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Gold-catalyzed intermolecular amination of allyl azides with ynamides: Efficient construction of 3-azabicyclo[3.1.0] scaffold

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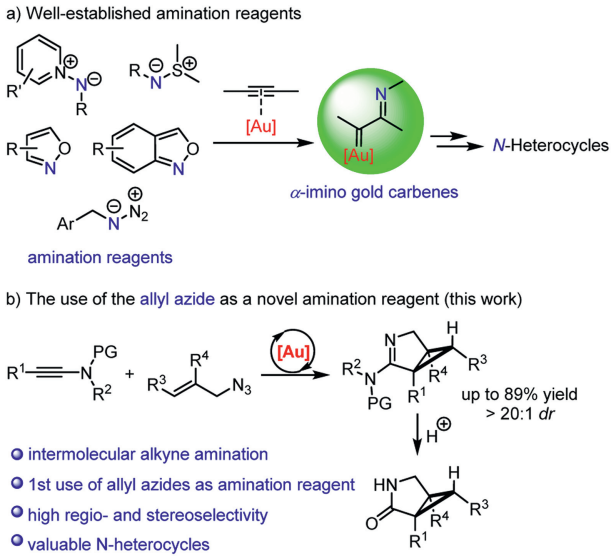
ABSTRACT

Gold-catalyzed amination reactions based on azides *via* α-imino gold carbene intermediates have attracted extensive attention in the past decades because this methodology leads to the facile and efficient construction of synthetically useful N-containing molecules, especially valuable *N*-heterocycles. However, successful examples of intermolecular generation of α-imino gold carbenes by using azides as amination reagents are rarely explored probably due to the weak nucleophilicity of azides. Herein, we disclose an efficient gold-catalyzed intermolecular aminative cyclopropanation of ynamides with the allyl azides, enabling flexible synthesis of a wide range of valuable 3-azabicyclo[3.1.0]hex–2-ene derivatives in good to excellent yields with excellent diastereoselectivities. Importantly, this protocol represents the first use of allyl azide as an efficient amination reagent in gold-catalyzed alkyne amination reactions.

The generation of α-imino metal carbenes is arguably the most important aspect of metal carbene chemistry attributed to their abundant carbene transfer reactions in the synthesis of valuable N-containing molecules [1–12]. Among these, gold-catalyzed alkyne amination reactions *via* α-imino gold carbene complexes have proved to be a powerful and intriguing method in organic synthesis because of their high regio- and stereoselectivity for rapid assembly of a variety of worthwhile *N*-heterocycles from readily available alkynes. In the past decades, a series of amination reagents, including nitrogen ylides [13–20], sulfilimines [21–27], isoxazoles [28–37], anthranils [38–46] and others [47–52], have been nicely exploited in the gold-catalyzed intermolecular alkyne amination reactions (Scheme 1a). Since the pioneer work on the gold-catalyzed azide-alkyne cyclization reaction *via* α-imino gold carbenes in 2005 by Toste and co-workers [53], an increasing number of protocols involving the generation of α-imino gold carbenes through gold-catalyzed amination reactions based on azides have been developed in the past decades owing to the ready availability of the azide precursors, environmental friendliness and good com-



Scheme 1. Amination reagents for gold-catalyzed intermolecular alkyne amination reactions *via* α-imino gold carbenes.

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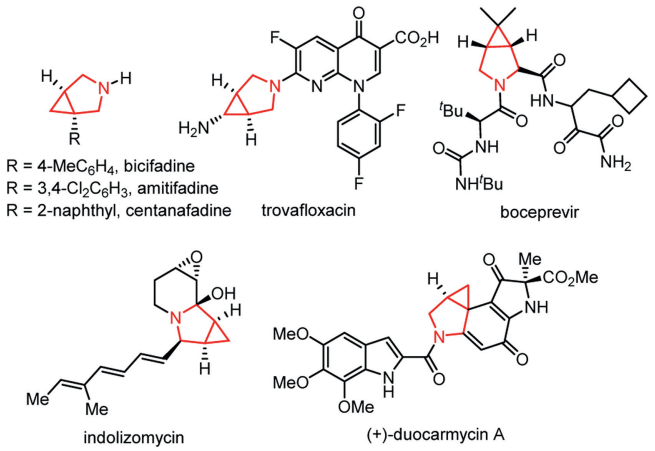


