**Ester (±)-21, Method A.** To a suspension of indolo[2,3-a]carbazole (18) (1.0 g, 3.9 mmol, 1.0 equiv) in 1,2-dichloroethane (130 mL) was added furanose (±)-19 (1.8 g, 8.2 mmol, 2.1 equiv) and CSA (100 mg, 0.43 mmol, 0.11 equiv). After heating at reflux for 48 h, the reaction mixture was cooled to room temperature, diluted with CH₂Cl₂ (100 mL), and washed with 10% NaHCO₃ solution. The organic layer was dried over Na₂SO₄ and chromatographed on silica gel (3:1 hexanes:EtOAc eluent) to afford indolocarbazole (±)-21 (1.37 g, 85% yield) as a yellow foam: mp 235-236 °C; IR (thin film/NaCl) 3501.3 (br m), 3047.5 (m), 3006.7 (m), 2950.6 (m), 1729.4 (s), 1640.2 (m), 1568.1 (m), 1441.1 (s), 1305.9 (s), 1230.3 (s), 1128.1 (s), 740.0 (s) cm⁻¹; ¹H NMR (500 MHz, acetone-d₆) δ 8.18 (app.t, J = 6.6 Hz, 1H), 8.18 (app.t, J = 5.4 Hz, 1H), 8.00 (m, 2H), 7.89 (d, J = 8.5 Hz, 1H), 7.75 (d, J = 8.2 Hz, 1H), 7.44 (td, J = 0.9, 7.6 Hz, 1H), 7.38 (td, J = 1.0, 7.9 Hz, 1H), 7.26 (app.t, J = 6.9 Hz, 1H), 7.25 (app.t, J = 7.1 Hz, 1H), 7.10 (dd, J = 4.9, 7.3 Hz, 1H), 5.18 (s, 1H), 3.99 (s, 3H), 3.44 (dd, J = 7.5, 14.0 Hz, 1H), 2.21 (s, 3H), 2.19 (dd, J = 4.9, 14.0 Hz, 1H); ¹³C NMR (125 MHz, acetone-d₆) δ 174.1, 140.8, 138.1, 127.7, 127.0, 125.6, 125.6, 125.5, 125.4, 121.6, 121.5, 121.2, 120.5, 120.4, 120.3, 115.0, 113.1, 112.8, 109.6, 99.9, 86.1, 86.0, 53.3, 43.2, 23.3; high resolution mass spectrum (EI) m/z 412.1419 [calcd for C₂₅H₂₆N₂O₄ (M⁺) 412.1423].

**Ester (±)-21, Method B.** To a solution of (±)-28 (100 mg, 0.26 mmol) in 1:1 MeOH/CH₂Cl₂ (14 mL) was added copper (I) chloride (700 mg, 7.1 mmol, 27 equiv) and the mixture warmed to reflux for 6 h. Solvent was removed in vacuo and the resulting residue subjected to silica gel chromatography (2:1,
hexane:EtOAc) to afford (±)-21 (102 mg, 95% yield) as a colorless solid (mp 235-239 °C).

**Thioether (±)-24 and Aldehyde (±)-23.** To a stirred solution of diol (±)-22 (100 mg, 0.26 mmol, 1.0 equiv) in 1:1 benzene:DMSO (1.8 mL) was added pyridinium trifluoroacetate (50 mg, 0.26 mmol, 1.0 equiv) followed by 1,3-dicyclohexylcarbodiimide (161 mg, 0.78 mmol, 3.0 equiv). The flask was quickly sealed with a septum, evacuated, and flushed with N₂ (3 x). The heterogeneous mixture was stirred for 7 h until reaction was complete as indicated by TLC. Benzene (4 mL) was added to the mixture and the 1,3-dicyclohexylurea (DCU) precipitate was filtered. The filtrate was washed with H₂O (3 x 10 mL), and the combined aqueous layers were back extracted with CH₂Cl₂ (3 x 15 mL). All organic layers were combined, dried over Na₂SO₄, and evaporated to an oily residue. A minimum amount of acetone (1 mL) was added to precipitate the remaining DCU. Filtration and evaporation gave a yellow oil. Flash chromatography (3:1 hexanes/EtOAc eluent) afforded two products. The first compound to elute was thioether 24 (15 mg, 13% yield) as a yellow foam: IR (thin film/NaCl) 3050.3 (w), 2922.7 (m), 2848.1 (w), 1725.3 (m), 1641.9 (m), 1570.4 (m), 1446.9 (s), 1302.6 (m), 1227.0 (m), 1099.4 (m), 745.1 (s) cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 9.34 (s, 1H), 8.16 (app.t, J = 8.5 Hz, 2H), 7.98 (d, J = 8.1 Hz, 1H), 7.94 (d, J = 8.3 Hz, 1H), 7.46-7.50 (comp m, 3H), 7.40 (app.t, J = 8.3 Hz, 1H), 7.29-7.33 (m, 2H), 7.04 (dd, J = 4.4, 7.2 Hz, 1H), 4.87 (d, J = 11.8 Hz, 1H), 4.70 (d, J = 11.9 Hz, 1H), 3.23 (dd, J = 7.3, 15.5 Hz, 1H), 2.80 (dd, J = 4.4, 15.6 Hz, 1H), 2.44 (s, 3H), 2.37 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 195.6, 137.7, 136.9, 126.5, 126.4, 125.3, 125.1, 124.7, 124.5, 121.4, 121.1, 120.8, 120.6, 120.4,
119.8, 113.1, 112.4, 112.2, 108.1, 102.3, 93.6, 86.3, 70.9, 35.4, 22.9, 14.6; high resolution mass spectrum (EI) m/z 442.1350 [calcd for C_{26}H_{22}N_{2}O_{3}S (M^+) 442.1351].

The second compound to elute was aldehyde (±)-23 (54 mg, 50% yield) as a white solid: mp 153-155 °C; IR (thin film/NaCl) 3426.4 (br m), 3049.7 (w), 2925.4 (m), 2853.1 (m), 1715.6 (m), 1640.0 (m), 1446.7 (s), 1302.8 (s), 1133.8 (s), 744.5 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.95 (s, 1H), 8.16 (app.t, \(J = 8.2\) Hz, 2H), 7.96 (d, \(J = 8.4\) Hz, 1H), 7.93 (d, \(J = 8.1\) Hz, 1H), 7.24-7.49 (m, 6H), 7.08 (app.t, \(J = 5.8\) Hz, 1H), 4.31 (br s, 1H), 2.71 (m, 2H), 2.24 (s, 3H); \(^13\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 199.3, 137.2, 136.9, 126.5, 126.4, 125.4, 125.2, 124.5, 124.2, 121.5, 120.9, 120.8, 120.6, 120.5, 119.9, 113.2, 112.2, 111.9, 108.1, 102.2, 88.0, 86.6, 42.6, 22.0; high resolution mass spectrum (EI) m/z 382.1313 [calcd for C_{24}H_{18}N_{2}O_{3} (M^+) 382.1317].

