Title: A New Synthetic Route for the Preparation of 1,10-Phenantridine Derivatives
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Figure S1. Pairs of enantiomeric conformers in the crystal structure of 5b (A) and 5d (B).
Experimental section

**General.** All reagents were used as purchased from commercial sources without further purification. Solvents were dried using standard techniques prior to use. All reactions were performed in standard glassware under an inert argon atmosphere. Evaporation was done using water aspirator and drying in vacuo at $10^{-2}$ Torr. Column chromatography: Merck silica gel 60, 40-63 µm (230-400 mesh). TLC: Precoated glass sheets with silica gel 60 F$_{254}$ (Merck), visualization by UV light. Melting points were determined on a Electrothermal Digital Melting Point apparatus and are uncorrected. NMR spectra were recorded on a Bruker AM 300 (300 MHz) with solvent signal as reference. FAB-mass spectra were taken on a ZA HF instrument with 4-nitrobenzyl alcohol as matrix.

**Preparation of 1a-1c.** A solution of the appropriate organolithium derivative (6.1 mmol) was added slowly to a suspension of 1,10-phenanthroline (1 g, 5.55 mmol) in dry ether (40 mL) at 0°C. After 2.5 hrs, benzylbromide (0.72 mL, 6.1 mmol) was added and the mixture was allowed to heat to room temperature. After 24 hrs the mixture was filtered and the solvents were removed under reduced pressure.

**1a.** Purification by column chromatography (SiO$_2$, cyclohexane/CH$_2$Cl$_2$ 2/1 to 1/1 and then cyclohexane/CH$_2$Cl$_2$ 1/1 with 1% CH$_3$OH), afforded compound 1a as a yellow oil in a 74% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 0.78 (t, $J$ = 7 Hz, 3 H), 1.08-1.19 (m, 2 H), 1.23-1.50 (m, 4 H), 3.79-3.86 (m, 1 H), 4.66 (d, $J$ = 15 Hz, 1 H), 5.49 (d, $J$ = 15 Hz, 1 H), 5.74 (dd, $J$ = 6 Hz, $J$ = 9 Hz, 1 H), 6.45 (d, $J$ = 9 Hz, 1 H), 7.17-7.29 (m, 6 H), 7.37-7.42 (m, 2 H), 8.00 (dd, $J$ = 8 Hz, $J$ = 2 Hz, 1 H), 8.83 (dd, $J$ = 6 Hz, $J$ = 2 Hz, 1 H). $^{13}$C-NMR (75 MHz, CDCl$_3$): $\delta$ 14.0, 22.6, 26.8, 35.3, 57.5, 59.4, 119.0, 120.3, 124.9, 125.5, 126.3, 126.7, 127.2, 127.9, 128.1, 129.4, 136.2,
Purification by column chromatography (SiO₂, hexane/CH₂Cl₂ 8/2), afforded compound 1b as a yellow oil in a 79% yield. ¹H-NMR (300 MHz, CDCl₃): δ 0.80 (t, J = 7.5 Hz, 3 H), 1.04-1.23 (m, 6 H), 1.25-1.38 (m, 2 H), 1.40-1.51 (m, 2 H), 3.79-3.85 (m, 1 H), 4.67 (d, J = 15 Hz, 1 H), 5.47 (d, J = 15 Hz, 1 H), 5.74 (dd, J = 6 Hz, J = 8 Hz, 1 H), 6.45 (d, J = 9 Hz, 1 H), 7.17-7.29 (m, 6 H), 7.40-7.42 (m, 2 H), 8.00 (dd, J = 8 Hz, J = 2 Hz, 1 H), 8.83 (dd, J = 6 Hz, J = 2 Hz, 1 H). ¹³C-NMR (75 MHz, CDCl₃): δ 14.0, 22.6, 24.5, 29.2, 31.7, 35.5, 57.4, 59.3, 119.0, 120.2, 124.8, 125.5, 126.2, 126.7, 127.2, 127.9, 128.4, 129.3, 136.1, 140.6, 141.1, 142.7, 147.5. Anal. calc. for C₂₅H₂₈N₂: C 84.23, H 7.92, N 7.86; found: C 84.01, H 8.11, N 7.88.

