Electronic Supplementary Material (ESI) for RSC Advances.

Expedient, catalyst-free, three-component synthesis of fused tetrahydropyridines in water

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Supporting Information

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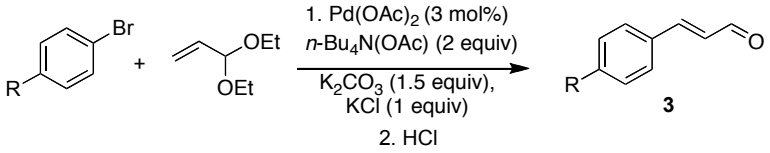
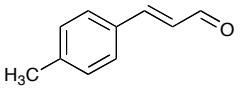
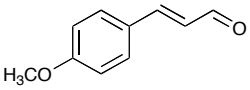
1. General procedure for the synthesis of substituted cinnamaldehydes 3.[1]



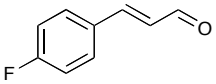
To a stirred solution of aryl halides (2.5 mmol) in 10 mL of DMF were added acrolein diethyl acetal (7.5 mmol), nBu$_4$NOAc (5 mmol), K_2CO_3 (3.75 mmol), KCl (2.5 mmol) and $Pd(OAc)_2$ (3 mol%). Stirring was continued at 90 ^oC for the completion of the reaction (5-10 h). After cooling, 2 N HCl was slowly added and the reaction mixture was stirred at room temperature for 10 min. The mixture was extracted with ether and washed with water. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica column chromatography using pet ether-ethyl acetate mixture as eluent (90:10 v/v).



3b (85%)



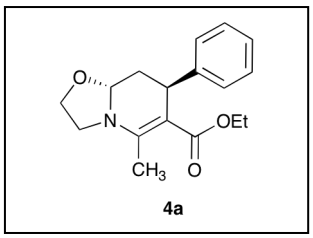
3c (79%)



3d (70%)

2. Characterization data of compounds 4 and 5.

Ethyl 5-methyl-7-phenyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4a):

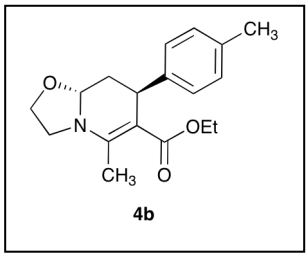


4a

Yellow viscous liquid; yield: 75%; IR (neat): 2932.2, 2844.2, 1681.3, 1553.8, 1287.0, 1118.2 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, J = 7.2 Hz, 3H, $CH_2\underline{CH_3}$), 1.64-1.73 (m, 1H, H_c), 2.27-2.30 (dt, J = 12.6, 3.3 Hz, 1H, H_b), 2.59 (s, 3H, CH_3), 3.52-3.63 (m, 2H, NCH_2), 3.82-3.88 (m, 1H, $O\underline{CH_2}CH_2N$), 3.94 (q, J = 7.2 Hz, 2H, $O\underline{CH_2}CH_3$), 4.17-4.22 (m, 2H, $O\underline{CH_2}CH_2N$ & Ph$\underline{CH}$), 4.39 (dd, J = 10.5, 3.6 Hz, 1H, H_d), 7.11-7.17 (m, 3H, ArH), 7.22-7.26 (m, 2H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.3 (CH_3), 18.0 (CH_3), 33.5 (Ph$\underline{C}$H), 38.1 (PhCH$\underline{C}H_2$), 46.3 (N$\underline{C}H_2$), 58.7 (COO$\underline{C}H_2$), 65.6 (O$\underline{C}H_2$), 84.5 (O$\underline{C}$HN), 95.5 (C=$\underline{C}CO_2$Et), 125.8 (Ar$\underline{C}$H), 127.5 (Ar$\underline{C}$H), 128.1 (Ar$\underline{C}$H), 146.5 (Ar-Quaternary), 152.5 (N$\underline{C}$=CCO_2Et), 168.7 ($\underline{C}$O). Anal Calcd for $C_{17}H_{21}NO_3$: C, 71.06; H, 7.37; N, 4.87. Found: C, 70.77; H, 7.28; N, 4.75.

[1] G. Battistuzzi, S. Cacchi and G. Fabrizi, *Org. Lett*., 2003, **5**, 777.

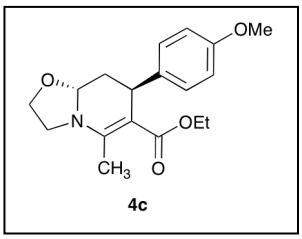
Ethyl 5-methyl-7-*p*-tolyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4b):



4b

Yellow viscous liquid; yield: 83%; IR (neat): 2954.1, 2866.8, 1670.1, 1567.6, 1420.9, 1289.0, 1123.5 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.54-1.64 (m, 1H), 2.17 (ddd, *J* = 12.0, 3.6, 2.4 Hz, 1H), 2.23 (s, 3H), 2.51 (s, 3H), 3.47-3.53 (m, 2H), 3.72-3.80 (m, 1H), 3.84-3.92 (m, 2H), 4.09-4.15 (m, 2H), 4.33 (dd, *J* = 10.5, 5.4 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.3, 18.0, 21.0, 33.6, 37.6, 46.3, 58.7, 65.6, 84.6, 95.7, 127.4, 128.8, 135.2, 143.5, 152.2, 168.7. Anal Calcd for $C_{18}H_{23}NO_3$: C, 71.73; H, 7.69; N, 4.65. Found: C, 71.41; H, 7.61; N, 4.54.

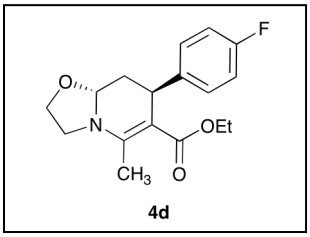
Ethyl 7-(4-methoxyphenyl) -5-methyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4c):



4c

Pale yellow viscous liquid; yield: 80%; IR (neat): 2920.6, 2852.2, 1674.7, 1564.5, 1426.1, 1292.0, 1118.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.01 (t, *J* = 6.9 Hz, 3H), 1.62-1.68 (m, 1H), 2.23 (dt, *J* = 11.7, 2.4 Hz, 1H), 2.60 (s, 3H), 3.55-3.62 (m, 2H), 3.80 (s, 3H), 3.82-3.88 (m, 1H), 3.95 (q, *J* = 7.2 Hz, 2H), 4.18-4.23 (m, 2H), 4.39 (dd, *J* = 10.5, 3.6 Hz, 1H) 6.79 (d, *J* = 8.7 Hz, 2H), 7.03 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.3, 17.9, 33.7, 37.2, 46.3, 55.2, 58.7 65.5, 84.5, 95.8, 113.4, 128.4, 138.6, 152.1, 157.7, 168.7. Anal Calcd for $C_{18}H_{23}NO_4$: C, 68.12; H, 7.30; N, 4.41. Found: C, 67.81; H, 7.18; N, 4.34.

Ethyl 7-(4-fluorophenyl)-5-methyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4d):

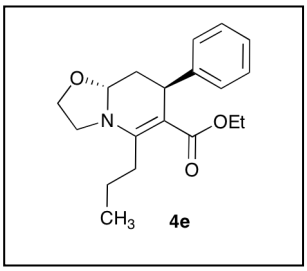


4d

Yellow viscous liquid; yield: 73%; IR (neat); 2930.5, 2873.3, 1676.4, 1566.5, 1505.5, 1290.0, 1222.9, 1118.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.93 (t, *J* = 7.2 Hz, 3H), 1.58-1.65 (m, 1H), 2.16 (ddd, *J* = 12.0, 3.6, 2.4 Hz, 1H), 2.51 (s, 3H), 3.48-3.54 (m, 2H), 3.74-3.92 (m, 3H) 4.11-4.15 (m, 2H), 4.30 (dd, *J* = 10.5, 3.6 Hz, 1H), 6.83-6.89 (m, 2H), 6.99-7.03 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.3, 18.0, 33.6, 37.4, 46.3, 58.8, 65.6, 84.3, 95.5, 114.8 (d, *J* = 21.0 Hz), 128.9 (d, *J* = 7.5 Hz), 142.2 (d, *J* = 3.0 Hz), 152.5, 161.2 (d, *J*

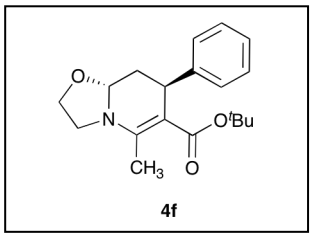
= 241.5 Hz), 168.5. Anal Calcd for $C_{17}H_{20}FNO_3$: C, 66.87; H, 6.60; N, 4.59. Found: C, 66.59; H, 6.49; N, 4.45.

Ethyl 7-phenyl-5-propyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4e):



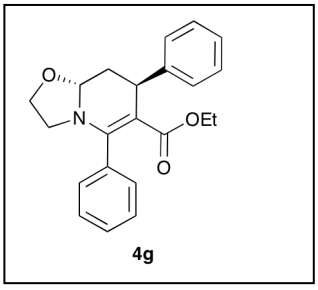
Yellow viscous liquid; yield: 81%; IR (neat): 2945.2, 2887.1, 1680.9, 1545.8, 1281.2, 1232.1, 1121.0 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.6 Hz, 3H), 1.03 (t, *J* = 7.5 Hz, 3H), 1.56-1.65 (m, 2H), 1.68-1.78 (m, 1H), 2.20 (ddd, *J* = 11.1, 3.6, 2.4 Hz, 1H), 2.55 (td, *J* = 12.0, 5.1 Hz, 1H), 3.12 (td, *J* = 11.4, 5.1 Hz, 1H), 3.50-3.55 (m, 2H), 3.74-3.82 (m, 1H), 3.88 (q, *J* = 6.6 Hz, 2H), 4.10-4.17 (m, 2H), 4.31 (dd, *J* = 10.5, 3.6 Hz, 1H), 7.03-7.07 (m, 3H), 7.10-7.18 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): 14.3, 14.5, 22.5, 33.4, 33.6, 38.0, 45.7, 58.7, 65.6, 84.5, 95.1, 125.8, 127.5, 128.1, 146.7, 156.6, 168.1. Anal Calcd for $C_{19}H_{25}NO_3$: C, 72.35; H, 7.99; N, 4.44. Found: C, 72.02; H, 7.81; N, 4.32.

***tert*-Butyl 5-methyl-7-phenyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4f):**



Colourless viscous liquid; yield: 82%; IR (neat): 2945.1, 2888.2, 1670.0, 1560.5, 1478.9, 1272.0, 1134.5 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.12 (s, 9H), 1.60-1.66 (m, 1H), 2.16 (dt, *J* = 12.0, 2.4 Hz, 1H),2.48 (s, 3H), 3.46-3.55 (m, 2H), 3.74-3.82(m, 1H) 4.07-4.15 (m, 2H), 4.34 (dd, *J* = 10.2, 3.3 Hz, 1H), 7.04-7.09 (m, 3H), 7.15-7.20 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): 16.5, 26.9, 32.5, 37.5, 44.9, 64.3, 76.7, 83.1, 96.0, 124.4, 126.4, 126.7, 145.8, 150.2, 167.0. Anal Calcd for $C_{19}H_{25}NO_3$: C, 72.35; H, 7.99; N, 4.44. Found: C, 71.99; H, 7.84; N, 4.32.

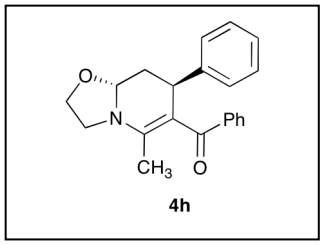
Ethyl 5,7-diphenyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridine-6-carboxylate (4g):



Yellow viscous liquid; yield: 62%; IR (neat): 2951.8, 2866.0, 1688.3, 1554.1, 1501.0, 1243.6, 1116.5 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.59 (t, *J* = 7.2 Hz, 3H), 1.74

(ddd, *J* = 12.0, 10.2, 5.7 Hz, 1H), 2.28 (ddd, *J* = 12.3, 3.6, 2.4 Hz, 1H), 2.89-2.97 (m, 1H), 3.09-3.16 (m, 1H), 3.57-3.67 (m, 2H), 3.68-3.77 (m, 1H), 3.90-3.96 (m, 1H), 4.32 (dd, *J* = 5.7, 2.4 Hz, 1H), 4.55 (dd, *J* = 10.2, 3.6 Hz, 1H), 7.09-7.25 (m, 6H), 7.32-7.39 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): 13.6, 33.9, 38.2, 48.1, 58.8, 65.5, 85.0, 99.4, 126.1, 127.6, 128.0, 128.3, 128.8, 138.6, 145.6, 154.0, 168.0. Anal Calcd for $C_{22}H_{23}NO_3$: C, 75.62; H, 6.63; N, 4.01. Found: C, 75.35; H, 6.49; N, 3.89. One aromatic carbon is merged with others.

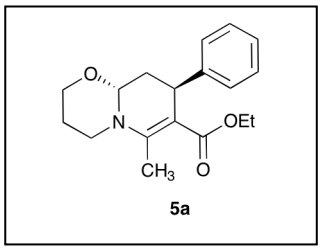
5-Methyl-7-phenyl-3,7,8,8a-tetrahydro-2*H*-oxazolo[3,2-*a*]pyridin-6-yl)(phenyl)methanone (4h):



4h

Pale yellow viscous liquid; yield: 61%; IR (neat): 2921.1, 2879.3, 1689.0, 1533.8, 1501.8, 1267.8, 1145.7 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.67-1.76 (m, 1H), 2.15 (s, 3H), 2.29-2.34 (m, 1H), 3.56-3.61 (m, 2H), 3.85-3.93 (m, 1H), 4.22-4.27 (m, 1H), 4.41 (d, *J* = 5.1 Hz, 1H), 4.52 (dd, *J* = 10.5, 3.6 Hz, 1H), 7.14 (d, *J* = 6.9 Hz, 2H), 7.20-7.34 (m, 6H), 7.42 (d, *J* = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): 20.0, 33.6, 39.0, 46.4, 65.7, 85.1, 107.9, 126.1, 127.6, 127.9, 128.0, 128.3, 129.8, 143.5, 145.7, 152.4, 195.8. Anal Calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.63; N, 4.39. Found: C, 78.72; H, 6.51; N, 4.35.

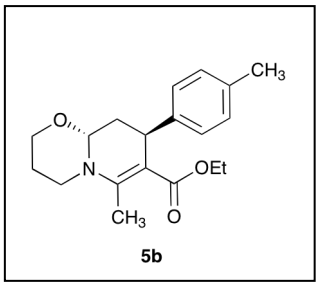
Ethyl 6-methyl-8-phenyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5a):



5a

Pale yellow viscous liquid; yield: 73%; IR (neat); 3023.8, 2929.2, 2849.2, 1683.2, 1576.7, 1437.1, 1119.0 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.92 (t, *J* = 7.2 Hz, 3H), 1.50-1.52 (m, 1H), 1.92-2.19 (m, 3H), 2.45 (s, 3H), 2.98 (td, *J* = 13.2, 2.7 Hz, 1H), 3.61 (td, *J* = 12.0, 2.7 Hz, 1H), 3.87 (q, *J* = 7.2 Hz, 2H), 4.03-4.10 (m, 3H), 4.24 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.13-7.16 (m, 3H), 7.24-7.29 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.0, 16.5, 25.9, 36.1, 37.3, 45.4, 59.1, 67.2, 83.8, 101.2, 125.8, 127.5, 128.2, 146.5, 152.6, 168.9. Anal Calcd for $C_{18}H_{23}NO_3$: C, 71.73; H, 7.69; N, 4.65. Found: C, 71.49; H, 7.60; N, 4.54.

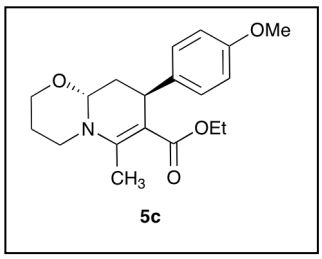
Ethyl 6-methyl-8-*p*-tolyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5b):



5b

Yellow viscous liquid; yield: 77%; IR (neat); 2957.5, 2926.5, 1682.8, 1579.2, 1438.0, 1207.3, 1122.4 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 3H), 1.43-1.48 (m, 1H), 1.81-2.10 (m, 3H), 2.24 (s, 3H), 2.38 (s, 3H) 2.88 (td, *J* = 13.0, 2.7 Hz, 1H), 3.56 (td, *J* = 12.0, 2.7 Hz, 1H), 3.77-3.88 (m, 2H), 3.95-4.00 (m, 3H), 4.15 (dd, *J* = 8.4, 4.5 Hz, 1H), 6.93-7.09 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.1, 16.5, 21.0, 26.0, 36.2, 36.9, 45.3, 59.1, 67.1, 83.9, 101.3, 127.2, 128.9, 135.2, 143.4, 152.6, 169.0. Anal Calcd for $C_{19}H_{25}NO_3$: C, 72.35; H, 7.99; N, 4.44. Found: C, 72.03; H, 7.85; N, 4.35.

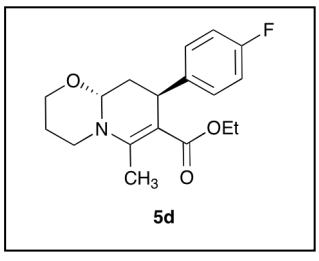
Ethyl 8-(4-methoxyphenyl) -6-methyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5c):



5c

Yellow viscous liquid; yield: 78%; IR (neat): 2978.5, 2943.1, 1677.9, 1589.6, 1443.9, 1265.1, 1117.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.2 Hz, 3H), 1.43-1.48 (m, 1H), 1.84-2.10 (m, 3H), 2.37 (s, 3H), 2.89 (td, *J* = 13.0, 2.7 Hz, 1H), 3.52 (td, *J* =12.0, 2.4 Hz, 1H), 3.70 (s, 3H), 3.82 (q, *J* = 7.2 Hz, 2H), 3.95-3.99 (m, 3H), 4.14 (dd, *J* = 8.4, 4.2 Hz, 1H), 6.72 (d, *J* =8.7 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.1, 16.5, 25.9, 36.2, 36.4, 45.3, 55.2, 59.1, 67.1, 83.9, 101.5, 113.5, 128.3, 138.5, 152.4, 157.7, 168.9. Anal Calcd for $C_{19}H_{25}NO_4$: C, 68.86; H, 7.60; N, 4.23. Found: C, 68.58; H, 7.53; N, 4.19.

