

Supporting Information

Dual C–H Functionalization of N-Aryl Tetrahydroisoquinolines: Highly Diastereoselective Synthesis of Dibenzo[*a,f*]quinolizines via Visible-Light Induced Oxidation and Inverse Electron-Demand Aza- Diels–Alder Reaction

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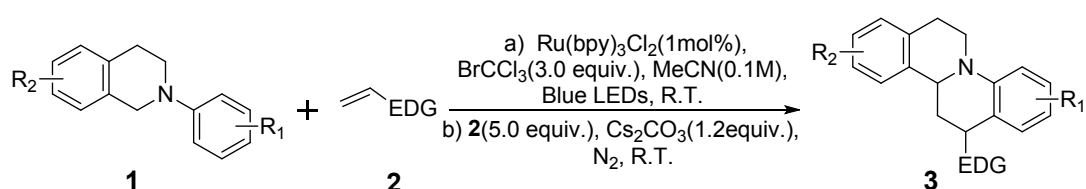
1. General information

All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography (TLC) plates were visualized by exposure to ultraviolet light and/or staining with phosphomolybdic acid followed by heating on a hot plate. Flash chromatography was carried out using silica gel (200-300 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker AM-400 (400 MHz). The spectra were recorded in CDCl_3 as solvent at room temperature, ^1H and ^{13}C NMR chemical shifts are reported in ppm relative to the residual solvent peak. The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale (CDCl_3 : $\delta_{\text{H}} = 7.26$ ppm, $\delta_{\text{C}} = 77.00$ ppm). Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet, comp = complex), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported as chemical shift. IR spectra were recorded using Nicolet NEXUS 670 FT-IR instrument and are reported in wavenumbers (cm^{-1}). HRMS were performed on a Bruker Apex II mass instrument (ESI).

2. General procedure for the synthesis of substrates

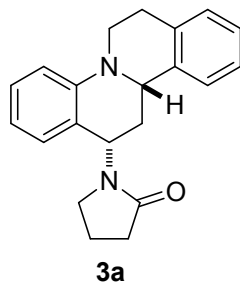
All substrates were either commercially available and used without further purification or were prepared according to literature procedures^{1,2}

3. General procedure for the synthesis of product 3 and analytical data



A mixture of tetrahydroisoquinoline **1** (0.2 mmol), [Ru(bpy)₃]Cl₂·6H₂O (1.5 mg, 1 mol%) and acetonitrile (2 mL) was degassed by three cycles of freeze-pump-thaw. BrCCl₃ (59.1 μL, 0.6 mmol, 3.0 equiv.) was added and the mixture was stirred under an inert atmosphere at room temperature while irradiated by blue LEDs (at a distance of approximately 5 cm so that the reaction mixture did not heat up during the reaction). Full conversion of the tetrahydroisoquinoline was ensured by TLC. Then, to the reaction mixture was added Electron-donating alkene **2** (1 mmol, 5.0 equiv.) and Cs₂CO₃ (78.2 mg, 0.24 mmol, 1.2 equiv.), then the reaction system was evacuated and refilled with argon three times. The reaction mixture was stirred overnight (16 h) in dark under an inert atmosphere at room temperature. After completion of the reaction, the crude mixture was purified by flash chromatography (silica gel, mixtures of petroleum/ethyl acetate) to afford the pure product **3**.

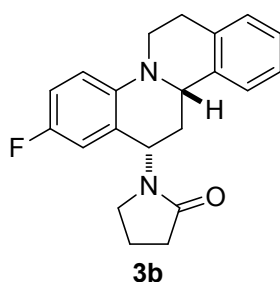
13-cis-1-(7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3a)



Following the general procedure, compound **3a** was obtained as a white solid in 86% yield (>20:1 d.r.); mp=175-178°C; $R_f=0.27$ (petroleum/ethyl acetate 1:1 v/v);

^1H NMR (400 MHz, CDCl_3): δ 7.25-7.15(m, 5H), 6.93 (app d, $J=7.6$ Hz, 1H), 6.86 (app d, $J=8.4$ Hz, 1H), 6.73 (app t, $J=7.2$ Hz, 1H), 5.77 (dd, $J=11.6, 6.4$ Hz, 1H), 4.55 (d, $J=11.8$ Hz, 1H), 3.87-3.83 (m, 1H), 3.19-3.04 (comp, 4H), 2.92-2.86 (m, 1H), 2.56-2.42 (comp, 3H), 2.10 (q, $J=11.6$ Hz, 1H), 1.98-1.93 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.6, 147.5, 137.2, 134.7, 128.5, 128.3, 127.1, 126.4, 126.2, 125.8, 121.4, 117.8, 113.0, 55.9, 48.6, 43.4, 42.3, 33.9, 31.4, 29.7, 18.1; IR (KBr, cm^{-1}): 3449, 2925, 1685, 1601, 1490, 1457, 1422, 1287, 1232, 750; HRMS (ESI) for $\text{C}_{21}\text{H}_{23}\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.319.1805, found 319.1801.

13-cis-1-(2-fluoro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3b)

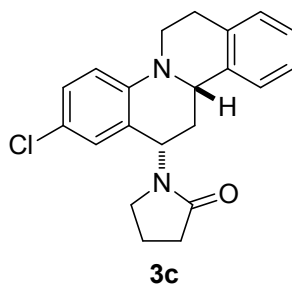


Following the general procedure, compound **3b** was obtained as a white solid in 82% yield (>20:1 d.r.); mp=150-153°C; $R_f=0.34$ (petroleum/ethyl acetate 1:1 v/v);

^1H NMR (400 MHz, CDCl_3): δ 7.23-7.16(m, 4H), 6.88 (app td, $J= 8.7, 3.2$ Hz, 1H), 6.81 (app dd, $J=9.0, 4.7$ Hz, 1H), 6.68 (app dd, $J=9.0, 2.9$ Hz, 1H), 5.74 (dd, $J=11.8, 6.6$ Hz, 1H), 4.50 (d, $J=10.2$ Hz, 1H), 3.82-3.76 (m, 1H), 3.24-3.17 (m, 1H), 3.14-3.03 (comp, 3H), 2.97-2.90 (m, 1H), 2.57-2.44 (comp, 3H), 2.09 (q, $J=11.9$ Hz, 1H),

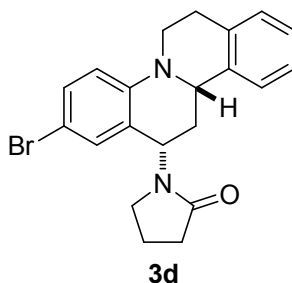
20.5-1.97 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.6, 157.3, 154.9, 144.2, 136.9, 134.4, 128.5, 126.4, 126.3, 125.9, 123.6(1), 123.5(5), 115.1(3), 115.0(5), 114.9(8), 114.8, 113.5, 113.2, 56.0, 48.5, 44.1, 42.3, 33.2, 31.2, 29.7, 18.1; IR (KBr, cm^{-1}): 3428, 2925, 1683, 1493, 1460, 1424, 1286, 1228, 924, 734; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{F}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.337.1711, found 337.1709.

13-cis-1-(2-chloro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3c)



Following the general procedure, compound **3c** was obtained as a white solid in 88% yield (>20:1 d.r.); mp=76-78°C; R_f =0.35 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.24-7.16(m, 4H), 7.10 (app dd, J = 8.8, 2.5 Hz, 1H), 6.87 (app d, J =9.0 Hz, 1H), 6.68 (d, J =9.0 Hz, 1H), 5.72 (dd, J =11.9, 6.4 Hz, 1H), 4.56 (d, J =9.5 Hz, 1H), 3.88-3.81 (m, 1H), 3.22-3.04 (comp, 4H), 2.93-2.87 (m, 1H), 2.60-2.44 (comp, 3H), 2.09 (q, J =11.8 Hz, 1H), 2.04-1.97 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 146.0, 136.8, 134.6, 128.5, 128.2, 126.5(3), 126.4(9), 126.4, 125.6, 123.0, 122.6, 114.2, 55.7, 48.3, 43.5, 42.3, 33.6, 31.2, 29.5, 18.1; IR (KBr, cm^{-1}): 3424, 2924, 1685, 1677, 1597, 1489, 1460, 1421, 1287, 1232, 1187, 1123, 781, 656; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{Cl}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.353.1415, found 353.1411.

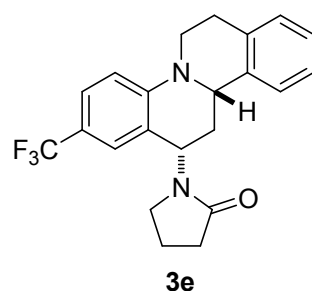
13-cis-1-(2-bromo-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3d)



Following the general procedure, compound **3d** was obtained as a white solid in 90% yield (>20:1 d.r.); mp=161-163°C; R_f =0.36 (petroleum/ethyl acetate 1:1 v/v); ^1H

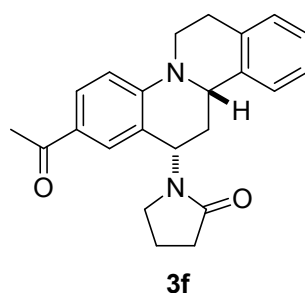
NMR (400 MHz, CDCl₃): δ 7.25-7.16(m, 5H), 6.99 (app dd, $J=2.2, 0.8$ Hz, 1H), 6.71 (d, $J=8.9$ Hz, 1H), 5.72 (dd, $J=11.8, 6.4$ Hz, 1H), 4.56 (d, $J=9.5$ Hz, 1H), 3.87-3.81 (m, 1H), 3.22-3.03 (comp, 4H), 2.93-2.87 (m, 1H), 2.60-2.44 (comp, 3H), 2.09 (q, $J=11.8$ Hz, 1H), 20.4-1.97 (m, 2H); ¹³C NMR (400 MHz, CDCl₃): δ 175.6, 146.3, 136.8, 134.6, 131.1, 129.3, 128.5, 126.5, 126.4, 125.6, 123.4, 114.5, 109.6, 55.7, 48.2, 43.4, 42.3, 33.6, 31.2, 29.4, 18.1; IR (KBr, cm⁻¹): 3448, 2926, 2241, 1679, 1592, 1487, 1460, 1420, 1287, 1231, 731; HRMS (ESI) for C₂₁H₂₂BrN₂O₁ [M+H]⁺ calcd.397.0910, found 397.0906.

13-cis-1-(2-(trifluoromethyl)-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3e)



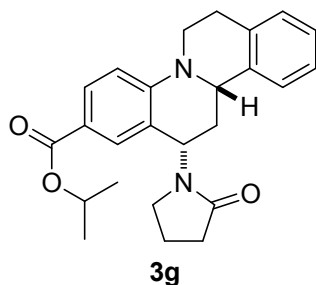
Following the general procedure, compound **3e** was obtained as a white solid in 86% yield (>20:1 d.r.); mp=80-82°C; R_f=0.39 (petroleum/ethyl acetate 1:1 v/v); ¹H NMR (400 MHz, CDCl₃): δ 7.37(d, $J=8.6$ Hz, 1H)7.25-7.18(m, 4H), 7.09 (s, 1H), 6.82 (d, $J=8.7$ Hz, 1H), 5.72 (dd, $J=12.0, 5.8$ Hz, 1H), 4.59 (d, $J=9.6$ Hz, 1H), 3.97-3.92 (m, 1H), 3.22-3.14 (m, 2H), 3.12-3.04 (m,2H), 2.92-2.86(m, 1H), 2.61-2.45 (comp, 3H), 2.09 (q, $J=11.9$ Hz, 1H), 2.05-1.98 (m, 2H); ¹³C NMR (400 MHz, CDCl₃): δ 175.8, 149.1, 136.8, 134.7, 128.4, 126.7, 126.5, 125.6(5), 125.6(1), 125.4, 123.5(4), 123.5(0), 120.6, 119.0, 118.7, 118.4, 118.2, 111.5, 55.3, 48.1, 43.4, 42.3, 33.5, 31.2, 29.3, 18.2; IR (KBr, cm⁻¹): 3371, 2925, 1686, 1619, 1544, 1460, 1421, 1333, 1302, 1266, 1157, 1113,739; HRMS (ESI) for C₂₂H₂₂F₃N₂O₁ [M+H]⁺ calcd.387.1679, found 387.1676.

13-cis-1-(2-acetyl-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3f)



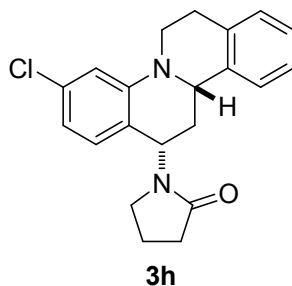
Following the general procedure, compound **3f** was obtained as a white solid in 72% yield (>20:1 d.r.); mp=191-194°C; $R_f=0.15$ (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.78 (dd, $J=8.8, 2.0$ Hz, 1H), 7.50 (m, 1H), 7.26-7.18 (m, 4H), 6.77 (d, $J=8.8$ Hz, 1H), 5.71 (dd, $J=12.0, 5.4$ Hz, 1H), 4.75 (dd, $J=11.2, 2.6$ Hz, 1H), 4.02-3.97 (m, 1H), 3.29-3.22 (m, 1H), 3.18 (t, $J=6.6$ Hz, 2H), 3.12-3.05 (m, 1H), 2.90 (app dt, $J=15.6, 4.0$ Hz, 1H), 2.64-2.50 (comp, 3H), 2.48 (s, 3H), 2.14-1.99 (comp, 3H); ^{13}C NMR (400 MHz, CDCl_3): δ 195.9, 175.7, 150.1, 136.7, 134.7, 129.8, 128.3, 126.9, 126.7, 126.5, 125.9, 125.2, 119.6, 110.5, 55.1, 47.9, 43.5, 42.2, 33.3, 31.2, 29.1, 25.9, 18.2; IR (KBr, cm^{-1}): 3318, 3052, 2927, 1681, 1600, 1509, 1423, 1290, 1252, 736; HRMS (ESI) for $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ calcd.361.1911, found 361.1909.

13-cis-isopropyl13-(2-oxopyrrolidin-1-yl)-7,11b,12,13-tetrahydro-6H-isoquinolino [2,1-a]quinoline-2-carboxylate (3g)



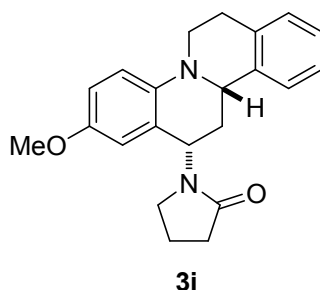
Following the general procedure, compound **3g** was obtained as a white solid in 82% yield (>20:1 d.r.); mp=156-160°C; $R_f=0.22$ (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.82 (dd, $J=8.8, 1.9$ Hz, 1H), 7.54 (s, 1H), 7.27-7.17 (m, 4H), 6.76 (d, $J=8.8$ Hz, 1H), 5.71 (dd, $J=12.0, 5.5$ Hz, 1H), 5.23-5.15 (m, 1H), 4.73 (d, $J=9.1$ Hz, 1H), 4.01-3.96 (m, 1H), 3.26-3.04 (comp, 4H), 2.88 (app dt, $J=15.6, 4.0$ Hz, 1H), 2.58-2.45 (comp, 3H), 2.09 (q, $J=12.0$ Hz, 1H), 2.05-1.93 (m, 3H), 1.33 (d, $J=2.4$ Hz, 3H), 1.32 (d, $J=2.4$ Hz, 3H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.8, 166.0, 159.9, 136.9, 134.8, 130.4, 128.4, 126.7, 126.5, 125.4, 119.7, 110.8, 55.3, 48.0, 43.5, 42.3, 33.5, 31.3, 29.2, 21.9(8), 21.9(6), 18.3; IR (KBr, cm^{-1}): 3365, 2979, 2929, 1686, 1609, 1509, 1422, 1384, 1791, 1249, 1181, 1107, 911, 732; HRMS (ESI) for $\text{C}_{25}\text{H}_{29}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ calcd.405.2173, found 405.2171.

13-cis-1-(3-chloro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3h)



Following the general procedure, compound **3h** was obtained as a white solid in 45% yield (>20:1 d.r.); mp=78-79°C; R_f =0.27 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.22-7.16 (m, 4H), 6.80 (app dd, J = 8.8, 2.4 Hz, 2H), 6.68 (app dd, J =8.0, 1.6 Hz, 1H), 5.70 (dd, J =11.6, 6.0 Hz, 1H), 4.59 (d, J =10.0 Hz, 1H), 3.88-3.82 (m, 1H), 3.20-3.03 (comp, 4H), 2.92-2.86 (m, 1H), 2.53-2.47 (comp, 3H), 2.11-1.95 (comp, 3H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 148.2, 136.8, 134.6, 134.0, 128.5, 128.0, 126.6, 126.4, 125.6, 119.5, 117.2, 112.4, 55.7, 48.1, 43.2, 42.3, 33.9, 31.3, 29.4, 18.1; IR (KBr, cm^{-1}): 3355, 2926, 1681, 1597, 1486, 1460, 1422, 1287, 732; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{Cl}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.353.1415, found 353.1412.

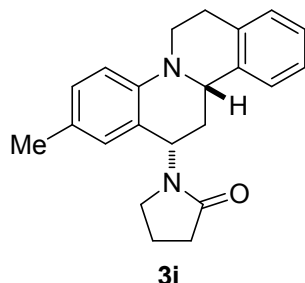
13-cis-1-(2-methoxy-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3i)



Following the general procedure, compound **3i** was obtained as a white solid in 66% yield (>20:1 d.r.); mp=66-67°C; R_f =0.22 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.26-7.14 (m, 4H), 6.87 (d, J =9.0 Hz, 1H), 6.77 (app dd, J =8.9, 2.9 Hz, 1H), 6.55 (d, J =2.6Hz, 1H), 5.74 (app dd, J =11.5, 7.3 Hz, 1H), 4.42 (d, J =10.9 Hz, 1H), 3.78-3.75 (m, 1H), 3.74 (s, 3H), 3.21-3.15 (m, 1H), 3.13-3.00 (m, 3H), 2.98-2.91 (m, 1H), 2.54-2.46 (m, 2H), 2.46-2.41 (m, 1H), 2.10 (q, J =11.7 Hz, 1H), 2.01-1.93 (m, 2H). ^{13}C NMR (400 MHz, CDCl_3): δ 175.6, 152.7, 142.4, 137.1, 134.4, 128.5, 126.3, 126.1, 126.0, 123.7, 116.0, 114.0, 112.7, 56.2, 55.7, 48.8, 44.5, 42.3, 33.1, 31.3, 29.8, 18.3; IR (KBr, cm^{-1}): 3352, 2926, 1678, 1495, 1461, 1422, 1287, 1235, 1041, 732; HRMS (ESI) for $\text{C}_{22}\text{H}_{25}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ calcd.349.1911, found

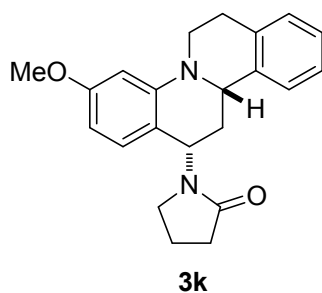
349.1909.

