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I_2/PhI(OAc)$_2$-assisted oxidative C–H amination protocols toward metal-free pragmatic synthesis of pyrrolo[2,3-*b*]indoles

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ABSTRACT

We report herein an I_2/PhI(OAc)$_2$ catalytic system for the pragmatic construction of C–N bonds through C–H/N–H oxidative coupling protocol. Divergent pyrrolo[2,3-*b*]indoles were efficiently prepared *via* I_2-catalyzed intramolecular C–H amination reactions from (*E*/*Z*)-2-indolylenamines under metal-free conditions. Various functional groups are tolerated under mild reaction conditions and the resulting pyrrolo[2,3-*b*]indoles were obtained with mostly good to excellent yields. It was interesting to observe that both the (*E*)- and (*Z*)-isomers of the starting materials were efficiently transformed into the targeted product. The I^+-mediated catalytic cycle was proposed based on mechanistic studies for this reaction.

Indoles are among the most privileged medicinal scaffolds that can be frequently found in natural products, as well as in various pharmaceutical candidates, dyes, and agrochemicals [1–3]. In particular, structurally diversified indolyl fused pyrrolines are biologically valuable natural alkaloids (Fig. 1). For instance, pyrrolo[2,3-*b*]indole derivatives such as pyrroindomycins **A** and **B** [4] were isolated from *Streptomyces rugosporus* in 1994 [5,6]. Recently, families of these compounds were found to behave potent antibiotic activities against bacterial pathogens, and in vitro activity against Gram-positive bacteria [7]. The 5,6-dihydroindolo[2,3-*b*]indoles also demonstrated excellent activities as Sirtuins inhibitors [8,9], or as growth inhibitors of Bacillus subtilis [10]. Due to their prominent pharmaceutical activities, pyrrolo[2,3-*b*]indole frameworks have always been the superior choice in medicinal studies, and their synthetic methodologies have also received extensive attention.

Recently, transition-metal-catalyzed annulations leading to fused aza-heteroaromatics have attracted particular attention. To date, the synthesis of indole-based fused polyheterocycles that were catalyzed by varied transitionmetals, such as Pd [8,11,12], Au [13], Cu/[O] [14], Fe [15] or Zn/NBS [16], were reported. Nevertheless, formidable limitations with the conventional cyclizations or cycloadditions still exist, mainly due to their reliance on specific active metal catalysts. As a supplement, metal-free protocols can offer quick but low-cost access to diverse molecular structures. Indeed, metal-free reactions have shown great potential for rapid production of complex aza-heterocyclic molecules in a single synthetic operation, which is of exceeding importance in the large-scale preparation of medicinal-related substances by avoiding metal pollution [17–20]. In 1996, an interesting heat-promoted stepwise Barton-Zard reaction was developed to deliver a pyrrolo[2,3-*b*]indole ring system [21]. Besides, stoichiometric amounts of hypervalent iodine reagent [22,23] or NIS [24] mediated amination of indole frameworks have also shown potential toward *N*-arylated and *N*-alkylated fused aza-heterocycles. These methods allowed the rapid construction of the indole skeletons by connecting N atoms on the side chain of the phenyl moieties. However, compared with the noticeable C3-functionalization, the C2-amination of indole is less explored. Some limited reports are available for intramolecular C2-amination of indole scaffolds. In this regard, Wang disclosed I_2-mediated oxidative coupling of intramolecular C–N bond formations to afford indole derivatives. Wang group disclosed an iodine-catalyzed intramolecular amination of tryptophan esters for the practical synthesis of pyrrolo[2,3-*b*]indoles (Scheme 1a) [25]. Sekar and co-workers developed I_2-mediated intramolecular C2 sulfonamidative cyclization of indoles to give indole fused tetracyclic compounds (Scheme 1b) [26]. While these methods should shed light to the synthesis of pyrrolo[2,3-*b*]indole derivatives, step-wise preparation of the starting materials, and/or pre-functionalization of an electron-withdrawing substituent at the certain position of indolyl precursors are often necessary.

