Supporting Information to Accompany

“Transformation of \(N, N\)-Dimethylaniline-\(N\)-oxides into \(N\)-methyldiindolines by a Tandem Polonovski-Mannich Reaction”

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**General Information:** All reactions were performed in single-neck oven- or flame-dried round-bottom flasks fitted with rubber septa under a positive pressure of argon, unless otherwise noted. Air- and moisture-sensitive liquids were transferred via syringe or stainless steel cannula. Organic solutions were concentrated by rotary evaporation below 35 °C at 10 Torr (diaphragm vacuum pump) unless otherwise noted. Analytical thin-layer chromatography (TLC) was performed using glass plates pre-coated with silica gel (0.25 mm, 60-Å pore size, 5–20 µm, Silicycle) impregnated with a fluorescent indicator (254 nm). TLC plates were visualized by exposure to ultraviolet light (UV), then were stained by submersion in aqueous ceric ammonium molybdate solution (CAM), ethanolic phosphomolybdic acid solution (PMA), acidic ethanolic p-anisaldehyde solution (anisaldehyde), or aqueous potassium permanganate solution (KMnO₄), followed by brief heating on a hot plate (215 °C, 10–15 s). Flash chromatography was performed as described by Still et al.¹, employing silica gel (60-Å pore size, 40–63 µm, standard grade, Silicycle) or basic alumina (60-Å pore size, 50–200 µm, Brockmann I, Sorbent Technologies or Acros Organics).

**Materials:** Commercial reagents and solvents were used as received with the following exceptions. Triethylamine, dichloromethane, ethyl ether, dimethylsulfoxide, tetrahydrofuran, hexane, toluene, and benzene were purified by the method of Pangborn, et. al.² Where noted, solvents were deoxygenated before use by bubbling with argon for 20 minutes.

**Instrumentation:** Proton nuclear magnetic resonance (¹H NMR) spectra and carbon nuclear magnetic resonance (¹³C NMR) spectra were recorded on Varian Mercury Plus 300 MHz/75 MHz or Varian Unity INOVA 500 MHz/125 MHz NMR spectrometers at 23 °C. Fluorine nuclear magnetic resonance (¹⁹F NMR) spectra were recorded on a Varian Mercury Plus 282 MHz spectrometer at 23 °C. Proton chemical shifts are expressed in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent (CHCl₃: δ 7.26, CD₂HOD: δ 3.31, CD₃SOCD₂H: δ 2.50, C₆D₆H: δ 7.16). Carbon chemical shifts are expressed in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the NMR solvent (CDCl₃: δ 77.16, CD₃OD: δ 49.00, CD₃SOCD₃: δ 39.52, C₆D₆: δ 128.00). Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet, br =


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broad, app = apparent), integration, and coupling constant ($J$) in Hertz (Hz). Infrared (IR) spectra were obtained using a Shimadzu IRAffinity-1 FT-IR spectrophotometer referenced to a polystyrene standard and data are represented as frequency of absorption (cm$^{-1}$). Accurate mass measurements were obtained on a Waters LCT premier (ESI source, flow injection analysis) or a Waters GCT premier (GC-MS fitted with an EI or CI source) at the Mass Spectrometry Facility at the University of California at Irvine.

(For clarity, intermediates that have not been assigned numbers in the text are numbered sequentially in the supplemental information beginning with 11.)

**Experimental Procedures:**

**Preparation of cyanoacetyl chloride:**

Oxalyl chloride (4.00 mL, 4.70 mmol, 1.10 equiv) was added dropwise to a solution of cyanoacetic acid (3.50 g, 4.20 mmol, 1 equiv) in dichloromethane (25 mL). The resultant solution was stirred at 23 °C whereupon $N,N$-dimethylformamide (10 $\mu$L) was added via syringe. The reaction flask was fitted with a bubbler and the reaction mixture was stirred at 23 °C for 2 h at which point gas evolution ceased. The bubbler was removed and the reaction flask was fitted with a distillation head and collection flask. The reaction flask was heated at 50 °C under reduced pressure (600 torr) and dichloromethane was removed by distillation. The reaction vessel was then cooled, filled with argon, and the collection flask was exchanged. The distillation apparatus pressure was reduced to 5 torr and the distillation assembly was then lowered into a preheated oil bath (100 °C). A distillation fraction was collected at 67 °C to afford cyanoacetyl chloride (1.39 g, 32%) as a light red oil.