13-cis-1-(2-methyl-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3i)



Following the general procedure, compound **3i** was obtained as a white solid in 64% yield (>20:1 d.r.); mp=88-90°C; R_f =0.30 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.24-7.14 (m, 4H), 6.98 (d, J =8.1 Hz, 1H), 6.79 (d, J =8.4 Hz, 1H), 6.75 (s, 1H), 5.74 (app dd, J =11.6, 6.7 Hz, 1H), 4.47 (d, J =11.0 Hz, 1H), 3.89-3.83 (m, 1H), 3.19-3.13 (m, 1H), 3.12-3.00 (m, 3H), 2.91-2.85 (m, 1H), 2.58-2.41 (m, 3H), 2.23 (s, 3H), 2.09 (q, J =12.1 Hz, 1H), 2.00-1.93 (m, 2H). ^{13}C NMR (400 MHz, CDCl_3): δ 175.6, 145.5, 137.2, 134.6, 128.9, 128.5, 127.6, 127.4, 126.3, 126.2, 125.8, 121.6, 113.7, 56.1, 55.7, 48.5, 43.6, 42.3, 33.8, 31.4, 29.7, 20.3, 18.1; IR (KBr, cm^{-1}): 3294, 2925, 1679, 1504, 1460, 1422, 1287, 1230, 733; HRMS (ESI) for $\text{C}_{22}\text{H}_{25}\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.333.1961, found 333.1959.

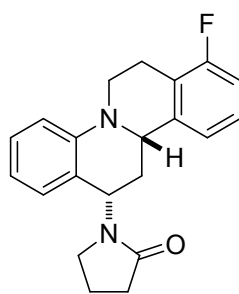
13-cis-1-(3-methoxy-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3k)



Following the general procedure, compound **3k** was obtained as a colorless oil in 11% yield (>20:1 d.r.); R_f =0.17 (petroleum/ethyl acetate 1:2 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.27-7.25 (m, 1H), 7.19-7.09 (m, 4H), 6.59 (d, J =8.4 Hz, 1H), 6.31 (d, J =8.4 Hz, 1H), 5.57 (app d, J =7.6 Hz, 1H), 4.44 (d, J =8.8 Hz, 1H), 4.03-3.98 (m, 1H), 3.75 (s, 3H), 3.14-3.03(m, 2H), 2.81-2.77 (m, 1H), 2.63-2.51 (comp, 3H), 2.46-2.39 (m, 1H), 2.31-2.25(m, 2H), 1.71-1.64 (m, 1H), 1.51-1.44 (m, 1H). ^{13}C NMR (400

MHz, CDCl₃): δ 174.6, 158.6, 149.2, 137.3, 134.5, 129.1, 128.6, 126.2, 126.1, 125.9, 109.7, 106.2, 100.2, 55.6, 55.4, 44.4, 44.1, 43.5, 34.0, 31.4, 28.7, 17.9; IR (KBr, cm⁻¹): 3444, 2928, 1664, 1599, 1475, 1422, 1269, 1231, 1106, 740; HRMS (ESI) for C₂₂H₂₅N₂O₂ [M+H]⁺ calcd.349.1911, found 349.1910.

13-cis-1-(8-fluoro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3l)

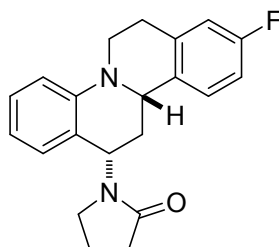


3l

Following the general procedure, compound **3l** was obtained as a white solid in 60% yield (>20:1 d.r.); mp=166-168°C; R_f=0.22 (petroleum/ethyl acetate 1:1 v/v); ¹H

NMR (400 MHz, CDCl₃): δ 7.22-7.16 (m, 2H), 7.03 (app d, *J*= 12 Hz, 1H), 6.95-6.90 (m, 3H), 6.68 (app t, *J*=7.2, 1H), 5.76 (dd, *J*=12.0, 6.4 Hz, 1H), 4.54 (d, *J*=11.2 Hz, 1H), 4.01-3.95 (m, 1H), 3.12-2.90 (comp, 5H), 2.53-2.45 (comp, 3H), 2.10 (q, *J*=11.6 Hz, 1H), 2.14-1.94 (m, 2H); ¹³C NMR (400 MHz, CDCl₃): δ 175.7, 161.4, 159.0, 147.3, 139.8, 139.7, 128.4, 126.2(7), 126.1(6), 127.0(7), 122.6, 122.4, 121.6, 121.4, 121.3, 118.4, 113.5, 112.7, 112.4, 55.8(4), 55.8(2), 48.5, 42.7, 42.4, 33.8, 31.5, 31.4, 22.5(9), 22.5(5), 18.1, 14.07; IR (KBr, cm⁻¹): 3352, 2926, 1686, 1601, 1492, 1459, 1421, 1287, 1254, 740; HRMS (ESI) for C₂₁H₂₂F₁N₂O₁ [M+H]⁺ calcd.337.1711, found 337.1713.

13-cis-1-(9-fluoro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3m)

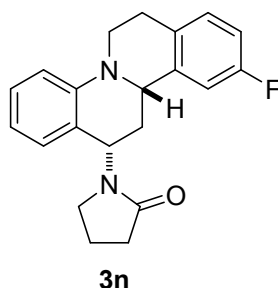


3m

Following the general procedure, compound **3m** was obtained as a white solid in 50%

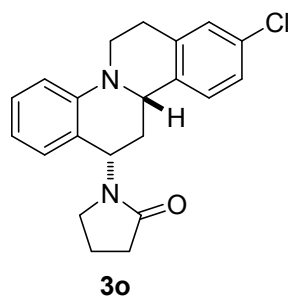
yield (>20:1 d.r.); mp=75-78°C; R_f =0.26 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.21-7.15 (m, 2H), 6.94-6.90 (m, 2H), 6.89-6.85 (m, 2H), 6.75 (app t, J =7.6, 1H), 5.76 (dd, J =11.6, 6.4 Hz, 1H), 4.51 (d, J =11.2 Hz, 1H), 3.93-3.87 (m, 1H), 3.20-3.02 (comp, 4H), 2.91-2.85 (m, 1H), 2.56-2.44 (comp, 3H), 2.08 (q, J =12 Hz, 1H), 2.02-1.94 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 162.4, 159.9, 147.3, 137.0, 136.9, 132.9(4), 132.9(2), 128.4, 127.5, 127.4, 127.1, 121.4, 118.1, 114.8, 114.6, 113.5, 113.3, 113.2, 55.4, 48.4, 43.3, 42.3, 33.8, 31.4, 29.7, 18.1; IR (KBr, cm^{-1}): 3289, 2925, 1686, 1601, 1491, 1459, 1422, 1277, 1227, 739; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{F}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.337.1711, found 337.1712.

13-cis-1-(10-fluoro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3n)



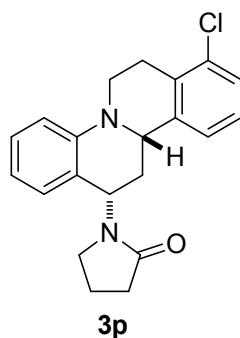
Following the general procedure, compound **3n** was obtained as a white solid in 56% yield (>20:1 d.r.); mp=180-183°C; R_f =0.35 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.19-7.10 (m, 2H), 6.96-6.85 (m, 4H), 6.75 (app t, J =7.2, 1H), 5.76 (dd, J =11.6, 6.4 Hz, 1H), 4.52 (d, J =10.8 Hz, 1H), 3.95-3.91 (m, 1H), 3.19-3.01 (comp, 4H), 2.88-2.85 (m, 1H), 2.54-2.43 (comp, 3H), 2.08 (q, J =12.0 Hz, 1H), 2.03-1.95(m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 162.6, 160.1, 147.1, 139.2, 139.1, 130.4, 130.3, 129.9(7), 129.8(9), 128.4, 127.1, 121.3, 118.1, 113.7, 113.5, 113.1, 112.5, 112.3, 55.8(1), 55.7(9), 48.4, 43.6, 42.3, 33.7, 31.4, 28.9, 18.1; IR (KBr, cm^{-1}): 3347, 2921, 1680, 1601, 1491, 1457, 1422, 1268, 739; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{F}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.337.1711, found 337.1710.

13-cis-1-(9-chloro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3o)



Following the general procedure, compound **3o** was obtained as a white solid in 85% yield (>20:1 d.r.); mp=78-80°C; R_f =0.26 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.21-7.16 (m, 4H), 6.93 (d, J = 7.6 Hz, 1H), 6.86 (d, J =8.0 Hz, 1H), 6.68 (t, J =7.6 Hz, 1H), 5.76 (dd, J =12.0, 6.4 Hz, 1H), 4.51 (d, J =10.4 Hz, 1H), 3.94-3.90 (m, 1H), 3.20-3.01 (comp, 4H), 2.91-2.85 (m, 1H), 2.56-2.43 (comp, 3H), 2.09 (q, J =12.0 Hz, 1H), 2.02-1.95 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 147.3, 136.7, 135.8, 132.1, 128.4, 128.3, 127.3, 127.2, 126.5, 121.4, 118.2, 113.2, 55.7, 48.5, 42.3, 42.4, 33.8, 31.4, 29.6, 18.2; IR (KBr, cm^{-1}): 3289, 2927, 1681, 1600, 1488, 1457, 1421, 1286, 735; HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{Cl}_1\text{N}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd.353.1415, found 353.1412.

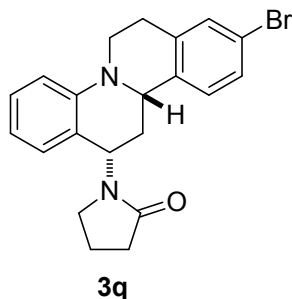
13-cis-1-(8-chloro-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3p)



Following the general procedure, compound **3p** was obtained as a white solid in 80% yield (>20:1 d.r.); mp=190-194°C; R_f =0.19 (petroleum/ethyl acetate 1:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.27 (d, J = 4.8 Hz, 1H), 7.26-7.16(m, 3H), 6.92 (dd, J = 8.0, 12.0 Hz, 2H), 6.77 (t, J =7.2 Hz, 1H), 5.76 (dd, J =11.6, 6.4 Hz, 1H), 4.53 (d, J =10.4 Hz, 1H), 4.02-3.96 (m, 1H), 3.18-2.94 (comp, 5H), 2.55-2.43 (comp, 3H), 2.09 (q, J =11.6 Hz, 1H), 2.04-1.93 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 147.2, 139.5, 134.0, 132.9, 128.4, 127.2, 127.0, 124.4, 121.5, 118.4, 113.5, 56.0, 48.5, 43.1, 42.3, 33.9, 31.4, 27.4, 18.1; IR (KBr, cm^{-1}): 3348, 2926, 1679, 1602, 1492, 1458,

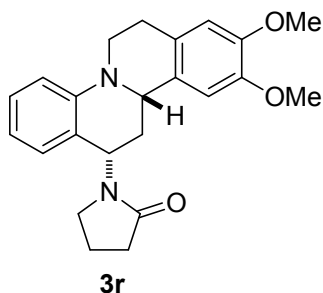
1422, 1287, 732; HRMS (ESI) for C₂₁H₂₂Cl₁N₂O₁ [M+H]⁺ calcd.353.1415, found 353.1413.

13-cis-1-(9-bromo-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3q)



Following the general procedure, compound **3q** was obtained as a white solid in 73% yield (>20:1 d.r.); mp=83-85°C; R_f=0.26 (petroleum/ethyl acetate 1:1 v/v); ¹H NMR (400 MHz, CDCl₃): δ 7.34-7.35 (m, 2H), 7.17 (t, *J*=7.6 Hz, 1H), 7.12 (d, *J*= 8.2 Hz, 1H), 6.91 (d, *J*=7.6 Hz, 1H), 6.86 (d, *J*=8.2, Hz, 1H), 6.76 (t, *J*=7.2 Hz, 1H), 5.76 (dd, *J*=11.6, 6.4 Hz, 1H), 4.49 (d, *J*=10.0 Hz, 1H), 3.94-3.87 (m, 1H), 3.20-3.01 (m, 4H), 2.91-2.84 (m, 1H), 2.54-2.43 (comp, 3H), 2.09 (q *J*=12.0 Hz, 1H), 20.5-1.97 (m, 2H); ¹³C NMR (400 MHz, CDCl₃): δ 175.7, 147.2, 137.1, 136.3, 131.3, 129.4, 128.4, 127.6, 127.2, 121.4, 120.2, 118.2, 113.2, 55.7, 48.5, 43.2, 42.4, 33.7, 31.4, 29.5, 18.1; IR (KBr, cm⁻¹): 3282, 2926, 1679, 1601, 1486, 1458, 1421, 1286, 735; HRMS (ESI) for C₂₁H₂₂Br₁N₂O₁ [M+H]⁺ calcd.397.0910, found 397.0906.

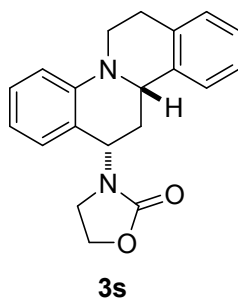
13-cis-1-(9,10-dimethoxy-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)pyrrolidin-2-one (3r)



Following the general procedure, compound **3r** was obtained as a colorless oil in 34% yield (>20:1 d.r.); R_f=0.19 (petroleum/ethyl acetate 1:1 v/v); ¹H NMR (400 MHz, CDCl₃): δ 7.19-7.16 (m, 1H), 6.94 (d, *J*= 7.6 Hz, 1H), 6.90 (d, *J*=8.2 Hz, 1H), 6.75 (t, *J*=7.6 Hz, 1H), 6.71 (s, 1H), 6.64(s, 1H), 5.77 (dd, *J*=11.6, 6.8 Hz, 1H), 4.47 (d, *J*=10.8 Hz, 1H), 3.95-3.90 (m, 1H), 3.879 (s, 3H), 3.876 (s, 3H), 3.20-2.97 (comp,

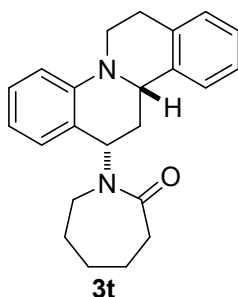
4H), 2.81-2.77 (m, 1H), 2.56-2.44 (m, 3H), 2.09 (q, $J=11.6$ Hz, 1H), 20.1-1.94 (m, 2H); ^{13}C NMR (400 MHz, CDCl_3): δ 175.7, 147.7, 147.6, 128.9, 128.4, 127.4, 126.7, 121.4, 118.1, 113.3, 111.1, 108.8, 56.1, 55.9, 55.8, 48.7, 43.3, 42.4, 34.2, 31.5, 29.3, 18.1; IR (KBr, cm^{-1}): 3357, 2926, 1683, 1601, 1513, 1492, 1459, 1422, 1379, 1274, 733; HRMS (ESI) for $\text{C}_{23}\text{H}_{27}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ calcd.379.2016, found 379.2013.

13-cis-3-(7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)oxazolidin-2-one (3s)



Following the general procedure, compound **3s** was obtained as a white solid in 69% yield (>20:1 d.r.); mp=169-172°C; $R_f=0.30$ (petroleum/ethyl acetate 2:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.27-7.16(m, 5H), 7.08 (d, $J=7.6$ Hz, 1H), 6.88 (d, $J=8.2$ Hz, 1H), 6.77 (t, $J=7.2$ Hz, 1H), 5.53 (dd, $J=11.5, 6.4$ Hz, 1H), 4.57 (d, $J=10.8$ Hz, 1H), 3.35-3.25 (m, 2H), 3.97-3.90 (m, 1H), 3.34-3.24 (m, 2H), 3.13-3.05 (m, 2H), 2.93-2.86 (m, 1H), 2.64-2.58 (m, 1H), 2.15 (q, $J=11.6$ Hz, 1H); ^{13}C NMR (400 MHz, CDCl_3): δ 178.8, 147.4, 137.0, 134.7, 128.8, 128.6, 127.4, 126.5, 126.4, 125.8, 120.5, 118.0, 113.1, 62.1, 55.6, 50.7, 43.3, 39.8, 33.9, 29.5; IR (KBr, cm^{-1}): 3451, 2925, 1746, 1602, 1490, 1456, 1424, 1380, 734; HRMS (ESI) for $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ calcd.321.1598, found 321.1596.

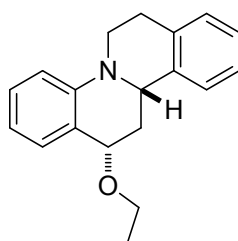
13-cis-1-(7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)azepan-2-one (3t)



Following the general procedure, compound **3t** was obtained as a white solid in 68% yield (>20:1 d.r.); mp=62-64°C; $R_f=0.24$ (petroleum/ethyl acetate 2:1 v/v); ^1H NMR

(400 MHz, CDCl₃): δ 7.27-7.24 (m, 1H), 7.23-7.13 (m, 4H), 7.00 (d, J = 7.6 Hz, 1H), 6.85 (d, J = 8.2 Hz, 1H), 6.73 (app t, J =7.6 Hz, 1H), 6.27 (dd, J =12.0, 6.8 Hz, 1H), 4.50 (d, J =10.8 Hz, 1H), 3.92-3.88 (m, 1H), 3.14-3.02 (comp, 4H), 3.00-2.87 (m, 1H), 2.67-2.64 (m, 2H), 2.57-2.52 (m, 1H), 2.00 (d, J =12.0 Hz, 1H), 1.83-1.77 (m, 2H), 1.68-1.55 (m, 3H), 1.35 (d, J =8.2 Hz, 1H); ¹³C NMR (400 MHz, CDCl₃): δ 176.7, 148.4, 137.3, 134.6, 128.5, 128.1, 127.9, 126.3, 126.2, 126.0, 122.6, 118.0, 113.0, 56.3, 51.8, 44.8, 43.2, 37.6, 35.0, 30.0, 29.9, 29.4, 23.6; IR (KBr, cm⁻¹): 3323, 2928, 2856, 1633, 1602, 1489, 1455, 1421, 1306, 1188, 909, 732; HRMS (ESI) for C₂₃H₂₇N₂O₁ [M+H]⁺ calcd.347.2118, found 347.2115.

