Supporting information for

p-Toluenesulfonic acid catalyzed single pot synthesis of tetracyclic 1,2-Dihydroquinolines: a metal free approach

Anil K. Hajare [a], Arun R. Jagdale [a], G. Gautham Shenoy [b] and Neelima Sinha[a,] *

[a] Novel Drug Discovery & Development, Lupin Limited, 1st Floor, Genesis Square Building, Hinjewadi, Phase-2, Village - Man, Tal. Mulshi, Dist. - Pune, Maharashtra, India, 411057.
[b] Manipal College of Pharmaceutical Sciences, Manipal University, Manipal, Karnataka, India, 576104.

Email: neelimasinha@lupin.com

Table Of Contents

General:

Commercially available reagents and solvents were used without further purification. ^{1}H and ^{13}C NMR experiments were performed on a Bruker AVANCE 400 MHz instrument, operating at 400 MHz for protons and 100 MHz for carbon nuclei. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ) in parts per million (multiplicity, coupling constant (Hz), integration). Data for ^{1}H NMR spectra are referenced to the center line of $CDCl_3$ (δ 7.26) as the internal standard. Data for ^{13}C NMR spectra are referenced to the center line of $CDCl_3$ (δ 77.0). High-resolution mass spectra were obtained on a mass spectrometer in ESI or EI mode using a TOF mass analyzer. Reaction progress was monitored by thin layer chromatography (TLC, Merck, Inc., silica gel 60 F254), visualized under UV light, and plates were developed using iodine. Flash chromatography was performed using silica gel 100 – 200 mesh. Melting points were determined using Buchi apparatus and are uncorrected.

General Procedure for the *p*-TSA-Catalyzed Synthesis of 2,2,4-Trialkyl-1,2-dihydroquinolines 8a−l (Method A). In a reaction vial containing a magnetic stir bar, was taken a mixture of aniline 6a−l (1.0 molar equiv), ketone 7 (5.0 molar equiv) and *p*-toluene sulfonic acid (0.2 molar equiv) in DMF (1 mL per mmol of substrate). Reaction vial was capped and stirred at the specified temperature (140°C) until complete disappearance of the starting material was observed by TLC. The reaction was transferred to a separatory funnel containing water and extracted with diethyl ether (3 × 30 mL). The combined organic extracts were dried over Na_2SO_4, and solvent was removed in vacuo. The residue was purified by flash column chromatography on silica gel (gradient elution with EtOAc and hexanes).

General Procedure for the *p*-TSA-Catalyzed Synthesis of 2,2,4-Trialkyl-1,2-dihydroquinolines 10a−g (Method B). In a reaction vial containing a magnetic stir bar, was taken a mixture of aniline (1.0 molar equiv), *p*-toluene sulfonic acid (0.2 molar equiv) in Ketone (2 mL per mmol of substrate) as a solvent. Reaction vial was capped and stirred at the specified temperature (100°C) until complete disappearance of starting material was observed by TLC. The solvent was evaporated under reduced pressure and residue was diluted with diethyl ether 50 mL., transferred to a separatory funnel containing water and extracted with diethyl ether (3 × 30 mL). The combined organic extracts were dried over Na_2SO_4, and solvent was removed in vacuo. The residue was purified by flash column chromatography on silica gel (gradient elution with EtOAc and hexanes).

1,2,3,5-Tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8a). Pale yellow solid; yield: 45%; mp: 83-84 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (td, *J* = 7.6, 1.2 Hz, 1H), 6.90 (dd, *J* = 7.6, 1.2 Hz, 1H), 6.63 (td, *J* = 7.6, 1.2 Hz, 1H), 6.46 (dd, *J* = 7.6, 1.2 Hz, 1H), 3.93 (br s, 1H), 2.72–2.63 (m, 2H), 2.58–2.47 (m, 2H), 2.10–1.98 (m, 2H), 1.97–1.85 (m, 2H), 1.84 – 1.64 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 142.6, 138.7, 132.1, 127.5, 123.3, 120.4, 117.1, 112.2, 65.1, 39.6, 32.2, 31.2, 23.9, 22.4; HRMS (ESI) calcd. for $C_{16}H_{19}N$ $[M+H]^+$ *m*/*z* = 225.1517, found 225.1513.

8-Methyl-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8b) White solid; yield: 46%; mp: 82-84 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.64 (d, *J* = 8.0, 1H), 6.55 (s, 1H), 6.41 (d, *J* = 8.0 Hz, 1H), 5.61 (br s, 1H), 2.55–2.40 (m, 4H), 2.11 (s, 3H), 1.92 (p, *J* = 7.2 Hz, 2H), 1.82–1.70 (m, 4H), 1.65–1.57 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.35 , 139.20 , 132.19 , 127.89 , 126.28 , 123.92 , 112.33 , 77.23 , 65.05 , 39.43 , 32.23 , 31.22 , 23.96 , 22.47 , 20.72; HRMS (ESI) calcd. for $C_{17}H_{21}N$ $[M+H]^+$ *m*/*z* = 239.1674, found 239.1671.

8-Chloro-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8c) White solid; yield: 21%; mp: 106-108 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.82 (dd, J = 8.4, 2.4 Hz, 1H), 6.69 (d, J = 2.4 Hz, 1H), 6.49 (d, J = 8.4 Hz, 1H), 6.06 (br s, 1H), 2.50–2.42 (m, 4H), 1.92 (quint, J = 7.6 Hz, 2H), 1.87–1.70 (m, 4H), 1.67–1.48 (m, 4H); ^{13}C NMR (100MHz, DMSO-d_6) δ 142.3, 139.9, 130.1, 126.7, 122.0, 120.3, 118.3, 112.8, 64.2, 40.4, 31.8, 30.5, 23.8, 21.7; HRMS (ESI) calcd. for $C_{16}H_{18}ClN$ $[M+H]^+$ m/z = 259.1128, found 259.1128.

7-Methoxy-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8e) White solid; yield: 76%; mp: 108-110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (d, J = 8.0 Hz, 1H), 6.19 (dd, J = 8.0, 2.4 Hz, 1H), 6.05 (d, J = 2.4 Hz, 1H), 3.97 (br s, 1H), 3.76 (s, 3H), 2.69–2.59 (m, 2H), 2.55–2.45 (m, 2H), 2.05–1.97 (m, 2H), 1.92–1.84 (m, 2H), 1.80–1.64 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 159.6, 143.9, 135.9, 131.7, 124.1, 114.2, 102.0, 98.3, 65.1, 55.1, 39.8, 32.0, 31.2, 23.9, 22.4; HRMS (ESI) calcd. for $C_{17}H_{21}NO$ $[M+H]^+$ m/z = 255.1623, found 255.1621.

7-Methyl-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8f) White solid; yield: 73%; mp: 77-79 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.60 (d, J = 7.6 Hz, 1H), 6.32 (s, 1H), 6.22 (d, J = 7.6Hz, 1H), 5.73 (br s, 1H), 2.50–2.41 (m, 4H), 2.11 (s, 3H), 1.91 (quint, J = 7.6 Hz, 2H), 1.84–1.70 (m, 4H), 1.65–1.49 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.5, 137.6, 137.3, 132.0, 123.1, 117.9, 112.8, 65.1, 39.7, 32.1, 31.2, 23.9, 22.4, 21.5; HRMS (ESI) calcd. for $C_{17}H_{21}N$ $[M+H]^+$ m/z = 239.1674, found 239.1669.

7-Chloro-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8g) White solid; yield: 30%; mp: 68-69 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 (d, J = 8.0 Hz, 1H), 6.56 (dd, J = 8.0, 2.0 Hz, 1H), 6.43 (d, J = 2.0 Hz, 1H), 3.97 (br s, 1H), 2.68–2.59 (m, 2H), 2.56–2.47 (m, 2H), 2.03 (quint, J = 7.6 Hz, 2H), 1.96–1.85 (m, 2H), 1.84–1.64 (m, 6H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 143.6, 138.9, 132.5, 131.4, 124.1, 118.8, 116.8, 111.7, 65.2, 40.1, 32.2, 31.1 , 23.9, 22.3; HRMS (ESI) calcd. for $C_{16}H_{18}ClN$ $[M+H]^+$ *m*/*z* = 259.1128, found 259.1123.

N-(1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentan]-7-yl)acetamide (8h) Yellow solid; yield: 76%; mp 236−238 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.57 (br s, 1H), 6.98 (d, *J* = 2.0 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 6.47 (dd, *J* = 8.0, 2.0 Hz, 1H), 5.94 (br s, 1H), 2.50–2.39 (m, 4H), 1.99 (s, 3H), 1.90 (quint, *J* = 7.2 Hz, 2H), 1.83–1.74 (m, 4H), 1.64–1.49 (m, 4H); ^{13}C-NMR (100 MHz, DMSO-d_6) δ 168.2, 144.2, 139.2, 136.9, 131.0, 123.1, 115.1, 106.7, 103.0, 64.6, 40.8, 32.1, 31.0, 24.5, 24.4, 22.3; HRMS (ESI) calcd. for $C_{18}H_{22}N_2O$ $[M+H]^+$ *m*/*z* = 282.1732, found 282.1728.

6-Methoxy-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8i) Pale **y**ellow solid; yield: 20%; mp 95-96 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.69–6.54 (m, 3H), 4.47 (br s, 1H), 3.85 (s, 3H), 2.71–2.64 (m, 2H), 2.57– 2.50 (m, 2H), 2.03 (quint, *J* = 7.2 Hz, 2H), 1.94–1.86 (m, 2H), 1.83–1.75 (m, 4H), 1.72–1.65 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 145.2, 138.6, 132.1, 132.1, 120.3, 116.1, 115.7, 109.0, 64.6, 55.6, 40.0, 32.2, 31.4, 23.9, 22.4; HRMS (ESI) calcd. for $C_{17}H_{21}NO$ $[M+H]^+$ *m*/*z* = 255.1623, found 255.1625.

6-Methyl-1,2,3,5-tetrahydrospiro[cyclopenta[c]quinoline-4,1'-cyclopentane] (8j) White solid; yield: 12%; mp 69-70 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 6.88 (d, *J* = 7.6 Hz, 1H), 6.82 (d, *J* = 7.6 Hz, 1H), 6.58 (t, *J* = 7.6 Hz, 1H), 3.83 (br s, 1H), 2.73–2.65 (m, 2H), 2.59–2.50 (m, 2H), 2.12 (s, 3H), 2.04 (quint, *J* = 7.2Hz, 2H), 1.97–1.87 (m, 2H), 1.85– 1.69 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 140.5, 138.2, 132.5, 128.9, 121.4, 119.7, 119.0, 116.4, 65.0, 40.0, 32.2, 31.4, 23.8, 22.5, 17.0; HRMS (ESI) calcd. for $C_{17}H_{21}N$ $[M+H]^+$ *m*/*z* = 239.1674, found 239.1669.

7',8',9',10'-tetrahydro-5'H-spiro[cyclohexane-1,6'-phenanthridine] (8k) Oil; yield: 40%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.00 (td, *J* = 7.6, 1.2 Hz, 1H), 6.71 (td, *J* = 7.6, 1.2 Hz, 1H), 6.59 (dd, *J* = 7.6, 1.2 Hz, 1H), 4.43 (br s, 1H), 2.43–2.37 (m, 2H), 2.17–2.11 (m, 2H), 1.82–1.66 (m, 8H), 1.65 –1.59 (m, 2H), 1.56–1.41 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 142.0, 135.9, 127.1, 125.2, 124.3, 122.1, 117.9, 113.6, 77.1, 54.6, 32.4, 25.4, 25.2, 24.9, 23.1, 22.4, 21.2; HRMS (ESI) calcd. for $C_{18}H_{23}N$ $[M+H]^+$ *m*/*z* = 253.1830, found 253.1831.

3'-methoxy-7',8',9',10'-tetrahydro-5'H-spiro[cyclohexane-1,6'-phenanthridine] (8l) White solid; yield: 63%; mp 102-103 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (d, *J* = 8.4 Hz, 1H), 6.27 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.18 (d, *J* = 2.4 Hz, 1H), 4.42 (br s, 1H), 3.79 (s, 3H), 2.41–2.33 (m, 2H), 2.16–2.09 (m, 2H), 1.80–1.41 (m, 14H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 143.4, 133.2, 124.9, 123.1, 118.0, 102.9, 99.4, 55.1, 54.7, 32.5, 25.4, 25.3, 24.7, 23.2, 22.5, 21.1; HRMS (ESI) calcd. for $C_{19}H_{25}NO$ $[M+H]^+$ *m*/*z* = 283.1936, found 283.1934.

2,2,4-Trimethyl-1,2-dihydroquinoline (10a) Oil; yield: 43%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (dd, *J* = 7.6, 1.2 Hz, 1H), 6.99 (td, *J* = 7.6, 1.2 Hz, 1H), 6.64 (td, *J* = 7.6, 1.2 Hz, 1H), 6.45 (dd, *J* = 7.6, 1.2 Hz, 1H), 5.33 (q, *J* = 1.2 Hz, 1H), 3.74 (br s, 1H), 1.99 (d, *J* = 1.2 Hz, 3H), 1.28 (s, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 143.2, 128.6, 128.39, 128.36, 123.6, 121.6, 117.2, 113.0, 51.8, 31.0, 18.6; HRMS (ESI) calcd. for $C_{12}H_{15}N$ $[M+H]^+$ *m*/*z* = 173.1204, found 173.1208.

7-Methoxy-2,2,4-trimethyl-1,2-dihydroquinoline (10b) White solid; yield: 56%; mp 68−69 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (d, *J* = 8.4 Hz, 1H), 6.22 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.06 (d, *J* = 2.4 Hz, 1H), 5.20 (s, 1H), 3.78 (br s, 1H), 3.75 (s, 3H), 1.96 (s, 3H), 1.27 (s, 6H); ^{13}C-

NMR (100 MHz, $CDCl_3$) δ 160.2, 144.7, 130.6, 128.2, 126.2, 124.7, 115.3, 102.3, 98.6, 55.1, 51.9, 31.1, 18.7; HRMS (ESI) calcd. for $C_{13}H_{17}NO$ $[M+H]^+$ *m*/*z* = 203.1310, found 203.1306.

2,2,4,7-tetramethyl-1,2-dihydroquinoline (10c) Oil; yield: 52%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (d, *J* = 7.6 Hz, 1H), 6.51 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.33 (d, *J* = 1.6 Hz, 1H), 5.29 (d, *J* = 1.6 Hz, 1H), 3.69 (br s, 1H), 2.27 (s, 3H), 2.01 (d, *J* = 1.6 Hz, 3H), 1.31 (s, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 143.23, 138.32, 128.53, 127.46, 123.60, 119.18, 118.10, 113.68, 51.86, 31.04, 21.45, 18.68; HRMS (ESI) calcd. for $C_{13}H_{17}N$ $[M+H]^+$ *m*/*z* = 187.1361, found 187.1361.

7-chloro-2,2,4-trimethyl-1,2-dihydroquinoline (10d) Oil; yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.97 (d, *J* = 8.0 Hz, 1H), 6.61 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.46 (d, *J* = 2.0 Hz, 1H), 5.33 (d, *J* = 1.6 Hz, 1H), 3.92 (s, 1H), 1.98 (s, 3H), 1.30 (s, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.22, 133.54, 128.44, 127.84, 124.67, 120.06, 116.95, 112.47, 52.15, 31.10, 18.57; HRMS (ESI) calcd. for C12H14ClN $[M+H]^+$ *m*/*z* = 207.0815, found 207.0817.