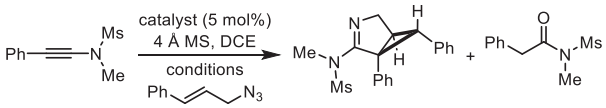
Fig. 1. 3-Azabicyclo[3.1.0] scaffold in pharmaceutical molecules and natural products.

patibility with functional groups of these reactions [53–68]. However, such protocols are only limited to intramolecular generation of α-imino gold carbenes probably due to the weak nucleophilicity of azides, and intermolecular generation of α-imino gold carbenes by the use of azides as amination reagents has not been evoked.

In 2015, our group described the first gold-catalyzed amination reactions by employing the benzyl azides and indolyl azides as a new type of amination reagents for the intermolecular generation of α-imino gold carbenes, leading to the facile formation of functionalized 2-aminoindoles and 3-amino-β-carbolines in generally high yields *via* formal [3 + 2] and [4 + 2] annulations, respectively [69,70]. On the basis of this work, in 2016, we further realized a gold-catalyzed intermolecular amination reaction initiated aza-Nazarov cyclization reaction based on 3-en-1-ynamides and a gold-catalyzed intermolecular tandem ynamide amination/C–H functionalization by both using the benzyl azides as amination reagents, thus allowing the flexible synthesis of synthetically useful 2-aminopyrroles and 2-aza-1,3-butadienes, respectively [71,72]. Inspired by the above results and by our recent study on developing ynamide chemistry for *N*-heterocycle synthesis [73–91], we envisioned that the use of allyl azides might act as amination reagents to react with ynamides for the intermolecular generation of alkene-tethered α-imino gold carbenes, followed by intramolecular cyclopropanation, eventually leading to valuable 3-azabicyclo[3.1.0] scaffold (Scheme 1b), which is widely existing in pharmaceutical molecules and natural products (Fig. 1) [92–101]. However, realizing this intermolecular annulation is highly challenging: (i) Whether the allyl azide is nucleophilic enough to inter-molecularly attack the ynamide; (ii) how to prevent the possible triazole formation *via* direct azide-alkyne [3 + 2] cycloaddition [102–105]; and (iii) how to control the regioselectivity and diastereoselectivity. Herein, we disclose such a gold-catalyzed ynamide amination initiated cyclopropanation by employing the allyl azide as amination reagent, thus representing the first use of allyl azide as a novel amination reagent. This protocol enables the efficient and flexible synthesis of a wide range of 3-azabicyclo[3.1.0]hex–2-enes in generally good to excellent yields. In addition, the formed 3-azabicyclo[3.1.0]hex–2-enes can be readily converted into valuable γ-lactams [106–108] through a facile hydrolysis of the amide moiety under the acidic conditions. Furthermore, a mechanistic rationale for this α-imino gold carbene-involved intermolecular annulation reaction is strongly supported by theoretical calculations.

We commenced our investigation by examining the reaction between readily prepared ynamide **1a** and allyl azide **2a** in DCE at

Table 1
Optimization of reaction conditions.[a]



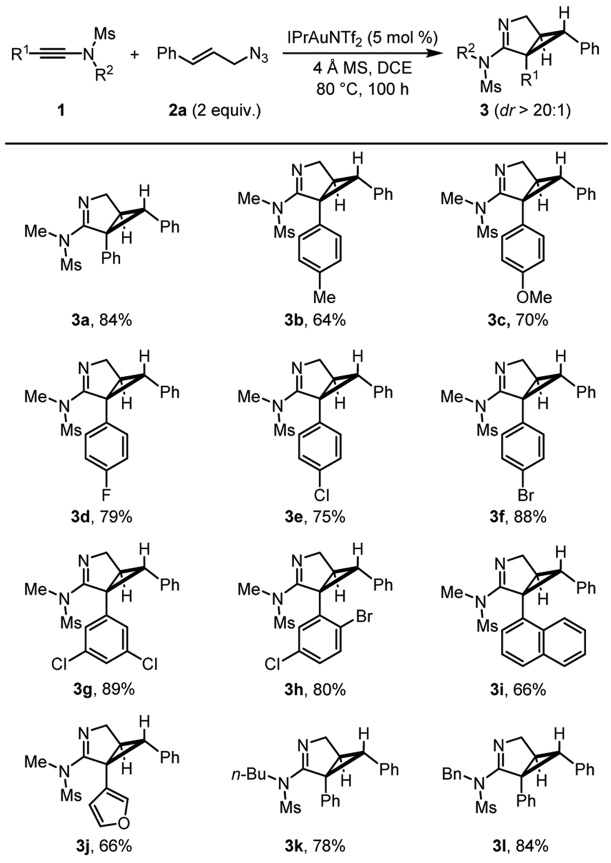
1a **2a** (2 equiv.) **3a** (*dr* > 20:1) **3aa**

