Supporting Information for

Nickel(II)/TPMPP Catalyzed Reductive Coupling of Oxalate and Tetrasulfides: Synthesis of Unsymmetric Disulfides

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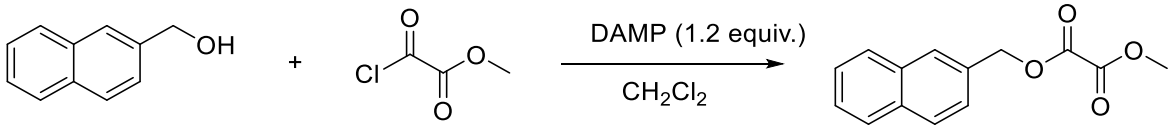
Contents

Ⅰ. General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were analytical grade and used without further purification. Anhydrous DMSO, was purchased from Beijing InnoChem Science & Technology Co., Ltd. Analytical thin-layer chromatography (TLC) was performed on silica gel, visualized by irradiation with UV light. For column chromatography, 300-400 mesh silica gel was used. ^{1}H-NMR and ^{13}C-NMR were recorded on a BRUKER 400 MHz spectrometer in $CDCl_3$. Chemical shifts (δ) were reported referenced to an internal tetramethylsilane standard or the $CDCl_3$ residual peak (δ 7.26) for ^{1}H NMR. Chemical shifts of ^{13}C NMR are reported relative to $CDCl_3$ (δ 77.16). Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), bs (broad singlet), d (doublet), t (triplet), m (multiplet); coupling constants (J) are in Hertz (Hz). IR spectra were recorded on a BRUKER VERTEX 70 spectrophotometer and are reported in terms of frequency of absorption (cm^{-1}). HRMS spectra were obtained by using GCT Premier TOF-MS with EI source. The starting materials were isolated by SepaBean machine Flash Chromatography, which was purchased from Santai Technologies Inc.

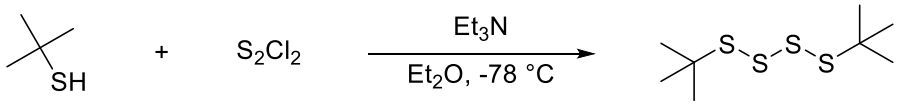
II. Synthesis of Substrates

General procedure for the synthesis of Methyl (naphthalen-2-ylmethyl) oxalate.[1]



To a solution of DMAP (3.0 mmol, 1.2 equiv.) in CH_2Cl_2 (10 mL) was dropwise added methyl chlorooxoacetate (3.0 mmol, 1.2 equiv.) at 0 °C. The reaction mixture was stirred at room temperature for 5 min, and a solution of naphthalen-2-ylmethanol (2.5 mmol) in CH_2Cl_2 (5 mL) was dropwise added. After stirring for 10 min, the reaction was quenched with water (20 mL), extracted twice with CH_2Cl_2 (20 mL). The combine organic layers was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The residue was purified by flash chromatography on silica gel to afford oxalate.

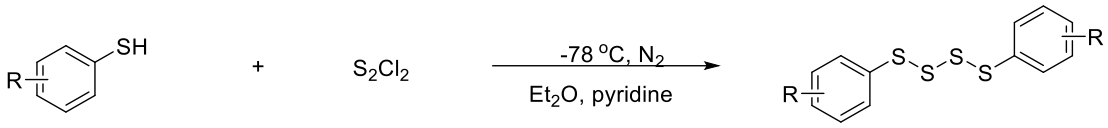
General procedure for the synthesis of 1,4-di-tert-butyltetrasulfane.[2,3]



A solution of S_2Cl_2 (2.21 mL, 27.7 mmol) in dry ether (70 mL) is cooled to -78°C in a dry ice/acetone bath. A solution of tert-butylthiol (6.25 mL, 55.4 mmol)

and Et_3N (7.68 mL, 55.4 mmol) in dry ether (70 mL) is added dropwise over 1 hour. After the addition is complete, the solution is stirred at -78°C for an additional 30 minutes after which is warmed to room temperature and quenched with water. The organic layer was separated and washed with water, Na_2CO_3 (sat.) and brine, dried over $MgSO_4$, filtered and concentrated in vacuo. The crude yellow oil was purified by column chromatography using hexanes as the eluent to yield the product as a yellow oil.

General procedure for the synthesis of 1,4-diphenyltetrasulfane.[2.4]

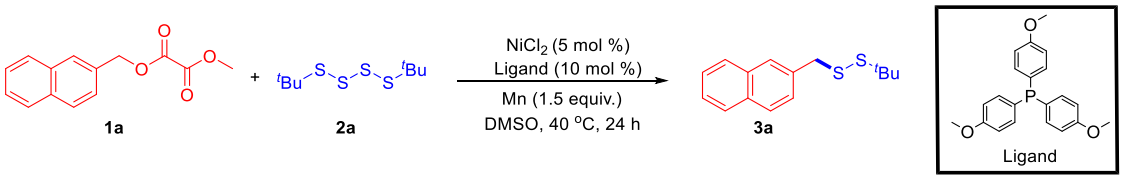


Under N_2 atmosphere, the desired thiol (1.0 equiv.) and pyridine (1.0 equiv.) were dissolved in 30 mL of anhydrous ether and cooled to -78 °C. S_2Cl_2 (0.6 equiv.) was added dropwise. A white precipitate formed within a few seconds and the reaction was continued for 2 hours. Then warm to room temperature. The reaction was quenched by adding 30 mL of water. Extract three times with dichloromethane. The organic phase was dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by silica gel column chromatography to obtain the target product

III. General Procedure and Product Characterization

1. General Procedure A

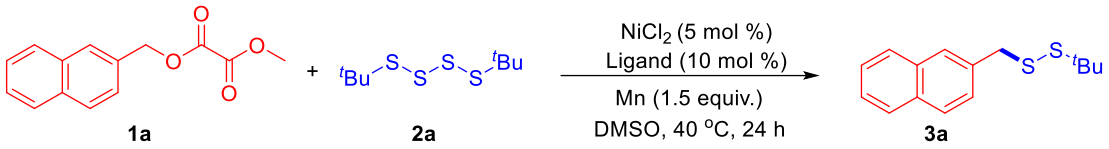
A representative procedure synthesis of 1-(tert-butyl)-2-(naphthalen-2-ylmethyl) disulfane (3a) is shown below.



In glovebox, an oven-dried screw-capped 8-mL vial equipped with a magnetic stir bar was charged with Methyl (naphthalen-2-ylmethyl) oxalate 1a (48.8 mg, 0.2 mmol), 1,4-di-tert-butyltetrasulfane 2a (58.1 mg, 0.24 mmol), $NiCl_2$ (5.0 mol %), Ligand (10 mol %), Mn (1.5 equiv.), DMSO (1 mL) was added via syringe and the mixture was stirred at 40 °C for 24 h. After 24 h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL × 3). The organic layer was dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product **3a** (85% yield).

2. General Procedure B

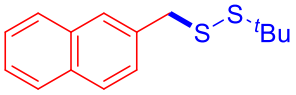
The procedure scale-up synthesis of 3a is shown below.



1a 2a 3a

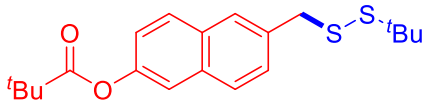
In glovebox, An oven-dried screw-capped 100-mL vial equipped with Methyl (naphthalen-2-ylmethyl) oxalate 1a (732.2 mg, 3 mmol), 1,4-di-tert-butyltetrasulfane 2a (871.3 mg, 3.6 mmol), $NiCl_2$ (5.0 mol %), Ligand (10 mol %), Mn (1.5 equiv.), DMSO (15.0 mL) was added via syringe. The reaction mixture was stirred for 24 h at 40 °C. After 24 h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL × 3). The organic layer was dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product **3a** (64 % yield, 0.503 g).

IV. Product Characterization



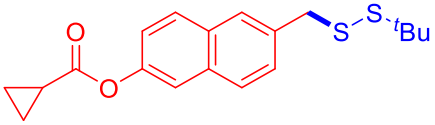
1-(tert-butyl)-2-(naphthalen-2-ylmethyl)disulfane (3a)

Yield: 85% (89.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 2957, 2920, 2853, 2369, 1732, 1507, 1361, 1271, 1162, 817, 750, 468. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 3H), 7.77 – 7.75 (m, 1H), 7.52 – 7.47 (m, 3H), 4.13 (s, 2H), 1.39 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 134.8, 133.3, 132.7, 128.4, 127.9, 127.8, 127.7, 127.3, 126.2, 125.9, 48.2, 46.1, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{15}H_{18}S_2$: 262.0850, found 262.0855.



6-((tert-butyldisulfaneyl)methyl)naphthalen-2-yl pivalate (3b)

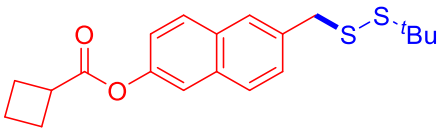
Yield: 68% (98.5 mg). White solid. **IR** (neat, ν, cm^{-1}): 2968, 2898, 2357, 1750, 1474, 1362, 1148, 1129, 907, 823, 480. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, J = 8.9 Hz, 1H), 7.73 (d, J = 8.4 Hz, 1H), 7.69 (d, J = 1.6 Hz, 1H), 7.49 (d, J = 2.3 Hz, 1H), 7.44 (dd, J = 8.4, 1.7 Hz, 1H), 7.17 (dd, J = 8.8, 2.3 Hz, 1H), 4.06 (s, 2H), 1.39 (s, 9H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.3, 148.9, 134.7, 133.1, 131.3, 129.2, 128.1, 128.1, 127.9, 121.6, 118.4, 48.2, 45.9, 39.2, 30.2, 27.3. **HRMS** (CI) m/z (M^+) calcd for $C_{20}H_{26}O_2S_2$: 362.1374, found 362.1376.



6-((tert-butyldisulfaneyl)methyl)naphthalen-2-yl cyclopropanecarboxylate (3c)

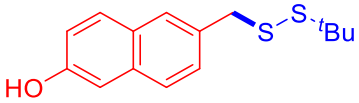
Yield: 55% (76.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 2917, 2853, 1730, 1374, 1212,

1147, 893, 479. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.9 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.69 (s, 1H), 7.52 (d, *J* = 2.3 Hz, 1H), 7.44 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.22 (dd, *J* = 8.7, 2.4 Hz, 1H), 4.05 (s, 2H), 1.87 (dq, *J* = 8.3, 4.6, 4.1 Hz, 1H), 1.33 (s, 9H), 1.18 (dt, *J* = 6.8, 3.5 Hz, 2H), 1.02 (dq, *J* = 7.6, 4.1 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.7, 148.6, 134.7, 133.1, 131.3, 129.2, 128.2, 128.1, 127.8, 121.6, 118.5, 48.2, 45.9, 30.2, 13.2, 9.4. **HRMS** (CI) m/z (M^+) calcd for $C_{19}H_{22}O_2S_2$: 346.1061, found 346.1059.



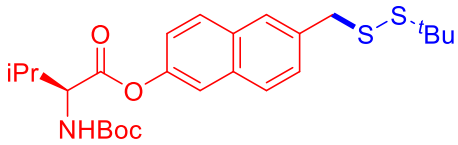
6-((tert-butyldisulfaneyl)methyl)naphthalen-2-yl cyclobutanecarboxylate (3d)

Yield: 25% (36.0 mg). White solid. **IR** (neat, ν, cm^{-1}): 2956, 2921, 2856, 1753, 1360, 1205, 1130, 1133, 896, 824, 478. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.8 Hz, 1H), 7.79 (d, *J* = 8.5 Hz, 1H), 7.75 (d, *J* = 1.7 Hz, 1H), 7.56 (d, *J* = 2.3 Hz, 1H), 7.49 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.25 (dd, *J* = 8.8, 2.3 Hz, 1H), 4.11 (s, 2H), 3.47 (pd, *J* = 8.5, 1.1 Hz, 1H), 2.59 – 2.46 (m, 2H), 2.39 (tdd, *J* = 10.3, 5.3, 2.2 Hz, 2H), 2.20 – 2.00 (m, 2H), 1.38 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.2, 148.7, 134.8, 133.1, 131.4, 129.3, 128.2, 128.1, 127.9, 121.6, 118.5, 48.3, 46.0, 38.3, 30.2, 25.5, 18.6. **HRMS** (CI) m/z (M^+) calcd for $C_{20}H_{24}O_2S_2$: 360.1218, found 360.1223.



6-((tert-butyldisulfaneyl)methyl)naphthalen-2-ol (3d')

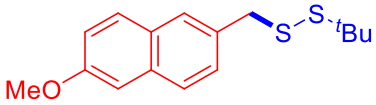
Yield: 40% (44.5 mg). White solid. **IR** (neat, ν, cm^{-1}): 3215, 2957, 2359, 1508, 1361, 1205, 1158, 864, 808, 470. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.7 Hz, 1H), 7.67 – 7.60 (m, 2H), 7.39 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.12 – 7.05 (m, 2H), 5.33 (s, 1H), 4.06 (s, 2H), 1.35 (s, 9H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.7, 134.0, 132.5, 129.8, 128.9, 128.1, 128.0, 127.0, 118.2, 109.6, 48.3, 46.1, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{15}H_{18}OS_2$: 278.0799, found 278.0795.



6-((tert-butyldisulfaneyl)methyl)naphthalen-2-yl (tert-butoxycarbonyl)-L-valinate (3e)

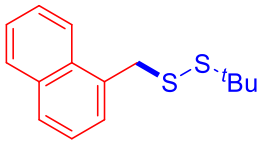
Yield: 30% (57.3 mg). White solid. **IR** (neat, ν, cm^{-1}): 3443, 2966, 1747, 1713, 1360, 1154, 1074, 482. **^{1}H NMR** (400 MHz, $CDCl_3$)) δ 7.82 (d, *J* = 8.9 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.72 (s, 1H), 7.53 (d, *J* = 2.2 Hz, 1H), 7.47 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.22 (dd, *J* = 8.9, 2.3 Hz, 1H), 5.13 (d, *J* = 9.1 Hz, 1H), 4.52 (dd, *J* = 9.1, 4.8 Hz, 1H), 4.07 (s, 2H), 2.50 – 2.25 (m, 1H), 1.48 (s, 9H), 1.37 – 1.30 (m, 9H), 1.12 (d, *J* = 6.8

Hz, 3H), 1.07 (d, J = 6.9 Hz, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.4, 155.9, 148.3, 135.1, 133.1, 131.5, 129.5, 128.2, 127.9, 121.3, 118.4, 80.2, 58.9, 48.3, 45.9, 31.5, 30.2, 28.5, 19.3, 17.9. **HRMS** (CI) m/z (M^+) calcd for $C_{25}H_{35}NO_4S_2$: 477.2007, found 477.2005.



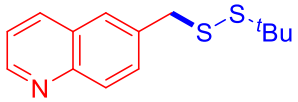
1-(tert-butyl)-2-((6-methoxynaphthalen-2-yl)methyl)disulfane (3f)

Yield: 86% (99.8 mg). White solid. **IR** (neat, ν, cm^{-1}): 2959, 2892, 2359, 1389, 1360, 1265, 1211, 1158, 1026, 857, 723, 472. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 (d, J = 8.7 Hz, 2H), 7.53 (d, J = 1.7 Hz, 1H), 7.30 (dd, J = 8.4, 1.8 Hz, 1H), 7.02 (dd, J = 8.9, 2.6 Hz, 1H), 6.98 (d, J = 2.6 Hz, 1H), 3.95 (s, 2H), 3.76 (s, 3H), 1.25 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.8, 133.9, 132.4, 129.3, 128.8, 127.9, 127.9, 127.3, 119.0, 105.8, 55.3, 48.1, 46.1, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{16}H_{20}OS_2$: 290.0956, found 292.0963.



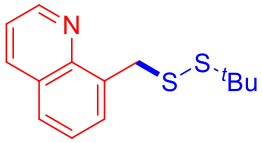
1-(tert-butyl)-2-(naphthalen-1-ylmethyl)disulfane (3g)

Yield: 67% (70.2 mg). White solid. **IR** (neat, ν, cm^{-1}): 3045, 2957, 2920, 2855, 2359, 1509, 1361, 1162, 774, 528, 413. **^{1}H NMR** 1H NMR (400 MHz, $CDCl_3$) δ 8.11 (dq, J = 8.5, 0.9 Hz, 1H), 7.83 (dd, J = 8.2, 1.4 Hz, 1H), 7.76 (dd, J = 7.3, 2.1 Hz, 1H), 7.54 (ddd, J = 8.4, 6.8, 1.5 Hz, 1H), 7.47 (ddd, J = 8.0, 6.8, 1.3 Hz, 1H), 7.42 – 7.36 (m, 2H), 4.39 (s, 2H), 1.34 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 134.1, 133.2, 131.5, 128.9, 128.6, 128.0, 126.4, 126.0, 125.4, 124.2, 48.3, 44.2, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{15}H_{18}S_2$: 262.0850, found 262.0855.



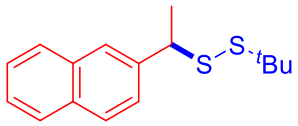
6-((tert-butyldisulfaneyl)methyl)quinoline (3h)

Yield: 70% (73.7 mg). White solid. **IR** (neat, ν, cm^{-1}): 2958, 2355, 1498, 1361, 1160, 892, 845, 778, 471. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.89 (dd, J = 4.3, 1.7 Hz, 1H), 8.12 (dt, J = 8.3, 1.2 Hz, 1H), 8.10 – 8.02 (m, 1H), 7.69 (d, J = 7.4 Hz, 2H), 7.39 (dd, J = 8.3, 4.2 Hz, 1H), 4.10 (s, 2H), 1.35 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.5, 147.8, 136.0, 136.0, 131.1, 130.0, 128.2, 127.8, 121.5, 48.3, 45.6, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{14}H_{17}NS_2$: 263.0802, found 263.0802.



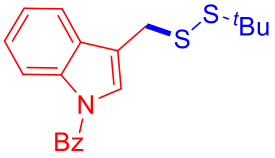
8-((tert-butyldisulfaneyl)methyl)quinoline (3i)

Yield: 79% (83.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 3003, 2360, 1709, 1359, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.93 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.09 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.74 – 7.65 (m, 2H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.36 (dd, *J* = 8.2, 4.2 Hz, 1H), 4.62 (s, 2H), 1.35 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.8, 146.2, 136.4, 136.3, 130.0, 128.5, 127.6, 126.1, 121.3, 48.1, 41.8, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{14}H_{17}NS_2$: 263.0802, found 263.0807.



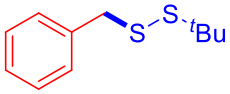
1-(tert-butyl)-2-(1-(naphthalen-2-yl)ethyl)disulfane (3j)

Yield: 16% (17.7 mg). Yellow solid. **IR** (neat, ν, cm^{-1}): 3029, 2960, 2921, 2855, 2361, 1455, 1361, 1164, 824, 746, 476. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (dt, *J* = 7.0, 2.8 Hz, 3H), 7.74 – 7.72 (m, 1H), 7.50 – 7.44 (m, 3H), 4.18 (q, *J* = 7.0 Hz, 1H), 1.76 (d, *J* = 7.0 Hz, 3H), 1.32 (s, 9H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.9, 133.4, 133.0, 128.5, 128.0, 127.8, 126.5, 126.3, 126.0, 125.9, 51.5, 48.1, 30.3, 21.4. **HRMS** (CI) m/z (M^+) calcd for $C_{16}H_{20}S_2$: 276.1006, found 276.1010.



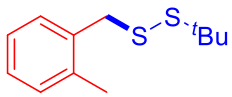
(3-((tert-butyldisulfaneyl)methyl)-1H-indol-1-yl)(phenyl)methanone (3k)

Yield: 76% (108.0 mg). White solid. **IR** (neat, ν, cm^{-1}): 3442, 2965, 2359, 1747, 1713, 1359, 1153, 1073, 890, 482. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.9 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.72 (s, 1H), 7.53 (d, *J* = 2.2 Hz, 1H), 7.47 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.22 (dd, *J* = 8.9, 2.3 Hz, 1H), 5.13 (d, *J* = 9.1 Hz, 1H), 4.52 (dd, *J* = 9.1, 4.8 Hz, 1H), 4.07 (s, 2H), 2.50 – 2.25 (m, 1H), 1.48 (s, 9H), 1.37 – 1.30 (m, 9H), 1.12 (d, *J* = 6.8 Hz, 3H), 1.07 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.4, 155.9, 148.3, 135.1, 133.1, 131.5, 129.5, 128.2, 127.9, 121.3, 118.4, 80.2, 58.9, 48.3, 45.9, 31.5, 30.2, 28.5, 19.3, 17.9. **HRMS** (CI) m/z (M^+) calcd for $C_{20}H_{21}NOS_2$: 355.1065, found 355.1058.



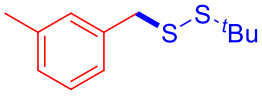
1-benzyl-2-(tert-butyl)disulfane (5a)

Yield: 53% (45.0 mg). White solid. **IR** (neat, ν, cm^{-1}): 2960, 2360, 1708, 1361, 1220, 1165, 763, 696, 529, 472. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 3.3 Hz, 4H), 7.28 – 7.23 (m, 1H), 3.93 (s, 2H), 1.34 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.5, 129.3, 128.7, 127.5, 48.2, 45.9, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{16}S_2$: 212.0693, found 212.0697.



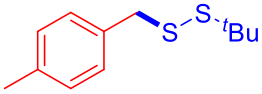
1-(tert-butyl)-2-(2-methylbenzyl)disulfane (5b)

Yield: 64% (57.9 mg). Pale yellow solid. **IR** (neat, ν, cm^{-1}): 3401, 2960, 1709, 1456, 1361, 1219, 1165, 763, 727, 529, 443. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 – 7.19 (m, 1H), 7.18 – 7.11 (m, 3H), 3.96 (s, 2H), 2.41 (s, 3H), 1.34 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 136.9, 135.3, 130.6, 130.5, 127.9, 126.1, 48.1, 44.2, 30.2, 19.5. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{18}S_2$: 226.0850, found 226.0854.



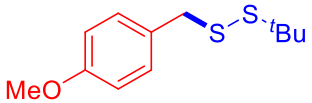
1-(tert-butyl)-2-(3-methylbenzyl)disulfane (5c)

Yield: 62% (56.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 3393, 2959, 2920, 1608, 1455, 1361, 1218, 1164, 880, 786, 718, 702, 434. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.4 Hz, 1H), 7.13 – 7.02 (m, 3H), 3.91 (s, 2H), 2.34 (s, 3H), 1.37 – 1.31 (m, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.3, 137.3, 130.0, 128.5, 128.3, 126.4, 48.2, 46.0, 30.2, 21.5. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{18}S_2$: 226.0850, found 226.0854.



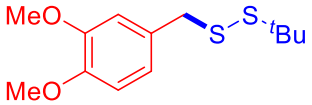
1-(tert-butyl)-2-(4-methylbenzyl)disulfane (5d)

Yield: 66% (59.7 mg). Pale yellow solid. **IR** (neat, ν, cm^{-1}): 2957, 2921, 2855, 1510, 1455, 1360, 1163, 822, 728, 510, 472. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19 (d, J = 7.8 Hz, 2H), 7.11 (d, J = 7.8 Hz, 2H), 3.90 (s, 2H), 2.32 (s, 3H), 1.34 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.2, 134.4, 129.3, 129.2, 48.1, 45.6, 30.2, 21.3. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{18}S_2$: 226.0850, found 226.0853.



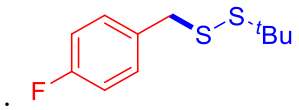
1-(tert-butyl)-2-(4-methoxybenzyl)disulfane (5e)

Yield: 75% (72.6 mg). White solid. **IR** (neat, ν, cm^{-1}): 2958, 2834, 1608, 1509, 1455, 1360, 1302, 1246, 1162, 1030, 820, 518. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (d, J = 8.5 Hz, 2H), 6.84 (d, J = 8.5 Hz, 2H), 3.90 (s, 2H), 3.78 (d, J = 0.9 Hz, 3H), 1.34 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.1, 130.5, 129.5, 114.1, 55.4, 48.1, 45.3, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{18}OS_2$: 242.0799, found 242.0804.



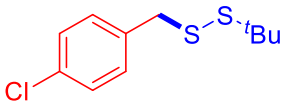
1-(tert-butyl)-2-(3,4-dimethoxybenzyl)disulfane (5f)

Yield: 75% (81.6 mg). Pale yellow solid. **IR** (neat, ν, cm^{-1}): 2959, 2898, 2360, 1508, 1456, 1257, 1140, 1022, 812, 550. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.87 – 6.78 (m, 3H), 3.90 (s, 2H), 3.89 (s, 3H), 3.86 (s, 3H), 1.35 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.9, 148.4, 129.8, 121.5, 112.2, 111.0, 55.9, 55.9, 48.1, 45.8, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{13}H_{20}O_2S_2$: 272.0905, found 272.0908.



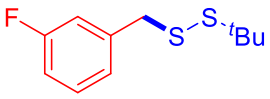
1-(tert-butyl)-2-(4-fluorobenzyl)disulfane (5g)

Yield: 47% (43.3 mg). White solid. **IR** (neat, ν, cm^{-1}): 3413, 2963, 1710, 1509, 1361, 1220, 1157, 837, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.23 (m, 2H), 7.00 (t, J = 8.7 Hz, 2H), 3.89 (s, 2H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.2 (J = 244.5), 133.3 (J = 3.3), 130.9 (J = 8.0), 115.5 (J = 21.4), 48.2, 44.8, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{15}FS_2$: 230.0599, found 230.0598.



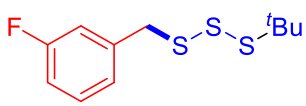
1-(tert-butyl)-2-(4-chlorobenzyl)disulfane (5h)

Yield: 80% (78.7 mg). White solid. **IR** (neat, ν, cm^{-1}): 2959, 2920, 2859, 2359, 1489, 1455, 1361, 1164, 1092, 1015, 831, 806, 497. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 (d, J = 8.5 Hz, 2H), 7.22 (d, J = 8.4 Hz, 2H), 3.87 (s, 2H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 136.4, 133.6, 131.0, 129.1, 48.6, 45.1, 30.4. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{15}ClS_2$: 246.0304, found 246.0303.



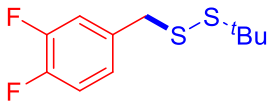
1-(tert-butyl)-2-(3-fluorobenzyl)disulfane (5i)

Yield: 49% (45.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 3003, 2359, 1710, 1359, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 (td, J = 8.0, 4.0 Hz, 1H), 7.07 (d, J = 7.8 Hz, 1H), 7.02 (dt, J = 9.5, 2.1 Hz, 1H), 6.95 (td, J = 8.5, 2.7 Hz, 1H), 3.89 (s, 2H), 1.38 (s, 1H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.9 (J = 248.0), 140.1 (J = 7.3), 130.1 (J = 8.2), 125.0 (J = 2.9), 116.2 (J = 21.3), 114.4 (J = 20.9), 49.1, 48.3, 45.2, 45.2, 42.7, 42.7, 30.1, 30.0. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{15}FS_2$: 230.0599, found 230.0602.



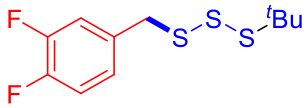
1-(tert-butyl)-3-(3-fluorobenzyl)trisulfane (5i')

Yield: 5% (5.2 mg). **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{15}FS_3$: 262.0320, found 262.0322.



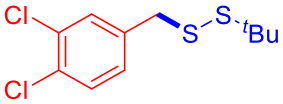
1-(tert-butyl)-2-(3,4-difluorobenzyl)disulfan(5j)

Yield: 74% (73.4 mg). White solid. **IR** (neat, ν, cm^{-1}): 3003, 2967, 2359, 1710, 1360, 1220, 778, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.17 – 7.05 (m, 2H), 7.00 (ddt, *J* = 8.1, 3.7, 1.6 Hz, 1H), 3.84 (s, 2H), 1.37 (s, 1H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.5 (*J* = 12.7), 151.0 (*J* = 12.5), 149.0 (*J* = 12.8), 148.6 (*J* = 12.4), 134.7 (*J* = 3.9), 125.3 (*J* = 3.6), 118.2 (*J* = 17.2), 117.3 (*J* = 17.1), 49.2, 48.3, 44.5, 44.5, 42.1, 30.1, 30.0. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{14}F_2S_2$: 248.0505, found 248.0509.



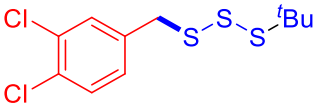
1-(tert-butyl)-3-(3,4-difluorobenzyl)trisulfane (5j')

Yield: 5% (5.6 mg). **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{14}F_2S_3$: 280.0226, found 280.0229.



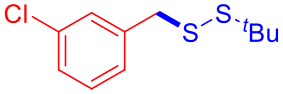
1-(tert-butyl)-2-(3,4-dichlorobenzyl)disulfane (5k)

Yield: 81% (90.7 mg). White solid. **IR** (neat, ν, cm^{-1}): 2963, 2359, 1709, 1360, 1220, 900, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 2H), 7.13 (dd, *J* = 8.2, 2.1 Hz, 1H), 3.82 (s, 2H), 1.37 (s, 1H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.9, 132.5, 131.5, 131.1, 130.5, 128.7, 48.4, 44.2, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{14}Cl_2S_2$: 279.9914, found 279.9916.



1-(tert-butyl)-3-(3,4-dichlorobenzyl)trisulfane (5k')

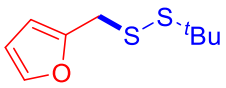
Yield: 5% (6.2 mg). **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{14}Cl_2S_3$: 311.9635, found 311.9645.



1-(tert-butyl)-2-(3-chlorobenzyl)disulfane (5l)

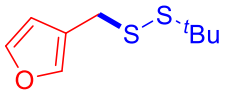
Yield: 72% (70.9 mg). White solid. **IR** (neat, ν, cm^{-1}): 2958, 2916, 2360, 1571, 1473, 1455, 1360, 1159, 1075, 884, 859, 792, 718, 689, 675, 433. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 16.29 (d, *J* = 2.1 Hz, 1H), 16.25 – 16.21 (m, 2H), 16.17 (dt, *J* = 5.4, 1.6 Hz, 1H), 12.86 (s, 2H), 10.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.6, 134.3, 129.9,

129.4, 127.6, 127.5, 48.3, 45.0, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{11}H_{15}ClS_2$: 246.0304, found 246.0308.



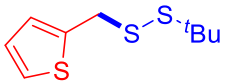
2-((tert-butyldisulfaneyl)methyl)furan (5m)

Yield: 82% (66.3 mg). Pale yellow solid. **IR** (neat, ν, cm^{-1}): 2960, 2922, 1772, 1456, 1362, 1162, 1069, 1020, 872, 785, 729, 599. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 6.42 – 6.38 (m, 1H), 3.77 (s, 2H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.4, 140.7, 121.2, 111.1, 48.1, 35.6, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_9H_{14}OS_2$: 202.0486, found 202.0490.