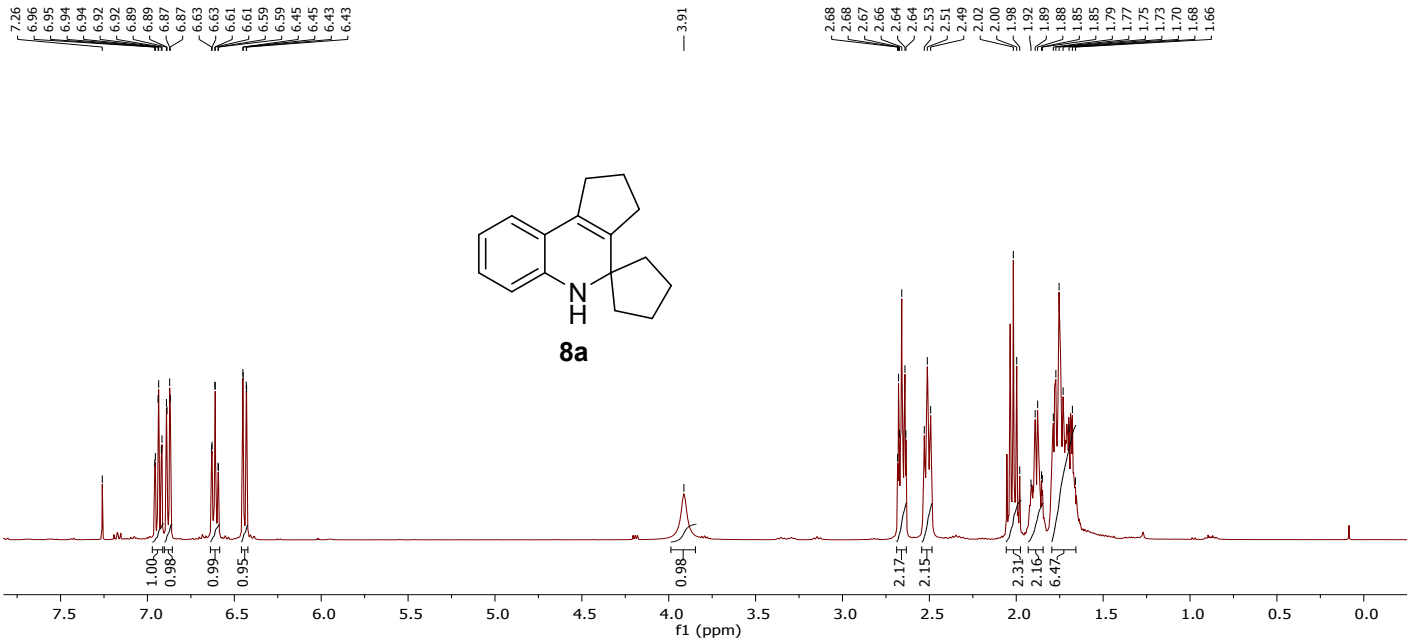
2,4-diethyl-2-methyl-1,2-dihydroquinoline (10e) Oil; yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.00 (td, *J* = 7.6, 1.2 Hz, 1H), 6.64 (td, *J* = 7.6, 1.2 Hz, 1H), 6.47 (dd, *J* = 8.0, 1.2 Hz, 1H), 5.24 (d, *J* = 1.2 Hz, 1H), 3.63 (br s, 1H), 2.43 (q, *J* = 7.2 Hz, 2H), 1.61 – 1.48 (m, 2H), 1.27 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 143.96, 134.80, 128.20, 125.11, 123.22, 120.60, 116.70, 112.83, 54.61, 36.51, 29.45, 24.62, 12.94, 8.59; HRMS (ESI) calcd. for $C_{14}H_{19}N$ $[M+H]^+$ *m*/*z* = 201.1517, found 201.1518.

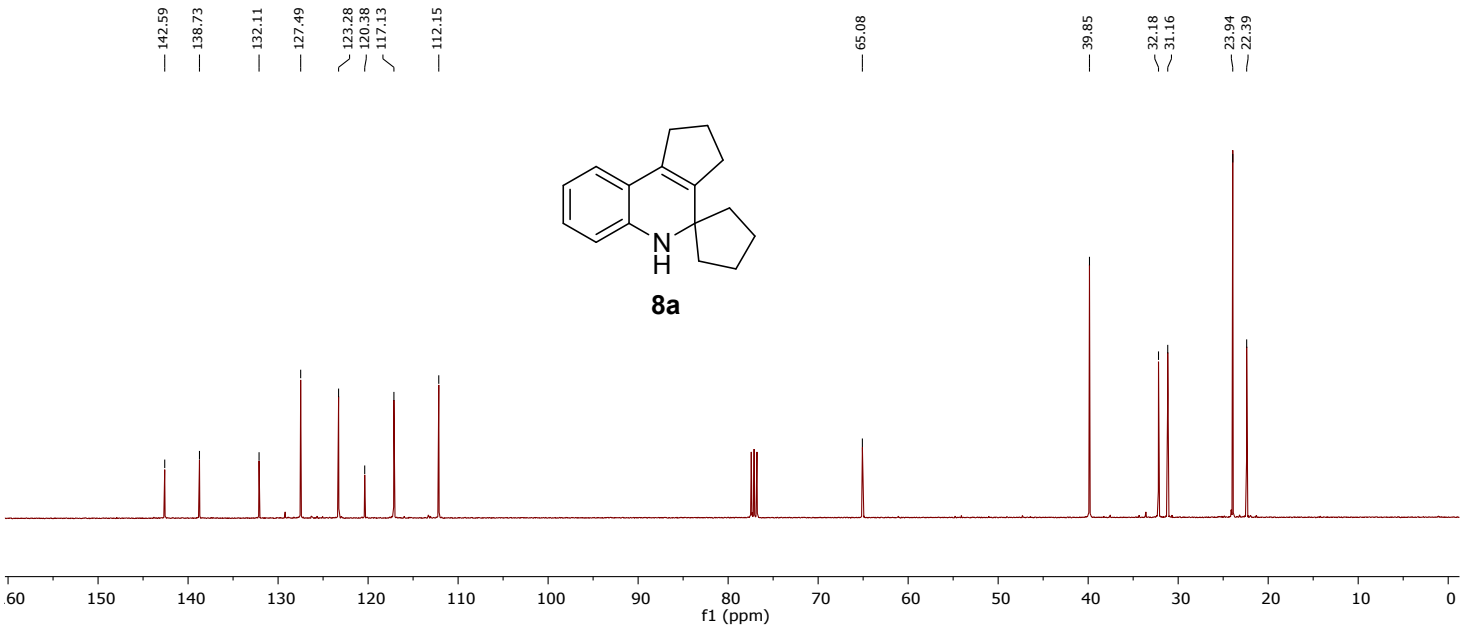
2,4-Diethyl-7-methoxy-2-methyl-1,2-dihydroquinoline (10f) White solid; yield: 60%; mp 60−61 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, *J* = 8.4 Hz, 1H), 6.20 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.06 (d, *J* = 2.4 Hz, 1H), 5.12 (t, *J* = 1.6 Hz, 1H), 3.78 (br s, 1H), 3.77 (s, 3H), 2.39 (q, *J* = 7.2

Hz, 2H), 1.58–1.48 (m, 2H), 1.25 (s, 3H), 1.18 (t, J = 7.6 Hz, 3H), 0.93 (t, J = 7.6 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 160.0, 145.4, 134.5, 124.2, 122.6, 114.3, 101.8, 98.4, 55.0, 54.8, 36.5, 29.5, 24.7, 12.9, 8.5; HRMS (ESI) calcd. for $C_{15}H_{21}NO$ $[M+H]^+$ m/z = 231.1623, found 231.1622.

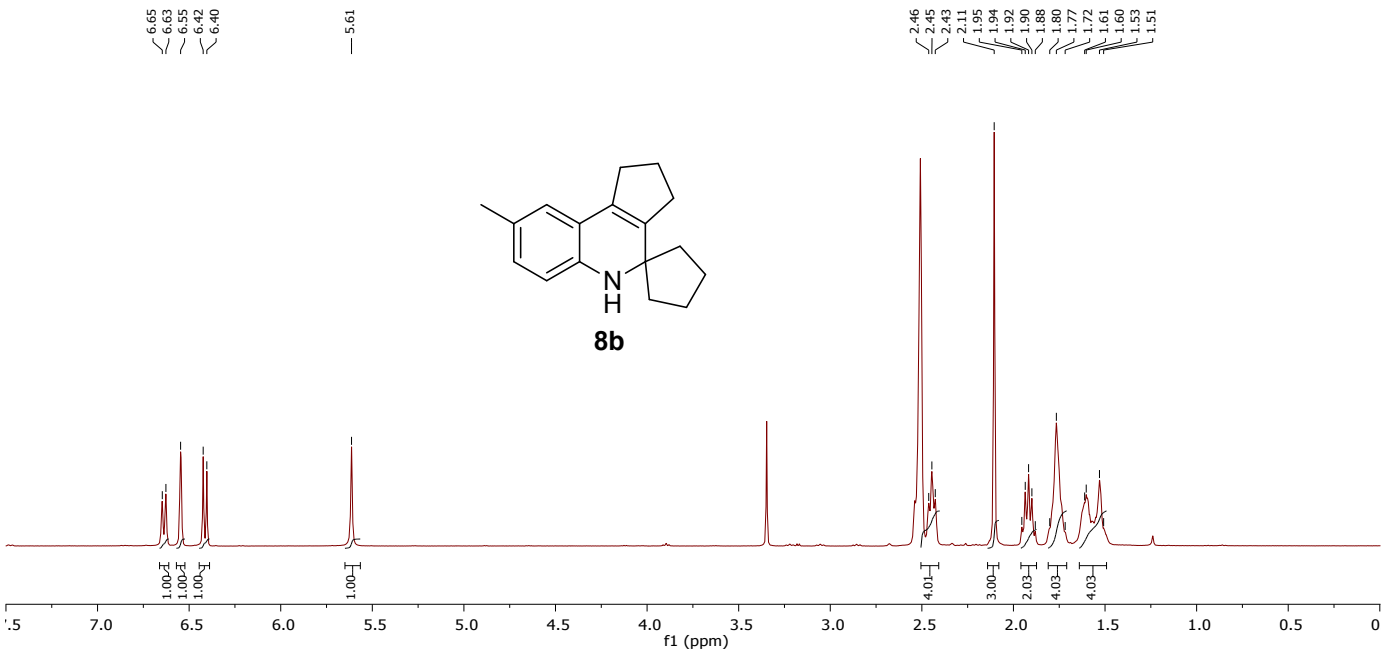
2,4-diethyl-2,7-dimethyl-1,2-dihydroquinoline (10g) Oil; yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (d, J = 7.6 Hz, 1H), 6.46 (d, J = 7.6 Hz, 1H), 6.33 (s, 1H), 5.18 (s, 1H), 3.87 (br s, 1H), 2.41 (q, J = 7.6 Hz, 2H), 2.24 (s, 3H), 1.60 – 1.47 (m, 2H), 1.25 (s, 3H), 1.18 (t, J = 7.6 Hz, 3H), 0.94 (t, J = 7.6 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 143.94, 138.11, 134.75, 124.13, 123.16, 118.12, 117.56, 113.46, 54.61, 36.50, 29.47, 24.67, 21.37, 13.00, 8.61; HRMS (ESI) calcd. for $C_{14}H_{19}N$ $[M+H]^+$ m/z = 215.1674, found 215.1674.

