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# Dual-regioselective direct C(sp<sup>2</sup>)-arylation of unprotected $\beta$ -enamino esters with 2-indolylmethanols catalyzed by Brønsted acid

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An efficient dual-regioselective strategy has been developed for the direct C(sp<sup>2</sup>)-arylation of  $\beta$ -enamino esters with 2-indolylmethanols, employing diphenyl phosphate as the catalyst without the need for amino group protection. 48 structurally diverse indole-enamino ester hybrids were synthesized in moderate to excellent yields (up to 98%). Notably, this approach effectively suppresses the competitive *N*-arylation byproduct formation commonly encountered in traditional approaches and represents the successful precise integration of  $\beta$ -enamino esters and indoles, offering a modular and efficient route to complex heterocyclic architectures with potential bioactivities.

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## 1. Introduction

$\beta$ -Enamino esters are structurally unique scaffolds that have attracted significant attention in drug discovery and bioactive molecule development (Fig. 1).<sup>1</sup> As versatile *C,N*-dinucleophilic synthons, they are widely used to construct nitrogen-containing heterocycles with high application value.<sup>2</sup> Recent advances have also enabled regioselective  $\beta$ -C–H functionalization (Scheme 1a), including trifluoromethylation,<sup>3</sup> trifluoromethylthiolation,<sup>4</sup> arylation,<sup>5</sup> thiolation,<sup>6</sup> phosphorylation,<sup>7</sup> and  $\beta$ -ketoalkylation.<sup>8</sup> Among these transformations, direct C(sp<sup>2</sup>)-H arylation of  $\beta$ -enamino esters has emerged as a key area of research. It usually utilizes electron-rich arenes,<sup>9</sup> *o*-silylaryl triflates,<sup>10</sup> aryl boronic esters,<sup>11</sup> and 3-aminoindazoles<sup>12</sup> as arylating agents. Nevertheless, existing methods often face some limitations, including contamination from transition metals and the requirements for pre-protecting amino groups. Consequently, the development of milder reaction conditions for the direct C(sp<sup>2</sup>)-arylation of  $\beta$ -enamino esters remains a significant challenge in synthetic chemistry.

Indolylmethanols have proven to be versatile reactants for the synthesis of indole derivatives, as they can easily be converted into delocalized cation intermediates with carbocation resonance structures mediated by Brønsted or Lewis acids, followed by the nucleophilic substitution reactions<sup>13</sup> and [3 + *n*] cycloadditions,<sup>14</sup> affording benzylic site functionalized, C3-functionalized or multicyclic indole derivatives. Shi reported the reaction of 2-indolylmethanol with cyclic enaminones to

access a series of C3-functionalized indole derivatives (Scheme 1b).<sup>15</sup> Zhen demonstrated a highly efficient, AlCl<sub>3</sub>-catalyzed protocol enabling regioselective reactions between 2-indolylmethanols and enamides (Scheme 1c).<sup>16</sup> However, these strategies still require protecting groups to manage the high reactivity of primary amines, and suppressing *N*-arylation side reactions remains a key challenge due to uncontrolled cascade processes arising from their inherent nucleophilicity. As a result, we envisioned that 2-indolylmethanols could be used as arylating agents in the C(sp<sup>2</sup>)-arylation with  $\beta$ -enamino esters. In continuation of our research on organocatalytic synthetic strategies,<sup>17</sup> herein we report a Brønsted acid-catalyzed direct C(sp<sup>2</sup>)-arylation of  $\beta$ -enamino esters (including 4-aminocoumarins and acyclic derivatives) with 2-indolylmethanols (Scheme 1d). This method efficiently yields a series of diverse indole-enamine hybrids with good to excellent yields. The procedure is dual regioselective, operationally simple, avoids the need for amino group protection, and ensures the absence of *N*-arylation byproducts.

