Iodine-catalyzed synthesis of sulfur-bridged enaminones and chromones via double C(sp^2)-H thiolation

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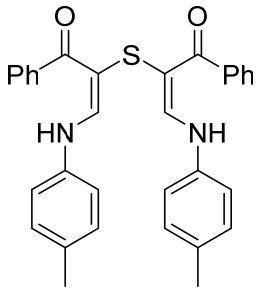
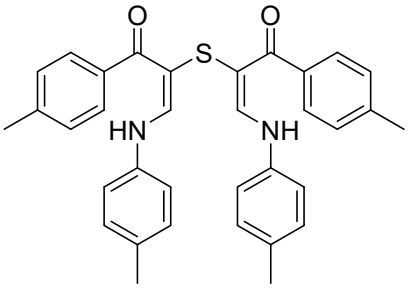
General experimental information

All experiments were carried out under air atmosphere. The enaminones **1** were synthesized following literature process,[1-2] other chemicals and solvents used in the experiments were obtained from commercial sources and used directly without further treatment. ^{1}H and ^{13}C NMR spectra were recorded in 400 MHz apparatus in $CDCl_3$. The frequencies for ^{1}H NMR and ^{13}C NMR test are 400 MHz and 100 MHz, respectively. The chemical shifts were reported in ppm with TMS as internal standard. Melting points were tested in X-4A instrument without correcting temperature and the HRMS data for all new products were obtained under ESI model with TOF analyzer.

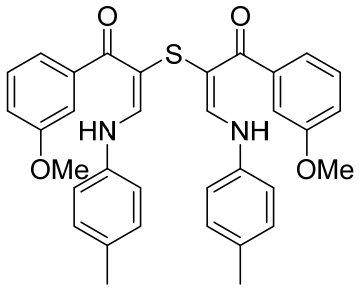
Characterization data of all products



(2E,2'E)-2,2'-Thiobis(1-phenyl-3-(p-tolylamino)prop-2-en-1-one) (**4a**). Yellow solid; mp 247-250 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.21 (d, *J* = 13.6 Hz, 2 H), 8.03 (d, *J* = 13.7 Hz, 2 H), 7.59-7.57 (m, 4 H), 7.52-7.43 (m, 6 H), 7.12-7.06 (m, 8 H), 2.30 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.1, 153.2, 140.0, 137.5, 133.9, 130.6, 130.2, 128.6, 128.4, 116.8, 107.4, 20.8; ESI-HRMS Calcd for $C_{32}H_{29}N_2O_2S$ $[M + H]^+$ 505.1944, found 505.1945.

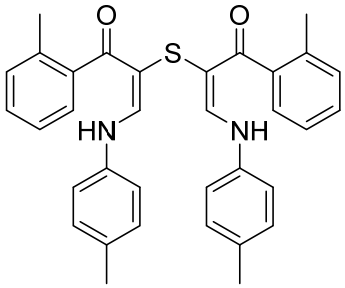


(2E,2'E)-2,2'-Thiobis(1-(p-tolyl)-3-(p-tolylamino)prop-2-en-1-one) (**4b**). Yellow solid; mp 210-214 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.23 (d, *J* = 13.6 Hz, 2 H), 8.05 (d, *J* = 13.6 Hz, 2 H), 7.50 (d, *J* = 7.8 Hz, 4 H), 7.25-7.23 (m, 4 H), 7.12-7.07 (m, 8 H), 2.42 (s, 6 H), 2.30 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.1, 152.8, 141.0, 137.7, 137.1, 133.7, 130.2, 129.0, 128.9, 116.8, 107.4, 21.5, 20.8; ESI-HRMS Calcd for $C_{34}H_{33}N_2O_2S$ $[M + H]^+$ 533.2257, found 533.2256.

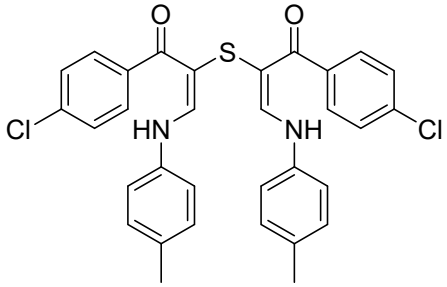


(2E,2'E)-2,2'-Thiobis(1-(3-methoxyphenyl)-3-(p-tolylamino)prop-2-en-1-one) (**4c**). Yellow solid; mp 200-201 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.19 (d, *J* = 13.7 Hz, 2

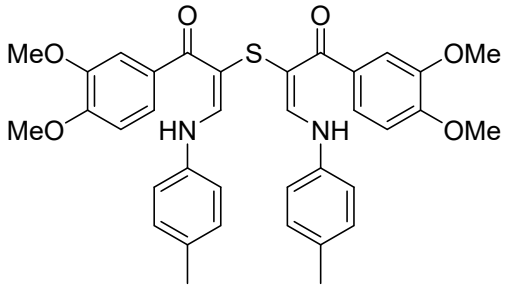
H), 8.07 (d, *J* = 13.7 Hz, 2 H), 7.36 (t, *J* = 7.9 Hz, 2 H), 7.14-7.02 (m, 14 H), 3.83 (s, 6 H), 2.30 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 194.8, 159.6, 153.3, 141.4, 137.5, 134.0, 130.2, 129.3, 121.0, 116.8, 116.6, 113.6, 107.3, 55.4, 20.8; IR (KBr, cm^{-1}) 3141, 3026, 2925, 2858, 1629, 1520, 1464, 1349, 1269, 1120, 1042, 813, 792; ESI-HRMS Calcd for $C_{34}H_{33}N_2O_4S$ $[M + H]^+$ 565.2156, found 565.2154.



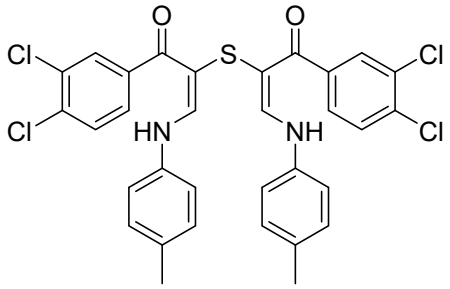
(2E,2'E)-2,2'-Thiobis(1-(o-tolyl)-3-(p-tolylamino)prop-2-en-1-one) (**4d**). Yellow solid; mp 205-208 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.02 (d, *J* = 13.3 Hz, 2 H), 7.81 (d, *J* = 13.7 Hz, 2 H), 7.34-7.20 (m, 8 H), 7.08-6.98 (m, 8 H), 2.27 (s, 6 H), 2.18 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 196.2, 153.6, 139.9, 137.2, 135.3, 134.2, 130.7, 130.2, 129.2, 127.0, 125.5, 116.9, 109.3, 20.8, 19.2; ESI-HRMS Calcd for $C_{34}H_{33}N_2O_2S$ $[M + H]^+$ 533.2257, found 533.2258.



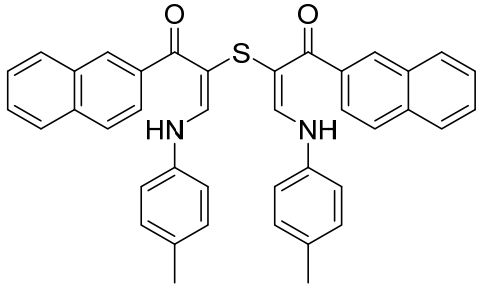
(2E,2'E)-2,2'-Thiobis(1-(4-chlorophenyl)-3-(p-tolylamino)prop-2-en-1-one) (**4e**). Yellow solid; mp 254-256 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.19 (d, *J* = 13.6 Hz, 2 H), 7.98 (d, *J* = 13.7 Hz, 2 H), 7.54-7.42 (m, 8 H), 7.15-7.05 (m, 8 H), 2.32 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 193.7, 153.0, 138.3, 137.3, 136.9, 134.3, 130.3, 130.0, 128.7, 116.8, 107.2, 20.8; ESI-HRMS Calcd for $C_{32}H_{27}Cl_2N_2O_2S$ $[M + H]^+$ 573.1165, found 573.1165.



(2E,2'E)-2,2'-Thiobis(1-(3,4-dimethoxyphenyl)-3-(p-tolylamino)prop-2-en-1-one) (**4f**). Yellow solid; mp 230-232 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.23 (d, *J* = 13.6 Hz, 2 H), 8.09 (d, *J* = 13.6 Hz, 2 H), 7.23-7.08 (m, 12 H), 6.92 (d, *J* = 8.2 Hz, 2 H), 3.96 (s, 6 H), 3.91 (s, 6 H), 2.31 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 194.4, 152.2, 151.5, 148.9, 137.8, 133.6, 132.5, 130.2, 122.6, 116.6, 112.0, 110.3, 107.0, 56.0, 20.8; IR (KBr, cm^{-1}) 3016, 2931, 2853, 2360, 1643, 1576, 1459, 1336, 1269, 1131, 1019, 864, 797; ESI-HRMS Calcd for $C_{36}H_{37}N_2O_6S$ $[M + H]^+$ 625.2367, found 625.2367.

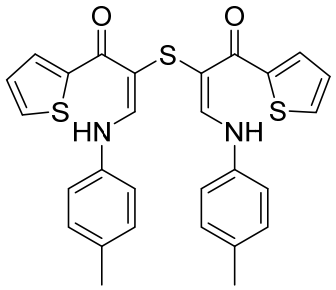


(2E,2'E)-2,2'-Thiobis(1-(3,4-dichlorophenyl)-3-(p-tolylamino)prop-2-en-1-one) (**4g**). Yellow solid; mp 233-235 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.18 (d, *J* = 13.2 Hz, 2 H), 7.98-7.94 (m, 2 H), 7.67 (s, 2 H), 7.54-7.52 (m, 2 H), 7.39 (d, *J* = 8.1 Hz, 2 H), 7.25 (d, *J* = 2.5 Hz, 1 H), 7.16-7.06 (m, 7 H), 2.32 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.1, 153.1, 139.7, 137.1, 135.0, 134.6, 133.1, 130.6, 130.4, 127.7, 116.9, 106.9, 20.8; ESI-HRMS Calcd for $C_{32}H_{25}Cl_4N_2O_2S$ $[M + H]^+$ 641.0385, found 641.0388.

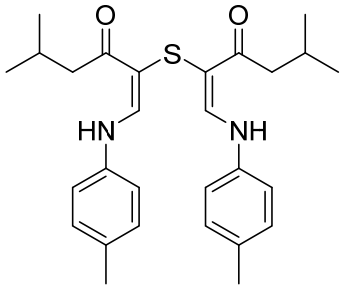


(2E,2'E)-2,2'-Thiobis(1-(naphthalen-2-yl)-3-(p-tolylamino)prop-2-en-1-one) (**4h**).

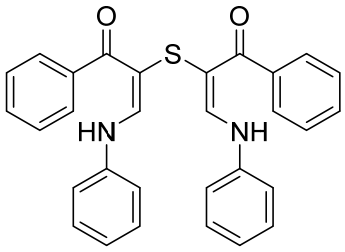
Yellow solid; mp 238-241 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.32 (d, *J* = 13.7 Hz, 2 H), 8.16-8.08 (m, 4 H), 7.92 (t, *J* = 6.8 Hz, 6 H), 7.71 (d, *J* = 9.7 Hz, 2 H), 7.61-7.54 (m, 4 H), 7.09 (s, 8 H), 2.29 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.1, 153.2, 137.5, 137.3, 134.4, 133.9, 132.6, 130.2, 128.8, 128.3, 127.8, 127.4, 126.8, 125.8, 116.8, 107.6, 20.8; ESI-HRMS Calcd for $C_{40}H_{33}N_2O_2S$ $[M + H]^+$ 605.2257, found 605.2257.



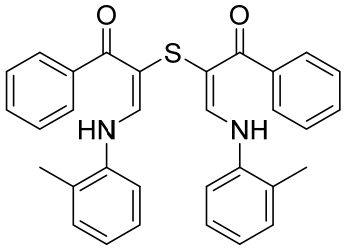
(2E,2'E)-2,2'-Thiobis(1-(thiophen-2-yl)-3-(p-tolylamino)prop-2-en-1-one) (**4i**). Yellow solid; mp 204-206 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.22 (d, *J* = 13.6 Hz, 2 H), 8.39 (d, *J* = 13.6 Hz, 2 H), 7.59-7.53 (m, 4 H), 7.15-7.13 (m, 10 H), 2.31 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 185.8, 151.1, 143.5, 137.7, 133.8, 131.1, 131.0, 130.2, 127.4, 116.9, 106.6, 20.8; ESI-HRMS Calcd for $C_{28}H_{25}N_2O_2S_3$ $[M + H]^+$ 517.1073, found 517.1073.



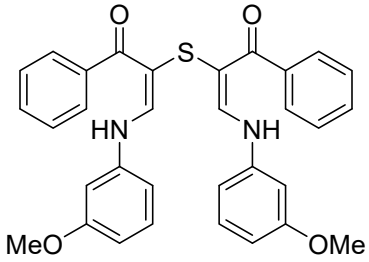
(2E,2'E)-2,2'-Thiobis(5-methyl-1-(p-tolylamino)hex-1-en-3-one) (**4j**). Yellow solid; mp 124-127 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.18 (d, *J* = 12.8 Hz, 2 H), 7.89 (d, *J* = 13.0 Hz, 2 H), 7.18-7.03 (m, 8 H), 2.75 (d, *J* = 6.9 Hz, 4 H), 2.33 (s, 6 H), 2.26-2.19 (m, 2 H), 0.98 (d, *J* = 6.6 Hz, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): 202.5, 152.7, 136.6, 135.0, 130.4, 117.3, 102.1, 49.1, 25.4, 22.8, 20.8; IR (KBr, cm^{-1}) 3037, 2963, 2925, 2864, 1869, 1613, 1571, 1469, 1349, 1291, 1160, 1056, 808; ESI-HRMS Calcd for $C_{28}H_{37}N_2O_2S$ $[M + H]^+$ 465.2570, found 465.2570.



(2E,2'E)-2,2'-Thiobis(1-phenyl-3-(phenylamino)prop-2-en-1-one) (**4k**). Yellow solid; mp 223-225 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.26 (d, *J* = 13.2 Hz, 2 H), 8.07 (d, *J* = 13.6 Hz, 2 H), 7.60-7.30 (m, 14 H), 7.19-7.06 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.3, 153.1, 139.9, 139.9, 130.8, 129.7, 128.7, 128.4, 124.2, 116.8, 107.9; IR (KBr, cm^{-1}) 3021, 1629, 1581, 1482, 1349, 1272, 1117, 1024, 874, 789; ESI-HRMS Calcd for $C_{30}H_{25}N_2O_2S$ $[M + H]^+$ 477.1631, found 477.1632.

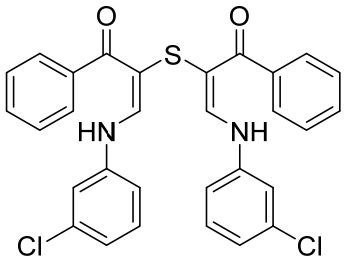


(2E,2'E)-2,2'-Thiobis(1-phenyl-3-(o-tolylamino)prop-2-en-1-one) (**4l**). Yellow solid; mp 188-191 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.57 (d, *J* = 13.3 Hz, 2 H), 7.99 (d, *J* = 13.4 Hz, 2 H), 7.52-7.38 (m, 10 H), 7.22 (d, *J* = 7.3 Hz, 2 H), 7.13-7.02 (m, 4 H), 6.85 (d, *J* = 7.8 Hz, 2 H), 2.65 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 194.9, 155.8, 140.3, 139.0, 131.5, 130.4, 129.5, 128.5, 128.3, 126.9, 124.8, 118.0, 107.7, 18.2; ESI-HRMS Calcd for $C_{32}H_{29}N_2O_2S$ $[M + H]^+$ 505.1944, found 505.1945.

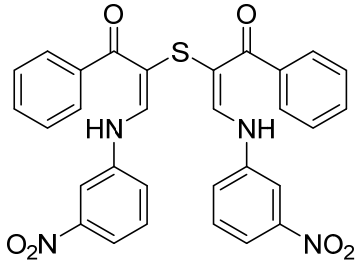


(2E,2'E)-2,2'-Thiobis(3-((3-methoxyphenyl)amino)-1-phenylprop-2-en-1-one) (**4m**). Brown solid; mp 180-184 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.19 (d, *J* = 13.5 Hz, 2 H), 8.03 (d, *J* = 13.5 Hz, 2 H), 7.60-7.44 (m, 10 H), 7.22 (t, *J* = 8.1 Hz, 2 H),

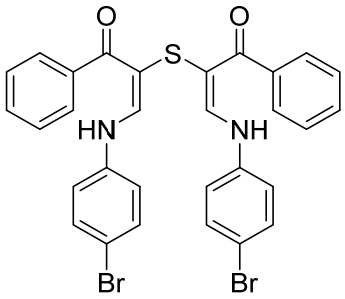
6.78-6.73 (m, 4 H), 6.63 (d, J = 8.2 Hz, 2 H), 3.80 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.3, 160.8, 153.0, 141.2, 139.8, 130.9, 130.5, 128.7, 128.4, 109.4, 109.3, 108.0, 103.1, 55.4; ESI-HRMS Calcd for $C_{32}H_{29}N_2O_4S$ $[M + H]^+$ 537.1843, found 537.1843.



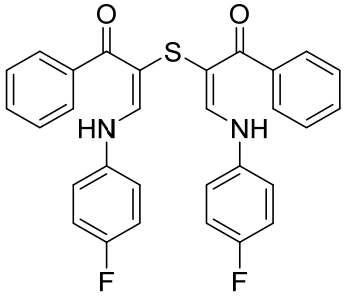
(2E,2'E)-2,2'-Thiobis(3-((3-chlorophenyl)amino)-1-phenylprop-2-en-1-one) (**4n**). Yellow solid; mp 220-225 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.30 (d, J = 13.3 Hz, 2 H), 8.00 (d, J = 13.3 Hz, 2 H), 7.61-7.47 (m, 10 H), 7.25-7.22 (m, 2 H), 4.14-7.04 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.5, 152.6, 141.2, 139.4, 135.5, 131.2, 130.8, 128.8, 128.5, 124.1, 116.8, 115.2, 108.8; ESI-HRMS Calcd for $C_{30}H_{23}Cl_2N_2O_2S$ $[M + H]^+$ 545.0852, found 545.0851.



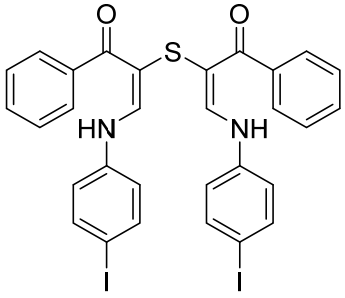
(2E,2'E)-2,2'-Thiobis(3-((3-nitrophenyl)amino)-1-phenylprop-2-en-1-one) (**4o**). Yellow solid; mp 144-145 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.25 (d, J = 11.3 Hz, 2 H), 7.97-7.89 (m, 6 H), 7.56-7.46 (m, 10 H), 7.37 (d, J = 8.0 Hz, 2 H), 6.16 (d, J = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): 191.8, 149.4, 143.4, 141.7, 138.6, 132.1, 130.6, 128.6, 127.5, 122.2, 117.8, 110.0, 95.7; ESI-HRMS Calcd for $C_{30}H_{23}N_4O_6S$ $[M + H]^+$ 567.1333, found 567.1328.



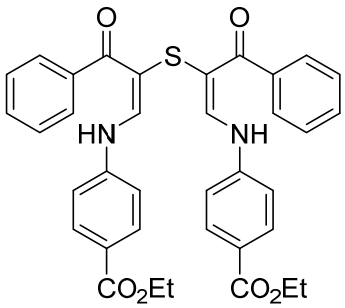
(2E,2'E)-2,2'-Thiobis(3-((4-bromophenyl)amino)-1-phenylprop-2-en-1-one) (**4p**). Yellow solid; mp 247-248 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.28 (d, *J* = 13.5 Hz, 2 H), 7.99 (d, *J* = 13.4 Hz, 2 H), 7.59-7.41 (m, 14 H), 7.05 (d, *J* = 8.5 Hz, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.5, 152.7, 139.5, 139.0, 132.7, 131.1, 128.7, 128.5, 118.3, 116.8, 108.5; ESI-HRMS Calcd for $C_{30}H_{23}Br_2N_2O_2S$ $[M + H]^+$ 632.9842, found 632.9841.