**Ester (±)-25.** A solution of aldehyde (±)-23 (100 mg, 0.263 mmol, 1.0 equiv) in DMSO (10 mL) was treated sequentially with a saturated solution of NaH\(_2\)PO\(_4\) that had been acidified to pH 2 with 1 N HCl (2.0 mL) and a solution of NaClO\(_2\) (200 mg, 2.21 mmol, 8.4 equiv). The mixture was stirred for 10 min and then treated with CH\(_2\)N\(_2\) in Et\(_2\)O until a yellow color persisted. The reaction mixture was diluted with H\(_2\)O (5 mL), extracted with Et\(_2\)O (3 x 10 mL), and the combined organic extracts dried over Na\(_2\)SO\(_4\). Flash chromatography (1:1 EtOAc:hexanes eluent) provided ester (±)-25 (92 mg, 85% yield) as a yellow foam: IR (thin film/NaCl) 3492.7 (br m), 3011.6 (m), 2951.6 (m), 2926.5 (m), 2851.6 (w), 1726.5 (s), 1640.2 (w), 1569.0 (w), 1440.8 (s), 1306.6 (s), 1230.9 (m), 1138.6 (s), 1093.4 (m), 743.4 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, acetone-d\(_6\)) \(\delta\) 8.20 (app.t, \(J = 7.3\) Hz, 2H), 8.01 (d, \(J = 8.2\) Hz, 2H), 7.95 (d, \(J = 8.1\) Hz, 1H), 7.85 (d, \(J = 8.2\) Hz, 1H), 7.29-7.44 (m, 6H), 7.08 (app.t, \(J = 5.8\) Hz, 1H), 4.28 (br s, 1H), 2.70 (m, 2H), 2.24 (s, 3H); \(^13\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 199.3, 137.2, 136.9, 126.5, 126.4, 125.4, 125.2, 124.5, 124.2, 121.5, 120.9, 120.8, 120.6, 120.5, 119.9, 113.2, 112.2, 111.9, 108.1, 102.2, 88.0, 86.6, 42.6, 22.0; high resolution mass spectrum (EI) m/z 382.1317 [calcd for C_{24}H_{18}N_{2}O_{3} (M^+) 382.1317].
Hz, 1H), 7.99 (d, J = 8.3 Hz, 1H), 7.75 (d, J = 8.2 Hz, 1H), 7.59 (d, J = 8.5 Hz, 1H), 7.45 (app.t, J = 7.5 Hz, 1H), 7.39 (app.t, J = 8.4 Hz, 1H), 7.26 (app.t, J = 7.4 Hz, 2H), 7.23 (dd, J = 4.5, 7.5 Hz, 1H), 5.54 (s, 1H), 3.04 (dd, J = 7.5, 14.9 Hz, 1H), 2.96 (s, 3H), 2.81 (dd, J = 4.7, 14.7 Hz, 1H), 2.46 (s, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ 172.2, 138.0, 137.0, 127.0, 126.2, 125.0, 124.9, 124.8, 124.5, 121.4, 120.8, 120.7, 120.3, 119.9, 119.7, 112.7, 112.1, 111.6, 108.2, 102.0, 85.9, 83.7, 53.4, 42.8, 21.9; high resolution mass spectrum (CI) m/z 413.1498 [calcd for C$_{25}$H$_{21}$N$_{2}$O$_{4}$ (M+H) 413.1501].

**Diol (±)-26** To a stirred room temperature solution of ester (±)-21 (1.0 g, 2.43 mmol, 1.0 equiv) in THF (24 mL) was added LiBH$_4$ (106 mg, 4.87 mmol, 2.0 equiv). After 20 min, the solvent was removed in vacuo to provide a white solid which was cooled to 0 °C and treated with 1.0 N HCl (50 mL). The suspension was stirred for 15 min and then extracted with CH$_2$Cl$_2$ (3 x 50 mL). The combined organic phases were dried over Na$_2$SO$_4$. Flash chromatography (1:1 hexanes:EtOAc eluent) afforded diol (±)-26 (815 mg, 87% yield) as a white solid: mp >190 °C (dec.); IR (thin film/NaCl) 3416.8 (br s), 3052.9 (m), 3010.5 (m), 2955.4 (w), 1732.7 (w), 1640.9 (m), 1568.5 (m), 1492.6 (m), 1459.0 (s), 1441.4 (s), 1309.0 (s), 1233.1 (s), 1031.9 (s), 741.0 (s) cm$^{-1}$; $^1$H NMR (500 MHz, acetone-d$_6$) δ 8.18 (d, J = 7.6 Hz, 1H), 8.15 (d, J = 7.8 Hz, 1H), 7.96 (s, 2H), 7.89 (d, J = 8.5 Hz, 1H), 7.65 (d, J = 8.1 Hz, 1H), 7.42 (app.t, J = 7.6 Hz, 1H), 7.36 (app.t, J = 8.2 Hz, 1H), 7.25 (app.t, J = 7.6 Hz, 1H), 7.23 (app.t, J = 7.4 Hz, 1H), 6.91 (dd, J = 5.2, 7.4 Hz, 1H), 4.57 (s, 1H), 4.18 (app.t, J = 5.9 Hz, 1H), 4.06 (dd, J = 5.4, 11.1 Hz, 1H), 3.90 (dd, J = 7.1, 11.1 Hz, 1H), 3.30 (dd, J = 7.6, 13.8 Hz, 1H), 2.23 (dd, J = 5.1, 13.8 Hz, 1H), 2.22 (s, 3H); $^{13}$C NMR (125 MHz, acetone-d$_6$) δ 140.2, 137.4, 127.6, 126.3,
125.4, 125.0, 124.6, 124.6, 120.7, 120.6, 119.9, 119.5, 114.6, 112.2, 112.0,
108.8, 100.1, 84.2, 83.8, 65.5, 40.6, 21.5; high resolution mass spectrum (EI)
$m/z$ 384.1472 [calcd for C$_{24}$H$_{20}$N$_2$O$_3$ (M$^+$) 384.1474].