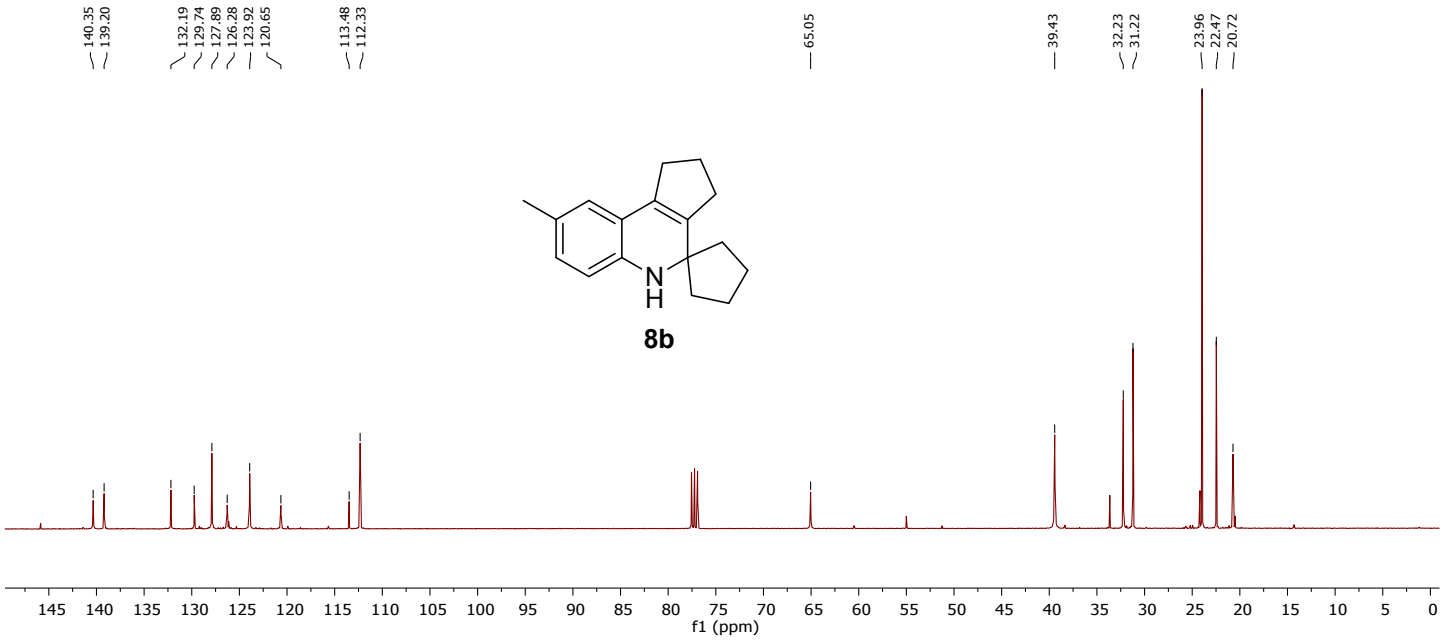
^{1}H and ^{13}C spectra of compound 8a



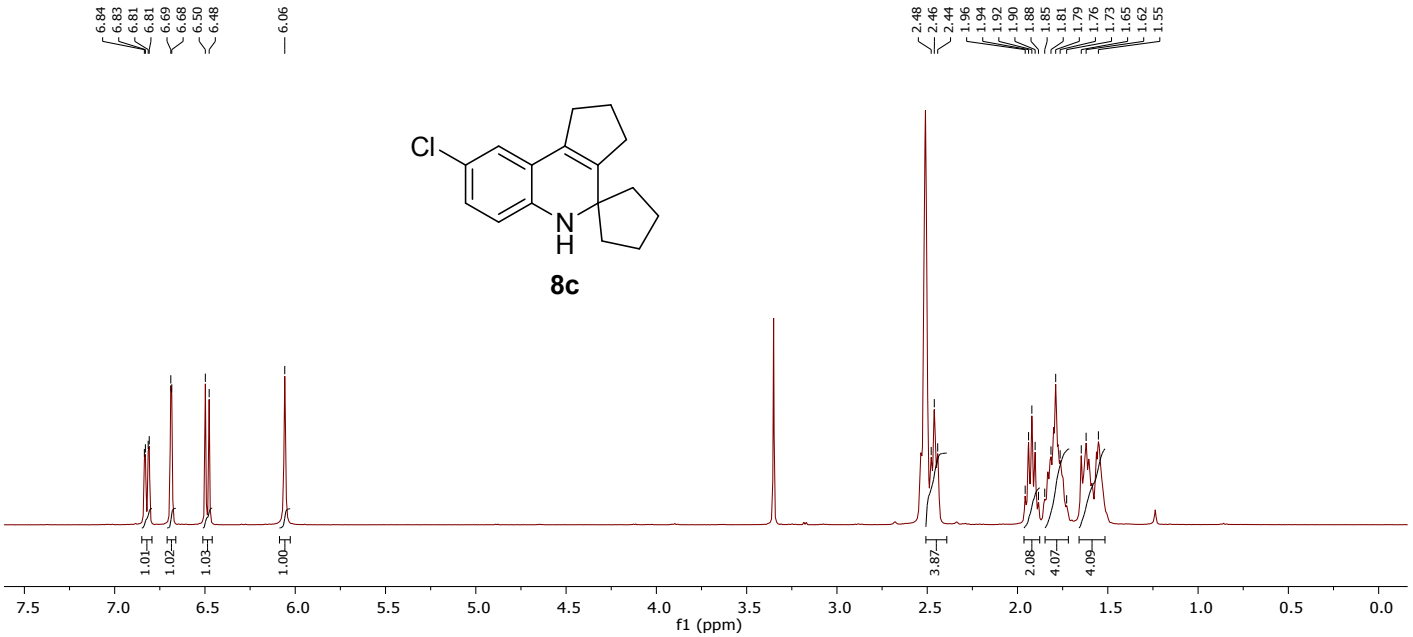


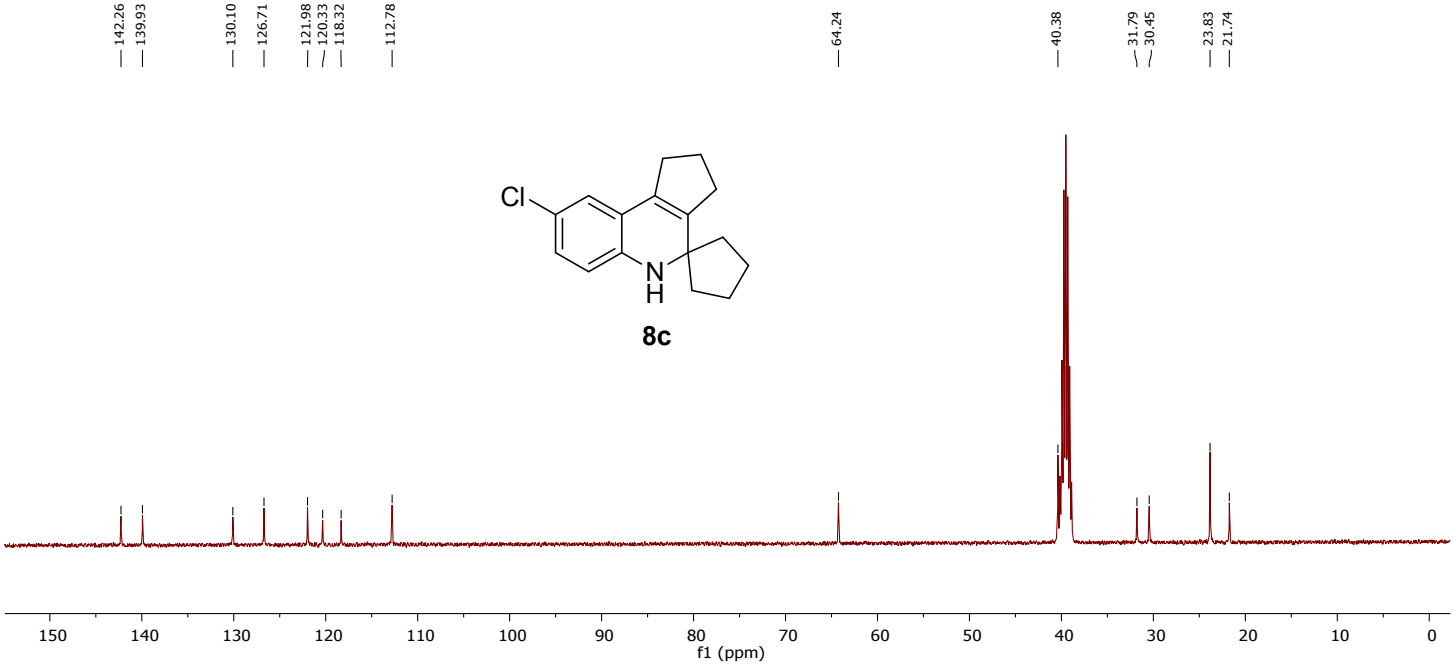
^{1}H and ^{13}C spectra of compound 8b



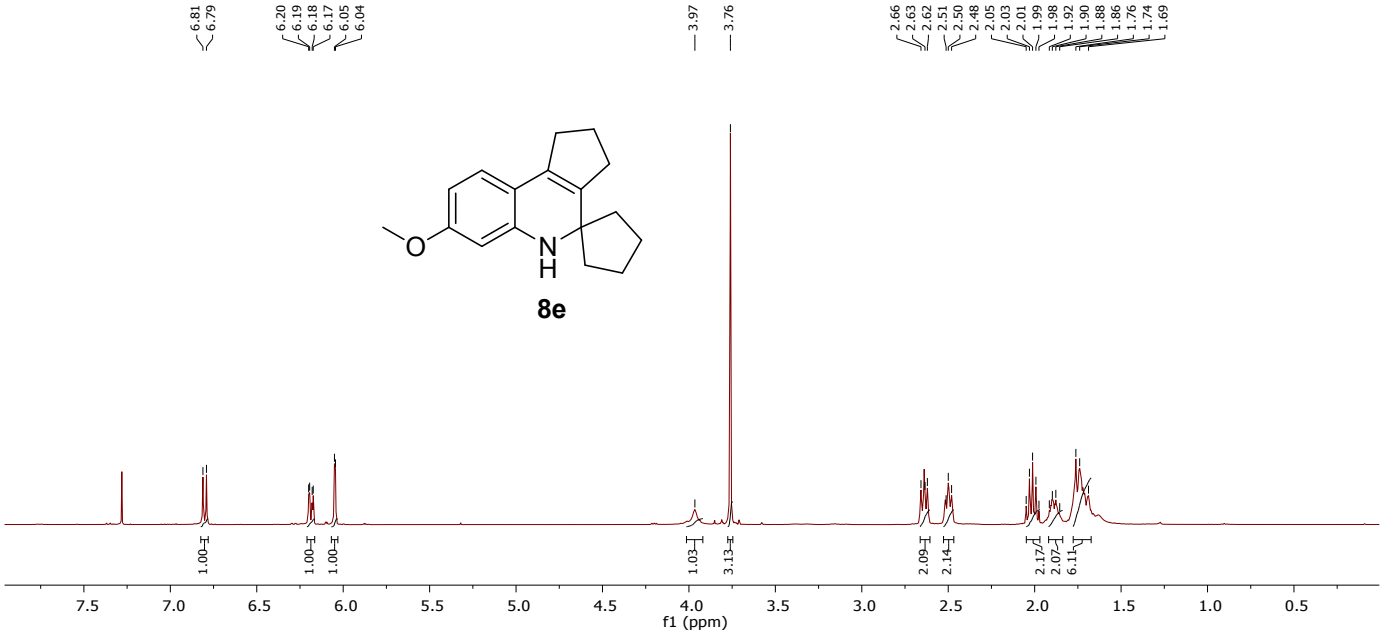


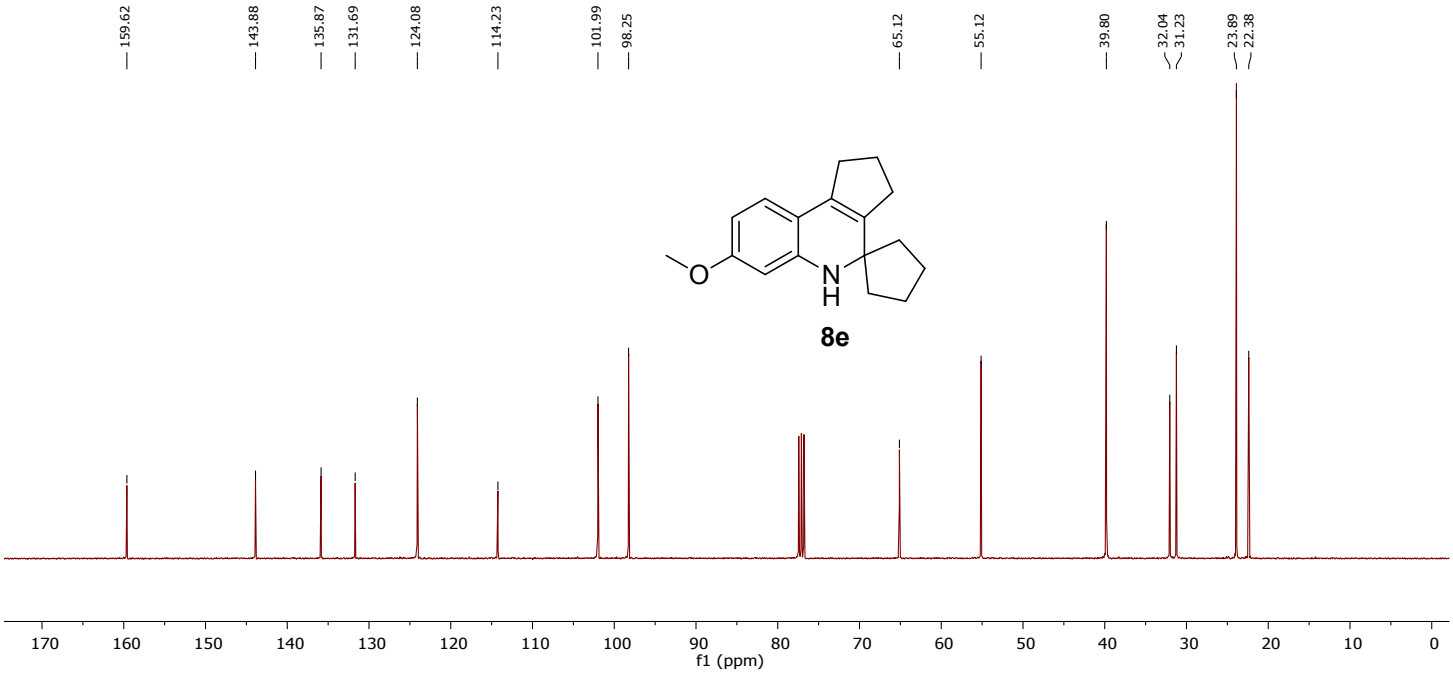
^{1}H and ^{13}C spectra of compound 8c



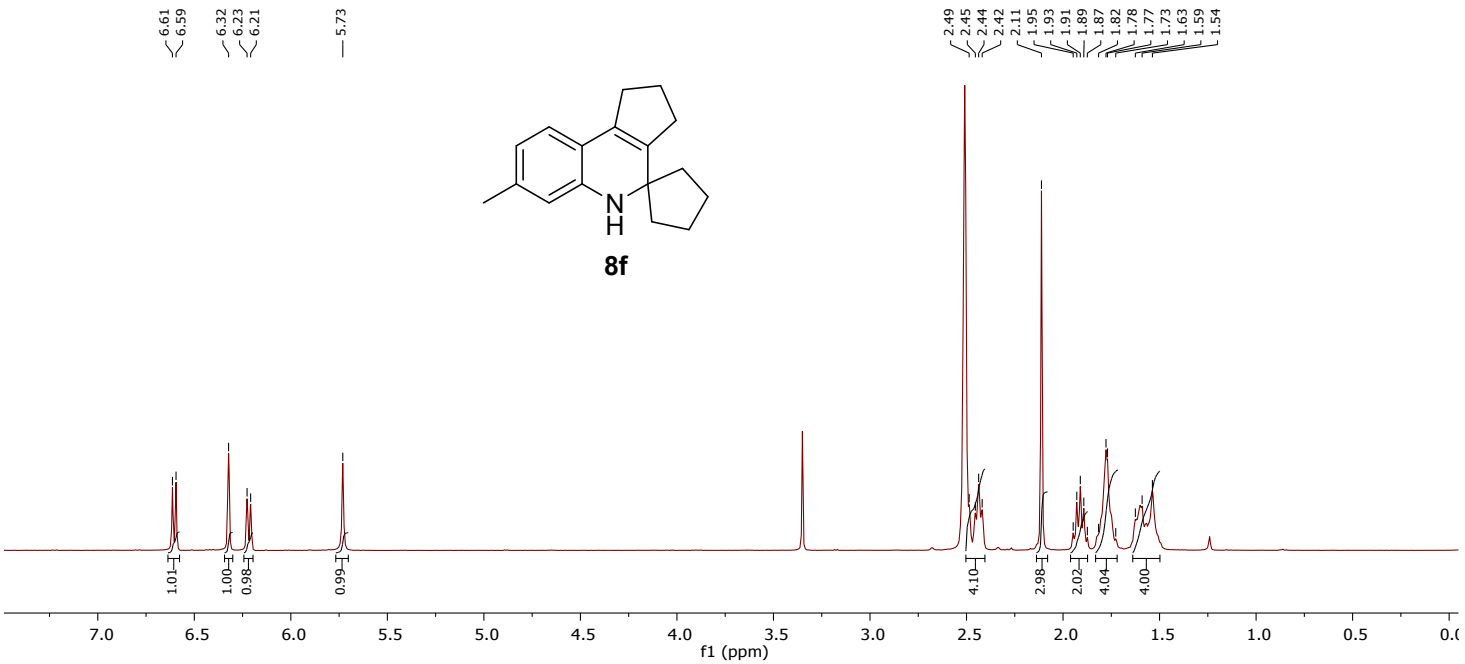


^{1}H and ^{13}C spectra of compound 8e

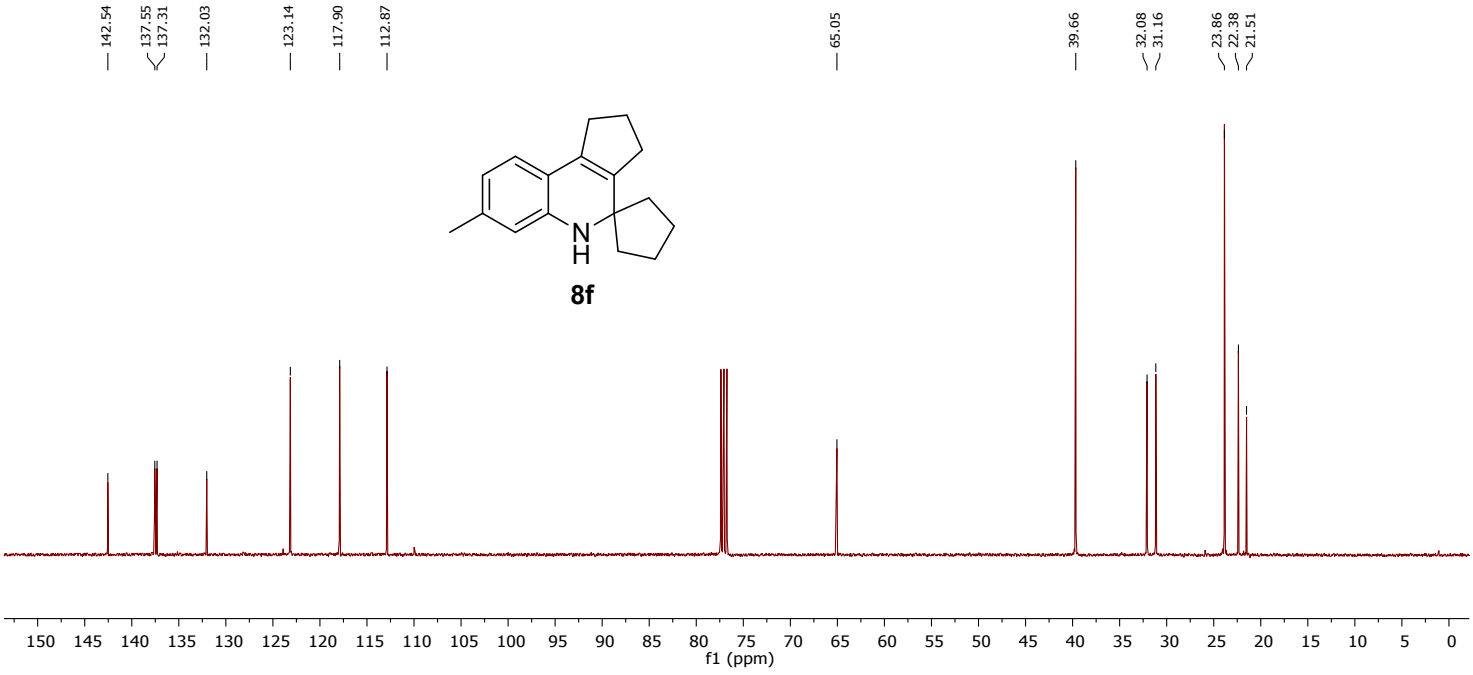




^{1}H and ^{13}C spectra of compound 8f