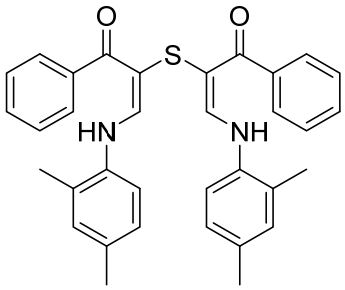
(2E,2'E)-2,2'-Thiobis(3-((4-fluorophenyl)amino)-1-phenylprop-2-en-1-one) (**4q**). Yellow solid; mp 229-233 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.32 (d, *J* = 13.5 Hz, 2 H), 7.98 (d, *J* = 13.5 Hz, 2 H), 7.59-7.45 (m, 10 H), 7.16-7.12 (m, 4 H), 7.02 (t, *J* = 8.6 Hz, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.3, 160.8, 158.4, 153.4, 139.7, 136.2, 130.9, 128.6, 128.5, 118.3, 118.2, 116.6, 116.4, 107.9; IR (KBr, cm^{-1}) 3067, 2976, 2861, 1642, 1600, 1469, 1352, 1272, 1117, 1029, 880, 720; ESI-HRMS Calcd for $C_{32}H_{23}F_2N_2O_2S$ $[M + H]^+$ 513.1443, found 513.1442.



(2E,2'E)-2,2'-Thiobis(3-((4-iodophenyl)amino)-1-phenylprop-2-en-1-one) (**4r**). Yellow solid; mp 241-242 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.26 (d, *J* = 13.3 Hz, 2 H), 7.99 (d, *J* = 13.4 Hz, 2 H), 7.62-7.45 (m, 14 H), 6.94 (d, *J* = 8.6 Hz, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.5, 152.5, 139.7, 139.5, 138.6, 131.1, 128.7, 128.5, 118.7, 108.6, 87.1; ESI-HRMS Calcd for $C_{30}H_{23}I_2N_2O_2S$ $[M + H]^+$ 728.9564, found 728.9564.

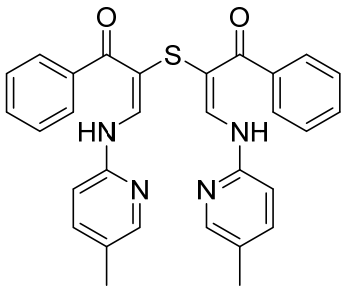


Diethyl 4,4'-(((1E,1'E)-thiobis(3-oxo-3-phenylprop-1-ene-2,1-diyl))bis(azanediyl)) dibenzoate (**4s**). Yellow solid; mp 219-222 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.39 (d, *J* = 13.3 Hz, 2 H), 8.10 (d, *J* = 13.3 Hz, 2 H), 8.01 (d, *J* = 8.7 Hz, 4 H), 7.62-7.47 (m, 10 H), 7.20 (d, *J* = 8.8 Hz, 4 H), 4.35 (q, *J* = 7.1 Hz, 4 H), 1.38 (t, *J* = 7.1 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.7, 165.9, 152.1, 143.6, 139.3, 131.5, 131.3, 128.8, 128.6, 125.9, 116.0, 109.5, 60.9, 14.3; ESI-HRMS Calcd for $C_{36}H_{33}N_2O_6S$ $[M + H]^+$ 621.2054, found 621.2054.

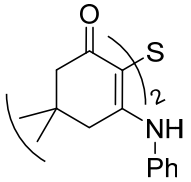


(2E,2'E)-2,2'-Thiobis(3-((2,4-dimethylphenyl)amino)-1-phenylprop-2-en-1-one) (**4t**). Yellow solid; mp 198-202 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.56 (d, *J* = 13.3 Hz, 2 H), 7.93 (d, *J* = 13.4 Hz, 2 H), 7.51-7.36 (m, 10 H), 7.02 (s, 2 H), 6.91 (d, *J* = 7.8 Hz, 2 H), 6.75 (d, *J* = 8.0 Hz, 2 H), 2.59 (s, 6 H), 2.27 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 194.8, 156.2, 140.4, 136.8, 134.7, 132.1, 130.3, 129.6, 128.5, 128.3, 127.4, 118.5, 107.2, 20.7, 18.1; IR (KBr, cm^{-1}) 3123, 3037, 2957, 2856, 2360, 1619,

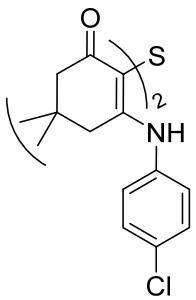
1592, 1461, 1384, 1269, 1112, 1024, 861, 723; ESI-HRMS Calcd for $C_{34}H_{33}N_2O_2S$ $[M + H]^+$ 533.2257, found 533.2257.



(2E,2'E)-2,2'-Thiobis(3-((4-methylpyridin-2-yl)amino)-1-phenylprop-2-en-1-one) (**4u**). Yellow solid; mp 250-254 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.40 (d, *J* = 12.6 Hz, 2 H), 8.83 (d, *J* = 12.7 Hz, 2 H), 8.02 (d, *J* = 5.1 Hz, 2 H), 7.62 (d, *J* = 6.9 Hz, 4 H), 7.54-7.44 (m, 6 H), 7.15 (s, 2 H), 6.77 (d, *J* = 4.9 Hz, 2 H), 2.37 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 196.3, 152.0, 151.5, 150.0, 147.9, 139.7, 130.9, 128.9, 128.4, 120.4, 113.3, 108.9, 21.1; IR (KBr, cm^{-1}) 3275, 3058, 2984, 2850, 1907, 1632, 1552, 1472, 1381, 1261, 1112, 1029, 819, 728; ESI-HRMS Calcd for $C_{30}H_{27}N_4O_2S$ $[M + H]^+$ 507.1849, found 507.1849.

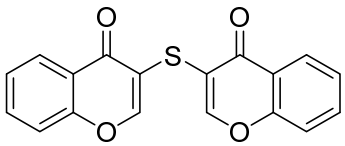


2,2'-Thiobis(5,5-dimethyl-3-(phenylamino)cyclohex-2-en-1-one) (**6a**).[3] Yellow solid; mp 198-204 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.65 (s, 2 H), 7.41 (t, *J* = 7.7 Hz, 4 H), 7.29 (s, 1 H), 7.24-7.22 (m, 5 H), 2.44 (s, 4 H), 2.37 (s, 4 H), 1.05 (s, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): 195.6, 167.7, 138.0, 129.4, 126.3, 124.7, 103.5, 49.5, 40.8, 32.6.

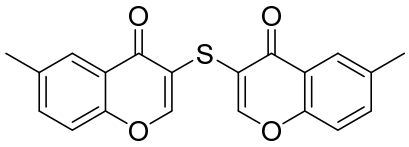


2,2'-Thiobis(3-((4-chlorophenyl)amino)-5,5-dimethylcyclohex-2-en-1-one) (**6b**).[3]

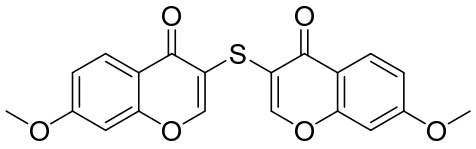
Yellow solid; mp 278-280 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.18 (s, 2 H), 7.35 (d, *J* = 8.6 Hz, 4 H), 7.20 (d, *J* = 8.6 Hz, 4 H), 2.58 (s, 4 H), 2.38 (s, 4 H), 0.98 (s, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): 194.8, 167.0, 137.3, 131.2, 129.4, 125.7, 104.8, 50.5, 41.3, 32.7; IR (KBr, cm^{-1}) 3064, 2957, 2938, 1600, 1590, 1492, 1382, 1277, 1120, 819, 736.



3,3'-Thiobis(4H-chromen-4-one) (**8a**)[4] White solid; mp 198-199 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (s, 2 H), 8.17 (d, *J* = 8.0 Hz, 2 H), 7.66 (t, *J* = 8.0 Hz, 2 H), 7.46-7.38 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): 175.6, 159.6, 156.3, 133.9, 126.1, 125.7, 123.9, 118.3, 116.5; IR (KBr, cm^{-1}) 3059, 1824, 1629, 1469, 1349, 1309, 1219, 1171, 1016, 888, 760.



3,3'-Thiobis(6-methyl-4H-chromen-4-one) (**8b**).[4] White solid; mp 224-225 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (s, 2 H), 7.95 (s, 2 H), 7.45 (d, *J* = 8.4 Hz, 2 H), 7.33 (d, *J* = 8.4 Hz, 2 H), 2.42 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 175.7, 159.5, 154.6, 135.8, 135.1, 125.3, 123.6, 118.0, 116.3, 20.9.



3,3'-Thiobis(7-methoxy-4H-chromen-4-one) (**8c**). White solid; mp 237-239 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (s, 2 H), 8.07 (d, *J* = 8.9 Hz, 2 H), 6.95 (d, *J* = 7.5 Hz, 2 H), 6.81 (s, 2 H), 3.88 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 175.0, 164.2, 159.2, 158.1, 127.4, 117.8, 116.5, 115.1, 100.4, 55.9; IR (KBr, cm^{-1}) 3074, 1850, 1618, 1440, 1360, 1307, 1227, 1187, 1019, 941, 776; ESI-HRMS Calcd for $C_{20}H_{15}O_6S$ $[M + H]^+$ 383.0584, found 383.0582.

References

(1) F. M. A. A. EL-Taweel and M. H. Elnagdi, *J. Heterocyclic Chem.,* 2001, **38**, 981.

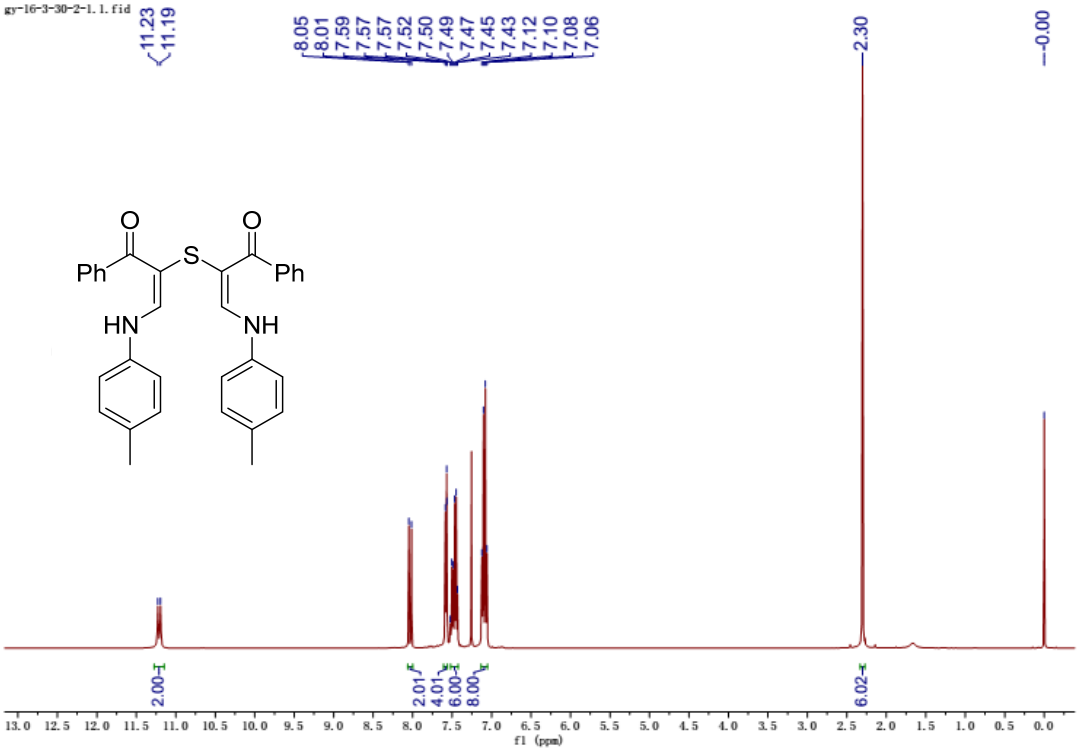
(2) G. W. Wang and C. B. Miao, *Green Chem*., 2006, **8**, 1080.

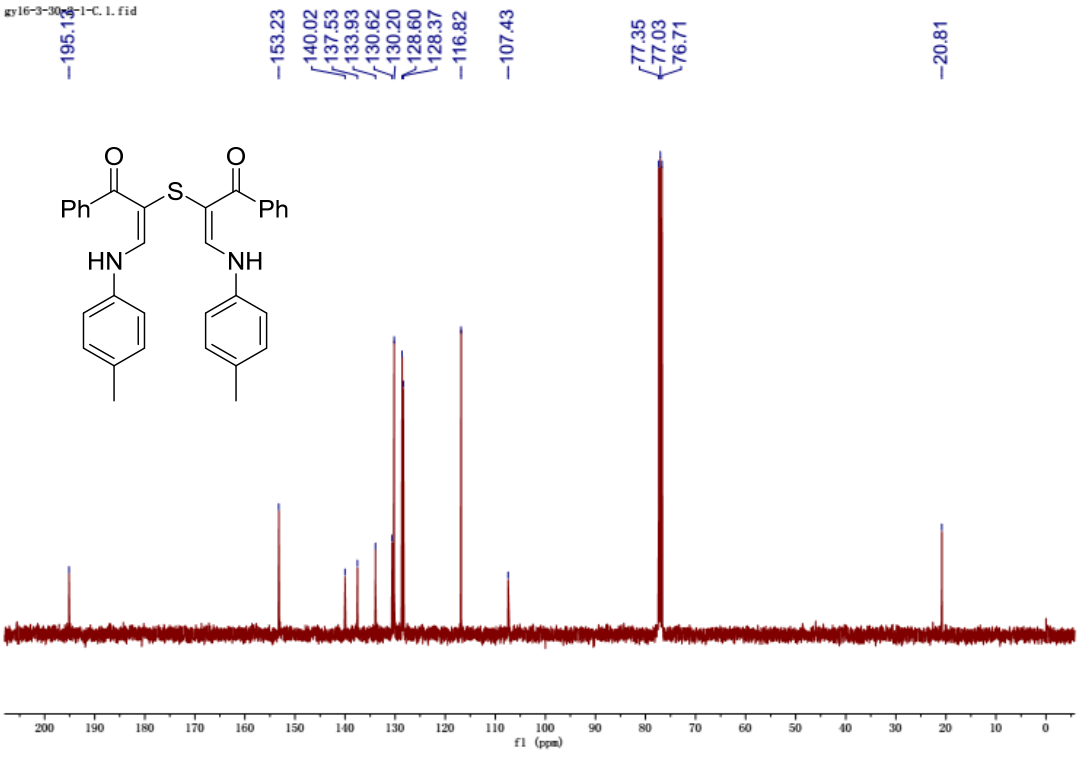
(3) L. F. Yang, C. G. Liu, X. P. Xu and S. J. Ji, *Org. Biomol. Chem*., **2016**, **14**, 2993.

(4) W. Loewe and A. Kennemann, *Archiv der Pharmazie,* 1984, **317**, 4372.

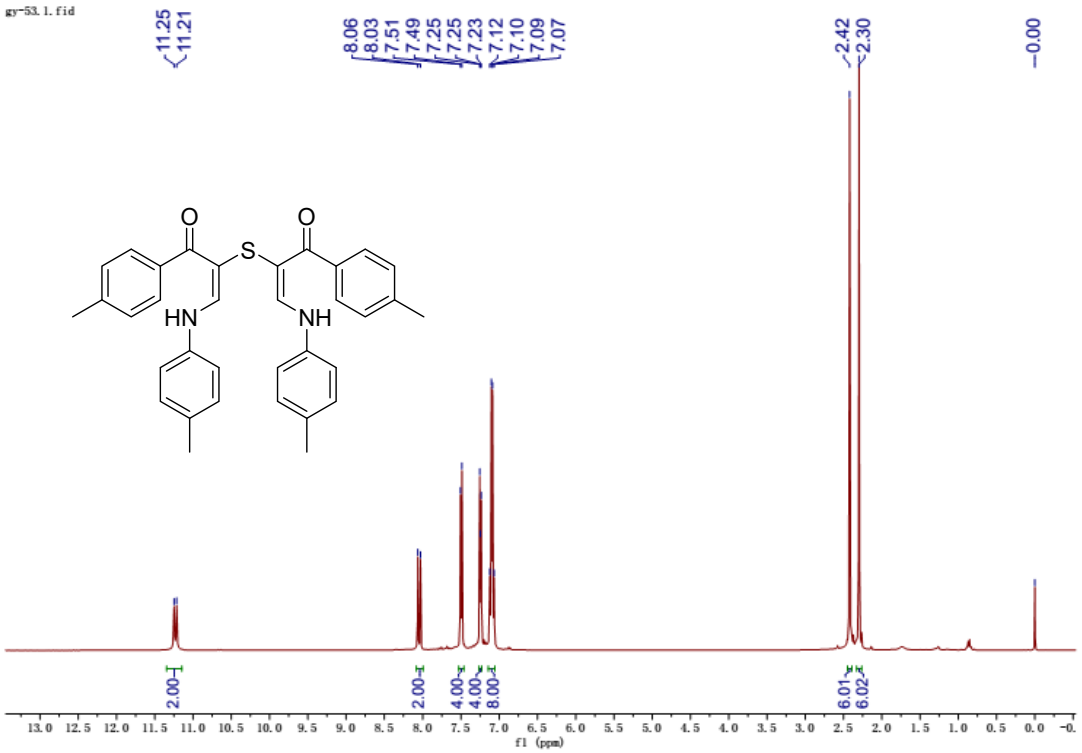
^{1}H and ^{13}C NMR spectra of all products