13-cis-13-ethoxy-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolone (3u)

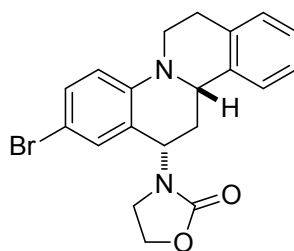


3u

Following the general procedure, compound **3u** was obtained as a white solid in 72% yield (>20:1 d.r.); mp=64-66°C; R_f=0.35 (petroleum/ethyl acetate 20:1 v/v); ¹H NMR

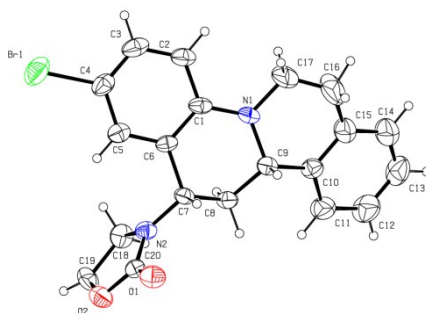
(400 MHz, CDCl₃): δ 7.43-7.41(d, J = 7.6 Hz, 1H), 7.27-7.24 (m, 1H), 7.22-7.14 (M, 4H), 6.80-6.74 (m, 2H), 4.82 (dd, J =10.8, 6.0 Hz, 1H), 4.52 (dd, J =11.6, 2.8 Hz, 1H), 3.88-3.84 (m, 1H), 3.78-3.74 (m, 1H), 3.65-3.62 (m, 1H), 3.19-3.07 (m, 2H), 2.90-2.77 (comp, 2H), 2.04 (q, J =12.0 Hz, 1H), 1.29 (t, J =7.2 Hz, 3H); ¹³C NMR (400 MHz, CDCl₃): δ 145.9, 138.0, 135.2, 128.4, 128.3, 126.8, 126.3, 126.1, 125.4(2), 125.3(7), 117.4, 112.3, 74.1, 63.6, 55.2, 44.0, 35.5, 29.3, 15.6; IR (KBr, cm⁻¹): 3042, 2972, 2926, 1601, 1576, 1489, 1456, 1383, 1295, 1265, 1123, 737; HRMS (ESI) for C₁₉H₂₂N₁O₁ [M+H]⁺ calcd.280.1696, found 280.1694.

13-cis-3-(2-bromo-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)oxazolidin-2-one (3v)

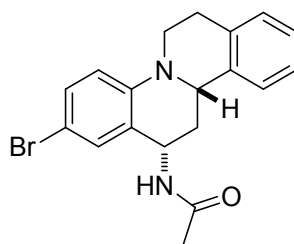


3v

Following the general procedure, compound **3v** was obtained as a white solid in 74% yield (>20:1 d.r.); mp=60-62°C; $R_f=0.34$ (petroleum/ethyl acetate 2:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.28-7.27(m, 1H), 7.25-7.16 (m, 5H), 6.72 (d, $J=8.8$ Hz, 1H), 5.47 (dd, $J=11.6, 6.4$ Hz, 1H), 4.58 (d, $J=10.8$ Hz, 1H), 4.40-4.28 (m, 2H), 3.88-3.85 (m, 1H), 3.34-3.29 (m, 2H), 3.13-3.03 (m, 2H), 2.92-2.86 (m, 1H), 2.61-2.56 (m, 1H), 2.12 (q, $J=12.0$ Hz, 1H); ^{13}C NMR (400 MHz, CDCl_3): δ 158.6, 146.3, 136.6, 134.6, 131.6, 129.6, 128.6, 126.7, 126.5, 125.7, 122.6, 114.6, 109.8, 62.2, 55.6, 50.4, 43.4, 39.8, 33.6, 29.4; IR (KBr, cm^{-1}): 3316, 2925, 2851, 1744, 1593, 1487, 1422, 1265, 736; HRMS (ESI) for $\text{C}_{20}\text{H}_{20}\text{Br}_1\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ calcd.399.0703, found 399.0700. Crystals of the major diastereomer of **3v** were obtained from hexane/dichloromethane through slow diffusion at room temperature. The relative stereochemistry was assigned by X-ray diffraction.



13-cis-N-(2-bromo-7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-13-yl)acetamide



3w

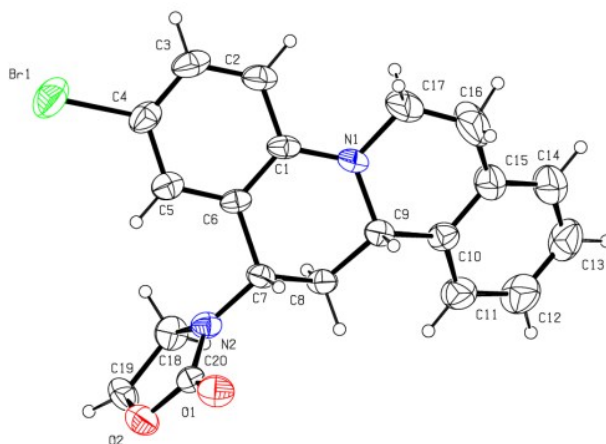
Following the general procedure, compound **3w** was obtained as a white solid in 63% yield (>20:1 d.r.); mp=218-220°C; $R_f=0.18$ (petroleum/ethyl acetate 2:1 v/v); ^1H NMR (400 MHz, CDCl_3): δ 7.24-7.13 (m, 6H), 6.69 (d, $J=9.6$ Hz, 1H), 5.61 (d, $J=8.8$ Hz, 1H), 5.43-5.36 (m, 1H), 4.57 (app $J=10$ Hz, 1H), 3.90-3.85 (m, 1H), 3.20-3.13 (m, 1H), 3.09-3.02 (m, 1H), 2.86-2.82 (m, 1H), 2.76-2.70 (m, 1H), 2.01 (s, 3H), 1.71 (m, 1H); ^{13}C NMR (400 MHz, CDCl_3): δ 169.8, 145.2, 137.1, 134.8, 131.3, 129.8, 128.6, 126.6, 126.3, 125.9, 125.6, 114.3, 109.2, 55.2, 46.1, 44.3, 35.8, 28.7, 23.4; IR

(KBr, cm^{-1}): 3430, 2920, 2372, 1636, 1545, 1488, 1371, 1282, 1229, 721; HRMS (ESI) for $\text{C}_{19}\text{H}_{20}\text{BrN}_2\text{O}_1$ $[\text{M}+\text{H}]^+$ calcd. 370.0681, found 370.0679.

4. References:

- (1) X.-Z. Shu, Y. F. Yang, X. F. Xia, K. G. Ji, X.-Y. Liu and Y. M. Liang, *Org. Biomol. Chem.*, 2010, **8**, 4077.
- (2) J. B. Feltenberger, R. Hayashi, Y. Tang, E. S. C. Babiash and R. P. Hsung, *Org. Lett.*, 2009, **11**, 3666.

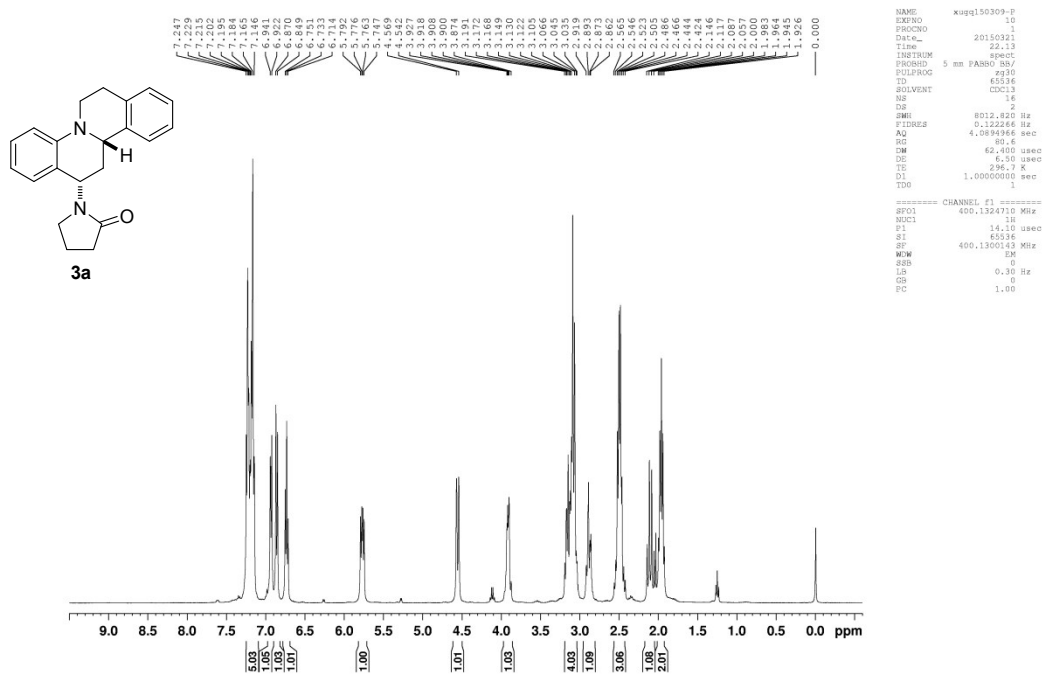
5. X-Ray Crystallographic Data:



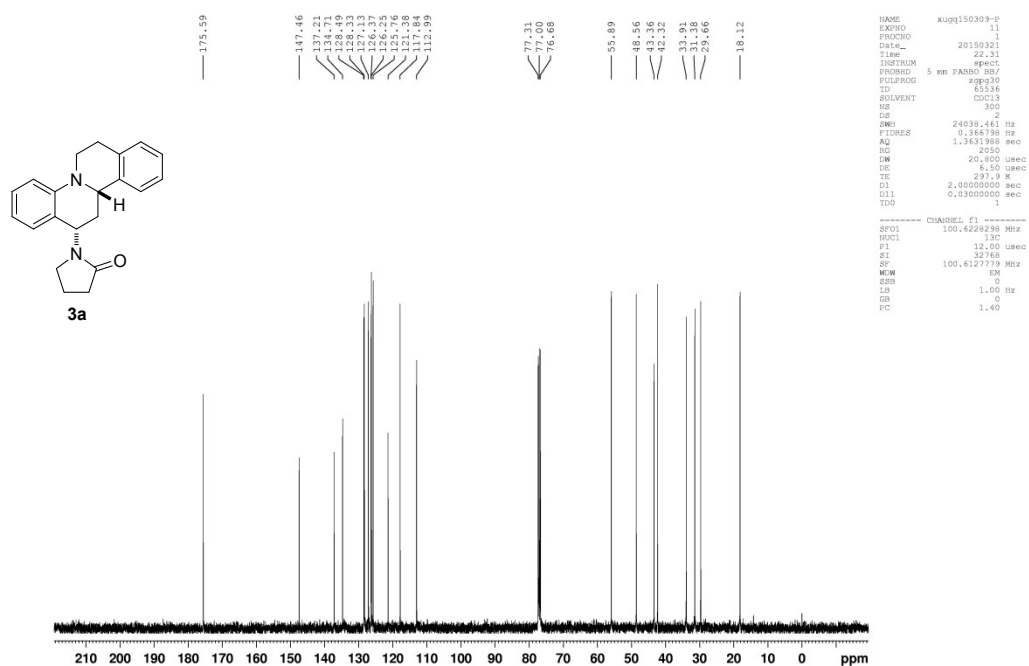
Bond precision:	C-C = 0.0107 Å	Wavelength=0.71073	
Cell:	a=11.3148(10)	b=7.1192(5)	c=21.3182(15)
	alpha=90	beta=93.830(8)	gamma=90
Temperature:	293 K		
	Calculated	Reported	
Volume	1713.4(2)	1713.4(2)	
Space group	P 21/n	P 1 21/n 1	
Hall group	-P 2yn	-P 2yn	
Moiety formula	C ₂₀ H ₁₈ Br N ₂ O ₂	C ₂₀ H ₁₈ Br N ₂ O ₂	
Sum formula	C ₂₀ H ₁₈ Br N ₂ O ₂	C ₂₀ H ₁₈ Br N ₂ O ₂	
Mr	398.26	398.27	
D _x , g cm ⁻³	1.544	1.544	
Z	4	4	
Mu (mm ⁻¹)	2.415	2.415	
F ₀₀₀	812.0	812.0	
F ₀₀₀ '	811.16		
h,k,lmax	13,8,26	13,8,26	
Nref	3380	3375	
Tmin,Tmax	0.469,0.560	0.630,1.000	
Tmin'	0.433		
Correction method=	# Reported T Limits: Tmin=0.630 Tmax=1.000		
AbsCorr =	MULTI-SCAN		
Data completeness=	0.999	Theta(max)= 26.020	
R(reflections)=	0.0716(1325)	wR2(reflections)= 0.1642(3375)	
S =	1.003	Npar= 226	