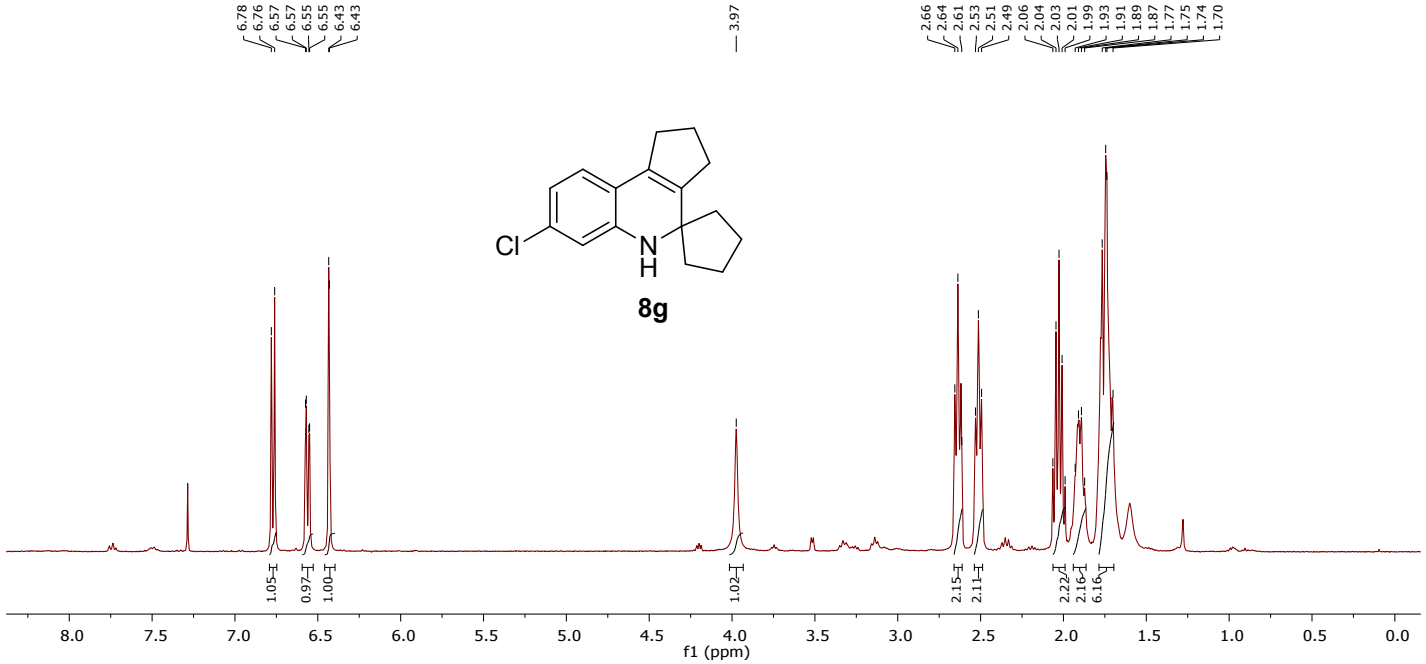


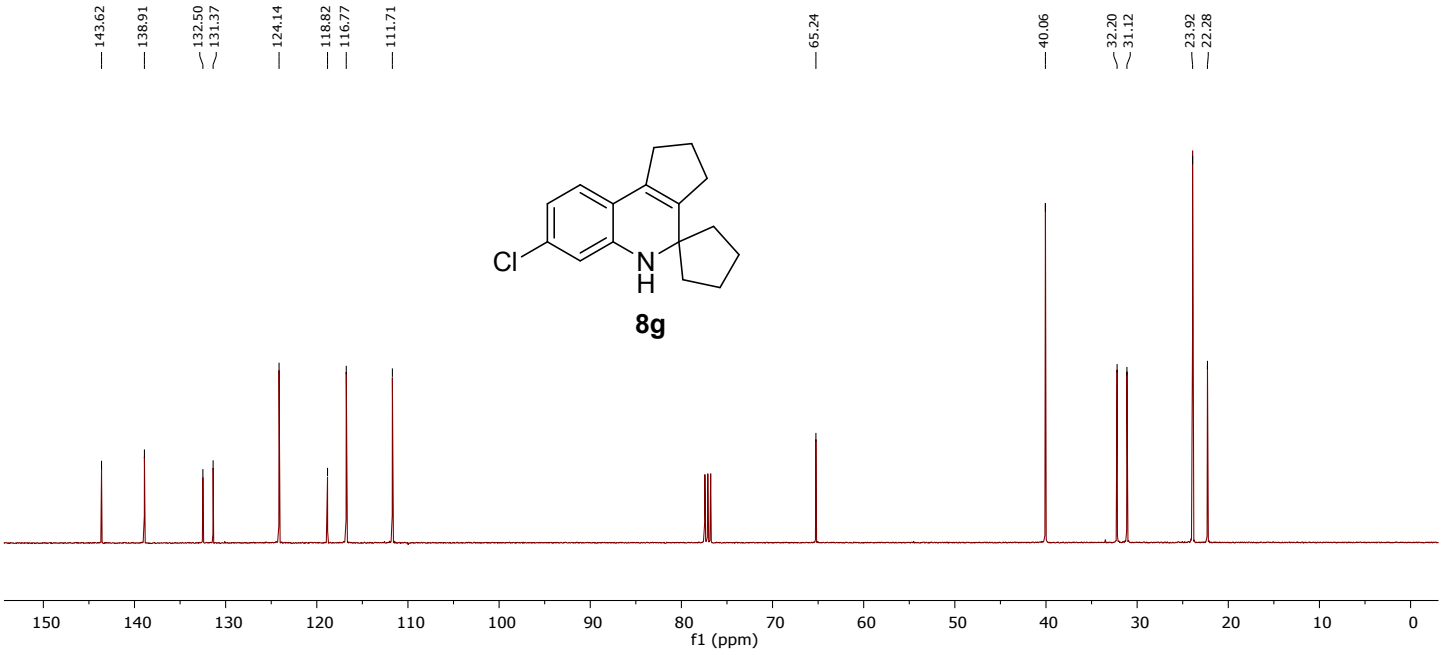
8f



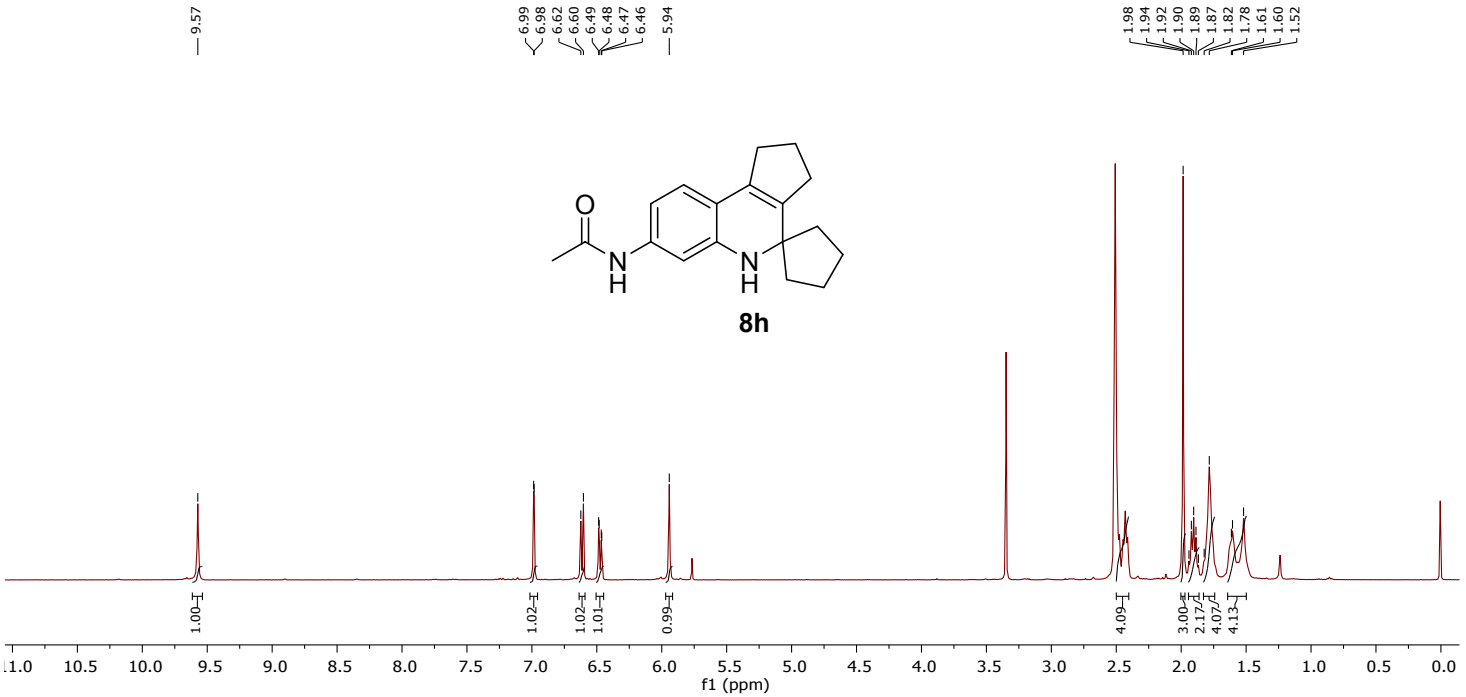
8f

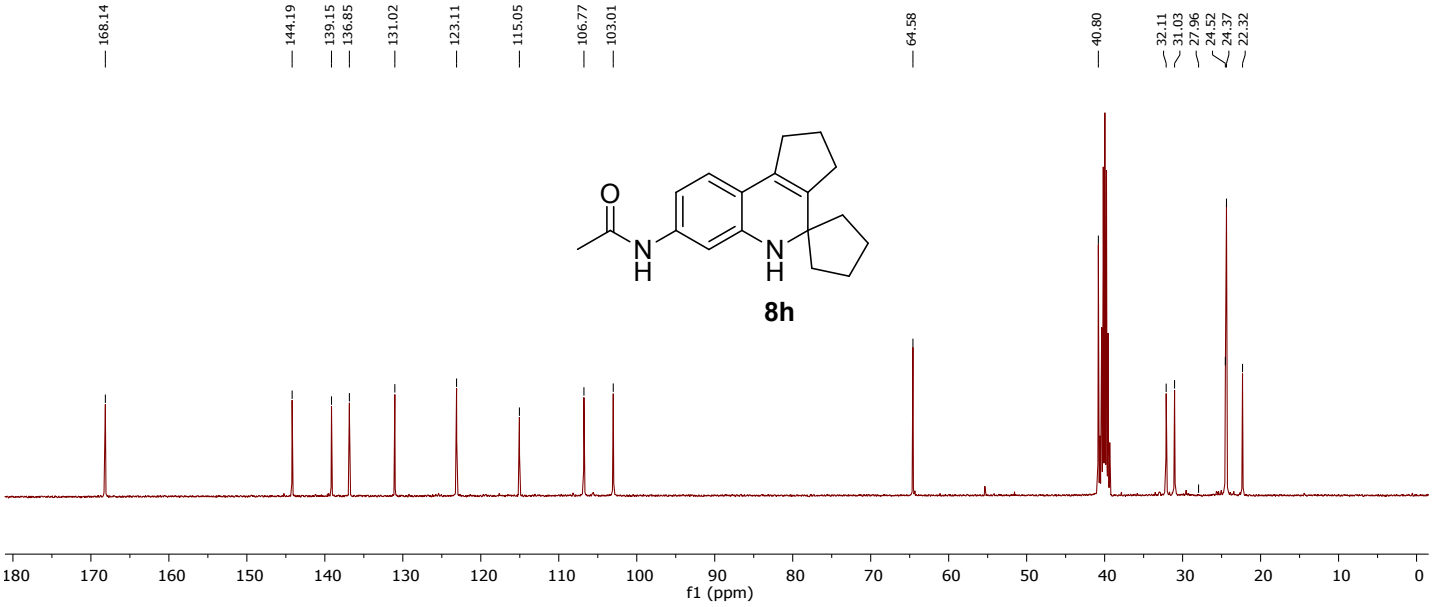
^{1}H and ^{13}C spectra of compound 8g



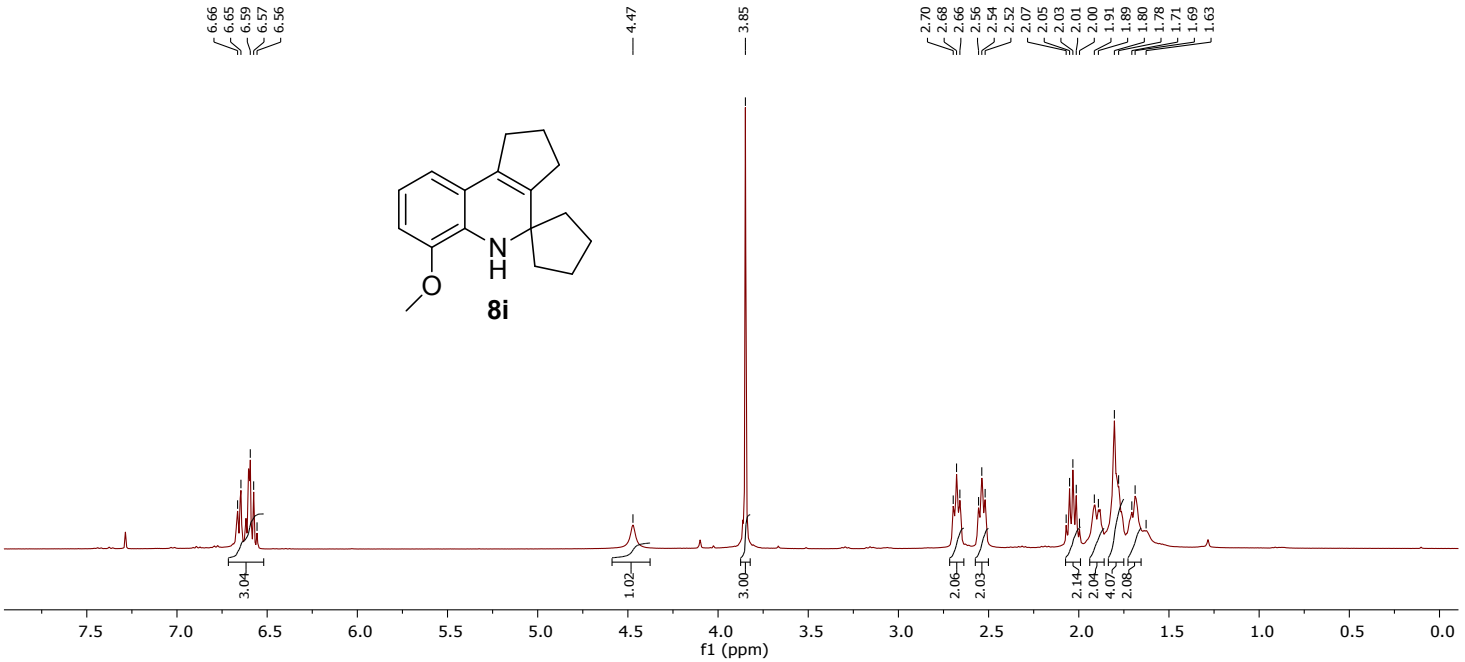


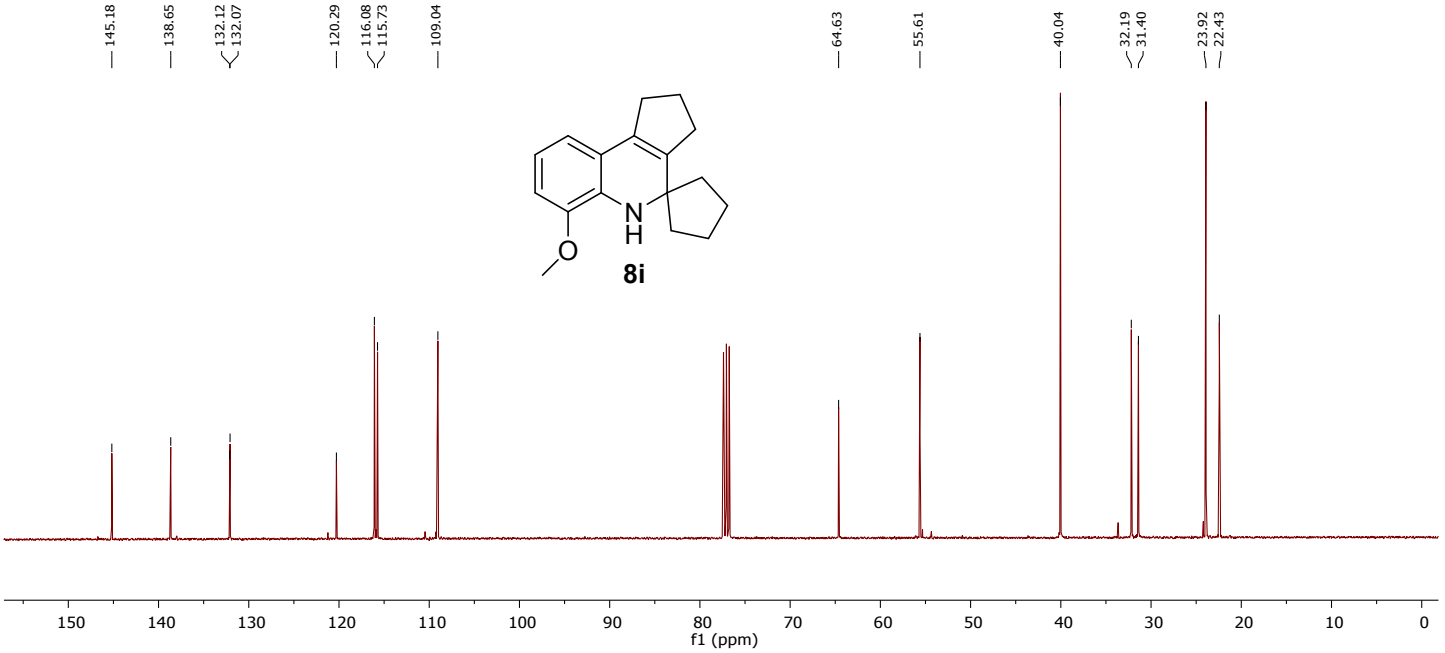
^{1}H and ^{13}C spectra of compound 8h


9.57
6.99
6.98
6.62
6.60
6.49
6.48
6.47
6.46
5.94
1.98
1.94
1.92
1.90
1.89
1.87
1.82
1.78
1.61
1.60
1.52
O
N
H
N
H
8h
1.00
1.02
1.02
1.01
0.99
4.09
3.00
2.17
4.07
4.13
f1 (ppm)

