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Communication

A practical synthesis of *β*-carbolines by tetra-*n*-butylammonium bromide (TBAB)-mediated cycloaromatization reaction of aldehydes with tryptophan derivatives

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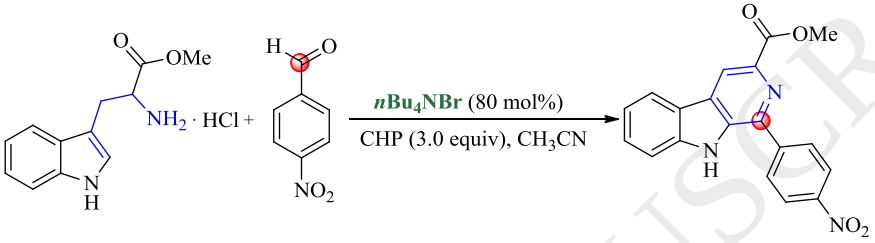
Graphical abstract



Br-mediated cycloaromatization
Without column chromatography
Multigram-scale synthesis
5.8 g, 67% yield

A mild and efficient *n*Bu4NBr-mediated oxidative cycloaromatization to prepare *β*-carbolines from readily available tryptophans and aldehydes is described. The reaction is practical and allows the synthesis of *β*-carbolines on gram-scale. Some of products crystallized from the reaction mixture and were easily removed by filtration, obviating the need for chromatographic separation.

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ABSTRACT

A mild and efficient *n*Bu4NBr-mediated oxidative cycloaromatization to prepare *β*-carbolines from readily available tryptophans and aldehydes is described. The reaction is practical and allows the synthesis of *β*-carbolines on gram-scale. Some of products crystallized from the reaction mixture and were easily removed by filtration, obviating the need for chromatographic

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Carbolines
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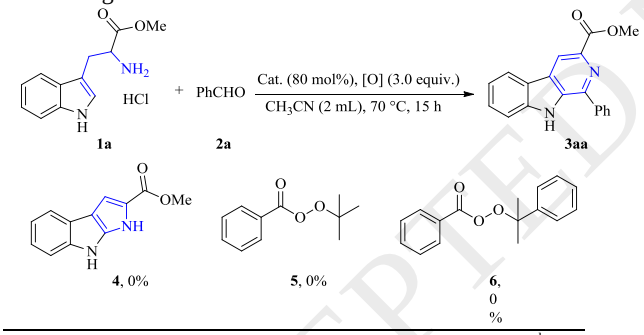
TBAB
tetra-*n*-Butylammonium bromide

separation.

Aromatic *β*-carboline derivatives are frequently found in naturally occurring substances and synthetic analogues [1]. They have proven to be useful in the treatment of malaria [2], cancer [3], AIDS [4], and other diseases [5]. They also act as photosensitizers in material science [6]. In view of the importance of the *β*-carboline motif, particularly in medicinal chemistry, the green and practical methods for the preparation of these compounds are desirable. In the traditional methods such as Pictet-Spengler reaction [7] and Bischler-Napieralski cyclization [8], a protic or Lewis acid is needed to obtain the imine intermediate and the reactions take place under harsh conditions. Moreover, a subsequent oxidation step is required to form the desired aromatic *β*-carbolines [9]. Recently, oxidative cycloaromatization reactions for the synthesis of *β*-carbolines have been developed by use of transition-metal catalysts including Pd [10], Cu [11], Ru [12], Rh [13], Au [14], and bimetallic Cu/Rh [15]. Although these reported protocols were successfully applied to syntheses of *β*-carboline derivatives, most of the reactions occurred in the presence of expensive metal catalysts, and in certain cases the starting materials were not easily accessible. In 2013, Wu and co-workers developed a metal-free cascade cycloaromatization reaction of aromatic ketones with tryptamines through the use of a combination of I_2 and H_2O_2. Diverse natural products containing a core structure of *β*-carboline were efficiently constructed in one-step under their reaction conditions [16]. During the last decade the readily available iodine reagents such as molecular iodine and iodide salts have been studied as environmentally friendly alternatives to metal catalysts in the oxidative C-H bond functionalization [17]. In contrast, the bromine-catalytic version of the oxidative C-H bond functionalization was mainly unexplored until 2013 when Glorius and Li independently reported tetraalkylammonium bromide catalyzed intramolecular dehydrogenative arylation of aldehydes that allows straightforward access to fluorenones and xanthones [18,19]. More recently, some novel transformations were achieved by use of bromine catalysts rather than iodine catalysts [20]. As part of our continuous efforts in searching for the metal-free halide (iodine and bromine)-mediated cross-dehydrogenative coupling (HCDC) reactions [20,21], we report herein an *n*Bu_4NBr-mediated cycloaromatization of tryptophan derivatives with aldehydes to prepare *β*-carbolines in one-step. This approach is characterized by its readily available starting materials, mild conditions, operational simplicity and multigram scale synthesis while relying on *n*Bu_4NBr as an environmentally benign and cheap mediator.

