Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Facile and Efficient transformation of thiols to disulfides *via* a radical pathway with *N*-anomeric amide

Xiaobo Xu,* Leyu Yan, Weijie Huang, Yanping Wang, Mengya Wang, Liming Feng, Panpan Wang, and Shengqiang Wang*

College of Chemistry and Pharmaceutical Engineering, Huanghuai University, Zhumadian 463000, China

Table of Contents

1. General Information

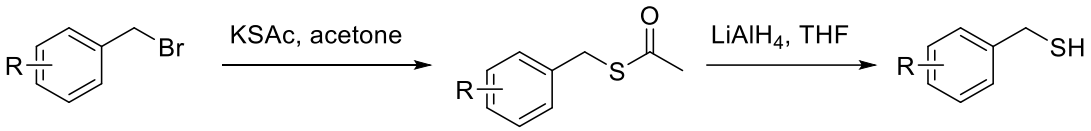
Unless otherwise noted, reactions were carried out in oven-dried glassware or sealed tube under ambient atmosphere. *N*, *N*-Dimethylformamide (DMF) was distilled from calcium hydride. Tetrahydrofuran (THF) was dried and distilled from sodium. Reactions were monitored by analytical thin-layer chromatography (TLC) on Merck silica gel 60 F_{254} plates (0.25 mm), visualized by ultraviolet light (254 nm) or by staining with ceric ammonium molybdate. ^{1}H NMR spectra were obtained on a Bruker AVANCE 400 MHz spectrometer at ambient temperature. Data were reported as follows: chemical shift on the δ scale using residual proton solvent as internal standard [δ 7.26 ($CHCl_3$); δ 2.50 (DMSO); δ 4.79 (H_2O); TMS: 0.00 ppm], multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets), integration, and coupling constant (J) in hertz (Hz). ^{13}C NMR spectra were obtained with proton decoupling on a Bruker AVANCE (100 MHz) spectrometer and were reported in ppm with residual solvent for internal standard [δ 77.0 ($CHCl_3$); δ 39.52 (DMSO)]. TEMPO = 2,2,6,6-Tetramethylpiperidinooxy.

2. Experimental procedures

2.1 Synthesis of *N*-anomeric amide (2)

N-anomeric amide (**2**) was prepared according to known method.[1]

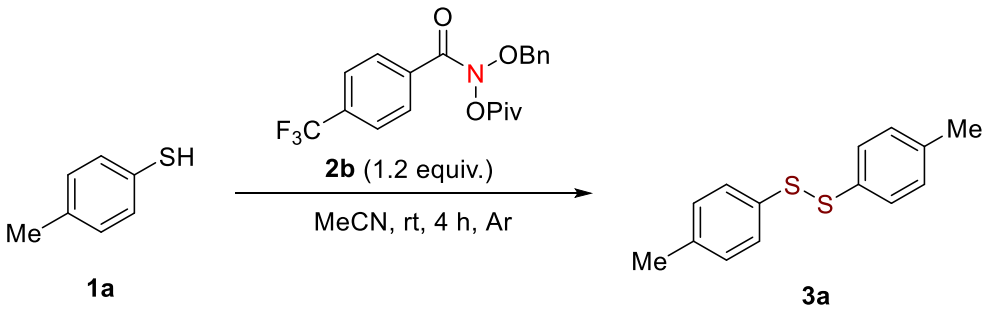
2.2 Synthesis of benzylic thiols



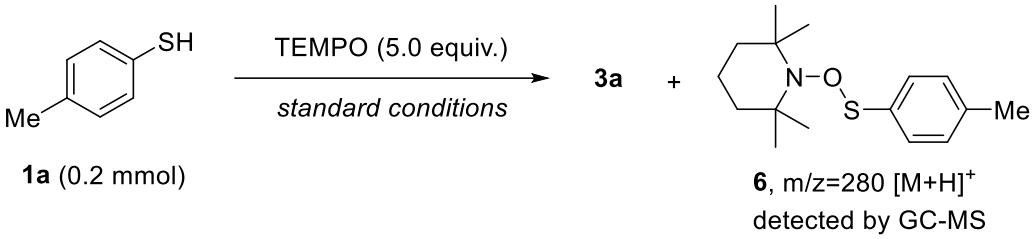
General procedure for commercially unavailable benzylic thiols[2]: To a suspension of potassium thioacetate (7.5 mmol, 1.5 equiv.) in acetone (10 mL) was added bromide substrate (5 mmol, 1.0 equiv.) under nitrogen atmosphere. The reaction mixture was kept stirring overnight. The solvent was evaporated and the solid residue was dissolved in CH_2Cl_2 (20 mL). The organic solution was extracted with water (5 × 20 mL), dried with Na_2SO_4, and concentrated to get a residue. The residue was purified by flash column chromatography.

To a cooled (0 ℃) solution of S-alkyl thioacetate in THF (10 mL) was added $LiAlH_4$ (1.1 equiv.) portionwise, and the reaction mixture was kept stirring for 30 min at 0 ℃. The reaction mixture was carefully quenched with saturated aqueous $NaHSO_4$ solution at 0 ℃, and then extracted with Et_2O (3 × 30 mL). The organic extracts were dried over $MgSO_4$, filtered and evaporated under reduced pressure to afford the crude product. The crude product was purified by flash column chromatography to give the thiols.

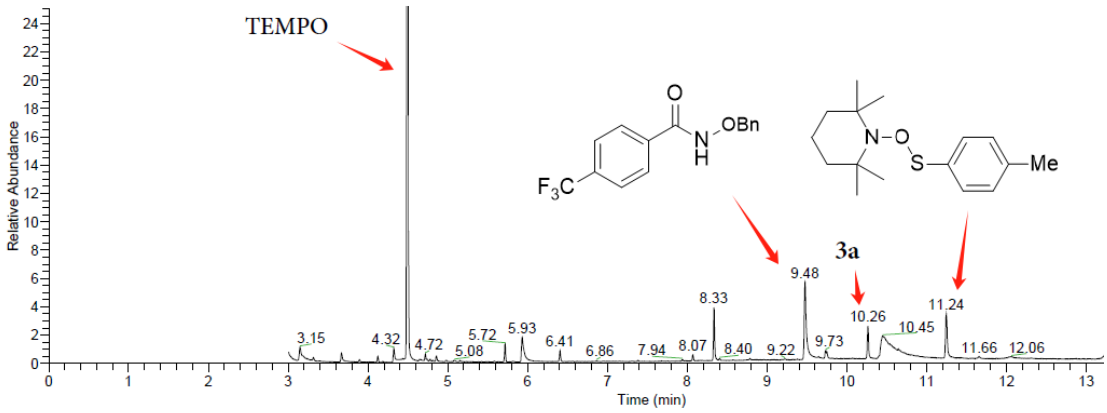
2.3 Synthesis of disulfides



General procedure for disulfides: To a solution of thiol **1a** (0.2 mmol) and *N*-anomeric amide **2b** (0.24 mmol) in dry MeCN (1.0 mL) under argon atmosphere at room temperature. After 4 h the reaction was completed, the reaction was quenched with H_2O (5 mL), and the resulting mixture was extracted with ethyl acetate (3 × 20 mL). The organic phases were combined, washed with saturated brine, dried over anhydrous Na_2SO_4 and then concentrated under vacuum. The resultant residue was purified by flash chromatography (petroleum ether/ethyl acetate = 50/1−10/1) on silica gel to afford the desired product **3a**.



Radical trapping experiments: To a solution of *p*-toluenethiol **1a** (0.2 mmol), *N*-anomeric amide **2b** (0.24 mmol) and radical scavenger TEMPO (5.0 equiv.) in MeCN (2.0 mL) at room temperature. The resulting reaction mixture was stirred for 4 h and H_2O (10 mL) was then added to the mixture. The resulting mixture was extracted with ethyl acetate (3 × 20 mL), and the combined organic phase was dried over Na_2SO_4, and concentrated under reduced pressure. The resultant residue was purified by flash chromatography on silica gel to afford the desired product **3a** in 16% yield.



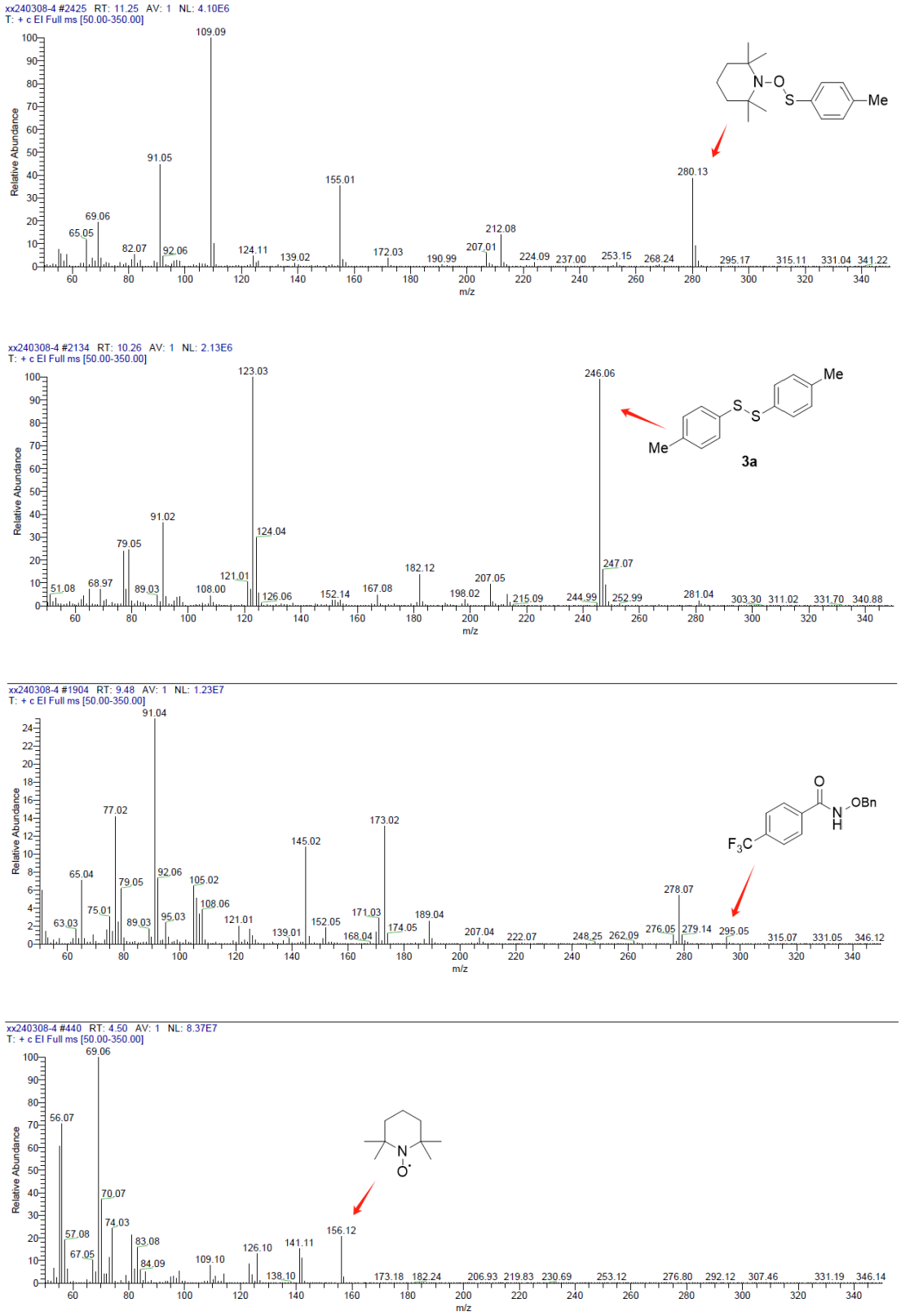
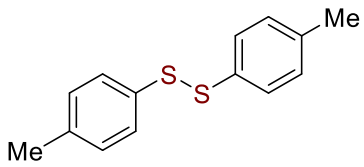


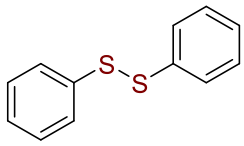
Figure 1. The GC-MS chart of **3a** and **6**.

3. Characterization of Products



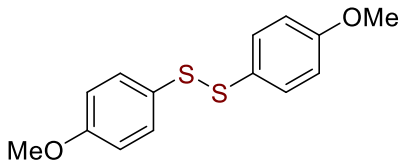
1,2-di-p-tolyldisulfane (3a)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (46 mg, yield 94%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38 (d, J = 7.6 Hz, 4H), 7.09 (d, J = 7.6 Hz, 4H), 2.31 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 137.41, 133.86, 129.77, 128.50, 21.04. MS (ESI+): Calculated $C_{14}H_{14}S_2$ as 246.05, [M^+] found 246.04 m/z. Characterized in accordance to the literature.[3]



1,2-diphenyldisulfane (3b)

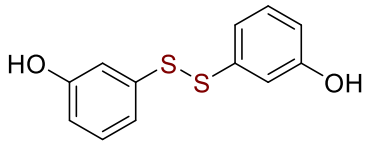
Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (38 mg, yield 88%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.49 (d, J = 7.6 Hz, 4H), 7.28 (t, J = 7.2 Hz, 4H), 7.20 (t, J = 7.2 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 136.99, 129.03, 127.47, 127.12. MS (ESI+): Calculated $C_{12}H_{10}S_2$ as 218.02, [M^+] found 218.10 m/z. Characterized in accordance to the literature.[3]



1,2-bis(4-methoxyphenyl)disulfane (3c)

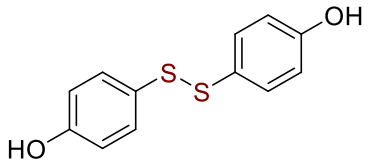
Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow oil (51 mg, yield 92%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39 (d, J = 7.6 Hz, 4H), 6.83 (d, J = 7.6 Hz, 4H), 3.79 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm)

159.85, 132.60, 128.35, 114.56, 55.30. MS (ESI+): Calculated $C_{14}H_{14}O_2S_2$ as 278.04, [M^+] found 278.00 m/z. Characterized in accordance to the literature.[3]



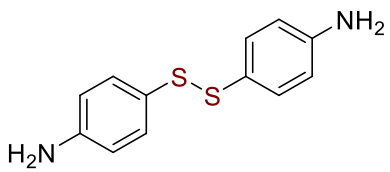
3,3'-disulfanediyldiphenol (3d)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow solid (34 mg, yield 68%), ^{1}H NMR (400 MHz, DMSO): δ (ppm) 9.77 (s, 2H), 7.18 (t, J = 8.0 Hz, 2H), 6.92 (d, J = 12.0 Hz, 4H), 6.68 (d, J = 8 Hz, 2H); ^{13}C NMR (101 MHz, DMSO): δ (ppm) 158.16, 136.76, 130.30, 117.25, 114.62, 113.04. MS (ESI+): Calculated $C_{12}H_{10}O_2S_2$ as 250.01, [M^+] found 249.96 m/z. Characterized in accordance to the literature.[4]



4,4'-disulfanediyldiphenol (3e)

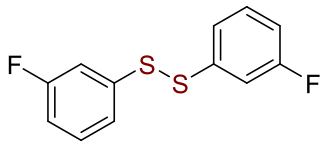
Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (38 mg, yield 77%), ^{1}H NMR (400 MHz, DMSO): δ (ppm) 9.84 (s, 2H), 7.26 (d, J = 8.0 Hz, 4H), 6.75 (d, J = 8.0 Hz, 4H); ^{13}C NMR (101 MHz, DMSO): δ (ppm) 158.27, 133.03, 125.09, 116.30. MS (ESI+): Calculated $C_{12}H_{10}O_2S_2$ as 250.01, [M^+] found 249.97 m/z. Characterized in accordance to the literature.[4]



4,4'-disulfanediyldianiline (3f)

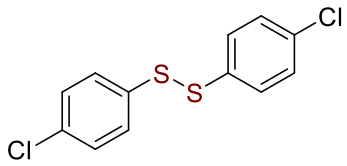
Followed the general procedure of disulfide synthesis (P3), product obtained as a white

solid (36 mg, yield 72%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.24 (d, J = 7.2 Hz, 4H), 6.56 (d, J = 7.2 Hz, 4H), 3.76 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 147.04, 133.89, 125.61, 115.31. MS (ESI+): Calculated $C_{12}H_{12}N_2S_2$ as 248.04, [M^+] found 248.00 m/z. Characterized in accordance to the literature.[5]



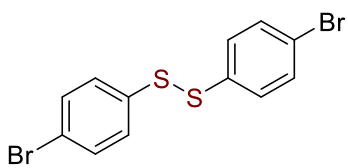
1,2-bis(3-fluorophenyl)disulfane (3g)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow oil (43 mg, yield 84%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.28-7.21 (m, 6H) ,6.94-6.90 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 164.26 (d, J = 248 Hz, 1C), 138.76 (d, J = 7 Hz, 1C), 130.50 (d, J = 8 Hz, 1C), 122.61 (d, J = 3 Hz, 1C), 114.43, (d, J = 22 Hz, 1C), 114.07 (d, J = 24 Hz, 1C). MS (ESI+): Calculated $C_{12}H_8F_2S_2$ as 254.00, [M^+] found 253.95 m/z. Characterized in accordance to the literature.[6]



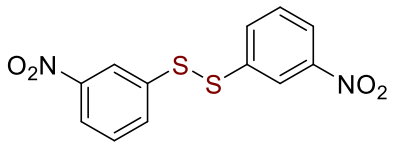
1,2-bis(4-chlorophenyl)disulfane (3h)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow solid (53 mg, yield 93%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39 (d, J = 7.6 Hz, 4H), 7.27 (d, J = 7.6 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 135.10, 133.61, 129.56, 129.28. MS (ESI+): Calculated $C_{12}H_8Cl_2S_2$ as 285.94, [M^+] found 285.92 m/z. Characterized in accordance to the literature.[3]



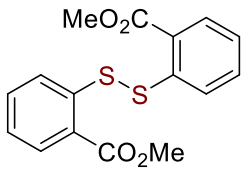
1,2-bis(4-bromophenyl)disulfane (3i)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (65 mg, yield 87%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.42 (d, J = 7.6 Hz, 4H), 7.33 (d, J = 7.6 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 135.69, 132.19, 129.36, 121.51. MS (ESI+): Calculated $C_{12}H_8Br_2S_2$ as 373.84, [M^+] found 373.86 m/z. Characterized in accordance to the literature.[3]



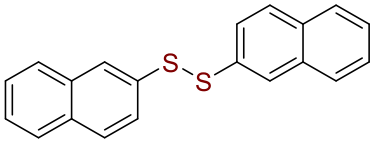
1,2-bis(3-nitrophenyl)disulfane (3j)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow solid (49 mg, yield 79%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.37 (s, 2H), 8.10 (d, J = 8.0 Hz, 2H), 7.81 (d, J = 7.6 Hz, 2H), 7.53 (t, J = 8.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 148.72, 138.42, 132.73, 130.18, 122.43, 121.82. MS (ESI+): Calculated $C_{12}H_8N_2O_4S_2$ as 307.99, [M^+] found 307.92 m/z. Characterized in accordance to the literature.[6]



