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An Approach to Synthesis of Thioesters and Selenoesters Promoted by Rongalite®

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Description of the Products

Compounds **3a**-**3h**, **3j**-**3m**, **3o**-**3p**, **4a**-**4d** are known, compounds **3i**, **3n** and **3q** are new and described below.

*S-Phenyl thiobenzoate (**3a**)*

White solid; mp 51-52 °C (Lit.[1] mp 54-58 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.45-7.62 (m, 8H), 8.02-8.05 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 127.49, 127.54, 128.81, 129.30, 129.55, 133.70, 135.14, 136.75, 190.08.

*S-4-Tolyl thiobenzoate (**3b**)*

White solid; mp 70-71 °C (Lit.[2] mp 65-66 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.41 (s, 3H),7.29-7.61 (m, 7H), 8.01-8.05 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.29, 123.71, 127.38, 128.63, 130.02, 133.48, 134.94, 136.64, 139.71, 190.44.

*S-4-Chlorophenyl thiobenzoate (**3c**)*

White solid; mp 71-72 °C (Lit.[3] mp 73-74°C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.44-7.63 (m, 7H), 8.00-8.03 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 125.81, 127.47, 128.78, 129.46, 133.82, 135.94, 136.27, 136.32, 189.55.

*S-Phenyl, 4-methoxythiobenzoate (**3d**)*

White solid; mp 93-95 °C (Lit.[4] mp 98-99 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.80 (s, 3H), 6.88 (d, *J* 9 Hz, 2H), 7.35-7.44 (m, 5H), 7.93 (d, *J* 9Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.55, 113.94, 127.71, 129.17, 129.36, 129.47, 129.72, 135.19, 164.03, 188.57.

*S-4-Tolyl 4-methoxythiobenzoate (**3e**)*[5]

White solid; mp 59-62 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.41 (s, 3H), 3.89 (s, 3H), 6.96 (d, *J* 9 Hz, 2H), 7.25-7.28 (m, 2H), 7.40 (d, *J* 8Hz, 2H), 8.01 (d, *J* 9Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 20.31, 54.50, 112.87, 123.10, 128.52, 128.65, 128.99, 134.08, 138.57, 162.93, 187.94.

*S-4-Chlorophenyl 4-methoxythiobenzoate (**3f**)*

White solid; mp 93-94 °C (Lit.[6] mp 98-101 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.81 (s, 3H), 6.88-6.91 (m, 2H), 7.35 (m, 4H), 7.92 (d, *J* 6.9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.57, 114.01, 126.22, 129.16, 129.41, 129.77, 135.81, 136.39, 164.18, 188.00.

*S-Phenyl 2-iodothiobenzoate (**3g**)*[7]

White solid; mp 54-56 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.19 (t, 1H), 7.45-7.60 (m, 6H), 7.70-7.73 (m, 1H), 7.95-7.98 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 127.54, 127.80, 128.04, 128.59, 129.39, 129.76, 132.44, 134.59, 140.85, 142.47, 192.32.

*S-4-Tolyl 2-iodothiobenzoate (**3h**)*[8]

White solid; mp 60-63 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.41 (s, 3H), 7.18 (t, 1H), 7.27-7.47 (m, 5H), 7.71 (d, *J* 8 Hz, 1H), 7.96 (d, *J* 8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.54, 123.99, 128.00, 128.57, 129.98, 130.23, 132.36, 134.55, 140.08, 140.82, 142.58, 192.80.

*S-4-Chlorophenyl 2-iodothiobenzoate (**3i**)*

White solid; mp 82-85 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.20-7.21 (m, 1H), 7.43 -7.51 (m, 5H), 7.69-7.72 (m, 1H), 7.96-7.99 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 91.56, 125.98, 128.07, 128.61, 129.64, 132.62, 135.82, 136.22, 140.95, 142.08, 191.71. ESI-MS: *m/z* (%): 376 ([M+2]$^+$, 32), 374 (M$^+$, 100). Anal. calc. for $C_{13}H_8ClIOS$: C, 41.68; H, 2.15; Found: C, 41.72; H, 2.21.

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*S-Phenyl thiopropionate (**3j**)*[9]

Oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.24 (t, *J* 7.50 Hz, 3H), 2.70 (q, *J* 7.49 Hz, 2H), 7.42-7.43 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 8.75, 36.09, 126.91, 128.12, 128.26, 133.50, 197.13.

*S-4-Tolyl thiopropionate (**3k**)*[3]

Oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.21 (t, *J* 7.47 Hz, 3H), 2.36 (s, 3H), 2.67 (q, *J* 7.50 Hz, 2H), 7.19 -7.30 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.53, 21.20, 36.90, 124.30, 129.89, 134.39, 139.43, 198.57.

*S-4-Chlorophenyl thiopropionate (**3l**)*[3]

Oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (t, *J* 7.48 Hz, 3H), 2.69 (q, *J* 7.48 Hz, 2H), 7.32 -7.41 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 8.43, 36.09, 126.87, 128.12, 128.26, 133.50, 197.16.

*S-Phenyl 2-Furancarbothioate (**3m**)*

White solid; mp 51-52 °C (Lit.[10] mp 51-52 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.58-6.60 (m, 1H), 7.27 (d, *J* 3.6 Hz, 1H), 7.46-7.63 (m, 5H), 7.64 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 111.39, 115.21, 125.23, 128.23, 128.55, 134.12, 145.45, 149.39, 177.59.

*S-4-Tolyl 2-Furancarbothioate (**3n**)*

White solid; mp 63-65°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.41 (s, 3H), 6.57-6.59 (m, 1H), 7.25-7.41 (m, 5H), 7.63 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.34, 112.35, 116.09, 122.64, 130.10, 135.06, 139.95, 146.37, 150.49, 179.06. ESI-MS: *m/z* (%): 218 (M^+, 100). Anal. calc. for $C_{12}H_{10}O_2S$: C, 66.03; H, 4.62; Found: C, 60.10; H, 4.59.

*S-Phenyl 3-phenyl-3-(phenylthio)propanethioate (**4a**)*[11]

White solid; mp 74-76°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.27 (d, *J* 6 Hz, 2H), 4.75 (t, *J* 6 Hz, 1H), 7.23-7.43 (m, 15H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 49.28, 49.42, 127.37, 127.70, 127.82, 128.54, 128.94, 129.18, 129.48, 133.67, 134.39, 139.97, 194.59.

*S-4-Tolyl 3-phenyl-3-(phenylthio)propanethioate (**4b**)*[12]

White solid; mp 82-83 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.34 (s, 3H), 2.38 (s, 3H), 3.26 (d, *J* 7.6 Hz, 2H), 4.70 (t, *J* 7.6 Hz, 1H), 7.07-7.32 (m, 13H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.10, 21.26, 49.08, 49.44, 127.50, 127.74, 128.38, 128.91, 129.63, 129.68, 129.99, 133.70, 134.26, 138.23, 139.66, 139.96, 194.59.

*S-4-Chlorophenyl 3-(4-chlorophenylthio)-3-phenylpropanethioate (**4c**)*

White solid; mp 63-65 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.24 (d, *J* 7.5 Hz, 2H) , 4.69 (t, *J* 7.6 Hz, 1H), 7.19-7.43 (m, 13H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 49.22, 49.43, 123.9, 125.6, 1207.7, 128.6, 129.4, 130.9, 131.8, 134.2, 135.5, 135.8, 139.5, 141.9, 193.9. ESI-MS: *m/z* (%): 422 ([M+4]$^+$, 10), 420 ([M+2]$^+$, 36), 418 (M^+, 100). Anal. calc. for $C_{21}H_{16}Cl_2OS_2$: C, 60.14; H, 3.85; Found: C, 60.08; H, 3.94.

*Se-Phenyl selenobenzoate (**5a**)*

Yellow solid; mp 40-41 °C (Lit.[12] mp 37-38 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.43-7.52 (m, 5H), 7.60-7.63 (m, 3H), 7.93-7.96 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 125.78, 127.30, 128.91, 129.02, 129.33, 133.84, 136.29, 138.52, 192.30.

*Se-phenyl 4-methoxyselenobenzoate (**5b**)*

White solid; mp 56-58 °C (Lit.[13] mp 61-62 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.96 (d, *J* 6.9Hz, 2H), 7.42-7.44 (m, 3H), 7.59-7.63 (m, 2H), 7.92 (d, *J* 6.9Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.52, 114.05, 125.97, 128.84, 129.22, 129.61, 131.25, 136.35, 164.14, 191.20.

*Se-Phenyl 2-iodoselenobenzoate (**5c**)*

Yellow solid; mp 59-60 °C (Lit.[14] mp 76 °C); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.18-7.18 (m, 1H), 7.43-7.7.45 (m, 4H), 7.63-7.69 (m, 3H), 7.96 (d, *J* 7.9Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 90.20, 126.65, 128.01, 128.39, 129.17, 129.45, 132.53, 135.75, 140.97, 143.76, 195.61.

*Se-Phenyl 2-Furancarboselenoate (**5d**)*[15]

Oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.59-6.60 (m, 1H), 7.21-7.23 (m, 1H), 7.41-7.44 (m, 3H), 7.58-7.63 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 112.77, 115.18, 124.74, 129.10, 129.32, 136.30, 146.57, 151.74, 180.72.

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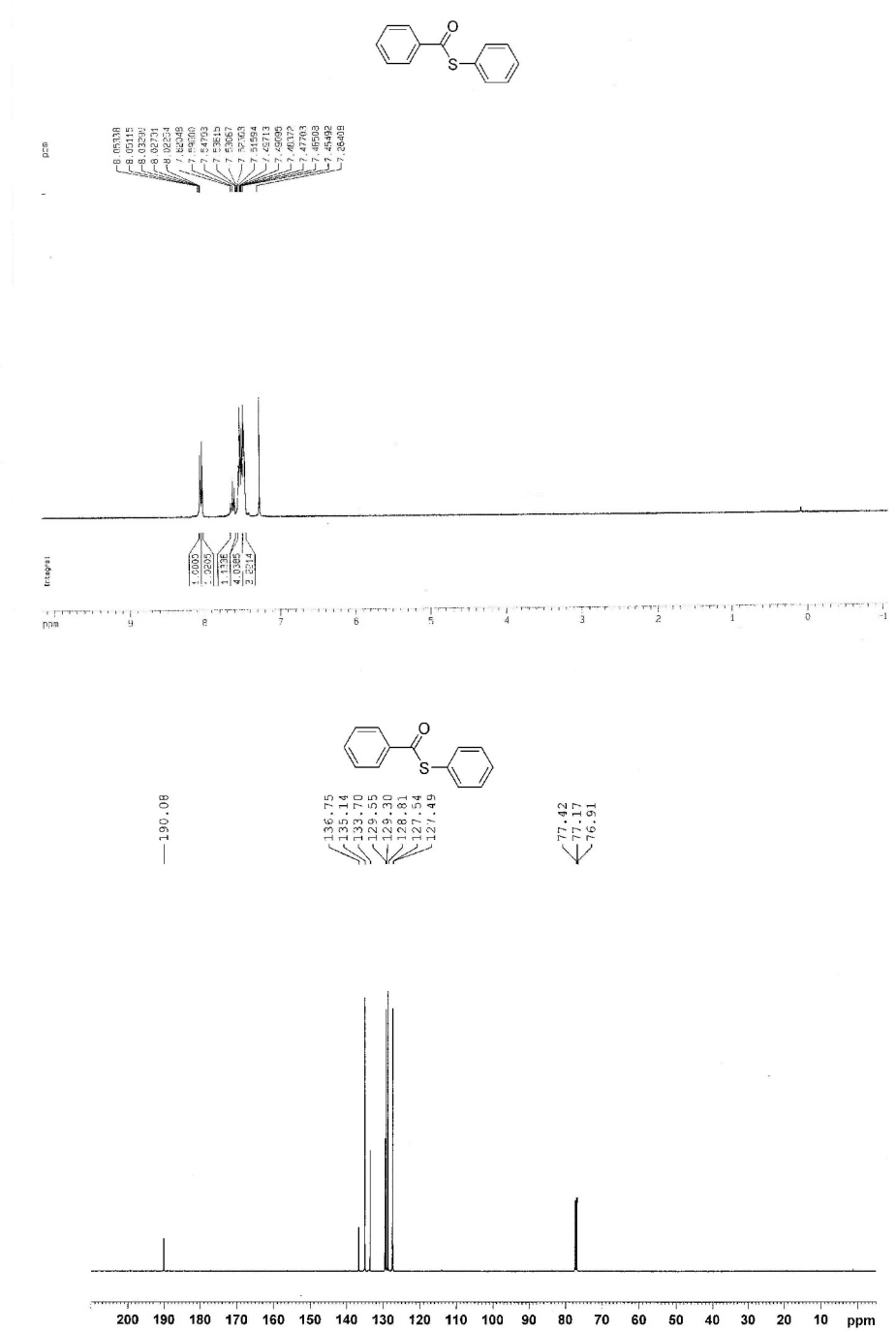

Figure S1. ^{1}H NMR of **3a** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3a** (125 MHz, $CDCl_3$).