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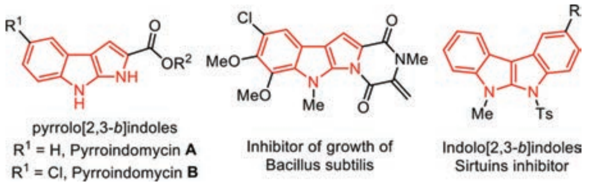
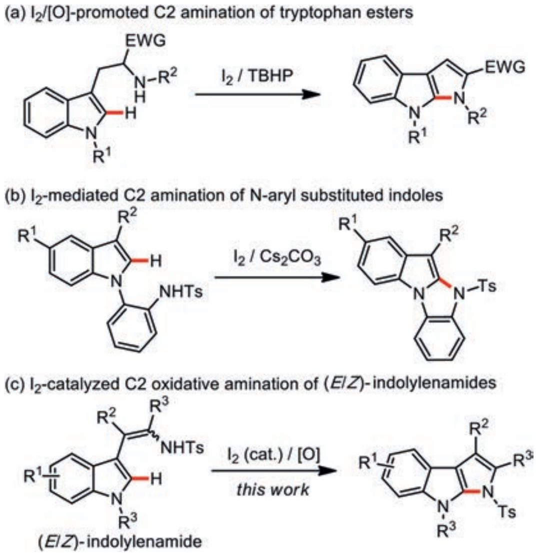


pyrrolo[2,3-*b*]indoles
R^1 = H, Pyrroindomycin **A**
R^1 = Cl, Pyrroindomycin **B**

Inhibitor of growth of Bacillus subtilis

Indolo[2,3-*b*]indoles Sirtuins inhibitor

Fig. 1. Selected biologically active pyrrolo[2,3-*b*]indole derivatives.



(a) I_2/[O]-promoted C2 amination of tryptophan esters

(b) I_2-mediated C2 amination of N-aryl substituted indoles

(c) I_2-catalyzed C2 oxidative amination of (*E*/*Z*)-indolylenamides

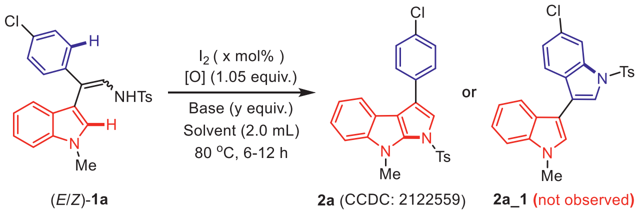
(*E*/*Z*)-indolylenamide

Scheme 1. I_2-mediated intramolecular C2-amination of 2-indolylenamides.

Our group previously reported a 1,3-insertion reaction of a Rh-carbenoid into the C(sp^2)-H bond of simple indoles [27], and subsequently sequential C–H oxidation to selectively produce (*E*/*Z*)-2-indolylenamides or (*Z*)-3-arylidene-2-oxindole imides [28]. As part of our interest in the fused polyheterocyclic alkaloids [29–33], we envisioned that an I_2-catalyzed C2-amination of C–H/N–H oxidative coupling should offer a pragmatic synthesis of structurally divergent pyrrolo[2,3-*b*]indoles (Scheme 1c). Herein, we report a highly efficient metal-free protocol to chemo- and regioselectively afford pyrrolo[2,3-*b*]indoles under very gentle conditions.

We sought to employ (*E*/*Z*)-mixture of 2-indolylenamide **1a** as a model substrate to investigate this C2-aminative cyclization reaction (Table 1). The optimization commenced with **1a** in the presence of iodine (5 mol%), oxidant [O], and Na_2HPO_4 (3.5 equiv.) in toluene (2.0 mL) at 80 °C. Indeed, the desired product **2a** could be formed when $Cu(OAc)_2$ (1.05 equiv.) was selected as the oxidant, albeit in quite a low yield (entry 1). The configuration of compound **2a** was determined by single-crystal X-ray diffraction analysis (CCDC: 2122559 (**2a**) in Supporting information for details). Product **2a** was formed in 29% yield when *t*BuOOH was added instead of $Cu(OAc)_2$ (entry 2). To our delight, improved yields were observed when BQ or TBHP was added, and a 62% yield of product **2a** was obtained in the presence of TBHP (entries 3 and 4). However, decreased efficiency was observed when I_2 loading was improved to 10 mol%, whereas a dramatically sluggish reaction was observed by reducing the amount of iodine to 2 mol%. These results indicated the crucial role of iodine species in this amidation protocol (entries 5 and 6). The reaction was optimistic for $PhI(OAc)_2$, which could afford the desired product **2a** in 68% yield (entries 7 and 8). Further screening of the reaction conditions revealed LiOH was a suitable base (entries 9–12). Pleasingly, we found that reducing the amount of LiOH to 1.2 equiv. did not restrain the transformation (entry 13). Among the solvent evaluation, the full transformation was observed in 1,4-dioxane in 8 h, and as a result, 83% yield of **2a** was produced (entries 14–16). Interestingly, a dilute 1,4-dioxane solution and reduced temperature were proved optimal for this reaction, which led to 96% yield of **2a** when

