Synthesis of Pyridazine and Pyrrole Analogues of 2-Aminotetralin as Potential Dopaminergics

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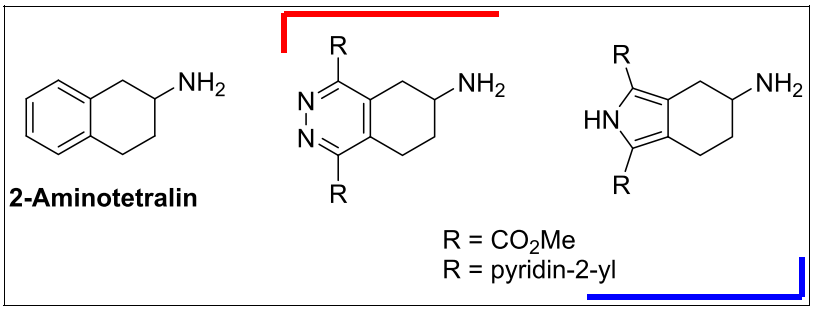

Syntheses of pyridazine and pyrrole analogues of 2-aminotetralin starting from 3-cyclohexene-1-carboxylic acid are reported. All syntheses involve the following key steps: Curtius rearrangement for amine functionality, inverse electron demand Diels–Alder addition with 1,2,4,5-tetrazine for pyridazine ring synthesis, and pyridazine-to-pyrrole ring contraction for pyrrole ring formation.

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INTRODUCTION

Mono- and dihydroxy-2-aminotetralin (**1**) structures are rigid analogues of dopamine (**2**) and exhibit a wide range pharmacological activity such as dopaminergic, serotonergic, and adrenergic effects (Fig. 1) [1–3]. The position of the hydroxyl groups is critical in determining the receptor preference [4]. Compounds **3–5** are examples of heterocyclic isosteres of hydroxy-2-aminotetralins, which were shown to possess DA agonist activity (Fig. 1) [5–13]. This encouraged us to replace the phenolic portion of 2-aminotetralin with ester and pyridine-substituted pyridazines and pyrroles may be the compounds with potential dopaminergic activities.

RESULTS AND DISCUSSIONS

We report here the fast, efficient preparation of pyridazine and pyrrole motifs of 2-aminotetralin directly starting from readily available compounds (Scheme 1). Carboxylic acid acts as the convenient precursor of an amine group. Our initial effort was to synthesize benzyl cyclohex-3-en-1-ylcarbamate (**7**). First, cyclohex-3-ene-1-carboxylic acid (**6**) was reacted with diphenylphosphoryl azide (DPPA) in the presence of triethylamine (NEt_3) at room temperature for 1 day in benzene. The initially formed acyl azide was rearranged with the loss of nitrogen to afford the isocyanate, which was trapped *in situ* with benzyl alcohol. Refluxing the mixture in benzene afforded the benzyl carbamate **7** in 76% yield through the Curtius rearrangement [14–17]. Inverse electron demand Diels–Alder additions (iEDDA) between 1,2,4,5-tetrazines and olefins are very well known for the preparation of pyridazines [18–27]. These reactions result in the formation of bicyclic intermediates via nitrogen elimination readily and subsequent rearranging, yielding dihydropyridazines, which may be further oxidized to pyridazines. A reaction of cyclohexene derivative **7** with dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (**8a**) in dry methylene chloride led to the formation of the corresponding 1,4-dihydropyridazine, which was then converted to the desired pyridazine **9a** in 68% overall yield by oxidizing with phenyliodo-bis(trifluoroacetate) (PIFA). Cycloaddition of **7** with 3,6-di(2-pyridyl)-1,2,4,5-tetrazine (**8b**) progressed more slowly than its analogue **8a** and a higher reaction temperature was required to carry out the aimed transformation. When the reaction was performed under reflux conditions in acetonitrile (MeCN), the formation of 3,6-di(pyridin-2-yl)-1,4-dihydro-1,2,4,5-tetrazine (**10b**) along with **10a** was observed. Since dipyridyl-tetrazine **8b** behaves as an oxidizing agent at the same time, using two equivalents of **8b** with **7** for 3 days led to the formation of **9b** (92%) and **10b** (92%) [28,29]. In the next step, a carbobenzyloxy protecting group of *N*-Cbz-protected amines **9a** and **9b** was easily removed with palladium-catalyzed hydrogenations to give the corresponding free amines **11a** and **11b** in 62% and 70% yield, respectively. Both of the pyridazines **11a** and **11b** were also converted into the corresponding pyrroles **12a**