Ethyl 8-(4-fluorophenyl) -6-methyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5d):

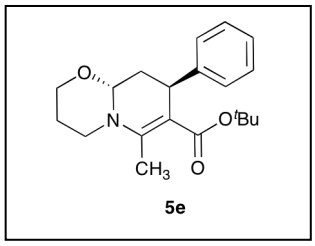


5d

Colourless viscous liquid; yield: 78%; IR (neat): 2989.3, 2865.7, 1681.2, 1545.7, 1476.1, 1232.8, 1332.1 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.86 (t, *J* = 7.2 Hz, 3H), 1.44-1.49 (m, 1H), 1.85-1.96 (m, 2H), 2.02-2.11 (m, 1H) 2.37 (s, 3H), 2.92 (td, *J* = 12.9, 2.7 Hz, 1H), 3.55 (td, *J* =12.0, 2.4 Hz, 1H), 3.81 (q, *J* = 7.2 Hz, 2H), 3.97-4.01 (m, 3H), 4.15 (dd, *J* =8.7, 4.2 Hz, 1H),

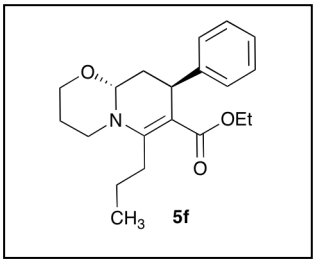
6.85-6.90 (m, 2H), 6.99-7.05 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 13.0, 15.4, 24.9, 35.2, 35.6, 44.4, 58.1, 66.2, 82.7, 100.1, 113.9 (d, J = 21.0 Hz), 127.6 (d, J = 7.5 Hz), 141.1 (d, J = 3.0 Hz), 151,7, 160.2 (d, J = 241.5 Hz), 167.8. Anal Calcd for $C_{18}H_{22}FNO_3$: C, 67.69; H, 6.94; N, 4.39. Found: C, 67.44; H, 6.87; N, 4.31.

***tert*-Butyl 6-methyl-8-phenyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5e):**



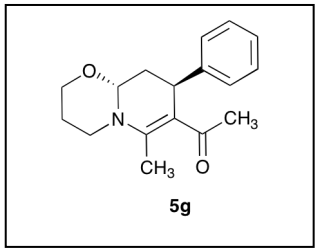
Yellow viscous liquid; yield: 81%; IR (neat): 2976.5, 2922.5, 1677.0, 1571.6, 1440.0, 1119.3 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.04 (s, 9H), 1.41-1.45 (m, 1H), 1.85-1.95 (m, 2H), 2.02-2.11 (m, 1H), 2.31 (s, 3H), 2.92 (td, J =12.9, 2.7 Hz, 1H), 3.58 (td, J = 12.0, 2.4 Hz, 1H), 3.90-4.04 (m, 3H), 4.20(dd, J = 7.5, 3.3 Hz, 1H), 7.08-7.11 (m, 2H), 7.16-7.21 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.3, 25.9, 27.9, 36.4, 37.9, 45.7, 67.3, 78.6, 83.9, 103.8, 125.7, 127.5, 128.1, 147.1, 150.9, 168.4. Anal Calcd for $C_{20}H_{27}NO_3$: C, 72.92; H, 8.26; N, 4.25. Found: C, 72.65; H, 8.11; N, 4.16.

Ethyl 8-phenyl-6-propyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5f):



Pale yellow semi-solid; yield: 70%; IR (neat): 2992.1, 2920.3, 1691.5, 1521.2, 1412.9, 1256.8, 1129.4 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, J = 7.2 Hz, 3H), 0.99 (t, J = 7.5 Hz, 3H), 1.45-1.55 (m, 3H), 1.82-2.06 (m, 3H), 2.61-2.75 (m, 1H), 2.87-2.99 (m, 2H), 3.53 (td, J = 12.0, 2.7 Hz, 1H), 3.76-3.85 (m, 2H), 3.87-4.02 (m, 3H), 4.16 (dd, J =8.4, 4.2 Hz, 1H), 7.04-7.09 (m, 2H), 7.16-7.22 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.0, 14.1, 22.1, 26.3, 30.5, 36.1, 37.1, 45.4, 58.9, 67.1, 84.0, 100.1, 125.8, 127.3, 128.2, 146.4, 156.7, 168.5. Anal Calcd for $C_{20}H_{27}NO_3$: C, 72.92; H, 8.26; N, 4.25. Found: C, 72.69; H, 8.21; N, 4.18.

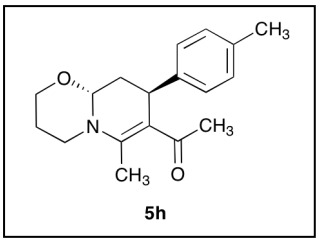
1-(6-Methyl-8-phenyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazin-7-yl)ethanone (5g):



5g

Pale yellow solid; yield: 78%; mp: 130-131 ^{o}C; IR (neat): 2956.8, 2849.3, 1634.2, 1527.1, 1425.2, 1361.7, 1085.5 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.52-1.61 (m, 1H), 1.85-2.05 (m, 2H), 1.93 (s, 3H), 2.16-2.20 (m, 2H), 2.49 (s, 3H), 2.96 (td, *J* = 12.9, 3.0 Hz, 1H), 3.56 (td, *J* = 12.9, 3.0 Hz, 1H), 3.95-4.11 (m, 2H), 4.17 (dd, *J* = 8.1, 5.4 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 7.19-7.24 (m, 1H), 7.28-7.33 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.8, 25.9, 29.6, 36.4, 38.6, 44.7, 67.0, 83.5, 109.3, 126.4, 127.5, 128.7, 145.1, 153.3, 198.4. Anal Calcd for $C_{20}H_{27}NO_3$: C, 75.25; H, 7.80; N, 5.16. Found: C, 74.97; H, 7.68; N, 5.11.

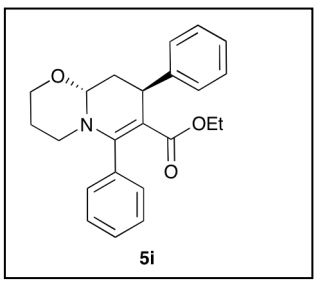
1-(6-Methyl-8-*p*-tolyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazin-7-yl)ethanone (5h):



5h

Pale yellow viscous liquid; yield: 74%; IR (neat): 2988.1, 2865.6, 1657.2, 1565.7, 1469.8, 1225.8, 1081.8 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.46-1.51 (m, 1H), 1.85 (s, 3H), 1.86-1.89 (m, 1H), 2.07-2.11 (m, 2H), 2.22 (s, 3H), 2.41 (s, 3H), 2.87 (td, *J* = 12.9, 3.0 Hz, 1H), 3.48 (td, *J* =12.0, 2.7 Hz, 1H), 3.83-4.06 (m, 3H), 4.09 (dd, *J* = 7.8, 6.0 Hz, 1H), 6.94 (d, *J* = 8.1 Hz, 2H), 7.04 (d, *J* = 8.1 Hz, 2H). Anal Calcd for $C_{18}H_{23}NO_2$: C, 75.76; H, 8.12; N, 4.91. Found: C, 75.65; H, 8.03; N, 4.82.

Ethyl 6,8-diphenyl-2,3,4,8,9,9a-hexahydropyrido[2,1-*b*][1,3]oxazine-7-carboxylate (5i):



5i

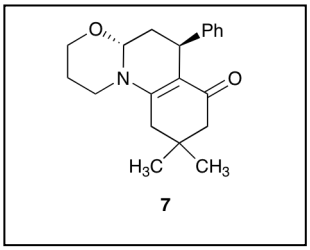
Pale yellow viscous liquid; yield: 65%; IR (neat): 2967.3, 2889.2, 1688.3, 1545.2, 1465.1, 1233.8, 1119.4 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.65 (t, *J* = 7.2 Hz, 3H), 1.25-1.30 (m, 1H), 1.72-1.85 (m, 1H), 2.14-2.22 (m, 1H), 2.26-2.35 (m, 1H) 2.79 (td, *J* = 12.9, 2.7 Hz, 1H), 3.20-3.25 (m, 1H), 3.64-3.69 (m, 3H), 4.04 (dd, *J* = 11.4, 4.8 Hz, 1H), 4.15 (t, *J* = 5.7 Hz, 1H), 4.42 (dd, *J* = 8.1, 4.2 Hz, 1H), 7.21-7.34 (m, 7H), 7.38-7.42 (m,3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 13.5, 26.0, 36.2, 37.2, 47.0, 58.9, 67.3, 84.0, 102.0, 126.0, 127.3, 127.9,

128.3, 128.4, 128.8, 137.5, 145.8, 154.4, 168.0. Anal Calcd for $C_{23}H_{25}NO_3$: C, 76.01; H, 6.93; N, 3.85. Found: C, 75.79; H, 6.89; N, 3.82.

3. General procedure for the synthesis of compounds 7 and 8.

To a stirred solution of amino alcohol **1** (1.3 mmol), dimedone **6** (1.0 mmol) and cinnamaldehyde **3a** (1 mmol) in acetonitrile (2 mL) was added CAN (10 mol%) and stirring was continued for 3 h at 25 ^{o}C. The reaction mixture was diluted with water and extracted with dichloromethane. The organic layer was washed with water followed by brine and evapored to dryness. The crude product was purified by silica column chromatography using pet ether-ethyl acetate mixture as eluent (60:40 v/v).

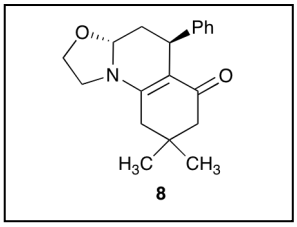
9,9-Dimethyl-6-phenyl-2,3,4a,5,6,8,9,10-octahydro-[1,3]oxazino[3,2-*a*]quinolin-7(1*H*)-one (7):



7

White solid; yield: 42%; mp: 160-161 ^{o}C; IR (neat) 3018.8, 2963.6, 2862.8, 1617.1, 1560.0, 1448.5, 1323.6, 1099.8, 1058.3 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.02 (s, 3H), 1.10 (s, 3H), 1.43-1.48 (m, 1H), 1.70-1.86 (m, 1H), 2.05-2.24 (m, 5H), 2.45 (d, *J* = 16.2 Hz, 1H), 3.12 (td, *J* = 12.9, 2.7 Hz, 1H), 3.53 (td, *J* = 12.9, 2.7 Hz, 1H), 3.83 (dd, *J* = 11.4, 4.8 Hz, 1H), 3.98-4.01 (m, 1H), 4.04-4.08 (m, 1H) 4.48 (t, *J* = 3.6 Hz, 1H), 6.98-7.04 (m, 1H), 7.07-7.15 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 27.0, 28.0, 29.8, 32.2, 33.0, 34.9, 40.6, 46.6, 49.6, 67.7, 85.3, 109.9, 125.3, 127.5, 127.7, 145.4, 156.0, 194.1 Anal Calcd for $C_{20}H_{25}NO_2$: C, 77.14; H, 8.09; N, 4.50. Found: C, 76.91; H, 8.01; N, 4.43.

8,8-Dimethyl-5-phenyl-3a,4,5,7,8,9-hexahydro-1*H*-oxazolo[3,2-*a*]quinolin-6(2*H*)-one (8):

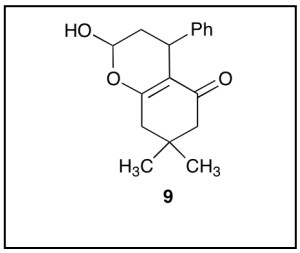


8

White solid; yield: 27%; mp:145-147 ^{o}C; IR (neat) 3008.3, 2937.0, 2880.4, 1621.8, 1547.1, 1438.7, 1302.9, 1070.9 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.06 (s, 3H), 1.11 (s, 3H), 1.54-1.60 (m, 1H), 2.17-2.20 (m, 2H), 2.25-2.33 (m, 1H) 2.38-2.45 (m, 2H), 3.43-3.56 (m, 2H), 3.79-3.85 (m, 1H), 4.16-4.26 (m, 2H), 4.38 (dd, *J* = 10.5, 3.9 Hz, 1H), 7.04-7.09 (m, 3H), 7.15-7.19 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.5, 29.4, 32.4, 33.3, 34.1, 41.4, 45.6, 49.7,

65.7, 85.2, 106.6, 126.0, 127.5, 128.3, 144.9, 155.8, 192.5. Anal Calcd for $C_{19}H_{23}NO_2$: C, 76.73; H, 7.80; N, 4.71. Found: C, 76.51; H, 7.74; N, 4.68.

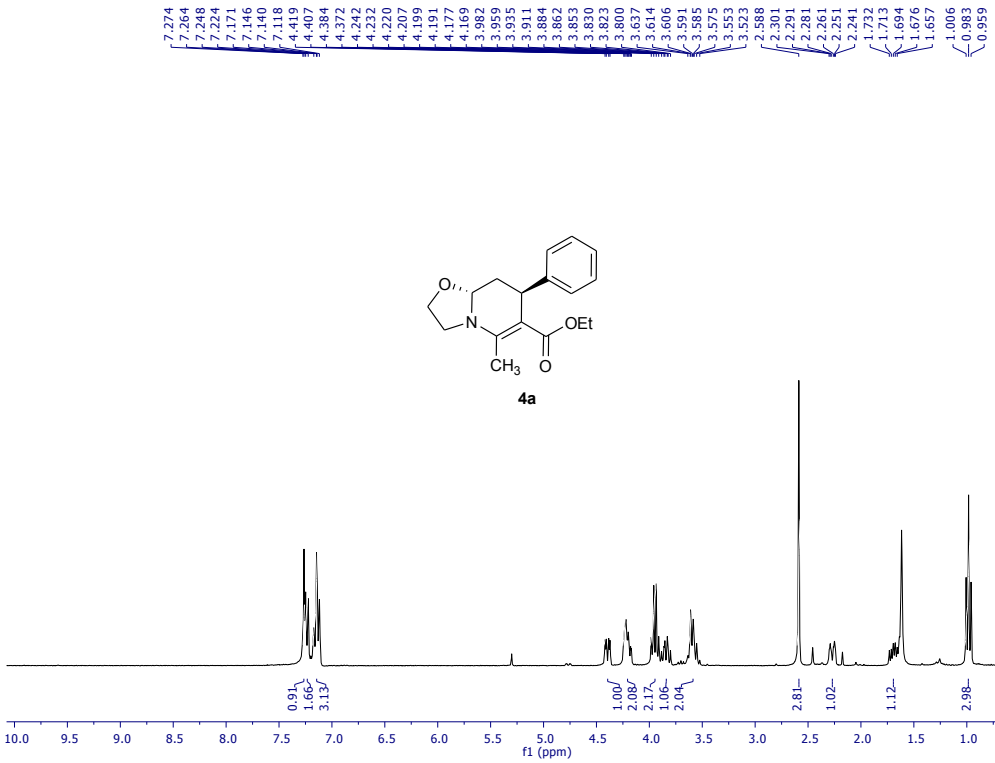
2-Hydroxy-7,7-dimethyl-4-phenyl-3,4,7,8-tetrahydro-2*H*-chromen-5(6*H*)-one (9):

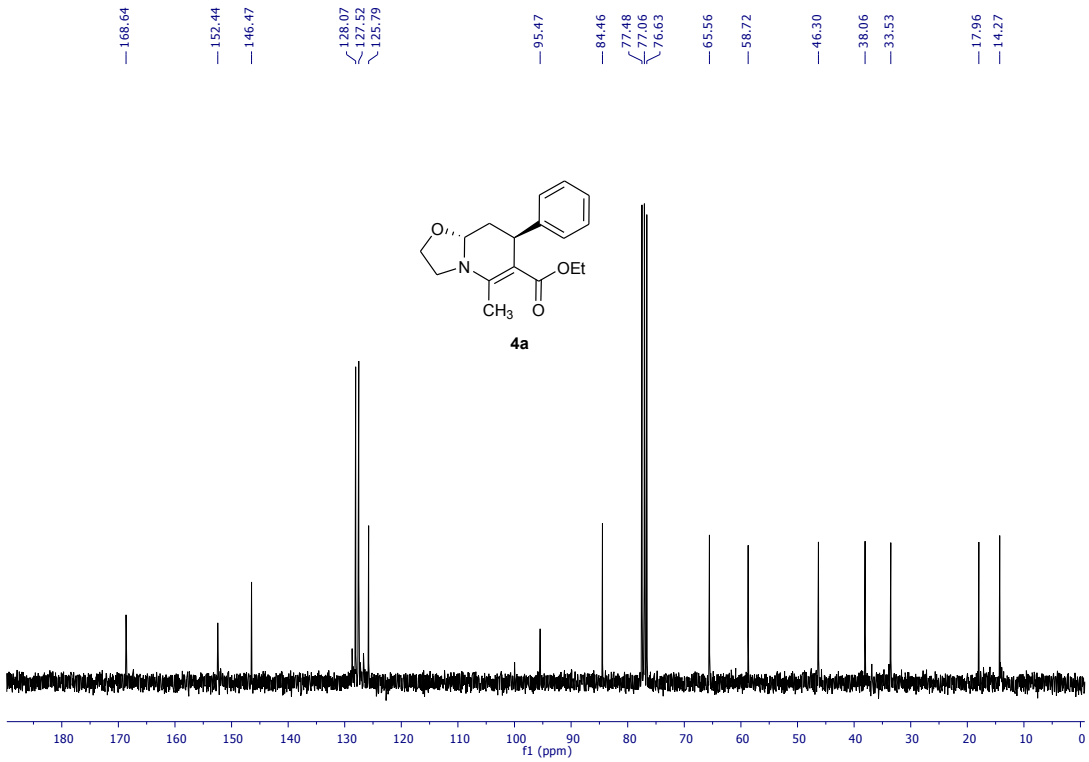


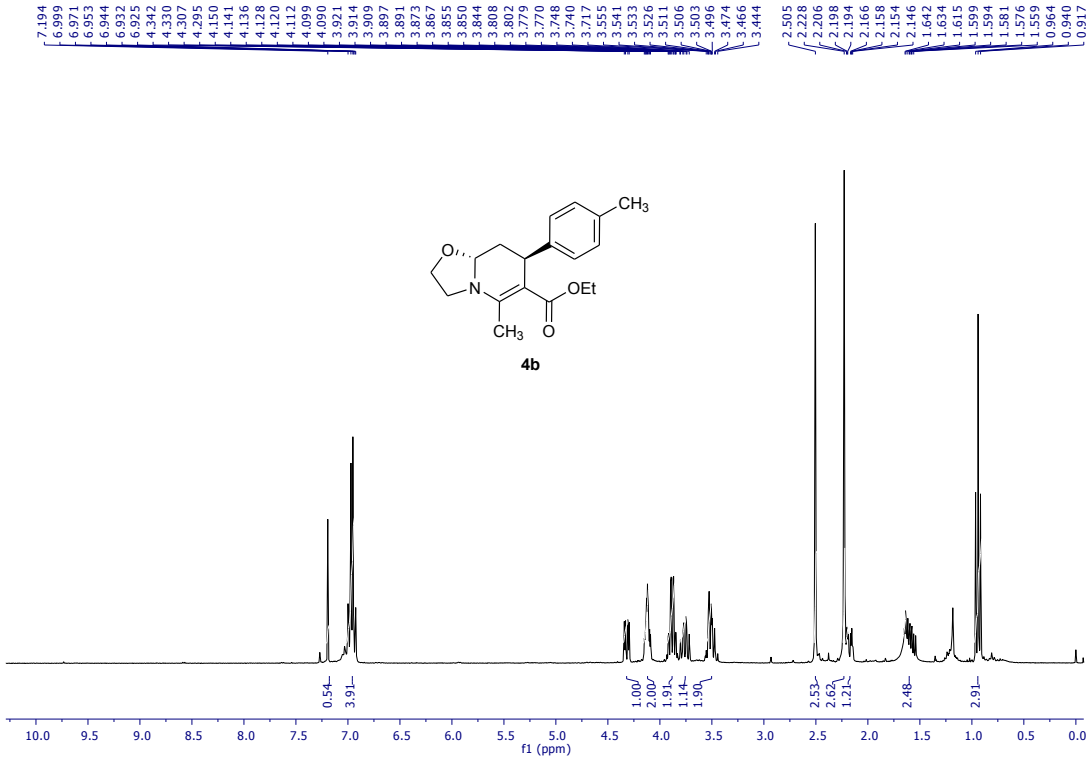
9

White solid; yield: 22%; mp: 200-201 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.12 (s, 3H), 1.18 (s, 3H), 1.95-2.05 (m, 1H), 2.14-2.19 (m, 1H), 2.22-2.36 (m, 2H), 2.42-2.51 (m, 2H), 3.65 (d, *J* = 6.9 Hz, 1H), 4.08-4.09 (m, 1H), 5.23 (t, *J* = 7.2 Hz, 1H), 7.11-7.22 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.1, 28.9, 32.2, 33.9, 36.8, 42.5, 50.7, 93.2, 111.3, 126.4, 127.4, 128.5,144.1, 169.4, 197.0