Purification by column chromatography (SiO₂, CH₂Cl₂/hexane: 1/1 to CH₂Cl₂), afforded compound 1c as a yellow oil in a 72% yield. ¹H-NMR (300 MHz, CDCl₃): δ 4.75 (d, J = 15.5 Hz, 1 H), 5.08 (d, J = 6 Hz, 1 H), 5.96 (d, J = 15.5 Hz, 1 H), 5.97 (dd, J = 9 Hz, J = 6 Hz, 1 H), 6.59 (d, J = 9 Hz, 1 H), 7.12-7.30 (m, 9 H), 7.32-7.35 (m, 2 H), 7.45-7.47 (m, 2 H), 7.99 (dd, J = 8 Hz, J = 2 Hz, 1 H), 8.80 (dd, J = 6 Hz, J = 2 Hz, 1 H). ¹³C-NMR (75 MHz, CDCl₃): δ 58.9, 60.5, 118.7, 120.4, 124.2, 125.3, 125.7, 126.1, 126.4, 126.8, 128.15, 128.2, 128.3, 129.7, 136.2, 140.2, 140.9, 143.8, 147.3. Anal. calc. for C₂₅H₂₀N₂: C 86.17, H 5.79, N 8.04; found: C 86.40, H 5.43, N 8.17.

Preparation of 3a-3e. A solution of the appropriate organolithium derivative (2.14 mmol) was added slowly to a suspension of 2 (0.5 g, 1.95 mmol) in dry ether (20 mL) at 0°C. After 3 hrs, benzylbromide (0.25 mL, 2.14 mmol) was added. After 24 hrs at room temperature the mixture was filtered and the solvents were removed under reduced pressure.
**3a.** Purification by column chromatography (SiO₂, CH₂Cl₂/cyclohexane: 1/4), afforded 3a as a yellow solid in a 55% yield. Mp: 144-146°C. ¹H-NMR (300 MHz, CDCl₃): δ 1.12 (d, J = 6.5 Hz, 3 H), 3.91-3.95 (m, 1 H), 5.20 (AB, J = 15.5 Hz, 2 H), 5.79 (dd, J = 6 Hz, J = 9 Hz, 1 H), 6.54 (d, J = 9 Hz, 1 H), 7.21 (d, J = 8.5 Hz, 1 H), 7.25-7.36 (m, 7 H), 7.64-7.66 (m, 2 H), 7.79 (d, J = 8.5 Hz, 1 H). ¹³C-NMR (75 MHz, CDCl₃): δ 21.1, 53.4, 58.8, 118.1, 119.0, 125.1, 125.4, 126.5, 127.1, 127.6, 127.8, 128.4, 128.7, 128.8, 129.1, 137.4, 140.1, 141.0, 141.5, 142.8, 154.6. FAB-MS: 363.2 [M + H].

Anal. calc. for C₂₆H₂₂N₂: C 86.15, H 6.12, N 7.73; found: C 86.54, H 6.13, N 7.33.

**3b.** Purification by column chromatography (SiO₂, CH₂Cl₂/cyclohexane 1/1 to 3/1), afforded 3b as a yellow oil in a 64% yield. ¹H-NMR (300 MHz, CDCl₃): δ 0.79 (t, J = 7.5 Hz, 3 H), 1.08-1.23 (m, 2 H), 1.27-1.52 (m, 4 H), 3.78-3.85 (m, 1 H), 4.94 (d, J = 15 Hz, 1 H), 5.52 (d, J = 15 Hz, 1 H), 5.80 (dd, J = 9.5 Hz, J = 6 Hz, 1 H), 6.55 (d, J = 6.5 Hz, 1 H), 7.18-7.33 (m, 5 H), 7.40-7.45 (m, 3 H), 7.55-7.57 (m, 2 H), 7.79 (d, J = 8.5 Hz, 1 H), 8.07 (d, J = 8.5 Hz, 1 H), 8.10-8.13 (m, 2 H). ¹³C-NMR (75 MHz, CDCl₃): δ 14.0, 22.6, 26.9, 35.1, 57.2, 59.1, 117.7, 118.5, 125.0, 125.5, 126.1, 126.7, 126.8, 127.4, 127.9, 128.2, 128.4, 128.8, 137.0, 139.9, 141.1, 142.4, 154.1. Anal. calc. for C₂₉H₂₈N₂: C 86.10, H 6.98, N 6.92; found: C 86.05, H 7.21, N 6.74.

