

Supporting Information

Palladium-Catalyzed Carbonylation of Thioacetates and Aryl Iodides for the Synthesis of *S*-Aryl Thioesters

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General Experimental Procedure of Carbonylation for the synthesis of thioesters;

S-Aryl thioacetate (2.0 mmol), aryl iodide (2.0 mmol), tris(dibenzylideneacetone)dipalladium(0) (45 mg, 0.05 mmol), 1,4-bis(diphenylphosphino)butane (60 mg, 0.14 mmol), 1,4-diazabicyclo[2.2.2]octane (269 mg, 2.4 mmol), toluene (6.0 mL), and acetone (2.0 mL) were added to the autoclave. The autoclave was closed, purged three times with carbon monoxide, pressurized with 8.8 atm of CO, and then stirred at 110 °C for 6 h. Excess CO was discharged at room temperature. The mixture was extracted with ethyl acetate, and washed with water/brine. The organic layer was dried over magnesium sulfate. Evaporation of the solvent under reduced pressure provided the crude product, which was purified by column chromatography on silica gel.

***S*-Phenyl benzothioate (3aa)¹**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl benzoate (**3aa**) (394 mg, 1.84 mmol, 92% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 52-53 °C [lit. mp 51-53 °C]; ¹H NMR (500 MHz, CDCl₃) δ 8.05–8.01 (m, 2H), 7.62–7.58 (m, 1H), 7.54–7.43 (m, 7H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 190.1, 136.6, 135.1, 133.6, 129.5, 129.2, 128.7, 127.4, 127.3; MS (EI) *m/z* = 214 (M⁺).

***S*-Phenyl 2-methylbenzothioate (3ba)¹**

1-Iodo-2-methylbenzene (**1b**) (436 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 2-methylbenzothioate (**3ba**) (411 mg, 1.80 mmol, 90% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 51-52 °C [lit. mp 46-48 °C]; ¹H NMR (500 MHz, CDCl₃) δ 7.94 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.57–7.49 (m, 2H), 7.49–7.39 (m, 4H), 7.32–7.28 (m, 1H), 7.28–7.26 (m, 1H), 2.49 (s, 3H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 192.2, 137.4, 136.7, 134.9, 132.0, 131.7, 129.5, 129.3, 128.6, 128.2, 125.8, 20.8; MS (EI) *m/z* = 228 (M⁺).

***S*-Phenyl 3-methylbenzothioate (3ca)¹**

1-Iodo-3-methylbenzene (**1c**) (436 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 3-methylbenzothioate (**3ca**) (402 mg, 1.76 mmol, 88% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 97-98 °C [lit. mp 95-97 °C]; ¹H NMR (500 MHz, cdcl₃) δ 7.88–7.78 (m, 2H), 7.55–7.49 (m, 2H), 7.49–7.43 (m, 3H), 7.43–7.39 (m, 1H), 7.39–7.35 (m, 1H), 2.43 (s, 3H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 190.2, 138.6, 136.6, 135.1, 134.4, 129.4, 129.2, 128.6, 127.9, 127.5, 124.7, 21.3; MS (EI) m/z = 228 (M⁺).

***S*-Phenyl 4-methylbenzothioate (3da)¹**

1-Iodo-4-methylbenzene (**1d**) (436 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-methylbenzothioate (**3da**) (443 mg, 1.94 mmol, 97% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 79-80 °C [lit. mp 78-80 °C]; ¹H NMR (500 MHz, CDCl₃) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.54–7.49 (m, 2H), 7.48–7.43 (m, 3H), 7.30–7.26 (m, 2H), 2.43 (s, 3H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 189.7, 144.6, 135.1, 134.1, 129.39, 129.38, 129.2, 127.54, 127.52, 21.7; MS (EI) m/z = 228 (M⁺).

***S*-Phenyl 4-methoxybenzothioate (3ea)¹**

1-Iodo-4-methoxybenzene (**1e**) (468 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-methoxybenzothioate (**3ea**) (479 mg, 1.96 mmol, 98% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 91-92 °C [lit. mp 92-94 °C]; ¹H NMR (600 MHz, CDCl₃) δ 8.01 (d, *J* = 9.1 Hz, 2H), 7.54–7.48 (m, 2H), 7.48–7.42 (m, 3H), 6.95 (d, *J* = 9.0 Hz, 2H), 3.87 (s, 3H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 188.6, 164.0, 135.1, 129.7, 129.4, 129.4, 129.2, 127.6, 113.9, 55.5; MS (EI) m/z = 244 (M⁺).

***S*-Phenyl 4-(trifluoromethyl)benzothioate (3fa)²**

1-Iodo-4-(trifluoromethyl)benzene (**1f**) (544 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**)

(304 mg, 2.0 mmol) afforded *S*-phenyl 4-(trifluoromethyl)benzothioate (**3fa**) (373 mg, 1.32 mmol, 66% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 60-62 °C; ¹H NMR (500 MHz, cdcl₃) δ 8.13 (d, *J* = 8.1 Hz, 2H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.57–7.45 (m, 5H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 189.4, 134.9 (q, *J*_{C-F} = 32.8 Hz), 125.9 (q, *J*_{C-F} = 3.7 Hz), 123.5 (q, *J*_{C-F} = 272.8 Hz); MS (EI) *m/z* = 282 (*M*⁺).

***S*-Phenyl 4-fluorobenzothioate (3ga)¹**

1-Fluoro-4-iodobenzene (**1g**) (444 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-fluorobenzothioate (**3ga**) (288 mg, 1.24 mmol, 62% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 65-67 °C[lit. mp 60-62 °C]; ¹H NMR (500 MHz, CDCl₃) δ 8.09–8.02 (m, 2H), 7.54–7.48 (m, 2H), 7.48–7.43 (m, 3H), 7.19–7.13 (m, 2H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 188.7, 166.1 (d, *J*_{C-F} = 255.4 Hz), 135.1, 133.0 (d, *J*_{C-F} = 3.0 Hz), 130.1 (d, *J*_{C-F} = 9.4 Hz), 129.6, 129.3, 127.1, 115.9 (d, *J*_{C-F} = 22.1 Hz); MS (EI) *m/z* = 232 (*M*⁺).

***S*-Phenyl 3,5-difluorobenzothioate (3ha)**

1,3-Difluoro-5-iodobenzene (**1h**) (450 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 3,5-difluorobenzothioate (**3ha**) (385 mg, 1.54 mmol, 77% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 105-107 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.53–7.44 (m, 7H), 7.03 (tt, *J* = 8.4, 2.3 Hz, 1H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 187.9 (t, *J*_{C-F} = 2.9 Hz), 162.9 (dd, *J*_{C-F} = 251.7, 11.8 Hz), 139.3 (t, *J*_{C-F} = 8.1 Hz), 134.9, 129.9, 129.4, 126.2, 110.5 (dd, *J*_{C-F} = 20.4, 6.7 Hz), 108.8 (t, *J*_{C-F} = 25.3 Hz); HRMS (FD) *m/z* calcd. for C₁₃H₈OF₂S [*M*⁺]: 250.0258, found: 250.0260.

***S*-Phenyl 4-chlorobenzothioate (3ia)¹**

1-Chloro-4-iodobenzene (**1i**) (477 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-chlorobenzothioate (**3ia**) (348 mg, 1.40 mmol, 70% yield); White solid; mp 55-57 °C [lit. mp 56-58 °C]; Eluent for chromatography on silica gel (hexane / ethyl

acetate = 20 / 1); ^1H NMR (500 MHz, CDCl_3) δ 7.96 (d, J = 2.1 Hz, 1H), 7.95 (d, J = 2.1 Hz, 1H), 7.53–7.48 (m, 2H), 7.48–7.42 (m, 5H); $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) δ 189.0, 140.0, 135.0, 134.9, 129.7, 129.3, 129.1, 128.8, 126.9; MS (EI) m/z = 248 (M^+).

***S*-Phenyl 4-acetylbenzothioate (**3ja**)²**

1-(4-Iodophenyl)ethanone (**1j**) (492 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-acetylbenzothioate (**3ja**) (338 mg, 1.32 mmol, 66% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 10 / 1); White solid; mp 109–111 °C; ^1H NMR (500 MHz, CDCl_3) δ 8.10 (d, J = 8.0 Hz, 2H), 8.05 (d, J = 7.9 Hz, 2H), 7.56–7.50 (m, 2H), 7.50–7.42 (m, 3H), 2.65 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) δ 197.3, 189.7, 140.6, 139.9, 135.0, 129.8, 129.4, 128.6, 127.7, 126.7, 26.9; MS (EI) m/z = 256 (M^+).

***S*-Phenyl 4-formylbenzothioate (**3ka**)³**

4-Iodobenzaldehyde (**1k**) (464 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-formylbenzothioate (**3ka**) (330 mg, 1.36 mmol, 68% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 10 / 1); White solid; mp 107–108 °C; ^1H NMR (500 MHz, CDCl_3) δ 10.11 (s, 1H), 8.17 (d, J = 8.2 Hz, 2H), 8.00 (d, J = 8.6 Hz, 2H), 7.56–7.50 (m, 2H), 7.50–7.44 (m, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) δ 191.3, 189.6, 140.9, 139.4, 134.9, 129.9, 129.8, 129.4, 128.0, 126.6; MS (EI) m/z = 242 (M^+).

Methyl 4-((phenylthio)carbonyl)benzoate (3la**)⁴**

Methyl 4-iodobenzoate (**1l**) (524 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded methyl 4-((phenylthio)carbonyl)benzoate (**3la**) (387 mg, 1.42 mmol, 71% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 148–149 °C [lit. mp 146–148 °C]; ^1H NMR (500 MHz, CDCl_3) δ 8.14 (d, J = 8.4 Hz, 2H), 8.07 (d, J = 8.6 Hz, 2H), 7.55–7.49 (m, 2H), 7.49–7.42 (m, 3H), 3.95 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) δ 189.6, 166.0, 139.9, 135.0, 134.4, 129.9, 129.7, 129.3, 127.4, 126.8, 52.5; MS (EI) m/z = 272 (M^+).

***S*-Phenyl 4-cyanobenzothioate (3ma)³**

4-Iodobenzonitrile (**1m**) (458 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-cyanobenzothioate (**3ma**) (369 mg, 1.54 mmol, 77% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 10 / 1); White solid; mp 125-127 °C [lit. mp 127-129 °C]; ¹H NMR (500 MHz, CDCl₃) δ 8.11 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.54–7.46 (m, 5H); ¹³C {¹H} NMR (126 MHz, CDCl₃) δ 189.1, 139.8, 134.9, 132.6, 130.0, 129.5, 127.9, 126.2, 117.8, 116.9; MS (EI) *m/z* = 239 (M⁺).

***S*-Phenyl 4-nitrobenzothioate (3na)²**

1-Iodo-4-nitrobenzene (**1n**) (489 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl 4-nitrobenzothioate (**3na**) (280 mg, 1.08 mmol, 54% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 154-155 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.34 (d, *J* = 9.0 Hz, 2H), 8.18 (d, *J* = 9.0 Hz, 2H), 7.55–7.46 (m, 5H); ¹³C {¹H} NMR (126 MHz, CDCl₃) δ 188.9, 150.7, 141.3, 134.9, 130.1, 129.5, 128.5, 126.2, 124.0; MS (EI) *m/z* = 259 (M⁺).

***S*-Phenyl naphthalene-1-carbothioate (3oa)⁵**

1-Iodonaphthalene (**1o**) (508 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl naphthalene-1-carbothioate (**3oa**) (444 mg, 1.68 mmol, 84% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 40 / 1); White solid; mp 50-52 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.54 (d, *J* = 7.6 Hz, 1H), 8.21 (dd, *J* = 7.2, 1.2 Hz, 1H), 8.02 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.61–7.45 (m, 8H); ¹³C {¹H} NMR (126 MHz, CDCl₃) δ 192.1, 134.9, 134.6, 133.8, 133.2, 129.5, 129.3, 129.3, 128.3, 128.3, 128.1, 127.9, 126.7, 125.3, 124.4; MS (EI) *m/z* = 264 (M⁺).

***S*-Phenyl pyridine-3-carbothioate (3pa)**

3-Iodopyridine (**1p**) (410 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl pyridine-3-carbothioate (**3pa**) (379 mg, 1.76 mmol, 88% yield); Eluent for

chromatography on silica gel (hexane / ethyl acetate = 2 / 1); White solid; mp 44-45 °C; ¹H NMR (500 MHz, CDCl₃) δ 9.25 (d, *J* = 1.7 Hz, 1H), 8.83 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.27 (ddd, *J* = 8.0, 2.2, 1.7 Hz, 1H), 7.56–7.46 (m, 5H), 7.45 (ddd, *J* = 8.0, 4.9, 0.8 Hz, 1H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 188.9, 154.0, 148.7, 135.0, 134.8, 132.3, 129.9, 129.4, 126.3, 123.6; HRMS (FD) *m/z* calcd. for C₁₂H₉NOS [M⁺]: 215.0399, found: 215.0401.

***S*-Phenyl thiophene-2-carbothioate (3qa)¹**

2-Iodothiophene (**1q**) (420 mg, 2.0 mmol), and *S*-phenyl thioacetate (**2a**) (304 mg, 2.0 mmol) afforded *S*-phenyl thiophene-2-carbothioate (**3qa**) (392 mg, 1.78 mmol, 89% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 54-55 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.90 (dd, *J* = 3.8, 1.2 Hz, 1H), 7.64 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.55–7.49 (m, 2H), 7.49–7.37 (m, 3H), 7.14 (dd, *J* = 5.0, 3.8 Hz, 1H); ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 182.0, 141.4, 135.1, 133.2, 131.6, 129.7, 129.3, 128.0, 126.9; MS (EI) *m/z* = 220 (M⁺).

***S*-*o*-Tolyl benzothioate (3ab)¹**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-*o*-tolyl ethanethioate (**2b**) (332 mg, 2.0 mmol) afforded *S*-*o*-tolyl benzothioate (**3ab**) (420 mg, 1.84 mmol, 92% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 8.12–7.98 (m, 2H), 7.64–7.56 (m, 1H), 7.54–7.43 (m, 3H), 7.42–7.32 (m, 2H), 7.28–7.22 (m, 1H), 2.40 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 189.6, 142.6, 136.7, 136.4, 133.6, 130.8, 130.2, 128.7, 127.5, 126.8, 126.7, 20.8; MS (EI) *m/z* = 228 (M⁺).

***S*-*m*-Tolyl benzothioate (3ac)⁶**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-*m*-tolyl ethanethioate (**2c**) (332 mg, 2.0 mmol) afforded *S*-*m*-tolyl benzothioate (**3ac**) (429 mg, 1.88 mmol, 94% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 46-47 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (dt, *J* = 8.5, 1.6 Hz, 2H), 7.62–7.55 (m, 1H), 7.50–7.44 (m,

2H), 7.38–7.28 (m, 3H), 7.28–7.23 (m, 1H), 2.39 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 190.4, 139.1, 136.7, 135.6, 133.6, 132.1, 130.4, 129.1, 128.7, 127.5, 126.9, 21.3; MS (EI) m/z = 228 (M^+).

***S*-*p*-Tolyl benzothioate (3ad)¹**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-*p*-tolyl ethanethioate (**2d**) (332 mg, 2.0 mmol) afforded *S*-*p*-tolyl benzothioate (**3ad**) (438 mg, 1.92 mmol, 96% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 78-80 °C [lit. mp 75-77 °C]; ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, J = 7.4 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H), 2.39 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 190.6, 139.8, 136.6, 135.0, 133.6, 130.1, 128.7, 127.4, 123.7, 21.4; MS (EI) m/z = 228 (M^+).

***S*-(4-Methoxyphenyl) benzothioate (3ae)¹**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-(4-methoxyphenyl) ethanethioate (**2e**) (364 mg, 2.0 mmol) afforded *S*-(4-methoxyphenyl) benzothioate (**3ae**) (464 mg, 1.90 mmol, 95% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 10 / 1); White solid; mp 97-99 °C [lit. mp 92-94 °C]; ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, J = 7.2 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.7 Hz, 2H), 7.42 (d, J = 8.9 Hz, 2H), 6.99 (d, J = 8.9 Hz, 2H), 3.84 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 191.1, 160.8, 136.6, 133.6, 128.7, 127.5, 117.8, 115.0, 55.4; MS (EI) m/z = 244 (M^+).

***S*-(4-Fluorophenyl) benzothioate (3ag)⁵**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-(4-fluorophenyl) ethanethioate (**2g**) (340 mg, 2.0 mmol) afforded *S*-(4-fluorophenyl) benzothioate (**3ag**) (381 mg, 1.64 mmol, 82% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 51-52 °C; ^1H NMR (500 MHz, CDCl_3) δ 8.06–7.98 (m, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.54–7.44 (m, 4H), 7.19–7.12 (m, 2H); $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) δ 190.1, 163.6 (d, $J_{\text{C-F}}$ = 250.1

Hz), 137.2 (d, $J = 8.7$ Hz), 136.4, 133.8, 128.8, 127.5, 122.6 (d, $J_{\text{C-F}} = 3.5$ Hz), 116.6 (d, $J_{\text{C-F}} = 22.1$ Hz); MS (EI) $m/z = 232$ (M^+).

***S*-(4-Chlorophenyl) benzothioate (3ai)⁵**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-(4chlorophenyl) ethanethioate (**2i**) (373 mg, 2.0 mmol) afforded *S*-(4-chlorophenyl) benzothioate (**3ai**) (428 mg, 1.72 mmol, 86% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 65-66 °C [lit. mp 64-66 °C]; ^1H NMR (400 MHz, CDCl_3) δ 8.05–7.96 (m, 2H), 7.61 (t, $J = 7.4$ Hz, 1H), 7.48 (t, $J = 7.7$ Hz, 2H), 7.45–7.39 (m, 4H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 189.6, 136.3, 136.0, 133.9, 129.5, 128.8, 127.5, 125.8; MS (EI) $m/z = 248$ (M^+).

***S*-Naphthalen-1-yl benzothioate (3aj)⁷**

Iodobenzene (**1a**) (408 mg, 2.0 mmol) and *S*-naphthalen-1-yl ethanethioate (**2j**) (405 mg, 2.0 mmol) afforded *S*-naphthalen-1-yl benzothioate (**3aj**) (481 mg, 1.82 mmol, 91% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 116-118 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.28–8.19 (m, 1H), 8.11 (d, $J = 8.3$ Hz, 2H), 7.97 (d, $J = 8.3$ Hz, 1H), 7.93–7.86 (m, 1H), 7.78 (dd, $J = 7.1, 1.2$ Hz, 1H), 7.61 (t, $J = 7.4$ Hz, 1H), 7.56 7.46 (m, 5H); $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 189.7, 136.7, 135.5, 134.6, 134.2, 133.7, 131.0, 128.8, 128.7, 127.7, 127.2, 126.4, 125.6, 125.4, 124.8; MS (EI) $m/z = 264$ (M^+).

General experimental procedure for the synthesis of *S*-aryl thioacetates from palladium-catalyzed coupling of KSAc and aryl iodides;

Aryl iodide (2.0 mmol), potassium thioacetate (228 mg, 2.0 mmol), tris(dibenzylideneacetone)dipalladium(0) (45 mg, 0.05 mmol), 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene (81 mg, 0.14 mmol), toluene (6.0 mL) and acetone (2.0 mL) were added to the reaction vial. The mixture was stirred at 70 °C for 6 h. The mixture was extracted with ethyl acetate, and washed with water/brine. The organic layer was dried over magnesium sulfate. Evaporation of the solvent under reduced pressure provided the crude product, which

was purified by column chromatography on silica gel (eluent: hexane / ethyl acetate = 20 / 1).

***S*-Phenyl thioacetate (**2a**)⁸**

Potassium thioacetate (228 mg, 2.0 mmol) and iodobenzene (**1a**) (408 mg, 2.0 mmol) afforded *S*-phenyl thioacetate (**2a**) (264 mg, 1.74 mmol, 87% yield); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.47–7.33 (m, 5H), 2.40 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 194.0, 134.4, 129.4, 129.2, 128.0, 30.2; MS (EI) m/z = 152 (M⁺).

***S*-*o*-Tolyl ethanethioate (**2b**)⁸**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-2-methylbenzene (**1b**) (436 mg, 2.0 mmol) afforded *S*-*o*-tolyl ethanethioate (**2b**) (273 mg, 1.64 mmol, 82% yield); Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, *J* = 7.4 Hz, 1H), 7.36–7.26 (m, 2H), 7.23–7.19 (m, 1H), 2.41 (s, 3H), 2.35 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 193.7, 142.0, 135.9, 130.8, 130.2, 127.4, 126.6, 30.2, 20.7; MS (EI) m/z = 166 (M⁺).

***S*-*m*-Tolyl ethanethioate (**2c**)**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-3-methylbenzene (**1c**) (436 mg, 2.0 mmol) afforded *S*-*m*-tolyl ethanethioate (**2c**) (266 mg, 1.60 mmol, 80% yield); Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.34–7.27 (m, 1H), 7.26–7.18 (m, 3H), 2.41 (s, 3H), 2.36 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 194.4, 139.1, 135.0, 131.5, 130.3, 129.0, 127.6, 30.2, 21.3; HRMS (FD) m/z calcd. for C₉H₁₀OS [M⁺]: 166.0447, found: 166.0450.

***S*-*p*-Tolyl ethanethioate (**2d**)⁹**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-4-methylbenzene (**1d**) (436 mg, 2.0 mmol) afforded *S*-*p*-tolyl ethanethioate (**2d**) (299 mg, 1.80 mmol, 90% yield); Orange oil; ¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 8.1 Hz, 2H), 2.40 (s, 3H), 2.37 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 194.6, 139.7, 134.4, 130.1, 124.4, 30.1, 21.3; MS (EI) m/z = 166 (M⁺).

***S*-(4-Methoxyphenyl) ethanethioate (**2e**)¹**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-4-methoxybenzene (**1e**) (468 mg, 2.0 mmol) afforded *S*-(4-methoxyphenyl) ethanethioate (**2e**) (306 mg, 1.68 mmol, 84% yield); Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 3.81 (s, 3H), 2.38 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 195.2, 160.6, 136.1, 118.6, 114.9, 55.3, 29.9; MS (EI) *m/z* = 182 (*M*⁺).

***S*-(4-Fluorophenyl) ethanethioate (**2g**)⁸**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-fluoro-4-iodobenzene (**1g**) (444 mg, 2.0 mmol) afforded *S*-(4-fluorophenyl) ethanethioate (**2g**) (242 mg, 1.42 mmol, 71% yield); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.38 (dd, *J* = 8.9, 5.3 Hz, 2H), 7.10 (t, *J* = 8.7 Hz, 2H), 2.41 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 194.0, 163.5 (d, *J*_{C-F} = 250.1 Hz), 136.5 (d, *J*_{C-F} = 8.6 Hz), 123.2 (d, *J*_{C-F} = 3.5 Hz), 116.5 (d, *J*_{C-F} = 22.1 Hz), 30.1; MS (EI) *m/z* = 170 (*M*⁺).

***S*-(4-Chlorophenyl) ethanethioate (**2l**)⁸**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-chloro-4-iodobenzene (**1l**) (477 mg, 2.0 mmol) afforded *S*-(4-chlorophenyl) ethanethioate (**2l**) (287 mg, 1.54 mmol, 77% yield); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 8.7 Hz, 2H), 7.32 (d, *J* = 8.7 Hz, 2H), 2.41 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 193.4, 135.8, 135.7, 129.4, 126.3, 30.2; MS (EI) *m/z* = 186 (*M*⁺).

***S*-Naphthalen-1-yl ethanethioate (**2o**)⁹**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodonaphthalene (**1o**) (508 mg, 2.0 mmol), afforded *S*-naphthalene-1-yl ethanethioate (**2o**) (328 mg, 1.62 mmol, 81% yield); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 8.11 (d, *J* = 8.1 Hz, 1H), 7.86 (d, *J* = 8.3 Hz, 1H), 7.80 (dd, *J* = 8.2, 1.1 Hz, 1H), 7.62 (dd, *J* = 7.1, 1.2 Hz, 1H), 7.51–7.38 (m, 3H), 2.39 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 194.1, 135.0, 134.2, 131.0, 128.7, 127.2, 126.5, 125.6, 125.5, 125.3, 30.3; MS (EI) *m/z* = 202 (*M*⁺).

General experimental procedure tandem reaction for the synthesis of *S*-aryl thioesters;

Potassium thioacetate (228 mg, 2.0 mmol), aryl iodide (4.0 mmol), tris(dibenzylideneacetone)dipalladium(0) (45 mg, 0.05 mmol), 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene (81 mg, 0.14 mmol), toluene (6.0 mL), and acetone (2.0 mL) were added to the reaction vial. The mixture was stirred at 70 °C for 6 h. The mixture was transferred to an autoclave, followed by the addition of 1,4-bis(diphenylphosphino)butane (119 mg, 0.14 mmol), and 1,4-diazabicyclo[2.2.2]octane (337 mg, 3.0 mmol). The autoclave was closed, purged three times with carbon monoxide, pressurized with 8.8 atm of CO, and then stirred at 110 °C for 6 h. Excess CO was discharged at room temperature. The mixture was extracted with ethyl acetate, and washed with water/brine. The organic layer was dried over magnesium sulfate. Evaporation of the solvent under reduced pressure provided the crude product, which was purified by column chromatography on silica gel.

***S*-*o*-Tolyl 2-methylbenzothioate (**3bb**)¹**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-2-methyl benzene (**1b**) (872 mg, 4.0 mmol) afforded *S*-*o*-tolyl 2-methylbenzothioate (**3bb**) (290 mg, 1.20 mmol, 60% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 60-61 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.88–7.76 (m, 2H), 7.43–7.30 (m, 5H), 7.27–7.23 (m, 1H), 2.43 (s, 3H), 2.40 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 191.9, 142.4, 137.3, 137.0, 136.3, 131.9, 131.7, 130.8, 130.2, 128.6, 127.6, 126.7, 125.9, 20.8, 20.7; MS (EI) m/z = 242 (M⁺).

***S*-*m*-Tolyl 3-methylbenzothioate (**3cc**)**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-3-methylbenzene (**1c**) (872 mg, 4.0 mmol) afforded *S*-*m*-tolyl 3-methylbenzothioate (**3cc**) (262 mg, 1.08 mmol, 54% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.97 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.42 (td, *J* = 7.5, 1.4 Hz, 1H), 7.39–7.22 (m, 5H), 2.49 (s, 3H), 2.43 (s, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ

190.6, 139.1, 138.6, 136.7, 135.7, 134.4, 132.1, 130.4, 129.1, 128.6, 127.9, 127.1, 124.7, 21.4, 21.3; HRMS (FD) m/z calcd. for $C_{15}H_{14}OS$ [M^+]: 242.0760, found: 242.0760.

***S-p*-Tolyl 4-methylbenzothioate (3dd)¹**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-4-methylbenzene (**1d**) (872 mg, 4.0 mmol) afforded *S-p*-tolyl 4-methylbenzothioate (**3dd**) (310 mg, 1.28 mmol, 64% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 121-123 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H), 7.28–7.23 (m, 4H), 2.42 (s, 3H), 2.40 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 190.2, 144.5, 139.7, 135.1, 134.1, 130.1, 129.4, 127.5, 123.9, 21.7, 21.4; MS (EI) m/z = 242 (M^+).

***S*-(4-Methoxyphenyl) 4-methoxybenzothioate (3ee)¹⁰**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodo-4-methoxybenzene (**1e**) (936 mg, 4.0 mmol) afforded *S*-(4-methoxyphenyl) 4-methoxybenzothioate (**3ee**) (280 mg, 1.02 mmol, 51% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 4 / 1); White solid; mp 142-144 °C [lit. mp 136-137 °C]; 1H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, J = 9.0 Hz, 2H), 7.41 (d, J = 8.9 Hz, 2H), 6.98 (d, J = 8.9 Hz, 2H), 6.95 (d, J = 9.0 Hz, 2H), 3.88 (s, 3H), 3.84 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 189.5, 163.9, 160.7, 136.7, 129.7, 129.4, 118.2, 114.9, 113.9, 55.5, 55.4; MS (EI) m/z = 274 (M^+).

***S*-(4-Fluorophenyl) 4-fluorobenzothioate (3gg)**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-fluoro-4-iodobenzene (**1g**) (888 mg, 4.0 mmol) afforded *S*-(4-fluorophenyl) 4-fluorobenzothioate (**3gg**) (250 mg, 1.00 mmol, 50% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 91-92 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, J = 8.9, 5.3 Hz, 2H), 7.48 (dd, J = 8.9, 5.2 Hz, 2H), 7.22–7.11 (m, 4H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 188.7, 166.2 (d, J_{C-F} = 249.6 Hz), 163.7 (d, J_{C-F} = 244.3 Hz), 137.2 (d, J_{C-F} = 8.6 Hz), 132.7 (d, J_{C-F} = 3.0 Hz), 130.1 (d, J_{C-F} = 9.4 Hz), 122.3 (d, J_{C-F} = 3.5 Hz), 116.6 (d, J_{C-F} = 22.2 Hz), 116.0 (d, J_{C-F} = 22.1 Hz);

HRMS (FD) m/z calcd. for $C_{13}H_8F_2OS$ [M^+]: 250.0258, found: 250.0260.

***S*-(4-Chlorophenyl) 4-chlorobenzothioate (3ii)⁶**

Potassium thioacetate (228 mg, 2.0 mmol) and 1-chloro-4-iodobenzene (**1i**) (954 mg, 4.0 mmol) afforded *S*-(4-chlorophenyl) 4-chlorobenzothioate (**3ii**) (306 mg, 1.08 mmol, 54% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 20 / 1); White solid; mp 135-137 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.7 Hz, 2H), 7.47 (d, J = 8.8 Hz, 2H), 7.45–7.39 (m, 4H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 188.6, 140.3, 136.3, 136.2, 134.7, 129.6, 129.2, 128.9, 125.4; MS (EI) m/z = 282 (M^+).