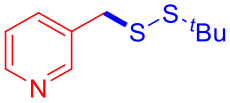
3-((tert-butyldisulfaneyl)methyl)furan (5n)

Yield: 72% (58.2 mg). White solid. **IR** (neat, ν, cm^{-1}): 2965, 1710, 1359, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 (dd, J = 1.9, 0.9 Hz, 1H), 6.30 (dd, J = 3.3, 1.9 Hz, 1H), 6.23 (dd, J = 3.2, 0.8 Hz, 1H), 3.99 – 3.90 (m, 2H), 1.32 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.8, 142.5, 110.7, 108.6, 48.2, 37.9, 30.0. **HRMS** (CI) m/z (M^+) calcd for $C_9H_{14}OS_2$: 202.0486, found 202.0490.



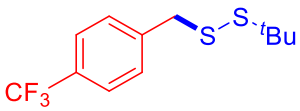
2-((tert-butyldisulfaneyl)methyl)thiophene (5o)

Yield: 82% (71.5 mg). White solid. **IR** (neat, ν, cm^{-1}): 2950, 2920, 2859, 1455, 1361, 1221, 1164, 850, 697, 480. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (dd, J = 5.0, 1.3 Hz, 1H), 7.00 – 6.88 (m, 2H), 4.14 (s, 2H), 1.35 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.0, 127.0, 126.9, 125.4, 48.4, 40.0, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_9H_{14}S_3$: 218.0258, found 218.0263.



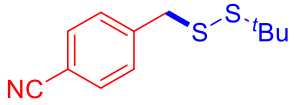
3-((tert-butyldisulfaneyl)methyl)pyridine (5p)

Yield: 81% (69.0 mg). White solid. **IR** (neat, ν, cm^{-1}): 2965, 1709, 1360, 1220, 714, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.50 (dd, J = 8.6, 3.6 Hz, 2H), 7.63 (d, J = 7.9 Hz, 1H), 7.29 – 7.17 (m, 1H), 3.86 (s, 2H), 1.31 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.2, 148.6, 136.7, 133.3, 123.5, 48.2, 42.0, 30.0. **HRMS** (CI) m/z (M^+) calcd for $C_{10}H_{15}NS_2$: 213.0646, found 213.0652.



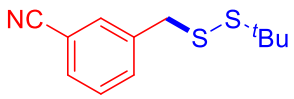
1-(tert-butyl)-2-(4-(trifluoromethyl)benzyl)disulfane (5q)

Yield: 67% (75.1 mg). White solid. **IR** (neat, ν, cm^{-1}):3378, 2963, 1617, 1417, 1363, 1321, 1162, 1120, 1065, 1019, 843, 618, 499. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.1 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 3.93 (s, 2H), 1.34 (s, 9H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.8 (*J* = 1.6), 129.6, 129.6 (*J* = 32.3), 128.3, 125.6 (*J* = 3.6), 122.9, 120.2, 48.4, 44.9, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{15}F_3S_2$: 280.0567, found 280.0571.



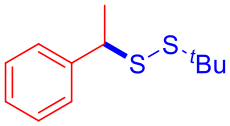
4-((tert-butyldisulfaneyl)methyl)benzonitrile (5r)

Yield: 83% (78.7 mg). White solid. **IR** (neat, ν, cm^{-1}): 2961, 2360, 2229, 1710, 1361, 1220, 1165, 552, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.2 Hz, 2H), 3.91 (s, 2H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.2, 132.3, 130.0, 118.8, 111.2, 48.4, 44.8, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{15}NS_2$: 237.0646, found 237.0651.



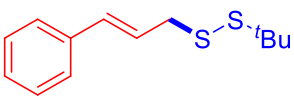
3-((tert-butyldisulfaneyl)methyl)benzonitrile (5s)

Yield: 88% (83.4 mg). White solid. **IR** (neat, ν, cm^{-1}): 2964, 2230, 1709, 1419, 1360, 1220, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 1.8 Hz, 1H), 7.57 – 7.52 (m, 2H), 7.43 (t, *J* = 7.7 Hz, 1H), 3.89 (s, 2H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.4, 133.7, 132.8, 131.1, 129.4, 118.7, 112.6, 48.4, 44.3, 30.1. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{15}NS_2$: 237.0646, found 237.0652.



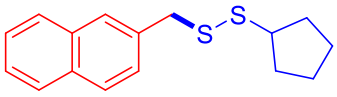
(R)-1-(tert-butyl)-2-(1-phenylethyl)disulfane (5t)

Yield: 20% (18.1 mg). White solid. **IR** (neat, ν, cm^{-1}): 2923, 2359, 1708, 1360, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 4.3 Hz, 4H), 7.26 (q, *J* = 5.0, 4.6 Hz, 1H), 4.01 (q, *J* = 7.0 Hz, 1H), 1.67 (d, *J* = 7.0 Hz, 3H), 1.31 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.5, 128.6, 127.8, 127.6, 51.3, 48.0, 30.2, 21.3. **HRMS** (CI) m/z (M^+) calcd for $C_{12}H_{18}S_2$: 226.0850, found 226.0855.



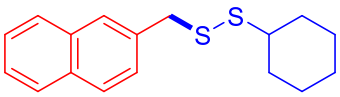
1-(tert-butyl)-2-cinnamyldisulfane (5v)

Yield: 64% (61.0 mg). Pale yellow liquid. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1419, 1359, 1219, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 2H), 7.37 – 7.33 (m, 2H), 7.30 – 7.27 (m, 1H), 6.53 (d, *J* = 15.7 Hz, 1H), 6.28 (ddd, *J* = 15.6, 8.2, 7.1 Hz, 1H), 3.57 (dd, J = 7.6, 1.2 Hz, 2H), 1.40 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 136.8, 133.6, 128.7, 127.8, 126.6, 124.8, 48.0, 43.9, 30.2. **HRMS** (CI) m/z (M^+) calcd for $C_{13}H_{18}S_2$: 238.0850, found 238.0852.



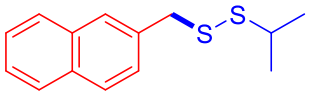
1-cyclopentyl-2-(naphthalen-2-ylmethyl)disulfane (6a)

Yield: 55% (60.3 mg). White solid. **IR** (neat, ν, cm^{-1}): 3413, 2955, 2866, 1709, 1350, 1220, 816, 748, 529, 478. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (dt, *J* = 8.2, 3.6 Hz, 3H), 7.68 (dd, *J* = 10.0, 1.7 Hz, 1H), 7.39 (qd, *J* = 5.7, 5.2, 2.2 Hz, 3H), 3.99 (s, 2H), 2.93 (tt, *J* = 7.3, 5.5 Hz, 1H), 1.85 – 1.69 (m, 2H), 1.66 – 1.57 (m, 2H), 1.57 – 1.49 (m, 2H), 1.46 – 1.39 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 135.1, 133.4, 132.8, 128.4, 128.1, 127.9, 127.8, 127.4, 126.3, 126.0, 50.2, 44.6, 33.1, 24.8. **HRMS** (CI) m/z (M^+) calcd for $C_{16}H_{18}S_2$: 274.0850, found 274.0856.



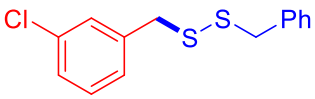
1-cyclohexyl-2-(naphthalen-2-ylmethyl)disulfane (6b)

Yield: 55% (63.4 mg). White solid. **IR** (neat, ν, cm^{-1}): 3051, 2925, 2850, 2359, 1710, 1360, 1220, 749, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 5.5, 3.6 Hz, 3H), 7.66 (d, *J* = 1.7 Hz, 1H), 7.40 – 7.36 (m, 3H), 3.97 (s, 2H), 2.27 (tt, *J* = 11.0, 3.7 Hz, 1H), 1.84 (dt, *J* = 13.1, 3.8 Hz, 2H), 1.61 (dt, *J* = 12.7, 3.7 Hz, 2H), 1.22 – 0.96 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 135.0, 133.3, 132.7, 128.3, 128.0, 127.8, 127.7, 127.3, 126.2, 125.9, 49.5, 45.1, 32.9, 26.1, 25.6. **HRMS** (CI) m/z (M^+) calcd for $C_{17}H_{20}S_2$: 288.1006, found 288.1013.



1-isopropyl-2-(naphthalen-2-ylmethyl)disulfane (6c)

Yield: 55% (54.6 mg). White solid. **IR** (neat, ν, cm^{-1}): 2961, 2360, 1709, 1360, 1220, 818, 751, 529, 479. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.71 (m, 3H), 7.66 (d, *J* = 1.7 Hz, 1H), 7.41 – 7.37 (m, 3H), 3.98 (s, 2H), 2.64 (p, *J* = 6.7 Hz, 1H), 1.14 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 135.0, 133.3, 132.7, 128.4, 128.0, 127.8, 127.7, 127.3, 126.2, 125.9, 45.0, 41.0, 22.6. **HRMS** (CI) m/z (M^+) calcd for $C_{14}H_{16}S_2$: 248.0693, found 248.0692.



1-benzyl-2-(3-chlorobenzyl)disulfane (6d)

Yield: 46% (51.5 mg). White solid. **IR** (neat, ν, cm^{-1}): 3061, 2924, 1597, 1454, 1228, 1076, 877, 787, 698, 472. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 3H), 7.31 – 7.26 (m, 4H), 7.22 (q, *J* = 1.4 Hz, 1H), 7.12 (ddd, *J* = 5.0, 3.6, 1.7 Hz, 1H), 3.68 (s, 2H), 3.53 (s, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.4, 139.3, 137.2, 134.2, 129.7, 129.5, 129.4, 128.6, 127.6, 127.5, 43.4, 42.5. **HRMS** (CI) m/z (M^+) calcd for $C_{14}H_{13}ClS_2$: 280.0147, found 280.0154.

V. References

[1] He, R.-D.; Li, C.-L.; Pan, Q.-Q.; Guo, P.; Liu, X.-Y.; Shu, X.-Z. Reductive Coupling between C–N and C–O Electrophiles. *J. Am. Chem. Soc.* **2019**, *141*, 12481–12486.

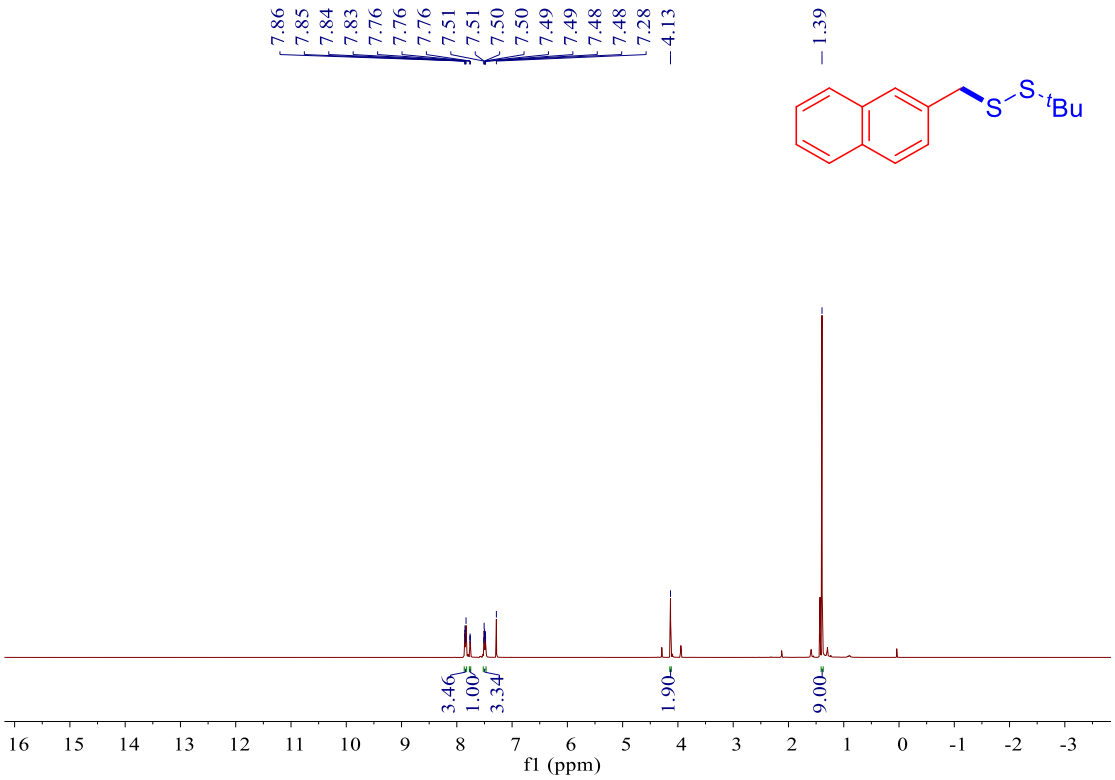
[2] Wu, Z.; Pratt, D. A. Radical Substitution Provides a Unique Route to Disulfides. *J. Am. Chem. Soc.* **2020**, *142*, 10284–10290.

[3] Chauvin, J. P. R.; Griesser, M.; Pratt, D. A. *Chem. Sci*. **2019**, *10*, 4999.

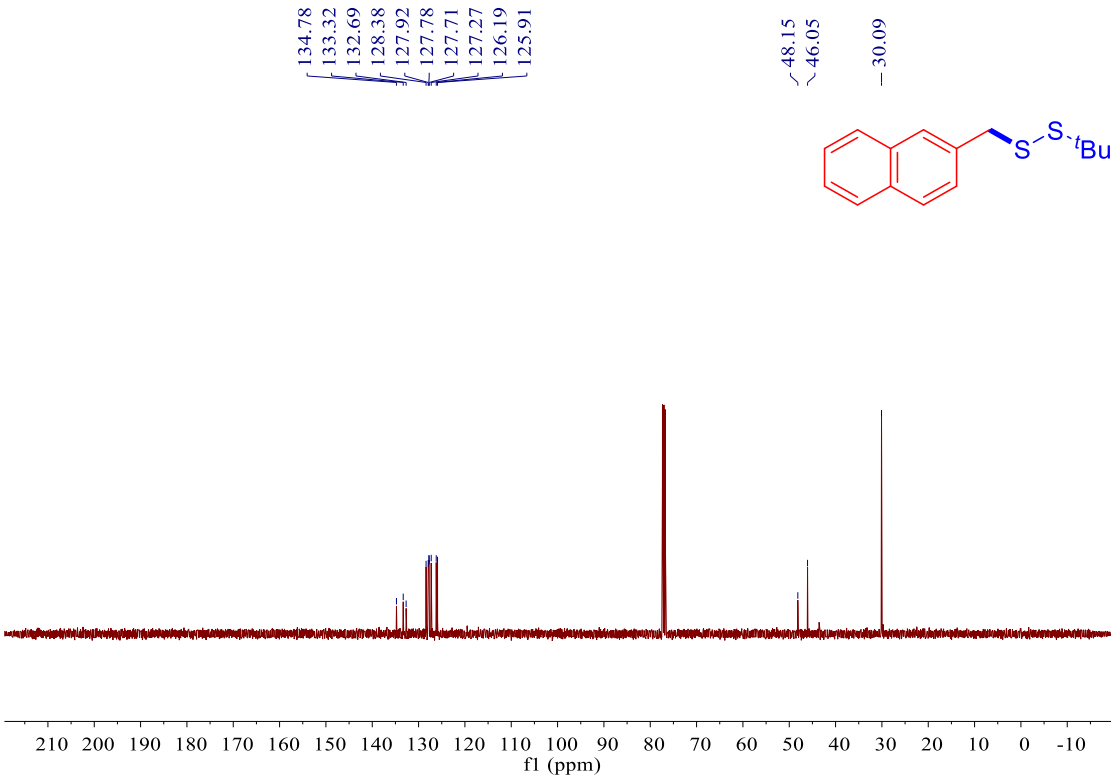
[4] Cerda, M. M.; Hammers, M. D.; Earp, M. S.; Zakharov, L. N.; Pluth, M. D. *Org. Lett*. **2017**, *19*, 2314−2317.

VI. Copies of ^{1}H NMR and ^{13}C NMR Spectra

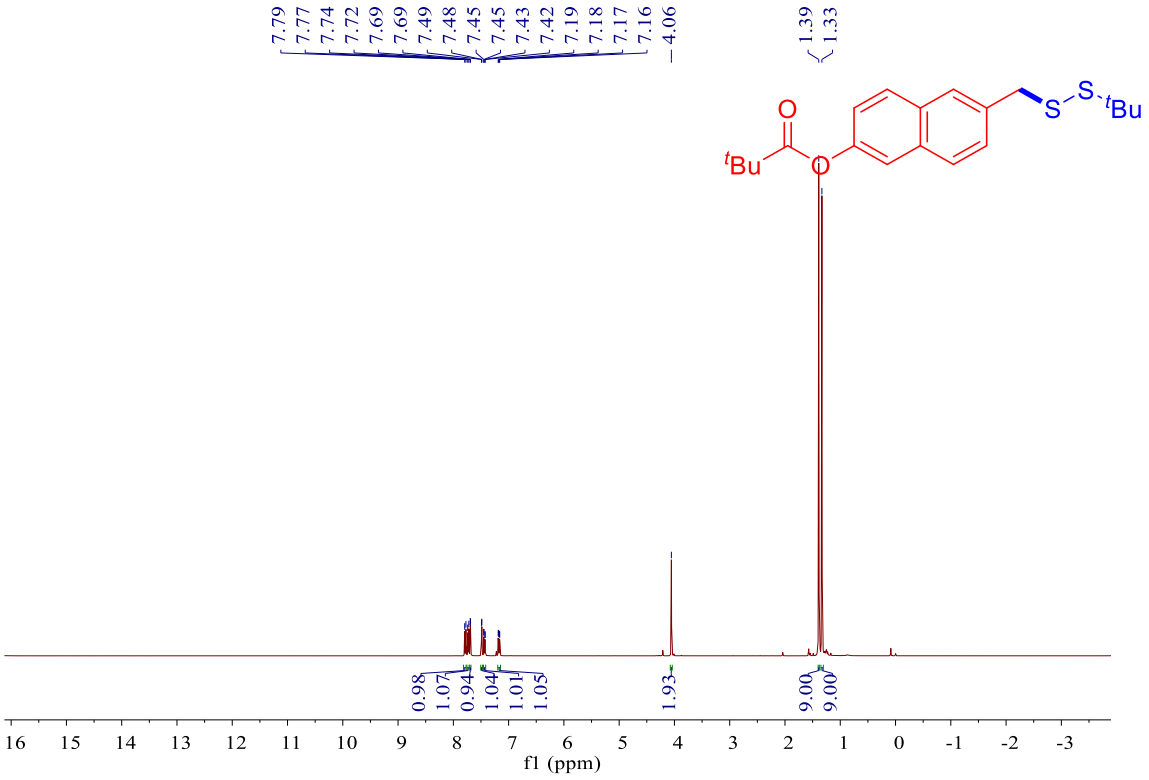
^{1}H NMR Spectra of **3a** (400 MHz, $CDCl_3$)



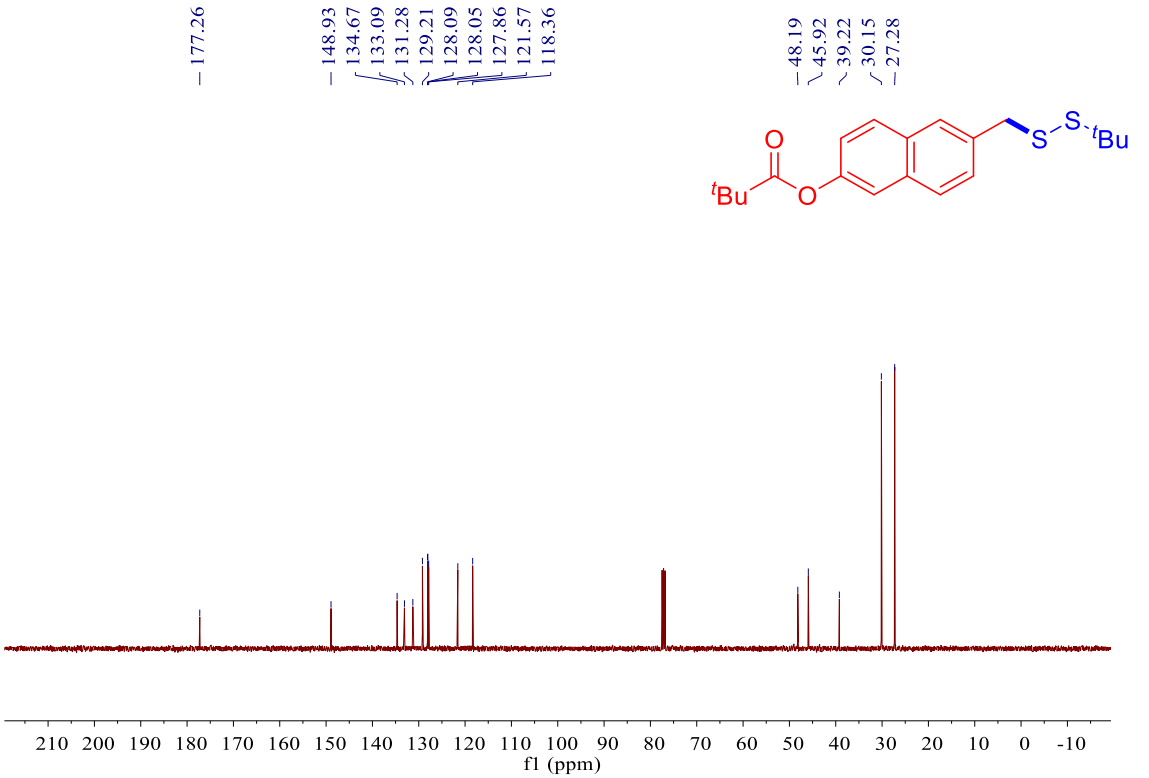
^{13}C NMR Spectra of **3a** (400 MHz, $CDCl_3$)



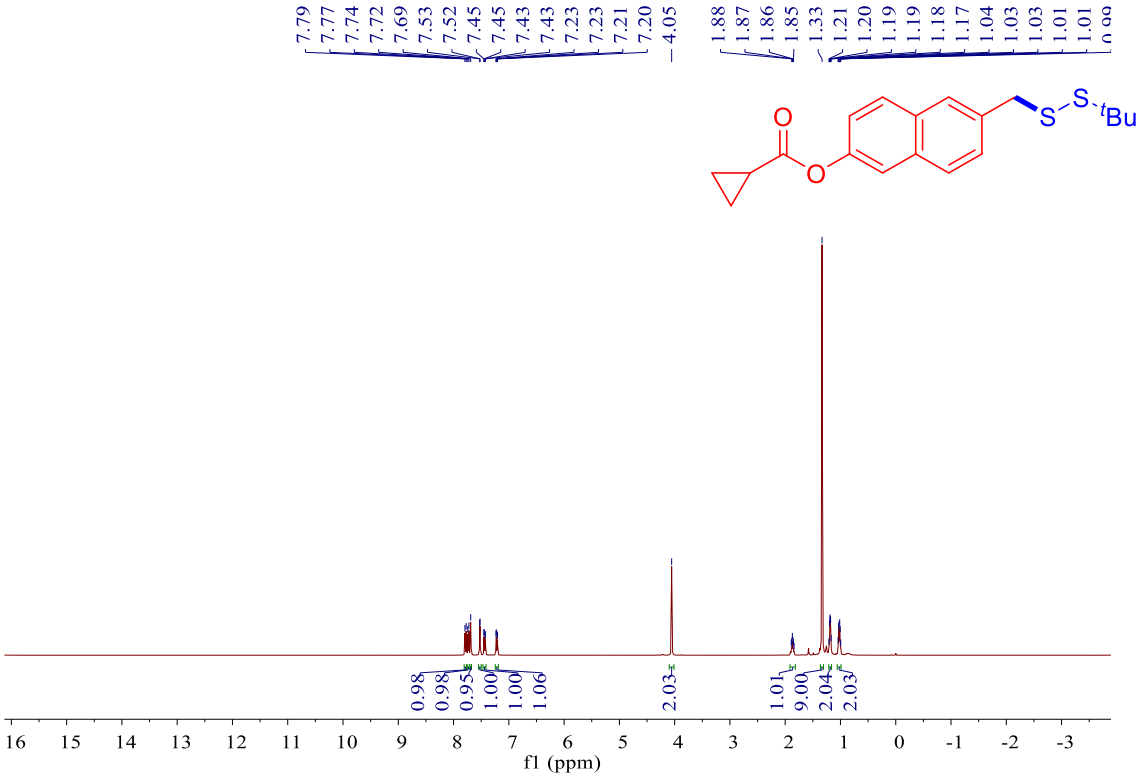
^{1}H NMR Spectra of **3b** (400 MHz, $CDCl_3$)



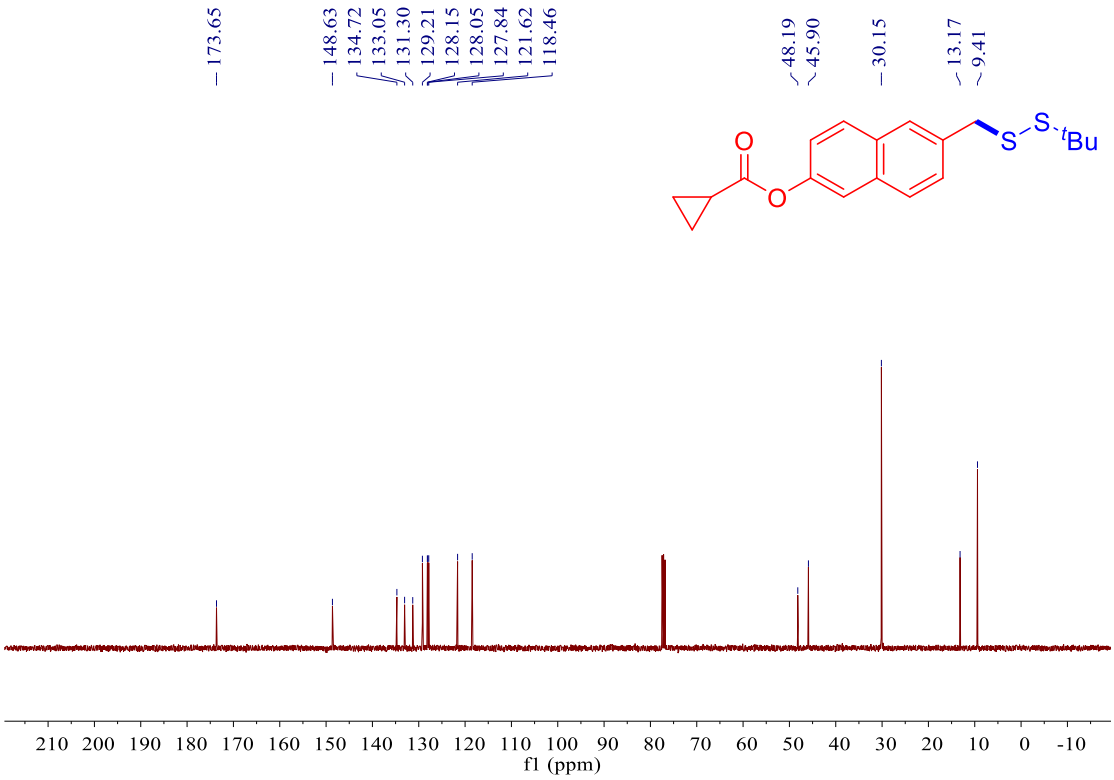
^{13}C NMR Spectra of **3b** (400 MHz, $CDCl_3$)



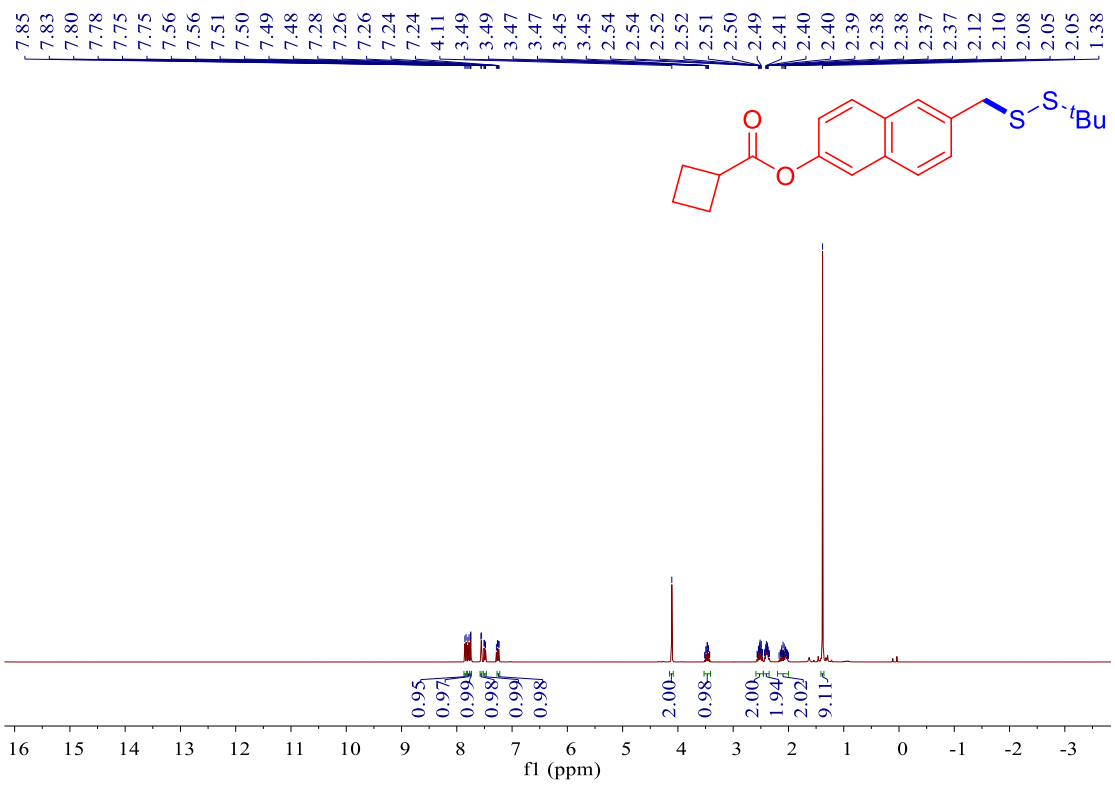
^{1}H NMR Spectra of **3c** (400 MHz, $CDCl_3$)



