Supporting information

Mild and Efficient Synthesis of Indoles and Isoquinolones *via* a Nickel-Catalyzed Larock-Type Heteroannulation Reaction

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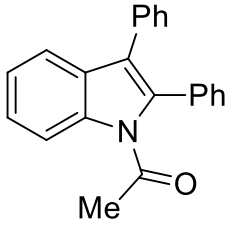
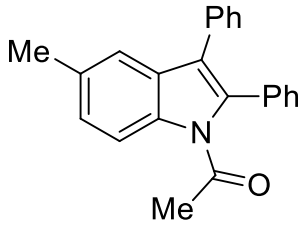
Physical data of the compounds

1-(2,3-Diphenyl-1*H*-indol-1-yl)ethanone (3)[1]



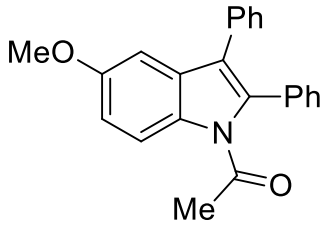
According to **GP1** with *N*-(2-iodophenyl)acetamide (53.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **3** as white solid (56.7 mg, 91%). Mp: 130-132 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.4 Hz, 1H), 7.56 (dd, *J* = 7.8, 0.6 Hz, 1H), 7.43-7.20 (m, 12H), 1.99 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.6, 136.7, 134.9, 133.0, 132.9, 130.7, 130.0, 129.2, 128.6, 128.6, 128.2, 126.9, 125.5, 123.4, 123.3, 119.5, 116.2, 27.9; **HRMS** (ESI) calculated for $C_{22}H_{18}NO$ $[M+H]^+$ m/z 312.1383, found 312.1382.

1-(5-Methyl-2,3-diphenyl-1*H*-indol-1-yl)ethanone (4)[2]



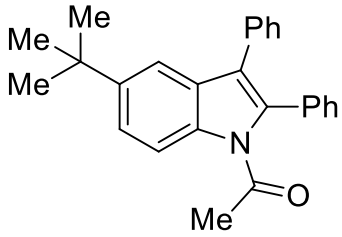
According to **GP1** with *N*-(2-iodo-4-methylphenyl)acetamide (56.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **4** as white solid (52.6 mg, 81%). Mp: 170-172 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.34 (d, *J* = 8.4 Hz, 1H), 7.36-7.19 (m, 12H), 2.43 (s, 3H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.4, 135.1, 135.0, 133.4, 133.1, 133.0, 130.7, 130.0, 129.4, 128.5, 128.2, 126.83, 126.76, 123.2, 119.3, 115.9, 27.8, 21.4; **HRMS** (ESI) calculated for $C_{23}H_{20}NO$ $[M+H]^+$ m/z 326.1539, found 326.1539.

1-(5-Methoxy-2,3-diphenyl-1*H*-indol-1-yl)ethanone (5)[2]



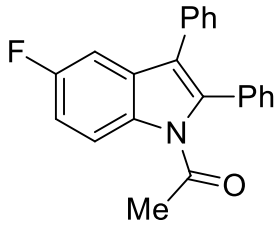
According to **GP1** with *N*-(2-iodo-4-methoxyphenyl)acetamide (58.7 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **5** as white solid (58.5 mg, 86%). Mp: 176-178 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.41-8.38 (m, 1H), 7.35-7.18 (m, 10H), 7.02-6.99 (m, 2H), 3.79 (s, 3H), 1.96 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.1, 156.6, 135.6, 133.0, 132.9, 131.5, 130.7, 130.1, 129.9, 128.6, 128.5, 128.2, 126.8, 123.2, 117.3, 113.8, 102.0, 55.6, 27.7; **HRMS** (ESI) calculated for $C_{23}H_{20}NO_2$ $[M+H]^+$ m/z 342.1489, found 342.1487.

1-(5-(*tert*-Butyl)-2,3-diphenyl-1*H*-indol-1-yl)ethanone (6)



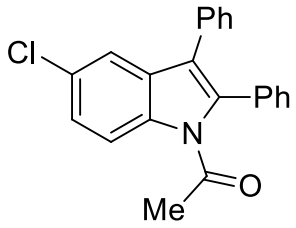
According to **GP1** with *N*-(4-(*tert*-butyl)-2-iodophenyl)acetamide (63.7 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (16.3 mg, 0.03 mmol, 0.15 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **6** as white solid (69.8 mg, 95%). Mp: 147-149 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.7 Hz, 1H), 7.54 (d, *J* = 1.8 Hz, 1H), 7.49 (dd, *J* = 8.7, 2.1 Hz, 1H), 7.35-7.21 (m, 10H), 1.98 (s, 3H), 1.36 (s, 9H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.3, 147.0, 135.2, 134.9, 133.2, 133.1, 130.8, 130.0, 129.0, 128.5, 128.2, 126.8, 123.6, 123.4, 115.8, 115.4, 34.7, 31.7, 27.8; **HRMS** (ESI) calculated for $C_{26}H_{26}NO$ $[M+H]^+$ m/z 368.2009, found 368.2007.

1-(5-Fluoro-2,3-diphenyl-1*H*-indol-1-yl)ethanone (7)[1]



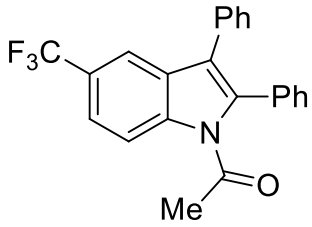
According to **GP1** with *N*-(4-fluoro-2-iodophenyl)acetamide (55.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (53.9 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **7** as white solid (56.5 mg, 86%). Mp: 175-177 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.44 (dd, *J* = 9.1, 4.7 Hz, 1H), 7.39-7.31 (m, 5H), 7.29-7.24 (m, 3H), 7.22-7.16 (m, 3H), 7.11 (td, *J* = 9.1, 2.7 Hz, 1H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.3, 159.9 (d, *J* = 239.0 Hz), 136.4, 133.1, 132.6, 132.5, 130.7, 130.3 (d, *J* = 9.3 Hz), 129.8, 128.9, 128.7, 128.3, 127.1, 123.0 (d, *J* = 3.8 Hz), 117.5 (d, *J* = 8.8 Hz), 113.0 (d, *J* = 24.8 Hz), 105.1 (d, *J* = 24.2 Hz), 27.8; **HRMS** (ESI) calculated for $C_{22}H_{17}FNO$ $[M+H]^+$ m/z 330.1289, found 330.1290.

1-(5-Chloro-2,3-diphenyl-1*H*-indol-1-yl)ethanone (8)[2]



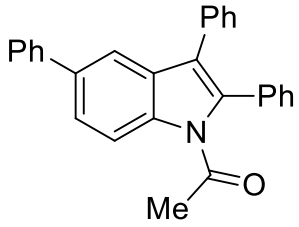
According to **GP1** with *N*-(4-chloro-2-iodophenyl)acetamide (59.3 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **8** as white solid (37.0 mg, 54%). Mp: 197-199 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.7 Hz, 1H), 7.51 (d, *J* = 2.1 Hz, 1H), 7.38-7.25 (m, 9H), 7.19-7.16 (m, 2H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.4, 136.1, 135.1, 132.4, 132.3, 130.7, 130.5, 129.9, 129.3, 128.9, 128.7, 128.4, 127.2, 125.5, 122.6, 119.1, 117.4, 27.8; **HRMS** (ESI) calculated for $C_{22}H_{17}ClNO$ $[M+H]^+$ m/z 346.0993, found 346.1000.

1-(2,3-Diphenyl-5-(trifluoromethyl)-1*H*-indol-1-yl)ethanone (9)



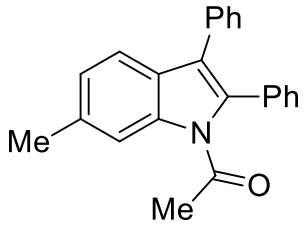
According to **GP1** with *N*-(2-iodo-4-(trifluoromethyl)phenyl)acetamide (65.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **9** as white solid (33.1 mg, 44%). Mp: 153-155 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.55 (d, *J* = 8.7 Hz, 1H), 7.83-7.80 (m, 1H), 7.64 (dd, *J* = 8.8, 1.5 Hz, 1H), 7.40-7.28 (m, 8H), 7.22-7.19 (m, 2H), 2.01 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.6, 138.2 (q, *J* = 1.1 Hz), 136.6, 132.2, 132.1, 130.7, 129.9, 129.1, 129.0, 128.8, 128.5, 127.3, 126.0 (q, *J* = 31.9 Hz), 124.7 (q, *J* = 270.3 Hz), 123.1, 122.1 (q, *J* = 3.7 Hz), 117.0 (q, *J* = 4.0 Hz), 116.5, 27.9; **HRMS** (ESI) calculated for $C_{23}H_{17}F_3NO$ $[M+H]^+$ m/z 380.1257, found 380.1255.

1-(2,3,5-Triphenyl-1*H*-indol-1-yl)ethanone (10)



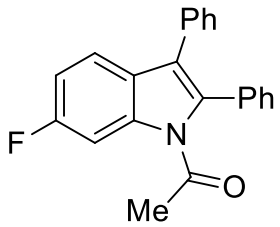
According to **GP1** with *N*-(3-iodo-[1,1'-biphenyl]-4-yl)acetamide (68.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **10** as white solid (57.0 mg, 74%). Mp: 152-154 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.39 (dd, *J* = 8.7, 0.3 Hz, 1H), 7.76-7.20 (m, 1H), 7.67-7.60 (m, 3H), 7.44-7.23 (m, 13H), 2.01 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.5, 141.5, 137.2, 136.2, 135.6, 132.9, 132.8, 130.7, 130.0, 129.7, 128.7, 128.6, 128.3, 127.4, 127.0, 126.9, 124.9, 123.5, 117.9, 116.5, 27.9; **HRMS** (ESI) calculated for $C_{28}H_{22}NO$ $[M+H]^+$ m/z 388.1696, found 388.1698.

1-(6-Methyl-2,3-diphenyl-1*H*-indol-1-yl)ethanone (11)[2]



According to **GP1** with *N*-(2-iodo-5-methylphenyl)acetamide (55.6 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (53.7 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (15.9 mg, 0.03 mmol, 0.15 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **11** as white solid (60.8 mg, 93%). Mp: 118-120 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.36-7.19 (m, 10H), 7.13 (dd, *J* = 8.0, 0.8 Hz, 1H), 2.52 (s, 3H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.7, 137.1, 135.6, 134.3, 133.2, 133.0, 130.7, 130.0, 128.54, 128.46, 128.2, 127.0, 126.8, 125.1, 123.2, 119.1, 116.3, 28.0, 22.0; **HRMS** (ESI) calculated for $C_{23}H_{20}NO$ $[M+H]^+$ m/z 326.1539, found 326.1538.

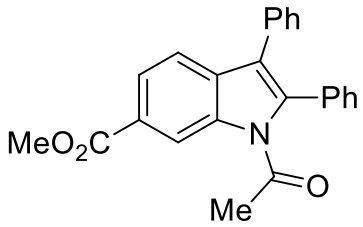
1-(6-Fluoro-2,3-diphenyl-1*H*-indol-1-yl)ethanone (12)



According to **GP1** with *N*-(5-fluoro-2-iodophenyl)acetamide (56.1 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **12** as white solid (25.3 mg, 38%). Mp: 145-147 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.23 (dd, *J* = 10.7, 2.4 Hz, 1H), 7.46 (dd, *J* = 8.7, 5.5 Hz, 1H), 7.38-7.31 (m, 5H), 7.29-7.24 (m, 3H), 7.22-7.18 (m, 2H), 7.05 (td, *J* = 8.9, 2.4 Hz, 1H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.5, 161.2 (d, *J* = 239.6 Hz), 136.9 (d, *J* = 12.7 Hz), 135.1 (d, *J* = 3.8 Hz), 132.7 (d, *J* = 2.3 Hz), 130.7, 129.9, 128.8, 128.7, 128.3, 127.1, 125.6 (d, *J* = 1.7 Hz), 123.0 (d, *J* = 1.1 Hz), 120.2 (d, *J* = 9.9 Hz), 111.9 (d, *J* = 24.2 Hz), 103.7 (d, *J* = 28.6 Hz), 27.8; **HRMS** (ESI) calculated for $C_{22}H_{16}FNONa$ $[M+Na]^+$ m/z 352.1108, found

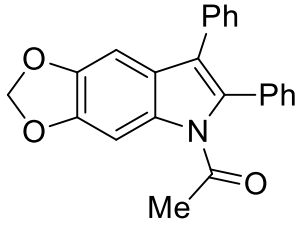
352.1201.1113.

Methyl 1-acetyl-2,3-diphenyl-1*H*-indole-6-carboxylate (13)



According to **GP1** with methyl 3-acetamido-4-iodobenzoate (63.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 30/1) to afford the desired product **13** as white solid (64.2 mg, 87%). Mp: 163-165 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 9.11 (d, *J* = 0.9 Hz, 1H), 8.00 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.40-7.32 (m, 5H), 7.30-7.26 (m, 3H), 7.22-7.18 (m, 2H), 3.96 (s, 3H), 2.02 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.3, 167.6, 137.8, 136.1, 132.7, 132.4, 132.3, 130.6, 129.9, 129.0, 128.7, 128.3, 127.1, 127.0, 125.0, 122.9, 119.2, 117.8, 52.1, 27.9; **HRMS** (ESI) calculated for $C_{24}H_{20}NO_3$ $[M+H]^+$ m/z 370.1438, found 370.1438.

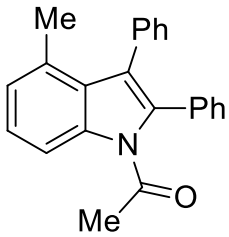
1-(6,7-Diphenyl-5*H*-[1,3]dioxolo[4,5-*f*]indol-5-yl)ethanone (14)[3]



According to **GP1** with *N*-(6-iodobenzo[*d*][1,3]dioxol-5-yl)acetamide (61.5 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **14** as white solid (37.9 mg, 53%). Mp: 194-196 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.35-7.29 (m, 5H), 7.28-7.23 (m, 3H), 7.18-7.15 (m, 2H), 6.91 (s, 1H), 5.99 (s, 2H), 1.96 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.6, 146.8, 145.1, 133.74, 133.72, 133.10, 133.08, 131.6, 130.8, 129.9, 128.5, 128.4, 128.2, 126.9, 123.5, 101.2, 98.4, 98.0, 27.9; **HRMS** (ESI)

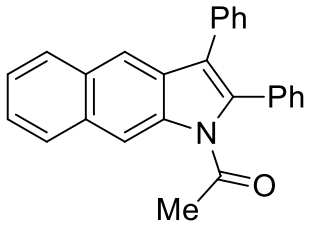
calculated for $C_{23}H_{18}NO_3$ $[M+H]^+$ m/z 356.1281, found 356.1281.

1-(4-Methyl-2,3-diphenyl-1*H*-indol-1-yl)ethanone (15)



According to **GP1** with *N*-(2-iodo-3-methylphenyl)acetamide (55.1 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **15** as white solid (24.8 mg, 38%). Mp: 171-173 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.35 (d, *J* = 8.4 Hz, 1H), 7.29-6.99 (m, 11H), 7.00 (d, *J* = 7.2 Hz, 1H), 2.01 (s, 3H), 1.97 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.5, 136.5, 135.6, 135.3, 133.0, 131.10, 131.07, 130.4, 128.2, 128.1, 127.5, 127.0, 125.6, 125.2, 124.7, 113.7, 27.9, 20.1; **HRMS** (ESI) calculated for $C_{23}H_{20}NO$ $[M+H]^+$ m/z 326.1539, found 326.1540.

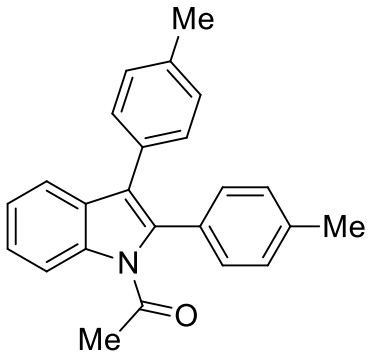
1-(2,3-Diphenyl-1*H*-benzo[*f*]indol-1-yl)ethanone (16)



According to **GP1** with *N*-(3-iodonaphthalen-2-yl)acetamide (62.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **16** as white solid (14.3 mg, 20%). Mp: 226-228 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.56 (d, *J* = 9.0 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 9.3 Hz, 1H), 7.56 (d, *J* = 8.7 Hz, 1H), 7.40-7.31 (m, 6H), 7.28 (s, 5H), 7.24-7.18 (m, 1H), 2.06 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 172.2, 135.4, 134.7, 133.5, 132.9, 131.2, 131.0, 130.6, 128.5, 128.34, 128.30, 128.2, 127.6, 127.4, 126.1, 125.7, 125.0, 124.3, 123.7, 122.9, 115.7, 28.2; **HRMS** (ESI) calculated

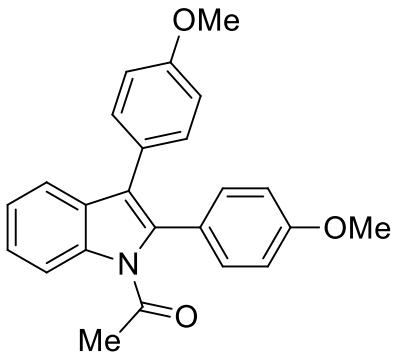
for $C_{26}H_{20}NO$ $[M+H]^+$ m/z 362.1539, found 362.1540.

1-(2,3-Di-*p*-tolyl-1*H*-indol-1-yl)ethanone (17)[2]



According to **GP1** with *N*-(2-iodophenyl)acetamide (52.2 mg, 0.2 mmol, 1.0 equiv), 1,2-di-*p*-tolylethyne (62.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **17** as white solid (62.8 mg, 93%). Mp: 156-158 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 7.5 Hz, 1H), 7.40-7.34 (m, 1H), 7.29-7.07 (m, 9H), 2.35 (s, 3H), 2.31 (s, 3H), 1.98 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.7, 138.4, 136.7, 136.4, 134.9, 130.6, 130.0, 129.9, 129.8, 129.4, 129.3, 128.9, 125.2, 123.6, 122.9, 119.5, 116.1, 27.9, 21.3, 21.2; **HRMS** (ESI) calculated for $C_{24}H_{22}NO$ $[M+H]^+$ m/z 340.1696, found 340.1692.

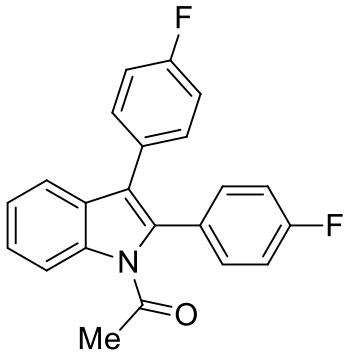
1-(2,3-Bis(4-methoxyphenyl)-1*H*-indol-1-yl)ethanone (18)[2]



According to **GP1** with *N*-(2-iodophenyl)acetamide (52.3 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-methoxyphenyl)ethyne (71.6 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **18** as white solid (65.8 mg, 89%). Mp: 139-141 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* =

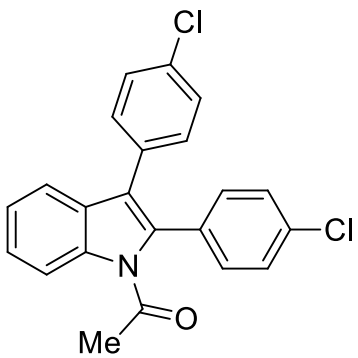
7.8 Hz, 1H), 7.41-7.36 (m, 1H), 7.31-7.22 (m, 3H), 7.18-7.13 (m, 2H), 6.91-6.82 (m, 4H), 3.83 (s, 3H), 3.79 (s, 3H), 2.01 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.7, 159.7, 158.4, 136.6, 134.6, 132.0, 131.1, 129.4, 125.4, 125.2, 125.1, 123.6, 122.6, 119.4, 116.2, 114.1, 113.7, 55.2, 55.1, 27.9; **HRMS** (ESI) calculated for $C_{24}H_{22}NO_3$ $[M+H]^+$ m/z 372.1594, found 372.1594.

1-(2,3-Bis(4-fluorophenyl)-1*H*-indol-1-yl)ethanone (19)



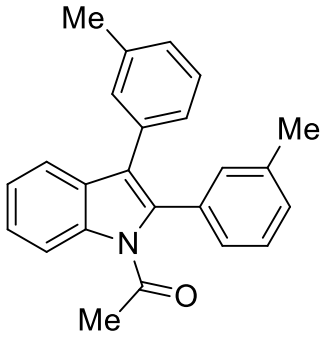
According to **GP1** with *N*-(2-iodophenyl)acetamide (53.2 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-fluorophenyl)ethyne (65.0 mg, 0.3 mmol, 1.5 equiv), $Ni(dppp)Cl_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **19** as white solid (56.0 mg, 81%). Mp: 154-156 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.1 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.44-7.38 (m, 1H), 7.33-7.23 (m, 3H), 7.18-7.14 (m, 2H), 7.10-6.96 (m, 4H), 2.03 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.1, 162.7 (d, *J* = 248.4 Hz), 161.8 (d, *J* = 245.0 Hz), 136.6, 133.9, 132.4 (d, *J* = 8.3 Hz), 131.5 (d, *J* = 8.3 Hz), 129.0, 128.8 (d, *J* = 5.0 Hz), 128.7 (d, *J* = 4.4 Hz), 125.7, 123.9, 122.7, 119.3, 116.2, 115.9 (d, *J* = 21.5 Hz), 115.4 (d, *J* = 21.5 Hz), 28.0; **HRMS** (ESI) calculated for $C_{22}H_{16}F_2NO$ $[M+H]^+$ m/z 348.1195, found 348.1191.

1-(2,3-Bis(4-chlorophenyl)-1*H*-indol-1-yl)ethanone (20)[2]



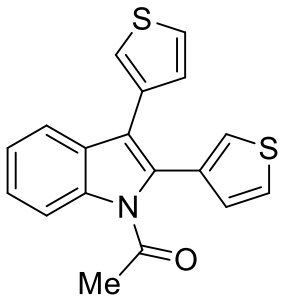
According to **GP1** with *N*-(2-iodophenyl)acetamide (52.3 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-chlorophenyl)ethyne (74.5 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **20** as white solid (36.3 mg, 48%). Mp: 192-194 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.41 (d, *J* = 8.1 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.45-7.39 (m, 1H), 7.38-7.23 (m, 7H), 7.15-7.10 (m, 2H), 2.06 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.0, 136.7, 135.0, 133.8, 133.1, 131.8, 131.2, 131.1, 129.1, 128.8, 128.7, 125.9, 124.0, 122.6, 119.4, 116.1, 28.1; **HRMS** (ESI) calculated for $C_{22}H_{16}Cl_2NO$ $[M+H]^+$ m/z 380.0603, found 380.0601.

1-(2,3-Di-*m*-tolyl-1*H*-indol-1-yl)ethanone (21)[2]



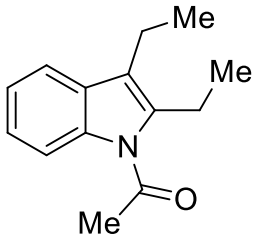
According to **GP1** with *N*-(2-iodophenyl)acetamide (52.6 mg, 0.2 mmol, 1.0 equiv), 1,2-di-*m*-tolylethyne (62.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **21** as pale yellow oil (51.5 mg, 76%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.40-7.35 (m, 1H), 7.30-6.98 (m, 9H), 2.31 (s, 3H), 2.27 (s, 3H), 1.99 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.7, 138.2, 137.6, 136.7, 135.0, 132.9, 132.8, 131.3, 130.7, 129.3, 128.4, 128.0, 127.9, 127.6, 127.1, 125.3, 123.6, 123.2, 119.6, 116.1, 27.9, 21.4, 21.3; **HRMS** (ESI) calculated for $C_{24}H_{22}NO$ $[M+H]^+$ m/z 340.1696, found 340.1692.

1-(2,3-Di(thiophen-3-yl)-1*H*-indol-1-yl)ethanone (22)



According to **GP1** with *N*-(2-iodophenyl)acetamide (53.2 mg, 0.2 mmol, 1.0 equiv), 1,2-di(thiophen-3-yl)ethyne (57.3 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **22** as yellow solid (54.2 mg, 84%). Mp: 123-125 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 7.2 Hz, 1H), 7.43-7.24 (m, 5H), 7.17 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.17 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.17 (dd, *J* = 4.8, 1.2 Hz, 1H), 2.08 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.2, 136.7, 133.0, 132.8, 129.8, 129.5, 128.7, 128.3, 126.53, 126.48, 125.6, 125.0, 123.8, 123.4, 119.5, 119.2, 116.4, 27.0; **HRMS** (ESI) calculated for $C_{18}H_{14}NOS_2$ $[M+H]^+$ m/z 324.0511, found 324.0513.

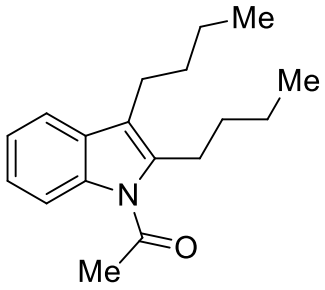
1-(2,3-Diethyl-1*H*-indol-1-yl)ethanone (23)



According to **GP1** with *N*-(2-iodophenyl)acetamide (53.0 mg, 0.2 mmol, 1.0 equiv), hex-3-yne (25.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **23** as yellow oil (35.8 mg, 83%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.77-7.34 (m, 1H), 7.51-7.48 (m, 1H), 7.27-7.21 (m, 2H), 3.03 (q, *J* = 7.3 Hz, 2H), 2.77 (s, 3H), 2.68 (q, *J* = 7.6 Hz, 2H), 1.25-1.19 (m, 6H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 170.0, 139.3, 135.7, 130.6, 123.4, 122.6, 121.2, 118.6, 114.6, 27.7, 20.3, 17.0, 15.0; **HRMS** (ESI) calculated for $C_{14}H_{18}NO$ $[M+H]^+$ m/z 216.1383, found

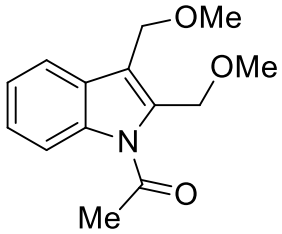
216.1379.

1-(2,3-Dibutyl-1*H*-indol-1-yl)ethanone (24)[2]



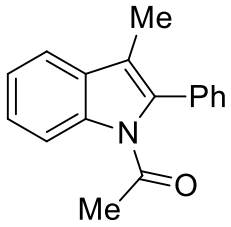
According to **GP1** with *N*-(2-iodophenyl)acetamide (52.8 mg, 0.2 mmol, 1.0 equiv), dec-5-yne (42.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **24** as yellow oil (40.3 mg, 74%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.75-7.72 (m, 1H), 7.49-7.45 (m, 1H), 7.25-7.19 (m, 2H), 3.01-2.96 (m, 2H), 2.74 (s, 3H), 2.67-2.61 (m, 2H), 1.63-1.51 (m, 4H), 1.47-1.34 (m, 4H), 0.95 (q, *J* = 7.2 Hz, 6H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 170.0, 138.2, 135.6, 130.9, 123.3, 122.5, 120.0, 118.6, 114.4, 32.43, 32.38, 27.6, 26.7, 23.6, 22.83, 22.76, 14.0, 13.9; **HRMS** (ESI) calculated for $C_{18}H_{26}NO$ $[M+H]^+$ m/z 272.2009, found 272.2002.

1-(2,3-Bis(methoxymethyl)-1*H*-indol-1-yl)ethanone (25)



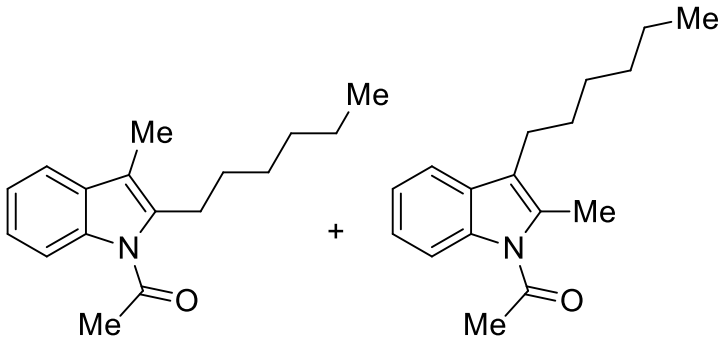
According to **GP1** with *N*-(2-iodophenyl)acetamide (52.6 mg, 0.2 mmol, 1.0 equiv), 1,4-dimethoxybut-2-yne (35.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product **25** as yellow oil (36.9 mg, 75%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.36-7.24 (m, 2H), 4.79 (s, 2H), 4.66 (s, 2H), 3.39 (s, 3H), 3.36 (s, 3H), 2.79 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 175.0, 136.4, 133.7, 128.8, 125.4, 123.2, 120.1, 119.4, 115.7, 64.5, 64.3, 57.9, 57.7, 26.2; **HRMS** (ESI) calculated for $C_{14}H_{18}NO_3$ $[M+H]^+$ m/z 248.1281, found 248.1284.

1-(3-Methyl-2-phenyl-1*H*-indol-1-yl)ethanone (26)[4]



According to **GP1** with *N*-(2-iodophenyl)acetamide (53.1 mg, 0.2 mmol, 1.0 equiv), prop-1-yn-1-ylbenzene (35.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **26** as yellow solid (11.3 mg, 23%). Mp: 74-76 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.1 Hz, 1H), 7.54-7.43 (m, 4H), 7.40-7.29 (m, 4H), 2.14 (s, 3H), 1.96 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.1, 136.8, 134.9, 133.6, 130.2, 128.7, 128.4, 125.3, 123.4, 118.6, 118.1, 116.3, 27.6, 9.2; **HRMS** (ESI) calculated for $C_{17}H_{16}NO$ $[M+H]^+$ m/z 250.1226, found 250.1225.

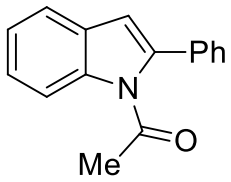
1-(2-Hexyl-3-methyl-1*H*-indol-1-yl)ethanone (27) and **1-(3-hexyl-2-methyl-1*H*-indol-1-yl)ethanone (27')[5]**



According to **GP1** with *N*-(2-iodophenyl)acetamide (52.7 mg, 0.2 mmol, 1.0 equiv), non-2-yne (38.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **27+27'** as yellow oil (45.6 mg, 89%, **27/27' = 1.3:1**). **^{1}H NMR** (300 MHz, $CDCl_3$) δ **27** 7.97-7.91 (m, 1H), 7.45-7.42 (m, 1H), 7.26-7.19 (m, 2H), 2.69 (s, 3H), 2.63 (t, *J* = 7.5 Hz, 2H), 2.55 (s, 3H), 1.60-1.53 (m, 2H), 1.35-1.25 (m, 6H), 0.90-0.86 (m, 3H); **27'** 7.77-7.71 (m, 1H), 7.45-7.42 (m, 1H), 7.26-7.19 (m, 2H), 2.98 (t, *J* = 7.7 Hz, 2H), 2.73 (s, 3H), 2.18 (s, 3H), 1.60-1.53 (m, 2H), 1.35-1.25

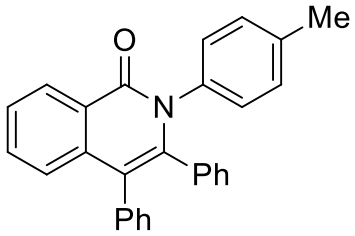
(m, 6H), 0.90-0.86 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ **27** 170.2, 135.7, 132.5, 130.6, 123.5, 122.7, 120.3, 118.2, 115.0, 31.7, 29.9, 29.2, 27.5, 23.8, 22.6, 14.3, 14.0; **27'** 169.9, 138.2, 135.4, 131.4, 123.4, 122.6, 118.4, 115.2, 114.4, 31.6, 29.8, 29.2, 27.5, 27.0, 22.6, 14.0, 8.6; **HRMS** (ESI) calculated for $C_{17}H_{24}NO$ $[M+H]^+$ m/z 258.1852, found 258.1852.

1-(2-Phenyl-1*H*-indol-1-yl)ethanone (28)[6]



According to **GP1** with *N*-(2-iodophenyl)acetamide (52.5 mg, 0.2 mmol, 1.0 equiv), ethynylbenzene (31.0 mg, 0.3 mmol, 1.5 equiv), $Ni(dppp)Cl_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **28** as yellow oil (10.6 mg, 23%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.36 (d, *J* = 8.1 Hz, 1H), 7.56 (d, *J* = 7.5 Hz, 1H), 7.48-7.42 (m, 5H), 7.39-7.26 (m, 2H), 6.63 (s, 1H), 2.08 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 171.4, 139.7, 137.7, 134.2, 129.0, 128.7, 128.6, 125.1, 123.7, 120.4, 116.0, 111.5, 27.9; **HRMS** (ESI) calculated for $C_{16}H_{14}NO$ $[M+H]^+$ m/z 236.1070, found 236.1066.

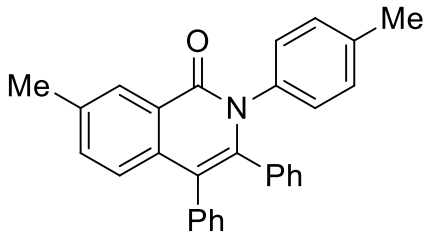
3,4-Diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (30)[7]



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.0 mg, 0.24 mmol, 1.2 equiv), $Ni(dppp)Cl_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **30** as white solid (73.7 mg, 95%). Mp: 216-218 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.57 (d, *J* = 7.5 Hz, 1H), 7.58-7.47 (m, 2H), 7.26-7.12 (m, 6H), 6.99 (s, 4H), 6.89 (s, 5H), 2.21 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 141.2, 137.6, 137.2, 136.8, 136.4, 134.9, 132.4, 131.6, 131.0, 129.2, 129.1, 128.2,

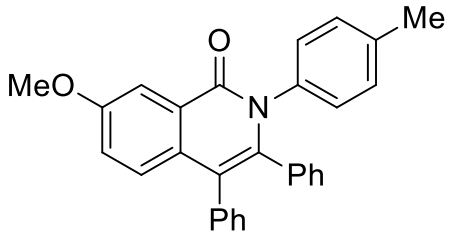
127.9, 127.1, 127.0, 126.8, 126.7, 125.5, 118.6, 21.0; **HRMS** (ESI) calculated for $C_{28}H_{22}NO$ $[M+H]^+$ m/z 388.1696, found 388.1704.

7-Methyl-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (31)



According to **GP2** with 2-iodo-5-methyl-*N*-(*p*-tolyl)benzamide (70.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **31** as white solid (77.9 mg, 97%). Mp: 204-206 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.39 (d, *J* = 8.7 Hz, 1H), 7.22-7.10 (m, 6H), 7.01-6.95 (m, 4H), 6.89 (s, 5H), 2.50 (s, 3H), 2.22 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 143.3, 137.1, 136.9, 136.8, 136.6, 135.3, 135.0, 133.8, 131.6, 131.1, 129.2, 129.1, 127.8, 127.02, 126.98, 126.7, 125.5, 125.4, 118.6, 21.3, 21.0; **HRMS** (ESI) calculated for $C_{29}H_{24}NO$ $[M+H]^+$ m/z 402.1852, found 402.1853.

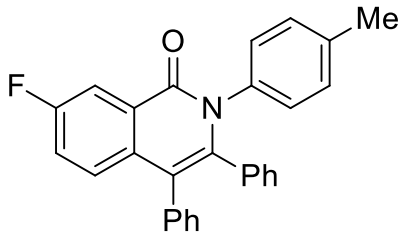
7-Methoxy-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (32)[7]



According to **GP2** with 2-iodo-5-methoxy-*N*-(*p*-tolyl)benzamide (73.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 8/1) to afford the desired product **32** as white solid (82.0 mg, 98%). Mp: 206-208 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.24-7.10 (m, 7H), 6.99-6.96 (m, 4H), 6.88 (s, 5H), 3.92 (s, 3H), 2.22 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.4, 158.8, 138.9, 137.1, 137.0, 136.6, 134.9, 131.6, 131.5, 131.2, 129.2,

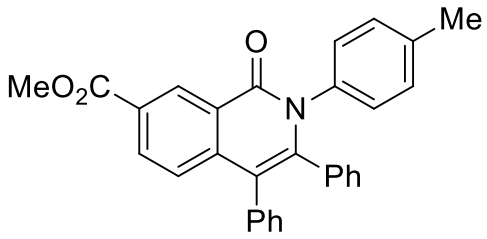
129.1, 127.8, 127.3, 127.0, 126.7, 22.7, 118.6, 108.2, 55.6, 21.0; **HRMS** (ESI) calculated for $C_{29}H_{24}NO_2$ $[M+H]^+$ m/z 418.1802, found 418.1804.

7-Fluoro-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (33)



According to **GP2** with 5-fluoro-2-iodo-*N*-(*p*-tolyl)benzamide (71.3 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.4 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **33** as white solid (78.2 mg, 96%). Mp: 187-189 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.19 (d, *J* = 9.0 Hz, 1H), 7.31-7.10 (m, 7H), 7.01-6.98 (m, 4H), 6.89 (s, 5H), 2.21 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.9 (d, *J* = 3.8 Hz), 161.5 (d, *J* = 246.8 Hz), 140.5 (d, *J* = 3.0 Hz), 137.4, 136.5, 136.2, 134.5, 134.2 (d, *J* = 2.3 Hz), 131.2 (d, *J* = 36.8 Hz), 129.1 (d, *J* = 27.8 Hz), 128.2, 128.1, 128.0, 127.2, 127.13, 127.07, 127.03, 126.9, 121.0 (d, *J* = 23.3 Hz), 118.2, 113.2 (d, *J* = 22.5 Hz), 21.1; **HRMS** (ESI) calculated for $C_{28}H_{21}FNO$ $[M+H]^+$ m/z 406.1602, found 406.1601.

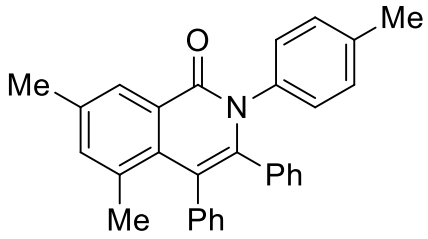
Methyl 1-oxo-3,4-diphenyl-2-(*p*-tolyl)-1,2-dihydroisoquinoline-7-carboxylate (34)



According to **GP2** with methyl 4-iodo-3-(*p*-tolylcarbamoyl)benzoate (79.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **34** as white solid (72.8 mg, 82%). Mp: 217-219 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 9.21 (dd, *J* = 1.8, 0.6 Hz, 1H), 8.17 (dd, *J* = 8.4, 1.8 Hz, 1H), 8.17 (dd, *J* = 8.7, 0.6 Hz, 1H), 7.24-7.16 (m, 3H), 7.14-7.09

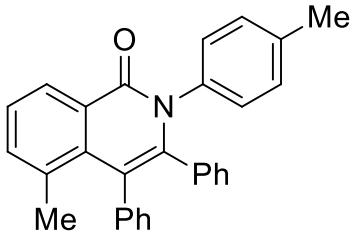
(m, 2H), 7.03-6.96 (m, 4H), 6.93-6.87 (m, 5H), 3.94 (s, 3H), 2.23 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.5, 162.4, 143.7, 140.9, 137.5, 136.4, 135.9, 134.5, 132.5, 131.5, 130.7, 130.5, 129.3, 129.0, 128.2, 128.1, 127.4, 127.1, 127.0, 125.8, 125.2, 118.4, 52.2, 21.0; **HRMS** (ESI) calculated for $C_{30}H_{24}NO_3$ $[M+H]^+$ m/z 446.1751, found 446.1754.

5,7-Dimethyl-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (35)



According to **GP2** with 2-iodo-3,5-dimethyl-*N*-(*p*-tolyl)benzamide (73.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **35** as white solid (40.5 mg, 49%). Mp: 159-161 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.34 (s, 1H), 7.25 (s, 1H), 7.07 (s, 5H), 6.99-6.91 (m, 4H), 6.88-6.79 (m, 5H), 2.45 (s, 3H), 2.21 (s, 3H), 1.75 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.9, 140.8, 139.9, 138.3, 137.1, 137.0, 136.6, 135.2, 135.0, 133.1, 132.0, 131.3, 129.2, 129.0, 127.3, 126.9, 126.74, 126.66, 126.5, 118.3, 23.7, 21.1; **HRMS** (ESI) calculated for $C_{30}H_{26}NO$ $[M+H]^+$ m/z 416.2009, found 416.2009.

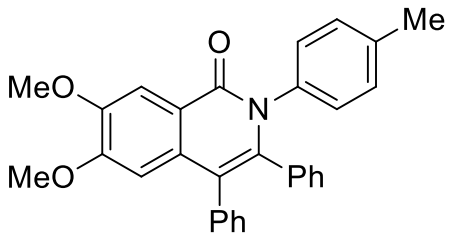
5-Methyl-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (36)



According to **GP2** with 2-iodo-3-methyl-*N*-(*p*-tolyl)benzamide (70.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.4 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **36** as white solid (60.5 mg, 75%).

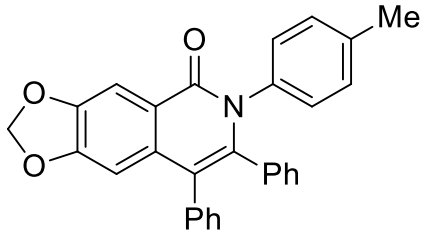
Mp: 186-188 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.55-8.52 (m, 1H), 7.40-7.39 (m, 2H), 7.08 (s, 5H), 6.99-6.92 (m, 4H), 6.84 (s, 5H), 2.20 (s, 3H), 1.78 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.9, 141.8, 139.8, 137.2, 136.9, 136.7, 135.5, 135.12, 135.10, 132.1, 131.2, 129.2, 129.0, 127.3, 127.1, 127.0, 126.80, 126.76, 126.6, 126.5, 118.3, 23.7, 21.0; **HRMS** (ESI) calculated for $C_{29}H_{24}NO$ $[M+H]^+$ m/z 402.1852, found 402.1857.

6,7-Dimethoxy-3,4-diphenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (37)[7]



According to **GP2** with 2-iodo-4,5-dimethoxy-*N*-(*p*-tolyl)benzamide (79.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 12/1) to afford the desired product **37** as white solid (78.9 mg, 88%). Mp: 293-295 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.23-7.12 (m, 5H), 7.02-6.95 (m, 4H), 6.89 (s, 5H), 6.61 (s, 1H), 4.01 (s, 3H), 3.72 (s, 3H), 2.22 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.9, 153.3, 149.1, 139.8, 137.0, 136.9, 136.6, 134.9, 132.9, 131.4, 131.0, 129.1, 129.0, 127.9, 126.9, 126.7, 119.4, 118.2, 108.1, 105.9, 56.1, 55.7, 21.0; **HRMS** (ESI) calculated for $C_{30}H_{26}NO_3$ $[M+H]^+$ m/z 448.1907, found 448.1910.

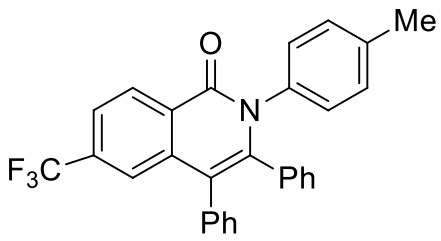
7,8-Diphenyl-6-(*p*-tolyl)-[1,3]dioxolo[4,5-*g*]isoquinolin-5(6*H*)-one (38)



According to **GP2** with 6-iodo-*N*-(*p*-tolyl)benzo[*d*][1,3]dioxole-5-carboxamide (76.5 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.3 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography

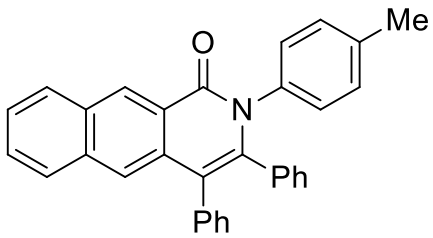
(petroleum ether/EtOAc = 10/1) to afford the desired product **38** as white solid (78.5 mg, 91%). Mp: 235-237 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.22-7.08 (m, 5H), 7.01-6.95 (m, 4H), 6.88 (s, 5H), 6.59 (s, 1H), 6.00 (s, 2H), 2.21 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.8, 152.0, 147.6, 140.0, 137.1, 136.9, 136.7, 135.0, 134.9, 131.5, 131.0, 129.13, 129.05, 127.9, 127.02, 126.96, 126.8, 121.0, 118.5, 106.0, 103.7, 101.6, 21.0; **HRMS** (ESI) calculated for $C_{29}H_{22}NO_3$ $[M+H]^+$ m/z 432.1594, found 432.1592.

3,4-Diphenyl-2-(*p*-tolyl)-6-(trifluoromethyl)isoquinolin-1(2*H*)-one (39)



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)-4-(trifluoromethyl)benzamide (81.0 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.2 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.8 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **39** as white solid (33.0 mg, 36%). Mp: 250-252 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.4 Hz, 1H), 7.71 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.54-7.51 (m, 1H), 7.26-7.19 (m, 3H), 7.13-7.10 (m, 2H), 7.04-6.86 (m, 9H), 2.24 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.9, 142.8, 137.7, 137.6, 136.3, 135.3, 134.3, 134.0 (q, *J* = 31.9 Hz), 131.4, 130.8, 129.39, 129.36, 128.9, 128.2, 127.6 (q, *J* = 1.1 Hz), 127.4, 127.24, 127.15, 123.7 (q, *J* = 271.5 Hz), 122.7 (q, *J* = 3.8 Hz), 121.9, 21.0; **HRMS** (ESI) calculated for $C_{29}H_{21}F_3NO$ $[M+H]^+$ m/z 456.1570, found 456.1572.

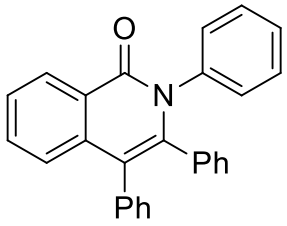
3,4-Diphenyl-2-(*p*-tolyl)benzo[*g*]isoquinolin-1(2*H*)-one (40)



According to **GP2** with 3-iodo-*N*-(*p*-tolyl)-2-naphthamide (77.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg,

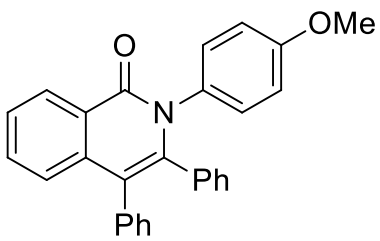
0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **40** as white solid (86.0 mg, 98%). Mp: 236-238 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 9.17 (s, 1H), 8.09-8.06 (m, 1H), 7.77-7.34 (m, 1H), 7.67 (s, 1H), 7.53-7.46 (m, 2H), 7.27-7.19 (m, 5H), 7.02 (s, 4H), 6.94-6.86 (m, 5H), 2.23 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 163.2, 140.1, 137.1, 136.8, 136.7, 135.3, 135.0, 133.9, 131.7, 131.6, 131.0, 129.6, 129.3, 129.2, 128.0, 127.02, 126.98, 126.8, 126.0, 124.3, 124.0, 118.6, 21.0; **HRMS** (ESI) calculated for $C_{32}H_{24}NO$ $[M+H]^+$ m/z 438.1852, found 438.1858.

2,3,4-Triphenylisoquinolin-1(2*H*)-one (41)[8]



According to **GP2** with 2-iodo-*N*-phenylbenzamide (64.7 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **41** as white solid (70.0 mg, 94%). Mp: 201-203 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.57 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.61-7.49 (m, 2H), 7.24-7.09 (m, 11H), 6.89 (s, 5H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.6, 141.1, 139.5, 137.6, 136.4, 134.8, 132.5, 131.6, 131.0, 129.5, 128.5, 128.2, 127.9, 127.5, 127.2, 127.1, 126.85, 126.83, 125.6, 118.8; **HRMS** (ESI) calculated for $C_{27}H_{20}NO$ $[M+H]^+$ m/z 374.1539, found 374.1540.

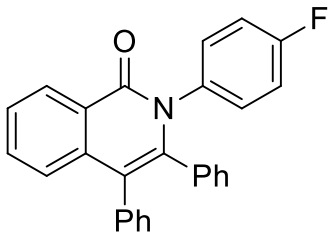
2-(4-Methoxyphenyl)-3,4-diphenylisoquinolin-1(2*H*)-one (42)[9]



According to **GP2** with 2-iodo-*N*-(4-methoxyphenyl)benzamide (71.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1

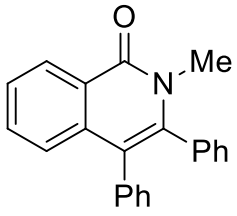
mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **42** as white solid (73.9 mg, 92%). Mp: 209-211 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.56 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.58-7.47 (m, 2H), 7.26-7.11 (m, 6H), 7.02-6.99 (m, 2H), 6.95-6.84 (m, 5H), 6.73-6.70 (m, 2H), 3.68 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 158.3, 141.3, 137.5, 136.3, 134.8, 132.3, 132.1, 131.5, 130.8, 130.2, 128.1, 127.8, 127.1, 127.0, 126.7, 125.4, 118.5, 113.7, 55.1; **HRMS** (ESI) calculated for $C_{28}H_{22}NO_2$ $[M+H]^+$ m/z 404.1645, found 404.1650.

2-(4-Fluorophenyl)-3,4-diphenylisoquinolin-1(2*H*)-one (43)[9]



According to **GP2** with *N*-(4-fluorophenyl)-2-iodobenzamide (68.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **43** as white solid (71.1 mg, 91%). Mp: 190-192 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.56 (d, *J* = 7.8 Hz, 1H), 7.61-7.50 (m, 2H), 7.27-7.06 (m, 8H), 6.91-6.86 (m, 7H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 161.4 (d, *J* = 245.3 Hz), 140.8, 137.5, 136.1, 135.3 (d, *J* = 3.8 Hz), 134.5, 132.6, 131.5, 131.1, 131.0, 130.9, 128.2, 128.0, 127.2, 127.2 (d, *J* = 30.0 Hz), 126.9, 125.6, 125.3, 119.0, 115.5 (d, *J* = 22.5 Hz); **HRMS** (ESI) calculated for $C_{27}H_{19}FNO$ $[M+H]^+$ m/z 392.1445, found 392.1449.

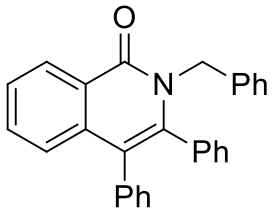
2-Methyl-3,4-diphenylisoquinolin-1(2*H*)-one (44)[9]



According to **GP2** with 2-iodo-*N*-methylbenzamide (52.2 mg, 0.20 mmol, 1.0 equiv),

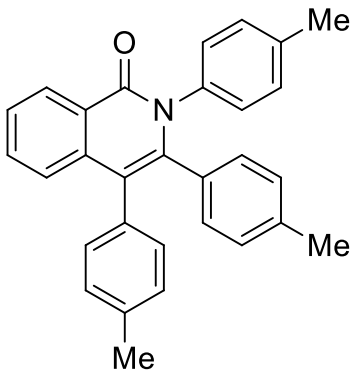
1,2-diphenylethyne (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **44** as white solid (26.6 mg, 43%). Mp: 152-154 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.58-8.55 (m, 1H), 7.56-7.46 (m, 2H), 7.26-7.11 (m, 9H), 7.08-7.05 (m, 2H), 3.36 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 141.2, 137.1, 136.4, 135.0, 132.0, 131.5, 129.9, 128.2, 127.9, 127.8, 126.8, 126.6, 125.3, 124.9, 118.9, 34.3; **HRMS** (ESI) calculated for $C_{22}H_{18}NO$ $[M+H]^+$ m/z 312.1383, found 312.1389.

2-Benzyl-3,4-diphenylisoquinolin-1(2*H*)-one (45)[9]



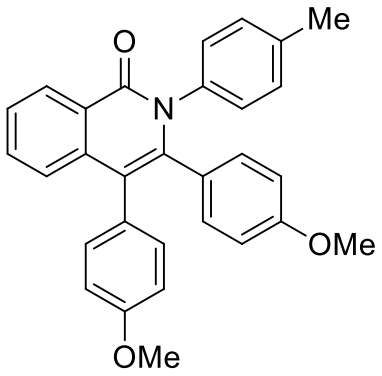
According to **GP2** with *N*-benzyl-2-iodobenzamide (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.8 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **45** as white solid (15.8 mg, 20%). Mp: 228-230 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.63-8.60 (m, 1H), 7.56-7.47 (m, 2H), 7.18-7.08 (m, 8H), 7.07-7.01 (m, 4H), 6.90-6.87 (m, 4H), 5.22 (s, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.6, 141.3, 137.7, 137.3, 136.3, 134.3, 132.2, 131.4, 130.4, 128.2, 128.1, 128.0, 127.8, 127.5, 126.9, 126.8, 126.70, 126.65, 125.4, 125.1, 119.4, 49.0; **HRMS** (ESI) calculated for $C_{28}H_{22}NO$ $[M+H]^+$ m/z 388.1696, found 388.1697.

2,3,4-Tri-*p*-tolylisoquinolin-1(2*H*)-one (46)



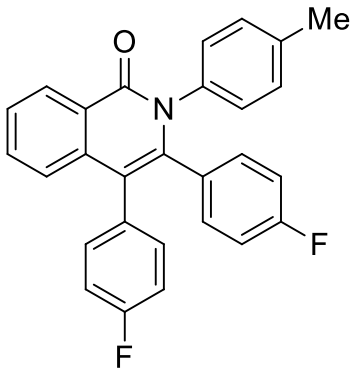
According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-di-*p*-tolylethyne (49.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **46** as white solid (78.5 mg, 94%). Mp: 84-86 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.54 (d, *J* = 7.8 Hz, 1H), 7.56-7.45 (m, 2H), 7.25-7.22 (m, 1H), 7.00-6.97 (m, 8H), 6.78-6.68 (m, 4H), 2.27 (s, 3H), 2.22 (s, 3H), 2.07 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.8, 141.2, 137.9, 137.0, 136.9, 136.6, 136.1, 133.5, 132.2, 132.0, 131.4, 130.8, 129.2, 129.1, 128.6, 128.1, 127.7, 126.5, 125.5, 125.4, 118.6, 21.1, 21.03, 21.01; **HRMS** (ESI) calculated for $C_{30}H_{26}NO$ $[M+H]^+$ m/z 416.2009, found 416.2014.

3,4-Bis(4-methoxyphenyl)-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (47)



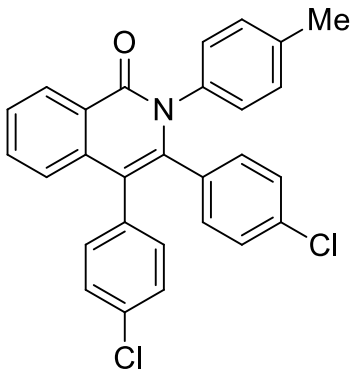
According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-methoxyphenyl)ethyne (58.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product **47** as white solid (80.7 mg, 90%). Mp: 245-247 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.54 (d, *J* = 7.5 Hz, 1H), 7.57-7.44 (m, 2H), 7.27-7.24 (m, 1H), 7.04-6.94 (m, 6H), 6.80-6.73 (m, 4H), 6.48-6.38 (m, 2H), 3.73 (s, 3H), 3.59 (s, 3H), 2.23 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.8, 158.1, 158.0, 141.2, 138.0, 137.1, 137.0, 132.6, 132.3, 132.1, 129.3, 129.0, 128.8, 128.2, 127.4, 126.6, 125.5, 125.4, 118.4, 113.4, 112.5, 55.1, 54.9, 21.0; **HRMS** (ESI) calculated for $C_{30}H_{26}NO_3$ $[M+H]^+$ m/z 448.1907, found 448.1910.

3,4-Bis(4-fluorophenyl)-2-(*p*-tolyl)isoquinolin-1(2H)-one (48)



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-fluorophenyl)ethyne (51.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.8 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 12/1) to afford the desired product **48** as white solid (82.3 mg, 97%). Mp: 186-188 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.58-8.54 (m, 1H), 7.62-7.50 (m, 2H), 7.22-7.19 (m, 1H), 7.10-7.02 (m, 4H), 6.97-6.82 (m, 6H), 6.67-6.59 (m, 2H), 2.25 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.7 (d, *J* = 245.3 Hz), 162.6, 161.4 (d, *J* = 246.8 Hz), 140.5, 137.6, 137.4, 136.6, 133.1 (d, *J* = 8.3 Hz), 132.7 (d, *J* = 8.3 Hz), 132.6, 132.2 (d, *J* = 3.8 Hz), 130.9 (d, *J* = 3.8 Hz), 129.5, 129.0, 128.4, 127.1, 125.6, 125.3, 118.0, 115.2 (d, *J* = 21.0 Hz), 114.4 (d, *J* = 21.8 Hz), 21.1; **HRMS** (ESI) calculated for $C_{28}H_{20}F_2NO$ $[M+H]^+$ m/z 424.1508, found 424.1506.

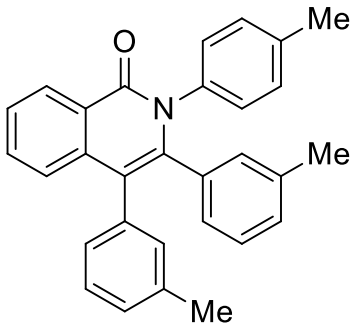
3,4-Bis(4-chlorophenyl)-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (49)



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-chlorophenyl)ethyne (59.7 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude

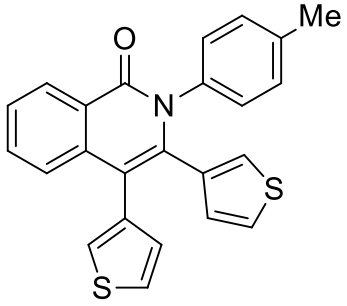
reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **49** as white solid (84.9 mg, 93%). Mp: 145-147 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.55 (d, *J* = 7.5 Hz, 1H), 7.60-7.49 (m, 2H), 7.24-7.17 (m, 3H), 7.06-7.02 (m, 4H), 6.96-6.91 (m, 4H), 6.83-6.81 (m, 2H), 2.25 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.5, 140.1, 137.7, 137.0, 136.3, 134.6, 133.4, 133.1, 133.0, 132.8, 132.6, 132.1, 129.5, 128.9, 128.43, 128.36, 127.6, 127.2, 125.5, 125.2, 117.7, 21.1; **HRMS** (ESI) calculated for $C_{28}H_{20}Cl_2NO$ $[M+H]^+$ m/z 456.0917, found 456.0919.

3,4-Di-*m*-tolyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (50)



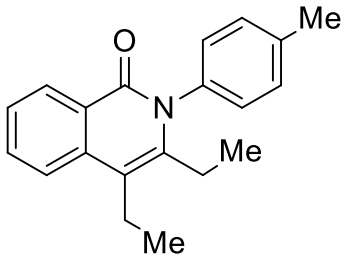
According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), 1,2-di-*m*-tolylethyne (50.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **50** as white solid (53.1 mg, 64%). Mp: 83-85 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.55 (d, *J* = 7.8 Hz, 1H), 7.58-7.50 (m, 2H), 7.27-7.24 (m, 1H), 7.14-7.06 (m, 1H), 6.98-6.90 (m, 7H), 6.80-6.67 (m, 4H), 2.22 (s, 6H), 2.02 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.7, 141.2, 137.7, 137.3, 137.1, 136.8, 136.3, 134.6, 132.3, 132.2, 131.8, 131.7, 129.0, 128.60, 128.57, 128.14, 128.06, 128.04, 127.72, 127.65, 127.4, 126.78, 126.74, 126.6, 125.6, 125.4, 118.7, 21.2, 21.02, 20.96; **HRMS** (ESI) calculated for $C_{30}H_{26}NO$ $[M+H]^+$ m/z 416.2009, found 416.2015.

3,4-Di(thiophen-3-yl)-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (51)



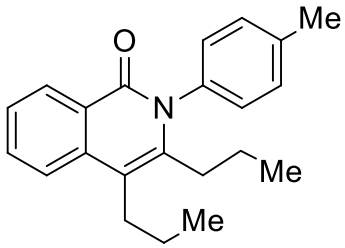
According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,2-di(thiophen-3-yl)ethyne (46.2 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **51** as white solid (71.7 mg, 90%). Mp: 207-209 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.53 (d, *J* = 7.8 Hz, 1H), 7.63-7.58 (m, 1H), 7.53-7.49 (m, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.25-7.21 (m, 1H), 7.07-6.98 (m, 5H), 6.89-6.85 (m, 2H), 6.73-6.72 (m, 1H), 6.53-6.52 (m, 1H), 2.28 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.6, 137.4, 137.3, 137.0, 136.8, 136.2, 134.8, 132.5, 130.1, 129.3, 129.2, 128.6, 128.2, 126.9, 126.6, 125.4, 125.3, 125.1, 124.9, 124.1, 114.2, 21.1; **HRMS** (ESI) calculated for $C_{24}H_{18}NOS_2$ $[M+H]^+$ m/z 400.0824, found 400.0831.

3,4-Diethyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (52)



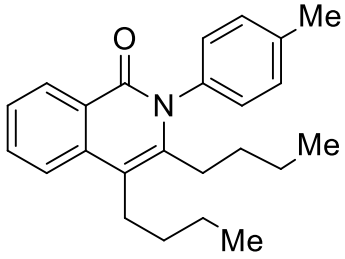
According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (68.0 mg, 0.20 mmol, 1.0 equiv), hex-3-yne (22.6 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **52** as white solid (56.4 mg, 97%). Mp: 111-113 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.45 (d, *J* = 7.8 Hz, 1H), 7.74-7.66 (m, 2H), 7.46-7.42 (m, 1H), 7.36-7.26 (m, 2H), 7.20-7.09 (m, 2H), 2.81 (q, *J* = 7.5 Hz, 2H), 2.50-2.43 (m, 5H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.00 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 163.2, 141.2, 138.2, 136.9, 132.4, 130.0, 128.6, 125.7, 125.4, 122.6, 114.6, 23.1, 21.2, 20.4, 14.7, 13.9; **HRMS** (ESI) calculated for $C_{20}H_{22}NO$ $[M+H]^+$ m/z 292.1696, found 292.1700.

