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Brønsted acid-catalyzed asymmetric dearomatization of indolyl ynamides: Practical and enantioselective synthesis of polycyclic indolines

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ABSTRACT

Catalytic asymmetric dearomatization of indoles and alkynes has received much attention in the past decade because this strategy offers an attractive and alternative way for the efficient synthesis of valuable chiral polycyclic indolines. However, these reactions have been mostly limited to transition-metal catalysts, and the related chiral Brønsted acid catalysis has been scarcely reported. Herein, we disclose a chiral phosphoric acid-catalyzed asymmetric dearomatization of indolyl ynamides by direct activation of alkynes. This metal-free method enables the practical and atom-economical construction of an array of valuable chiral polycyclic indolines in moderate to good yields with high enantioselectivities

Polycyclic indolines are important scaffolds for natural products and pharmaceutical molecules, such as indoline alkaloids (Fig. 1) [1–6]. The preparation of tetracyclic indolines, which contain hydrocarbazole skeletons, was reported by Wang *et al.* (Scheme 1a) [7]. However, tetracyclic indolines containing hydro-γ-carboline skeletons receive less attention. To the best of our knowledge, there is only one example for the synthesis of tetracyclic-γ-carbolines reported by Zu *et al.* [8], which affords racemic tetracyclic-γ-carbolines through Brønsted acid-catalyzed Grob fragmentation/Mannich cyclization followed by intramolecular cyclization. Considering the potential application for biological investigations of tetracyclic hydro-γ-carbolines [9], it is urgent to develop a new strategy for enantioselective synthesis of indoline compounds bearing tetracyclic hydro-γ-carbolines.

Catalytic asymmetric dearomatization (CADA) [10–19] is a powerful strategy to construct chiral quaternary carbon centers in the original aromatic rings, such as phenyl [20–27], naphthyl [28–37] and indolyl [38–79] rings. In recent years, CADA reactions involving indoles have been widely reported. For example, You *et al.* developed a series of elegant protocols on the transition-metal-catalyzed asymmetric allylic dearomatization of indoles [38–44]. In addition, other types of CADA reactions catalyzed by Au [45–48], Cu [49,50] and Pd [51–54] have been also nicely demonstrated. Furthermore, various methodologies on the metal-free CADA reactions of indoles have been established by employing chiral Brønsted acids (BA) [55–66], organobases [67–78] and other organocatalysts [79,80] as catalysts. Among these, catalytic asymmetric dearomatization of indoles and alkynes has received particular attention because this strategy offers an attractive and alternative way for the efficient synthesis of valuable chiral polycyclic indolines [8,45,48,55-66,81]. For example, Bandini *et al.* reported a gold-catalyzed enantioselective tandem cyclization of indoles and alkynes, constructing polycyclic indolines [48]. Besides, Wang [2] and Unsworth [81] applied similar strategies for the assembly of polycyclic indoles using silver as the catalyst (Scheme 1a). Conversely, chiral Brønsted acid-catalyzed dearomatization reactions of indoles and alkynes have been scarcely reported, probably due to the difficulties in activation of alkynes and controlling the enantioselectivities. To the best of our knowledge, there are only two examples of chiral phosphoric acid (CPA)-catalyzed asymmetric dearomatization of indoles and alkynes (Schemes 1b and c) reported by Wang [57] and Shi [55]. However, electron-deficient alkynes were directly or indirectly utilized in both cases, where chiral Brønsted acids were essentially employed to activate the imine moieties, but not carbon–carbon triple bonds. In other

