

Supporting Information © Wiley-VCH 2007

● Wilcy-VOI1 2007

69451 Weinheim, Germany

One-Pot Multicomponent Synthesis of Indoles from 2-Iodobenzoic Acid

Olivier Leogane, and Hélène Lebel*

General information: Unless otherwise noted, all non-aqueous reactions were performed under an oxygen-free atmosphere of argon with rigid exclusion of moisture from reagents and glassware. The solvents were dried using standard methods prior to use. NaN3 and sodium tert-butoxide are commercially available and were purchased from Aldrich. Sodium tert-butoxide was handled under glove-box atmosphere. 2-iodo benzoic acid, alkynes, aldehydes, ketones and amines are commercially available and were purified using standard methods prior to use. Analytical thin layer chromatography (TLC) was performed using EM Reagent 0.25 mm silica gel 60-F plates. Visualization of the developed chromatogram was performed by UV absorbance, aqueous cerium molybdate, ethanolic phosphomolybdic acid, or aqueous potassium permanganate. Flash chromatography was performed using EM Silica Gel 60 (230-400 mesh) with the indicated solvent system. Optical rotations were measured on a Perkin-Elmer 341 digital polarimeter at 589 nm. Data are reported as follows: $[\alpha]_1^{\text{temp.}}$, concentration (c g/100mL), and solvent. ¹H NMR spectra were recorded in CDCl₃, unless otherwise noted, on a Bruker AV-400, a Bruker ARX-400, a Bruker AMX-300 or a Bruker AV-300 spectrometer (400, 400, 300 and 300 MHz respectively). Chemical shifts are reported in ppm on the δ scale from an internal standard of residual chloroform (7.27 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, q = quintet, m = multiplet and br = broad). coupling constant in Hz, integration. ¹³C NMR spectra were recorded in CDCl₃, unless otherwise noted, on a Bruker AV-400, a Bruker ARX-400, a Bruker AMX-300 or a Bruker AV-300 spectrometer (100, 100, 75 and 75 MHz respectively) with complete proton decoupling. Chemical shifts are reported in ppm from the central peak of CDCl₃ (77.0 ppm) on the δ scale. 2DCOSY experiments were realized for complex structures. Mass spectra were obtained on a LC-MSD TOF (ESI) Agilent Technologies high resolution from the Centre régional de spectrométrie de masse de l'Université de Montréal. Analytical gas chromatography with a mass spectroscopy (GC-MS) was carried out on a Hewlett Packard 6890 series gas chromatograph equipped with a split mode capillary injector and electron impact mass detector. Unless otherwise noted, injector and detector temperatures were 250 °C and the carrier gas was hydrogen (2mL/min) with a HP-5MS column. Data are reported as follows: column type, oven temperature, and retention time (t_r) .

Table S1. Palladium-Catalyzed Indolization of Carbamate 1.

Entry	Na ₂ CO ₃ (equiv.)	LiCl (equiv.)	Alcyne (equiv.)	time (hours)	1	Conv. (%) ^a	3
1	-	-	1.5	16	100	0	0
2	-	1	1.5	16	100	0	0
3	1.5	1	1.5	3	17	49	2
4	1.5	1	1.5	3	18	57	10
5	3	1	3	3	5	27	52
6	1.1	1	3	3	10	62 (48%)	^b 17
7	3	1	3	16	-	-	85% ^b
8	3	10	3	16	-	-	58% ^b
9	3	1 + NaC (1 equiv		16	-	-	67% ^b

^aDetermined by GC-MS. ^bIn parentheses, isolated yields.

Synthesis and caracterization of CBz-protected Indole 2.