**Aldehyde (±)-27.** To a stirred solution of diol (±)-26 (500 mg, 1.3 mmol, 1.0 equiv) in 1:1 benzene:DMSO (8.7 mL) was added pyridinium trifluoroacetate (250 mg, 1.3 mmol, 1.0 equiv) followed by 1,3-dicyclohexylcarbodiimide (810 mg, 3.9 mmol, 3.0 equiv). The flask was quickly sealed with a septum, evacuated, and flushed with N$_2$ (3 x). The heterogeneous mixture was stirred for 7 h until reaction was complete as indicated by TLC. Benzene (15 mL) was added to the mixture and the 1,3-dicyclohexylurea (DCU) precipitate was filtered. The filtrate was washed with H$_2$O (3 x 20 mL), and the combined aqueous layers were back extracted with CH$_2$Cl$_2$ (3 x 30 mL). All organic layers were combined, dried over Na$_2$SO$_4$, and evaporated to an oily residue. A minimum amount of acetone (2 mL) was added to precipitate the remaining DCU. Filtration and evaporation gave a yellow oil, which was purified by flash chromatography (3:1 hexanes:EtOAc eluent) to afford aldehyde (±)-27 (373 mg, 73% yield, 63% yield 2 steps) as a yellow powder: mp $>225$ °C (dec.); IR (thin film/NaCl) 3486.7 (br m), 3054.6 (m), 3007.7 (m), 2945.3 (m), 2843.4 (w), 1723.9 (m), 1641.8 (m), 1568.6 (m), 1458.7 (m), 1441.1 (s), 1309.2 (s), 1232.5 (s), 1128.8 (m), 1004.2 (m), 741.7 (s) cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 9.70 (s, 1H), 7.99 (app.t, $J = 7.3$ Hz, 2H), 7.78 (s, 2H), 8.02 (d, $J = 8.4$ Hz, 1H), 7.29 (app.t, $J = 7.4$ Hz, 1H), 7.24 (app.t, $J = 7.2$ Hz, 1H), 7.22 (d, $J = 8.4$ Hz, 1H), 7.17 (app.t, $J = 7.9$ Hz, 1H), 7.15 (app.t, $J = 7.2$ Hz, 1H), 6.59 (dd, $J = 5.0$, 7.4 Hz, 1H), 3.08 (s, 1H), 2.76 (dd, $J = 7.6$, 14.6 Hz, 1H), 1.99 (s, 3H), 1.83 (dd, $J = 5.0$, 14.7 Hz, 1H); $^{13}$C NMR (125 MHz,
CDCl₃ δ 199.4, 139.3, 136.9, 126.3, 126.3, 125.1, 124.7, 124.1, 121.2, 121.1, 120.8, 120.3, 120.3, 119.9, 113.1, 112.9, 112.2, 108.0, 97.7, 87.7, 84.0, 39.7, 23.0; high resolution mass spectrum (EI) m/z 382.1319 [calcd for C₂₄H₁₈N₂O₃ (M⁺) 382.1317].

**Hydroxy ketone (±)-28.** A suspension of aldehyde (±)-27 (75 mg, 0.196 mmol, 1.0 equiv) in Et₂O (5.0 mL) was treated with BF₃•OEt₂ (27 µL, 0.220 mmol, 1.1 equiv), and stirred vigorously for 6 h. After addition of CH₂Cl₂ (25 mL) to solubilize the suspension, the resulting solution was evaporated onto SiO₂ (100 mg) and chromatographed (2:1 hexanes:EtOAc eluent) to provide ketone (±)-28 (45 mg, 60% yield) as a white powder: mp 235-239 °C (dec.); IR (thin film/NaCl) 3328.6 (br m), 3048.0 (w), 2923.7 (m), 2852.1 (w), 1731.4 (s), 1637.4 (m), 1441.5 (s), 1395.3 (m), 1312.0 (s), 1130.1 (m), 740.8 (s) cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 8.15 (d, J = 7.7 Hz, 1H), 8.10 (d, J = 7.7 Hz, 1H), 7.97 (d, J = 8.5 Hz, 1H), 7.92 (d, J = 8.2 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.43 (app.t, J = 7.7 Hz, 1H), 7.39 (app.t, J = 7.8 Hz, 1H), 7.32 (app.t, J = 7.4 Hz, 1H), 7.28 (app.t, J = 7.5 Hz, 1H), 7.25 (d, J = 8.1 Hz, 1H), 7.06 (d, J = 7.3 Hz, 1H), 4.89 (d, J = 6.0 Hz, 1H), 3.55 (dd, J = 7.5, 14.3 Hz, 1H), 3.49 (d, J = 6.5 Hz, 1H), 2.99 (d, J = 14.4 Hz, 1H), 2.54 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 199.8, 140.2, 136.3, 126.4, 125.7, 125.4, 125.1, 124.8, 124.6, 121.4, 120.8, 120.4, 120.2, 119.8, 115.2, 112.7, 112.4, 112.1, 107.9, 100.3, 84.0, 81.6, 44.7, 29.5; high resolution mass spectrum (EI) m/z 382.1315 [calcd for C₂₄H₁₈N₂O₃ (M⁺) 382.1317].

**Diol (±)-29.** To a stirred room temperature solution of ketone (±)-28 (100 mg, 0.26 mmol, 1.0 equiv) in 1:1 MeOH: CH₂Cl₂ (10 mL) was added NaBH₄ (27 mg, 0.70
mmol, 2.7 equiv). After 5 min solvent was removed under reduced pressure to afford a white solid which was cooled to 0 °C and then treated with 1.0 N HCl (10 mL). After 5 min at 0 °C, the mixture was warmed to room temperature, stirred for 15 min at 25 °C, and extracted with CH₂Cl₂ (3 x 10 mL). The combined organic layers were dried with Na₂SO₄ and chromatographed (2:1 hexanes:EtOAc eluent) to afford diol (±)-29 (95 mg, 95% yield) as a white solid: mp 235-238 °C (dec.); IR (thin film/NaCl) 3529.6 (br m), 3043.9 (m), 2930.2 (w), 1642.5 (m), 1564.1 (m), 1445.1 (s), 1314.4 (s), 1230.1 (m), 1129.9 (m), 1073.3 (m), 739.3 (s), 694.3 (m) cm⁻¹; ¹H NMR (500 MHz, acetone-d₆) δ 8.16 (d, J = 7.6 Hz, 1H), 8.11 (d, J = 7.9 Hz, 1H), 8.00 (d, J = 8.5 Hz, 1H), 7.88 (d, J = 8.3 Hz, 1H), 7.85 (d, J = 8.3 Hz, 1H), 7.49 (d, J = 8.1 Hz, 1H), 7.40 (app.t, J = 7.6 Hz, 1H), 7.31 (ddd, J = 1.3, 7.0, 11.4 Hz, 1H), 7.24 (app.t, J = 7.3 Hz, 1H), 7.17 (app.t, J = 7.2 Hz, 1H), 6.71 (dd, J = 1.0, 5.8 Hz, 1H), 4.21 (m, 1H), 3.99 (dd, J = 3.3, 9.1 Hz, 1H), 3.59 (br s, 1H), 2.80 (d, J = 13.6 Hz, 1H), 2.74 (ddd, J = 3.0, 5.7, 14.9 Hz, 1H), 2.63 (ddd, J = 1.0, 3.3, 15.0 Hz, 1H), 2.32 (s, 3H); ¹³C NMR (125 MHz, DMSO-d₆) δ 139.4, 136.1, 127.5, 126.6, 125.2, 124.2, 123.8, 123.7, 119.9, 119.1, 118.8, 118.5, 118.3, 115.1, 111.3, 110.5, 108.7, 92.2, 79.5, 73.3, 64.0, 34.4, 29.5; high resolution mass spectrum (EI) m/z 384.1469 [calcd for C₂₄H₂₀N₂O₃ (M⁺) 384.1474].