Cyanoacetyl chloride: $^1$H NMR (300 MHz, CDCl$_3$), $\delta$: 3.99 (s, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$), $\delta$: 163.9, 111.0, 35.6.
Preparation of diketene:\(^3\)

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\text{N} \\
\text{El}_2\text{O} \\
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Acetyl chloride (5.00 mL, 70.3 mmol, 1 equiv) was added dropwise to a vigorously stirred solution of triethylamine (11 mL, 78.9 mmol, 1.12 equiv) in diethyl ether (100 mL). The resultant slurry was stirred at 23 °C for 16 h whereupon the reaction flask was fitted with a distillation head and collection flask. The reaction flask was heated at 60 °C and diethyl ether was removed by distillation. The reaction vessel was then cooled, filled with argon, and the collection flask exchanged. The distillation apparatus pressure was reduced to 60 torr and the distillation assembly was then lowered into a preheated oil bath (120 °C). A distillation fraction was collected at 58 °C to afford diketene (1.60 g, 59%) as a clear, colorless oil.

Diketene: \(^1\)H NMR (300 MHz, CDCl\(_3\)), \(\delta\): 5.00–4.71 (m, 1H), 4.49 (m, 1H), 3.90 (t, \(J = 1.7\) Hz, 2H). \(^13\)C NMR (75 MHz, CDCl\(_3\)), \(\delta\): 165.2, 147.6, 87.3, 42.5.

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A stirred solution of N,N-dimethylaniline-N-oxide 11a (1.00 g, 7.30 mmol, 1 equiv, dried by azeotropic distillation with benzene) in dichloromethane (100 mL) was cooled to –78 °C whereupon diketene (0.68 mL, 8.03 mmol, 1.1 equiv) was added dropwise via syringe. The resultant solution was stirred for 1 h whereupon triethylamine (2.00 mL, 14.6 mmol, 2.1 equiv) was added. The reaction mixture was stirred for 30 min, then was warmed to 23 °C, stirred at that temperature for 8 h, then was concentrated. Purification of the residue by flash column chromatography (gradient elution 2 → 10% ethyl acetate–hexanes) afforded 1-(2-(dimethylamino)phenyl)propan-2-one 9 (543 mg, 42%) as a clear oil.

1-(2-(dimethylamino)phenyl)propan-2-one 9: TLC: 20% ethyl acetate–hexanes, Rf = 0.50 (UV, anisaldehyde). 1H NMR (300 MHz, CDCl3), δ: 7.35–7.23 (m, 1H), 7.17 (d, 2H), 7.07 (td, J1 = 7.3 Hz, J2 = 1.3 Hz, 1H), 3.74 (s, 2H), 2.62 (s, 6H), 2.09 (s, 3H). 13C NMR (75 MHz, CDCl3), δ: 207.0, 152.7, 131.1, 128.2, 123.9, 120.3, 112.9, 47.3, 44.4, 28.6. FTIR (NaCl, thin film), cm⁻¹: 3061, 2979, 2940, 2828, 2784, 1700, 1599. HRMS: ESI+ [M+Na⁺] Calcd. for C11H15NONa: 200.1043. Found: 200.1051.
A stirred solution of \(N,N\text{-dimethylaniline-N-oxide\,}11a\) (810 mg, 5.90 mmol, 1 equiv, dried by azeotropic distillation with benzene) in dichloromethane (100 mL) was cooled to \(-78\,^\circ\text{C}\) whereupon cyanoacetyl chloride (674 mg, 6.50 mmol, 1.1 equiv) was added dropwise via syringe. The resultant solution was stirred for 1 h whereupon triethylamine (1.60 mL, 11.8 mmol, 2.00 equiv) was added. The reaction mixture was stirred for 30 min, then was warmed to 23 \(^\circ\text{C}\), stirred at that temperature for 8 h, then was concentrated. Purification of the residue by flash column chromatography (gradient elution 2 \(\rightarrow\) 10% ethyl acetate–hexanes) afforded 2-(2-(dimethylamino)phenyl)acetonitrile (8) (350 mg, 37%) as a clear oil.