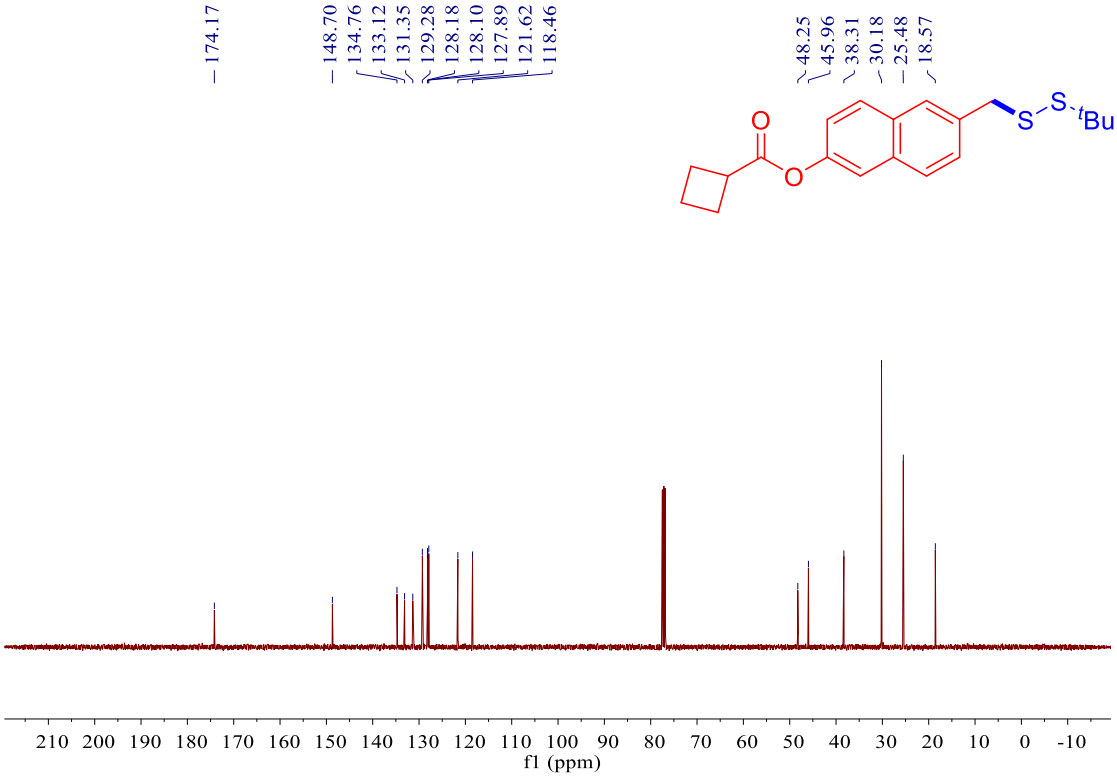
^{13}C NMR Spectra of **3c** (400 MHz, $CDCl_3$)



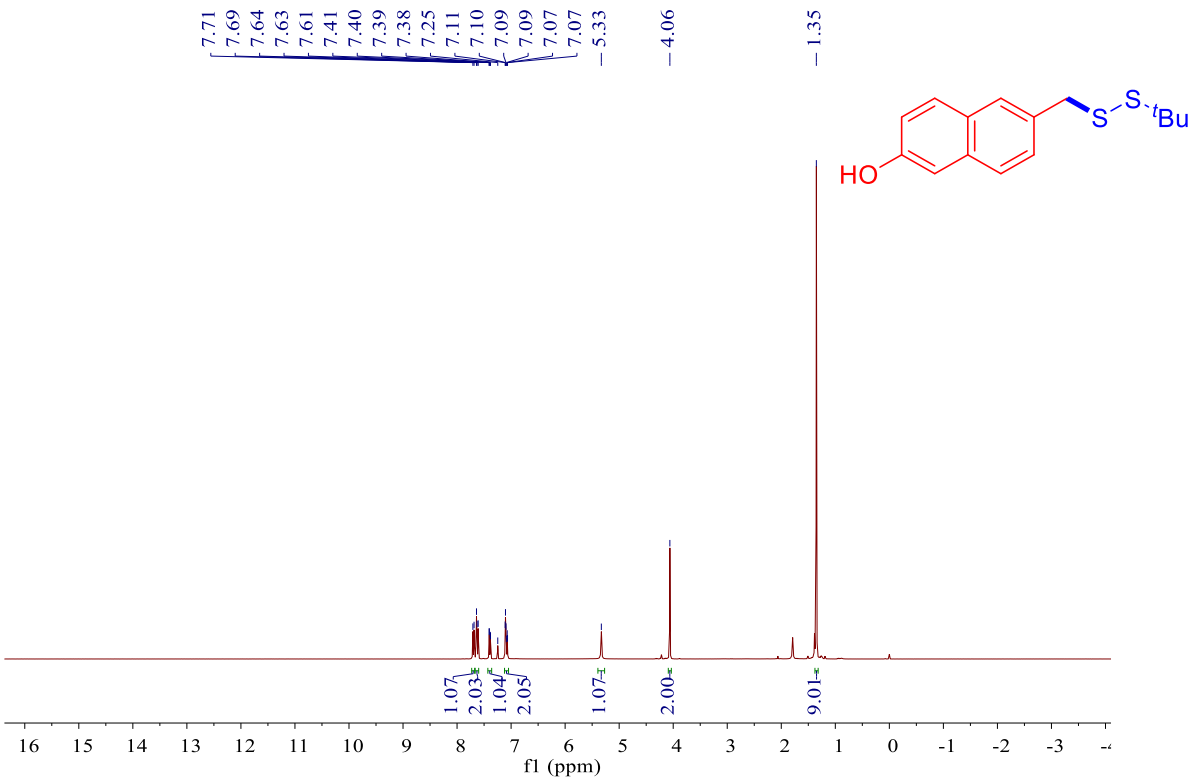
^{1}H NMR Spectra of **3d** (400 MHz, $CDCl_3$)



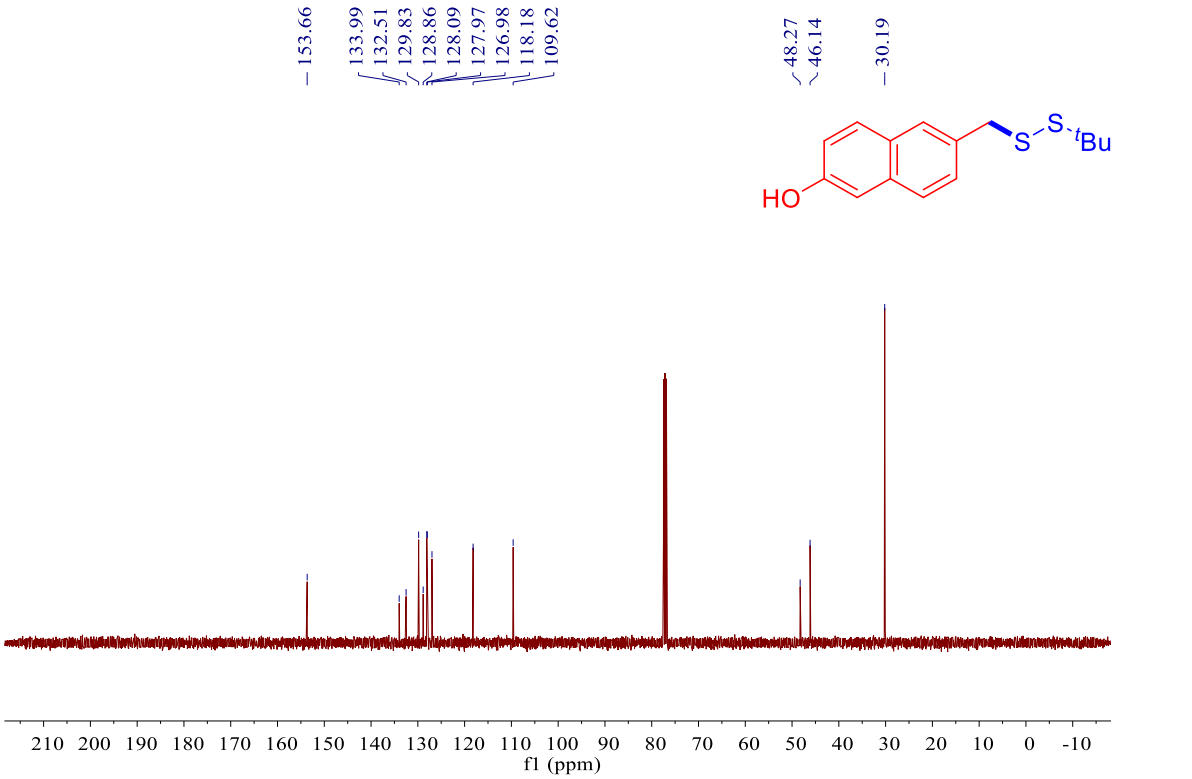
^{13}C NMR Spectra of **3d** (400 MHz, $CDCl_3$)



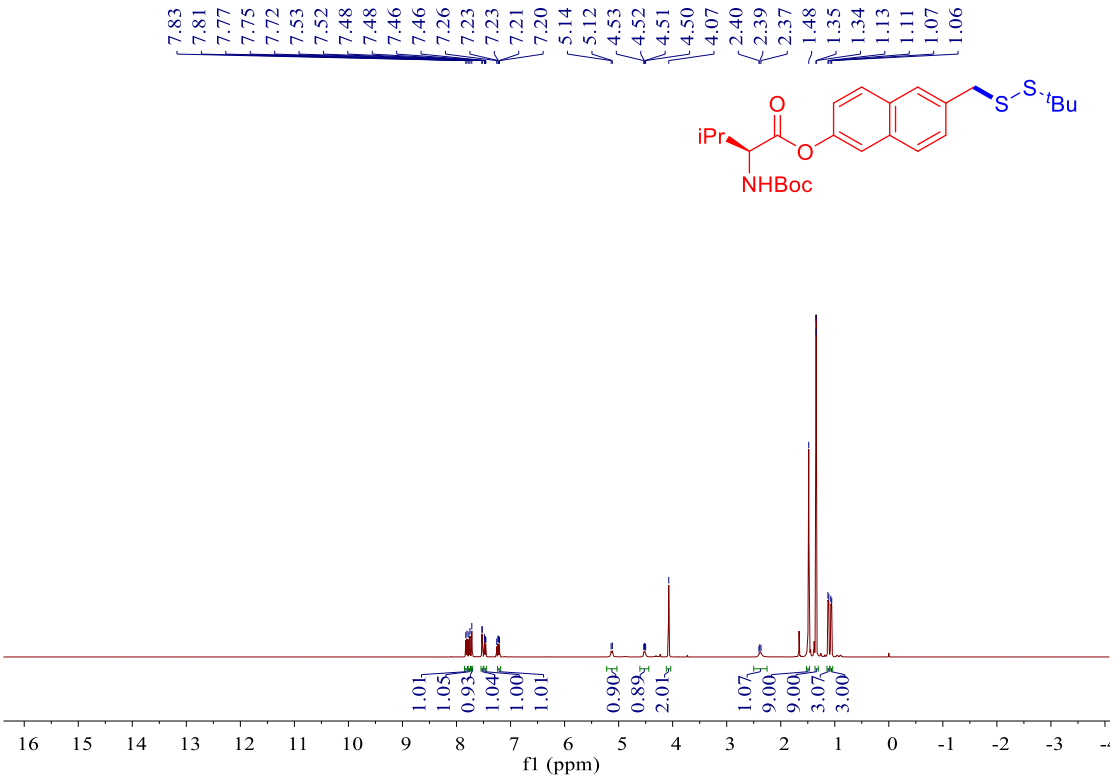
^{1}H NMR Spectra of **3d'** (400 MHz, $CDCl_3$)



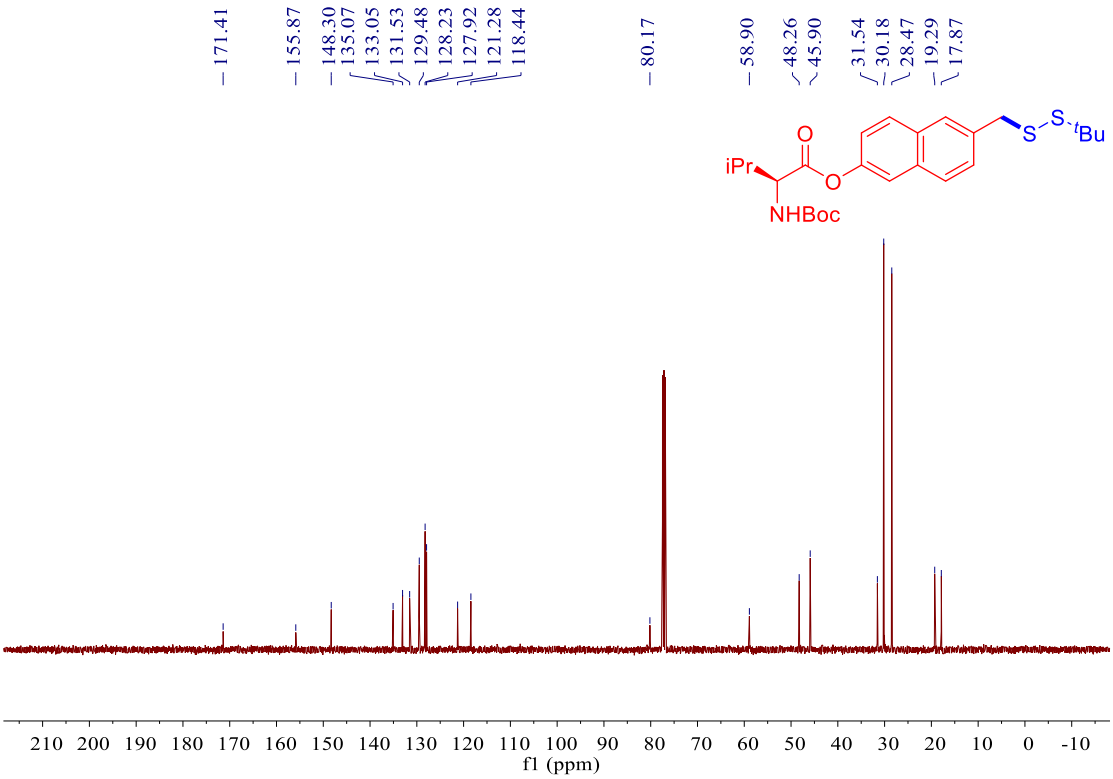
^{13}C NMR Spectra of **3d'** (400 MHz, $CDCl_3$)



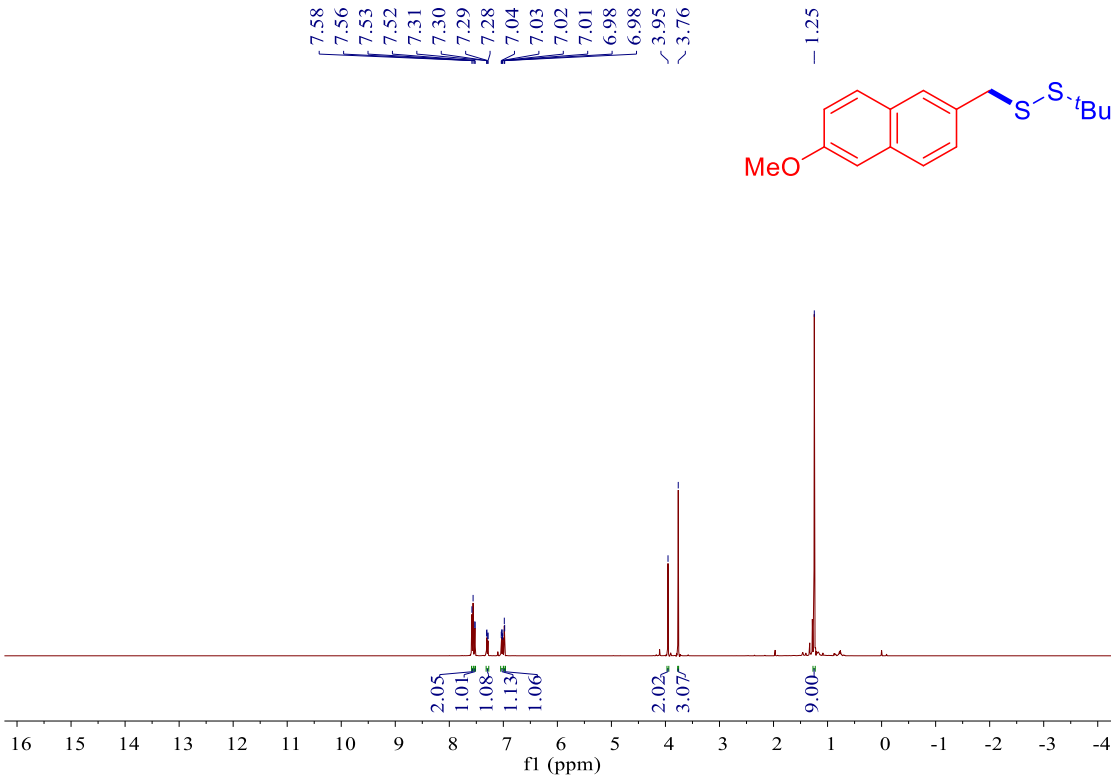
^{1}H NMR Spectra of **3e** (400 MHz, $CDCl_3$)



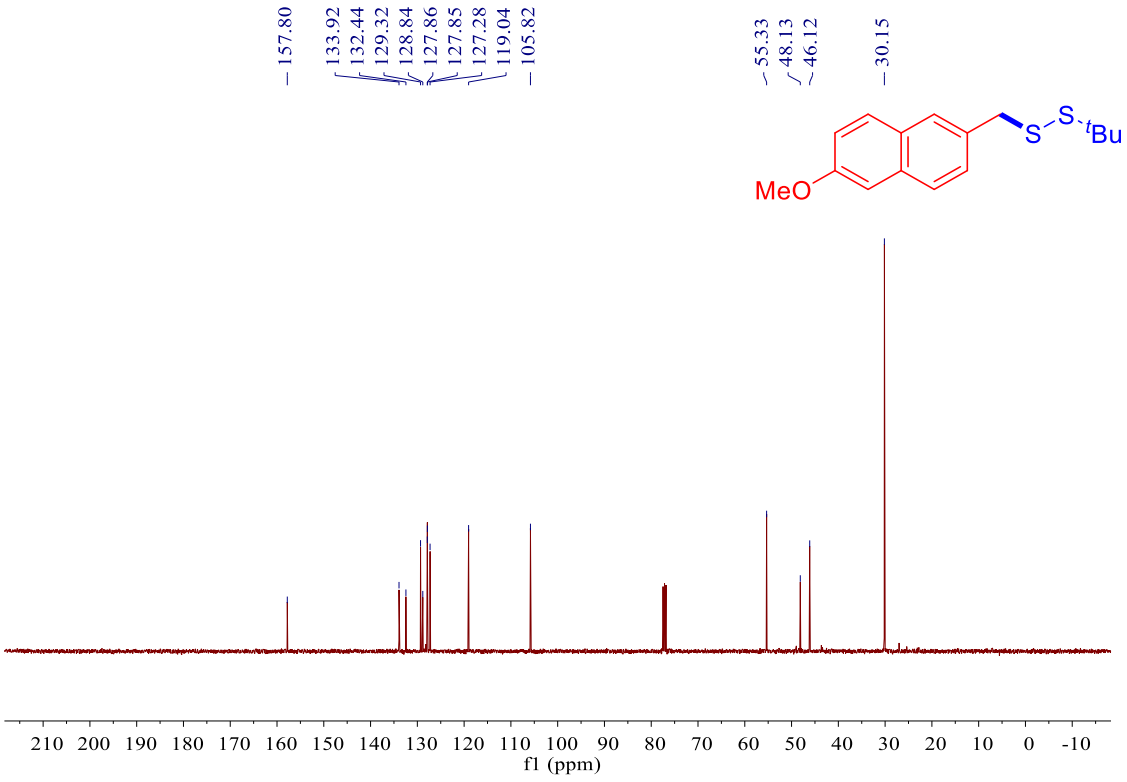
^{13}C NMR Spectra of **3e** (400 MHz, $CDCl_3$)



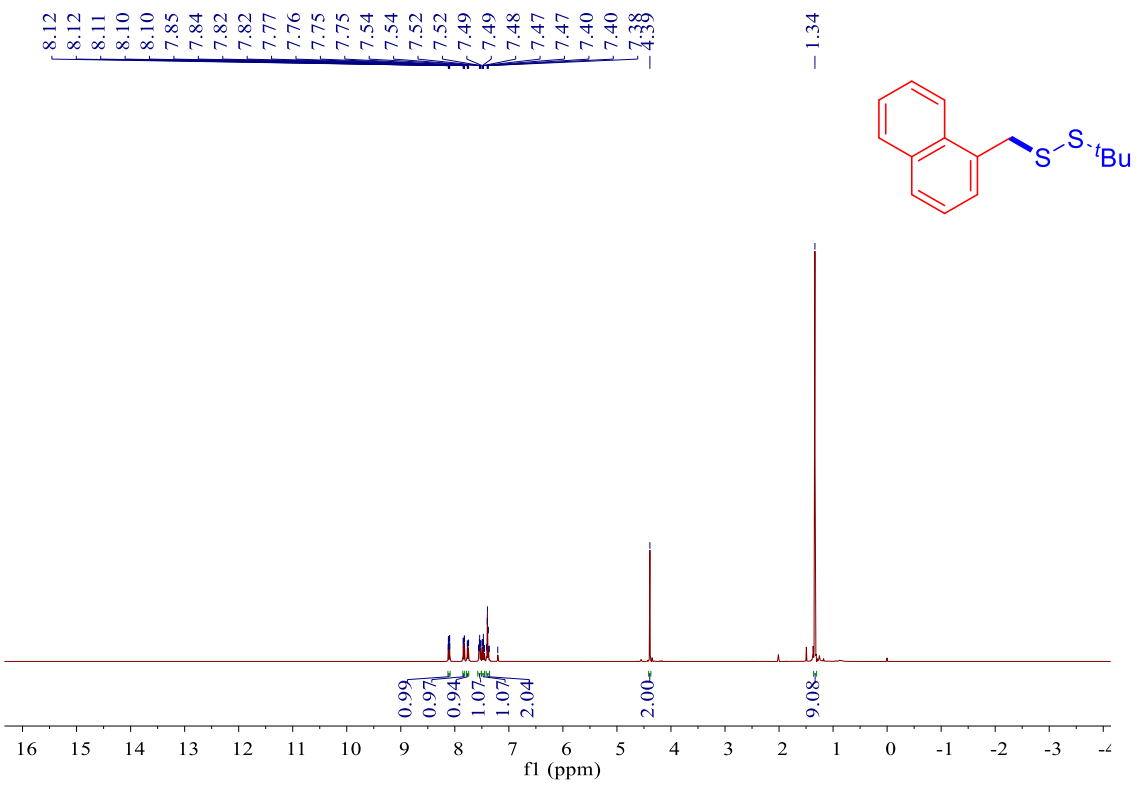
^{1}H NMR Spectra of **3f** (400 MHz, $CDCl_3$)



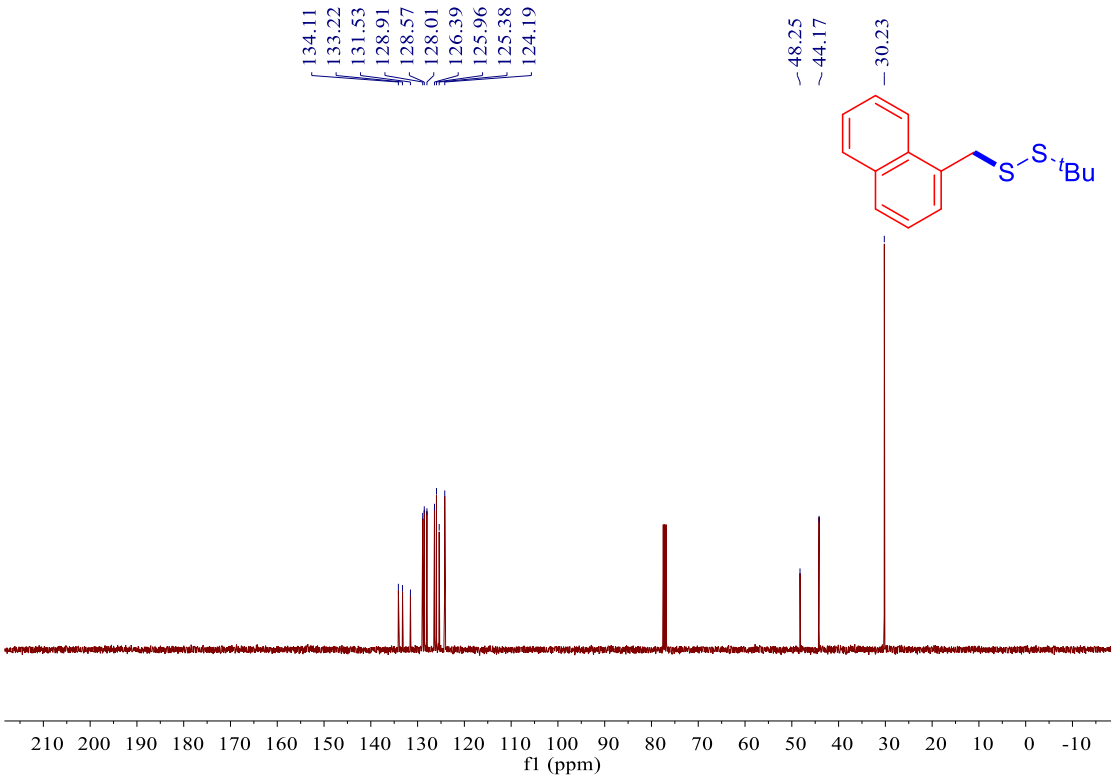
^{13}C NMR Spectra of **3f** (400 MHz, $CDCl_3$)



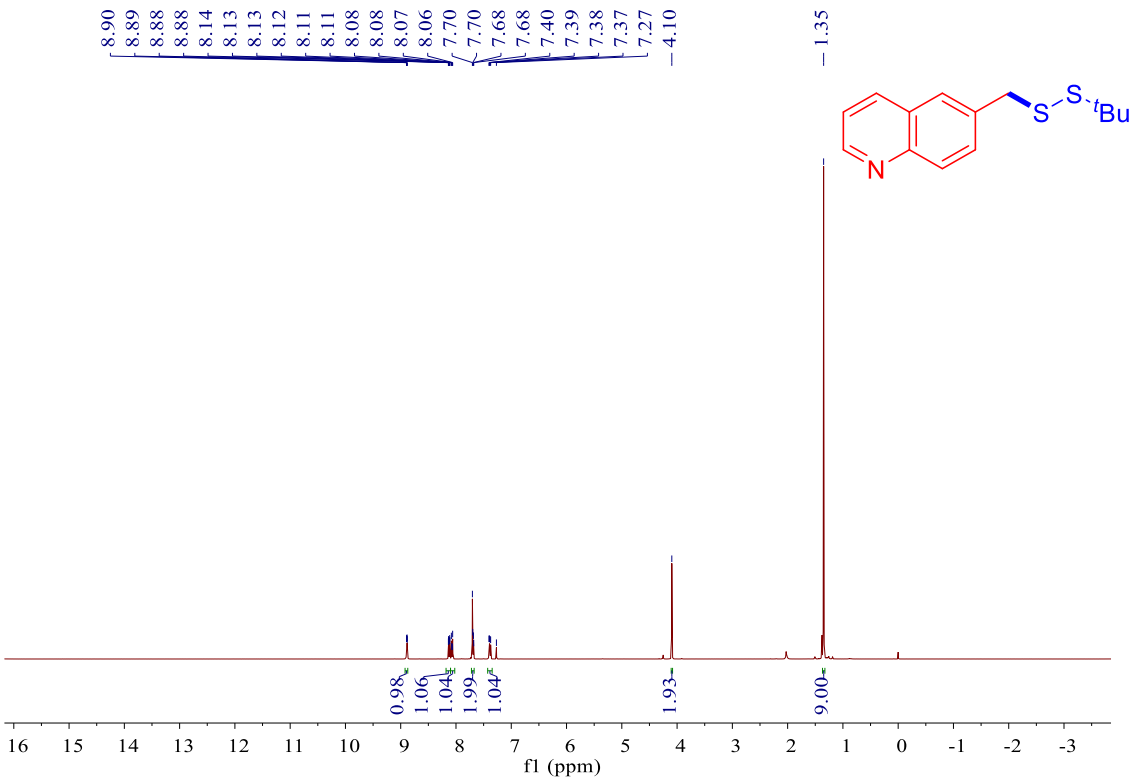
^{1}H NMR Spectra of **3g** (400 MHz, $CDCl_3$)



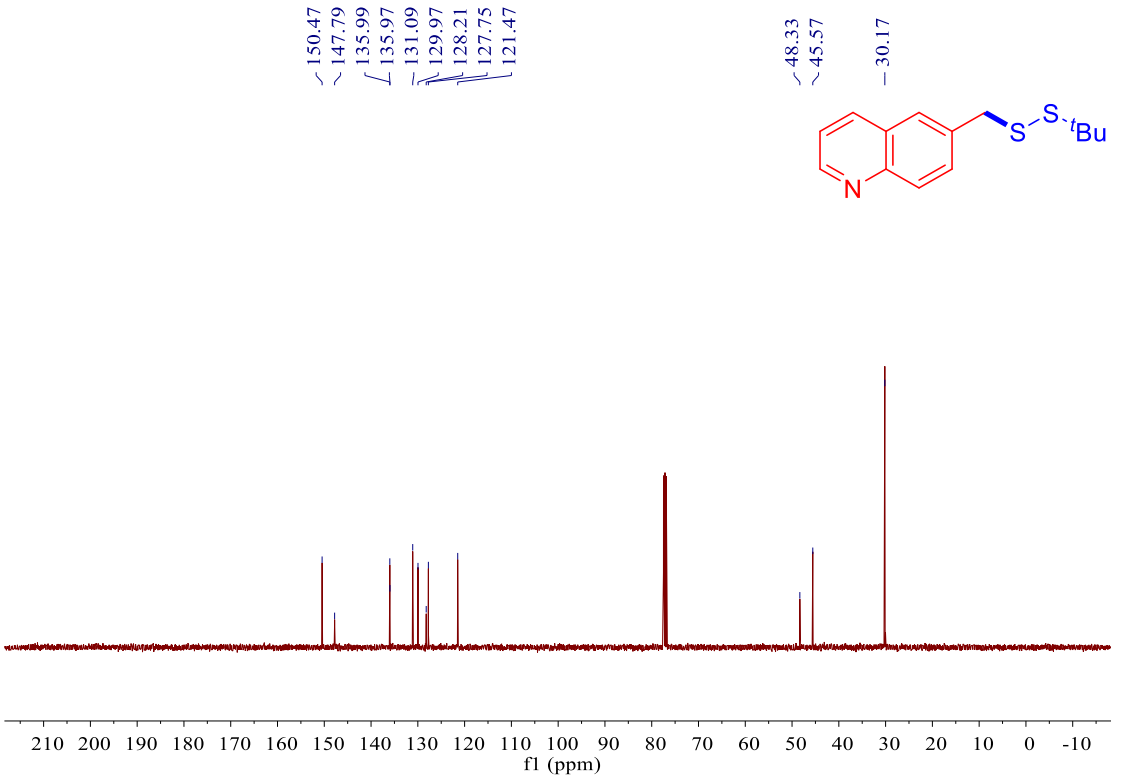
^{13}C NMR Spectra of **3g** (400 MHz, $CDCl_3$)



