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铱催化 *N*-芳基-2-氨基吡啶和碳酸亚乙烯酯合成吲哚衍生物

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摘要 吲哚及其衍生物是一类重要的杂环化合物. 在本次研究中, 设计并开发了铱催化的 *N*-芳基-2-氨基吡啶和碳酸亚乙烯酯作为新的 C2 合成子进行环合反应合成吲哚衍生物的方法. 该方法有望为各种吲哚衍生物的合成提供一种简便且有用的途径.

关键词 吲哚; 铱催化; 环合反应; 碳酸亚乙烯酯; 氨基吡啶

Iridium-Catalyzed Synthesis of Indole Derivatives from *N*-Aryl-2-aminopyridines and Vinylene Carbonate

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Abstract Indoles and their derivatives are an important class of N-heterocycles. In this article, iridium-catalyzed annulation reactions of *N*-aryl-2-aminopyridines to synthesize indole derivatives are designed and developed, which utilize vinylene carbonate as a new C2 synthon. This protocol is expected to provide a facile and useful access to various indole derivatives.

Keywords indoles; iridium-catalyzed; annulation reactions; vinylene carbonate; aminopyridines

1 Introduction

Indoles and their derivatives are an important class of N-heterocycles, which are widely prevalent in natural bioactive molecules, pharmaceuticals, and functional materials.[1] As a result, a great deal of effort has been dedicated to developing methodologies for preparing indole scaffolds.[2] Among them, the most notable example is Fischer indole synthesis, which prepares indoles from arylhydrazine and carbonyl compounds (Scheme 1a).[3] Subsequently, Larock's group developed palladium-catalyzed coupling reactions of carbonyl compounds and *o*-haloaniline derivants to perfect indole synthesis.[4] However, most of them also face some unavoidable drawbacks, such as limited substrate scope, the necessity for strong bases and harsh reaction conditions.

Considering the limitations of the traditional synthesis method, in recent years, many transition metal-catalysed coupling reactions utilizing a directing group were exploited to the synthesis of indoles (Scheme 1b).[5] In 2015, Kim's group[6] demonstrated a rhodium(III)-catalyzed coupling reactions of anilines with diazo compounds to synthesize highly substituted indoles. In 2019, Huang's group[7] demonstrated that sulfoxonium ylides are important building blocks in annulation reactions, and reported a Ru(II)-catalyzed intermolecular annulation of *N*-aryl-2-aminopyridines and sulfoxonium ylides to synthesize indole derivatives. Recently, Tao and Xia's group[8] reported a Pd-catalyzed [3 + 2] annulation of *N*-aryl-2-aminopyridines and alkynes, which provides an

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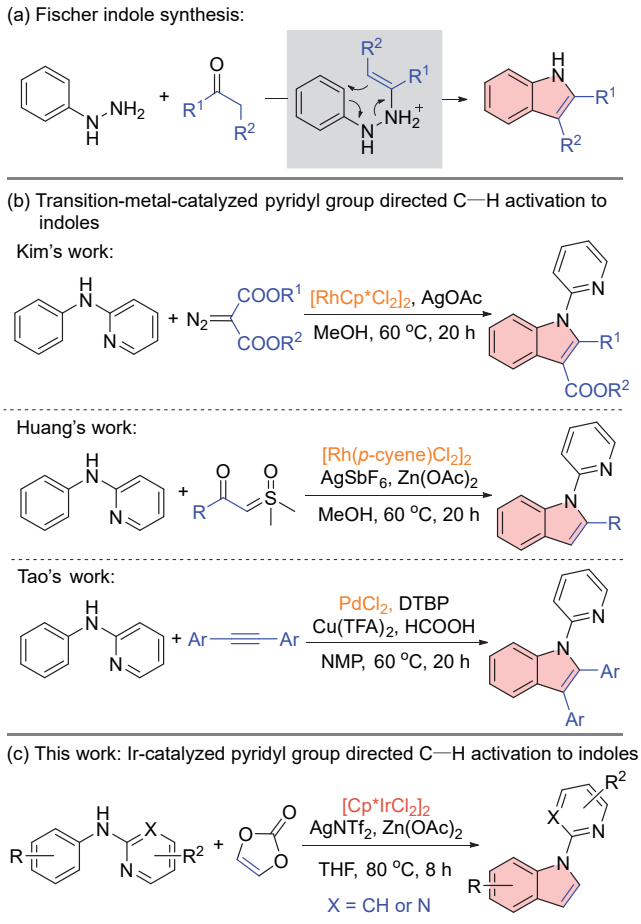
efficient strategy for the synthesis of highly substituted indoles. Despite these considerable progress, there is still a great demand for the development of the efficient synthesis of indole derivatives.

Recently, vinylene carbonate, as an ideal synthetic precursor of acetylene surrogate, has been widely used in annulation reactions to synthesize various polycyclic heteroaromatic scaffolds.[9] Inspired by these elegant progresses and our previous research,[10] herein, we design and develop an Ir-catalyzed annulation reactions of *N*-aryl-2-aminopyridines to synthesize indole derivatives (Scheme 1c), which utilizing vinylene carbonate as a new C2 synthon.