4. Copies of NMR spectra



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2.154
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0.940
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CH3
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1.00
2.00
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2.53
2.62
1.21
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2.91
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
3.0
2.5
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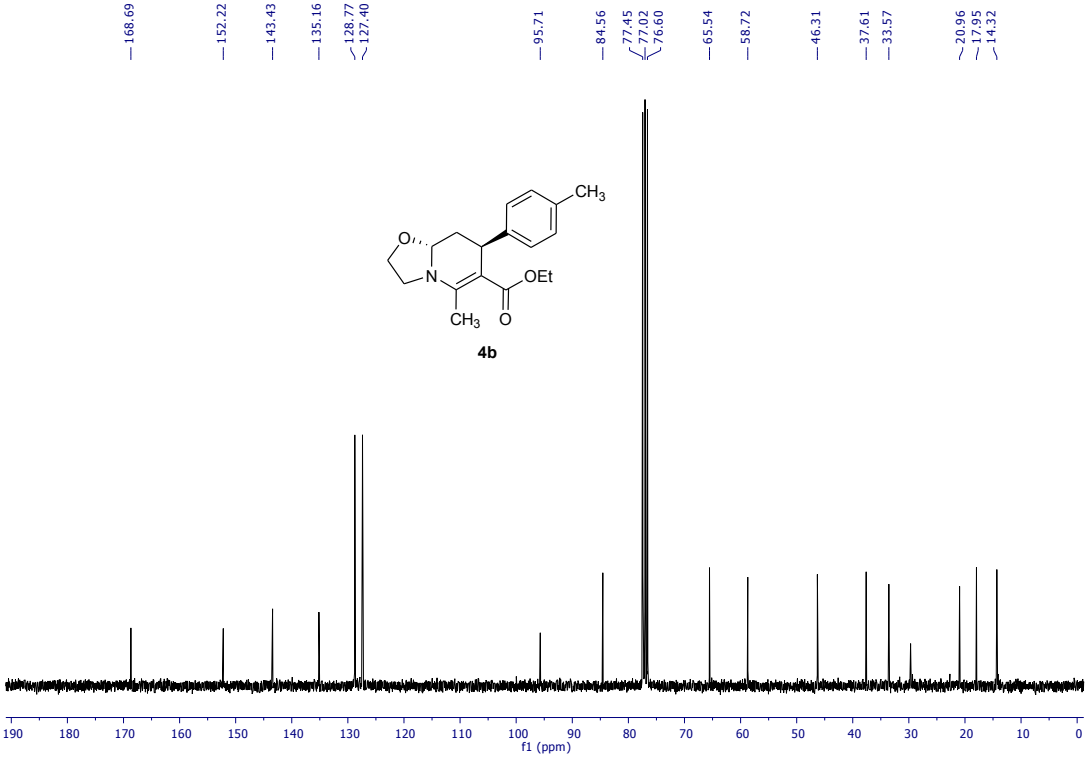
0.0
f1 (ppm)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46.31
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33.57
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17.95
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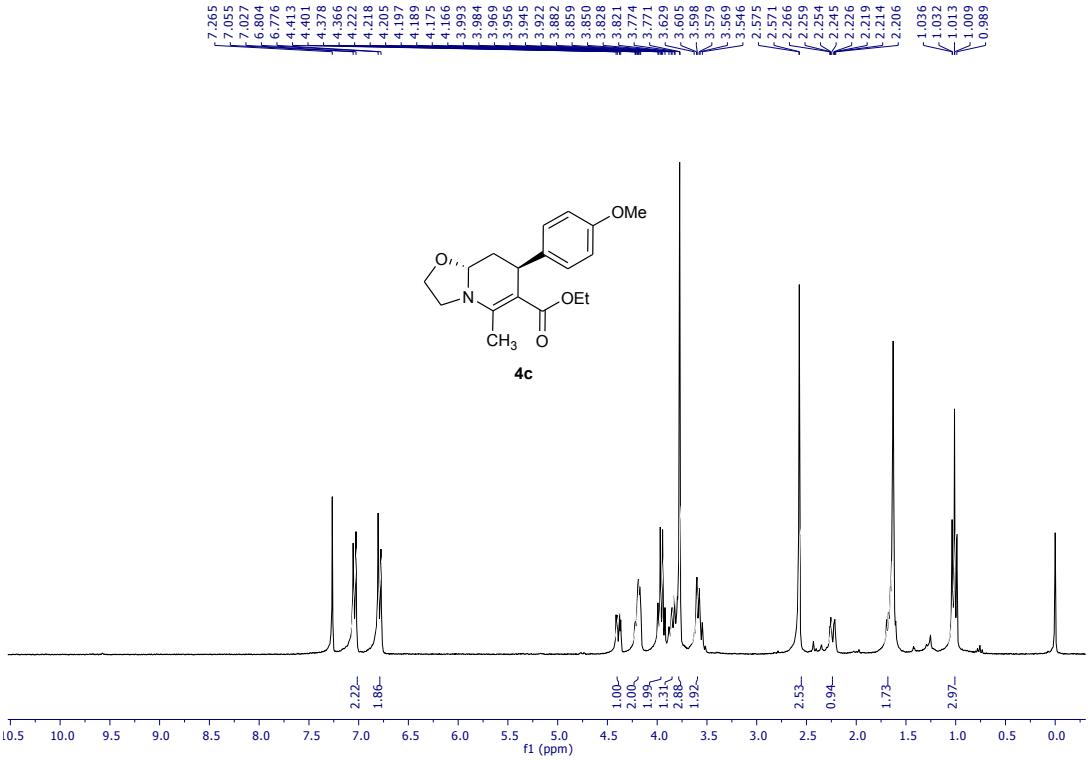
f1 (ppm)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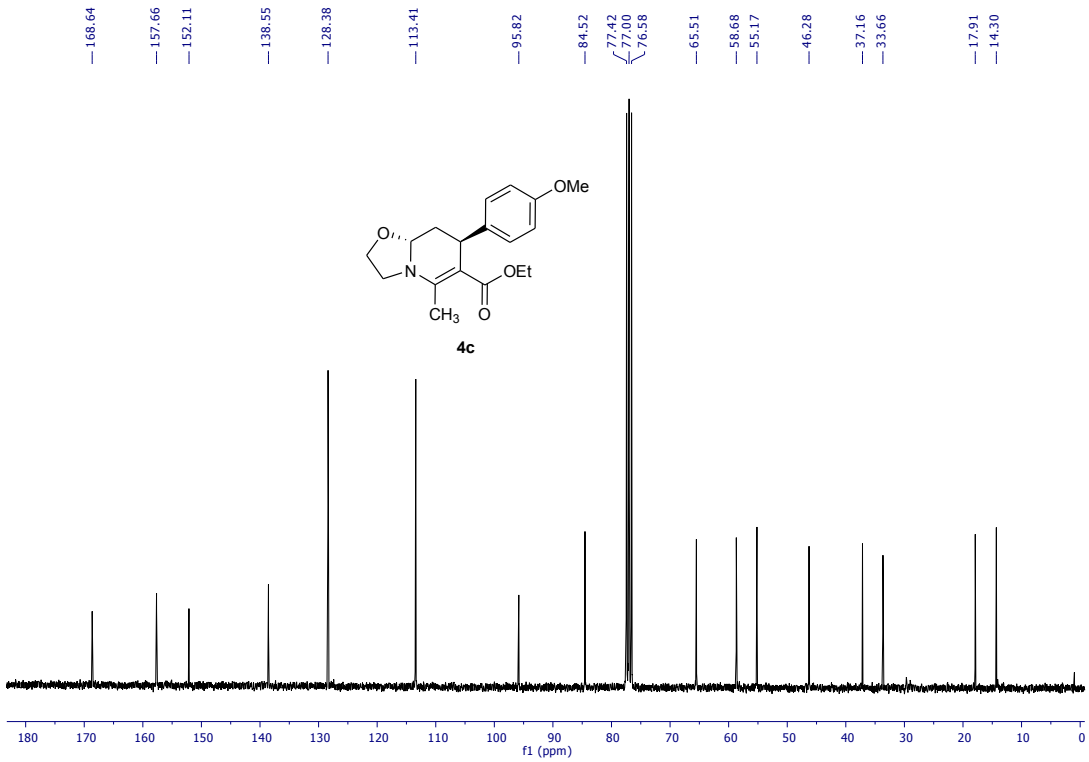
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N
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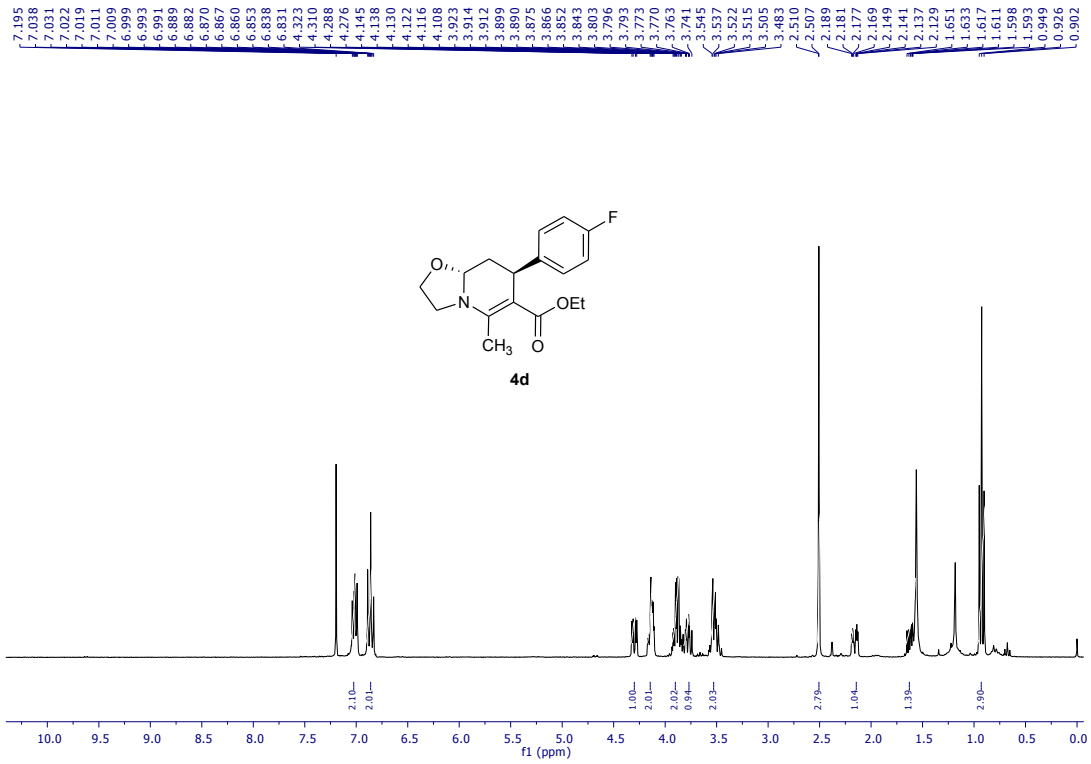
CH3
O
4c
f1 (ppm)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O
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1.00
2.01
2.02
0.94
2.03
2.79
1.04
1.39
2.90
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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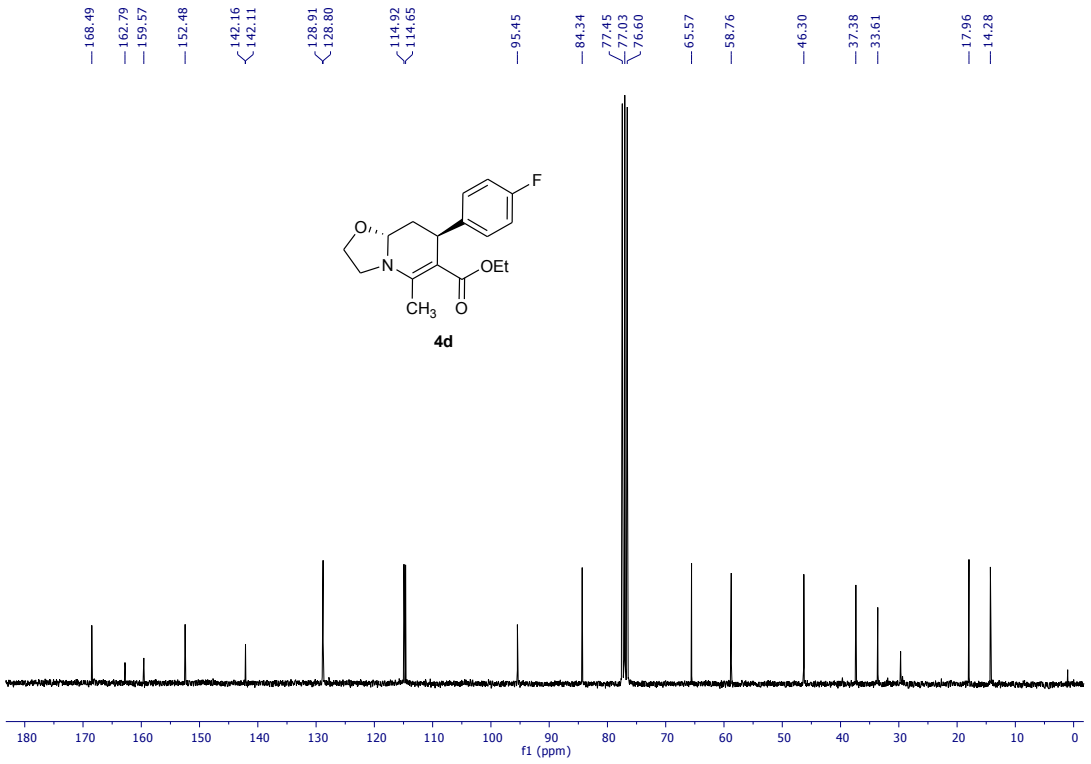
f1 (ppm)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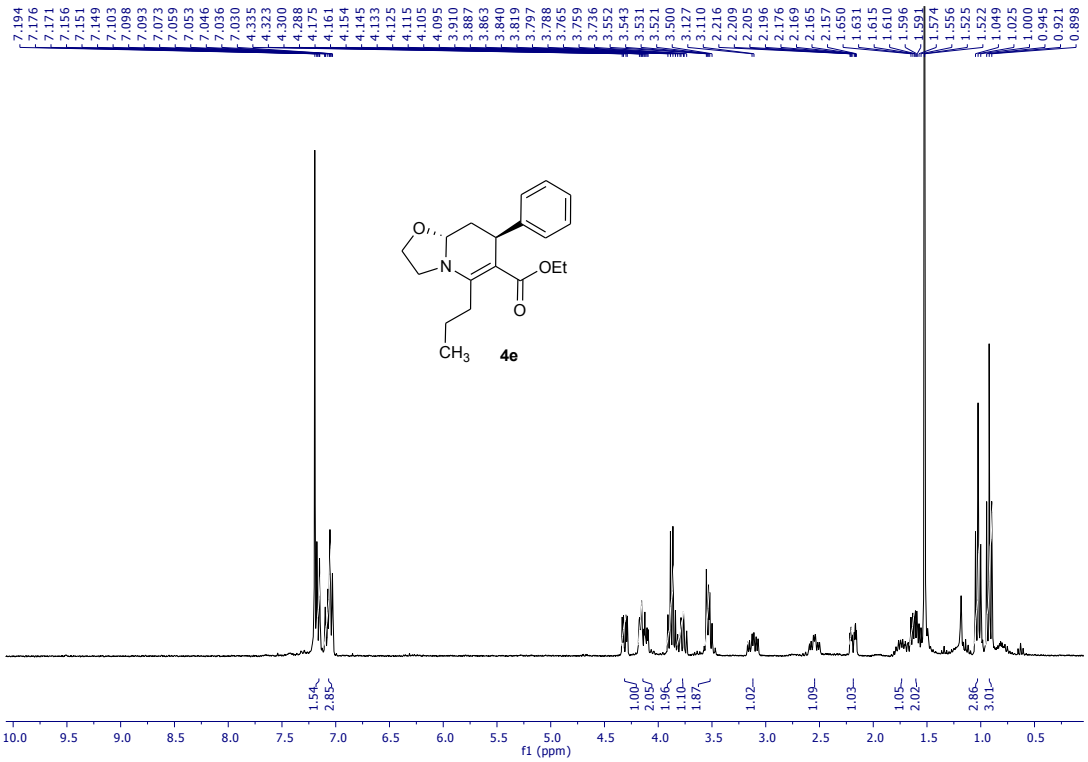
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.098
7.093
7.073
7.059
7.053
7.046
7.036
7.030
4.335
4.323
4.300
4.288
4.175
4.161
4.154
4.145
4.133
4.125
4.115
4.105
4.095
3.910
3.887
3.863
3.840
3.819
3.797
3.788
3.765
3.759
3.736
3.552
3.543
3.531
3.521
3.500
3.127
3.110
2.216
2.209
2.205
2.196
2.176
2.169
2.165
2.157
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1.631
1.615
1.610
1.596
1.591
1.574
1.556
1.525
1.522
1.049
1.025
1.000
0.945
0.921
0.898
O
N
OEt
O
CH3
4e
1.54
2.85
1.00
2.05
1.96
1.10
1.87
1.02
1.09
1.03
1.05
2.02
2.86
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.0
3.5
3.0
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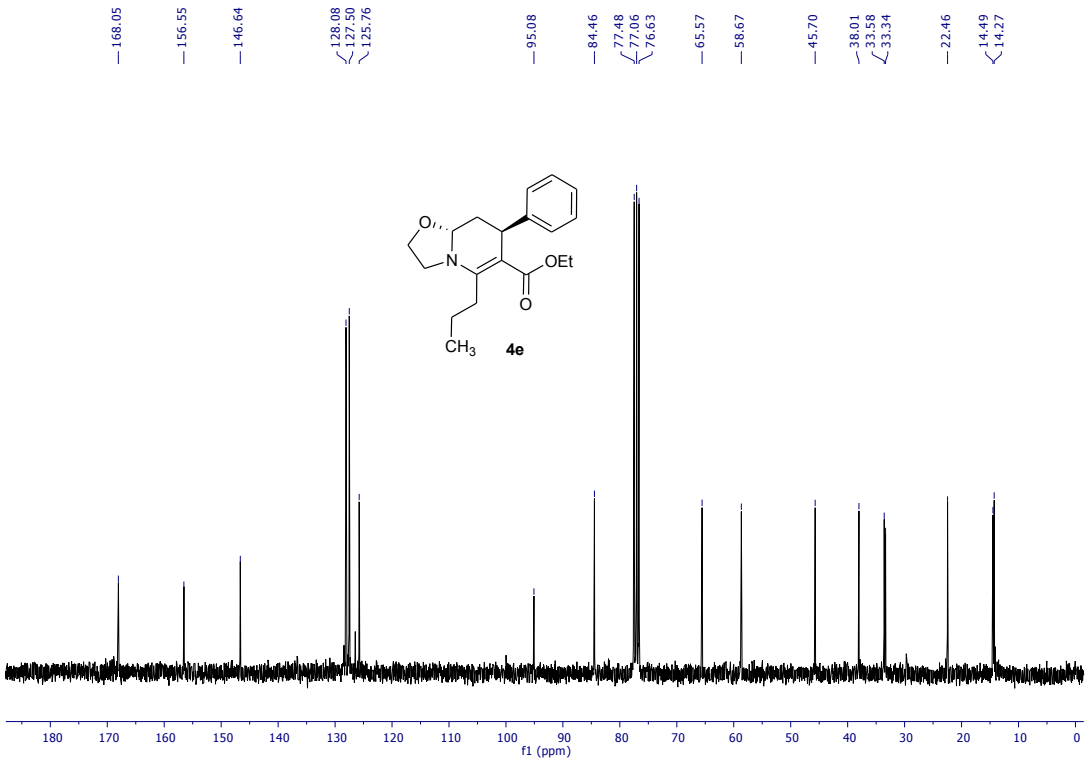
2.0
1.5
1.0
0.5
f1 (ppm)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77.06
76.63
65.57
58.67
45.70
38.01
33.58
33.34
22.46
14.49
14.27
O
N
OEt
O
CH3
4e
180
170
160
150
140
130
120
110
100
90
80
70
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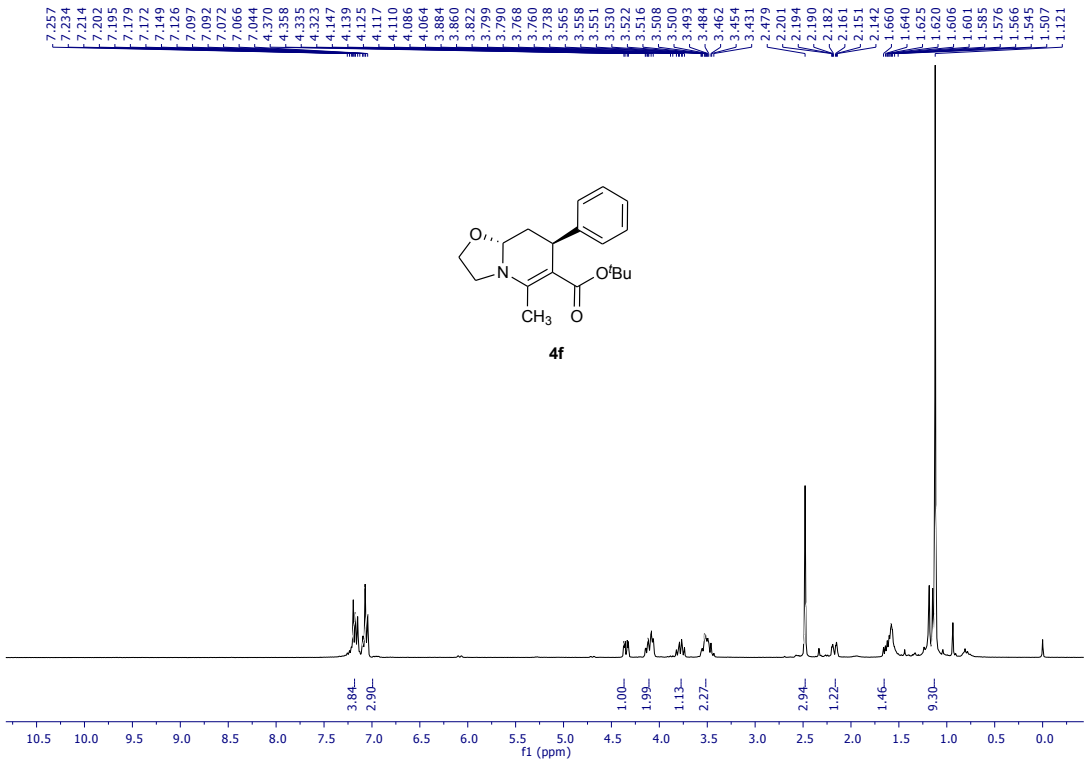
50
40
30
20
10
0
f1 (ppm)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7.202
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7.179
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7.149
7.126
7.097
7.092
7.072
7.066
7.044
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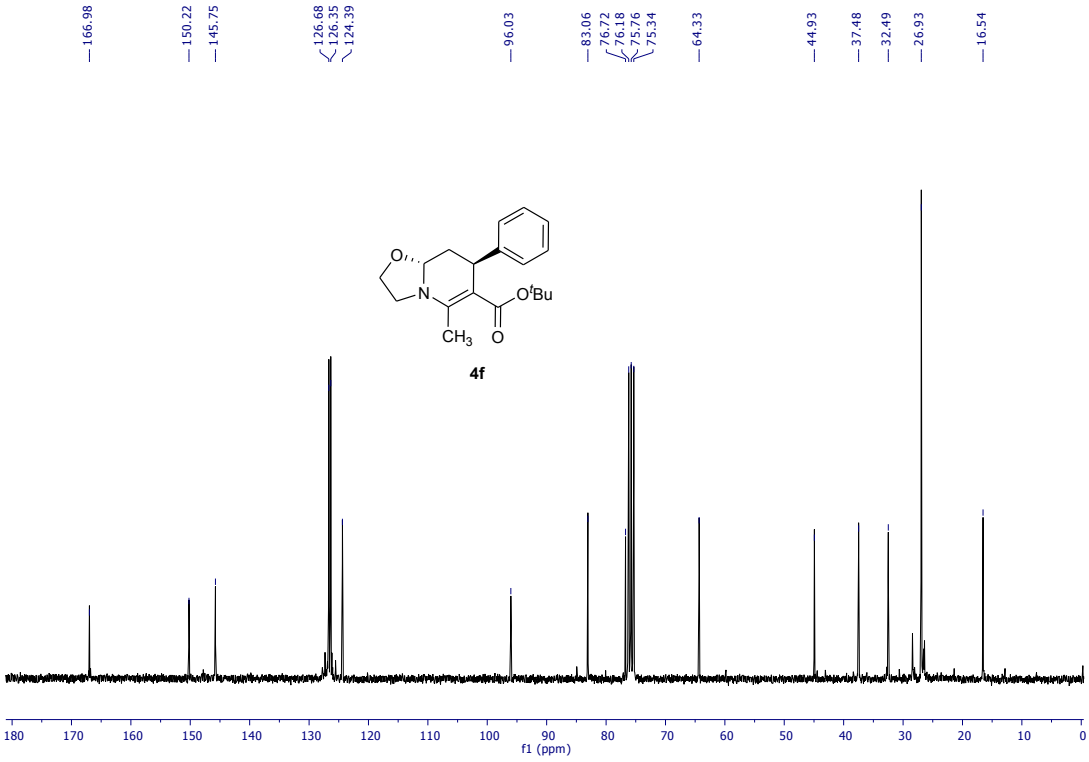
4.358
4.335
4.323
4.147
4.139
4.125
4.117
4.110
4.086
4.064
3.884
3.860
3.822
3.799
3.790
3.768
3.760
3.738
3.565
3.558
3.551
3.530
3.522
3.516
3.508
3.500
3.493
3.484
3.462
3.454
3.431
2.479
2.201
2.194
2.190
2.182
2.161
2.151
2.142
1.660
1.640
1.625
1.620
1.606
1.601
1.585
1.576
1.566
1.545
1.507
1.121
O
N
O^{t}Bu
CH$_3$
O
4f
3.84
2.90
1.00
1.99
1.13
2.27
2.94
1.22
1.46
9.30
10.5 10.0 9.5 9.0 8.5 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 0.0
f1 (ppm)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145.75
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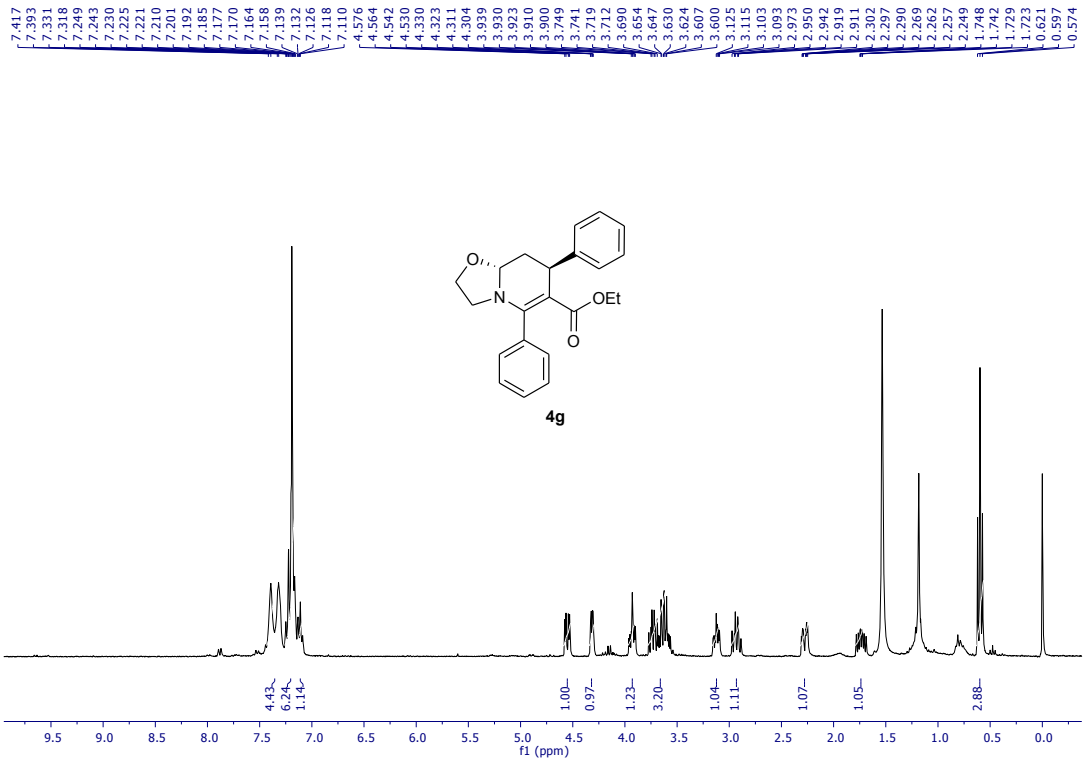
124.39
96.03
83.06
76.72
76.18
75.76
75.34
64.33
44.93
37.48
32.49
26.93
16.54
O
N
O^{t}Bu
CH$_3$
O
4f
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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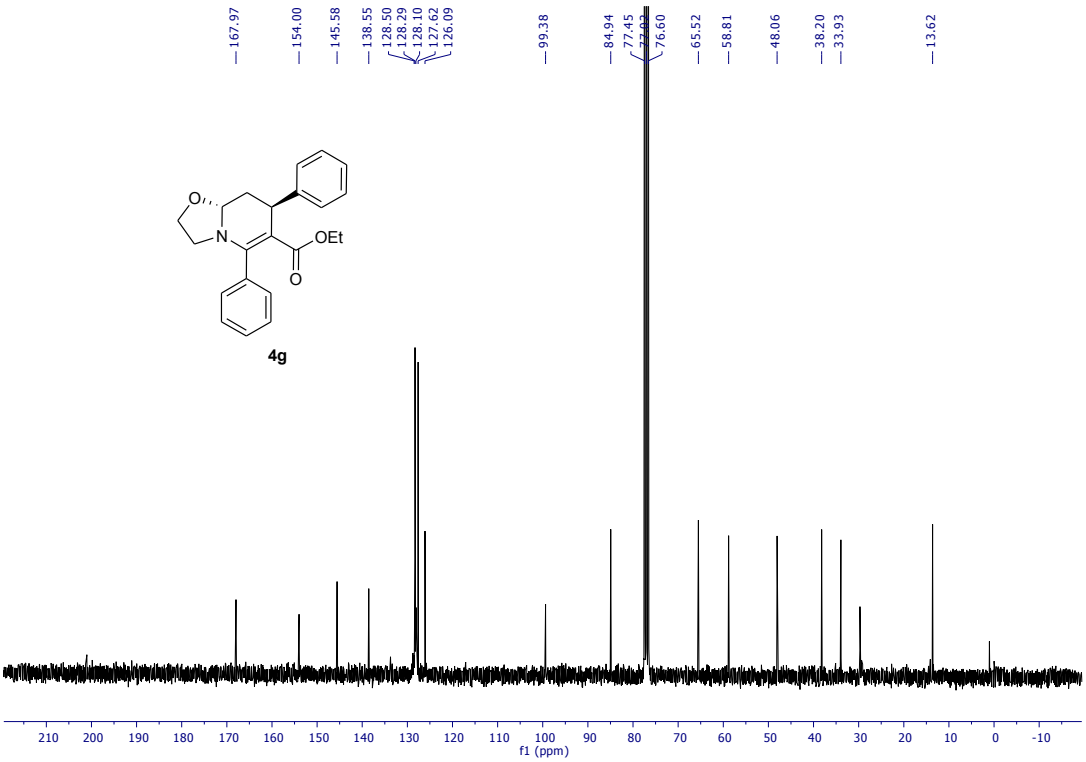
7.225
7.221
7.210
7.201
7.192
7.185
7.177
7.170
7.164
7.158
7.139
7.132
7.126
7.118
7.110
4.576
4.564
4.542
4.530
4.330
4.323
4.311
4.304
3.939
3.930
3.923
3.910
3.900
3.749
3.741
3.719
3.712
3.690
3.654
3.647
3.630
3.624
3.607
3.600
3.125
3.115
3.103
3.093
2.973
2.950
2.942
2.919
2.911
2.302
2.297
2.290
2.269
2.262
2.257
2.249
1.748
1.742
1.729
1.723
0.621
0.597
0.574
O
N
OEt
O
4g
4.43
6.24
1.14
1.00
0.97
1.23
3.20
1.04
1.11
1.07
1.05
2.88
f1 (ppm)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154.00
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138.55
128.50
128.29
128.10
127.62
126.09
99.38
84.94
77.45
76.60
65.52
58.81
48.06
38.20
33.93
13.62
O
N
OEt
O
4g
f1 (ppm)