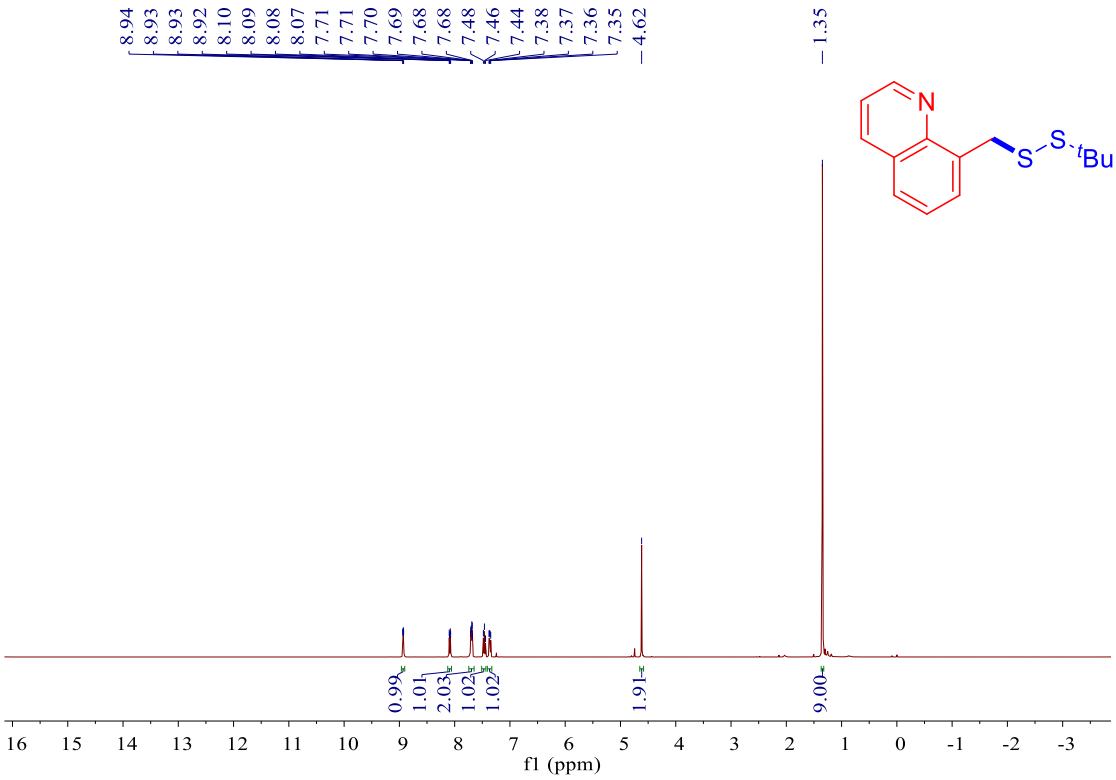
^{1}H NMR Spectra of **3h** (400 MHz, $CDCl_3$)



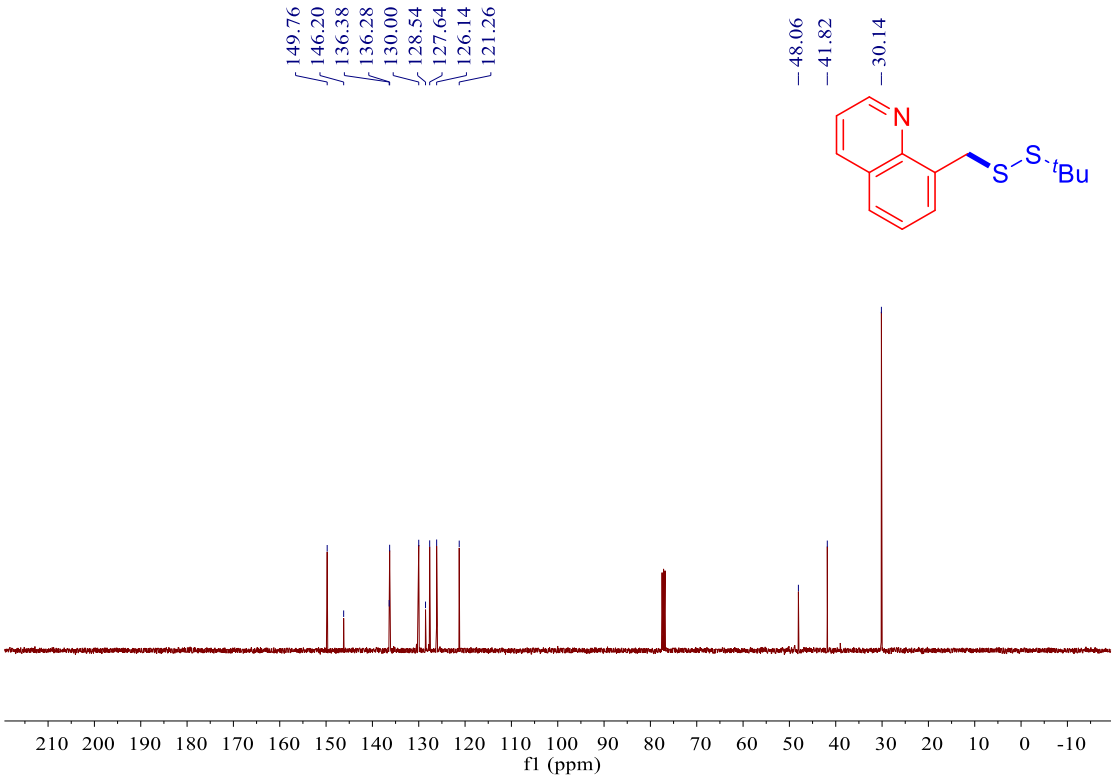
^{13}C NMR Spectra of **3h** (400 MHz, $CDCl_3$)



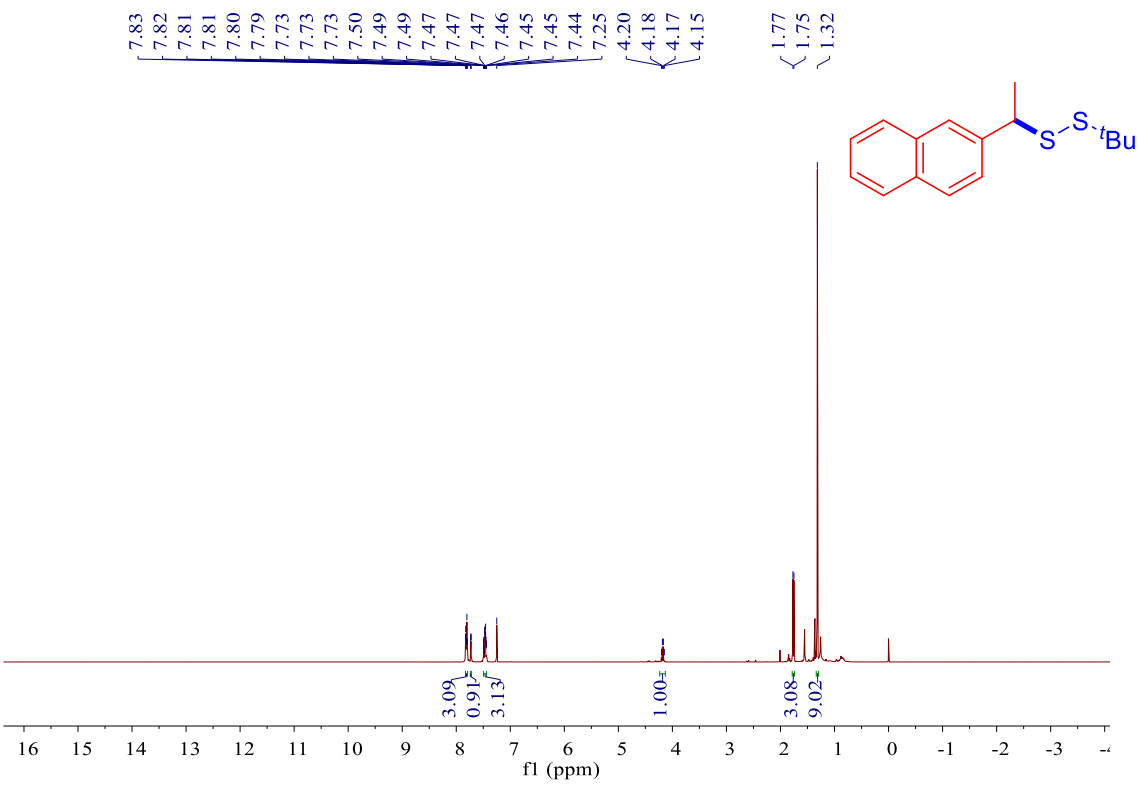
^{1}H NMR Spectra of **3i** (400 MHz, $CDCl_3$)



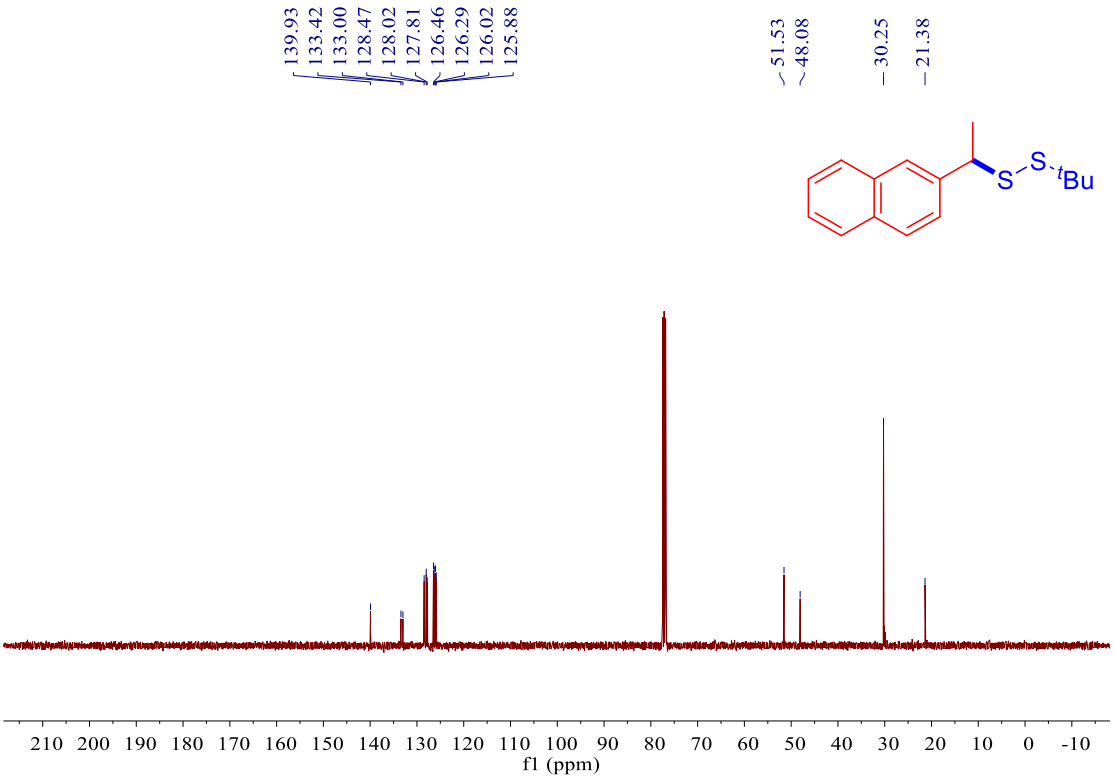
^{13}C NMR Spectra of **3i** (400 MHz, $CDCl_3$)



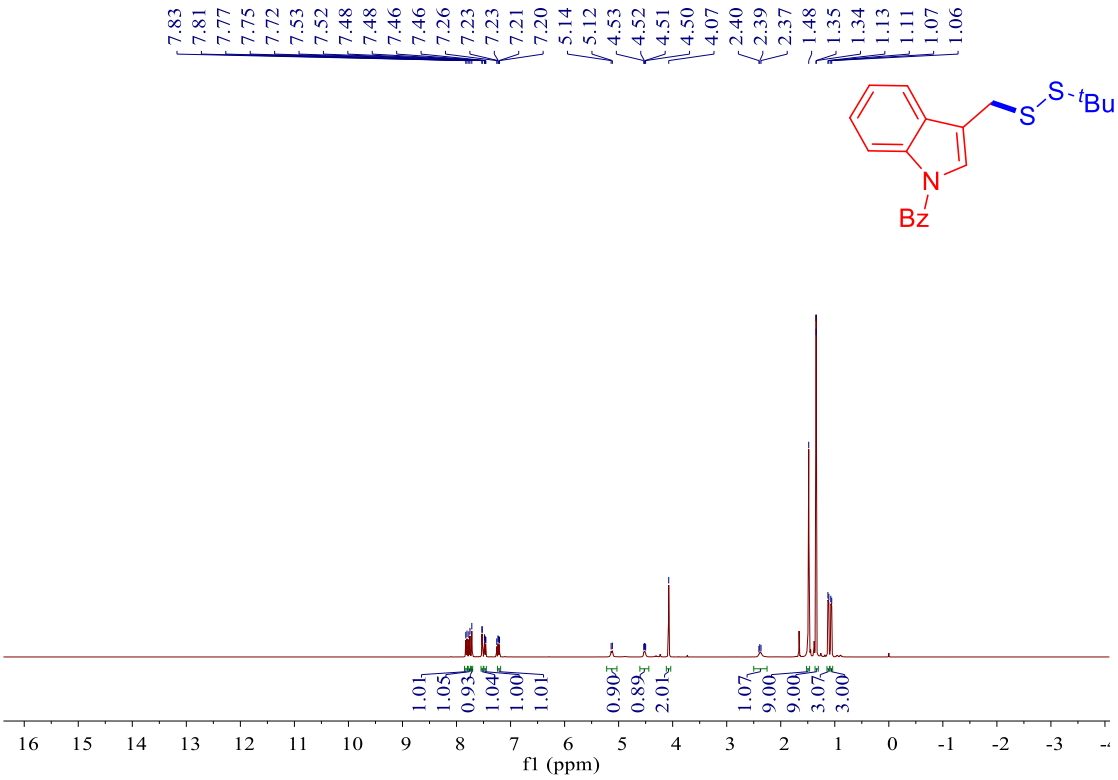
^{1}H NMR Spectra of **3j** (400 MHz, $CDCl_3$)



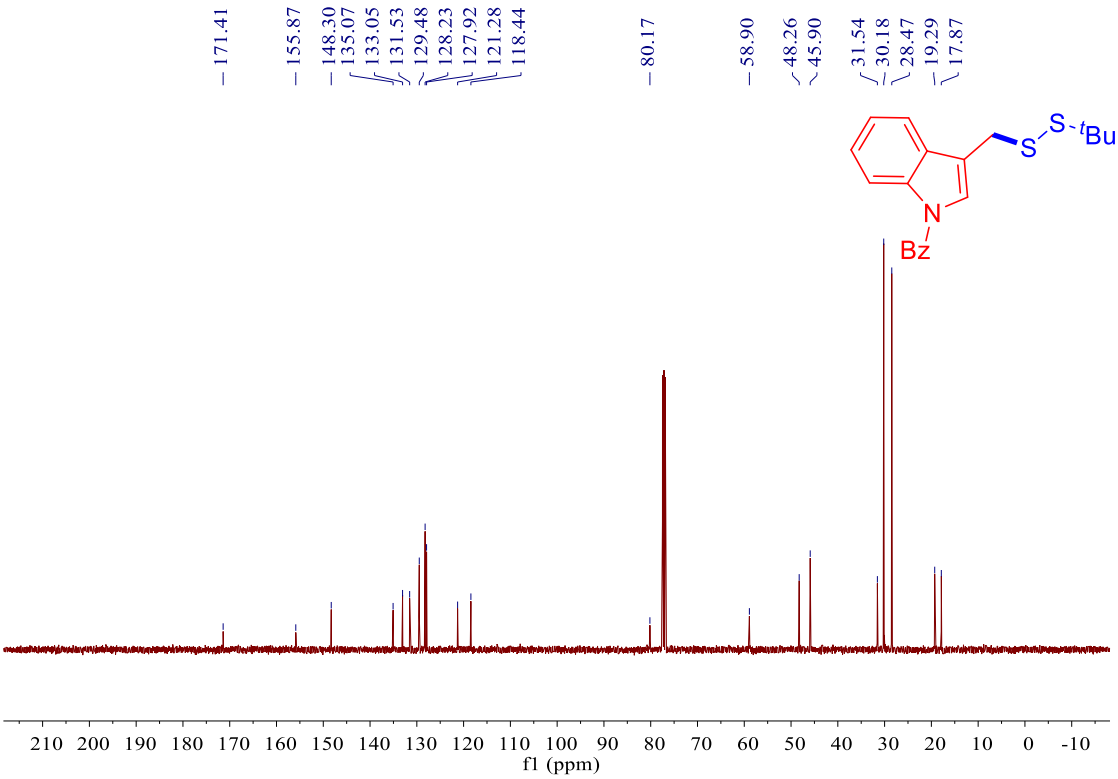
^{13}C NMR Spectra of **3j** (400 MHz, $CDCl_3$)



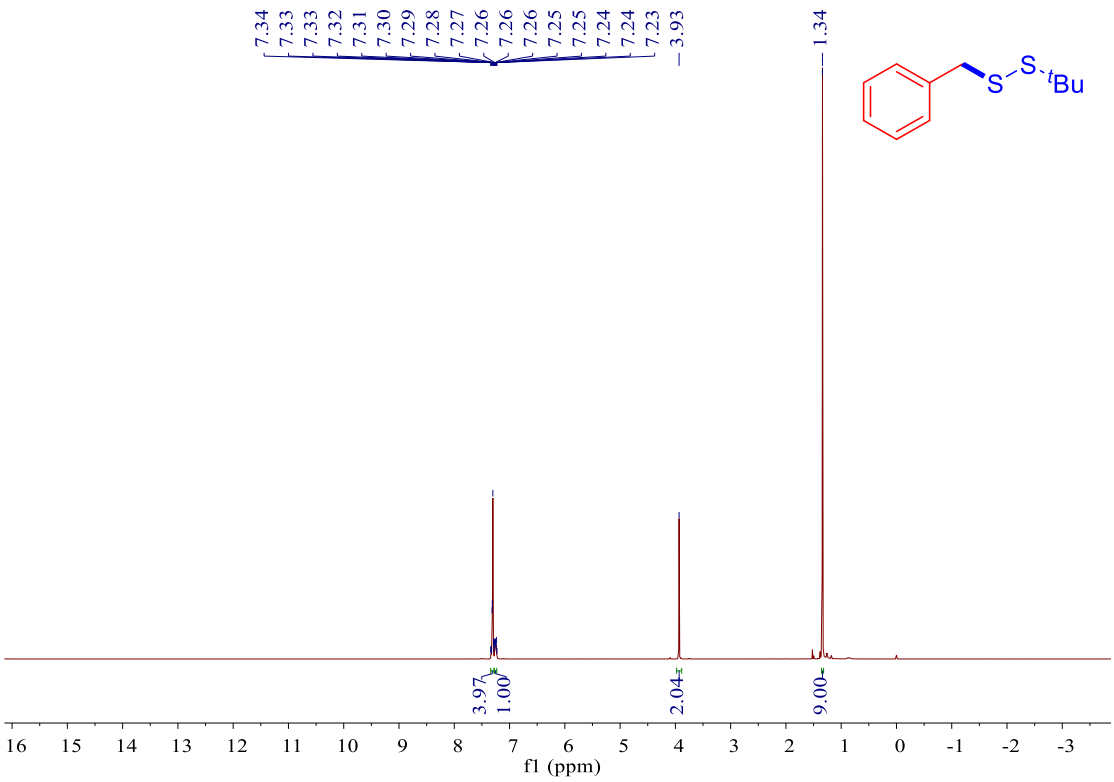
^{1}H NMR Spectra of **3k** (400 MHz, $CDCl_3$)



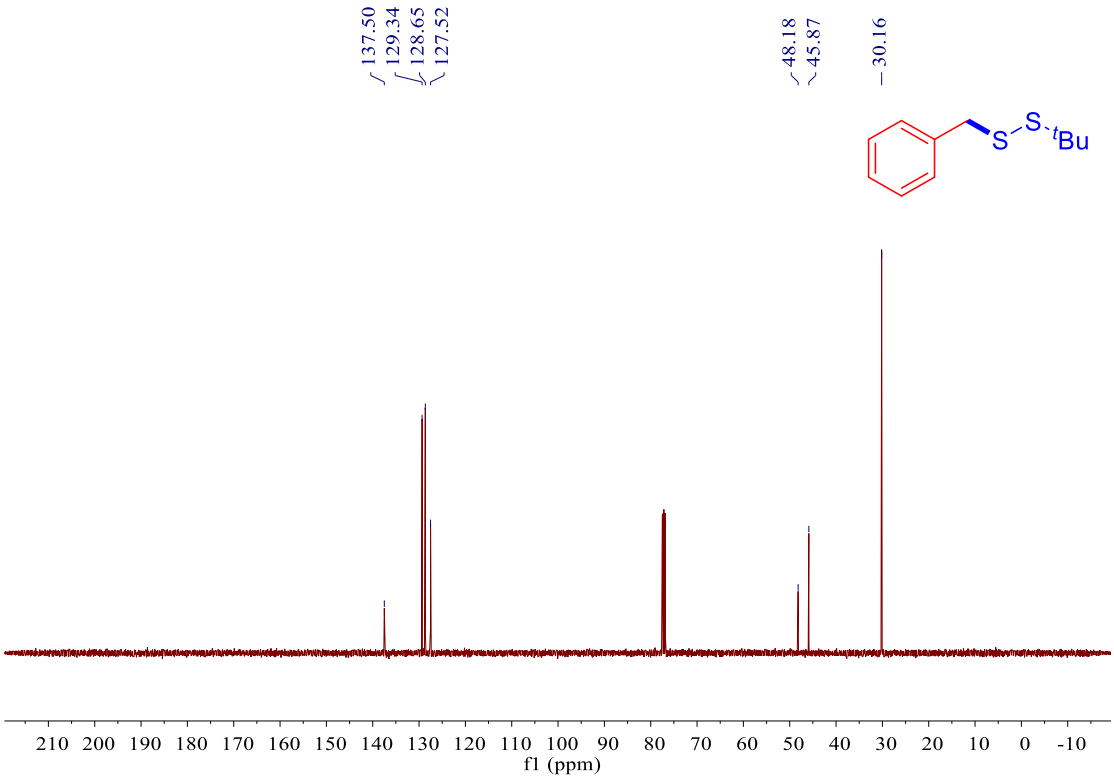
^{13}C NMR Spectra of **3k** (400 MHz, $CDCl_3$)



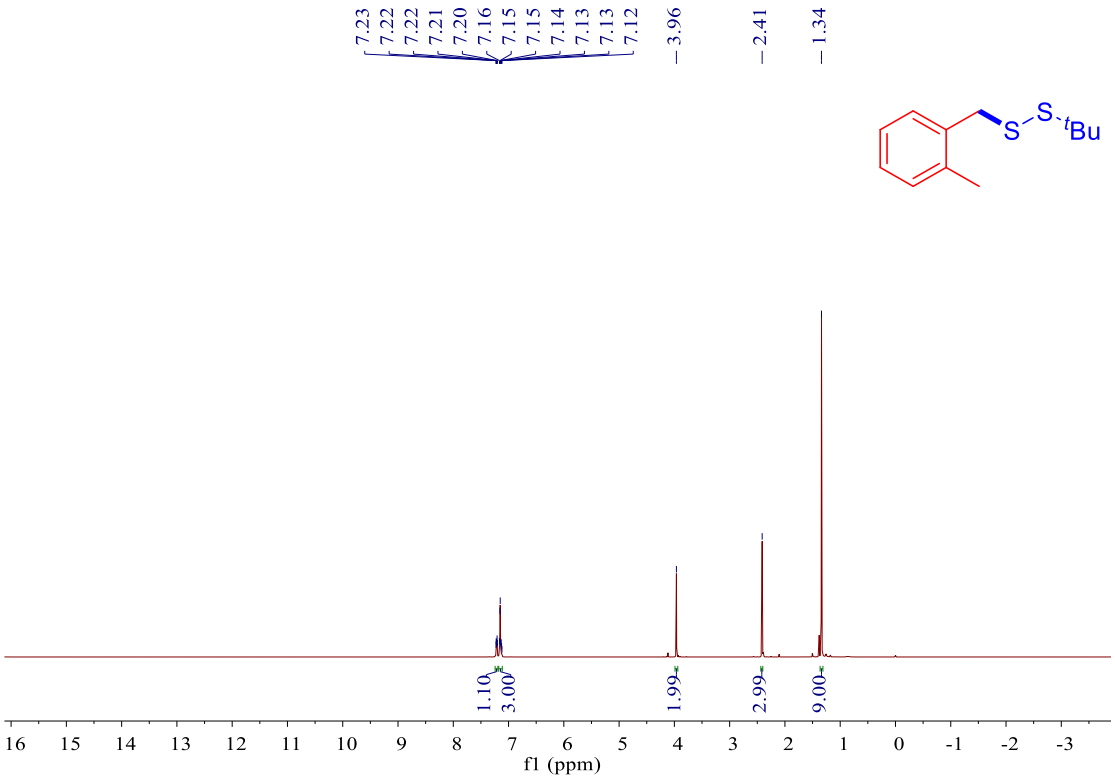
^{1}H NMR Spectra of **5a** (400 MHz, $CDCl_3$)



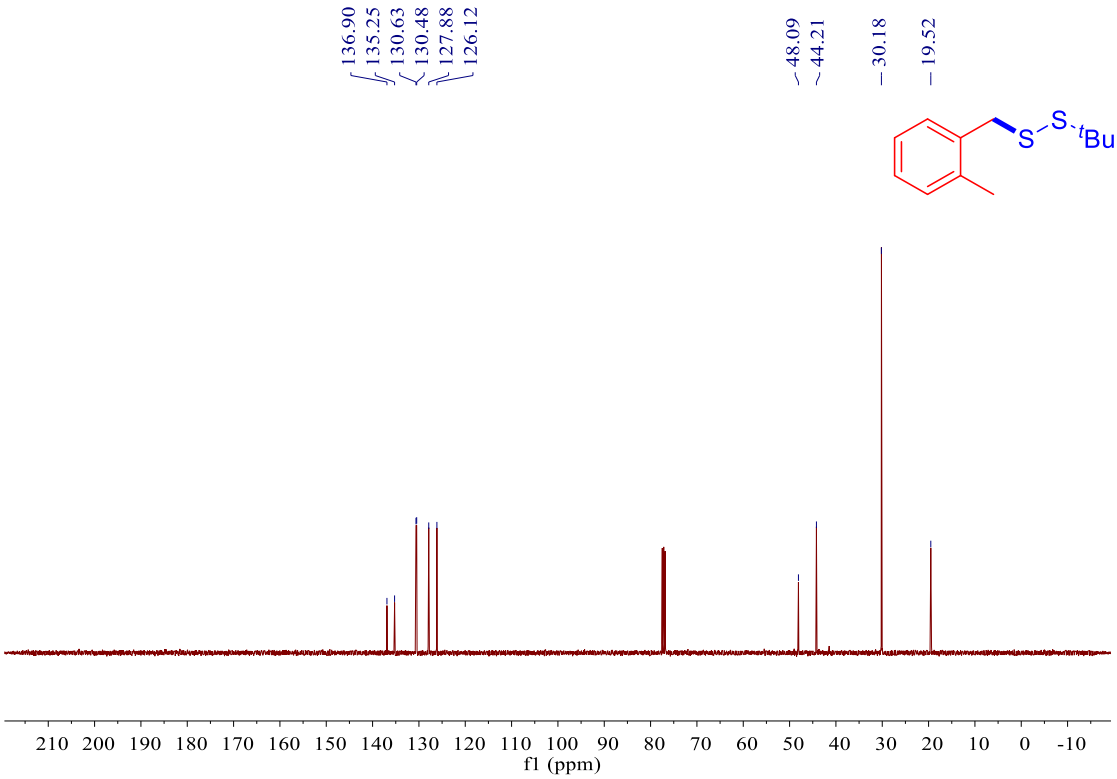
^{13}C NMR Spectra of **5a** (400 MHz, $CDCl_3$)



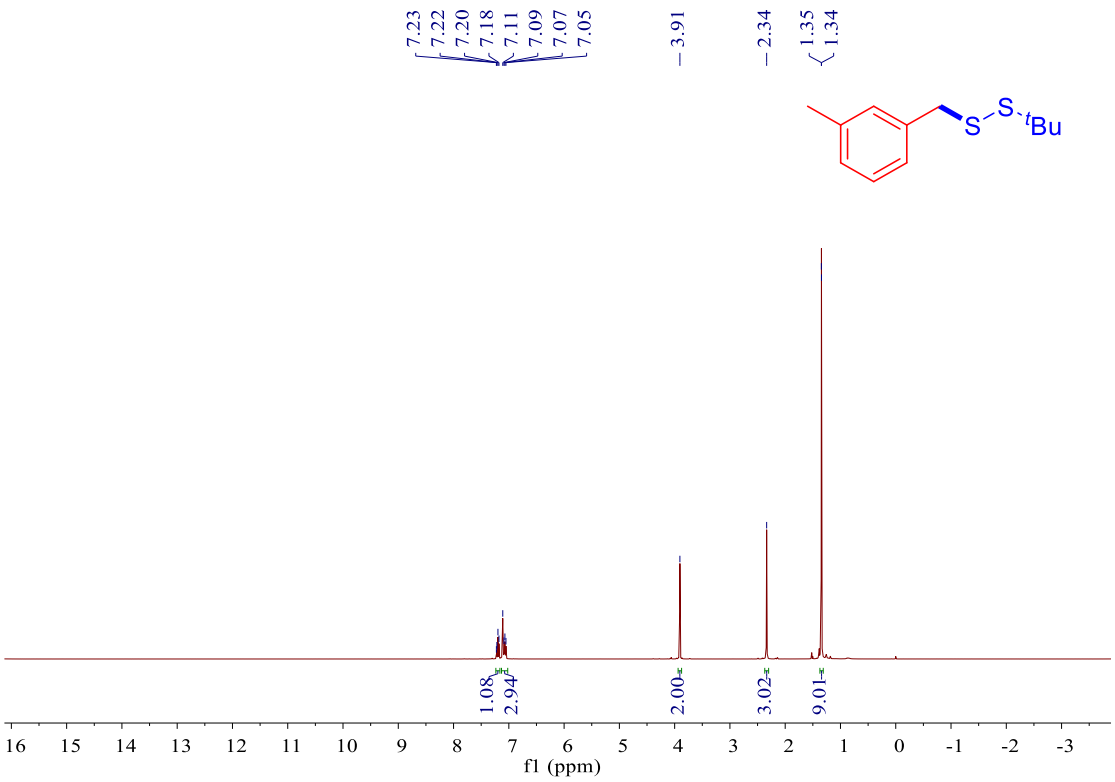
^{1}H NMR Spectra of **5b** (400 MHz, $CDCl_3$)