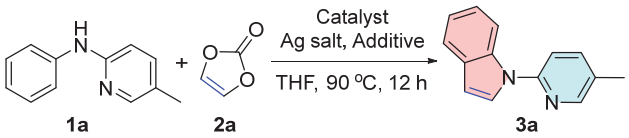


Scheme 1 Representative synthetic methods for indole derivatives

2 Results and discussion

Inspired by our recent research and the reported work involving vinylene carbonate, our investigation commenced with the utilization of *N*-aryl-2-aminopyridines (**1a**) and vinylene carbonate (**2a**) as the model substrates using 5 mol% $[Cp^*RhCl_2]_2$ in tetrahydrofuran (THF) with $AgSbF_6$ as the sliver salt and PivOH as the additive at 90 ℃ (Table 1, Entry 1). Unfortunately, the cycloaddition product **3a** was not obtained after attempts even other common catalysts, such as $[Ru(p\text{-cymene})Cl_2]_2$, $[Cp^*Co(CO)I_2]$ and $Pd(OAc)_2$ (Table 1, Entries 2～4). Subsequently, our focus shifted to Ir-complexes, which is known for their outstanding performance in metal-catalyzed coupling reactions.[11] To our delight, the desired product **3a** was first obtained in 46% yield using $[Cp^*IrCl_2]_2$ as catalyst (Table 1, Entry 5). Considering the importance of additives in current reaction, other additives were investigated (Table 1, Entries 6～9), such as HOAc, NaOAc, $Cu(OAc)_2$ and $Zn(OAc)_2$, and the results revealed that $Zn(OAc)_2$ was beneficial for the reaction, the yield of **3a** increased to 56%. Then several solvents were investigated, among which THF gave the superior result compared with 1,2-dichloroethane (DCE), ethyl acetate (EA) and PhMe (Table 1, Entries 10～12). Subsequently, screening of sliver salt revealed that $AgNTf_2$ was the optimal choice and 73% yield was obtained (Table 1, Entries 13～15). In order to further complete the catalytic system, screening of other parameters, such as the amount of reactants, reaction temperature and reaction time, the optimal conditions are established as follows: *N*-phenylpyridin-2-amine (**1a**, 0.2 mmol), vinylene carbonate (**2a**, 0.4 mmol), $[Cp^*IrCl_2]_2$ (5 mol%), $AgNTf_2$ (20 mol%), $Zn(OAc)_2$ (5 mol%), in THF (2 mL) at 80 ℃ for 8 h, where the yield of **3a** reached 75% (Table 1, Entry 16).

Table 1 Optimization of reaction conditions[a]



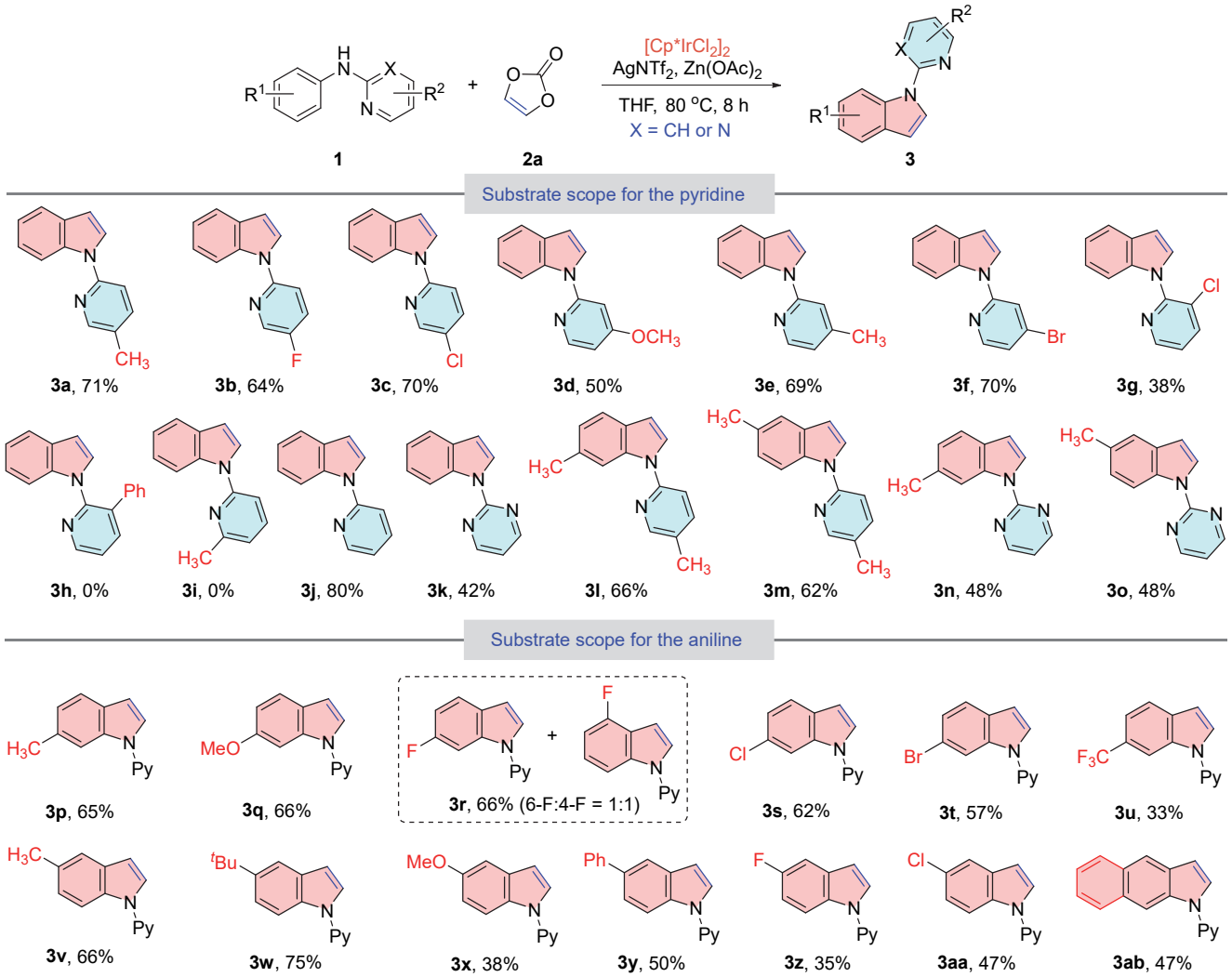
Entry	Catalyst	Ag salt	Additive	Solvent	Yield[b]/%
1	$[Cp^*RhCl_2]_2$	$AgSbF_6$	PivOH	THF	Trace
2	$[Ru(p\text{-cymene})Cl_2]_2$	$AgSbF_6$	PivOH	THF	Trace
3	$[Cp^*Co(CO)I_2]$	$AgSbF_6$	PivOH	THF	Trace
4	$Pd(OAc)_2$	$AgSbF_6$	PivOH	THF	Trace
5	$[Cp^*IrCl_2]_2$	$AgSbF_6$	PivOH	THF	46
6	$[Cp^*IrCl_2]_2$	$AgSbF_6$	HOAc	THF	31
7	$[Cp^*IrCl_2]_2$	$AgSbF_6$	NaOAc	THF	Trace
8	$[Cp^*IrCl_2]_2$	$AgSbF_6$	$Cu(OAc)_2$	THF	Trace
9	$[Cp^*IrCl_2]_2$	$AgSbF_6$	$Zn(OAc)_2$	THF	56
10	$[Cp^*IrCl_2]_2$	$AgSbF_6$	$Zn(OAc)_2$	DCE	35
11	$[Cp^*IrCl_2]_2$	$AgSbF_6$	$Zn(OAc)_2$	EA	18
12	$[Cp^*IrCl_2]_2$	$AgSbF_6$	$Zn(OAc)_2$	PhMe	51
13	$[Cp^*IrCl_2]_2$	$AgBF_4$	$Zn(OAc)_2$	THF	Trace
14	$[Cp^*IrCl_2]_2$	$AgNTf_2$	$Zn(OAc)_2$	THF	73
15	$[Cp^*IrCl_2]_2$	AgOTf	$Zn(OAc)_2$	THF	54
16[c]	$[Cp^*IrCl_2]_2$	$AgNTf_2$	$Zn(OAc)_2$	THF	75, 71[d]

