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Supplementary Information

Synthesis of 2,3-dihydro-1*H*-pyrroles by intramolecular cyclization of *N*-(3-butynyl)- sulfonamides

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***tert*-Butyl hept-3-yn-1-ylcarbamate** (**3c**)

Di-*tert*-butyl dicarbonate (171.6 mg, 0.69 mmol) was added to a solution of 3-heptynamine (64 mg, 0.58 mmol), triethylamine (241 μL, 1.73 mmol) and CH_2Cl_2 (5 mL) at 0 ºC. The resulting mixture was stirred at rt for 16 h, added with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.69) to give **3c** (109.1 mg, 0.52 mmol, 89%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.94 (t, *J* = 7.4 Hz, 3H), 1.42 (s, 9H), 1.44–1.52 (m, 2H), 2.08–2.12 (m, 2H), 2.30–2.32 (m, 2H), 3.21 (q, *J* = 6.0 Hz, 2H), 4.78 (br, 1H); ^{13}C NMR (CDCl3, 125MHz) δ 13.4, 20.2, 20.7, 22.4, 28.4, 39.8, 79.3, 81.9, 155.7; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{12}H_{21}NO_2Na$) 234.1470, found 234.1466.

***N*-(Hept-3-yn-1-yl)acetamide** (**3d**)

Acetyl chloride (81 μL, 1.14 mmol) was added to a solution of 3-heptynamine (63.0 mg, 0.57 mmol), triethylamine (238 μL, 1.71 mmol) and CH_2Cl_2 (5 mL) at 0 ºC. The resulting mixture was stirred at rt for 16 h, added with water (7 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with $HCl_{(aq)}$ (1 N, 5 mL), sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:1; R_f 0.27) to give **3d** (109.1 mg, 0.52 mmol, 86%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.94 (t, *J* = 7.3 Hz, 3H), 1.47 (q, *J* = 7.3 Hz, 2H), 1.96 (s, 3H), 2.08–2.11 (m, 2H), 2.31–2.34 (m, 2H), 3.31 (q, *J* = 6.2 Hz, 2H), 5.83 (br, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.4, 19.6, 20.6, 22.3, 23.2, 38.6, 82.0, 82.1, 170.1; HRMS-EI (m/z) calcd for $[M]^+$ ($C_9H_{15}NO$) 153.1154, found 153.1150.

Benzyl hept-3-yn-1-ylcarbamate (**3e**)

Benzyl chloroformate (69 μL, 0.48 mmol) was added to a solution of 3-heptynamine (48.6 mg, 0.44 mmol), triethylamine (86 μL, 0.61 mmol) and CH_2Cl_2 (5 mL) at 0 ºC. The resulting mixture was stirred at rt for 9 h, added with water (7 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with $HCl_{(aq)}$ (1 *N*, 10 mL), sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.54) to give **3e** (72.2 mg, 0.29 mmol, 67%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.94 (t, *J* = 7.4 Hz, 3H), 1.44–1.51 (m, 2H), 2.09 (m, 2H), 2.35 (t, *J* = 6.2 Hz, 2H), 3.29 (q, *J* = 6.2 Hz, 2H), 5.04 (br, 1H), 5.09 (s, 2H), 7.29–7.35 (m, 5H); ^{13}C NMR ($CDCl_3$, 125MHz) δ 13.4, 20.1, 20.7, 22.3, 40.2, 66.7, 76.8, 82.1, 128.1,

128.5, 136.5, 165.2; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{15}H_{19}NO_2Na$) 268.1313, found 268.1317.

***N*-(Hept-3-yn-1-yl)benzamide** (**3f**)

Benzoyl chloride (74 µL, 0.64 mmol) was added to a solution of 3-heptynamine (47.3 mg, 0.42 mmol), triethylamine (120 µL, 0.85 mmol) and CH_2Cl_2 (5 mL) at 0 ºC. The resulting mixture was stirred at rt for 16 h, added with water (7 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.34) to give **3f** (45.4 mg, 0.21 mmol, 50%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300MHz) δ 0.94 (t, *J* = 7.3 Hz, 3H), 1.49 (q, *J* = 7.3 Hz, 2H), 2.10–2.15 (m, 2H), 2.44–2.48 (m, 2H), 3.55 (q, *J* = 6.2 Hz, 2H), 6.51 (br, 1H), 7.41–7.46 (m, 3H), 7.76 (d, *J* = 7.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.4, 19.7, 20.7, 22.3, 38.9, 76.6, 82.3, 126.9, 128.4, 128.5, 128.6, 130.1, 131.4, 167.6; HRMS-EI (m/z) calcd for $[M]^+$ ($C_{14}H_{17}NO_2$) 215.1310, found 215.1304.

Ethyl hept-3-yn-1-ylcarbamate (**3g**)

Ethyl chloroformate (62 µL, 0.64 mmol) was added to a solution of 3-heptynamine (47.7 mg, 0.42 mmol), triethylamine (120 µL, 0.85 mmol) and CH_2Cl_2 (5 mL) at 0 ºC. The resulting mixture was stirred at rt for 16 h, added with water (7 mL) and extracted with CH2Cl2 (12 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.62) to give **3g** (40.5 mg, 0.22 mmol, 52%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300MHz) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.22 (t, *J* = 7.0 Hz, 3H), 1.48 (q, *J* = 7.2 Hz, 2H), 2.07–2.12 (m, 2H), 2.31–2.35 (m, 2H), 3.26 (q, *J* = 6.2 Hz, 2H), 4.09 (q, *J* = 6.9Hz, 2H), 4.91 (br, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.4, 14.6, 20.2, 20.7, 22.3, 40.1, 60.8, 82.0, 156.5; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{10}H_{17}NO_2Na$) 206.1157, found 206.1154.

***trans*-2-(Phenylethynyl)cyclopentan-1-ol** (**6pa**)

n-Butyl lithium (2.5 *M* in hexanes, 1.9 mL, , 4.76 mmol) was added to a solution of phenylacetylene (523 µL, 4.76 mmol) and THF (25 mL) at -78 ºC. The resulting solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (441 µL, 3.57 mmol) and cyclopentene oxide (207 µL, 2.38 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (10 mL) and extracted with diethyl ether (30 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column

chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.48) to give **6pa** (440.3 mg, 2.36 mmol, 99%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.56–1.65 (m, 1H), 1.71–1.83 (m, 4H), 2.04–2.18 (m, 2H), 2.73–2.80 (m, 1H), 4.27 (dd, *J* = 6.3 Hz, *J* = 5.6 Hz, 1H), 7.24–7.28 (m, 3H), 7.36–7.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.7, 30.9, 33.4, 40.2, 79.3, 81.8, 91.5, 127.7, 128.2, 131.5; HRMS-EI (m/z) calcd for $[M]^+$ ($C_{13}H_{14}O$) 186.1045, found 186.1048.

***trans*-2-(Hex-1-yn-1-yl)cyclopentan-1-ol (6pb)**[1]

n-Butyl lithium (1.6 M in hexanes, 1.5 mL, , 2.38 mmol) was added to a solution of 1-hexyne (273 µL, 2.38 mmol) and THF (10 mL) at -78 ºC. The resulting solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (220 µL, 1.79 mmol) and cyclopentene oxide (103 µL, 1.19 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (10 mL) and extracted with diethyl ether (30 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.50) to give **6pb** (392.3 mg, 2.36 mmol, 99%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.85 (t, *J* = 7.1 Hz, 3H), 1.30–1.74 (m, 9H), 1.89–2.04 (m, 2H), 2.07–2.13 (m, 2H), 2.43–2.50 (m, 1H), 3.31 (q, *J* = 5.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.5, 18.4, 21.5, 21.8, 31.0, 31.9, 33.1, 39.6, 79.4, 81.7.

***trans*-2-((Trimethylsilyl)ethynyl)cyclopentan-1-ol (6pc)**

n-Butyl lithium (2.5 M in hexanes, 1.9 mL, , 4.76 mmol) was added to a solution of trimethylsilylacetylene (673 µL, 4.76 mmol) and THF (10 mL) at -78 ºC. The resulting solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (441 µL, 3.57 mmol) and cyclopentene oxide (208 µL, 2.38 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (10 mL) and extracted with diethyl ether (30 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:4; R_f 0.67) to give **6pc** (400.6 mg, 2.19 mmol, 92%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.09 (s, 9H), 1.48–1.54 (m, 1H), 1.57–1.72 (m, 3H), 1.91–2.05 (m, 2H), 2.53 (dt, *J* = 7.6 Hz, *J* = 5.5 Hz, 2H), 4.13 (q, *J* = 5.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 0.10, 21.8, 31.1, 33.4, 40.5, 79.2, 85.5, 108.9; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{10}H_{19}OSi$) 183.1205, found 183.1204.

***trans*-2-(Phenylethynyl)cyclohexanol (6ha)**[2]

n-Butyl lithium (1.6 *M* in hexanes, 0.9 mL, 1.54 mmol) was added to a solution of phenylacetylene (168 µL, 1.53 mmol) and THF (6 mL) at -78 ºC. The resulting

solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (180 µL, 1.53 mmol) and cyclohexene oxide (103 µL, 1.02 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (10 mL) and extracted with diethyl ether (30 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.75) to give **6ha** (194.3 mg, 0.97 mmol, 95%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500MHz) δ 1.17–1.38 (m, 3H), 1.42–1.50 (m, 1H), 1.66–1.72 (m, 1H), 1.75–1.79 (m, 1H), 2.02–2.08 (m, 2H), 2.32 (br, 1H), 2.41 (m, 1H), 3.53 (td, *J* = 9.9 Hz, *J* = 3.8 Hz, 1H), 7.26–7.28 (m, 3H), 7.38–7.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 125MHz) δ 24.2, 24.9, 31.0, 33.1, 39.6, 73.6, 82.7, 90.8, 123.3, 127.9, 128.0, 128.8, 128.9, 131.7.

***trans*-2-(Hex-1-yn-1-yl)cyclohexanol (6hb)**[3]

n-Butyl lithium (1.6 *M* in hexanes, 1.2 mL, 2.04 mmol) was added to a solution of 1-hexyne (234 µL, 2.04 mmol) and THF (8 mL) at -78 ºC. The resulting solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (252 µL, 2.04 mmol) and cyclohexene oxide (103 µL, 1.02 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (10 mL) and extracted with diethyl ether (30 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:9; R_f 0.50) to give **6hb** (160.3 mg, 0.89 mmol, 87%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.88 (t, *J* = 7.1 Hz, 3H), 1.15–1.46 (m, 7H), 1.59–1.74 (m, 3H), 1.89–2.02 (m, 2H), 2.14–2.19 (m, 3H), 2.31 (br, 1H), 3.35 (ddd, *J* = 9.7 Hz, *J* = 9.7 Hz, *J* = 4.0 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 18.4, 24.9, 24.3, 25.0, 31.4, 31.9, 33.0, 39.1, 73.9, 81.1, 82.9.

***trans*-2-((Trimethylsilyl)ethynyl)cyclohexanol (6hc)**[4]

n-Butyl lithium (2.5 *M* in hexanes, 2.4 mL, , 6.12 mmol) was added to a solution of trimethylsilylacetylene (865 µL, 6.12 mmol) and THF (30 mL) at -78 ºC. The resulting solution was stirred at -78 ºC for 30 min, added with boron trifluoride diethyl etherate (567 µL, 4.59 mmol) and cyclohexene oxide (309 µL, 3.06 mmol), stirred for another 4 h at -78 ºC, quenched with sat. $NH_4Cl_{(aq)}$ (15 mL) and extracted with diethyl ether (45 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was further purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.59) to give **6hc**[4] (571.2 mg, 2.91 mmol, 95%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.11 (s, 9H),

1.09–1.37 (m, 4H), 1.59–1.63 (m, 1H), 1.69–1.72 (m, 1H), 1.91–1.98 (m, 2H), 2.16–2.21 (m, 1H), 2.36 (br, 1H), 3.37-3.41 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 0.15, 24.2, 24.8, 30.9, 32.9, 40.0, 73.4, 86.8, 107.9.

2-(*cis*-2-(Phenylethynyl)cyclopentyl)isoindoline-1,3-dione (**7pa**)

Diisopropyl azodicarboxylate (722 μL, 3.57 mmol) was added to a solution of **6pa** (443.3 mg, 2.38 mmol), triphenylphosphine (936.4 mg, 3.57 mmol) and THF (25 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (525.3 mg, 3.57 mmol), stirred for another 36 h at rt and concentrated. The crude product was was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.53) to give **7pa** (398.6 mg, 1.26 mmol, 53%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500MHz) δ 1.52–1.63 (m, 1H), 2.09–2.15 (m, 3H), 2.17–2.25 (m, 1H), 2.59–2.67 (m, 1H), 3.22 (dt, *J* = 9.5 Hz, *J* = 7.7 Hz, 1H), 4.89 (dd, *J* = 16.3 Hz, *J* = 8.8 Hz, 1H), 6.86 (d, *J* = 7.3 Hz, 2H), 6.99–7.09 (m, 3H), 7.62–7.65 (m, 2H), 7.77–7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 25.3, 27.9, 33.6, 36.8, 53.4, 83.6, 88.9, 122.9, 123.2, 127.3, 127.8, 131.0, 131.9, 133.7, 168.7; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{21}H_{17}NO_2Na$) 338.1157, found 338.1160.

2-(*cis*-2-(Hex-1-yn-1-yl)cyclopentyl)isoindoline-1,3-dione (**7pb**)[1]

Diisopropyl azodicarboxylate (188 μL, 1.20 mmol) was added to a solution of **6pb** (132.4 mg, 0.80 mmol), triphenylphosphine (314.7 mg, 1.20 mmol) and THF (8 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (176.6 mg, 1.20 mmol), stirred for another 16 h at rt and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.66) to give **7pb** (130.2 mg, 0.44 mmol, 55%) as a colorless, viscous liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.49 (t, *J* = 7.0Hz, 3H), 0.78–0.97 (m, 4H), 1.35–1.53 (m, 1H), 1.72 (td, *J* = 6.9 Hz, *J* = 2.3 Hz, 2H), 1.92–2.06 (m, 4H), 2.39–2.51 (m, 1H), 2.85–2.93 (m, 1H), 4.03 (dt, *J* = 6.9 Hz, *J* = 9.0 Hz, 1H), 7.59–7.65 (m, 2H), 7.71–7.77 (m, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 13.3, 18.0, 21.4, 25.2, 27.8, 30.5, 33.7, 36.3, 53.1, 78.7, 83.5, 122.8, 131.9, 133.5, 168.6.

2-(*cis*-2-((Trimethylsilyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**7pc**)

Diisopropyl azodicarboxylate (223 μL, 1.42 mmol) was added to a solution of **6pc** (129.4 mg, 0.71 mmol), triphenylphosphine (372.3 mg, 1.42 mmol) and THF (7 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (208.9 mg, 1.42 mmol), stirred for another 36 h at rt and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.67) to give **7pc** (132.6 mg, 0.42 mmol, 59%) as a colorless solid. M.p. 118.0–120.0 ºC; ^{1}H NMR ($CDCl_3$, 500MHz) δ

-0.31 (s, 9H), 1.44–1.54 (m, 1H), 2.00–2.16 (m, 4H), 2.49–2.57 (m, 1H), 2.95–3.00 (m, 1H), 4.81 (dt, *J* = 9.3 Hz, *J* = 7.1 Hz, 1H), 7.65–7.68 (m, 2H), 7.78–7.81 (m, 2H); ^{13}C NMR ($CDCl_3$, 125MHz) δ -0.61, 25.6, 27.9, 33.7, 37.3, 53.2, 87.6, 105.7, 122.9, 132.1, 133.7, 168.6; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{18}H_{22}NO_2Si$) 312.1420, found, 312.1414.

***cis*-2-(Phenylethynyl)cyclohexyl)isoindoline-1,3-dione (7ha)**[2]

Diisopropyl azodicarboxylate (297 μL, 1.51 mmol) was added to a solution of **6ha** (150.8 mg, 0.75 mmol), triphenylphosphine (395.0 mg, 1.51 mmol) and THF (10 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (221.6 mg, 1.51 mmol), stirred for another 36 h at rt and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.22) to give **7ha** (158.4 mg, 0.48 mmol, 64%) as a colorless solid. M.p. 152–153 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.29–1.38 (m, 1H), 1.59 (d, *J* = 11.7 Hz, 1H), 1.70–1.79 (m, 2H), 1.86 (d, *J* = 11.6 Hz, 1H), 1.95–2.00 (m, 2H), 3.19 (dq, *J* = 13.1 Hz, *J* = 3.6 Hz, 1H), 3.31 (m, 1H), 4.20 (dt, *J* = 13.1 Hz, *J* = 3.8 Hz, 1H), 7.23–7.26 (m, 3H), 7.38–7.40 (m, 2H), 7.67 (dd, *J* = 5.5 Hz, *J* = 3.0 Hz, 2H), 7.81 (dd, *J* = 5.4 Hz, *J* = 3.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 25.2, 26.4, 31.0, 33.9, 55.2, 83.6, 89.4, 123.0, 123.8, 127.6, 128.1, 131.6, 131.9, 133.8, 168.6.

2-(*cis*-2-(Hex-1-yn-1-yl)cyclohexyl)isoindoline-1,3-dione (7hb)[3]

Diisopropyl azodicarboxylate (903 μL, 4.58 mmol) was added to a solution of **6hb** (551.7 mg, 3.06 mmol), triphenylphosphine (1.20 g, 4.58 mmol) and THF (10 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (674.6 mg, 4.58 mmol), stirred for another 36 h at rt and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:7; R_f 0.40) to give **7hb**[3] (565.4 mg, 1.83 mmol, 60%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.77 (t, *J* = 7.2 Hz, 3H), 1.20–1.38 (m, 7H), 1.49–1.53 (m, 1H), 1.55–1.66 (m, 2H), 1.73–1.85 (m, 3H), 2.05 (m, 2H), 3.00–3.06 (m, 1H), 4.03 (dt, *J* = 13.0 Hz, *J* = 4.0 Hz, 1H), 7.60–7.65 (m, 2H), 7.71-7.77 (m, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 13.5, 18.3, 20.9, 21.8, 24.9, 26.2, 30.7, 31.0, 32.9, 55.3, 80.0, 83.5, 122.8, 131.8, 133.5, 168.5.

2-(*cis*-2-((trimethylsilyl)ethynyl)cyclohexyl)isoindoline-1,3-dione (7hc)

Diisopropyl azodicarboxylate (857 μL, 4.35 mmol) was added to a solution of **6hc** (570.0 mg, 2.90 mmol), triphenylphosphine (1.14 g, 4.35 mmol) and THF (30 mL) at 0 ºC. The resulting mixture was stirred at 0 ºC for 30 min, added with phthalimide (640.0 mg, 4.35 mmol), stirred for another 36 h at rt and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:8; R_f 0.46) to give **7hc** (717.4 mg, 2.20 mmol,

76%) as a colorless solid. M.p. 130–131 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.06 (s, 9H), 1.12–1.30 (m, 1H), 1.54–1.71 (m, 3H), 1.77–1.93 (m, 3H), 3.05–3.13 (m, 2H), 4.07 (dt, *J* = 13.0 Hz, *J* = 3.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75MHz) δ -0.22, 20.9, 24.9, 26.2, 30.5, 33.8, 55.1, 88.1, 106.2, 122.8, 122.9, 131.8, 131.9, 133.6, 133.7, 168.4, 168.5; HRMS-MALDI (m/z) calcd for $[M+H]^+$ ($C_{19}H_{24}NO_2Si$) 326.1571, found, 326.1571.

4-Methyl-*N*-(*cis*-2-(phenylethynyl)cyclopentyl)benzenesulfonamide (**8pa**)

A solution of **7pa** (150.6 mg, 0.48 mmol), hydrazine monohydrate (116 μL, 2.4 mmol) and methanol (5 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (5 mL) and added with triethylamine (268 μL, 1.92 mmol). The above solution was added with *p*-toluenesulfonyl chloride (183.2 mg, 0.96 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (3 mL) and extracted with CH_2Cl_2 (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.57) to give **8pa** (127.6 mg, 0.37 mmol, 78%) as a colorless solid. M.p. 113.5–114.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.58–1.61 (m, 1H), 1.75–1.79 (m, 1H), 1.83–1.93 (m, 4H), 2.43 (s, 3H), 2.85–2.89 (m, 1H), 3.66–3.69 (m, 1H), 5.08 (d, *J* = 8.1 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 2H), 7.32–7.34 (m, 3H), 7.36–7.38 (m, 2H), 7.76 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125MHz) δ 20.9, 21.5, 30.7, 31.4, 35.4, 56.7, 85.1, 88.1, 122.8, 127.1, 128.2, 128.3, 129.6, 131.6, 137.7, 143.3; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{20}H_{21}NO_2NaS$) 362.1191, found 362.1190.

***N*-(*cis*-2-(Hex-1-yn-1-yl)cyclopentyl)-4-methylbenzenesulfonamide (8pb**)

A solution of **7pb** (118.2 mg, 0.41 mmol), hydrazine monohydrate (97 μL, 2.0 mmol) and methanol (4 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (4 mL) and added with triethylamine (223 μL, 1.60 mmol). The above solution was added with *p*-toluenesulfonyl chloride (152.5 mg, 0.80 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8

mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.57) to give **8pb** (85.7 mg, 0.27 mmol, 67%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.86–0.90 (m, 3H), 1.32–1.36 (m, 2H), 1.38–1.46 (m, 3H), 1.60–1.67 (m, 2H), 1.67–1.77 (m, 3H), 2.09–2.13 (m, 2H), 2.38 (s, 3H), 2.53–2.54 (m, 1H), 3.42–3.47 (m, 1H), 4.95 (d, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 8.2 Hz, 2H), 7.72 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125MHz) δ 13.5, 18.3, 20.9, 21.4, 21.9, 30.8, 30.9, 31.1, 34.8, 56.3, 78.4, 85.4, 127.1, 129.5, 137.6, 143.2; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{18}H_{25}NO_2SNa$) 342.1504, found, 342.1506.

4-Methyl-*N*-(*cis*-2-(phenylethynyl)cyclohexyl)benzenesulfonamide (8ha)[2]

A solution of **7ha** (153.2 mg, 0.47 mmol), hydrazine monohydrate (25 μL, 0.51 mmol) and methanol (12 mL) was heated to reflux for 3 h, added with conc. hydrochloric acid (3 mL) at 0 ºC, filtered and concentrated. The residue was diluted with hydrochloric acid (2 *N*, 15 mL), washed with ethyl acetate (15 mL × 3), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (324 μL, 2.33 mmol). The above solution was added with *p*-toluenesulfonyl chloride (354.9 mg, 1.86 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with hydrochloric acid (1 *N*, 5 mL), sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:7; R_f 0.31) to give **8ha** (118.3 mg, 0.33 mmol, 72%) as a colorless solid. M.p. 144.0–145.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.21–1.26 (m, 1H), 1.41–1.49 (m, 2H), 1.56–1.57 (m, 1H), 1.60–1.64 (m, 2H), 1.67–1.71 (m, 1H), 1.82–1.85 (m, 1H), 2.40 (s, 3H), 2.87 (q, *J* = 3.6 Hz, 1H), 3.27–3.33 (m, 1H), 4.79 (d, *J* = 9.2 Hz, 1H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.29–7.30 (m, 3H), 7.36–7.38 (m, 2H), 7.76 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 21.5, 24.7, 30.0, 30.3, 34.6, 53.9, 84.9, 88.0, 123.0, 126.9, 127.0, 128.1, 128.3, 129.7, 131.7, 138.6, 143.2.

***N*-(*cis*-2-(Hex-1-yn-1-yl)cyclohexyl)-4-methylbenzenesulfonamide (8hb)**[3]

A solution of **7hb** (145.4 mg, 0.47 mmol), hydrazine monohydrate (114 μL, 2.35 mmol) and methanol (12 mL) was heated to reflux for 3 h, added with conc. hydrochloric acid (3 mL) at 0 ºC, filtered and concentrated. The residue was diluted with hydrochloric acid (2 *N*, 15 mL), washed with ethyl acetate (15 mL × 3), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether

(25 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (197 μL, 1.41 mmol). The above solution was added with *p*-toluenesulfonyl chloride (134.5 mg, 0.71 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with hydrochloric acid (1 *N*, 5 mL), sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.64) to give **8hb** (104.6 mg, 0.32 mmol, 67%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.90 (t, *J* = 7.2 Hz, 3H), 1.35–1.48 (m, 7H), 1.53–1.71 (m, 6H), 2.14 (dt, *J* = 6.8 Hz, *J* = 2.1 Hz, 2H), 2.40 (s, 3H), 2.57 (d, *J* = 2.1 Hz, 1H), 3.10–3.21 (m, 1H), 4.77 (d, *J* = 9.0 Hz, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.74 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 18.4, 20.7, 21.5, 22.0, 24.7, 29.9, 30.5, 31.1, 33.8, 55.7, 78.1, 85.1, 126.8, 129.6, 138.6, 143.1.

***N*-(*cis*-2-(Phenylethynyl)cyclopentyl)methanesulfonamide (9pa)**

A solution of **7pa** (150.7 mg, 0.48 mmol), hydrazine monohydrate (116 μL, 2.4 mmol) and methanol (5 mL) was heated to reflux for 3 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (5 mL) and added with triethylamine (268 μL, 1.92 mmol). The above solution was added with methanesulfonyl chloride (75 μL, 0.96 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (3 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:2; R_f 0.47) to give **9pa** (89.7 mg, 0.34 mmol, 70%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.57–1.69 (m, 1H), 1.70–1.81 (m, 1H), 1.86–2.08 (m, 4H), 2.98 (s, 3H), 3.15–3.21 (m, 1H), 3.79–3.89 (m, 1H), 4.86 (d, *J* = 8.6 Hz, 1H), 7.26–7.30 (m, 3H), 7.37–7.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.9, 30.7, 31.5, 35.7, 41.4, 56.8, 84.8, 88.2, 122.7, 128.0, 128.1, 131.5; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{14}H_{18}NO_2S$) 264.1058, found 264.1053.

***N*-(*cis*-2-(hex-1-yn-1-yl)cyclopentyl)methanesulfonamide (9pb)**

A solution of **7pb** (118.2 mg, 0.40 mmol), hydrazine monohydrate (97 μL, 2.0 mmol) and methanol (4 mL) was heated to reflux for 12 h, added with conc.

hydrochloric acid (2 mL) at 0 ºC, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (4 mL) and added with triethylamine (223 µL, 1.60 mmol). The above solution was added with methanesulfonyl chloride (62 µL, 0.80 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:2; R_f 0.63) to give **9pb** (61.4 mg, 0.25 mmol, 62%) as a colorless liquid. ^{1}H NMR ($CDCl_3$,500 MHz) δ 0.87 (t, *J* = 7.3Hz, 3H), 1.32–1.39 (m, 2H), 1.41–1.47 (m, 2H), 1.51–1.58 (m, 1H), 1.63–1.70, (m, 1H), 1.72–1.89 (m, 3H), 1.91–1.98 (m, 1H), 2.15 (dt, *J* = 7.0 Hz, *J* = 2.1 Hz, 2H), 2.89–2.93 (m, 1H), 2.95 (s, 3H), 3.66–3.72 (m, 1H), 4.71 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.5, 18.3, 21.0, 21.9, 30.9, 31.0, 31.6, 35.2, 41.4, 56.6, 78.5, 85.5; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{12}H_{21}NO_2SNa$) 266.1191, found, 266.1193.

***N*-(*cis*-2-(Phenylethynyl)cyclohexyl)methanesulfonamide** (**9ha**)

A solution of **7ha** (153.2 mg, 0.47 mmol), hydrazine monohydrate (25 µL, 0.51 mmol) and methanol (10 mL) was heated to reflux for 3 h, added with conc. hydrochloric acid (3 mL) at 0 ºC, filtered and concentrated. The residue was diluted with hydrochloric acid (2 *N*, 15 mL), washed with ethyl acetate (15 mL × 3), basified with $NaOH_{(aq)}$ (15%, ~5 mL) and extracted with diethyl ether (25 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (343 µL, 2.46 mmol). The above solution was added with methanesulfonyl chloride (83 µL, 1.07 mmol) at 0 ºC, stirred at rt for 16 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with hydrochloric acid (1 *N*, 5 mL), sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 2:3; R_f 0.40) to give **9ha** (145.5 mg, 0.52 mmol, 64%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.23–1.38 (m, 1H), 1.49–1.55 (m, 1H), 1.59–1.66 (m, 3H), 1.73–1.83 (m, 2H), 1.91–1.95 (m, 1H), 2.99 (s, 3H), 3.20 (q, *J* = 3.4 Hz, 1H), 3.36–3.51 (m, 1H), 4.68 (d, *J* = 9.1 Hz, 1H), 7.28–7.31 (m, 3H), 7.38–7.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.9, 24.6, 30.2, 30.4, 35.1, 42.4, 54.0, 84.9, 88.0, 122.8, 128.1, 128.2, 128.3, 128.4, 131.6; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{15}H_{20}NO_2S$) 278.1215, found 278.1216.

***N*-(*cis*-2-(Hex-1-yn-1-yl)cyclohexyl)methanesulfonamide (9hb)**

A solution of 2-(1-hexynyl)-cyclohexanamine (51.3 mg, 0.29 mmol, prepared from the procedure for **8hb**), triethylamine (120 μL, 0.86 mmol) and dichloromethane (5 mL) was added with methanesulfonyl chloride (41 μL, 0.37 mmol) at 0 ºC, stirred at rt for 12 h, added with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.32) to give **9hb** (48.7 mg, 0.19 mmol, 66%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.89 (t, *J* = 7.1 Hz, 3H), 1.31–1.51 (m, 7H), 1.53–1.81 (m, 6H), 2.16 (dt, *J* = 6.9 Hz, *J* = 2.1 Hz, 2H), 2.92, (br, 1H), 2.96 (s, 3H), 3.27–3.37 (m, 1H), 4.57 (d, *J* = 9.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 13.5, 18.3, 20.7, 22.0, 24.6, 30.2, 30.5, 31.1, 34.4, 42.3, 53.9, 78.1, 85.1; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{13}H_{24}NO_2S$) 258.1528, found, 258.1531.

4-Methyl-*N*-(*cis*-2-((4-nitrophenyl)ethynyl)cyclopentyl)benzenesulfonamide (8pc)

A solution of **11pc** (38.4 mg, 0.11 mmol), hydrazine monohydrate (30 μL, 0.55 mmol) and methanol (1 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~3 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (1 mL) and added with triethylamine (62 μL, 0.44 mmol). The above solution was added with *p*-toluenesulfonyl chloride (41.9 mg, 0.22 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (1 mL) and extracted with CH_2Cl_2 (5 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.46) to give **8pc** (32.6 mg, 0.08 mmol, 77%) as a light yellow solid. M.p. 152.0–153.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.51–1.58 (m, 1H), 1.62–1.68 (m, 1H), 1.76–1.94 (m, 4H), 2.38 (s, 3H), 2.94 (dd, *J* = 10.9 Hz, *J* = 6.8 Hz, 1H), 3.69 (quintet, *J* = 7.5 Hz, 1H), 5.10 (d, *J* = 8.6 Hz, 1H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.46 (d, *J* = 8.6 Hz, 2H), 7.75 (d, *J* = 8.2 Hz, 2H), 8.11 (d, *J*= 8.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 21.8, 30.5, 31.2, 35.8, 56.9, 83.2, 94.3, 123.5, 127.1, 129.6, 129.9, 132.4, 137.7, 143.5, 146.9; HRMS-ESI neg (m/z) calcd for $[M-H]^-$ ($C_{20}H_{19}N_2O_4S$) 383.1066, found, 383.1068.

***N*-(*cis*-2-((3-Chlorophenyl)ethynyl)cyclopentyl)-4-methylbenzenesulfonamide** (**8pi**)

A solution of **11pi** (120.0 mg, 0.34 mmol), hydrazine monohydrate (83 μL, 1.72 mmol) and methanol (3.5 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2.5 mL) and added with triethylamine (190 μL, 1.36 mmol). The above solution was added with *p*-toluenesulfonyl chloride (129.6 mg, 0.68 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.47) to give **8pi** (96.3 mg, 0.26 mmol, 76%) as a colorless solid. M.p. 118.0–119.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.49-1.57 (m, 1H), 1.64–1.71 (m, 1H), 1.76–1.89 (m, 4H), 2.38 (s, 3H), 2.83–2.87 (m, 1H), 3.62–3.68 (m, 1H), 5.02 (s, 1H), 2.38 (s, 3H), 7.19–7.27 (m, 6H), 7.75 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 21.5, 30.6, 31.4, 35.5, 56.8, 83.5, 89.6, 124.6, 127.1, 128.4, 129.5, 129.6, 129.7, 131.5, 134.0, 137.7, 143.3; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{20}H_{20}NO_2SClNa$) 396.0801, found, 396.0793.

***N*-(*cis*-2-((2-chlorophenyl)ethynyl)cyclopentyl)-4-methylbenzenesulfonamide** (**8pj**)

A solution of **11pj** (84.9 mg, 0.24 mmol), hydrazine monohydrate (60 μL, 1.21 mmol) and methanol (2.5 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2 mL) and added with triethylamine (123 μL, 0.88 mmol). The above solution was added with *p*-toluenesulfonyl chloride (83.9 mg, 0.44 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.44) to give **8pj** (70.7 mg, 0.19 mmol, 79%) as a colorless solid. M.p. 109.0–110.0 ºC; ^{1}H

NMR ($CDCl_3$, 500 MHz) δ 1.49–1.59 (m, 1H), 1.68–1.75 (m, 1H), 1.78–1.90 (m, 4H), 2.37 (s, 3H), 2.84 (dd, *J* = 11.1 Hz, *J* = 6.1 Hz, 1H), 3.64–3.70 (m, 1H), 5.22 (d, *J* = 8.7 Hz, 1H), 7.16–7.24 (m, 4H), 7.34–7.38 (m, 2H), 7.76 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 21.4, 30.5, 31.0, 35.5, 56.8, 81.9, 94.0, 122.7, 126.5, 127.1, 129.1, 129.2, 129.5, 133.0, 135.8, 137.7, 143.2; HRMS-APCI (m/z) calcd for $[M+Na]^+$ ($C_{20}H_{20}NO_2SClNa$) 396.0801, found, 396.0806.

***N*-(*cis*-2-((2-Fluorophenyl)ethynyl)cyclopentyl)-4-methylbenzenesulfonamide (8pk)**

A solution of **11pk** (90.2 mg, 0.27 mmol), hydrazine monohydrate (66 μL, 1.35 mmol) and methanol (3 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2 mL) and added with triethylamine (123 μL, 0.88 mmol). The above solution was added with *p*-toluenesulfonyl chloride (83.9 mg, 0.44 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.44) to give **8pk** (74.4 mg, 0.21 mmol, 77%) as a colorless solid. M.p. 100.0–101.0 ºC; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.48–1.58 (m, 1H), 1.68–1.74 (m, 1H), 1.77–1.88 (m, 4H), 2.36 (s, 3H), 2.82 (q, *J* = 6.1 Hz, 1H), 3.62–3.68 (m, 1H), 5.09 (d, *J* = 8.5 Hz, 1H), 7.03–7.07 (m, 2H), 7.21–7.33 (m, 4H), 7.76 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.8, 21.4, 30.5, 31.2, 35.4, 56.7, 78.3, 93.8, 111.3 (d, J_{C-F} = 15.4 Hz), 115.3 (d, J_{C-F} = 20.9 Hz), 123.9 (d, J_{C-F} = 3.1 Hz), 127.1, 129.5, 129.8 (d, J_{C-F} = 7.8 Hz), 133.1, 137.6, 143.2, 162.8 (d, J_{C-F} = 248.7 Hz); HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{20}H_{20}NO_2FSNa$) 380.1096, found, 380.1102.

4-Methyl-*N*-(*cis*-2-((4-nitrophenyl)ethynyl)cyclohexyl)benzenesulfonamide (8hc)

A solution of **11hc** (101.7 mg, 0.27 mmol), hydrazine monohydrate (66 μL, 1.36 mmol) and methanol (15 mL) was heated to reflux for 3 h, filtered and concentrated. The residue was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (92 μL, 0.67 mmol). The above solution was added with *p*-toluenesulfonyl chloride (51.0 mg, 0.27 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaHCO_{3(aq)}$ (10 mL) and sat.

$NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.38) to give **8hc** (76.1 mg, 0.19 mmol, 70%) as a colorless solid. M.p. 182.0–184.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.20–1.25 (m, 1H), 1.45–1.50 (m, 1H), 1.53–1.60 (m, 2H), 1.62–1.69 (m, 1H), 1.83–1.86 (m, 1H), 2.37 (s, 3H), 3.00 (d, *J* = 3.2 Hz, 1H), 3.24–3.34 (m, 1H), 5.19 (d, *J* = 9.0 Hz, 1H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 9.0 Hz, 2H), 7.76 (d, *J* =8.1 Hz, 2H), 8.09 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.9, 21.5, 24.6, 29.8, 30.0, 53.8, 83.1, 94.4, 123.4, 126.9, 130.0, 130.1, 132.5, 138.4, 143.3, 146.8; HRMS-ESI neg (m/z) calcd for $[M-H]^-$ ($C_{21}H_{21}N_2O_4S$) 397.1222, found, 397.1226.

Methyl 4-((cis-2-(4-methylphenylsulfonamido)cyclohexyl)ethynyl)benzoate (**8hd**)

A solution of **7hc** (397.2 mg, 1.22 mmol), hydrazine monohydrate (296 µL, 6.1 mmol) and methanol (15 mL) was heated to reflux for 3 h, filtered and concentrated. The residue was dissolved in CH_2Cl_2 (15 mL), added with triethylamine (428 µL, 3.07 mmol) and stirred at rt for 30 min. The above solution was then added with *p*-toluenesulfonyl chloride (175.6 mg, 0.92 mmol) at 0 ºC, stirred at rt for 15 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with hydrochloric acid (1 *N*, 5 mL), sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:8; R_f 0.50) to give 4-methyl-*N*-(*cis*-2-((trimethylsilyl)ethynyl)cyclohexyl)-benzenesulfonamide (280.6 mg, 0.80 mmol, 66%) as a colorless solid. M.p. 125.0–126.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.14 (s, 9H), 1.31–1.75 (m, 8H), 2.39 (s, 3H), 2.59–2.63 (m, 1H), 3.12–3.23 (m, 1H), 4.76 (d, *J* = 15.5 Hz, 1H), 7.25–7.27 (m, 2H), 7.72–7.75 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 0.13, 20.6, 21.5, 24.7, 29.7, 30.1, 34.8, 53.4, 89.5, 105.1, 126.8, 126.9, 129.6, 129.7, 138.6, 143.2; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{18}H_{28}NO_2Si$) 350.1610, found, 350.1607. Tetrabutylammonium fluoride (1.0 *M* in THF, 410 µL, 0.41 mmol) was added to a solution of the above TMS-alkyne (119.3 mg, 0.34 mmol) and THF (3 mL) at 0 ºC. The reaction mixture was stirred at rt for 1 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:4; R_f 0.37) to give *N*-(*cis*-2-ethynylcyclohexyl)-4-methylbenzenesulfonamide (86.7 mg, 0.31 mmol, 92%) as a colorless solid. M.p. 99.0–101.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.12–1.19 (m, 1H), 1.34–1.44 (m, 2H), 1.49–1.57 (m, 2H), 1.61–1.67 (m, 2H),

1.72–1.77 (m, 1H), 2.09 (d, *J* = 2.4 Hz, 1H), 2.40 (s, 3H), 2.61–2.66 (m, 1H), 3.17–3.24 (m, 1H), 4.82 (d, *J* = 9.1 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.5, 21.5, 24.7, 29.6, 29.9, 33.8, 53.4, 72.7, 82.8, 126.8, 126.9, 129.6, 129.7, 138.5, 143.3; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{15}H_{20}NO_2S$) 278.1215, found, 278.1213. A solution of the above terminal alkyne (92.9 mg, 0.34 mmol), copper(I) iodide (3.2 mg, 17 μmol) and THF (5 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (11.8 mg, 17 μmol), methyl 4-iodobenzoate (131.7 mg, 0.50mmol) and triethylamine (0.1 mL, 0.67 mmol) sequentially, heated to reflux for 10 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:4; R_f 0.30) to give **8hd** (74.1 mg, 0.18 mmol, 55%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.19–1.21 (m, 1H), 1.37–1.43 (m, 1H), 1.47–1.53 (m, 3H), 1.59–1.71 (m, 2H), 1.83–1.86 (m, 1H), 2.39 (s, 1H), 2.93–2.96 (m, 1H), 3.26–3.34 (m, 1H), 3.91 (s, 1H), 4.80 (d, *J* = 9.2 Hz, 1H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.96 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 20.9, 21.5, 24.7, 30.0, 30.2, 34.7, 52.2, 53.8, 84.2, 91.5, 126.9, 127.7, 129.5, 129.7, 131.7, 138.5, 143.3, 166.5; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{23}H_{25}NO4NaS$) 434.1402, found, 434.1402.

***N*-(*cis*-2-((4-Chlorophenyl)ethynyl)cyclohexyl)-4-methylbenzenesulfonamide** (**8he**)

A solution of *N*-(*cis*-2-ethynylcyclohexyl)-4-methylbenzenesulfonamide (37.2 mg, 0.13 mmol, as prepared in **8hd**), copper(I) iodide (1.3 mg, 7 μmol) and THF (5 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (4.7 mg, 7 μmol), 1-chloro-4-iodobenzene (41.6 mg, 0.17 mmol) and triethylamine (38 μL, 0.27 mmol) sequentially, heated to reflux for 45 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.39) to give **8he** (33.5 mg, 0.086 mmol, 64%) as a colorless solid. M.p. 139.0–141.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.10–1.23 (m, 1H), 1.40–1.50 (m, 3H), 1.55–1.59 (m, 2H), 1.66–1.70 (m, 1H), 1.81–1.83 (m, 1H), 2.39 (s, 1H), 2.89–2.91 (m, 1H), 3.24–3.33 (m, 1H), 4.87 (d, *J* = 9.2 Hz, 1H), 7.24-7.30 (m, 6H), 7.76 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 20.9, 21.5, 24.7, 29.9, 30.2, 34.7, 53.8, 83.7, 89.2, 121.5, 126.8, 128.6, 129.7, 132.9, 134.1, 138.4, 143.3; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{21}NO_2SCl$) 386.0982, found, 386.0974.

4-Methyl-*N*-(*cis*-2-(*p*-tolylethynyl)cyclohexyl)benzenesulfonamide (**8hf**)

A solution of **11hf** (93.9 mg, 0.27 mmol), hydrazine monohydrate (70 μL, 1.37 mmol) and methanol (15 mL) was heated to reflux for 3 h, filtered and

concentrated. The residue was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (125 μL, 0.88 mmol). The above solution was added with *p*-toluenesulfonyl chloride (83.9 mg, 0.44 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.56) to give **8hf** (71.9 mg, 0.20 mmol, 72%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.20–1.24 (m, 1H), 1.40–1.47 (m, 2H), 1.51–1.56 (m, 1H), 1.59–1.64 (m, 2H), 1.66–1.69 (m, 1H), 1.81–1.84 (m, 1H), 2.33 (s, 3H), 2.38 (s, 3H), 2.85 (q, *J* = 3.9 Hz, 1H), 3.26–3.31 (m, 1H), 4.90 (d, *J* = 9.2 Hz, 1H), 7.09 (d, *J* = 8.1 Hz, 2H), 7.25–7.27 (m, 4H), 7.76 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125MHz) δ 20.8, 21.4, 21.5, 24.7, 29.9, 30.3, 34.6, 53.8, 84.9, 87.2, 119.9, 126.8, 128.9, 129.6, 131.4, 138.1, 138.5, 143.2; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{22}H_{25}NO_2NaS$) 390.1504, found, 390.1508.

***N*-(*cis*-2-((4-(*tert*-Butyl)phenyl)ethynyl)cyclohexyl)-4-methylbenzenesulfonamide (8hg)**

A solution of **11hg** (58.5 mg, 0.15 mmol), hydrazine monohydrate (37 μL, 0.76 mmol) and methanol (15 mL) was heated to reflux for 3 h, filtered and concentrated. The residue was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (84 μL, 0.60 mmol). The above solution was added with *p*-toluenesulfonyl chloride (57.2 mg, 0.30 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.44) to give **8hg** (43.9 mg, 0.11 mmol, 71%) as a colorless solid. M.p. 139.5-142.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.20–1.28 (m,1H), 1.30 (s, 9H), 1.41–1.48 (m, 2H), 1.59–1.64 (m, 2H), 1.66–1.70 (m, 1H), 1.80–1.84 (m, 1H), 2.40 (s, 3H), 2.85 (q, *J* = 4.1 Hz, 1H), 3.26–3.32 (m, 1H), 4.80 (d, *J* = 9.3 Hz, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.31 (s, 4H), 7.76 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 21.5, 24.7, 29.7, 30.0, 30.4, 31.2, 34.6, 53.9, 85.0, 87.3, 119.9, 125.3, 126.9, 129.7, 131.4, 138.6, 143.2, 151.5; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{25}H_{31}NO_2NaS$) 432.1973, found, 432.1980.

***N*-(*cis*-2-((4-Methoxyphenyl)ethynyl)cyclohexyl)-4-methylbenzenesulfonamide (8hh)**

A solution of **11hh** (75.0 mg, 0.21 mmol), hydrazine monohydrate (51 μL, 1.04 mmol) and methanol (15 mL) was heated to reflux for 3 h, filtered and

concentrated. The residue was dissolved in CH_2Cl_2 (15 mL) and added with triethylamine (87 μL, 0.62 mmol). The above solution was added with *p*-toluenesulfonyl chloride (47.6 mg, 0.25 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (10 mL) and extracted with CH_2Cl_2 (12 mL × 3). The combined organic layers were washed with sat. $NaHCO_{3(aq)}$ (10 mL) and sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.25) to give **8hh** (42.0 mg, 0.11 mmol, 53%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.19–1.26 (m,2H), 1.41–1.47 (m, 1H), 1.51–1.53 (m, 1H), 1.59–1.64 (m, 2H), 1.66–1.70 (m, 1H), 1.79–1.86 (m, 1H), 2.40 (s, 3H), 2.84 (q, *J* = 4.0 Hz, 1H), 3.25–3.30 (m, 1H), 3.80 (s, 3H), 4.82 (d, *J* = 12.2 Hz, 1H), 6.82 (d, *J* = 8.8 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.9, 24.7, 30.0, 30.4, 34.6, 53.9, 55.3, 84.8, 86.4, 113.9, 115.1, 126.9, 129.7, 133.1, 138.7, 140.0, 143.2; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{22}H_{25}NO_3SNa$) 406.1453, found, 406.1457.

***N*-(*cis*-2-((4-Nitrophenyl)ethynyl)cyclopentyl)-methanesulfonamide** (**9pc**)

A solution of **11pc** (72.5 mg, 0.20 mmol), hydrazine monohydrate (49 μL, 1.0 mmol) and methanol (2 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2 mL) and added with triethylamine (112 μL, 0.80 mmol). The above solution was added with methanesulfonyl chloride (31 μL, 0.40 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:1; R_f 0.52) to give **9pc** (37.3 mg, 0.12 mmol, 60%) as a light yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.58–1.79 (m,2H), 1.83–1.96 (m, 2H), 1.98–2.11 (m, 2H), 2.99 (s, 3H), 3.23 (dt, *J* = 4.1 Hz, *J* = 6.9 Hz, 1H), 3.83–3.94 (m, 1H), 4.87 (d, *J* = 8.7 Hz, 1H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 8.12 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 21.1, 30.7, 31.7, 36.2, 41.8, 57.0, 83.3, 94.3, 123.5, 129.8, 132.4, 146.9; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{14}H_{17}N_2O_4S$) 309.0909, found, 309.0906.

Methyl 4-((*cis*-2-(methylsulfonamido)cyclopentyl)ethynyl)-benzoate (**9pd**)

A solution of **11pd** (100.2 mg, 0.27 mmol), hydrazine monohydrate (66 μL, 1.35 mmol) and methanol (3 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (3 mL) and added with triethylamine (151 μL, 1.08 mmol). The above solution was added with methanesulfonyl chloride (42 μL, 0.54 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:2; R_f 0.45) to give **9pd** (36.1 mg, 0.11 mmol, 45%) as a viscous liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.55–1.79 (m, 2H), 1.86–1.96 (m, 2H), 1.98–2.08 (m, 2H), 2.97 (s, 3H), 3.19 (dt, *J* = 4.2 Hz, *J* = 6.9 Hz, 1H), 3.80–3.93 (m, 1H), 3.87 (s, 3H), 4.90 (d, *J* = 8.7 Hz, 1H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.92 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.1, 30.7, 31.7, 36.0, 41.6, 52.1, 56.9, 84.3, 91.6, 127.5, 129.4, 131.5, 166.4; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{16}H_{19}NO4SNa$) 344.0932, found, 344.0925.

***N*-(*cis*-2-((4-Chlorophenyl)ethynyl)cyclopentyl)-methanesulfonamide** (**9pe**)

A solution of **11pe** (70.0 mg, 0.20 mmol), hydrazine monohydrate (49 μL, 1.0 mmol) and methanol (2 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2 mL) and added with triethylamine (111 μL, 0.80 mmol). The above solution was added with methanesulfonyl chloride (31 μL, 0.40 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 2:3; R_f 0.57) to give **9pe** (39.9 mg, 0.13 mmol, 67%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.56–1.79 (m, 2H), 1.84–2.09 (m, 4H), 2.98 (s, 3H), 3.17 (dt, *J* = 4.1 Hz, *J* = 6.7 Hz, 1H), 3.80–3.90 (m, 1H), 4.80 (d, *J* = 8.6 Hz, 1H), 7.23–7.32 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.1, 30.7, 31.7, 35.9, 41.7, 56.9, 84.0, 89.2, 121.2, 128.6,

132.9, 134.2; HRMS-MALDI (m/z) calcd for $[M+Na]^+$ ($C_{21}H_{21}NO_2SCl$) 320.0482, found, 320.0485.

***N*-(*cis*-2-((4-(*tert*-Butyl)phenyl)ethynyl)cyclopentyl)methanesulfonamide (9pg)**

A solution of **11pg** (74.5 mg, 0.20 mmol), hydrazine monohydrate (49 μL, 1.0 mmol) and methanol (2 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (2 mL) and added with triethylamine (111 μL, 0.80 mmol). The above solution was added with methanesulfonyl chloride (31 μL, 0.40 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.47) to give **9pg** (25.3 mg, 0.08 mmol, 40%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (s, 9H), 1.60–1.65 (m,1H), 1.70–1.81(m, 1H), 1.87–1.97 (m, 2H), 1.98–2.09 (m, 2H), 3.00 (s, 3H), 3.15–3.22 (m, 1H), 3.80–3.90 (m, 1H), 4.76 (d, J = 8.4 Hz, 1H), 7.28–7.38 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.2, 31.0, 31.1, 31.9, 34.7, 35.8, 41.6, 57.0, 85.3, 87.4, 119.6, 125.4, 131.3, 151.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{18}H_{25}NO_2SNa$) 342.1504, found, 342.1505.

***N*-(*cis*-2-((4-Methoxyphenyl)ethynyl)cyclopentyl)-methanesulfonamide (9ph)**

A solution of **11ph** (120.9 mg, 0.35 mmol), hydrazine monohydrate (85 μL, 1.75 mmol) and methanol (3.5 mL) was heated to reflux for 12 h, added with conc. hydrochloric acid (2 mL) at 0 ºC, heated to reflux for another 1 h, filtered and concentrated. The residue was diluted with water (10 mL), washed with diethyl ether (10 mL × 2), basified with $NaOH_{(aq)}$ (15%, ~6 mL) and extracted with diethyl ether (20 mL × 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude amine was dissolved in CH_2Cl_2 (3.5 mL) and added with triethylamine (195 μL, 1.40 mmol). The above solution was added with methanesulfonyl chloride (54 μL, 0.70 mmol) at 0 ºC, stirred at rt for 12 h, quenched with water (2 mL) and extracted with CH_2Cl_2 (8 mL × 3). The combined organic layers were washed with sat. $NaCl_{(aq)}$ (10 mL), dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 2:3; R_f 0.48) to give **9ph** (79.3 mg, 0.27 mmol, 78%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz)

δ 1.56–1.64 (m,1H), 1.70–1.77 (m, 1H), 1.83–1.92 (m, 2H), 1.94–2.04 (m, 2H), 2.97 (s, 3H), 3.14 (dt, *J* = 4.2 Hz, *J* = 6.9 Hz, 1H), 3.76 (s, 3H), 3.79–3.85 (m, 1H), 4.87 (d, *J* = 8.5 Hz, 1H), 6.78–6.81 (m, 2H), 7.29–7.32 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.1, 30.9, 31.8, 35.8, 41.5, 55.2, 56.9, 84.9, 86.6, 113.9, 114.8, 133.0, 159.5; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{15}H_{20}NO_3S$) 294.1164, found, 294.1164.

2-(*cis*-2-Ethynylcyclopentyl)isoindoline-1,3-dione (**10p**)

Tetrabutylammonium fluoride (70% in H_2O, 169.9 mg, 0.65 mmol) was added to a solution of **7pc** (167.5 mg, 0.54 mmol) and THF (5 mL) at 0 ºC. The reaction mixture was stirred at rt for 10 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.68) to give **10p** (109.1 mg, 0.46 mmol, 85%) as a colorless solid. M.p. 68.0–69.5 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.48–1.58 (m, 1H), 1.86 (d, *J* = 2.6 Hz, 1H), 2.05–2.12 (m, 3H), 2.15–2.22 (m, 1H), 2.48–2.56 (m, 1H), 2.98–3.04 (m, 1H), 4.81 (dt, *J* = 9.0 Hz, *J*=7.3 Hz, 1H), 7.66–7.69 (m, 2H), 7.79–7.83 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 25.3, 28.1, 33.5, 35.8, 52.9, 71.4, 83.2, 123.1, 131.9, 133.8, 168.7. HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{15}H_{14}NO_2$) 240.1025, found, 240.1022.

2-(*cis*-2-Ethynylcyclohexyl)isoindoline-1,3-dione (**10h**)

Tetrabutylammonium fluoride (1.0 *M* in THF, 837 µL, 0.84 mmol) was added to a solution of **7hc** (227.0 mg, 0.70 mmol) and THF (10 mL) at 0 ºC. The reaction mixture was stirred at rt for 1 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:7; R_f 0.40) to give **10h** (154.4 mg, 0.61 mmol, 87%) as a light yellow solid. M.p. 140–142 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.25–1.42 (m, 1H), 1.63–1.66 (m, 1H), 1.69–1.82 (m, 2H), 1.85–2.03 (m, 3H), 2.06 (d, *J* = 2.5 Hz, 1H), 3.03–3.21 (m, 2H), 4.14 (dt, *J* = 13.1 Hz, *J* = 3.9 Hz, 1H), 7.71–7.73 (m, 1H), 7.83–7.86 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.9, 25.1, 26.3, 30.9, 33.0, 54.8, 71.6, 83.7, 123.1, 131.9, 133.8, 168.6; HRMS-EI (m/z) calcd for $[M]^+$ ($C_{16}H_{15}NO_2$) 253.1103, found, 253.1095.

2-(*cis*-2-((4-Nitrophenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pc**)

A solution of **10p** (175.2 mg, 0.73 mmol) and copper(I) iodide (7.0 mg, 36.5 µmol) in THF (7 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (25.6 mg, 36.5 µmol), 1-iodo-4-nitrobenzene (373.5 mg, 1.5 mmol), triethylamine (209 µL, 1.5 mmol) sequentially, heated to reflux for 12 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:4; R_f 0.50) to give **11pc** (239.2 mg, 0.66 mmol, 91%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ

1.60–1.65 (m, 1H), 2.09–2.30 (m, 4H), 2.59–2.71 (m, 1H), 3.26 (q, *J* = 8.5 Hz, 1H), 4.91 (q, *J* = 8.8 Hz, 1H), 7.00 (d, *J* = 8.5 Hz, 2H), 7.67–7.72 (m, 2H), 7.78–7.82 (m, 2H), 7.91 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.4, 27.9, 33.4, 36.8, 53.3, 82.1, 95.2, 123.0, 123.1, 127.1, 130.1, 131.8, 134.0, 146.4, 168.6; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{17}N_2O_4$) 361.1188, found, 361.1181.

Methyl 4-((*cis*-2-(1,3-dioxoisoindolin-2-yl)cyclopentyl)ethynyl)benzoate (**11pd**)

A solution of **10p** (136.4 mg, 0.57 mmol) and copper(I) iodide (5.0 mg, 28.5 μmol) in THF (6 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (20.4 mg, 28.5 μmol), methyl 4-iodobenzoate (298.7 mg, 1.14 mmol), triethylamine (159 μL, 1.14 mmol) sequentially, heated to reflux for 12 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.47) to give **11pd** (174.3 mg, 0.47 mmol, 82%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.49–1.66 (m, 1H), 2.06–2.27 (m, 4H), 2.56–2.69 (m, 1H), 3.22 (dd, *J* = 16.5 Hz, *J* = 9.6 Hz, 1H), 3.80 (s, 3H), 4.89 (dd, *J* = 16.5 Hz, *J* = 8.5 Hz, 1H), 6.88 (d, *J* = 8.5 Hz, 2H), 7.63–7.69 (m, 4H), 7.75–7.81 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.4, 27.9, 33.5, 36.8, 52.0, 53.4, 83.1, 92.4, 123.0, 127.9, 128.7, 129.0, 131.0, 131.8, 133.9, 166.4, 168.7; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{23}H_{20}NO_4$) 374.1392, found, 374.1393.

2-(*cis*-2-((4-Chlorophenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pe**)

A solution of **10p** (167.3 mg, 0. 70 mmol) and copper(I) iodide (6.7 mg, 35 μmol) in THF (7 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (24.6 mg, 35 μmol), 1-chloro-4-iodobenzene (333.8 mg, 1.40 mmol), triethylamine (195 μL, 1.40 mmol) sequentially, heated to reflux for 16 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.50) to give **11pe** (221.2 mg, 0.68 mmol, 97%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.53–1.62 (m, 1H), 2.07–2.27 (m, 4H), 2.56–2.69 (m, 1H), 3.16–3.24 (m, 1H), 4.88 (dd, *J* =16.4 Hz, *J* = 8.8 Hz, 1H), 6.76–6.80 (m, 2H), 6.98–7.02 (m, 2H), 7.63–7.70 (m, 2H), 7.76–7.84 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.4, 27.9, 33.6, 36.7, 53.5, 82.6, 90.1, 121.7, 123.0, 128.2, 131.9, 132.3, 133.4, 133.9, 168.8; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{18}H_{22}NO_2Si$) 350.0948, found, 350.0955.

2-(*cis*-2-((4-(*tert*-Butyl)phenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pg**)

A solution of **10p** (153.4 mg, 0.64 mmol) and copper(I) iodide (5.7 mg, 32 μmol) in THF (6 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (21.1 mg, 32 μmol), 1-bromo-4-*tert*-butylbenzene (225 μL, 1.30 mmol), triethylamine (181 μL, 1.30 mmol) sequentially, heated to reflux for 12 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.58) to give **11pg** (98.5 mg, 0.26 mmol, 41%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.19 (s, 9H), 1.50–1.65 (m, 1H), 2.07–2.28 (m, 4H), 2.57–2.69 (m, 1H), 3.17–3.26 (m, 1H), 4.88 (dt, *J* = 7.2 Hz, *J* = 8.8 Hz, 1H), 6.78–6.83 (m, 2H), 7.03–7.07 (m, 2H), 7.62–7.69 (m, 2H), 7.77–7.83 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.3, 27.9, 31.0, 33.7, 34.5, 36.9, 53.5, 83.7, 88.1, 120.2, 123.0, 124.8, 130.8, 132.0, 133.7, 150.5, 168.7; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{25}H_{25}NO_2Na$) 394.1783, found, 394.1779.

2-(*cis*-2-((4-Methoxyphenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11ph**)

A solution of **10p** (95.7 mg, 0.40 mmol) and copper(I) iodide (3.8 mg, 20 μmol) in THF (4 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (14.0 mg, 20 μmol), 4-iodoanisole (187 mmol, 0.80 mmol), triethylamine (112 μL, 0.80 mmol) sequentially, heated to reflux for 12 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.50) to give **11ph** (105.3 mg, 0.30 mmol, 76%) as a colorless solid. M.p. 95.0–97.0 ºC; ^{1}H NMR ($CDCl_3$, 300MHz) δ 1.52–1.63 (m, 1H), 2.02–2.26 (m, 4H), 2.56–2.68 (m, 1H), 3.15–3.24 (m, 1H), 3.67 (s, 3H), 4.87 (dt, *J* = 7.3 Hz, *J* = 8.9 Hz, 1H), 6.52–6.57 (m, 2H), 6.77–6.81 (m, 2H), 7.61–7.68 (m, 2H), 7.76–7.82 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.4, 27.9, 33.7, 36.9, 53.5, 55.1, 83.4, 87.3, 113.4, 115.4, 123.0, 132.0, 132.5, 133.7, 158.8, 168.8; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{22}H_{20}NO_3$) 346.1443, found, 346.1438.

2-(*cis*-2-((3-Chlorophenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pi**)

A solution of **10p** (92.9 mg, 0.39 mmol) and copper(I) iodide (3.8 mg, 20 μmol) in triethylaime (4 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (14.0 mg, 20 μmol), 3-chloro-1-iodobenzene (72 μL, 0.58 mmol), stirred at rt for 30 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.47) to give **11pi** (131.1 mg, 0.37 mmol, 96%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.53–1.62 (m, 1H), 2.09–2.15 (m, 3H), 2.15–2.23 (m, 1H), 2.60–2.67 (m, 1H), 3.20 (dt, *J* = 7.5 Hz, *J* = 9.8 Hz, 1H), 4.89 (dt, *J* = 7.5 Hz, *J* = 8.9 Hz, 1H), 6.70 (t, *J* = 1.7 Hz, 1H), 6.78 (dt, *J* = 7.8 Hz, *J* = 1.3 Hz, 1H), 6.95 (t, *J* = 7.8 Hz, 1H), 7.03–7.05 (m, 1H), 7.64–7.68 (m, 2H), 7.78–7.82 (m, 2H); ^{13}C

NMR ($CDCl_3$, 125 MHz) δ 25.4, 27.9, 33.5, 36.8, 53.5, 82.4, 90.5, 123.0, 124.9, 127.7, 129.0, 129.2, 131.0, 131.9, 133.6, 133.9, 168.7; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{17}NO_2Cl$) 350.0948, found, 350.0941.

2-(*cis*-2-((2-Chlorophenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pj**)

A solution of **10p** (100.4 mg, 0.42 mmol) and copper(I) iodide (3.8 mg, 20 μmol) in triethylaime (4 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (14.0 mg, 20 μmol), 2-chloro-1-iodobenzene (77 μL, 0.63 mmol), stirred at rt for 30 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.59) to give **11pj** (119.8 mg, 0.34 mmol, 82%) as a white solid. M.p. 108.0–109.5 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.54–1.64 (m, 1H), 2.11–2.19 (m, 3H), 2.25–2.33 (m, 1H), 2.53–2.60 (m, 1H), 3.26–3.32 (m, 1H), 4.90 (dt, *J* = 7.1 Hz, *J* = 9.0 Hz, 1H), 6.94 (t, *J* = 7.6 Hz, 1H), 6.99–7.03 (m, 2H), 7.11 (d, *J* = 8.1 Hz, 1H), 7.62–7.64 (m, 2H), 7.76–7.78 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 25.4, 28.3, 33.7, 37.0, 53.1, 80.4, 94.3, 123.0, 126.0, 127.6, 128.5, 128.8, 132.1, 133.2, 133.7, 135.3, 168.7; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{17}NO_2Cl$) 350.0948, found, 350.0956.

2-(*cis*-2-((2-Fluorophenyl)ethynyl)cyclopentyl)isoindoline-1,3-dione (**11pk**)

A solution of **10p** (110.0 mg, 0.46 mmol) and copper(I) iodide (3.8 mg, 20 μmol) in triethylaime (5 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (14.0 mg, 20 μmol), 2-fluoro-1-iodobenzene (81 μL, 0.69 mmol), stirred at rt for 30 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.56) to give **11pk** (133.5 mg, 0.40 mmol, 87%) as a light yellow liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.53–1.63 (m, 1H), 2.09–2.17 (m, 3H), 2.21–2.30 (m, 1H), 2.55–2.63 (m, 1H), 3.23–3.28 (m, 1H), 4.90 (dt, *J* = 7.2 Hz, *J* = 8.9 Hz, 1H), 6.75–6.82 (m, 2H), 6.87 (td, *J* = 7.6 Hz, *J*=1.8 Hz. 1H), 7.03–7.07 (m, 1H), 7.61–7.65 (m, 2H), 7.76–7.80 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 25.4, 28.1, 33.6, 36.9, 53.2, 76.9, 94.2, 111.7 (d, $J_{C\text{-}F}$ = 15.6 Hz), 115.0 (d, $J_{C\text{-}F}$ = 20.9 Hz), 122.9, 123.4, 129.0 (d, $J_{C\text{-}F}$ = 7.6 Hz), 132.0, 133.1, 133.6, 162.4 (d, $J_{C\text{-}F}$ = 249.8 Hz), 168.7; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{17}NO_2F$) 334.1243, found, 334.1247.

2-(*cis*-2-((4-Nitrophenyl)ethynyl)cyclohexyl)isoindoline-1,3-dione (**11hc**)

A solution of **10h** (77.2 mg, 0.30 mmol) and copper(I) iodide (3.0 mg, 15 μmol) in THF (5 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (10.7 mg, 15 μmol), 1-iodo-4-nitrobenzene (113.8 mg, 0.46 mmol), triethylamine (84 μL, 0.60 mmol) sequentially, heated to reflux for 12 h and concentrated. The crude product was purified by column

chromatography (SiO_2, ethyl acetate/hexanes, 1:7; R_f 0.36) to give **11hc** (101.7 mg, 0.27 mmol, 89%) as a colorless solid. M.p. 167.0–169.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.30–1.47 (m, 1H), 1.65–1.80 (m, 2H), 1.81–1.90 (m, 1H), 1.95–2.02 (m, 2H), 3.16 (dq, *J* = 13.2 Hz, *J* = 3.6 Hz, 1H), 3.32 (d, *J* = 3.3Hz, 1H), 4.23 (dt, *J* = 13.2 Hz, *J*=3.6 Hz, 1H), 7.54 (d, *J* = 8.9 Hz, 2H), 7.68–7.70 (m, 2H), 7.80–7.83 (m, 2H), 8.12 (d, *J* = 8.9 Hz. 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 21.2, 25.3, 26.2, 30.8, 34.2, 54.8, 82.3, 95.8, 123.1, 123.4, 130.8, 131.8, 132.3, 133.9, 146.7, 168.6; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{22}H_{19}N_2O_4$) 375.1345, found, 375.1351.

2-(*cis*-2-(*p*-Tolylethynyl)cyclohexyl)isoindoline-1,3-dione (**11hf**)

A solution of **10h** (53.2 mg, 0.21 mmol) and copper(I) iodide (2.1 mg, 11 μmol) in THF (5 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (7.1 mg, 11 μmol), 4-bromotoluene (51 μL, 0.42 mmol), triethylamine (56 μL, 0.40 mmol) sequentially, heated to reflux for 16 h and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:7; R_f 0.39) to give **11hf** (23.9 mg, 0.07 mmol, 33%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.29–1.38 (m, 1H), 1.57–1.60 (m, 1H), 1.68–1.80 (m, 2H), 1.84–1.88 (m, 1H), 1.94–2.00 (m, 2H), 2.29 (s, 3H), 3.20 (qd, *J* = 13.1 Hz, *J* = 3.6 Hz, 1H), 3.31 (d, *J* = 3.1 Hz, 1H), 4.20 (dt, *J* = 13.1 Hz, *J* = 3.6 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz. 2H), 7.63–7.65 (m, 2H), 7.77–7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.1, 21.4, 25.2, 31.0, 33.9, 55.2, 83.7, 88.6, 120.7, 123.0, 128.8, 131.4, 131.9, 133.7, 137.5, 168.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{23}H_{21}NO_2Na$) 366.1470, found, 366.1472.

2-(*cis*-2-((4-(*tert*-Butyl)phenyl)ethynyl)cyclohexyl)isoindoline-1,3-dione (**11hg**)

A solution of **10h** (113.4 mg, 0.45 mmol), copper(I) iodide (4.2 mg, 23 μmol), triethylamine (1 mL) and THF (2 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (15.7 mg, 23 μmol) and 1-bromo-4-*tert*-butylbenzene (155 μL, 0.89 mmol) sequentially, heated in the microwave reactor (80 ºC) for 20 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.46) to give **11hg** (58.8 mg, 0.15 mmol, 34%) as a colorless solid. M.p. 149.0–151.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.27 (s, 9H), 1.31–1.35 (m, 1H), 1.57–1.62 (m, 1H), 1.71–1.79 (m, 2H), 1.84–1.87 (m, 1H), 1.94–2.00 (m, 2H), 3.19 (qd, *J* = 13.1 Hz, *J* = 3.7 Hz, 1H), 3.30 (d, *J* = 3.0 Hz, 1H), 4.19 (dt, *J* = 13.1 Hz, *J*=3.7 Hz, 1H), 7.27 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz. 2H), 7.65–7.67 (m, 2H), 7.79–7.81 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.1, 25.2, 26.3, 31.1,

31.2, 33.9, 34.6, 55.3, 83.7, 88.6, 102.8, 123.0, 125.1, 131.2, 132.0, 131.9, 133.7, 150.7, 168.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{26}H_{27}NO_2Na$) 408.1939, found, 408.1930.

2-(*cis*-2-((4-Methoxyphenyl)ethynyl)cyclohexyl)isoindoline-1,3-dione (**11hh**)

A solution of **10h** (40.5 mg, 0.16 mmol), copper(I) iodide (1.5 mg, 8 μmol), triethylamine (1 mL) and THF (3 mL) was stirred at rt for 10 min. The reaction mixture was added with $PdCl_2(PPh_3)_2$ (5.6 mg, 8 μmol) and 4-iodoanisole (56.0 mg, 0.24 mmol) sequentially, heated in the microwave reactor (80 ºC) for 20 min and concentrated. The crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:6; R_f 0.16) to give **11hh** (25.8 mg, 0.072 mmol, 45%) as a colorless solid. M.p. 110.0–112.0 ºC; ^{1}H NMR ($CDCl_3$, 300MHz) δ 1.26–1.37 (m, 1H), 1.56–1.75 (m, 2H), 1.81–1.85 (m, 2H), 1.93–2.02 (m, 2H), 3.14 (qd, *J* = 13.1 Hz, *J* = 3.5 Hz, 1H), 3.28 (d, *J* = 2.8 Hz, 1H), 3.76 (s, 1H), 4.19 (dt, *J* = 13.1 Hz, *J* = 3.9 Hz, 1H), 6.77 (d, *J* = 8.8 Hz, 2H), 7.32 (d, *J* = 8.8 Hz. 2H), 7.64–7.68 (m, 2H), 7.78–7.81 (m, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 21.2, 25.2, 26.4, 31.1, 33.9, 55.2, 55.3, 83.4, 87.8, 113.7, 116.0, 123.0, 132.0, 132.9, 133.7, 133.8, 159.1, 168.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{23}H_{21}NO_3Na$) 382.1419, found, 382.1426.

***cis*-2-Butyl-1-tosyl-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole** (**12pb**)

A suspension of **8pa** (30.5 mg, 0.12 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.59) to give **12pb** (16.2 mg, 0.051 mmol, 53%) as a viscous liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.89 (t, *J* = 7.4 Hz, 3H), 1.28–1.37 (m, 2H), 1.41–1.51 (m, 4H), 1.52–1.62 (m, 2H), 1.80–1.87 (m, 1H), 1.96–2.01 (m, 1H), 2.26 (quintet, *J* = 7.8 Hz, 1H), 2.39 (s, 3H), 2.51–2.58 (m, 1H), 2.90–2.94 (m, 1H), 4.24–4.28 (m, 1H), 4.65 (s, 1H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.9, 21.5, 22.3, 23.1, 28.9, 30.0, 32.5, 36.5, 44.5, 67.4, 115.1, 127.3, 129.5, 135.2, 143.2, 144.0; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{18}H_{26}NO_2S$) 320.1684, found 320.1692.

***cis*-2-Butyl-1-(methylsulfonyl)-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole** (**13pb**)

A suspension of **9pb** (28.8 mg, 0.12 mmol), $PdCl_2$ (1.1 mg, 5.8 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.66) to give **13pb** (10.6 mg, 0.044 mmol, 37%) as a

viscous liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.89 (t, *J* = 7.4 Hz, 3H), 1.27–1.36 (m, 2H), 1.44–1.59 (m, 5H), 1.62–1.68 (m, 1H), 1.79–1.86 (m, 1H), 1.93–1.96 (m, 1H), 2.17–2.23 (m, 1H), 2.34–2.41 (m, 1H), 2.80 (s, 3H), 3.31–3.35 (m, 1H), 4.34–4.38 (m, 1H), 4.79 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.9, 22.3, 23.0, 28.6, 29.7, 32.8, 35.6, 36.8, 44.7, 67.9, 114.1, 143.8; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{12}H_{21}NO_2SNa$) 266.1191, found 266.1195.

***cis*-2-(4-Nitrophenyl)-1-tosyl-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole (12pc)**

A suspension of **8pc** (28.0 mg, 0.07 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.50) to give **12pc** (24.3 mg, 0.063 mmol, 87%) as a yellow solid. M.p. 168.0–170.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.50–1.52 (m, 3H), 1.61–1.66 (m, 1H), 2.01–2.02 (m, 2H), 2.41 (s, 3H), 2.90 (t, *J* = 7.6 Hz, 1H), 4.44–4.78 (m, 1H), 5.28 (s, 1H), 7.27 (d, *J* = 7.7 Hz, 2H), 7.46 (d, *J* = 7.7 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 8.18 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.6, 23.2, 31.5, 35.8, 45.6, 68.0, 123.2, 125.2, 127.9, 128.3, 129.5, 133.0, 139.9, 142.3, 144.0, 147.5; HRMS-ESI neg (m/z) calcd for $[M-H]^-$ ($C_{20}H_{19}N_2O_4S$) 383.1066, found 383.1060.

***cis*-1-(Methylsulfonyl)-2-(4-nitrophenyl)-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole (13pc)**

A suspension of **9pc** (35.9 mg, 0.12 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:1; R_f 0.56) to give **13pc** (30.2 mg, 0.10 mmol, 84%) as a yellow liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.56–1.72 (m, 3H), 1.80–1.88 (m, 1H), 1.97–2.05 (m, 2H), 2.78 (s, 3H), 3.60 (tt, *J* = 8.2 Hz, *J* = 2.4 Hz, 1H), 4.56–4.62 (m, 1H), 5.48 (d, *J* = 2.2 Hz, 1H), 7.60–7.63 (m, 2H), 8.11–8.15 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.1, 31.9, 35.3, 36.1, 46.0, 68.3, 123.1, 124.0, 128.3, 139.2, 142.2, 147.5; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{14}H_{17}N_2O_4S$) 309.0909, found 309.0903.

Methyl 4-(cis-1-(methylsulfonyl)-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrol-2-yl)benzoate (13pd)

A suspension of **9pd** (35.0 mg, 0.11 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and

maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:2; R_f 0.50) to give **13pd** (26.7 mg, 0.085 mmol, 76%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.52–1.70 (m, 3H), 1.75–1.87 (m, 1H), 1.97–2.03 (m, 2H), 2.76 (s, 3H), 3.56 (tt, *J* = 8.3 Hz, *J* = 2.3 Hz, 1H), 3.88 (s, 3H), 4.60 (dt, *J* = 8.3 Hz, *J* = 5.4 Hz, 1H), 5.36 (d, *J* = 2.1 Hz, 1H), 7.52 (d, *J* = 8.3 Hz, 2H), 7.96 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.1, 32.1, 35.5, 36.2, 45.7, 52.1, 68.3, 122.0, 127.6, 129.1, 130.0, 137.1, 143.3, 166.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{16}H_{19}NO_4SNa$) 344.0932, found 344.0926.

***cis*-2-(4-Chlorophenyl)-1-(methylsulfonyl)-1,3a,4,5,6,6a-hexahydrocyclopenta [b]pyrrole** (**13pe**)

A suspension of **9pe** (30.5 mg, 0.10 mmol), $PdCl_2$ (1.2 mg, 6.7 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:2; R_f 0.59) to give **13pe** (21.4 mg, 0.070 mmol, 70%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.51–1.63 (m, 2H), 1.65–1.69(m, 1H), 1.76–1.83 (m, 1H), 1.95–2.02 (m, 2H), 2.75 (s, 3H), 3.53 (tt, *J* = 8.2 Hz, *J* = 2.4 Hz, 1H), 4.58 (dt, *J* = 8.2 Hz, *J* = 5.4 Hz, 1H), 5.25 (d, *J* = 2.2 Hz, 1H), 7.26 (dt, *J* = 8.6 Hz, *J* = 2.1 Hz, 2H), 7.39 (dt, *J* = 8.6 Hz, *J* = 2.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.1, 32.1, 35.4, 36.3, 45.5, 68.3, 120.6, 128.0, 129.0, 131.0, 134.4, 143.0; HRMS-MALDI (m/z) calcd for $[M+H]^+$ ($C_{14}H_{17}ClNO_2S$) 298.0663, found 298.0653.

***cis*-2-(4-(*tert*-Butyl)phenyl)-1-(methylsulfonyl)-1,3a,4,5,6,6a-hexahydrocyclop enta[b]pyrrole** (**13pg**)

A suspension of **9pg** (23.4 mg, 0.07 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:3; R_f 0.63) to give **13pg** (18.2 mg, 0.062 mmol, 78%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29 (s, 9H), 1.54–1.60 (m, 2H), 1.63–1.67 (m, 1H), 1.75–1.81 (m, 1H), 1.95–2.02 (m, 2H), 2.76 (s, 3H), 3.52 (tt, *J* = 8.2 Hz, *J* = 2.2 Hz, 1H), 4.58–4.64 (m, 1H), 5.20 (d, *J* =2.2 Hz, 1H), 7.30–7.34 (m, 2H), 7.38–7.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 75MHz) δ 23.1, 31.2, 32.2, 34.6, 35.7, 36.3, 45.4, 68.3, 119.7, 124.8, 127.4, 129.7, 144.1, 151.7; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{18}H_{26}NO_2S$) 320.1684, found 320.1680.

***cis*-2-(4-Methoxyphenyl)-1-(methylsulfonyl)-1,3a,4,5,6,6a-hexahydrocyclope nta[b]pyrrole** (**13ph**)

A suspension of **9ph** (35.3 mg, 0.12 mmol), $PdCl_2$ (1.1 mg, 6.2 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 2:3; R_f 0.52) to give **13ph** (24.5 mg, 0.083 mmol, 69%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.57–1.68 (m, 3H), 1.71–1.83 (m, 1H), 1.94–2.03 (m, 2H), 2.74 (s, 3H), 3.50 (tt, *J* = 8.3 Hz, *J* = 2.2 Hz, 1H), 3.78 (s, 3H), 4.56–4.63 (m, 1H), 5.13 (d, *J* = 2.0 Hz, 1H), 6.81–6.85 (m, 2H), 7.37–7.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 23.1, 32.3, 35.8, 36.4, 45.4, 55.2, 68.3, 113.3, 118.5, 124.9, 129.2, 130.6, 143.8; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{15}H_{20}NO_3S$) 294.1164, found, 294.1172.

***cis*-2-(3-Chlorophenyl)-1-tosyl-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole (12pi)**

A suspension of **8pi** (29.0 mg, 0.081 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.62) to give **12pi** (26.7 mg, 0.071 mmol, 92%) as a colorless solid. M.p. 114.0–116.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.49–1.54 (m, 3H), 1.58–1.65 (m, 1H), 1.98–2.01 (m, 2H), 2.41 (s, 3H), 2.87 (t, *J* = 8.0 Hz, 1H), 4.45–4.49 (m, 1H), 5.10 (s, 1H), 7.24–7.28 (m, 4H), 7.39–7.40 (m, 1H), 7.43 (s, 1H), 7.49 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.5, 23.2, 31.7, 45.3, 67.8, 122.6, 126.0, 127.6, 128.0, 128.4, 129.0, 129.3, 133.5, 133.6, 135.2, 142.8, 143.7; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{20}H_{21}NO_2SCl$) 374.0982, found 374.0989.

***cis*-2-(2-Chlorophenyl)-1-tosyl-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole (12pj)**

A suspension of **8pj** (40.1 mg, 0.11 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.58) to give **12pj** (33.1 mg, 0.089 mmol, 83%) as a colorless solid. M.p. 167.0–168.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.52–1.59 (m, 2H), 1.62–1.68 (m, 2H), 1.91–1.98 (m, 1H), 2.14–2.17 (m, 1H), 2.39 (s, 3H), 3.10–3.14 (m, 1H), 4.48 (dt, *J* = 2.4 Hz, *J* = 8.1 Hz, 1H), 5.13 (d, *J* = 1.3 Hz, 1H), 7.21–7.24 (m, 4H), 7.31–7.35 (m, 1H), 7.37–7.39 (m, 1H), 7.53 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.5, 23.3, 32.0, 36.1, 45.6, 67.0, 122.3,

125.9, 128.0, 129.3, 129.7, 131.2, 132.0, 133.1, 134.5, 140.1, 143.4; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{20}H_{21}NO_2SCl$) 374.0928, found 374.0984.

***cis*-2-(2-fluorophenyl)-1-tosyl-1,3a,4,5,6,6a-hexahydrocyclopenta[b]pyrrole (12pk)**

A suspension of **8pk** (36.7 mg, 0.11 mmol), $PdCl_2$ (1.0 mg, 5.6 μmol) and acetonitrile (1 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2, ethyl acetate/hexanes, 1:5; R_f 0.53) to give **12pk** (27.0 mg, 0.076 mmol, 74%) as a colorless solid. M.p. 101.0–103.5 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.49–1.66 (m, 4H), 1.91–2.10 (m, 2H), 2.40 (s, 3H), 2.95–3.00 (m, 1H), 4.42 (dt, J = 3.5 Hz, J = 7.8 Hz, 1H), 5.19 (s, 1H), 7.01–7.14 (m, 2H), 7.24–7.31 (m, 3H), 7.45–7.53 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.5, 23.2, 31.8, 36.0, 45.6, 67.0, 115.5 (d, $J_{C\text{-}F}$ =21.7 Hz), 121.3 (d, $J_{C\text{-}F}$ = 13.5 Hz), 123.3 (d, $J_{C\text{-}F}$ = 3.0 Hz) , 128.0, 129.3, 129.8 (d, $J_{C\text{-}F}$ = 7.9 Hz), 130.8, 133.8, 137.3, 143.5, 159.9 (d, $J_{C\text{-}F}$ = 248.1 Hz); HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{20}H_{21}NO_2FS$) 358.1277, found, 358.1274.

***cis*-2-Butyl-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indole (12hb)**

A suspension of **8hb** (37.6 mg, 0.11 mmol), AuCl (1.3 mg, 5.6 μmol) and acetonitrile (3 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 1:8; R_f 0.33) to give **12hb** (17.6 mg, 0.05 mmol, 47%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 300MHz) δ 0.91 (t, J = 7.2 Hz, 3H), 1.09–1.27 (m, 3H), 1.31–1.41 (m, 4H), 1.47–1.66 (m, 5H), 1.87–1.96 (m, 1H), 2.31–2.36 (m, 1H), 2.39 (s, 3H), 2.49–2.58 (m, 1H), 4.01 (dt, J = 9.8 Hz, J = 7.4 Hz, 1H), 4.84 (s, 1H), 7.25 (d, J = 8.1 Hz, 2H), 7.66 (d, J = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.9, 20.9, 21.6, 21.7, 22.4, 26.0, 28.7, 29.4, 29.5, 39.1, 62.9, 118.6, 126.9, 129.5, 136.7, 138.5, 143.1; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{19}H_{28}NO_2S$) 334.1841, found, 334.1851.

***cis*-2-(4-Nitrophenyl)-1-tosyl-3a,4,5,6,7,7a-hexahydro-1H-indole (12hc)**

A suspension of **8hc** (45.2 mg, 0.11 mmol), AuCl (1.3 mg, 5.6 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 1:4; R_f 0.34) to give **12hc** (23.0 mg, 0.053 mmol, 51%) as a colorless solid. M.p. 158.0–160.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.10–1.16 (m, 2H), 1.34–1.41 (m, 1H), 1.45–1.46 (m, 1H), 1.54–1.69 (m, 3H), 2.07–2.10 (m, 1H), 2.39–2.44 (m, 1H), 2.42 (s, 3H), 4.25 (dt, J = 10.3 Hz, J = 6.9

Hz, 1H), 5.59 (d, *J* = 2.0 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.57 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.7 Hz, 2H), 8.20 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.3, 21.6, 22.0, 25.8, 28.8, 40.6, 63.9, 123.3, 127.4, 127.7, 127.8, 129.7, 135.5, 139.9, 141.8, 144.0, 147.6; HRMS-EI (m/z) calcd for $[M]^+$ ($C_{21}H_{22}N_2O_4S$) 398.1300, found, 398.1292.

Methyl 4-(*cis*-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indol-2-yl)benzoate (**12hd**)

A suspension of **8hd** (58.2 mg, 0.14 mmol), AuCl (1.6 mg, 6.8 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 1:4; R_f 0.40) to give **12hd** (29.0 mg, 0.067 mmol, 50%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.81–0.87 (m, 1H), 1.10–1.15 (m, 1H), 1.32–1.36 (m, 1H), 1.40–1.44 (m, 1H), 1.54–1.67 (m, 3H), 2.06–2.11 (m, 1H), 2.36–2.39 (m, 1H), 2.41 (s, 3H), 3.91 (s, 3H), 4.24 (dt, *J* = 10.2 Hz, *J* = 6.9 Hz, 1H), 5.49 (d, *J* = 1.9 Hz, 1H), 7.24–7.26 (m, 3H), 7.56–7.62 (m, 4H), 8.01 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 21.6, 22.1, 26.0, 28.8, 40.4, 52.1, 63.8, 125.8, 127.1, 127.4, 129.3, 129.5, 134.0, 134.1, 135.7, 138.0, 142.7, 143.7, 166.8; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{23}H_{25}NO_4SNa$) 434.1402, found 434.1401.

***cis*-2-(4-Chlorophenyl)-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indole** (**12he**)

A suspension of **8he** (27.0 mg, 0.069 mmol), AuCl (1.0 mg, 4.3 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 1:8; R_f 0.37) to give **12he** (15.3 mg, 0.039 mmol, 57%) as a white solid. M.p. 153.0–156.0 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.08–1.16 (m, 2H), 1.32–1.35 (m, 1H), 1.40–1.45 (m, 1H), 1.53–1.66 (m, 2H), 2.02–2.09 (m, 1H), 2.32–2.36 (m, 1H), 2.41 (s, 3H), 4.22 (dt, *J* = 10.1 Hz, *J* = 6.8 Hz, 1H), 5.36 (d, *J* = 1.9 Hz, 1H), 7.23–7.26 (m, 3H), 7.31 (d, *J* = 8.6 Hz, 2H), 7.46–7.51 (m, 2H), 7.56–7.58 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.2, 21.6, 22.2, 26.0, 28.8, 40.2, 63.7, 124.0, 127.4, 128.2, 128.5, 129.5, 132.1, 134.4, 135.8, 142.4, 143.6; HRMS-APCI (m/z) calcd for $[M+H]^+$ ($C_{21}H_{23}NO_2SCl$) 388.1138, found 388.1130.

***cis*-2-(*p*-Tolyl)-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indole** (**12hf**)

A suspension of **8hf** (35.0 mg, 0.10 mmol), AuCl (1.2 mg, 5.6 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified

with NEt_3, ethyl acetate/hexanes, 1:7; R_f 0.53) to give **12hf** (22.4 mg, 0.061 mmol, 64%) as a viscous liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.10–1.16 (m, 2H), 1.29–1.40 (m, 2H), 1.60–1.64 (m, 3H), 2.04–2.07 (m, 1H), 2.31–2.34 (m, 1H), 2.36 (s, 3H), 2.40 (s, 3H), 4.21 (dt, *J* = 10.1 Hz, *J* = 6.8 Hz, 1H), 5.30 (d, *J* = 1.7 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 8.1 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 21.3, 21.6, 22.3, 26.1, 28.8, 40.0, 63.6, 122.6, 127.1, 127.4, 128.6, 129.3, 130.8, 136.0, 138.6, 143.4; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{22}H_{26}NO_2SNa$) 368.1684, found, 368.1691.

***cis*-2-(4-(*tert*-Butyl)phenyl)-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indole (12hg)**

A suspension of **8hg** (51.4 mg, 0.13 mmol), AuCl (1.5 mg, 6.4 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 2:9; R_f 0.58) to give **12hg** (28.2 mg, 0.069 mmol, 55%) as a white solid. M.p. 159.0–161.0 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.07–1.13 (m, 2H), 1.33 (s, 9H), 1.36–1.47 (m, 2H), 1.54–1.64 (m, 3H), 2.04–2.08 (m, 1H), 2.31–2.34 (m, 1H), 2.41 (s, 3H), 4.22 (dt, *J* = 10.1 Hz, *J* = 6.8 Hz, 1H), 5.33 (d, *J* = 1.6 Hz, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 21.6, 22.3, 26.1, 28.8, 31.3, 34.7, 40.0, 63.7, 123.0, 124.8, 126.9, 127.5, 129.3, 130.7, 136.0, 143.4, 151.6; HRMS-ESI (m/z) calcd for $[M+Na]^+$ ($C_{25}H_{31}NO_2SNa$) 432.1987, found 432.1965.

***cis*-2-(4-Methoxyphenyl)-1-tosyl-3a,4,5,6,7,7a-hexahydro-*1H*-indole (12hh)**

A suspension of **8hh** (11.6 mg, 0.13 mmol), AuCl (0.6 mg, 2.6 μmol) and acetonitrile (2 mL) was heated to 85 ºC by the microwave reactor and maintained at 85 ºC for 20 min. After cooled to rt, the solvent was remove, and the crude product was purified by column chromatography (SiO_2 basified with NEt_3, ethyl acetate/hexanes, 1:6; R_f 0.46) to give **12hh** (4.8 mg, 0.013 mmol, 42%) as a colorless liquid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.10–1.19 (m, 2H), 1.32–1.45 (m, 2H), 1.60–1.64 (m, 3H), 2.02–2.11 (m, 1H), 2.29–2.35 (m, 1H), 2.40 (s, 3H), 3.82 (s, 3H), 4.21 (dt, *J* = 9.9 Hz, *J* = 6.7 Hz, 1H), 5.23 (s, 1H), 6.88 (d, *J* = 8.4 Hz, 2H), 7.22–7.27 (m, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.2, 21.6, 22.3, 26.1, 28.8, 40.0, 55.3, 63.6, 113.4, 121.5, 126.2, 127.5, 128.6, 129.4, 136.1, 143.1, 143.4, 160.0; HRMS-ESI (m/z) calcd for $[M+H]^+$ ($C_{22}H_{25}NO_3SNa$) 406.1453, found 406.1453.

References

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3 A. D. Jones, D. W. Knight, D. E. Hibbs, *J. Chem. Soc., Perkin Trans. 1* 2001, 1182.
4 I. D. Jurberg, Y. Odabachian, F. Gagosz, *J. Am. Chem. Soc*. 2010, **132**, 3543.

X-ray crystallography for compound **14h**

Summary of Data CCDC 1522351

Compound Name:

Formula: C21 H25 N1 O2 S1

Unit Cell Parameters: a 11.8441(4) b 11.9103(5) c 13.4579(5) P212121

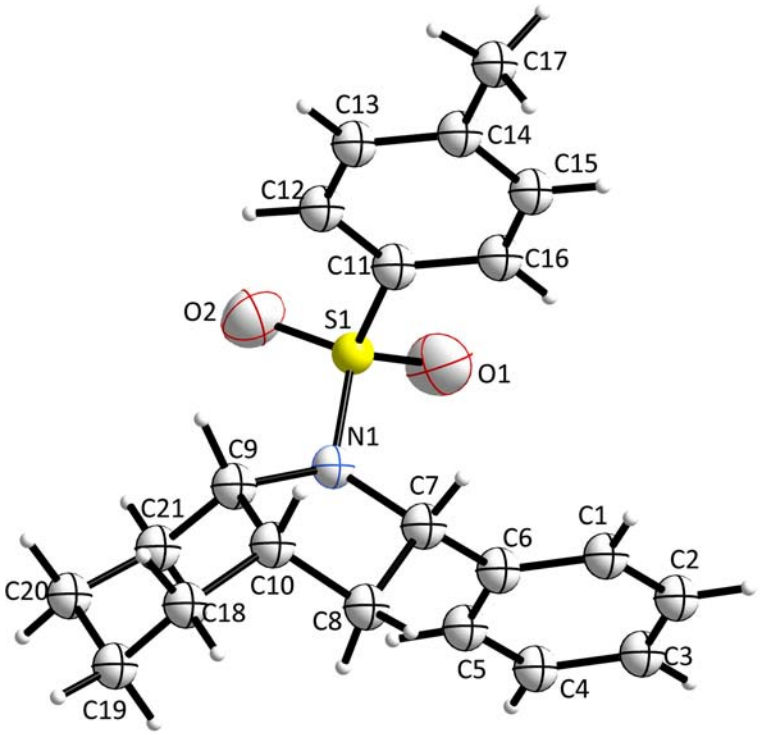


Table 1. Crystal data and structure refinement for 16AU09_0m.

Identification code	16au09_0m	
Empirical formula	C21 H25 N O2 S	
Formula weight	355.48	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 11.8441(4) Å	α= 90°.
	b = 11.9103(5) Å	β= 90°.
	c = 13.4579(5) Å	γ = 90°.

Volume	1898.46(12) $Å^3$
Z	4
Density (calculated)	1.244 Mg/m^3
Absorption coefficient	0.184 mm^{-1}
F(000)	760
Crystal size	0.30 x 0.15 x 0.05 mm^3
Theta range for data collection	2.28 to 28.41°.
Index ranges	-15<=h<=14, -15<=k<=12, -17<=l<=17
Reflections collected	25711
Independent reflections	4681 [R(int) = 0.0288]
Completeness to theta = 28.41°	98.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9909 and 0.9468
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4681 / 0 / 227
Goodness-of-fit on F^2	1.034
Final R indices [I>2sigma(I)]	R1 = 0.0433, wR2 = 0.0855
R indices (all data)	R1 = 0.0757, wR2 = 0.0971
Absolute structure parameter	0.50(7)
Largest diff. peak and hole	0.199 and -0.294 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for 16AU09_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	9815(1)	13879(1)	11051(1)	54(1)
N(1)	10186(1)	13530(1)	12165(1)	47(1)
O(1)	8684(1)	14286(1)	11098(1)	70(1)
O(2)	10694(1)	14577(1)	10665(1)	70(1)
C(1)	7345(2)	12840(2)	13017(2)	68(1)
C(2)	6361(2)	13346(3)	13352(2)	80(1)
C(3)	6397(2)	14366(2)	13793(2)	76(1)
C(4)	7410(2)	14903(2)	13905(2)	75(1)
C(5)	8393(2)	14402(2)	13581(2)	64(1)
C(6)	8374(2)	13369(2)	13126(1)	50(1)
C(7)	9431(2)	12794(2)	12777(1)	50(1)
C(8)	10237(2)	12402(2)	13594(2)	59(1)
C(9)	11396(1)	13309(2)	12391(1)	50(1)
C(10)	11349(2)	12254(2)	13039(2)	58(1)
C(11)	9781(2)	12643(2)	10337(1)	52(1)
C(12)	10732(2)	12286(2)	9836(2)	69(1)
C(13)	10706(2)	11296(2)	9308(2)	75(1)
C(14)	9739(2)	10655(2)	9251(2)	63(1)
C(15)	8791(2)	11032(2)	9748(2)	68(1)
C(16)	8809(2)	12007(2)	10289(2)	63(1)
C(17)	9724(2)	9570(2)	8686(2)	94(1)
C(18)	12417(2)	12062(3)	13647(2)	83(1)
C(19)	12856(2)	13095(3)	14142(2)	91(1)
C(21)	11906(2)	14316(2)	12901(2)	66(1)
C(20)	13027(2)	14034(3)	13398(2)	87(1)

Table 3. Bond lengths [Å] and angles [°] for 16AU09_0m.