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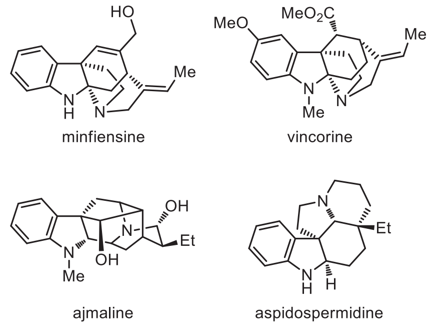
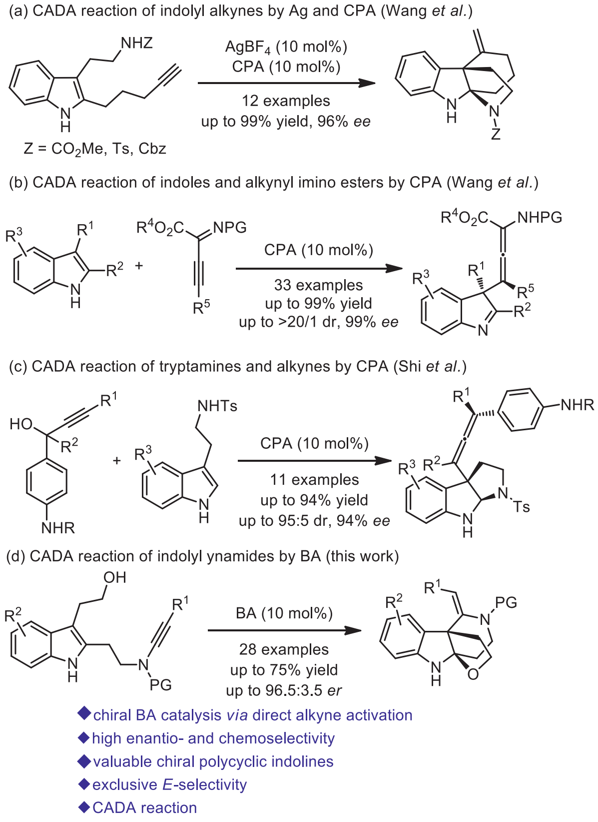


Fig. 1. Bioactive polycyclic indolines.

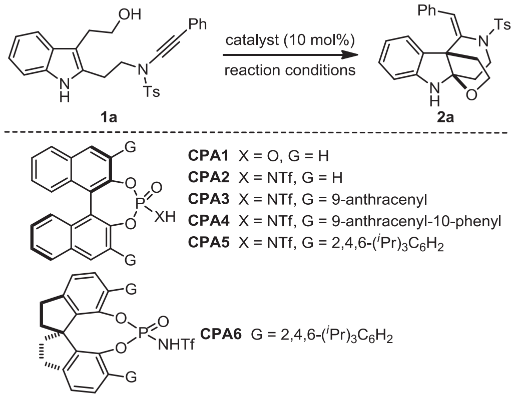


Scheme 1. CADA reactions of indoles and alkynes.

words, CADA reaction of indoles and alkynes *via* chiral BA catalysis by direct alkyne activation has not been reported yet.

Ynamides have proven to be important synthons in organic synthesis and have attracted much attention in the past decade [82–96]. In recent years, ynamide chemistry involving asymmetric transition-metal catalysis [97–101] and metal-free catalysis [102–106] has been successively established by our group. In 2019, our group reported a BA-catalyzed intramolecular alkoxylation/1,3-rearrangement of ynamides, indicating that BA could directly and efficiently activate ynamides [105]. Based on this discovery, we further realized CPA-catalyzed intramolecular CADA reactions of ynamides with naphthols, phenols and pyrroles [103,104]. Based on the above BA-catalyzed asymmetric reactions of ynamides and other transition-metal-free catalytic reactions by our group [107–111], we envision that indolyl ynamides might be activated by BA and the keteniminiums generated from ynamides could be attacked by 3-position of indole moieties, followed by an intramolecular cyclization with the hydroxyl group tethered on the indole partner, eventually leading to chiral polycyclic indolines.

Table 1
Optimization of reaction conditions.[a]



Entry	Catalyst	Reaction conditions	Yield (%)[b]	*er* (%)[c]
1[d]	**CPA1**	Toluene, 60 °C, 24 h	<5	-
2	**CPA2**	Toluene, 60 °C, 3 h	34	50:50
3	**CPA3**	Toluene, 60 °C, 24 h	51	81:19
4	**CPA4**	Toluene, 30 °C, 5 h	46	86.5:13.5
5	**CPA5**	Toluene, 30 °C, 5 h	37	60:40
6	**CPA6**	Toluene, 30 °C, 4 h	22	54.5:45.5
7	**CPA4**	PhCl, 30 °C, 4 h	44	85:15
8	**CPA4**	DCM, 30 °C, 4 h	56	82:18
9	**CPA4**	TBME, 30 °C, 4 h	72	94:6
10	**CPA4**	THF, 30 °C, 24 h	58	83:17
11	**CPA4**	MeCN, 30 °C, 24 h	35	63.5:36.5
12[e]	**CPA4**	TBME, 30 °C, 4 h	70	92.5:7.5
13[f]	**CPA4**	TBME, 30 °C, 4 h	64	92:8

Ts: 4-toluenesulfonyl; TBME: *tert*-butyl methyl ether.