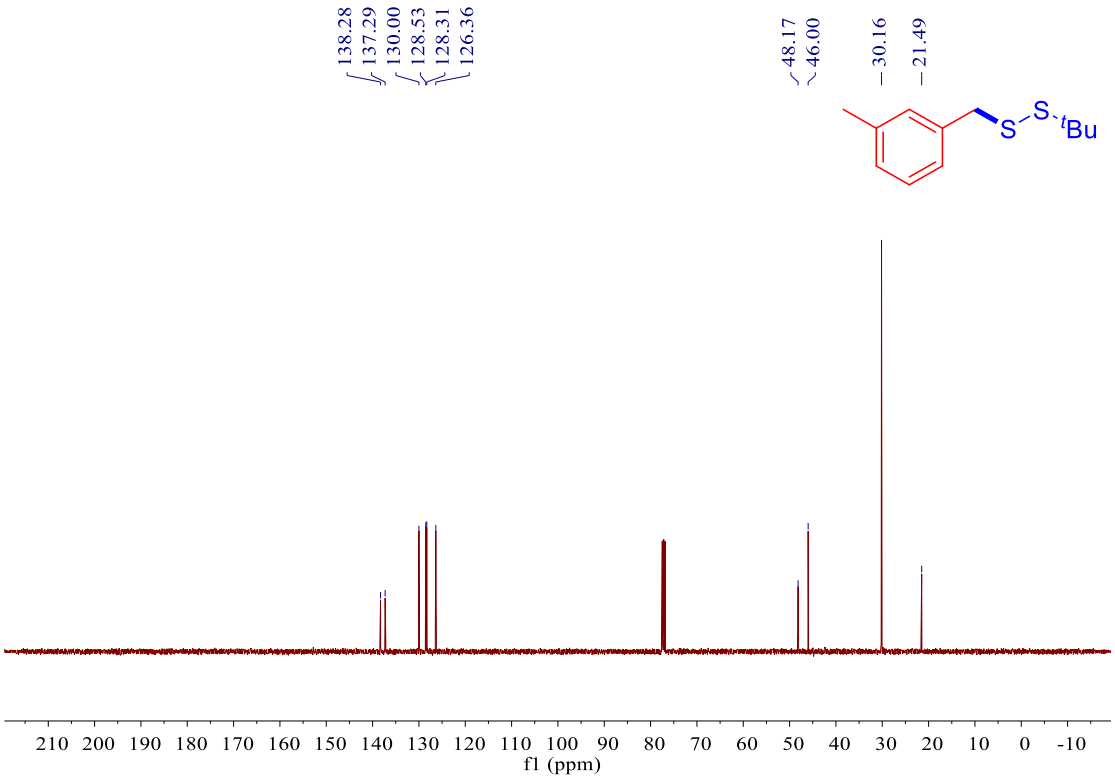
^{13}C NMR Spectra of **5b** (400 MHz, $CDCl_3$)



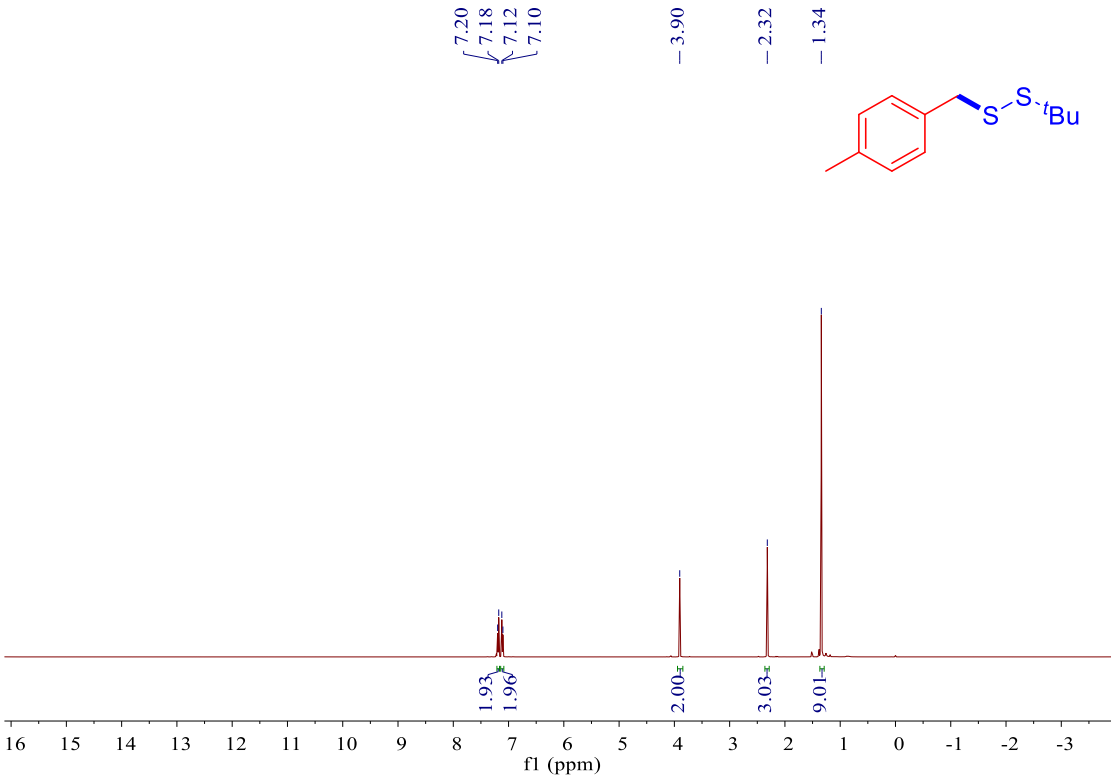
^{1}H NMR Spectra of **5c** (400 MHz, $CDCl_3$)



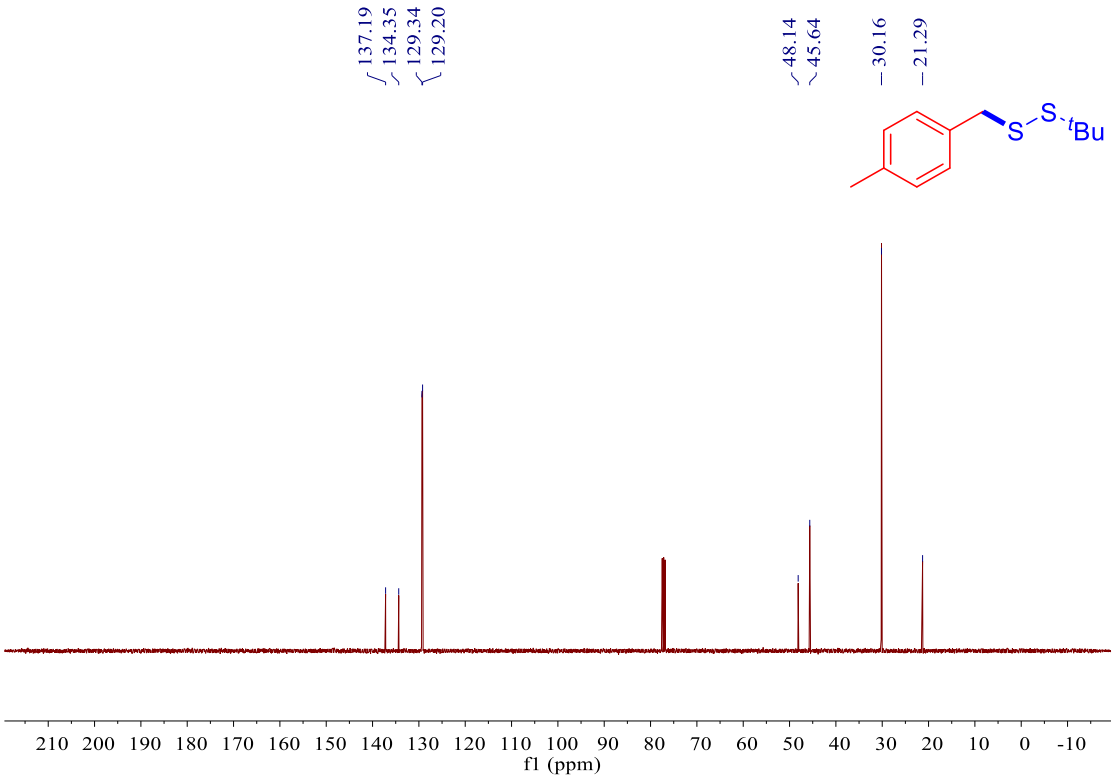
^{13}C NMR Spectra of **5c** (400 MHz, $CDCl_3$)



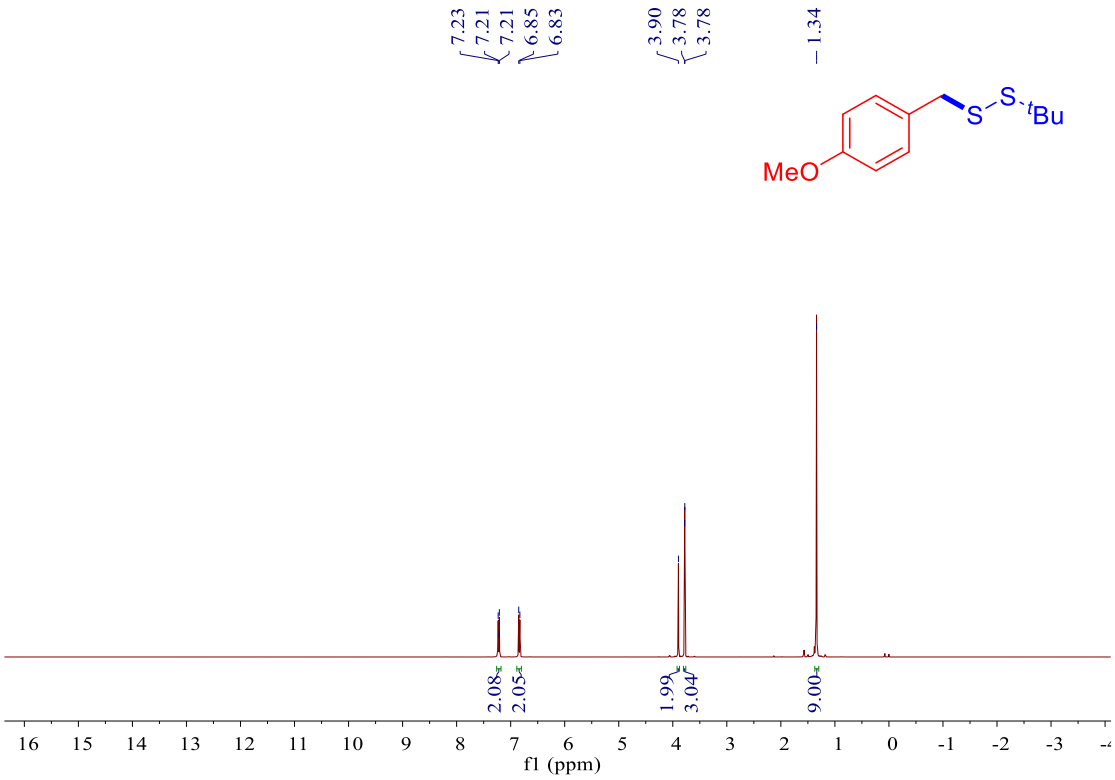
^{1}H NMR Spectra of **5d** (400 MHz, $CDCl_3$)



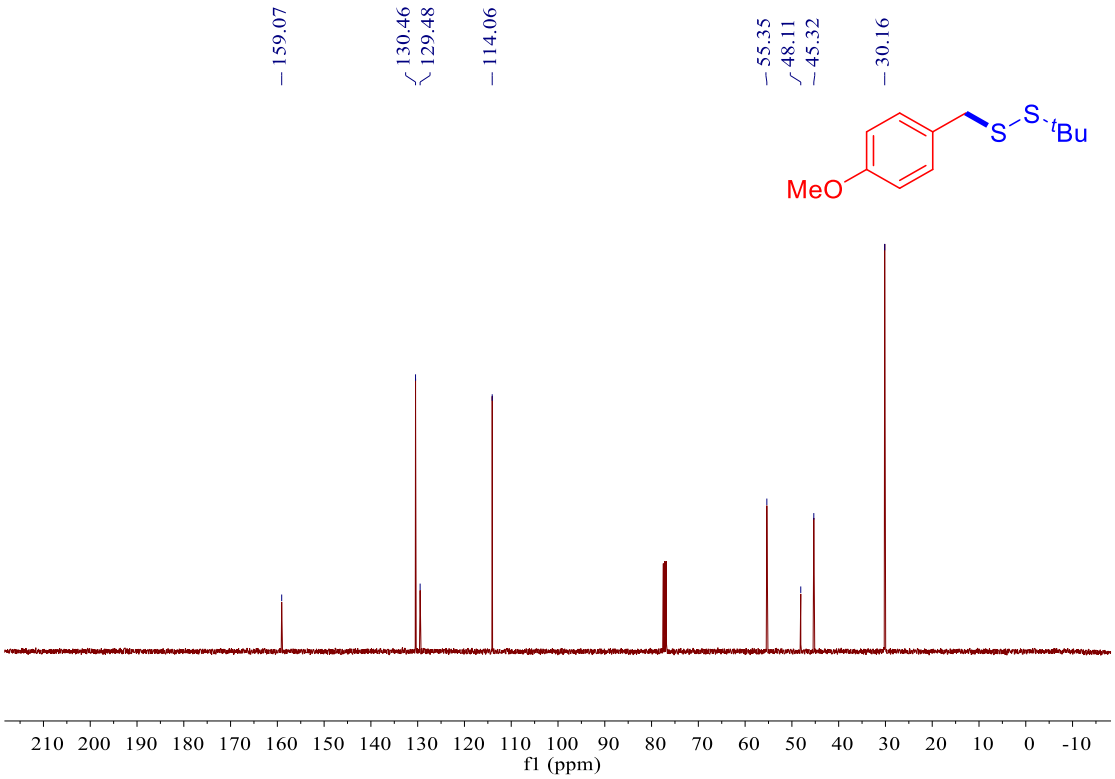
^{13}C NMR Spectra of **5d** (400 MHz, $CDCl_3$)



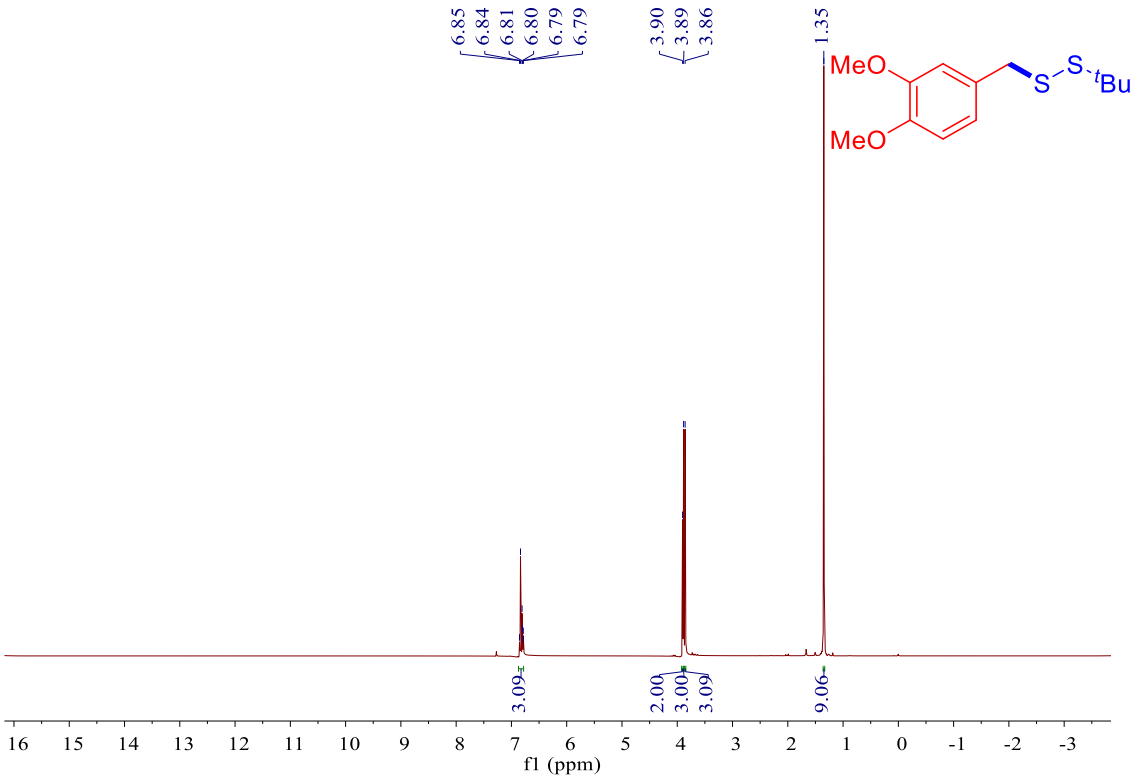
^{1}H NMR Spectra of **5e** (400 MHz, $CDCl_3$)



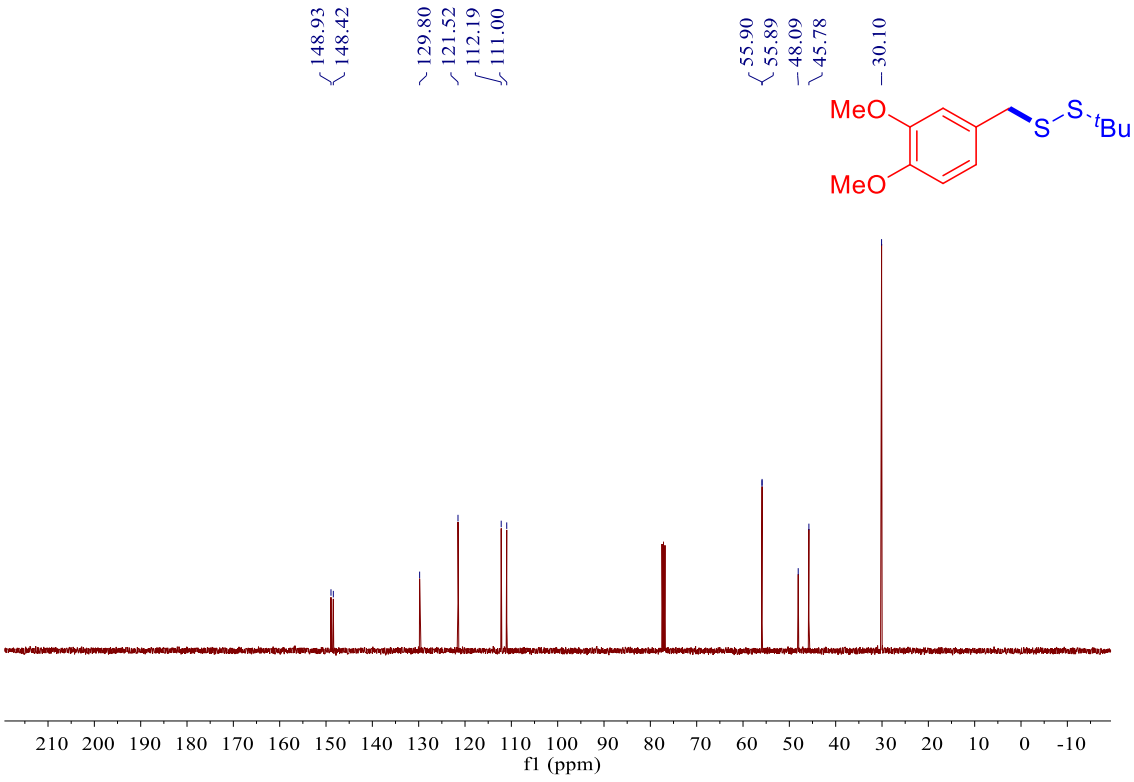
^{13}C NMR Spectra of **5e** (400 MHz, $CDCl_3$)



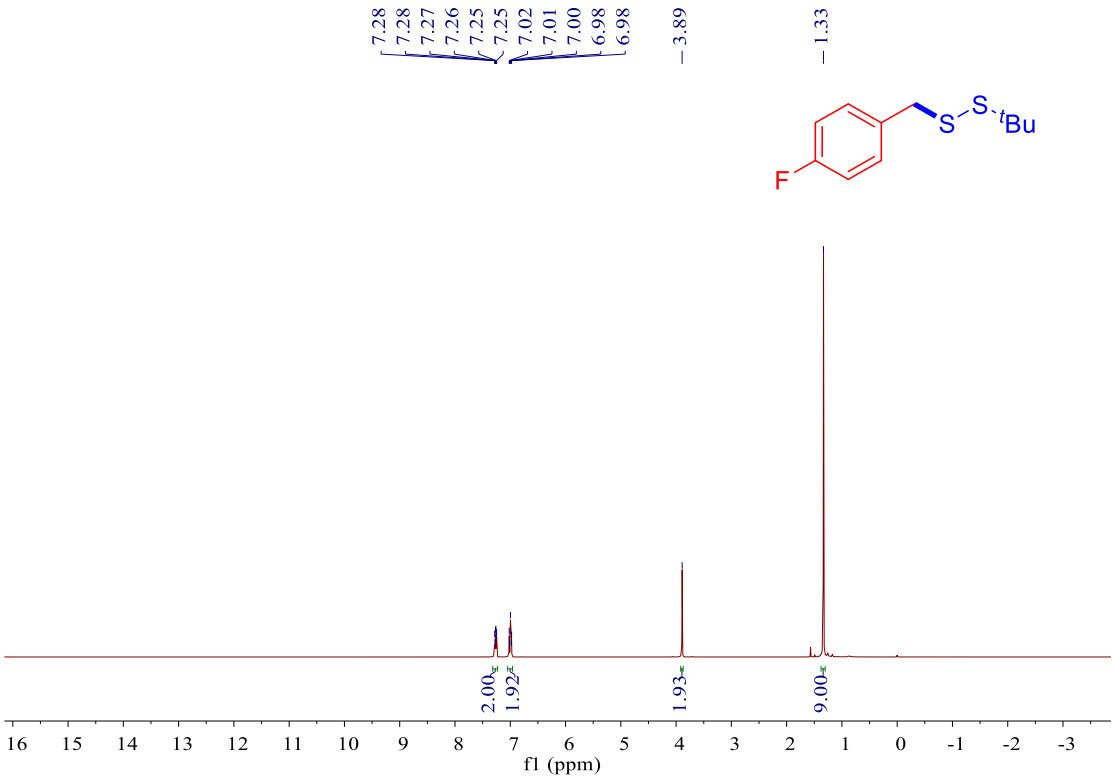
^{1}H NMR Spectra of **5f** (400 MHz, $CDCl_3$)



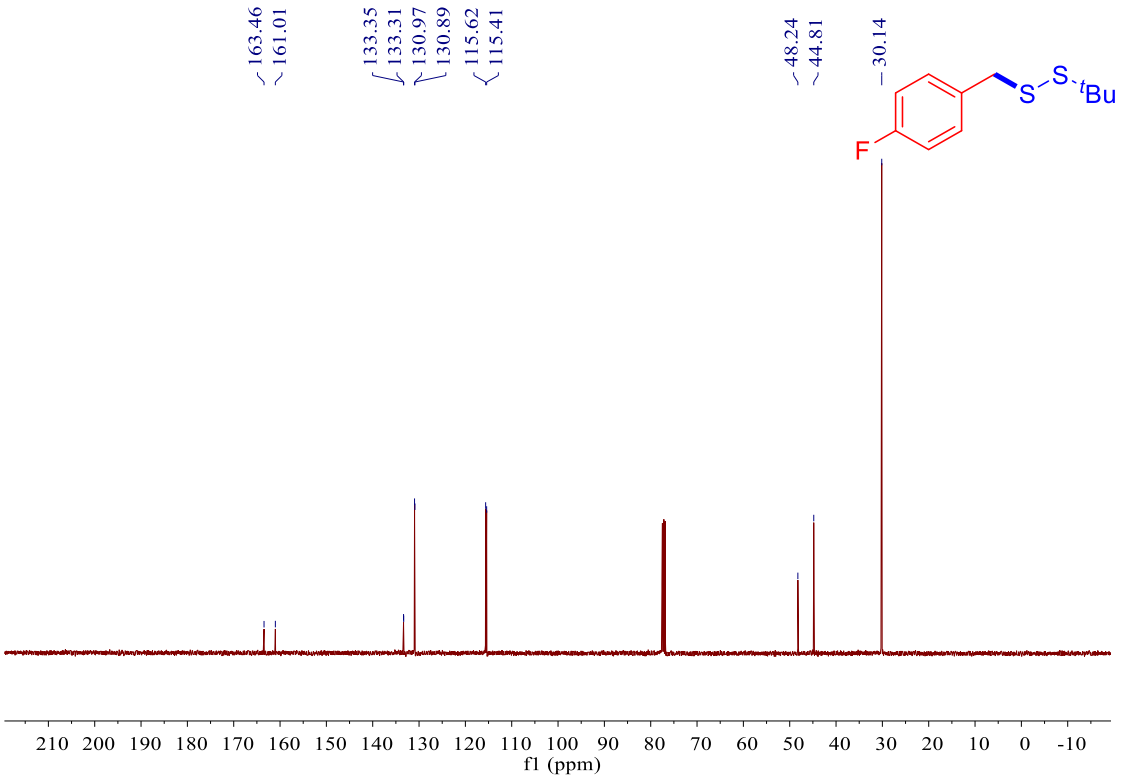
^{13}C NMR Spectra of **5f** (400 MHz, $CDCl_3$)



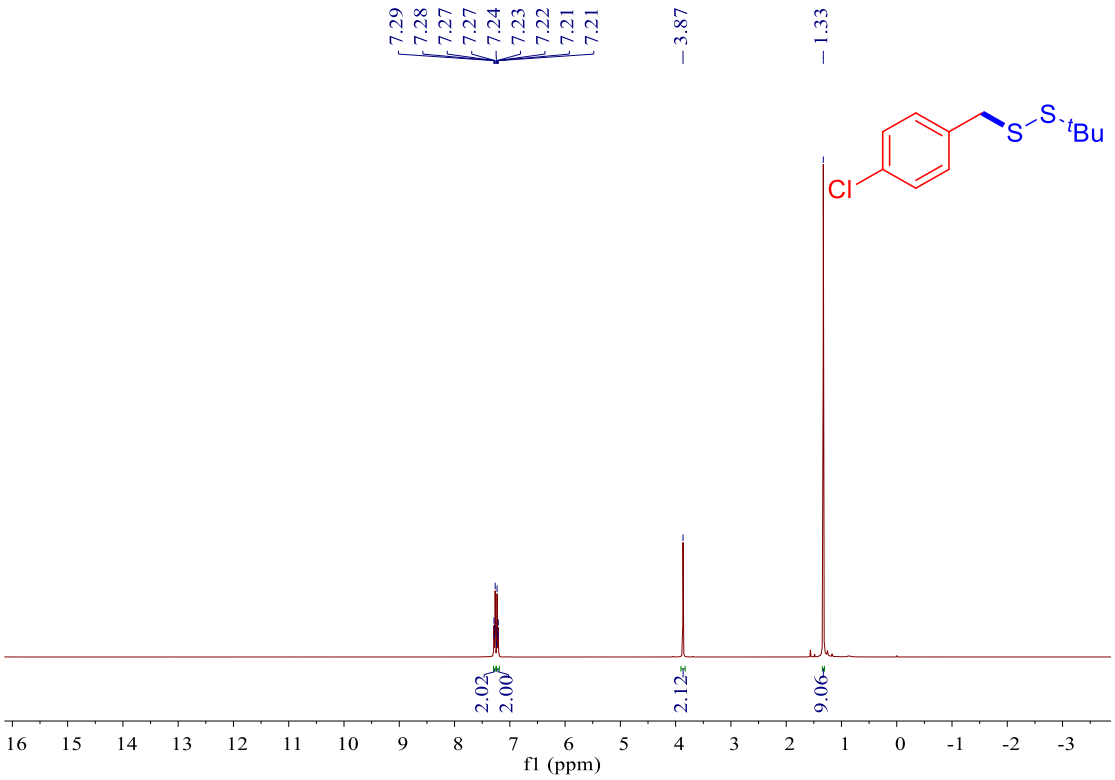
^{1}H NMR Spectra of **5g** (400 MHz, $CDCl_3$)



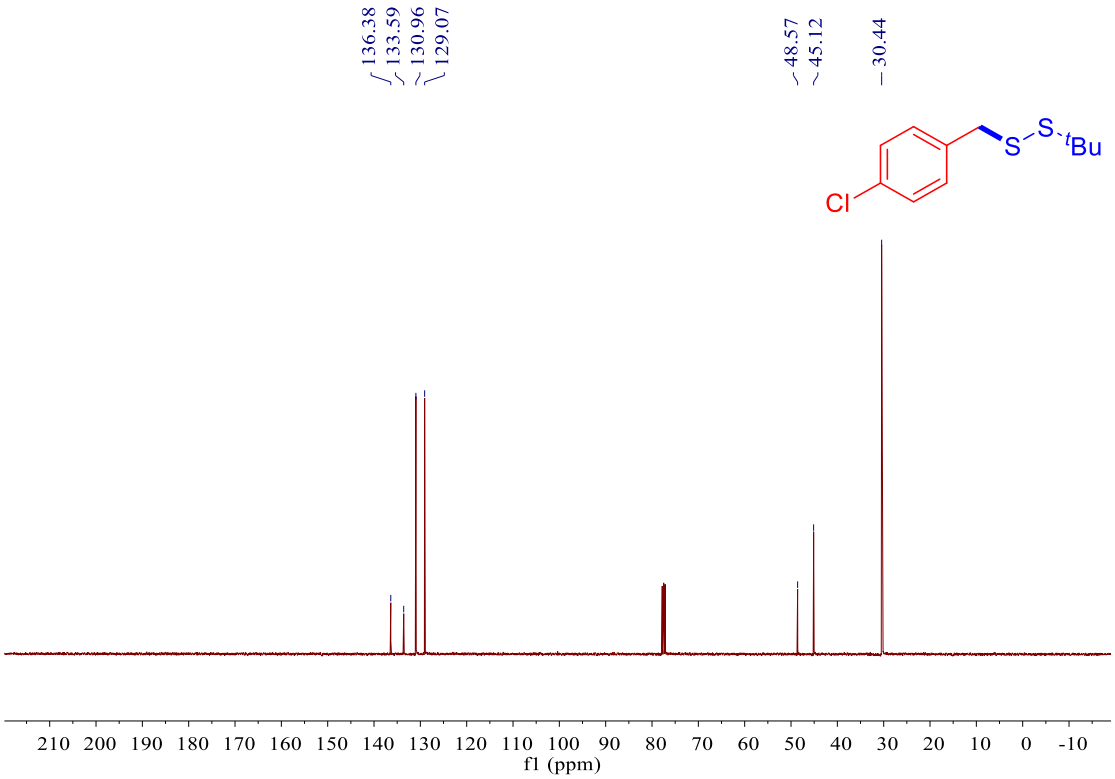
^{13}C NMR Spectra of **5g** (400 MHz, $CDCl_3$)