S(1)-O(1)	1.4256(13)
S(1)-O(2)	1.4297(15)
S(1)-N(1)	1.6172(15)
S(1)-C(11)	1.758(2)
N(1)-C(9)	1.489(2)
N(1)-C(7)	1.499(2)
C(1)-C(6)	1.379(3)
C(1)-C(2)	1.388(3)
C(2)-C(3)	1.353(4)
C(3)-C(4)	1.368(3)
C(4)-C(5)	1.379(3)
C(5)-C(6)	1.374(3)
C(6)-C(7)	1.502(3)
C(7)-C(8)	1.528(3)
C(8)-C(10)	1.524(3)
C(9)-C(21)	1.508(3)
C(9)-C(10)	1.530(3)
C(10)-C(18)	1.524(3)
C(11)-C(12)	1.379(3)
C(11)-C(16)	1.380(3)
C(12)-C(13)	1.377(3)
C(13)-C(14)	1.378(3)
C(14)-C(15)	1.382(3)
C(14)-C(17)	1.500(3)
C(15)-C(16)	1.370(3)
C(18)-C(19)	1.492(4)
C(19)-C(20)	1.515(4)
C(21)-C(20)	1.524(3)

O(1)-S(1)-O(2)	120.17(10)
O(1)-S(1)-N(1)	107.56(9)
O(2)-S(1)-N(1)	106.74(9)
O(1)-S(1)-C(11)	106.72(9)
O(2)-S(1)-C(11)	107.76(9)
N(1)-S(1)-C(11)	107.30(8)
C(9)-N(1)-C(7)	111.07(14)

C(9)-N(1)-S(1)	119.76(12)
C(7)-N(1)-S(1)	119.84(12)
C(6)-C(1)-C(2)	120.6(2)
C(3)-C(2)-C(1)	120.4(2)
C(2)-C(3)-C(4)	119.7(2)
C(3)-C(4)-C(5)	120.2(2)
C(6)-C(5)-C(4)	120.9(2)
C(5)-C(6)-C(1)	118.1(2)
C(5)-C(6)-C(7)	122.24(18)
C(1)-C(6)-C(7)	119.6(2)
N(1)-C(7)-C(6)	113.75(16)
N(1)-C(7)-C(8)	101.58(15)
C(6)-C(7)-C(8)	115.79(16)
C(10)-C(8)-C(7)	102.88(16)
N(1)-C(9)-C(21)	109.71(16)
N(1)-C(9)-C(10)	103.04(14)
C(21)-C(9)-C(10)	114.13(16)
C(8)-C(10)-C(18)	118.12(19)
C(8)-C(10)-C(9)	102.48(16)
C(18)-C(10)-C(9)	113.49(19)
C(12)-C(11)-C(16)	119.3(2)
C(12)-C(11)-S(1)	120.43(16)
C(16)-C(11)-S(1)	120.29(15)
C(13)-C(12)-C(11)	119.9(2)
C(12)-C(13)-C(14)	121.4(2)
C(13)-C(14)-C(15)	117.9(2)
C(13)-C(14)-C(17)	121.1(2)
C(15)-C(14)-C(17)	121.0(2)
C(16)-C(15)-C(14)	121.3(2)
C(15)-C(16)-C(11)	120.20(19)
C(19)-C(18)-C(10)	113.9(2)
C(18)-C(19)-C(20)	111.1(2)
C(9)-C(21)-C(20)	111.9(2)
C(19)-C(20)-C(21)	109.7(2)

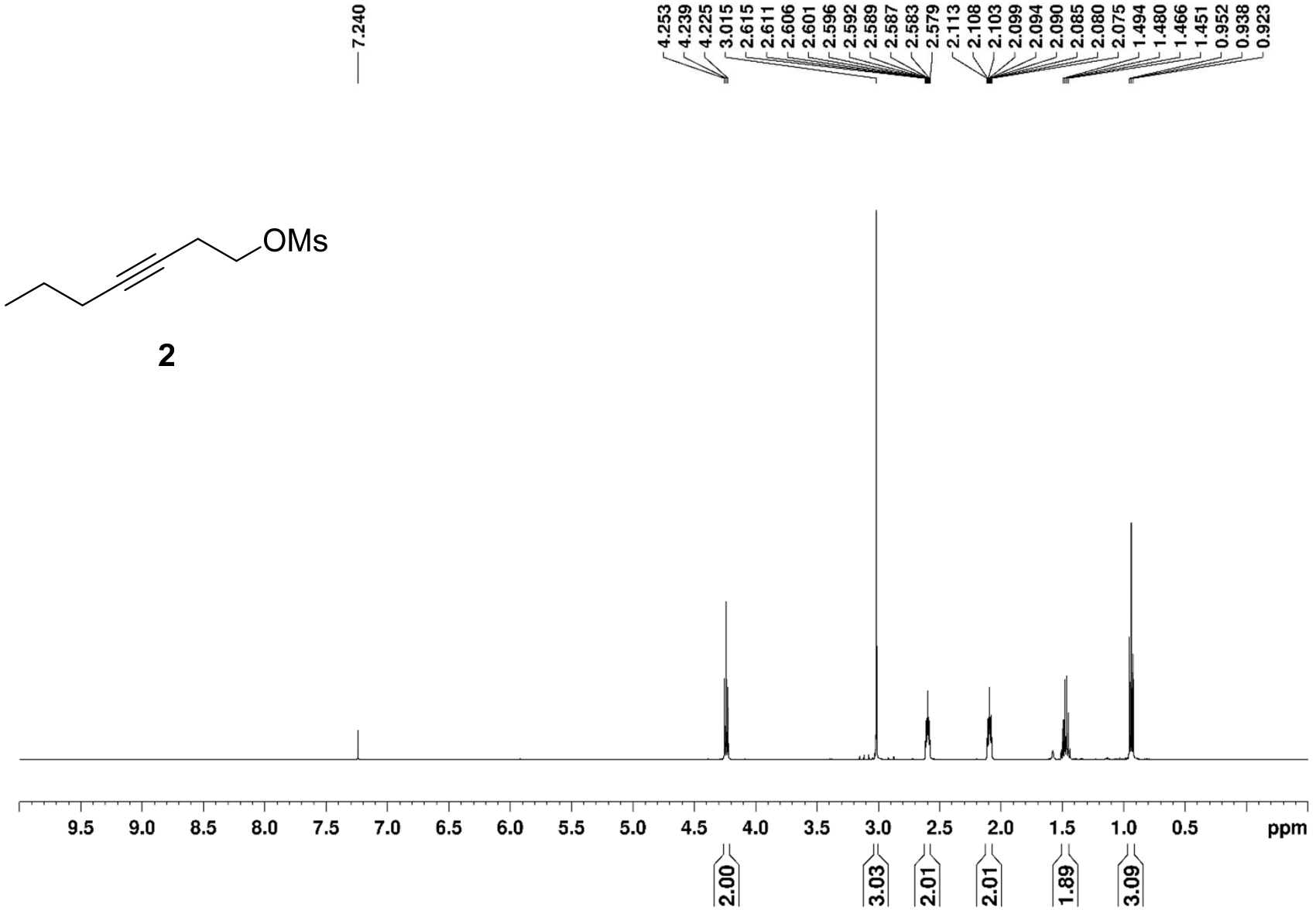
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 16AU09_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

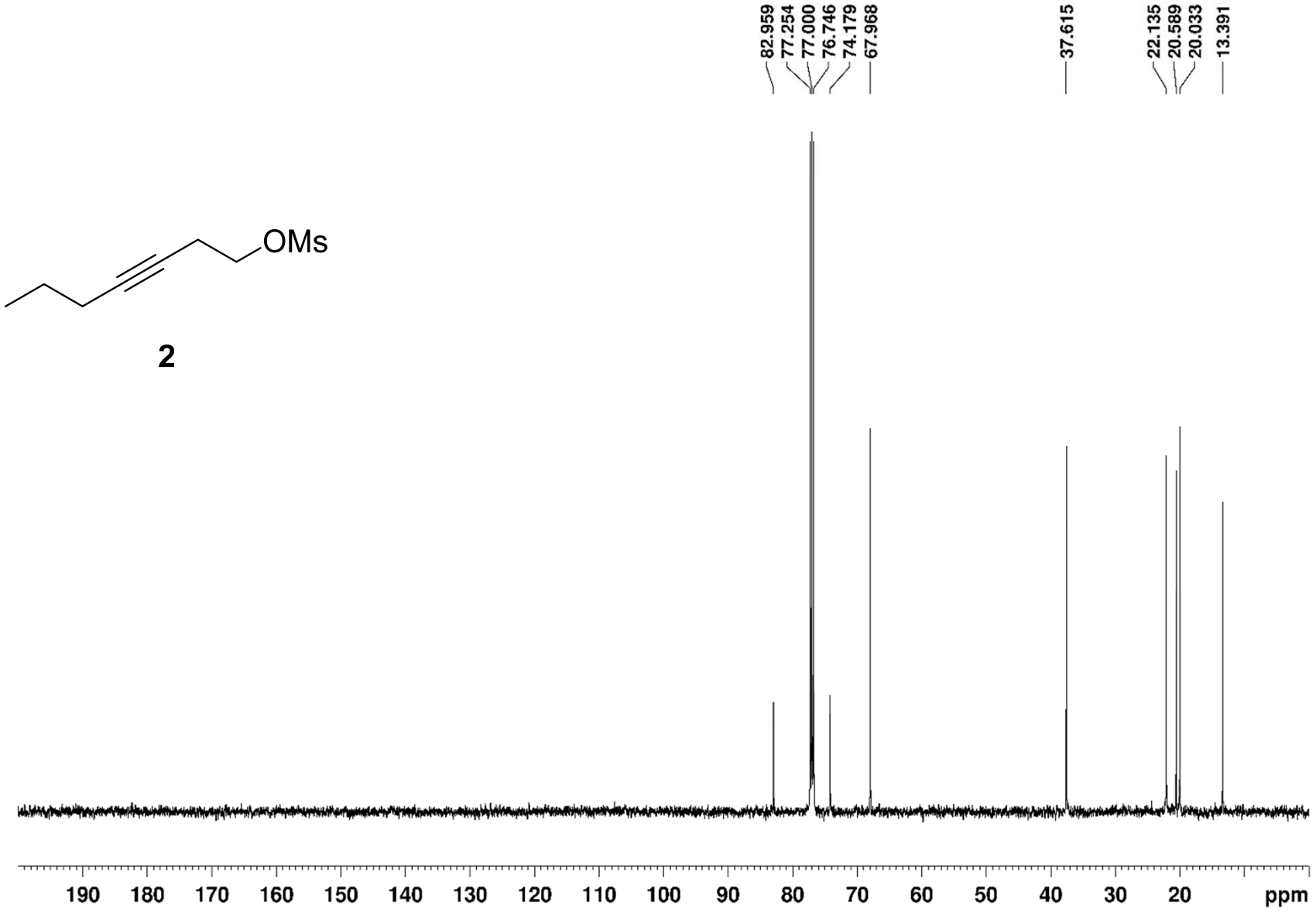
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	50(1)	64(1)	48(1)	11(1)	-1(1)	7(1)
N(1)	42(1)	55(1)	44(1)	6(1)	2(1)	3(1)
O(1)	60(1)	86(1)	64(1)	10(1)	-5(1)	26(1)
O(2)	75(1)	74(1)	60(1)	21(1)	3(1)	-10(1)
C(1)	56(1)	68(1)	78(1)	1(1)	9(1)	-6(1)
C(2)	49(1)	102(2)	89(2)	4(2)	9(1)	-8(1)
C(3)	58(1)	100(2)	69(2)	-4(2)	14(1)	16(1)
C(4)	70(1)	84(2)	71(1)	-17(1)	13(1)	10(1)
C(5)	59(1)	72(2)	63(1)	-8(1)	6(1)	-4(1)
C(6)	46(1)	60(1)	44(1)	7(1)	4(1)	0(1)
C(7)	51(1)	49(1)	49(1)	2(1)	3(1)	-3(1)
C(8)	61(1)	58(1)	58(1)	16(1)	5(1)	5(1)
C(9)	41(1)	63(1)	44(1)	4(1)	1(1)	3(1)
C(10)	54(1)	59(1)	60(1)	7(1)	-3(1)	10(1)
C(11)	37(1)	77(1)	40(1)	10(1)	-3(1)	5(1)
C(12)	40(1)	100(2)	67(1)	-11(1)	9(1)	-8(1)
C(13)	50(1)	108(2)	69(1)	-15(2)	10(1)	15(1)
C(14)	65(1)	71(1)	53(1)	3(1)	-12(1)	9(1)
C(15)	48(1)	88(2)	68(1)	2(1)	-8(1)	-6(1)
C(16)	36(1)	92(2)	60(1)	-7(1)	-1(1)	3(1)
C(17)	105(2)	85(2)	93(2)	-10(2)	-12(2)	22(2)
C(18)	66(1)	102(2)	82(2)	28(2)	-7(1)	22(2)
C(19)	65(1)	142(3)	67(2)	16(2)	-19(1)	5(2)
C(21)	61(1)	70(2)	69(1)	5(1)	-6(1)	-10(1)
C(20)	62(1)	117(2)	83(2)	3(2)	-19(1)	-16(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 16AU09_0m.

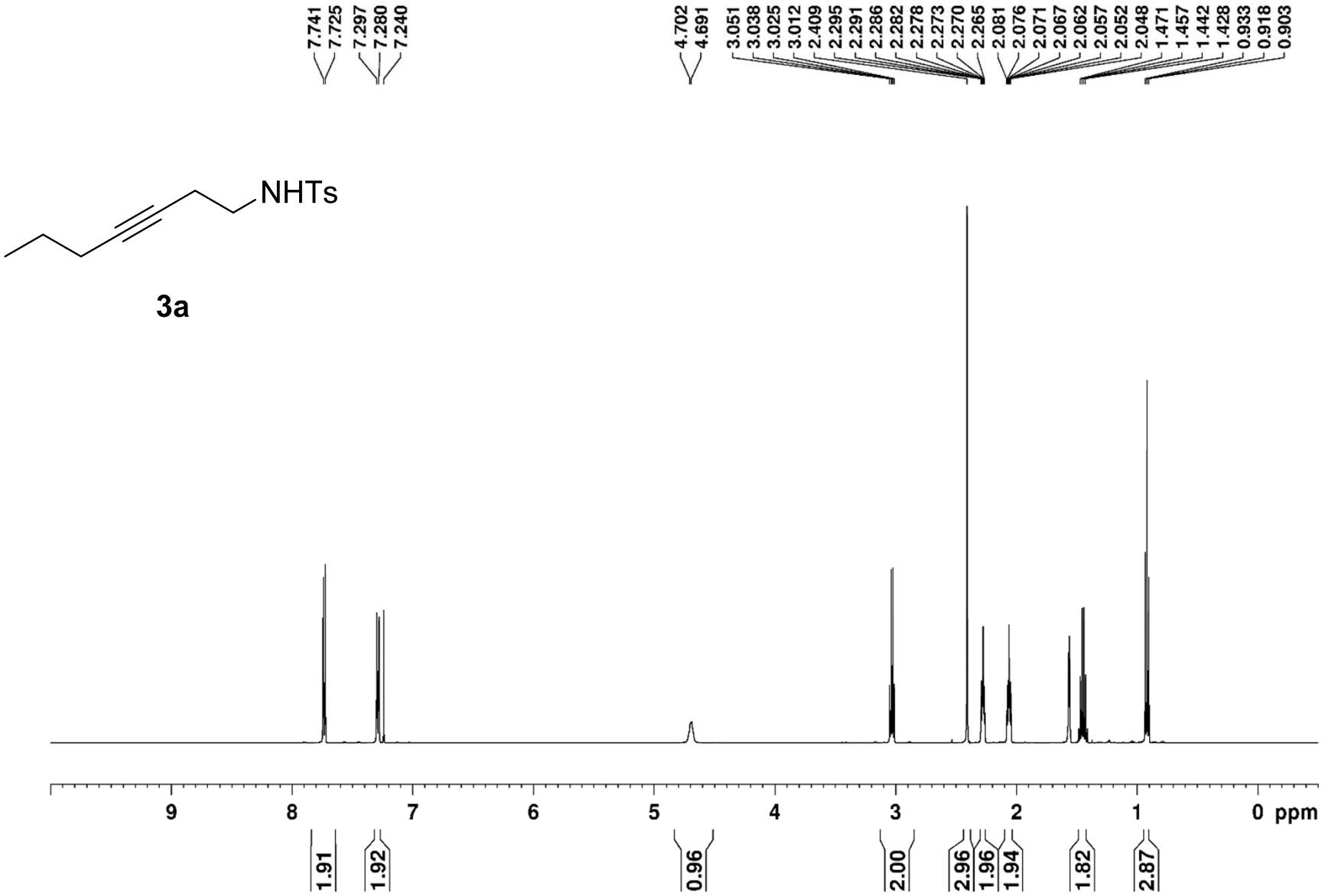
	x	y	z	U(eq)
H(1)	7311	12138	12717	81
H(2)	5672	12982	13272	96
H(3)	5736	14701	14019	91
H(4)	7436	15608	14201	90
H(5)	9079	14768	13672	77
H(7)	9215	12142	12376	60
H(8A)	10305	12961	14114	70
H(8B)	9986	11699	13884	70
H(9A)	11808	13149	11775	59
H(10A)	11261	11609	12594	69
H(12)	11388	12714	9855	83
H(13)	11355	11055	8984	91
H(15)	8128	10616	9715	81
H(16)	8164	12240	10625	75
H(17A)	10449	9448	8386	142
H(17B)	9156	9604	8178	142
H(17C)	9557	8964	9133	142
H(18A)	12262	11501	14152	100
H(18B)	12999	11765	13214	100
H(19A)	13568	12927	14465	109
H(19B)	12326	13336	14649	109
H(21A)	11382	14594	13397	80
H(21B)	12024	14907	12416	80
H(20A)	13571	13804	12900	105
H(20B)	13320	14693	13733	105



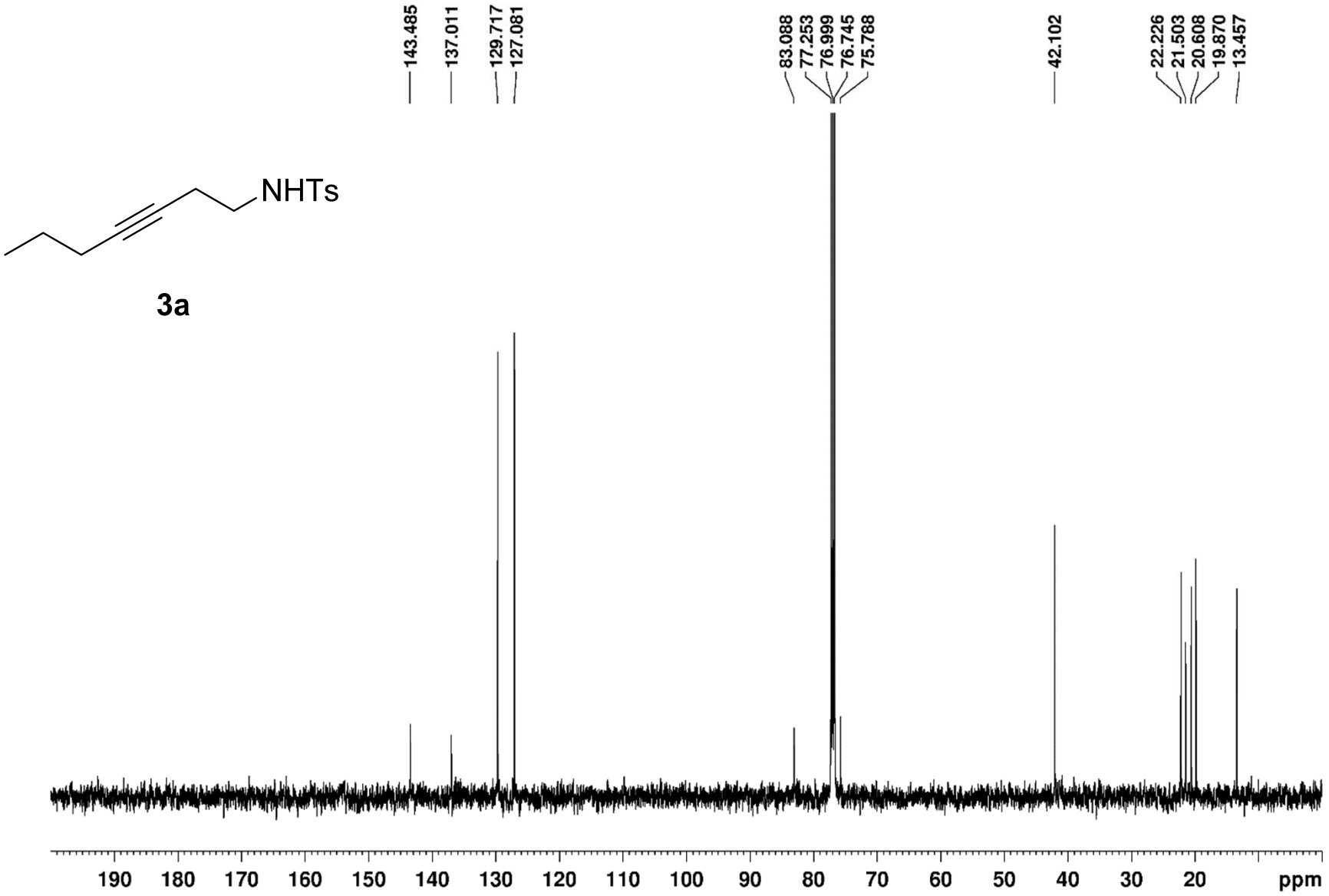
^{1}H NMR of compound **2** (500 MHz, $CDCl_3$)



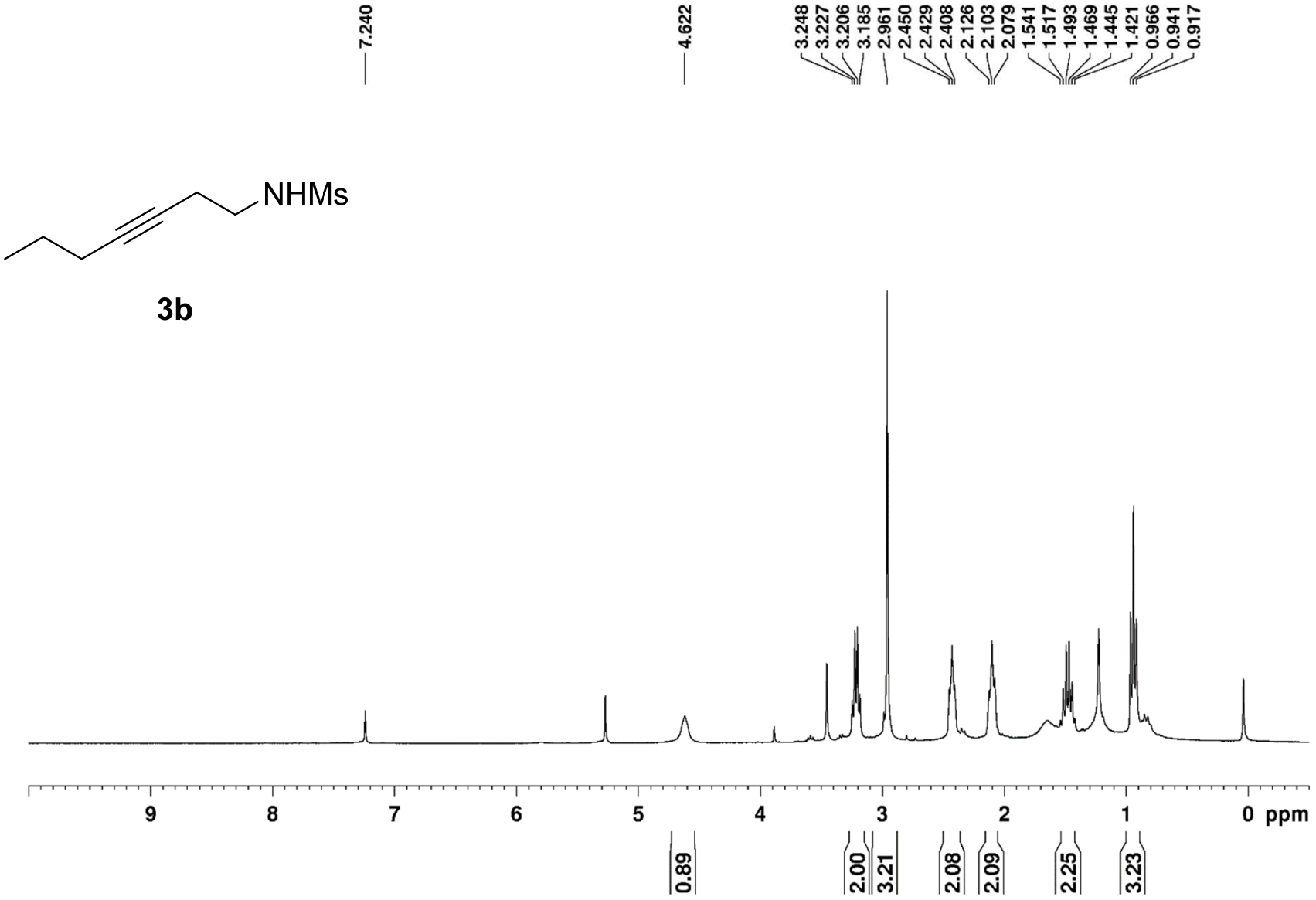
^{13}C NMR of compound **2** (125 MHz, $CDCl_3$)



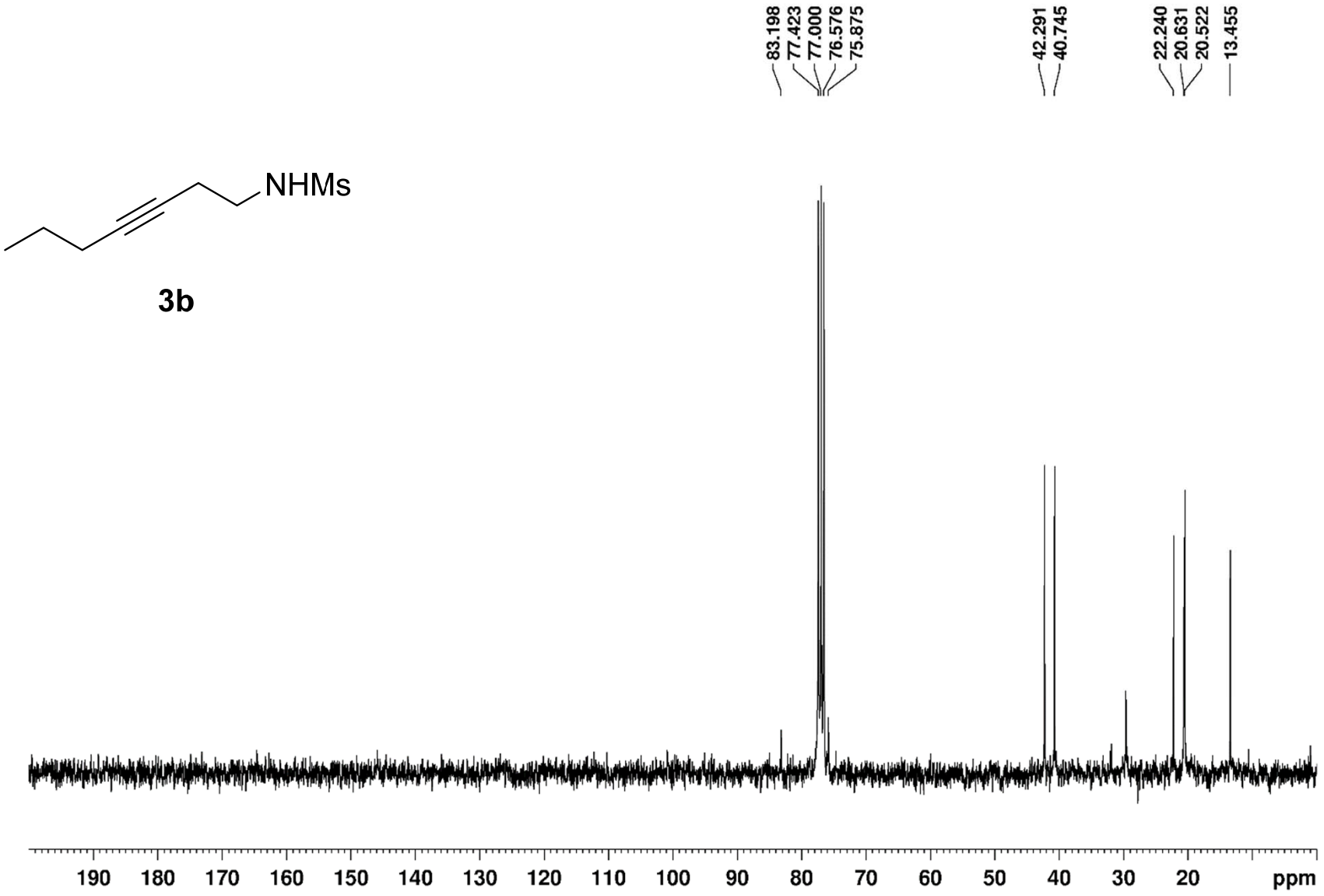
^{1}H NMR of compound **6pa** (500 MHz, $CDCl_3$)



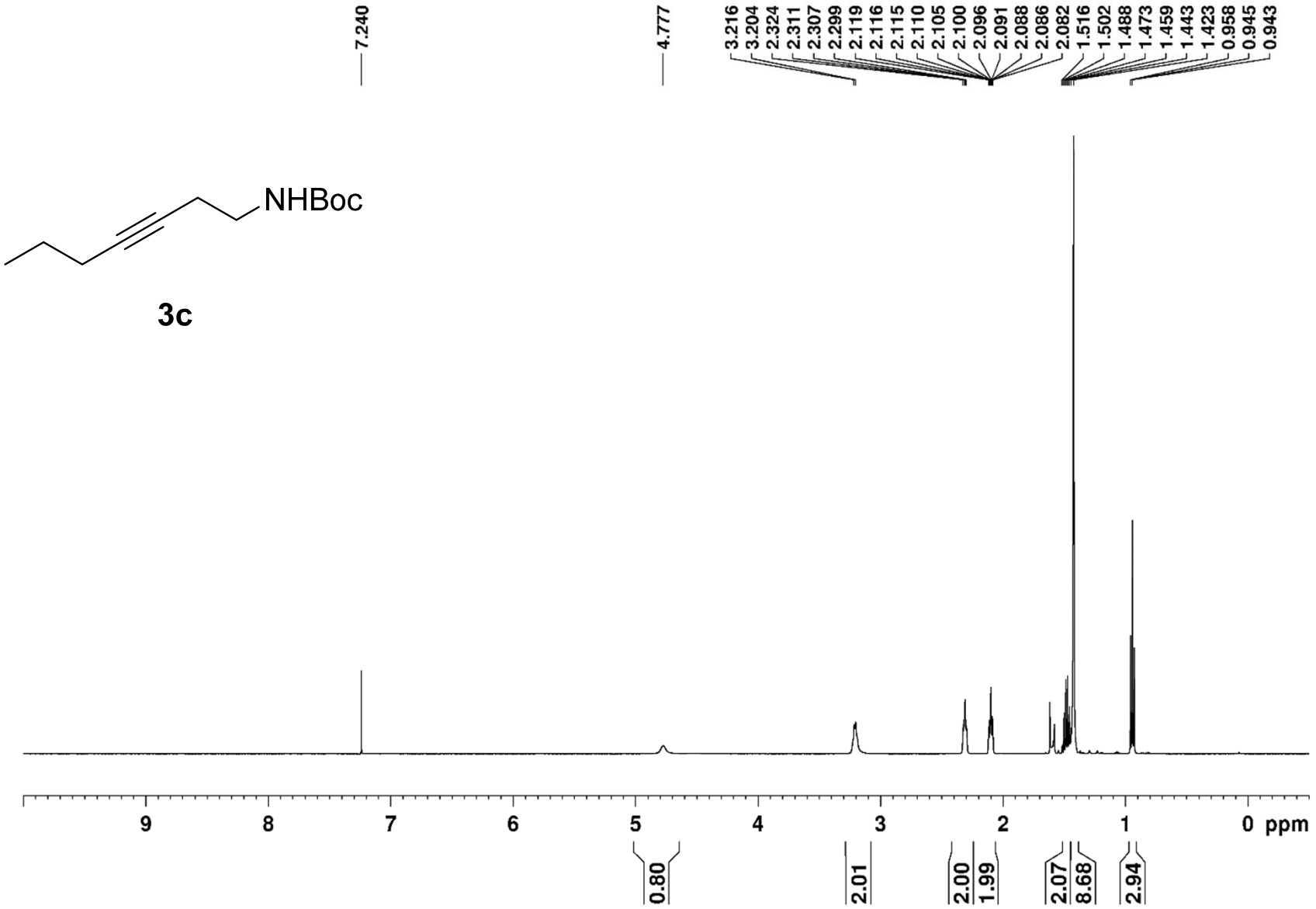
^{13}C NMR of compound **3a** (125 MHz, $CDCl_3$)



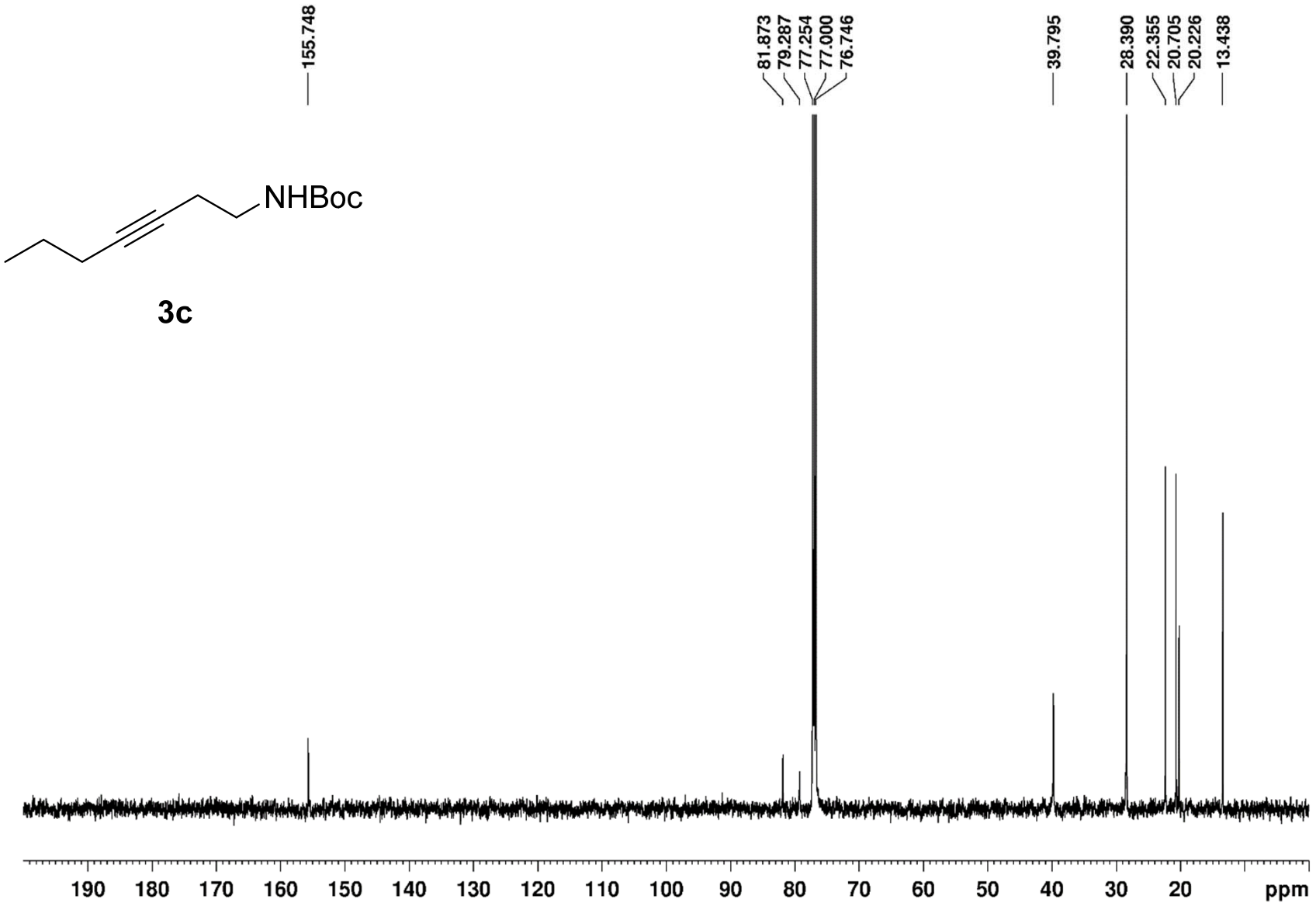
^{1}H NMR of compound **3b** (300 MHz, $CDCl_3$)



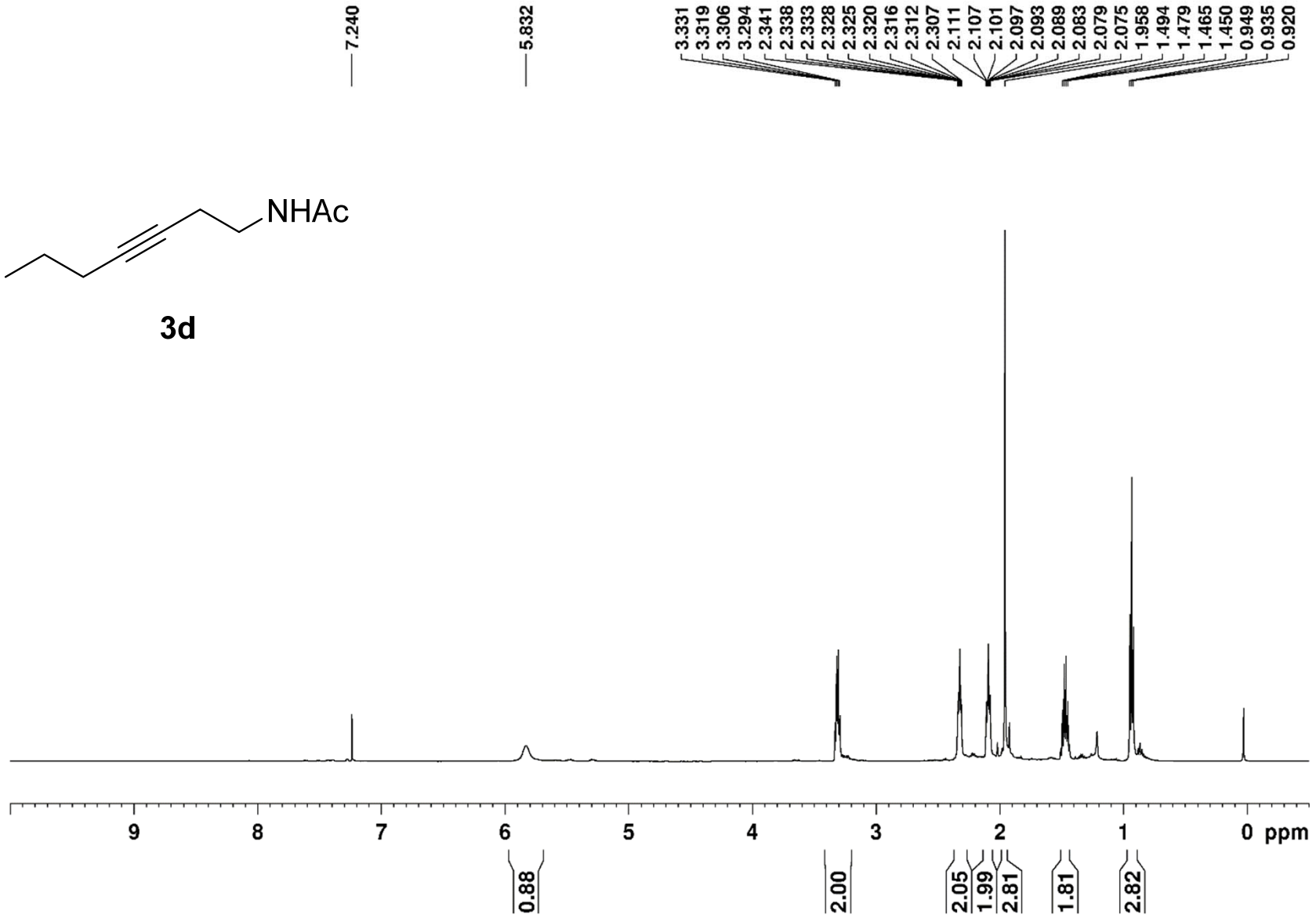
^{13}C NMR of compound **3b** (75 MHz, $CDCl_3$)



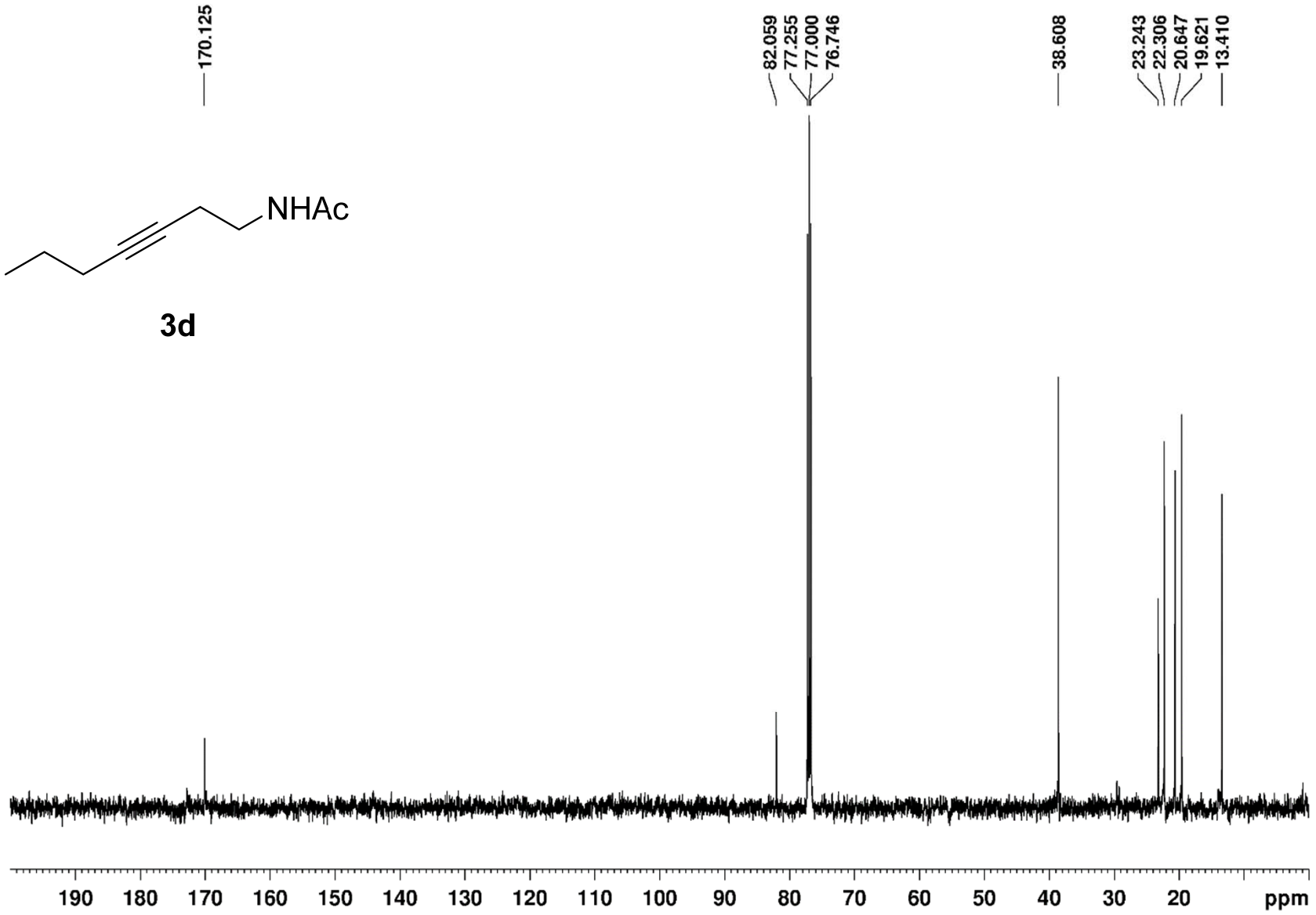
^{1}H NMR of compound **3c** (500 MHz, $CDCl_3$)



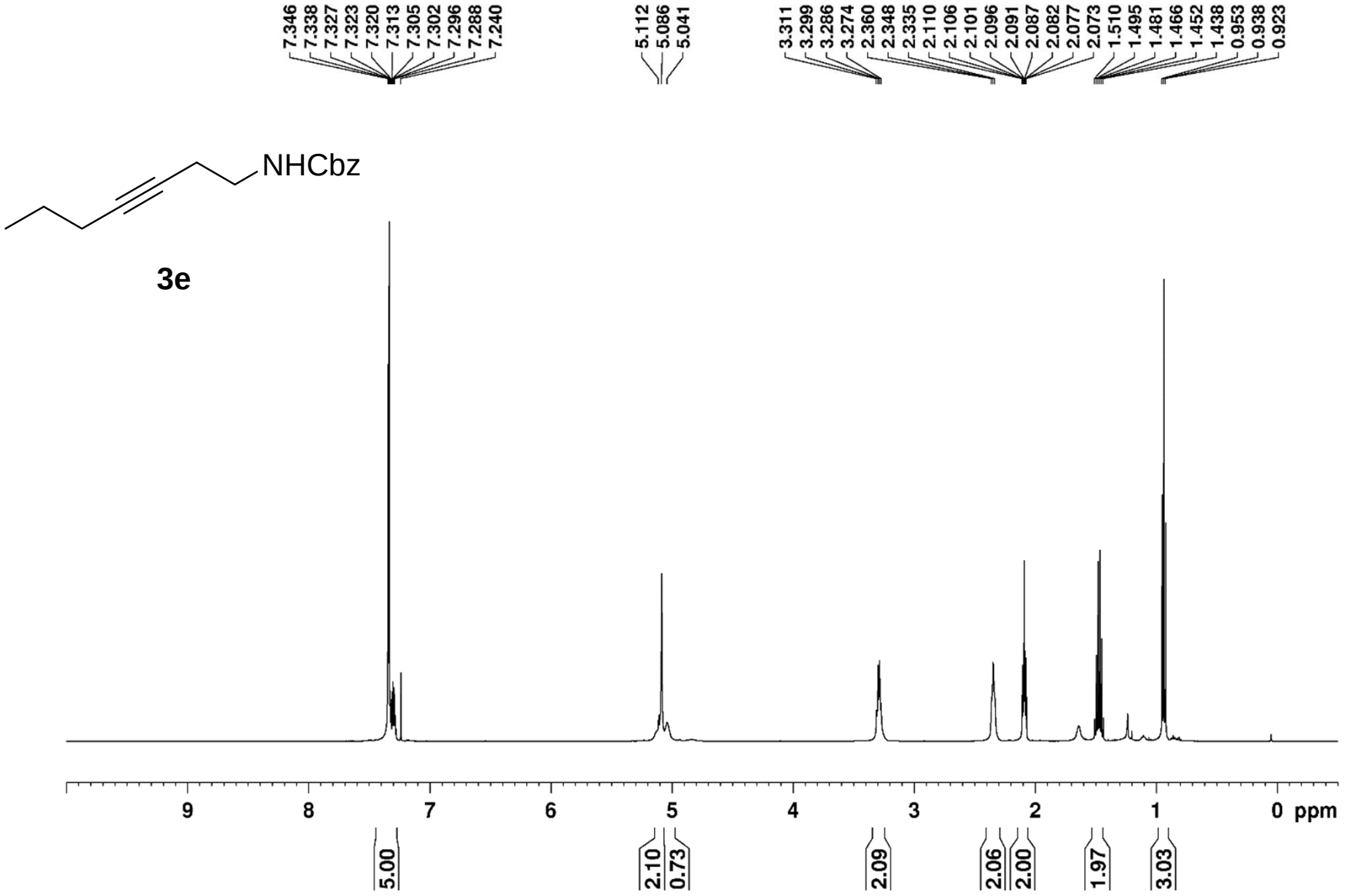
^{13}C NMR of compound **3c** (125 MHz, $CDCl_3$)



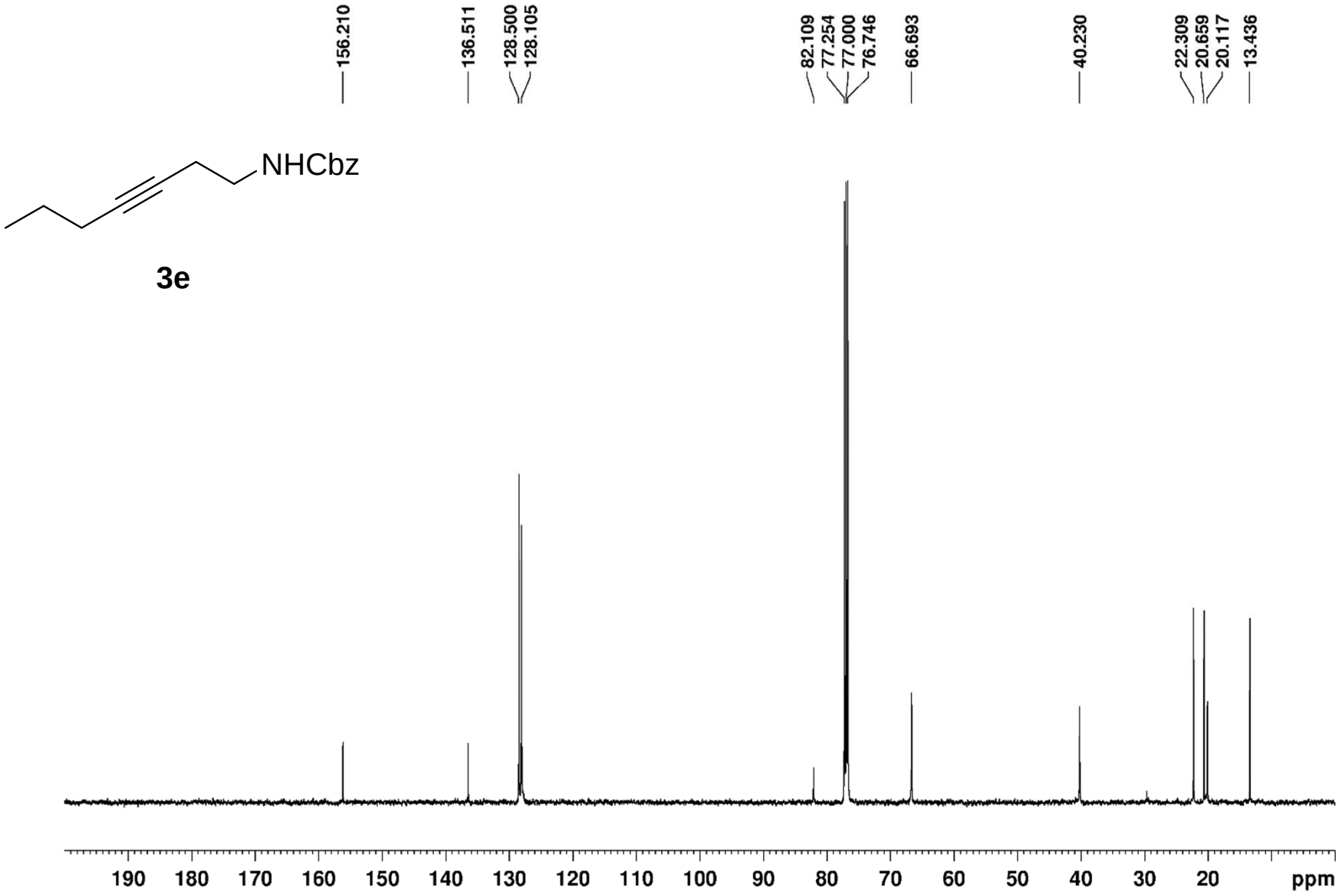
^{1}H NMR of compound **3d** (500 MHz, $CDCl_3$)



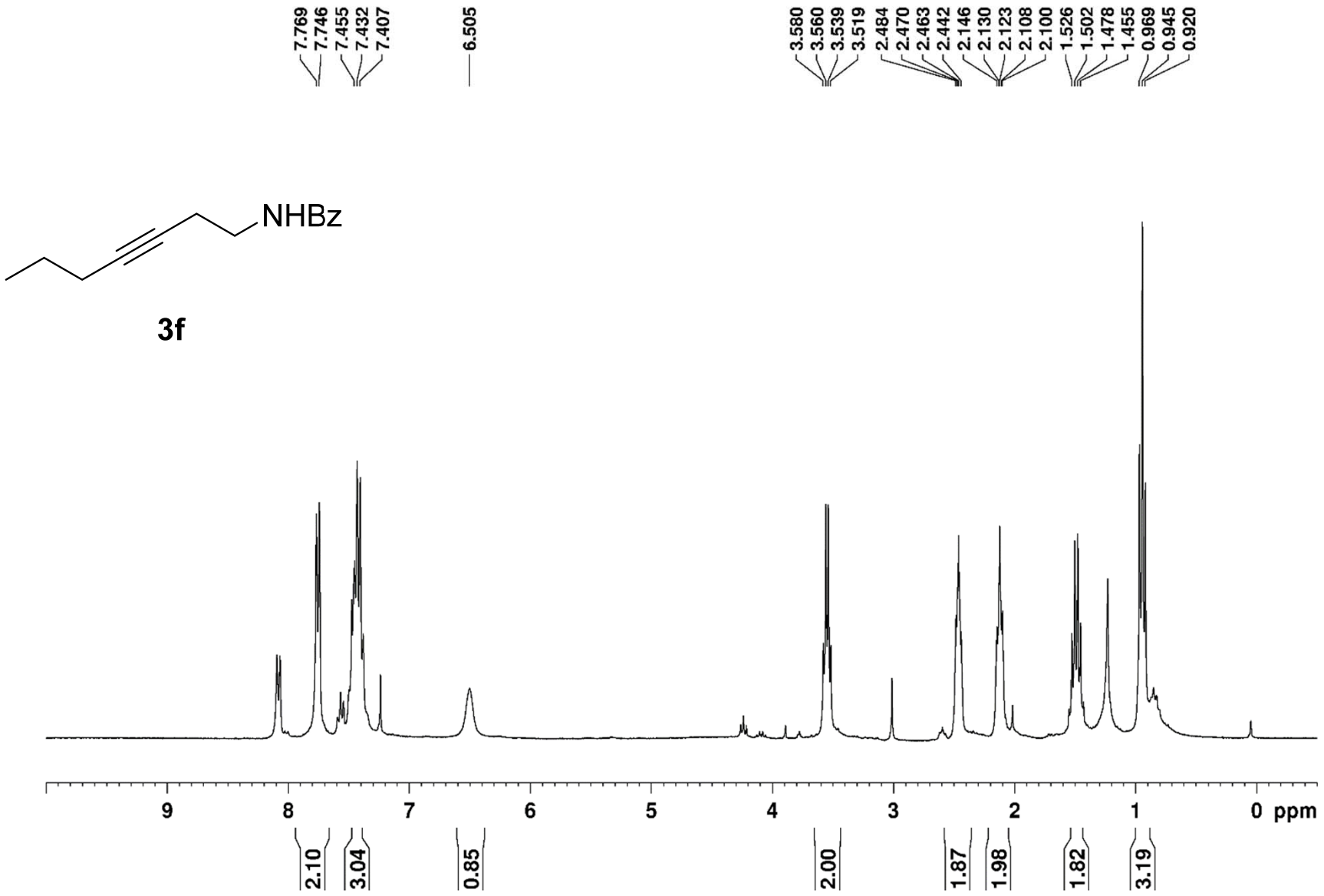
^{13}C NMR of compound **3d** (125 MHz, $CDCl_3$)



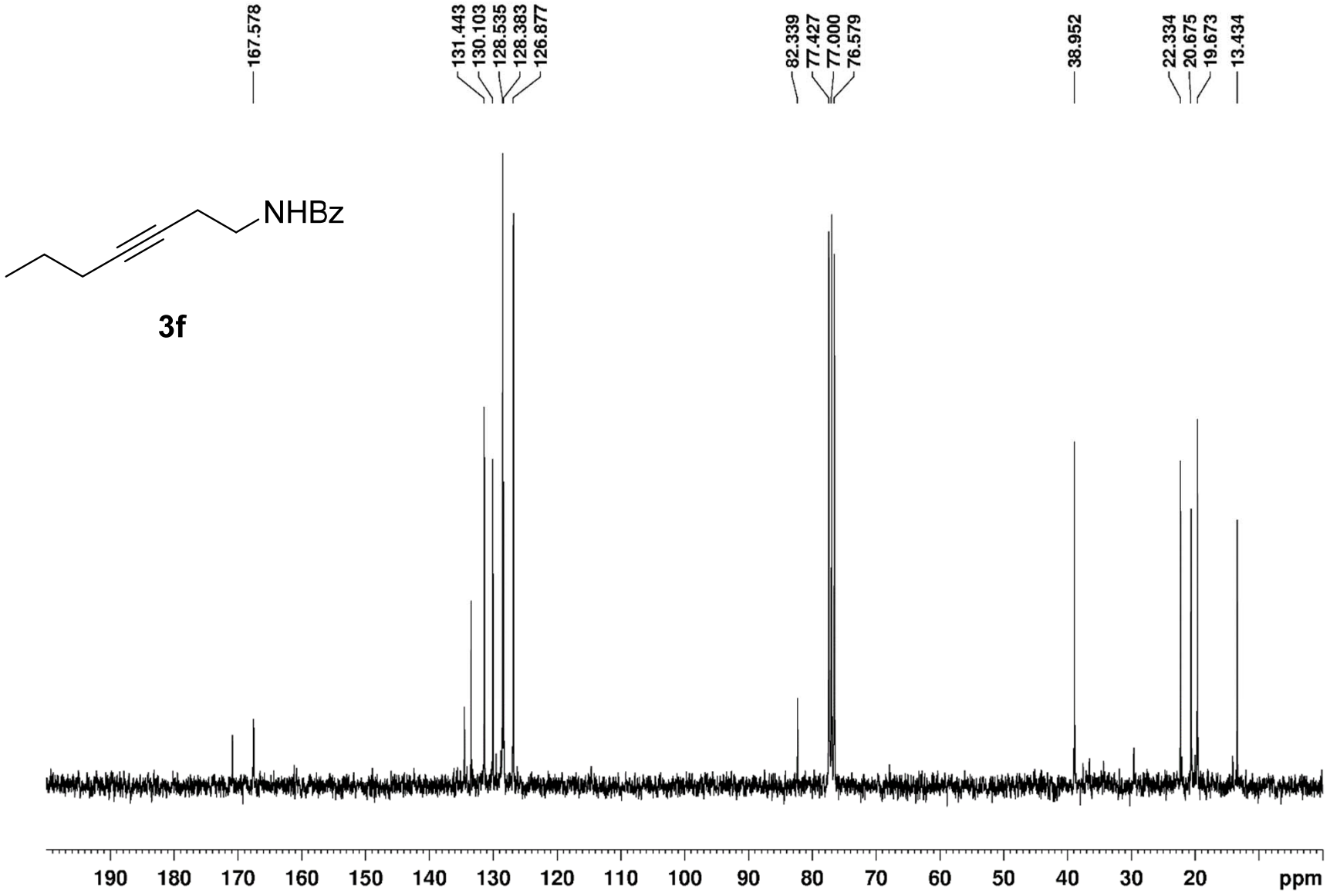
^{1}H NMR of compound **3e** (500 MHz, $CDCl_3$)



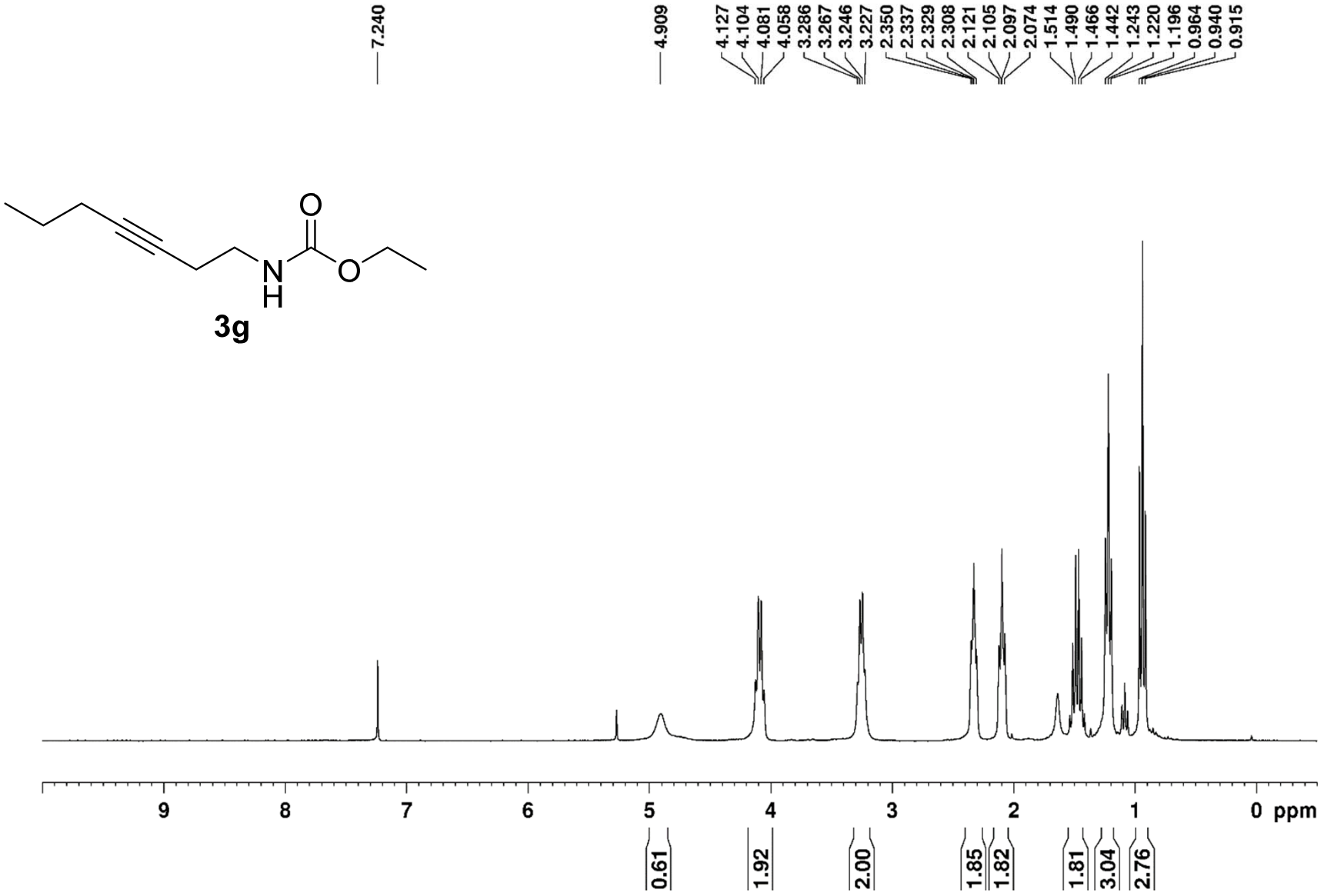
^{13}C NMR of compound **3e** (125 MHz, $CDCl_3$)



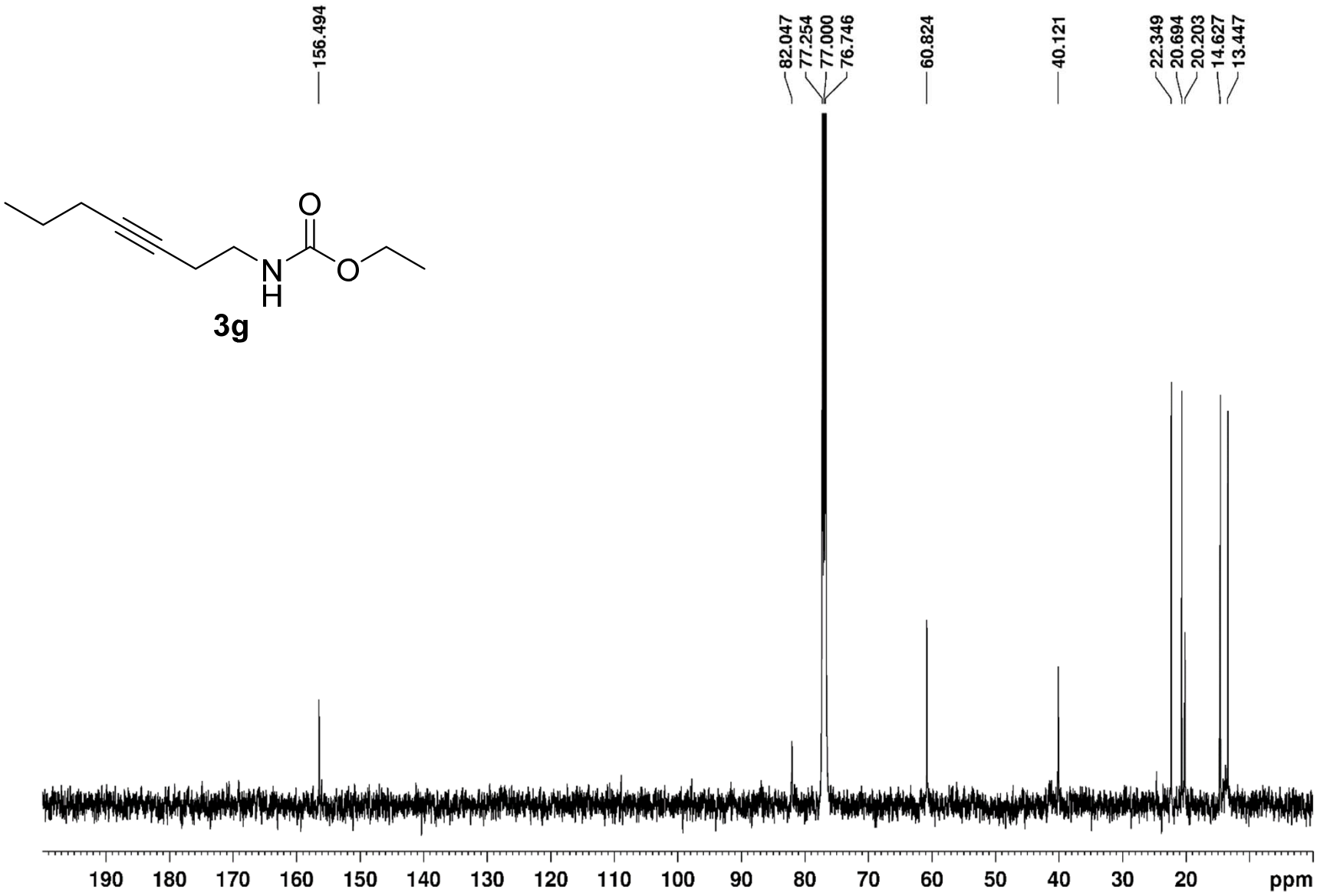
^{1}H NMR of compound **3f** (300 MHz, $CDCl_3$)



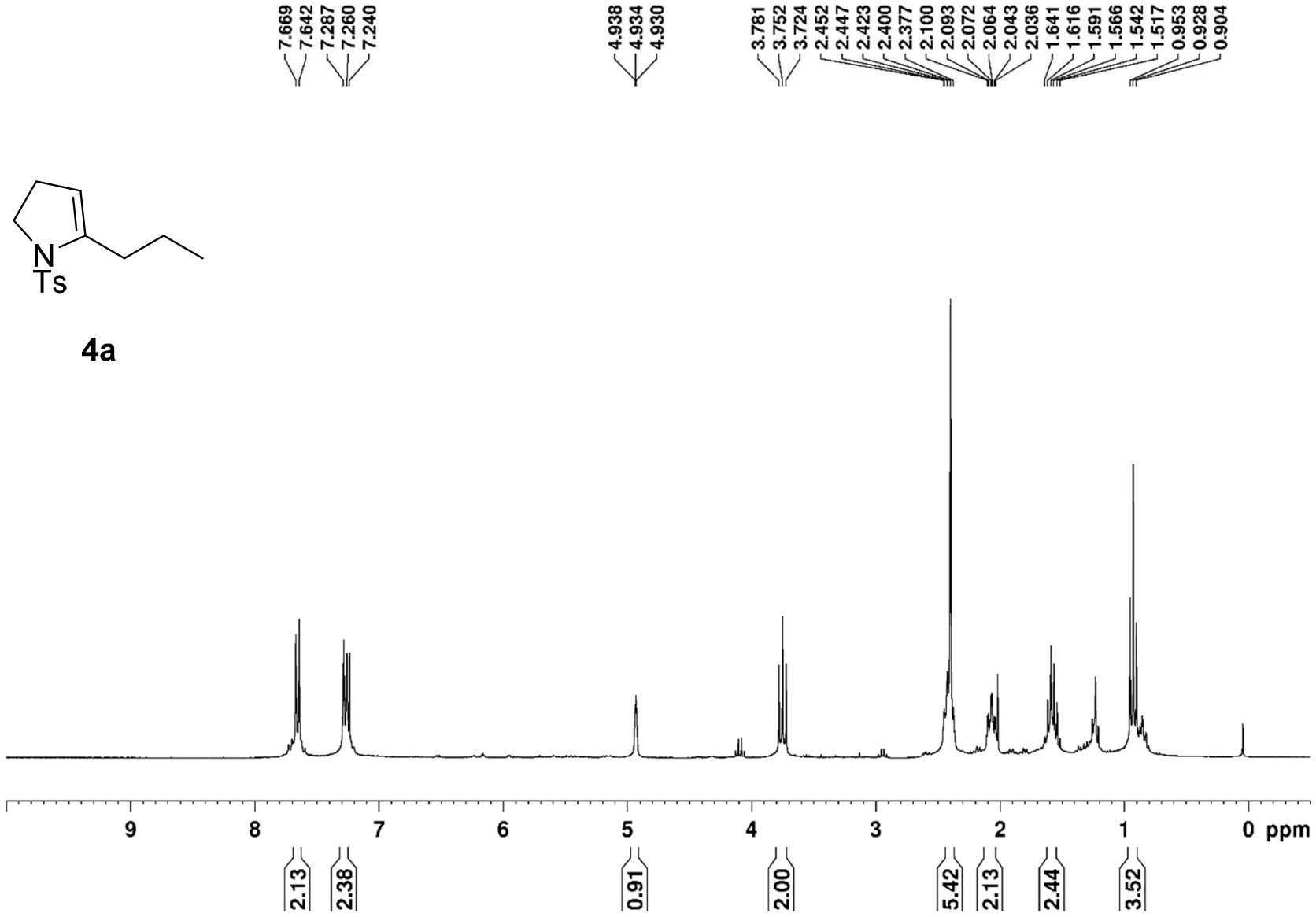
^{13}C NMR of compound **3f** (75 MHz, $CDCl_3$)



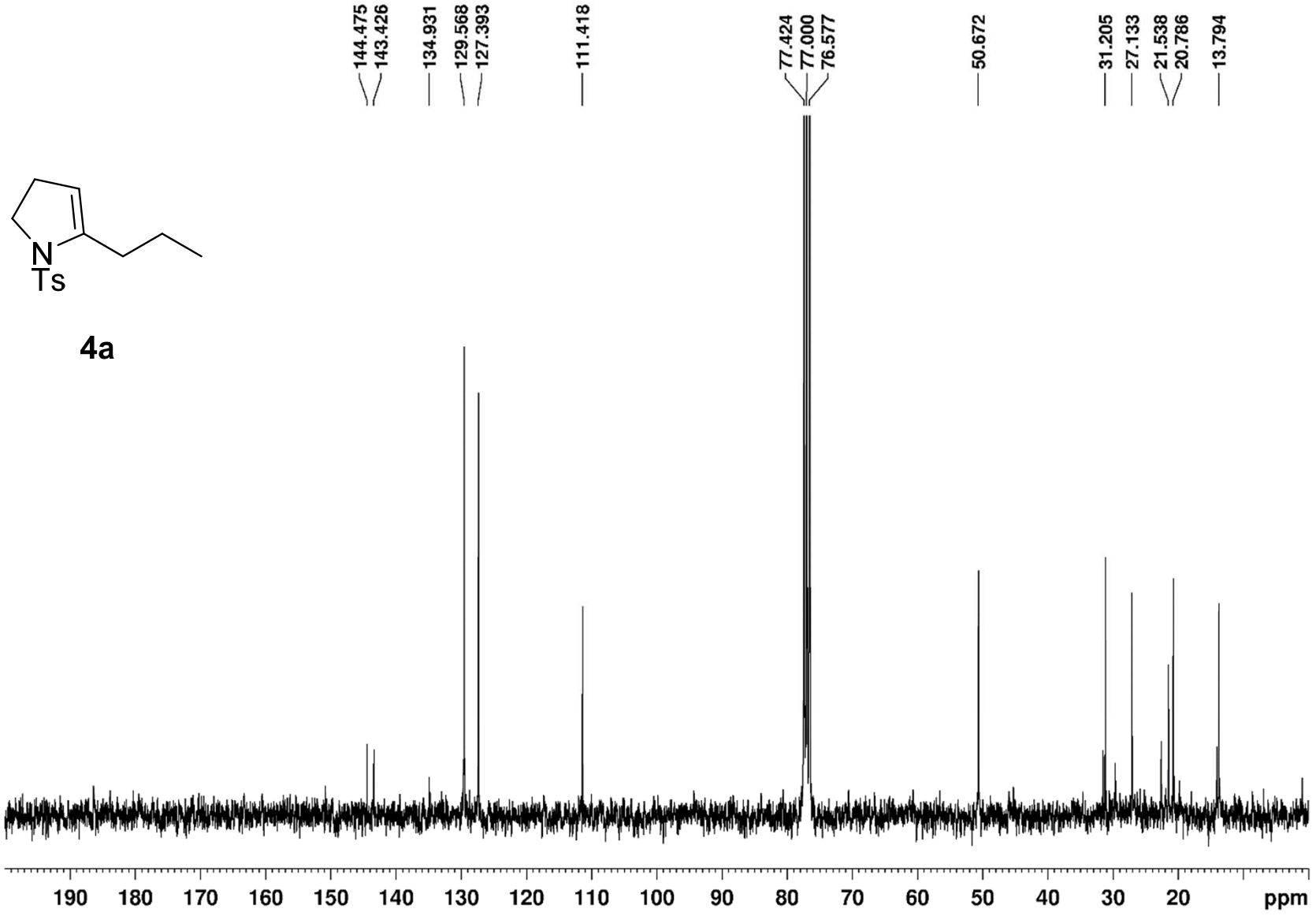
^{1}H NMR of compound **3g** (300 MHz, $CDCl_3$)



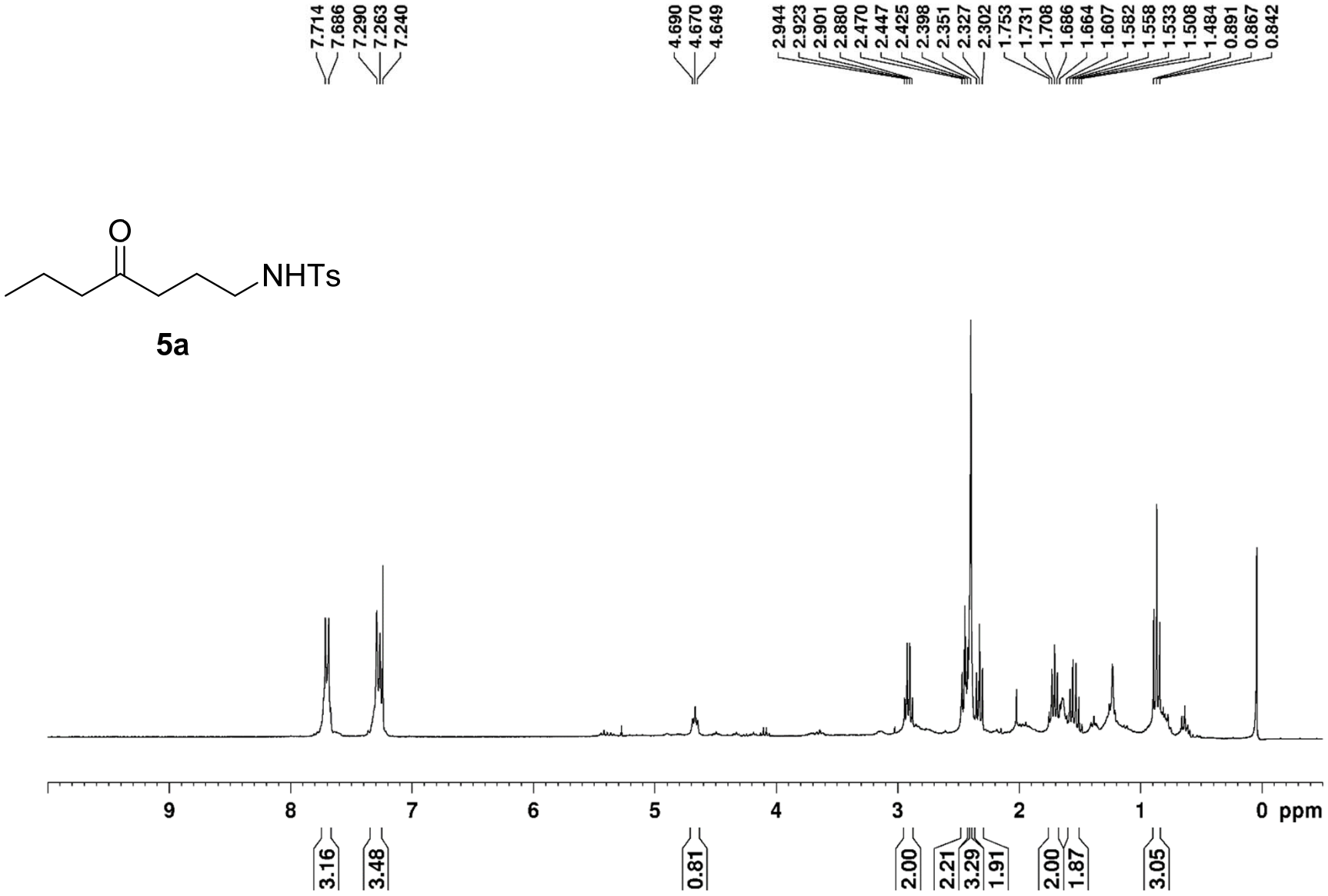
^{13}C NMR of compound **3g** (125 MHz, $CDCl_3$)



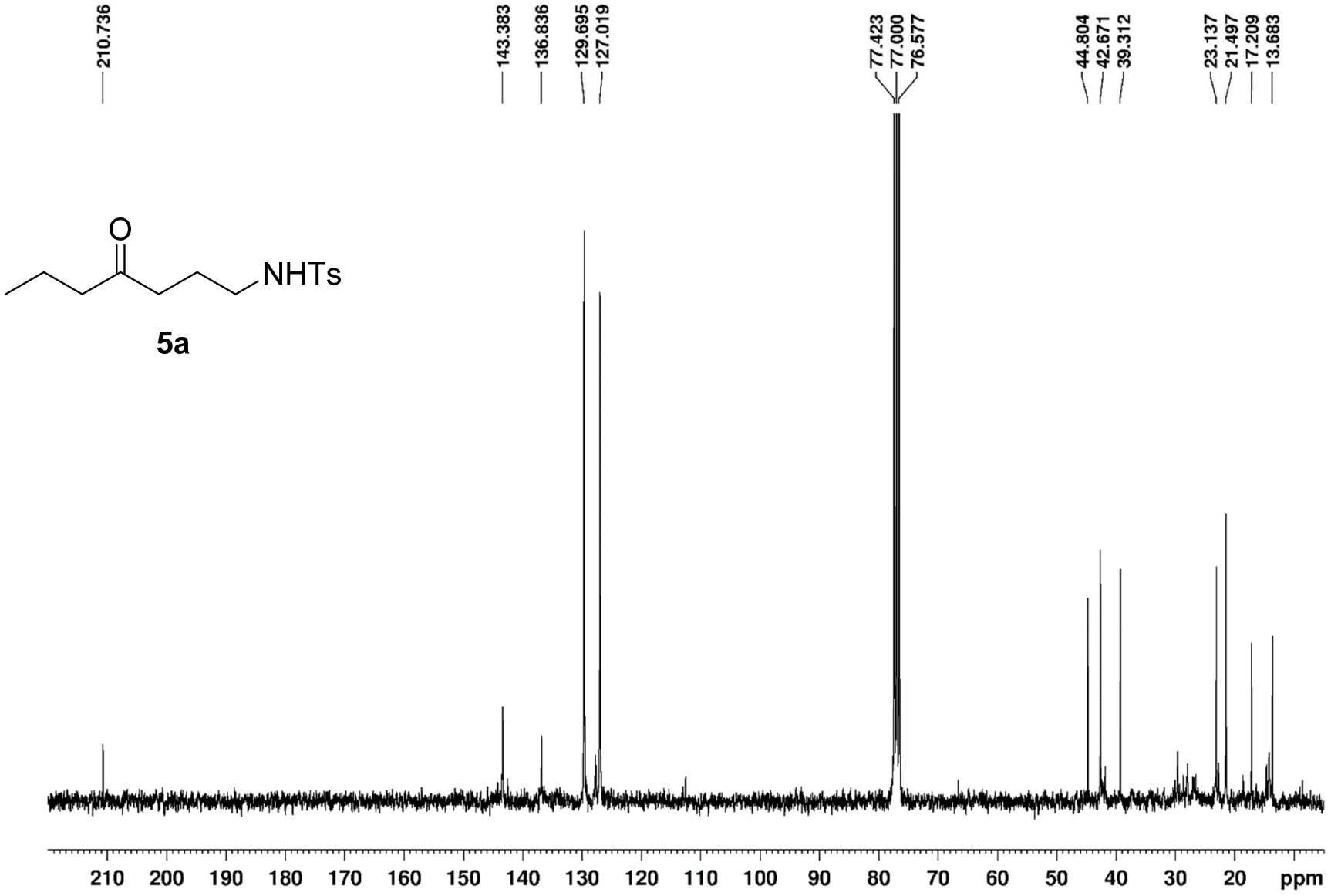
^{1}H NMR of compound **4a** (300 MHz, $CDCl_3$)



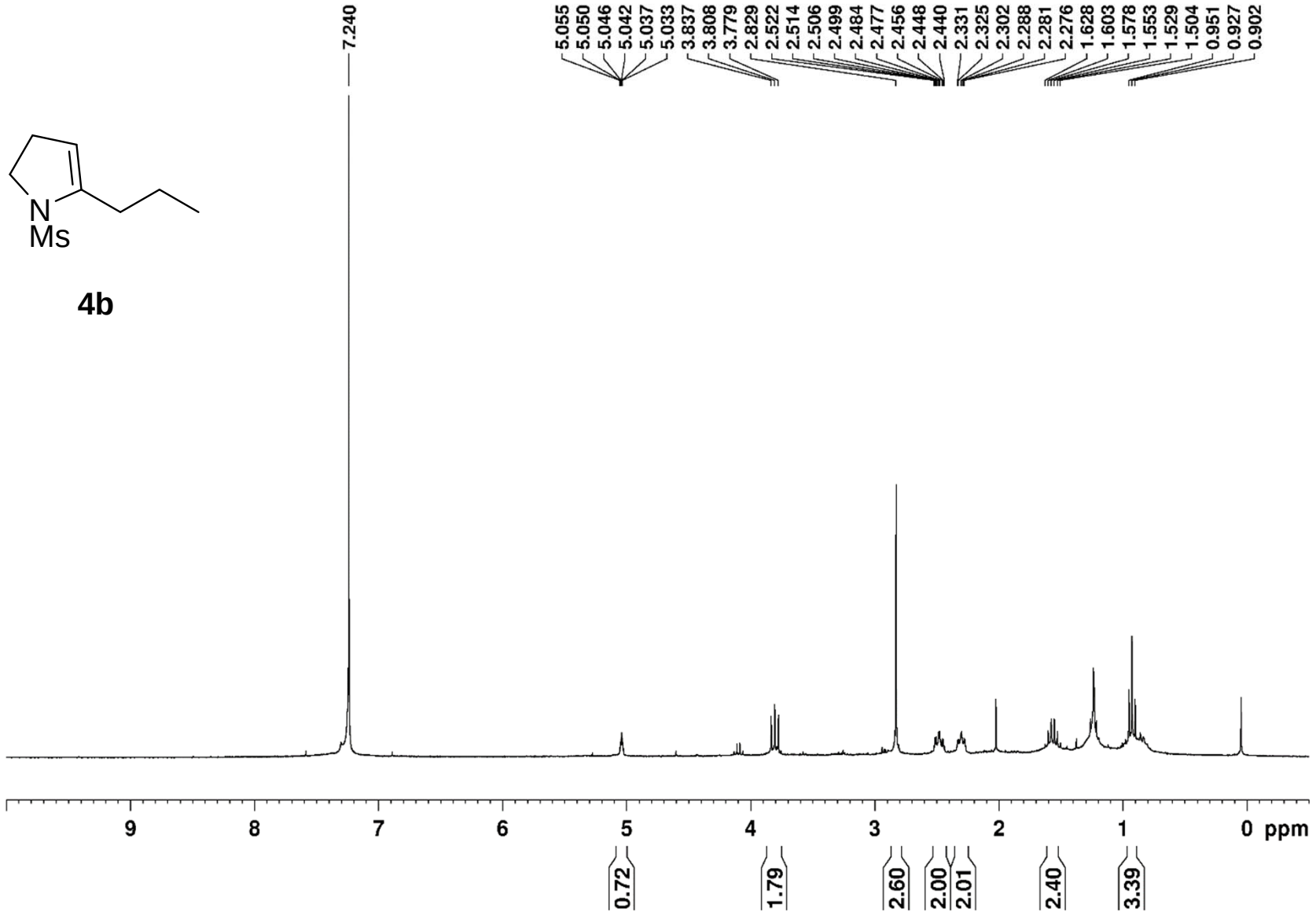
^{13}C NMR of compound **4a** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **5a** (300 MHz, $CDCl_3$)

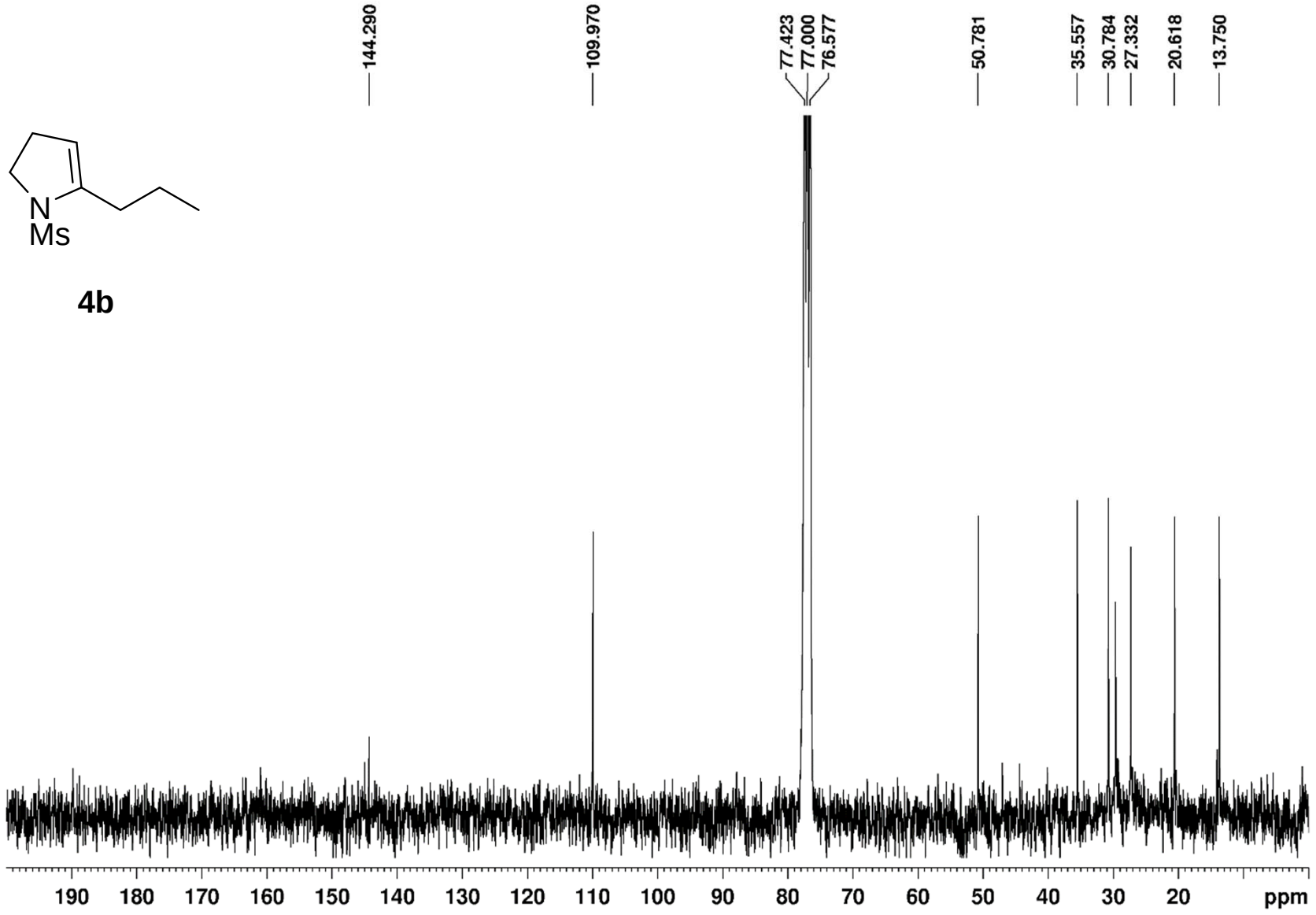


^{13}C NMR of compound **5a** (75 MHz, $CDCl_3$)

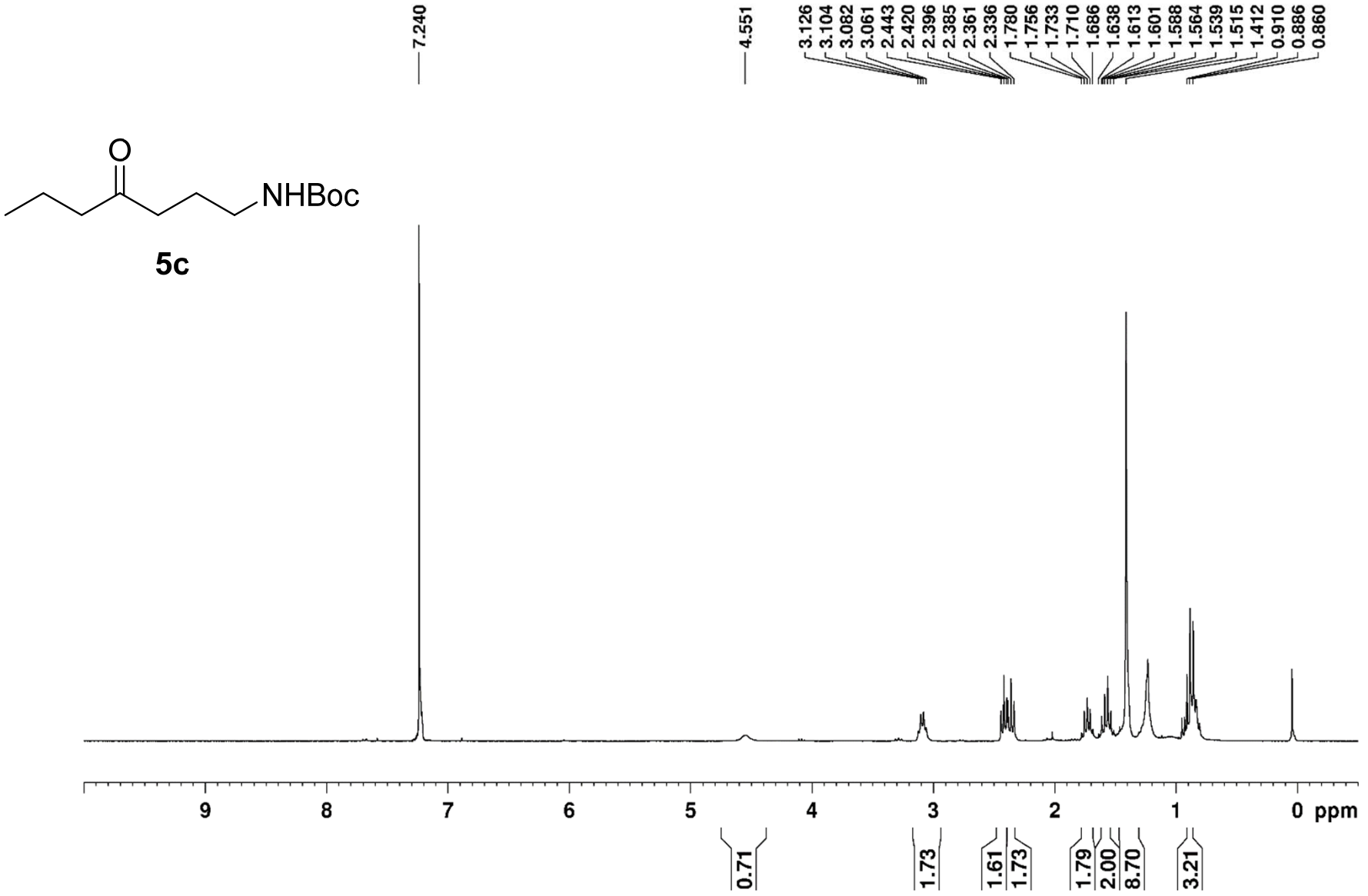


4b

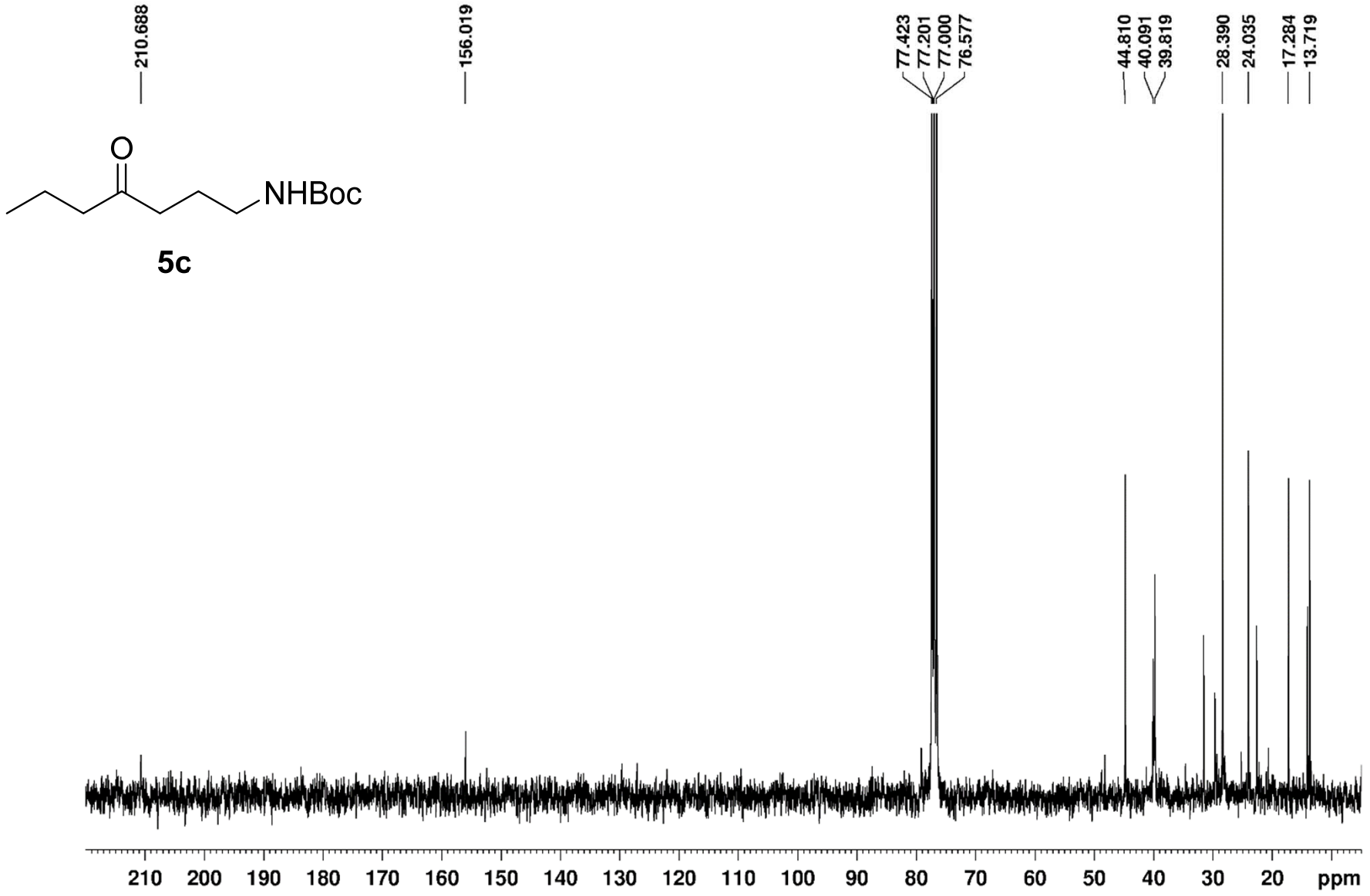
^{1}H NMR of compound **4b** (300 MHz, $CDCl_3$)