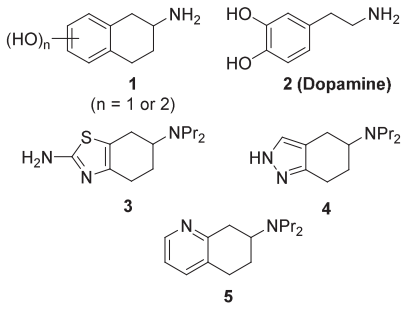


Figure 1. Structures of dihydroxy-2-aminotetralin (**1**), dopamine (**2**), and some heterocyclic isosteres **3–5**.

and **12b** via a ring contraction process with 20 equivalents of zinc dust in refluxing acetic acid according to the Boger procedure, which is a highly reliable synthetic approach [30–32]. Furthermore, an alternative pathway including pyridazine-to-pyrrole ring contraction and deprotection steps starting from pyridazine **9a**/**9b** afforded the desired products **12a**/**12b**.

CONCLUSION

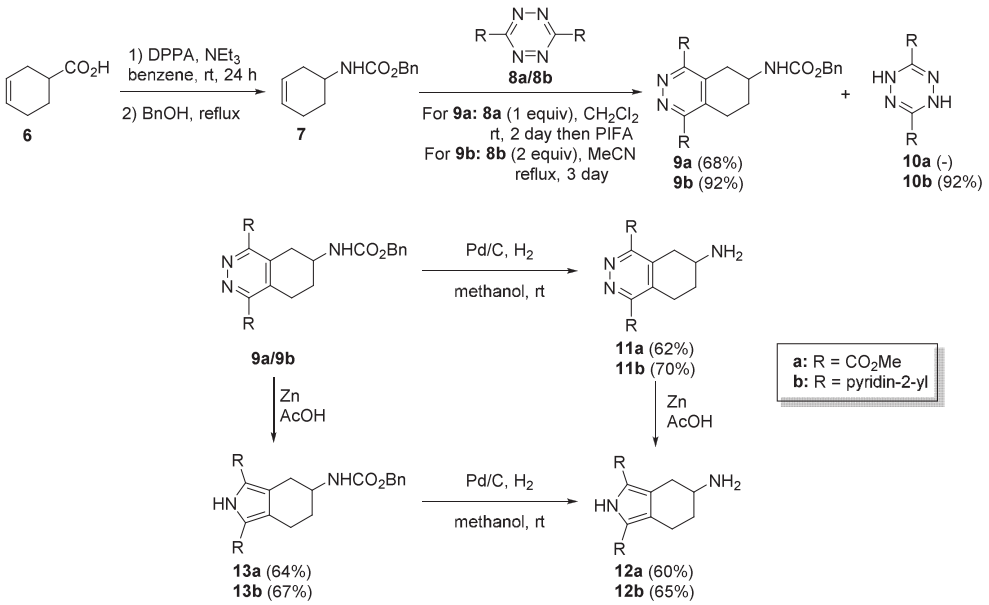
We present the fast, efficient preparation of pyridazine and pyrrole analogues of 2-aminotetralin. New heterocyclic analogues may be candidates for the development of dopaminergic agents. Further work is focusing on the enantioselective synthesis of these pyridazine and pyrrole analogues and evaluation of their biological activity.

EXPERIMENTAL SECTION

All reactions were carried out under nitrogen and monitored by TLC and/or ^{1}H-NMR spectroscopy. All solvents were dried and distilled before use. Column chromatography was performed on silica gel (60 mesh, Merck) or activated neutral aluminum oxide (Sigma Aldrich). TLC was carried out on silica gel 60 HF_{254} aluminum plates (Fluka). Melting points are uncorrected. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on 400 M Hz NMR spectrometers. Apparent splitting is given in all cases in ppm and coupling constants J in Hz. All new compounds gave satisfactory HRMS. All substances reported in this paper are in their racemic forms.