## 2. Results and discussion

Initially, we selected 2-indolylmethanol **1a** and 4-amino-coumarin **2a** (cyclic  $\beta$ -enamino ester) as model substrates in 1,2-dichloroethane (DCE) at room temperature (Table 1). Several Lewis acids were tested (entries 1–6), and Indium triflate [In(OTf)<sub>3</sub>] proved most effective, affording the desired product **3a** in 76% yield (entry 5). The *N*-arylation byproduct was not observed. Considering the potential risks of metal residues and efficiency limitations of metal catalysts, we turned our attention to Brønsted acids (entries 7–11). Notably, diphenyl phosphate (**C1**) delivered **3a** in 83% yield (entry 7), while other Brønsted acids including phenylphosphoric acid (**C2**), *p*-toluenesulfonic

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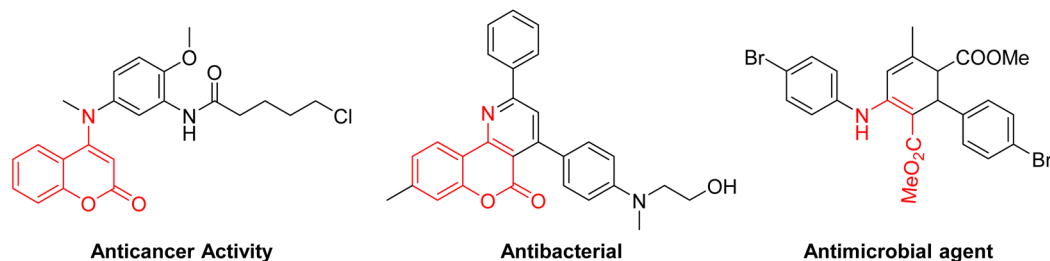



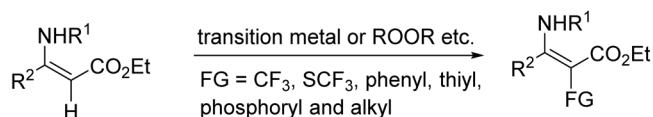
Fig. 1 Bioactive compounds including  $\beta$ -enamino ester scaffolds.

acid (TsOH), trifluoroacetic acid (TFA), and trifluoromethanesulfonic acid (TfOH) also provided moderate to good yields (entries 8–11, 57–81%). In order to further improve the reaction, different solvents were also investigated (entries 12–18). The halogenated solvents such as dichloromethane (DCM, entry 12) and chloroform (entry 13) could afford the desired product **3a** in 88% and 85% yield, respectively. In contrast, other polar solvents such as tetrahydrofuran (THF), ethyl acetate, acetone and dimethylformamide (DMF) exhibited limited efficiency (<20%, entries 14–17). Toluene resulted in

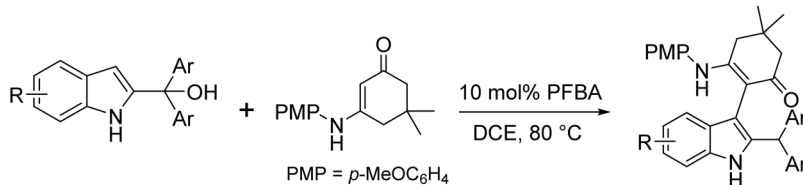
only a moderate yield (65%, entry 18). If the reaction temperature was increased to 40 °C, the reaction time could sharply shortened to 30 minutes and the yield reached 92% (entry 19). Remarkably, if the catalyst loading was reduced from 20 mol% to 10 mol%, the yield could remain (entry 20, 92%).

With the optimal reaction conditions established, the scope of this reaction was then examined. The results were summarized in Table 2. A wide range of 2-indolylmethanols **1** could smoothly react with 4-aminocoumarin **2a**, delivering the regio-selective products **3**. In detail, the influence of substituents on

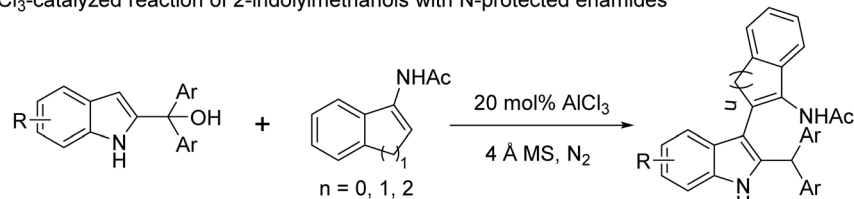
(a) Direct  $\beta$ -C-H functionalization of  $\beta$ -enamino esters<sup>3–8</sup>