**3c.** Column chromatography (SiO₂, CH₂Cl₂/cyclohexane 1/10 to 1/5), afforded 3c, as a mixture of diastereoisomereres that was used for the next step without further purification. The mixture was obtained as a yellow oil in a 72% yield. ¹H-NMR (300 MHz, CDCl₃): δ 0.75 and 0.80 (2 t, J = 7.5 Hz, 3 H), 0.84 and 0.95 (2 d, J = 7 Hz, 3 H), 1.08-1.25 (m, 2 H), 1.47-1.84 (m, 1 H), 3.62 and 3.70 (2 dd, J = 6 Hz, J = 8 Hz, 1 H), 4.67 and 4.72 (2 d, J = 15.5 Hz, 1 H), 5.75 and 5.81 (2 dd, J = 6 Hz, J = 9.5 Hz, 1 H), 5.97 and 5.99 (2 d, J = 15.5 Hz, 1 H), 6.56 and 6.57 (2 d, J = 9.5 Hz, 1 H), 7.14-7.23 (m, 5 H), 7.35-7.53 (m, 5 H), 7.76 and 7.77 (2 d, J =
8.5 Hz, 1 H), 8.04 and 8.05 (2 d, J = 8.5 Hz, 1 H), 8.16-8.22 (m, 2 H).

3d. Purification by column chromatography (SiO₂, CH₂Cl₂/cyclohexane 1/1), afforded 3d as a yellow glassy product in a 57% yield. ¹H-NMR (300MHz, CDCl₃): δ 0.93 (s, 9H), 3.70 (d, J = 6 Hz, 1H), 4.53 (d, J = 15.5 Hz, 1H), 5.74 (dd, J = 6 Hz, J = 9.5 Hz, 1H), 6.37 (d, J = 15.5 Hz, 1H), 6.63 (d, J = 9.5 Hz, 1H), 7.11-7.25 (m, 7H), 7.46-7.77 (m, 3H), 7.78 (d, J = 8.5 Hz, 1H), 8.02 (d, J = 8.5 Hz, 1H), 8.28-8.31 (m, 2H). ¹³C-NMR (75 MHz, CDCl₃): δ 25.5, 40.7, 62.7, 67.8, 117.6, 117.9, 124.7, 125.8, 126.0, 126.7, 126.8, 127.3, 128.1, 128.2, 128.5, 128.9, 129.1, 137.2, 140.3, 141.6, 141.8, 143.3, 153.6. Anal. calc. for C₂₉H₂₈N₂: C 86.10, H 6.98, N 6.92; found: C 86.10, H 6.82, N 7.08.

3e. Purification by column chromatography (SiO₂, CH₂Cl₂/hexane 9/1 to 1/1), afforded 3e as a yellow glassy product in a 71% yield. ¹H-NMR (300 MHz, CDCl₃): δ 4.98 (d, J = 15.5 Hz, 1 H), 5.09 (d, J = 6 Hz, 1 H), 5.99 (d, J = 15.5 Hz, 1 H), 6.02 (dd, J = 9 Hz, J = 6 Hz, 1 H), 6.63 (d, J = 9 Hz, 1 H), 7.12-7.31 (m, 8 H), 7.35-7.45 (m, 5 H), 7.49-7.56 (m, 2 H), 7.80 (d, J = 8.5 Hz, 1 H), 8.05-8.13 (m, 3 H). Anal. calc. for C₃₁H₂₄N₂: C 87.70, H 5.70, N 6.60; found: C 87.45, H 5.99, N 6.56.

Preparation of 4a-4b and 3e. A solution of the appropriate organolithium derivative (12.2 mmol) was added slowly to a suspension of 1,10-phenanthroline (1 g, 5.55 mmol) in dry ether (40 mL). After 24 hrs benzylbromide (0.8 mL, 6.66 mmol) was added. After 24 hrs the solvents were removed under reduced pressure.