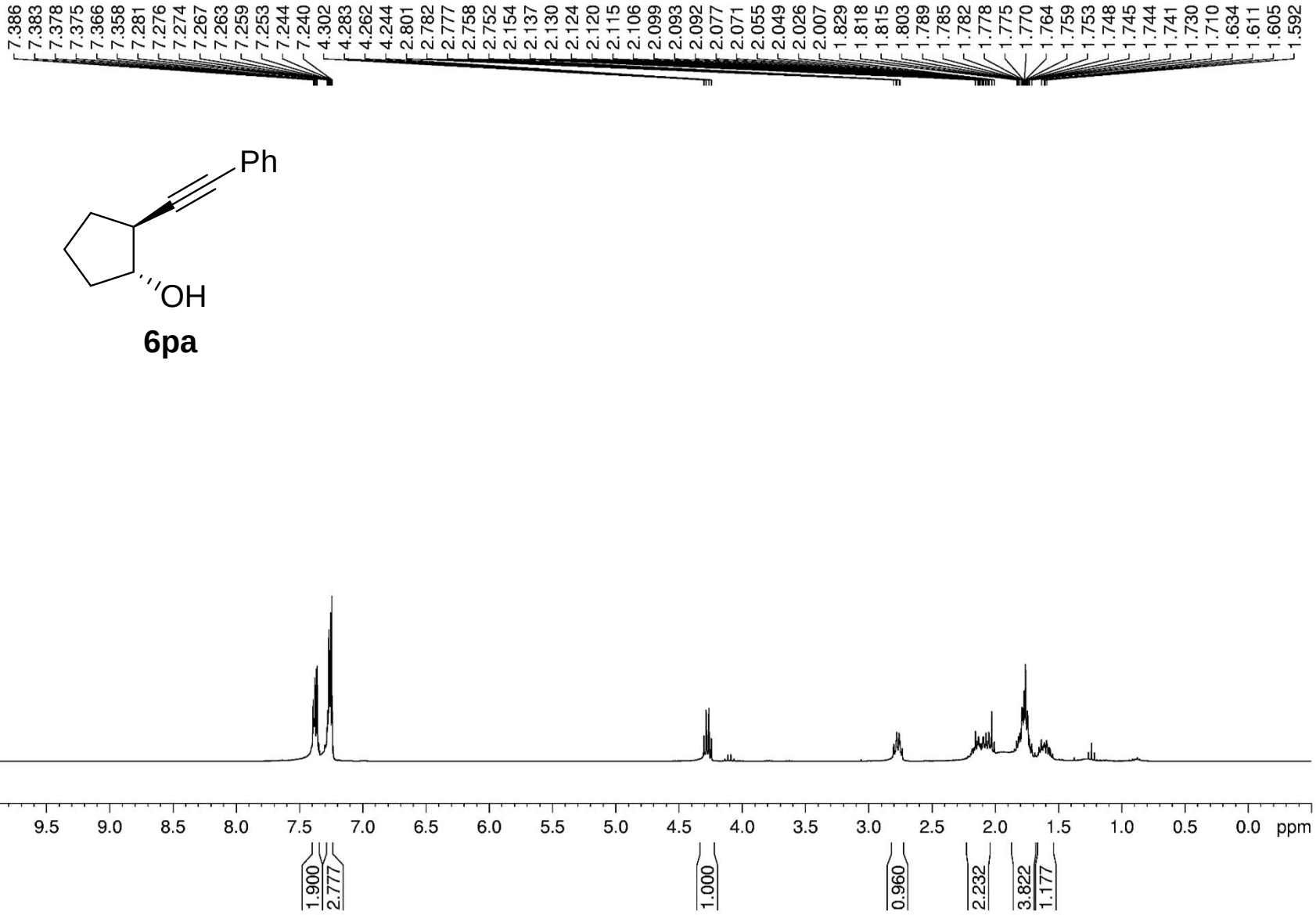
^{13}C NMR of compound **4b** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **5c** (300 MHz, $CDCl_3$)

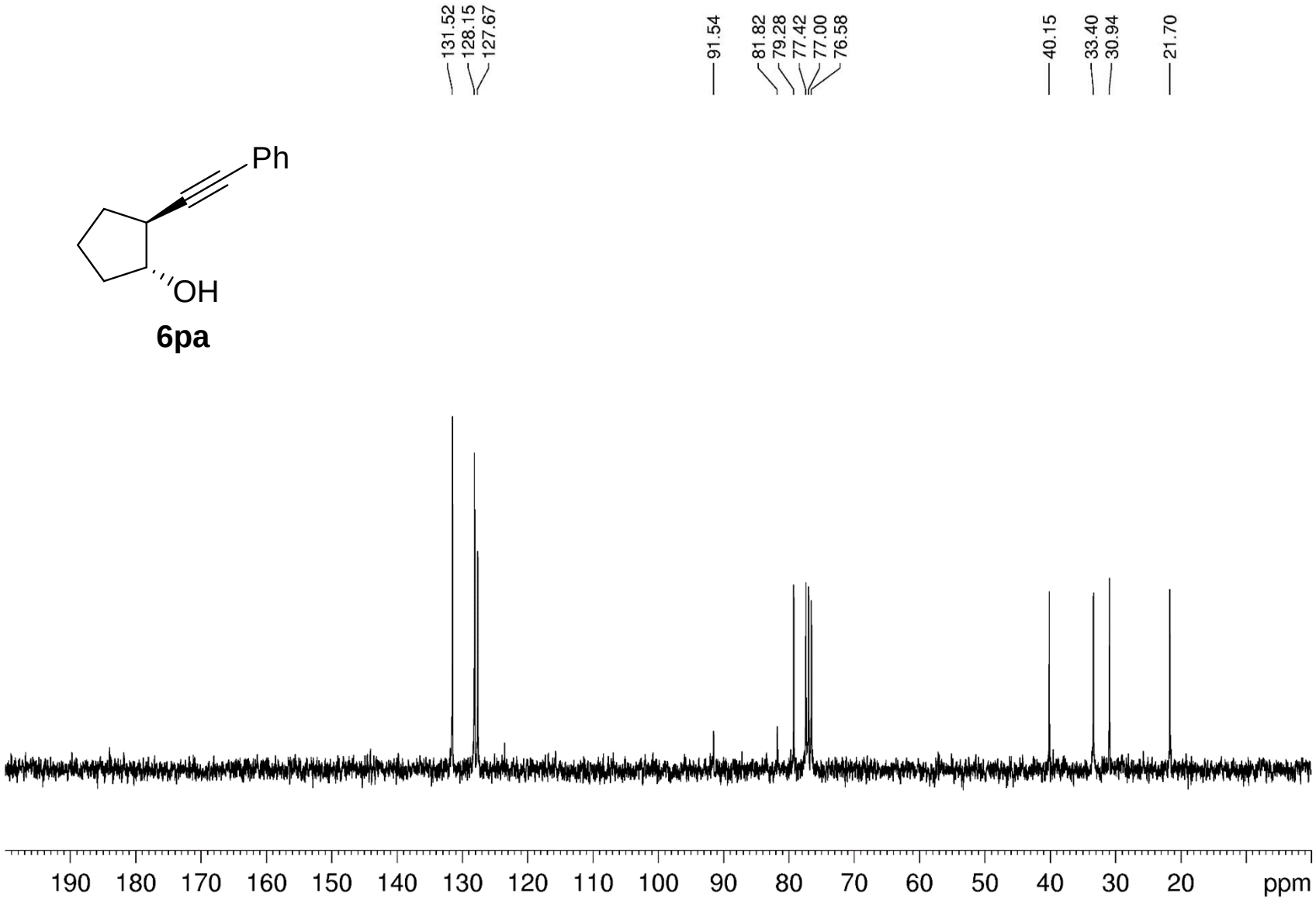


^{13}C NMR of compound **5c** (75 MHz, $CDCl_3$)

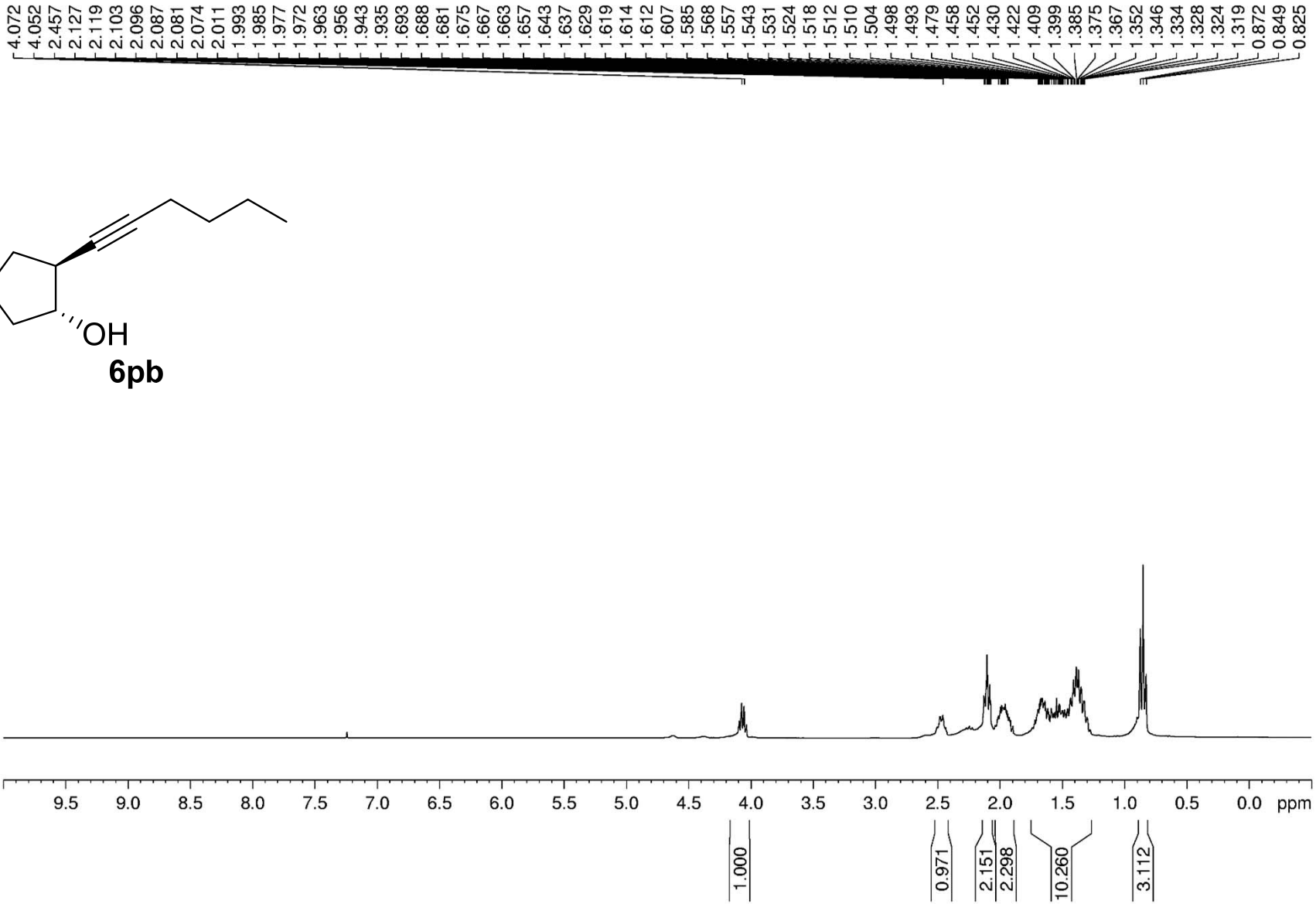


6pa

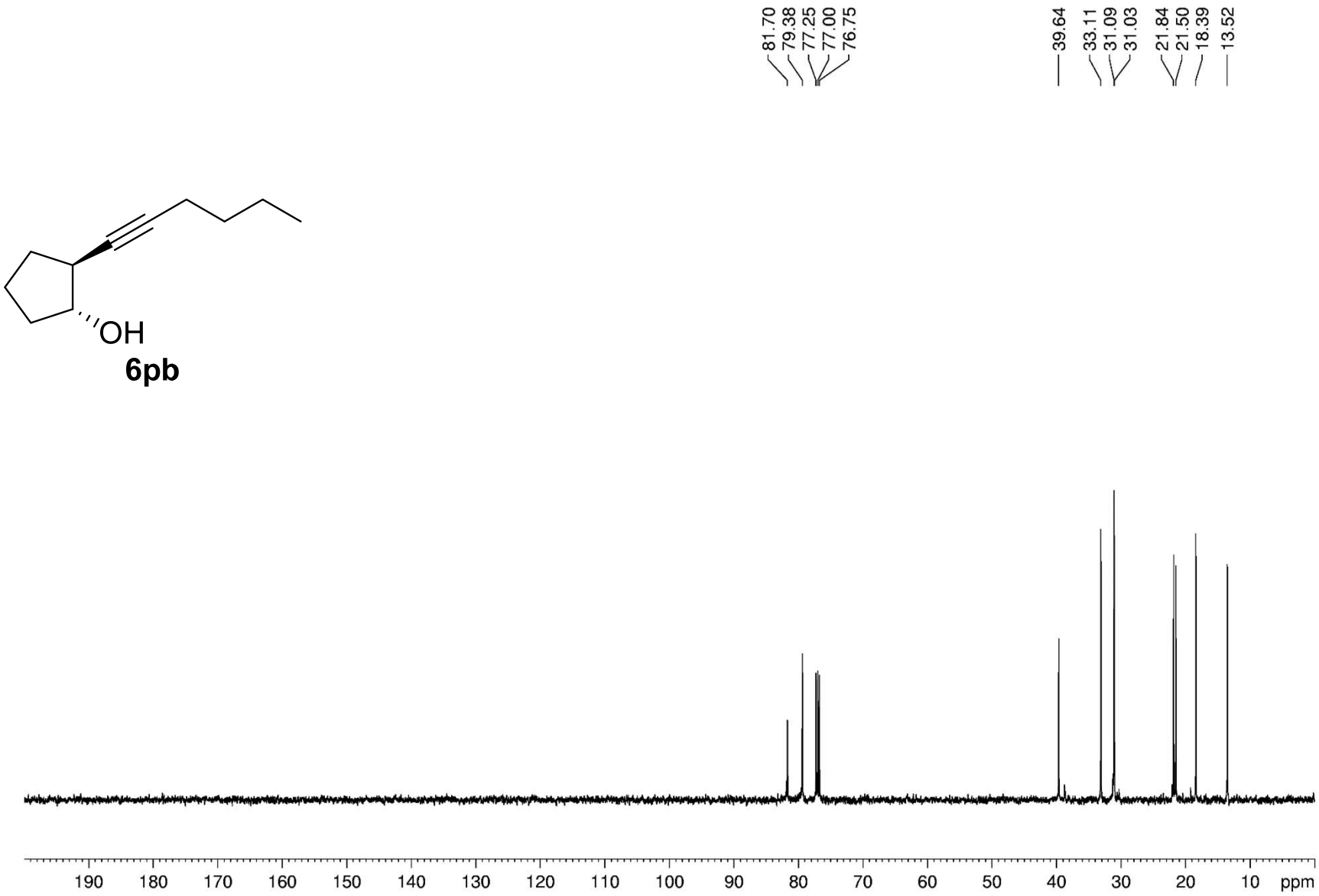
^{1}H NMR of compound **6pa** (300 MHz, $CDCl_3$)



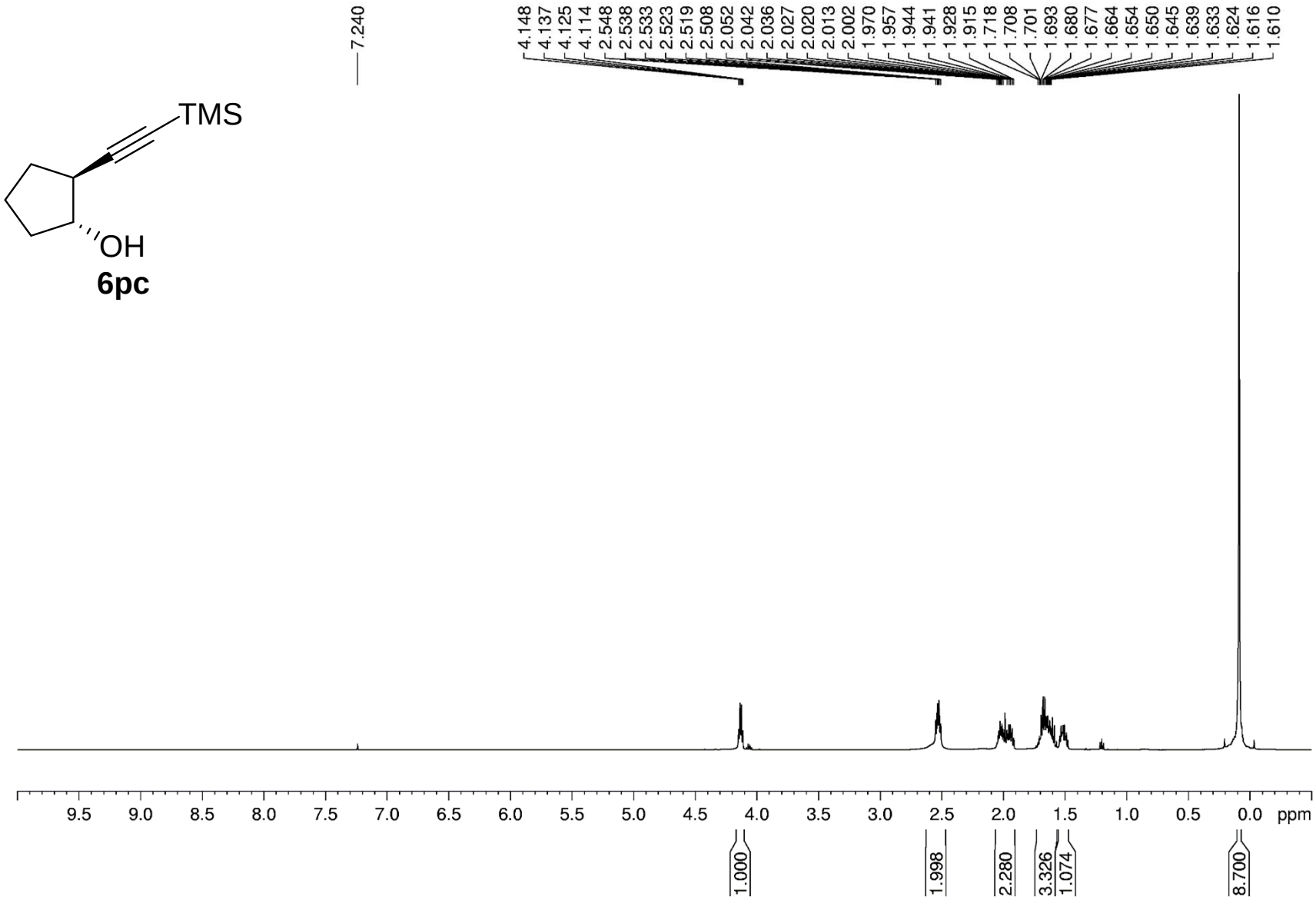
^{13}C NMR of compound **6pa** (75 MHz, $CDCl_3$)



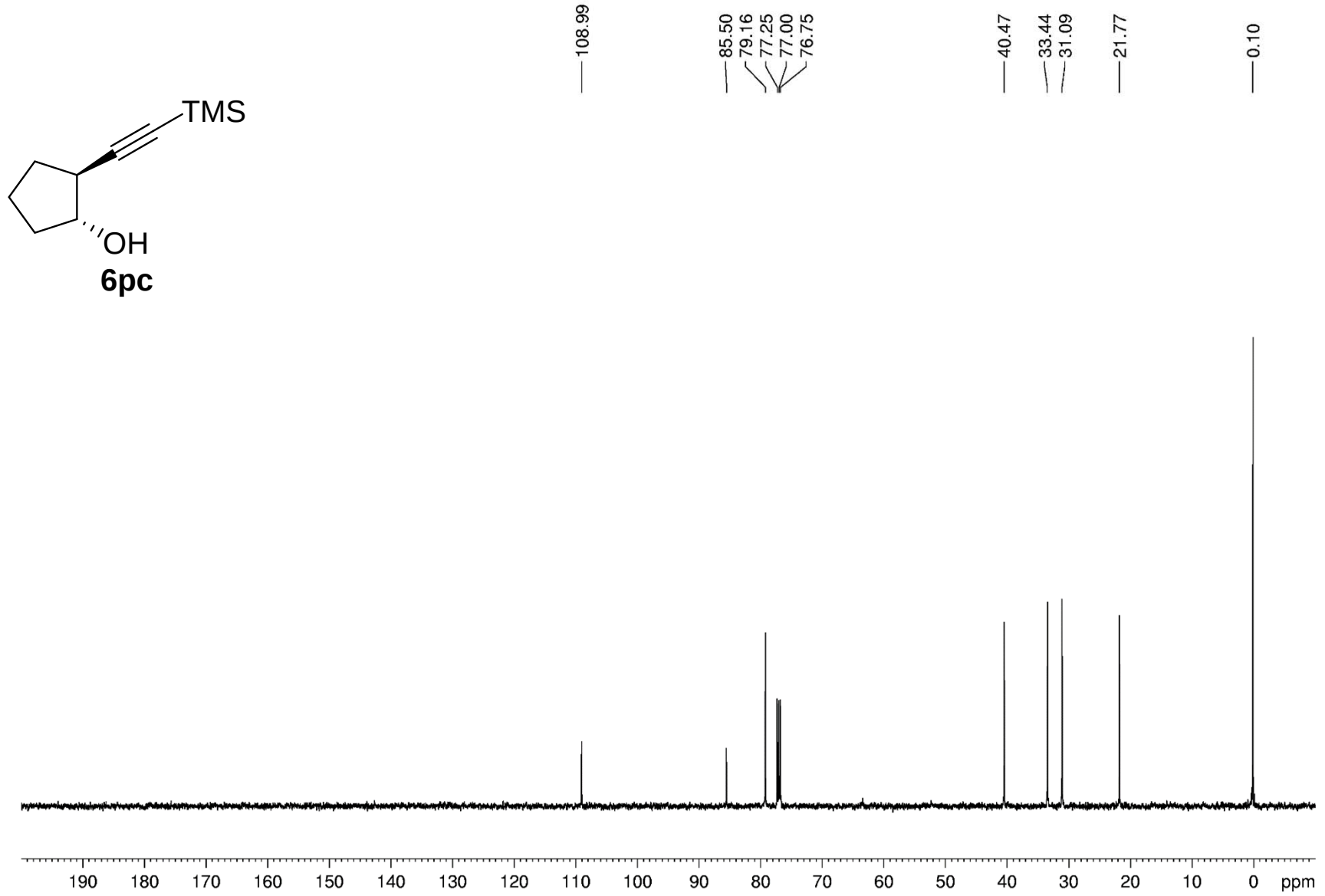
^{1}H NMR of compound **6pb** (300 MHz, $CDCl_3$)



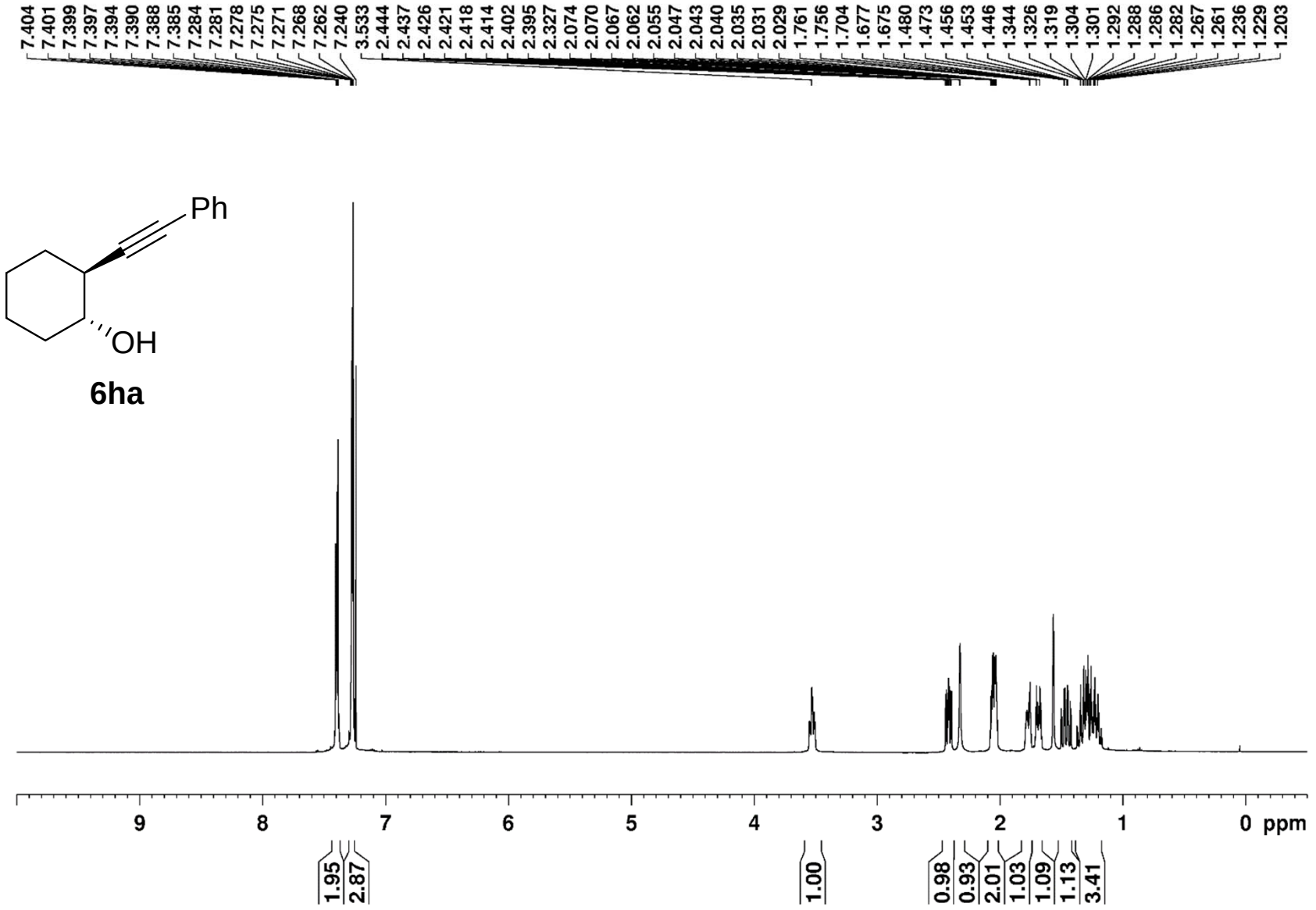
^{13}C NMR of compound **6pb** (125 MHz, $CDCl_3$)



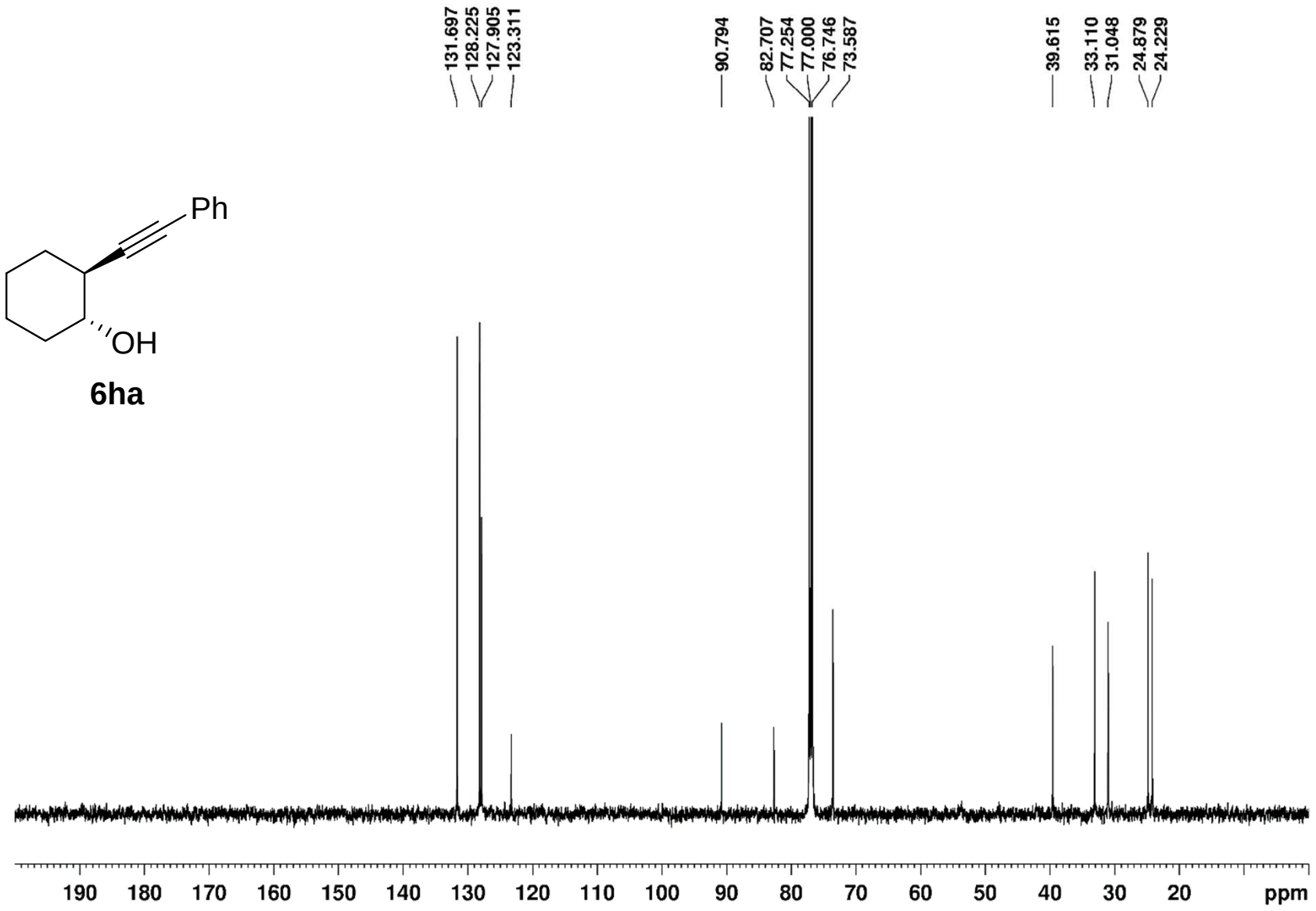
^{1}H NMR of compound **6pc** (500 MHz, $CDCl_3$)



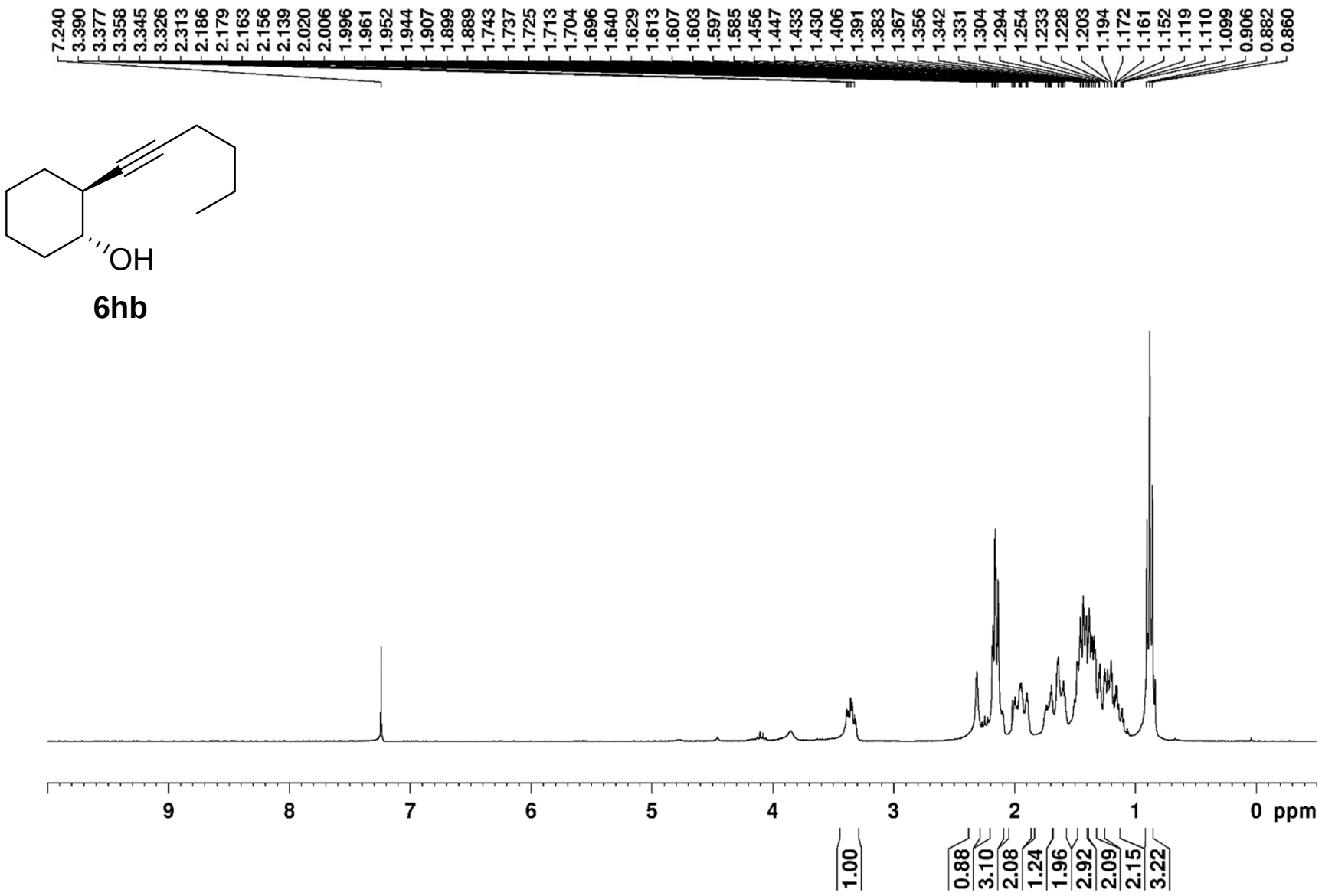
^{13}C NMR of compound **6pc** (75 MHz, $CDCl_3$)



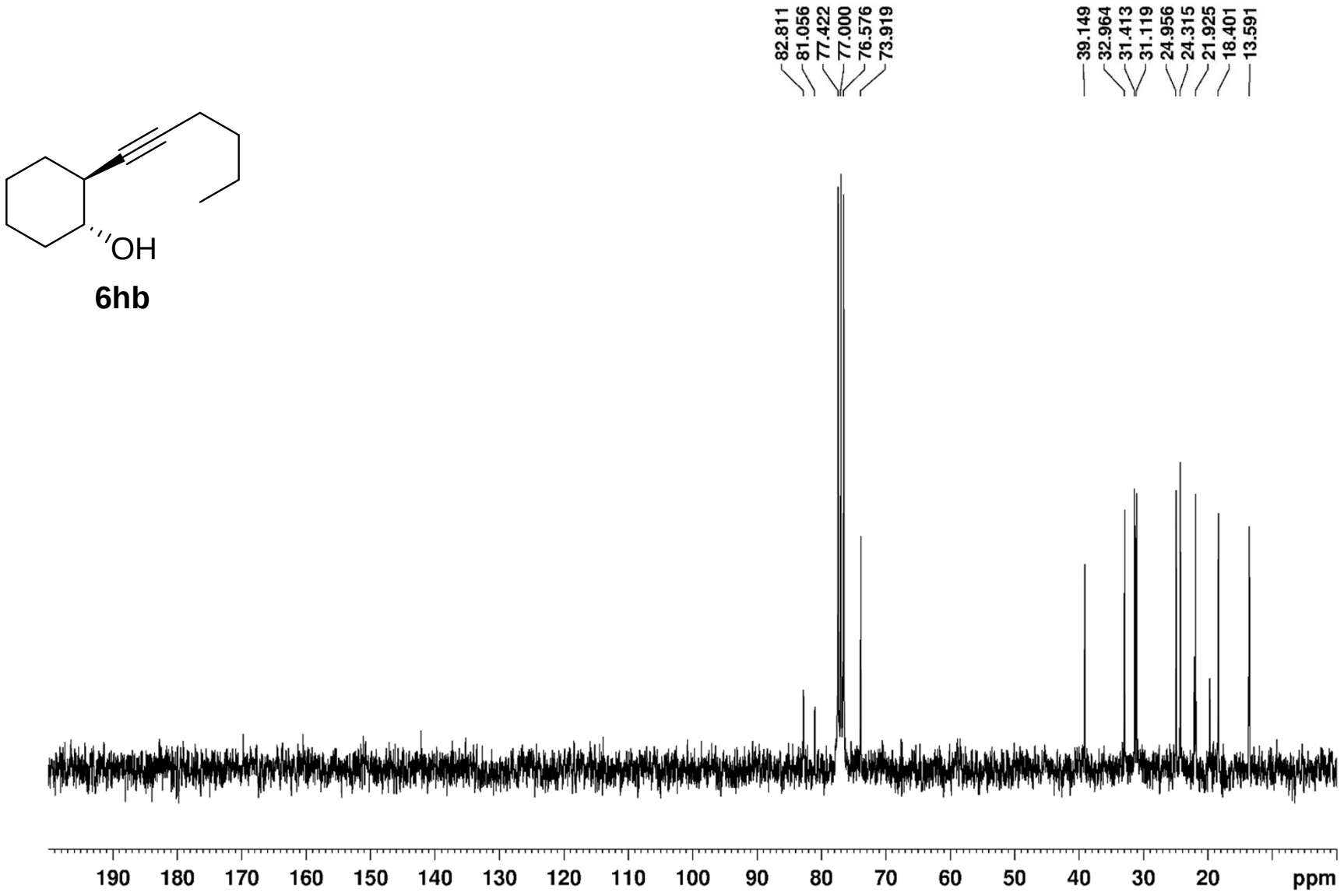
^{1}H NMR of compound **6ha** (500 MHz, $CDCl_3$)



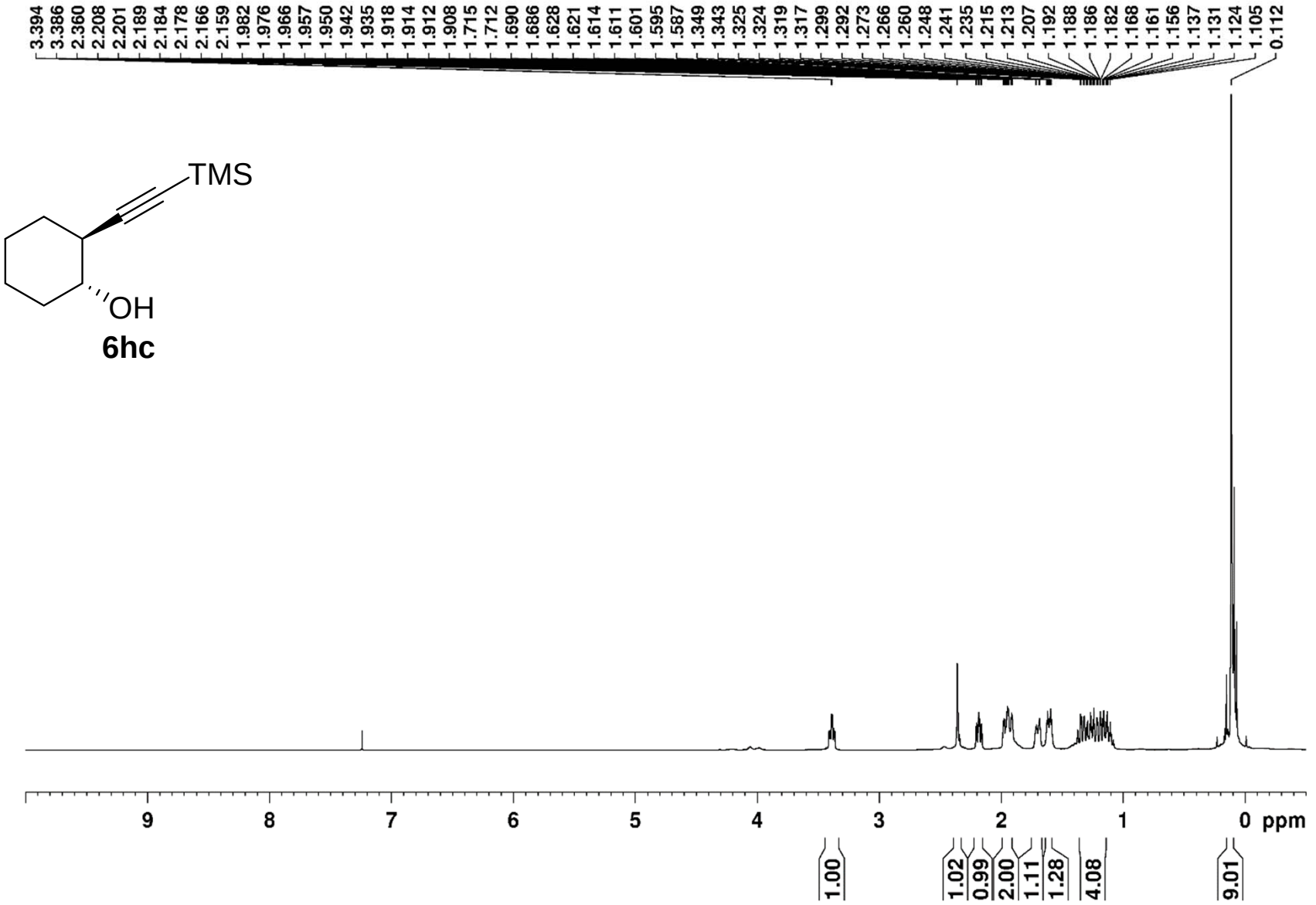
^{13}C NMR of compound **6ha** (125 MHz, $CDCl_3$)



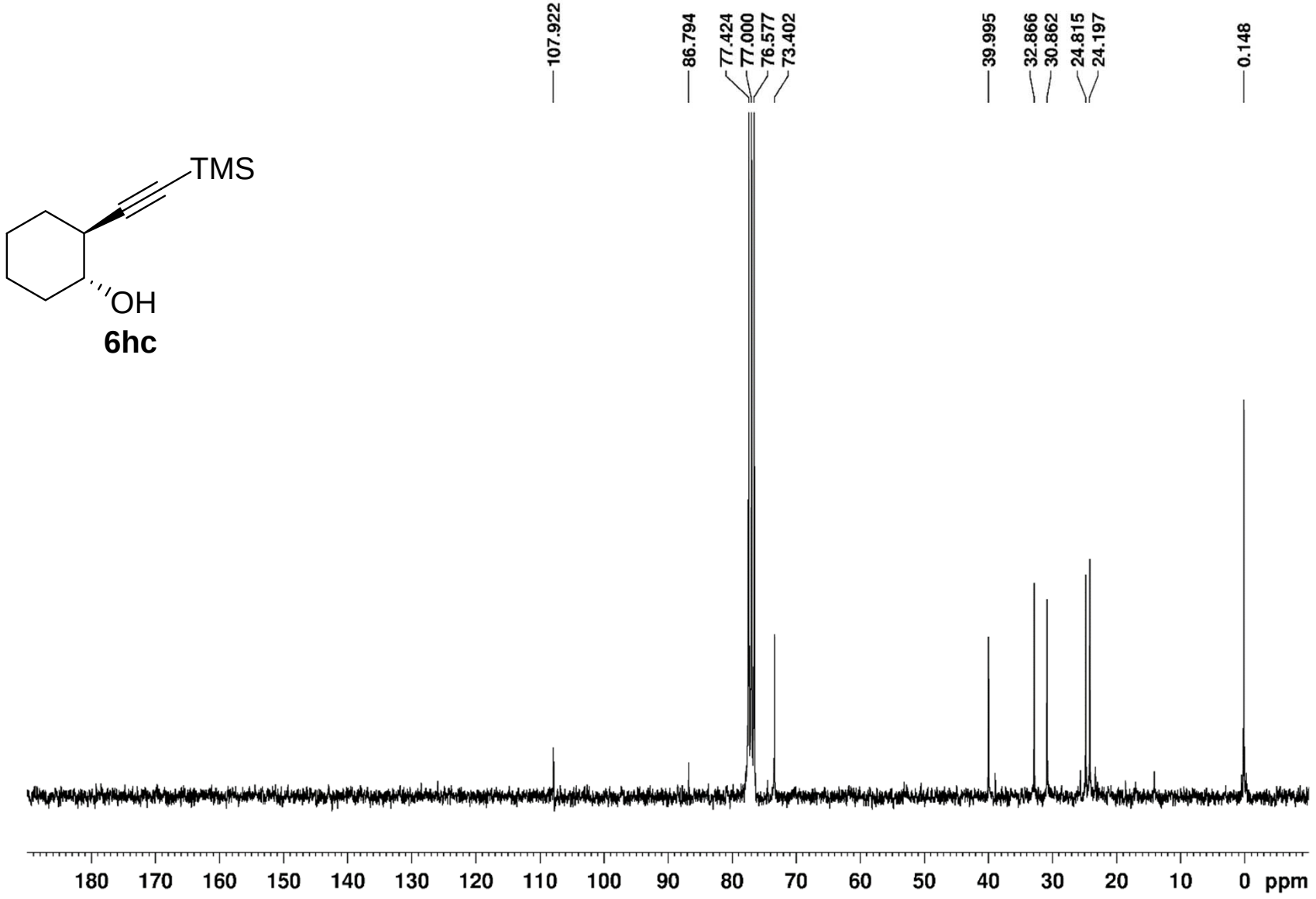
^{1}H NMR of compound **6hb** (300 MHz, $CDCl_3$)



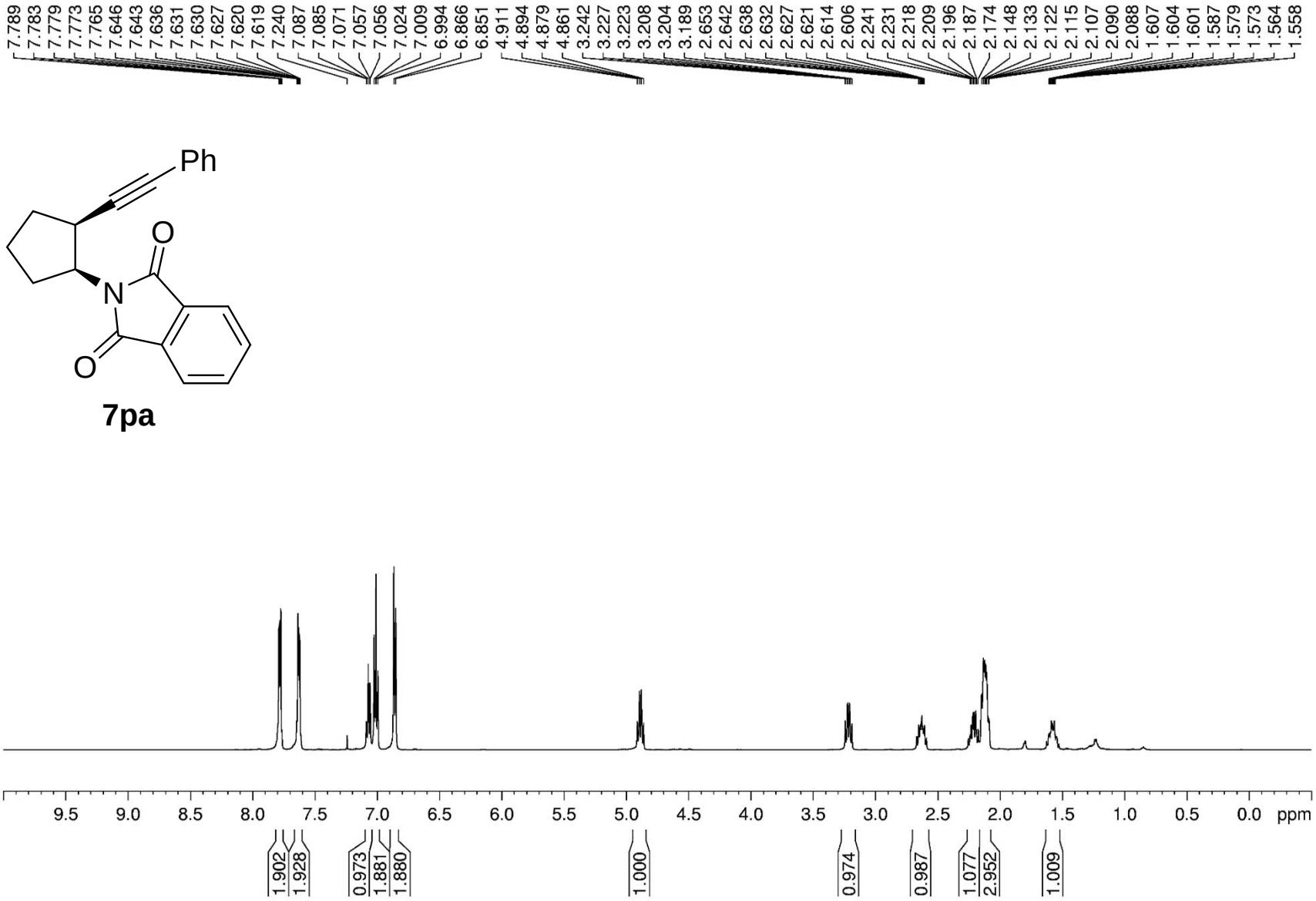
^{13}C NMR of compound **6hb** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **6hc** (500 MHz, $CDCl_3$)

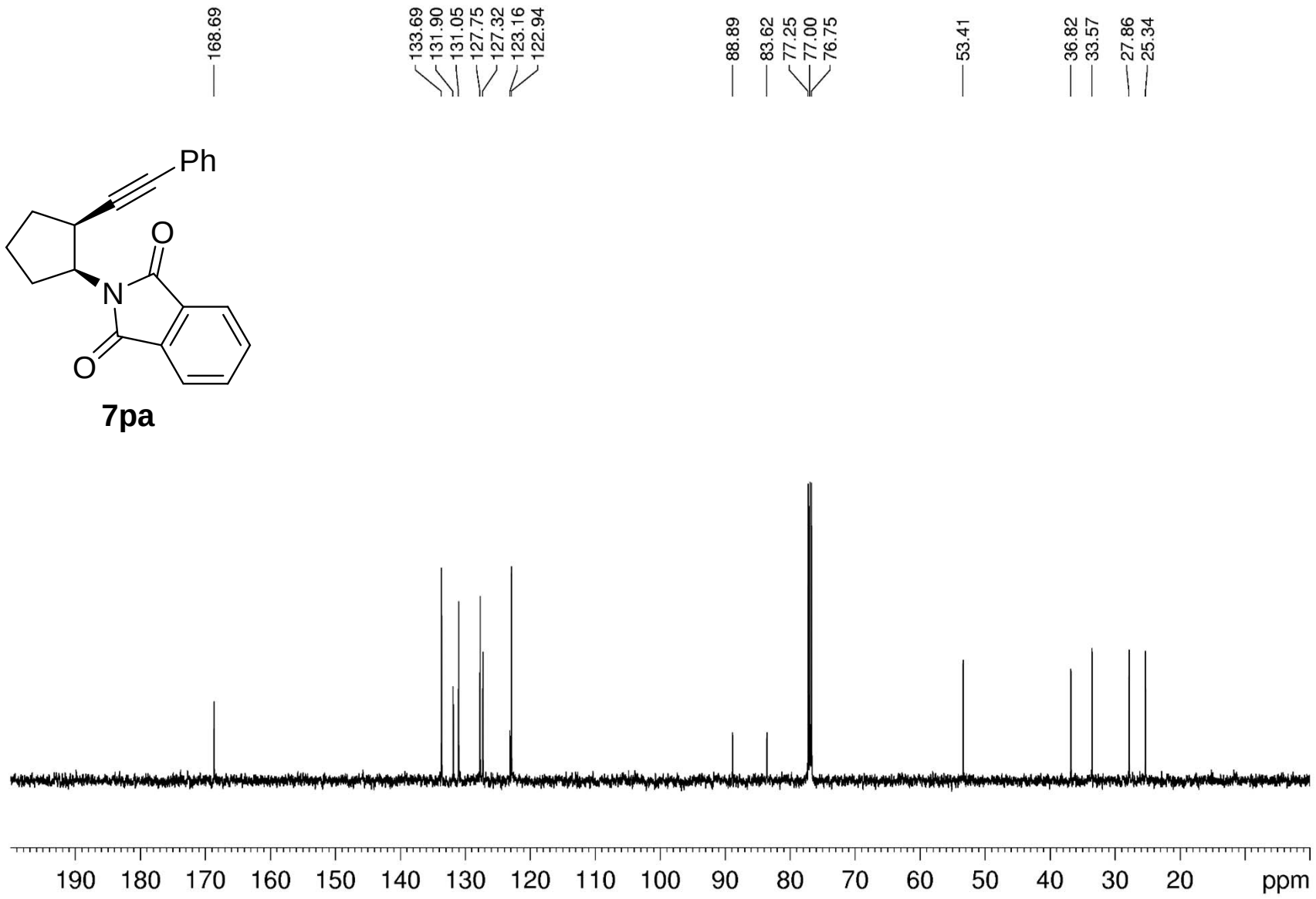


^{13}C NMR of compound **6hc** (75 MHz, $CDCl_3$)

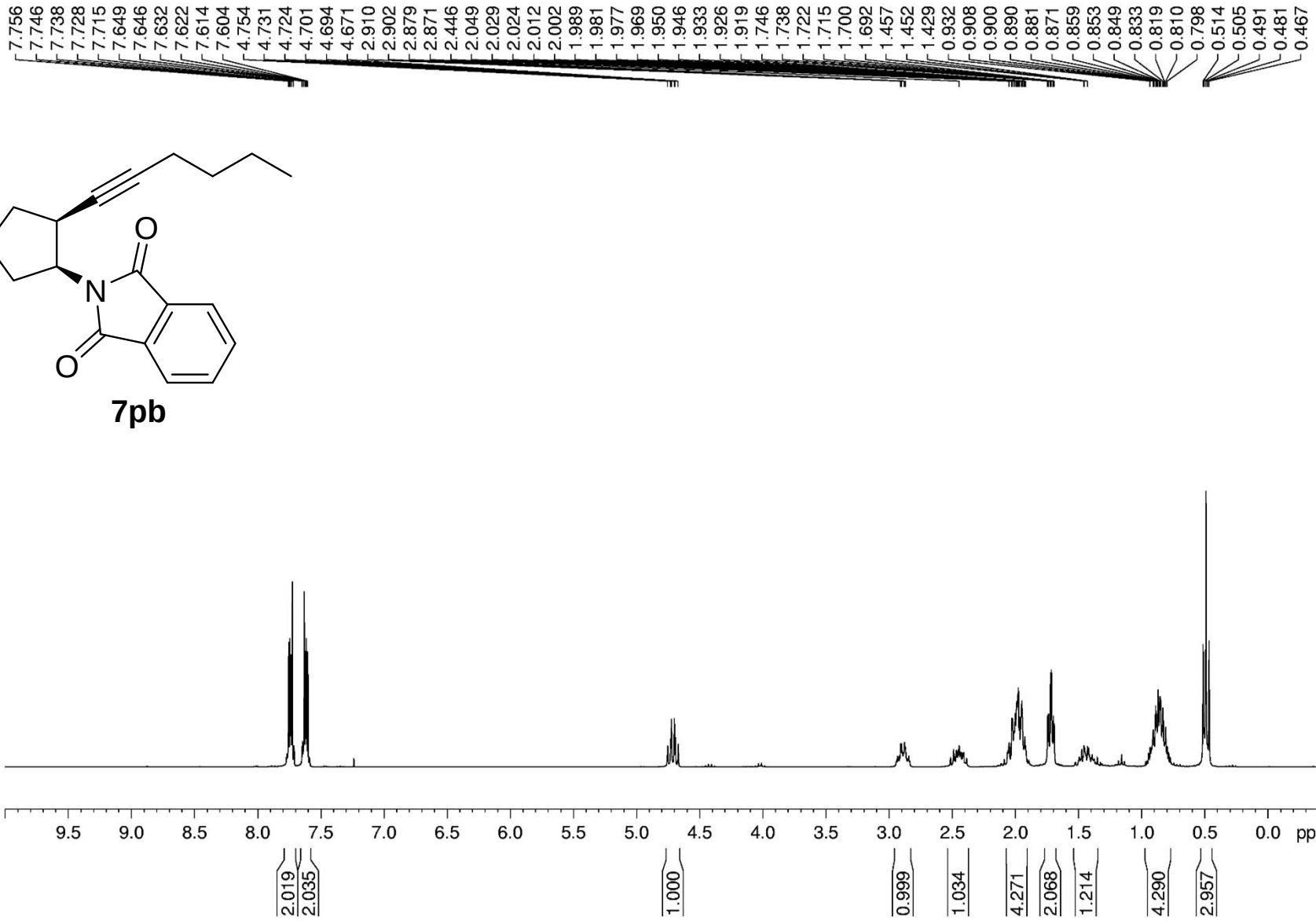


7pa

^{1}H NMR of compound **7pa** (500 MHz, $CDCl_3$)

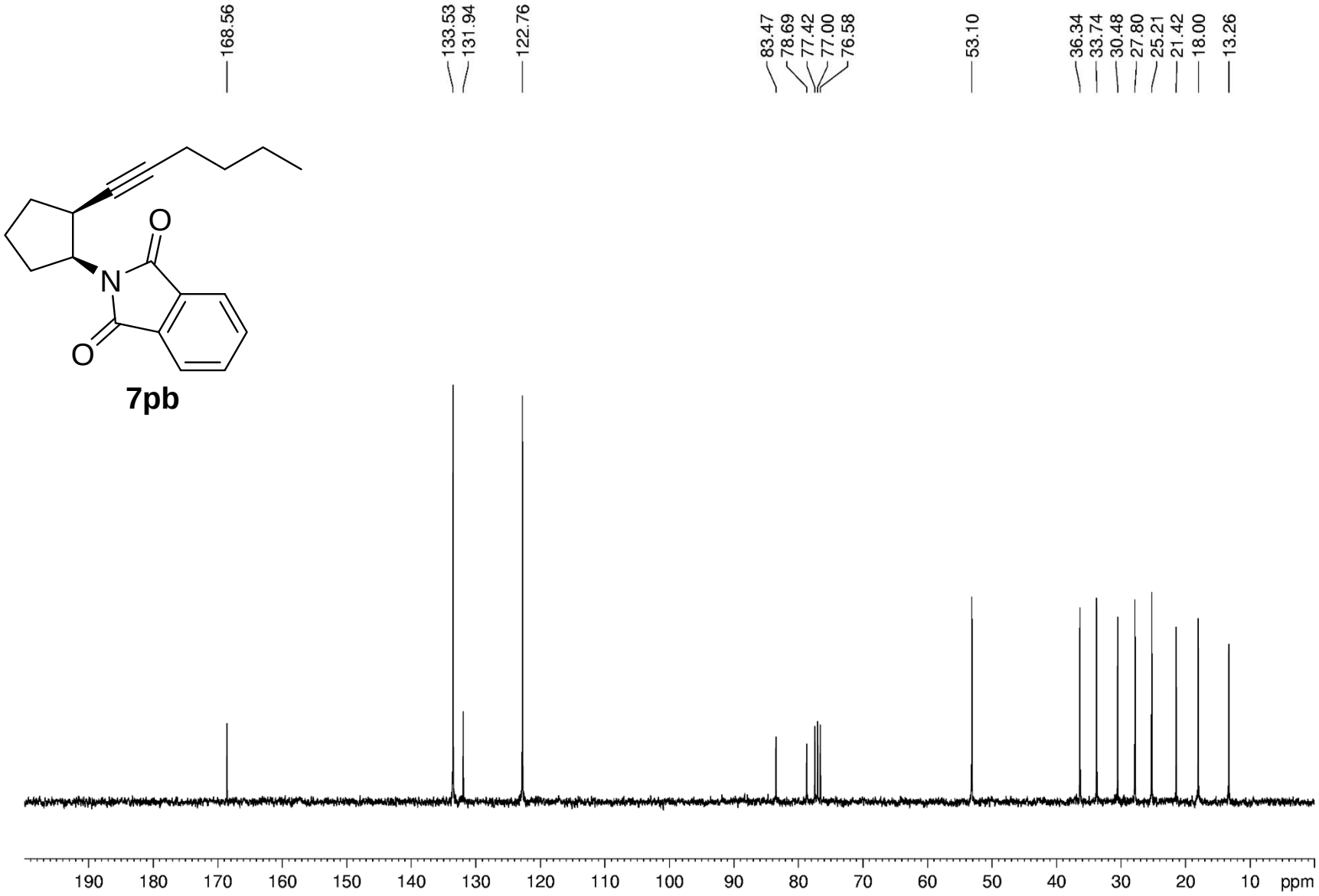


^{13}C NMR of compound **7pa** (125 MHz, $CDCl_3$)

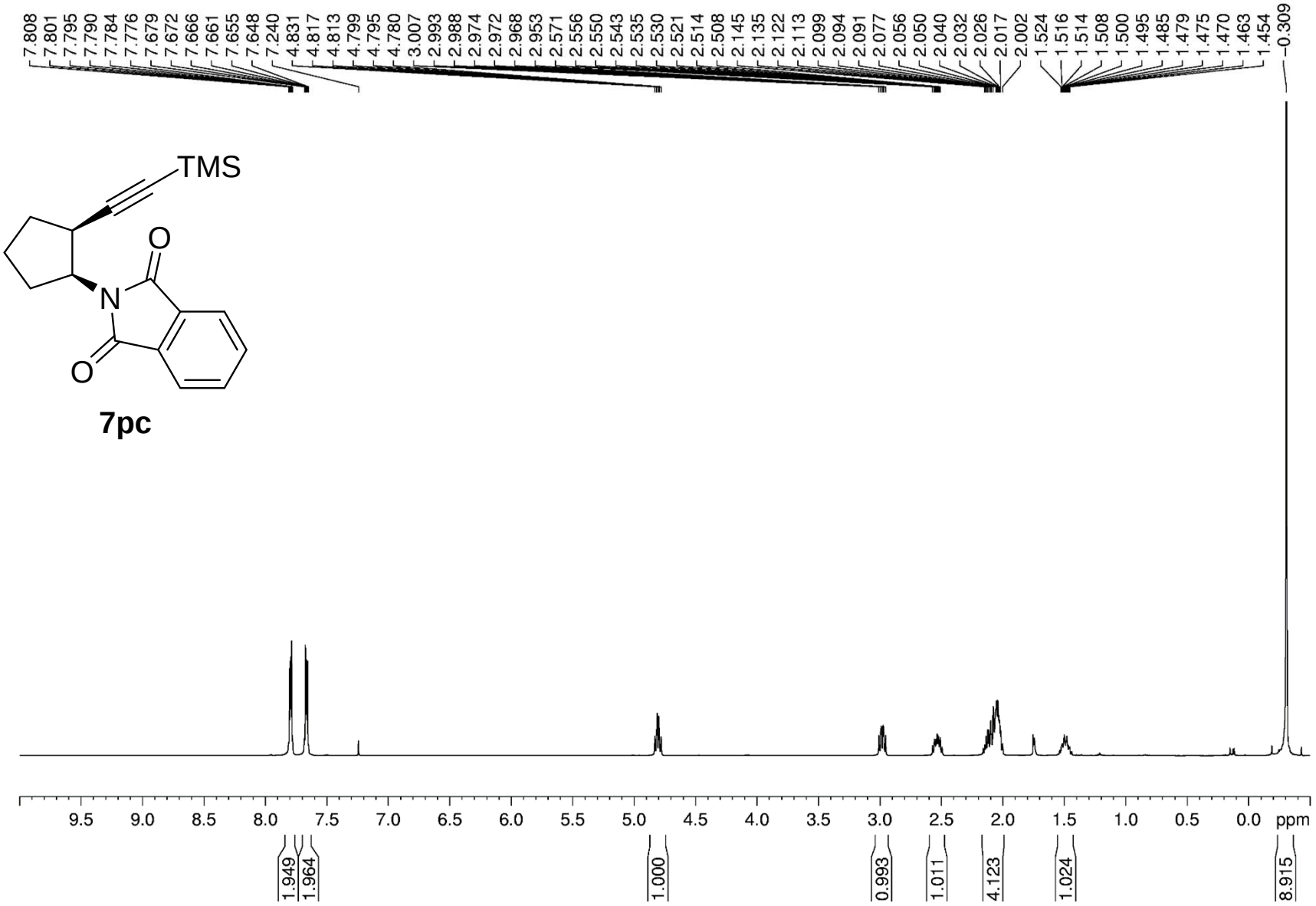


7pb

^{1}H NMR of compound **7pb** (300 MHz, $CDCl_3$)

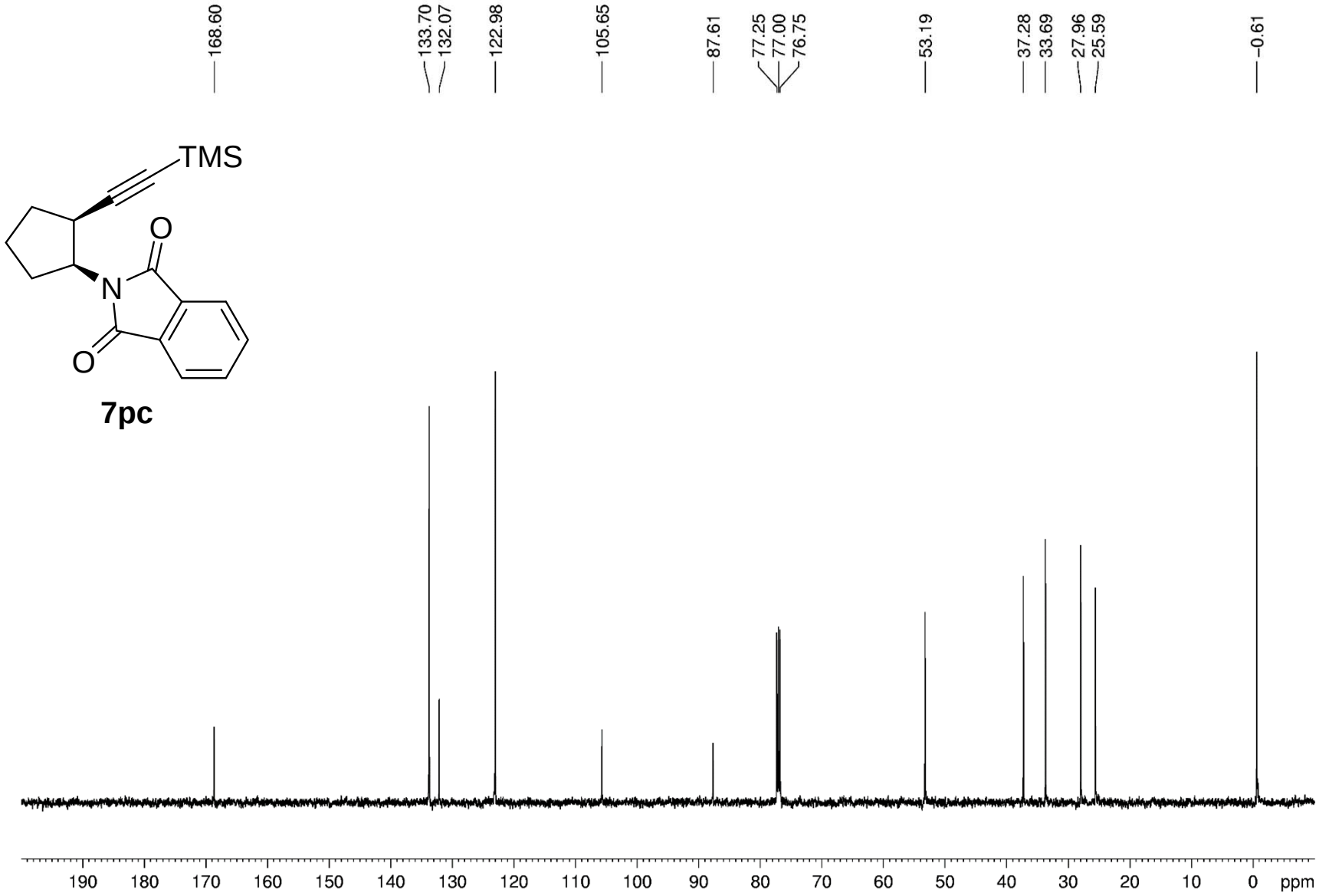


^{13}C NMR of compound **7pb** (75 MHz, $CDCl_3$)

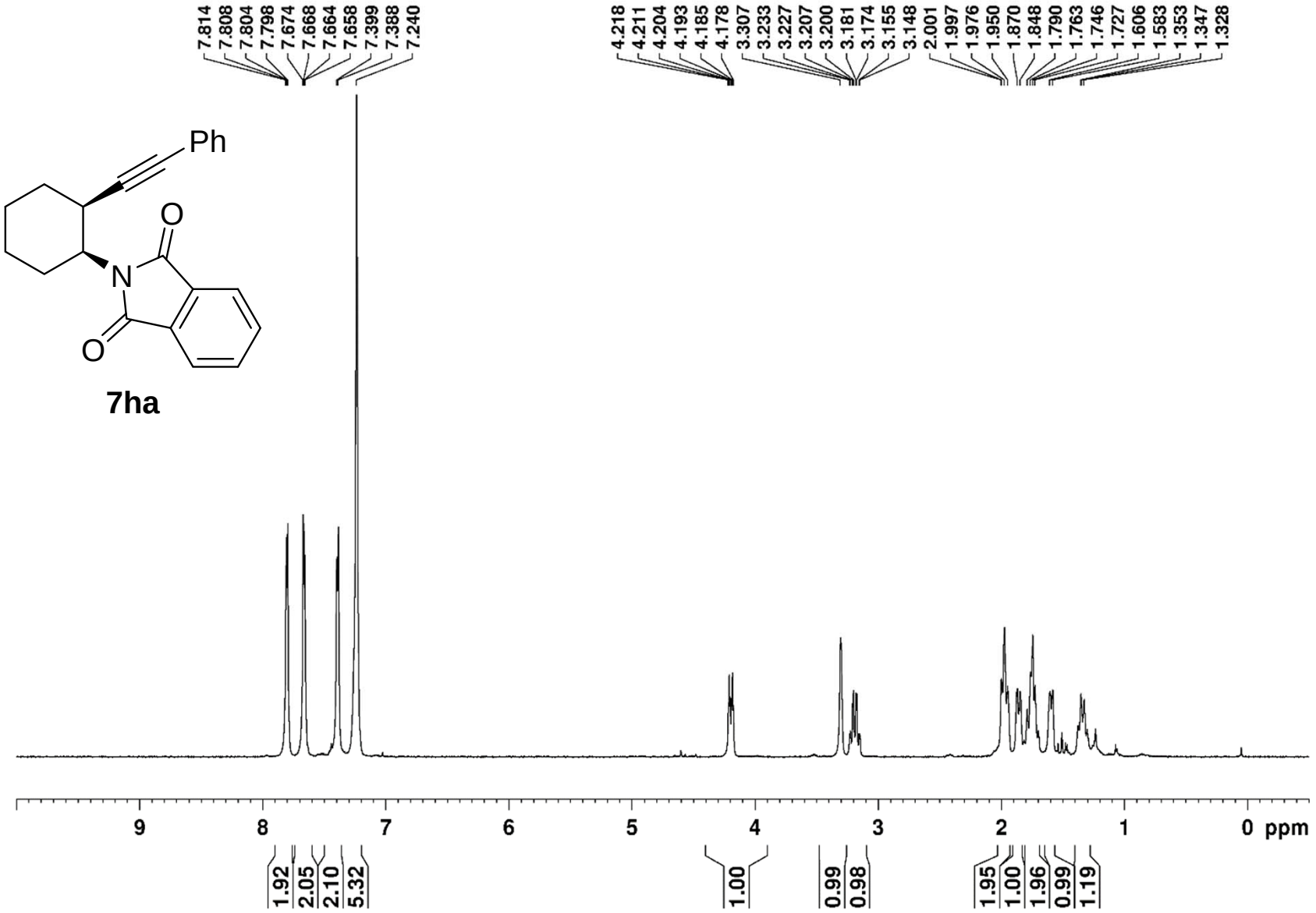


7pc

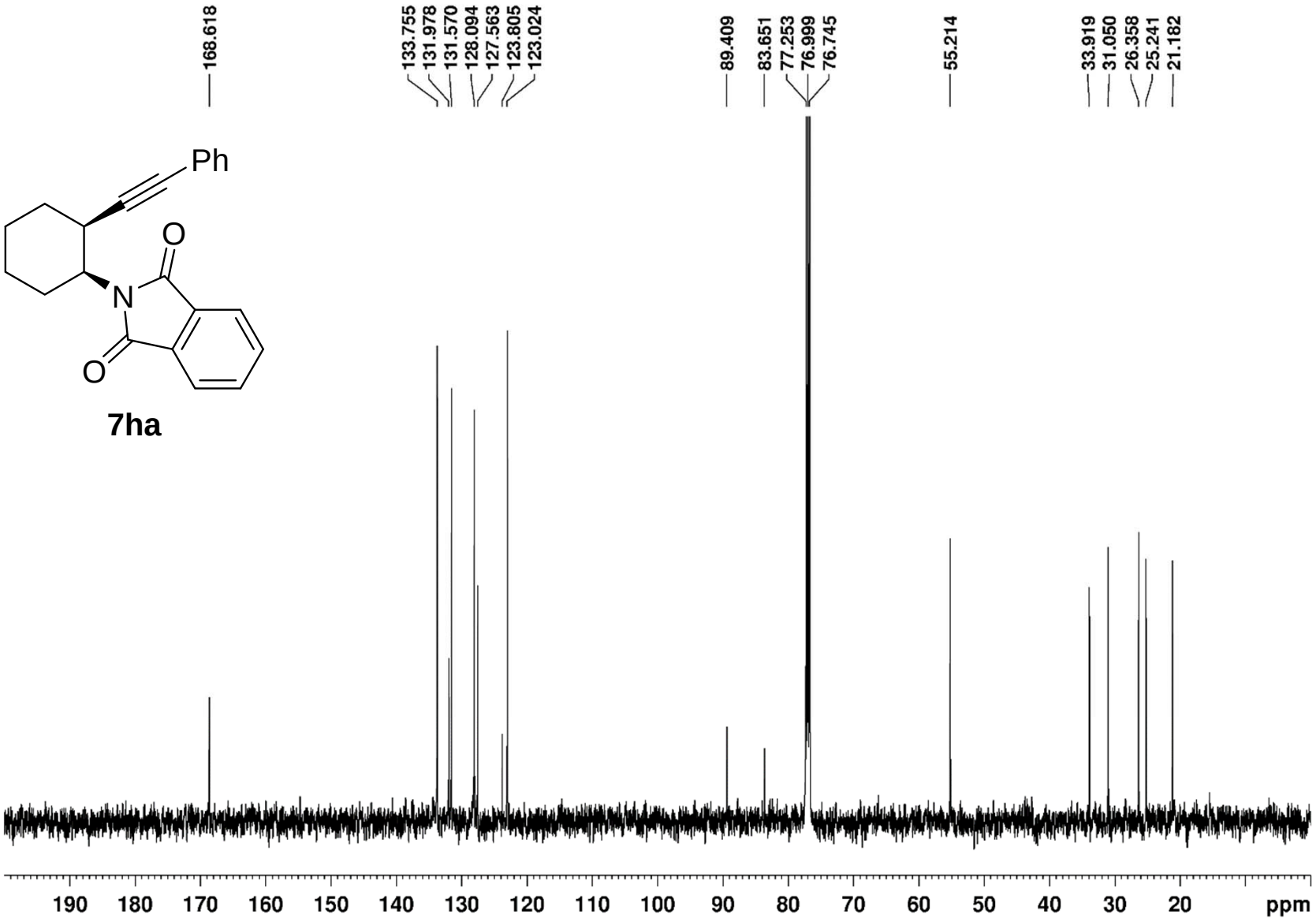
^{1}H NMR of compound **7pc** (500 MHz, $CDCl_3$)



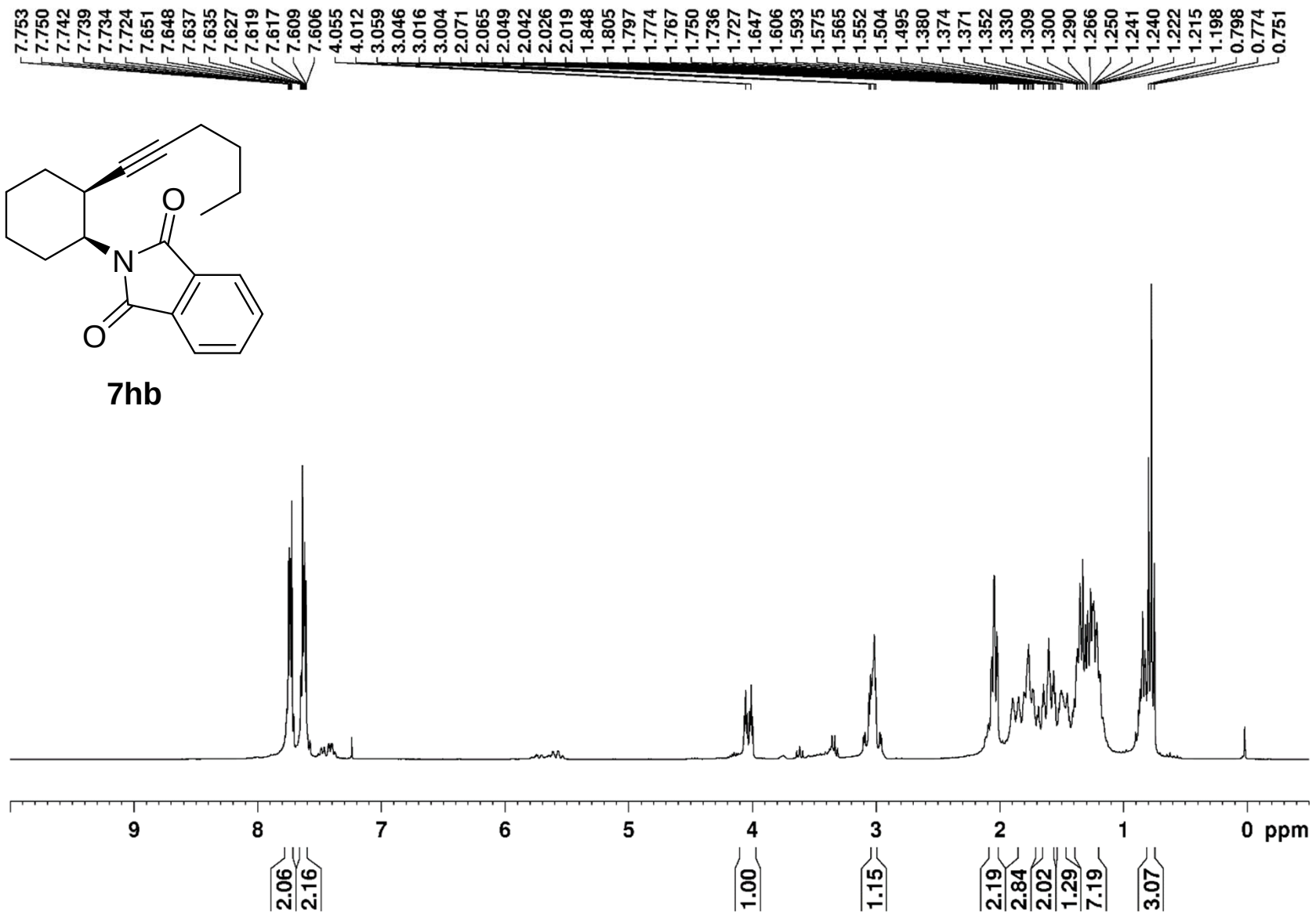
^{13}C NMR of compound **7pc** (125 MHz, $CDCl_3$)



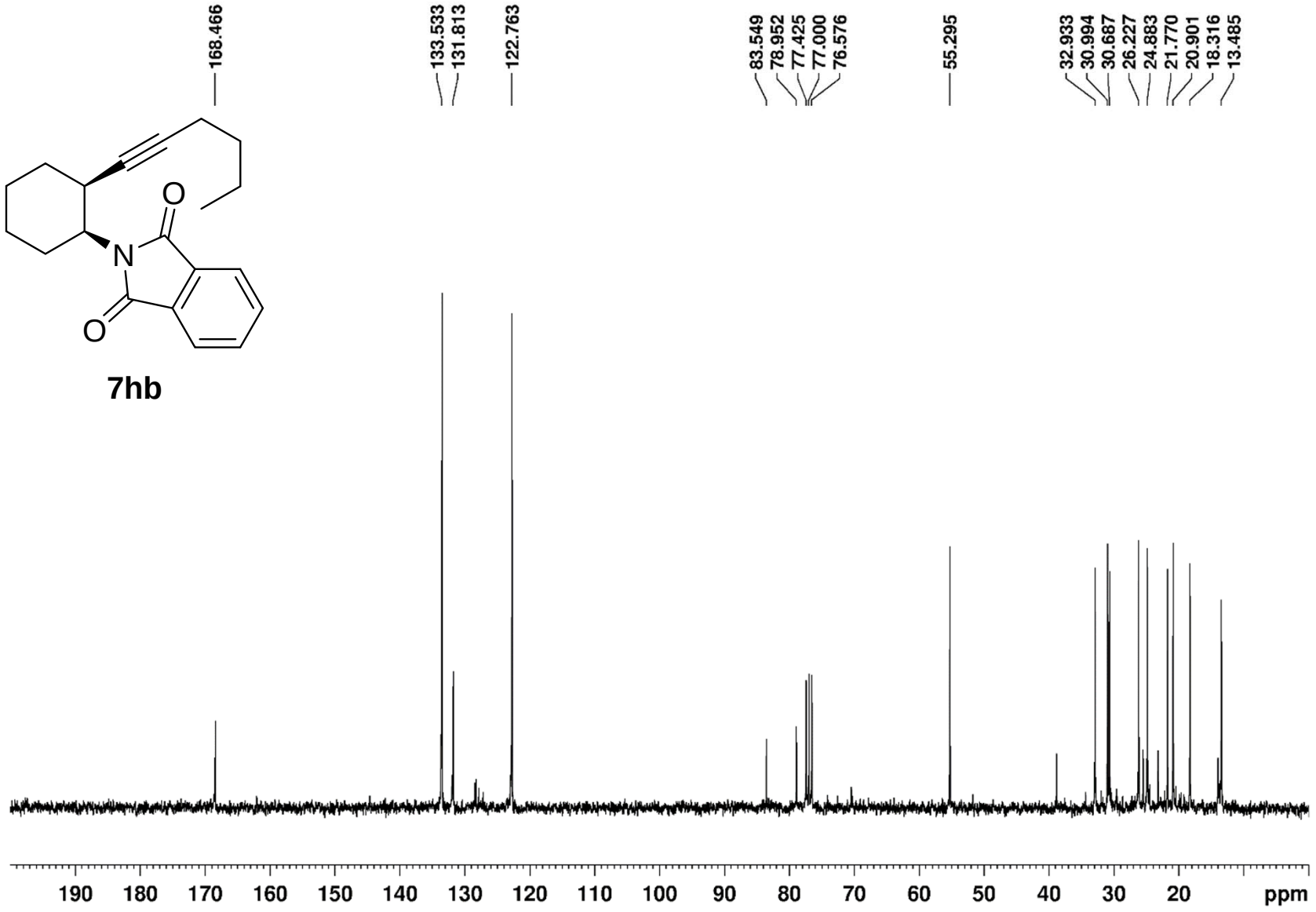
^{1}H NMR of compound **7ha** (500 MHz, $CDCl_3$)



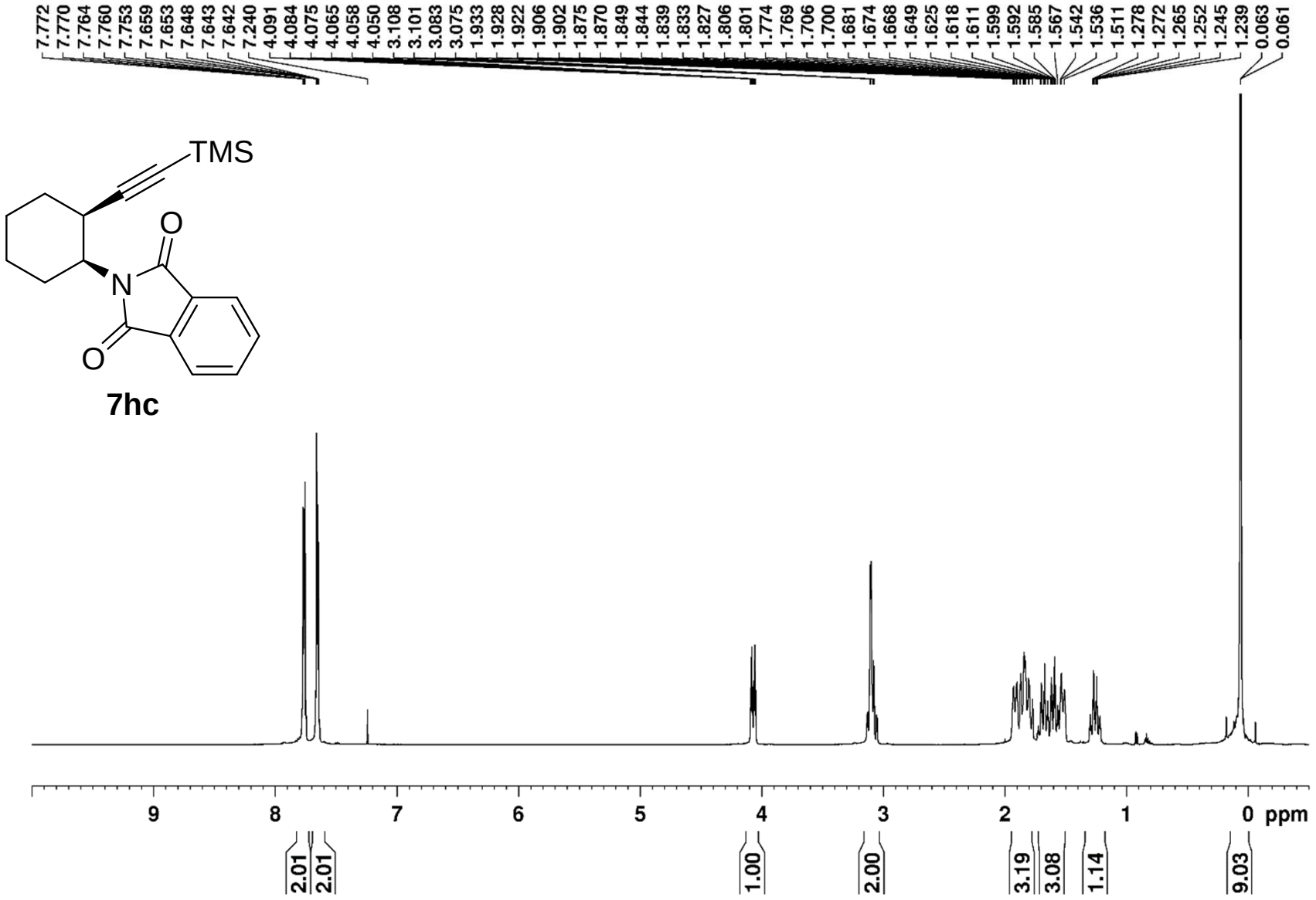
^{13}C NMR of compound **7ha** (125 MHz, $CDCl_3$)



^{1}H NMR of compound **7hb** (300 MHz, $CDCl_3$)

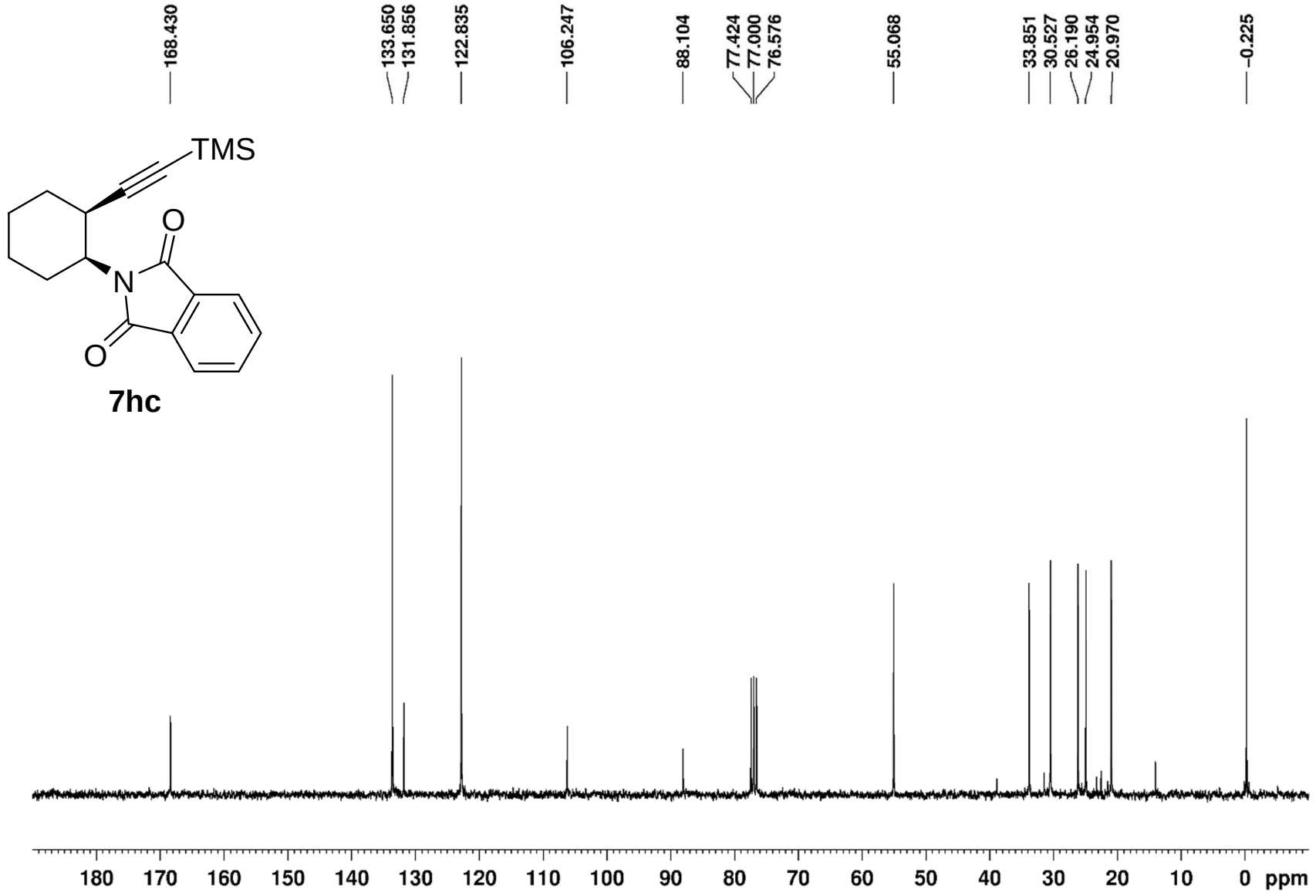


^{13}C NMR of compound **7hb** (75 MHz, $CDCl_3$)