**Bis p-bromobenzoate (±)-30.** Diol (±)-29 (30 mg, 0.078 mmol, 1.0 equiv), p-bromobenzoyle chloride (36 mg, 0.164 mmol, 2.1 equiv), Et₃N (23 µL, 0.164 mmol, 2.1 equiv), and 4-dimethylaminopyridine (2 mg, 0.016 mmol, 0.1 equiv) were heated to reflux in CH₂Cl₂ (1.0 mL) for 10 min. The reaction mixture was adsorbed onto SiO₂ and chromatographed (2:1 hexanes:EtOAc eluent) to afford (±)-30 (45 mg, 77%
yield) as a white solid which when crystallized from CHCl₃/MeOH provided crystals suitable for X-ray analysis: mp 198-200 °C; IR (thin film/NaCl) 3044.9 (w), 2928.5 (w), 1725.4 (s), 1642.6 (w), 1589.4 (s), 1398.4 (m), 1258.6 (s), 1230.5 (m), 1091.8 (br s), 1009.9 (s), 844.4 (w), 739.5 (s) cm⁻¹; ^1H NMR (500 MHz, CDCl₃) δ 8.15 (d, J = 8.5 Hz, 1H), 8.13 (d, J = 9.3 Hz, 1H), 8.00 (d, J = 8.2 Hz, 1H), 7.98 (d, J = 8.2 Hz, 1H), 7.44 (app.t, J = 8.4 Hz, 1H), 7.42 (app.t, J = 7.5 Hz, 1H), 7.30 (s, 4H), 7.27 (m, 1H), 7.26 (app.t, J = 8.2 Hz, 1H), 7.21 (app.t, J = 7.4 Hz, 1H), 7.10 (ddd, J = 1.2, 7.2, 8.4 Hz, 1H), 7.00 (d, J = 8.4 Hz, 2H), 6.76 (d, J = 5.5 Hz, 1H), 6.08 (d, J = 8.3 Hz, 2H), 5.85 (m, 1H), 5.84 (s, 1H), 2.94 (ddd, J = 3.6, 5.7, 15.4 Hz, 1H), 2.78 (dd, J = 2.8, 15.5 Hz, 1H), 2.39 (s, 3H); ^13C NMR (125 MHz, CDCl₃) δ 164.9, 164.8, 138.6, 136.3, 131.8, 131.6, 131.1, 130.5, 129.0, 127.8, 127.1, 127.0, 126.8, 126.0, 125.0, 124.6, 124.5, 121.0, 120.7, 120.2, 120.0, 119.8, 119.8, 114.1, 112.5, 111.5, 107.7, 90.0, 79.6, 73.3, 63.7, 32.4, 30.2; high resolution mass spectrum (EI) m/z 748.0214 [calcd for C₃₈H₂₆N₂O₅Br₂ (M⁺) 748.0208].

**Methoxy ketone (±)-31.** Montmorillonite clay K-10 (1.2 g) was mixed with trimethylorthoformate (1.78 mL, 16.3 mmol, 14.8 equiv) and immediately rinsed (3 mL CHCl₃) into a stirred solution of aldehyde (±)-27 (414 mg, 1.1 mmol, 1.0 equiv) in CHCl₃ (11 mL). After approximately 0.5 h formation of the dimethyl acetal 33 was complete as indicated by TLC (3:1 hexanes:EtOAc). The reaction mixture was filtered and the filtrate evaporated in vacuo. The residue was dissolved in diethyl ether (110 mL) under N₂ and treated with BF₃•OEt₂ (2.85 mL, 23.1 mmol, 21.0 equiv). The resultant mixture was stirred for 4 days at 25 °C. After this time, Et₃N (6.1 mL) and CH₂Cl₂ (100 mL) were added and the product was adsorbed onto silica
gel in vacuo. Flash chromatography (2:1 hexanes:EtOAc eluent) provided methoxy ketone (±)-31 (214 mg, 50% yield) as a yellow foam: mp 275-280 °C (dec.); IR (thin film/NaCl) 3046.6 (br m), 3003.8 (br w), 2927.9 (m), 2835.6 (m), 1736.6 (s), 1640.5 (m), 1565.8 (m), 1492.7 (m), 1442.9 (s), 1311.5 (s), 1144.3 (m), 1126.1 (s), 740.2 (s) cm⁻¹; ¹H NMR (500 MHz, DMSO-d₆) δ 8.21 (d, J = 7.7 Hz, 1H), 8.16 (d, J = 7.8 Hz, 1H), 7.97 (d, J = 8.2 Hz, 1H), 7.95 (d, J = 8.2 Hz, 1H), 7.88 (d, J = 8.6 Hz, 1H), 7.68 (d, J = 8.1 Hz, 1H), 7.46 (td, J = 1.0, 7.4 Hz, 1H), 7.37 (td, J = 1.1, 7.7 Hz, 1H), 7.36 (d, J = 7.2 Hz, 1H), 7.30 (app. t, J = 7.6 Hz, 1H), 7.23 (app. t, J = 7.4 Hz, 1H), 5.02 (s, 1H), 3.94 (dd, J = 7.2, 13.7 Hz, 1H), 3.39 (s, 3H), 2.62 (d, J = 13.9 Hz, 1H), 2.52 (s, 3H); ¹³C NMR (125 MHz, DMSO-d₆) δ 199.8, 139.4, 135.7, 125.0, 124.8, 124.5, 124.1, 124.0, 120.0, 119.8, 119.4, 119.2, 114.9, 112.1, 111.3, 109.2, 99.0, 88.2, 84.4, 58.9, 45.4, 29.2; high resolution mass spectrum (EI) m/z 396.1474 [calcd for C₂₅H₂₀N₂O₃ (M⁺) 396.1474].