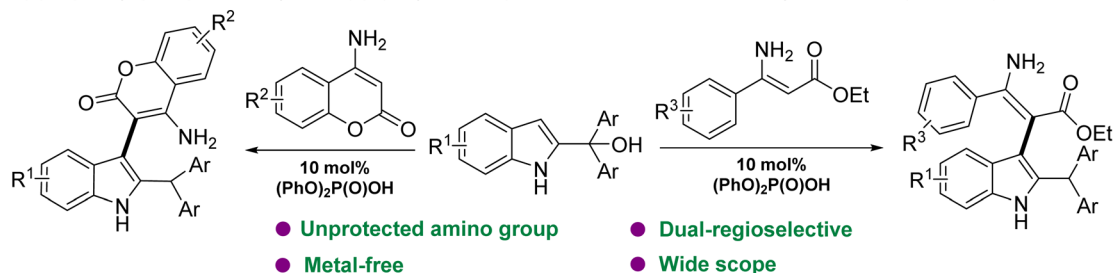
(b) Pentafluorobenzoic acid-catalyzed reaction of 2-indolylmethanols with N-protected enamines<sup>15</sup>



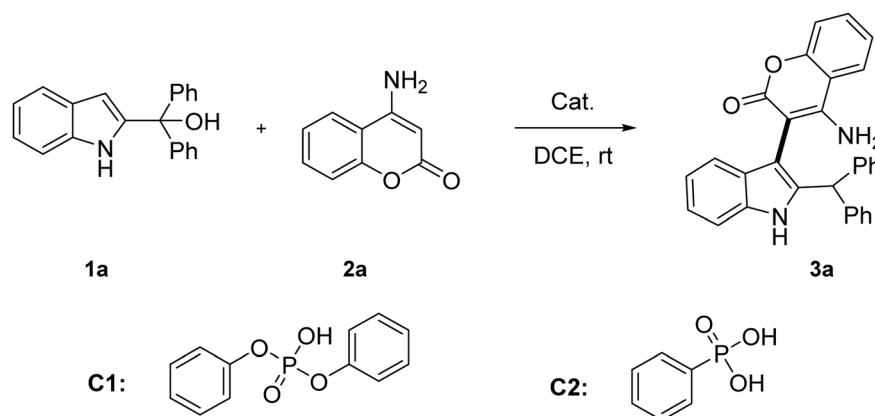
(c)  $\text{AlCl}_3$ -catalyzed reaction of 2-indolylmethanols with N-protected enamides<sup>16</sup>



(d) Diphenyl phosphate catalyzed  $\text{C}(\text{sp}^2)$ -arylation of  $\beta$ -enamino esters with 2-indolylmethanols (*this work*)



Scheme 1 Synthetic strategies using  $\beta$ -enamino derivatives as substrates.