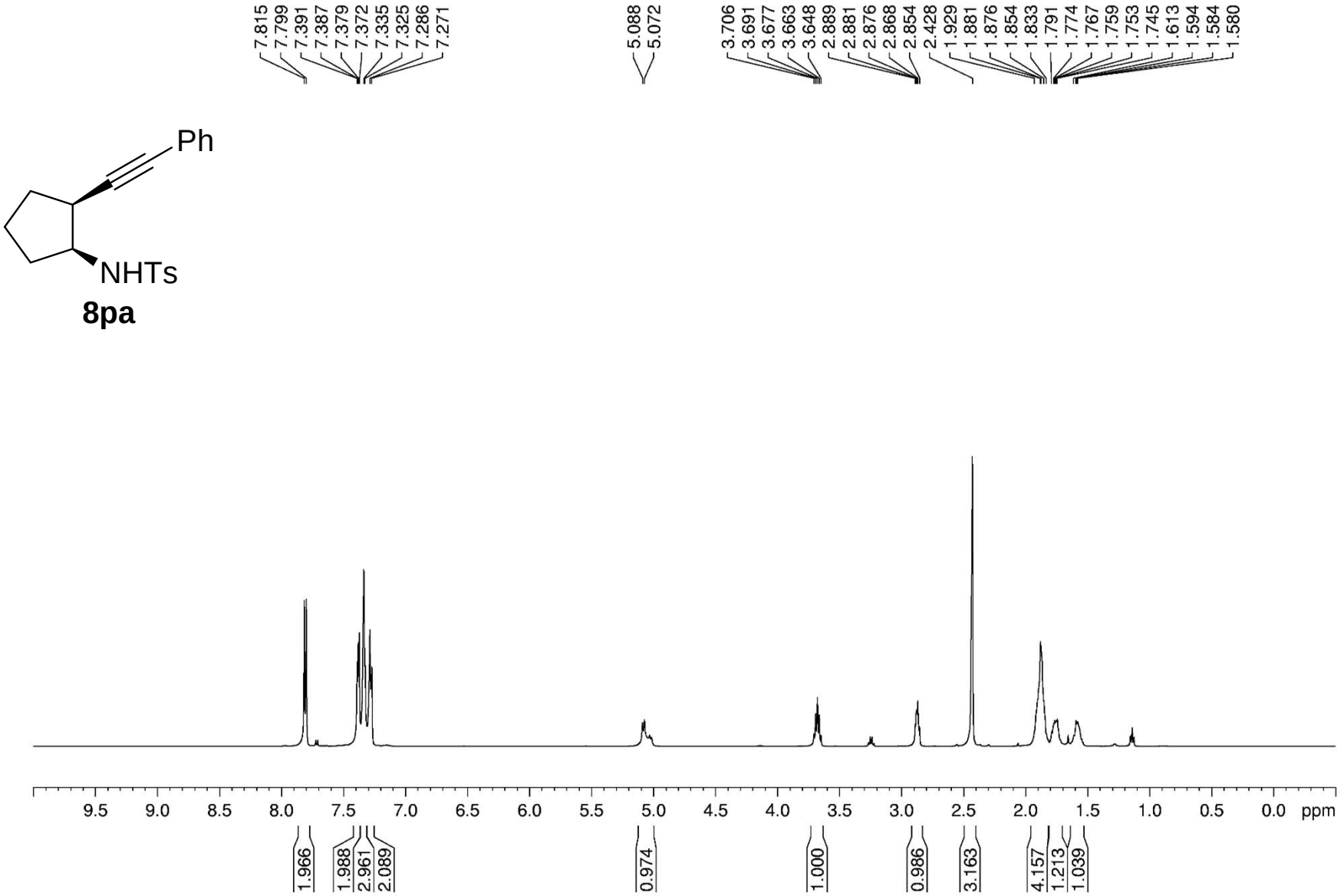


7hc

^{1}H NMR of compound **7hc** (500 MHz, $CDCl_3$)

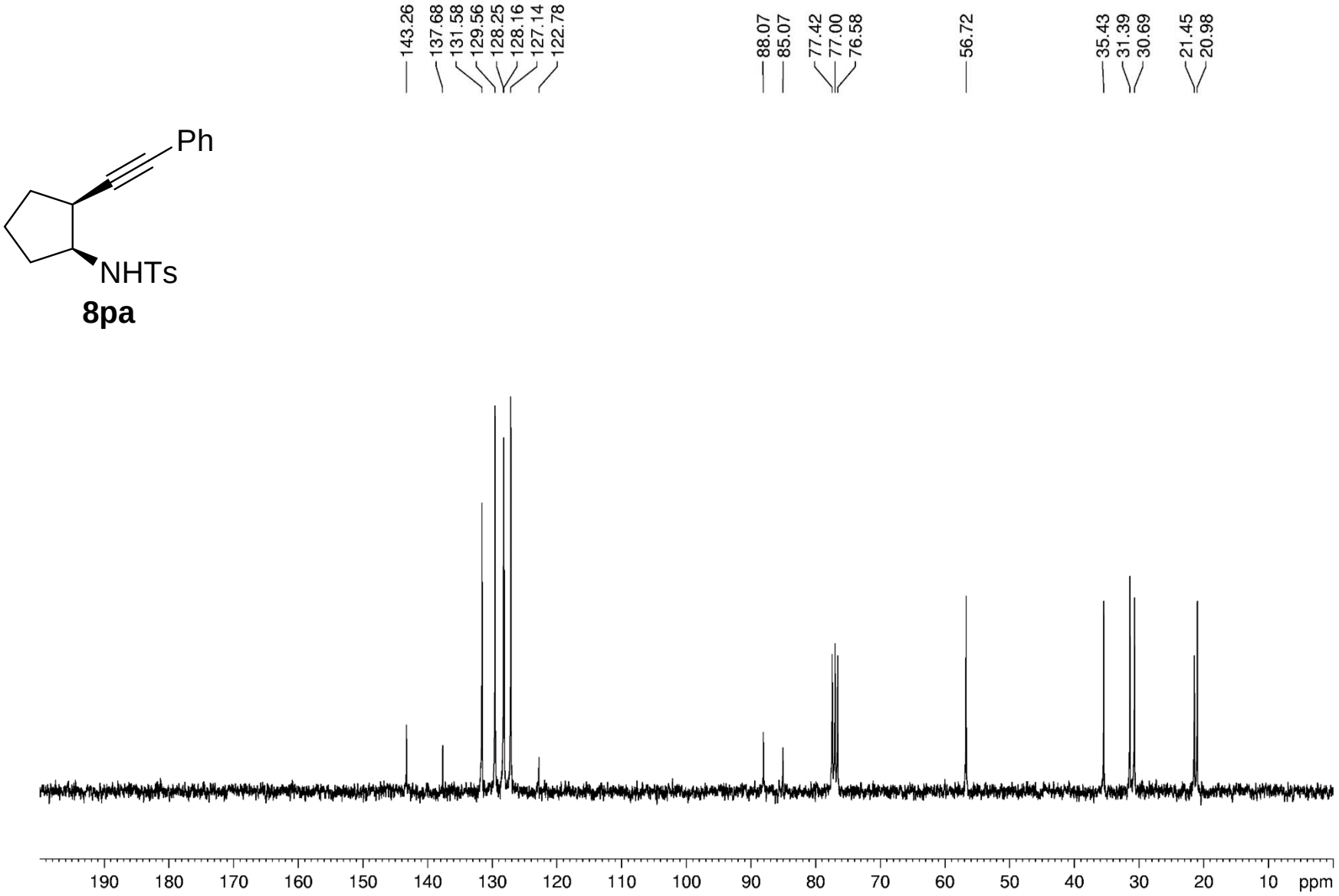


^{13}C NMR of compound **7hc** (75 MHz, $CDCl_3$)

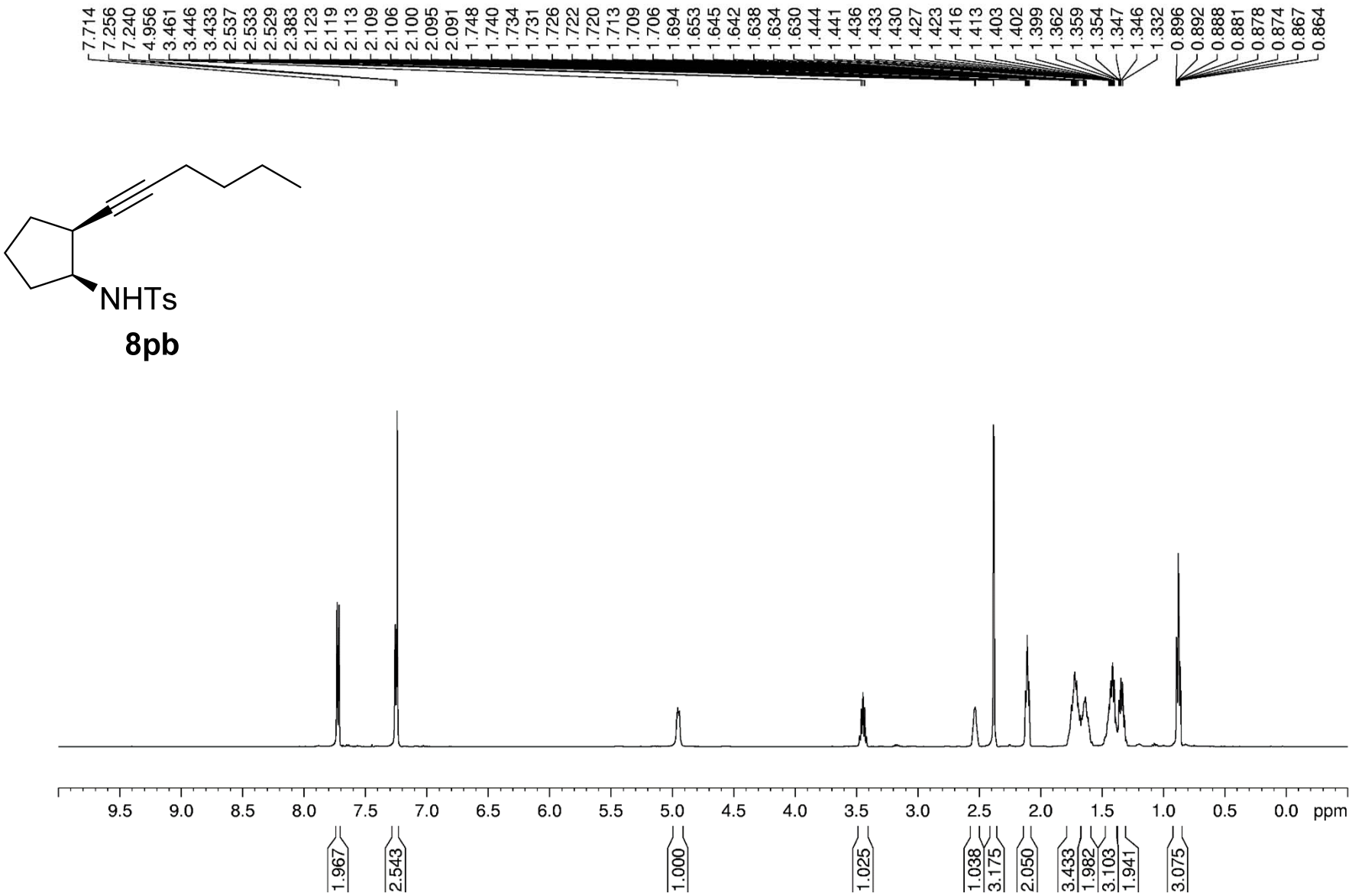


8pa

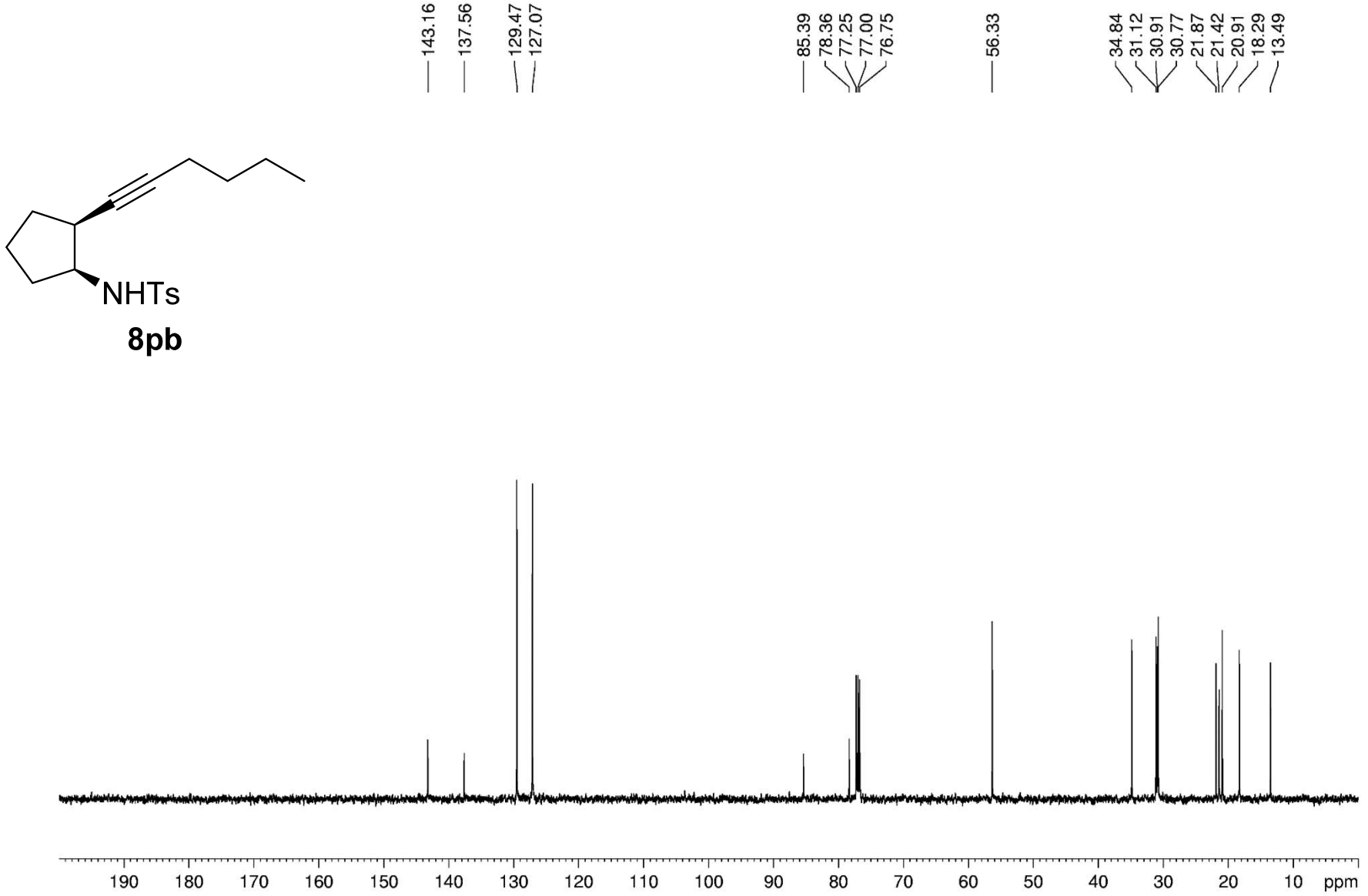
^{1}H NMR of compound **8pa** (500 MHz, $CDCl_3$)



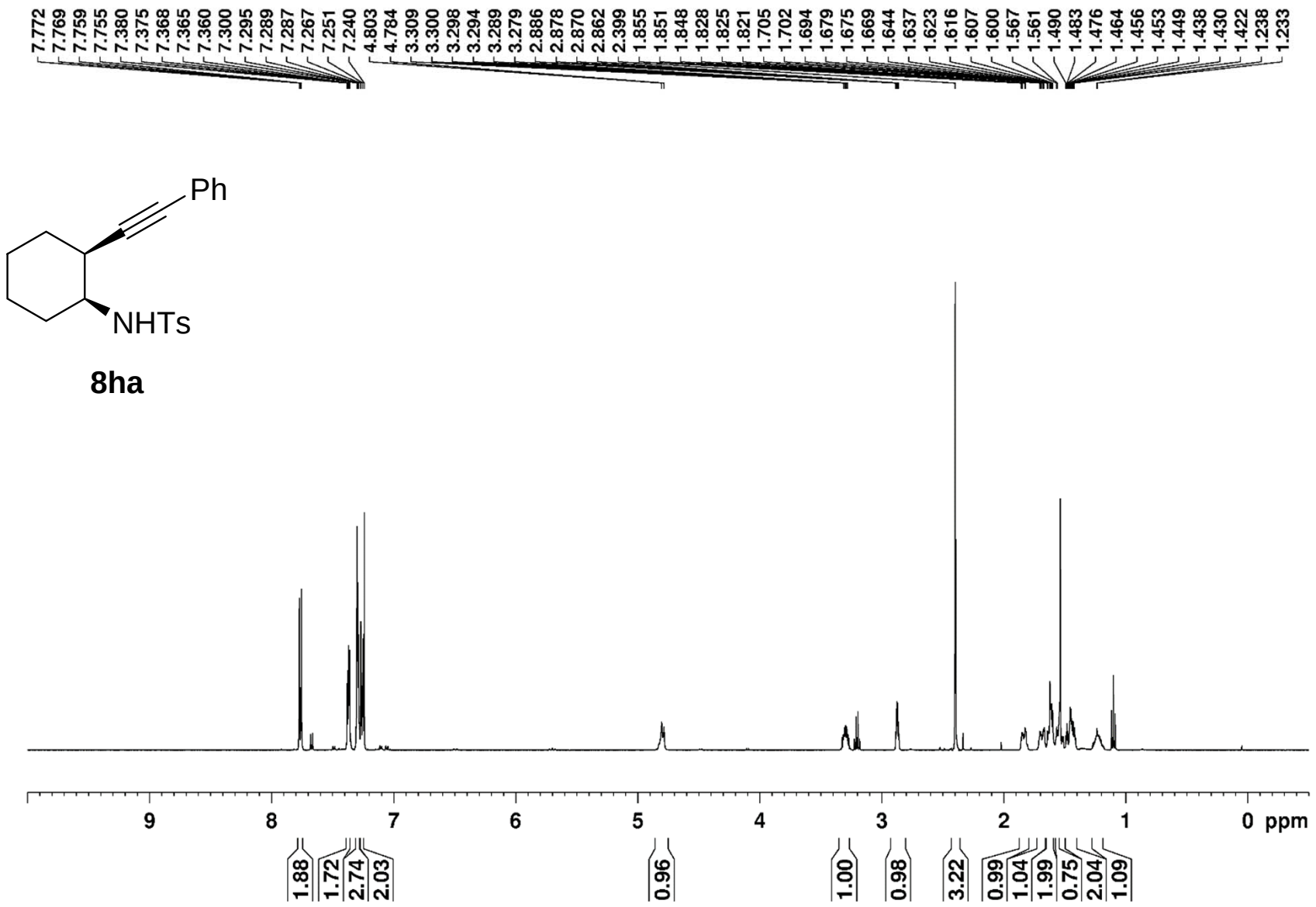
^{13}C NMR of compound **8pa** (125 MHz, $CDCl_3$)



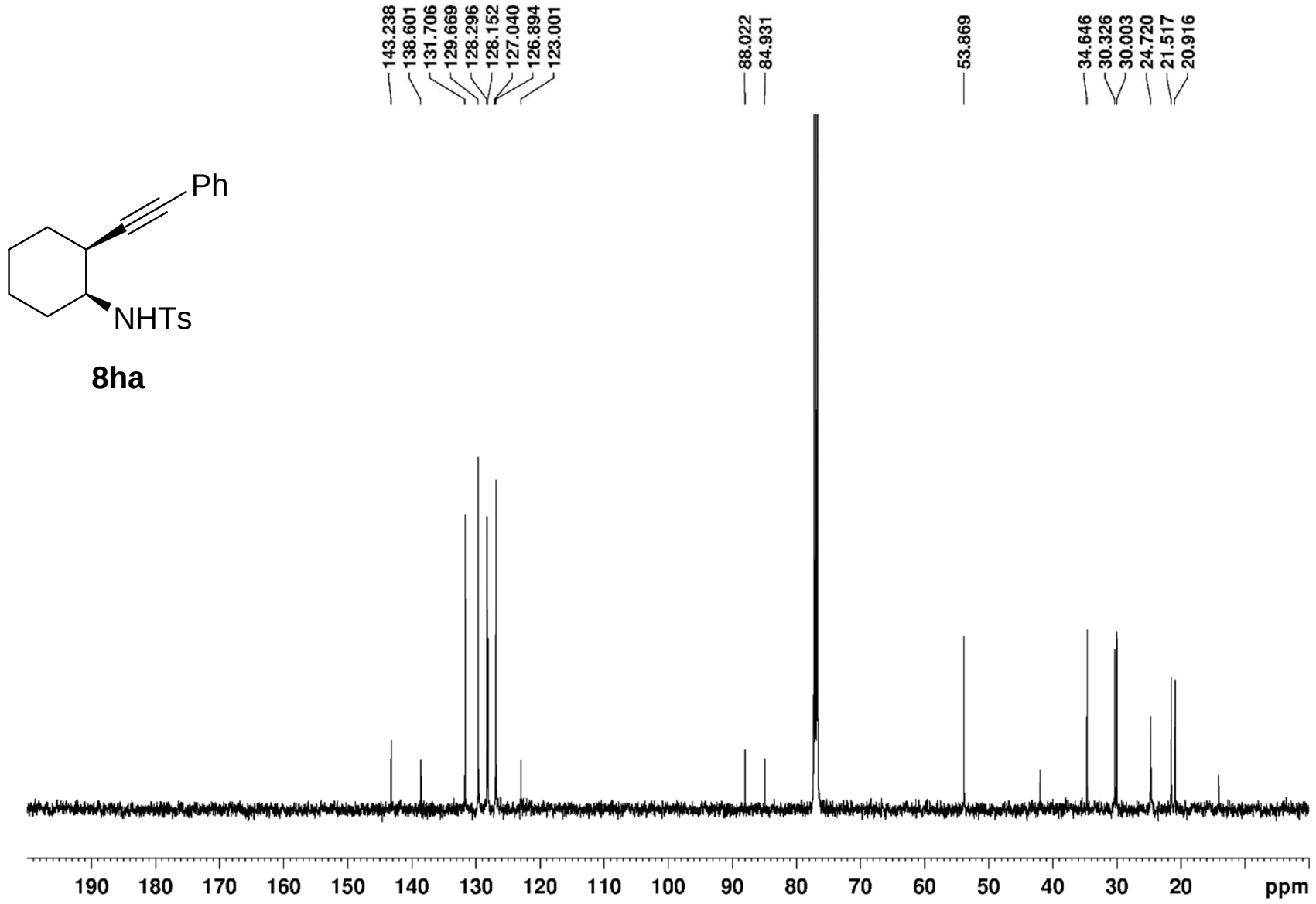
^{1}H NMR of compound **8pb** (500 MHz, $CDCl_3$)



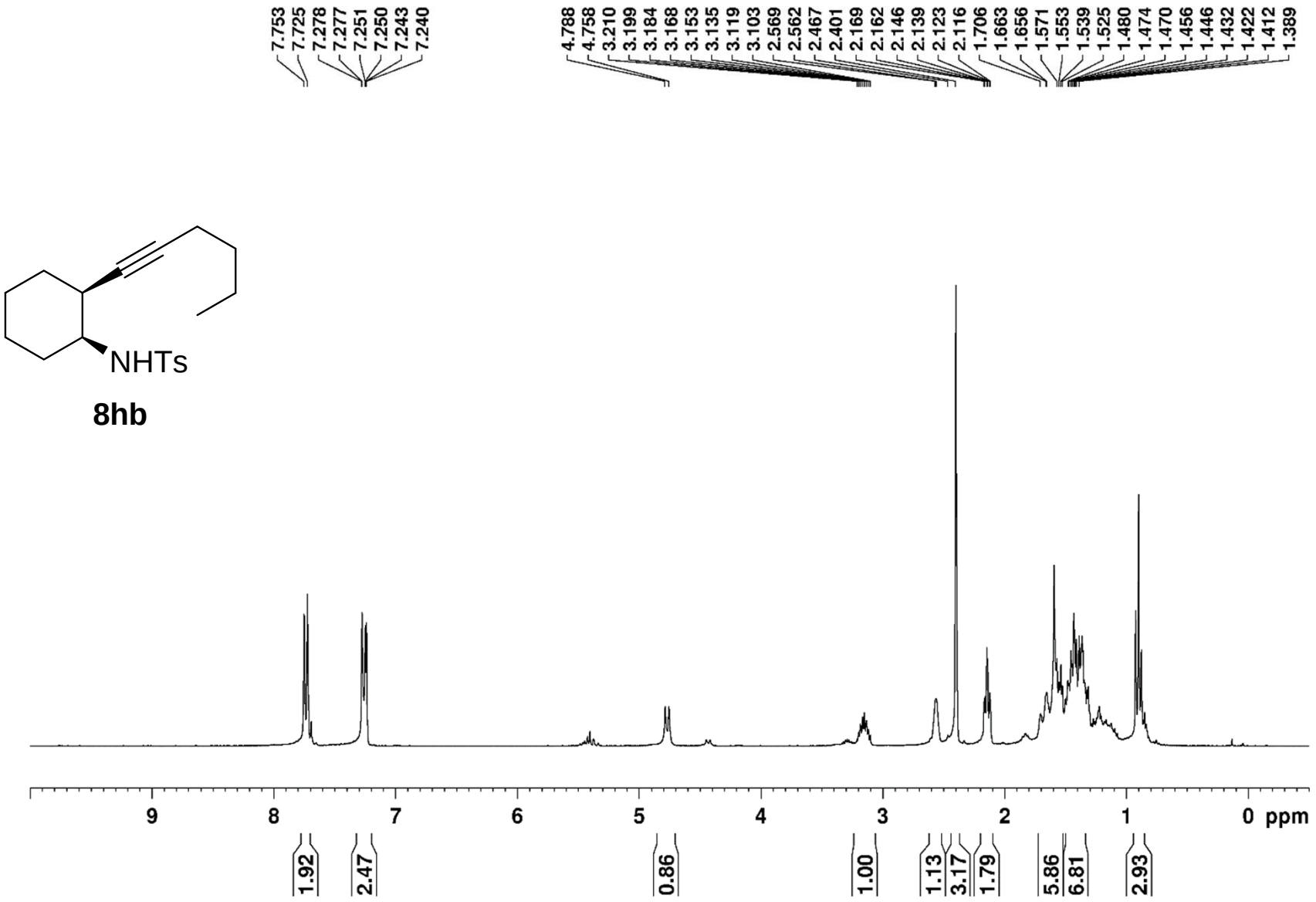
^{13}C NMR of compound **8pb** (125 MHz, $CDCl_3$)



^{1}H NMR of compound **8ha** (500 MHz, $CDCl_3$)

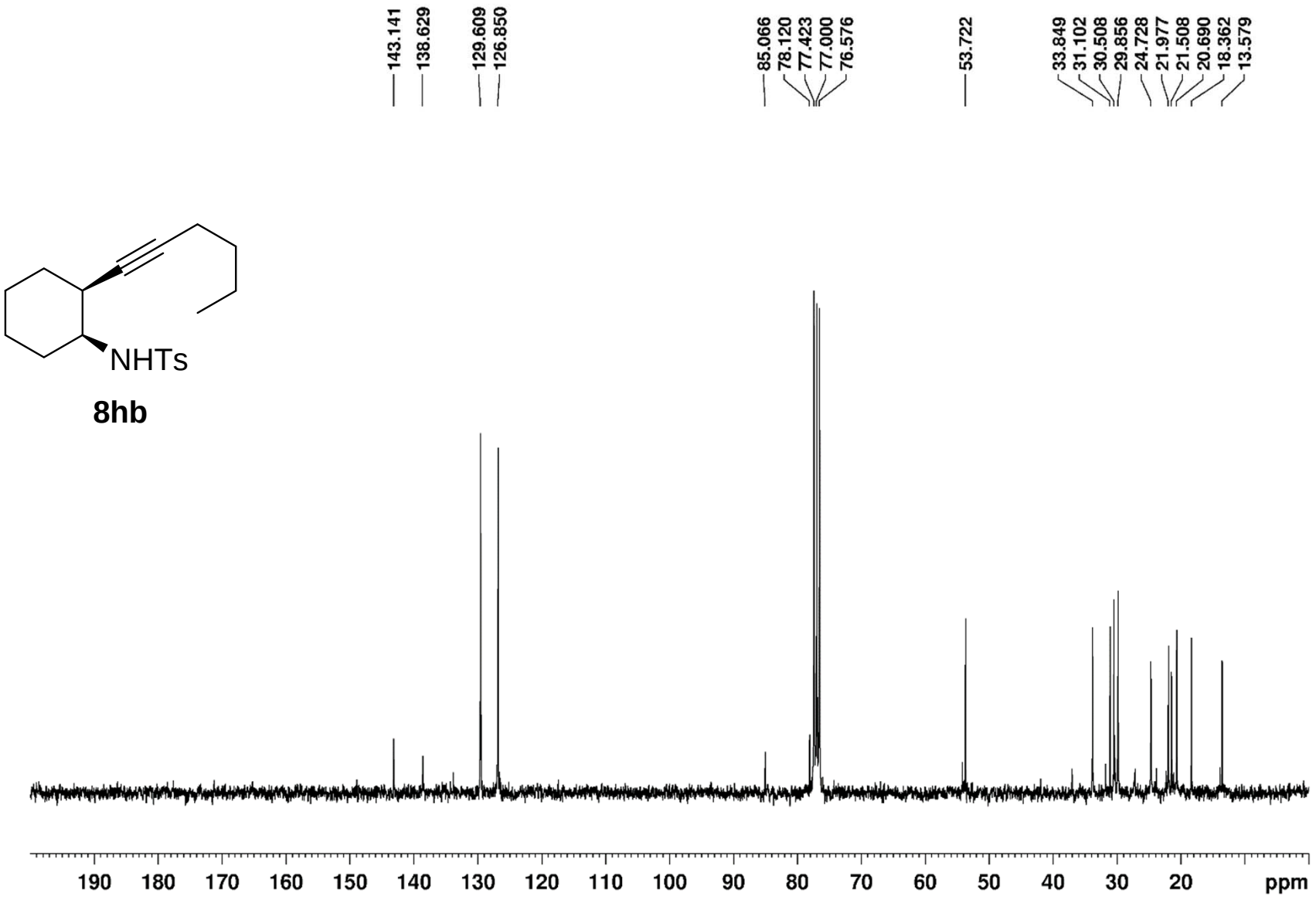


^{13}C NMR of compound **8ha** (125 MHz, $CDCl_3$)

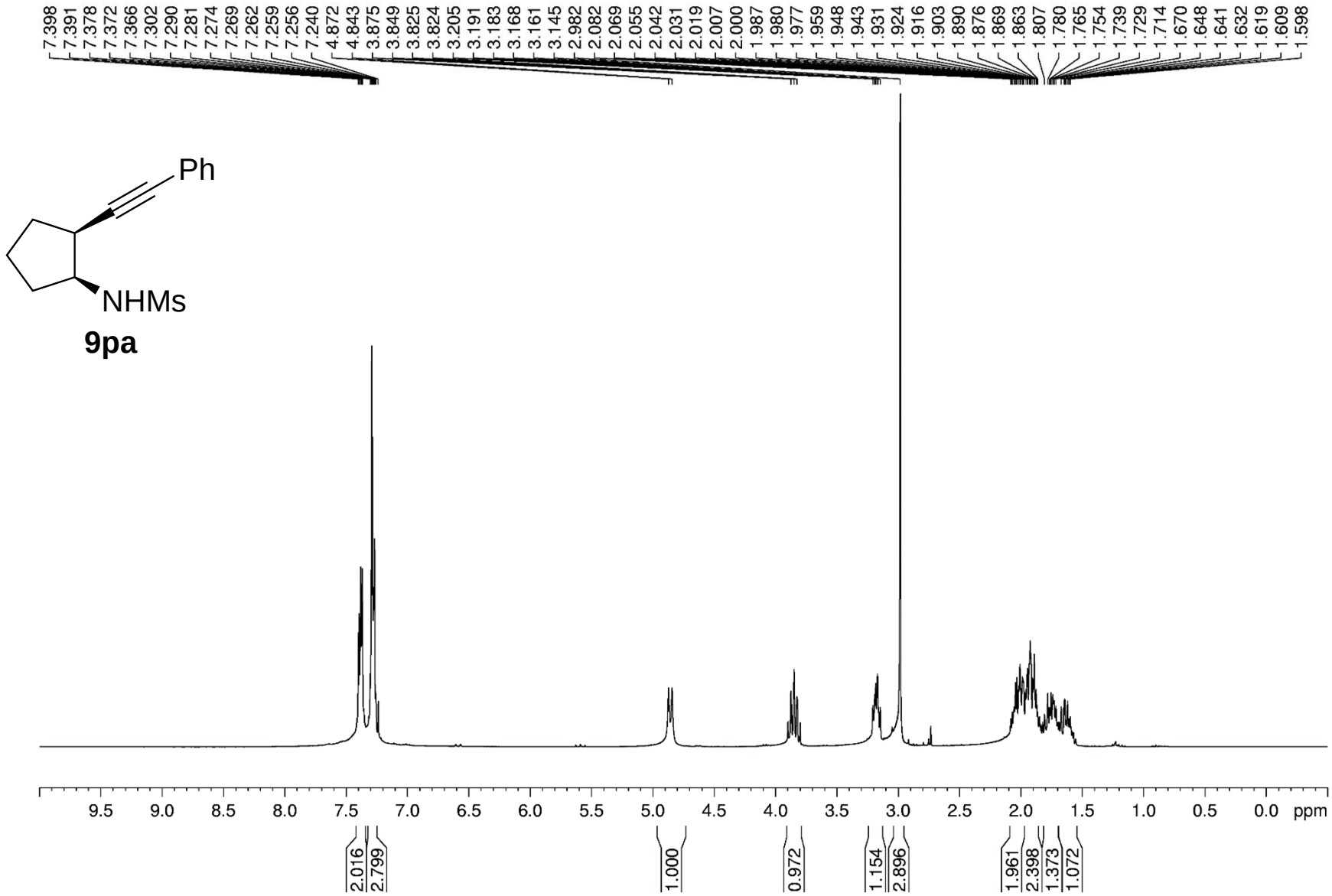


8hb

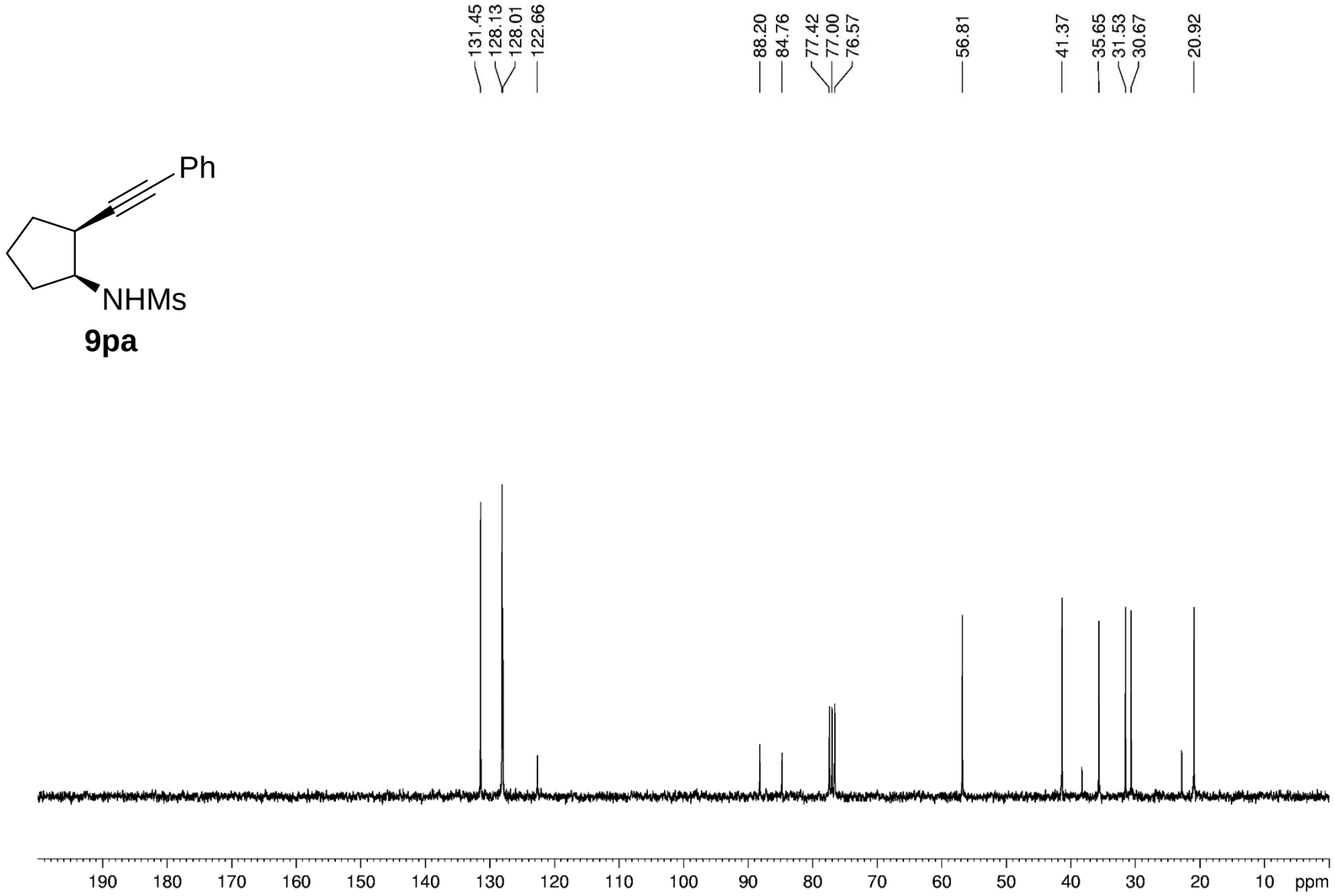
^{1}H NMR of compound **8hb** (300 MHz, $CDCl_3$)



^{13}C NMR of compound **8hb** (75 MHz, $CDCl_3$)

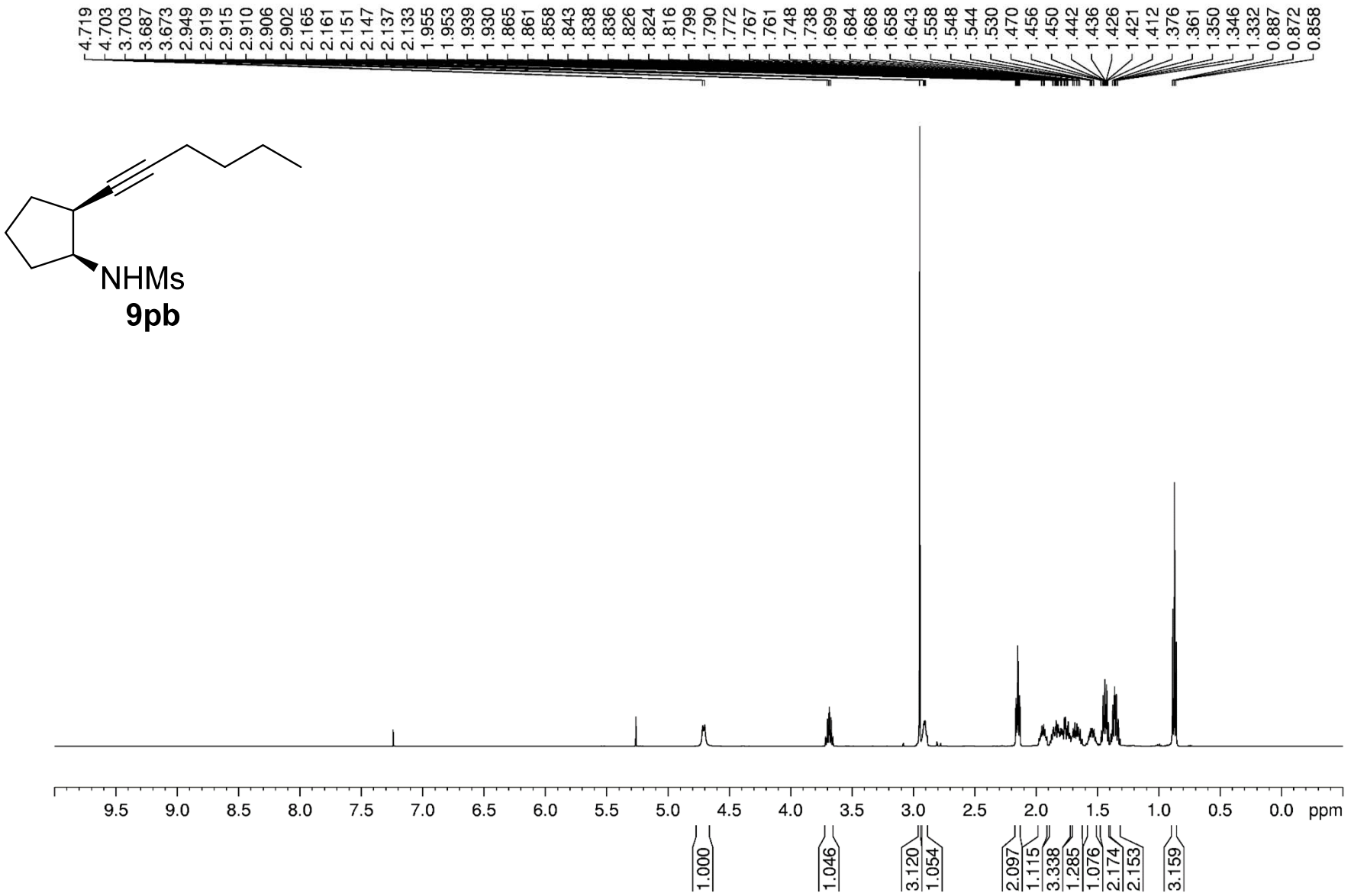


^{1}H NMR of compound **9pa** (300 MHz, $CDCl_3$)

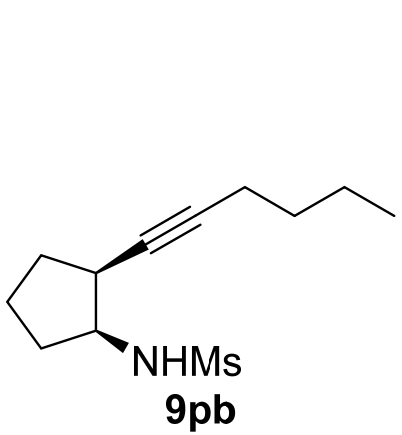


9pa

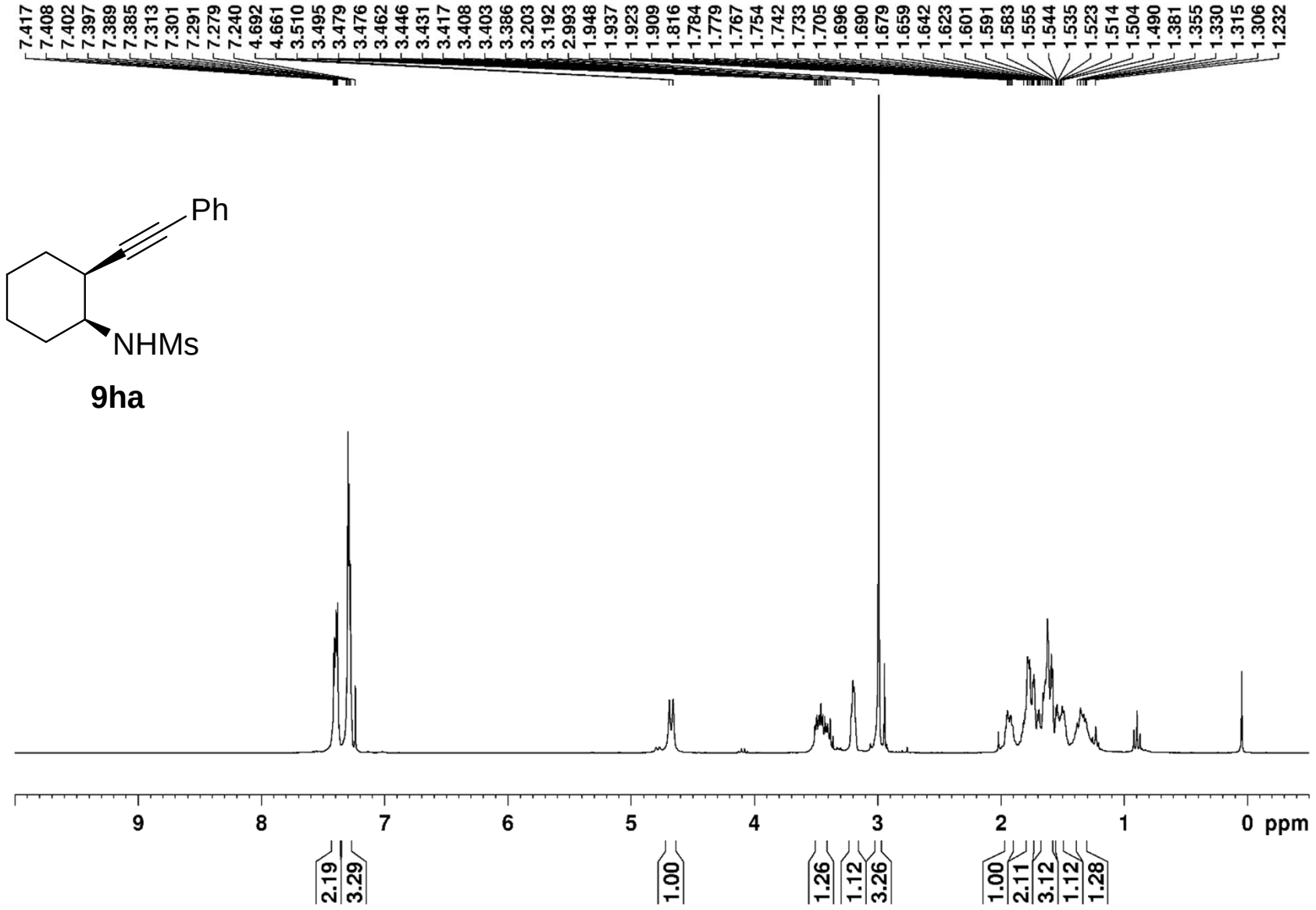
^{13}C NMR of compound **9pa** (75 MHz, $CDCl_3$)



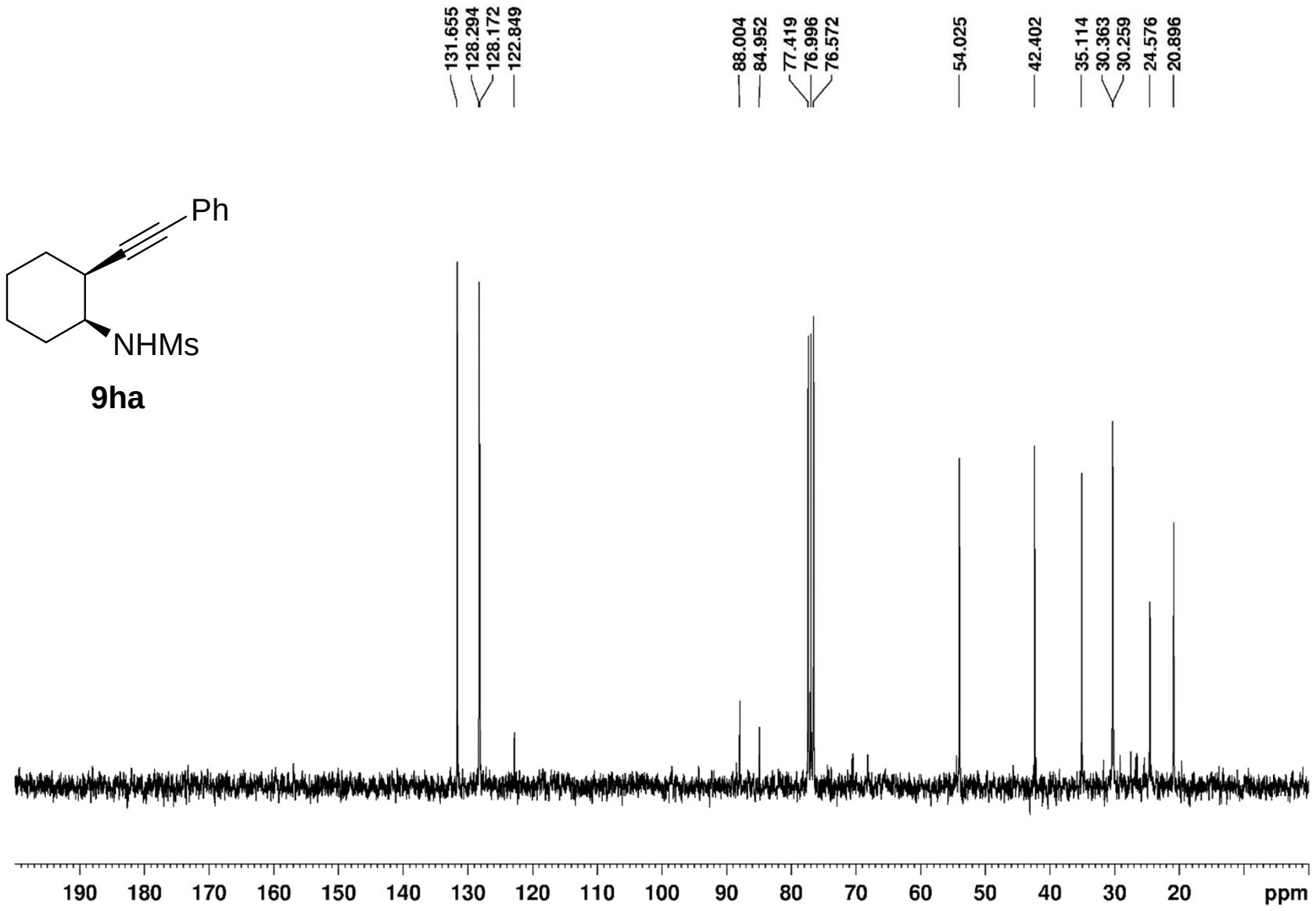
^{1}H NMR of compound **9pb** (500 MHz, $CDCl_3$)



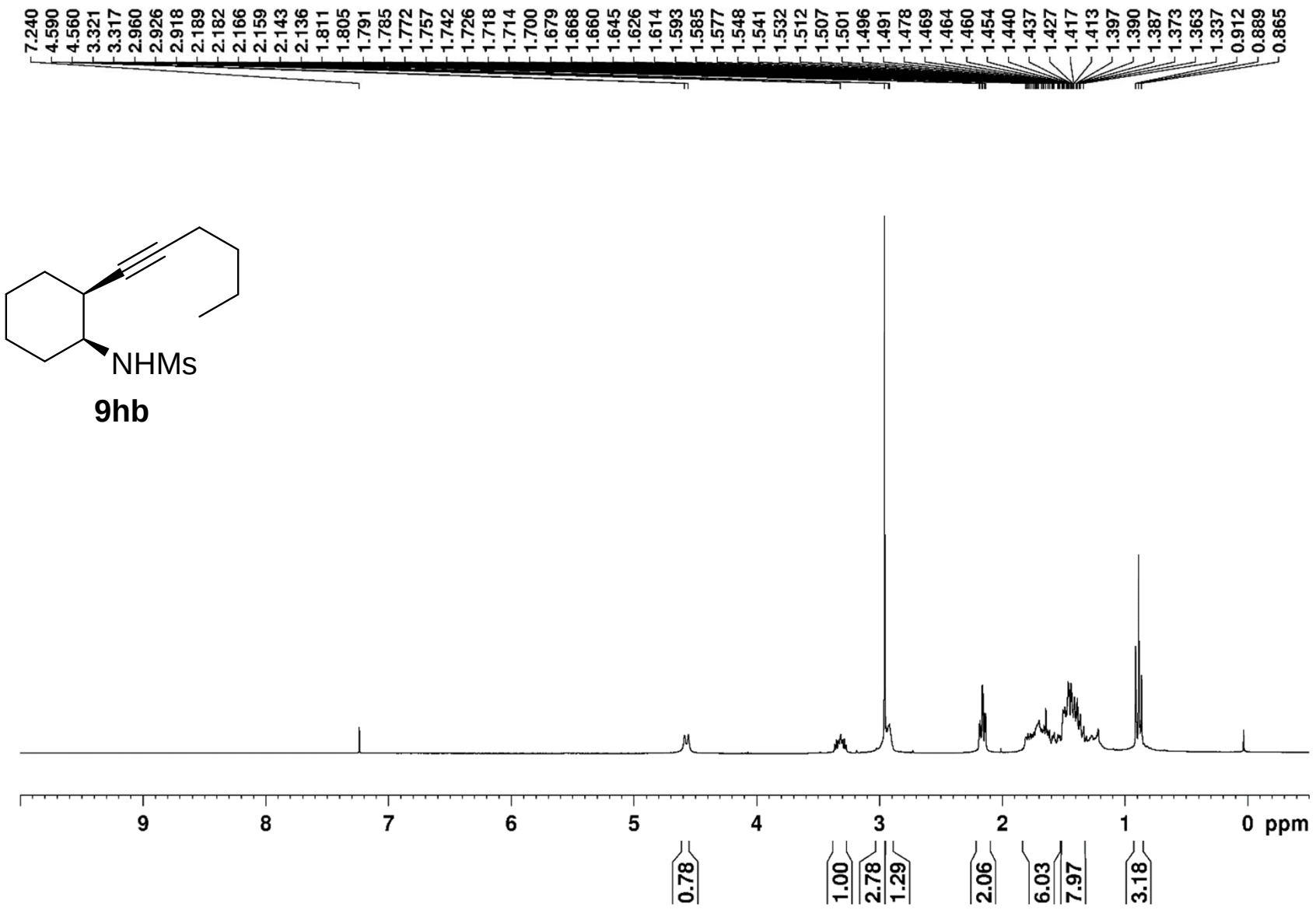
^{13}C NMR of compound **9pb** (125 MHz, $CDCl_3$)



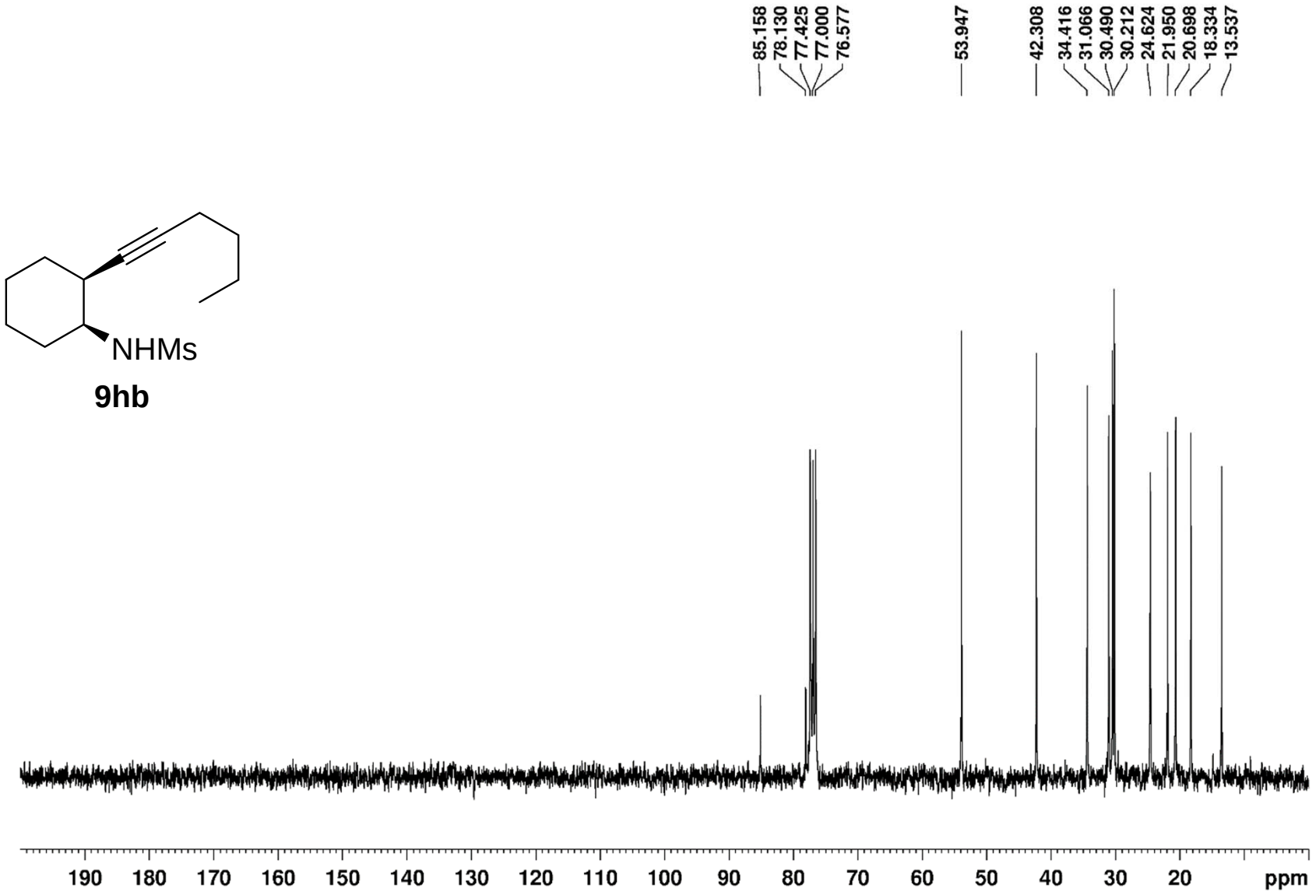
^{1}H NMR of compound **9ha** (300 MHz, $CDCl_3$)



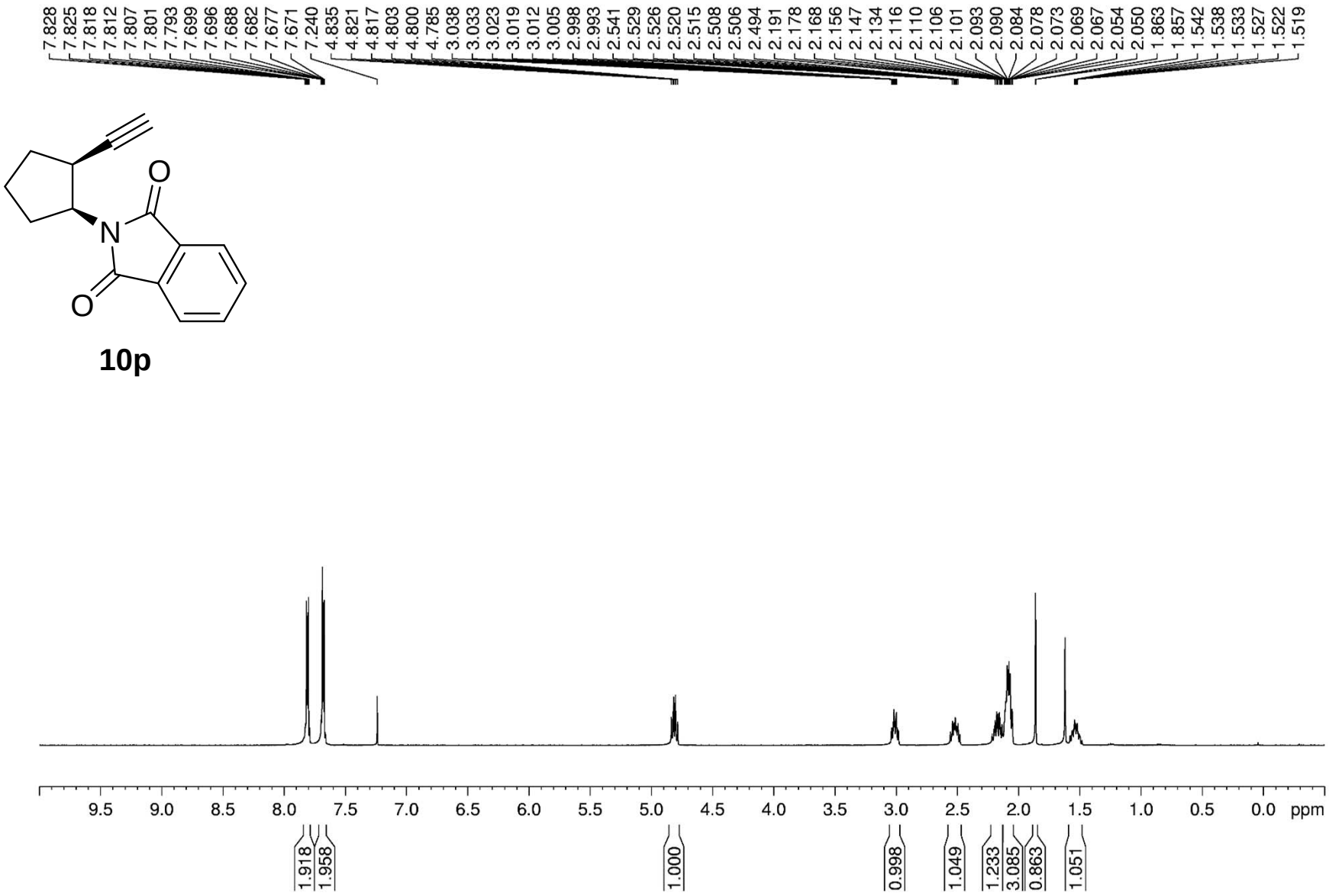
^{13}C NMR of compound **9ha** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **9hb** (300 MHz, $CDCl_3$)

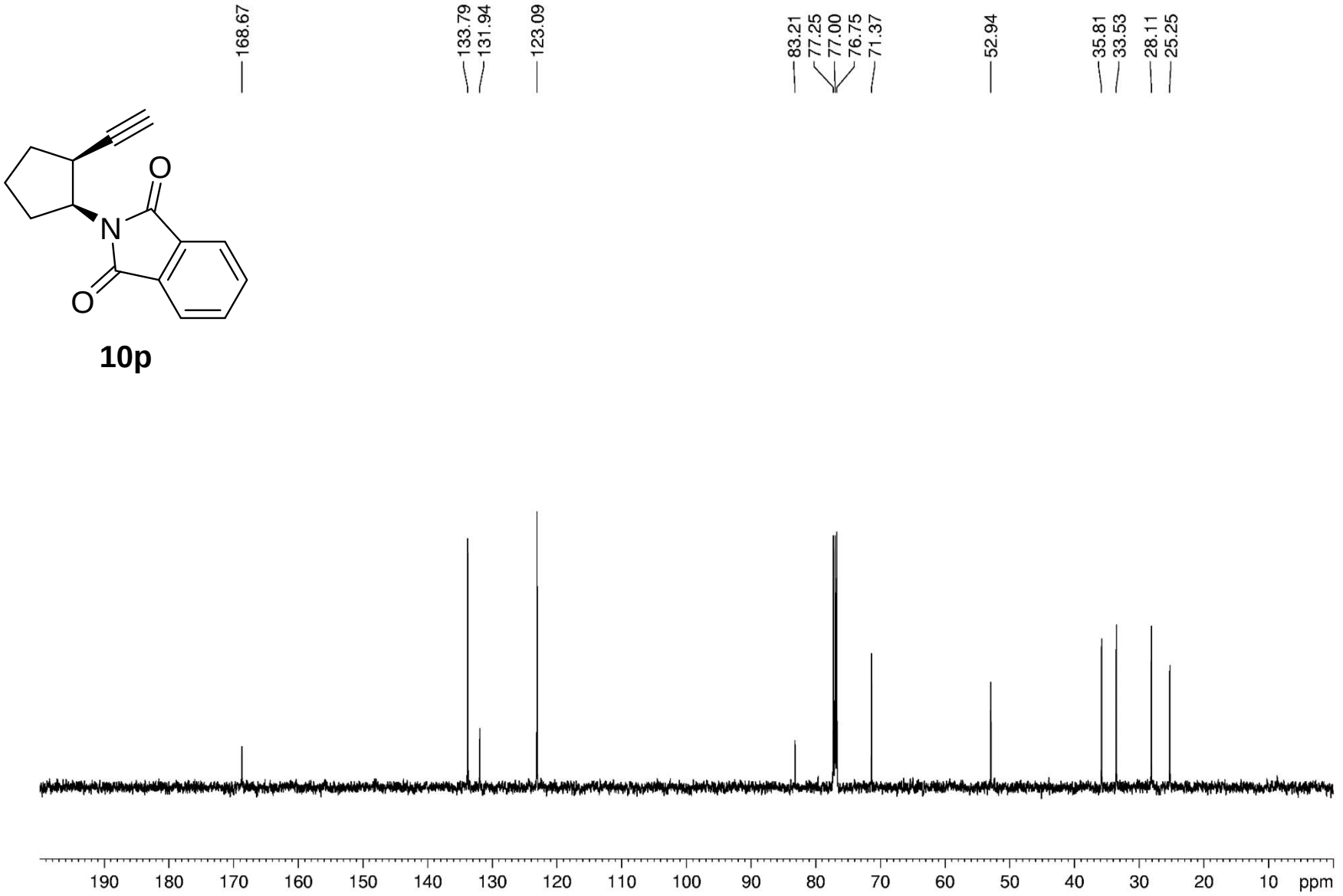


^{13}C NMR of compound **9hb** (75 MHz, $CDCl_3$)



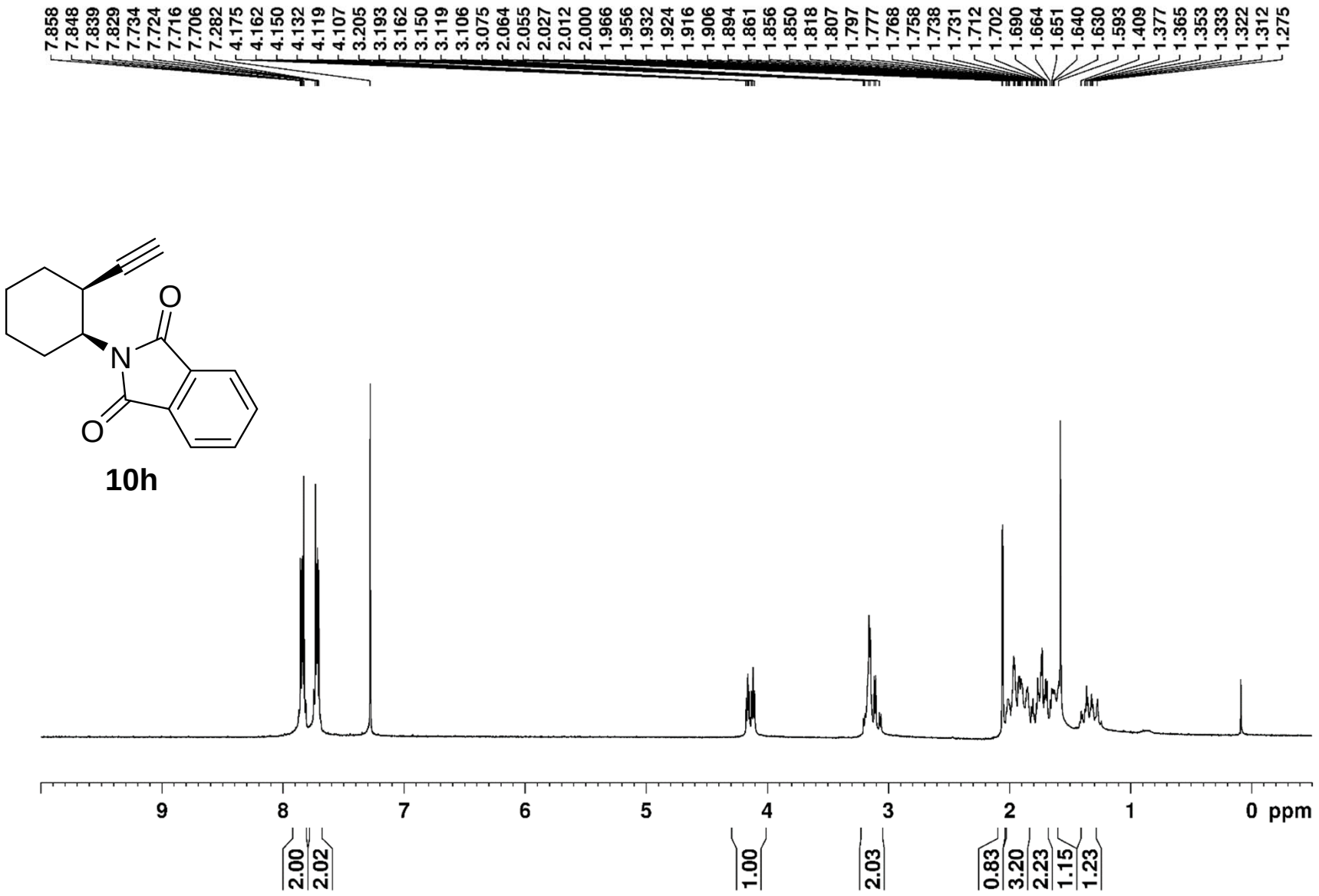
10p

^{1}H NMR of compound **10p** (500 MHz, $CDCl_3$)

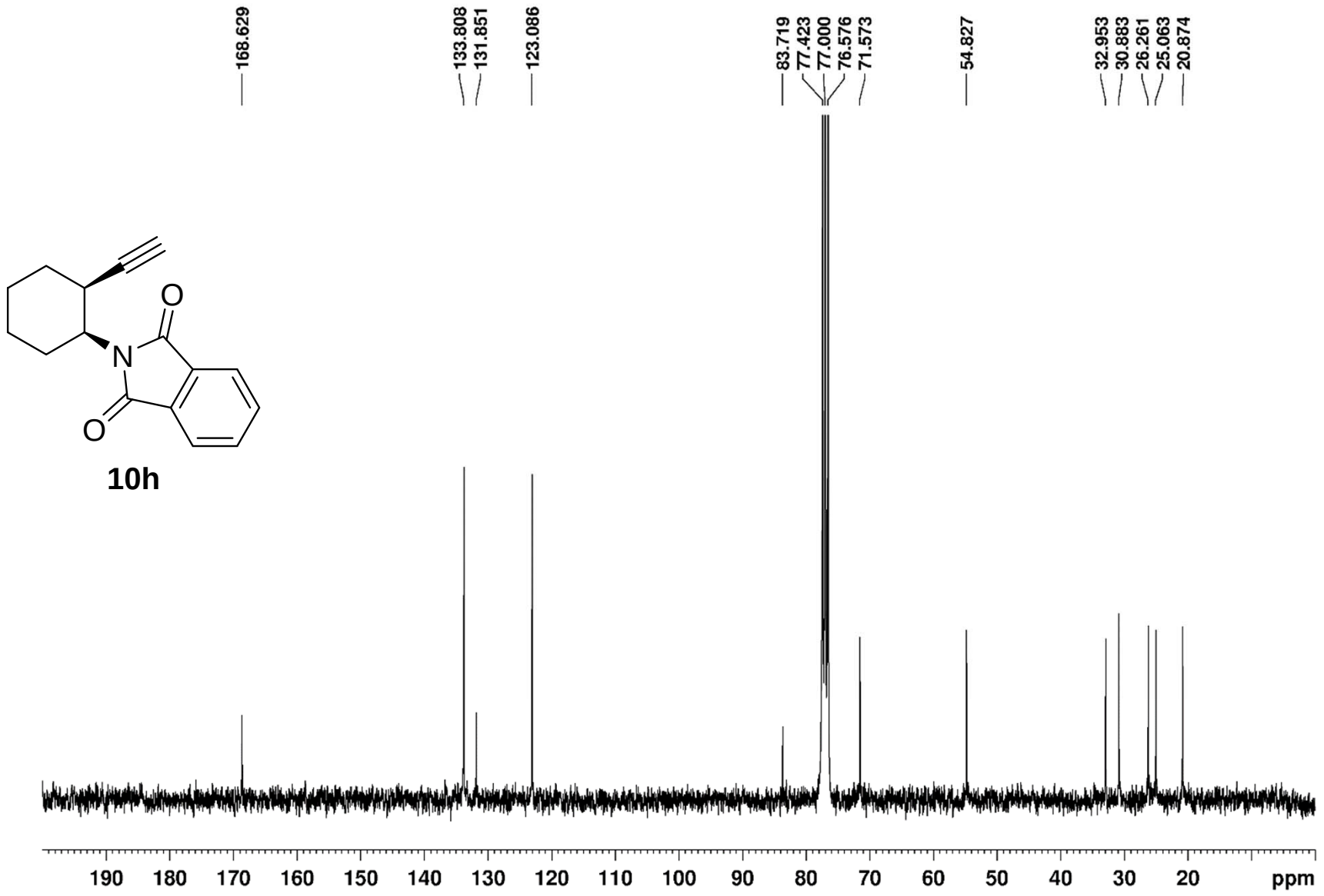


10p

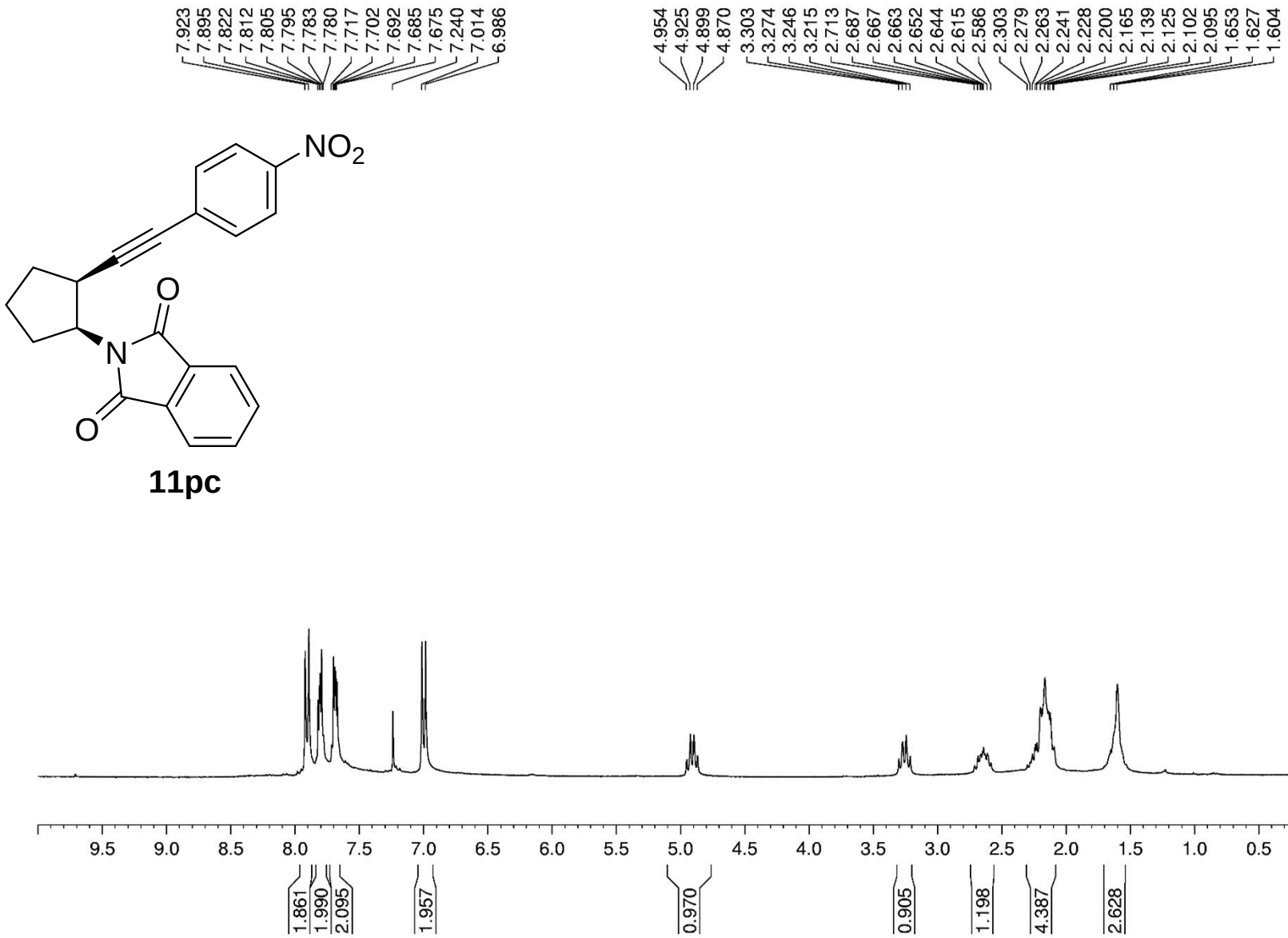
^{13}C NMR of compound **10p** (125 MHz, $CDCl_3$)



^{1}H NMR of compound **10h** (300 MHz, $CDCl_3$)

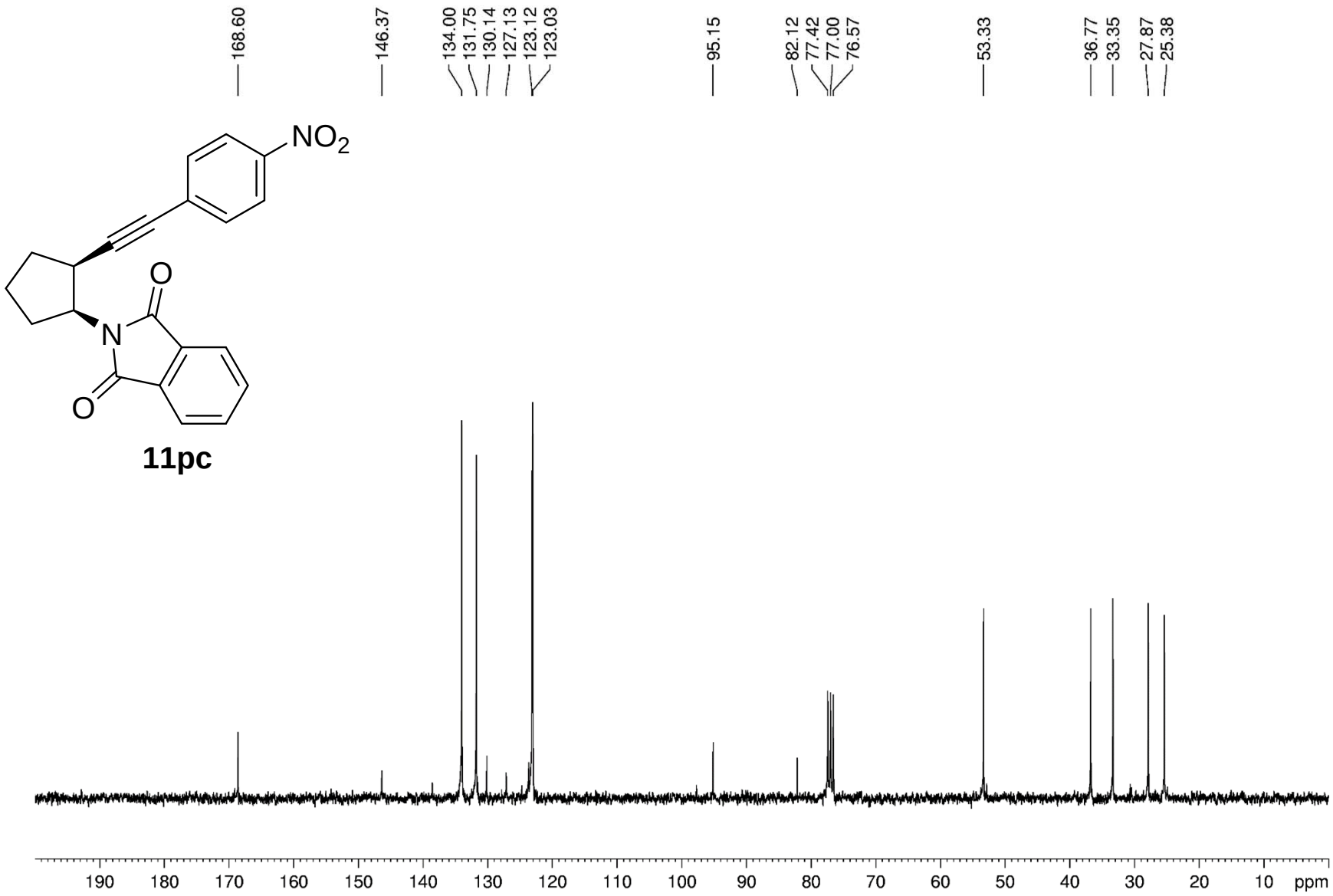


^{13}C NMR of compound **10h** (75 MHz, $CDCl_3$)

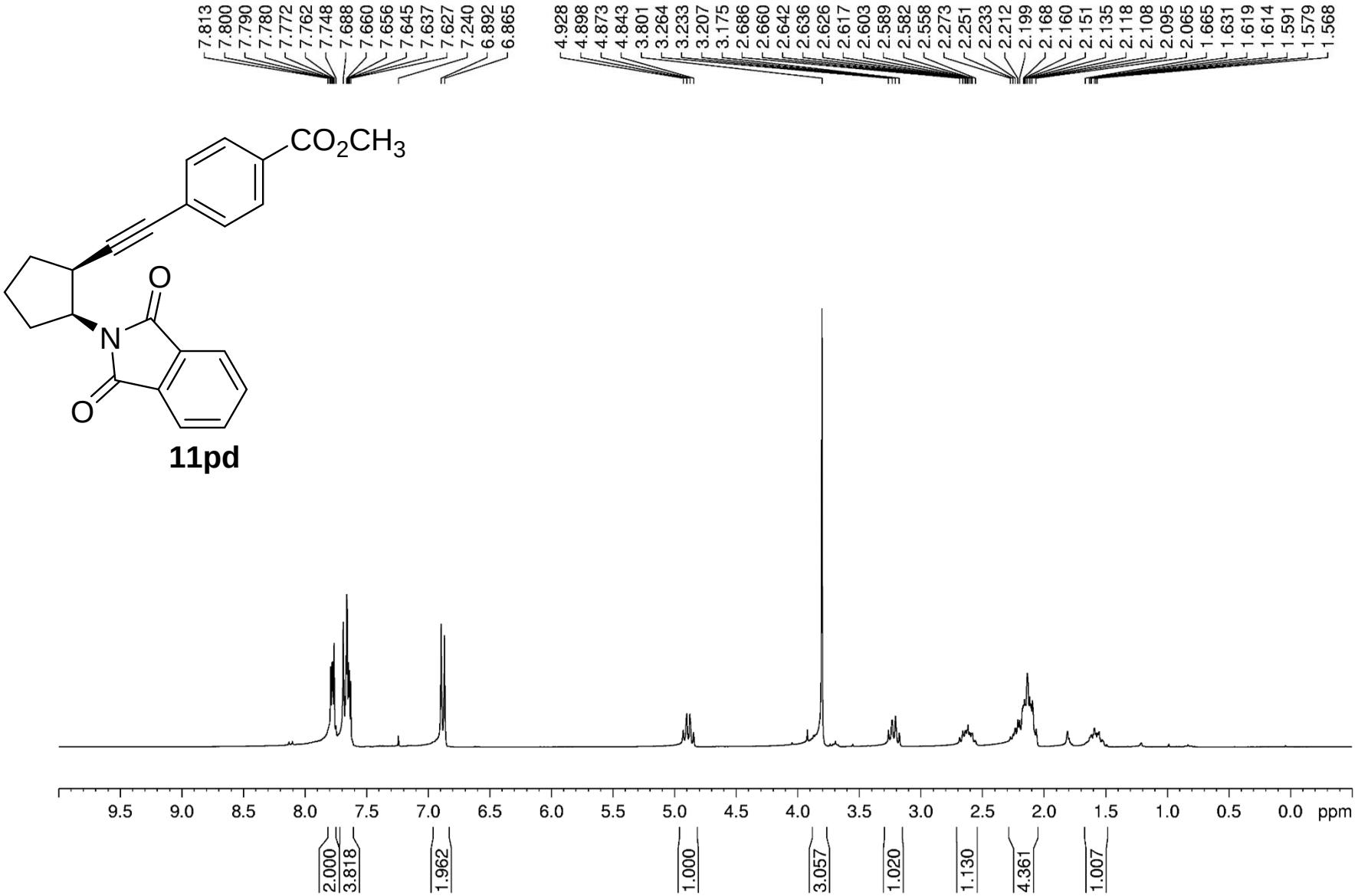


11pc

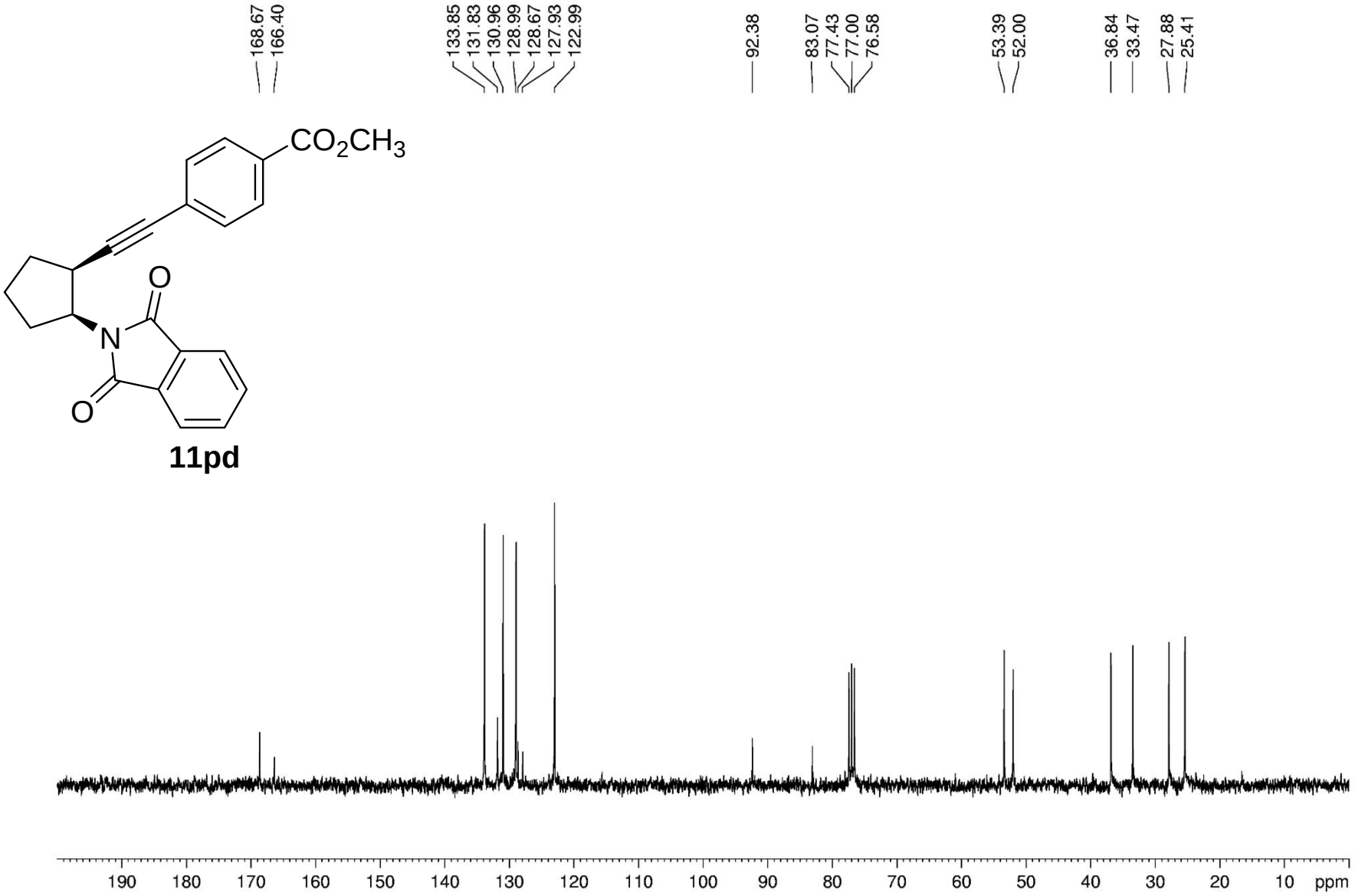
^{1}H NMR of compound **11pc** (300 MHz, $CDCl_3$)



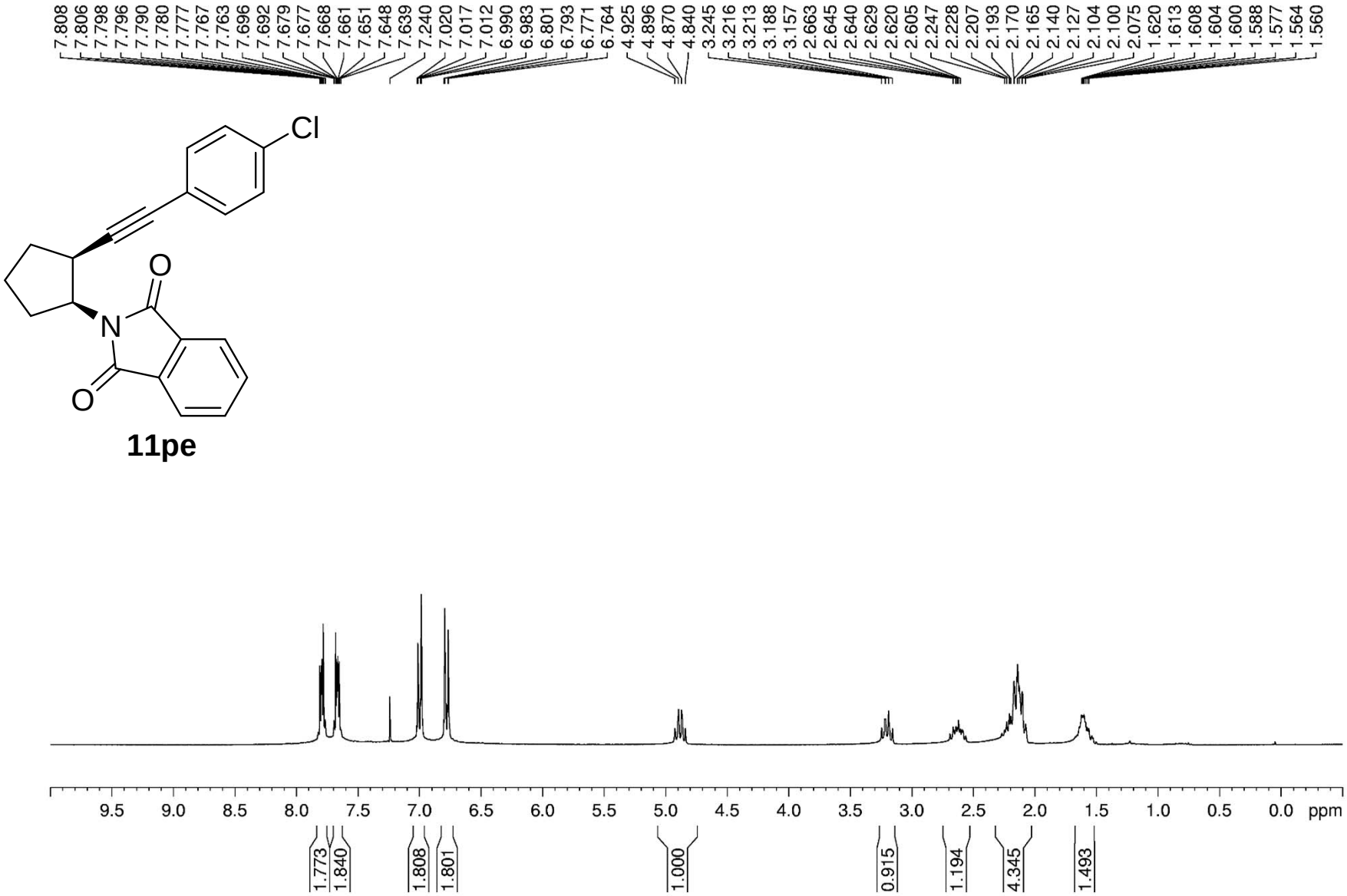
^{13}C NMR of compound **11pc** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **11pd** (300 MHz, $CDCl_3$)

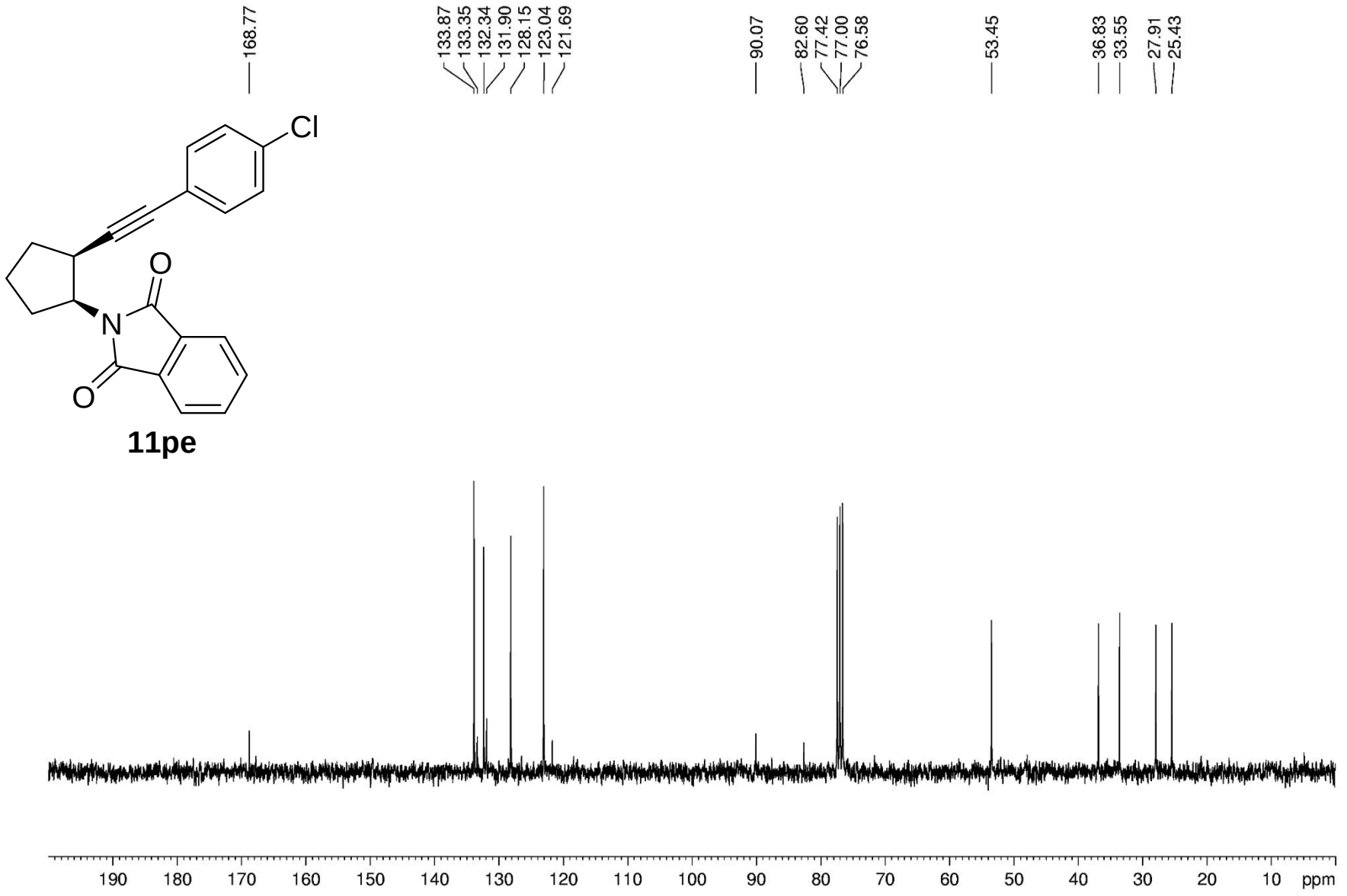


^{13}C NMR of compound **11pd** (75 MHz, $CDCl_3$)

