, 136.9, 134.7, 129.9, 129.8, 128.8, 128.3, 125.7, 121.5.

LR MS (EI, 70 eV, m/z): 278 [M^+]

Methyl 2-(phenylselanyl)benzoate



$C_{14}H_{12}O_2Se$, 291.21 g/mol

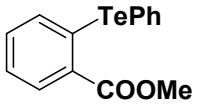
Yield 71.7 mg, 0.25 mmol, 82% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.05 (dd, *J* = 7.0 Hz, *J* = 2.2 Hz, 1H), 7.74-7.68 (m, 2H), 7.50-7.38 (m, 3H), 7.23-7.12 (m, 2H), 6.93-6.89 (m, 1H), 3.97 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 167.2, 140.4, 137.5, 132.6, 131.2, 129.7, 129.1, 128.9, 128.8, 126.9, 124.7, 52.3.

LR MS (EI, 70 eV, m/z): 292 [M^+]

Methyl 2-(phenyltellanyl)benzoate



$C_{14}H_{12}O_2Te$, 339.85 g/mol

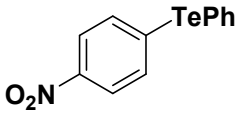
Yield 99.3 mg, 0.29 mmol, 97% (isolated)

^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 8.13-8.08 (m, 1H), 7.98 (dd, *J* = 8.0 Hz, *J* = 1.3 Hz, 2H), 7.47 (tt, *J* = 7.4 Hz, *J* = 2.0 Hz, 1H), 7.40-7.33 (m, 2H), 7.24-7.10 (m, 3H), 3.99 (s, 3H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 168.4, 141.7, 133.3, 132.7, 131.4, 129.7, 129.2, 128.9, 127.1, 125.6, 117.7, 52.7.

LR MS (EI, 70 eV, m/z): 342 [M^+]

(4-Nitrophenyl)(phenyl)tellane



$C_{12}H_9NO_2Te$, 326.81 g/mol

Yield 40.5 mg, 0.12 mmol, 41% (isolated)

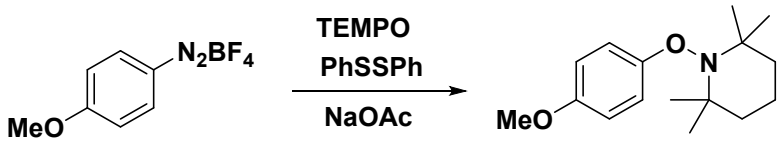
^{1}H-NMR (300 MHz, $CDCl_3$): δ_H [ppm] = 7.99-7.94 (m, 2H), 7.89-7.84 (m, 2H), 7.58 (dt, *J* = 8.9 Hz, *J* = 2.1 Hz, 2H), 7.45 (tt, *J* = 7.4 Hz, *J* = 1.6 Hz, 1H), 7.37-7.30 (m, 2H).

^{13}C-NMR (75 MHz, $CDCl_3$): δ_C [ppm] = 147.1, 140.3, 135.2, 130.1, 129.3, 127.9, 123.7, 112.6.

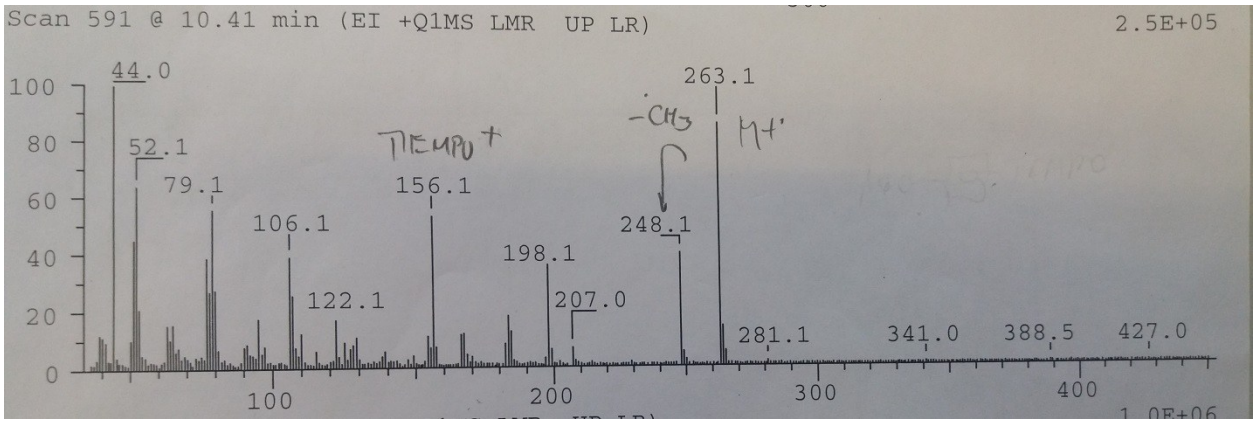
LR MS (EI, 70 eV, m/z): 329 [M^+]

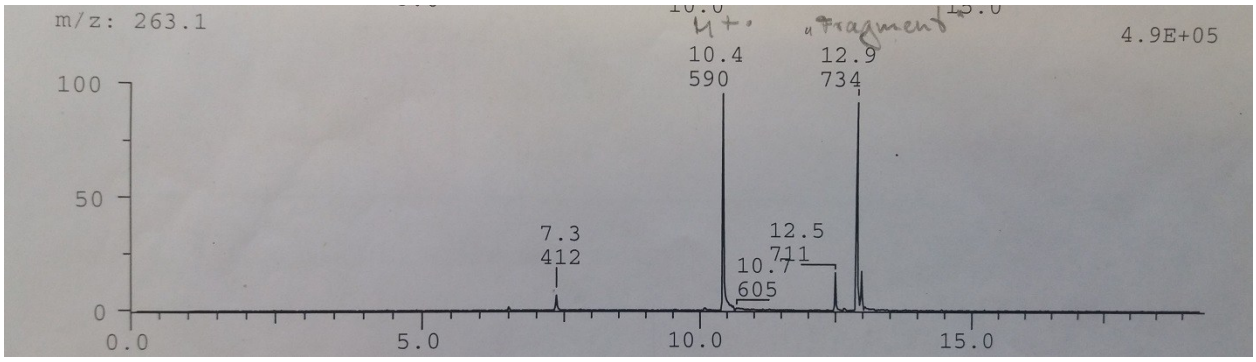
Radical trapping experiment

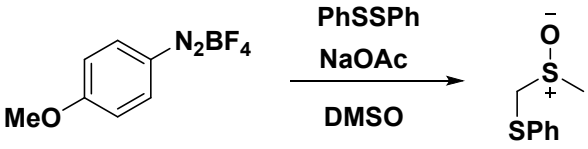
See standard procedure above, but with addition of TEMPO (1 equiv., 0.5 mmol) after 30 min.

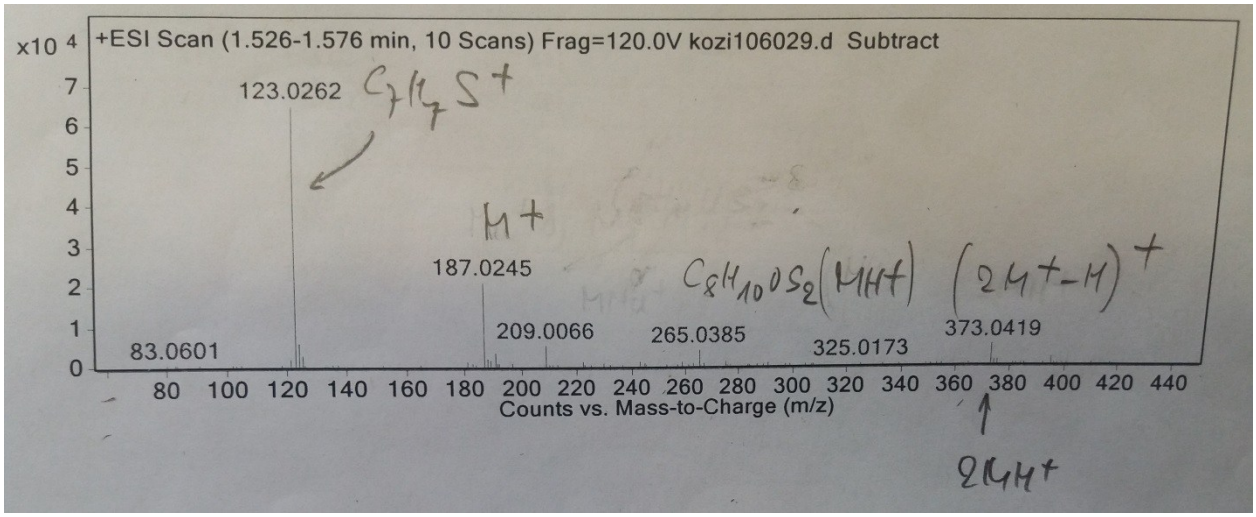


Calculated mass: 263.18







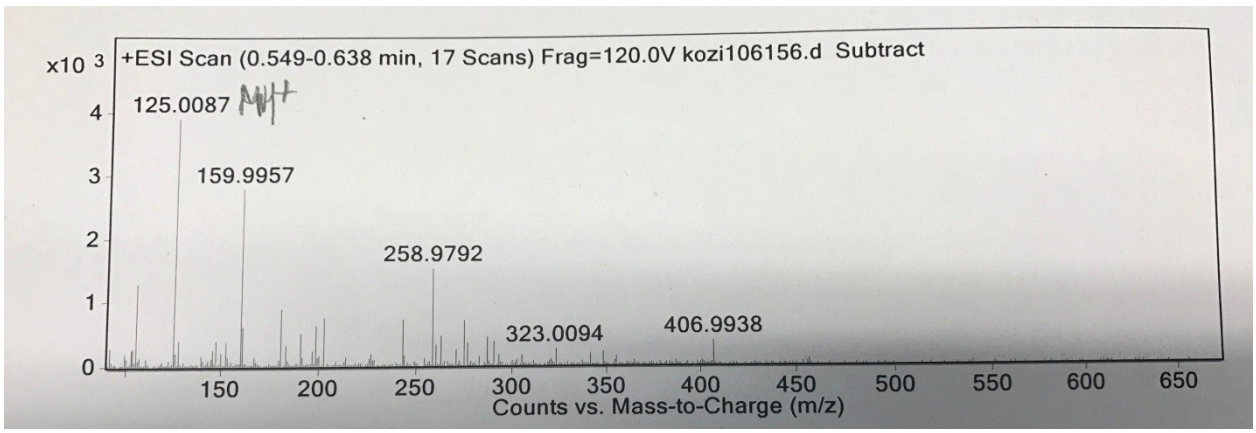


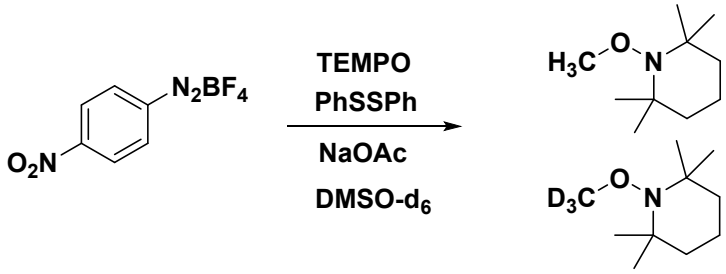
Spectrum Identification Results: + Scan (1.526-1.576 min) Sub (kozi106029.d)

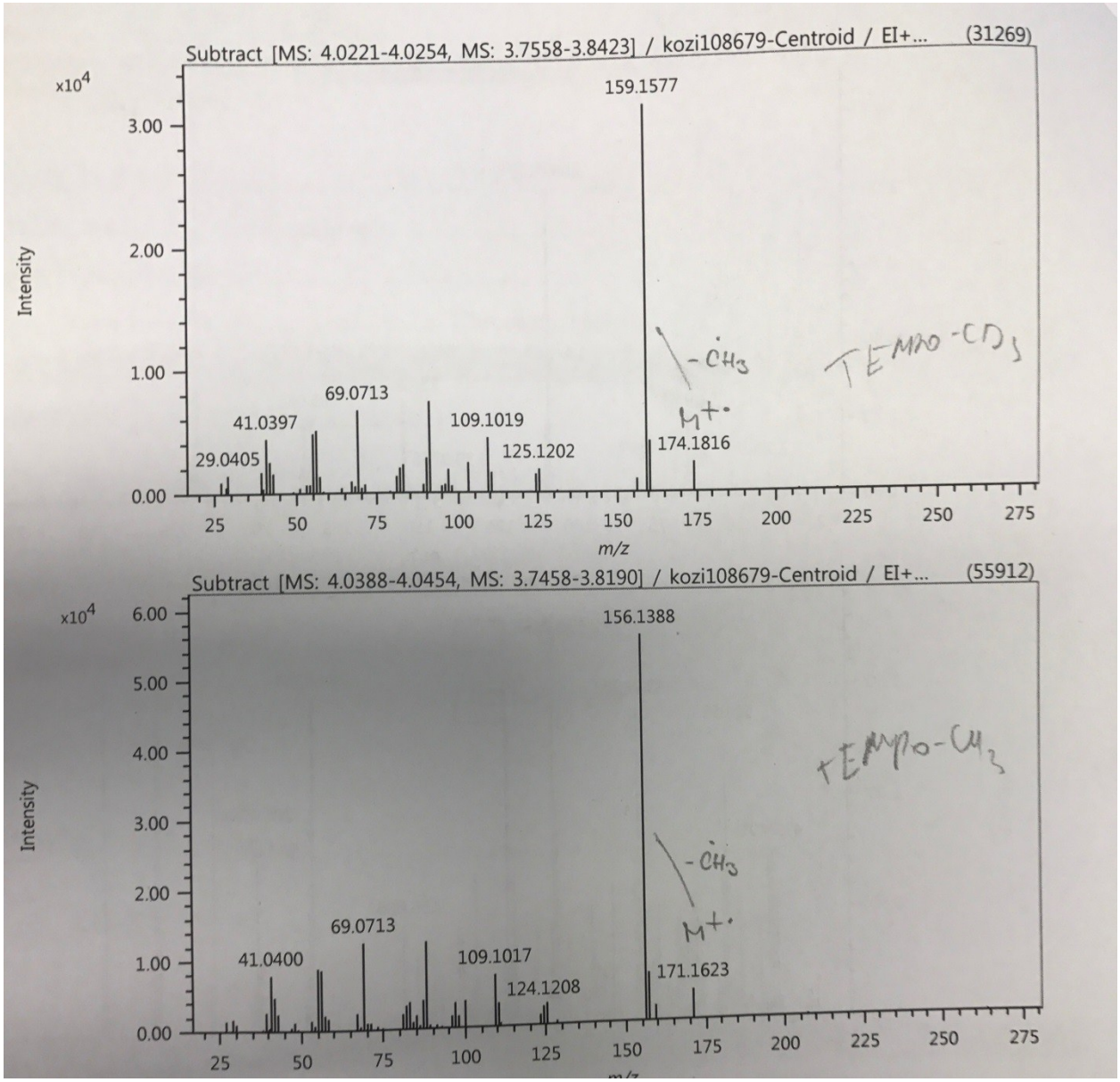
Best	Formula	Score	Mass	Mass (MFG)	Diff (ppm)	Diff (abs. ppm)	Diff (mDa)	ID Source	Score (MFG)	DBE
✓	C8 H11 O S2	96.18	187.0252	187.0251	-0.49	0.49	-0.09	MFG	96.18	3.5

Species	Ion Formula	m/z	Height	Score (MFG)	Score (MS)	Score (MFG, MS/MS)	Score (mass)	Score (iso. abund)	Score (iso. spacing)
M*+	C8 H11 O S2	187.0246	21004.3	96.18	96.18		99.91	93.26	92.24

m/z	m/z (Calc)	Diff (ppm)	Diff (mDa)	Height	Height (Calc)	Height %	Height % (Calc)	Height Sum %	Height Sum % (Calc)
187.0245	187.0246	0.34	0.1	21768	21004.3	100	100	85.7	82.7
188.0278	188.0274	-2.28	-0.4	1877.4	2183.7	8.6	10.4	7.4	8.6
189.0227	189.0211	-8.96	-1.7	1625.9	2025.1	7.5	9.6	6.4	8
190.025	190.0238	-6.6	-1.3	129.6	187.9	0.6	0.9	0.5	0.7

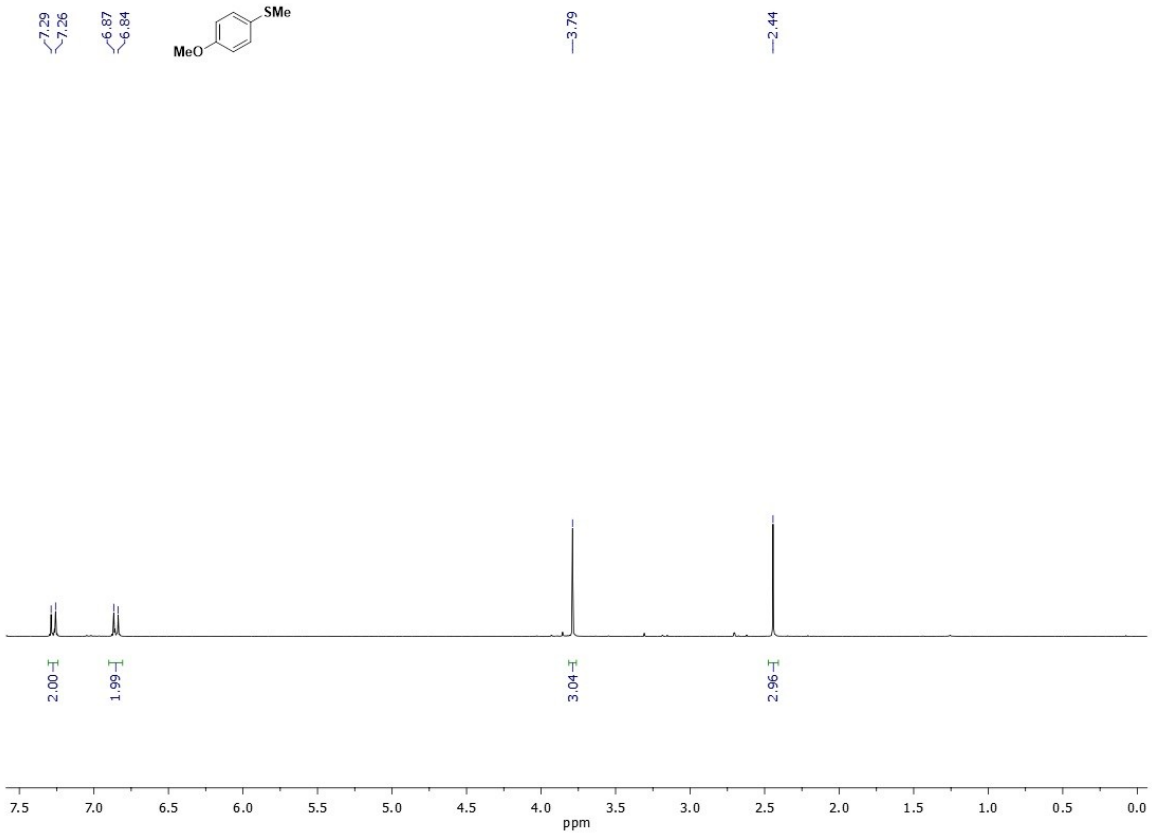



N2BF4
O2N
TEMPO
PhSSPh
NaOAc
DMSO-d6
H3C
O
N
D3C
O
N

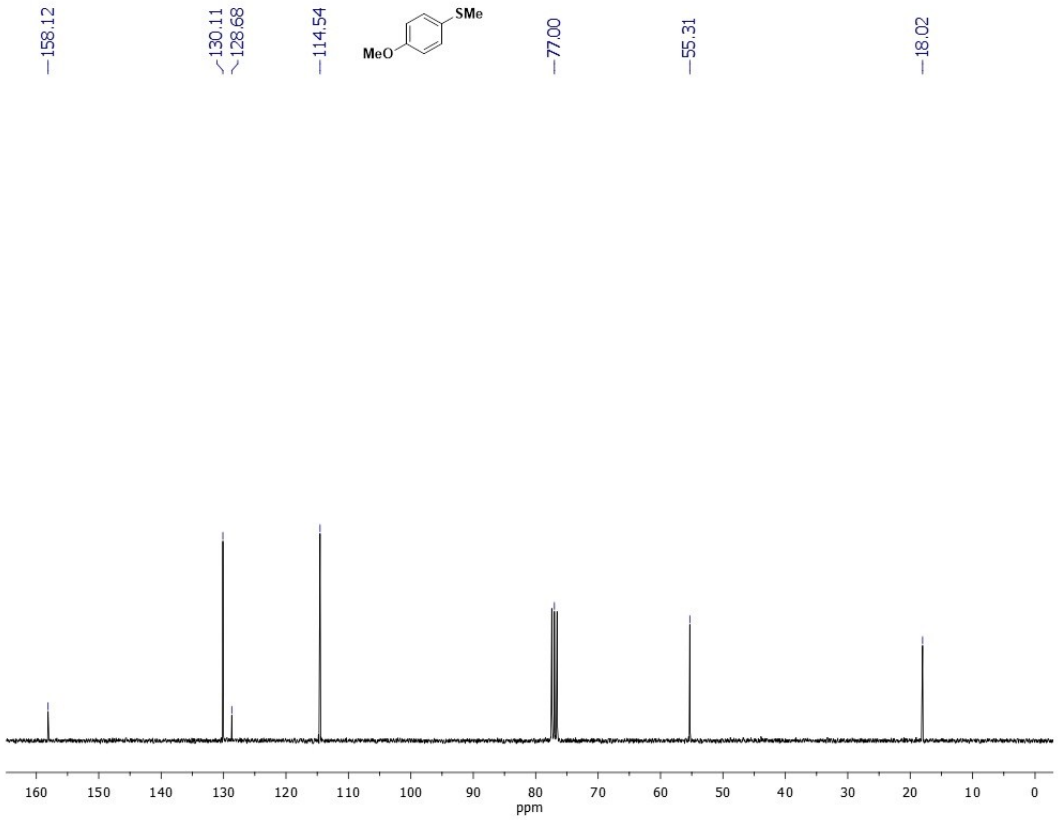

Subtract [MS: 4.0221-4.0254, MS: 3.7558-3.8423] / kozi108679-Centroid / EI+... (31269)
x10^4
Intensity
159.1577
69.0713
41.0397
109.1019
125.1202
29.0405
174.1816
-·CH3
M+·
TEMPO-CD3
m/z
Subtract [MS: 4.0388-4.0454, MS: 3.7458-3.8190] / kozi108679-Centroid / EI+... (55912)
x10^4
Intensity
156.1388
69.0713
41.0400
109.1017
124.1208
171.1623
-·CH3
M+·
TEMPO-CH3
m/z