7.442
7.434
7.430
7.424
7.416
7.408
7.402
7.372
7.368
7.359
7.347
7.339
7.330
7.325
7.318
7.306
7.288
7.282
7.273
7.263
7.254
7.227
7.213
7.204
7.150
7.126
4.548
4.536
4.513
4.501
4.418
4.399
4.274
4.264
4.255
4.245
4.229
4.215
3.910
3.902
3.878
3.874
3.848
3.611
3.596
3.583
3.574
3.563
2.338
2.330
2.326
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2.297
2.290
2.285
2.279
2.153
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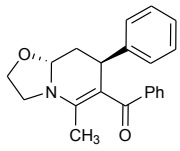
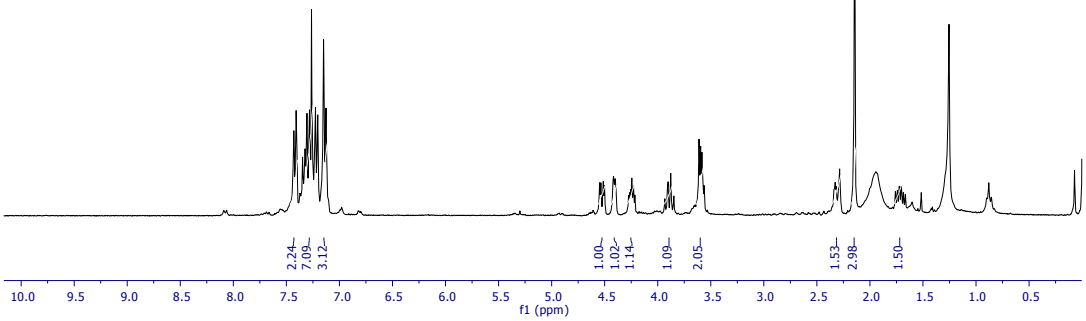
1.741
1.725
1.720
1.706
1.701
1.685
1.666
1.255