Table 1 Optimization of the reaction conditions<sup>a</sup>

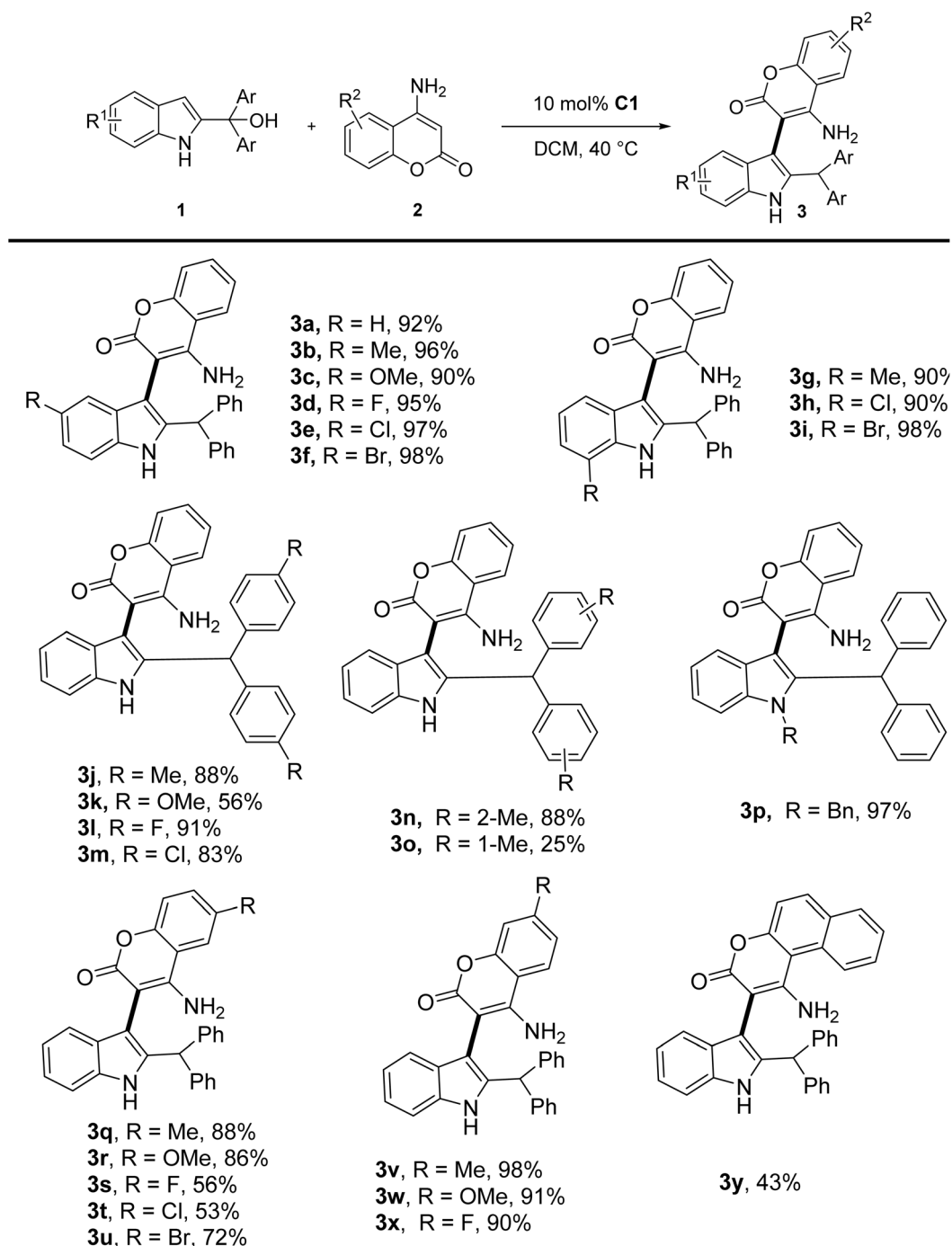
Entry	Catalyst	Solvent	Yield <sup>b</sup> (%)
1	ZnCl <sub>2</sub>	DCE	42
2	FeCl <sub>3</sub>	DCE	63
3	AlCl <sub>3</sub>	DCE	66
4	BF <sub>3</sub> ·Et <sub>2</sub> O	DCE	70
5	In(OTf) <sub>3</sub>	DCE	76
6	Cu(OTf) <sub>2</sub>	DCE	70
7	<b>C1</b>	DCE	83
8	<b>C2</b>	DCE	57
9	TsOH	DCE	75
10	TFA	DCE	81
11	TfOH	DCE	78
12	<b>C1</b>	DCM	88
13	<b>C1</b>	CHCl <sub>3</sub>	85
14	<b>C1</b>	THF	10
15	<b>C1</b>	EtOAc	20
16	<b>C1</b>	Acetone	17
17	<b>C1</b>	DMF	13
18	<b>C1</b>	Toluene	62
19 <sup>c,e</sup>	<b>C1</b>	DCM	92
20 <sup>c,d,e</sup>	<b>C1</b>	DCM	92

<sup>a</sup> Unless otherwise noted, all reactions were conducted with **1a** (0.2 mmol), **2a** (0.24 mmol, 1.2 equiv.) and **cat.** (0.04 mmol, 20 mol%) in the solvent (2.0 mL) stirred at room temperature for 6 h. <sup>b</sup> Isolated yield. <sup>c</sup> At 40 °C. <sup>d</sup> 10 mol% **C1** (diphenyl phosphate) was used. <sup>e</sup> Reaction time was shortened to 30 min.

the indole ring of 2-indolylmethanols was examined. The reaction efficiency was largely insensitive to the electronic nature of the indole ring. 2-Indolylmethanols bearing either electron-donating or electron-withdrawing groups at C5 and C7 positions of the indole moiety generally afforded the corresponding products in excellent yields (90–98%, **3b–3i**). Substituent effects on the benzyl ring were also investigated. *Para*-Substituted derivatives bearing methyl, fluoro, or chloro groups could maintain the yields above 83% (**3j**, **3l–m**), while a strongly electron-donating *para*-methoxy group significantly reduced the yield to 56% (**3k**) probably due to less dehydration to form the carbocation intermediate. Steric effects were evaluated through *meta*- and *ortho*-methyl substitutions. While the *meta*-methyl analog (**3m**) maintained an 88% yield, the *ortho*-methyl derivative (**3o**) showed dramatically diminished reactivity (25%