2-(2-(dimethylamino)phenyl)acetonitrile 8: TLC: 20% ethyl acetate–hexanes, \(R_f = 0.53\) (UV, anisaldehyde). \(^1\text{H NMR (300 MHz, CDCl}_3\text{), }\delta:\ 7.44 (\text{dd, } J_1 = 7.6 \text{ Hz, } J_2 = 0.9 \text{ Hz, } 1\text{H}), 7.35–7.28 (m, 1\text{H}), 7.18 (\text{dd, } J_1 = 8.0 \text{ Hz, } J_2 = 1.1 \text{ Hz, } 1\text{H}), 7.12 (\text{td, } J_1 = 7.5 \text{ Hz, } J_2 = 1.2 \text{ Hz, } 1\text{H}), 3.85 (s, 2\text{H}), 2.67 (s, 6\text{H}). \(^{13}\text{C NMR (75 MHz, CDCl}_3\text{), }\delta:\ 152.6, 129.6, 129.2, 125.9, 124.3, 120.5, 119.0, 45.0, 19.6. FTIR (NaCl, thin film), cm\(^{-1}\): 2982, 2943, 2864, 2830, 2789, 2248, 1733, 1637. HRMS: ES\(^+\) [M+H\(^+\)] Calcd. for C\(_{10}\)H\(_{13}\)O\(_2\)N\(_2\): 161.1078. Found: 161.1074.
General procedure for the Synthesis of N,N-Dimethylaniline-N-Oxides

A solution of 3-chloroperbenzoic acid (70% w/w, 860 mg, 3.48 mmol, 1.10 equiv) in dichloromethane (10 mL) was transferred via cannula to a stirred solution of ethyl 2-(2-(dimethylamino)phenyl)acetate 1a (720 mg, 3.23 mmol, 1 equiv) in dichloromethane (10 mL). The resultant mixture was stirred for 30 minutes at 23 °C, then was loaded directly onto a basic alumina column and eluted with 25% methanol–dichloromethane. The combined filtrates were concentrated to give 2-(2-ethoxy-2-oxoethyl)-N,N-dimethylaniline oxide 2a (710 mg, 98% yield) as a yellow oil.

2-(2-ethoxy-2-oxoethyl)-N,N-dimethylaniline oxide 2a: TLC 10% ethyl acetate-hexanes, \( R_f = 0.00 \) (UV, KMnO₄). \( ^1H \) NMR (300 MHz, CDCl₃), \( \delta: 7.48–7.37 \) (m, 1H), 7.35–7.18 (m, 3H), 4.59 (s, 2H), 4.16 (q, \( J = 7.1 \) Hz, 2H), 3.63 (s, 6H), 1.25 (t, \( J = 7.1 \) Hz, 3H). \( ^{13}C \) NMR (75 MHz, CDCl₃), \( \delta: 172.3, 151.3, 135.7, 129.8, 129.2, 127.9, 119.8, 63.6, 60.9, 40.9, 14.4 \). FTIR (NaCl, thin film), cm\(^{-1}\): 3441, 1728, 1489. HRMS: EI+ [M + Na⁺] Calcd. for C₁₂H₁₇NO₃Na: 246.1106. Found: 246.1106.

4-chloro-2-(2-ethoxy-2-oxoethyl)-N,N-dimethylaniline oxide 2b: 92% yield. TLC 10% ethyl acetate-hexanes, \( R_f = 0.00 \) (UV, KMnO₄). \( ^1H \) NMR (300 MHz, CDCl₃), \( \delta: 7.37 \) (d, \( J = 8.2 \) Hz, 1H), 7.30–7.17 (m, 2H), 4.50 (s, 2H), 4.14 (q, \( J = 7.1 \) Hz, 2H), 3.58 (s, 6H), 1.23 (t, \( J = 7.1 \) Hz,
$^1$H NMR (125 MHz, CDCl$_3$), δ: 171.7, 149.9, 135.3, 134.6, 131.9, 127.8, 121.3, 63.6, 61.0, 40.7, 14.3. FTIR (NaCl, thin film), cm$^{-1}$: 3390, 2986, 1728, 1643. HRMS: El$^+$ [M+Na]$^+$ Calcd. for C$_{12}$H$_{16}$ClNO$_3$Na: 280.0716. Found: 280.0712.