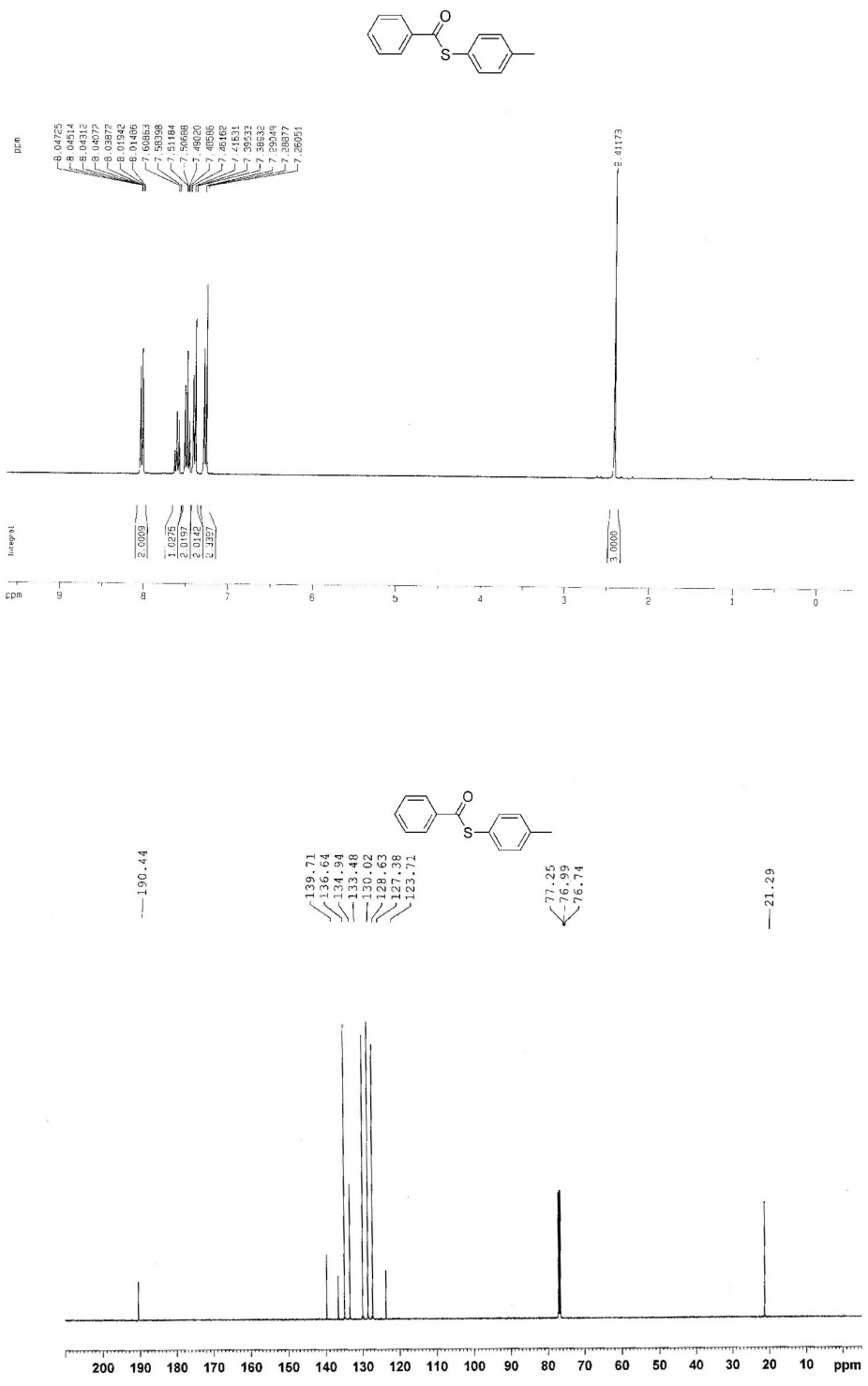

Figure S2. ^{1}H NMR of **3b** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3b** (125 MHz, $CDCl_3$).

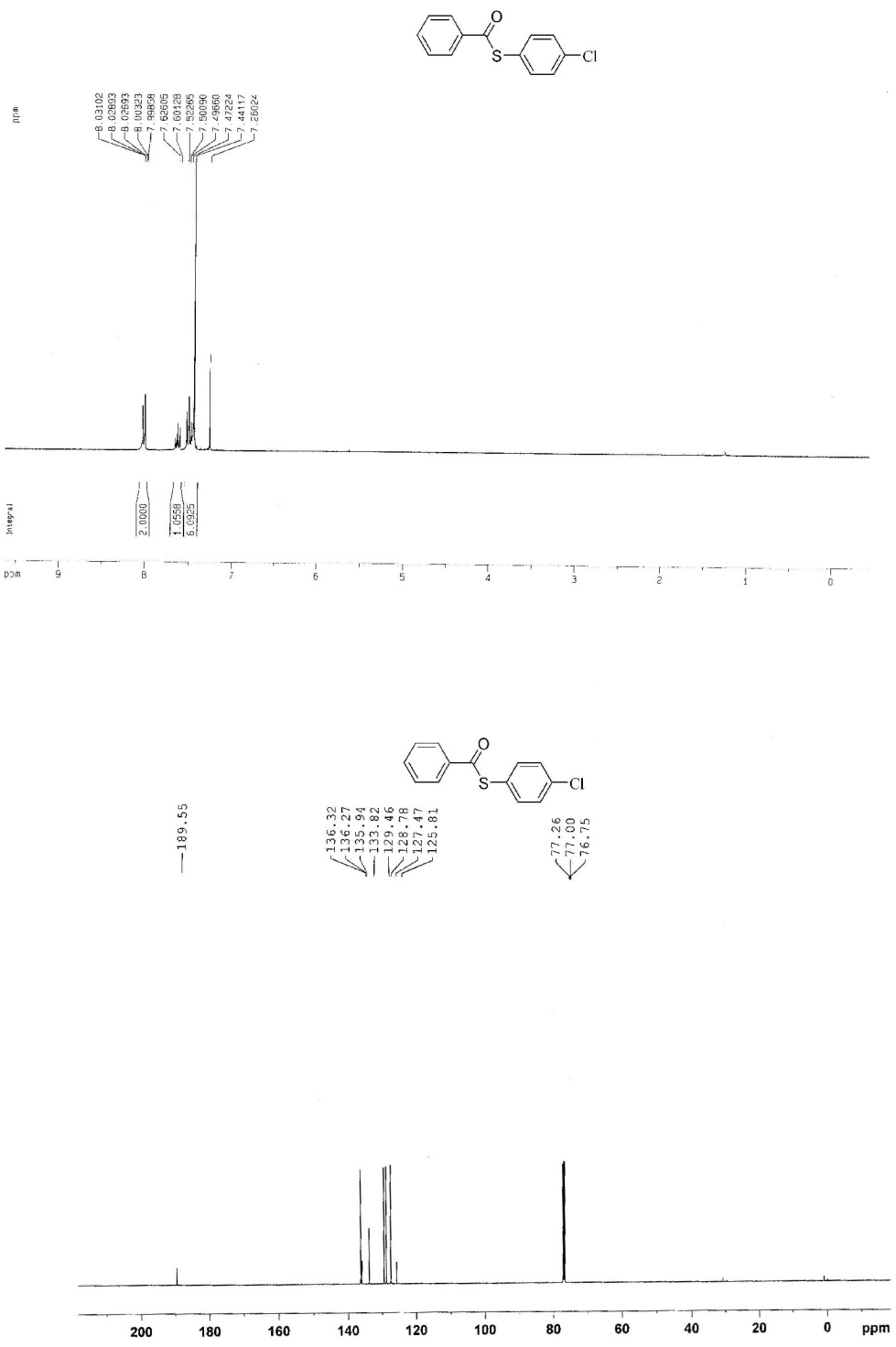

Figure S3. ^{1}H NMR of **3c** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3c** (125 MHz, $CDCl_3$).