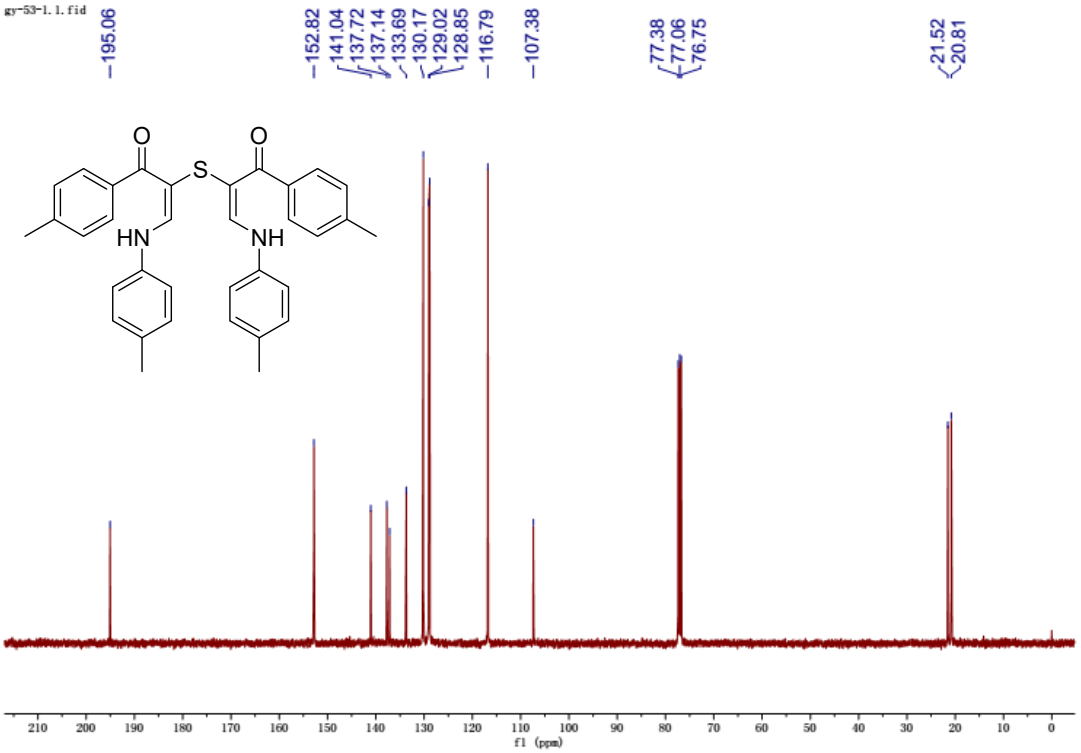
^{1}H and ^{13}C NMR spectra of **4a**



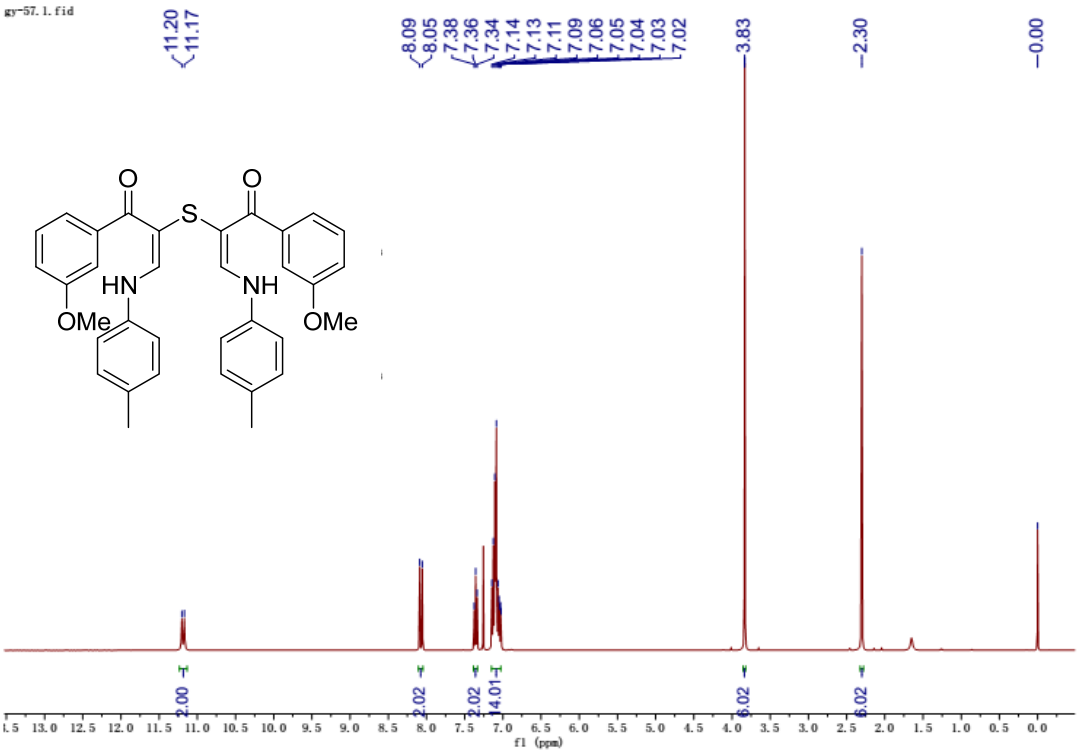


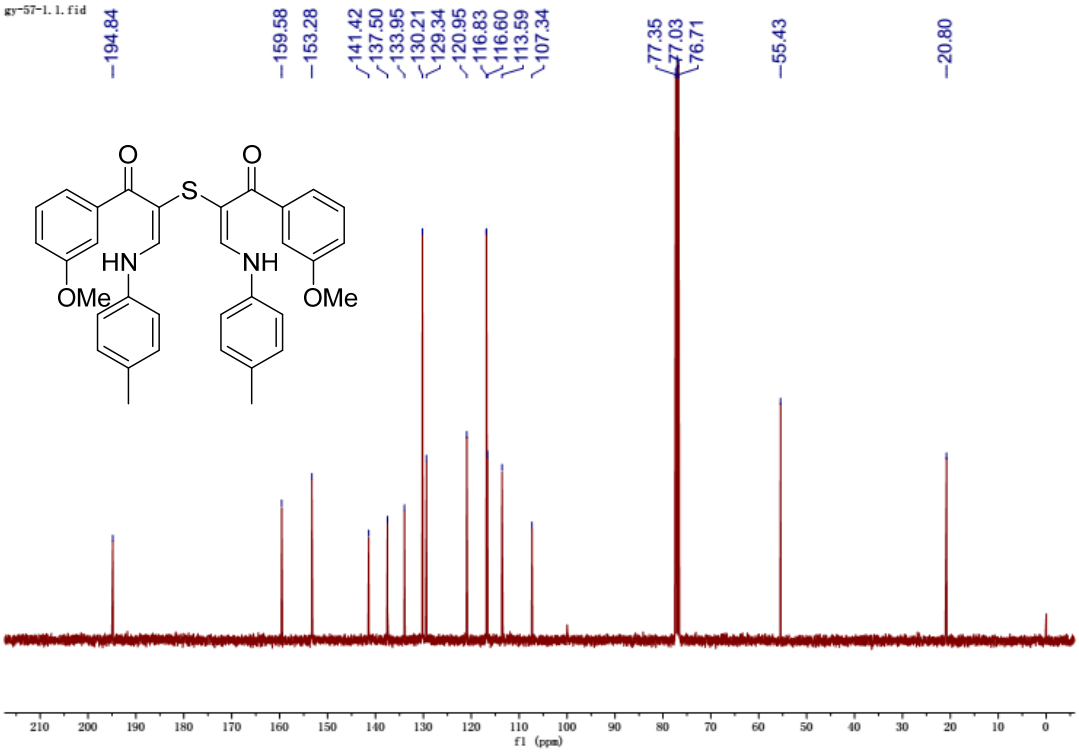
^{1}H and ^{13}C NMR spectra of **4b**



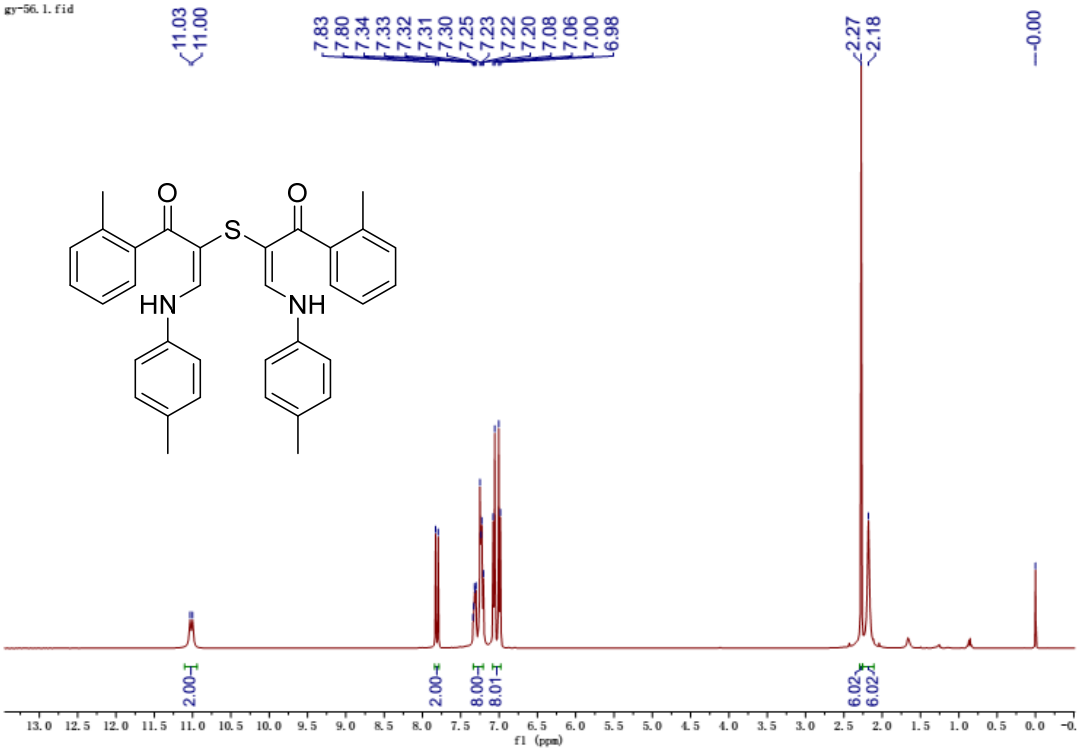


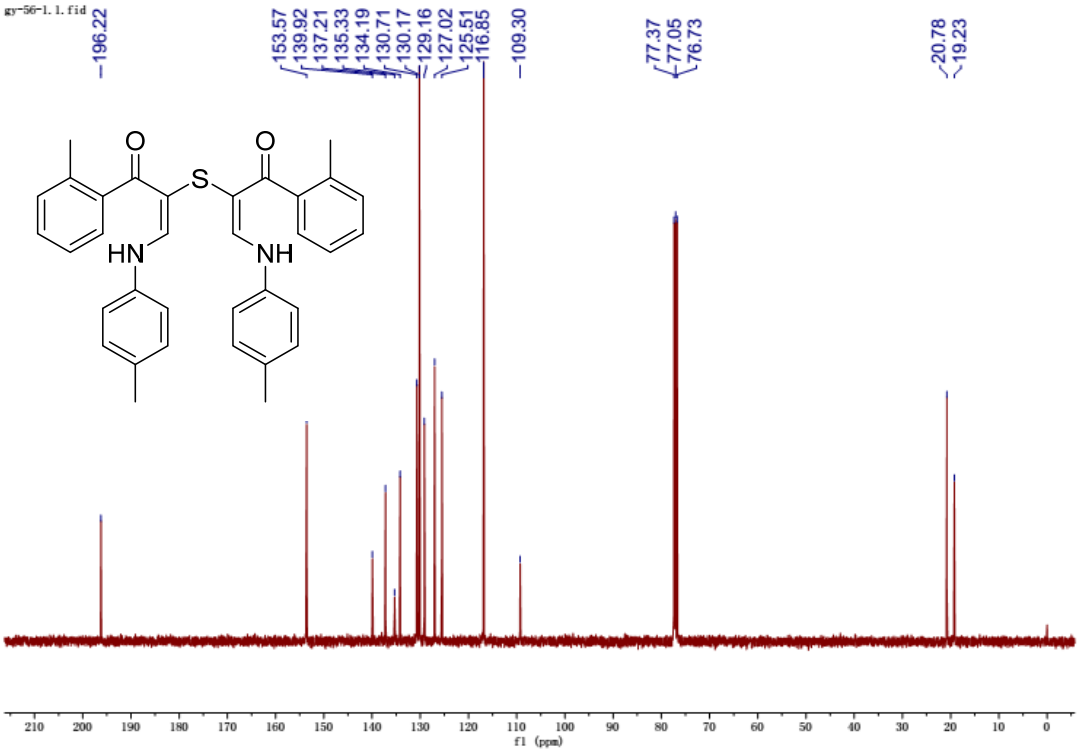
^{1}H and ^{13}C NMR spectra of **4c**



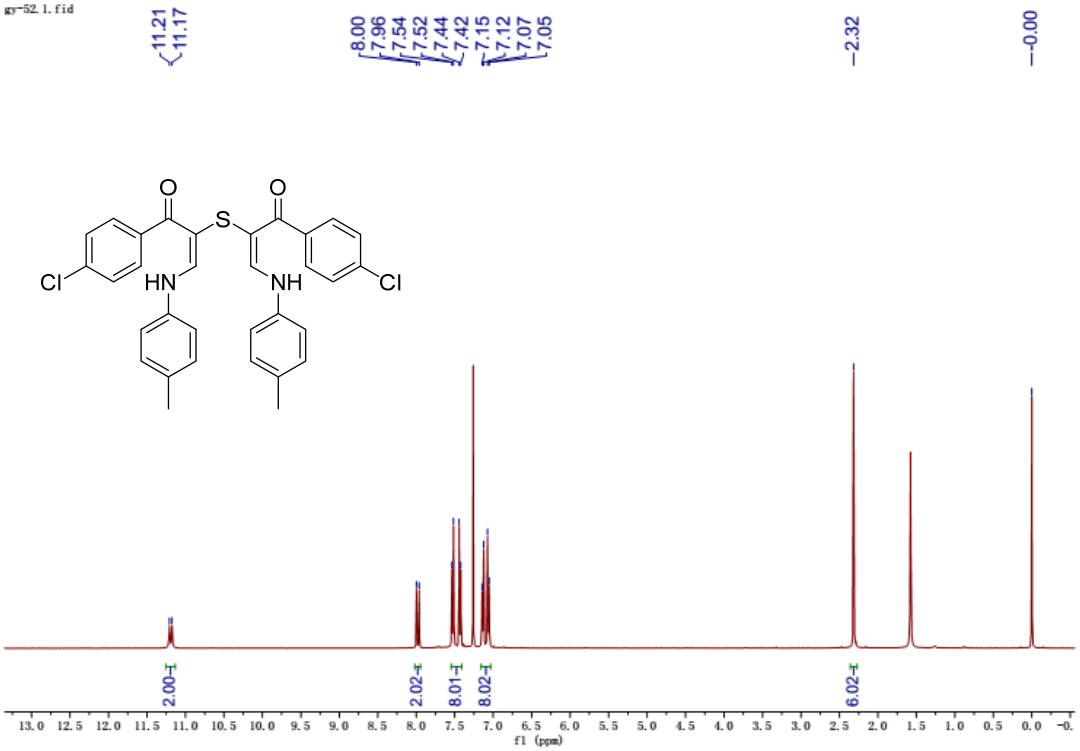


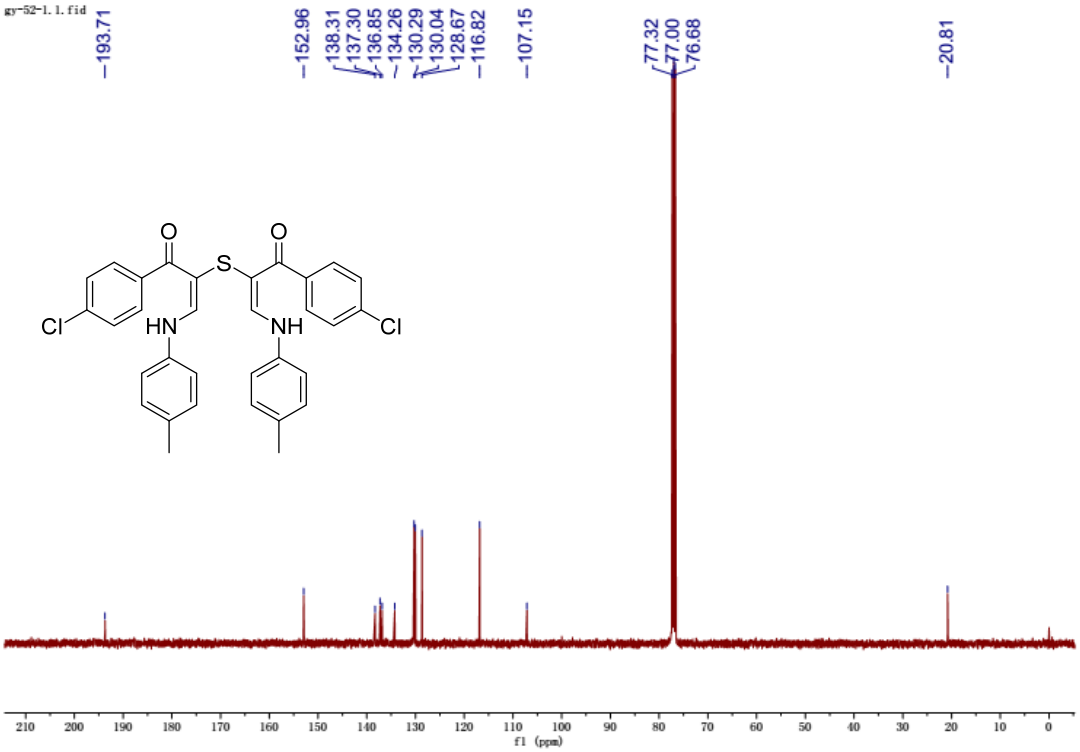
^{1}H and ^{13}C NMR spectra of **4d**



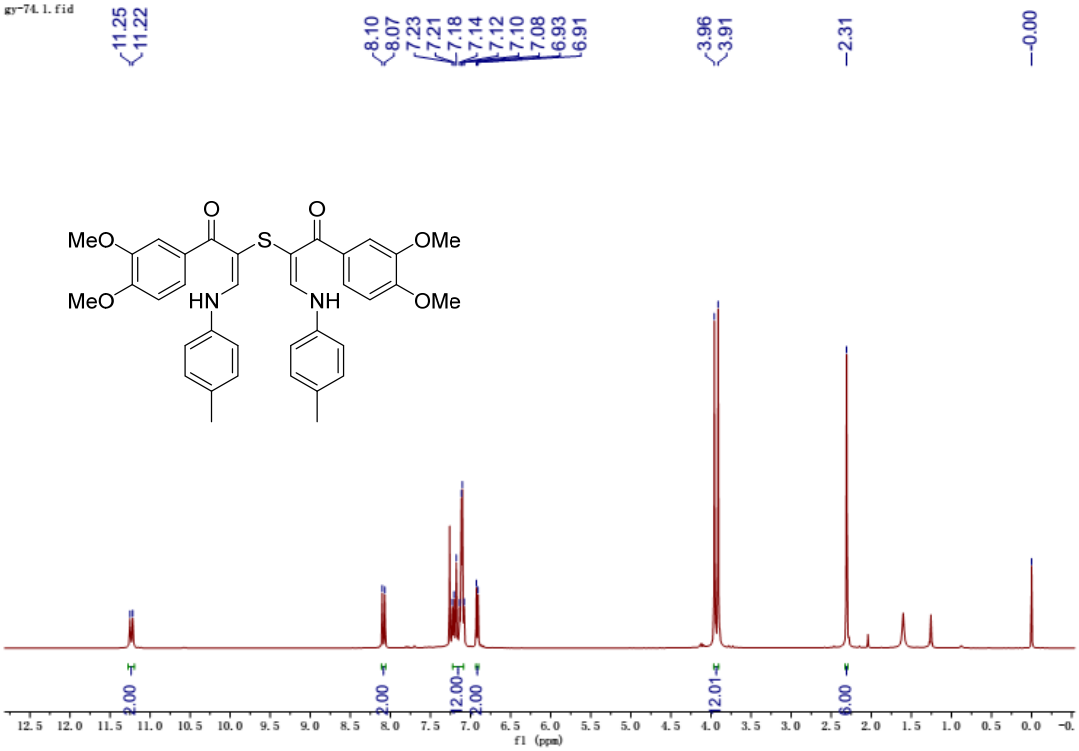