**Alcohol (±)-37. Method A.** To a stirred solution of ketone (±)-31 (12 mg, 0.03 mmol, 1.0 equiv) in 1:1 MeOH: CH₂Cl₂ (1.0 mL) was added NaBH₄ (3 mg, 0.08 mmol, 2.7 equiv) at room temperature. After 5 min the solvent was removed in vacuo to afford a white solid which was cooled to 0 °C and treated with 1.0 N HCl (1 mL). After 5 min at 0 °C, the mixture was warmed to room temperature, stirred for 15 min at 25 °C, and extracted with CH₂Cl₂ (3 x 1 mL). The combined organic layers were dried with Na₂SO₄ and chromatographed (2:1 hexanes:EtOAc eluent) to afford alcohol (±)-37 (12 mg, 95% yield) as a white solid: mp 340-344 °C (dec.); IR (thin film/NaCl) 3528.3 (br m), 3048.1 (m), 3000.2 (m), 2928.4 (m), 1643.7 (m), 1564.8 (m), 1493.3 (m), 1445.1 (s), 1344.4 (m), 1311.6 (s), 1231.2 (s), 1109.5
(br s) cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 8.14 (d, J = 7.7 Hz, 1H), 8.11 (d, J = 7.7 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.85 (d, J = 8.2 Hz, 1H), 7.81 (d, J = 8.5 Hz, 1H), 7.39 (td, J = 1.0, 8.1 Hz, 1H), 7.35 (ddd, J = 0.14, 7.1, 8.4 Hz, 1H), 7.25 (m, 3H), 6.54 (d, J = 5.6 Hz, 1H), 4.34 (m, 1H), 3.66 (d, J = 3.0 Hz, 1H), 3.53 (s, 3H), 2.71 (dd, J = 3.5, 14.9 Hz, 1H), 2.45 (m, 1H), 2.30 (s, 3H), 1.66 (br s, 1H);
¹³C NMR (125 MHz, CDCl₃) δ 139.6, 136.6, 128.3, 127.2, 126.5, 126.2, 124.8, 124.4, 123.9, 120.5, 120.3, 119.6, 119.3, 114.9, 112.1, 110.9, 107.6, 90.6, 83.1, 79.7, 60.5, 57.4, 33.7, 29.9; high resolution mass spectrum (EI) m/z 398.1633 [calcd for C₂₅H₂₂N₂O₃ (M⁺) 398.1630].

**Alcohol (±)-37. Method B.** To a stirred suspension of NaH (6.1 mg of a 60% dispersion in mineral oil, 0.15 mmol, 1.1 equiv) in THF (1.0 mL) was added a solution of alcohol (±)-29 (55 mg, 0.143 mmol, 1.0 equiv) in THF (5 mL). The resulting mixture was stirred for 10 min with the visible evolution of gas and for an additional 15 min thereafter. Addition of MeI (9.0 µL, 0.15 mmol, 1.1 equiv) produced a single product as evidenced by TLC (5:1 hexanes:acetone). After approximately 50 min the reaction was quenched by the sequential addition of 1.0 N HCl (1.0 mL) and H₂O (2.0 mL). Extraction of the solution with CH₂Cl₂ (3 x 10 mL), drying over Na₂SO₄, evaporation to a residue in vacuo, and chromatography (5:1 hexanes:acetone eluent) provided methyl ether (±)-37 (42 mg, 70% yield) as a yellow foam.

**Bis Methyl ether (±)-35, Method A.** A stirred room temperature solution of alcohol (±)-37 (19 mg) in DMSO (3 mL) was treated with excess MeI (5-10 equiv) and KOH (5-10 equiv) for 5 min. After this time, the reaction was diluted with H₂O (5 mL) and extracted with CH₂Cl₂ (3 x 10 mL). The combined organic layers were dried over Na₂SO₄, and
evaporated. Purification by flash chromatography (1:1 hexanes:EtOAc eluent) provided bis-methyl ether (±)-35 (20 mg, 95% yield) as a yellow foam: mp 218-224 °C (dec.); IR (thin film/NaCl) 3048.0 (m), 3003.5 (m), 2930.1 (m), 2829.9 (m), 1642.3 (m), 1565.2 (m), 1492.8 (w), 1460.9 (s), 1444.8 (s), 1396.0 (s), 1314.4 (s), 1230.2 (s), 1118.9 (s), 1035.8 (m), 740.7 (s) cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 8.16 (d, J = 7.9 Hz, 1H), 8.09 (d, J = 7.0 Hz, 1H), 7.88 (d, J = 8.3 Hz, 1H), 7.86 (d, J = 8.3 Hz, 1H), 7.81 (d, J = 8.5 Hz, 1H), 7.41 (app.t, J = 7.3 Hz, 1H), 7.32 (ddd, J = 1.2, 7.2, 8.4 Hz, 1H), 7.28 (app.t, J = 7.4 Hz, 1H), 7.27 (d, J = 7.7 Hz, 1H), 7.20 (app.t, J = 7.4 Hz, 1H), 6.57 (d, J = 5.1 Hz, 1H), 3.99 (dt, J = 3.0, 6.4 Hz, 1H), 3.75 (d, J = 2.9 Hz, 1H), 3.52 (s, 3H), 2.85 (ddd, J = 0.9, 3.9, 15.0 Hz, 1H), 2.38 (s, 3H), 2.33 (ddd, J = 2.7, 5.5, 14.9 Hz, 1H), 2.32 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 139.6, 136.3, 127.7, 126.6, 126.0, 124.9, 124.2, 124.0, 120.5, 119.6, 119.2, 119.0, 114.6, 111.4, 111.2, 107.3, 91.3, 84.3, 80.0, 68.6, 57.5, 55.7, 30.3, 29.9; high resolution mass spectrum (EI) m/z 412.1784 [calcd for C₂₆H₂₄N₂O₃ (M⁺) 412.1787].

**Bis Methyl Ether (±)-35, Method B.** A procedure identical to that describe above for the alkylation of 37 was utilized for the conversion of diol (±)-29 (20 mg) to (±)-35 in 93% yield.

**Oxime (±)-36.** A suspension of ketone (±)-31 (30 mg, 0.08 mmol, 1.0 equiv), hydroxylamine hydrochloride (17 mg, 0.24 mmol, 3.0 equiv), and NaOAc (20 mg, 0.24 mmol, 3.0 equiv) in 50% aqueous EtOH (2.0 mL) was heated gently to reflux for 30 min. After cooling to room temperature, the solvent was removed in vacuo and the derived residue was purified by flash chromatography (2:1 hexanes:EtOAc eluent) to provide oxime (±)-36 (27 mg, 85% yield) as a yellow powder: mp >280
°C (dec.); IR (thin film/NaCl) 3249.5 (br m), 2918.3 (s), 2848.4 (s), 1728.1 (m),
1640.2 (m), 1443.1 (s), 1398.1 (m), 1312.0 (m), 1124.5 (s), 740.7 (s) cm⁻¹; ¹H
NMR (500 MHz, DMSO-d₆) δ 10.43 (s, 1H), 8.17 (d, J = 7.8 Hz, 1H), 8.13 (d, J =
7.4 Hz, 1H), 7.91 (d, J = 8.4 Hz, 1H), 7.89 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.4
Hz, 1H), 7.67 (d, J = 8.2 Hz, 1H), 7.44 (app.t, J = 7.6 Hz, 1H), 7.34 (app.t, J =
7.7 Hz, 1H), 7.27 (app.t, J = 7.5 Hz, 1H), 7.20 (app.t, J = 7.4 Hz, 1H), 6.98 (d, J =
5.5 Hz, 1H), 4.70 (s, 1H), 3.61 (d, J = 14.1 Hz, 1H), 3.42 (s, 3H), 2.97 (dd, J =
5.7, 14.3 Hz, 1H), 2.42 (s, 3H); ¹³C NMR (125 MHz, DMSO-d₆) δ 145.3, 139.3,
135.9, 126.0, 125.1, 124.9, 124.6, 124.2, 124.0, 120.0, 119.6, 119.4, 119.1,
119.1, 115.0, 111.8, 111.0, 109.1, 95.9, 83.7, 82.2, 58.3, 29.7, 28.4; high
resolution mass spectrum (EI) m/z 411.1582 [calcd for C$_{25}$H$_{21}$N$_{3}$O$_{3}$ (M$^+$)
411.1583].