Entry	Catalyst	Conditions	Yield (%)[b]		
			3a	**3aa**	**1a**
1	$IPrAuNTf_2$	r.t., 100 h	30	<5	60
2	$IPrAuNTf_2$	80 °C, 100 h	85	<5	<5
3[c]	$IPrAuNTf_2$	80 °C, 100 h	72	<5	<5
4[d]	$IPrAuNTf_2$	80 °C, 100 h	63	<5	<5
5	$BrettPhosAuNTf_2$	80 °C, 100 h	6	53	38
6[e]	Au(III)[c]	80 °C, 100 h	21	42	30
7	$Ph_3PAuNTf_2$	80 °C, 100 h	18	21	42
8	Cy-JohonPhosAuNTf$_2$	80 °C, 100 h	22	32	49
9	$[Rh(CO)_2Cl]_2$	80 °C, 0.5 h	<1	<1	<1
10	$AgNTf_2$	80 °C, 100 h	<1	53	45
11	$Zn(OTf)_2$	80 °C, 100 h	<1	74	20
12[f]	$IPrAuNTf_2$	80 °C, 100 h	85	<5	<5

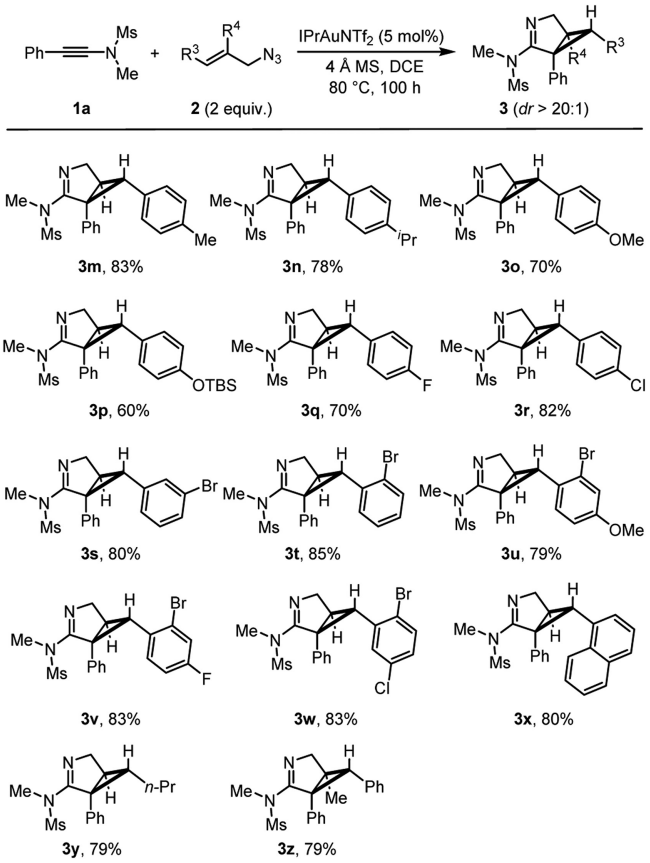
Ms = methylsulfonyl; DCE = 1,2-dichloroethane.
[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), catalyst (0.005 mmol), 0.1 mol/L, r.t.–80 °C, 0.5–100 h, 4 Å MS (20 mg), in vials.
[b] Yields are measured by ^1H NMR using diethyl phthalate as the internal standard.
[c] Toluene.
[d] MeCN.
[e] Dichloro(2-picolinato) gold(III).
[f] 10 mol% gold catalyst was used.