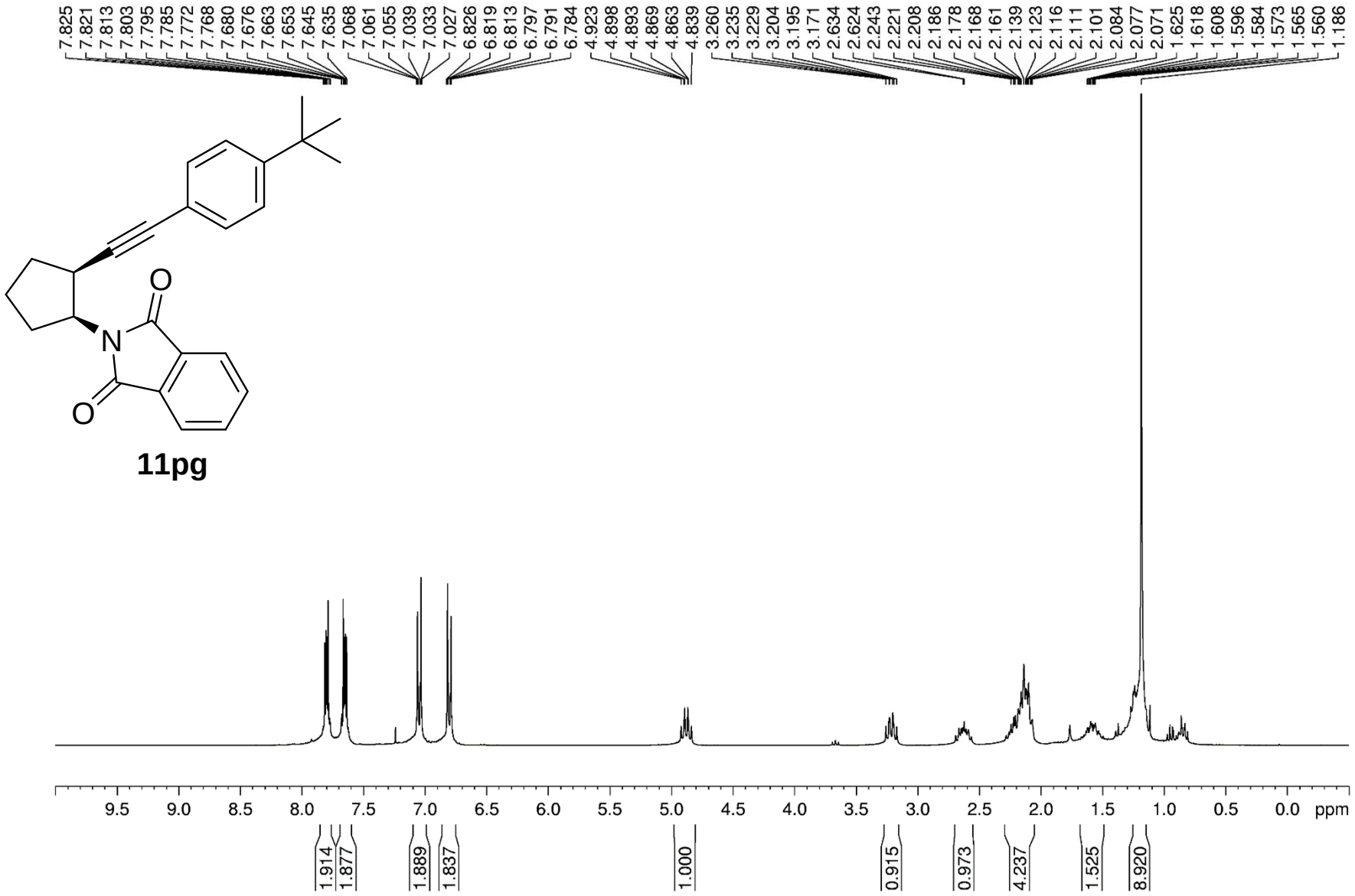


11pe

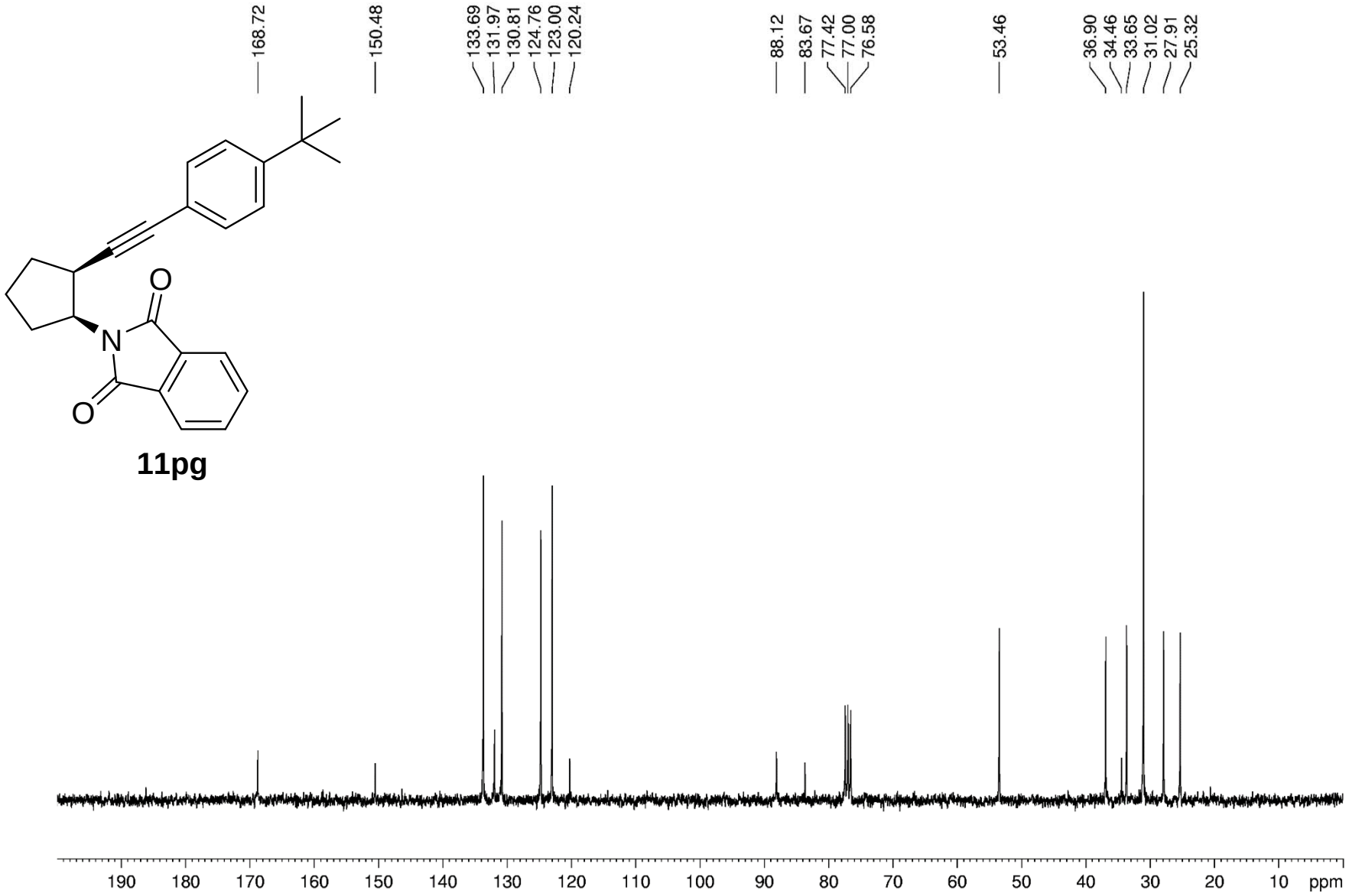
^{1}H NMR of compound **11pe** (300 MHz, $CDCl_3$)



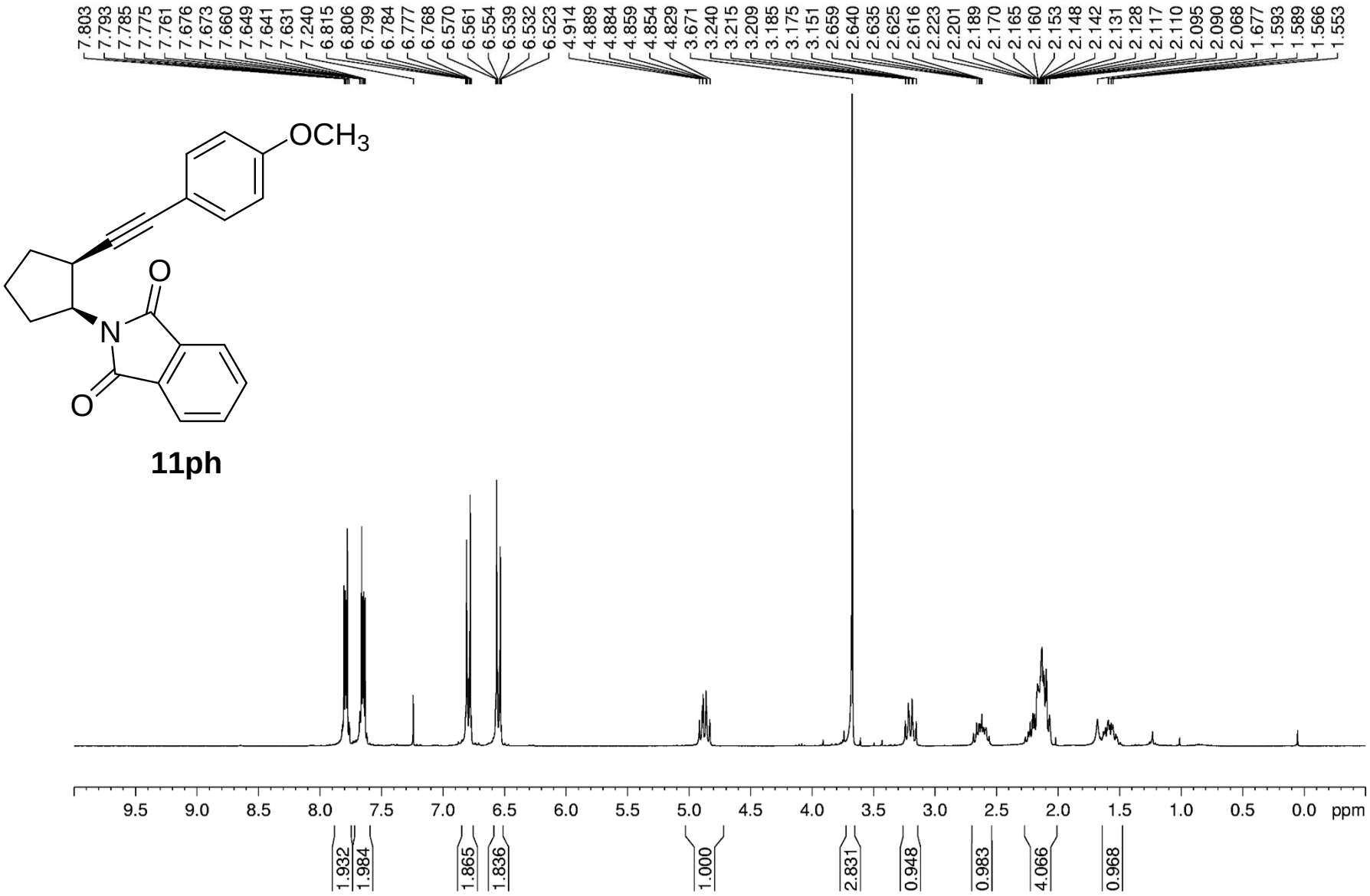
^{13}C NMR of compound **11pe** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **11pg** (300 MHz, $CDCl_3$)

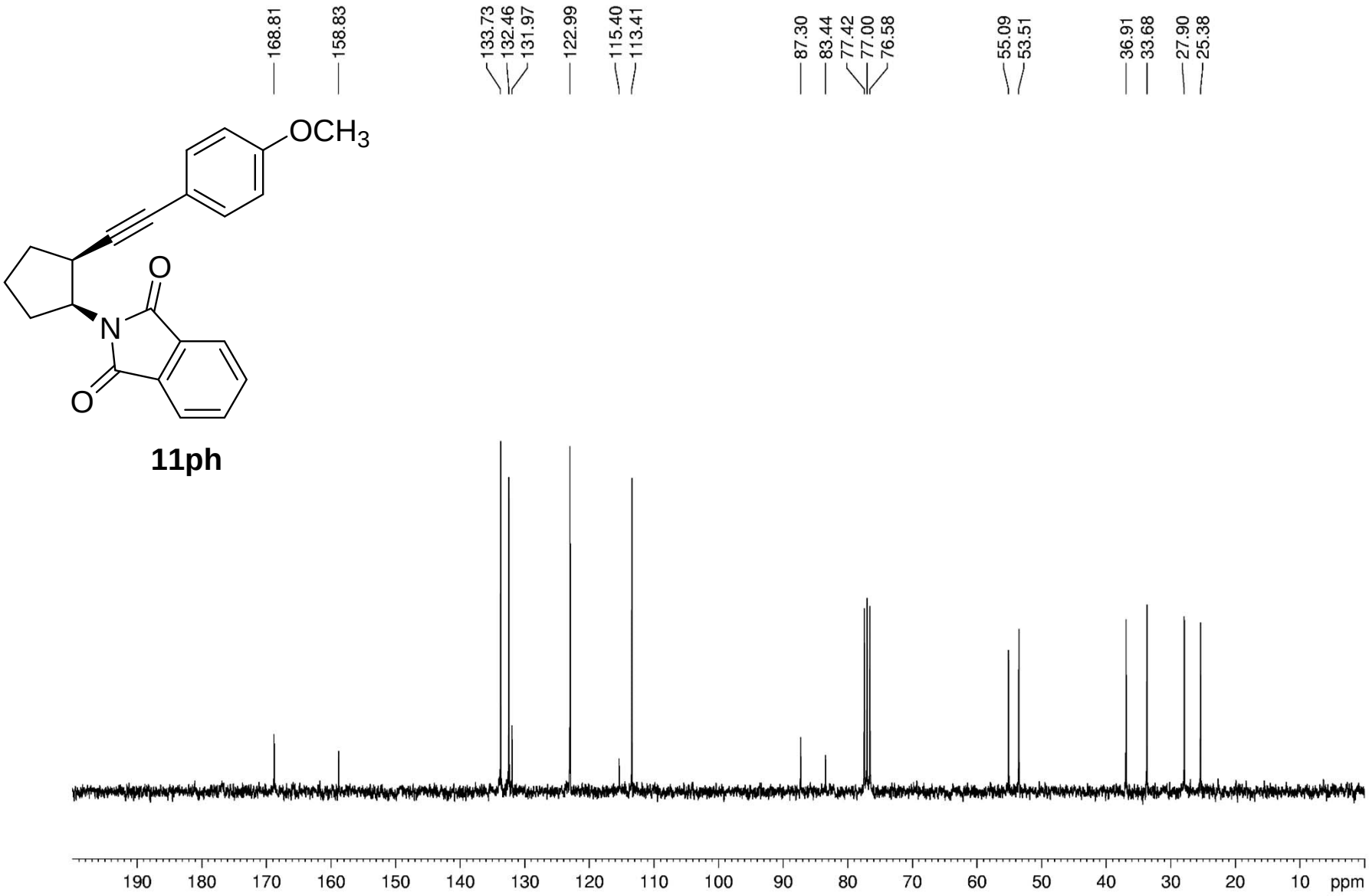


^{13}C NMR of compound **11pg** (75 MHz, $CDCl_3$)

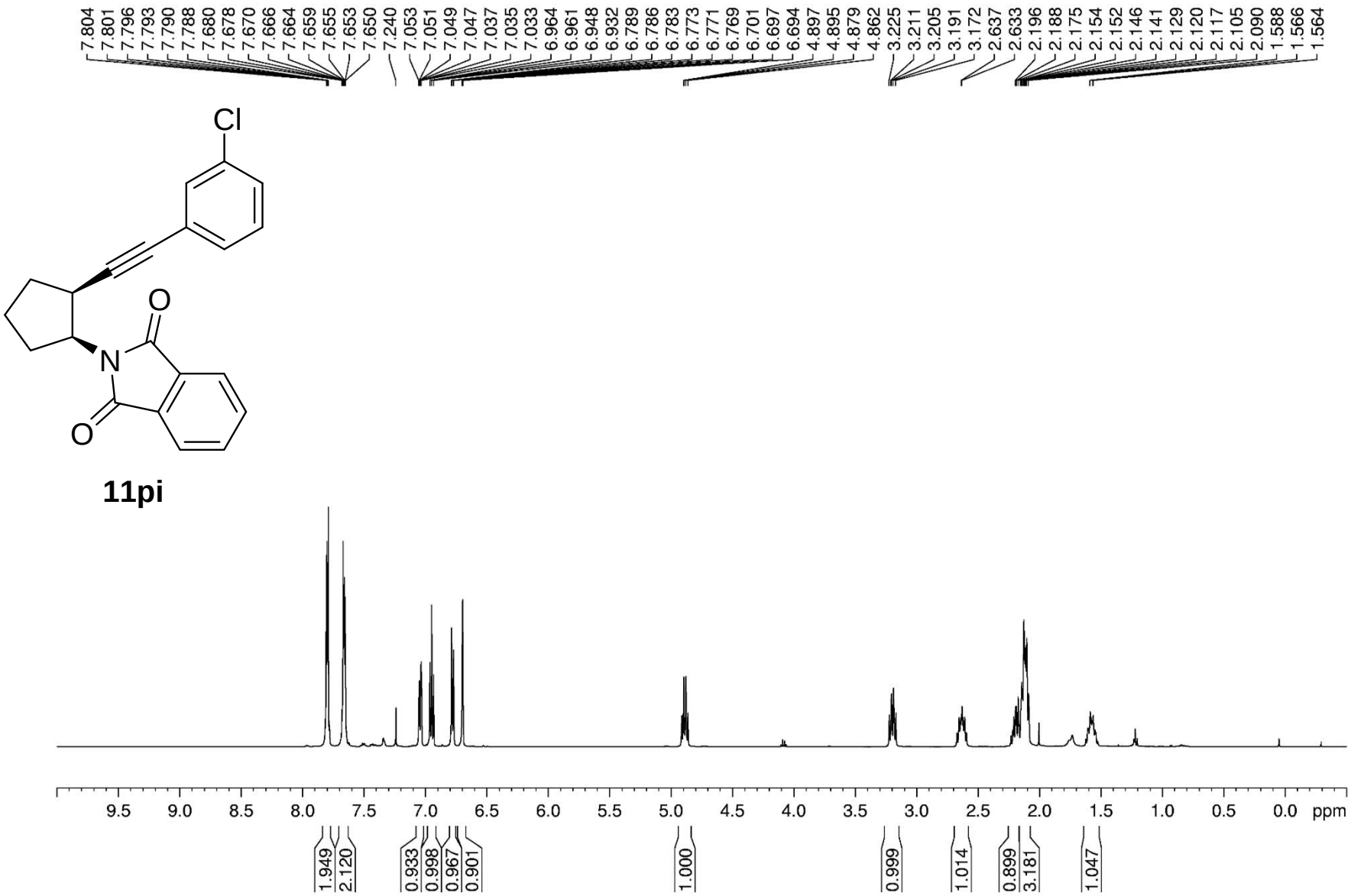


11ph

^{1}H NMR of compound **11ph** (300 MHz, $CDCl_3$)

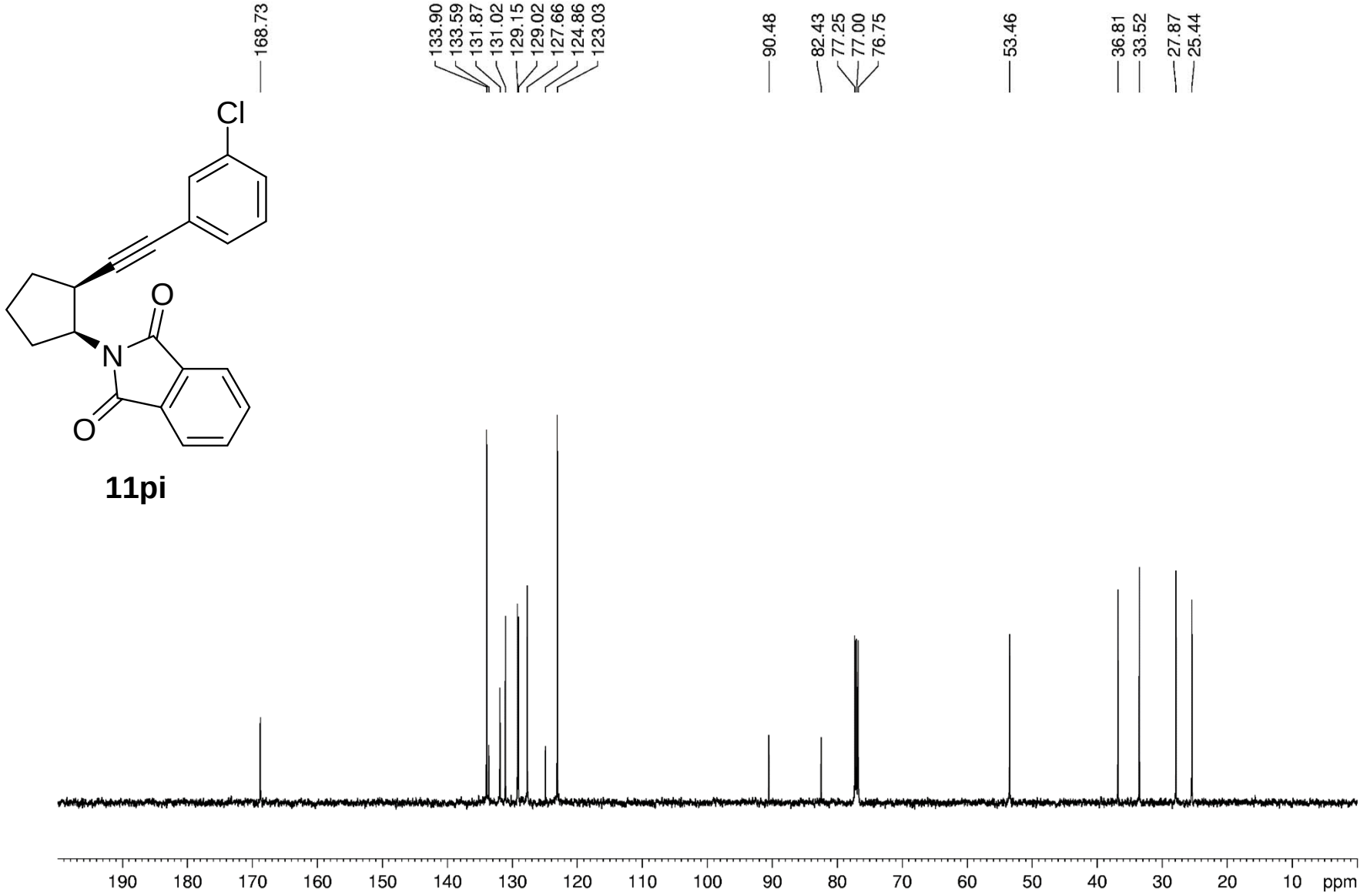


^{13}C NMR of compound **11ph** (75 MHz, $CDCl_3$)

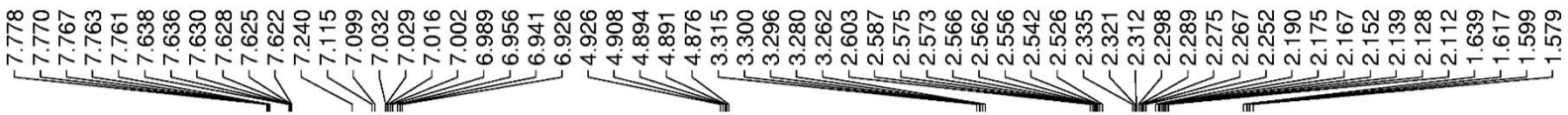


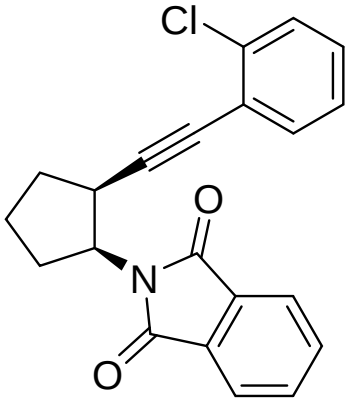
11pi

^{1}H NMR of compound **11pi** (500 MHz, $CDCl_3$)

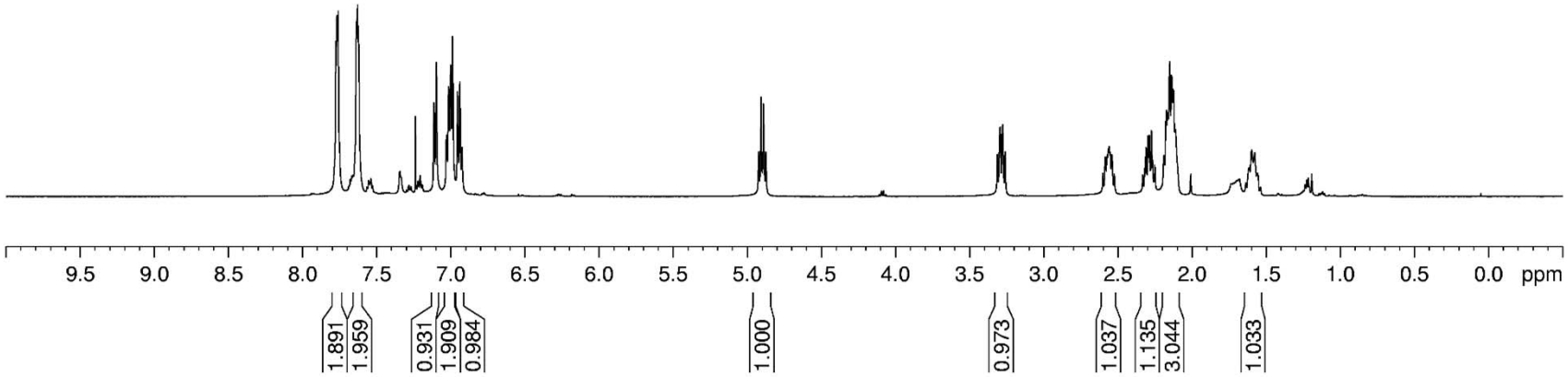


^{13}C NMR of compound **11pi** (125 MHz, $CDCl_3$)

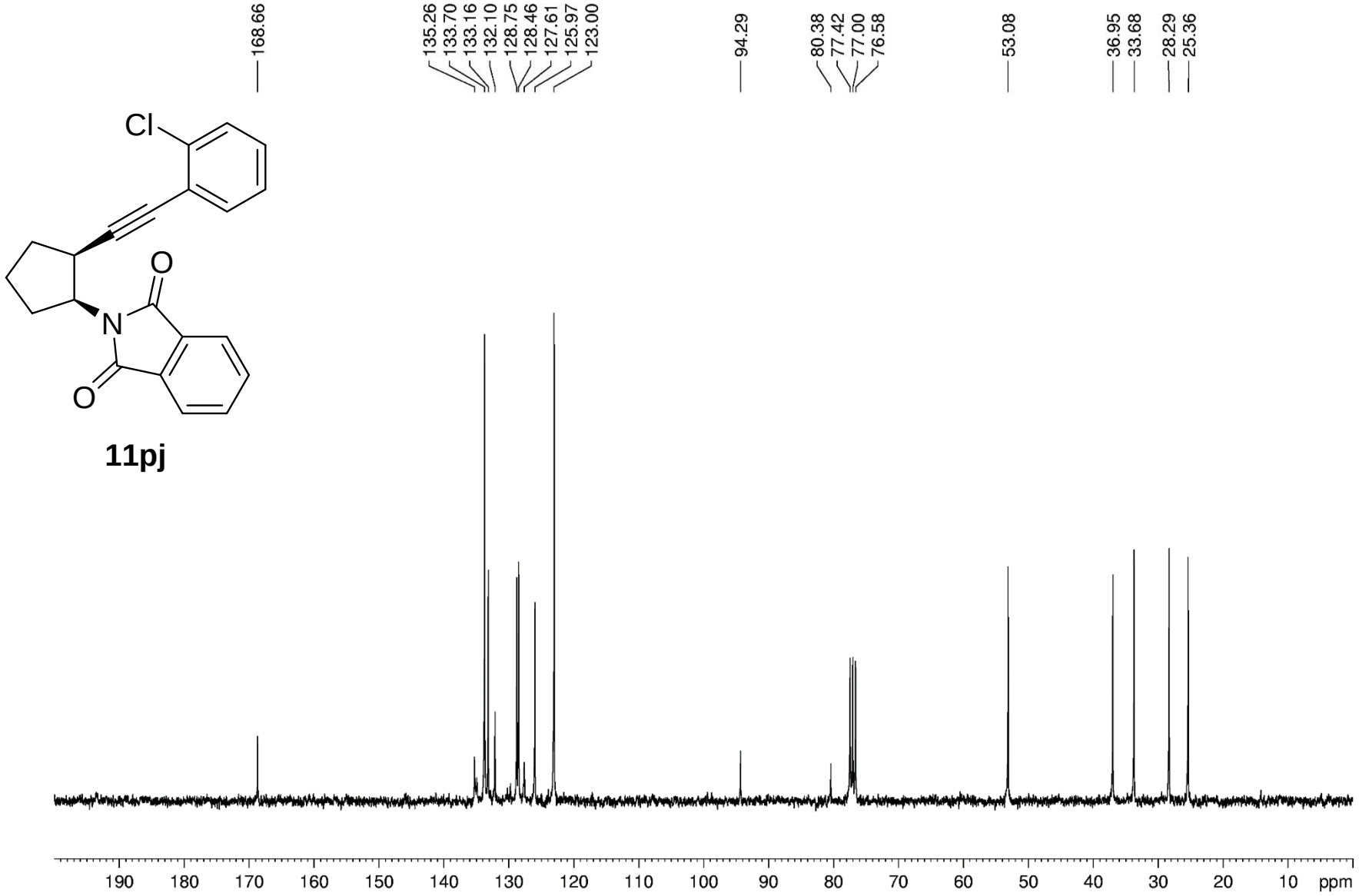




11pj

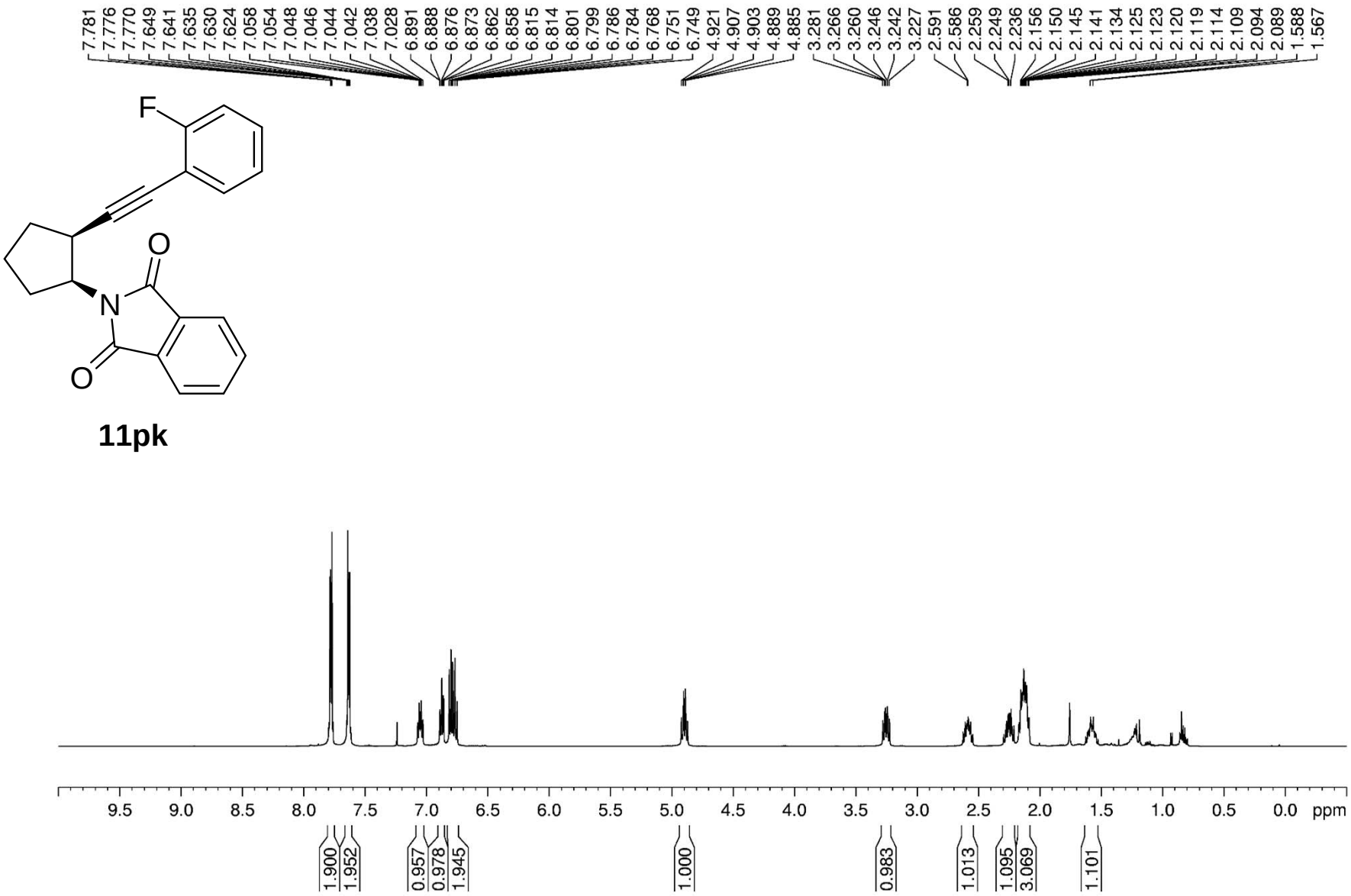


^{1}H NMR of compound **11pj** (500 MHz, $CDCl_3$)



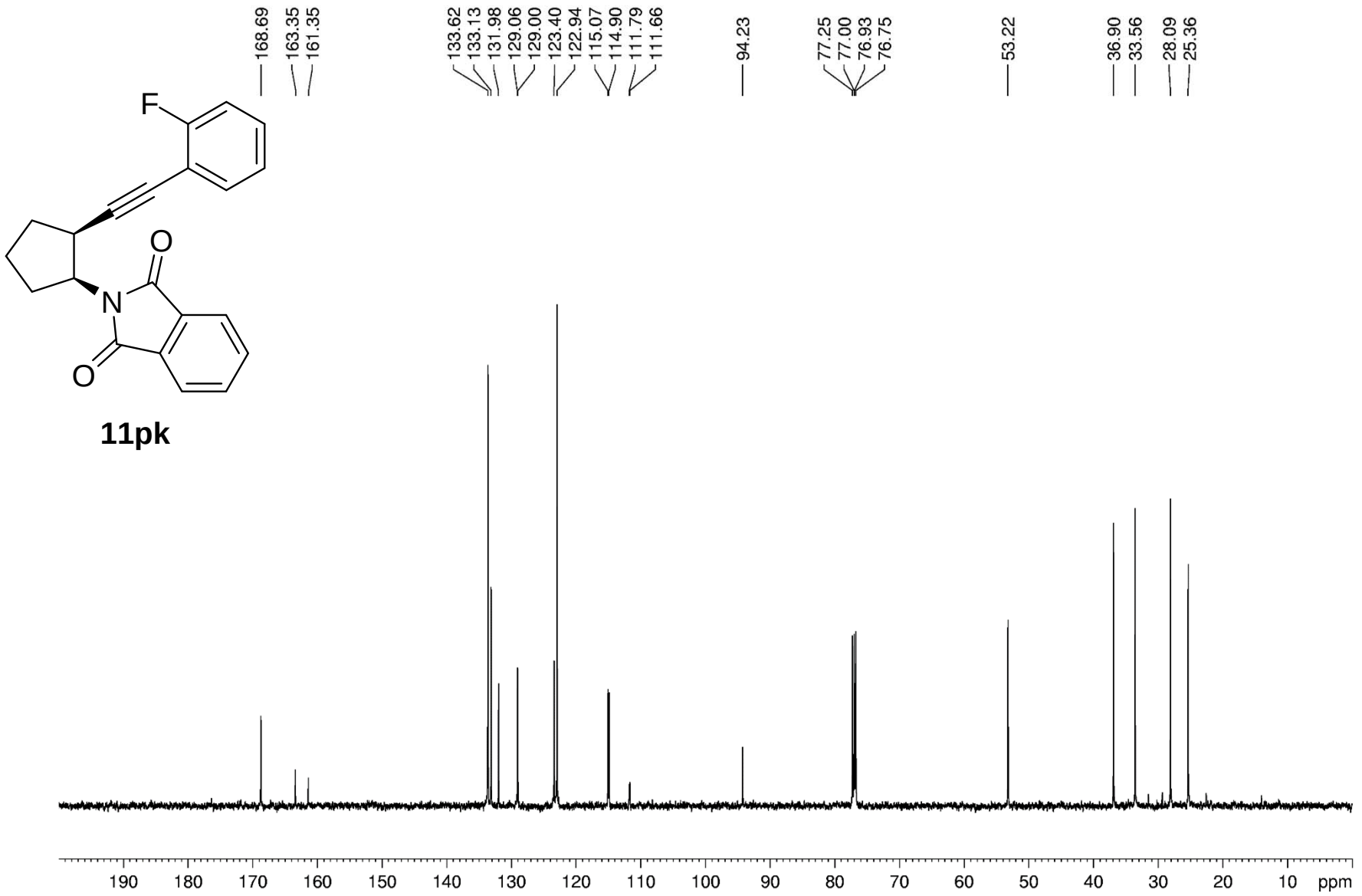
11pj

^{13}C NMR of compound **11pj** (75 MHz, $CDCl_3$)

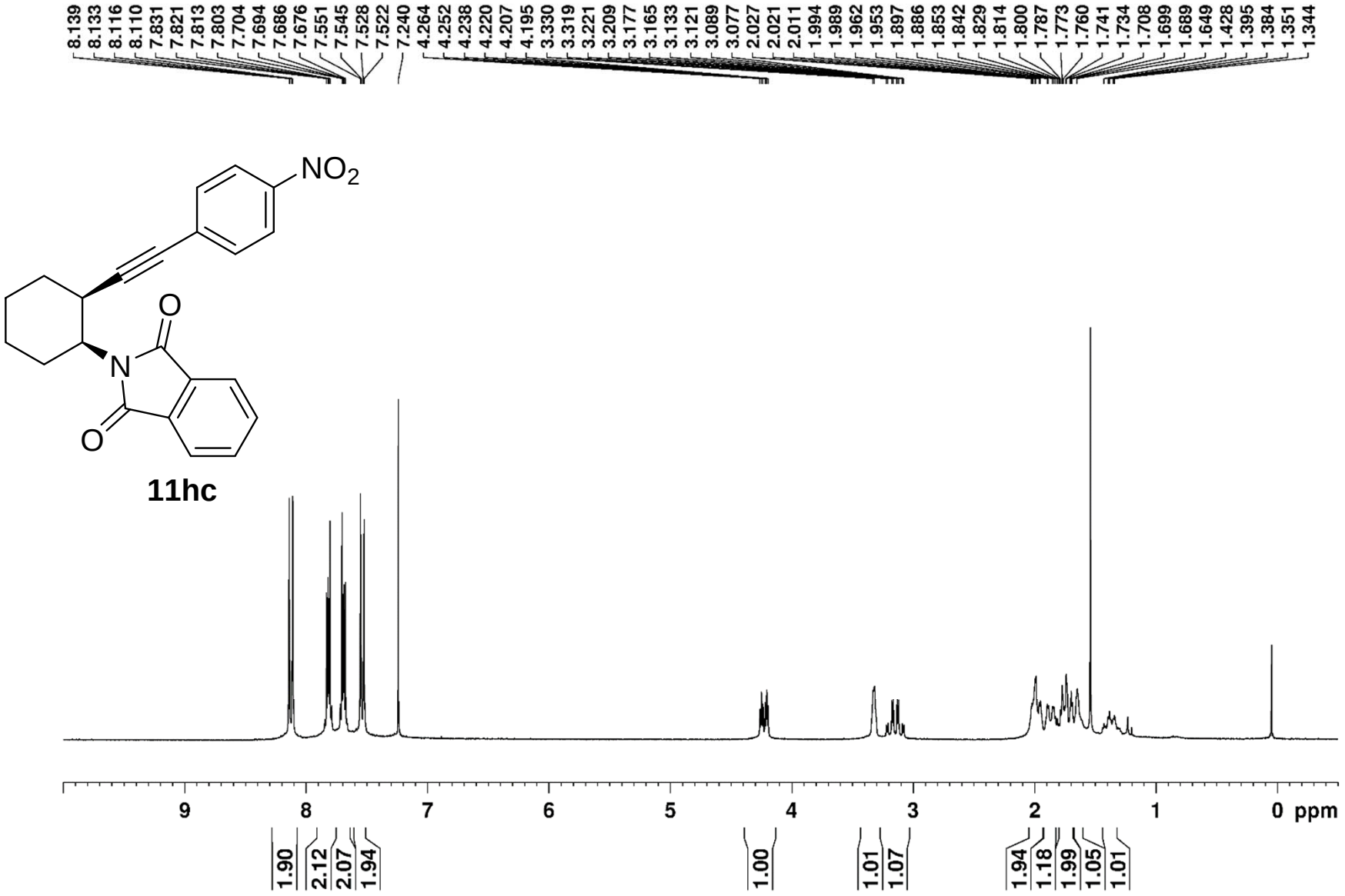


11pk

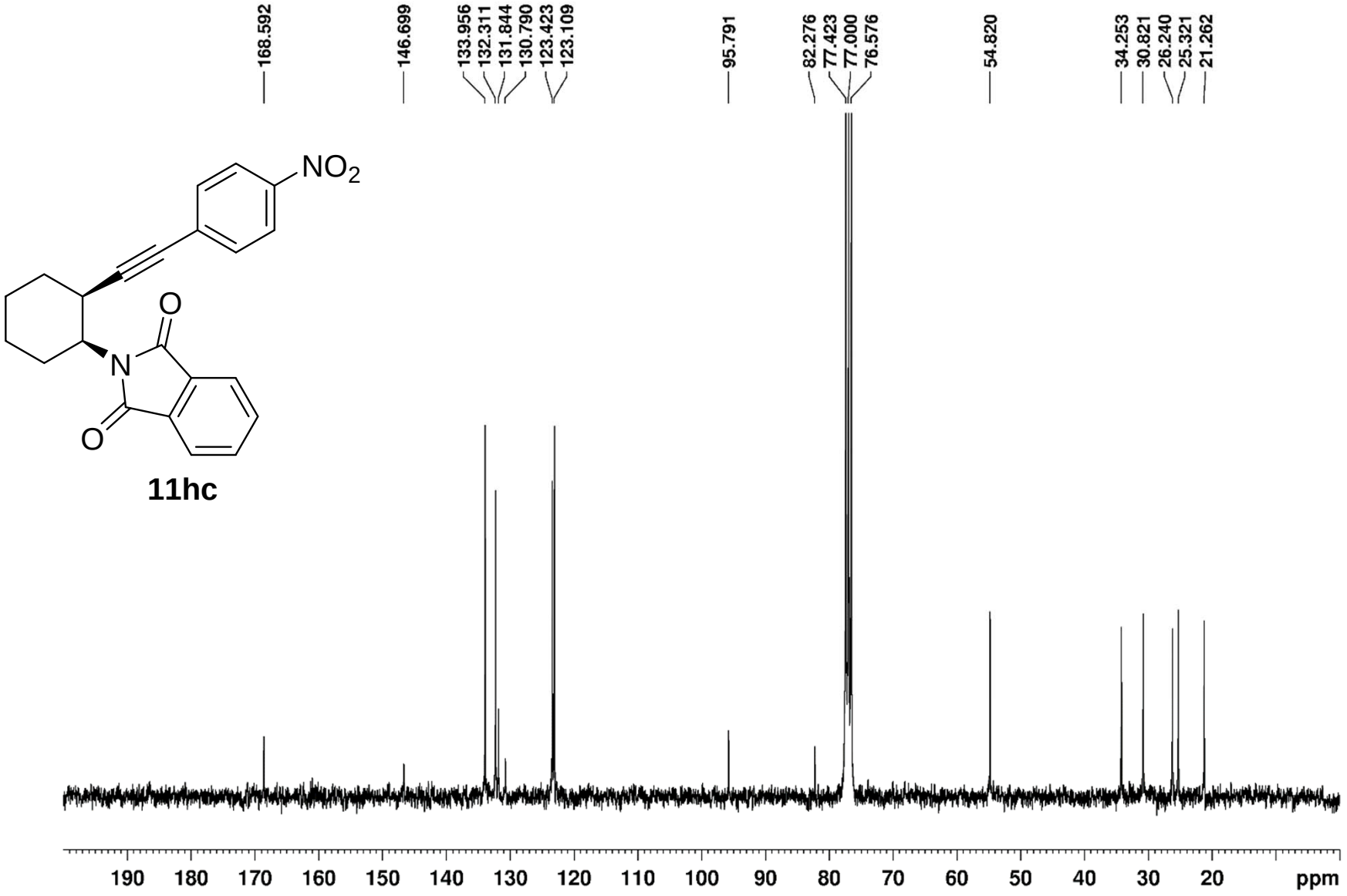
^{1}H NMR of compound **11pk** (500 MHz, $CDCl_3$)



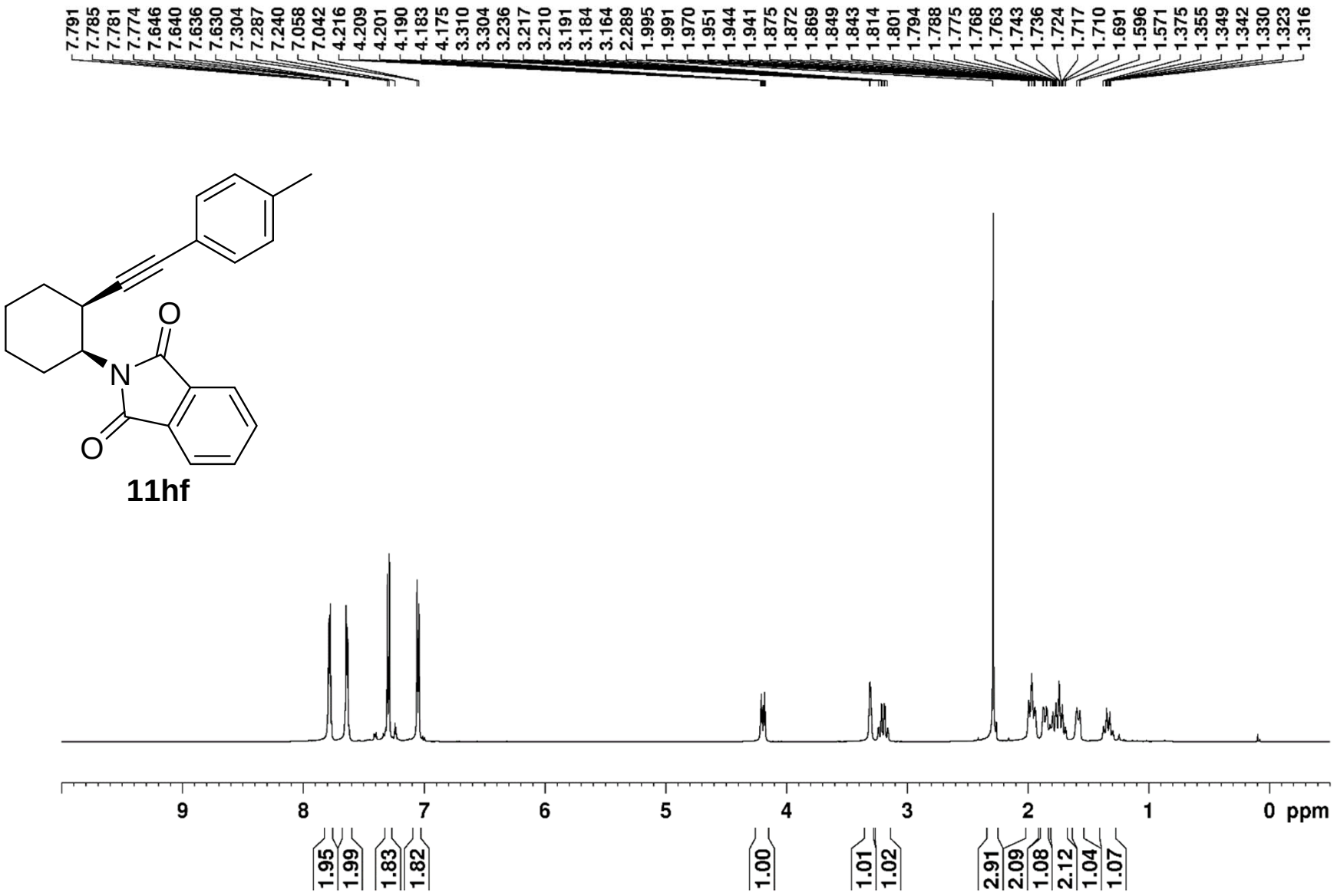
^{13}C NMR of compound **11pk** (125 MHz, $CDCl_3$)



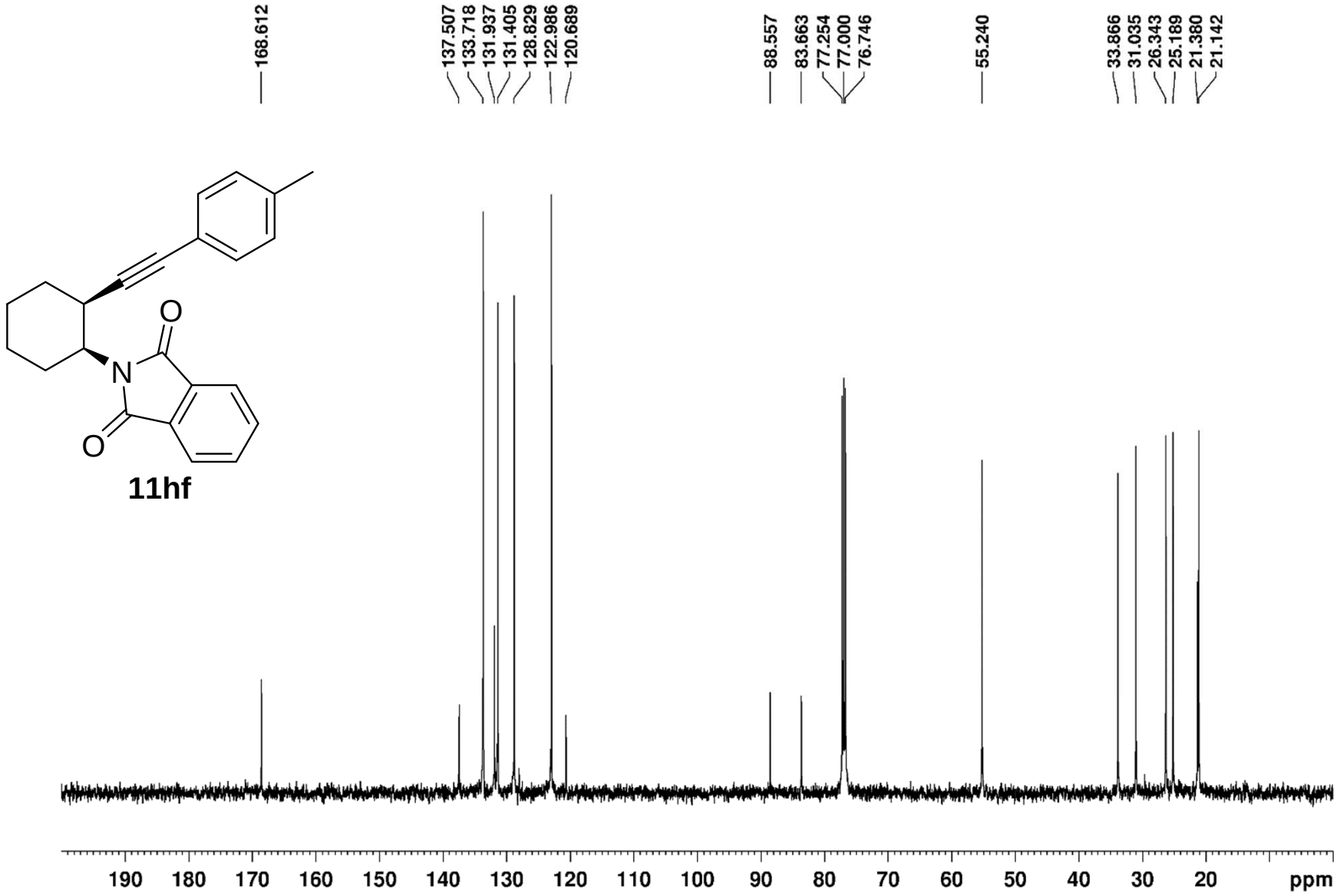
^{1}H NMR of compound **11hc** (300 MHz, $CDCl_3$)



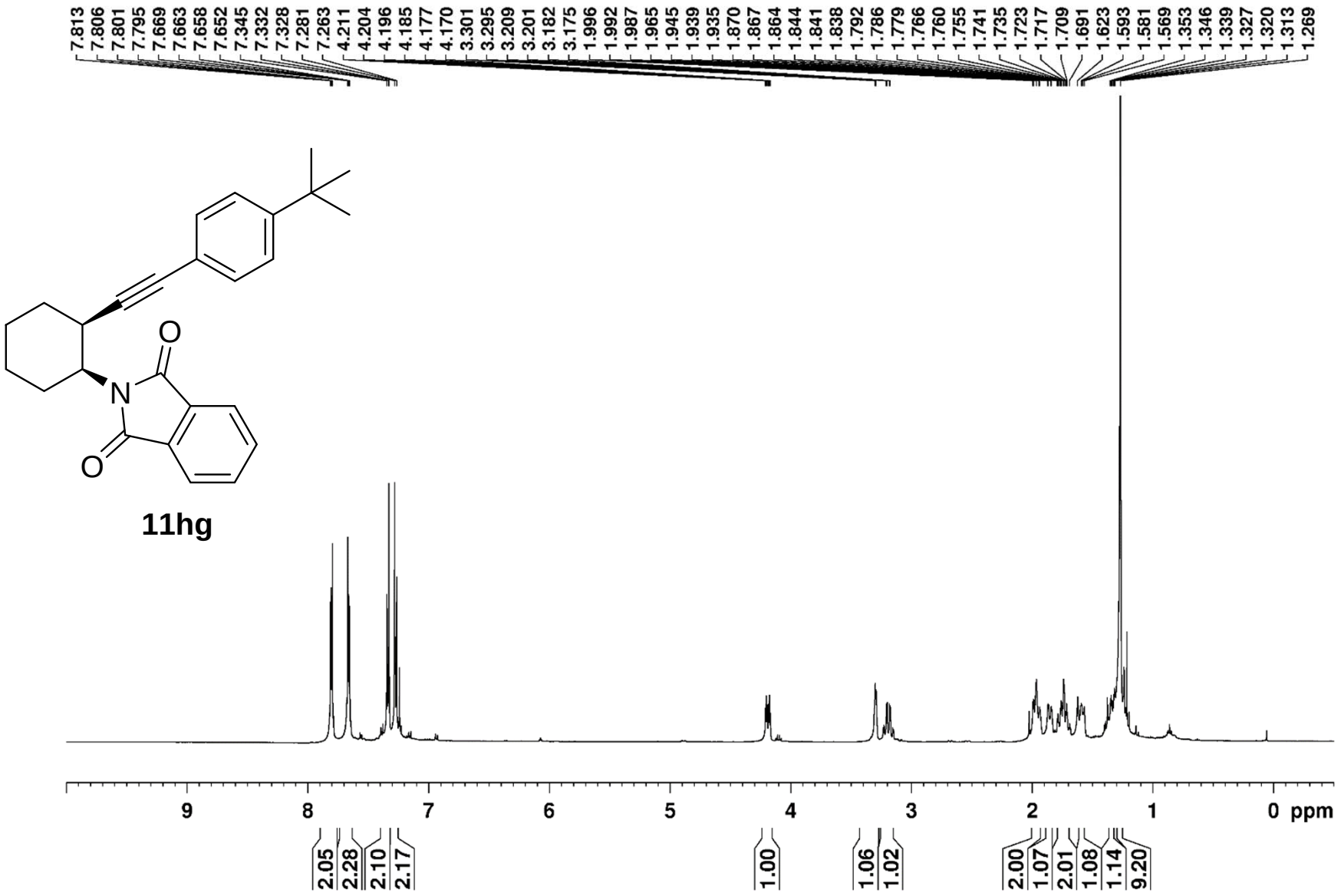
^{13}C NMR of compound **11hc** (75 MHz, $CDCl_3$)



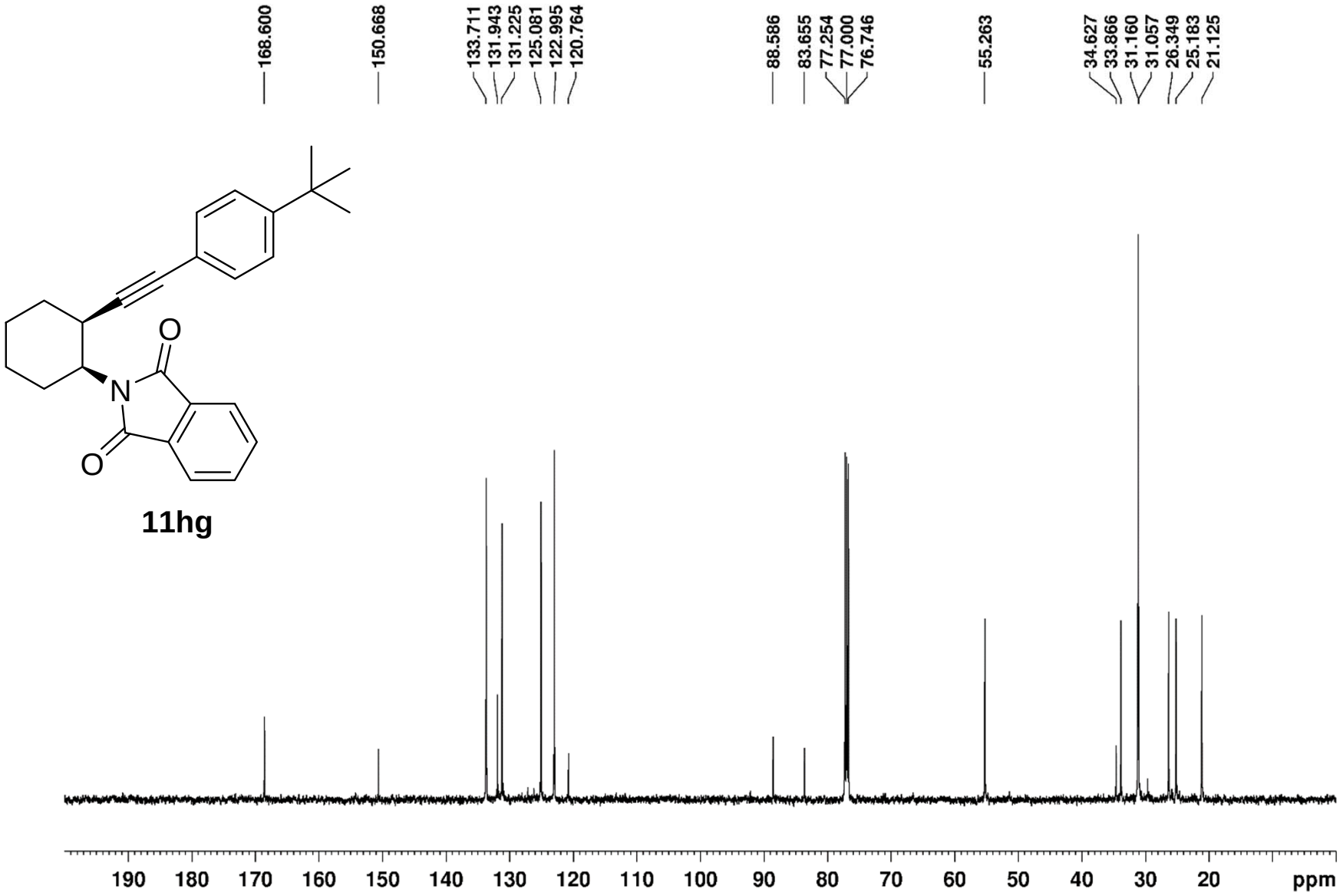
^{1}H NMR of compound **11hf** (500 MHz, $CDCl_3$)



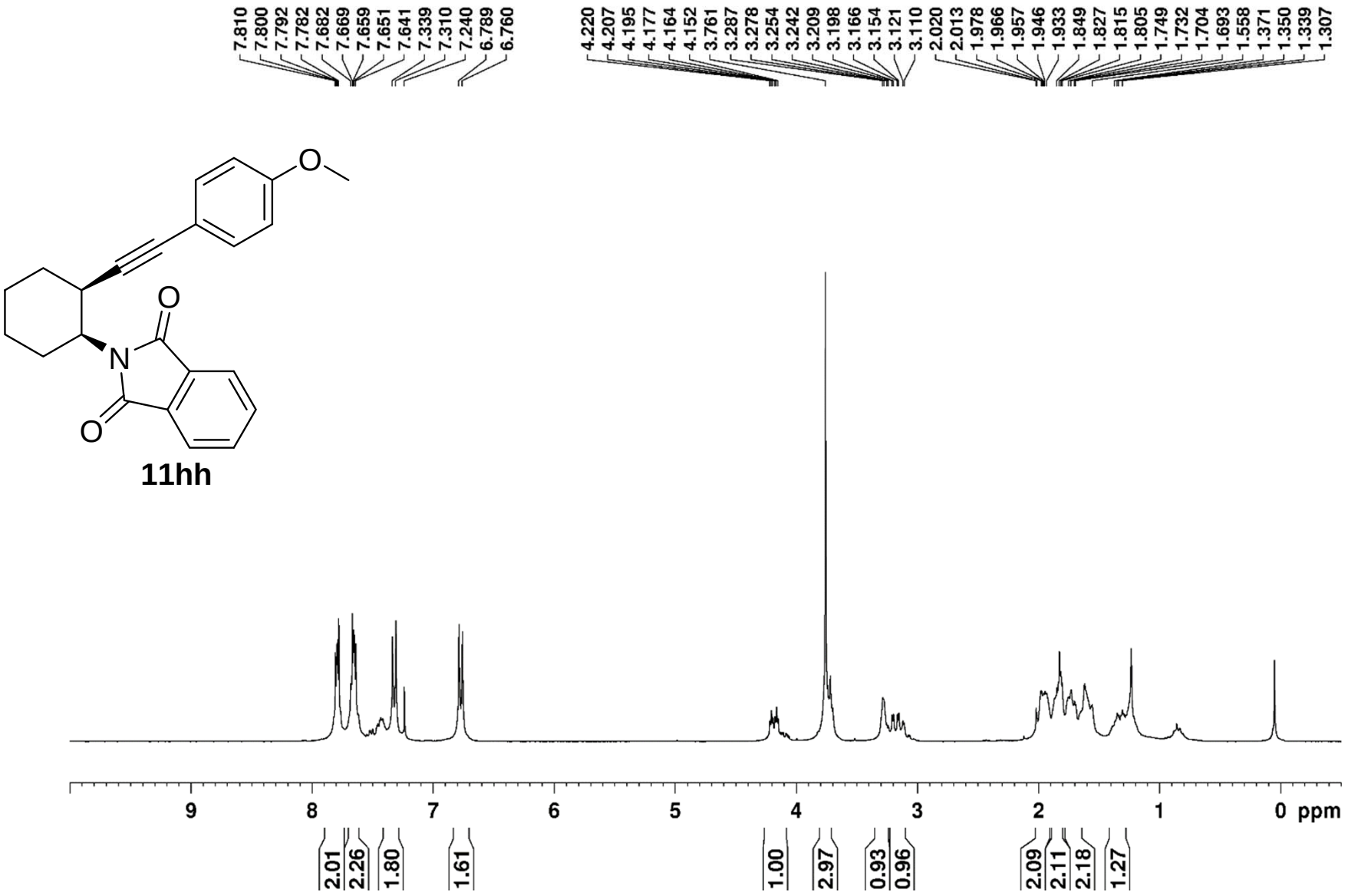
^{13}C NMR of compound **11hf** (125 MHz, $CDCl_3$)



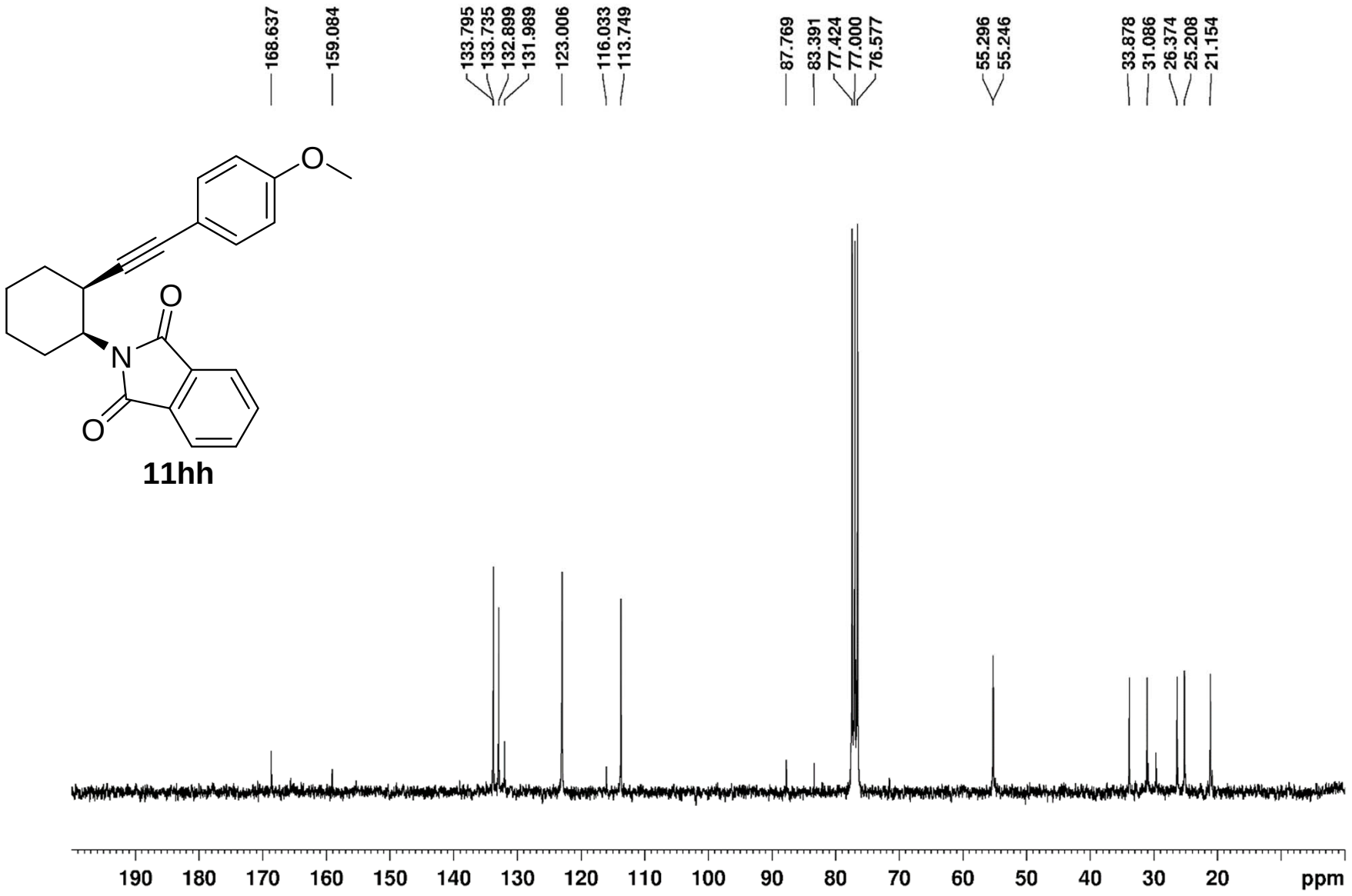
^{1}H NMR of compound **11hg** (500 MHz, $CDCl_3$)



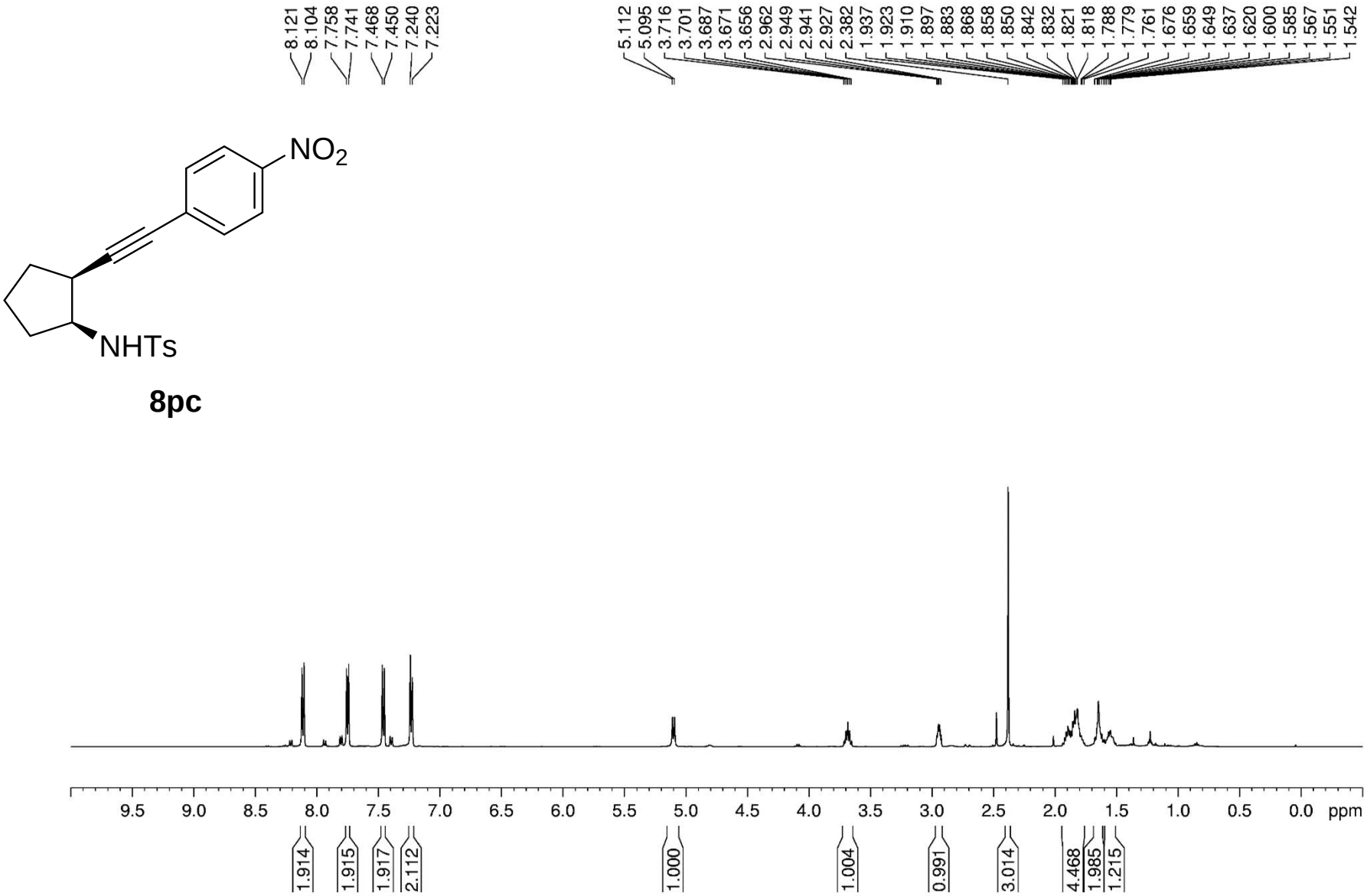
^{13}C NMR of compound **11hg** (125 MHz, $CDCl_3$)



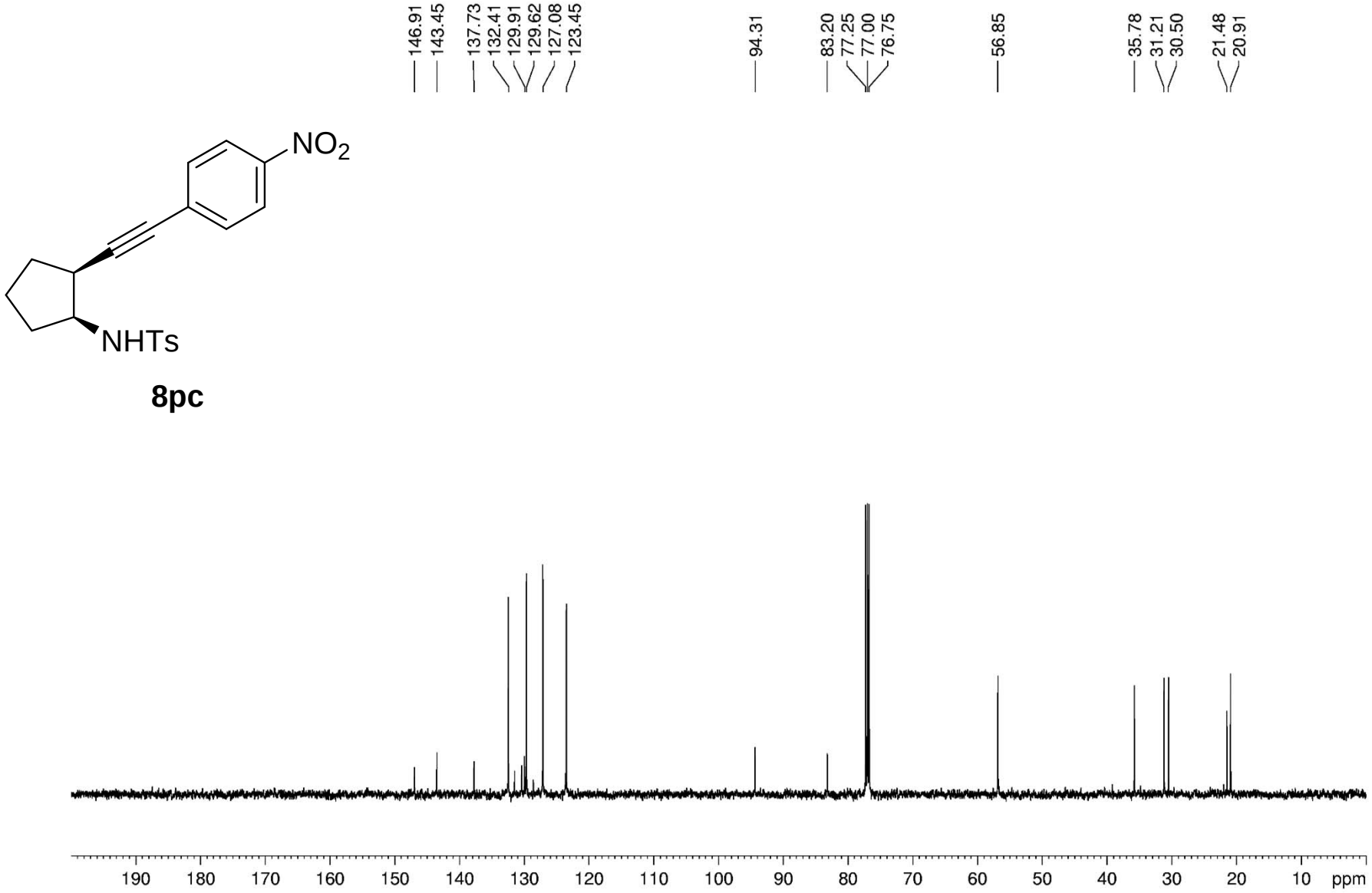
^{1}H NMR of compound **11hh** (300 MHz, $CDCl_3$)



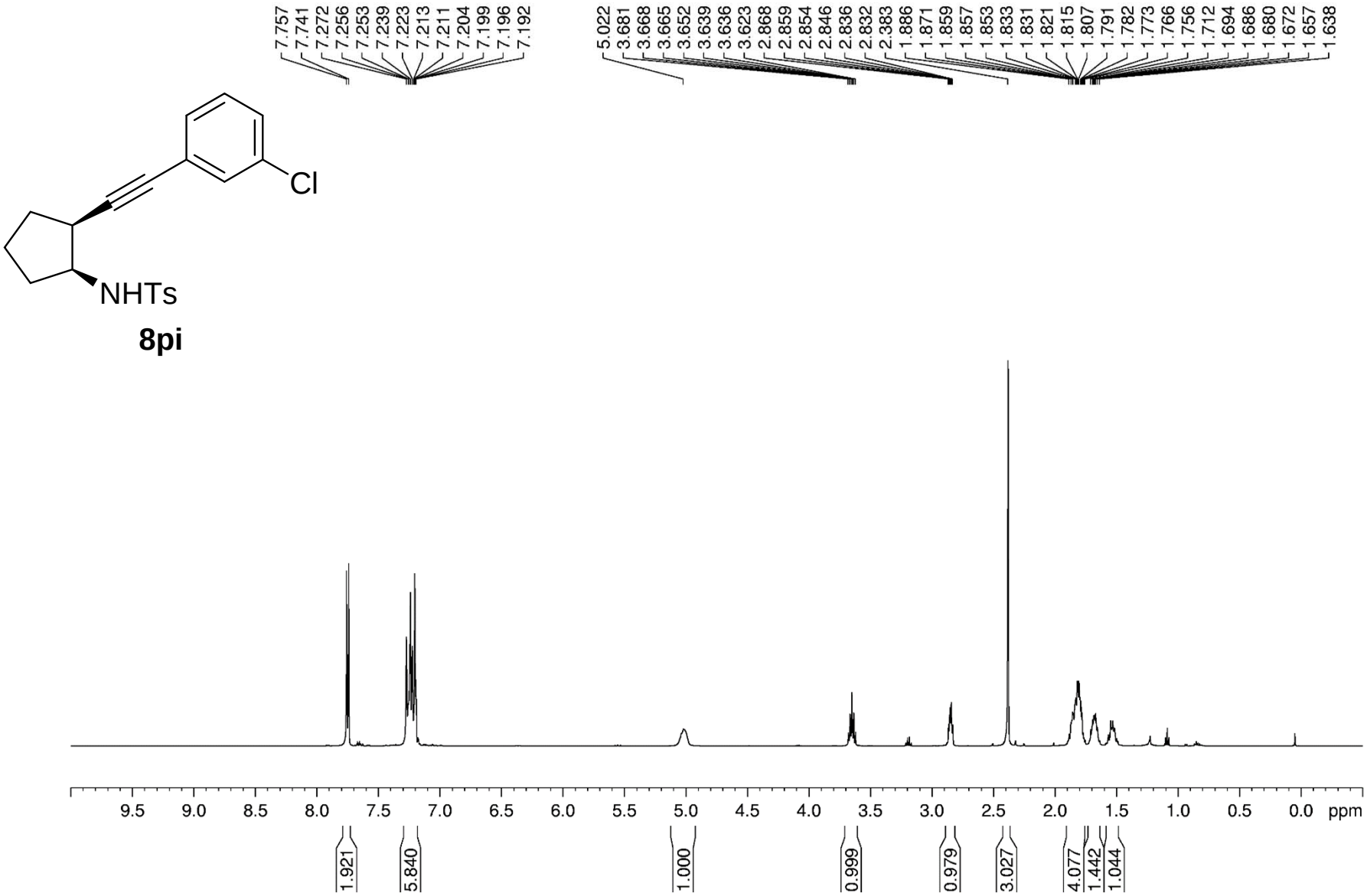
^{13}C NMR of compound **11hh** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **8pc** (500 MHz, $CDCl_3$)

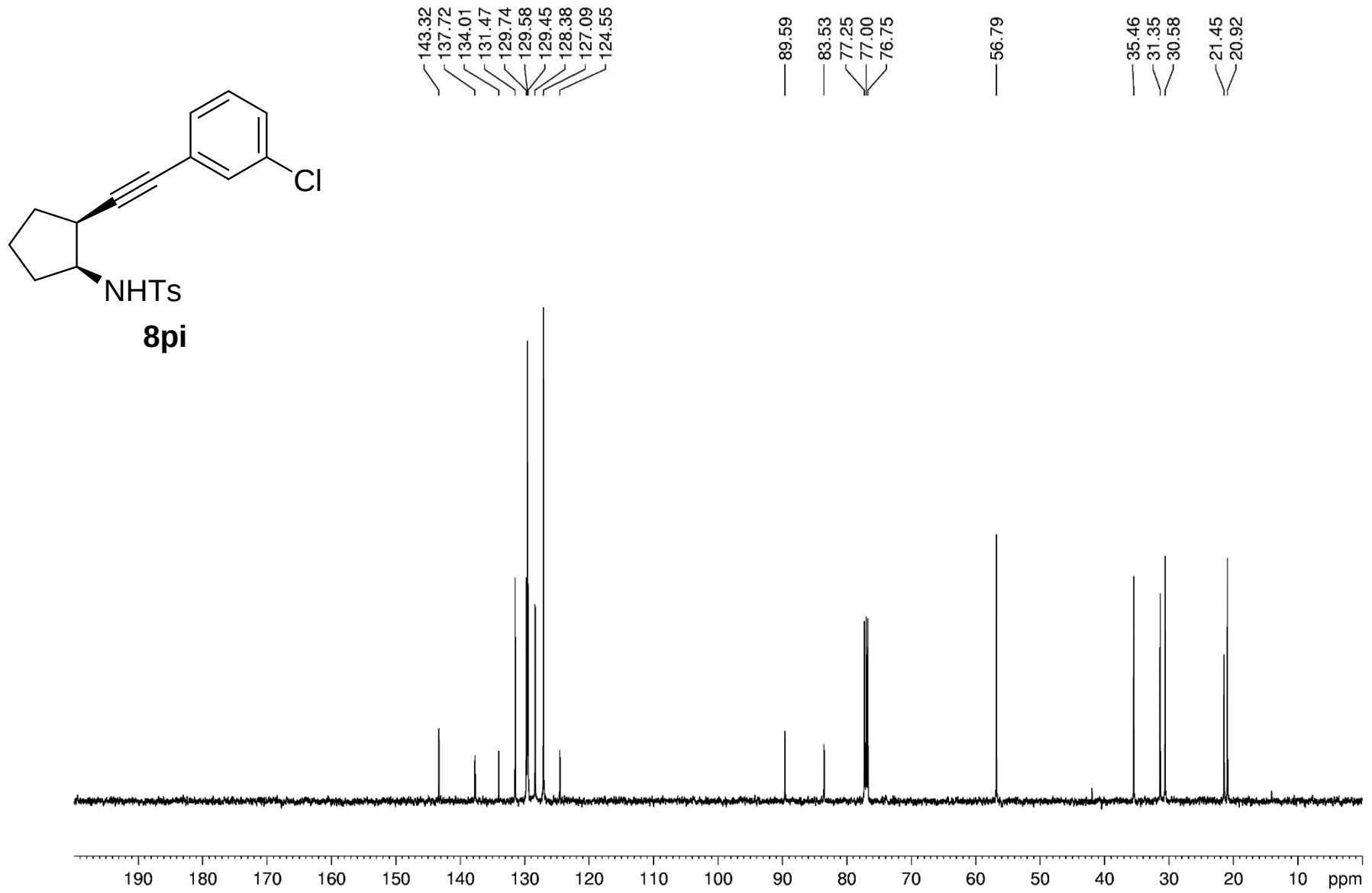


^{13}C NMR of compound **8pc** (125 MHz, $CDCl_3$)

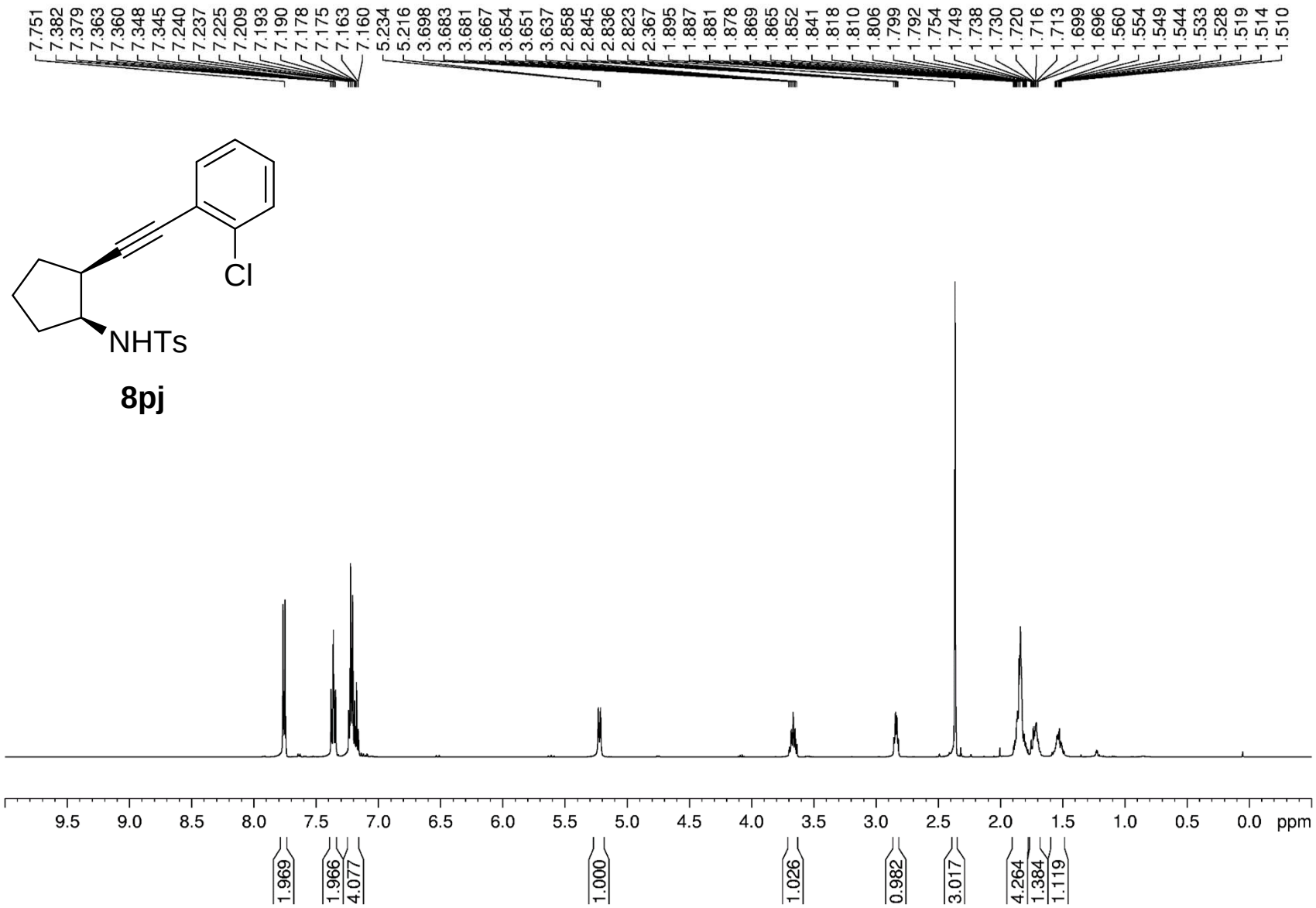


8pi

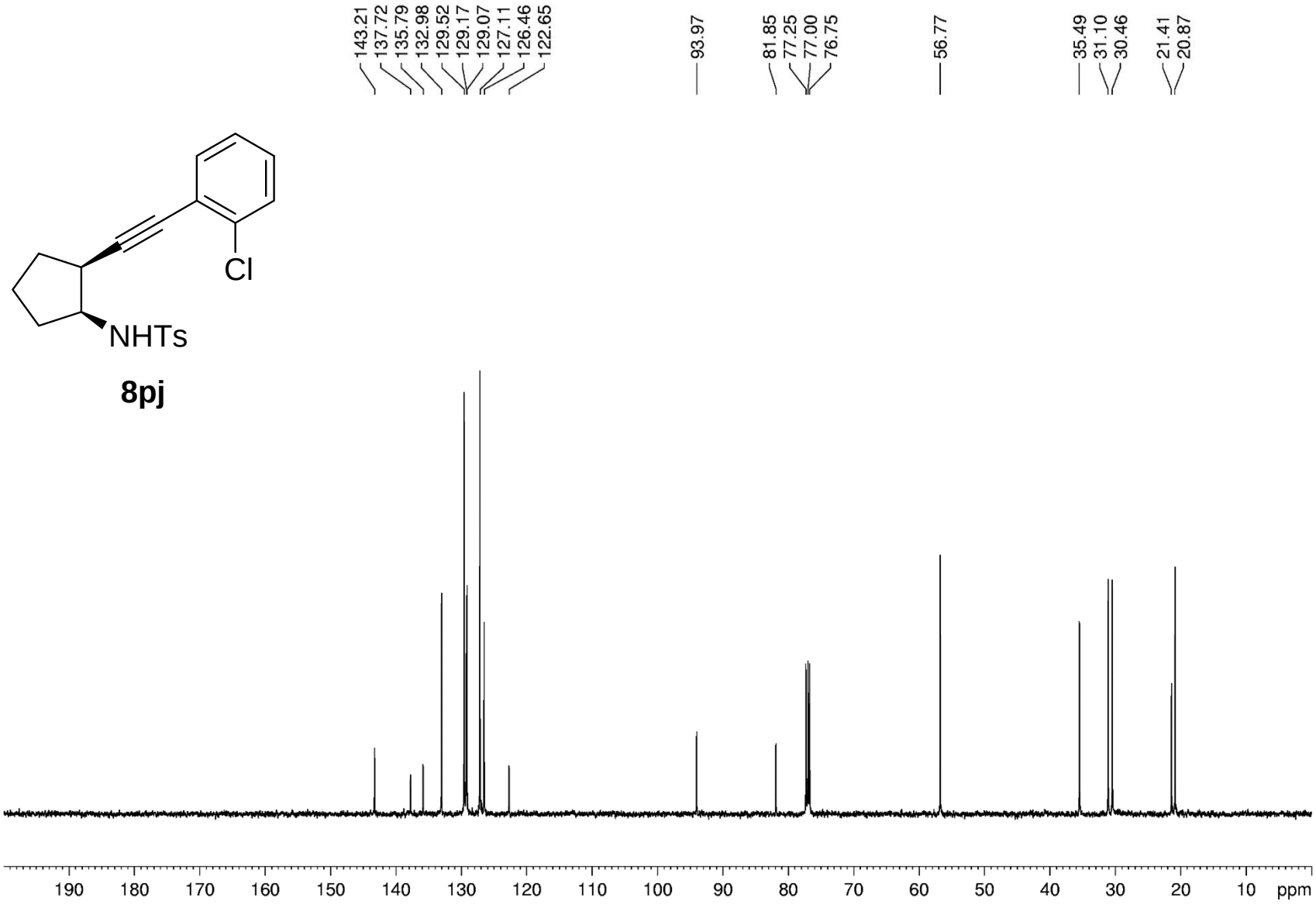
^{1}H NMR of compound **8pi** (500 MHz, $CDCl_3$)



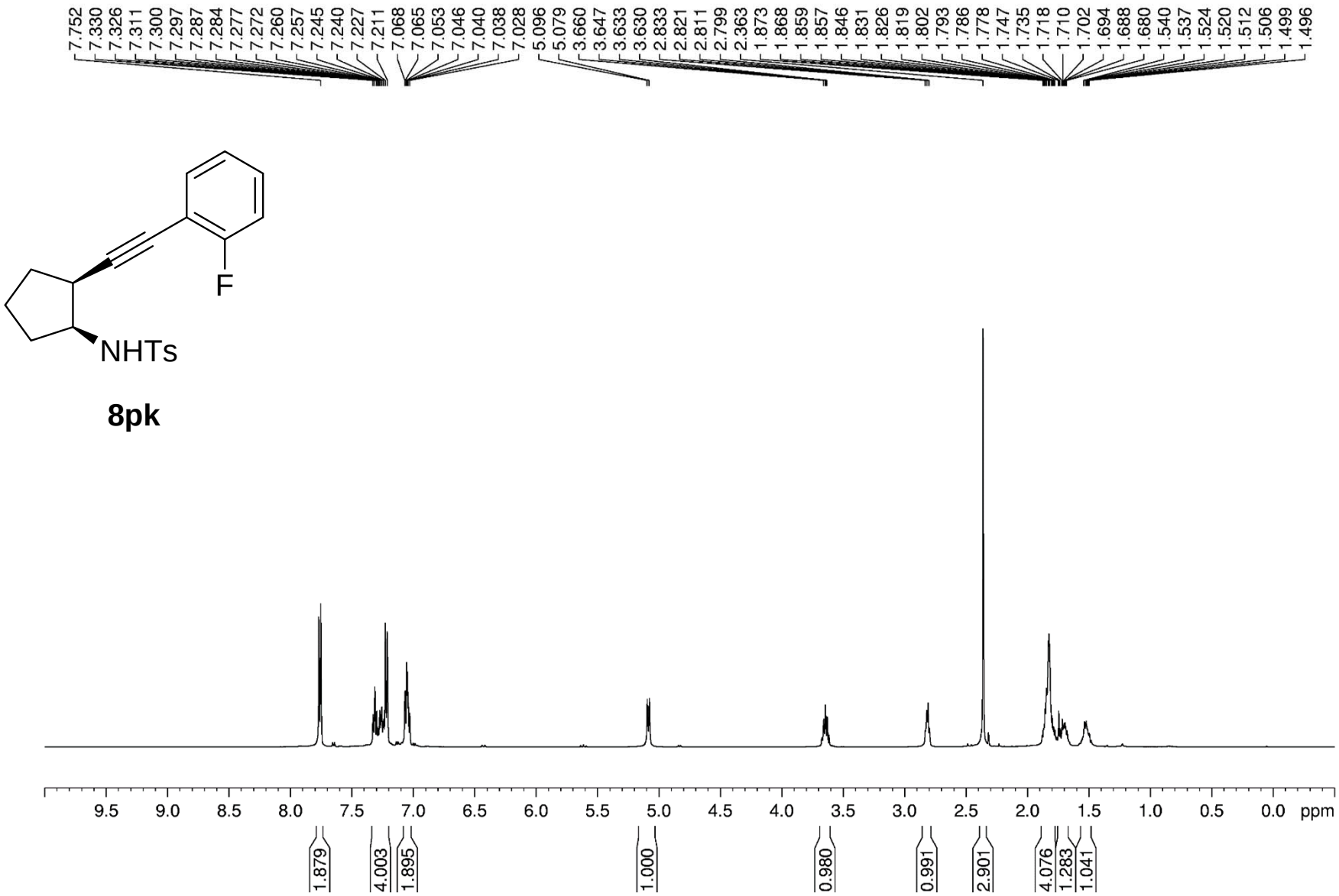
^{13}C NMR of compound **8pi** (125 MHz, $CDCl_3$)



