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Accessing polyarene-fused ten-membered lactams *via* oxidative *N*-heterocyclic carbene (NHC)-catalyzed high-order [7 + 3] annulation

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ABSTRACT

A new oxidative *N*-heterocyclic carbene (NHC)-catalyzed high-order [7+3] annulation reaction of γ -indolyl phenols as 1,7-dinucleophiles and α,β -alkynals with the aid of Sc(OTf)₃ is reported, enabling the highly regioselective access to unprecedented polyarene-fused ten-membered lactams bearing a bridged aryl-aryl-indole scaffold in moderate to good yields. This protocol demonstrates a broad substrate scope, good compatibility with substituents and complete regioselectivity, providing an organocatalytic modular synthetic strategy for creating medium-sized lactams.

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Medium-sized rings (MSRs), namely, 8–11-membered cyclic systems, are widely distributed in a wide range of important natural products, bioactive substances and other industrially relevant compositions of matter [1–7]. In direct comparison with smaller or larger-sized rings, medium-sized rings, because of their inherent structural rigidity and disparity in three-dimensional spatial properties, demonstrate special characteristics in binding affinities to biological receptors or cell permeability [8]. Remarkably, molecules incorporating MSRs are widely applied for pharmaceutical purposes because they exhibit a broad spectrum of biological activities [9–11]. Among MSRs, ten-membered lactams are highly valuable molecules in both academia and industry and play a significant role in modern organic chemistry [12–20], due to their occurrence in nature (Fig. 1) [21,22] and versatile utility as synthetic intermediates [23]. These contributions have been observed in ten-membered lactams, which has intensified interest in their synthetic accessibility. As a consequence of unfavorable factors associated with torsional and transannular strain as well as competing pathways preferring the formation of normal rings [24–27], the synthesis of ten-membered lactams is rather challenging. In this regard, there are only limited examples, which mainly focus on intramolecular photocyclization [28,29], ring-closing olefin

metathesis [30,31], ring expansion [32–34], the Ugi reaction [35] and recent palladium-catalyzed decarboxylative [5+5] cyclization [36]. Despite these limited advances, the development of novel catalytic strategies for the modular and viable construction of ten-membered lactam skeletons is in great demand, considering the significance of enriching synthetic toolboxes to access MSRs, as well as the potential for downstream studies utilizing these lactams to find new bioactive lead compounds in pharmaceutical chemistry.

In recent decades, oxidative *N*-heterocyclic carbene (NHC) catalyzed annulations have been recognized as powerful synthetic tools for unconventional access to heterocyclic targets [37–46], which often involve several commonly used catalytic cyclization modes, including formal [2+2] [47–49], [3+2] [50–52], [3+3] [43,53–55], [4+2] [56–59], and [4+3] [60–63] annulation *via* α,β -unsaturated/alkynyl acylazoliums, to access small- to normal-sized and seven-membered rings under oxidative conditions (Scheme 1a). In sharp contrast, NHC-catalyzed high-order cyclization to form MSRs (8–11-membered rings), especially those with ten-membered lactam motifs, has rarely been investigated [64,65]. To continue our interest in high-order cyclization cascades [66–68], we envisioned that privileged polyarene-fused ten-membered lactams could be synthesized through an NHC-catalyzed [7+3] annulation reaction starting from suitable γ -indolyl nucleophiles bearing 1,7-dinucleophilic sites and α,β -alkynals as alkynyl acylazolium precursors (Scheme 1b). Such a catalytic approach may suf-

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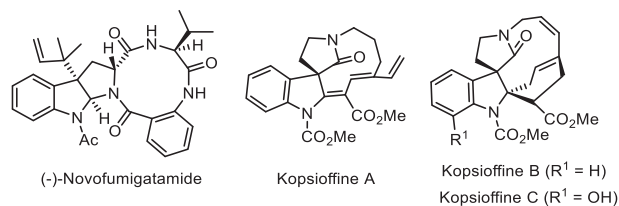
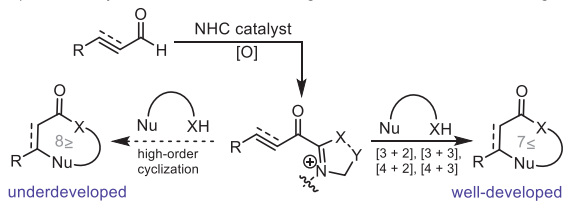
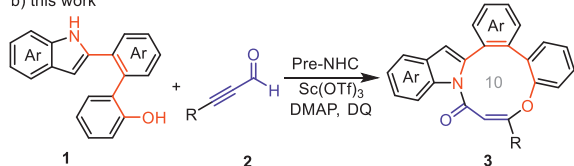