[a] Reaction conditions: **1a** (0.05 mmol), catalyst (0.005 mmol), solvents (1 mL), 30–60 °C, 4–24 h, in vials.

[b] Measured by ^{1}H NMR using diethyl phthalate as the internal reference.

[c] Determined by HPLC.

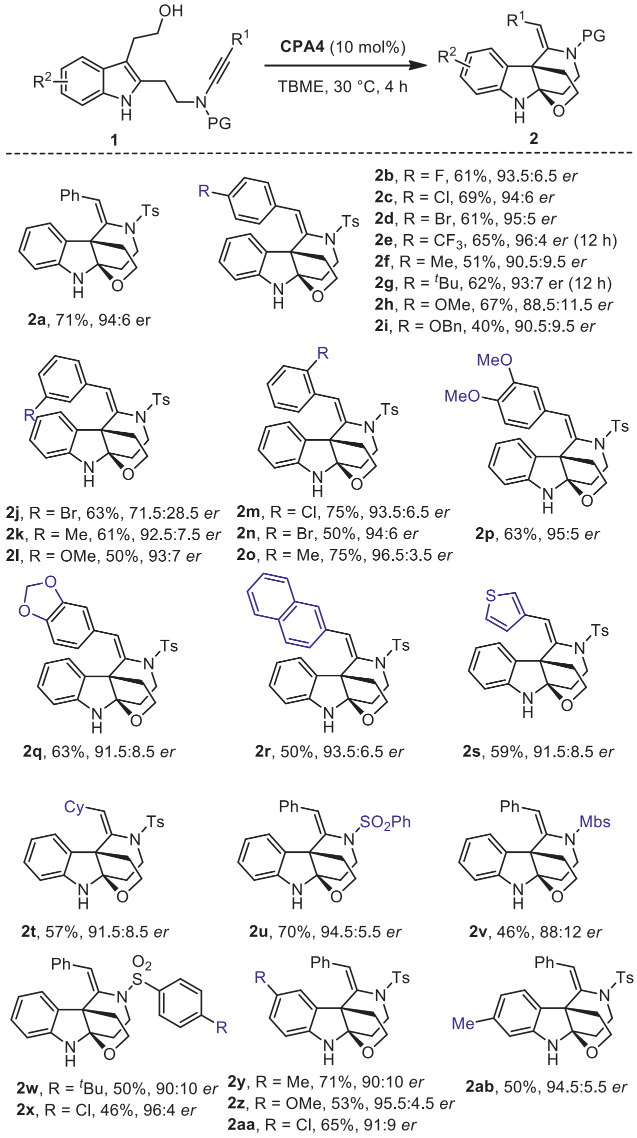
[d] >90% of **1a** remained unreacted.

[e] Using 15 mol% catalyst instead of 10 mol%.

[f] 0.5 mL of solvent was used.

Herein, we disclose a CPA-catalyzed asymmetric dearomatization of indolyl-ynamides [112], allowing atom-economical, practical and enantioselective construction of an array of valuable chiral polycyclic indolines containing hydro-γ-carboline skeletons in moderate to good yields with wide substrate scope and high enantioselectivities (Scheme 1d). Importantly, this protocol represents the first CADA reaction of indoles and alkynes *via* chiral BA catalysis by direct alkyne activation.

We initiated our investigation on condition optimization by using indolyl ynamide **1a** as the model substrate (Table 1). At the outset, **CPA1** was employed as catalyst, but the desired product could not be afforded in the presence of toluene as solvent at 60 °C (Table 1, entry 1). Other typical chiral phosphoric acids, which were used in our previous CADA reactions [103], also failed to deliver the desired product, see Supporting information for details. To our delight, the use of more acidic phosphoramide **CPA2** [113–116] as catalyst led to the formation of the desired polycyclic indoline **2a** in 34% yield albeit with no enantioselectivity (Table 1, entry 2). Encouraged by this result, we screened several BINOL-derived phosphoramide catalysts **CPA3**–**CPA5** and lowered the reaction temperature to 30 °C (Table 1, entries 3–5). When **CPA4** bearing 9-anthracene-10-phenyl group on the 3,3′-position of the BINOL framework was used as catalyst, the desired product **2a** could be obtained in 46% yield with 86.5:13.5 *er* (Table 1, entry 4). Using chiral spiro phosphoramide **CPA6** bearing two 2,4,6-triisopropylphenyl groups could not give a higher *ee* value (Table 1, entry 6) compared to **CPA5** bearing the same substituents (Table 1, entry 5). Subsequent examination of typical solvents revealed that