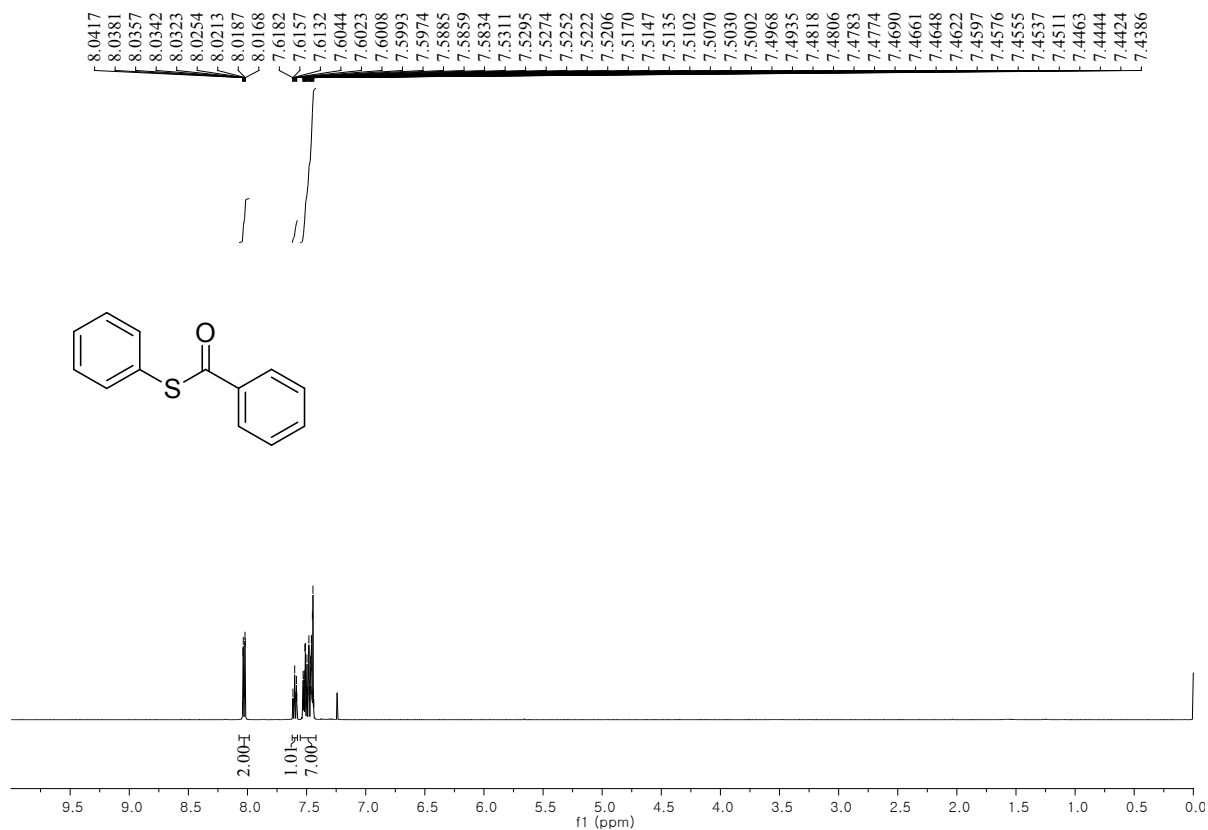
***S*-Naphthalen-1-yl naphthalene-1-carbothioate (3oo)**

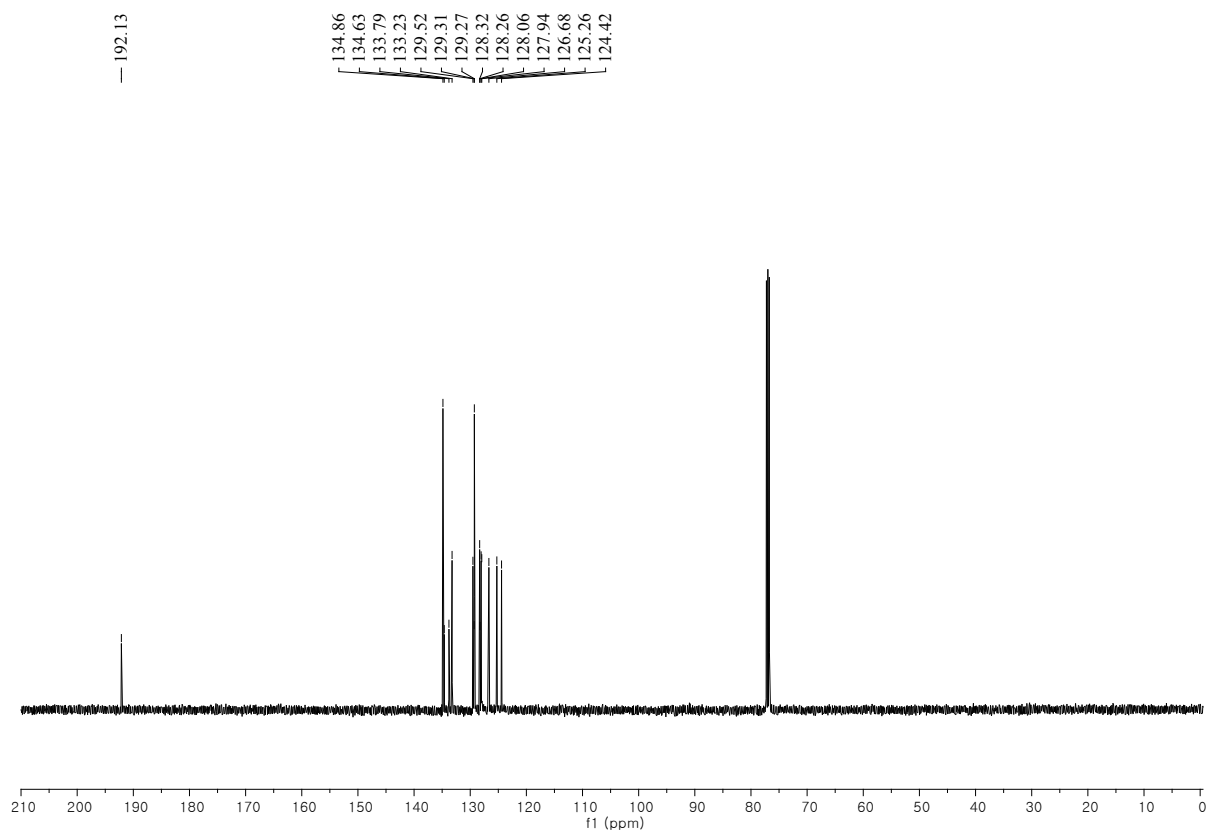
Potassium thioacetate (228 mg, 2.0 mmol) and 1-iodonaphthalene (**1o**) (1,016 mg, 4.0 mmol) afforded *S*-naphthalene-1-yl naphthalene-1-carbothioate (**3oo**) (352 mg, 1.12 mmol, 56% yield); Eluent for chromatography on silica gel (hexane / ethyl acetate = 10 / 1); White solid; mp 144-146 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.55–8.48 (m, 1H), 8.41 (dd, J = 7.2, 1.2 Hz, 1H), 8.38–8.29 (m, 1H), 8.06 (d, J = 8.3 Hz, 1H), 8.01 (d, J = 8.3 Hz, 1H), 7.96–7.84 (m, 3H), 7.62–7.51 (m, 6H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 191.8, 135.4, 134.7, 134.5, 134.3, 133.9, 133.4, 131.1, 129.4, 128.8, 128.4, 128.2, 128.2, 127.3, 126.7, 126.5, 125.7, 125.3, 124.5; HRMS (FD) m/z calcd. for $C_{21}H_{14}OS$ [M^+]: 314.0760, found: 314.0764.

Reference

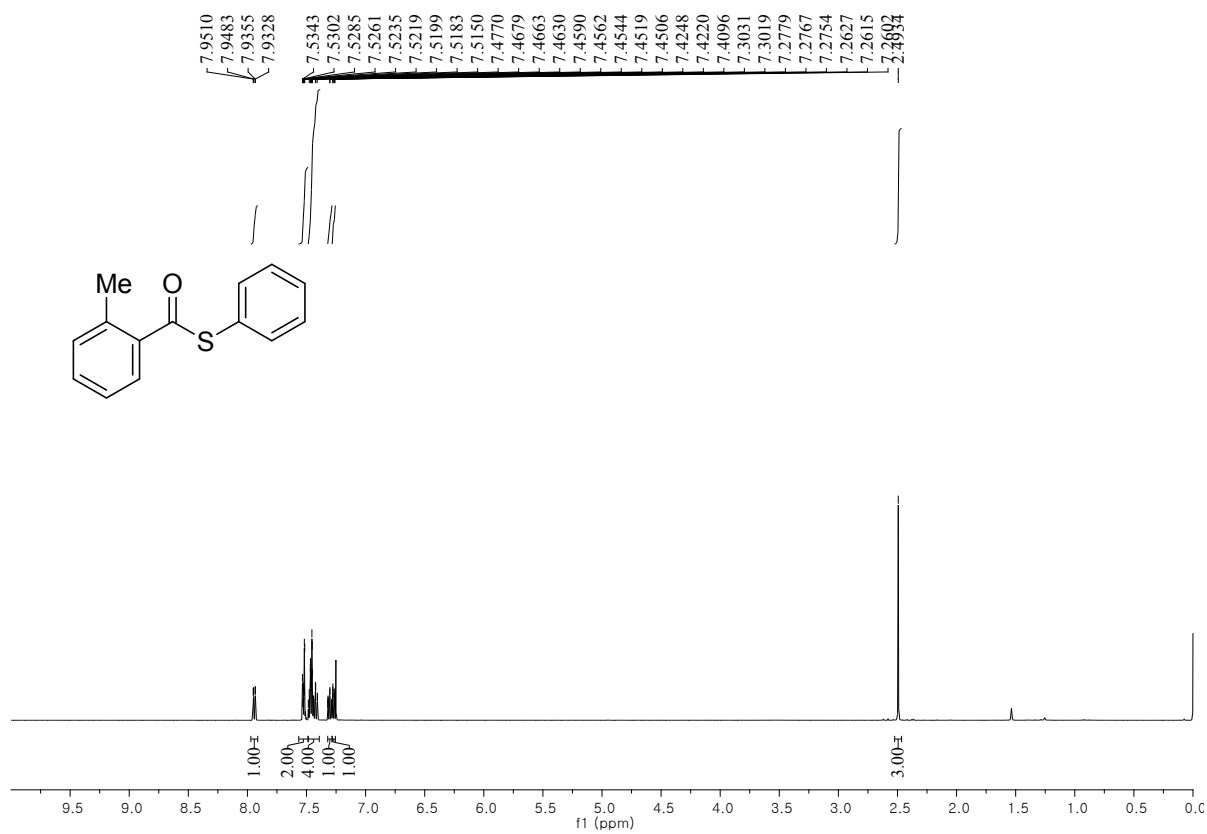
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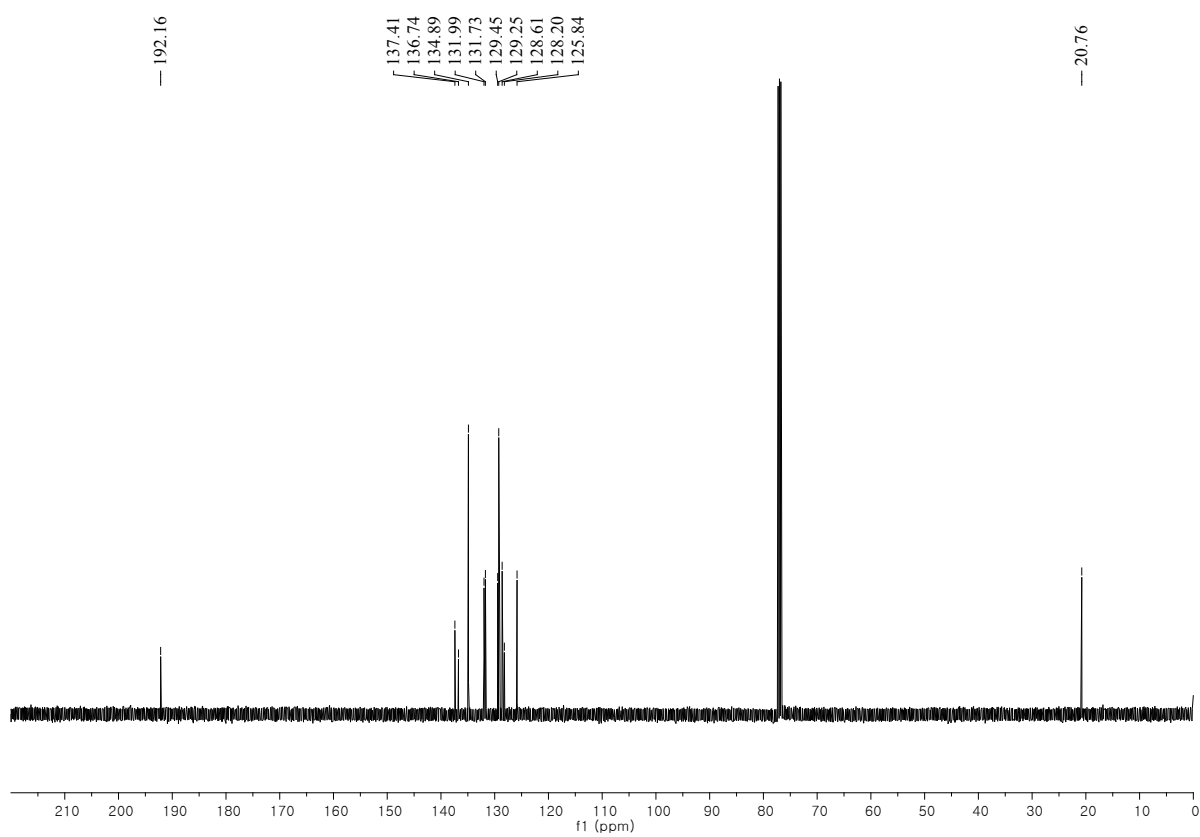
***S*-Phenyl benzothioate (3aa)**



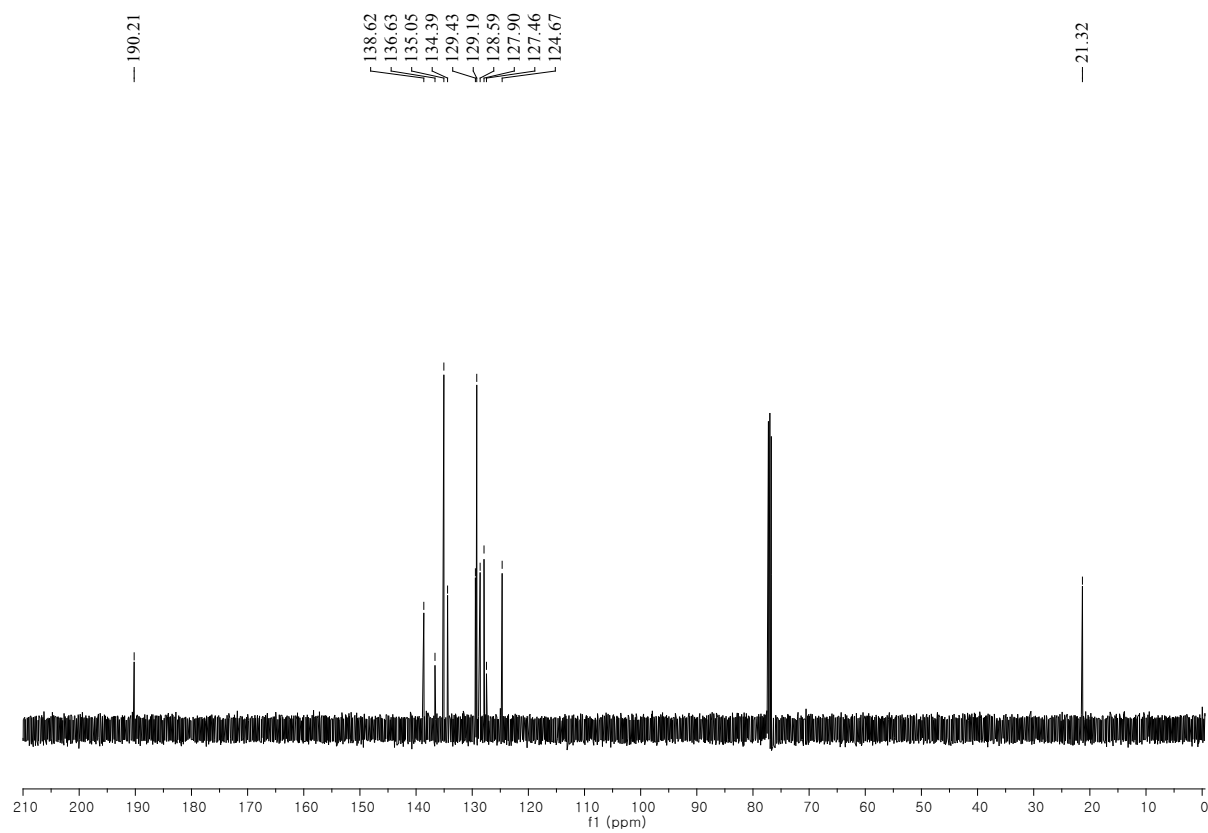
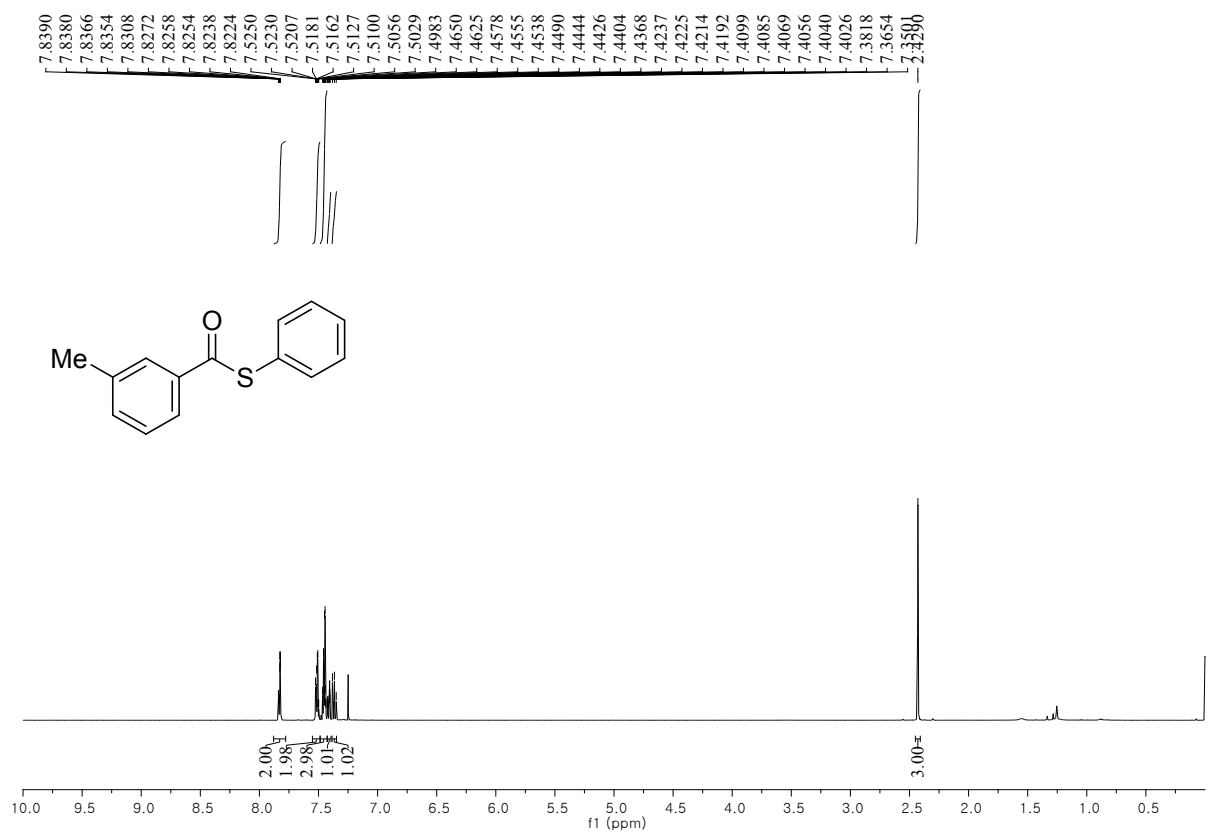


S-Phenyl 2-methylbenzothioate (3ba)

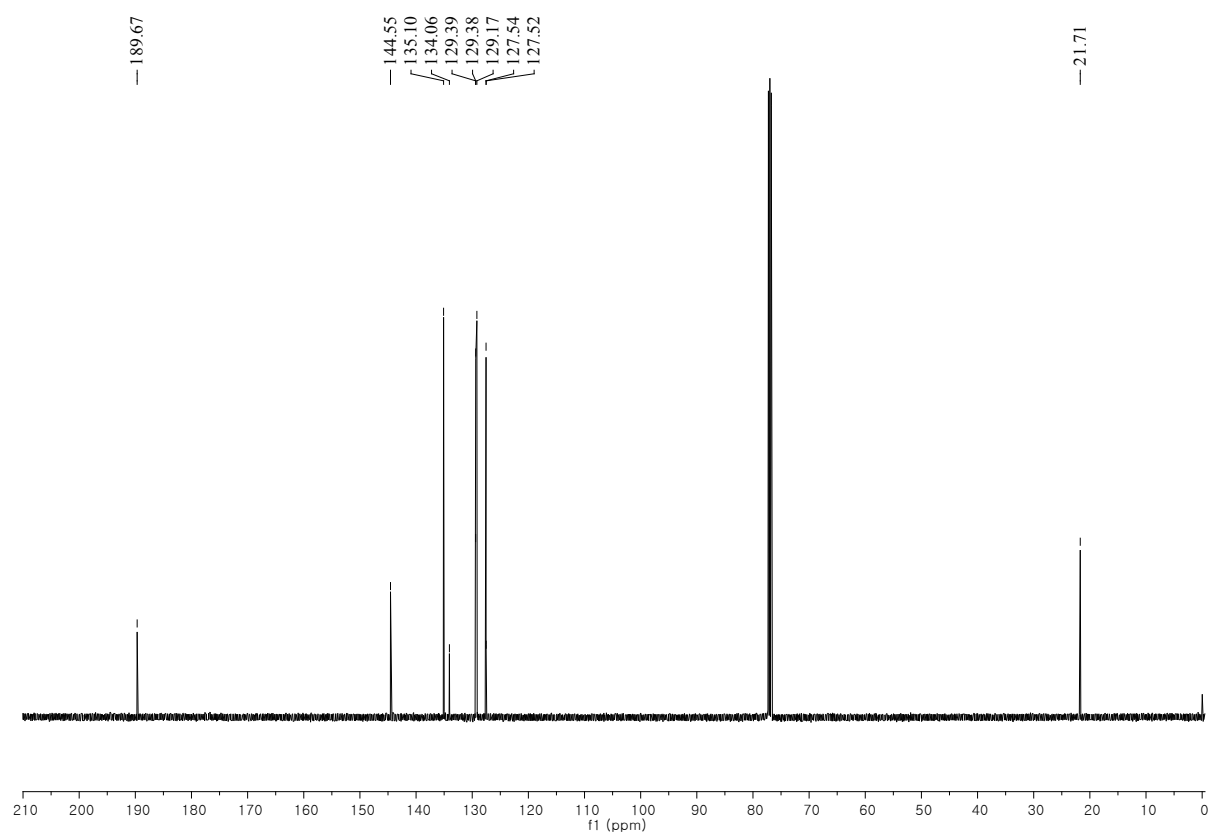
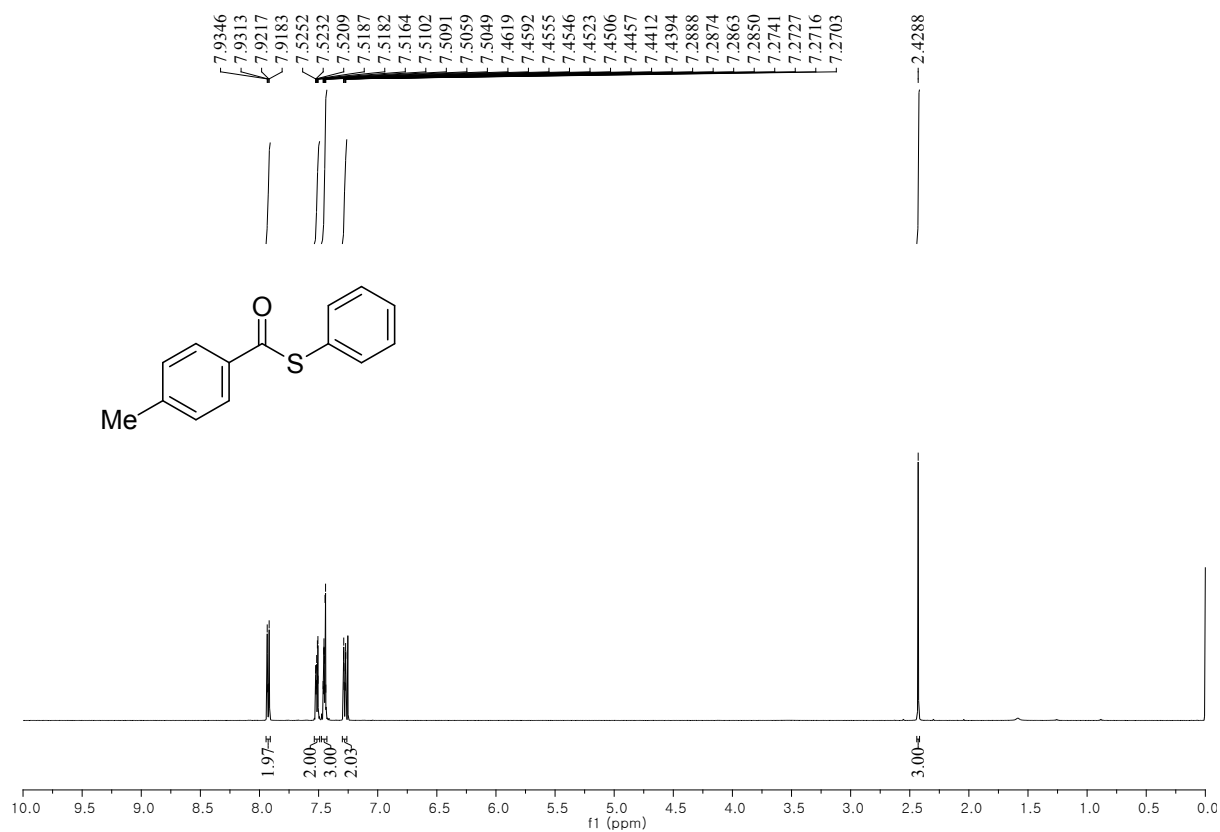




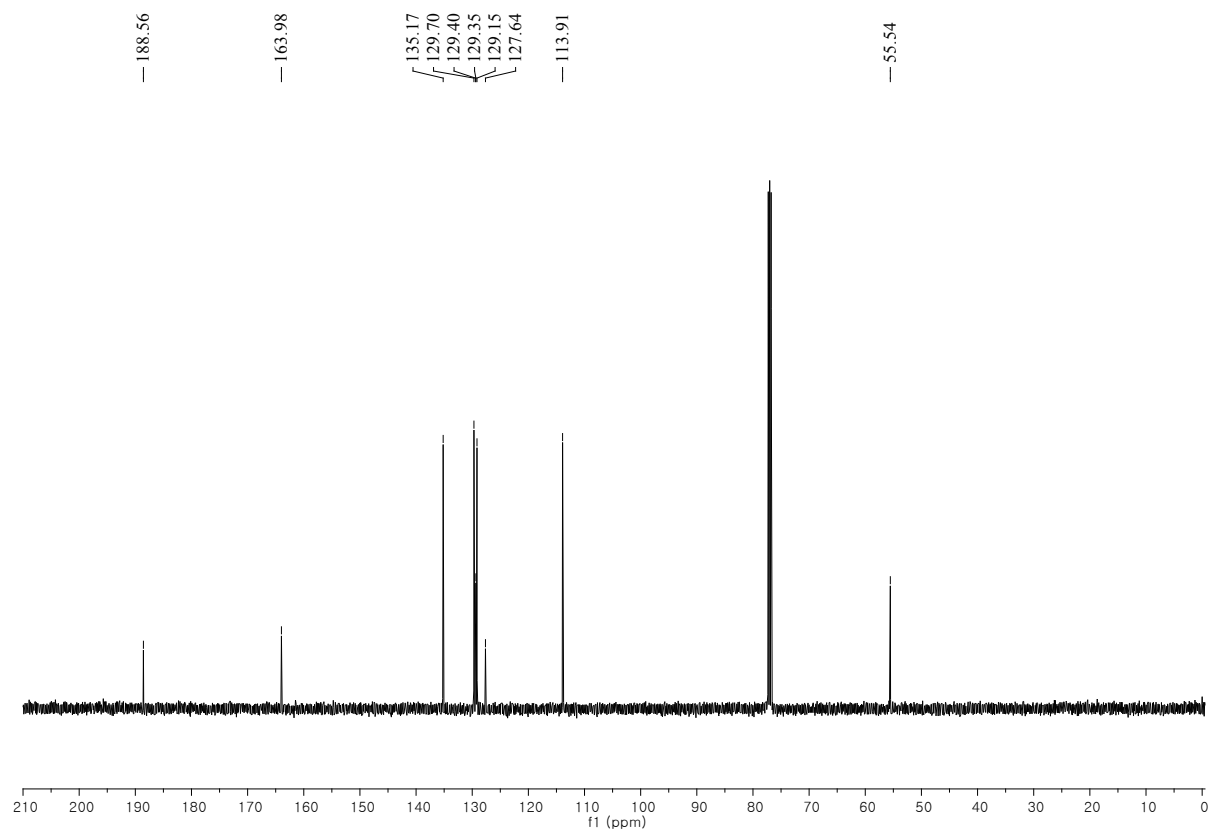
S-Phenyl 3-methylbenzothioate (3ca)