Table 1
Optimization of the reaction conditions.[a]



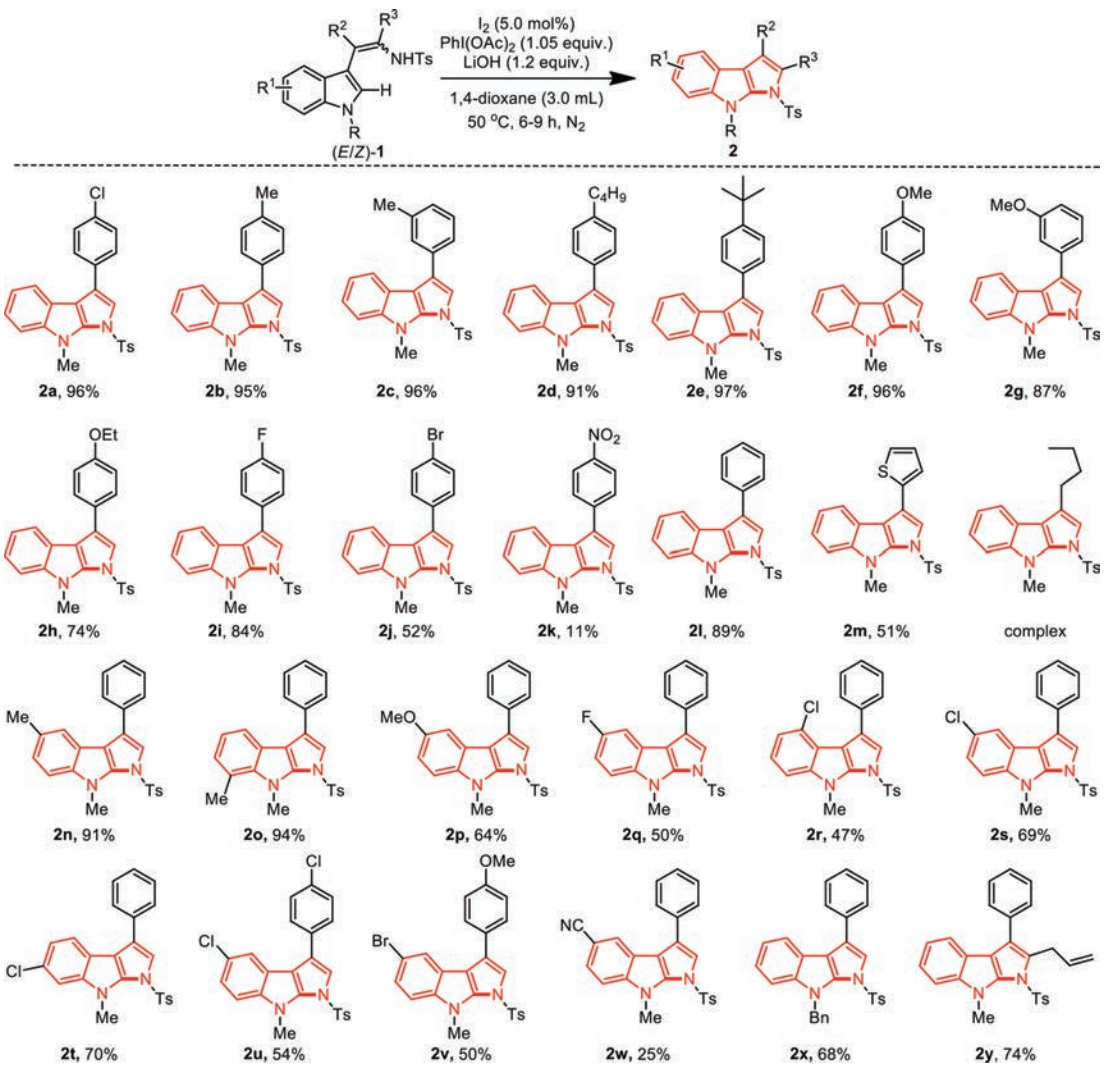
(*E*/*Z*)-**1a**

2a (CCDC: 2122559)