6. NMR spectra of compound 3

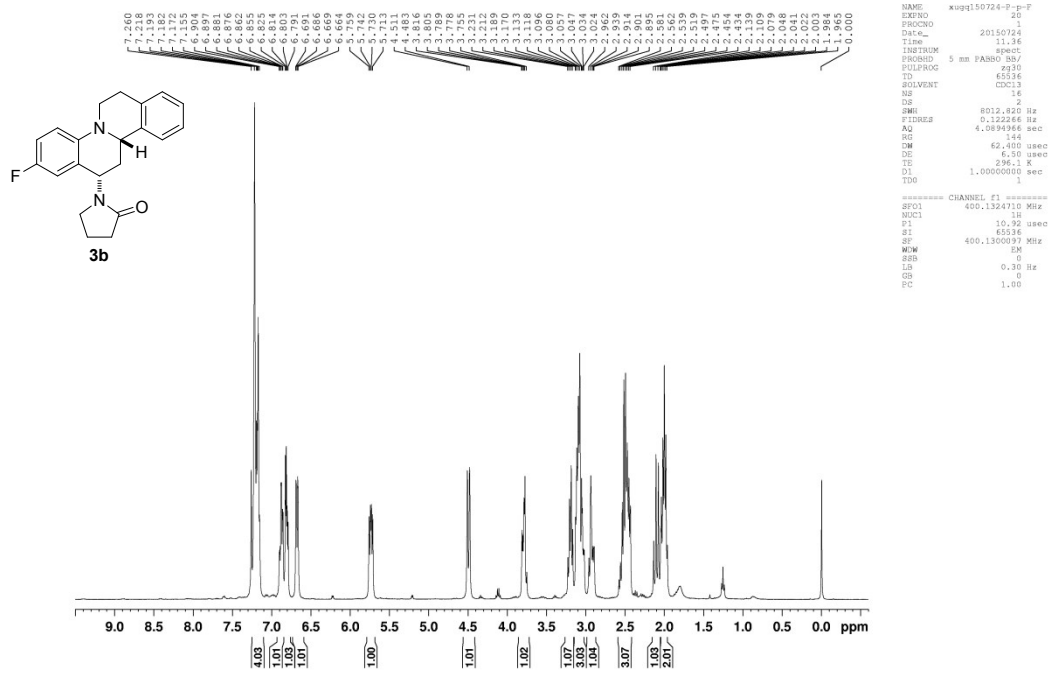
¹H NMR of **3a** in CDCl₃



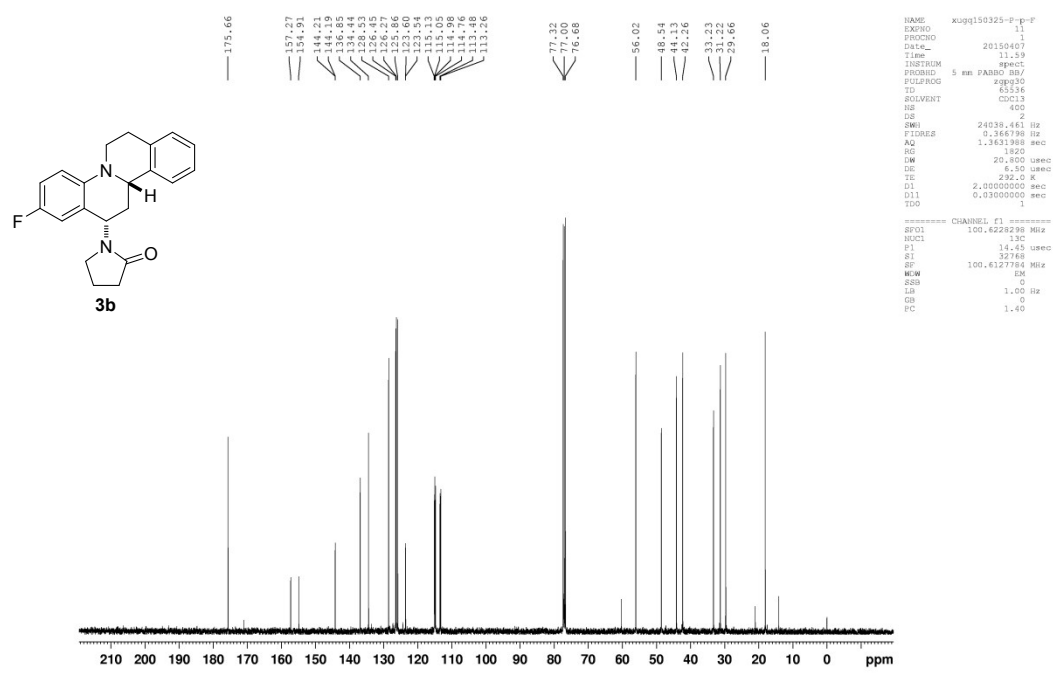
¹³C NMR of **3a** in CDCl₃



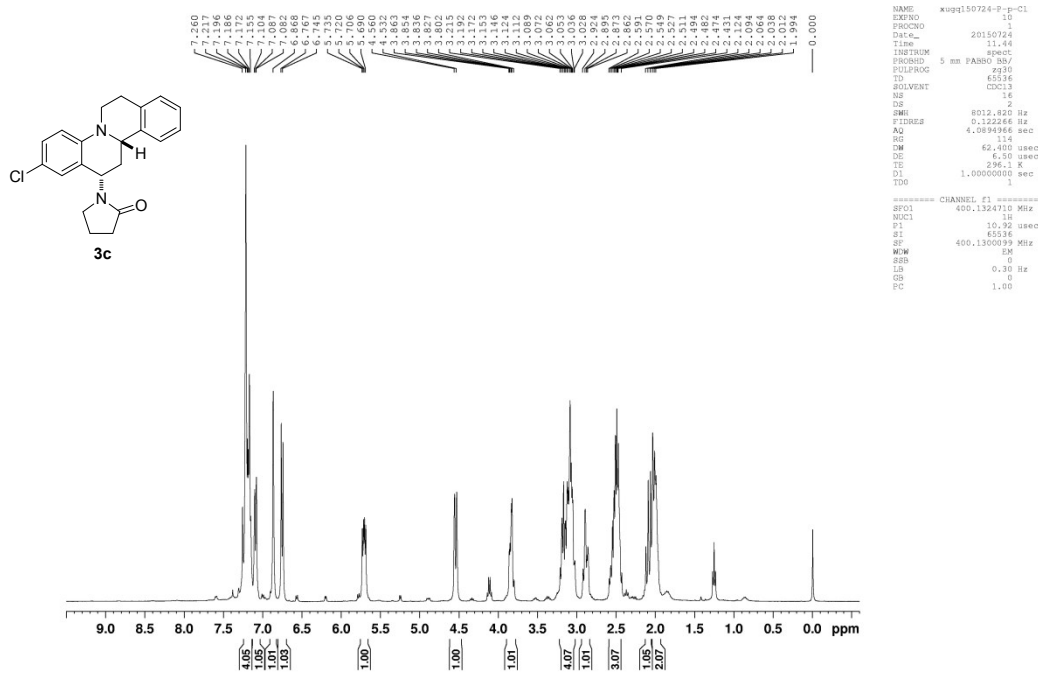
¹H NMR of **3b** in CDCl₃



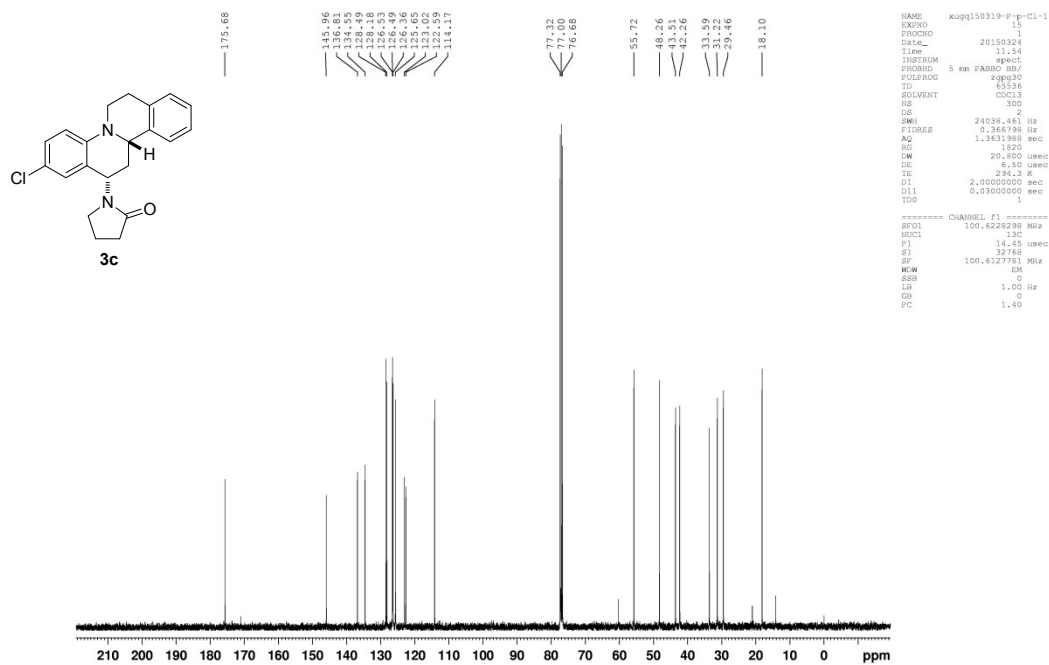
¹³C NMR of **3b** in CDCl₃



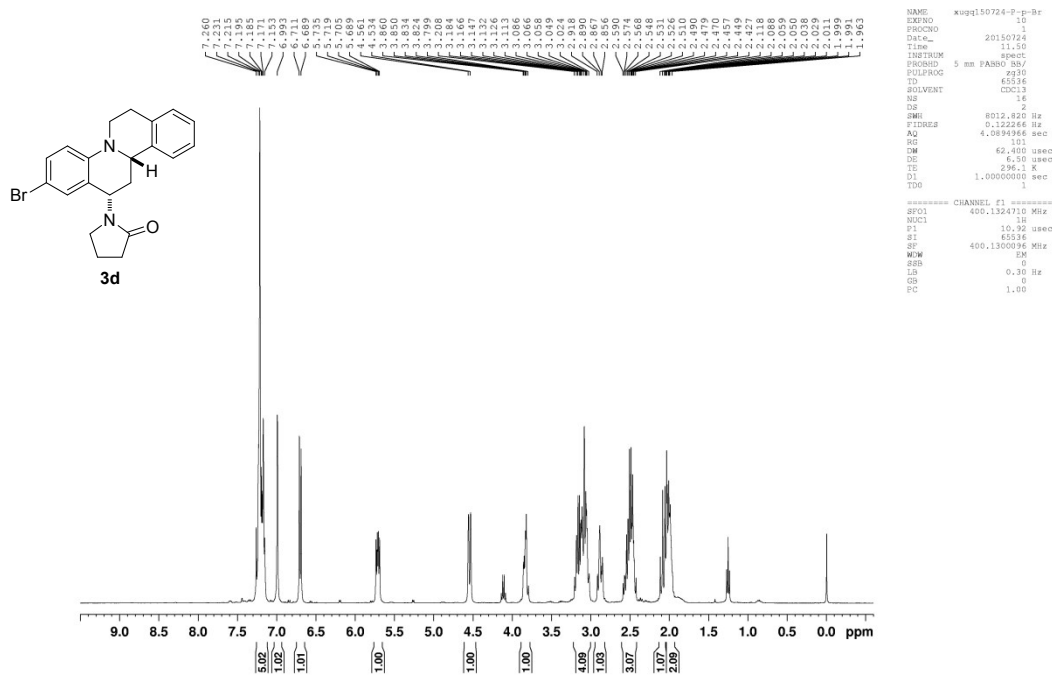
¹H NMR of **3c** in CDCl₃



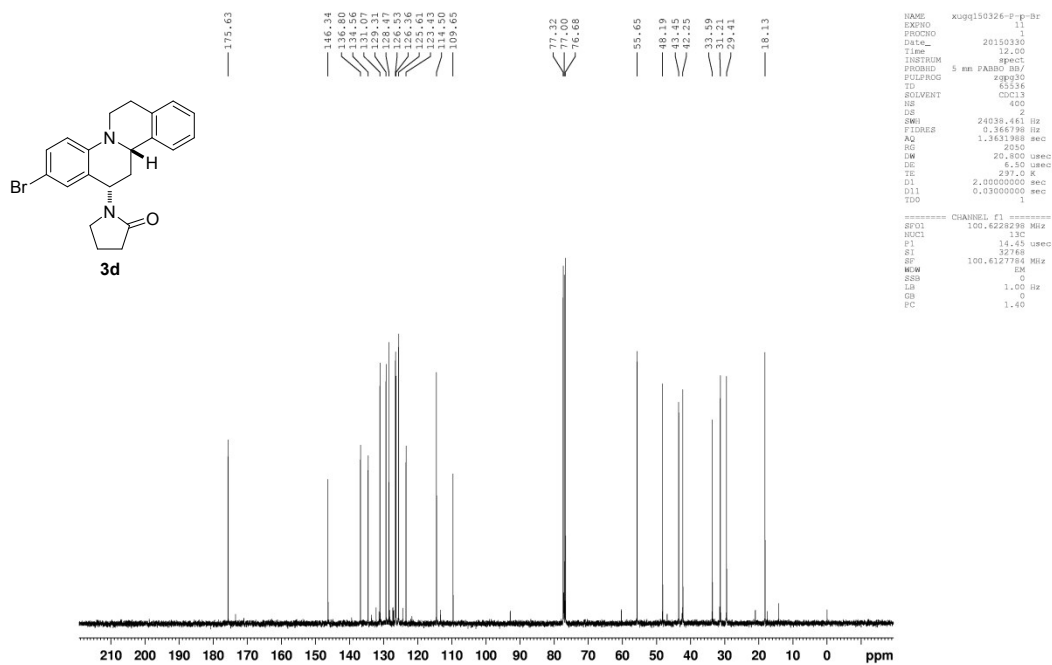
¹³C NMR of **3c** in CDCl₃



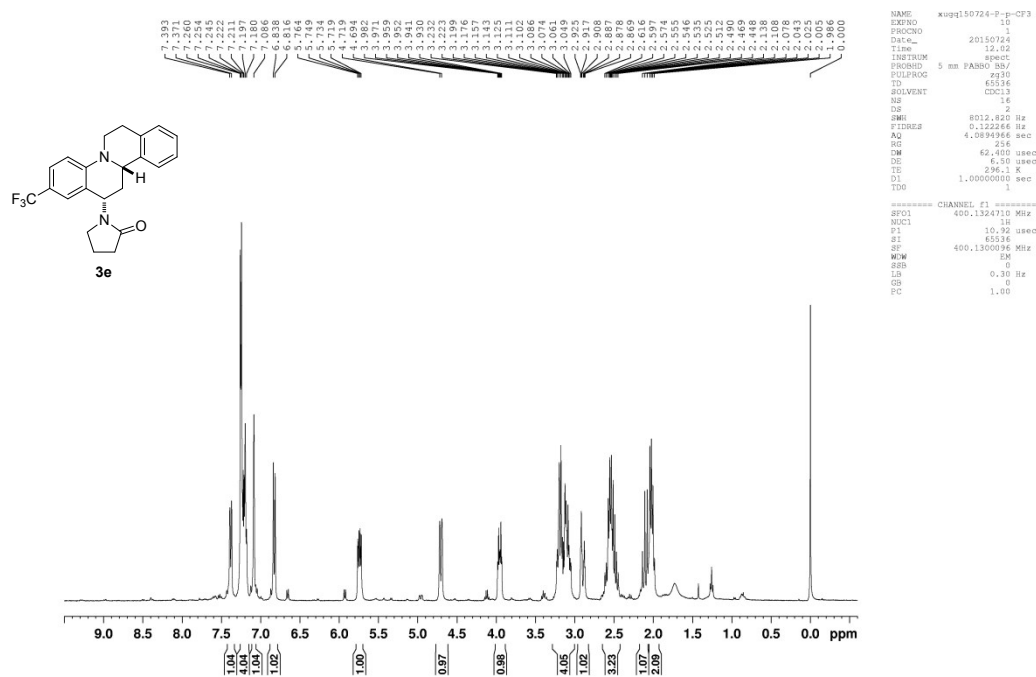
¹H NMR of **3d** in CDCl₃



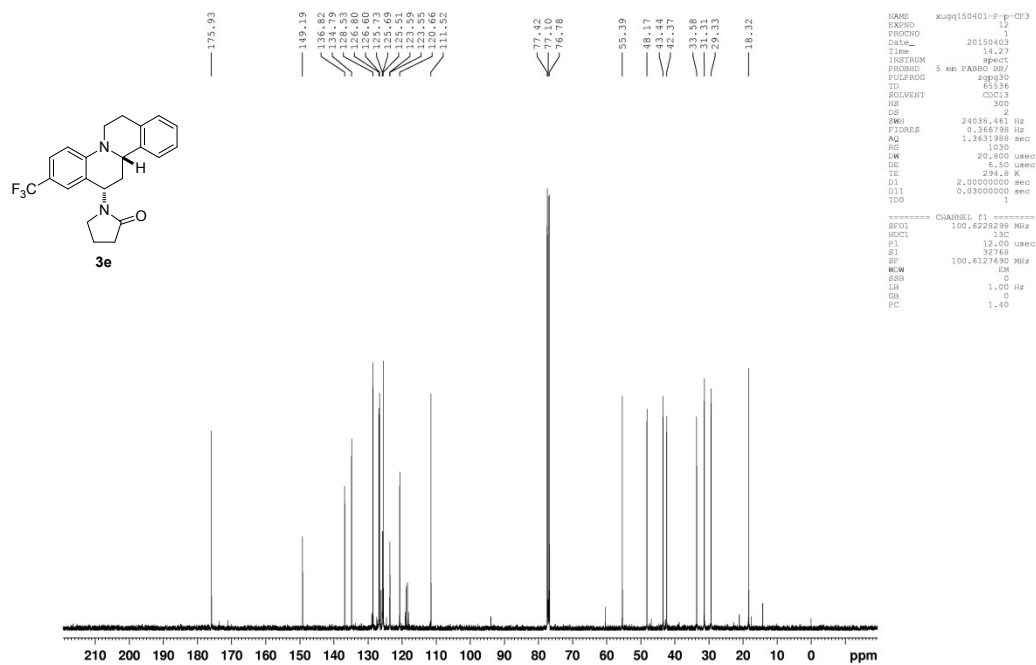
¹³C NMR of **3d** in CDCl₃



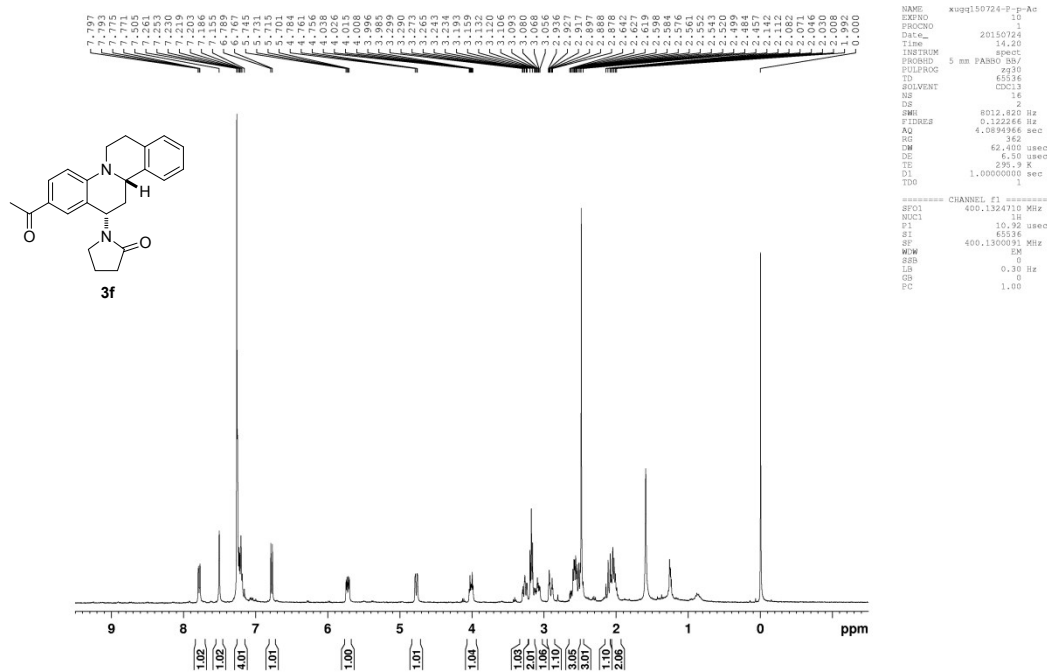
¹H NMR of **3e** in CDCl₃



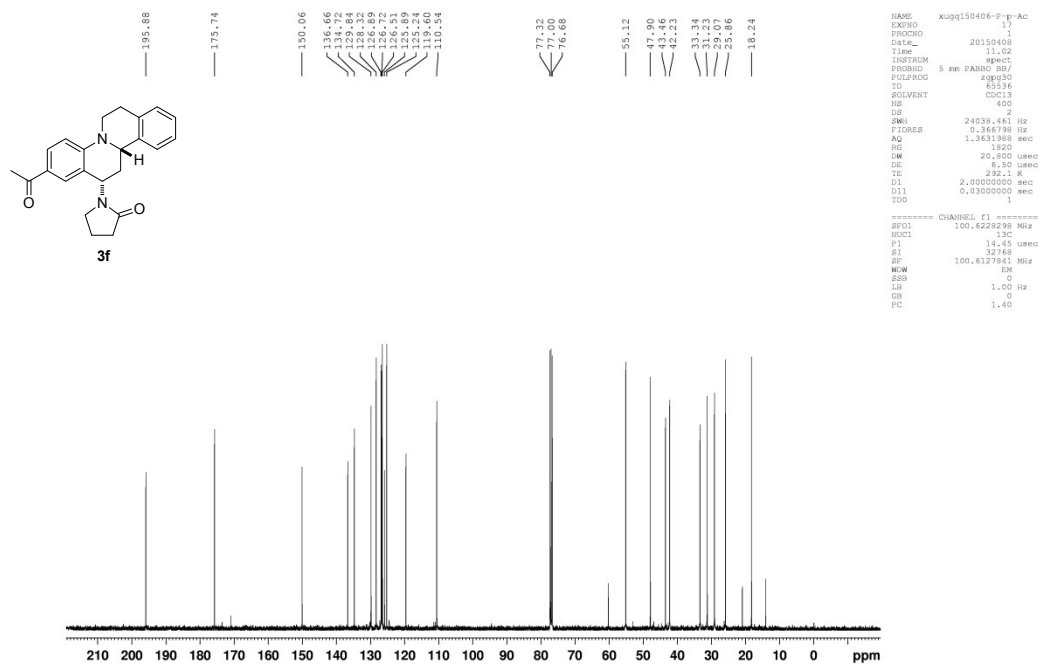
¹³C NMR of **3e** in CDCl₃



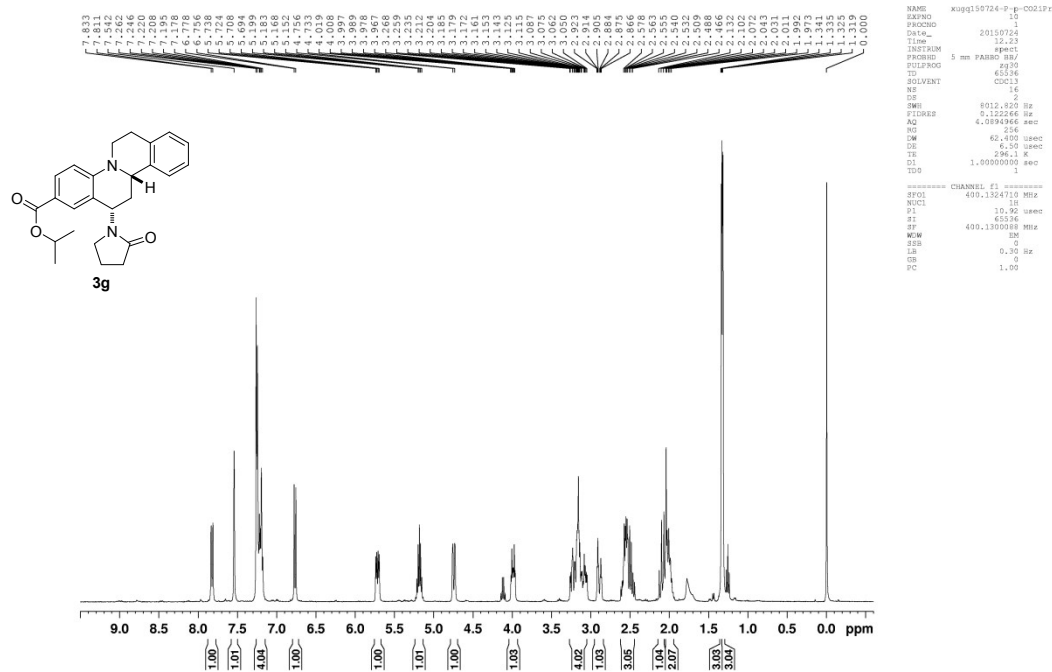
¹H NMR of **3f** in CDCl₃



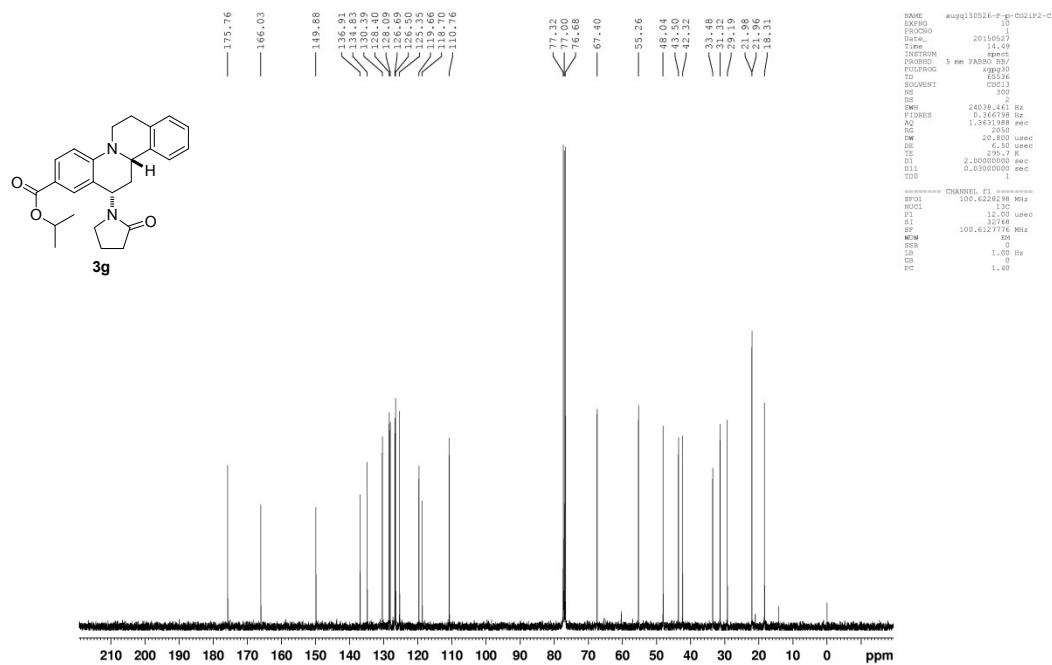
¹³C NMR of **3f** in CDCl₃