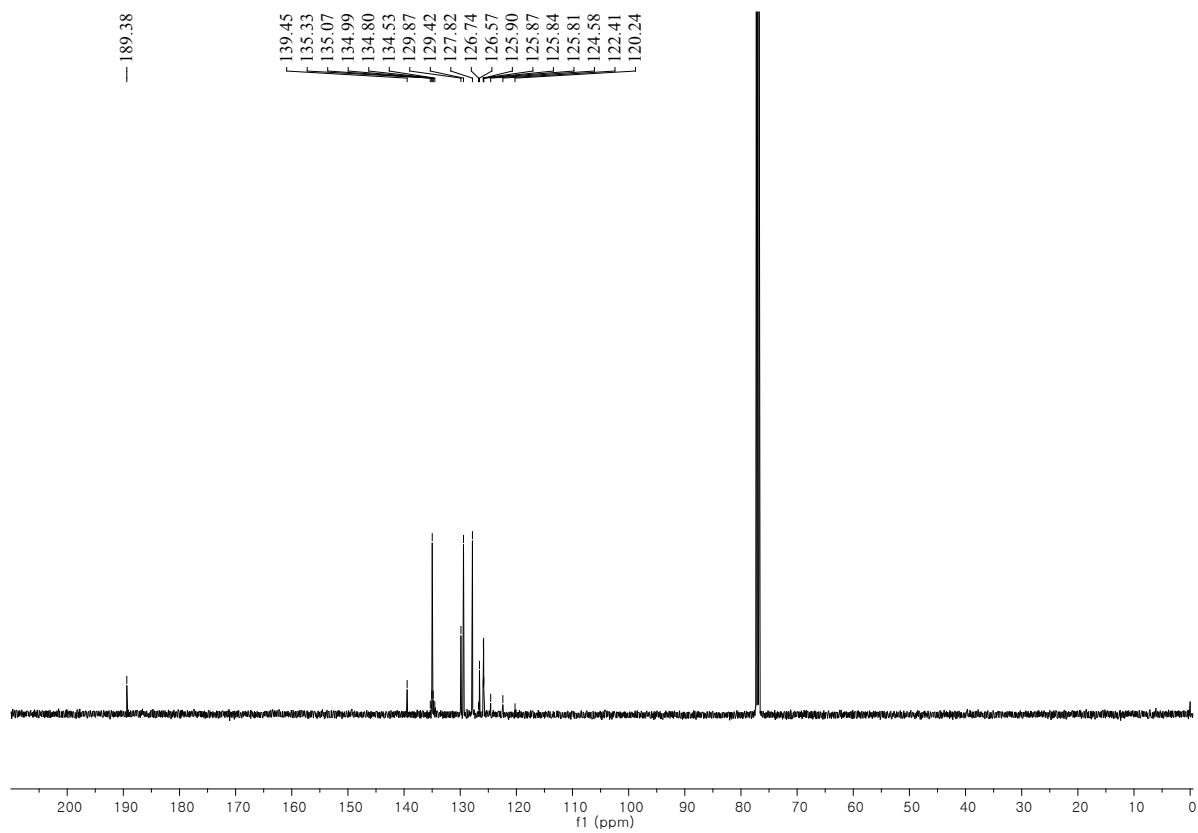
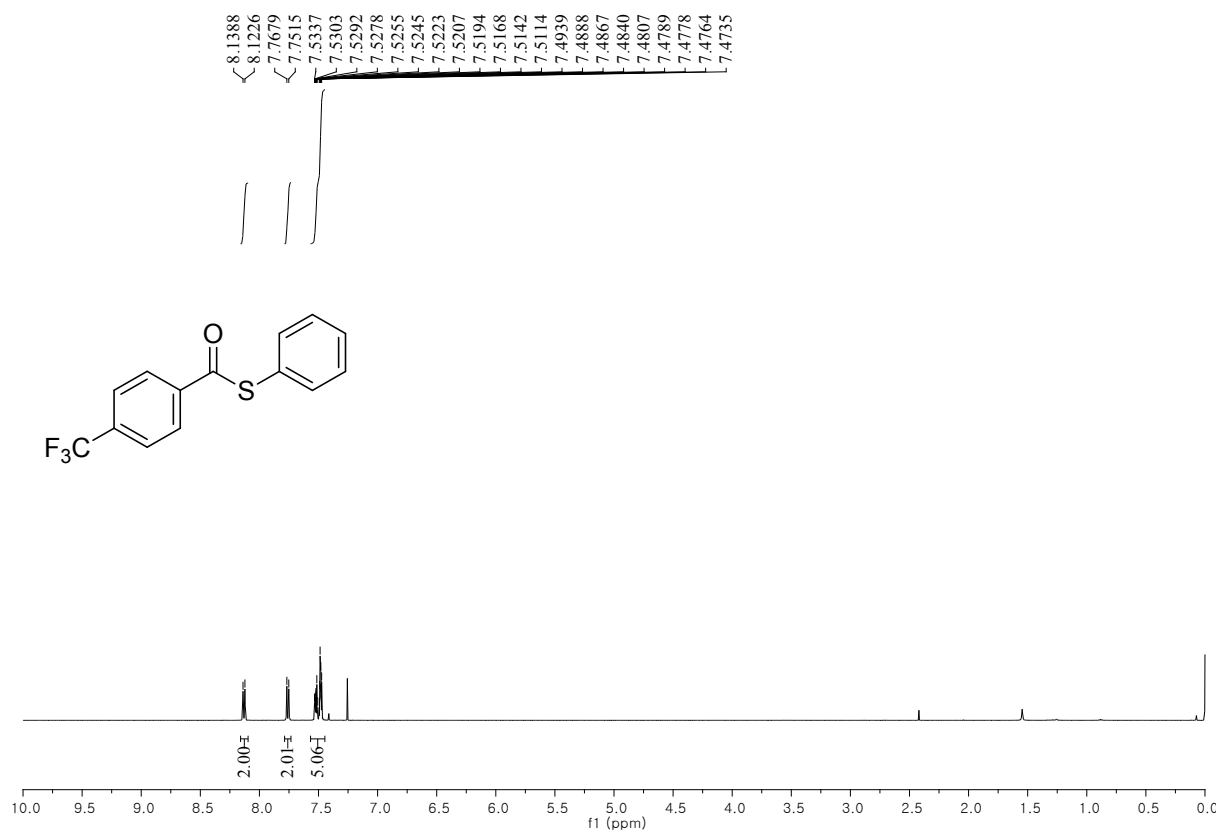
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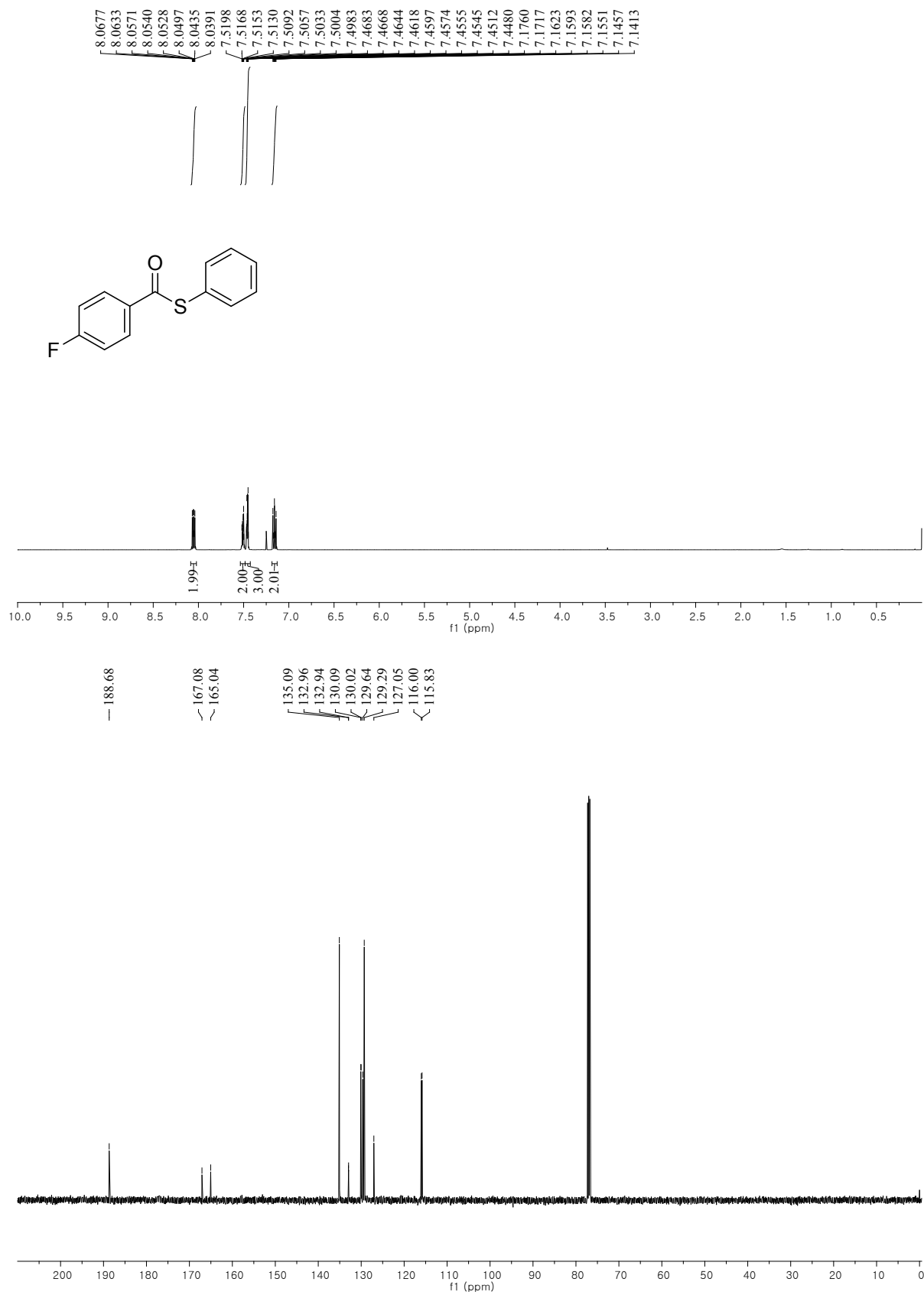
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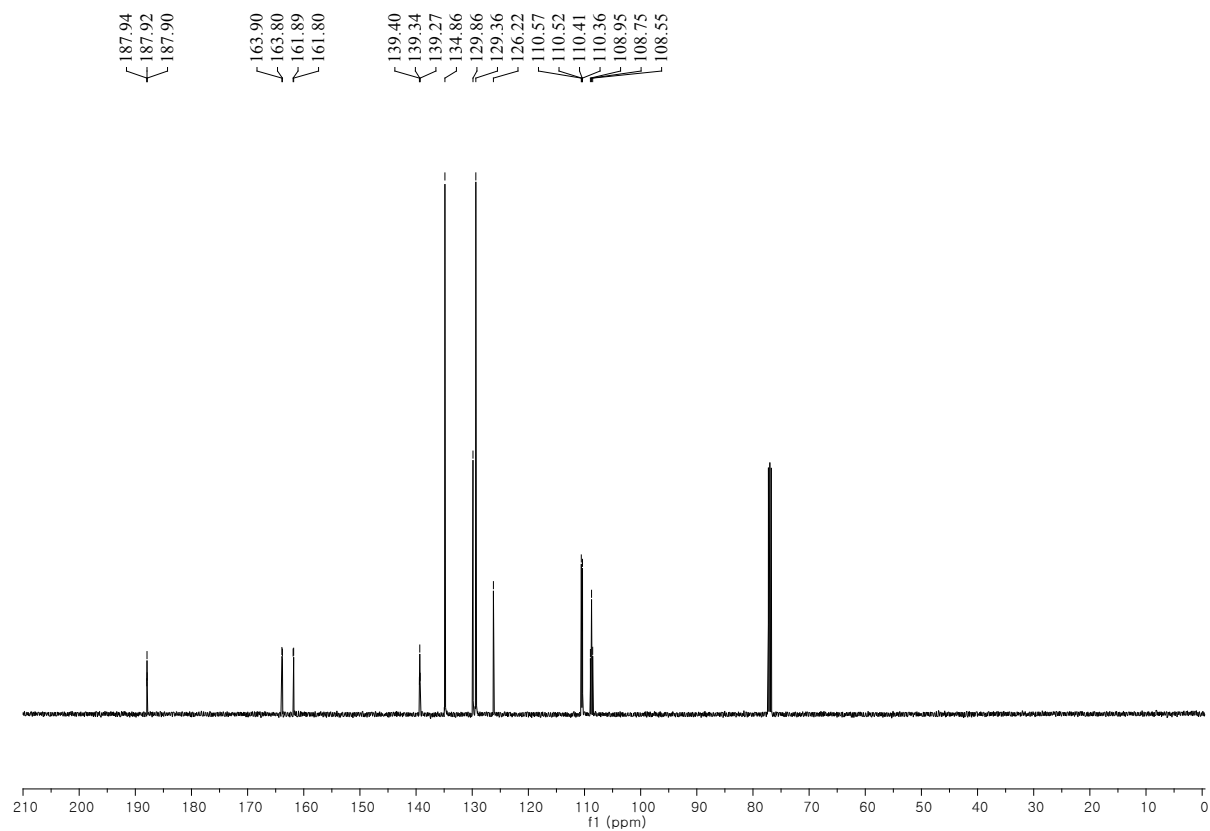
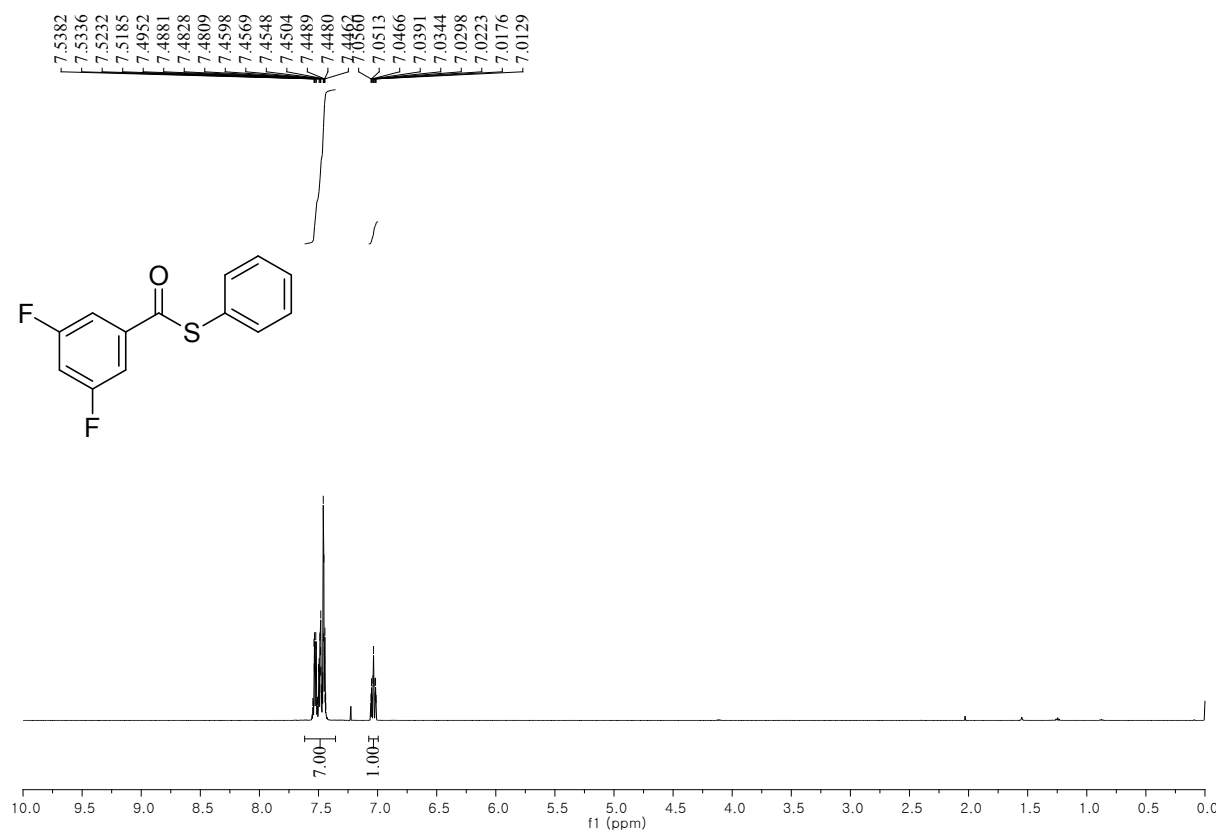
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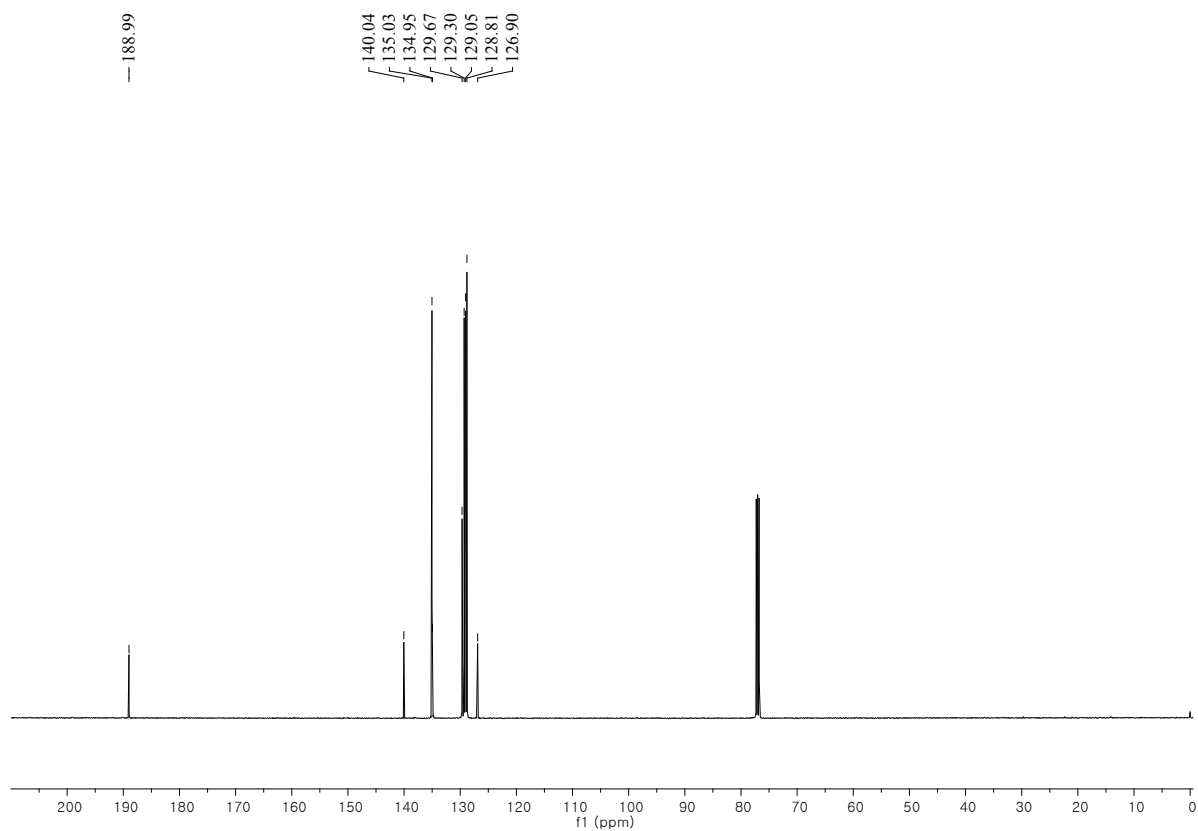
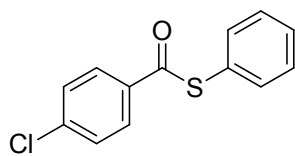
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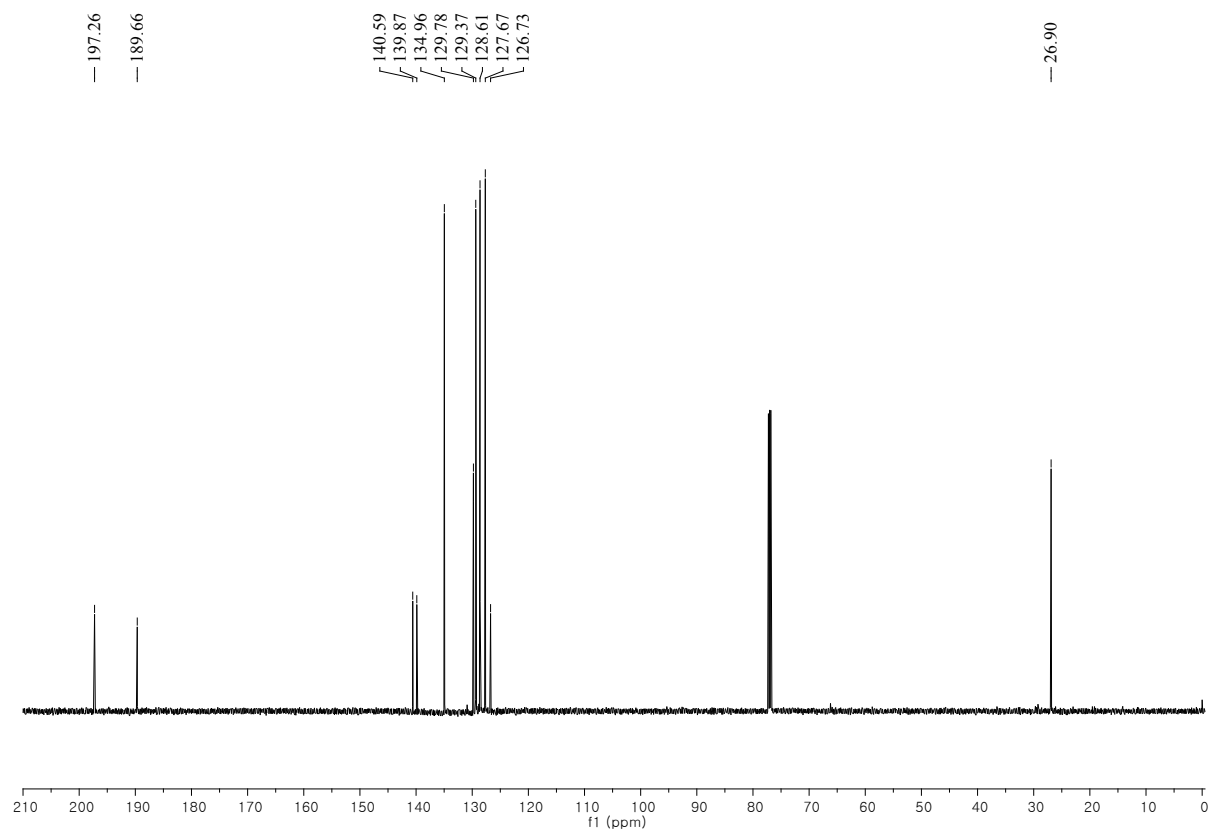
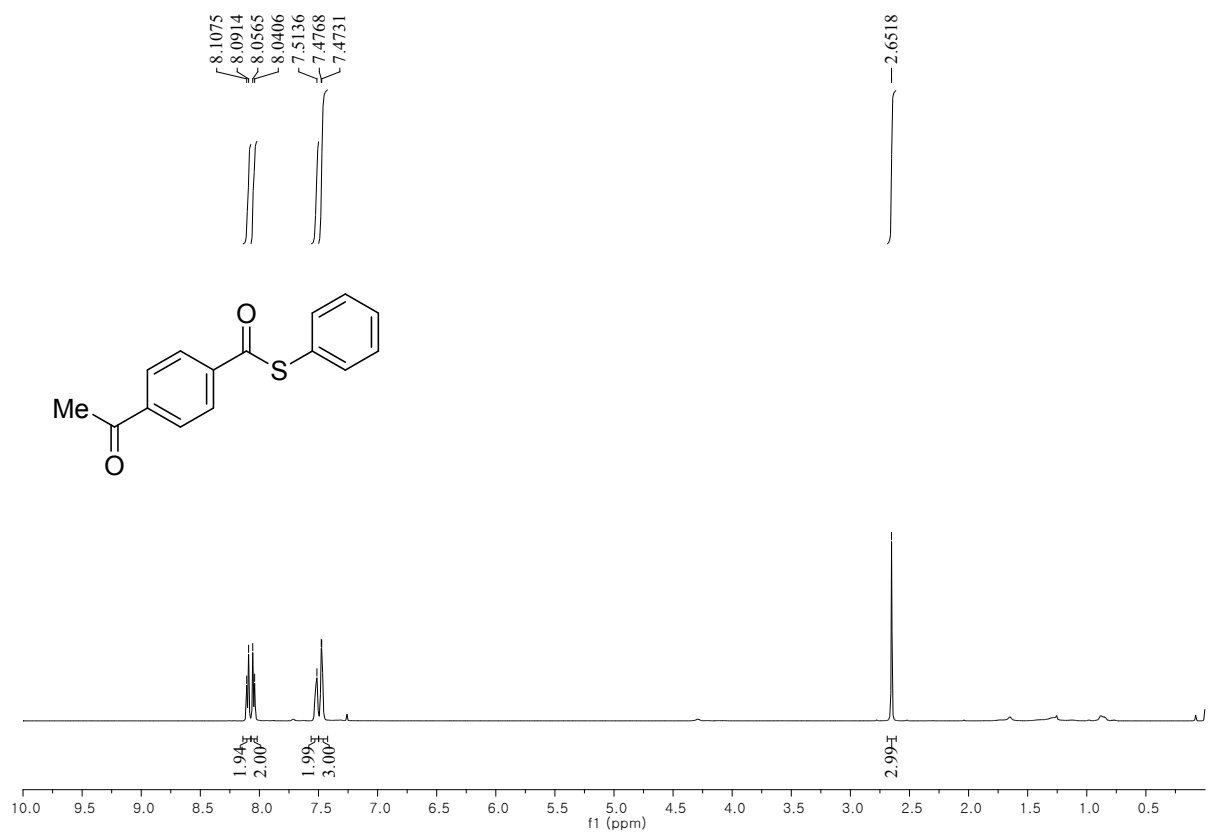
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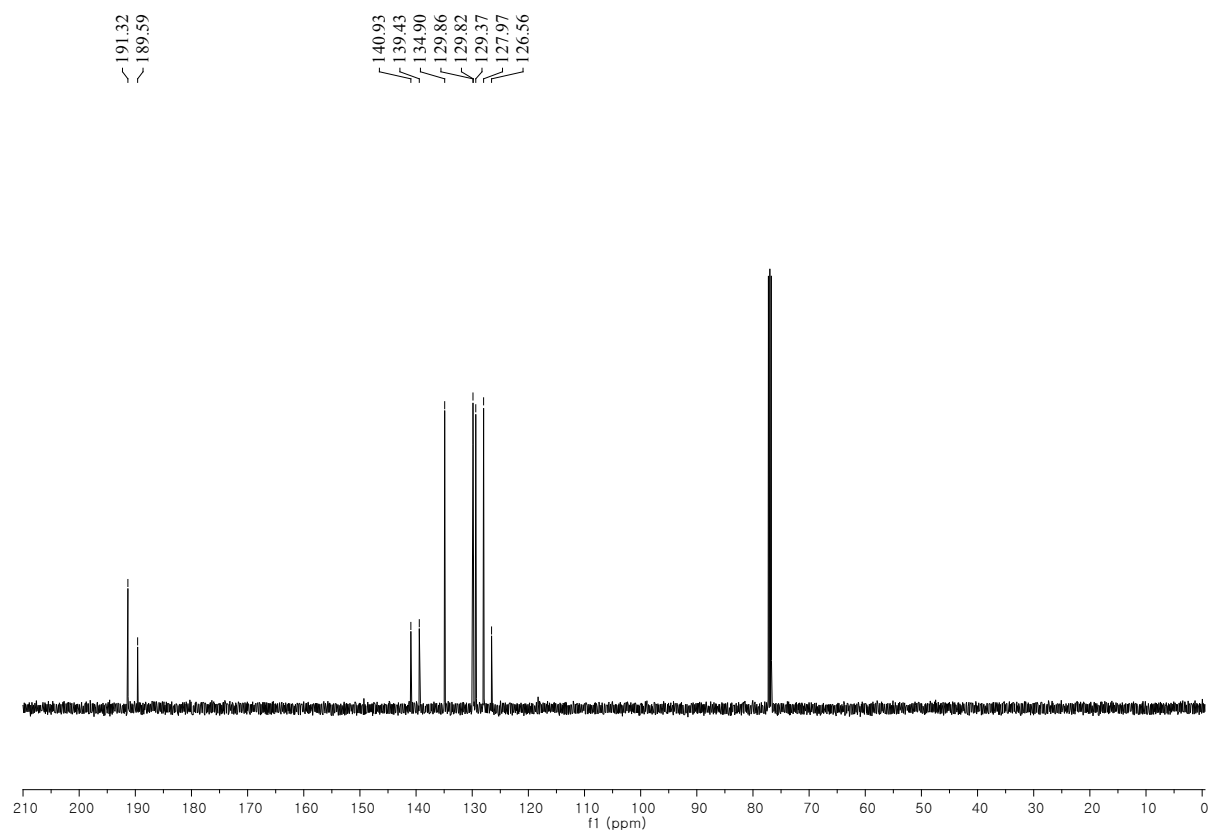
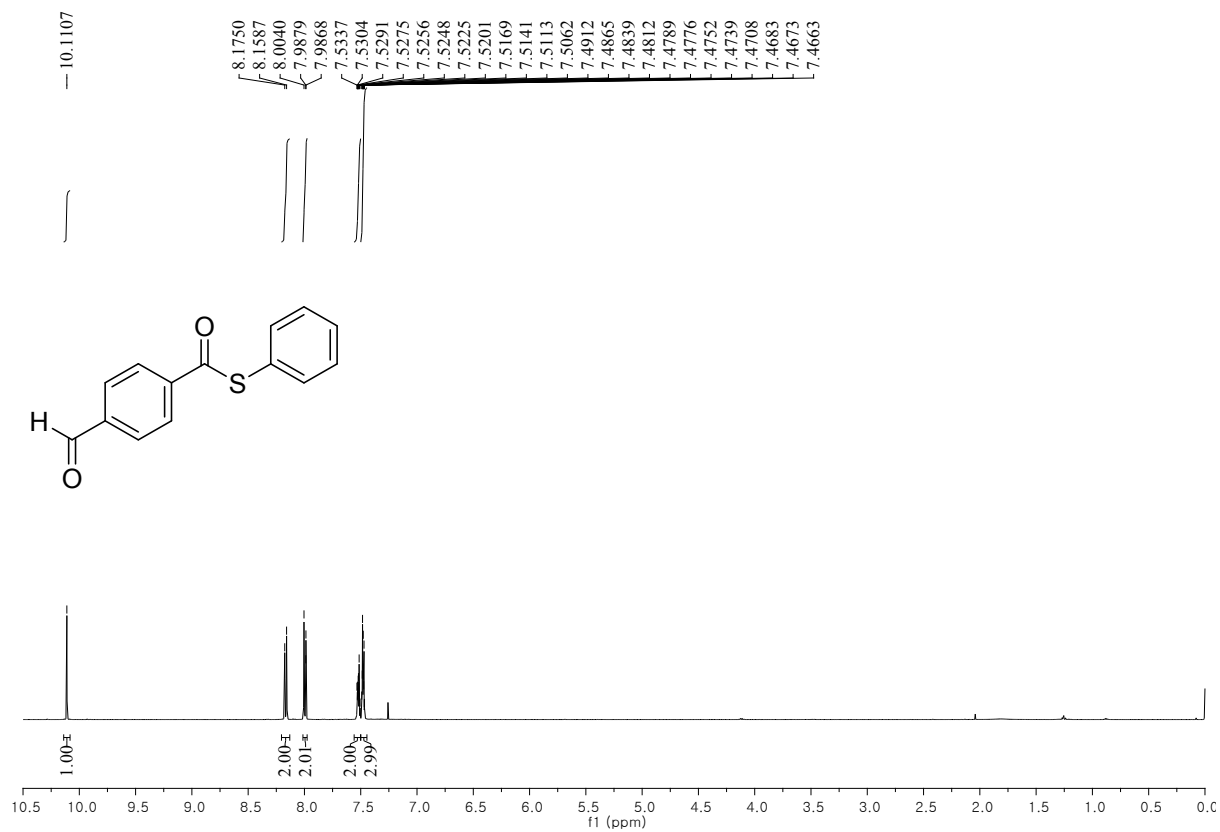
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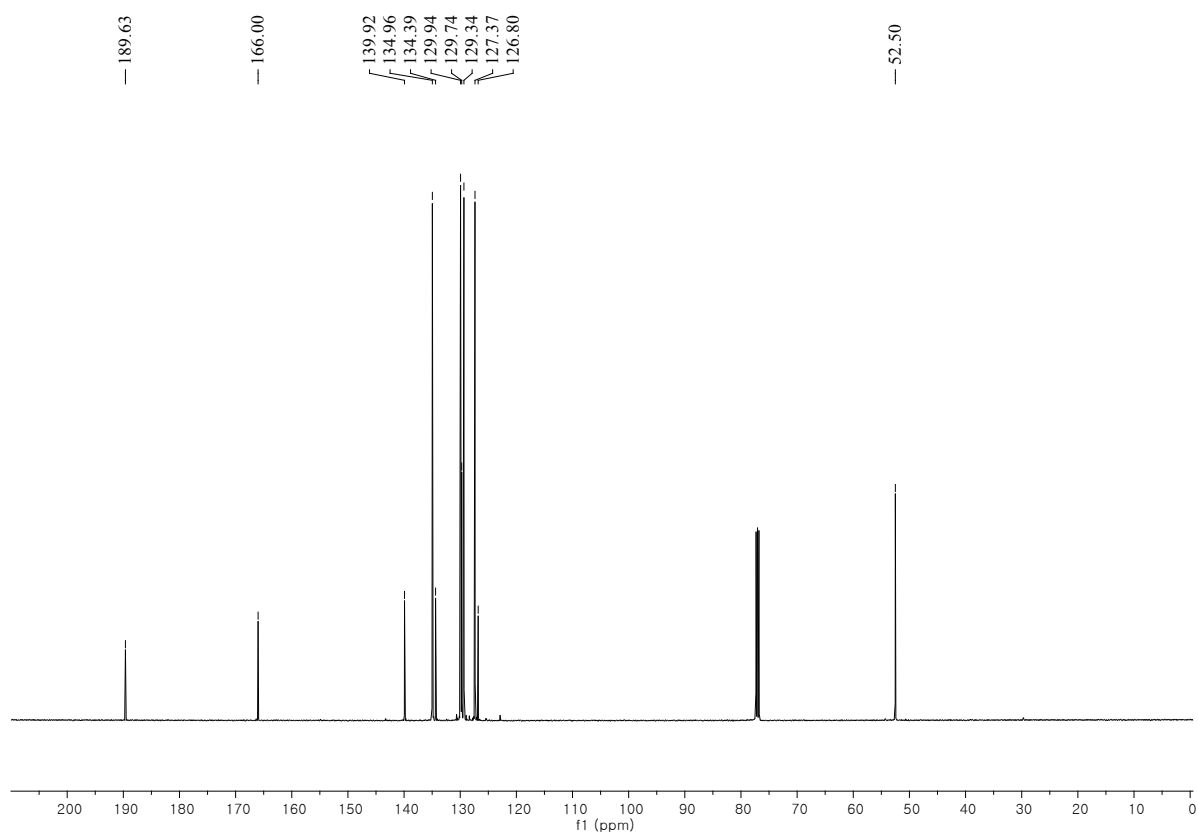
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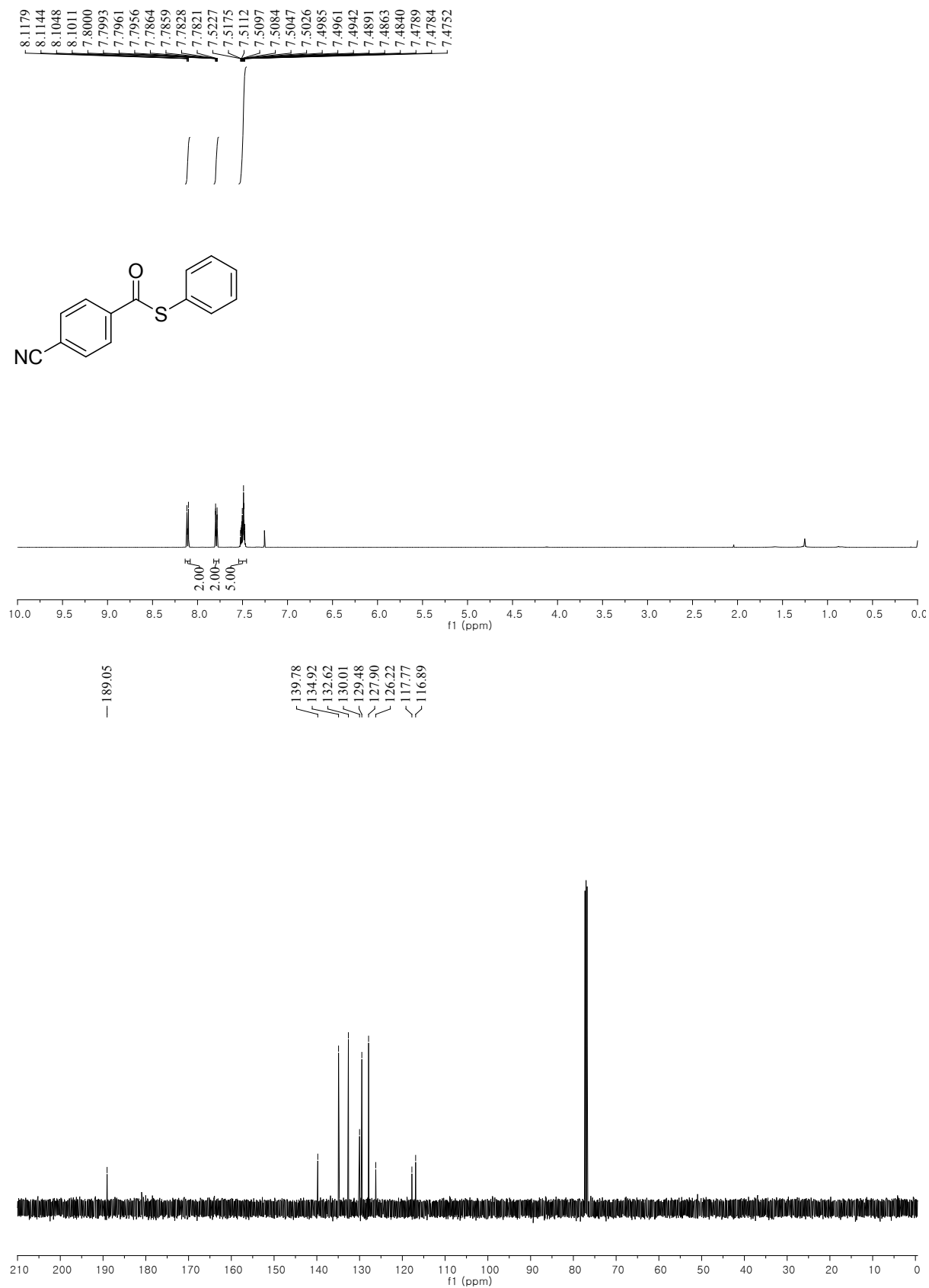
S-Phenyl 4-formylbenzothioate (3ka)