3,4-Dipropyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (53)[7]



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), oct-4-yne (26.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **53** as yellow oil (58.8 mg, 92%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.44 (d, *J* = 7.8 Hz, 1H), 7.69-7.63 (m, 2H), 7.44-7.39 (m, 1H), 7.31-7.28 (m, 2H), 7.13-7.10 (m, 2H), 2.75-2.69 (m, 2H), 2.42 (s, 3H), 2.39-2.33 (m, 2H), 1.70-1.62 (m, 2H), 1.45-1.35 (m, 2H), 1.09 (t, *J* = 7.4 Hz, 3H), 0.71 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 163.1, 140.4, 138.1, 137.1, 136.9, 132.3, 129.9, 128.53, 128.50, 125.7, 125.3, 122.7, 113.5, 32.2, 29.7, 23.6, 22.8, 21.2, 14.4, 14.1; **HRMS** (ESI) calculated for $C_{22}H_{26}NO$ $[M+H]^+$ m/z 320.2009, found 320.2011.

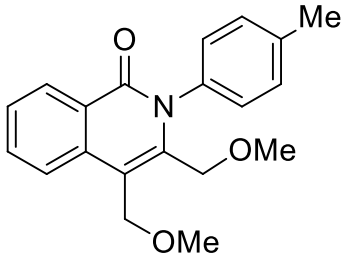
3,4-Dibutyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (54)[10]



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), dec-5-yne (34.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **54** as yellow oil (62.3 mg, 90%). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.44 (d, *J* = 7.8 Hz, 1H), 7.69-7.62 (m, 2H), 7.43-7.38 (m, 1H), 7.30-7.27 (m, 2H), 7.13-7.10 (m, 2H), 2.76-2.71 (m, 2H), 2.41 (s, 3H), 2.41-2.36 (m, 2H), 1.67-1.47 (m, 4H), 1.45-1.33 (m, 2H), 1.16-1.07 (m, 2H), 1.00 (t, *J* = 7.1 Hz, 3H), 0.70 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 163.0, 140.2, 138.0, 137.0,

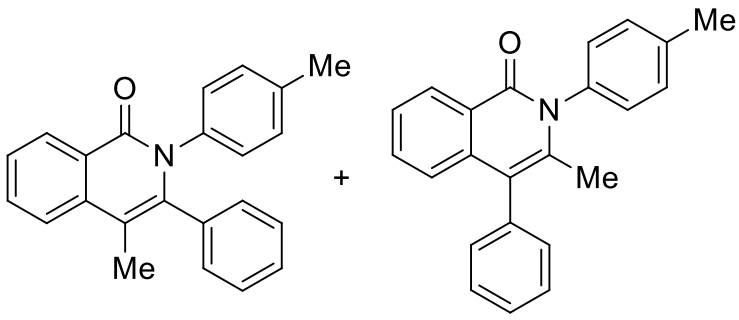
136.7, 132.2, 129.8, 128.5, 128.4, 125.5, 125.2, 122.5, 113.4, 32.4, 31.2, 29.6, 27.2, 23.0, 22.5, 21.1, 13.8, 13.2; **HRMS** (ESI) calculated for $C_{24}H_{30}NO$ $[M+H]^+$ m/z 348.2322, found 348.2322.

3,4-Bis(methoxymethyl)-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (55)



According to **GP2** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,4-dimethoxybut-2-yne (29.0 mg, 0.24 mmol, 1.2 equiv), $Ni(dppp)Cl_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product **55** as white solid (55.9 mg, 86%). Mp: 166-168 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.43 (d, *J* = 8.1 Hz, 1H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.74-7.69 (m, 1H), 7.52-7.47 (m, 1H), 7.32-7.26 (m, 2H), 7.19-7.16 (m, 2H), 4.69 (s, 2H), 4.11 (s, 2H), 3.50 (s, 3H), 3.04 (s, 3H), 2.43 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.9, 138.6, 138.4, 136.3, 135.8, 132.6, 129.7, 128.7, 128.2, 127.2, 126.2, 123.7, 113.2, 67.3, 67.1, 58.2, 58.1, 21.1; **HRMS** (ESI) calculated for $C_{20}H_{22}NO_3$ $[M+H]^+$ m/z 324.1594, found 324.1601.

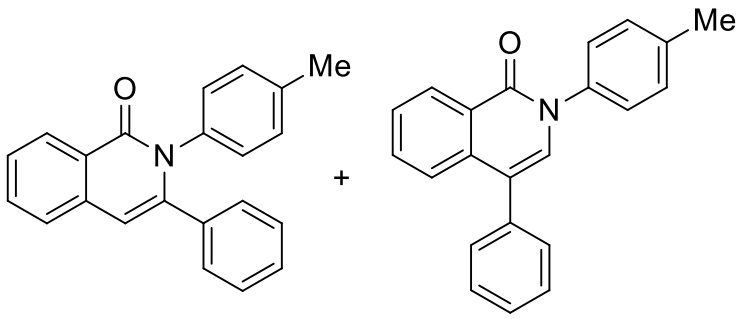
4-Methyl-3-phenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (56) and **3-methyl-4-phenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (56')[10]**



According to **GP1** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), prop-1-yn-1-ylbenzene (29.0 mg, 0.24 mmol, 1.2 equiv), $Ni(dppp)Cl_2$ (10.8 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc =

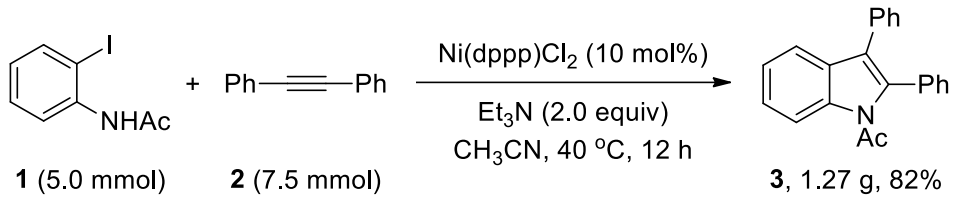
20/1) to afford the desired product **56+56'** as white solid (60.3 mg, 93%, **56/56' = 1.1:1**). **^{1}H NMR** (300 MHz, $CDCl_3$) δ **56+56'** 8.45 (d, *J* = 7.8 Hz, 1H), 8.37 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.65-7.63 (m, 2H), 7.45-7.27 (m, 6H), 7.23-7.19 (m, 4H), 7.09-7.05 (m, 5H), 6.97-6.95 (m, 3H), 6.89-6.86 (m, 2H), 6.82-6.79 (m, 2H), 2.32 (s, 3H), 2.11 (s, 3H), 1.98 (s, 3H), 1.70 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ **56** 162.5, 140.3, 137.5, 137.0, 136.9, 135.4, 132.4, 130.4, 129.08, 129.06, 128.4, 127.7, 127.6, 126.5, 125.7, 123.2, 110.1, 20.9, 14.7; **56'** 163.0, 138.2 137.7, 137.3, 136.8, 132.1, 130.9, 130.1, 128.7, 128.1, 127.9, 127.4, 125.8, 124.8, 124.6, 117.4, 21.1, 19.4; **HRMS** (ESI) calculated for $C_{23}H_{20}NO$ $[M+H]^+$ m/z 326.1539, found 326.1542.

3-Phenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (57) and **4-phenyl-2-(*p*-tolyl)isoquinolin-1(2*H*)-one (57')[10]**

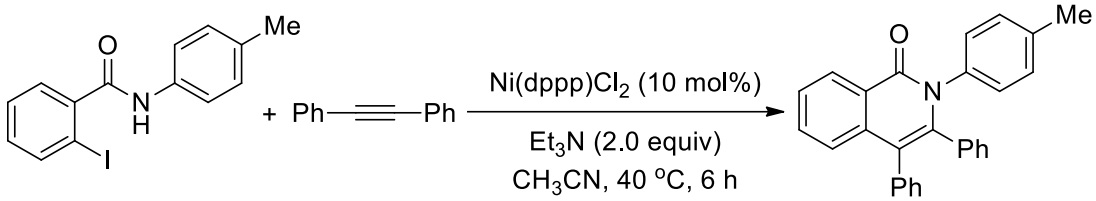


According to **GP1** with 2-iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), ethynylbenzene (25.2 mg, 0.24 mmol, 1.2 equiv), $Ni(dppp)Cl_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product **57+57'** as yellow oil (56.7 mg, 91%, **57/57' = 1:1**). **^{1}H NMR** (300 MHz, $CDCl_3$) δ **57+57'** 8.57 (d, *J* = 7.8 Hz, 1H), 8.46 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.70-7.40 (m, 11H), 7.37-7.25 (m, 4H), 7.17 (s, 6H), 7.07-6.98 (m, 4H), 6.58 (s, 1H), 2.41 (s, 3H), 2.27 (s, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ **57** 163.2, 143.7, 137.4, 136.7, 136.4, 136.2, 132.7, 129.3, 129.2, 129.0, 128.3, 127.8, 127.7, 126.8, 126.0, 125.4, 107.8, 21.1; **57'** 161.7, 138.7, 138.0, 136.4, 136.3, 132.4, 131.3, 129.92, 129.86, 128.7, 128.6, 127.9, 127.1, 126.5, 126.3, 124.7, 119.5, 21.1; **HRMS** (ESI) calculated for $C_{22}H_{18}NO$ $[M+H]^+$ m/z 312.1383, found 312.1389.

Gram-scale reactions



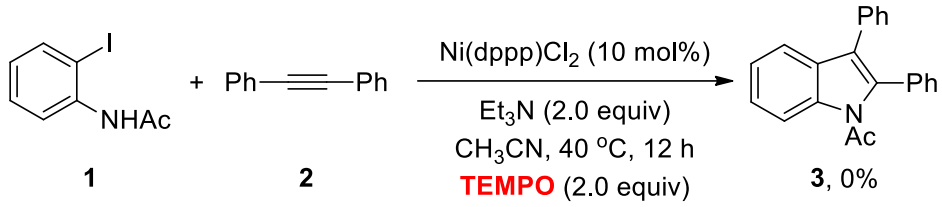
N-(2-iodophenyl)acetamide **1** (1.31 g, 5.0 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (1.34 g, 7.5 mmol, 1.5 equiv), Ni(dppp)Cl_2 (0.27 g, 0.5 mmol, 0.1 equiv), and Et_3N (1.40 mL, 10.0 mmol, 2.0 equiv) were placed in a dry 100 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (15.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h monitored with TLC. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **3** (1.27 g, 82%).



2-Iodo-*N*-(*p*-tolyl)benzamide **29** (1.35 g, 4.0 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (0.86 g, 4.8 mmol, 1.2 equiv), Ni(dppp)Cl_2 (0.22 g, 0.4 mmol, 0.1 equiv), and Et_3N (1.10 mL, 8.0 mmol, 2.0 equiv) were placed in a dry 100 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (30.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h monitored with TLC. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **30** (1.39 g, 90%).

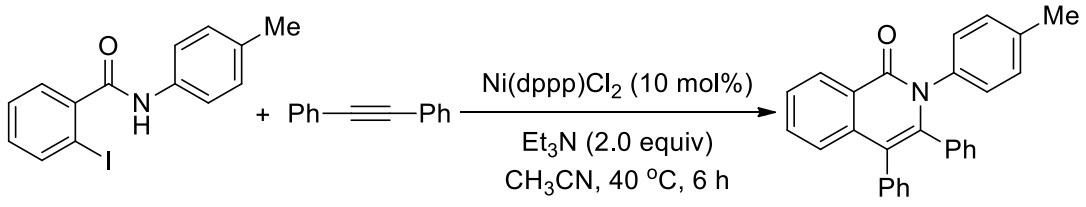
Mechanistic studies

Radical inhibition experiments:



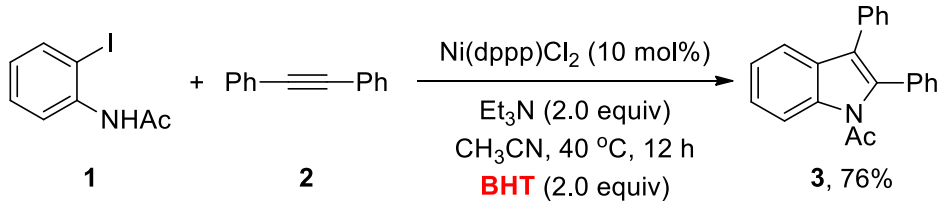
1 **2** **3**, 0%

N-(2-iodophenyl)acetamide **1** (53.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (54.3 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), Et_3N (56 μL, 0.40 mmol, 2.0 equiv), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (63.0 mg, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. The formation of **3** was completely suppressed.



29 **2** **30**, 0%

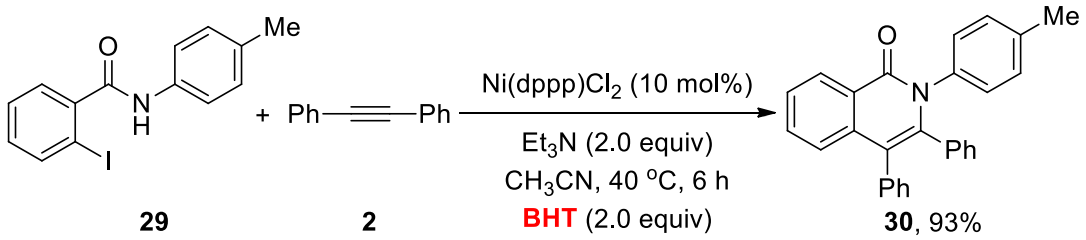
2-Iodo-*N*-(*p*-tolyl)benzamide **29** (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), Et_3N (56 μL, 0.4 mmol, 2.0 equiv), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (62.5 mg, 0.4 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. The formation of **30** was completely suppressed.



1 **2** **3**, 76%

N-(2-iodophenyl)acetamide **1** (53.1 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2**

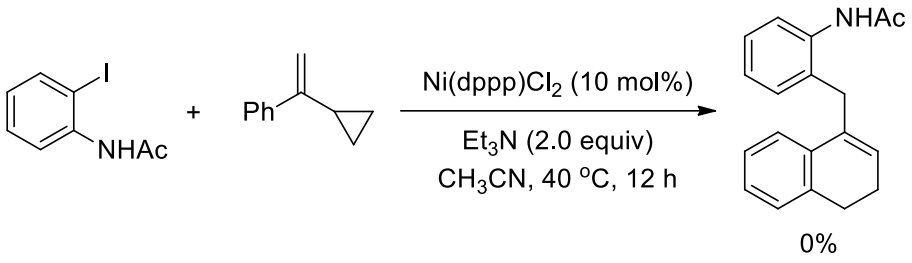
(54.0 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv), Et_3N (56 μL, 0.40 mmol, 2.0 equiv), and 2,6-di-*tert*-butyl-4-methylphenol (BHT) (88.5 mg, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **3** (47.4 mg, 76%).



29 **2** **30**, 93%

2-Iodo-*N*-(*p*-tolyl)benzamide **29** (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl_2 (10.9 mg, 0.02 mmol, 0.1 equiv), Et_3N (56 μL, 0.4 mmol, 2.0 equiv), and 2,6-di-*tert*-butyl-4-methylphenol (BHT) (88.2 mg, 0.4 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **30** (72.3 mg, 93%).

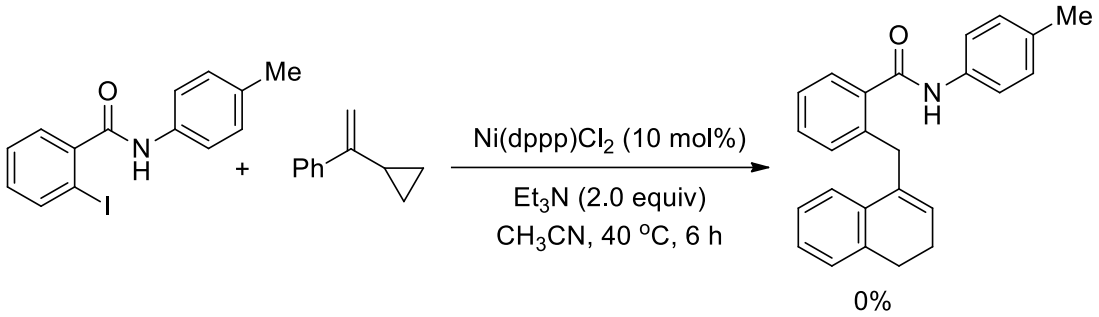
Radical clock experiments:



0%

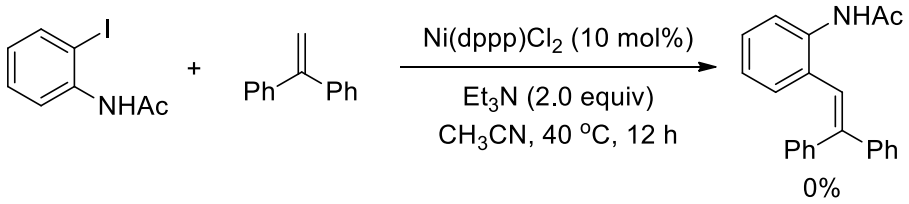
N-(2-iodophenyl)acetamide (53.1 mg, 0.20 mmol, 1.0 equiv), (1-cyclopropylvinyl)benzene (59.2 mg, 0.40 mmol, 2.0 equiv), Ni(dppp)Cl_2 (11.0 mg,

0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no ring-opening product was observed.

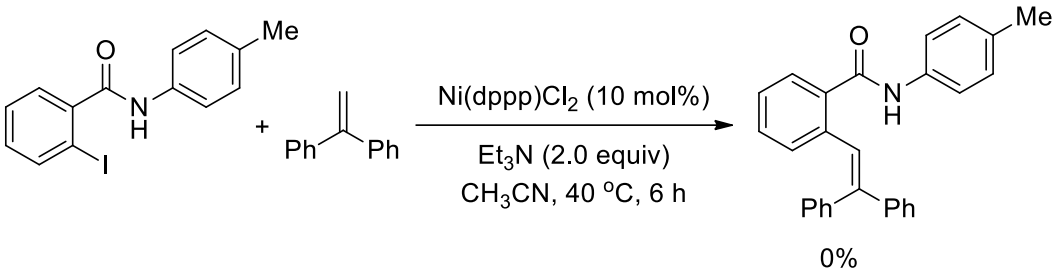


2-Iodo-*N*-(*p*-tolyl)benzamide (67.9 mg, 0.20 mmol, 1.0 equiv), (1-cyclopropylvinyl)benzene (44.9 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no ring-opening product was observed.

Radical trapping experiments:

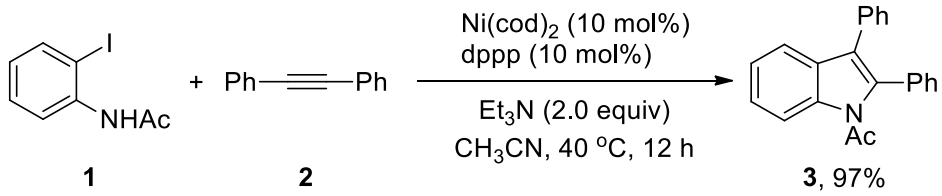


N-(2-iodophenyl)acetamide (52.8 mg, 0.20 mmol, 1.0 equiv), ethene-1,1-diyldibenzene (72.3 mg, 0.40 mmol, 2.0 equiv), Ni(dppp)Cl_2 (11.1 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no radical trapping product was observed.

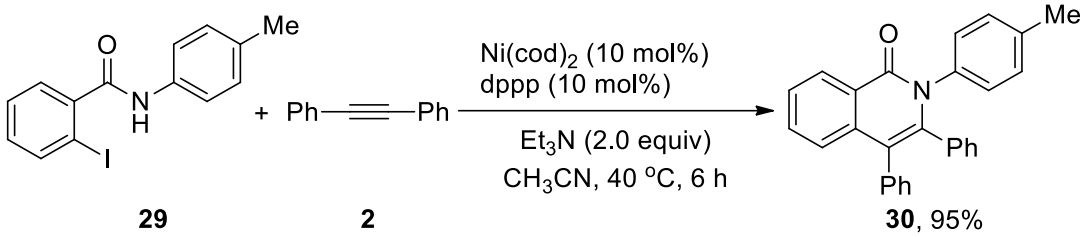


2-Iodo-*N*-(*p*-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), ethene-1,1-diyldibenzene (36.1 mg, 0.20 mmol, 1.0 equiv), Ni(dppp)Cl_2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no radical trapping product was observed.

Reactions with Ni(0) catalyst:



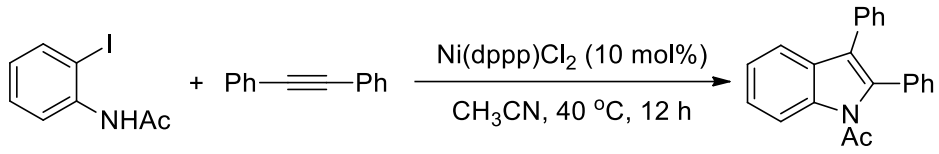
N-(2-iodophenyl)acetamide **1** (52.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (54.2 mg, 0.30 mmol, 1.5 equiv), Ni(cod)$_2$ (5.6 mg, 0.02 mmol, 0.1 equiv), dppp (12.7 mg, 0.03 mmol, 0.15 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product **3** (60.3 mg, 97%).



2-Iodo-*N*-(*p*-tolyl)benzamide **29** (67.1 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (43.6 mg, 0.24 mmol, 1.2 equiv), Ni(cod)$_2$ (5.5 mg, 0.02 mmol, 0.1 equiv), dppp

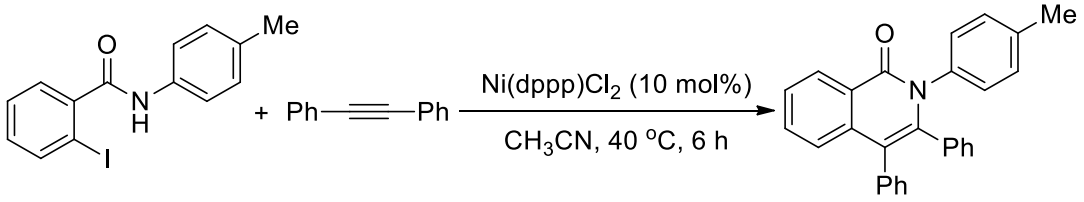
(12.4 mg, 0.03 mmol, 0.15 equiv), and Et_3N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH_2Cl_2. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product **30** (73.5 mg, 95%).

Role of Et_3N:



1 **2** **3**, 0%

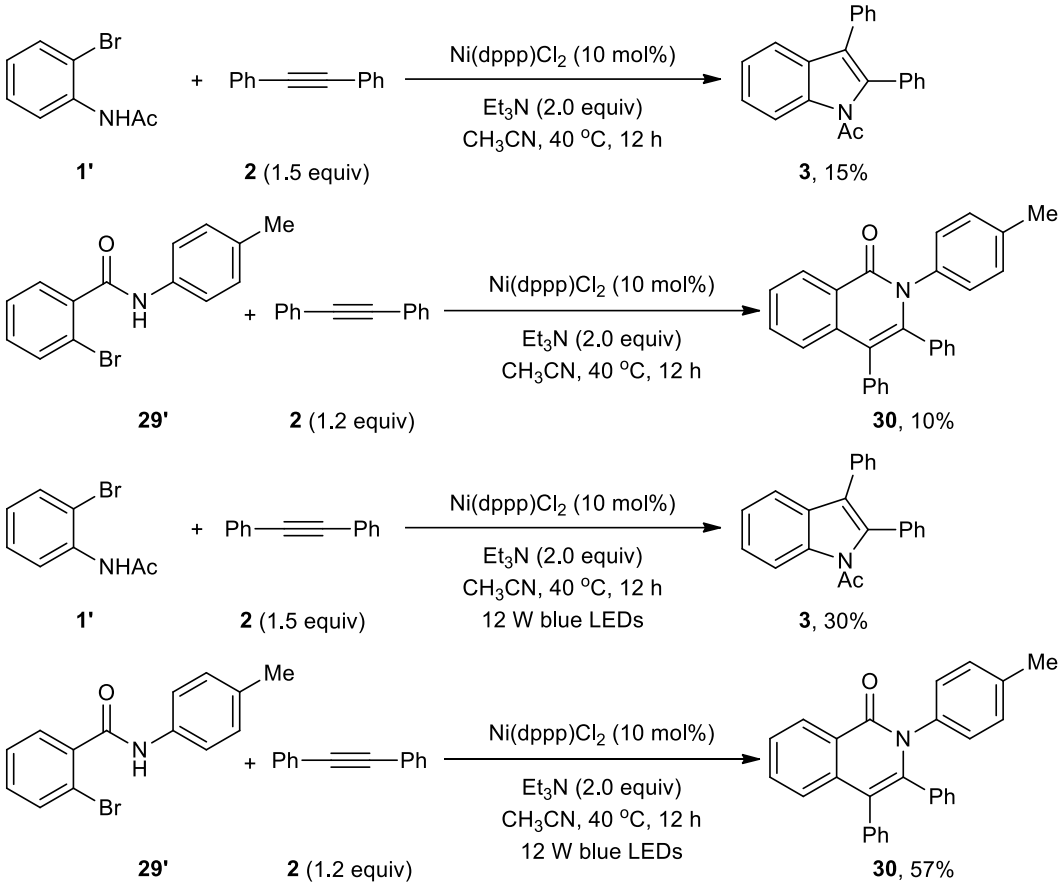
N-(2-iodophenyl)acetamide **1** (53.1 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (54.1 mg, 0.30 mmol, 1.5 equiv), and Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product **3** was observed.



29 **2** **30**, 0%

2-Iodo-*N*-(*p*-tolyl)benzamide **29** (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne **2** (42.8 mg, 0.24 mmol, 1.2 equiv), and Ni(dppp)Cl_2 (11.2 mg, 0.02 mmol, 0.1 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH_3CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no product **30** was observed.

Nickel-catalyzed Larock-type heteroannulation reactions of ortho-bromo substrates with 1,2-diphenylethyne



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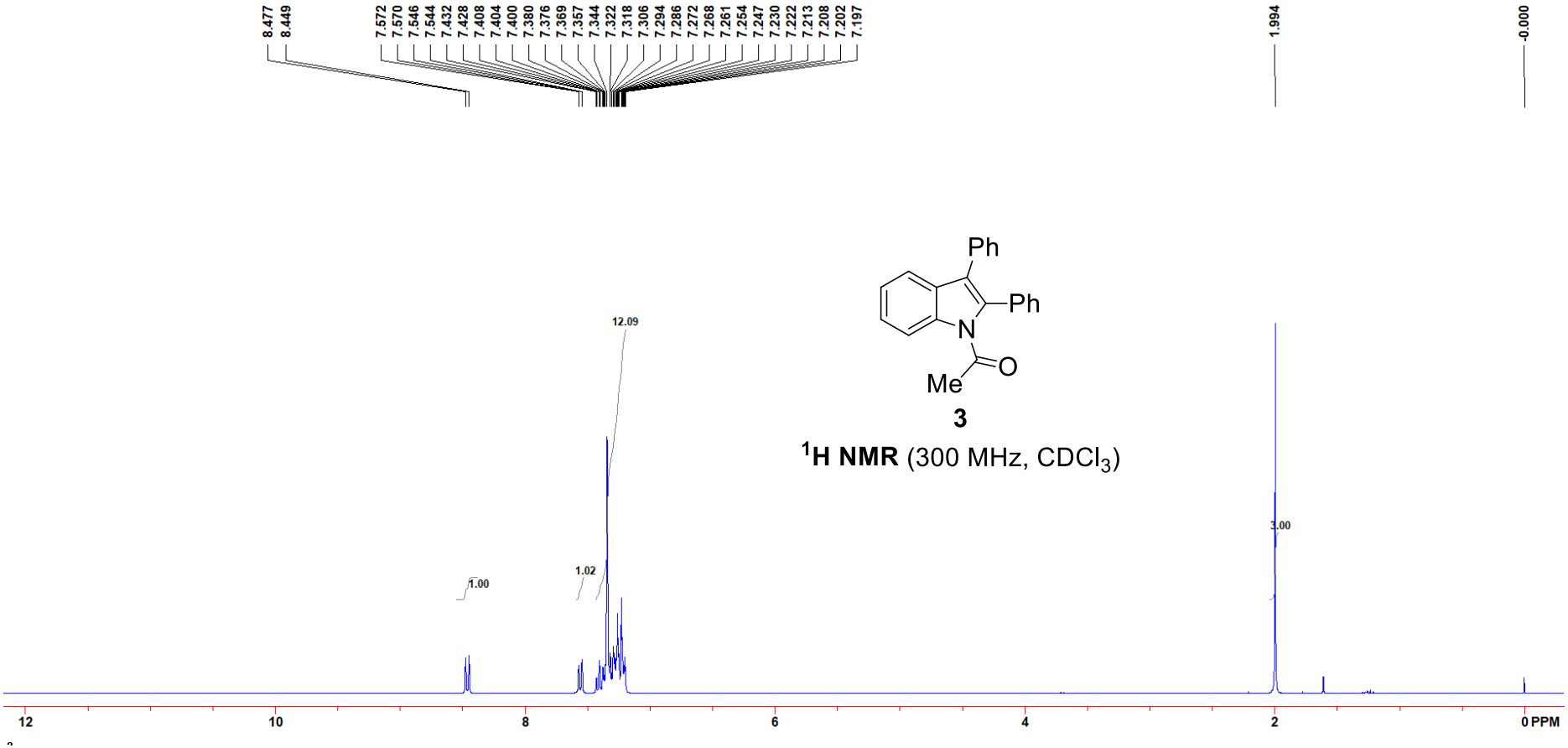
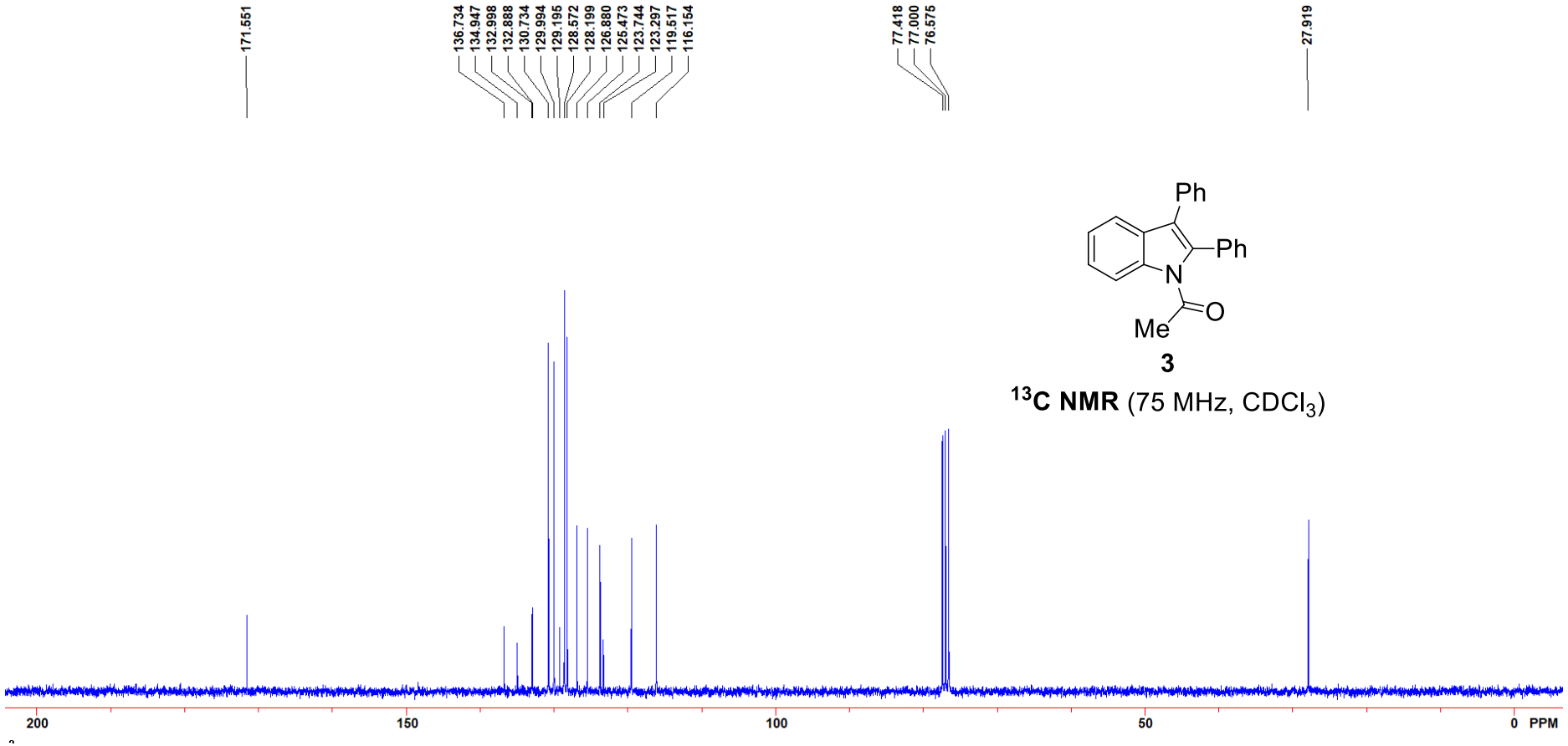


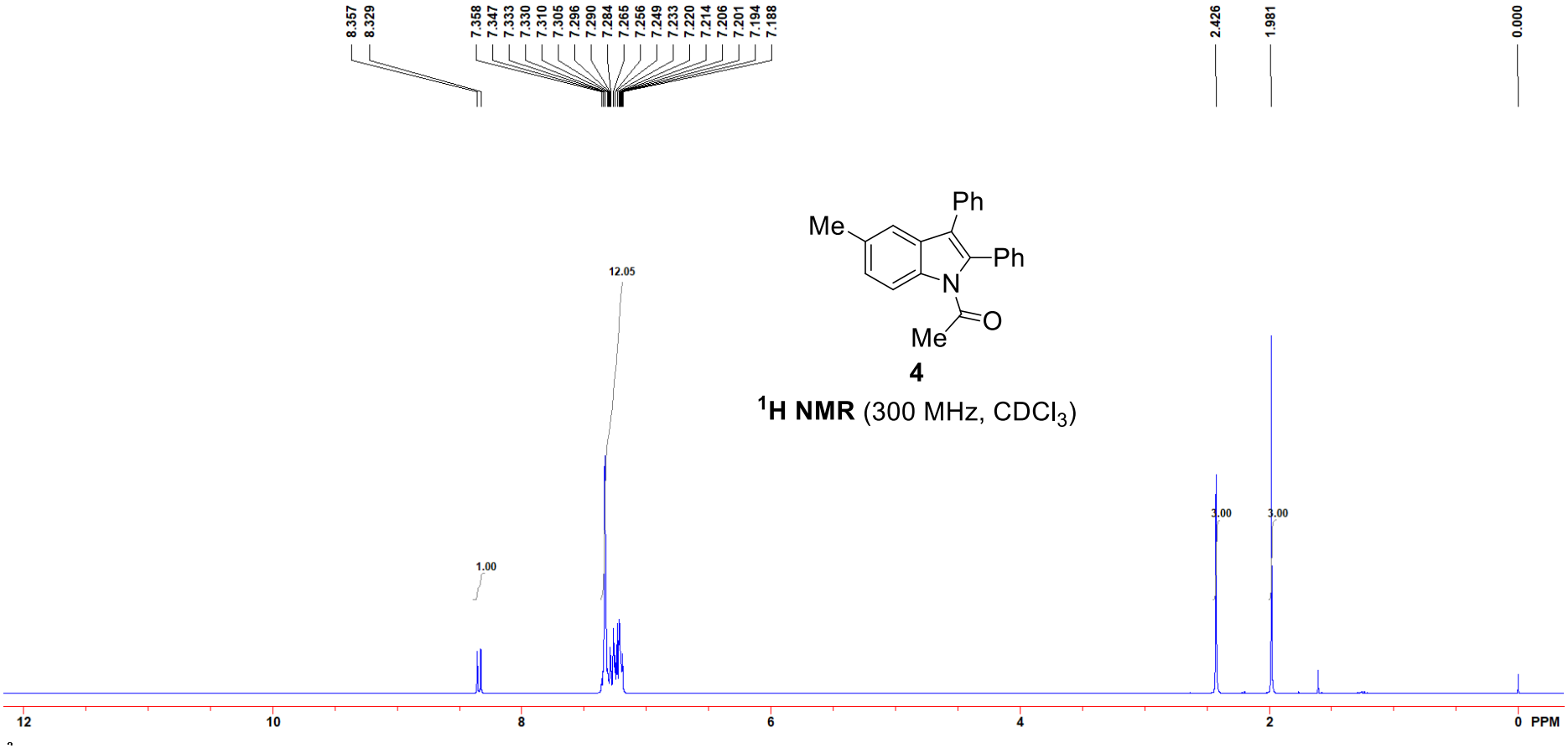
3

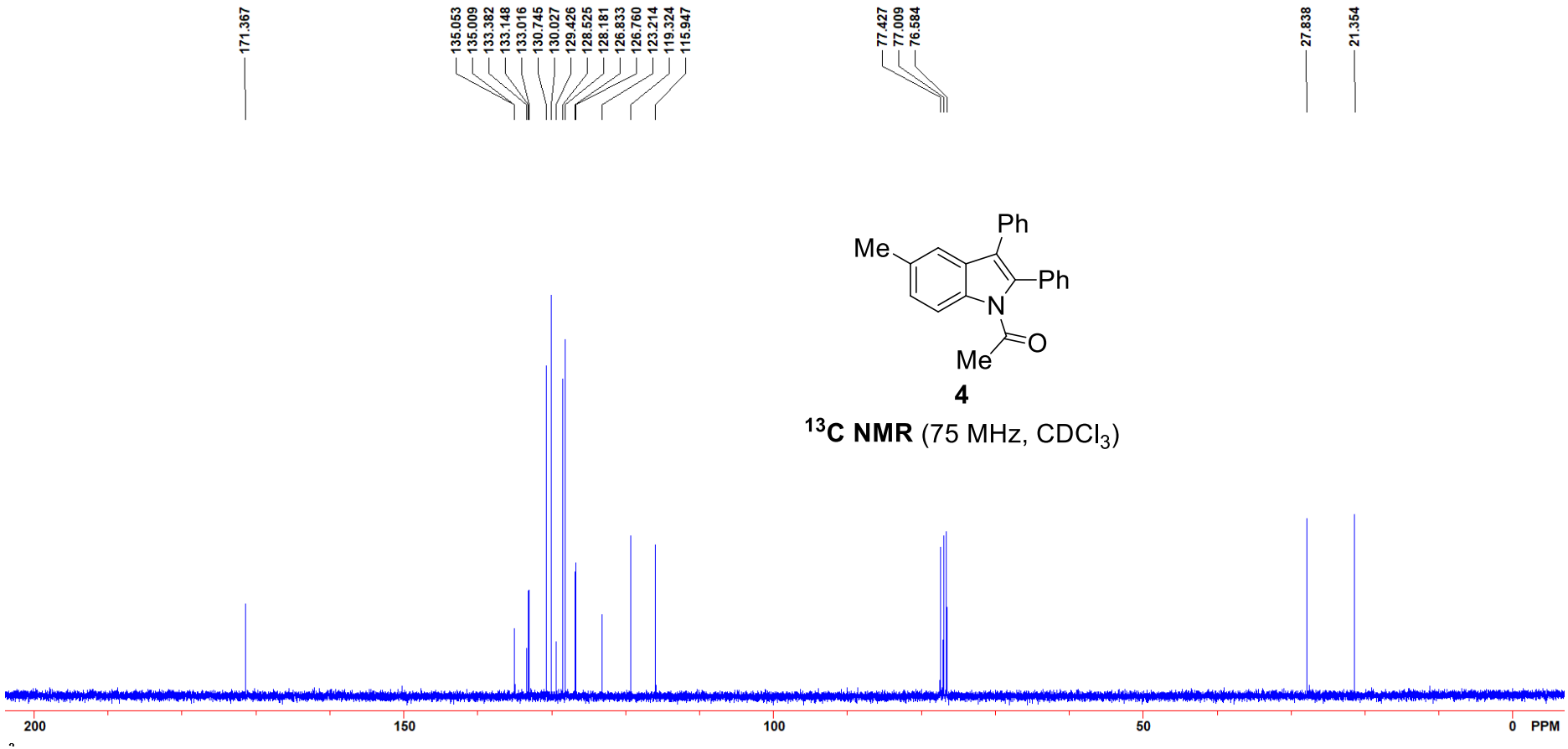
^{1}H NMR (300 MHz, $CDCl_3$)



3

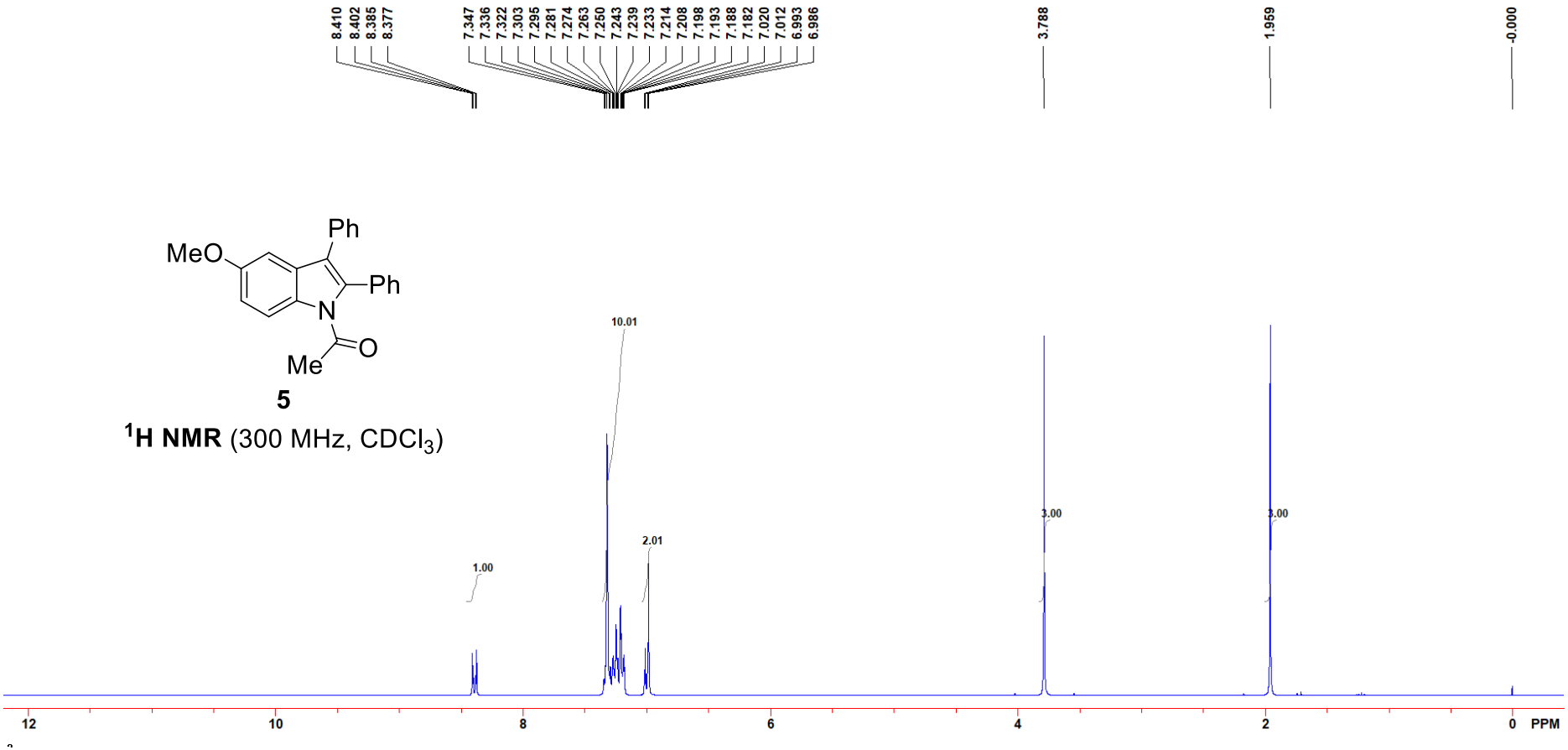
^{13}C NMR (75 MHz, $CDCl_3$)

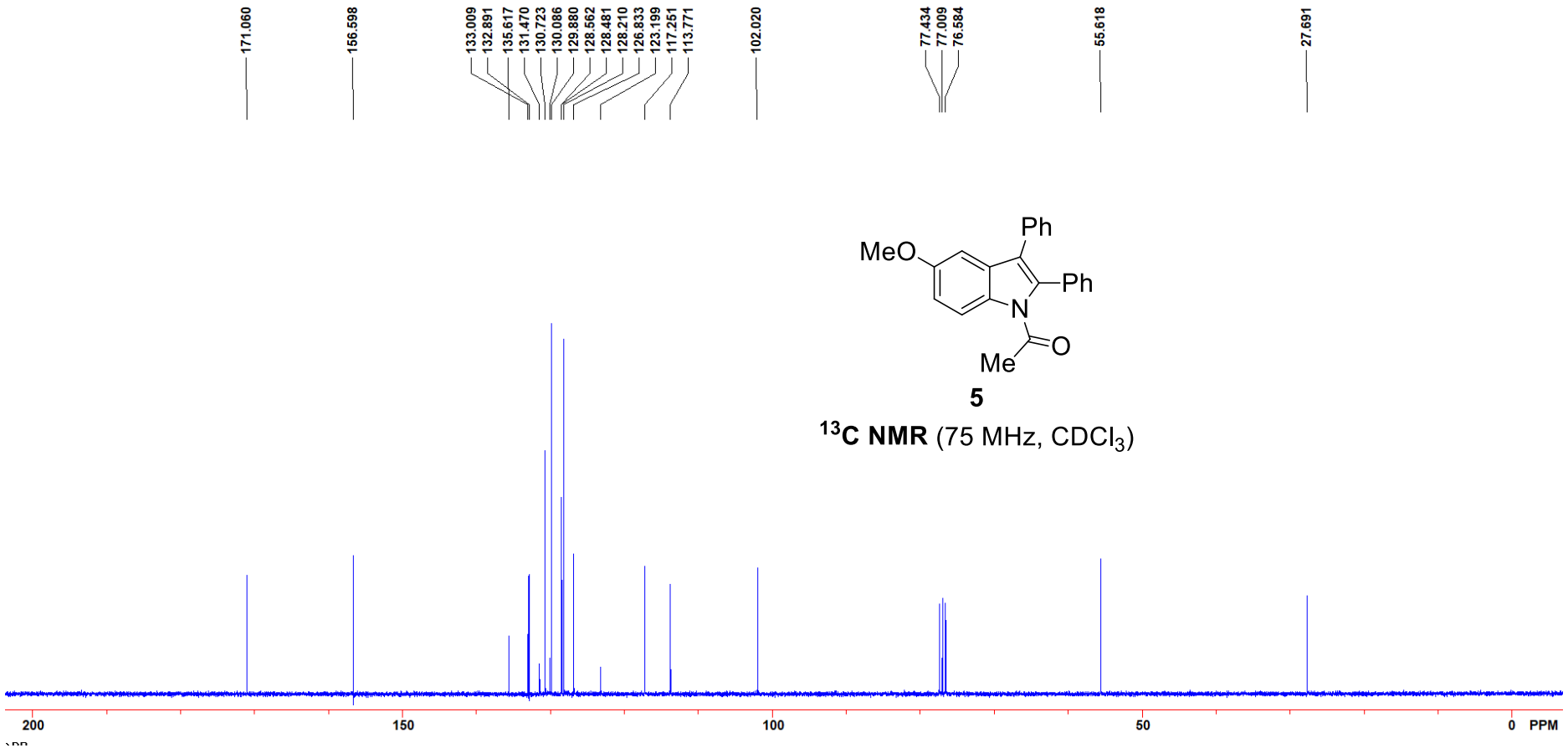

8.357
8.329
7.358
7.347
7.333
7.330
7.310
7.305
7.296
7.290
7.284
7.265
7.256
7.249
7.233
7.220
7.214
7.206
7.201
7.194
7.188
2.426
1.981
0.000
Ph
Me
Ph
N
O
Me
4
1H NMR (300 MHz, CDCl3)
12.05
1.00
3.00
3.00
12
10
8
6
4
2
0 PPM



4

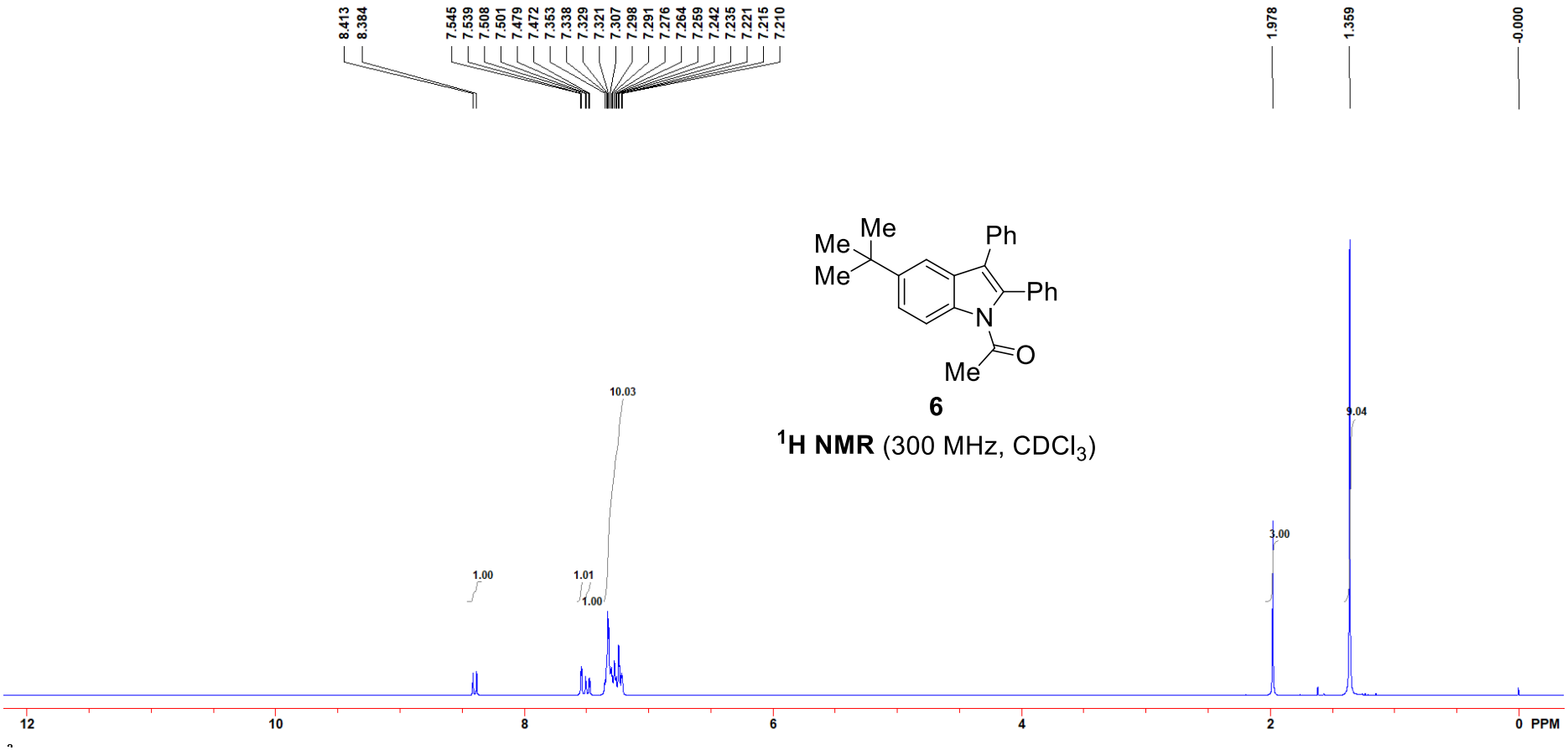
^{13}C NMR (75 MHz, $CDCl_3$)

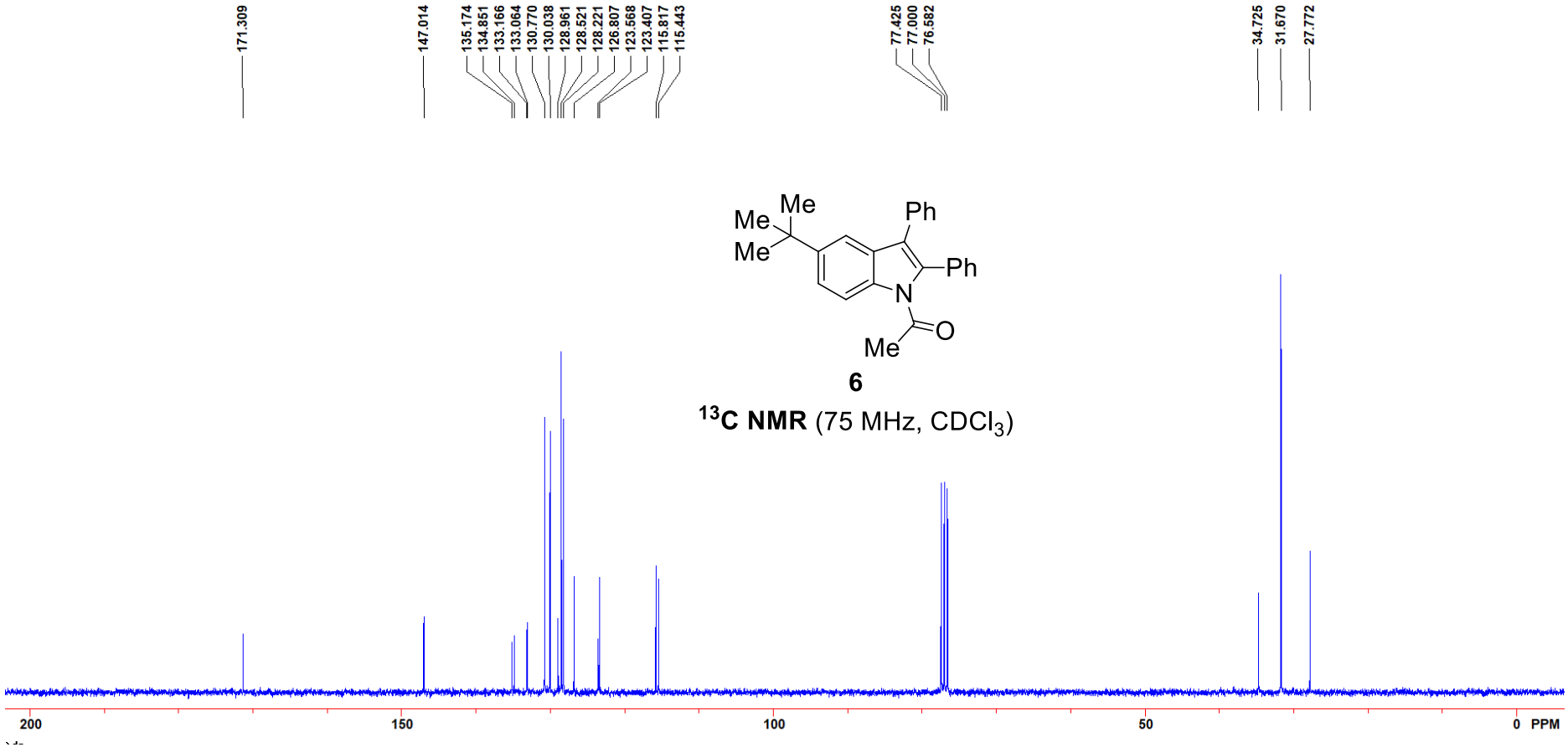

8.410
8.402
8.385
8.377
7.347
7.336
7.322
7.303
7.295
7.281
7.274
7.263
7.250
7.243
7.239
7.233
7.214
7.208
7.198
7.193
7.188
7.182
7.020
7.012
6.993
6.986
3.788
1.959
-0.000
MeO
Ph
Ph
N
O
Me
5
1H NMR (300 MHz, CDCl3)
10.01
1.00
2.01
3.00
3.00
12
10
8
6
4
2
0
PPM



5

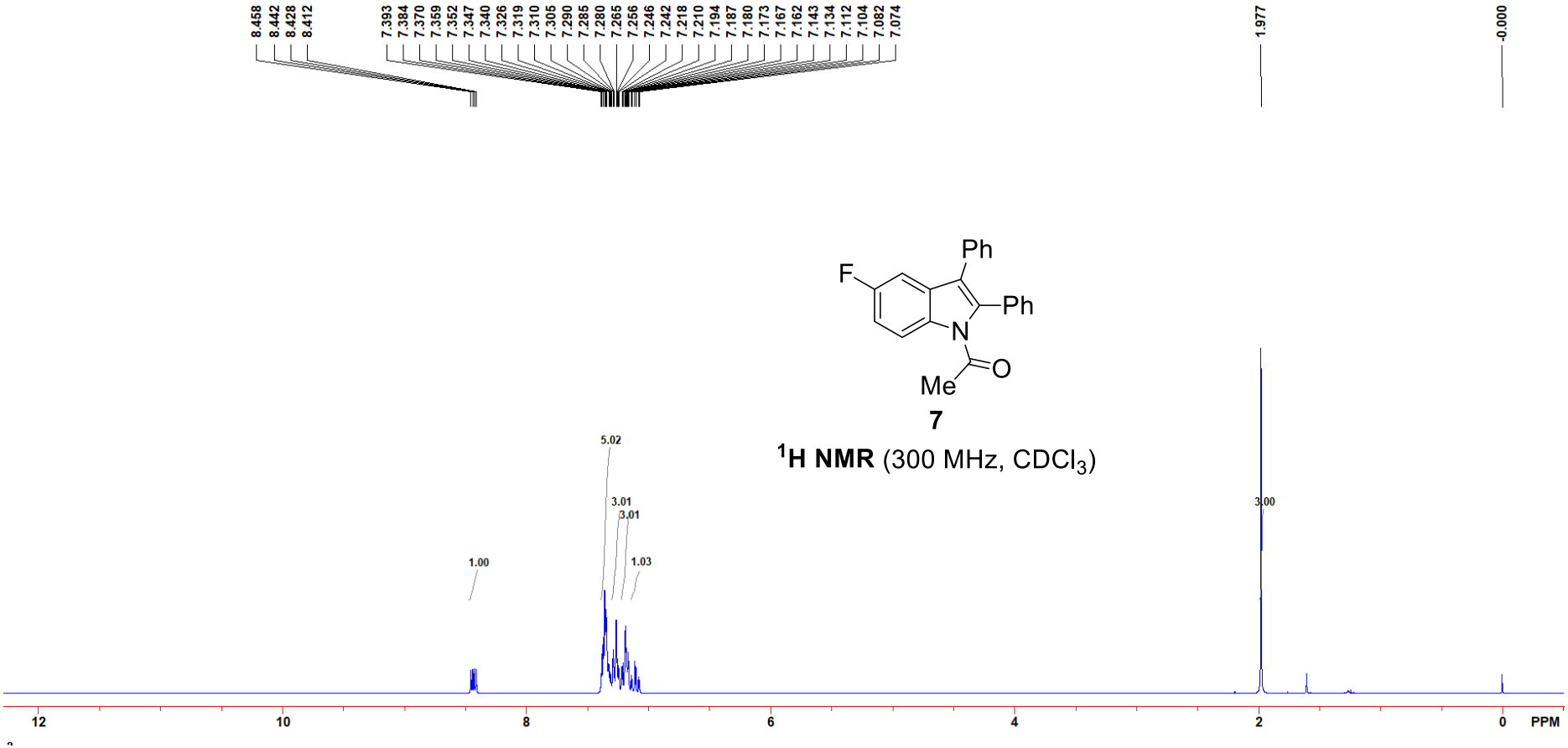
^{13}C NMR (75 MHz, $CDCl_3$)

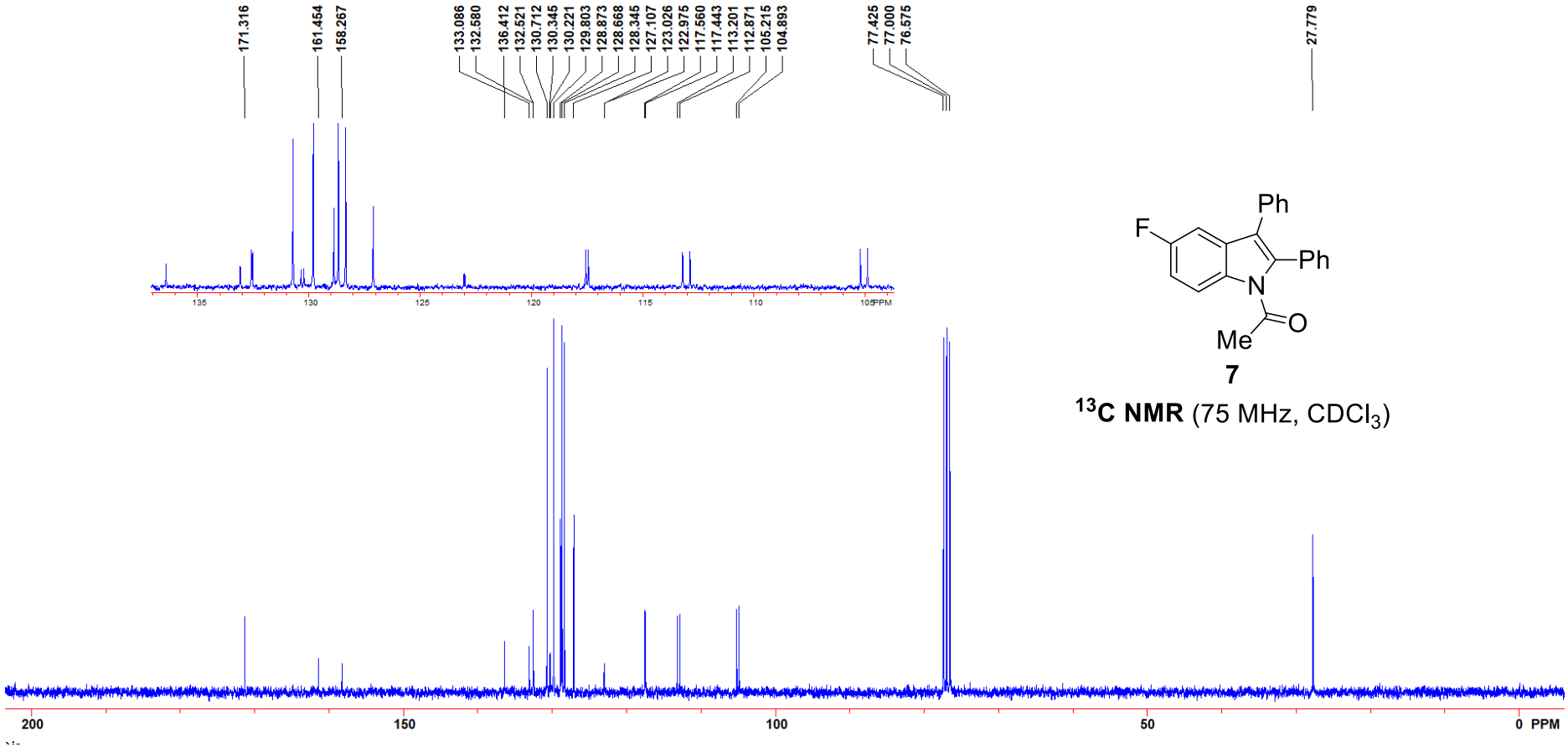

8.413
8.384
7.545
7.539
7.508
7.501
7.479
7.472
7.353
7.338
7.329
7.321
7.307
7.298
7.291
7.276
7.264
7.259
7.242
7.235
7.221
7.215
7.210
1.978
1.359
-0.000
Me
Me
Me
Ph
Ph
N
O
Me
6
1H NMR (300 MHz, CDCl3)
10.03
1.00
1.01
1.00
3.00
9.04
12
10
8
6
4
2
0 PPM