**Amine (±)-38.** A mixture of oxime (±)-36 (20 mg, 0.049
mmol, 1.0 equiv) and PtO$_2$ (5 mg) in a 60% aqueous
acetic acid (6.0 mL) was placed in a flasks capped with
a H$_2$ filled balloon. The reaction was monitored by TLC
(1:1 hexanes:EtOAc) and upon completion (2 h) was
filtered through celite. The filtrate was evaporated in
vacuo and the residue was dissolved in 1.0 N HCl (4.0 mL) and washed with
EtOAc (1 x 4.0 mL). The aqueous layer was rendered basic with 3.0 N NaOH
and then extracted with EtOAc (3 x 5.0mL). The combined organic layers were
dried over Na$_2$SO$_4$ and evaporated to a residue which was used without further
purification.

An analytical sample of the derived primary amine could be obtained by
preparative TLC of the above residue (5% MeOH/CH$_2$Cl$_2$ eluent): mp >225 °C
(dec.); IR (thin film/NaCl) 3373.6 (br w), 3048.2 (br w), 2926.7 (br m), 2851.2
(br w), 1641.4 (m), 1563.9 (m), 1492.2 (m), 1459.0 (s), 1444.2 (s), 1314.0 (s), 1231.1 (s), 1110.2 (s), 741.3 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.16 (d, \(J = 8.0\) Hz, 1H), 8.12 (d, \(J = 7.8\) Hz, 1H), 7.91 (d, \(J = 8.2\) Hz, 1H), 7.89 (d, \(J = 8.2\) Hz, 1H), 7.86 (d, \(J = 8.4\) Hz, 1H), 7.43 (app. t, \(J = 7.6\) Hz, 1H), 7.38 (app. t, \(J = 7.6\) Hz, 1H), 7.27 (m, 3H), 6.55 (dd, \(J = 1.0, 5.5\) Hz, 1H), 3.71 (d, \(J = 3.7\) Hz, 1H), 3.65 (br m, 1H), 3.41 (s, 3H), 2.64 (dt, \(J = 5.3, 14.6\) Hz, 1H), 2.58 (ddd, \(J = 1.1, 3.4, 14.4\) Hz, 1H), 2.3 (s, 3H), 1.24 (br s, 2H); \(^1\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 139.5, 136.8, 126.8, 126.1, 125.8, 124.9, 124.6, 124.2, 120.6, 120.5, 119.8, 119.7, 119.5, 119.4, 114.7, 112.2, 111.4, 108.0, 91.2, 84.0, 80.3, 57.6, 42.7, 34.5, 29.7; high resolution mass spectrum (EI) \(m/z\) 397.1789 [calcd for C\(_{25}\)H\(_{23}\)N\(_3\)O\(_2\) (M\(^+\)) 397.1790].

The derived residue was dissolved in THF (2.0 mL) and treated with an excess of formic acetic anhydride (3 equiv, prepared by treatment of 1.0 equiv acetic anhydride with 1.2 equiv formic acid followed by reflux for 2 h) in THF. TLC analysis showed rapid formation of a less polar substance. The solvent was evaporated with a stream of N\(_2\) followed by high vacuum (ca. 1 torr) for 15 min. The derived residue was dissolved in THF (2.0 mL), cooled to 0 °C, and treated with BH\(_3\)•DMS (61 \(\mu\)L of a 2.0 N solution in toluene). The solution was heated to reflux for 2 h and then cooled to 0 °C. Methanolic HCl (4.0 mL) was added and the solution was refluxed for an additional hour. After cooling to room temperature, volatiles were removed \textit{in vacuo} leaving a solid residue to which was added 1.0 N NaOH (1.5 mL). The mixture was extracted with EtOAc (3 x 3.5 mL) and the combined organic layers were dried over Na\(_2\)SO\(_4\). Purification of the residue by flash chromatography (10% MeOH/CH\(_2\)Cl\(_2\) eluent) provided methyl amine (\(\pm\))-38 (14 mg, 70% yield 2 steps) as a white powder: mp 238-242 °C (dec.); IR (thin film/NaCl) 3344.1 (w), 3043.9 (m),
3000.7 (m), 2929.4 (m), 2850.6 (m), 2796.2 (m), 1642.5 (m), 1562.9 (m), 1442.1 (s), 1396.0 (m), 1341.5 (m), 1311.0 (s), 1232.2 (s), 1111.4 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.19 (d, \(J = 7.9\) Hz, 1H), 8.16 (d, \(J = 7.3\) Hz, 1H), 7.93 (d, \(J = 8.2\) Hz, 1H), 7.90 (d, \(J = 8.2\) Hz, 1H), 7.85 (d, \(J = 8.5\) Hz, 1H), 7.44 (td, \(J = 1.0, 7.7\) Hz, 1H), 7.39 (ddd, \(J = 1.3, 7.1, 8.4\) Hz, 1H), 7.31 (app.t, \(J = 7.7\) Hz, 1H), 7.27 (app.t, \(J = 8.0\) Hz, 1H), 7.26 (d, \(J = 7.6\) Hz, 1H), 6.51 (dd, \(J = 1.3, 6.1\) Hz, 1H), 3.83 (d, \(J = 3.5\) Hz, 1H), 3.30 (s, 3H), 3.29 (dd, \(J = 4.1, 4.7\) Hz, 1H), 2.63 (ddd, \(J = 1.5, 4.7, 14.5\) Hz, 1H), 2.38 (ddd, \(J = 4.0, 6.1, 14.6\) Hz, 1H), 2.34 (s, 3H), 1.69 (s, 3H), 0.84 (br s, 1H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 139.2, 136.6, 128.2, 127.3, 126.0, 124.7, 124.2, 123.8, 120.3, 120.0, 119.5, 119.2, 119.0, 119.0, 114.2, 111.7, 110.8, 107.5, 91.3, 84.0, 80.5, 57.5, 50.8, 33.4, 30.6, 29.7; high resolution mass spectrum (EI) \(m/z\) 411.1944 [calcd for \(C_{26}H_{25}N_3O_2\) (M\(^+\)) 411.1947].