Benzyl 2,3-di-*n***-propylindole-1-carboxylate (2).** To a solution of Pd(OAc)₂ (7.9 mg, 0.035 mmol), LiCl (29.7 mg, 0.700 mmol), Na₂CO₃ (81.6 mg, 0.770 mmol) and benzyl 2-iodophenylcarbamate (1) (247 mg, 0.700 mmol), in DMF (3.5 mL) at 25 °C, was added 4-octyne (0.308 mL, 2.10 mmol). The resulting mixture was then stirred at 120 °C for 3 hours. The reaction mixture was cooled to room temperature and filtered on a short pad of celite and eluted with EtOAc (70 mL). The resulting organic layer was washed with saturated NH₄Cl (2 x 30 mL), and brine (30 mL). The organic layer was dried over Na₂SO₄ and the solvent was removed under reduced pressure. The desired CBz-protected indole **2** (107 mg, 48%) was obtained as a pale yellow oil after flash chromatography (5% Et₂O/pentane). R_f 0.70 (10% Et₂O/pentane); ¹H NMR (400 MHz, CDCl₃) δ 8.13-8.09 (m, 1H), 7.52-7.49 (m, 2H), 7.47-7.37 (m, 4H), 7.24-7.20 (m, 2H), 5.46 (s, 2H), 2.95-2.91 (m, 2H), 2.65-2.61 (m, 2H), 1.68-1.52 (m, 4H), 0.99 (t, *J* = 7 Hz, 3H), 0.88 (t, *J* = 7 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 151.8, 137.2, 135.8, 135.0, 130.4, 128.74, 128.71, 128.65, 123.5, 122.6, 119.5, 118.2, 115.7, 68.5, 28.2, 26.1, 23.5, 23.4, 14.2, 14.0; IR (neat) 3034, 2957, 2930, 1728, 1457, 1392, 1320, 1222, 1139, 1107, 745 cm⁻¹; HMRS (ESI) calcd for C₂₂H₂₅NO₂Na [M+Na]⁺: 358.1777. Found: 358.1771.

General procedure for the synthesis of indoles through a one-pot Curtius rearrangement / Pd-catalyzed indolization with internal alkynes.

To a solution of sodium azide (0.110 g, 1.70 mmol), sodium *tert*-butoxide (14.4 g, 0.150 mmol) and 2-iodobenzoic acid (0.248 g, 1.00 mmol) in DMF (5.0 mL) at 25 °C, was added benzyl chloroformate (150 μ L, 1.10 mmol). The resulting mixture was then stirred at 75 °C. After 5 hours of reaction, the reaction mixture was allowed to cool at room temperature, then Pd(OAc)₂ (11.2 mg, 0.050 mmol), Na₂CO₃ (0.318 g, 3.00 mmol), and the alkyne (3 mmol) were introduced and the mixture was heated at 120 °C for 16 hours. The reaction mixture was cooled to room temperature and filtered on a short pad of celite which was washed with EtOAc (100 mL). The resulting organic layer was washed with saturated NH₄Cl (2 x 40 mL), and brine (40 mL). The organic layer was dried over Na₂SO₄ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel with a pre-absorption on silica.

Characterization of Indoles 3 - 6

$$CO_2H$$
 Pr

2,3-Di-*n***-propylindole** (3).¹ The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **3** (169 mg, 84%) was obtained as a pale yellow oil after flash chromatography (10% Et₂O/pentane). R_f 0.39 (10% Et₂O/pentane); ¹H NMR (400 MHz, CDCl₃) δ 7.72 (s (br), 1H), 7.56 (d, J = 8 Hz, 1H), 7.31-7.28 (m, 1H), 7.16-7.08 (m, 2H), 2.76-2.69 (m, 4H), 1.77-1.66 (m, 4H), 1.04-0.98 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 135.3, 135.1, 128.8, 120.7, 118.8, 118.4, 112.1, 110.4, 28.1, 26.2, 24.1, 23.1, 14.2, 14.0; IR 3406, 2956, 2929, 2869, 1461, 1244, 740 cm⁻¹; HMRS (ESI) calcd for $C_{14}H_{20}N$ [M+H] $^+$: 202.1590. Found: 202.1585.

$$CO_2H$$
 Ph

2,3-Diphenylindole (**4**). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **4** (207 mg, 77%) was obtained as a white solid after flash chromatography (15% EtOAc/pentane). R_f 0.21 (10% Et₂O/pentane); mp 109 °C (Lit. mp 108-110 °C);² ¹H NMR (400 MHz, CDCl₃) δ 8.23 (s (br), 1H), 7.70 (d, J = 8 Hz, 1H), 7.46-7.24 (m, 12H), 7.17 (t, J = 8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 135.8, 135.0, 134.0, 132.6, 130.1, 128.6, 128.5, 128.1, 127.7, 126.2, 122.7, 120.4, 119.7, 115.0, 110.9; IR 3410, 3056, 1600, 1454, 1243, 1070, 959, 738, 696 cm⁻¹; HMRS (ESI) calcd for $C_{20}H_{16}N$ [M+H]⁺: 270.1277. Found: 270.1272.