yield). Notably, *N*-benzyl group protection of the indole nitrogen atom still enabled efficient formation of **3p** in 97% yield. To further examine the generality of the reaction, the scope of the reaction with respect to 4-aminocoumarin derivatives were also investigated. The electronic nature of the substituents of 4-aminocoumarins had noticeable effects on the outcome of the reaction. Substrates with electron-donating group substituent at C6 position afforded products with better yields than that with electron-withdrawing groups (**3q–3r** vs. **3s–3u**). Intriguingly, substituent at the C7 position exhibited different effects. Both the substrates with electron-donating group and electron-withdrawing group delivered the products in excellent yields (**3v–3x**). For the naphthalene-derived substrate, the yield of product **3y** decreased to 43%, likely due to its unique aromatic system architecture. The distinct difference in electronic



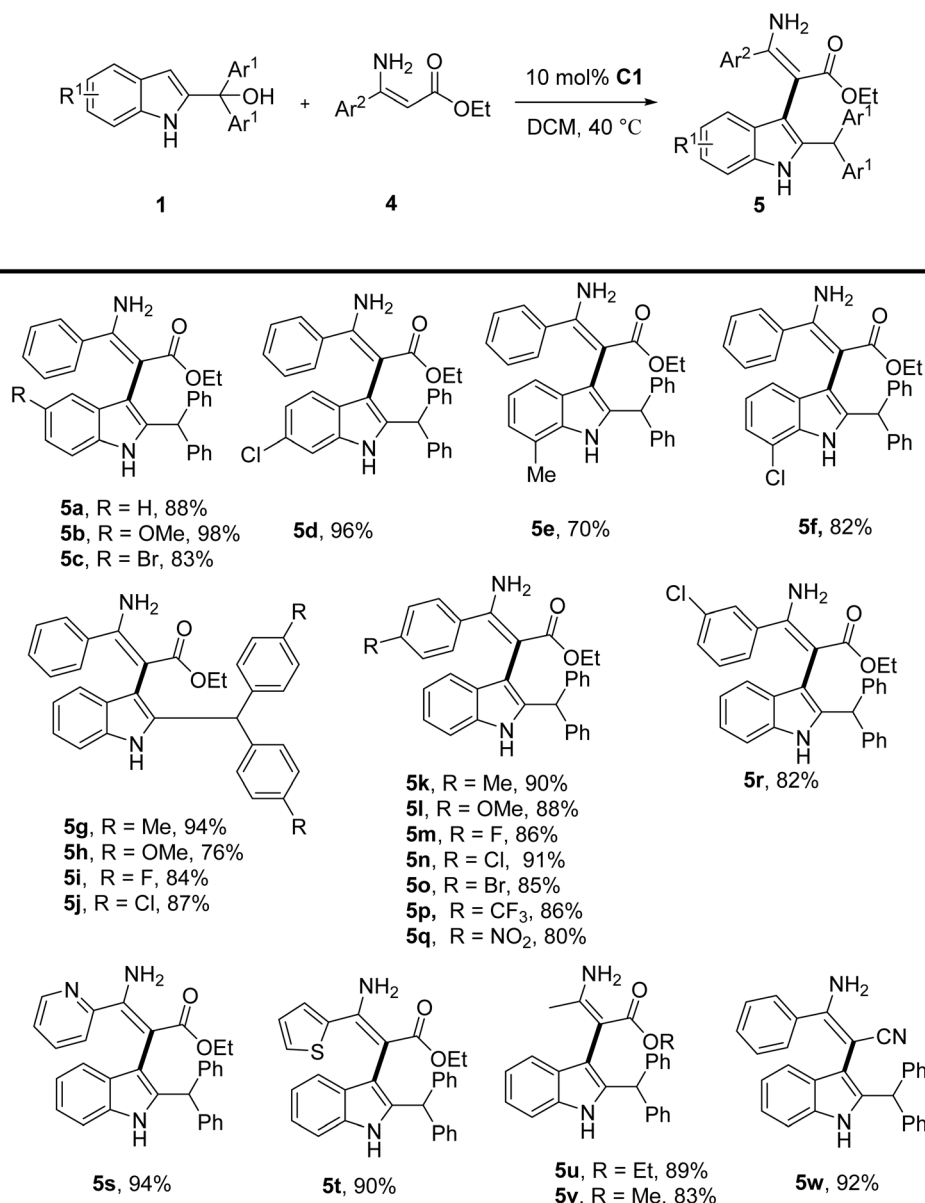
Table 2 Scope of the reaction between 4-aminocoumarins and 2-indolylmethanols<sup>a</sup>