2a_1 (not observed)

Entry	I_2 (mol%)	Oxidant	Base (equiv.)	Solvent	Temp. (°C)	Yield (%) [b]
1	5	$Cu(OAc)_2$	Na_2HPO_4 (3.5)	Toluene	80	15
2	5	*t*BuOOH	Na_2HPO_4 (3.5)	Toluene	80	29
3	5	BQ	Na_2HPO_4 (2.5)	Toluene	80	54
4	5	TBHP	Na_2HPO_4 (2.5)	Toluene	80	62
5	10	TBHP	Na_2HPO_4 (2.5)	Toluene	80	52
6	2	TBHP	Na_2HPO_4 (2.5)	Toluene	80	trace
7	5	$PhI(OAc)_2$	Na_2HPO_4 (2.5)	Toluene	80	68
8[c]	5	$PhI(OAc)_2$	Na_2HPO_4 (2.5)	Toluene	80	40
9	5	$PhI(OAc)_2$	K_2CO_3 (2.5)	Toluene	80	35
10	5	$PhI(OAc)_2$	pyridine (2.5)	Toluene	80	12
11	5	$PhI(OAc)_2$	LiOH (2.5)	Toluene	80	78
12	5	$PhI(OAc)_2$	NaOH (2.5)	Toluene	80	31
13	5	$PhI(OAc)_2$	LiOH (1.2)	Toluene	80	80
14	5	$PhI(OAc)_2$	LiOH (1.2)	1,4-Dioxane	80	83
15	5	$PhI(OAc)_2$	LiOH (1.2)	MeCN	80	39
16	5	$PhI(OAc)_2$	LiOH (1.2)	DCM	80	49
17	5	$PhI(OAc)_2$	LiOH (1.2)	1,4-Dioxane (3.0 mL)	50	96
18	5	$PhI(OAc)_2$	LiOH (1.2)	1,4-Dioxane (3.0 mL)	30	54

[a] Reaction conditions: (*E*/*Z*)-**1a** (0.20 mmol), I_2 (x mol%), oxidant (1.05 equiv.), and base in 2.0 mL of solvent at 30–80 °C for 6–12 h.
[b] isolated yield based on **1a**.
[c] $PhI(OAc)_2$ (1.5 equiv.). TBHP = *tert*-Butyl hydrogen peroxide.

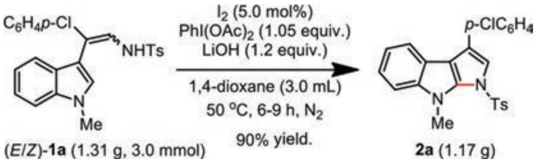


Scheme 2. Pragmatic synthesis of pyrrolo[2,3-*b*]indoles. Reaction conditions: (*E*/*Z*)-**1** (0.20 mmol), I_2 (5 mol%), $PhI(OAc)_2$ (1.05 equiv.), and LiOH (1.2 equiv.) in 3.0 mL of 1,4-dioxane at 80 °C, 5–8 h unless otherwise noted. Isolated yield based on (*E*/*Z*)-**1**.

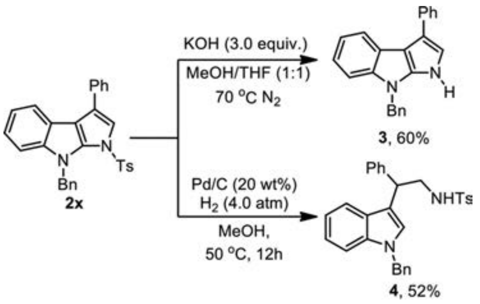
3.0 mL of 1,4-dioxane was utilized at 50 °C (entry 17). Lowering the temperature to room temperature resulted in inferior transformation and only a modest yield of **2a** was obtained (entry 18). Interestingly, the possible side product **2a_1**, which was presumably formed from (*E*)-**1a**, was not observed in this catalytic system. And the fact that the almost quantitative yield of **2a** demonstrated that this product should be originated from both (*Z*)-**1a** and (*E*)-**1a**, and thus mechanistically a common imine (C=NTs) intermediate should be the key species from the (*E*/*Z*)-**1a** substrates.