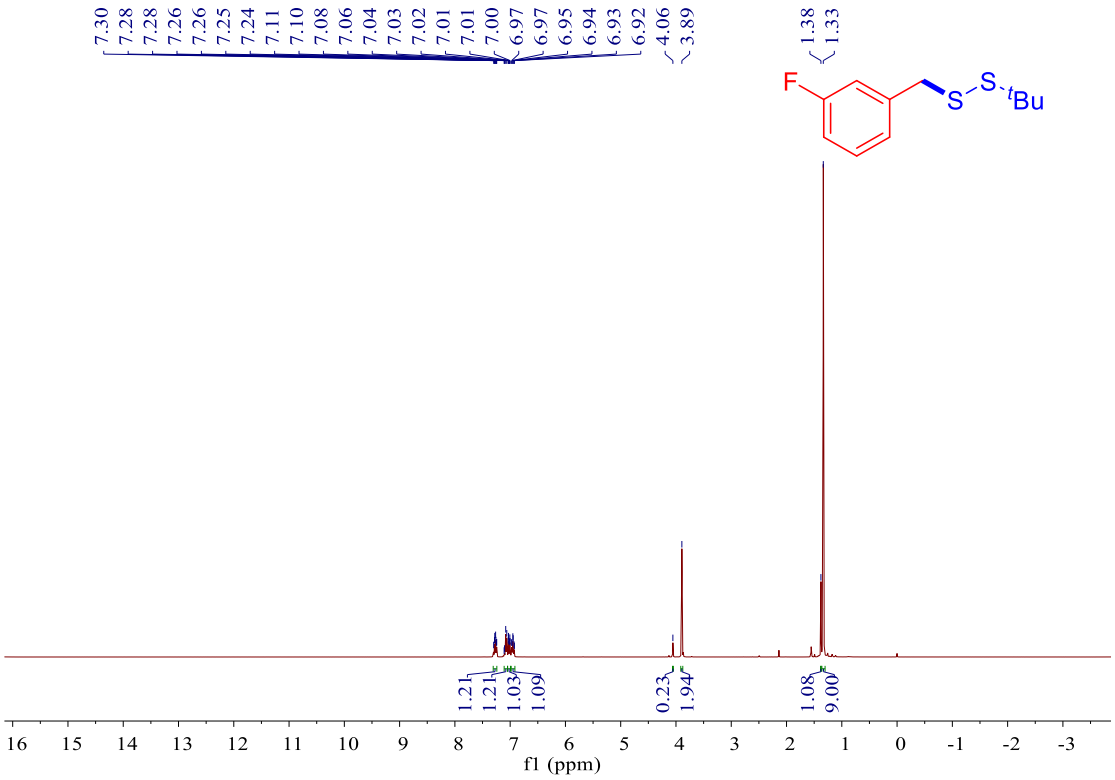
^{1}H NMR Spectra of **5h** (400 MHz, $CDCl_3$)



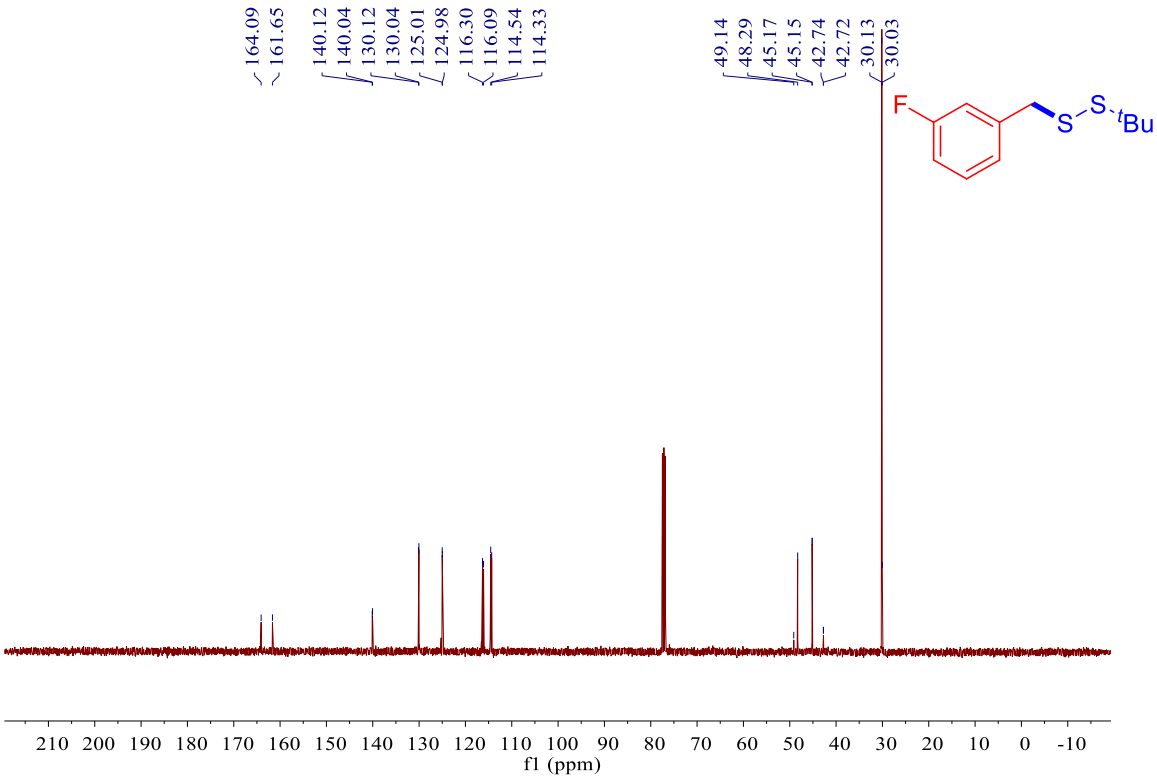
^{13}C NMR Spectra of **5h** (400 MHz, $CDCl_3$)



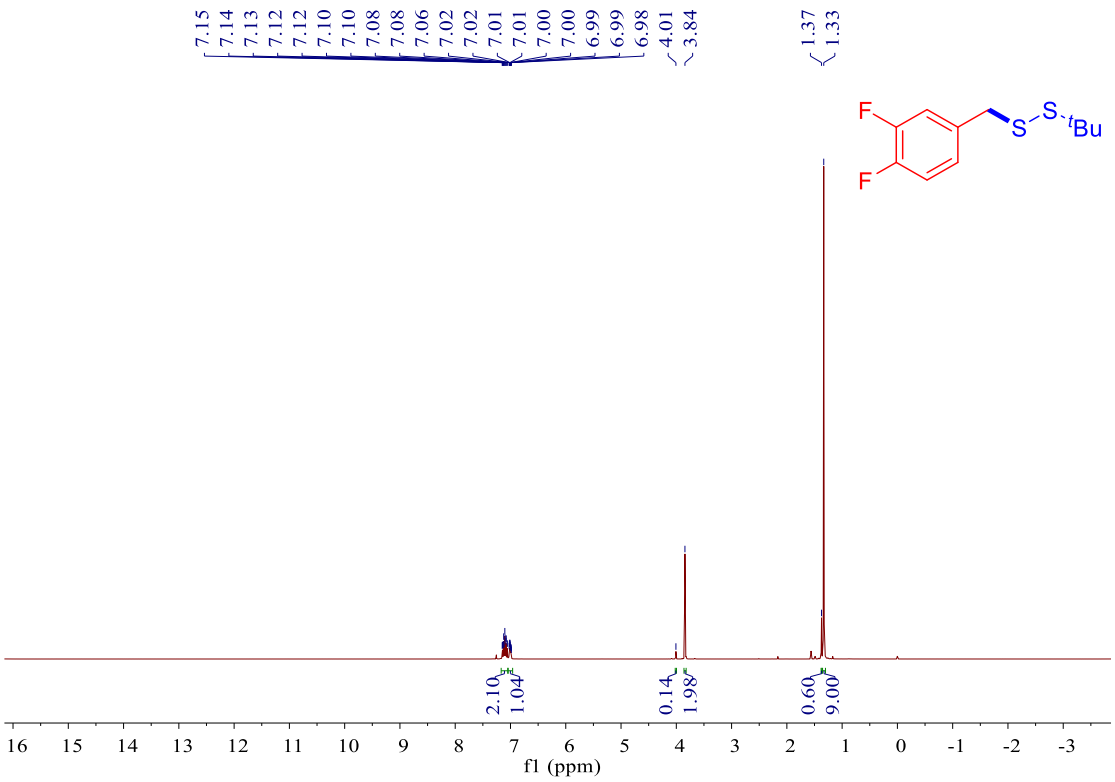
^{1}H NMR Spectra of **5i** (400 MHz, $CDCl_3$)



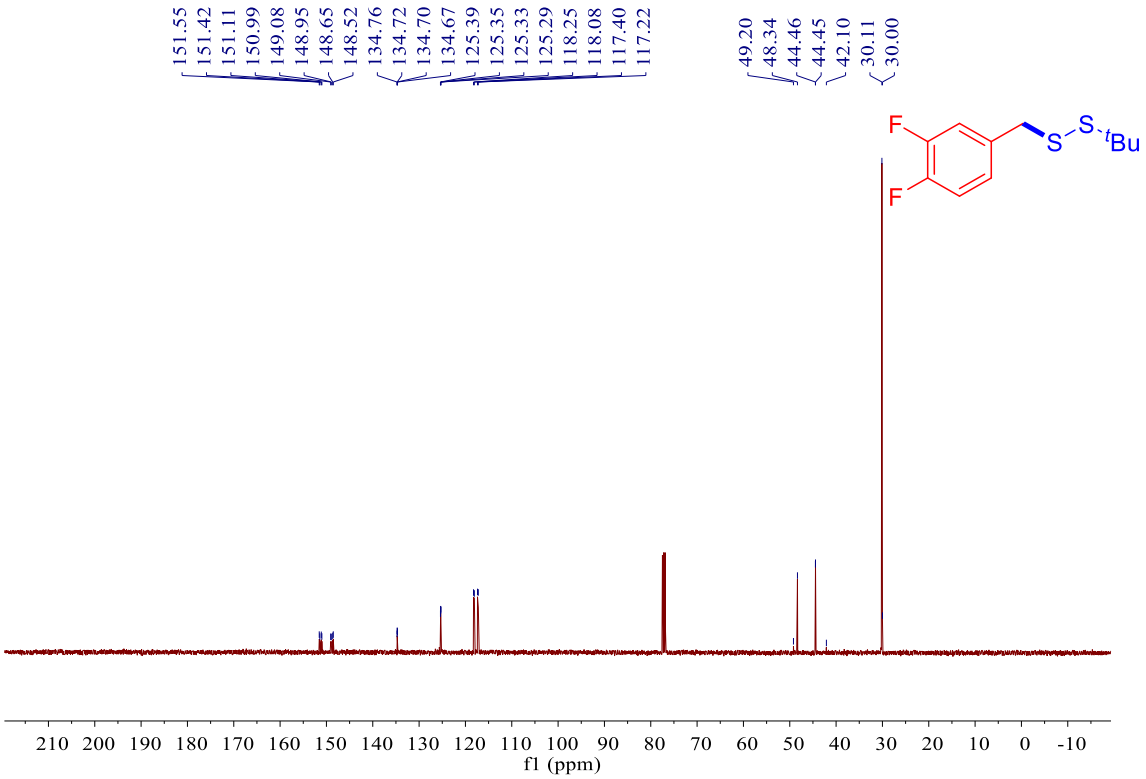
^{13}C NMR Spectra of **5i** (400 MHz, $CDCl_3$)



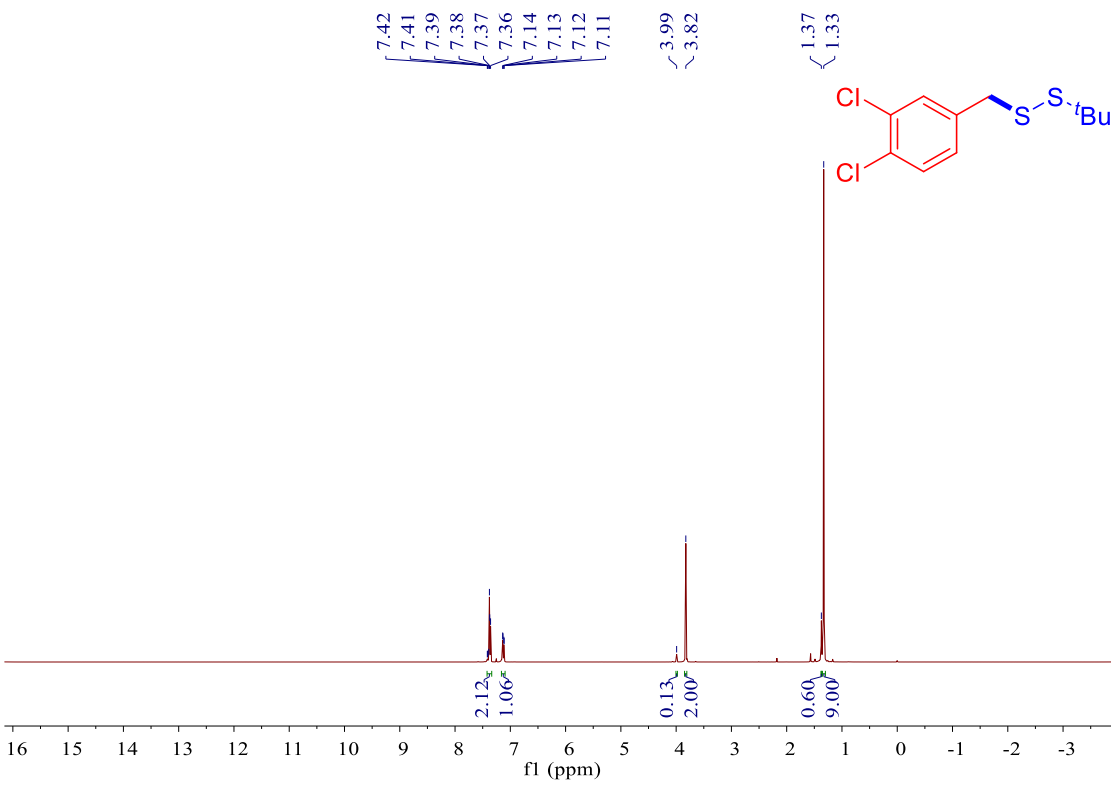
^{1}H NMR Spectra of **5j** (400 MHz, $CDCl_3$)



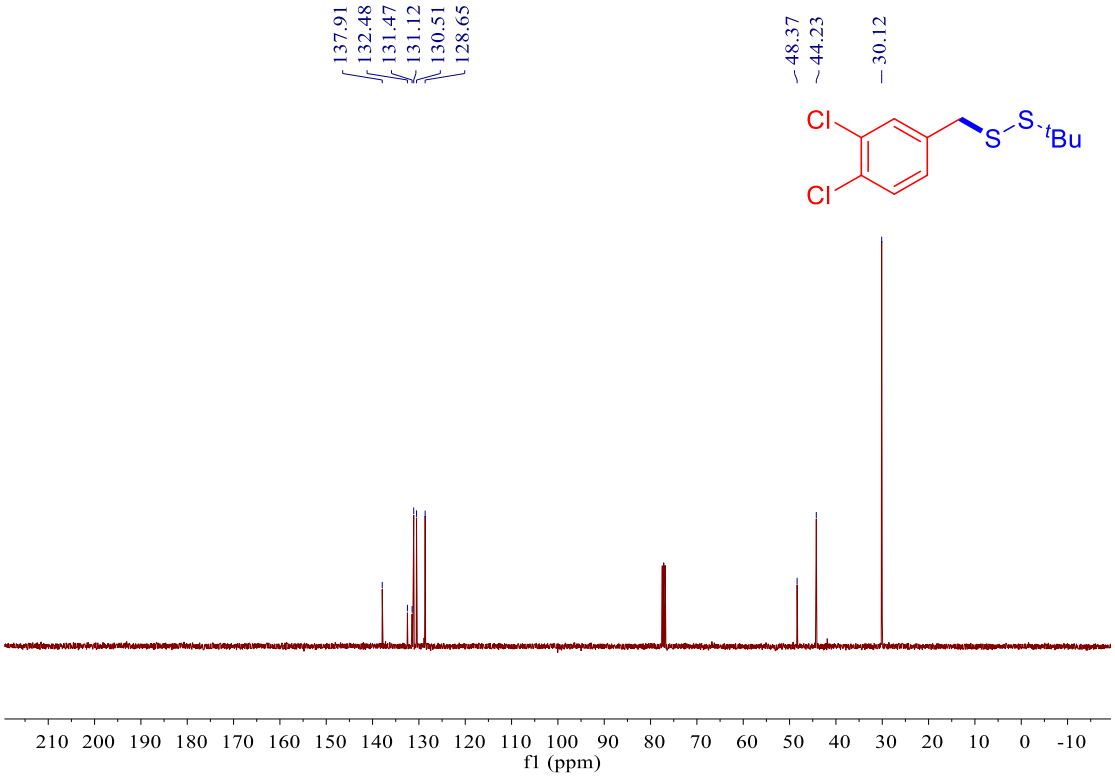
^{13}C NMR Spectra of **5j** (400 MHz, $CDCl_3$)



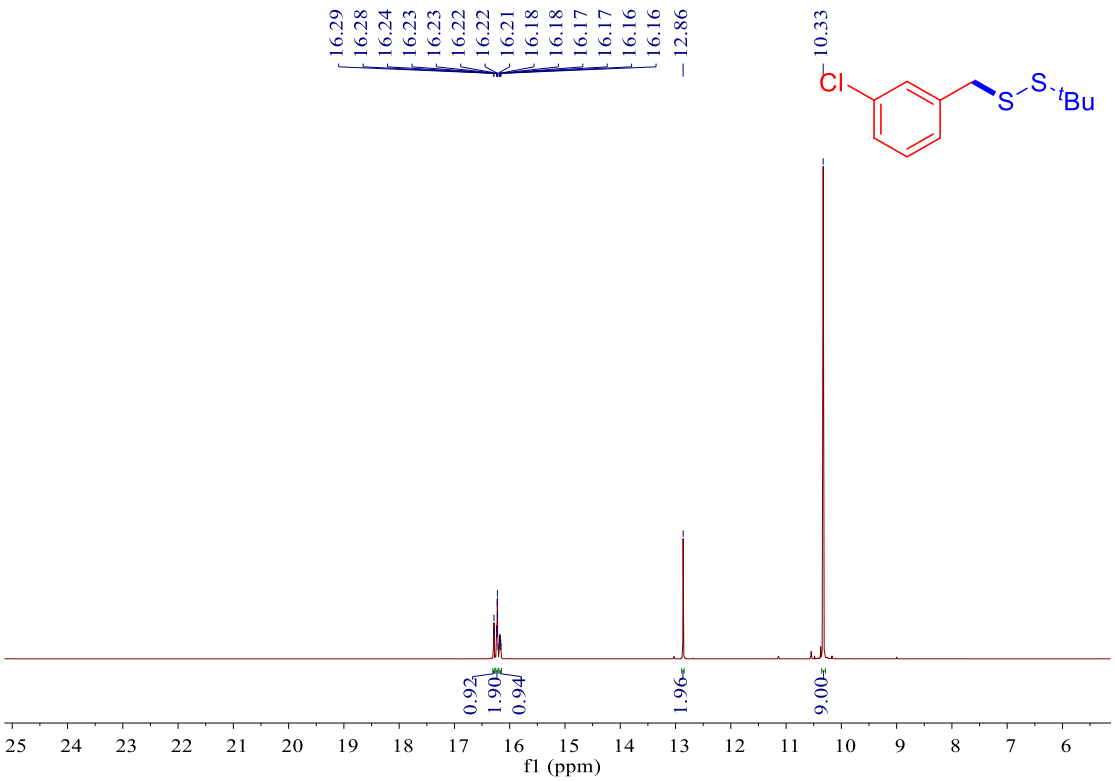
^{1}H NMR Spectra of **5k** (400 MHz, $CDCl_3$)



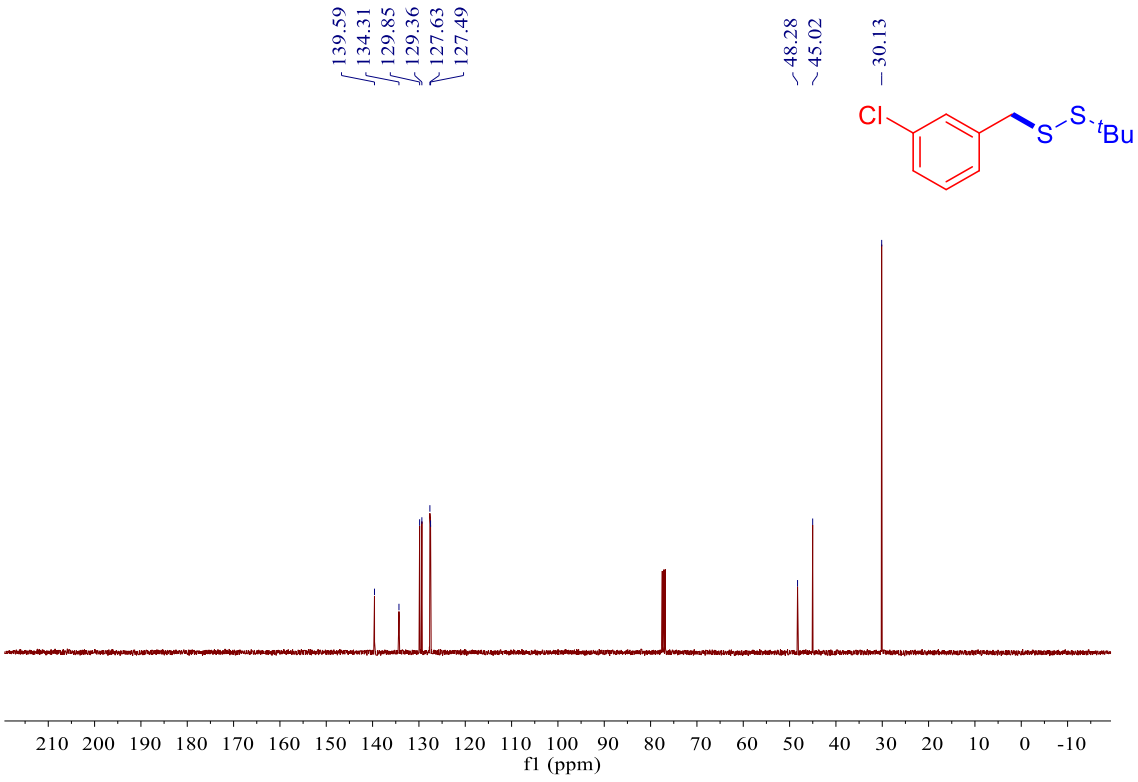
^{13}C NMR Spectra of **5k** (400 MHz, $CDCl_3$)



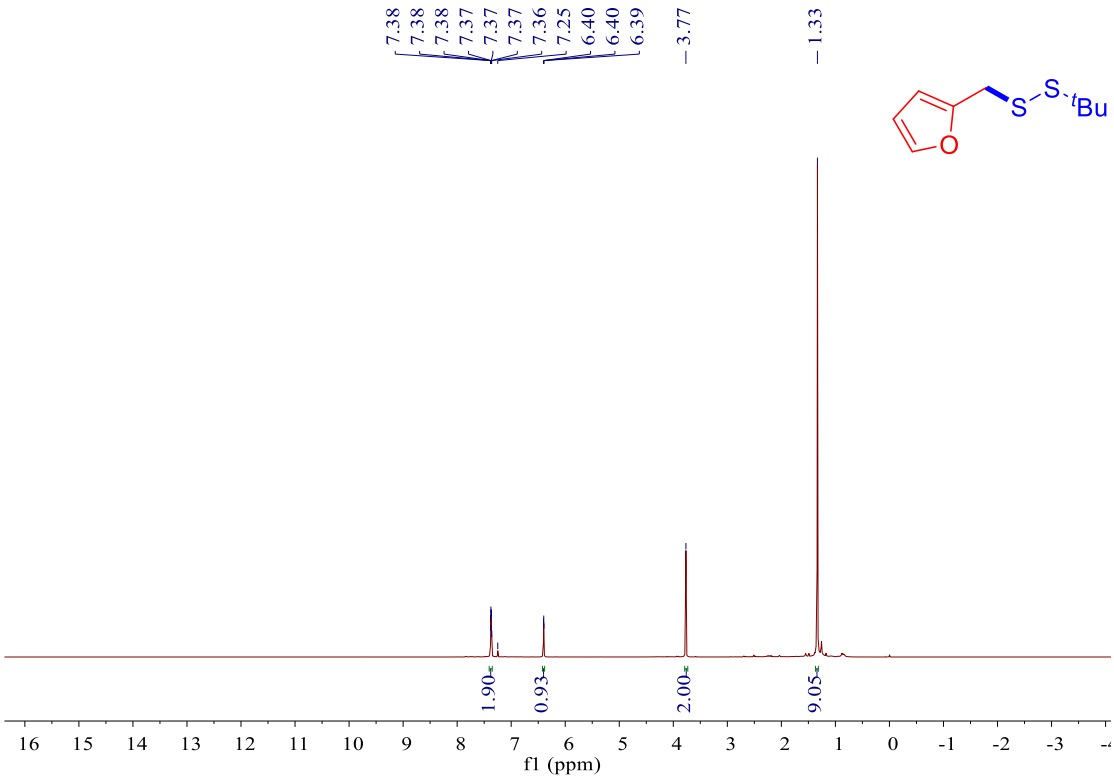
^{1}H NMR Spectra of **5l** (400 MHz, $CDCl_3$)



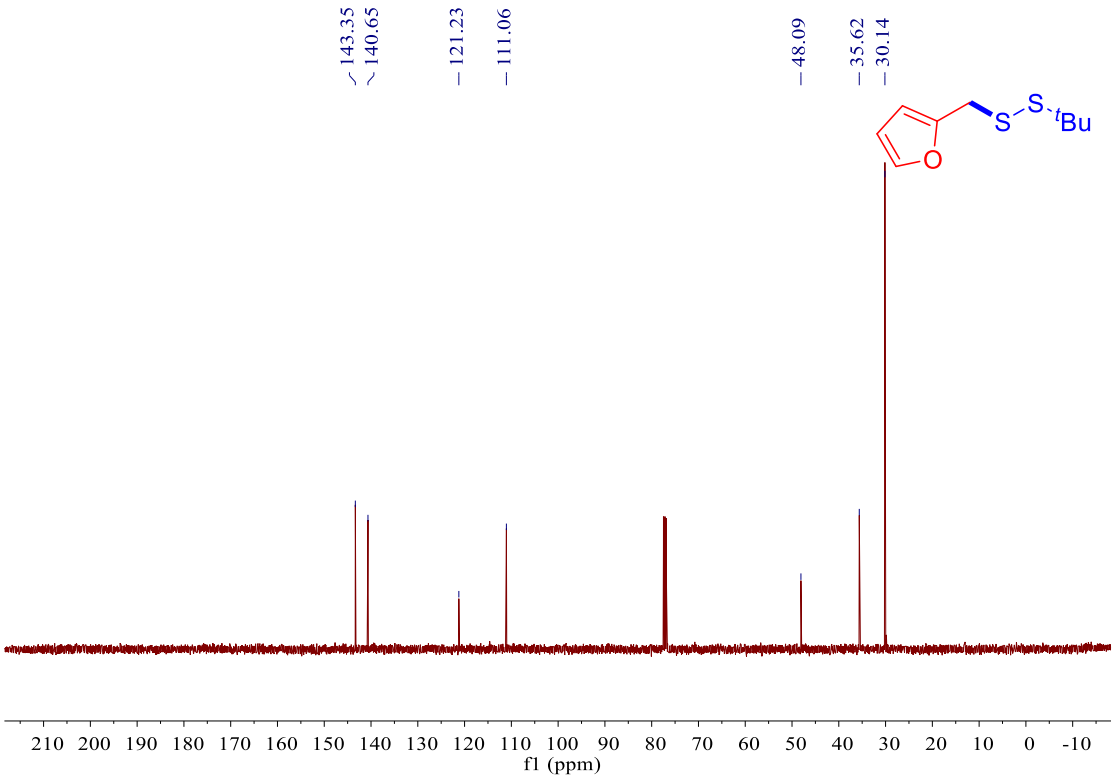
^{13}C NMR Spectra of **5l** (400 MHz, $CDCl_3$)



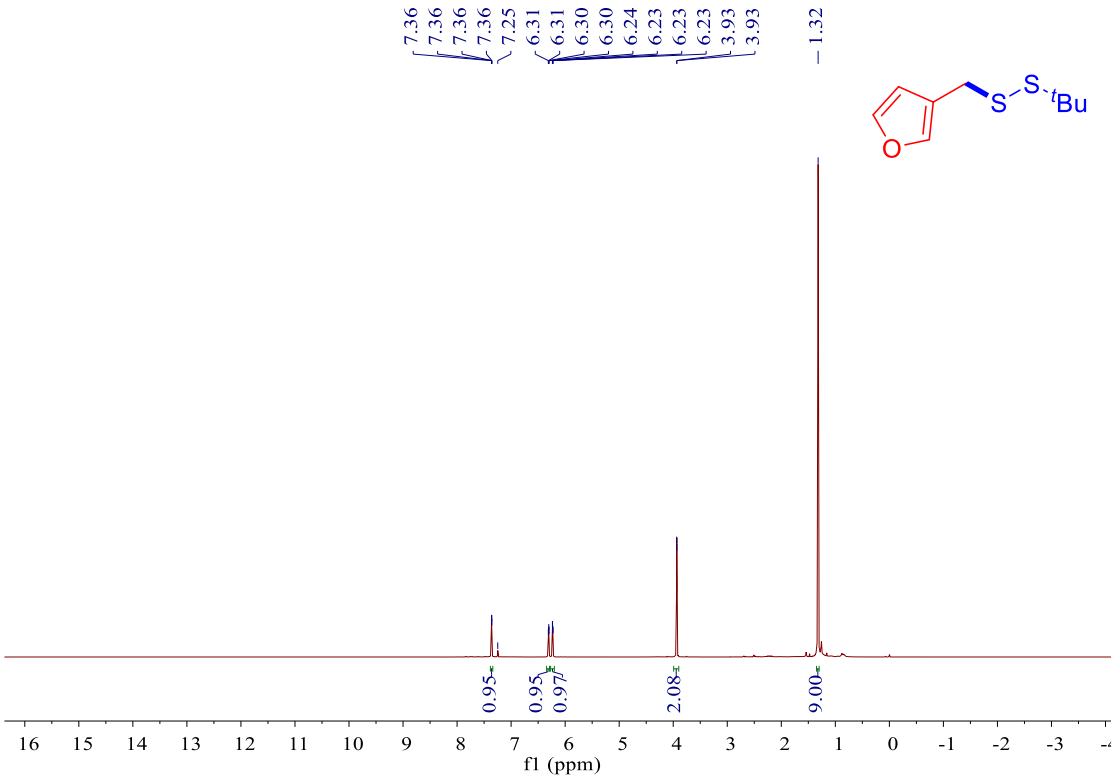
^{1}H NMR Spectra of **5m** (400 MHz, $CDCl_3$)