Methyl 4-((phenylthio)carbonyl)benzoate (3la)



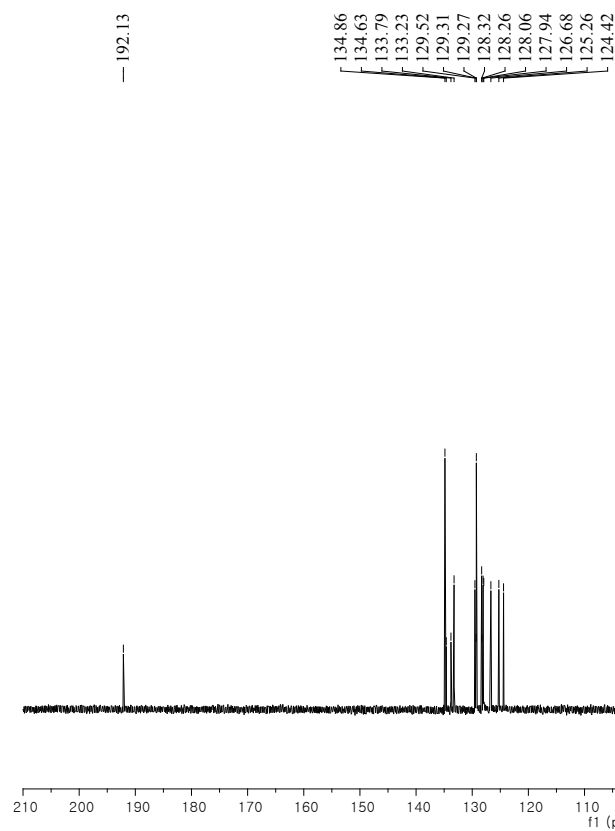
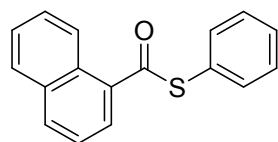
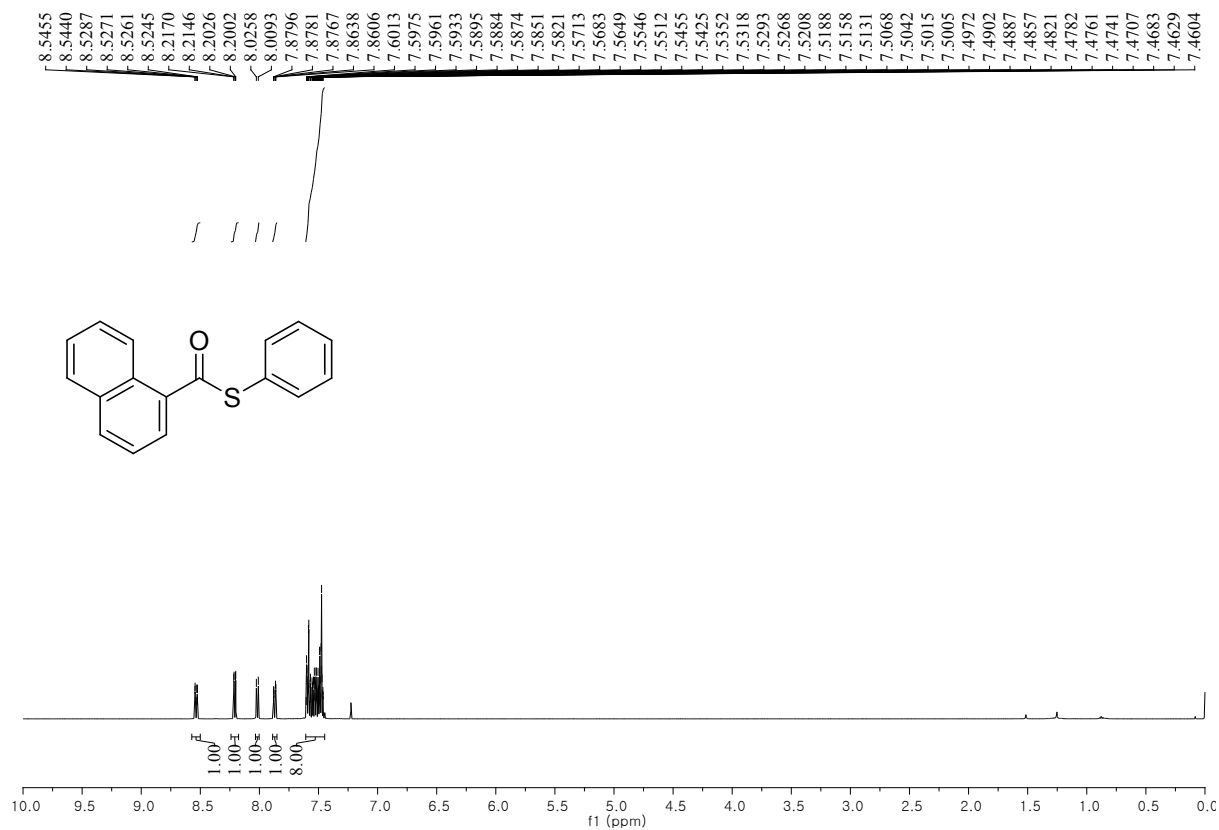
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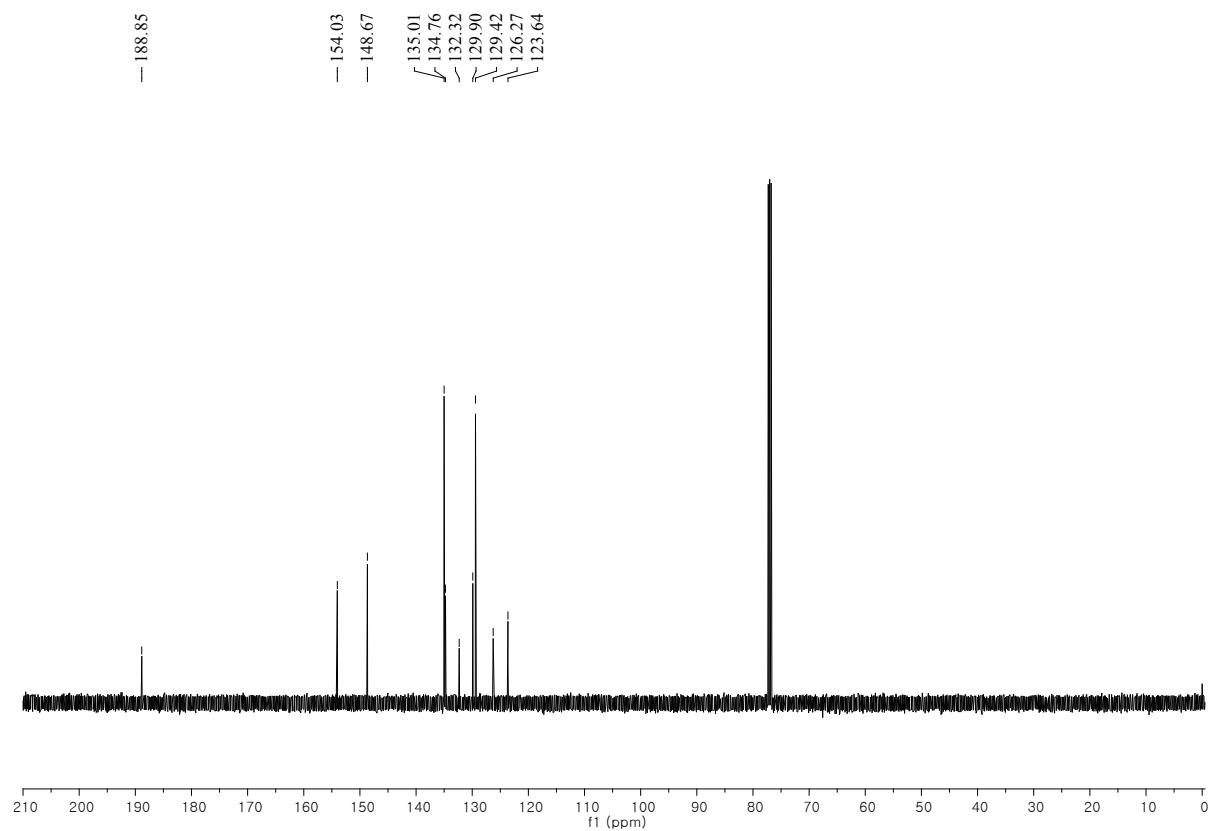
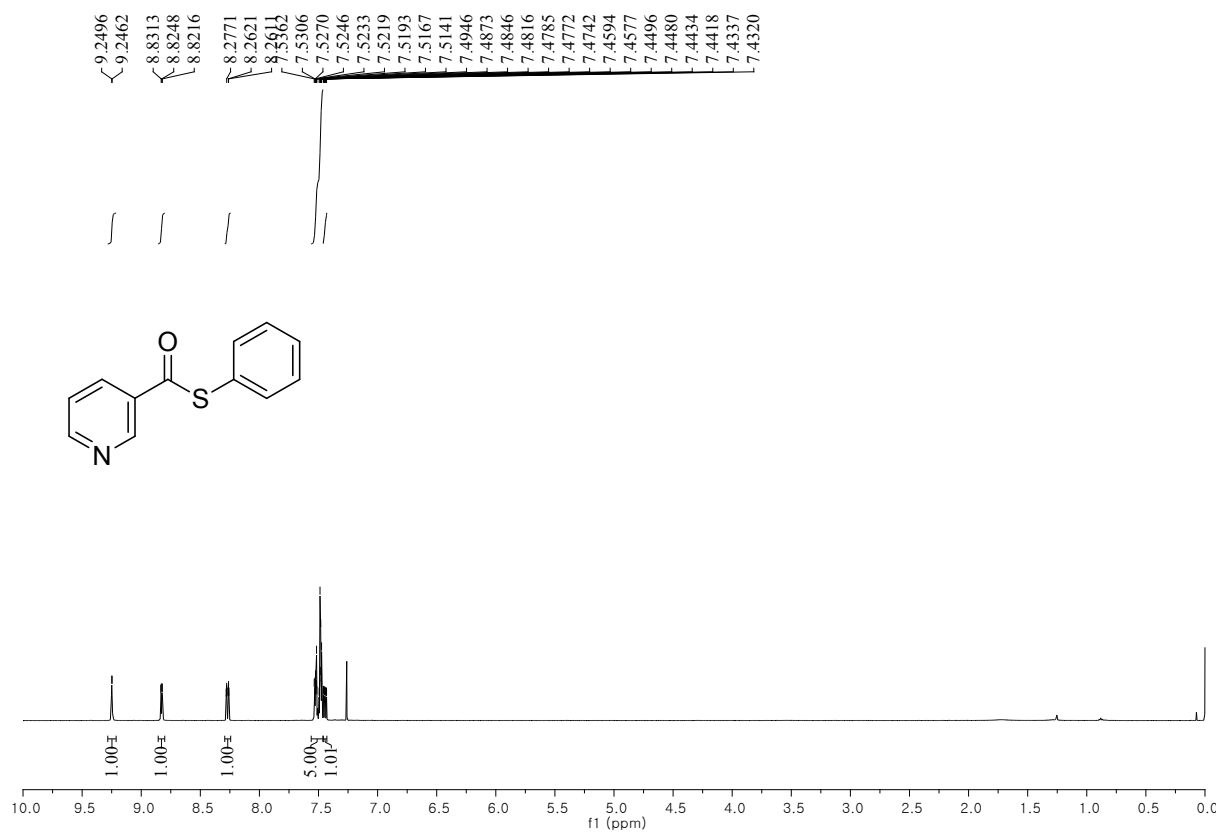
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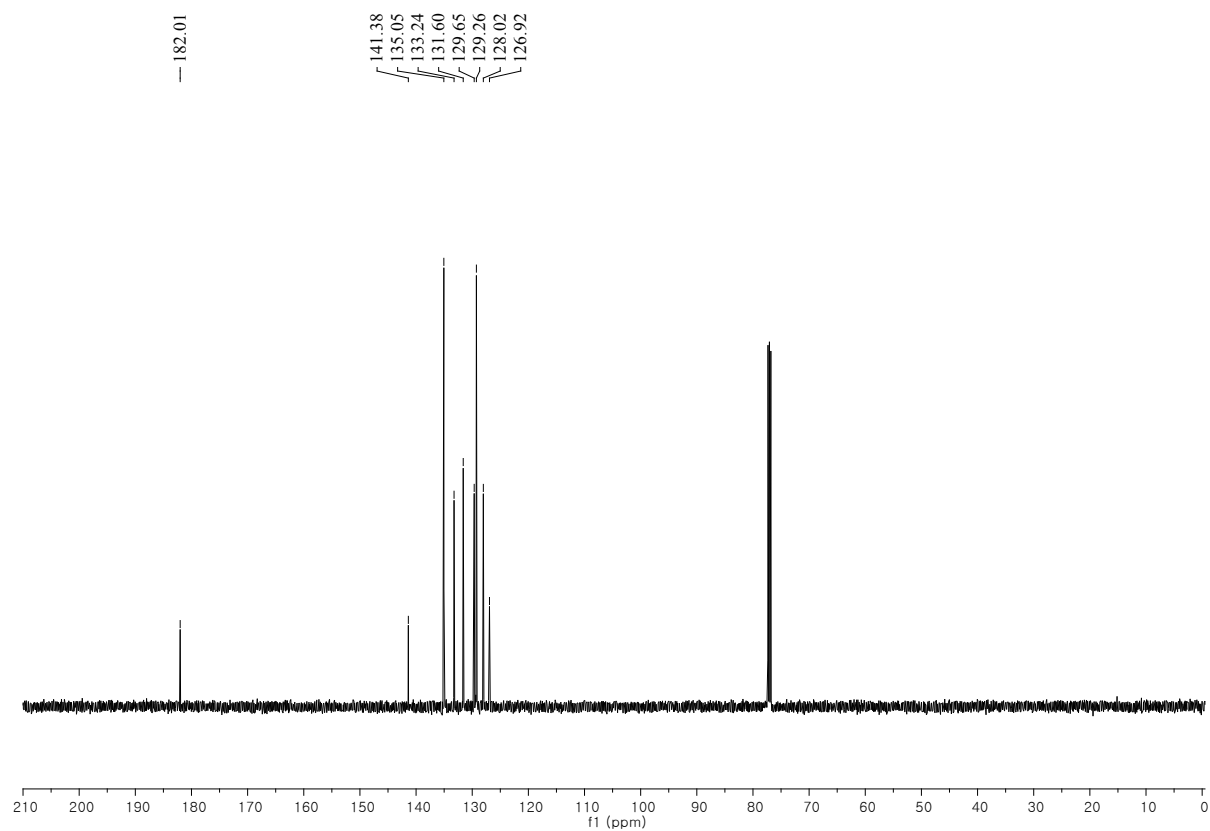
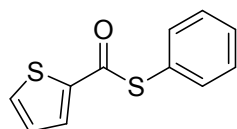
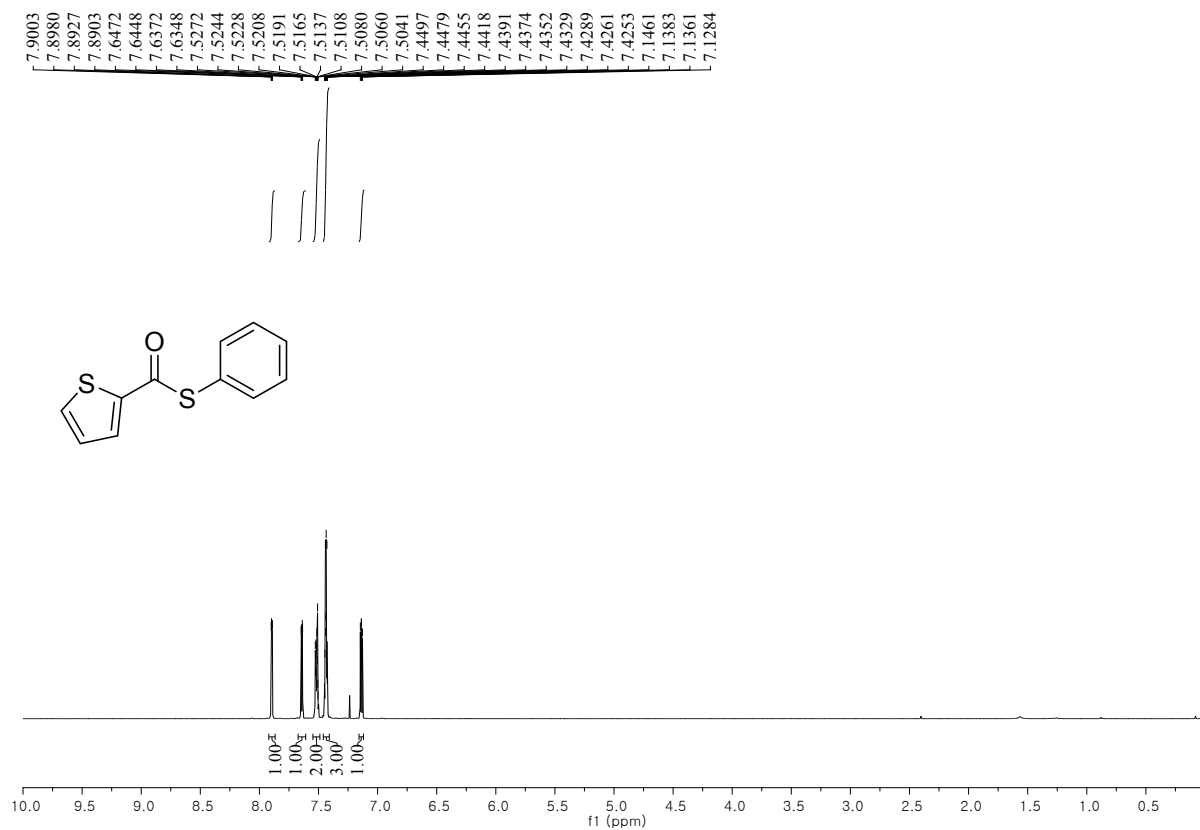
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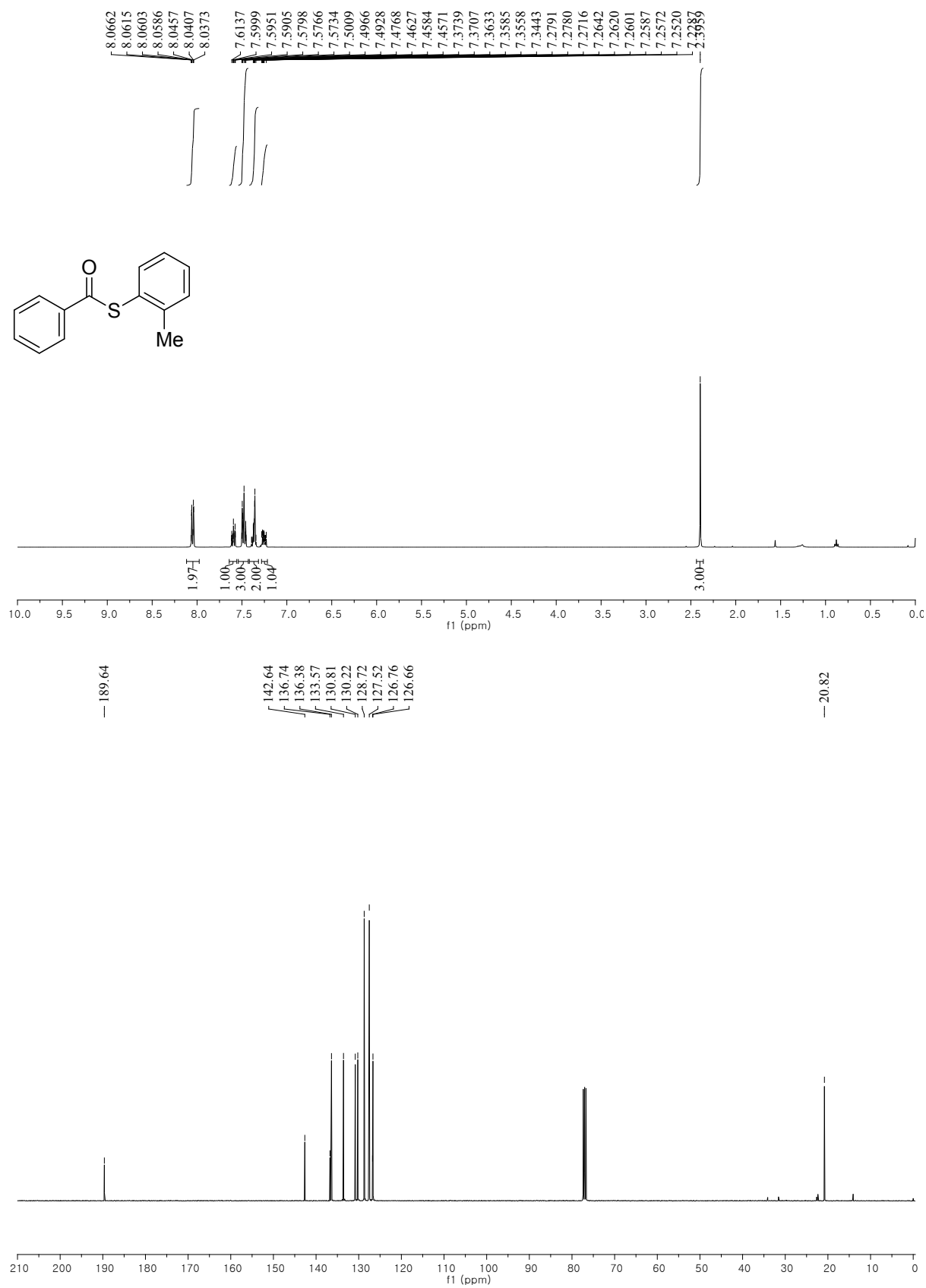
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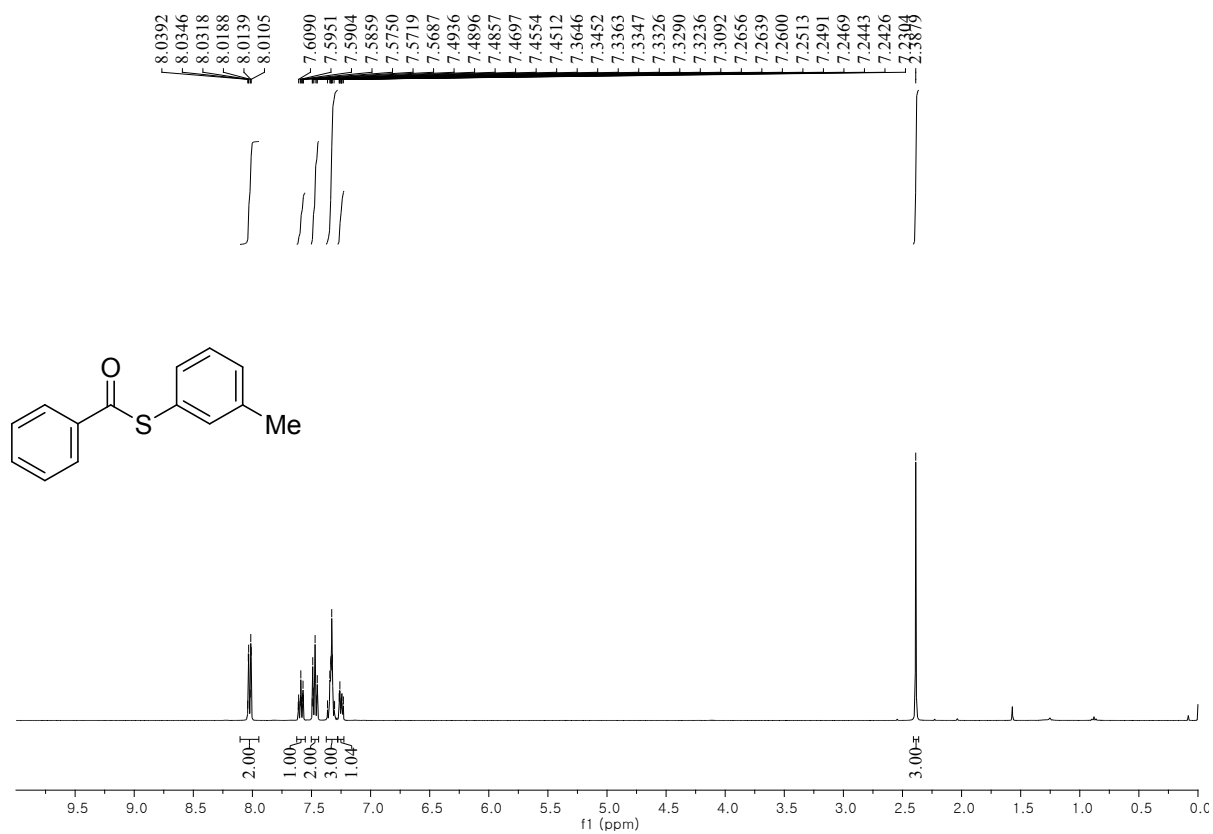
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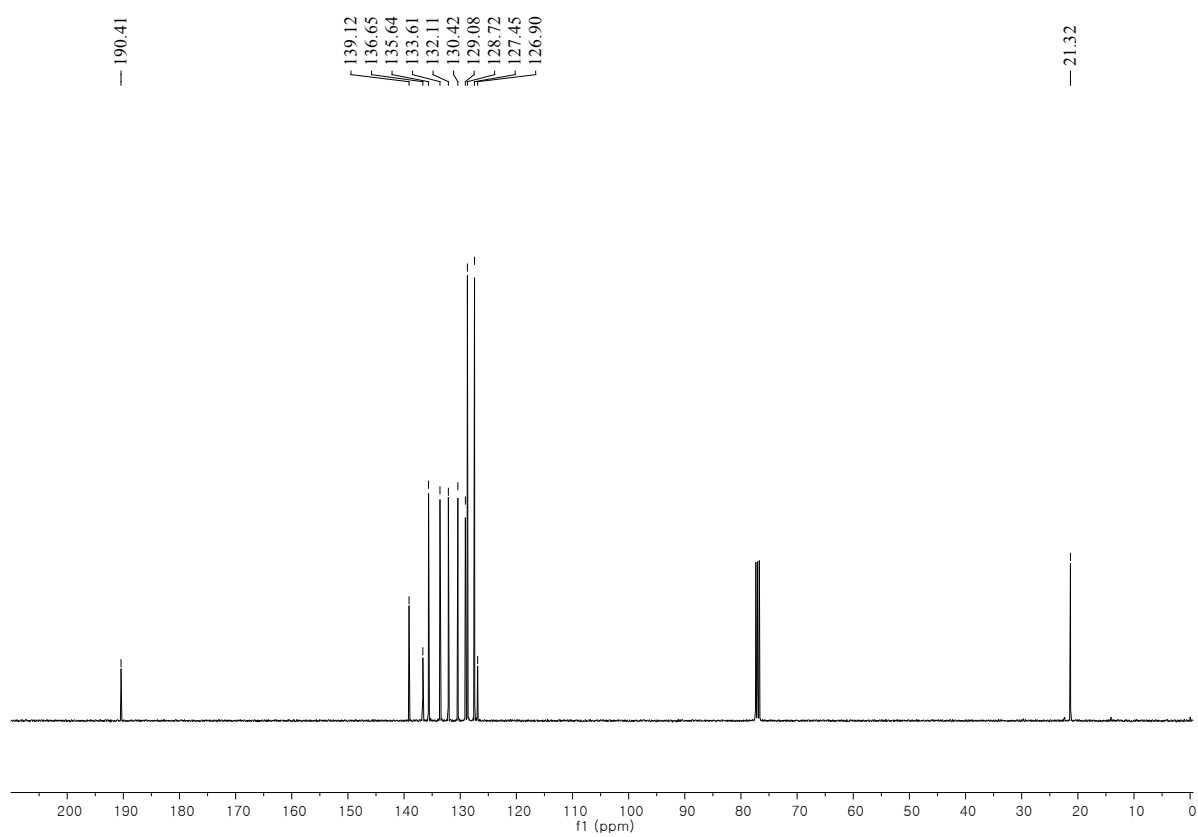