² Lu, B. Z.; Zhao, W. Y.; Wei, H. X.; Dufour, M.; Farina, V.; Senanayake, C. H. Org. Lett. **2006**, 8, 3271-3274.

¹ Larock, R. C.; Yum, E. K.; Refvik, M. D. J. Org. Chem. 1998, 63, 7652-7662.

2-tert-Butyl-3-methylindole (**5**). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **5** (105 mg, 56%) was obtained as a colorless oil after flash chromatography (5% Et₂O/pentane). R_f 0.42 (10% Et₂O/pentane); ¹H NMR (400 MHz, CDCl₃) δ 7.86 (s (br), 1H), 7.53 (d, J = 8 Hz, 1H), 7.31 (d, J = 8 Hz, 1H), 7.16-7.09 (m, 2H), 2.43 (s, 3H), 1.49 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 141.6, 133.6, 130.4, 120.9, 118.9, 117.7, 110.1, 105.2, 32.7, 29.9, 10.2; IR 3438, 3055, 2956, 2866, 1460, 1299, 1246, 1007, 737 cm⁻¹; HMRS (ESI) calcd for $C_{13}H_{18}N$ [M+H]⁺: 188.1434. Found: 188.1428.

2-(Trimethylsilyl)-3-phenylindole (6). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **6** (218 mg, 82%) was obtained as an orange solid after flash chromatography (10% EtOAc/pentane). R_f 0.45 (10% Et₂O/pentane); mp 103 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.15 (s (br), 1H), 7.57 (d, J = 8 Hz, 1H), 7.49-7.41 (m, 5H), 7.37-7.33 (m, 1H), 7.23 (t, J = 8 Hz, 1H), 7.10 (t, J = 8 Hz, 1H), 0.23 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 137.8, 136.5, 134.0, 130.3 (2C), 128.0 (2C), 126.5, 122.6, 119.8, 119.5, 110.7, -0.4; IR 3412, 3054, 2956, 2894, 1604, 1480, 1245, 1152, 833, 749, 699 cm⁻¹; HMRS (ESI) calcd for $C_{17}H_{20}NSi$ [M+H]⁺: 266.1359. Found: 266.1363.

General procedure for the synthesis of indoles through a one-pot Curtius rearrangement / Pd-catalyzed indolization with carbonyl compounds.

To a solution of sodium azide (0.110 g, 1.70 mmol), sodium *tert*-butoxide (14.4 mg, 0.150 mmol) and 2-iodobenzoic acid (0.248 g, 1.00 mmol) in DMF (5.0 mL) at 25 °C, was added benzyl chloroformate (150 µL, 1.10 mmol). The resulting mixture was then stirred at 75 °C. After 5 hours of reaction, the reaction mixture was allowed to cool at room temperature, then Pd(OAc)₂ (11.2 mg, 0.050 mmol for ketone and 10.1 mg, 0.045 mmol for aldehyde), DABCO (0.318 g, 3.00 mmol), and the ketone (3 mmol) or the aldehyde (0.90 mmol) were introduced, and the mixture was then heated at 120 °C for 16 hours. The reaction mixture was cooled to room temperature and filtered on a short pad of celite and eluted with EtOAc (100 mL). The resulting organic solution was washed with saturated NH₄Cl (2 x 40 mL), and brine (40 mL). The organic layer was dried over Na₂SO₄ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel with a pre-absorption on silica.