2-(2-ethoxy-2-oxoethyl)-4-fluoro-$N,N$-dimethylaniline oxide 2c: 83% yield. TLC 10% ethyl acetate-hexanes, $R_f$ = 0.00 (UV, KMnO$_4$). $^1$H NMR (300 MHz, CDCl$_3$), δ: 7.44 (dd, $J_1$ = 8.9, $J_2$ = 4.7 Hz, 1H), 7.06–6.88 (m, 2H), 4.56 (s, 2H), 4.17 (dd, $J_1$ = 14.2, $J_2$ = 7.1 Hz, 2H), 3.63 (s, 6H), 1.26 (t, $J$ = 7.1 Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$), δ: 171.7, 161.6 (d, $J = 248.0$ Hz), 147.5, 132.8, 122.0 (d, $J = 22.5$ Hz), 121.6 (d, $J = 8.9$ Hz), 114.4 (d, $J = 22.5$ Hz), 63.8, 61.0, 40.7, 14.3. $^{19}$F NMR (282 MHz, CDCl$_3$), δ: -113.1. FTIR (NaCl, thin film), cm$^{-1}$: 3055, 2985, 1728. HRMS: El$^+$ [M + Na]$^+$ Calcd. for C$_{12}$H$_{16}$FNO$_3$Na: 264.1012. Found: 264.1012.

2-(2-ethoxy-2-oxoethyl)-$N,N,4$-trimethylaniline oxide 2d: 72% yield. TLC 10% ethyl acetate-hexanes, $R_f$ = 0.00 (UV, KMnO$_4$). $^1$H NMR (300 MHz, CDCl$_3$), δ: 7.30 (d, $J = 9.1$ 1H), 7.17–6.96 (m, 2H), 4.59 (s, 2H), 4.17 (q, $J = 7.2$ Hz, 2H), 3.62 (s, 6H), 2.33 (s, 3H), 1.31 (t, $J = 7.1$ Hz, 3H). $^{13}$C (75 MHz, CDCl$_3$), δ: 172.3, 148.8, 138.8, 135.9, 129.1, 128.2, 119.6, 63.3, 60.5, 40.5, 20.5, 14.1. FTIR (NaCl, thin film), cm$^{-1}$: 3417, 1659, 1504. HRMS: El$^+$ [M + Na]$^+$ Calcd. for C$_{13}$H$_{18}$NO$_3$Na: 260.1263. Found: 260.1262.
2-(2-ethoxy-2-oxoethyl)-4-methoxy-N,N-dimethylaniline oxide 2e: 85% yield. TLC 10% ethyl acetate-hexanes, R_f = 0.00 (UV, KMnO_4). ^1^H NMR (300 MHz, CDCl_3), δ: 7.33 (d, J = 9.8 Hz, 1H), 6.81–6.68 (m, 2H), 4.49 (s, 2H), 4.13 (q, J = 7.1 Hz, 2H), 3.76 (s, 3H), 3.58 (s, 6H), 1.22 (t, J = 7.1 Hz, 4H). ^13^C NMR (75 MHz, CDCl_3), δ: 172.1, 159.1, 144.2, 131.1, 121.0, 120.3, 112.5, 63.6, 60.8, 55.6, 40.8, 14.3. FTIR (NaCl, thin film), cm⁻¹: 3414, 1731, 1498. HRMS: El+ [M+Na]^+ Calcd. for C_{13}H_{19}NO_4: 276.1212. Found: 276.1201.