Benzyl cyclohex-3-en-1-ylcarbamate (7). 3-Cyclohexene-1-carboxylic acid (**6**) (1.0 g, 7.9 mmol) was added to a solution of benzene (90 mL), triethylamine (NEt_3) (963 mg, 9.5 mmol), and diphenylphosphoryl azide (DPPA) (2.62 g, 9.5 mmol). The mixture was vigorously stirred at room temperature for 1 day. Benzyl alcohol (13.34 g, 123.5 mmol) was added to the reaction medium. After refluxing for 2 days, the reaction mixture was cooled room temperature, and the solvent was evaporated under reduced pressure. The crude reaction mixture was purified by column chromatography on silica gel (EtOAc/*n*-hexane (1:1), then EtOAc). The obtained solid was recrystallized from CH_2Cl_2/*n*-hexane (9:1) to give **7**. Yield: 76%; white crystals; m.p. = 64–65°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.38–7.28 (m, 5H), 5.68 (bd, A part of AB system, J = 10.0 Hz, 1H), 5.59 (bd, B part of AB system,

Scheme 1. Synthesis of pyridazine **11** and pyrrole **12** as analogues of 2-aminotetralin.



J = 10.0 Hz, 1H), 5.10 (s, 2H), 4.81 (bs, 1H, NH), 3.92–3.80 (m, 1H), 2.40 (d, J = 17.2 Hz, 1H), 2.22–2.04 (m, 2H), 1.94–1.80 (m, 2H), 1.63–1.51 (m, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.9, 136.8, 128.6, 128.3, 128.2, 127.1, 124.4, 66.7, 46.3, 32.1, 28.4, 23.5. HRMS (Q-TOF): m/z $[M + H]^+$ calcd for $C_{14}H_{17}NO_2$: 232.1338, found: 232.1324.

Dimethyl 6-(((benzyloxy)carbonyl)amino)-5,6,7,8-tetrahydrophthalazine-1,4-dicarboxylate (9a). Benzyl cyclohex-3-en-1-ylcarbamate (**7**) (500 mg, 2.16 mmol) and dimethyl-1,2,4,5-tetrazine-3,6-dicarboxylate (**8a**) (471 mg, 2.38 mmol) were dissolved in 25 mL of CH_2Cl_2. The mixture was stirred at room temperature for 2 days. Then [bis(trifluoroacetoxy)iodo]benzene (PIFA) (1.12 g, 2.59 mmol) was added and the mixture stirred for an additional 6 h at the same temperature. At the end of the reaction, the solvent was evaporated under reduced pressure. The crude reaction mixture was purified by column chromatography on silica gel (*n*-hexane, then EtOAc). The obtained solid was recrystallized from CH_2Cl_2/*n*-hexane (9:1) to give **9a**. Yield: 68%; white crystals; m.p. = 121–122°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.39–7.29 (m, 5H), 5.10 (s, 2H), 4.97 (bd, J = 6.1 Hz 1H), 4.04–4.01 (m, 7H), 3.38 (dd, A part of AB system, J = 18.5 Hz, J = 5.0 Hz, 1H), 3.20 (dt, J = 19.3 Hz, J = 5.3 Hz, 1H), 3.14–3.03 (m, 1H), 2.90 (dd, B part of AB system, J = 18.5 Hz, J = 8.6 Hz, 1H), 2.22–2.11 (m, 1H), 1.85–1.71 (m, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 165.1, 165.0, 155.7, 152.9, 152.5, 137.5, 136.8, 136.3, 128.7, 128.3, 128.2, 67.0, 53.4, 53.35, 45.1, 32.4, 26.9, 24.3. HRMS (Q-TOF): m/z $[M + H]^+$ calcd for $C_{20}H_{21}N_3O_6$: 400.1509, found: 400.1506.

Reaction of benzyl cyclohex-3-en-1-ylcarbamate (7) with 3,6-di(2-pyridyl)-1,2,4,5-tetrazine (8b). Benzyl cyclohex-3-en-1-ylcarbamate (**7**) (500 mg, 2.16 mmol) and 3,6-di(2-pyridyl)-1,2,4,5-tetrazine (**8b**) (1.02 mg, 4.32 mmol) were dissolved in 25 mL CH_3CN. The reaction was warmed at reflux for 3 days and the solvent was evaporated under reduced pressure. The crude reaction mixture was purified by column chromatography on silica gel (EtOAc). **10b** was obtained in the first fraction and **9b** was obtained in the second fraction. Also, **9a** was recrystallized from CH_2Cl_2/*n*-hexane (9:1).