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), catalyst (5 mol%), Ag salt (20 mol%), additive (5 mol%), THF (2 mL) at 90 ℃ for 12 h. [b] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as the internal standard. [c] At 80 ℃ for 8 h. [d] Isolated yields.

With suitable conditions established, we subsequently started to surveyed the scope and generality of various substituted pyridines for this [3＋2] annulation, and the results are showned in Scheme 2. As for directing group pyridine fragments, it was found that this [3＋2] annulation

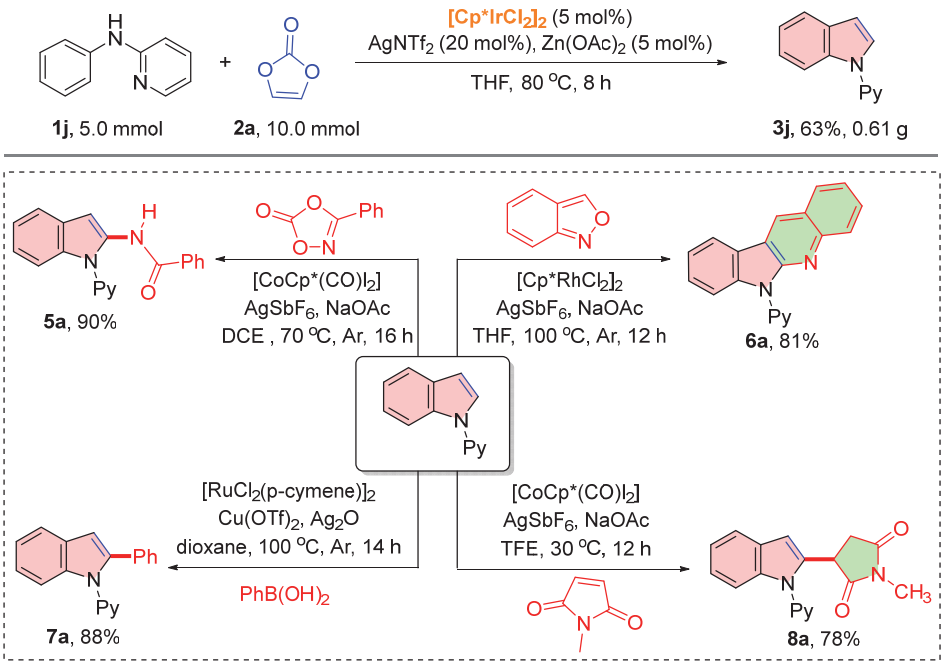
was not sensitive to the electronegativity of substituents, both electron-withdrawing group (F, Cl) or electron-donating group (Me) at C(3)- or C(4)-position of the pyridine rings reacted successfully, delivering the corresponding indoles (**3a**～**3f**) in 50%～71% yields. This reaction seems to be tremendously influenced by the steric hindrance effect, and the yield of C(5)-cholro-substituted pyridine **3g** was decreased dramatically to 38%, even the cycloaddition product **3h** was not obtained. Besides, the stability of Ir-com- plexes intermediate might be influenced by C(2)-substituted pyridine generating the desired product **3i**, but **3i** was not obtained. Moreover, anilines containing pyridine **3j** and *N*-pyrimidyl **3k** are also suitable for the reaction with 73% and 42% yields, respectively. Finally, polysubstituted substrates could also react with **2a** to give products **3l**～**3o** in acceptable yield. After determining the optimal guiding group **3j**, various aniline fragments were also examined under the optimized conditions. Generally, this reaction has good functional group compatibility, anilines bearing electron-donating (Me, OMe, *t*Bu, Ph) or electron-withdrawing groups (F, Cl, Br, CF_3) on the benzene ring could successfully couple with vinylene carbonate to give indole derivatives in moderate to good yields (**3p**～**3aa**). Among them, anilines containing the F or Cl group at the *para*-position were not sensitive in this catalytic system, affording **3z** and **3aa** in 35% and 47% yields, respectively. It is worth noting that the large *N*-(naphthalene-2-yl)pyridine-2-amine (**3ab**) was also found to be effective for this [3＋2] annulation, and provided the desired products in 47% yields.

In order to explore the synthetic utility of our catalytic system, a scaled-up reaction with 5 mmol of *N*-phenylpyridin-2-amine (**1j**) was carried out, and indole **3j** was obtained in 63% yields. Considering the importance of indole compounds,[12] some modification of the coupling product **3j** was attempted. As expected, the obtained indole **3j** could be further converted into various substituted indoles (**5a**～**8a**) under different catalytic systems, which clearly disclosed the synthetic utility of this method (Scheme 3).



Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), $[Cp^*IrCl_2]_2$ (5 mol%), $AgNTf_2$ (20 mol%), $Zn(OAc)_2$ (5 mol%), THF (2 mL) at 80 ℃ for 8 h. Isolated yields.