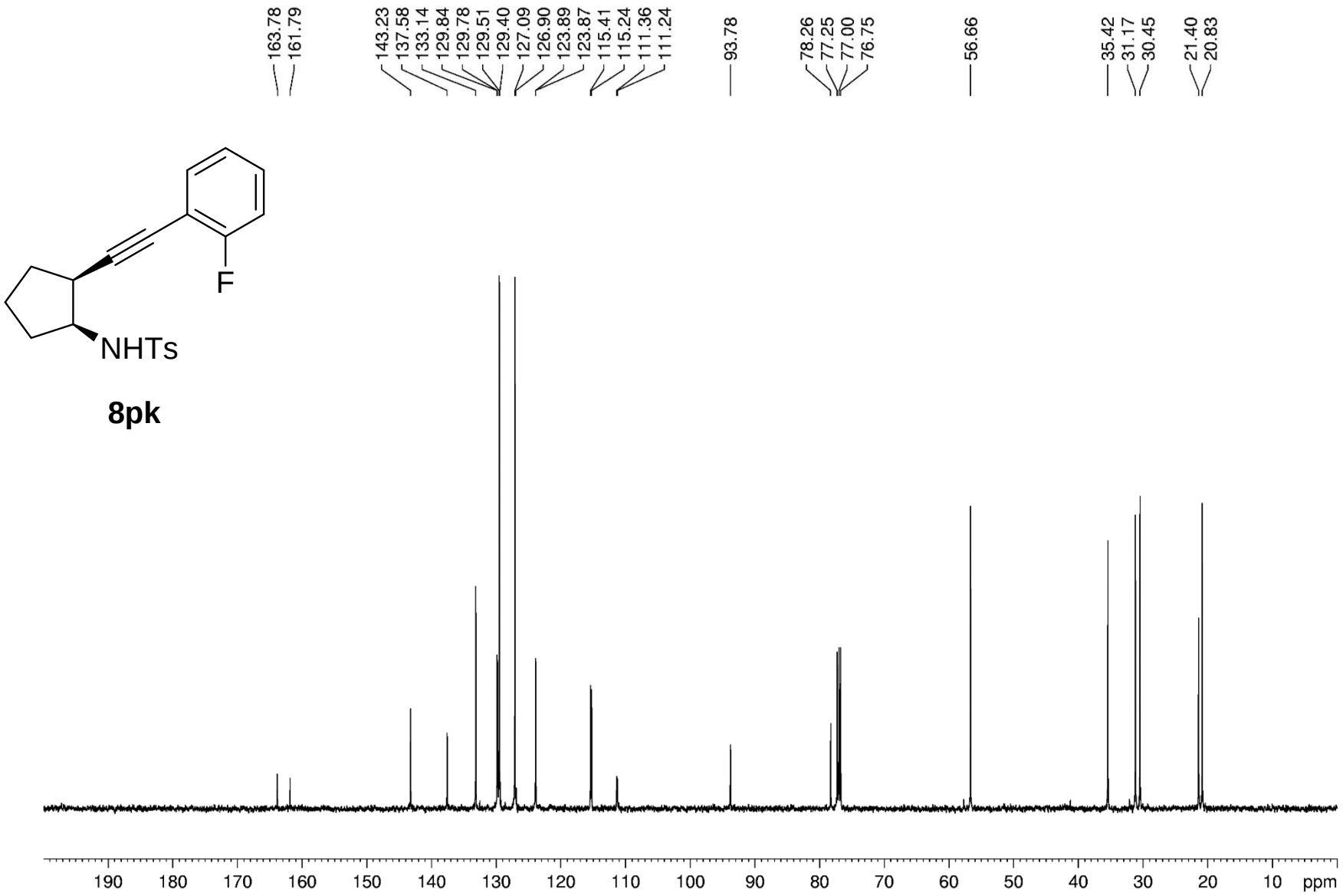
^{1}H NMR of compound **8pj** (500 MHz, $CDCl_3$)



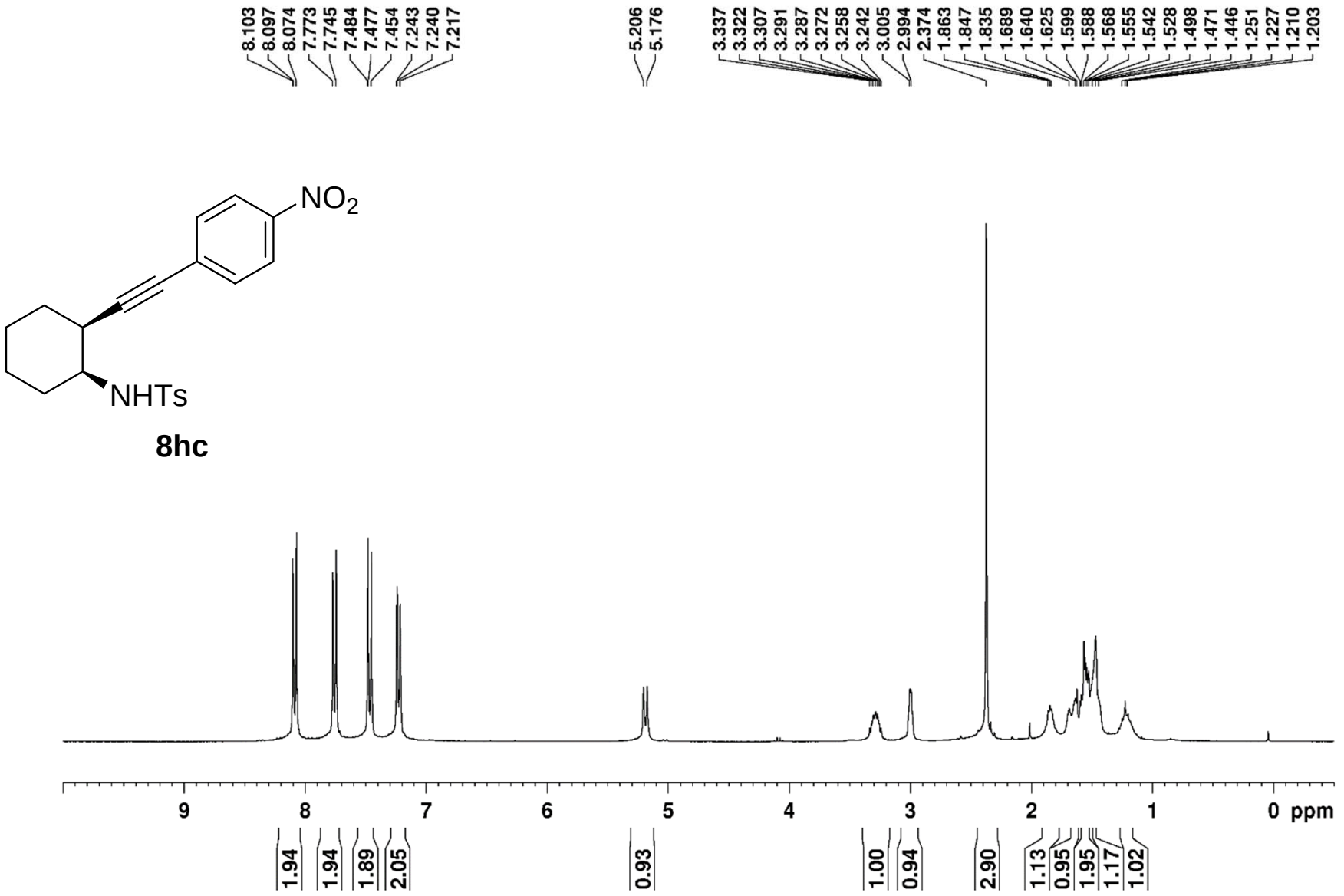
^{13}C NMR of compound **8pj** (125 MHz, $CDCl_3$)



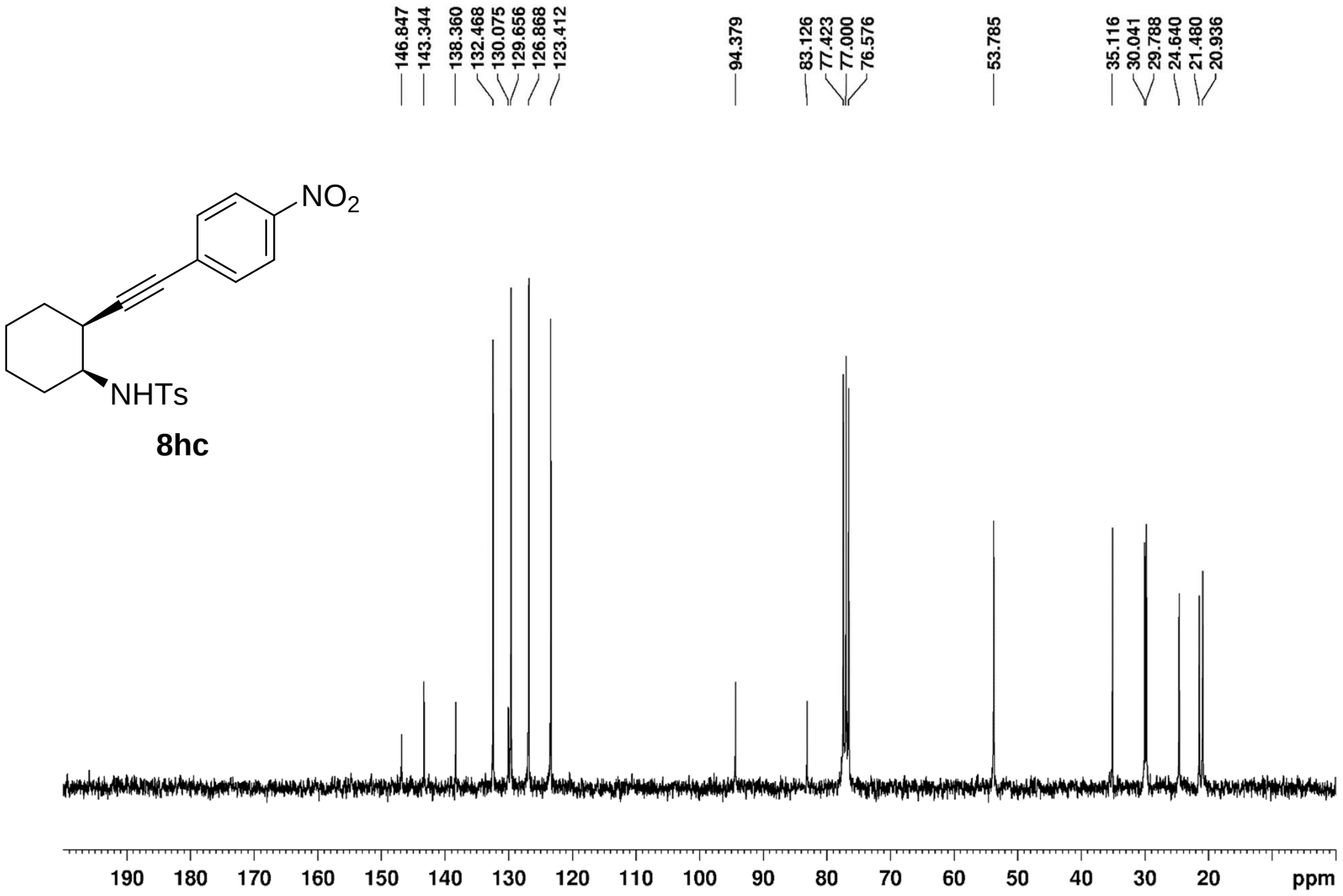
^{1}H NMR of compound **8pk** (500 MHz, $CDCl_3$)



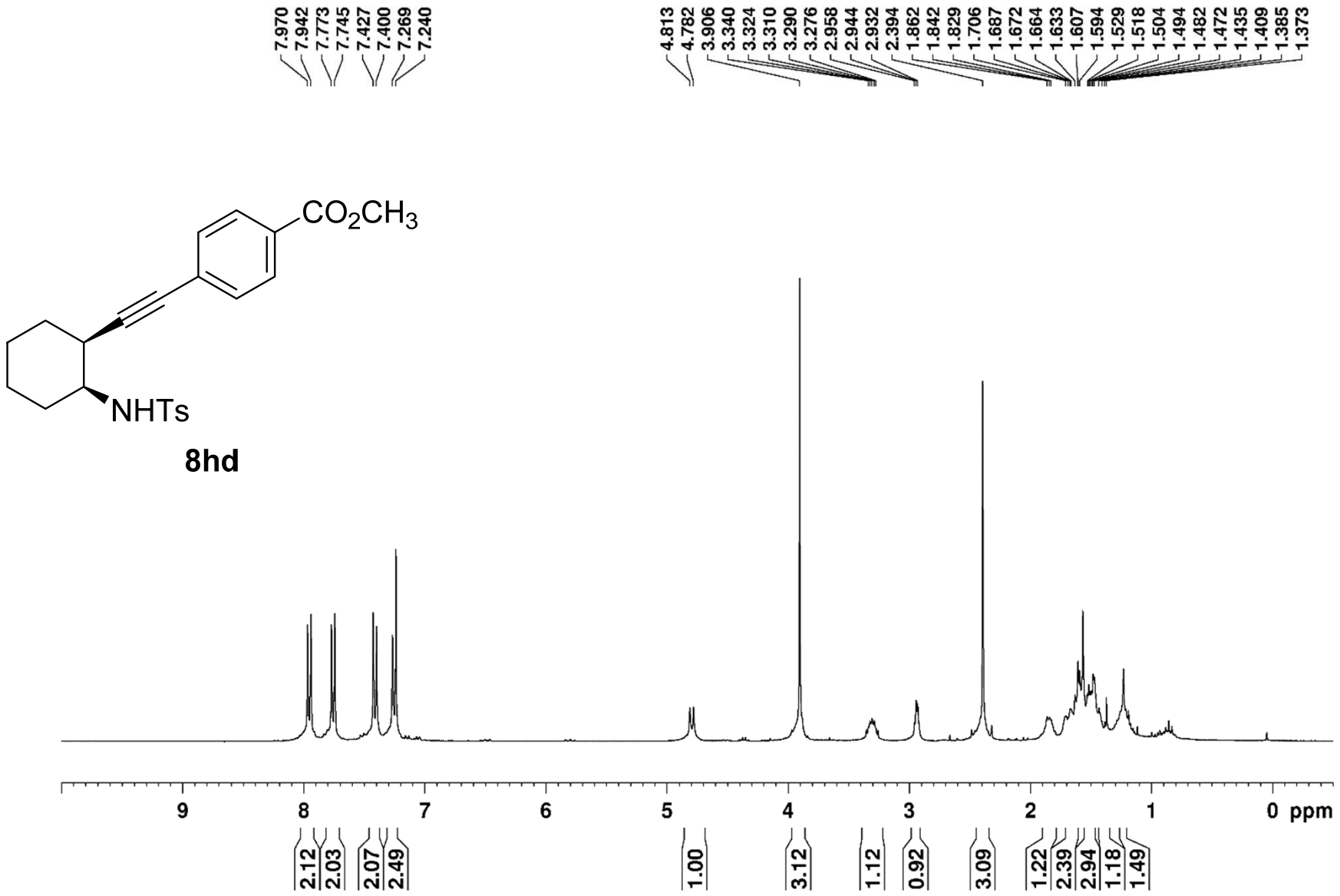
^{13}C NMR of compound **8pk** (125 MHz, $CDCl_3$)



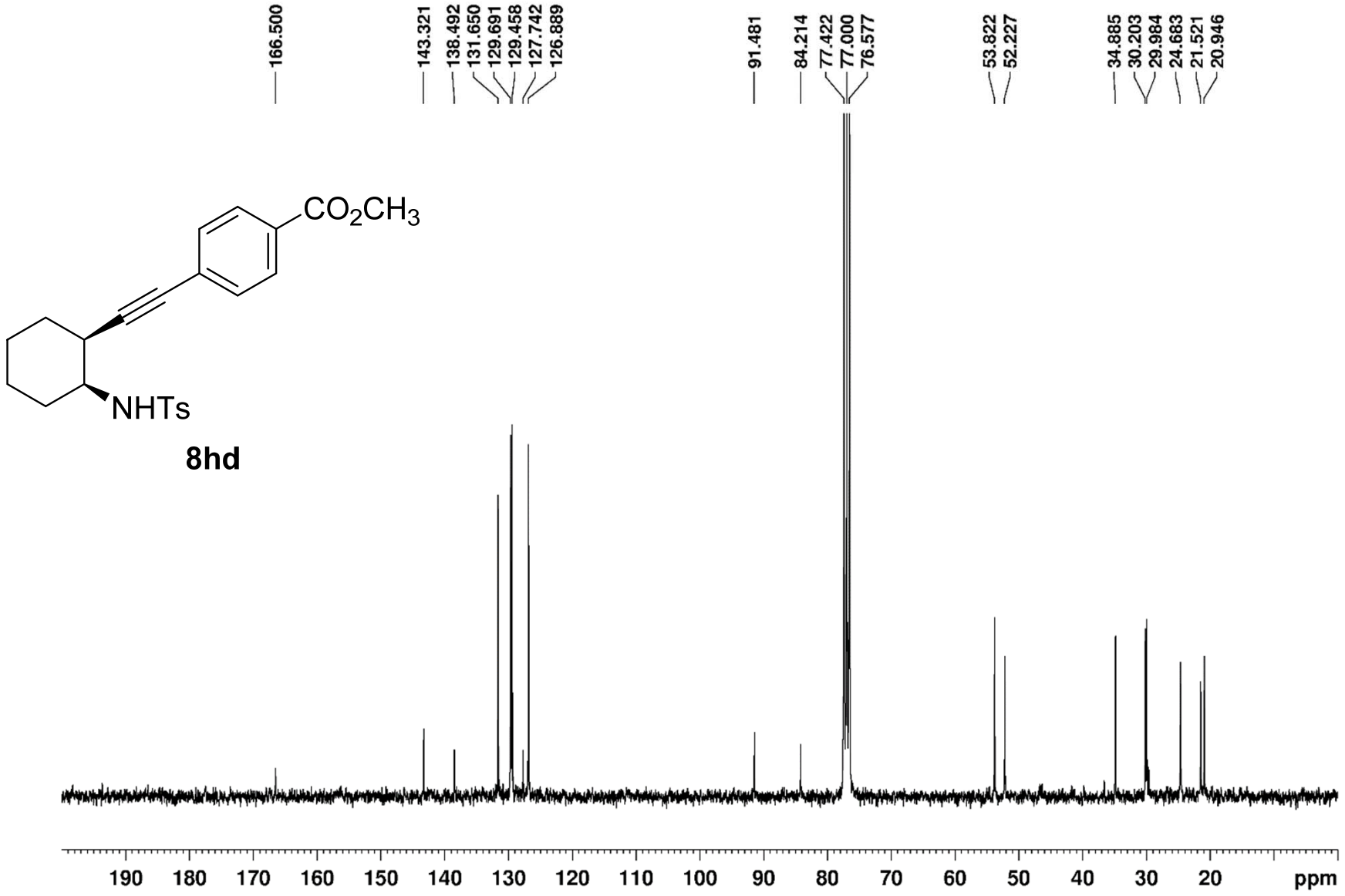
^{1}H NMR of compound **8hc** (300 MHz, $CDCl_3$)



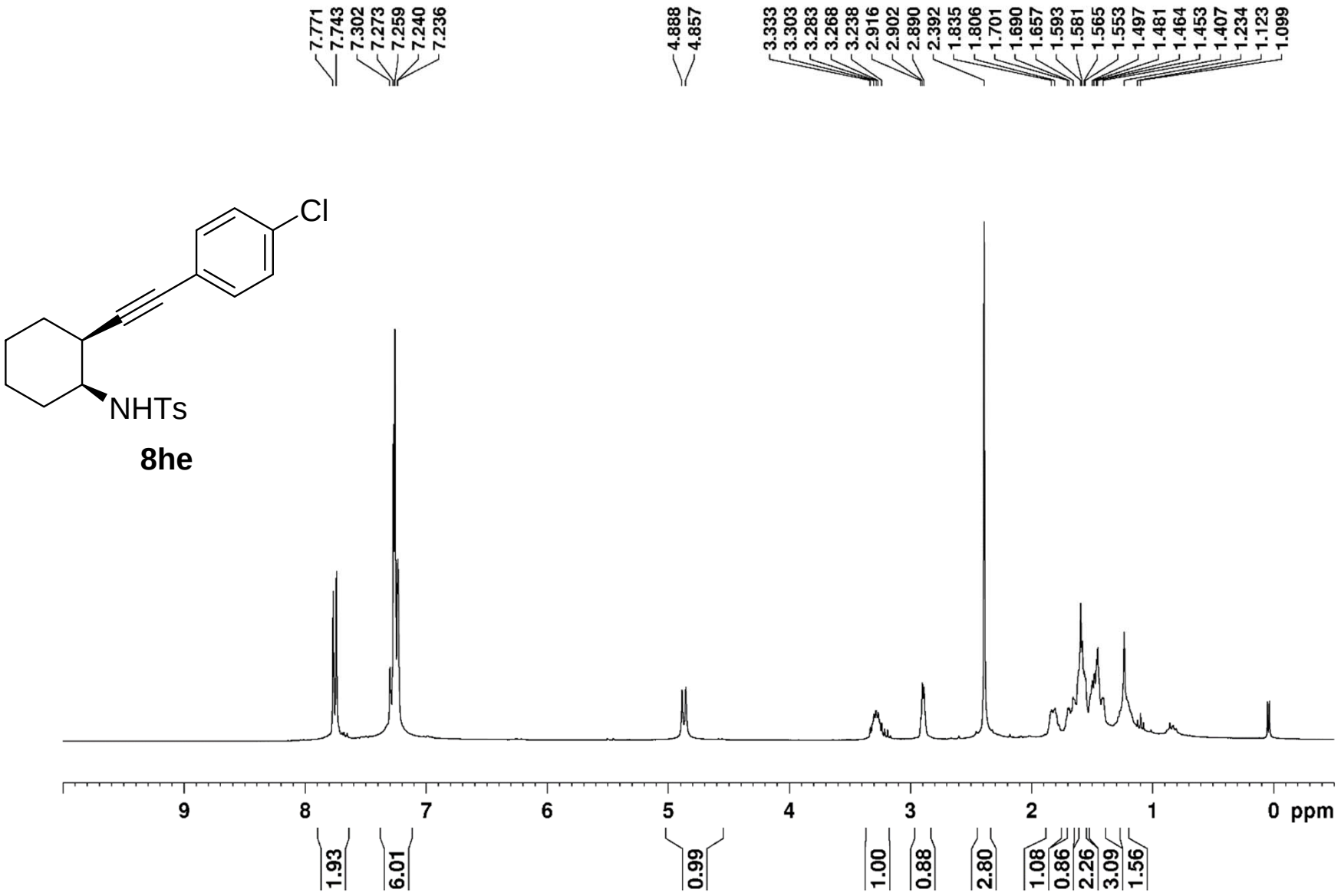
^{13}C NMR of compound **8hc** (75 MHz, $CDCl_3$)



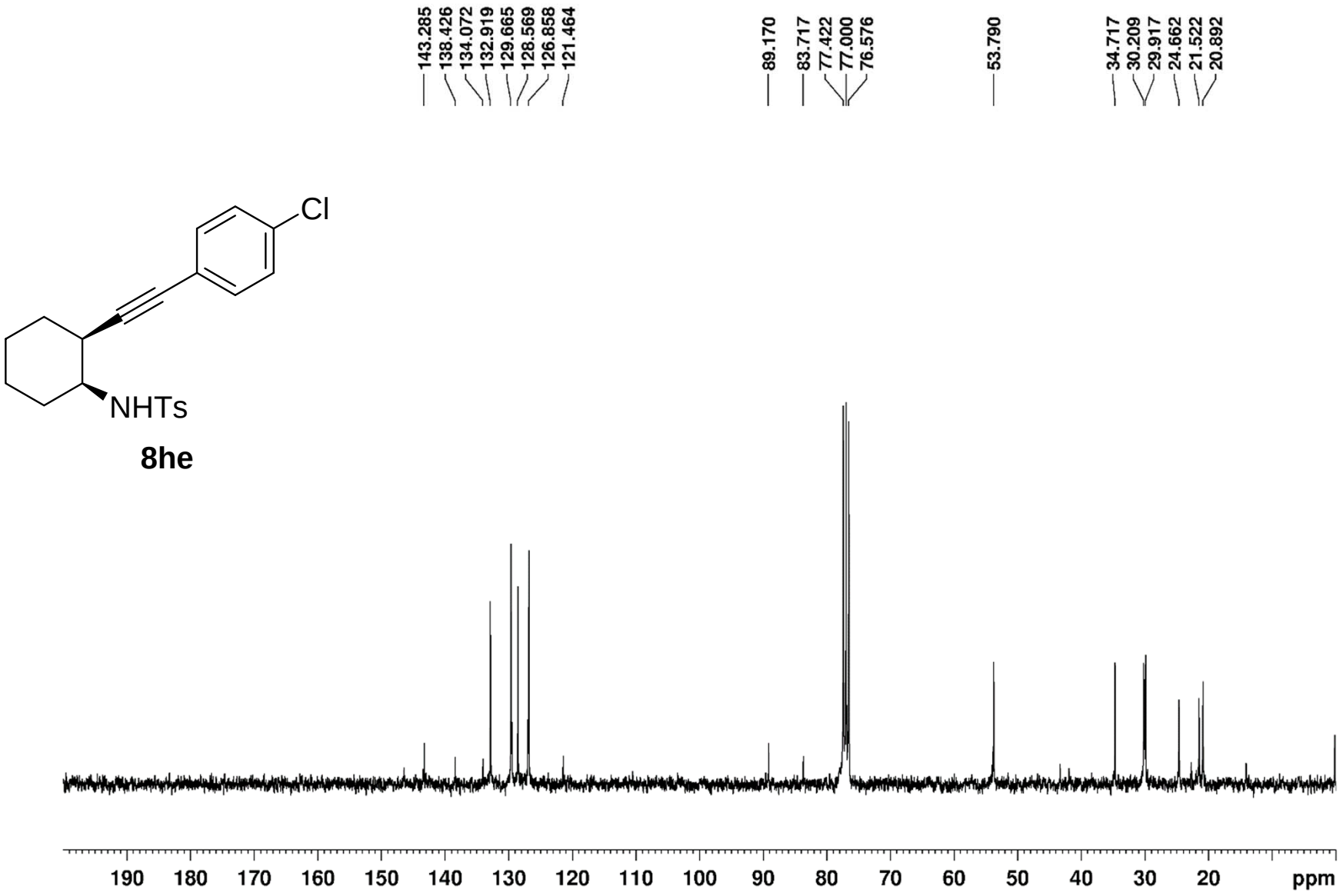
^{1}H NMR of compound **8hd** (300 MHz, $CDCl_3$)



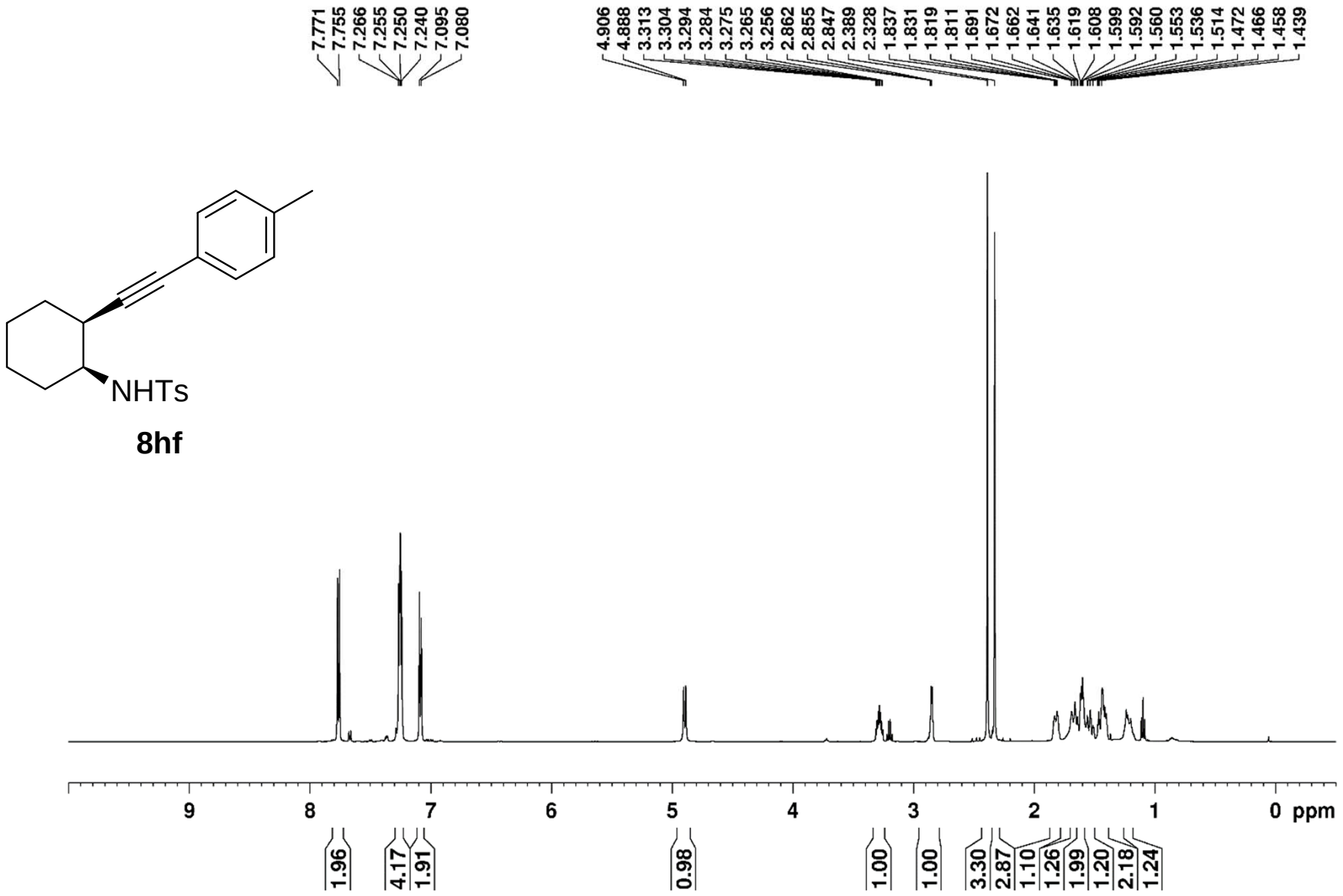
^{13}C NMR of compound **8hd** (75 MHz, $CDCl_3$)



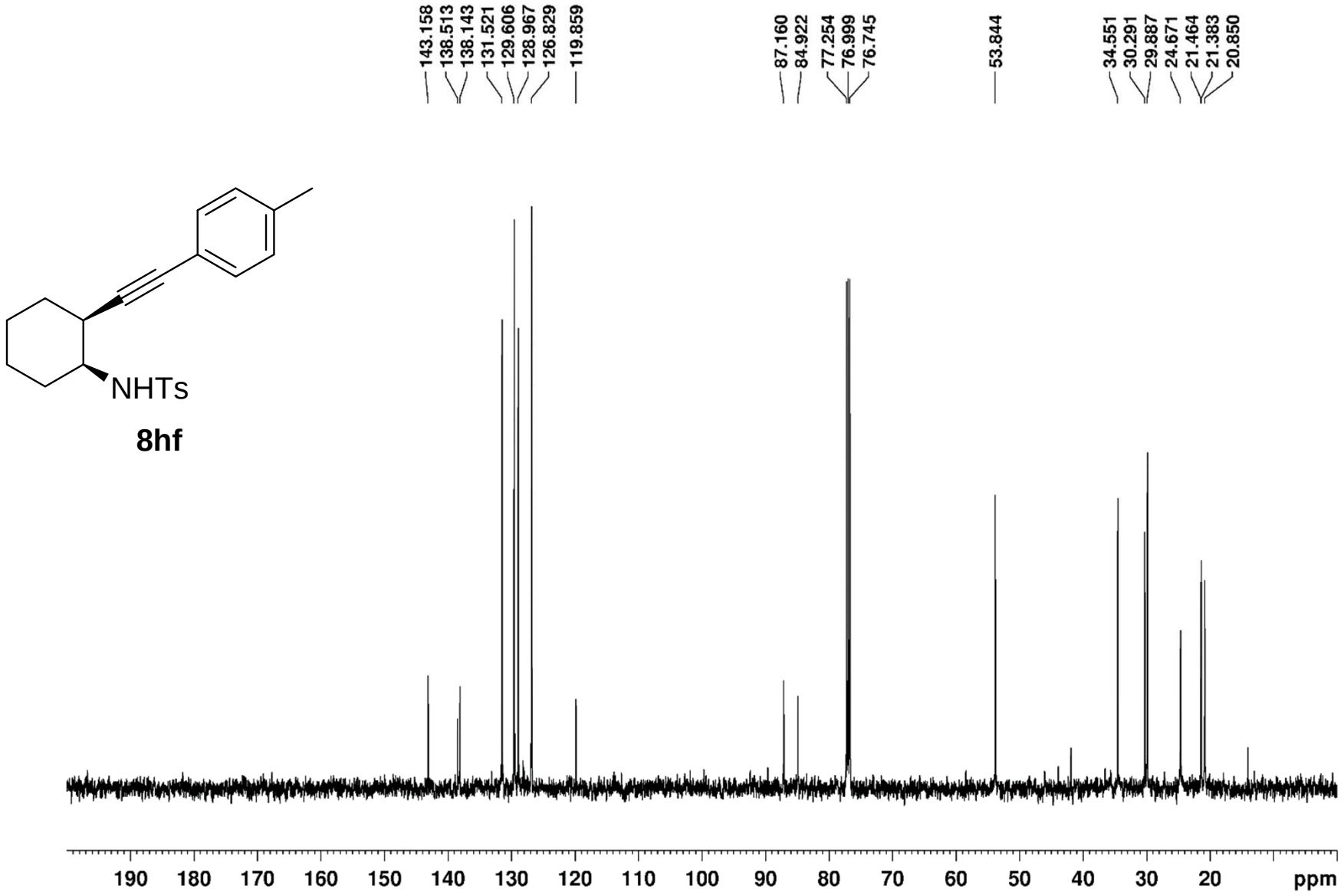
^{1}H NMR of compound **8he** (300 MHz, $CDCl_3$)



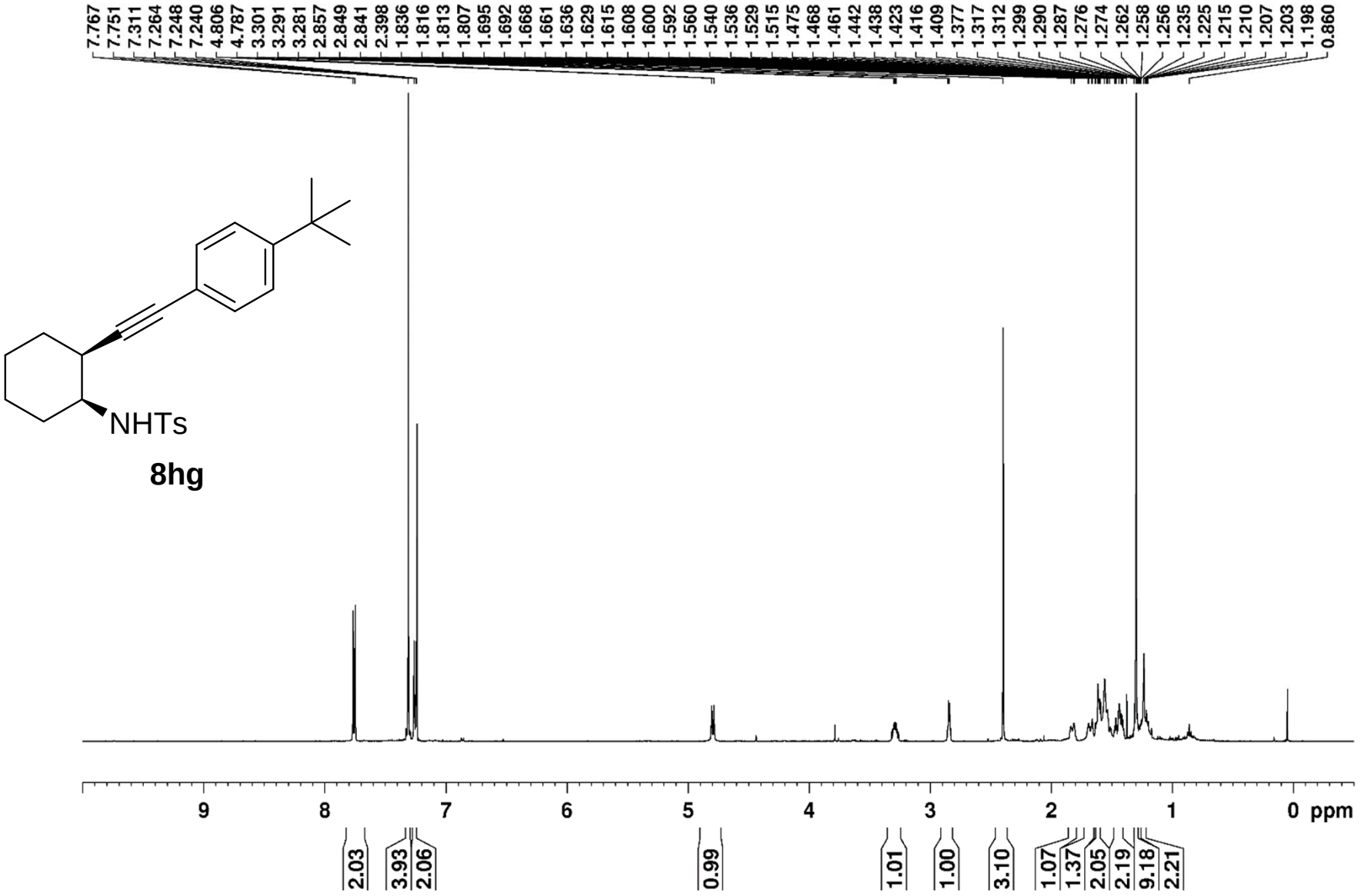
^{13}C NMR of compound **8he** (75 MHz, $CDCl_3$)



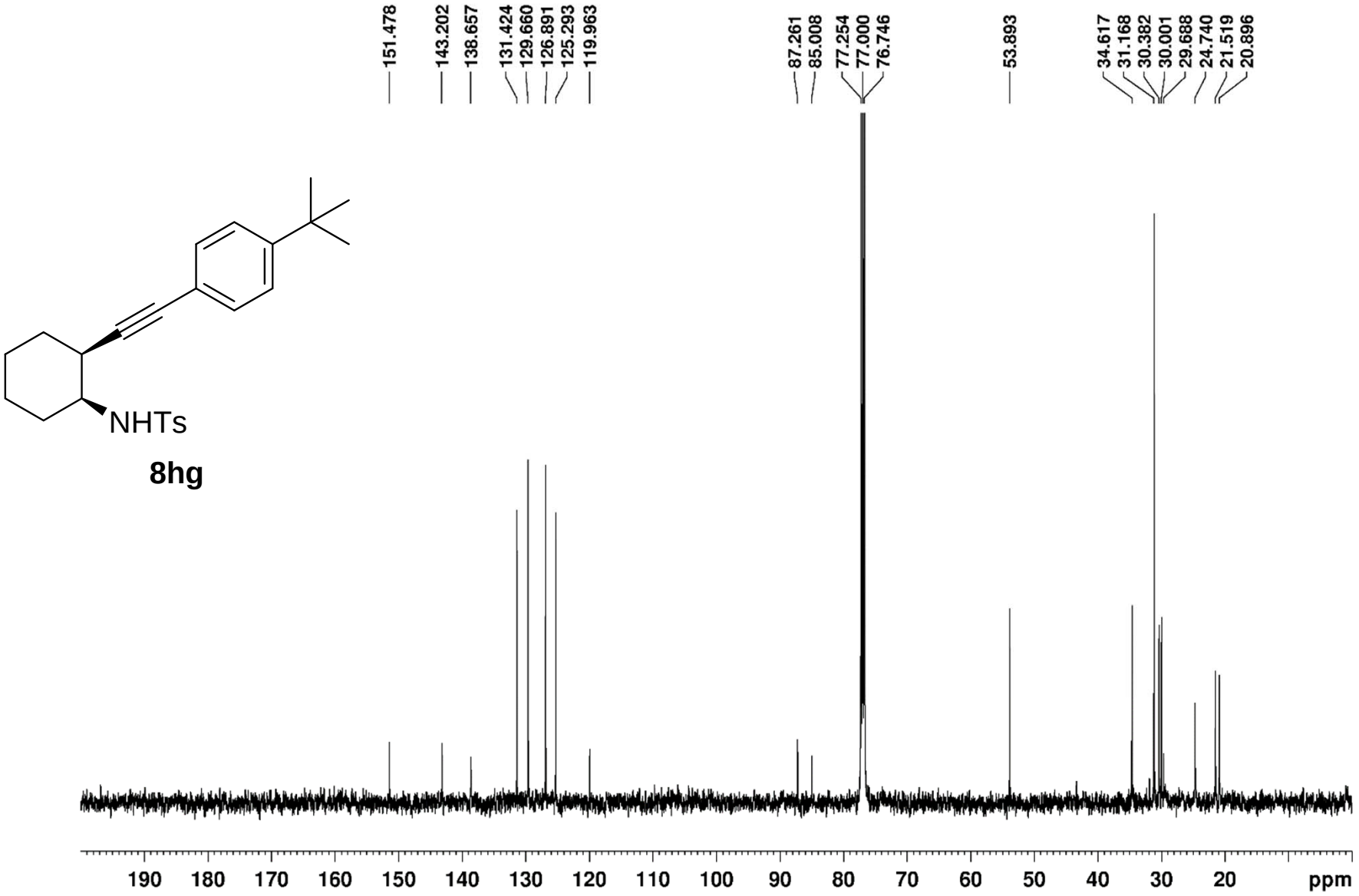
^{1}H NMR of compound **8hf** (500 MHz, $CDCl_3$)



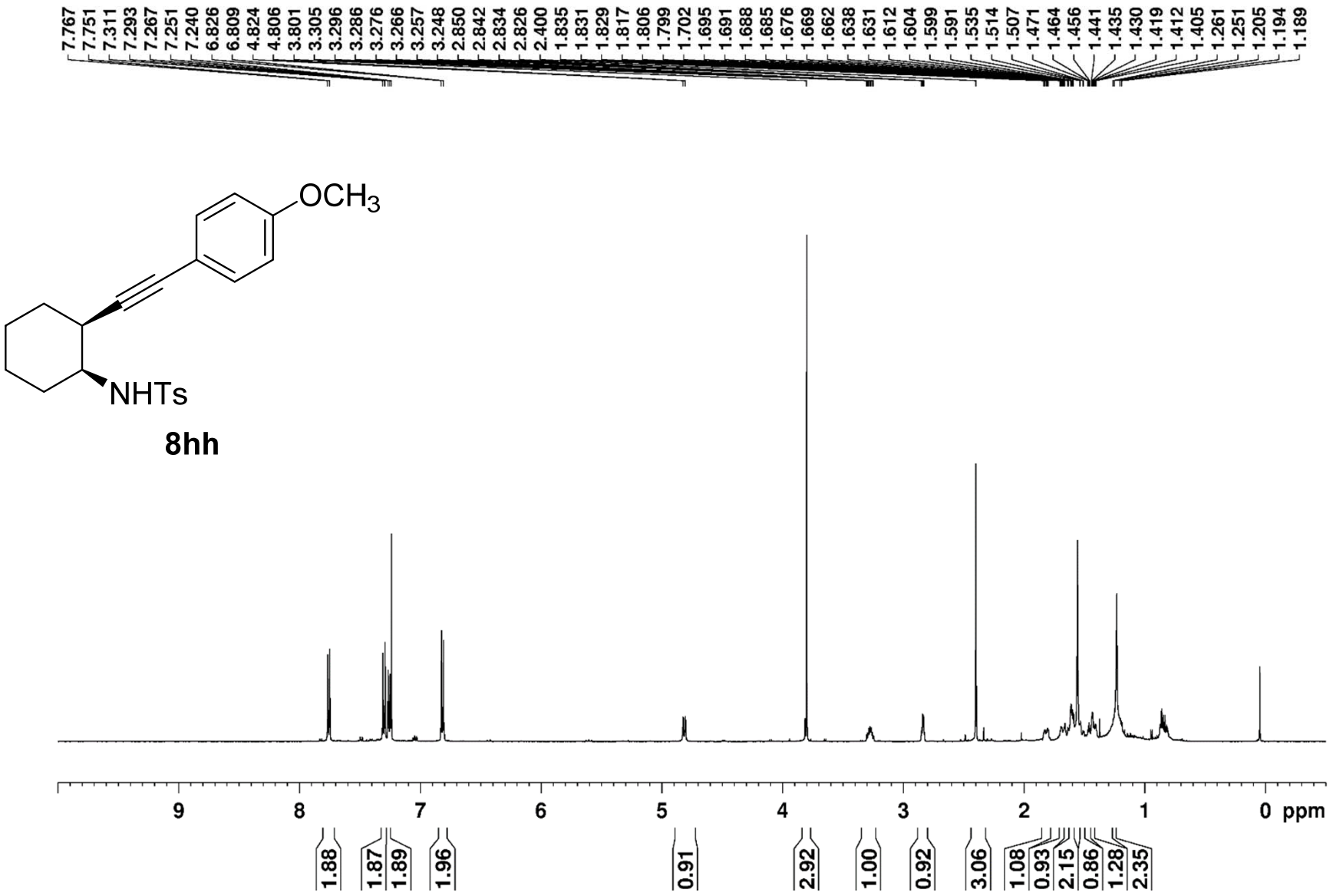
^{13}C NMR of compound **8hf** (125 MHz, $CDCl_3$)



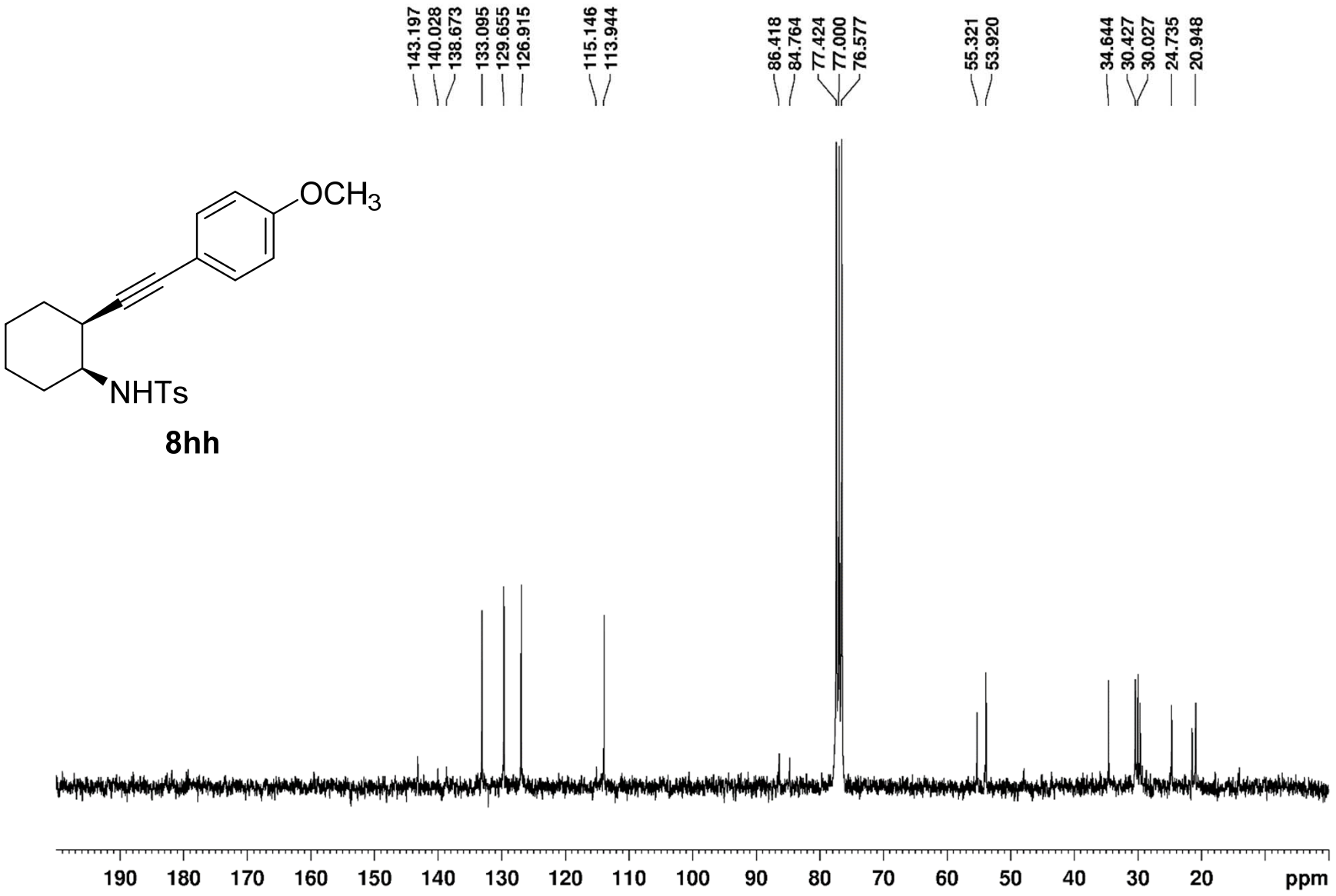
^{1}H NMR of compound **8hg** (500 MHz, $CDCl_3$)



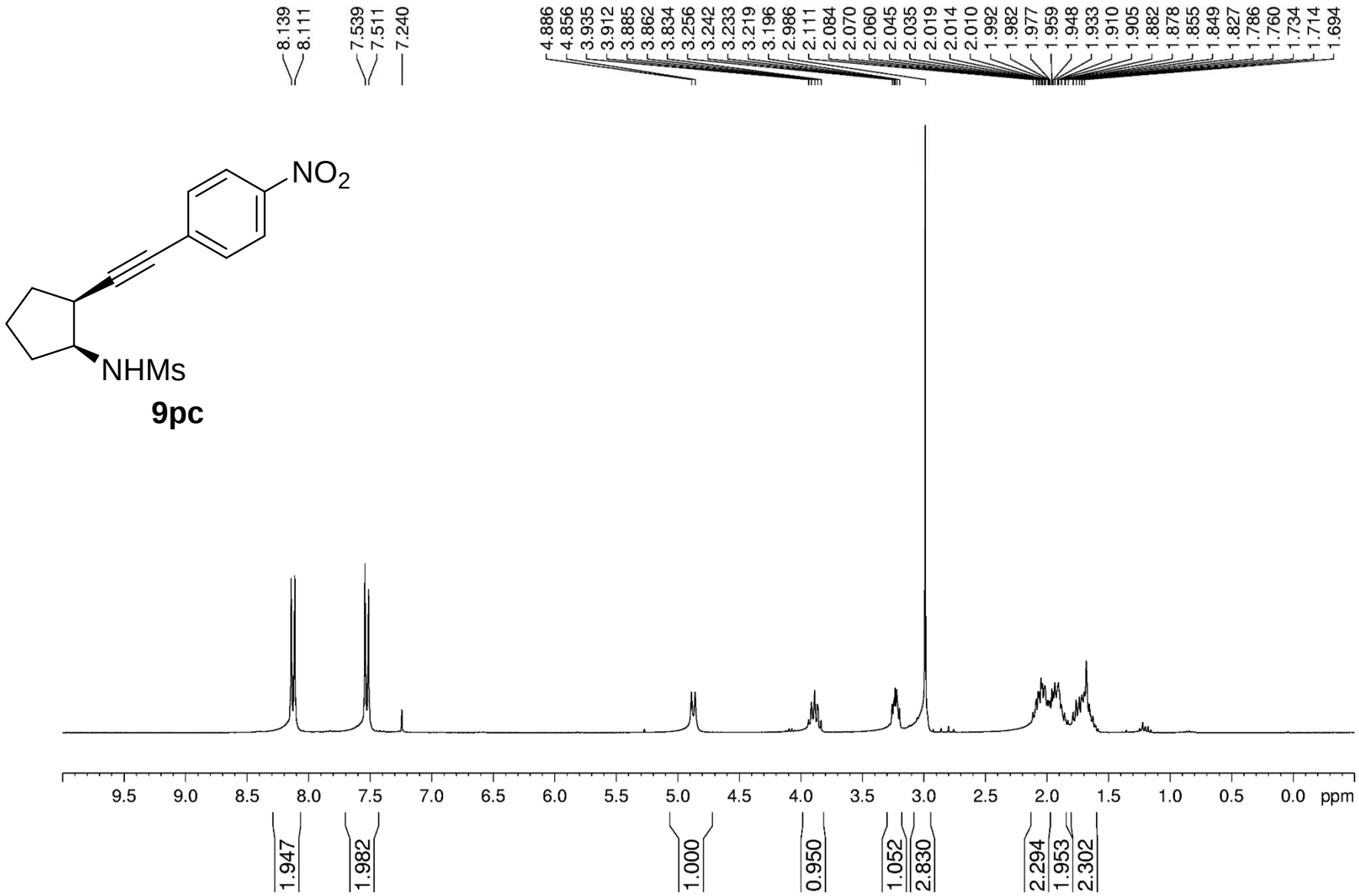
^{13}C NMR of compound **8hg** (125 MHz, $CDCl_3$)



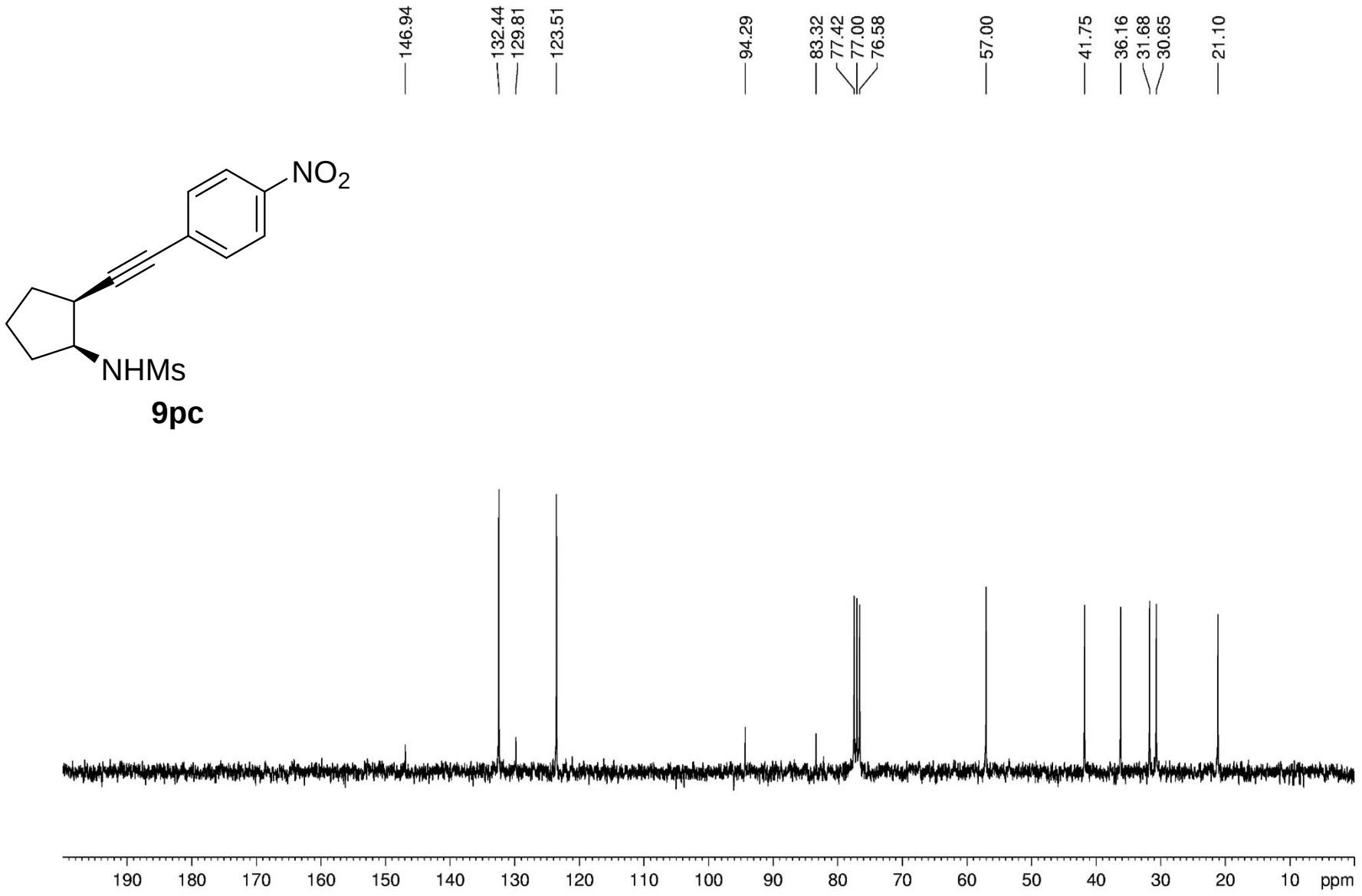
^{1}H NMR of compound **8hh** (500 MHz, $CDCl_3$)



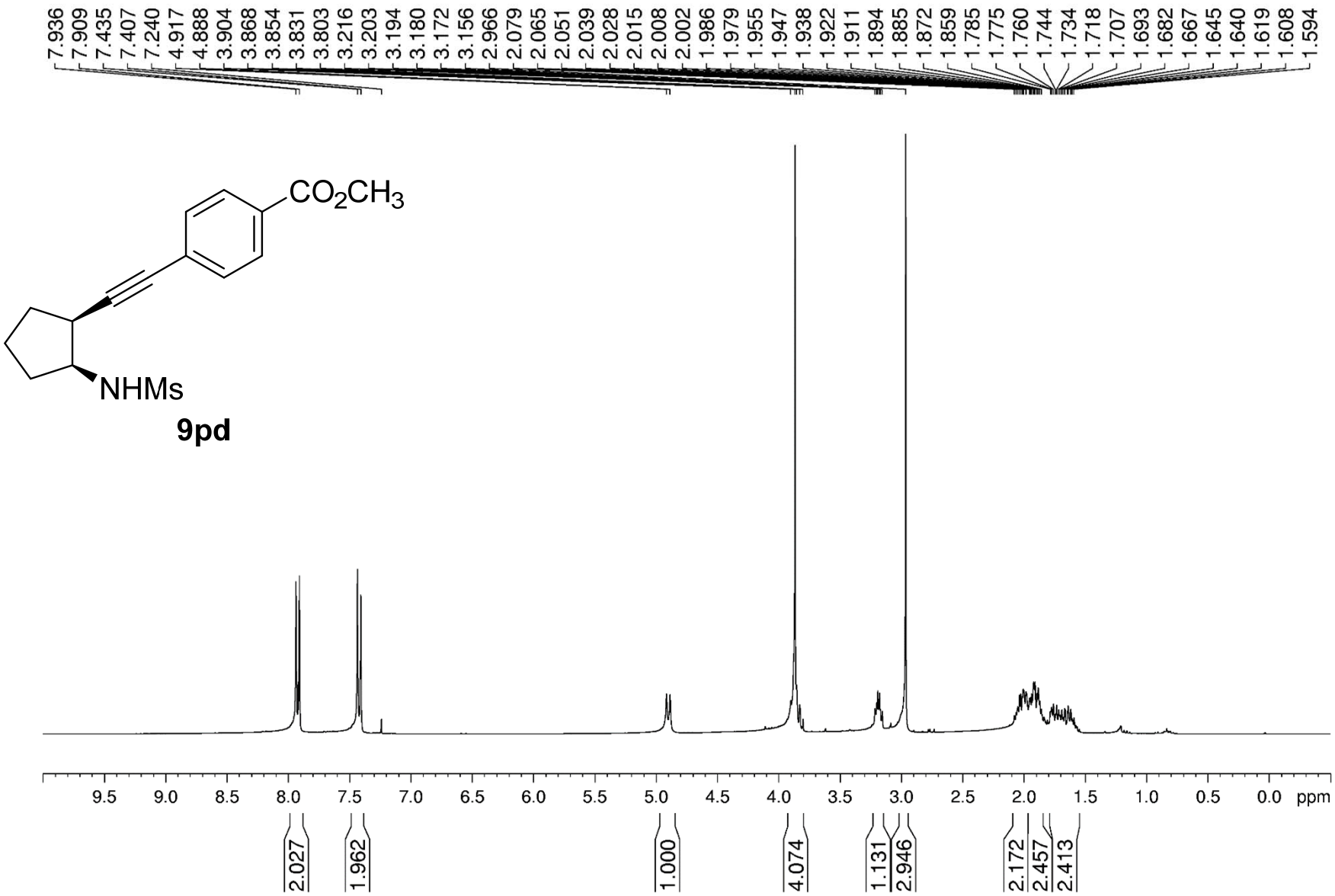
^{13}C NMR of compound **8hh** (75 MHz, $CDCl_3$)



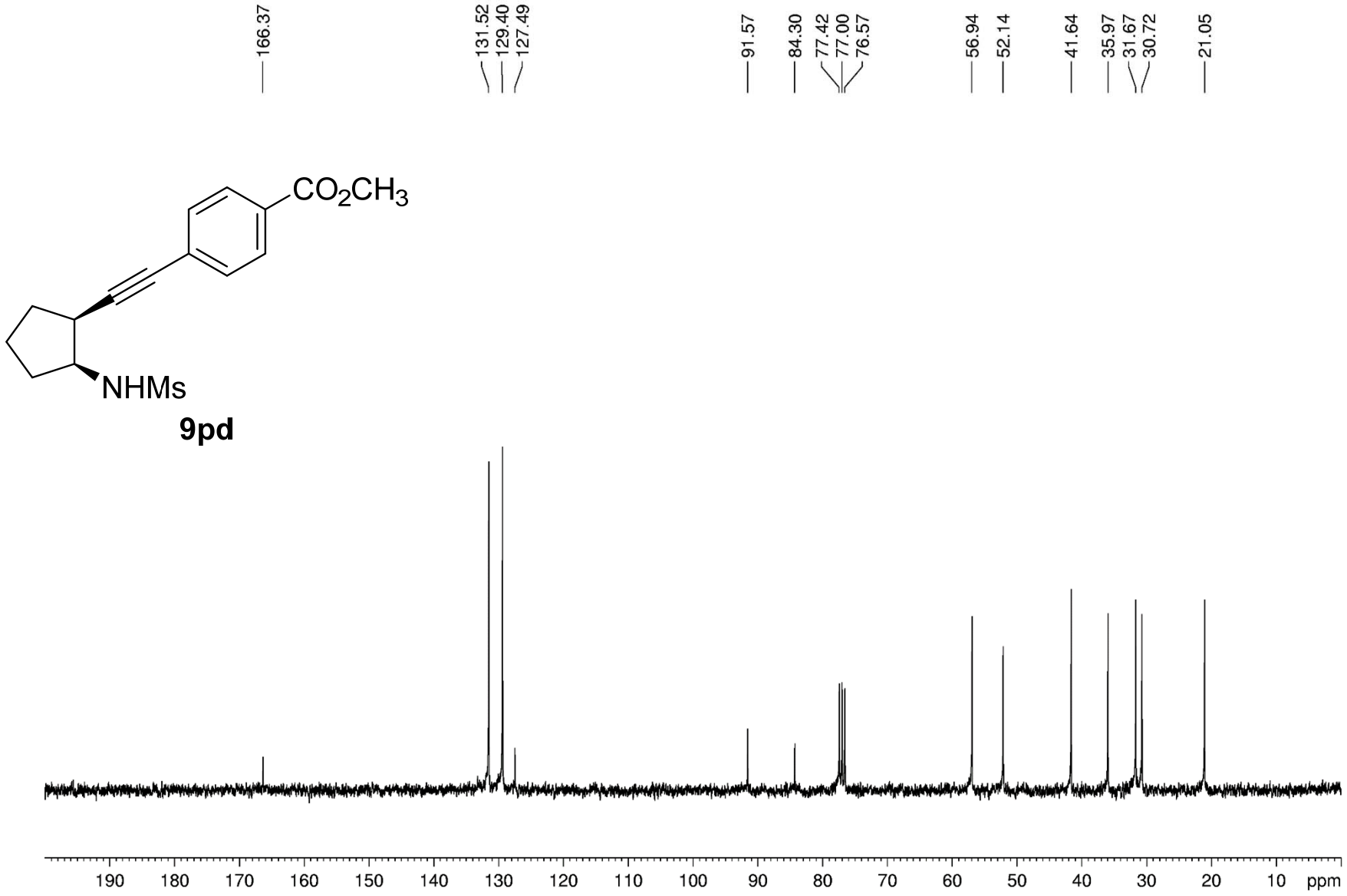
^{1}H NMR of compound **9pc** (300 MHz, $CDCl_3$)



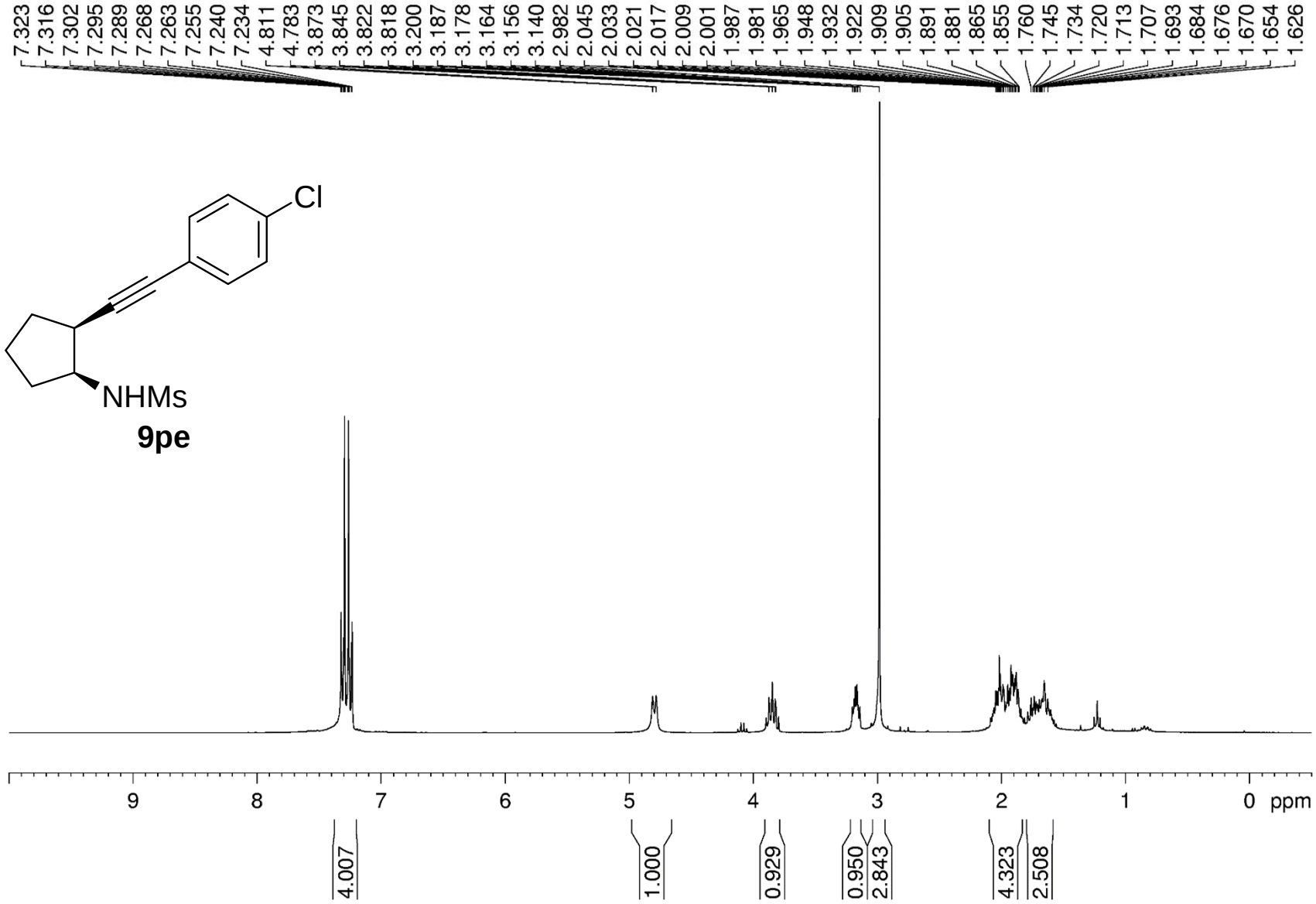
^{13}C NMR of compound **9pc** (75 MHz, $CDCl_3$)



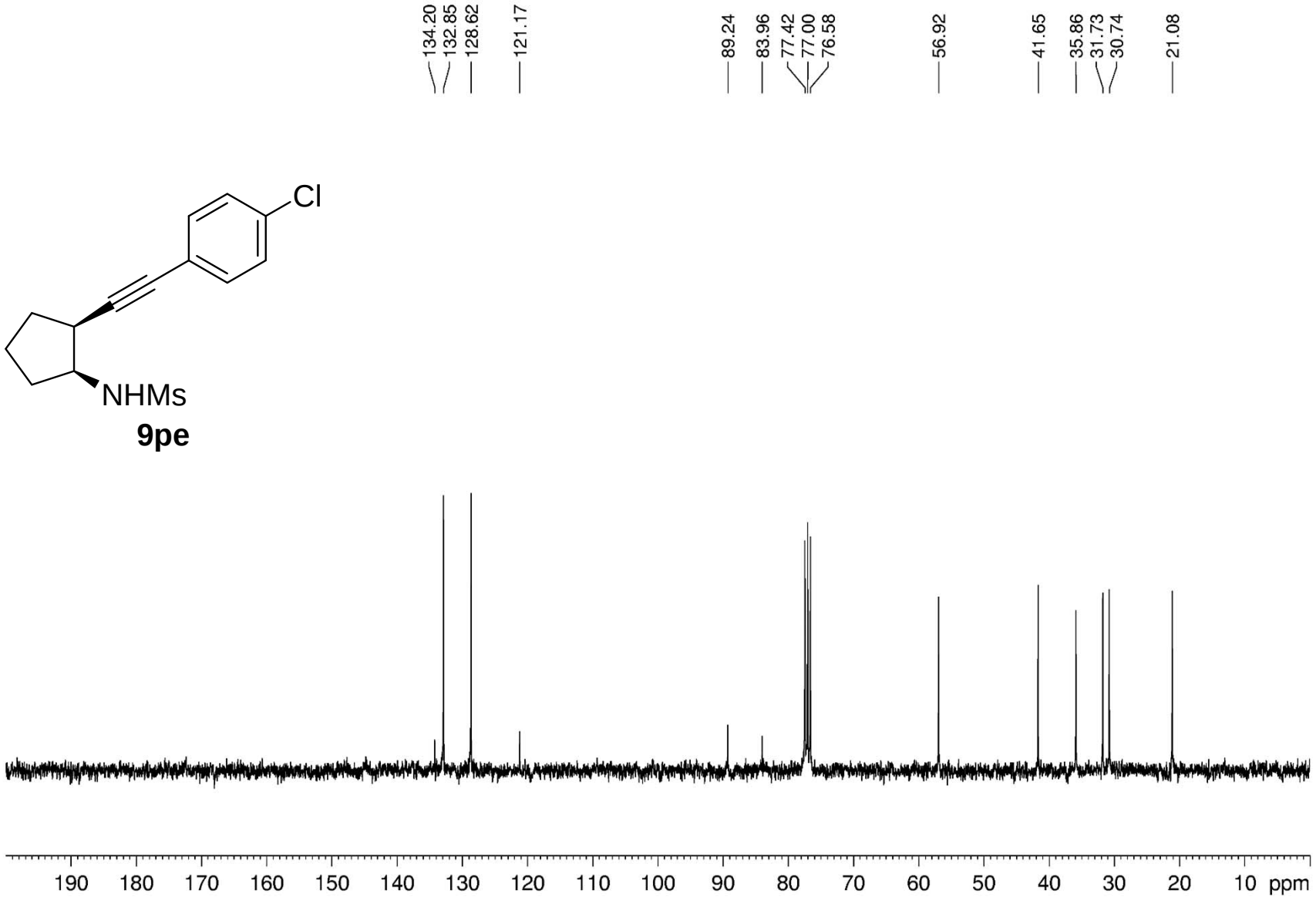
^{1}H NMR of compound **9pd** (300 MHz, $CDCl_3$)



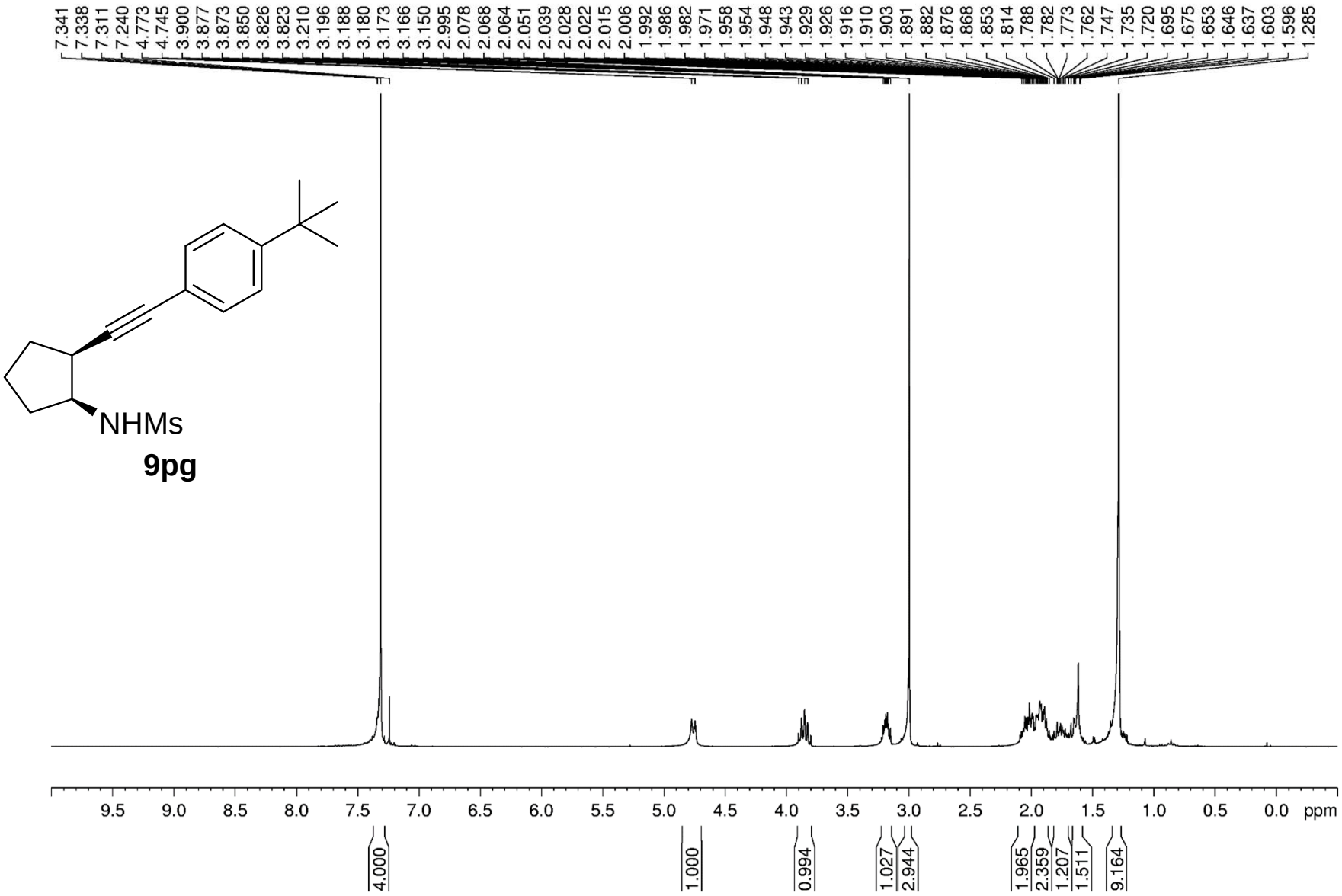
^{13}C NMR of compound **9pd** (75 MHz, $CDCl_3$)



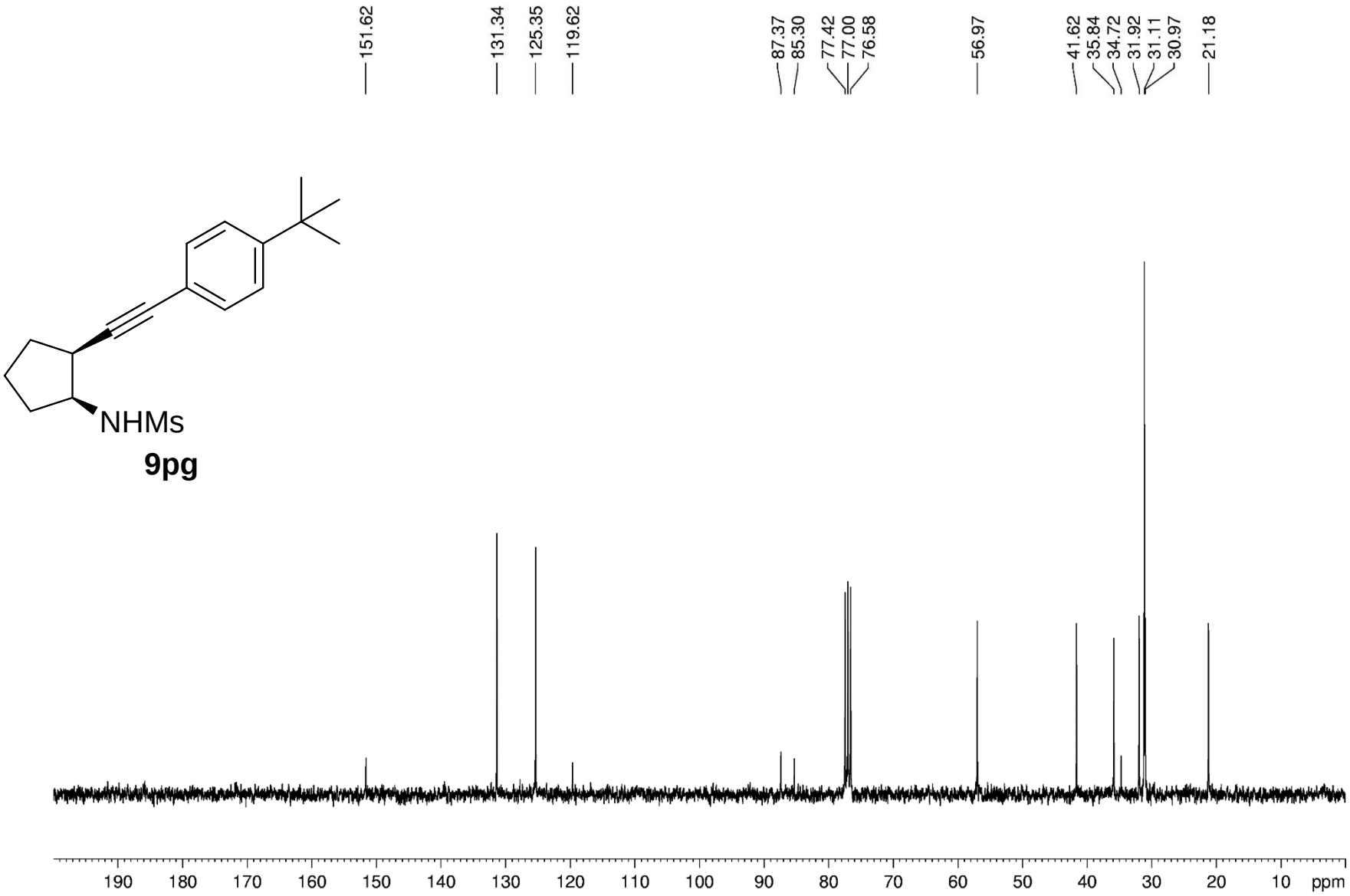
^{1}H NMR of compound **9pe** (300 MHz, $CDCl_3$)



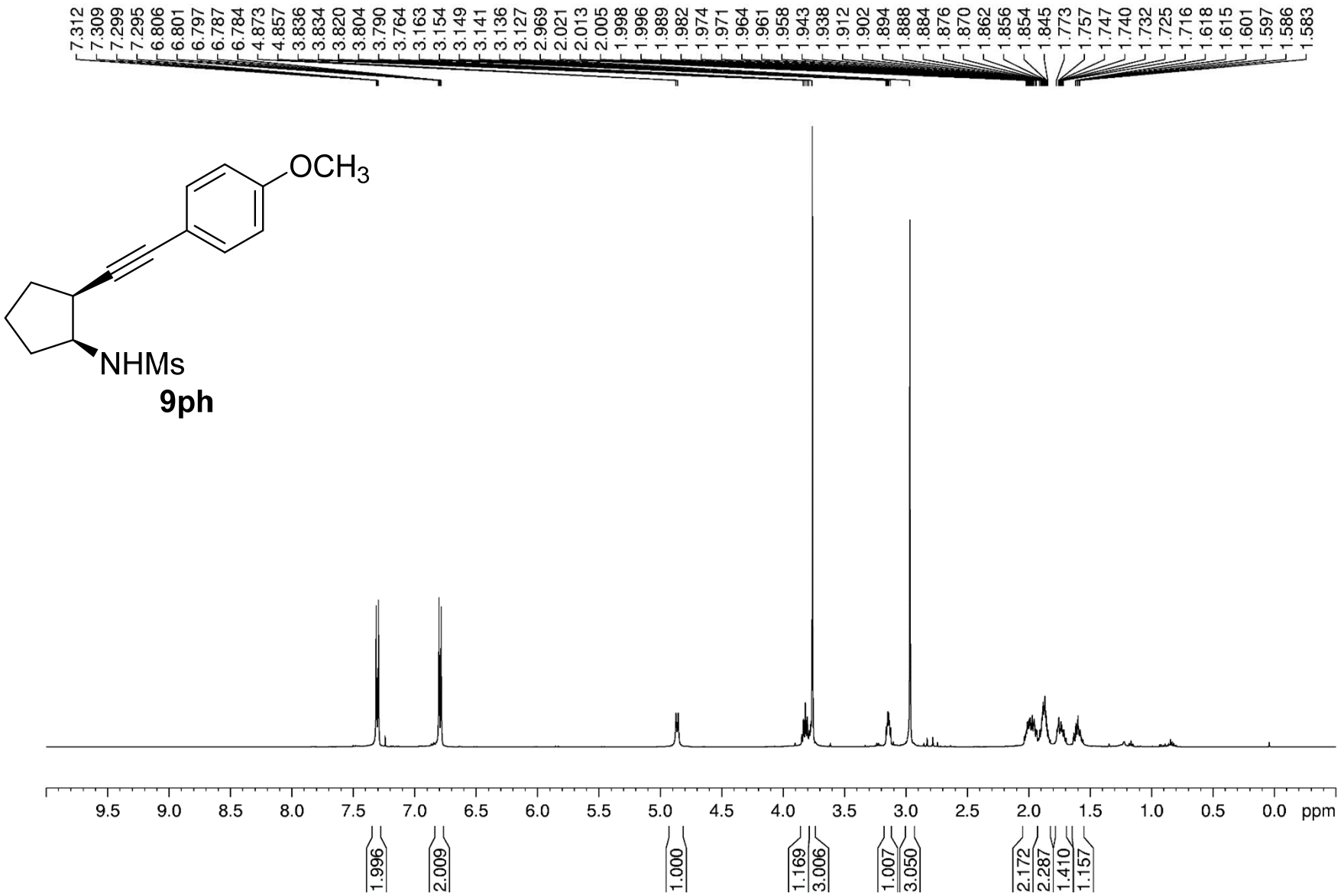
^{13}C NMR of compound **9pe** (75 MHz, $CDCl_3$)



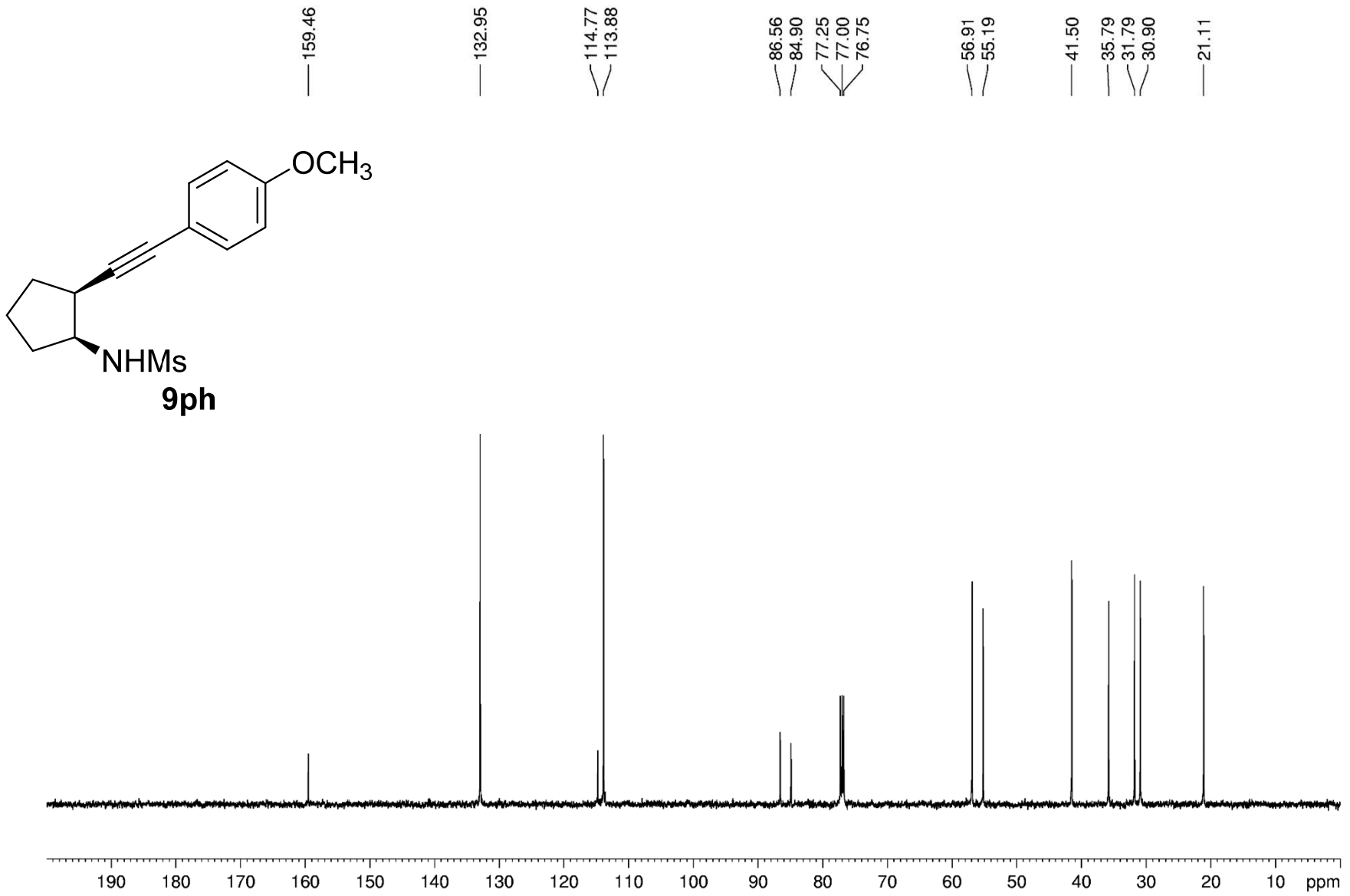
^{1}H NMR of compound **9pg** (300 MHz, $CDCl_3$)



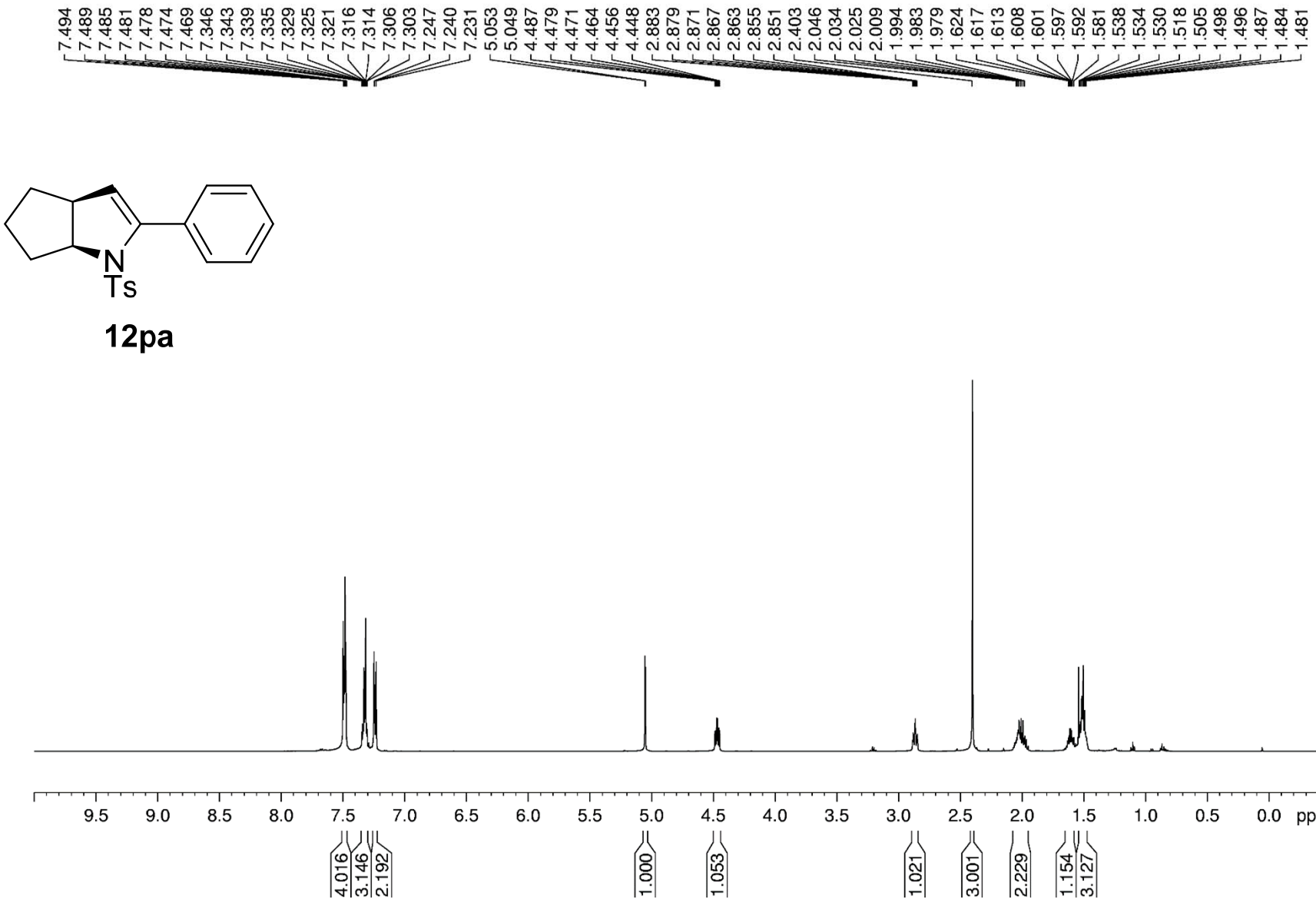
^{13}C NMR of compound **9pg** (75 MHz, $CDCl_3$)



^{1}H NMR of compound **9ph** (500 MHz, $CDCl_3$)

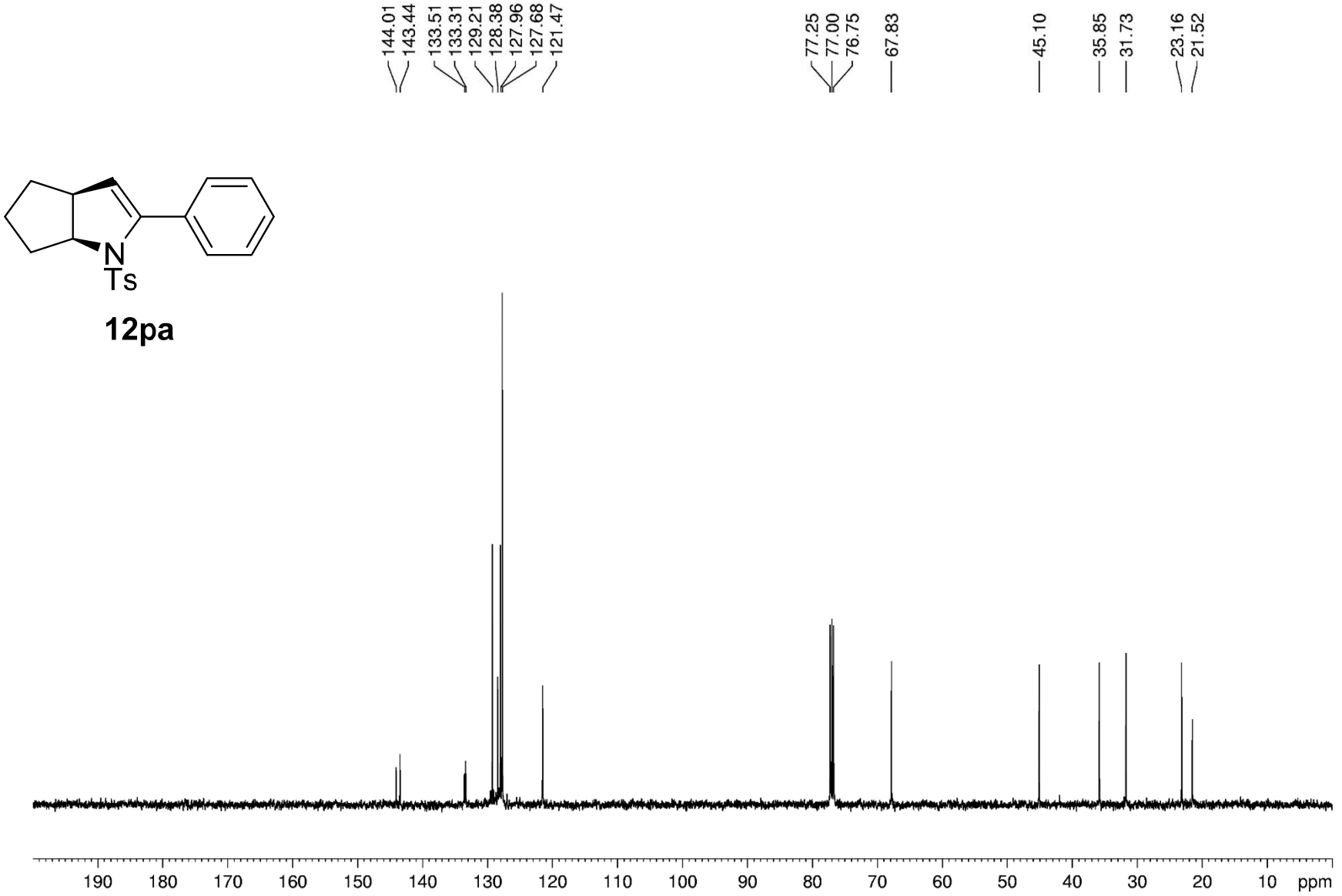


^{13}C NMR of compound **9ph** (125 MHz, $CDCl_3$)

