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Supporting information

Dimethyl Sulfoxide as a Mild Oxidant in S-P(O) Bond Construction: Simple and Metal-Free Approaches to Phosphinothioates

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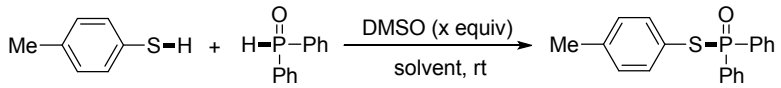
Optimization of reaction conditions

Table S1. Oxidative dehydrogenative phosphorylation of 4-methylbenzenethiol 1a with diphenylphosphine oxide 2a in the presence of DMSO as a stoichiometric oxidant under different conditions[a]



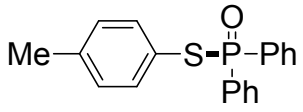
1a **2a** **3aa**

Entry	x	Reaction time	Solvent	Yield[b] (%)
1	2	12	DCM	13
2	5	12	DCM	15
3	10	12	DCM	25
4	20	12	DCM	34
5	20	12	Acetone	26
6	20	12	THF	25
7	20	12	EtOAc	40
8	20	12	CH_3CN	32
9	20	24	EtOAc	44
10	20	48	EtOAc	63
11	20	60	EtOAc	69
12	20	72	EtOAc	76

[a] Reaction conditions: **1a** (0.60 mmol), **2a** (0.30 mmol), and DMSO in solvent (1.5 mL) at room temperature under N_2. [b] Isolated yields.

Physical data of the compounds

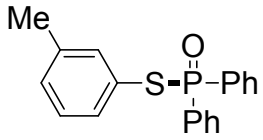
S-p-tolyl diphenylphosphinothioate (3aa)[1]



According to **GP1** with 4-methylbenzenethiol **1a** (74.5 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum

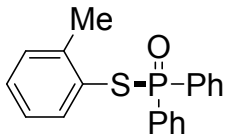
ether/EtOAc = 3/1) to afford the desired product **3aa** as white solid (91.3 mg, 94%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.7 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.8 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3aa** as white solid (77.5 mg, 80%). Mp: 106-108 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.88-7.81 (m, 4H), 7.49-7.40 (m, 6H), 7.35-7.29 (m, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 2.22 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.0 (d, *J* = 2.2 Hz), 135.2 (d, *J* = 3.9 Hz), 132.7 (d, *J* = 106.1 Hz), 132.1 (d, *J* = 3.3 Hz), 131.5 (d, *J* = 9.9 Hz), 129.8 (d, *J* = 1.7 Hz), 128.4 (d, *J* = 13.2 Hz), 122.2 (d, *J* = 5.0 Hz), 21.0; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.36; **HRMS** (ESI) calculated for $C_{19}H_{18}OPS$ $[M+H]^+$ m/z 325.0810, found 325.0806.

***S*-*m*-tolyl diphenylphosphinothioate (3ba)**[2]



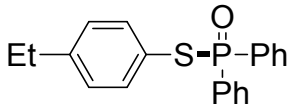
According to **GP1** with 3-methylbenzenethiol **1b** (75.0 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.4 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ba** as white solid (80.8 mg, 83%). According to **GP2** with 1,2-di-*m*-tolyldisulfane **4b** (44.8 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.6 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ba** as white solid (75.7 mg, 78%). Mp: 96-98 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86-7.82 (m, 4H), 7.49-7.46 (m, 2H), 7.43-7.39 (m, 4H), 7.26-7.22 (m, 2H), 7.07-7.02 (m, 2H), 2.20 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 138.7 (d, *J* = 1.7 Hz), 135.8 (d, *J* = 3.8 Hz), 132.4 (d, *J* = 106.1 Hz), 132.1 (d, *J* = 4.4 Hz), 132.0 (d, *J* = 2.8 Hz), 131.4 (d, *J* = 9.9 Hz), 129.6 (d, *J* = 2.3 Hz), 128.7 (d, *J* = 1.7 Hz), 128.3 (d, *J* = 12.7 Hz), 125.5 (d, *J* = 5.5 Hz), 20.9; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.35; **HRMS** (ESI) calculated for $C_{19}H_{18}OPS$ $[M+H]^+$ m/z 325.0810, found 325.0816.

***S*-*o*-tolyl diphenylphosphinothioate (3ca)**[3]



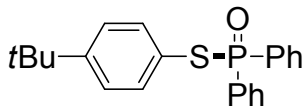
According to **GP1** with 2-methylbenzenethiol **1c** (75.1 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ca** as white solid (82.4 mg, 85%). According to **GP2** with 1,2-di-*o*-tolyldisulfane **4c** (44.5 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ca** as white solid (72.8 mg, 75%). Mp: 70-72 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.85-7.78 (m, 4H), 7.51-7.38 (m, 7H), 7.17-7.10 (m, 2H), 7.02-6.96 (m, 1H), 2.34 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 142.7 (d, *J* = 2.6 Hz), 136.6 (d, *J* = 3.8 Hz), 132.8 (d, *J* = 106.0 Hz), 132.1 (d, *J* = 2.8 Hz), 131.3 (d, *J* = 10.4 Hz), 130.6 (d, *J* = 1.7 Hz), 129.1 (d, *J* = 2.2 Hz), 128.3 (d, *J* = 13.2 Hz), 126.3 (d, *J* = 1.7 Hz), 125.3 (d, *J* = 5.0 Hz), 21.2; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.18; **HRMS** (ESI) calculated for $C_{19}H_{18}OPS$ $[M+H]^+$ m/z 325.0810, found 325.0807.

***S*-(4-ethylphenyl) diphenylphosphinothioate (3da)**[3]



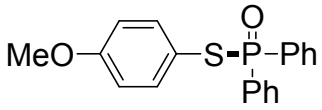
According to **GP1** with 4-ethylbenzenethiol **1d** (83.0 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.2 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3da** as white solid (85.8 mg, 85%). Mp: 84-86 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.88-7.81 (m, 4H), 7.51-7.39 (m, 6H), 7.37-7.31 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 2H), 2.54 (q, *J* = 7.6 Hz, 2H), 1.14 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.3 (d, *J* = 2.7 Hz), 135.3 (d, *J* = 3.8 Hz), 132.6 (d, *J* = 106.1 Hz), 132.1 (d, *J* = 2.8 Hz), 131.5 (d, *J* = 10.4 Hz), 128.6 (d, *J* = 1.7 Hz), 128.4 (d, *J* = 13.2 Hz), 122.4 (d, *J* = 5.6 Hz), 28.3, 15.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.49; **HRMS** (ESI) calculated for $C_{20}H_{20}OPS$ $[M+H]^+$ m/z 339.0967, found 339.0959.

***S*-(4-(*tert*-butyl)phenyl) diphenylphosphinothioate (3ea)**[1]



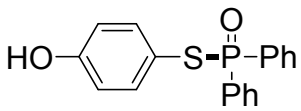
According to **GP1** with 4-(*tert*-butyl)benzenethiol **1e** (100.0 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.8 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ea** as white solid (91.6 mg, 83%). Mp: 119-121 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.87-7.80 (m, 4H), 7.50-7.34 (m, 8H), 7.22-7.19 (m, 2H), 1.22 (s, 9H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 152.1 (d, *J* = 2.2 Hz), 135.1 (d, *J* = 3.8 Hz), 132.7 (d, *J* = 106.1 Hz), 132.1 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 9.8 Hz), 128.3 (d, *J* = 13.2 Hz), 126.1 (d, *J* = 1.7 Hz), 122.3 (d, *J* = 5.0 Hz), 34.4, 31.0; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.56; **HRMS** (ESI) calculated for $C_{22}H_{24}OPS$ $[M+H]^+$ m/z 367.1280, found 367.1282.

***S*-(4-methoxyphenyl) diphenylphosphinothioate (3fa)**[1]



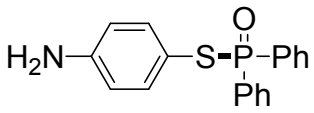
According to **GP1** with 4-methoxybenzenethiol **1f** (84.0 mg, 0.6 mmol, 2.0 equiv), and diphenylphosphine oxide **2a** (61.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3fa** as white solid (92.0 mg, 90%). According to **GP2** with 1,2-bis(4-methoxyphenyl)disulfane **4f** (50.7 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3fa** as white solid (93.5 mg, 92%). Mp: 132-134 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.87-7.80 (m, 4H), 7.50-7.38 (m, 6H), 7.34-7.31 (m, 2H), 6.74-6.68 (m, 2H), 3.69 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 160.3 (d, *J* = 2.3 Hz), 136.9 (d, *J* = 3.8 Hz), 132.6 (d, *J* = 105.5 Hz), 132.1 (d, *J* = 3.3 Hz), 131.5 (d, *J* = 10.4 Hz), 128.4 (d, *J* = 13.2 Hz), 115.9 (d, *J* = 5.5 Hz), 114.7 (d, *J* = 1.7 Hz), 55.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.39; **HRMS** (ESI) calculated for $C_{19}H_{18}O_2PS$ $[M+H]^+$ m/z 341.0760, found 341.0755.

***S*-(4-hydroxyphenyl) diphenylphosphinothioate (3ga)**[3]



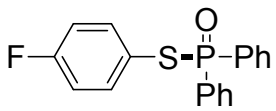
According to **GP1** with 4-mercaptophenol **1g** (75.7 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.4 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ga** as white solid (94.1 mg, 96%). According to **GP2** with 4,4'-disulfanediyldiphenol **4g** (45.9 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (61.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ga** as white solid (93.0 mg, 95%). Mp: 137-139 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 9.61 (br s, 1H), 7.89-7.83 (m, 4H), 7.57-7.44 (m, 6H), 7.09-7.06 (m, 2H), 6.53-6.50 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 159.1 (d, *J* = 2.2 Hz), 137.3 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 106.1 Hz), 132.5 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 10.4 Hz), 128.7 (d, *J* = 13.2 Hz), 117.4 (d, *J* = 2.2 Hz), 112.1 (d, *J* = 5.6 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 43.12; **HRMS** (ESI) calculated for $C_{18}H_{16}O_2PS$ $[M+H]^+$ m/z 327.0603, found 327.0604.

***S*-(4-aminophenyl) diphenylphosphinothioate (3ha)**[3]



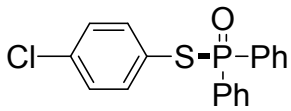
According to **GP1** with 4-aminobenzenethiol **1h** (75.1 mg, 0.6 mmol, 2.0 equiv), and diphenylphosphine oxide **2a** (61.3 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ha** as white solid (70.2 mg, 72%). Mp: 167-169 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.86-7.80 (m, 4H), 7.51-7.39 (m, 6H), 7.19-7.10 (m, 2H), 6.48-6.40 (m, 2H), 3.62 (br s, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.8, 136.9 (d, *J* = 2.9 Hz), 132.9 (d, *J* = 105.0 Hz), 132.0 (d, *J* = 3.0 Hz), 131.6 (d, *J* = 10.8 Hz), 128.4 (d, *J* = 12.6 Hz), 115.5, 111.9 (d, *J* = 4.9 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.36; **HRMS** (ESI) calculated for $C_{18}H_{17}NOPS$ $[M+H]^+$ m/z 326.0763, found 326.0762.

***S*-(4-fluorophenyl) diphenylphosphinothioate (3ia)**[1]



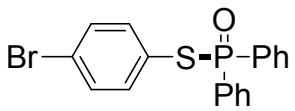
According to **GP1** with 4-fluorobenzenethiol **1i** (76.9 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.4 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ia** as white solid (80.3 mg, 82%). According to **GP2** with 1,2-bis(4-fluorophenyl)disulfane **4i** (45.8 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.9 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ia** as white solid (71.5 mg, 73%). Mp: 93-95 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.87-7.80 (m, 4H), 7.53-7.39 (m, 8H), 6.91-6.86 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 163.3 (dd, J = 248.4, 2.2 Hz), 137.3 (dd, J = 8.5, 3.6 Hz), 132.3 (d, J = 3.3 Hz), 132.1 (d, J = 106.6 Hz), 131.4 (d, J = 10.4 Hz), 128.5 (d, J = 13.2 Hz), 121.2 (dd, J = 5.0, 3.3 Hz), 116.2 (dd, J = 22.0, 1.7 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.66; **HRMS** (ESI) calculated for $C_{18}H_{15}FOPS$ $[M+H]^+$ m/z 329.0560, found 329.0551.

***S*-(4-chlorophenyl) diphenylphosphinothioate (3ja)**[2]



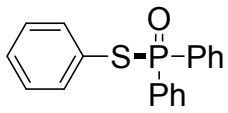
According to **GP1** with 4-chlorobenzenethiol **1j** (87.4 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ja** as white solid (76.0 mg, 74%). According to **GP2** with 1,2-bis(4-chlorophenyl)disulfane **4j** (51.7 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.9 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ja** as white solid (60.0 mg, 58%). Mp: 95-97 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.87-7.80 (m, 4H), 7.54-7.36 (m, 8H), 7.18-7.15 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.5 (d, J = 3.9 Hz), 135.5 (d, J = 2.0 Hz), 132.4 (d, J = 2.9 Hz), 132.3 (d, J = 107.0 Hz), 131.6 (d, J = 10.8 Hz), 129.3, 128.6 (d, J = 13.6 Hz), 124.7 (d, J = 4.9 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.67; **HRMS** (ESI) calculated for $C_{18}H_{15}ClOPS$ $[M+H]^+$ m/z 345.0264, found 345.0256.

***S*-(4-bromophenyl) diphenylphosphinothioate (3ka)**[3]



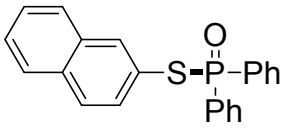
According to **GP1** with 4-bromobenzenethiol **1k** (114.2mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ka** as white solid (69.8 mg, 60%). Mp: 93-95 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86-7.82 (m, 4H), 7.53-7.50 (m, 2H), 7.46-7.42 (m, 4H), 7.31 (s, 4H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.7 (d, *J* = 2.9 Hz), 132.2 (d, *J* = 107.0 Hz), 132.4, 132.2, 131.5 (d, *J* = 10.8 Hz), 128.6 (d, *J* = 13.6 Hz), 125.4 (d, *J* = 4.9 Hz), 123.7 (d, *J* = 2.9 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.45; **HRMS** (ESI) calculated for $C_{18}H_{15}BrOPS$ $[M+H]^+$ m/z 388.9759, found 388.9747.

***S*-phenyl diphenylphosphinothioate (3la)**[2]



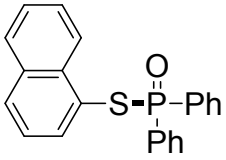
According to **GP1** with benzenethiol **1l** (66.1 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.6 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3la** as white solid (73.2 mg, 79%). According to **GP2** with 1,2-diphenyldisulfane **4l** (40.0 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3la** as white solid (75.4 mg, 81%). Mp: 86-88 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.88-7.81 (m, 4H), 7.50-7.38 (m, 8H), 7.24-7.14 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 135.2 (d, *J* = 3.8 Hz), 132.4 (d, *J* = 106.7 Hz), 132.2 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 10.4 Hz), 129.0 (d, *J* = 1.7 Hz), 128.8 (d, *J* = 2.3 Hz), 128.4 (d, *J* = 13.2 Hz), 126.0 (d, *J* = 5.0 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.79; **HRMS** (ESI) calculated for $C_{18}H_{16}OPS$ $[M+H]^+$ m/z 311.0654, found 311.0653.

***S*-naphthalen-2-yl diphenylphosphinothioate (3ma)**[1]



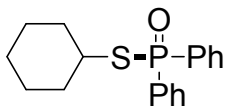
According to **GP1** with naphthalene-2-thiol **1m** (96.6 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ma** as white solid (86.6 mg, 80%). According to **GP2** with 1,2-di(naphthalen-2-yl)disulfane **4m** (58.7 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.4 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ma** as white solid (80.5 mg, 74%). Mp: 108-110 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.90-7.83 (m, 4H), 7.71-7.61 (m, 3H), 7.49-7.36 (m, 9H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 135.2 (d, *J* = 4.9 Hz), 133.4 (d, *J* = 1.7 Hz), 132.8 (d, *J* = 1.7 Hz), 132.4 (d, *J* = 106.7 Hz), 132.1 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 9.9 Hz), 131.4, 128.5, 128.4 (d, *J* = 13.2 Hz), 127.6, 127.4, 126.7, 126.3, 123.4 (d, *J* = 5.0 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.63; **HRMS** (ESI) calculated for $C_{22}H_{18}OPS$ $[M+H]^+$ m/z 361.0810, found 361.0806.

***S*-naphthalen-1-yl diphenylphosphinothioate (3na)**[3]



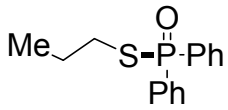
According to **GP1** with naphthalene-1-thiol **1n** (96.1 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.4 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3na** as white solid (64.6 mg, 60%). Mp: 100-102 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.1 Hz, 1H), 7.85-7.78 (m, 5H), 7.74-7.70 (m, 2H), 7.48-7.38 (m, 4H), 7.35-7.24 (m, 5H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 135.4 (d, *J* = 4.9 Hz), 134.9 (d, *J* = 3.0 Hz), 134.0, 132.5 (d, *J* = 106.0 Hz), 132.1 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 10.8 Hz), 129.9 (d, *J* = 2.9 Hz), 128.3 (d, *J* = 13.6 Hz), 128.2, 126.6, 126.1, 125.9, 125.4, 123.6 (d, *J* = 5.9 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.63; **HRMS** (ESI) calculated for $C_{22}H_{18}OPS$ $[M+H]^+$ m/z 361.0810, found 361.0813.

***S*-cyclohexyl diphenylphosphinothioate (3oa)**[3]



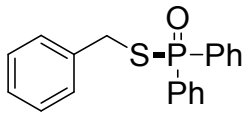
According to **GP1** with cyclohexanethiol **1o** (69.7 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3oa** as white solid (52.0 mg, 55%). According to **GP2** with 1,2-dicyclohexyldisulfane **4o** (41.5 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.8 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3oa** as white solid (39.4 mg, 42%). Mp: 75-77 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.91-7.84 (m, 4H), 7.51-7.43 (m, 6H), 3.36-3.25 (m, 1H), 1.96-1.93 (m, 2H), 1.69-1.64 (m, 2H), 1.58-1.45 (m, 3H), 1.34-1.17 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 134.1 (d, J = 106.1 Hz), 132.0 (d, J = 3.3 Hz), 131.3 (d, J = 9.9 Hz), 128.4 (d, J = 13.1 Hz), 44.4 (d, J = 1.7 Hz), 35.5 (d, J = 3.9 Hz), 25.6, 25.2; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.97; **HRMS** (ESI) calculated for $C_{18}H_{22}OPS$ $[M+H]^+$ m/z 317.1123, found 317.1126.

***S*-propyl diphenylphosphinothioate (3pa)**[2]



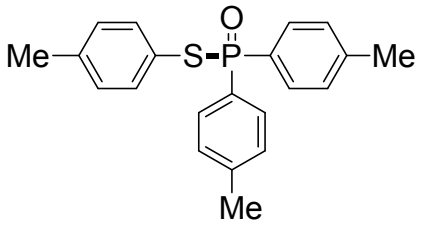
According to **GP1** with propane-1-thiol **1p** (46.0 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (60.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3pa** as white solid (36.9 mg, 45%). According to **GP2** with 1,2-dipropyldisulfane **4p** (27.1 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.9 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3pa** as colorless oil (64.8 mg, 78%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.92-7.85 (m, 4H), 7.54-7.43 (m, 6H), 2.82-2.74 (m, 2H), 1.71-1.59 (m, 2H), 0.93 (t, J = 7.4 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 133.4 (d, J = 106.7 Hz), 132.1 (d, J = 3.3 Hz), 131.3 (d, J = 10.4 Hz), 128.5 (d, J = 12.6 Hz), 31.1 (d, J = 2.2 Hz), 23.9 (d, J = 5.0 Hz), 13.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 43.30; **HRMS** (ESI) calculated for $C_{15}H_{18}OPS$ $[M+H]^+$ m/z 277.0810, found 277.0815.

***S*-benzyl diphenylphosphinothioate (3qa)**[3]



According to **GP1** with phenylmethanethiol **1q** (74.5 mg, 0.6 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (61.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3qa** as white solid (63.3 mg, 65%). According to **GP2** with 1,2-dibenzyldisulfane **4q** (45.0 mg, 0.18 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (60.9 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3qa** as colorless oil (51.7 mg, 53%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.90-7.83 (m, 4H), 7.53-7.41 (m, 6H), 7.23-7.14 (m, 5H), 4.02 (d, *J* = 9.3 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.8 (d, *J* = 4.9 Hz), 133.1 (d, *J* = 105.9 Hz), 132.2 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 9.8 Hz), 128.9, 128.6, 128.5 (d, *J* = 4.9 Hz), 127.3, 33.1 (d, *J* = 2.0 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 42.83; **HRMS** (ESI) calculated for $C_{19}H_{18}OPS$ $[M+H]^+$ m/z 325.0815, found 325.0810.

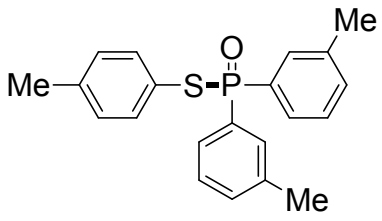
***S*-*p*-tolyl di-*p*-tolylphosphinothioate (3ab)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.8 mg, 0.6 mmol, 2.0 equiv) and di-*p*-tolylphosphine oxide **2b** (70.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ab** as colorless oil (80.9 mg, 77%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.0 mg, 0.18 mmol, 0.6 equiv) and di-*p*-tolylphosphine oxide **2b** (71.1 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ab** as colorless oil (71.5 mg, 68%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.75-7.68 (m, 4H), 7.34-7.31 (m, 2H), 7.23-7.20 (m, 4H), 6.99 (d, *J* = 8.1 Hz, 2H), 2.35 (s, 6H), 2.23 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$)

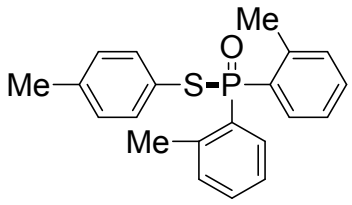
δ 142.5 (d, *J* = 3.3 Hz), 138.7 (d, *J* = 2.3 Hz), 135.0 (d, *J* = 3.3 Hz), 131.4 (d, *J* = 10.4 Hz), 129.7 (d, *J* = 1.7 Hz), 129.5 (d, *J* = 108.9 Hz), 129.0 (d, *J* = 13.7 Hz), 122.6 (d, *J* = 5.0 Hz), 21.4, 20.9; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 42.01; **HRMS** (ESI) calculated for $C_{21}H_{22}OPS$ $[M+H]^+$ m/z 353.1123, found 353.1123.

***S*-*p*-tolyl di-*m*-tolylphosphinothioate (3ac)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.6 mg, 0.6 mmol, 2.0 equiv) and di-*m*-tolylphosphine oxide **2c** (70.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ac** as white solid (83.6 mg, 79%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.6 mg, 0.18 mmol, 0.6 equiv) and di-*m*-tolylphosphine oxide **2c** (69.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ac** as white solid (80.3 mg, 76%). Mp: 59-61 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.69-7.59 (m, 4H), 7.39-7.28 (m, 6H), 7.00 (d, *J* = 7.8 Hz, 2H), 2.33 (s, 6H), 2.24 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 138.9 (d, *J* = 2.2 Hz), 138.2 (d, *J* = 13.2 Hz), 135.2 (d, *J* = 3.9 Hz), 132.8 (d, *J* = 2.8 Hz), 132.4 (d, *J* = 104.9 Hz), 132.0 (d, *J* = 9.9 Hz), 129.7 (d, *J* = 1.7 Hz), 128.4 (d, *J* = 10.4 Hz), 128.2 (d, *J* = 13.7 Hz), 122.4 (d, *J* = 5.0 Hz), 21.2, 20.9; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 42.00; **HRMS** (ESI) calculated for $C_{21}H_{22}OPS$ $[M+H]^+$ m/z 353.1123, found 353.1119.

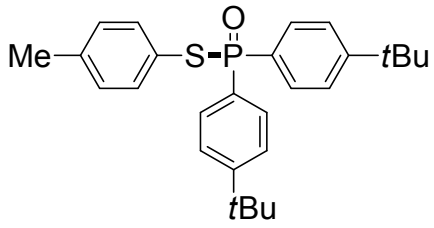
***S*-*p*-tolyl di-*o*-tolylphosphinothioate (3ad)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.5 mg, 0.6 mmol, 2.0 equiv), and di-*o*-tolylphosphine oxide **2d** (70.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum

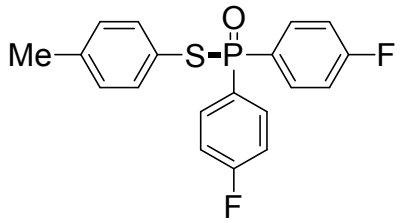
ether/EtOAc = 3/1) to afford the desired product **3ad** as white solid (56.2 mg, 53%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.0 mg, 0.18 mmol, 0.6 equiv) and di-*o*-tolylphosphine oxide **2d** (71.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ad** as white solid (48.8 mg, 46%). Mp: 99-101 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.40-7.33 (m, 4H), 7.23-7.16 (m, 4H), 7.02 (d, *J* = 7.5 Hz, 2H), 2.40 (s, 6H), 2.27 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 141.9 (d, *J* = 9.9 Hz), 139.0 (d, *J* = 2.2 Hz), 135.6 (d, *J* = 3.8 Hz), 132.7 (d, *J* = 11.5 Hz), 132.0 (d, *J* = 3.3 Hz), 131.8 (d, *J* = 12.1 Hz), 131.6 (d, *J* = 101.7 Hz), 129.8 (d, *J* = 1.7 Hz), 125.4 (d, *J* = 13.2 Hz), 122.4 (d, *J* = 5.0 Hz), 21.3 (d, *J* = 3.8 Hz), 21.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 43.77; **HRMS** (ESI) calculated for $C_{21}H_{22}OPS$ $[M+H]^+$ m/z 353.1123, found 353.1117.

***S*-*p*-tolyl bis(4-(*tert*-butyl)phenyl)phosphinothioate (3ae)**[3]



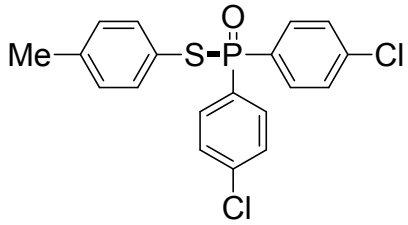
According to **GP1** with 4-methylbenzenethiol **1a** (74.9 mg, 0.6 mmol, 2.0 equiv) and bis(4-(*tert*-butyl)phenyl)phosphine oxide **2e** (95.6 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ae** as white solid (95.4 mg, 73%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.6 mg, 0.18 mmol, 0.6 equiv) and bis(4-(*tert*-butyl)phenyl)phosphine oxide **2e** (94.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ae** as white solid (106.3 mg, 81%). Mp: 120-122 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.81-7.74 (m, 4H), 7.45-7.41 (m, 4H), 7.35-7.29 (m, 2H), 6.97 (d, *J* = 7.8 Hz, 2H), 2.22 (s, 3H), 1.29 (s, 18H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 155.6 (d, *J* = 2.8 Hz), 138.8 (d, *J* = 2.2 Hz), 135.2 (d, *J* = 3.3 Hz), 131.4 (d, *J* = 10.4 Hz), 129.7 (d, *J* = 1.7 Hz), 129.6 (d, *J* = 108.8 Hz), 125.4 (d, *J* = 13.2 Hz), 122.9 (d, *J* = 5.6 Hz), 34.9, 31.0, 21.0; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.53; **HRMS** (ESI) calculated for $C_{27}H_{34}OPS$ $[M+H]^+$ m/z 437.2062, found 437.2059.

***S*-*p*-tolyl bis(4-fluorophenyl)phosphinothioate (3af)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.3 mg, 0.6 mmol, 2.0 equiv) and bis(4-fluorophenyl)phosphine oxide **2f** (72.1 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3af** as colorless oil (75.1 mg, 69%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.8 mg, 0.18 mmol, 0.6 equiv) and bis(4-fluorophenyl)phosphine oxide **2f** (72.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3af** as colorless oil (64.8 mg, 60%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.88-7.79 (m, 4H), 7.35-7.26 (m, 2H), 7.16-7.10 (m, 4H), 7.02 (d, *J* = 7.8 Hz, 2H), 2.26 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.2 (dd, *J* = 252.8, 3.3 Hz), 139.4 (d, *J* = 2.2 Hz), 135.2 (d, *J* = 3.9 Hz), 134.1 (dd, *J* = 11.9, 9.1 Hz), 130.0 (d, *J* = 1.1 Hz), 128.4 (dd, *J* = 110.4, 2.7 Hz), 121.8 (d, *J* = 5.5 Hz), 115.9 (dd, *J* = 21.4, 14.3 Hz), 21.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 39.28; **HRMS** (ESI) calculated for $C_{19}H_{16}F_2OPS$ $[M+H]^+$ m/z 361.0622, found 361.0617.

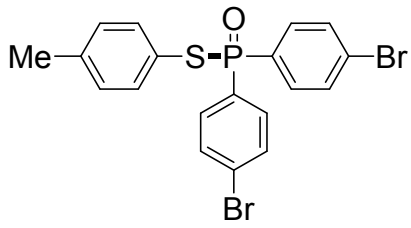
S-*p*-tolyl bis(4-chlorophenyl)phosphinothioate (3ag)[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.6 mg, 0.6 mmol, 2.0 equiv) and bis(4-chlorophenyl)phosphine oxide **2g** (82.3 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ag** as white solid (71.2 mg, 60%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.0 mg, 0.18 mmol, 0.6 equiv) and bis(4-chlorophenyl)phosphine oxide **2g** (82.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ag** as white solid (76.6

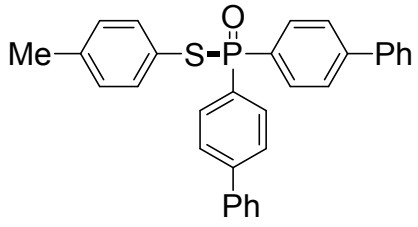
mg, 65%). Mp: 109-111 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.79-7.72 (m, 4H), 7.44-7.40 (m, 4H), 7.35-7.27 (m, 2H), 7.03 (d, *J* = 7.8 Hz, 2H), 2.27 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.5 (d, *J* = 2.7 Hz), 139.1 (d, *J* = 3.9 Hz), 135.2 (d, *J* = 3.9 Hz), 132.9 (d, *J* = 11.6 Hz), 130.9 (d, *J* = 108.2 Hz), 130.1 (d, *J* = 1.7 Hz), 128.9 (d, *J* = 13.7 Hz), 121.5 (d, *J* = 5.0 Hz), 21.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 39.13; **HRMS** (ESI) calculated for $C_{19}H_{16}Cl_2OPS$ $[M+H]^+$ m/z 393.0031, found 393.0031.

***S*-*p*-tolyl bis(4-bromophenyl)phosphinothioate (3ah)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (75.3 mg, 0.6 mmol, 2.0 equiv) and bis(4-bromophenyl)phosphine oxide **2h** (109.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ah** as white solid (77.5 mg, 54%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.1 mg, 0.18 mmol, 0.6 equiv) and bis(4-bromophenyl)phosphine oxide **2h** (108.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ah** as white solid (65.2 mg, 45%). Mp: 132-134 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.71-7.64 (m, 4H), 7.59-7.56 (m, 4H), 7.35-7.27 (m, 2H), 7.03 (d, *J* = 7.8 Hz, 2H), 2.26 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.6 (d, *J* = 2.2 Hz), 135.3 (d, *J* = 3.8 Hz), 133.0 (d, *J* = 11.0 Hz), 131.9 (d, *J* = 13.1 Hz), 131.4 (d, *J* = 107.7 Hz), 130.2 (d, *J* = 1.7 Hz), 127.8 (d, *J* = 3.9 Hz), 121.4 (d, *J* = 5.5 Hz), 21.1; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 39.50; **HRMS** (ESI) calculated for $C_{19}H_{16}Br_2OPS$ $[M+H]^+$ m/z 480.9021, found 480.9023.

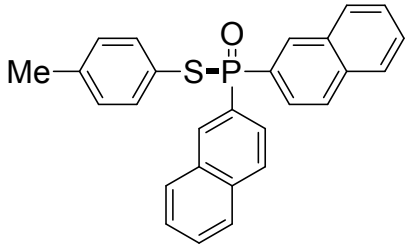
***S*-*p*-tolyl di([1,1'-biphenyl]-4-yl)phosphinothioate (3ai)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.5 mg, 0.6 mmol, 2.0 equiv) and

di([1,1'-biphenyl]-4-yl)phosphine oxide **2i** (107.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ai** as white solid (106.4 mg, 74%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.1 mg, 0.18 mmol, 0.6 equiv) and di([1,1'-biphenyl]-4-yl)phosphine oxide **2i** (106.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ai** as white solid (112.2 mg, 78%). Mp: 125-127 °C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.99-7.92 (m, 4H), 7.65-7.61 (m, 4H), 7.55-7.53 (m, 4H), 7.41-7.30 (m, 8H), 6.98 (d, *J* = 7.8 Hz, 2H), 2.19 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 144.8 (d, *J* = 2.8 Hz), 139.6, 139.0 (d, *J* = 2.3 Hz), 135.2 (d, *J* = 3.3 Hz), 132.0 (d, *J* = 10.4 Hz), 130.5, 129.9 (d, *J* = 1.7 Hz), 128.8, 128.1, 127.1, 127.0 (d, *J* = 10.4 Hz), 122.3 (d, *J* = 5.0 Hz), 21.0; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.09; **HRMS** (ESI) calculated for $C_{31}H_{26}OPS$ $[M+H]^+$ m/z 477.1436, found 477.1435.

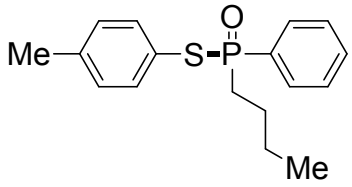
***S*-*p*-tolyl di(naphthalen-2-yl)phosphinothioate (3aj)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (75.0 mg, 0.6 mmol, 2.0 equiv) and di(naphthalen-2-yl)phosphine oxide **2j** (91.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3aj** as colorless oil (90.9 mg, 71%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.4 mg, 0.18 mmol, 0.6 equiv) and di(naphthalen-2-yl)phosphine oxide **2j** (91.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3aj** as colorless oil (95.7 mg, 75%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.43 (s, 1H), 7.90-7.82 (m, 8H), 7.59-7.49 (m, 4H), 7.41-7.35 (m, 2H), 6.95 (d, *J* = 8.1 Hz, 2H), 2.18 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.1 (d, *J* = 1.7 Hz), 135.3 (d, *J* = 3.8 Hz), 134.8 (d, *J* = 2.2 Hz), 133.9 (d, *J* = 9.3 Hz), 132.4 (d, *J* = 14.3 Hz), 129.9 (d, *J* = 1.1 Hz), 129.8 (d, *J* = 107.2 Hz), 129.0, 128.32, 128.30 (d, *J* = 13.2 Hz), 127.7, 126.9, 126.2 (d, *J* = 11.6 Hz), 122.2 (d, *J* = 4.9 Hz), 21.0; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.53; **HRMS** (ESI)

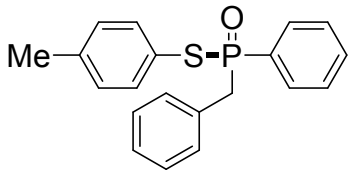
calculated for $C_{27}H_{22}OPS$ $[M+H]^+$ m/z 425.1123, found 425.1123.