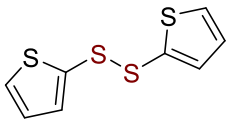
dimethyl 2,2'-disulfanediyldibenzoate (3k)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (43 mg, yield 65%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.06 (d, J = 7.6 Hz, 2H), 7.75 (d, J = 8.4 Hz, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.24 (t, J = 7.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 166.89, 140.33, 133.06, 131.44, 127.25, 125.80, 125.46, 52.37. MS (ESI+): Calculated $C_{16}H_{14}O_4S_2$ as 334.03, [M^+] found 333.99 m/z. Characterized in accordance to the literature.[5]



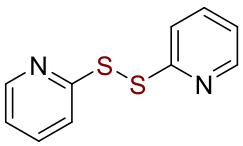
1,2-di(naphthalen-2-yl)disulfane (3l)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (45 mg, yield 71%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.97 (s, 2H), 7.77 (d, J = 7.6 Hz, 4H), 7.71 (d, J = 6.8 Hz, 2H), 7.62-7.60 (m, 2H), 7.44 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 134.24, 133.45, 132.47, 128.95, 127.74, 127.44, 126.71, 126.52, 126.21, 125.63. MS (ESI+): Calculated $C_{20}H_{14}S_2$ as 318.05, [M^+] found 318.03 m/z. Characterized in accordance to the literature.[7]



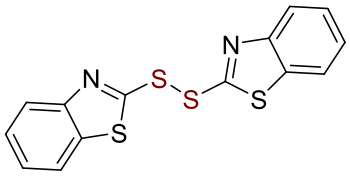
1,2-di(thiophen-2-yl)disulfane (3m)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow solid (39 mg, yield 85%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.49 (d, J = 5.2 Hz, 2H), 7.15 (s, 2H), 7.01 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 135.66, 135.62, 132.25, 127.73. MS (ESI+): Calculated $C_8H_6S_4$ as 229.94, [M^+] found 229.86 m/z. Characterized in accordance to the literature.[8]



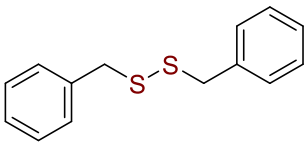
1,2-di(pyridin-2-yl)disulfane (3n)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (28 mg, yield 64%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.47 (s, 2H), 7.61 (s, 4H), 7.10 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 158.83, 149.48, 137.34, 121.04, 119.58. MS (ESI+): Calculated $C_{10}H_8N_2S_2$ as 220.01, [M^+] found 219.99 m/z. Characterized in accordance to the literature.[8]



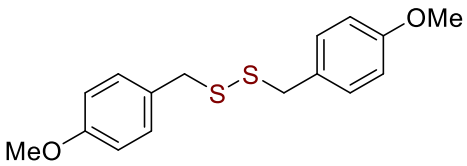
1,2-bis(benzo[d]thiazol-2-yl)disulfane (3o)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (33 mg, yield 50%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.94 (d, J = 8.4 Hz, 2H), 7.77 (d, J = 8.0 Hz, 2H), 7.47 (t, J = 8.0 Hz, 2H), 7.36 (t, J = 7.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 167.86, 154.51, 136.10, 126.58, 125.29, 122.66, 121.30. MS (ESI+): Calculated $C_{14}H_8N_2S_4$ as 331.96, [M^+] found 332.09 m/z. Characterized in accordance to the literature.[8]



1,2-dibenzyldisulfane (5a)

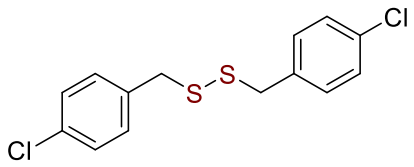
Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (40 mg, yield 82%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.33-7.22 (m, 5H), 3.59 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 137.37, 129.39, 128.46, 127.40, 43.26. MS (ESI+): Calculated $C_{14}H_{14}S_2$ as 246.05, [M^+] found 246.01 m/z. Characterized in accordance to the literature.[9]



1,2-bis(4-methoxybenzyl)disulfane (5b)

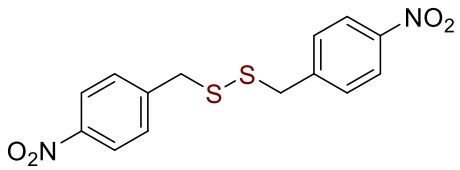
Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (45 mg, yield 74%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.20 (d, J = 8.0 Hz, 4H), 6.89 (d, J = 8.0 Hz, 4H), 3.81 (s, 6H), 3.62 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 159.01, 130.50, 129.38, 113.93, 113.87, 55.27, 42.75. MS (ESI+): Calculated

$C_{16}H_{18}O_2S_2$ as 306.07, [M^+] found 305.93 m/z. Characterized in accordance to the literature.[9]



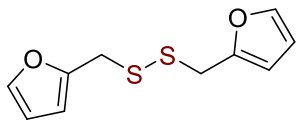
1,2-bis(4-chlorobenzyl)disulfane (5c)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (51 mg, yield 81%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.29 (d, J = 7.6 Hz, 4H), 7.15 (d, J = 7.6 Hz, 4H), 3.57 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 135.79, 133.37, 130.63, 128.65, 42.45. MS (ESI+): Calculated $C_{14}H_{12}Cl_2S_2$ as 313.98, [M^+] found 313.88 m/z. Characterized in accordance to the literature.[9]



1,2-bis(4-nitrobenzyl)disulfane (5d)

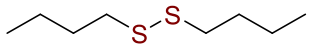
Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (52 mg, yield 78%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.20 (d, J = 7.6 Hz, 4H), 7.39 (d, J = 7.6 Hz, 4H), 3.70 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 147.34, 144.68, 130.06, 123.81, 42.25. MS (ESI+): Calculated $C_{14}H_{12}N_2O_4S_2$ as 336.02, [M^+] found 336.12 m/z. Characterized in accordance to the literature.[9]



1,2-bis(furan-2-ylmethyl)disulfane(5e)

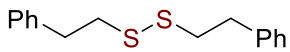
Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow oil (34 mg, yield 76%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.39 (dd, J = 0.9,

1.9 Hz, 2H), 6.33 (dd, J = 1.9, 3.3 Hz, 2H), 6.23 (d, J = 3.3 Hz, 2H), 3.68 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 150.12, 142.42, 110.72, 108.93, 35.57. MS (ESI+): Calculated $C_{10}H_{10}O_2S_2$ as 226.01, [M^+] found 226.14 m/z. Characterized in accordance to the literature.[4]



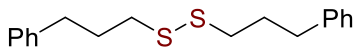
1,2-dibutyldisulfane (5f)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (29 mg, yield 82%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 2.68 (t, J = 7.6 Hz, 4H), 1.68 (p, J = 7.3 Hz, 4H), 1.38-1.27 (m, 4H), 0.88 (t, J = 6.0 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 38.83, 31.27, 21.62, 13.66. MS (ESI+): Calculated $C_8H_{18}S_2$ as 178.08, [M^+] found 178.07 m/z. Characterized in accordance to the literature.[8]



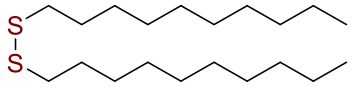
1,2-diphenethyldisulfane(5g)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (40 mg, yield 73%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.32-7.28 (m, 4H), 7.23-7.18 (m, 6H), 3.00-2.95 (m, 4H), 2.94-2.91 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 140.00, 128.59, 128.49, 126.39, 40.17, 35.70. MS (ESI+): Calculated $C_{16}H_{18}S_2$ as 274.08, [M^+] found 274.09 m/z. Characterized in accordance to the literature.[10]



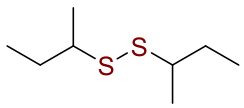
1,2-bis(3-phenylpropyl)disulfane(5h)

Followed the general procedure of disulfide synthesis (P3), product obtained as a white solid (39 mg, yield 65%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.22-7.19 (m, 4H), 7.18-7.10 (m, 6H), 2.66-2.60 (m, 8H), 1.98-1.90 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 141.43, 128.59, 128.52, 126.07, 38.21, 34.46, 30.67. MS (ESI+): Calculated $C_{18}H_{22}S_2$ as 302.12, [M^+] found 302.01 m/z. Characterized in accordance to the literature.[10]



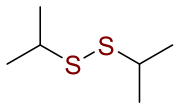
1,2-didecyldisulfane (5i)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (52 mg, yield 76%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 2.68 (t, J = 7.6 Hz, 4H), 1.68 (p, J = 7.3 Hz, 4H), 1.38-1.27 (m, 28H), 0.88 (t, J = 6.0 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 39.18, 31.89, 29.55, 29.51, 29.31, 29.24, 29.21, 28.52, 22.67, 14.10. MS (ESI+): Calculated $C_{20}H_{42}S_2$ as 346.27, [M^+] found 346.15 m/z. Characterized in accordance to the literature.[4]



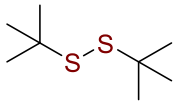
1,2-di-sec-butyldisulfane (5j)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (22 mg, yield 62%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 2.74 (q, J = 6.4 Hz, 2H), 1.77-1.66 (m, 2H), 1.57-1.46 (m, 2H), 1.29 (d, J = 6.8 Hz, 6H), 0.98 (t, J = 7.2 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 48.27, 48.24, 29.00, 20.07, 11.48. MS (ESI+): Calculated $C_8H_{18}S_2$ as 178.08, [M^+] found 178.05 m/z. Characterized in accordance to the literature.[11]



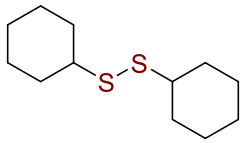
1,2-diisopropyldisulfane (5k)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (21 mg, yield 70%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 3.00-2.94 (m, 2H), 1.30 (d, J = 6.8 Hz, 12H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 41.39, 22.57. MS (ESI+): Calculated $C_6H_{14}S_2$ as 150.05, [M^+] found 150.04 m/z. Characterized in accordance to the literature.[12]



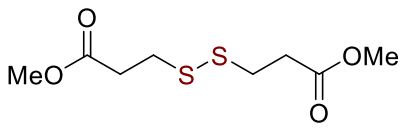
1,2-di-tert-butyldisulfane (5l)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (21 mg, yield 58%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 1.35 (s, 18H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 48.94, 46.08, 34.54. MS (ESI+): Calculated $C_8H_{18}S_2$ as 178.08, [M^+] found 178.09 m/z. Characterized in accordance to the literature.[8]



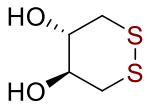
1,2-dicyclohexyldisulfane(5m)

Followed the general procedure of disulfide synthesis (P3), product obtained as a colorless oil (40 mg, yield 88%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 2.71-2.66 (m, 1H), 2.06-2.03 (m, 2H), 1.79-1.77 (m, 2H), 1.63-1.61 (m, 1H), 2.37-1.21 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 49.79, 32.74, 25.98, 25.61. MS (ESI+): Calculated $C_{12}H_{22}S_2$ as 230.12, [M^+] found 230.10 m/z. Characterized in accordance to the literature.[10]



dimethyl 3,3'-disulfanediyldipropionate (5n)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow oil (30 mg, yield 64%), ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 3.71 (s, 6H), 2.93 (t, J = 7.2 Hz, 4H), 2.74 (t, J = 7.2 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 172.10, 51.87, 33.86, 33.05. MS (ESI+): Calculated $C_8H_{14}O_4S_2$ as 238.03, [M^+] found 238.00 m/z. Characterized in accordance to the literature.[8]



1,2-dithiane-4,5-diol (5o)

Followed the general procedure of disulfide synthesis (P3), product obtained as a yellow oil (23 mg, yield 75%), ^{1}H NMR (400 MHz, DMSO): δ (ppm) 5.22 (s, 2H), 3.34 (d, J = 4.0 Hz, 2H), 3.04 (d, J = 13.2 Hz, 2H), 2.76-2.70 (m, 2H); ^{13}C NMR (101 MHz, DMSO): δ (ppm) 73.40, 40.23. MS (ESI+): Calculated $C_4H_8O_2S_2$ as 152.00, [M^+] found 151.95 m/z. Characterized in accordance to the literature.[10]

4. References

[1] S. H. Kennedy, B. D. Dherange, K. J. Berger and M. D. Levin, *Nature*, 2021, **593**, 223-227.

[2] Z. Yang, C.-H. Yang, S. Chen, X. Chen, L. Zhang and H. Ren, *Chem. Commun.*, 2017, **53**, 12092-12095.

[3] S. M. Weierbach, R. P. Reynolds, S. M. Stephens, K. V. Vlasakakis, R. T. Ritter, O. M. White, N. H. Patel, E. C. Hayes, S. Dunmire and K. M. Lambert, *J. Org. Chem.*, 2023, **88**, 11392-11410.

[4] A. Talla, B. Driessen, N. J. W. Straathof, L. G. Milroy, L. Brunsveld, V. Hessel and T. Noël, *Adv. Synth. Catal.*, 2015, **357**, 2180-2186.

[5] Y. Dou, X. Huang, H. Wang, L. Yang, H. Li, B. Yuan, G. Yang, *Green Chem.,* 2017, **19**, 2491-2495.

[6] X. Li, J. Du, Y. Zhang, H. Chang, W. Gao and W. Wei, *Org. Biomol. Chem.*, 2019, **17**, 3048-3055.

[7] D. R. Dreyer, H. P. Jia, A. D. Todd, J. Geng and C. W. Bielawski, *Org Biomol Chem.*, 2011, **9**, 7292-7295.

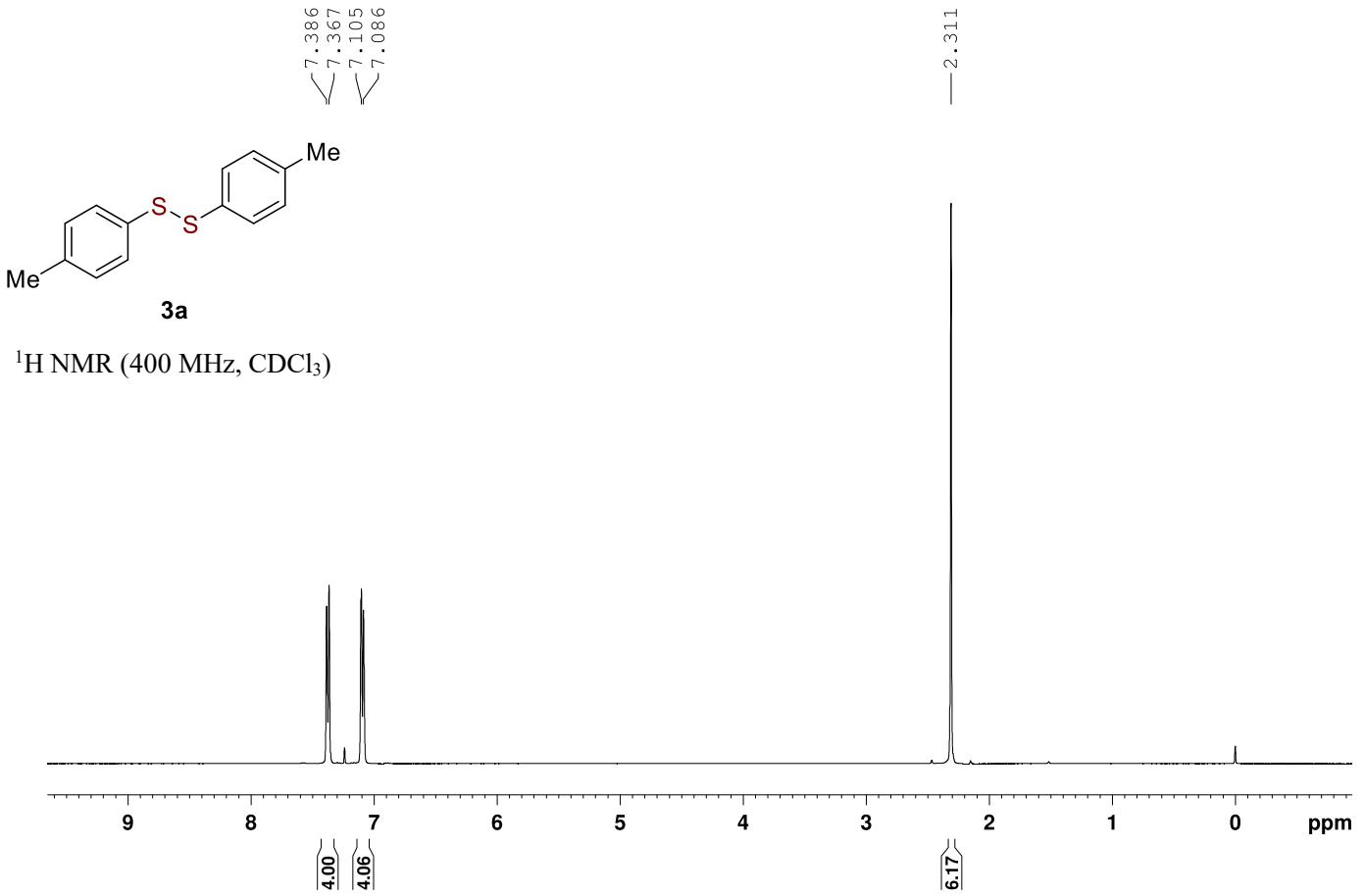
[8] C. Zhu, D. Wu, H. Liu, C. Meng and T. Tang, *Green Chem.*, 2022, **24**, 9033-9039.

[9] V. Panduranga, G. Prabhu, Basavaprabhu, N. R. Panguluri and V. V. Sureshbabu, *Synthesis*, 2016, **48**, 1711-1718.

[10] L. Wang, L. Chen, Z. Qin, K. Ni, X. Li, Z. Yu, Z. Kuang, X. Qin, H. Duan and J. An, *Molecules*, 2023, **28**, 6789.