Characterization of Indoles 7 - 9

3-Benzylindole (7).³ The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **7** (100 mg, 50%) was obtained as a pale yellow solid after flash chromatography (15% EtOAc/pentane). R_f 0.18 (10% Et₂O/pentane); mp 105 °C; 1 H NMR (400 MHz, CDCl₃) δ 7.95 (s (br), 1H), 7.53 (d, J = 8 Hz, 1H), 7.37 (d, J = 8 Hz, 1H), 7.31-7.28 (m, 4H), 7.21-7.24 (m, 2H), 7.09 (t, J = 8 Hz, 1H), 6.93-6.92 (m, 1H), 4.13 (s, 2H); 13 C NMR (100 MHz, CDCl₃) δ 141.2, 128.6, 128.3 (2C), 127.4, 125.8, 122.2, 122.0, 119.3, 119.1, 115.8, 111.0, 31.6; IR 3396, 3057, 2909, 2842, 1600, 1456, 1339, 1222, 1087, 1009, 740 cm⁻¹; HMRS (ESI) calcd for $C_{15}H_{14}N$ [M+H] $^+$: 208.1121. Found: 208.1117.

$$CO_2H$$
 OBn

3-(2-(benzyloxy)ethyl)-indole (8). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **8** (120 mg, 53%) was obtained as an orange oil after flash chromatography (15% EtOAc/pentane). R_f 0.19 (10% Et₂O/pentane); 1 H NMR (400 MHz, CDCl₃) δ 7.95 (s (br), 1H), 7.61 (d, J = 8 Hz, 1H), 7.37-7.28 (m, 6H), 7.19 (t, J = 8 Hz, 1H), 7.11 (t, J = 8 Hz, 1H), 7.06 (s (br), 1H), 4.57 (s, 2H), 3.79 (t, J = 7 Hz, 2H), 3.10 (t, J = 7 Hz, 2H); 13 C NMR (100 MHz, CDCl₃) δ 138.4, 136.0, 128.3 (2C), 127.6, 127.50, 127.46, 122.0, 121.8, 119.1, 118.7, 112.8, 111.0, 72.9, 70.5, 25.7; IR 3339, 2981, 1446, 1378, 1122, 1079, 946, 630 cm⁻¹; HMRS (ESI) calcd for $C_{17}H_{17}NO$ [M+Na] $^+$: 274.1202. Found: 274.1202.

$$CO_2H$$
 O_2H

6,7,8,9-Tetrahydrocarbazole (**9**). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired carbazole **9** (96 mg, 56%) was obtained as a white solid after flash chromatography (10% EtOAc/pentane). R_f 0.26 (10% Et₂O/pentane); mp 117 °C (Lit. mp 117 °C);⁴ ¹H NMR (400 MHz, CDCl₃) δ 7.66 (s (br), 1H), 7.46 (d, J = 7 Hz, 1H), 7.29-7.27 (m, 1H), 7.13-7.05 (m, 2H), 2.75-2.70 (m, 4H), 1.95-1.84 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 135.6, 134.0, 127.8, 120.9, 119.1, 117.7, 110.3 (2C), 23.3, 23.22, 23.20, 20.9; IR 3393, 2927, 2852, 1469, 1450, 1234, 1143, 1009, 736 cm⁻¹; HMRS (ESI) calcd for C₁₂H₁₄N [M+H]⁺: 172.1120. Found 172.1119.

³ Jia, Y.; Zhu, J. J. Org. Chem. **2006**, 71, 7826-7834.

⁴ Adam, G.; Andrieux, J.; Plat, M. *Tetrahedron*, **1985**, *41*, 399-407.

General procedure for the synthesis of indoles through a one-pot Curtius rearrangement / urea formation / Pd-catalyzed indolization with internal alkynes.

To a solution of sodium azide (0.110 g, 1.70 mmol), sodium *tert*-butoxide (14.4 mg, 0.150 mmol) and 2-iodobenzoic acid (0.248 g, 1.00 mmol) in NMP (5.0 mL) at 25 °C, was added phenyl chloroformate (140 µL, 1.10 mmol). The resulting mixture was then stirred at 75 °C. After 5 hours of reaction, the amine derivative was added (1.50 mmol), and the stirring maintained for another 3 hours at 75 °C. Finally, the reaction mixture was allowed to cool at room temperature, then Pd(OAc)₂ (11.2 mg, 0.050 mmol) and the alkyne (3 mmol) were introduced and the mixture was heated at 120 °C for 16 hours. The reaction mixture was cooled to room temperature and filtered on a short pad of celite which was washed with EtOAc (100 mL). The resulting organic layer was washed with aqueous KOH (0.5 M, 2 x 40 mL), saturated NH₄Cl (40 mL), and brine (40 mL). The organic layer was dried over Na₂SO₄ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel with a pre-absorption on silica.

