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Synthesis of 3,3-Disubstituted Oxindoles by Visible-Light-Mediated Radical Reactions of Aryl Diazonium Salts with N-Arylacrylamides

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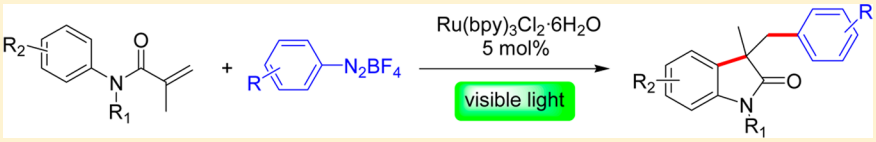
Supporting Information



ABSTRACT: A mild and efficient visible-light-mediated diarylation of *N*-arylacrylamides with aryl diazonium salts under mild conditions has been developed. This method provides convenient access to a variety of useful 3,3-disubstituted oxindoles by constructing two C−C bonds in one step.

The prevalence of the oxindole ring system that represents a key structural component in natural products and pharmaceutical chemistry is well-established.[1] Moreover, functionalized oxindoles have also found wide utility as versatile starting materials for the synthesis of a broad range of heterocyclic compounds. As such, the search for sustainable and more efficient methods for the preparation of oxindoles is of constant interest.[2] Among many different approaches to 3,3′-disubstituted oxindoles, palladium-catalyzed oxidative difunctionalization alkenes in N-arylacrylamides provides direct and potentially more efficient access to oxindoles,[3] particularly because additional functional groups can be introduced into the oxindole framework by a single transformation. These methods, however, have always presented some limitations, such as the use of equivalent oxidant and harsh reaction conditions. An alternative method is the radical-mediated cyclization of N-arylacrylamides, which is performed with or without the aid of transition-metal catalysts. Yang and co-workers reported an efficient carbon phosphorylation of N-arylacrylamides with silver catalysts under mild conditions.[4] The radical alkylarylation,[5] arylcarbonylation,[6] azidoarylation,[7] and arylsulfonylation[8] of N-arylacrylamides have since been disclosed by several groups, allowing the effective formation of oxindole framework. To the best of our knowledge, the similar protocol using aryl diazonium salts as aryl radicals is not well-documented.

Aryl radicals have been widely used to create carbon−carbon and carbon−heteroatom bonds in organic synthesis over the past several decades.[9] Because they are one of the most powerful synthetic tools, various methods for the generation of aryl radicals have been developed.[10] Among many different approaches to aryl radicals, the photoinduced reduction of aryl diazonium salts through electron transfer (PET) using [Ru(bpy)$_3$]Cl$_2$ or organic dyes are particularly attractive.[11] Recently, many examples for radical arylation by photoredox catalysis have been reported.[12] König and co-workers developed an efficient visible-light-mediated arylation of alkenes, alkynes, enones, enol acetates, and heteroarenes using diazonium salts by photoredox catalysis.[13] Zhou and co-workers reported a metal-free, visible-light-induced [4 + 2] benzannulation of biaryldiazonium salts with alkynes to form phenanthrenes with eosin Y as the photoredox catalyst.[14] Inspired by these pioneering works and in connection with radical-mediated cyclizations of N-arylacrylamides, we herein report an efficient procedure for the preparation of a series of 3,3-disubstituted oxoindoles via visible-light-promoted diarylation of N-arylacrylamides with aryl diazonium salts (Scheme 1).

At first, we examined the reaction of N-arylacrylamide **1a** with diazonium salt **2a** under various conditions, and the results are summarized in Table 1. When the reaction of **1a** with **2a** was carried out in the presence of Ru(bpy)$_3$Cl$_2$·6H$_2$O (5 mol %) under N$_2$ in MeCN for 12 h, the yield of **3a** was only 46% (Table 1, entry 1). When the solvent was changed to MeOH, the yield improved to 65%, whereas other solvents such as DMF and THF were found to be less effective for this reaction (Table 1, entries 2−4). Increasing the amount of diazonium salt **2a** led to an improvement in the yield (Table 1, entries 5 and 6). Using 2.5 equiv of **2a** gave a good yield, but when the amount of **2a** was increased to 4.0 equiv, the yield of **3a** did not increase further (Table 1, entry 7). Under the same reaction conditions, employment of the catalyst Ru(bpy)$_3$(PF$_6$)$_2$ afforded a similar result. In contrast, photocatalytic active organic dye eosin Y resulted in low yield. As expected, no reaction occurred in the absence of photocatalyst (entry 10). When the reaction was carried out under ambient light, the reaction was much more sluggish and had a dramatically decreased yield (entry 11). The molecular structure of **3a** was confirmed by its X-ray single-crystal diffraction (see the Supporting Information).