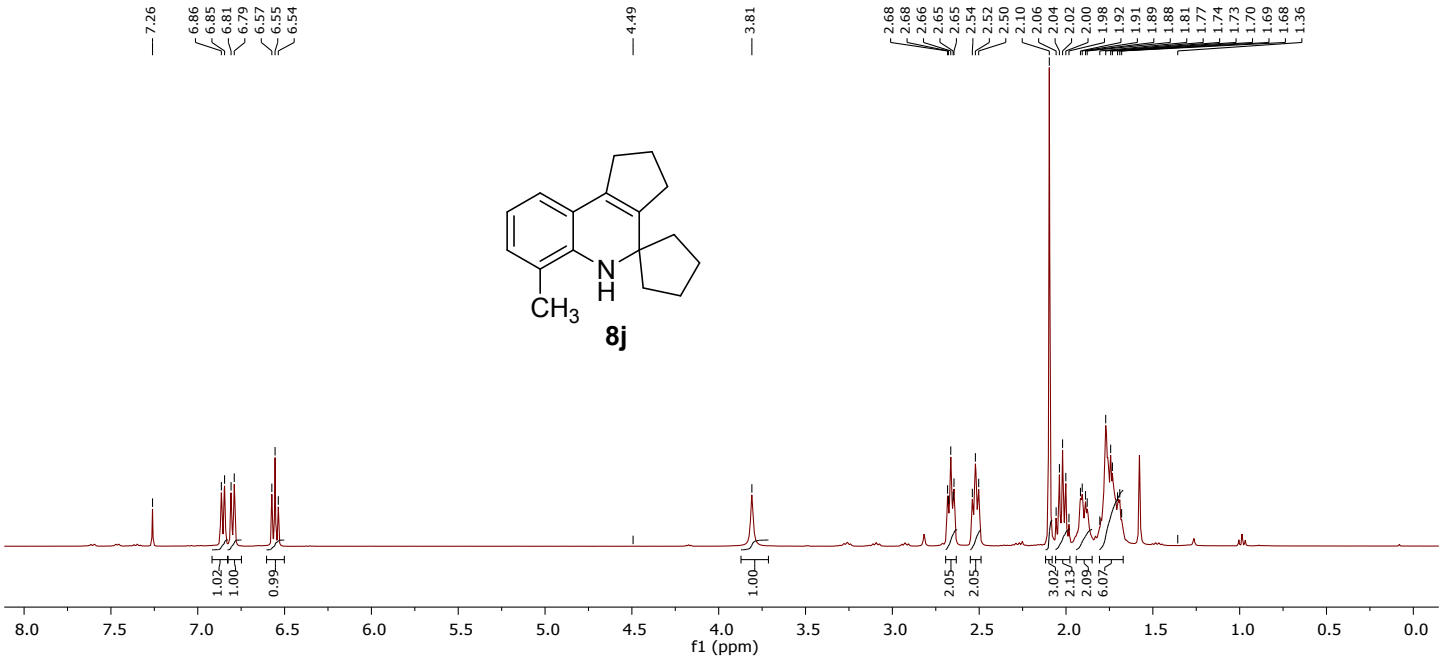

168.14
144.19
139.15
136.85
131.02
123.11
115.05
106.77
103.01
64.58
40.80
32.11
31.03
27.96
24.52
24.37
22.32
O
N
H
N
H
8h
f1 (ppm)