With the optimized reaction conditions in hand (Table 1, entry 17), we next explored the substrate scope and the results are presented in Scheme 2. Different aromatic substituents at R^2 position of (*E*/Z)-2-indolylenamides **1** were firstly studied. The results indicated that varied functional groups with electron-donating properties were well tolerated. For example, aryl groups at R^2 could be attached with Me (**2b** and **2c**), MeO (**2f** and **2g**), EtO (**2h**), *n*Bu (**2d**), and *t*Bu (**2e**), delivering the corresponding products in good to excellent yields. Other than chloride (**2a**), the synthetically useful halide groups, such as F (**2i**) and Br (**2j**) at the aryl moiety of R^2 also worked smoothly to afford the expected products in 84% and 52% yields, respectively. Note that the strong electron-withdrawing nitro group at the aryl group of R^2 was less reactive, providing the desired product **2k** in a hamper yield. Pleasingly, the heteroaryl 3-thienyl group was compatible for this C2-amidation of indole scaffold, delivering the desired product **2m** in 51% yield. Unfortunately, the alkyl-substitution located at the R^2 position of **1** was intolerable under this I_2-catalyzed oxidative condition, because the enamide **1** with a butyl-group at R^2 could be easily decomposed and no desired product could be detected.

For the substituents R^1 that were attached on to the benzenoid ring, results showed different functional groups with electron-donating or electron-withdrawing properties were all compatible (Scheme 2). Methyl groups which located at the 5- or 7-positions of indolyl benzoid moiety worked well to produce the desired products **2n** and **2o** in nice yields. Whereas 64% yield of **2p** was obtained for the cyclization of 5-MeO substituted substrate. In addition, 2-indolylenamides **1**, which bearing an electron-withdrawing halo-substitution such as fluoro (**2q**), chloro (**2r**, **2s**, **2t** and **2y**), and bromo (**2v**), were all well-tolerated without difficulty, regardless of their locations at the indole phenyl moieties. It was worth noting that the strong electron-withdrawing group like cyanide (**2w**) was also admissible tolerant, highlighting the possible subsequent divergent conversions of the products. In the case of the *N*-protecting group in the indolyl enamide, the benzyl group was found to be an optimal choice. The Bn-protect product **2x** could be isolated from a Bn-substituted 2-indolylenamide precursor **1** in 68% yield under the standard conditions. Gratifyingly, by modifying the substituted enamide as a substrate, we were able



Scheme 3. Gram-scale synthesis of product **2a**.



Scheme 4. Deprotection of the N-Ts.

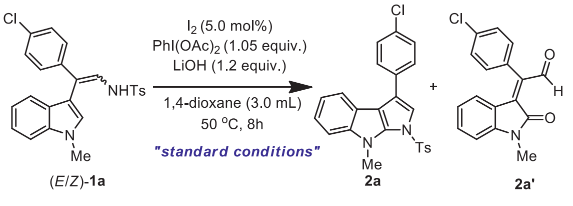
to obtain the fully substituted pyrrole product **2y** in 74% yield under the I_2-catalyzed optimal conditions.

The utility of this C–H/N–H coupling reaction can be demonstrated by a preparative gram-scale reaction, as the product **2a** can be isolated in 90% yield on the 3.0 mmol scale of (*E*/*Z*)-**1a** (Scheme 3).

Deprotection of the tosyl group from compound **2x** using KOH (3.0 equiv.) as a base [34] in MeOH/THF solvent was successful to obtain product **3** in a synthetically useful yield (60%). The attempts to remove the benzyl group in **2x** under Pd/C and low-pressure H_2 conditions were failed. However, a ring-opening product **4** was isolated under 4.0 atm of H_2 reduction conditions in MeOH at 50 °C (Scheme 4).