Under the optimized conditions, N-arylacrylamide **1a** was reacted with various aryl diazonium salts **2a**−**h** (2.5 equiv) to give

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Scheme 1. Photocatalytic Radical-Mediated Cyclizations of N-Arylacrylamides

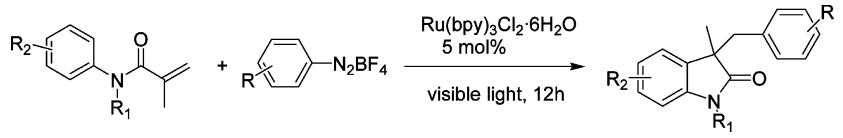
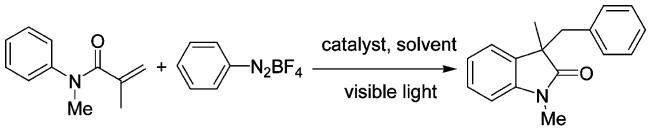


Table 1. Optimization of Reaction Conditions for 3a[a]

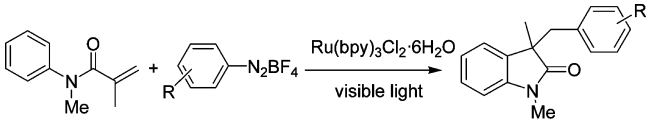


1a 2a 3a

entry	catalyst (mol %)	ratio (**1a**:**2a**)	solvent	yield (%)[b]
1	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:1.5	MeCN	46
2	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:1.5	DMF	51
3	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:1.5	THF	45
4	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:1.5	MeOH	65
5	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:2	MeOH	71
6	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:2.5	MeOH	80
7	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:4	MeOH	81
8	$Ru(bpy)_3(PF_6)_2$ (5)	1:2.5	MeOH	80
9	Eosin Y (5)	1:2.5	MeOH	52
10[c]		1:2.5	MeOH	
11[d]	$Ru(bpy)_3Cl_2·6H_2O$ (5)	1:2.5	MeOH	36

[a]Reaction conditions: **1a** (0.3 mmol), catalyst (5 mol %), and solvent (3.0 mL) under visible-light irradiation for 12 h. [b]Isolated yield. [c]Reaction was carried out without catalyst. [d]Reaction was carried out under ambient light.

Table 2. Scope of Aryl Diazonium Salts[a]



1 2a-h 3a-h

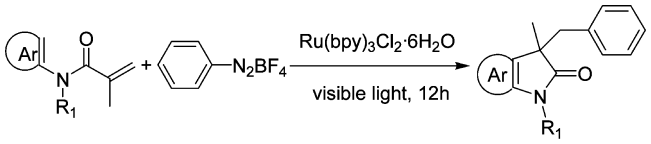
entry	R	product	yield (%)
1	H	**3a**	80
2	4-F	**3b**	82
3	4-Br	**3c**	76
4	4-Me	**3d**	63
5	2-Me	**3e**	58
6	2-F	**3f**	71
7	2-Cl	**3g**	67
8	2-Br	**3h**	60

[a]Reaction conditions: **1** (0.3 mmol), $Ru(bpy)_3Cl_2·6H_2O$ (5 mol %), and aryl diazonium salts **2** (0.75 mmol) in MeOH (3.0 mL) under visible-light irradiation for 12 h.

the desired oxoindoles **3a**–**h** in moderate to good yields (Table 2). Aryl diazonium salts possessing electron-withdrawing groups gave the desired products in higher yields than the substrates with electron-donating groups. Moreover, the procedure seemed to be sensitive to steric effects. Generally, substitutents in the para position on the aryl group proceeded well (entries 2–4). In comparison, ortho substituents on the aryl group reduced the yields (entries 5–8).

To establish the scope of this developed radical-mediated cyclization protocol, we subsequently tested a wide range of N-arylacrylamides (Table 3). It was found that various N-protected

Table 3. Scope of N-Arylacrylamides[a]



1a-s 2a 4a-s

entry	R_1	Ar	product	yield (%)
1	Et	Ph	**4a**	72
2	*i*-Pr	Ph	**4b**	67
3	*n*-Bu	Ph	**4c**	70
4	Bn	Ph	**4d**	63
5	Ac	Ph	**4e**	0
6	H	Ph	**4f**	0
7	Me	4-Me-Ph	**4g**	70
8	Me	4-MeO-Ph	**4h**	75
9	Me	4-Cl-Ph	**4i**	63
10	Me	4-Br-Ph	**4j**	60
11	Me	4-F-Ph	**4k**	61
12	Me	4-CN-Ph	**4l**	50
13	Me	4-CF_3–Ph	**4m**	47
14	Me	3,5-di-Me-Ph	**4n**	72
15	Me	3-Cl-Ph	**4o** + **4o′** (5:2)	55
16	Me	3-Me-Ph	**4p** + **4p′** (6:5)	64
17	Me	2-Me-Ph	**4q**	45
18	Me	2-Naphtyl	**4r**	66
19	Me	2-Pyridyl	**4s**	53