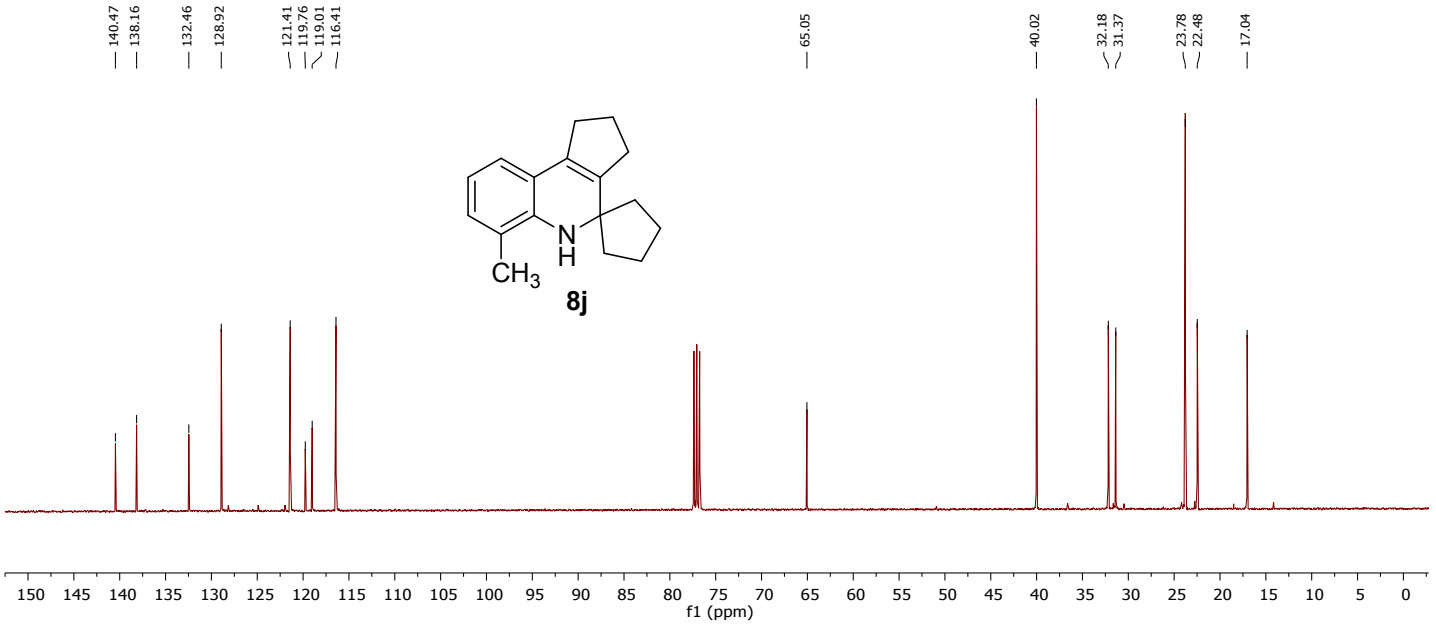
^{1}H and ^{13}C spectra of compound 8i



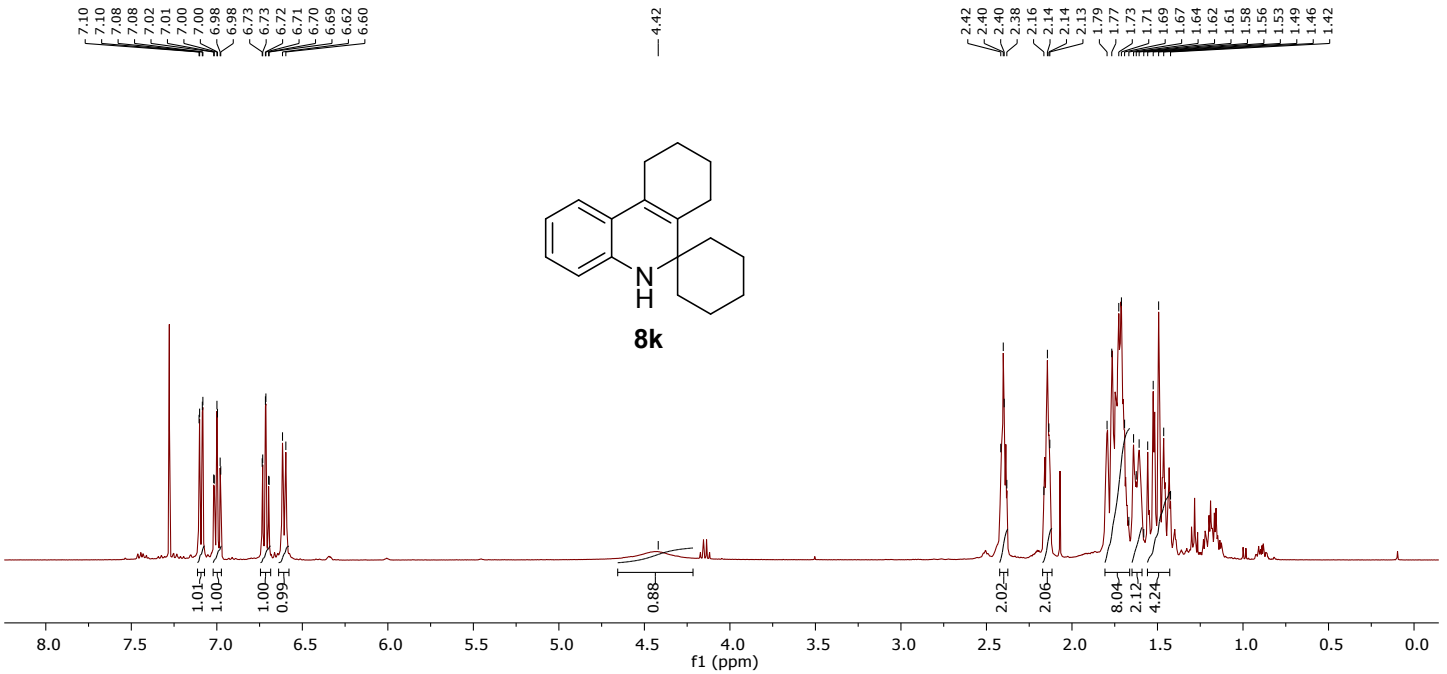


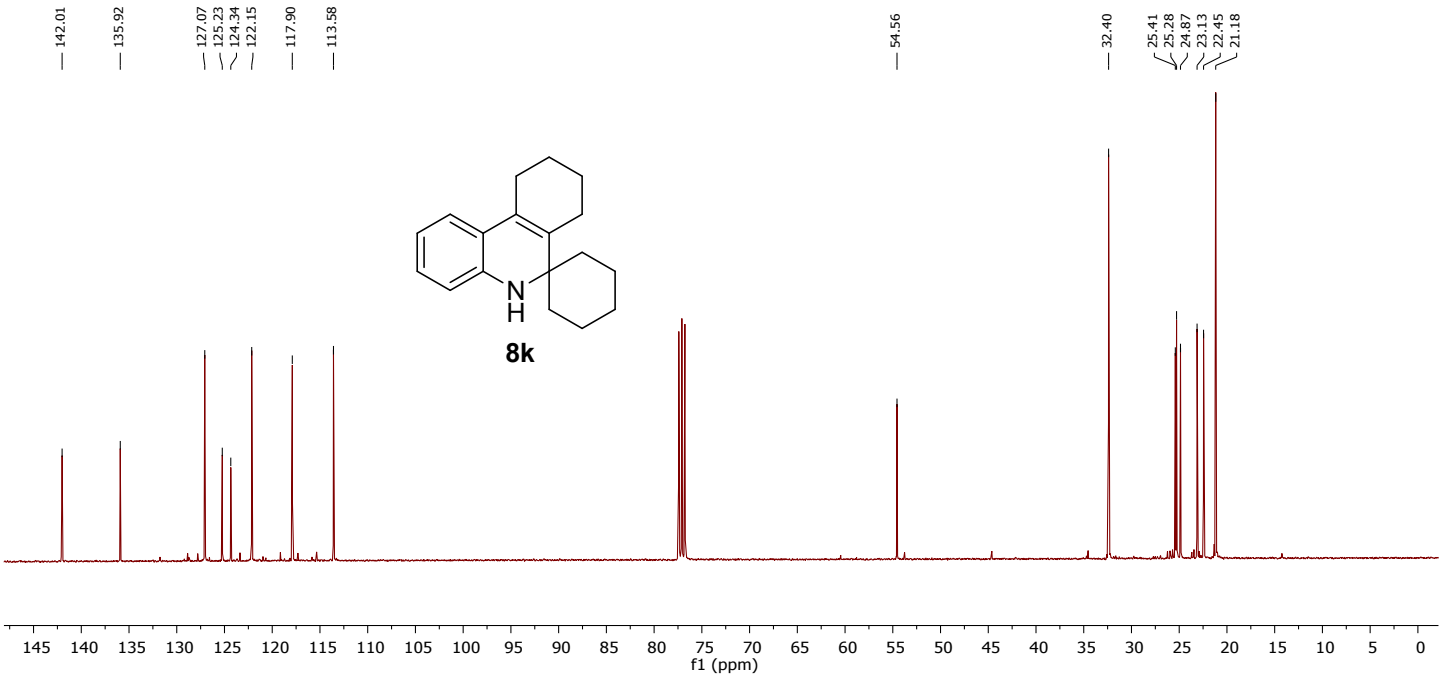
^{1}H and ^{13}C spectra of compound 8j



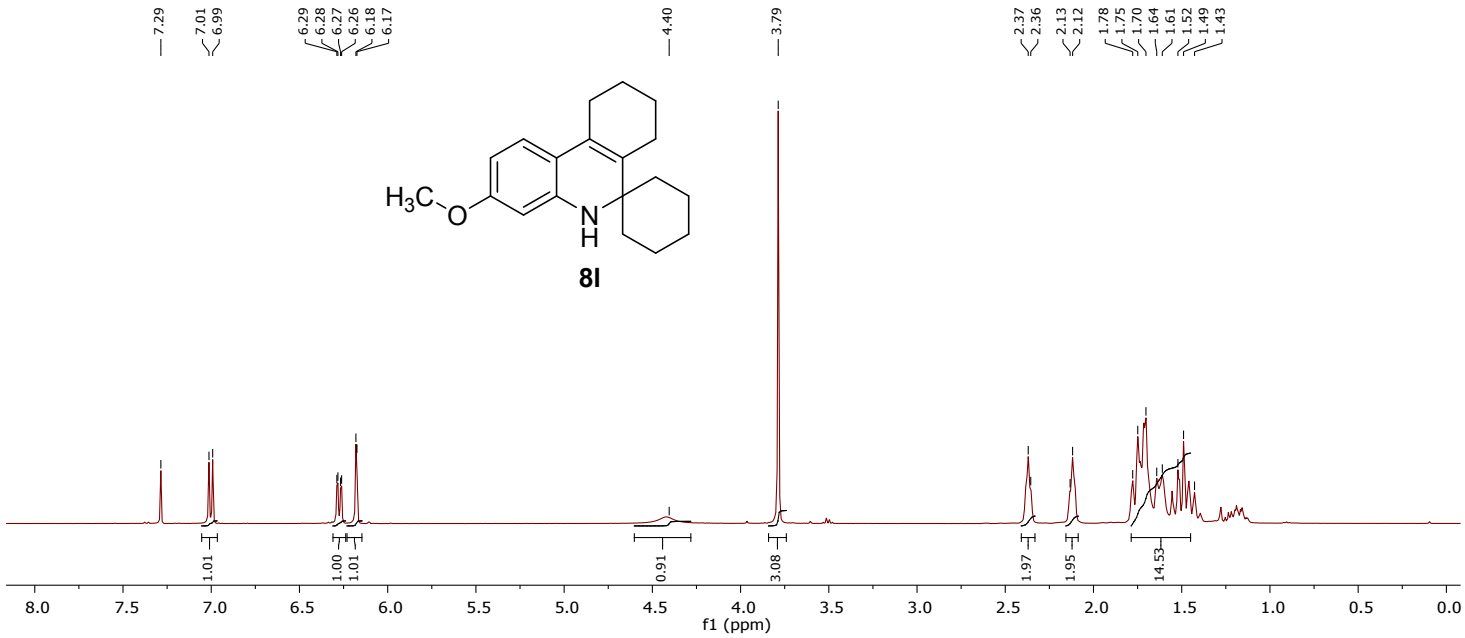


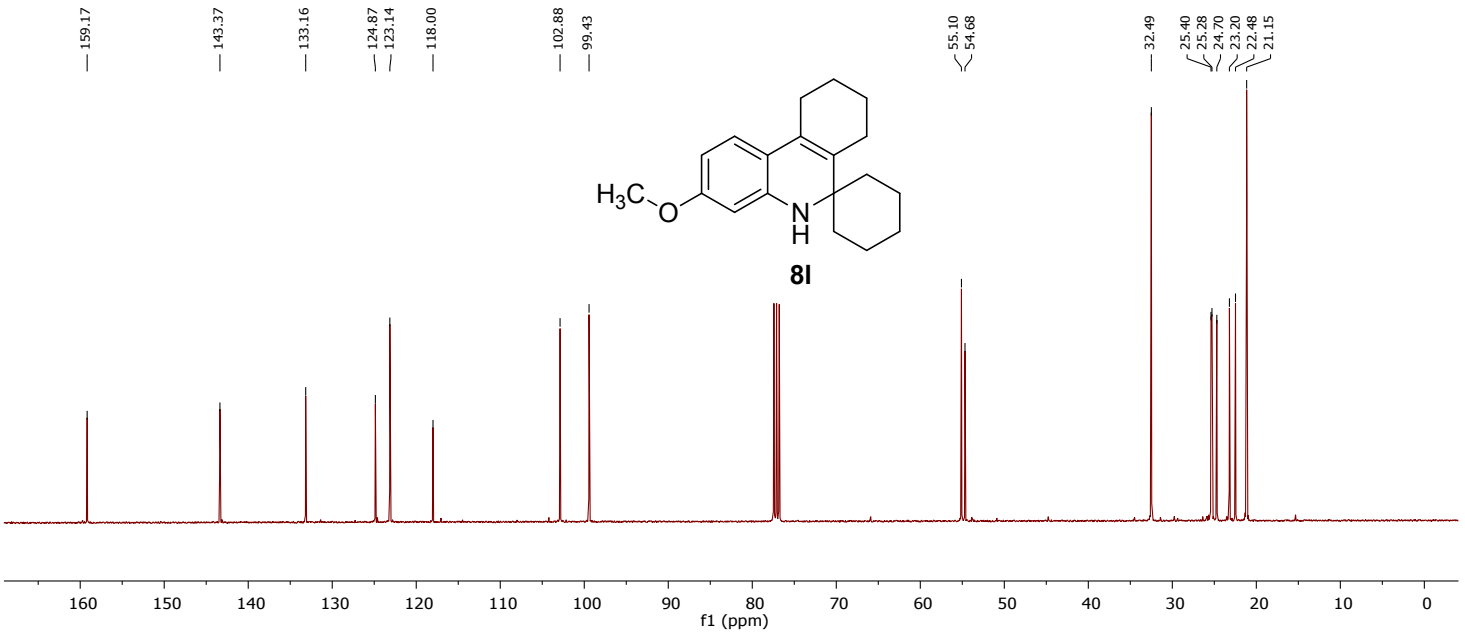
^{1}H and ^{13}C spectra of compound 8k



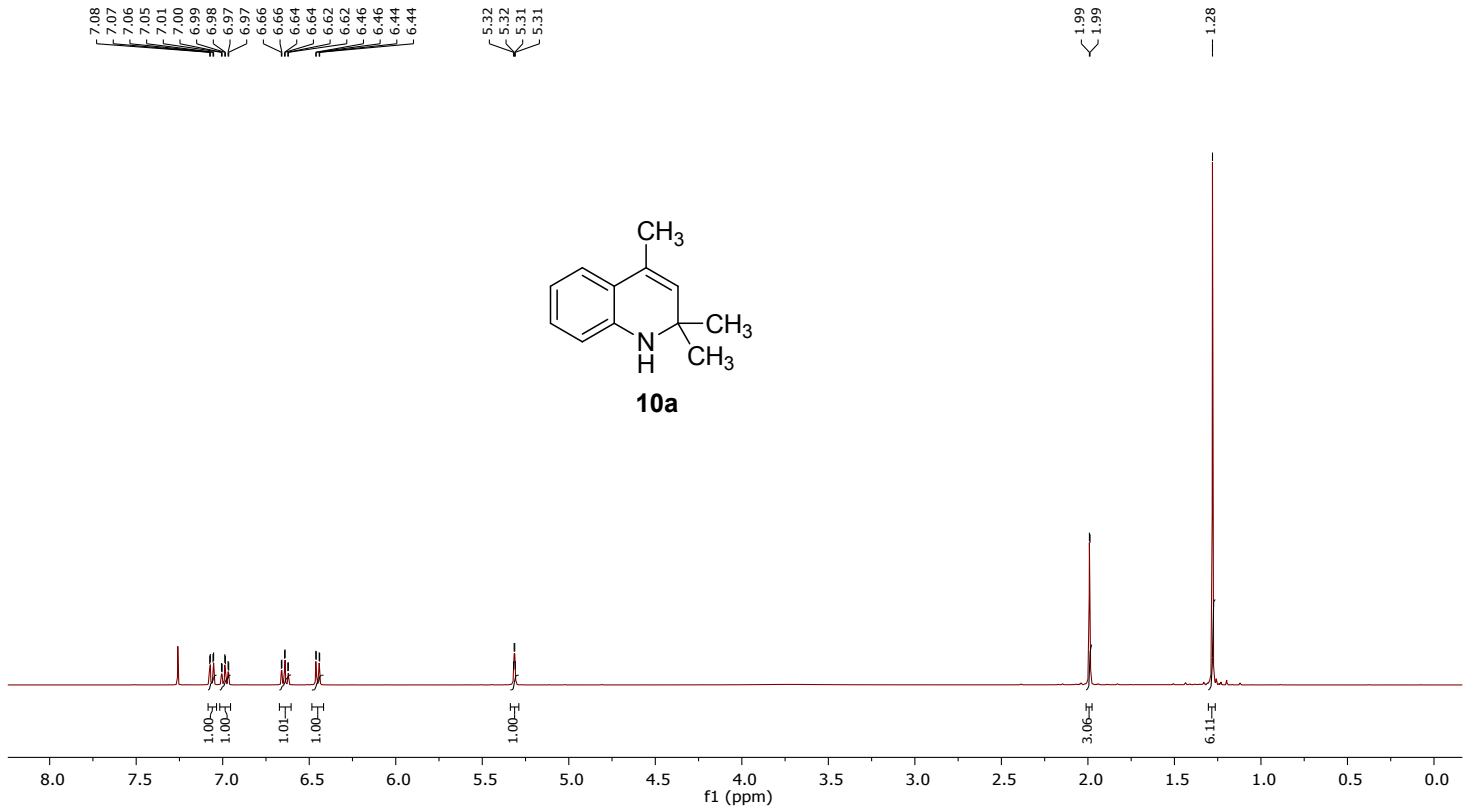


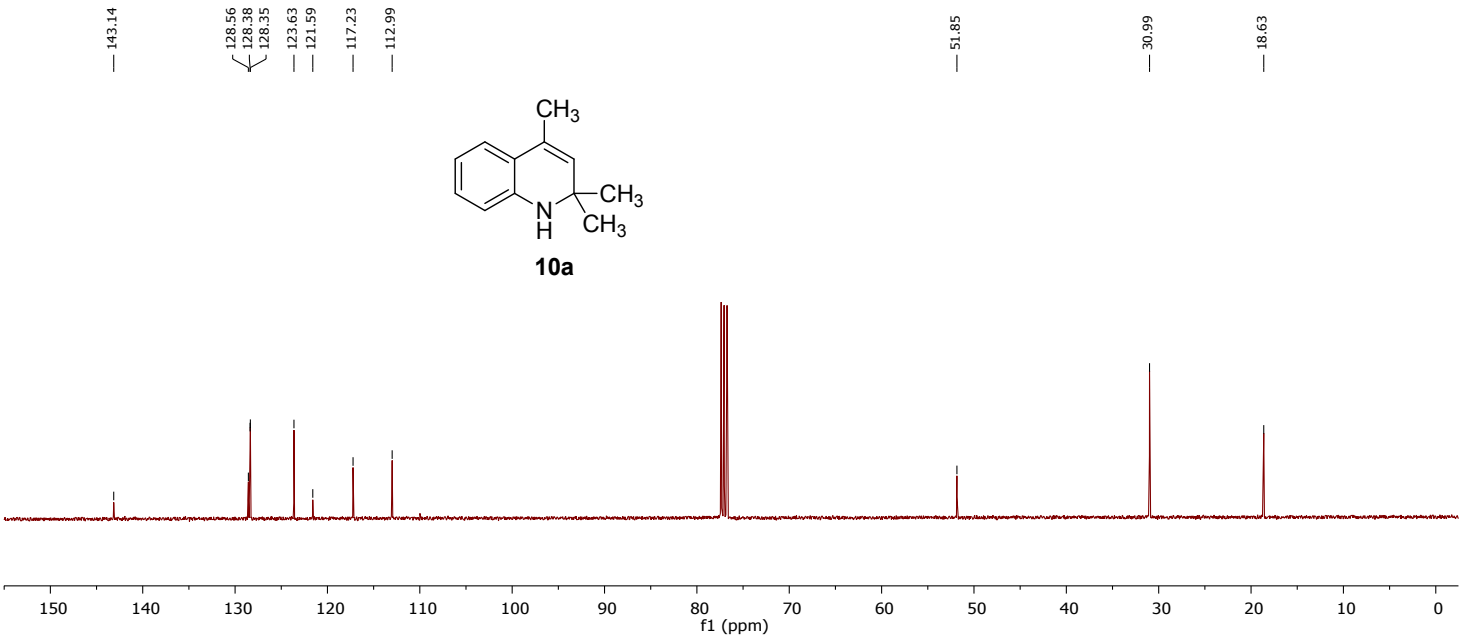
^{1}H and ^{13}C spectra of compound 8l



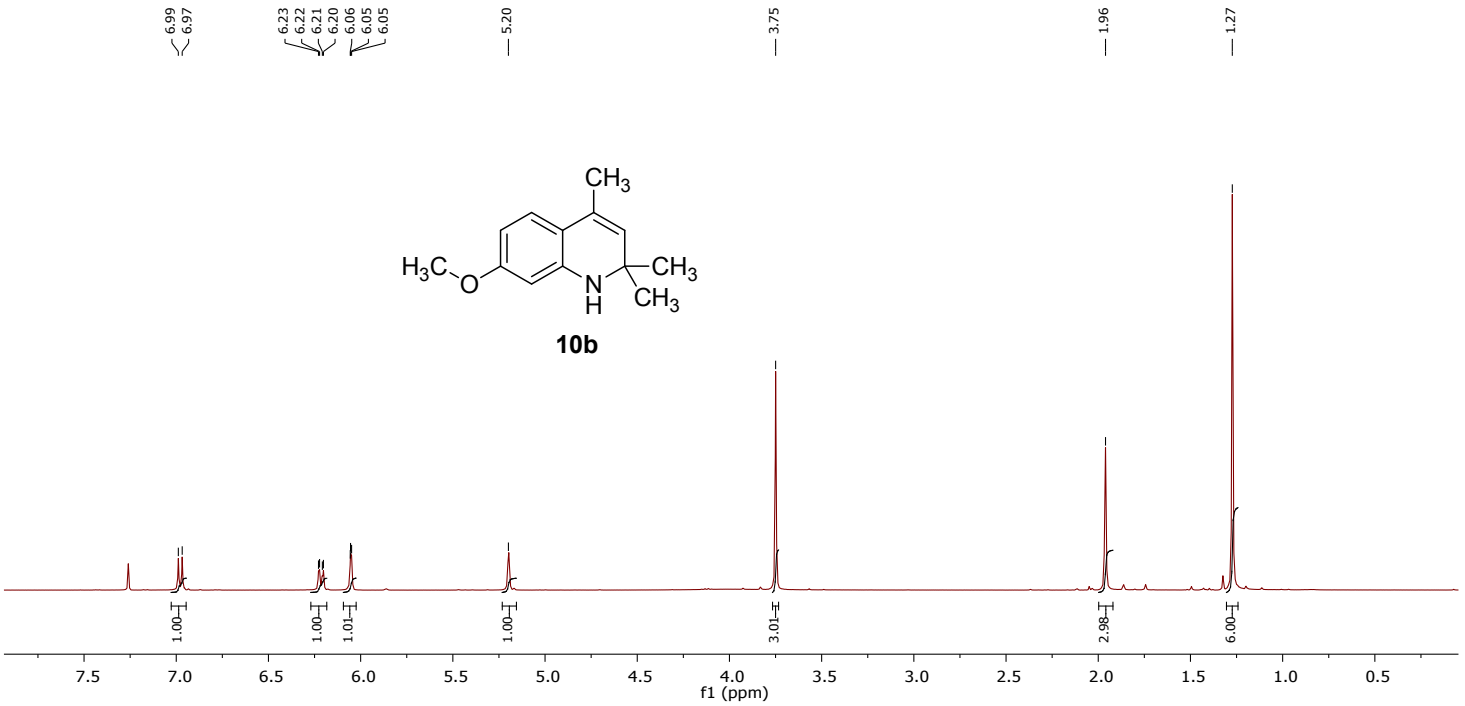


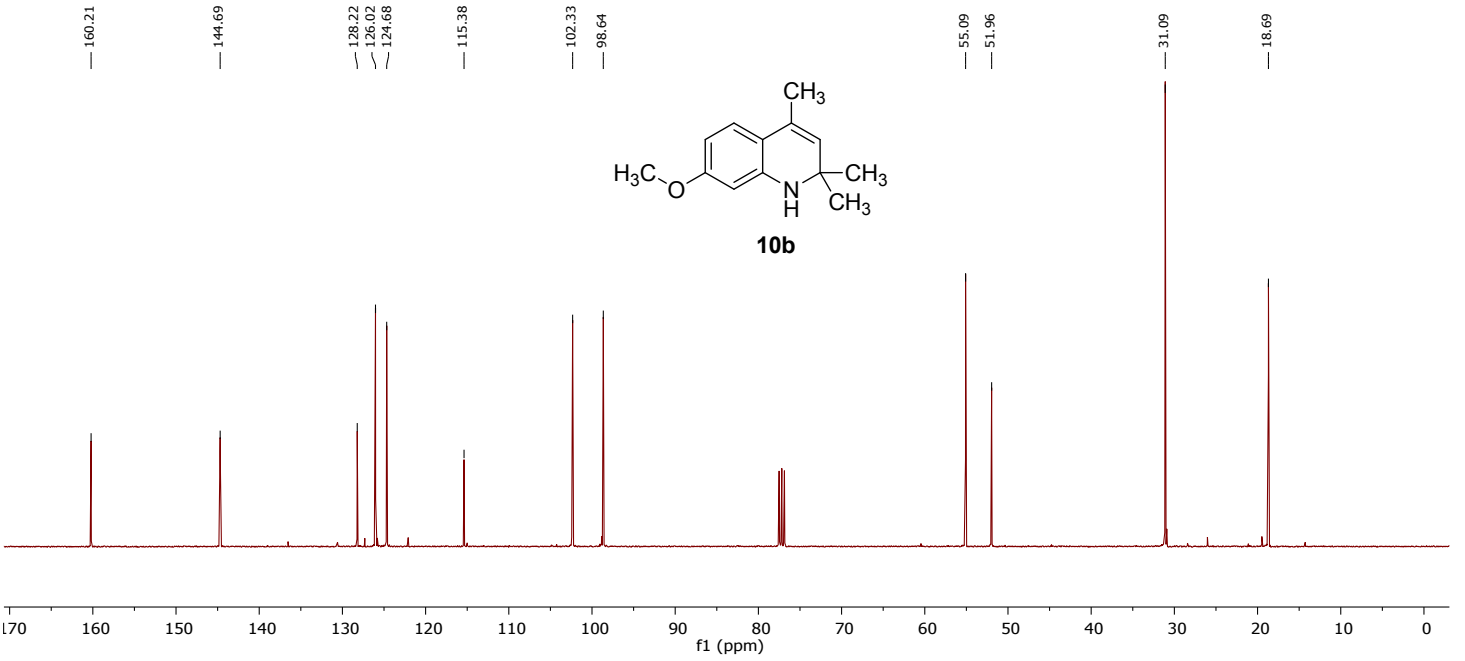
^{1}H and ^{13}C spectra of compound 10a