O
N
Ph
CH3
O

4h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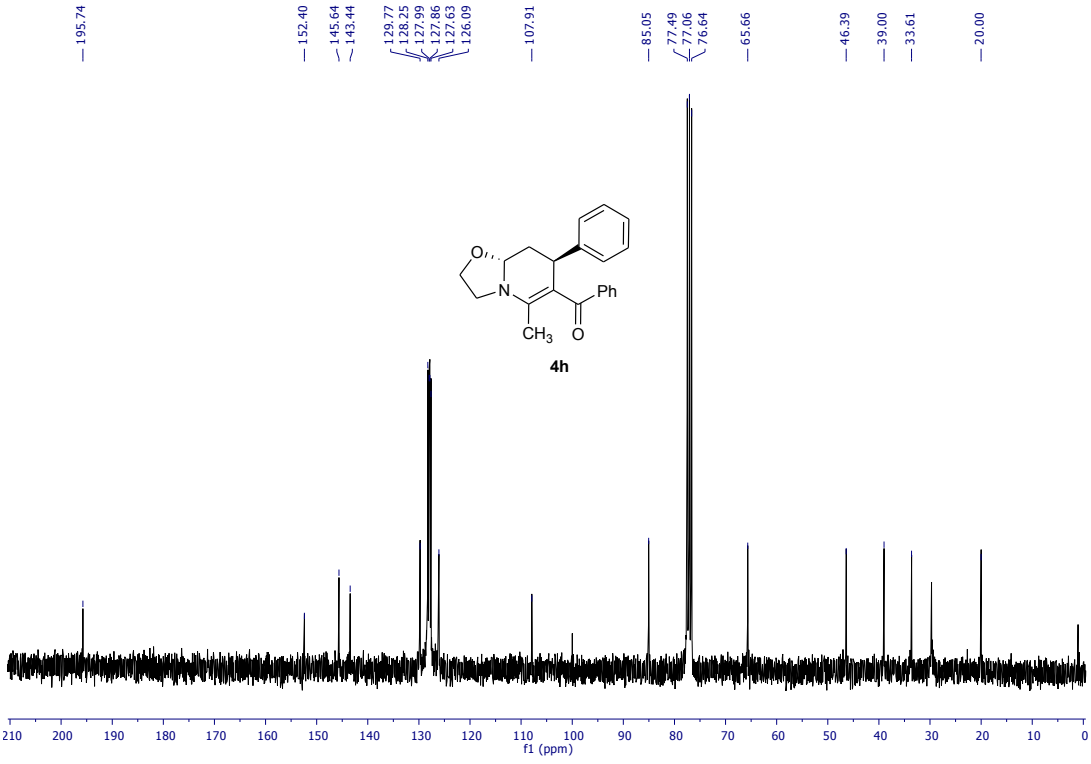
7.09
3.12
1.00
1.02
1.14
1.09
2.05
1.53
2.98
1.50
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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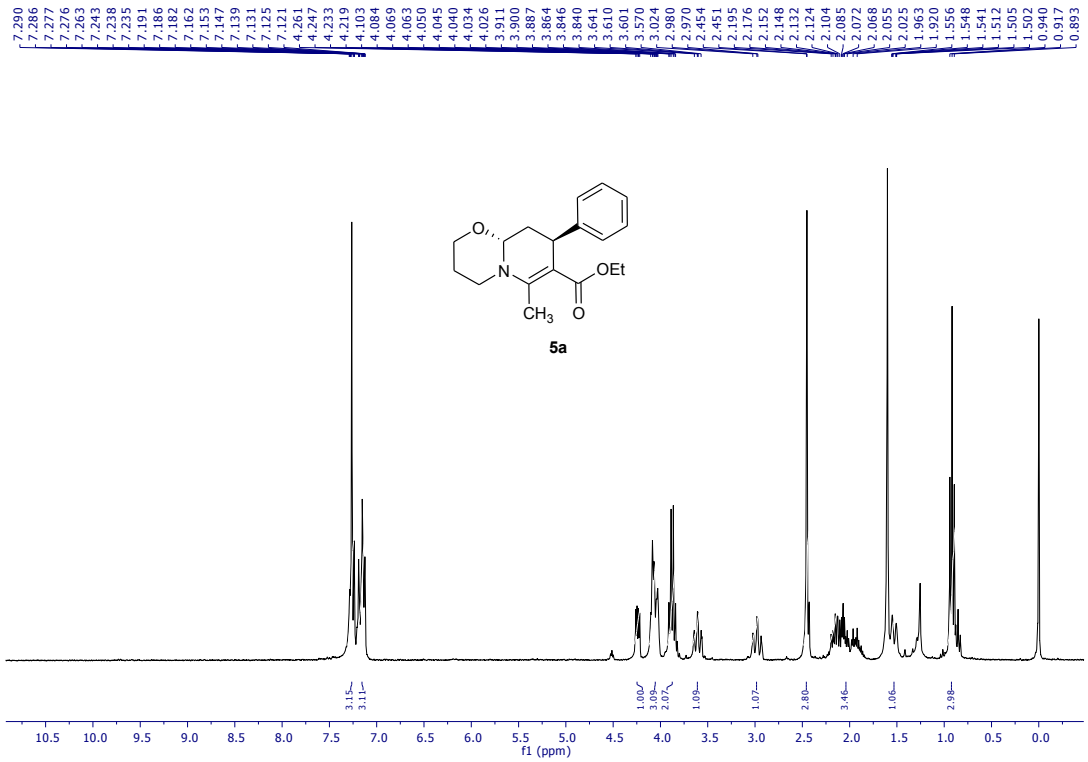
128.25
127.99
127.86
127.63
126.09
107.91
85.05
77.49
77.06
76.64
65.66
46.39
39.00
33.61
20.00
O
N
Ph
CH3
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4h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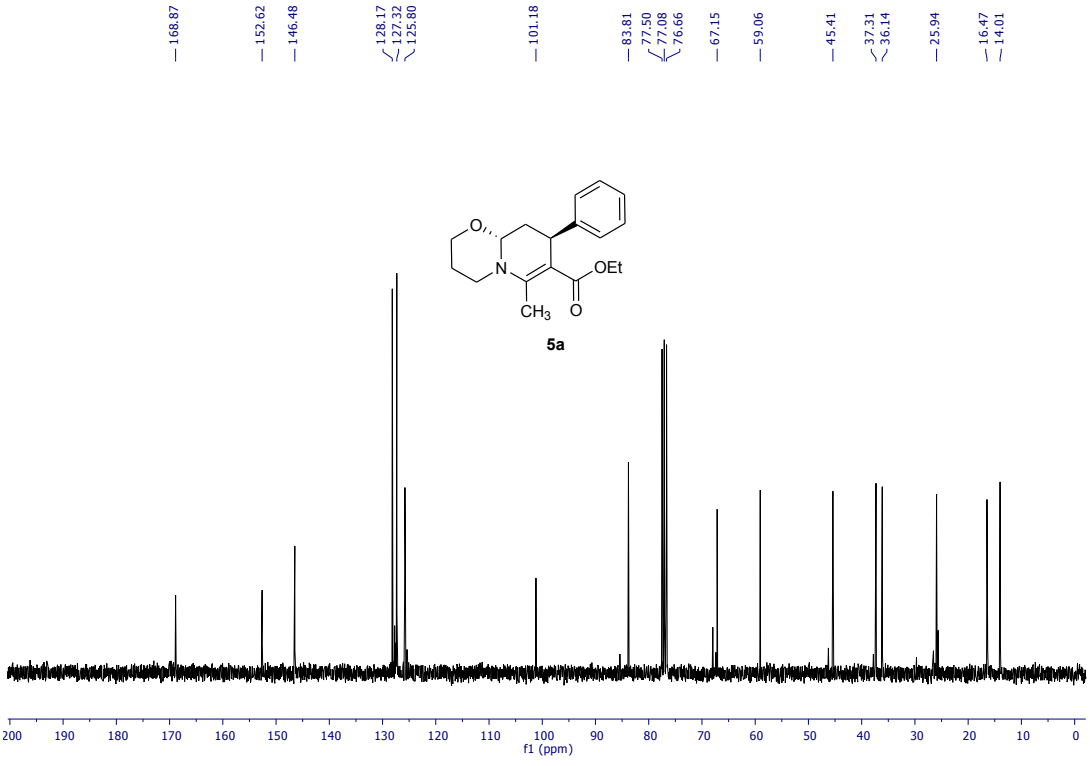
7.235
7.191
7.186
7.182
7.162
7.153
7.147
7.139
7.131
7.125
7.121
4.261
4.247
4.233
4.219
4.103
4.084
4.069
4.063
4.050
4.045
4.040
4.034
4.026
3.911
3.900
3.887
3.864
3.846
3.840
3.641
3.610
3.601
3.570
3.024
2.980
2.970
2.454
2.451
2.195
2.176
2.152
2.148
2.132
2.124
2.104
2.085
2.072
2.068
2.055
2.025
1.963
1.920
1.556
1.548
1.541
1.512
1.505
1.502
0.940
0.917
0.893
O
N
OEt
CH3
O
5a
3.15
3.11
1.00
3.05
2.07
1.09
1.07
2.80
3.46
1.06
2.98
10.5 10.0 9.5 9.0 8.5 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 0.0
f1 (ppm)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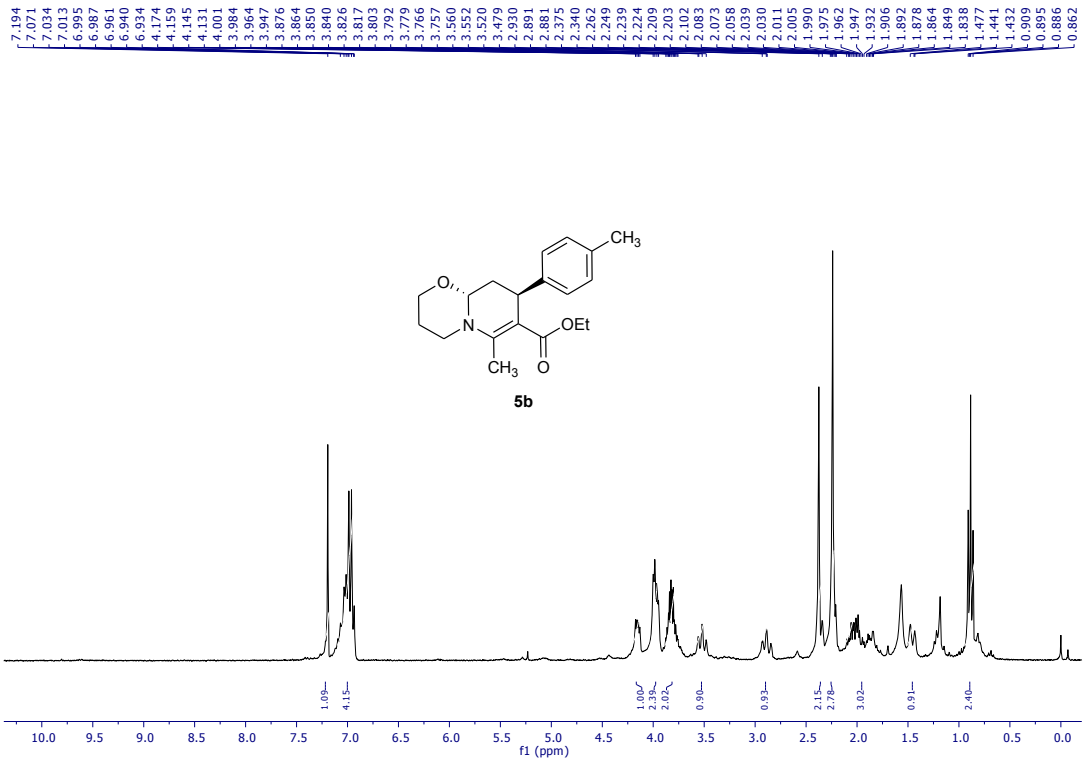
127.32
125.80
101.18
83.81
77.50
77.08
76.66
67.15
59.06
45.41
37.31
36.14
25.94
16.47
14.01
O
N
OEt
CH3
O
5a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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6.940
6.934
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4.145
4.131
4.001
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3.964
3.947
3.876
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3.850
3.840
3.826
3.817
3.803
3.792
3.779
3.766
3.757
3.560
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3.520
3.479
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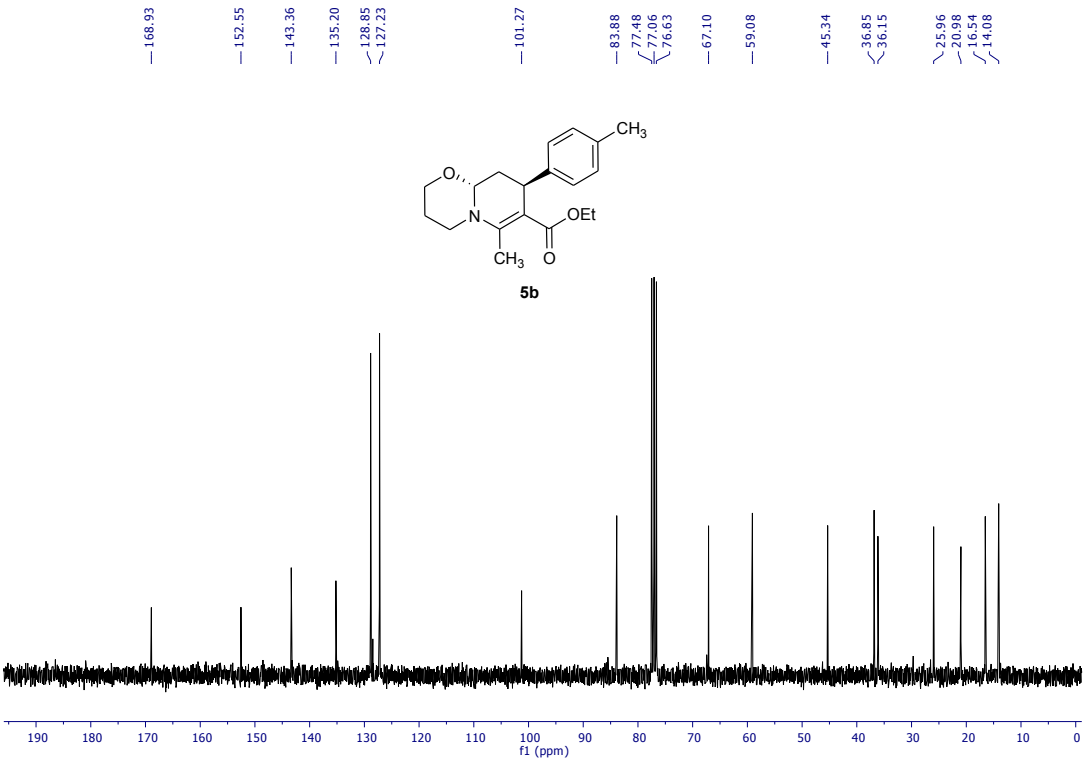
2.891
2.881
2.375
2.340
2.262
2.249
2.239
2.224
2.209
2.203
2.102
2.083
2.073
2.058
2.039
2.030
2.011
2.005
1.990
1.975
1.962
1.947
1.932
1.906
1.892
1.878
1.864
1.849
1.838
1.477
1.441
1.432
0.909
0.895
0.886
0.862
CH3
O
N
OEt
CH3
O
5b
1.09
4.15
1.00
2.39
2.02
0.90
0.93
2.15
2.78
3.02
0.91
2.40
10.0
9.5
9.0
8.5
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
0.0
f1 (ppm)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101.27
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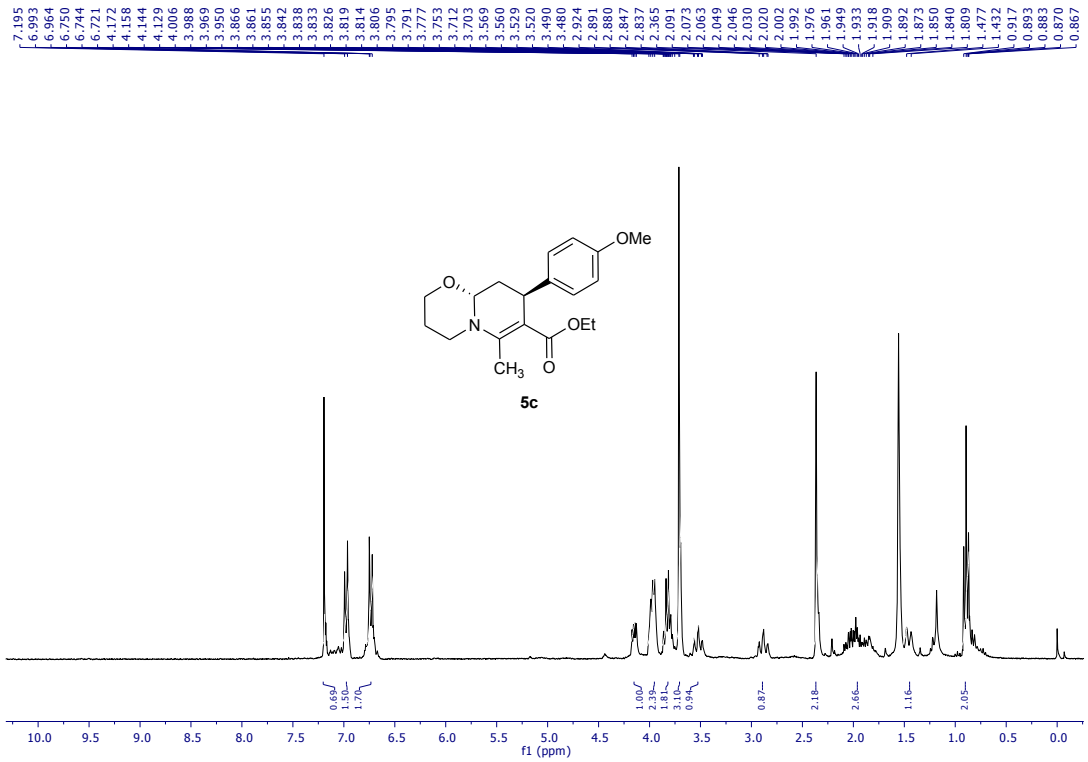
77.06
76.63
67.10
59.08
45.34
36.85
36.15
25.96
20.98
16.54
14.08
CH3
O
N
OEt
CH3
O
5b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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4.158
4.144
4.129
4.006
3.988
3.969
3.950
3.866
3.861
3.855
3.842
3.838
3.833
3.826
3.819
3.814
3.806
3.795
3.791
3.777
3.753
3.712
3.703
3.569
3.560
3.529
3.520
3.490
3.480
2.924
2.891
2.880
2.847
2.837
2.365
2.091
2.073
2.063
2.049
2.046
2.030
2.020
2.002
1.992
1.976
1.961
1.949
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1.918
1.909
1.892
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1.850
1.840
1.809
1.477
1.432
0.917
0.893
0.883
0.870
0.867
OMe
O
N
OEt
CH3
O
5c
0.69
1.50
1.70
1.00
2.39
1.81
3.10
0.94
0.87
2.18
2.66
1.16
2.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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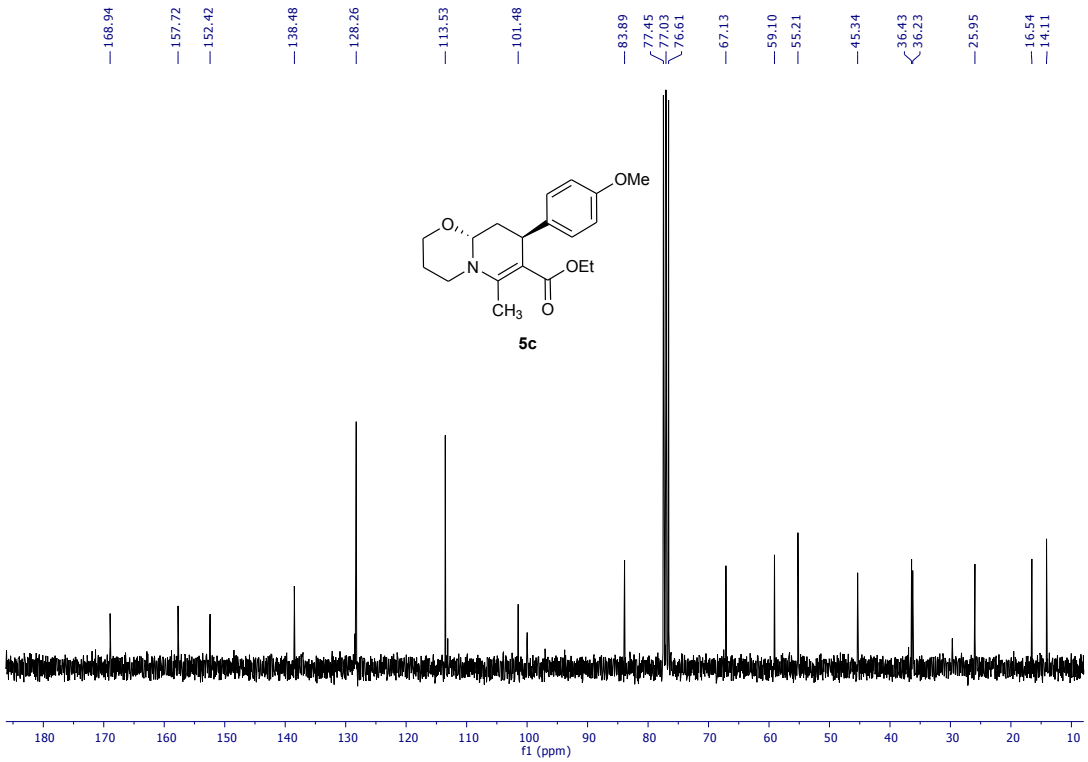
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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101.48
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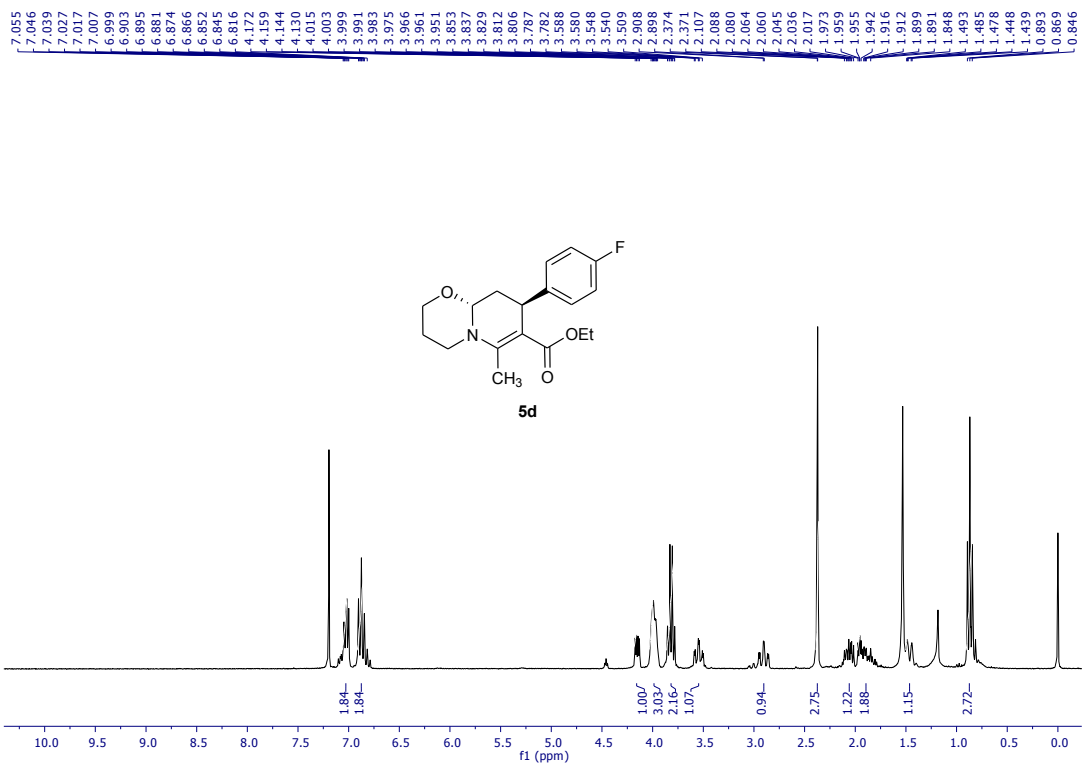
77.03
76.61
67.13
59.10
55.21
45.34
36.43
36.23
25.95
16.54
14.11
OMe
O
N
OEt
CH3
O
5c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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6.852
6.845
6.816
4.172
4.159
4.144
4.130
4.015
4.003
3.999
3.991
3.983
3.975
3.966
3.961
3.951
3.853
3.837
3.829
3.812
3.806
3.787
3.782
3.588
3.580
3.548
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3.509
2.908
2.898
2.374
2.371
2.107
2.088
2.080
2.064
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2.045
2.036
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1.955
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1.891
1.848
1.493
1.485
1.478
1.448
1.439
0.893
0.869
0.846
F
O
N
OEt
CH3
O
5d
1.84
1.84
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1.07
0.94
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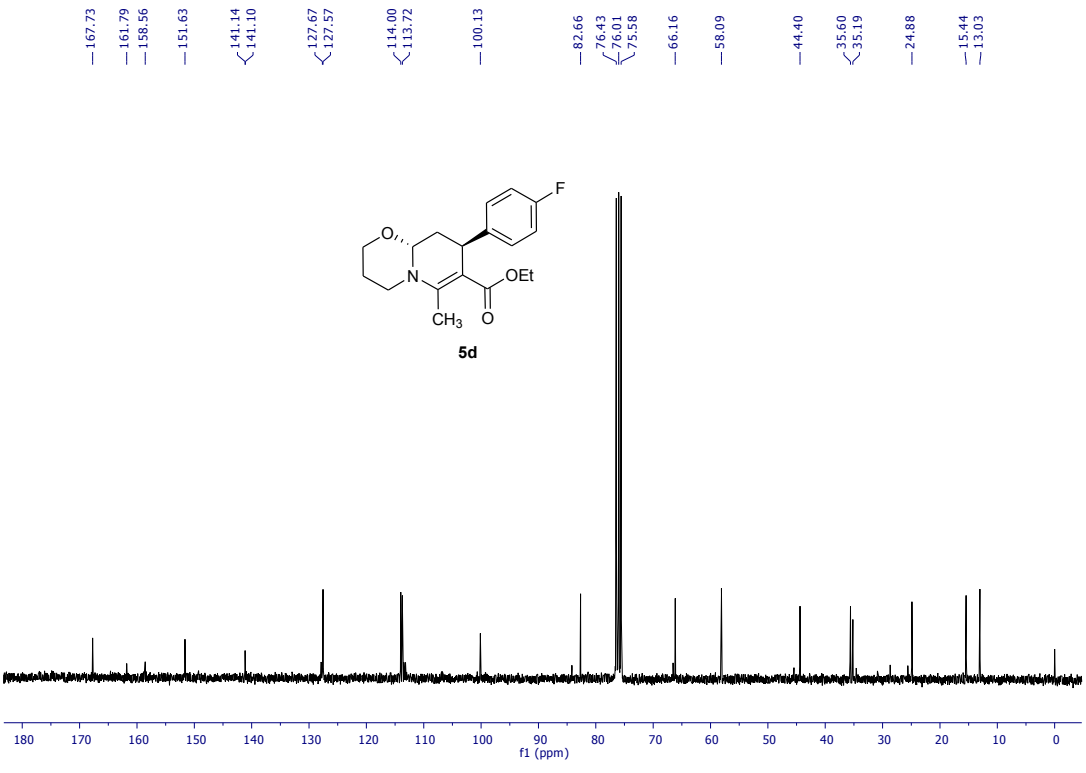
1.22
1.88
1.15
2.72
f1 (ppm)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127.57
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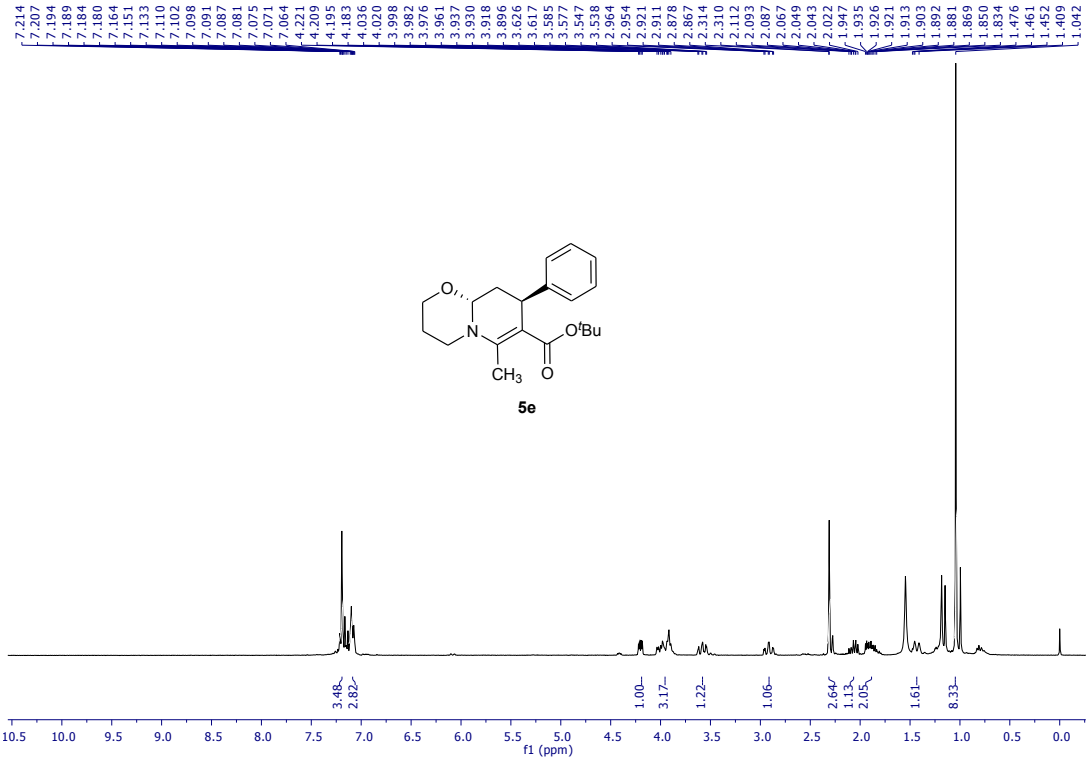
113.72
100.13
82.66
76.43
76.01
75.58
66.16
58.09
44.40
35.60
35.19
24.88
15.44
13.03
F
O
N
OEt
CH3
O
5d
f1 (ppm)