***S*-*o*-Tolyl benzothioate (3ab)**

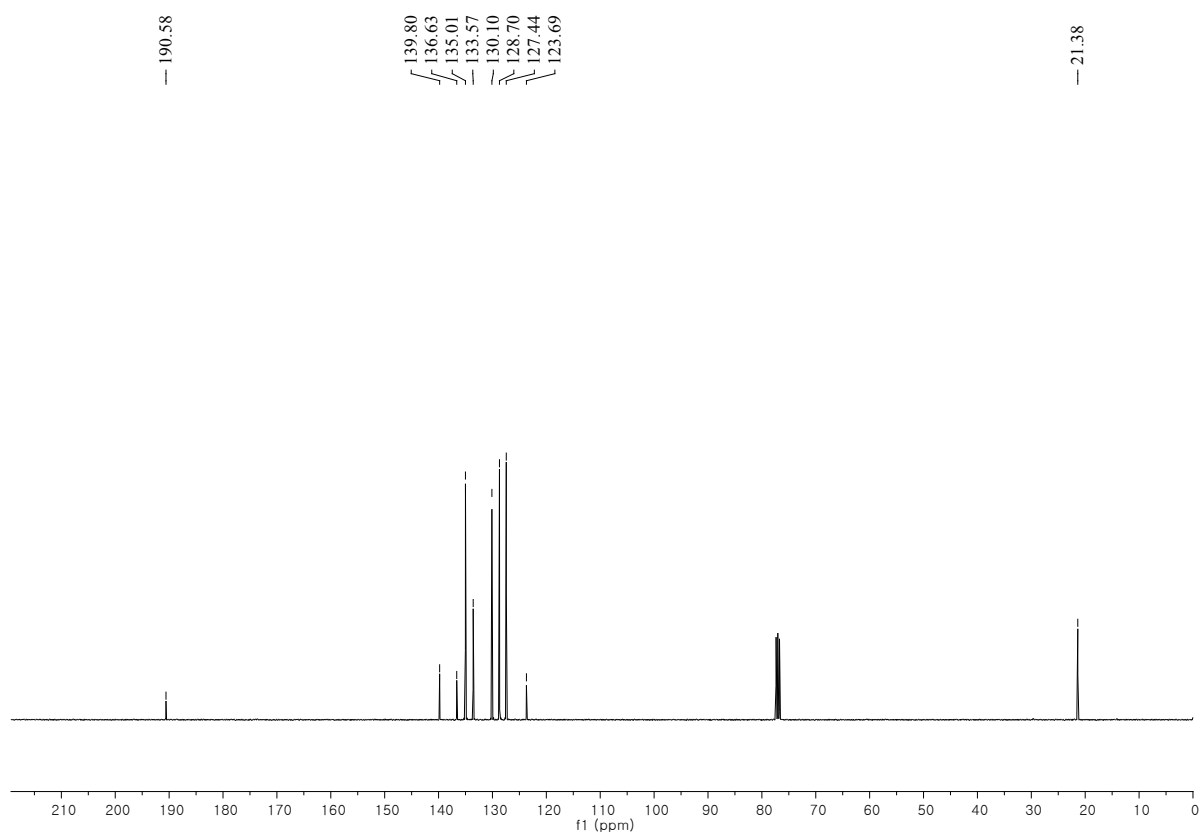
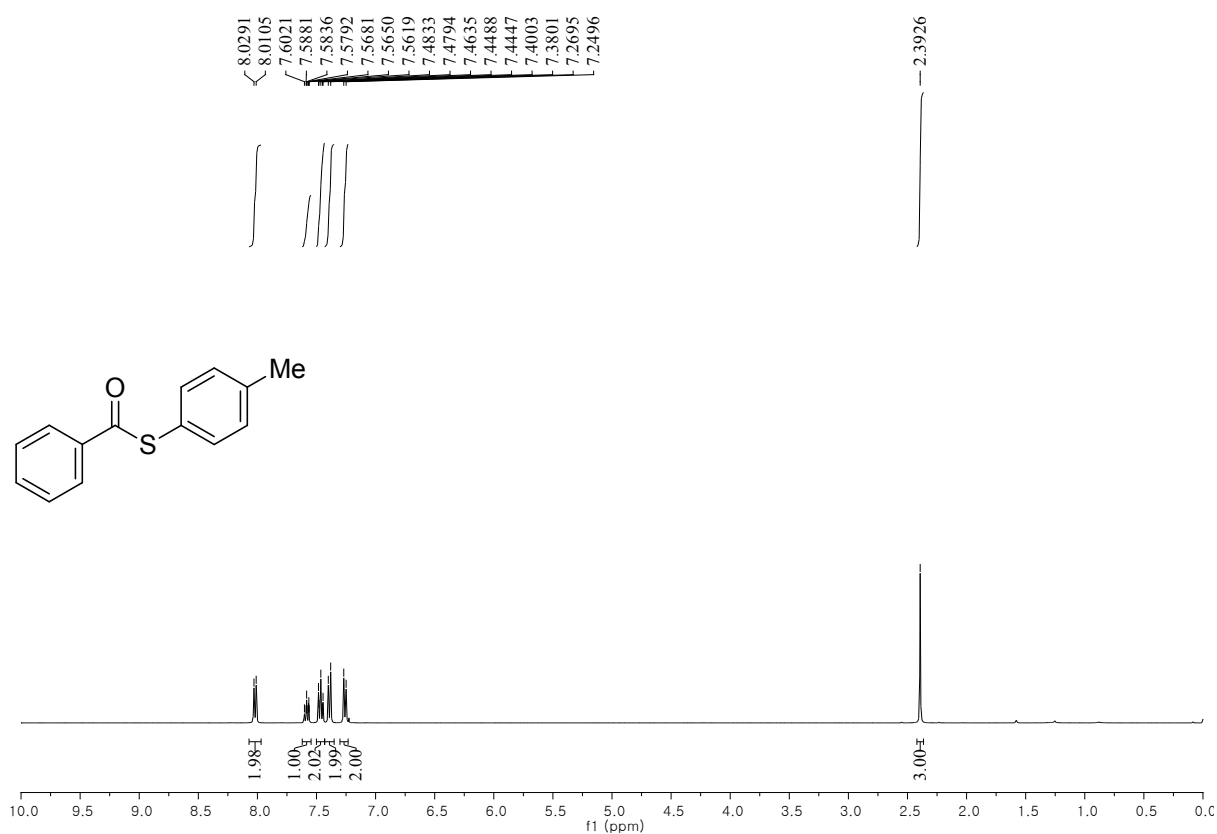


***S*-*m*-Tolyl benzothioate (3ac)**

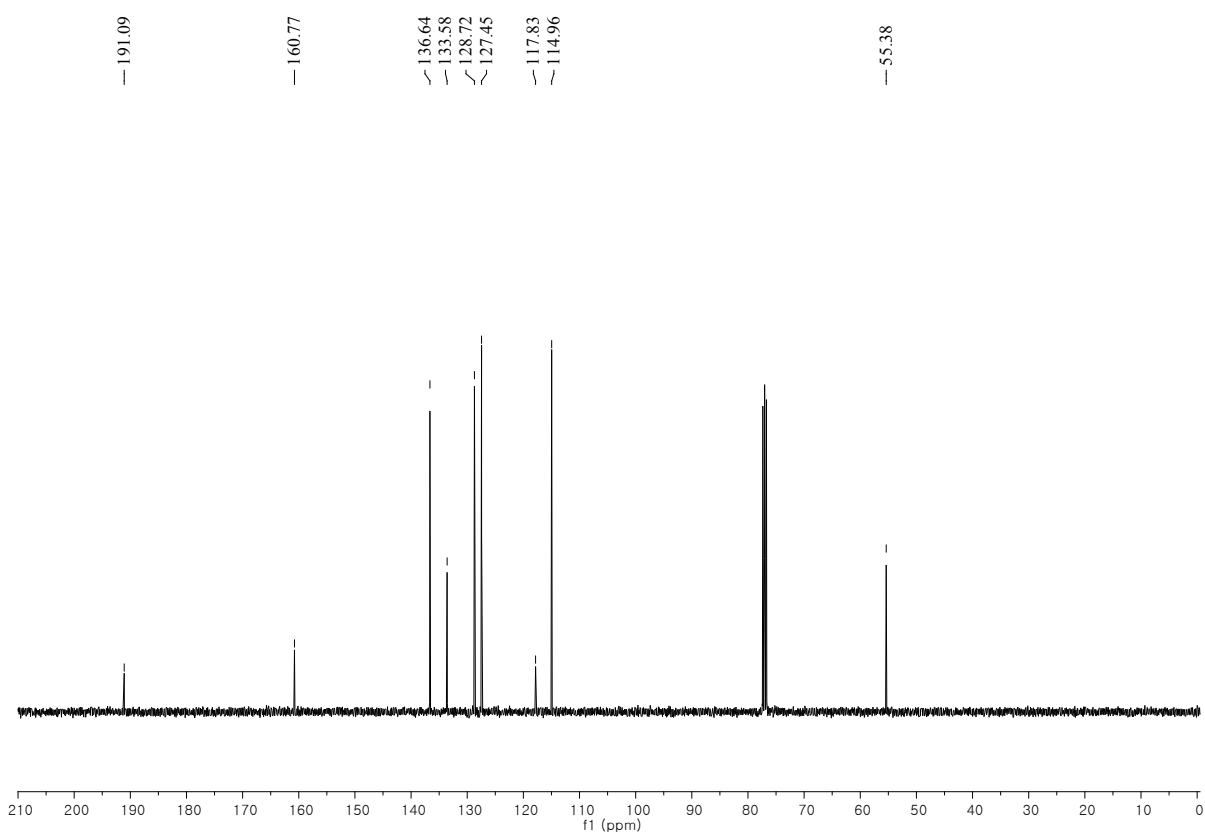
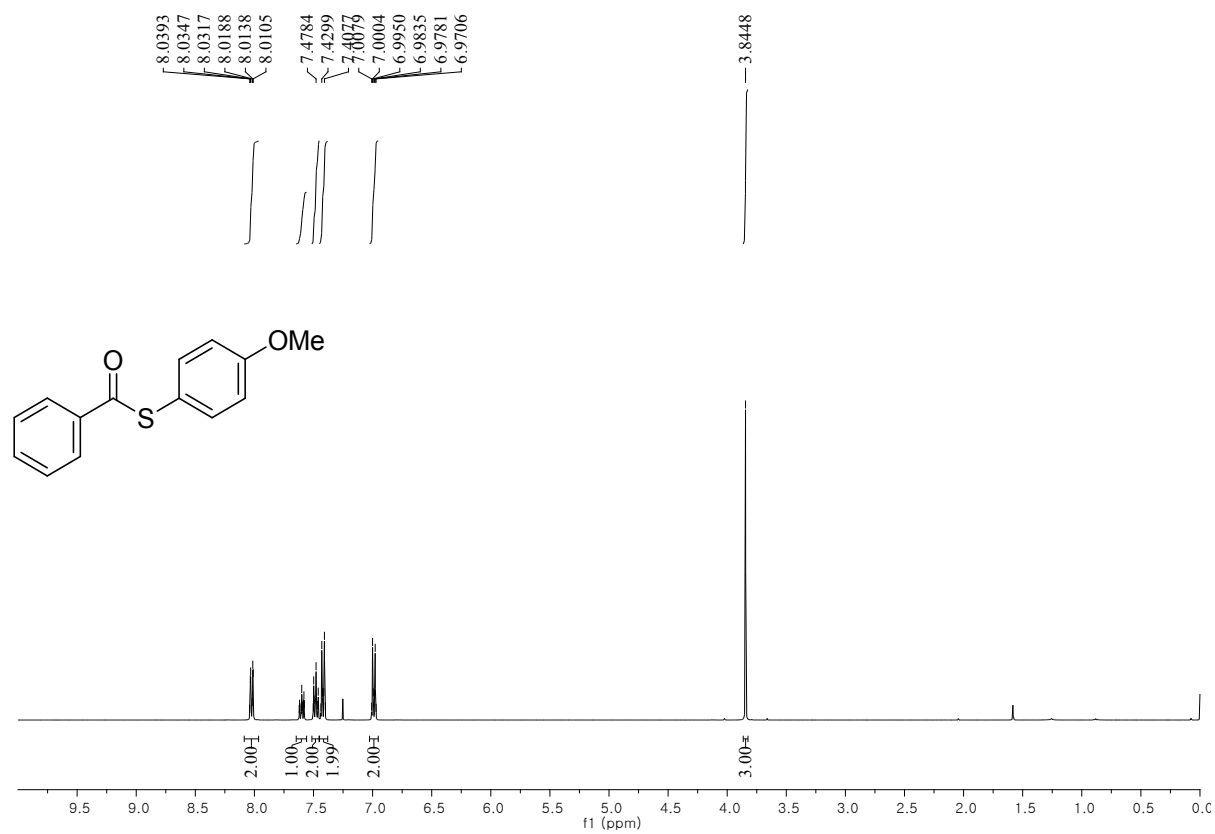




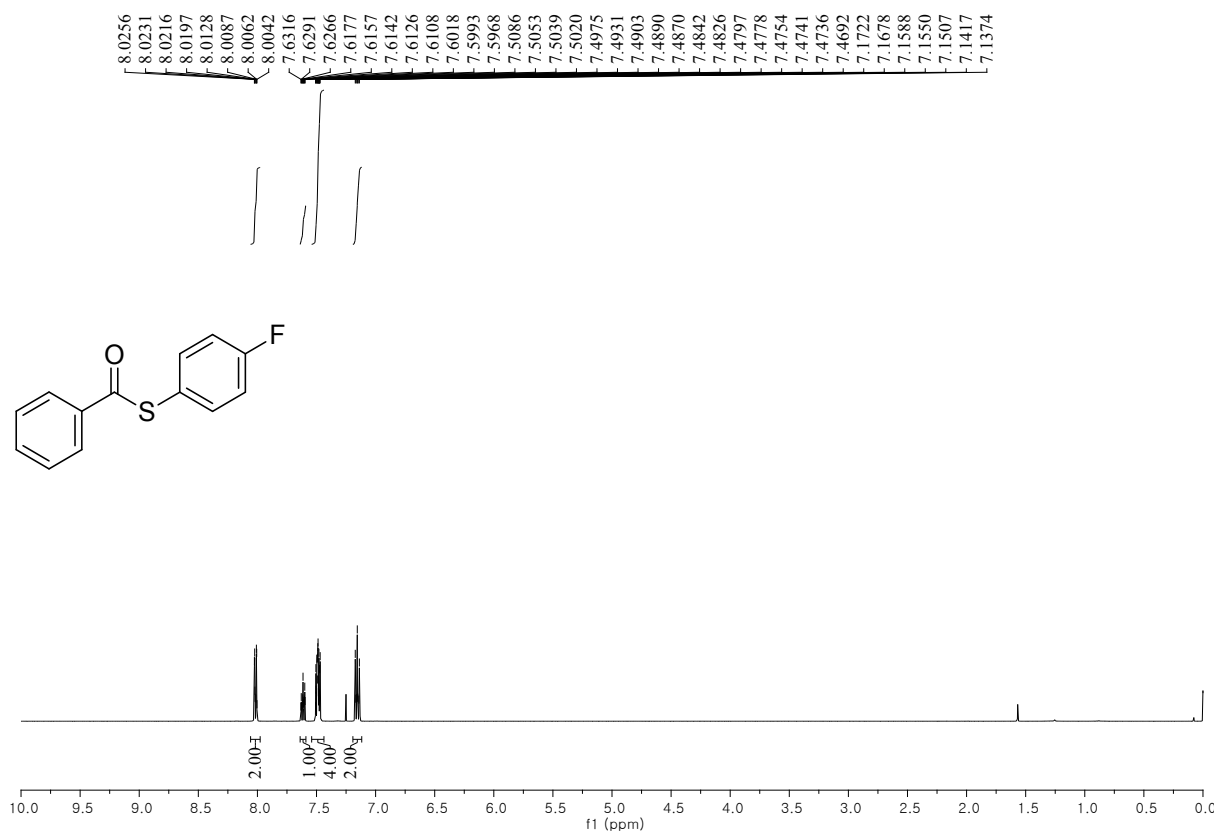
***S-p*-Tolyl benzothioate (3ad)**

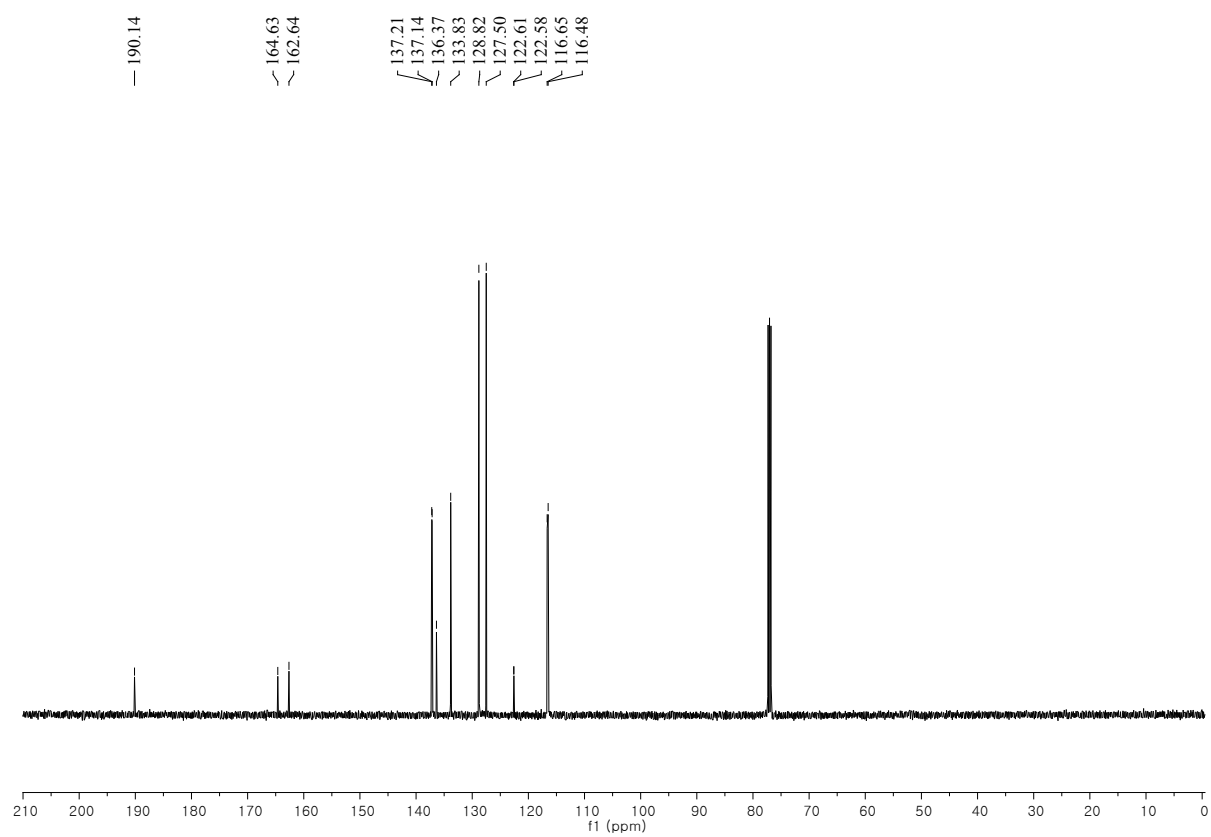


***S*-(4-Methoxyphenyl) benzothioate (3ae)**

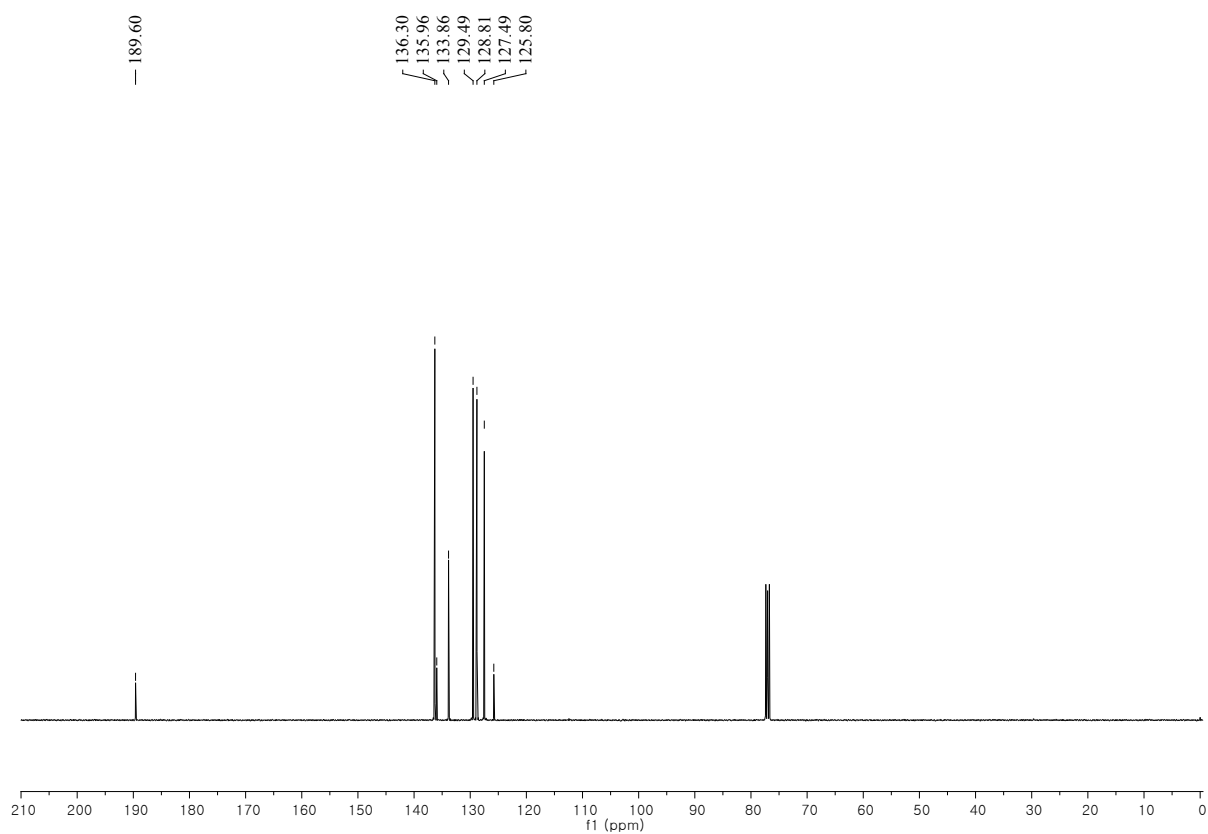
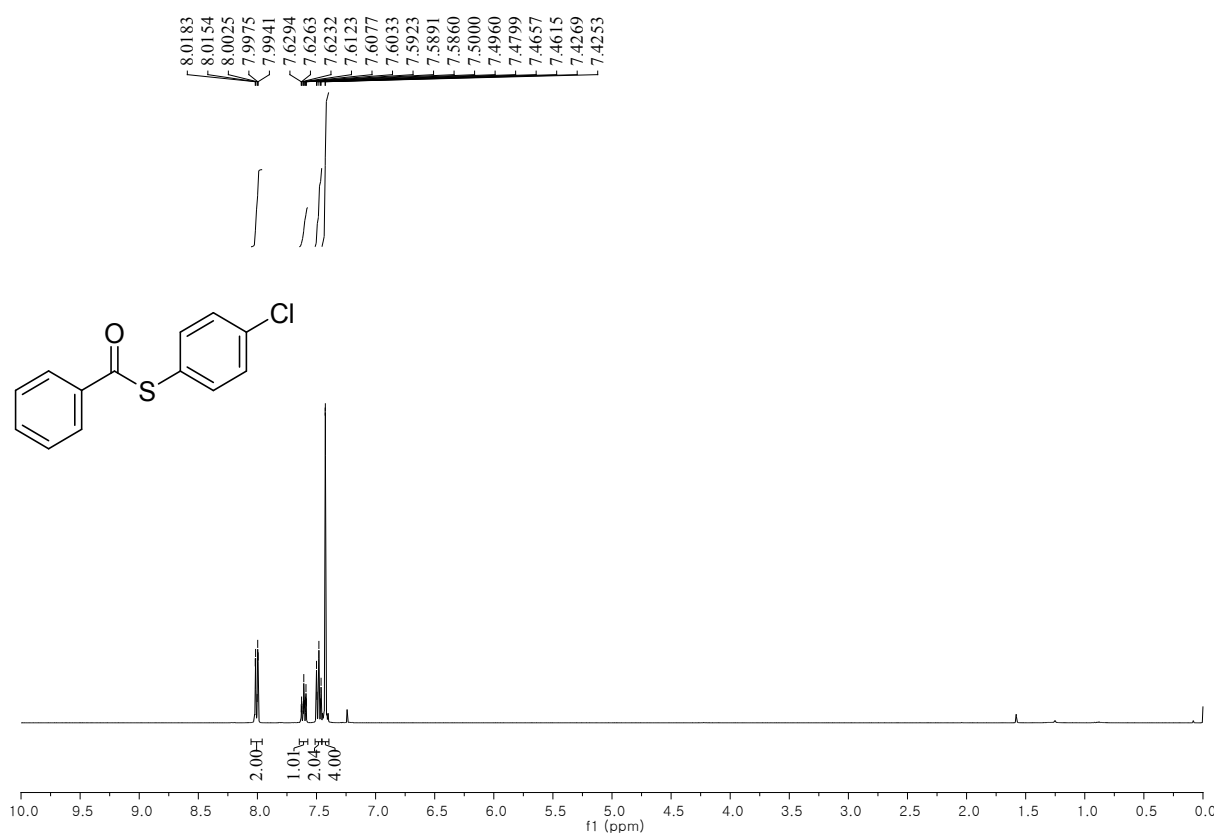