Spectroscopic Compound Characterizations

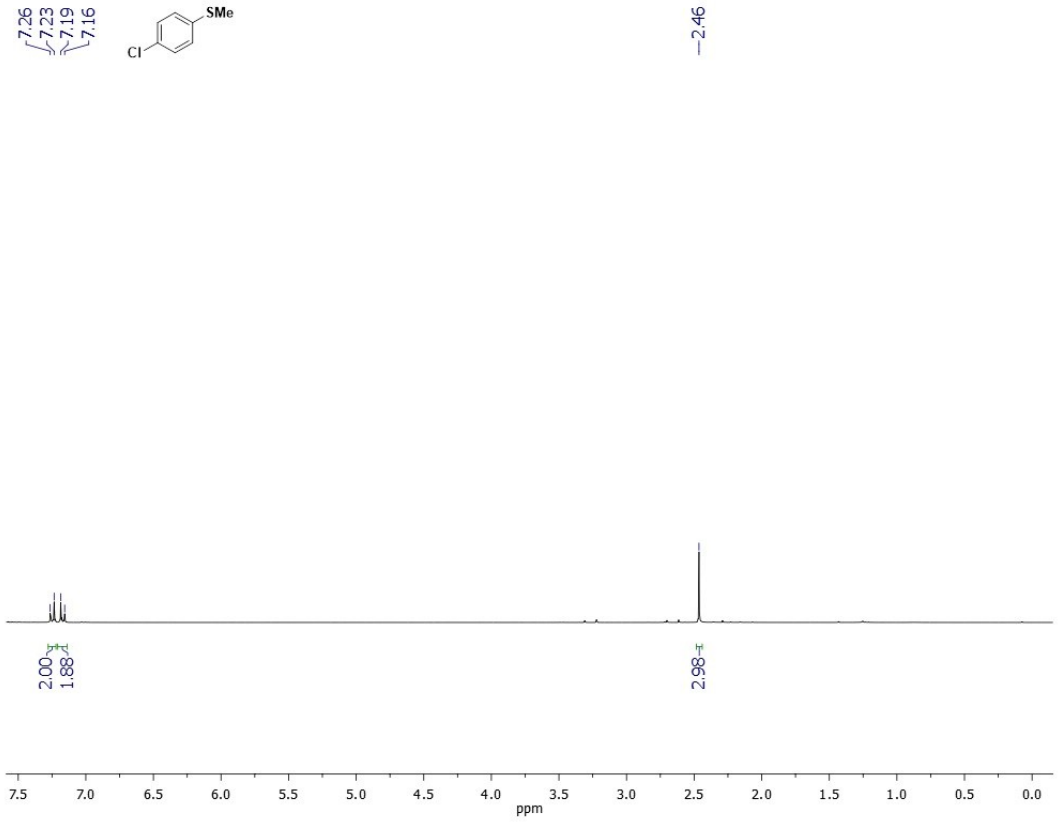
^{1}H-NMR (300 MHz, $CDCl_3$)



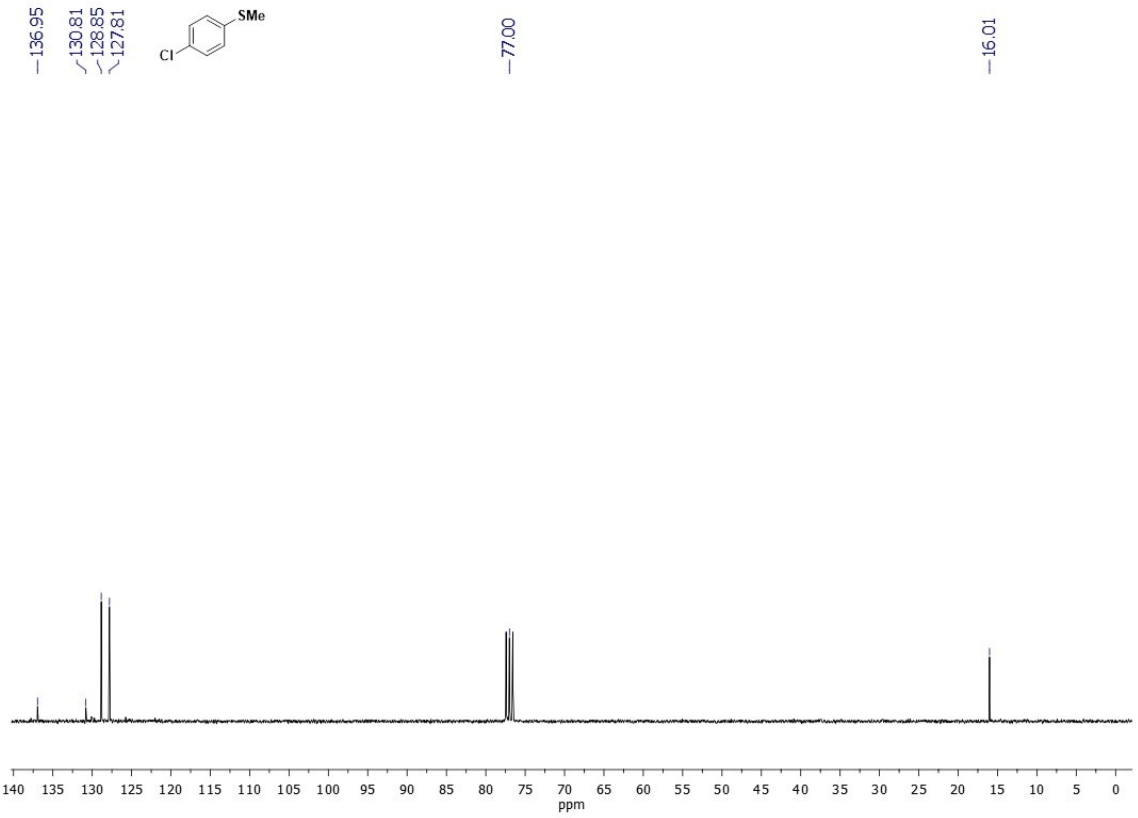
^{13}C-NMR (75 MHz, $CDCl_3$)



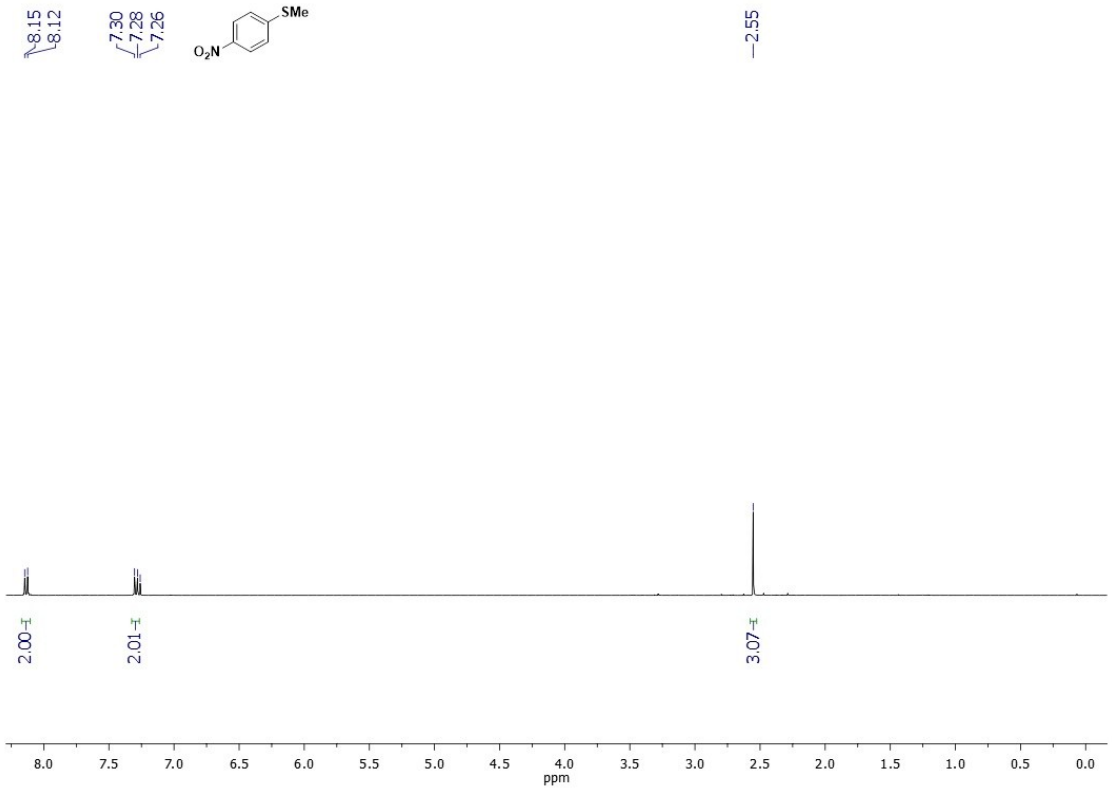
^{1}H-NMR (300 MHz, $CDCl_3$)



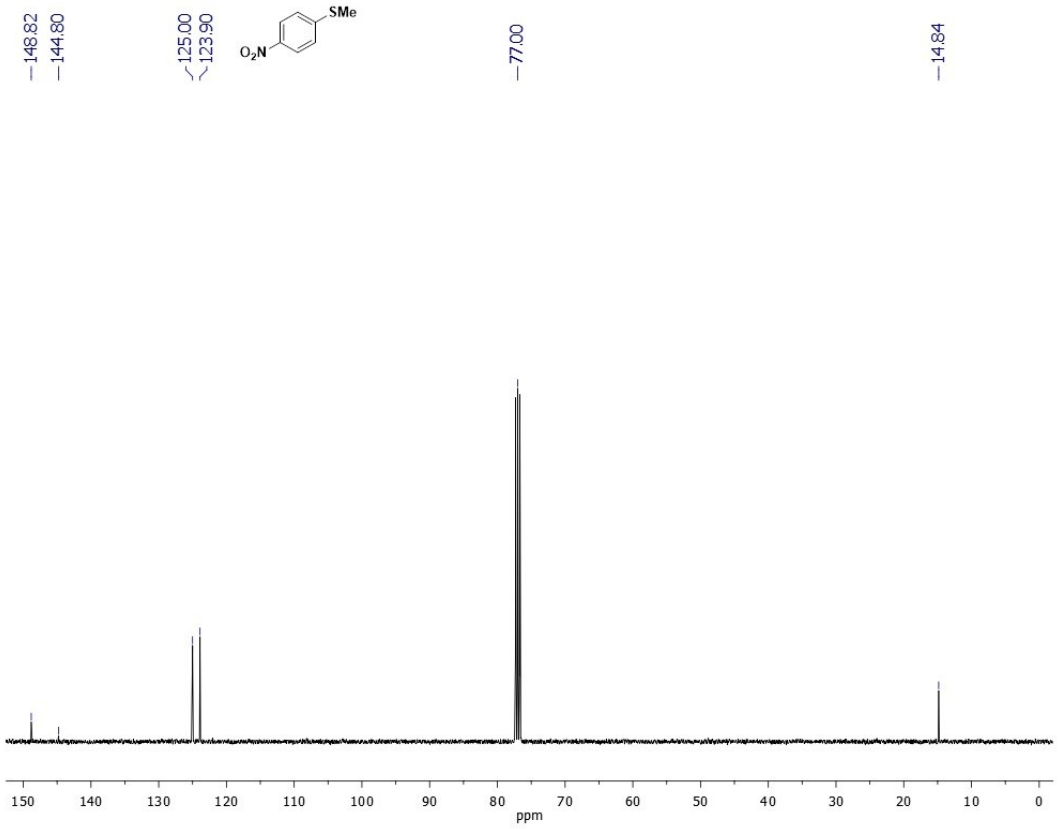
^{13}C-NMR (75 MHz, $CDCl_3$)


136.95
130.81
128.85
127.81
SMe
Cl
77.00
16.01
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
ppm

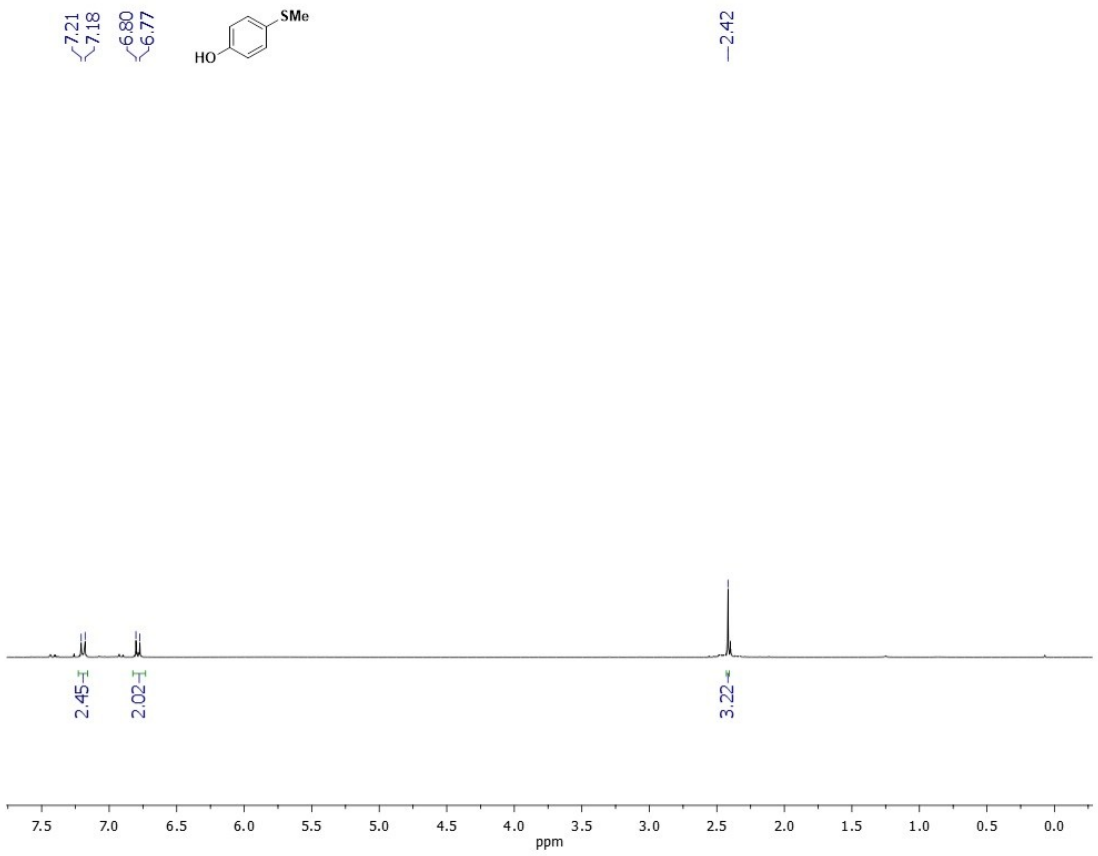
^{1}H-NMR (400 MHz, $CDCl_3$)



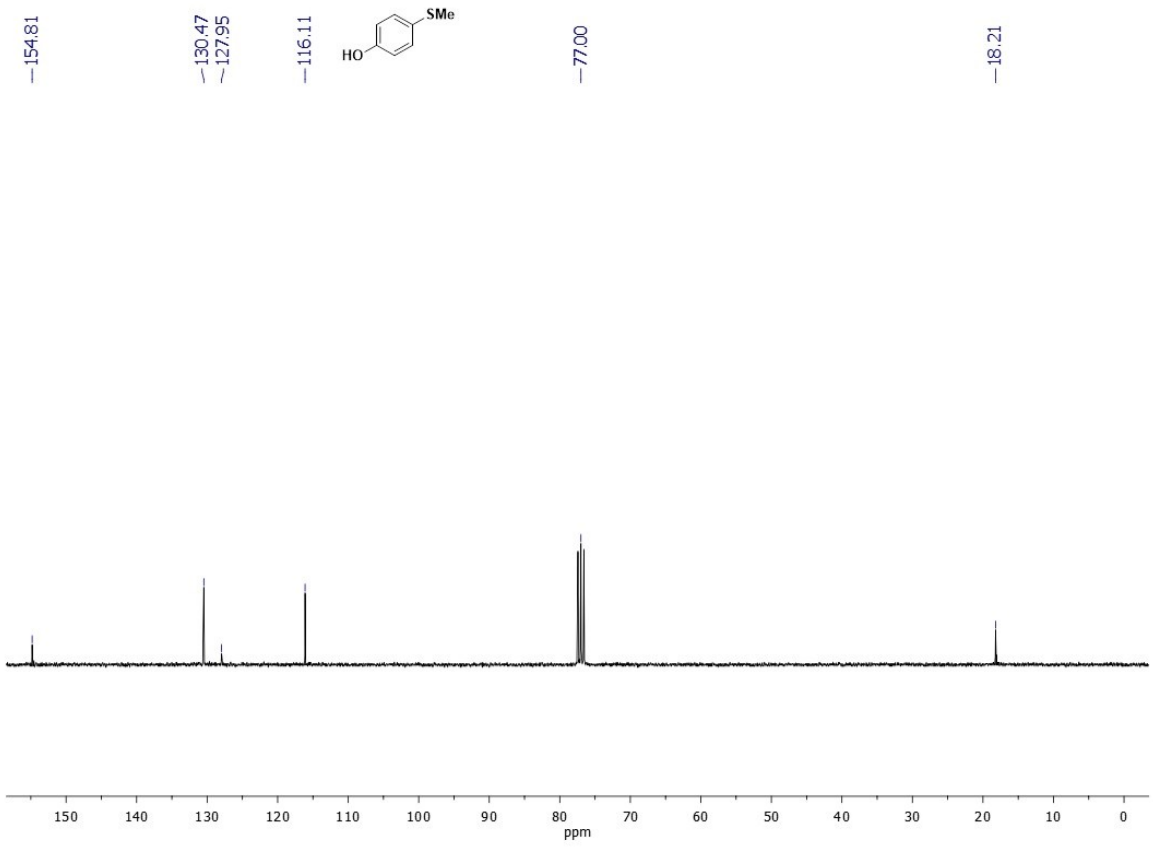
^{13}C-NMR (101 MHz, $CDCl_3$)



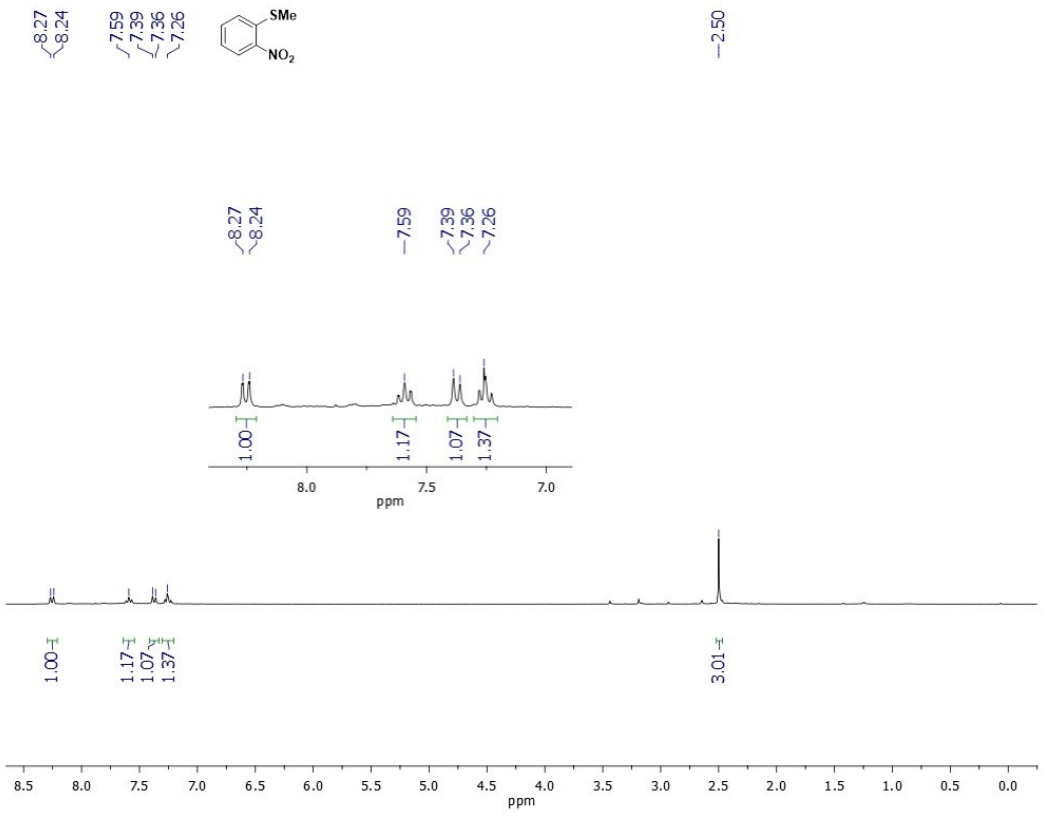
^{1}H-NMR (300 MHz, $CDCl_3$)