Scheme 2. Scope of the CADA reaction of indolyl ynamides **1**. Reaction conditions: **1** (0.15 mmol), **CPA4** (0.015 mmol), TBME (3 mL), 30 °C, 4 h, in vials; yields are those for the isolated products; ers are determined by HPLC analysis. PG = protecting group, Mbs = 4-methoxybenzenesulfonyl.

the use of *tert*-butyl methyl ether (TBME) as solvent significantly improved the enantioselectivity, generating **2a** in 72% yield with 94:6 *er* (Table 1, entry 9). Finally, raising the amount of **CPA4** to 15 mol% (Table 1, entry 12) or increasing the concentration of the reaction (Table 1, entry 13) only led to comparable results.

With the optimized reaction conditions in hand (Table 1, entry 9), we next explored the substrate scope of this CADA *via* CPA catalysis by assessing a variety of substituents on the indolyl ynamides **1**. As shown in Scheme 2, the reaction proceeded smoothly with the model substrate **1a** to provide **2a** in 71% yield with 94:6 *er*. Then, various 4-substituents on the phenyl ring attached to the terminal of ynamide were screened. A range of EWGs including F, Cl, Br, CF_3, and EDGs such as Me, ^{t}Bu, OMe, OBn were well tolerated, resulting in the formation of the corresponding chiral polycyclic indolines **2b**–**2i** in moderate to good yields (40%–69%) with good to excellent enantioselectivities (88.5:11.5 to 96:4 *er*). The scopes of different substituents on the 3- and 2-position of phenyl ring were also investigated. The 3-Br substituted substrate gave **2j** in moderate enantioselectivity (71:5:28.5 *er*) while ynamides bearing 3-Me, -OMe and 2-Cl, -Br, -Me groups could afford the desired products **2k**–**2o** in 50%–75% yields with 92.5:7.5 to 96.5:3.5 *er*. Besides, substrates containing disubstituted aryl groups afforded **2p** and **2q** in 63% yield with 91.5:8.5 and 95:5 *er* respectively. Apart from the phenyl rings, other aryl rings such as 2-naphthyl and 3-thienyl rings were also tolerated in this reaction (**2r** and **2s**). Moreover, cyclohexyl substituted ynamide could be smoothly converted into the desired **2t** in 57% yield with 91.5:8.5 *er*. The use of different N-protecting groups of ynamides led to the formation of **2u**-**2x** in 46%–70% yields with 88:12–96:4 *er*. Finally, various substituted indolyl ynamides were also compatible with this reaction (**2y**–**2ab**). Of note, exclusive *E*-selectivity of the newly generated exo-cyclic olefin moieties was observed in all cases, and this reaction is also highly chemoselective without the formation of the competing *N*-cyclization product. The absolute configuration of **2d** was verified by X-ray crystallographic analysis (Fig. 2).

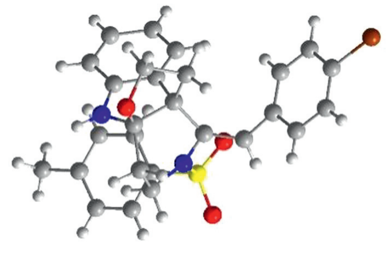
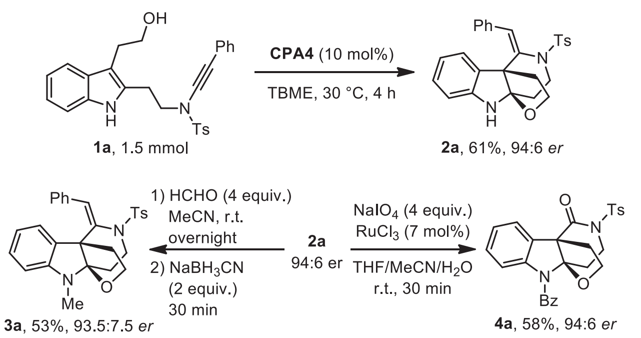


Fig. 2. Structure of compound **2d** in its crystal.

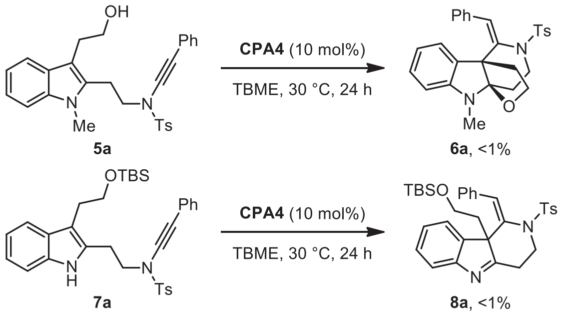


Scheme 3. Preparative scale reaction and synthetic applications of **2a**.