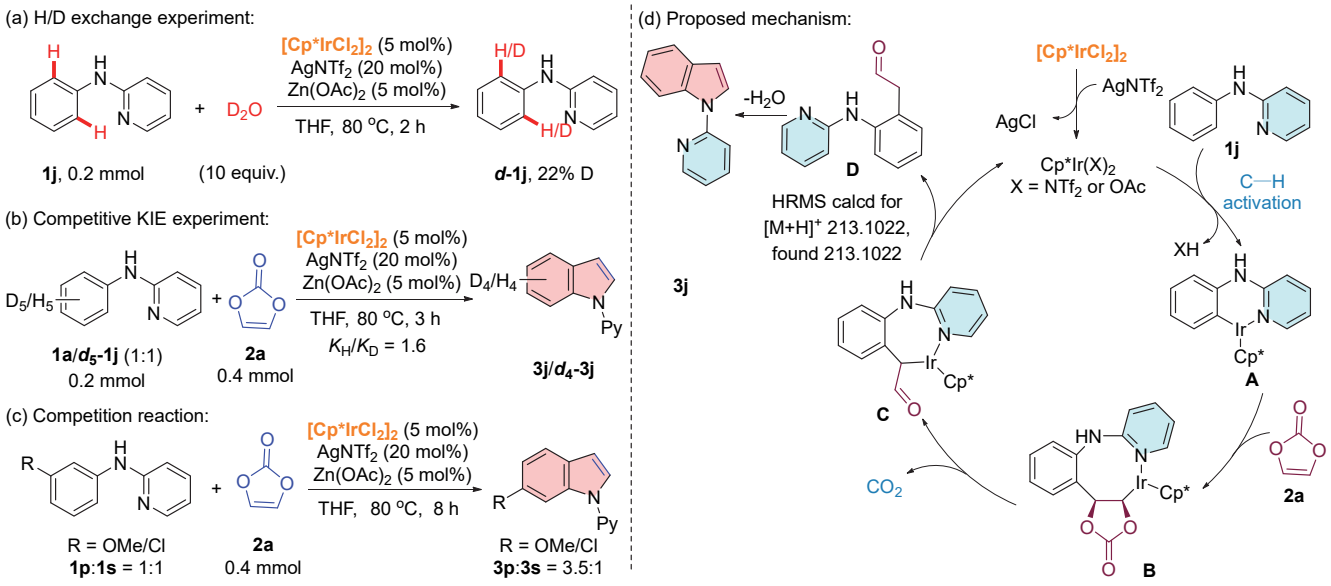
Scheme 2 Substrate scope for the pyridine



Scheme 3 Gram-scale synthesis and derivatization of the coupling product **3j**

Some mechanistic experiments were then carried out to explore the experimental mechanism (Scheme 4). Initially, the reaction was carried out by using **1j** with D_2O under the standard conditions for 2 h in the absence of vinylene carbonate (**2a**), which showed that 22% deuterium incorporation at the *ortho*-position of **1j** (Scheme 4a). This indicated that the C—H bond cleavage of this transformation was reversible. Then, the intermolecular competitive kinetic isotope effect (KIE) experiment of **1j** and ***d*$_5$-1j** was implemented under the standard conditions for 3 h and revealed a KIE value of 1.6 (Scheme 4b), which implying that *ortho*-C—H bond cleavage might not be involved in the rate-determining process. Finally, intermolecular competition experiment between **1q** and **1s** was conducted, and the **3q** and **3s** were obtained in a ratio of 3.5∶1, which indicated the higher reactivity for the electron-rich substrate (Scheme 4c).

Based on these preliminary results and previous reports,[13] a proposed mechanism for this transformation was proposed in Scheme 4d. Firstly, substrate **1j** transforms into an iridium intermediate **A** via C—H bond activation. Subsequently, intermediate **A** reacts with vinylene carbonate **2a** to form eight-member intermediate **B**, which can translate into intermediate **C** by a decarboxylation. Then



Scheme 4 Mechanistic studies and proposed mechanism

intermediate **D** is obtained from protonation of intermediate **C**, while releasing active Ir-complexes to complete the catalytic cycle. Finally, the coupling product **3j** is formed via eliminating water.

3 Conclusions

In summary, an iridium-catalyzed [3+2] annulation of *N*-phenylpyridin-2-amine with readily available vinylene carbonate has been developed, which opens an efficient approach for the synthesis of indole derivatives. This method features broad substrate scopes and good yields.

4 Experimental section

4.1 General experimental information

All the solvents involved in the reaction were dried by standard methods. All the drugs involved in the reaction were purchased from chemical reagent suppliers such as Anergy, Bidet, *etc.*, and were used without further purification. All products were separated by silica gel (200～300 mesh) column chromatography with petroleum ether (PE) (60～90 ℃) and ethyl acetate (EA). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance 500 spectrometer at ambient temperature with $CDCl_3$ as solvent and tetramethylsilane (TMS) as the internal standard. High resolution mass spectrometry (HRMS) data were recorded by an Agilent LC 1200/MS QTOF6520. Melting points were measured by a WRS-1B type melting point apparatus and were uncorrected.

4.2 General procedure for synthesis of indole **3**

The synthesis of 1-(5-methylpyridin-2-yl)-1*H*-indole (**3a**) was taken as an example. 5-Methyl-*N*-phenylpyridin-2-amine (**1a**) (0.2 mmol), carbonate ethylene (**2a**) (0.4 mmol), $[Cp^*IrCl_2]_2$ (5 mol%), $AgNTf_2$ (20 mol%) and $Zn(OAc)_2$ (5 mol%) were dissolved in THF (2 mL). The reaction mixture was stirred at 80 ℃ for 8 h. The resulting mixture was cooled to room temperature. After concentrating on the solvent, the product was purified by flash chromatography with *V*(petroleum ether, PE)∶*V*(ethyl acetate, EA)=50∶1 to give the product **3a**.

1-(5-Methylpyridin-2-yl)-1*H*-indole (**3a**): 71% yield as a white solid. m.p. 107～109 ℃; R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.40 (d, *J*=2.1 Hz, 1H), 8.13 (dd, *J*=8.3, 1.0 Hz, 1H), 7.70 (d, *J*=3.5 Hz, 1H), 7.67 (d, *J*=7.8 Hz, 1H), 7.63 (dd, *J*=8.3, 2.4 Hz, 1H), 7.41 (d, *J*=8.3 Hz, 1H), 7.29 (ddd, *J*=8.4, 7.1, 1.2 Hz, 1H), 7.20 (ddd, *J*=7.9, 7.0, 1.0 Hz, 1H), 6.71 (dd, *J*=3.5, 0.8 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.4, 149.0, 139.1, 135.1, 130.3, 129.8, 126.2, 123.0, 121.1, 121.1, 114.5, 112.7, 105.1, 17.9; HRMS (ESI-TOF) calcd for $C_{14}H_{13}N_2$ $[M+H]^+$ 209.1073, found 209.1080.