<sup>a</sup> Reaction conditions: 1 (0.2 mmol), 2a (0.24 mmol, 1.2 equiv.), C1 (0.02 mmol, 10 mol%) in DCM (2.0 mL) stirred at 40 °C for 30 min.

sensitivity between C6 and C7 substituents demonstrated the position-dependent nature of electronic effects in this system.

Since 4-aminocoumarins can be viewed as cyclic  $\beta$ -enamino esters, we next explored acyclic  $\beta$ -enamino esters as substrates to further broaden the reaction scope. To our delight, under the

same conditions, the reaction of 2-indolylmethanols 1 with the acyclic  $\beta$ -enamino esters 4 also proceeded smoothly, leading to the corresponding product 5 in generally good to excellent yields (Table 3). For 2-indolylmethanols, regardless of the electronic properties and positions of the substituent on the

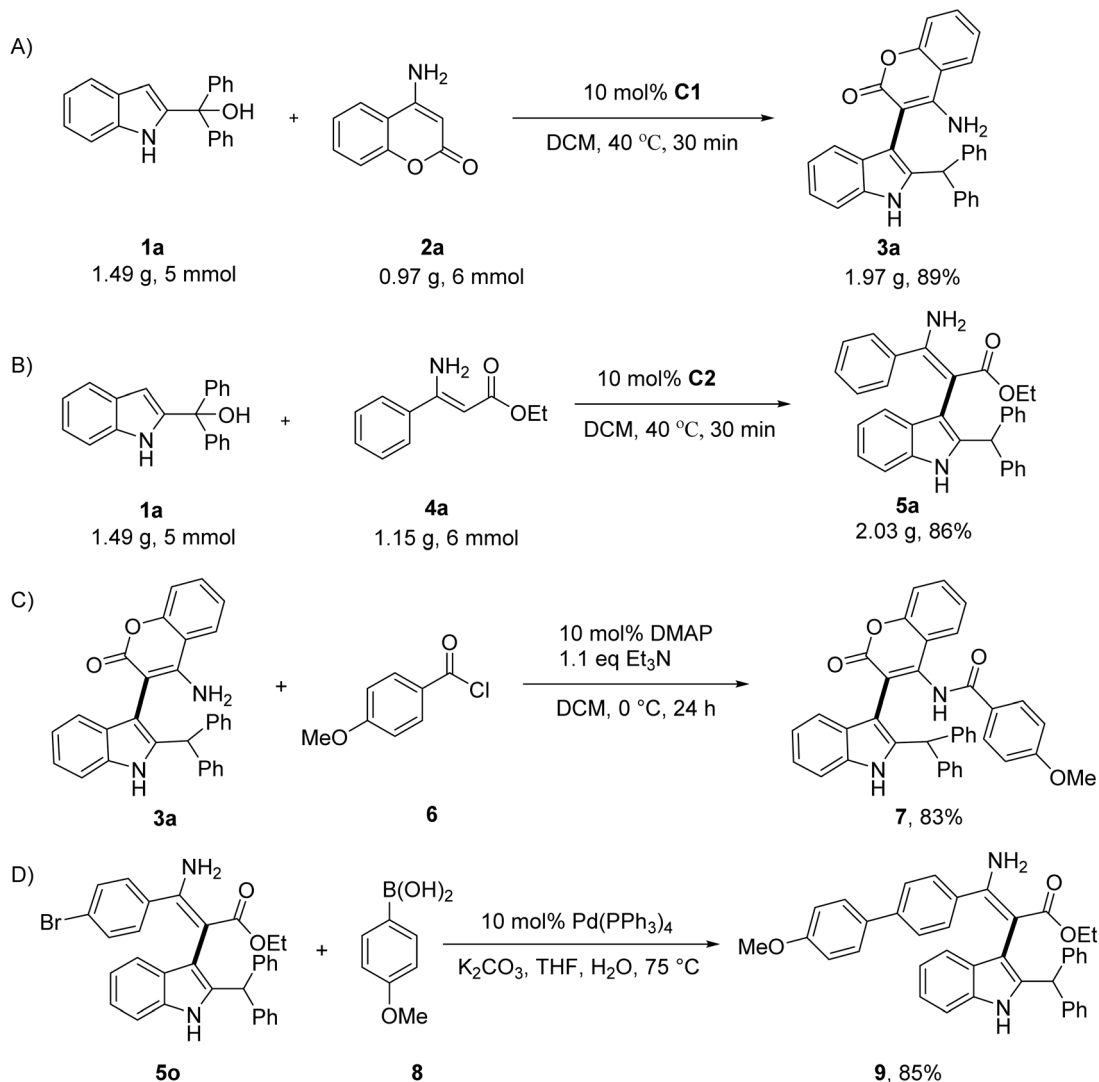
Table 3 Scope of the reaction between acyclic  $\beta$ -enamino esters and 2-indolymethanols<sup>a</sup>