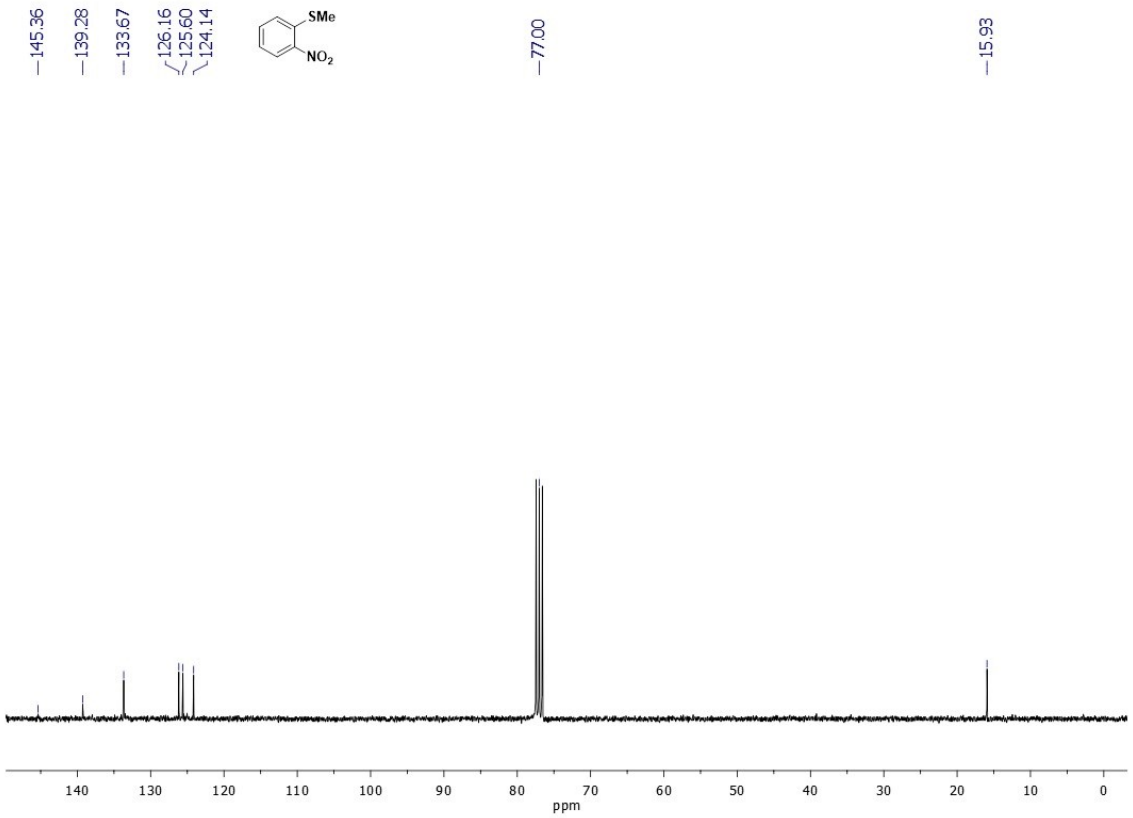
^{13}C-NMR (75 MHz, $CDCl_3$)



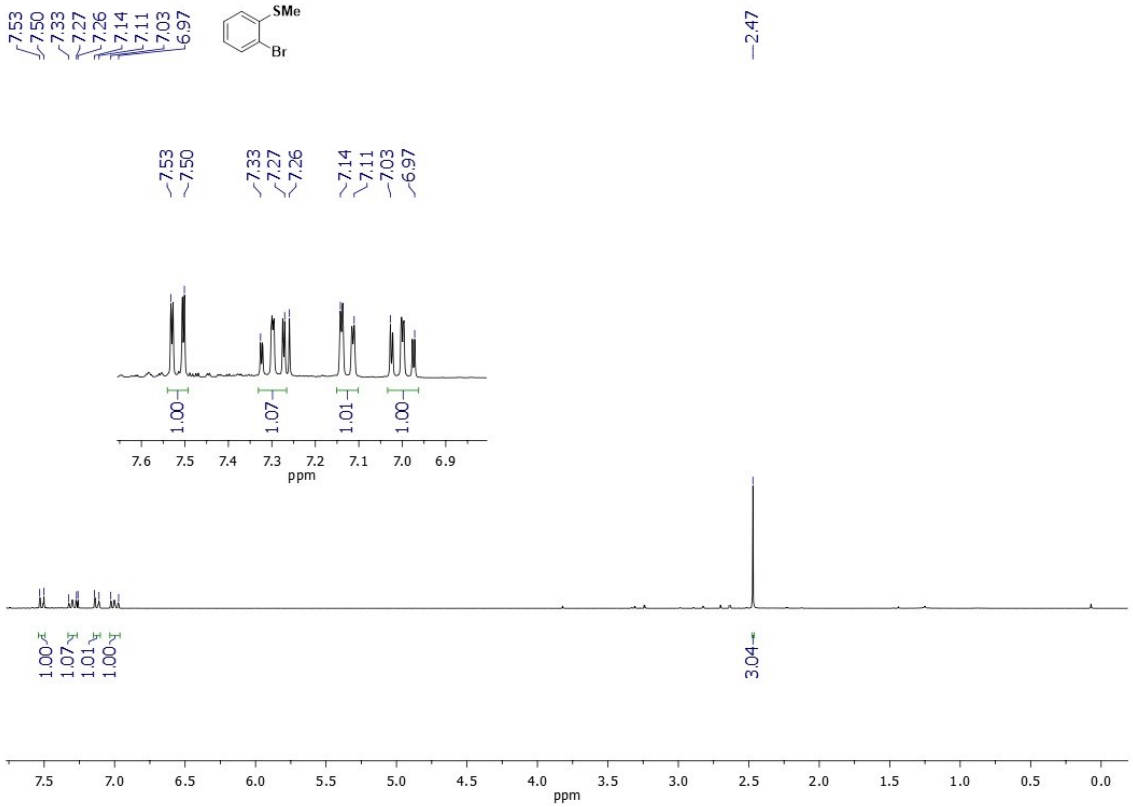
^{1}H-NMR (300 MHz, $CDCl_3$)



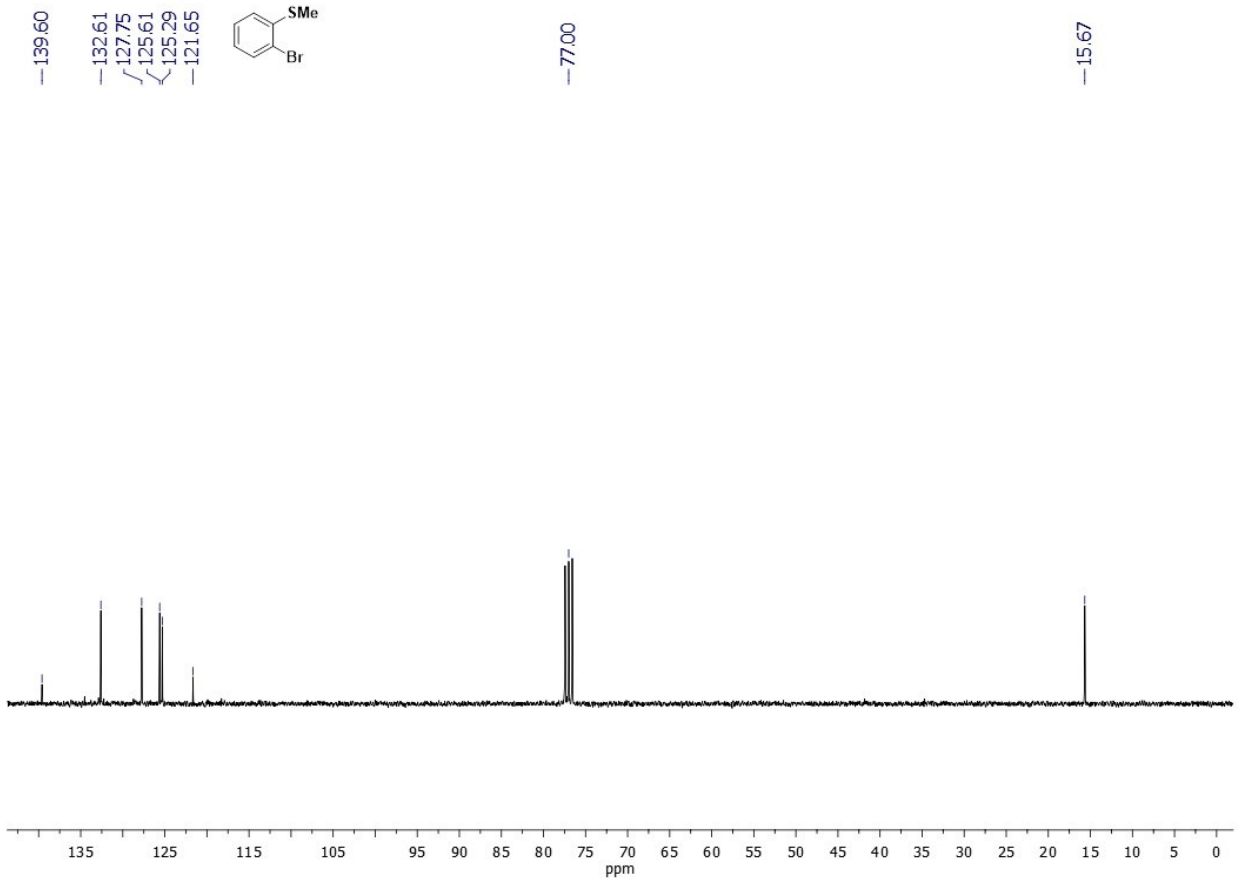
^{13}C-NMR (75 MHz, $CDCl_3$)



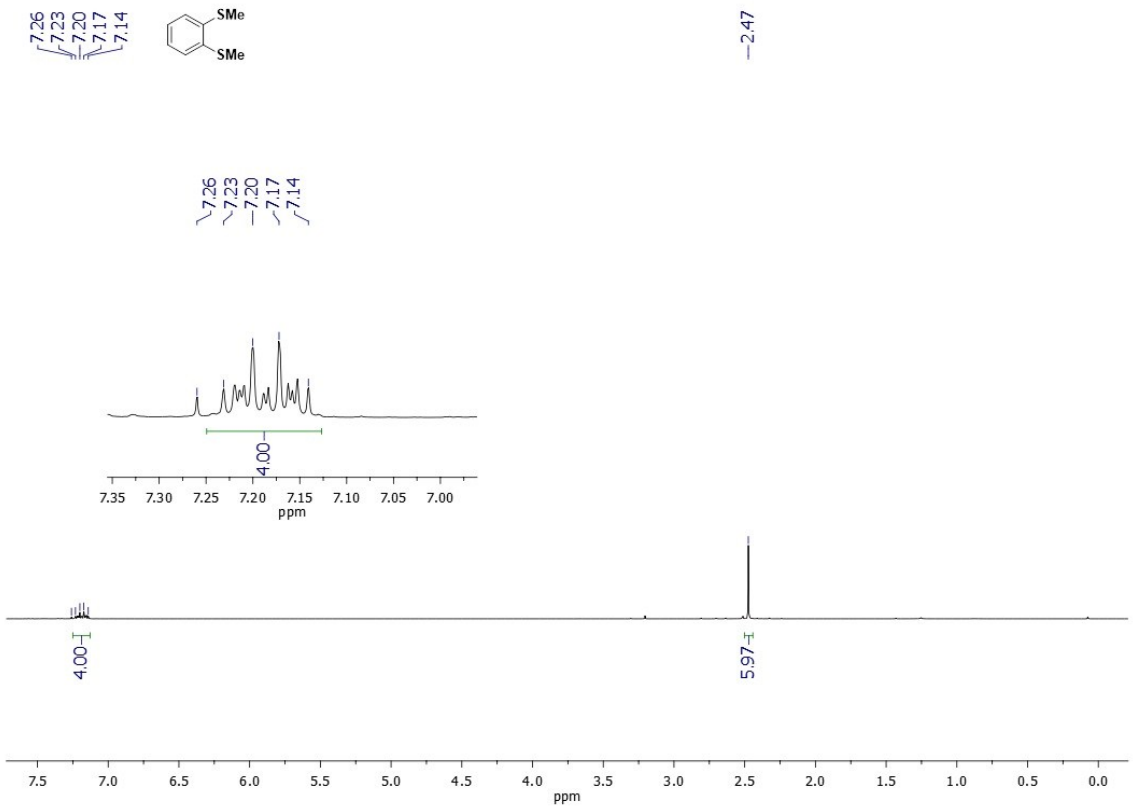
^{1}H-NMR (300 MHz, $CDCl_3$)



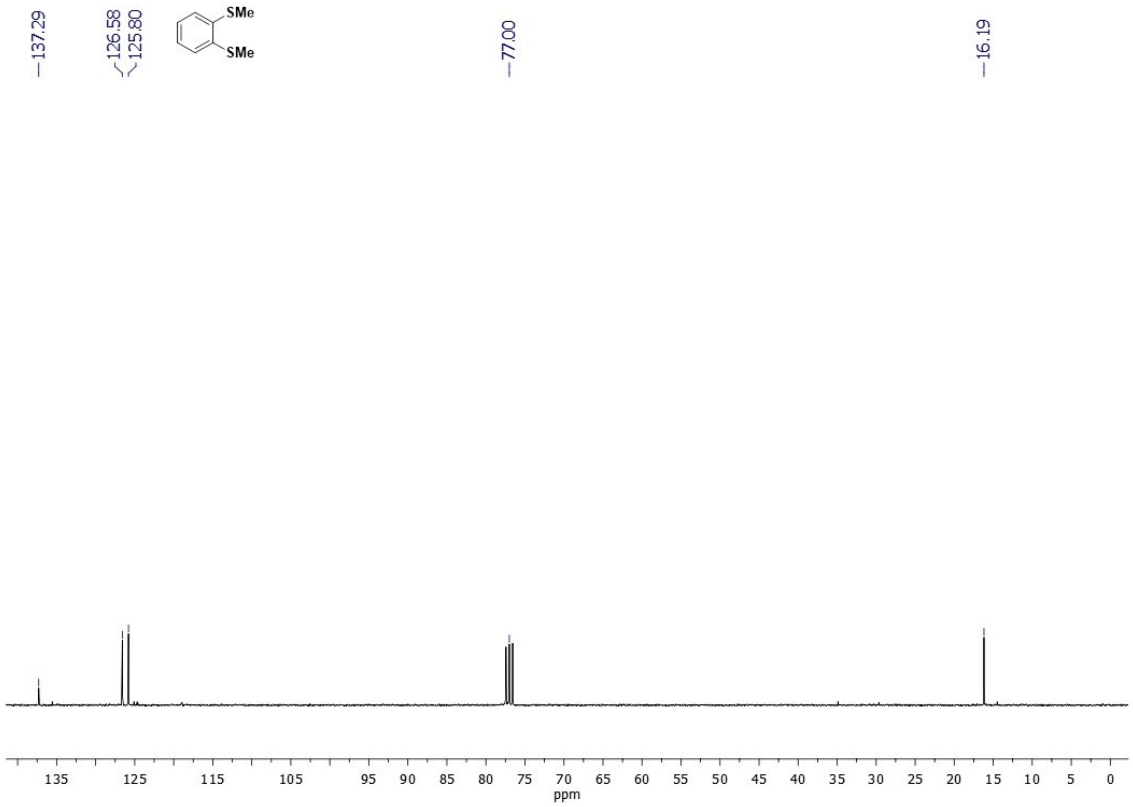
^{13}C-NMR (75 MHz, $CDCl_3$)



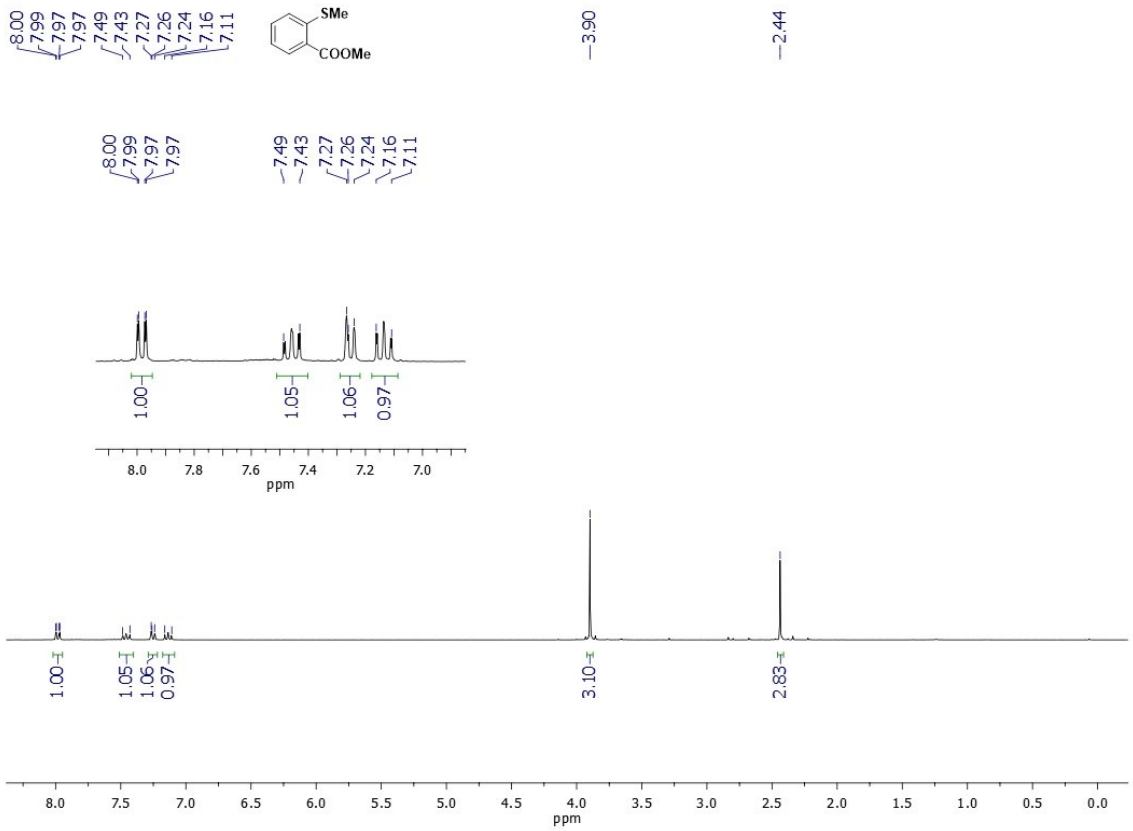
^{1}H-NMR (300 MHz, $CDCl_3$)



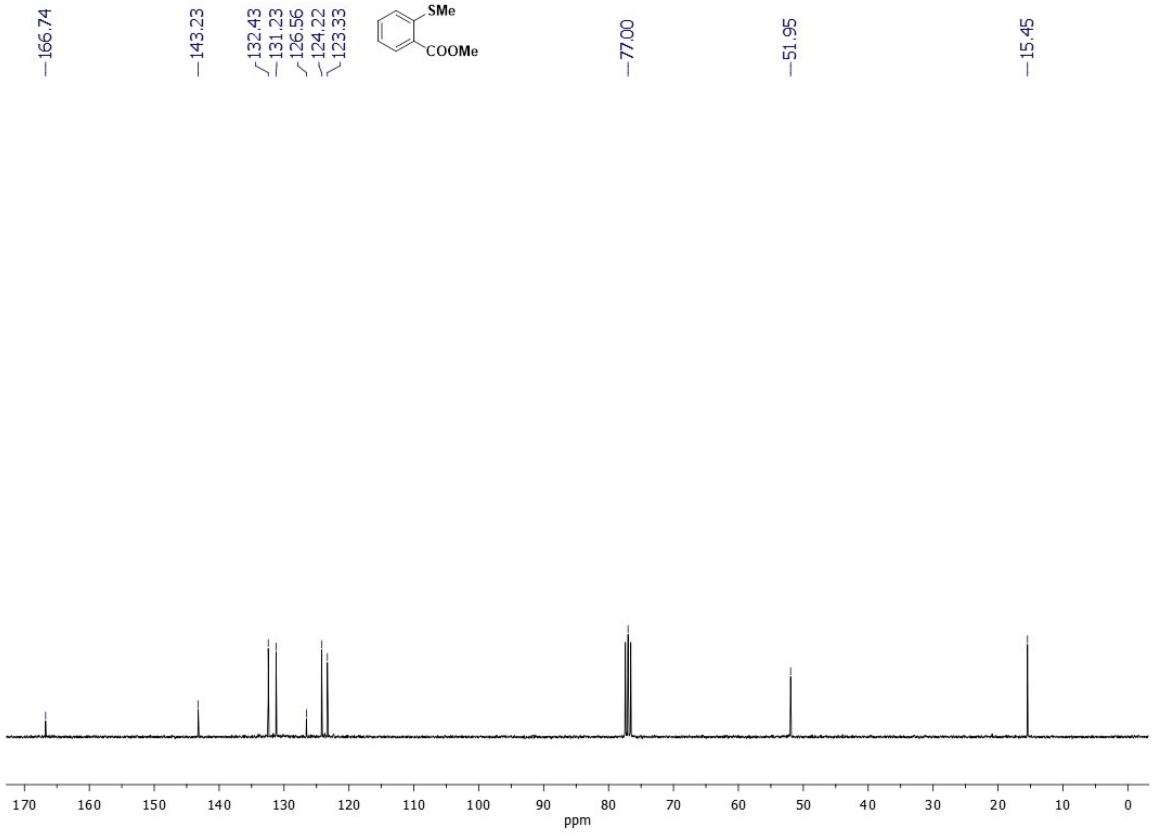
^{13}C-NMR (75 MHz, $CDCl_3$)