Table 1
Screening of the reaction conditions.[a]



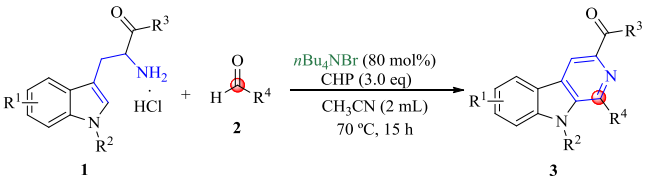
Entry	Cat.	[O]	Yield (%)[b]
1	I_2	TBHP	trace
2	*n*Bu_4NI	TBHP	52
3	*n*Bu_4NBr	TBHP	60
4	*n*Bu_4NBr	CHP	91
5	*n*Bu_4NBr	CHP	20[c]
6	Et_4NBr	CHP	42
7	NBS	CHP	35
8	NaBr	CHP	trace
9	$BF_3{\cdot}Et_2O$	CHP	0
10	$ZnCl_2$	CHP	17
11	PPA	CHP	0
12	PhCOOH	CHP	25
13	TFA	CHP	46
14	TFAA	CHP	31

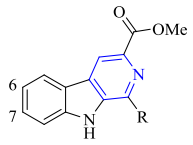
[a] Reaction conditions: **1a** (0.25 mmol), **2a** (1.5 equiv., 0.38 mmol), catalyst (80 mol%), oxidant (3.0 equiv.), under air.
[b] Isolated yields.
[c] *n*Bu_4NBr (50 mol%).

At the outset of our investigation, the readily available tryptophan methyl ester hydrochloride **1a** and benzaldehyde **2a** were employed as the model substrates, and various catalysts were examined using hydroperoxides as oxidants (Table 1). When I_2 was used as

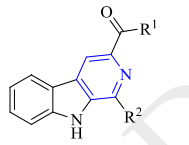
catalyst, trace amounts of product **3aa** was detected and starting material was reclaimed (entry 1). We previously reported that the combination of I_2/TBHP promoted an intramolecular annulation of **1a** to afford pyrrolo[2,3-b]indole **4** using 1,4-dioxane as the solvent (Table 1) [21a]. Interestingly, no intramolecular annulation product was observed under the present reaction conditions. The reaction was then investigated with *n*Bu_4NI/TBHP system, and the desired product **3aa** was obtained in 52% yield (entry 2). When *n*Bu_4NBr was used instead of *n*Bu_4NI, a higher yield of **3aa** was observed (entry 3). We further found that the combination of *n*Bu_4NBr with cumene hydroperoxide (CHP) promoted the cycloaromatization more efficiently to provide *β*-carboline **3aa** in 91% yield (entry 4). In 2011, Wan and coworkers reported an *n*Bu_4NI-catalyzed cross-dehydrogenative coupling reaction of aldehyde with TBHP to prepare *tert*-butyl perester **5** using H_2O as the solvent (Table 1) [22]. It should be noted that, no evidence for the formation of peresters **5** and **6** was observed under the present reaction conditions (entries 1-4). The use of 50 mol% of *n*Bu_4NBr significantly decreased the yield of product (entry 5). Other bromides such as Et_4NBr, NBS and NaBr were found to be less effective than *n*Bu_4NBr (entries 6-8). We also examined other catalysts such as Lewis and protic acids. However, they proved to be less efficient or ineffective (entries 9-14).