Benzyl (1,4-di(pyridin-2-yl)-5,6,7,8-tetrahydrophthalazin-6-yl)carbamate (9b). Yield: 92%; brown crystals; m.p. = 75–76°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.67 (dd, J = 9.1 Hz, J = 4.8 Hz, 2H), 7.99 (t, J = 8.4 Hz, 2H), 7.85 (t, J = 7.7 Hz, 2H), 7.38–7.27 (m, 7H), 5.08 (bd, J = 6.8 Hz 1H)), 5.05 (s, 2H), 4.09–3.93 (m, 1H), 3.32 (dd, A part of AB system, J = 18.3 Hz, J = 4.8 Hz, 1H), 3.25–3.15 (m, 2H), 3.08 (dd, B part of AB system, J = 18.3 Hz, J = 8.3 Hz, 1H), 2.14–2.03 (m, 1H), 1.81–1.65 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.2, 157.8, 156.3, 156.1, 155.7, 148.4, 137.0, 136.9, 136.4, 136.2, 135.1, 128.5 (2C), 128.1 (2C), 125.1, 125.0, 123.6, 123.6, 66.7, 45.7, 33.4, 27.6, 25.0. HRMS (Q-TOF): m/z $[M + H]^+$ calcd for $C_{26}H_{23}N_5O_2$: 438.1930, found: 438.1923.

3,6-Di(pyridin-2-yl)-1,4-dihydro-1,2,4,5-tetrazine (10b). Yield: 92%; orange solid; m.p. = 196–197 (lit[29] mp 191–193°C). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.59 (bs, 2H, NH) 8.57 (d, J = 4.9 Hz, 2H), 8.05 (d, J = 7.7 Hz, 2H), 7.75 (dt, J = 7.7 Hz, J = 1.8 Hz, 2H), 7.34 (ddd, J = 7.7 Hz, J = 4.9 Hz, J = 1.2 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 148.4, 147.5, 146.6, 136.7, 124.9, 121.3.

General procedure for hydrogenation of carbamat 9a and 9b. Carbamat **9** (1.0 mmol) was dissolved in MeOH (20 mL) and 10% Pd/C was added. The mixture was stirred under H_2 atmosphere for 20 h. The Pd/C was removed by filtration, and the solvent was evaporated. Chromatography (SiO_2, EtOAc/MeOH (20:1)) afforded **11** as solid. The obtained solid was crystallized from MeOH/diethyl ether (9:1).

Dimethyl 6-amino-5,6,7,8-tetrahydrophthalazine-1,4-dicarboxylate (11a). Yield: 62%; green crystals; m.p. = 144–145°C. ^{1}H-NMR (400 MHz, MeOD-d_4): δ = 5.00 (bs, 2H), 4.06 (s, 3H), 4.05 (s, 3H), 3.89–3.57 (m, 3H), 3.24–3.11 (m, 2H), 2.41–2.32 (m, 1H), 2.01–1.86 (m, 1H). ^{13}C-NMR (100 MHz, MeOD-d_4): δ = 165.9, 165.8, 153.8, 153.4, 139.6, 137.8, 53.9, 53.8, 46.7, 31.0, 26.0, 25.3. HRMS (Q-TOF): m/z $[M + H]^+$ calcd for $C_{12}H_{15}N_3O_4$: 266.1141, found: 266.1127.

1,4-Di(pyridin-2-yl)-5,6,7,8-tetrahydrophthalazin-6-amine (11b). Yield: 70%; green crystals; m.p. = 219–220°C. ^{1}H-NMR (400 MHz, MeOD-d_4): δ =8.74 (d, J = 4.9 Hz, 2H), 8.05 (t, J = 7.7 Hz, 2H), 7.88 (ddd, J = 7.8 Hz, J = 4.2 Hz, J = 0.7 Hz, 2H), 7.56 (dd, J = 7.5 Hz, J = 4.9 Hz, 2H), 4.87 (bs, 2H), 3.20–2.95 (m, 4H), 2.78 (dd, J = 17.9 Hz, J = 9.2 Hz, 1H), 2.10–2.00 (m, 1H), 1.67–1.54 (m, 1H). ^{13}C-NMR (100 MHz, MeOD-d_4): δ = 160.4, 160.0, 156.7, 156.67, 149.9, 149.87, 138.9, 138.8, 138.78, 138.3, 126.1 (2C), 125.4 (2C), 46.7, 36.4, 31.2, 26.6. HRMS (Q-TOF): m/z $[M + H]^+$ calcd for $C_{18}H_{17}N_5$: 304.1562, found: 304.1556.