1-(5-Fluoropyridin-2-yl)-1*H*-indole (**3b**):[14] 64% yield as a white solid. m.p. 74～76 ℃; R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.43 (d, *J*=3.0 Hz, 1H), 8.11 (dd, *J*=8.4, 1.0 Hz, 1H), 7.71～7.63 (m, 2H), 7.56 (ddd, *J*=8.9, 7.4, 3.0 Hz, 1H), 7.47 (dd, *J*=8.9, 3.6 Hz, 1H), 7.30 (ddd, *J*=8.4, 7.1, 1.3 Hz, 1H), 7.22 (ddd, *J*=8.0, 7.1, 1.0 Hz, 1H), 6.72 (dd, *J*=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.05, 156.04, 148.83, 148.81, 136.79, 136.59, 135.18, 130.38, 126.19, 125.79, 125.63, 123.35, 121.46, 121.29, 115.61, 115.58, 112.59, 105.6; HRMS (ESI-TOF) calcd for $C_{13}H_{10}FN_2$ $[M+H]^+$ 213.0823, found 213.0827.

1-(5-Chloropyridin-2-yl)-1*H*-indole (**3c**):[14] 70% yield as a pale yellow solid foam. R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.51 (d, *J*=2.5 Hz, 1H), 8.18 (dd, *J*=8.4, 1.0 Hz, 1H), 7.77 (ddd, *J*=8.7, 2.6, 1.0 Hz, 1H), 7.70～7.62 (m, 2H), 7.44 (dt, *J*=8.8, 0.9 Hz, 1H), 7.32 (ddd, *J*=8.4, 7.0, 1.3 Hz, 1H), 7.23 (ddd, *J*=8.0, 7.1, 1.0 Hz, 1H), 6.73 (dd, *J*=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.8, 147.6, 138.25, 135.10, 130.57, 127.61, 125.86, 123.52, 121.69, 121.32, 115.09, 113.12, 106.2; HRMS (ESI-TOF) calcd for $C_{13}H_{10}ClN_2$ $[M+H]^+$ 229.0527, found 229.0528.

(4-Methoxypyridin-2-yl)-1*H*-indole (**3d**): 50% yield as a pale yellow oil. R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.39 (d, *J*=5.8 Hz, 1H), 8.16 (dd, *J*=8.4, 0.9 Hz, 1H), 7.72 (d, *J*=3.5 Hz, 1H), 7.66 (d, *J*=7.7 Hz, 1H), 7.32～7.28 (m, 1H), 7.21 (td, *J*=7.5, 0.9 Hz, 1H), 7.00 (d, *J*=2.2 Hz, 1H), 6.74 (dd, *J*=5.8, 2.3 Hz, 1H), 6.71 (d, *J*=3.4 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 167.6, 154.0, 150.1, 135.2, 130.5, 126.3, 123.1, 121.3, 121.2, 112.9, 107.1, 105.4, 100.7, 55.6; HRMS (ESI-TOF) calcd for $C_{14}H_{13}N_2O$ $[M+H]^+$ 247.0842, found 247.0844.

1-(4-Methylpyridin-2-yl)-1*H*-indole (**3e**):[14] 69% yield as a pale-yellow oil. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.43 (d, *J*=5.1 Hz, 1H), 8.19 (d, *J*=8.4 Hz, 1H), 7.72 (d, *J*=3.5 Hz, 1H), 7.67 (dt, *J*=7.9, 1.0 Hz, 1H), 7.33～7.28 (m, 2H), 7.21 (ddd, *J*=8.0, 7.1, 1.0 Hz, 1H), 7.00 (dd, *J*=5.1, 1.3 Hz, 1H), 6.71 (dd, *J*=3.5, 0.8 Hz, 1H), 2.45 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.73, 149.92, 148.71, 135.22, 130.48, 126.20, 123.10, 121.48, 121.24, 121.15, 115.45, 113.0, 105.3, 21.4; HRMS (ESI-TOF) calcd for $C_{14}H_{13}N_2$ $[M+H]^+$ 231.0893, found 231.0898.

1-(4-Bromopyridin-2-yl)-1*H*-indole (**3f**):[14] 70% yield as yellow oil. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.38 (d, *J*=5.3 Hz, 1H), 8.24 (dd, *J*=8.4, 0.9 Hz, 1H), 7.71～7.64 (m, 3H), 7.35～7.30 (m, 2H), 7.23 (ddd, *J*=8.0, 7.1, 1.0 Hz, 1H), 6.73 (dd, *J*=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 153.4, 149.6, 135.1, 134.3, 130.7, 125.7, 123.6, 123.2, 121.8, 121.3, 117.4, 113.3, 106.6; HRMS (ESI-TOF) calcd for $C_{13}H_{10}BrN_2$ $[M+H]^+$ 273.0022, found 273.0020.

1-(3-Chloropyridin-2-yl)-1*H*-indole (**3g**):[14] 38% yield as a colorless oil. R_f=0.4 [*V*(PE)∶*V*(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.53 (dd, *J*=4.6, 1.7 Hz, 1H), 7.94 (dd, *J*=8.0, 1.6 Hz, 1H), 7.69 (d, *J*=7.6 Hz, 1H), 7.55 (dd, *J*=3.5, 1.4 Hz, 1H), 7.51 (dd, *J*=8.2, 1.1 Hz, 1H), 7.31～7.23 (m, 2H), 7.18 (tdd, *J*=7.1, 2.0, 1.1 Hz, 1H), 6.73 (dd, *J*=3.4, 1.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.1, 147.2, 140.1, 136.0, 129.2, 127.7, 126.1,

123.1, 122.8, 121.1, 121.1, 112.2, 104.7; HRMS (ESI-TOF) calcd for $C_{13}H_{10}ClN_2$ $[M+H]^+$ 229.0527, found 229.0528.