6

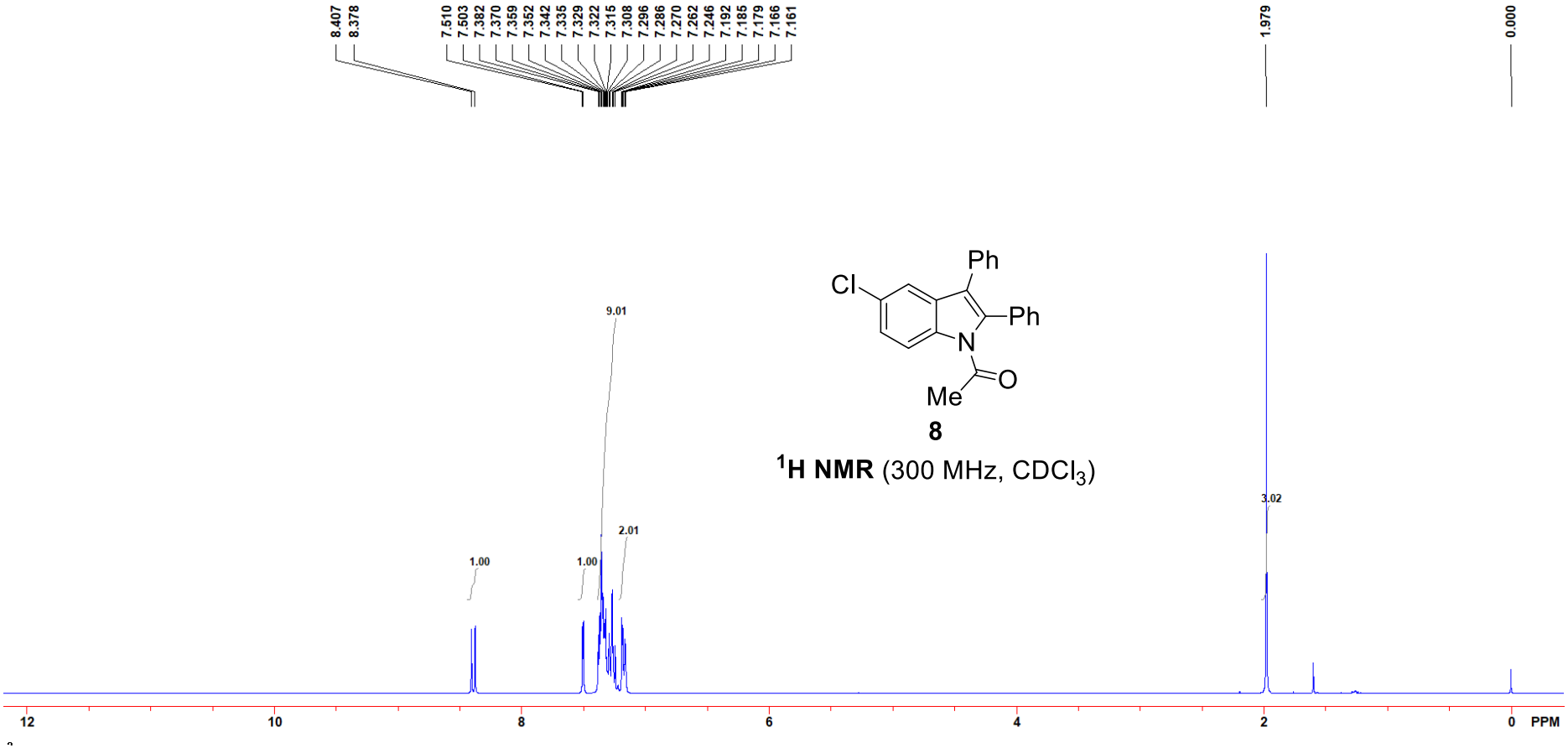
^{13}C NMR (75 MHz, $CDCl_3$)

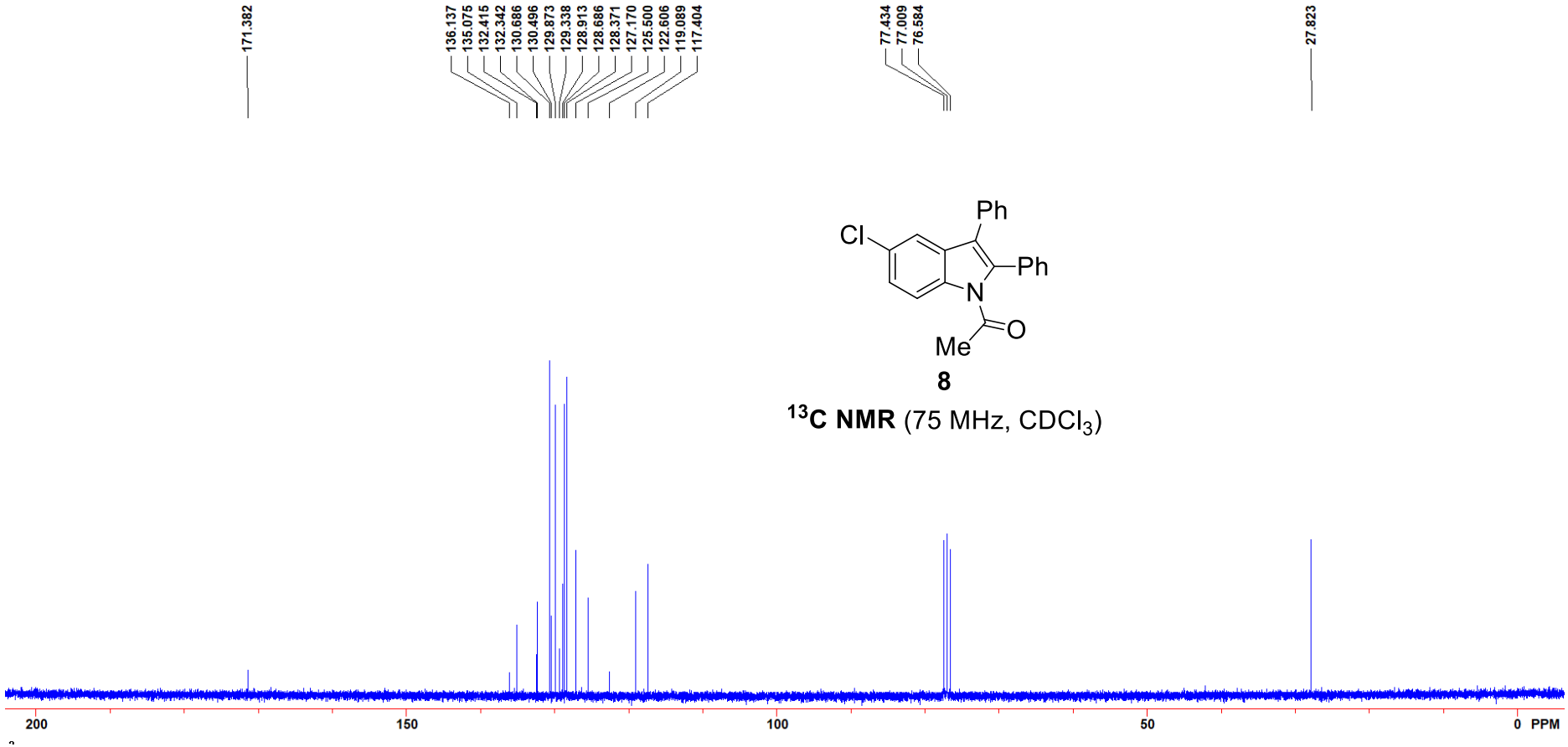

8.458
8.442
8.428
8.412
7.393
7.384
7.370
7.359
7.352
7.347
7.340
7.326
7.319
7.310
7.305
7.290
7.285
7.280
7.265
7.256
7.246
7.242
7.218
7.210
7.194
7.187
7.180
7.173
7.167
7.162
7.143
7.134
7.112
7.104
7.082
7.074
1.977
-0.000
F
Ph
Ph
N
O
Me
7
1H NMR (300 MHz, CDCl3)
5.02
3.01
3.01
1.03
1.00
3.00
12
10
8
6
4
2
0
PPM



7

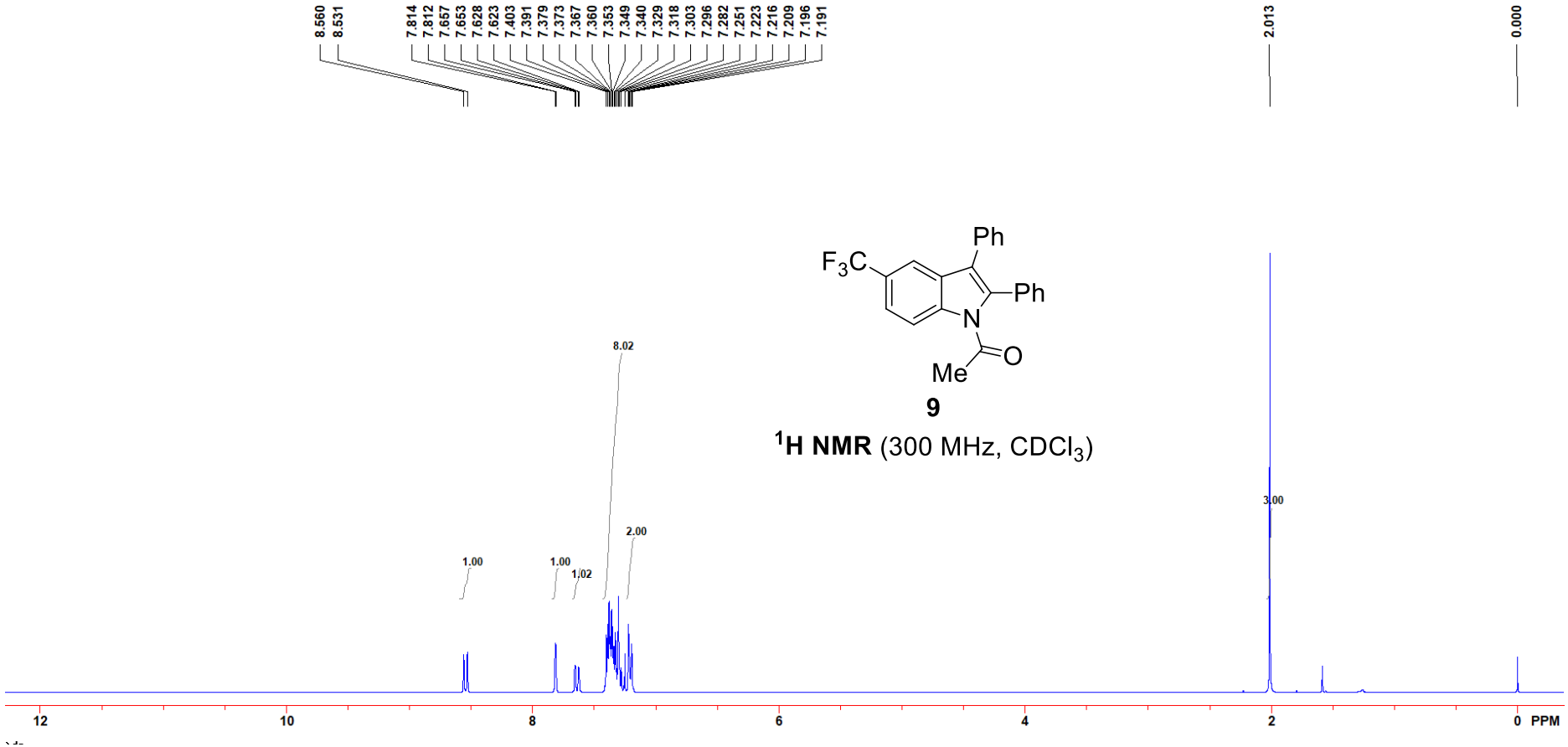
^{13}C NMR (75 MHz, $CDCl_3$)

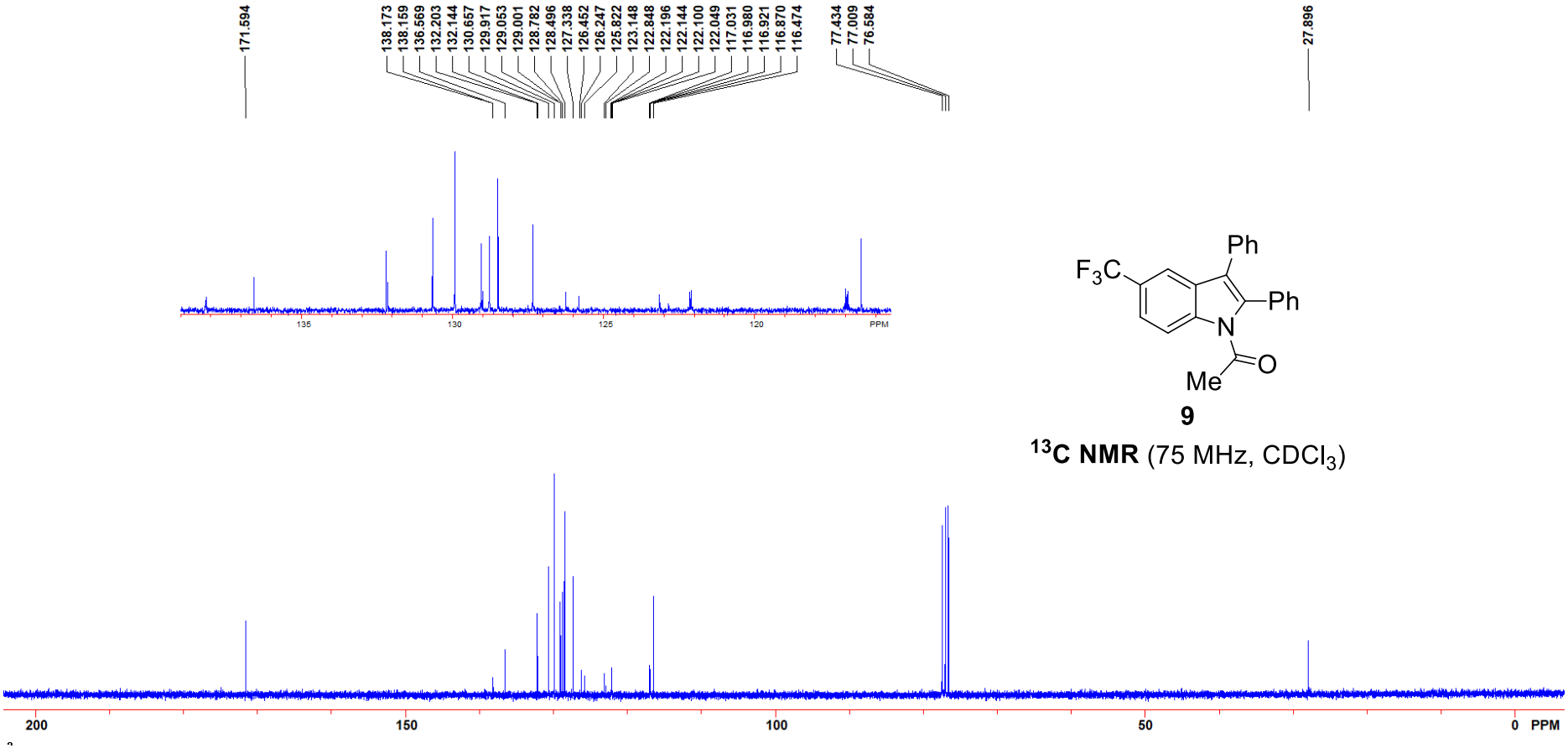

8.407
8.378
7.510
7.503
7.382
7.370
7.359
7.352
7.342
7.335
7.329
7.322
7.315
7.308
7.296
7.286
7.270
7.262
7.246
7.192
7.185
7.179
7.166
7.161
1.979
0.000
Cl
Ph
Ph
N
O
Me
8
1H NMR (300 MHz, CDCl3)
9.01
2.01
1.00
1.00
3.02
12
10
8
6
4
2
0
PPM



8

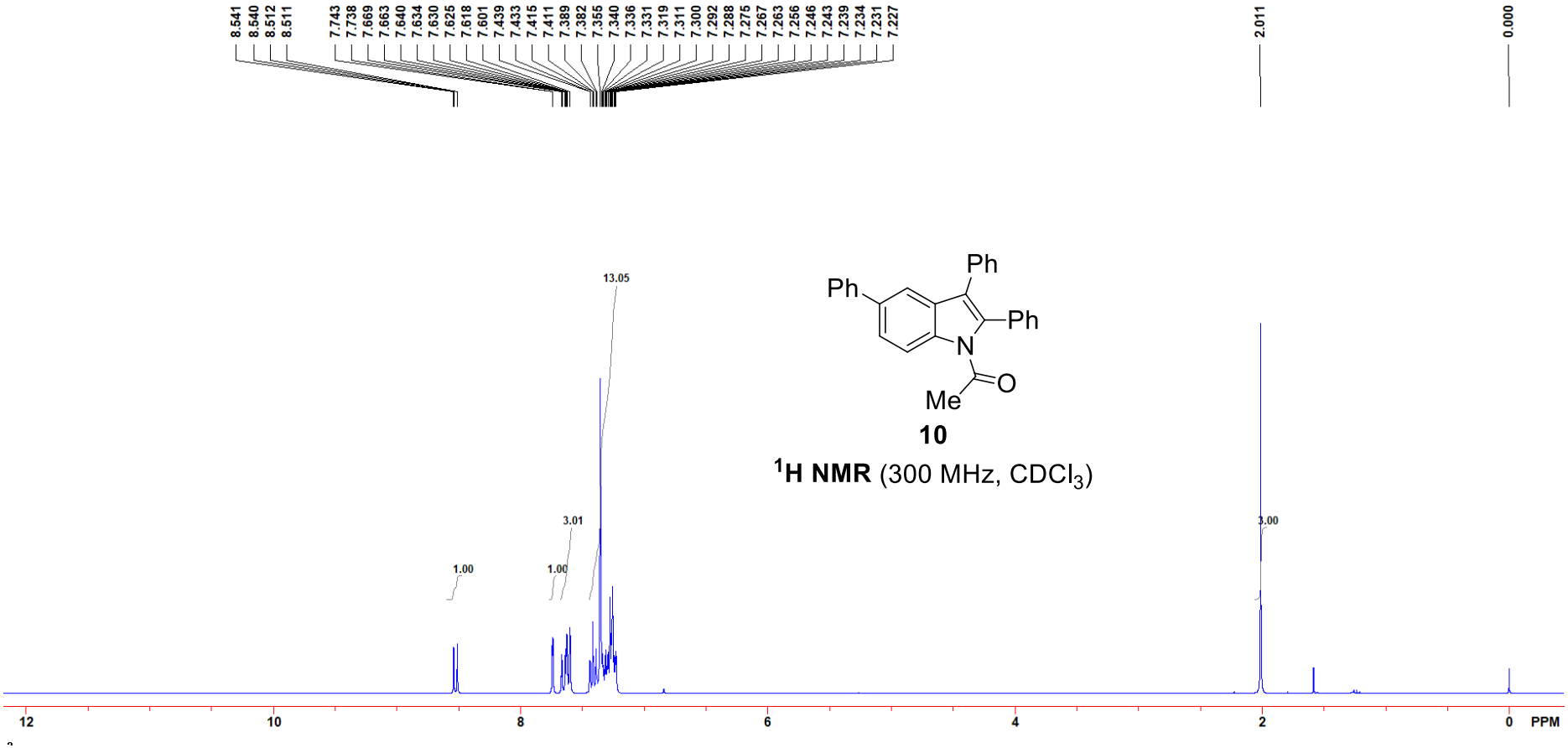
^{13}C NMR (75 MHz, $CDCl_3$)


8.560
8.531
7.814
7.812
7.657
7.653
7.628
7.623
7.403
7.391
7.379
7.373
7.367
7.360
7.353
7.349
7.340
7.329
7.318
7.303
7.296
7.282
7.251
7.223
7.216
7.209
7.196
7.191
2.013
0.000
Ph
F3C
Ph
N
Me
O
9
1H NMR (300 MHz, CDCl3)
8.02
2.00
1.00
1.00
1.02
3.00
12
10
8
6
4
2
0 PPM



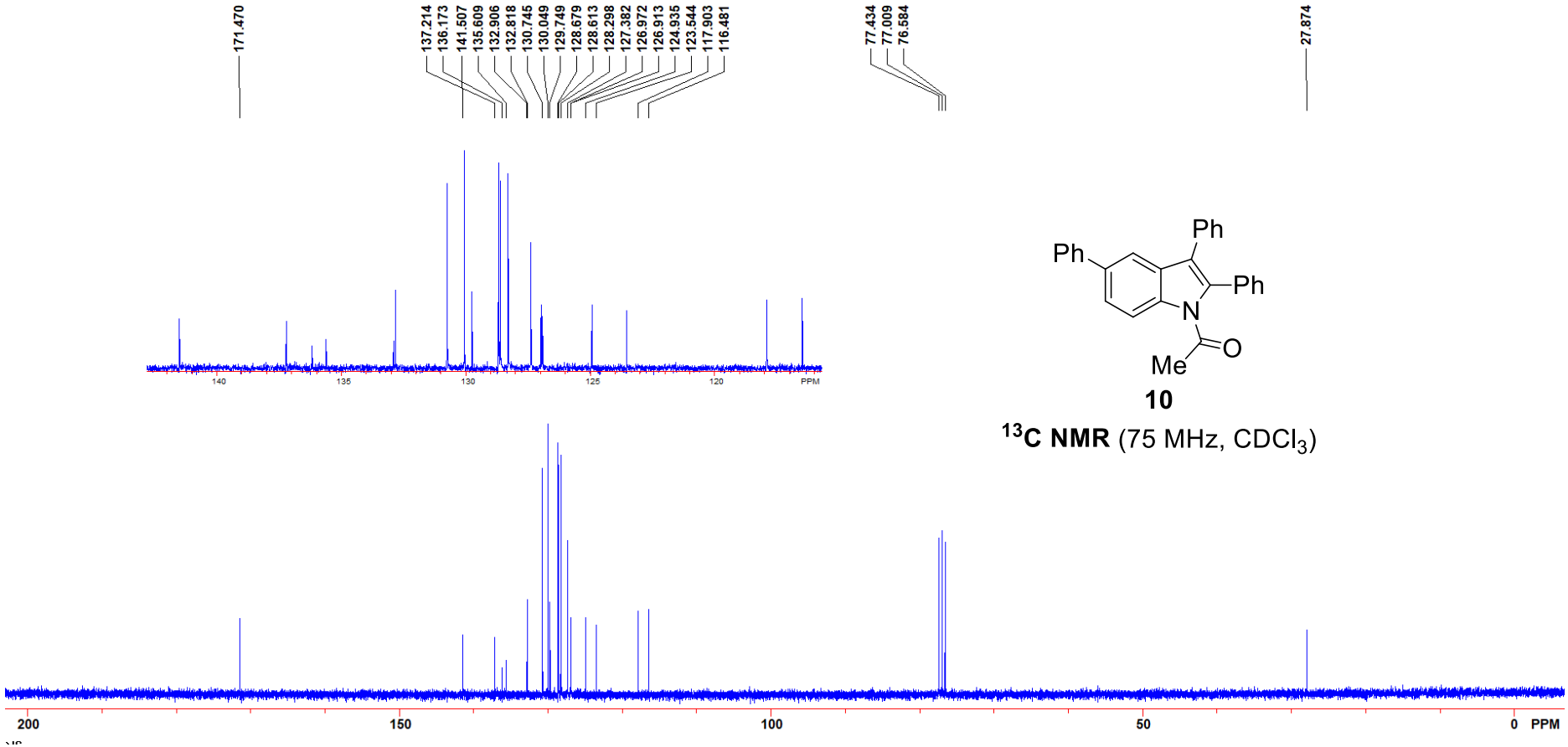
9

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



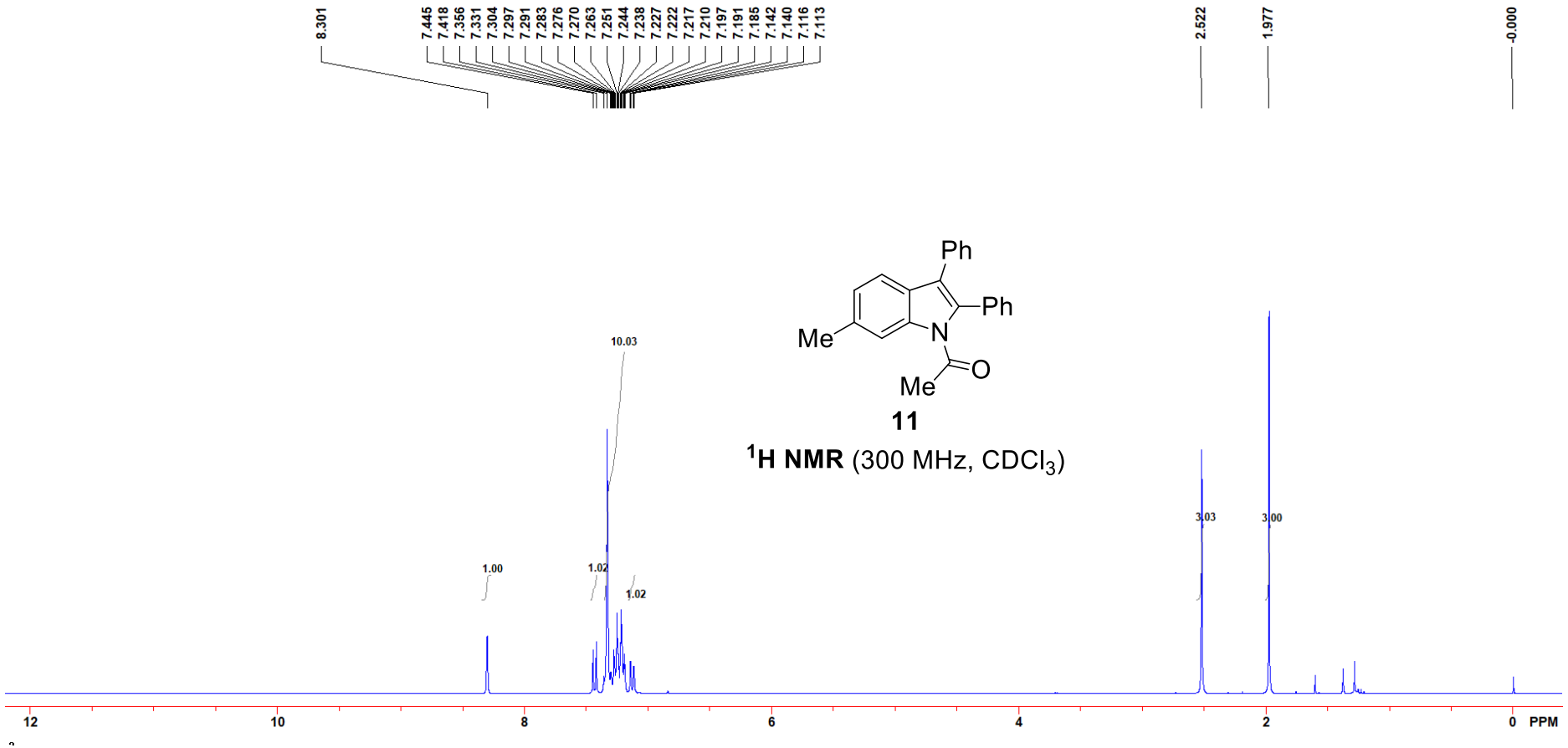
10

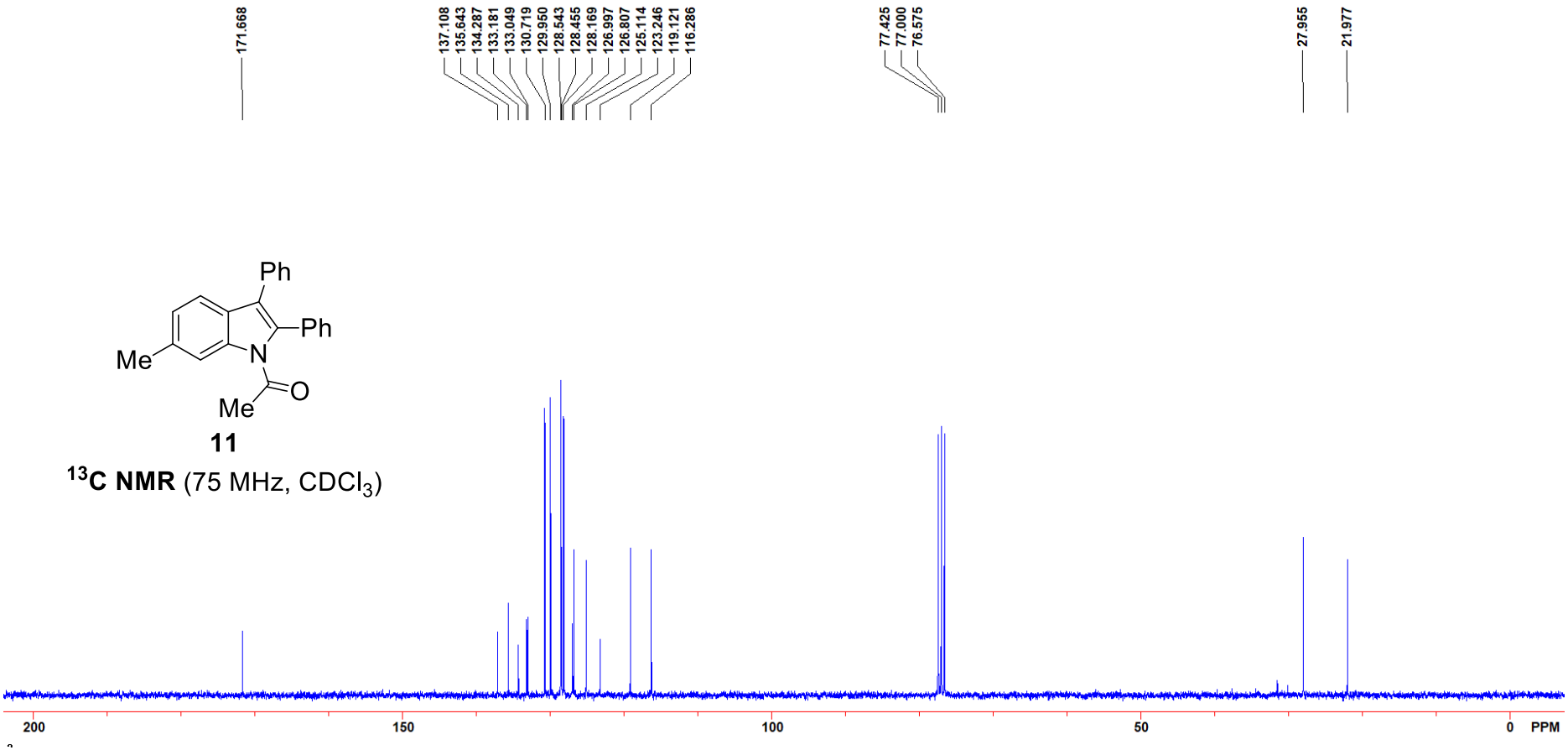
^{1}H NMR (300 MHz, $CDCl_3$)



10

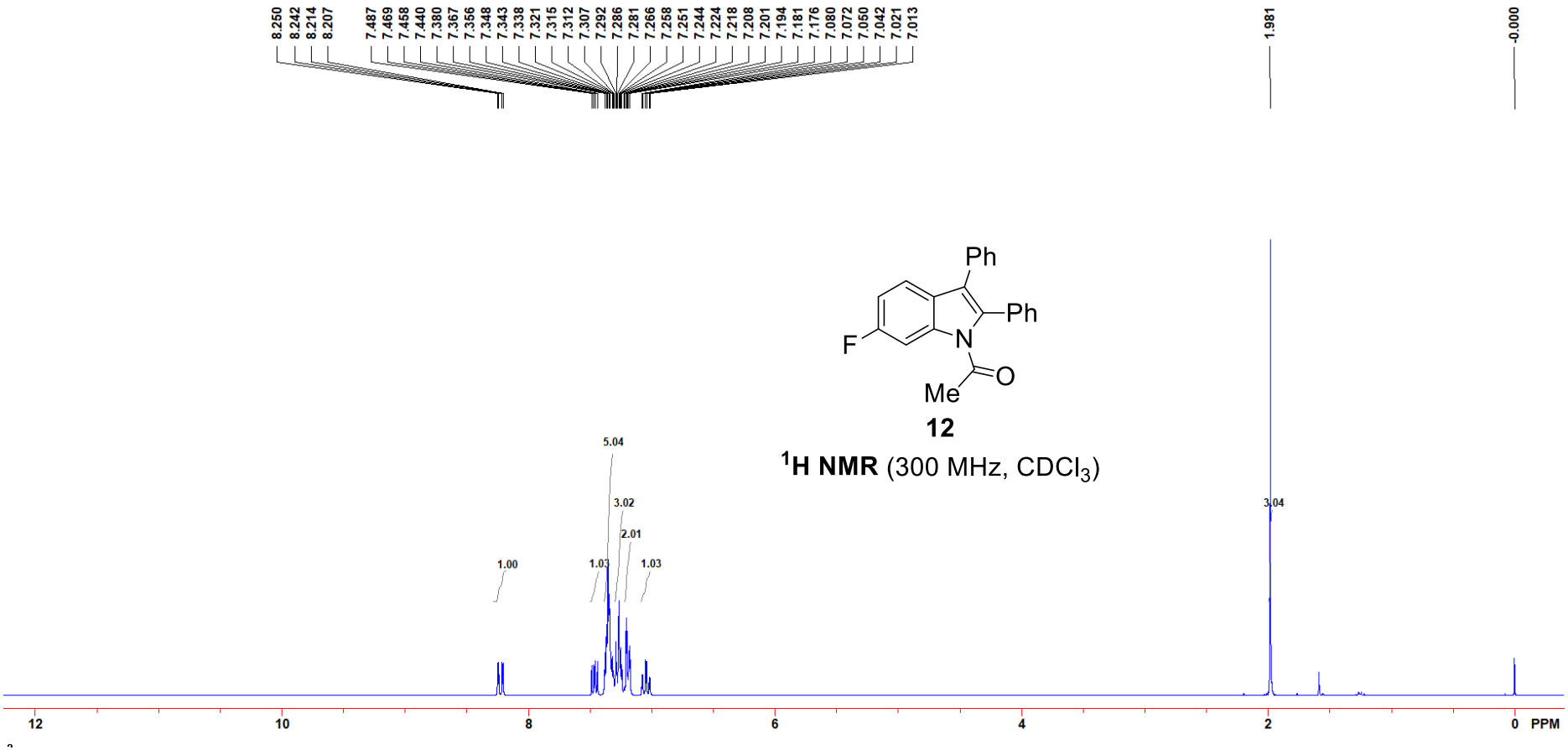
^{13}C NMR (75 MHz, $CDCl_3$)

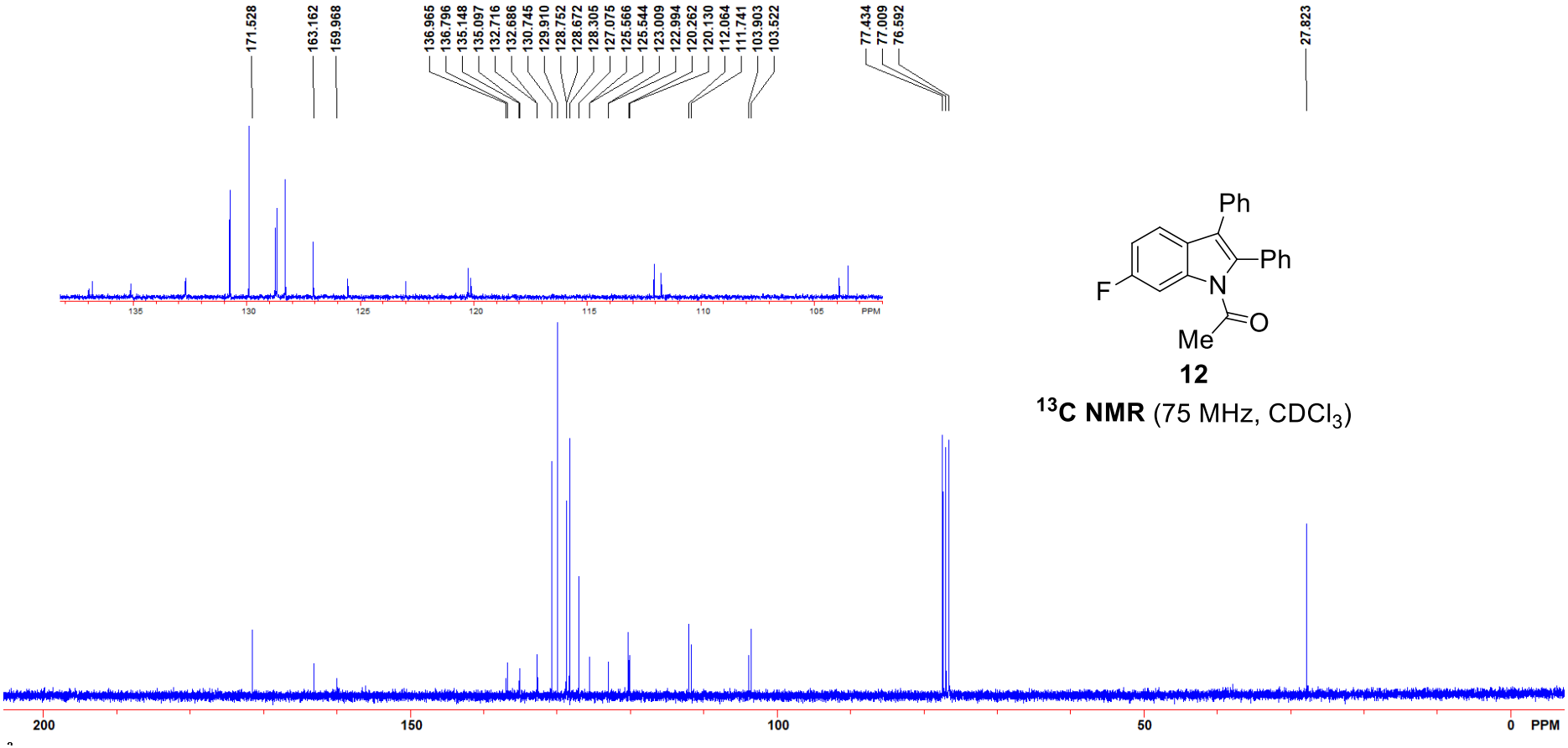

8.301
7.445
7.418
7.356
7.331
7.304
7.297
7.291
7.283
7.276
7.270
7.263
7.251
7.244
7.238
7.227
7.222
7.217
7.210
7.197
7.191
7.185
7.142
7.140
7.116
7.113
2.522
1.977
-0.000
Ph
Ph
Me
N
O
Me
11
1H NMR (300 MHz, CDCl3)
10.03
1.00
1.02
1.02
3.03
3.00
12
10
8
6
4
2
0
PPM



11

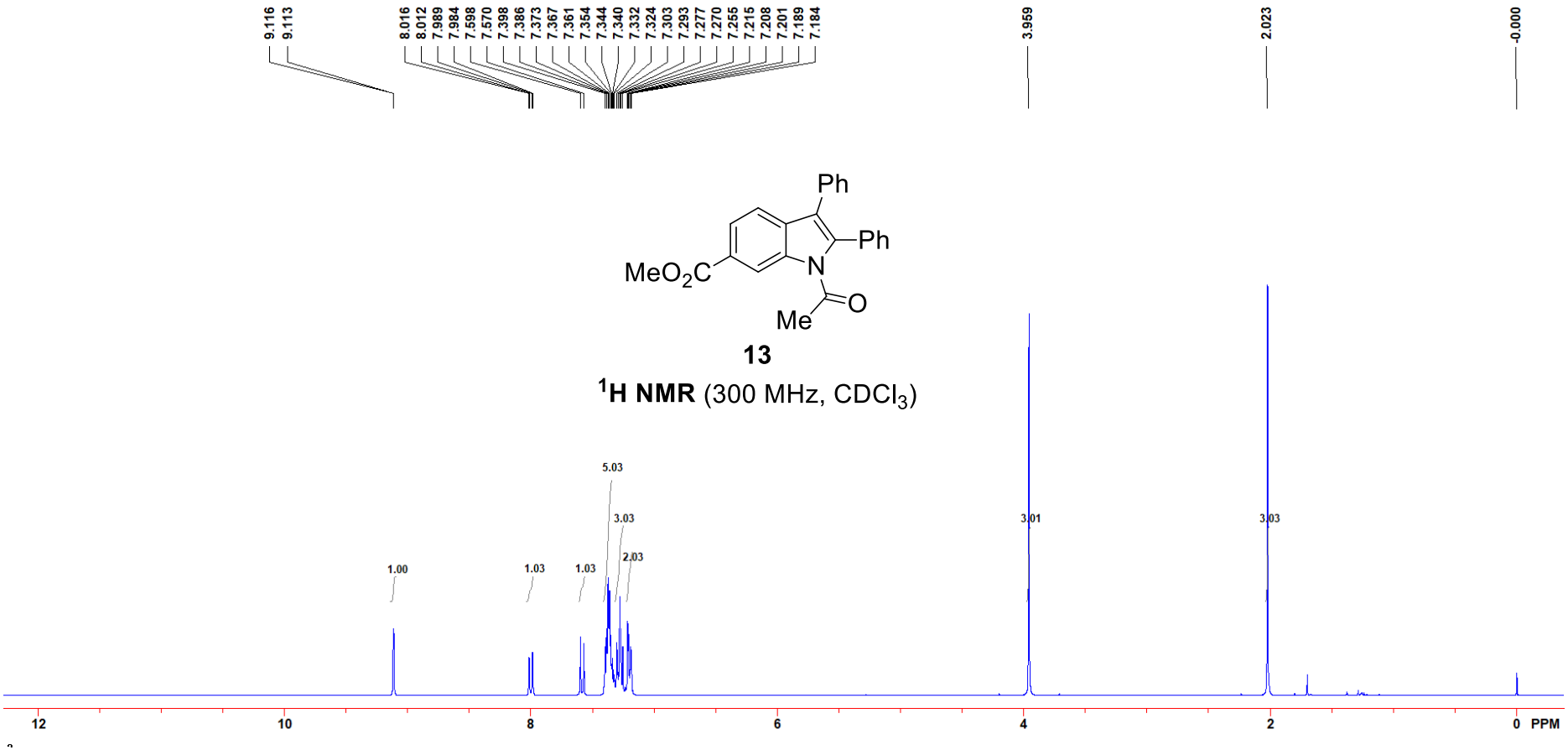
^{13}C NMR (75 MHz, $CDCl_3$)

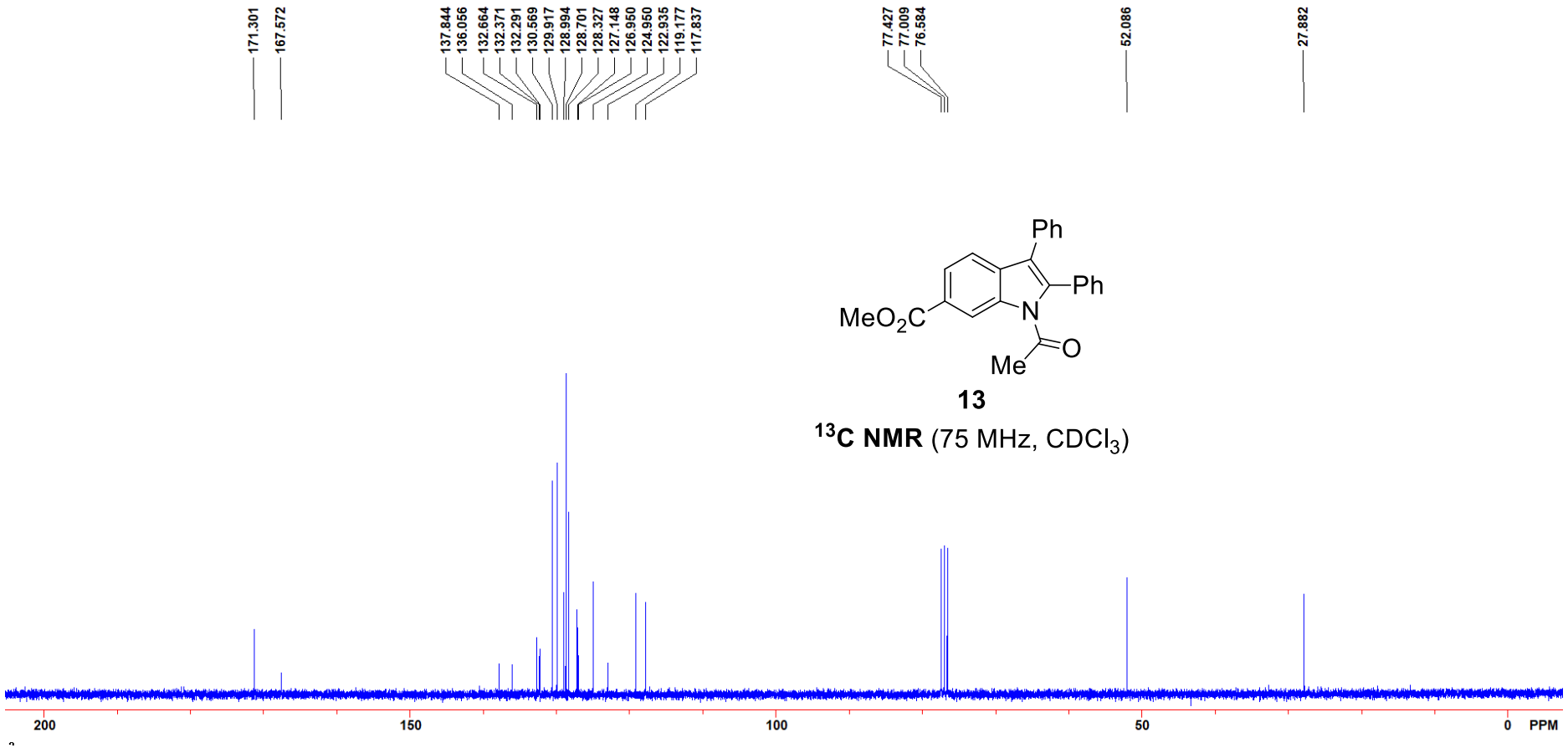

8.250
8.242
8.214
8.207
7.487
7.469
7.458
7.440
7.380
7.367
7.356
7.348
7.343
7.338
7.321
7.315
7.312
7.307
7.292
7.286
7.281
7.266
7.258
7.251
7.244
7.224
7.218
7.208
7.201
7.194
7.181
7.176
7.080
7.072
7.050
7.042
7.021
7.013
1.981
-0.000
Ph
Ph
F
N
O
Me
12
1H NMR (300 MHz, CDCl3)
5.04
3.02
2.01
1.00
1.03
1.03
3.04
12
10
8
6
4
2
0 PPM



12

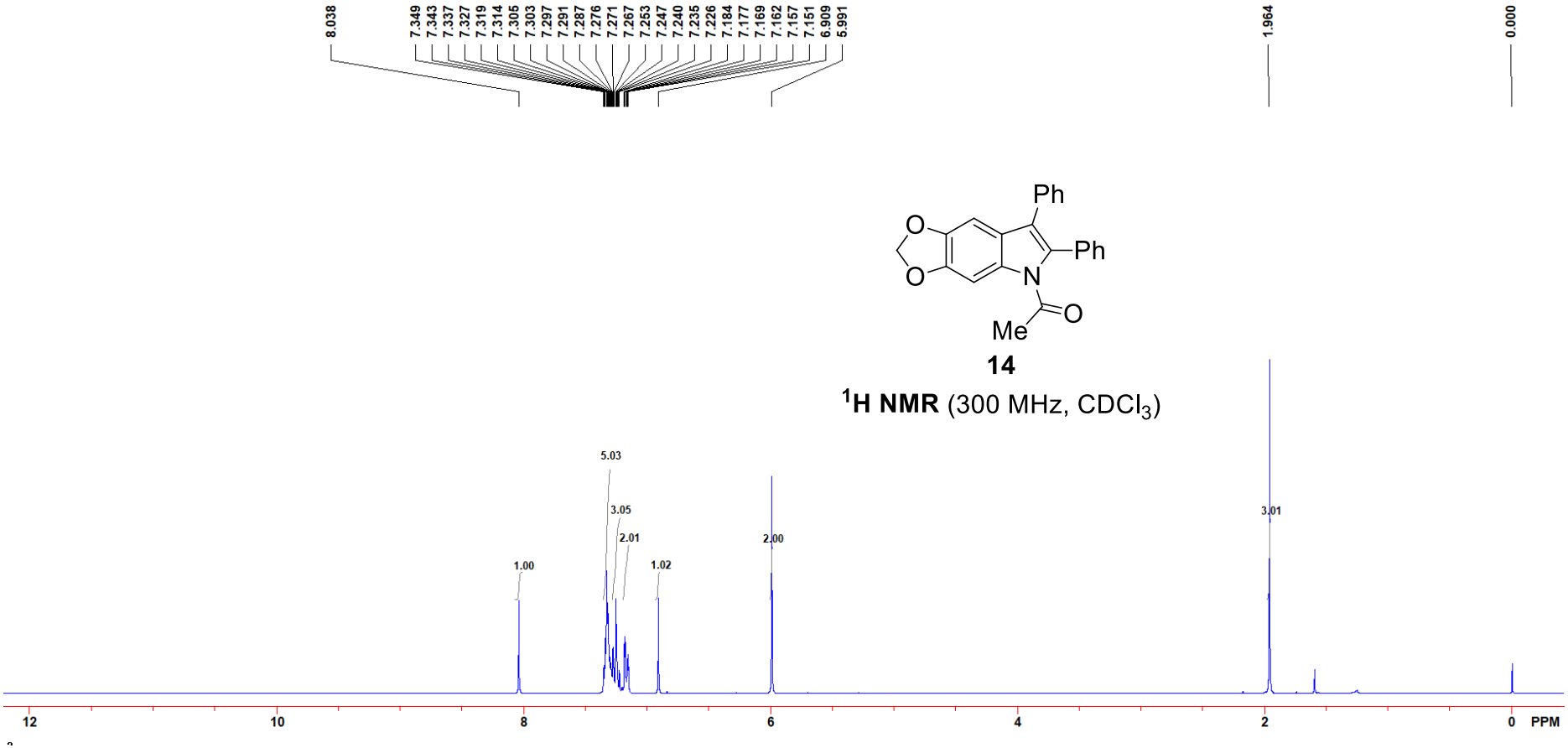
^{13}C NMR (75 MHz, $CDCl_3$)

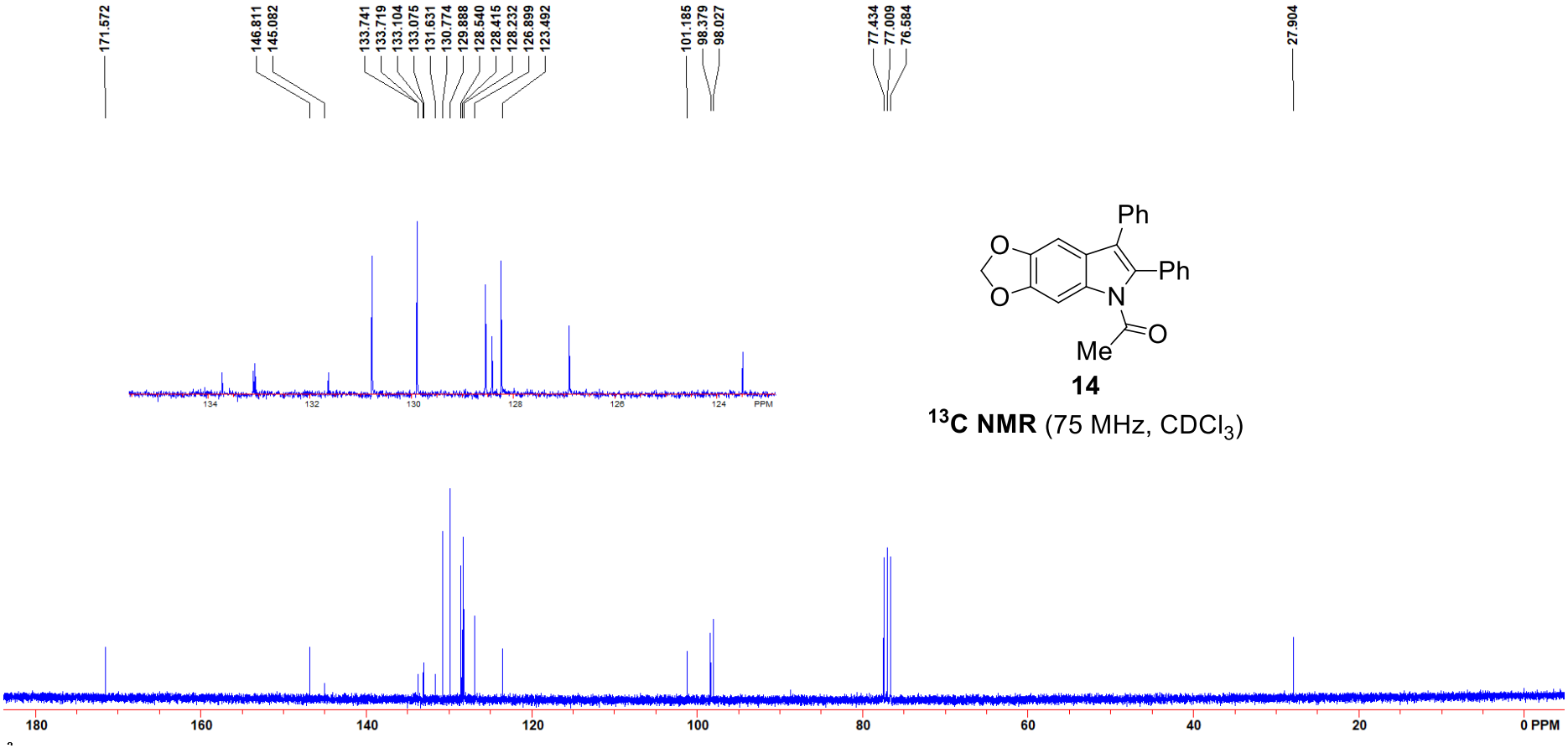

9.116
9.113
8.016
8.012
7.989
7.984
7.598
7.570
7.398
7.386
7.373
7.367
7.361
7.354
7.344
7.340
7.332
7.324
7.303
7.293
7.277
7.270
7.255
7.215
7.208
7.201
7.189
7.184
3.959
2.023
-0.000
Ph
Ph
MeO2C
N
O
Me
13
1H NMR (300 MHz, CDCl3)
5.03
3.03
2.03
1.00
1.03
1.03
3.01
3.03
12
10
8
6
4
2
0 PPM



13

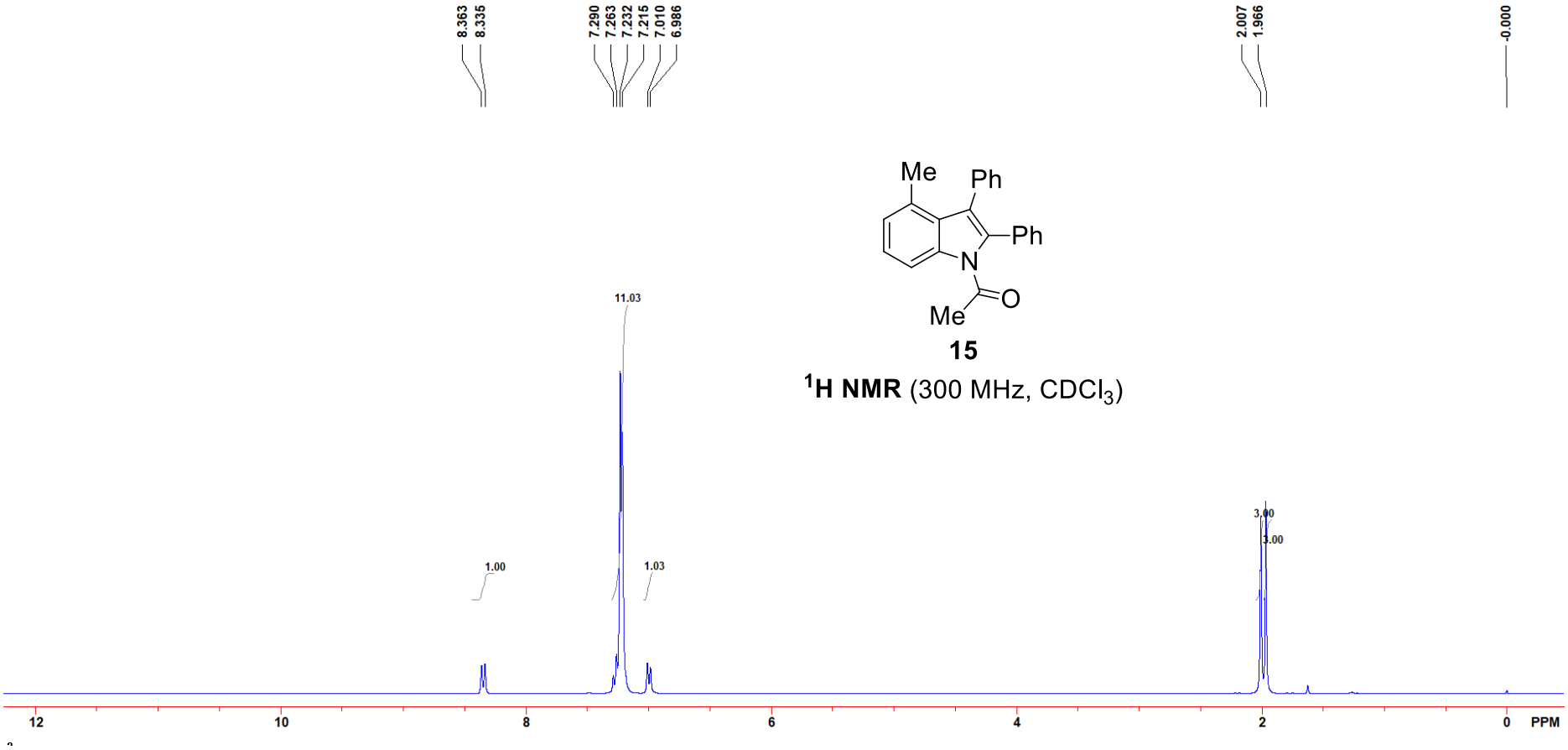
^{13}C NMR (75 MHz, $CDCl_3$)


8.038
7.349
7.343
7.337
7.327
7.319
7.314
7.305
7.303
7.297
7.291
7.287
7.276
7.271
7.267
7.253
7.247
7.240
7.235
7.226
7.184
7.177
7.169
7.162
7.157
7.151
6.909
5.991
1.964
0.000
Ph
O
O
Ph
N
O
Me
14
1H NMR (300 MHz, CDCl3)
5.03
3.05
2.01
1.00
1.02
2.00
3.01
12
10
8
6
4
2
0
PPM