General procedure of ring contraction of pyridazines to pyrroles. Zinc dust (25.04 mmol) was added to a solution of pyridazine **11** (1.25 mmol) in 10 mL of glacial acetic acid and the reaction was stirred at room temperature for overnight. The reaction mixture was filtered through Celite, the filtrate was neutralized with the addition of saturated aqueous $NaHCO_3$, and extracted with EtOAc (2 × 25). The combined organic layer was dried over Na_2SO_4 and evaporated under vacuo. The obtained solid was crystallized from MeOH/diethyl ether (9:1) to give **12.**

Dimethyl 5-amino-4,5,6,7-tetrahydro-2H-isoindole-1,3-dicarboxylate (12a). Yield: 60%; white crystals; m. p. = 264–265°C. ^{1}H-NMR (400 MHz, MeOD-d_4): δ = 4.92 (bs, 3H), 3.89 (s, 3H), 3.88 (s, 3H), 3.59–3.50 (m, 1H), 3.40 (dd, *J* = 16.6 Hz, *J* = 5.0 Hz, 1H), 3.11 (dt, *J* = 17.2 Hz, *J* = 4.8 Hz, 1H), 2.89–2.72 (m, 2H), 2.25–2.16 (m, 1H), 1.92–1.80 (m, 1H). ^{13}C-NMR (100 MHz, MeOD-d_4): δ = 162.5, 162.4, 127.1, 124.9, 122.5, 122.4, 52.1, 52.06, 48.9, 28.8, 28.2, 21.7. HRMS (Q-TOF): *m*/*z* [M + H]$^+$ calcd for $C_{12}H_{16}N_2O_4$: 253.1188, found: 253.1179.

1,3-Di(pyridin-2-yl)-4,5,6,7-tetrahydro-2H-isoindol-5-amine (12b). Yield: 65%; light green crystals; m.p. = 301–302°C. ^{1}H-NMR (400 MHz, MeOD-d_4): δ = 8.58 (d, *J* = 4.8 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 2H), 7.63 (dd, *J* = 8.0 Hz, *J* = 4.6 Hz, 2H), 7.21 (t, *J* = 5.7 Hz, 2H), 4.89 (bs, 3H), 3.67–3.58 (m, 1H), 3.47 (dd, *J* = 15.6 Hz, *J* = 5.0 Hz, 1H), 3.18 (dt, *J* = 16.5 Hz, *J* = 5.0 Hz, 1H), 3.10–2.99 (m, 1H), 2.95 (dd, *J* = 15.6 Hz, *J* = 9.3 Hz, 1H), 2.34–2.25 (m, 1H), 2.04–1.94 (m, 1H). ^{13}C-NMR (100 MHz, MeOD-d_4): δ = 151.7 (2C), 150.4, 150.3, 138.1(2C), 128.6, 128.4, 121.9, 121.86, 121.0, 120.8, 120.6, 118.7, 49.4, 30.3, 28.9, 22.8. HRMS (Q-TOF): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{18}N_4$: 291.1610, found: 291.1602.