4a. Purification by column chromatography (SiO₂, cyclohexane/CH₂Cl₂ 7/3), afforded compound 4a as a yellow oil in a 79% yield. ¹H-NMR (300 MHz, CDCl₃): δ 0.76 (t, J = 7.5, 3 H), 0.91 (t, J = 7.5, 3 H), 1.01-1.42 (m, 8 H), 1.73-1.83 (m, 2 H), 2.93 (t, J = 7.5, 2 H), 3.72-3.79 (m, 1 H), 4.85 (d, J = 15 Hz , 1 H), 5.27 (d, J = 15 Hz , 1 H), 5.77 (dd, J = 5.5 Hz, J = 9 Hz, 1 H), 6.51 (d, J = 9 Hz, 1 H), 7.14 (d, J = 8 Hz, 1 H, 7.16 (AB, J = 8 Hz, 2 H), 7.22-7.31
(m, 3 H), 7.62-7.59 (m, 2 H), 7.89 (d, J = 9 Hz, 1 H). $^{13}$C-NMR (75 MHz, CD$_2$Cl$_2$): δ 14.4, 14.5, 23.1, 23.2, 27.5, 32.3, 39.3, 58.2, 59.6, 119.3, 121.2, 125.6, 125.8, 127.2, 127.3, 128.2, 128.4, 129.2, 136.8, 140.7, 142.1, 142.7, 160.6. Anal. calc. for C$_{27}$H$_{32}$N$_2$: C 84.33, H 8.39, N 7.28; found: C 84.55, H 8.41, N 7.04.

**4b.** Purification by column chromatography (SiO$_2$, hexane/CH$_2$Cl$_2$ 8/2), afforded 4b as a yellow oil in a 74% yield. $^1$H-NMR (300 MHz, CDCl$_3$): δ 0.75-0.92 (m, 6 H), 1.00-1.43 (m, 16 H), 1.74-1.84 (m, 2 H), 2.91 (t, J = 7.5, 3 H), 3.72-3.79 (m, 1 H), 4.86 (d, J = 15 Hz, 2 H), 5.25 (d, J = 15 Hz, 2 H), 5.77 (dd, J = 6 Hz, J = 9 Hz, 1 H), 6.51 (d, J = 9 Hz, 1 H), 7.01-7.20 (m, 6 H), 7.62-7.60 (m, 2 H), 7.89 (d, J = 8 Hz, 1 H). Anal. calc. for C$_{31}$H$_{40}$N$_2$: C 84.49, H 9.15, N 6.36; found: C 84.10, H 9.49, N 6.41.

**3e.** Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$/hexane: 9/1 to CH$_2$Cl$_2$/hexane: 1/1), afforded compound 3e (0.70 g, 2 mmol) as a yellow glassy product in an 34% yield.

**Preparation of 5a-5d.** MnO$_2$ (6 g) was added to a solution of the appropriate phenanthroline derivative (1 mmol) in CH$_2$Cl$_2$ (30 mL). When the characteristic yellow colour had disappeared, MgSO$_4$ was added; the mixture was filtered on celite and evaporated.

**5a.** Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH:98/2), afforded 5a as a brownish glassy product in a 81% yield from 1a and 82% from 1b. $^1$H-NMR (300 MHz, CDCl$_3$): δ 6.81 (broad s, 2 H), 6.98 (d, J = 9 Hz, 1 H), 7.08-7.25 (m, 5 H), 7.42 (dd, J = 4 Hz, J = 8 Hz, 1 H), 7.58 (AB, J = 8 Hz, 2 H), 7.85 (d, J = 9 Hz, 1 H), 8.12 (dd, J = 8 Hz, J = 2 Hz, 1 H), 8.79 (dd, J = 4 Hz, J = 2 Hz, 1 H). $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 50.2, 120.7, 121.8, 122.5, 122.6, 125.9, 126.0, 126.7, 127.0, 127.9, 130.0, 136.1, 137.1, 139.4, 139.7, 147.0, 164.0. Anal. calc. for C$_{19}$H$_{14}$N$_2$O.H$_2$O: C 74.98, H 5.30, N 9.20; found: C 74.91, H 5.32, N 9.01.

**5b.** Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH:999/1), afforded 5b as a colourless solid in a 73% yield. $^1$H-NMR (300 MHz, CDCl$_3$): δ 0.85 (t, J = 7.5 Hz, 3 H), 1.18-
1.30 (m, 2 H), 1.38-1.46 (m, 2 H), 2.70 (t, \( J = 7.5 \), 2 H), 6.76 (broad s, 2 H), 6.95 (d, \( J = 9 \) Hz, 1 H), 7.11-7.22 (m, 5 H), 7.28 (d, \( J = 9 \) Hz, 1 H), 7.54 (AB, \( J = 9 \) Hz, 2 H), 7.85 (d, \( J = 9 \) Hz, 1 H), 8.03 (d, \( J = 9 \) Hz, 1 H). \(^{13}\)C-NMR (75 MHz, CD$_2$Cl$_2$): \( \delta \) 14.2, 23.1, 32.2, 39.1, 51.4, 121.3, 122.6, 122.9, 126.3, 126.5, 126.6, 128.5, 128.9, 137.0, 137.6, 139.5, 140.4, 140.7, 161.2, 164.2. FAB-MS: 343.2 [M + H]+. Anal. calc. for C$_{23}$H$_{22}$N$_2$O: C 80.67, H 6.48, N 8.18; found: C 80.61, H 6.66, N 8.21.