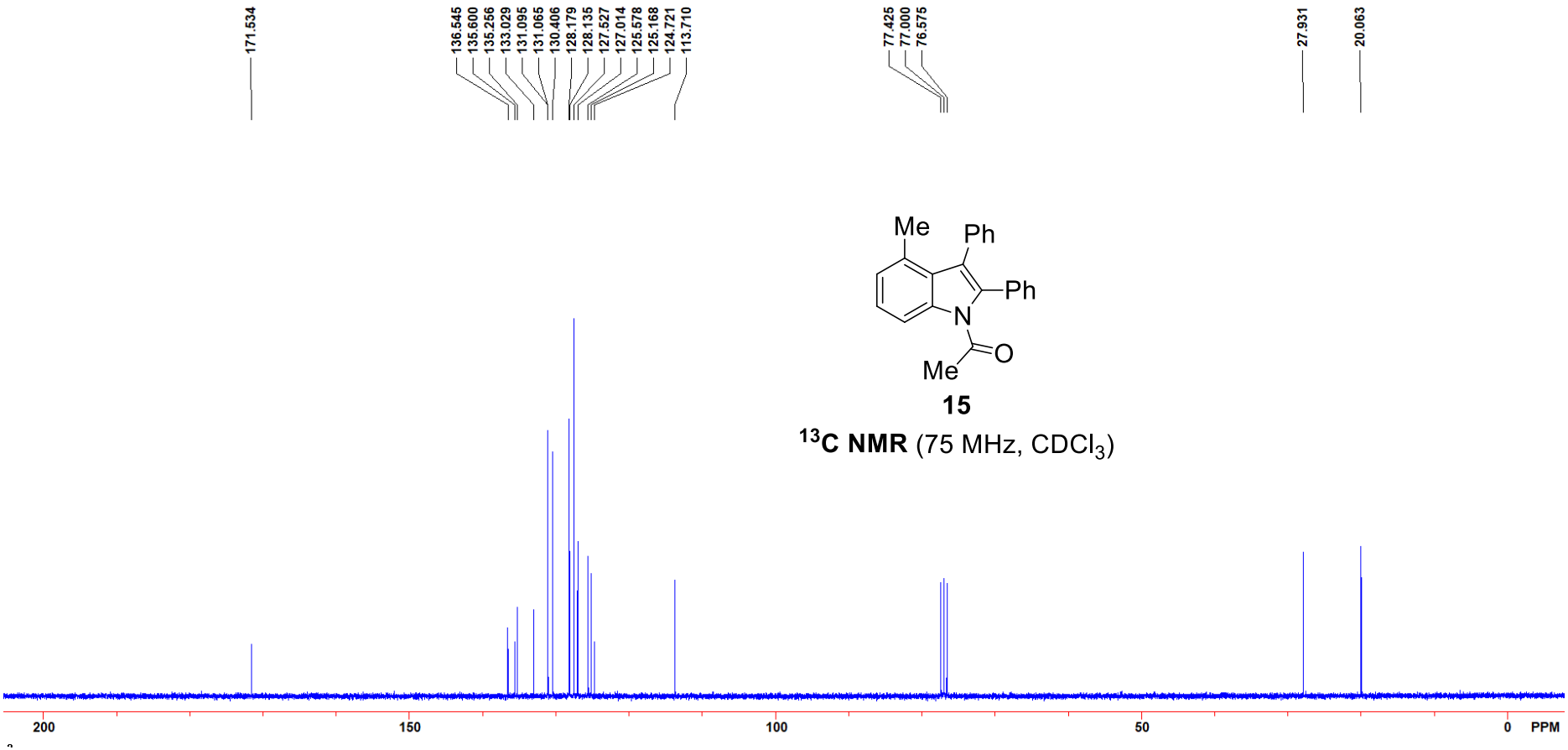
14

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



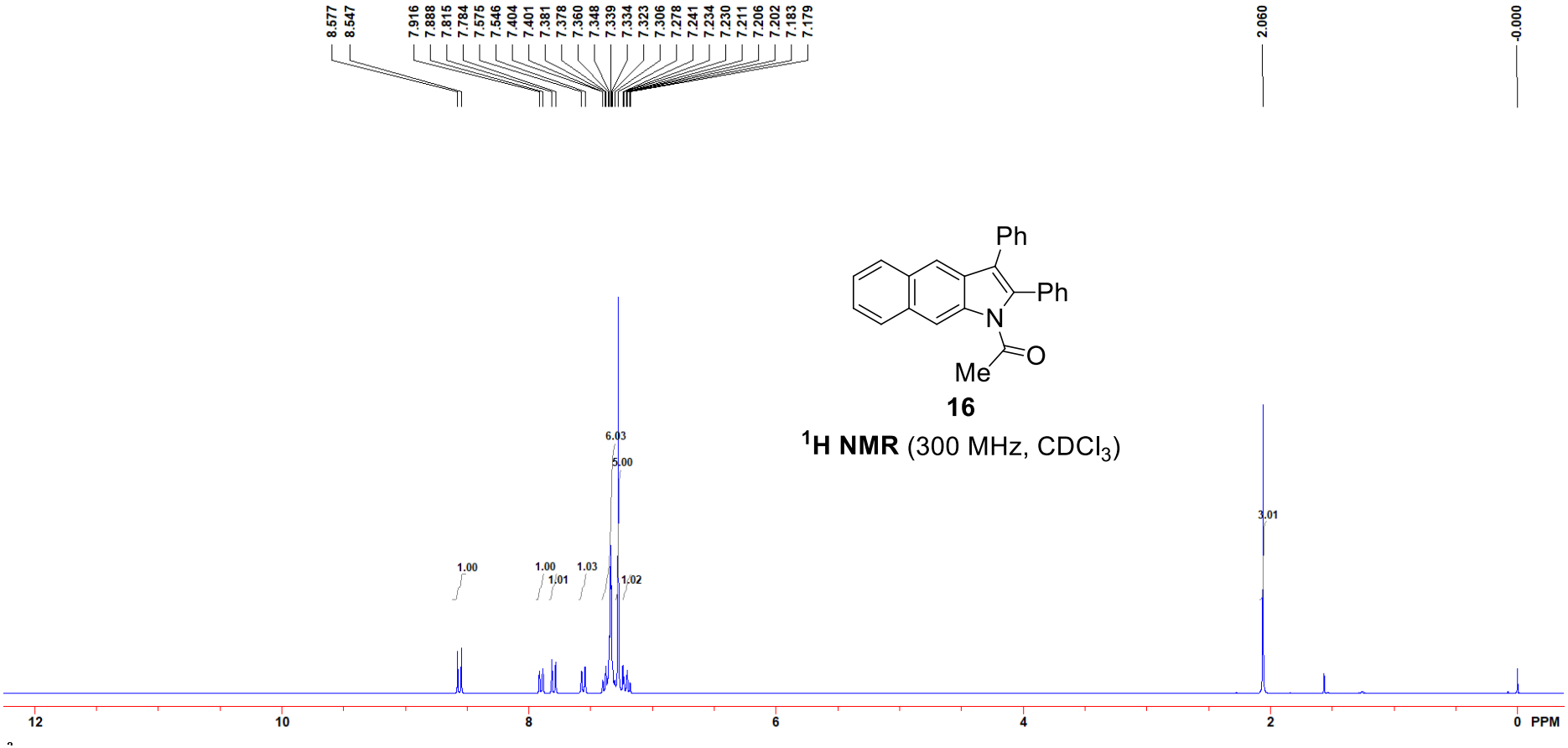
15

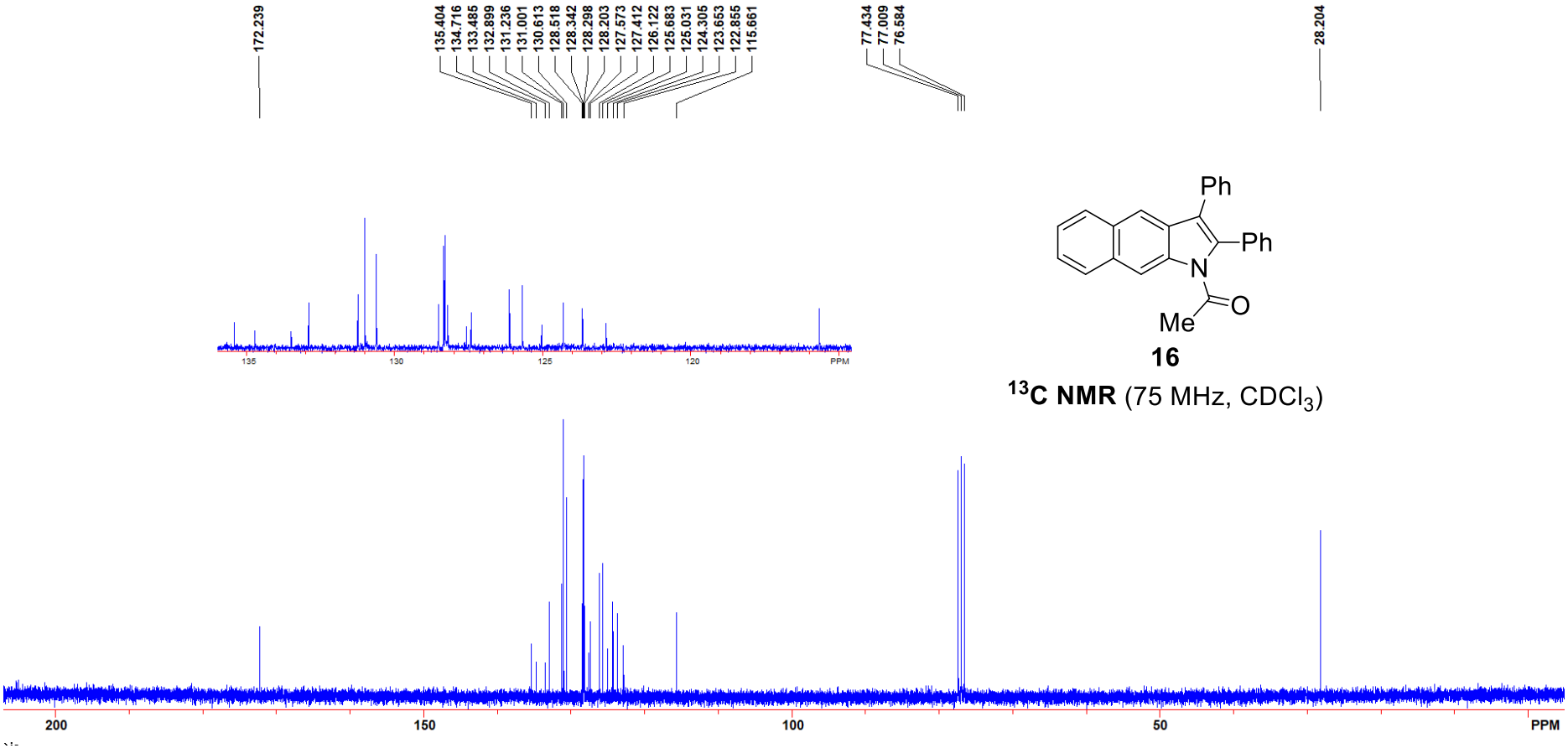
^{1}H NMR (300 MHz, $CDCl_3$)



15

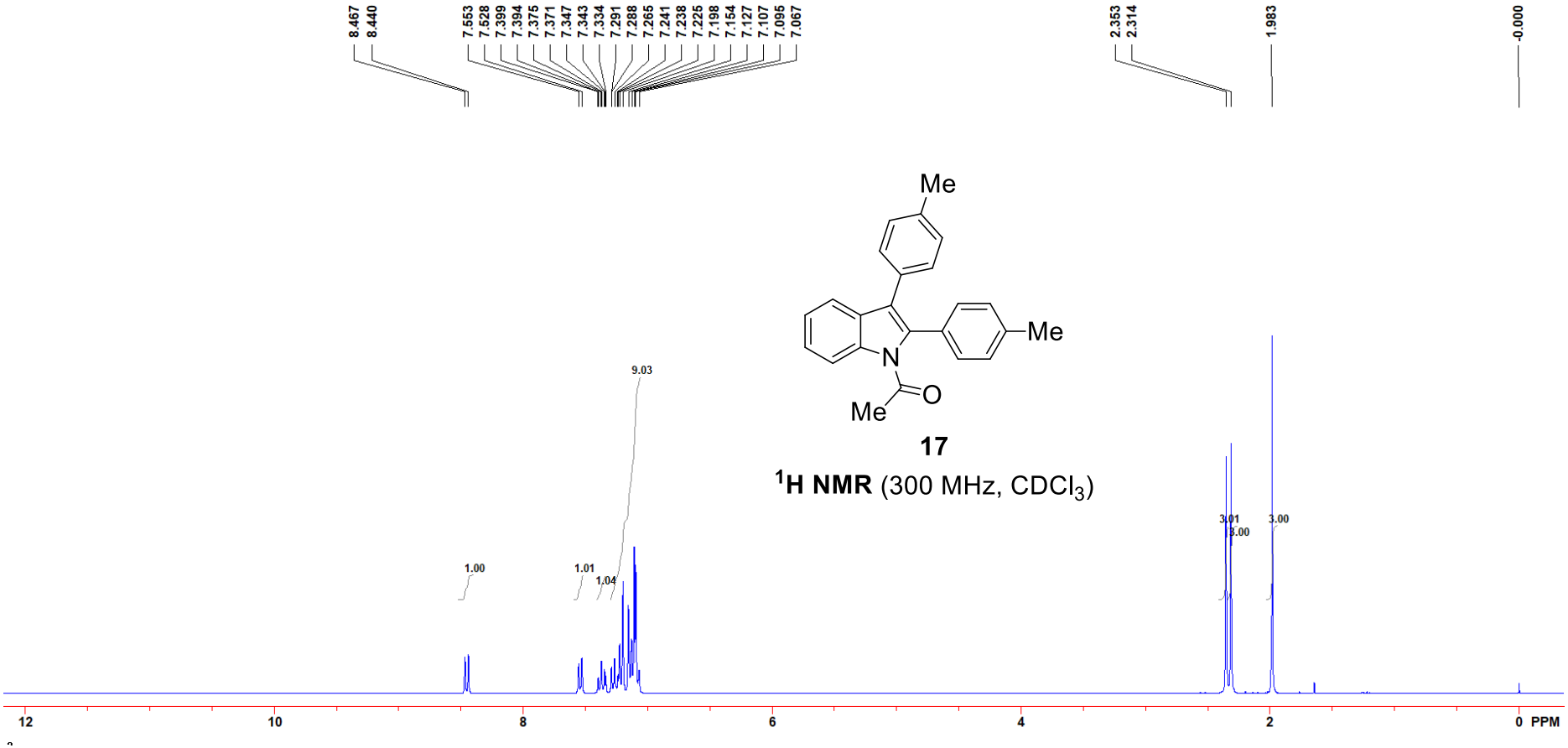
^{13}C NMR (75 MHz, $CDCl_3$)

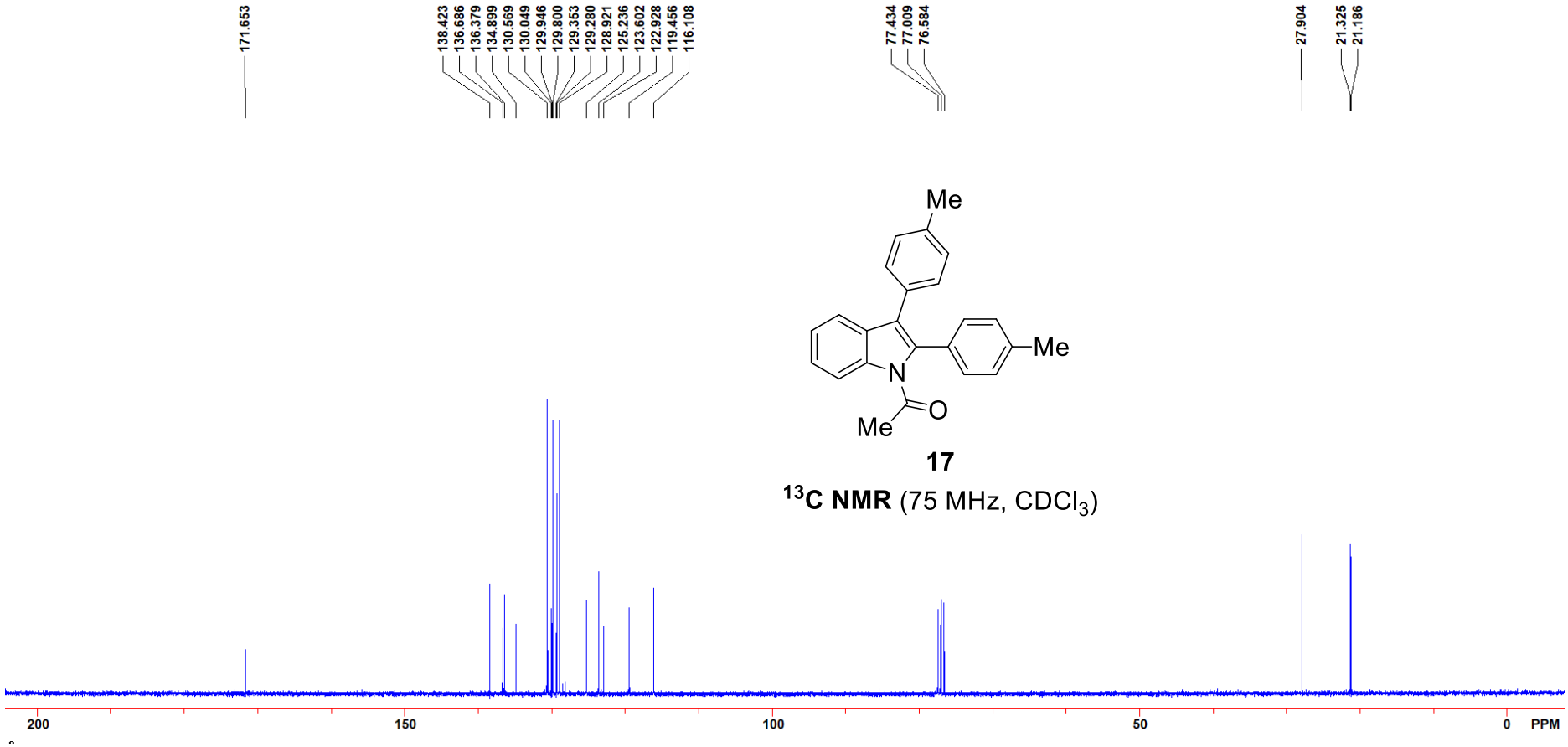

8.577
8.547
7.916
7.888
7.815
7.784
7.575
7.546
7.404
7.401
7.381
7.378
7.360
7.348
7.339
7.334
7.323
7.306
7.278
7.241
7.234
7.230
7.211
7.206
7.202
7.183
7.179
2.060
-0.000
Ph
Ph
N
O
Me
16
1H NMR (300 MHz, CDCl3)
6.03
5.00
3.01
1.00
1.00
1.01
1.03
1.02
12
10
8
6
4
2
0 PPM



16

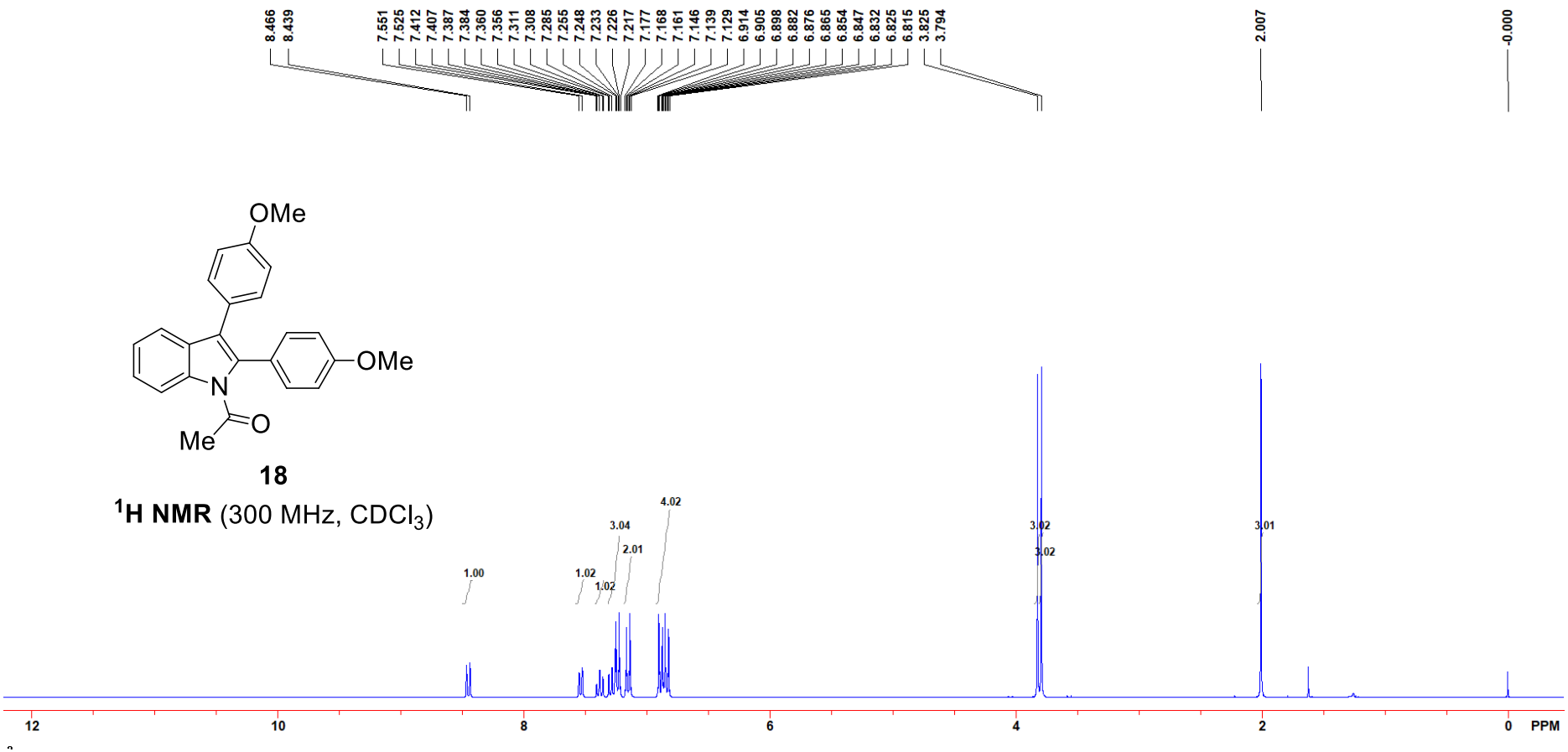
^{13}C NMR (75 MHz, $CDCl_3$)

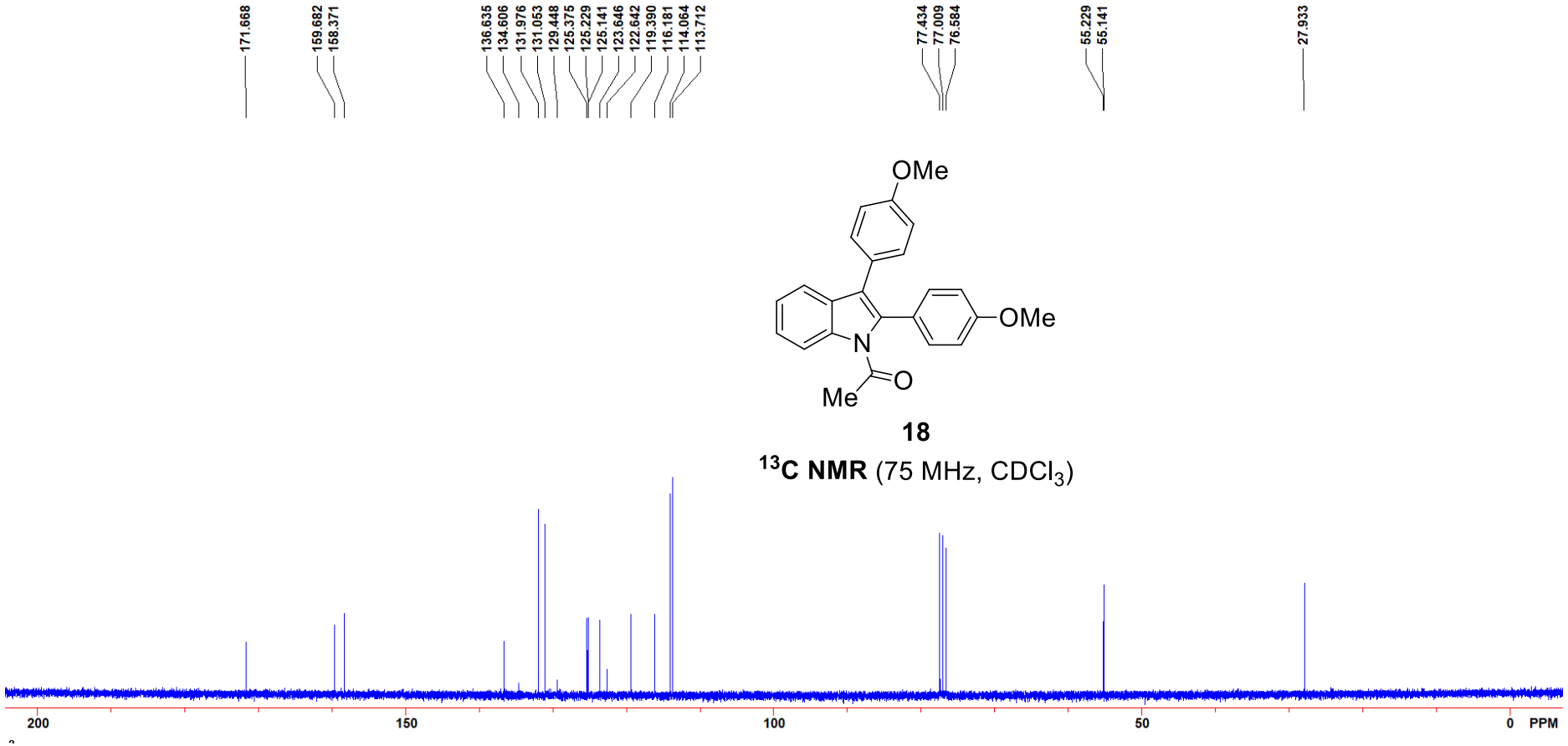

8.467
8.440
7.553
7.528
7.399
7.394
7.375
7.371
7.347
7.343
7.334
7.291
7.288
7.265
7.241
7.238
7.225
7.198
7.154
7.127
7.107
7.095
7.067
2.353
2.314
1.983
0.000
Me
Me
N
O
Me
17
1H NMR (300 MHz, CDCl3)
9.03
1.00
1.01
1.04
3.01
3.00
3.00
12
10
8
6
4
2
0 PPM



17

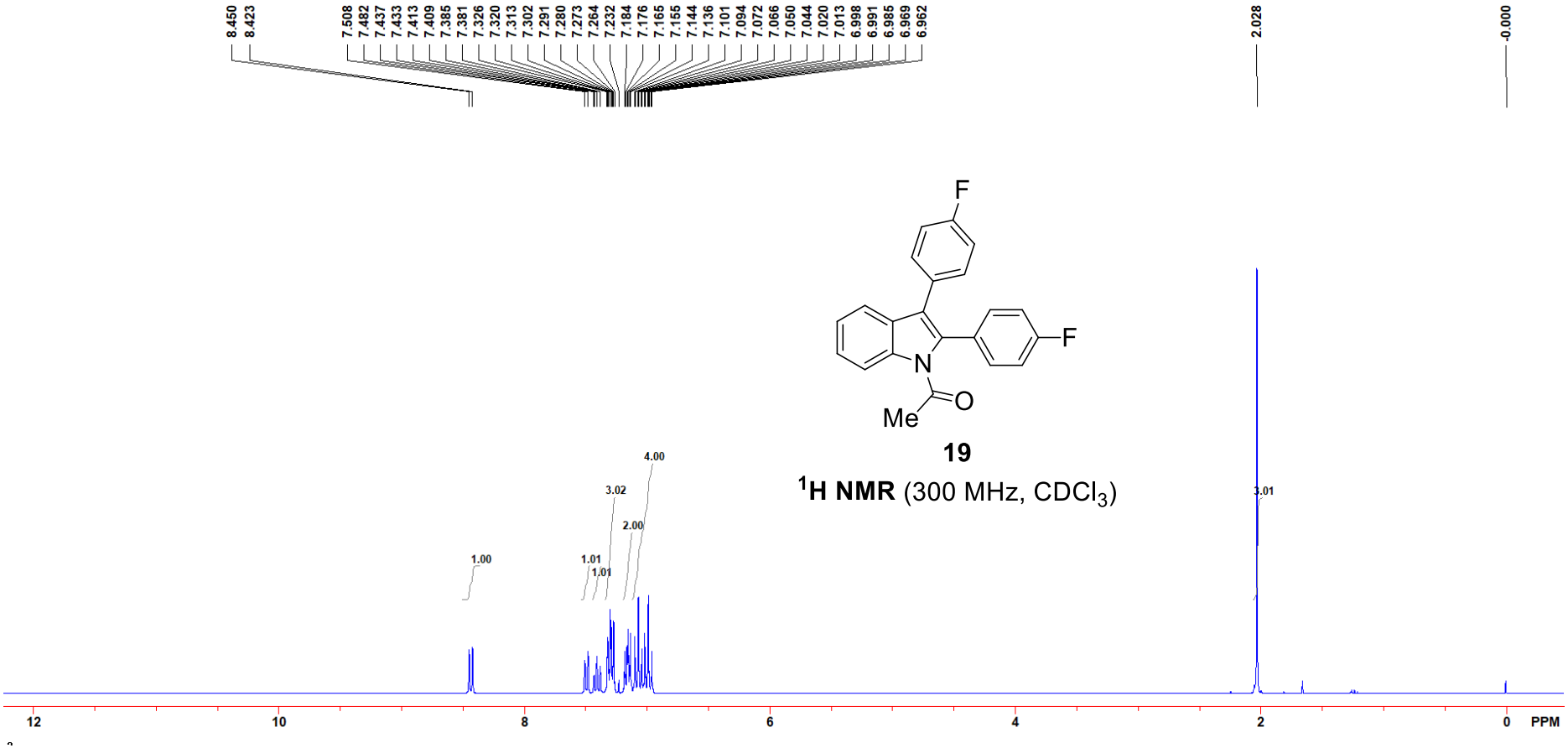
^{13}C NMR (75 MHz, $CDCl_3$)

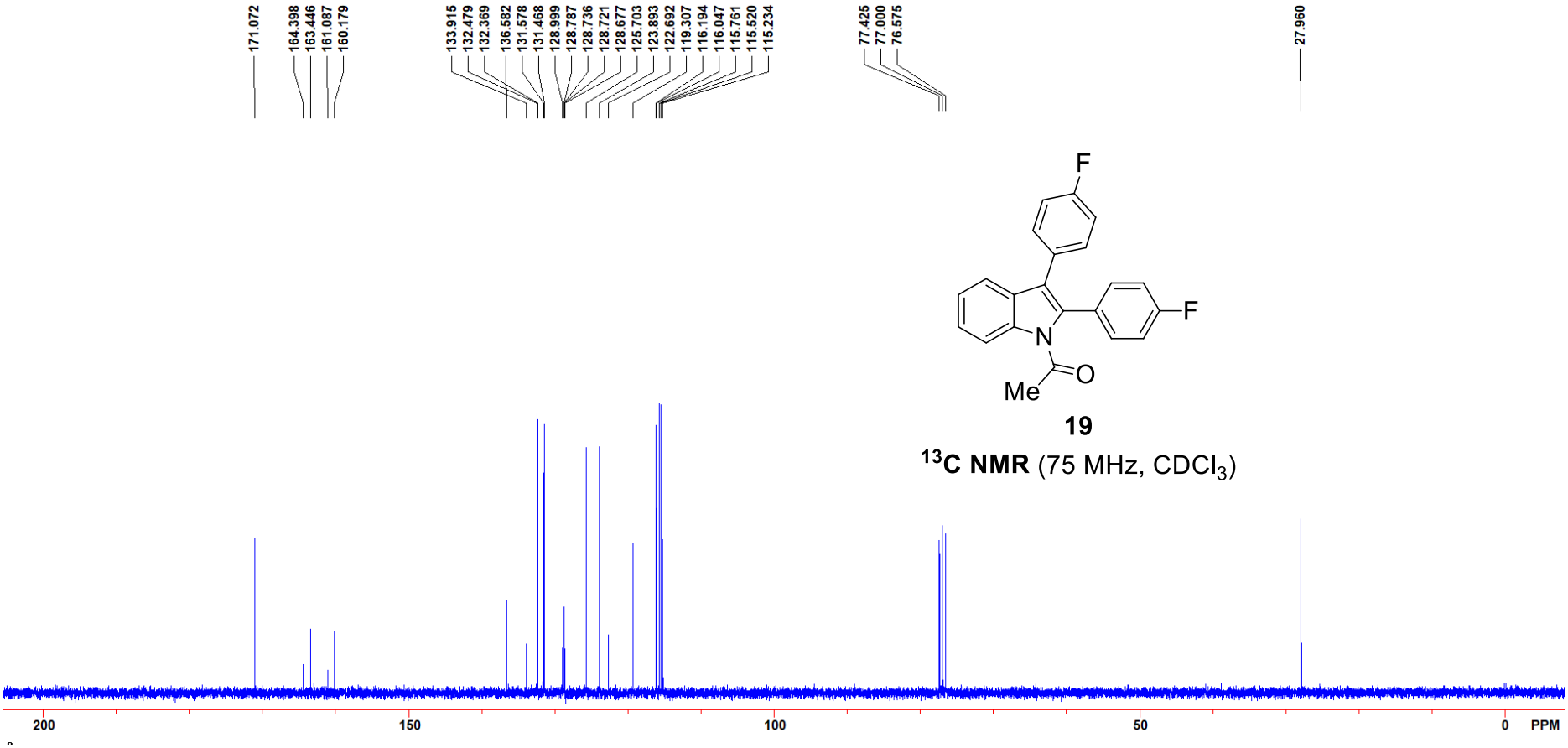

8.466
8.439
7.551
7.525
7.412
7.407
7.387
7.384
7.360
7.356
7.311
7.308
7.285
7.255
7.248
7.233
7.226
7.217
7.177
7.168
7.161
7.146
7.139
7.129
6.914
6.905
6.898
6.882
6.876
6.865
6.854
6.847
6.832
6.825
6.815
3.825
3.794
2.007
-0.000
OMe
OMe
N
O
Me
18
1H NMR (300 MHz, CDCl3)
1.00
1.02
1.02
3.04
2.01
4.02
3.02
3.02
3.01
12
10
8
6
4
2
0
PPM



18

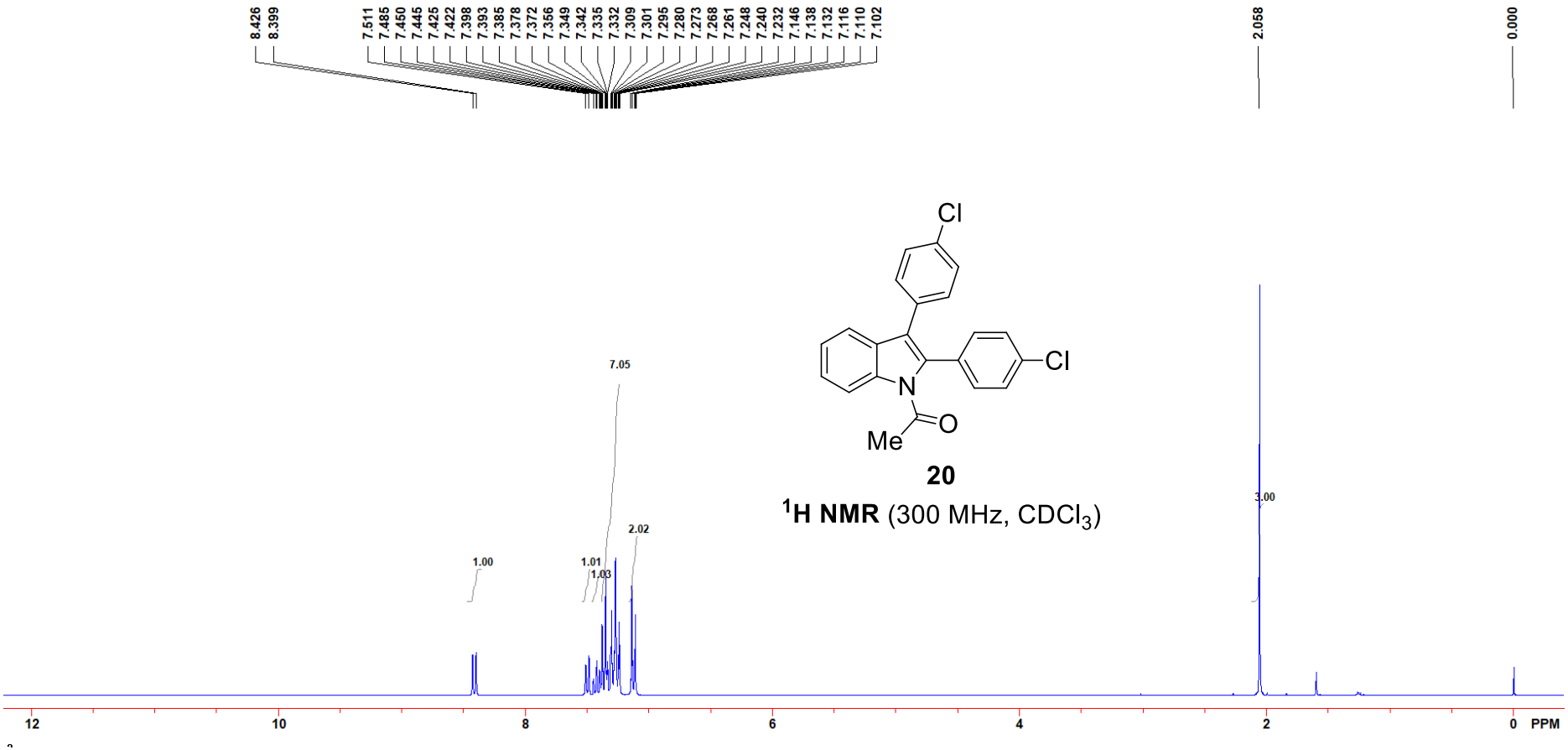
^{13}C NMR (75 MHz, $CDCl_3$)


8.450
8.423
7.508
7.482
7.437
7.433
7.413
7.409
7.385
7.381
7.326
7.320
7.313
7.302
7.291
7.280
7.273
7.264
7.232
7.184
7.176
7.165
7.155
7.144
7.136
7.101
7.094
7.072
7.066
7.050
7.044
7.020
7.013
6.998
6.991
6.985
6.969
6.962
2.028
-0.000
F
F
N
O
Me
19
1H NMR (300 MHz, CDCl3)
4.00
3.02
2.00
1.00
1.01
1.01
3.01
12
10
8
6
4
2
0
PPM



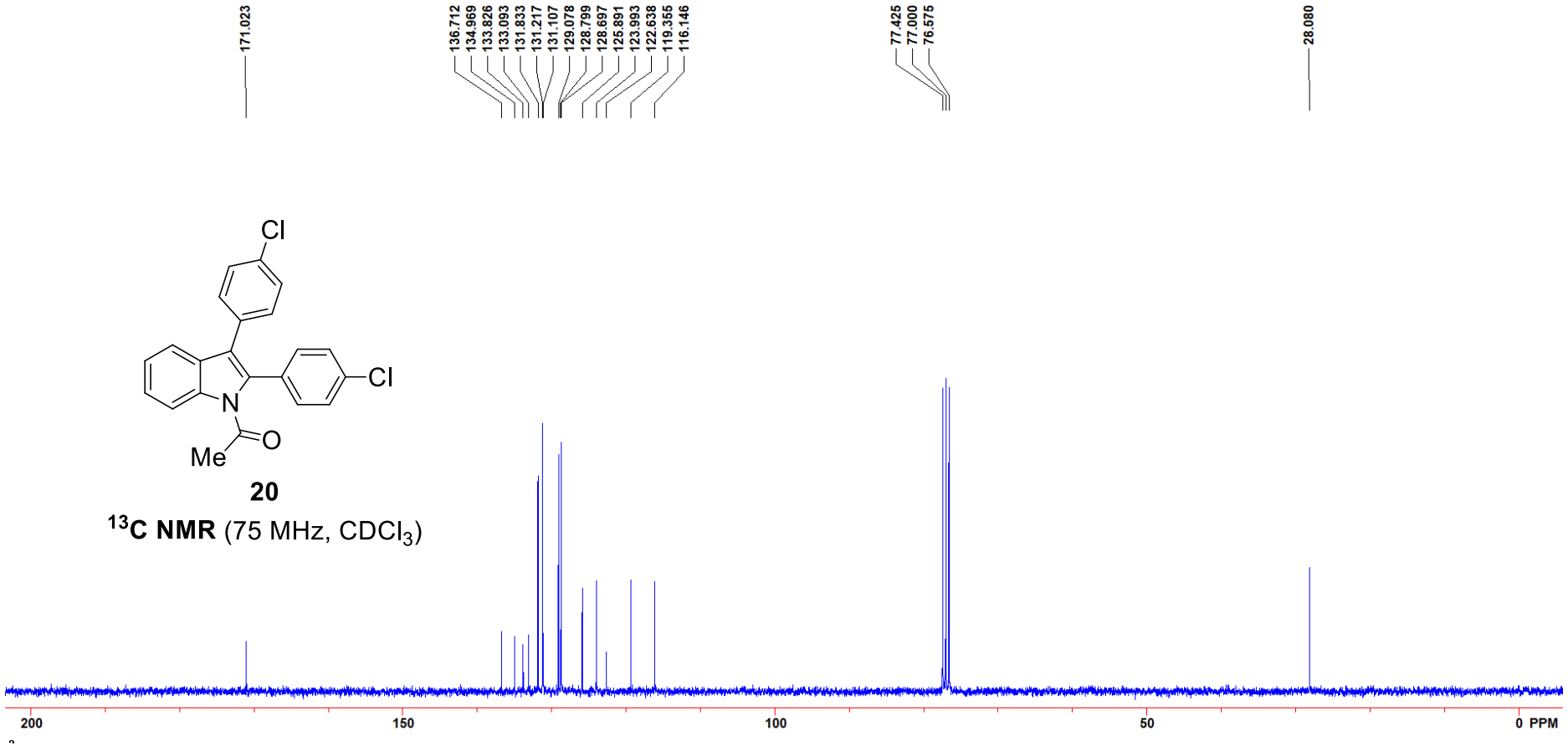
19

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



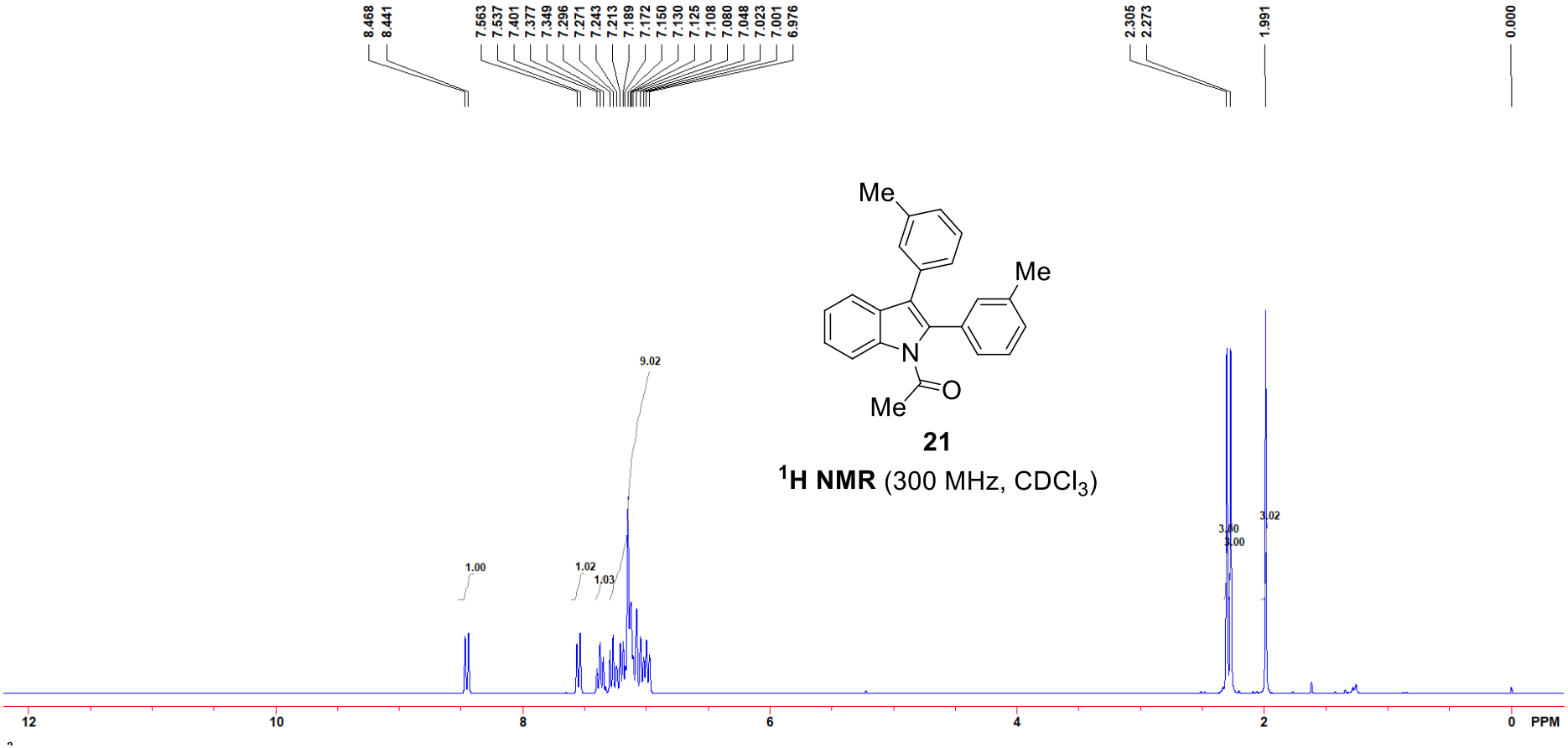
20

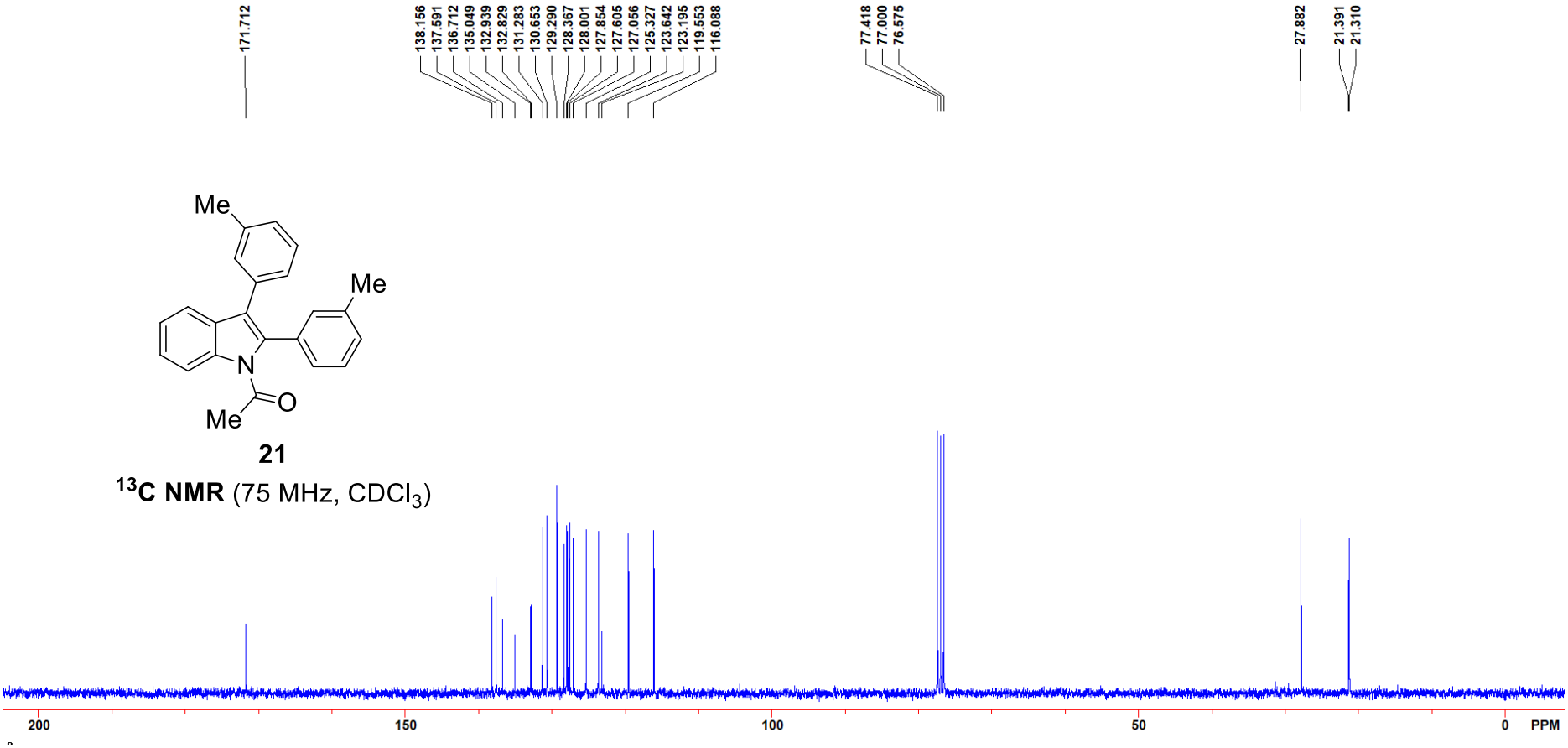
^{1}H NMR (300 MHz, $CDCl_3$)



20

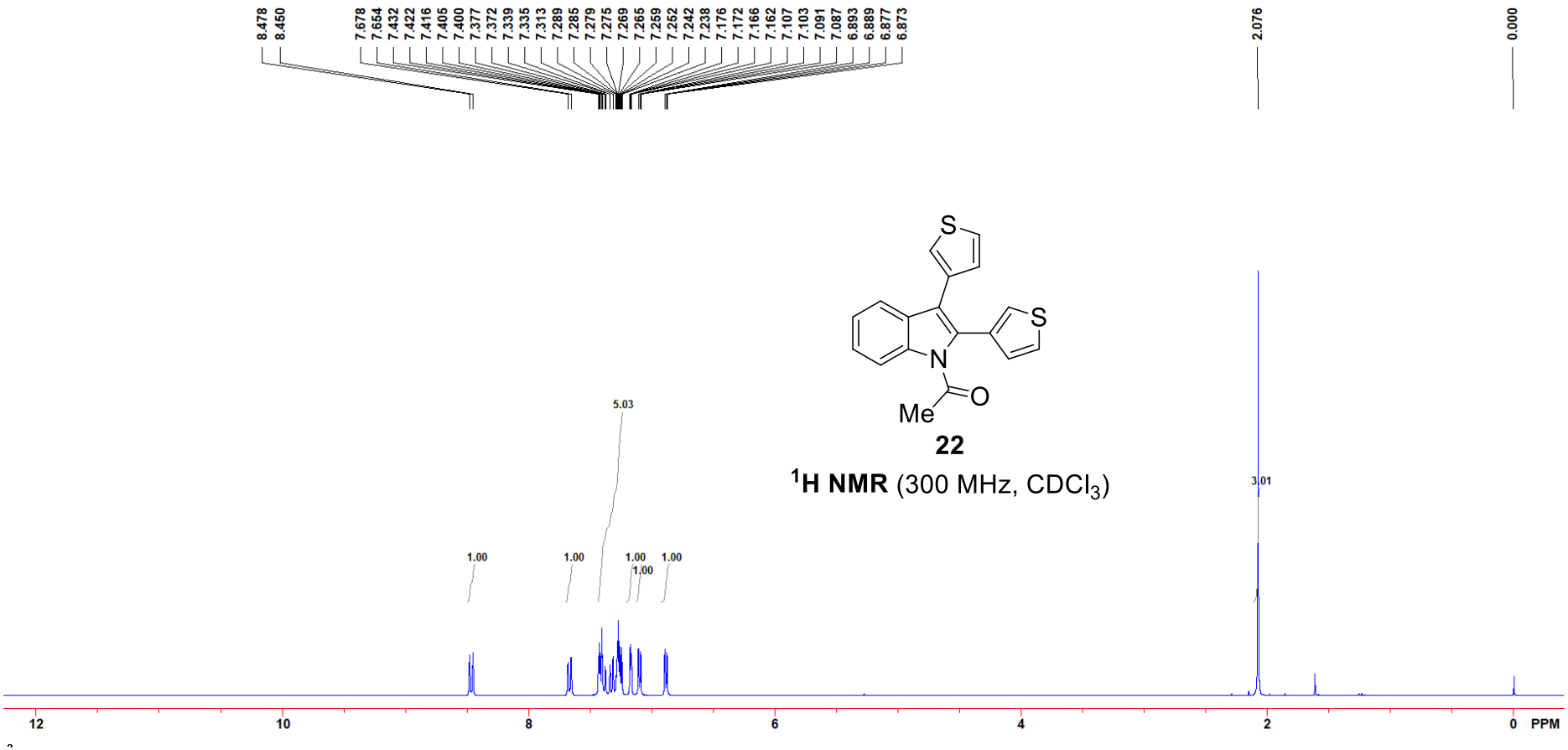
^{13}C NMR (75 MHz, $CDCl_3$)

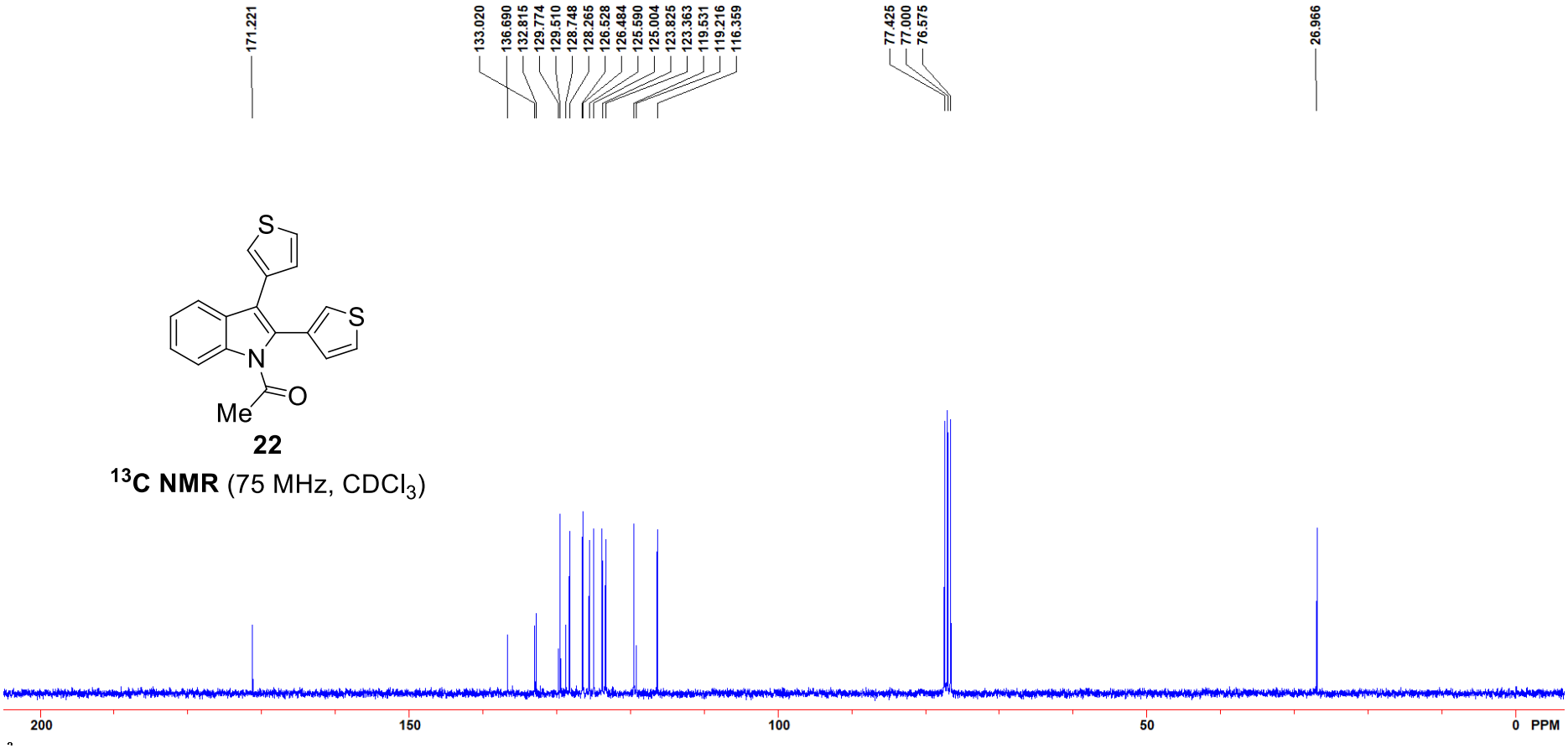

8.468
8.441
7.563
7.537
7.401
7.377
7.349
7.296
7.271
7.243
7.213
7.189
7.172
7.150
7.130
7.125
7.108
7.080
7.048
7.023
7.001
6.976
2.305
2.273
1.991
0.000
Me
Me
N
O
Me
21
1H NMR (300 MHz, CDCl3)
9.02
1.00
1.02
1.03
3.00
3.00
3.02
12
10
8
6
4
2
0
PPM



21

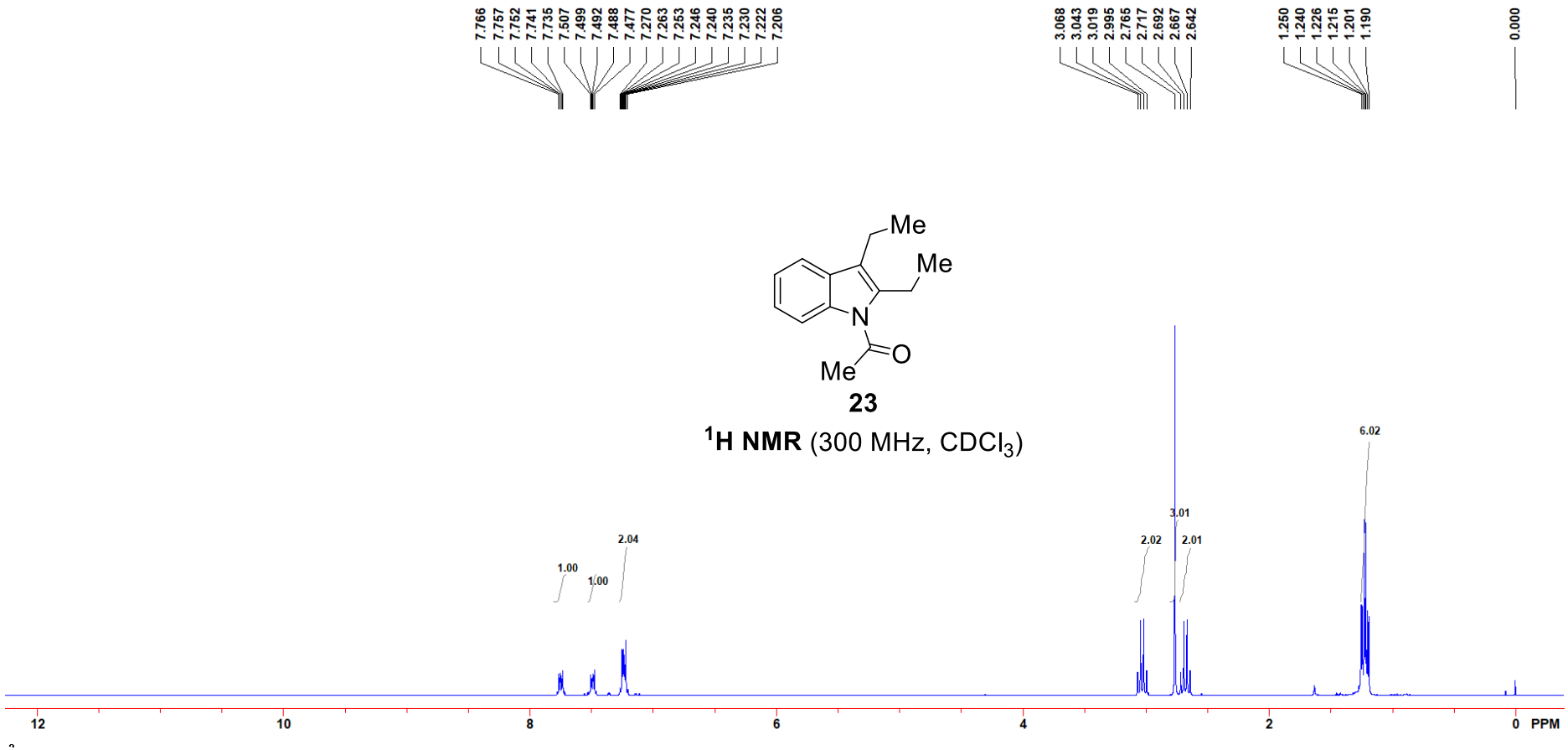
^{13}C NMR (75 MHz, $CDCl_3$)

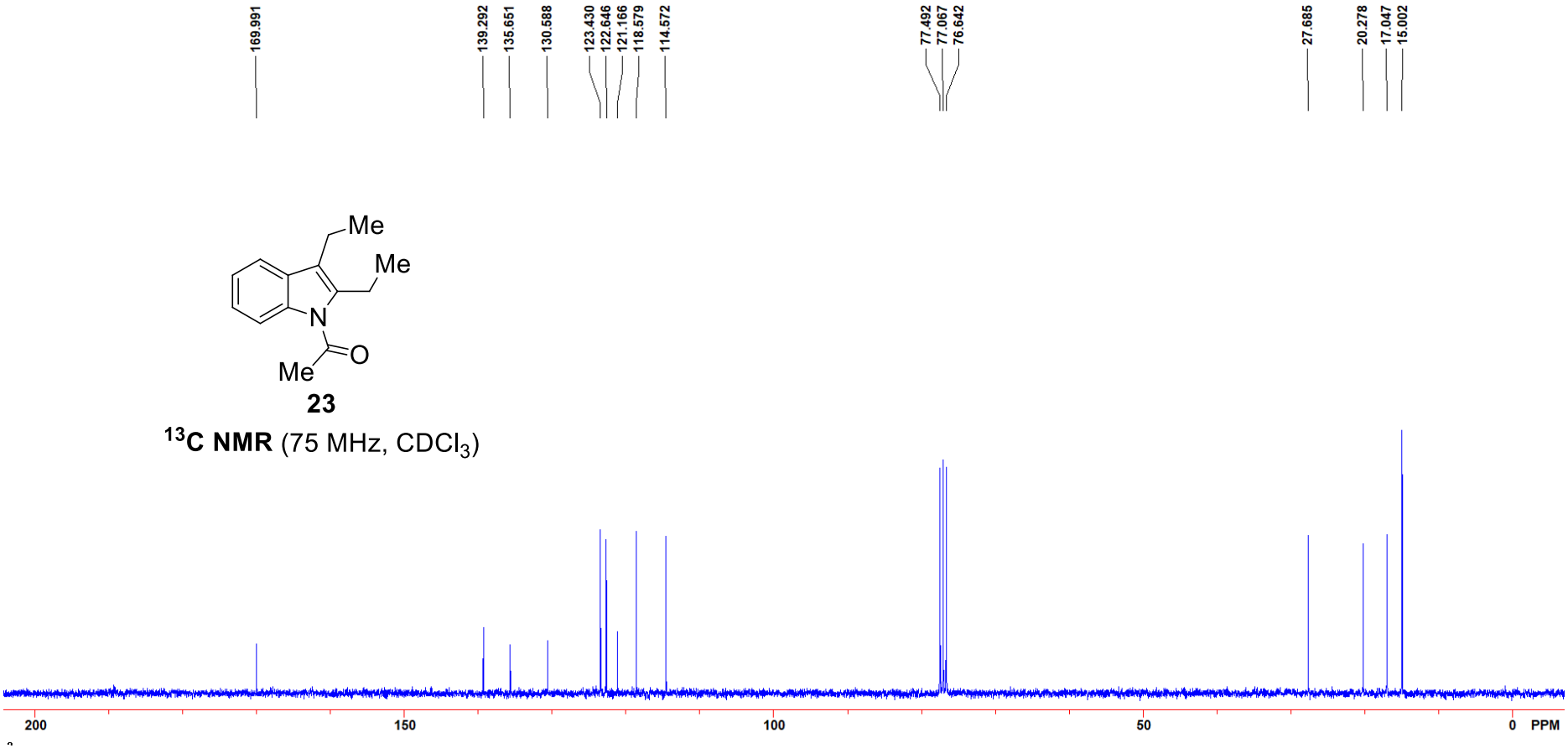

8.478
8.450
7.678
7.654
7.432
7.422
7.416
7.405
7.400
7.377
7.372
7.339
7.335
7.313
7.289
7.285
7.279
7.275
7.269
7.265
7.259
7.252
7.242
7.238
7.176
7.172
7.166
7.162
7.107
7.103
7.091
7.087
6.893
6.889
6.877
6.873
2.076
0.000
S
S
N
O
Me
22
1H NMR (300 MHz, CDCl3)
5.03
3.01
1.00
1.00
1.00
1.00
1.00
12
10
8
6
4
2
0
PPM