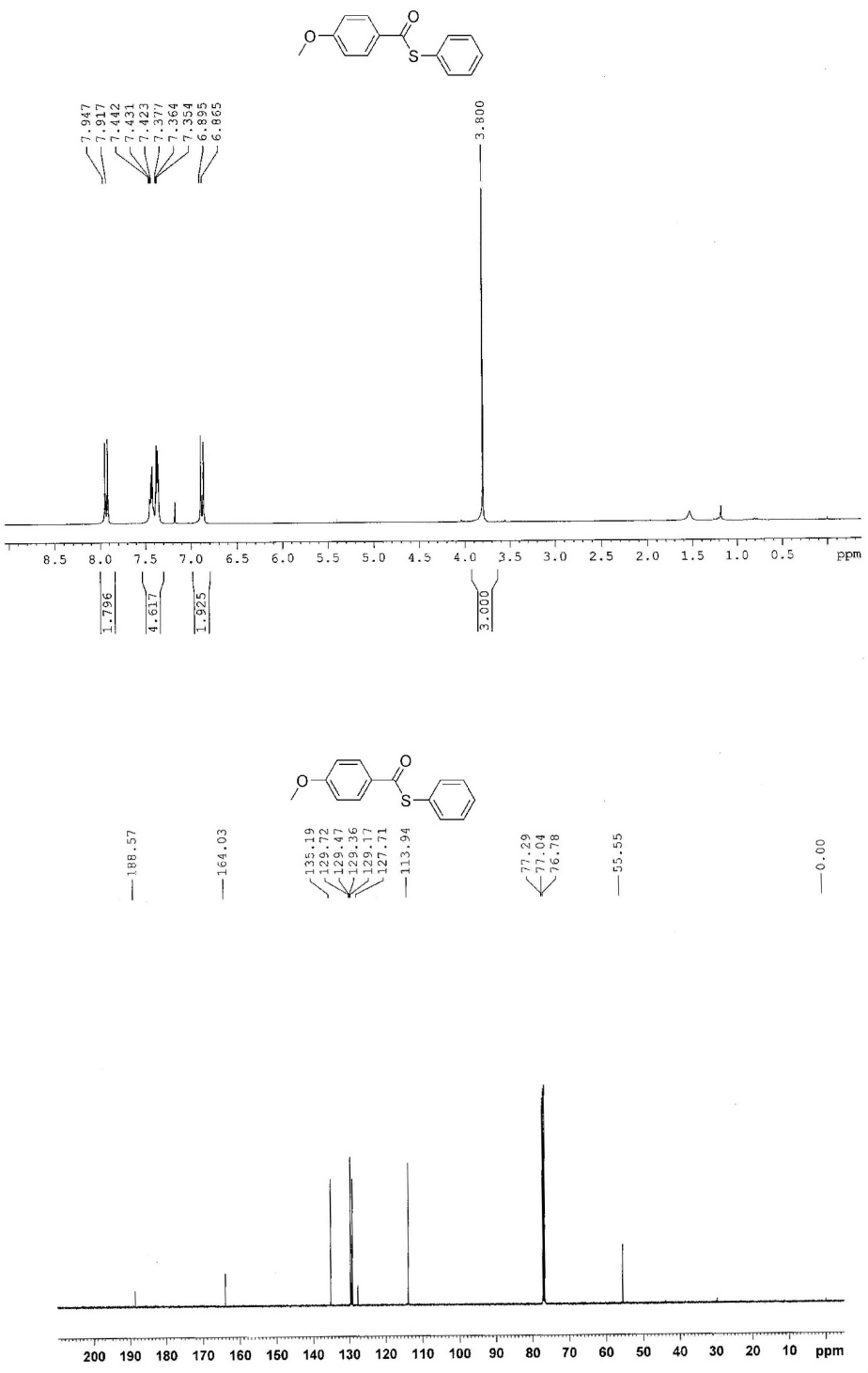

Figure S4. ^{1}H NMR of **3d** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3d** (125 MHz, $CDCl_3$).

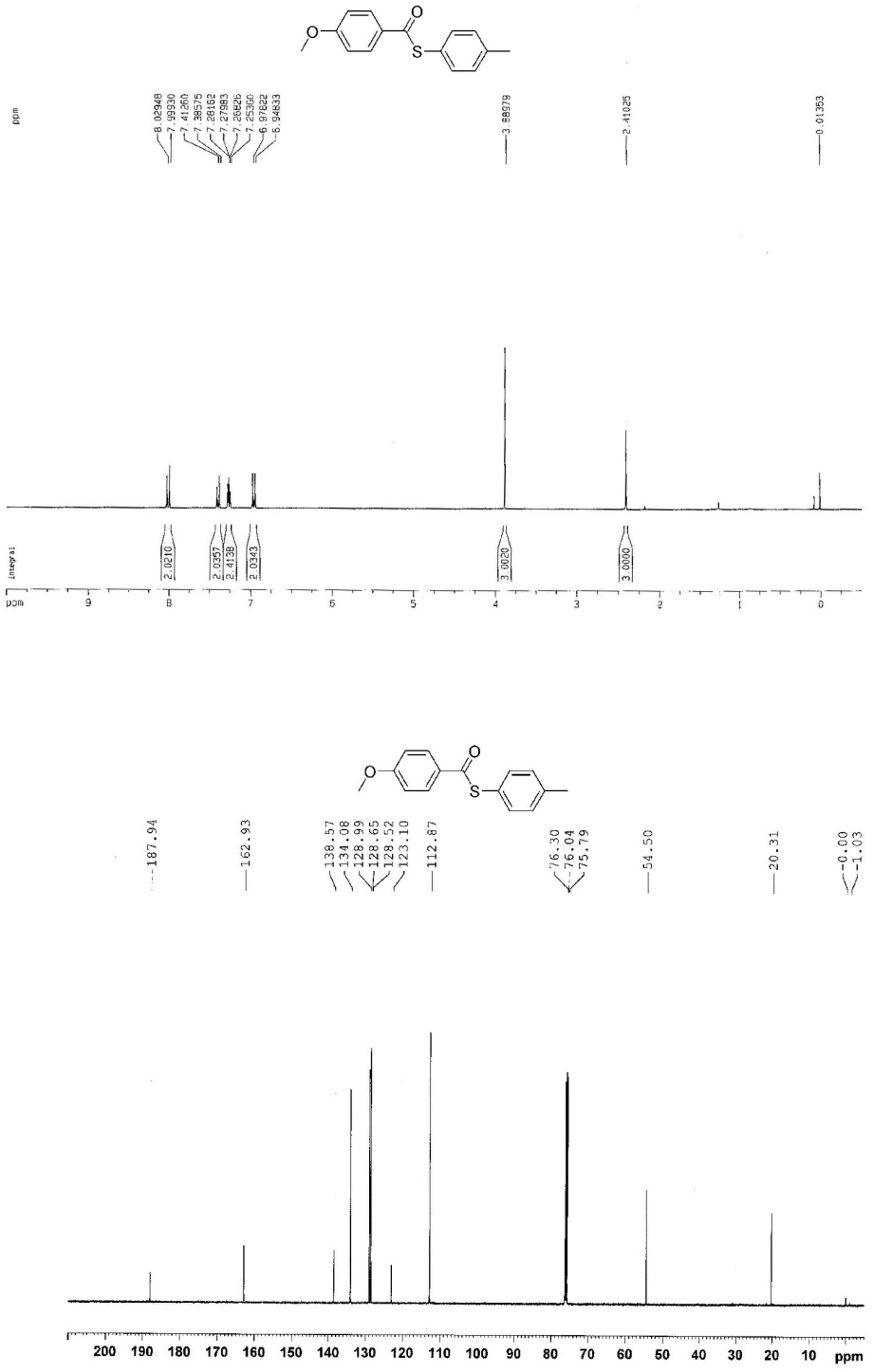

Figure S5. ^{1}H NMR of **3e** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3e** (125 MHz, $CDCl_3$).

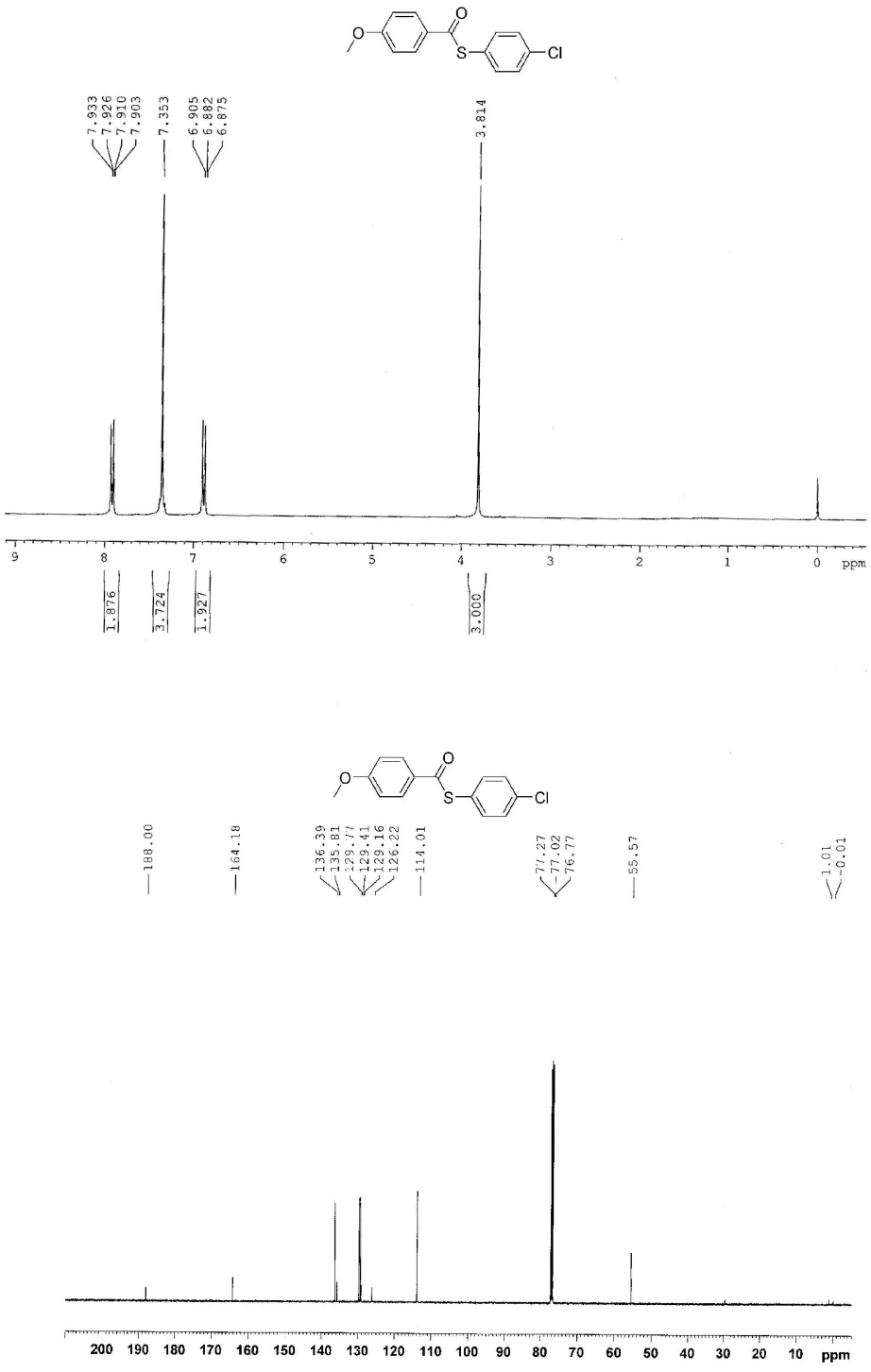

Figure S6. ^{1}H NMR of **3f** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3f** (125 MHz, $CDCl_3$).