[a]Reaction conditions: **1** (0.3 mmol), $Ru(bpy)_3Cl_2·6H_2O$ (5 mol %), and aryl diazonium salts **2** (0.75 mmol) in MeOH (3.0 mL) under visible-light irradiation for 12 h.

substrates, such as isopropyl, butyl, or benzyl could be used as an effective substituent group for this transformation (entries 1–4). However, the use of acetyl or N-free N-arylacrylamides resulted in no reaction (entries 5 and 6). The effect of various groups on the aromatic ring was also examined, and a strong dependence on the position of the substituents was observed. Substrates with both moderate electron-donating and electron-withdrawing substituents in the para position worked comparatively well (entries 7–11). A strong electron-withdrawing group resulted in slightly lower yields (entries 12 and 13). As was anticipated, when the substrates were substituted in the meta position, a mixture of **4o** + **4o′** (5:2) and **4p** + **4p′** (6:5) isomers was observed. (entries 15 and 16). When N-arylacrylamide **1q** bearing a ortho-substitued group was checked, the desired oxoindole **4q** was obtained in low yield (entry 17). The substrate disubstituted by methyl groups also gave a satisfactory yield of 72% (entry 14). Likewise, this reaction was also sustainable with the naphtyl and pyridyl groups as substituents (entries 18 and 19).

On the basis of the above results and related literature,[4−8,14] a plausible reaction mechanism is depicted in Scheme 2. Photoexcitation of $[Ru(bpy)]^{2+}$ by visible light generates excited $[Ru(bpy)]^{2+*}$, which is oxidatively quenched via single-electron transfer (SET) to the diazonium salt **2** to give $[Ru(bpy)]^{3+}$and an

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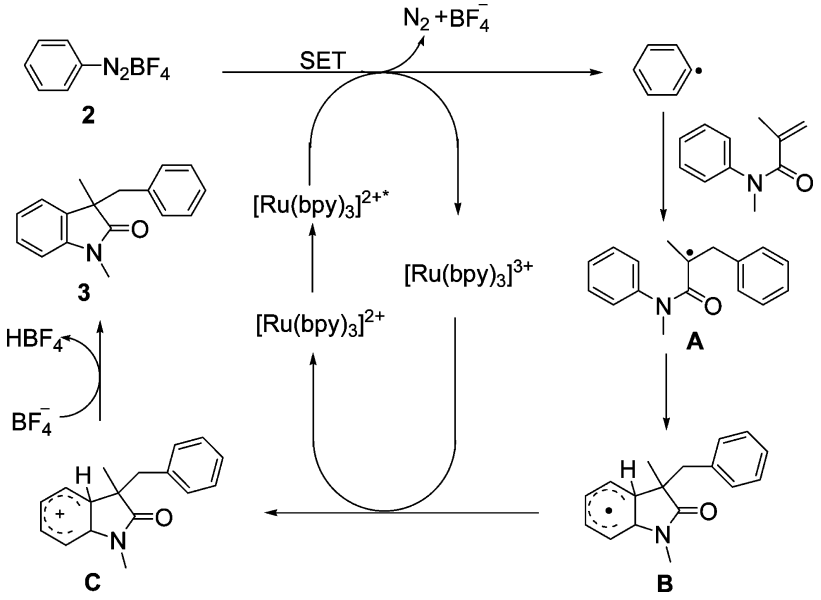
Scheme 2. Plausible Reaction Mechanism



ary radical. Subsequently, the addition of an aryl radical to N-arylacrylamides **1** generates the alkyl radical **A** followed by an intramolecular radical cyclization to give intermediate **B**. A single-electron oxidation of the intermediate **B** by $[Ru(bpy)]^{3+}$ regenerates the photocatalyst and forms the intermediate **C**, which dehydrogenated to give the product, **3**.

In conclusion, we have developed a visible-light-mediated radical reaction that allows the assembly of 3,3-disubstituted oxoindoles from two readily accessible building blocks, N-arylacrylamides and aryl diazonium salts, in moderate to good yields. The process showed considerable synthetic advantages in terms of product diversity, simplicity of the reaction procedure, and mild reaction conditions.

EXPERIMENTAL SECTION

General Methods. Chemicals used were obtained from commercial suppliers and used without further purifications. ^{1}H and ^{13}C NMR spectra were measured in $CDCl_3$ and recorded at 400 or 100 MHz, with TMS as an internal standard. Column chromatography over silica gel (300−400 mesh) and a petroleum ether/ethyl acetate combination was used as the eluent.

General Procedure for the Synthesis of 3,3-Disubstituted Oxoindoles. To a mixture of N-arylacrylamides **1** (0.30 mmol) and aryl diazonium tetrafluoroborate (0.75 mmol) in methanol (3.0 mL) was added Ru $(bpy)_3Cl_2{\cdot}6H_2O$ (0.0015 mmol). The solution was stirred at room temperature under visible-light irradiation for 12 h. The solvent was evaporated under vacuum. Then, the crude products were directly purified using flash column chromatography on silica gel (petroleum ether/ethyl acetate 10:1 as the eluant) to afford the desired oxindoles.