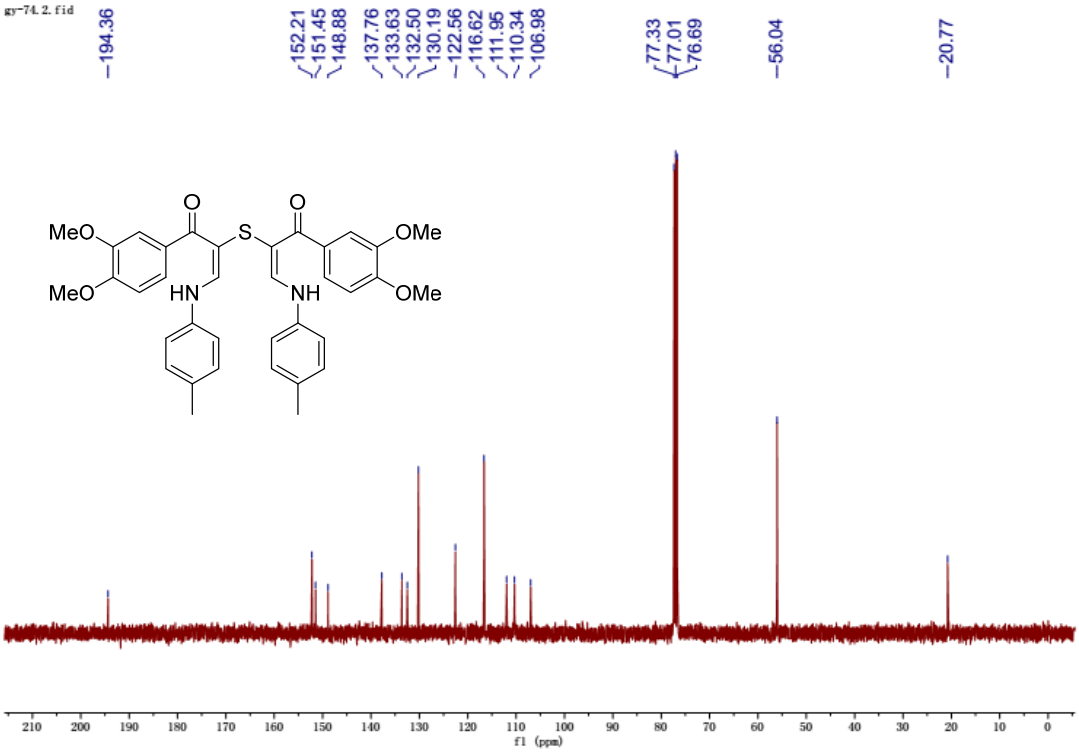
^{1}H and ^{13}C NMR spectra of **4e**



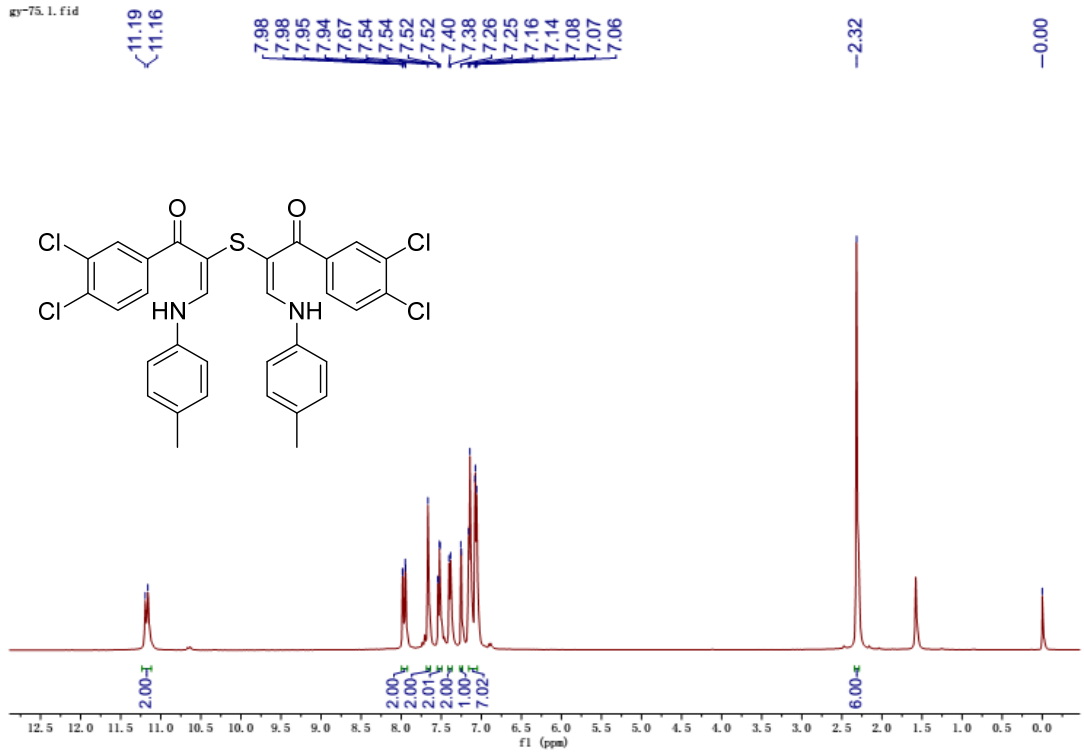


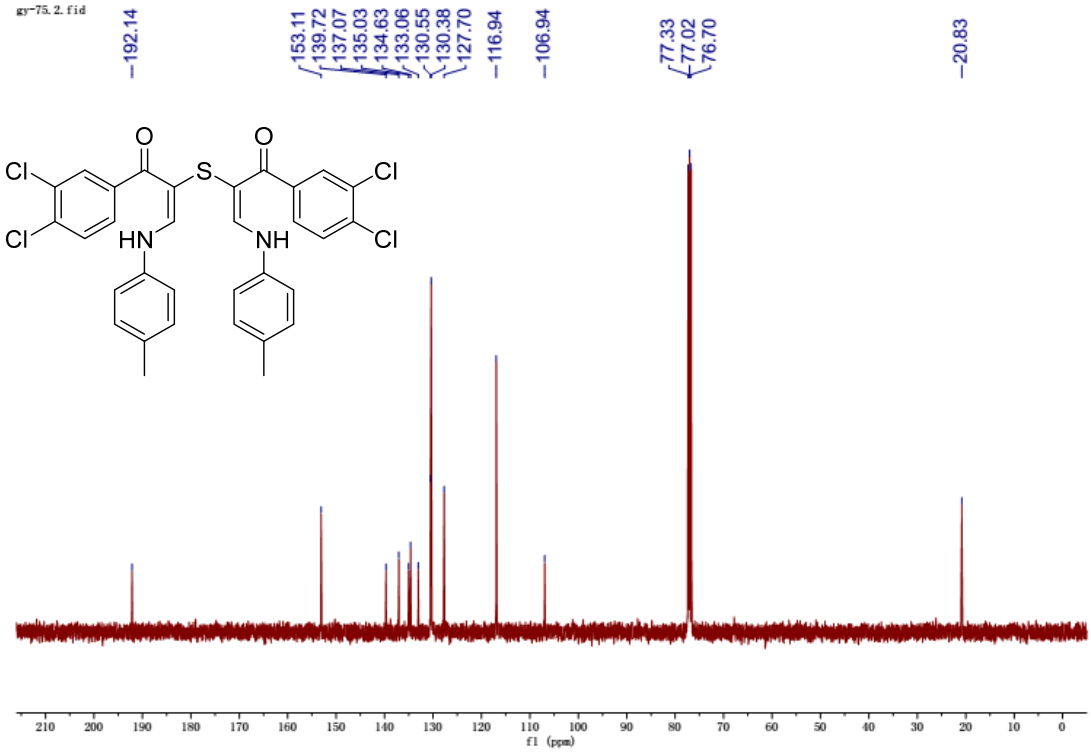
^{1}H and ^{13}C NMR spectra of **4f**



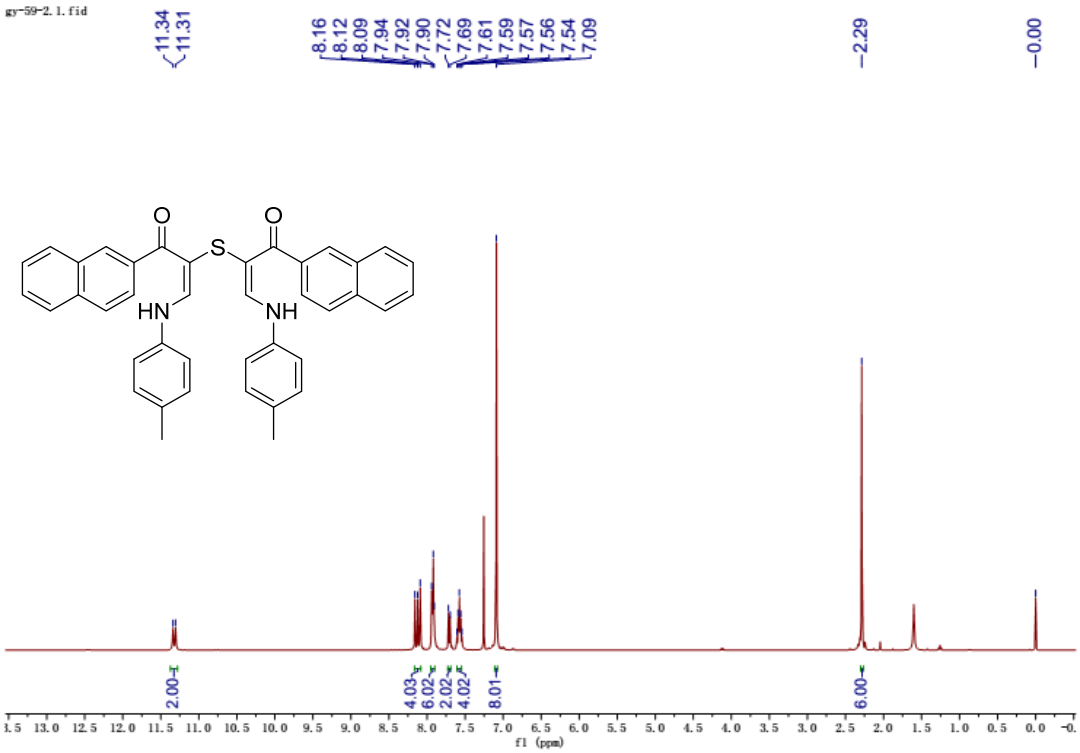


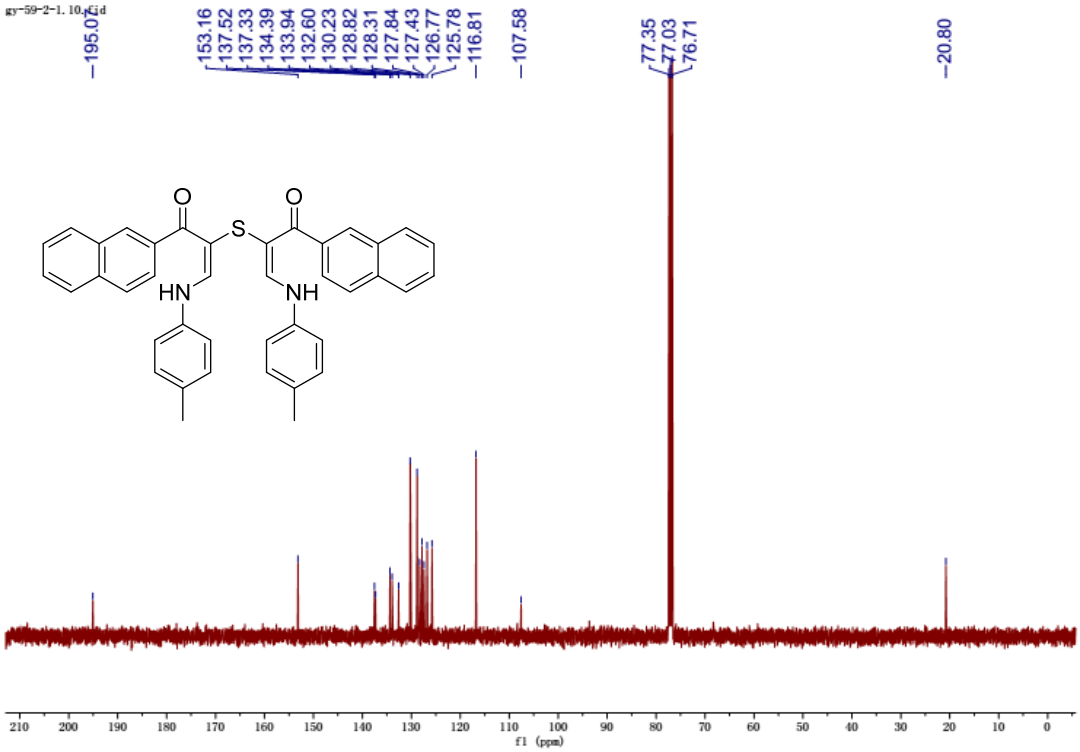
^{1}H and ^{13}C NMR spectra of **4g**



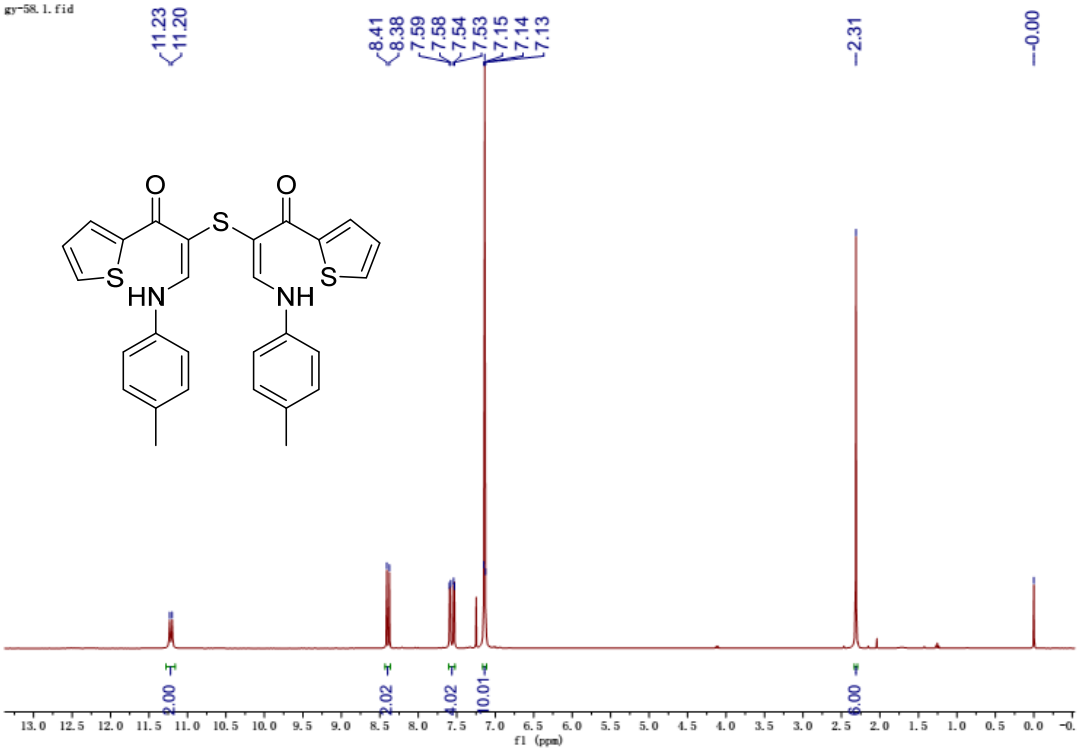


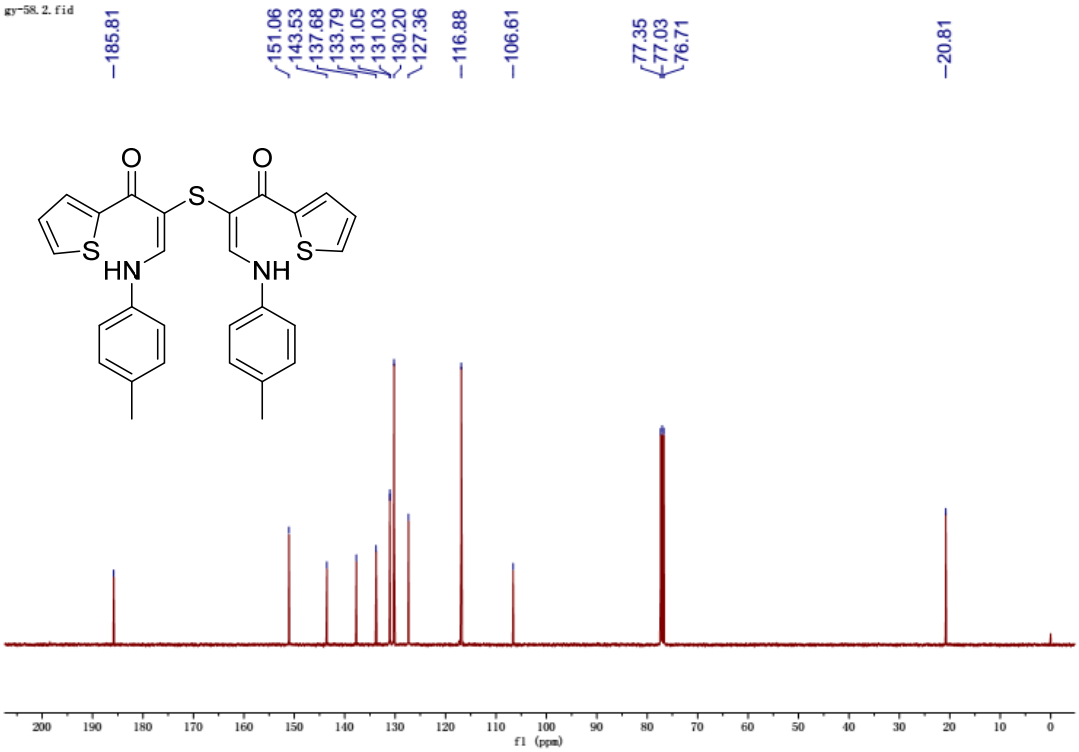
^{1}H and ^{13}C NMR spectra of **4h**



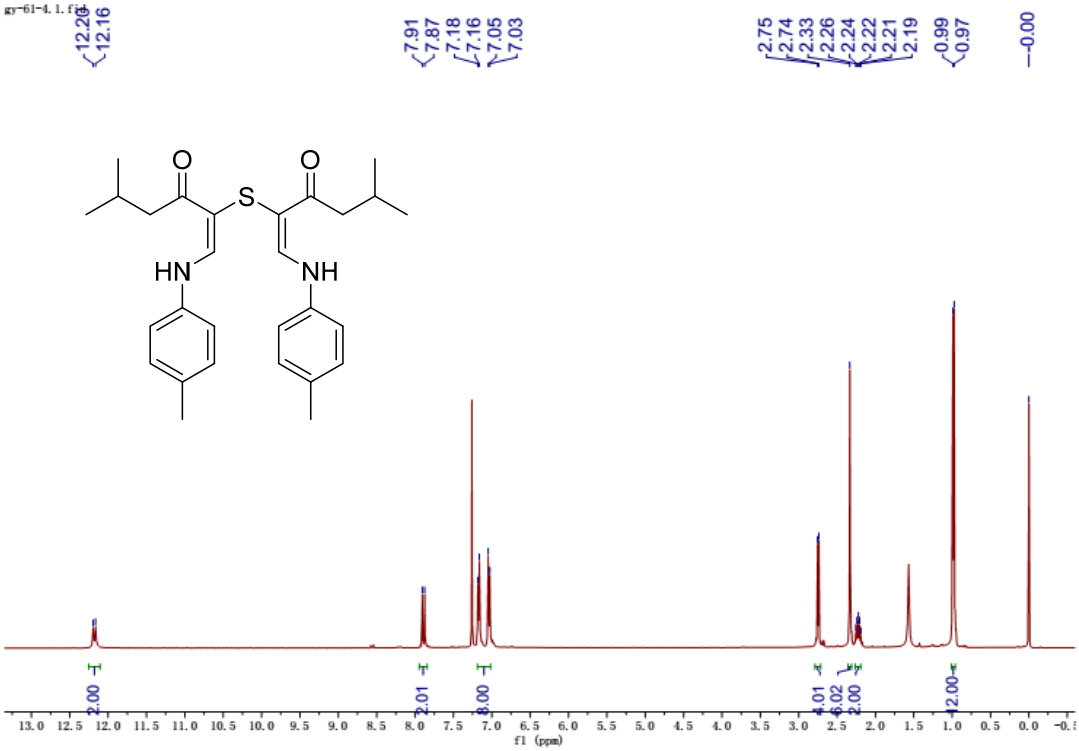