***S*-*p*-tolyl butyl(phenyl)phosphinothioate (3ak)**[3]



According to **GP1** with 4-methylbenzenethiol **1a** (74.4 mg, 0.6 mmol, 2.0 equiv) and butyl(phenyl)phosphine oxide **2k** (55.5 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ak** as colorless oil (63.2 mg, 69%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (44.6 mg, 0.18 mmol, 0.6 equiv) and butyl(phenyl)phosphine oxide **2k** (54.8 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3ak** as colorless oil (56.6 mg, 62%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.78-7.71 (m, 2H), 7.52-7.39 (m, 3H), 7.35-7.28 (m, 2H), 7.04 (d, *J* = 8.1 Hz, 2H), 2.28 (s, 3H), 2.24-2.04 (m, 2H), 1.68-1.47 (m, 2H), 1.38-1.26 (m, 2H), 0.85 (d, *J* = 7.2 Hz, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.0 (d, *J* = 2.2 Hz), 135.3 (d, *J* = 3.3 Hz), 132.4 (d, *J* = 98.3 Hz), 131.9 (d, *J* = 2.8 Hz), 131.1 (d, *J* = 9.9 Hz), 129.9 (d, *J* = 1.7 Hz), 128.3 (d, *J* = 12.7 Hz), 122.2 (d, *J* = 5.5 Hz), 32.7 (d, *J* = 71.0 Hz), 24.2 (d, *J* = 4.4 Hz), 23.6 (d, *J* = 15.9 Hz), 21.0, 13.4; **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 45.29; **HRMS** (ESI) calculated for $C_{17}H_{22}OPS$ $[M+H]^+$ m/z 305.1123, found 305.1115.\

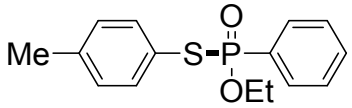
***S*-*p*-tolyl benzyl(phenyl)phosphinothioate (3al)**



According to **GP1** with 4-methylbenzenethiol **1a** (74.3 mg, 0.6 mmol, 2.0 equiv) and benzyl(phenyl)phosphine oxide **2l** (65.7 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3al** as white solid (81.8 mg, 81%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.1 mg, 0.18 mmol, 0.6 equiv) and benzyl(phenyl)phosphine oxide **2l** (66.0 mg, 0.3 mmol, 1.0 equiv). The crude

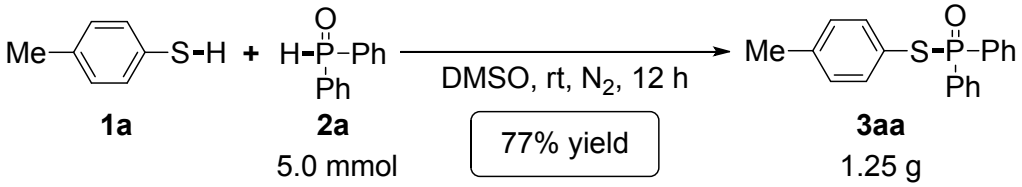
reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3al** as white solid (75.7 mg, 75%). Mp: 134-136 °C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.66-7.60 (m, 2H), 7.41-7.33 (m, 5H), 7.21-7.12 (m, 3H), 7.07-7.00 (m, 4H), 3.61-3.46 (m, 2H), 2.24 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 138.9 (d, *J* = 1.9 Hz), 135.1 (d, *J* = 2.9 Hz), 131.9 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 8.8 Hz), 131.3 (d, *J* = 99.3 Hz), 130.6 (d, *J* = 8.8 Hz), 129.9 (d, *J* = 5.9 Hz), 129.8, 128.2 (d, *J* = 2.9 Hz), 128.0 (d, *J* = 12.8 Hz), 126.8 (d, *J* = 2.9 Hz), 121.9 (d, *J* = 4.9 Hz), 41.1 (d, *J* = 65.1 Hz), 20.9; **^{31}P NMR** (202.5 MHz, $CDCl_3$) δ 47.40; **HRMS** (ESI) calculated for $C_{20}H_{20}OPS$ $[M+H]^+$ m/z 339.0967, found 339.0971.

***O*-ethyl *S*-*p*-tolyl phenylphosphonothioate (3am)**[3]



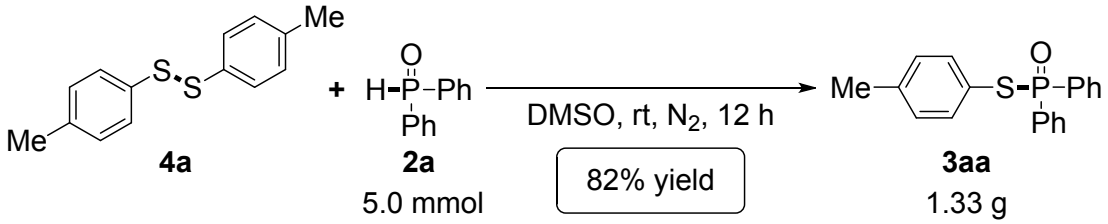
According to **GP1** with 4-methylbenzenethiol **1a** (74.6 mg, 0.6 mmol, 2.0 equiv) and ethyl phenylphosphinate **2m** (51.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3am** as colorless oil (43.5 mg, 50%). According to **GP2** with 1,2-di-*p*-tolyldisulfane **4a** (45.1 mg, 0.18 mmol, 0.6 equiv) and ethyl phenylphosphinate **2m** (51.0 mg, 0.3 mmol, 1.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3am** as colorless oil (43.9 mg, 50%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.70-7.63 (m, 2H), 7.51-7.47 (m, 1H), 7.40-7.33 (m, 2H), 7.18-7.16 (m, 2H), 7.02-6.99 (m, 2H), 4.39-4.26 (m, 2H), 2.29 (s, 3H), 1.39 (d, *J* = 7.1 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.1 (d, *J* = 3.3 Hz), 135.3 (d, *J* = 4.4 Hz), 132.3 (d, *J* = 3.3 Hz), 131.7 (d, *J* = 149.5 Hz), 131.4 (d, *J* = 10.5 Hz), 129.8 (d, *J* = 2.2 Hz), 128.1 (d, *J* = 14.8 Hz), 122.8 (d, *J* = 5.5 Hz), 62.3 (d, *J* = 7.1 Hz), 21.0, 16.2 (d, *J* = 6.5 Hz); **^{31}P NMR** (121.5 MHz, $CDCl_3$) δ 41.91; **HRMS** (ESI) calculated for $C_{15}H_{18}O_2PS$ $[M+H]^+$ m/z 293.0760, found 293.0758.

Lager scale experiments



1a **2a** 5.0 mmol — 77% yield — **3aa** 1.25 g

4-Methylbenzenethiol **1a** (1.24 g, 10.0 mmol, 2.0 equiv) and diphenylphosphine oxide **2a** (1.01g, 5.0 mmol, 1.0 equiv) were placed in a 100 mL round bottom flask. Then DMSO (25.0 mL) was added. The reaction mixture was stirred at room temperature under N_2 for 12 h, and the reaction was monitored with TLC. After the reaction was completed, H_2O (50.0 mL) was added, and the mixture was extracted by EtOAc (3x50.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3aa** (1.25 g, 77%).



4a **2a** 5.0 mmol — 82% yield — **3aa** 1.33 g

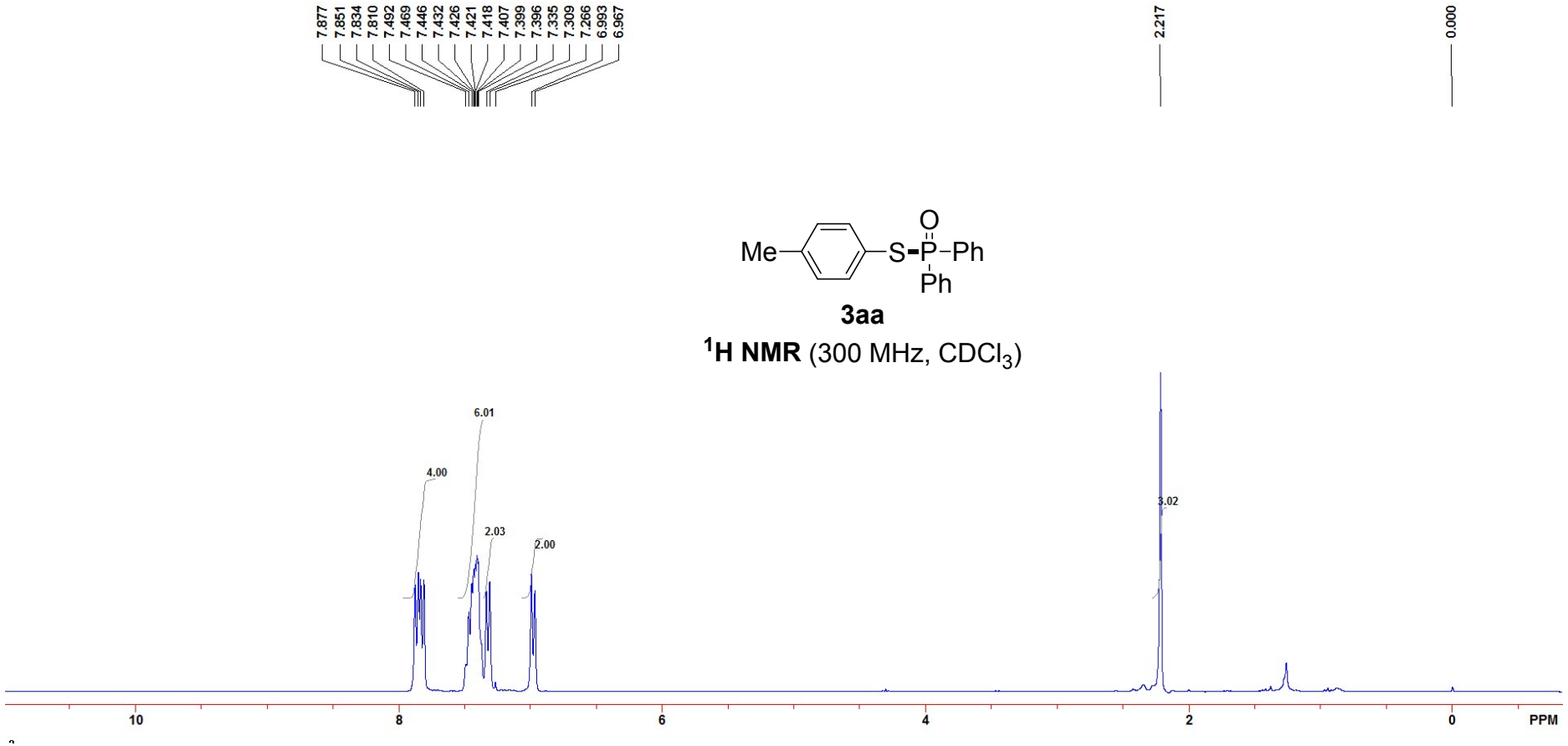
1,2-Di-*p*-tolyldisulfane **4a** (0.74 g, 3.0 mmol, 0.6 equiv) and diphenylphosphine oxide **2a** (1.01g, 5.0 mmol, 1.0 equiv) were placed in a 100 mL round bottom flask. Then DMSO (25.0 mL) was added. The reaction mixture was stirred at room temperature under N_2 for 12 h, and the reaction was monitored with TLC. After the reaction was completed, H_2O (50.0 mL) was added, and the mixture was extracted by EtOAc (3x50.0 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 3/1) to afford the desired product **3aa** (1.33 g, 82%).

References:

[1] Li, S.; Chen, T.; Saga, Y.; Han, L.-B. *RSC Adv.* **2015**, *5*, 71544.

[2] Wang, J.; Huang, X.; Ni, Z.; Wang, S.; Wu, J.; Pan, Y. *Green Chem.* **2015**, *17*, 314.

[3] Sun, J.-G.; Yang, H.; Li, P.; Zhang, B. *Org. Lett.* **2016**, *18*, 5114.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7.432
7.426
7.421
7.418
7.407
7.399
7.396
7.335
7.309
7.266
6.993
6.967
2.217
0.000
Me
S
P
O
Ph
Ph
3aa
1H NMR (300 MHz, CDCl3)
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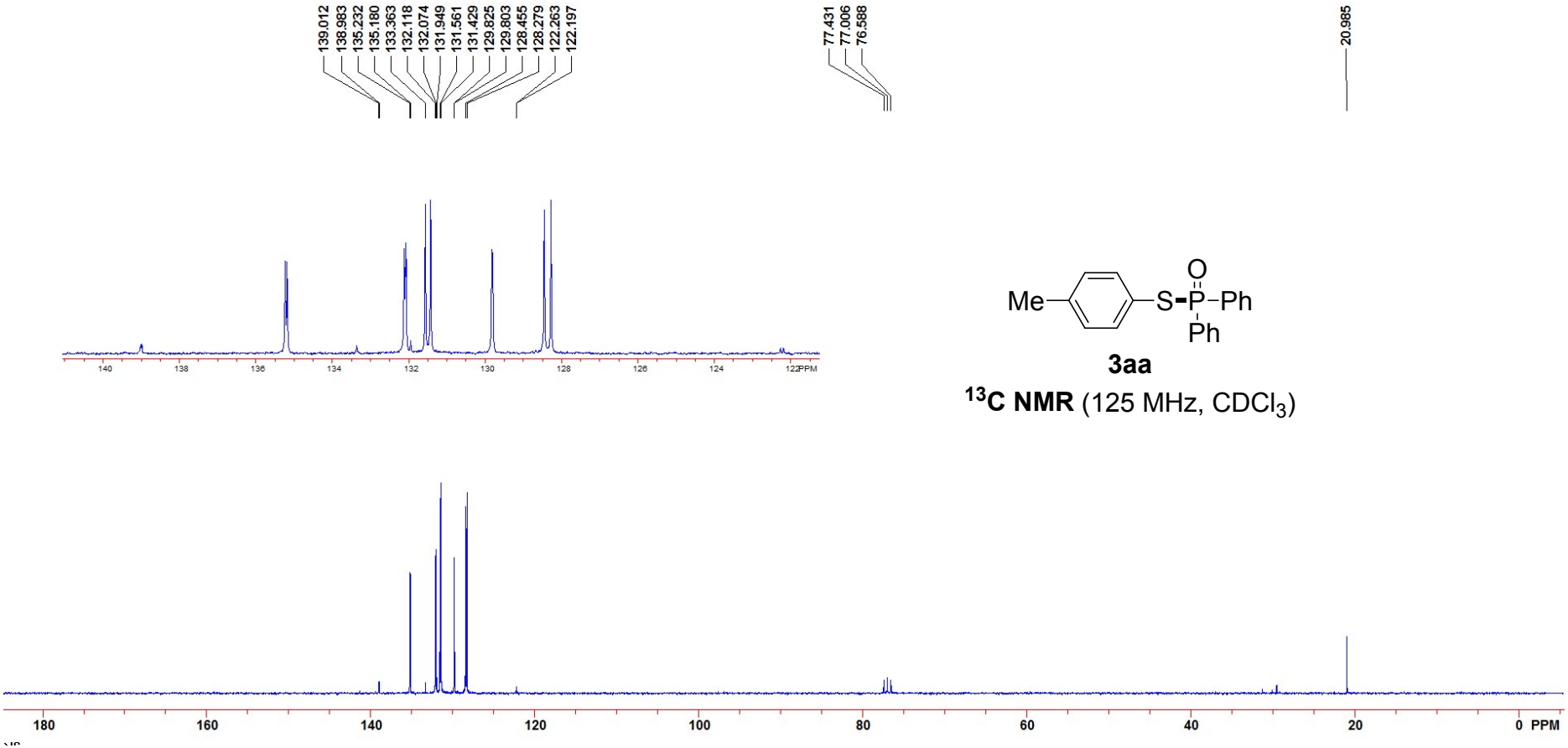
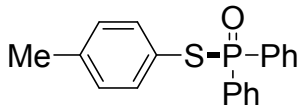
4.00
2.03
2.00
3.02
10
8
6
4
2
0
PPM



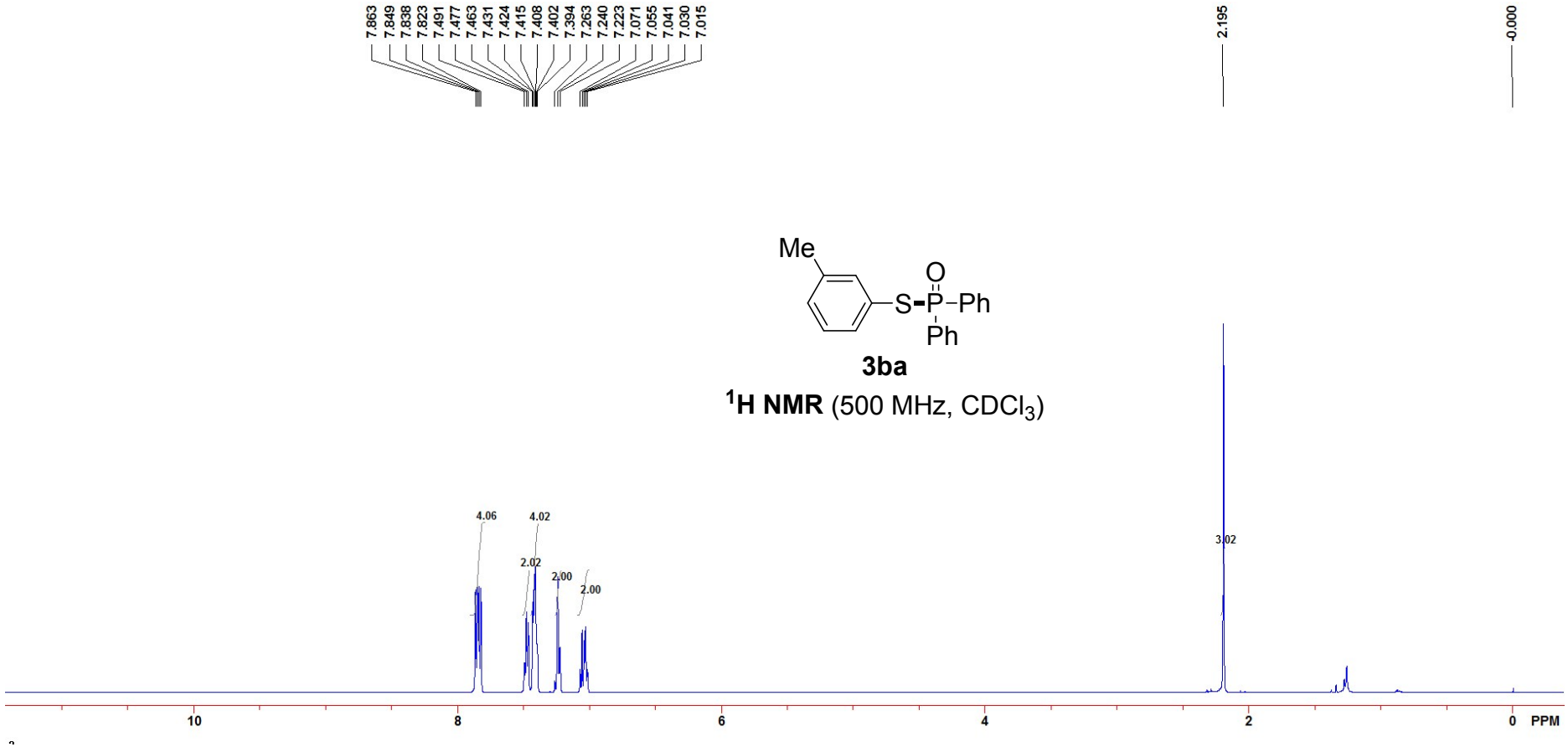
3aa

^{13}C NMR (125 MHz, $CDCl_3$)



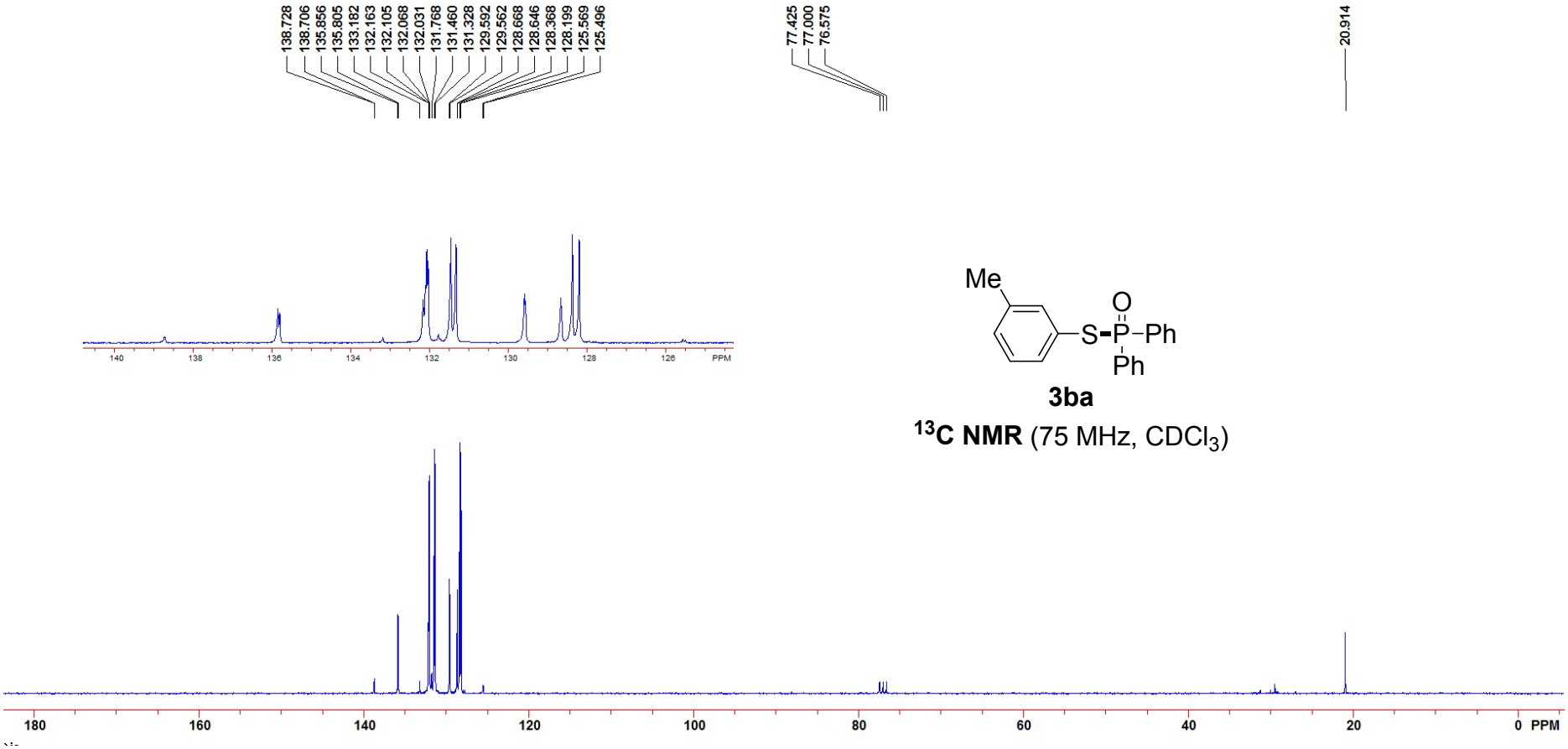
3aa

^{31}P NMR (121.5 MHz, $CDCl_3$)



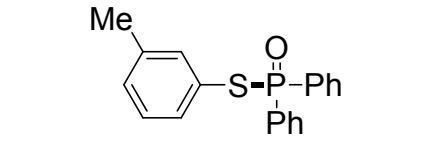
3ba

^{1}H NMR (500 MHz, $CDCl_3$)



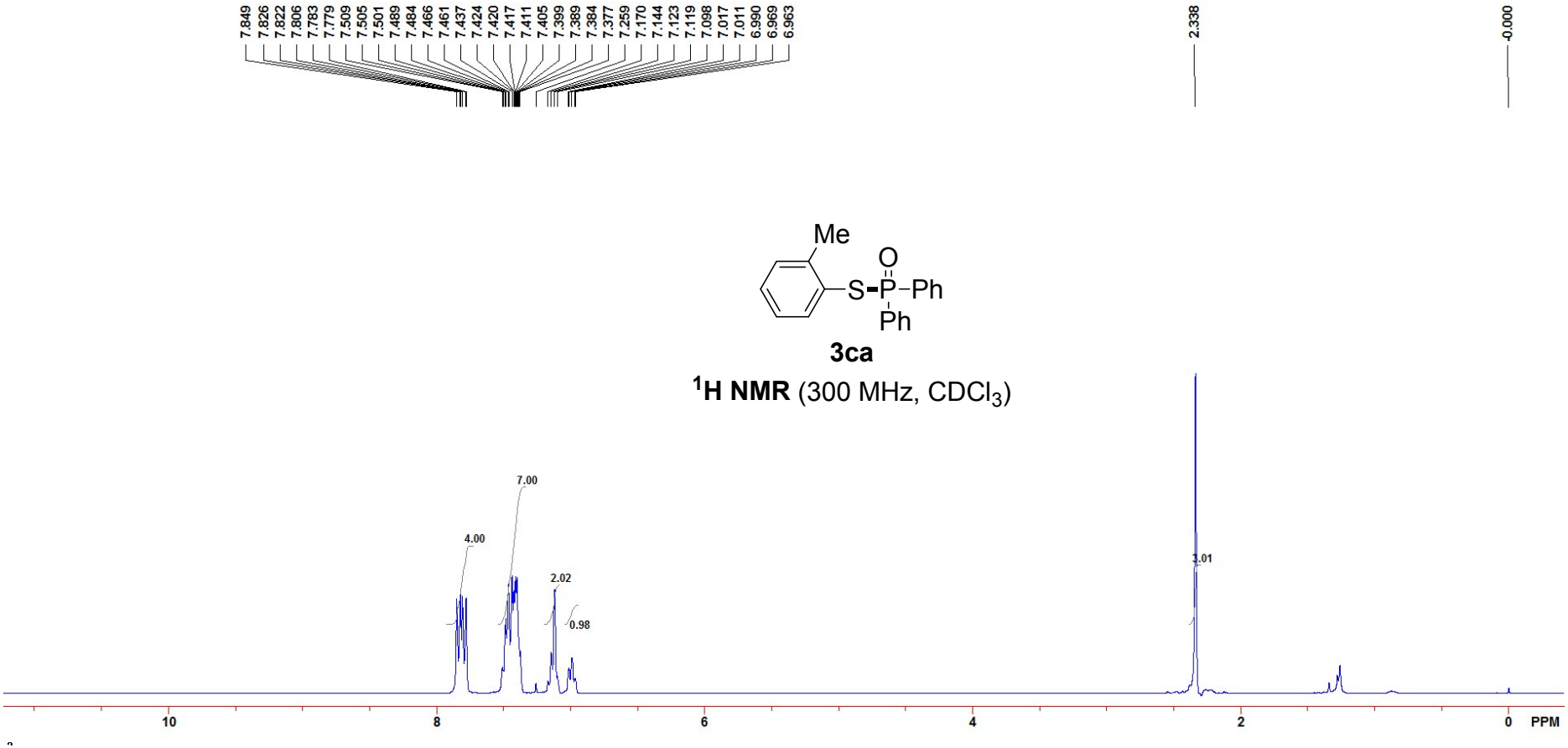
3ba

^{13}C NMR (75 MHz, $CDCl_3$)



3ba

^{31}P NMR (121.5 MHz, $CDCl_3$)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7.822
7.806
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7.779
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7.501
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7.484
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7.437
7.424
7.420
7.417
7.411
7.405
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7.384
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7.144
7.123
7.119
7.098
7.017
7.011
6.990
6.969
6.963
2.338
0.000
Me
O
S
P
Ph
Ph
3ca
1H NMR (300 MHz, CDCl3)
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2.02
0.98
3.01
10
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4
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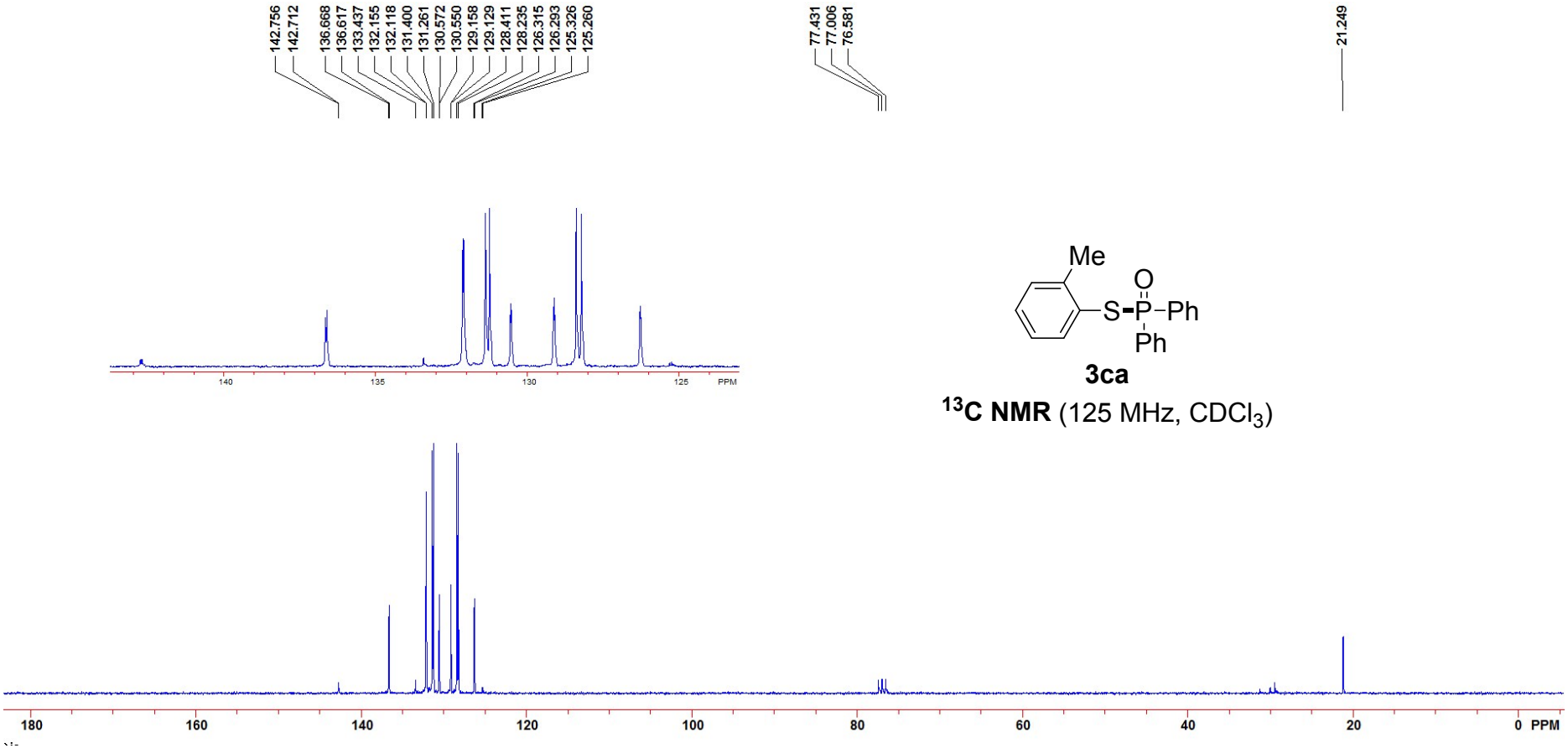
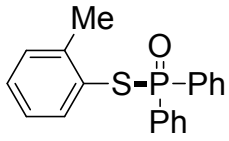
PPM



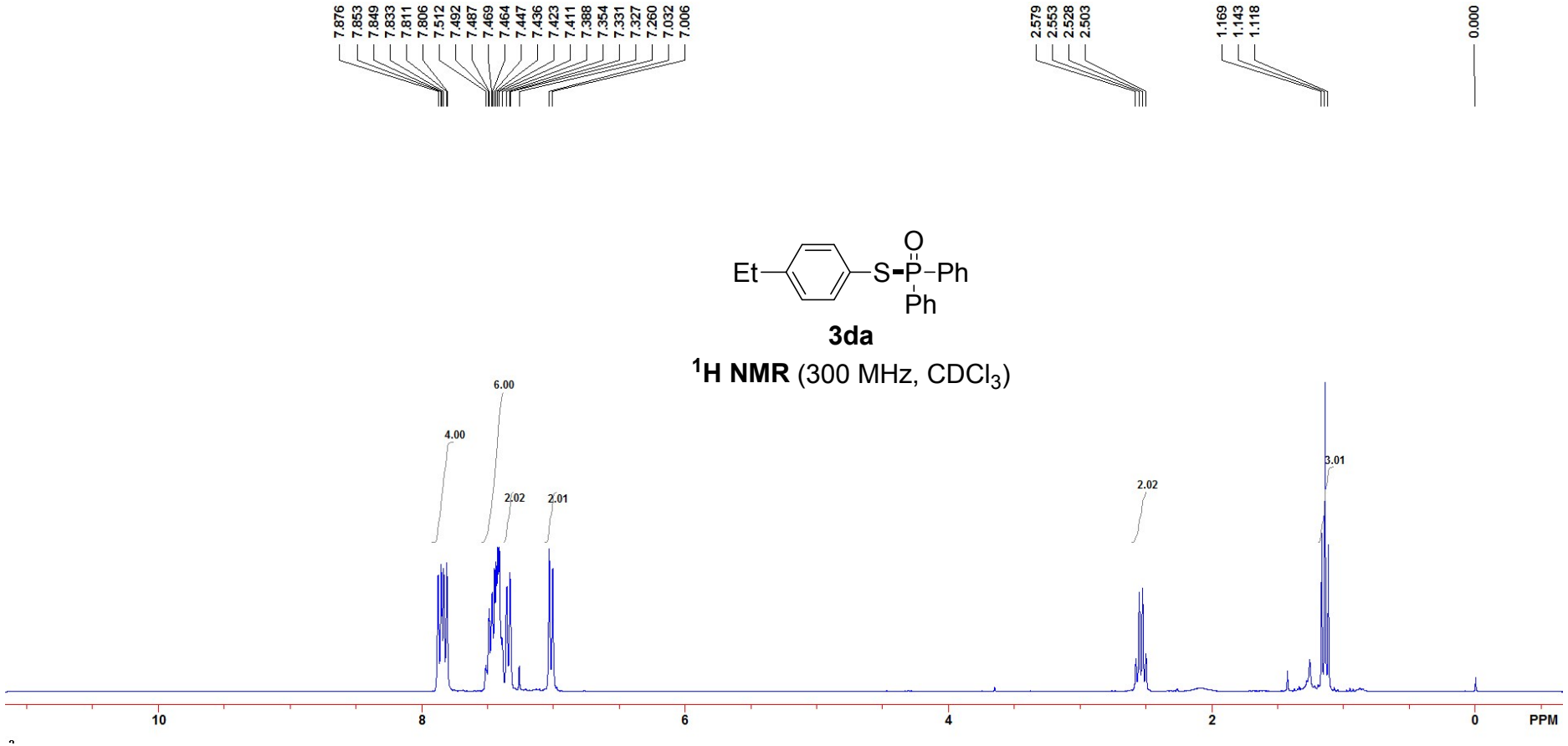
3ca

^{13}C NMR (125 MHz, $CDCl_3$)