room temperature using $IPrAuNTf_2$ as gold catalyst. To our delight, the desired fused cyclopropanation product **3a** could be obtained in 30% yield with 60% of ynamide **1a** recovered (Table 1, entry 1). Encouraged by this preliminary attempt, we then increased the reaction temperature to 80 °C, and found that 3-azabicyclo[3.1.0]hex–2-ene **3a** could be obtained in 85% NMR yield with the full conversion of **1a** (Table 1, entry 2). Subsequently, solvent screening showed that typical solvents such as toluene and MeCN failed to improve the reaction yield (Table 1, entries 3 and 4). Various gold catalysts, including $BrettPhosAuNTf_2$, Au(III), $Ph_3PAuNTf_2$ and Cy-JohonPhosAuNTf$_2$, were then investigated but also failed to further improve the yield, and significant formation of hydrolysis product **3aa** was observed in these cases (Table 1, entries 5–8). Of note, only triazole formation (97%) *via* azide-alkyne [3 + 2] cycloaddition was observed under rhodium catalysis, which was similar to Huang's protocol (Table 1, entry 9) [102]. In addition, the use of other representative transition metal catalysts such as $AgNTf_2$ and $Zn(OTf)_2$ also failed to deliver the desired product **3a**, and hydrolysis product **3aa** was obtained as the main product in these cases (Table 1, entries 10 and 11). Finally, it was found that the reaction time could be shortened to 60 h by increasing the catalyst amount to 10 mol%, but no obvious improvement of the yield was observed (Table 1, entry 12). Importantly, excellent diastereoselectivities (> 20:1) were achieved in all these cases.

Having established the optimized reaction conditions (Table 1, entry 2), we next explored the substrate scope of this gold-catalyzed intermolecular amination reaction of ynamides with allyl azides. As shown in Scheme 2, we initially investigated the scope of a wide array of substituted ynamides **1** under the optimized conditions. It was found that ynamides with various substitutions at the para position of the phenyl ring were suitable substrates to furnish the corresponding 3-azabicyclo[3.1.0]hex–2-ene products **3a**–**3f** in 64%–88% yields, and ynamides bearing electron-deficient groups gave better yields than the ones containing the electron-rich groups on the aryl. Ynamides bearing the disubstituted aryl, such as 3,5-dichloro and 3–chloro-5–bromo substitu-



Scheme 2. Reaction scope of ynamides **1**. Reactions run in vials; **1** (0.3 mmol), **2a** (0.6 mmol), $IPrAuNTf_2$ (0.015 mmol), DCE (3 mL), 4 Å MS (60 mg), 80 °C, 100 h; yields are those for the isolated products.



Scheme 3. Reaction scope of allyl azides **2**. Reactions run in vials; **1a** (0.3 mmol), **2** (0.6 mmol), $IPrAuNTf_2$ (0.015 mmol), DCE (3 mL), 4 Å MS (60 mg), 80 °C, 100 h; yields are those for the isolated products.

tion, were well tolerated in this annulation, affording the desired 3-azabicyclo[3.1.0]hex–2-enes **3g** and **3h** in excellent yields. In addition, the reaction was also extended to the naphthyl and furyl substituted ynamides, and the expected cyclopropanation products **3i** and **3j** were formed in both of 66% yields. Finally, ynamides with diverse R^2 substituents (^{n}Bu, Bn) were able to undergo smooth annulation to deliver the desired products **3k** in 78% yield and **3l** in 84% yield, respectively. Prominently, excellent diastereoselectivities ($dr > 20:1$) were obtained in all these cases, and no triazole formation *via* azide-alkyne [3 + 2] cycloaddition was detected in such a gold catalysis.