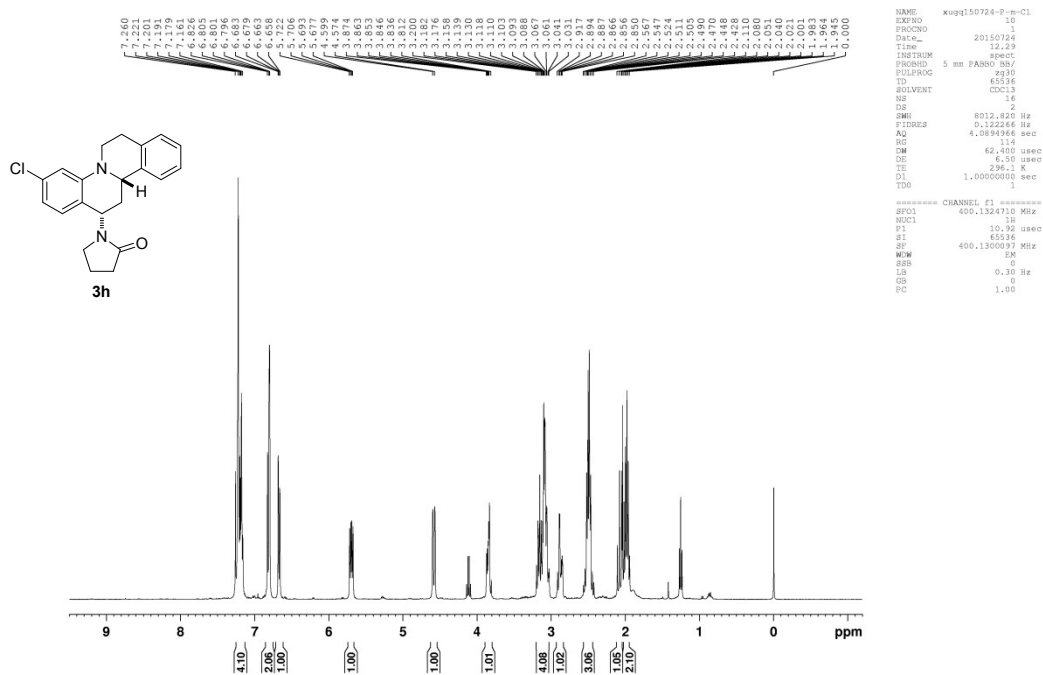
¹H NMR of **3g** in CDCl₃



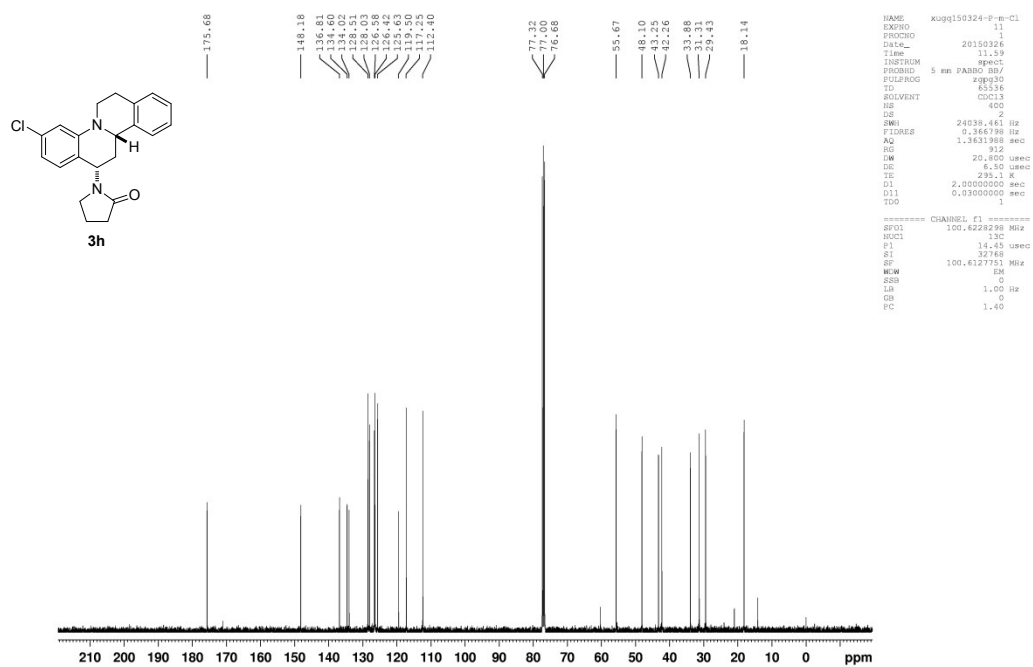
¹³C NMR of **3g** in CDCl₃



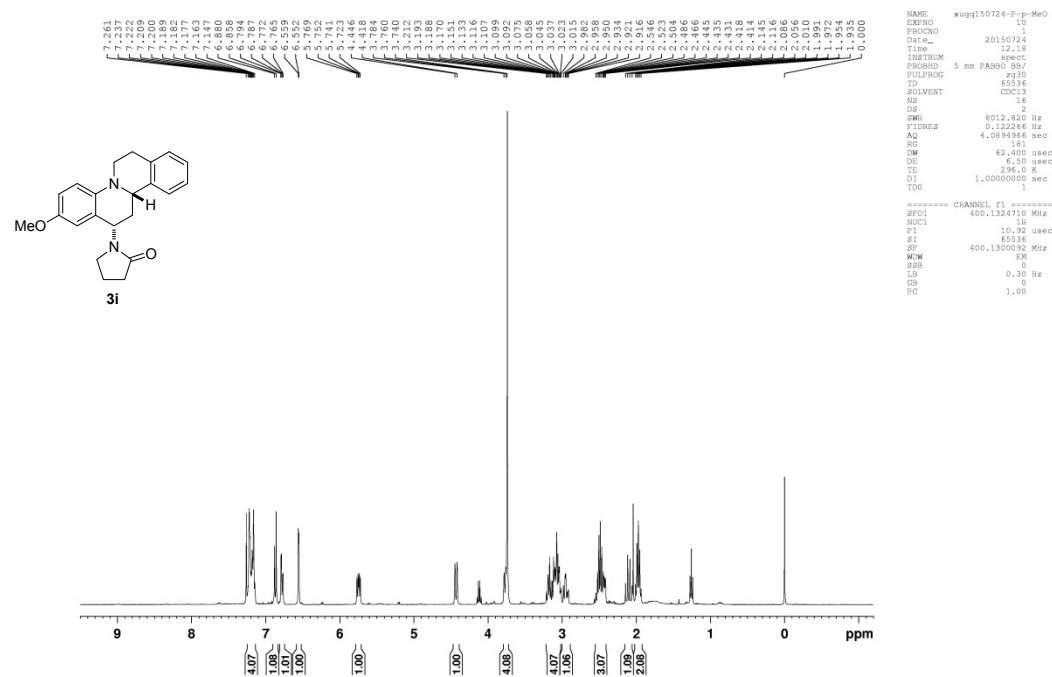
¹H NMR of **3h** in CDCl₃



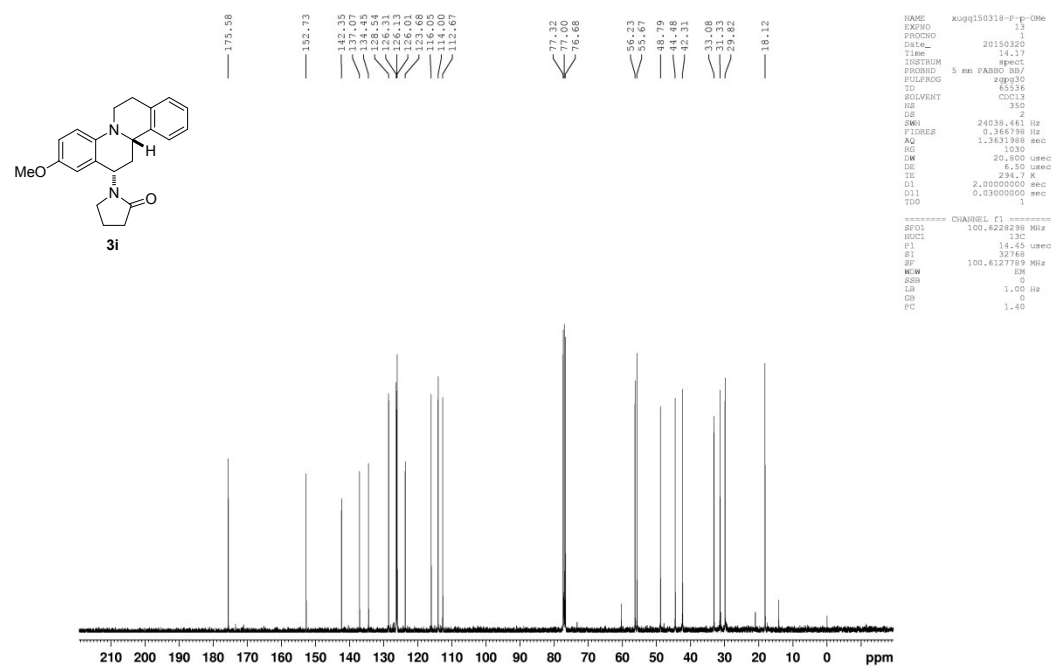
¹³C NMR of **3h** in CDCl₃



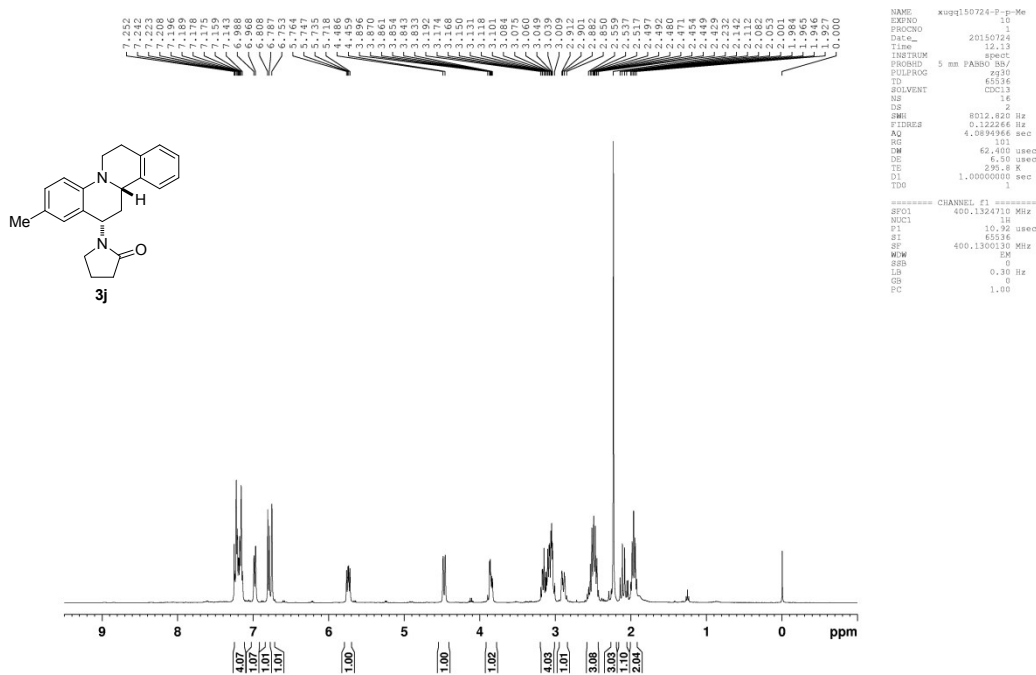
¹H NMR of **3i** in CDCl₃



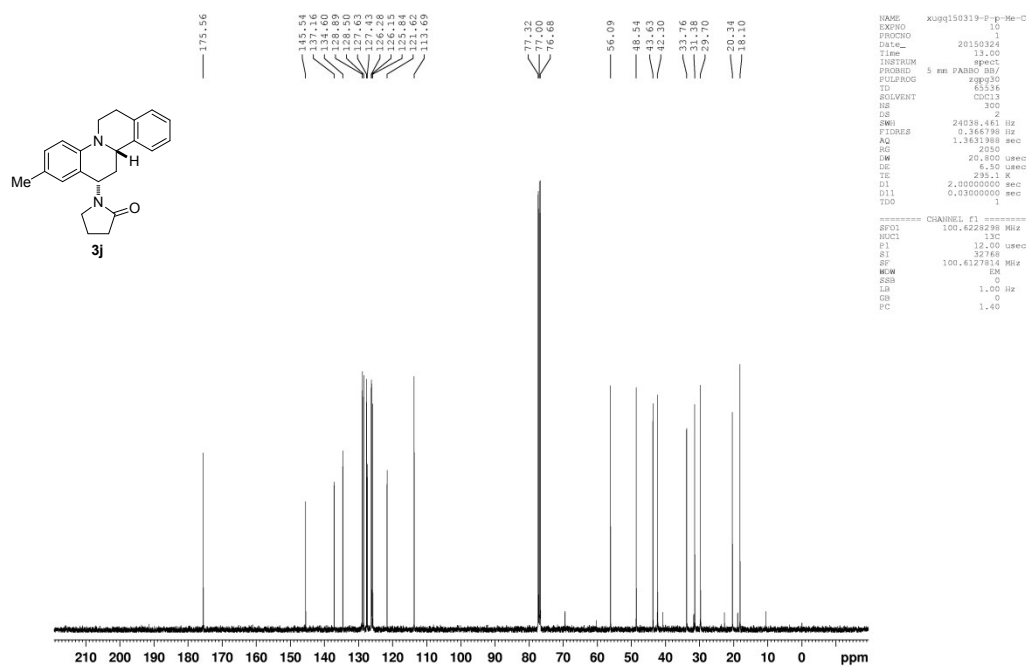
¹³C NMR of **3i** in CDCl₃



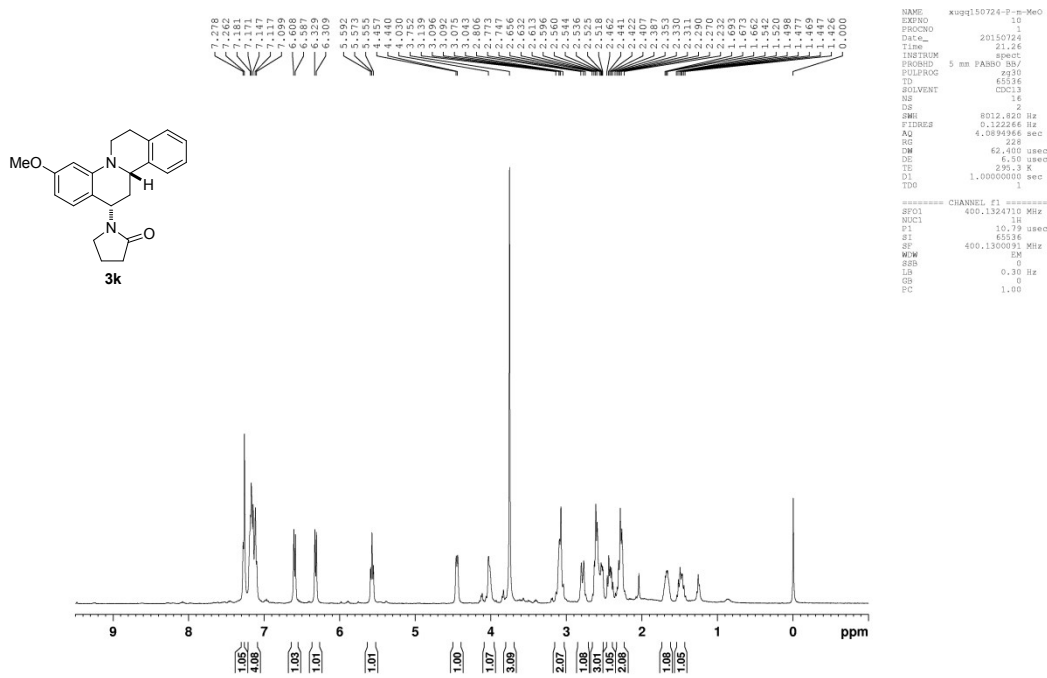
¹H NMR of **3j** in CDCl₃



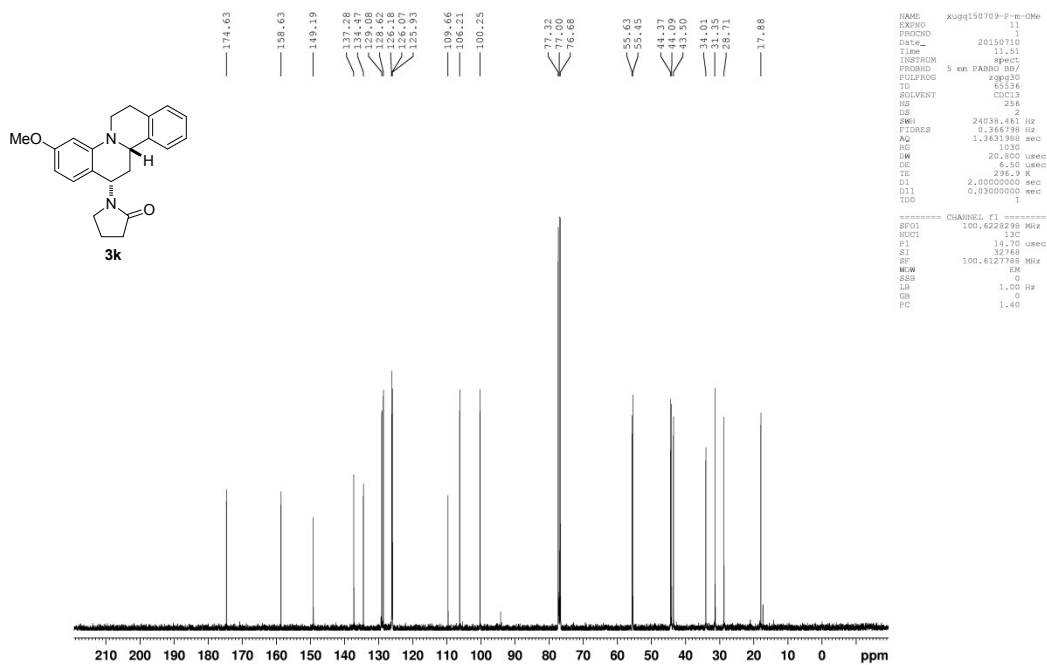
¹³C NMR of **3j** in CDCl₃



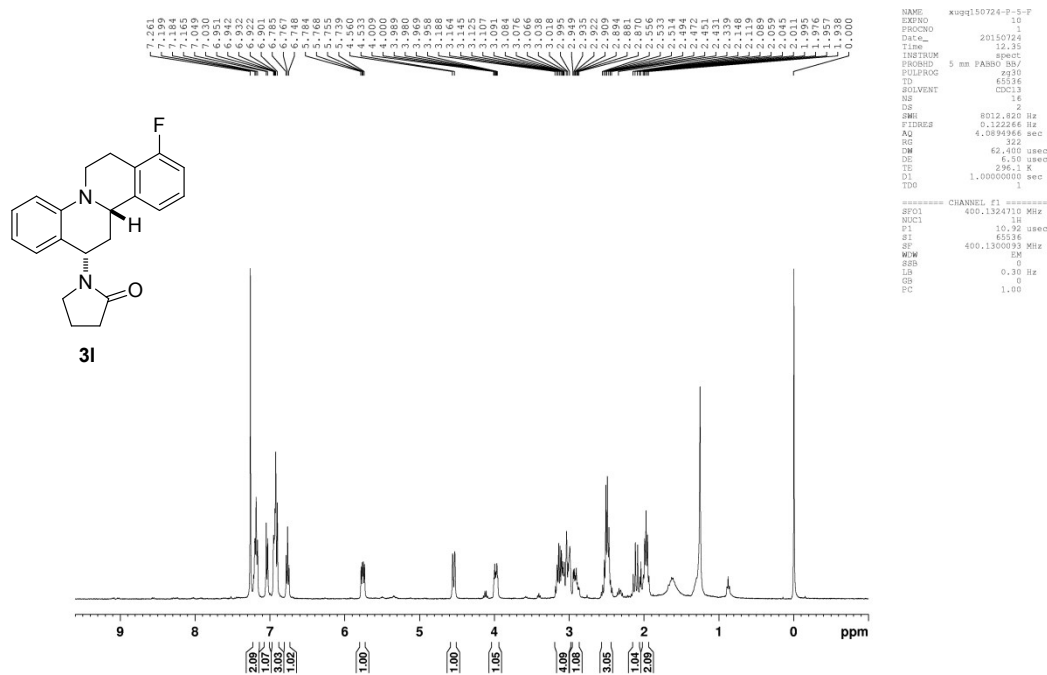
¹H NMR of **3k** in CDCl₃



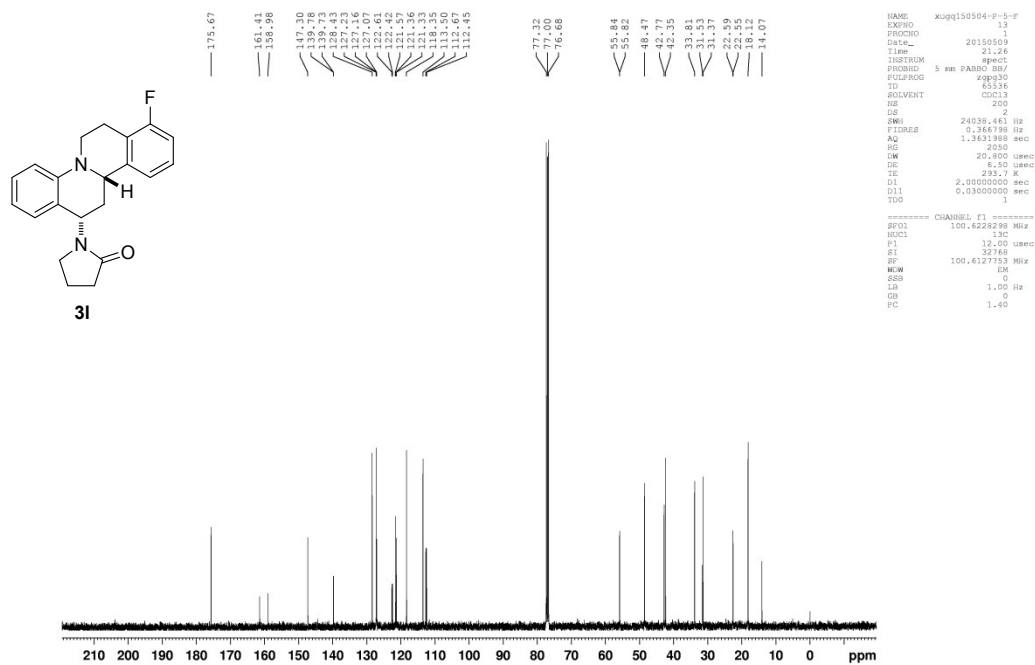
¹³C NMR of **3k** in CDCl₃



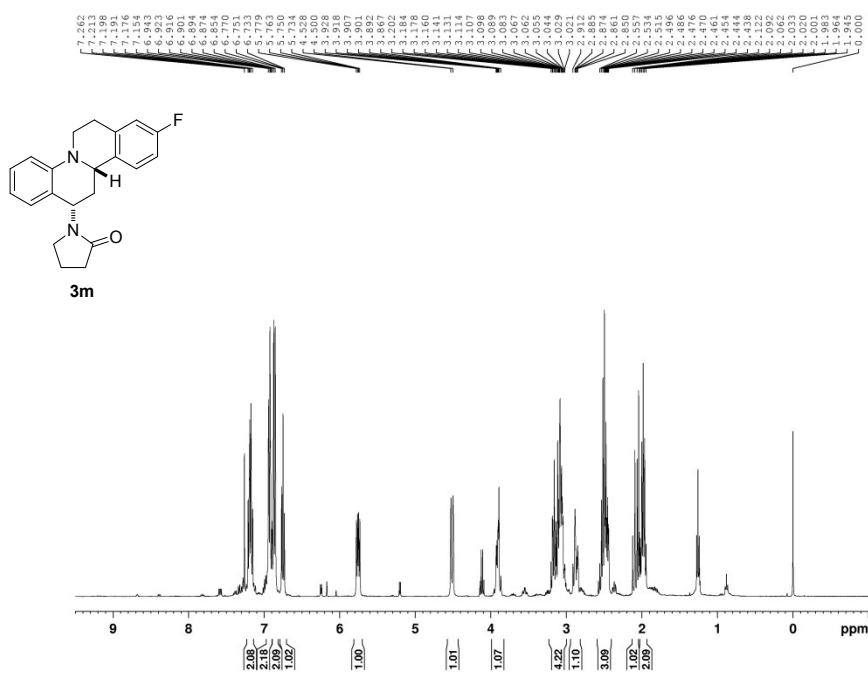
¹H NMR of **31** in CDCl₃



¹³C NMR of **31** in CDCl₃



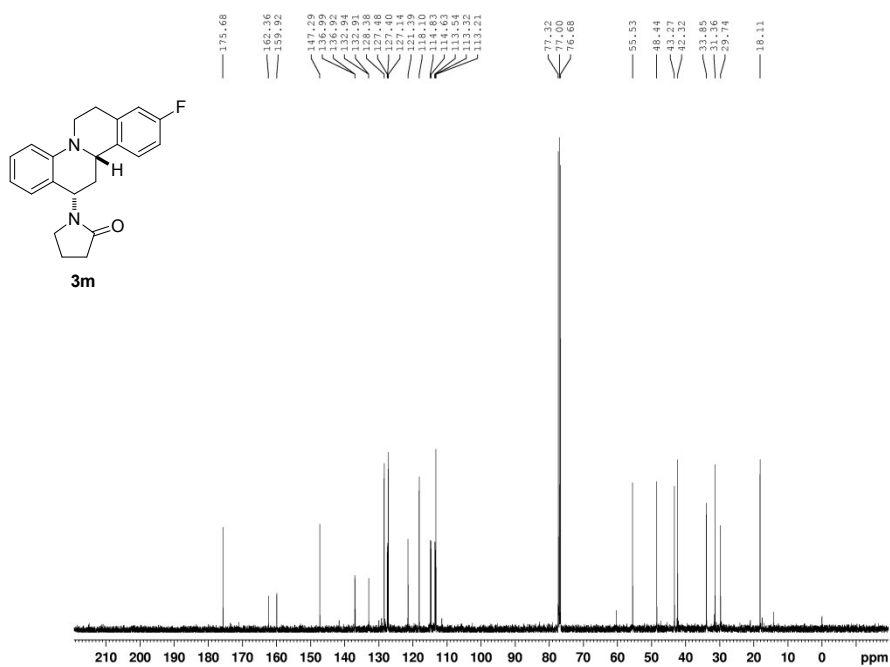
¹H NMR of **3m** in CDCl₃



```