Characterization of Indole N-Carboxamide derivatives

Morpholino(2,3-diphenyl-indol-1-yl)methanone (10). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **10** (245 mg, 64%) was obtained as a white solid after flash chromatography (3:1, Et₂O/pentane). R_f 0.12 (3:1, Et₂O/pentane); mp 160°C; 1 H NMR (400 MHz, DMSO, 70 °C) δ 7.58 (d, J = 8 Hz, 1H), 7.52 (d, J = 8 Hz, 1H), 7.40-7.29 (m, 11H), 7.23 (t, J = 8 Hz, 1H), 3.59-3.51 (m, 2H), 3.38-3.32 (m, 2H), 3.27-3.16 (m, 4H); 13 C NMR (100 MHz, DMSO, 70 °C) δ 151.3, 134.9, 134.5, 133.1, 130.4, 129.3, 129.2, 127.9, 127.84, 127.79, 127.2, 126.1, 123.2, 121.3, 118.9, 116.8, 110.8, 65.1, 44.9; IR (neat) 3041, 2969, 2862, 1682, 1441, 1415, 1206, 1107, 756, 738, 697 cm⁻¹; HMRS (ESI) calcd for $C_{25}H_{23}N_2O_2$ [M+H]⁺: 383.1777. Found 383.1759.

(2,3-diphenyl-indol-1-yl)(piperidin-1-yl)methanone (11). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole 11 (259 mg, 68%) was obtained as a white solid after flash chromatography (3:1, Et₂O/pentane). R_f 0.31 (3:1, Et₂O/pentane); mp 145 °C; ¹H NMR (400 MHz, DMSO, 70 °C) δ 7.58 (d, J = 8 Hz, 1H), 7.45 (d, J = 8 Hz, 1H), 7.38-7.27 (m, 11H), 7.21 (t, J = 8 Hz, 1H), 3.33-3.23 (m, 4H), 1.55-1.46 (m, 4H), 1.26-1.19 (m, 2H); ¹³C NMR (100 MHz, DMSO, 70 °C) δ 151.0, 134.9, 134.6, 133.2, 130.4, 129.3, 129.2, 127.9, 127.7, 127.6, 127.0, 126.0, 123.0, 121.0, 118.8, 116.4, 110.6, 45.3, 24.7, 22.9; IR (neat) 2938, 2923, 1677, 1445, 1426, 1237, 1146, 743, 696 cm⁻¹; HMRS (ESI) calcd for $C_{26}H_{25}N_2O$ [M+H]⁺: 381.1950. Found 381.1961.

(Piperidin-1-yl)(2,3-dipropyl-1H-indol-1-yl)methanone (12). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole **12** (169 mg, 54%) was obtained as a pale orange oil after flash chromatography (20% Et₂O/pentane). R_f 0.20 (10% Et₂O/pentane); ¹H NMR (400 MHz, DMSO, 70 °C) δ 7.50 (d, J = 8 Hz, 1H), 7.25-7.21 (d, J = 8 Hz, 1H), 7.15 (t, J = 8 Hz, 1H), 7.08 (t, J = 8 Hz, 1H), 3.44-3.31 (m, 4H), 2.77 (t, J = 7 Hz, 2H), 2.66 (t, J = 7 Hz, 2H), 1.68-1.49 (m, 10H), 0.97-0.89 (m, 6H); ¹³C NMR (100 MHz, DMSO, 70 °C) δ 151.9, 135.5, 134.2, 127.8, 121.4, 119.7, 117.9, 114.3, 109.9, 45.6, 25.7, 25.1, 25.0, 23.1, 22.7, 21.9, 13.0; IR (neat) 2932, 2866, 1674, 1455, 1422, 1230, 1017, 740 cm⁻¹; HMRS (ESI) calcd for C₂₀H₂₈N₂ONa [M+Na]⁺: 335.2094. Found 335.2086.