5c. Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH:99/1), afforded 5c as a brownish glassy product in a 66% yield. \(^1\)H-NMR (300 MHz, CDCl$_3$): \( \delta \) 0.87 (t, \( J = 7.5 \) Hz, 3 H), 1.16-1.30 (m, 6 H), 1.39-1.51 (m, 2 H), 2.69 (t, \( J = 7.5 \) Hz, 2 H), 6.77 (broad s, 2 H), 6.94 (d, \( J = 9.5 \) Hz, 1 H), 7.08-7.29 (m, 6 H), 7.55 (AB, \( J = 8.5 \) Hz, 2 H), 7.85 (d, \( J = 9.5 \) Hz, 1 H), 8.03 (d, \( J = 8.5 \) Hz, 1 H). \(^{13}\)C-NMR (75 MHz, CDCl$_3$): \( \delta \) 14.0, 22.5, 29.0, 29.5, 31.6, 38.8, 50.9, 120.8, 122.0, 122.3, 122.5, 125.8, 126.0, 127.9, 128.3, 136.4, 137.0, 139.0, 139.6, 139.8, 160.5, 164.0. Anal. calc. for C$_{25}$H$_{26}$N$_2$O: C 81.05, H 7.07, N 7.56; found: C 80.64, H 7.51, N 7.27.

5d. Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$/CH$_3$OH:99/1), afforded 5d as a slightly yellow solid in a quantitative yield from 3a, 67 % yield from 3b, 13 % yield from 3c and 28 % yield from 3d. \(^1\)H-NMR (300 MHz, CDCl$_3$): \( \delta \) 6.86 (broad s, 2H), 6.95 (d, \( J = 9.5 \) Hz, 1H), 7.15-7.21 (m, 4H), 7.28-7.37 (m, 4H), 7.57-7.61 (m, 4H), 7.87 (d, \( J = 9 \) Hz, 1H), 7.92 (d, \( J = 8.5 \) Hz, 1H), 8.22 (d, \( J = 8.5 \) Hz, 1H). \(^{13}\)C-NMR (75 MHz, CDCl$_3$): \( \delta \) 51.0, 119.7, 121.2, 122.5, 122.6, 126.2, 126.3, 126.9, 127.5, 128.2, 128.7, 129.0, 129.3, 137.4, 137.6, 138.9, 139.5, 139.6, 155.1, 163.8. Anal. calc. for C$_{25}$H$_{18}$N$_2$O: C 82.85, H 5.01, N 7.73; found: C 82.76, H 5.21, N 7.71.

Preparation of 6a. MnO$_2$ (10 g) was added to a solution of 1c (0.3 g, 0.86 mmol) in CH$_2$Cl$_2$ (30 mL). After 3 hrs MgSO$_4$ was added; the mixture was filtered on celite and evaporated. Purification by
column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH:99/1) afforded 6a (0.12 g, 0.42 mmol) as a yellow oil in a 49% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 6.42 ( broad s, 2 H), 6.52-6.54 (m, 2 H), 6.61 (s, 1 H), 6.92-6.95 (m, 3 H), 7.43-7.56 (m, 6 H), 7.67 (d, $J = 8.5$ Hz, 1 H), 8.17 (dd, $J = 8.5$ Hz, $J = 2$ Hz, 1 H), 8.50 (d, $J = 8.5$ Hz, 1 H), 8.95 (dd, $J = 6$ Hz, $J = 2$ Hz, 1 H). Anal. calc. for C$_{25}$H$_{18}$N$_2$O: C 82.85, H 5.01, N 7.73; found: C 82.81, H 5.32, N 7.51.