3-Benzyl-1,3-dimethylindolin-2-one (3a).[3h] White solid (60.2 mg, 80%). mp 86−88 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (t, J = 7.6 Hz, 1H), 7.12 (d, J = 7.6 Hz, 1H), 7.01−7.06 (m, 4H), 6.84 (d, J = 5.6 Hz, 2H), 6.60 (d, J = 7.6 Hz, 1H), 3.11 (d, J = 13.2 Hz, 1H), 3.00 (d, J = 13.2 Hz, 1H), 2.98 (s, 3H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.1, 143.3, 136.3, 133.2, 123.0, 127.9, 127.6, 126.6, 123.4, 122.2, 107.9, 50.1, 44.7, 26.0, 22.9. MS (EI, 70 eV) m/z: 251 $[M]^+$. Anal. Calcd for $C_{17}H_{17}NO$: C, 81.24; H, 6.82; N, 5.57. Found: C, 81.63; H, 6.47; N, 5.60.

3-(4-Fluorobenzyl)-1,3-dimethylindolin-2-one (3b). White solid (66.2 mg, 82%). mp 126−127 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15−7.20 (m, 2H), 7.04 (t, J = 7.2 Hz, 1H), 6.70−6.80 (m, 4H), 6.61 (d, J = 7.6 Hz, 1H), 3.11 (d, J = 13.2 Hz, 1H), 2.97 (s, 3H), 2.96 (d, J = 13.2 Hz, 1H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.8, 161.2 (d, J = 242.4 Hz), 143.3, 132.9, 132.0 (d, J = 3.3 Hz), 131.3 (d, J = 8.1 Hz), 128.0, 123.2, 122.3, 114.3 (d, J = 20.5 Hz), 107.9, 50.1, 43.8, 25.9, 22.8. MS (EI, 70 eV) m/z: 269 $[M]^+$. Anal. Calcd for $C_{17}H_{16}FNO$: C, 75.82; H, 5.99; N, 5.20. Found: C, 76.07; H, 6.30; N, 5.26.

3-(4-Bromobenzyl)-1,3-dimethylindolin-2-one (3c). White solid (75.0 mg, 76%). mp 110−112 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14−7.21 (m, 4H), 7.04 (t, J = 7.6 Hz, 1H), 6.70 (d, J = 7.6 Hz, 2H), 6.63 (d, J = 7.6 Hz, 1H), 3.10 (d, J = 12.8 Hz, 1H), 2.99 (s, 3H), 2.94 (d, J = 12.8 Hz, 1H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 143.2, 135.3, 132.7, 131.6, 130.7, 128.1, 123.2, 122.3, 120.6, 108.1, 49.9, 43.9, 26.0, 23.0. MS (EI, 70 eV) m/z: 329 $[M]^+$. Anal. Calcd for $C_{17}H_{16}BrNO$: C, 61.83; H, 4.88; N, 4.24. Found: C, 61.70; H, 5.03; N, 4.10.

3-(4-Methylbenzyl)-1,3-dimethylindolin-2-one (3d). White solid (50.1 mg, 63%). mp 116−118 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (t, J = 7.6 Hz, 1H), 7.10 (d, J = 7.2 Hz, 1H), 7.01 (t, J = 7.2 Hz, 1H), 6.85 (d, J = 7.2 Hz, 2H), 6.73 (d, J = 7.6 Hz, 2H), 6.62 (d, J = 7.6 Hz, 1H), 3.06 (d, J = 13.2 Hz, 1H), 3.00 (s, 3H), 2.97 (d, J = 13.2 Hz, 1H), 2.20 (s, 3H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.2, 143.3, 135.9, 133.3, 133.2, 129.9, 128.3, 127.8, 123.5, 122.1, 107.9, 49.99, 44.2, 26.0, 22.9, 21.1. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.07; H, 7.58; N, 5.40.

3-(2-Methylbenzyl)-1,3-dimethylindolin-2-one (3e). Colorless oil (46.1 mg, 58%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (t, J = 7.6 Hz, 1H), 6.90−7.04 (m, 5H), 6.86 (d, J = 7.2 Hz, 1H), 6.71 (d, J = 8.0 Hz, 1H), 3.07−3.11 (m, 5H), 2.01 (s, 3H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.7, 143.2, 137.3, 134.9, 133.3, 130.6, 130.3, 127.9, 126.7, 125.2, 123.8, 122.1, 107.9, 49.5, 40.1, 26.2, 22.7, 20.1. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.20; H, 7.01; N, 5.21.