7.214
7.207
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7.091
7.087
7.081
7.075
7.071
7.064
4.221
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3.577
3.547
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2.921
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2.878
2.867
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2.087
2.067
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2.043
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1.935
1.926
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1.913
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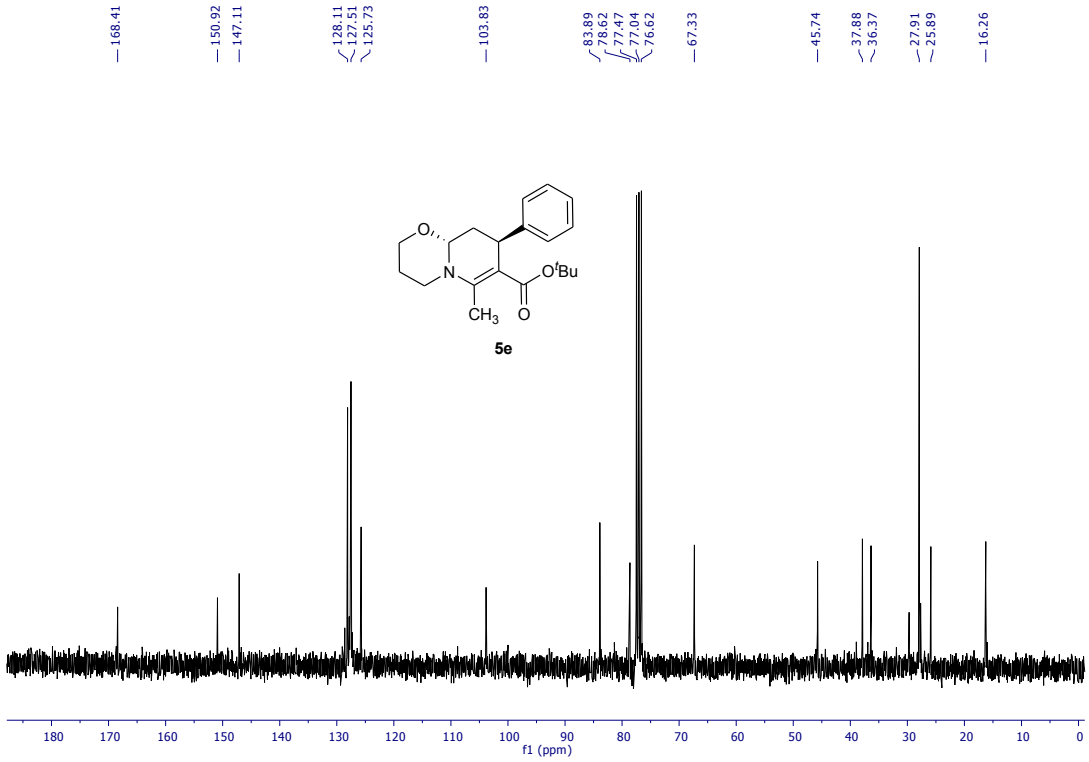
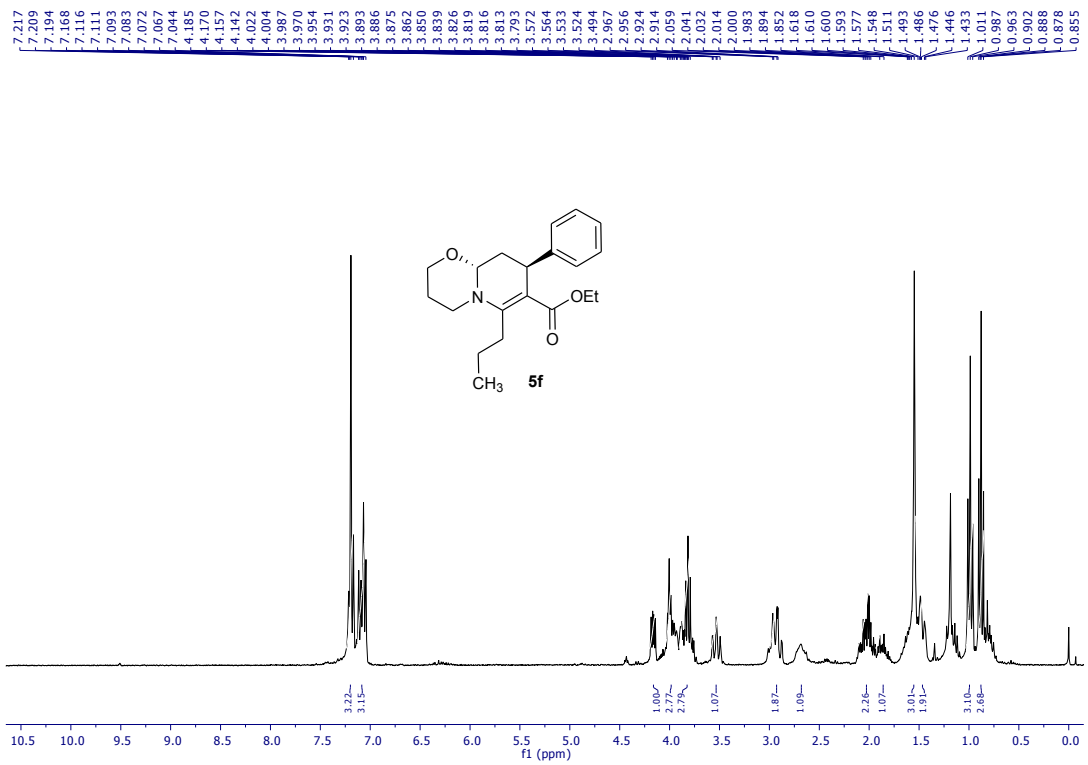
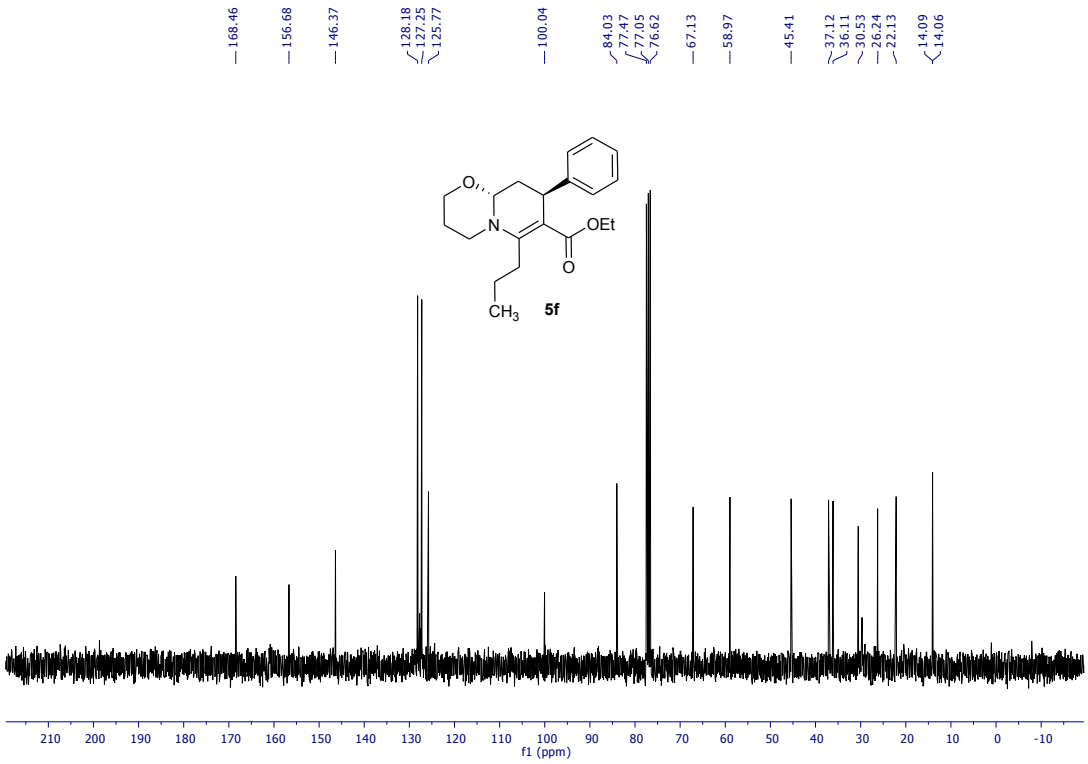
1.409
1.042
O
N
O^{t}Bu
CH$_3$
O
5e
3.48
2.82
1.00
3.17
1.22
1.06
2.64
1.13
2.05
1.61
8.33
f1 (ppm)