With optimized conditions in hand (Table 1, entry 4), we next studied the scope and limitations of the *β*-carboline synthesis by first varying the aldehyde component in the reaction with tryptophan methyl ester hydrochloride **1a** (Scheme 1, **3aa-ay**). Benzaldehydes with electron-withdrawing (*e.g.*, −Hal, $-NO_2$, −CN, $-CO_2Me$, $-CF_3$) and moderately electron-donating groups ($-CH_3$, −*i*Pr) were well-tolerated, leading to the desired products **3ab-ao** in high yields (80%-99%). The moderate yields were observed when 4-methoxy and 4-benzyloxy benzaldehydes were subjected to the reaction (**3ap** and **3aq**). 4-Methylthiobenzaldehyde was converted to the corresponding *β*-carboline **3ar** in 58% yield. Interestingly, this annulation was accompanied by a concomitant oxidation of thioether, resulting in the formation of sulfoxide substituted *β*-carboline **3ar'** in 41% yield. The reactions of 2-naphthaldehyde, 2-thiophenecarbaldehyde and 2-pyridinecarbaldehyde occurred smoothly to afford the corresponding products **3as-au** in good yields. In the case of formaldehyde, the reaction gave the desired product **3av** in 62% yield. Its structure was confirmed unambiguously by single crystal X-ray diffraction (Supporting information). Unfortunately, the aliphatic aldehydes showed low reactivity, giving products **3aw** and **3ax** in low yields and large amounts of recovered starting material. Surprisingly, when the annulation was attempted with salicylaldehyde, an unexpected bromination reaction of product **3ay** occurred to give 6-bromo-*β*-carboline **3ay'** in 25% yield.

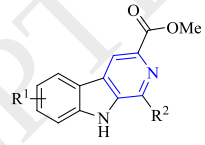




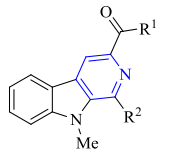
3aa, R = Ph, 91% (1.2 g, 80%)
3ab, R = 4-ClC_6H_4, 93%
3ac, R = 2-ClC_6H_4, 91%
3ad, R = 2,4-diClC_6H_3, 95%
3ae, R = 4-BrC_6H_4, 99%
3af, R = 3-BrC_6H_4, 97%
3ag, R = 4-FC_6H_4, 87%
3ah, R = 4-$NO_2C_6H_4$, 96% (5.8 g, 67%)
3ai, R = 3-CNC_6H_4, 90%
3aj, R = 4-$COOMeC_6H_4$, 90%
3ak, R = 4-$CF_3C_6H_4$, 85%
3al, R = 2-MeC_6H_4, 89%
3am, R = 3-MeC_6H_4, 97%
3an, R = 4-MeC_6H_4, 81%
3ao, R = 4-iPrC_6H_4, 80%
3ap, R = 4-$MeOC_6H_4$, 58%
3aq, R = 4-$BnOC_6H_4$, 69%
3ar, R = 4-$MeSC_6H_4$, 58%
3ar', R = 4-$MeSOC_6H_4$, 41%
3as, R = 2-naphthyl, 88%
3at, R = 2-thiophenyl, 73%
3au, R = 2-quinolyl, 90%
3av, R = H, 62%
3aw, R = *n*Pr, 34%
3ax, R = Cy, 45%
3ay, R = 2-HOC_6H_4, 52%
3ay', R =2-HOC_6H_4, 6-Br, 25%



3ba, R^1 = OEt, R^2 = Ph, 84%
3bi, R^1 = OEt, R^2 = 3-CNC_6H_4, 88%
3ca, R^1 = OBn, R^2 = Ph, 49%



3da, R^1 = 7-Cl, R^2 = Ph, 41%
3dh, R^1 = 7-Cl, R^2 = 4-$NO_2C_6H_4$, 87%
3ea, R^1 = 6-Br, R^2 = Ph, 15%
3eh, R^1 = 6-Br, R^2 = 4-$NO_2C_6H_4$, 84%
3fh, R^1 = 6-CN, R^2 = 4-$NO_2C_6H_4$, 82%
3ga, R^1 = 6-Me, R^2 = Ph, 64%

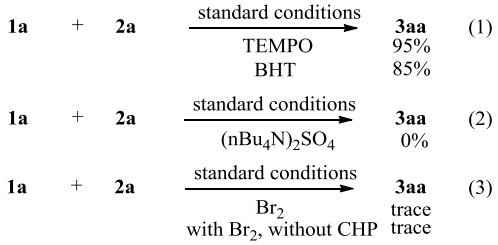


3ha, R^1 = OMe, R^2 = Ph, 77%
3hi, R^1 = OMe, R^2 = 3-CNC_6H_4, 95%
3ia, R^1 = NH_2, R^2 = Ph, 53%

Scheme 1. Scope of the substrates. Reaction conditions: 1 (0.25 mmol), 2 (1.5 equiv., 0.38 mmol), *n*Bu_4NBr (80 mol%), CHP (3.0 equiv.), under air; isolated yields.