22

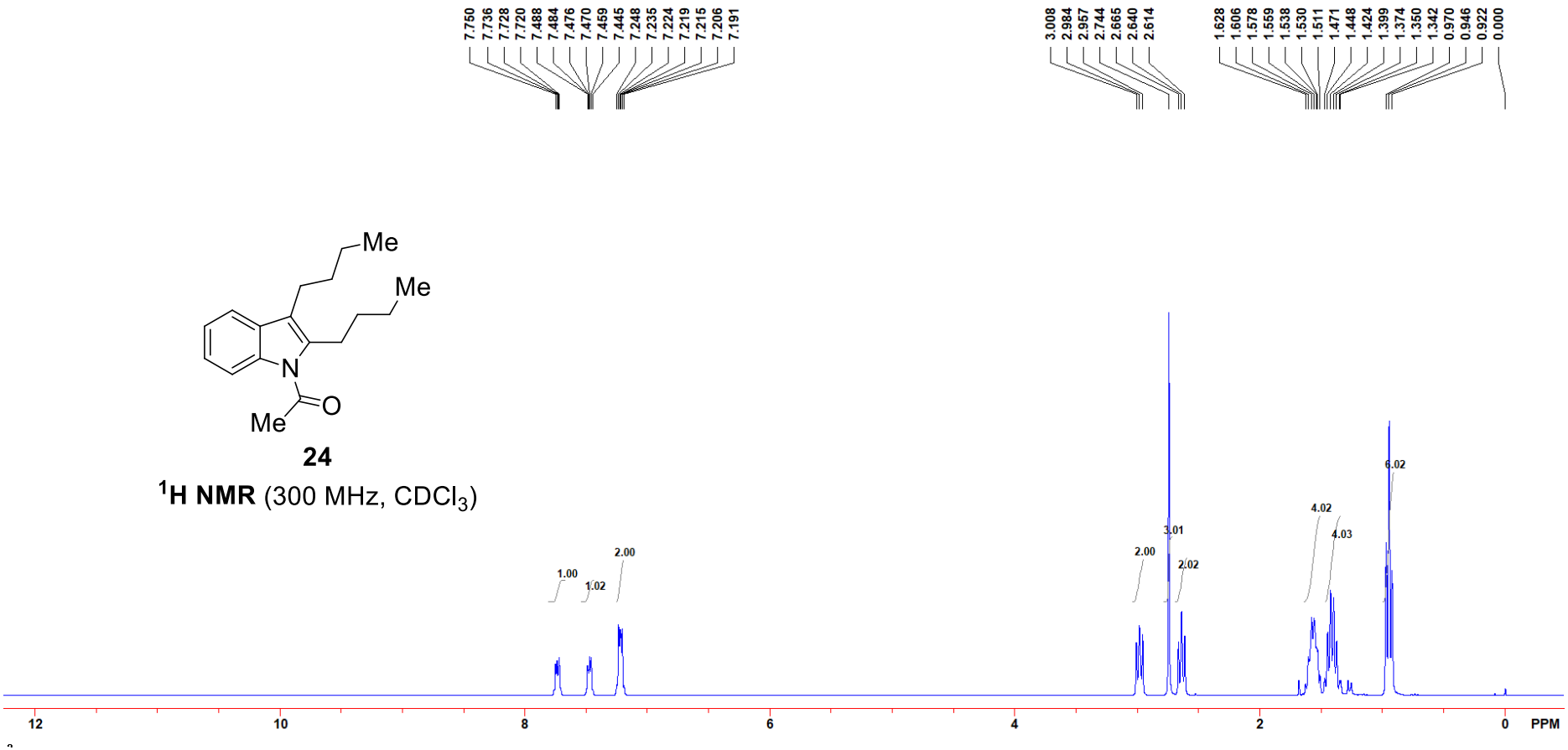
^{13}C NMR (75 MHz, $CDCl_3$)

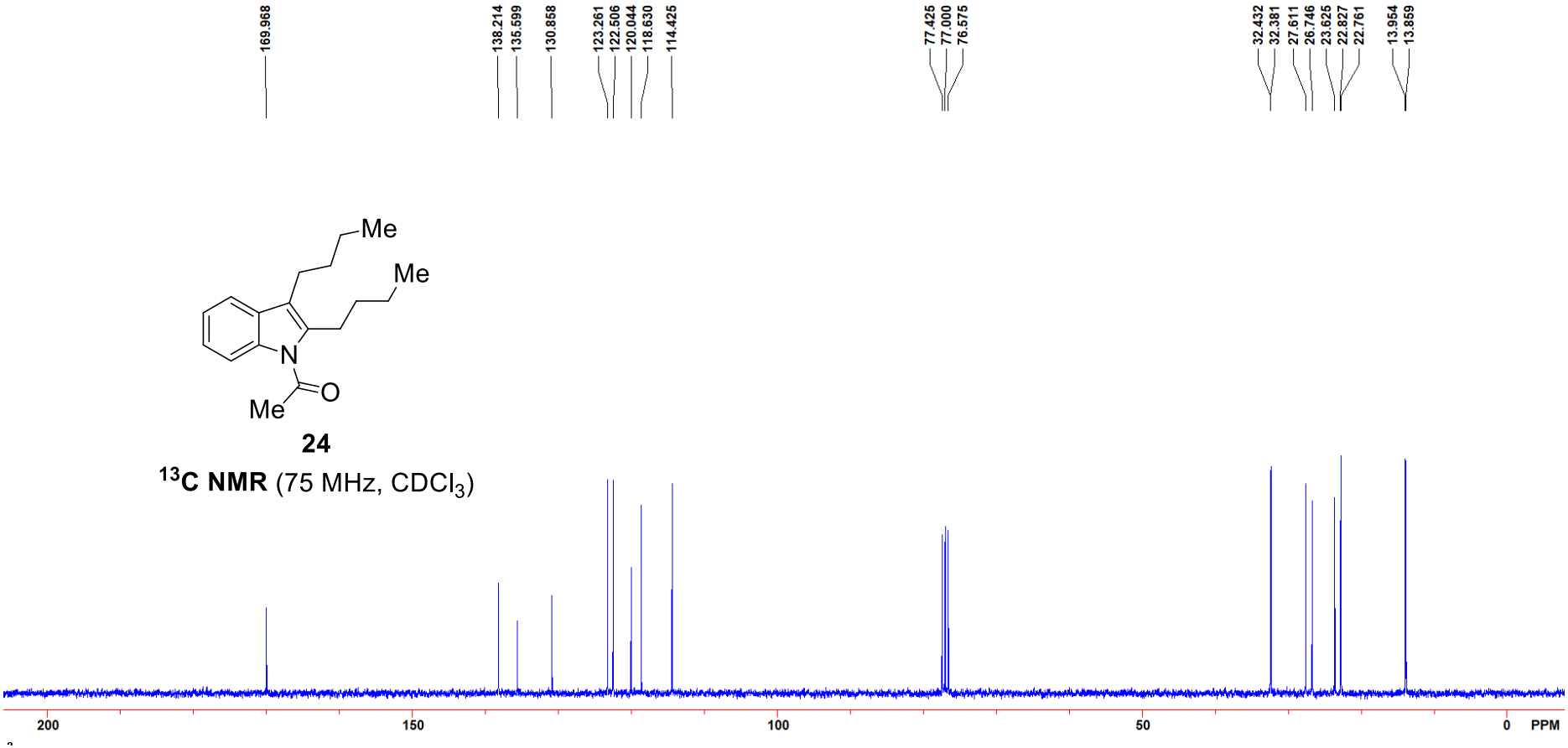

7.766
7.757
7.752
7.741
7.735
7.507
7.499
7.492
7.488
7.477
7.270
7.263
7.253
7.246
7.240
7.235
7.230
7.222
7.206
3.068
3.043
3.019
2.995
2.765
2.717
2.692
2.667
2.642
1.250
1.240
1.226
1.215
1.201
1.190
0.000
Me
Me
N
O
Me
23
1H NMR (300 MHz, CDCl3)
1.00
1.00
2.04
2.02
3.01
2.01
6.02
12
10
8
6
4
2
0 PPM



23

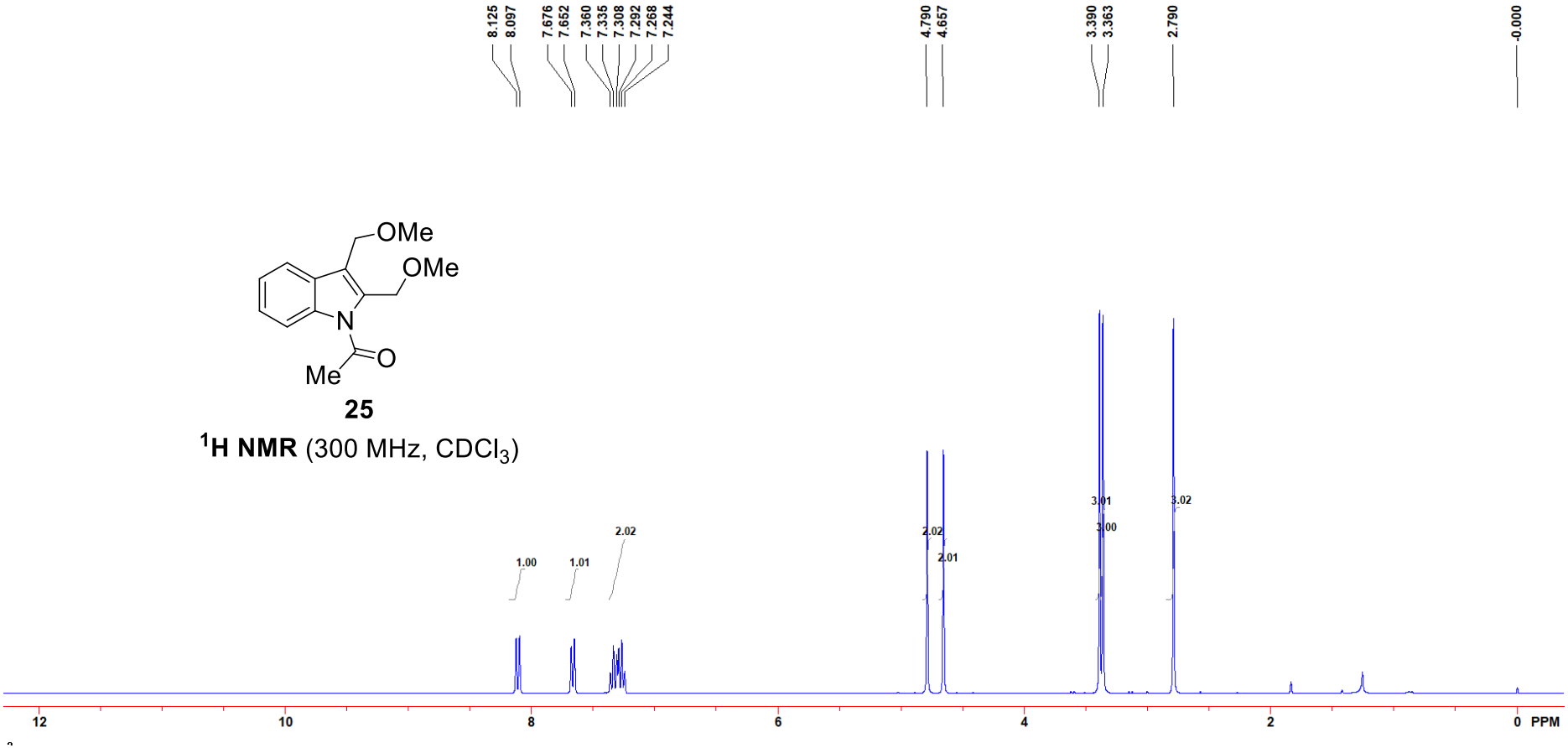
^{13}C NMR (75 MHz, $CDCl_3$)

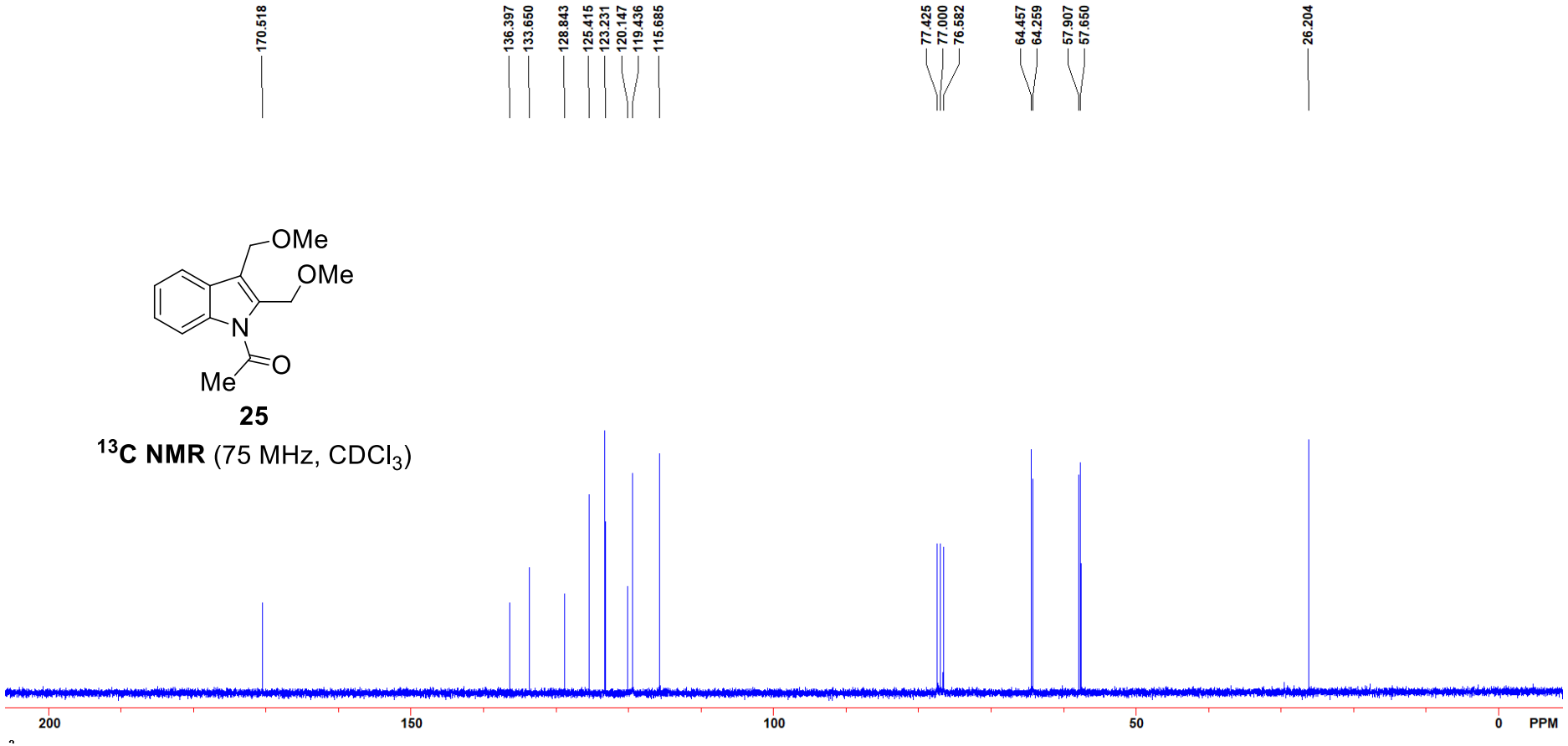

7.750
7.736
7.728
7.720
7.488
7.484
7.476
7.470
7.459
7.445
7.248
7.235
7.224
7.219
7.215
7.206
7.191
3.008
2.984
2.957
2.744
2.665
2.640
2.614
1.628
1.606
1.578
1.559
1.538
1.530
1.511
1.471
1.448
1.424
1.399
1.374
1.350
1.342
0.970
0.946
0.922
0.000
Me
Me
N
O
Me
24
1H NMR (300 MHz, CDCl3)
1.00
1.02
2.00
2.00
3.01
2.02
4.02
4.03
6.02
12
10
8
6
4
2
0
PPM



24

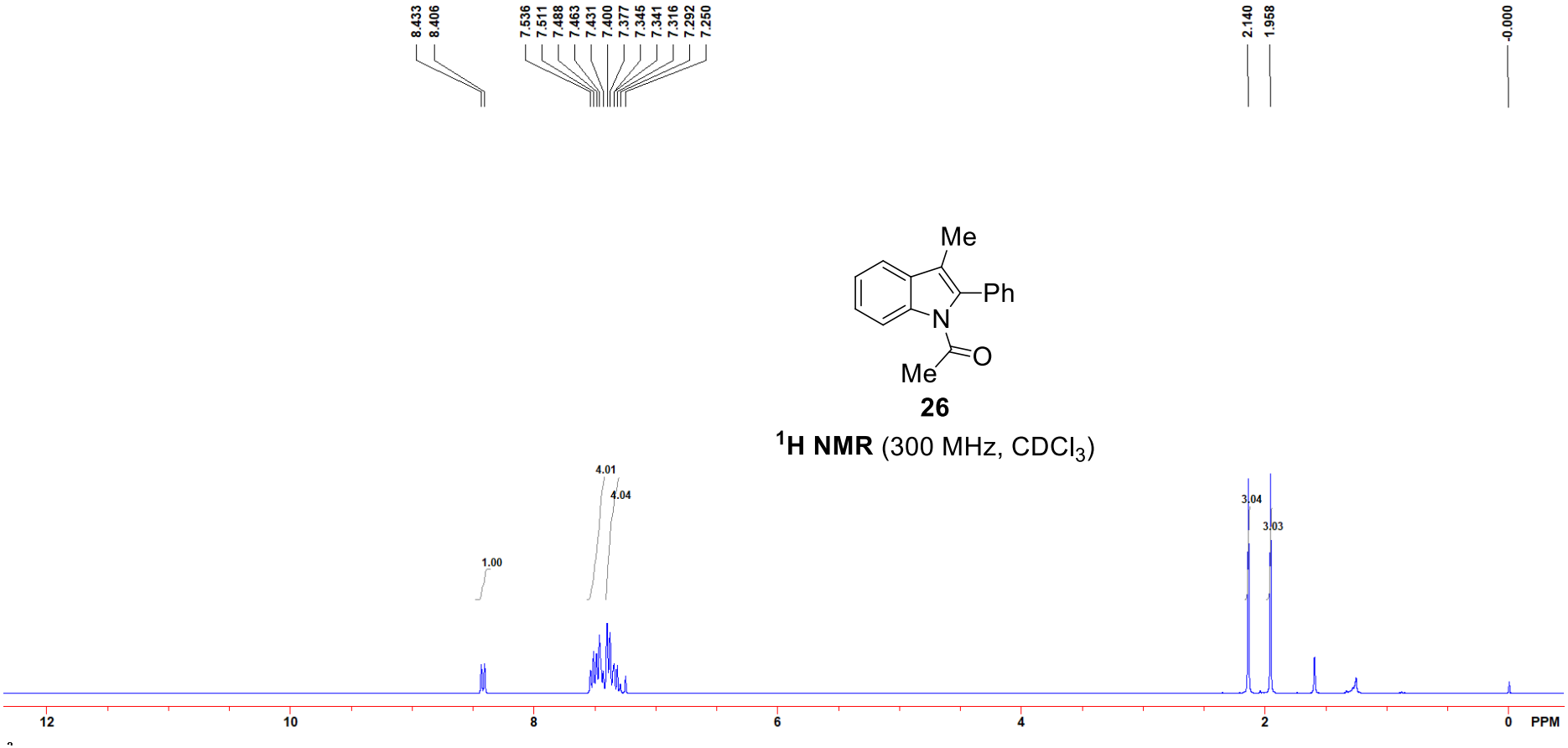
^{13}C NMR (75 MHz, $CDCl_3$)


8.125
8.097
7.676
7.652
7.360
7.335
7.308
7.292
7.268
7.244
4.790
4.657
3.390
3.363
2.790
-0.000
OMe
OMe
N
O
Me
25
1H NMR (300 MHz, CDCl3)
1.00
1.01
2.02
2.02
2.01
3.01
3.00
3.02
12
10
8
6
4
2
0 PPM



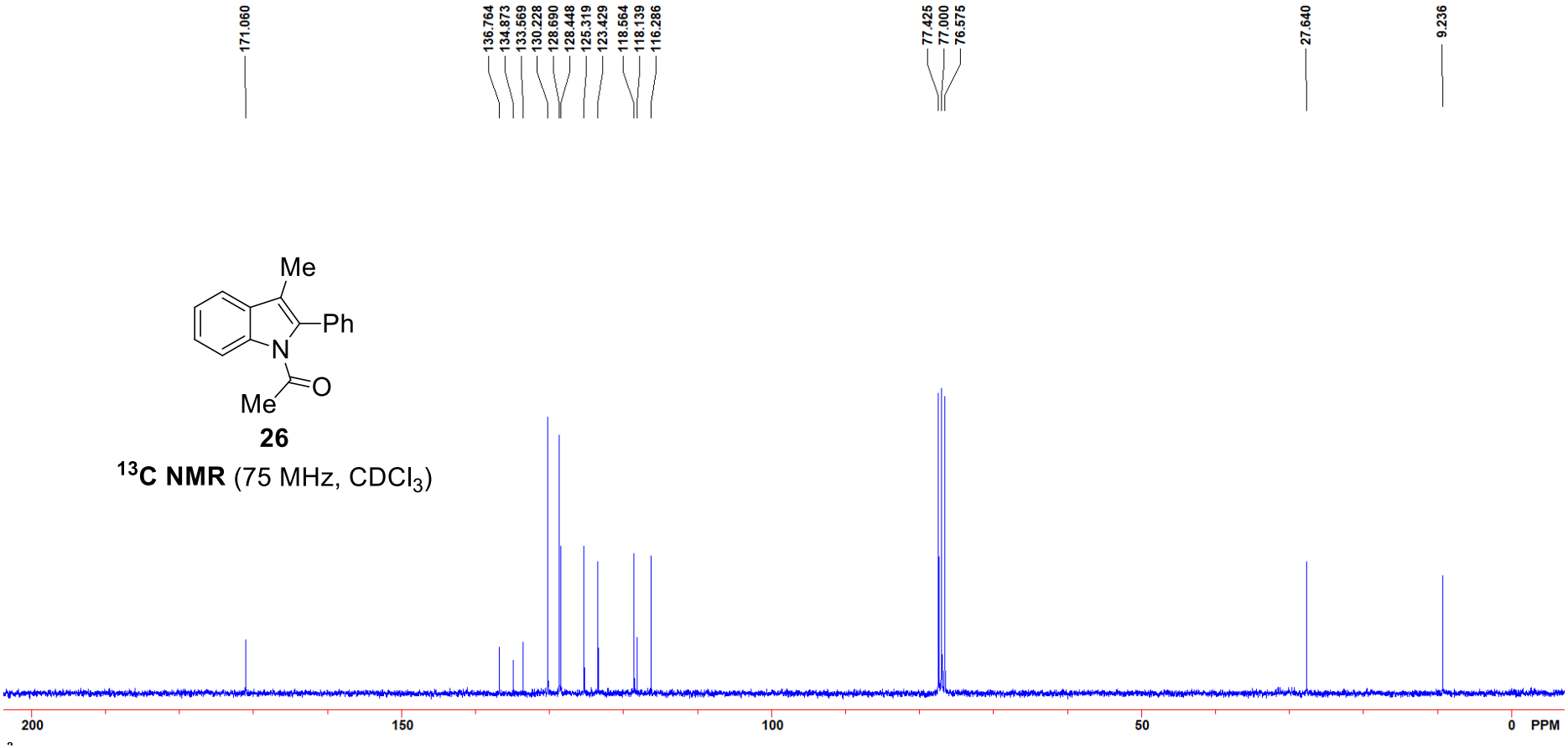
25

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



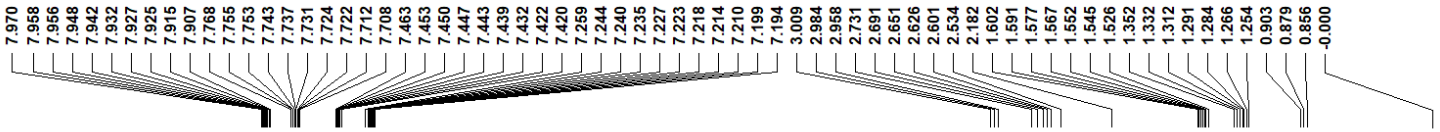
26

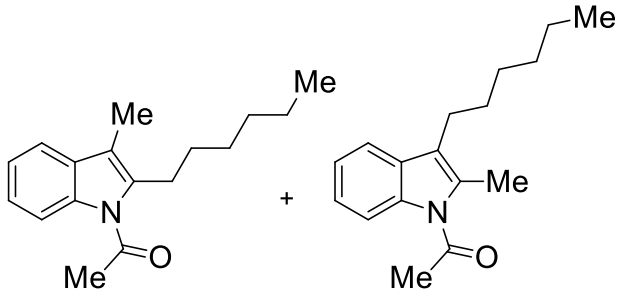
^{1}H NMR (300 MHz, $CDCl_3$)



26

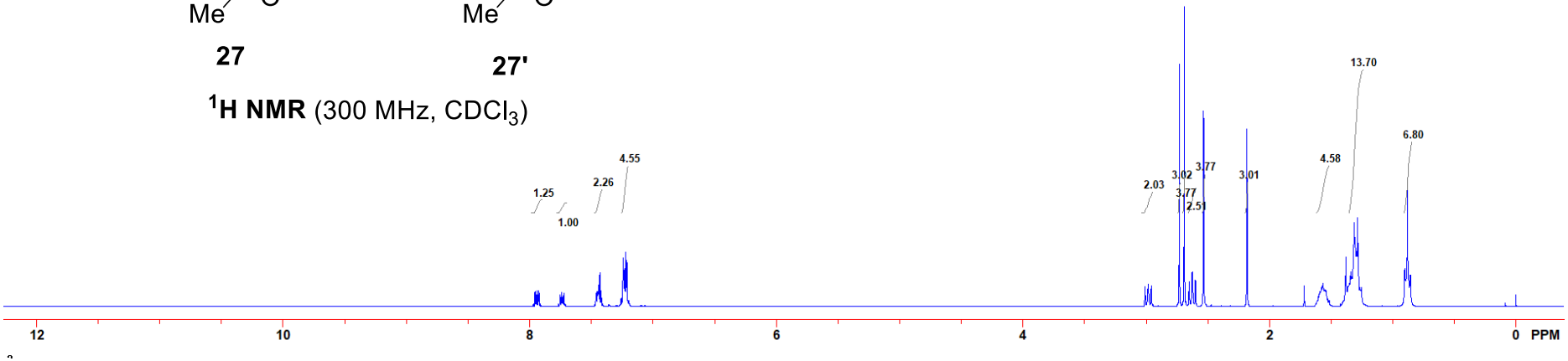
^{13}C NMR (75 MHz, $CDCl_3$)

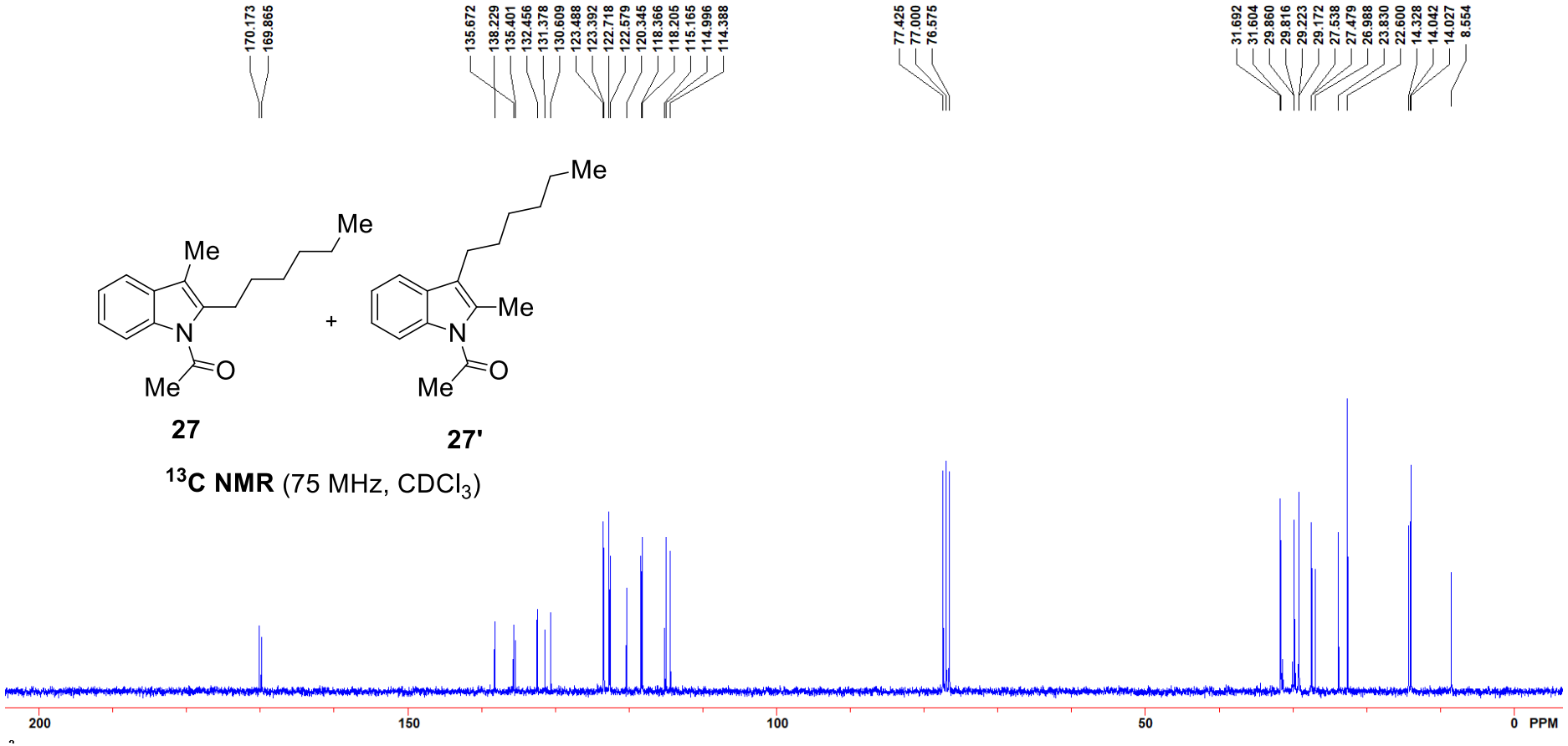




27 **27'**

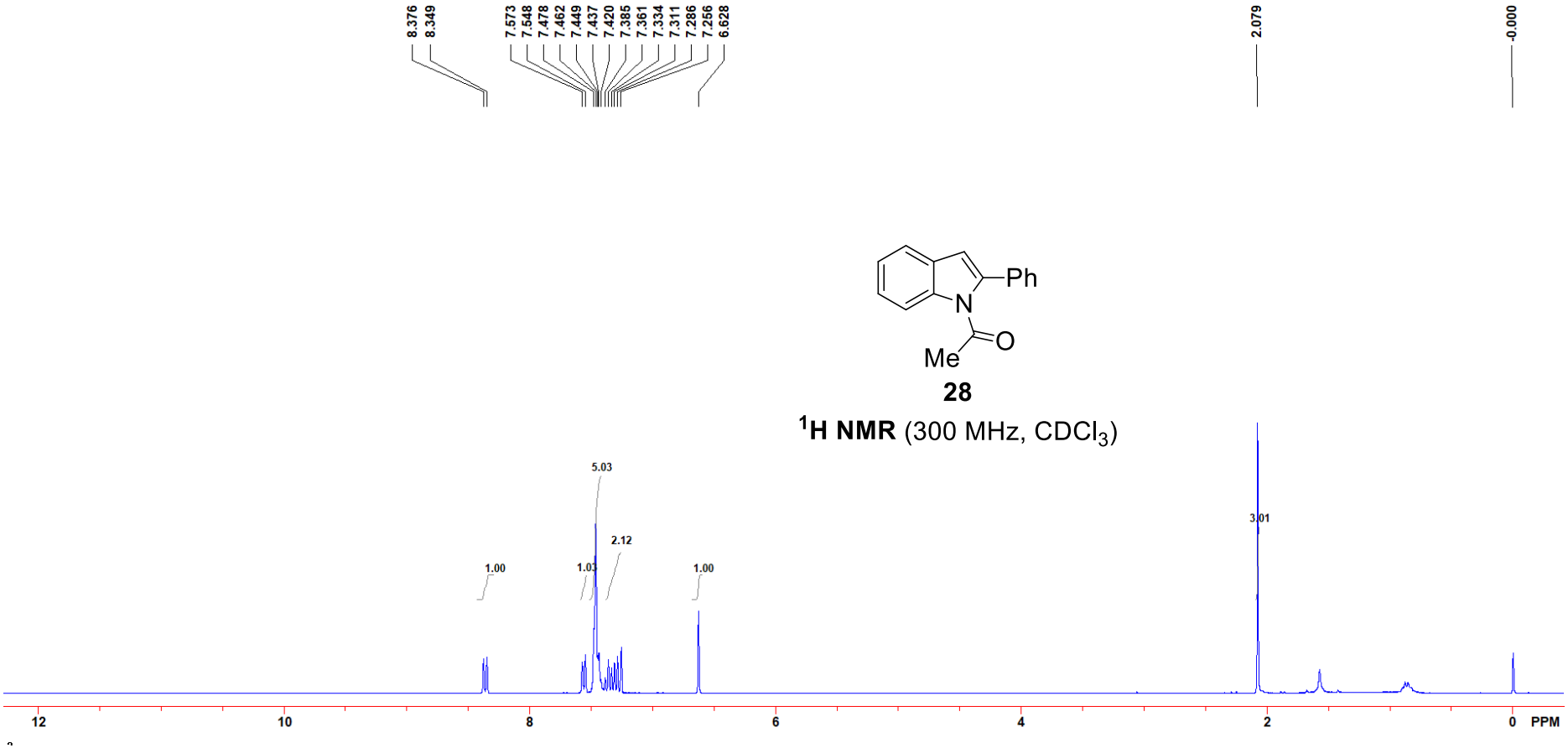
^{1}H NMR (300 MHz, $CDCl_3$)

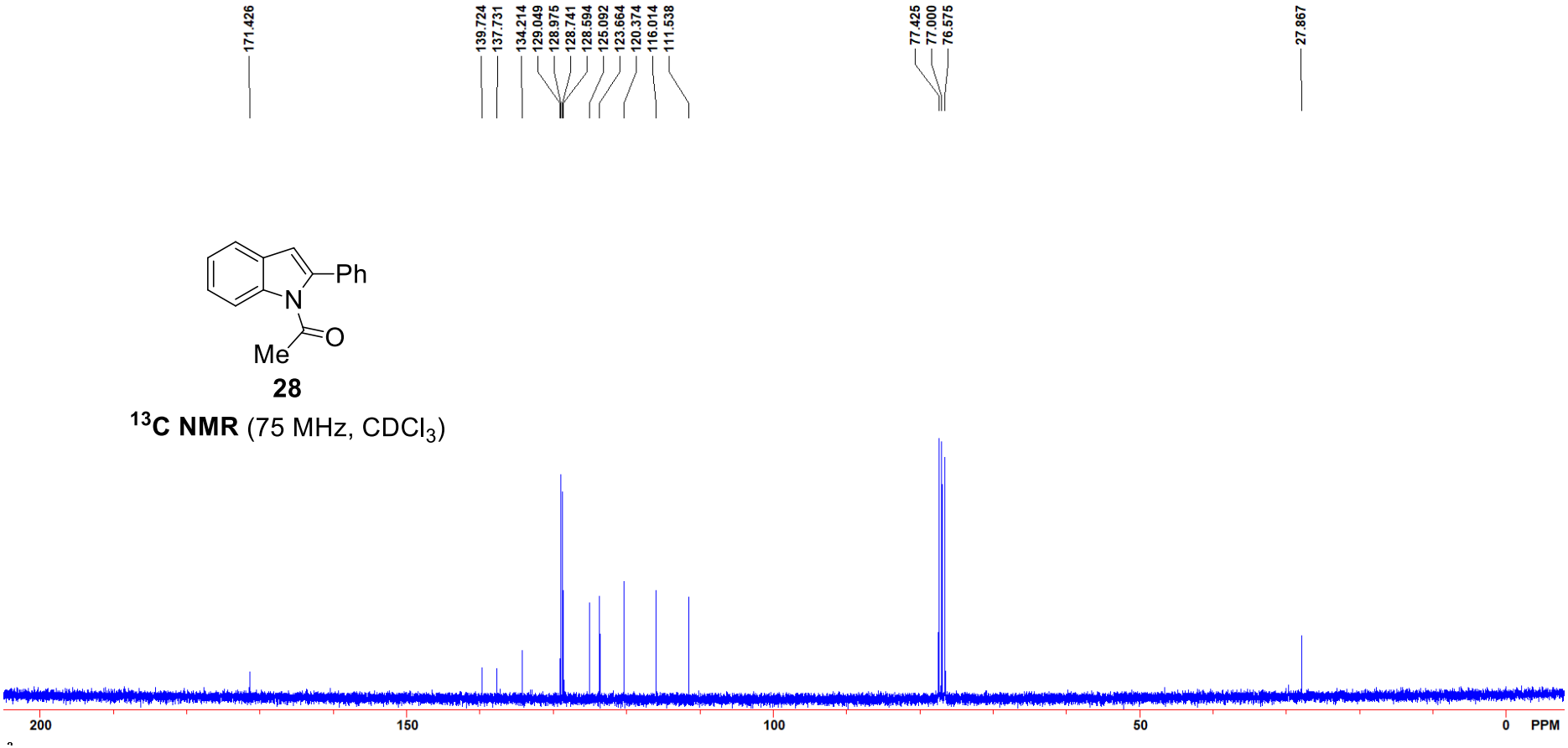




27 **27'**

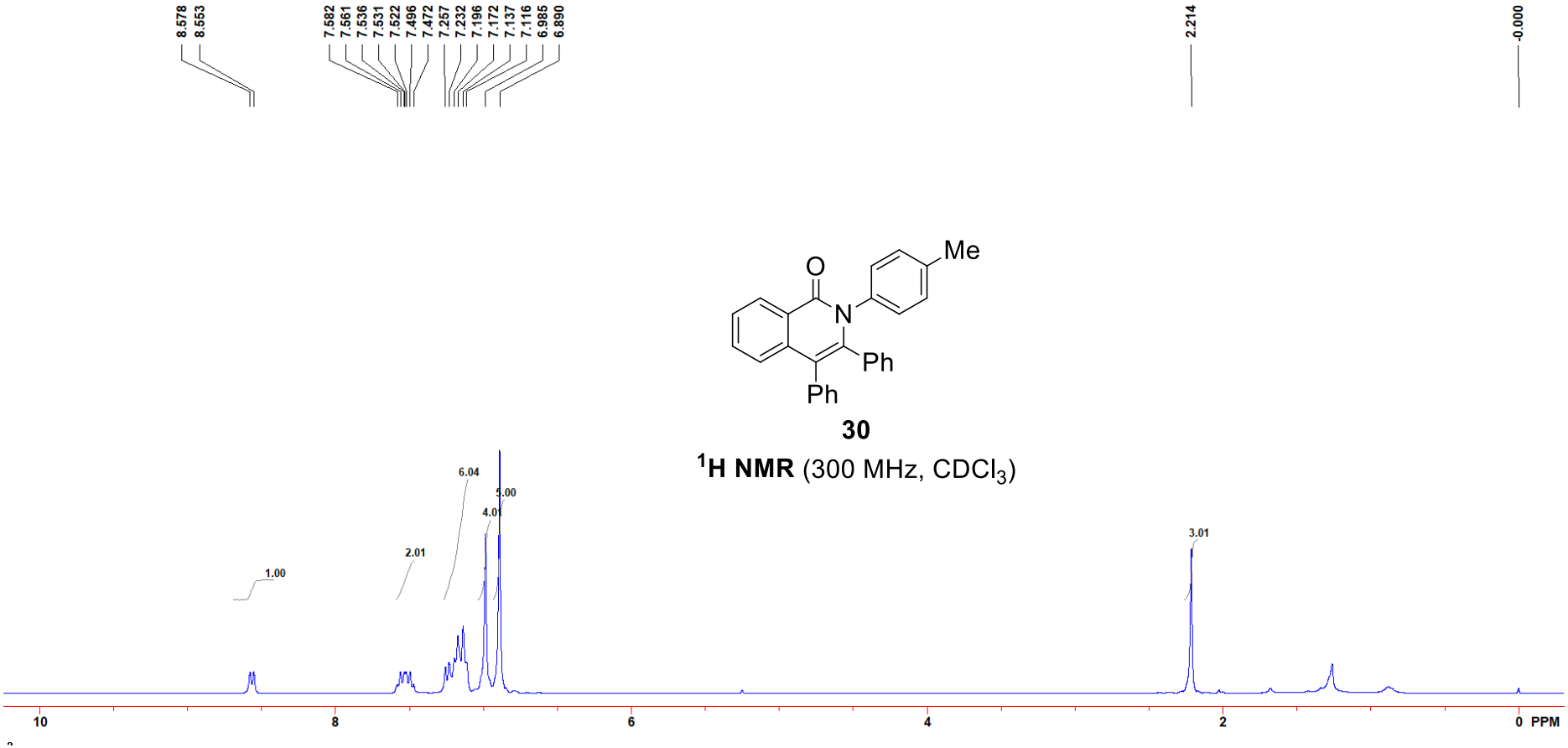
^{13}C NMR (75 MHz, $CDCl_3$)


8.376
8.349
7.573
7.548
7.478
7.462
7.449
7.437
7.420
7.385
7.361
7.334
7.311
7.286
7.256
6.628
2.079
-0.000
Ph
N
O
Me
28
1H NMR (300 MHz, CDCl3)
5.03
2.12
1.00
1.03
1.00
3.01
12
10
8
6
4
2
0
PPM



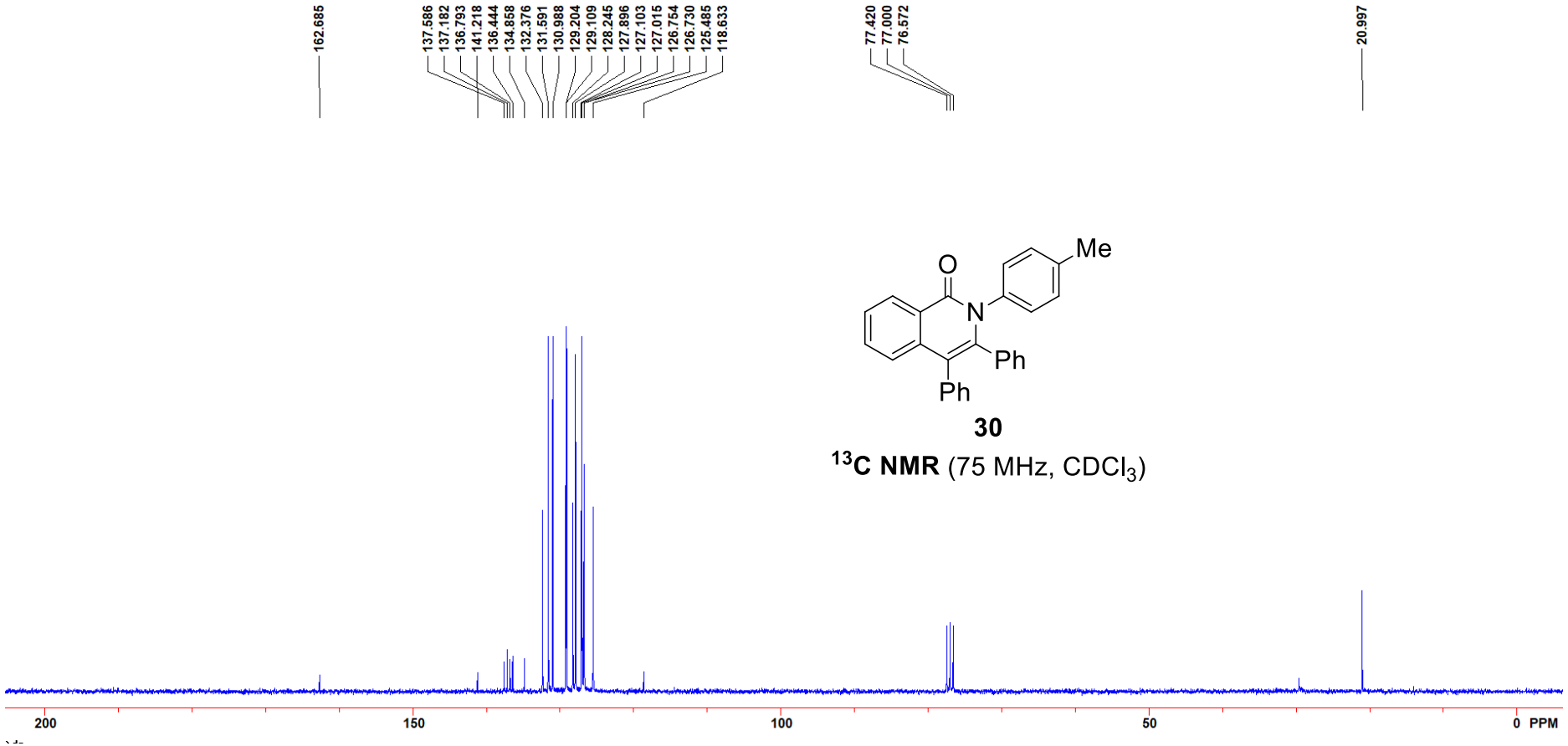
28

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



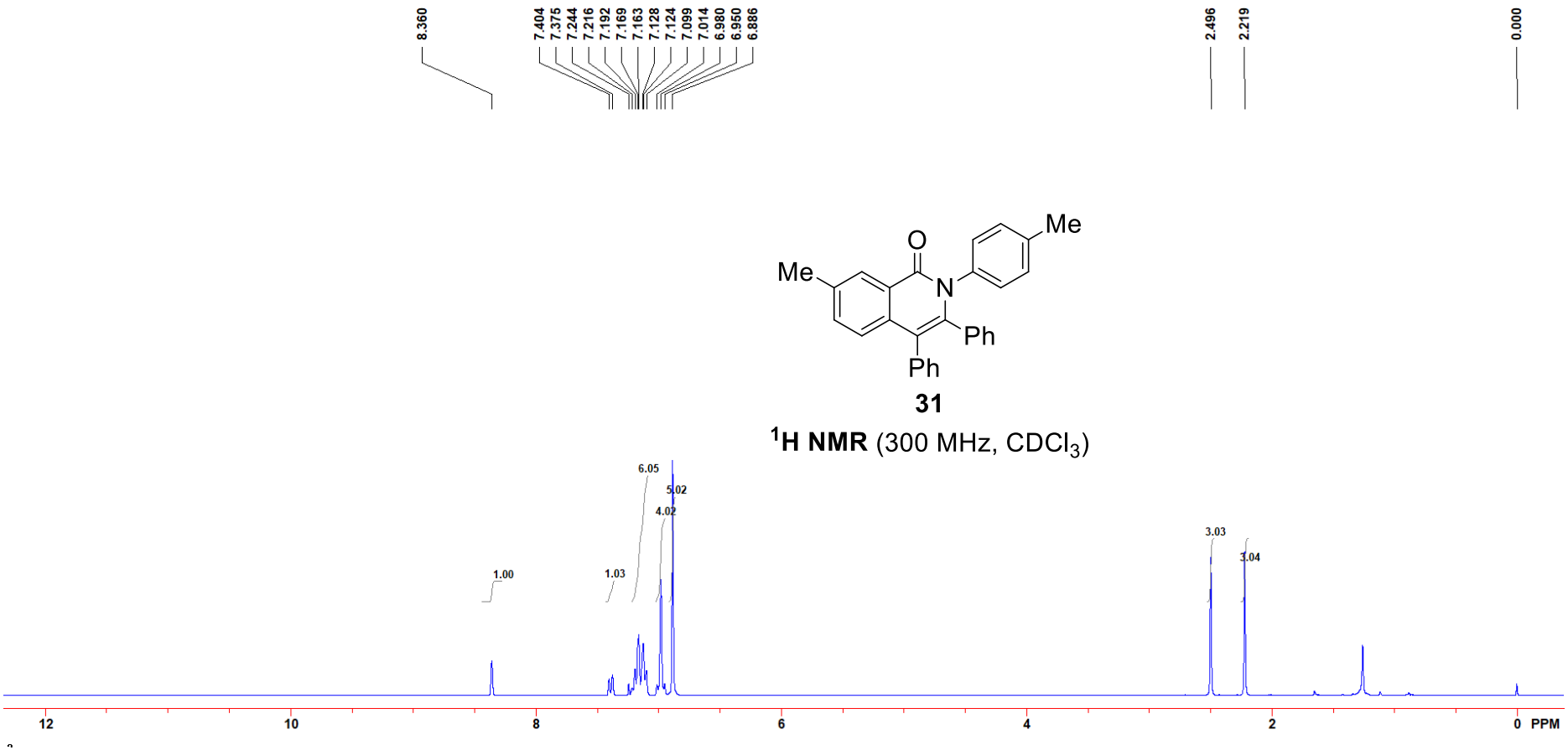
30

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



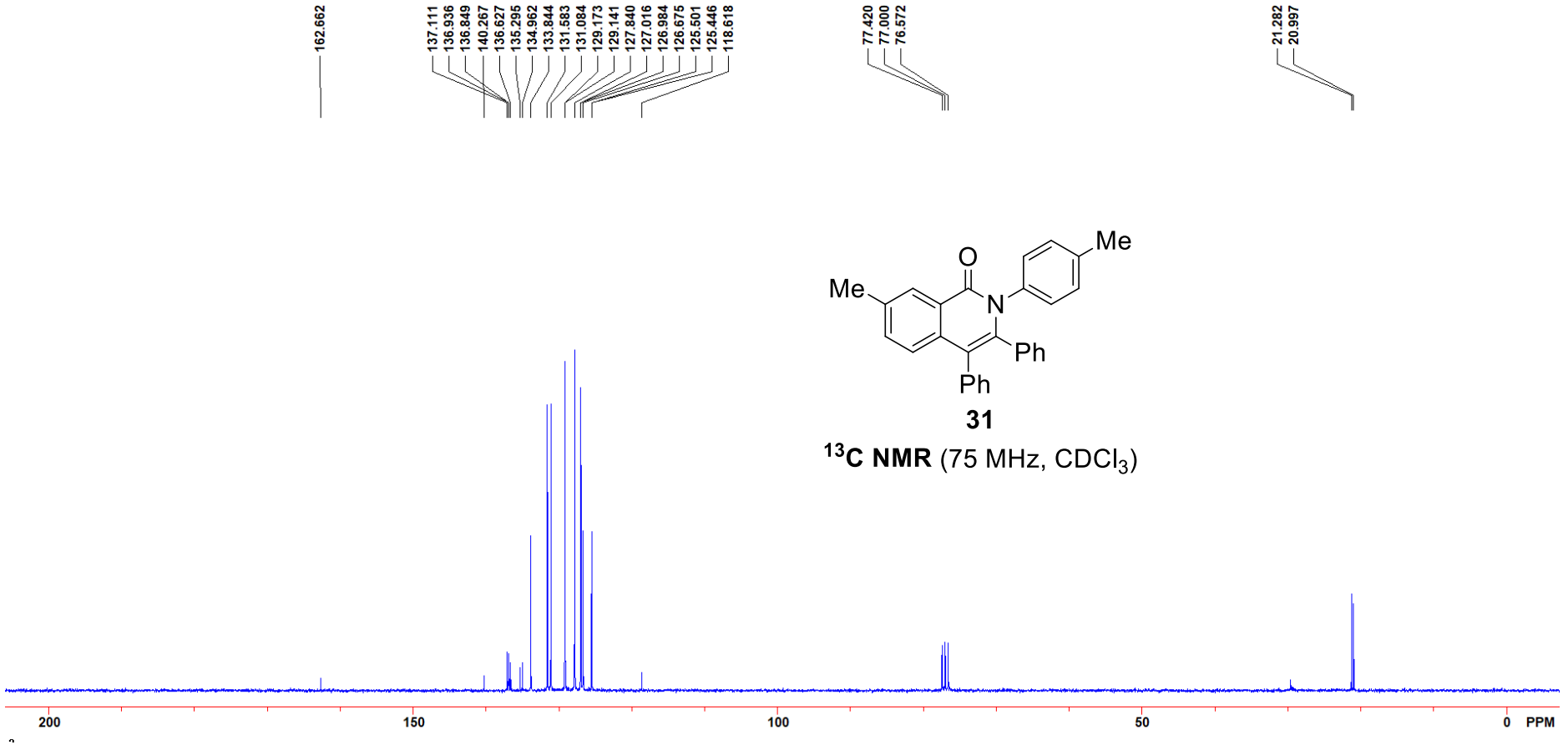
30

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



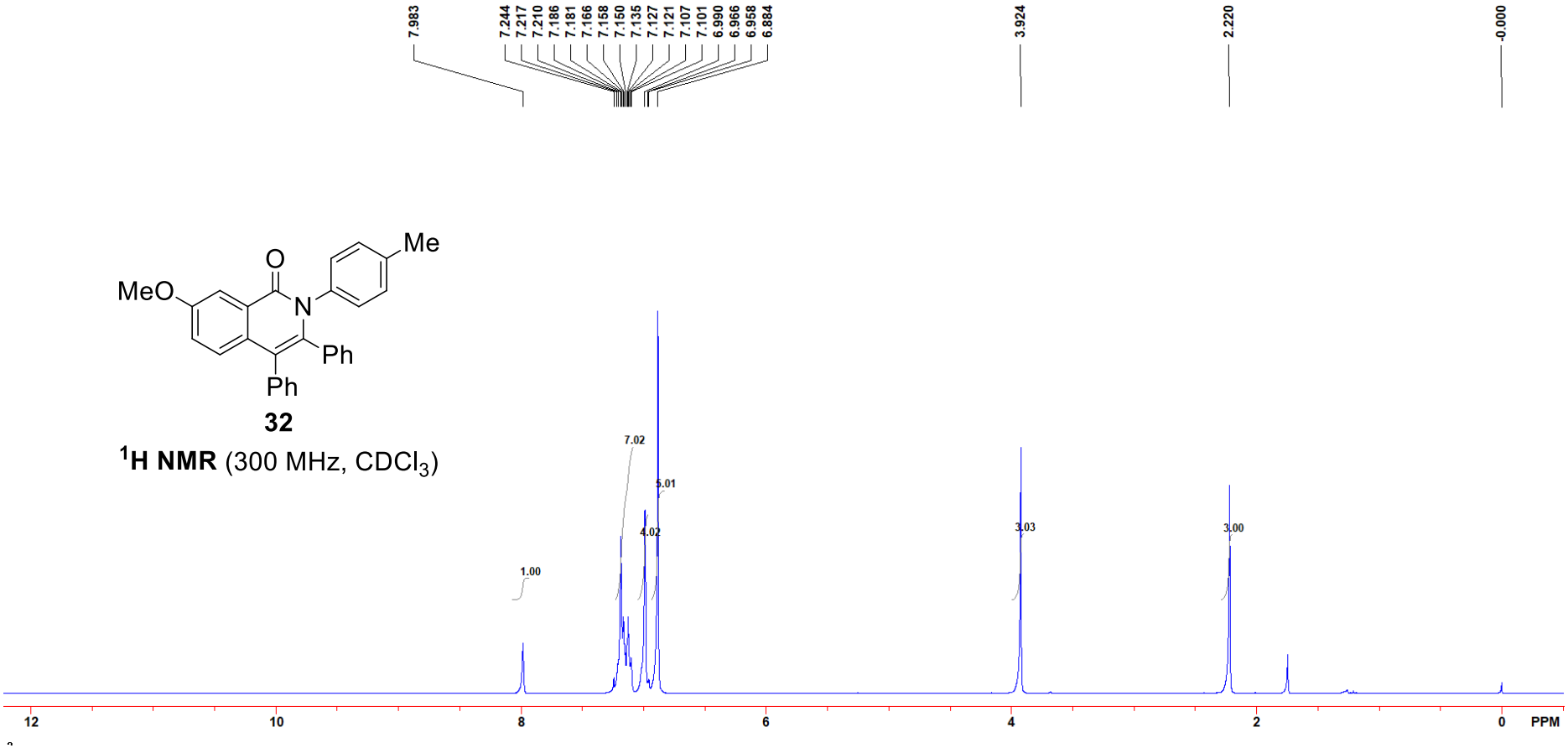
31

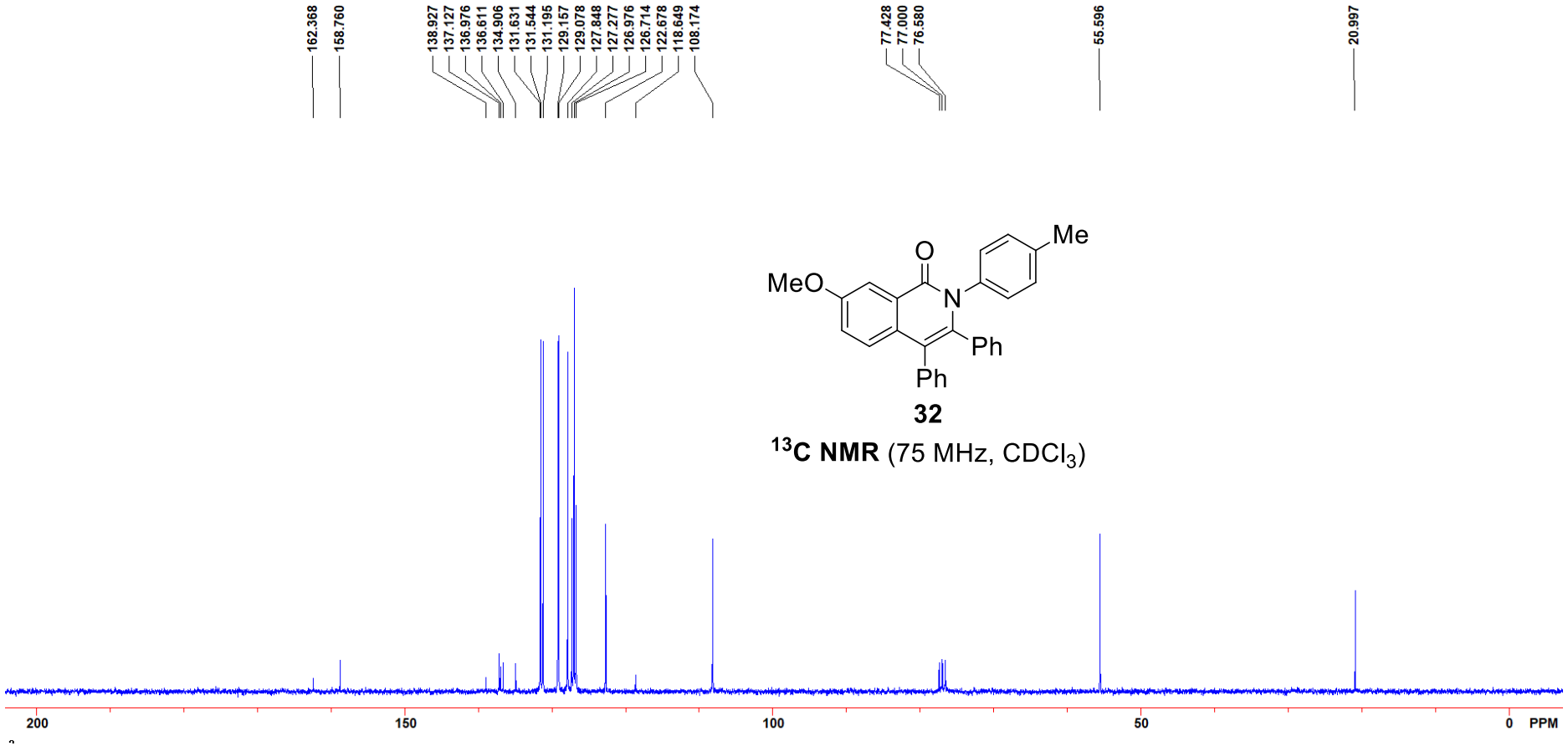
^{1}H NMR (300 MHz, $CDCl_3$)



31

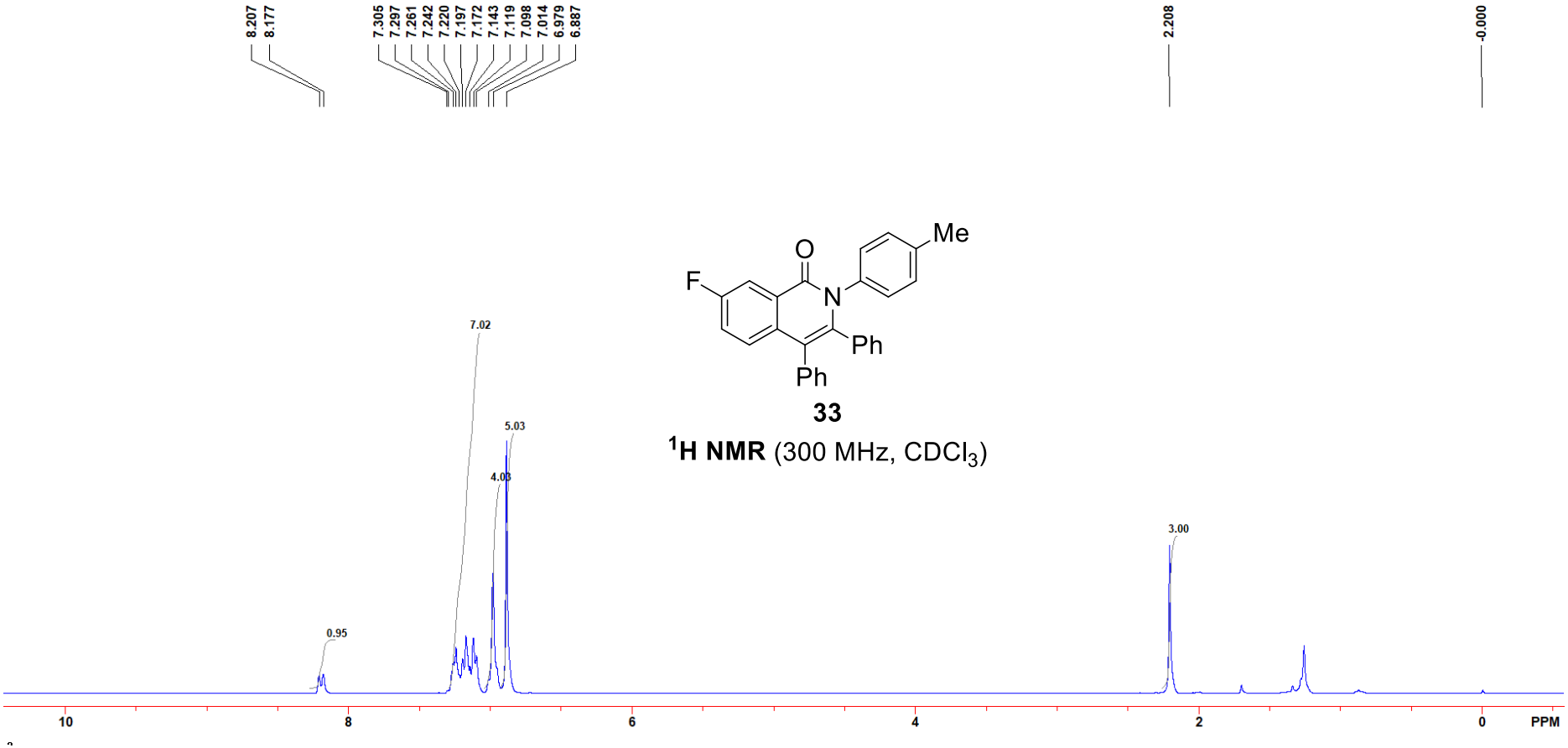
^{13}C NMR (75 MHz, $CDCl_3$)