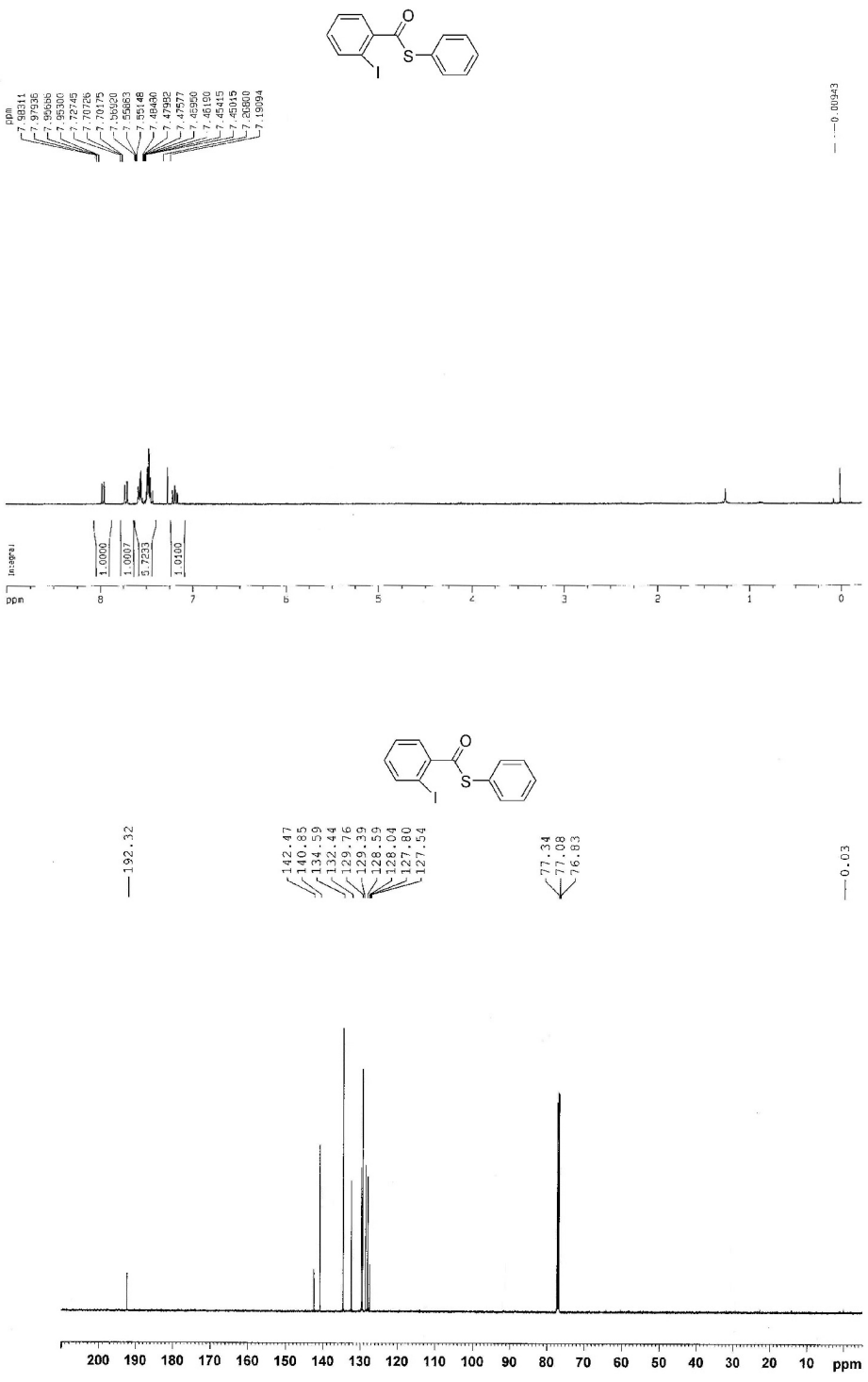

Figure S7. ^{1}H NMR of **3g** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3g** (125 MHz, $CDCl_3$).

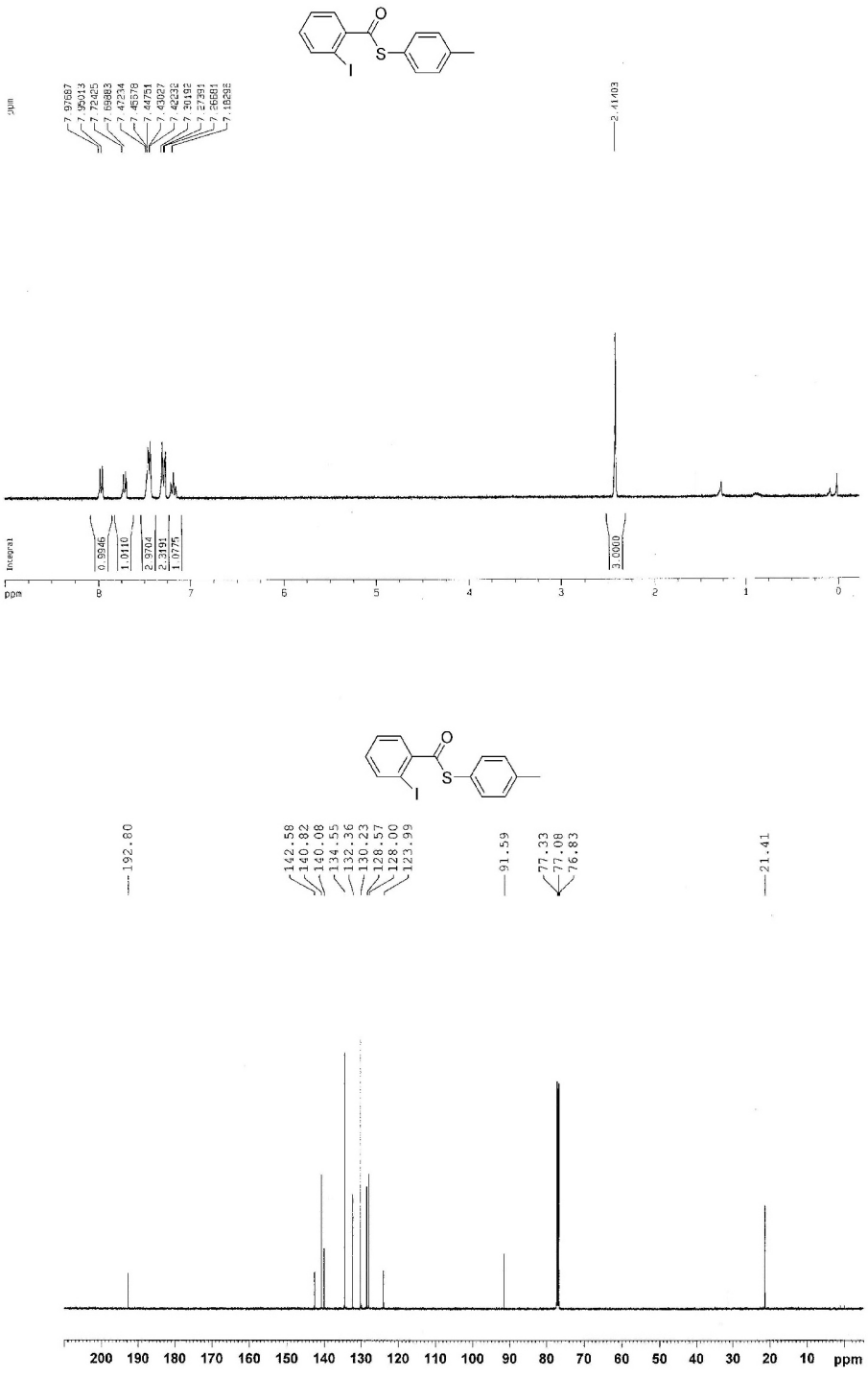

Figure S8. ^{1}H NMR of **3h** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3h** (125 MHz, $CDCl_3$).

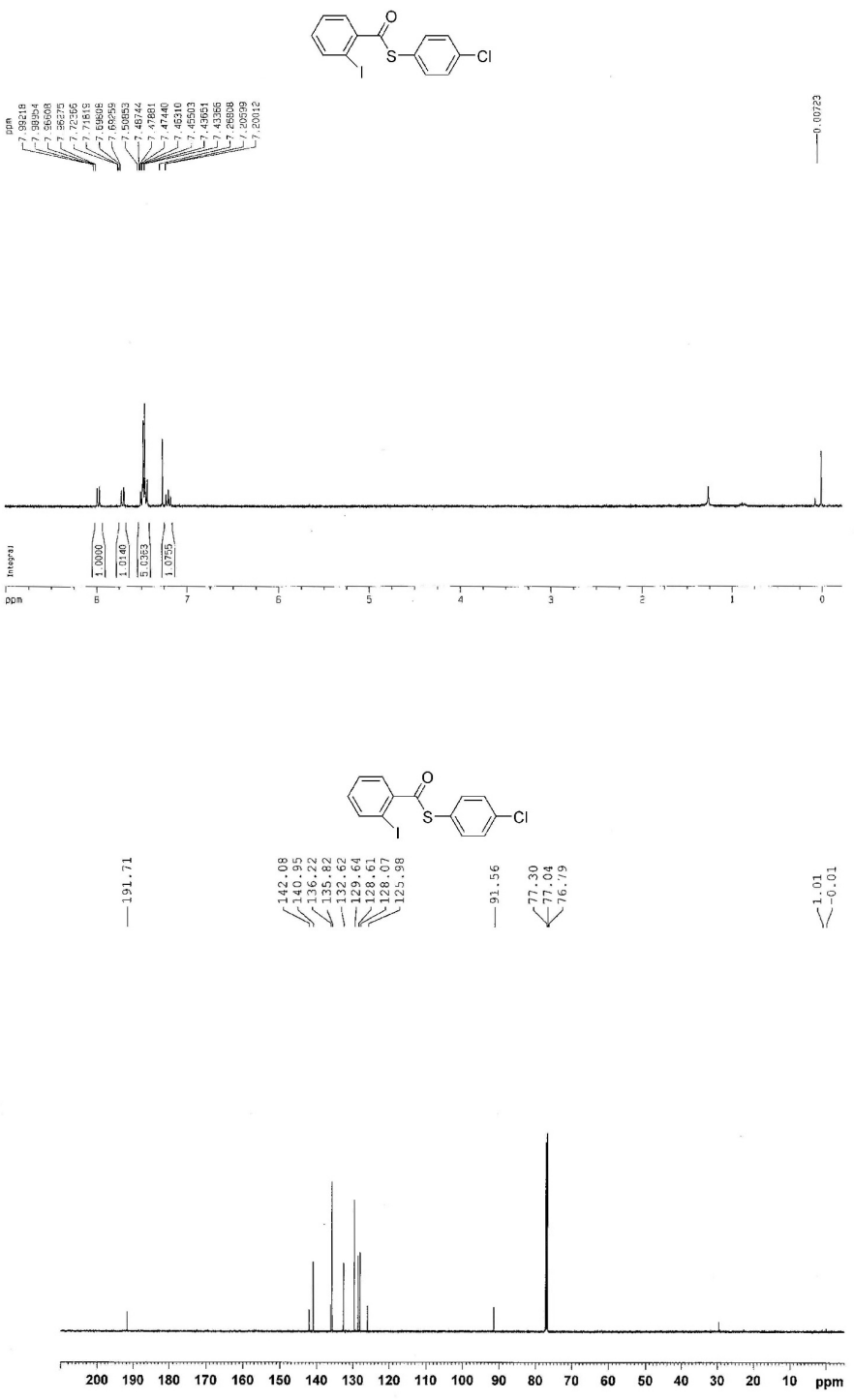

Figure S9. ^{1}H NMR of **3i** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3i** (125 MHz, $CDCl_3$).