168.41
150.92
147.11
128.11
127.51
125.73
103.83
83.89
78.62
77.47
77.04
76.62
67.33
45.74
37.88
36.37
27.91
25.89
16.26
O
N
O^{t}Bu
CH$_3$
O
5e
f1 (ppm)


7.217
7.209
7.194
7.168
7.116
7.111
7.093
7.083
7.072
7.067
7.044
4.185
4.170
4.157
4.142
4.022
4.004
3.987
3.970
3.954
3.931
3.923
3.893
3.886
3.875
3.862
3.850
3.839
3.826
3.819
3.816
3.813
3.793
3.572
3.564
3.533
3.524
3.494
2.967
2.956
2.924
2.914
2.059
2.041
2.032
2.014
2.000
1.983
1.894
1.852
1.618
1.610
1.600
1.593
1.577
1.548
1.511
1.493
1.486
1.476
1.446
1.433
1.011
0.987
0.963
0.902
0.888
0.878
0.855
O
N
OEt
O
CH3
5f
3.22
3.15
1.00
2.77
2.79
1.07
1.87
1.09
2.26
1.07
3.01
1.91
3.10
2.68
10.5 10.0 9.5 9.0 8.5 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 0.0
f1 (ppm)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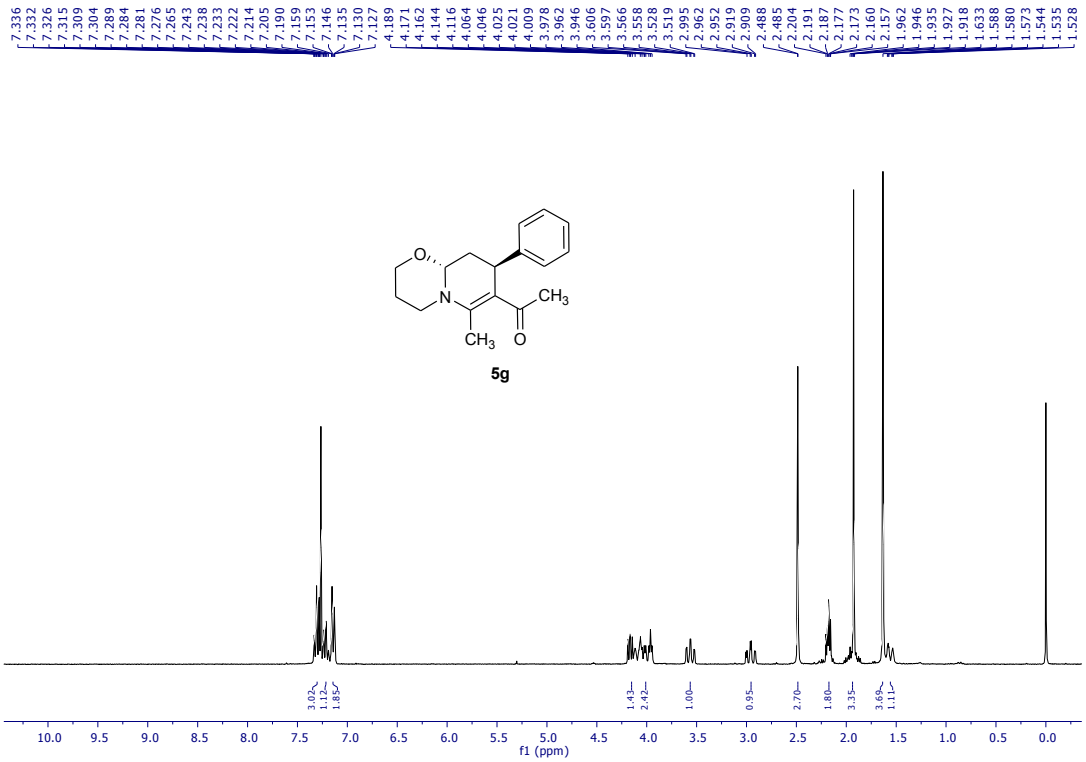
156.68
146.37
128.18
127.25
125.77
100.04
84.03
77.47
77.05
76.62
67.13
58.97
45.41
37.12
36.11
30.53
26.24
22.13
14.09
14.06
O
N
OEt
O
CH3
5f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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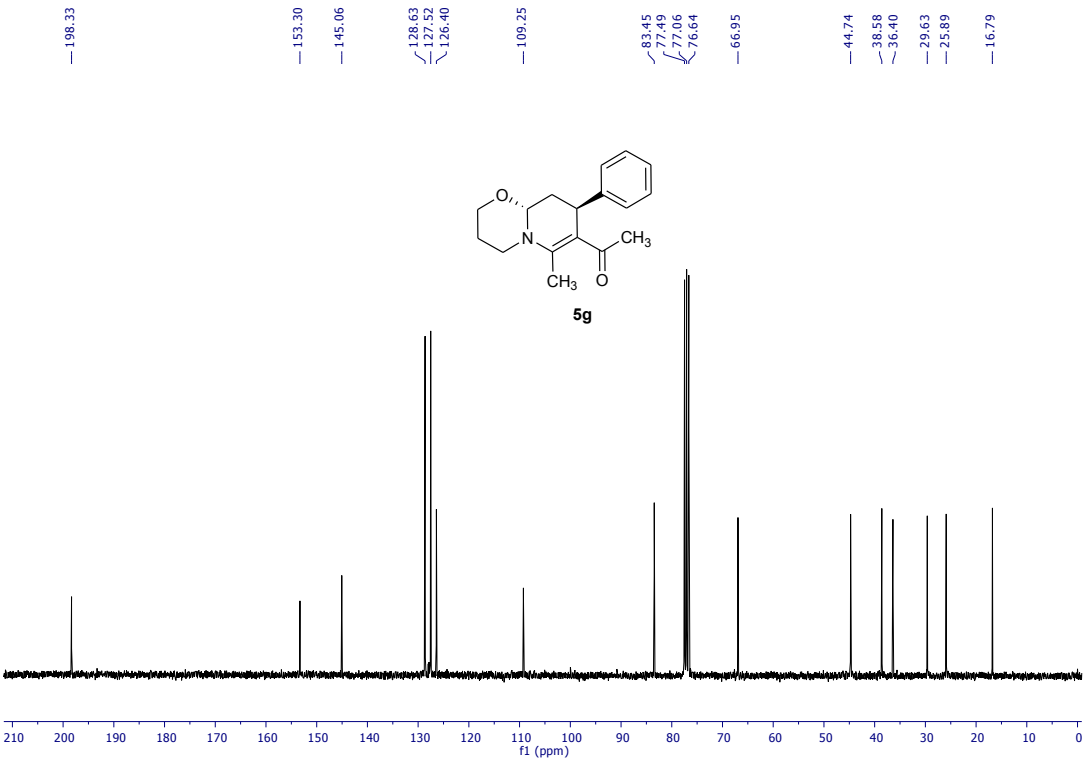
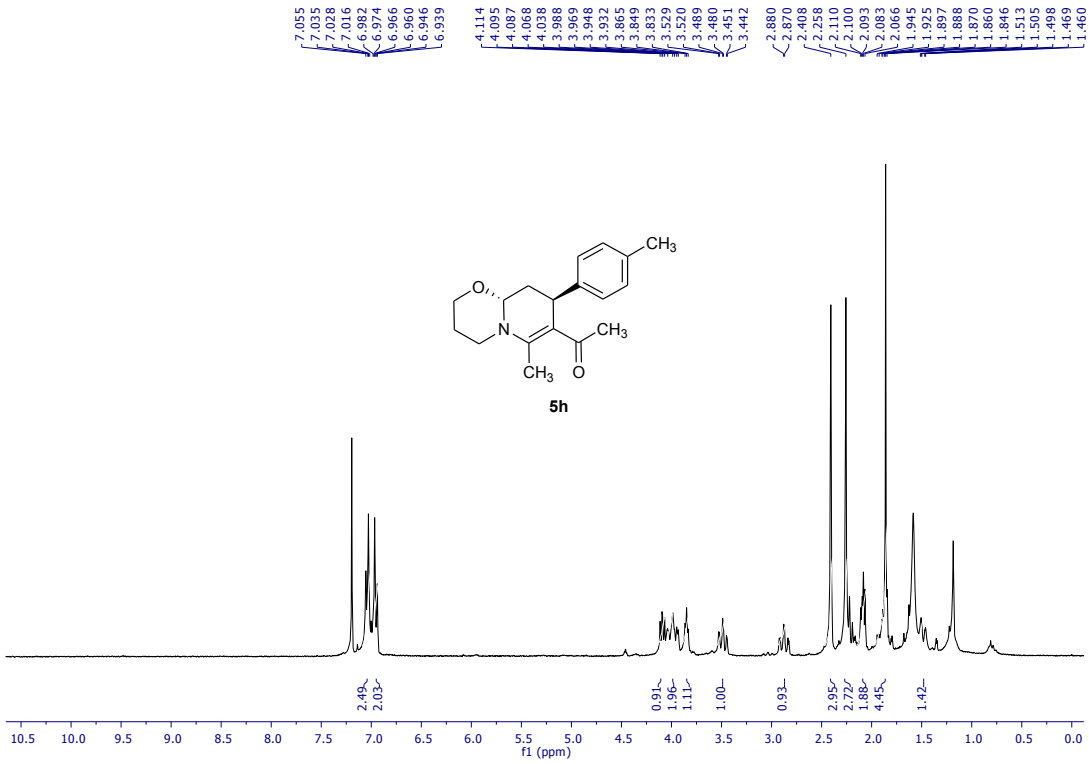
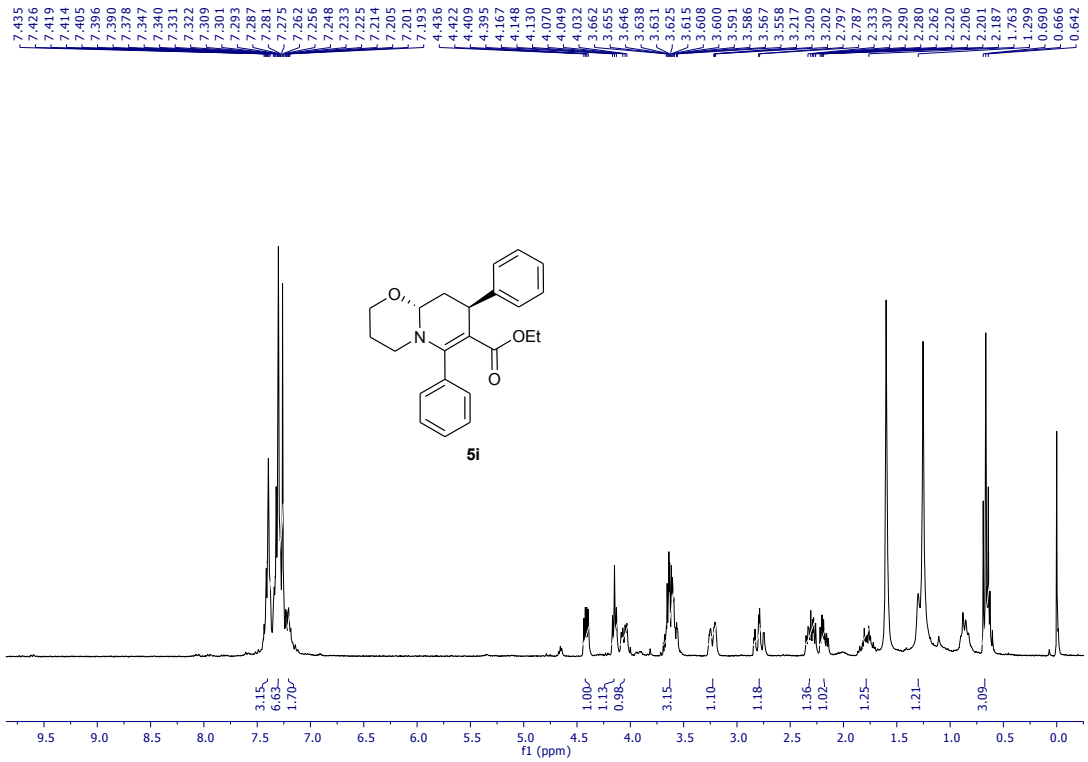
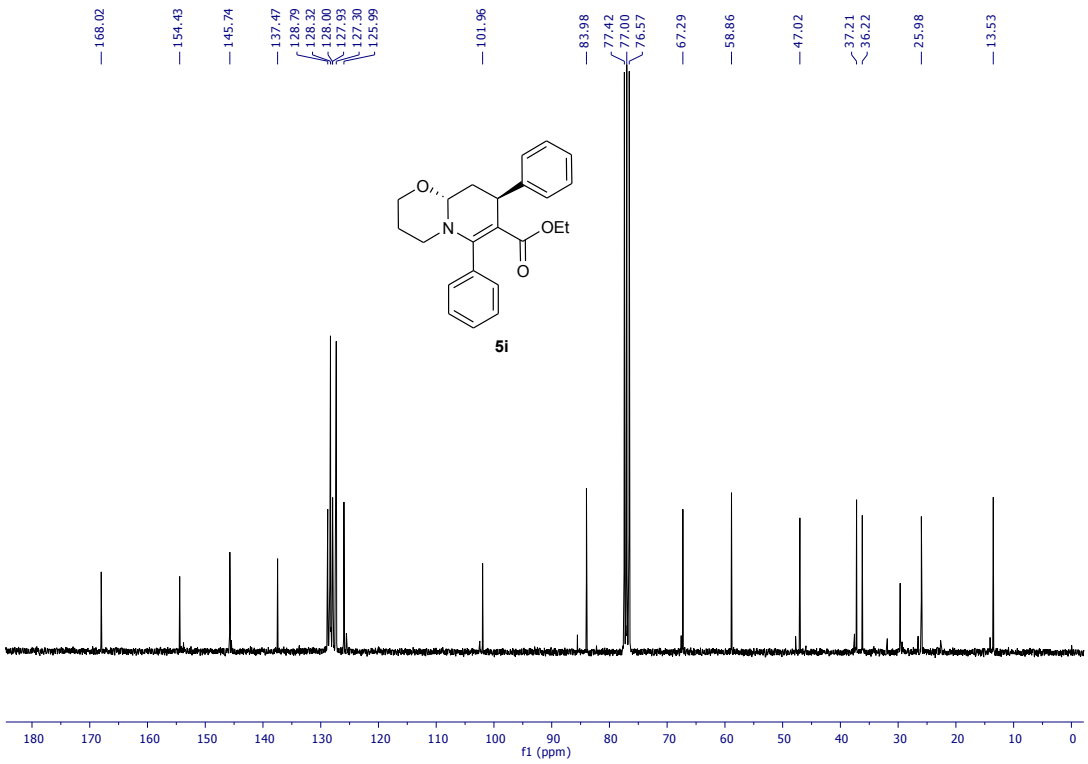
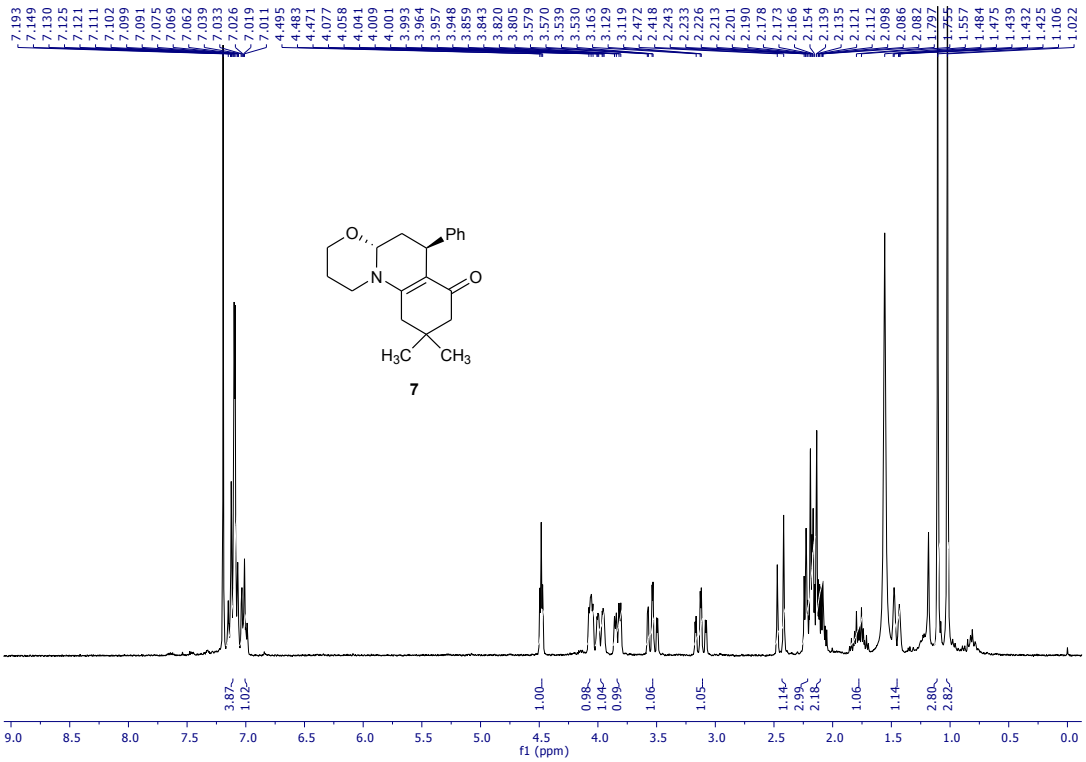
7.332
7.326
7.315
7.309
7.304
7.289
7.284
7.281
7.276
7.265
7.243
7.238
7.233
7.222
7.214
7.205
7.190
7.159
7.153
7.146
7.135
7.130
7.127
4.189
4.171
4.162
4.144
4.116
4.064
4.046
4.025
4.021
4.009
3.978
3.962
3.946
3.606
3.597
3.566
3.558
3.528
3.519
2.995
2.962
2.952
2.919
2.909
2.488
2.485
2.204
2.191
2.187
2.177
2.173
2.160
2.157
1.962
1.946
1.935
1.927
1.918
1.633
1.588
1.580
1.573
1.544
1.535
1.528
O
N
CH3
CH3
O
5g
3.02
1.12
1.85
1.43
2.42
1.00
0.95
2.70
1.80
3.35
3.60
1.11
10.0 9.5 9.0 8.5 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 0.0
f1 (ppm)