7.983
7.244
7.217
7.210
7.186
7.181
7.166
7.158
7.150
7.135
7.127
7.121
7.107
7.101
6.990
6.966
6.958
6.884
3.924
2.220
-0.000
MeO
O
N
Me
Ph
Ph
32
1H NMR (300 MHz, CDCl3)
1.00
7.02
5.01
4.02
3.03
3.00
12
10
8
6
4
2
0
PPM



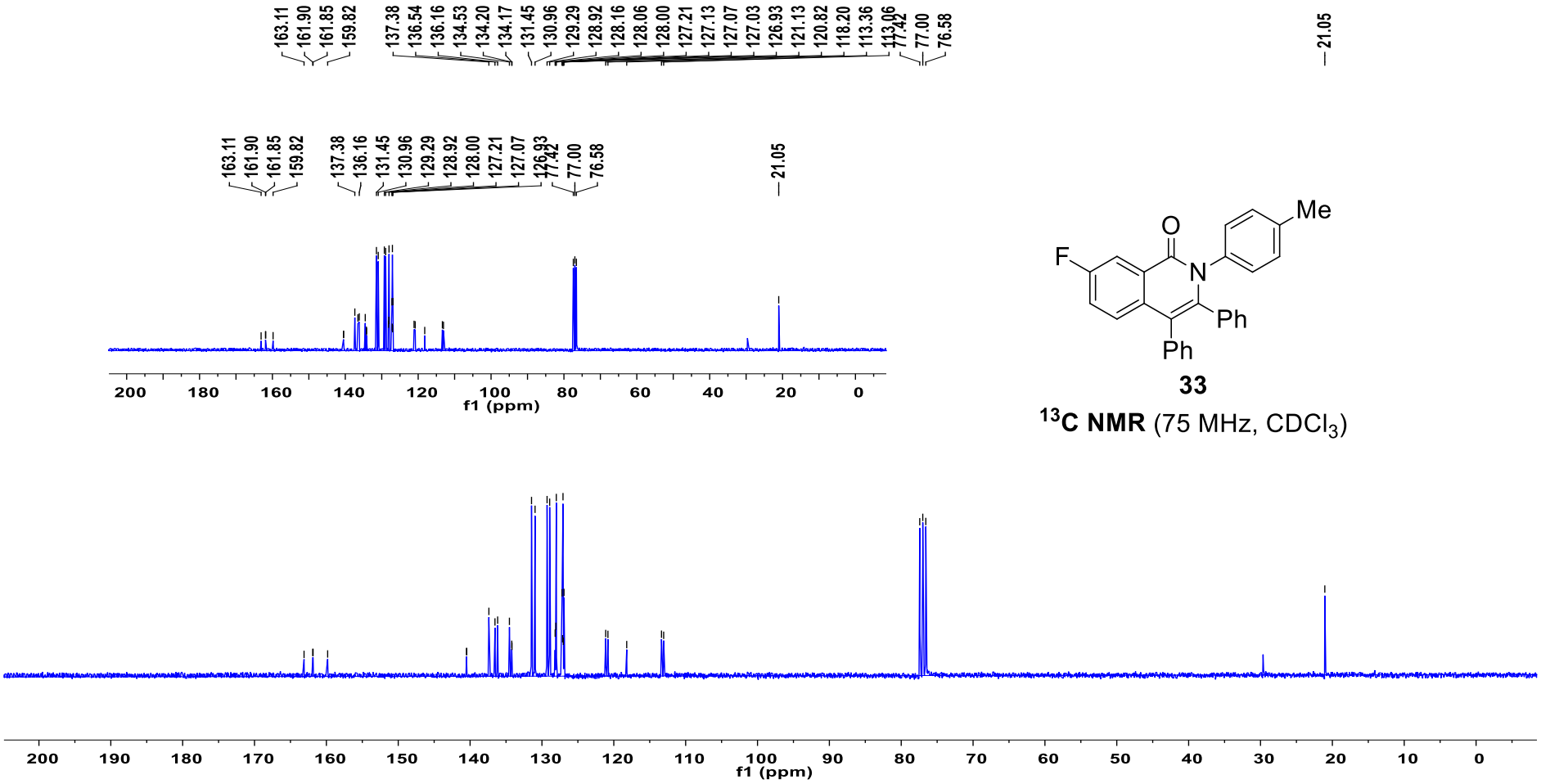
32

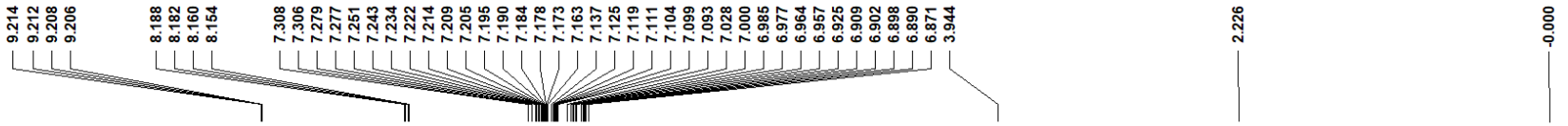
^{13}C NMR (75 MHz, $CDCl_3$)

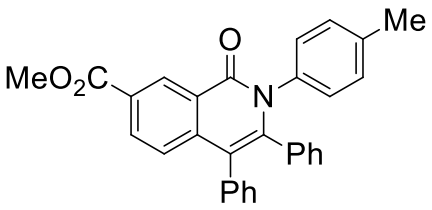


33

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

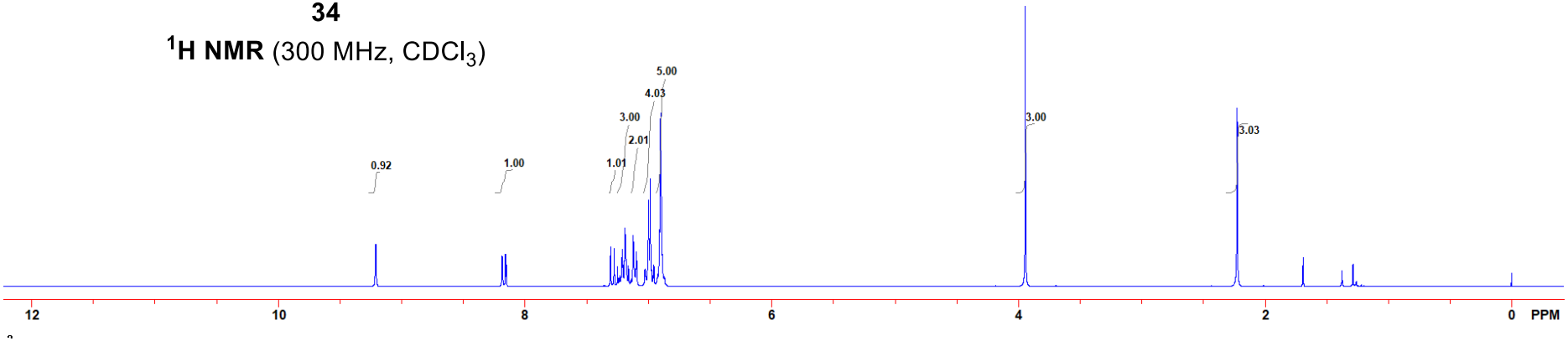

163.11
161.90
161.85
159.82
137.38
136.54
136.16
134.53
134.20
134.17
131.45
130.96
129.29
128.92
128.16
128.06
128.00
127.21
127.13
127.07
127.03
126.93
121.13
120.82
118.20
113.36
113.06
77.42
77.00
76.58
21.05
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
O
N
F
Me
Ph
Ph
33
13C NMR (75 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

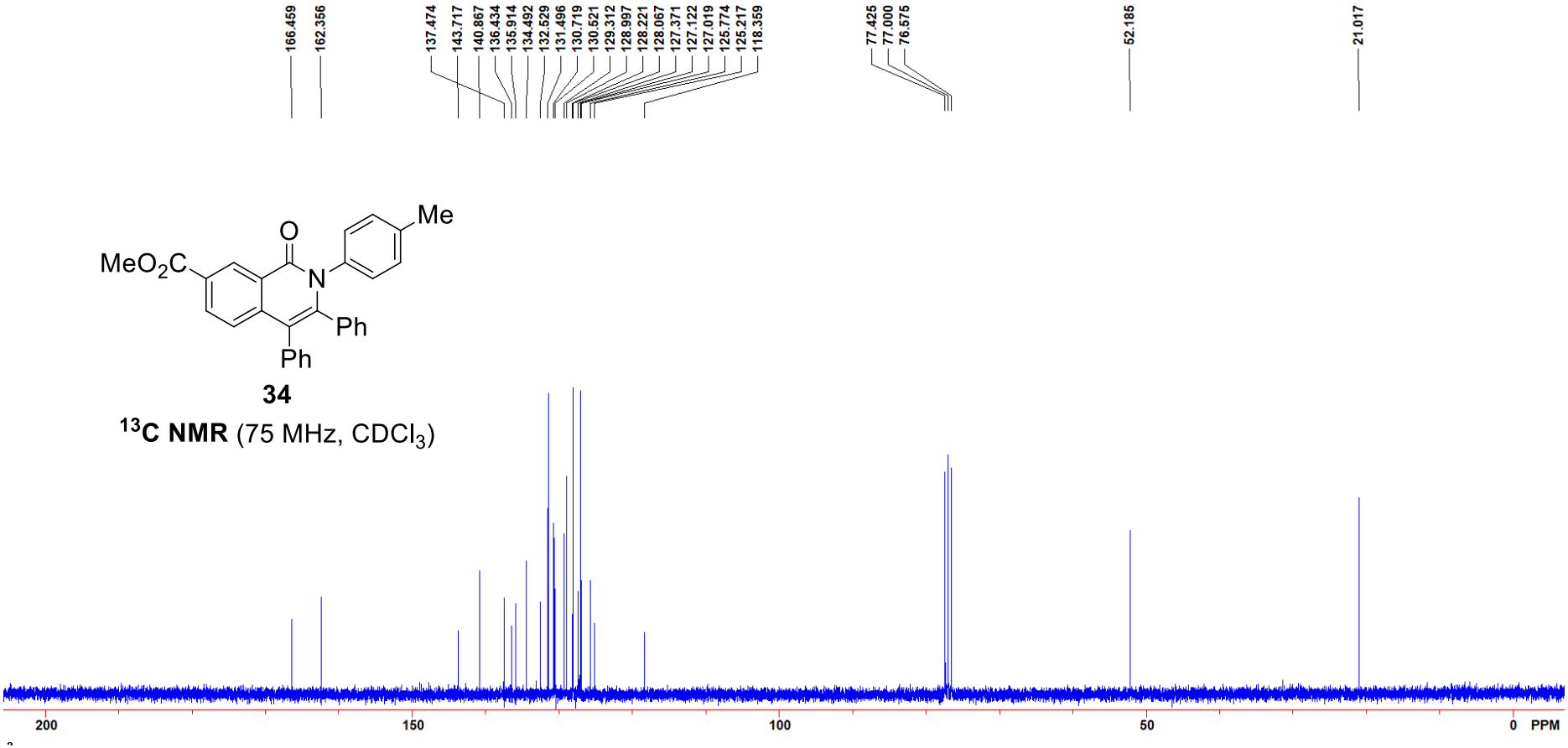




34

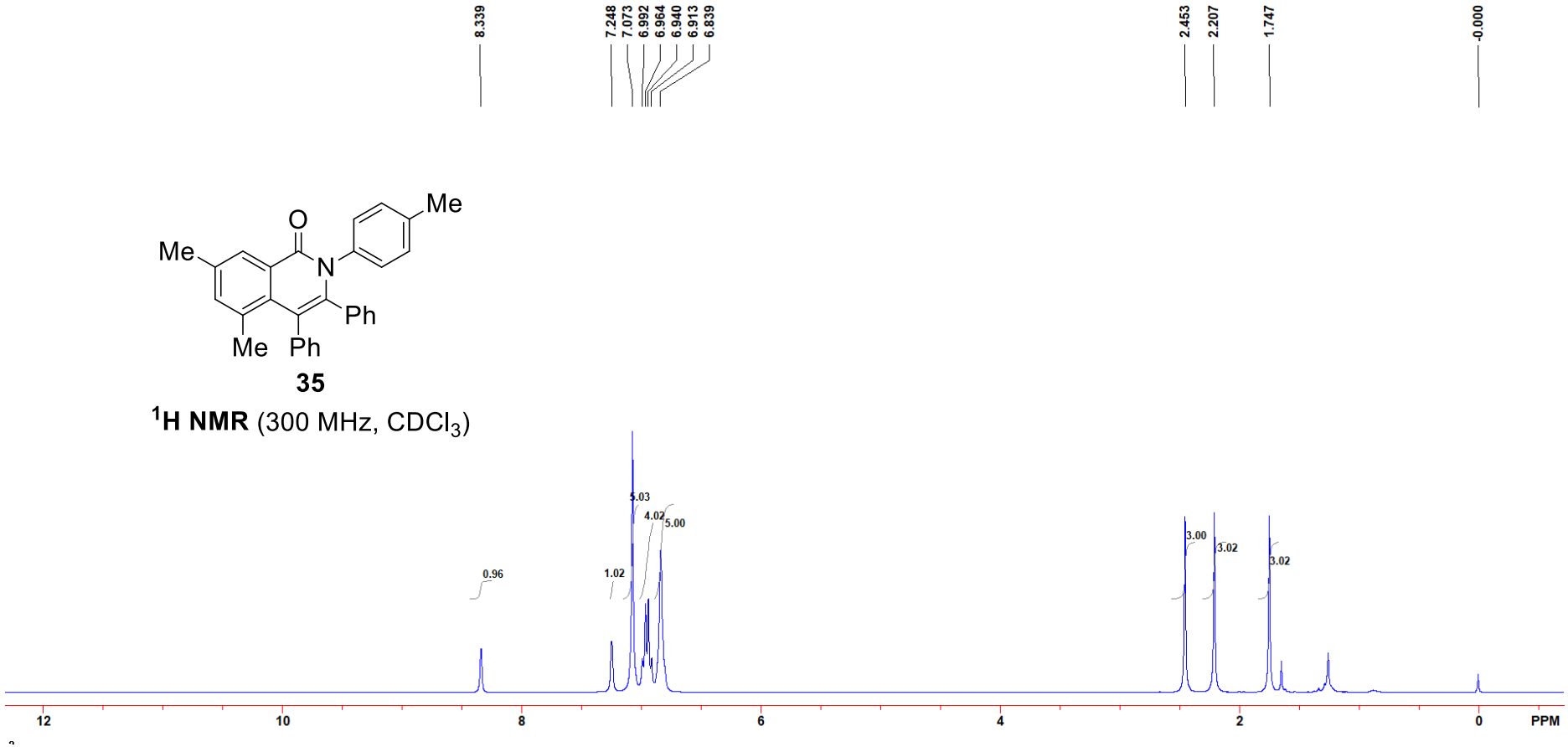
^{1}H NMR (300 MHz, $CDCl_3$)



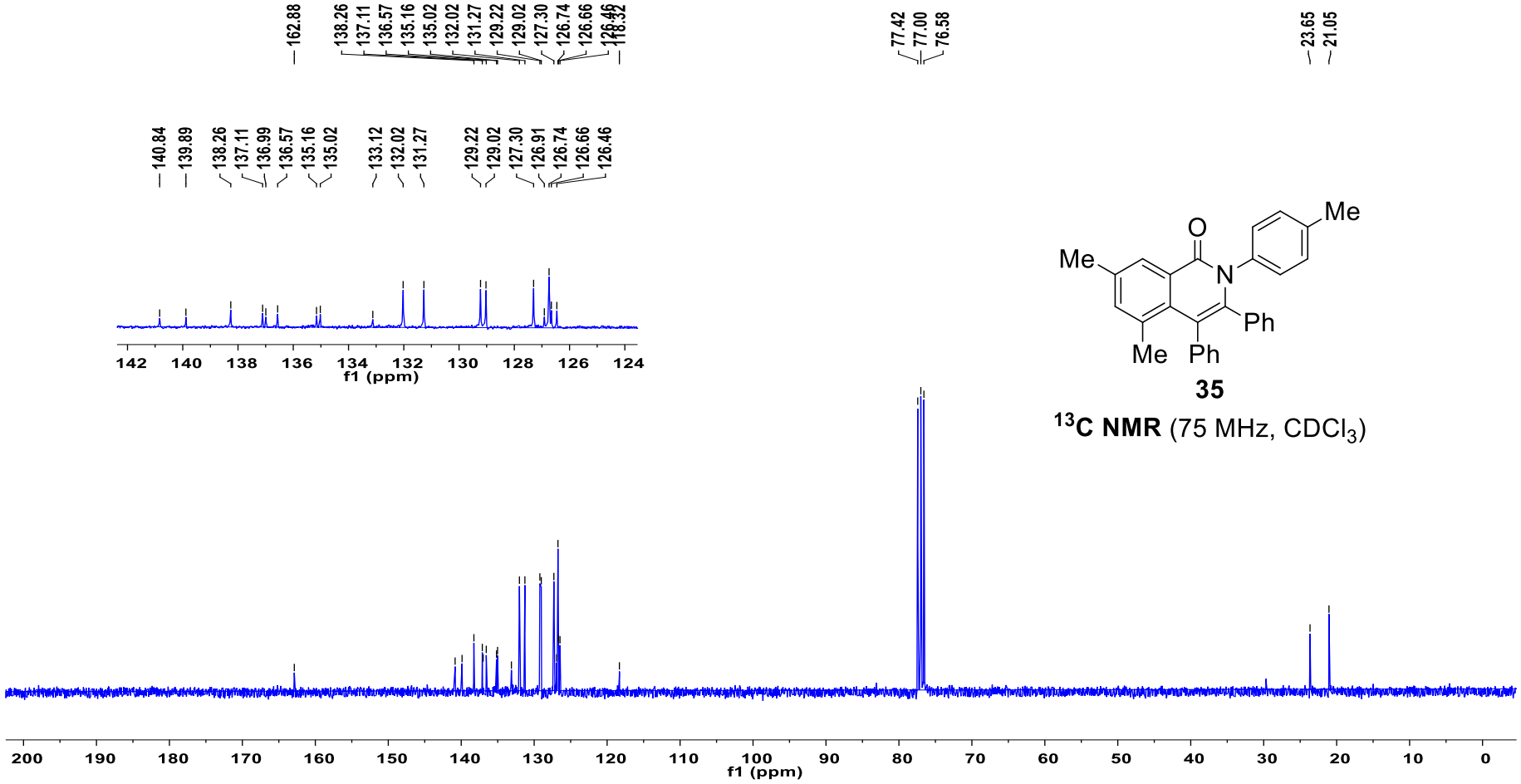


34

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

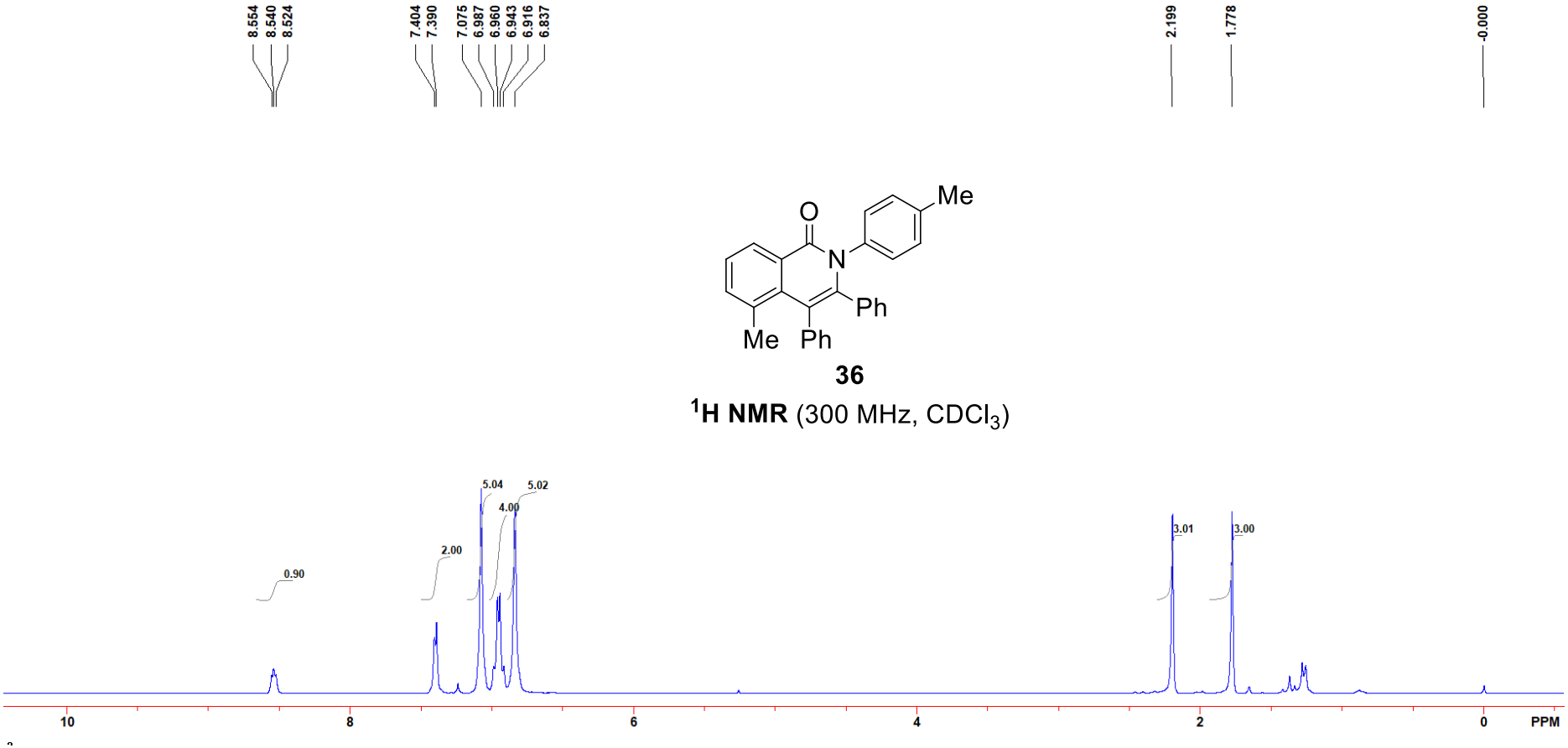


^{1}H NMR (300 MHz, $CDCl_3$)



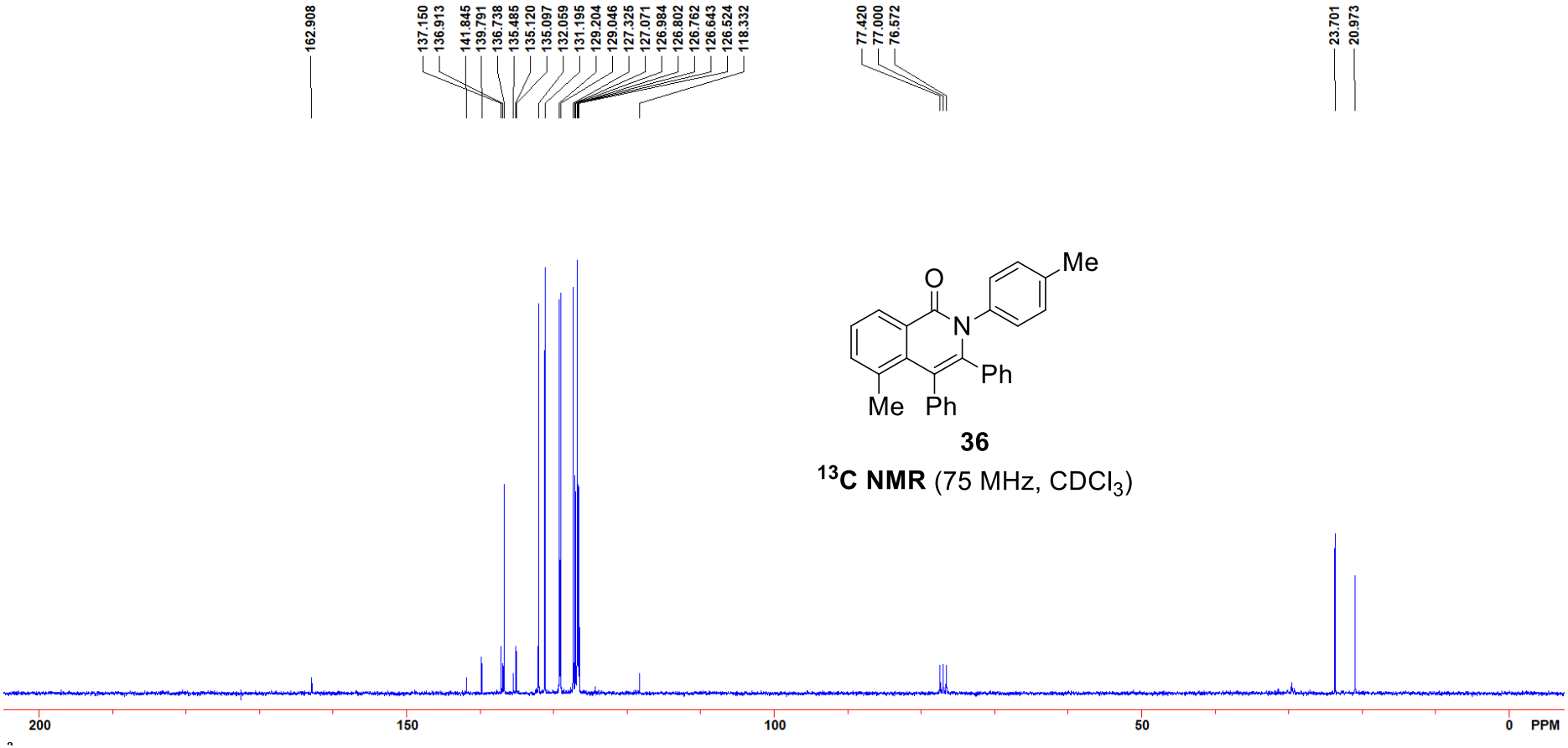
35

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



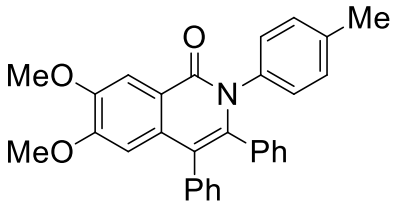
36

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



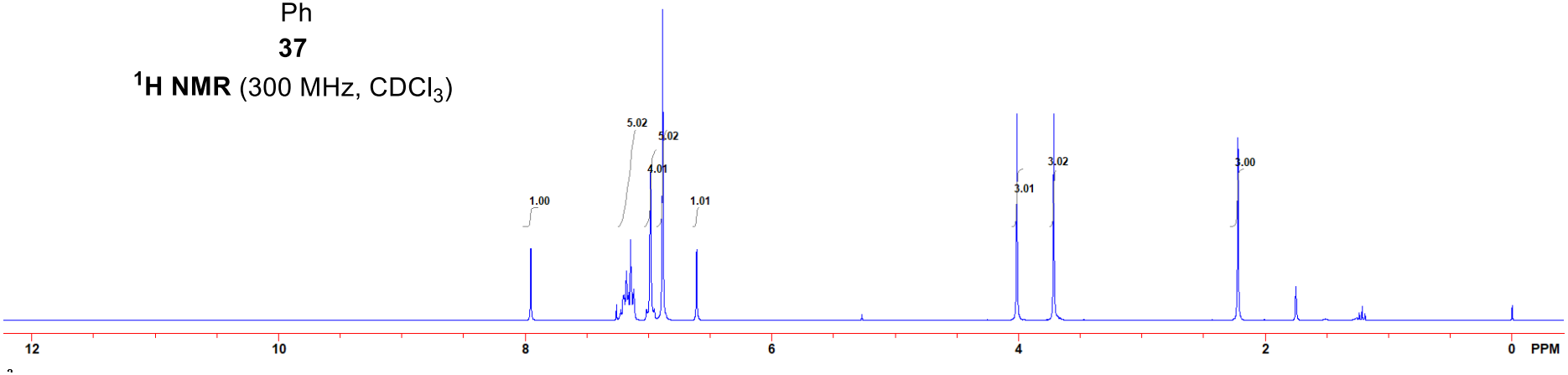
36

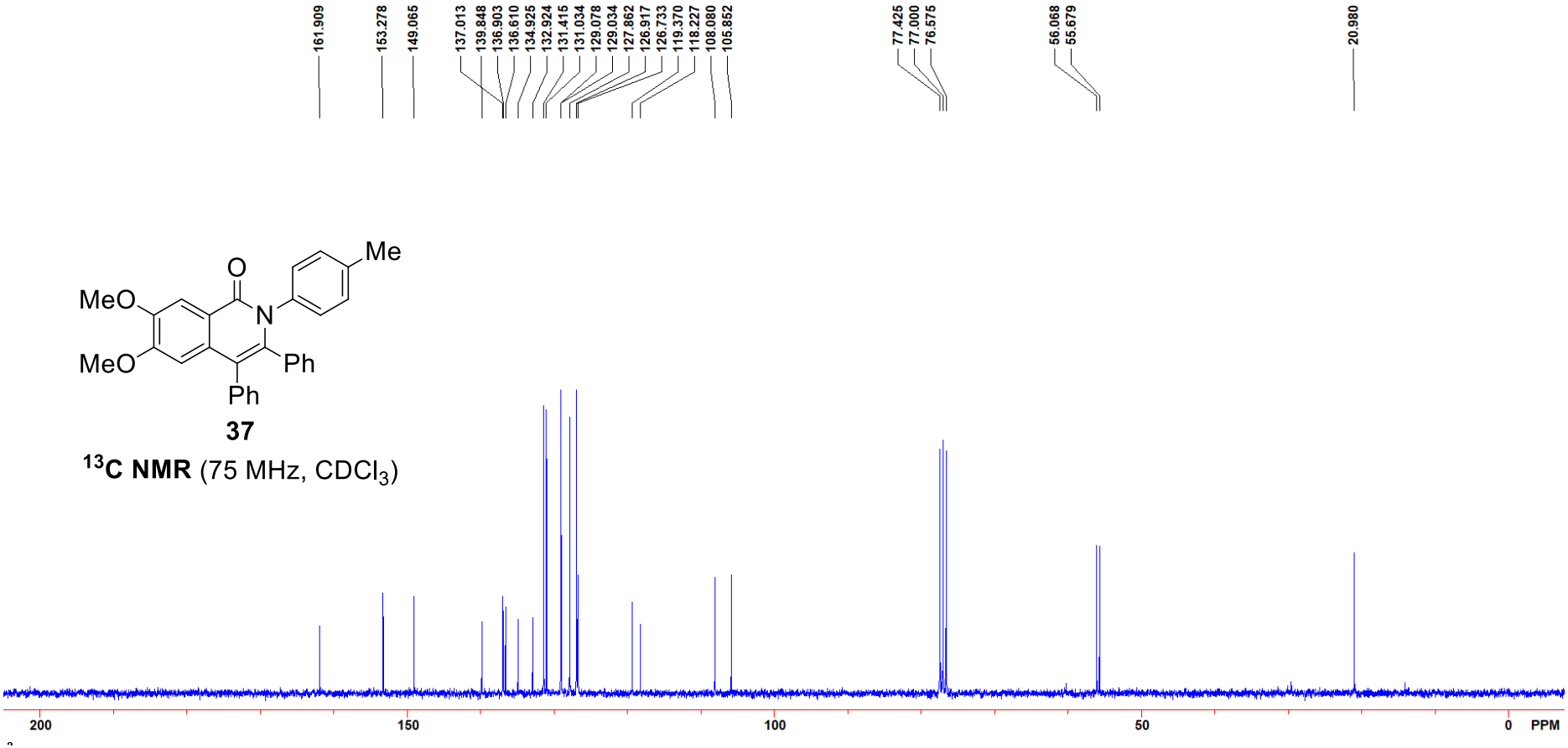
^{13}C NMR (75 MHz, $CDCl_3$)



37

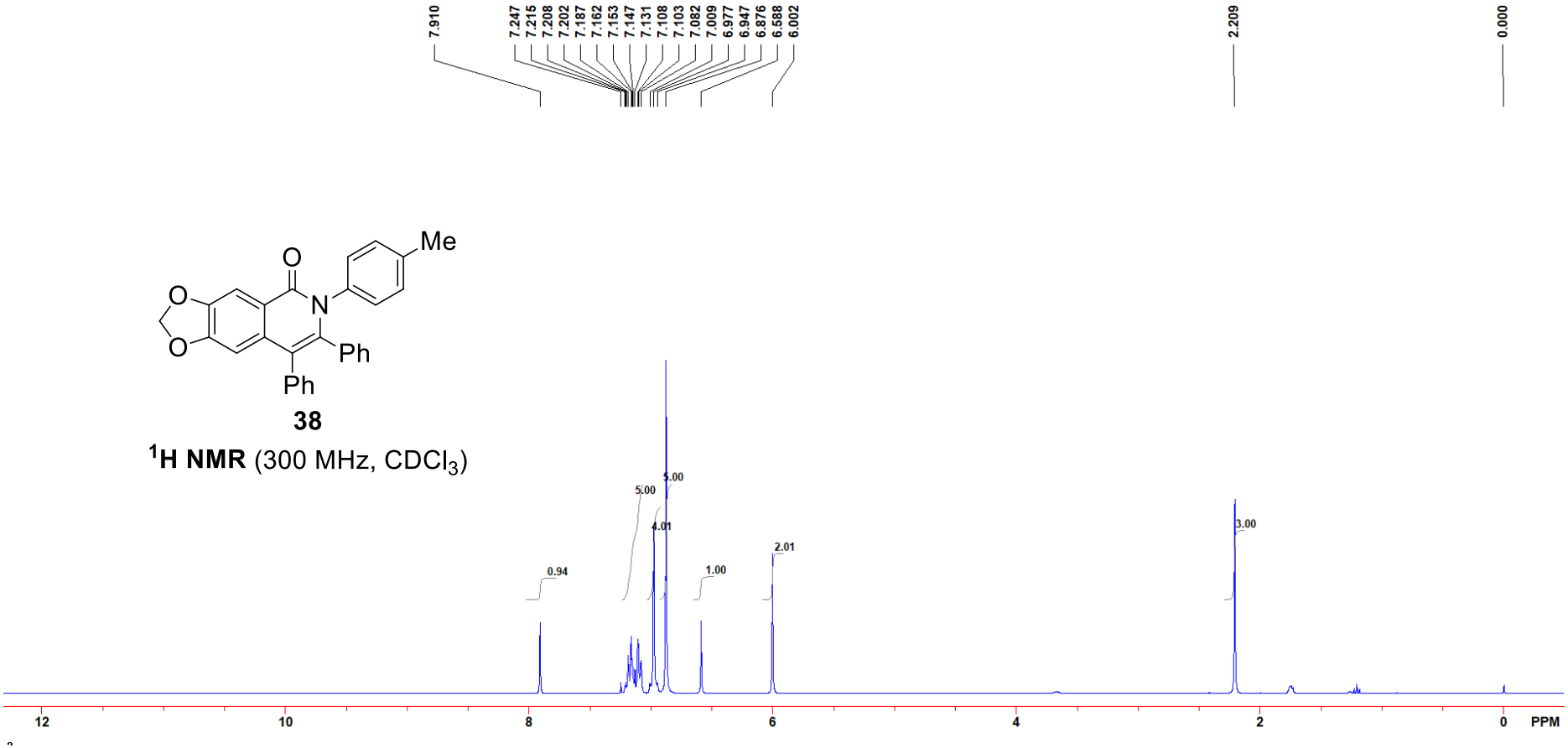
^{1}H NMR (300 MHz, $CDCl_3$)



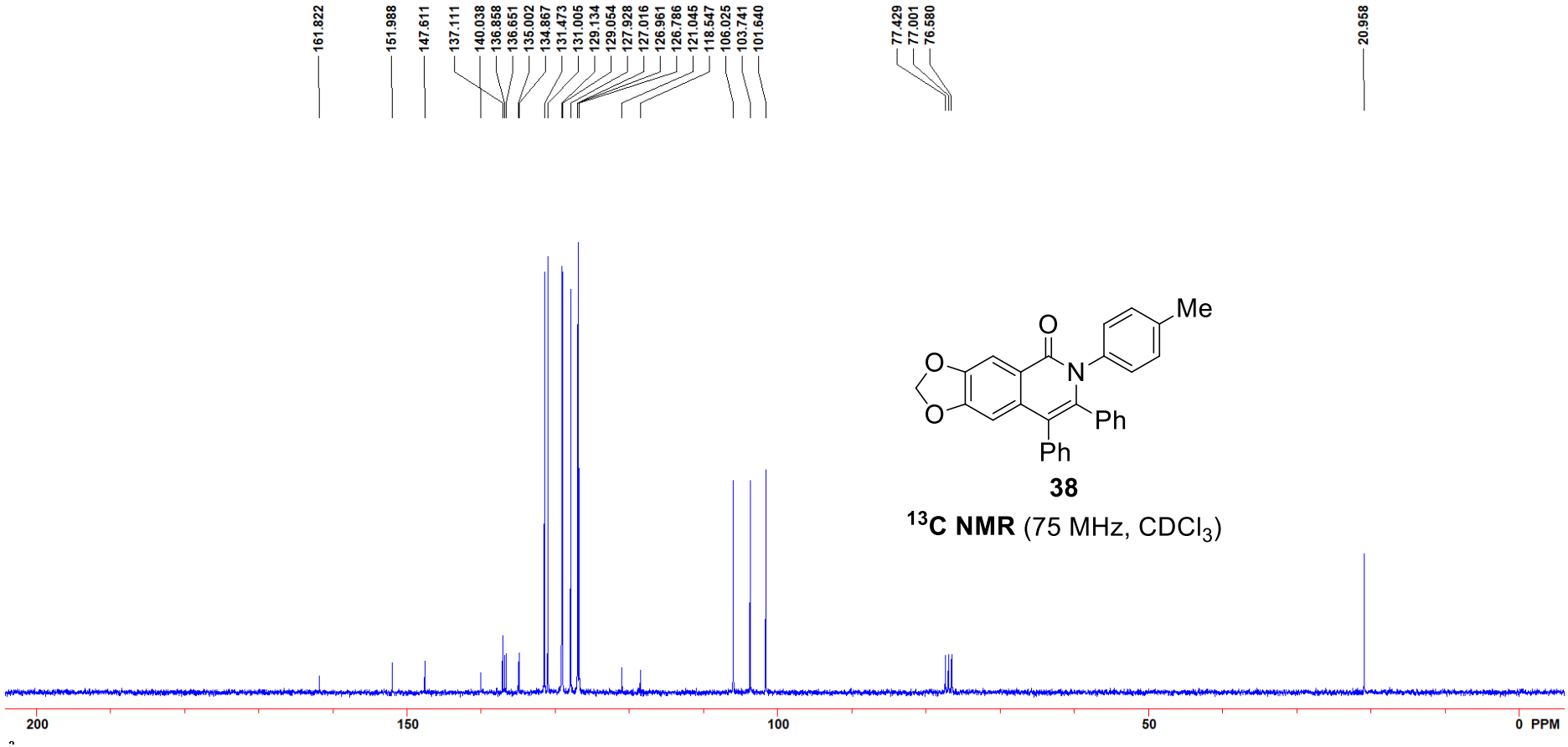


37

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

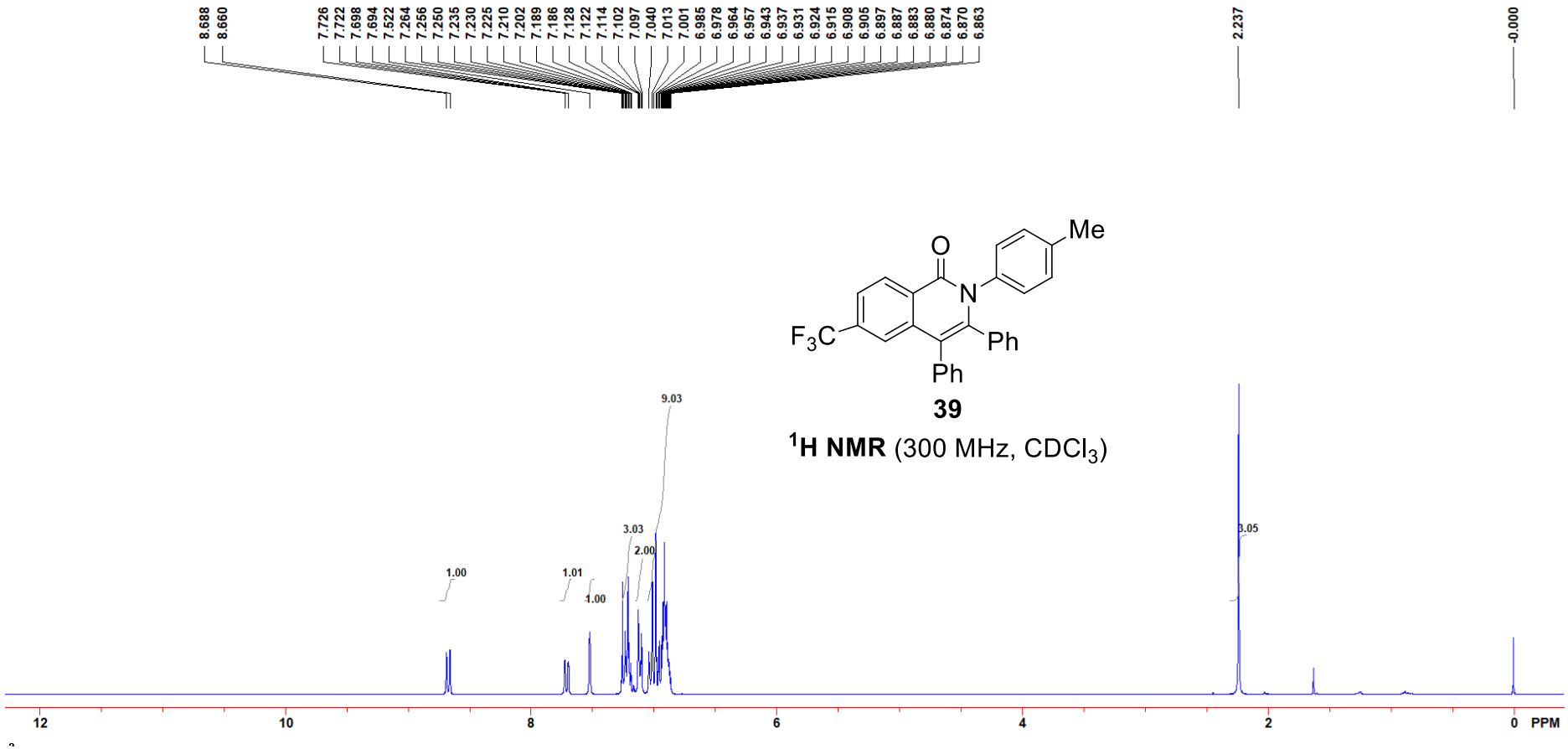


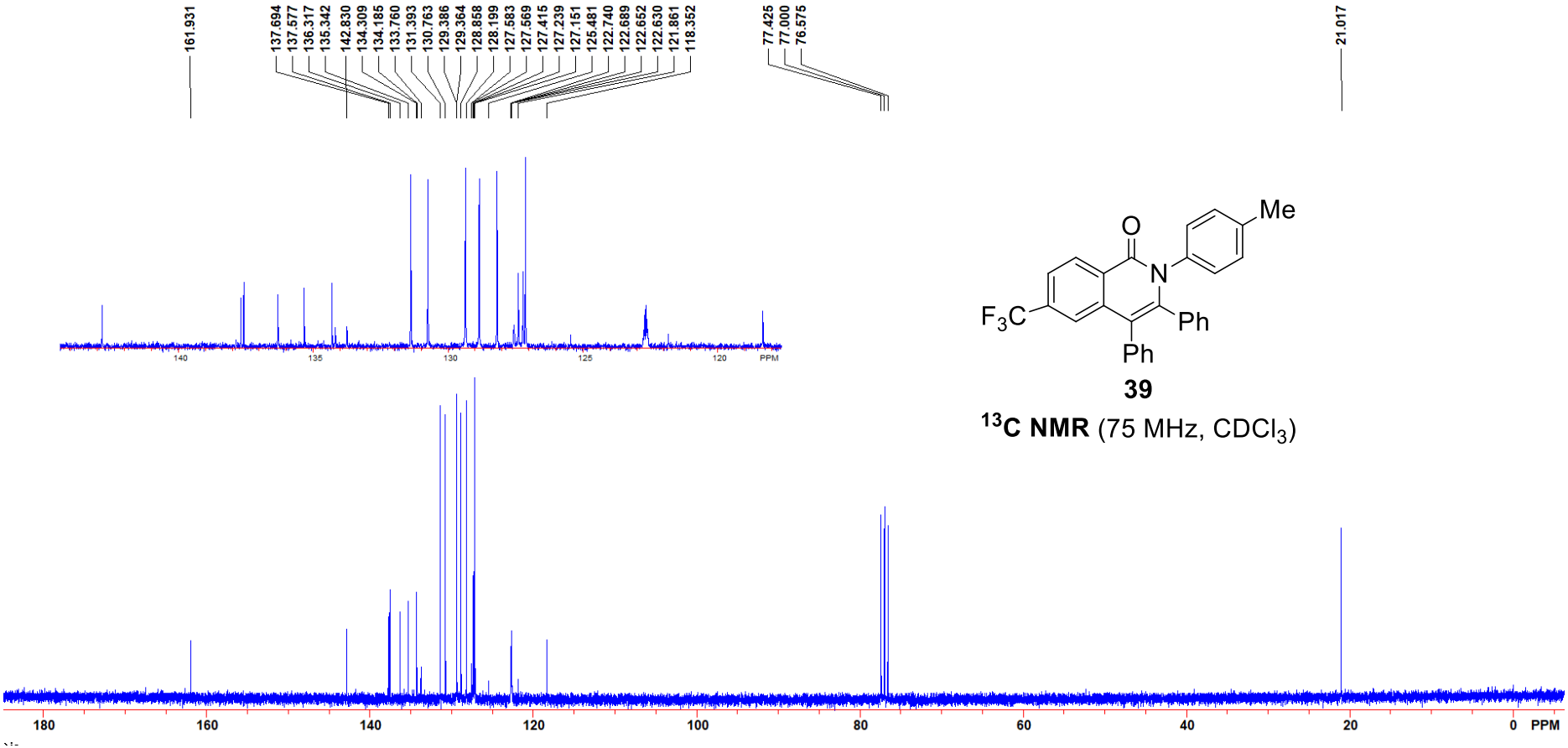
^{1}H NMR (300 MHz, $CDCl_3$)



38

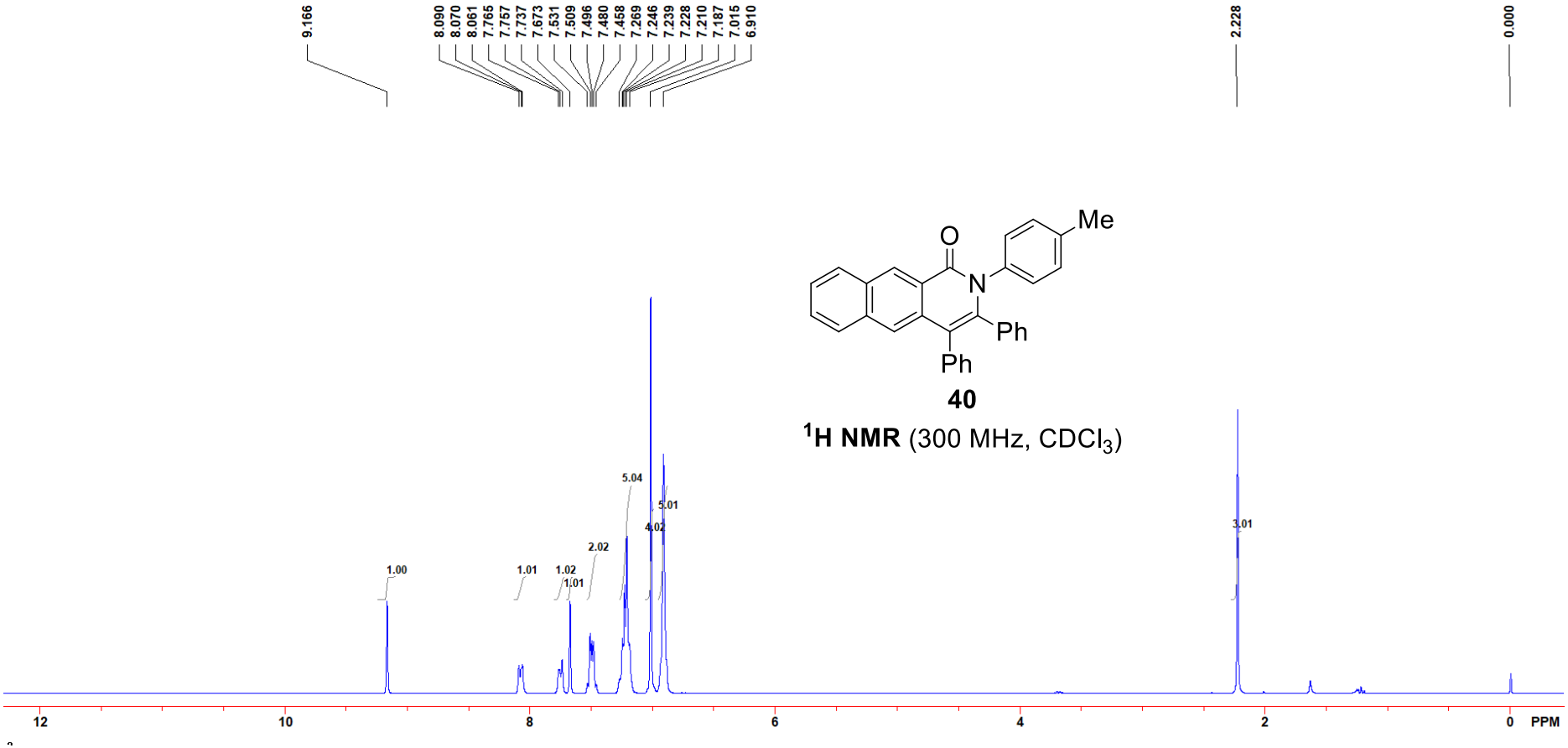
^{13}C NMR (75 MHz, $CDCl_3$)

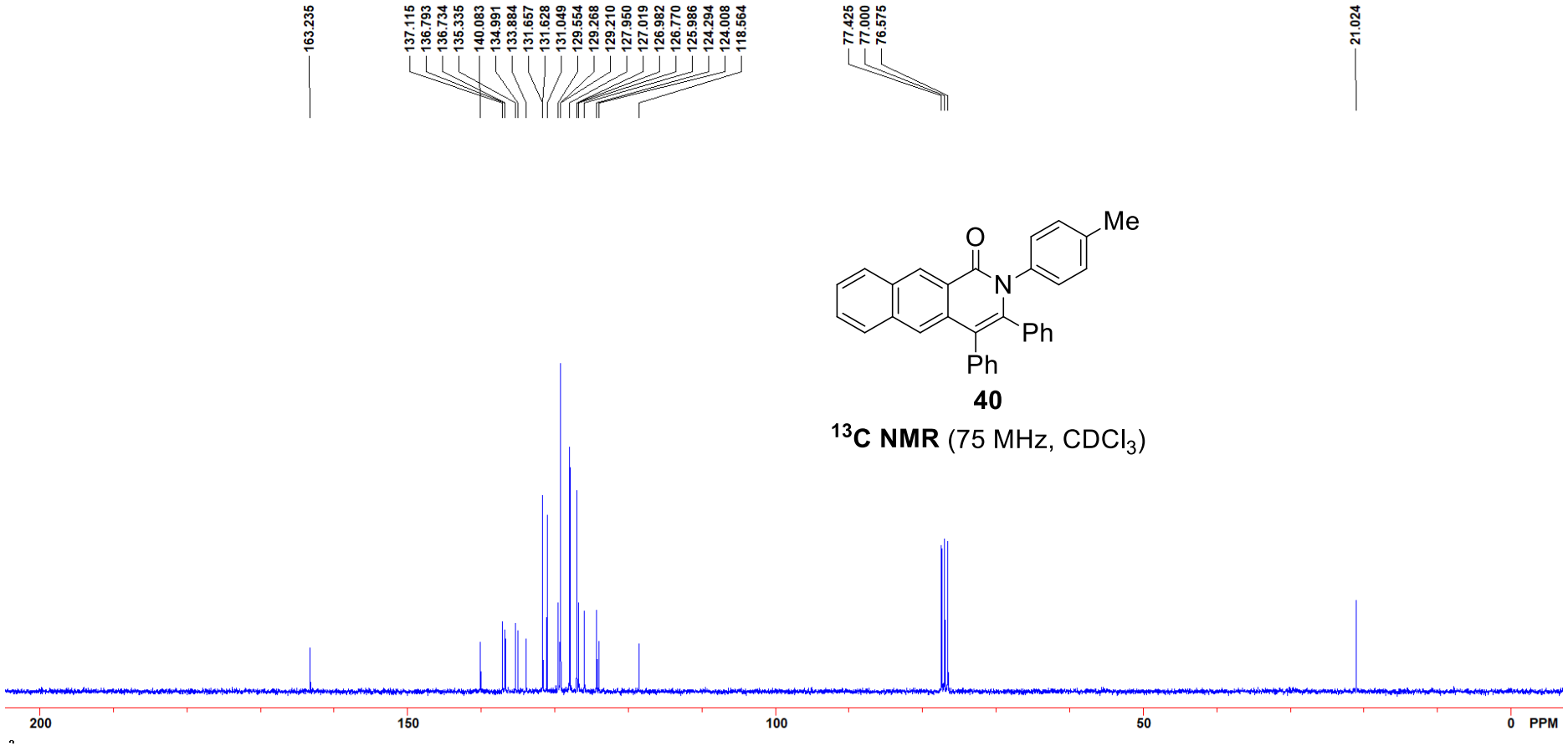

8.688
8.660
7.726
7.722
7.698
7.694
7.522
7.264
7.256
7.250
7.235
7.230
7.225
7.210
7.202
7.189
7.186
7.128
7.122
7.114
7.102
7.097
7.040
7.013
7.001
6.985
6.978
6.964
6.957
6.943
6.937
6.931
6.924
6.915
6.908
6.905
6.897
6.887
6.883
6.880
6.874
6.870
6.863
2.237
-0.000
O
N
Me
F3C
Ph
Ph
39
1H NMR (300 MHz, CDCl3)
9.03
3.03
2.00
1.00
1.01
1.00
3.05
12
10
8
6
4
2
0
PPM



39

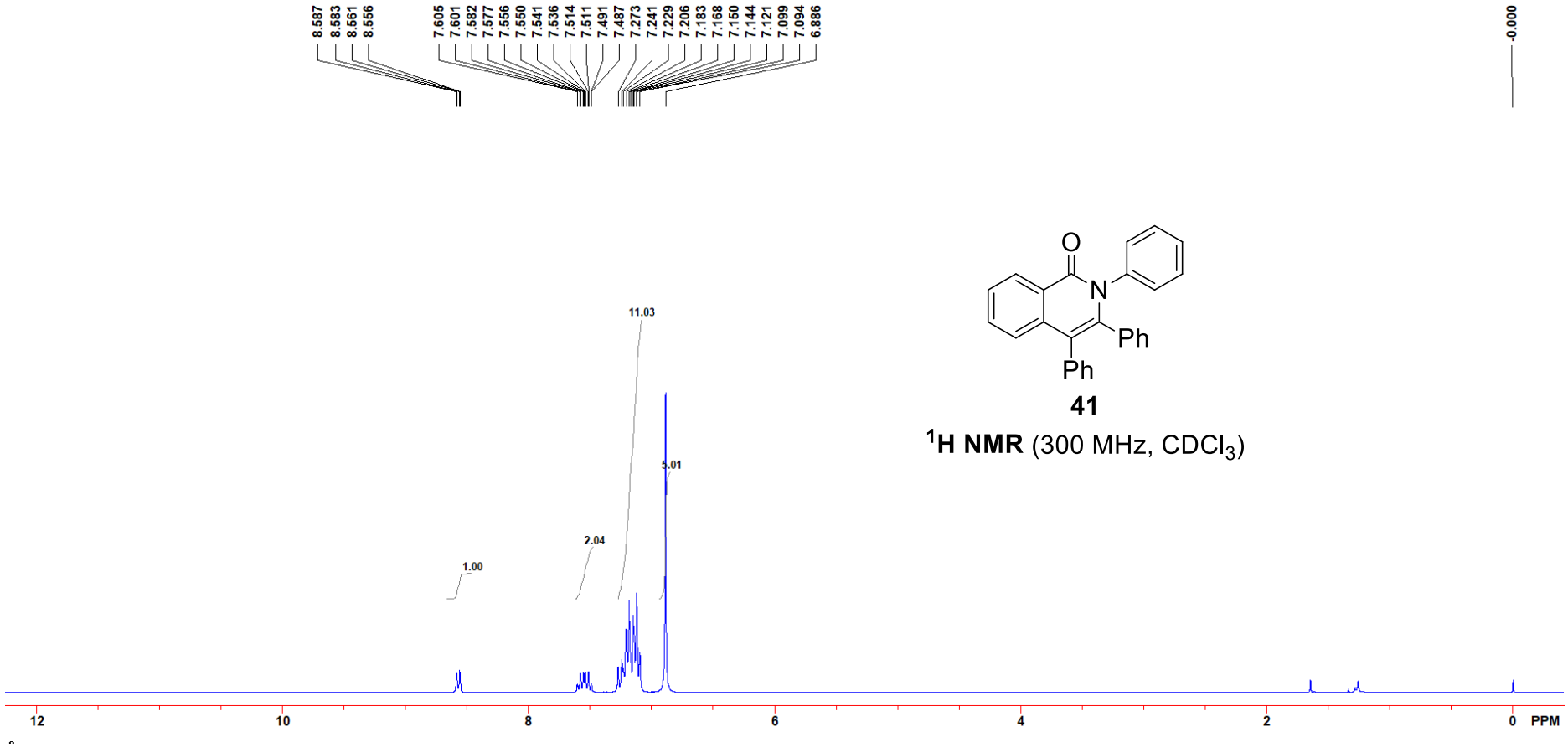
^{13}C NMR (75 MHz, $CDCl_3$)


9.166
8.090
8.070
8.061
7.765
7.757
7.737
7.673
7.531
7.509
7.496
7.480
7.458
7.269
7.246
7.239
7.228
7.210
7.187
7.015
6.910
2.228
0.000
O
N
Me
Ph
Ph
40
1H NMR (300 MHz, CDCl3)
1.00
1.01
1.02
1.01
2.02
5.04
5.01
4.02
3.01
12
10
8
6
4
2
0
PPM