**Ketones (±)-44 and (±)-45.** To a suspension of aldehyde (±)-23 (56 mg, 0.147 mmol, 1.0 equiv) in Et\(_2\)O (15.0 mL) was added BF\(_3\)•OEt\(_2\) (20 \(\mu\)L, 0.161 mmol, 1.1 equiv). The mixture was stirred vigorously for 7 h and then treated with CH\(_2\)Cl\(_2\) (25 mL) to solubilize the suspension. The resulting solution was adsorbed onto silica *in vacuo* and chromatographed (3:1 hexanes:EtOAc eluent) to provide two products. The first compound to elute, hydroxy ketone (±)-44 (12 mg, 21% yield), was isolated as a yellow foam: IR (thin film/NaCl) 3461.9 (br m), 2924.5 (m), 1731.8 (s), 1570.7 (m), 1447.5 (m), 1389.4 (m), 1307.5 (s), 1227.4 (s), 1133.2 (s), 747.2 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.19 (d, \(J = 4.8\) Hz, 1H), 8.17 (d, \(J = 4.7\) Hz,
1H), 7.98 (s, 2H), 7.68 (d, J = 8.3 Hz, 1H), 7.45-7.51 (comp m, 2H), 7.32-7.40 (comp m, 3H), 6.79 (dd, J = 4.8, 6.1 Hz, 1H), 5.13 (s, 1H), 3.65 (s, 1H), 3.41 (dd, J = 6.3, 15.9 Hz, 1H), 3.36 (dd, J = 4.7, 15.9 Hz, 1H), 2.22 (s, 3H); 13C NMR (125 MHz, CDCl3) δ 204.0, 138.4, 137.2, 126.2, 125.9, 125.8, 125.5, 125.4, 125.3, 121.8, 121.2, 120.9, 120.6, 120.5, 120.4, 113.2, 112.8, 111.7, 108.2, 99.6, 83.8, 78.9, 42.6, 23.7; high resolution mass spectrum (EI) m/z 382.1319 [calcd for C24H18N2O3 (M⁺) 382.1317].

The second compound to elute, ketone (±)-45 (36 mg, 64% yield), was isolated as a white solid: mp >230 °C (dec); IR (thin film/NaCl) 3458.1 (br m), 3053.6 (w), 2924.4 (m), 2855.9 (m), 1706.1 (s), 1655.1 (m), 1568.7 (m), 1447.9 (s), 1404.9 (s), 1342.9 (s), 1036.7 (m), 747.1 (s) cm⁻¹; 1H NMR (500 MHz, DMSO-d₆, 315 K) δ 8.19 (d, J = 7.9 Hz, 1H), 8.17 (d, J = 7.8 Hz, 1H), 7.98 (d, J = 8.4 Hz, 1H), 7.96 (d, J = 8.4 Hz, 1H), 7.83 (d, J = 8.2 Hz, 1H), 7.71 (d, J = 8.2 Hz, 1H), 7.45 (app.t, J = 7.6 Hz, 1H), 7.44 (app.t, J = 8.2 Hz, 1H), 7.36 (dd, J = 3.0, 7.5 Hz, 1H), 7.24-7.28 (m, 2H), 6.93 (s, 1H), 6.71 (s, 1H), 2.81 (dd, J = 7.5, 14.7 Hz, 1H), 2.47 (dd, J = 2.8, 14.8 Hz, 1H), 2.06 (s, 3H); 13C NMR (125 MHz, DMSO-d₆) δ 206.0, 137.5, 137.0, 125.3, 124.9, 124.9, 124.3, 124.0, 123.9, 120.3, 120.2, 119.7, 119.7, 119.5, 112.3, 112.2, 109.5, 109.4, 93.0, 87.8, 87.6, 42.8, 26.7; high resolution mass spectrum (EI) m/z 382.1312 [calcd for C24H18N2O3 (M⁺) 382.1317].

**Epoxide (±)-46.** To a solution of diol (±)-26 (100 mg, 0.26 mmol, 1.0 equiv) and p-toluene sulfonyl chloride (52 mg, 0.27mmol, 1.05 equiv) in THF (2.6 mL) was added powdered KOH (36 mg, 0.65 mmol, 2.5 equiv) followed by n-Bu₄NBr (8 mg, 0.03 mmol, 0.1 equiv). The reaction mixture was stirred for 1 h, and solvent was removed in vacuo. Purification by
flash chromatography (3:1→1:1 hexanes:EtOAc) provided epoxide 46 (86 mg, 90% yield) as a white foam: mp >260 °C (dec.); IR (thin film/NaCl) 3049.6 (w), 3010.0 (w), 2947.3 (br w), 1638.2 (m), 1567.7 (m), 1445.6 (s), 1347.0 (m), 1307.5 (s), 1225.4 (m), 1026.0 (m), 744.9 (s) cm\(^{-1}\); \(^1\)H NMR (500 MHz, DMSO-d\(_6\)) \(\delta\) 8.21 (app.t, \(J = 8.2\) Hz, 2H), 8.02 (d, \(J = 8.4\) Hz, 1H), 7.98 (d, \(J = 8.1\) Hz, 1H), 7.78 (d, \(J = 8.5\) Hz, 1H), 7.72 (d, \(J = 8.2\) Hz, 1H), 7.45 (app.t, \(J = 7.6\) Hz, 1H), 7.36 (app.t, \(J = 7.7\) Hz, 1H), 7.28 (app.t, \(J = 8.2\) Hz, 1H), 7.24 (app.t, \(J = 7.8\) Hz, 1H), 7.15 (d, \(J = 6.0\) Hz, 1H), 3.28 (dd, \(J = 6.1, 14.9\) Hz, 1H), 3.23 (d, \(J = 3.8\) Hz, 1H), 2.98 (d, \(J = 4.2\) Hz, 1H), 2.41 (s, 3H), 1.90 (d, \(J = 14.8\) Hz, 1H); \(^{13}\)C NMR (125 MHz, DMSO-d\(_6\)) \(\delta\) 138.4, 136.5, 126.8, 125.2, 125.1, 124.9, 123.9, 120.4, 120.2, 120.1, 119.9, 119.6, 119.5, 112.6, 111.7, 111.6, 109.3, 96.7, 84.7, 66.5, 50.4, 37.4, 24.6; high resolution mass spectrum (EI) \(m/z\) 366.1363 [calcd for \(\text{C}_{24}\text{H}_{18}\text{N}_{2}\text{O}_{2}\) (M\(^{+}\)) 366.1368].