3-(2-Fluorobenzyl)-1,3-dimethylindolin-2-one (3f). White solid (57.3 mg, 71%). mp 103−105 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (t, J = 8.0 Hz, 1H), 7.11 (d, J = 7.6 Hz, 1H), 6.98−7.06 (m, 3H), 6.88 (t, J = 7.2 Hz, 1H), 6.79 (t, J = 8.8 Hz, 1H), 6.64 (d, J = 7.6 Hz, 1H), 3.24 (d, J = 13.2 Hz, 1H), 3.08 (s, 3H), 3.03 (d, J = 13.2 Hz, 1H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.2, 160.9 (d, J_{C-F} = 244.7 Hz), 142.9, 132.8, 131.8 (d, J_{C-F} = 4.1 Hz), 128.4 (d, J_{C-F} = 8.4 Hz), 127.9, 123.8 (d, J_{C-F} = 15.0 Hz), 123.7, 123.3 (d, J_{C-F} = 3.7 Hz), 122.2, 115.0 (d, J_{C-F} = 23.3 Hz), 107.7, 49.5, 36.3, 26.1, 22.9. MS (EI, 70 eV) m/z: 269 $[M]^+$. Anal. Calcd for $C_{17}H_{16}FNO$: C, 75.82; H, 5.99; N, 5.20. Found: C, 76.01; H, 5.76; N, 5.08.

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3-(2-Chlorobenzyl)-1,3-dimethylindolin-2-one (3g). White solid (57.3 mg, 67%). mp 98−100 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15−7.19 (m, 2H), 7.07−7.11 (m, 2H), 7.02−7.04 (m, 2H), 6.94−6.98 (m, 1H), 6.66 (d, J = 7.6 Hz, 1H), 3.40 (d, J = 13.6 Hz, 1H), 3.19 (d, J = 13.6 Hz, 1H), 3.13 (s, 3H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.3, 142.9, 134.8, 134.7, 132.6, 131.4, 129.5, 128.0, 127.9, 126.1, 124.2, 122.1, 107.7, 49.6, 39.8, 26.2, 23.2. MS (EI, 70 eV) m/z: 285 $[M]^+$. Anal. Calcd for $C_{17}H_{16}ClNO$: C, 71.45; H, 5.64; N, 4.90. Found: C, 71.76; H, 5.93; N, 4.96.

3-(2-Bromobenzyl)-1,3-dimethylindolin-2-one (3h). Colorless oil (59.2 mg, 60%). mp 103−105 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, J = 8.4 Hz, 1H), 7.17 (t, J = 7.6 Hz, 1H), 7.09−7.11 (m, 3H), 6.94−6.97 (m, 2H), 6.67 (d, J = 8.4 Hz, 1H), 3.40 (d, J = 13.6 Hz, 1H), 3.24 (d, J = 13.6 Hz, 1H), 3.14 (s, 3H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.3, 142.9, 136.5, 132.9, 132.5, 131.2, 128.3, 127.9, 126.8, 125.9, 124.4, 122.1, 107.7, 49.5, 42.3, 26.2, 23.2. MS (EI, 70 eV) m/z: 329 $[M]^+$. Anal. Calcd for $C_{17}H_{16}BrNO$: C, 61.83; H, 4.88; N, 4.24. Found: C, 61.70; H, 5.11; N, 4.15.

3-Benzyl-1-ethyl-3-methylindolin-2-one (4a). White solid (57.2 mg, 72%). mp 76−78 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16−7.21 (m, 2H), 7.01−7.06 (m, 4H), 6.81 (d, J = 6.8 Hz, 2H), 6.61 (d, J = 8.0 Hz, 1H), 3.67−3.72 (m, 1H), 3.34−3.39 (m, 1H), 3.14 (d, J = 12.8 Hz, 1H), 3.01 (d, J = 12.8 Hz, 1H), 1.48 (s, 3H), 0.86 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.5, 142.4, 136.3, 133.4, 129.9, 127.8, 127.6, 126.5, 123.4, 121.9, 108.0, 49.9, 44.8, 34.3, 23.0, 12.2. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.30; H, 7.61; N, 5.19.

3-Benzyl-1-isopropyl-3-methylindolin-2-one (4b). White solid (56.1 mg, 67%). mp 119−121 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, J = 7.6 Hz, 1H), 7.15 (d, J = 7.6 Hz, 1H), 6.99−7.05 (m, 4H), 6.75−6.79 (m, 3H), 4.39−4.46 (m, 1H), 3.15 (d, J = 12.8 Hz, 1H), 2.98 (d, J = 12.8 Hz, 1H), 1.48 (s, 3H), 1.27 (d, J = 7.2 Hz, 3H), 1.02 (d, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.4, 141.9, 136.3, 133.7, 129.9, 127.6, 126.5, 123.5, 121.6, 109.7, 49.7, 45.1, 43.2, 23.1, 19.3, 18.9. MS (EI, 70 eV) m/z: 279 $[M]^+$. Anal. Calcd for $C_{19}H_{21}NO$: C, 81.68; H, 7.58; N, 5.01. Found: C, 81.34; H, 7.29; N, 5.12.