Fig. 1. Representative fused ten-membered lactams.

a) NHC-catalyzed annulations for forming five-, six-, seven-membered rings



b) this work



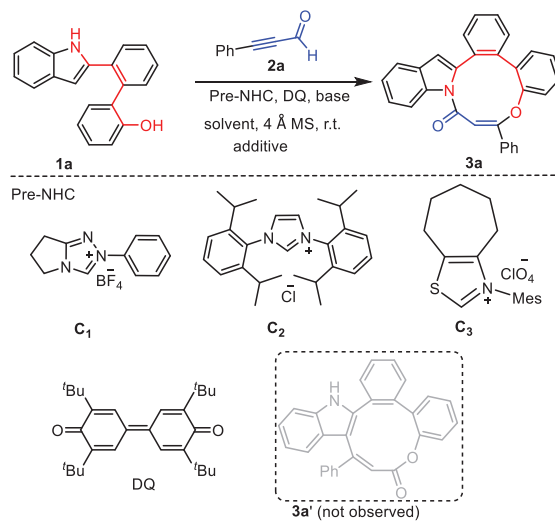
Scheme 1. Access to polyarene-fused ten-membered lactams.

fer from problems in the choice of 1,7- or 1,3-dinucleophilic indolyl substrates and control of regioselectivity. Herein, we report an organocatalytic high-order [7 + 3] annulation reaction of γ -indolyl phenols with α,β -alkynals, leading to the regioselective synthesis of unprecedented polyarene-fused ten-membered lactams bearing two vicinal chiral axes in moderate to good yields. Notably, the current high-order annulation reaction features complete regioselectivity, enabling the direct fabrication of rotationally hindered bridged aryl-aryl-indole scaffolds.

Initially, γ -2-indolyl phenol **1a** and α,β -alkynal **2a** were chosen as model substrates for screening the reaction conditions (Table 1). The catalytic transformation of **1a** with **2a** was investigated under NHC catalysis using 3,3',5,5'-tetra-*tert*-butyldiphenylquinone (DQ) as the oxidant (Table 1). We were encouraged to find that the reaction using the triazolium salt **C₁** as an NHC precursor and DBU as a base in the presence of Sc(OTf)₃ provided the desired pentacyclic ten-membered lactam **3a** in 65% yield without observing its regioisomer **3a'**, revealing that the reaction demonstrates complete regioselectivity (entry 1). Based on these preliminary results, various reaction parameters, such as the NHC source, base, solvent and additive, were then carefully investigated. Exchanging triazolium salt **C₁** with imidazolium salt **C₂** or thiazolium salt **C₃** returned inferior yields of **3a** (entries 2 and 3). After screening several bases commonly used in NHC catalysis, 4-dimethylaminopyridine (DMAP) was found to be the best choice for this transformation (80%, entry 5), as reactions involving other organic (Et₃N) and inorganic (Cs₂CO₃ and K₂CO₃) bases were tested but resulted in traces or no desired products (entries 4, 6 and 7). The application Mg(OTf)₂ and Zn(OTf)₂ as Lewis acid catalysts could make this reaction to proceed readily; however, the yields of **3a** obtained with both catalysts remain inferior to those achieved with Sc(OTf)₃ (entries 8 and 9 vs. entry 5). Next, the effect of the solvents was evaluated. The yields of product **3a** associated with Pre-NHC **C₁** in the presence of Sc(OTf)₃ and DMAP in several solvents are summarized as follows (entries 10–15): MeCN (60%), ethyl acetate (EA, 67%), toluene (58%), 1,2-dichloroethane (DCE, trace), and 1,4-dioxane (54%), indicating that none of them