Dimethyl 5-(((benzyloxy)carbonyl)amino)-4,5,6,7-tetrahydro-2H-isoindole-1,3-dicarboxylate (13a). Zinc dust (1.64 g, 25.04 mmol) was added to a solution of pyridazine **9a** (500 mg, 1.25 mmol) in 10 mL of glacial acetic acid, and the reaction was stirred at room temperature for overnight. The reaction mixture was filtered through Celite, the filtrate was neutralized with the addition of saturated aqueous $NaHCO_3$, and extracted with EtOAc (2 × 25). The combined organic layer was dried over Na_2SO_4 and evaporated under vacuo. Chromatography (SiO_2, EtOAc) afforded as a white solid. The obtained solid was recrystallized from MeOH/diethyl ether (1:1) to give **13a**. Yield: 64%; white crystals; m.p. = 200–201°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 9.56 (bs, 1H, NH), 7.38–7.28 (m, 5H), 5.10 (s, 2H), 4.90 (bd, *J* = 8.0 Hz 1H), 4.10–4.00 (m, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.18 (dd, A part of AB system, *J* = 16.9 Hz, *J* = 5.1 Hz, 1H), 2.93 (dt, A part of AB system, *J* = 17.7 Hz, *J* = 5.8 Hz, 1H), 2.83 (dt, B part of AB system, *J* = 17.7 Hz, *J* = 6.7 Hz, 1H), 2.66 (dd, B part of AB system, *J* = 16.9 Hz, *J* = 7.6 Hz, 1H), 2.08–1.94 (m, 1H), 1.84–1.71 (m, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 161.2, 161.18, 155.9, 136.6, 128.7, 128.2 (2C), 127.0, 125.8, 121.3, 120.9, 66.8, 51.83, 51.80, 46.6, 29.9, 28.4, 20.5. HRMS (Q-TOF): *m*/*z* [M + H]$^+$ calcd for $C_{20}H_{22}N_2O_6$: 387.1556, found: 387.1549.

Benzyl (1,3-di(pyridin-2-yl)-4,5,6,7-tetrahydro-2H-isoindol-5-yl)carbamate (13b). Zinc dust (1.64 g, 25.04 mmol) was added to a solution of pyridazine **9b** (500 mg, 1.25 mmol) in 10 mL of glacial acetic acid, and the reaction was stirred at room temperature for overnight. The reaction mixture was filtered through Celite, the filtrate was neutralized with the addition of saturated aqueous $NaHCO_3$, and extracted with EtOAc (2 × 25). The combined organic layer was dried over Na_2SO_4 and evaporated under vacuo. Chromatography (AlO_3, EtOAc) afforded as a green solid. The obtained solid was crystallized from MeOH/diethyl ether (9:1) to give **(13b)**. Yield: 67%; green crystals; m.p. = 186–188°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 10.54 (bs, 1H, NH), 8.56 (d, *J* = 4.5 Hz, 2H), 7.68–7.61 (m, 2H), 7.48–7.28 (m, 7H), 7.05 (dd, *J* = 7.2 Hz, *J* = 5.0 Hz, 2H), 5.11 (s, 2H), 4.99 (bd, *J* = 8.4 Hz, 1H), 4.27–4.16 (bs, 1H), 3.28 (dd, A part of AB system, *J* = 15.6 Hz, *J* = 5.2 Hz, 1H), 2.99 (t, *J* = 6.3 Hz, 2H), 2.81 (dd, B part of AB system, *J* = 15.6 Hz, *J* = 6.3 Hz, 1H), 2.15–1.90 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 156.0, 150.5, 150.4, 149.5, 136.7, 136.4, 128.6 (2C), 128.23, 128.20 (2C), 127.7, 127.2, 120.42, 120.35, 119.8, 119.3, 119.27, 118.7, 66.7, 46.9, 31.3, 28.7, 21.5. HRMS (Q-TOF): *m*/*z* [M + H]$^+$ calcd for $C_{26}H_{24}N_4O_2$: 425.1978, found: 425.1969.

Hydrogenation of carbamates 13a and 13b. Carbamat **13** (1.0 mmol) was dissolved in MeOH (20 mL) and 10% Pd/C was added. The mixture was stirred under H_2 atmosphere for 20 h. The Pd/C was removed by filtration, and the solvent was evaporated. Chromatography (SiO_2, EtOAc/MeOH (20:1)) afforded **12** as solid. The obtained solid was crystallized from MeOH/diethyl ether (9:1). Dimethyl 5-amino-4,5,6,7-tetrahydro-2*H*-isoindole-1,3-dicarboxylate (**12a**) was obtained in 63% yield. 1,3-Di(pyridin-2-yl)-4,5,6,7-tetrahydro-2*H*-isoindol-5-amine (**12b**) was obtained in 68% yield.

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SUPPORTING INFORMATION

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