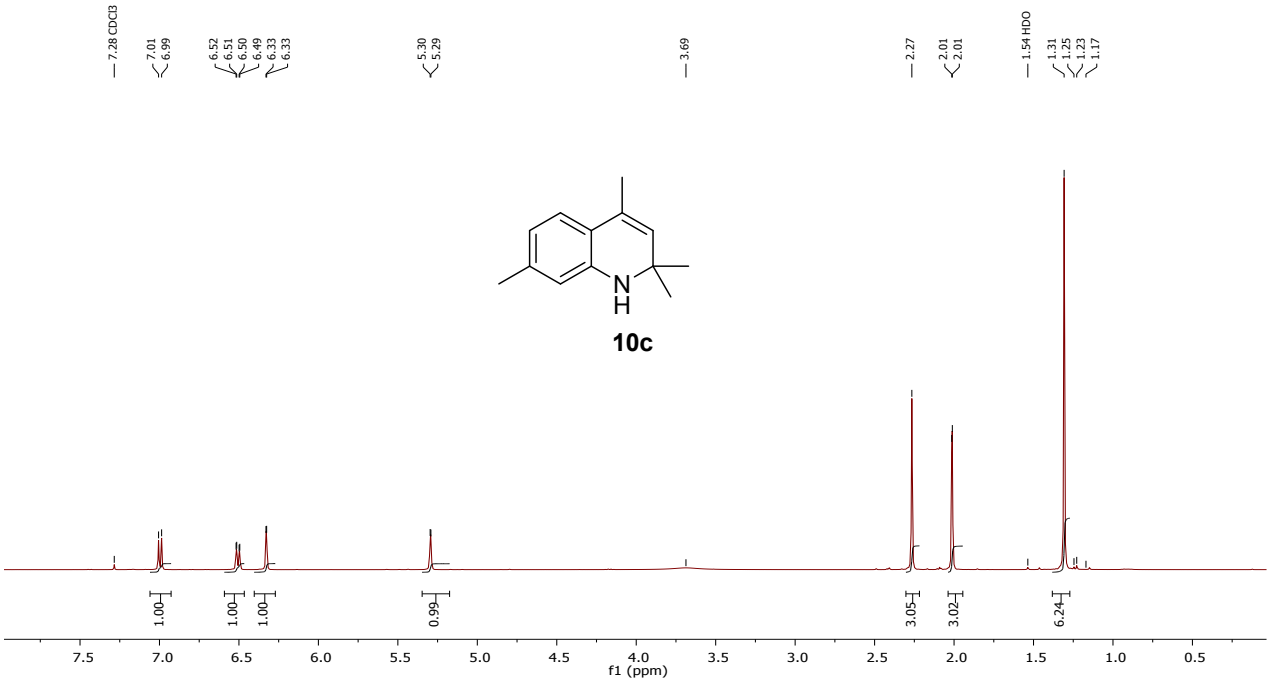


^{1}H and ^{13}C spectra of compound 10b

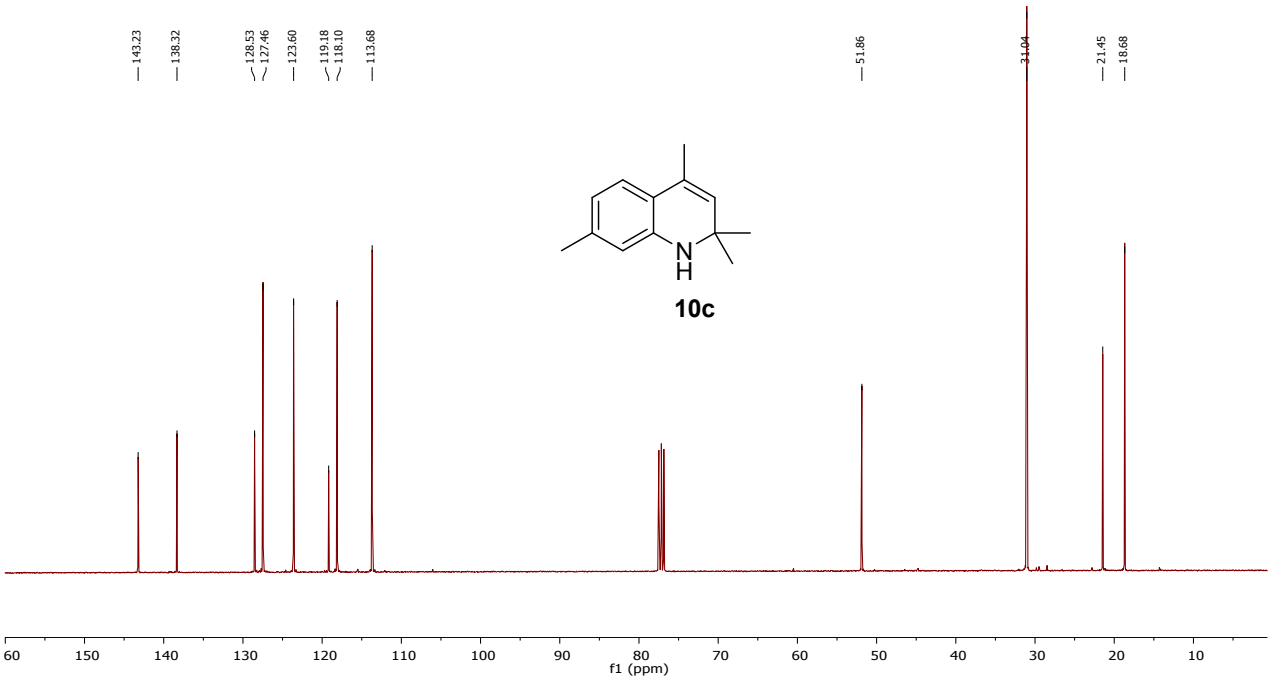




^{1}H and ^{13}C spectra of compound 10c

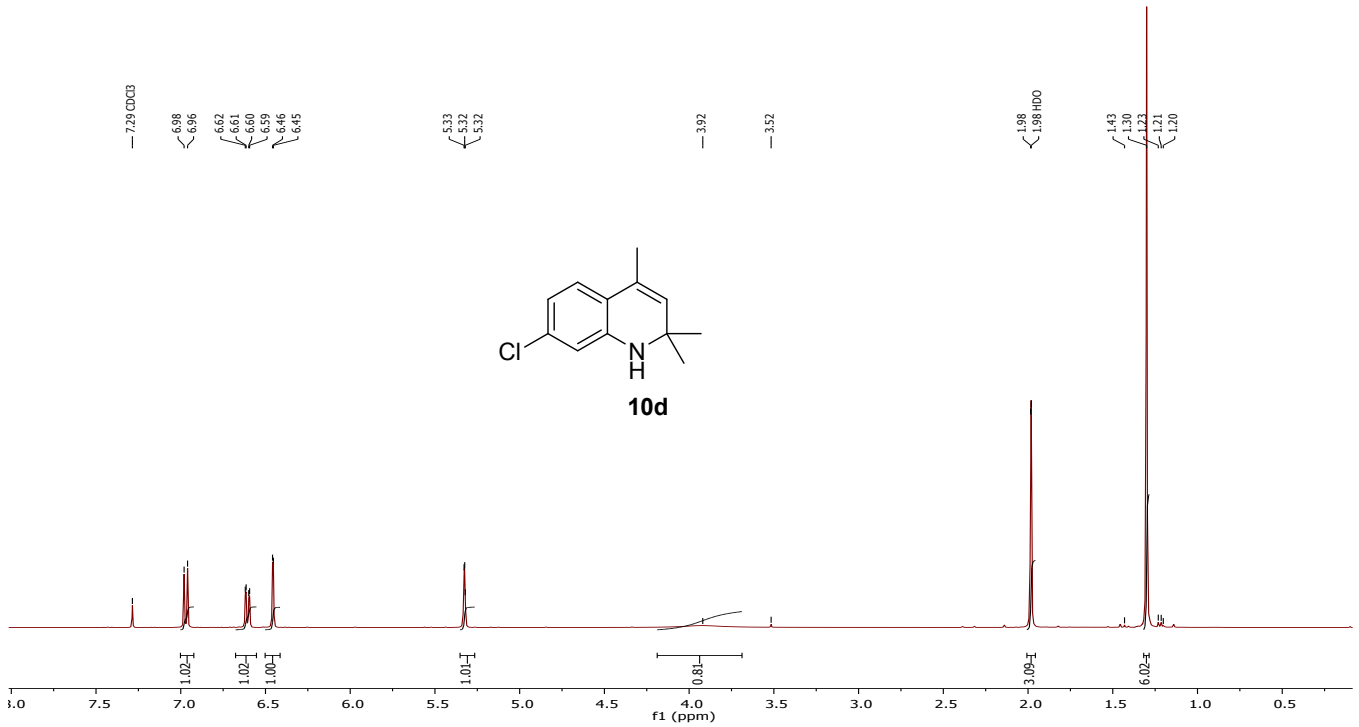

7.28 CDCl3
7.01
6.99
6.52
6.51
6.50
6.49
6.33
6.33
5.30
5.29
3.69
2.27
2.01
2.01
1.54 HDO
1.31
1.25
1.23
1.17
1.00
1.00
1.00
0.99
3.05
3.02
6.24
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

10c

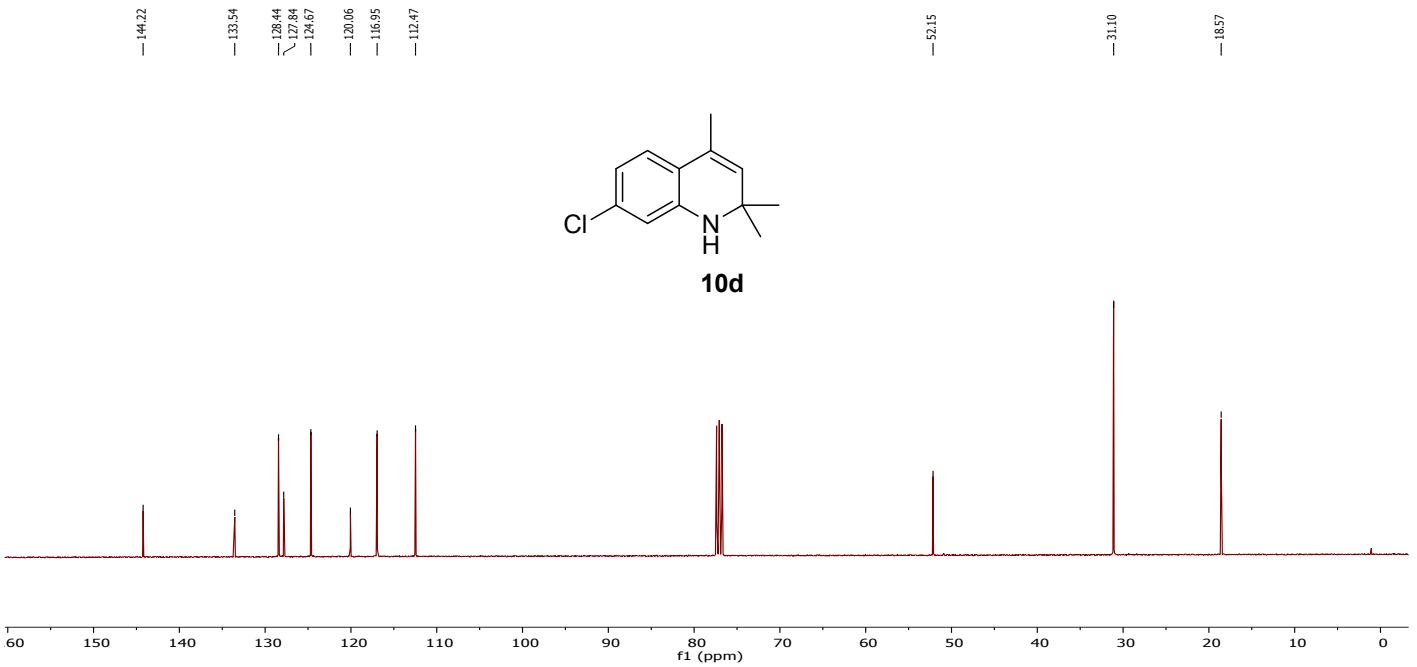

143.23
138.32
128.53
127.46
123.60
119.18
118.10
113.68
51.86
31.04
21.45
18.68
60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