3ca

^{31}P NMR (121.5 MHz, $CDCl_3$)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7.833
7.811
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7.492
7.487
7.469
7.464
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7.423
7.411
7.388
7.354
7.331
7.327
7.260
7.032
7.006
2.579
2.553
2.528
2.503
1.169
1.143
1.118
0.000
Et
S
P
O
Ph
Ph
3da
1H NMR (300 MHz, CDCl3)
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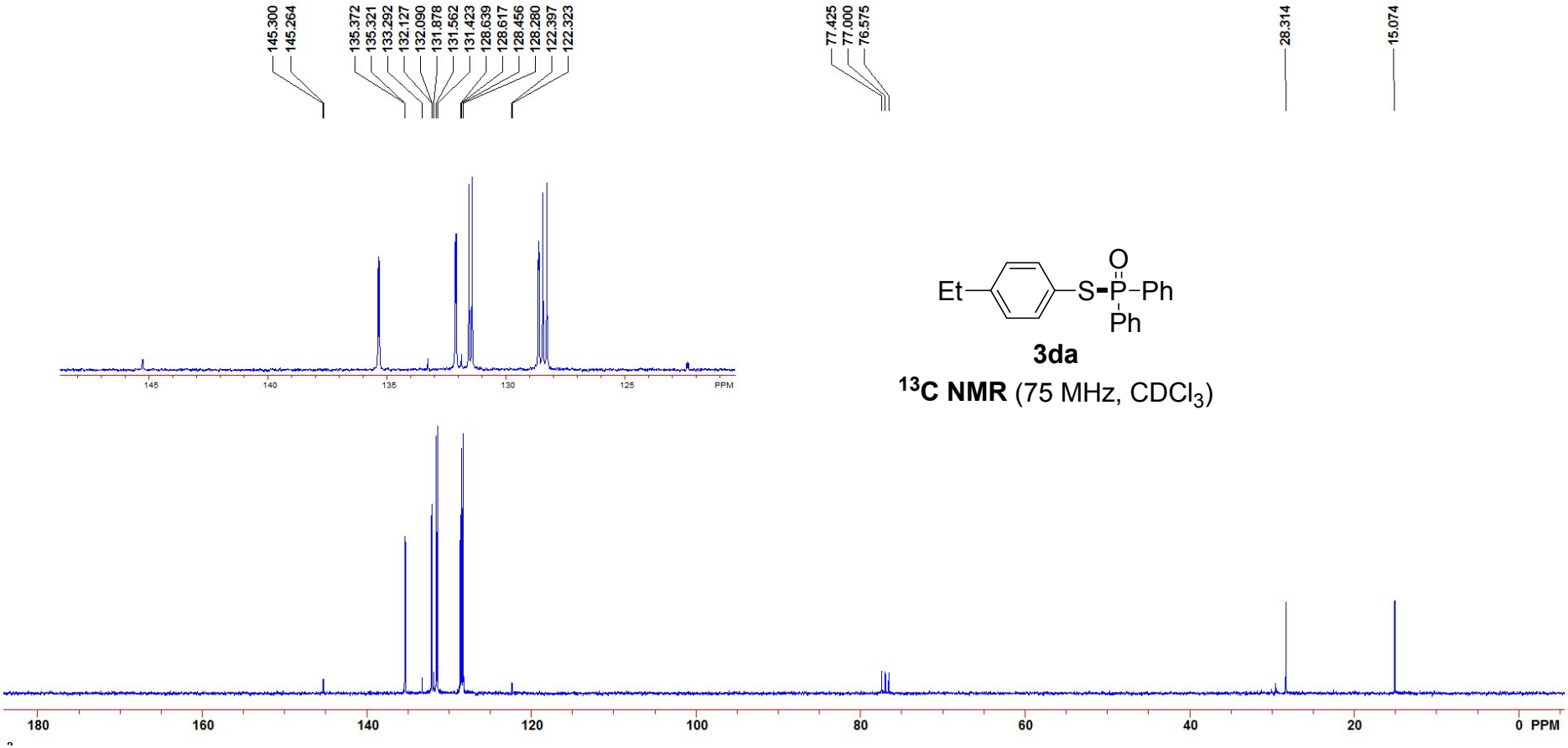
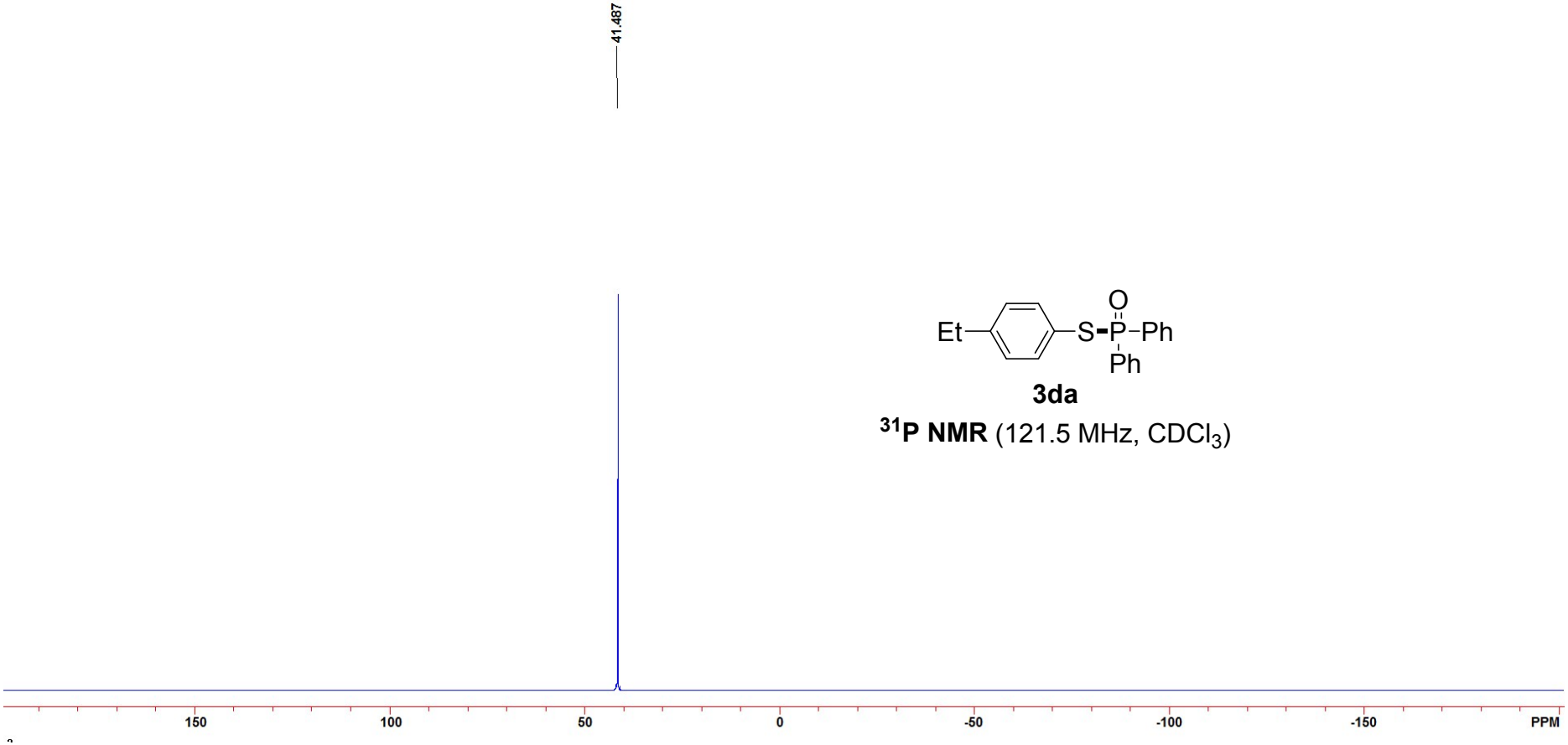
4.00
2.02
2.01
2.02
3.01
10
8
6
4
2
0
PPM



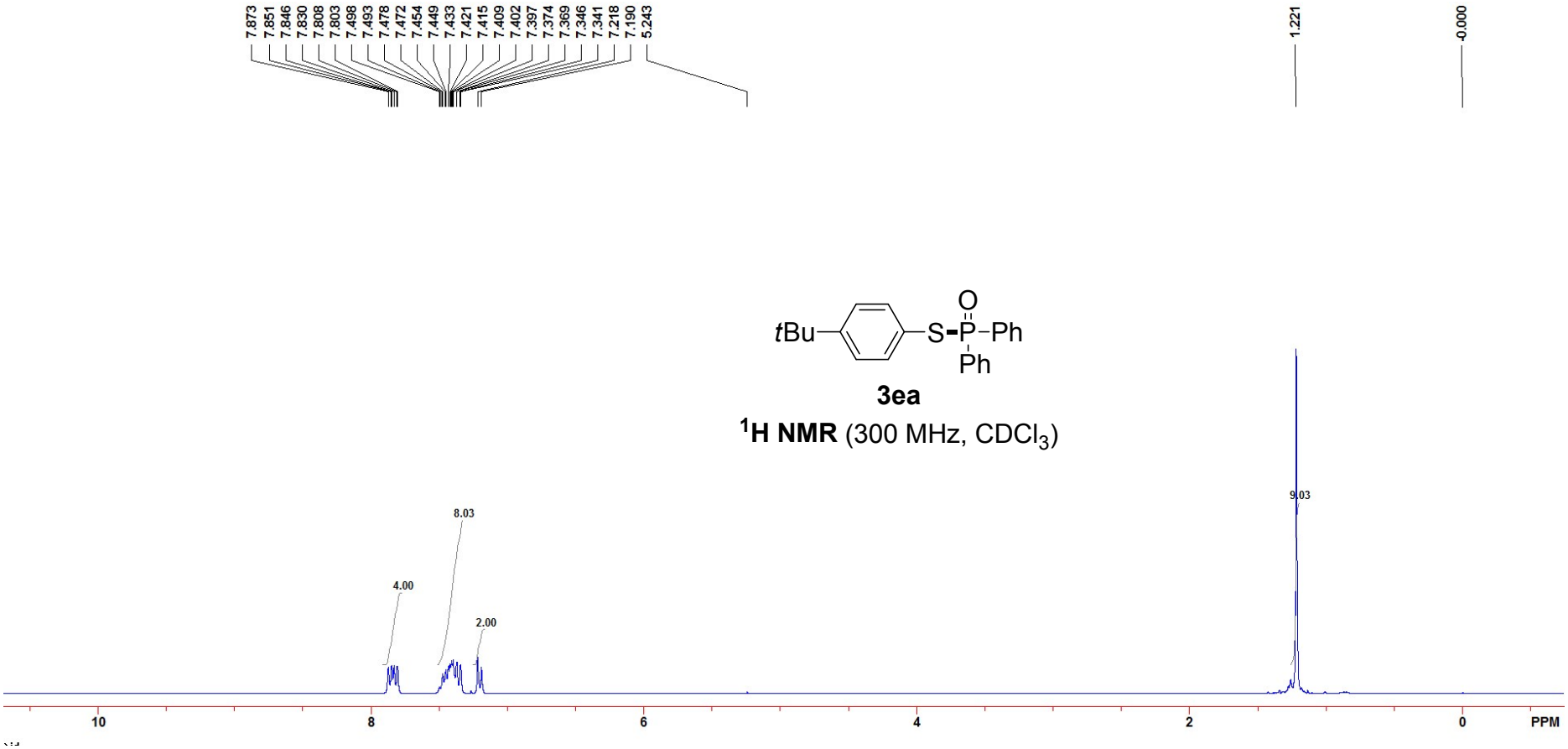
3da

^{13}C NMR (75 MHz, $CDCl_3$)



3da

^{31}P NMR (121.5 MHz, $CDCl_3$)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7.493
7.478
7.472
7.454
7.449
7.433
7.421
7.415
7.409
7.402
7.397
7.374
7.369
7.346
7.341
7.218
7.190
5.243
1.221
0.000
tBu
S
P
O
Ph
Ph
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9.03
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4.00
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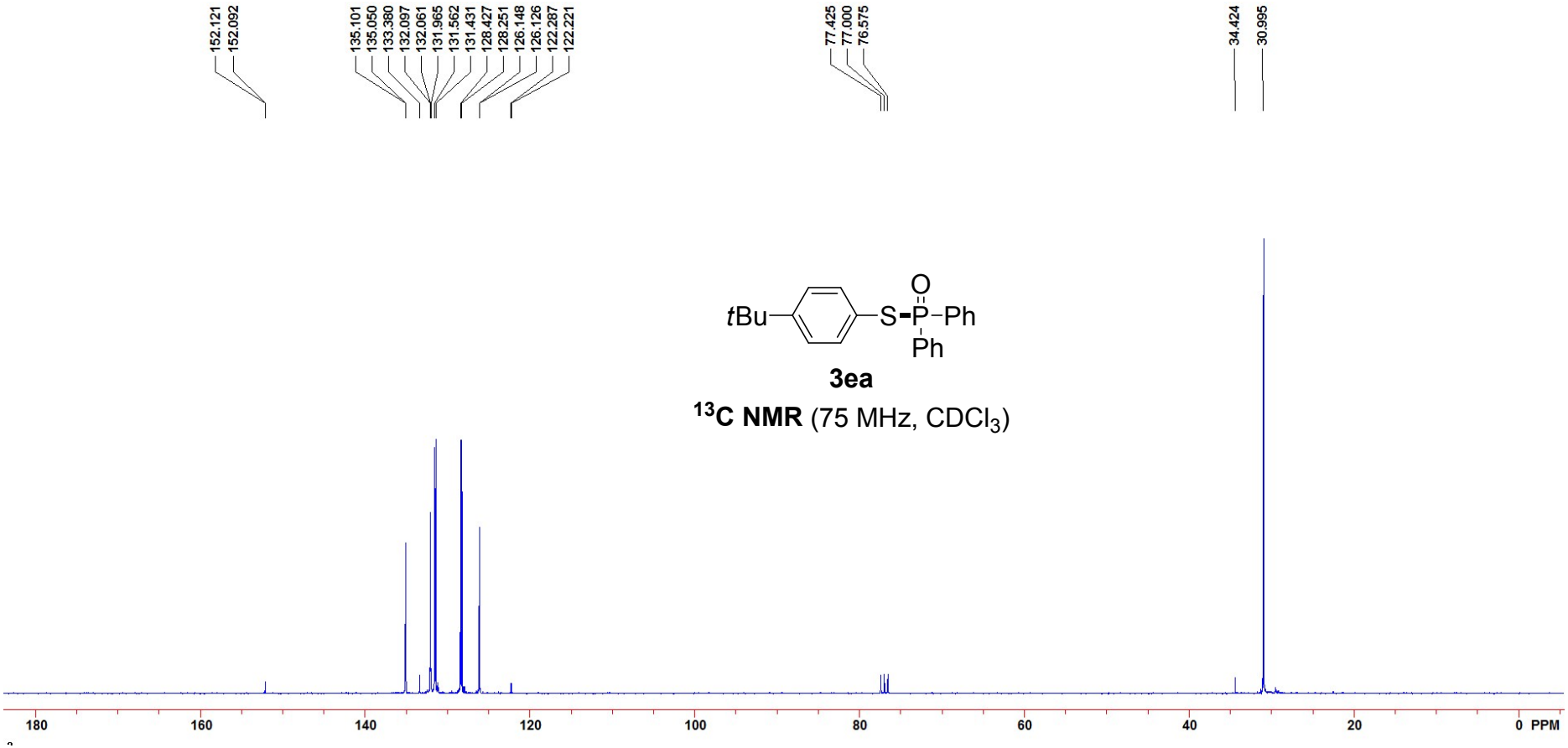
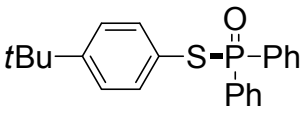
10
8
6
4
2
0
PPM



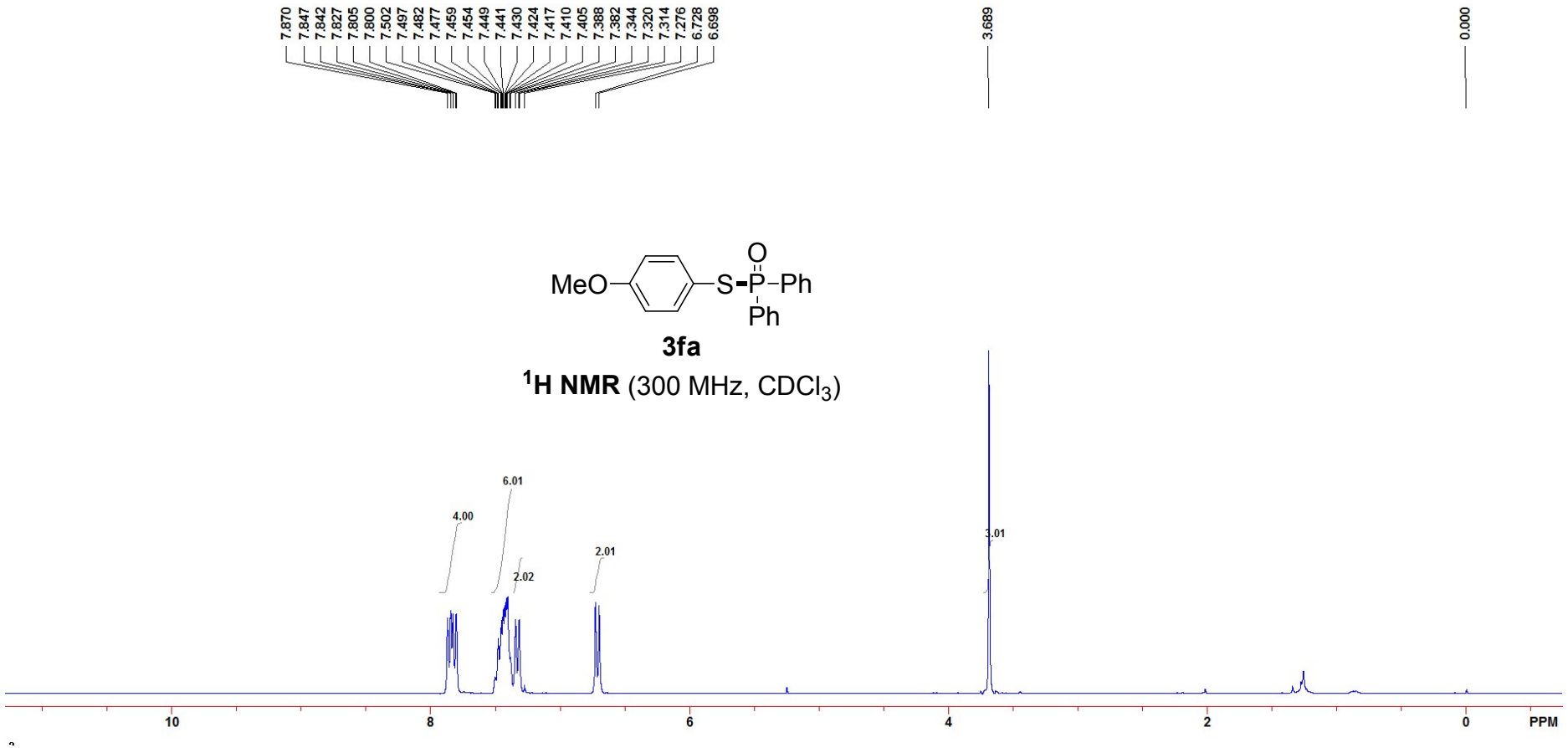
3ea

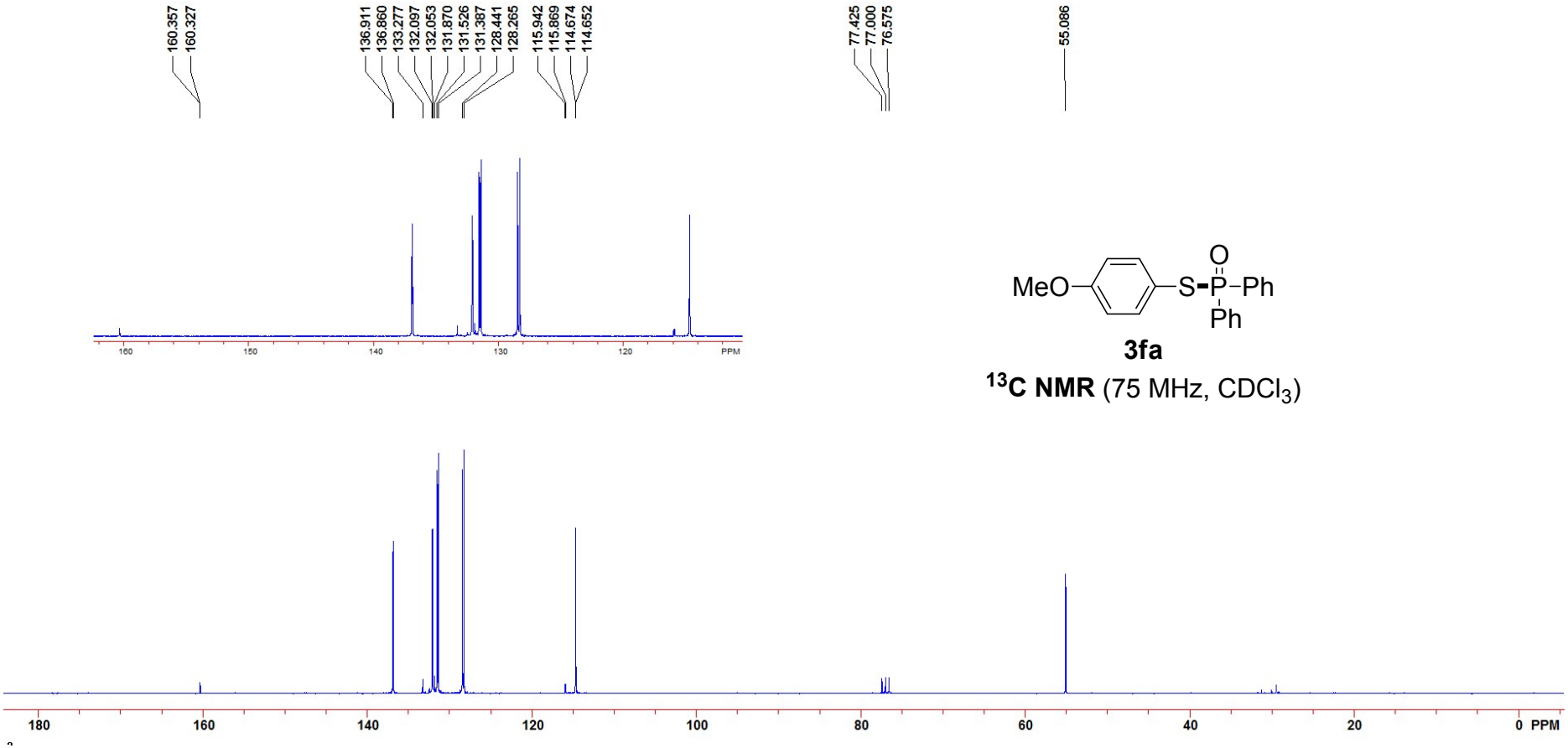
^{13}C NMR (75 MHz, $CDCl_3$)



3ea

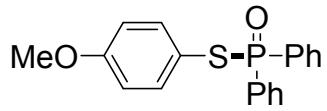
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.870
7.847
7.842
7.827
7.805
7.800
7.502
7.497
7.482
7.477
7.459
7.454
7.449
7.441
7.430
7.424
7.417
7.410
7.405
7.388
7.382
7.344
7.320
7.314
7.276
6.728
6.698
3.689
0.000
MeO
S
P
O
Ph
Ph
3fa
1H NMR (300 MHz, CDCl3)
6.01
4.00
2.02
2.01
3.01
10
8
6
4
2
0
PPM



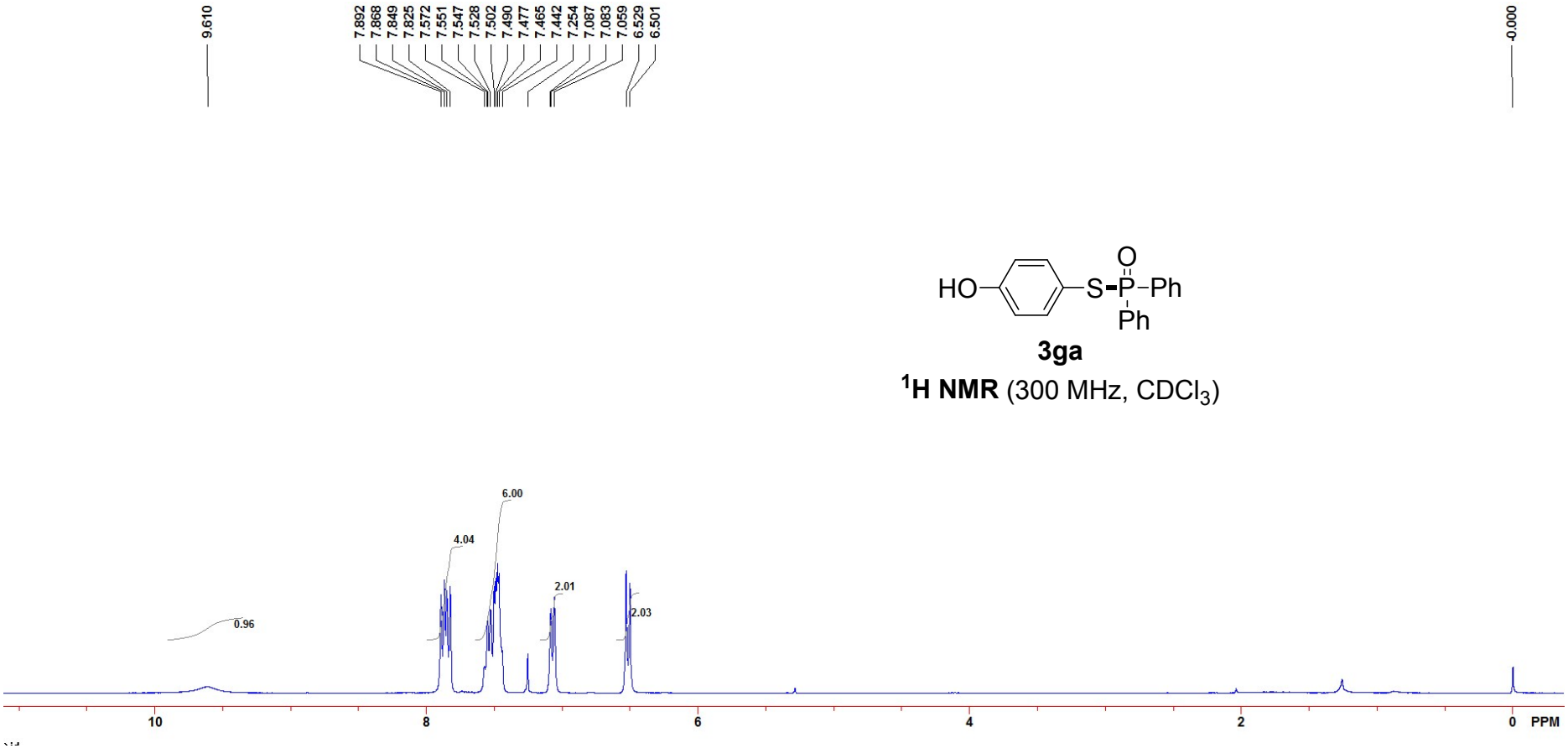
3fa

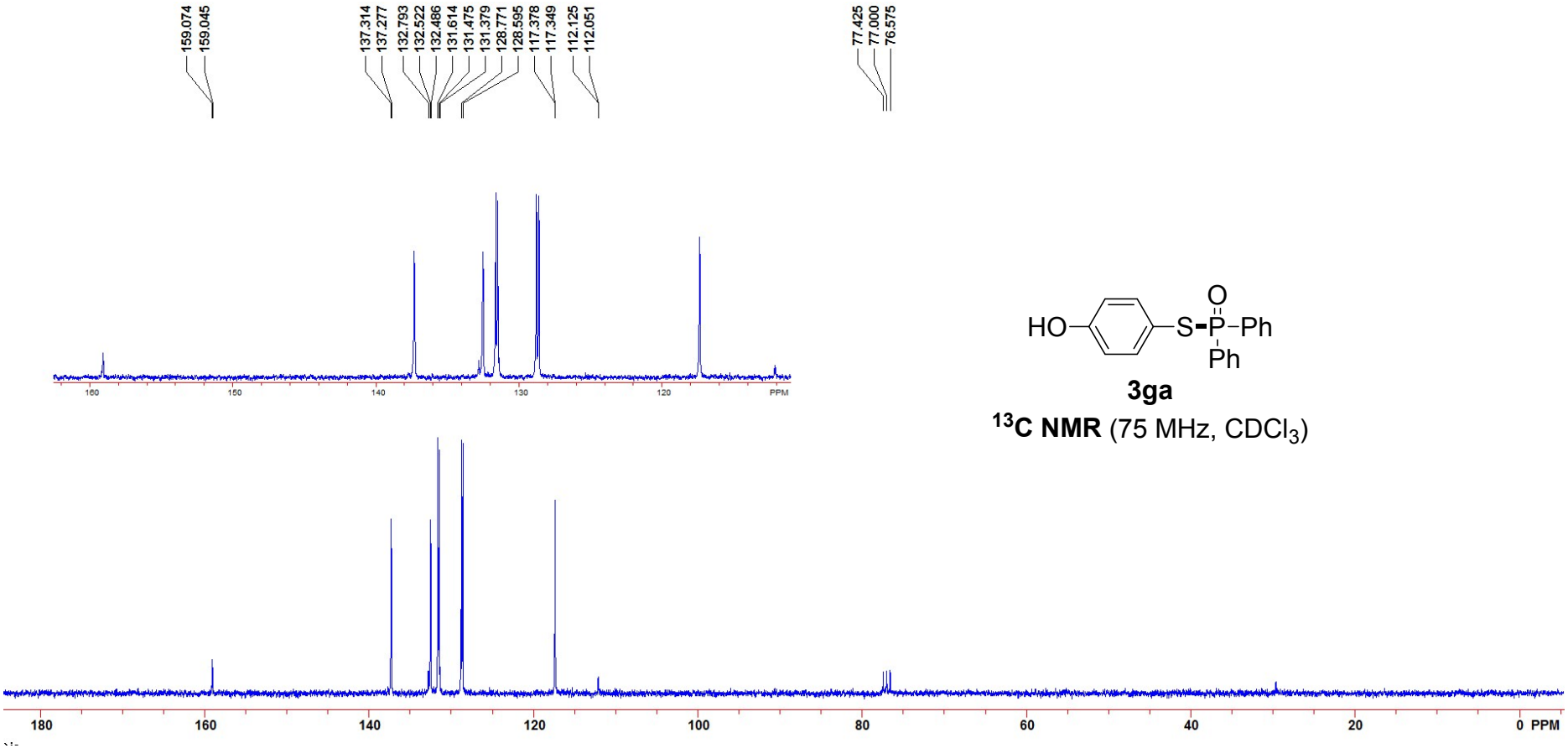
^{13}C NMR (75 MHz, $CDCl_3$)



3fa

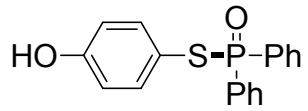
^{31}P NMR (121.5 MHz, $CDCl_3$)


9.610
7.892
7.868
7.849
7.825
7.572
7.551
7.547
7.528
7.502
7.490
7.477
7.465
7.442
7.254
7.087
7.083
7.059
6.529
6.501
-0.000
HO
S
P
O
Ph
Ph
3ga
1H NMR (300 MHz, CDCl3)
6.00
4.04
2.01
2.03
0.96
10
8
6
4
2
0
PPM



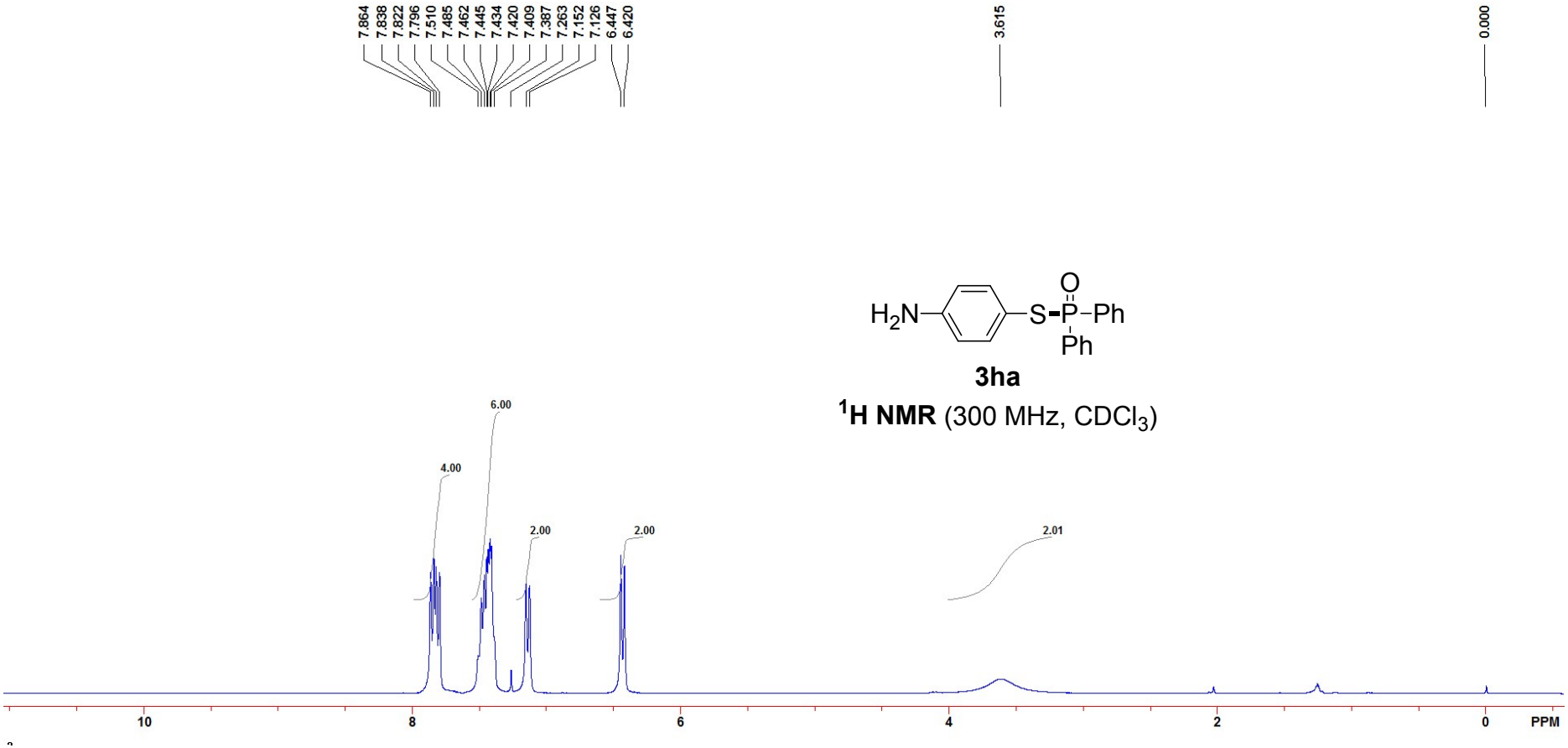
3ga

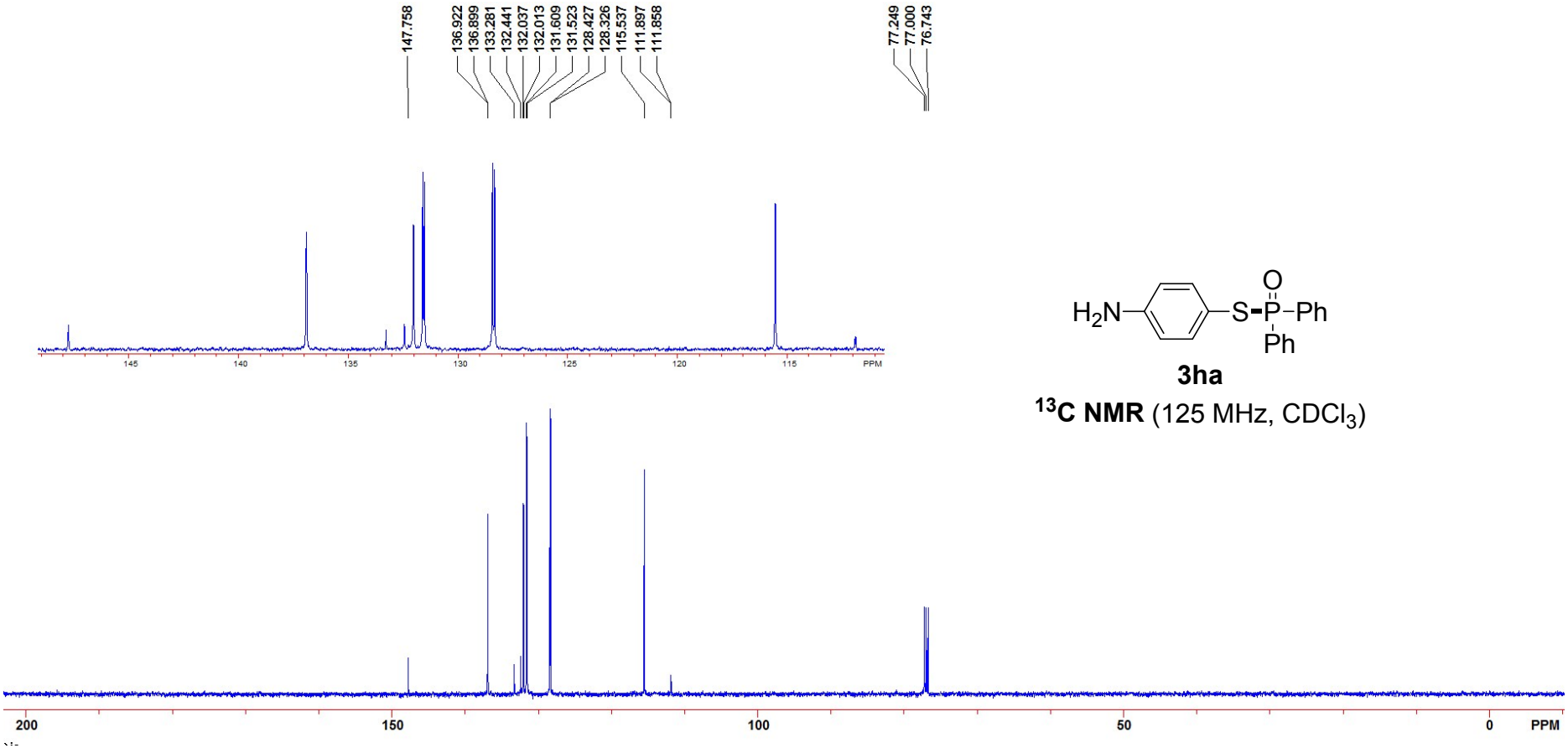
^{13}C NMR (75 MHz, $CDCl_3$)