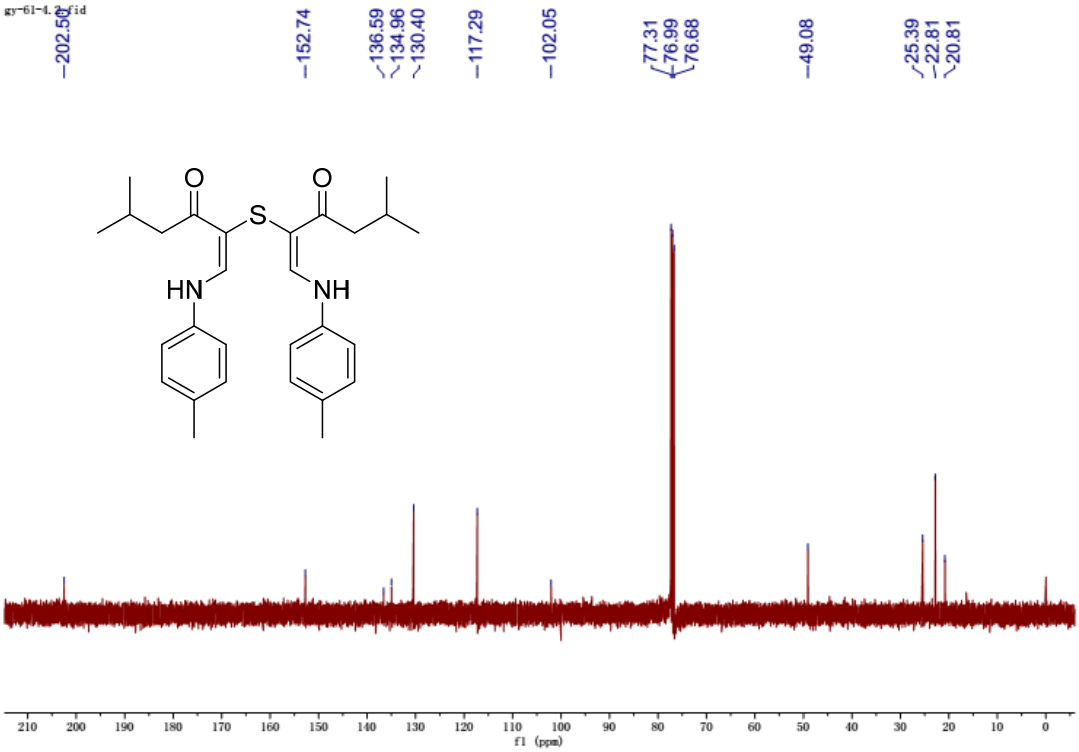
^{1}H and ^{13}C NMR spectra of **4i**



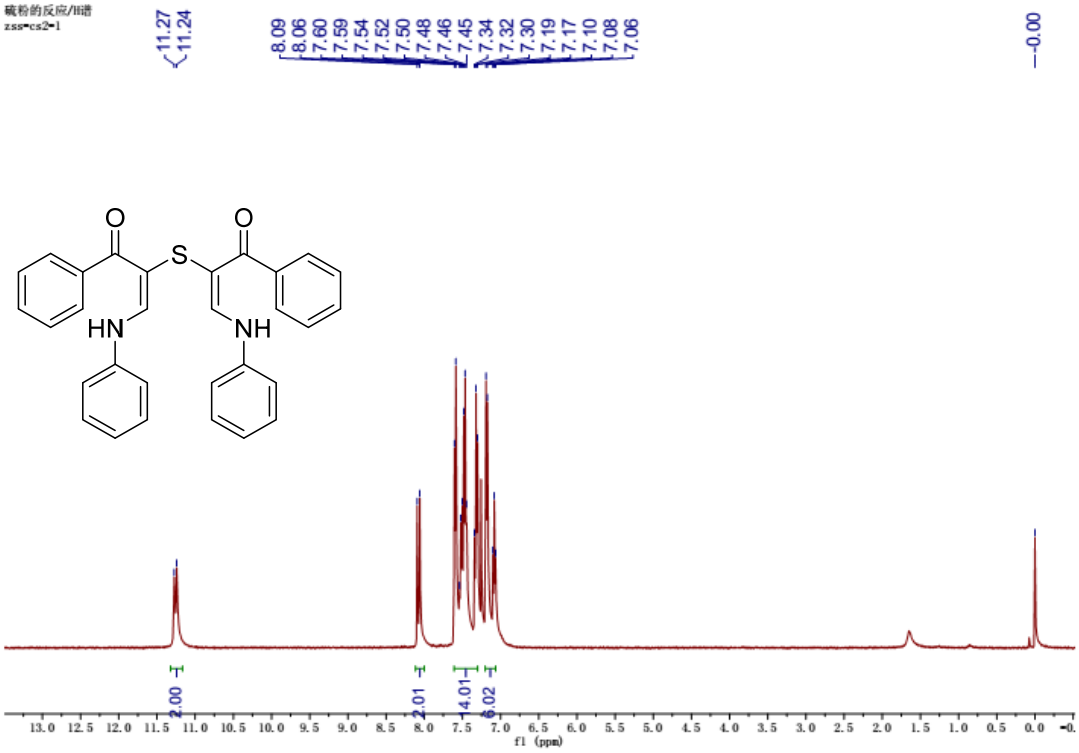


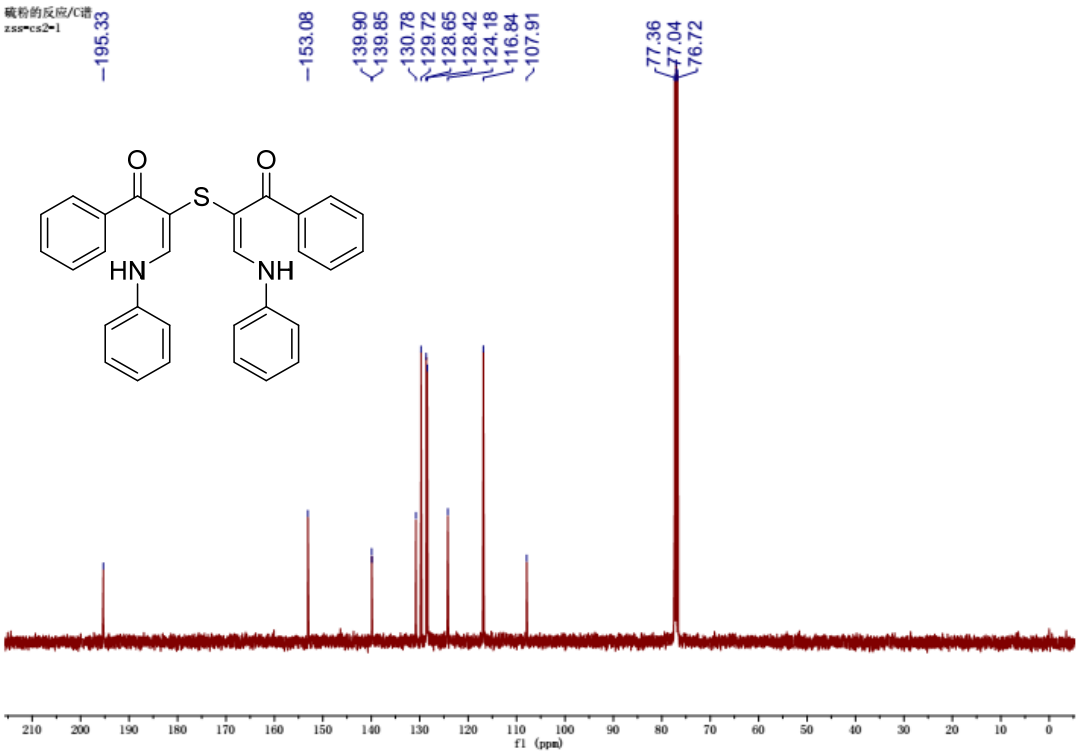
^{1}H and ^{13}C NMR spectra of **4j**



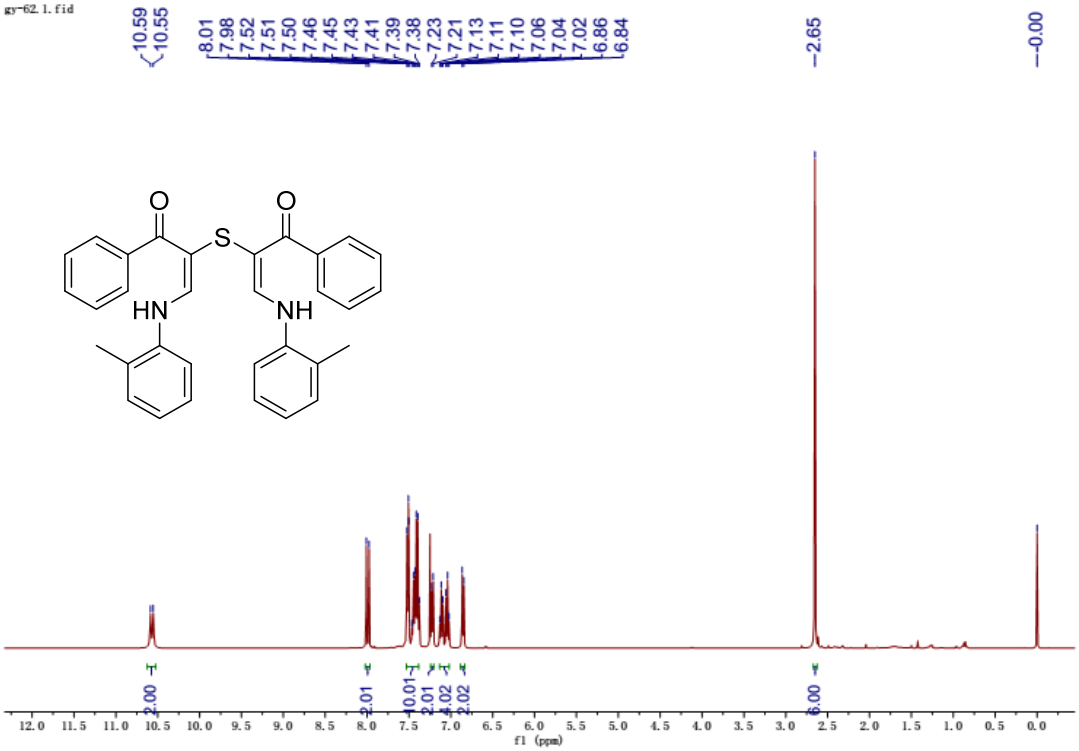


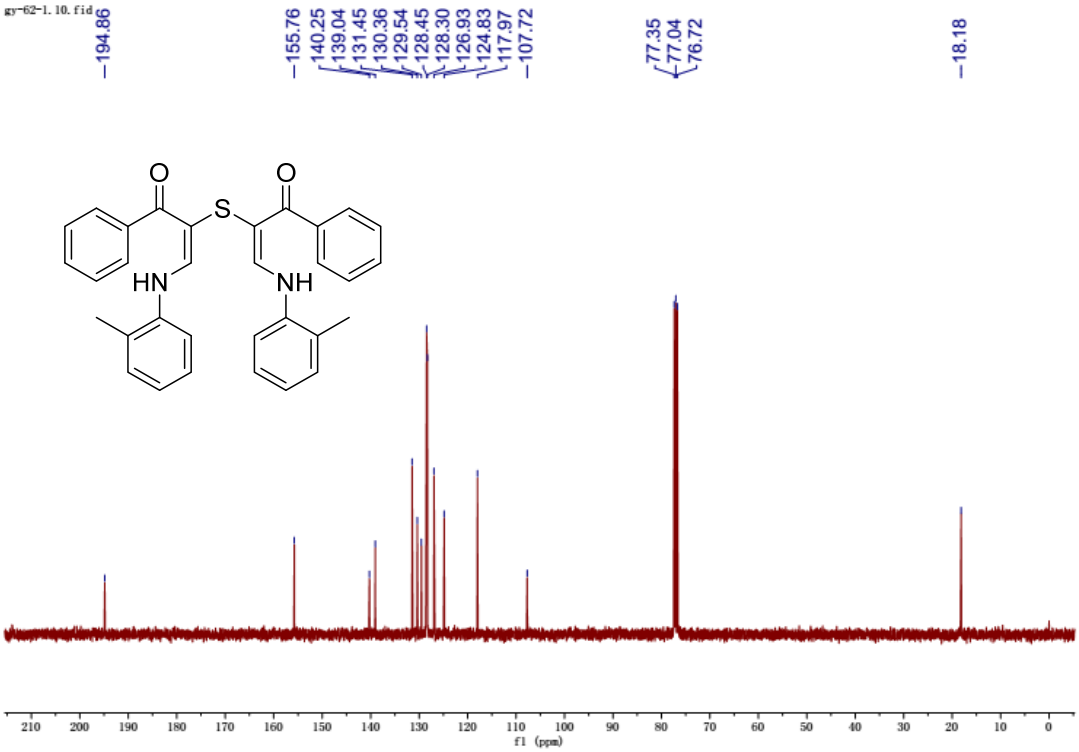
^{1}H and ^{13}C NMR spectra of **4k**



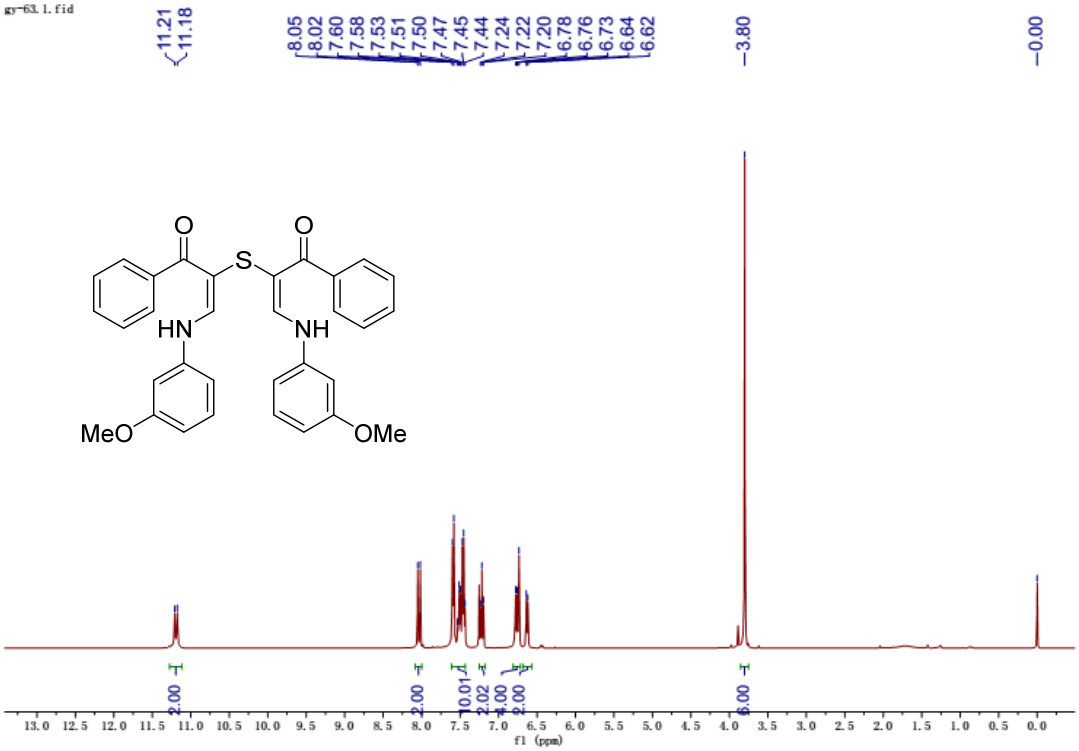


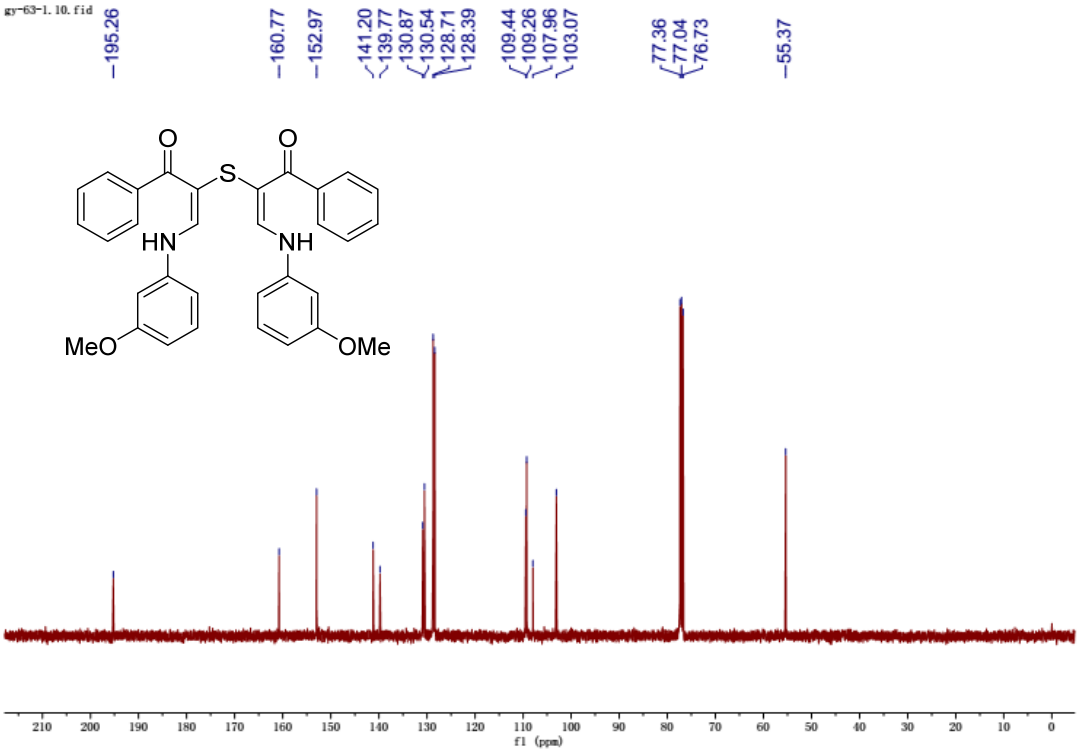
^{1}H and ^{13}C NMR spectra of **4l**



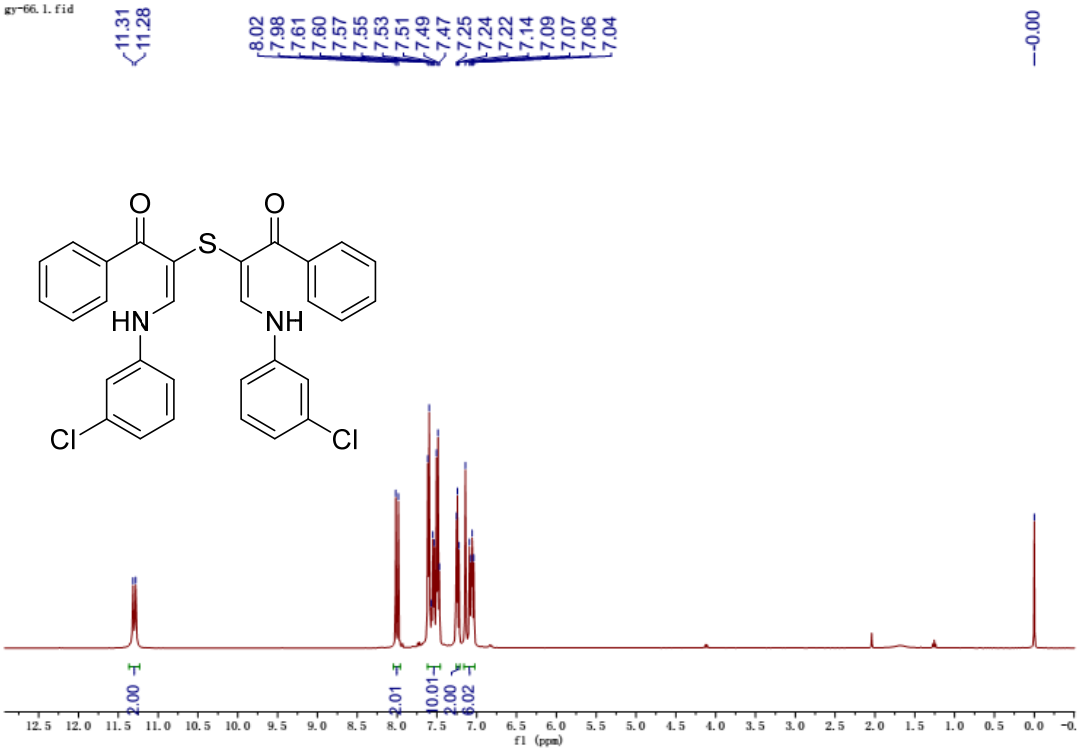