NAME      kugq150504-P-6-F
EXPNO    11
PROCNO   11
Date_    20150509
Time     21.32
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        2
SWH       8012.826 Hz
FIDRES   0.122266 Hz
AQ        4.083986 sec
RG         128
DM        62.000 usec
DE         6.50 usec
TE        293.2 K
D1        1.0000000 sec
TD0       1
===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1      13
P1        10.79 usec
SI        65536
SF        400.1300895 MHz
WWSW     EM
SSB       0
LB        0.30 Hz
GB        0
PC         1.00
    
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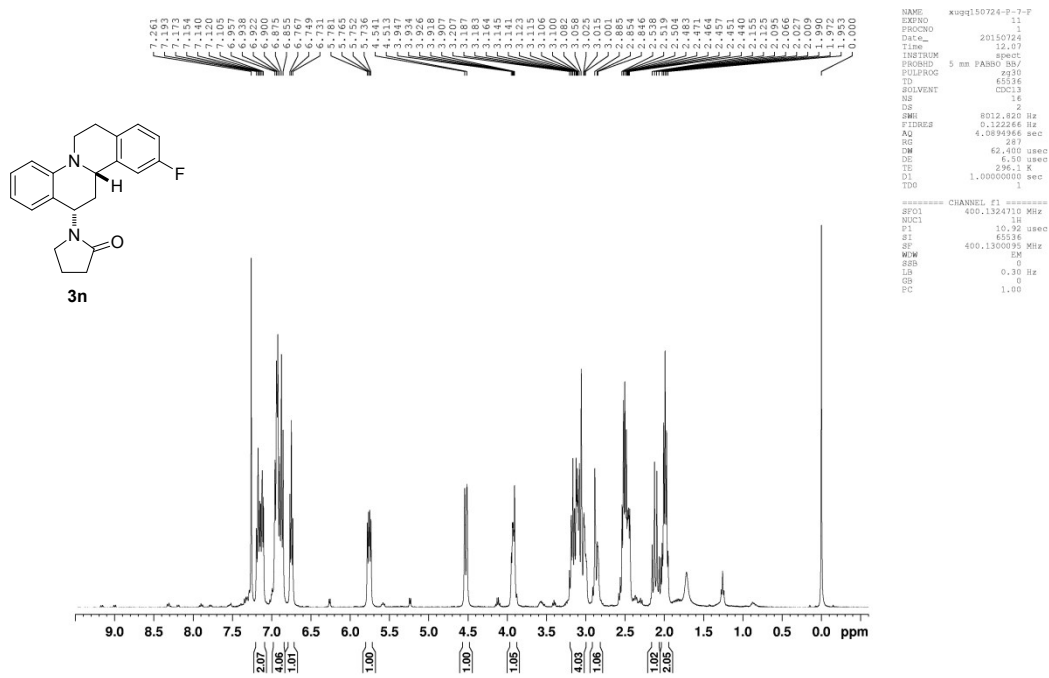
¹³C NMR of **3m** in CDCl₃



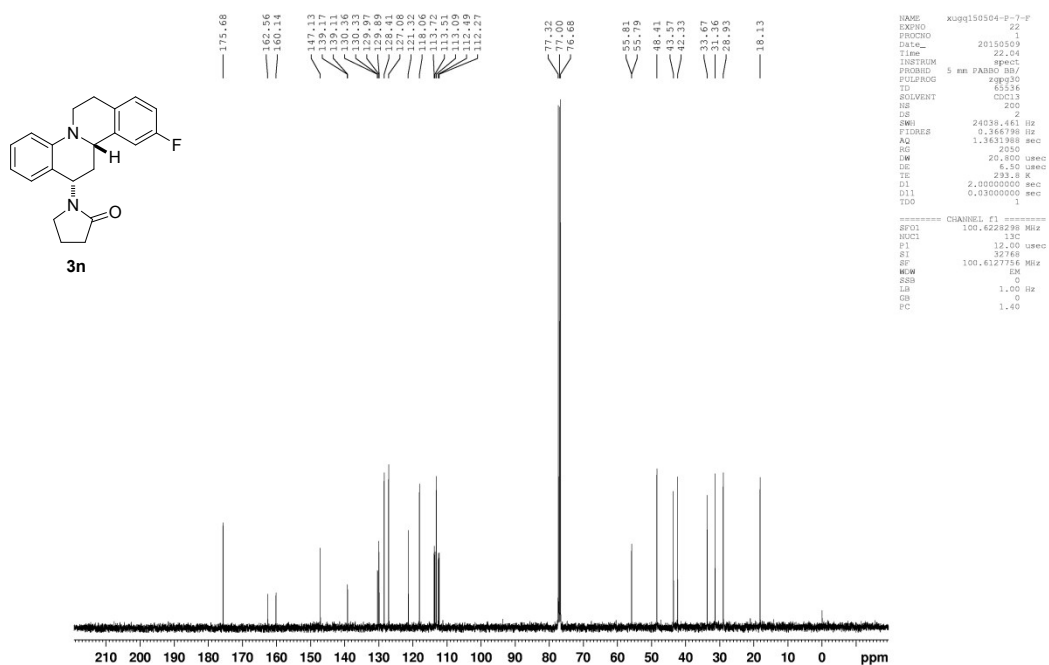
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NAME      kugq150504-P-6-F
EXPNO    11
PROCNO   11
Date_    20150509
Time     21.45
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        2