Next, we explored the reaction scope of various allyl azides **2**. As depicted in Scheme 3, different aryl substituted allyl azides tethered with either electron-donating or -withdrawing substituents on different position of the phenyl ring, such as Me, ^{i}Pr, OMe, OTBS, F, Cl, and Br, were well tolerated in this intermolecular amination reaction with ynamide **1a** to generate the desired 3-azabicyclo[3.1.0]hex–2-ene products **3m**–**3t** in generally good to excellent yields. Besides, the reaction proceeded smoothly with disubstituted aryl allyl azides, providing the corresponding bicyclic cyclopropanation products **3u**–**3w** in 79%–83% yields. 1-Naphthyl-substituted allyl azide also provided an excellent universal fit for this annulation reaction, affording the expected product **3x** in 80% yield. Apart from the aryl substituted allyl azides, the alkyl substituted allyl azide was proved to be an appropriate substrate to produce the alkyl substituted cyclopropane compound **3y** in 79% yield. Finally, the allyl azide containing 1,2-disubstituted olefin moiety could also be smoothly converted into the corresponding multisubstituted 3-azabicyclo[3.1.0]hex–2-ene **3z** bearing two vicinal all carbon quaternary stereocenters which was difficult

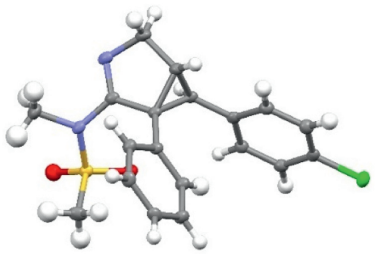
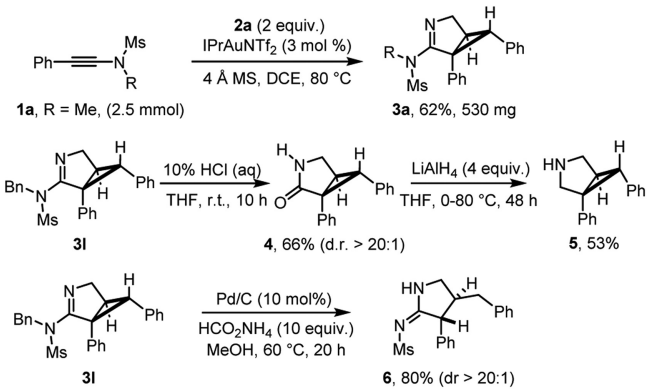


Fig. 2. Structure of compound **3r** in its crystal.

to construct by traditional methods in organic synthesis. Again, excellent diastereoselectivities ($dr > 20:1$) were achieved in all cases. The relative configuration of product **3r** was verified by X-ray crystallographic analysis and that of others was assigned analogously (Fig. 2) [106–108]. Thus, this intermolecular protocol provides a highly efficient and flexible route for the construction of valuable 3-azabicyclo[3.1.0] scaffold. Our attempts to extend the reaction to the chiral amine-derived ynamide for the asymmetric synthesis has been unsuccessful, and attempts to develop the direct asymmetric catalysis by employing Shi's chiral *N*-heterocyclic carbene (NHC)-derived chiral gold catalysts also failed [109–115].

Further preparative scale reaction and synthetic application of the as-synthesized 3-azabicyclo[3.1.0]hex–2-enes **3** were then explored (Scheme 4). Initially, preparative scale could be achieved for the synthesis of **3a** (530 mg, 62%) with a catalyst loading of 3 mol%. Then, synthetic applications of 3-azabicyclo[3.1.0]hex–2-ene **3l** were investigated. For example, hydrolysis of the amide moiety of 3-azabicyclo[3.1.0]hex–2-ene **3l** *via* the facile use of HCl (aq.) led



Scheme 4. Preparative scale reaction and synthetic application.

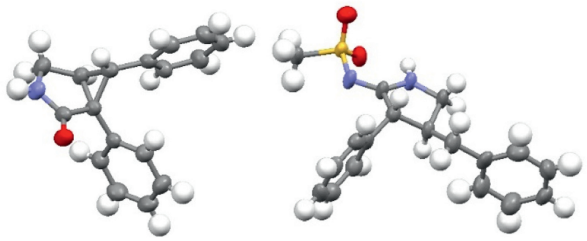
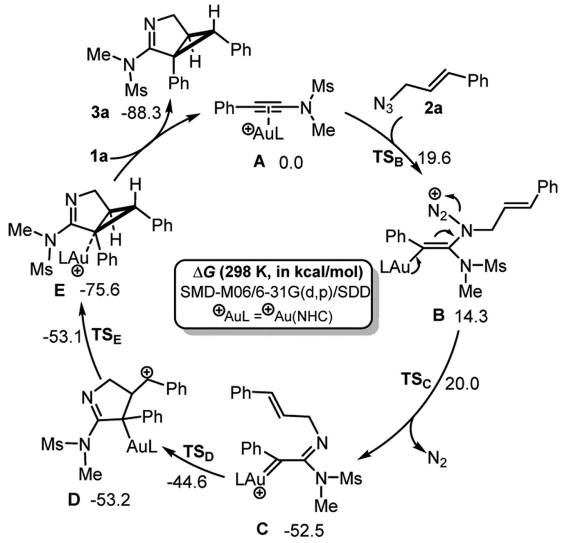