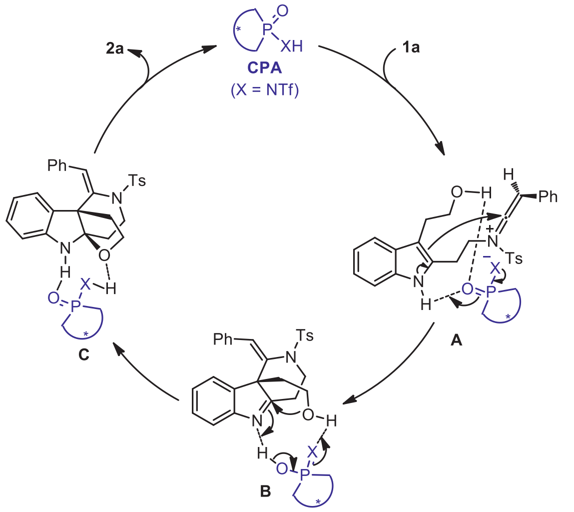
Subsequently, synthetic applications of the chiral polycyclic indoline **2a** were investigated (Scheme 3). First, a preparative scale reaction of **1a** was conducted under the standard condition, and **2a** could be obtained in 61% yield with *er* of 94:6 in a 1.5 mmol scale reaction. Further transformations of product **2a** were then explored. As indicated in Scheme 2, the treatment of **2a** with formalin followed by reduction with $NaBH_3CN$ could lead to the methylation of indoline N-H, affording **3a** in 53% yield with 93.5:6.5 *er*. Moreover, the enamide moiety of **2a** could be readily oxidized by $NaIO_4$ in the presence of $RuCl_3$ as catalyst, and the N-H of indoline was simultaneously substituted by the Bz group, affording **4a** in 58% yield with 94:6 *er*. This result was similar to the transformation previously reported by our group [103].

To enhance the understanding of the reaction mechanism, several control experiments were carried out. As illustrated in Scheme 4, the reaction of N-Me indolyl ynamide **5a** was first conducted under standard conditions, but failed to deliver the desired **6a**. In addition, when the hydroxyl group was protected by TBS group, the corresponding dearomatization product **8a** could not be observed. These results indicate that both the N-H of indole and the hydroxyl group are indispensable for the reaction.

Based on the above experimental results and our previous studies on chiral BA catalysis [102–105], a plausible mechanism is de-



Scheme 4. Control experiments.



Scheme 5. Plausible mechanism.

picted in Scheme 5. The indolyl ynamide **1a** is first converted into keteniminium complex **A** in the presence of CPA. Subsequently, CPA forms ion pair with keteniminium and possible hydrogen bond with both the hydroxyl group and the N-H of indole, which is similar to the reported CPA activation mode [103]. Then, the 3-position of indole attacks the keteniminium and dearomatization of indole occurs meanwhile, delivering iminium intermediate **B**. Subsequent intramolecular cyclization assisted by CPA generates intermediate **C** and finally leads to the formation of polycyclic indoline **2a** with the release of CPA catalyst. In other words, CPA here plays a key role in activating both the ynamide moiety and the iminium intermediate and thus controlling the enantioselectivity *via* hydrogen bonding and ion pair interactions.

In summary, we have developed a novel CPA-catalyzed asymmetric dearomatization of indolyl-ynamides, which represents the first CADA reaction of indoles and alkynes *via* chiral BA catalysis by direct alkyne activation. This metal-free method allows atom-economical, practical and enantioselective construction of an array of valuable chiral polycyclic indolines containing hydro-γ-carboline skeletons in moderate to good yields with wide substrate scope and high enantioselectivities (up to 96.5:3.5 *er*) under mild conditions. Thus, this reaction shows an excellent multifold selectivity, including chemo-, regio- and enantioselectivity. The polycyclic indolines are expected to find their utilities in organic synthesis and medicinal chemistry. Control experiments indicate that CPA plays a key role in activating both the ynamide moiety and the iminium intermediate and thus controlling the enantioselectivity *via* hydrogen bonding and ion pair interactions. We envision that this strategy involving direct Brønsted acid activation of ynamides can be applied in other CADA reactions based on ynamides.

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.2022.06.070.

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