Table 1

Screening of reaction conditions for the reaction of **1a** with **2a**.^a



Entry	Cat.	Base	Additive (mol%)	Solvent	Yield (%) ^b
1	C₁	DBU	Sc(OTf) ₃ (20)	THF	65
2	C₂	DBU	Sc(OTf) ₃ (20)	THF	Trace
3	C₃	DBU	Sc(OTf) ₃ (20)	THF	40
4	C₁	Et ₃ N	Sc(OTf) ₃ (20)	THF	NR
5	C₁	DMAP	Sc(OTf) ₃ (20)	THF	80
6	C₁	Cs ₂ CO ₃	Sc(OTf) ₃ (20)	THF	Trace
7	C₁	K ₂ CO ₃	Sc(OTf) ₃ (20)	THF	NR
8	C₁	DMAP	Mg(OTf) ₂ (20)	THF	72
9	C₁	DMAP	Zn(OTf) ₂ (20)	THF	66
10	C₁	DMAP	Sc(OTf) ₃ (20)	MeCN	60
11	C₁	DMAP	Sc(OTf) ₃ (20)	EA	67
12	C₁	DMAP	Sc(OTf) ₃ (20)	Toluene	58
13	C₁	DMAP	Sc(OTf) ₃ (20)	DCE	Trace
14	C₁	DMAP	Sc(OTf) ₃ (20)	1,4-Dioxane	54
15	C₁	DMAP	Sc(OTf) ₃ (10)	THF	49
16 ^c	C₁	DMAP	Sc(OTf) ₃ (20)	THF	56
17	C₁	DMAP	–	THF	57
18 ^d	C₁	DMAP	Sc(OTf) ₃ (20)	THF	Trace
19 ^e	C₁	DMAP	Sc(OTf) ₃ (20)	THF	NR
20	–	DMAP	Sc(OTf) ₃ (20)	THF	NR

^a Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.6 mmol, 3.0 equiv.), DQ (3.0 equiv.), Pre-NHC (15 mol%), base (2.0 equiv.), 4 Å MS (200 mg), Sc(OTf)₃ (20 mol%), dry solvent (4 mL) at 25 °C for 4 d. Mes = mesityl(2,4,6-trimethylphenyl).

^b Isolated yield of **3a**.

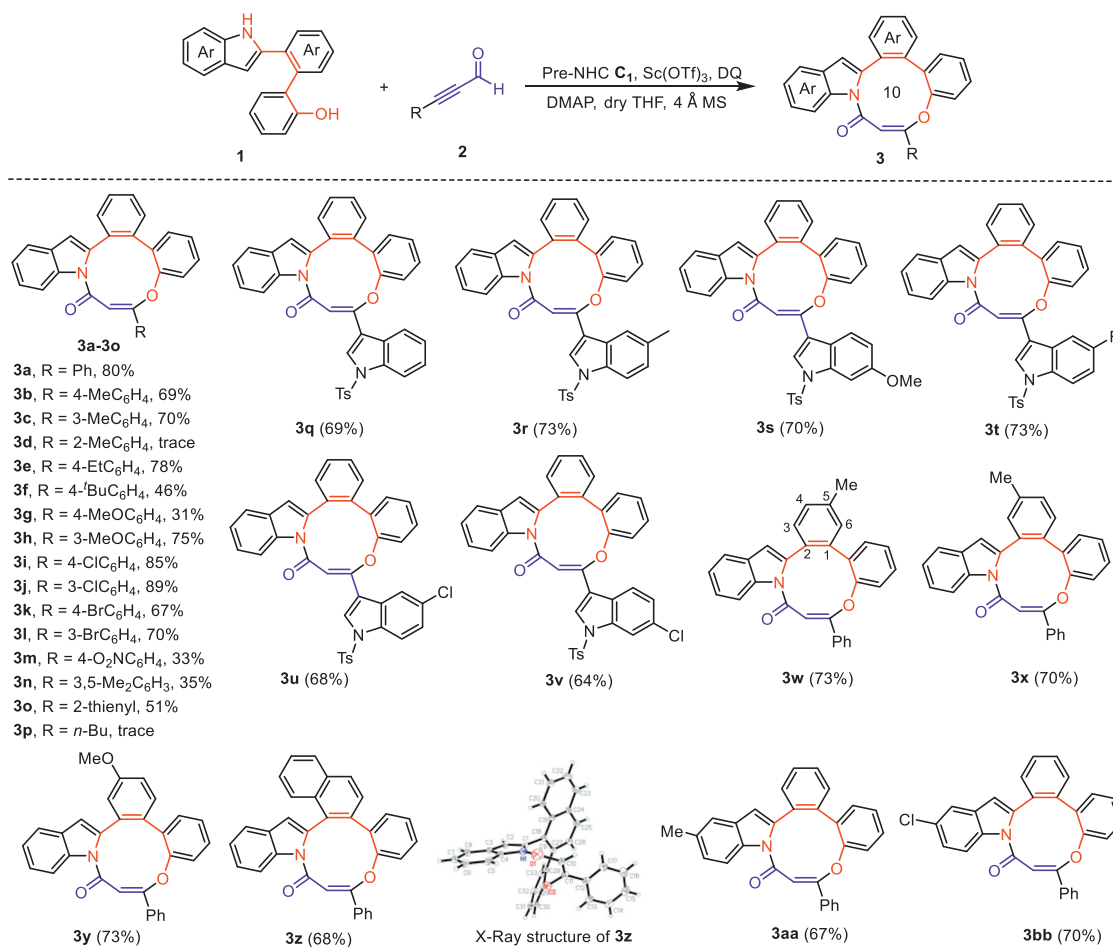
^c Use of DMAP (1.0 equiv.).