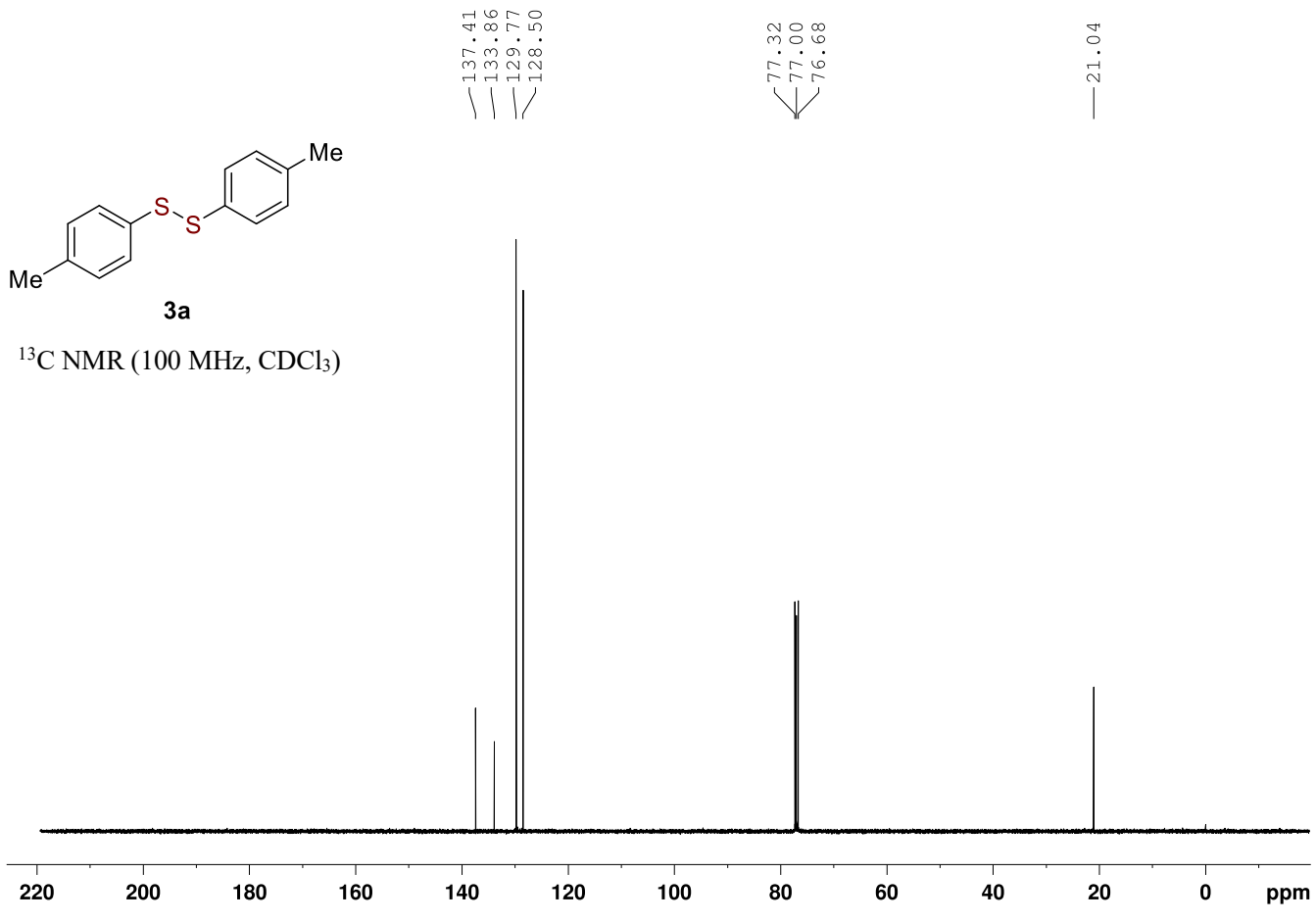
[11] Y. Feng, J. Nie, S. Xie, Z. He, H. Hong, J. Li, Y. Huang, L. Chen and Y. Li, *Chem. Commun.*, 2024, **60**, 1140-1143.

[12] D. Chauhan, P. Kumar, C. Joshi, N. Labhsetwar, S. K. Ganguly and S. L. Jain, *New J. Chem.*, 2015, **39**, 6193-6200.



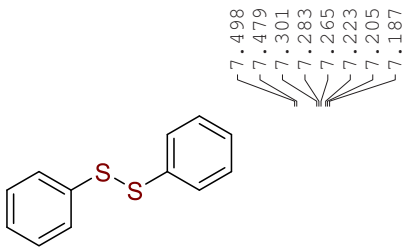
3a

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



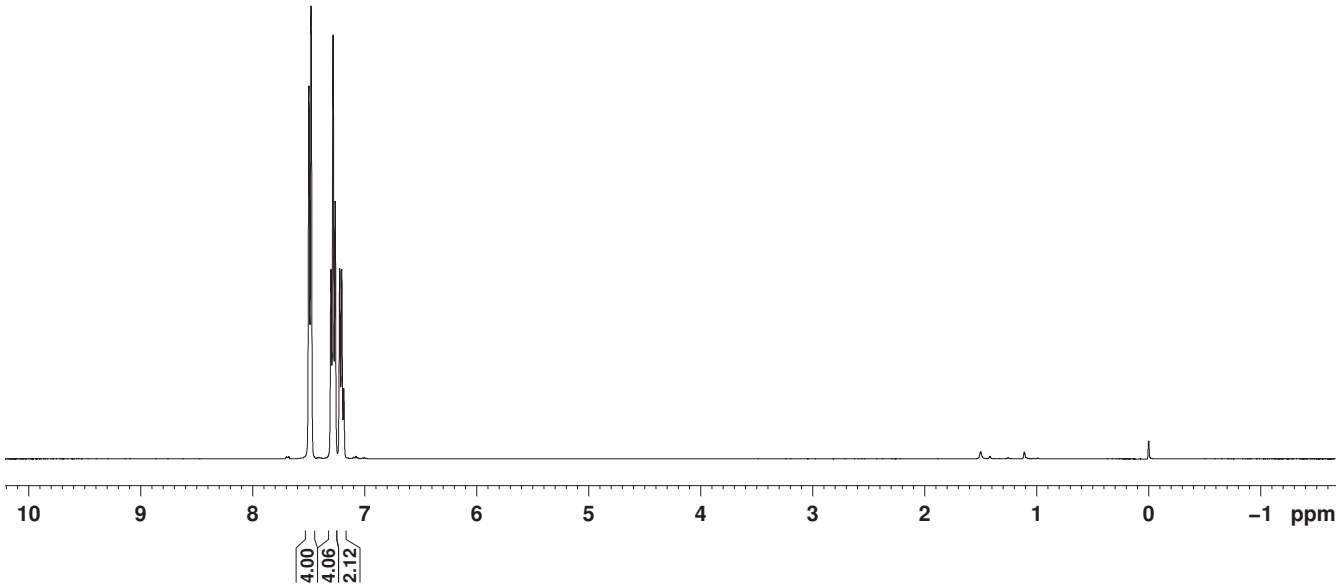
3a

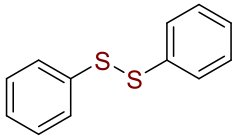
^{13}C NMR (100 MHz, $CDCl_3$)



3b

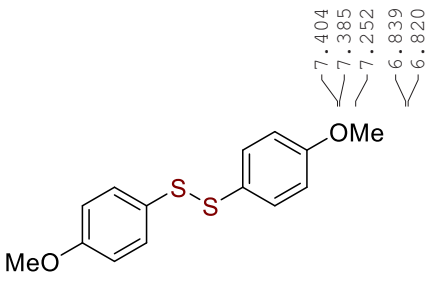
^{1}H NMR (400 MHz, $CDCl_3$)





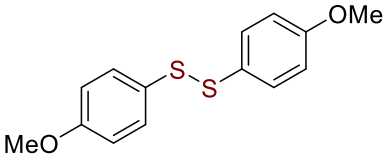
3b

^{13}C NMR (100 MHz, $CDCl_3$)



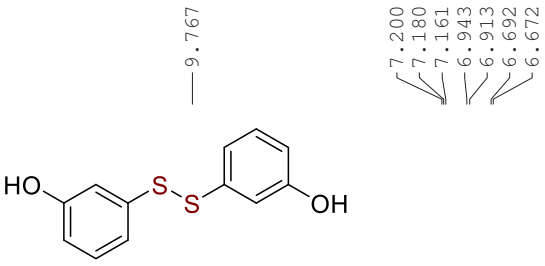
3c

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



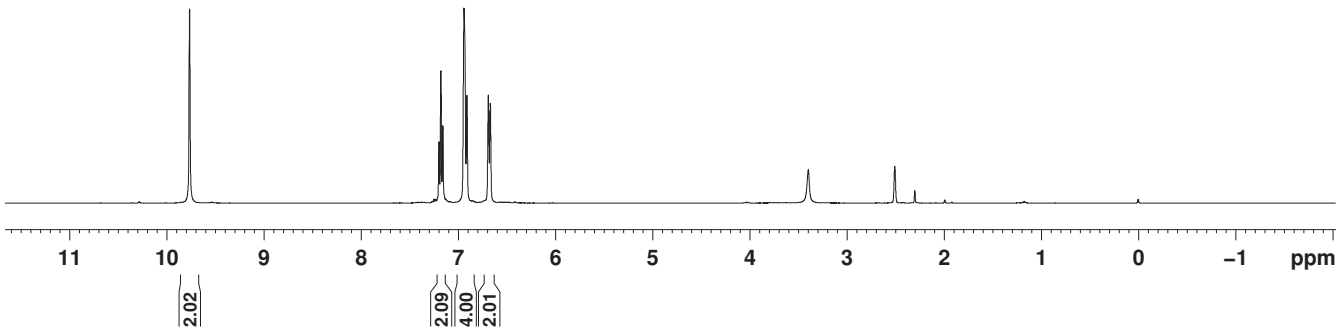
3c

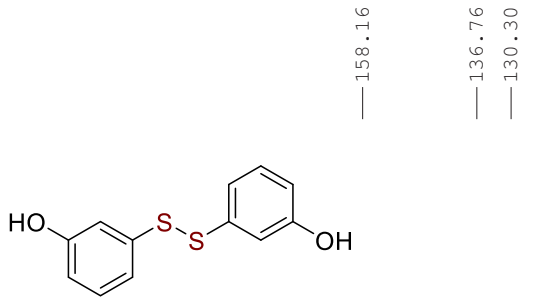
^{13}C NMR (100 MHz, $CDCl_3$)



3d

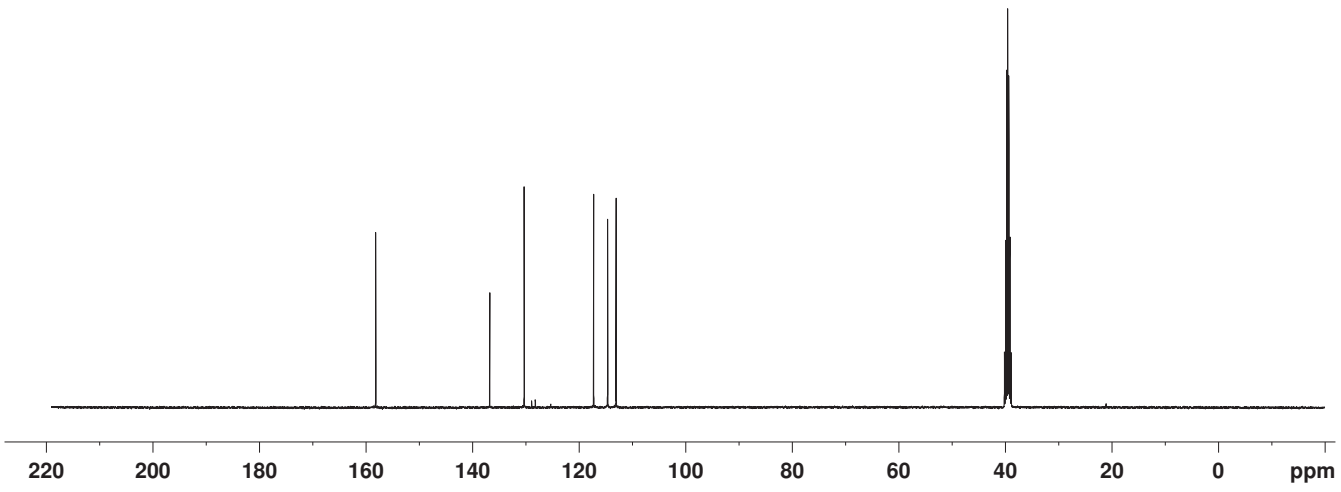
^{1}H NMR (400 MHz, d^{6}-DMSO)

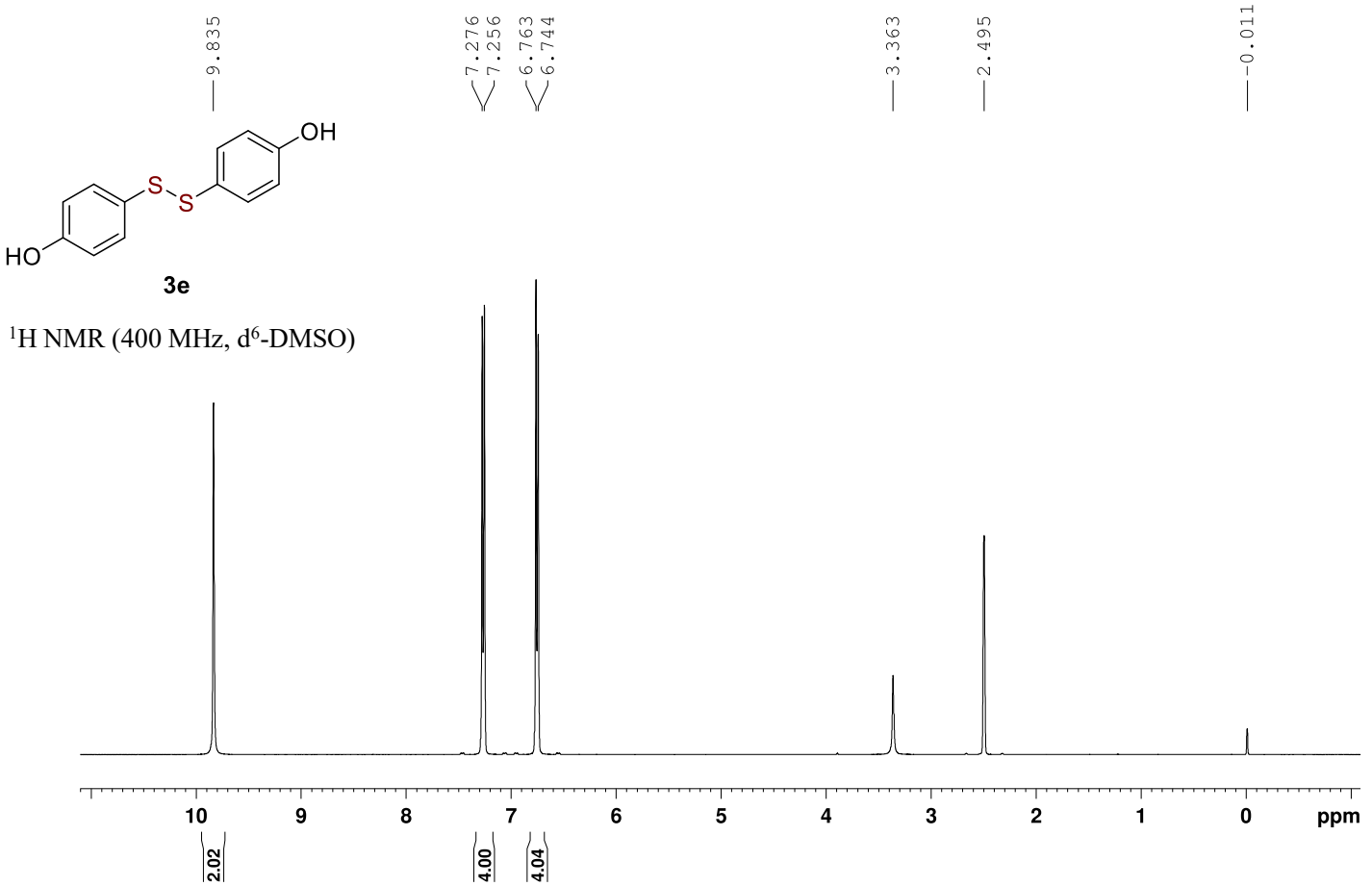




3d

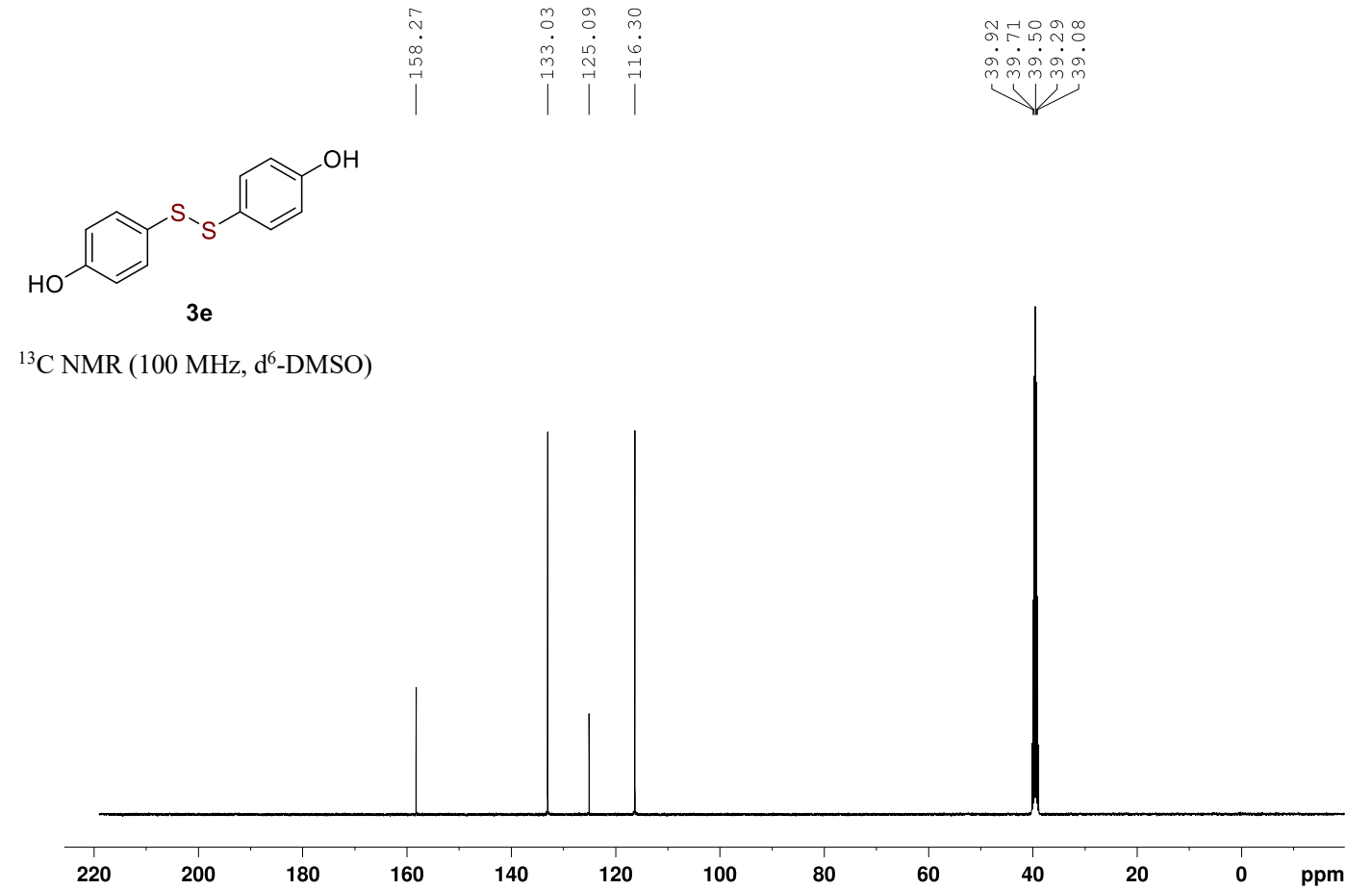
^{13}C NMR (100 MHz, d^{6}-DMSO)