***S*-(4-Fluorophenyl) benzothioate (3ag)**

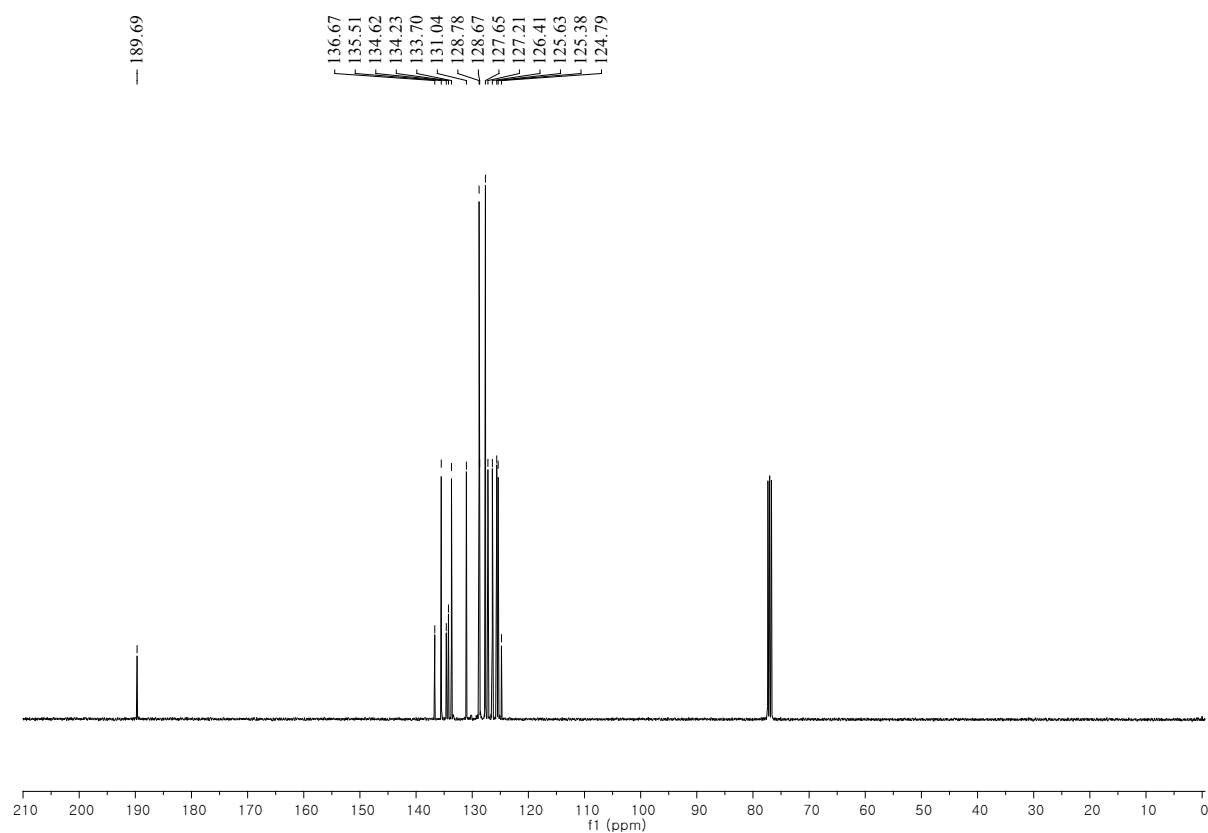
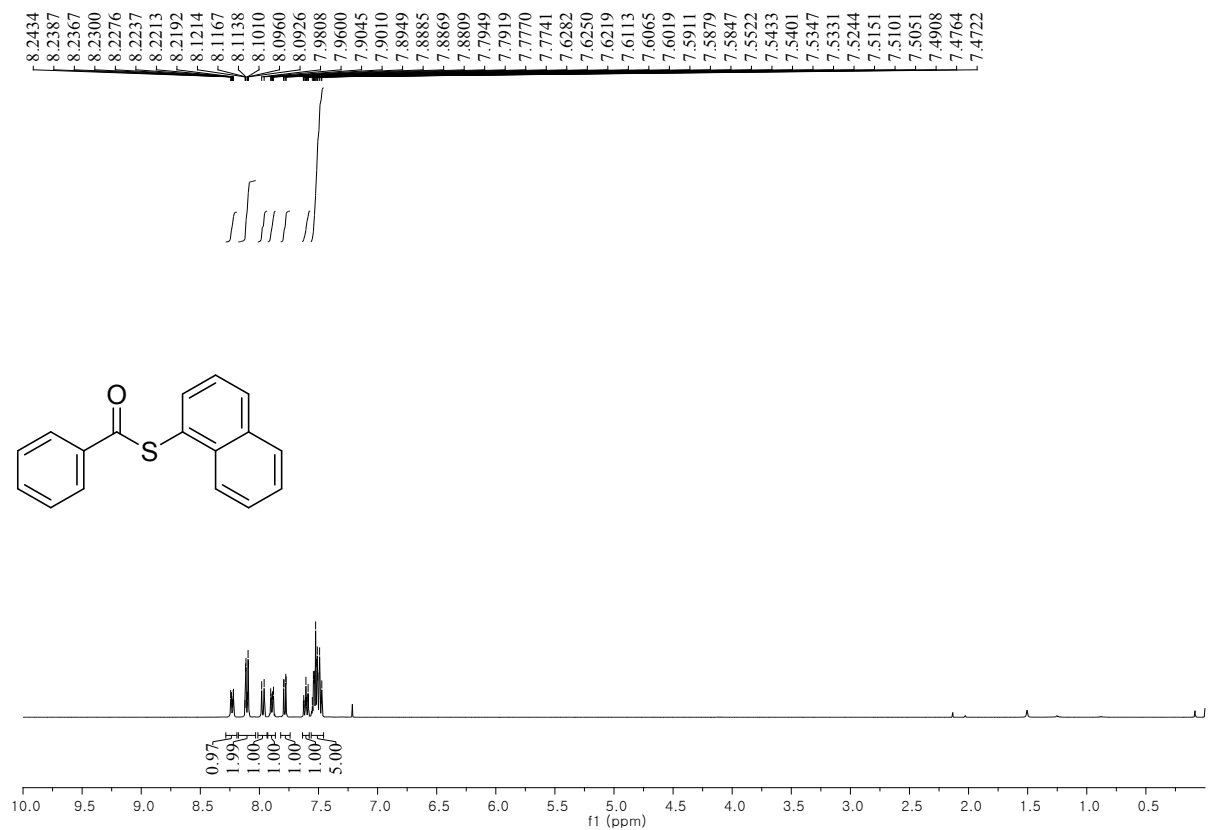




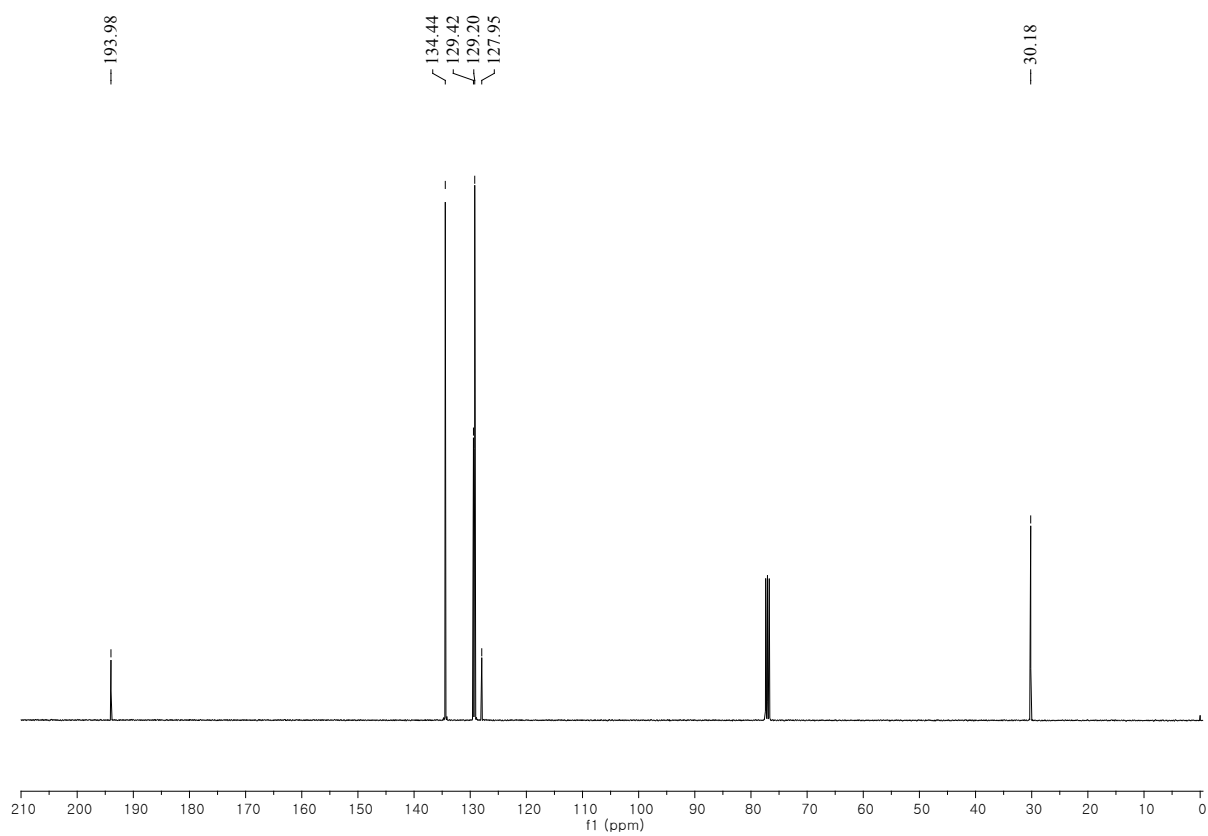
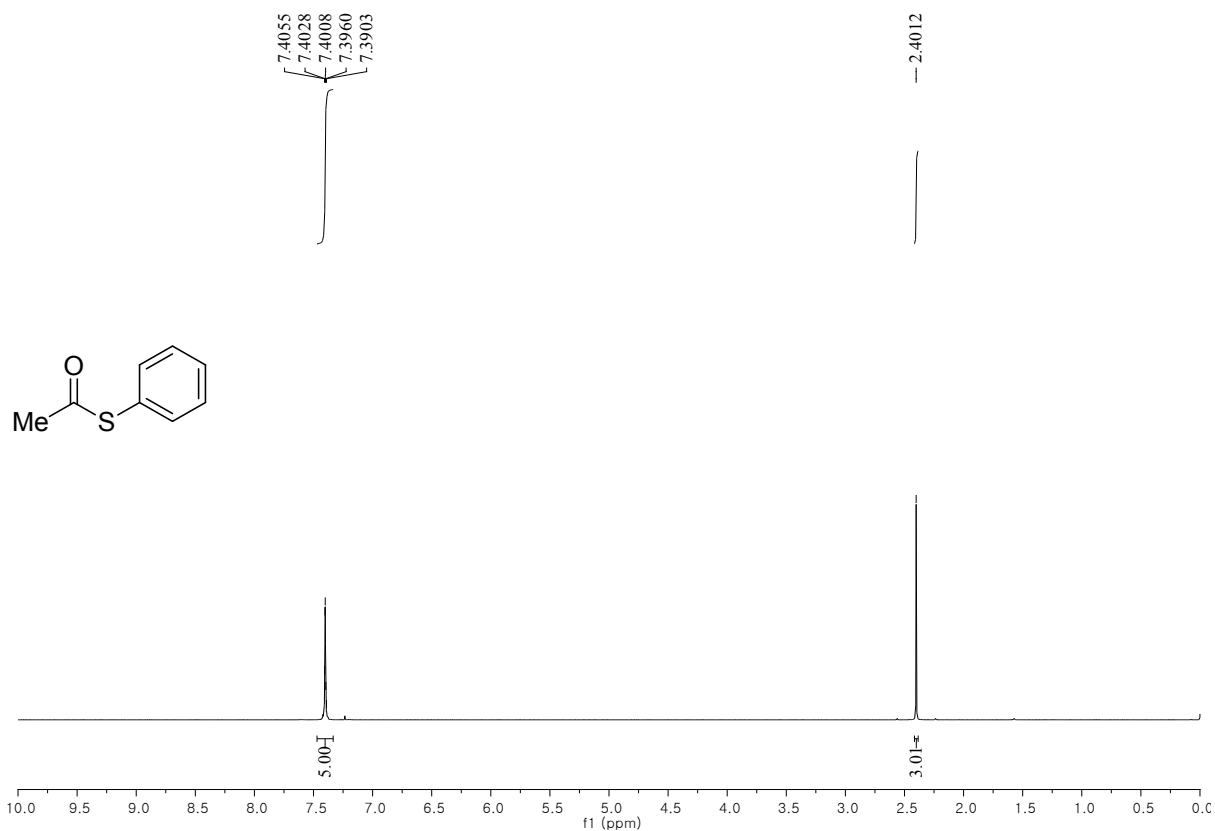
***S*-(4-Chlorophenyl) benzothioate (3ai)**



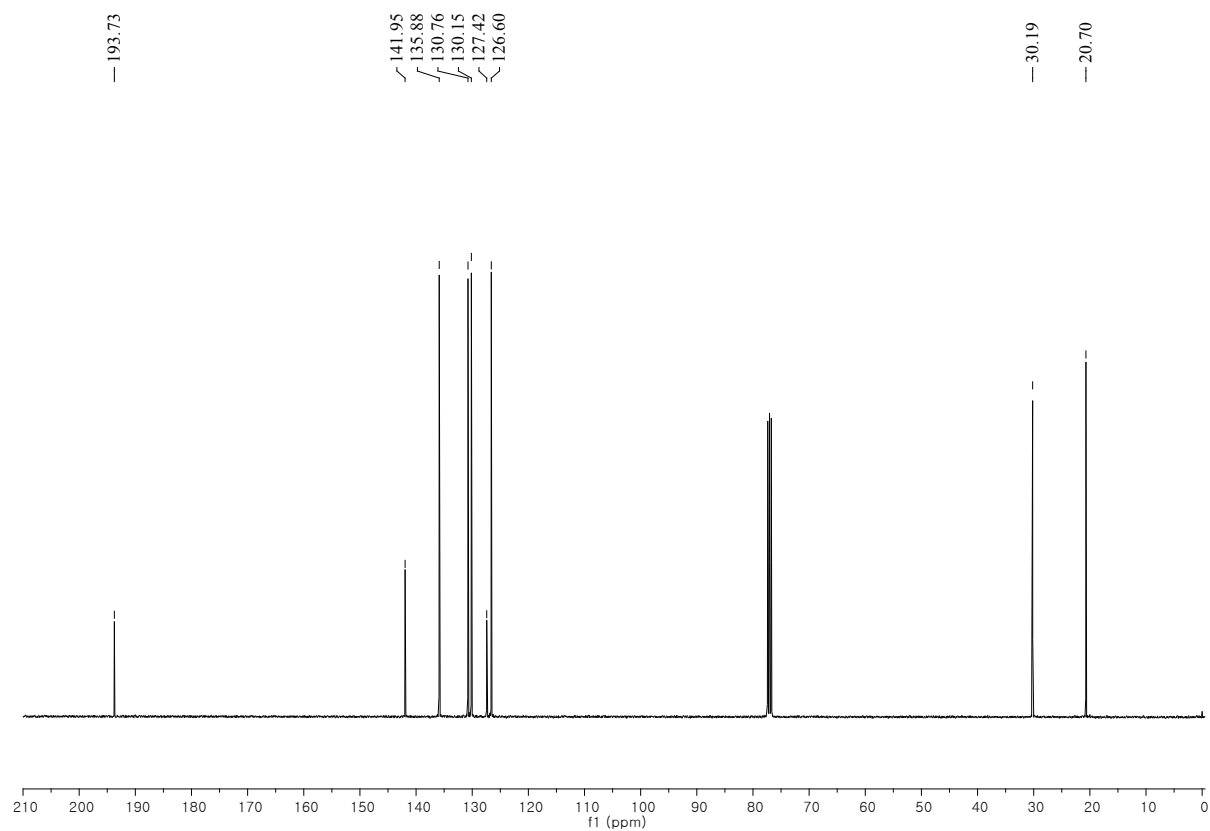
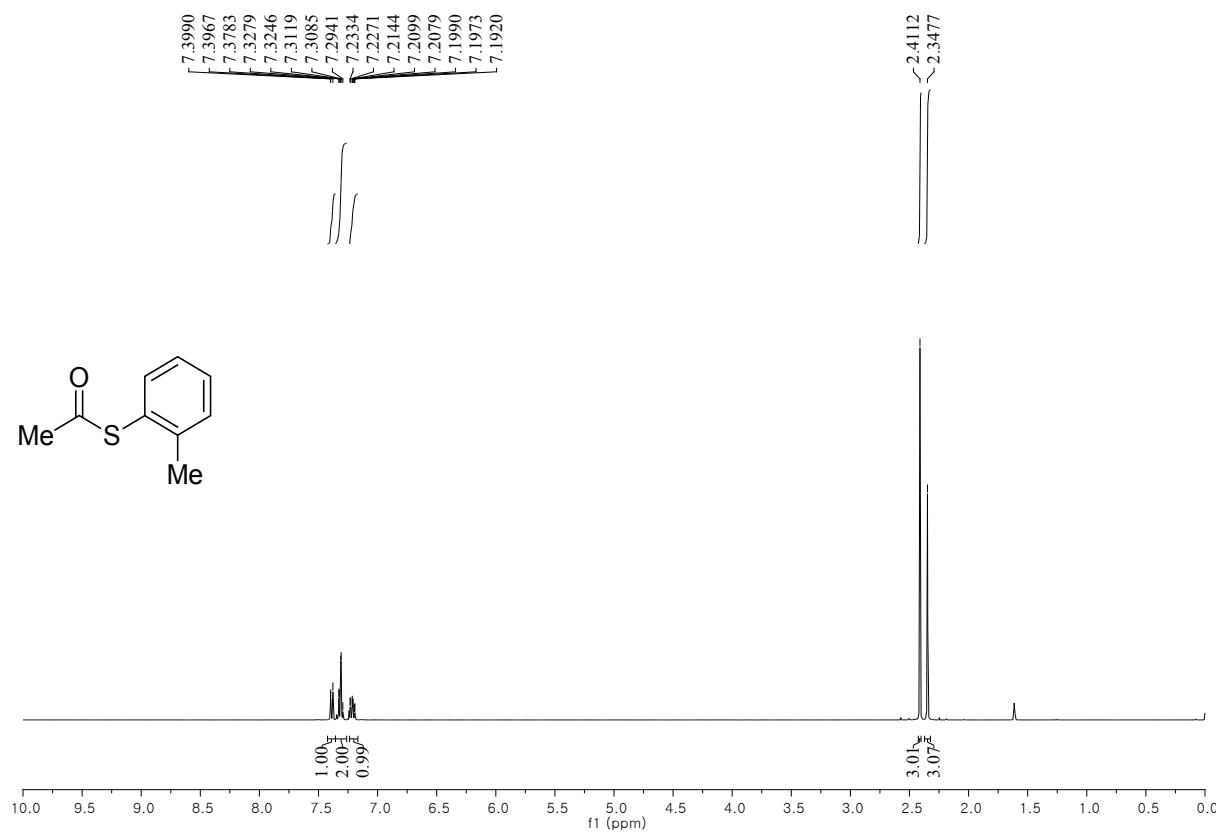
S-Naphthalen-1-yl benzothioate (3aj)



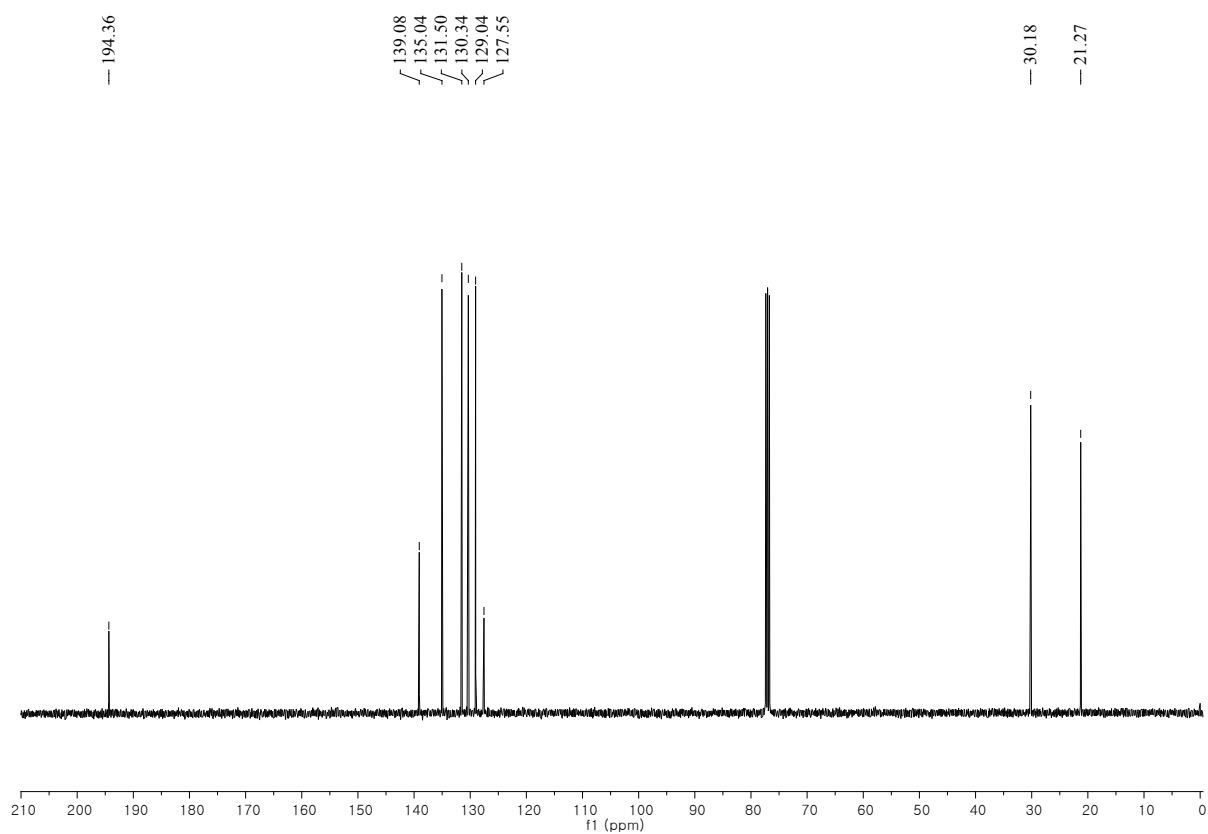
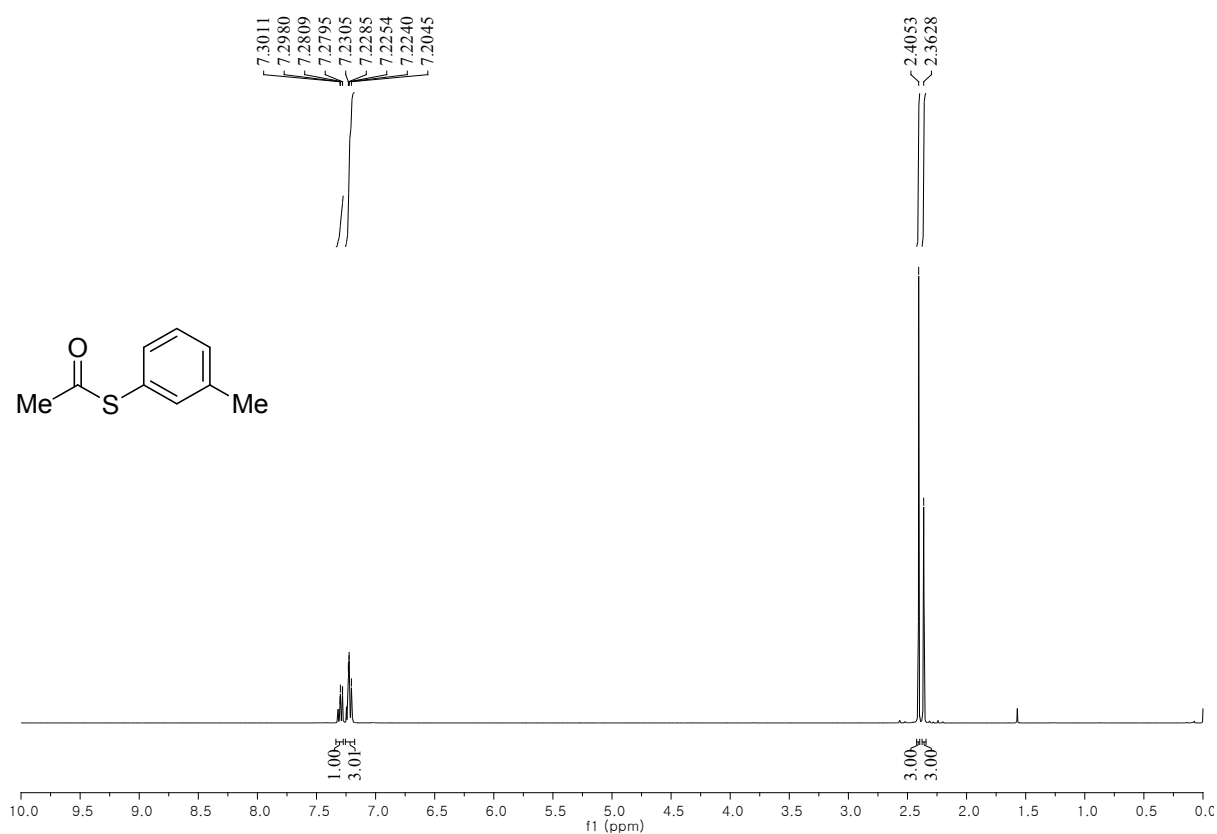
***S*-Phenyl thioacetate (2a)**