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.363198 sec
RG         2050
DM        20.800 usec
DE         6.50 usec
TE        293.2 K
D1        2.0000000 sec
D11       0.03000000 sec
TD0       1
===== CHANNEL f1 =====
SFO1     100.6228298 MHz
NUC1      13
P1        12.00 usec
SI        32768
SF        100.6127766 MHz
WWSW     EM
SSB       0
LB        1.00 Hz
GB        0
PC         1.40
    
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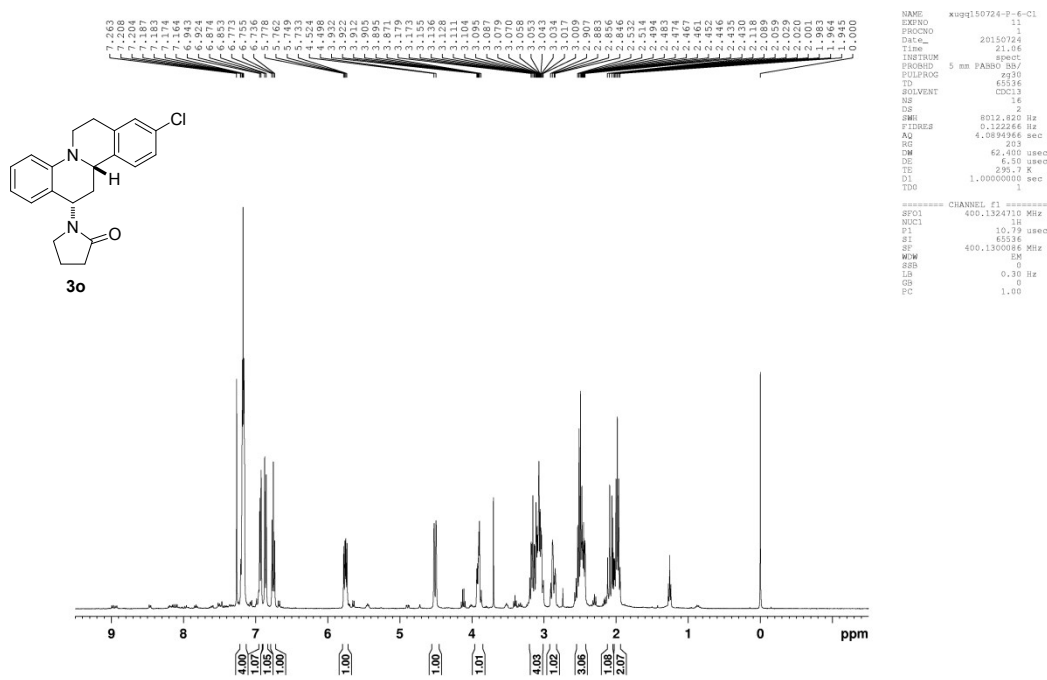
¹H NMR of **3n** in CDCl₃



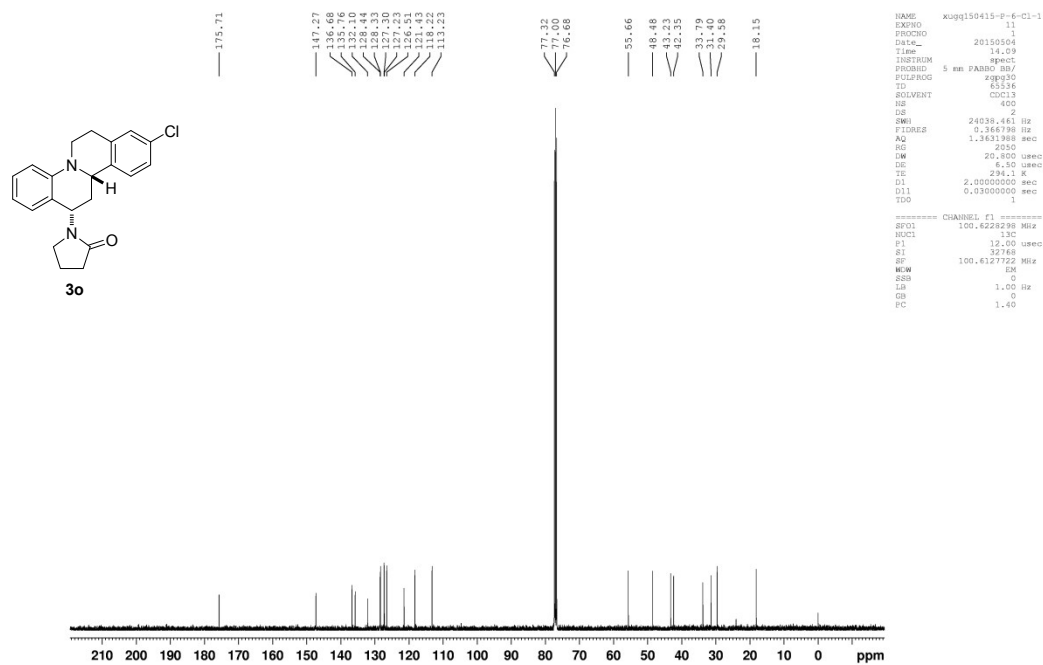
¹³C NMR of **3n** in CDCl₃



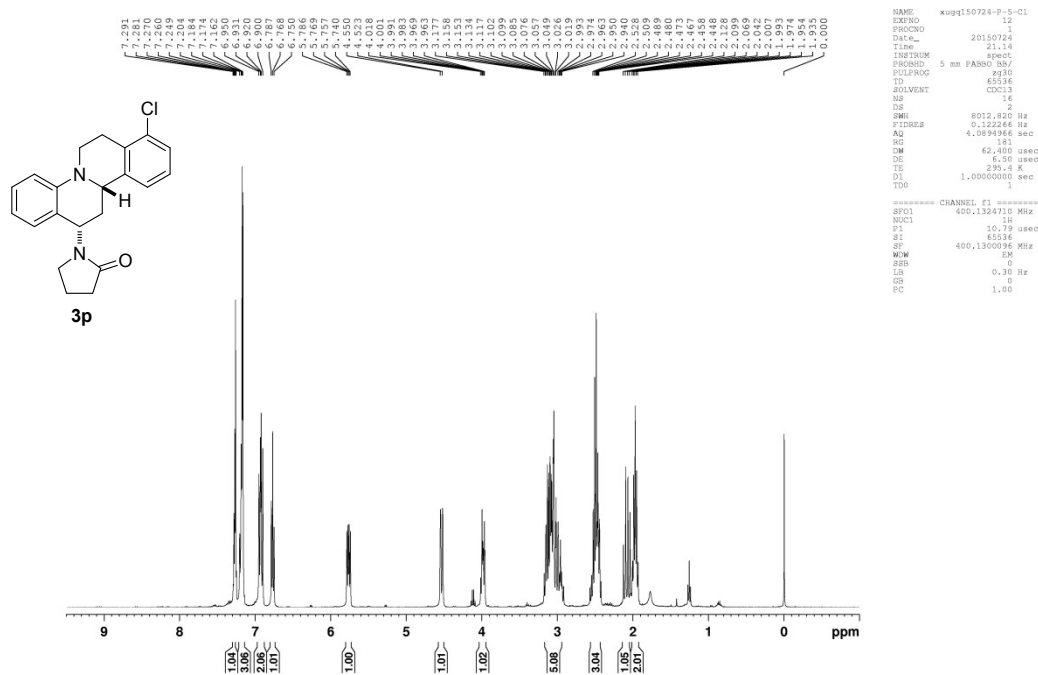
¹H NMR of **3o** in CDCl₃



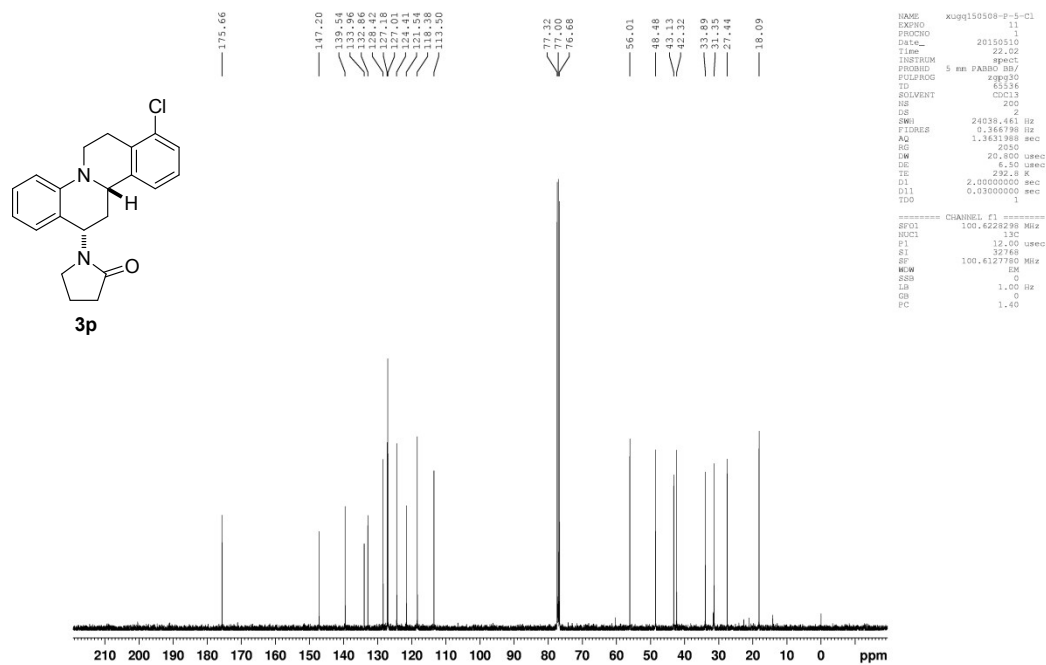
¹³C NMR of **3o** in CDCl₃



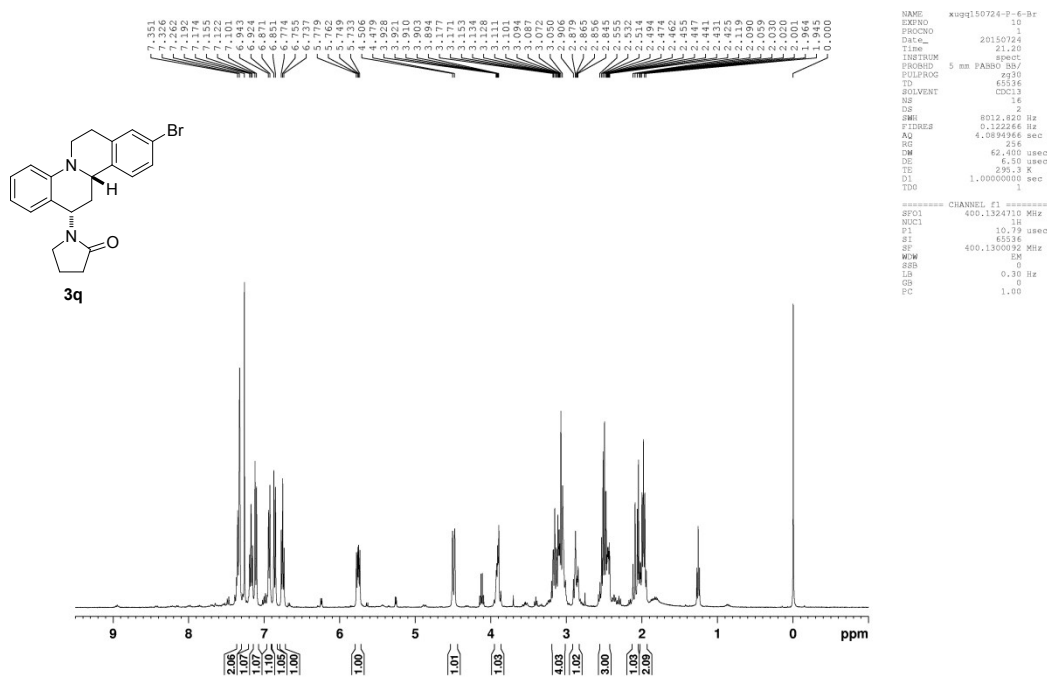
¹H NMR of **3p** in CDCl₃



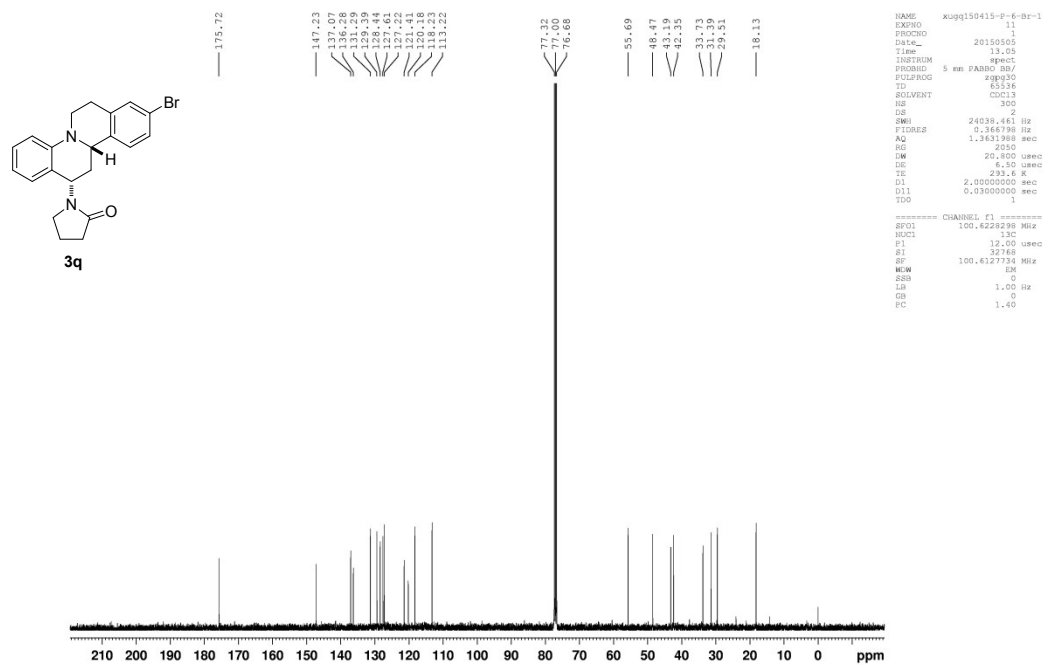
