A General [3+2+1] Annulation Strategy for the Preparation of Pyridine N-Oxides

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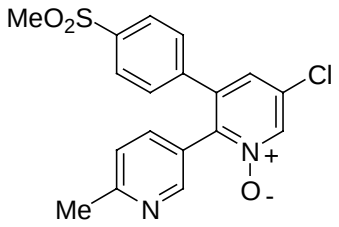
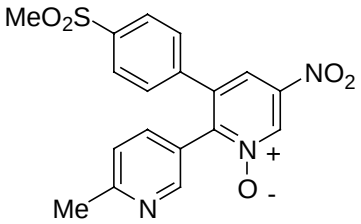
Characterization Data

Data for Table 1, Entry 1.



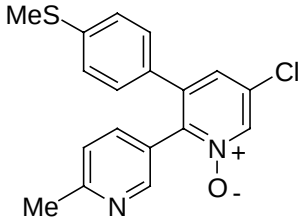
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J*= 2 Hz, 1H), 8.68 (d, *J*= 2 Hz, 1H), 8.11 (d, *J*= 8 Hz, 1H), 7.90-7.93 (m, 2H), 7.75 (d, *J*= 2 Hz, 1H), 7.60-7.67 (m, 3H), 3.24 (s, 3H), 2.53 (s, 3H).); ^{13}C NMR (100 MHz $CDCl_3$) δ 157.1, 148.0, 143.7, 142.2, 141.2, 141.1, 139.7, 138.2, 131.9, 130.7, 127.2, 126.9, 125.8, 124.0, 43.2, 21.9. LC-MS (M^++1) 376.1.

Data for Table 1, Entry 2.



^{1}H NMR (400 MHz, $CDCl_3$) δ 9.18 (d, *J*= 2 Hz, 1H), 8.25 (d, *J*= 2 Hz, 1H), 8.04 (d, *J*= 2 Hz, 1H), 7.85-7.88 (m, 2H), 7.67 (dd, *J*= 8, 2 Hz, 1H), 7.31-7.38 (m, 2H), 7.20 (d, *J*= 8 Hz, 1H), 3.04 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (100 MHz $CDCl_3$) δ 160.5, 151.2, 150.0, 145.1, 141.1, 140.9, 138.5, 135.5, 130.2, 129.5, 128.1, 127.1, 122.9, 120.7, 44.3, 24.5.

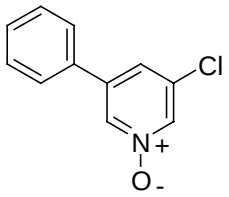
Data for Table 1, Entry 3.



^{1}H NMR (400 MHz, d_6-DMF) δ 8.39 (d, *J*= 2 Hz, 1H), 8.27 (d, *J*= 2 Hz, 1H), 7.62 (dd, *J*= 8, 2 Hz, 1H), 7.31 (d, *J*= 2 Hz, 1H), 7.14 (d, *J*= 8 Hz, 1H), 7.07-7.11 (m, 2H), 6.95-6.98 (m, 2H), 2.53 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz $CDCl_3$) δ 158.8, 150.3,

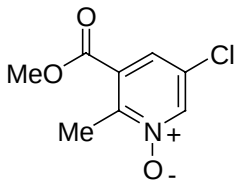
144.5, 140.5, 140.0, 138.6, 137.7, 132.1, 131.6, 129.4, 127.4, 125.8, 123.9, 122.5, 24.3, 15.0. LC-MS (M^++1) 345.0.

Data for Compound 7.



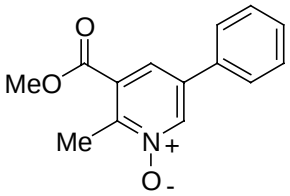
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.36 (s, 1H), 8.22 (s, 1H), 7.42-7.56 (m, 6H); ^{13}C NMR (75 MHz $CDCl_3$) δ 140.5, 136.9, 136.2, 134.1, 133.3, 130.0, 129.6, 127.0, 125.2. LC-MS (M^++1) 206.1.

Data for Table 2, Entry 1.



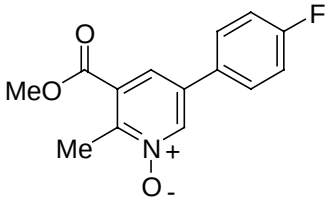
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J*= 2 Hz, 1H), 7.69 (d, *J*= 2 Hz, 1H), 3.95 (s, 3H), 2.72 (s, 3H); ^{13}C NMR (100 MHz $CDCl_3$) δ 164.3, 150.0, 140.6, 129.6, 129.4, 126.6, 53.2, 14.5. LC-MS (M^++1) 202.1.