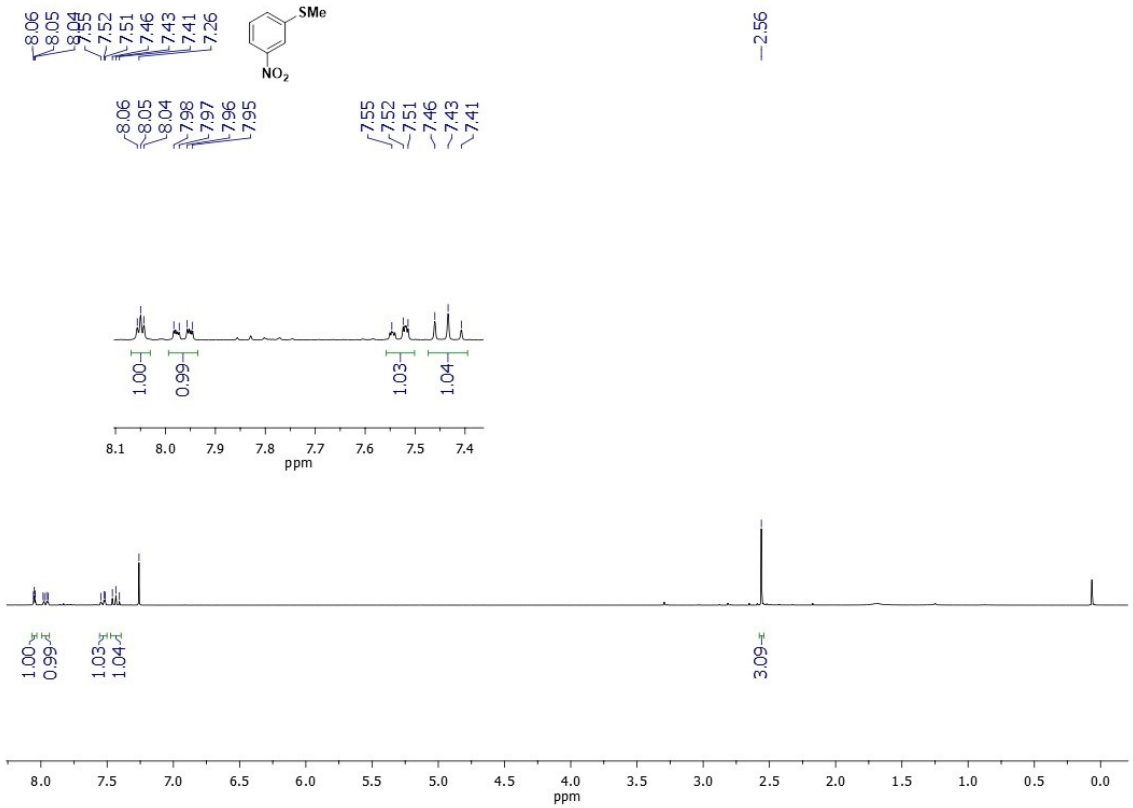
^{1}H-NMR (300 MHz, $CDCl_3$)



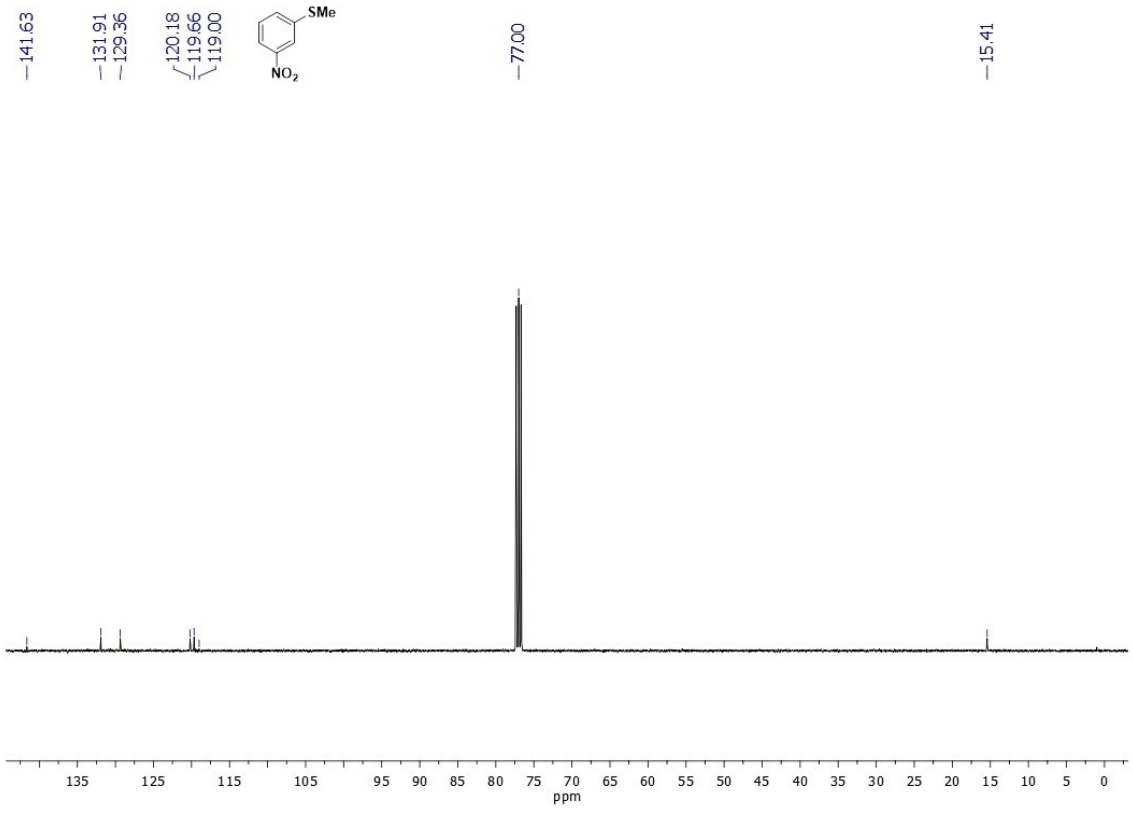
^{13}C-NMR (75 MHz, $CDCl_3$)



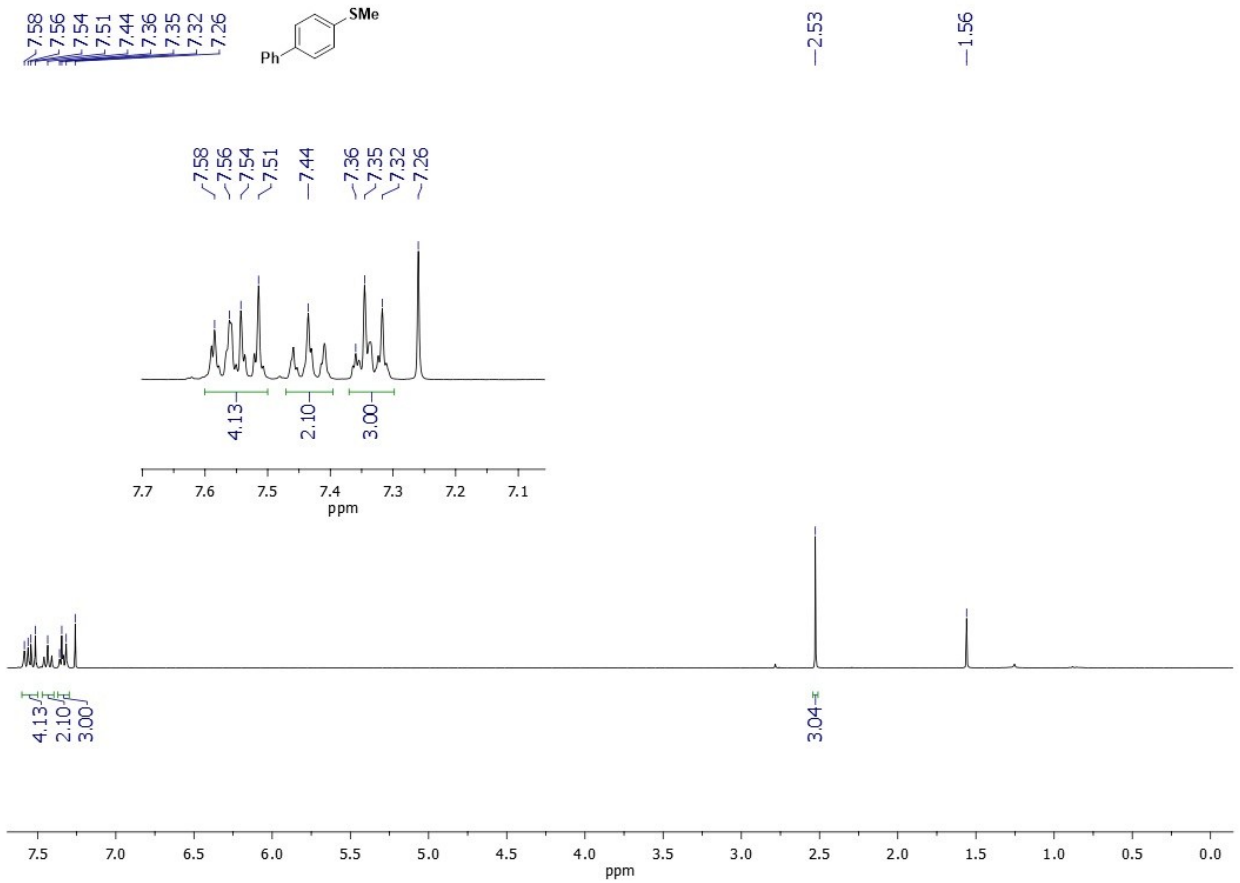
^{1}H-NMR (300 MHz, $CDCl_3$)



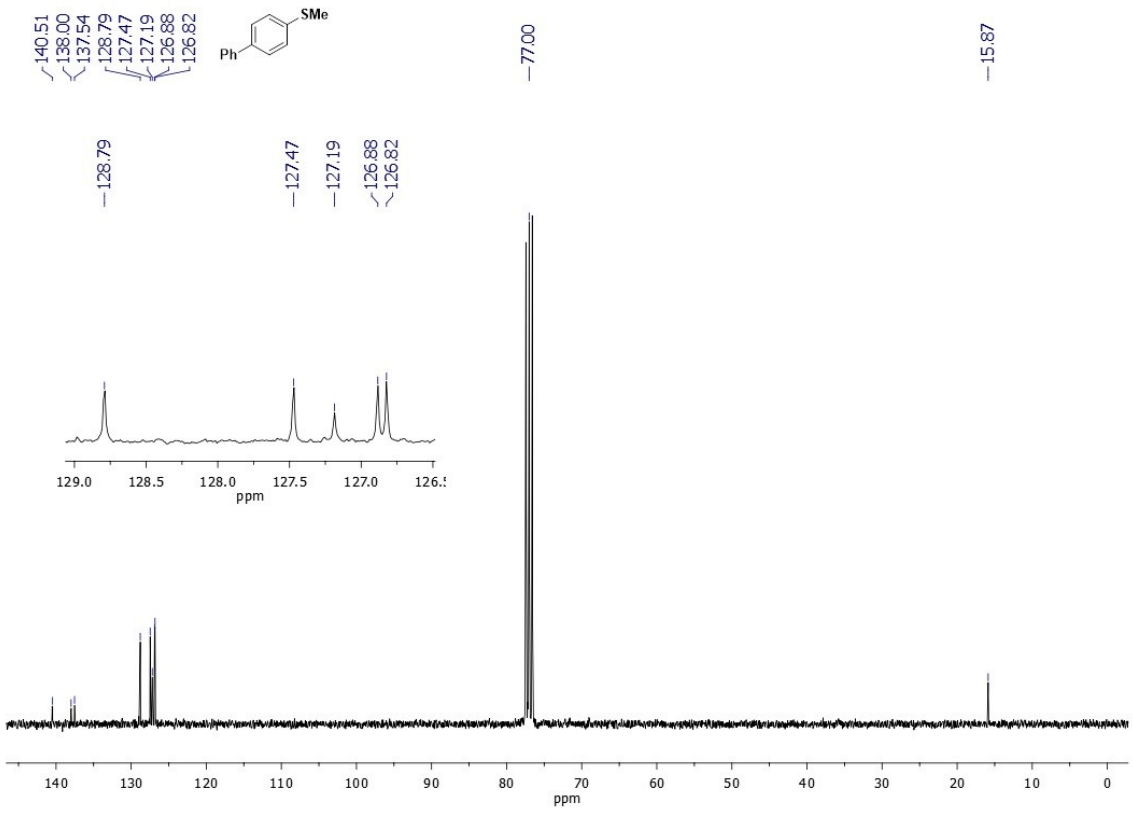
^{13}C-NMR (101 MHz, $CDCl_3$)



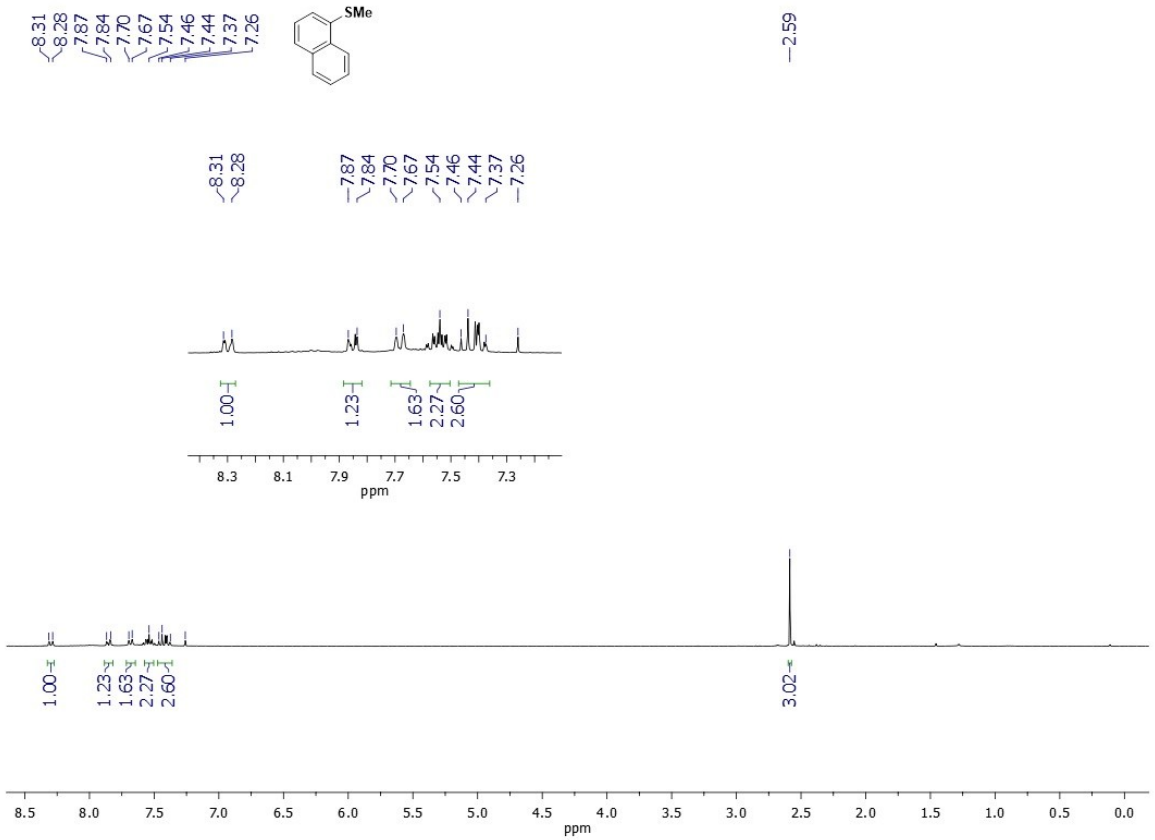
^{1}H-NMR (300 MHz, $CDCl_3$)



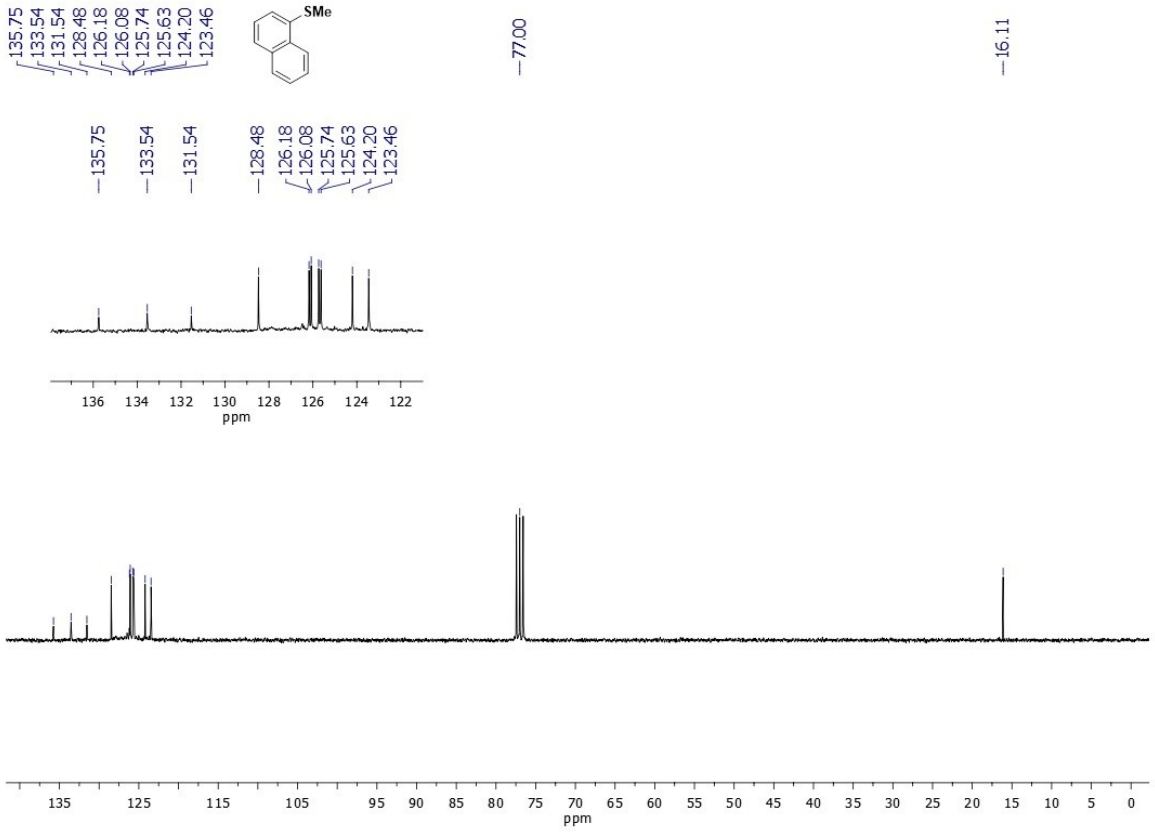
^{13}C-NMR (101 MHz, $CDCl_3$)



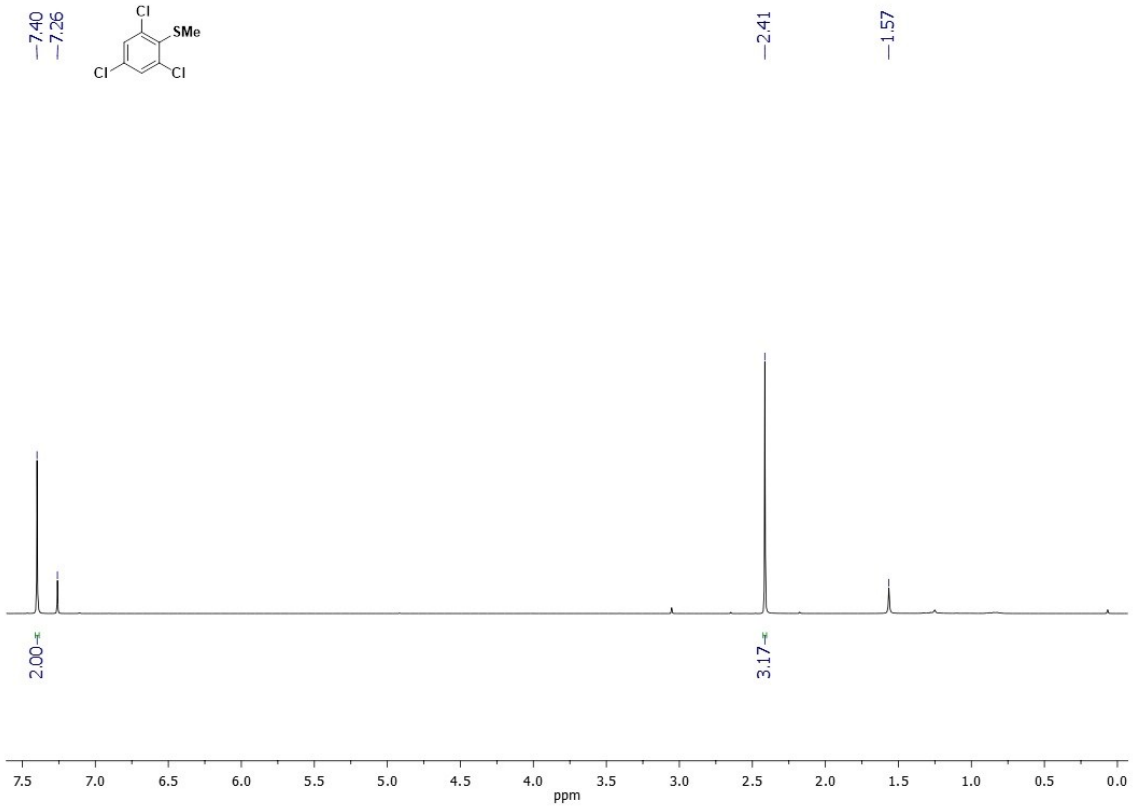
^{1}H-NMR (300 MHz, $CDCl_3$)