1-(Pyridin-2-yl)-1*H*-indole (**3j**):[14] 73% yield as a pale yellow oil. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.58 (ddd, J=4.9, 1.9, 0.9 Hz, 1H), 8.23 (dd, *J*=8.3, 1.0 Hz, 1H), 7.86～7.72 (m, 2H), 7.69 (dt, *J*=7.7, 1.0 Hz, 1H), 7.50 (d, *J*=8.2 Hz, 1H), 7.32 (ddd, *J*=8.3, 7.1, 1.3 Hz, 1H), 7.25～7.20 (m, 1H), 7.17 (ddd, *J*=7.4, 4.9, 0.9 Hz, 1H), 6.74 (dd, *J*=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.5, 149.0, 138.5, 135.1, 130.5, 126.1, 123.2, 121.3, 121.2, 120.1, 114.7, 113.0, 105.6; HRMS (ESI-TOF) calcd for $C_{13}H_{11}N_2$ $[M+H]^+$ 195.0917, found 195.0915.

1-(Pyrimidin-2-yl)-1*H*-indole (**3k**):[14] 42% yield as a pale yellow oil. R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.74 (dd, *J*=4.8, 1.7 Hz, 1H), 8.62 (dd, *J*=8.6, 1.3 Hz, 2H), 7.56 (dd, *J*=7.8, 1.6 Hz, 1H), 7.28 (ddd, *J*=8.6, 5.5, 1.8 Hz, 2H), 7.15 (ddt, *J*=8.5, 7.3, 1.2 Hz, 1H), 6.95 (s, 1H), 6.63 (dd, *J*=7.7, 4.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.2, 157.9, 135.5, 131.4, 125.9, 123.7, 122.2, 120.9, 116.3, 116.2, 107.0; HRMS (ESI-TOF) calcd for $C_{12}H_{10}N_3$ $[M+H]^+$ 196.0869, found 196.0871.

6-Methyl-1-(5-methylpyridin-2-yl)-1*H*-indole (**3l**): 66% yield as a colorless oil. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.29 (d, *J*=2.2 Hz, 1H), 7.84 (s, 1H), 7.51 (dd, *J*=5.7, 2.8 Hz, 2H), 7.44 (d, *J*=8.0 Hz, 1H), 7.27 (d, *J*=8.2 Hz, 1H), 6.97 (dd, *J*=8.0, 1.3 Hz, 1H), 6.55 (dd, *J*=3.5, 0.8 Hz, 1H), 2.41 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.5, 149.0, 139.1, 135.5, 132.8, 129.6, 128.1, 125.6, 122.8, 120.7, 114.5, 112.6, 104.9, 22.1, 17.9; HRMS (ESI-TOF) calcd for $C_{15}H_{15}N_2$ $[M+H]^+$ 223.1230, found 223.1233.

5-Methyl-1-(5-methylpyridin-2-yl)-1*H*-indole (**3m**):[14] 62% yield as a white solid. m.p. 108～110 ℃; R_f=0.7 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.38 (d, *J*=2.3 Hz, 1H), 8.02 (d, *J*=8.5 Hz, 1H), 7.68 (d, *J*=3.4 Hz, 1H), 7.61 (dd, *J*=8.4, 2.3 Hz, 1H), 7.50 (d, *J*=1.3 Hz, 1H), 7.33 (d, *J*=8.2 Hz, 1H), 7.12 (dd, *J*=8.4, 1.6 Hz, 1H), 6.63 (d, *J*=3.4 Hz, 1H), 2.48 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.4, 148.9, 139.2, 133.4, 130.6, 130.4, 129.5, 126.2, 124.5, 120.9, 114.2, 112.4, 104.8, 21.4, 17.9; HRMS (ESI-TOF) calcd for $C_{15}H_{15}N_2$ $[M+H]^+$ 223.1230, found 223.1232.

6-Methyl-1-(pyrimidin-2-yl)-1*H*-indole (**3n**):[14] 48% yield as a white solid. m.p. 111～113 ℃; R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.70 (d, *J*=4.8 Hz, 2H), 8.64 (d, *J*=1.5 Hz, 1H), 8.21 (d, *J*=3.7 Hz, 1H), 7.51 (d, *J*=7.9 Hz, 1H), 7.09 (dd, *J*=8.0, 1.4 Hz, 1H), 7.03 (t, *J*=4.8 Hz, 1H), 6.66 (d, *J*=3.6 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.2, 157.9, 135.8, 133.6, 129.2, 125.3, 123.7, 120.5, 116.4, 116.0, 106.9, 22.2; HRMS (ESI-TOF) calcd for $C_{13}H_{12}N_3$ $[M+H]^+$ 210.1026, found 210.1025.

5-Methyl-1-(pyrimidin-2-yl)-1*H*-indole (**3o**):[14] 48% yield as a pale yellow solid. m.p. 89～91 ℃; R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.71～8.63 (m, 3H), 8.23 (d, *J*=3.6 Hz, 1H), 7.42 (d, *J*=1.6 Hz, 1H), 7.17 (dd, *J*=8.5, 1.7 Hz, 1H), 7.01 (t, *J*=4.8 Hz, 1H), 6.63 (d, *J*=3.6 Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.2, 157.9, 133.7, 131.6, 131.6, 125.9, 125.1, 120.8, 116.0, 106.7, 21.5; HRMS (ESI-TOF) calcd for $C_{13}H_{12}N_3$ $[M+H]^+$ 210.1026, found 210.1024.

6-Methyl-1-(pyridin-2-yl)-1*H*-indole (**3p**):[14] 65% yield as a pale yellow oil. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.58 (dd, *J*=4.9, 1.1 Hz, 1H), 8.02 (s, 1H), 7.85～7.80 (m, 1H), 7.65 (d, *J*=3.5 Hz, 1H), 7.58 (d, *J*=8.0 Hz, 1H), 7.47 (d, *J*=8.2 Hz, 1H), 7.17 (ddd, *J*=7.3, 4.9, 0.9 Hz, 1H), 7.05 (dd, *J*=8.1, 1.4 Hz, 1H), 6.67 (dd, *J*=3.5, 0.9 Hz, 1H), 2.52 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.7, 149.0, 138.5, 135.5, 133.1, 128.3, 125.5, 123.0, 120.7, 120.0, 114.8, 113.0, 105.5, 22.1; HRMS (ESI-TOF) calcd for $C_{14}H_{13}N_2$ $[M+H]^+$ 209.1073, found 209.1072.