3ga

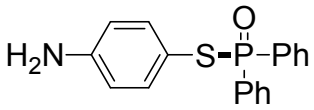
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.864
7.838
7.822
7.796
7.510
7.485
7.462
7.445
7.434
7.420
7.409
7.387
7.263
7.152
7.126
6.447
6.420
3.615
0.000
H_2N
S
P
O
Ph
Ph
3ha
^{1}H NMR (300 MHz, $CDCl_3$)
6.00
4.00
2.00
2.00
2.01
10
8
6
4
2
0
PPM



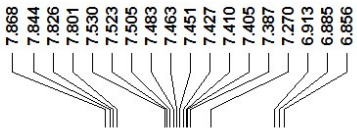
3ha

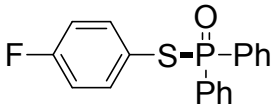
^{13}C NMR (125 MHz, $CDCl_3$)



3ha

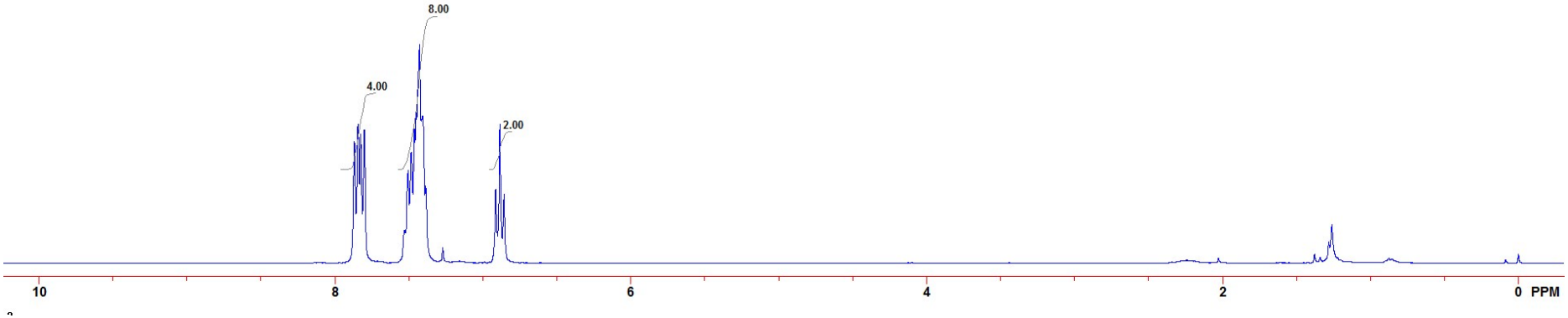
^{31}P NMR (121.5 MHz, $CDCl_3$)

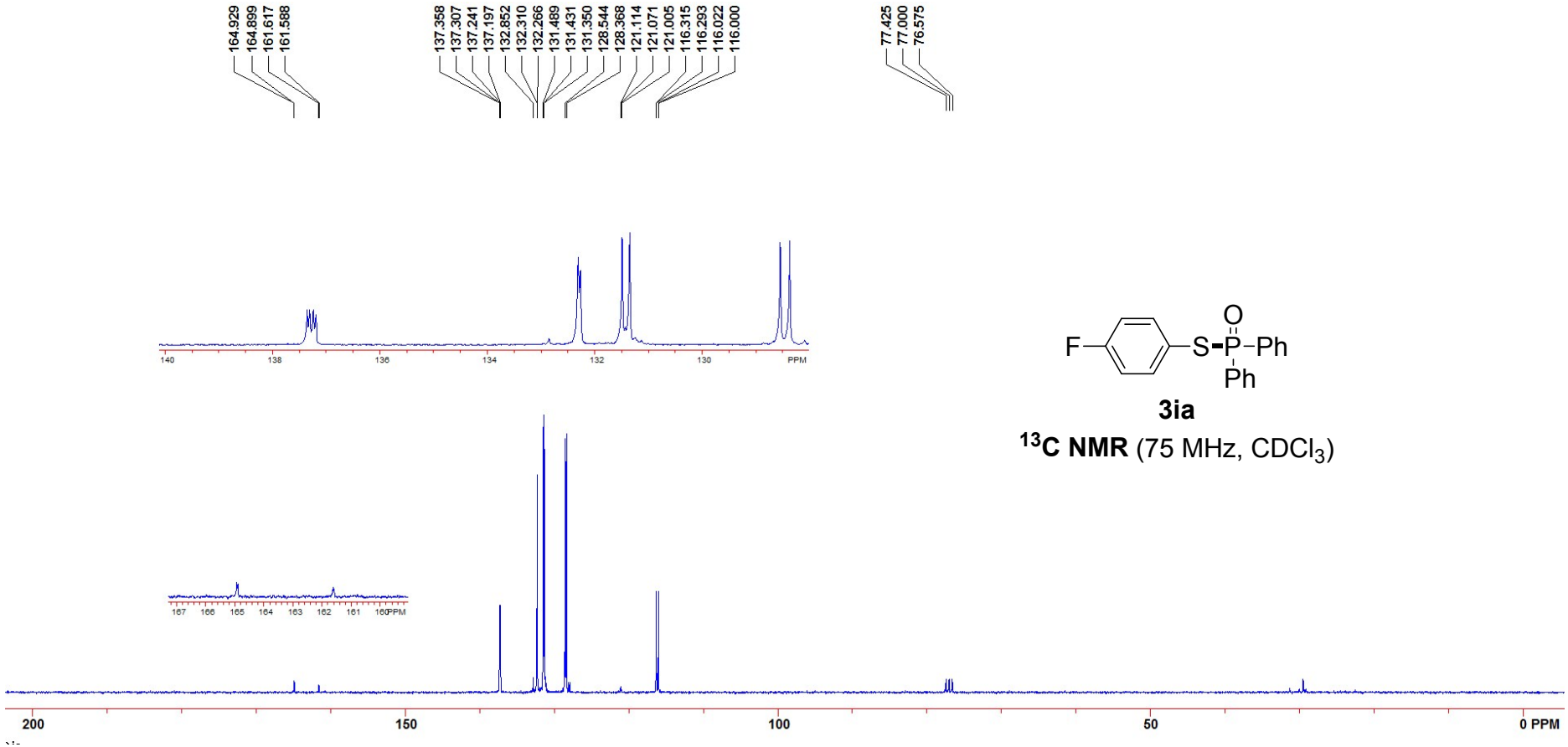




3ia

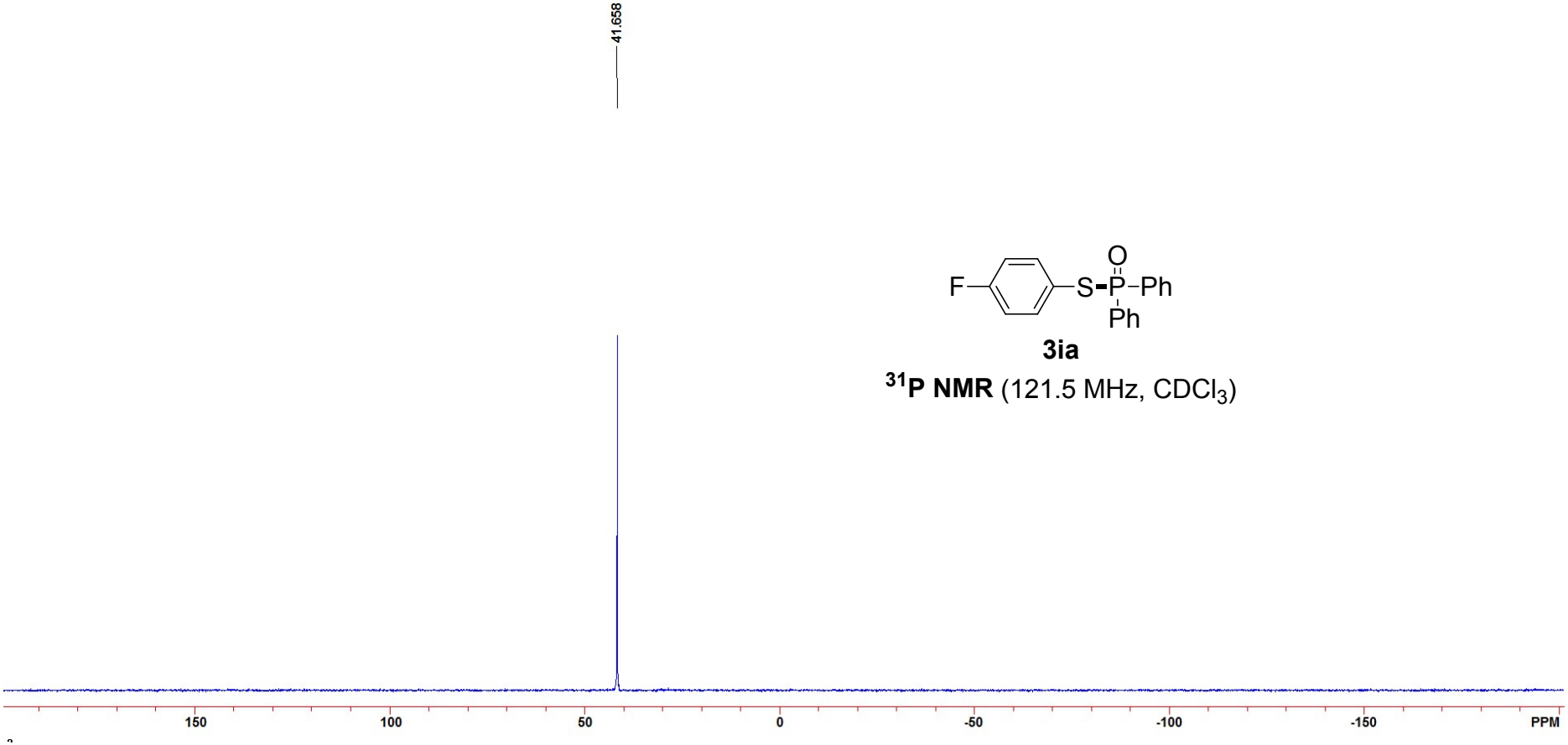
^{1}H NMR (300 MHz, $CDCl_3$)





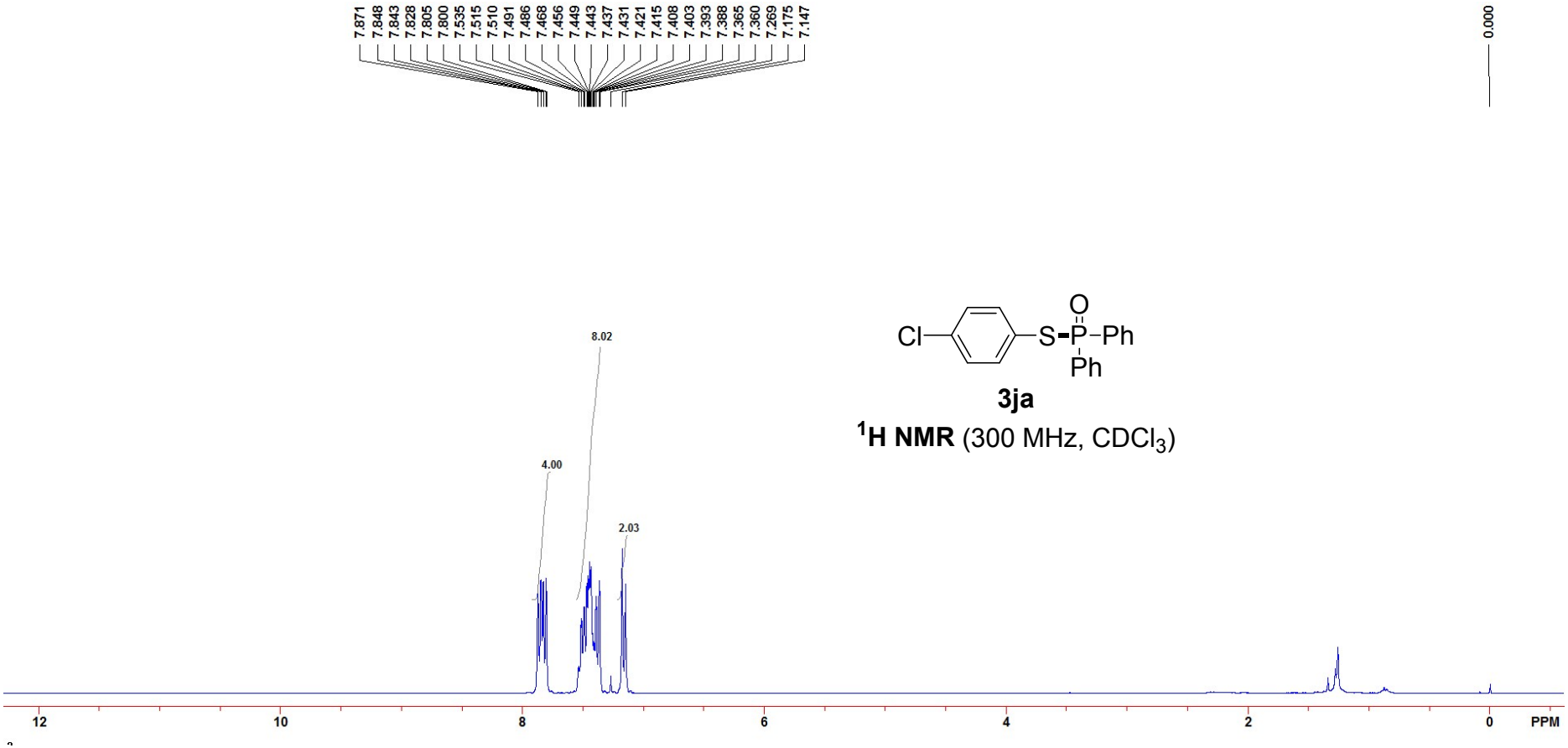
3ia

^{13}C NMR (75 MHz, $CDCl_3$)



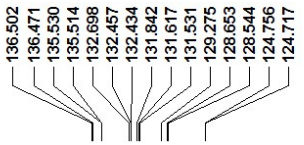
3ia

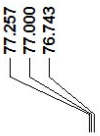
^{31}P NMR (121.5 MHz, $CDCl_3$)

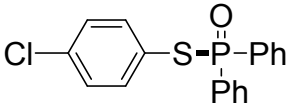


3ja

^{1}H NMR (300 MHz, $CDCl_3$)

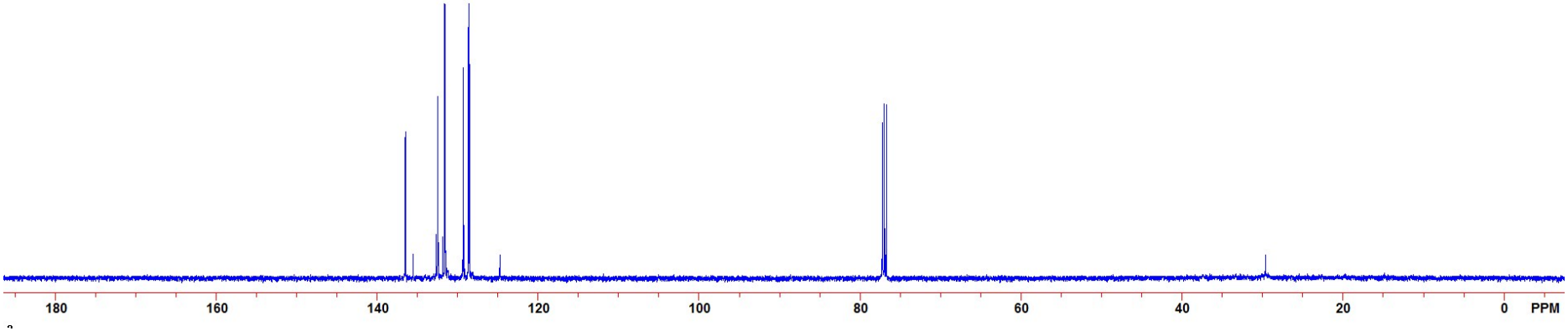


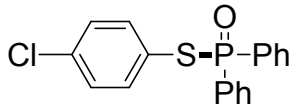




3ja

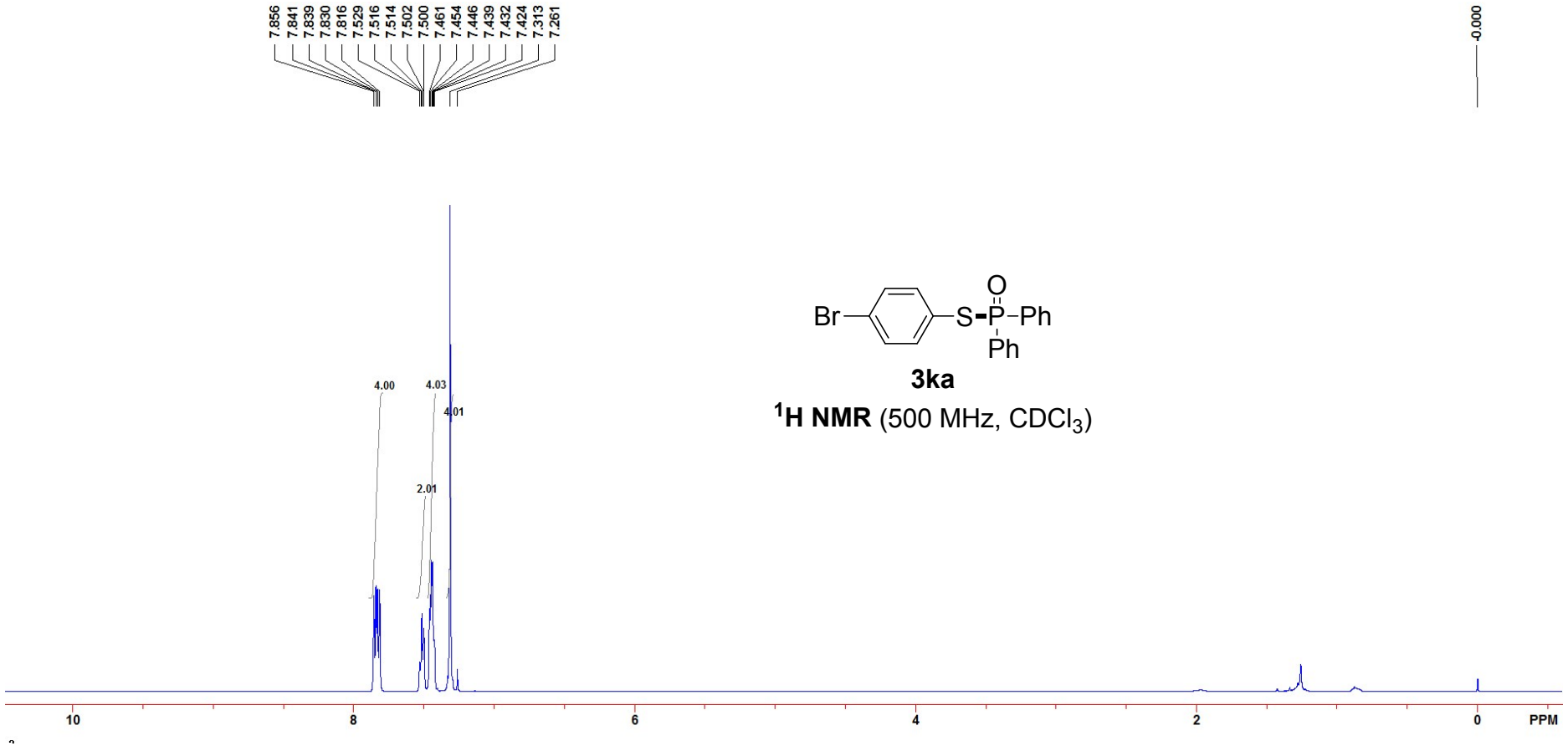
^{13}C NMR (125 MHz, $CDCl_3$)





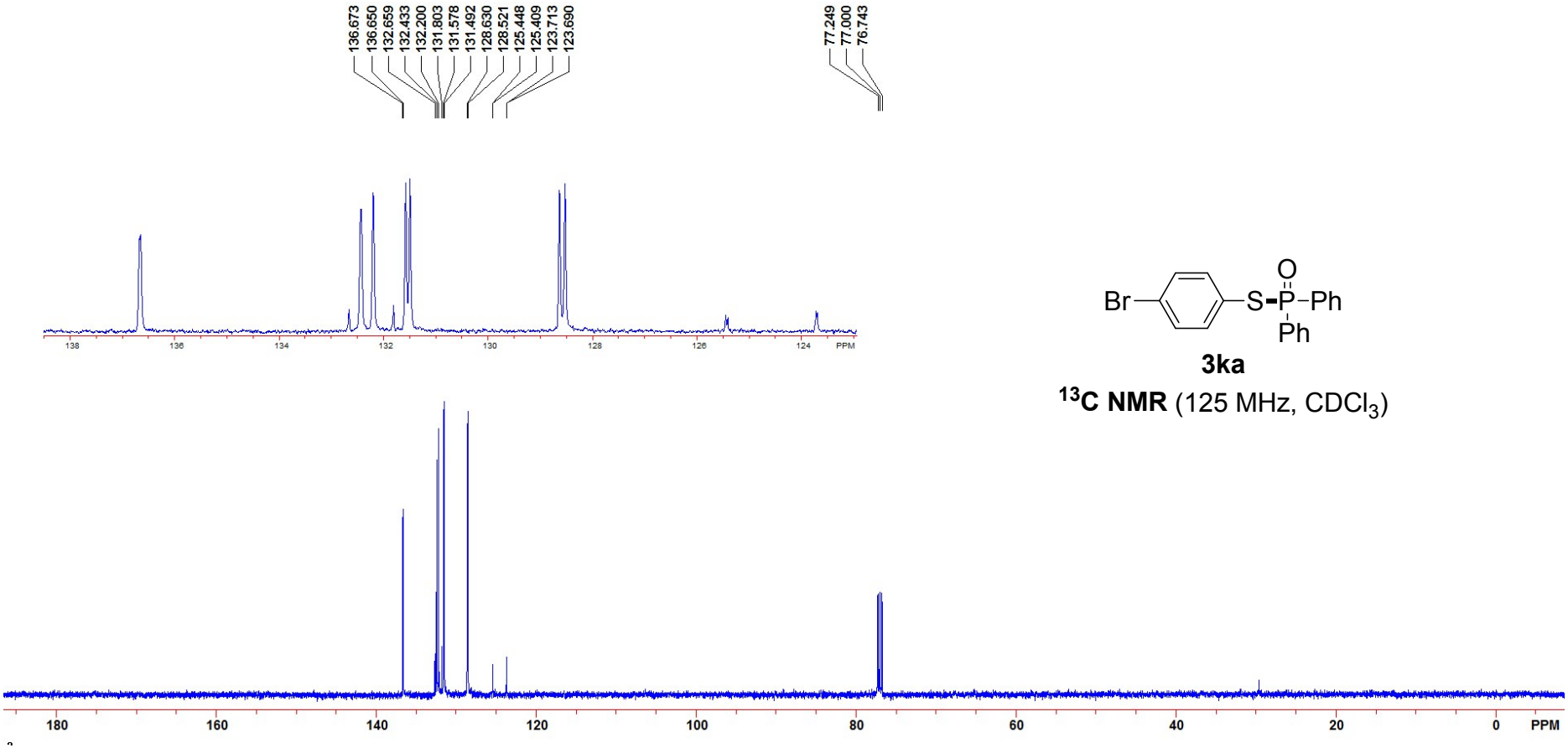
3ja

^{31}P NMR (121.5 MHz, $CDCl_3$)



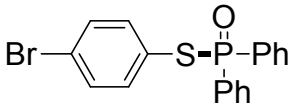
3ka

^{1}H NMR (500 MHz, $CDCl_3$)



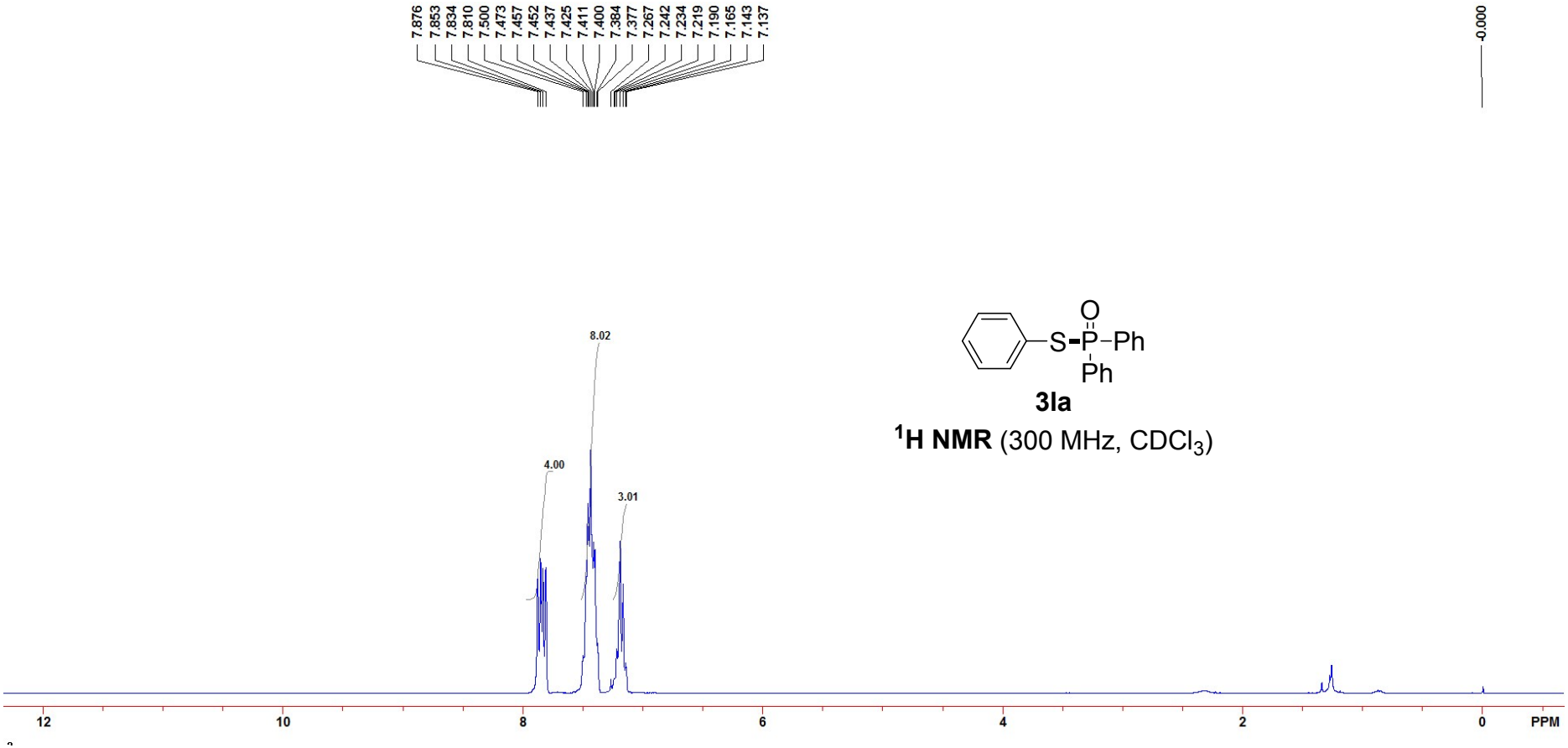
3ka

^{13}C NMR (125 MHz, $CDCl_3$)



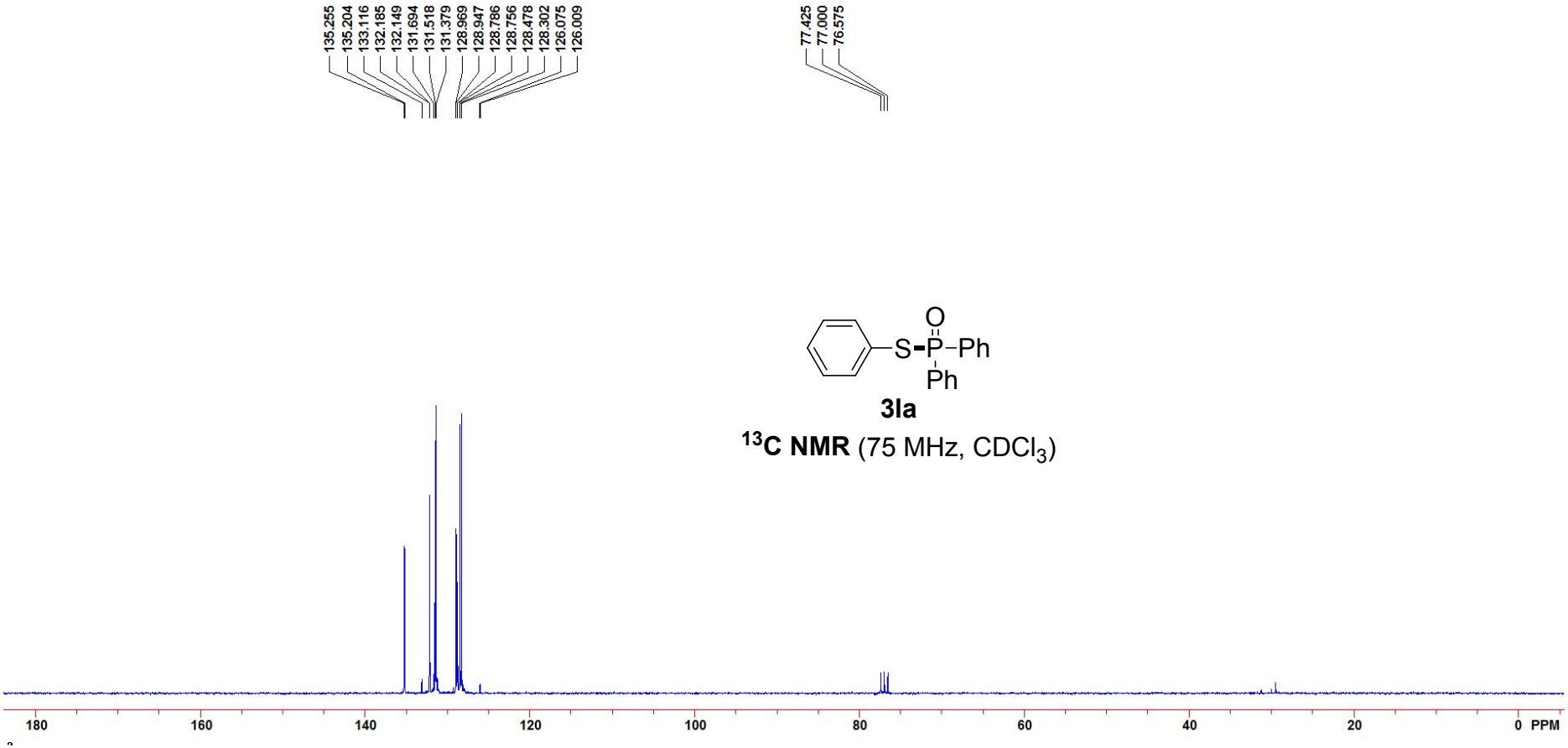
3ka

^{31}P NMR (121.5 MHz, $CDCl_3$)



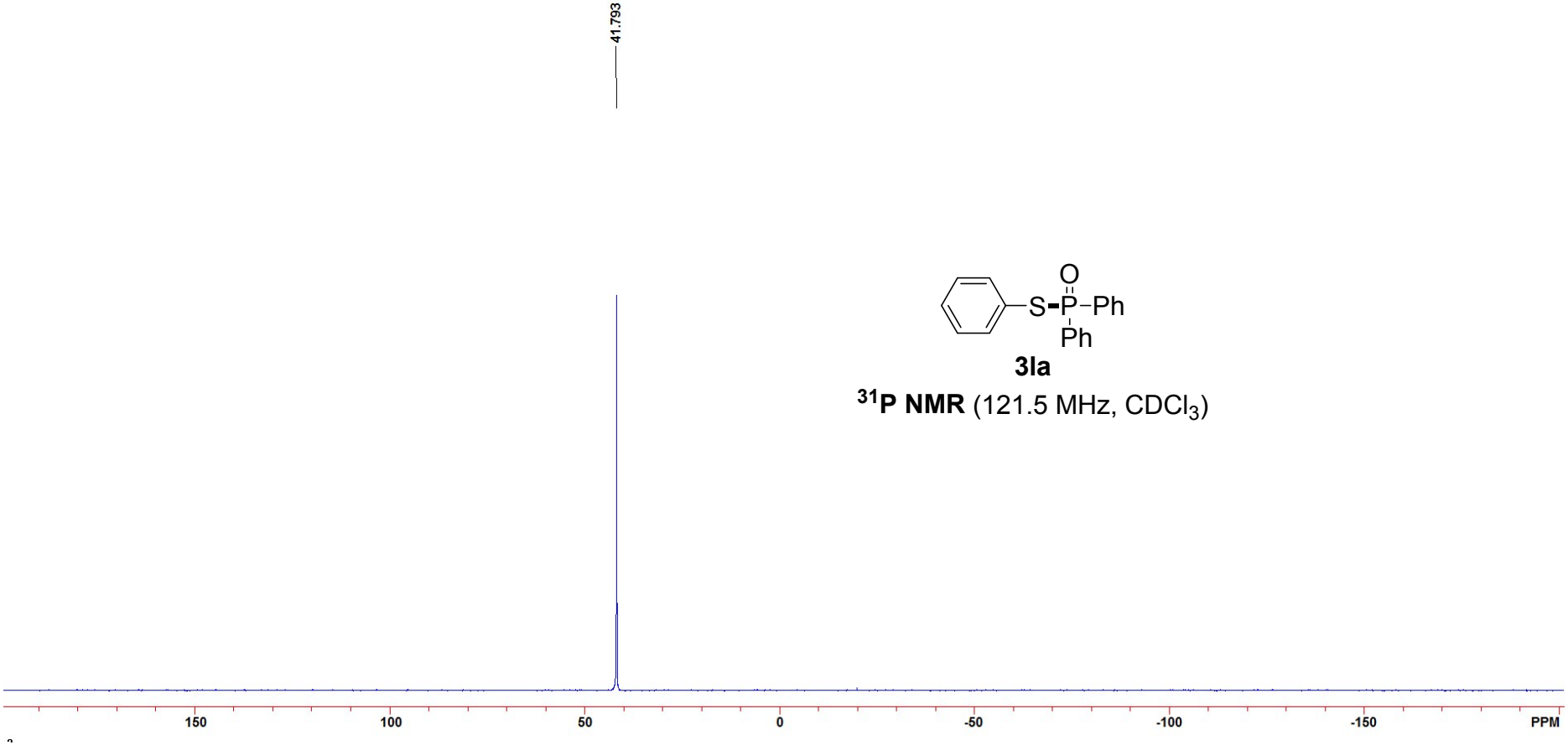
3la

^{1}H NMR (300 MHz, $CDCl_3$)