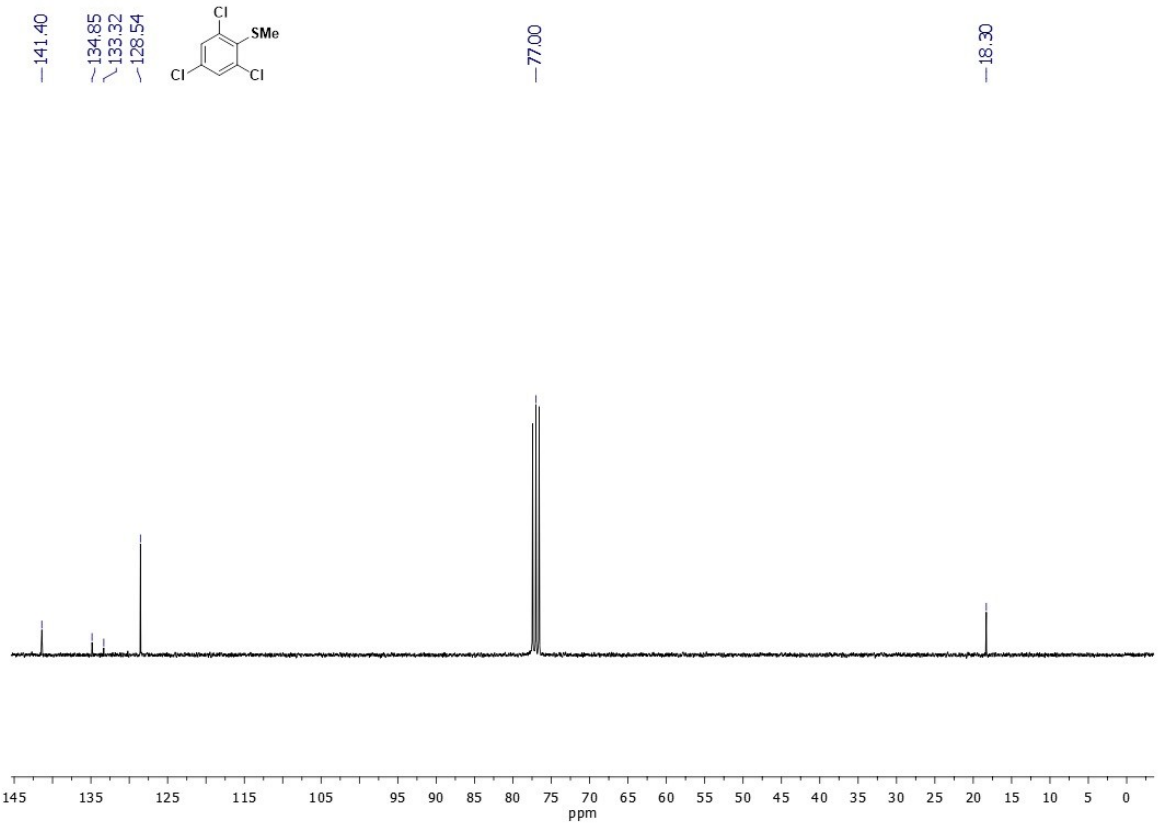
^{13}C-NMR (75 MHz, $CDCl_3$)



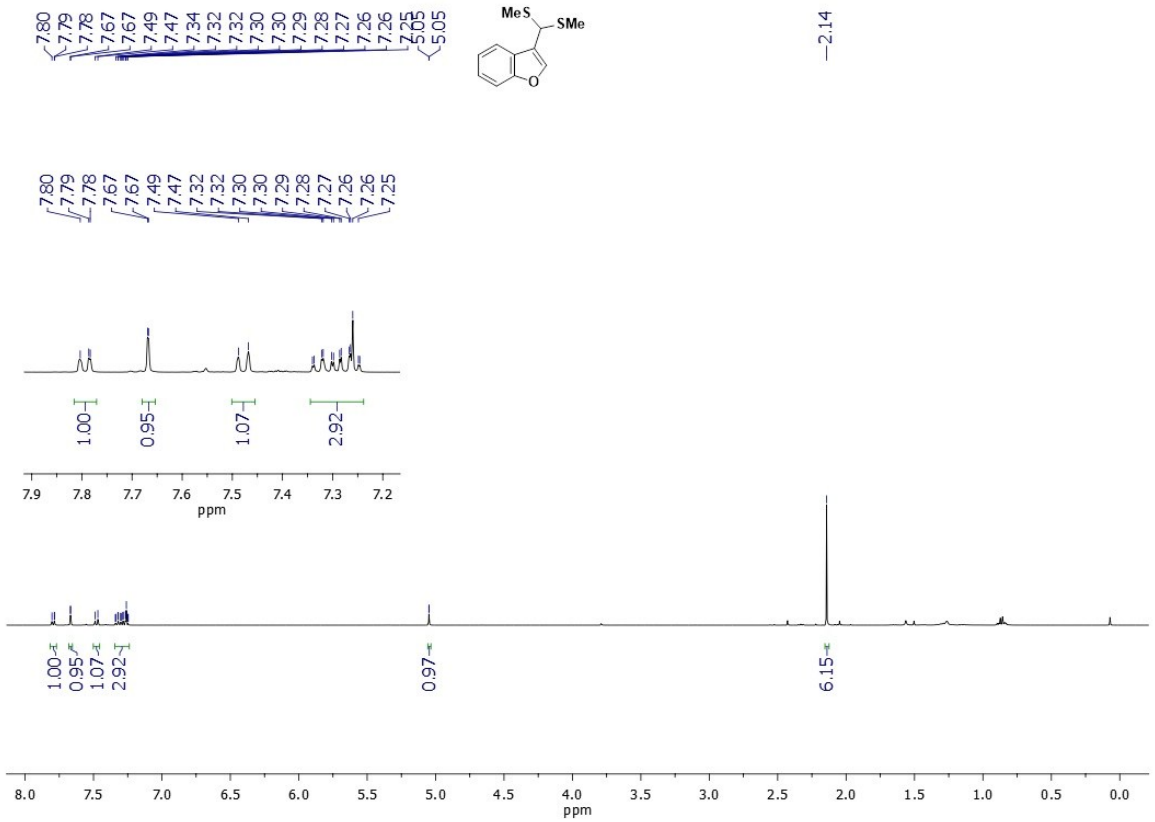
^{1}H-NMR (300 MHz, $CDCl_3$)



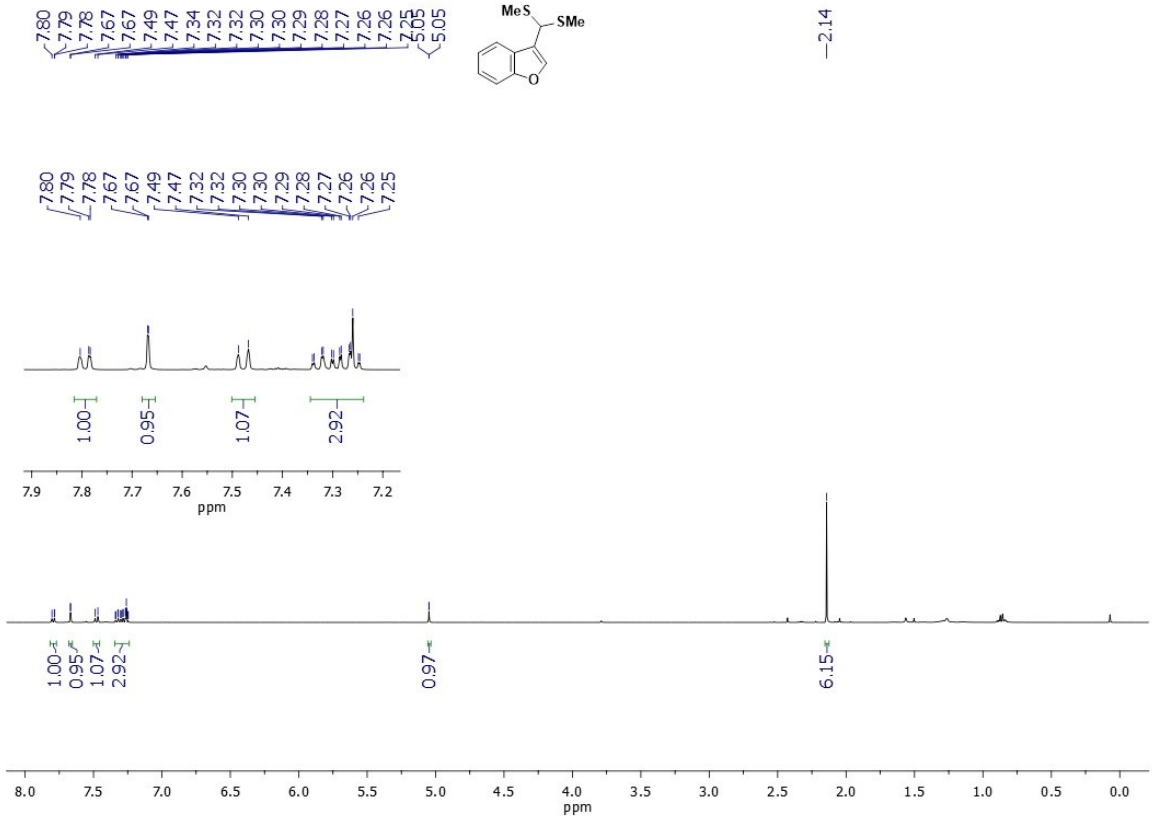
^{13}C-NMR (75 MHz, $CDCl_3$)



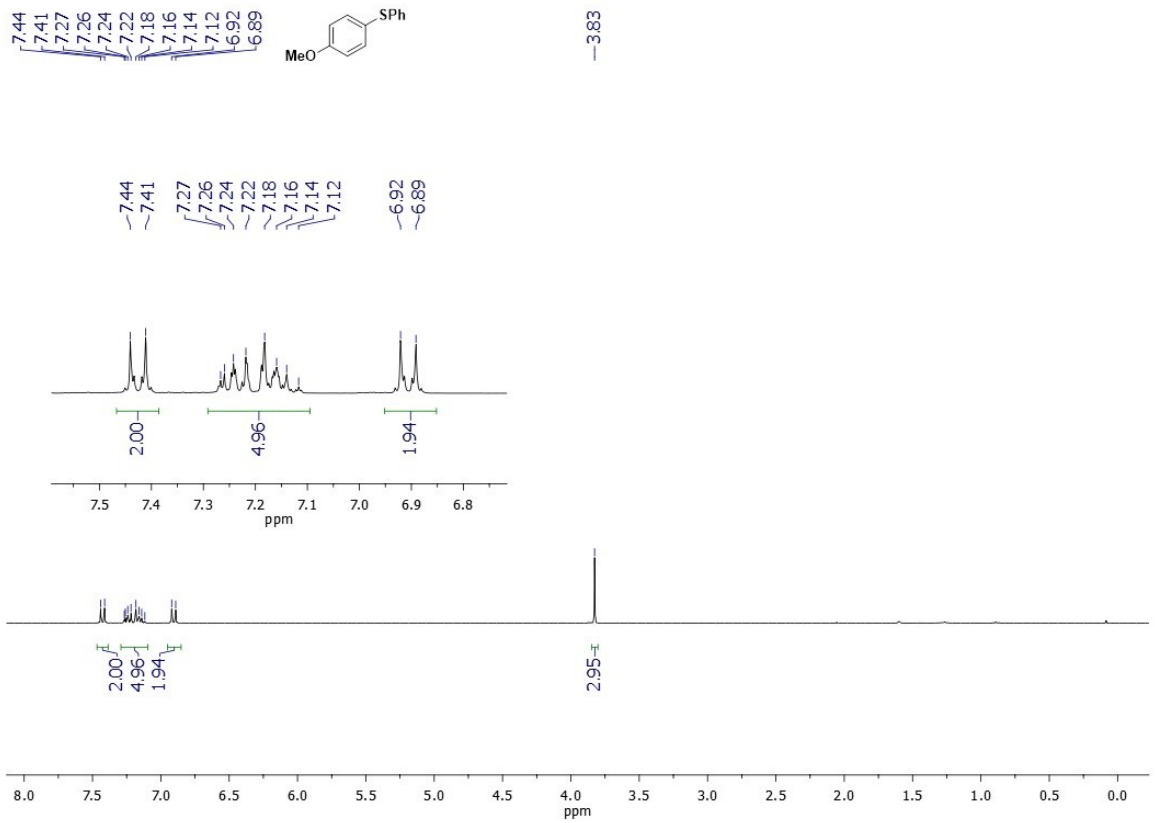
^{1}H-NMR (400 MHz, $CDCl_3$)



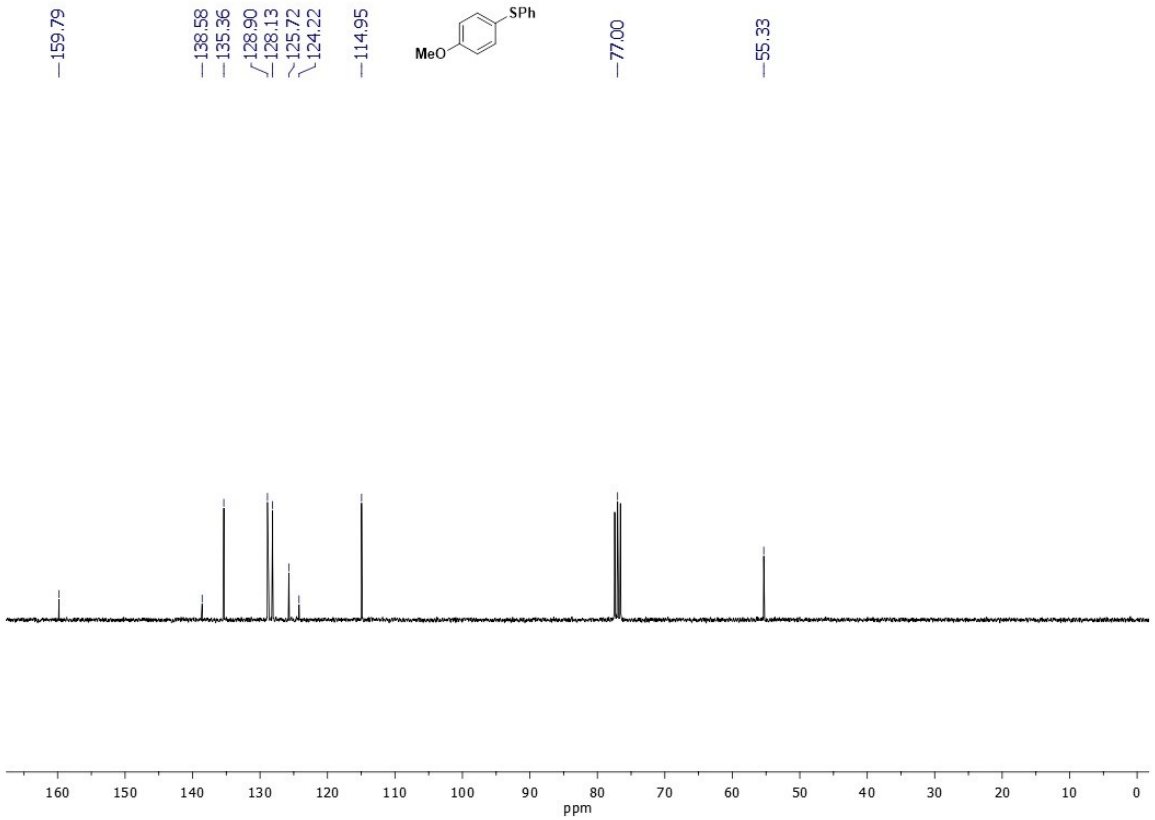
^{13}C-NMR (75 MHz, $CDCl_3$)



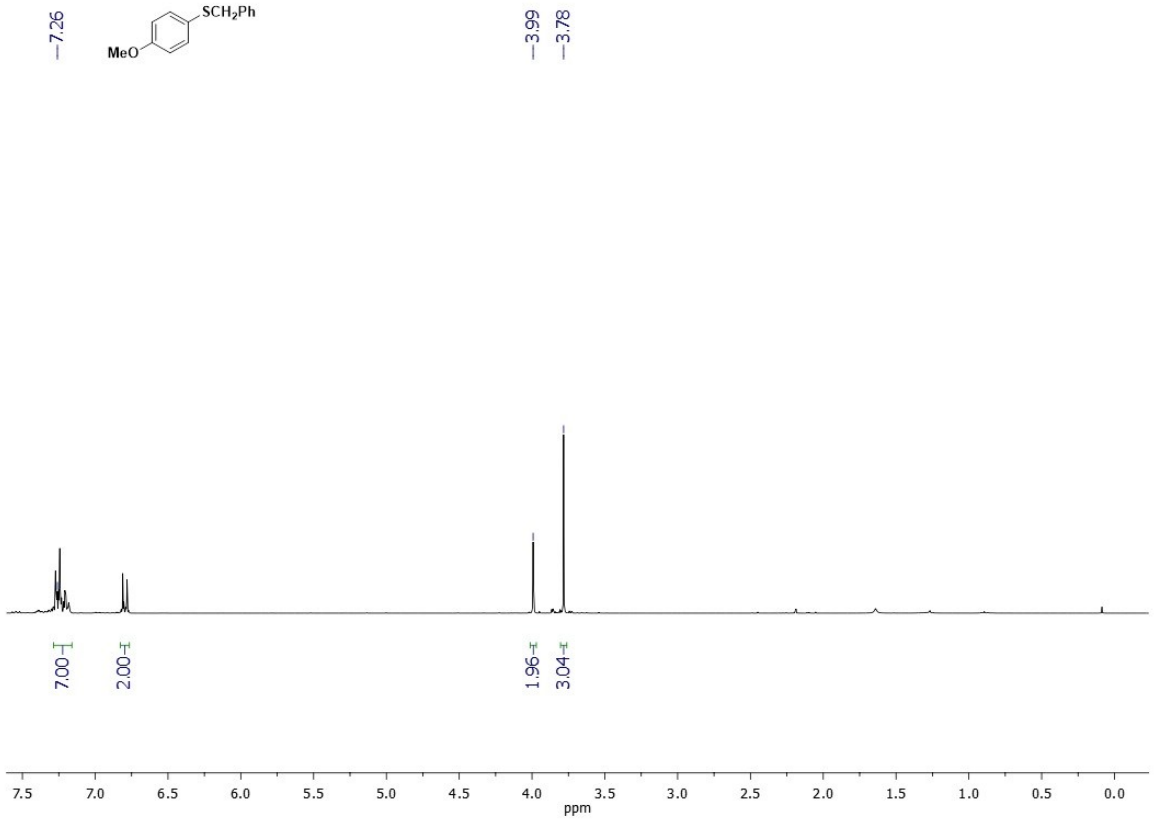
^{1}H-NMR (300 MHz, $CDCl_3$)



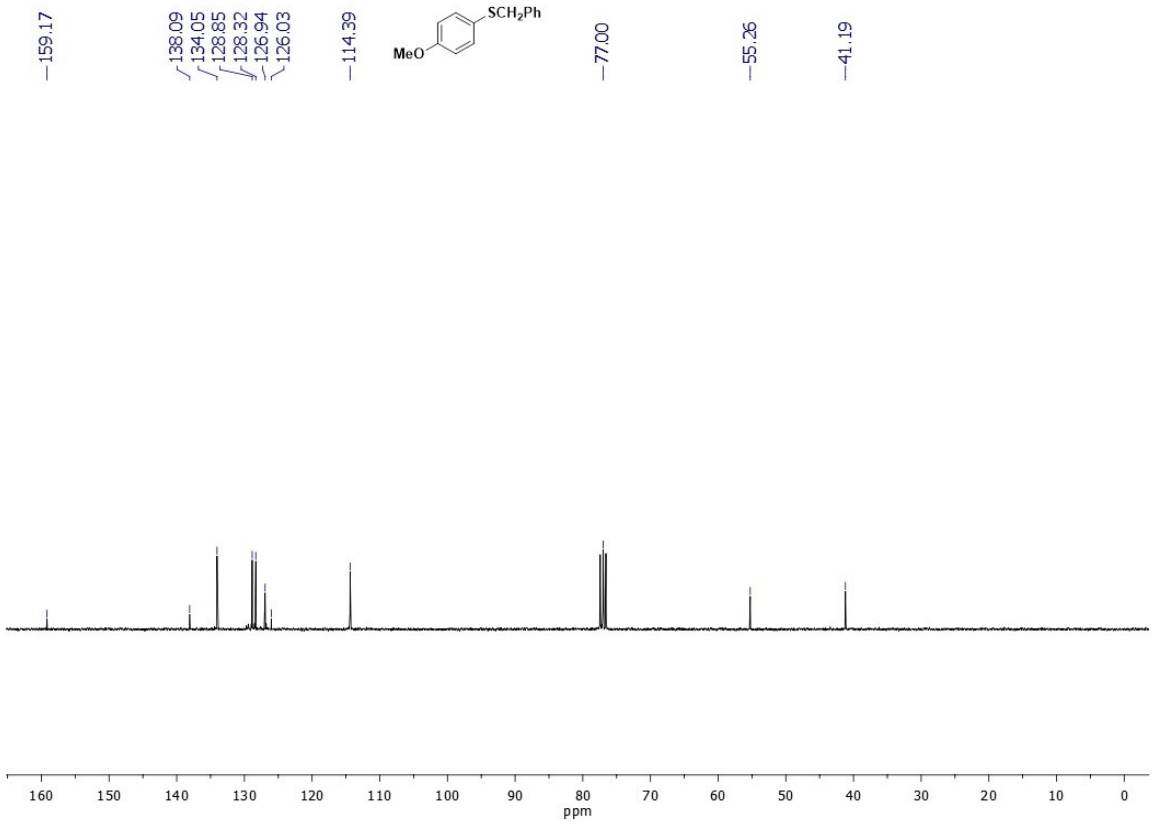
^{13}C-NMR (75 MHz, $CDCl_3$)