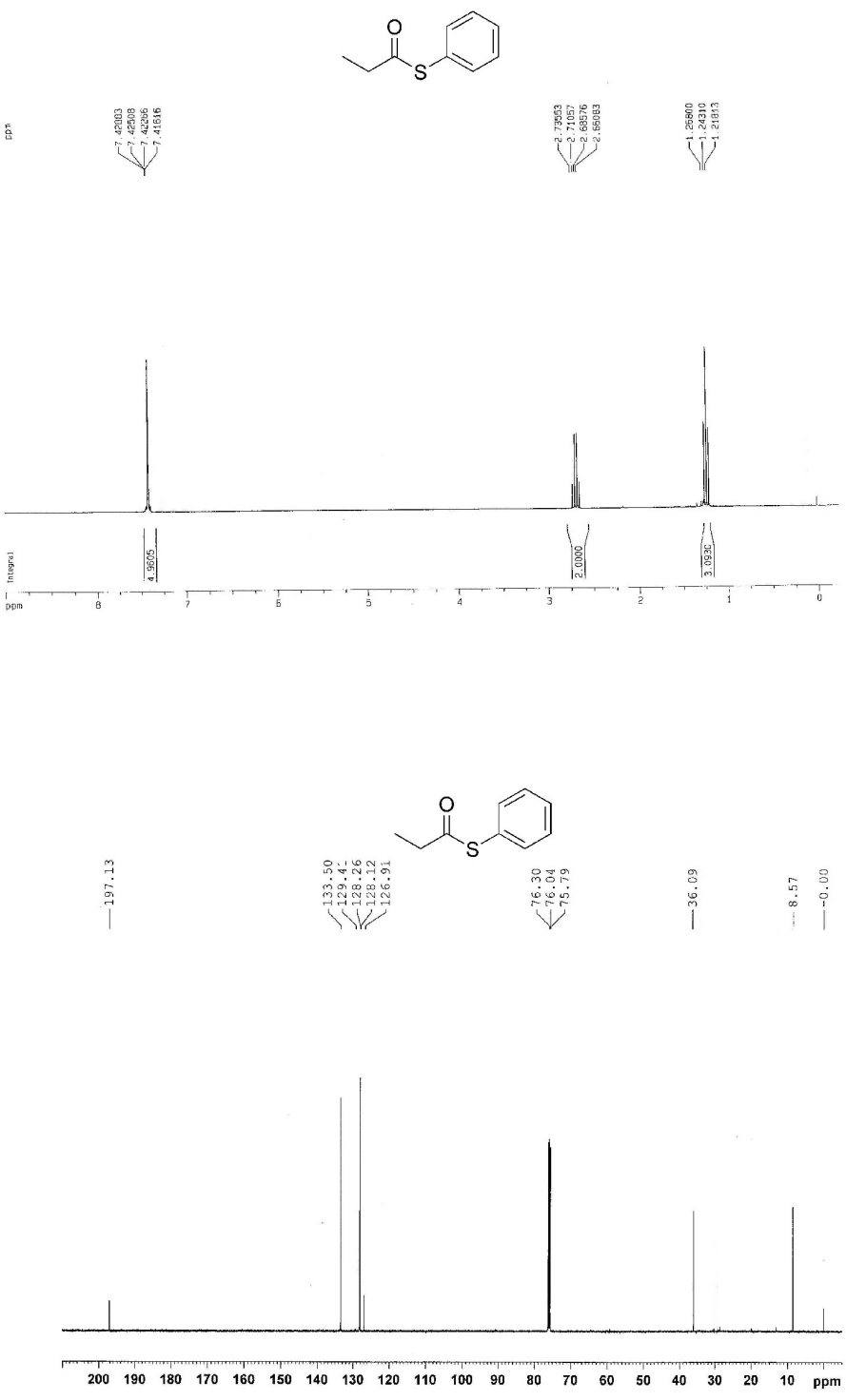

Figure S10. ^{1}H NMR of **3j** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3j** (125 MHz, $CDCl_3$).

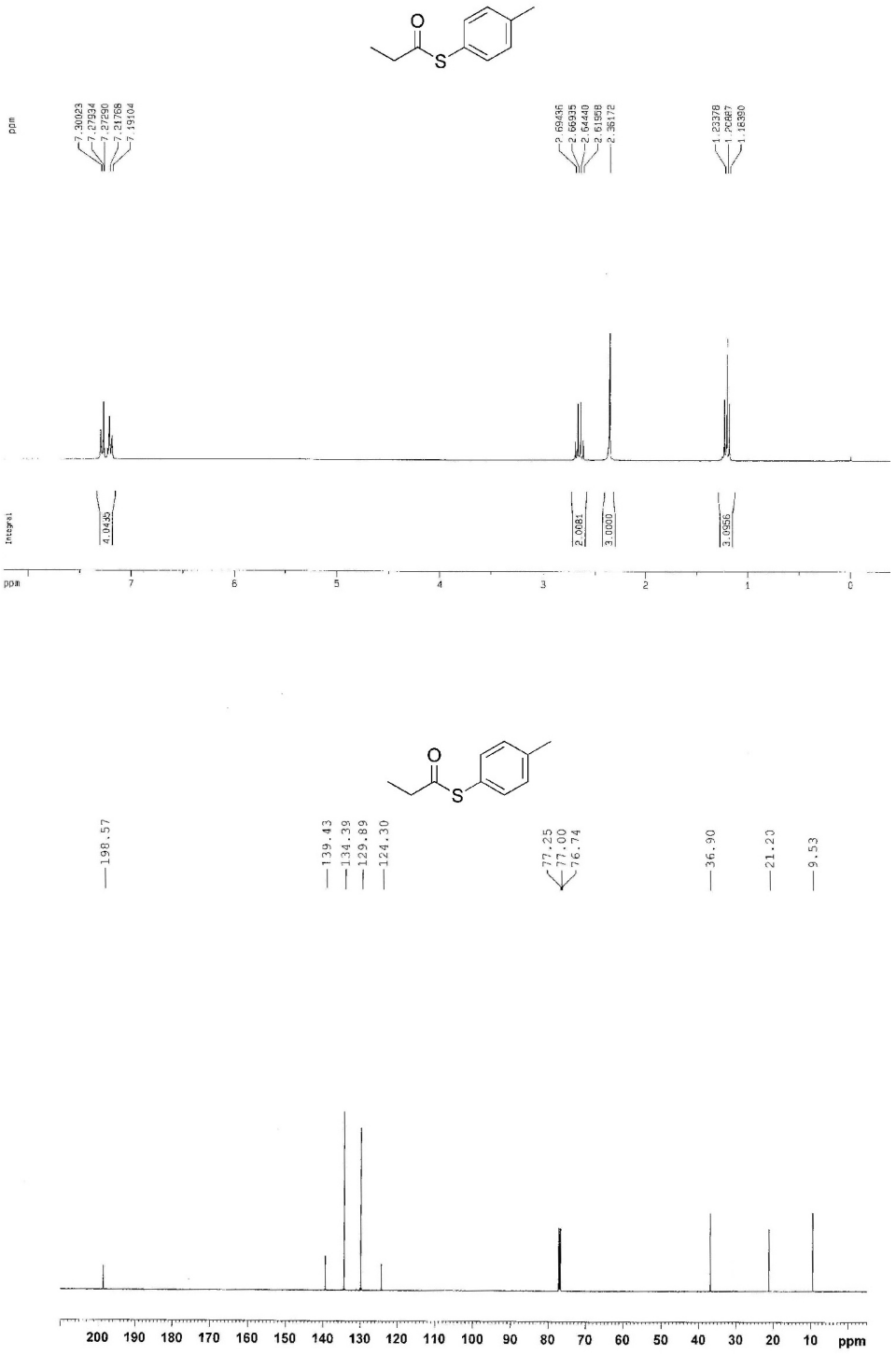

Figure S11. ^{1}H NMR of **3k** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3k** (125 MHz, $CDCl_3$).

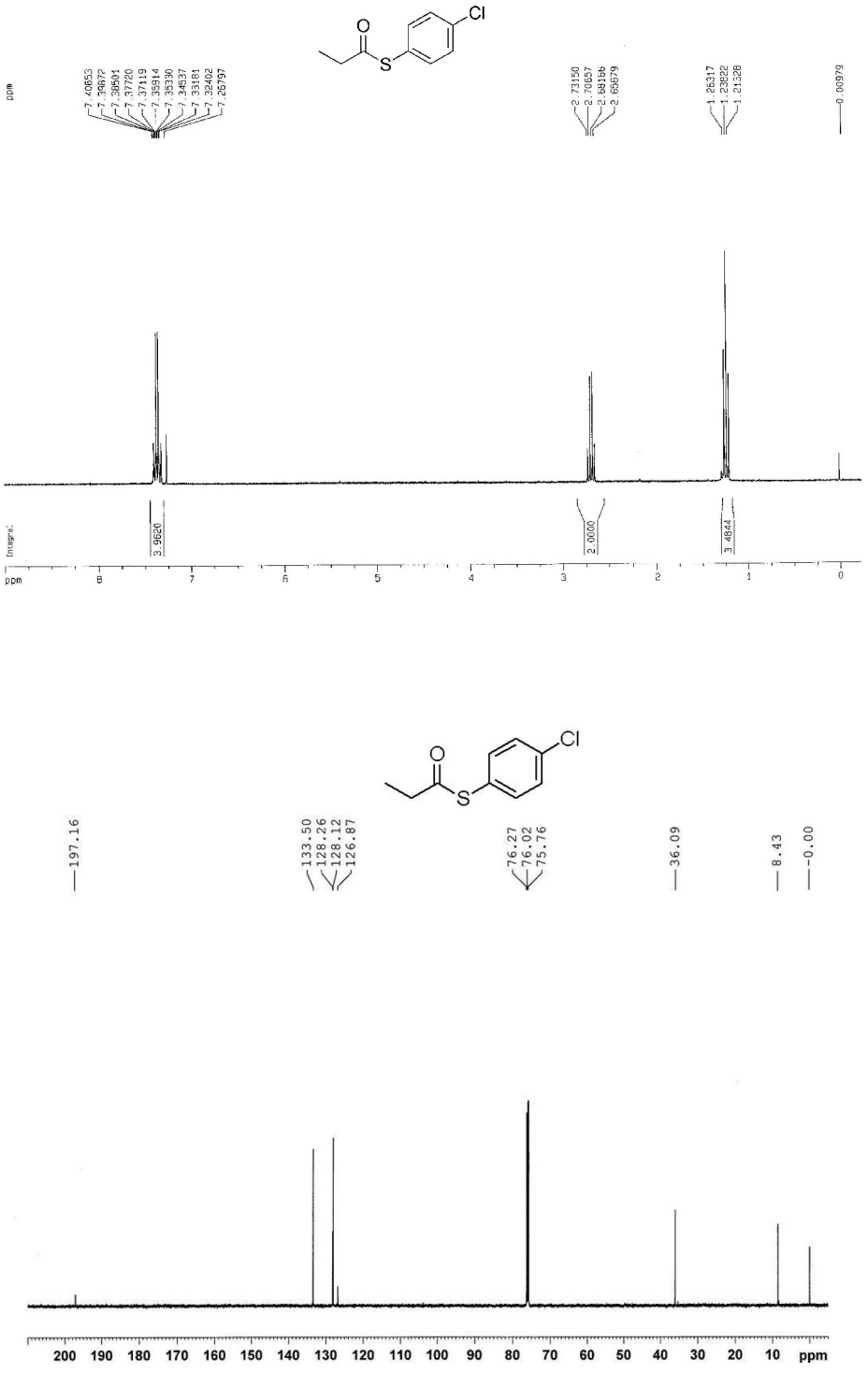

Figure S12. ^{1}H NMR of **3l** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3l** (125 MHz, $CDCl_3$).