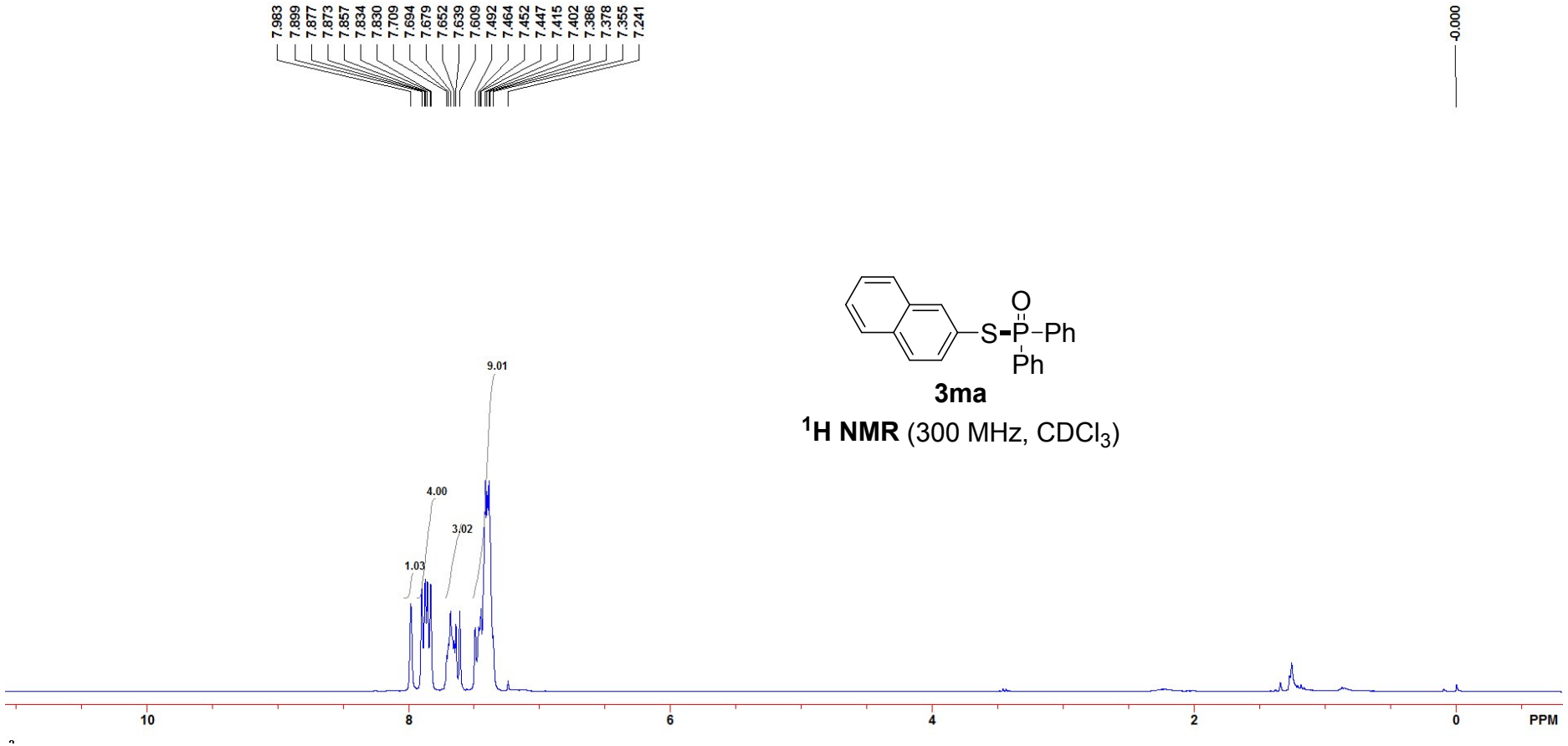
3la

^{13}C NMR (75 MHz, $CDCl_3$)



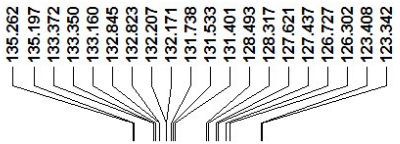
3la

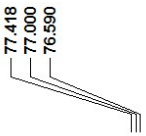
^{31}P NMR (121.5 MHz, $CDCl_3$)

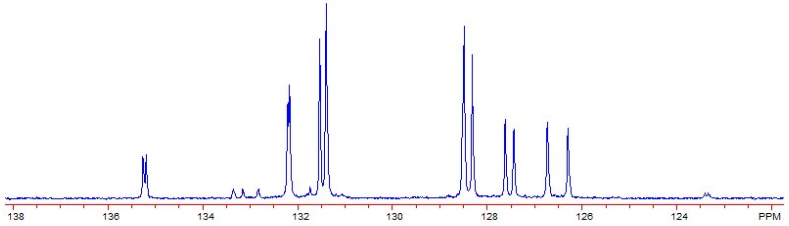


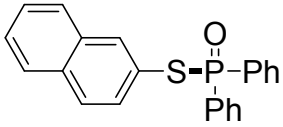
3ma

^{1}H NMR (300 MHz, $CDCl_3$)



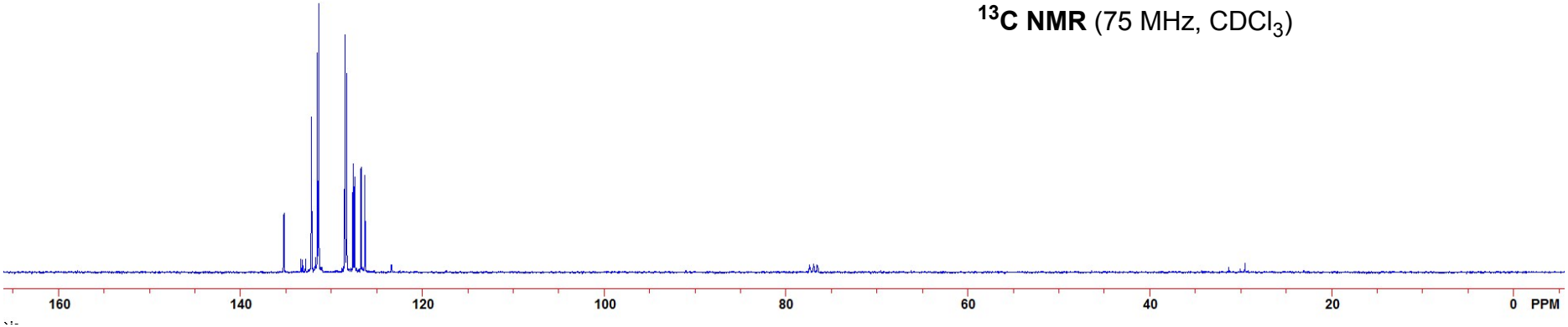


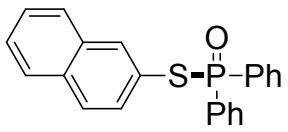




3ma

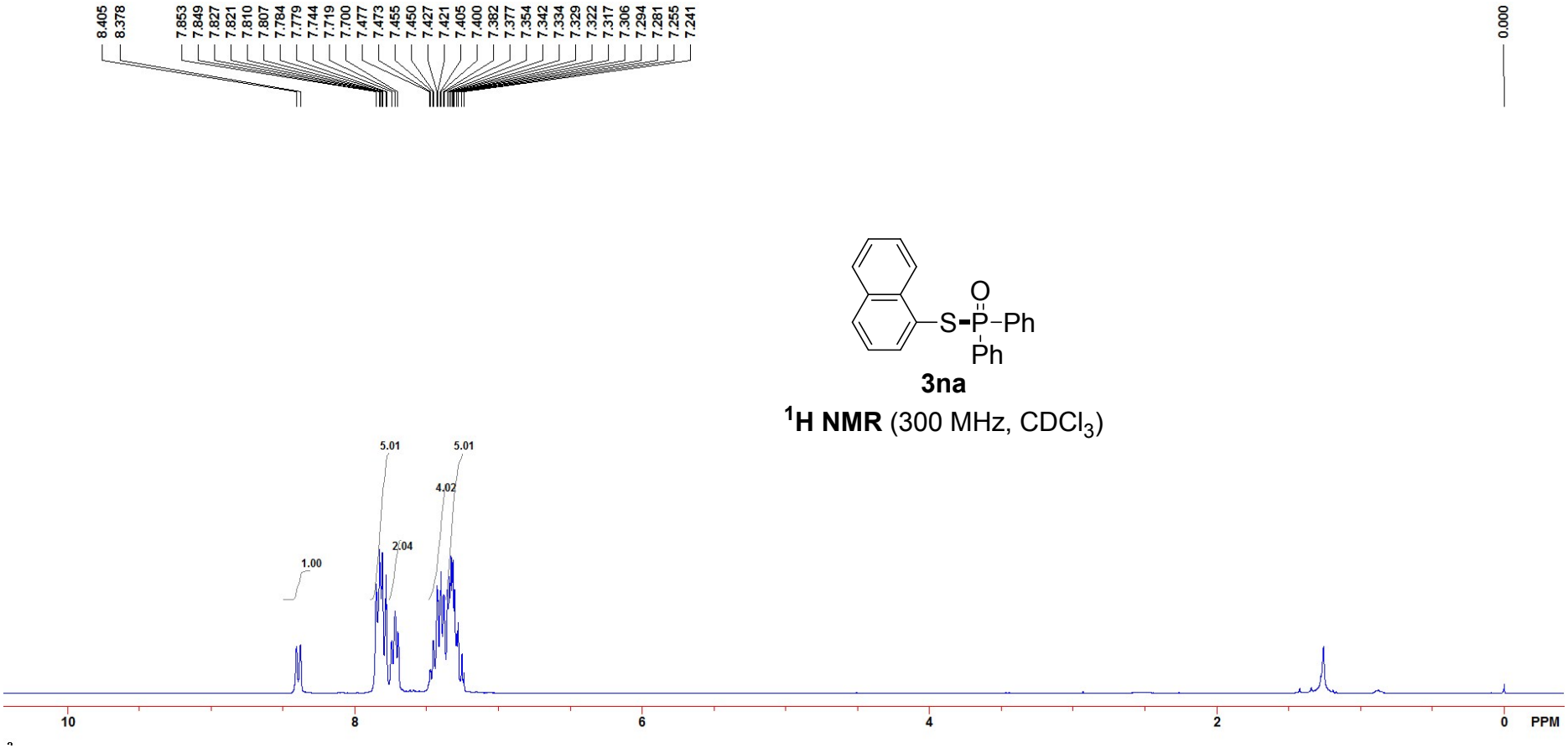
^{13}C NMR (75 MHz, $CDCl_3$)

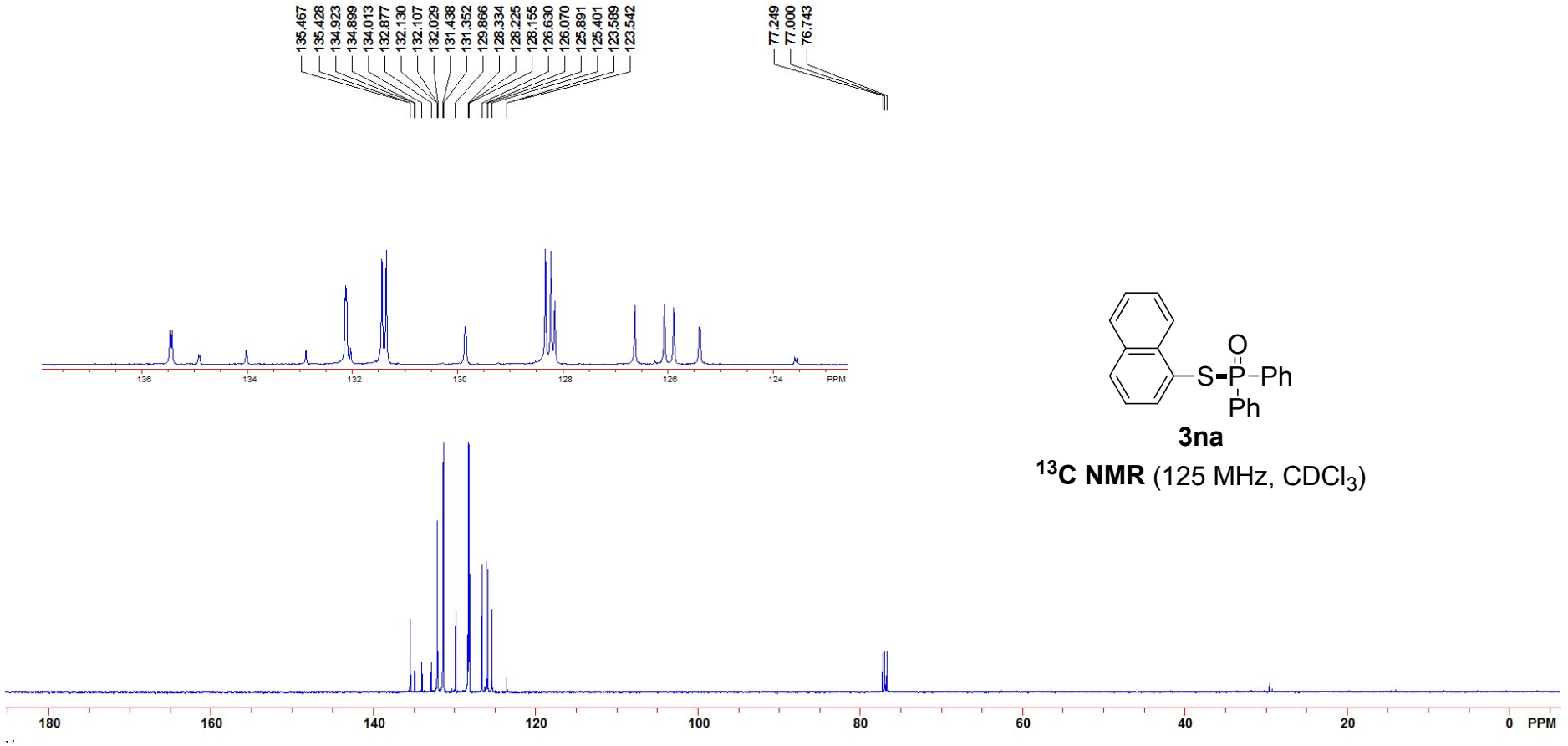




3ma

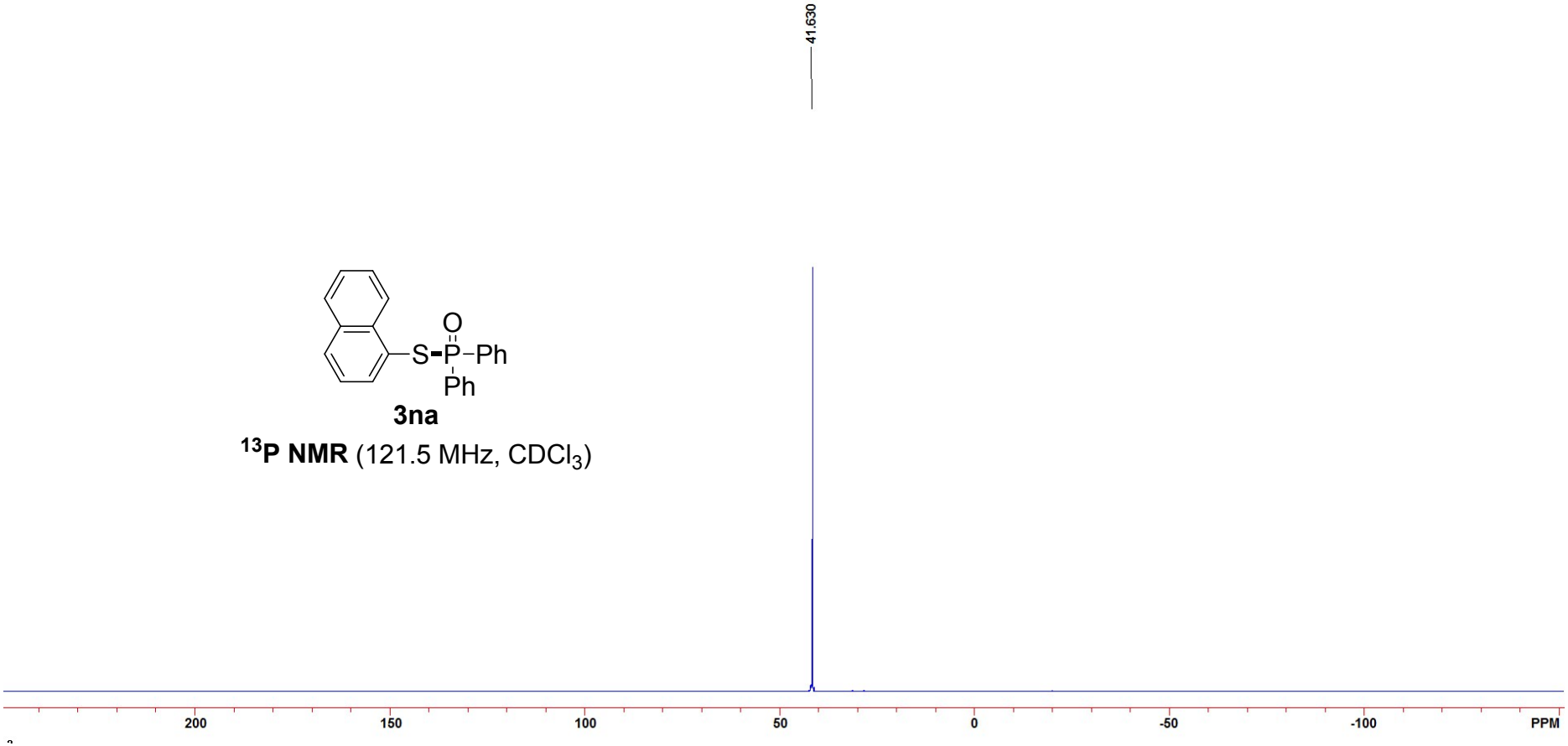
^{31}P NMR (121.5 MHz, $CDCl_3$)


8.405
8.378
7.853
7.849
7.827
7.821
7.810
7.807
7.784
7.779
7.744
7.719
7.700
7.477
7.473
7.455
7.450
7.427
7.421
7.405
7.400
7.382
7.377
7.354
7.342
7.334
7.329
7.322
7.317
7.306
7.294
7.281
7.255
7.241
0.000
O
S
P
Ph
Ph
3na
1H NMR (300 MHz, CDCl3)
5.01
5.01
4.02
2.04
1.00
10
8
6
4
2
0
PPM



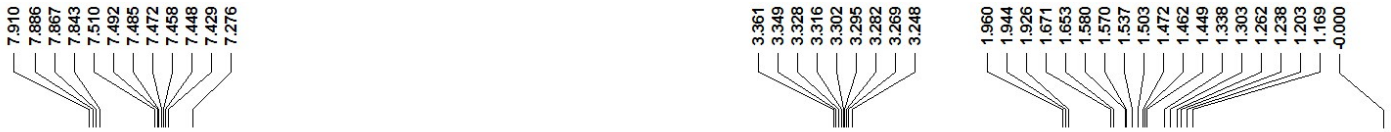
3na

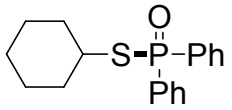
^{13}C NMR (125 MHz, $CDCl_3$)



3na

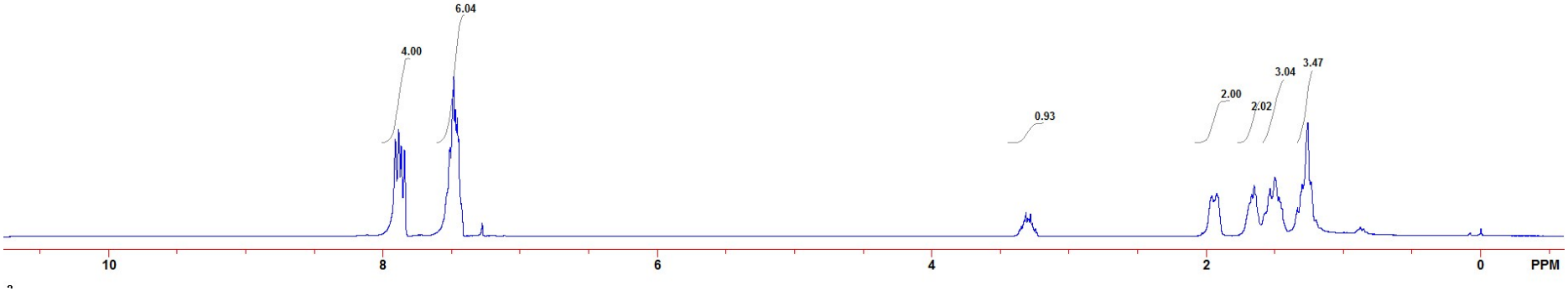
^{13}P NMR (121.5 MHz, $CDCl_3$)

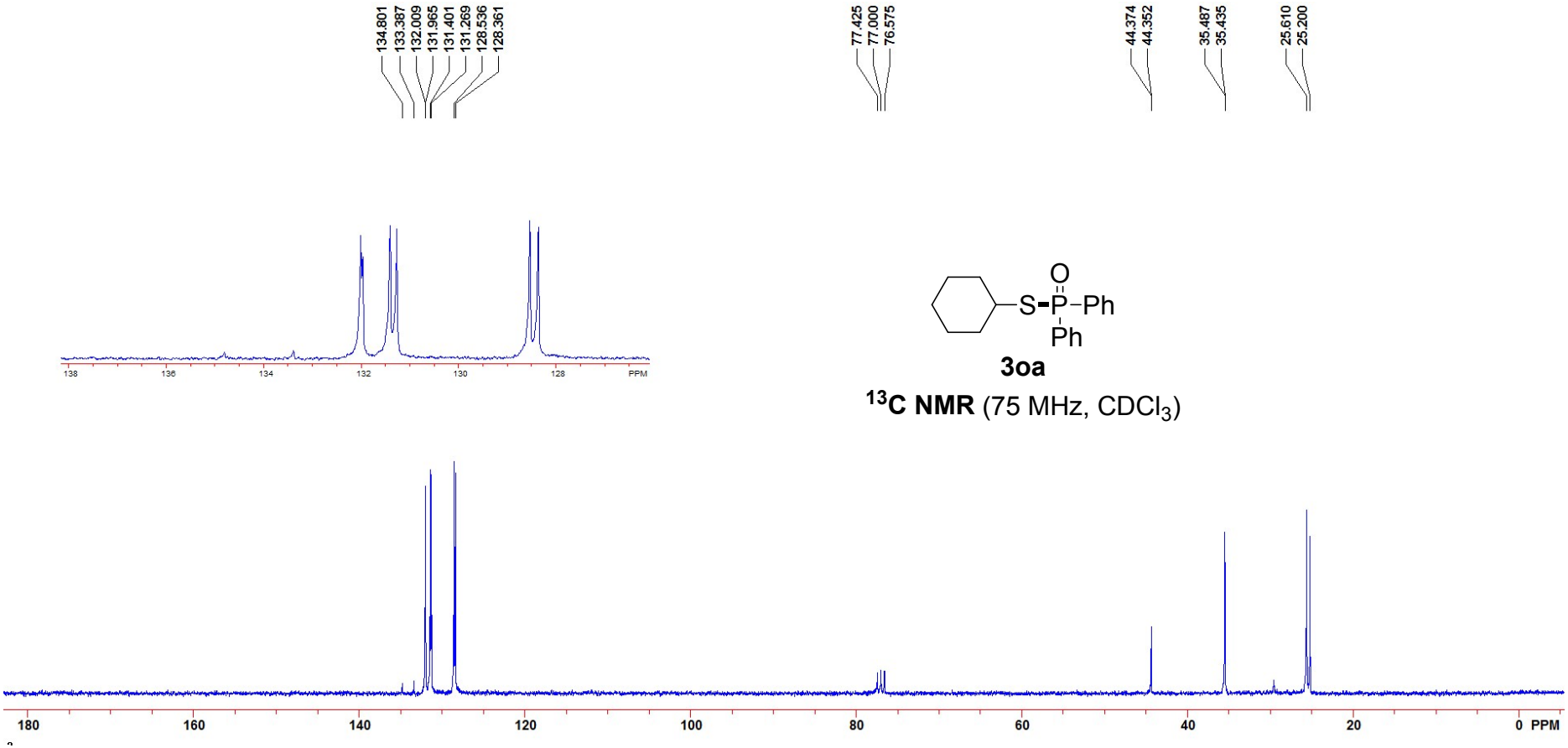




3oa

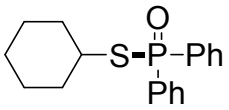
^{1}H NMR (300 MHz, $CDCl_3$)





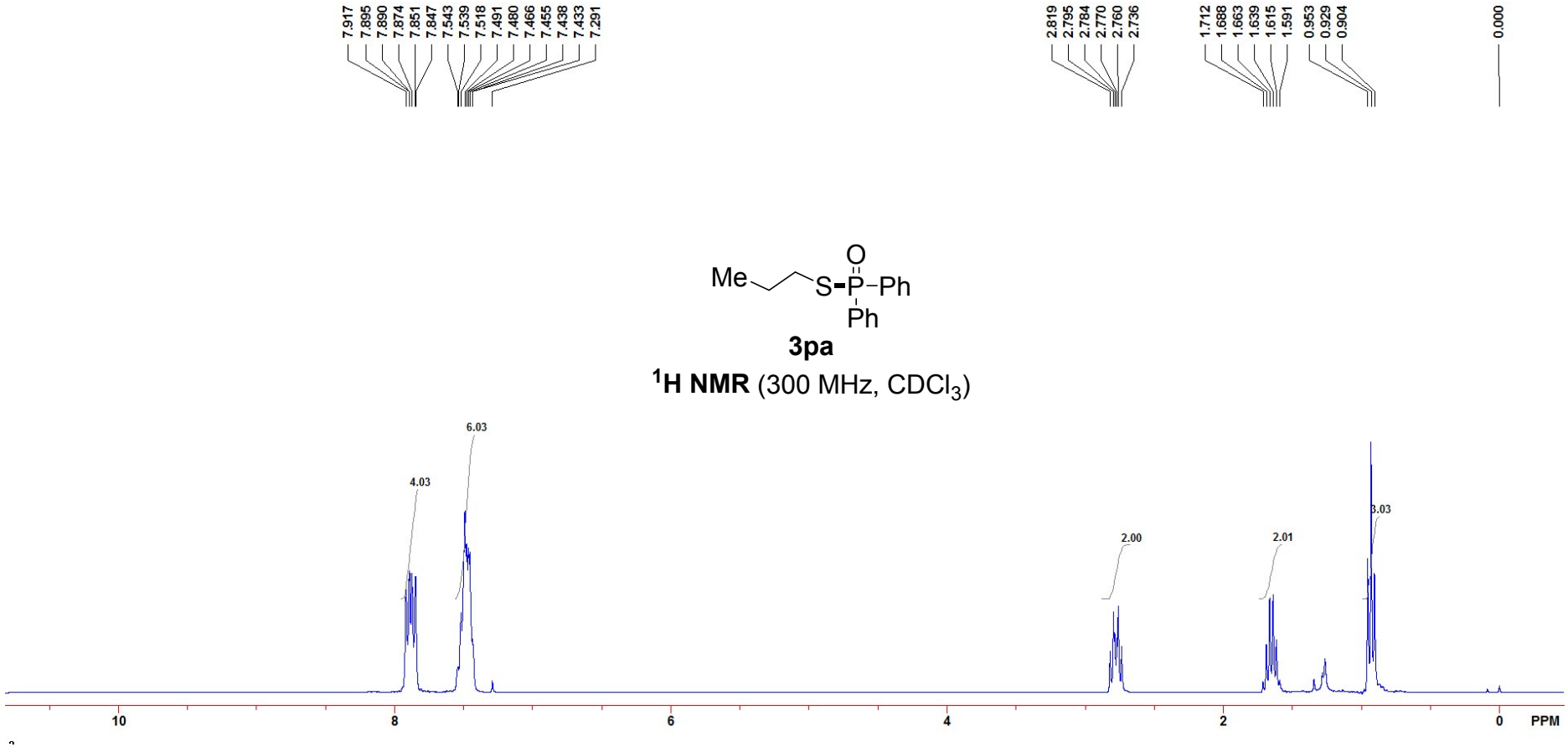
3oa

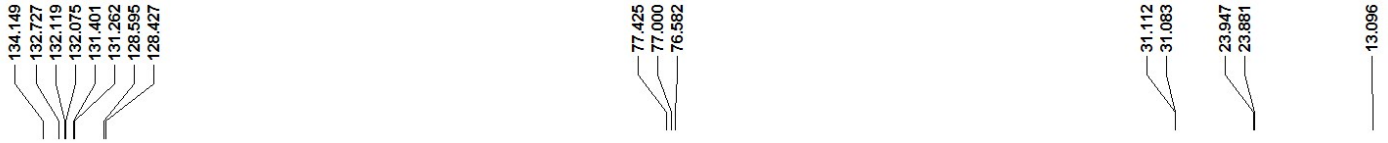
^{13}C NMR (75 MHz, $CDCl_3$)

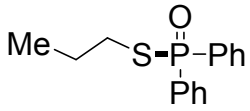


3oa

^{31}P NMR (121.5 MHz, $CDCl_3$)

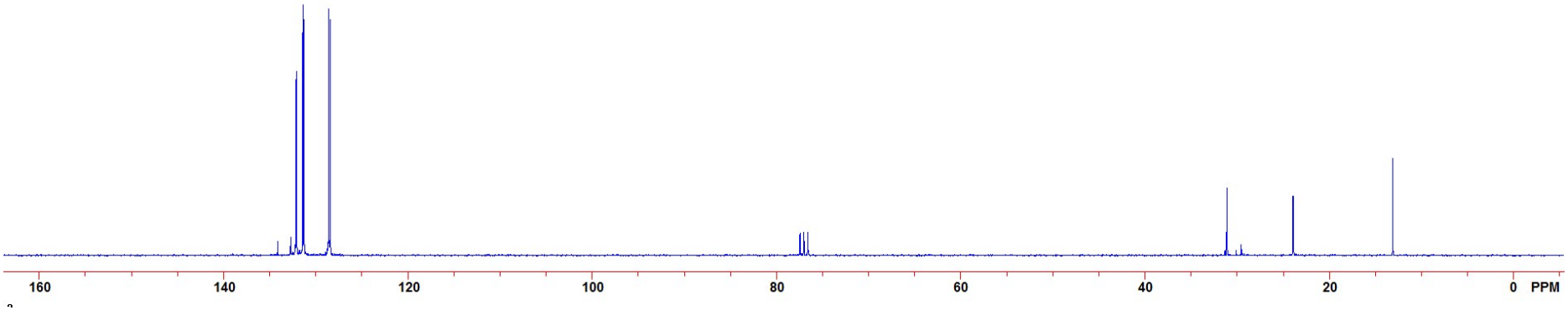

7.917
7.895
7.890
7.874
7.851
7.847
7.543
7.539
7.518
7.491
7.480
7.466
7.455
7.438
7.433
7.291
2.819
2.795
2.784
2.770
2.760
2.736
1.712
1.688
1.663
1.639
1.615
1.591
0.953
0.929
0.904
0.000
Me
S
P
O
Ph
Ph
3pa
1H NMR (300 MHz, CDCl3)
6.03
4.03
2.00
2.01
3.03
10
8
6
4
2
0
PPM

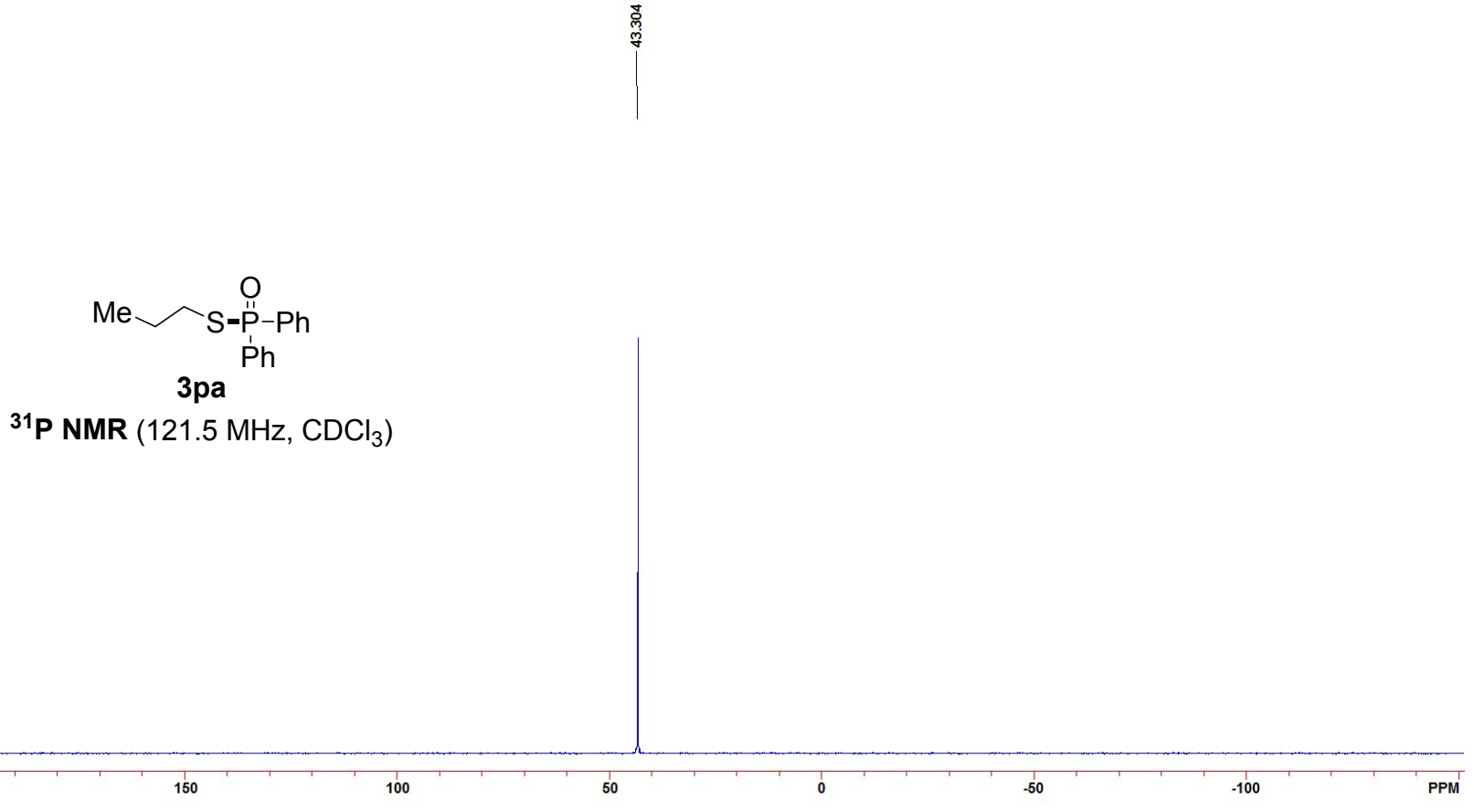




3pa

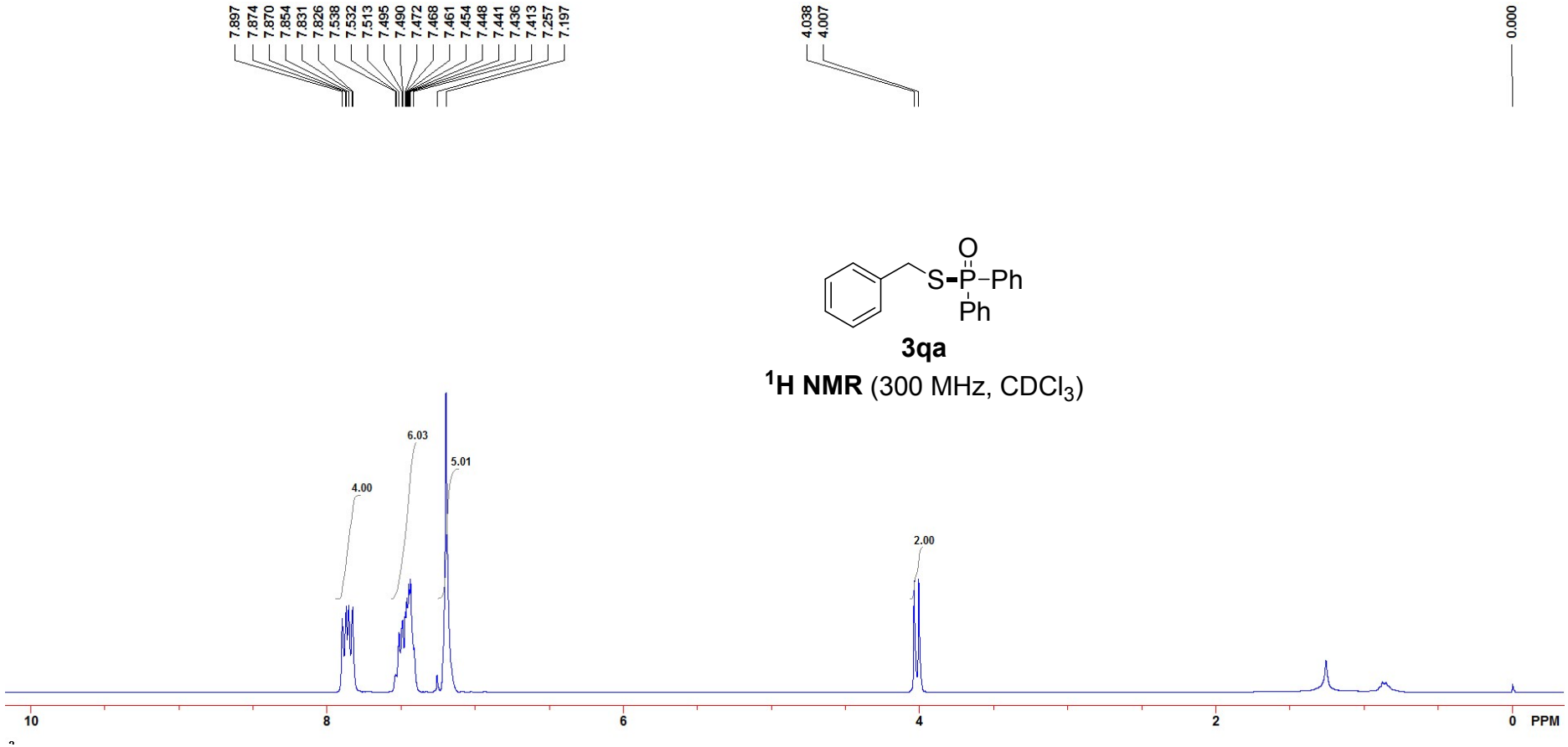
^{13}C NMR (125 MHz, $CDCl_3$)