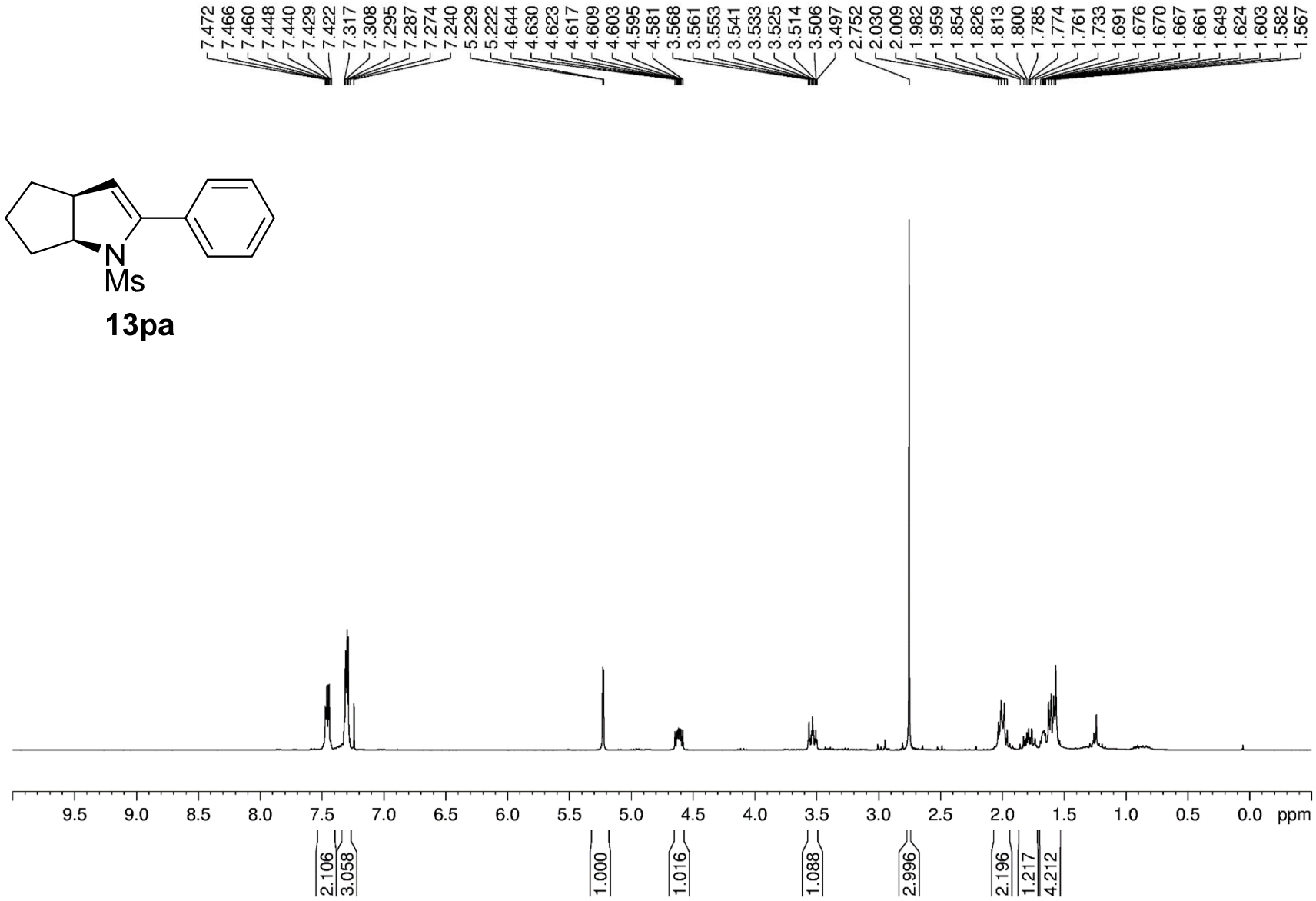


12pa

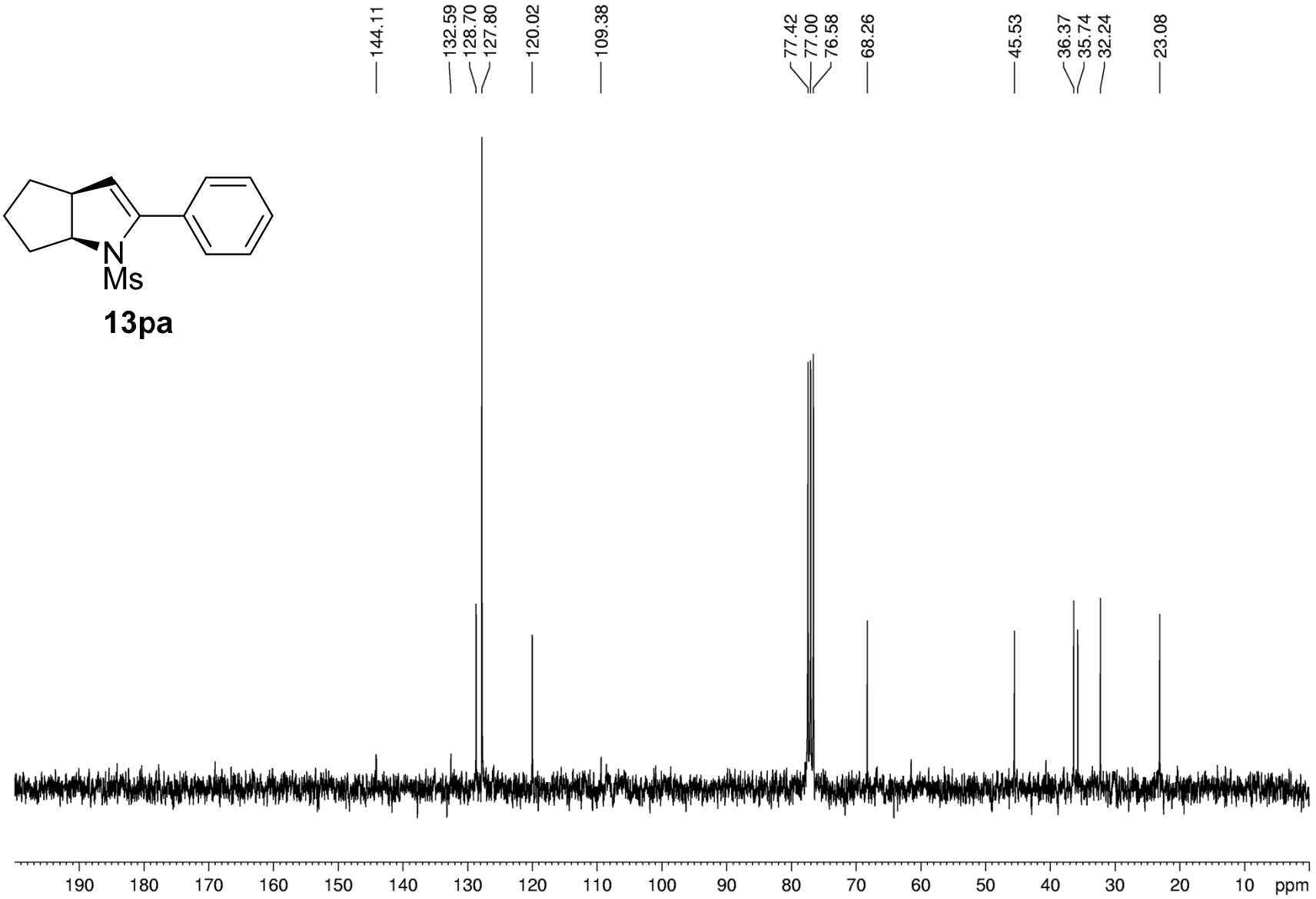
^{1}H NMR of compound **12pa** (500 MHz, $CDCl_3$)



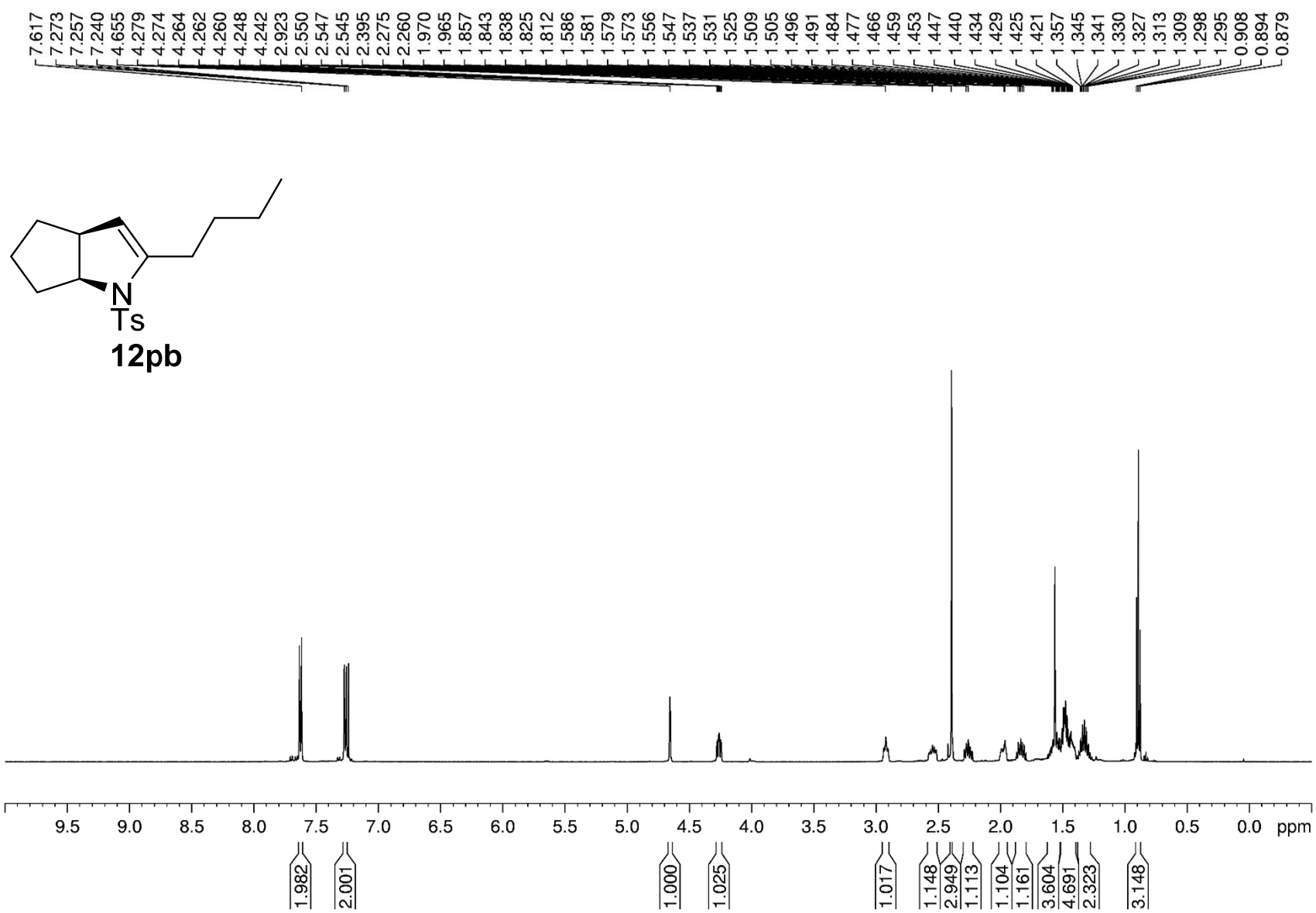
^{13}C NMR of compound **12pa** (125 MHz, $CDCl_3$)



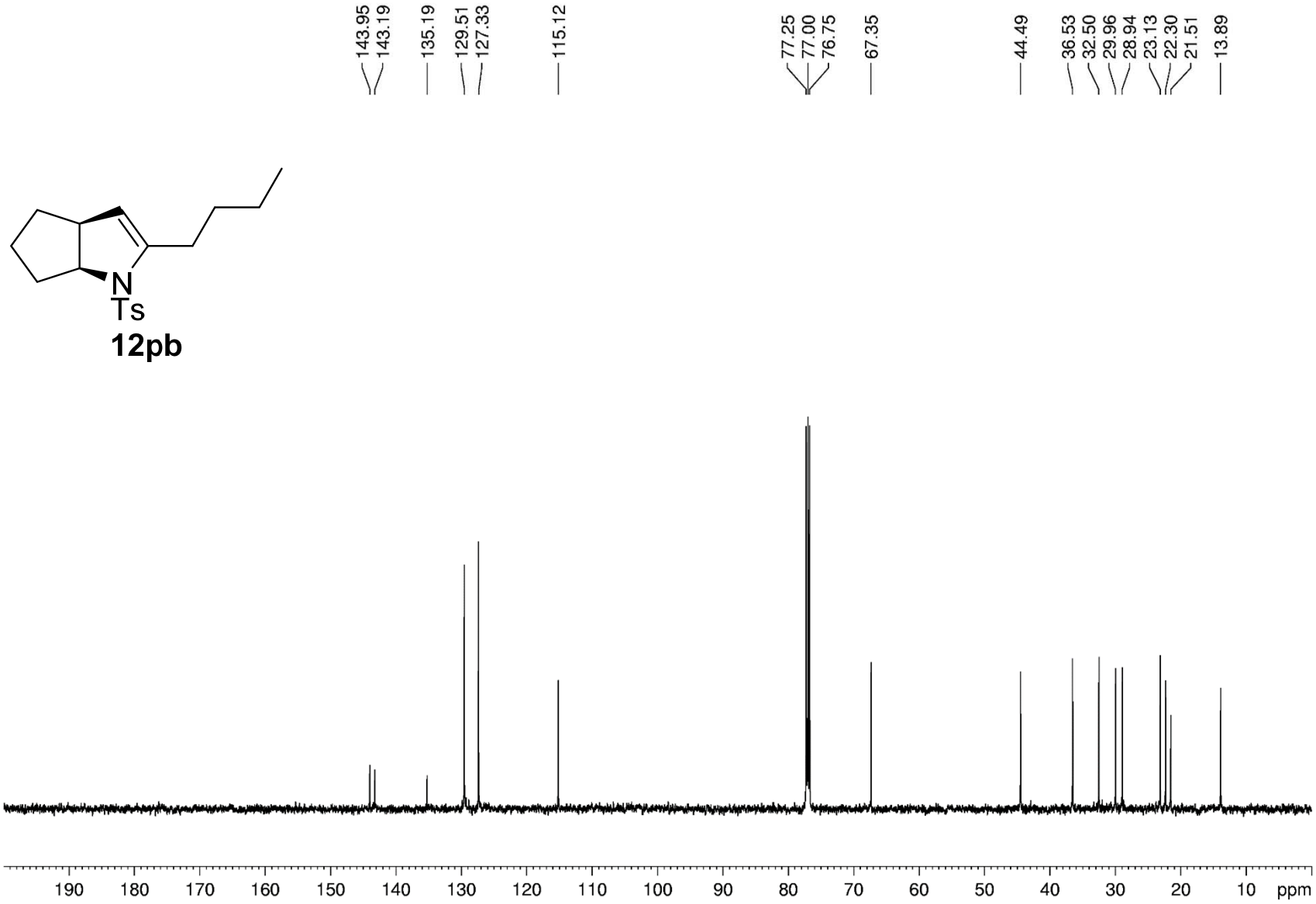
^{1}H NMR of compound **13pa** (300 MHz, $CDCl_3$)



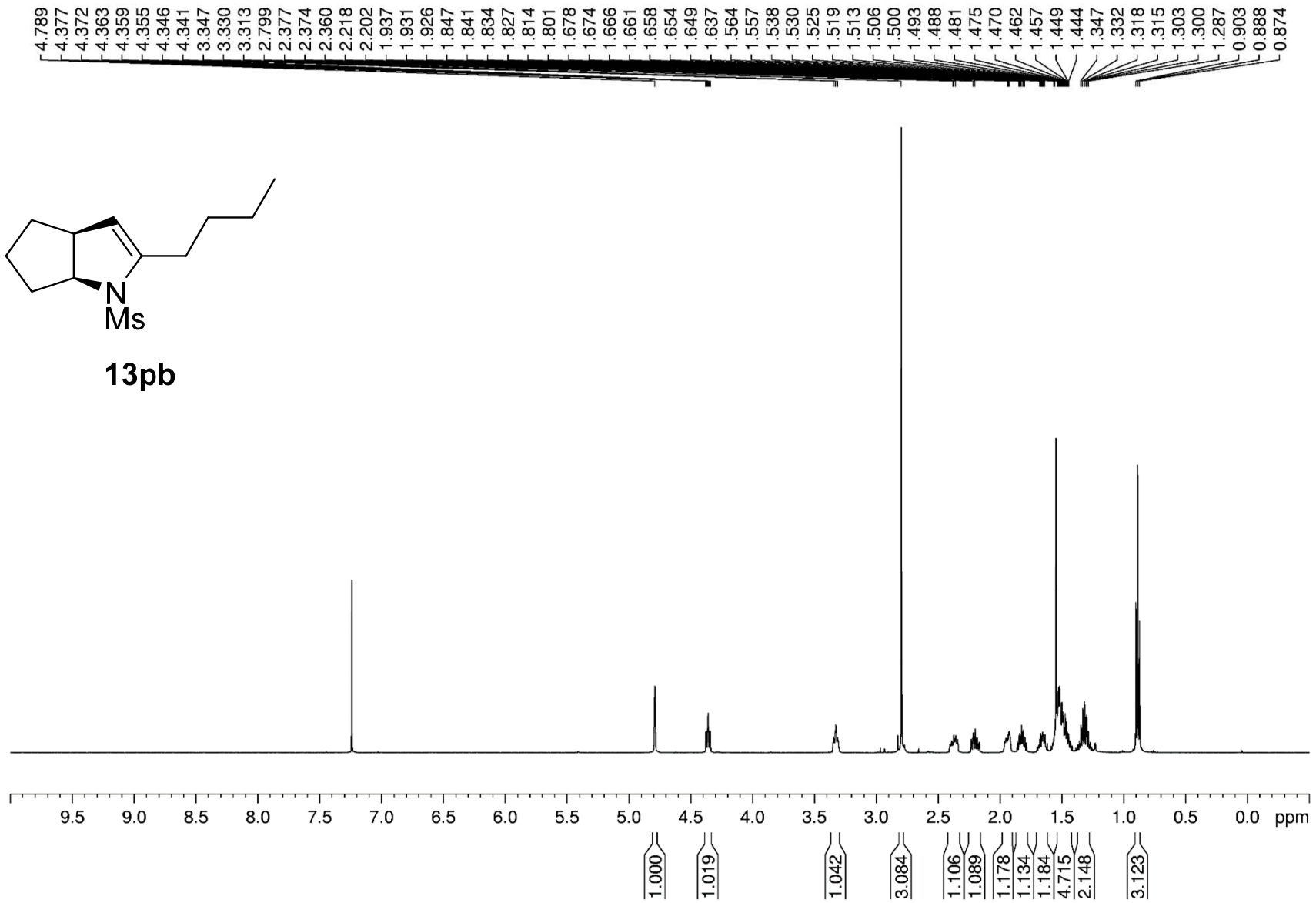
^{13}C NMR of compound **13pa** (75 MHz, $CDCl_3$)



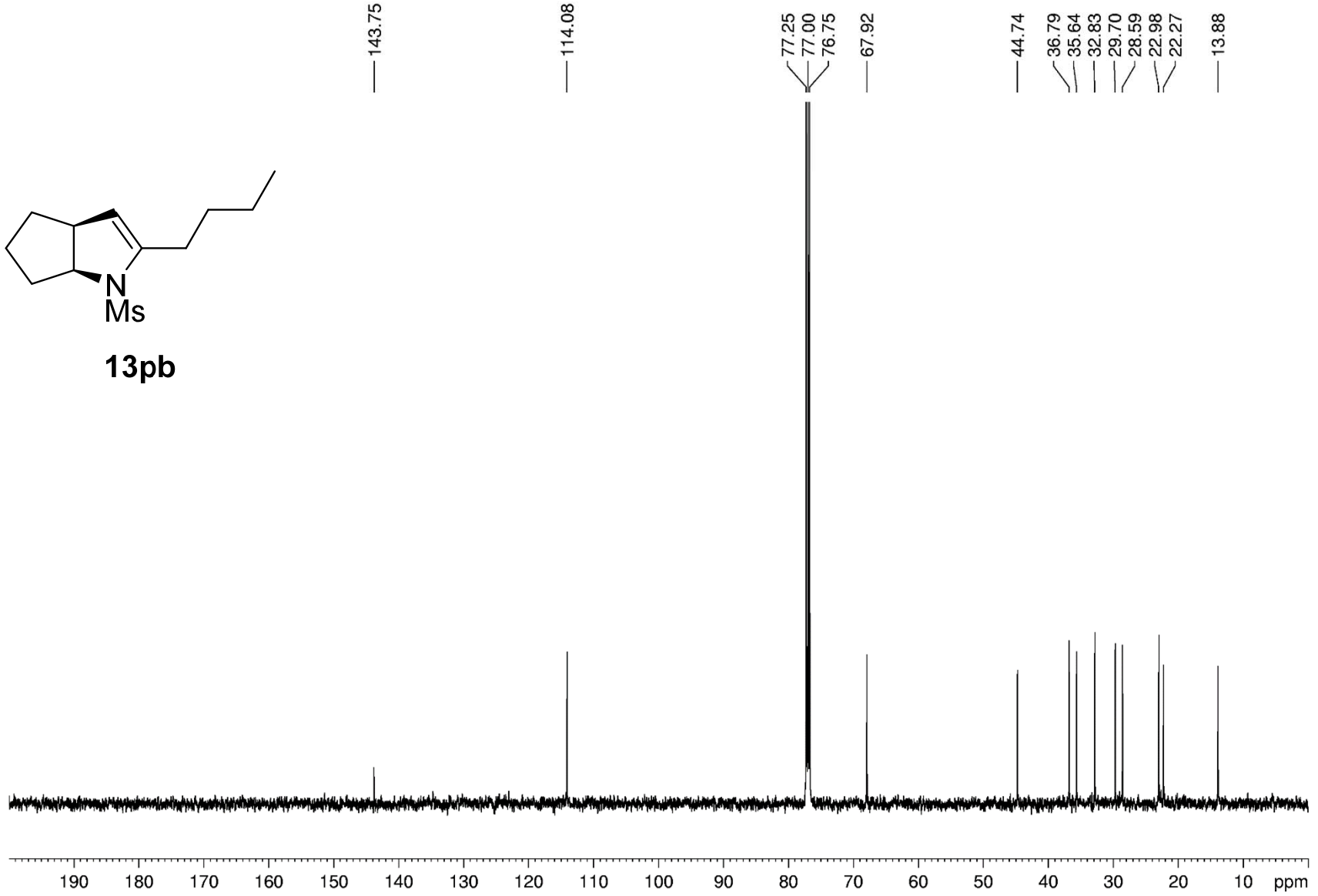
^{1}H NMR of compound **12pb** (500 MHz, $CDCl_3$)



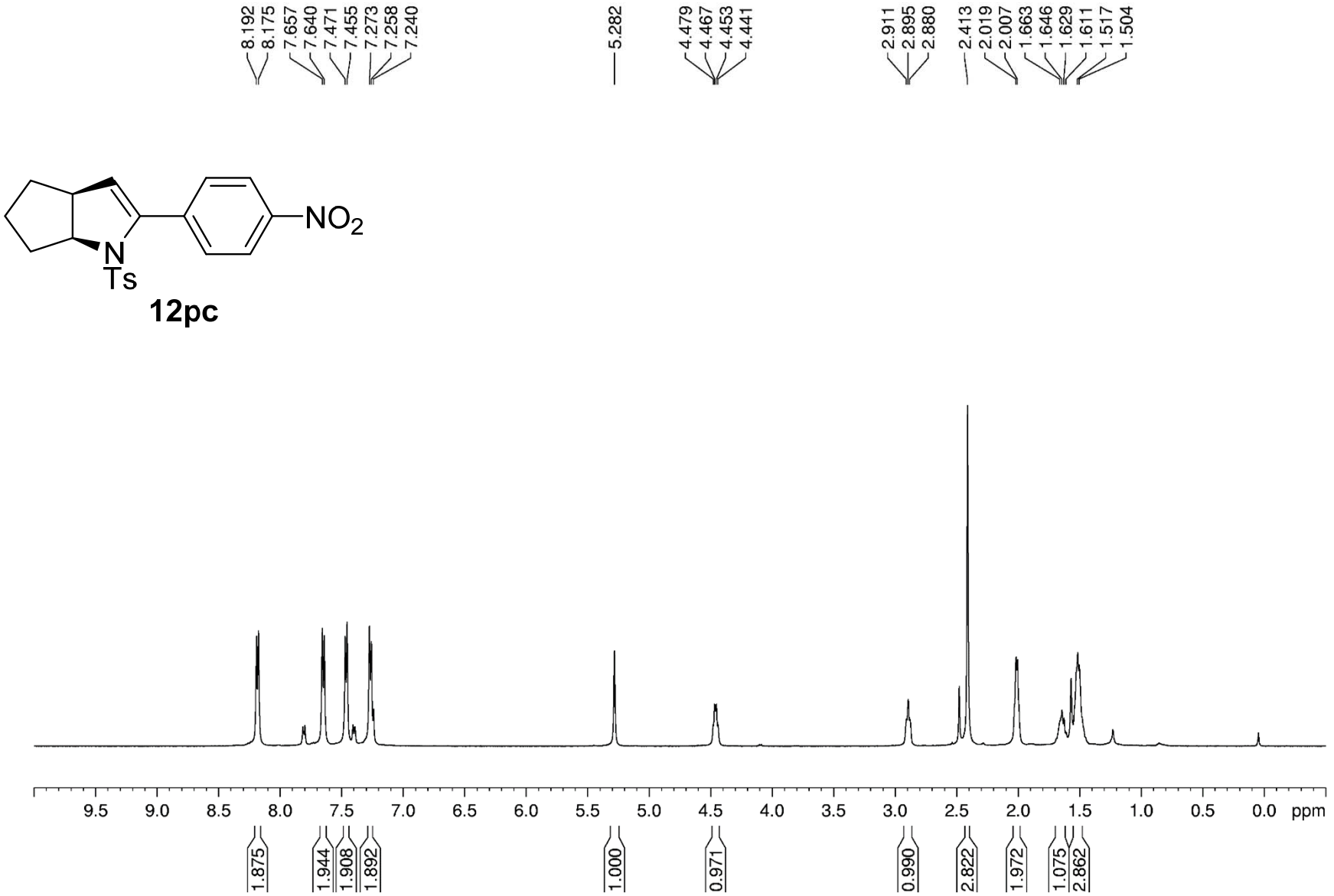
^{13}C NMR of compound **12pb** (125 MHz, $CDCl_3$)



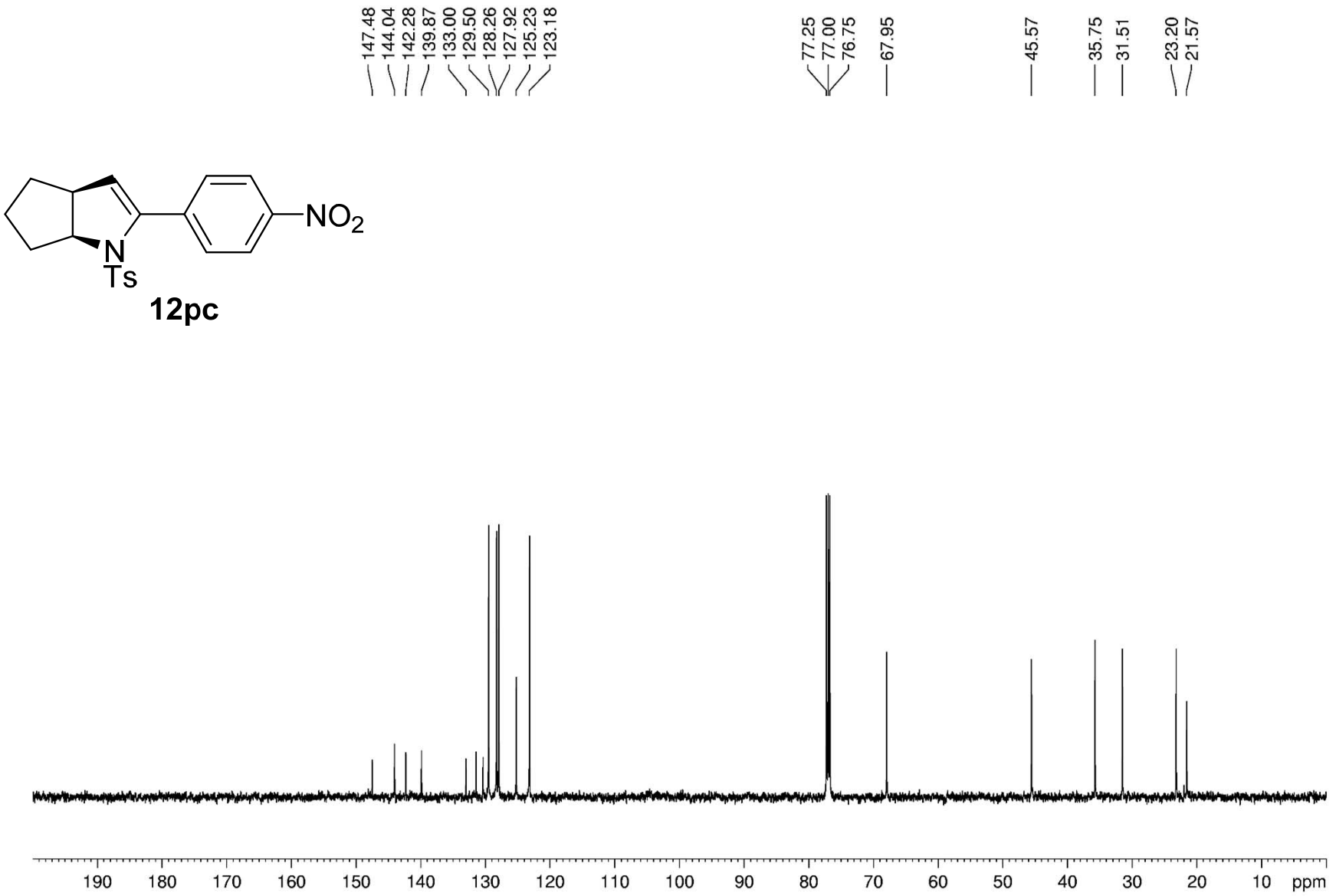
^{1}H NMR of compound **13pb** (500 MHz, $CDCl_3$)



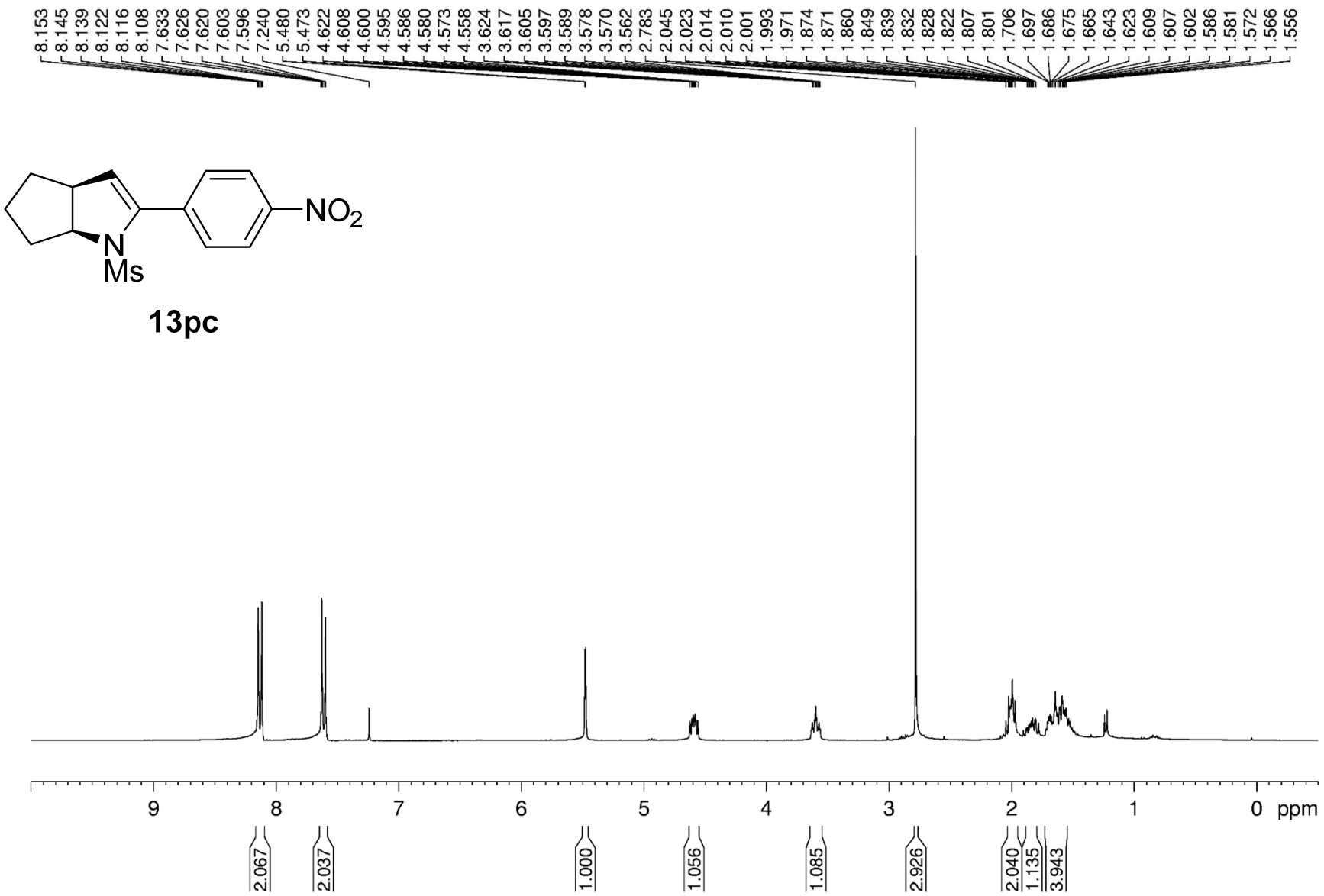
^{13}C NMR of compound **13pb** (125 MHz, $CDCl_3$)



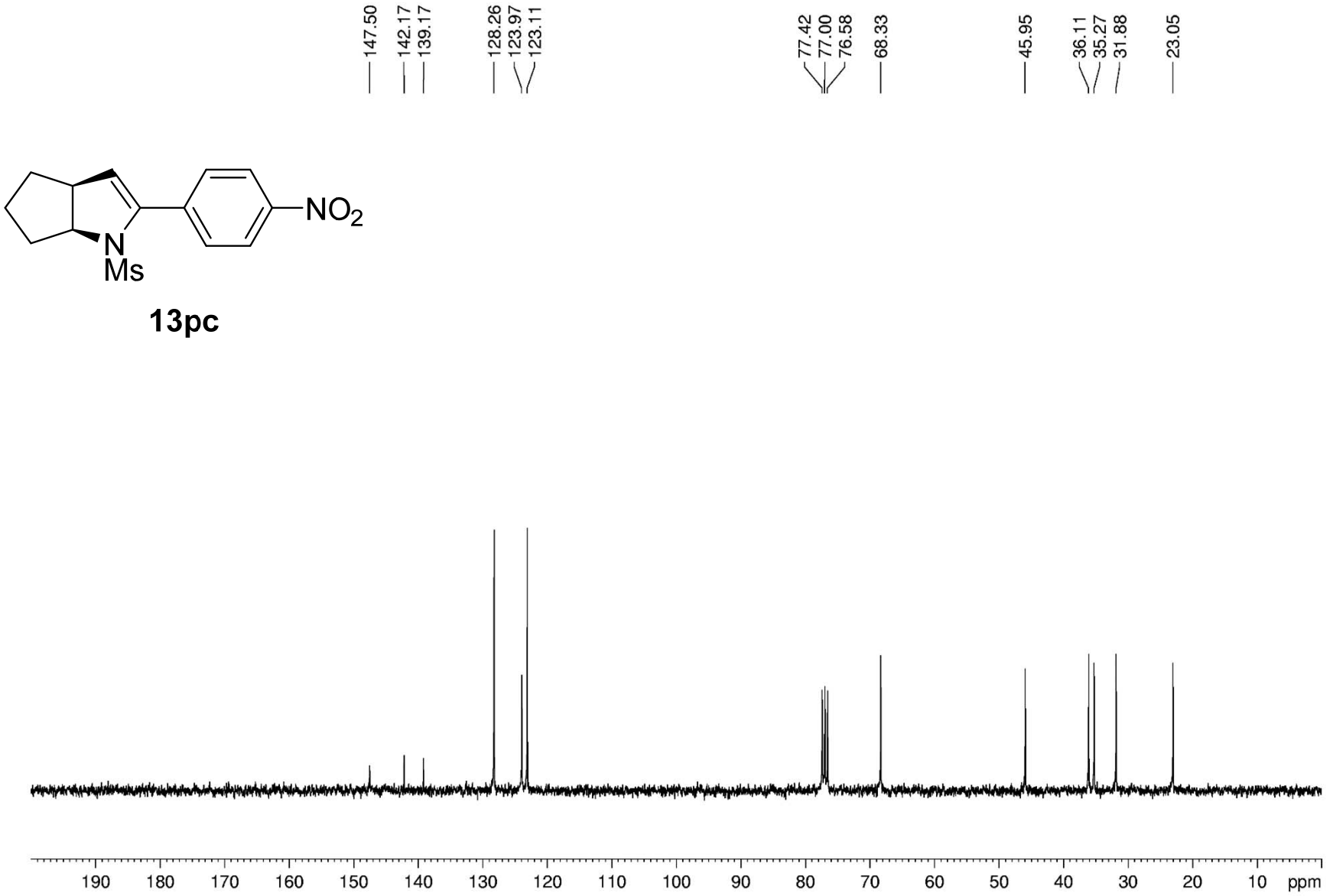
^{1}H NMR of compound **12pc** (500 MHz, $CDCl_3$)



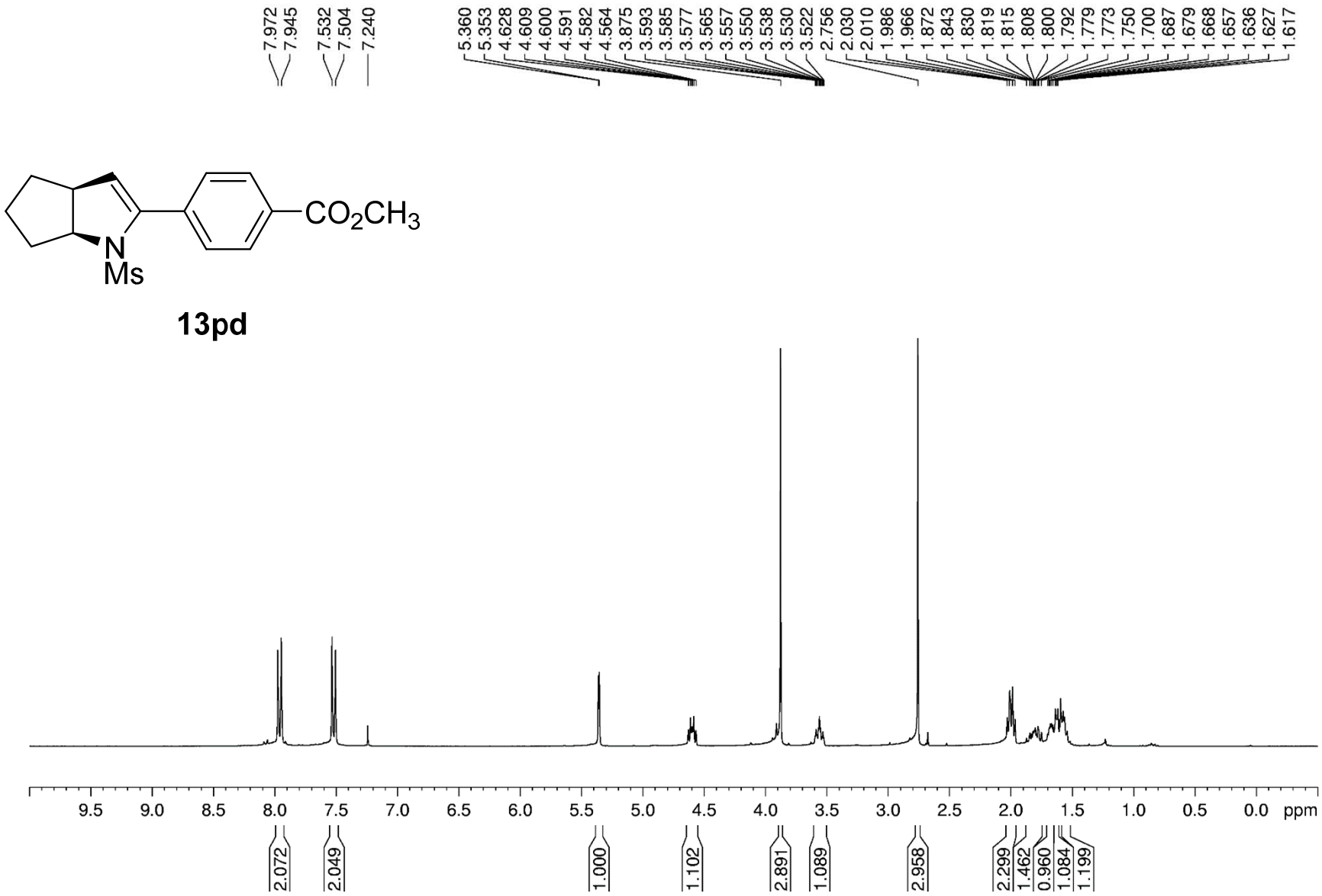
^{13}C NMR of compound **12pc** (125 MHz, $CDCl_3$)



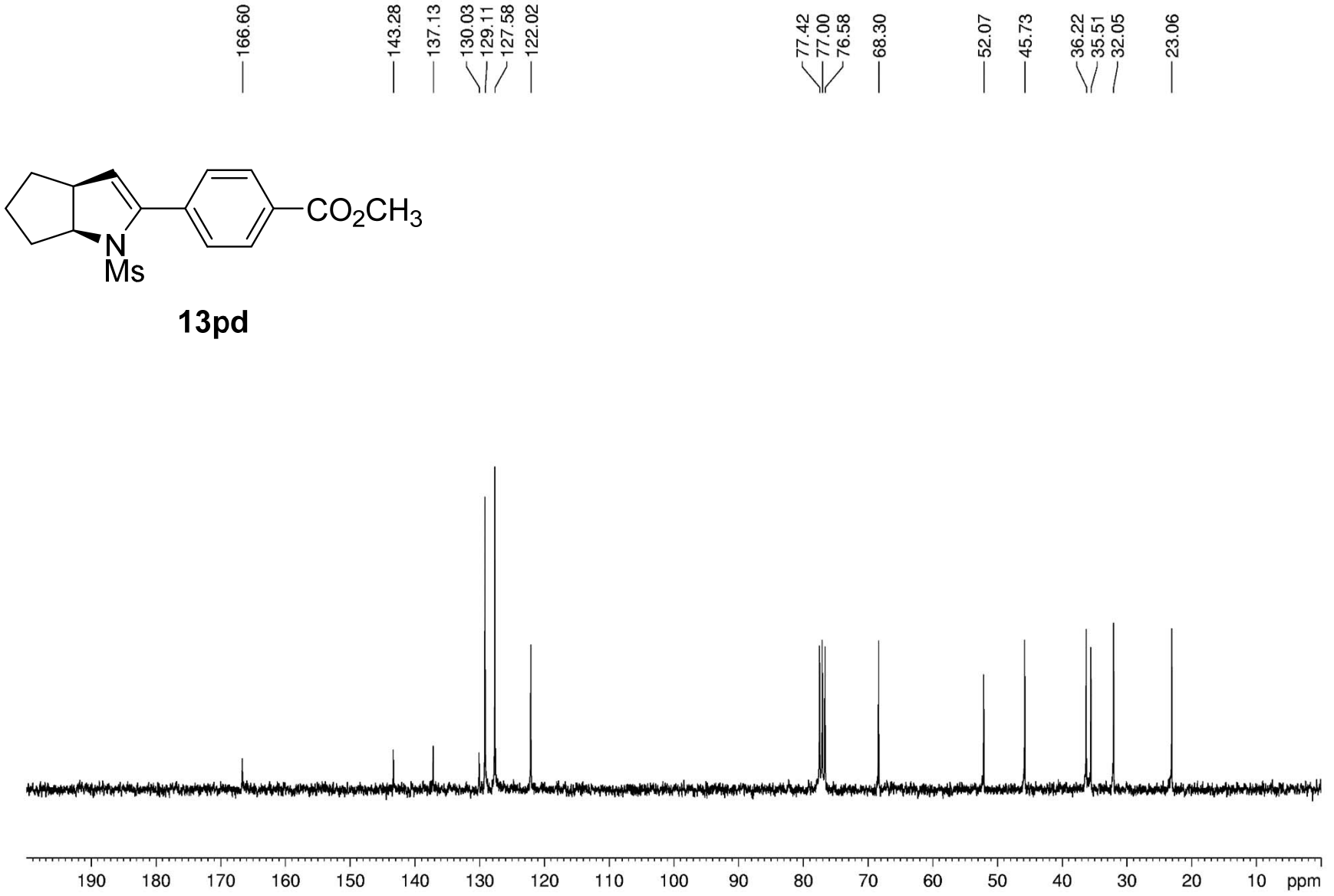
^{1}H NMR of compound **13pc** (500 MHz, $CDCl_3$)



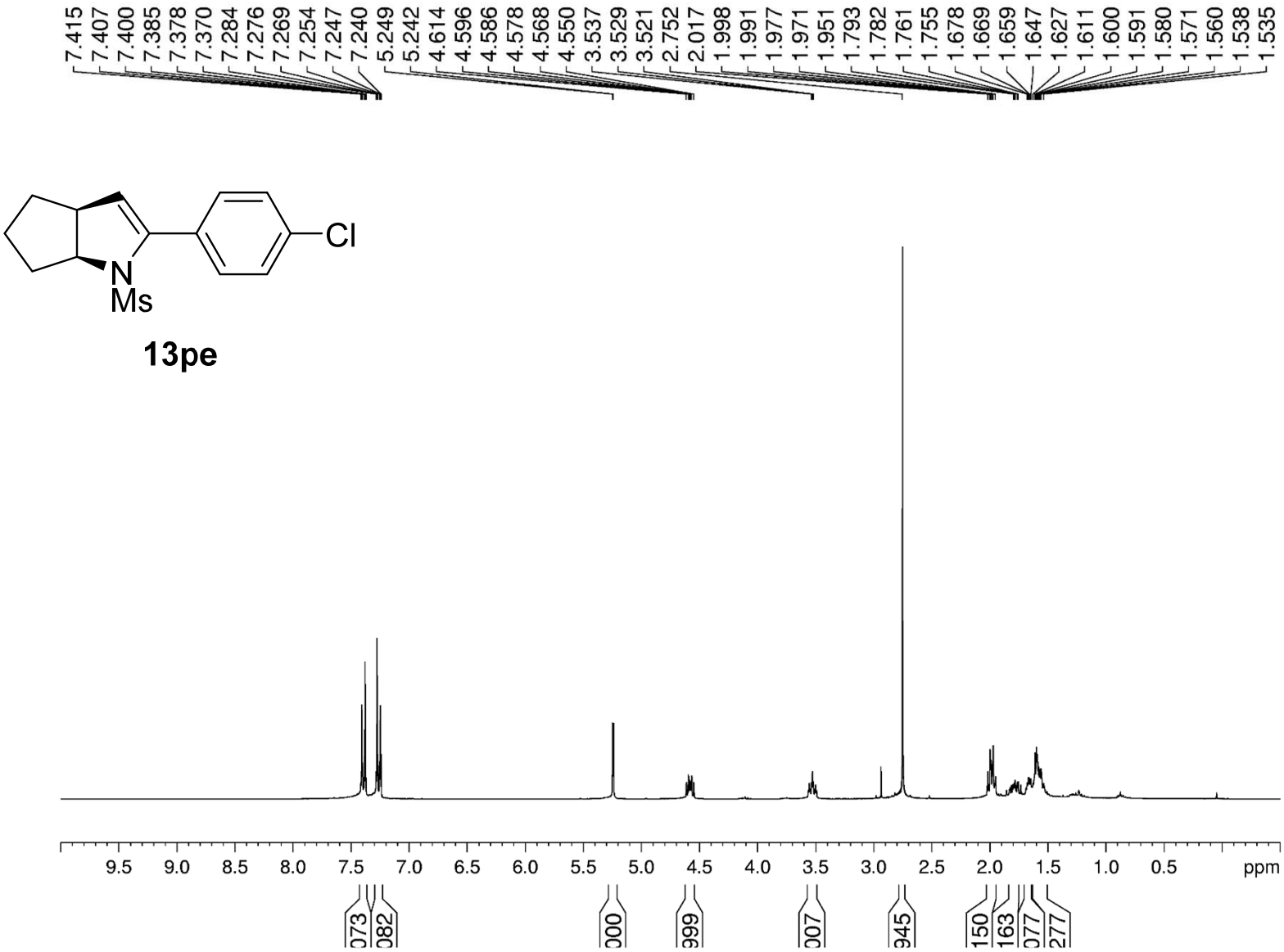
^{13}C NMR of compound **13pc** (75 MHz, $CDCl_3$)



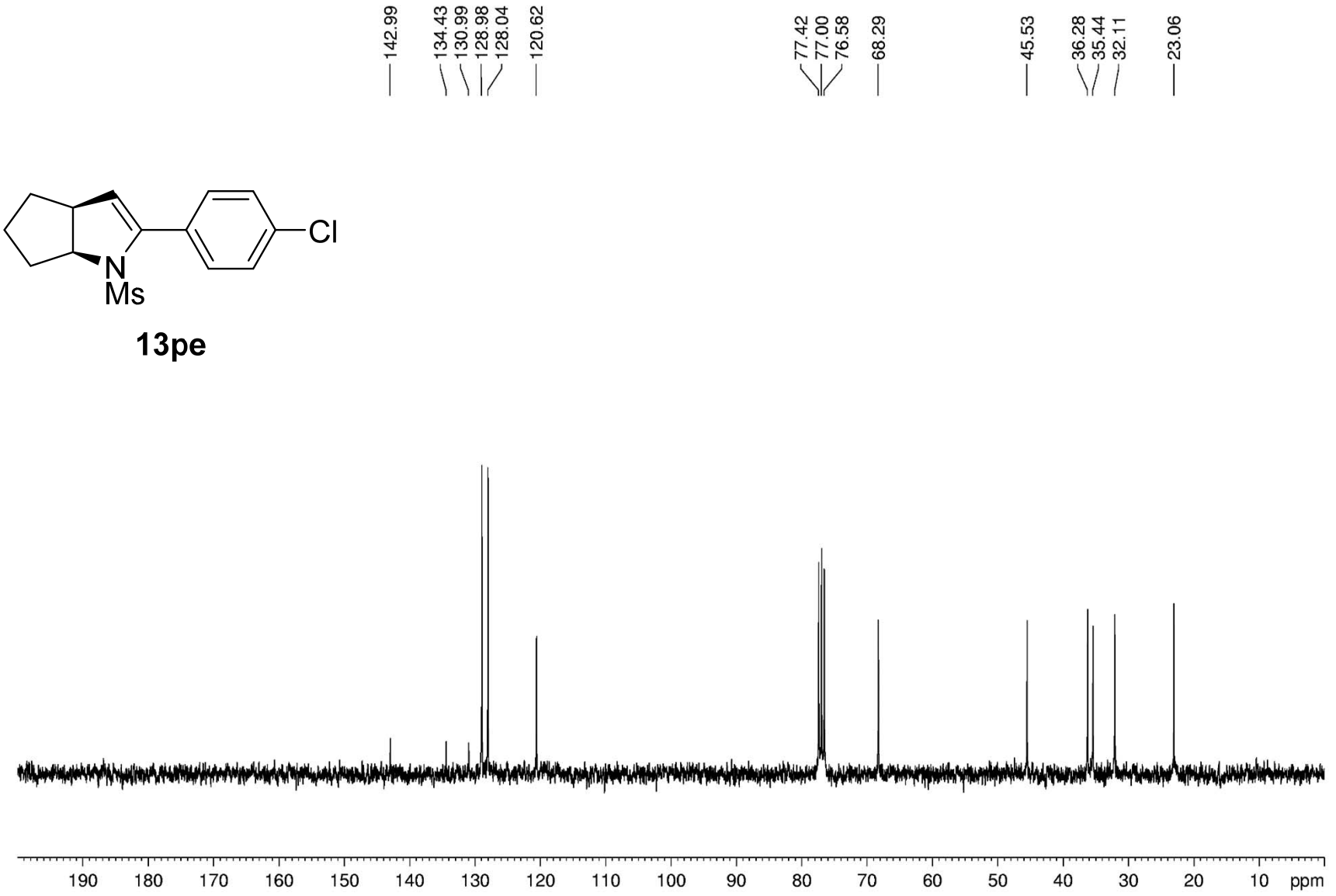
^{1}H NMR of compound **13pd** (300 MHz, $CDCl_3$)



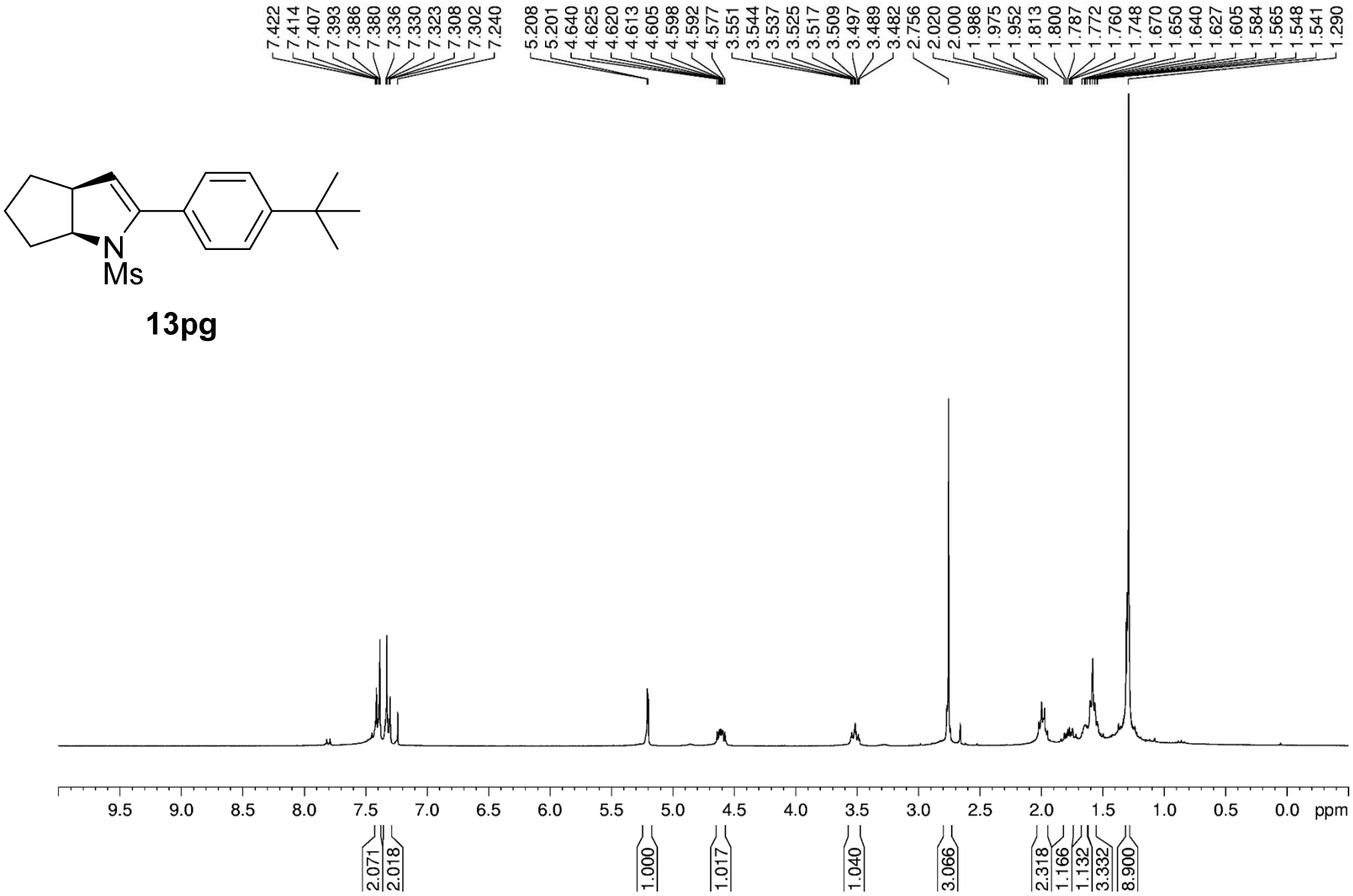
^{13}C NMR of compound **13pd** (75 MHz, $CDCl_3$)



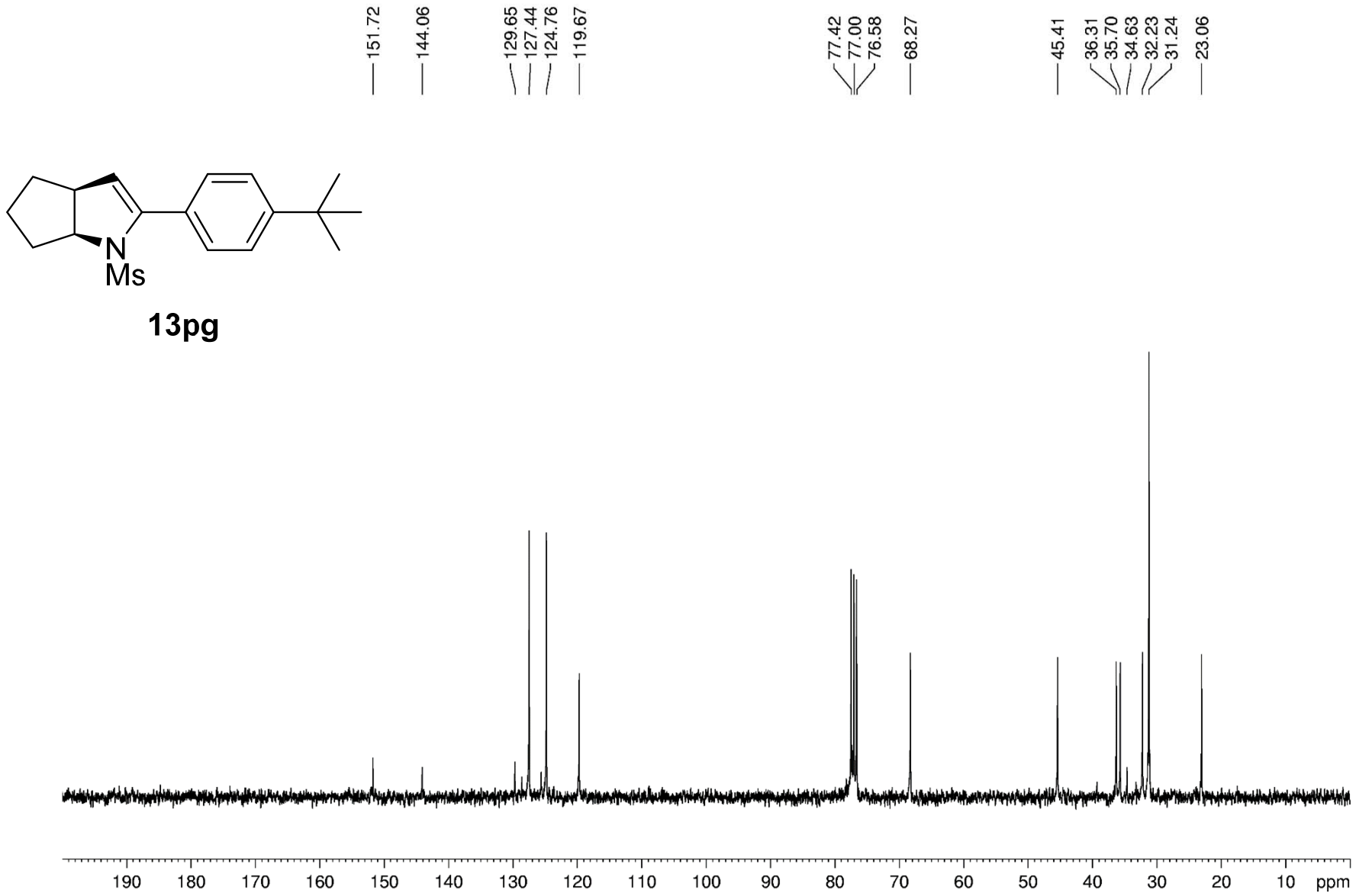
^{1}H NMR of compound **13pe** (300 MHz, $CDCl_3$)



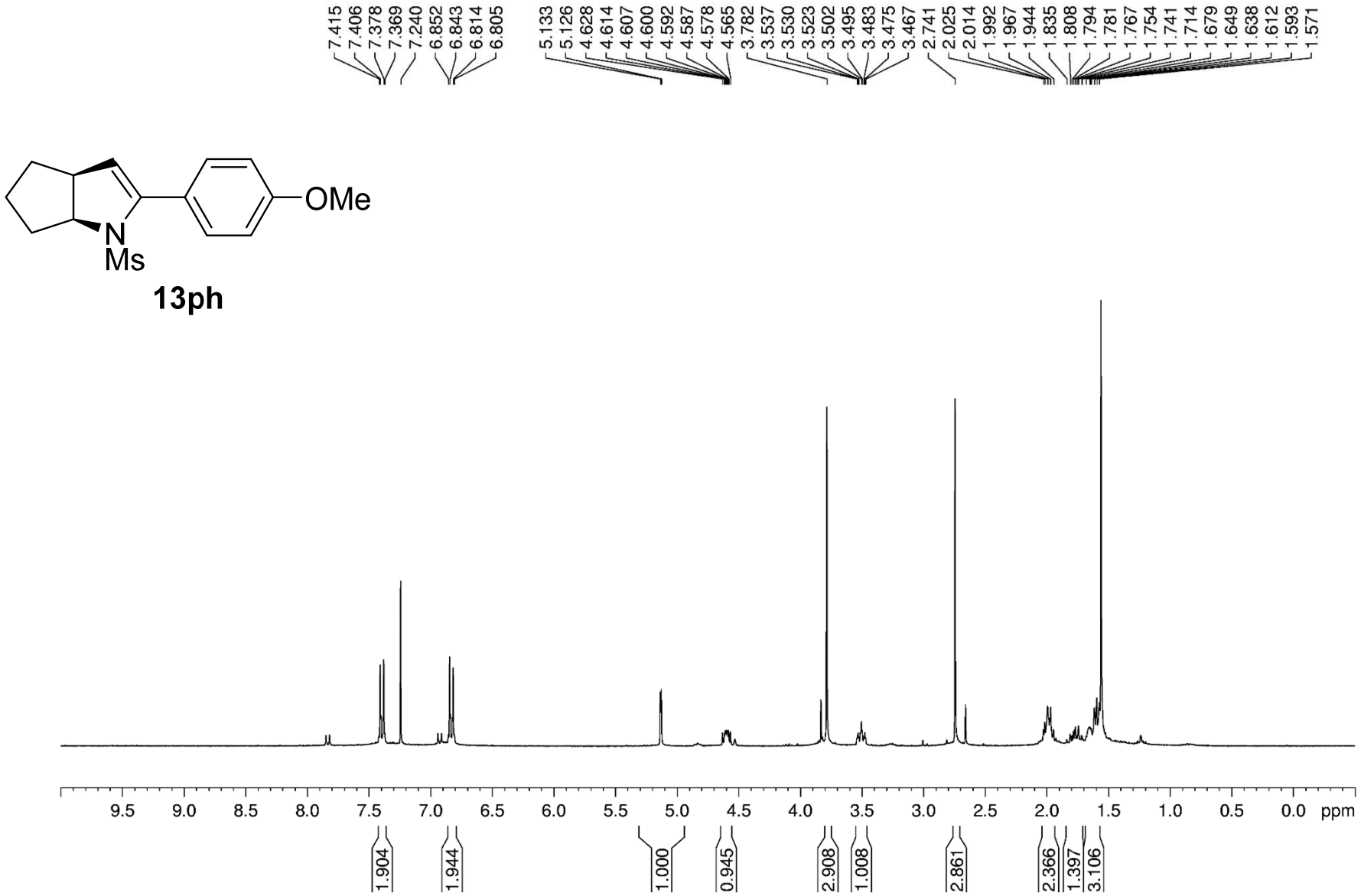
^{13}C NMR of compound **13pe** (75 MHz, $CDCl_3$)



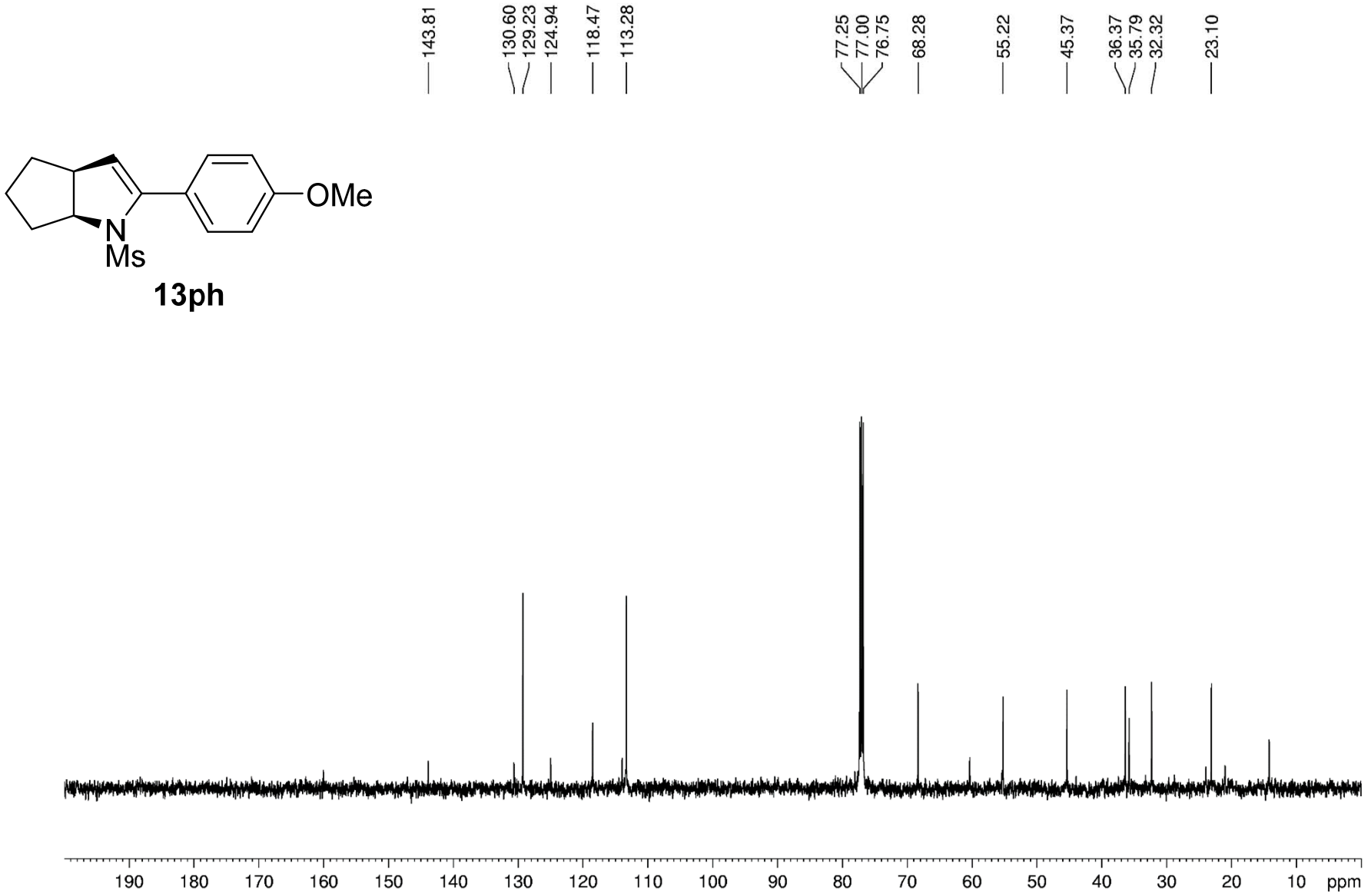
^{1}H NMR of compound **13pg** (300 MHz, $CDCl_3$)



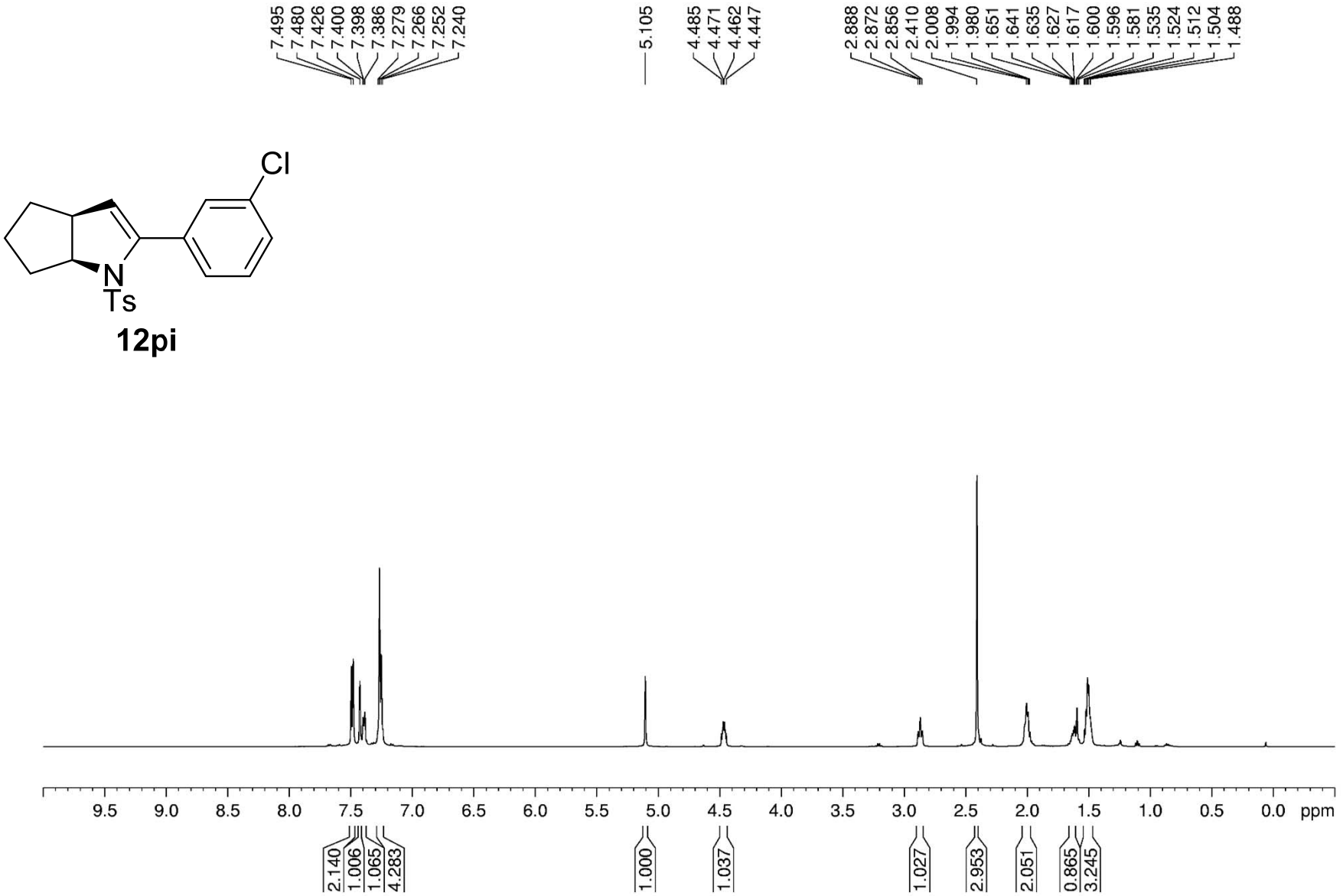
^{13}C NMR of compound **13pg** (75 MHz, $CDCl_3$)



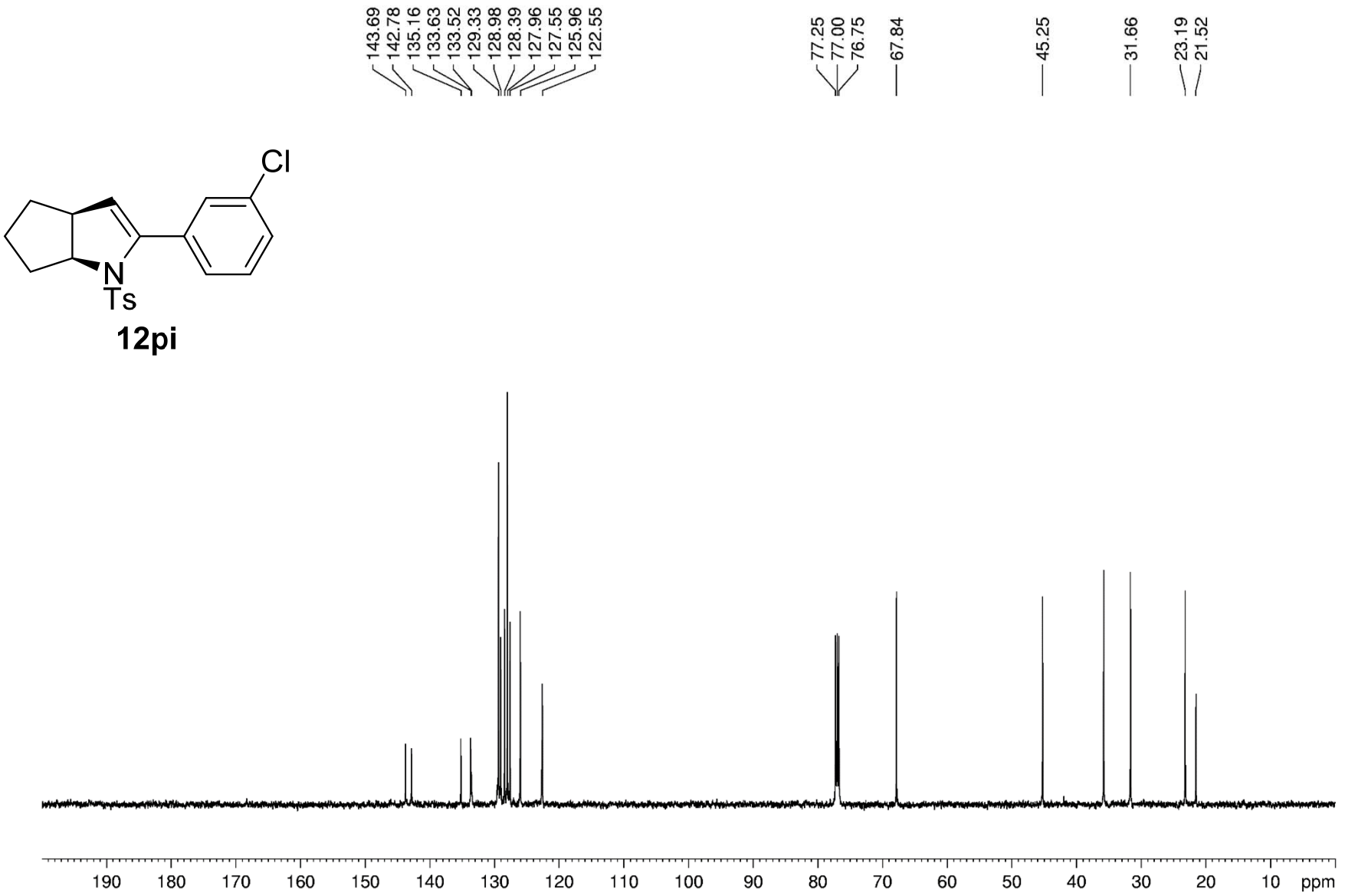
^{1}H NMR of compound **13ph** (300 MHz, $CDCl_3$)



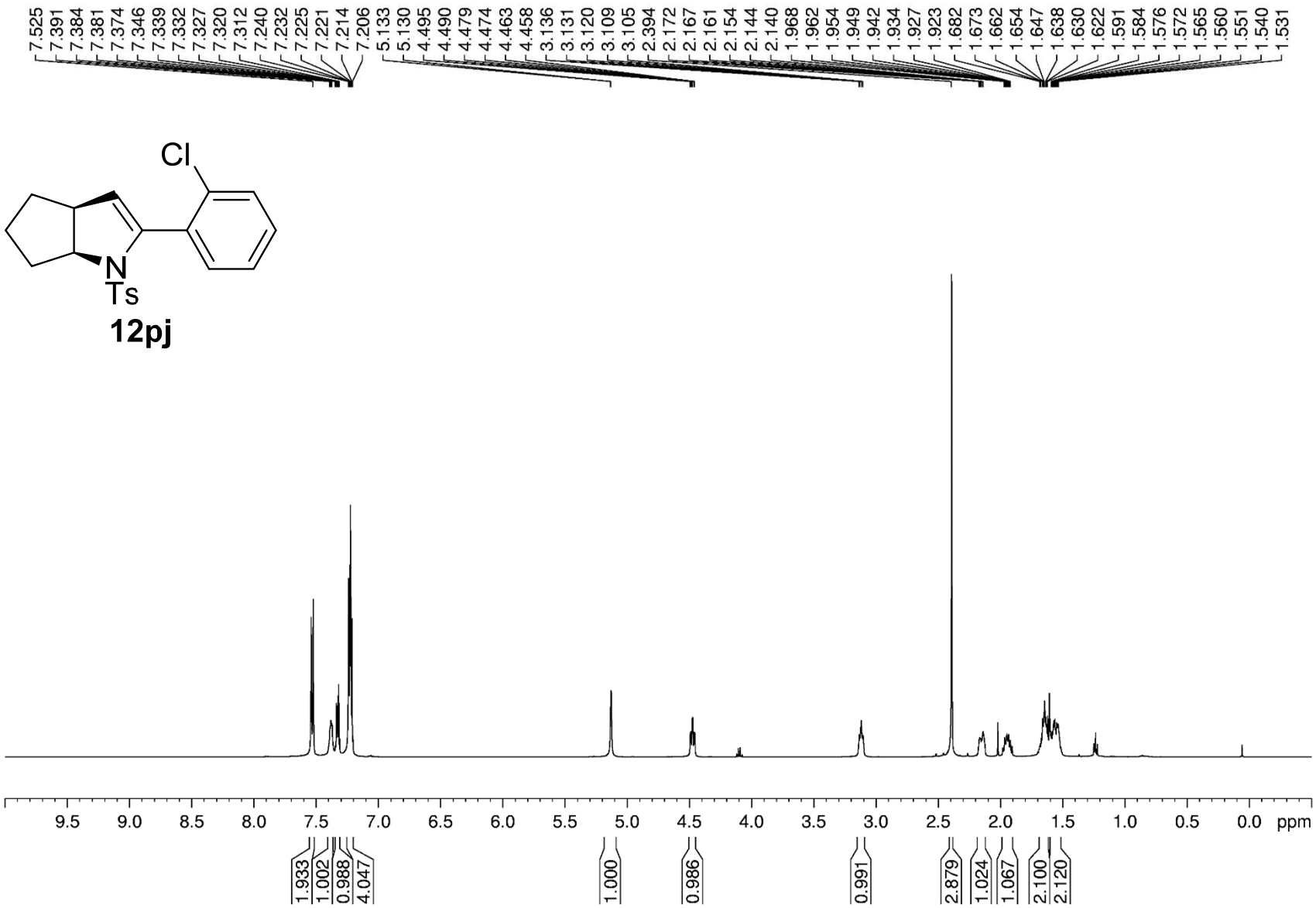
^{13}C NMR of compound **13ph** (125 MHz, $CDCl_3$)



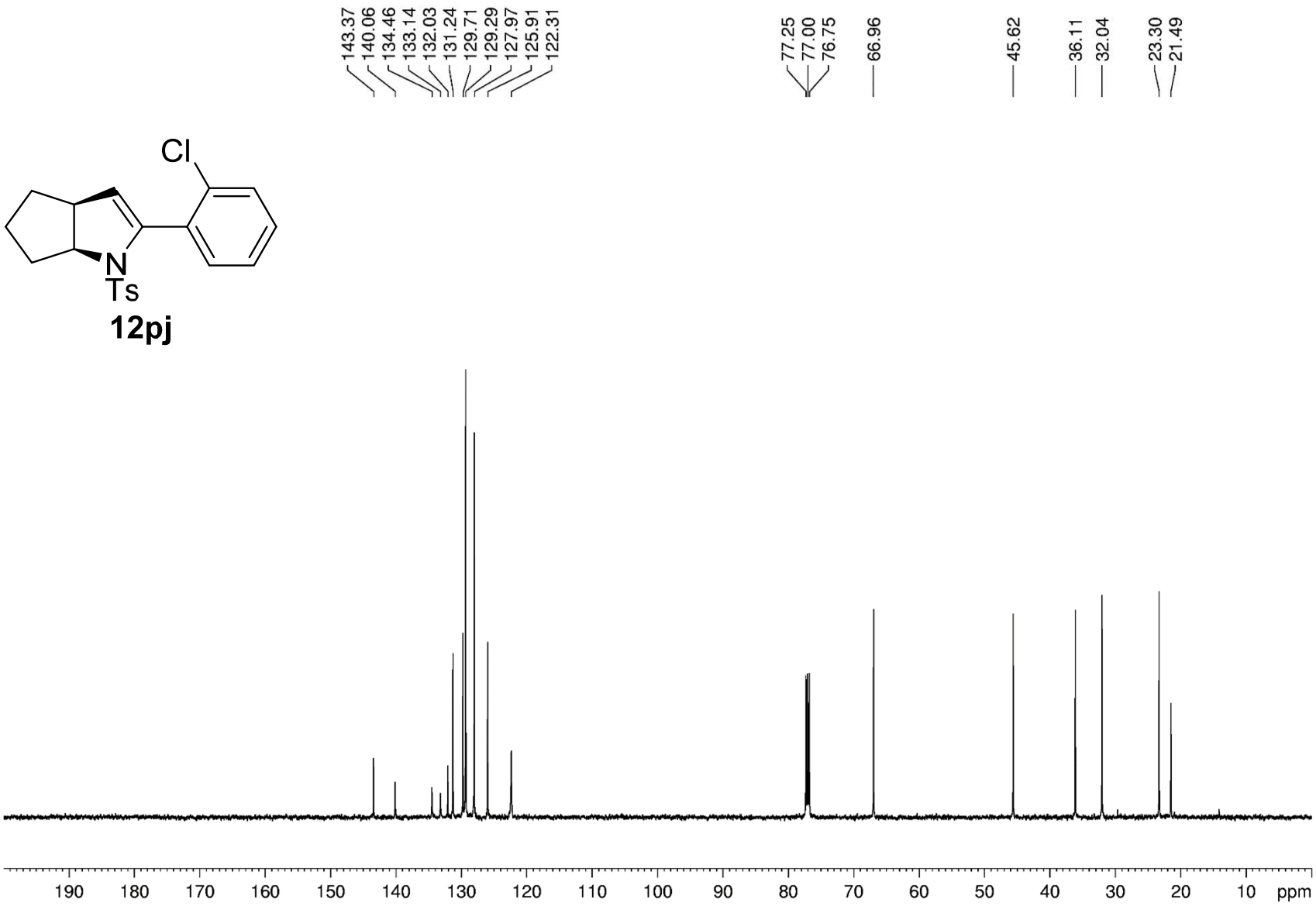
^{1}H NMR of compound **12pi** (500 MHz, $CDCl_3$)



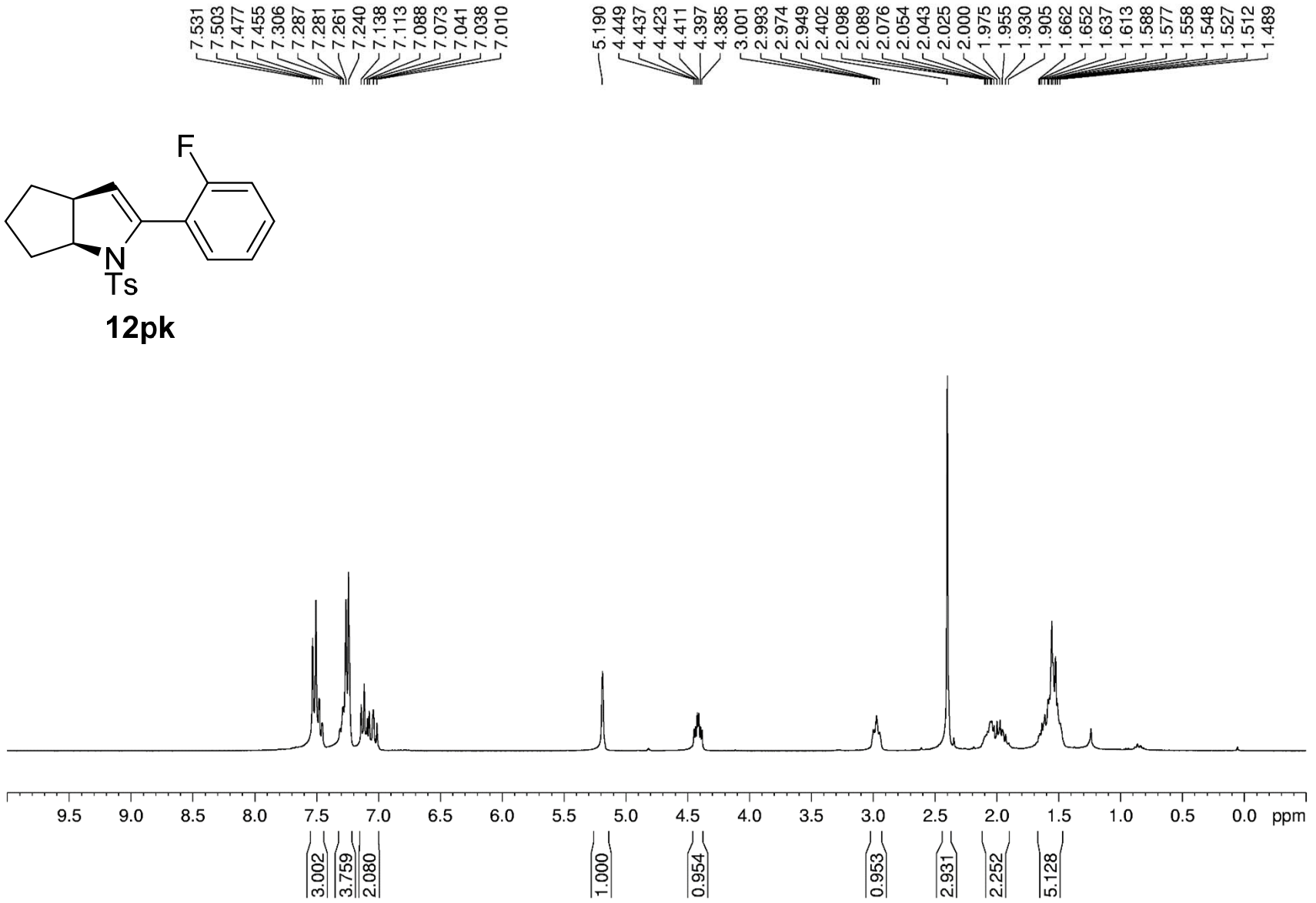
^{13}C NMR of compound **12pi** (125 MHz, $CDCl_3$)



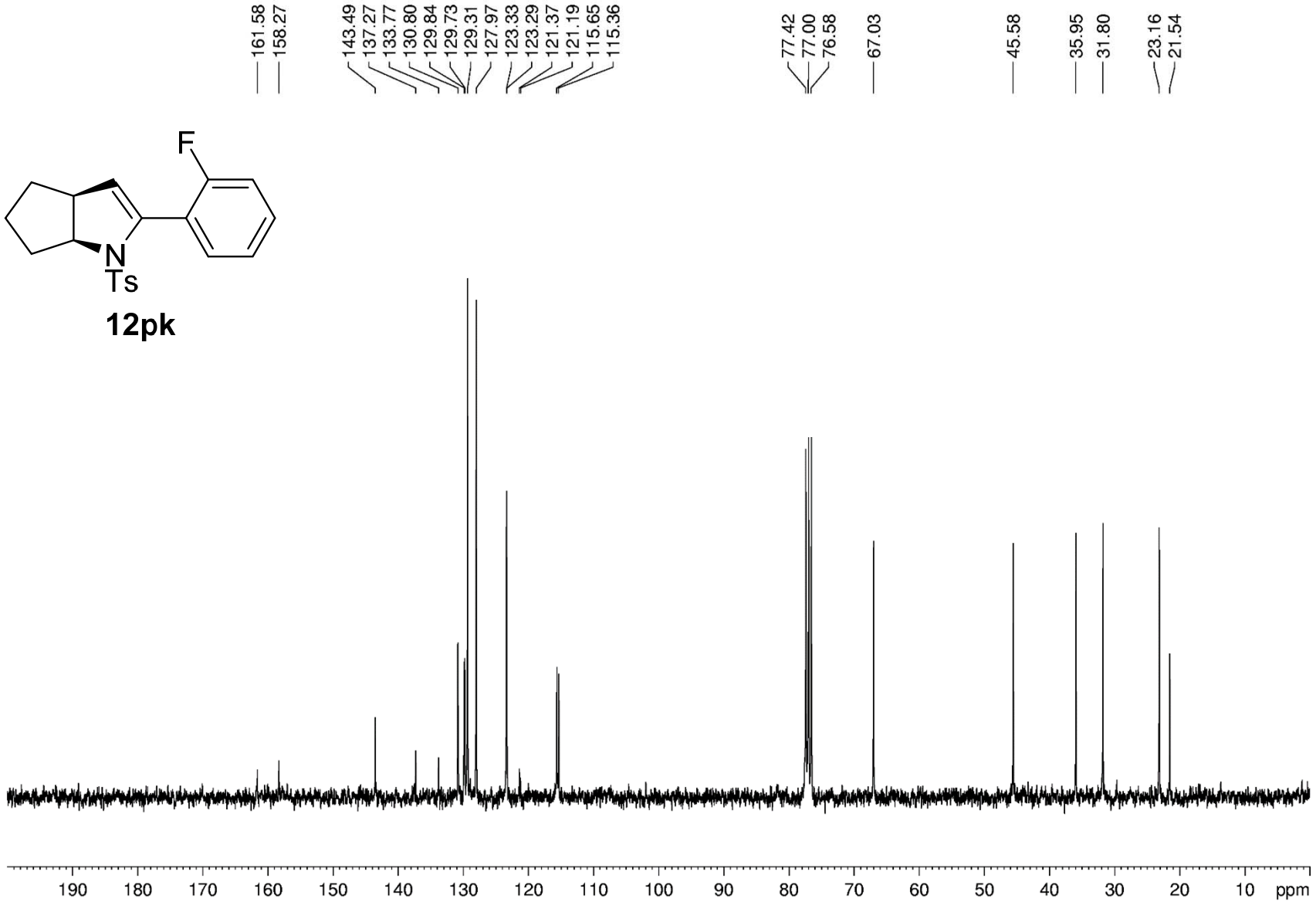
^{1}H NMR of compound **12pj** (500 MHz, $CDCl_3$)



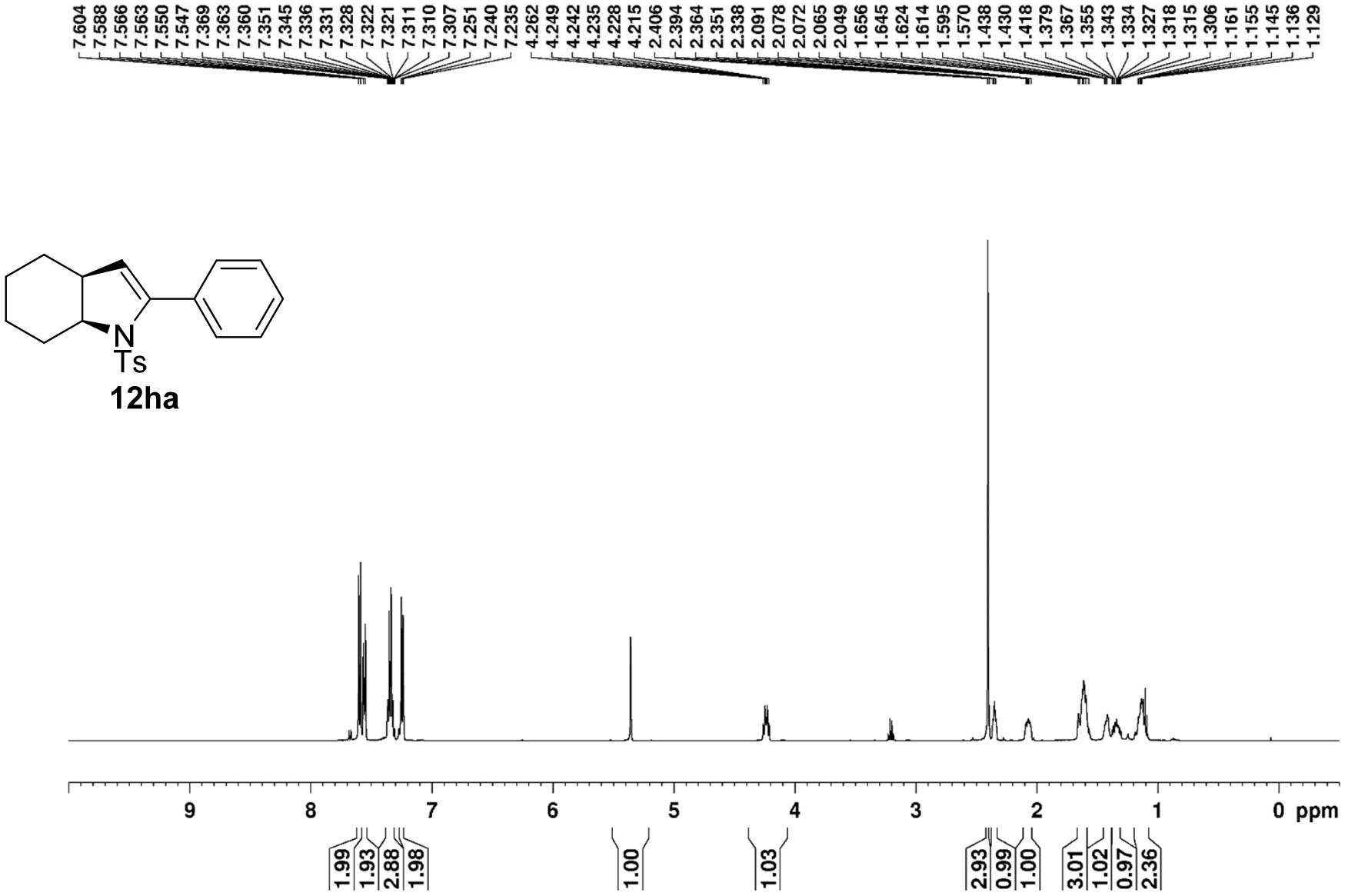
^{13}C NMR of compound **12pj** (125 MHz, $CDCl_3$)