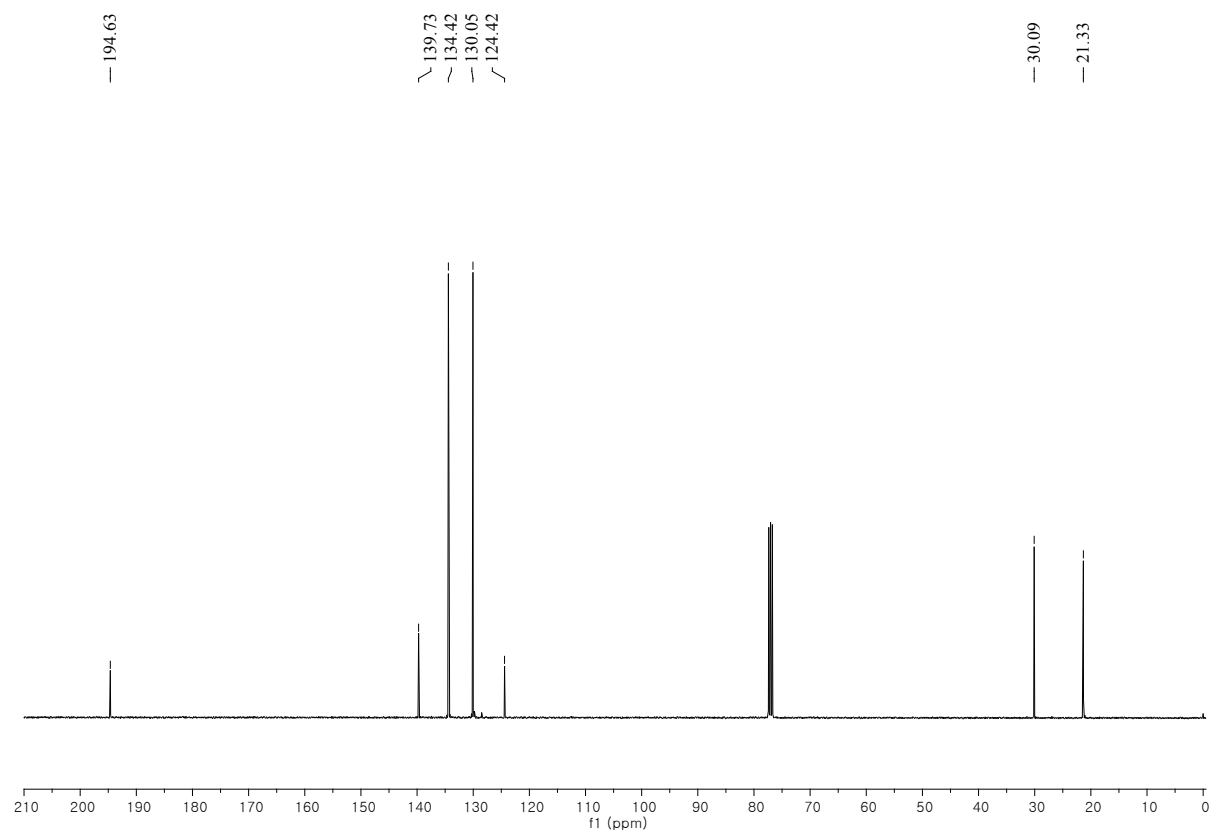
***S*-o-Tolyl ethanethioate (2b)**



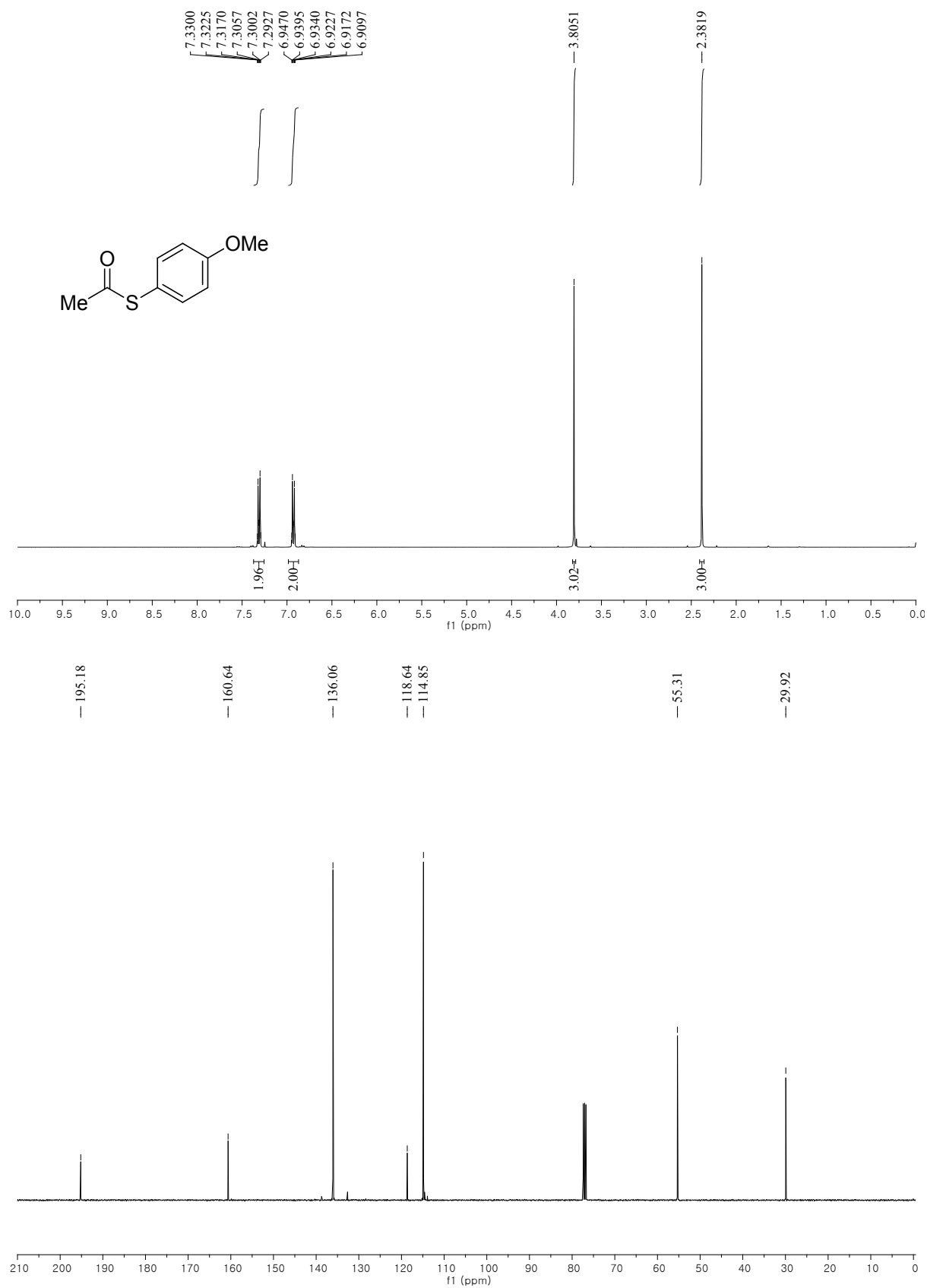
***S*-m-Tolyl ethanethioate (2c)**



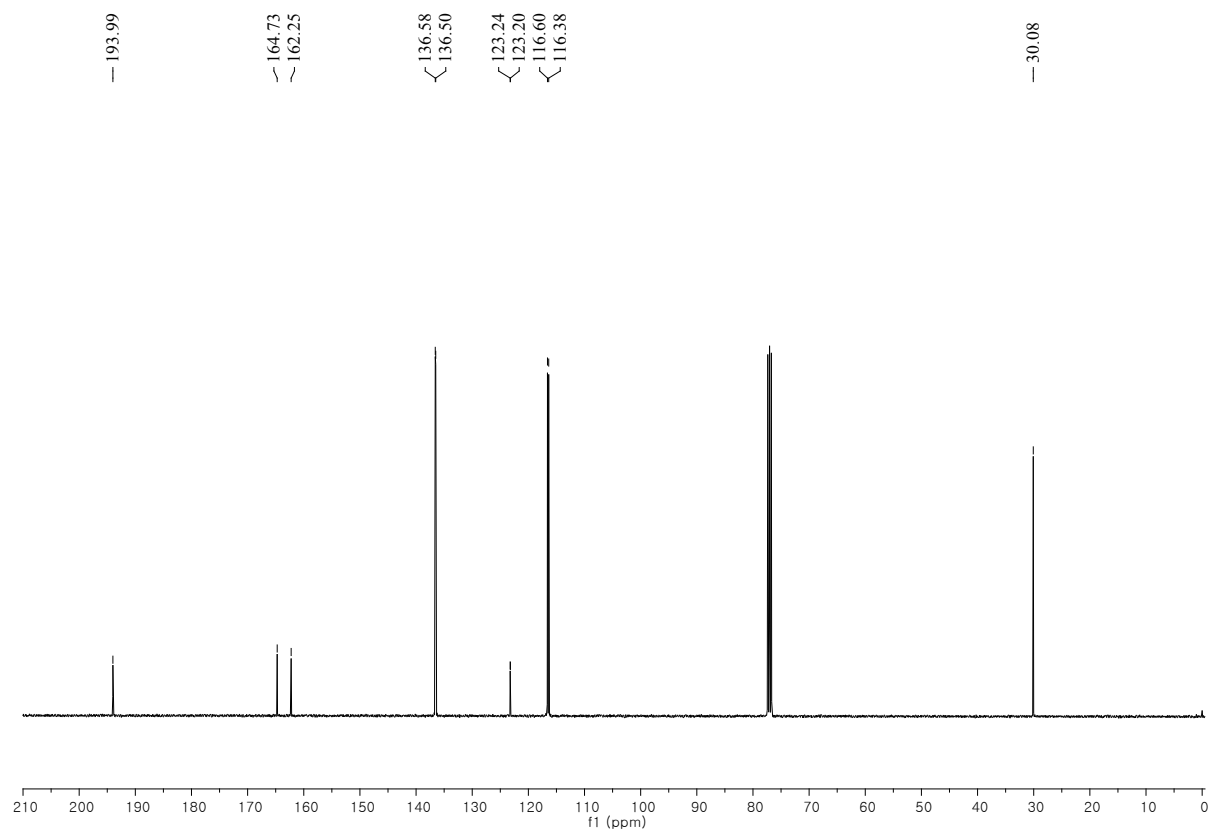
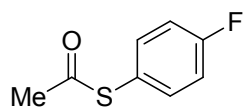
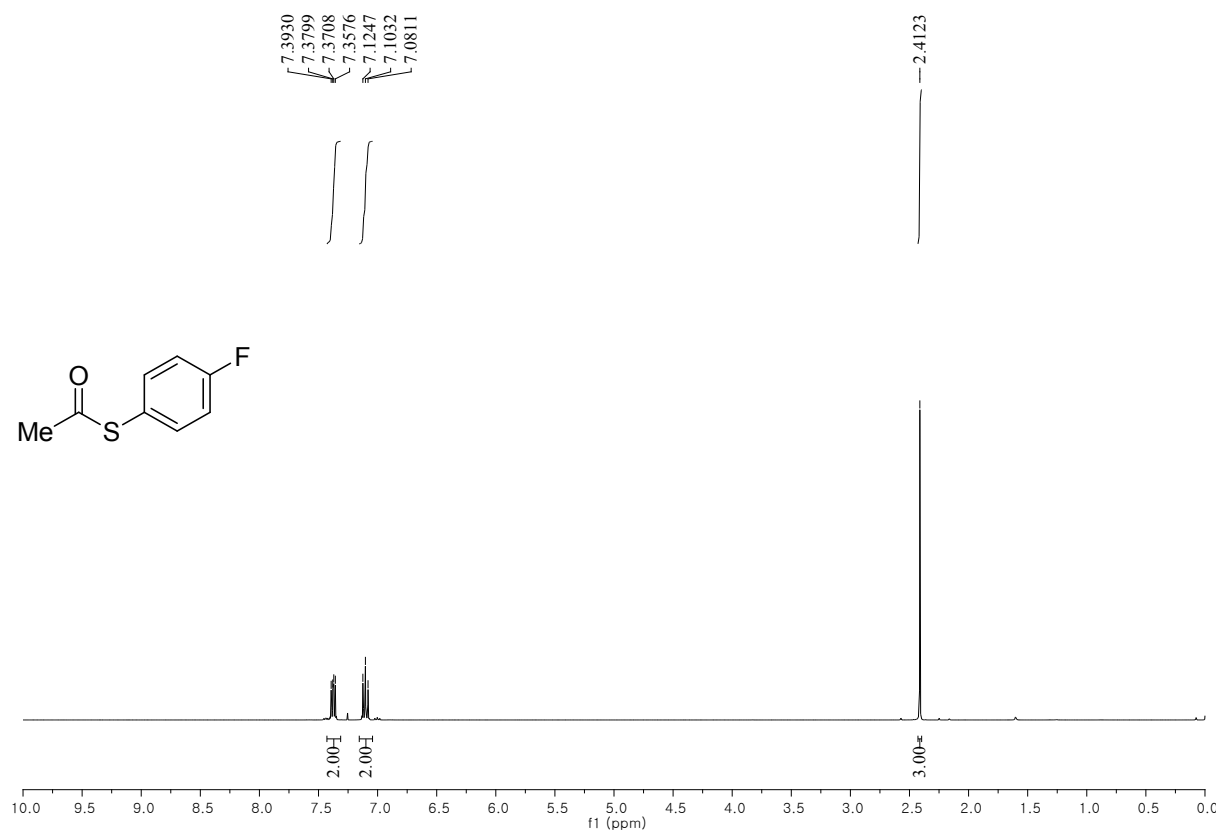
***S-p*-Tolyl ethanethioate (2d)**



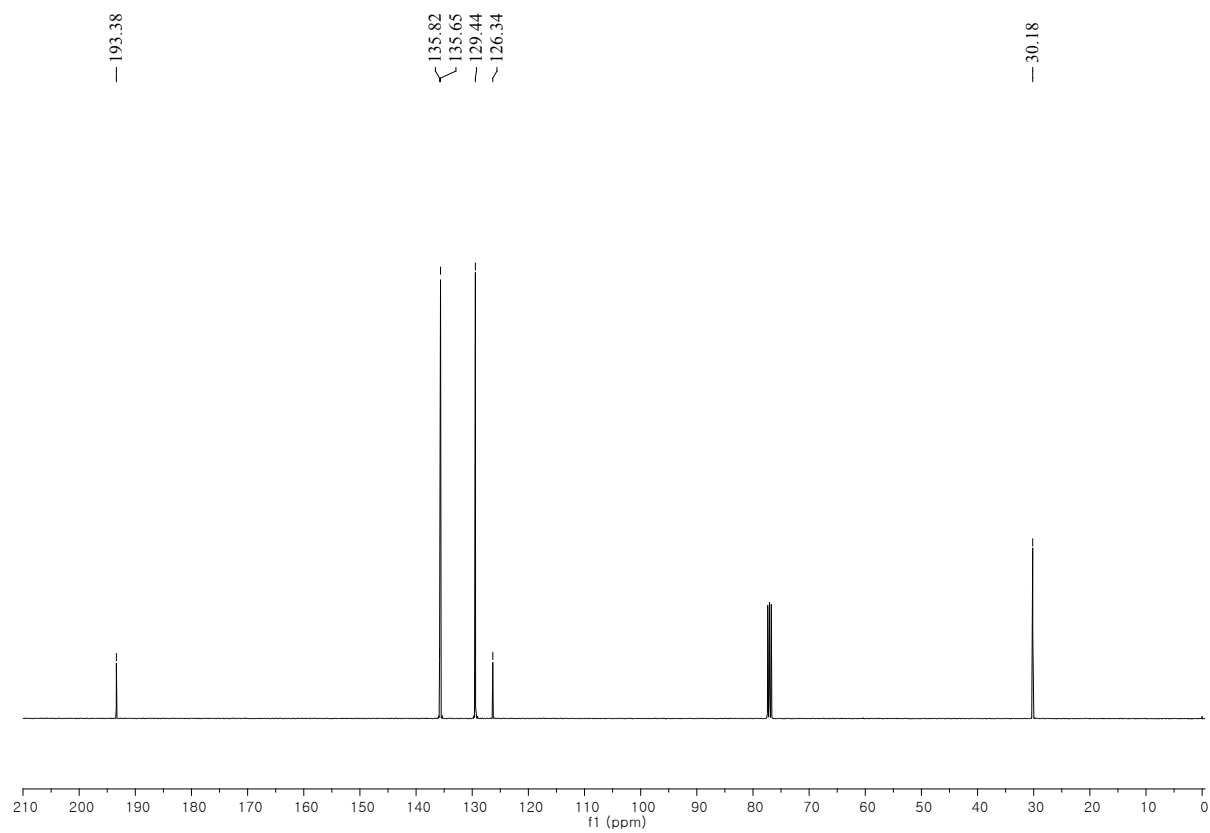
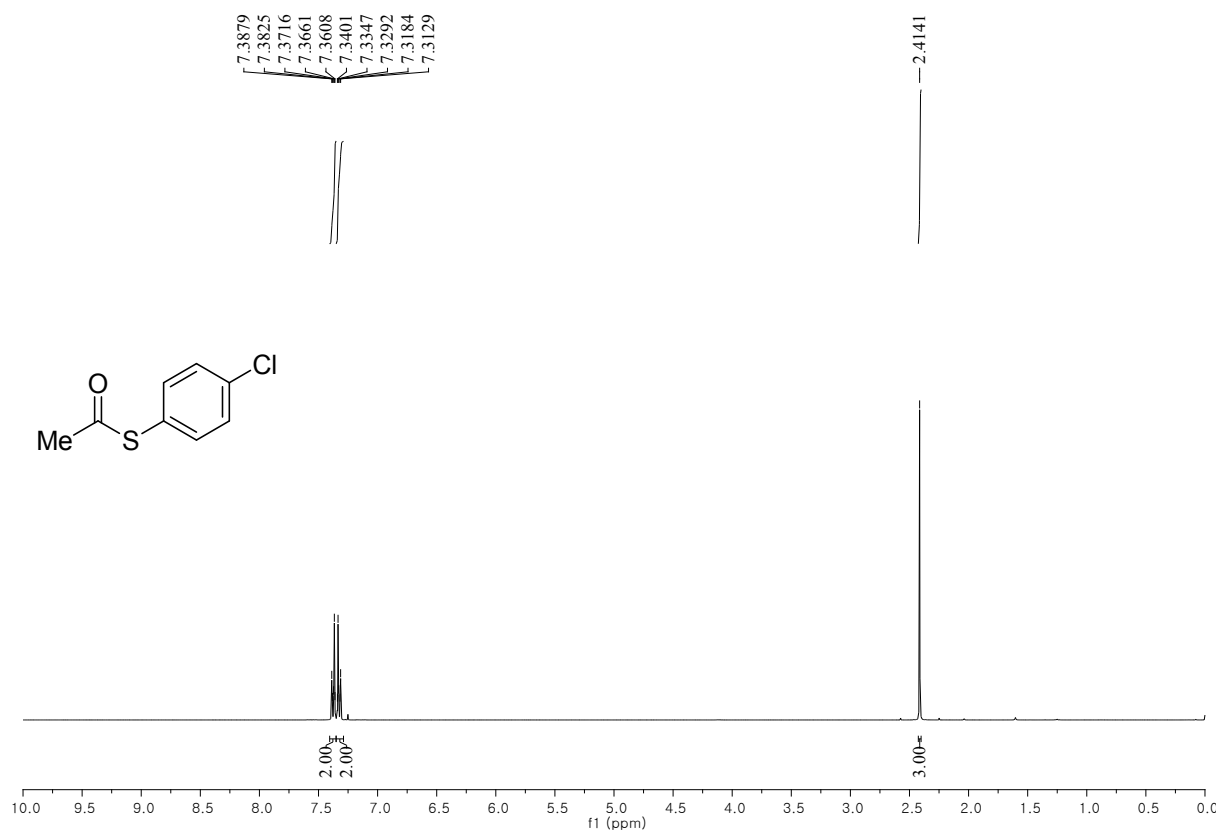
***S*-(4-methoxyphenyl) ethanethioate (2e)**



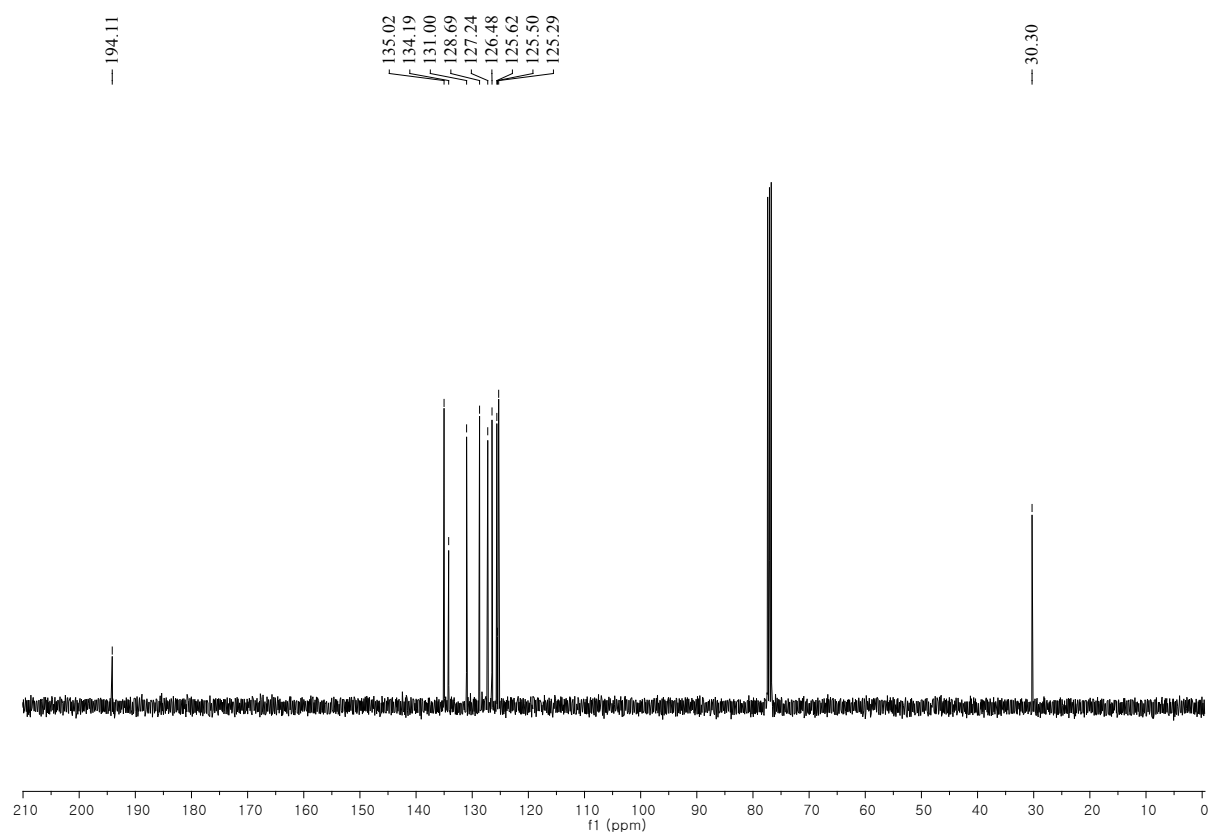
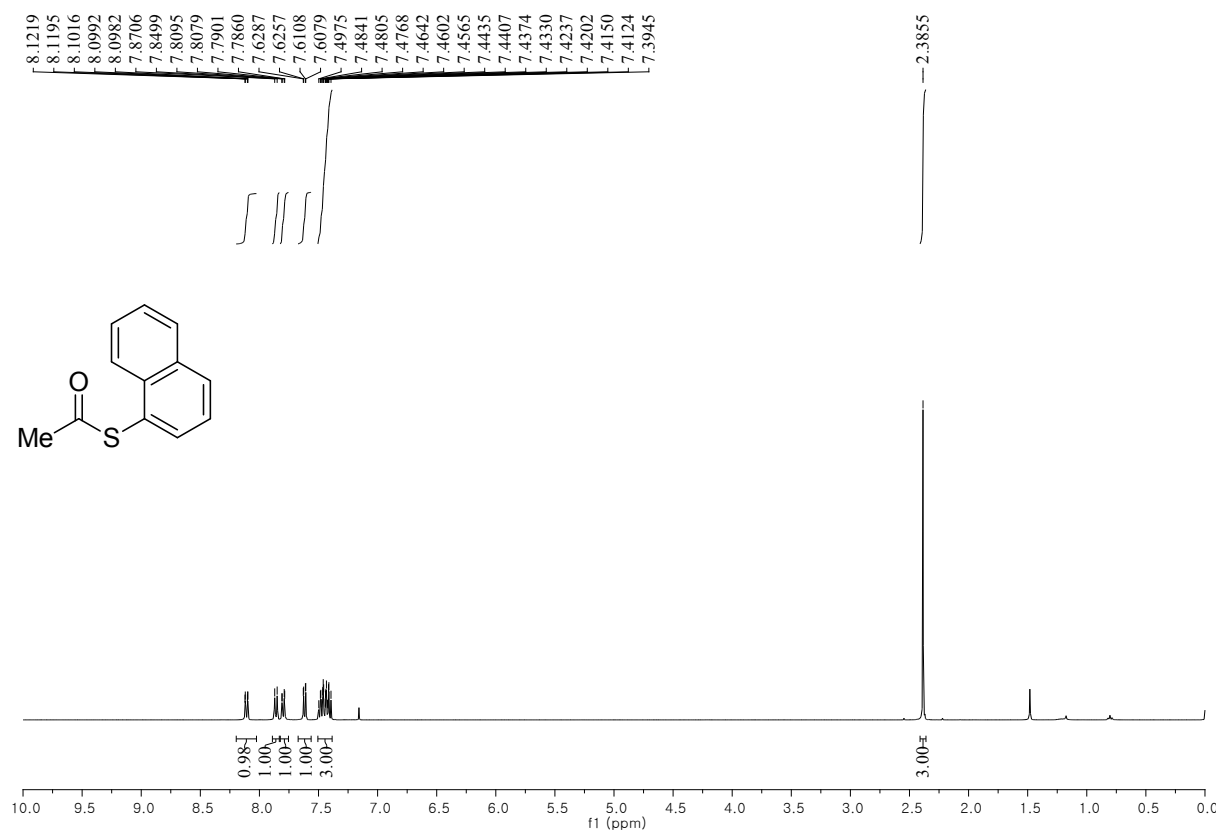
***S*-(4-Fluorophenyl) ethanethioate (2g)**



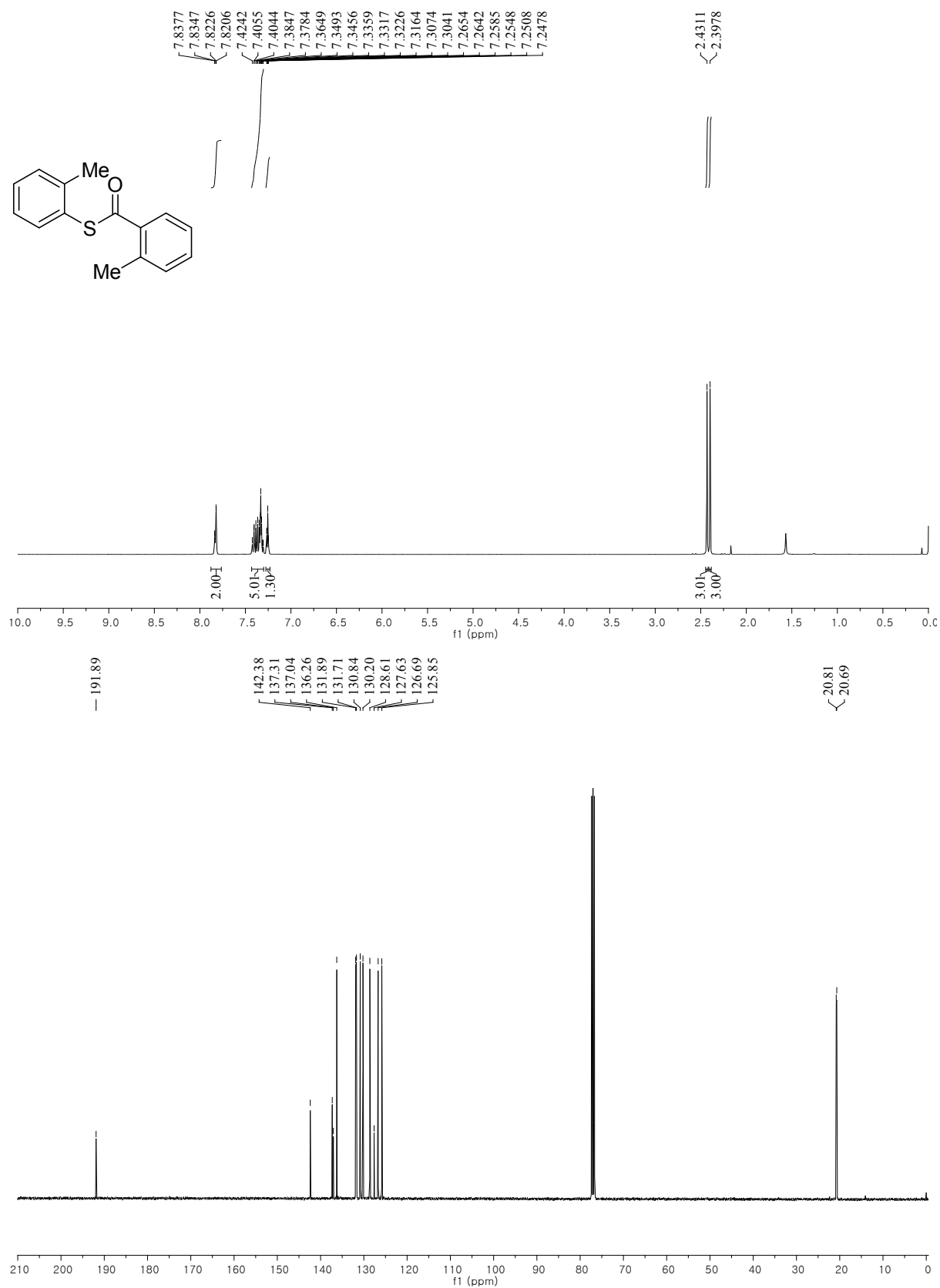
***S*-(4-Chlorophenyl) ethanethioate (2i)**