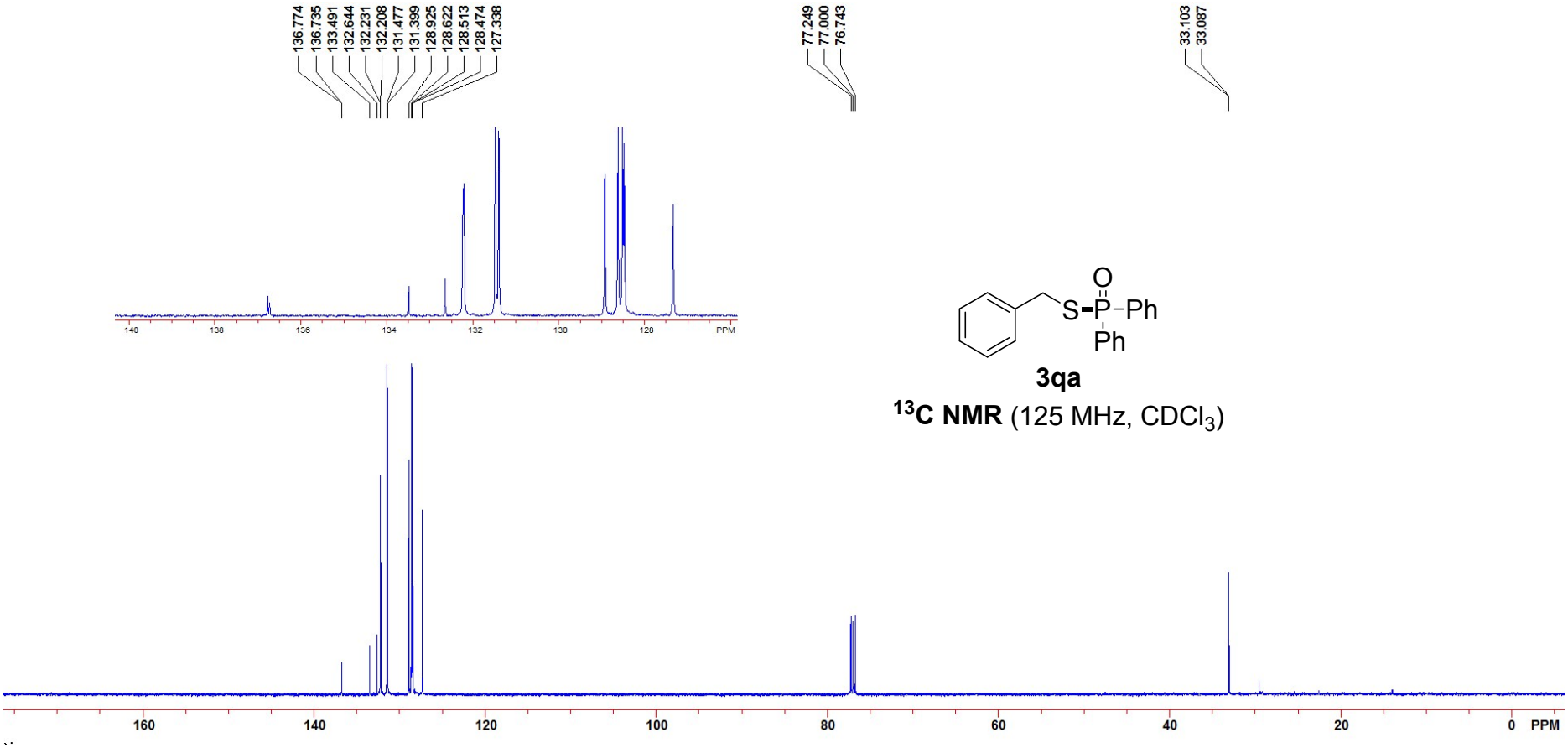


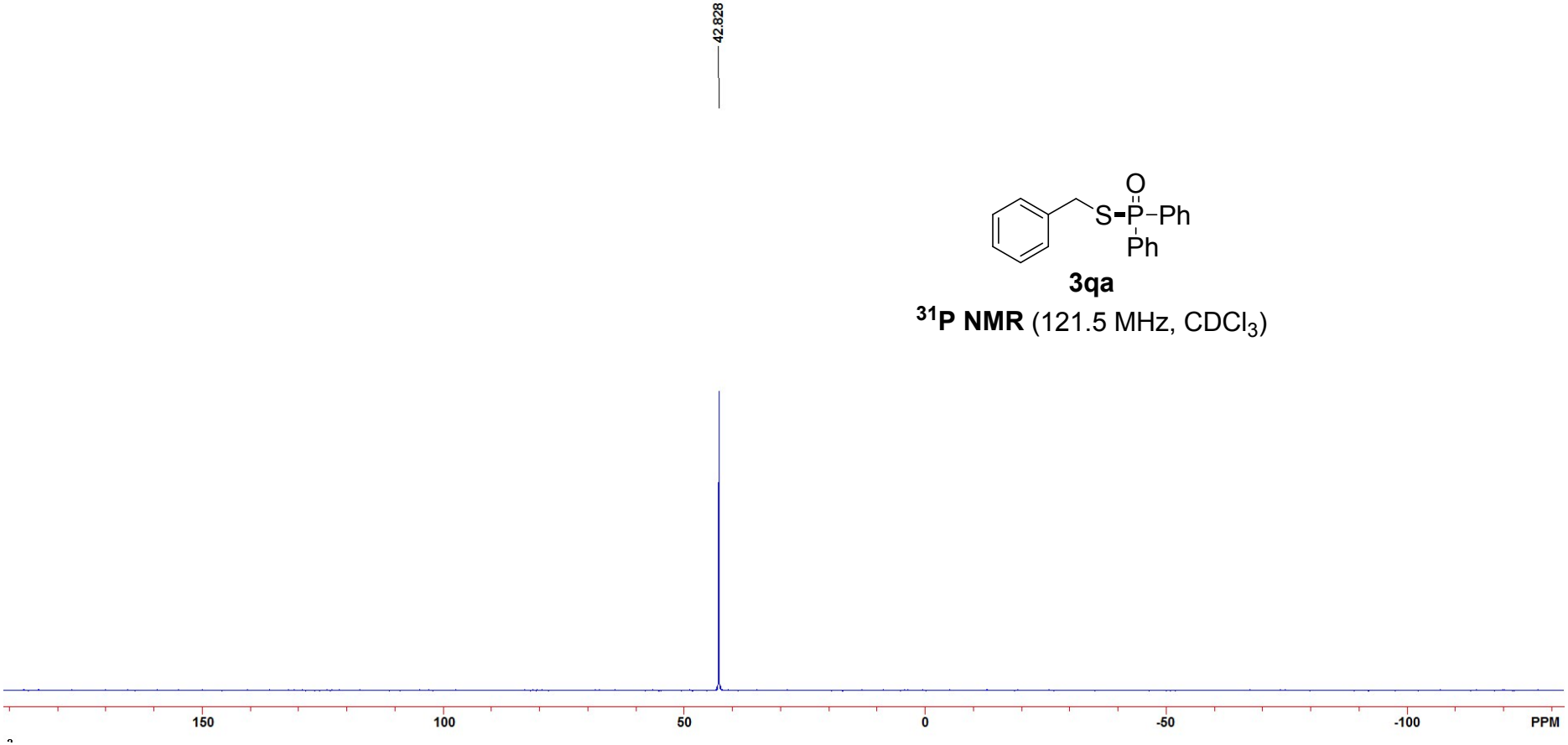


3pa

^{31}P NMR (121.5 MHz, $CDCl_3$)

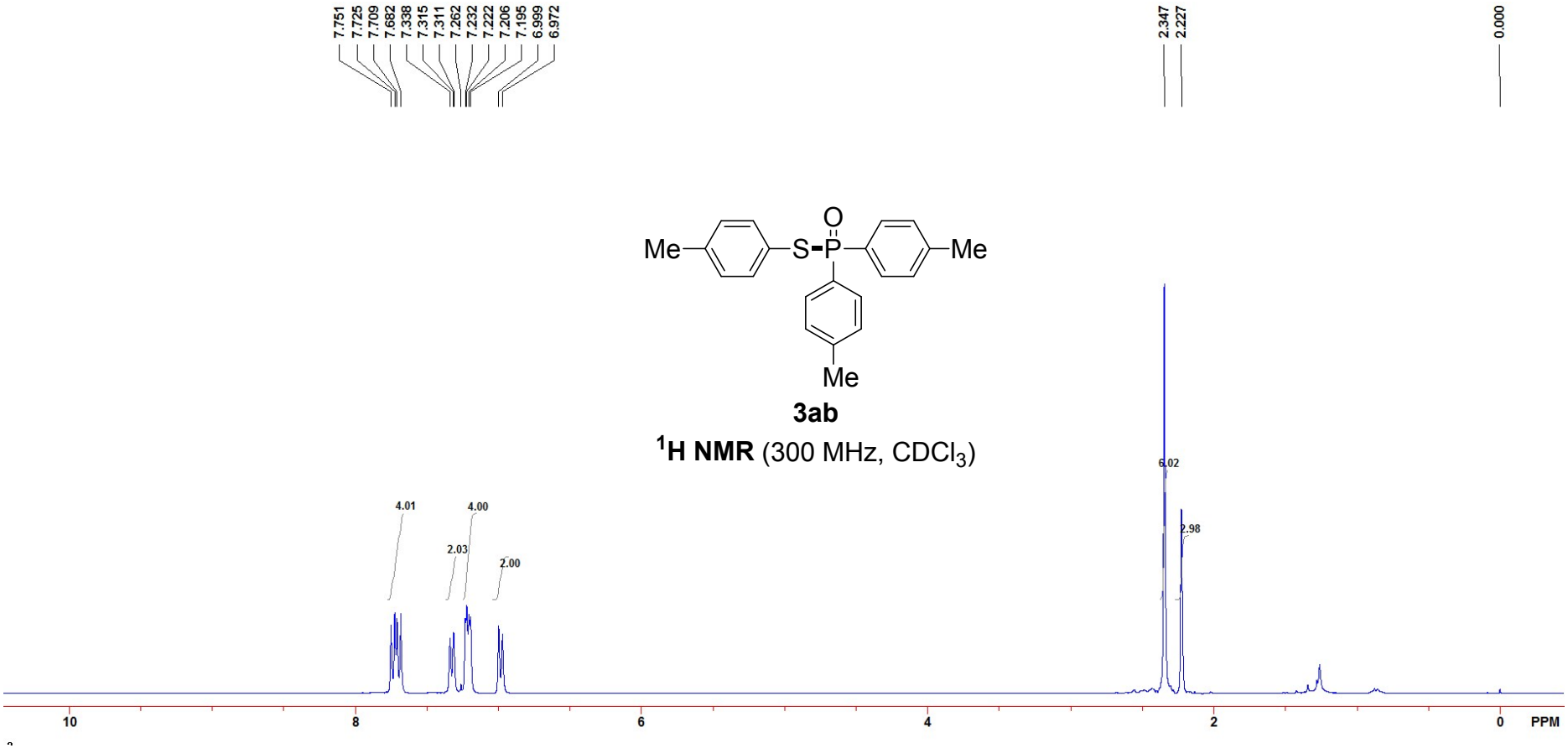

7.897
7.874
7.870
7.854
7.831
7.826
7.538
7.532
7.513
7.495
7.490
7.472
7.468
7.461
7.454
7.448
7.441
7.436
7.413
7.257
7.197
4.038
4.007
0.000
O
S
P
Ph
Ph
3qa
1H NMR (300 MHz, CDCl3)
6.03
5.01
4.00
2.00
10
8
6
4
2
0
PPM

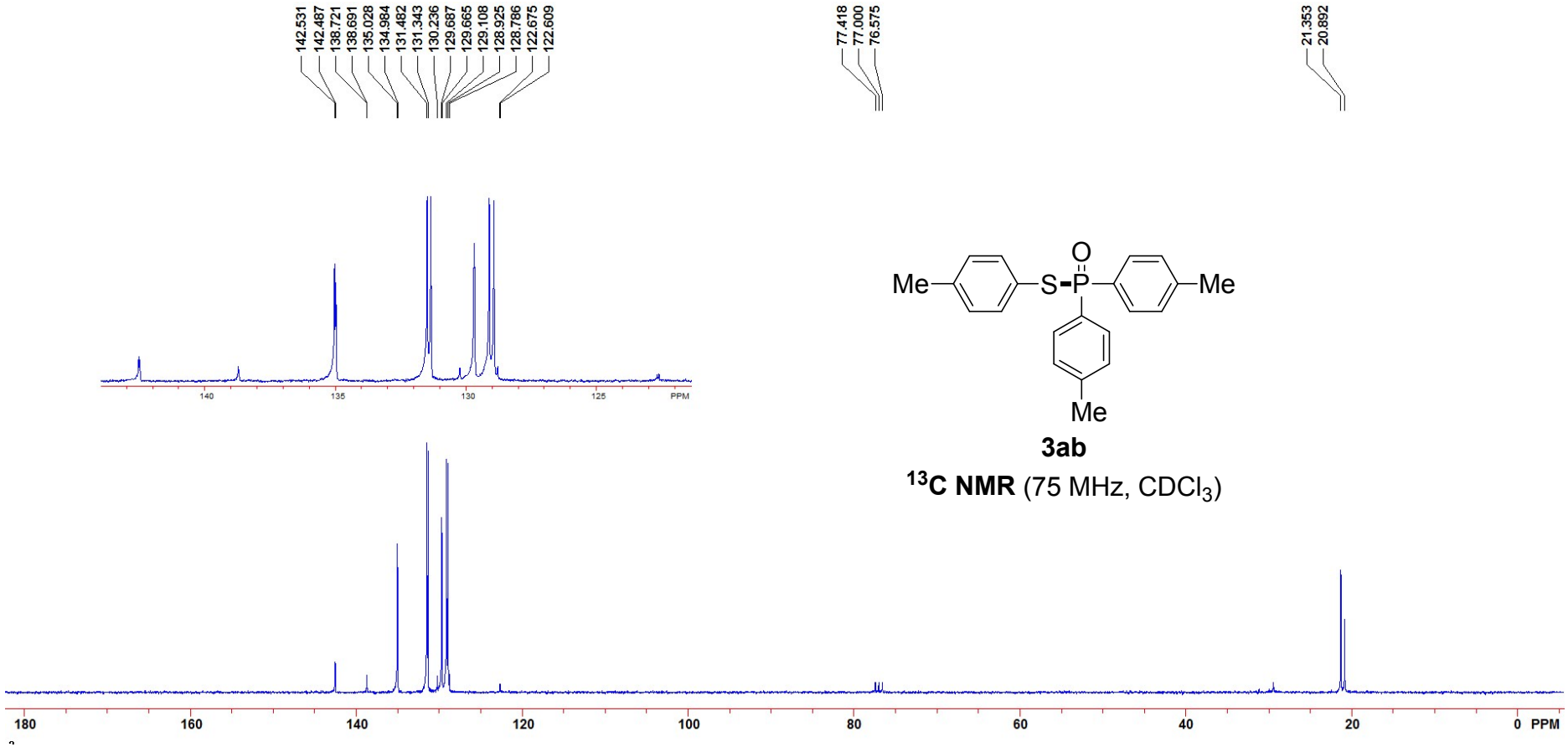

136.774
136.735
133.491
132.644
132.231
132.208
131.477
131.399
128.925
128.622
128.513
128.474
127.338
77.249
77.000
76.743
33.103
33.087
O
S
P
Ph
Ph
3qa
13C NMR (125 MHz, CDCl3)
160
140
120
100
80
60
40
20
0
PPM



3qa

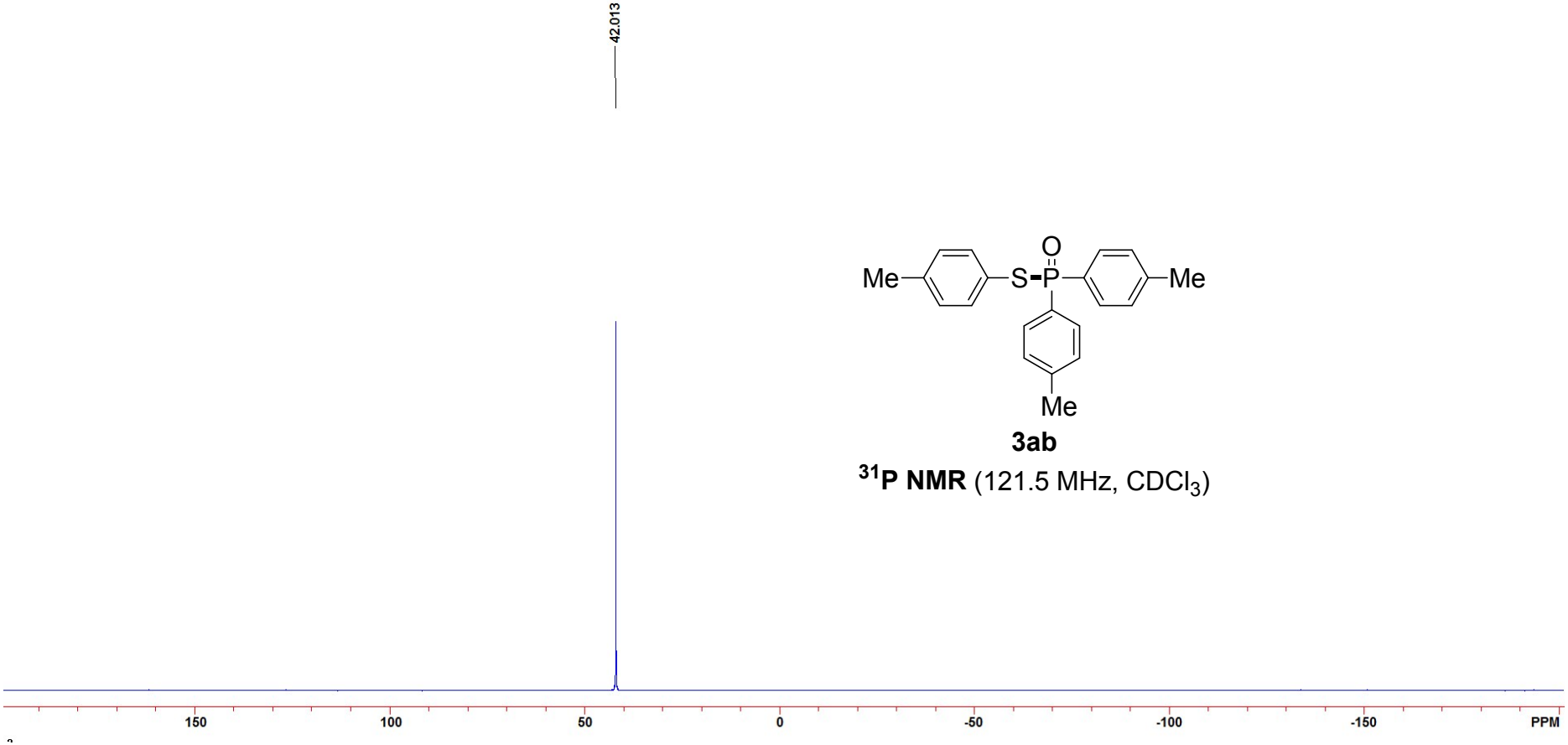
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.751
7.725
7.709
7.682
7.338
7.315
7.311
7.262
7.232
7.222
7.206
7.195
6.999
6.972
2.347
2.227
0.000
Me
S
P
O
Me
Me
3ab
1H NMR (300 MHz, CDCl3)
4.01
4.00
2.03
2.00
6.02
2.98
10
8
6
4
2
0
PPM



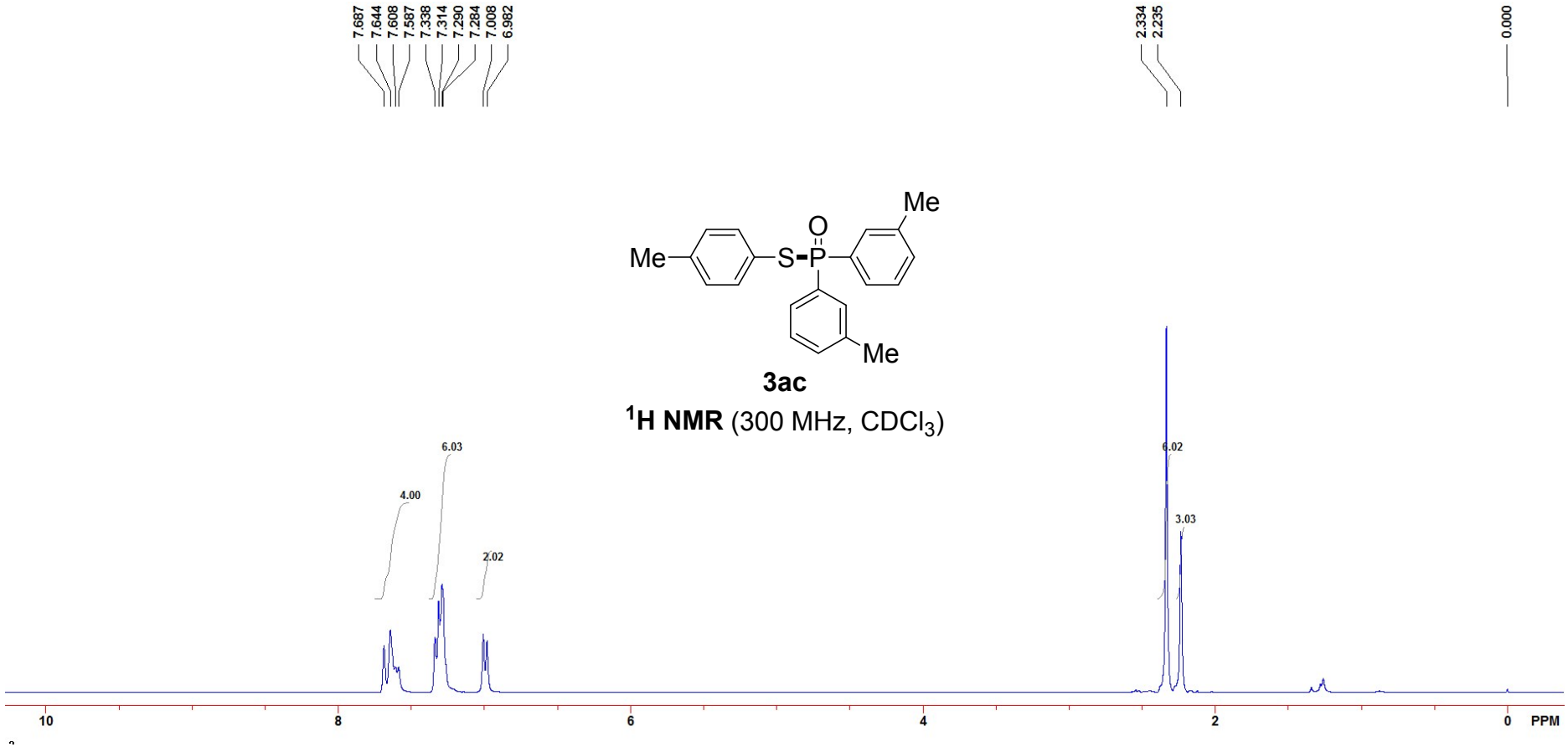
3ab

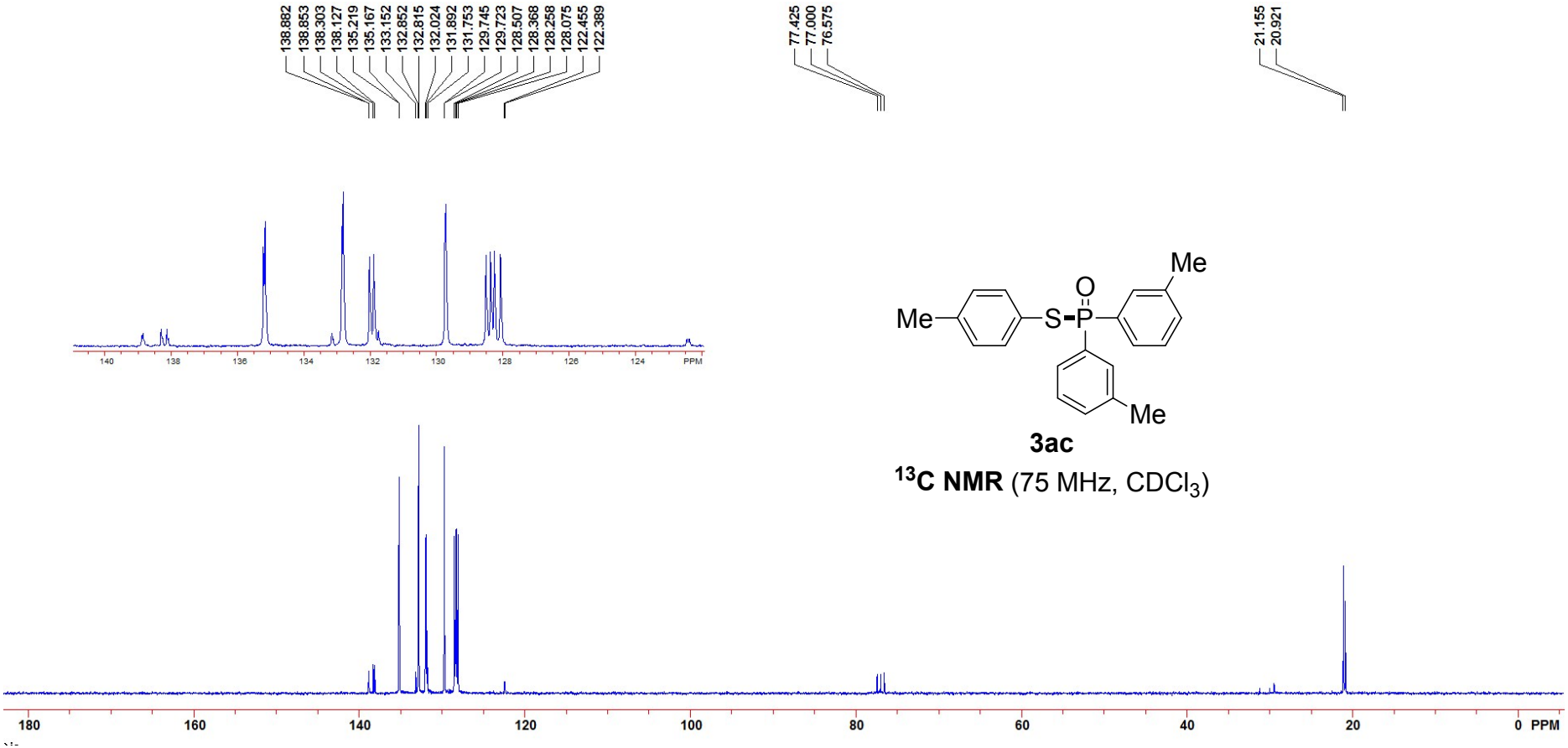
^{13}C NMR (75 MHz, $CDCl_3$)



3ab

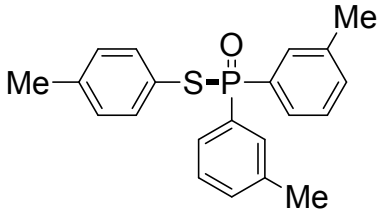
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.687
7.644
7.608
7.587
7.338
7.314
7.290
7.284
7.008
6.982
2.334
2.235
0.000
Me
O
Me
S
P
Me
3ac
1H NMR (300 MHz, CDCl3)
6.03
4.00
2.02
6.02
3.03
10
8
6
4
2
0
PPM



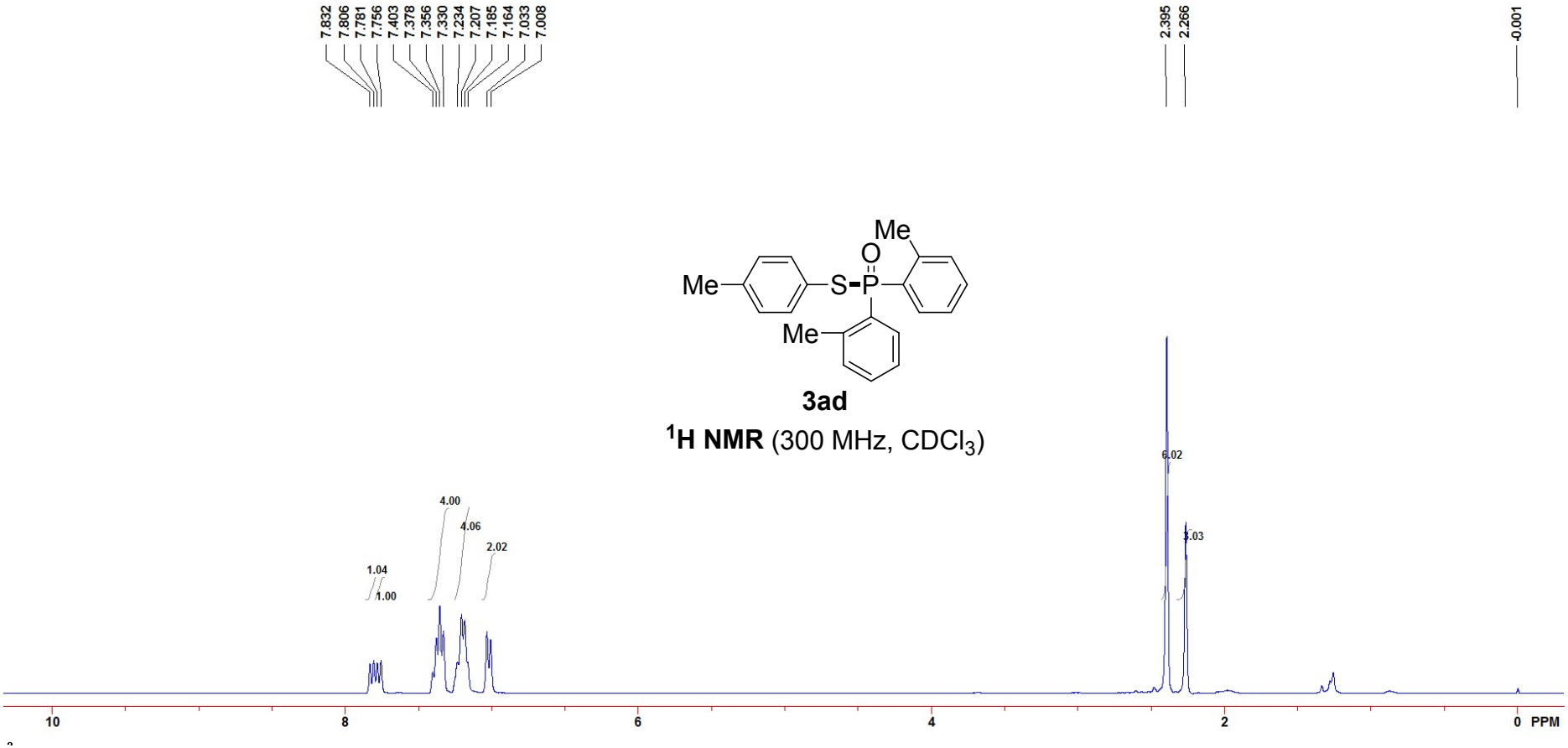
3ac

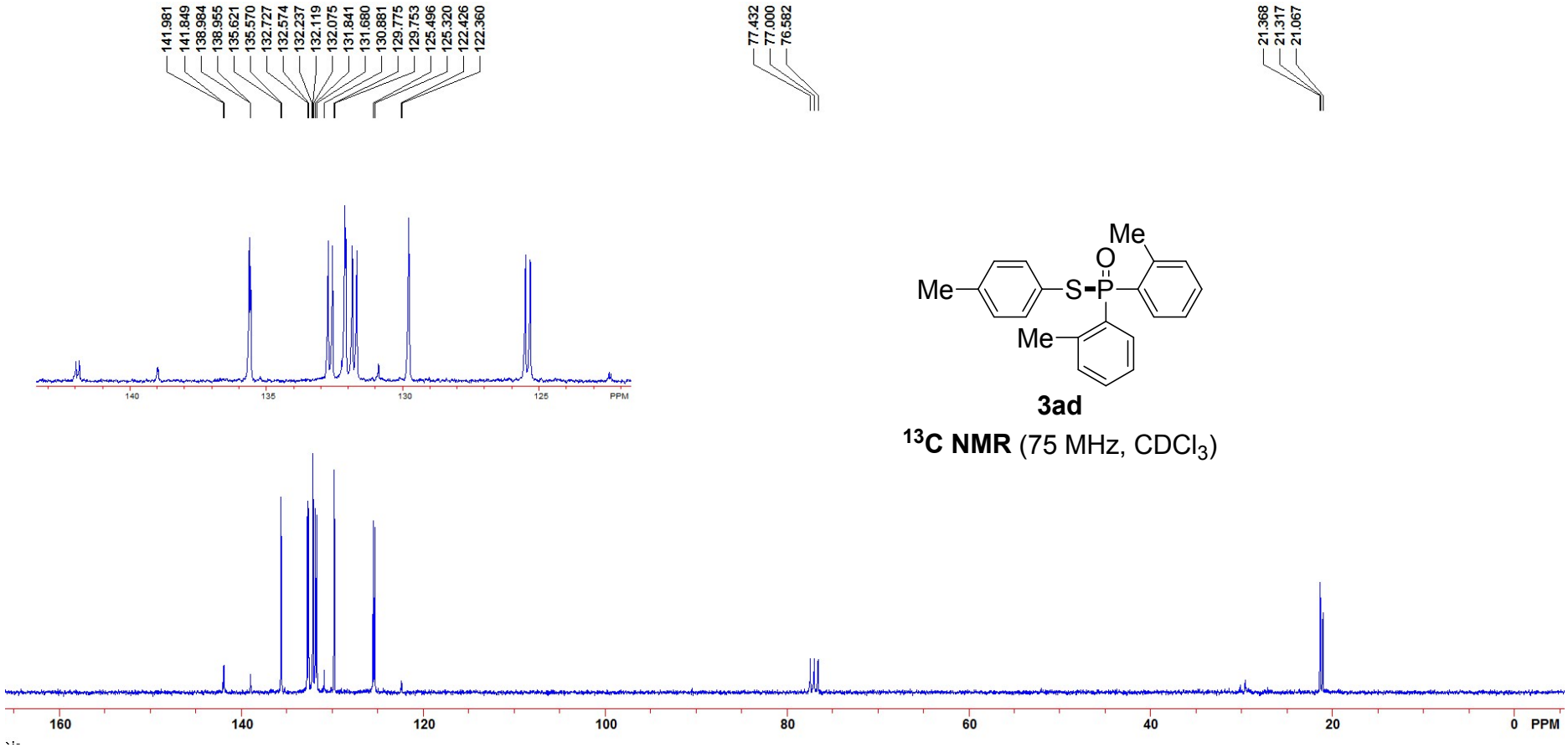
^{13}C NMR (75 MHz, $CDCl_3$)