Fig. 3. Structure of compounds **4** (left) and **6** (right) in its crystal.



Scheme 5. Plausible reaction mechanism.

to the cyclopropane-fused γ-lactam **4** in 66% yield, and subsequent reduction could afford pyrrolidine **5**, which is broadly existed in natural products and bioactive molecules [106–108]. Moreover, **3l** could be readily transferred into the corresponding pyrrolidine **6** in 80% yield with excellent diastereoselectivity (>20:1) *via* selective ring opening of cyclopropane and debenzylation process under the reductive condition by using Pd/C and HCO_2NH_4. The structures of γ-lactam **4** and pyrrolidine **6** were confirmed by X-ray diffraction analysis (Fig. 3).

On the basis of our previous studies and density functional theory (DFT) calculations [69–72,109], a plausible reaction mechanism for this gold-catalyzed intermolecular amination of ynamides with allyl azides is proposed. As shown in Scheme 5, ynamide **1a** is first activated by the gold catalyst to afford intermediate **A**, which undergoes regioselective attack by the allyl azide **2a** *via* transition state $\mathbf{TS_B}$ with a free energy barrier of 19.6 kcal/mol, leading to the formation of vinyl gold intermediate **B**. Subsequently, intermediate **B** overcomes a free energy barrier of 5.7 kcal/mol *via* $\mathbf{TS_C}$ with a release of N_2, resulting in the generation of α-imino gold carbene intermediate **C**. Then, an intramolecular attack occurs between the gold carbene and the alkene moiety within intermediate **C** to deliver the carbocation intermediate **D** with a free energy barrier of 7.9 kcal/mol *via* $\mathbf{TS_D}$. Subsequent intramolecular cyclopropanation of the intermediate **D** occurs almost without barriers to yield the cyclopropane intermediate **E**. Finally, intermediate **E** gives rise to the desired 3-azabicyclo[3.1.0]hex–2-ene **3a** *via* a substrate exchange. The whole process proceeds smoothly and highly exergonically with a release of free energy amounting to 88.3 kcal/mol.

In summary, we have developed an efficient gold-catalyzed intermolecular amination reaction of ynamides with allyl azides, leading to the flexible synthesis of a range of synthetically useful 3-azabicyclo[3.1.0]hex–2-ene derivatives in good to excellent yields with excellent diastereoselectivities. Importantly, this protocol represents the first use of allyl azide as an efficient amination reagent in gold-catalyzed alkyne amination reactions. Of note, the as-synthesized 3-azabicyclo[3.1.0]hex–2-enes can be readily converted into valuable γ-lactams *via* hydrolysis of the amide moieties under the acidic conditions, thus fulfilling the facile removing of the directing group of ynamides. Moreover, theoretical calculations provide further evidence for the proposed α-imino gold carbene-involved mechanism. Further investigation of the asymmetric version of this annulation and other related intermolecular alkyne aminations are ongoing in our lab.

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.

CRediT authorship contribution statement

Chong-Yang Shi: Writing – original draft, Validation, Investigation, Data curation. **Jian-Xing Gong:** Validation, Investigation, Data curation. **Zhen Li:** Investigation, Data curation. **Chao Shu:** Investigation, Data curation. **Long-Wu Ye:** Writing – review & editing, Writing – original draft, Validation, Funding acquisition, Conceptualization. **Qing Sun:** Writing – review & editing, Funding acquisition. **Bo Zhou:** Writing – review & editing. **Xin-Qi Zhu:** Writing – review & editing, Conceptualization.

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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.2024.109895.

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