Mechanistic studies were carried out to understand the mechanism of this reaction (Table 2). When typical radical scavengers, such as TEMPO and BHT were added, the product **2a** could be isolated in modest to good yields, suggesting that a free radical pathway should be unlikely involved in this indolyl C2 amination reaction (entry 1). LiOH worked as a base and was found to be necessary for this reaction (entry 2). With a catalytic amount of I_2, several different types of oxidants could participate in this reaction to deliver the desired product **2a** (Table 1, entries 1–4). It was found that the stoichiometric amounts of I_2 could promote this cyclization protocol smoothly while in the absence of an oxidant (entry 3), however, no reaction occurred when $PhI(OAc)_2$ alone was added while in the absence of I_2 (entry 4). For the stoichiometric amounts of I_2-promoted reactions of (*E*/*Z*)-**1**, we found that most of the reactions were achieved in humble yields, and inseparable mixture of the desired product with unknown side product were observed. These results clearly indicated that an I_2–mediated catalytic cycle was involved in this transformation, and the oxidant should oxidize the molecular I_2 to offer a high reactive (hypo)iodites "I^+" species in this iodine catalytic cycle. Indeed, "I^+" species have been previously studied as powerful electrophiles which can react with C(sp^2)-H substrates to give iodo-intermediates [35,36].

Table 2
Mechanistic studies.



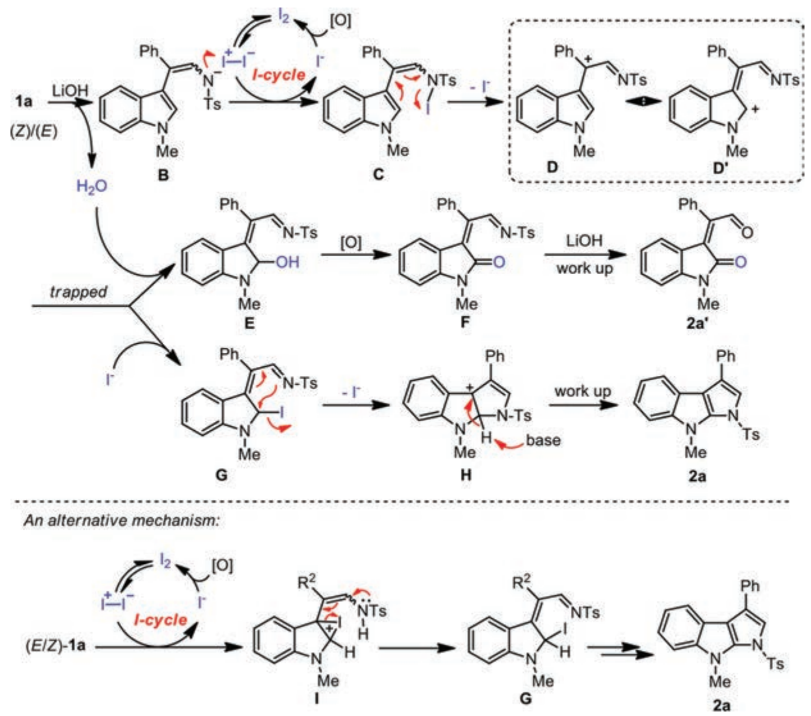
Entry	Variations beyond the "standard conditions"	**2a** (%)
1	With BHT or TEMPO as a radical scavenger	83 or 57
2	no LiOH	<10
3	I_2 (1.5 equiv.), no $PhI(OAc)_2$	80
4	$PhI(OAc)_2$ (2.0 equiv.), no I_2	n.r.
5	NaI (1.5 equiv.) instead of I_2 (5 mol%), no $PhI(OAc)_2$	n.r.
6	NaI (1.5 equiv.) instead of I_2 (5 mol%)	**2a** (trace) + **2a'** (28)
7	NIS (10 mol%) instead of I_2 (5 mol%)	81
8	NIS (1.5 equiv.), instead of I_2 (5 mol%), no $PhI(OAc)_2$	45

BHT = 2,6-di-*tert*–butyl–4-methylphenol. TEMPO = 2,2,6,6-tetramethyl-1-piperidinyloxy.