3-Benzyl-1-butyl-3-methylindolin-2-one (4c). Colorless oil (61.5 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15−7.19 (m, 2H), 7.02−7.05 (m, 4H), 6.83 (d, J = 7.2 Hz, 2H), 6.62 (d, J = 7.6 Hz, 1H), 3.57−3.62 (m, 1H), 3.30−3.38 (m, 1H), 3.13 (d, J = 13.2 Hz, 1H), 3.02 (d, J = 13.2 Hz, 1H), 1.47 (s, 3H), 1.19−1.30 (m, 2H), 1.06−1.14 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 142.9, 136.3, 133.4, 123.0, 127.8, 127.6, 126.5, 123.4, 121.9, 108.2, 49.9, 44.6, 39.6, 29.4, 23.4, 20.1, 13.9. MS (EI, 70 eV) m/z: 293 $[M]^+$. Anal. Calcd for $C_{20}H_{23}NO$: C, 81.87; H, 7.90; N, 4.77. Found: C, 81.73; H, 8.10; N, 4.83.

1,3-Dibenzyl-3-methylindolin-2-one (4d).[3i] Colorless oil (61.8 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 (d, J = 7.2 Hz, 2H), 7.11−7.15 (m, 4H), 7.04−7.07 (m, 4H), 6.88 (d, J = 7.6 Hz, 2H), 6.66 (d, J = 7.2 Hz, 2H), 6.41 (d, J = 7.2 Hz, 1H), 4.98 (d, J = 16.0 Hz, 1H), 4.47 (d, J = 16.0 Hz, 1H), 3.24 (d, J = 12.8 Hz, 1H), 3.12 (d, J = 12.8 Hz, 1H), 1.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.9, 142.5, 136.4, 135.5, 133.2, 130.2, 128.7, 127.9, 127.8, 127.2, 126.7, 126.6, 123.3, 122.3, 109.3, 50.3, 44.4, 43.6, 24.2. MS (EI, 70 eV) m/z: 327 $[M]^+$. Anal. Calcd for $C_{23}H_{21}NO$: C, 84.37; H, 6.46; N, 4.28. Found: C, 84.51; H, 6.20; N, 4.35.

3-Benzyl-1,3,5-trimethylindolin-2-one (4g). White solid (55.6 mg, 70%). mp 72−74 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.04−7.05 (m, 3H), 6.97 (d, J = 8.0 Hz, 1H), 6.93 (s, 1H), 6.84−6.85 (m, 2H), 6.49 (d, J = 8.0 Hz, 1H), 3.09 (d, J = 13.2 Hz, 1H), 2.98 (d, J = 13.2 Hz, 1H), 2.95 (s, 3H), 2.33 (s, 3H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.0, 140.9, 136.4, 133.2, 131.6, 129.9, 128.1, 127.6, 126.5 124.3, 107.6, 50.0, 44.7, 26.0, 22.9, 21.3. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.22; H, 7.53; N, 5.36.

3-Benzyl-5-methoxy-1,3-dimethylindolin-2-one (4h). Colorless oil (63.2 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06−7.07 (m, 3H), 6.87−6.89 (m, 2H), 6.69−6.71 (m, 2H), 6.51 (d, J = 8.0 Hz, 1H), 3.77 (s, 3H), 3.09 (d, J = 13.2 Hz, 1H), 2.98 (d, J = 13.2 Hz, 1H), 2.96 (s, 3H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 155.8, 136.9, 136.3, 134.5, 130.0, 127.6, 126.6, 112.1, 111.1, 108.1, 55.9, 50.4, 44.6, 26.1, 22.9. MS (EI, 70 eV) m/z: 281 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 77.13; H, 7.05; N, 5.01.

3-Benzyl-5-chloro-1,3-dimethylindolin-2-one (4i). Colorless oil (53.9 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.07−7.15 (m, 5H), 6.84−6.85 (m, 2H), 6.51 (d, J = 8.0 Hz, 1H), 3.12 (d, J = 12.8 Hz, 1H), 2.97 (d, J = 12.8 Hz, 1H), 2.95 (s, 3H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.5, 141.8, 135.8, 134.9, 129.9, 127.8, 127.7, 127.6, 126.7, 123.9, 108.7, 50.4, 44.7, 26.1, 22.7. MS (EI, 70 eV) m/z: 285 $[M]^+$. Anal. Calcd for $C_{17}H_{16}ClNO$: C, 71.45; H, 5.64; N, 4.90. Found: C, 71.69; H, 5.31; N, 4.99.

3-Benzyl-5-bromo-1,3-dimethylindolin-2-one (4j). White solid (59.2 mg, 60%). mp 96−98 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23−7.31 (m, 2H), 7.07−7.08 (m, 3H), 6.84−6.85 (m, 2H), 6.47 (d, J = 8.0 Hz, 1H), 3.11 (d, J = 13.2 Hz, 1H), 2.97 (d, J = 13.2 Hz, 1H), 2.95 (s, 3H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.4, 142.4, 135.8, 135.3, 130.7, 129.9, 127.8, 126.8, 126.7, 114.9, 109.3, 50.3, 44.7, 26.1, 22.7. MS (EI, 70 eV) m/z: 329 $[M]^+$. Anal. Calcd for $C_{17}H_{16}BrNO$: C, 61.83; H, 4.88; N, 4.24. Found: C, 61.58; H, 5.09; N, 4.39.