Preparation of 6b. MnO$_2$ (20 g) was added to a solution of 3e (0.86 g, 2 mmol) in CH$_2$Cl$_2$ (50 mL). After 2.5 hrs MgSO$_4$ was added and the mixture was filtered on celite and washed with CH$_2$Cl$_2$. Purification by crystallisation in a mixture of benzene/ether/petroleum ether afforded 6b (0.42 g, 0.95 mmol) as colorless crystals in a 67% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 6.50-6.53 (m, 2 H), 6.53 (broad s, 2 H), 6.64 (s, 1 H), 6.91-7.02 (m, 3 H), 7.47-7.75 (m, 8 H), 7.74 (d, $J = 8.5$ Hz, 1 H), 8.03 (dd, $J = 8.5$ Hz, 1 H), 8.11-8.14 (m, 2 H), 8.29 (d, $J = 8.5$ Hz, 1 H), 8.54 (d, $J = 8.5$ Hz, 1 H). $^1$H-NMR (75 MHz, CDCl$_3$): $\delta$ 57.7, 116.3, 120.0, 123.3, 124.1, 126.3, 126.9, 127.3, 128.0, 128.2, 128.6, 128.9, 129.0, 129.3, 129.6, 129.8, 136.3, 137.6, 138.4, 138.9, 141.1, 141.6, 154.7, 158.3, 177.8. Anal. calc. for C$_{31}$H$_{22}$N$_2$O: C 84.91, H 5.06, N 6.39; found: C 84.76, H 5.22, N 6.23.

Preparation of 7a-7d and 8a-b. A solution of the appropriate phenanthroline derivative (1.71 mmol) in a 47% aqueous hydrobromic acid solution (50 mL) was heated under reflux. After 1 to 5 hr the solution was allowed to cool to room temperature and then neutralised with a 1 M NaOH solution. The aqueous solution was extracted twice with CH$_2$Cl$_2$. The organic layers were collected, dried over MgSO$_4$ and then filtered before removal of the solvent. 7a. Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH 99/1 to 98/2), afforded 7a as a white solid in a quantitative yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 6.86 (dd, $J = 10$ Hz, $J = 2$ Hz, 1 H), 7.53-7.64 (m, 3 H), 7.89 (d, $J = 9.5$ Hz, 1 H),
8.21 (dd, $J = 10$ Hz, $J = 6$ Hz, 1 H), 8.92 (dd, $J = 6$ Hz, $J = 2$ Hz, 1 H), 10.69 (broad s, 1 H). $^{13}$C-NMR (75 MHz, CDCl$_3$): $\delta$ 117.3, 120.9, 123.1, 123.6, 125.2, 128.2, 135.2, 135.9, 136.5, 140.1, 149.2, 161.9. Anal. calc. for C$_{12}$H$_8$N$_2$O: C 73.46, H 4.11, N 14.28; found: C 73.46, H 4.19, N 14.21.

$^7b$ was obtained as a yellow oil in a 96% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 0.97 (t, $J = 7.5$ Hz, 3 H), 1.36-1.48 (m, 2 H), 1.77-1.87 (m, 2 H), 2.97 (t, $J = 7.5$ Hz, 2 H), 6.80 (d, $J = 9.5$ Hz, 1 H), 7.39 (d, $J = 8.5$ Hz, 1 H), 7.48 (AB, $J = 8.5$ Hz, 2 H), 7.84 (d, $J = 9.5$ Hz, 1 H), 8.06 (d, $J = 8.5$ Hz, 1 H), 10.72 (broad s, 1 H). $^{13}$C-NMR (75 MHz, CDCl$_3$): $\delta$ 13.9, 22.4, 31.6, 38.4, 117.3, 120.7, 123.3, 123.5, 124.1, 126.5, 134.9, 135.9, 136.0, 140.3, 162.0, 162.5. Anal. calc. for C$_{16}$H$_{16}$N$_2$O: C 76.16, H 6.39, N 11.10; found: C 75.98, H 6.60, N 10.96.

$^7c$. Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH 95/5), afforded $^5c$ as a yellow oil in a 93% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 0.90 (t, $J = 7.5$ Hz, 3 H), 1.25-1.46 (m, 6 H), 1.80-1.90 (m, 2 H), 2.99 (t, $J = 7.5$ Hz, 2 H), 6.82 (d, $J = 9.5$ Hz, 1 H), 7.42 (d, $J = 8.5$ Hz, 1 H), 7.52 (AB, $J = 8.5$ Hz, 2 H), 7.89 (d, $J = 9.5$ Hz, 1 H), 8.09 (d, $J = 8.5$ Hz, 1 H), 10.75 (broad s, 1 H). Anal. calc. for C$_{18}$H$_{20}$N$_2$O: C 77.11, H 7.19, N 9.99; found: C 76.88, H 7.56, N 9.71.