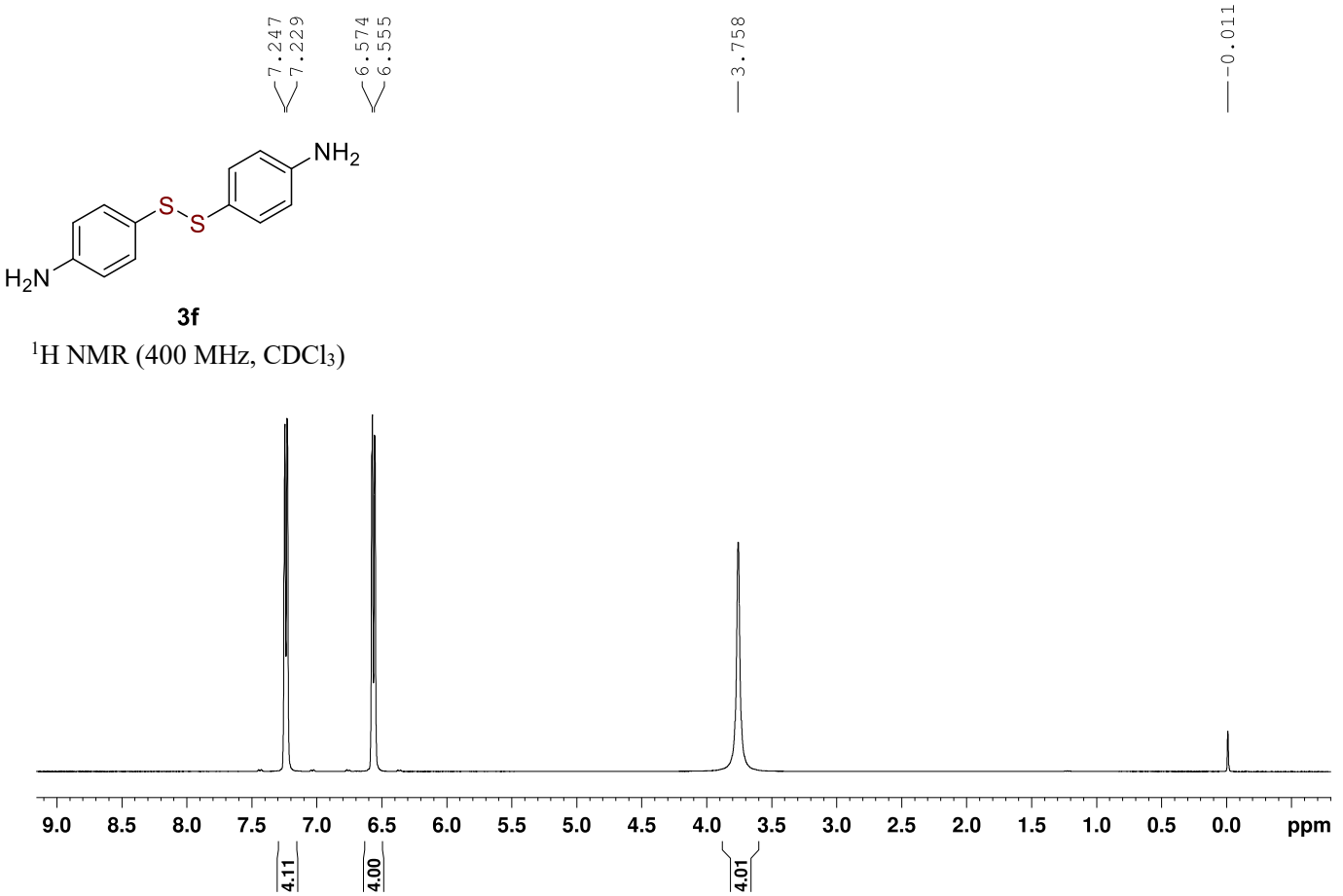
3e

^{1}H NMR (400 MHz, d^{6}-DMSO)



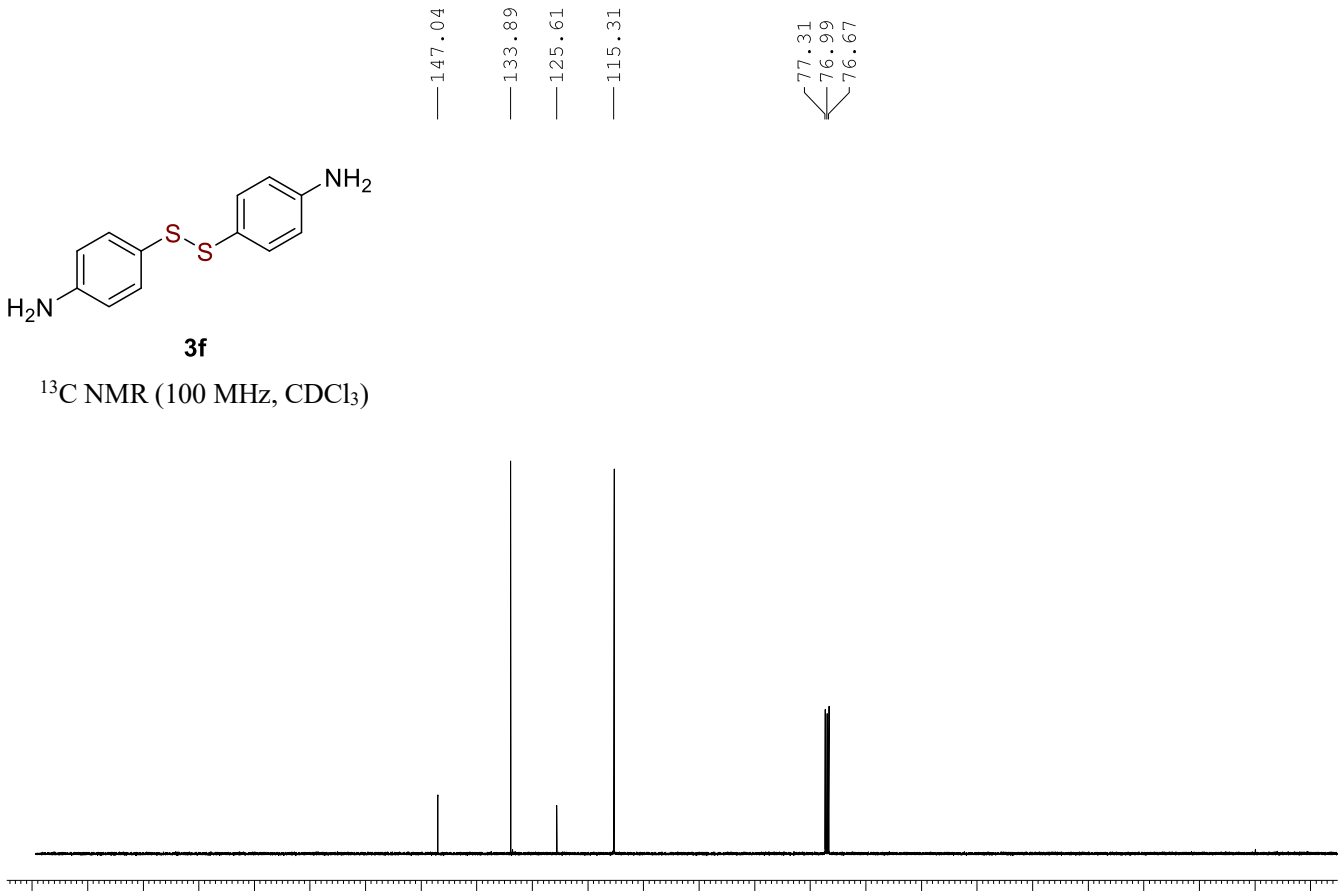
3e

^{13}C NMR (100 MHz, d^{6}-DMSO)



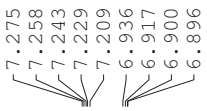
3f

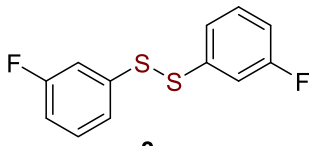
^{1}H NMR (400 MHz, $CDCl_3$)



3f

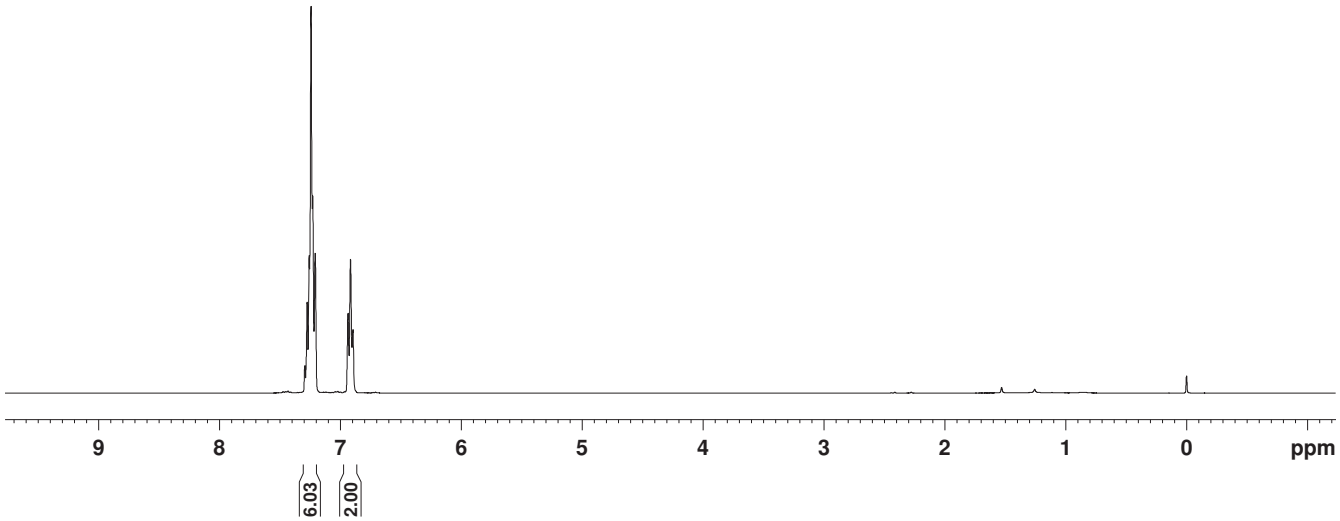
^{13}C NMR (100 MHz, $CDCl_3$)

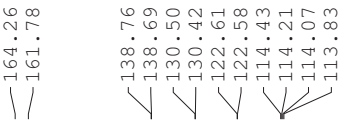


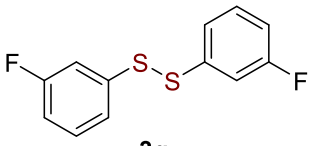


3g

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

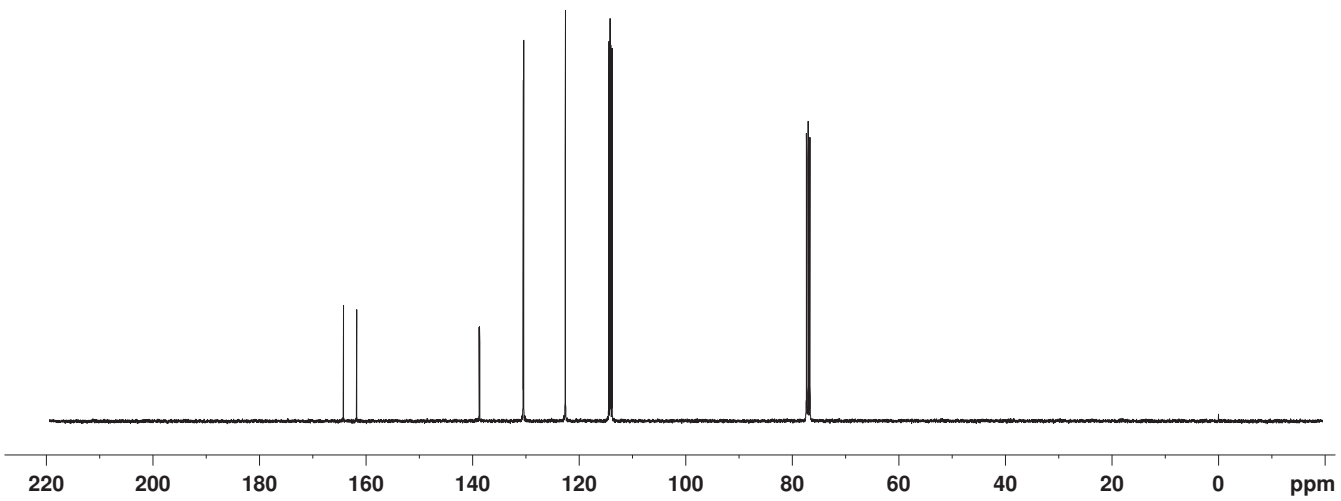


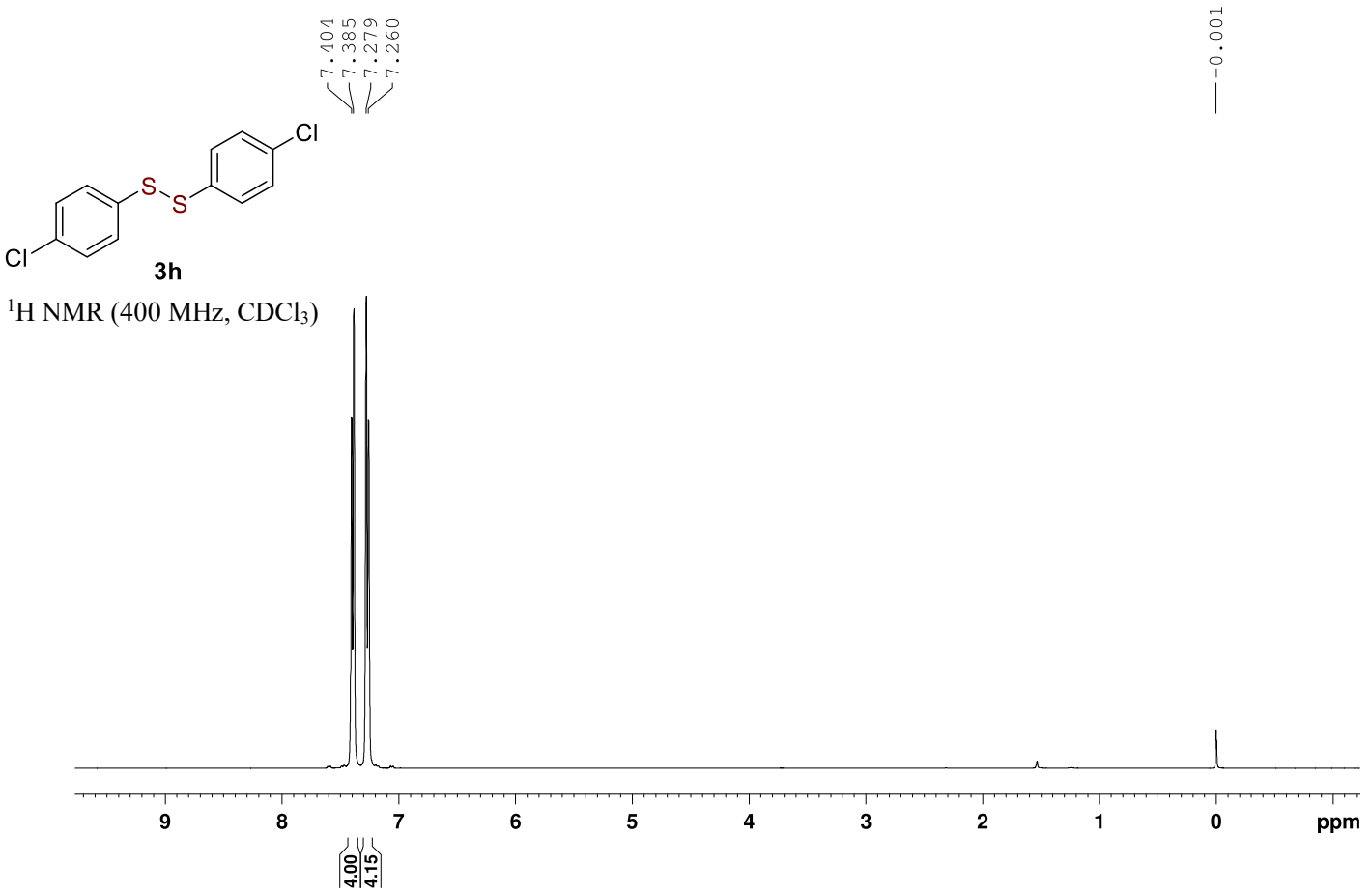




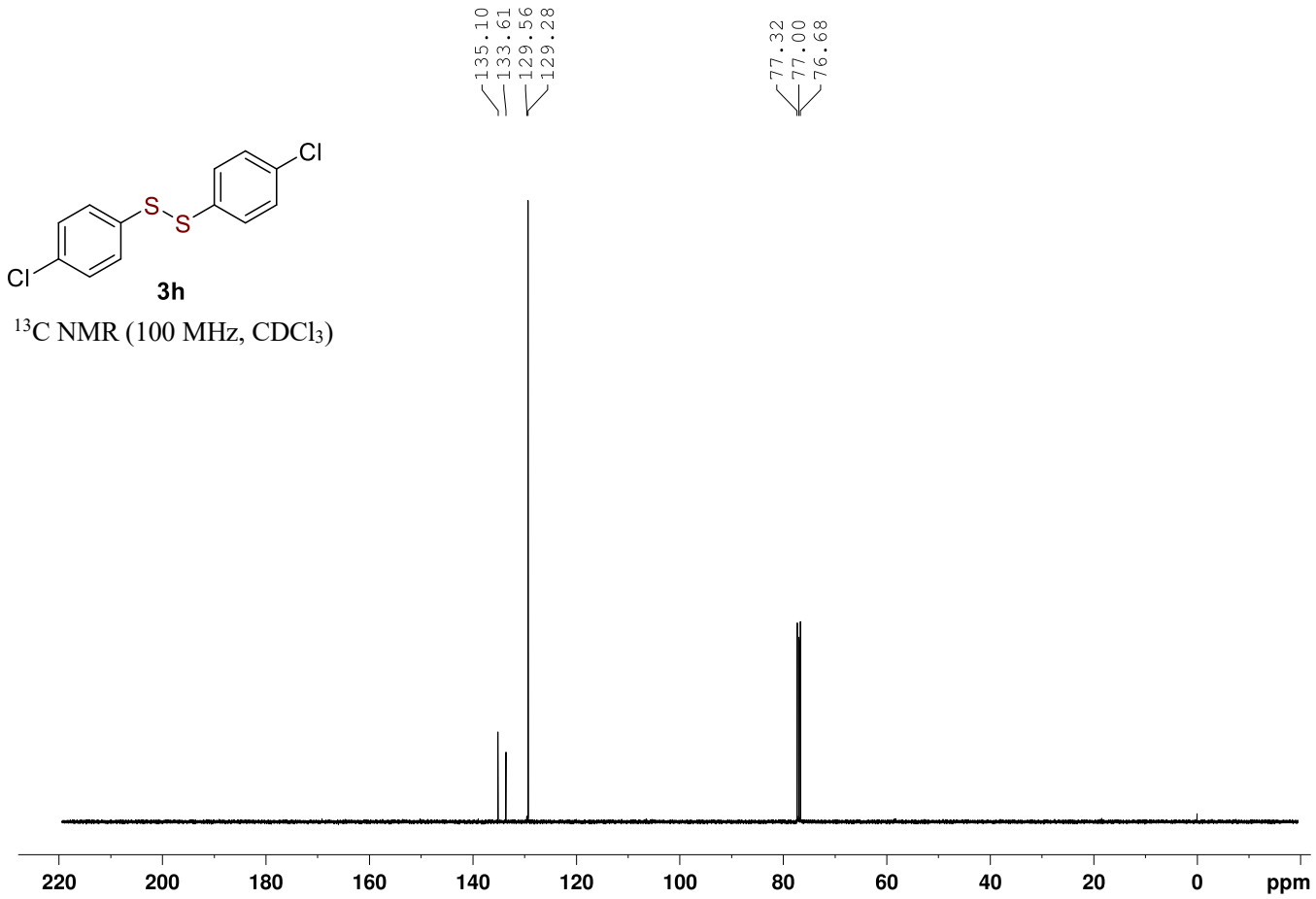
3g

^{13}C NMR (100 MHz, $CDCl_3$)