3-Benzyl-5-fluoro-1,3-dimethylindolin-2-one (4k). Colorless oil (47.8 mg, 61%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.07 (bs, 3H), 6.86 (bs, 4H), 6.49−6.51 (m, 2H), 3.13 (d, J = 12.8 Hz, 1H), 2.97 (d, J = 12.8 Hz, 1H), 2.96 (s, 3H), 1.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 159.2 (d, J = 239.1 Hz), 139.2, 135.9, 134.9 (d, J = 15.3 Hz), 129.8, 127.7, 126.7, 114.0 (d, J = 23.2 Hz), 111.5 (d, J = 24.4 Hz), 108.2 (d, J = 8.7 Hz), 50.6, 44.6, 26.1, 22.8. MS (EI, 70 eV) m/z: 269 $[M]^+$. Anal. Calcd for $C_{17}H_{16}FNO$: C, 75.82; H, 5.99; N, 5.20. Found: C, 75.58; H, 6.23; N, 5.04.

3-Benzyl-1,3-dimethyl-2-oxoindoline-5-carbonitrile (4l). White solid (41.5 mg, 50%). mp 122−124 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (dd, J = 8.4 Hz, J = 1.6 Hz, 1H), 7.37 (d, J = 1.2 Hz, 1H), 7.07−7.09 (m, 3H), 6.80 (dd, J = 7.6 Hz, J = 1.6 Hz, 2H), 6.60 (d, J = 8.0 Hz, 1H), 3.16 (d, J = 13.2 Hz, 1H), 3.01 (d, J = 13.2 Hz, 1H), 3.00 (s, 3H), 1.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 147.0, 135.2, 134.1, 133.2, 129.6, 127.8, 126.9, 126.6, 119.4, 108.2, 105.1, 49.9, 44.5, 26.1, 22.5. MS (EI, 70 eV) m/z: 276 $[M]^+$. Anal. Calcd for $C_{18}H_{16}N_2O$: C, 78.24; H, 5.84; N, 10.14. Found: C, 78.60; H, 5.58; N, 10.25.

3-Benzyl-5-(trifluoromethyl)-1,3-dimethylindolin-2-one (4m). White solid (45.0 mg, 47%). mp 85−87 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 8.0 Hz, 1H), 7.33 (s, 1H), 7.05−7.06 (m, 3H), 6.81 (d, J = 7.2 Hz, 2H), 6.65 (d, J = 8.0 Hz, 1H), 3.14 (d, J = 12.8 Hz, 1H), 3.01 (d, J = 12.8 Hz, 1H), 3.00 (s, 3H), 1.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.9, 146.2, 135.6, 133.7, 129.8, 127.8, 127.4 (q, J = 269.6 Hz), 126.8, 125.6 (q, J = 4.6 Hz), 124.4 (q, J = 31.8 Hz), 120.5 (q, J = 3.6 Hz), 107.5, 50.1, 44.6, 26.1, 22.5. MS (EI, 70 eV) m/z: 319 $[M]^+$. Anal. Calcd for $C_{18}H_{16}F_3NO$: C, 67.70; H, 5.05; N, 4.39. Found: C, 67.48; H, 5.22; N, 4.21.

3-Benzyl-1,3,4,6-tetramethylindolin-2-one (4n). White solid (60.3 mg, 72%). mp 77−79 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.98−7.00 (m, 3H), 6.78−6.80 (m, 2H), 6.63 (s, 1H), 6.25 (s, 1H), 3.22 (d, J = 13.2 Hz, 1H), 3.16 (d, J = 13.2 Hz, 1H), 2.89 (s, 3H), 2.47 (s, 3H), 2.26 (s, 3H), 1.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.1, 143.6, 137.7, 136.8, 133.8, 129.2, 127.6, 126.9, 126.4, 125.5, 106.6, 51.0, 42.6, 25.9, 21.8, 21.6, 18.5. MS (EI, 70 eV) m/z: 279 $[M]^+$. Anal. Calcd for $C_{19}H_{21}NO$: C, 81.68; H, 7.58; N, 5.01. Found: C, 81.35; H, 7.86; N, 5.12.