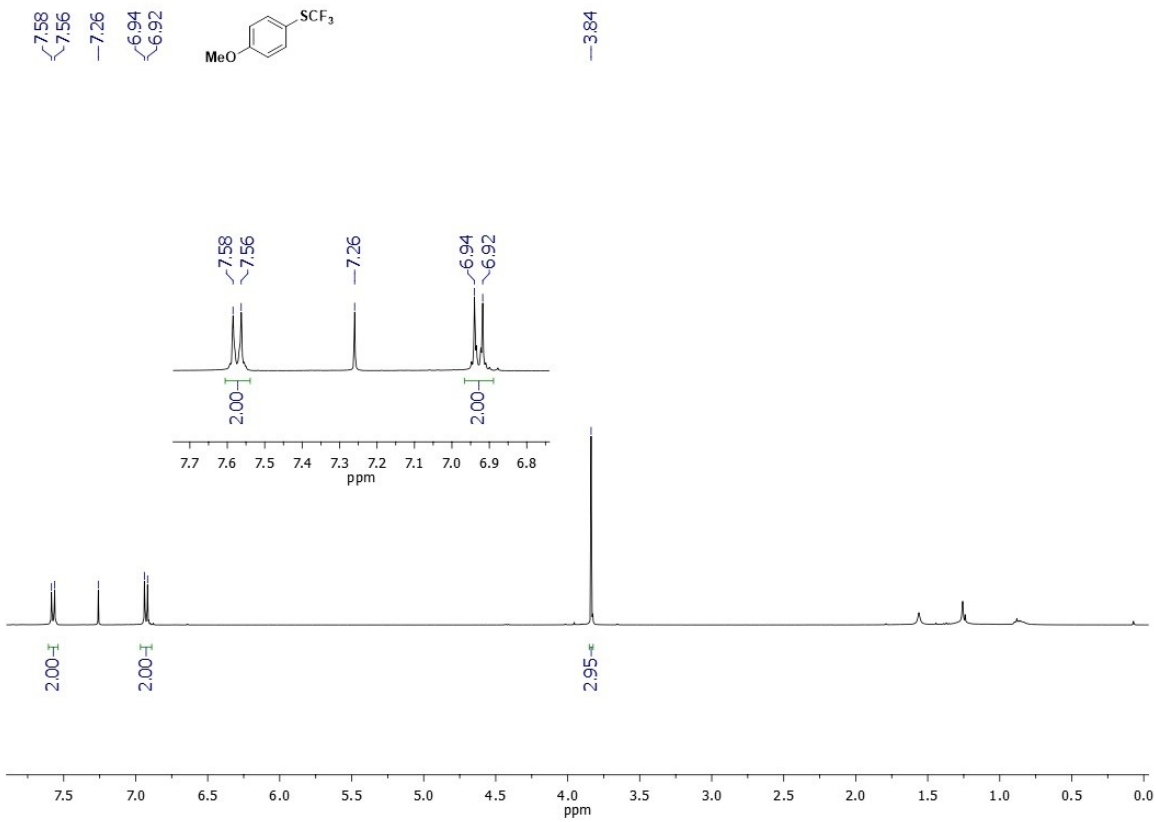
^{1}H-NMR (300 MHz, $CDCl_3$)



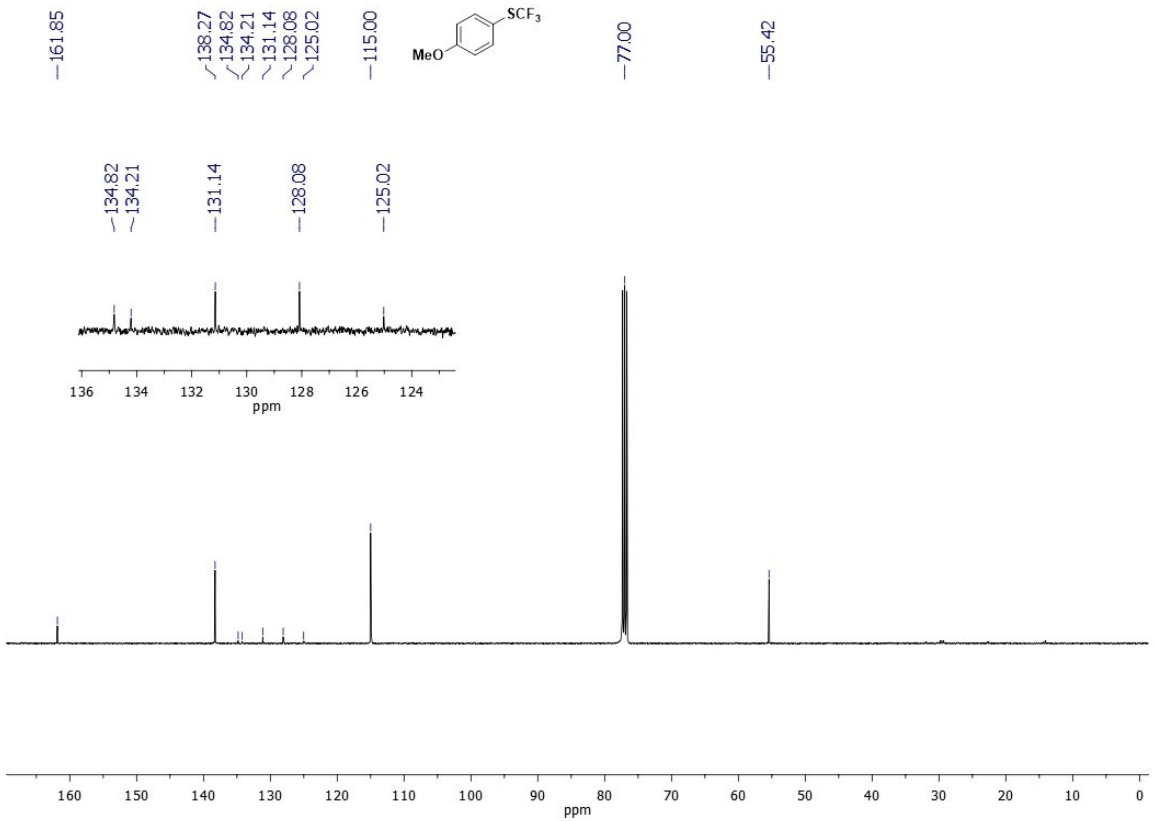
^{13}C-NMR (75 MHz, $CDCl_3$)



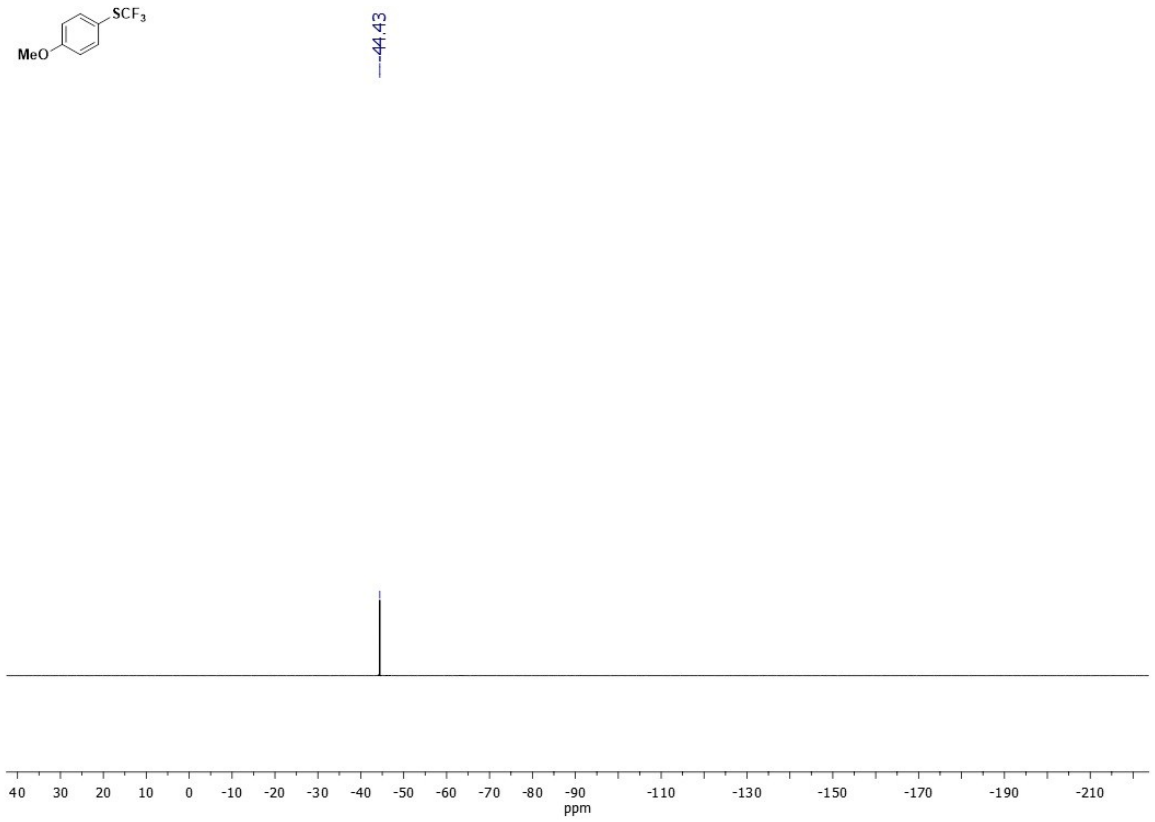
^{1}H-NMR (400 MHz, $CDCl_3$)



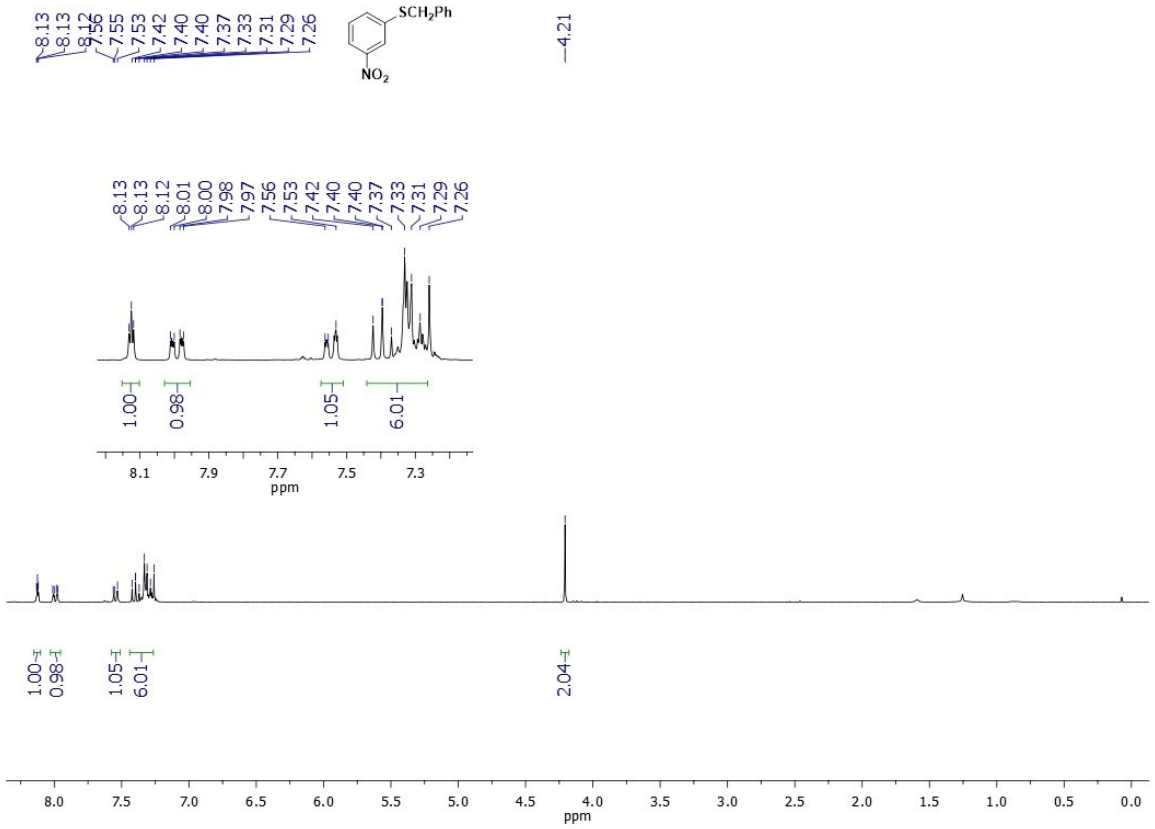
^{13}C-NMR (75 MHz, $CDCl_3$)



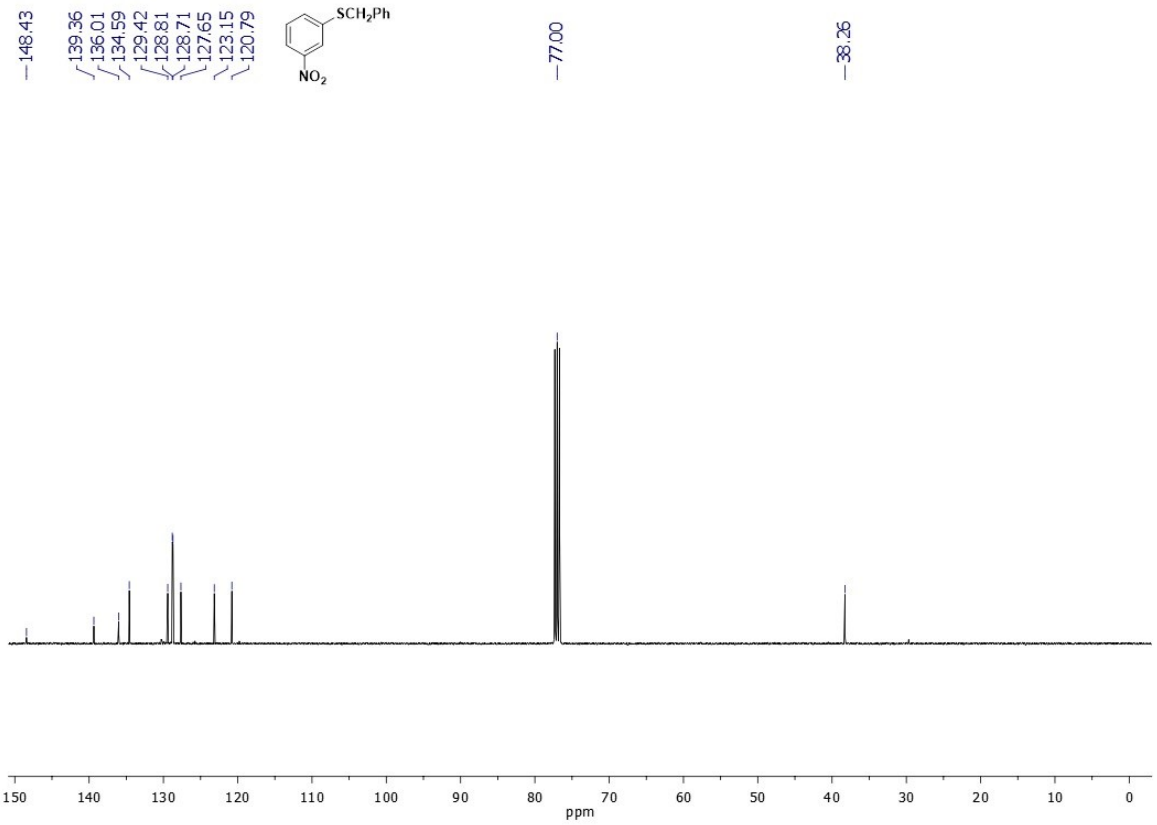
^{19}F-NMR (282 MHz, $CDCl_3$)



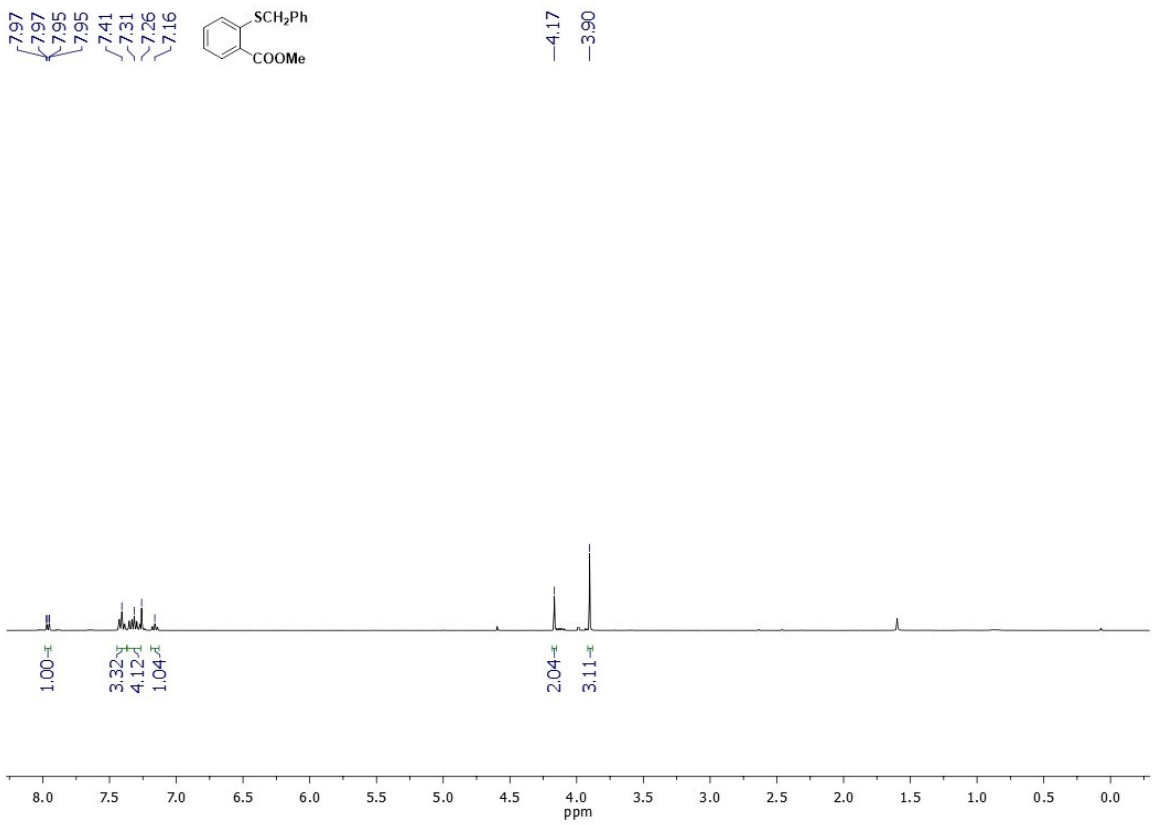
^{1}H-NMR (300 MHz, $CDCl_3$)



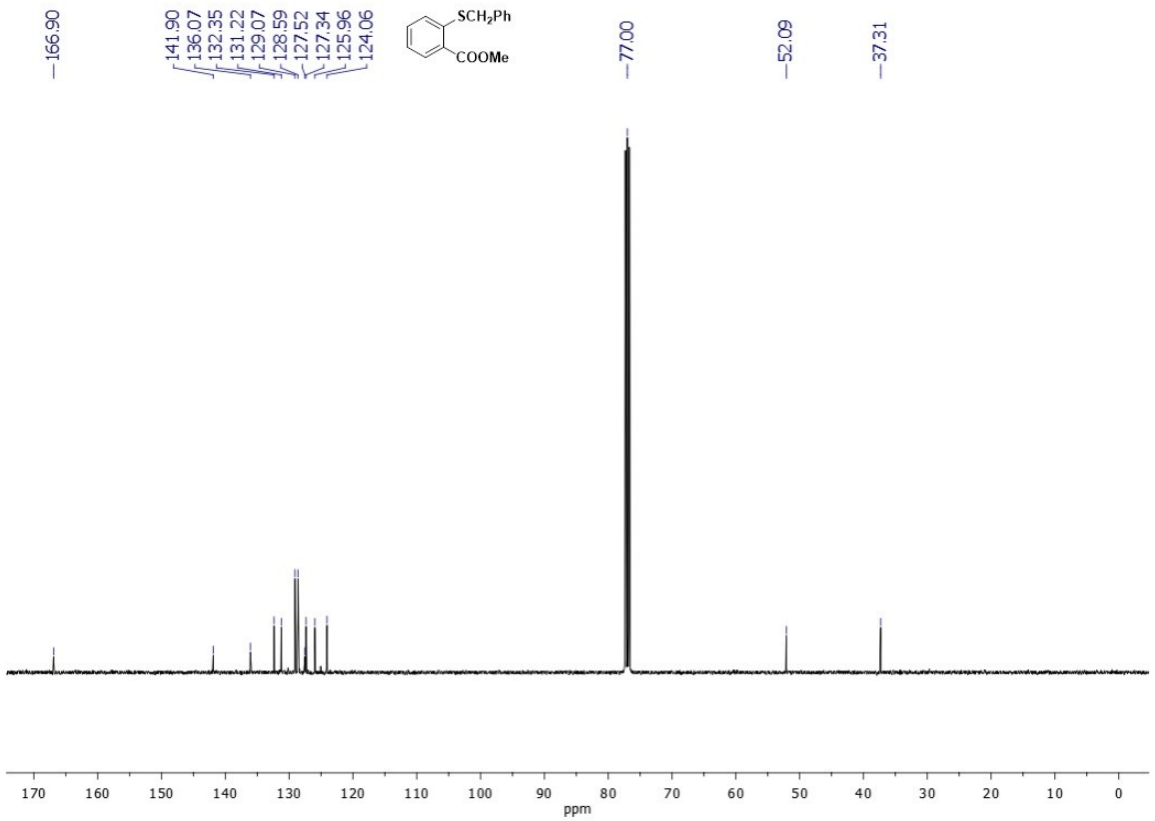
^{13}C-NMR (101 MHz, $CDCl_3$)