Data for Table 2, Entry 2.



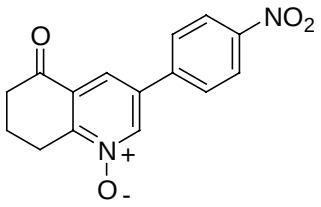
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (s, 1H), 7.91 (s, 1H), 8.36 (s, 1H), 7.39-7.55 (m, 5H), 3.94 (s, 3H), 2.80 (s, 3H); ^{13}C NMR (75 MHz $CDCl_3$) δ 165.4, 149.4, 139.7, 136.4, 134.5, 129.5, 129.4, 129.4, 126.8, 125.7, 53.0, 14.8. LC-MS (M^++1) 244.1.

Data for Table 2, Entry 3.



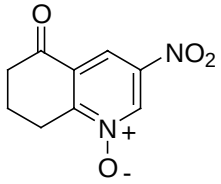
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.58 (s, 1H), 7.84 (s, 1H), 8.36 (s, 1H), 7.46-7.52 (m, 2H), 7.11-7.20 (m, 2H), 3.95 (s, 3H), 2.80 (s, 3H); ^{13}C NMR (75 MHz $CDCl_3$) δ 165.2, 149.4, 139.5, 135.4, 129.5, 128.7, 128.6, 125.3, 116.6, 116.4, 53.0, 14.7. LC-MS (M^++1) 262.1.

Data for Compound 11.



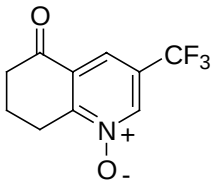
^{1}H NMR (400 MHz, d_6-DMF) δ 9.03 (d, *J*= 2 Hz, 1H), 8.58 (d, *J*= 8 Hz, 2H), 8.28 (d, *J*= 8 Hz, 2H), 8.24 (d, *J*= 2 Hz, 1H), 3.20-3.25 (m, 2H), 2.96-3.00 (m, 2H), 2.08-2.15 (m, 2H); ^{13}C NMR (100 MHz $CDCl_3$) δ 150.7, 148.9, 148.1, 142.2, 136.7, 134.2, 131.1, 128.5, 124.3, 117.5, 23.6, 21.6, 19.3.

Data for Compound 12.



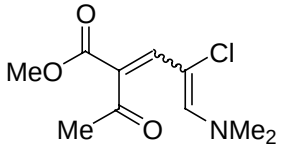
^{1}H NMR (400 MHz, d_6-DMF) δ 8.83 (d, *J*= 2 Hz, 1H), 8.42 (d, *J*= 2 Hz, 1H), 3.37-3.42 (m, 2H), 2.88-2.96 (m, 2H), 1.78-1.83 (m, 2H); ^{13}C NMR (100 MHz $CDCl_3$) δ 160.0, 154.5, 149.3, 133.3, 131.4, 114.9, 24.1, 21.4, 18.6.

Data for Compound 13.



^{1}H NMR (400 MHz, d_6-DMF) δ 8.88 (d, *J*= 2 Hz, 1H), 8.45 (d, *J*= 2 Hz, 1H), 3.25-3.32 (m, 2H), 2.85-2.93 (m, 2H), 1.90-1.96 (m, 2H); ^{13}C NMR (100 MHz $CDCl_3$) δ 155.5, 150.5, 149.1, 134.3, 131.2, 116.1, 23.9, 21.4, 18.9.

Data for Compound 14.



~2:1 mixture of isomers: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (s, 0.5H), 6.83 (s, 1H), 6.73 (s, 0.5H), 5.64 (s, 1H), 3.78 (s, 1.5H), 3.69 (s, 3H), 3.18 (s, 3H), 2.47 (s, 6H), 2.34 (s, 3H), 2.18 (s, 1.5H); ^{13}C NMR (400 MHz, $CDCl_3$) 192.9, 170.0, 165.8, 149.0, 143.3, 125.6 (broad), 121.0, 112.9 (broad), 97.5, 51.8, 51.2, 43.2, 38.7, 26.4, 20.5. Anal. Calc'd for $C_{10}H_{14}ClNO_3$ C, 51.84; H, 6.09; N, 6.05. Found: C, 51.97; H, 6.01; N, 5.93.