<sup>a</sup> Reaction conditions: all reactions were carried out using **1** (0.2 mmol), **4a** (0.24 mmol, 1.2 equiv.), **C1** (0.02 mmol, 10 mol%) in DCM (2.0 mL) stirred at 40 °C for 30 min.

indole ring ( $R^1$ ) (**5a–5f**) or benzyl aromatic ring (Ar) (**5g–5j**), the reaction pattern was similar to that with 4-aminocoumarins. It demonstrated the wide applicability of this regioselective reaction. For acyclic  $\beta$ -enamino esters, the varied substituents on different positions of  $\beta$ -phenyl ring afforded the desired product in 80–91% yield (**5k–5r**). Even strongly electron-withdrawing groups such as  $-\text{CF}_3$  and  $-\text{NO}_2$  were well tolerated (**5p** and **5q**, 86% and 80% yield). Other aromatic heterocyclic substituents such as pyridyl and thiophenyl also had

good performance, giving the corresponding product (**5s** and **5t**) in 94% and 90% yield, respectively. If the alkyl group (methyl) was used instead of phenyl group, the reaction yield still remained high (**5u–5v**). Different ester moiety proved good efficiency as well (**5v**, 83%). Additionally, the successful replacement of the ester group with a cyano ( $-\text{CN}$ ) group in the acyclic  $\beta$ -enamino system could result in nitrile derivative **5w** in 92% yield. The results presented the versatility of the catalytic system in accommodating both cyclic and acyclic  $\beta$ -enamino





Scheme 2 Gram-scale reactions and synthetic transformations.

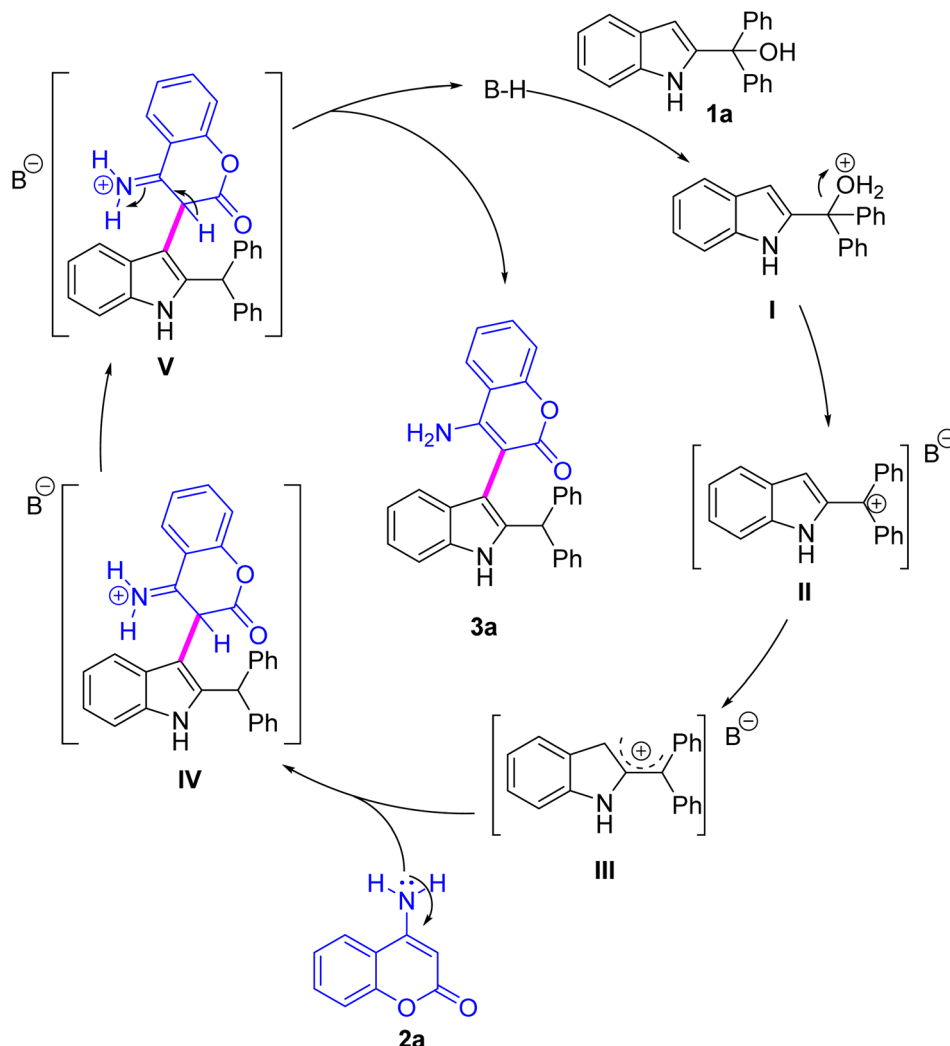
ester frameworks, significantly expanding the synthetic utility of this methodology for constructing diverse enamine-indole hybrid architectures.