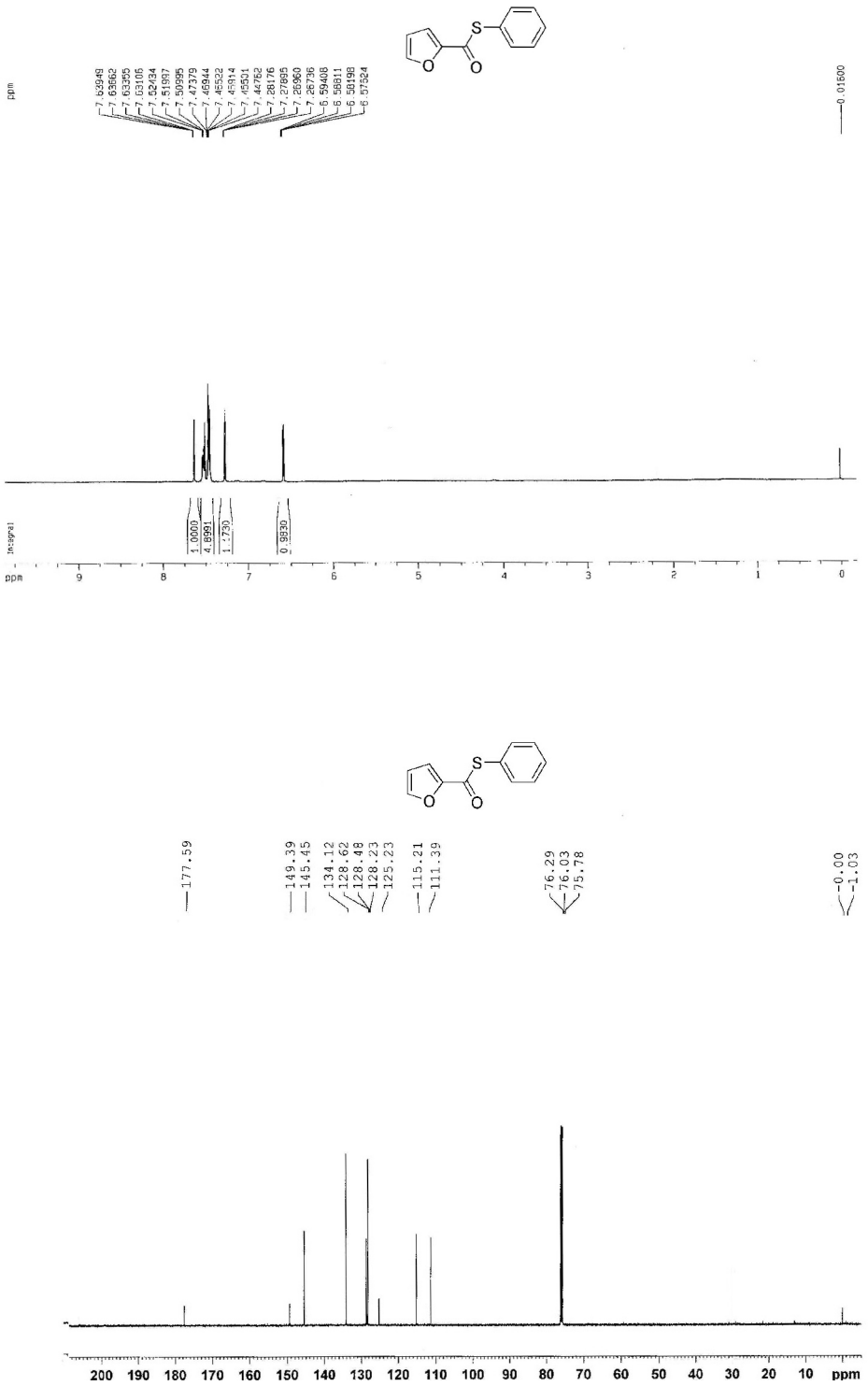

Figure S13. ^{1}H NMR of **3m** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3m** (125 MHz, $CDCl_3$).

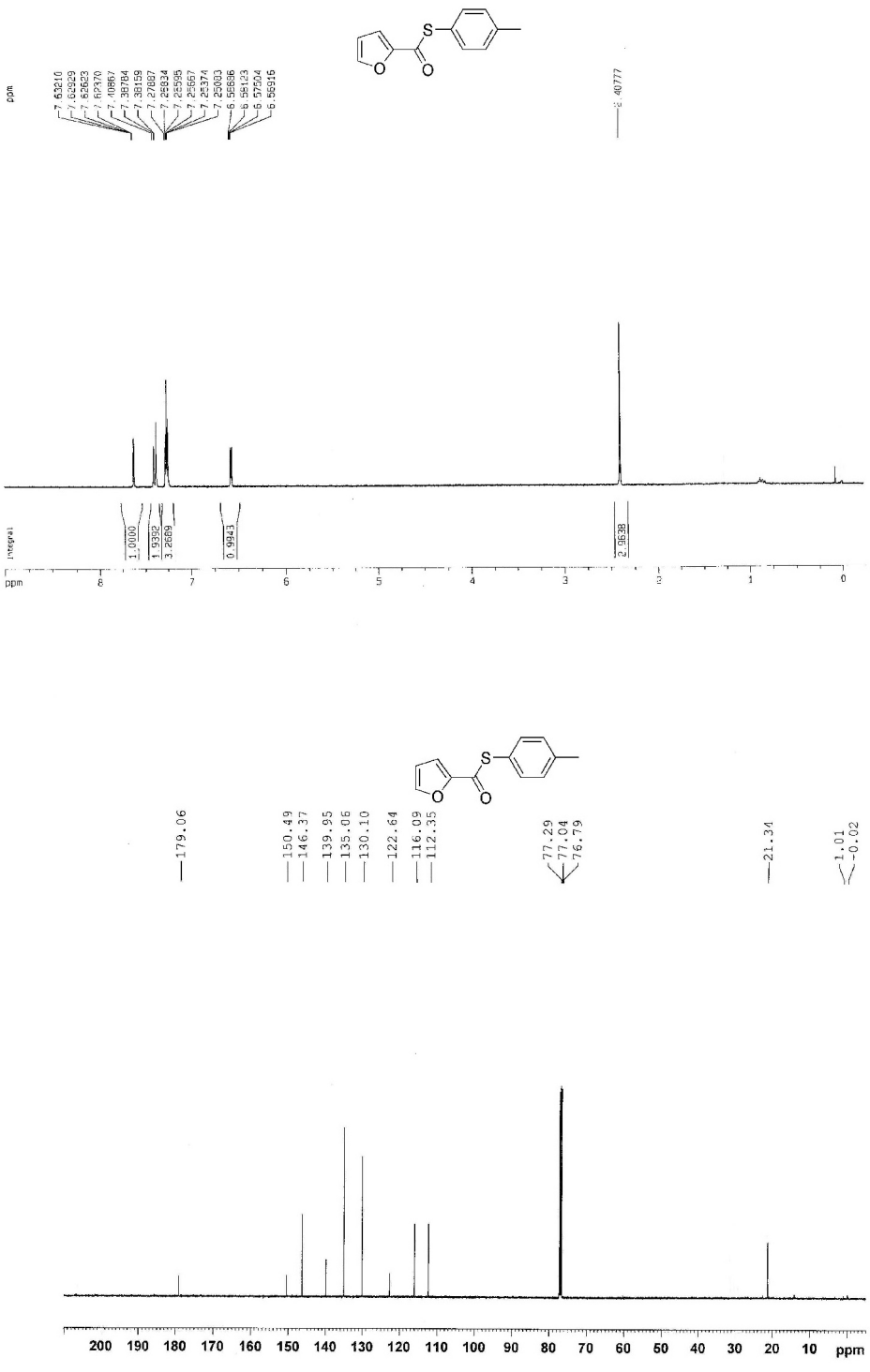

Figure S14. ^{1}H NMR of **3n** (300 MHz, $CDCl_3$) and ^{13}C NMR of **3n** (125 MHz, $CDCl_3$).

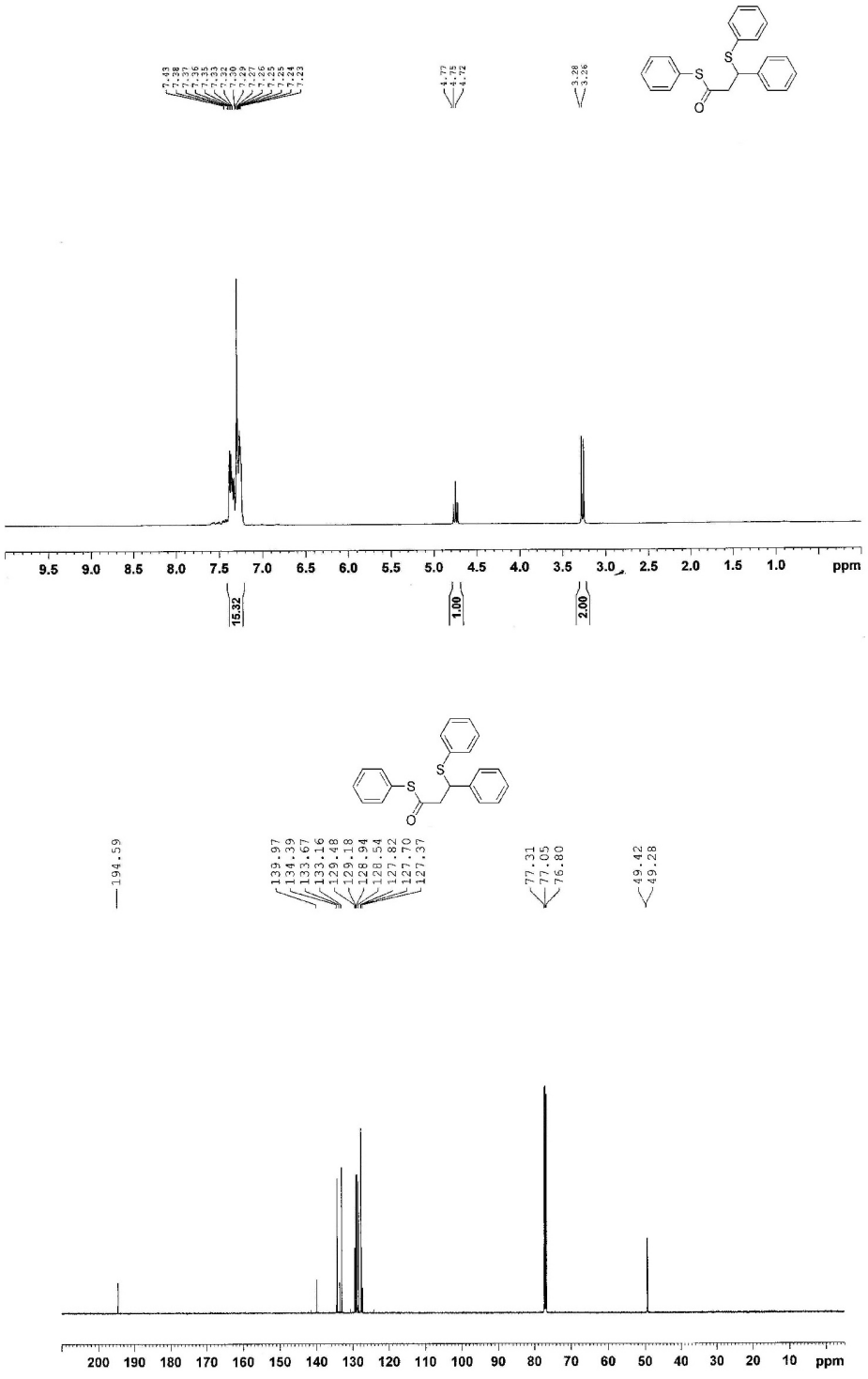

Figure S15. ^{1}H NMR of **4a** (300 MHz, $CDCl_3$) and ^{13}C NMR of **4a** (125 MHz, $CDCl_3$).