To confirm the possible existence of I^+, NaI (1.5 equiv.) was added instead of the catalytic amount of I_2 (5 mol%), we found that in this case, no reaction occurred when in the absence of oxidant (entry 5). Surprisingly, when NaI (1.5 equiv.) combined with 1.5 equiv. of $PhI(OAc)_2$ were added, the desired product **2a** was only traced, but the hydrolysis side product **2a'** was isolated in 28% yield (entry 6). The formation of aldehyde **2a'** was observed in our previously Cp^*Ir^{III}-catalyzed sequential C–H oxidation of (*E*/*Z*)-indolyl-3-enamines **1** followed by a TsOH-promoted hydrolysis reaction [28]. The observation of **2a'** in the current I_2-catalyzed reaction possibly indicated an indolyl C2-H oxidation to form the indolin-2-one intermediate be involved in this reaction. Further mechanistic studies using catalytic (entry 7) or stoichiometric (entry 8) amounts of NIS also strongly support the involvement of the I^+-cation species in the I-cycle in this reaction.

Based on the abovementioned results, we propose a possible mechanistic rationalization as illustrated in Scheme 5. The reaction was initiated by the reaction of (*E*/*Z*)-**1a** with LiOH, to form a nitrogen-centered anion species **B**, which was followed by nucleophilic iodination reaction with molecular I_2 to give intermediate **C** upon release of I^- anion. In the oxidant [O] promoted I-cycle, I^- could be oxidized to I_2 to complete the catalytic cycle [37]. In the following transformation, due to the electron-rich property of the indole, the conjugated π-system pushed an intramolecular heterolytic cleavage of the N-I bond in **C** to form ionic imine species **D** and its resonance imine isomer **D'**. The formation of imine (C=NTs) **D** or **D'** could explain why both of the stereoisomers (*E*)-**1a** and (*Z*)-**1a** all transformed into compound **2a**. In addition, for the reaction of (*E*/*Z*)-**1** with an electron-donating functional group at its R^2 position, such as a Me, *t*Bu, and OMe group, the corresponding products **2e** and **2f** were obtained in almost quantitative yields, whereas for the reaction of (*E*/*Z*)-**1** with a strong electron-withdrawing functional group, the corresponding products **2k** and **2w**, the yields of the expected products were only humble. These results should support the formation of carbocation **D** and resonance isomer **D'**. In the subsequent reactions, **D'** could be trapped by H_2O to give the intermediate **E**. The following oxidation of **E** produced indolin-2-one **F**, which was followed by a hydrolysis reaction to deliver the aldehyde side product **2a'**. Meanwhile, intermediate **D'** could also be trapped by I^- to generate the 2-iodoindoline species **G**. The subsequent nucleophilic attack and cyclization reaction of **G** produced pyrroloindoline cation **H**, and finally, this species transformed into product **2a** while with the assistance of LiOH.

An alternative mechanism was described as below in Scheme 5. In this mechanism, the indole moiety goes through electrophilic iodination **I**, and then ring-opening of the resultant iodonium salt gives the intermediate **G**. While this mechanism seemed reasonable, there's a key point that this putative pathway cannot be clar-



Scheme 5. Proposed mechanism.

ified: since the electronic effect was not very obvious from this alternative mechanism, thus, the reason is not clear why substrate (*E*/*Z*)-**1** with electron-donating groups, which are located at R^2 position, was much more reactive than those of substrates **1** with electron-withdrawing functional groups (**2k** and **2w**). However, in the mechanistic rationalization, as illustrated above in Scheme 5, the stability of the carbocation **D** and **D'** could explain this phenomenon very well.

In summary, I_2/PhI(OAc)$_2$ catalytic protocol was developed to promote the intramolecular oxidative C–H/N–H amination of (*E*/*Z*)-2-indolylenammides. The reaction worked smoothly under environmentally benign conditions and the desired pyrrolo[2,3-*b*]indoles were generated in mostly good to excellent yields under metal-free conditions. In light of the green and environmentally benign strategy that utilizing inexpensive I_2 as a catalyst to promote C–N bond formation sequences toward fused aza-heterocycles, this method should be potentially served as a pragmatic synthetic approach for the late-stage modification of pyrrolo[2,3-*b*]indoles, such as Pyrroindomycin analogues. Detailed mechanistic studies indicated an I^+-mediated catalytic cycle was proposed in this reaction.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.05.083.

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