**Ketone (±)-47.** A solution of epoxide (±)-46 (50 mg, 0.14 mmol, 1.0 equiv) in CDCl\(_3\) (13.7 mL) was treated with BF\(_3\)•OEt\(_2\) (18μL, 0.15 mmol, 1.05 equiv) and stirred at room temperature for 15 h. Following removal of solvent *in vacuo*, flash chromatography provided ketone 47 (47 mg, 92% yield) as a white foam: mp 180-183°C; IR (thin film/NaCl) 3420.6 (br w), 3051.0 (w), 2924.3 (w), 1927.2 (w), 1717.8 (s), 1638.0 (m), 1568.9 (m), 1446.0 (s), 1347.6 (s), 1310.8 (s), 1219.7 (s), 1071.7 (m) cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.19 (d, \(J = 7.8\) Hz, 1H), 8.13 (d, \(J = 7.8\) Hz, 1H), 8.00 (d, \(J = 8.4\) Hz, 1H), 7.98 (d, \(J = 8.1\) Hz, 1H), 7.51 (d, \(J = 8.1\) Hz, 1H), 7.30-7.47 (comp m, 4H), 7.16 (d, \(J = 8.4\) Hz, 1H), 6.69 (d, \(J = 7.4\) Hz, 1H), 4.89 (d, \(J = 9.7\) Hz, 1H), 4.01 (dd, \(J = 0.9, 9.8\) Hz, 1H), 3.17 (d, \(J = 13.6\) Hz, 1H), 2.97 (dd, \(J = 7.5, 13.6\) Hz, 1H), 2.37 (s, 3H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 203.4, 138.6, 138.6, 127.1, 125.6, 125.3, 125.3,
124.5, 121.8, 121.6, 120.7, 120.5, 120.4, 120.3, 113.0, 112.7, 109.5, 108.3, 83.1, 75.7, 71.9, 45.6, 27.6; high resolution mass spectrum (EI) m/z 366.1363 [calcd for C_{24}H_{18}N_{2}O (M^+) 366.1368].

**Ether (±)-50.** A solution of diol (±)-29 (38 mg, 0.10 mmol, 1.0 equiv) and Bu₂Sn(OMe)₂ (25 µL, 0.11 mmol, 1.1 equiv) in benzene (5.0 mL) was heated to reflux with azeotropically removal of H₂O (Dean-Stark apparatus) for 1 h. The solvent was removed in vacuo, followed by addition of CH₃CN (5.0 mL), MeI (6.8 µL, 0.11 mmol, 1.1 equiv), and Ag₂O (25 mg, 0.11, 1.1 equiv). The resulting mixture was heated at reflux over 4 h, diluted with H₂O (3 mL), and extracted with CH₂Cl₂ (3 x 2 mL). The combined organic extracts were dried over Na₂SO₄ and purified by flash chromatography (5:1 hexanes:acetone) to provide recovered diol (±)-29 (8 mg) and ether (±)-50 (6 mg, 15% yield) as a yellow foam: mp 213-217 °C (dec.); IR (thin film/NaCl) 3535.7 (br w), 3413.5 (w), 2963.2 (m), 2924.4 (s), 2853.7 (m), 1437.1 (m), 1314.6 (s), 735.0 (m) cm⁻¹; ¹H NMR (500 MHz, acetone-d₆) δ 8.18 (d, J = 7.0 Hz, 1H), 8.11 (d, J = 7.7 Hz, 1H), 7.97 (d, J = 8.6 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.87 (d, J = 8.2 Hz, 1H), 7.54 (d, J = 8.1 Hz, 1H), 7.42 (app.t, J = 7.3 Hz, 1H), 7.30 (app.t, J = 8.4 Hz, 1H), 7.25 (app.t, J = 7.7 Hz, 1H), 7.17 (app.t, J = 7.7 Hz, 1H), 6.72 (d, J = 4.7 Hz, 1H), 4.27 (dd, J = 3.3, 10.0 Hz, 1H), 3.80 (m, 2H), 2.88 (ddd, J = 1.3, 3.6, 15.4 Hz, 1H), 2.58 (ddd, J = 2.8, 5.5, 15.4 Hz, 1H), 2.34 (s, 3H), 2.30 (s, 3H); ¹³C NMR (125 MHz, acetone-d₆) δ 140.5, 137.0, 128.5, 126.6, 125.1, 125.1, 124.5, 120.7, 119.9, 119.8, 119.5, 115.7, 112.1, 111.4, 108.9, 93.3, 80.6, 74.7, 74.6, 74.1, 55.5, 30.1; high resolution mass spectrum (EI) m/z 398.1630 [calcd for C_{25}H_{22}N_{2}O₃ (M^+) 398.1630].
X-RAY CRYSTALLOGRAPHY REPORT FOR INDOLOCARBAZOLE (±)-30

EXPERIMENTAL DETAILS

A. Crystal Data
Empirical Formula .................................. C_{38.5}H_{26}N_{2.0}O_{1.5}Br_{2}
Formula Weight ..................................... 780.45
Crystal Color/Habit .................................. colorless needle
Crystal Dimensions (mm) ......................... 0.08 X 0.11 X 0.30
Crystal System ...................................... monoclinic
No. Reflections Used for Unit ................. 25(10.2-18.0°)
Lattice Parameters:

a .................................................. 30.141 (5) Å
b .................................................. 15.689 (2) Å
c .................................................. 14.803 (3) Å
β .................................................. 91.45 (2)°
V .................................................. 6998 (3) Å³
Space Group ...................................... C2/c (#15)
Z value ............................................. 8
Dcalc ............................................. 1.481 g/cm³
F000 .............................................. 3144
μ(MoKα) .......................................... 23.41 cm⁻¹

B. Intensity Measurements
Diffractometer .................................... Enraf-Nonius CAD-4
Radiation .......................................... MoKα (λ = 0.71069 Å)
Temperature ...................................... 23°C
Attenuator ........................................... Zr foil (factor = 20.4)
Take-off Angle ................................... 2.8°
Detector Aperture ................................ 2.0-2.5 mm hor/2.0 mm vert.
Crystal to Detector Distance ................... 21 cm
Scan Type .......................................... ω-2θ
Scan Rate .......................................... 1.0 - 16.5°/min (in omega)
Scan Width.................................................(0.95 + 0.83 tanθ)°
2θmax..........................................................52.6°
No. of Reflections Measured
   Total:..................................................7660
   Unique:...............................................7369 (Rint = .041)
Corrections..............................................Lorentz-polarization
                                         Absorption
                                         (trans. factors: 0.72 - 1.51)
                                         Decay (-27.00% decline)

C. Structure Solution and Refinement
Structure Solution.....................................Direct Methods
Refinement..............................................Full-matrix least-squares
Function Minimized...................................Σ w ( |Fo| - |Fc| )²
Least-squares Weights.................................4Fo²/σ²(Fo²)
p-factor...................................................0.03
Anomalous Dispersion...................................All non-hydrogen atoms
No. Observations (I>3.00σ(I)).............................2877
No. Variables............................................436
Reflection/Parameter Ratio.............................6.60
Residuals..................................................R; Rw 0.077; 0.080
Goodness of Fit Indicator............................4.61
Max Shift/Error in Final Cycle.........................0.00
Maximum Peak in Final Diff. Map......................1.21 e-/Å³
Minimum Peak in Final Diff. Map......................-1.23 e-/Å³

Positional parameters and B(eq) for indolocarbazole (±)-30

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