^d MnO₂ or DDQ instead of DQ.

^e Without DQ.

can improve the efficiency of this transformation as compared with THF. Decreasing the amount of Sc(OTf)₃ (10 mol%) or DMAP (1.0 equiv.) resulted in a markedly reduced yield of **3a** (entries 15 and 16). Without Sc(OTf)₃, the yield of **3a** decreased remarkably to 57%, showing that Sc(OTf)₃ plays an important role in increasing the yield of **3a** (entry 17). The reaction did not proceed with the use of MnO₂ or 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) as the oxidant (entry 18). Control experiments carried out without the NHC precursor or DQ did not yield **3a**, indicating the indispensable role of the carbene catalyst as well as the oxidant in this reaction (entries 19 and 20).

With the optimized reaction conditions (as stated in Table 1, entry 5), we sought to examine the generality and limitations of the organocatalytic high-order annulation with respect to γ -indolyl phenols and α,β -alkynals. The results are summarized in Scheme 2. First, the electronic properties and positions of the substituents on the arene ring of α,β -alkynals were carefully surveyed by repeating the reactions of **1a**, which afforded a wide range of pentacyclic ten-membered lactams **3b–3o** with good func-



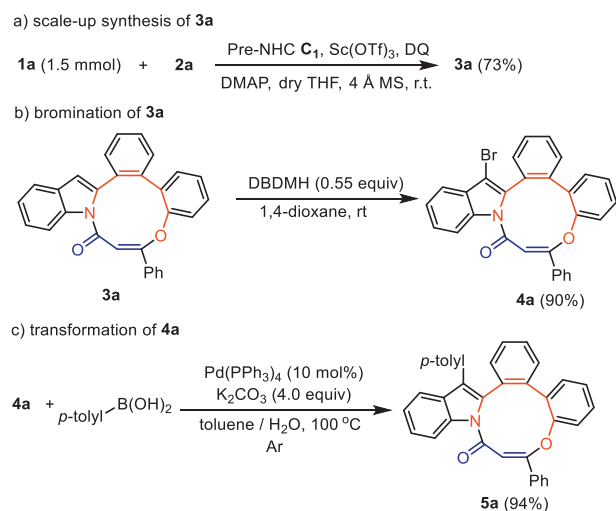
Scheme 2. Substrate scope for the synthesis of products **3**. Reactions were conducted with **1** (0.20 mmol), **2** (0.60 mmol), DQ (3.0 equiv.), Pre-NHC **C**₁ (15 mol%), DMAP (2.0 equiv.), 4 Å MS (200 mg), Sc(OTf)₃ (20 mol%), and dry THF (4 mL) at 25 °C under air conditions for 4 days. Yield of the isolated products based on substrate **1**.