3-Benzyl-4-chloro-1,3-dimethylindolin-2-one and 3-Benzyl-6-chloro-1,3-dimethylindolin-2-one (4o + 4o′). Colorless oil (47.0 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05−7.08 (m, 2H), 6.96−6.99 (m, 5H), 6.89−6.92 (m, 2H), 6.84−6.85 (m, 0.9H), 6.61 (s, 0.4H), 6.43 (d, J = 7.6 Hz, 1H), 3.51 (d, J = 12.8 Hz, 1H), 3.17 (d, J = 12.8 Hz, 1H), 3.10 (d, J = 13.6 Hz, 0.4H), 2.94−3.00 (m, 2H), 2.92 (s, 3H), 1.65 (s, 3H), 1.45 (s, 1.2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.0, 179.3, 145.0, 144.4, 136.5, 135.9, 133.6, 131.5, 130.8, 129.9, 129.7, 129.12, 129.1, 127.8, 127.6, 126.7, 126.5, 124.3, 123.4, 121.9, 115.6, 108.6, 106.3, 52.3, 49.8, 44.5, 41.4, 26.14, 26.10, 22.8, 20.8. MS (EI, 70 eV) m/z: 285 $[M]^+$. Anal. Calcd for $C_{17}H_{16}ClNO$: C, 71.45; H, 5.64; N, 4.90. Found: C, 71.80; H, 5.93; N, 4.71.

3-Benzyl-1,3,4-trimethylindolin-2-one and 3-Benzyl-1,3,6-trimethylindolin-2-one (4p + 4p′). Colorless oil (51.0 mg, 64%).

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^{1}H NMR (400 MHz, $CDCl_3$): δ 6.97−7.07 (m, 7.3H), 6.76−6.87 (m, 5.7H), 6.45 (S, 0.8H), 6.37 (d, J = 7.6 Hz, 1H), 3.00−3.22 (m, 3.7H), 2.97 (s, 2.5H), 2.89 (s, 3H), 2.52 (s, 3H), 2.32 (s, 2.5H), 1.59 (s, 3H), 1.44 (s, 2.5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.5, 179.8, 143.6, 143.3, 137.9, 136.6, 136.5, 134.2, 130.2, 130.0, 129.9, 129.2, 127.7, 127.6, 127.5, 126.5, 126.4, 124.9, 123.2, 122.7, 108.9, 105.6, 51.3, 49.8, 44.6, 42.6, 25.98, 25.96, 23.1, 21.9, 21.6, 18.7. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.85; H, 7.60; N, 5.06.

3-Benzyl-1,3,7-trimethylindolin-2-one (4q). Colorless oil (35.8 mg, 45%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05−7.07 (m, 3H), 6.89−6.95 (m, 3H), 6.82 (d, J = 6.4 Hz, 2H), 3.25 (s, 3H), 3.09 (d, J = 12.8 Hz, 1H), 2.97 (d, J = 12.8 Hz, 1H), 2.39 (s, 3H), 1.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.8, 141.1, 136.4, 133.8, 131.5, 129.9, 127.5, 126.5,122.0, 121.3, 119.4, 49.4, 45.0, 29.3, 23.2, 18.9. MS (EI, 70 eV) m/z: 265 $[M]^+$. Anal. Calcd for $C_{18}H_{19}NO$: C, 81.47; H, 7.22; N, 5.28. Found: C, 81.72; H, 7.62; N, 5.08.

3-Benzyl-1,3-dimethyl-1*H*-benzo[*g*]indol-2(3*H*)-one (4r). White solid (59.6 mg, 66%). mp 115−117 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59−7.62 (m, 1H), 7.45 (t, J = 7.6 Hz, 1H), 7.31−7.36 (m, 2H), 7.16 (t, J = 8.0 Hz, 1H), 6.74−6.83 (m, 3H), 6.52 (t, J = 7.6 Hz, 1H), 6.36 (t, J = 7.2 Hz, 2H), 3.32 (d, J = 12.8 Hz, 1H), 3.25 (s, 3H), 2.83 (d, J = 12.8 Hz, 1H), 1.82 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 137.2, 136.5, 136.4, 132.8, 129.5, 127.2, 126.6, 126.4, 126.1, 126.09, 123.2, 122.1, 120.3, 107.9, 52.8, 49.2, 29.4, 26.9. MS (EI, 70 eV) m/z: 301 $[M]^+$. Anal. Calcd for $C_{21}H_{19}NO$: C, 83.69; H, 6.35; N, 4.65. Found: C, 83.36; H, 6.05; N, 4.78.

3-Benzyl-1,3-dimethyl-1*H*-pyrrolo[2,3-*b*]pyridin-2(3*H*)-one (4s). Colorless oil (40.1 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (d, J = 4.8 Hz, 1H), 7.26 (s, 1H), 7.10 (bs, 3H), 6.87−6.92 (m, 3H), 3.12 (d, J = 13.6 Hz, 1H), 3.10 (s, 3H), 3.02 (d, J = 13.6 Hz, 1H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.8, 156.8, 146.8, 135.7, 130.8, 129.9, 127.9, 127.5, 126.9, 117.7, 49.6, 44.1, 25.2, 22.3. MS (EI, 70 eV) m/z: 252 $[M]^+$. Anal. Calcd for $C_{16}H_{16}N_2O$: C, 76.16; H, 6.39; N, 11.10. Found: C, 76.42; H, 6.01; N, 10.89.

■ ASSOCIATED CONTENT

Supporting Information

^{1}H and ^{13}C NMR spectra for all compounds and X-ray crystal structure of **3a** (CIF). This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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