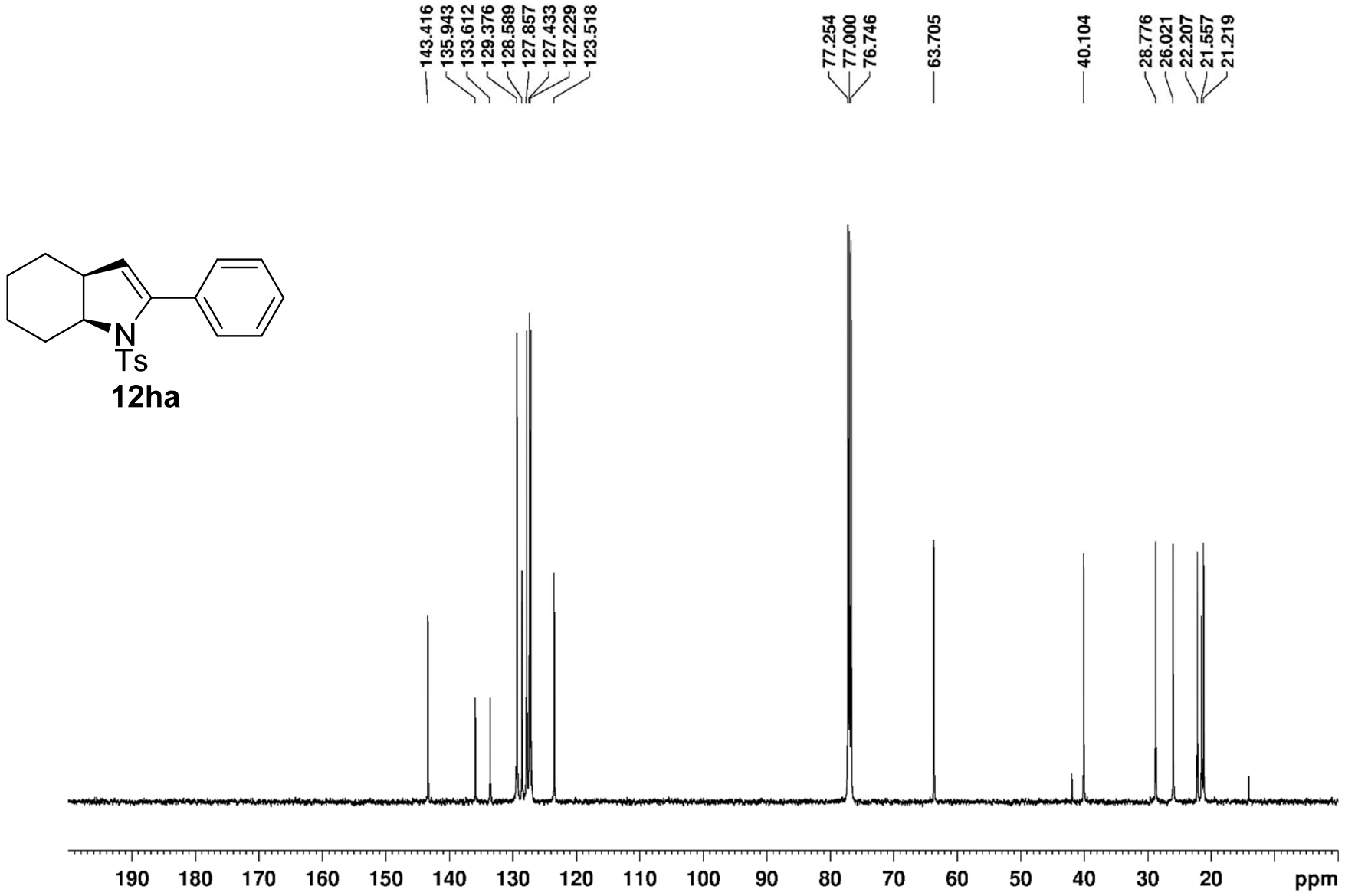
^{1}H NMR of compound **12pk** (300 MHz, $CDCl_3$)



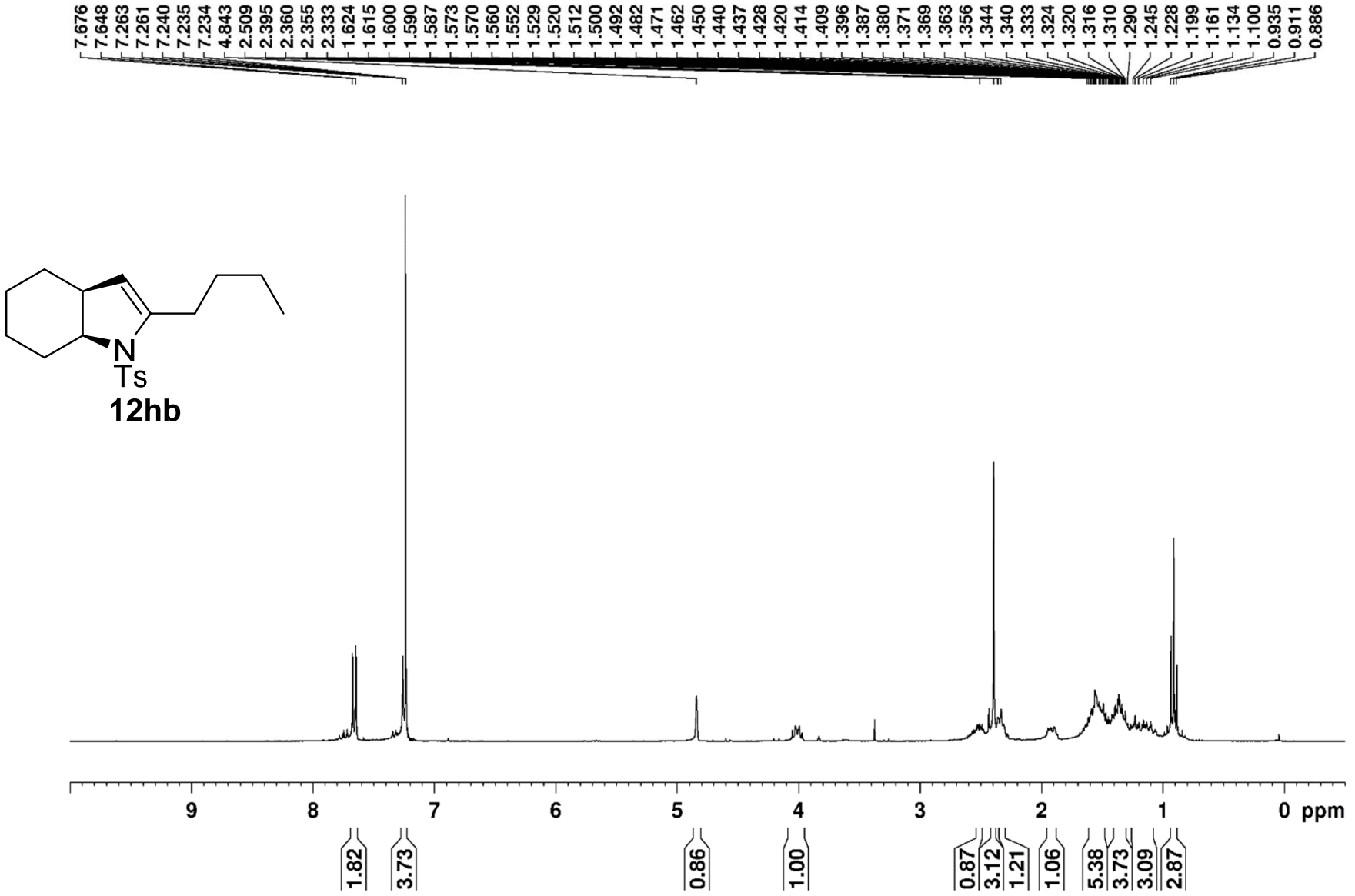
^{13}C NMR of compound **12pk** (75 MHz, $CDCl_3$)



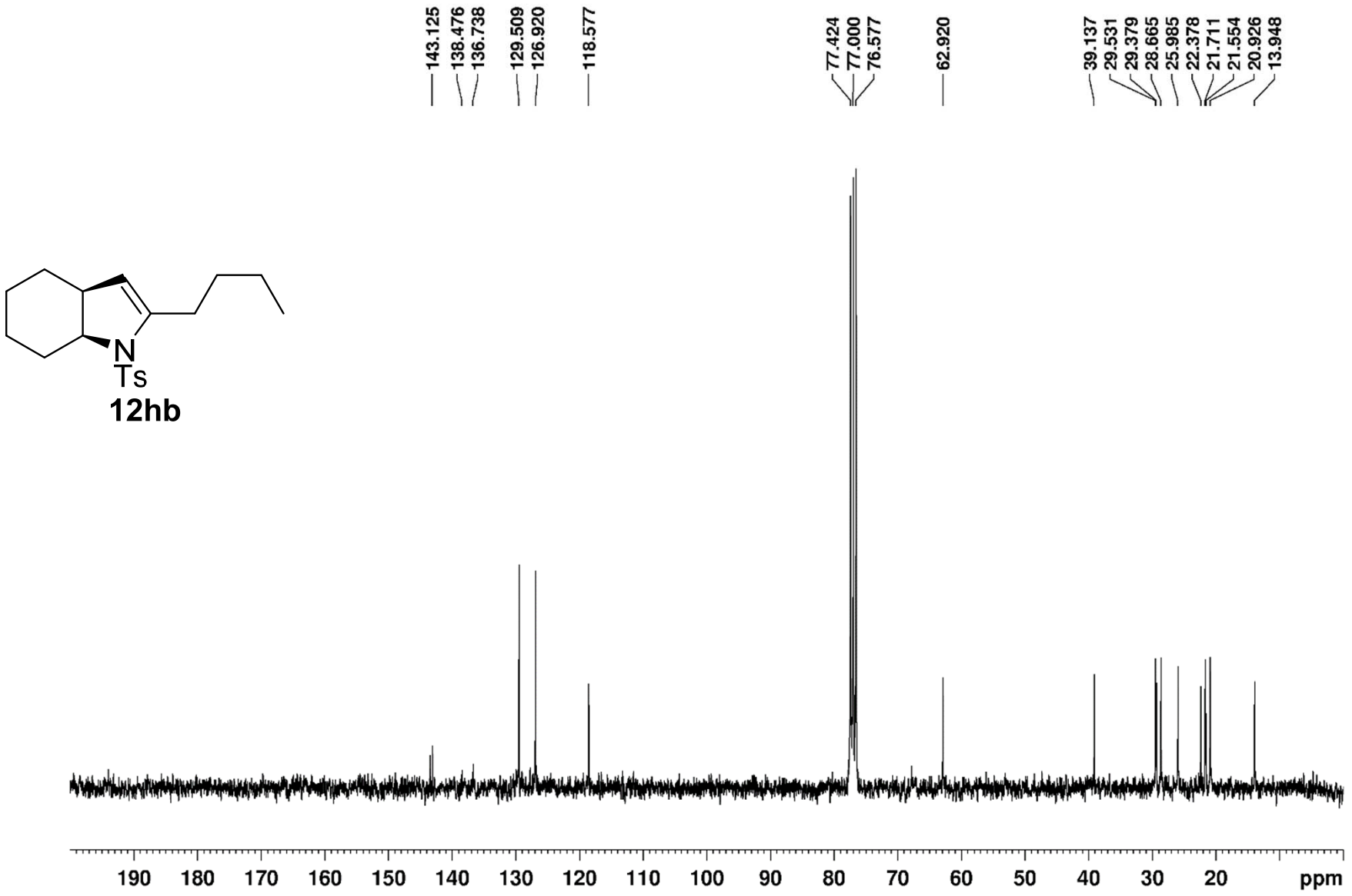
^{1}H NMR of compound **12ha** (500 MHz, $CDCl_3$)



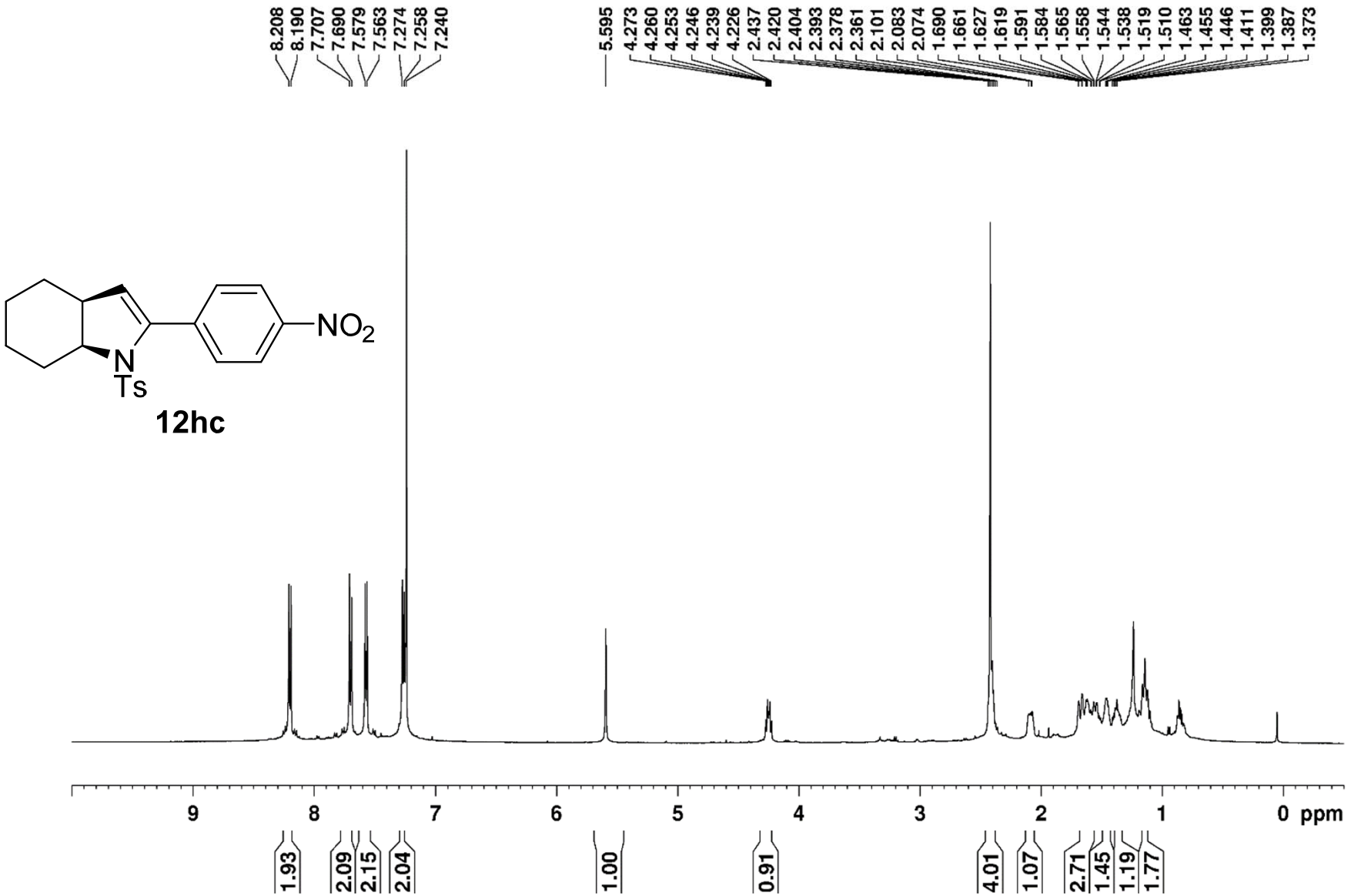
^{13}C NMR of compound **12ha** (125 MHz, $CDCl_3$)



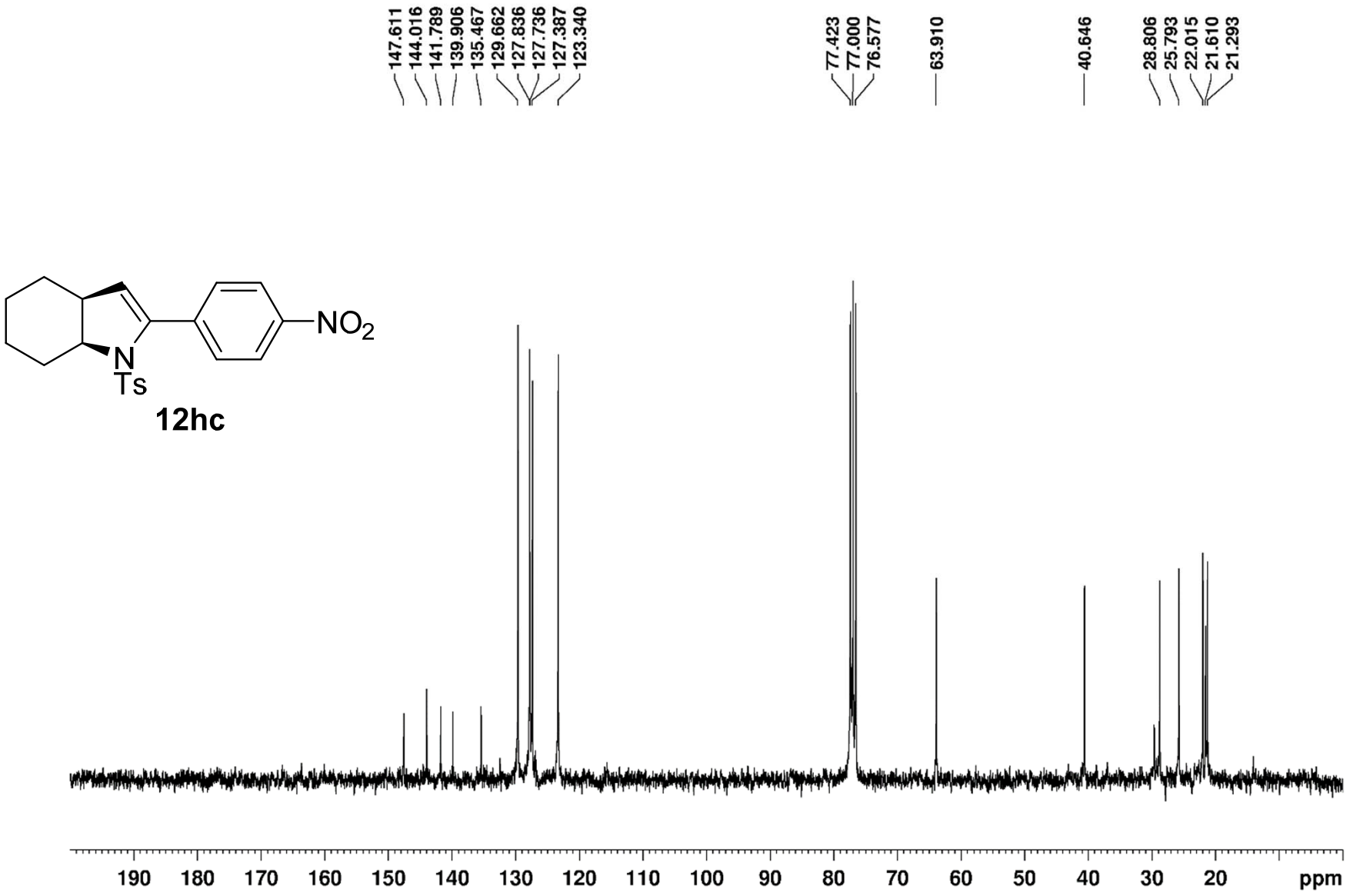
^{1}H NMR of compound **12hb** (300 MHz, $CDCl_3$)



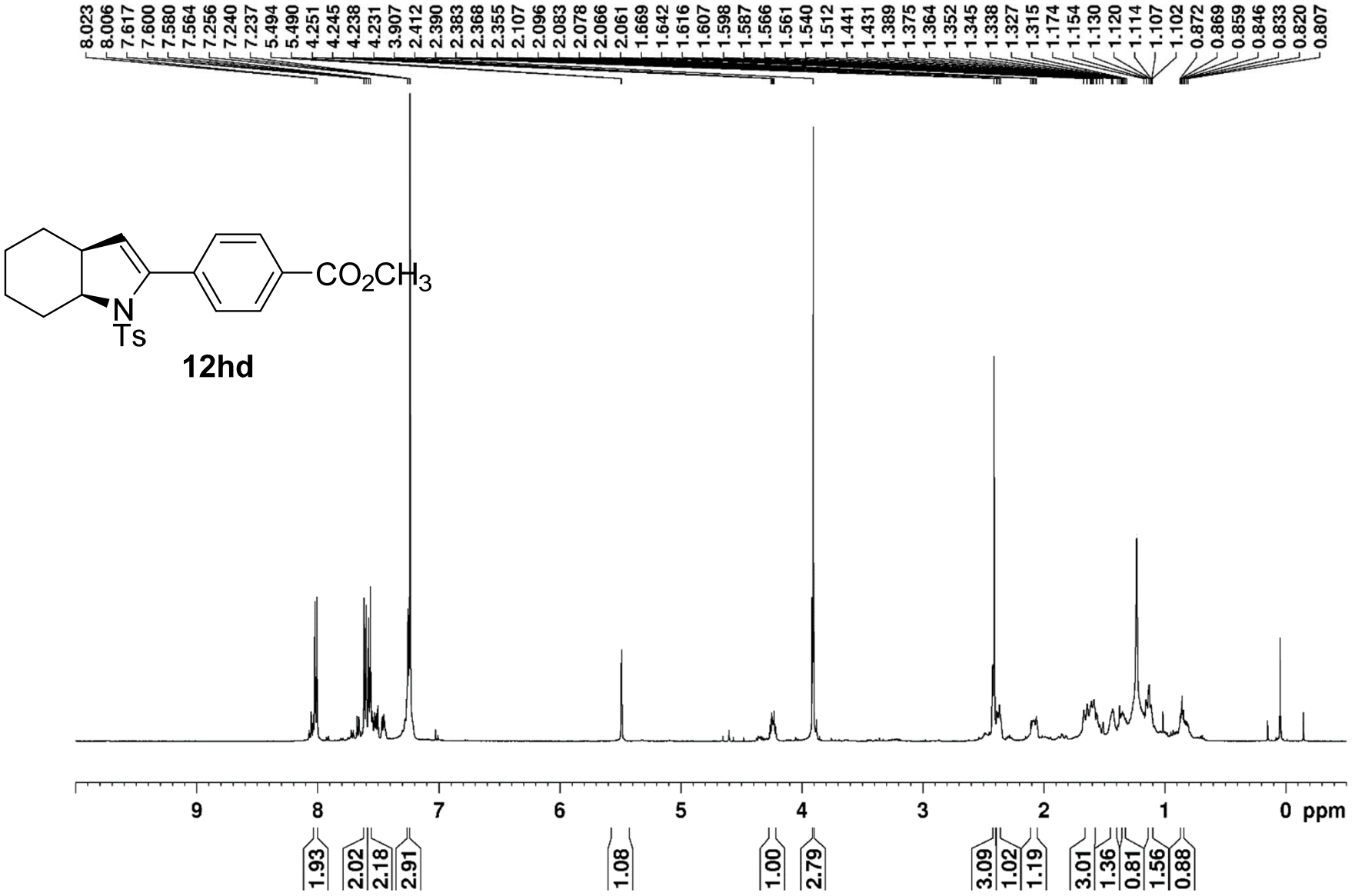
^{13}C NMR of compound **12hb** (75 MHz, $CDCl_3$)



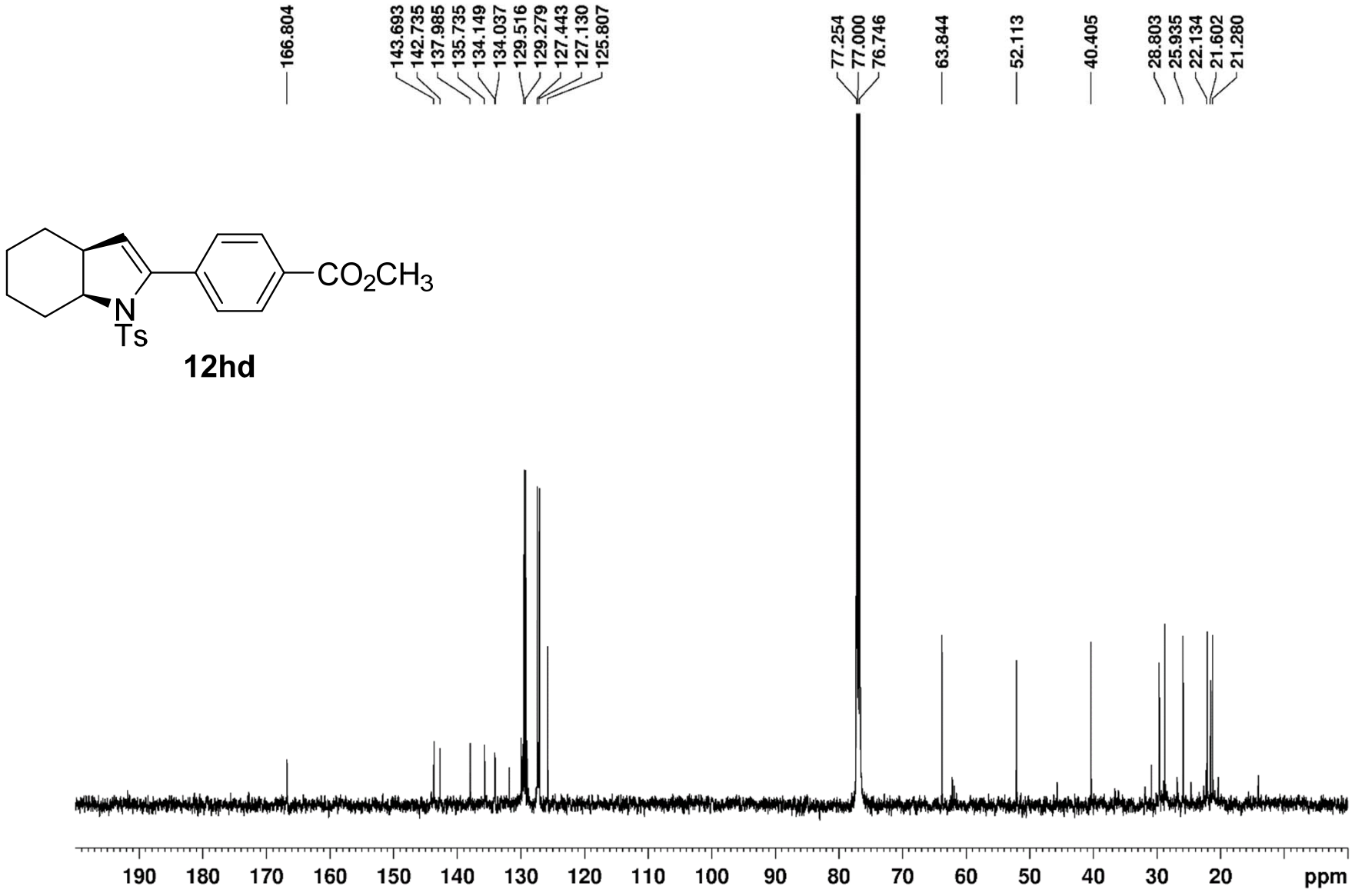
^{1}H NMR of compound **12hc** (500 MHz, $CDCl_3$)



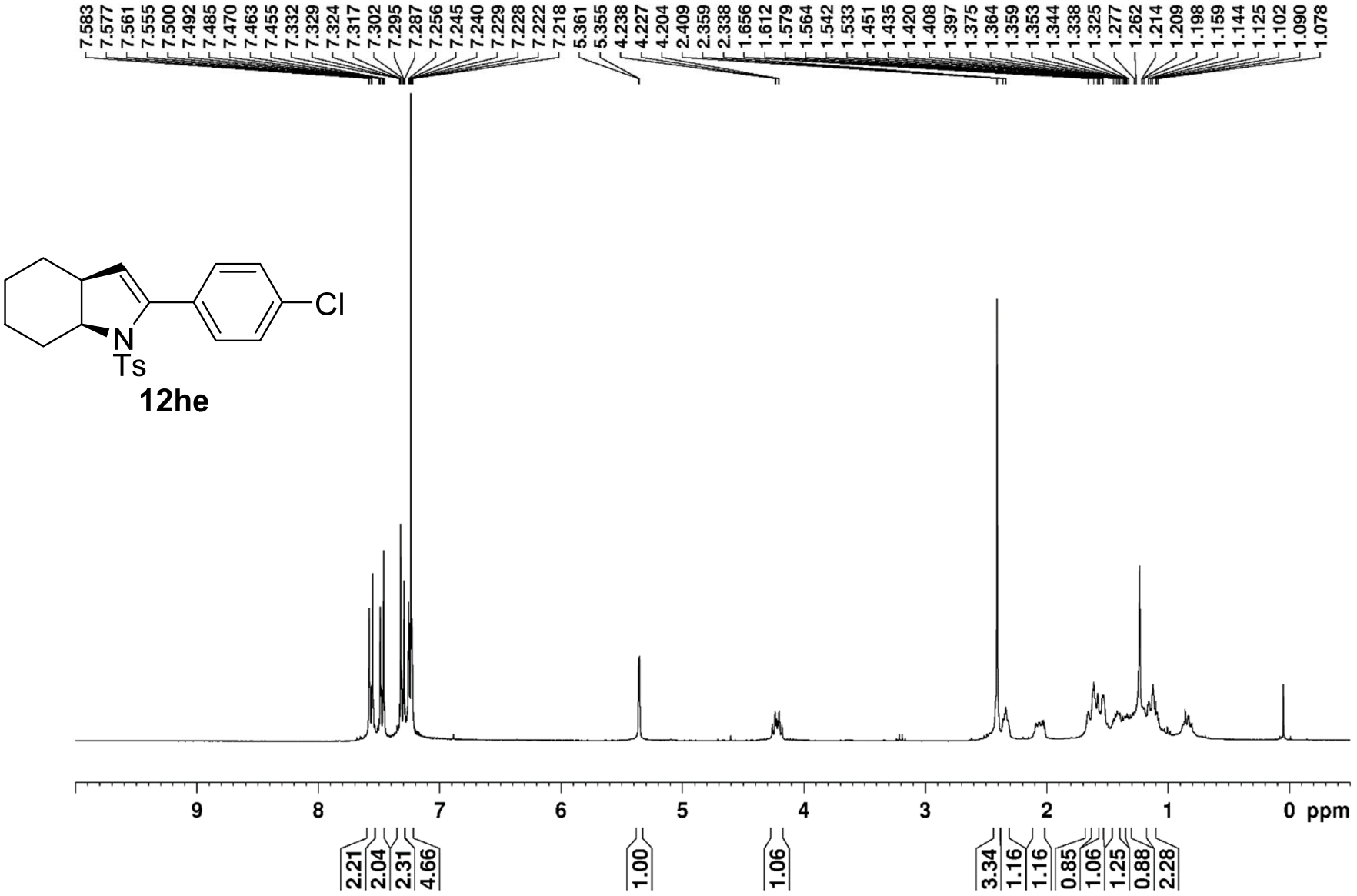
^{13}C NMR of compound **12hc** (75 MHz, $CDCl_3$)



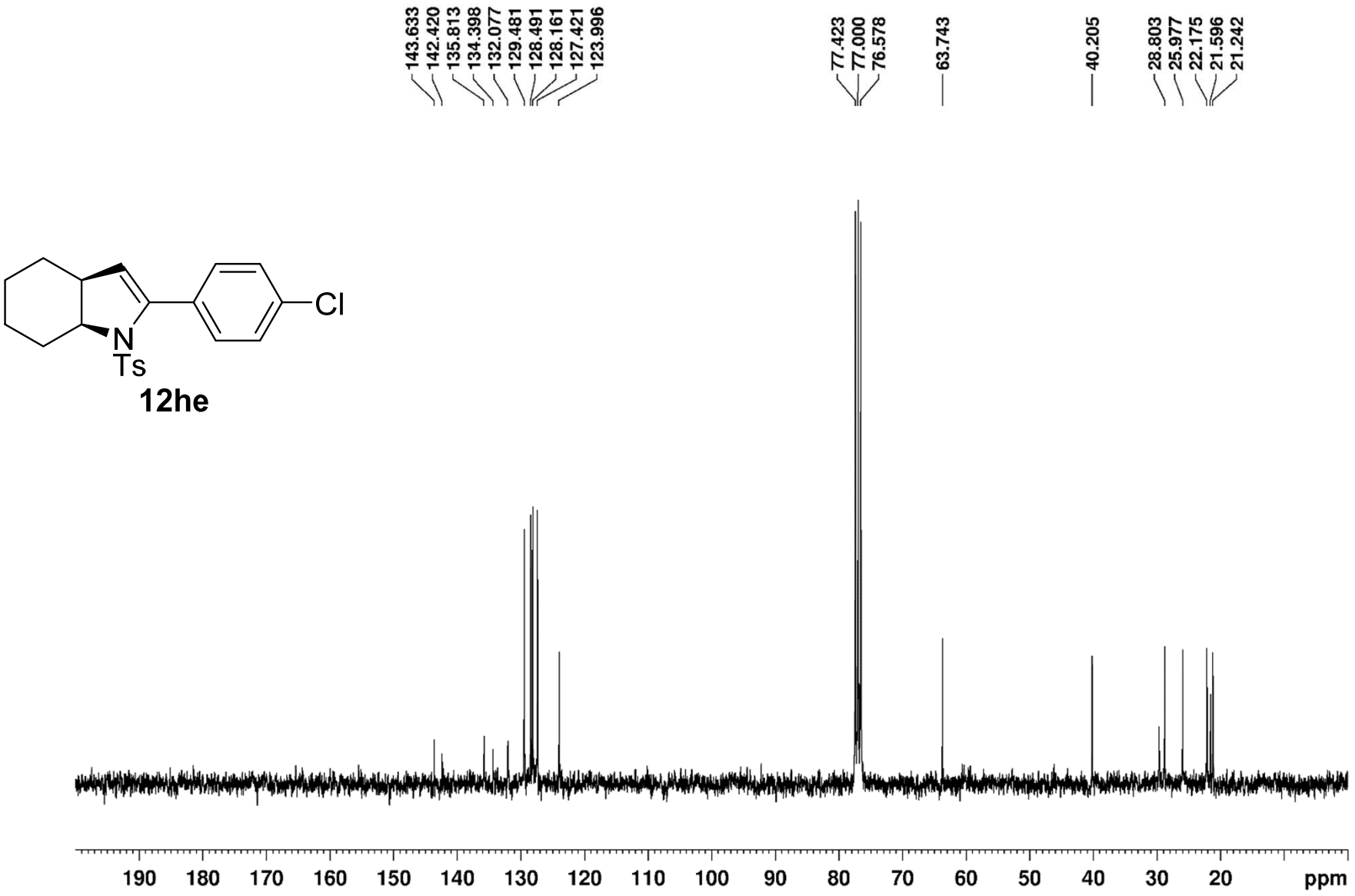
^{1}H NMR of compound **12hd** (500 MHz, $CDCl_3$)



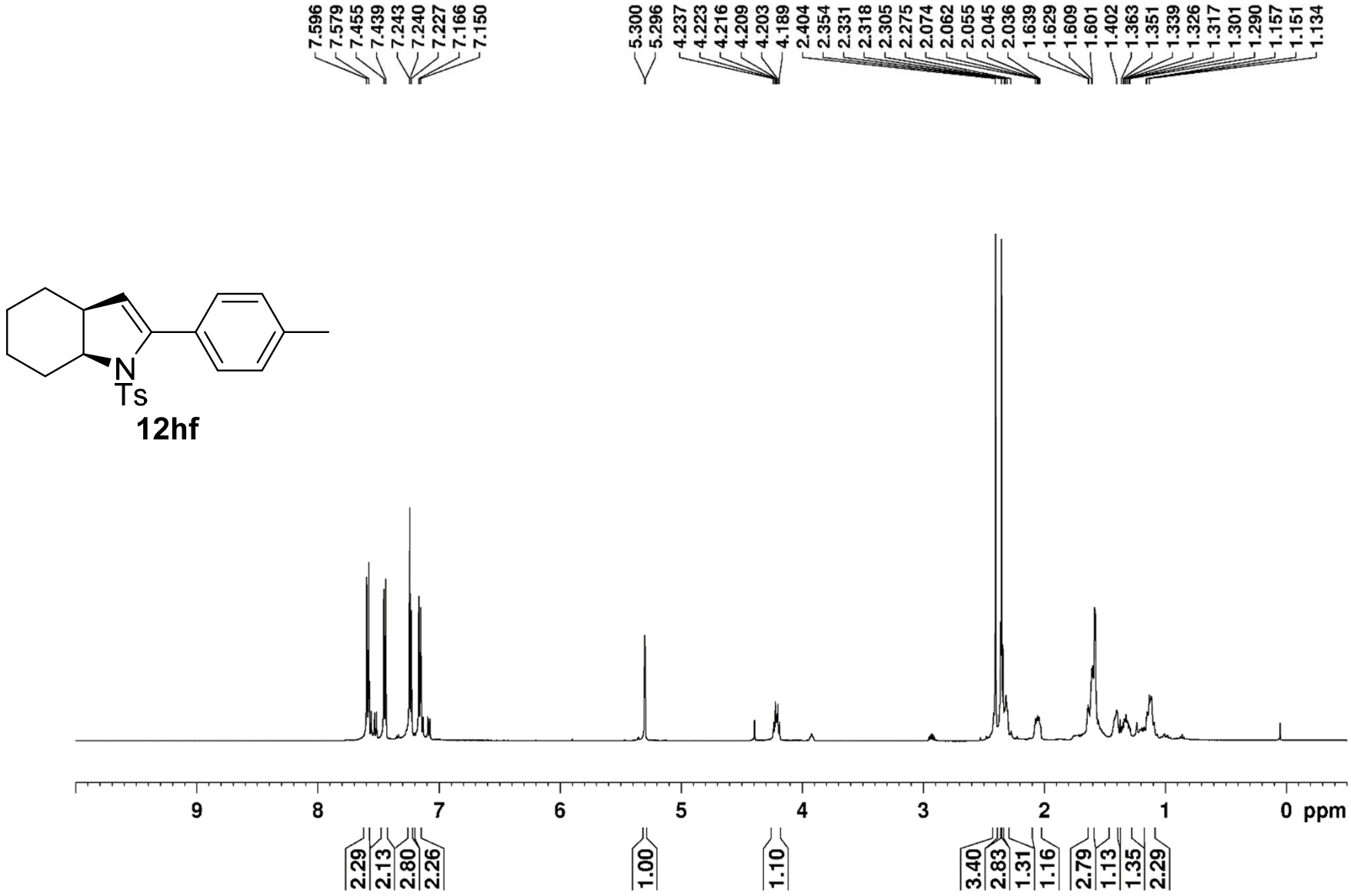
^{13}C NMR of compound **12hd** (125 MHz, $CDCl_3$)



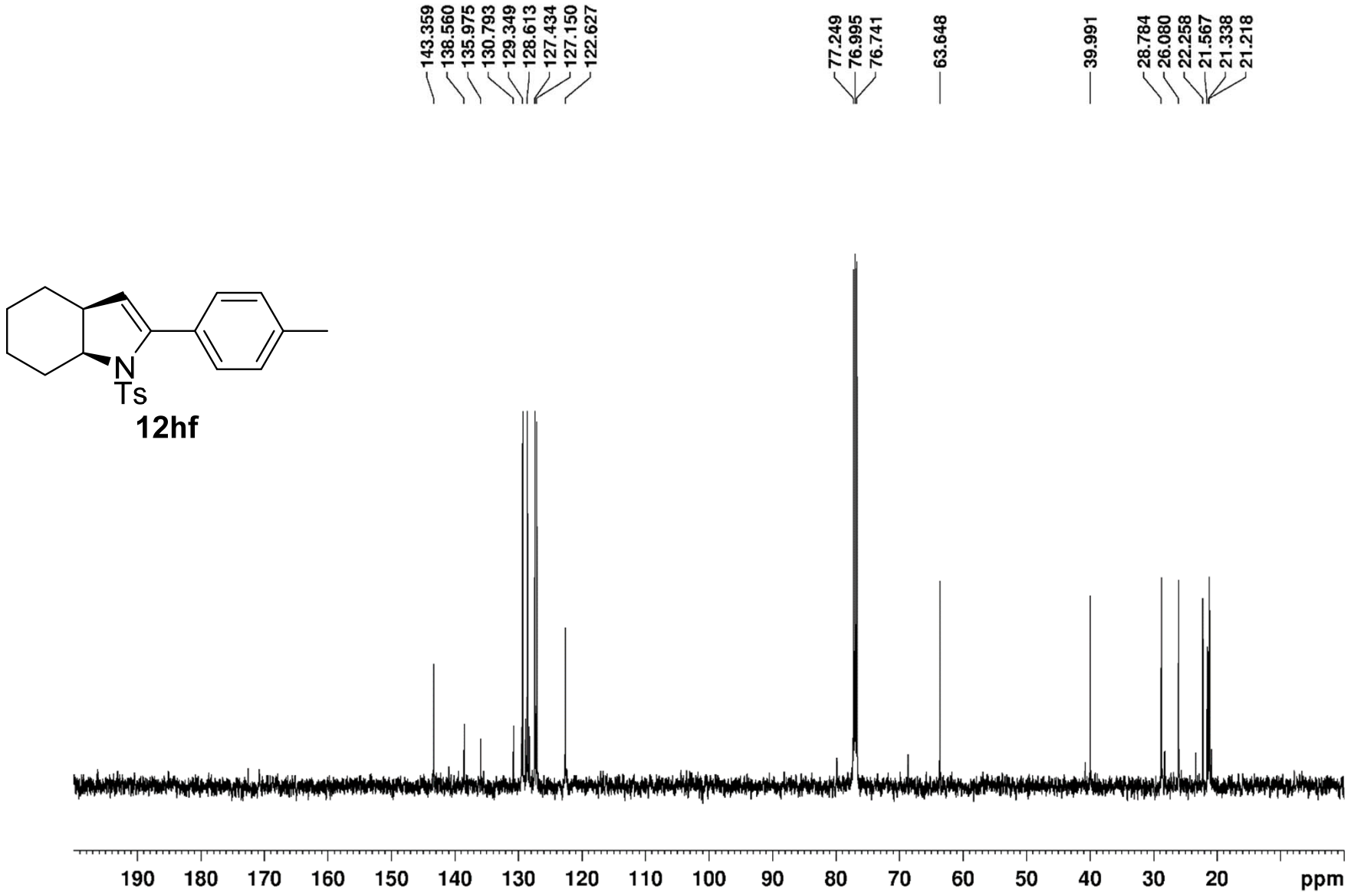
^{1}H NMR of compound **12he** (300 MHz, $CDCl_3$)



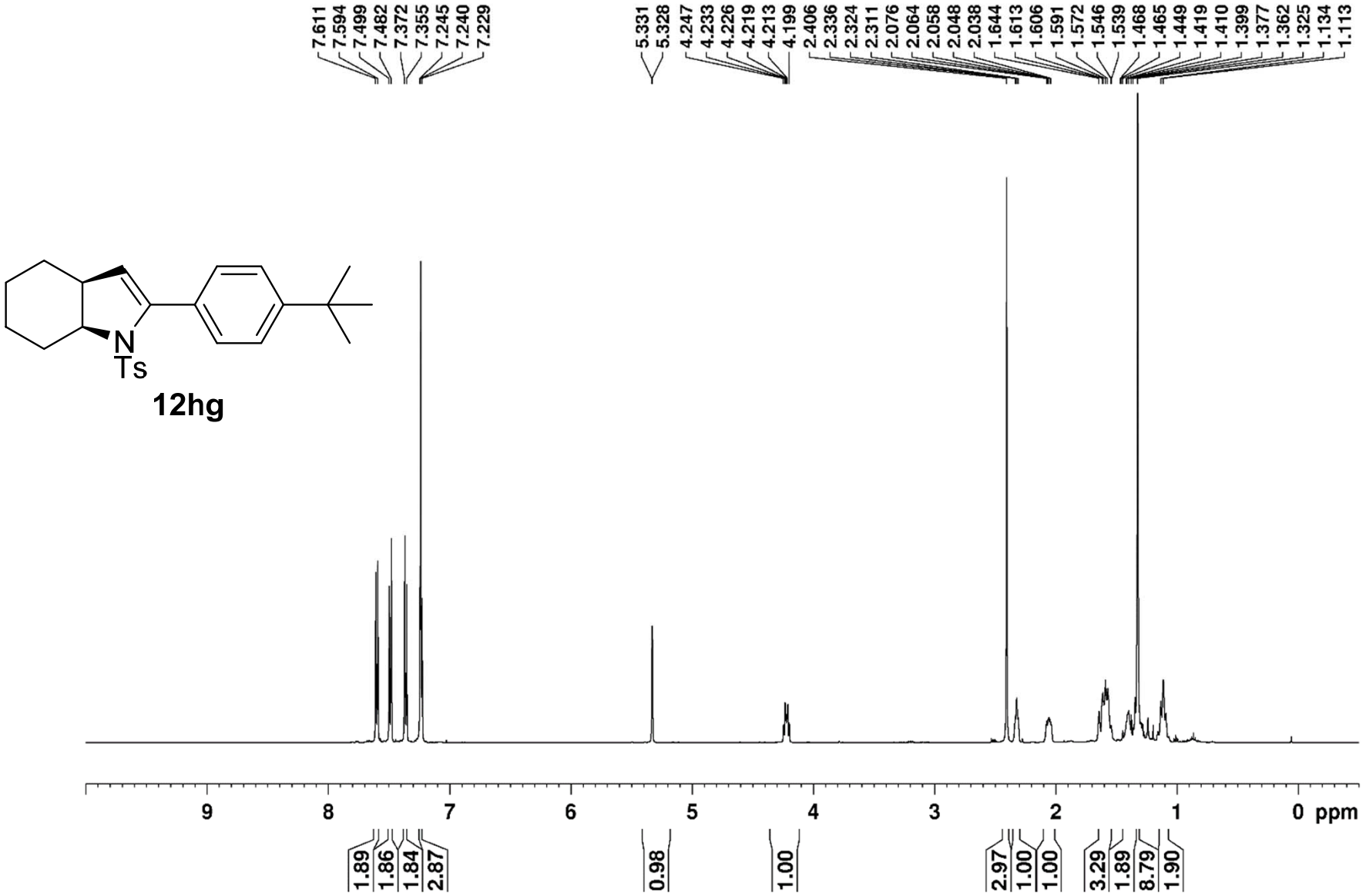
^{13}C NMR of compound **12he** (75 MHz, $CDCl_3$)



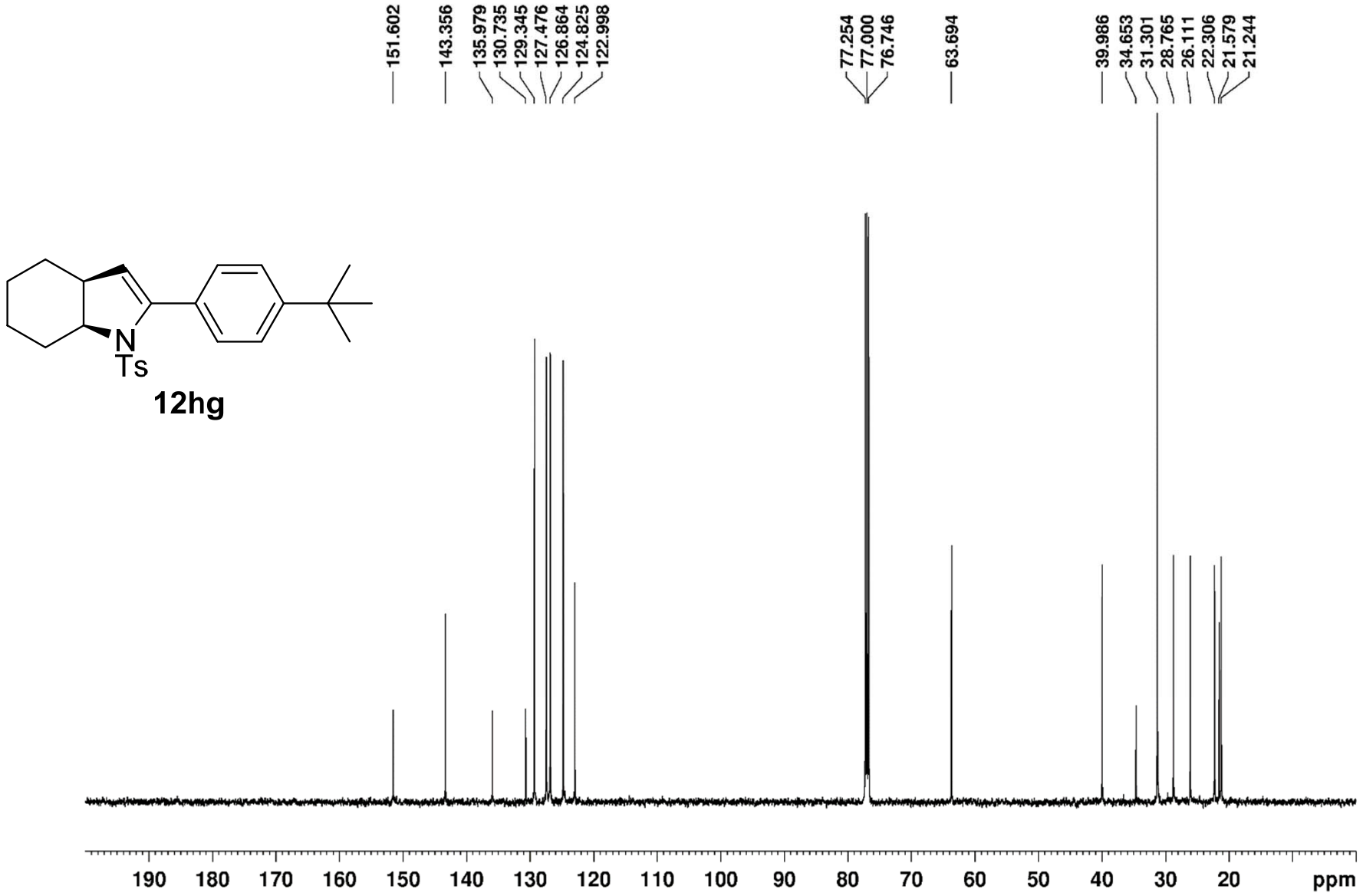
^{1}H NMR of compound **12hf** (500 MHz, $CDCl_3$)



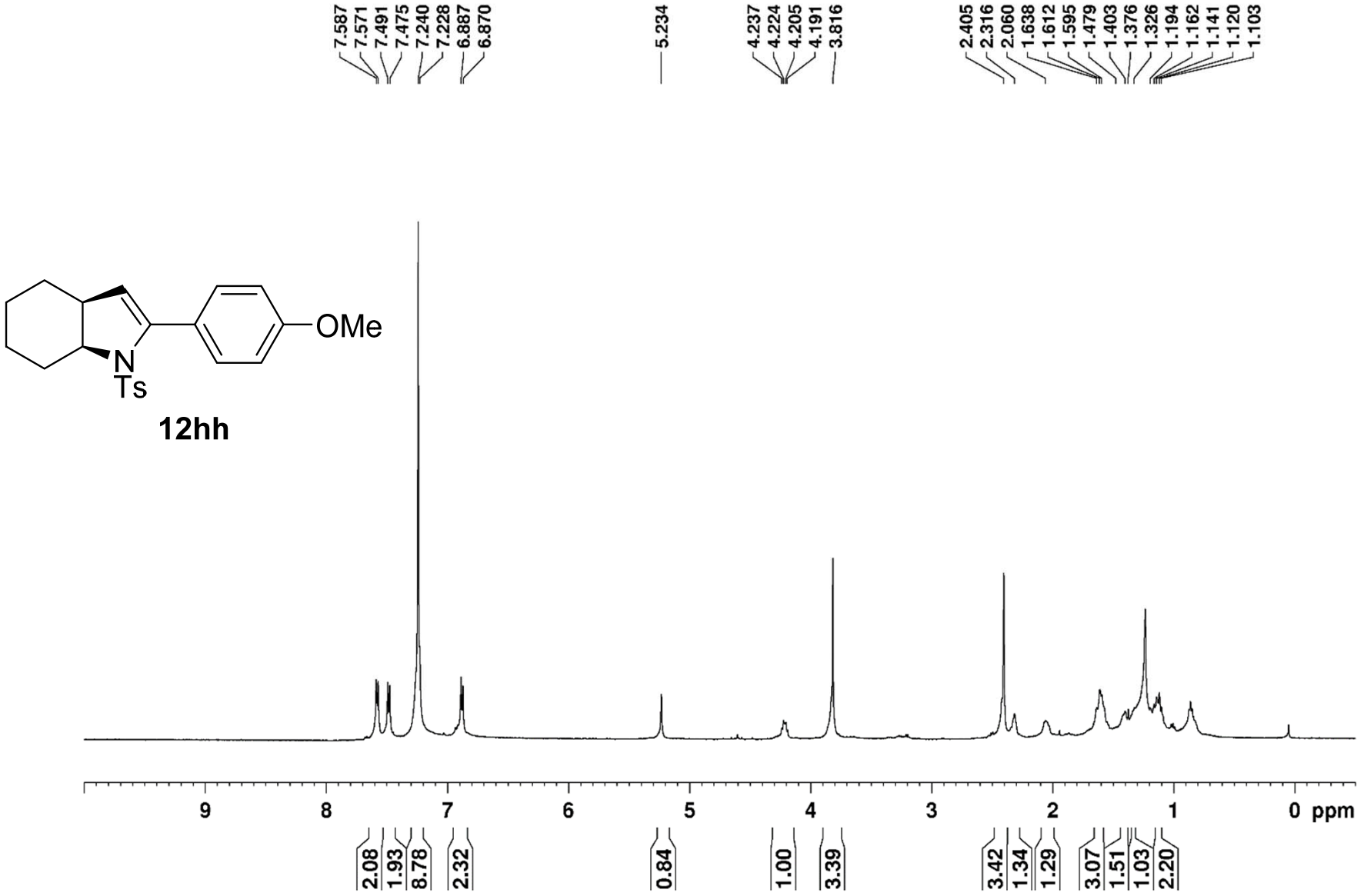
^{13}C NMR of compound **12hf** (125 MHz, $CDCl_3$)



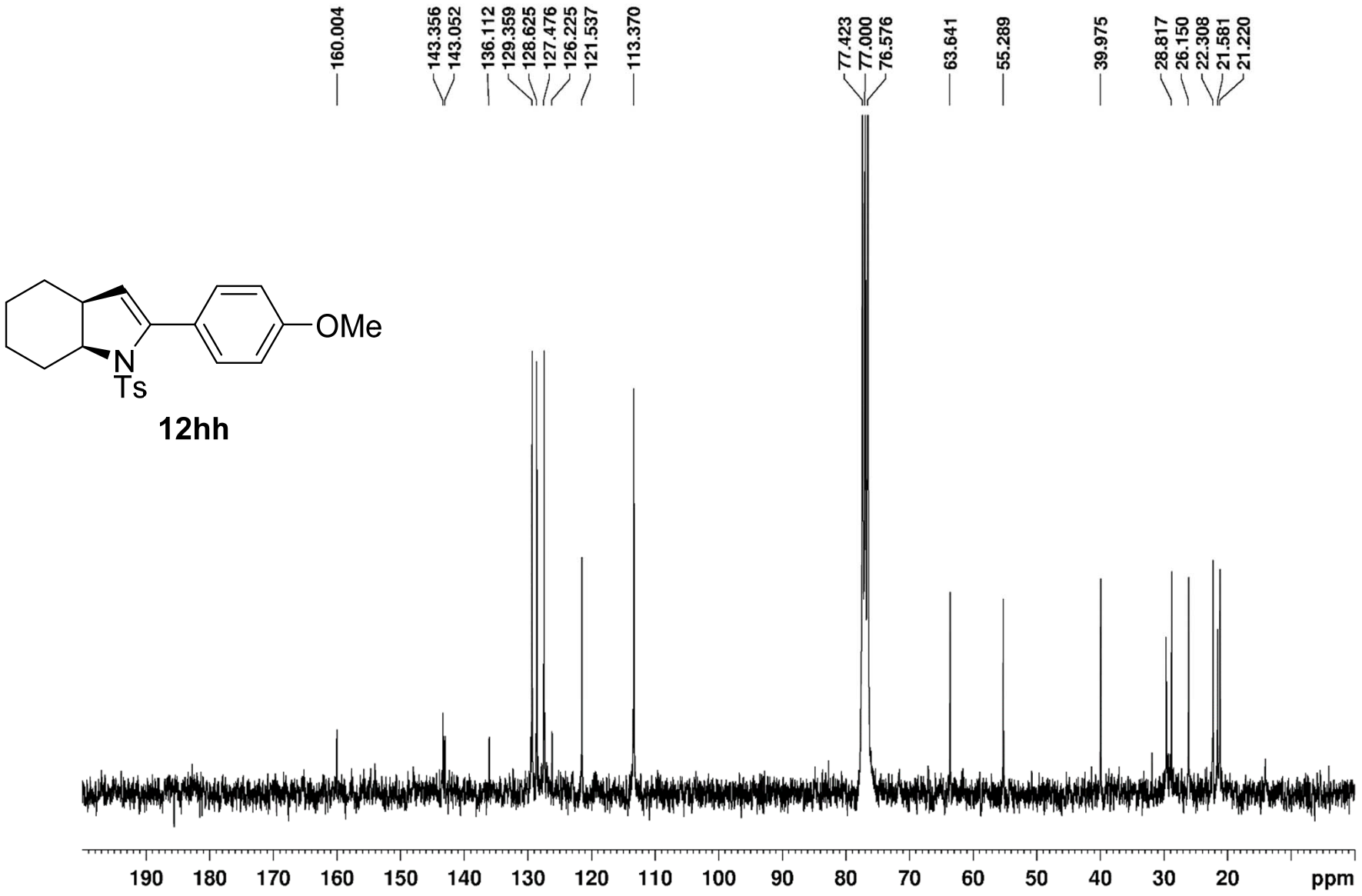
^{1}H NMR of compound **12hg** (500 MHz, $CDCl_3$)



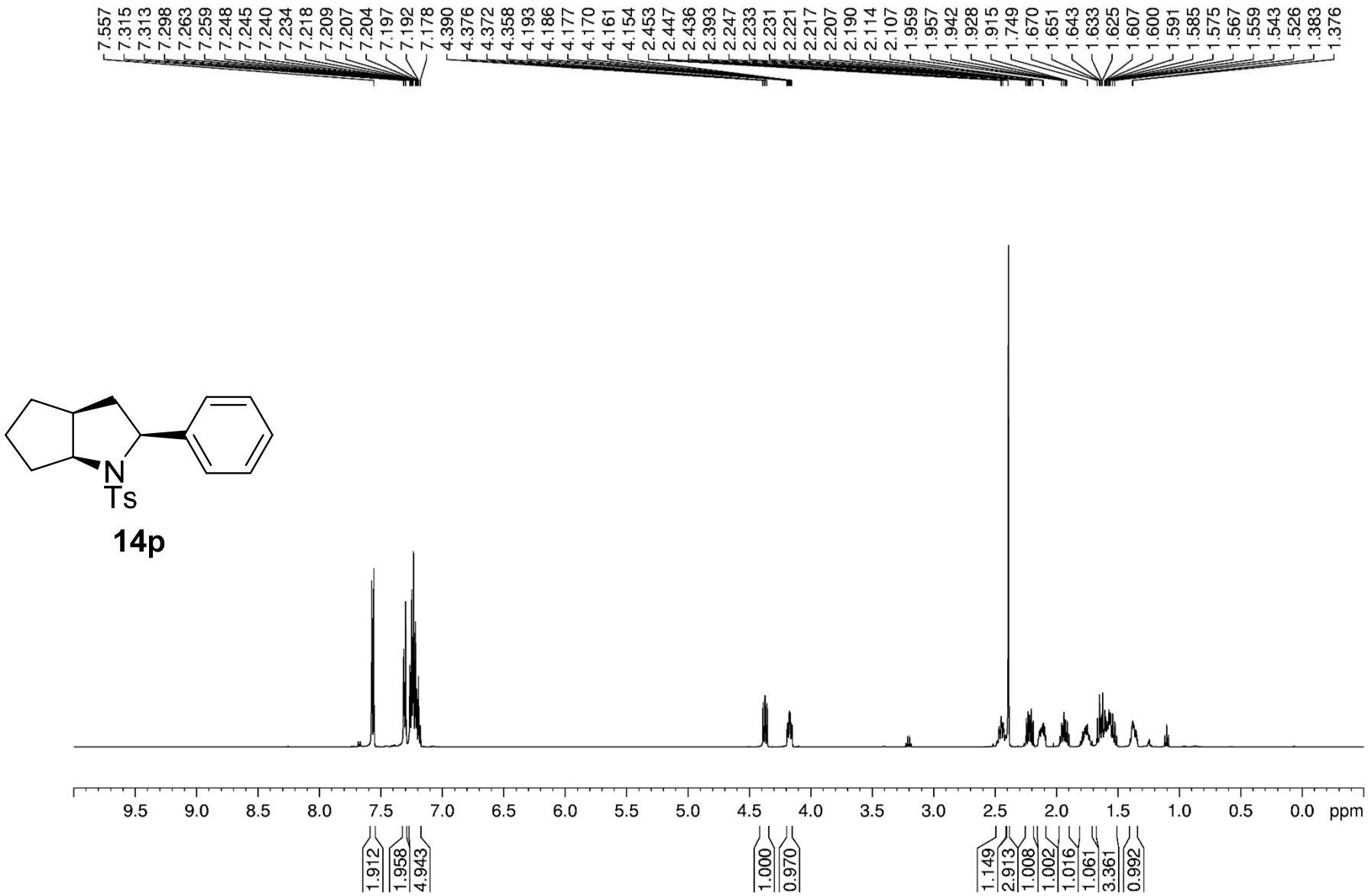
^{13}C NMR of compound **12hg** (125 MHz, $CDCl_3$)



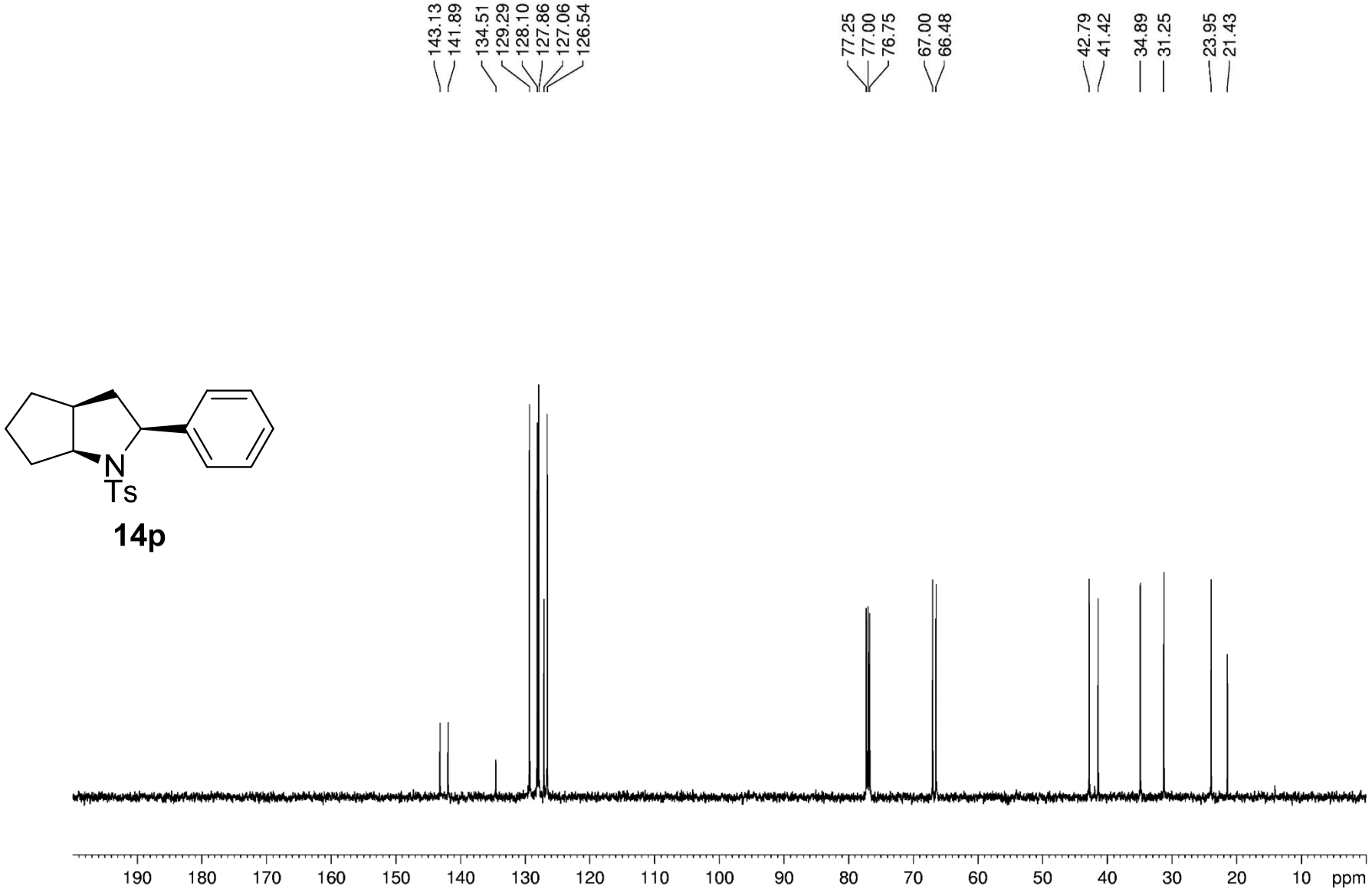
^{1}H NMR of compound **12hh** (500 MHz, $CDCl_3$)



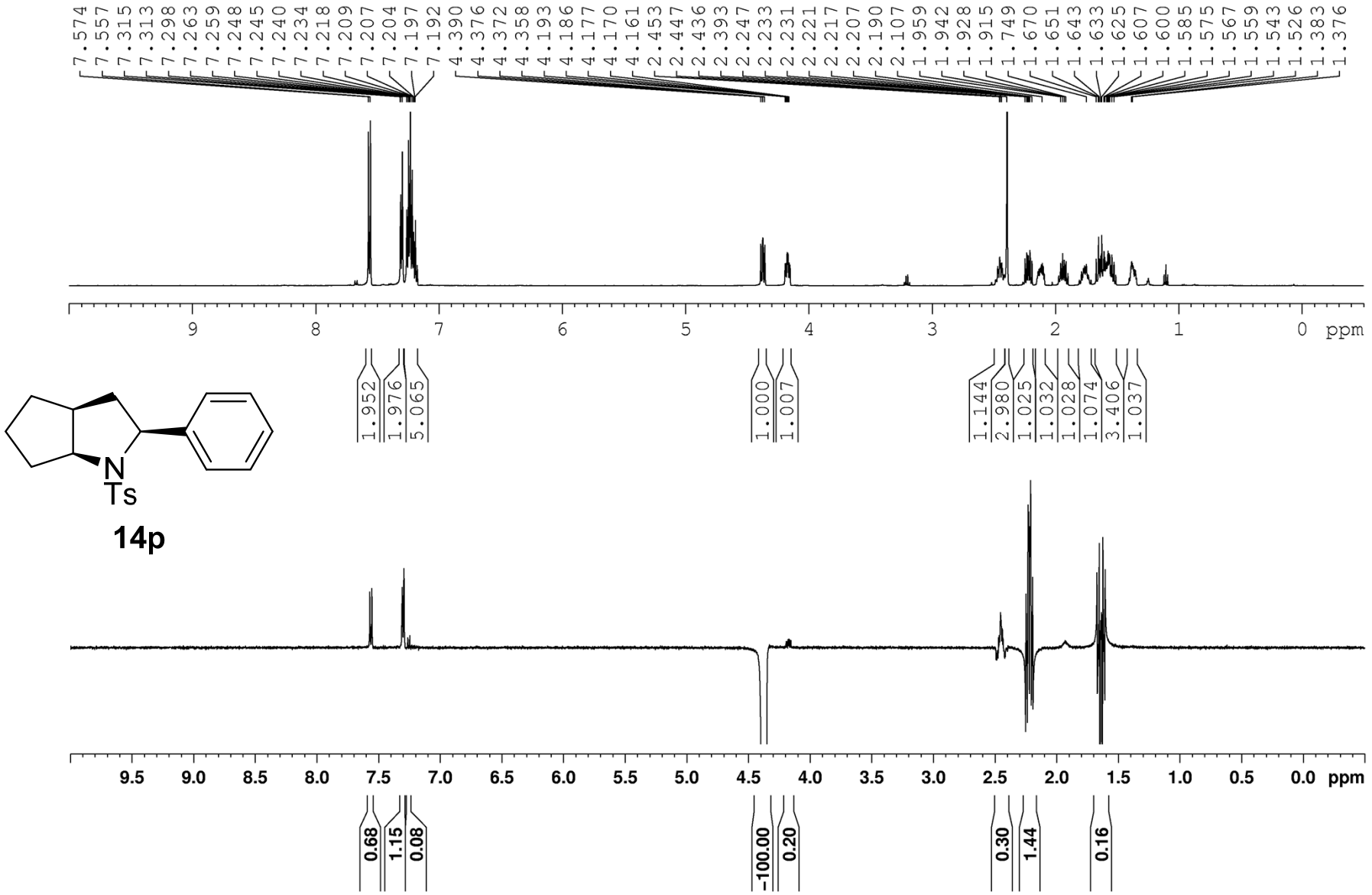
^{13}C NMR of compound **12hh** (75 MHz, $CDCl_3$)



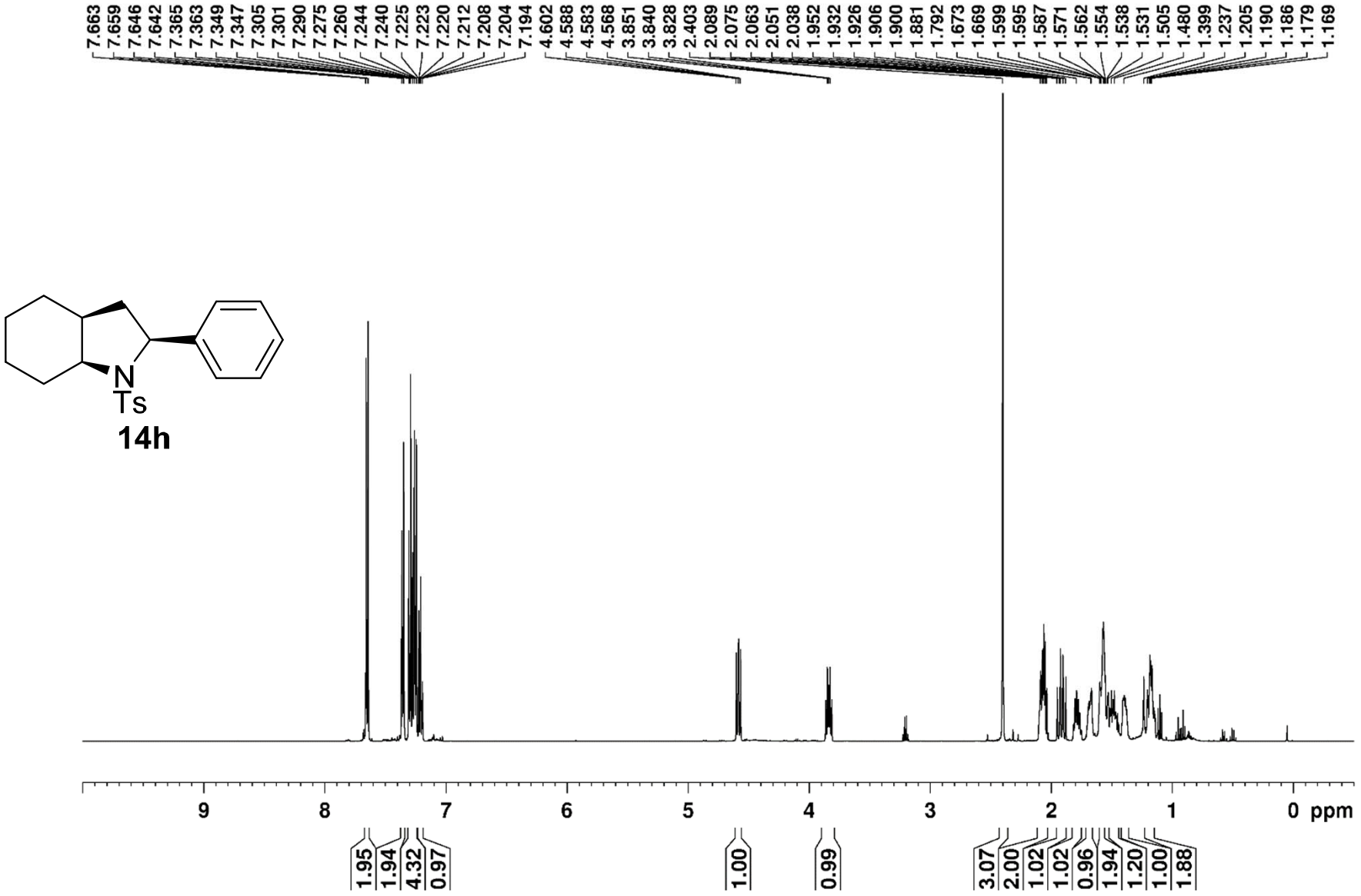
^{1}H NMR of compound **14p** (500 MHz, $CDCl_3$)



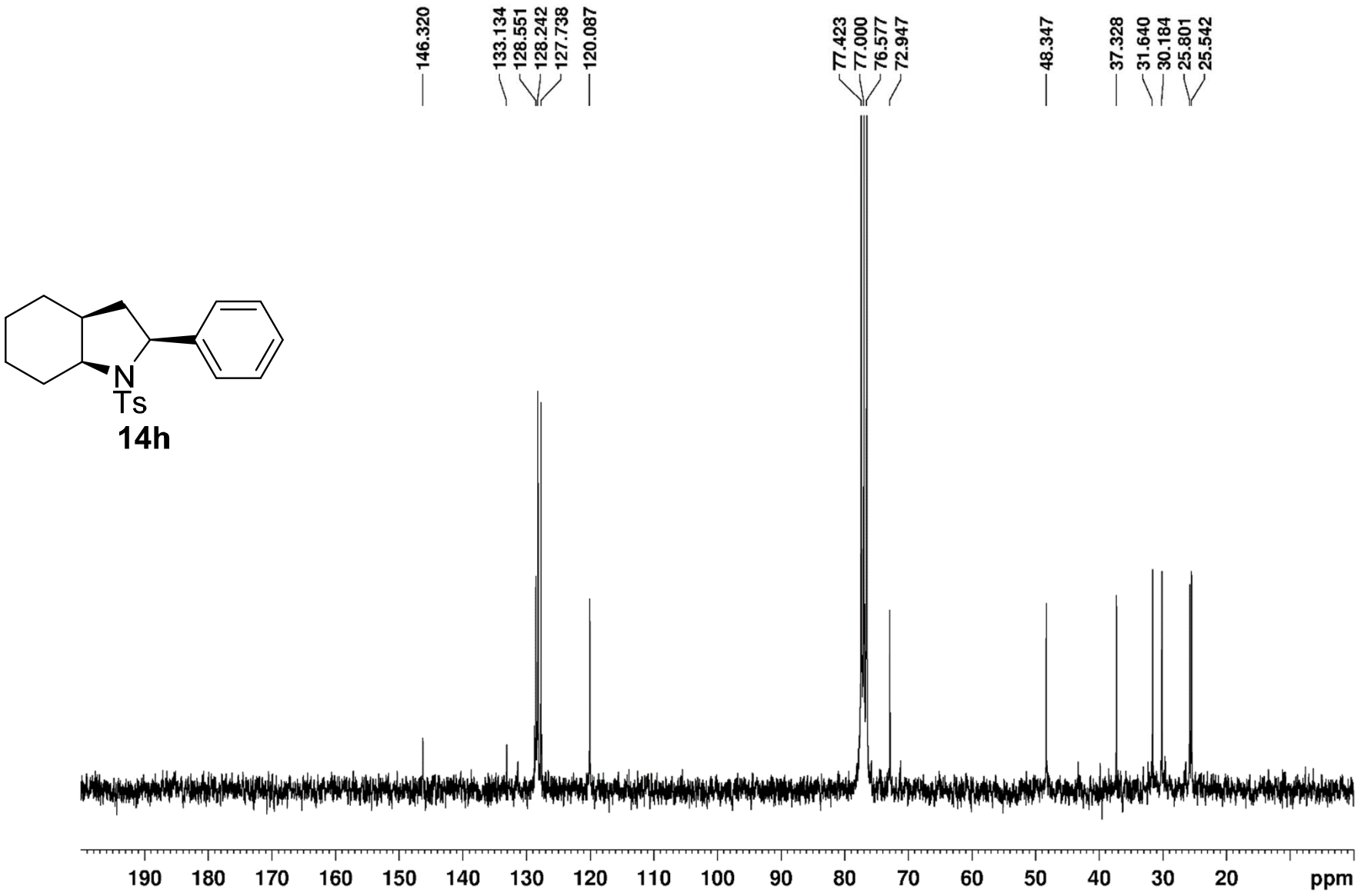
^{13}C NMR of compound **14p** (125 MHz, $CDCl_3$)



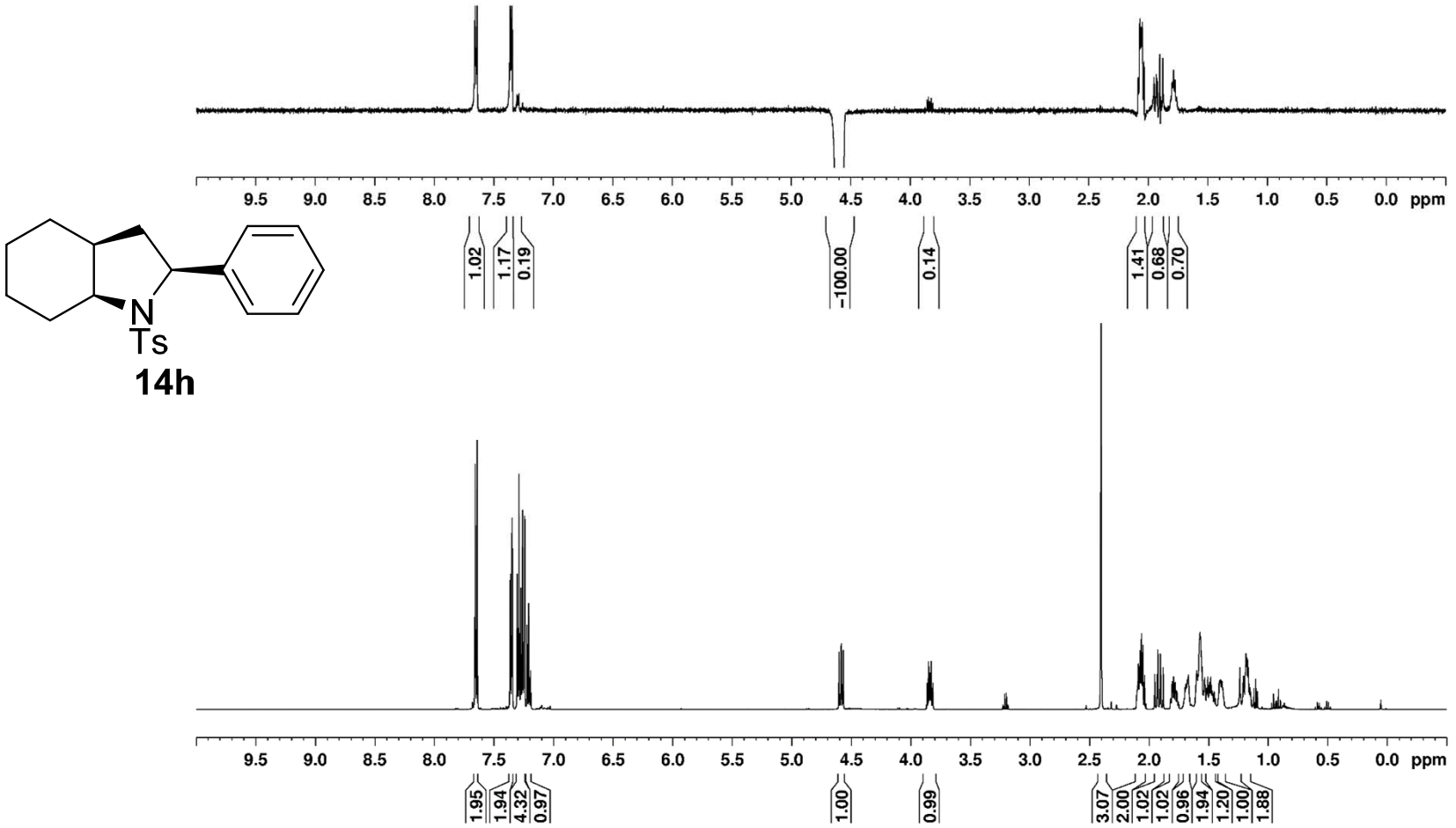
NOE of compound **14p** (500 MHz, $CDCl_3$)



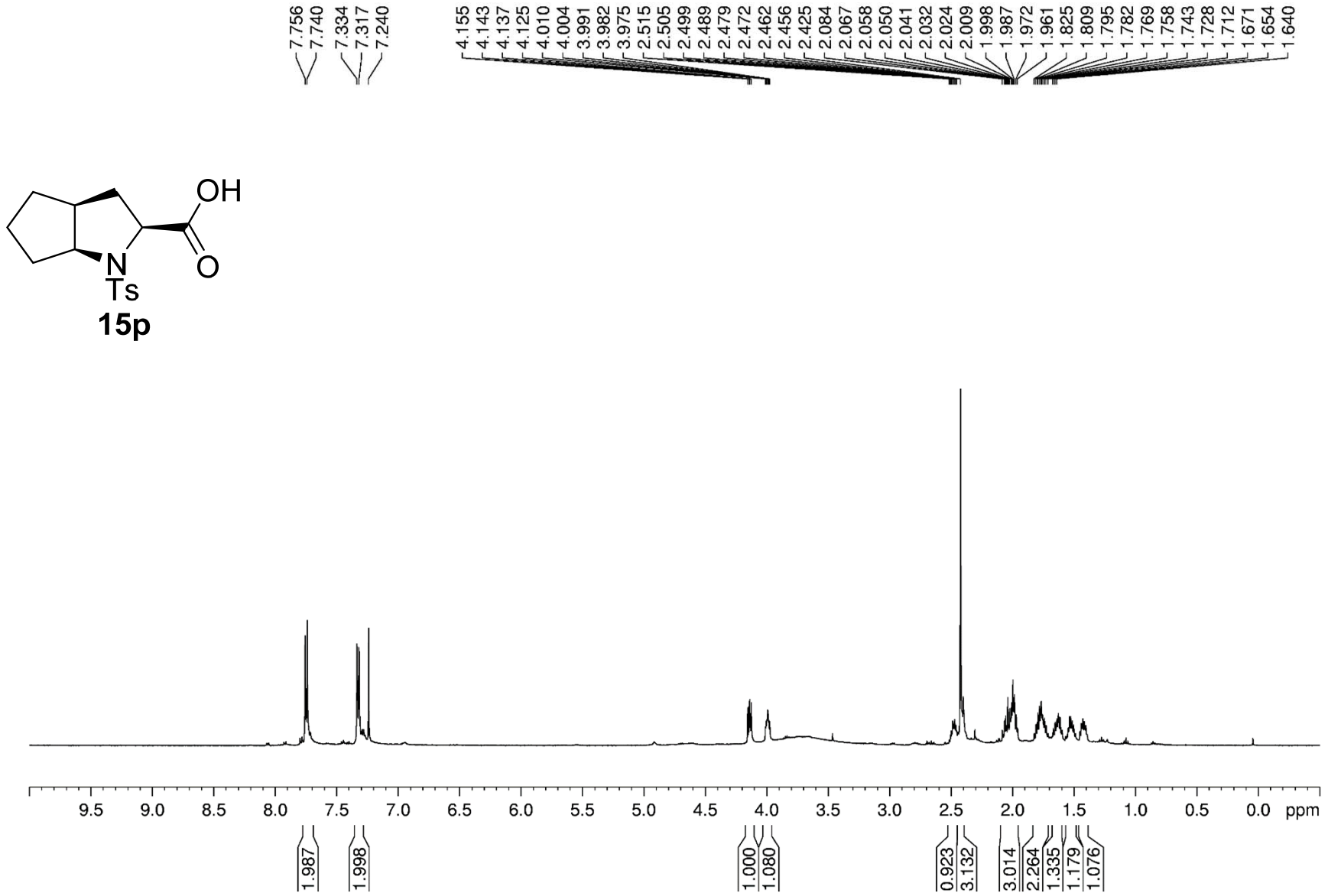
^{1}H NMR of compound **14h** (500 MHz, $CDCl_3$)



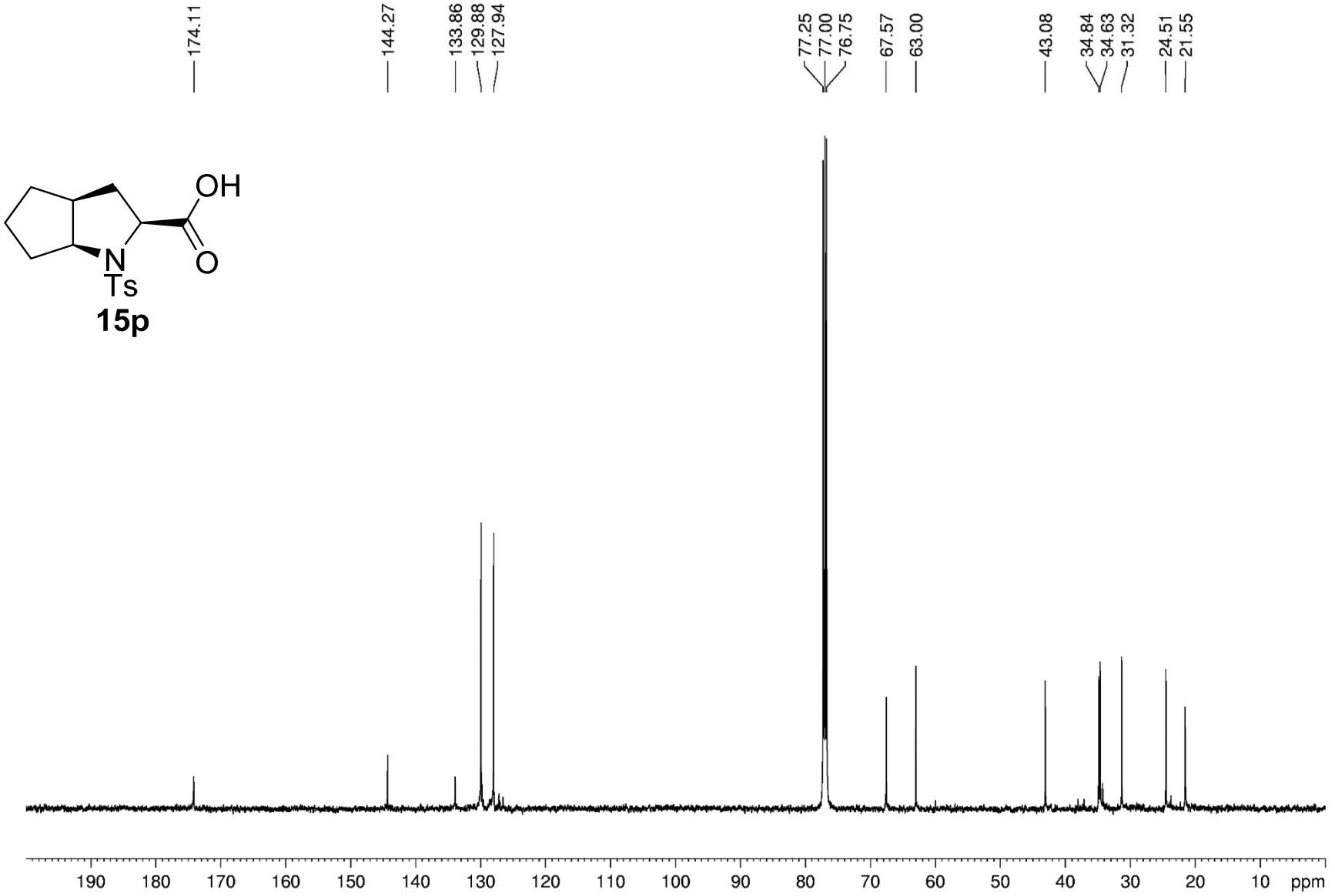
^{13}C NMR of compound **14h** (125 MHz, $CDCl_3$)



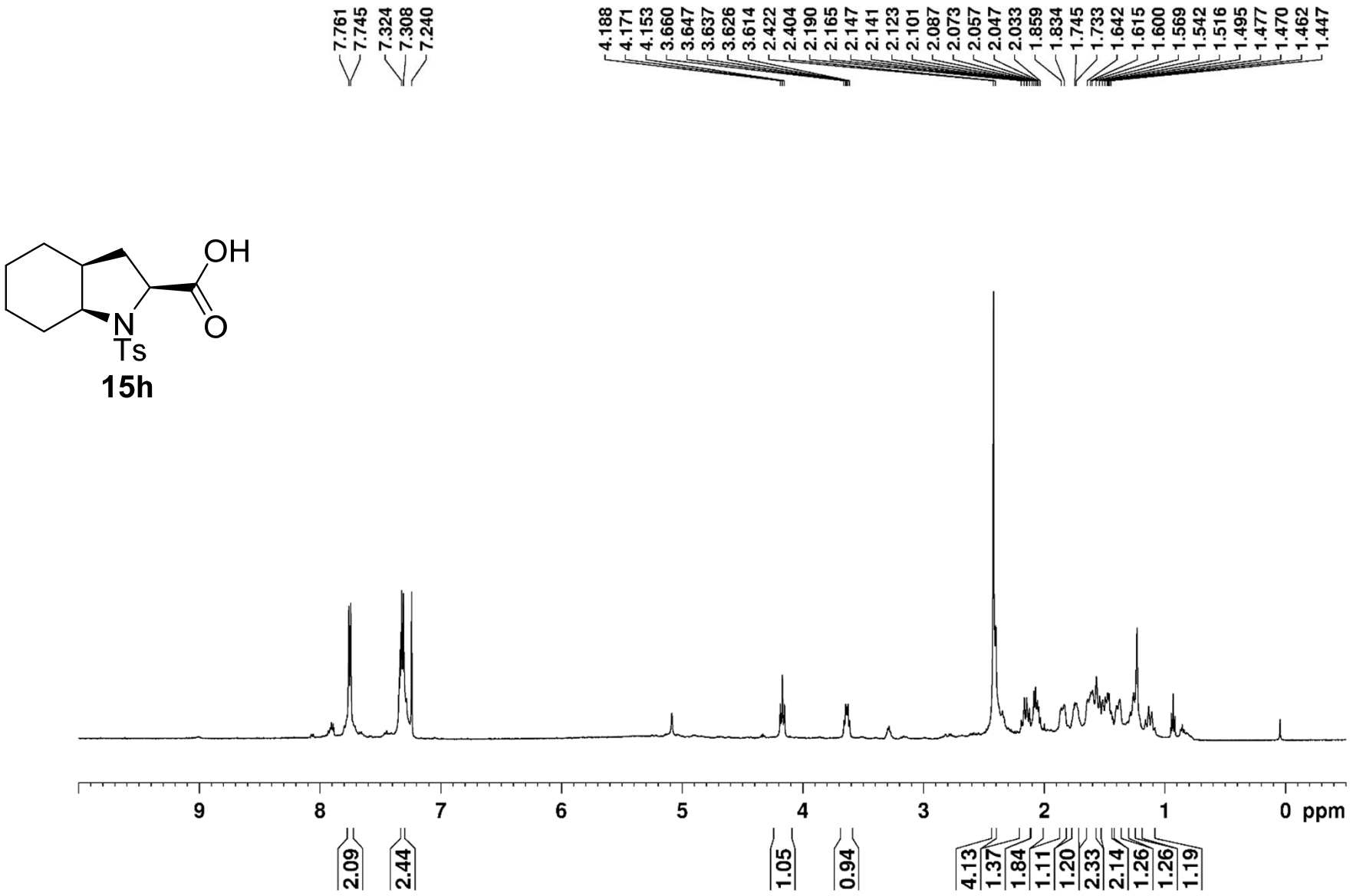
NOE of compound **14h** (500 MHz, $CDCl_3$)



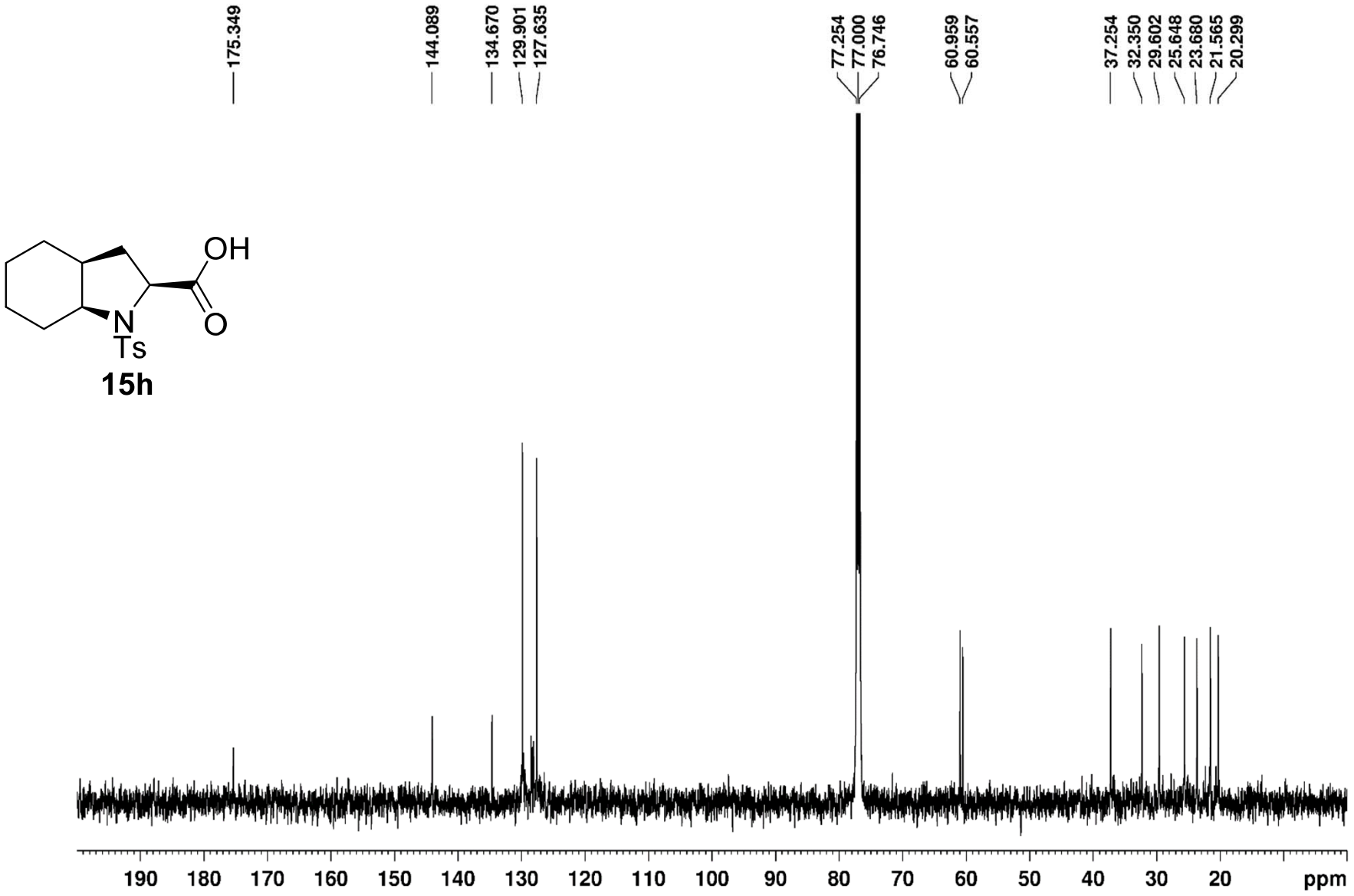
^{1}H NMR of compound **15p** (500 MHz, $CDCl_3$)



^{13}C NMR of compound **15p** (125 MHz, $CDCl_3$)



^{1}H NMR of compound **15h** (500 MHz, $CDCl_3$)



^{13}C NMR of compound **15h** (125 MHz, $CDCl_3$)