6-Methoxy-1-(pyridin-2-yl)-1*H*-indole (**3q**):[14] 66% yield as a colorless oil. R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.55 (dd, *J*=4.9, 1.1 Hz, 1H), 8.19 (dd, *J*=9.1, 2.4 Hz, 1H), 7.78 (d, *J*=7.7 Hz, 1H),, 7.70 (d, *J*=3.5 Hz, 1H), 7.44 (d, *J*=9.2 Hz, 1H), 7.12 (d, *J*=2.3 Hz, 2H), 6.96 (dd, *J*=9.1, 2.2 Hz, 1H), 6.65 (d, *J*=3.4 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 155.1, 152.6, 148.9, 138.4, 131.2, 130.3, 126.3, 119.8, 114.2, 114.0, 112.8, 105.4, 103.0, 55.8; HRMS (ESI-TOF) calcd for $C_{14}H_{13}N_2O$ $[M+H]^+$ 225.1022, found 225.1022.

6-Fluoro-1-(pyridin-2-yl)-1*H*-indole/4-fluoro-1-(pyridin-2-yl)-1*H*-indole (**3r**):[14] 66% yield as a pale yellow oil. R_f=0.5 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.57 (td, *J*=5.1, 1.9 Hz, 2H), 8.08 (dt, *J*=10.8, 2.3 Hz, 1H), 7.99 (d, *J*=8.3 Hz, 1H), 7.82 (qd, *J*=7.5, 1.7 Hz, 2H), 7.67 (dd, *J*=8.9, 3.5 Hz, 2H), 7.56 (dd, *J*=8.6, 5.5 Hz, 1H), 7.52～7.47 (m, 1H), 7.46～7.42 (m, 1H), 7.25～7.15 (m, 3H), 6.95 (td, *J*=9.0, 2.4 Hz, 1H), 6.87 (dd, *J*=10.0, 7.9 Hz, 1H), 6.82 (d, *J*=3.5 Hz, 1H), 6.69 (dd, *J*=3.4, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 161.5, 159.6, 157.2, 155.3, 152.5, 152.3, 149.1, 149.0, 138.6, 137.6, 137.5, 135.3, 135.2, 126.8, 126.1, 126.1, 126.0, 123.8, 123.7, 121.6, 121.5, 120.6, 120.3, 119.5, 119.3, 114.8, 114.1, 110.0, 109.8, 109.2, 109.2, 106.3, 106.2, 105.7, 101.3, 100.7, 100.5; HRMS (ESI-TOF) calcd for $C_{13}H_{10}FN_2$ $[M+H]^+$ 213.0823, found 213.0823.

6-Chloro-1-(pyridin-2-yl)-1*H*-indole (**3s**):[14] 62% yield as a white solid. m.p. 110～112 ℃; R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.59 (ddd, *J*=4.8, 1.9, 0.9 Hz, 1H), 8.33 (d, *J*=1.8 Hz, 1H), 7.84 (ddd, *J*=8.2, 7.4, 1.9 Hz, 1H), 7.67 (d, *J*=3.5 Hz, 1H), 7.56 (d, *J*=8.4 Hz, 1H), 7.45 (d, *J*=8.2 Hz, 1H), 7.22～7.16 (m, 2H), 6.69 (dd, *J*=3.5, 0.9 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ: 152.3, 149.0, 138.7, 135.5, 129.2, 128.9, 126.5, 122.0, 121.8, 120.5, 114.4, 113.6, 105.6; HRMS (ESI-TOF) calcd for $C_{13}H_{10}ClN_2$ $[M+H]^+$ 229.0527, found 229.0527.

6-Bromo-1-(pyridin-2-yl)-1*H*-indole (**3t**):[14] 57% yield as a pale yellow solid foam. R_f=0.6 [*V*(PE)∶*V*(EA)=5∶1]; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.58 (dd, *J*=5.0, 1.8 Hz, 1H), 8.48 (d, *J*=1.7 Hz, 1H), 7.83 (td, *J*=7.8, 1.9 Hz, 1H),

7.65 (d, J=3.5 Hz, 1H), 7.51 (d, J=8.4 Hz, 1H), 7.43 (d, J=8.2 Hz, 1H), 7.32 (dd, J=8.4, 1.7 Hz, 1H), 7.19 (dd, J=7.4, 4.8 Hz, 1H), 6.68 (d, J=3.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.2, 149.1, 138.7, 135.9, 129.2, 126.4, 124.6, 122.2, 120.5, 116.9, 116.5, 114.5, 105.7; HRMS (ESI-TOF) calcd for $C_{13}H_{10}BrN_2$ $[M+H]^+$ 273.0022, found 273.0022.

1-(Pyridin-2-yl)-6-(trifluoromethyl)-1*H*-indole (**3u**):[14] 33% yield as a white solid. m.p. 109～111 ℃; R_f=0.6 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.66～8.56 (m, 2H), 7.87 (ddd, J=8.2, 7.4, 1.9 Hz, 1H), 7.82 (d, J=3.5 Hz, 1H), 7.74 (dt, J=8.4, 0.8 Hz, 1H), 7.46 (ddd, J=13.8, 8.3, 1.3 Hz, 2H), 7.24 (ddd, J=7.4, 4.9 Hz, 1H), 6.77 (dd, J=3.5, 0.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.1, 149.2, 138.9, 134.3, 132.8, 128.4, 126.3, 125.4, 125.2, 124.2, 121.4, 120.8, 118.1, 118.1, 118.1, 118.0, 114.7, 111.1, 111.1, 105.6; HRMS (ESI-TOF) calcd for $C_{14}H_{10}F_3N_2$ $[M+H]^+$ 263.0791, found 263.0793

5-Methyl-1-(pyridin-2-yl)-1*H*-indole (**3v**):[14] 66% yield as a pale yellow oil. R_f=0.6 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.56 (dd, J=4.9, 1.8 Hz, 1H), 8.10 (d, J=8.4 Hz, 1H), 7.83～7.78 (m, 1H), 7.71 (d, J=3.4 Hz, 1H), 7.51～7.45 (m, 2H), 7.17～7.12 (m, 2H), 6.65 (d, J=3.4 Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.6, 148.9, 138.5, 138.5, 133.4, 130.8, 130.7, 126.0, 124.7, 120.9, 119.9, 114.3, 112.7, 105.3, 21.4; HRMS (ESI-TOF) calcd for $C_{14}H_{12}N_2Na$ $[M+H]^+$ 231.0893, found 231.0896.