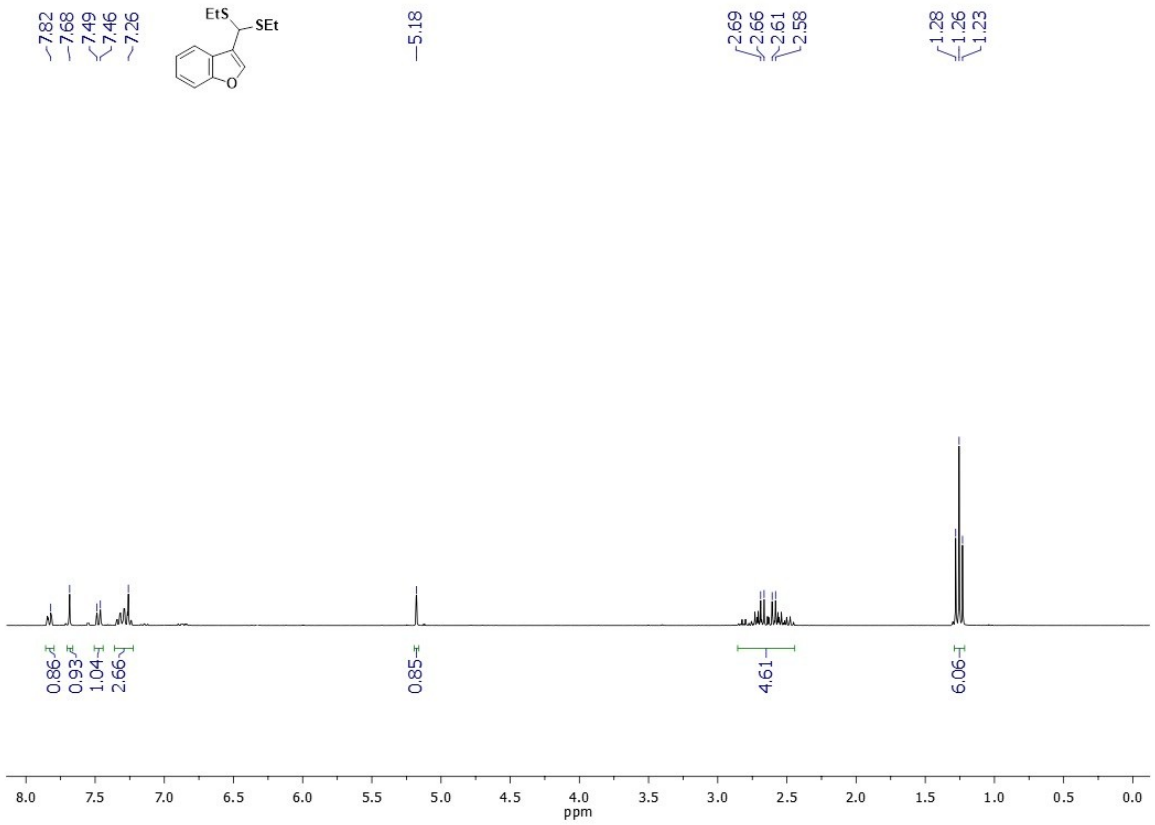
^{1}H-NMR (400 MHz, $CDCl_3$)



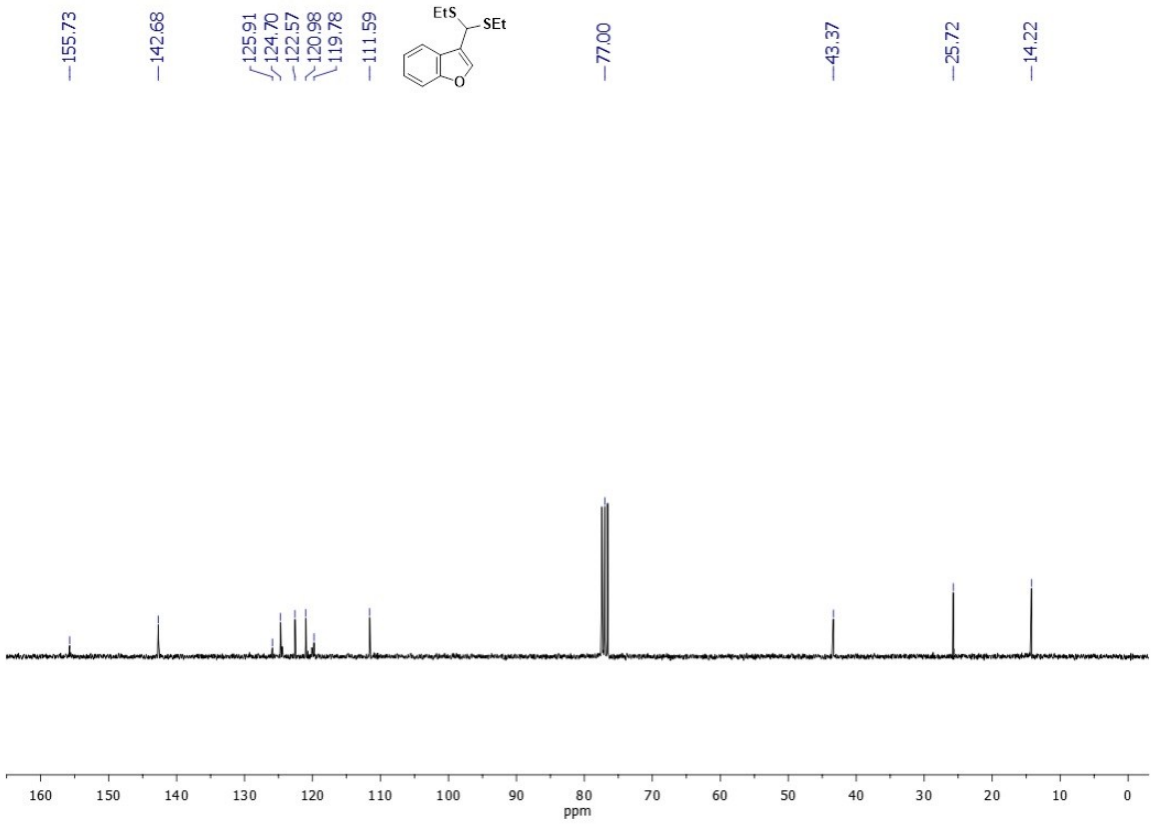
^{13}C-NMR (75 MHz, $CDCl_3$)



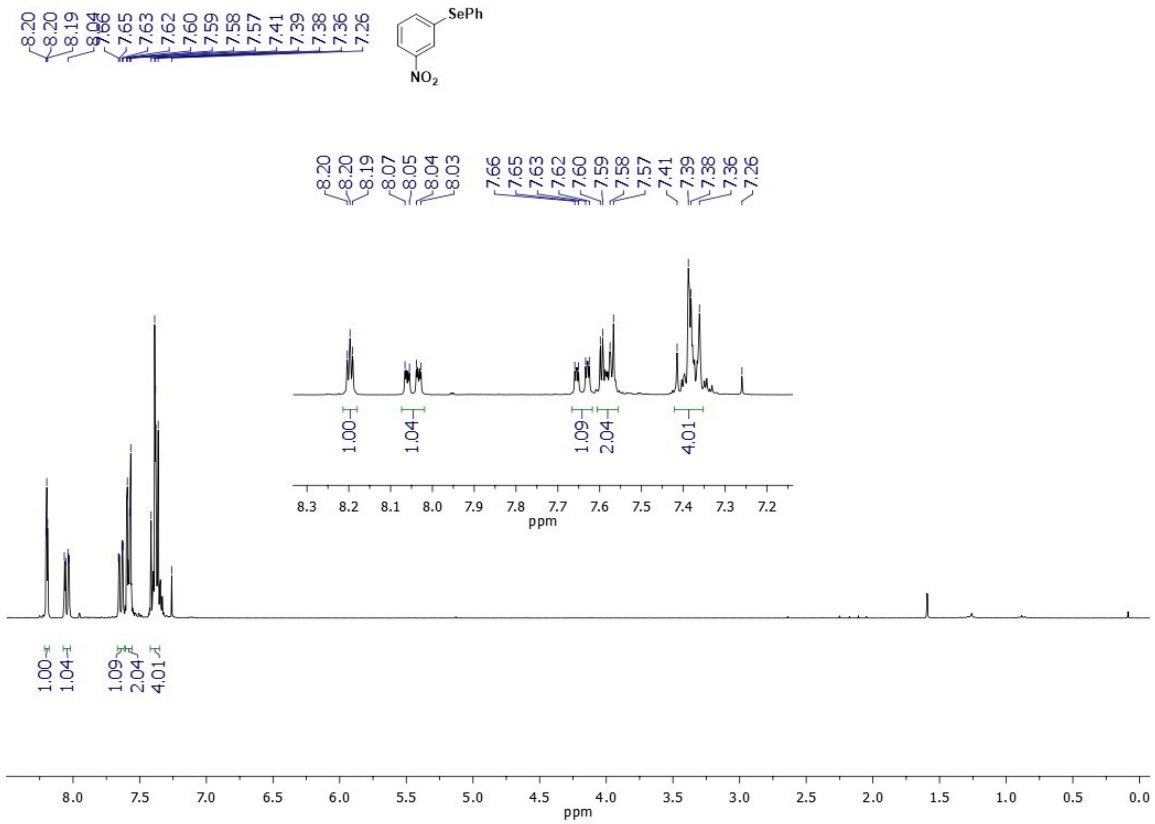
^{1}H-NMR (300 MHz, $CDCl_3$)



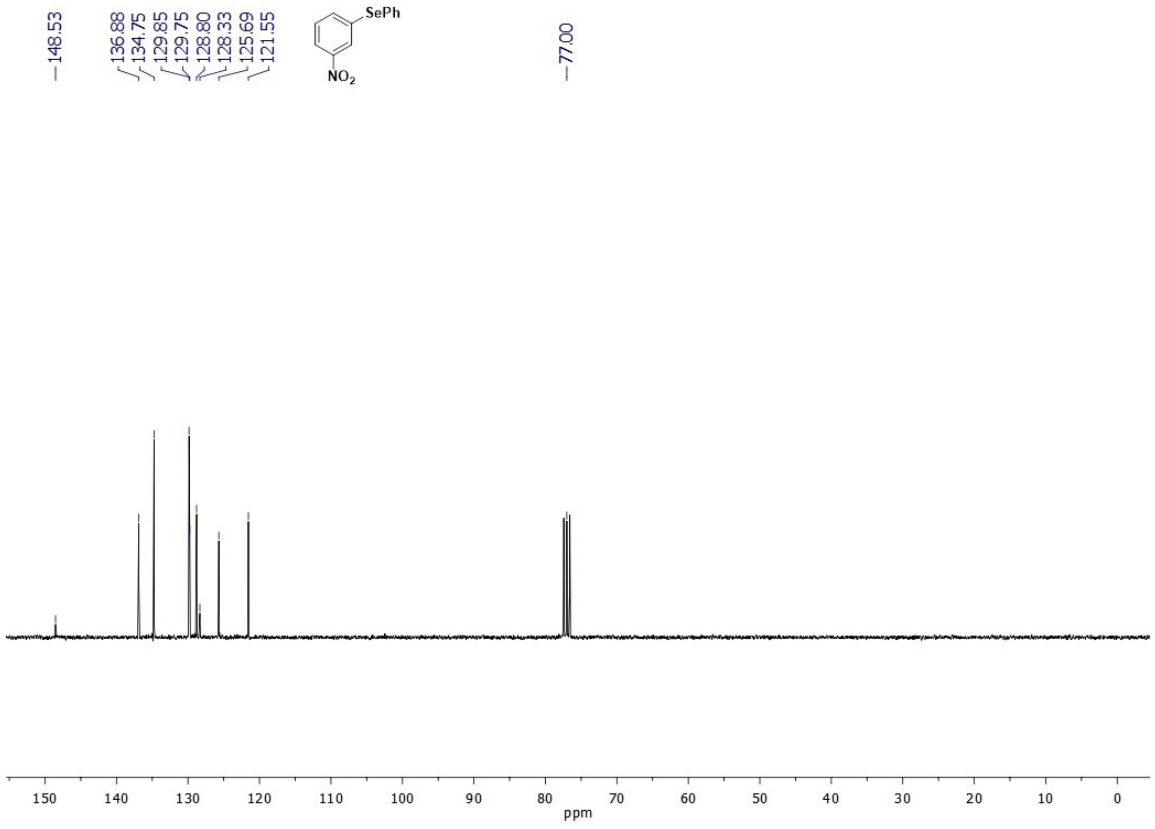
^{13}C-NMR (75 MHz, $CDCl_3$)



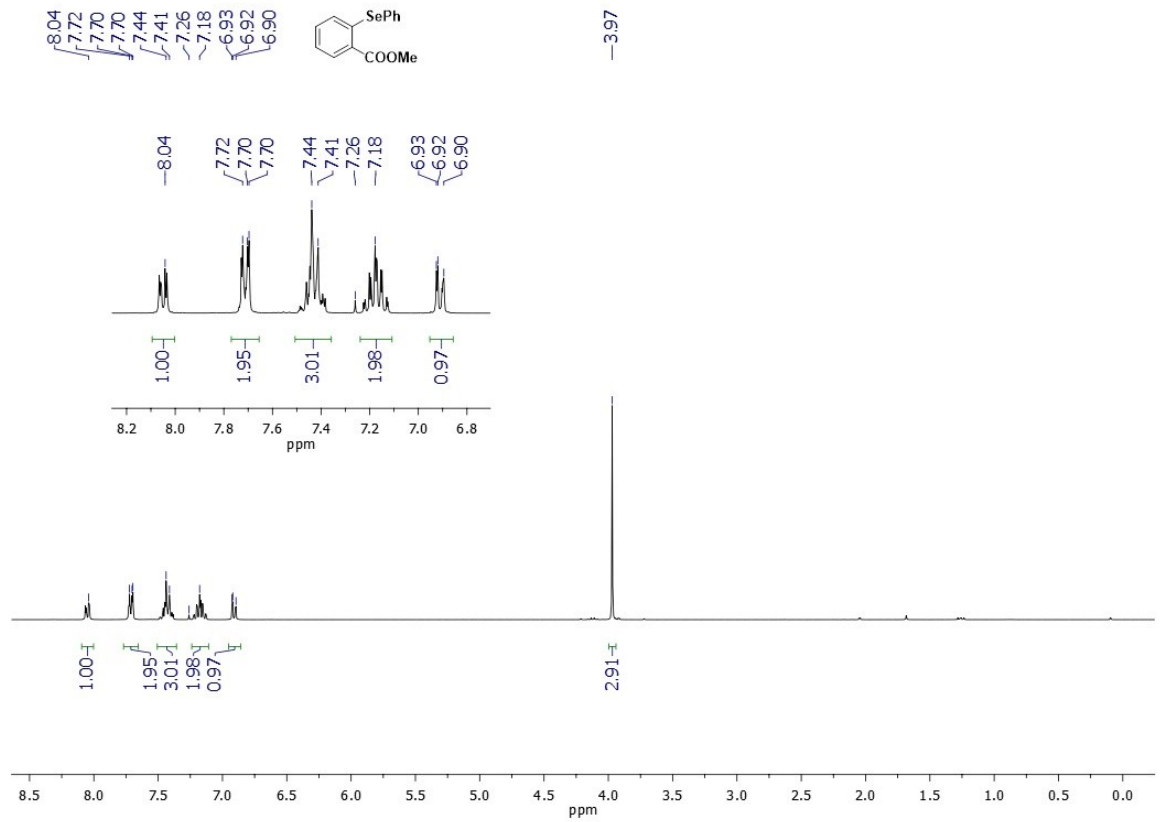
^{1}H-NMR (300 MHz, $CDCl_3$)



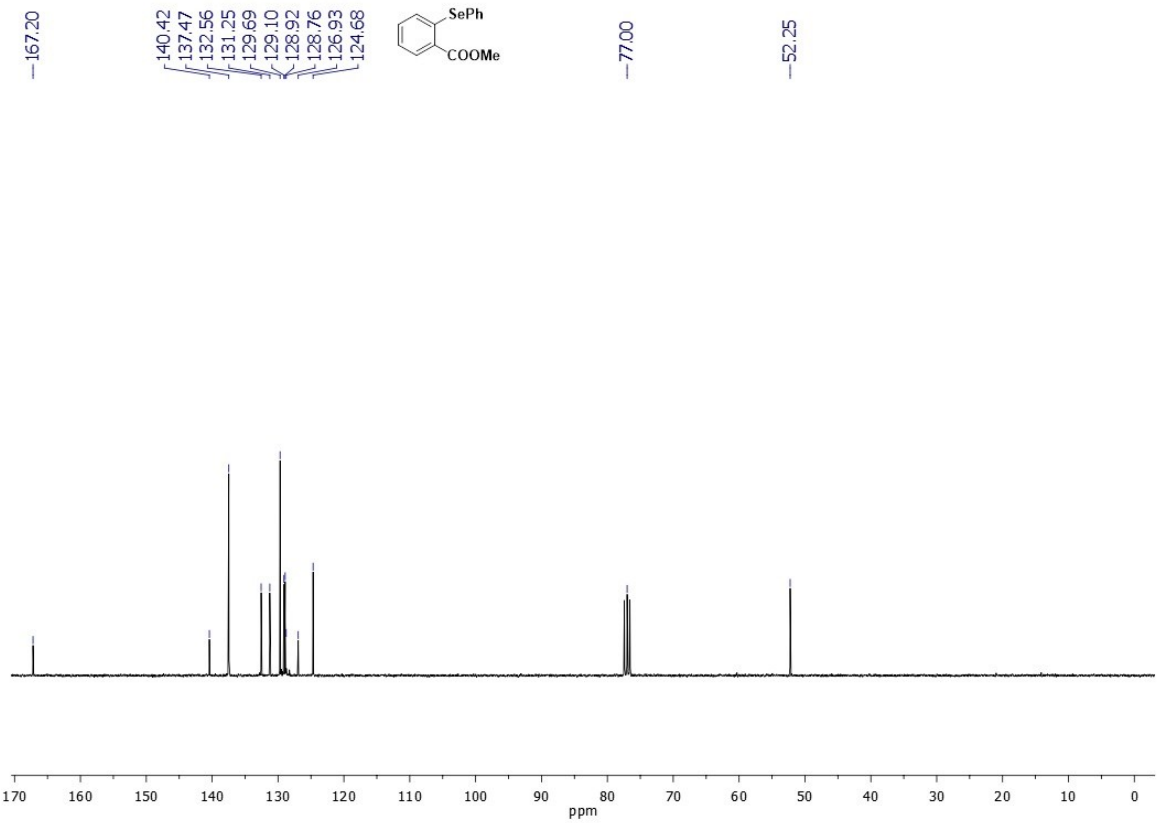
^{13}C-NMR (101 MHz, $CDCl_3$)



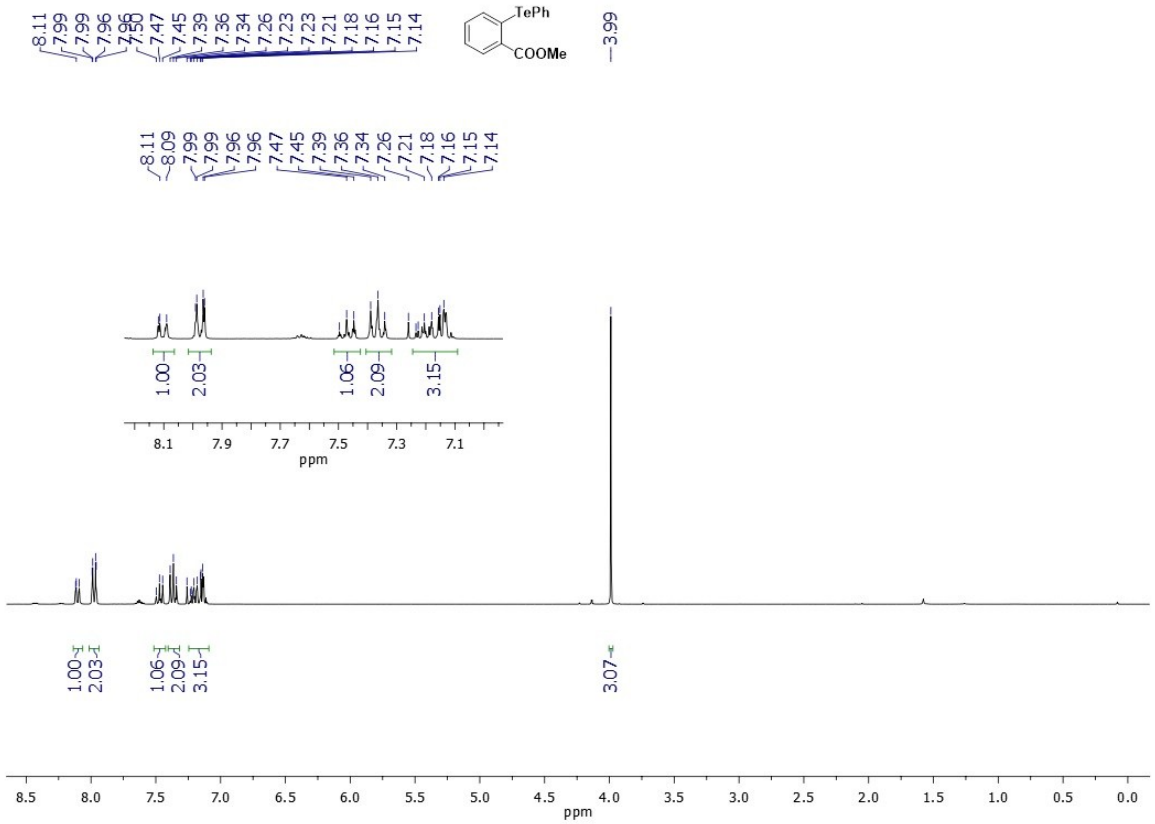
^{1}H-NMR (300 MHz, $CDCl_3$)