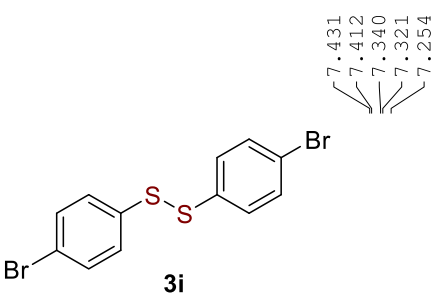




^{1}H NMR (400 MHz, $CDCl_3$)

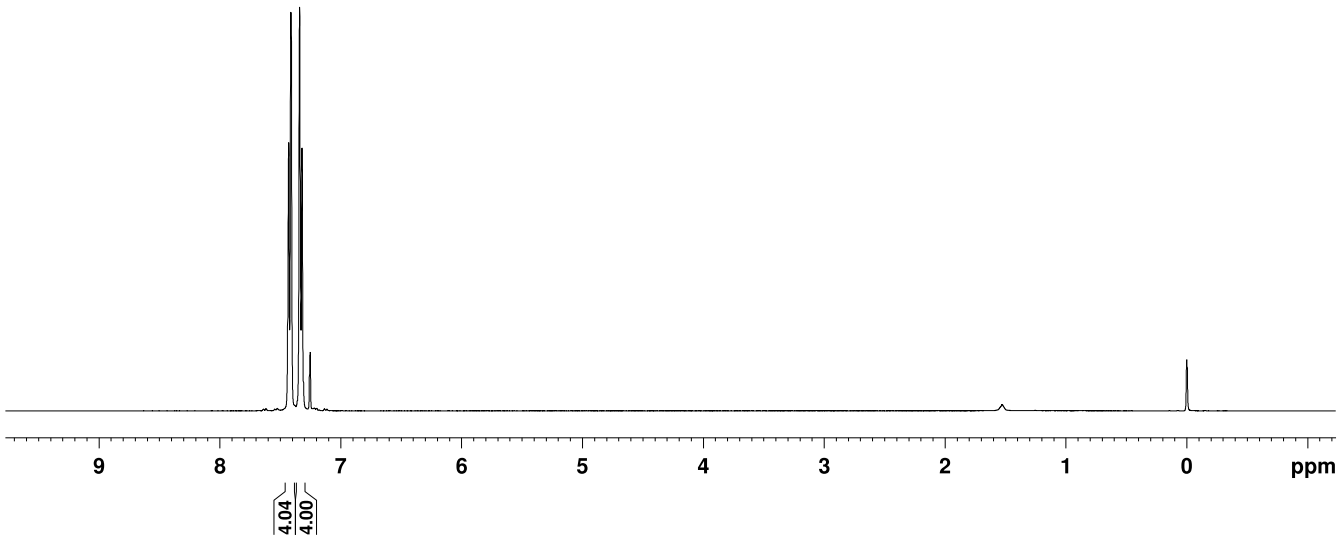


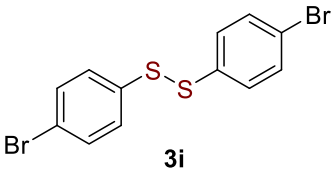
^{13}C NMR (100 MHz, $CDCl_3$)



3i

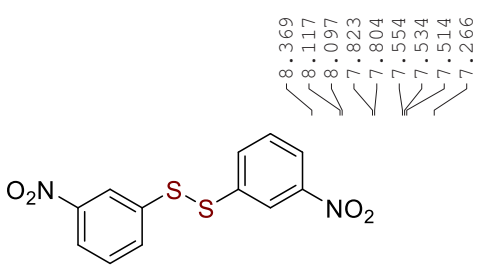
^{1}H NMR (400 MHz, $CDCl_3$)





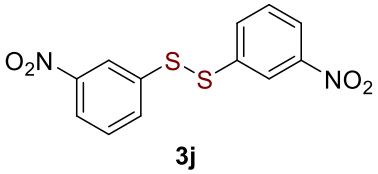
3i

^{13}C NMR (100 MHz, $CDCl_3$)



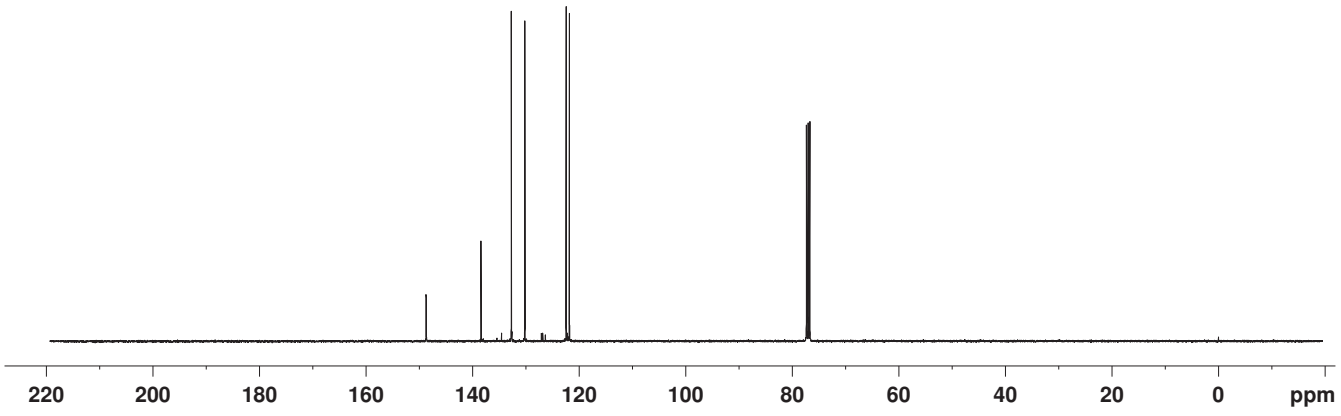
3j

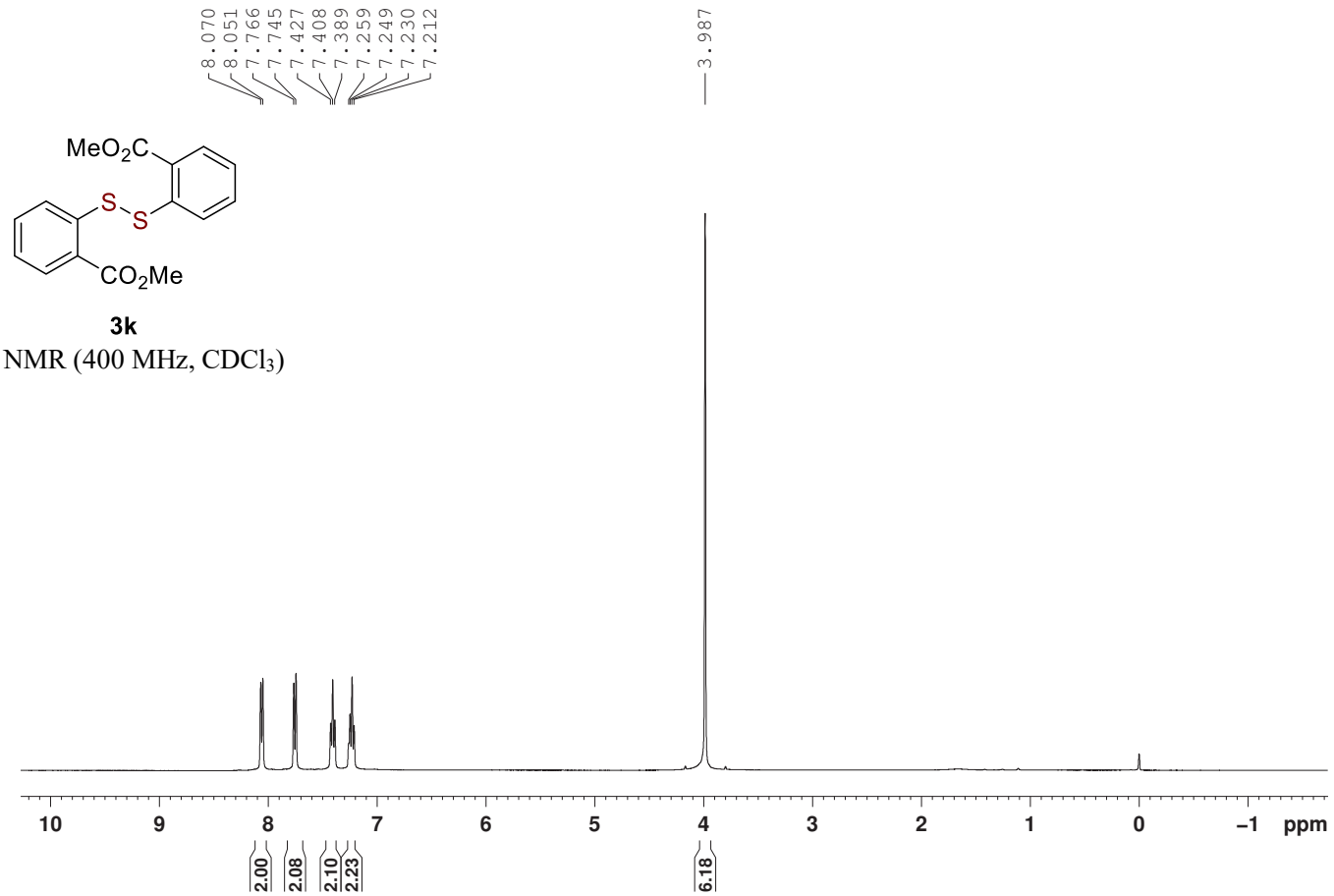
^{1}H NMR (400 MHz, $CDCl_3$)



3j

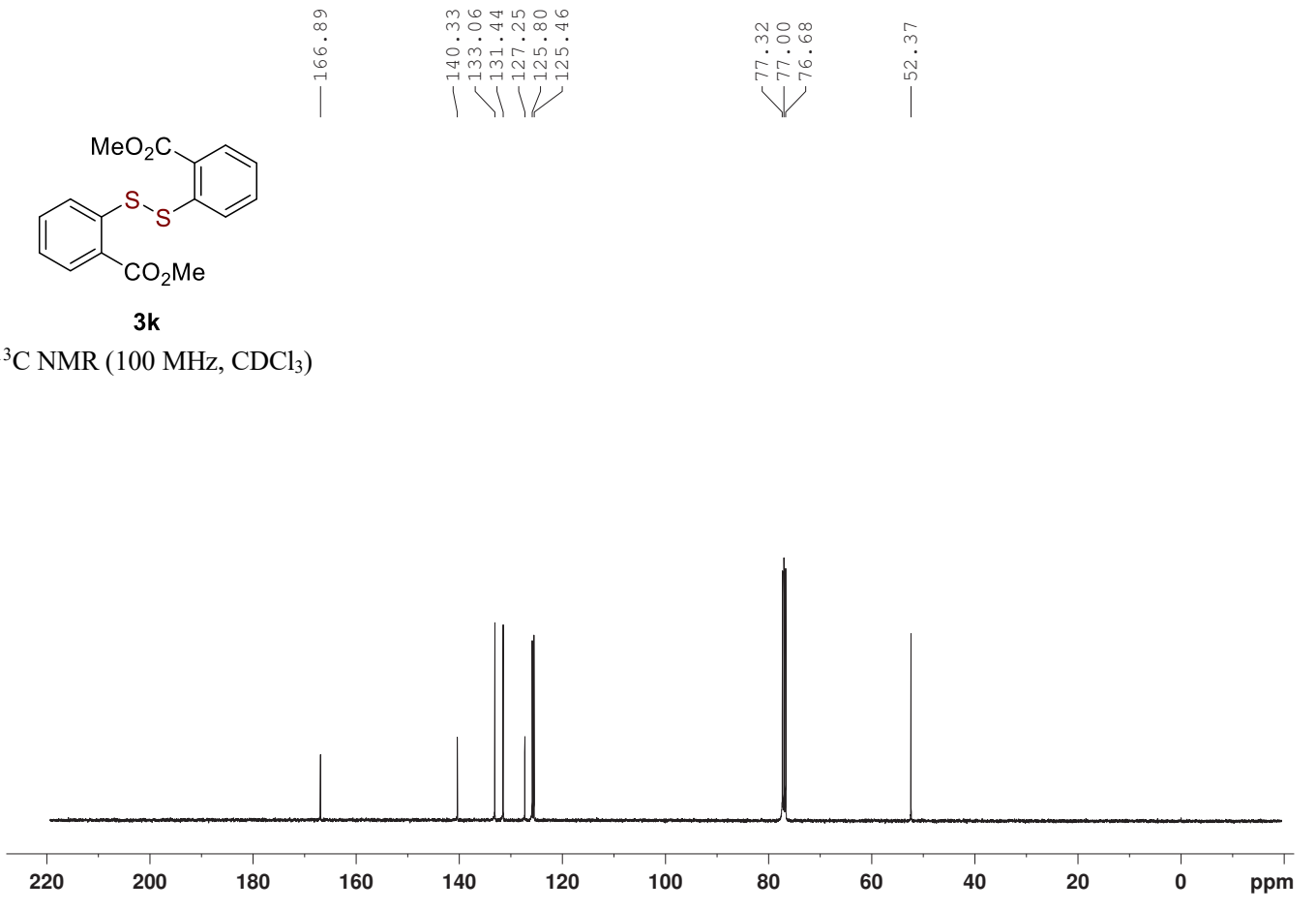
^{13}C NMR (100 MHz, $CDCl_3$)





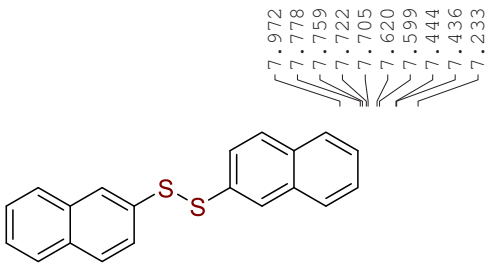
3k

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



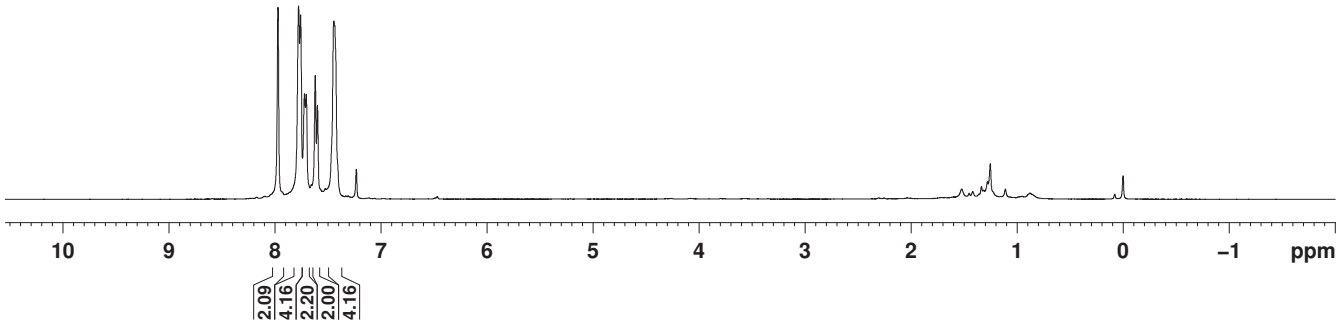
3k

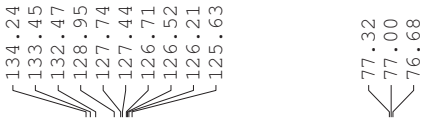
^{13}C NMR (100 MHz, $CDCl_3$)

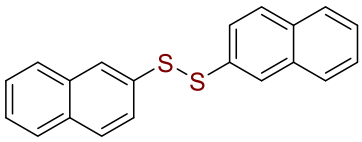


3l

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

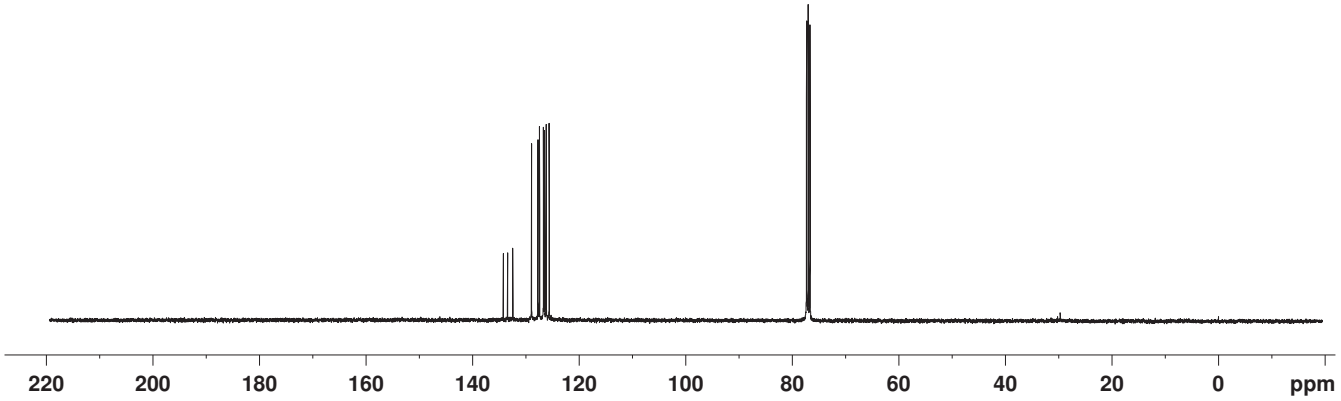


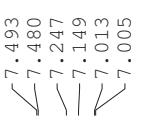


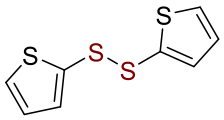


3l

^{13}C NMR (100 MHz, $CDCl_3$)