3ac

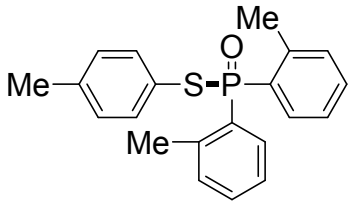
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.832
7.806
7.781
7.756
7.403
7.378
7.356
7.330
7.234
7.207
7.185
7.164
7.033
7.008
2.395
2.266
-0.001
Me
O
Me
S
P
Me
3ad
1H NMR (300 MHz, CDCl3)
4.00
4.06
2.02
1.04
1.00
6.02
3.03
10
8
6
4
2
0 PPM



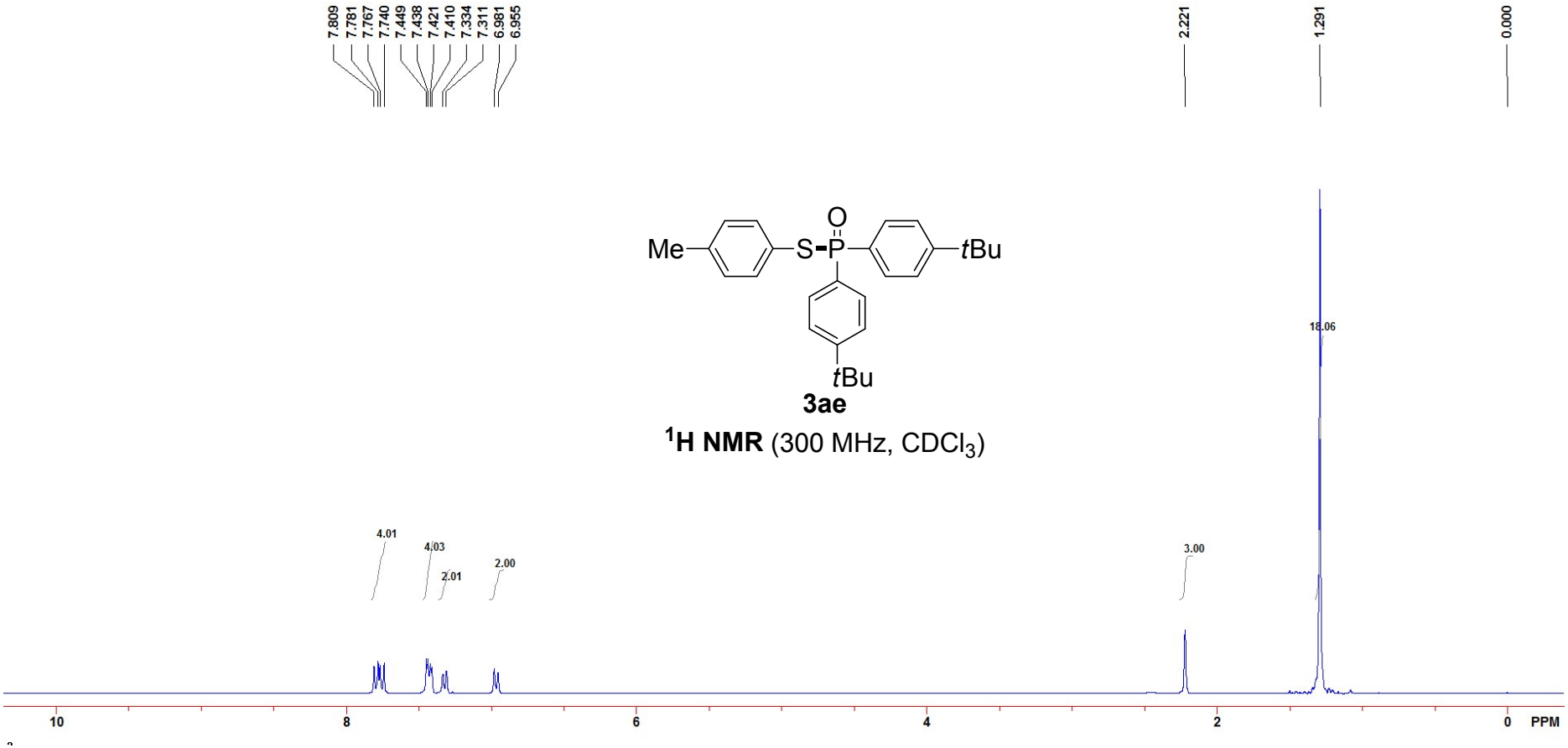
3ad

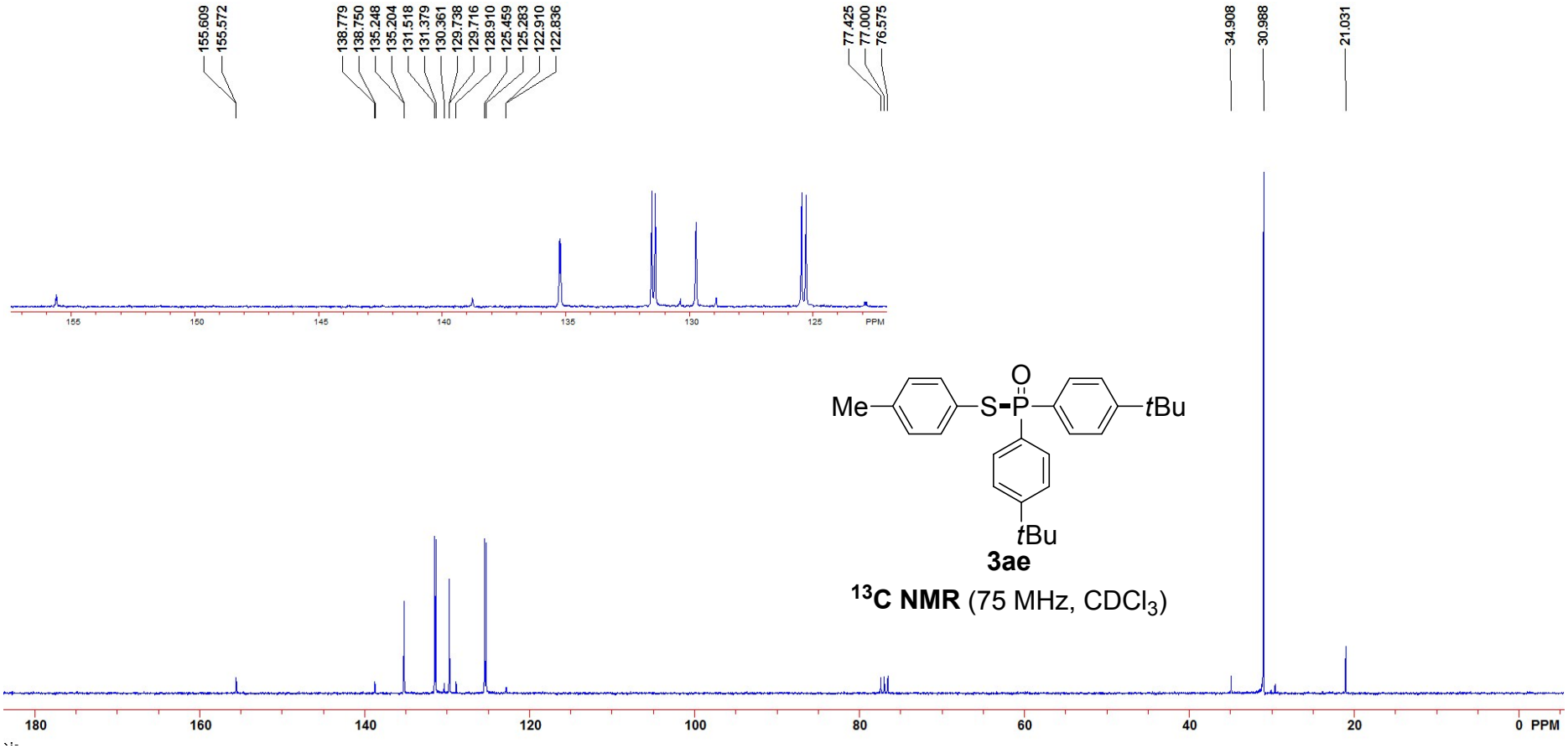
^{13}C NMR (75 MHz, $CDCl_3$)



3ad

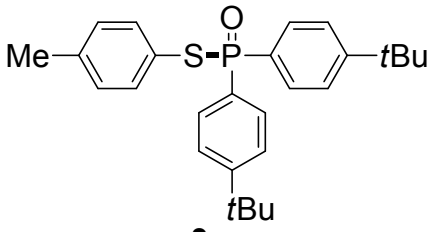
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.809
7.781
7.767
7.740
7.449
7.438
7.421
7.410
7.334
7.311
6.981
6.955
2.221
1.291
0.000
Me
S
P
O
tBu
tBu
3ae
1H NMR (300 MHz, CDCl3)
4.01
4.03
2.01
2.00
3.00
18.06
10
8
6
4
2
0
PPM



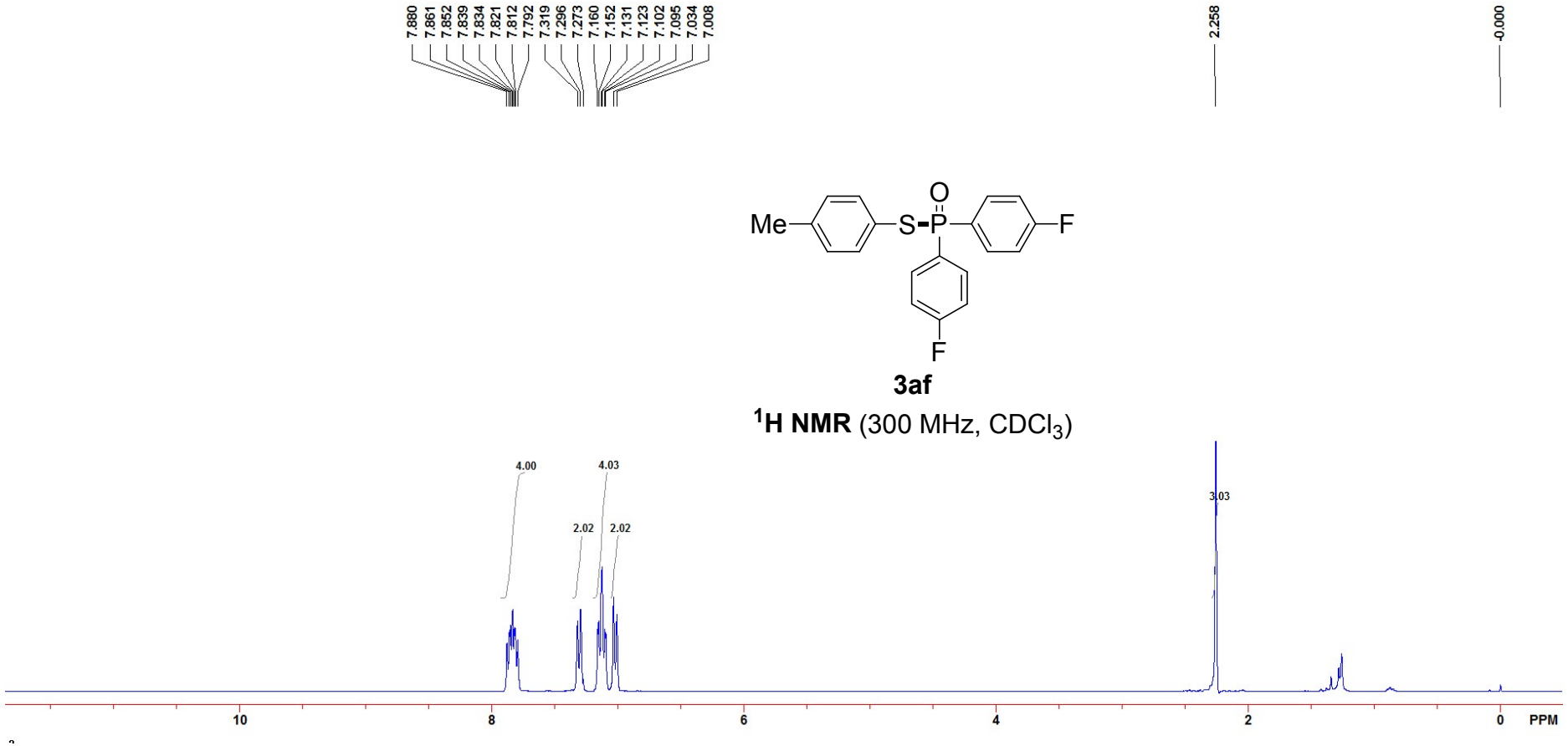
3ae

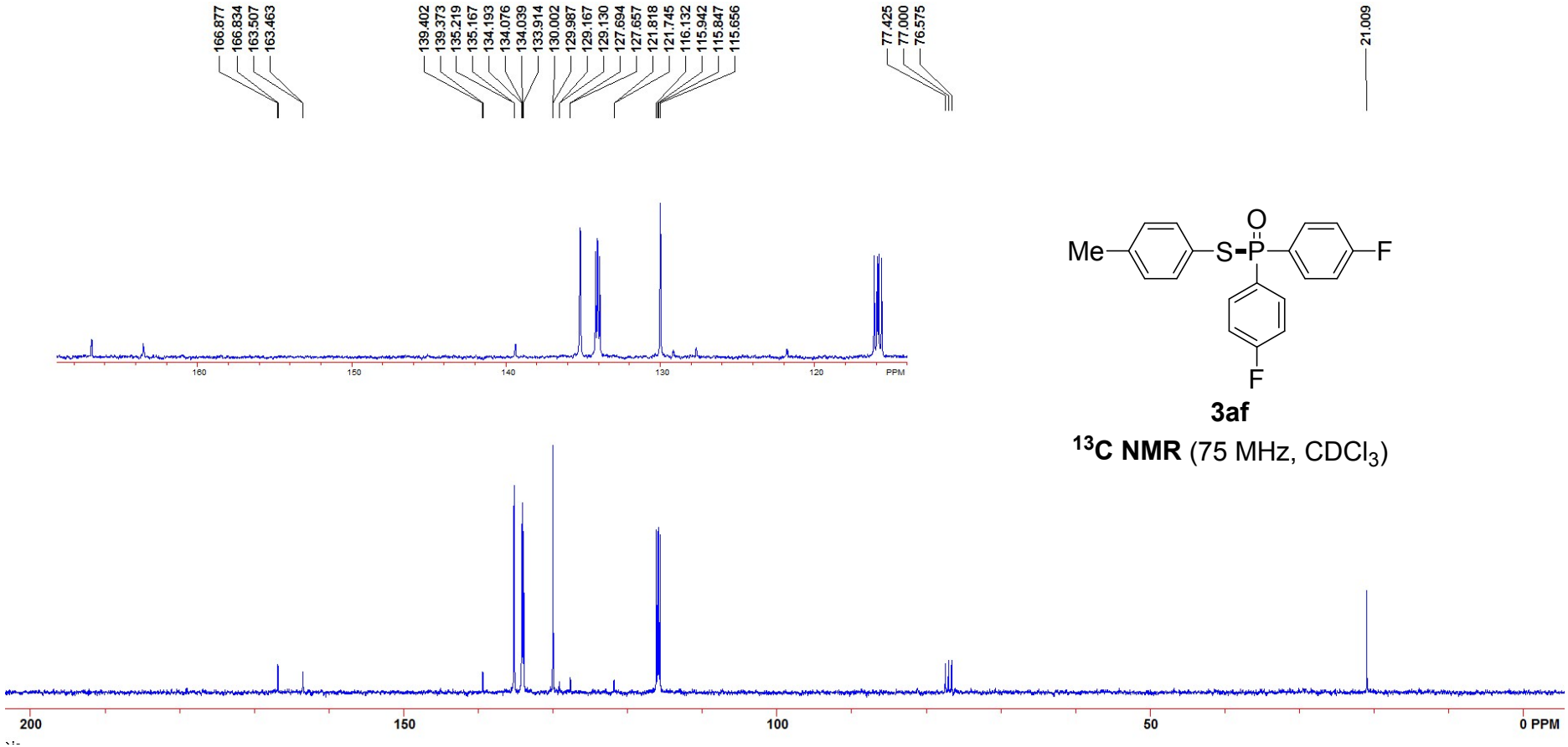
^{13}C NMR (75 MHz, $CDCl_3$)



3ae

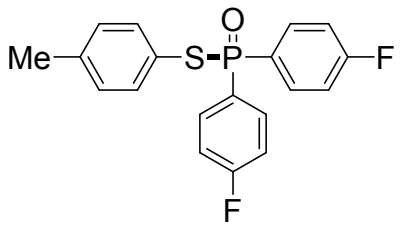
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.880
7.861
7.852
7.839
7.834
7.821
7.812
7.792
7.319
7.296
7.273
7.160
7.152
7.131
7.123
7.102
7.095
7.034
7.008
2.258
0.000
Me
S
P
O
F
F
3af
1H NMR (300 MHz, CDCl3)
4.00
4.03
2.02
2.02
3.03
10
8
6
4
2
0
PPM



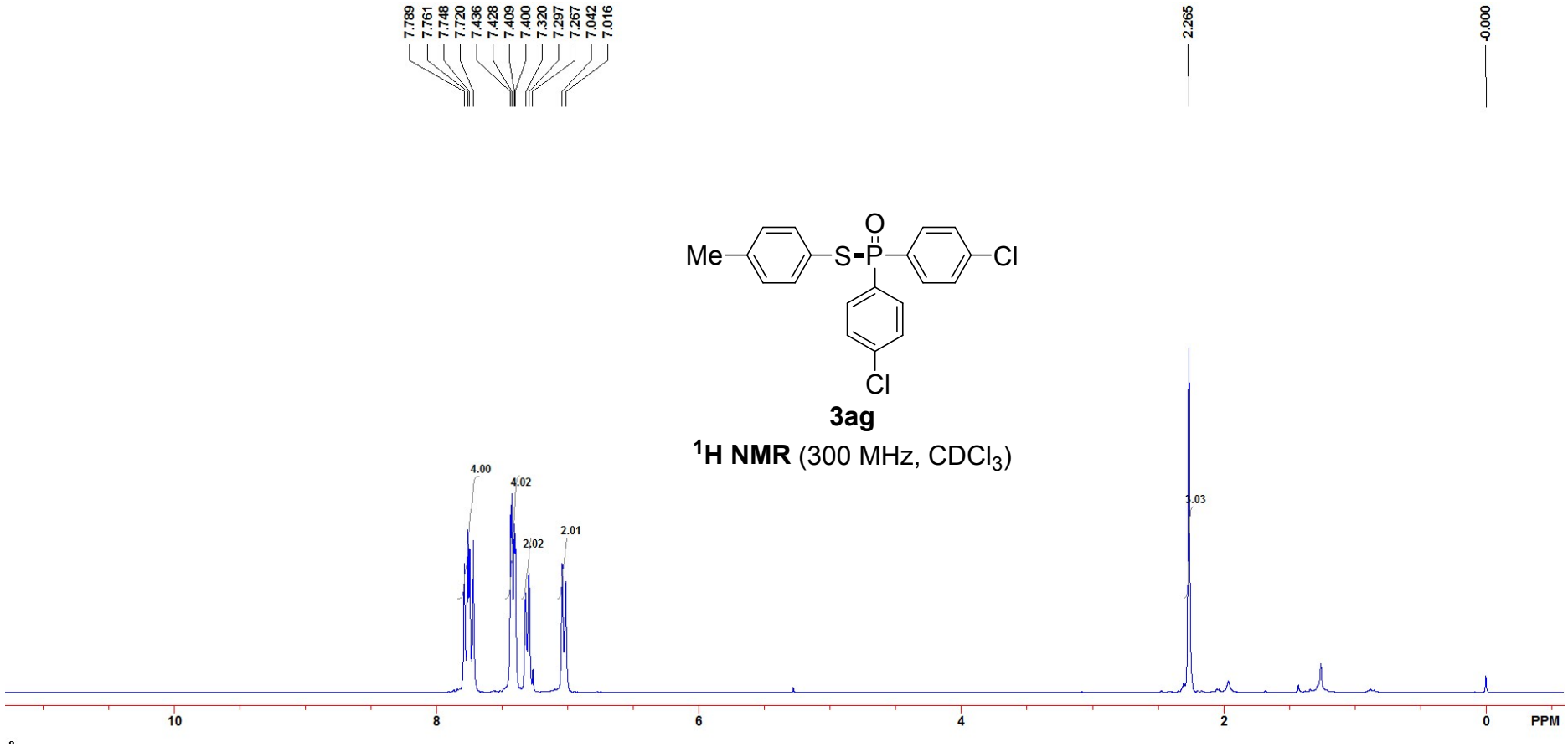
3af

^{13}C NMR (75 MHz, $CDCl_3$)



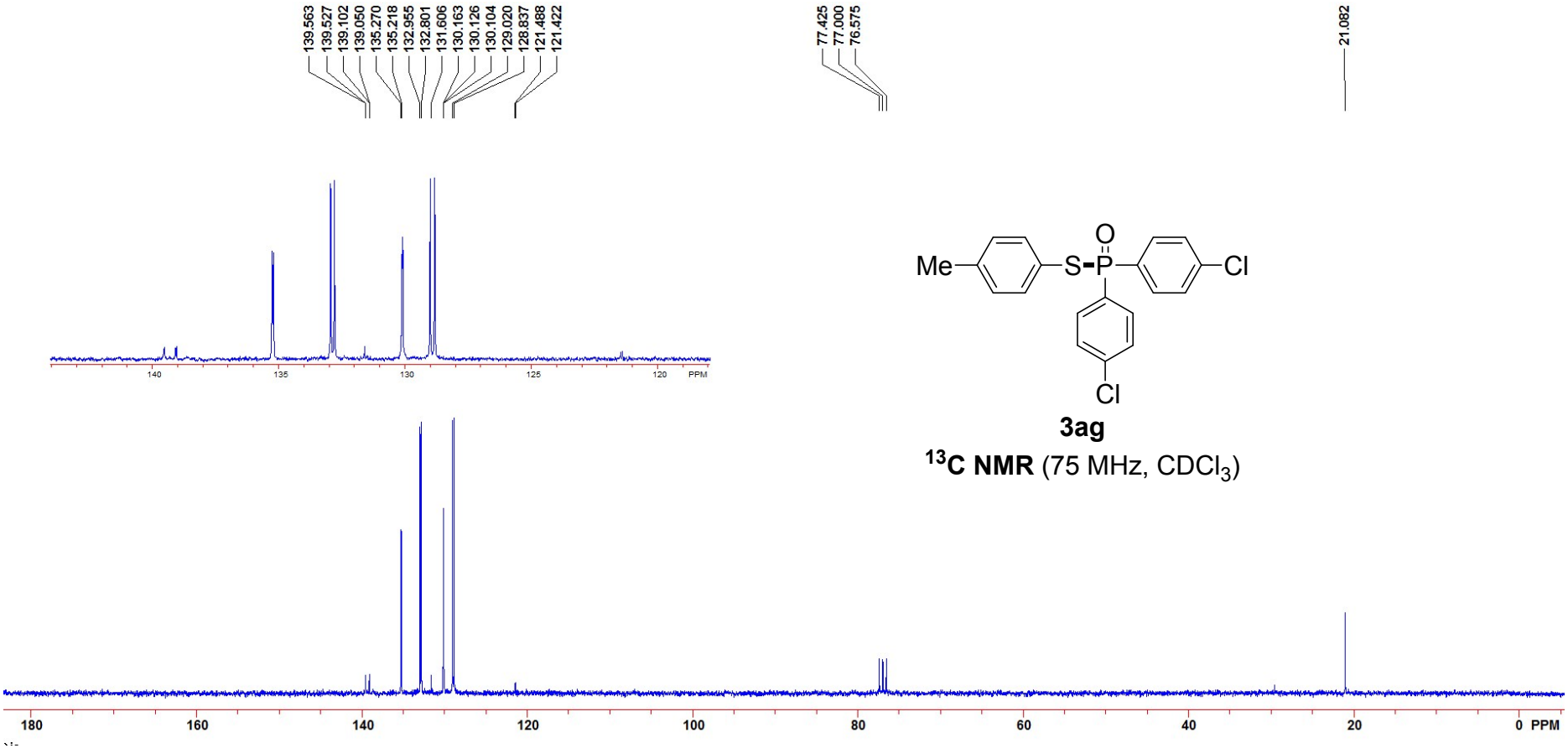
3af

^{31}P NMR (121.5 MHz, $CDCl_3$)



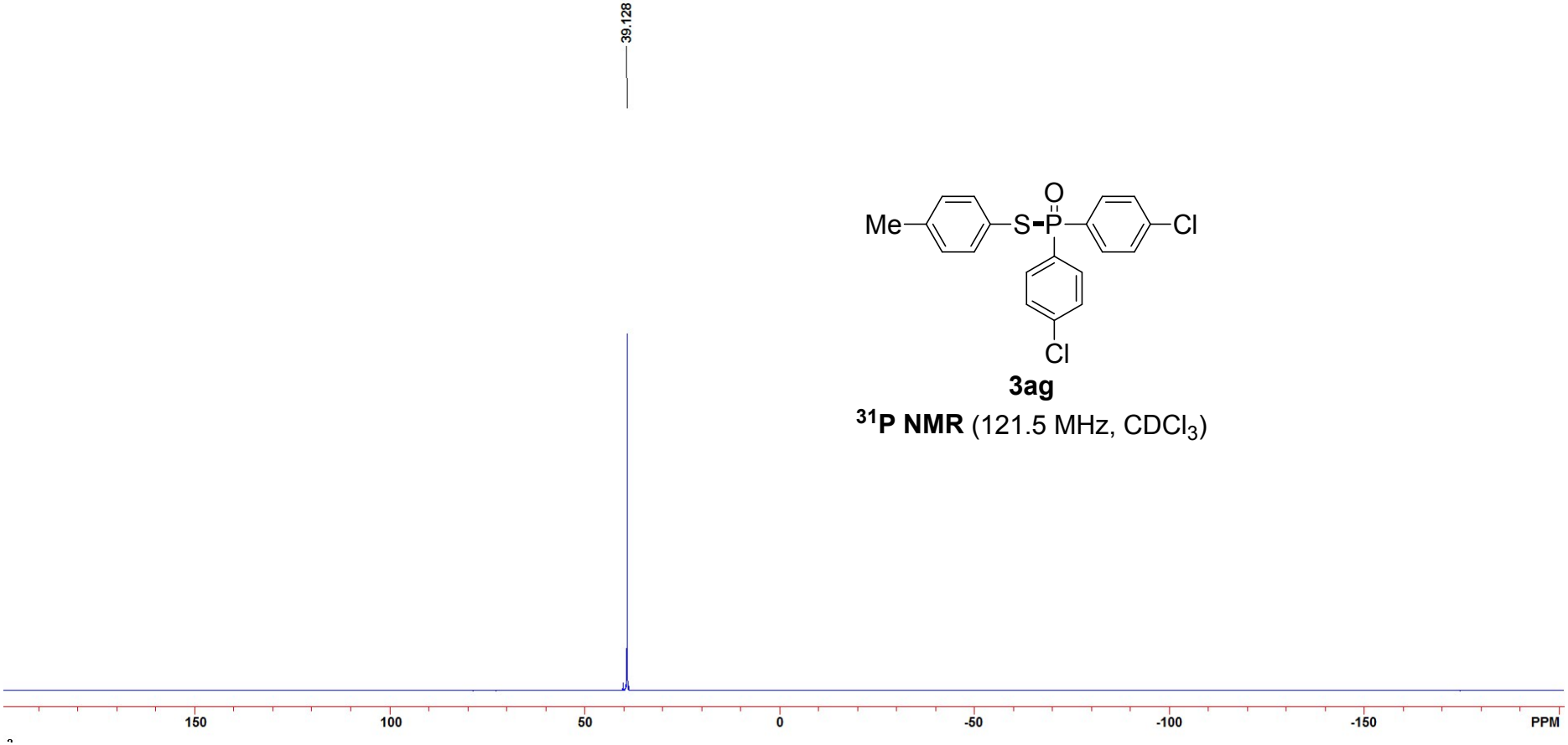
3ag

^{1}H NMR (300 MHz, $CDCl_3$)



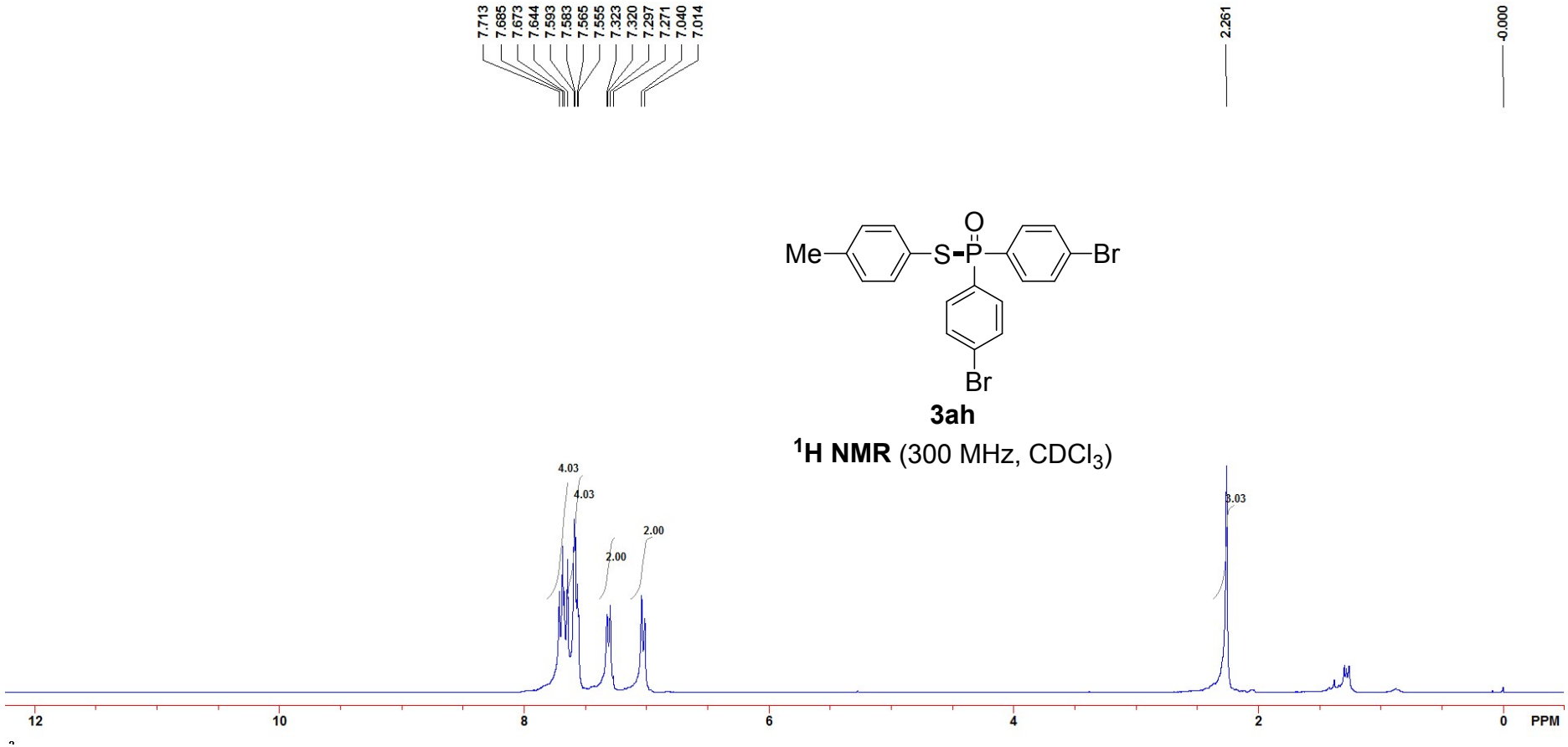
3ag

^{13}C NMR (75 MHz, $CDCl_3$)



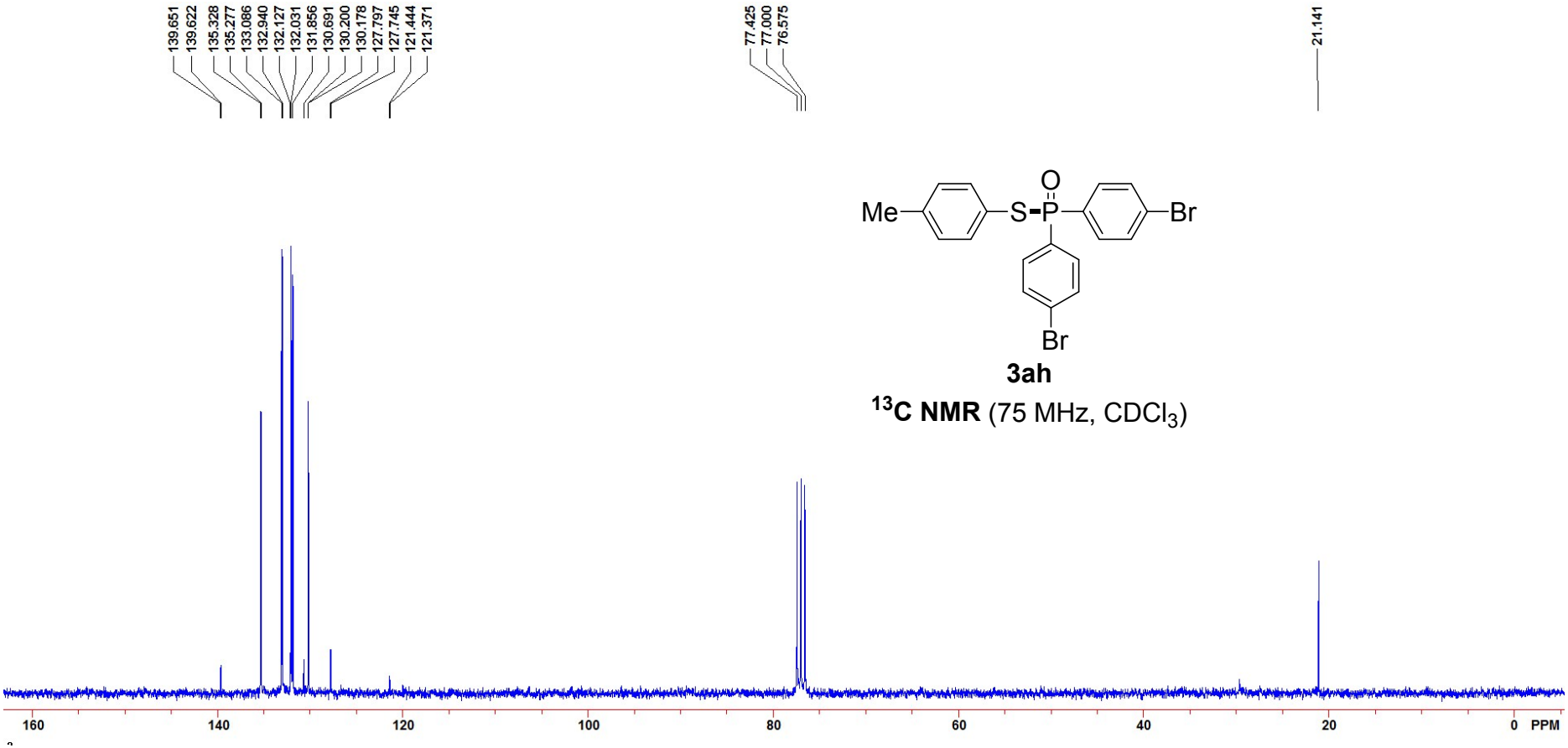
3ag

^{31}P NMR (121.5 MHz, $CDCl_3$)