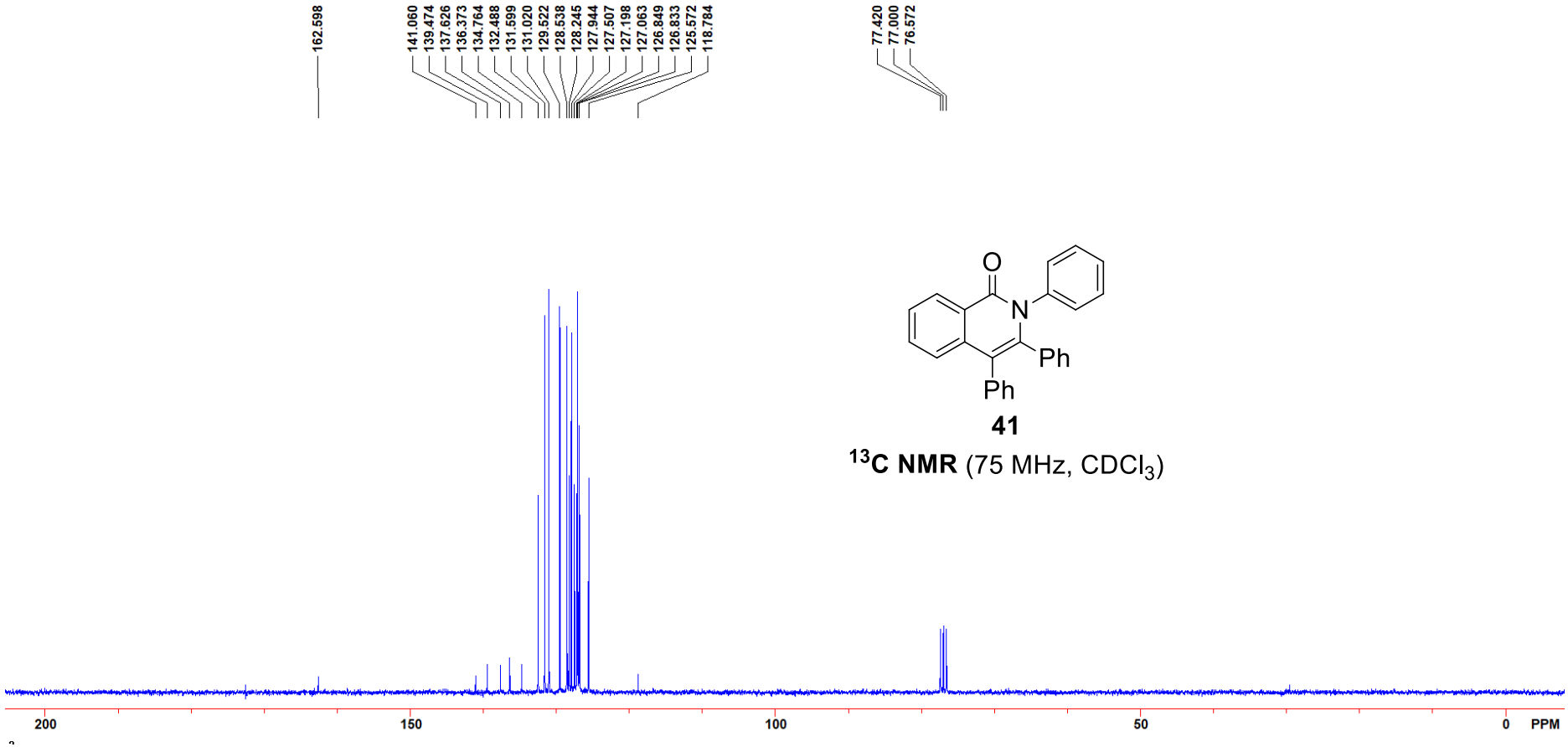
40

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



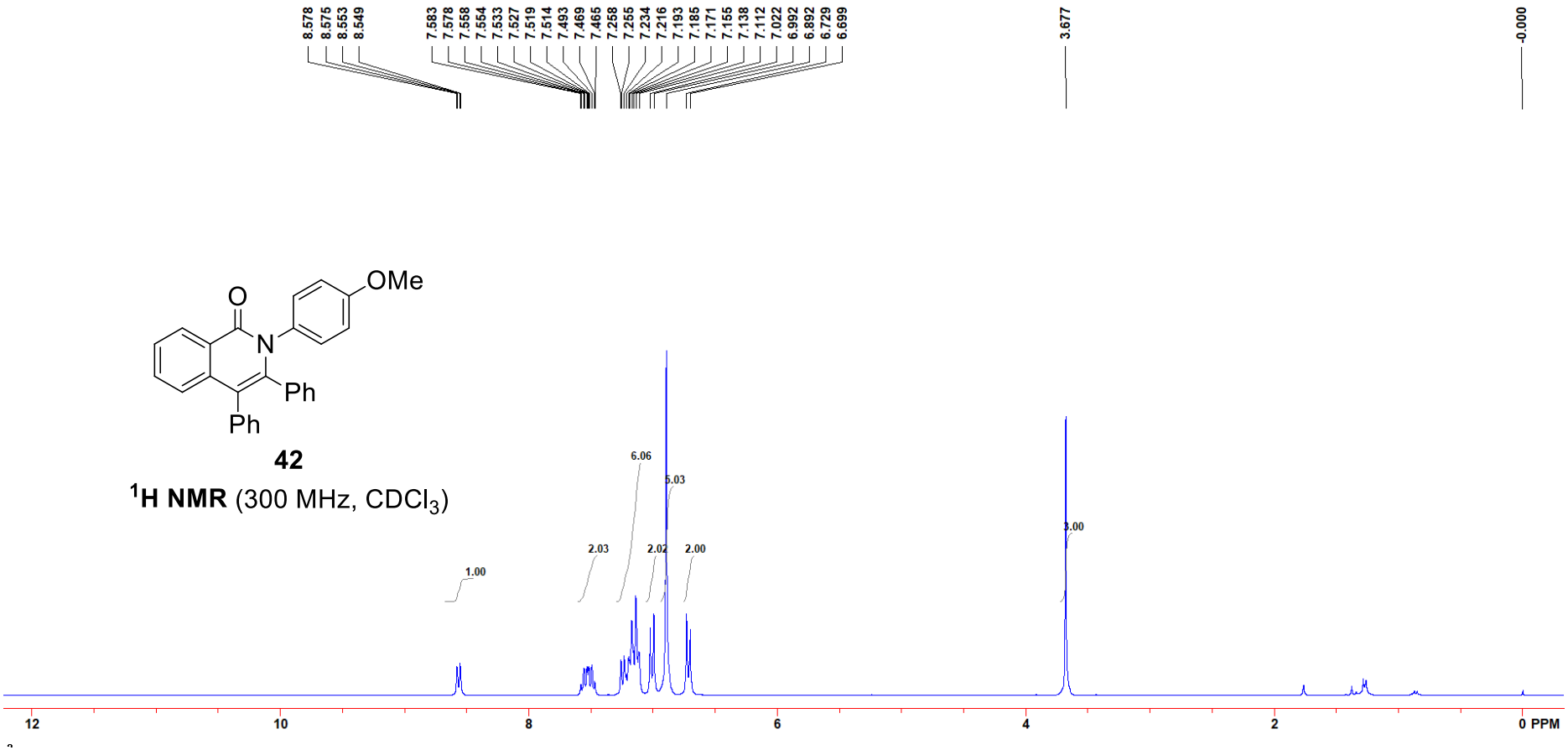
41

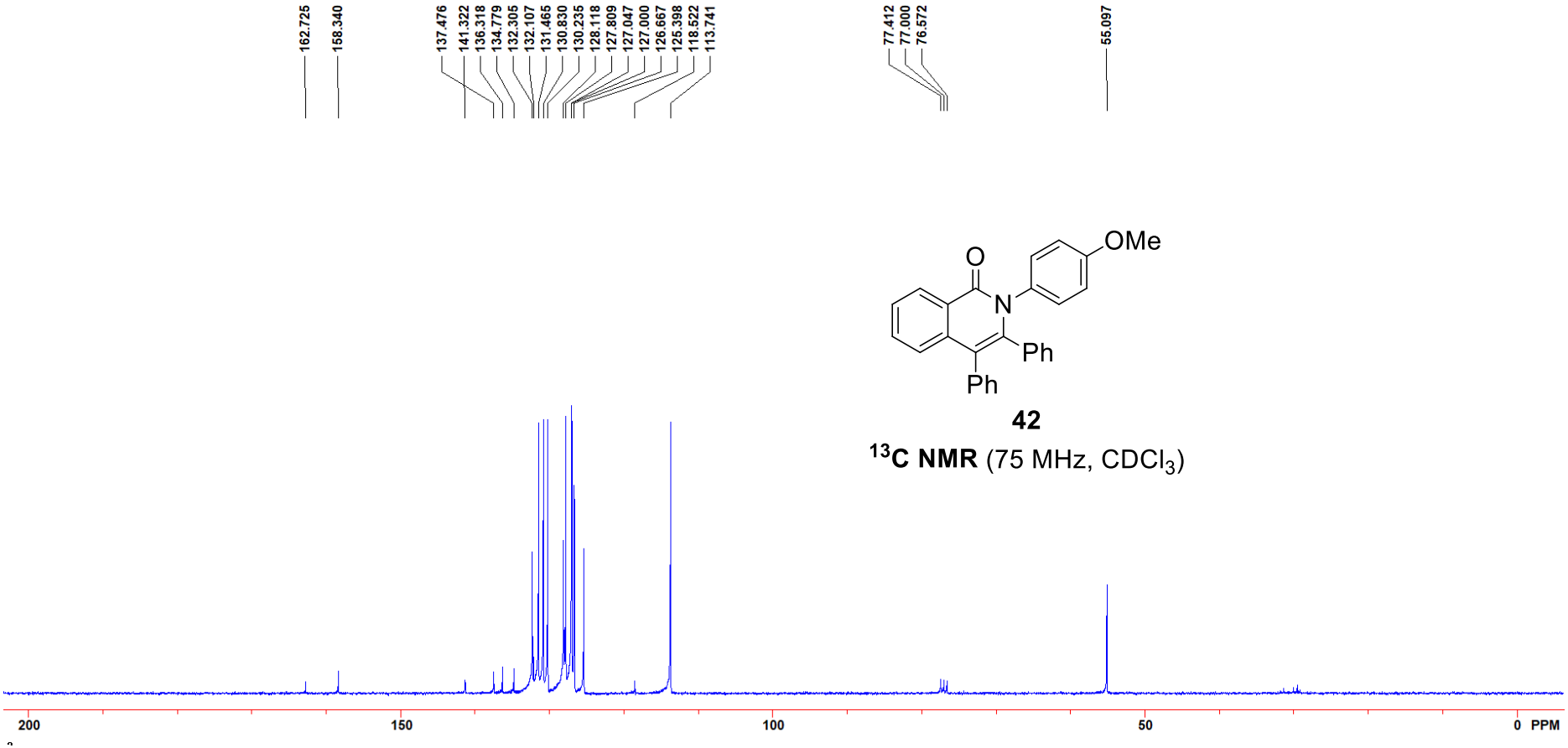
^{1}H NMR (300 MHz, $CDCl_3$)



41

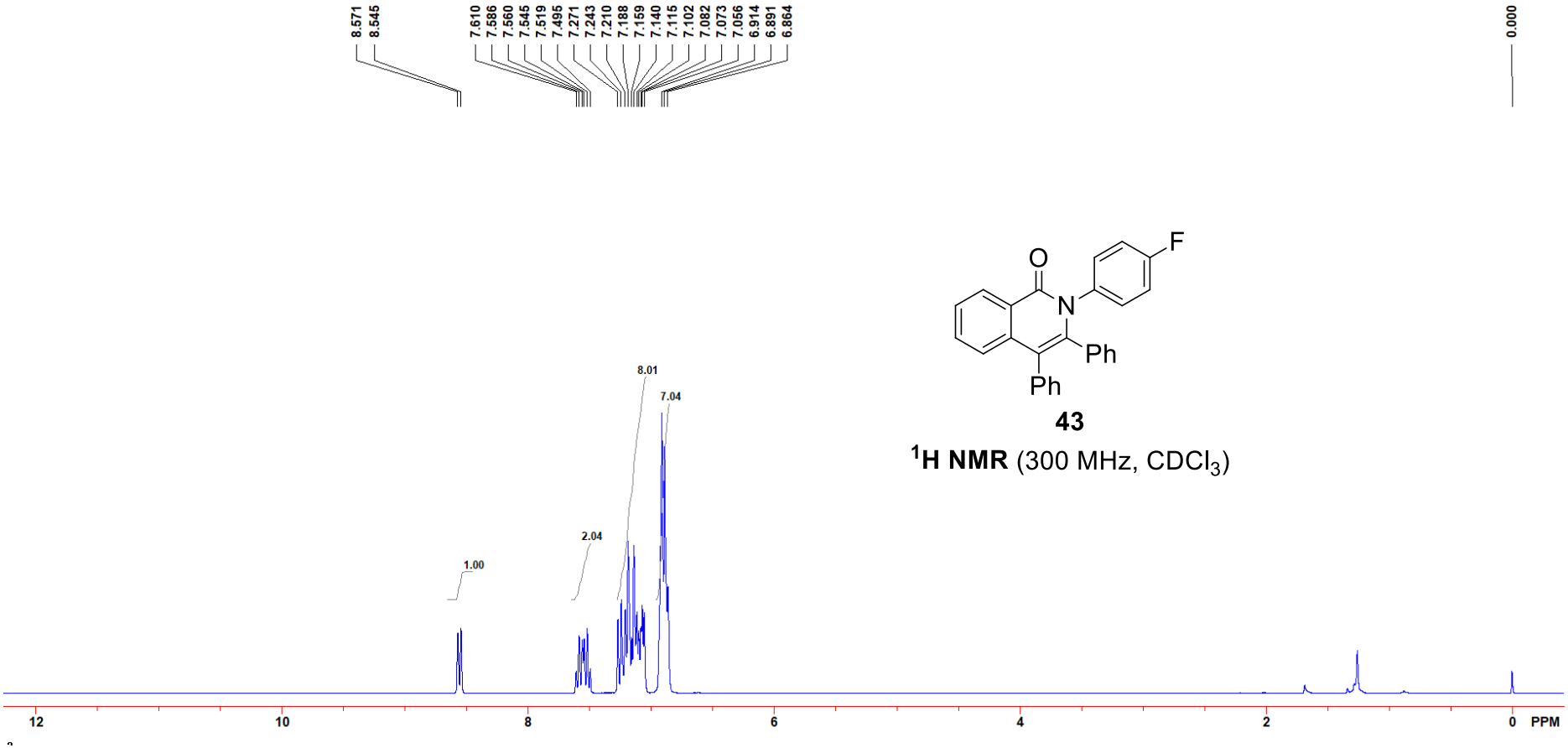
^{13}C NMR (75 MHz, $CDCl_3$)


8.578
8.575
8.553
8.549
7.583
7.578
7.558
7.554
7.533
7.527
7.519
7.514
7.493
7.469
7.465
7.258
7.255
7.234
7.216
7.193
7.185
7.171
7.155
7.138
7.112
7.022
6.992
6.892
6.729
6.699
3.677
-0.000
O
N
OMe
Ph
Ph
42
1H NMR (300 MHz, CDCl3)
1.00
2.03
6.06
5.03
2.02
2.00
3.00
12
10
8
6
4
2
0 PPM



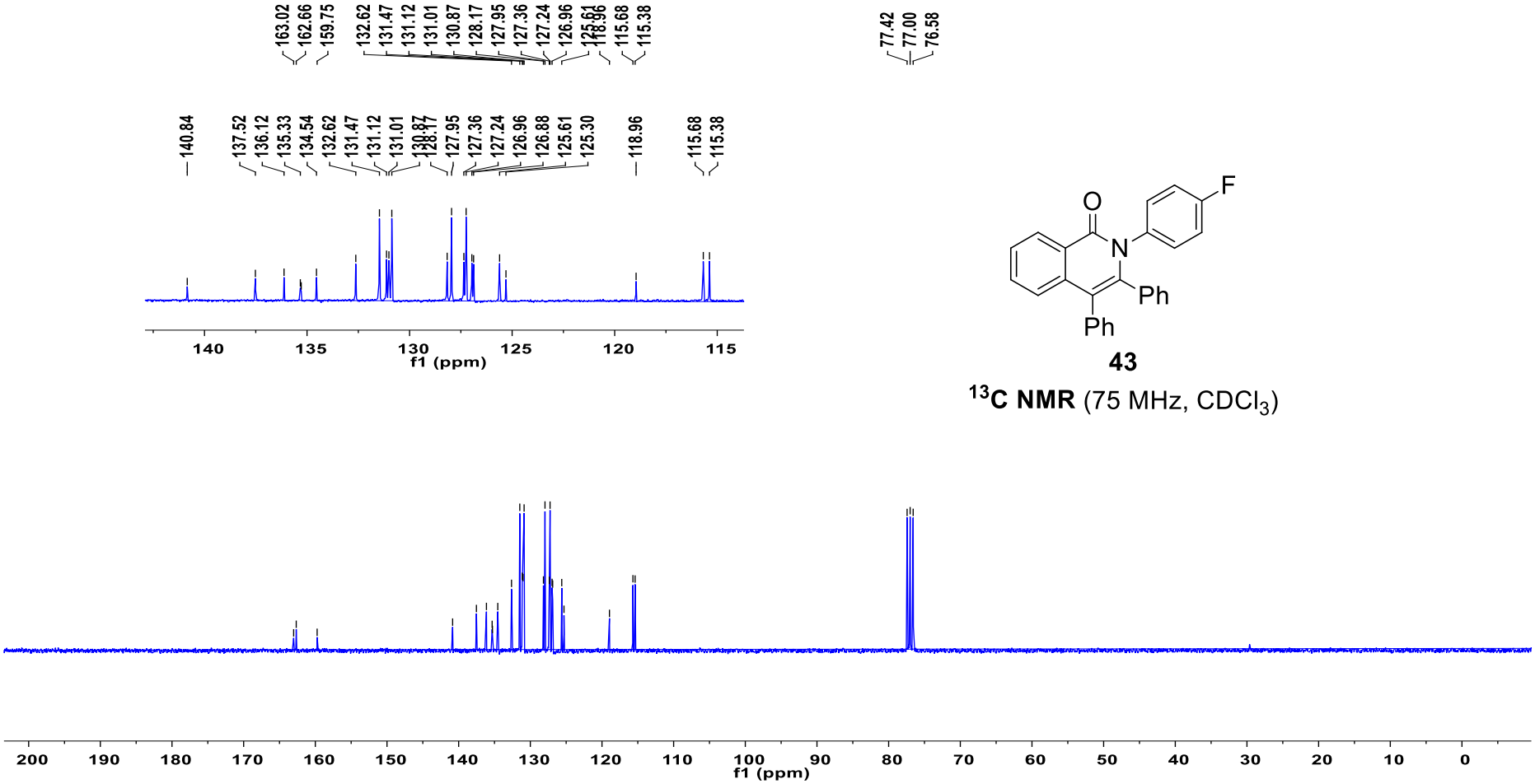
42

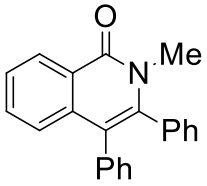
^{13}C NMR (75 MHz, $CDCl_3$)



43

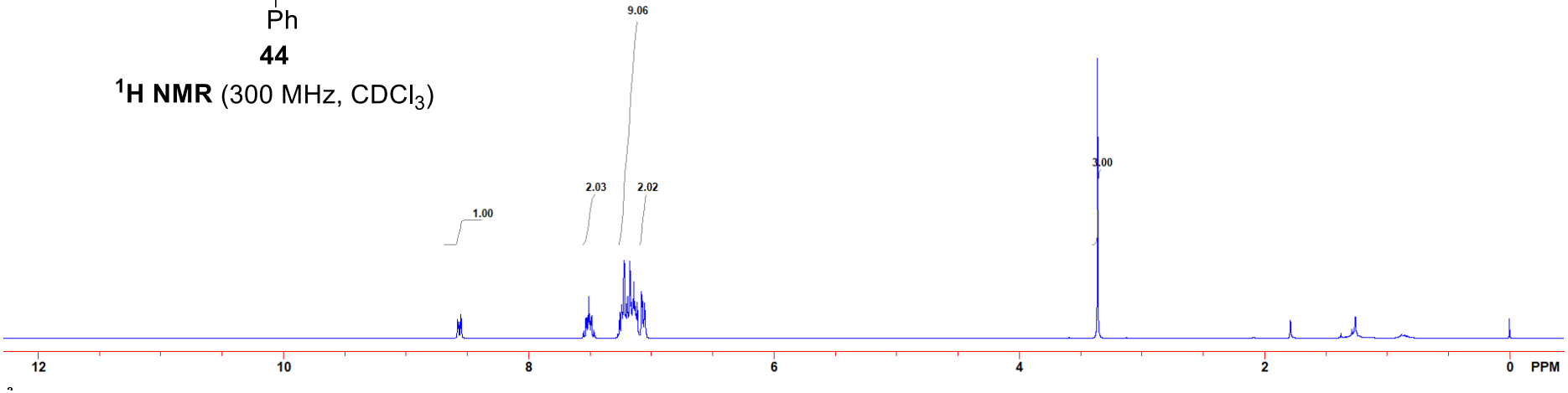
^{1}H NMR (300 MHz, $CDCl_3$)

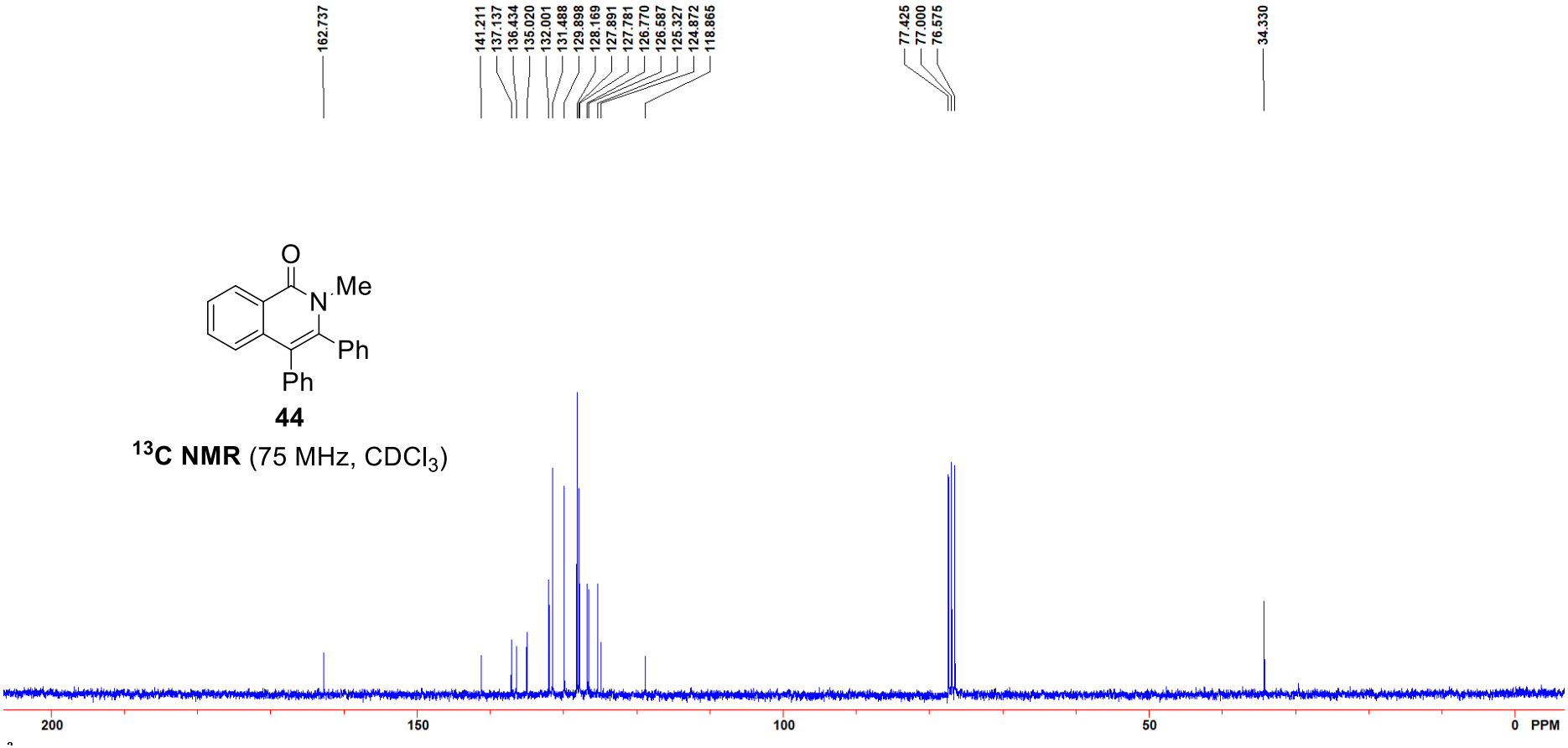

163.02
162.66
159.75
132.62
131.47
131.12
131.01
130.87
128.17
127.95
127.36
127.24
126.96
125.61
118.96
115.68
115.38
77.42
77.00
76.58
140.84
137.52
136.12
135.33
134.54
132.62
131.47
131.12
131.01
130.87
128.17
127.95
127.36
127.24
126.96
126.88
125.61
125.30
118.96
115.68
115.38
140
135
130
125
120
115
f1 (ppm)
O
N
F
Ph
Ph
43
13C NMR (75 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



44

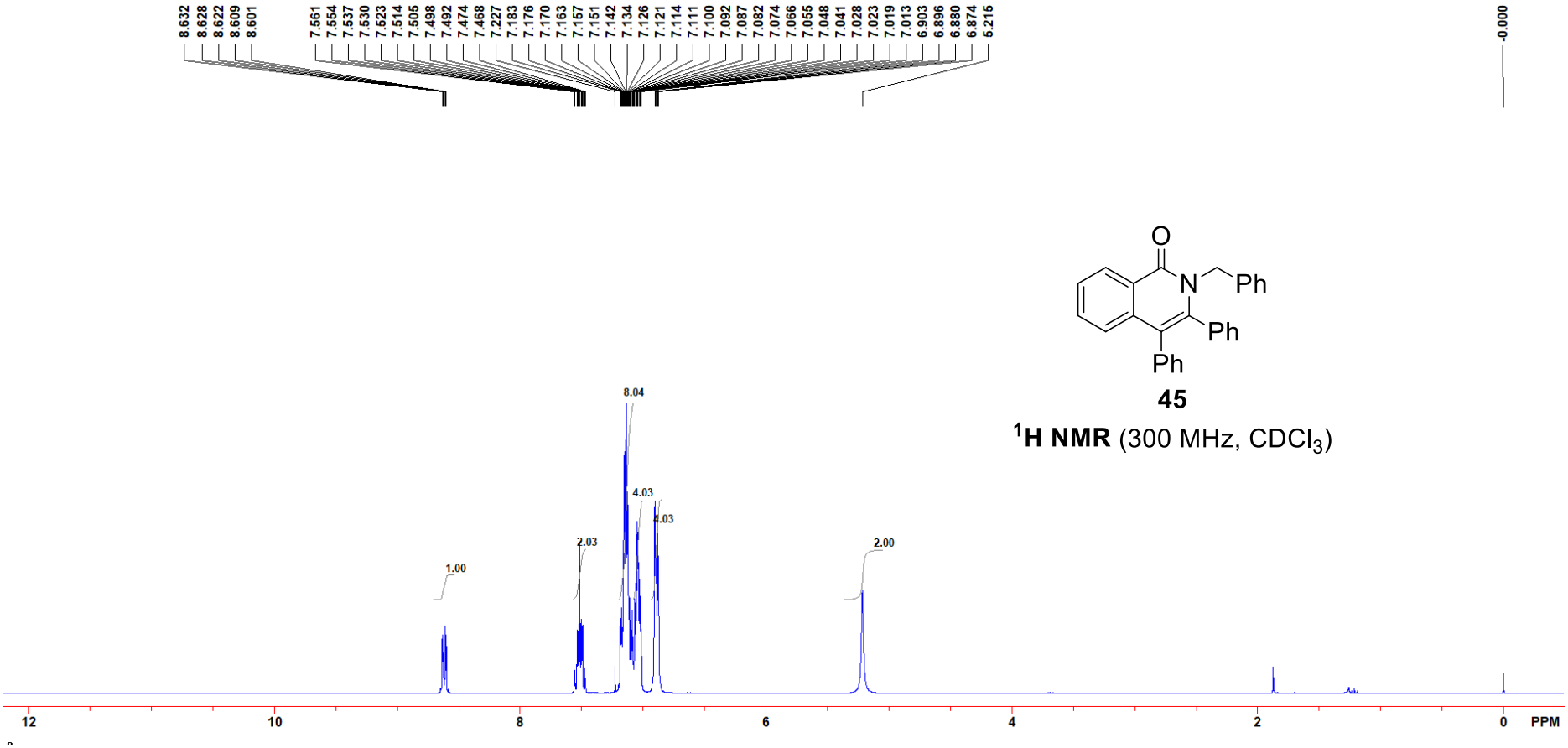
^{1}H NMR (300 MHz, $CDCl_3$)

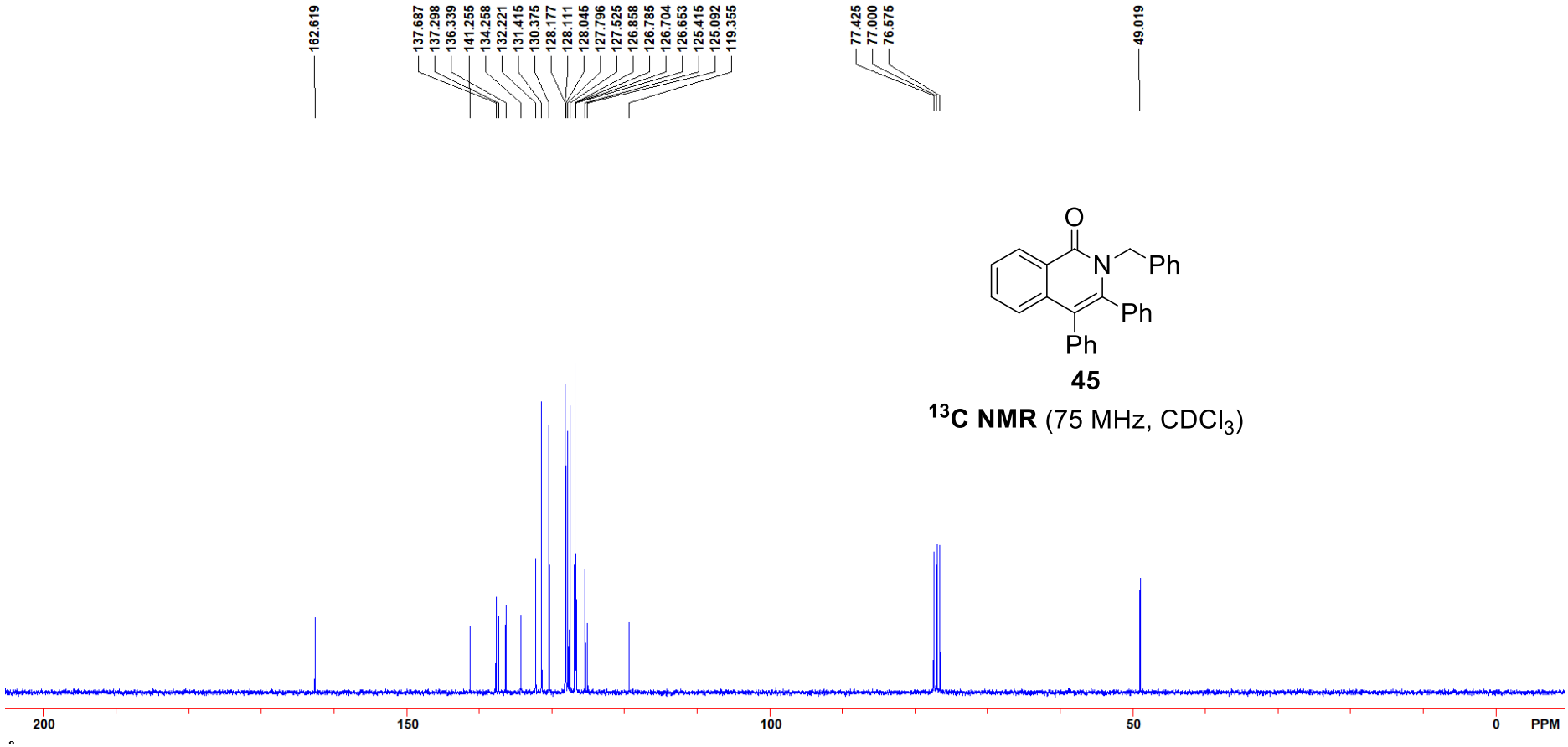




44

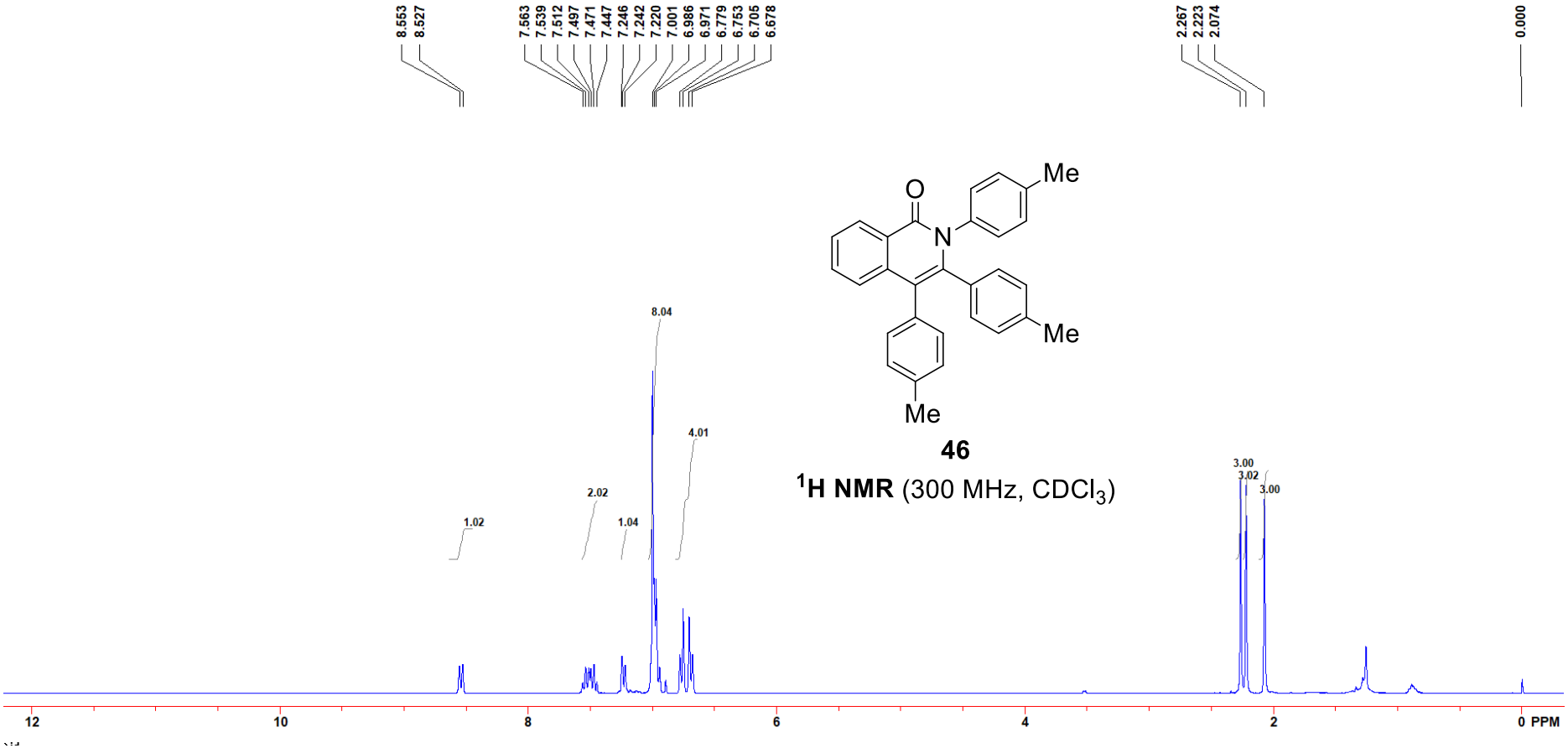
^{13}C NMR (75 MHz, $CDCl_3$)

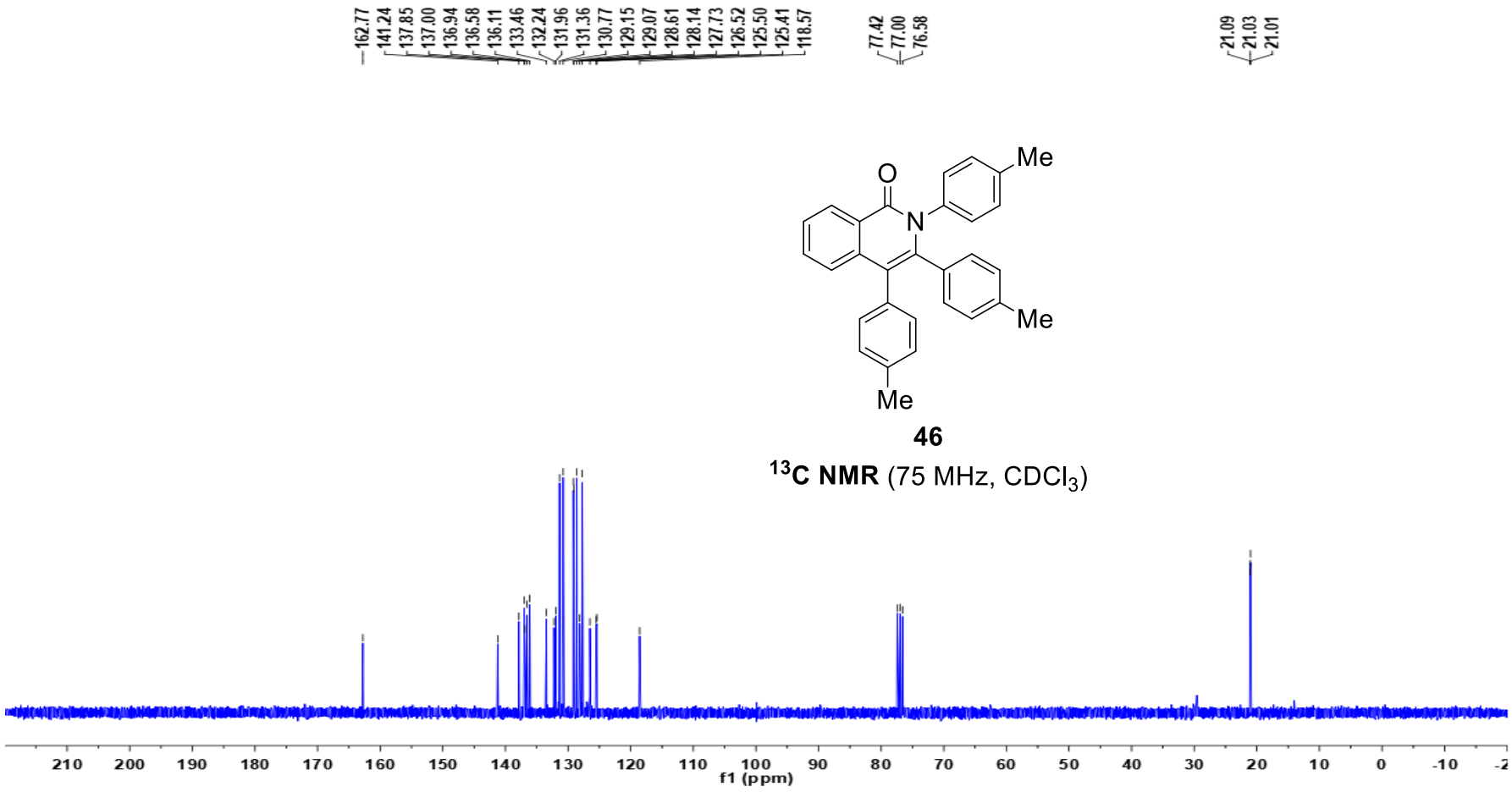

8.632
8.628
8.622
8.609
8.601
7.561
7.554
7.537
7.530
7.523
7.514
7.505
7.498
7.492
7.474
7.468
7.227
7.183
7.176
7.170
7.163
7.157
7.151
7.142
7.134
7.126
7.121
7.114
7.111
7.100
7.092
7.087
7.082
7.074
7.066
7.055
7.048
7.041
7.028
7.023
7.019
7.013
6.903
6.896
6.880
6.874
5.215
-0.000
O
N
Ph
Ph
Ph
45
1H NMR (300 MHz, CDCl3)
8.04
4.03
4.03
2.03
2.00
1.00
12
10
8
6
4
2
0
PPM



45

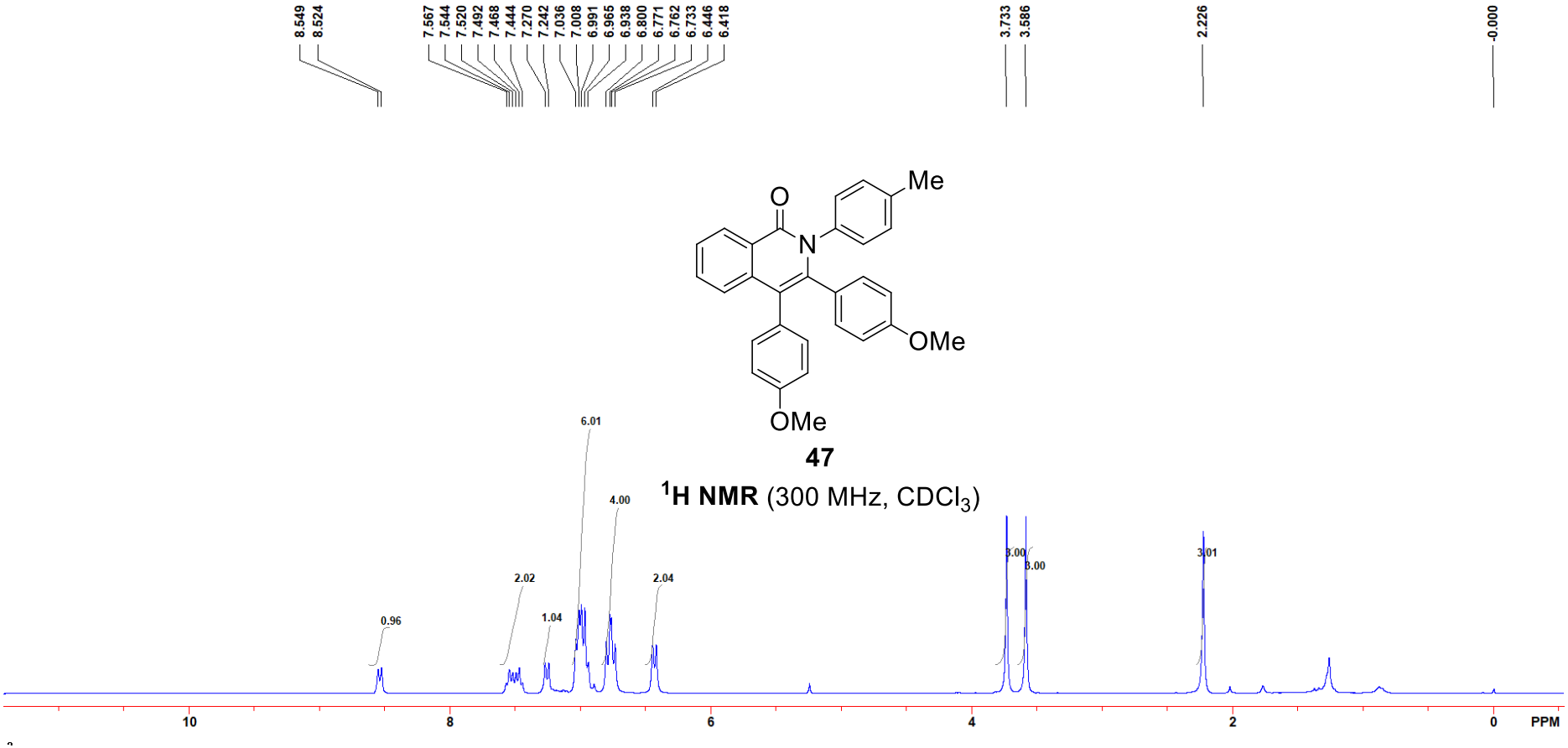
^{13}C NMR (75 MHz, $CDCl_3$)

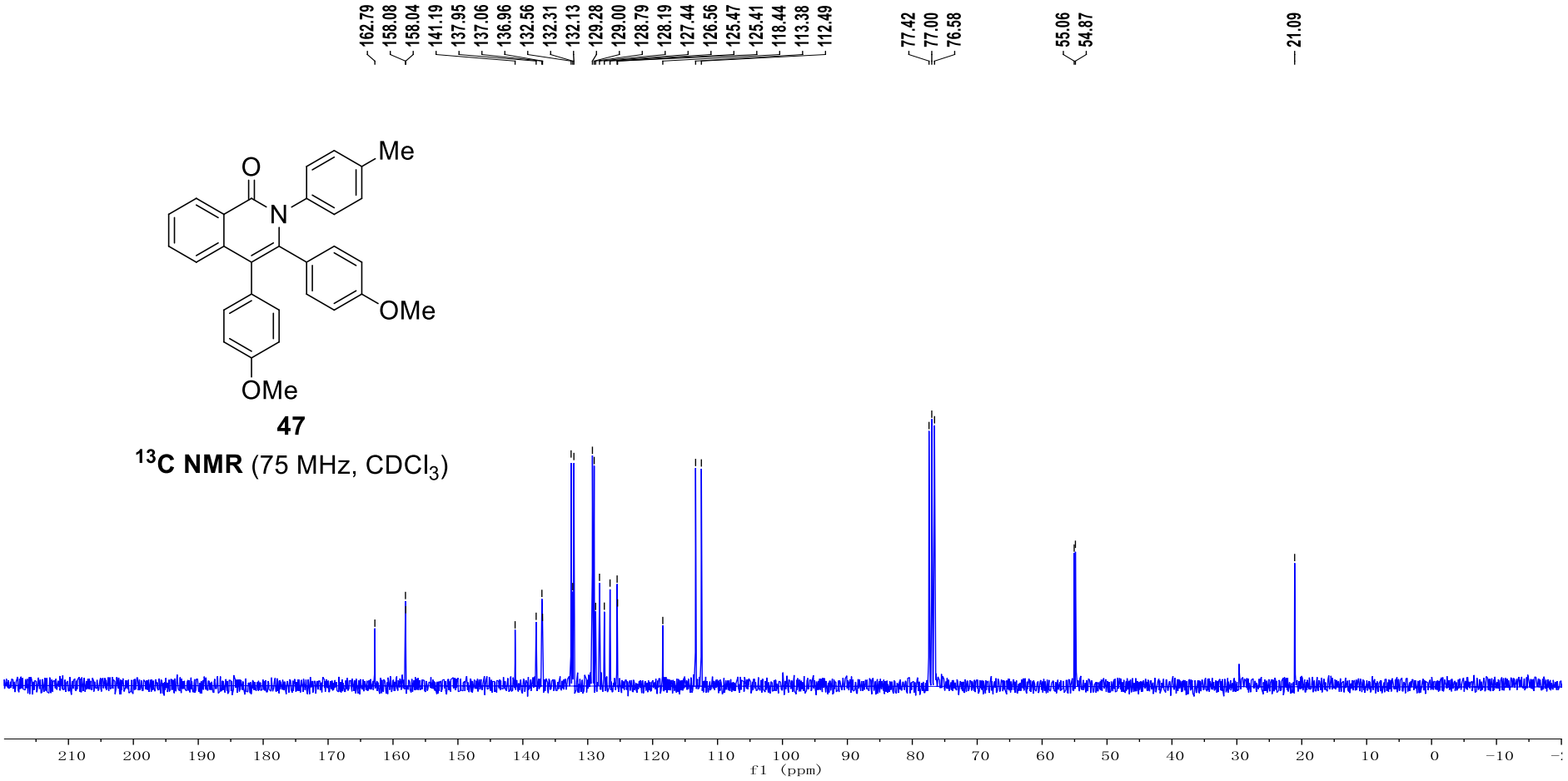

8.553
8.527
7.563
7.539
7.512
7.497
7.471
7.447
7.246
7.242
7.220
7.001
6.986
6.971
6.779
6.753
6.705
6.678
2.267
2.223
2.074
0.000
O
N
Me
Me
Me
46
1H NMR (300 MHz, CDCl3)
1.02
2.02
1.04
8.04
4.01
3.00
3.02
3.00
12
10
8
6
4
2
0 PPM

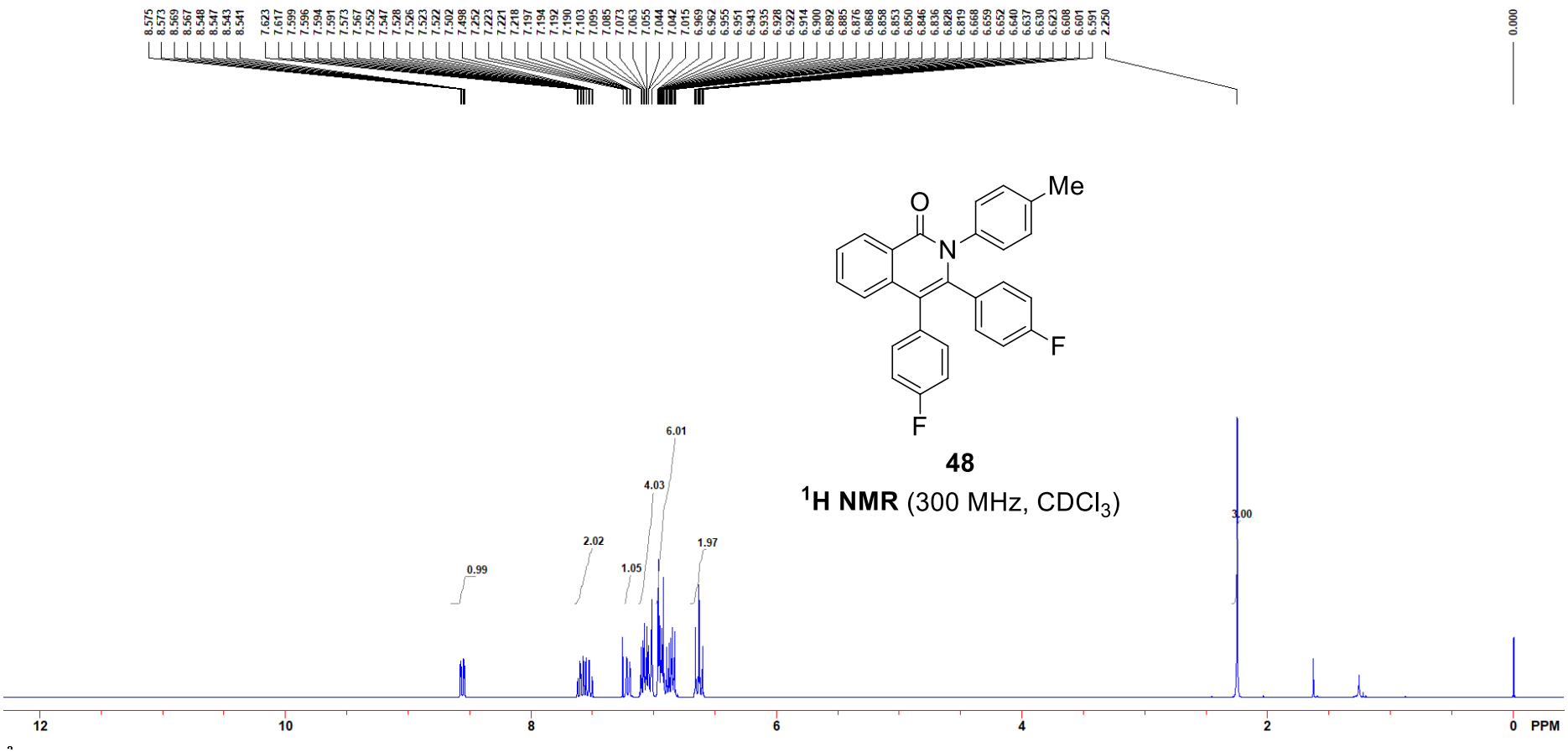


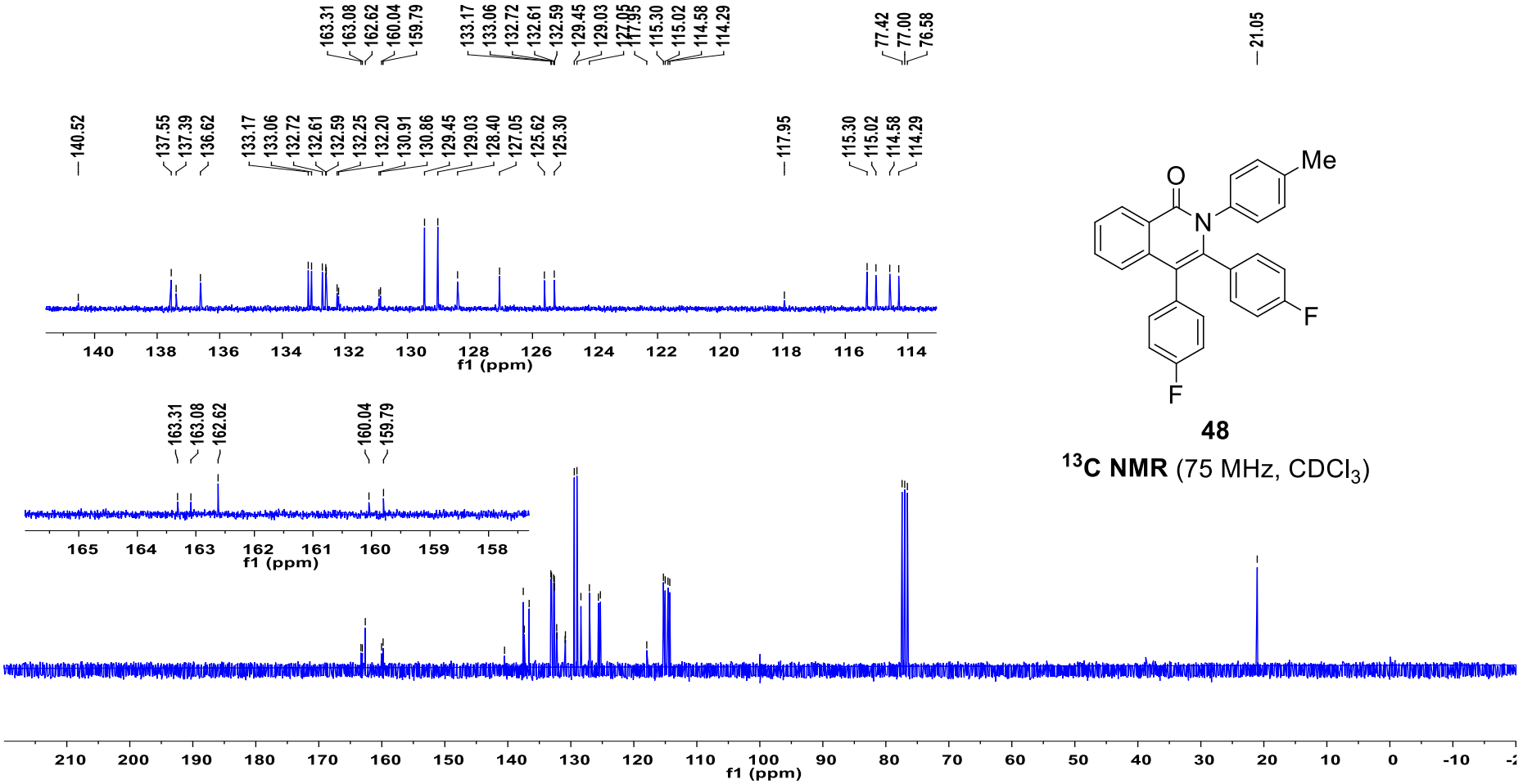
46

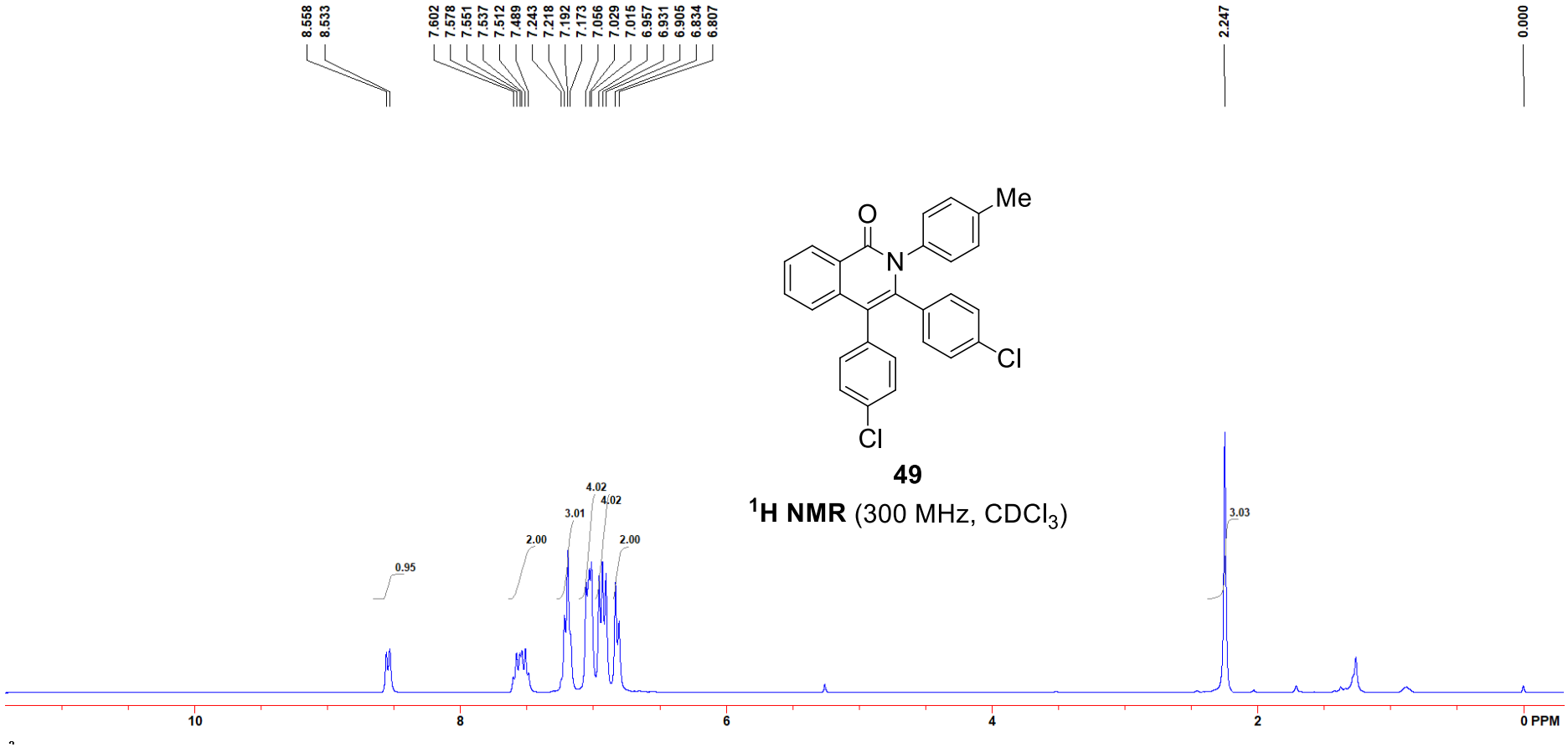
^{13}C NMR (75 MHz, $CDCl_3$)


8.549
8.524
7.567
7.544
7.520
7.492
7.468
7.444
7.270
7.242
7.036
7.008
6.991
6.965
6.938
6.800
6.771
6.762
6.733
6.446
6.418
3.733
3.586
2.226
-0.000
O
N
Me
OMe
OMe
47
1H NMR (300 MHz, CDCl3)
6.01
4.00
2.02
2.04
1.04
0.96
3.00
3.00
3.01
10
8
6
4
2
0
PPM


162.79
158.08
158.04
141.19
137.95
137.06
136.96
132.56
132.31
132.13
129.28
129.00
128.79
128.19
127.44
126.56
125.47
125.41
118.44
113.38
112.49
77.42
77.00
76.58
55.06
54.87
21.09
O
N
Me
OMe
OMe
47
13C NMR (75 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

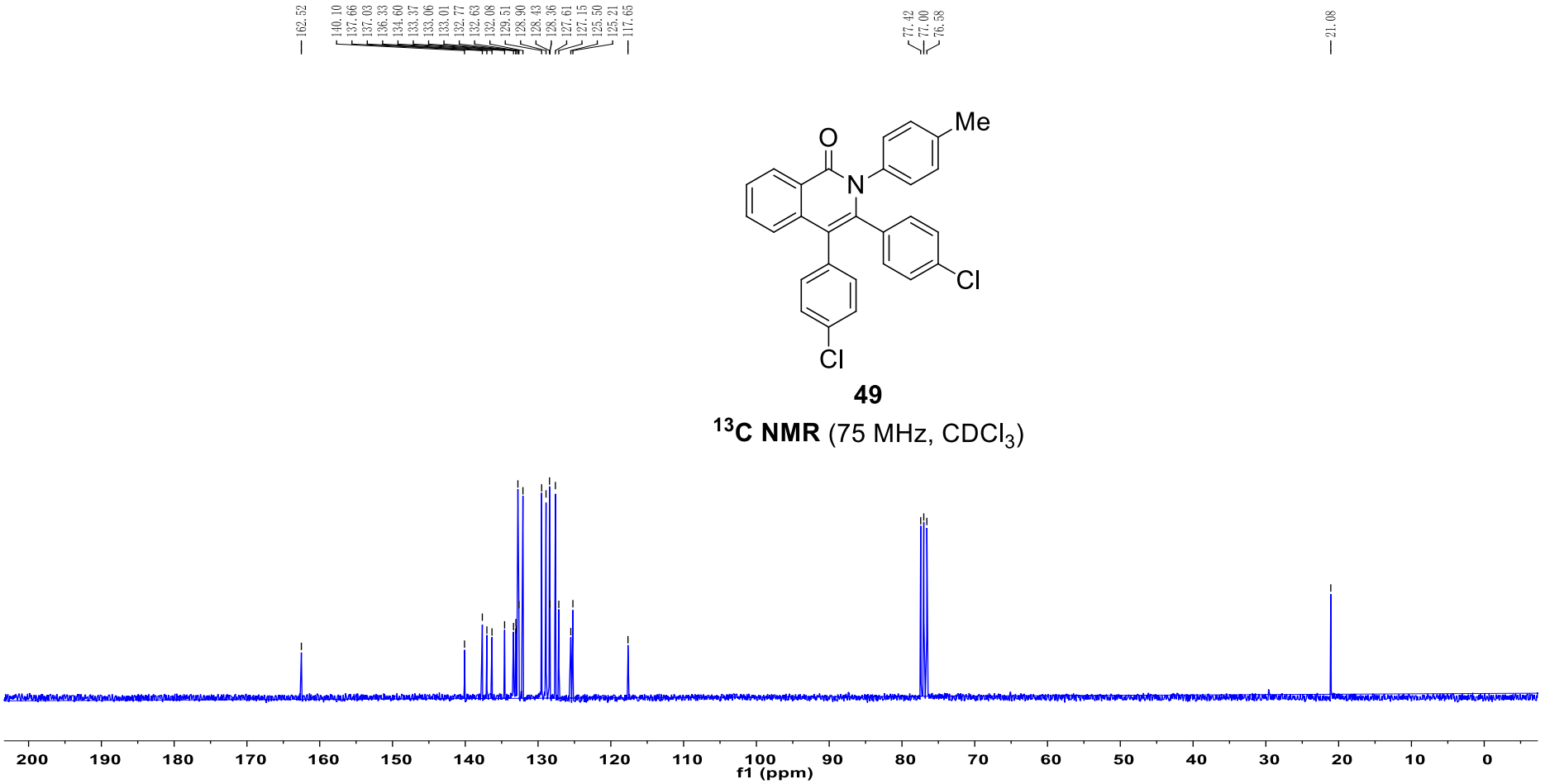

8.575 8.573 8.569 8.567 8.548 8.547 8.543 8.541 7.623 7.617 7.599 7.596 7.594 7.591 7.573 7.567 7.552 7.547 7.528 7.526 7.523 7.522 7.502 7.498 7.252 7.223 7.221 7.218 7.197 7.194 7.192 7.190 7.103 7.095 7.085 7.073 7.063 7.055 7.044 7.042 7.015 6.969 6.962 6.955 6.951 6.943 6.935 6.928 6.922 6.914 6.900 6.892 6.885 6.876 6.868 6.858 6.853 6.850 6.846 6.836 6.828 6.819 6.668 6.659 6.652 6.640 6.637 6.630 6.623 6.608 6.601 6.591 2.250
0.000
O
N
Me
F
F
48
1H NMR (300 MHz, CDCl3)
6.01
4.03
2.02
1.97
0.99
1.05
3.00
12
10
8
6
4
2
0
PPM