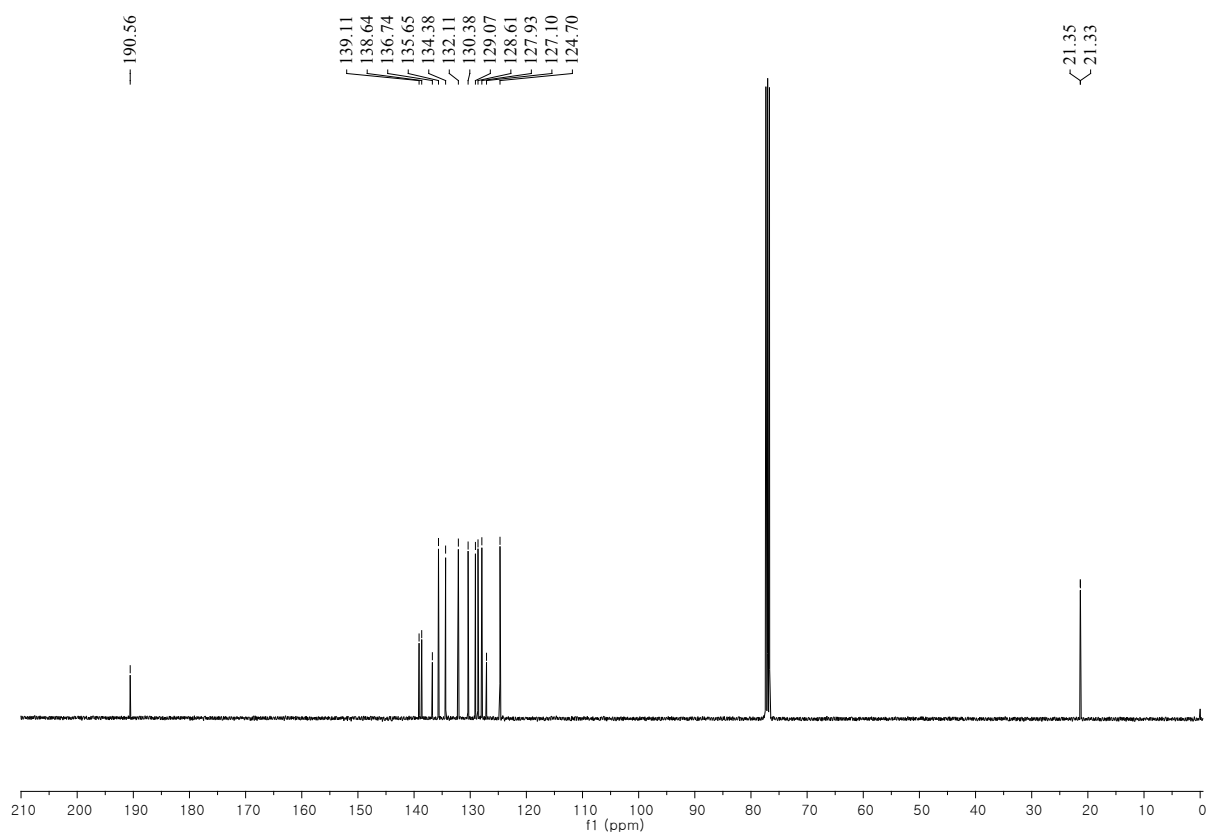
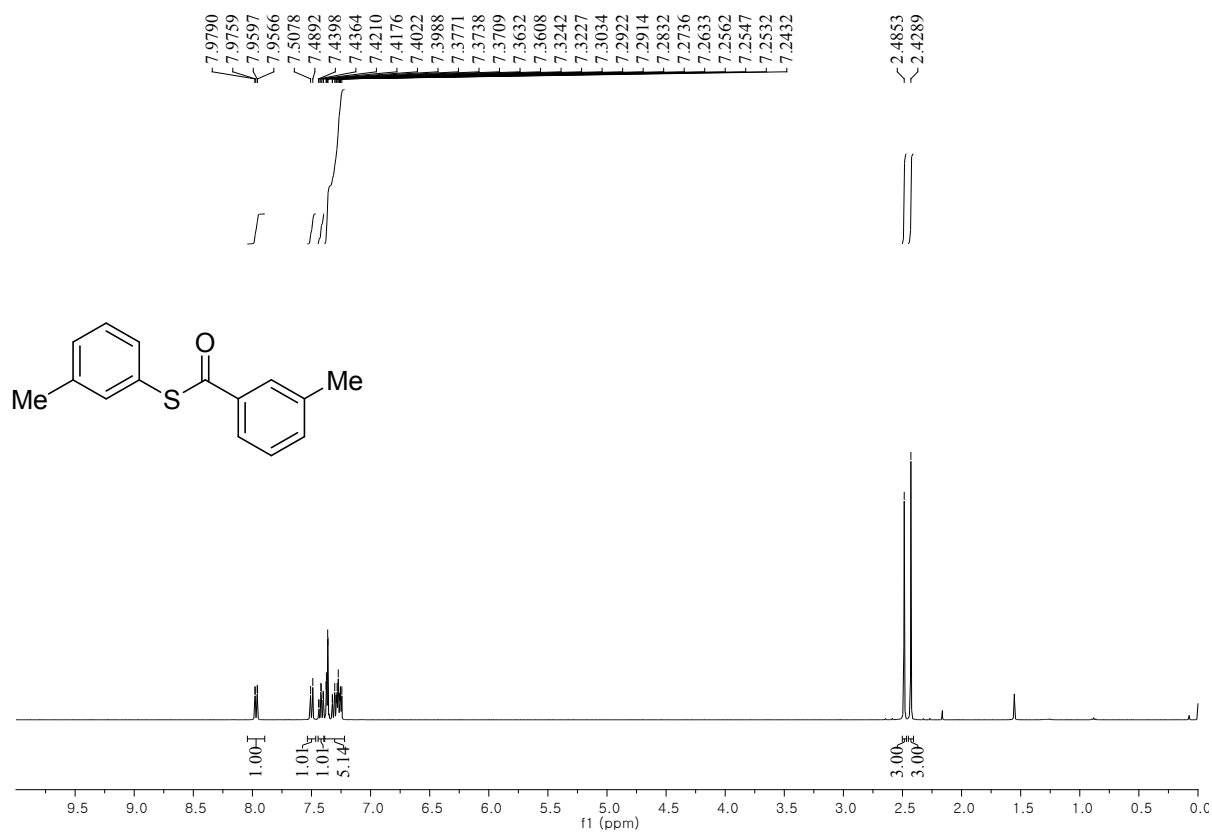
S-Naphthalen-1-yl ethanethioate (2o)



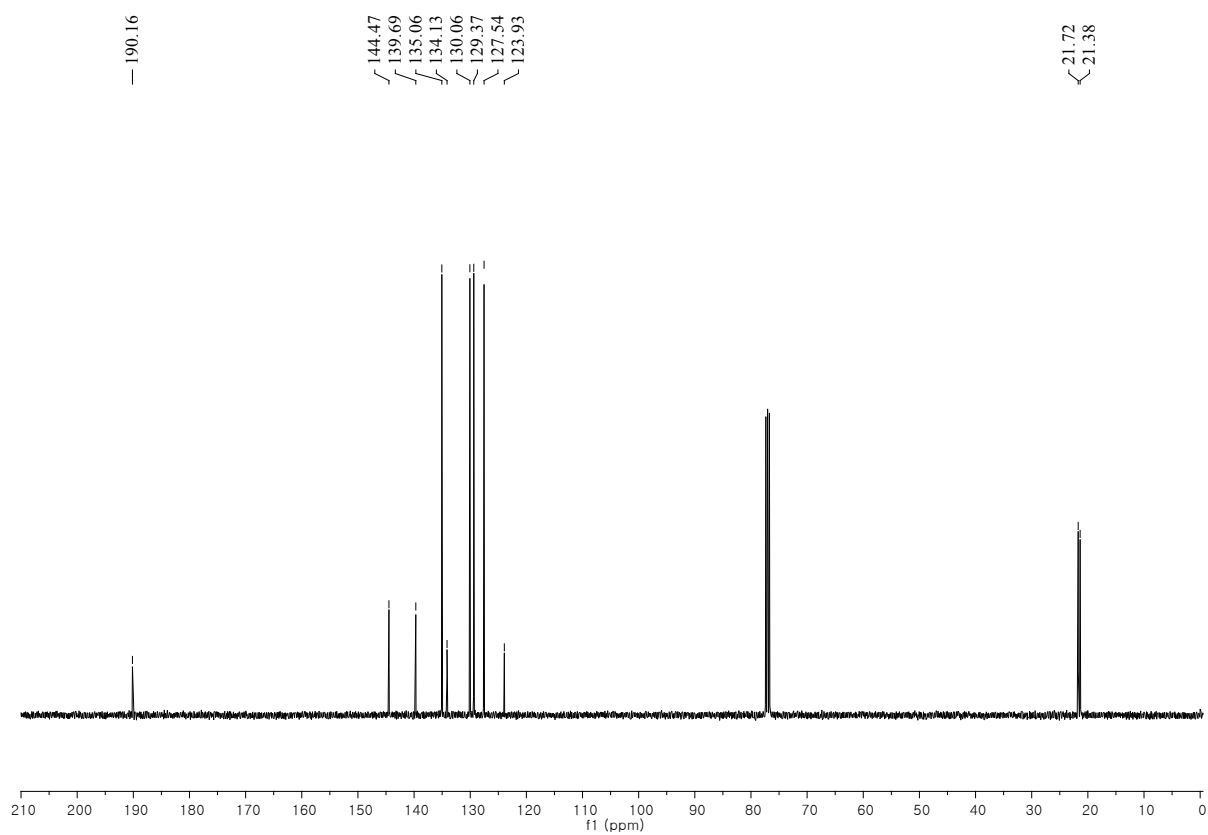
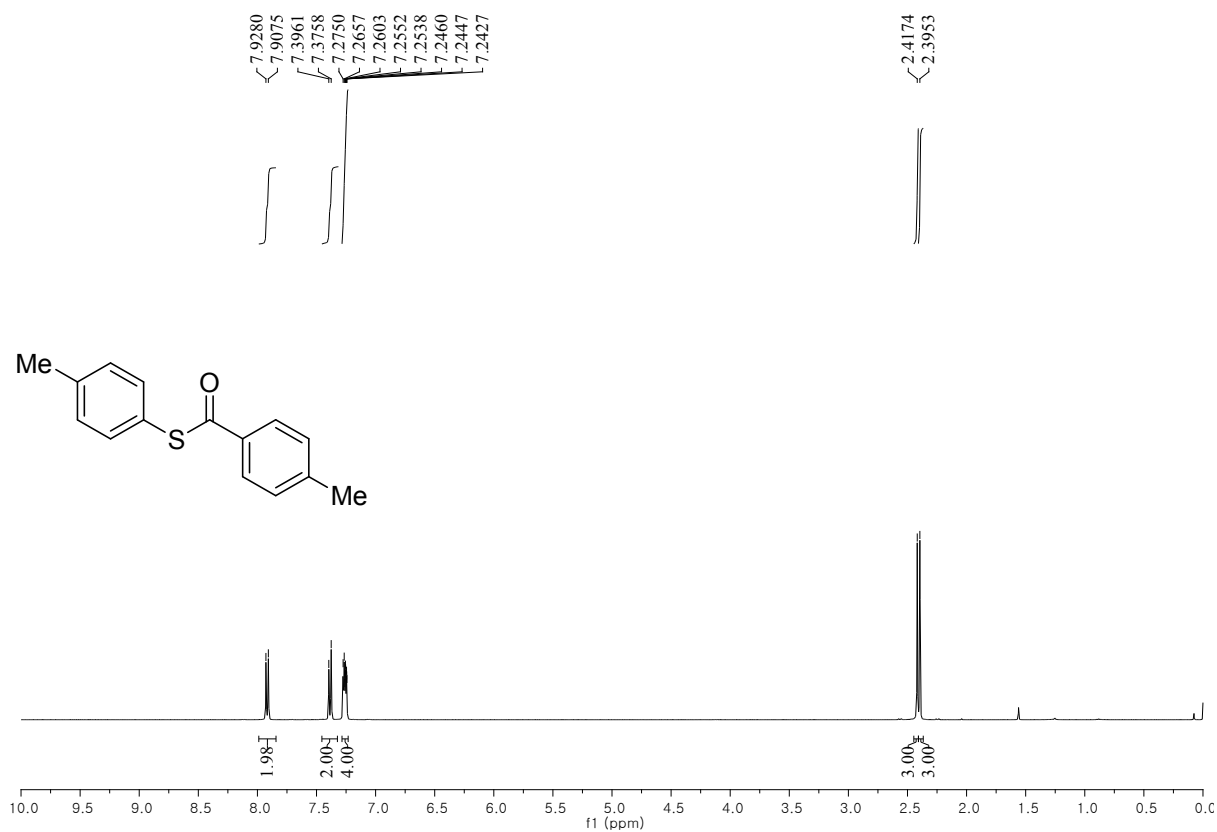
***S*-*o*-Tolyl 2-methylbenzothioate (3bb)**



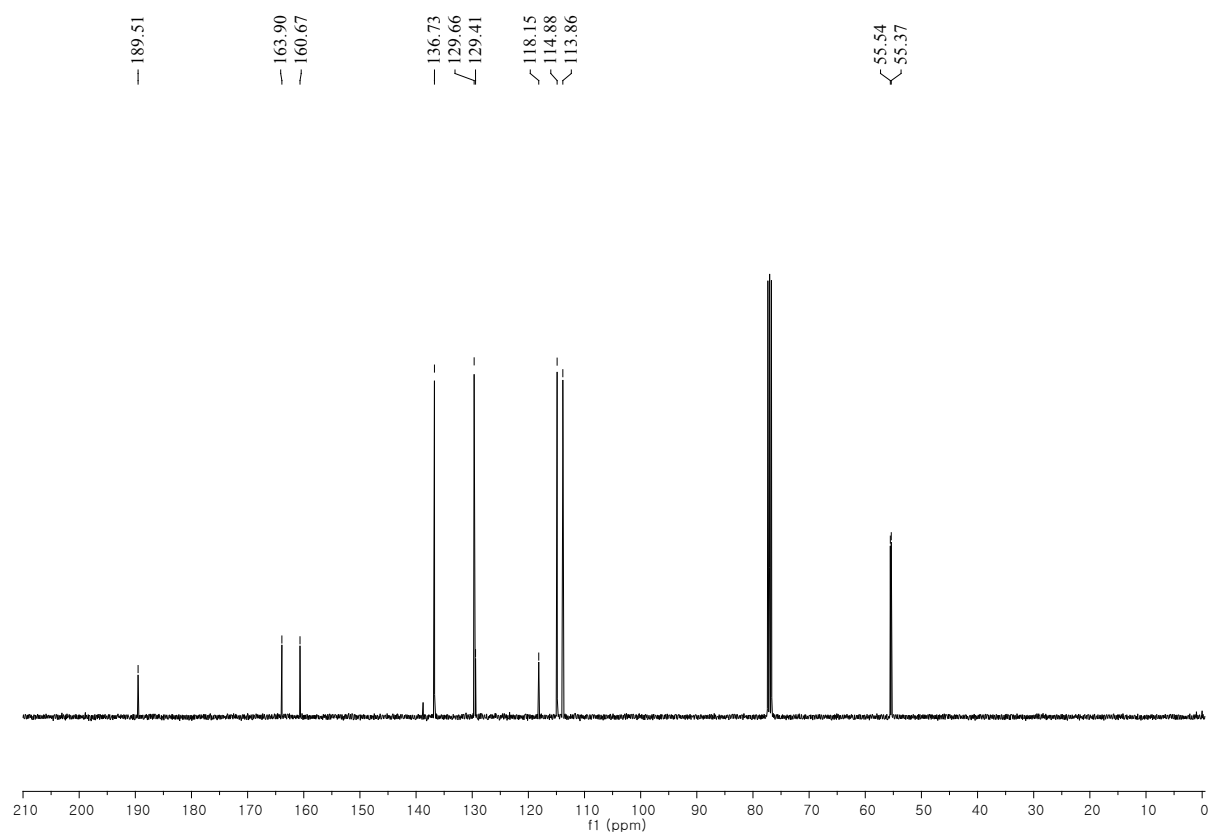
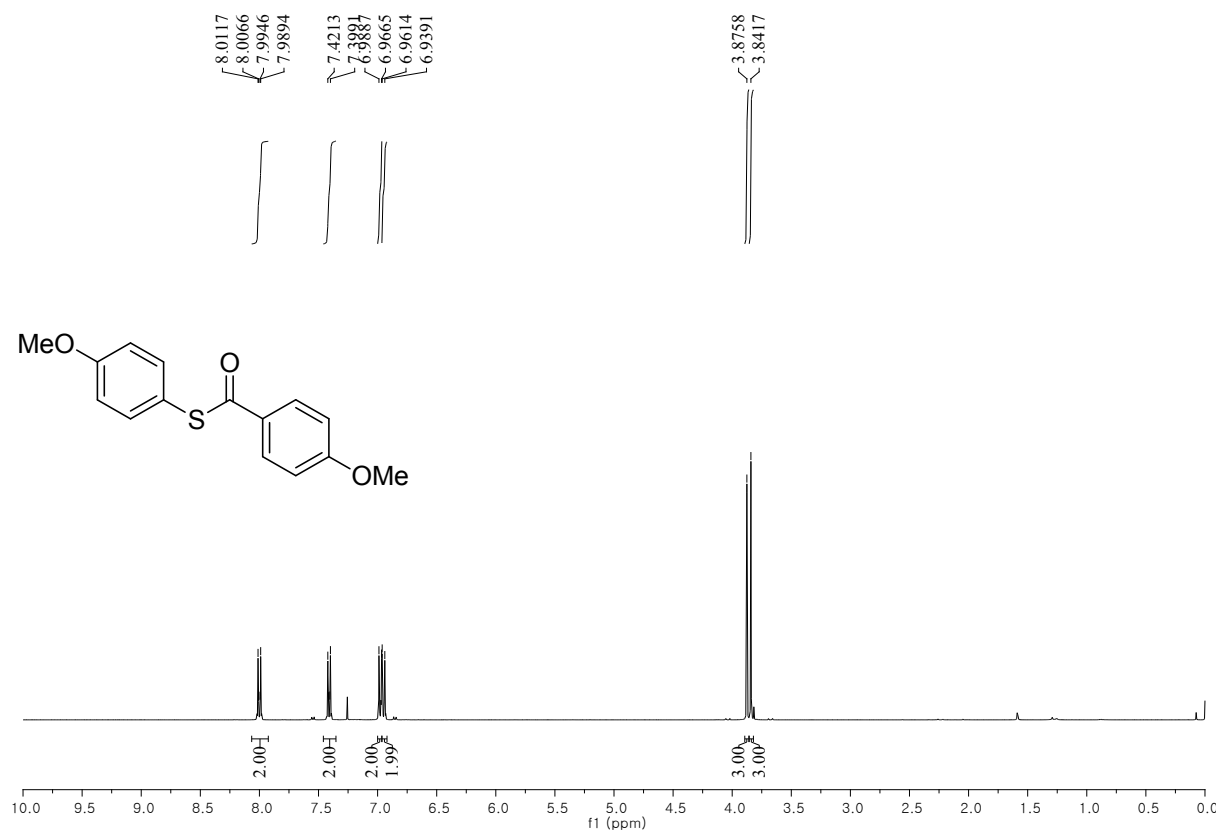
***S-m*-Tolyl 3-methylbenzothioate (3cc)**



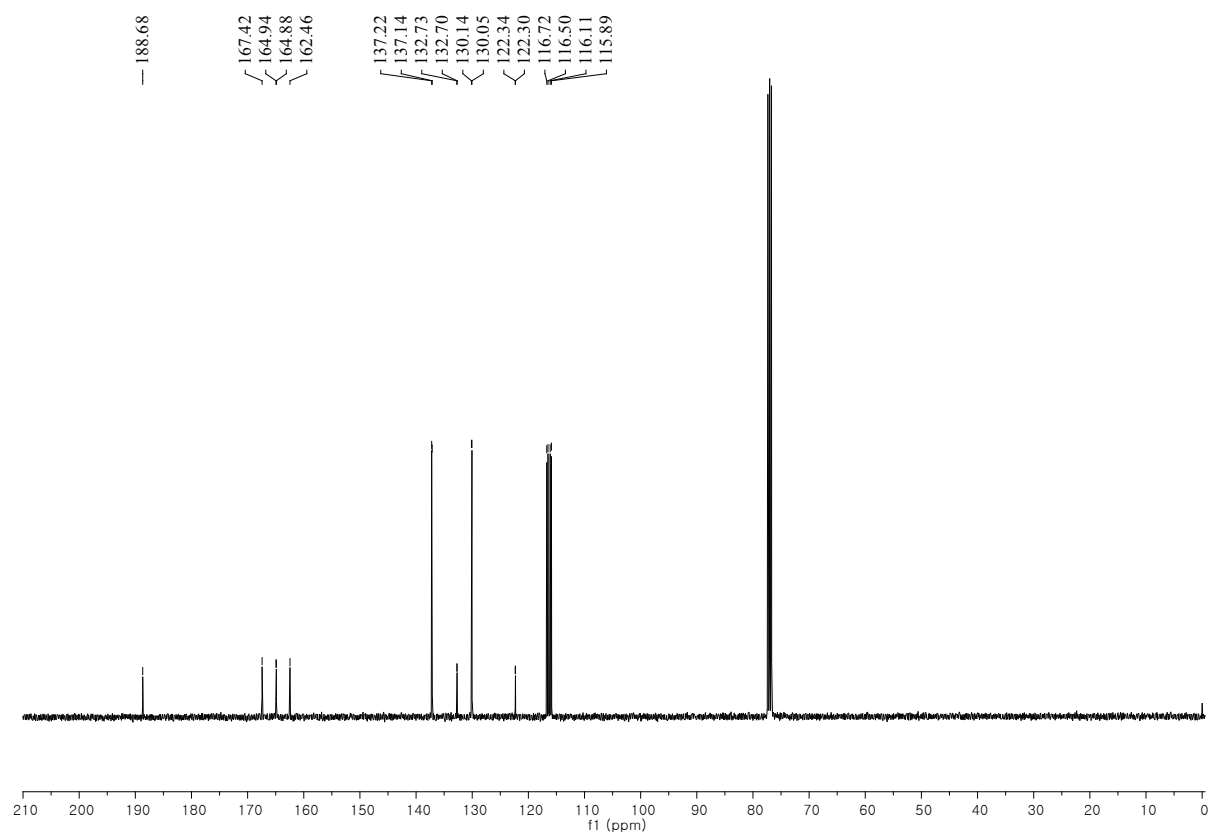
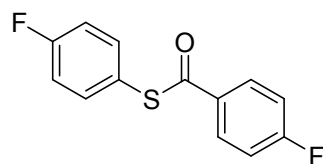
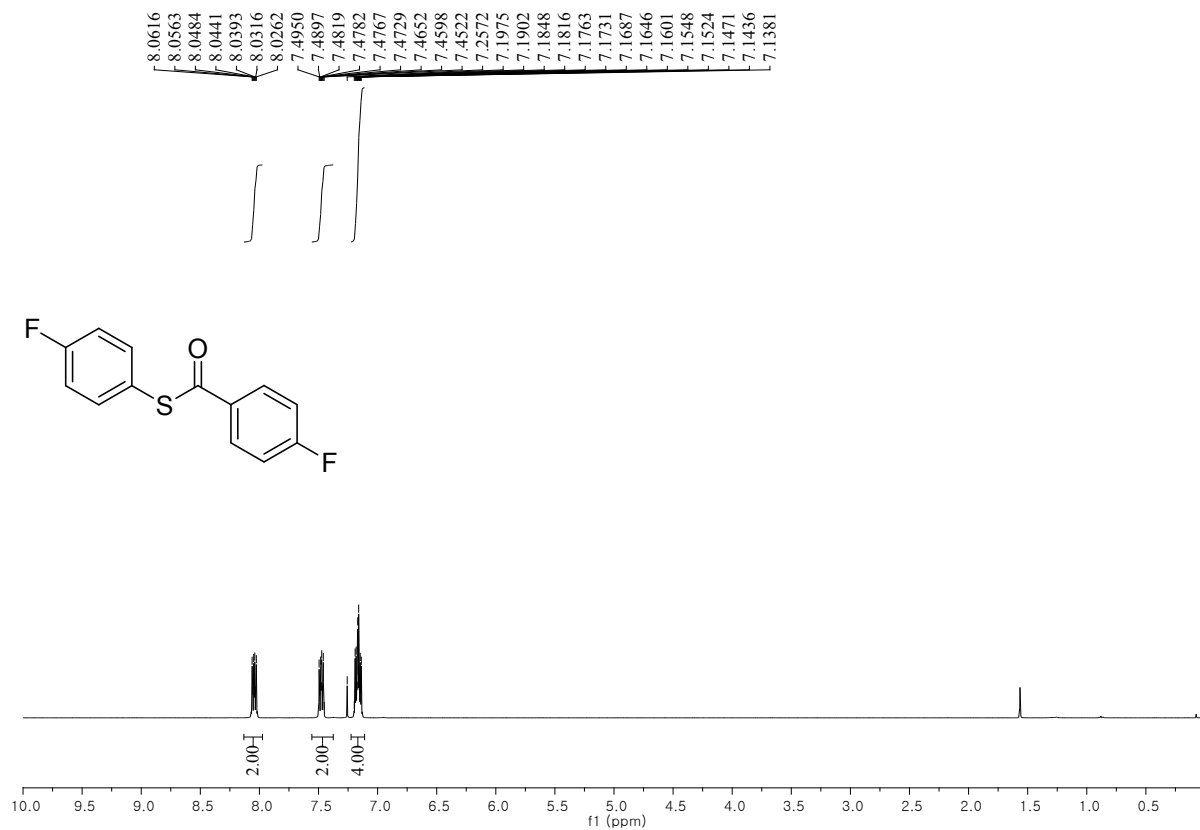
***S-p*-Tolyl 4-methylbenzothioate (3dd)**



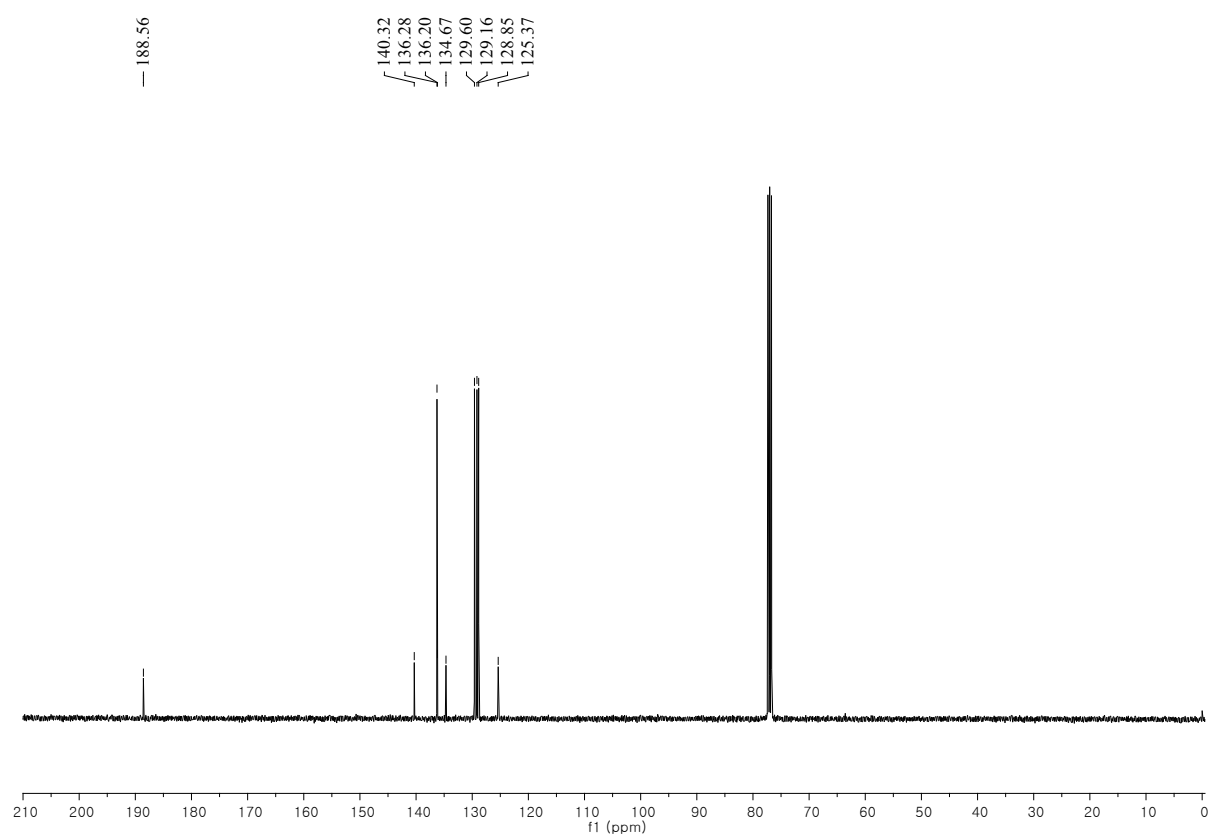
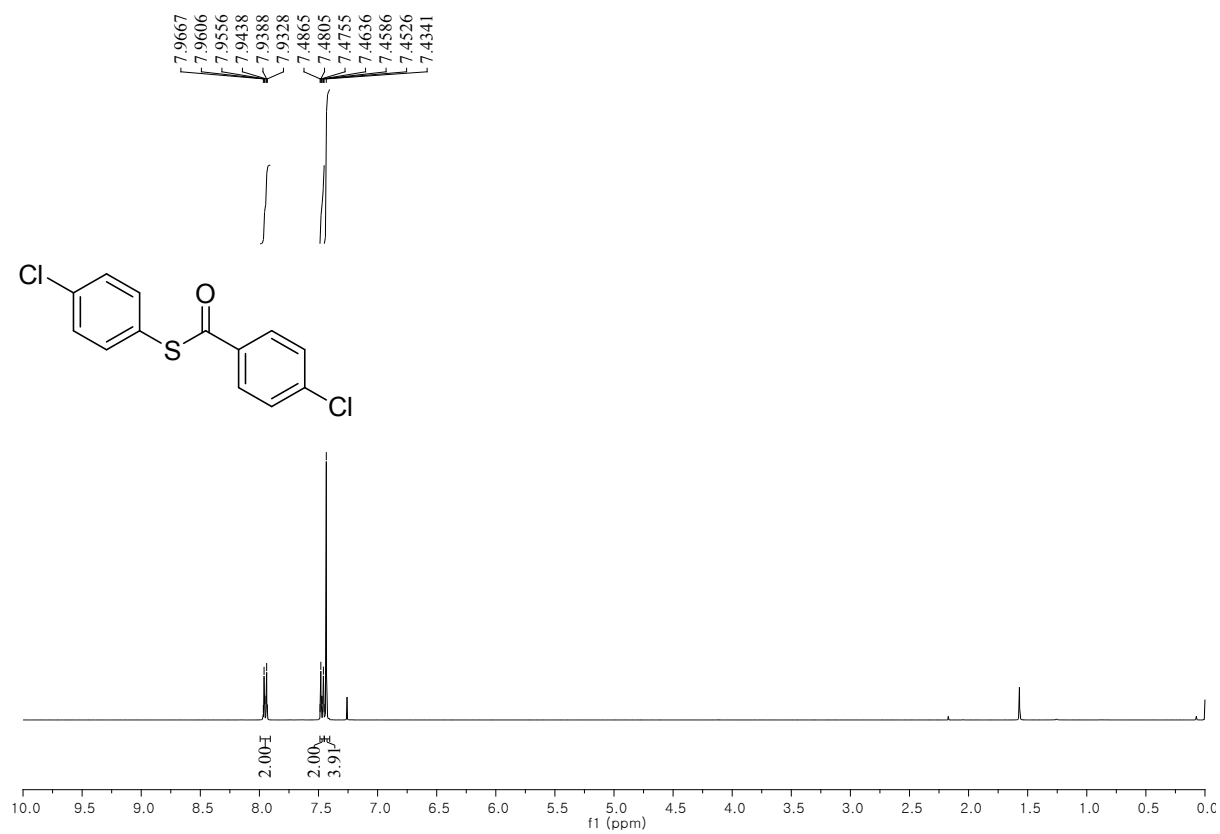
***S*-(4-Methoxyphenyl) 4-methoxybenzothioate (3ee)**



***S*-(4-Fluorophenyl) 4-fluorobenzothioate (3gg)**



***S*-(4-Chlorophenyl) 4-chlorobenzothioate (3ii)**



S-Naphthalen-1-yl naphthalene-1-carbothioate (3oo)