To demonstrate the practical utility of this reaction, gram-scale syntheses and chemical transformations were conducted. As displayed in Scheme 2A and 2B, gram-scale reactions were performed with 5 mmol of **1a** with 6 mmol of **2a** and **4a**, affording the corresponding products **3a** and **5a** without significant losses of yield compared with the small-scale reaction (89% and 86% yield, respectively). Several transformations were also performed (Scheme 2C and 2D). Treatment of **3a** with *p*-methoxybenzoyl chloride under mild acylation conditions converted the primary amine to amide **7** in 83% yield (Scheme 2C). Suzuki–Miyaura coupling of **5a** with 4-methoxyphenylboronic acid smoothly delivered biaryl product **9** in 85% yield (Scheme 2D). These transformations demonstrated

compatibility of the products and highlighted their value as versatile intermediates for complex molecule synthesis.

Based on experimental evidence and literature precedents,<sup>13</sup> we propose a plausible Brønsted acid-catalyzed mechanism for the reaction of 2-indolylmethanol with  $\beta$ -enamino ester (Scheme 3). The catalytic cycle begins with Brønsted acid protonation of 2-indolylmethanol **1a**, generating an oxonium intermediate (**I**). Subsequent dehydration forms the highly reactive carbocation intermediate (**II**), which undergoes extensive resonance stabilization to yield the electrophilic species (**III**). The  $\beta$ -enamino ester **2a** then undergoes nucleophilic attack at the electrophilic carbon of **III**, establishing the key C–C bond and forming iminium intermediate (**IV**). The inherent instability of **IV** promotes deprotonation at the activated site, simultaneously restoring the aromatic indole system and releasing the final product (**3a**). This step also regenerates the Brønsted acid catalyst, completing the catalytic cycle.





Scheme 3 Plausible mechanism of the reaction.

### 3. Conclusions

In summary, we have developed an efficient, dual-regioselective organocatalytic strategy for the direct C(sp<sup>2</sup>)-arylation of β-enamino esters (including 4-aminocoumarins and acyclic derivatives) with 2-indolylmethanols using diphenyl phosphate as the catalyst, without the need for amino group protection. This transformation exhibited broad substrate scope, accommodating a wide range of 2-indolylmethanols and β-enamino esters to deliver structurally diverse indole-enamine hybrids in good to excellent yields (48 examples, up to 98% yield). The protocol demonstrated excellent scalability in gram-scale reactions and offered versatile opportunities for further synthetic transformations, enhancing its practical utility. Notably, the reaction proceeded with high efficiency and complete dual regioselectivity, providing a robust and user-friendly method for accessing these valuable heterocyclic frameworks. Further investigations on asymmetric variants of this transformation are in progress.

### Conflicts of interest

There are no competing interest to declare.

### Data availability

The data supporting this article have been included as part of the SI. Supplementary information: experimental procedures and NMR spectra. See DOI: <https://doi.org/10.1039/d5ra05581d>.

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