5-(*tert*-Butyl)-1-(pyridin-2-yl)-1*H*-indole (**3w**): 75% yield as a pale yellow oil. R_f=0.6 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.56 (dd, J=5.3, 1.7 Hz, 1H), 8.13 (d, J=8.8 Hz, 1H), 7.83～7.79 (m, 1H), 7.73 (d, J=3.5 Hz, 1H), 7.67 (d, J=1.9 Hz, 1H), 7.50 (d, J=8.2 Hz, 1H), 7.39 (dd, J=8.8, 2.0 Hz, 1H), 7.15 (ddd, J=7.3, 4.9, 0.9 Hz, 1H), 6.69 (d, J=3.4 Hz, 1H), 1.42 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.6, 149.0, 144.3, 138.4, 133.2, 130.4, 126.0, 121.4, 119.9, 117.1, 114.3, 112.6, 105.8, 34.7, 31.9; HRMS (ESI-TOF) calcd for $C_{17}H_{19}N_2$ $[M+H]^+$ 251.1543, found 251.1544.

5-Methoxy-1-(pyridin-2-yl)-1*H*-indole (**3x**):[14] 38% yield as a pale yellow oil. R_f=0.4 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.55 (dd, J=4.9, 1.1 Hz, 1H), 8.17 (d, J=9.2 Hz, 1H), 7.82～7.77 (m, 1H), 7.70 (d, J=3.5 Hz, 1H), 7.46 (d, J=8.2 Hz, 1H), 7.13 (dd, J=12.6, 3.8 Hz, 2H), 6.95 (dd, J=9.0, 2.6 Hz, 1H), 6.64 (dd, J=3.4, 0.8 Hz, 1H), 3.88 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 155.1, 152.6, 148.9, 138.5, 131.2, 130.3, 126.4, 119.8, 114.2, 114.1, 112.8, 105.5, 103.0, 55.8; HRMS (ESI-TOF) calcd for $C_{14}H_{12}N_2ONa$ $[M+Na]^+$ 247.0842, found 247.0842.

5-Phenyl-1-(pyridin-2-yl)-1*H*-indole (**3y**): 50% yield as a yellow oil. R_f=0.6 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.60 (dd, J=4.9, 1.1 Hz, 1H), 8.29 (d, J=8.7 Hz, 1H), 7.89 (d, J=1.8 Hz, 1H), 7.86～7.82 (m, 1H), 7.77 (d, J=3.5 Hz, 1H), 7.69 (dd, J=8.2, 1.3 Hz, 2H), 7.56 (dd, J=8.7, 1.8 Hz, 1H), 7.53 (d, J=8.2 Hz, 1H), 7.47 (dd, J=8.5, 7.0 Hz, 2H), 7.36～7.32 (m, 1H), 7.19 (ddd, J=7.4, 4.9, 0.9 Hz, 1H), 6.78 (dd, J=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.5, 149.0, 142.2, 138.6, 134.8, 134.6, 131.0, 128.8, 127.4, 126.6, 122.9, 120.2, 119.6, 114.5, 113.4, 106.0; HRMS (ESI-TOF) calcd for $C_{19}H_{15}N_2$ $[M+H]^+$ 271.1230, found 271.1230.

5-Fluoro-1-(pyridin-2-yl)-1*H*-indole (**3z**):[14] 35% yield as a white solid. m.p. 62～64 ℃; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.56 (dd, J=4.9, 1.1 Hz, 1H), 8.23 (dd, J=9.1, 4.6 Hz, 1H), 7.83 (ddd, J=8.4, 7.3, 2.0 Hz, 1H), 7.73 (d, J=3.5 Hz, 1H), 7.45 (d, J=8.7 Hz, 1H), 7.29 (dd, J=9.2, 2.6 Hz, 1H), 7.18 (ddd, J=7.4, 4.8, 0.9 Hz, 1H), 7.03 (td, J=9.1, 2.6 Hz, 1H), 6.67 (dd, J=3.4, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 159.6, 157.7, 152.4, 148.9, 138.7, 131.8, 131.0, 127.4, 120.3, 114.4, 114.3, 114.3, 111.4, 111.2, 106.22, 106.0, 105.6, 105.6; HRMS (ESI-TOF) calcd for $C_{13}H_{10}FN_2$ $[M+H]^+$ 213.0823, found 213.0822.

5-Chloro-1-(pyridin-2-yl)-1*H*-indole (**3aa**):[14] 47% yield as a white solid foam. R_f=0.6 [V(PE)∶V(EA)=5∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.57 (dd, J=4.9, 1.1 Hz, 1H), 8.20 (d, J=8.8 Hz, 1H), 7.85～7.80 (m, 1H), 7.71 (d, J=3.5 Hz, 1H), 7.62 (d, J=2.1 Hz, 1H), 7.44 (dd, J=8.2, 0.9 Hz, 1H), 7.24 (dd, J=8.9, 2.1 Hz, 1H), 7.19 (dd, J=7.5, 4.9 Hz, 1H), 6.65 (dd, J=3.5, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.3, 149.0, 138.6, 133.6, 131.5, 127.1, 126.9, 123.4, 120.5, 120.4, 114.5, 114.4, 105.1; HRMS (ESI-TOF) calcd for $C_{13}H_{10}ClN_2$ $[M+H]^+$ 229.0527, found 229.0528.

1-(Pyridin-2-yl)-1*H*-benzo[*f*]indole (**3ab**): 47% yield as a white solid. m.p. 56～58 ℃; R_f=0.7 [V(PE)∶V(EA)=6∶1]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.74 (s, 1H), 8.63 (ddd, J=4.9, 2.0, 0.8 Hz, 1H), 8.14 (s, 1H), 8.01～7.94 (m, 3H), 7.85 (td, J=7.2, 1.3 Hz, 1H), 7.62～7.56 (m, 1H), 7.45～7.38 (m, 2H), 7.19～7.15 (m, 1H), 6.86 (dd, J=3.7, 0.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.7, 149.0, 138.6, 135.2, 131.6, 131.1, 129.6, 129.5, 128.3, 128.0, 124.3, 123.6, 119.6, 118.7, 114.0, 109.4, 105.5; HRMS (ESI-TOF) calcd for $C_{17}H_{12}N_2Na$ $[M+Na]^+$ 267.0893, found 267.0891.

Supporting Information Screening of reaction conditions, synthesis of substrate, characterization of products, copies of ^{1}H NMR and ^{13}C NMR. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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