(2,3-diphenyl-1H-indol-1-yl)(pyrrolidin-1-yl)methanone (13). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole 13 (227 mg, 62%) was obtained as a white solid after flash chromatography (3:1, Et₂O/pentane). R_f 0.17 (3:1, Et₂O/pentane); mp 109 °C; ¹H NMR (400 MHz, DMSO, 70 °C) δ 7.57 (d, J = 8 Hz, 1H), 7.46 (d, J = 8 Hz, 1H), 7.38-7.26 (m, 11H), 7.21 (t, J = 8 Hz, 1H), 3.29-3.21 (m (br), 4H), 1.79-1.66 (m (br), 4H); ¹³C NMR (100 MHz, DMSO, 70 °C) δ 150.3, 134.4, 134.1, 133.3, 130.6, 129.3, 128.9, 127.9, 127.7, 127.5, 127.1, 126.0, 123.0, 120.9, 118.8, 116.4, 110.6, 46.3, 24.0; IR (neat) 2974, 2879, 1699, 1678, 1399, 1238, 740, 700 cm⁻¹; HMRS (ESI) calcd for C₂₅H₂₃N₂O [M+H]⁺: 367.1805. Found 367.1804.

N-((*R*)-1-Phenylethyl)-2,3-dipropylindole-1-carboxamide (14). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole 14 (206 mg, 59%) was obtained as a white solid after flash chromatography (10% Et₂O/pentane). R_f 0.27 (10% Et₂O/pentane); mp 119 °C; $[\alpha]_D^{25} = -1.8$ (c 1.0, MeOH); ¹H NMR (400 MHz, DMSO) δ 8.97 (d, J = 8 Hz, 1H), 7.49-7.44 (m, 4H), 7.36 (t, J = 7 Hz, 3H), 7.28-7.25 (m, 1H), 7.15-7.06 (m, 2H), 5.08-5.00 (m, 1H), 2.83-2.74 (m, 2H), 2.62 (t, J = 7 Hz, 2H), 1.60-1.55 (m, 2H), 1.52 (d, J = 7 Hz, 3H), 1.39-1.34 (m, 2H), 0.92 (t, J = 7 Hz, 3H), 0.76 (t, J = 7 Hz, 3H); ¹³C NMR (100 MHz, DMSO) δ 151.3, 144.0, 135.9, 134.9, 128.2 (2C), 126.8, 126.1, 121.7 120.3, 118.1, 114.6, 111.6, 50.3, 26.4, 25.4,

23.4, 22.6, 22.1, 13.9, 13.6; IR 3288, 3033, 2958, 2931, 1662, 1519, 1457, 1320, 1205, 731 cm $^{-1}$; HMRS (ESI) calcd for $C_{23}H_{28}N_2ONa$ [M+Na] $^{+}$: 371.2094. Found 371.2087.

N-Phenethyl-2,3-dipropylindole-1-carboxamide (15). The title compound was prepared from 2-iodobenzoic acid (248 mg, 1.00 mmol) according to the general procedure. The desired indole 15 (136 mg, 39%) was obtained as a white solid after flash chromatography (10% Et₂O/pentane). R_f 0.15 (10% Et₂O/pentane); mp 74 °C; ¹H NMR (400 MHz, DMSO) δ 8.46 (s (br), 1H), 7.45 (d, J = 7 Hz, 1H), 7.36-7.31 (m, 4H), 7.26-7.23 (m, 2H), 7.08-7.01 (m, 2H), 3.61-3.56 (m, 2H), 2.93 (t, J = 7 Hz, 2H), 2.80 (t, J = 8 Hz, 2H), 2.61 (t, J = 7 Hz, 2H), 1.60-1.50 (m, 2H), 1.49-1.40 (m, 2H), 0.91 (t, J = 7 Hz, 3H), 0.84 (t, J = 7 Hz, 3H); ¹³C NMR (100 MHz, DMSO) δ 152.0, 139.0, 135.9, 134.6, 128.7, 128.3 (2C), 126.1, 121.5, 120.2, 118.0, 114.7, 111.8, 41.6, 34.4, 26.4, 25.4, 23.4, 22.7, 13.9, 13.7; IR 3288, 3029, 2958, 2930, 1667, 1533, 1456, 1321, 1210, 1026, 730 cm⁻¹; HMRS (ESI) calcd for C₂₃H₂₉N₂O [M+H]⁺: 349.2274. Found 349.2264.