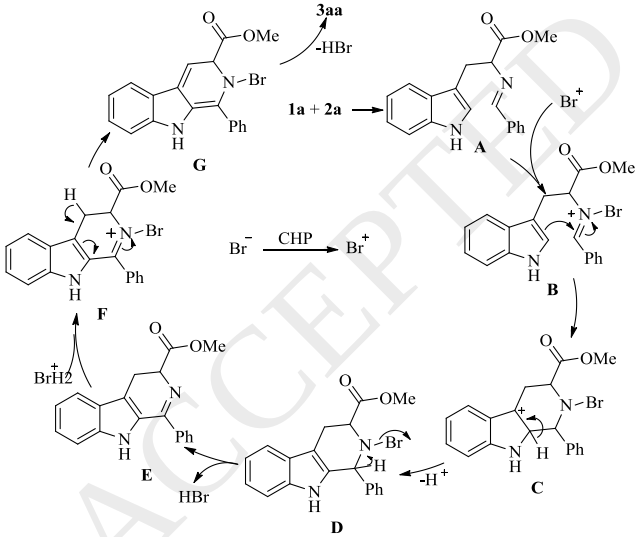
We continued the studies by varying the tryptophan ester component. The ethyl and benzyl esters of tryptophan were both compatible with the reaction conditions (**3ba**, **3bi** and **3ca**). When benzaldehyde **2a** was used as the aldehyde component, the groups in the 6-, or 7-position of indole ring had a marked effect on the efficiency of the reaction. They decreased the yields of products regardless whether they were EWG or EDG (**3da**, **3ea** and **3ga**). In contrast, the high reactive 4-nitrobenzaldehyde reacted with these tryptophan methyl esters to afford *β*-carbolines in good yields (**3dh**, **3eh** and **3gh**). The present annulation was also attempted on *N*-methyl tryptophan methyl ester **1h**. The desired products **3ha** and **3hi** were obtained in good yields. We finally found that even a primary amide group could be tolerated under the reaction conditions (**3ia**). In the cases of **3ad-af**, **3ah**, **3ai**, **3bi**, **3dh-fh** and **3hi**, they precipitated from the reaction mixture as crystalline complexes at the end of the reaction (Scheme 1, blue colour). The analytically pure products could be readily isolated by filtration. Moreover, the present synthesis is very practical and allows the preparation of *β*-carbolines on gram-scale (**3aa**, 1.2 g, 80% yield; **3ah**, 5.8 g, 67% yield). It should be noted that the present reaction could not be applicable to the 5 or 8-substituted tryptophan esters due to the production of complex product mixtures.

The control experiments were performed to explore the mechanism of the cycloaromatization after we established the substrate scope (Scheme 2). The addition of radical inhibitors TEMPO or BHT had no effect on the yield of the **3aa** (eq. 1). When bromine-free phase transfer catalyst such as $(n\mathrm{Bu_4N})_2\mathrm{SO_4}$ was used in place of $n\mathrm{Bu_4NBr}$, **3aa** was not observed (eq. 2). To explore the potential bromine species, $\mathrm{Br_2}$ was further used as the mediator. The reactions afforded complex mixtures containing trace amounts of **3aa** (eq. 3).



Scheme 2**.** Control experiments.

Based on our results and previous reports [23], a mechanistic pathway is proposed in Scheme 3. First, the reaction of **1a** with **2a** yields an imine **A**, which undergoes bromination with Br^+ to afford *N*-bromoiminium **B** [23b,24]. The electrophilicity of imine **B** is enhanced by the halogen bond interaction [24a]. Second, the intramolecular cyclization of **B** yields **C**, which converts into **D** through the elimination of proton. Third, the elimination of HBr from **D** yields the partially aromatized intermediate **E**. Finally, the bromination of **E** takes place to give intermediate **F,** followed by elimination of HBr from **G** to afford product **3aa** [9g,9h].



Scheme 3. Proposed mechanism.

In summary, we have developed a practical and scalable method for the synthesis of *β*-carbolines. The protocol uses readily available tryptophans and aldehydes as starting materials, and $n\mathrm{Bu_4NBr}$ as the environmentally friendly mediator, providing target products in high yields. Considering the valuable structure of the products, simple isolation procedure and acid/metal-free conditions, this methodology should find broad applications.

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