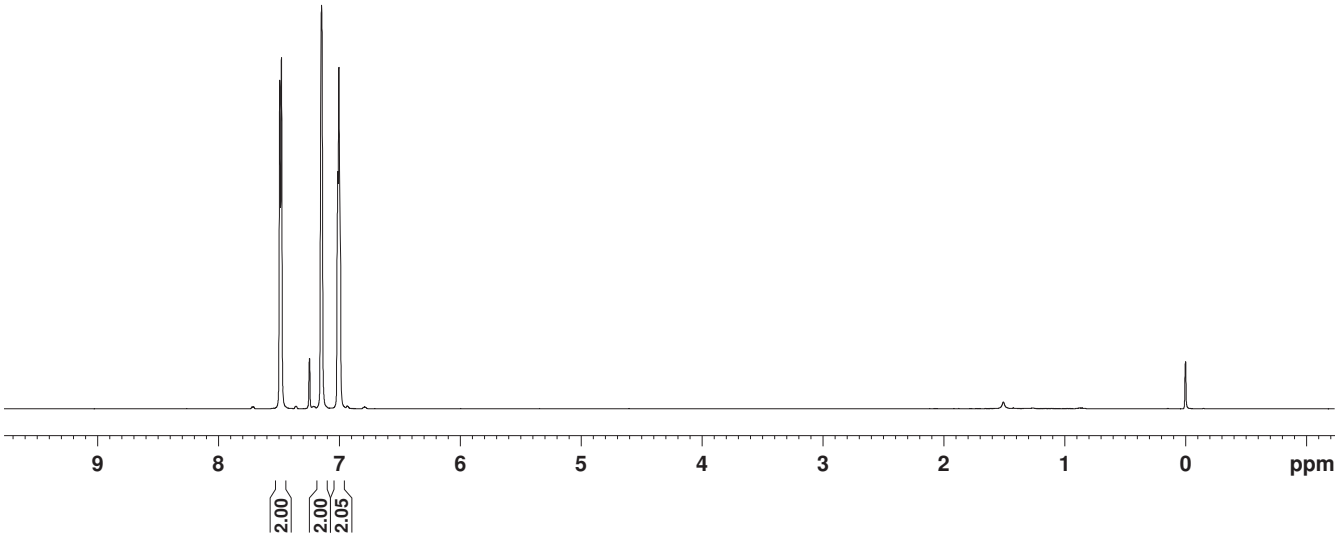


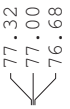


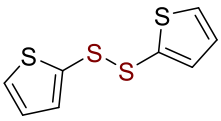


3m

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

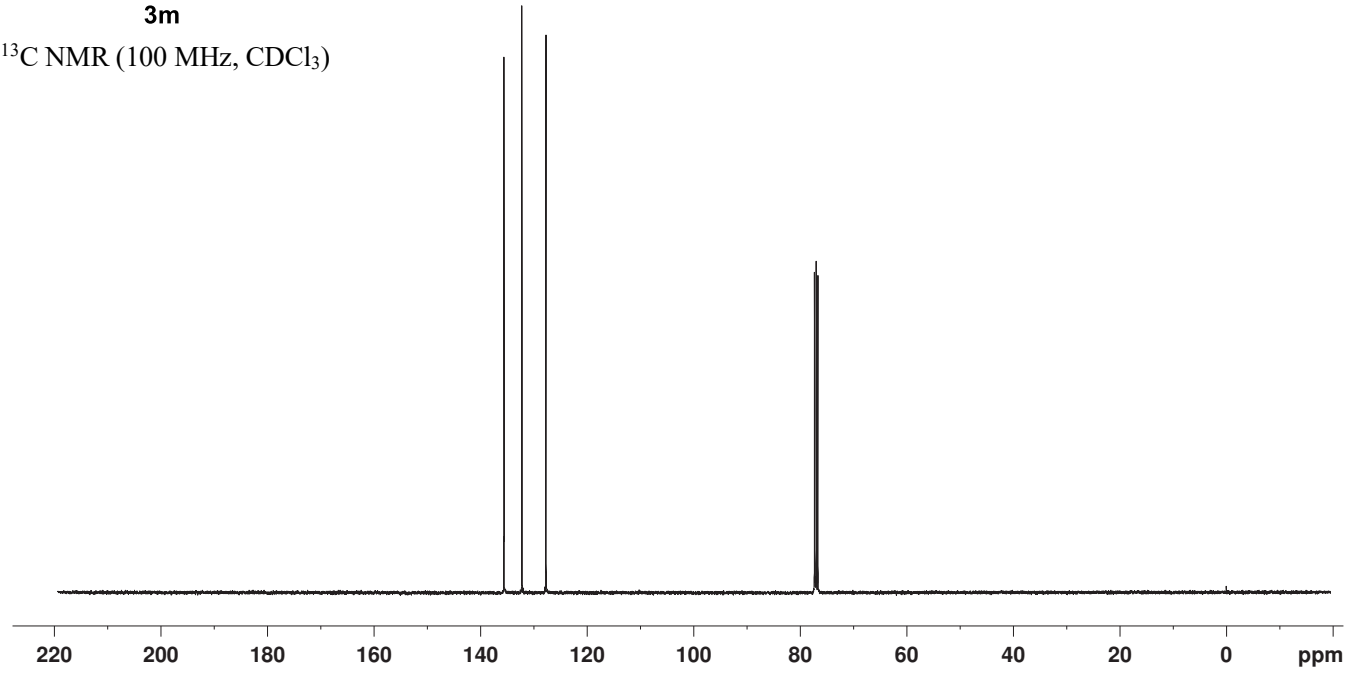


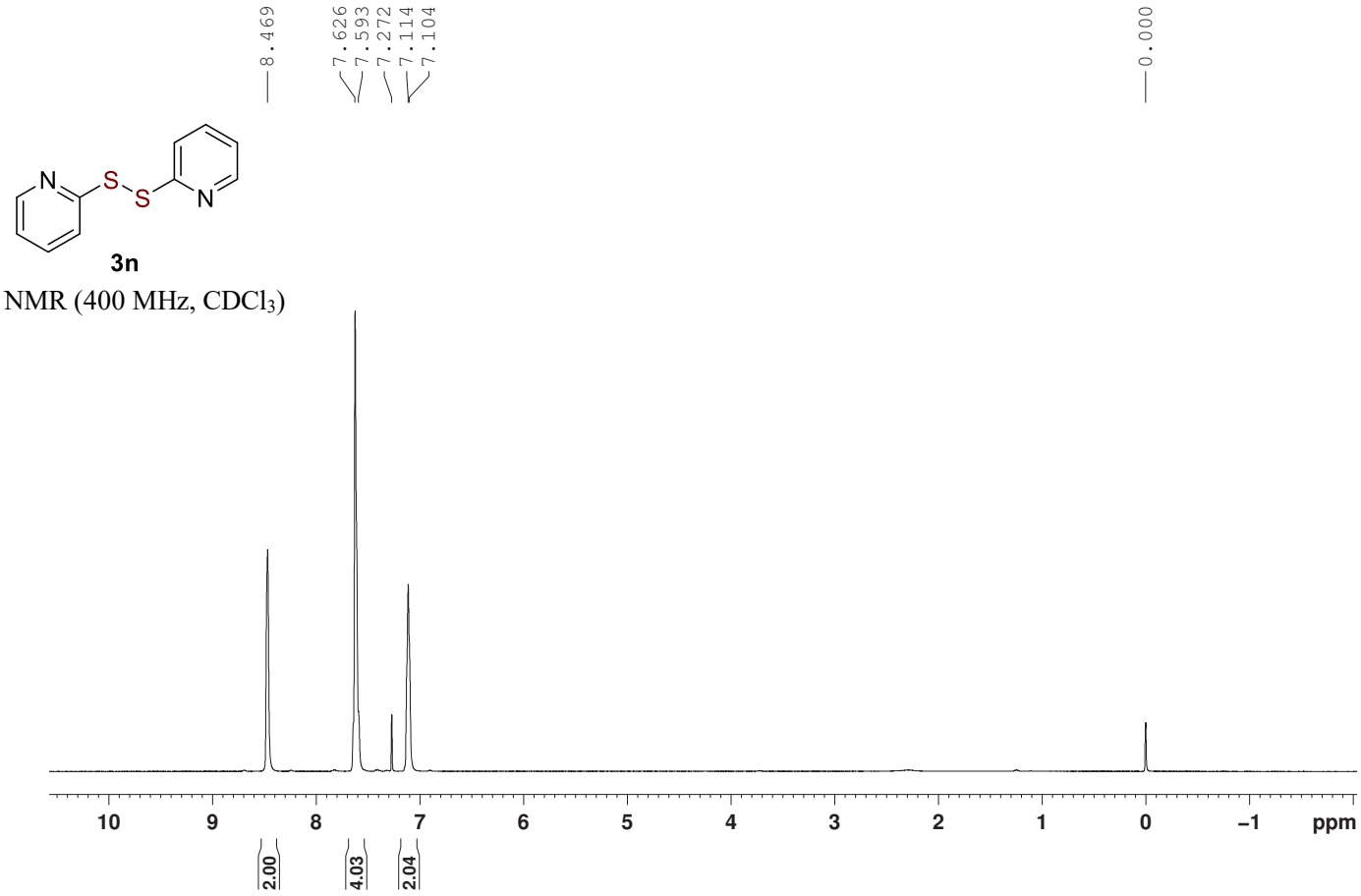




3m

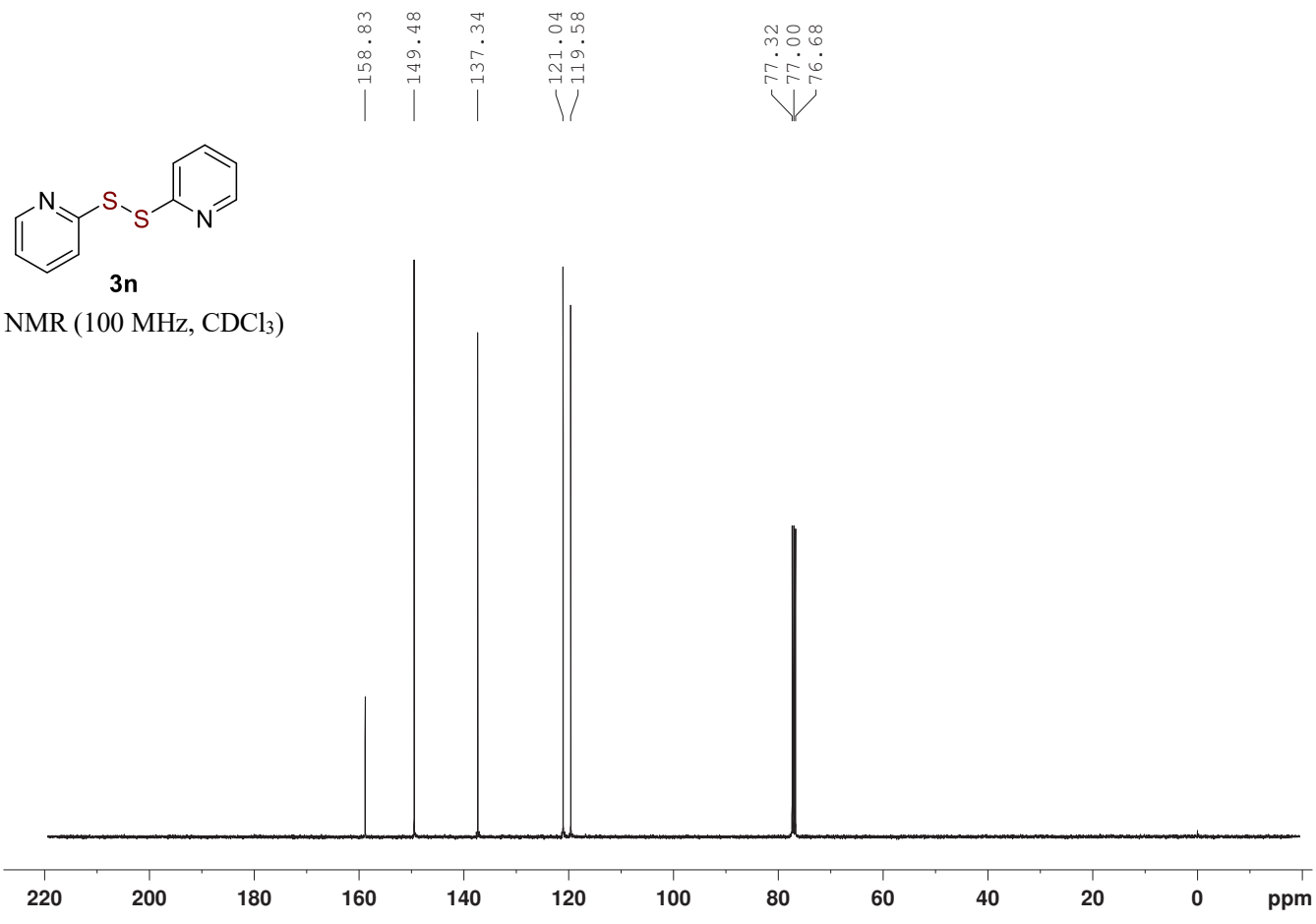
^{13}C NMR (100 MHz, $CDCl_3$)