10c

^{1}H and ^{13}C spectra of compound 10d

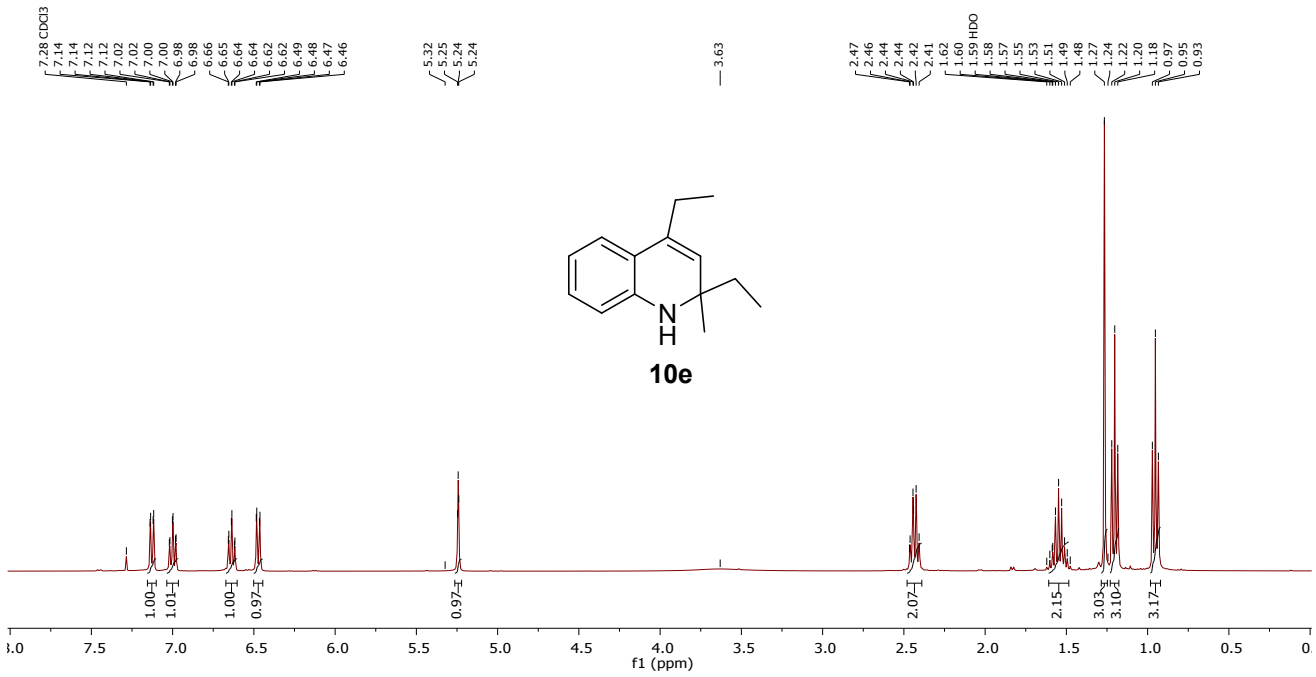


10d

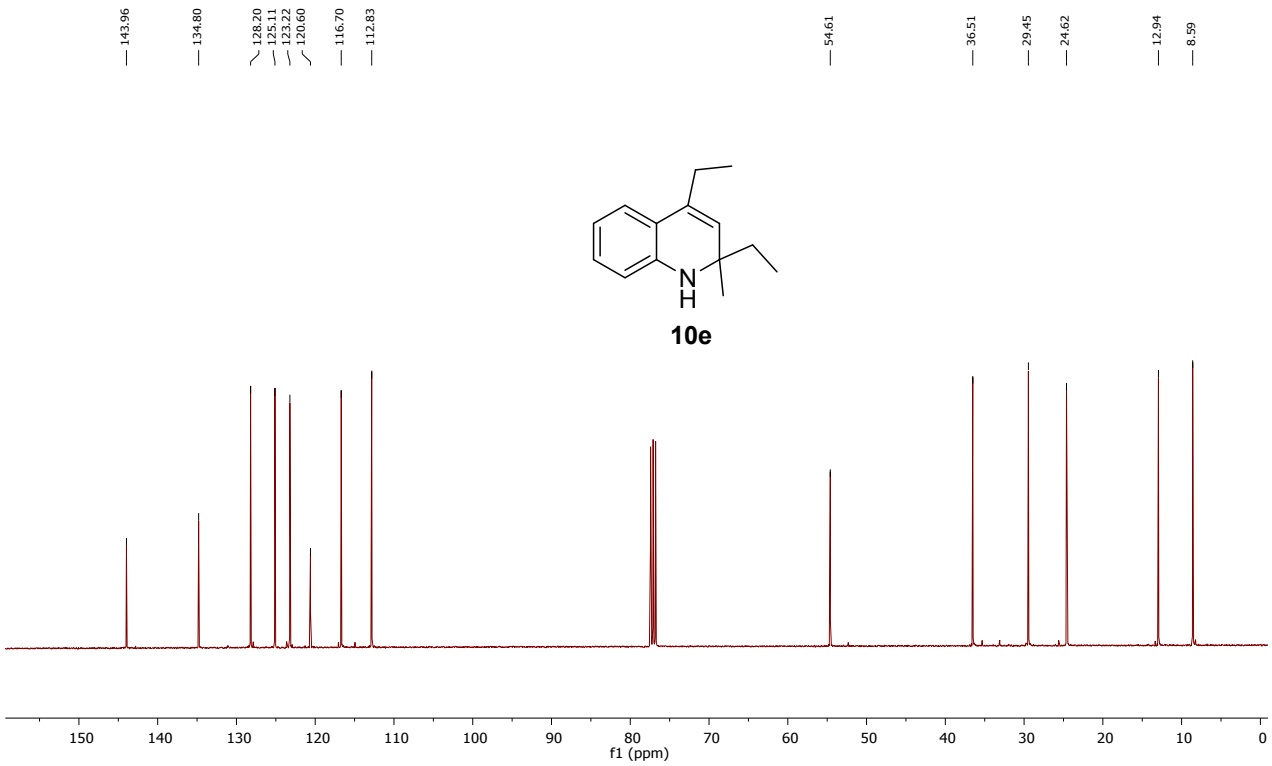


10d

^{1}H and ^{13}C spectra of compound 10e

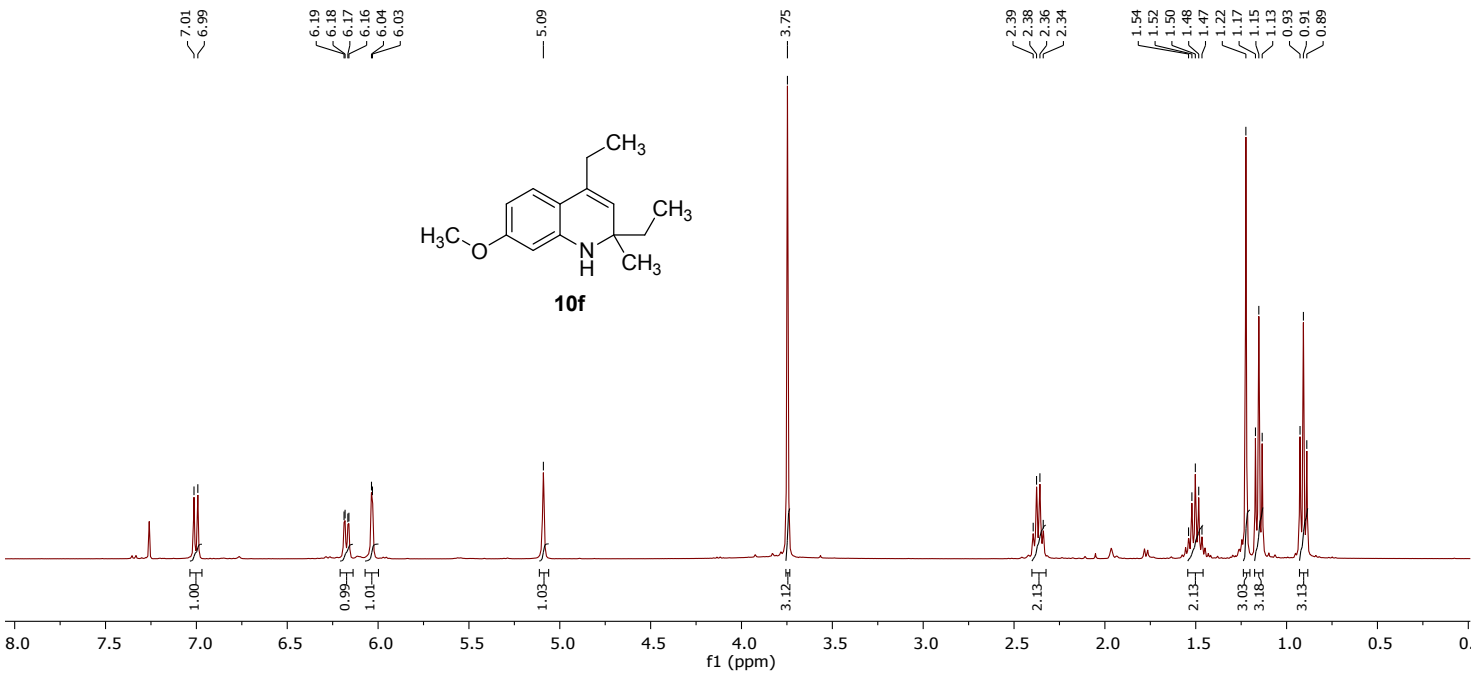


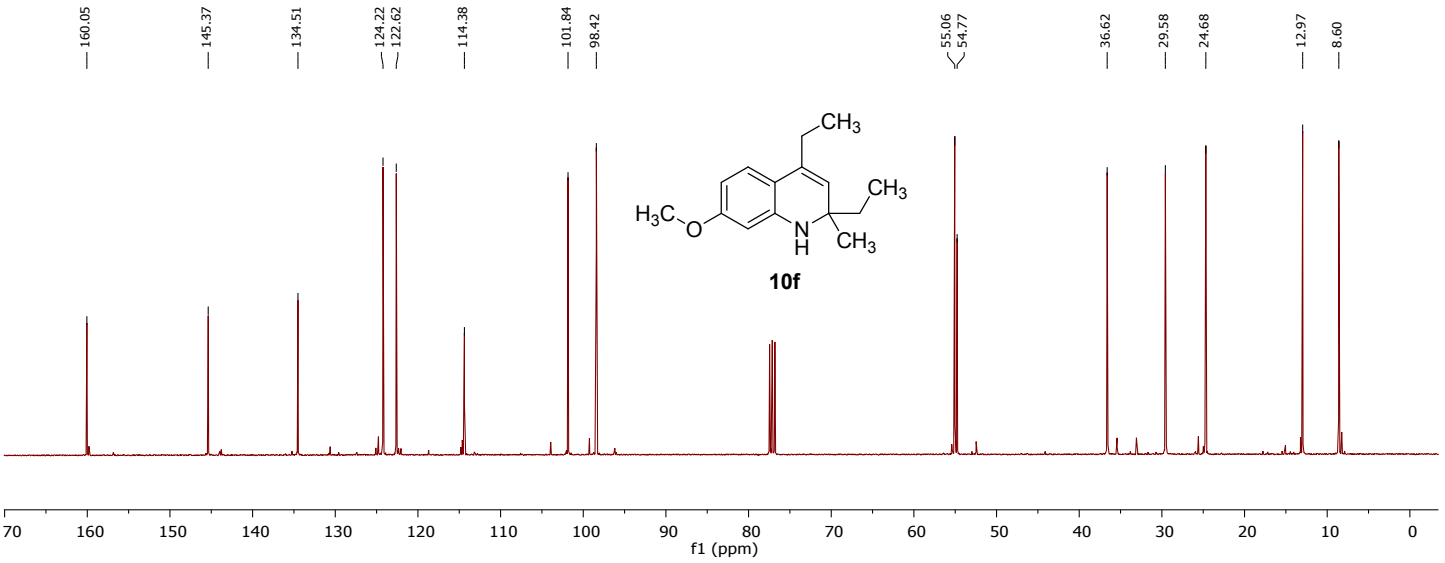
10e



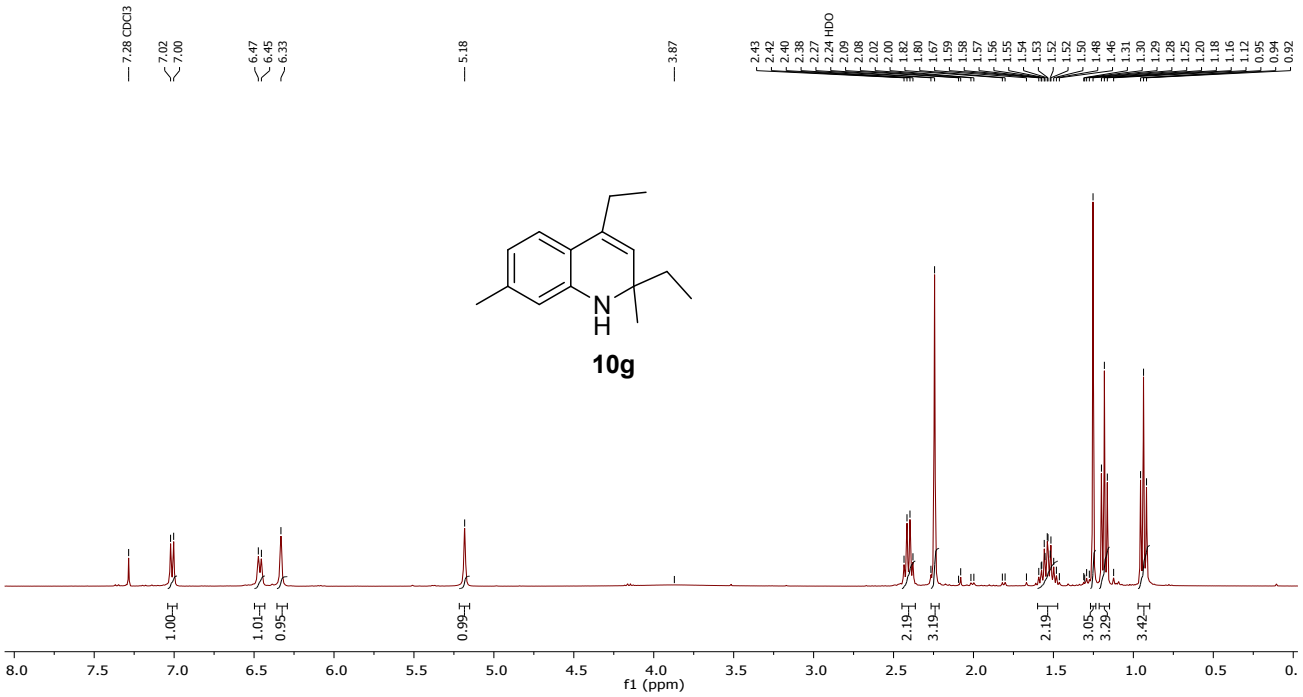
10e

^{1}H and ^{13}C spectra of compound 10f

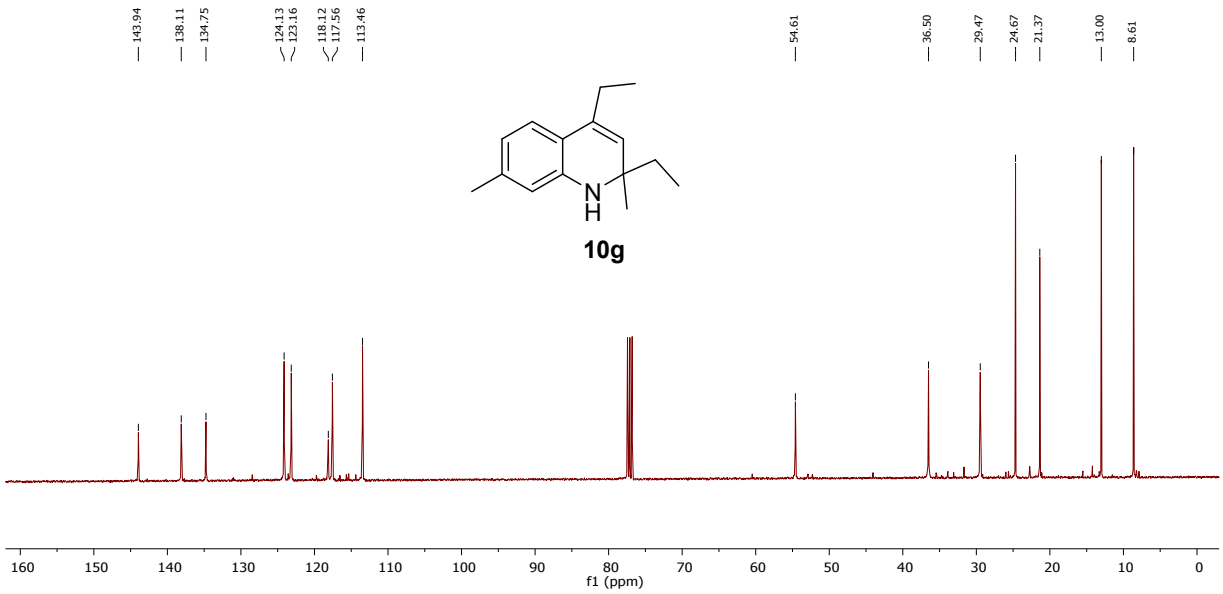

7.01
6.99
6.19
6.18
6.17
6.16
6.04
6.03
5.09
3.75
2.39
2.38
2.36
2.34
1.54
1.52
1.50
1.48
1.47
1.22
1.17
1.15
1.13
0.93
0.91
0.89
CH3
CH3
H3C
O
N
H
CH3
10f
1.00
0.99
1.01
1.03
3.12
2.13
2.13
3.03
3.18
3.13
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


160.05
145.37
134.51
124.22
122.62
114.38
101.84
98.42
55.06
54.77
36.62
29.58
24.68
12.97
8.60
CH3
CH3
H3C
O
N
H
CH3
10f
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{1}H and ^{13}C spectra of compound 10g



10g



10g