$^7d$. Purification by column chromatography (SiO$_2$, CH$_2$Cl$_2$ to CH$_2$Cl$_2$/CH$_3$OH 99/1), afforded $^7d$ as a yellow solid in a 90% yield. $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 6.77 (d, $J = 9.5$ Hz, 1H), 7.44-7.50 (m, 5H), 7.78 (d, $J = 9.5$ Hz, 1H), 7.90 (d, $J = 8.5$ Hz, 1H) 8.10-8.13 (m, 3H), 10.61 (broad s, 1H). $^{13}$C-NMR (75 MHz, CDCl$_3$): $\delta$ 117.6, 120.4, 120.8, 123.6, 124.9, 127.0, 127.4, 128.9, 130.0, 135.0, 136.0, 136.8, 138.0, 140.3, 155.9, 162.1. Anal. calc. for C$_{18}$H$_{12}$N$_2$O: C 79.39, H 4.44, N 10.29; found: C 79.01, H 4.31, N 10.35.

$^8a$. Purification by column chromatography (SiO$_2$, CH$_3$Cl/CH$_3$OH: 98/2 to 95/5), afforded compound $^8a$ as a white solid in a quantitative yield. $^1$H-NMR (300 MHz, DMSO-$d_6$): $\delta$ 6.88 (s, 1 H), 7.59-7.65 (m, 4
H), 7.68 (d, J = 9 Hz, 1 H), 7.82-7.86 (m, 2 H), 8.29 (dd, J = 8 Hz, J = 2 Hz, 1 H), 8.42 (d, J = 9 Hz, 1 H), 8.94 (dd, J = 4 Hz, J = 2 Hz, 1 H), 10.60 (broad s, 1 H). \(^{13}\)C-NMR (75 MHz, CDCl\(_3\)): δ 110.4, 122.0, 122.2, 123.2, 124.1, 127.1, 128.9, 129.1, 130.5, 133.8, 136.6, 137.0, 138.6, 148.6, 149.5, 176.6. FAB-MS: 273.1 [M + H]\(^+\).


8b. Purification by column chromatography (SiO\(_2\), CH\(_2\)Cl\(_2\) to CH\(_2\)Cl\(_2\)/CH\(_3\)OH: 99/1 to CH\(_2\)Cl\(_2\)/CH\(_3\)OH: 98/2), afforded 8b as a yellow solid in a 96% yield. \(^{1}\)H-NMR (300 MHz, CDCl\(_3\)): δ 6.80 (s, 1 H), 7.49-7.61 (m, 7 H), 7.75-7.79 (m, 2 H), 8.00 (d, J = 8.5 Hz, 1 H), 8.10-8.13 (m, 2 H), 8.24 (d, J = 8.5 Hz, 1 H), 8.32 (d, J = 9 Hz, 1 H), 10.54 (broad s, 1 H). \(^{13}\)C-NMR (75 MHz, CDCl\(_3\)): δ 111.1, 121.2, 1221.7, 122.8, 123.9, 126.3, 127.3, 127.9, 129.0, 129.6, 129.8, 130.7, 134.3, 136.9, 137.1, 138.4, 138.5, 147.8, 156.0, 179.5. Anal. calc. for C\(_{24}\)H\(_{16}\)N\(_2\)O: C 82.74, H 4.63, N 8.04; found: C 82.80, H 4.90, N 7.81.

X-ray crystal structures: Single crystals of 5b, 5d and 7d were mounted on a Nonius Kappa-CCD area detector diffractometer (Mo K\(\alpha\) = 0.71073 Å). The complete conditions of data collection (Denzo software) and structure refinements are given below. The cell parameters were determined from reflections taken from one set of 10 frames (1.0° steps in phi angle), each at 20 s exposure. The structures were solved using direct methods (SHELXS97) and refined against \(F^2\) using the SHELXL97 software.\(^{[1]}\) The absorption was not corrected. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were generated according to stereo-chemistry and refined using a riding model in SHELXL97. Crystallographic data (excluding structure factors) have been deposited in the Cambridge Crystallographic Data Centre as Supplementary publication n° CCDC 615458 (5b); CCDC 615459 (5d) and CCDC 615460 (7d). Copies of the data can be obtained free of charge on application to CCDC, 12
Union Road, Cambridge CB2 1EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).