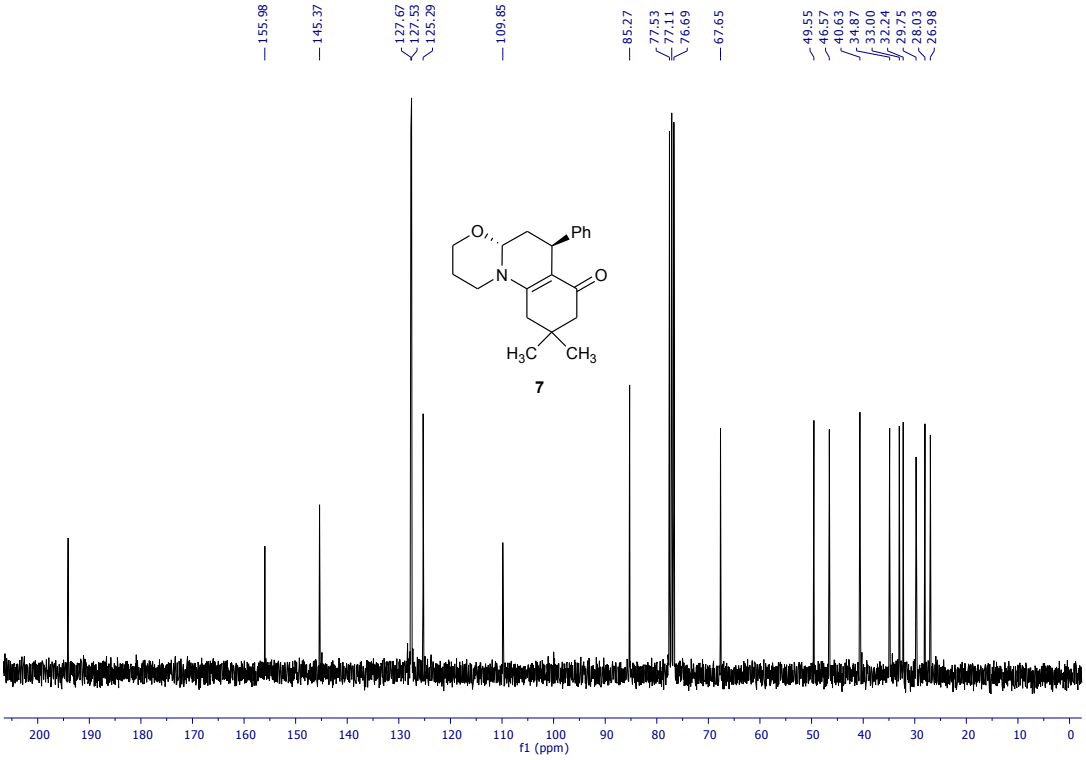

198.33
153.30
145.06
128.63
127.52
126.40
109.25
83.45
77.49
77.06
76.64
66.95
44.74
38.58
36.40
29.63
25.89
16.79
O
N
CH3
CH3
O
5g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

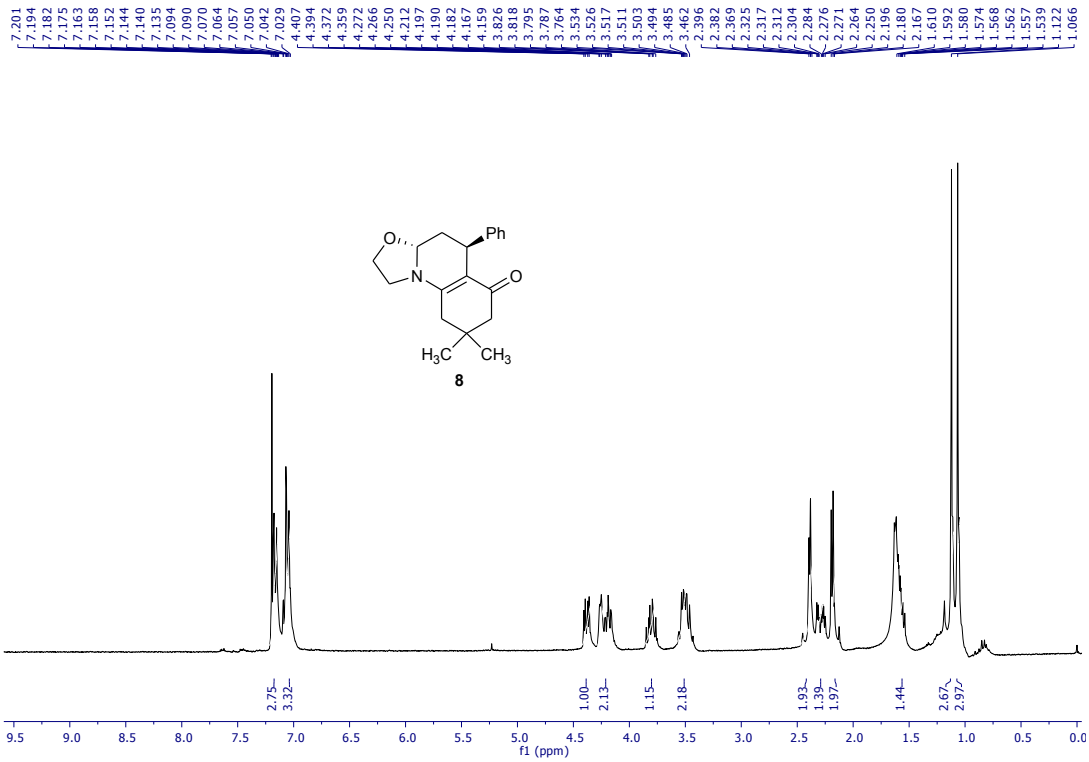

7.055
7.035
7.028
7.016
6.982
6.974
6.966
6.960
6.946
6.939
4.114
4.095
4.087
4.068
4.038
3.988
3.969
3.948
3.932
3.865
3.849
3.833
3.529
3.520
3.489
3.480
3.451
3.442
2.880
2.870
2.408
2.258
2.110
2.100
2.093
2.083
2.066
1.945
1.925
1.897
1.888
1.870
1.860
1.846
1.513
1.505
1.498
1.469
1.460
CH3
O
N
CH3
CH3
O
5h
2.49
2.03
0.91
1.96
1.11
1.00
0.93
2.95
2.72
1.88
4.45
1.42
10.5
10.0
9.5
9.0
8.5
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
0.0
f1 (ppm)

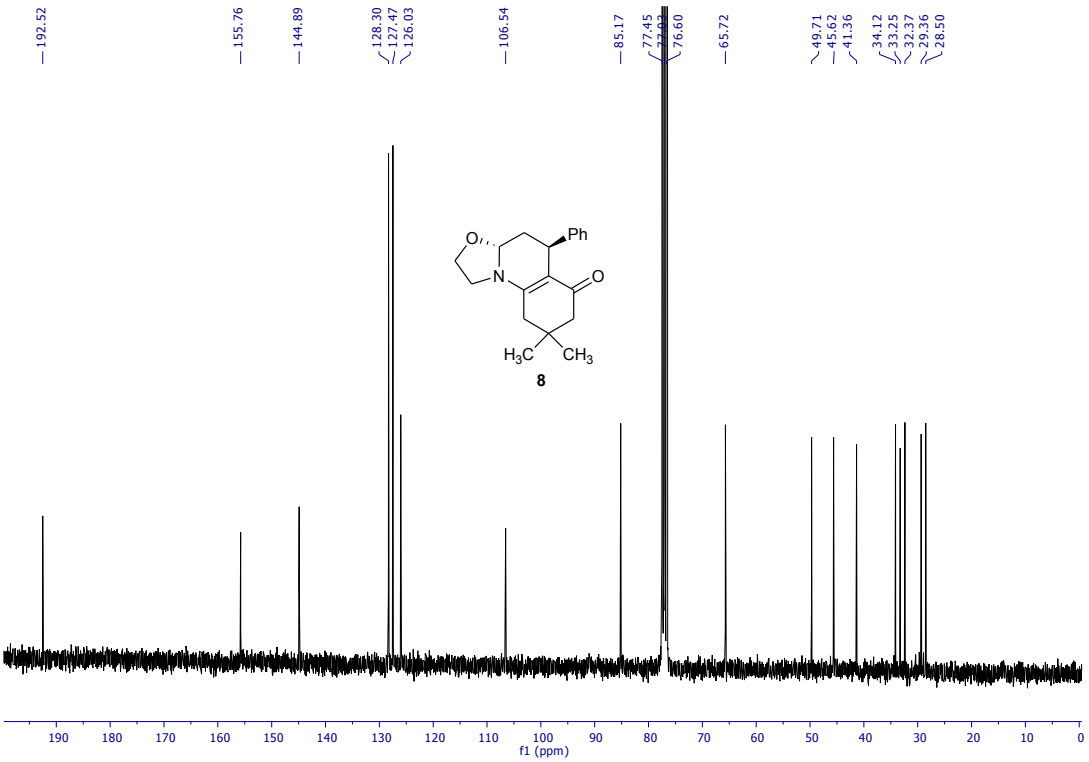

7.435
7.426
7.419
7.414
7.405
7.396
7.390
7.378
7.347
7.340
7.331
7.322
7.309
7.301
7.293
7.287
7.281
7.275
7.262
7.256
7.248
7.233
7.225
7.214
7.205
7.201
7.193
4.436
4.422
4.409
4.395
4.167
4.148
4.130
4.070
4.049
4.032
3.662
3.655
3.646
3.638
3.631
3.625
3.615
3.608
3.600
3.591
3.586
3.567
3.558
3.217
3.209
3.202
2.797
2.787
2.333
2.307
2.290
2.280
2.262
2.220
2.206
2.201
2.187
1.763
1.299
0.690
0.666
0.642
O
N
OEt
O
5i
3.15
6.63
1.70
1.00
1.13
0.98
3.15
1.10
1.18
1.36
1.02
1.25
1.21
3.09
f1 (ppm)

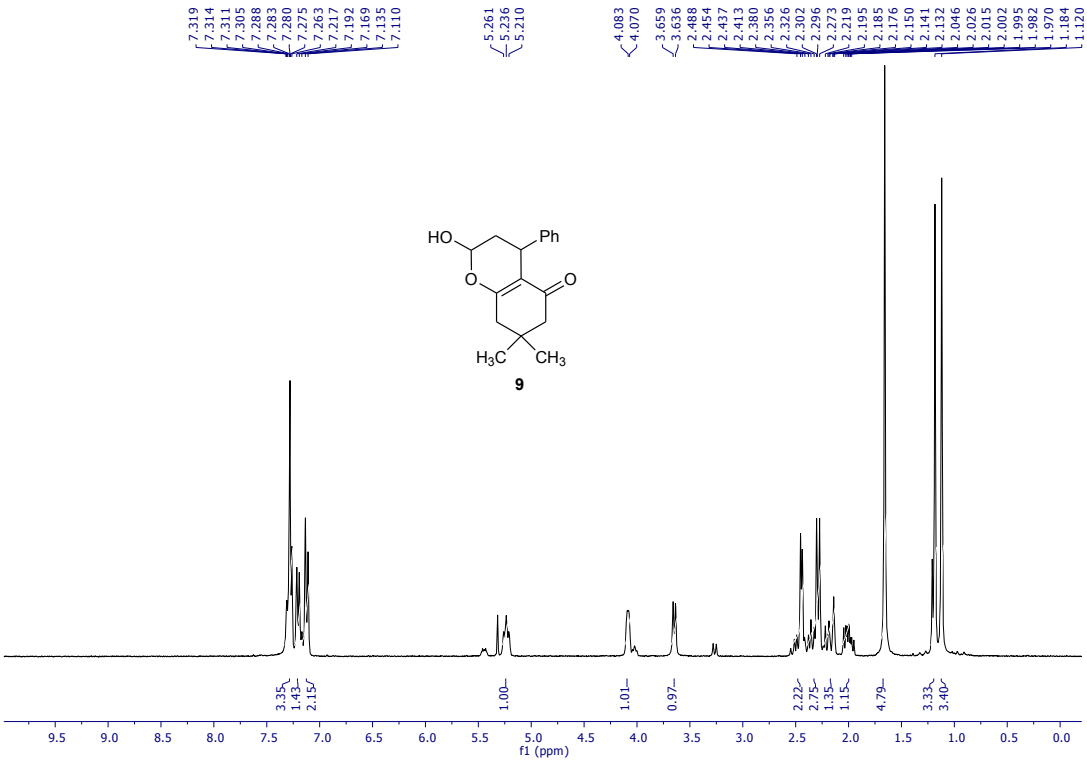

168.02
154.43
145.74
137.47
128.79
128.32
128.00
127.93
127.30
125.99
101.96
83.98
77.42
77.00
76.57
67.29
58.86
47.02
37.21
36.22
25.98
13.53
O
N
OEt
O
5i
f1 (ppm)


7.193 7.149 7.130 7.125 7.121 7.111 7.102 7.099 7.091 7.075 7.069 7.062 7.039 7.033 7.026 7.019 7.011 4.495 4.483 4.471 4.077 4.058 4.041 4.009 4.001 3.993 3.964 3.957 3.948 3.859 3.843 3.820 3.805 3.579 3.570 3.539 3.530 3.163 3.129 3.119 2.472 2.418 2.243 2.233 2.226 2.213 2.201 2.190 2.178 2.173 2.166 2.154 2.139 2.135 2.121 2.112 2.098 2.086 2.082 1.797 1.755 1.557 1.484 1.475 1.439 1.432 1.425 1.106 1.022
O
N
Ph
O
H3C
CH3
7
3.87 1.02 1.00 0.98 1.04 0.99 1.06 1.05 1.14 2.99 2.18 1.06 1.14 2.80 2.82
9.0 8.5 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 0.0
f1 (ppm)


155.98 145.37 127.67 127.53 125.29 109.85 85.27 77.53 77.11 76.69 67.65 49.55 46.57 40.63 34.87 33.00 32.24 29.75 28.03 26.98
O
N
Ph
O
H3C
CH3
7
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.201
7.194
7.182
7.175
7.163
7.158
7.152
7.144
7.140
7.135
7.094
7.090
7.070
7.064
7.057
7.050
7.042
7.029
4.407
4.394
4.372
4.359
4.272
4.266
4.250
4.212
4.197
4.190
4.182
4.167
4.159
3.826
3.818
3.795
3.787
3.764
3.534
3.526
3.517
3.511
3.503
3.494
3.485
3.462
2.396
2.382
2.369
2.325
2.317
2.312
2.304
2.284
2.276
2.271
2.264
2.250
2.196
2.180
2.167
1.610
1.592
1.580
1.574
1.568
1.562
1.557
1.539
1.122
1.066
O
N
Ph
O
H3C
CH3
8
2.75
3.32
1.00
2.13
1.15
2.18
1.93
1.39
1.97
1.44
2.67
2.97
f1 (ppm)


192.52
155.76
144.89
128.30
127.47
126.03
106.54
85.17
77.45
77.03
76.60
65.72
49.71
45.62
41.36
34.12
33.25
32.37
29.36
28.50
O
N
Ph
O
H3C
CH3
8
f1 (ppm)


7.319
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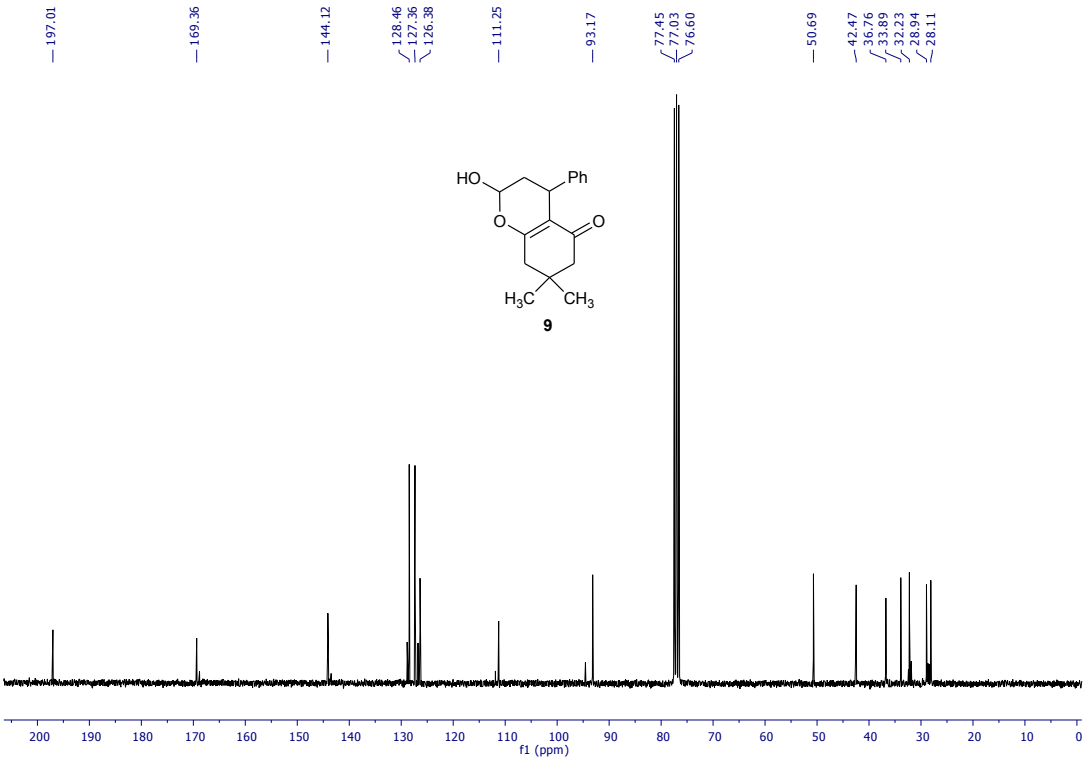
7.311
7.305
7.288
7.283
7.280
7.275
7.263
7.217
7.192
7.169
7.135
7.110
5.261
5.236
5.210
4.083
4.070
3.659
3.636
2.488
2.454
2.437
2.413
2.380
2.356
2.326
2.302
2.296
2.273
2.219
2.195
2.185
2.176
2.150
2.141
2.132
2.046
2.026
2.015
2.002
1.995
1.982
1.970
1.184
1.120
HO
Ph
O
O
H3C
CH3
9
3.35
1.43
2.15
1.00
1.01
0.97
2.22
2.75
1.35
1.15
4.79
3.33
3.40
f1 (ppm)


197.01
169.36
144.12
128.46
127.36
126.38
111.25
93.17
77.45
77.03
76.60
50.69
42.47
36.76
33.89
32.23
28.94
28.11
HO
Ph
O
O
H3C
CH3
9
f1 (ppm)