tional group compatibility and complete regioselectivity. The reaction proceeded readily with various substituents, such as methyl (**2b**, **2c** and **2d**), ethyl (**2e**), *tert*-butyl (**2f**), methoxy (**2g** and **2h**), chloro (**2i** and **2j**), bromo (**2k** and **2l**) and nitro (**2m**) at the *para*- or *meta*-positions of the phenyl ring. Among them, a substrate bearing a strong electron-donating (methoxy **2g**) group at the *para*-position seems reluctant to undergo this process, as demonstrated by the generation of product **3g** in 31% yield, which may be caused by unfavorable Michael addition of the hydroxyl group of γ -indolyl phenols to α,β -alkynals when a strong electron donating group exists in the latter. Moreover, an inferior outcome was observed (**3m**, 33%) when a strong electron-withdrawing group, such as nitro (**2m**) was used. The presence of a nitro group is detrimental to the yield of the product, probably due to its ability to greatly enhance the reactivity of α,β -alkynals, increasing the complexity of the reaction. In addition to the mono-substituent in the phenyl ring of α,β -alkynals, this protocol is also applicable for disubstituted counterparts, such as 3,5-dimethyl (**2n**), albeit with a moderate yield. Moreover, the thienyl-incorporating substrate **2o** was possible in this transformation, affording product **3o** in 51% yield. However, *n*-butyl substituted α,β -alkynal **2p** was not suitable for this transformation.

Subsequently, swapping the phenyl group with the *N*-Ts indolyl functionality on the α,β -alkynal moiety enabled regioselective access to a series of unprecedented pentacyclic ten-membered lactams **3q-3v** with yields ranging from 64% to 73%. Specifically, the substituent variation in the *N*-Ts protected indole ring of substrate **2** was briefly investigated by combining γ -indolyl phenol

1a under standard conditions. Different substituents, such as C5-methyl (**2r**), C6-methoxy (**2s**), C5-fluoro (**2t**), and chloro (C5, **2u**; C6, **2v**) were tested, and all of these compounds performed well to further demonstrate the compatibility of this transformation. Next, a brief investigation was conducted on the possible structural changes in the indole ring and the internal arene ring of the γ -indolyl phenols. For the internal arene ring, both methyl (**1b** and **1c**) and methoxy (**1d**) groups at the C4- or C5-position were accommodated in this transformation, delivering corresponding products **3w-3z** as single regioisomers in 68%–73% yields. As exemplified by product **3y**, this organocatalytic high-order annulation is also adaptable to γ -indolyl phenol **1e** anchored by a sterically crowded naphthalene linkage in good yield. For the indole ring, substituents, such as methyl (**1f**) and chloro (**1g**), at the C5 position were compatible with this catalytic system, giving pentacyclic ten-membered lactams **3aa** and **3bb** in 67% and 70% yields, respectively. In the case of **3z**, its structure was unambiguously determined by X-ray diffraction analysis (CCDC 2372468, see Supporting information).

To demonstrate the synthetic utility of this transformation, a scale-up reaction of **1a** with **2a** was conducted on a 1.5 mmol scale, leading to a slightly decreased yield of product **3a** (73%) (Scheme 3a). Next, the bromination of **3a** in the presence of 1,3-dibromo-5,5-dimethylhydantoin (DBDMH) gave brominated pentacyclic ten-membered lactam **4a** in 90% yield (Scheme 3b). The Suzuki–Miyaura cross-coupling reaction of **4a** with *p*-tolylboronic acid was conducted by using Pd(PPh₃)₄ as the catalyst, affording product **5a** in 94% yield (Scheme 3c) [69].



Scheme 3. Scale-up reaction and bromination of **3a**.