¹³C NMR of **3p** in CDCl₃



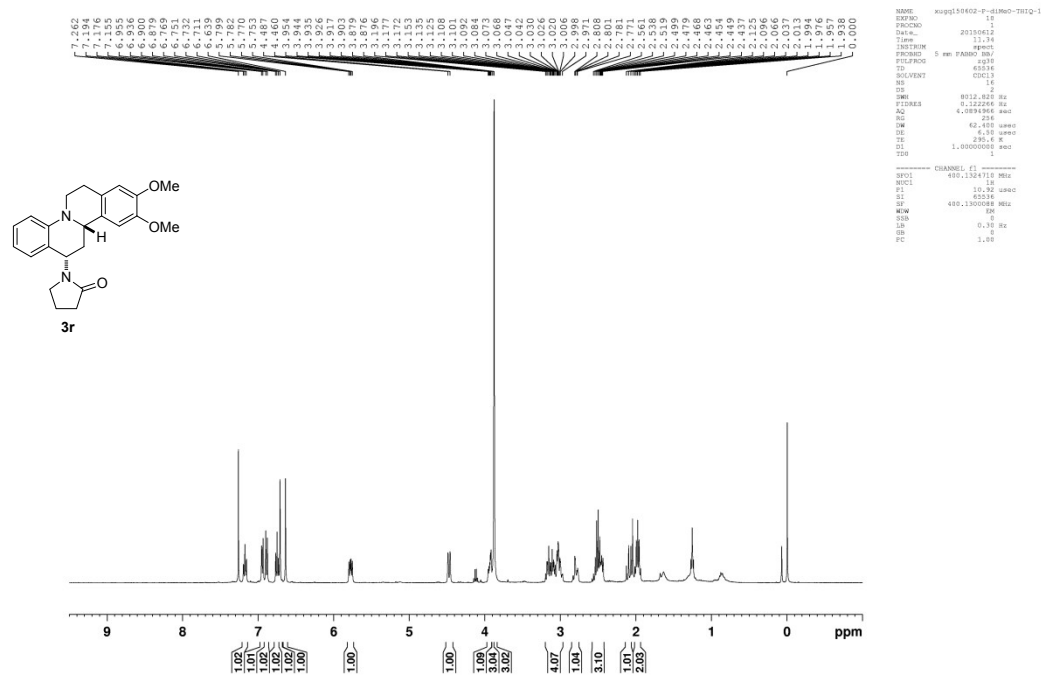
¹H NMR of **3q** in CDCl₃



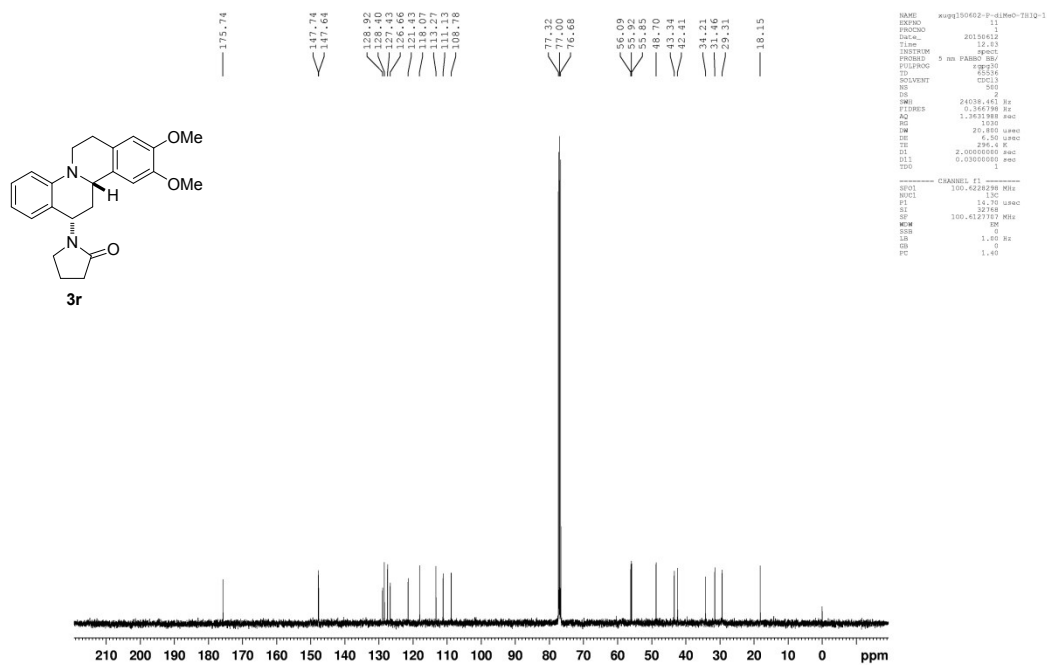
¹³C NMR of **3q** in CDCl₃



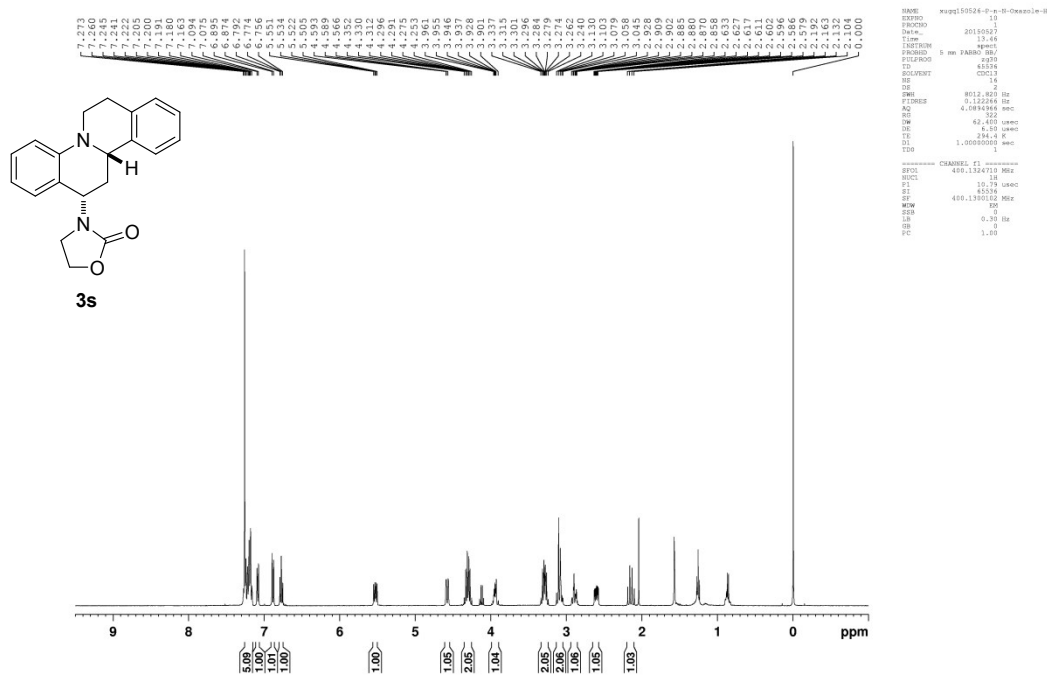
¹H NMR of **3r** in CDCl₃



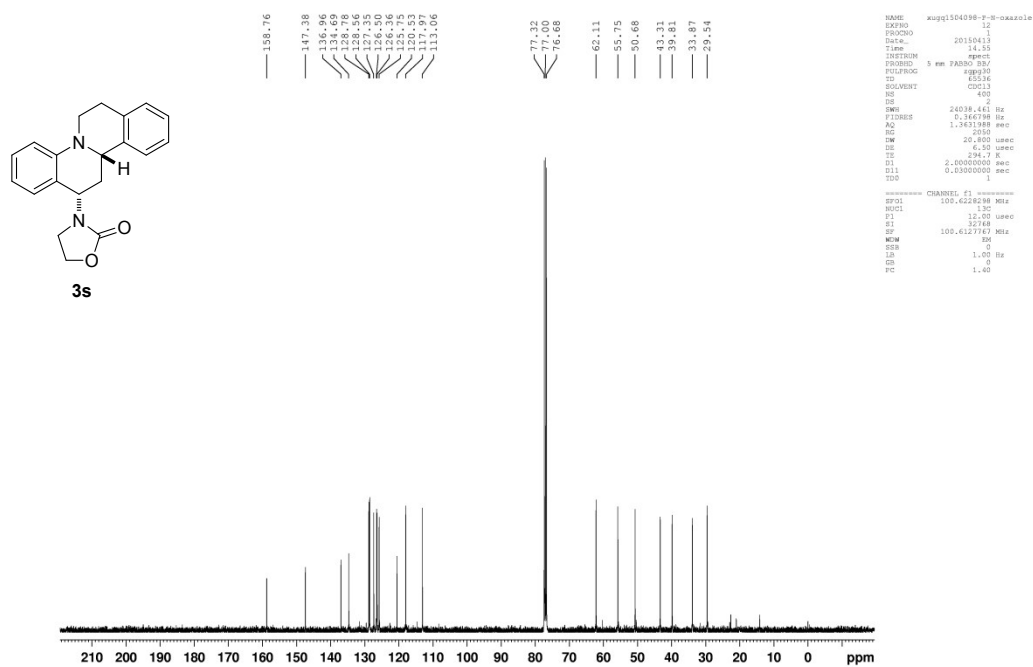
¹³C NMR of **3r** in CDCl₃



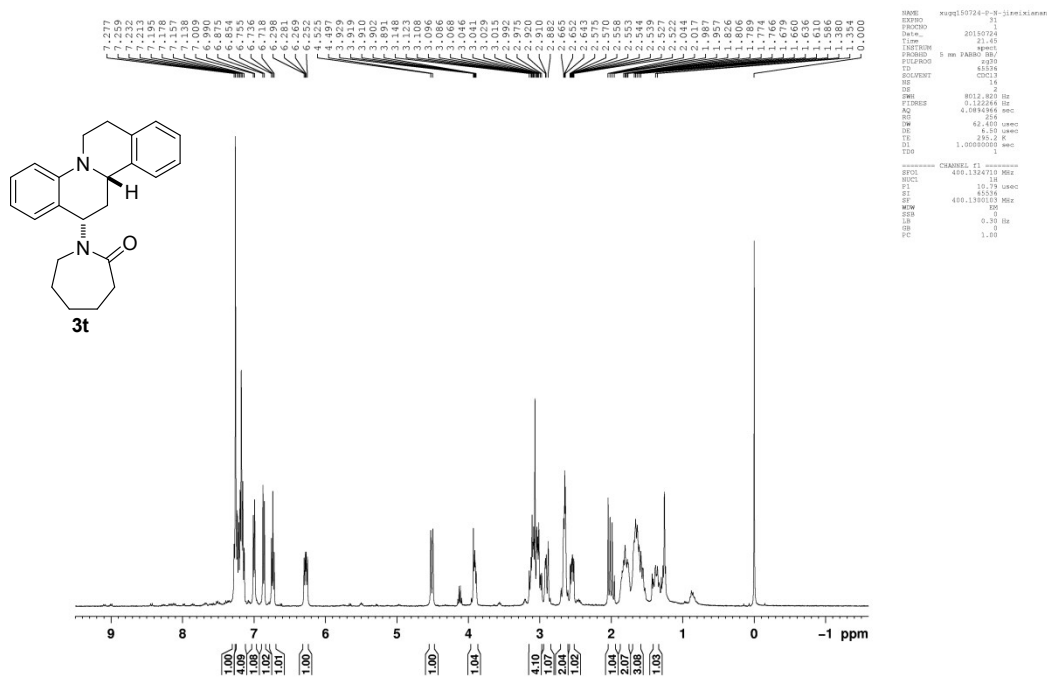
¹H NMR of **3s** in CDCl₃



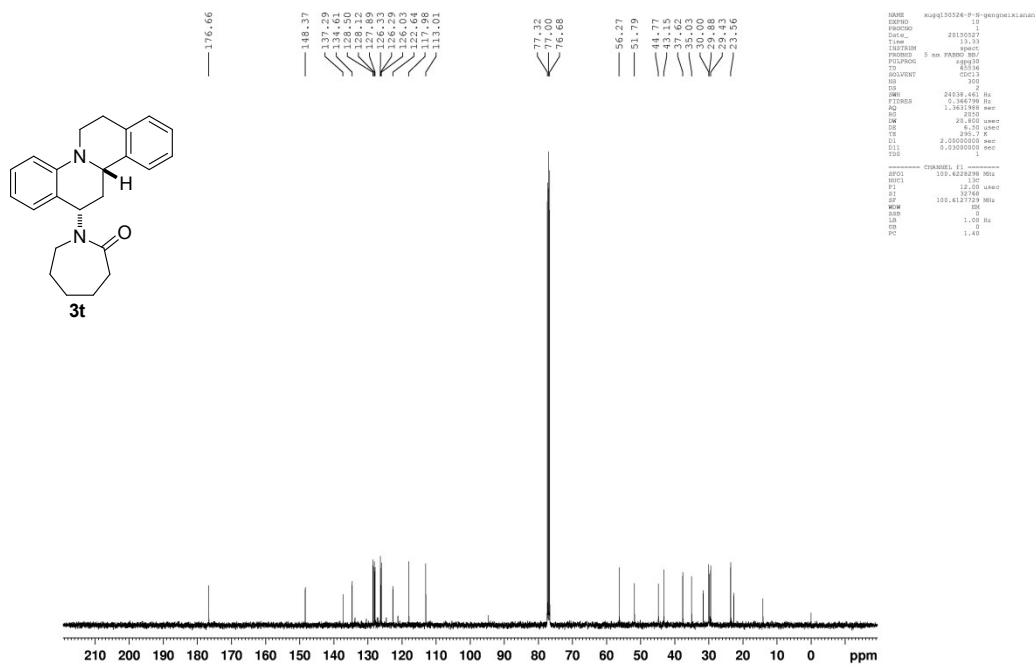
¹³C NMR of **3s** in CDCl₃



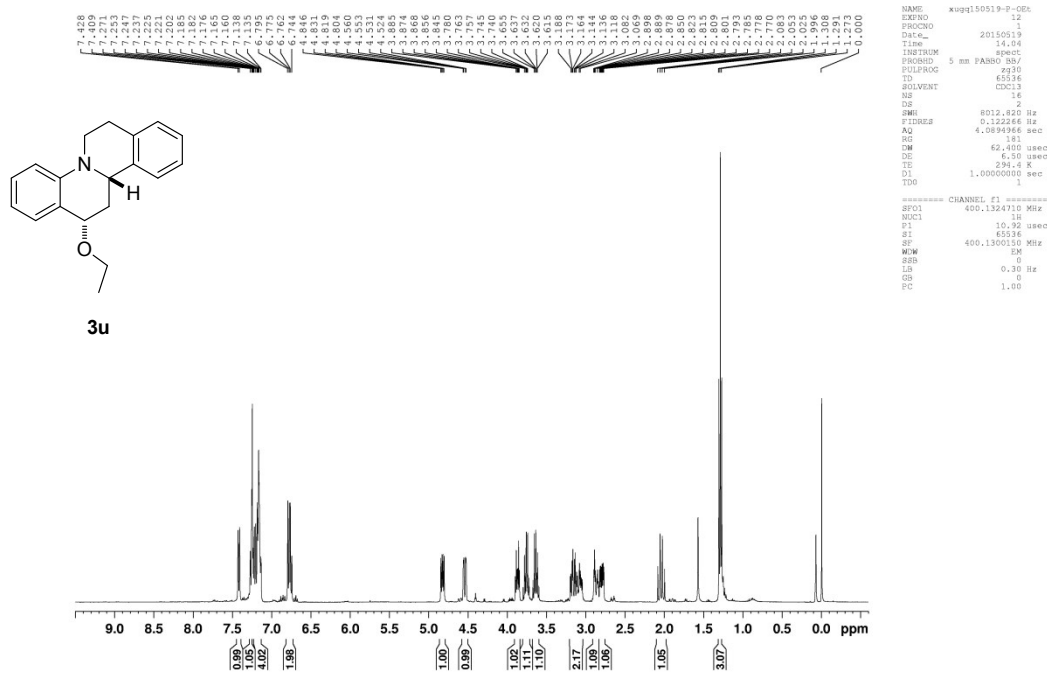
¹H NMR of **3t** in CDCl₃



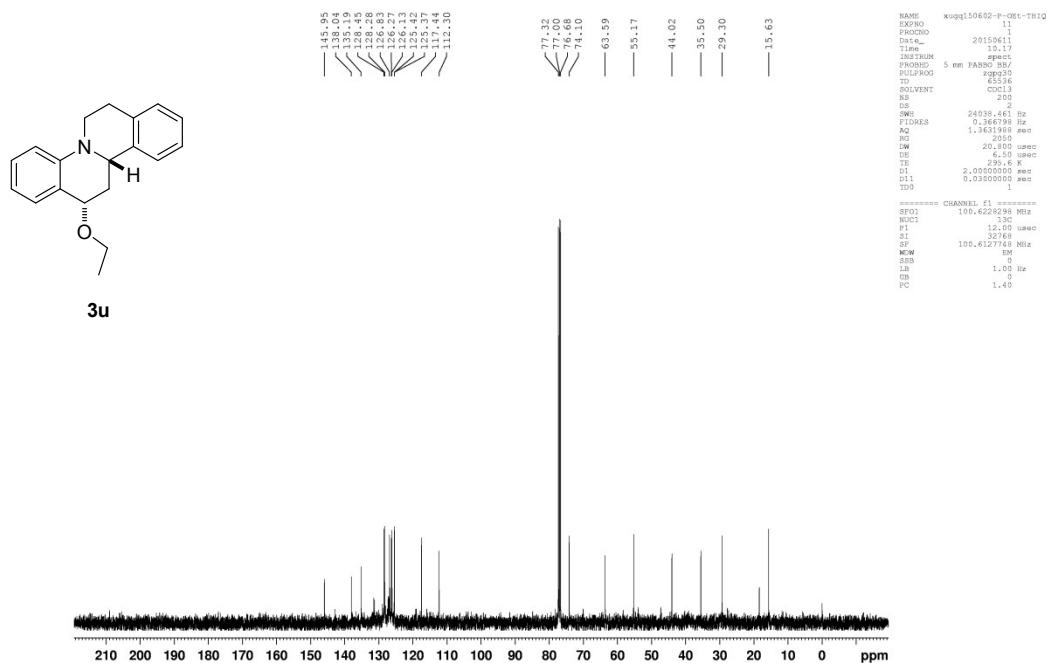
¹³C NMR of **3t** in CDCl₃



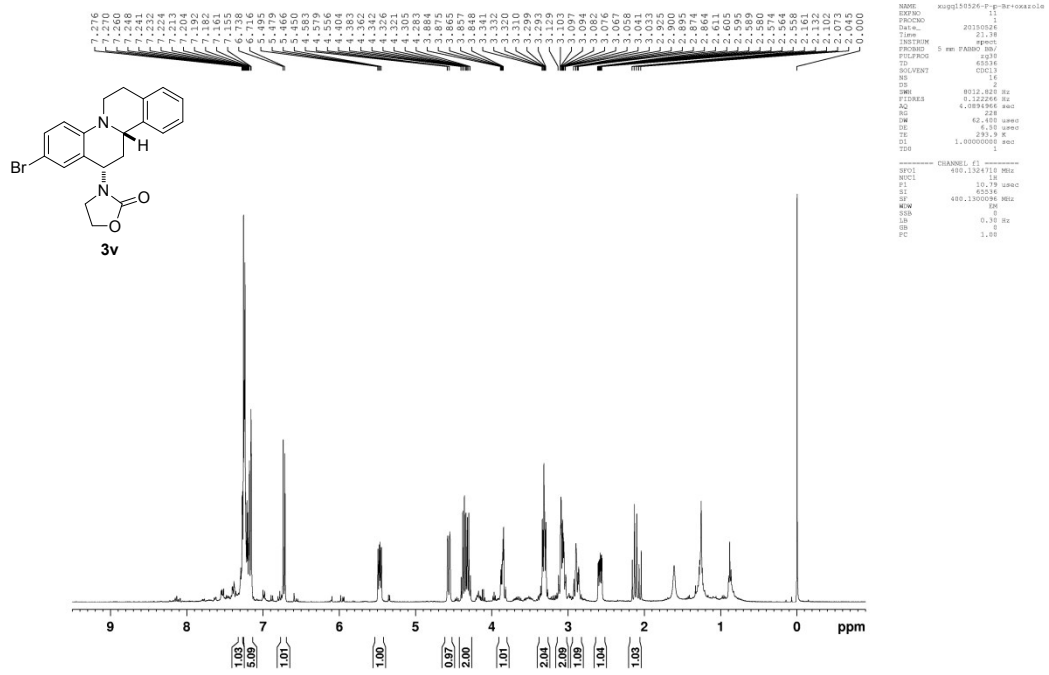
¹H NMR of **3u** in CDCl₃



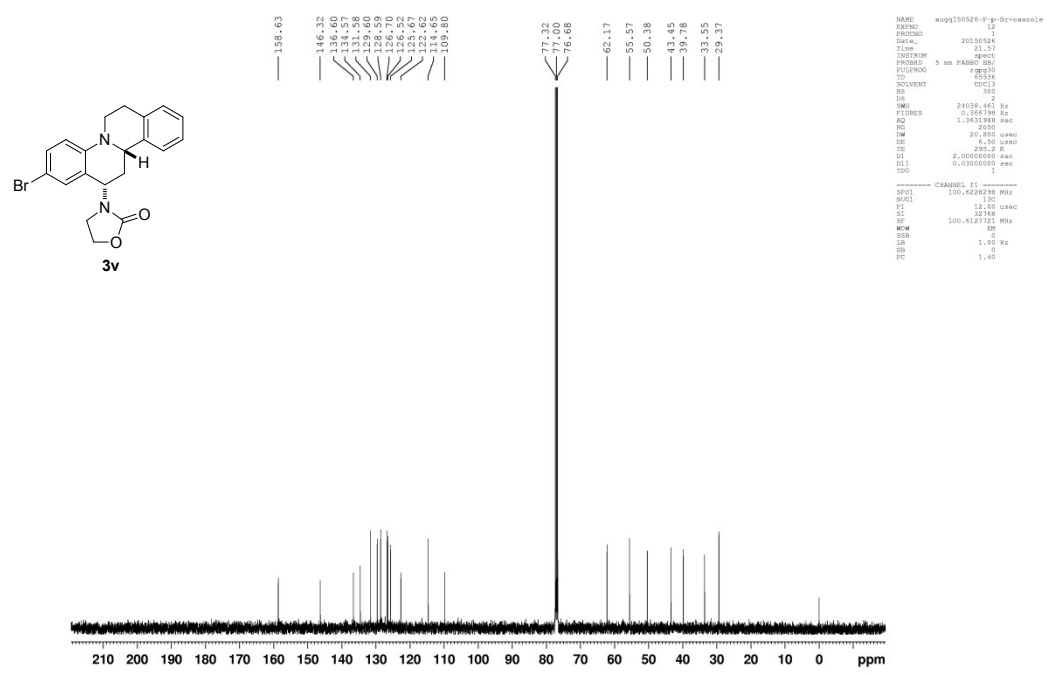
¹³C NMR of **3u** in CDCl₃



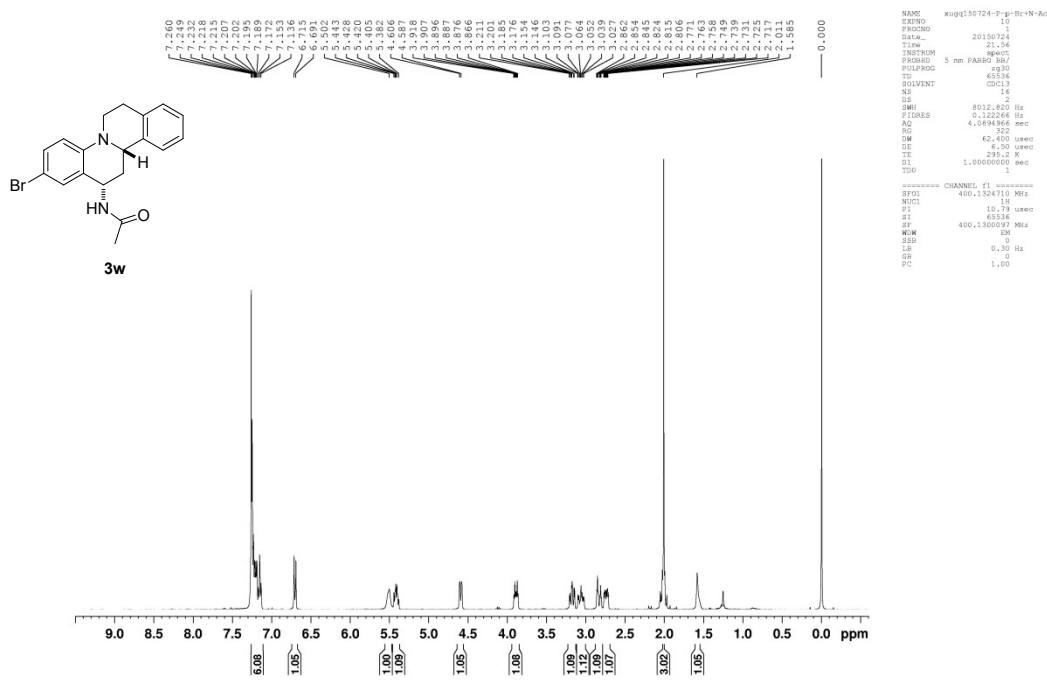
¹H NMR of **3v** in CDCl₃



¹³C NMR of **3v** in CDCl₃



¹H NMR of **3w** in CDCl₃



¹³C NMR of **3w** in CDCl₃