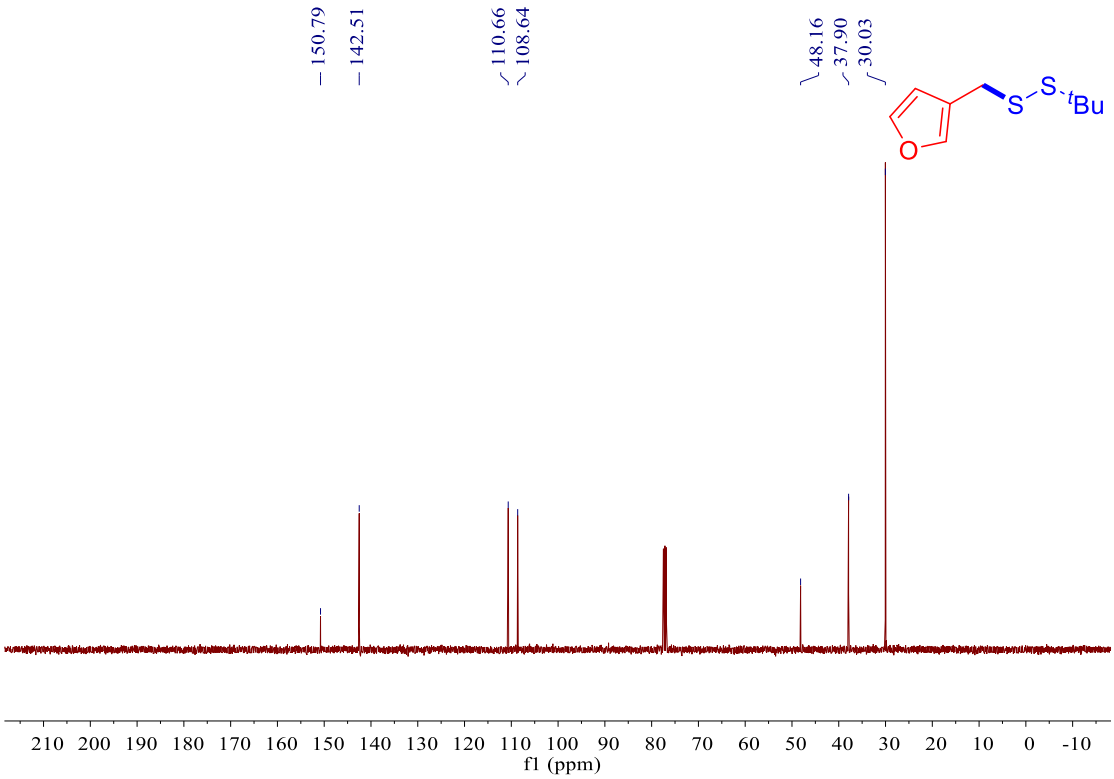
^{13}C NMR Spectra of **5m** (400 MHz, $CDCl_3$)



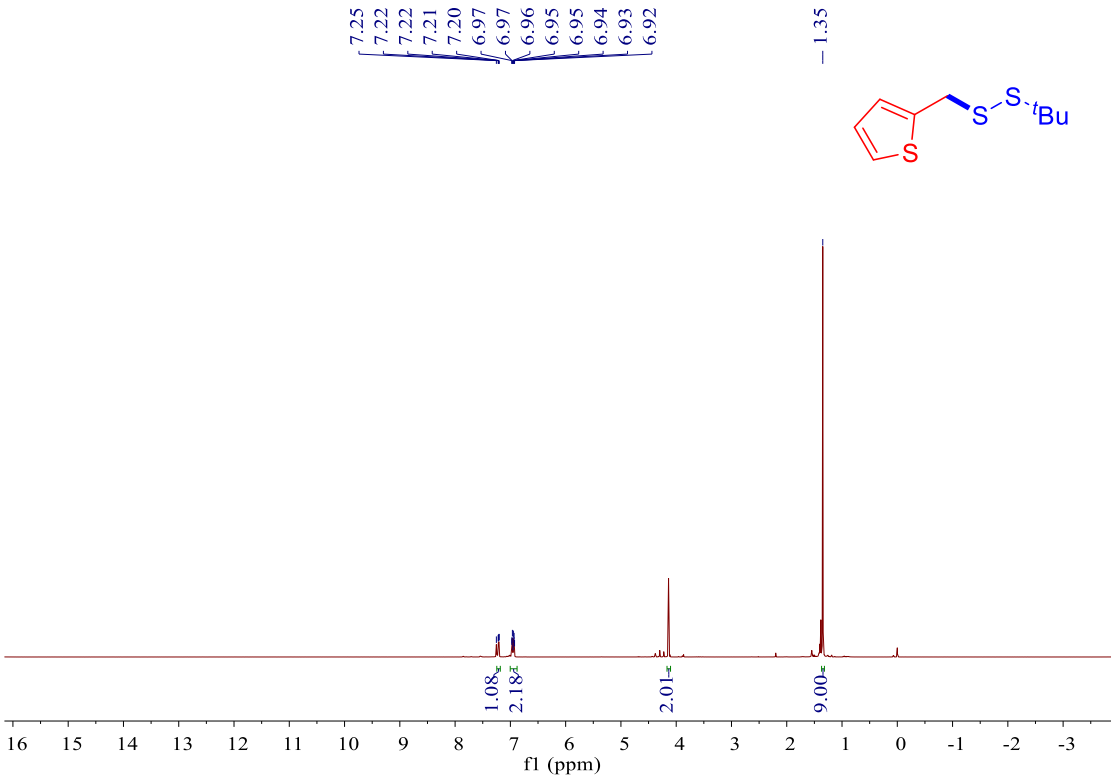
^{1}H NMR Spectra of **5n** (400 MHz, $CDCl_3$)



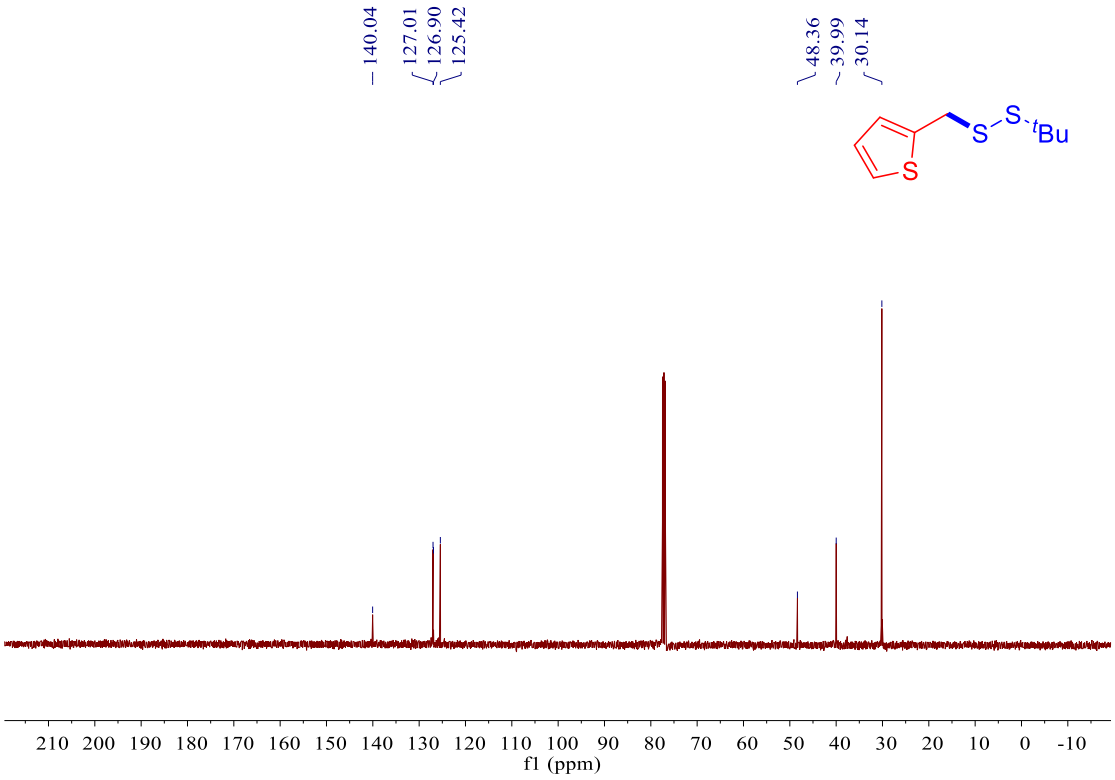
^{13}C NMR Spectra of **5n** (400 MHz, $CDCl_3$)



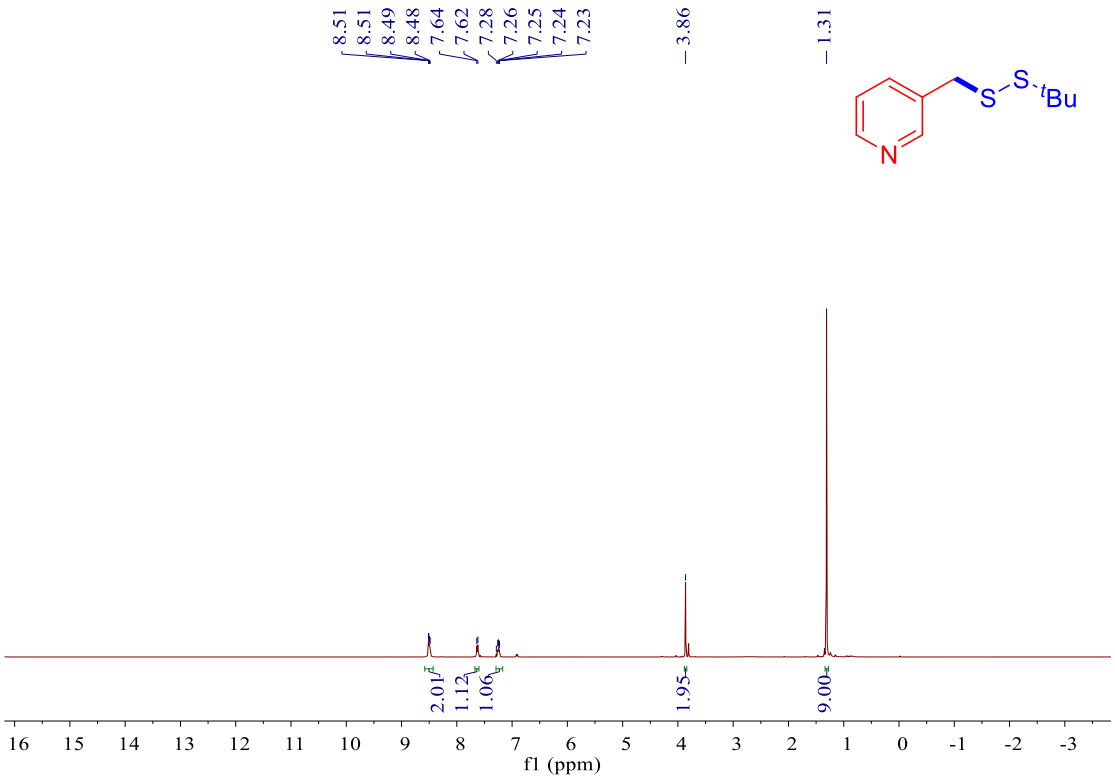
^{1}H NMR Spectra of **5o** (400 MHz, $CDCl_3$)



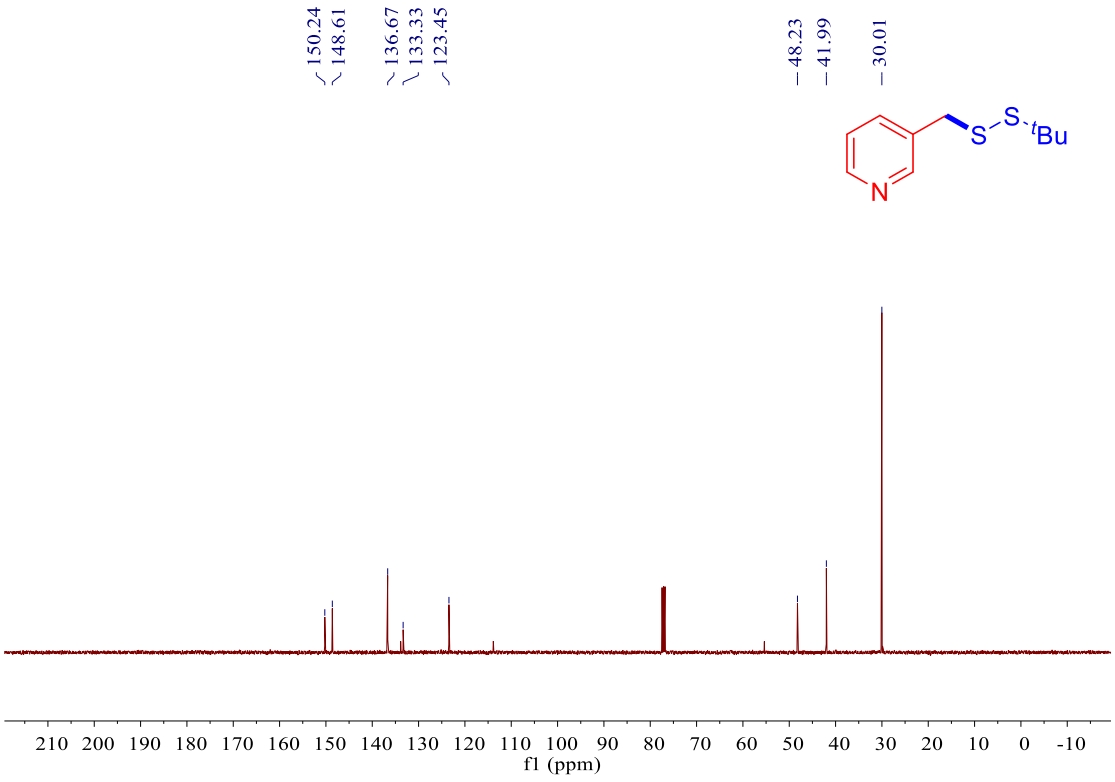
^{13}C NMR Spectra of **5o** (400 MHz, $CDCl_3$)



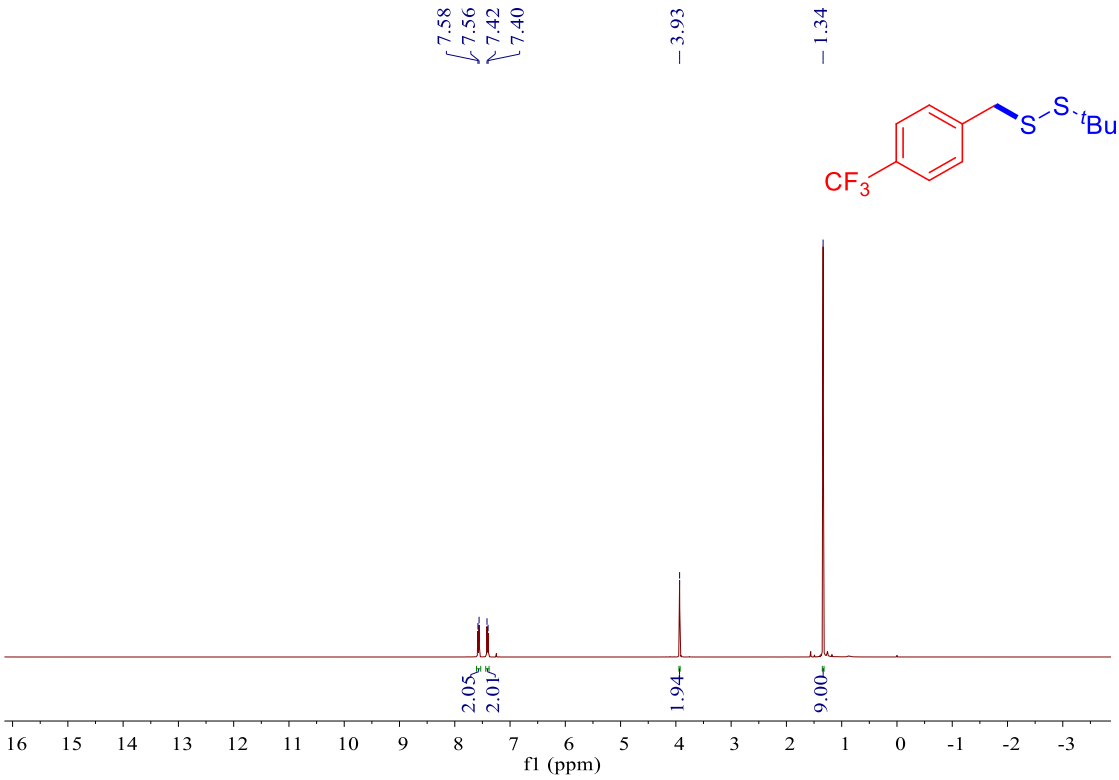
^{1}H NMR Spectra of **5p** (400 MHz, $CDCl_3$)



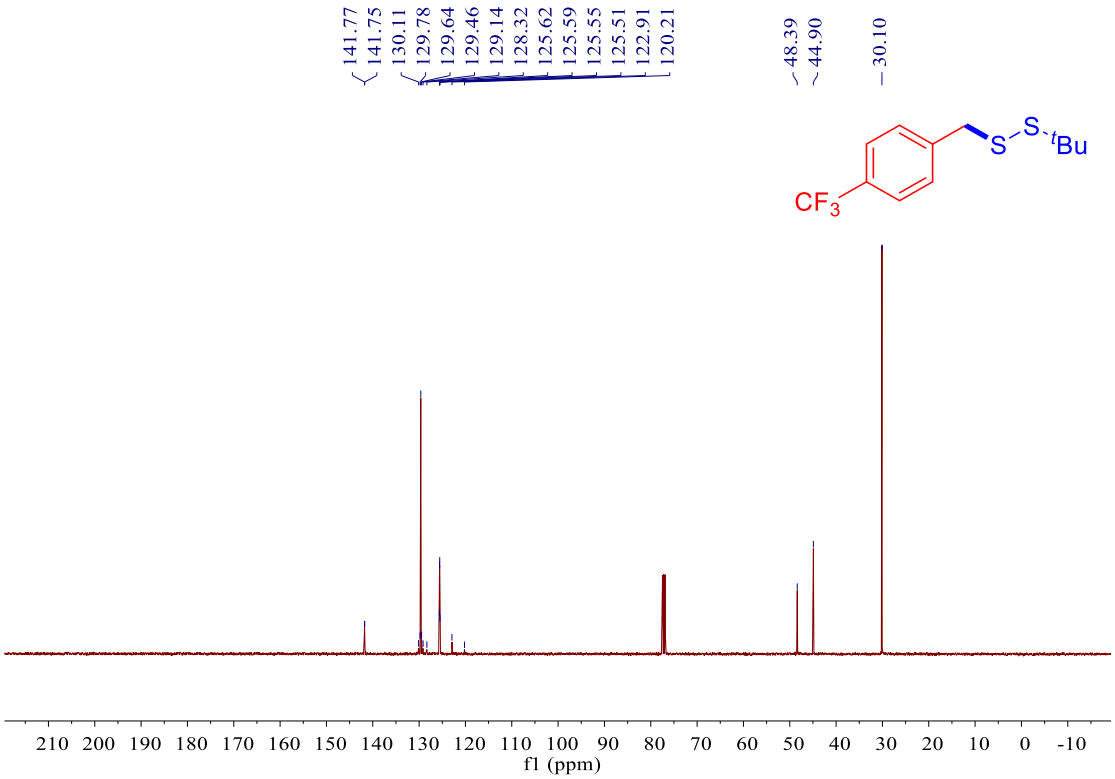
^{13}C NMR Spectra of **5p** (400 MHz, $CDCl_3$)



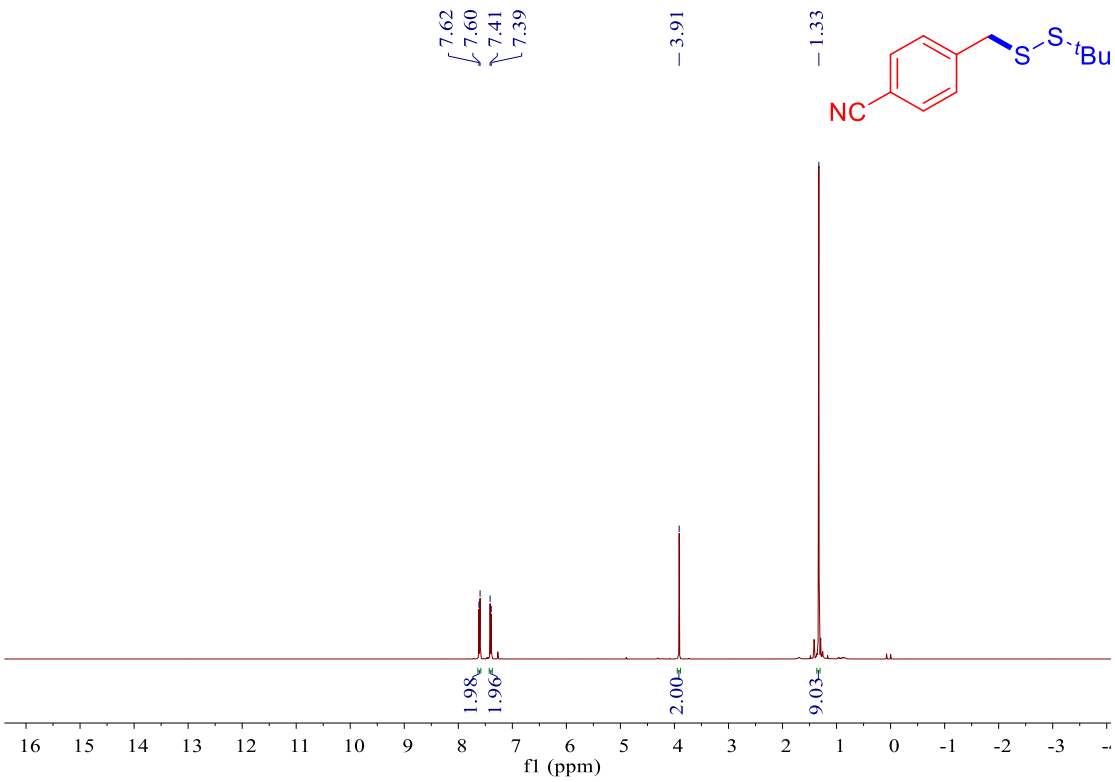
^{1}H NMR Spectra of **5q** (400 MHz, $CDCl_3$)



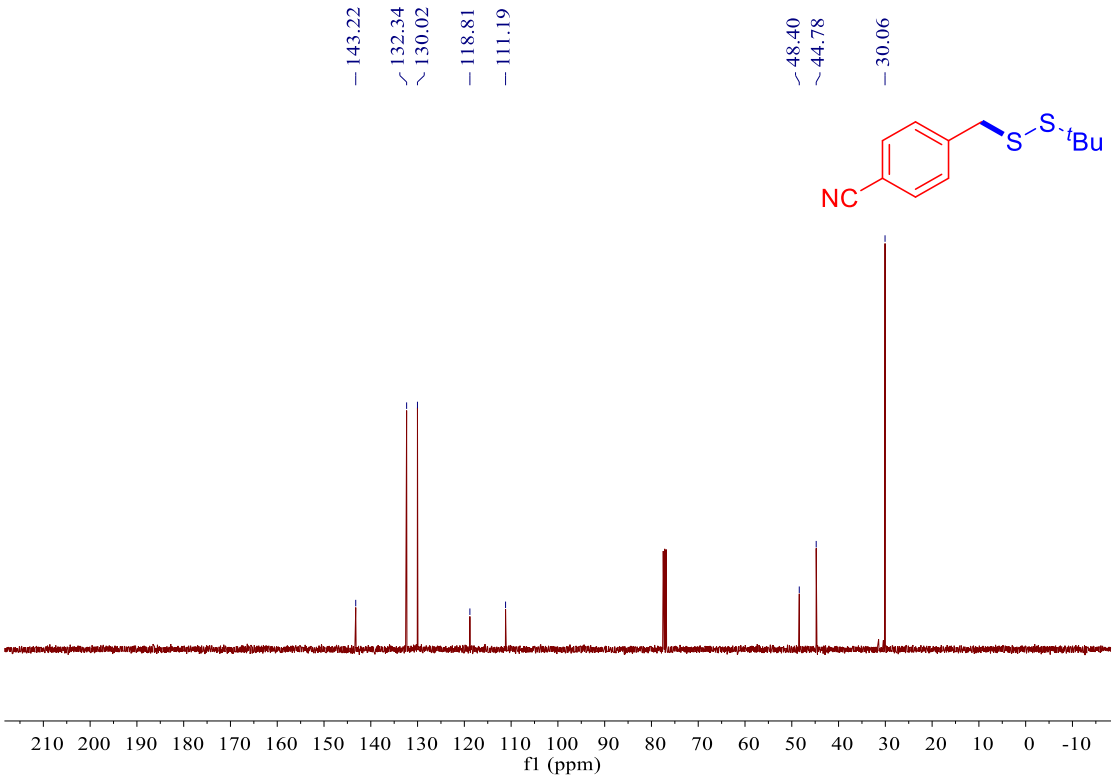
^{13}C NMR Spectra of **5q** (400 MHz, $CDCl_3$)



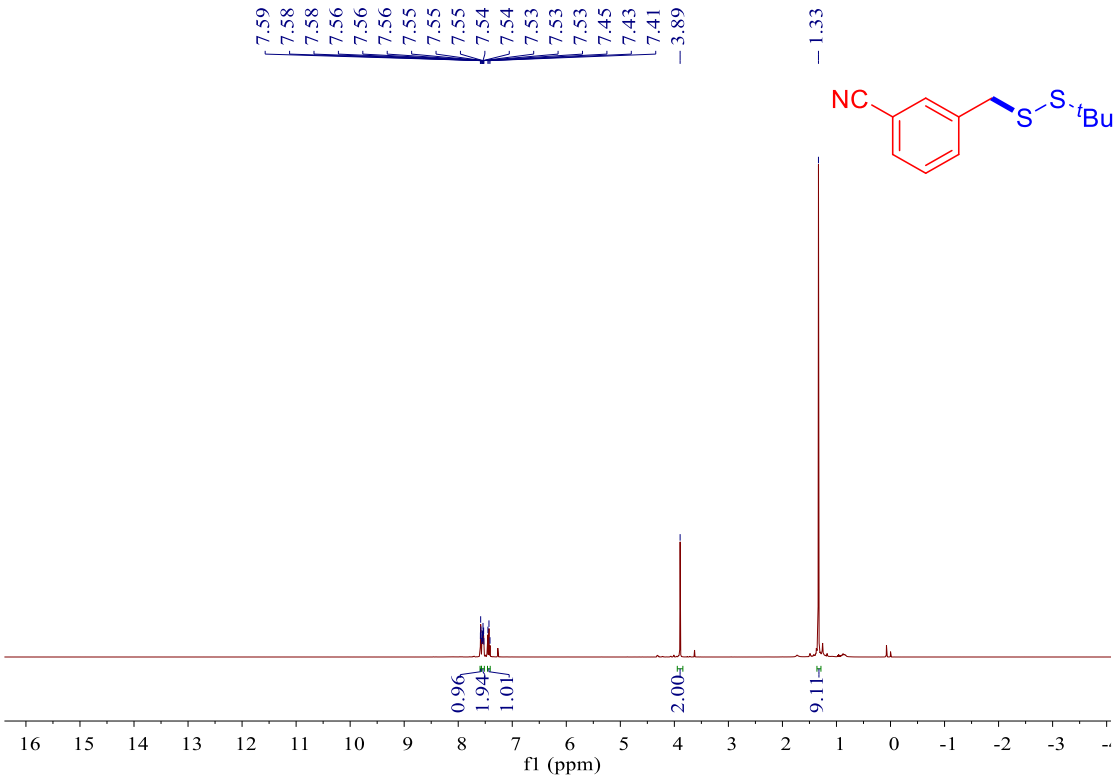
^{1}H NMR Spectra of **5r** (400 MHz, $CDCl_3$)



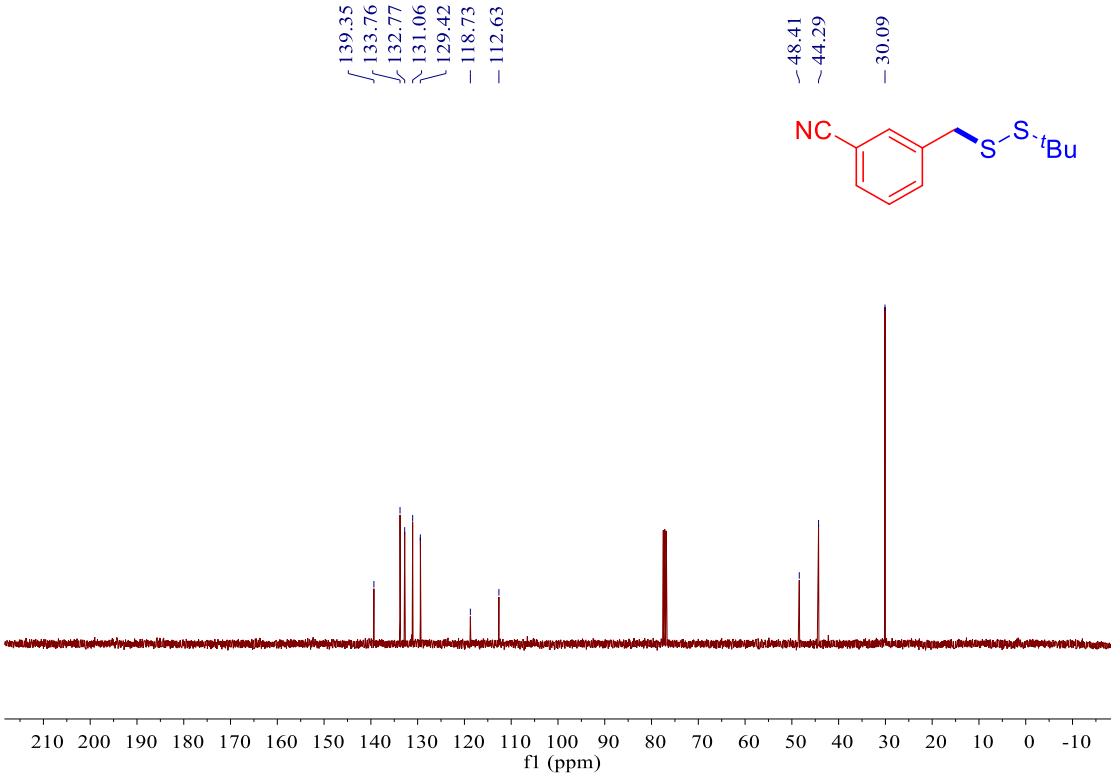
^{13}C NMR Spectra of **5r** (400 MHz, $CDCl_3$)