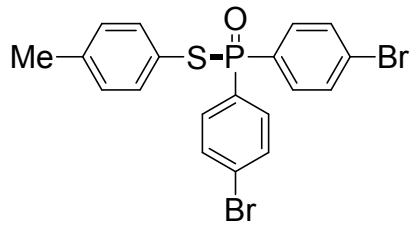
3ah

^{1}H NMR (300 MHz, $CDCl_3$)



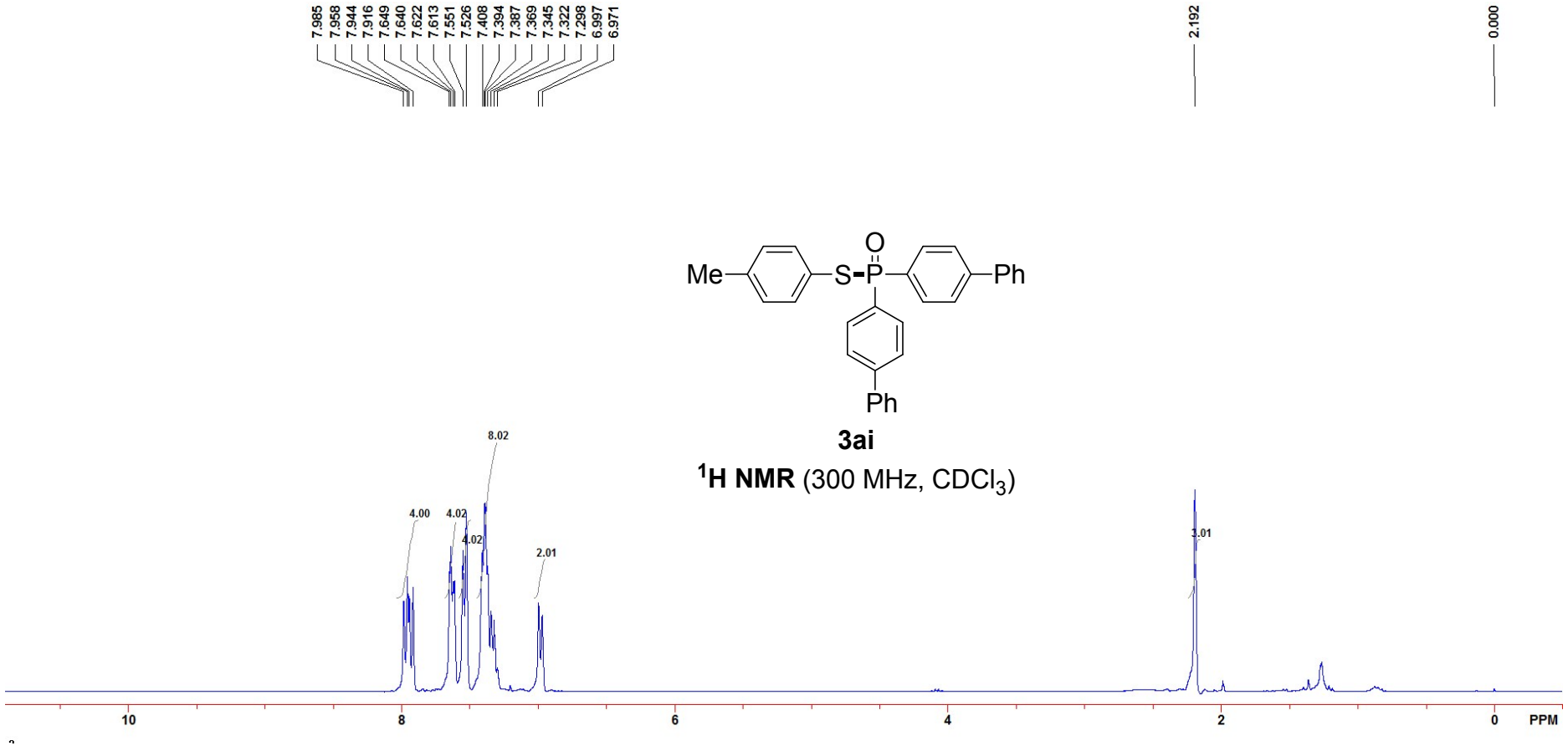
3ah

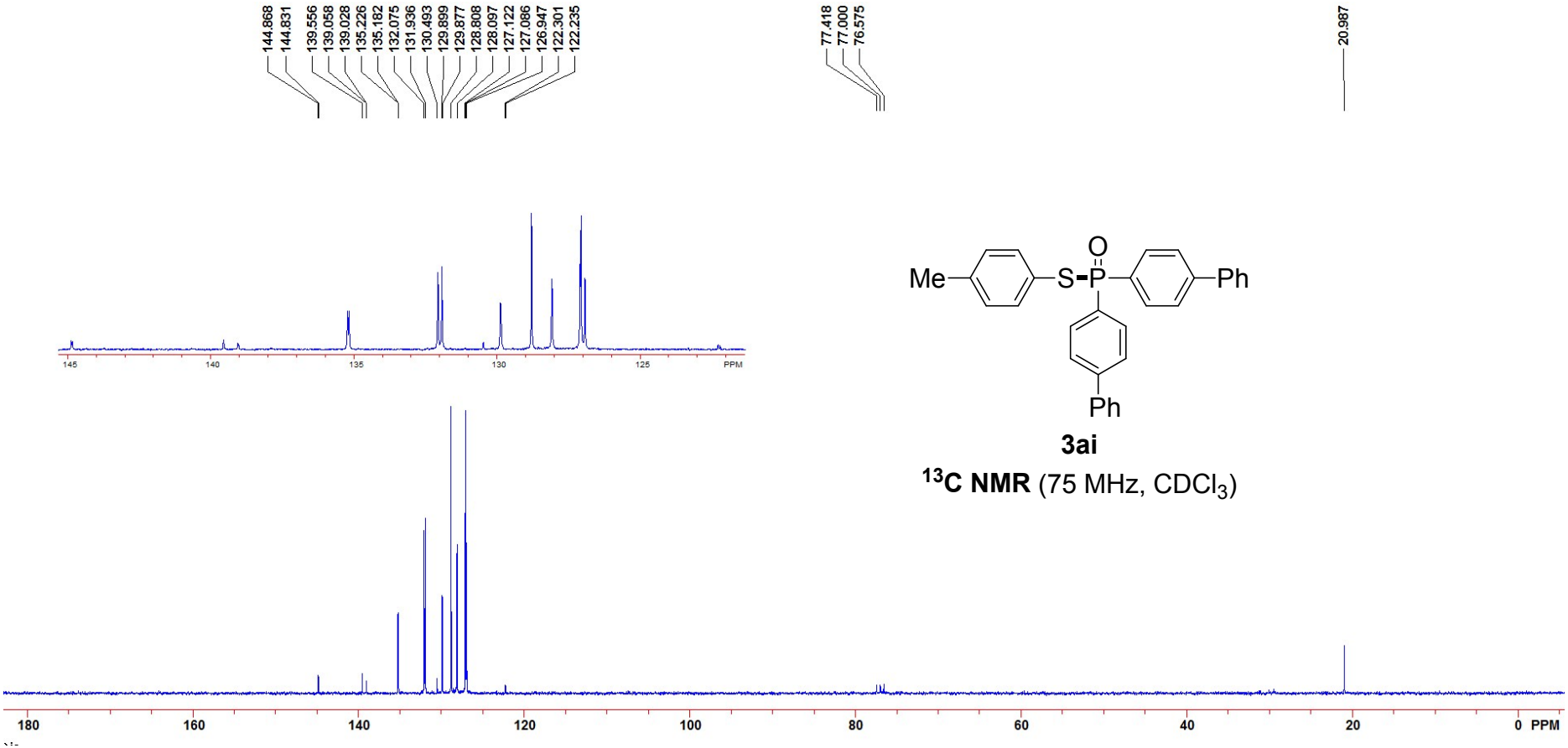
^{13}C NMR (75 MHz, $CDCl_3$)



3ah

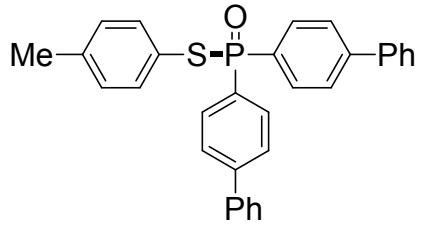
^{31}P NMR (121.5 MHz, $CDCl_3$)


7.985
7.958
7.944
7.916
7.649
7.640
7.622
7.613
7.551
7.526
7.408
7.394
7.387
7.369
7.345
7.322
7.298
6.997
6.971
2.192
0.000
Me
S
P
O
Ph
Ph
3ai
1H NMR (300 MHz, CDCl3)
8.02
4.00
4.02
4.02
2.01
3.01
10
8
6
4
2
0
PPM



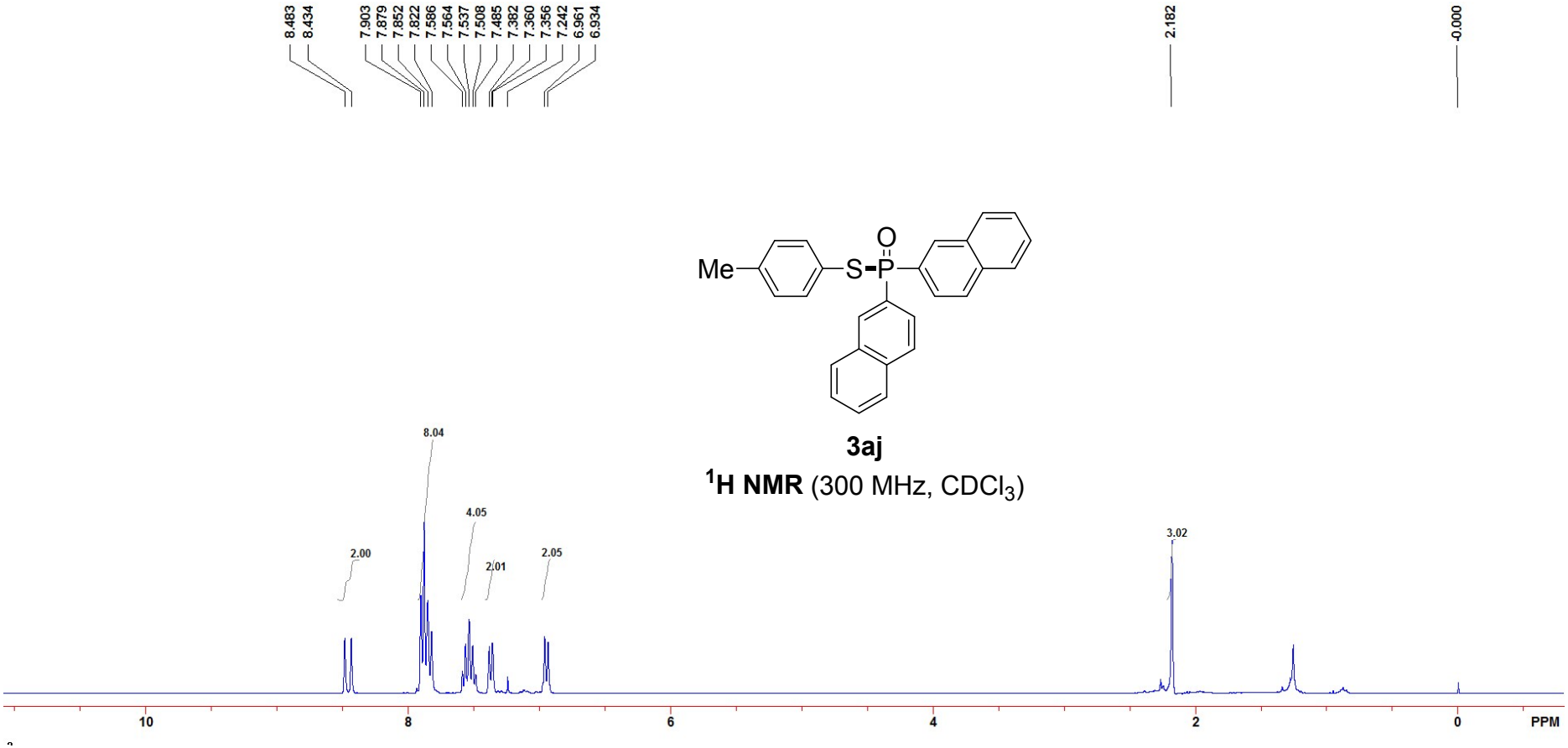
3ai

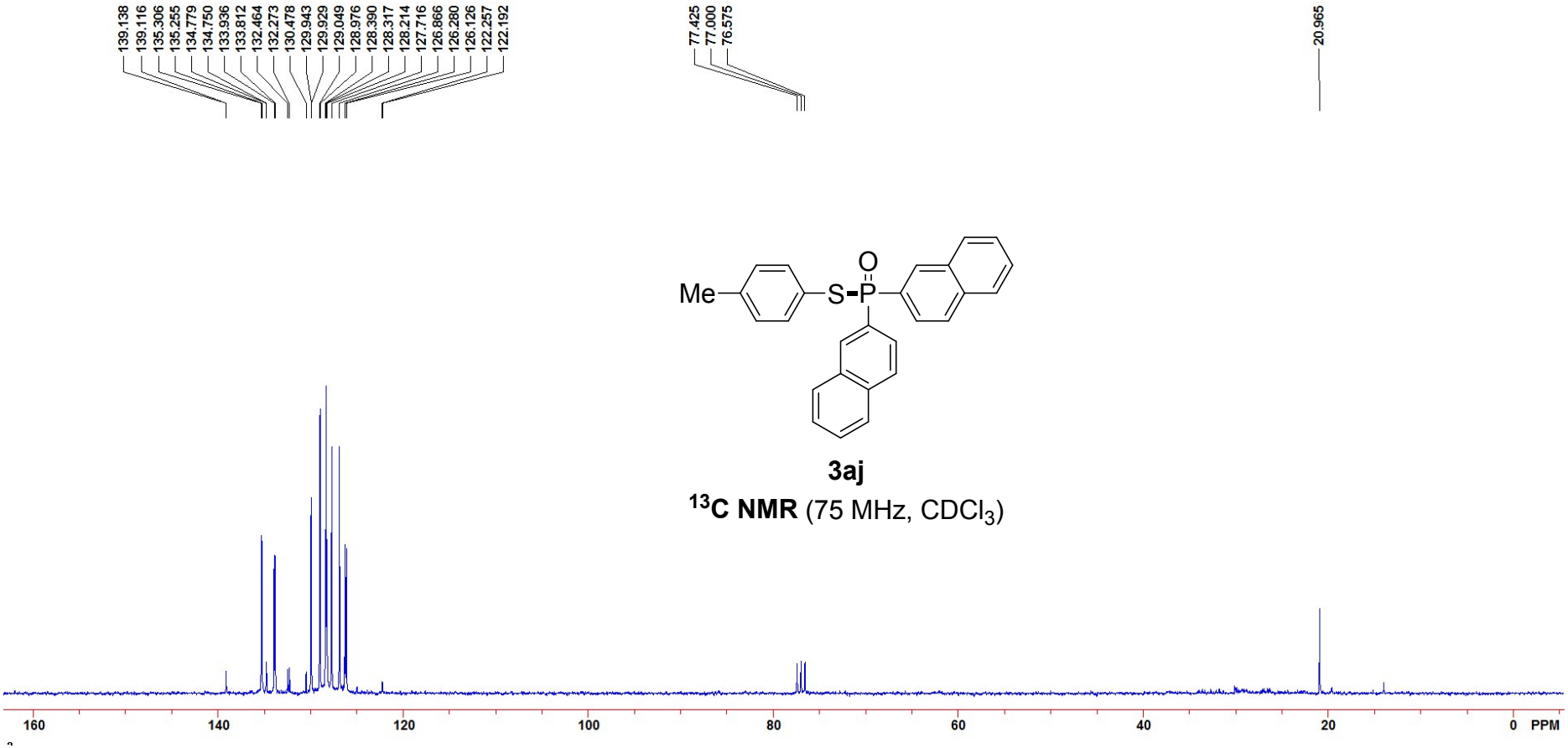
^{13}C NMR (75 MHz, $CDCl_3$)



3ai

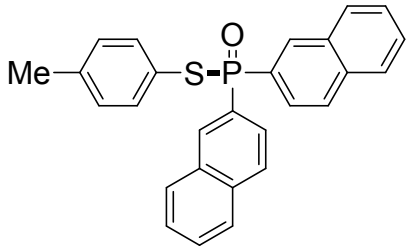
^{31}P NMR (121.5 MHz, $CDCl_3$)


8.483
8.434
7.903
7.879
7.852
7.822
7.586
7.564
7.537
7.508
7.485
7.382
7.360
7.356
7.242
6.961
6.934
2.182
0.000
Me
S
P
O
3aj
1H NMR (300 MHz, CDCl3)
8.04
4.05
2.00
2.01
2.05
3.02
10
8
6
4
2
0
PPM



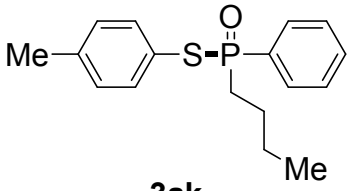
3aj

^{13}C NMR (75 MHz, $CDCl_3$)



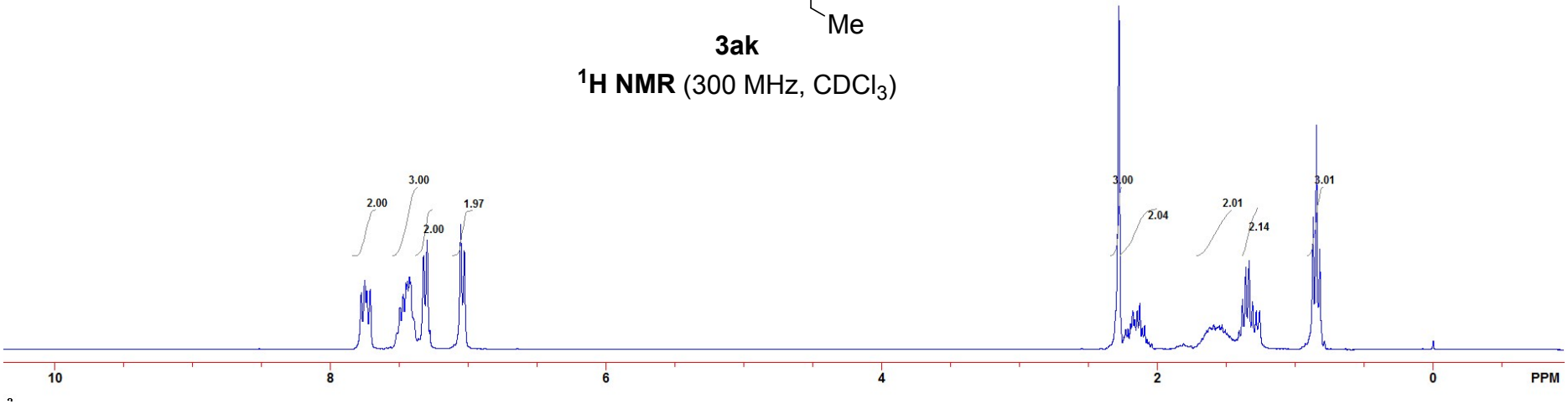
3aj

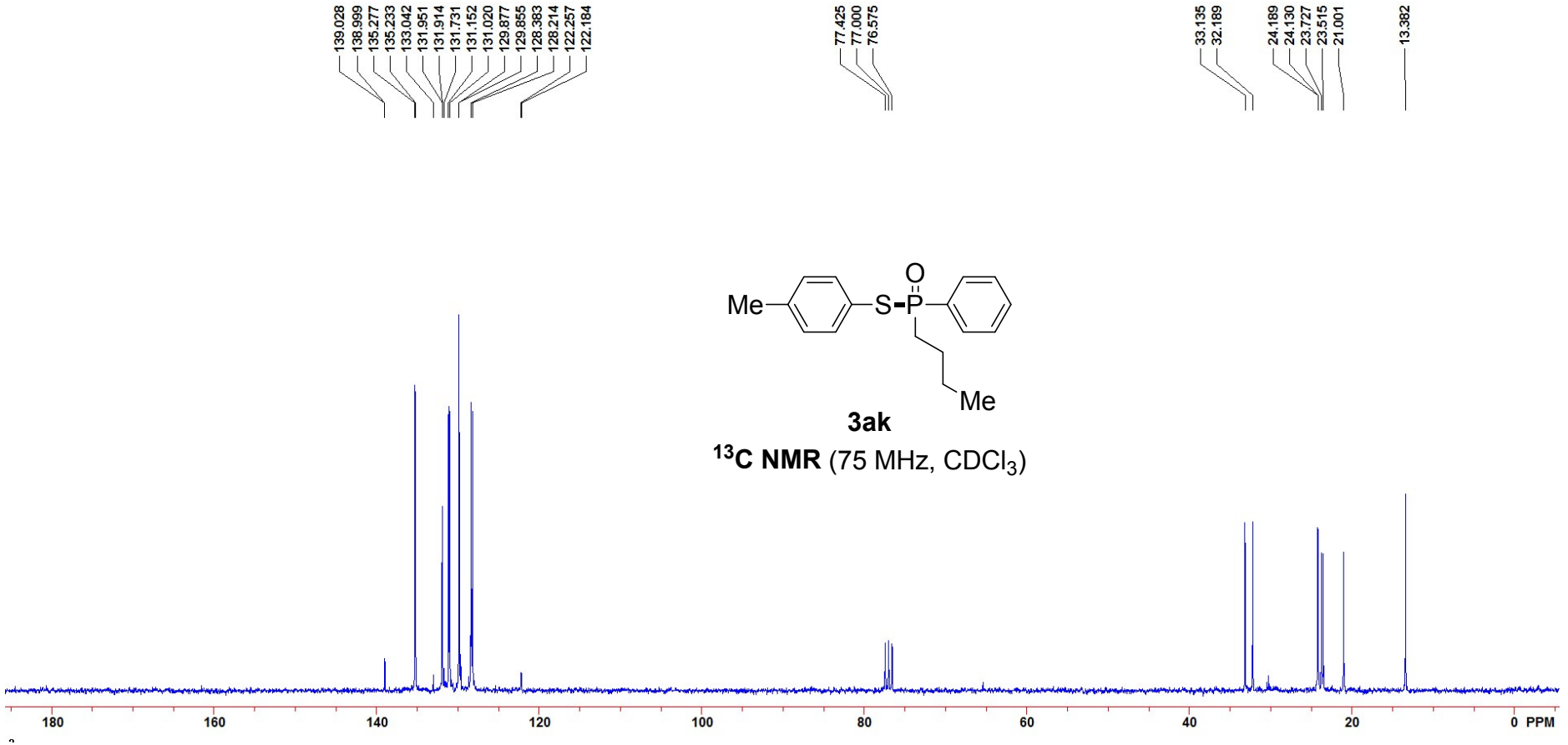
^{31}P NMR (121.5 MHz, $CDCl_3$)



3ak

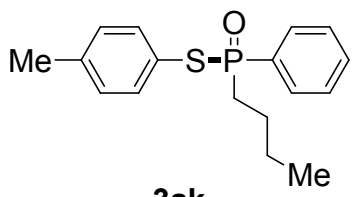
^{1}H NMR (300 MHz, $CDCl_3$)





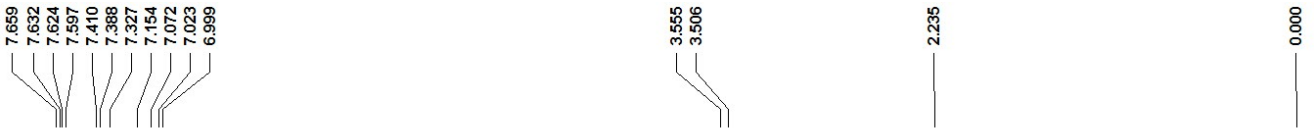
3ak

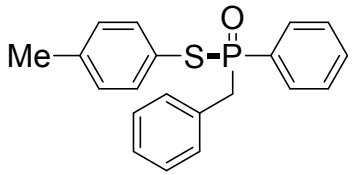
^{13}C NMR (75 MHz, $CDCl_3$)



3ak

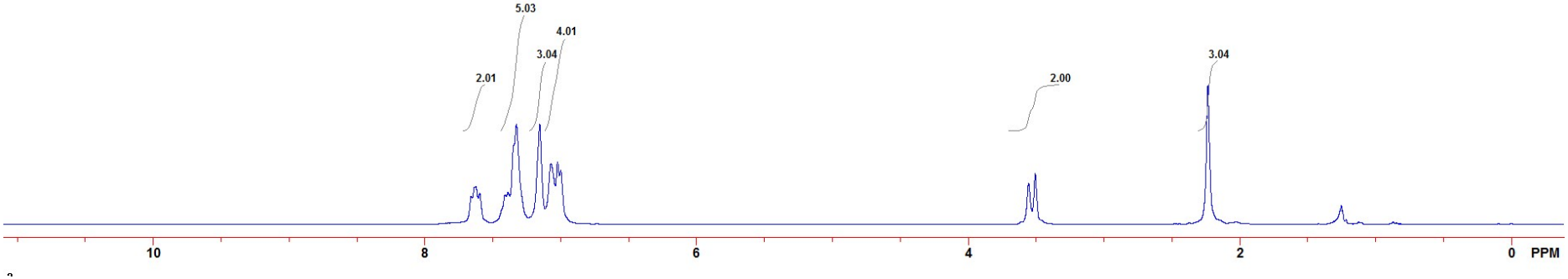
^{31}P NMR (121.5 MHz, $CDCl_3$)

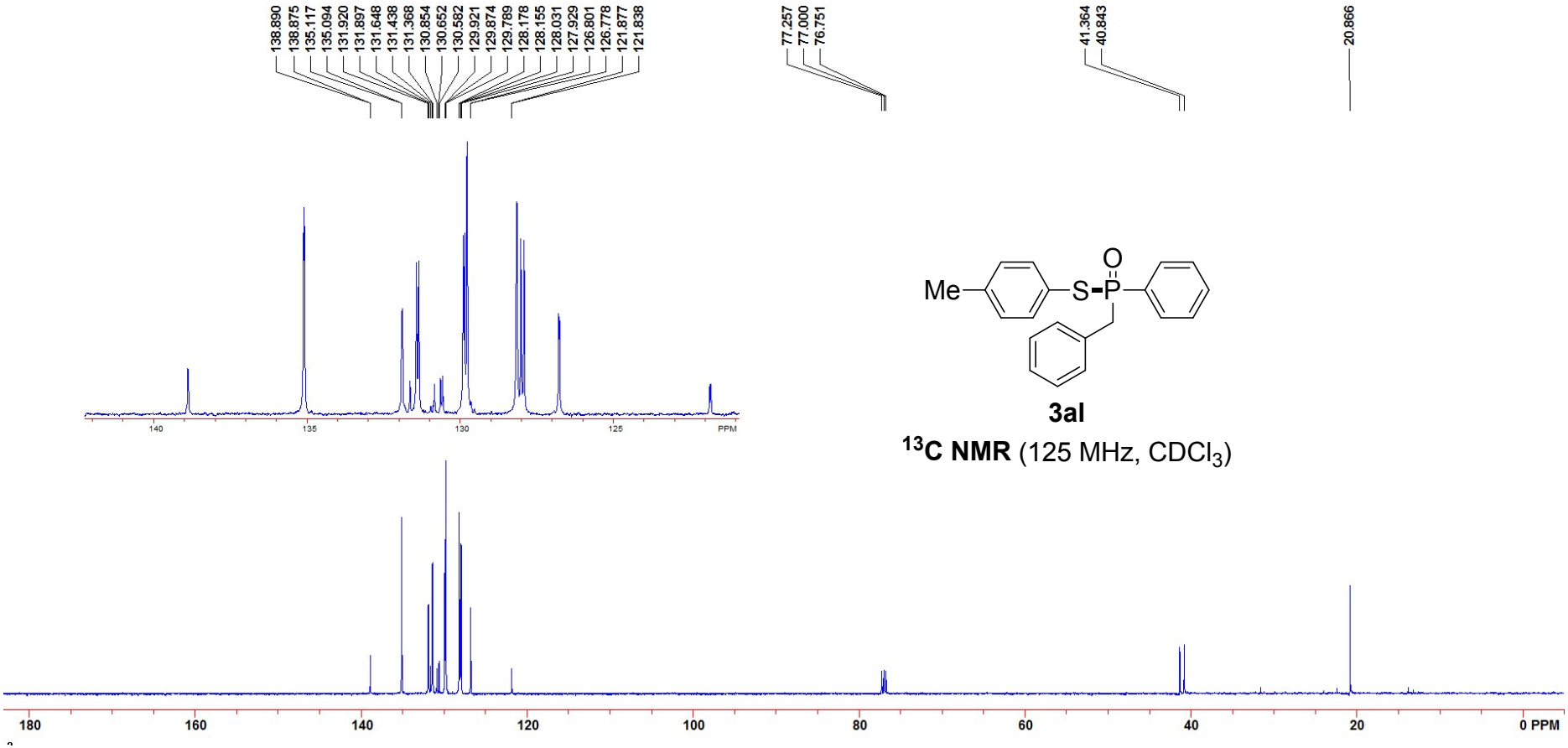




3al

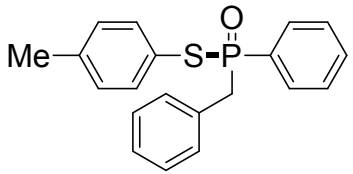
^{1}H NMR (300 MHz, $CDCl_3$)





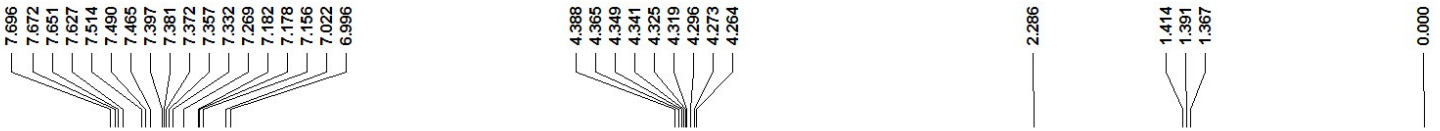
3al

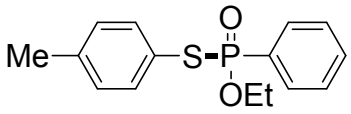
^{13}C NMR (125 MHz, $CDCl_3$)



3al

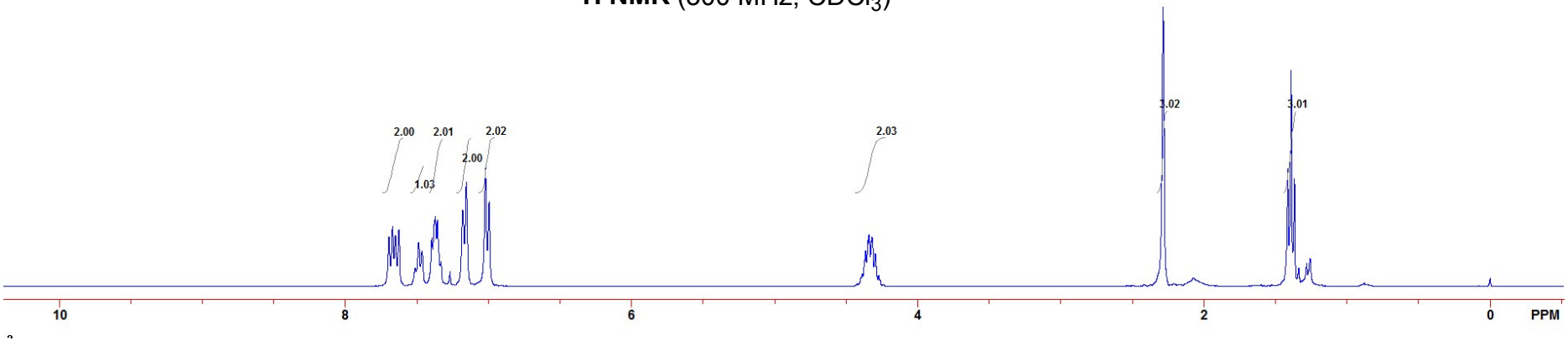
^{31}P NMR (202.5 MHz, $CDCl_3$)

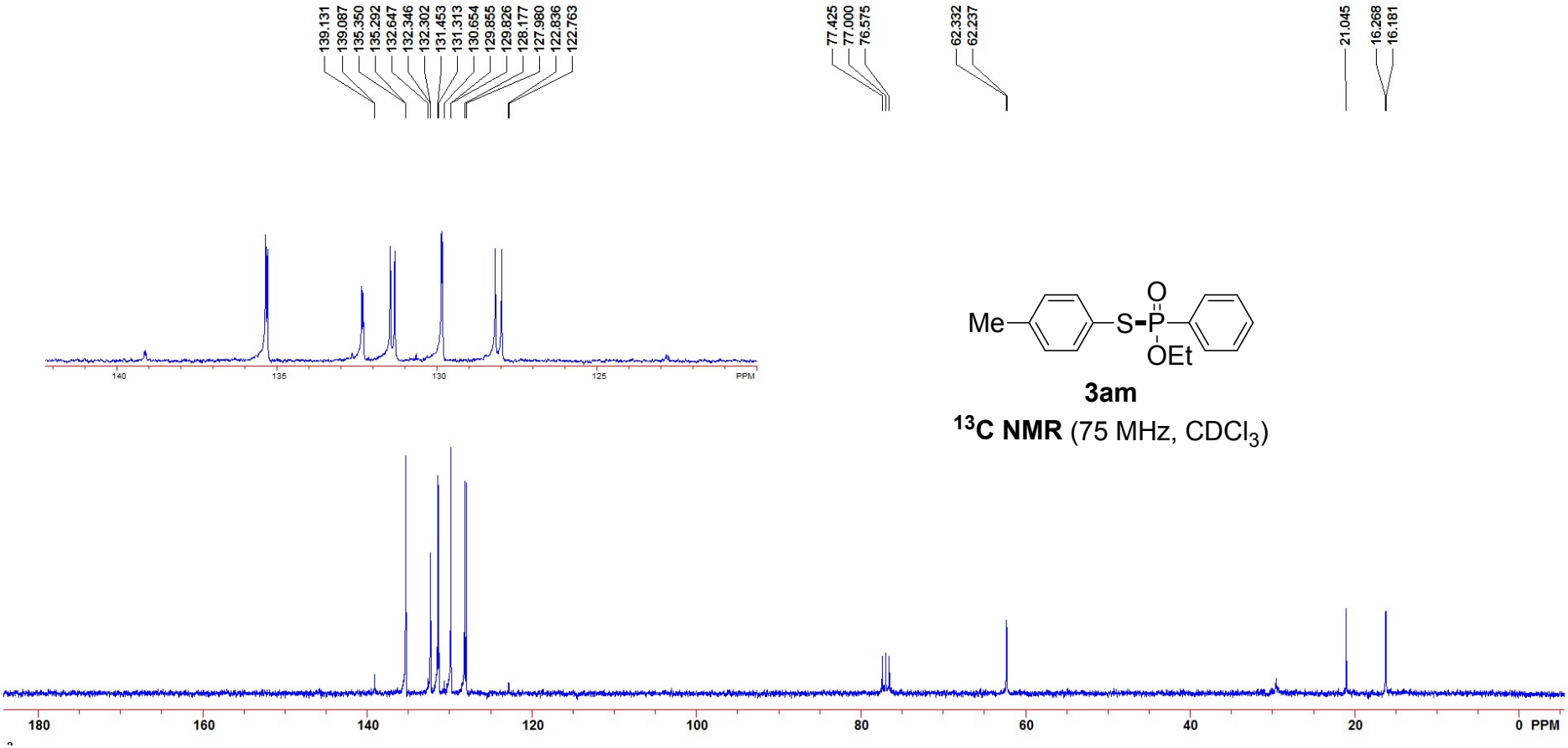




3am

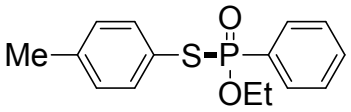
^{1}H NMR (300 MHz, $CDCl_3$)





3am

^{13}C NMR (75 MHz, $CDCl_3$)



3am

^{31}P NMR (121.5 MHz, $CDCl_3$)