**General procedure for the Synthesis of Indolines:**

A stirred solution of 2-(2-ethoxy-2-oxoethyl)-N,N-dimethylaniline oxide (223 mg, 1.00 mmol, 1 equiv, dried by azeotropic distillation with benzene) in dichloromethane (10 mL) was cooled to –78 °C whereupon acetic anhydride (0.112 mL, 1.10 mmol, 1.10 equiv) was added dropwise via syringe. The resultant solution was stirred for 45 minutes whereupon triethylamine (276 µL, 2.00 mmol, 2.00 equiv) was added. The solution was slowly warmed to 23 °C over the course of 1 h, then was concentrated. Purification of the residue by flash column chromatography (gradient elution 2 → 5% ethyl acetate–hexanes) afforded ethyl 1-methylindoline-3-carboxylate 4a (107 mg, 52%) as a pale yellow oil.

ethyl 1-methylindoline-3-carboxylate 4a: TLC 10% ethyl acetate-hexanes, R_f = 0.36 (UV, KMnO_4). ^1^H NMR (300 MHz, CDCl_3), δ: 7.33–7.23 (m, 1H), 7.15 (tdd, J_1 = 7.6 Hz, J_2 = 1.3 Hz, J_3 = 0.8 Hz, 1H), 6.70 (td, J = 13.5 Hz, 1.0 Hz, 1H), 6.51 (d, J = 7.9 Hz, 1H), 4.28–4.18 (qd, J = 6.3, 2.6 Hz, 2H), 4.10 (t, J = 8.7 Hz, 1H), 3.66 (t, J = 8.6 Hz, 1H), 3.53 (t, J = 9.3 Hz, 1H), 2.79 (s, 3H), 1.31 (t, J = 7.1 Hz, 3H). ^13^C NMR (75 MHz, CDCl_3), δ: 172.1, 152.8, 128.9, 126.9, 124.9, 118.0, 107.8, 61.3, 57.6, 46.1, 36.0, 14.4. FTIR (NaCl, thin film), cm⁻¹: 3056, 2987, 1640, 1493. HRMS: El+ [M + Na]^+ Calcd. for C_{12}H_{15}NO_2Na: 228.1001. Found: 228.1005.
ethyl 5-chloro-1-methylindoline-3-carboxylate 4b: 52% yield. TLC 10% ethyl acetate-hexanes, 
Rf = 0.30 (UV, KMnO4). 1H NMR (300 MHz, CDCl3), δ: 7.34–7.16 (m, 1H), 7.11 (ddd, J1 = 8.4, J2 = 2.2, J3 = 0.7 Hz, 1H), 6.41 (d, J = 8.4 Hz, 1H), 4.27 (q, J = 7.1 Hz, 2H), 4.10 (t, J = 8.7 Hz, 1H), 3.71 (dd, J1 = 9.3 Hz, J2 = 8.0 Hz, 1H), 3.58 (t, J = 9.4 Hz, 1H), 1.32 (t, J = 7.1 Hz, 3H). 13C NMR (125 MHz, CDCl3), δ: 171.4, 151.5, 128.6, 128.5, 125.1, 122.5, 108.3, 61.5, 57.7, 45.7, 35.9, 14.4. FTIR (NaCl, thin film), cm⁻¹: 2955, 2862, 1738, 1697, 1605. HRMS: EI+ [M+Na]+ Calcd. for C12H14ClNO2Na: 262.0611. Found: 262.0611.

ethyl 5-fluoro-1-methylindoline-3-carboxylate 4c: 53% yield. TLC 10% ethyl acetate-hexanes, 
Rf = 0.36 (UV, KMnO4). 1H NMR (300 MHz, CDCl3), δ: 7.02 (ddd, J1 = 8.4, J2 = 2.7, J3 = 1.2 Hz, 1H), 6.94–6.73 (m, 1H), 6.39 (dd, J1 = 8.6, J2 = 4.2 Hz, 1H), 4.24 (qd, J1 = 7.1, J2 = 1.4 Hz, 2H), 4.06 (t, J = 8.8 Hz, 1H), 3.62 (dd, J1 = 9.1, J2 = 8.2 Hz, 1H), 3.53 (t, J = 9.2 Hz, 1H), 2.75 (s, 3H), 1.32 (t, J = 7.1 Hz, 3H). 13C NMR (75 MHz, CDCl3), δ: 171.5, 156.6 (d, J = 235.0 Hz), 149.3, 128.2 (d, J = 8.3 Hz), 114.8 (d, J = 23.2 Hz), 112.6 (d, J = 24.9 Hz), 107.9 (d, J = 8.3 Hz), 61.5, 58.2, 45.9, 36.7, 14.4. 19F NMR (282 MHz, CDCl3), δ: -127.2. FTIR (NaCl, thin film), cm⁻¹: 2922, 2854, 1743. HRMS: EI+ [M+Na]+ Calcd. for C12H15NO2Na: 224.1087. Found: 224.1079.