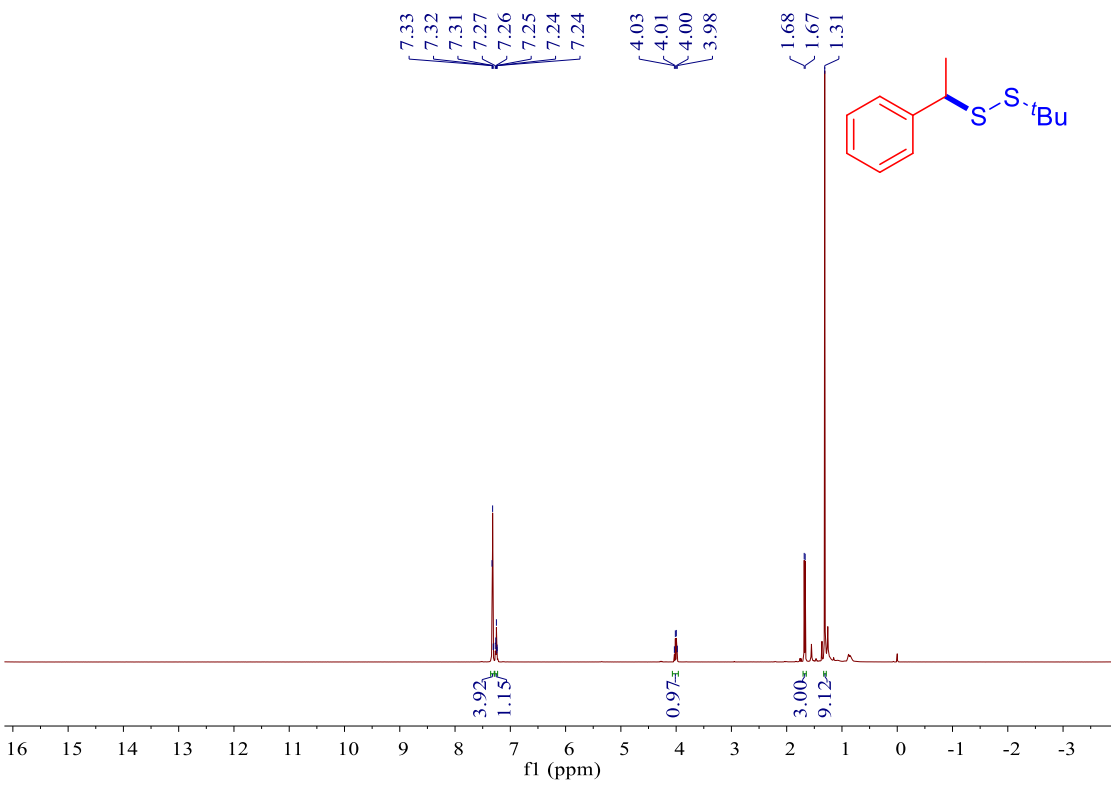
^{1}H NMR Spectra of **5s** (400 MHz, $CDCl_3$)



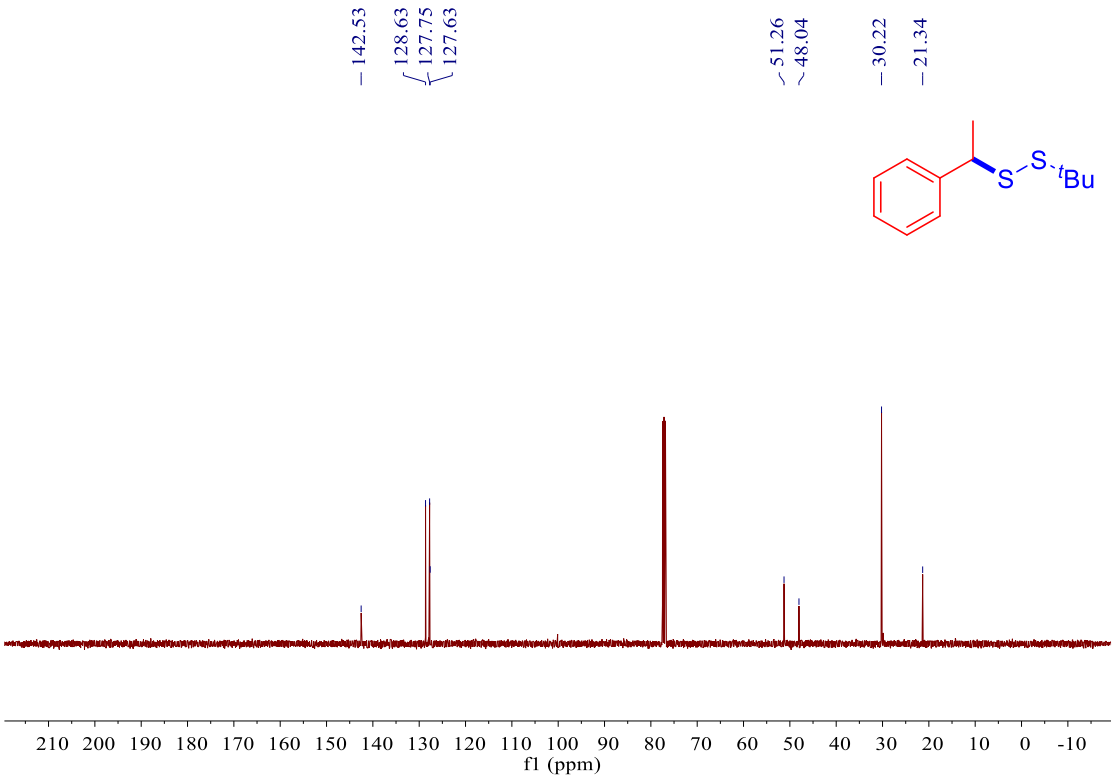
^{13}C NMR Spectra of **5s** (400 MHz, $CDCl_3$)



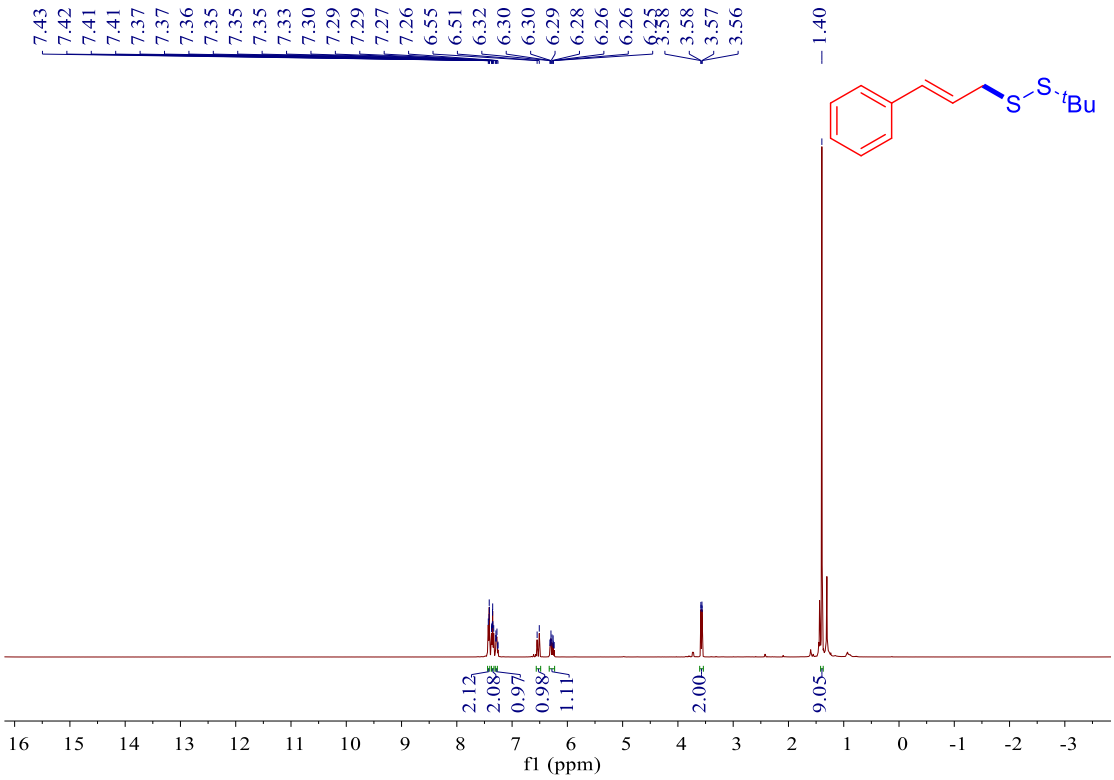
^{1}H NMR Spectra of **5t** (400 MHz, $CDCl_3$)



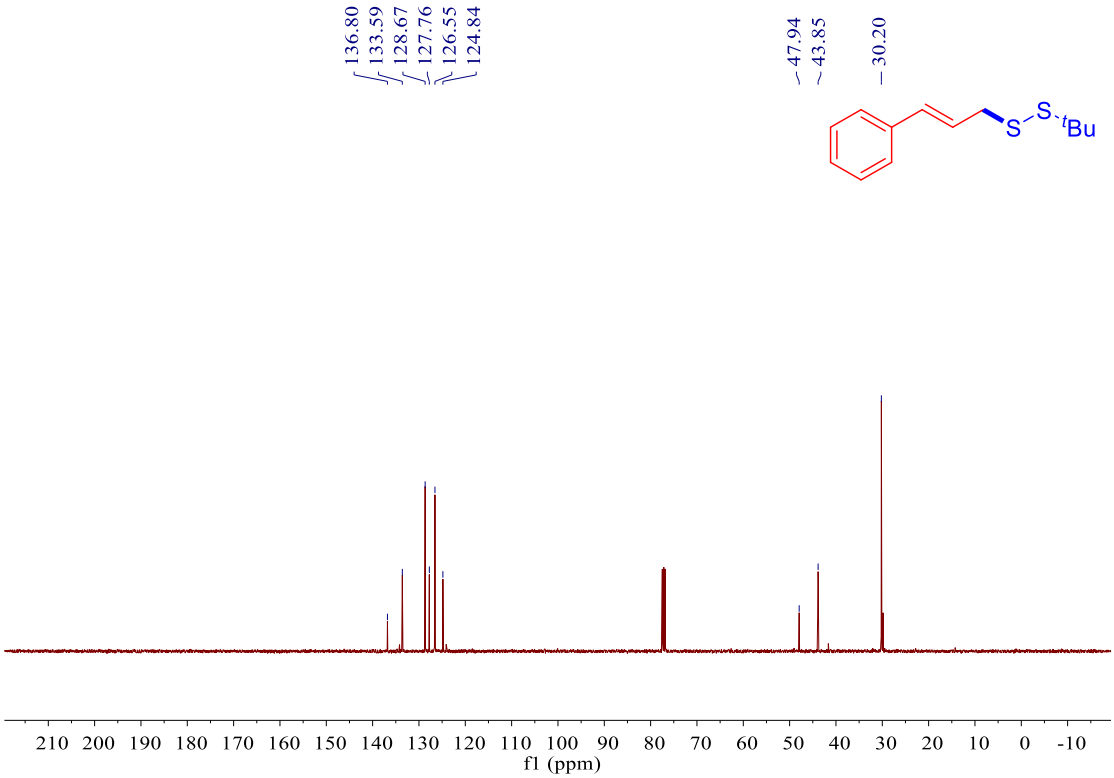
^{13}C NMR Spectra of **5t** (400 MHz, $CDCl_3$)



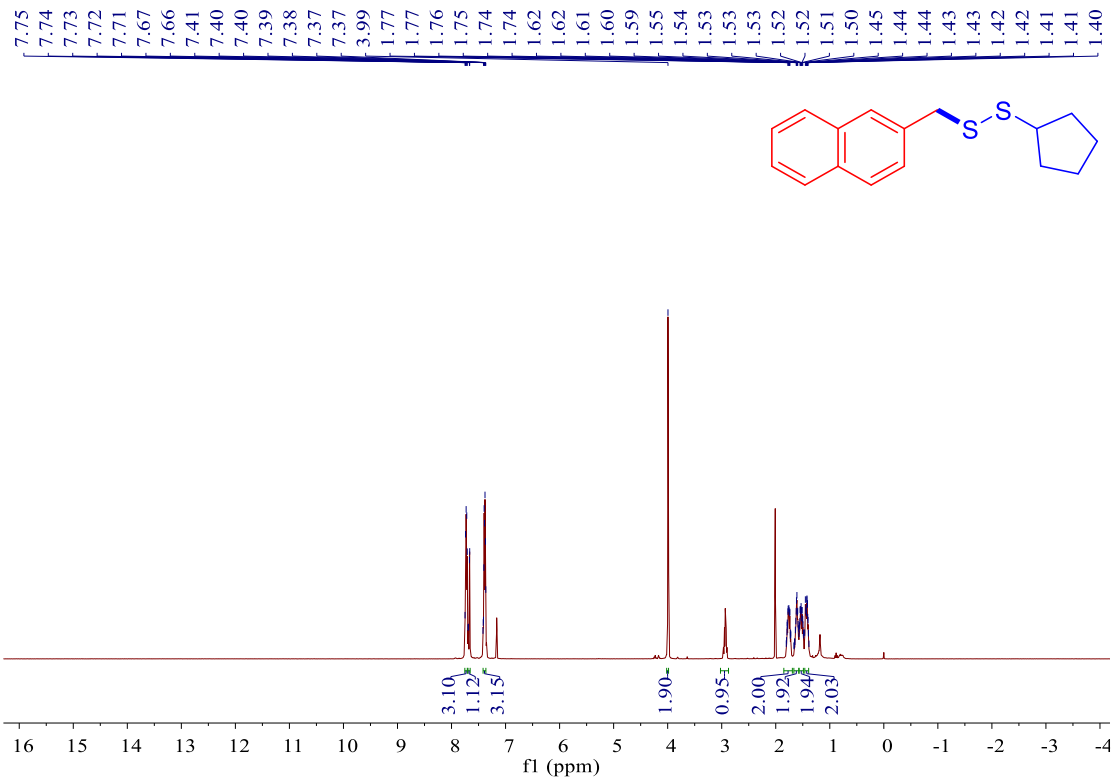
^{1}H NMR Spectra of **5v** (400 MHz, $CDCl_3$)



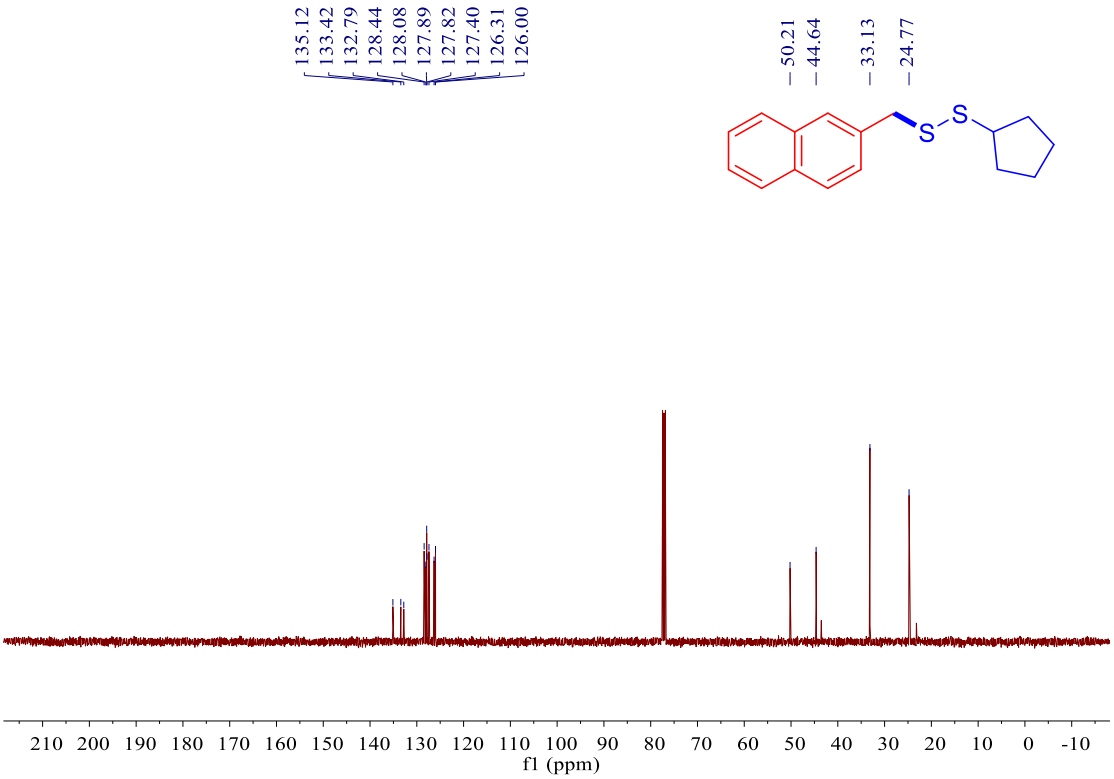
^{13}C NMR Spectra of **5v** (400 MHz, $CDCl_3$)



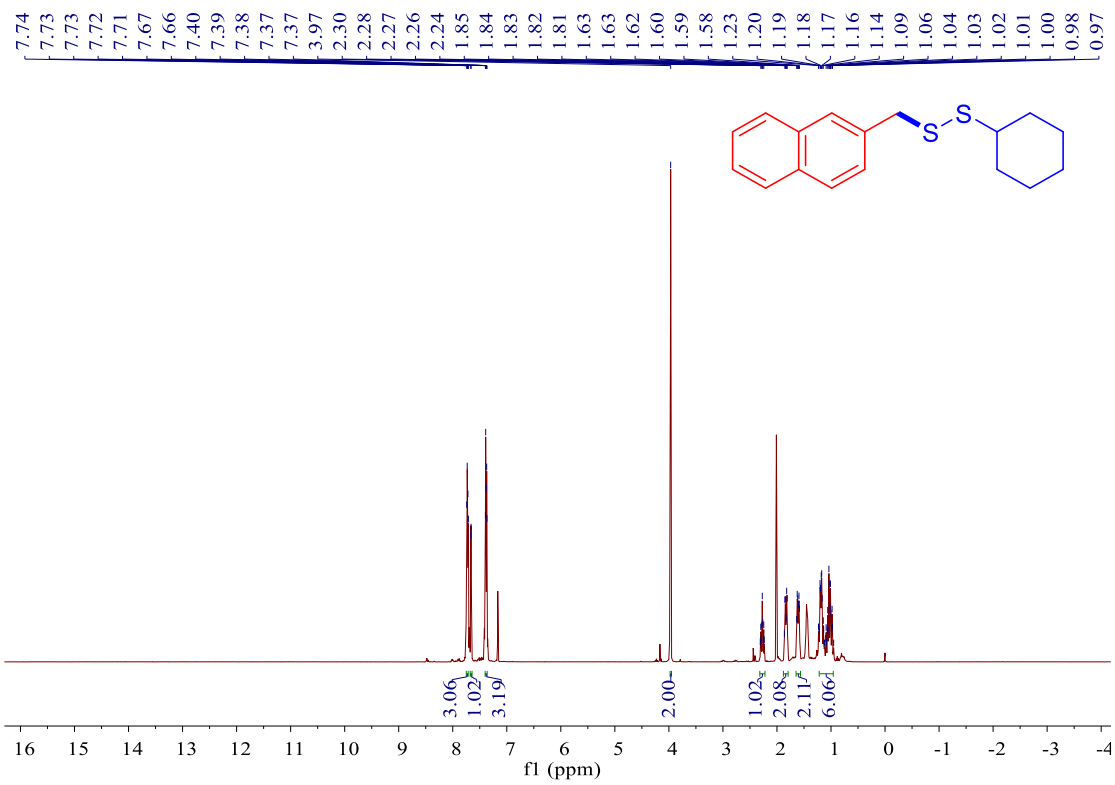
^{1}H NMR Spectra of **6a** (400 MHz, $CDCl_3$)



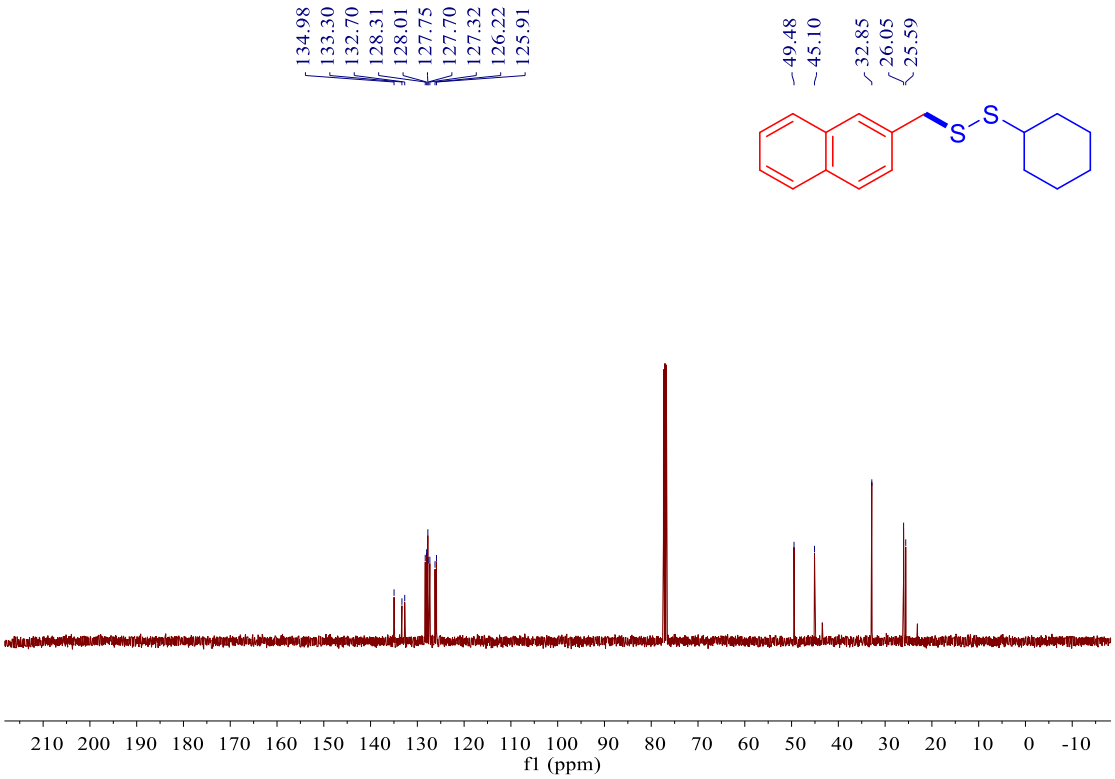
^{13}C NMR Spectra of **6a** (400 MHz, $CDCl_3$)



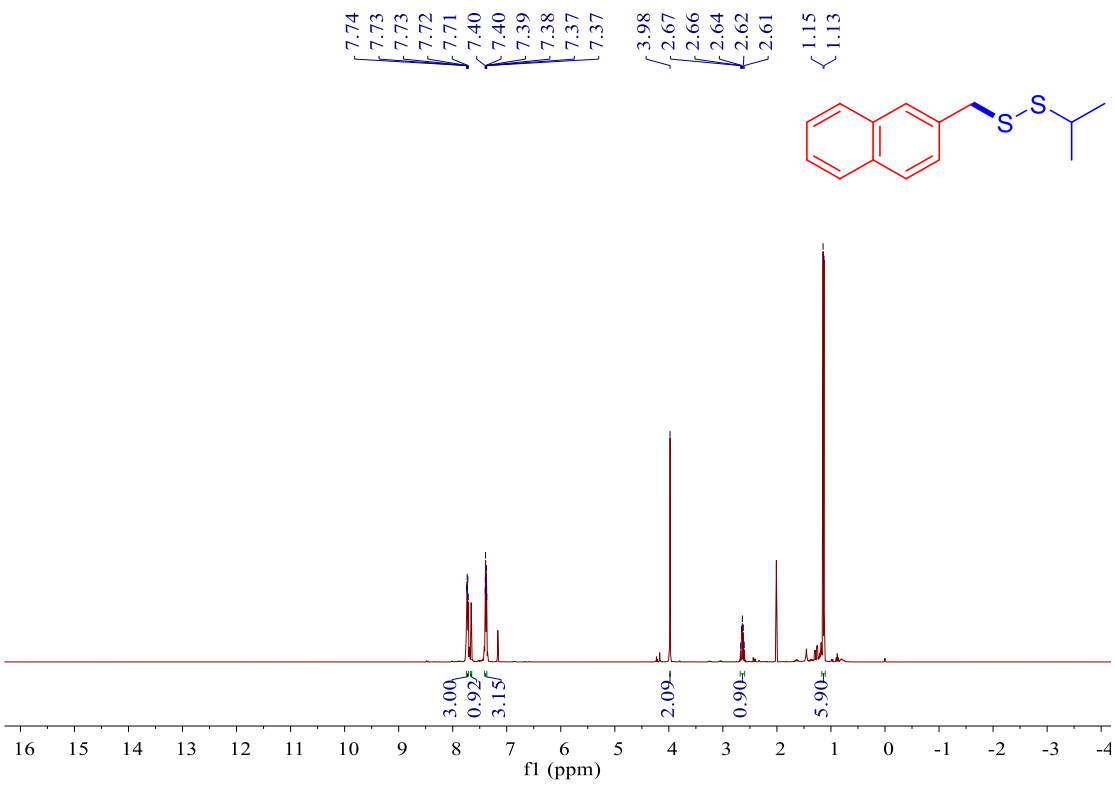
^{1}H NMR Spectra of **6b** (400 MHz, $CDCl_3$)



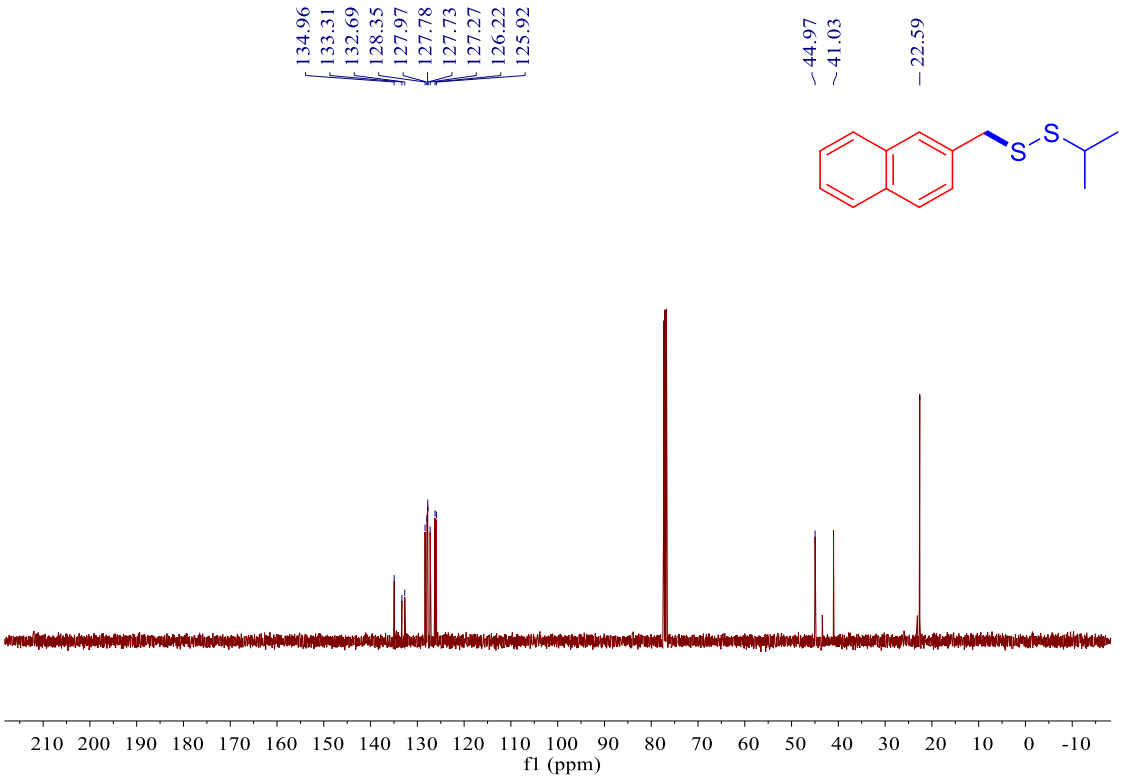
^{13}C NMR Spectra of **6b** (400 MHz, $CDCl_3$)



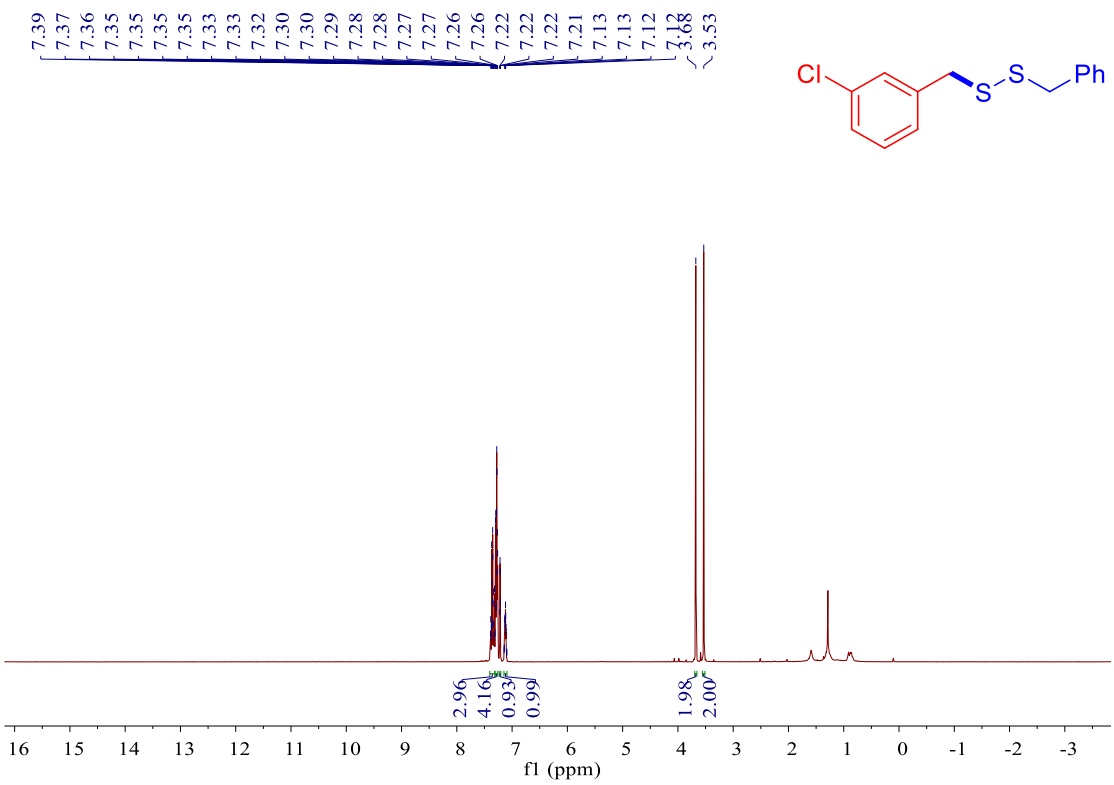
^{1}H NMR Spectra of **6c** (400 MHz, $CDCl_3$)



^{13}C NMR Spectra of **6c** (400 MHz, $CDCl_3$)



^{1}H NMR Spectra of **6d** (400 MHz, $CDCl_3$)



^{13}C NMR Spectra of **6d** (400 MHz, $CDCl_3$)