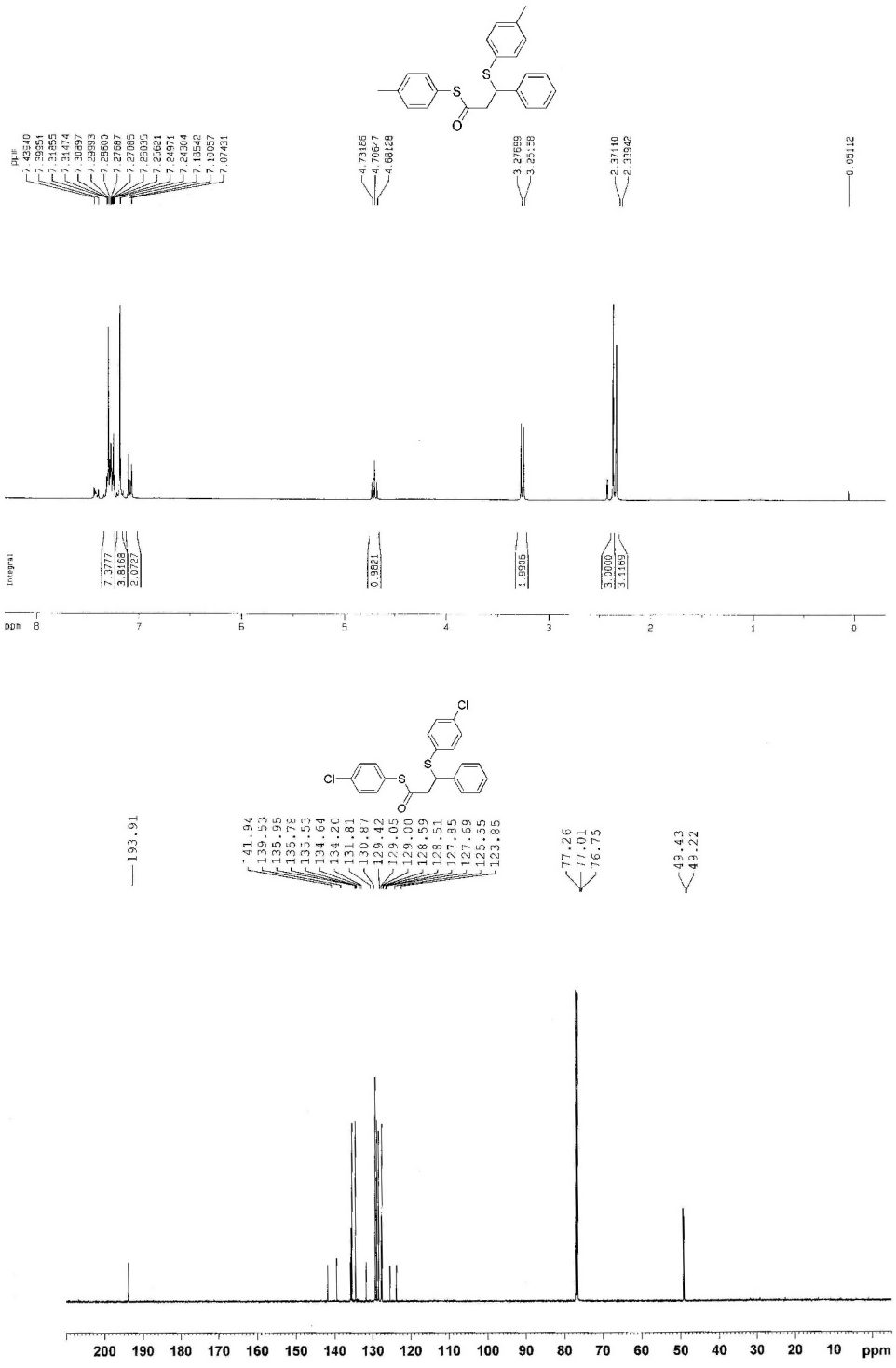

Figure S16. ^{1}H NMR of **4b** (300 MHz, $CDCl_3$) and ^{13}C NMR of **4b** (125 MHz, $CDCl_3$).

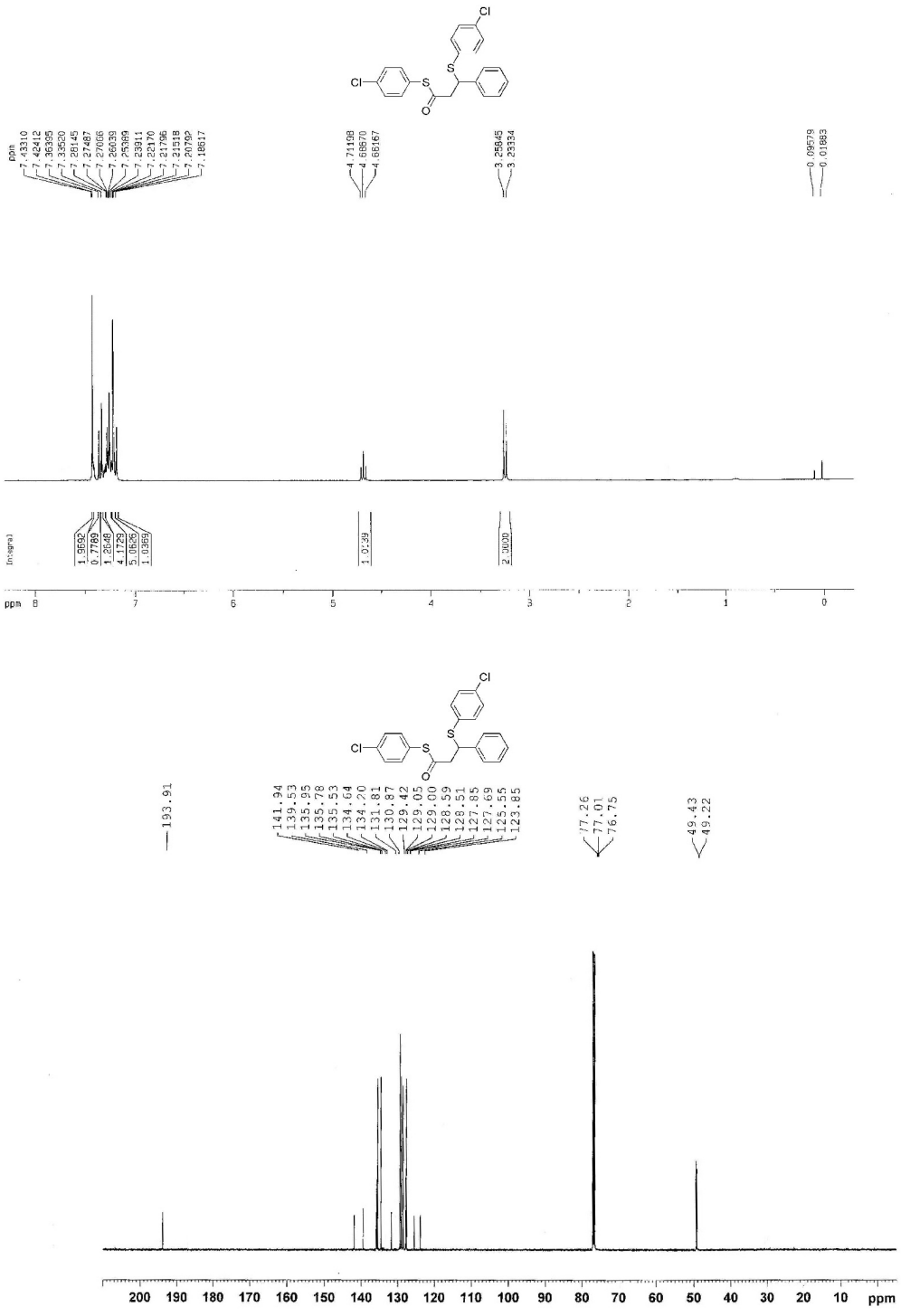

Figure S17. ^{1}H NMR of **4c** (300 MHz, $CDCl_3$) and ^{13}C NMR of **4c** (125 MHz, $CDCl_3$).

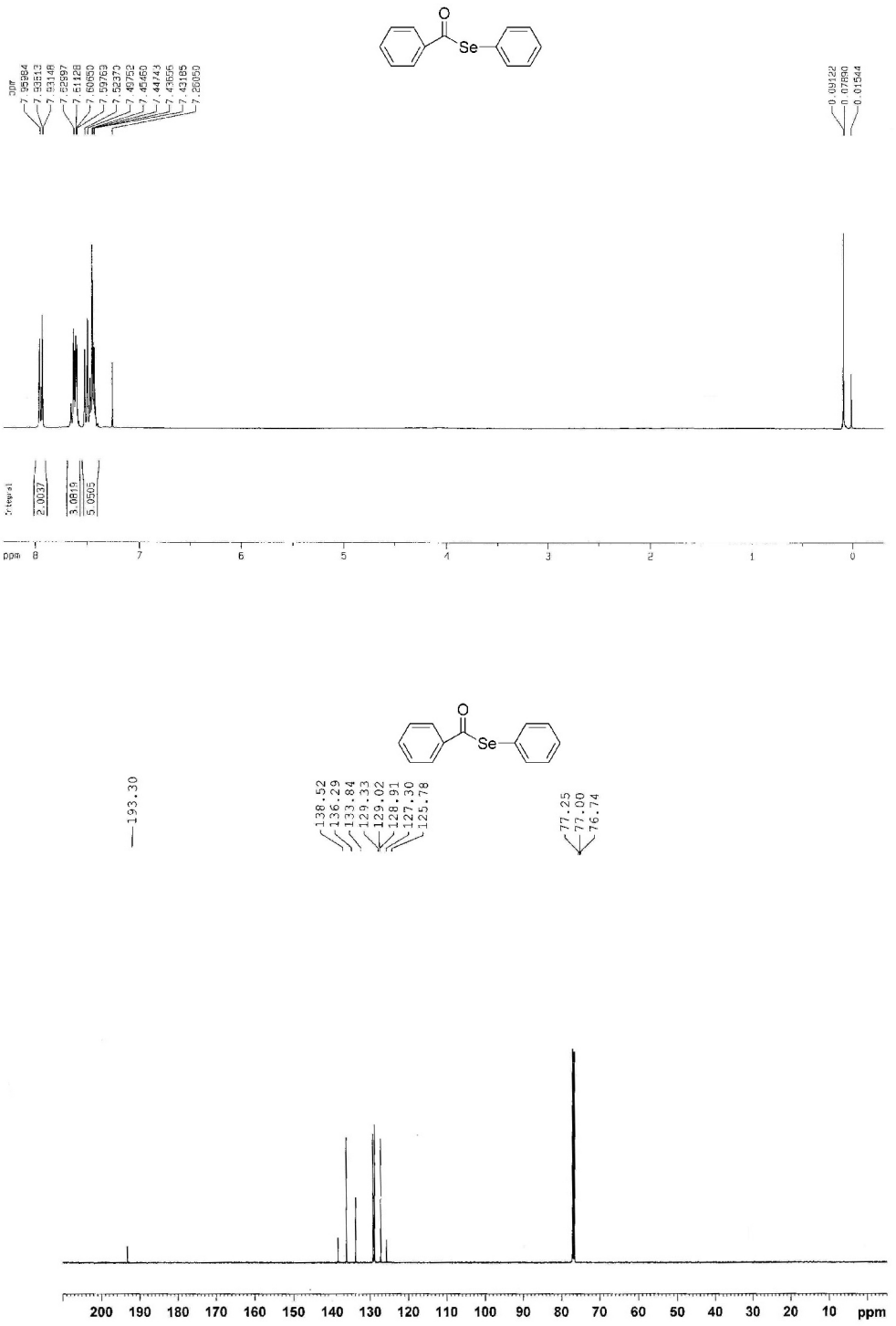

Figure S18. ^{1}H NMR of **5a** (300 MHz, $CDCl_3$) and ^{13}C NMR of **5a** (125 MHz, $CDCl_3$).