ethyl 1,5-dimethylindoline-3-carboxylate 4d: 57% yield. TLC 10% ethyl acetate-hexanes, 
Rf = 0.54 (UV, KMnO4). 1H NMR (300 MHz, CDCl3), δ: 7.15–7.07 (m, 1H), 7.01–6.90 (m, 1H), 6.43
(d, J = 7.9 Hz, 1H), 4.23 (qd, J1 = 7.1, J2 = 1.7 Hz, 2H), 4.06 (t, J = 8.6 Hz, 1H), 3.59 (dd, J1 = 9.1, J2 = 8.1 Hz, 1H), 3.49 (t, J = 9.2 Hz, 1H), 2.75 (s, 3H), 2.26 (s, 3H), 1.31 (t, J = 7.1 Hz, 3H).

13C NMR (125 MHz, CDCl3), δ: 172.1, 150.9, 129.2, 127.6, 127.2, 125.6, 107.9, 61.3, 58.2, 46.2, 36.7, 20.9, 14.4. FTIR (NaCl, thin film), cm⁻¹: 2978, 2862, 1736, 1501.


ethyl 5-methoxy-1-methylindoline-3-carboxylate 4e: 47% yield. TLC 10% ethyl acetate-hexanes, Rf = 0.23 (UV, KMnO4). 1H NMR (300 MHz, CDCl3), δ: 7.15 (d, J = 8.1 Hz, 1H), 6.23 (dd, J1 = 8.1, J2 = 2.3 Hz, 1H), 6.07 (d, J = 2.2 Hz, 1H), 4.22 (qd, J1 = 7.1, J2 = 3.2 Hz, 2H), 4.03 (br t, J = 8.5 Hz, 1H), 3.78 (s, 3H), 3.73 – 3.64 (m, 1H), 3.53 (t, J = 9.2 Hz, 1H), 2.77 (s, 3H), 1.30 (t, J = 7.1 Hz, 3H). 13C NMR (75 MHz, CDCl3), δ: 172.2, 161.3, 125.1, 119.3, 102.3, 94.8, 61.2, 58.0, 55.5, 45.3, 35.7, 14.4. FTIR (NaCl, thin film), cm⁻¹: 2978, 2862, 1736, 1501. HRMS: EI+ [M+Na]+ Calculated for C13H19NO3: 258.1106. Found: 258.1111.

1-methylindoline-3-carbonitrile 10: 61% yield (synthesized from unpurified N-oxide). TLC 10% ethyl acetate-hexanes, Rf = 0.36 (UV, KMnO4). 1H NMR (300 MHz, CDCl3), δ: 7.44 (dd, J1 = 7.6, J2 = 0.9 Hz, 1H), 7.35 – 7.28 (m, 1H), 7.18 (dd, J1 = 8.0, J2 = 1.1 Hz, 1H), 7.12 (td, J1 = 7.5, J2 = 1.2 Hz, 1H), 3.85 (s, 2H), 2.67 (s, 6H). 13C NMR (75 MHz, CDCl3), δ: 152.3, 129.9, 124.5, 123.9, 119.5, 119.0, 108.4, 59.3, 35.8, 31.2. FTIR (NaCl, thin film), cm⁻¹: 2922, 2854, 1743. HRMS: EI+ [M + H]+ Calculated for C10H11N2: 159.0922. Found: 159.0917.