163.31
163.08
162.62
160.04
159.79
133.17
133.06
132.72
132.61
132.59
129.45
129.03
127.05
117.95
115.30
115.02
114.58
114.29
77.42
77.00
76.58
21.05
140.52
137.55
137.39
136.62
133.17
133.06
132.72
132.61
132.59
132.25
132.20
130.91
130.86
129.45
129.03
128.40
127.05
125.62
125.30
117.95
115.30
115.02
114.58
114.29
f1 (ppm)
O
N
Me
F
F
48
13C NMR (75 MHz, CDCl3)



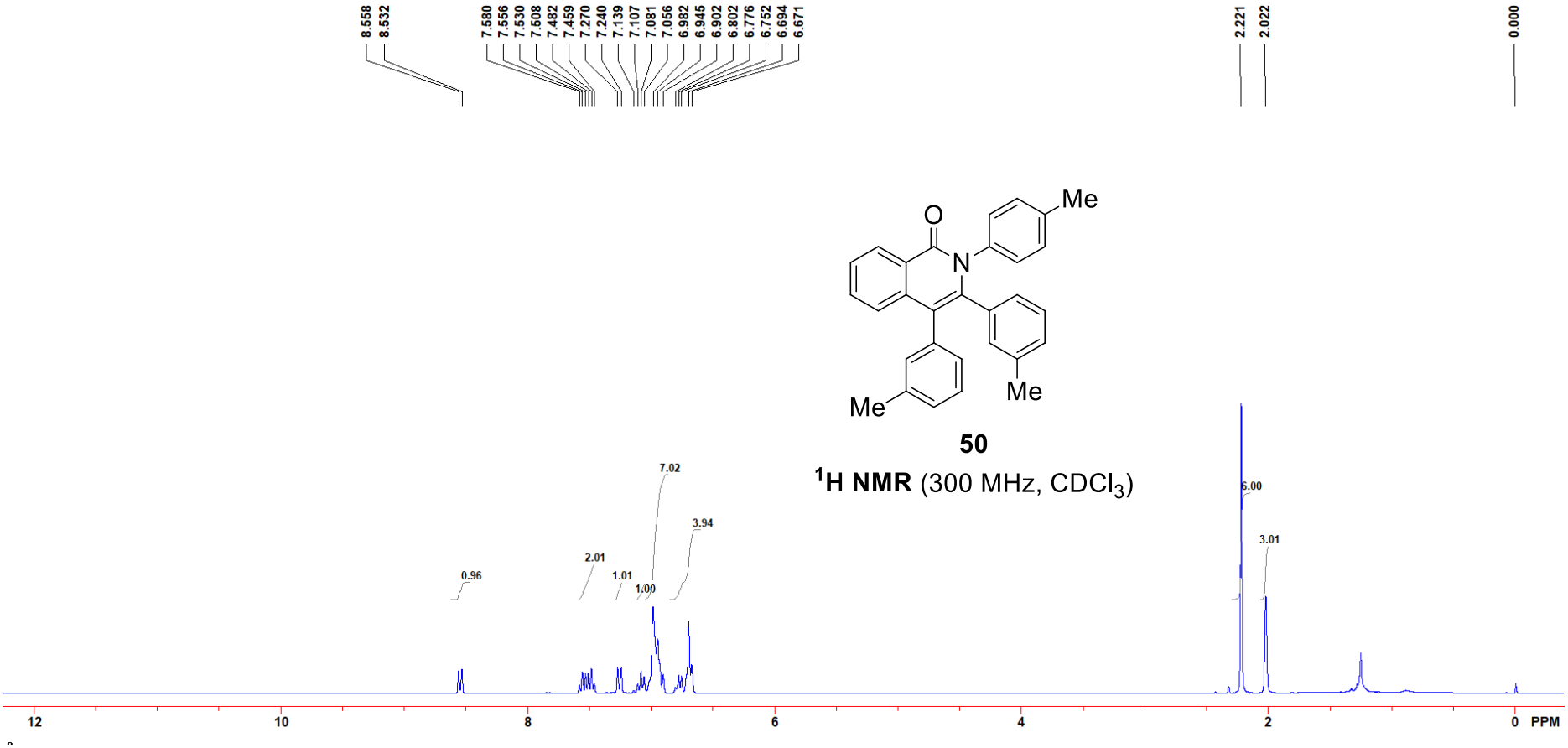
49

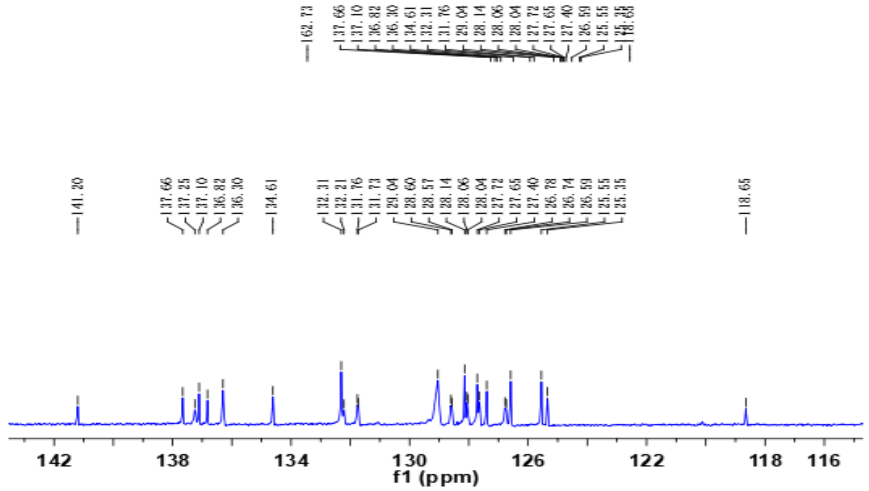
^{1}H NMR (300 MHz, $CDCl_3$)

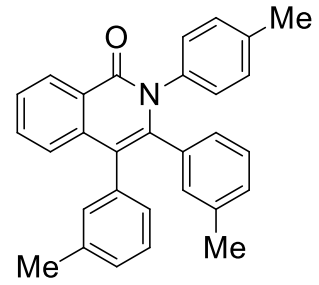


49

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

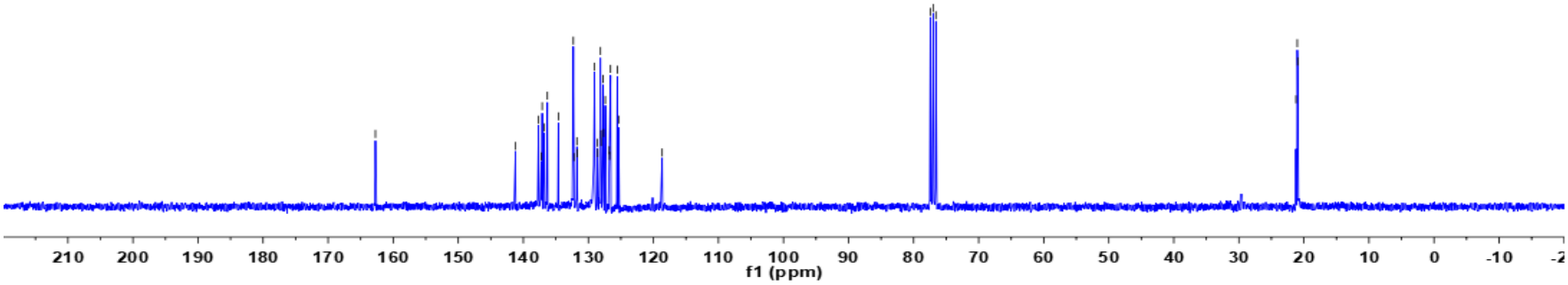

8.558
8.532
7.580
7.556
7.530
7.508
7.482
7.459
7.270
7.240
7.139
7.107
7.081
7.056
6.982
6.945
6.902
6.802
6.776
6.752
6.694
6.671
2.221
2.022
0.000
O
N
Me
Me
Me
50
1H NMR (300 MHz, CDCl3)
7.02
3.94
2.01
1.01
1.00
0.96
6.00
3.01
12
10
8
6
4
2
0
PPM

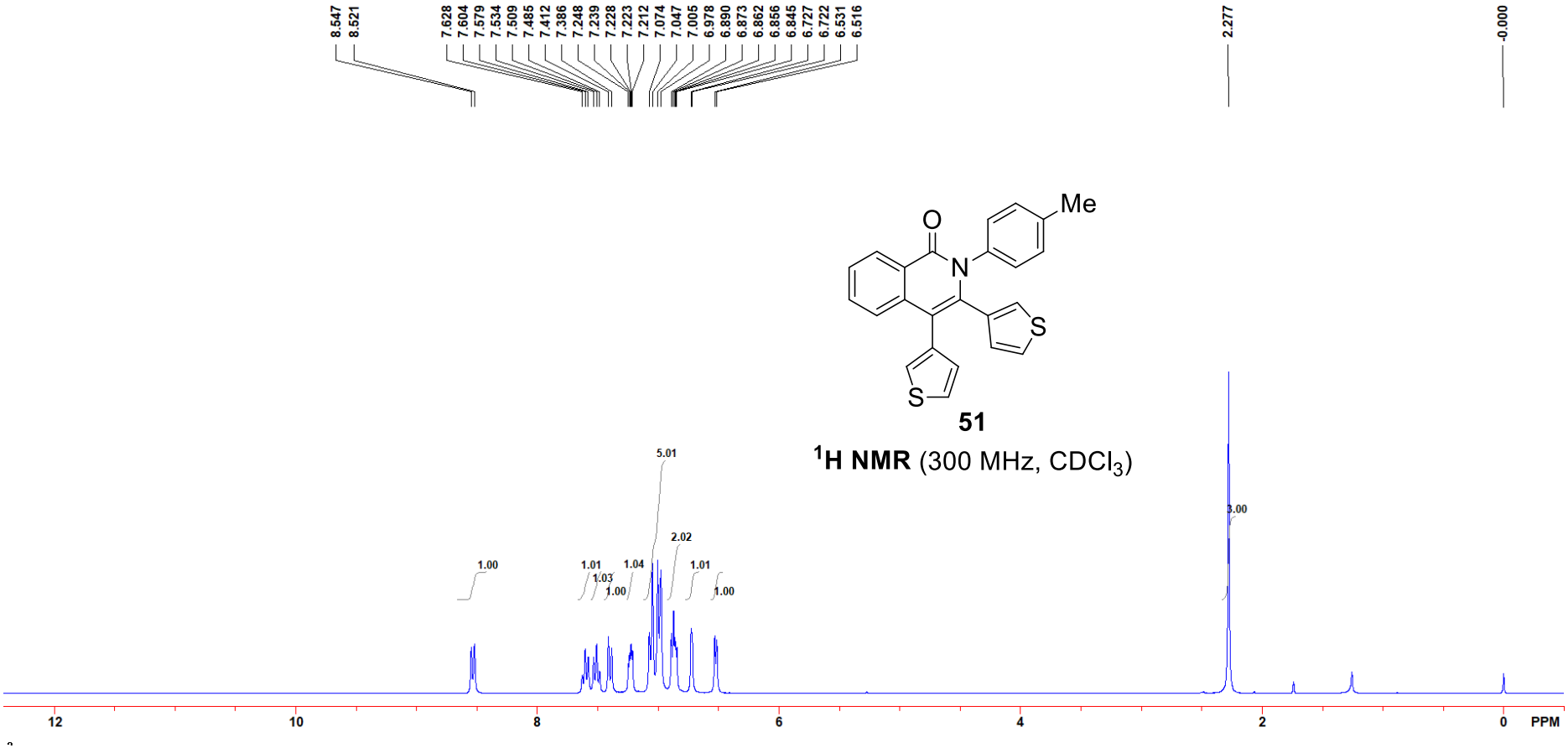


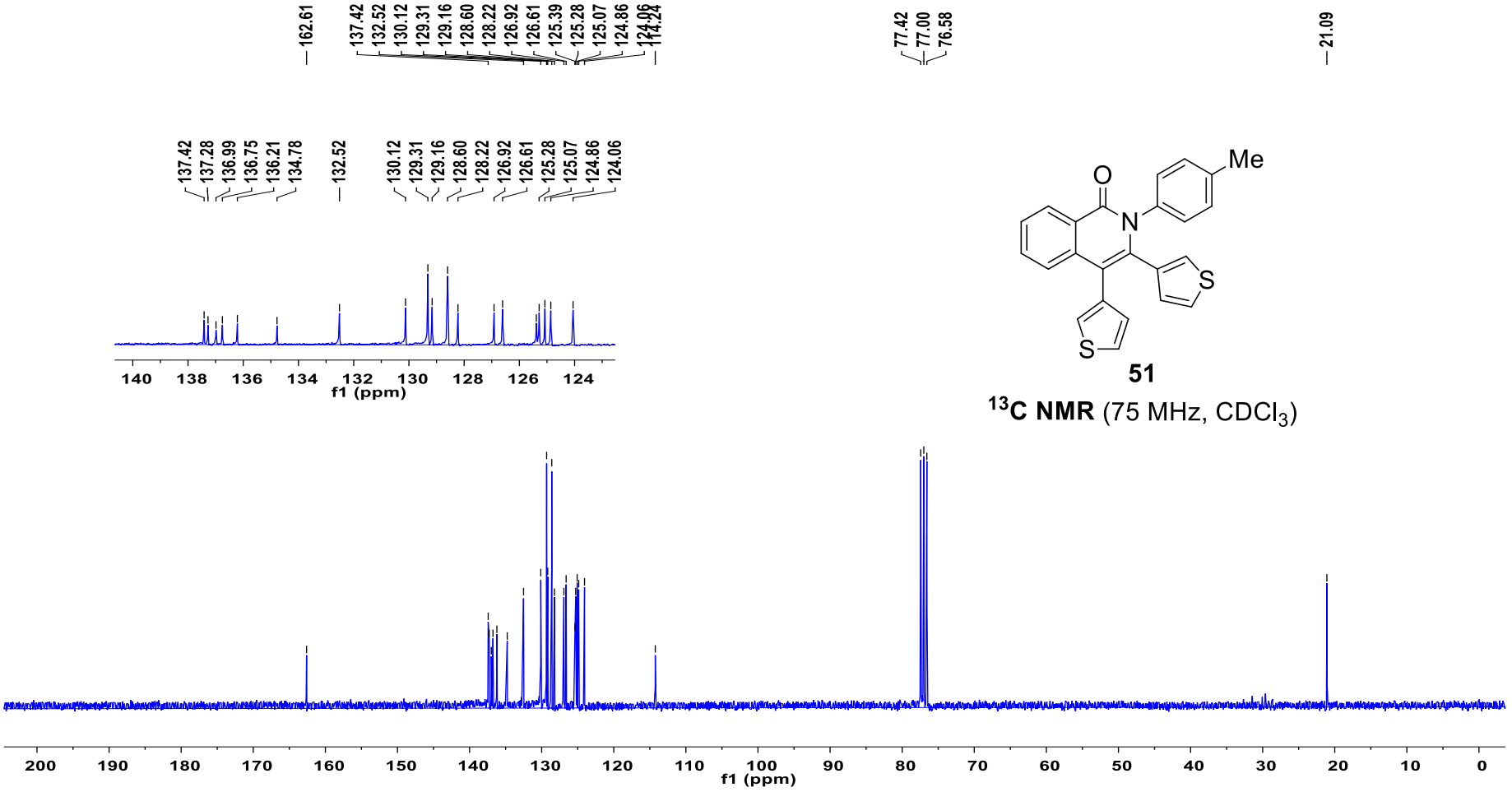


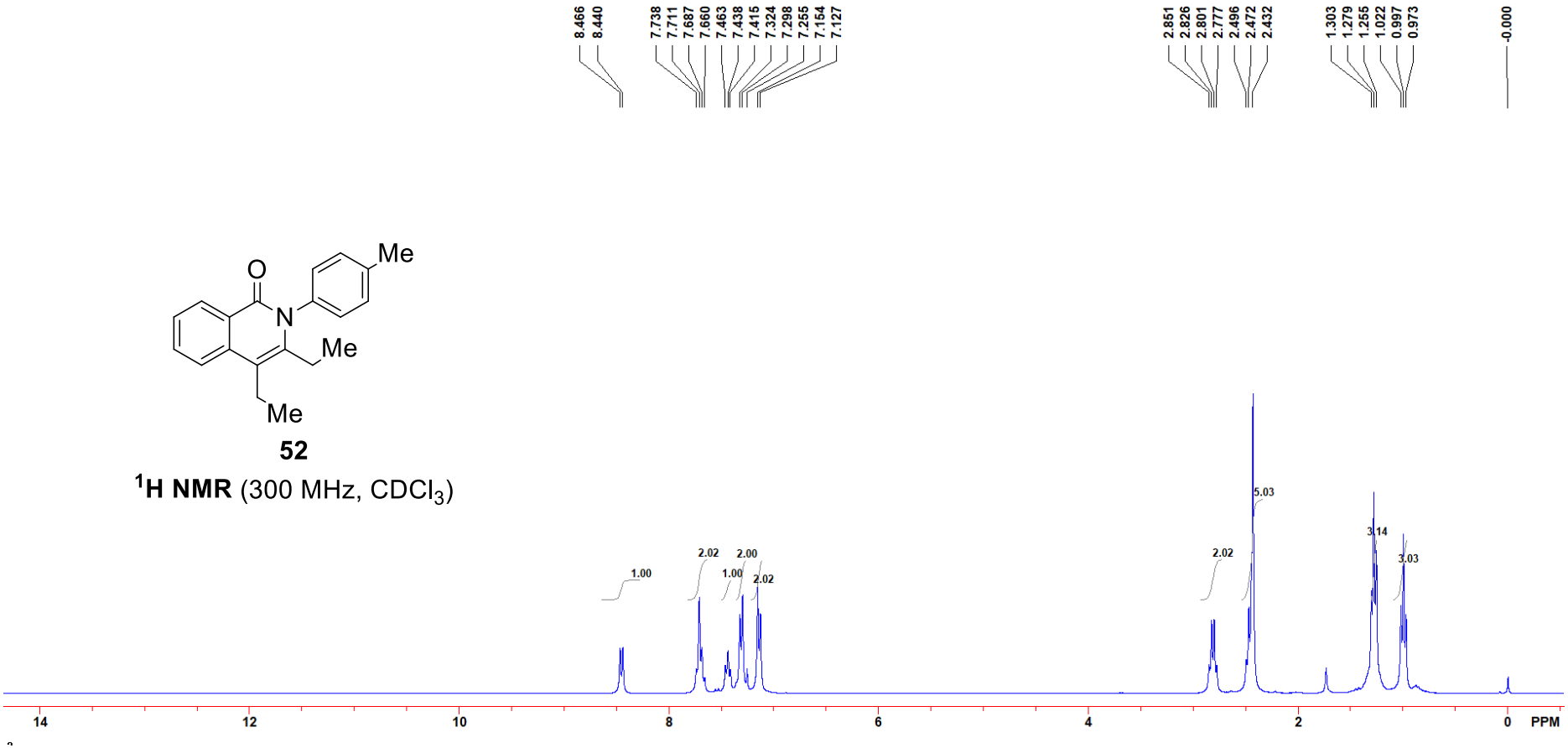
50

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



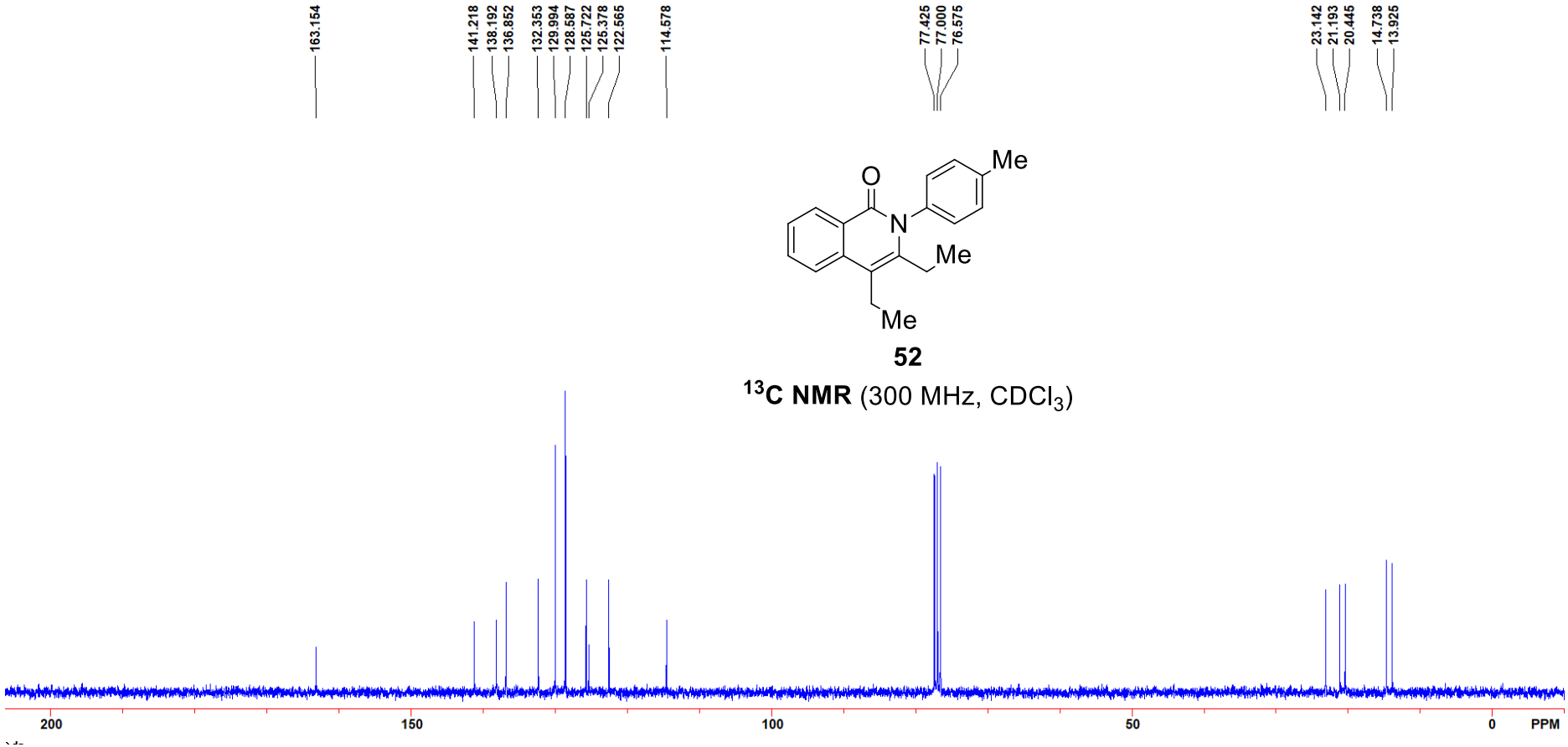

8.547
8.521
7.628
7.604
7.579
7.534
7.509
7.485
7.412
7.386
7.248
7.239
7.228
7.223
7.212
7.074
7.047
7.005
6.978
6.890
6.873
6.862
6.856
6.845
6.727
6.722
6.531
6.516
2.277
-0.000
O
N
Me
S
S
51
1H NMR (300 MHz, CDCl3)
5.01
3.00
2.02
1.00
1.01
1.03
1.00
1.04
1.01
1.00
12
10
8
6
4
2
0
PPM


162.61
137.42
132.52
130.12
129.31
129.16
128.60
128.22
126.92
126.61
125.39
125.28
125.07
124.86
114.24
77.42
77.00
76.58
21.09
137.42
137.28
136.99
136.75
136.21
134.78
132.52
130.12
129.31
129.16
128.60
128.22
126.92
126.61
125.28
125.07
124.86
124.06
140
138
136
134
132
130
128
126
124
f1 (ppm)
O
N
Me
S
S
51
13C NMR (75 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



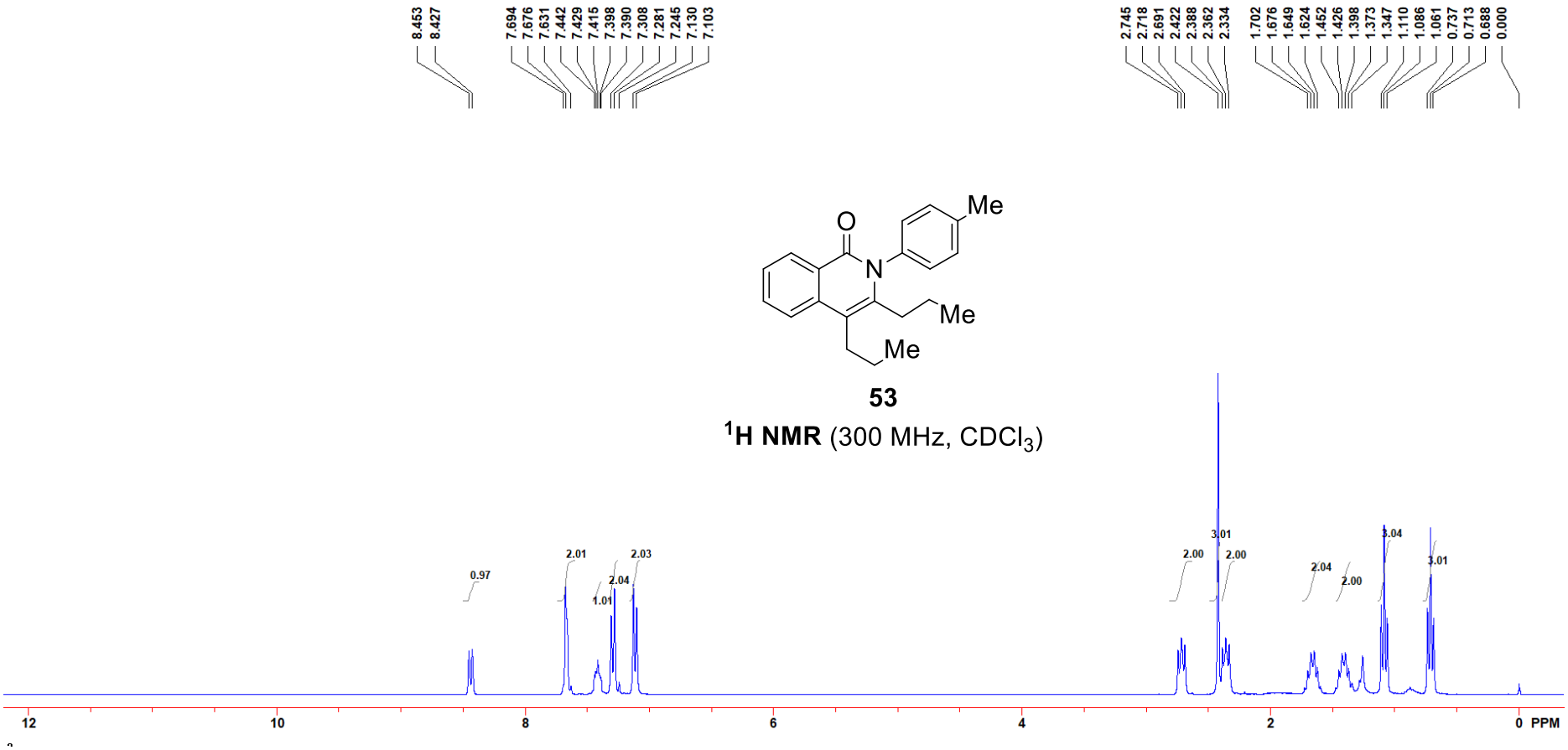
52

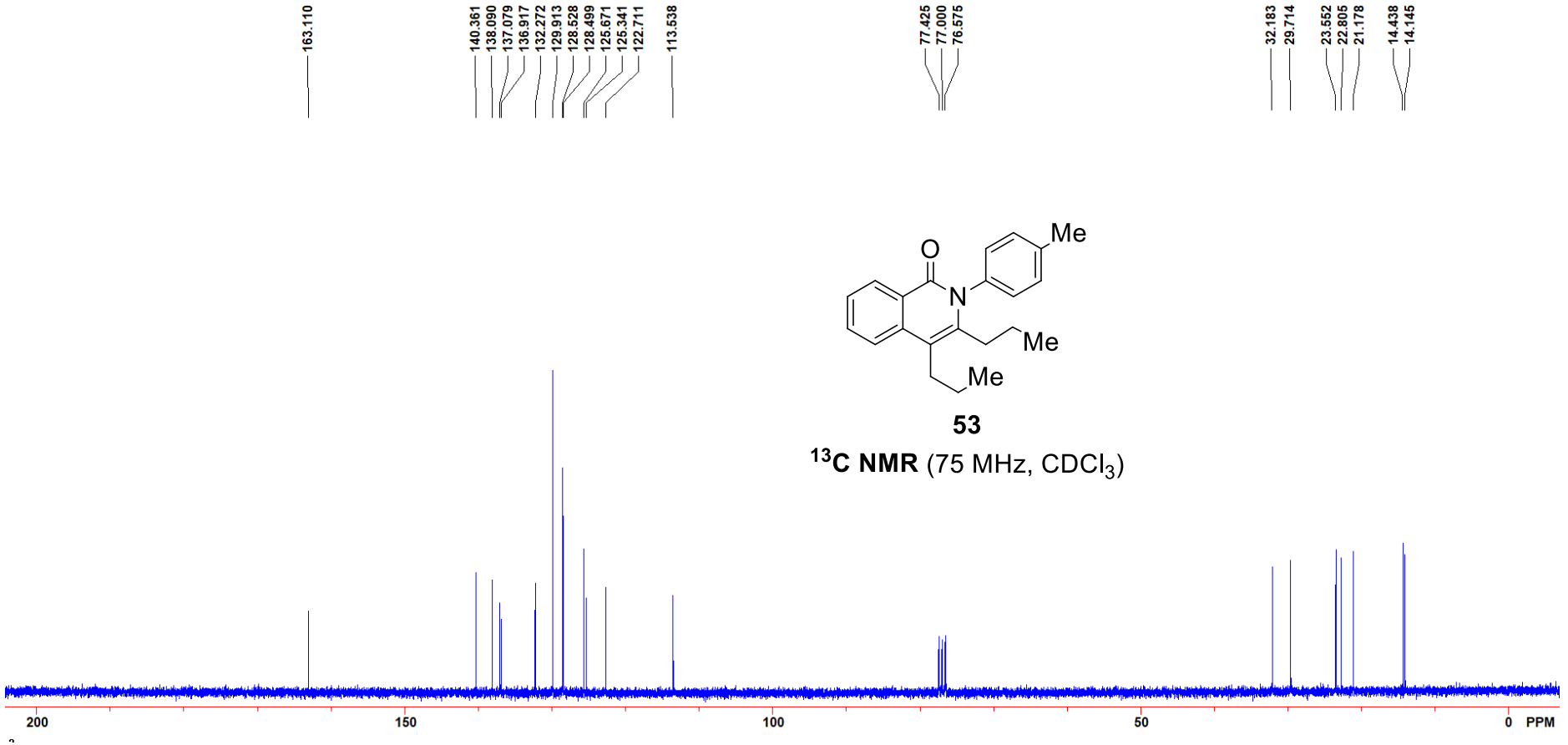
^{1}H NMR (300 MHz, $CDCl_3$)



52

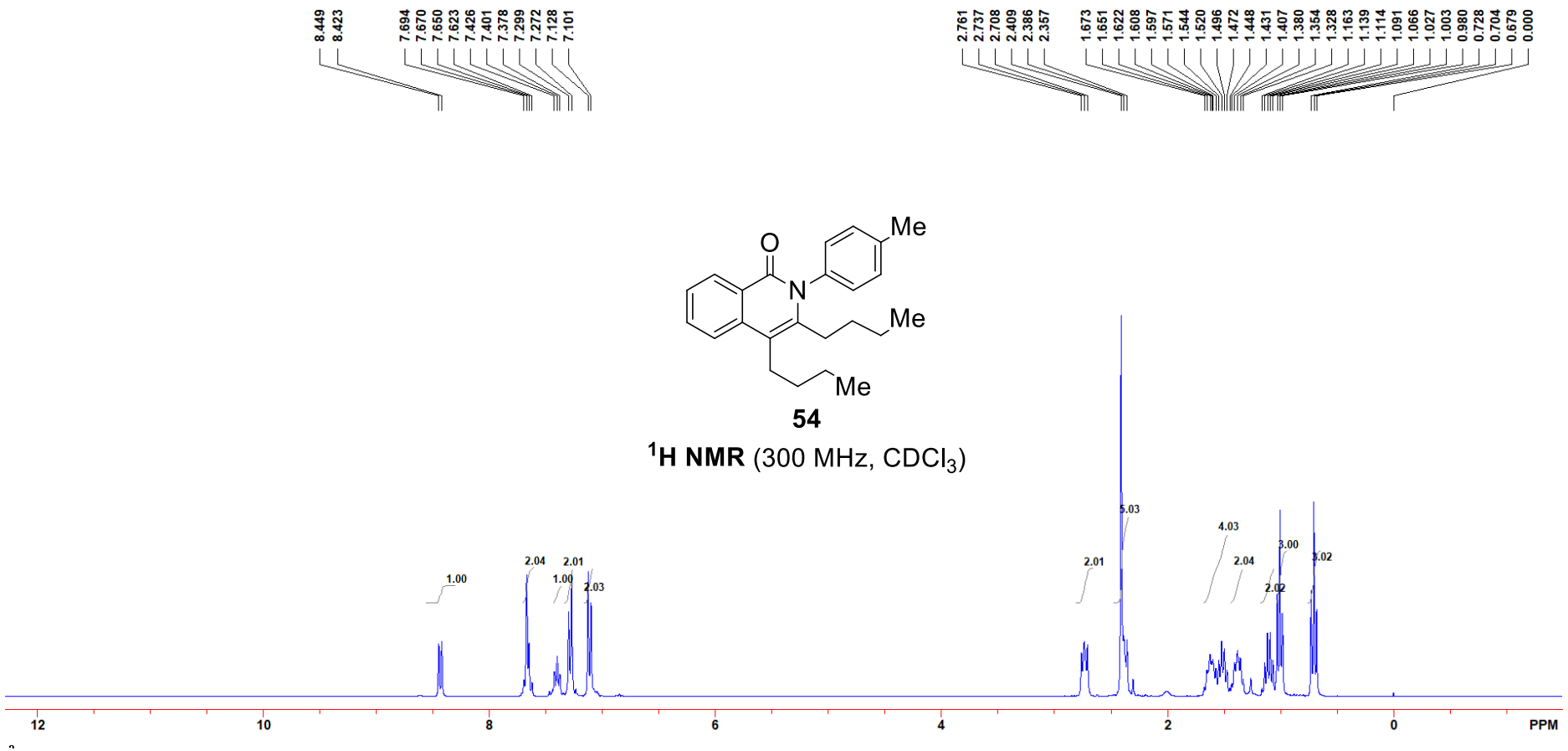
^{13}C NMR (300 MHz, $CDCl_3$)

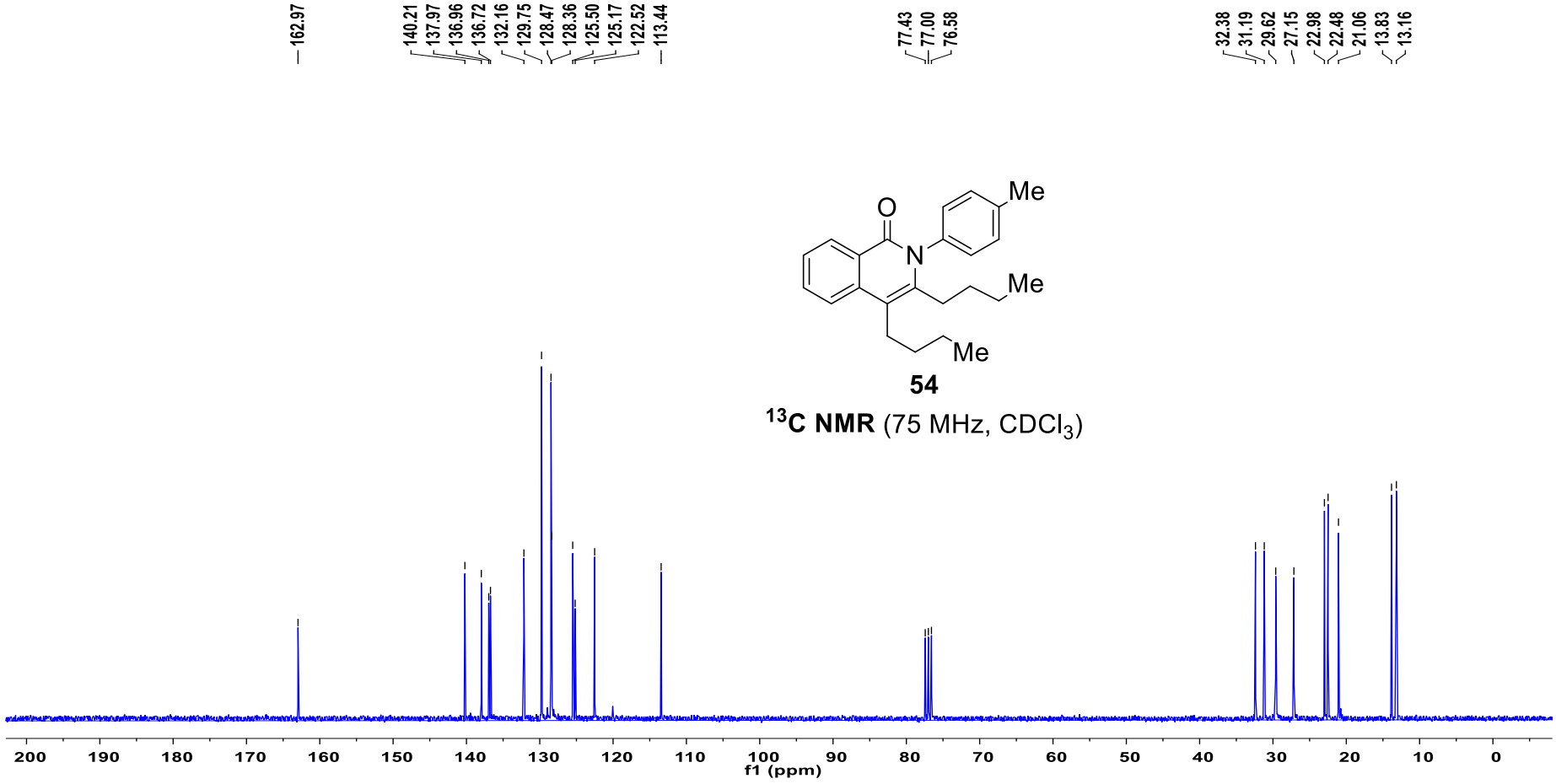

8.453
8.427
7.694
7.676
7.631
7.442
7.429
7.415
7.398
7.390
7.308
7.281
7.245
7.130
7.103
2.745
2.718
2.691
2.422
2.388
2.362
2.334
1.702
1.676
1.649
1.624
1.452
1.426
1.398
1.373
1.347
1.110
1.086
1.061
0.737
0.713
0.688
0.000
O
N
Me
Me
Me
53
1H NMR (300 MHz, CDCl3)
0.97
2.01
1.01
2.04
2.03
2.00
3.01
2.00
2.04
2.00
3.04
3.01
12
10
8
6
4
2
0 PPM



53

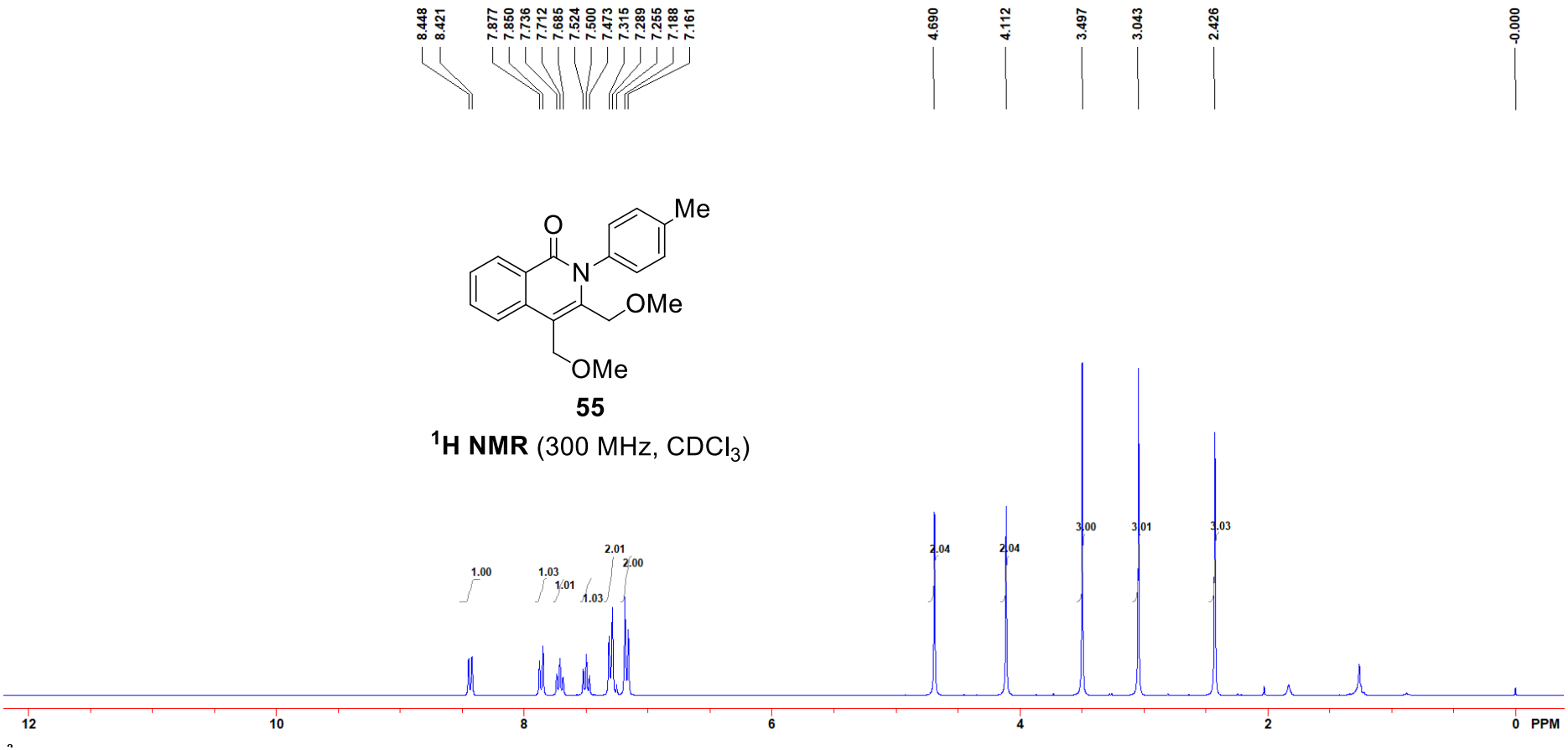
^{13}C NMR (75 MHz, $CDCl_3$)

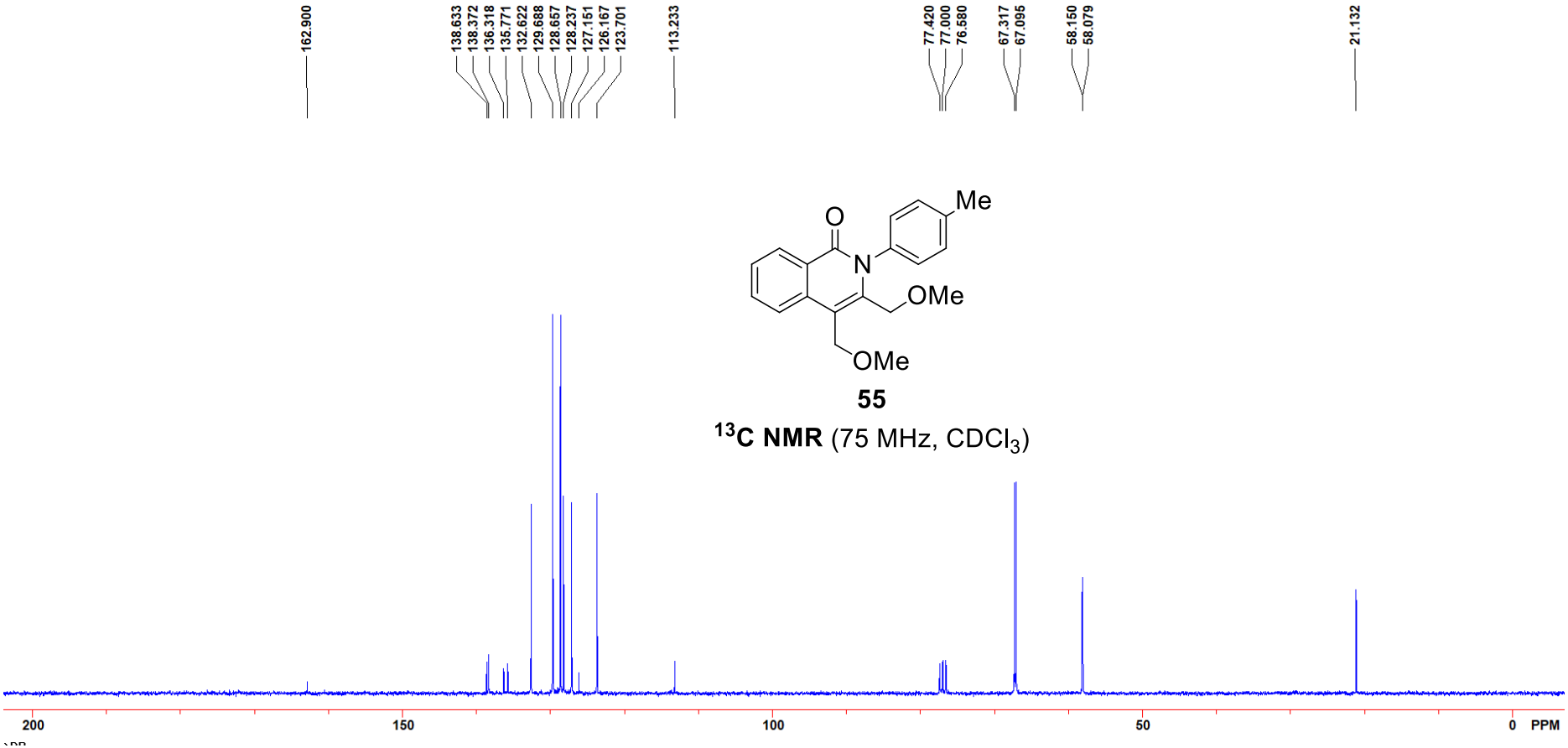

8.449
8.423
7.694
7.670
7.650
7.623
7.426
7.401
7.378
7.299
7.272
7.128
7.101
2.761
2.737
2.708
2.409
2.386
2.357
1.673
1.651
1.622
1.608
1.597
1.571
1.544
1.520
1.496
1.472
1.448
1.431
1.407
1.380
1.354
1.328
1.163
1.139
1.114
1.091
1.066
1.027
1.003
0.980
0.728
0.704
0.679
0.000
O
N
Me
Me
Me
54
1H NMR (300 MHz, CDCl3)
1.00
2.04
2.01
1.00
2.03
2.01
5.03
4.03
2.04
3.00
2.02
3.02
12
10
8
6
4
2
0
PPM



54

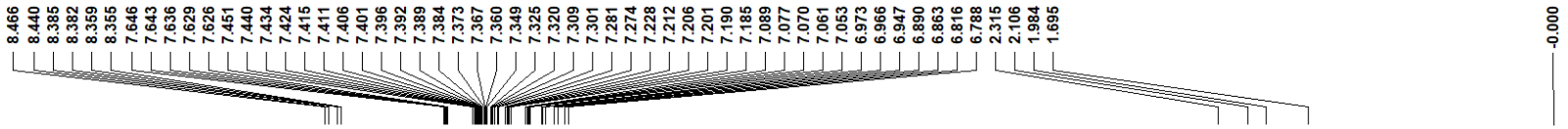
^{13}C NMR (75 MHz, $CDCl_3$)

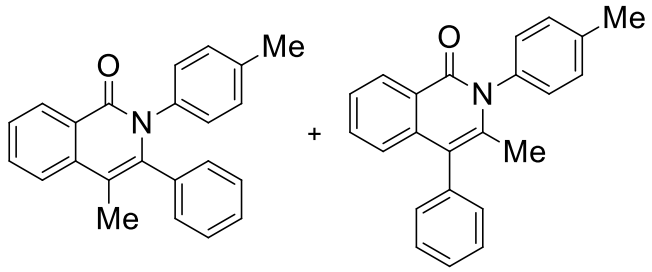

8.448
8.421
7.877
7.850
7.736
7.712
7.685
7.524
7.500
7.473
7.315
7.289
7.255
7.188
7.161
4.690
4.112
3.497
3.043
2.426
-0.000
Me
O
N
OMe
OMe
55
1H NMR (300 MHz, CDCl3)
1.00
1.03
1.01
1.03
2.01
2.00
2.04
2.04
3.00
3.01
3.03
12
10
8
6
4
2
0 PPM



55

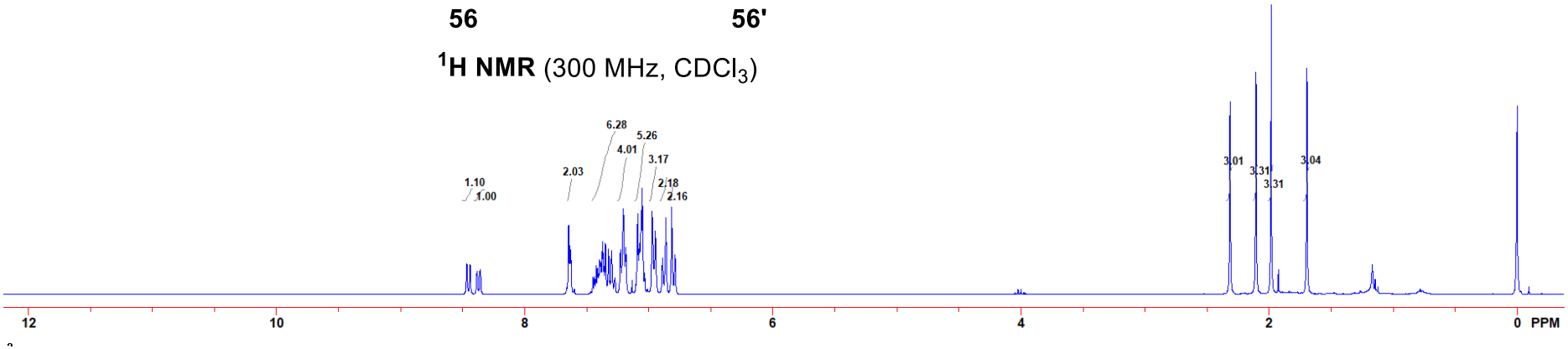
^{13}C NMR (75 MHz, $CDCl_3$)

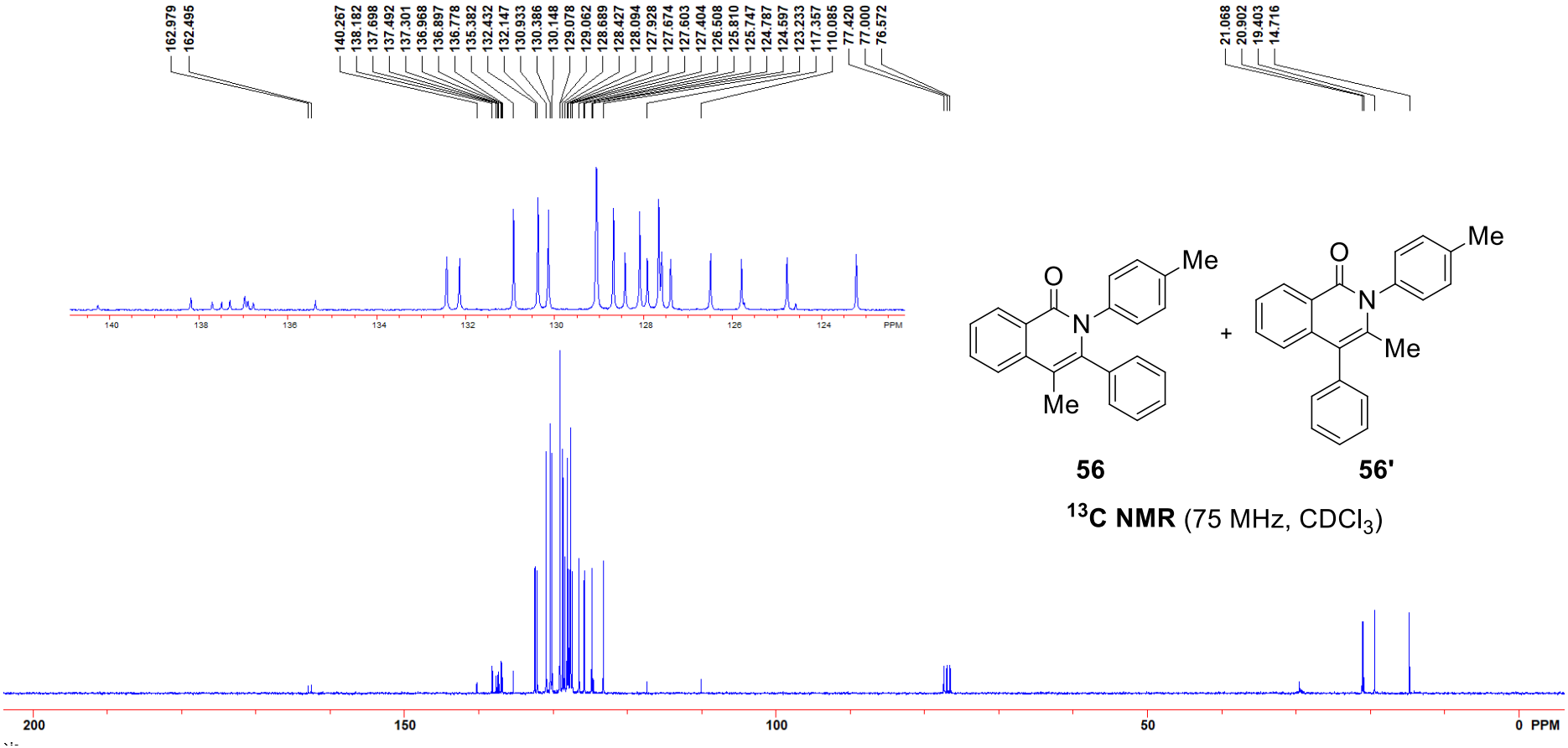




56 **56'**

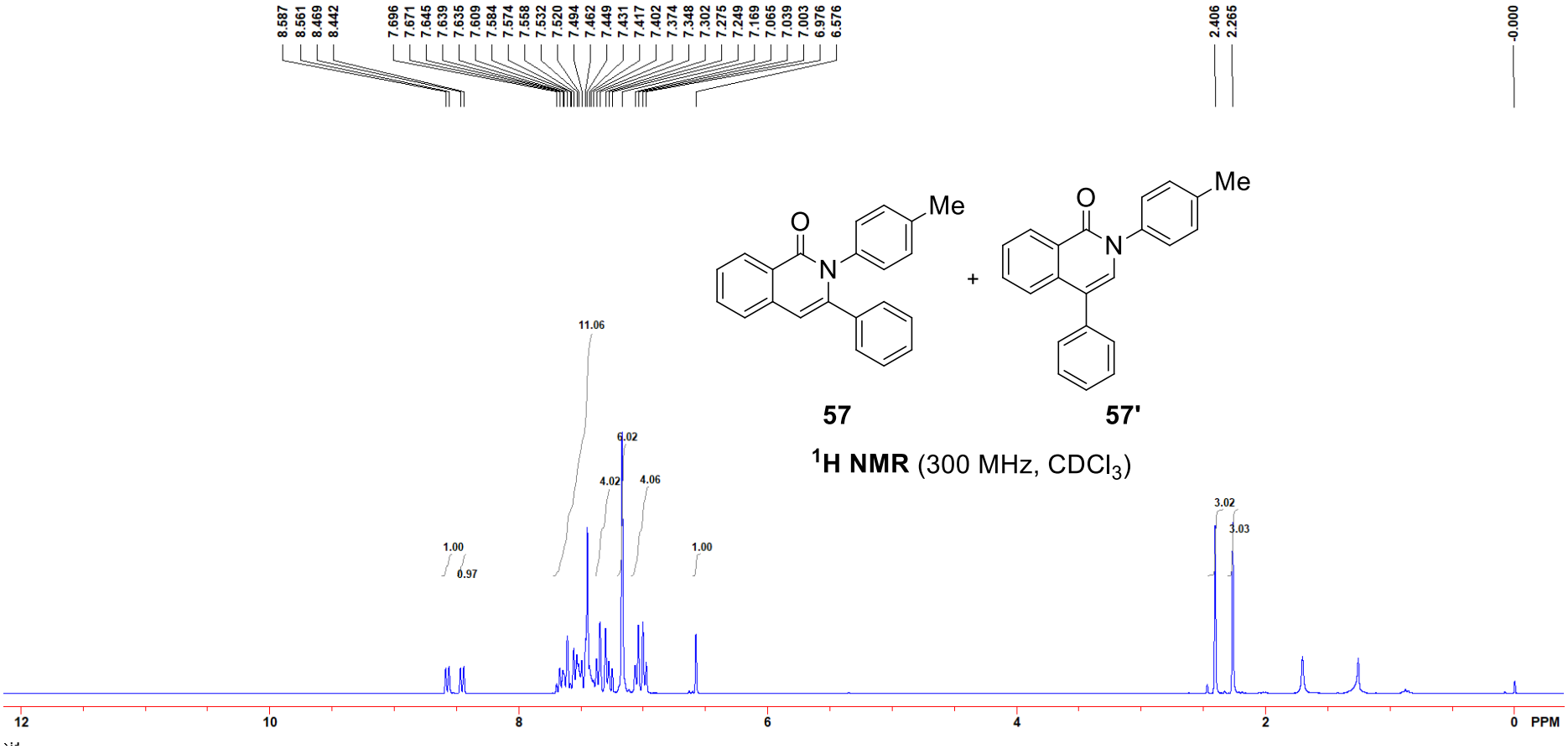
^{1}H NMR (300 MHz, $CDCl_3$)

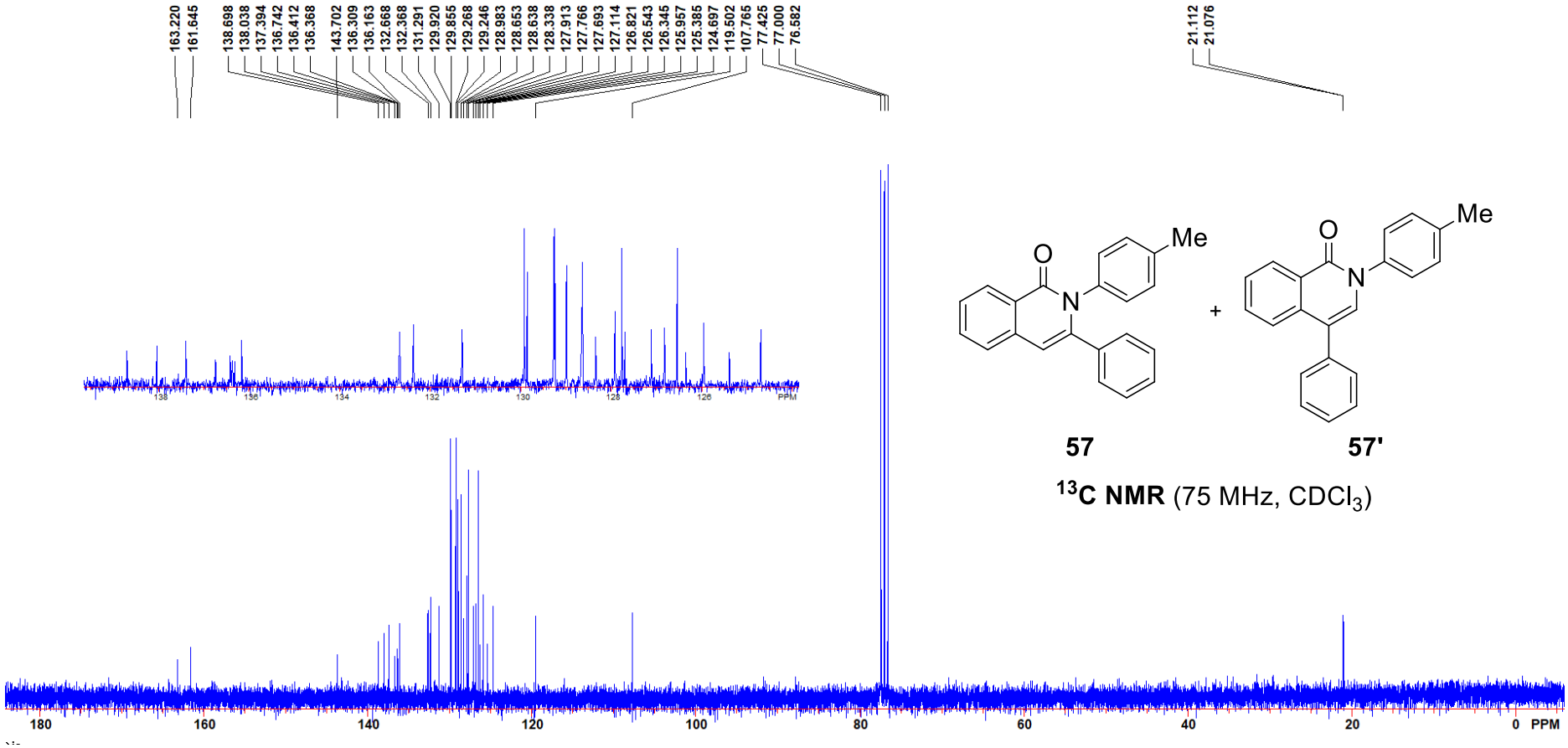




56 **56'**

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


8.587
8.561
8.469
8.442
7.696
7.671
7.645
7.639
7.635
7.609
7.584
7.574
7.558
7.532
7.520
7.494
7.462
7.449
7.431
7.417
7.402
7.374
7.348
7.302
7.275
7.249
7.169
7.065
7.039
7.003
6.976
6.576
2.406
2.265
-0.000
O
N
Me
+
57
57'
1H NMR (300 MHz, CDCl3)
11.06
6.02
4.02
4.06
1.00
0.97
1.00
3.02
3.03
12
10
8
6
4
2
0
PPM


163.220
161.645
138.698
138.038
137.394
136.742
136.412
136.368
143.702
136.309
136.163
132.668
132.368
131.291
129.920
129.855
129.268
129.246
128.983
128.853
128.638
128.338
127.913
127.766
127.693
127.114
126.821
126.543
126.345
125.957
125.385
124.697
119.502
107.765
77.425
77.000
76.582
21.112
21.076
Me
O
N
+
Me
O
N
57
57'
13C NMR (75 MHz, CDCl3)
180
160
140
120
100
80
60
40
20
0 PPM