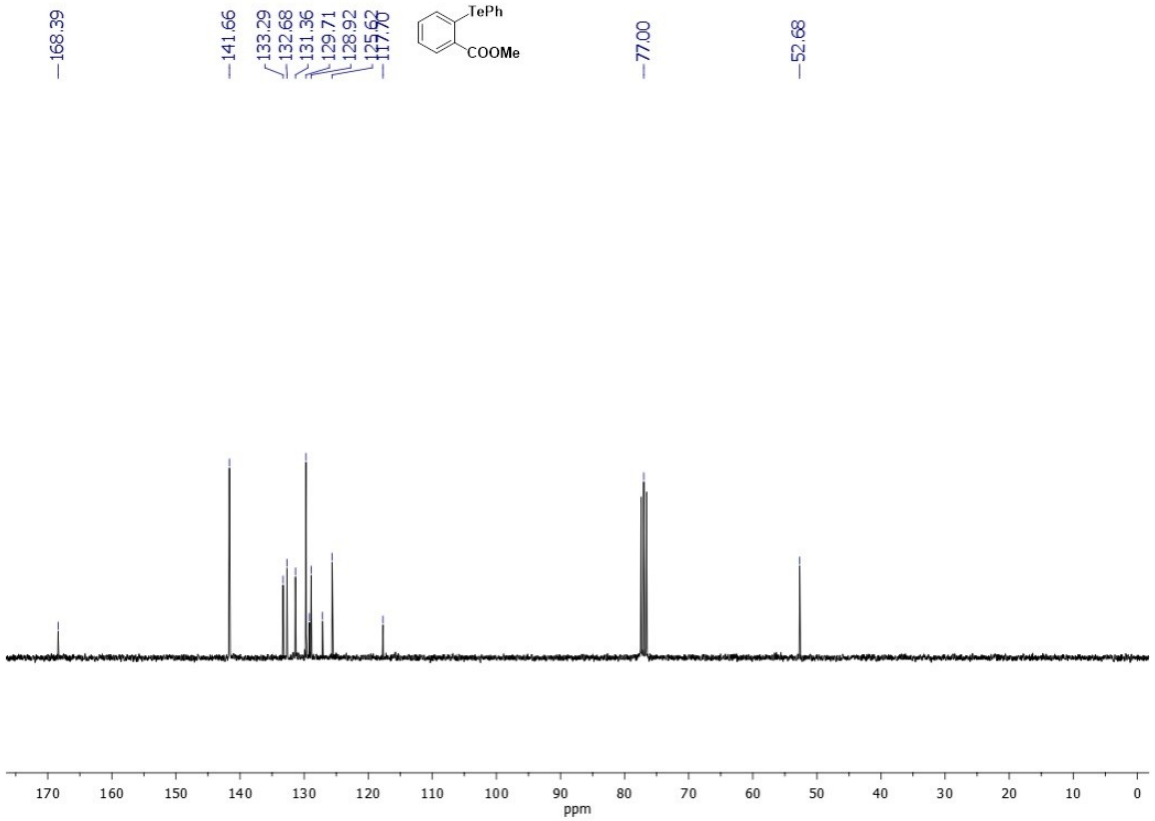
^{13}C-NMR (75 MHz, $CDCl_3$)



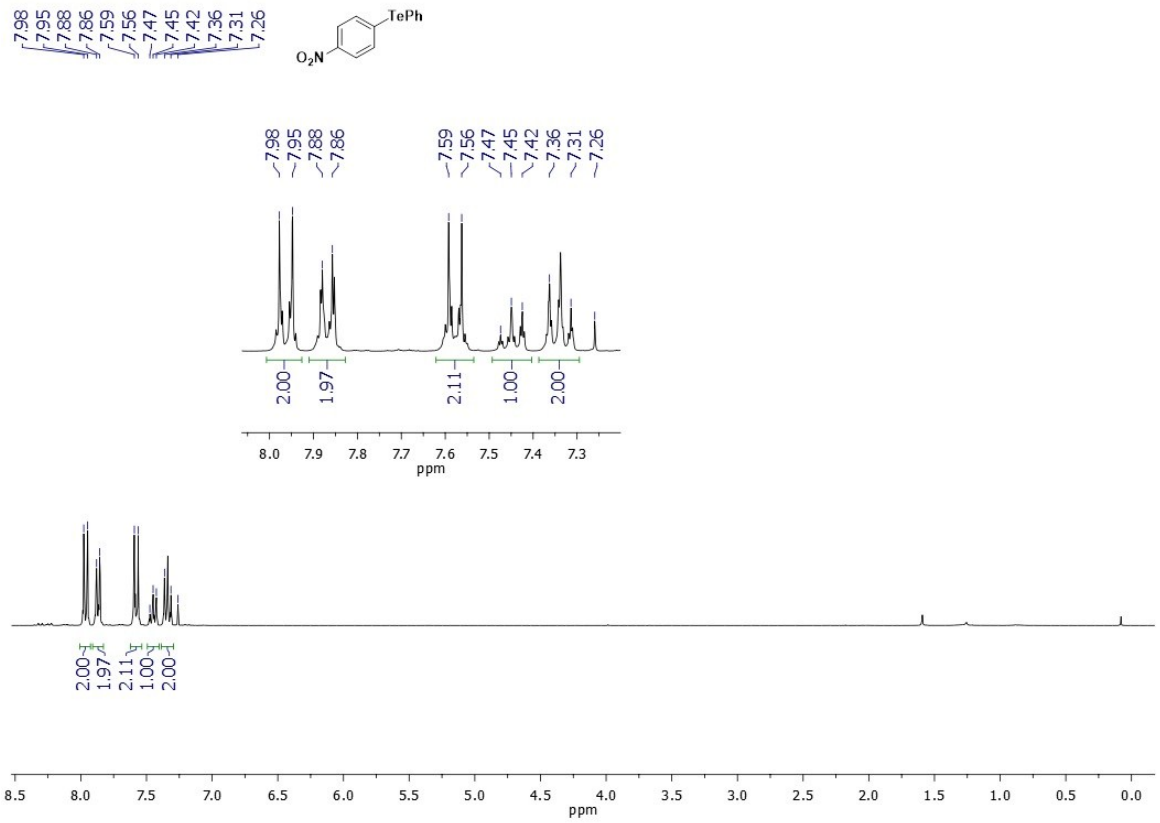
^{1}H-NMR (300 MHz, $CDCl_3$)



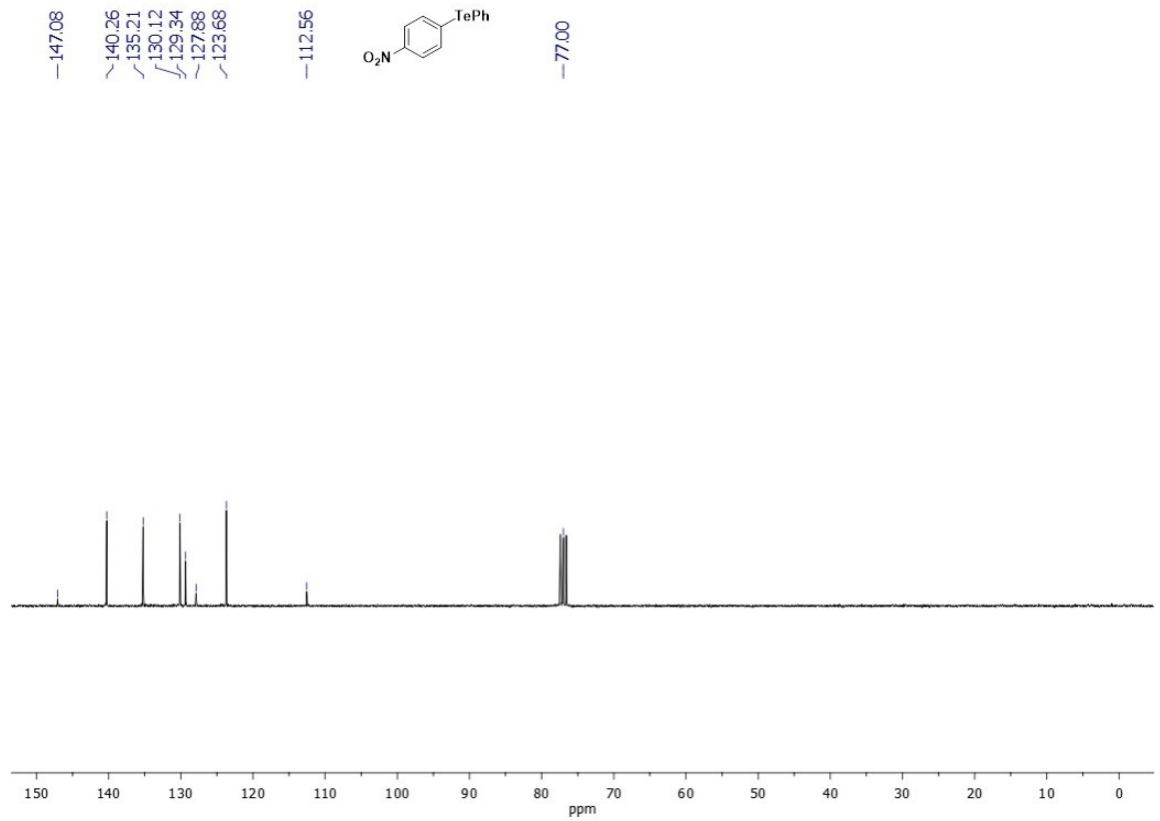
^{13}C-NMR (75 MHz, $CDCl_3$)



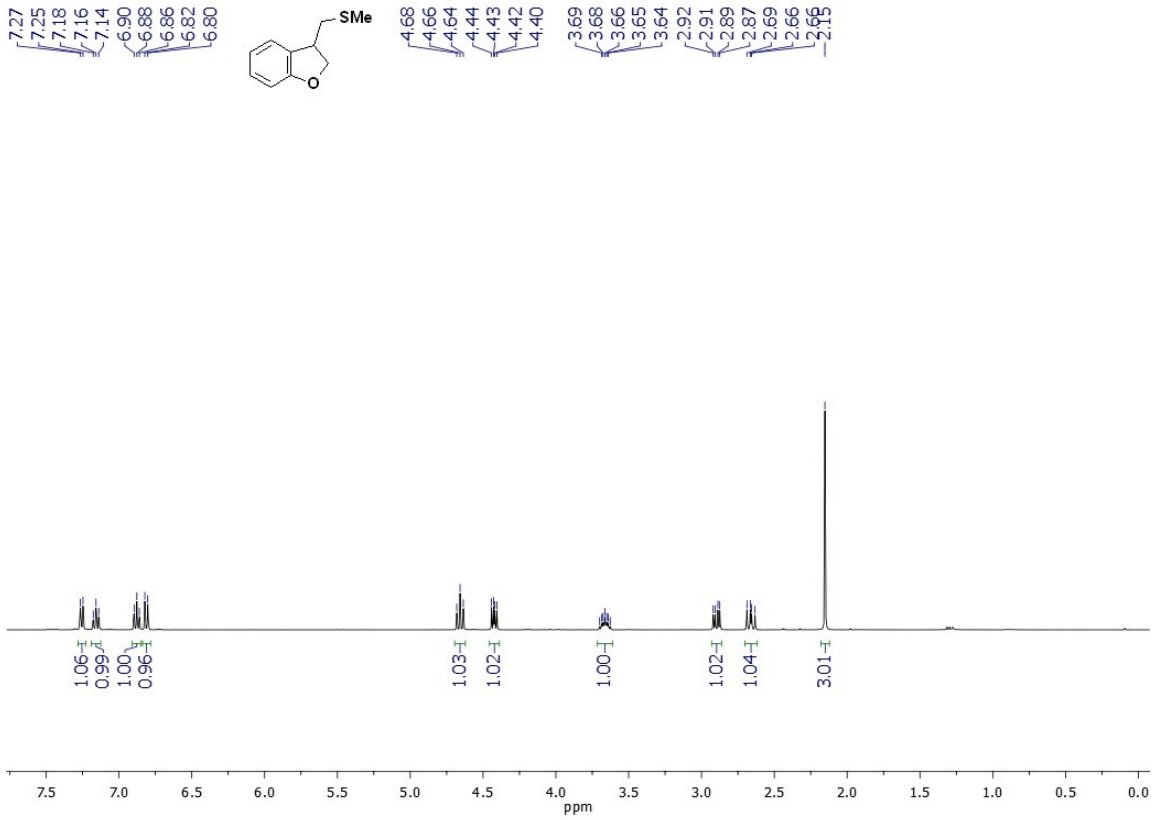
^{1}H-NMR (300 MHz, $CDCl_3$)



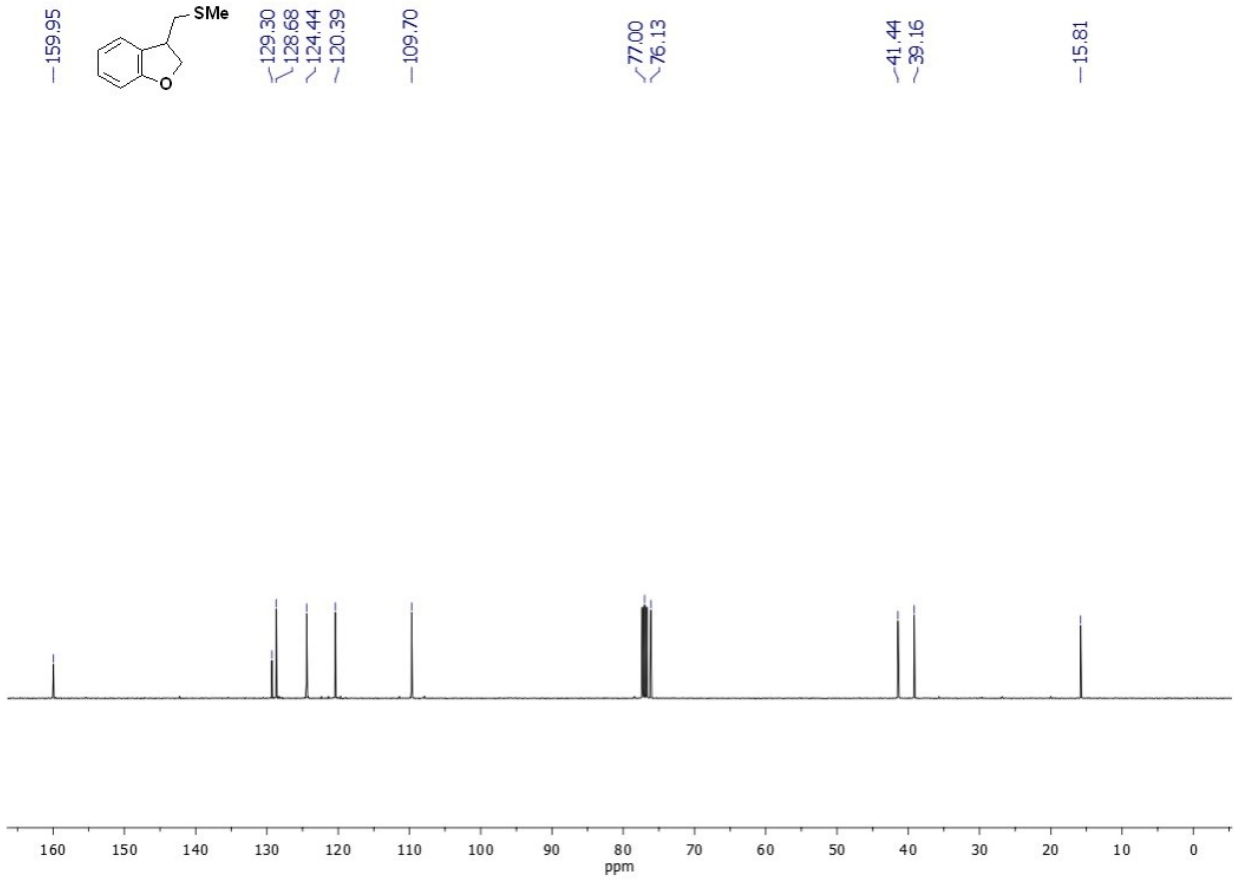
^{13}C-NMR (75 MHz, $CDCl_3$)



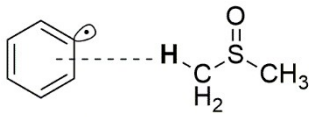
^{1}H-NMR (400 MHz, $CDCl_3$)



^{13}C-NMR (101 MHz, $CDCl_3$)

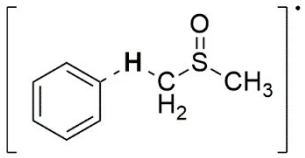

159.95
SMe
O
129.30
128.68
124.44
120.39
109.70
77.00
76.13
41.44
39.16
15.81
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Calculated geometries of the optimized structures and transition states:



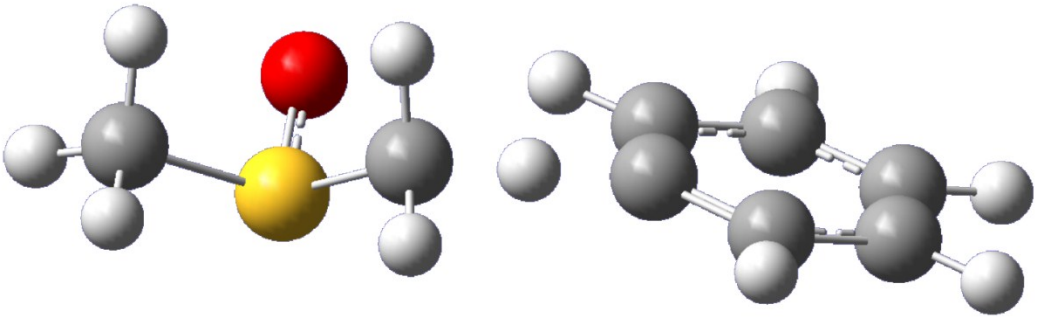
solvated complex

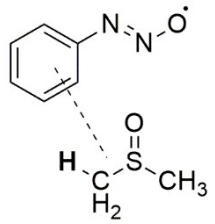
C	-3.75654000	-0.35292600	-0.17780300
C	-3.39646500	0.99452800	-0.24593100
C	-2.05530100	1.37374700	-0.09357500
C	-1.14888500	0.35898900	0.12264900
C	-1.44364500	-0.98519200	0.19797400
C	-2.79071000	-1.33851600	0.04167800
H	-4.79731100	-0.63707500	-0.29706000
H	-4.15411900	1.75379500	-0.41643200
H	-1.75810100	2.41696100	-0.14347800
H	1.17871000	1.62544600	1.07466900
H	-0.66435000	-1.72206400	0.37143000
H	-3.08238300	-2.38372300	0.09150300
C	2.17728200	1.42647200	0.68214700
H	2.49817800	2.24910300	0.04000800
H	2.88418200	1.26552800	1.49923400
S	2.06244100	-0.08797600	-0.29437700
O	1.74965700	-1.20170500	0.70616100
C	3.82056300	-0.25402500	-0.67649400
H	3.94354000	-1.18709500	-1.22722900
H	4.13499500	0.58892300	-1.29527700
H	4.38254600	-0.28700900	0.25950600



transition state

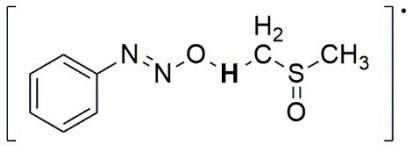
C	-3.72533200	-0.42086800	-0.10887600
C	-3.43064800	0.94387600	-0.13590800
C	-2.10633700	1.37761200	-0.01126800
C	-1.12199800	0.41427600	0.13688100
C	-1.37559600	-0.94768700	0.16739800
C	-2.70606900	-1.36345200	0.04139200
H	-4.75476000	-0.75179000	-0.20499000
H	-4.22790500	1.67184000	-0.25369900
H	-1.86363500	2.43632000	-0.02984800
H	0.18409400	0.82706700	0.27221300
H	-0.56247700	-1.65921500	0.28513400
H	-2.94401200	-2.42309800	0.06108700
C	1.42657200	1.16914300	0.39646000
H	1.52862400	2.13947200	-0.09035200
H	1.63949900	1.16863600	1.46710800
S	2.30201600	-0.12796500	-0.46626900
O	1.95360500	-1.41821300	0.27776500
C	3.97928700	0.30274300	0.05879900
H	4.65304400	-0.44349900	-0.36468700
H	4.22959000	1.29450700	-0.32298700
H	4.01756700	0.27904000	1.14992200





solvated complex

C	-1.90524600	-1.46853900	1.38567200
C	-1.74940300	-0.12792600	1.04812700
C	-1.67297500	0.23012400	-0.30258100
C	-1.76033000	-0.73671200	-1.30359300
C	-1.91583700	-2.07630900	-0.95327300
C	-1.98839400	-2.44488700	0.38919700
H	-1.96403100	-1.75130100	2.43165000
H	-1.69198600	0.63241600	1.82137200
H	-1.70035100	-0.42984300	-2.34294600
H	-1.98067900	-2.82989900	-1.73113700
H	-2.11013600	-3.48815600	0.66122600
N	-1.51623200	1.57213600	-0.76170900
N	-1.30014400	2.42815400	0.09815600
O	-1.12911600	3.61977100	0.03557500
H	0.94576300	1.00043300	1.11986700
C	1.75730500	1.13738100	0.40030200
H	2.37919900	1.98160100	0.69992300
H	1.37087400	1.28666000	-0.61155200
S	2.80213300	-0.33570400	0.41109000
O	3.82479800	-0.13098300	-0.70656700
C	1.57388200	-1.48282800	-0.24643800
H	2.05852900	-2.45385300	-0.35424400
H	0.73791900	-1.55768700	0.45428200
H	1.23593200	-1.11325700	-1.21754800



transtion state

C	2.69438100	-1.72231000	-0.39732800
C	1.69265600	-0.75783900	-0.42283400
C	2.00977400	0.56433400	-0.09041000
C	3.31659600	0.91988600	0.24146300
C	4.31251700	-0.05479300	0.26976600
C	4.00276200	-1.37607900	-0.04791400
H	2.45578200	-2.74865900	-0.65728100
H	0.67905900	-1.02103700	-0.70724800
H	3.53578300	1.95592200	0.47989300
H	5.32823400	0.21992400	0.53476900
H	4.77812700	-2.13540000	-0.03199200
N	1.06277000	1.63584300	-0.08595100
N	-0.11065600	1.25595000	-0.08463700
O	-0.96336400	2.28147500	-0.09327900
H	-2.07099100	1.71392400	-0.08586700
C	-3.15663900	1.04535300	-0.14425200
H	-3.62860400	1.39198200	-1.06401800
H	-3.71548500	1.27970400	0.76421300
S	-2.73461800	-0.69740600	-0.28393600
O	-4.10830000	-1.36921500	-0.34410800
C	-2.14150200	-0.95160500	1.40072200
H	-1.93345400	-2.01746900	1.50191200
H	-1.22537600	-0.37572200	1.54782000
H	-2.92393600	-0.64698700	2.09872400