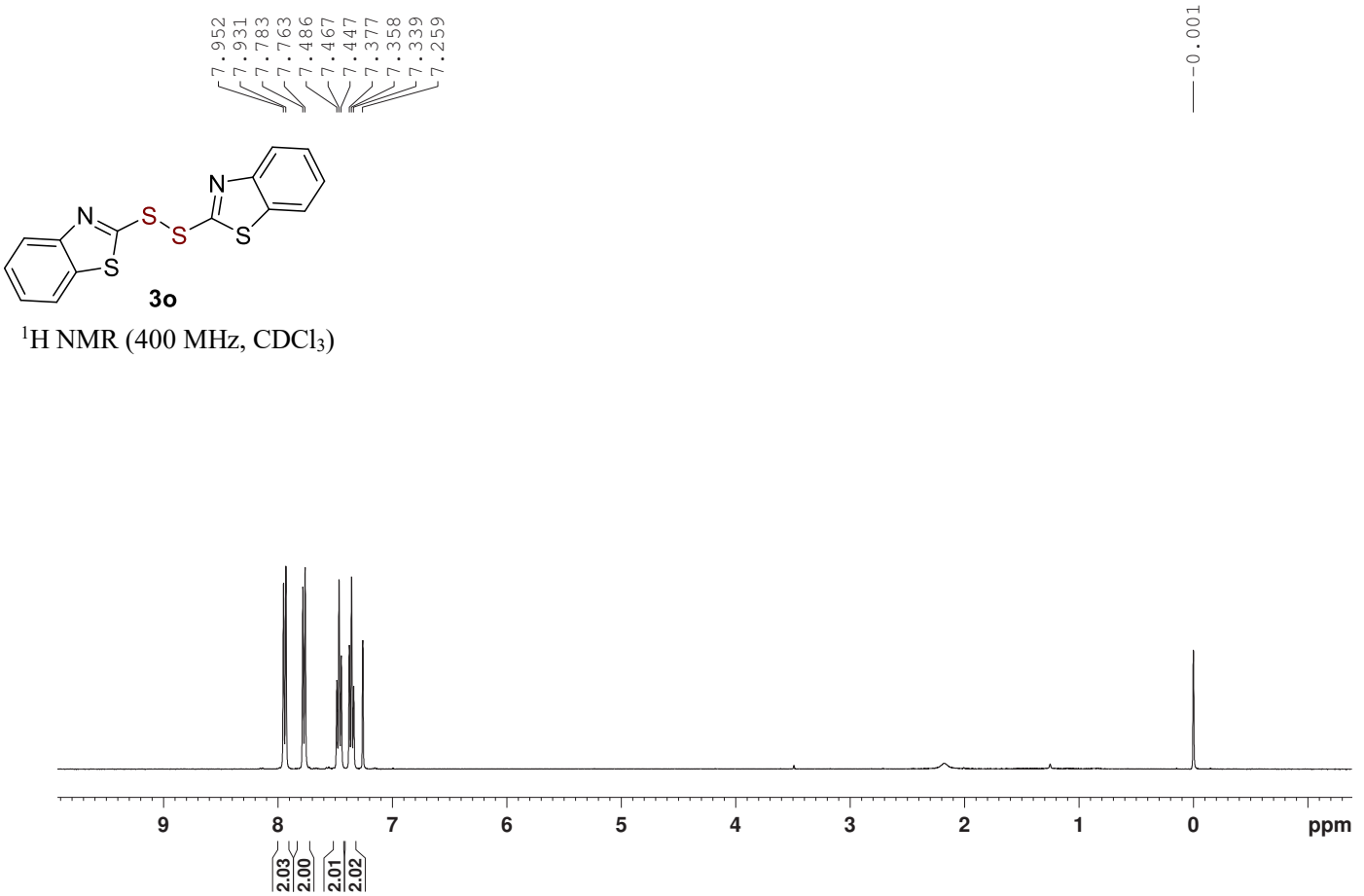
3n

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

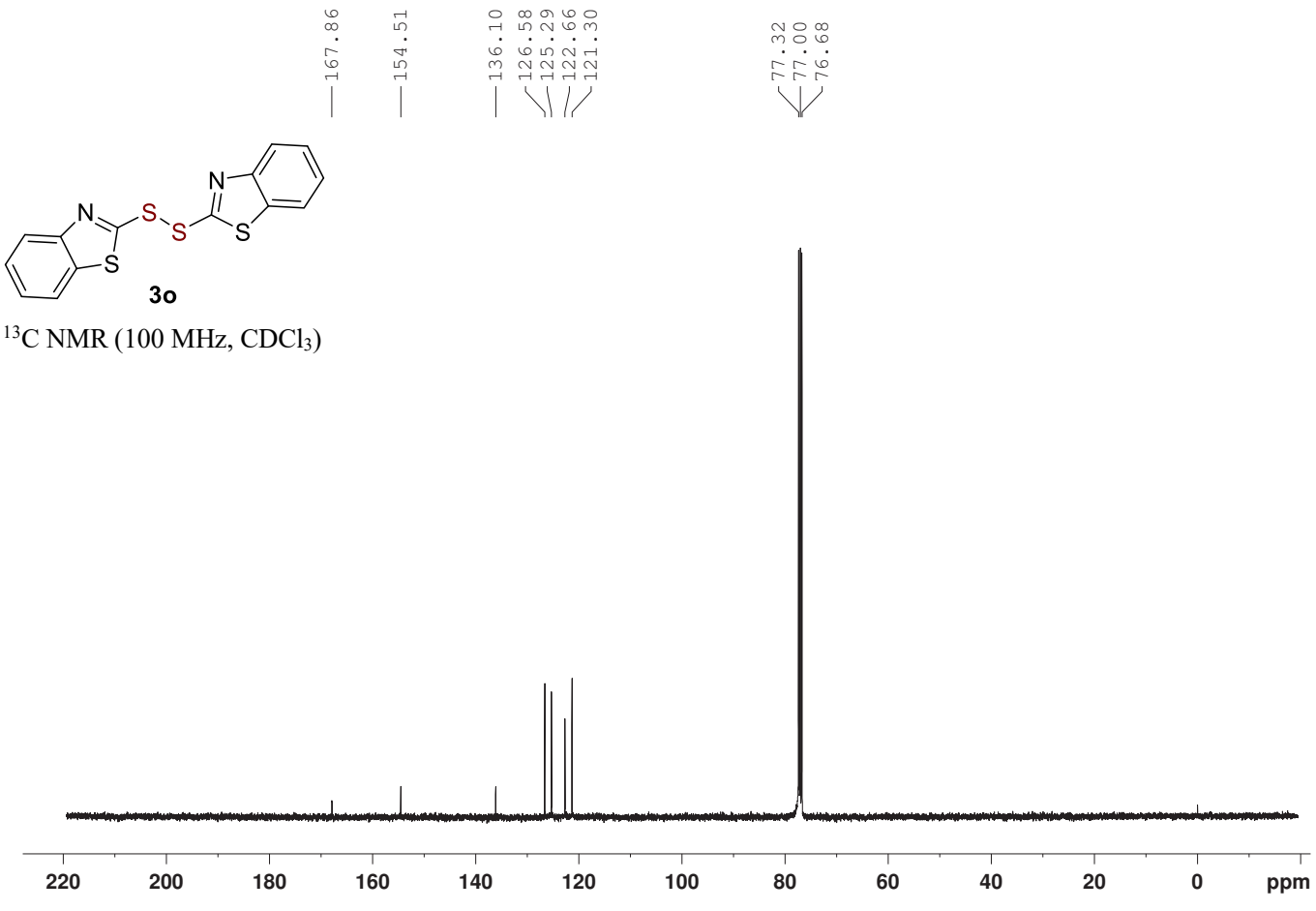


3n

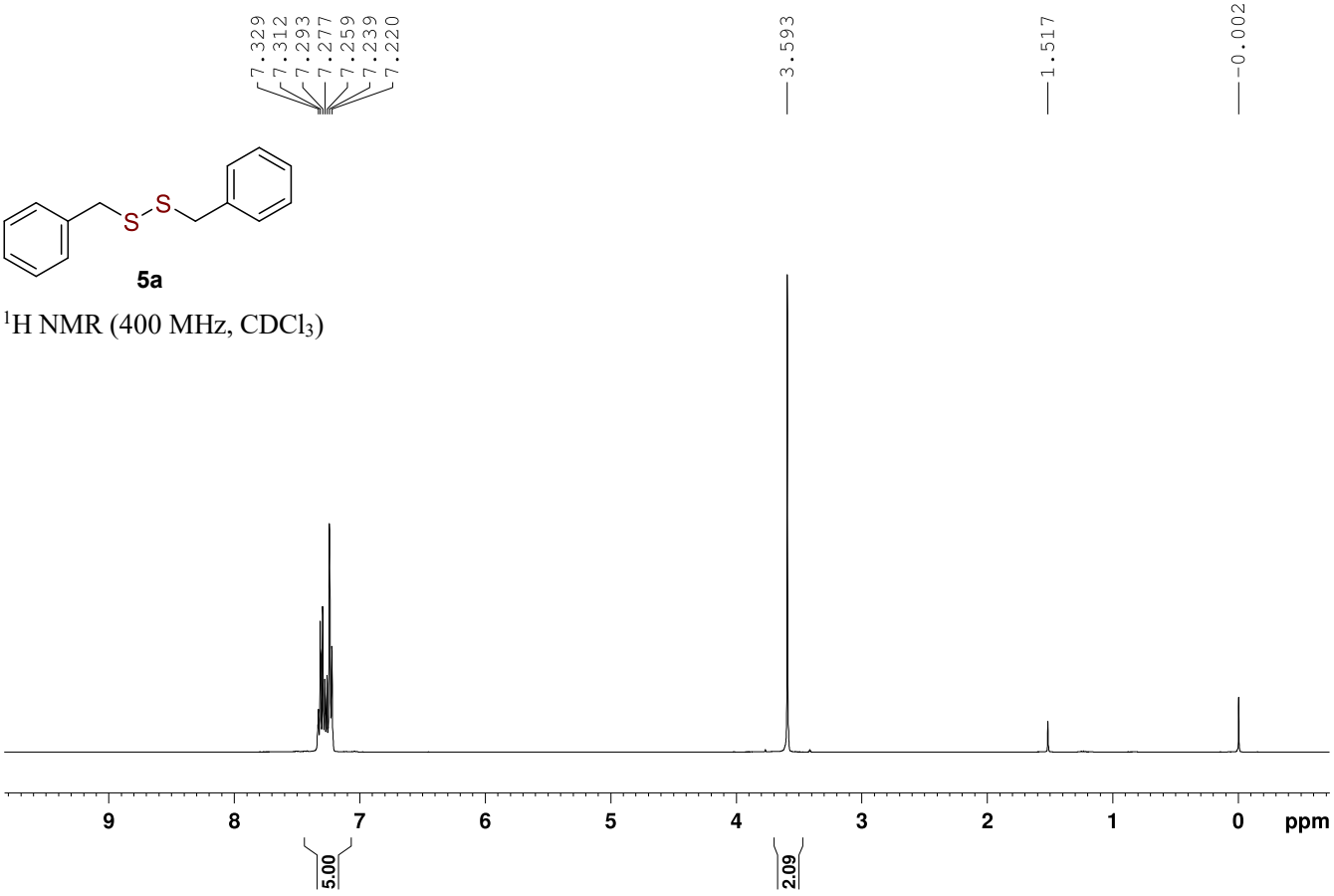
^{13}C NMR (100 MHz, $CDCl_3$)



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

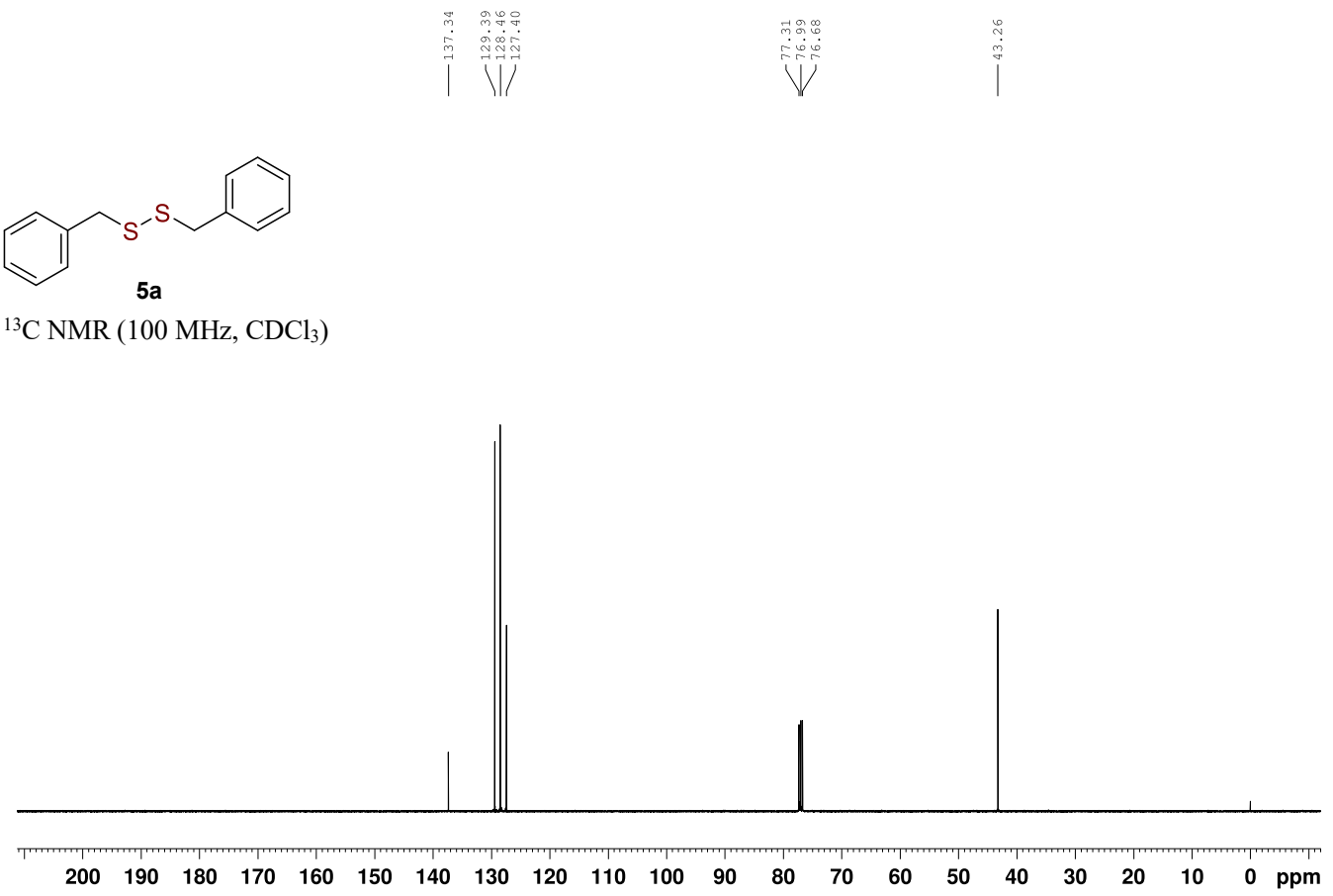


^{13}C NMR (100 MHz, $CDCl_3$)



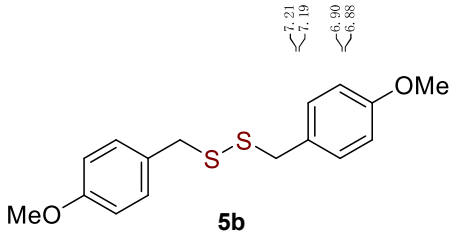
5a

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



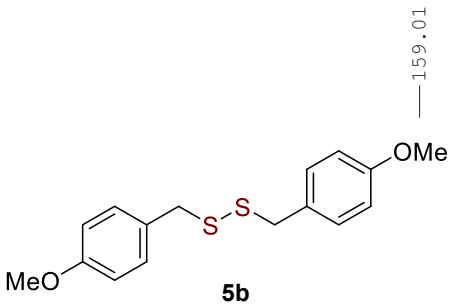
5a

^{13}C NMR (100 MHz, $CDCl_3$)



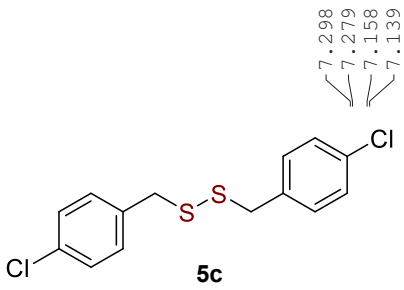
5b

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



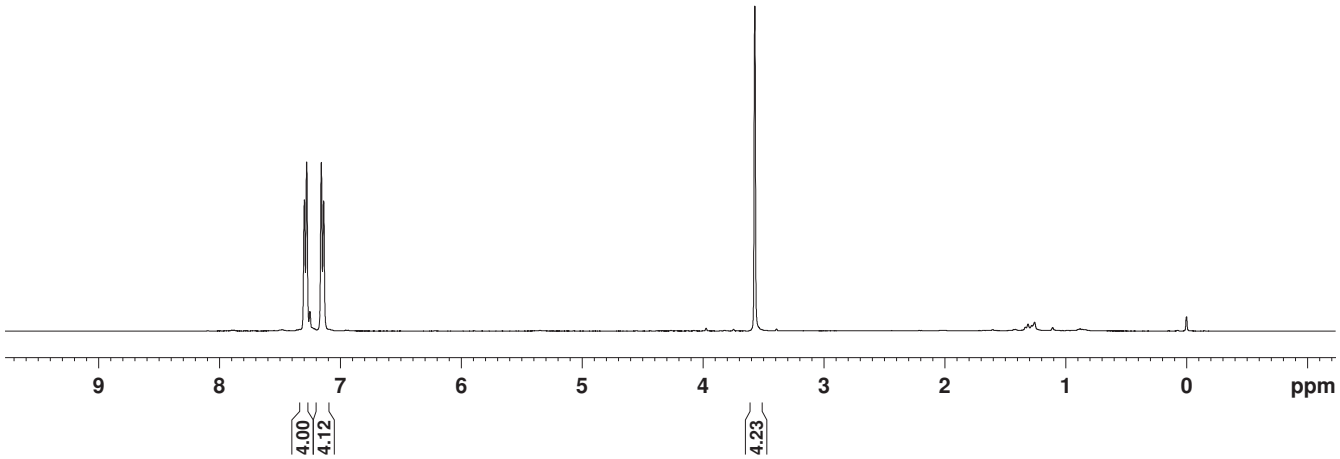
5b

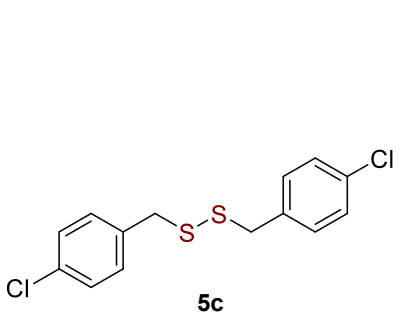
^{13}C NMR (100 MHz, $CDCl_3$)



5c

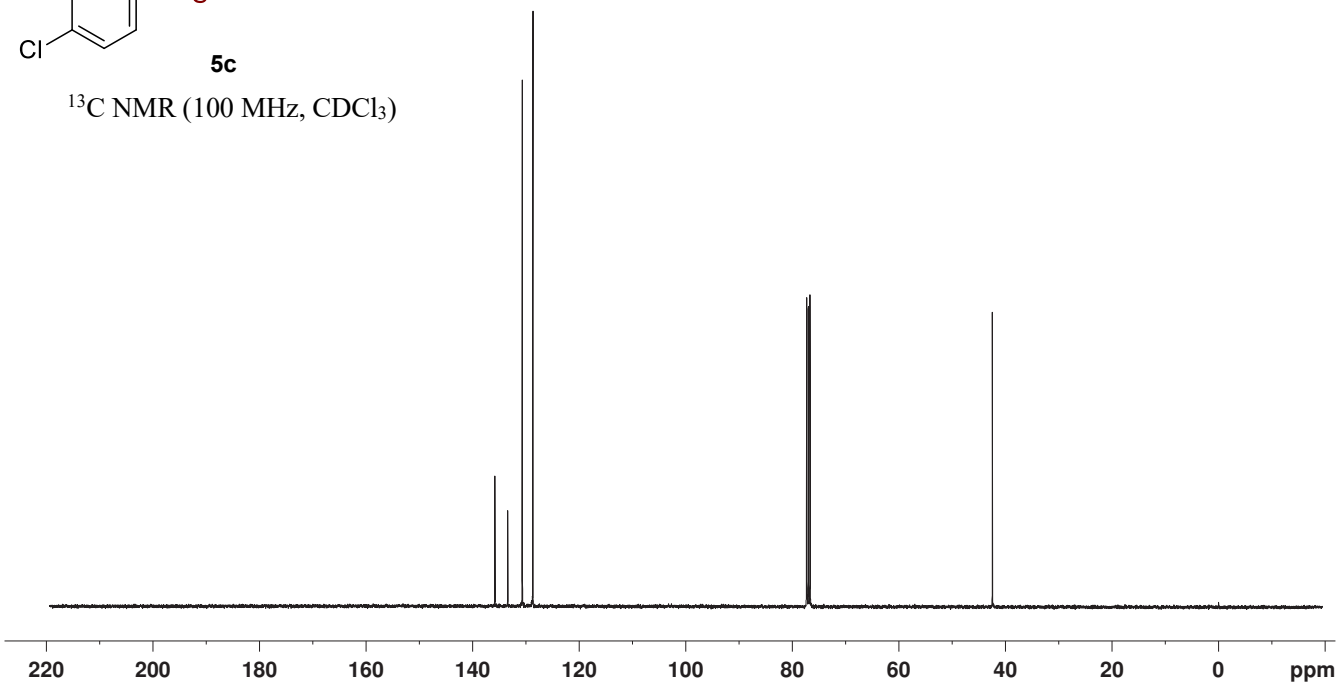
^{1}H NMR (400 MHz, $CDCl_3$)

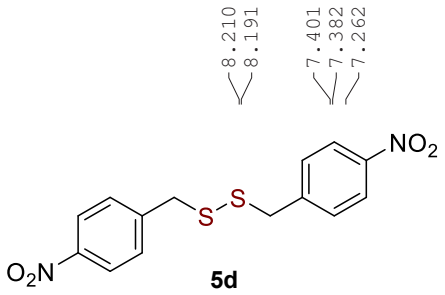




5c

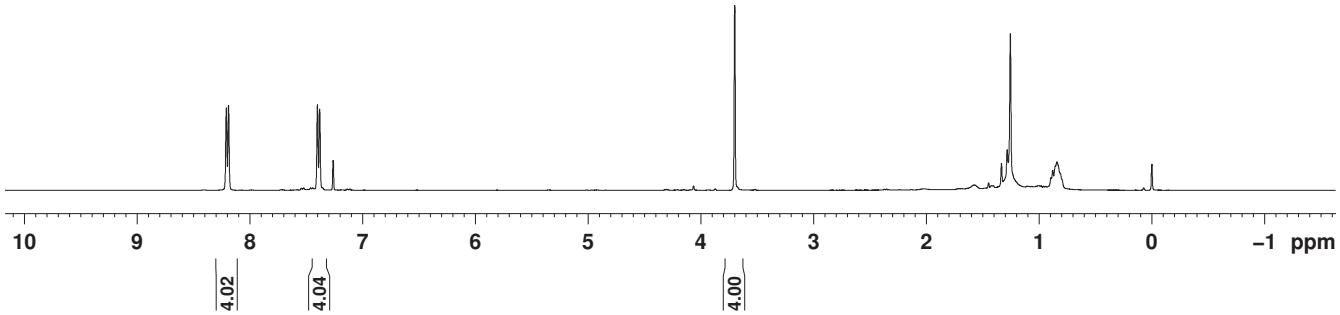
^{13}C NMR (100 MHz, $CDCl_3$)

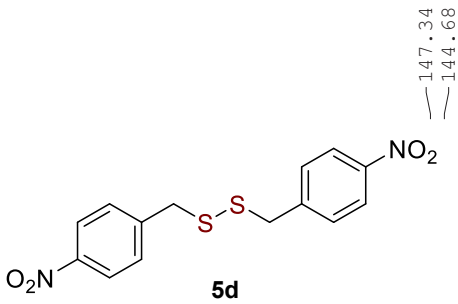




5d

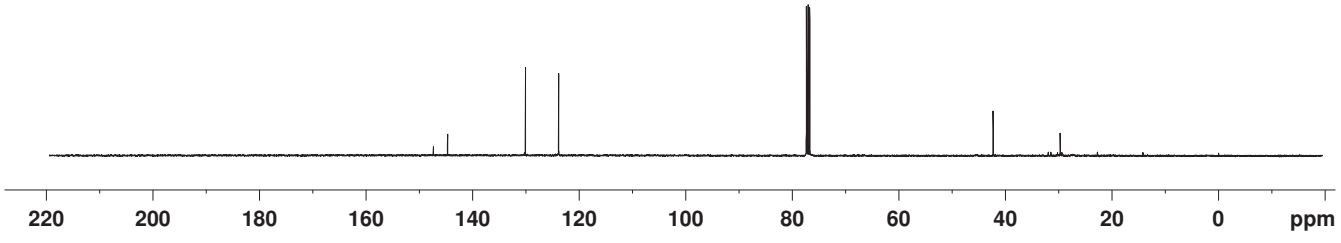
^{1}H NMR (400 MHz, $CDCl_3$)