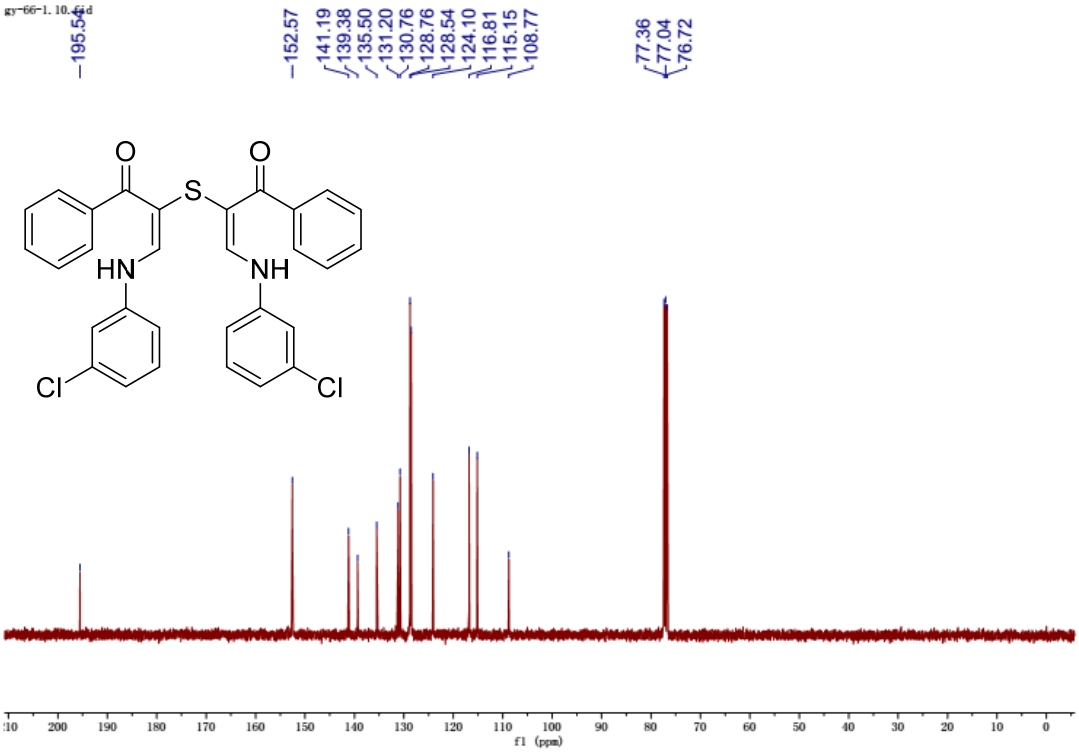
^{1}H and ^{13}C NMR spectra of **4m**



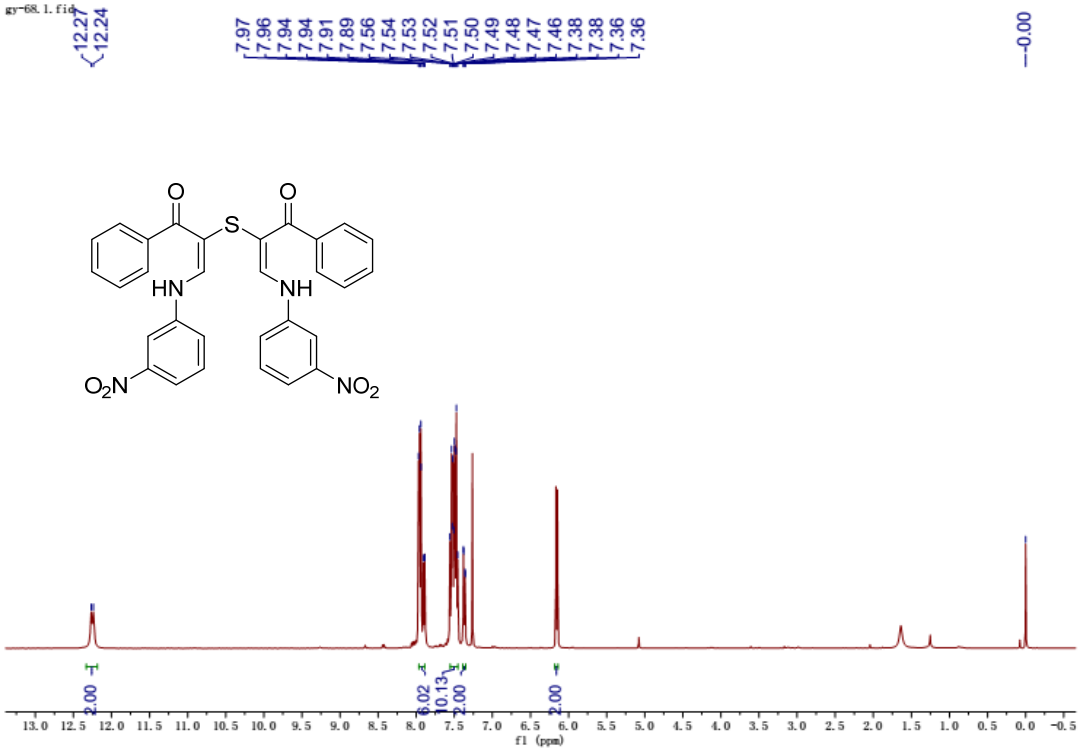


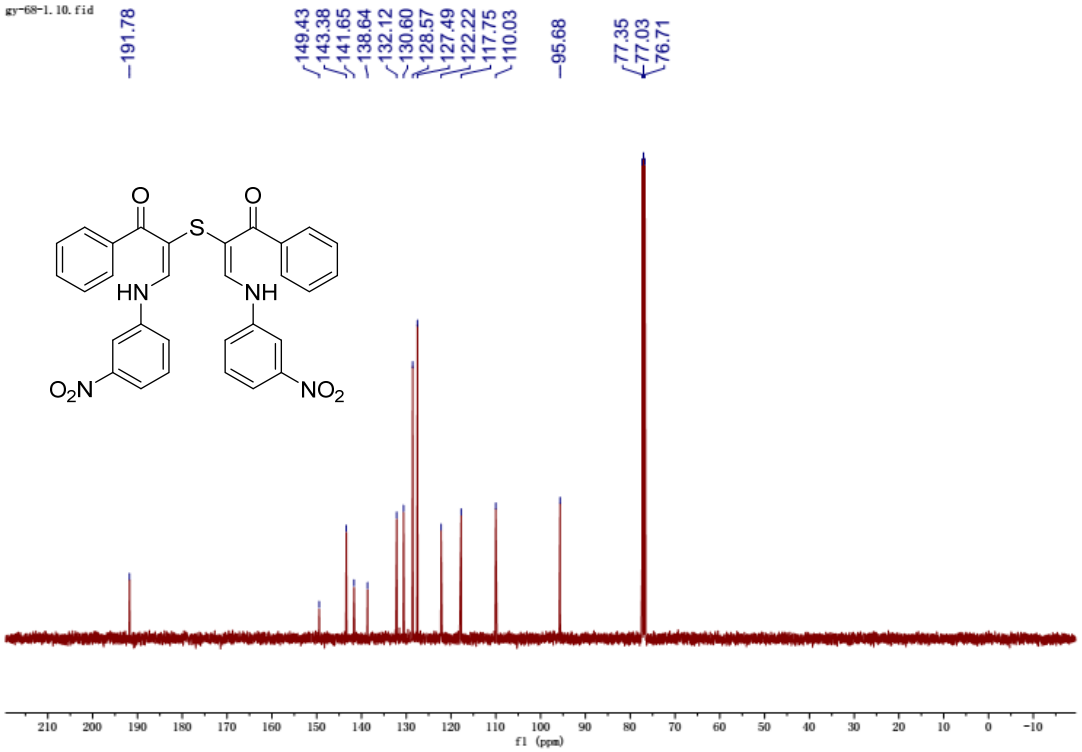
^{1}H and ^{13}C NMR spectra of **4n**



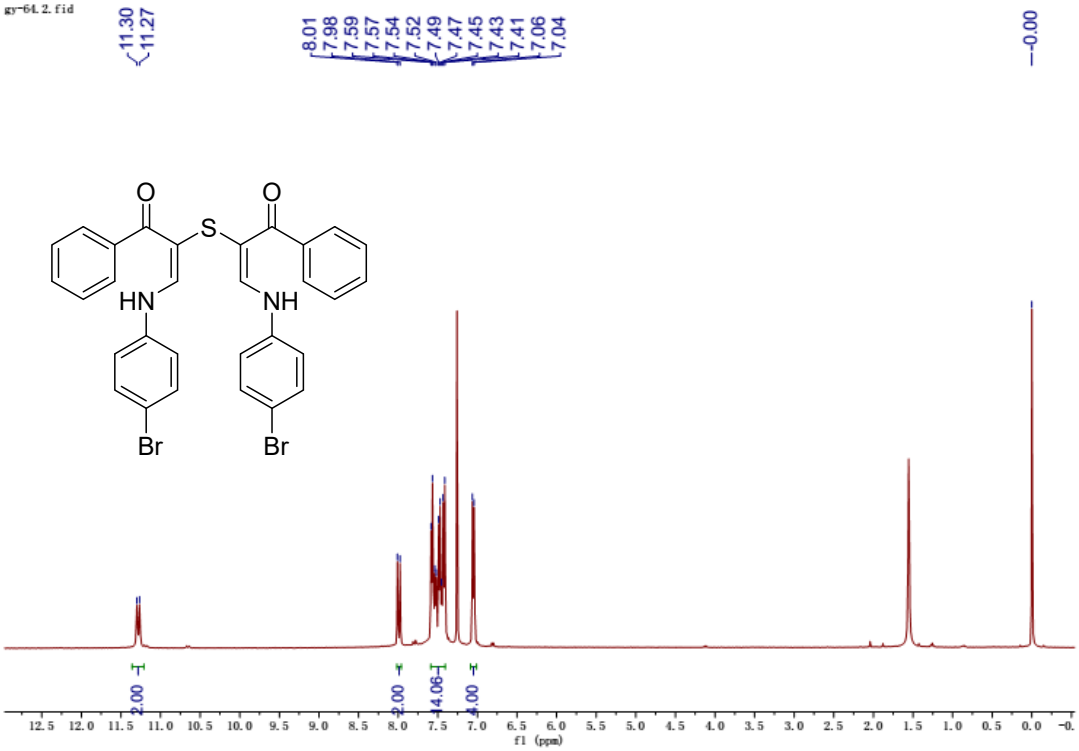


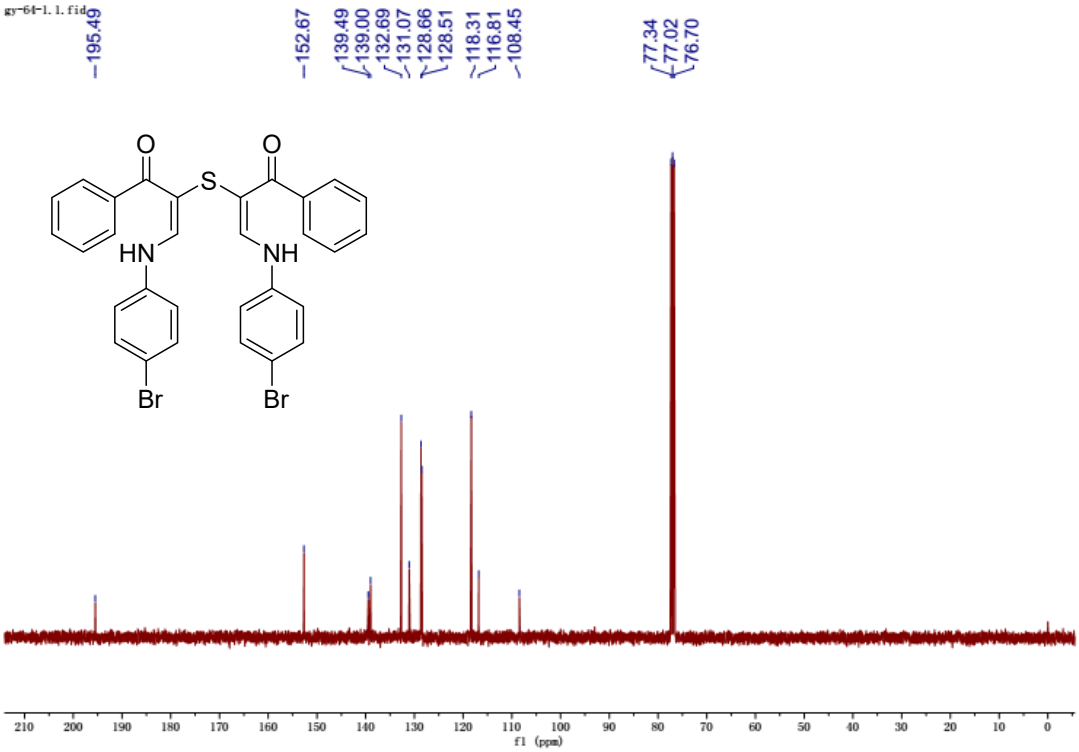
^{1}H and ^{13}C NMR spectra of **4o**



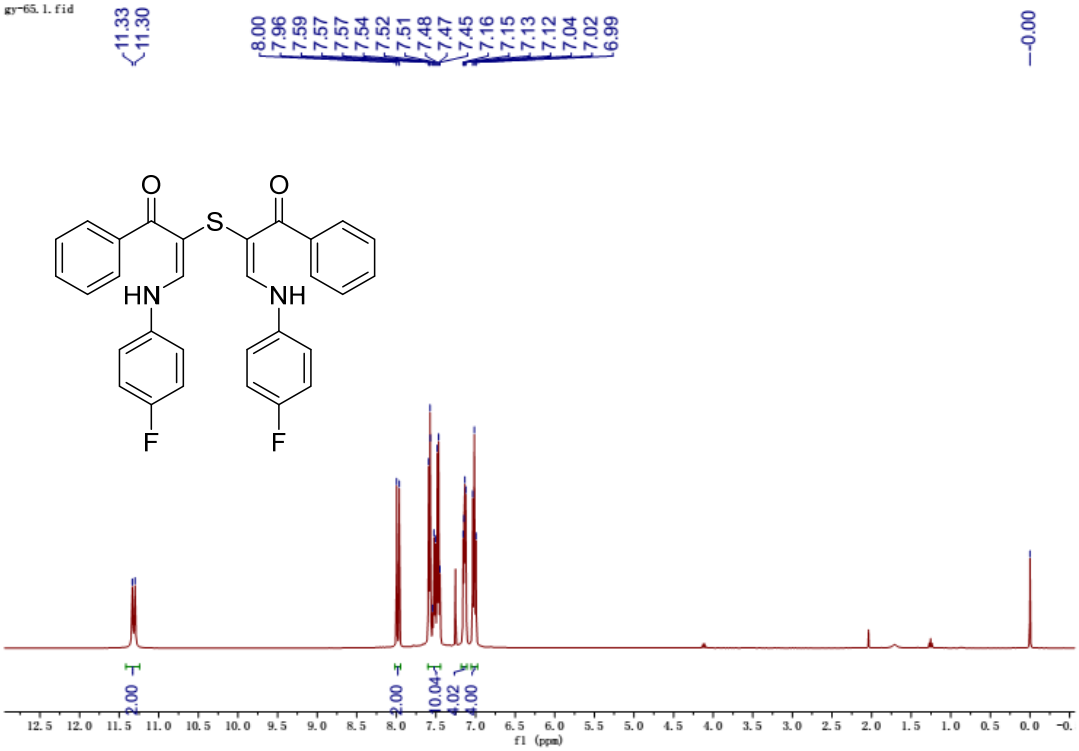


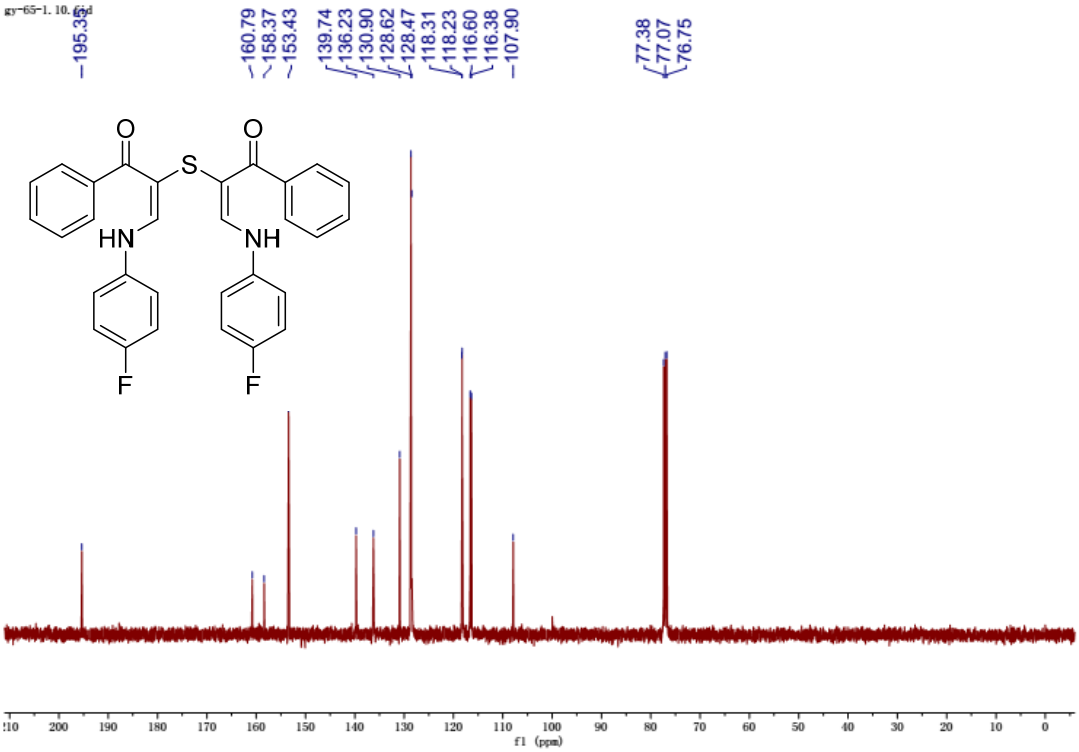
^{1}H and ^{13}C NMR spectra of **4p**



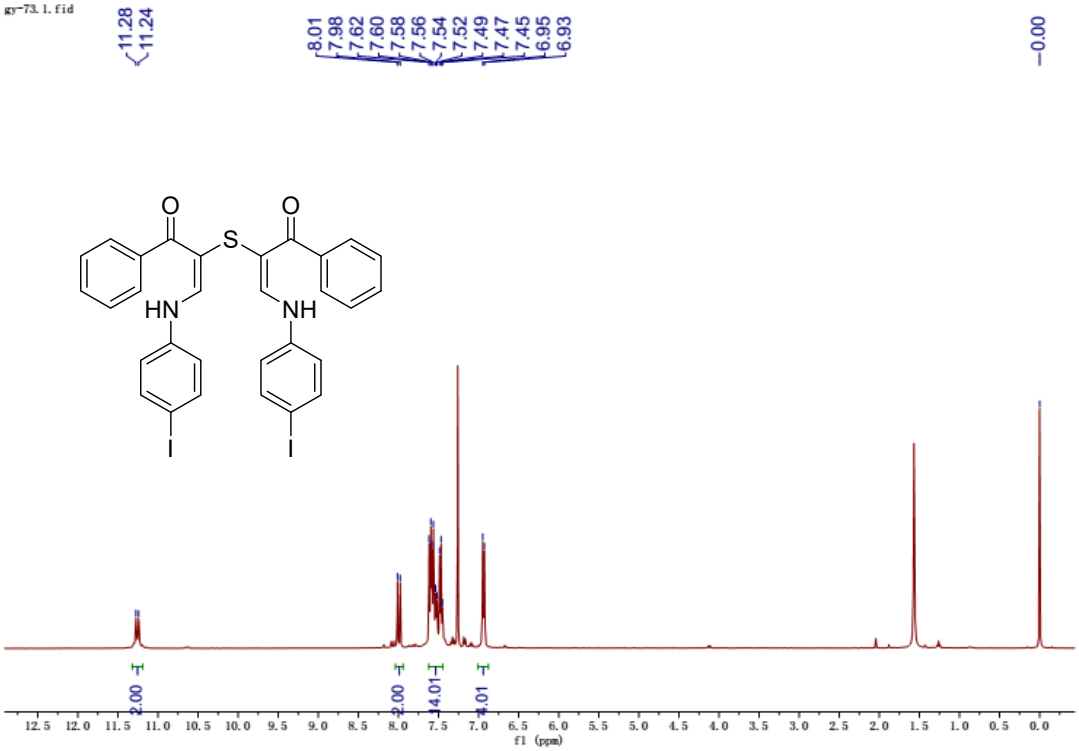


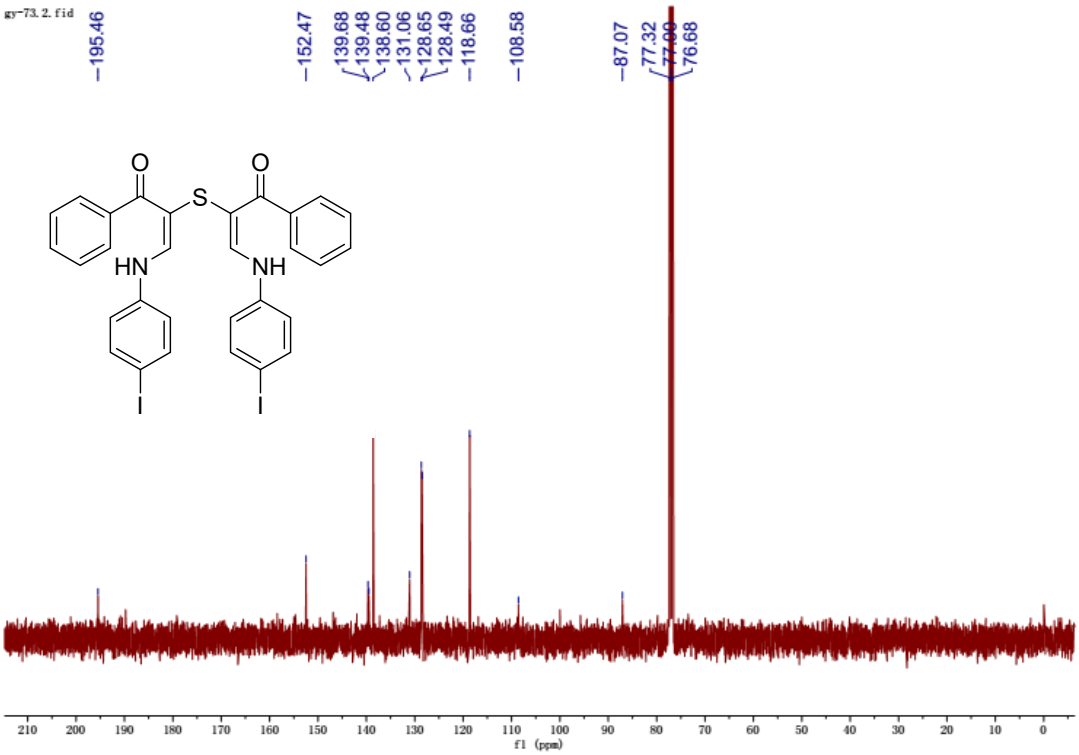