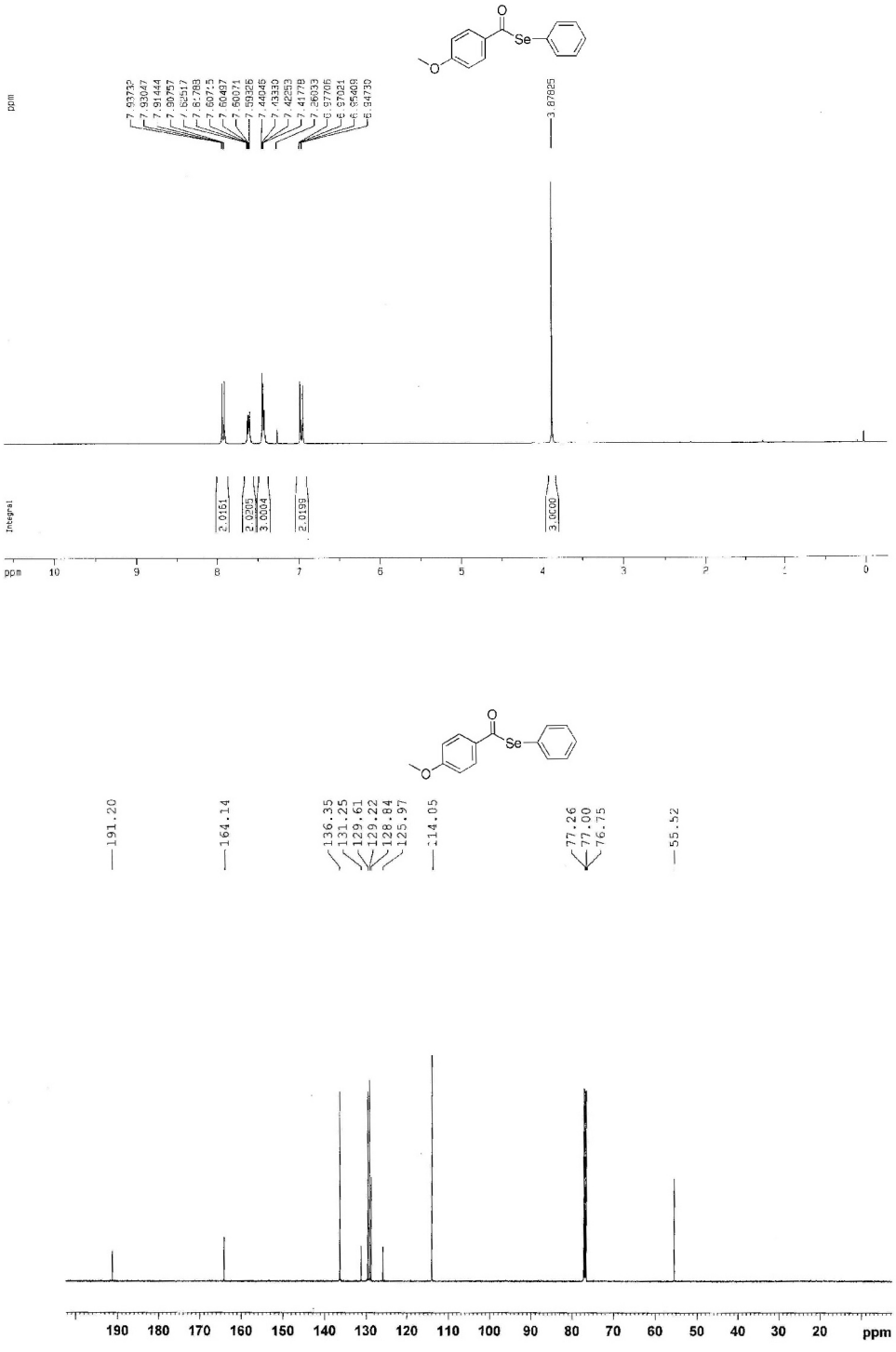

Figure S19. ^{1}H NMR of **5b** (300 MHz, $CDCl_3$) and ^{13}C NMR of **5b** (125 MHz, $CDCl_3$).

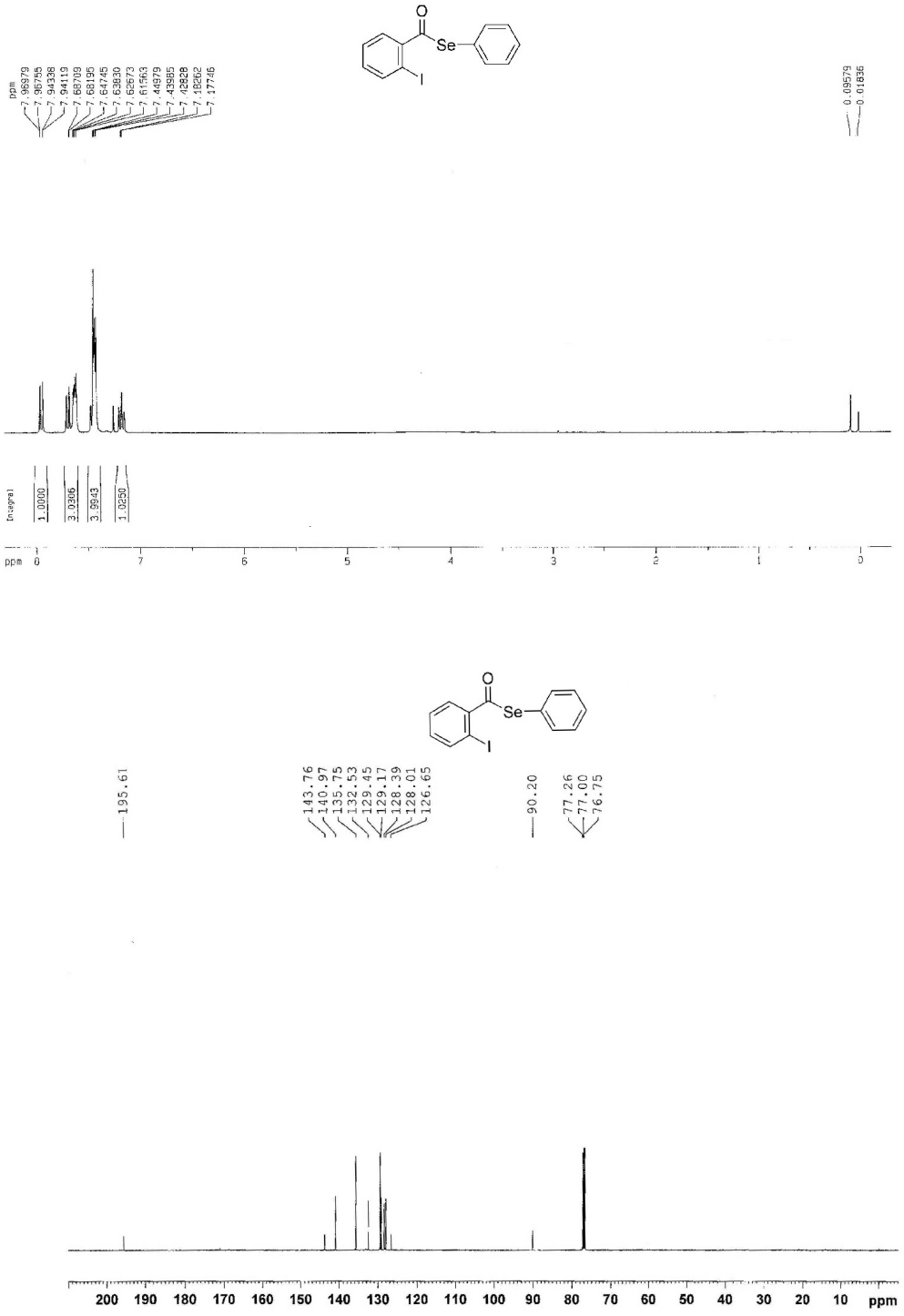

Figure S20. ^{1}H NMR of **5c** (300 MHz, $CDCl_3$) and ^{13}C NMR of **5c** (125 MHz, $CDCl_3$).

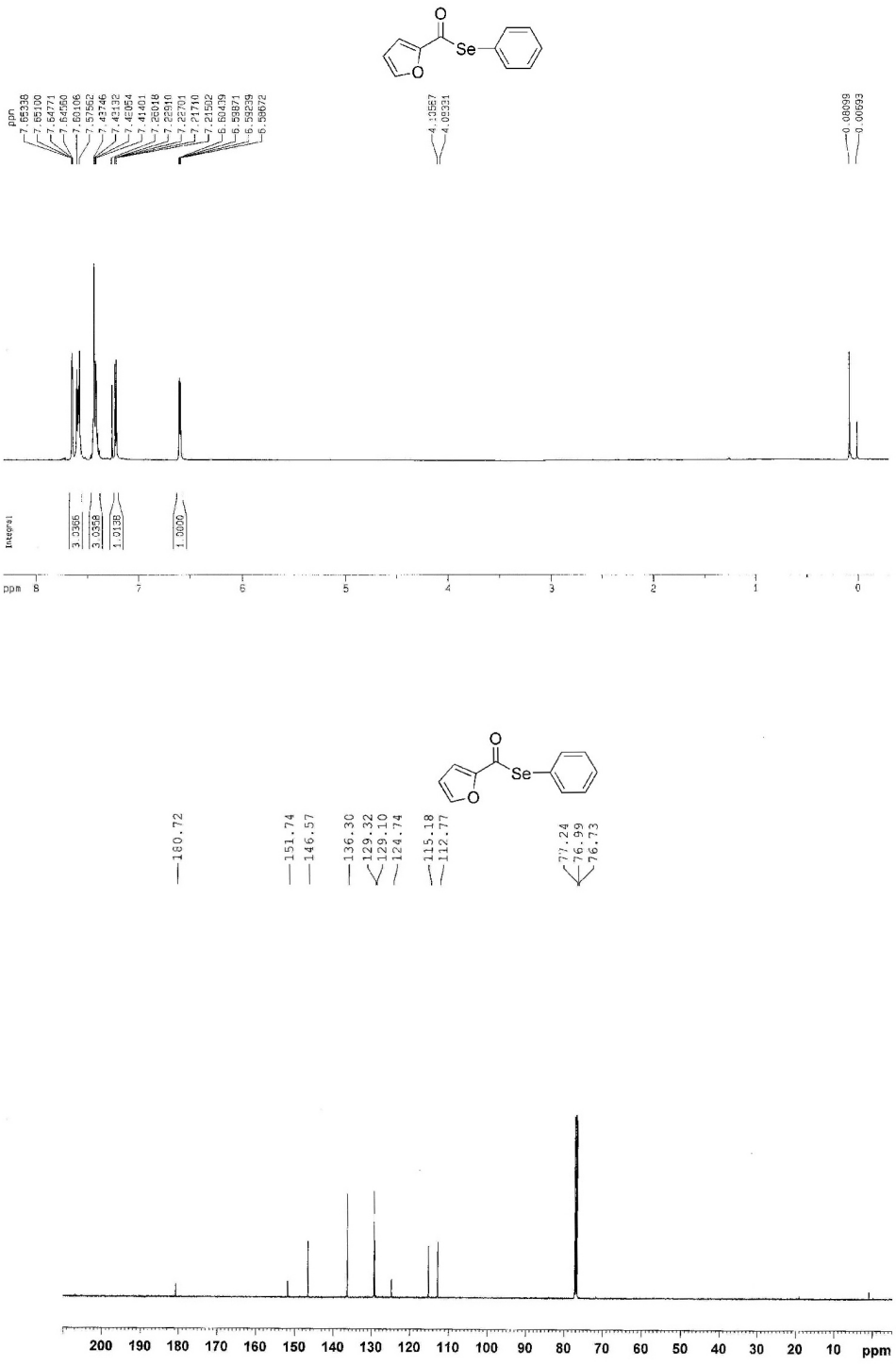

Figure S21. ^{1}H NMR of **5d** (300 MHz, $CDCl_3$) and ^{13}C NMR of **5d** (125 MHz, $CDCl_3$).