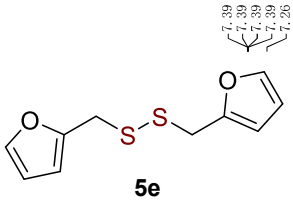




5d

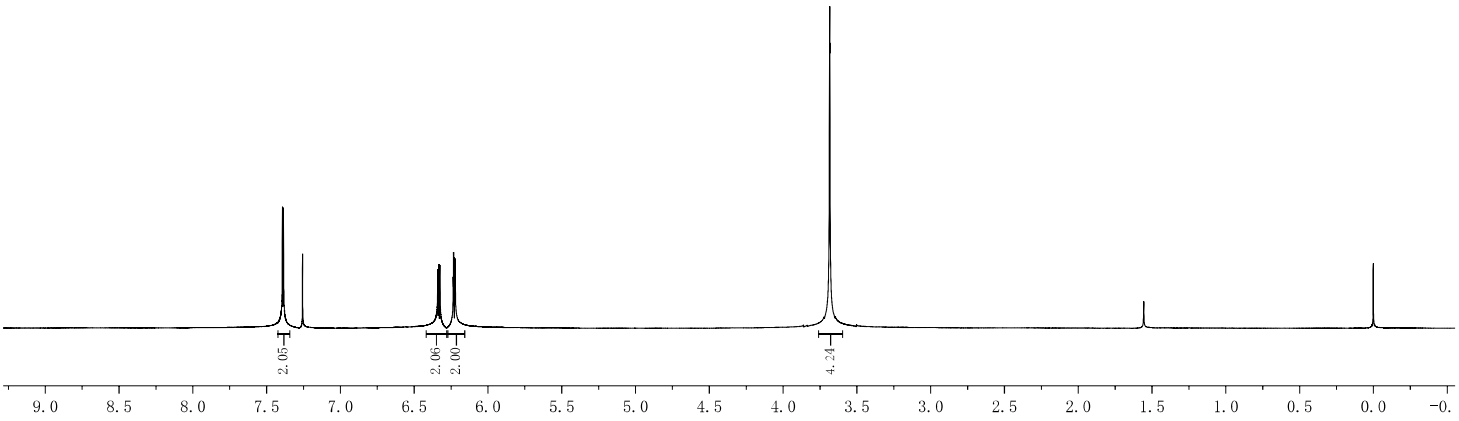
^{13}C NMR (100 MHz, $CDCl_3$)

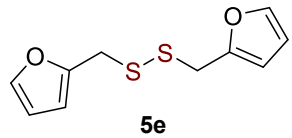




5e

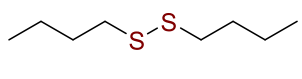
^{1}H NMR (400 MHz, $CDCl_3$)





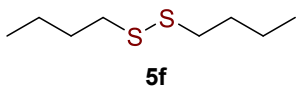
5e

^{13}C NMR (100 MHz, $CDCl_3$)



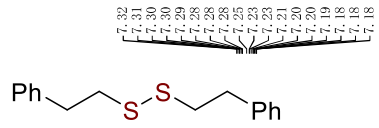
5f

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



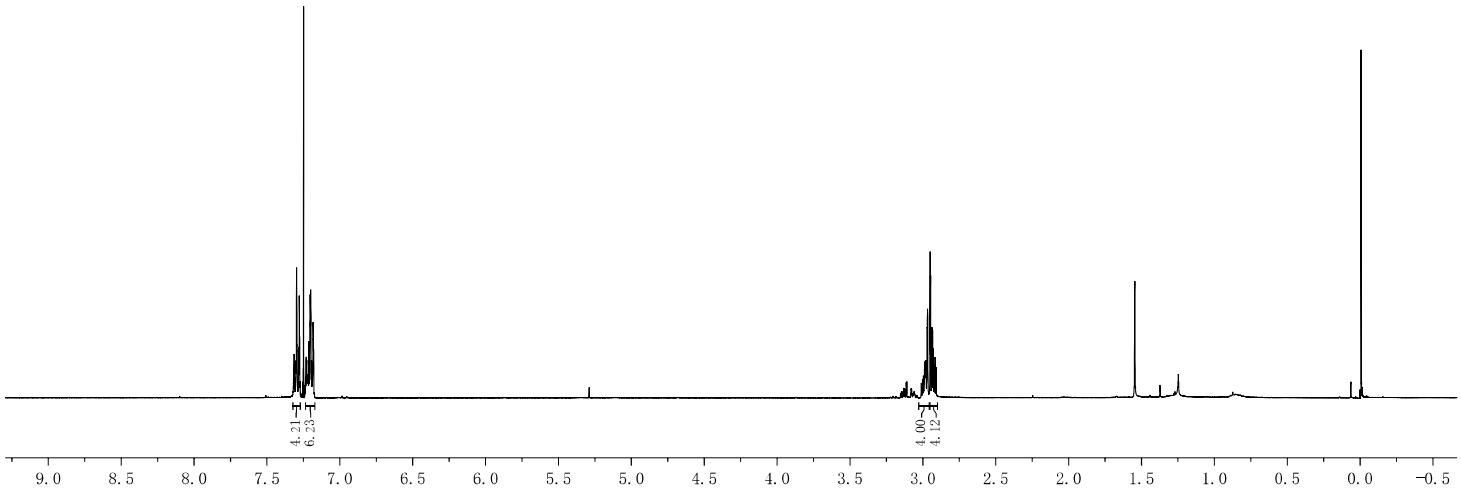
5f

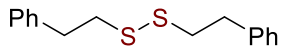
^{13}C NMR (100 MHz, $CDCl_3$)



5g

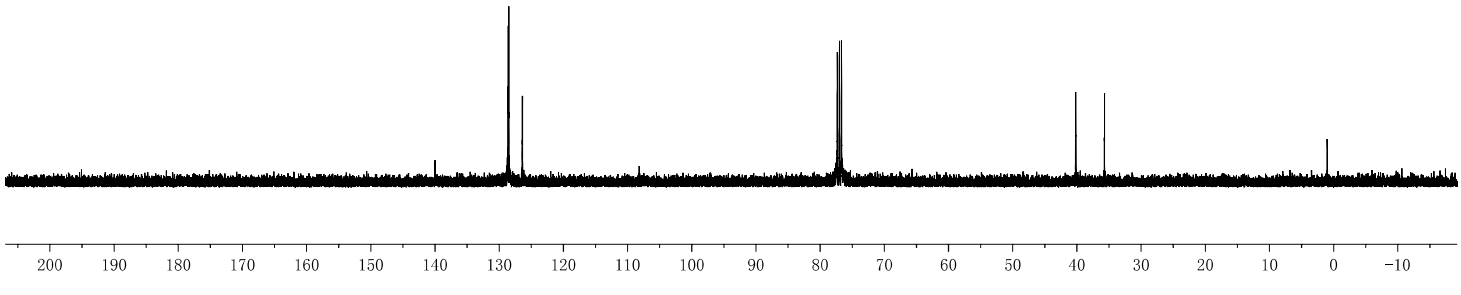
^{1}H NMR (400 MHz, $CDCl_3$)

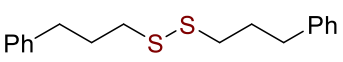




5g

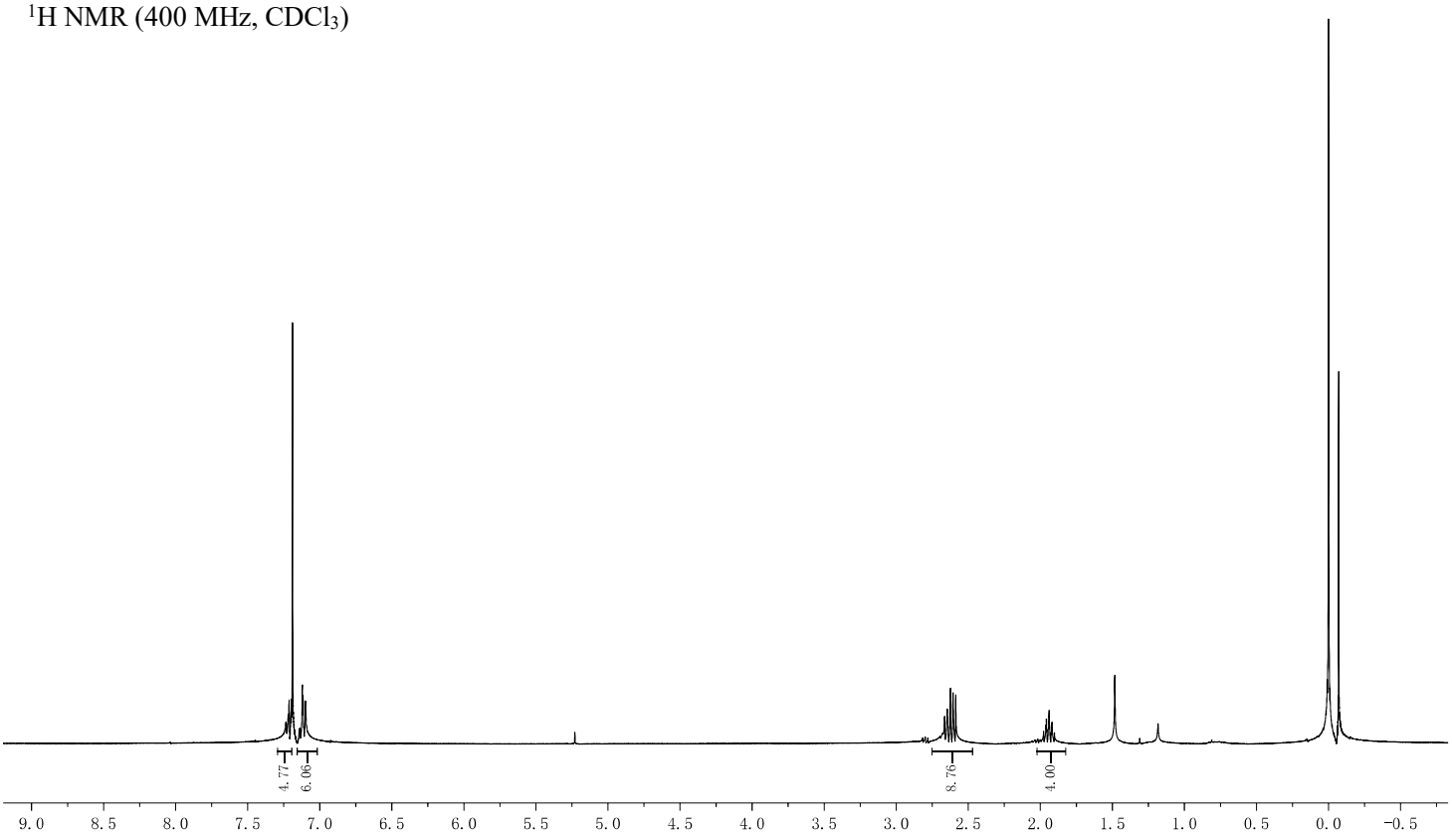
^{13}C NMR (100 MHz, $CDCl_3$)

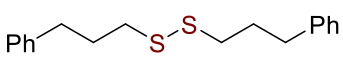




5h

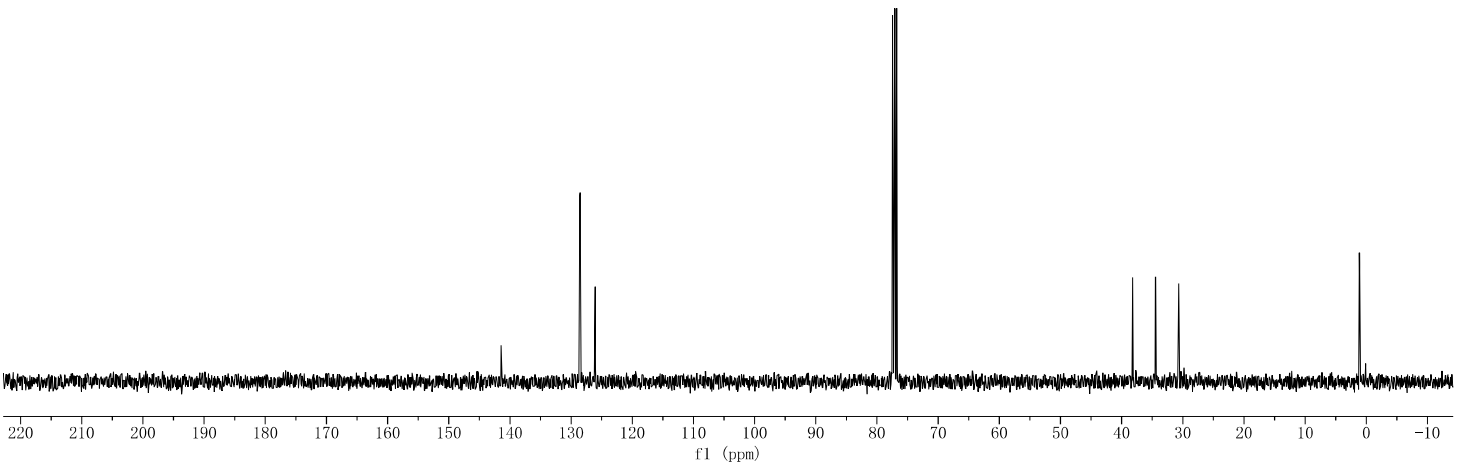
^{1}H NMR (400 MHz, $CDCl_3$)

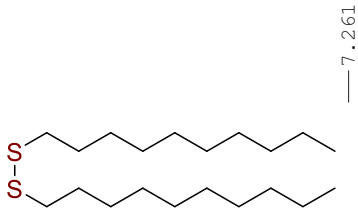




5h

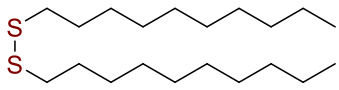
^{13}C NMR (100 MHz, $CDCl_3$)





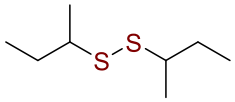
5i

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



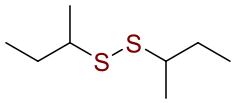
5i

^{13}C NMR (100 MHz, $CDCl_3$)



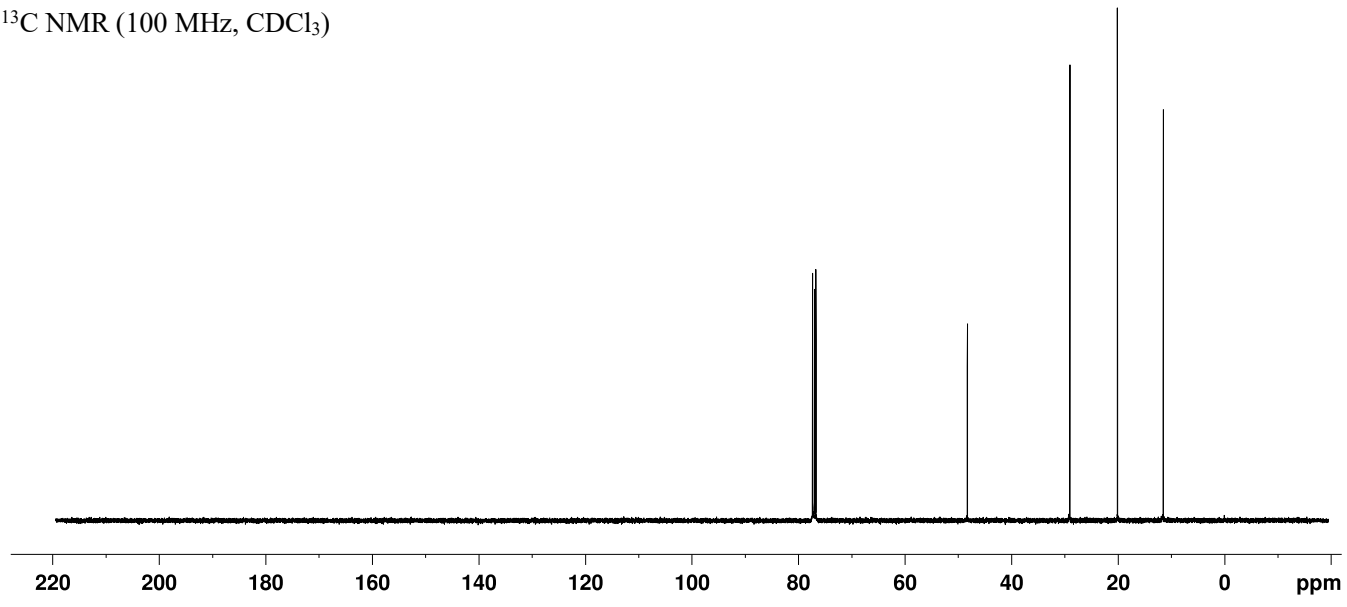
5j

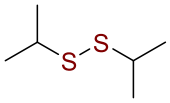
^{1}H NMR (400 MHz, $CDCl_3$)



5j

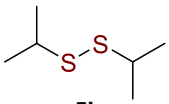
^{13}C NMR (100 MHz, $CDCl_3$)





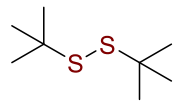
5k

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



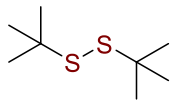
5k

^{13}C NMR (100 MHz, $CDCl_3$)



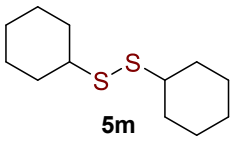
5l

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



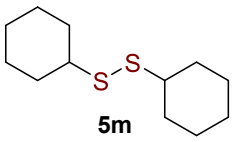
5l

^{13}C NMR (100 MHz, $CDCl_3$)



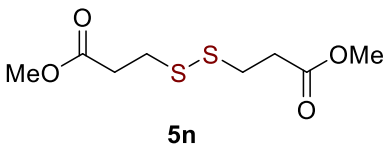
5m

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



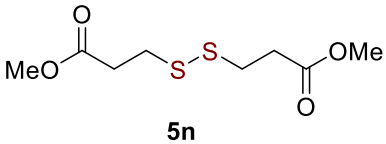
5m

^{13}C NMR (100 MHz, $CDCl_3$)



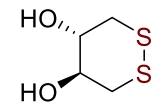
5n

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



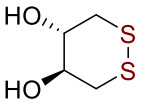
5n

^{13}C NMR (100 MHz, $CDCl_3$)



5o

^{1}H NMR (400 MHz, d^{6}-DMSO)



5o

^{13}C NMR (100 MHz, d^{6}-DMSO)