^{1}H and ^{13}C NMR spectra of **4q**



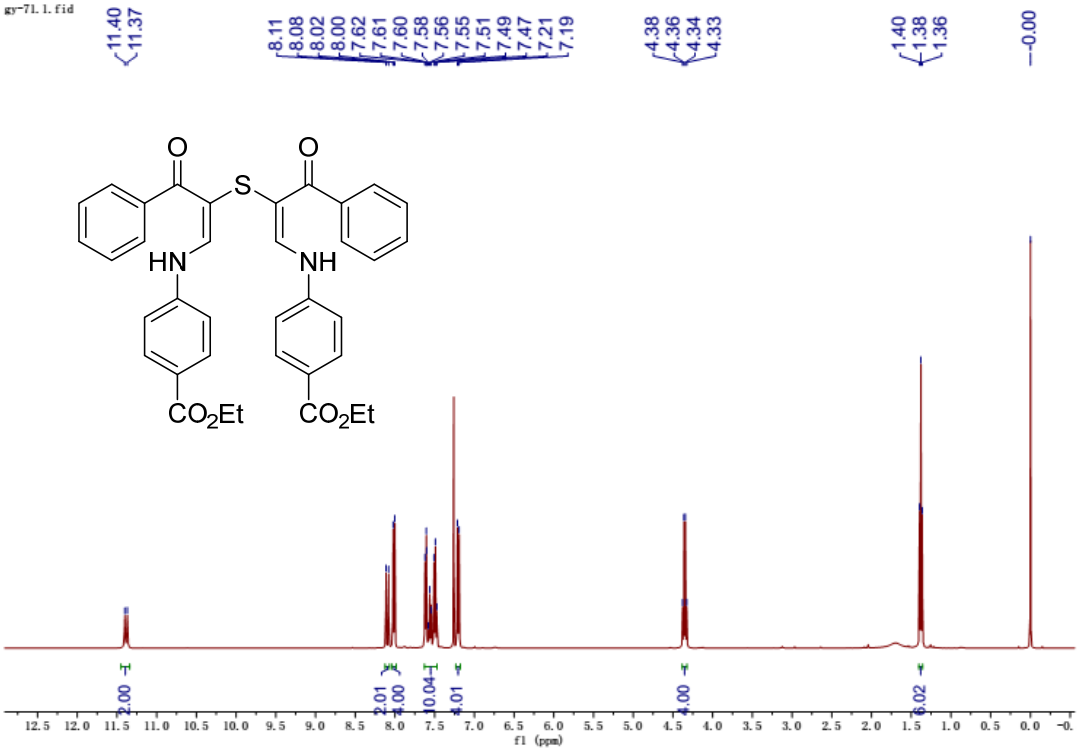


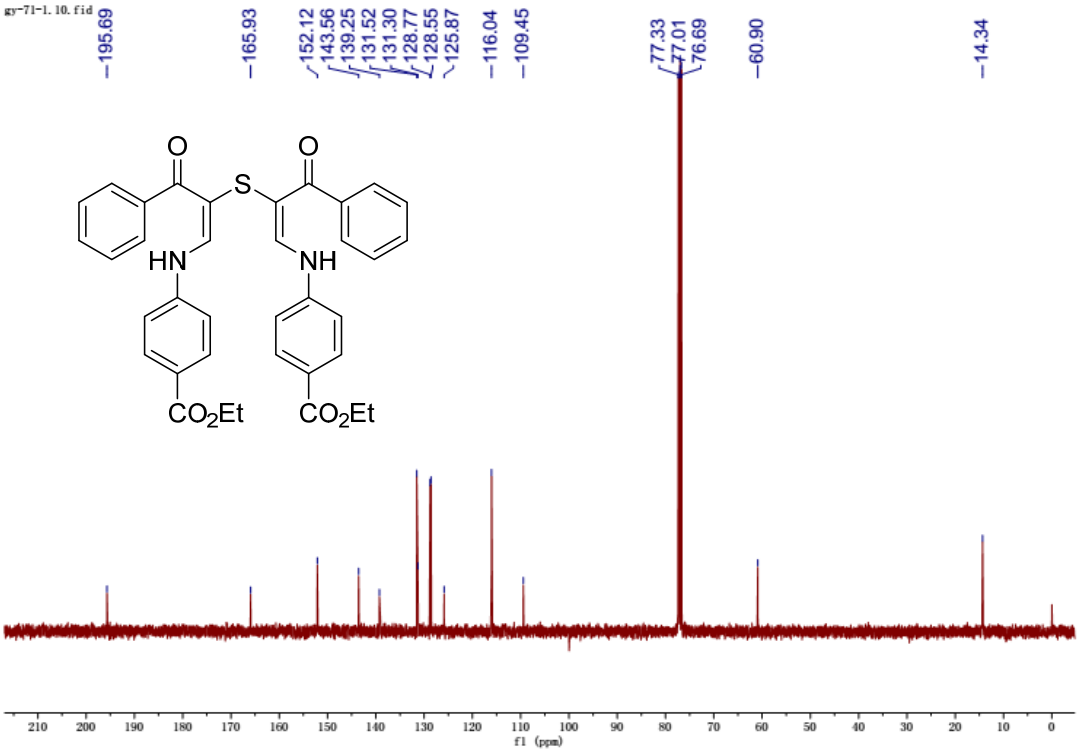
^{1}H and ^{13}C NMR spectra of **4r**



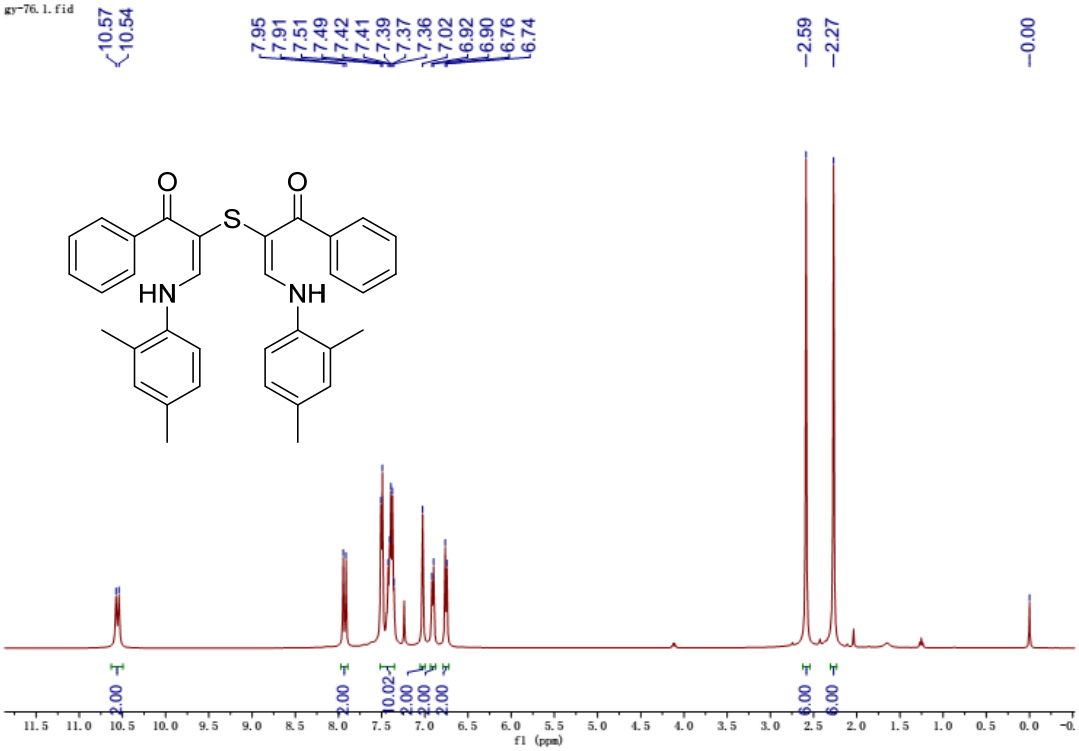


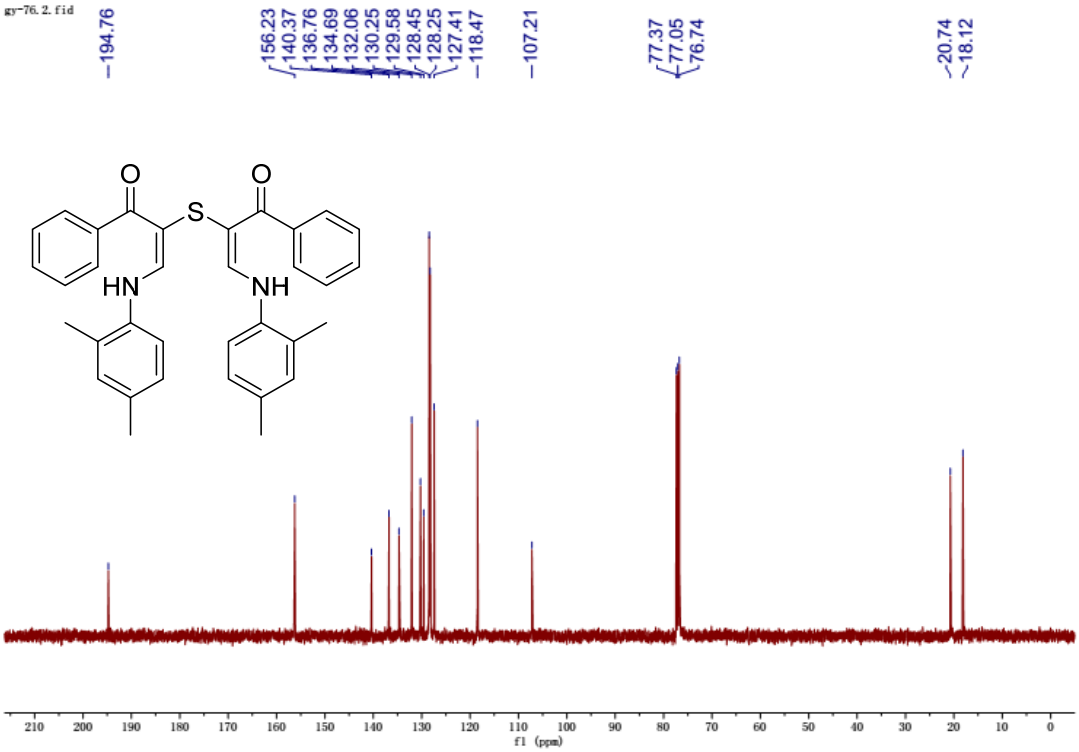
^{1}H and ^{13}C NMR spectra of **4s**



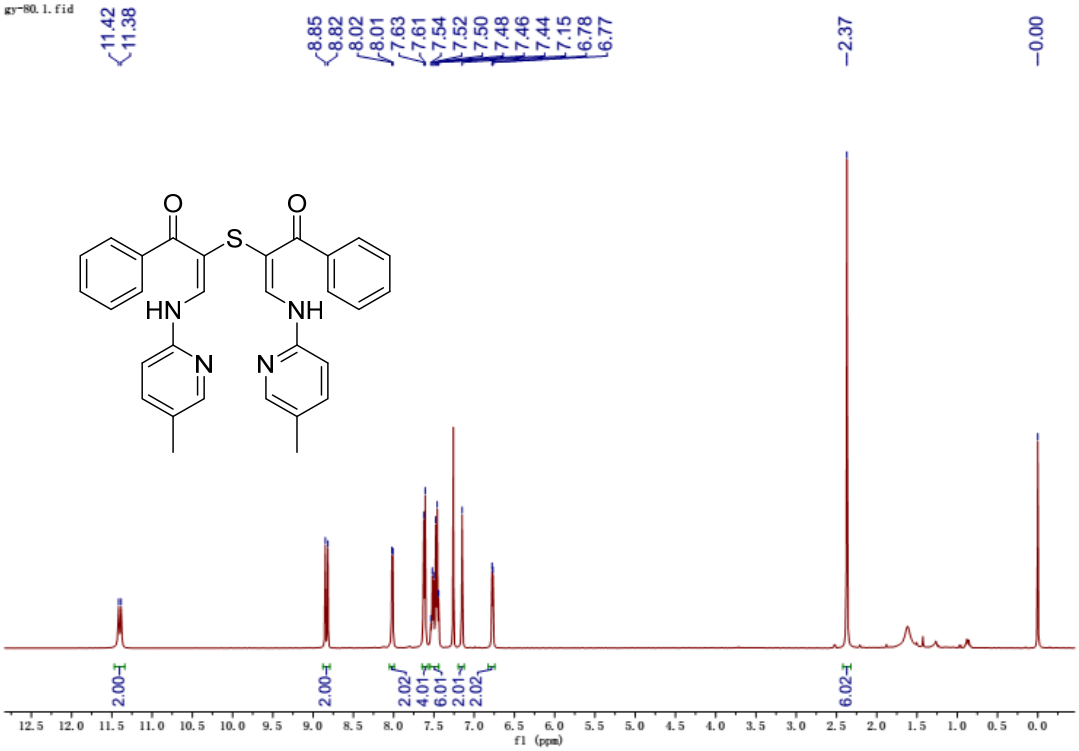


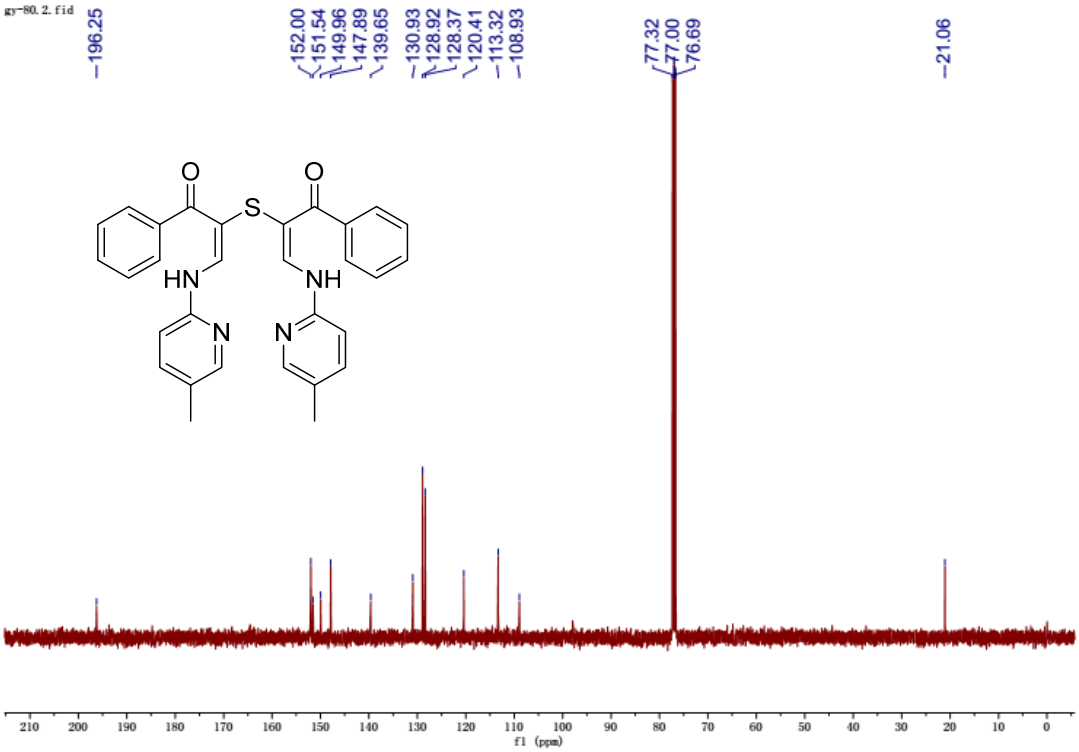
^{1}H and ^{13}C NMR spectra of **4t**



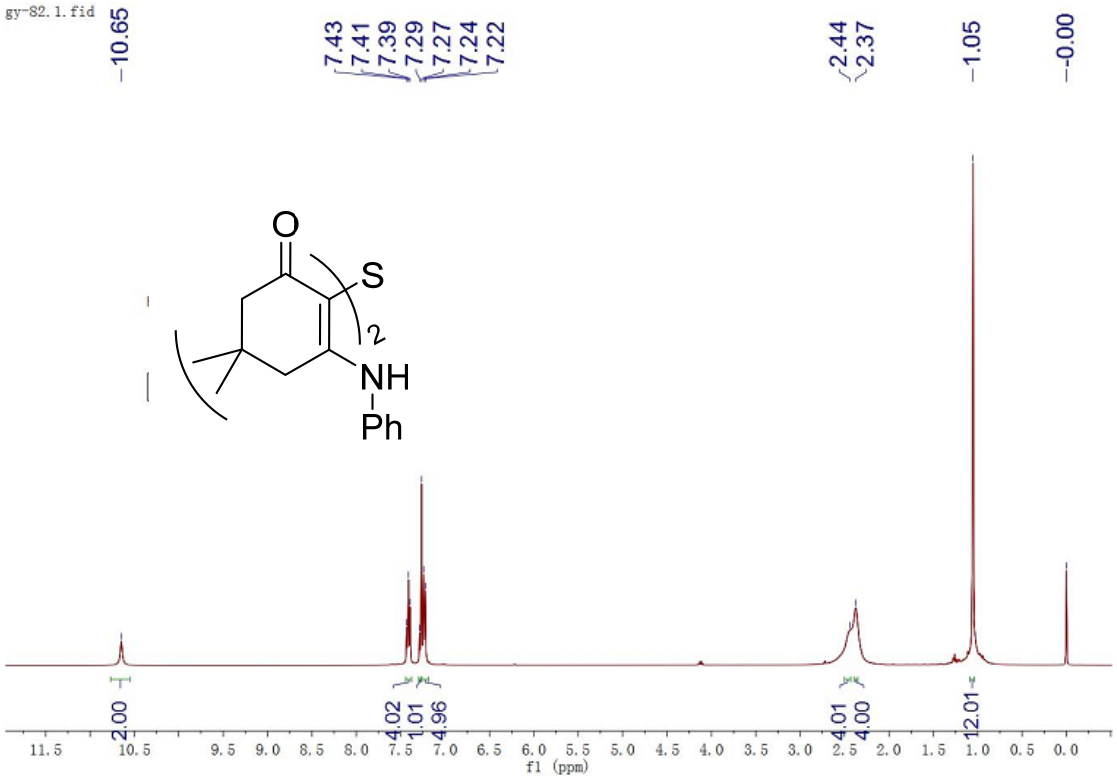


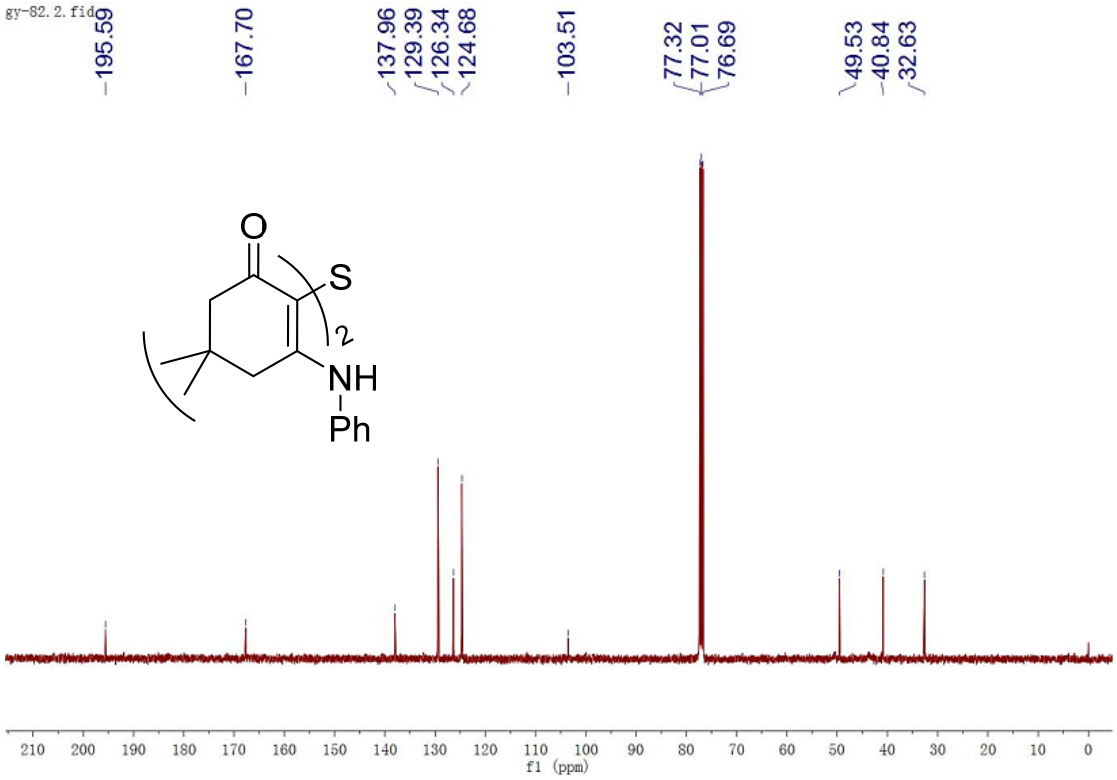