Based on the control experiments (Table 1, entries 17, 19 and 20) and previous reports [70,71], a plausible mechanism is proposed in Scheme 4. With γ -indolyl phenol **1a** and α,β -alkynal **2a** as representative examples, this process initially involves *in-situ*-generation of a free NHC catalyst from Pre-NHC **C₁** under basic conditions, and the subsequent addition of the NHC catalyst to **2a** generates Breslow intermediate **I**, which is oxidized by DQ in the presence of $\text{Sc}(\text{OTf})_3$ to form alkynyl acyl azolium-[Sc]-complex **II**. The Michael addition of the oxygen anion of γ -indolyl phenolic anion **III**, derived from **1a** under basic conditions, to intermediate **II** affords allenolate intermediate **IV**, which undergoes proton transfer (PT) and subsequent lactamization to afford product **3a** and simultaneously regenerates the NHC catalyst and [Sc]-complex for the next catalytic cycles.

In summary, we have illustrated a new oxidative NHC catalytic high-order [7+3] annulation strategy using γ -indolyl phenols as 1,7-dinucleophiles and α,β -alkynals with the aid of $\text{Sc}(\text{OTf})_3$, producing a wide range of unprecedented polyarene-fused ten-

membered lactams in moderate to good yields with complete regioselectivity. The reaction proceeds under mild conditions via NHC catalysis, is highly regioselective for constructing polycyclic medium-sized lactams incorporating vicinal biaxially chiral bridged aryl-aryl-indole motif and shows good substrate scope around each of the reacting functional groups. Further investigations to apply this reaction to biologically active targets and its asymmetric version are currently underway in our laboratory.

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

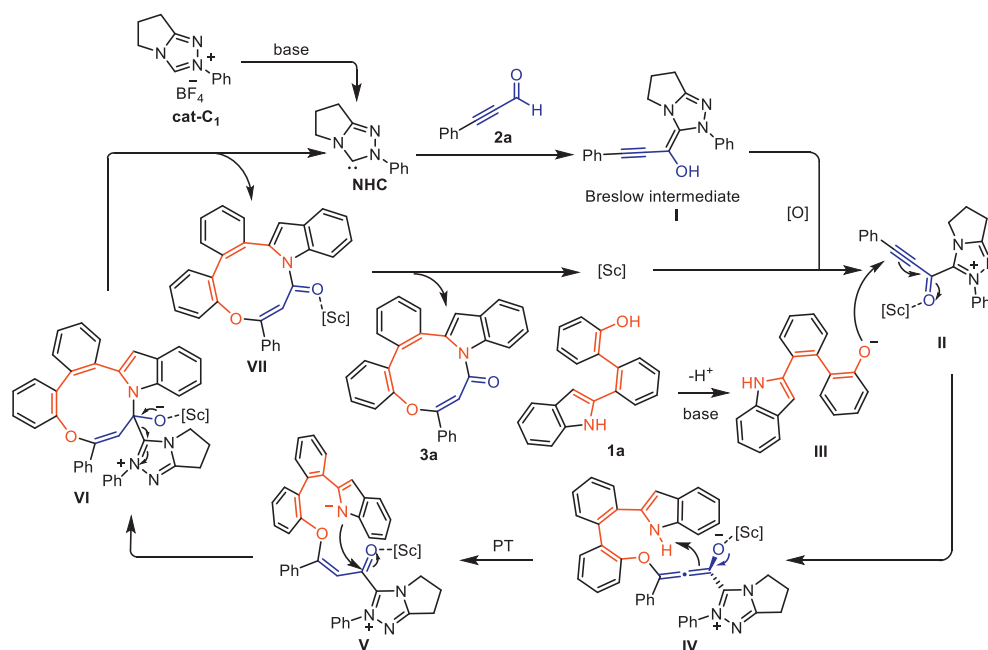
Chen-Chang Cui: Writing – original draft, Investigation, Formal analysis, Data curation. **Shao-Qing Shi:** Investigation, Formal analysis. **Lu-Yao Wang:** Data curation. **Feng Lin:** Investigation, Data curation. **Man-Su Tu:** Supervision, Project administration, Investigation, Funding acquisition, Formal analysis. **Wen-Juan Hao:** Writing – review & editing, Supervision, Project administration. **Bo Jiang:** Writing – review & editing, Supervision, Methodology, Funding acquisition.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2024.110541.



Scheme 4. Proposed reaction pathway.

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