^{1}H and ^{13}C NMR spectra of **4u**



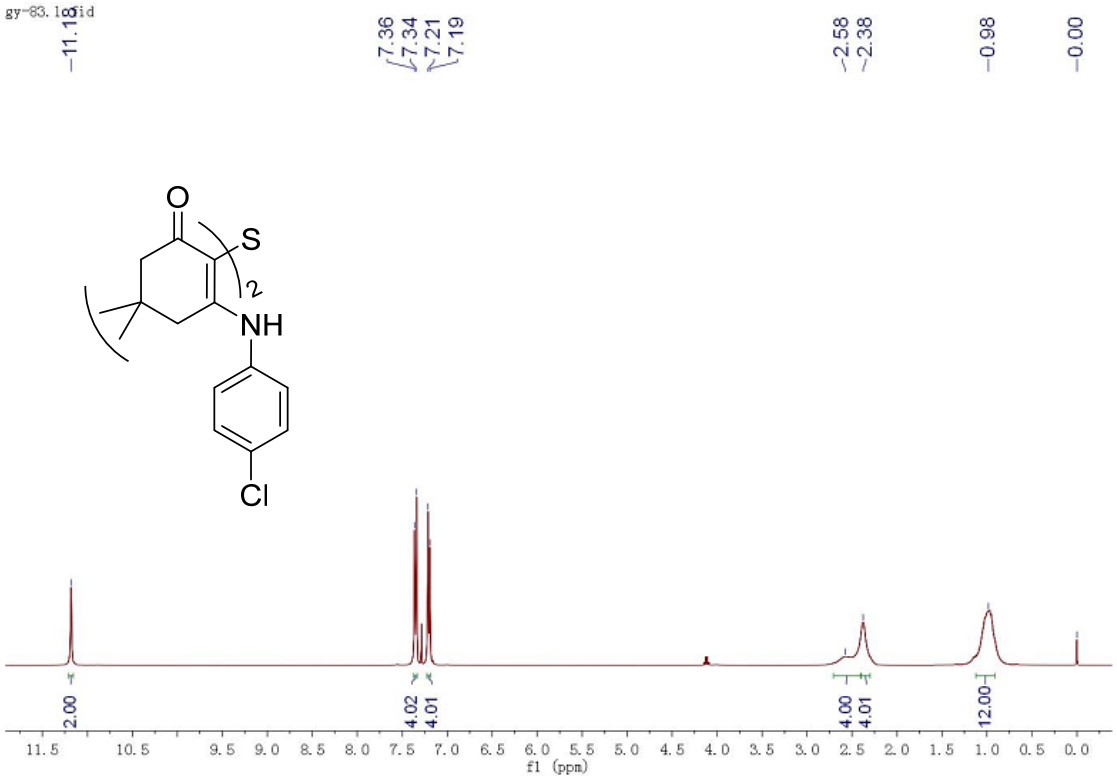


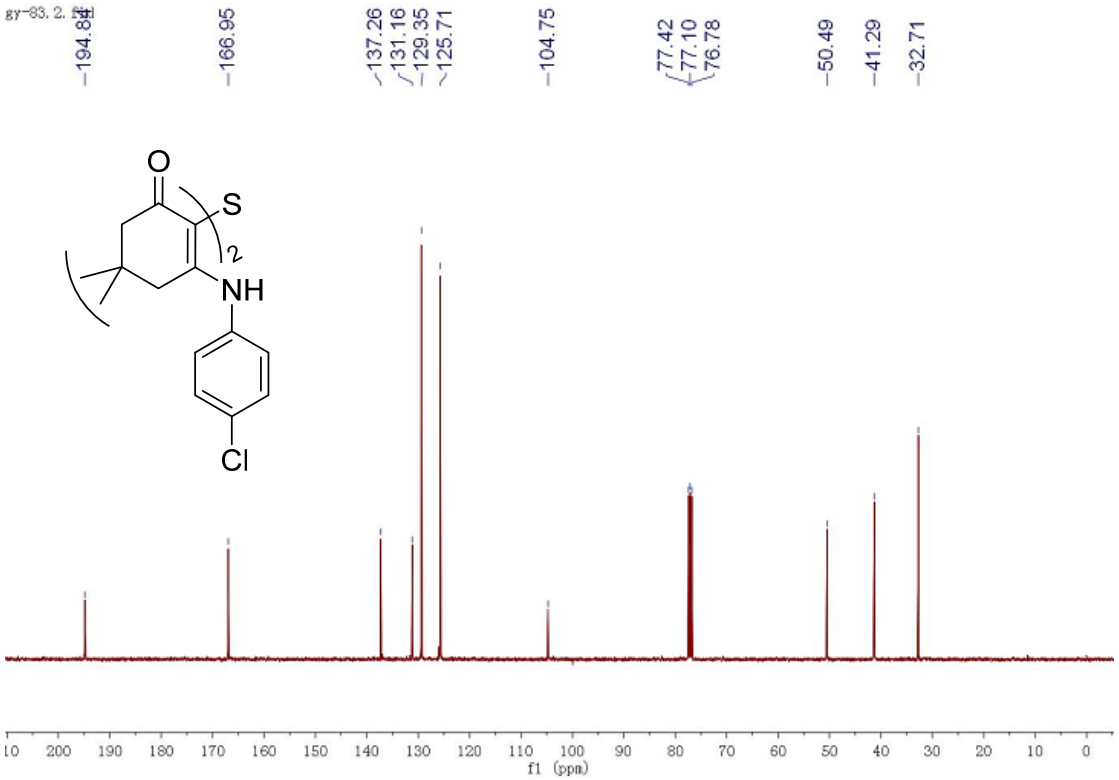
^{1}H and ^{13}C NMR spectra of **6a**



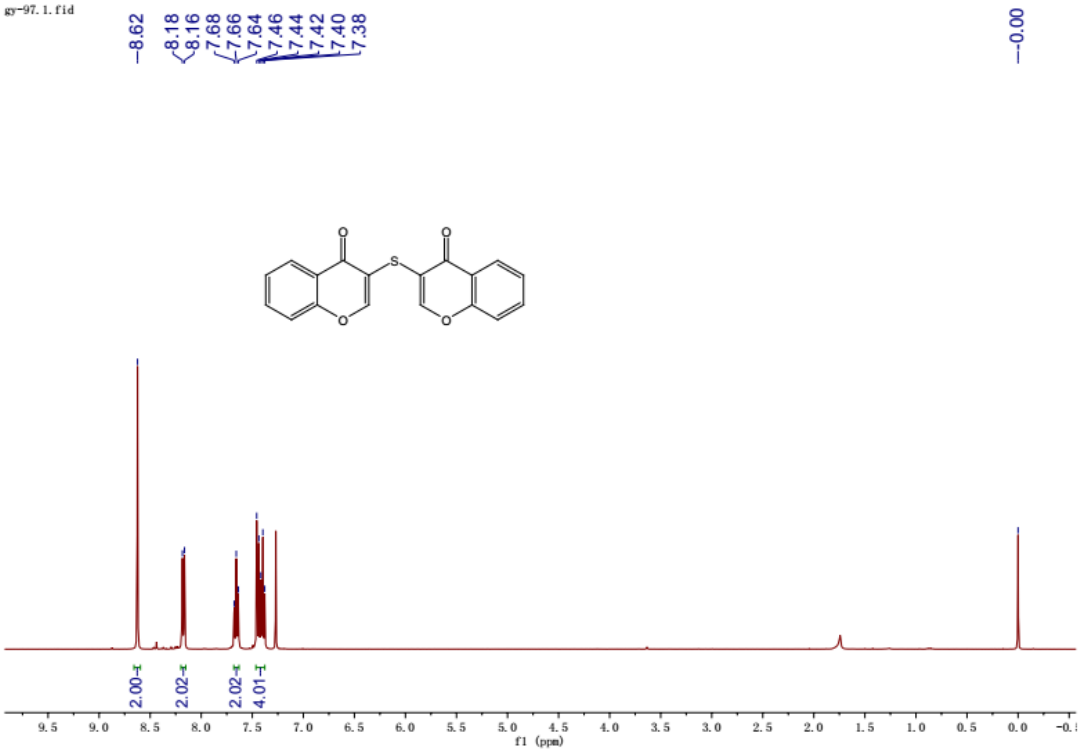


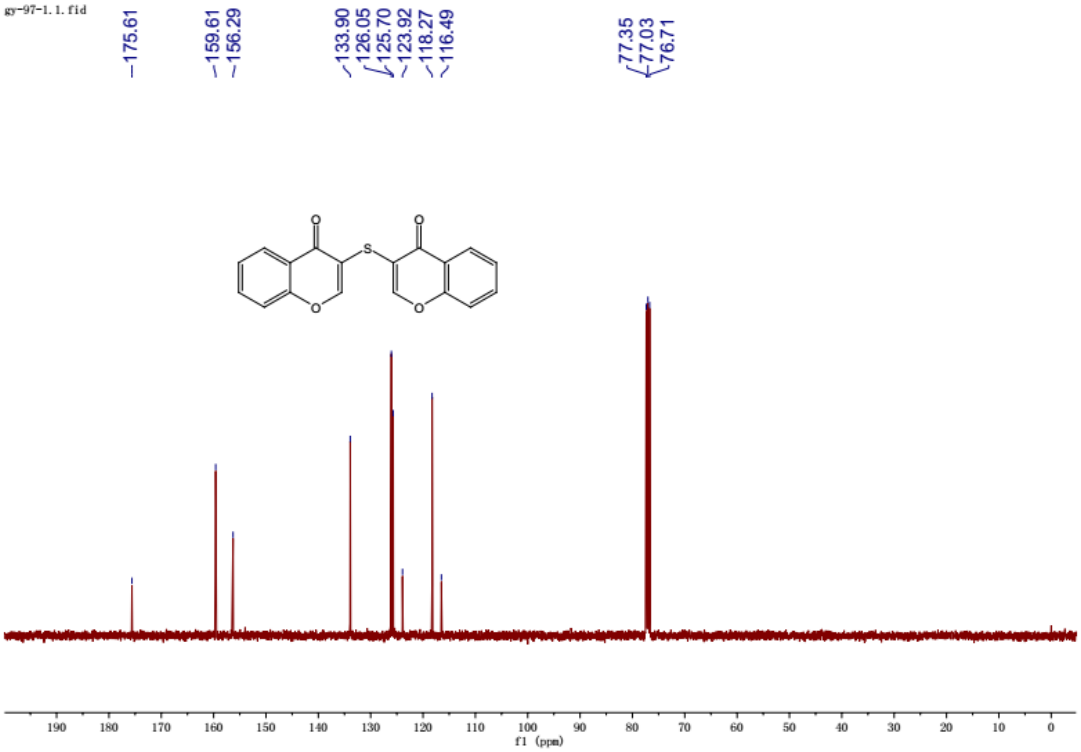
^{1}H and ^{13}C NMR spectra of **6b**



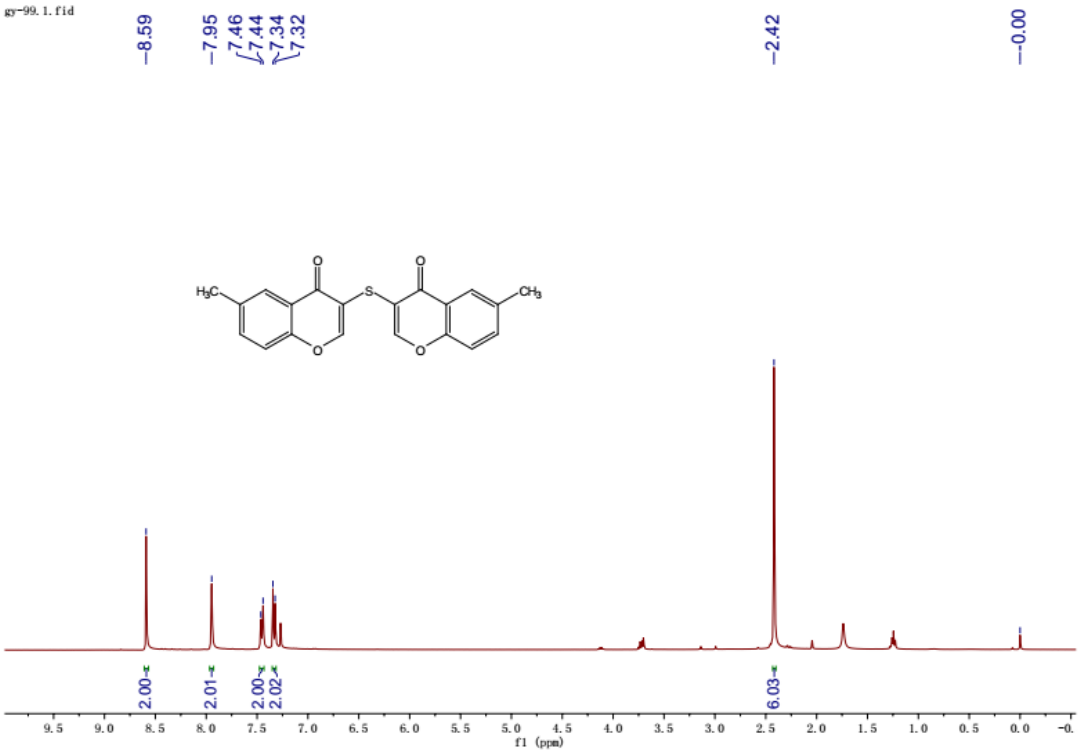


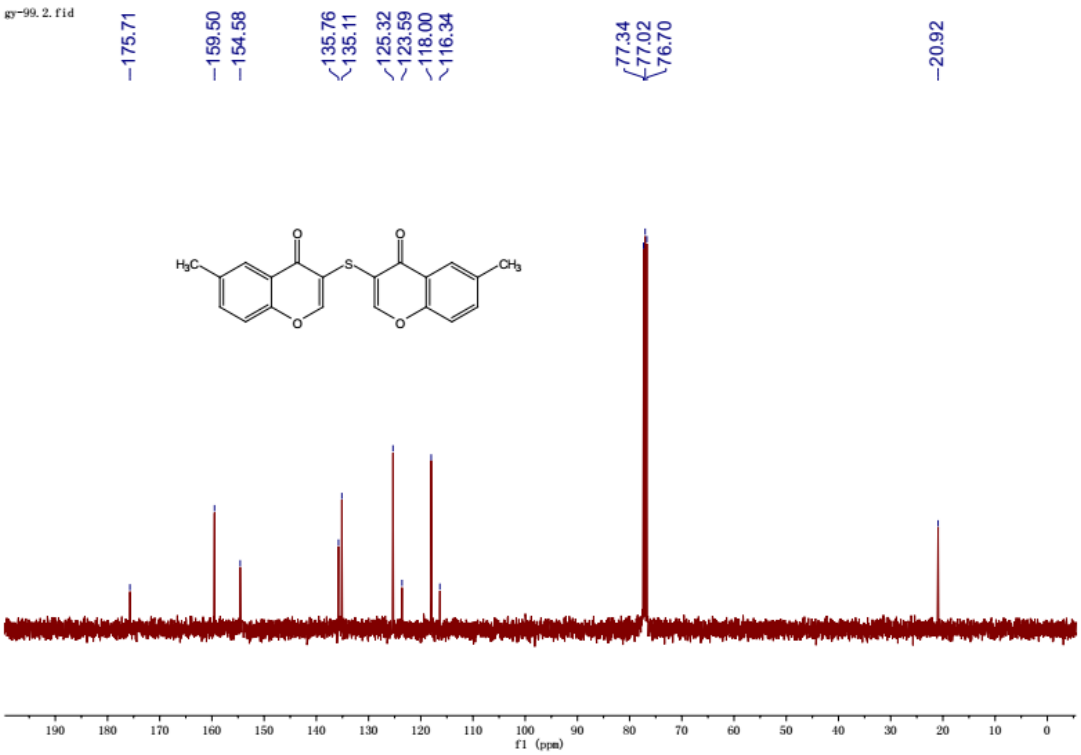
^{1}H and ^{13}C NMR spectra of **8a**





^{1}H and ^{13}C NMR spectra of **8b**





^{1}H and ^{13}C NMR spectra of **8c**