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# Hypervalent Iodine Reagents Enable C(sp$^2$)−H Amidation of (Hetero)arenes with Iminophenylacetic Acids

Yue Pan,[▽] Zhengyi Liu,[▽] Peng Zou, Yali Chen, and Yiyun Chen*

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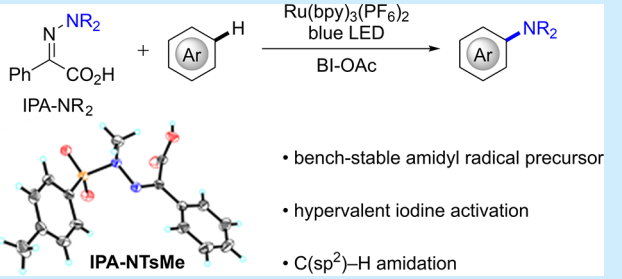
**ABSTRACT:** Sulfonamide-containing (hetero)arenes are widely present in bioactive molecules. Here, we report the sulfonamidyl (hetero)arenes synthesis by the C(sp$^2$)−H amidation from bench-stable amidyl-iminophenylacetic acids. The hypervalent iodine reagents covalently activated iminophenylacetic acids for the facile sulfonamidyl radical generation under mild photocatalytic oxidative conditions. Diversified indoles, pyrroles, imidazopyridines, and fused arenes underwent the C(sp$^2$)−H amidation with excellent chemoselectivity and regioselectivity. This reaction performs well under neutral aqueous conditions with potential biological applications.




Sulfonamides are widely present in many bioactive molecules, including drug molecules such as Rosuvastatin, Sulthiame, and Sulfamethoxazole (Scheme 1a).[1] The C(sp$^2$)−H amidation is useful to introduce amidyl groups to (hetero)arenes for the construction of nitrogen-containing compounds.[2] Among those C−H amidation methods, the Minisci-type radical amidation is attractive by providing complementary reactivity to transition-metal catalysis.[3] Compared to the amidyl radical generation from amides, the use of prefunctionalized amidyl radical precursors generated amidyl radicals under mild redox conditions,[4] which has improved functional group compatibility and will help expand the synthetic utilities including biomolecule-compatible applications.[5] Among those diversified prefunctionalized precursors, the use of *N*-chloroamides, oxime esters, *O*-aryloxy amides, and pyridinium salts has enabled the amidyl radical generation by single-electron reduction for C(sp$^2$)−H amidations (Scheme 1b).[6] However, the C(sp$^2$)−H amidation of (hetero)arenes from single-electron-oxidizable precursors remains underexplored.[7]

Hypervalent iodine reagents acetoxylbenziodoxole (BI-OAc) demonstrate mild single-electron oxidation reactivity and transition-metal-like property in photoredox catalysis, which enabled the carboxylates and alcohols oxidation under mild photo oxidative conditions.[8] We recently discovered that BI-OAc activated alkoxy-iminophenylacetic acids (IPA-OR) by forming the BI-IPA-OR complex, which reduced the oxidation potential significantly and enabled the alkoxyl radical generation via the N−O bond cleavage for the selective C−H alkynylation (Scheme 1c).[9] We speculate that the hypothetical coordination of BI-OAc to amidyl-iminophenylacetic acids (IPA-NR$_2$) may also yield the amidyl radical via the N−N bond cleavage under mild single-electron-oxidation conditions.[9,10] In this Letter, we report the development of amidyl-iminophenylacetic acids to generate sulfonamidyl radicals by hypervalent iodine(III) activation for the selective C(sp$^2$)−H sulfonamidation of (hetero)arenes.

We started the investigation by preparing amidyl-iminophenylacetic acids IPA-NTsMe **1** in 76% yield in 10 grams scale, which purification was simple filtration and recrystallization from the reaction mixtures of methyl benzoylformates and tosyl hydrazines (Scheme 2).[11] The IPA-NTsMe **1** could be stored on the benchtop for an extended time without decomposition. The X-ray crystallography (CCDC No. 2009399) indicated the exclusive *Z*-isomer, which was stable in the solid state and underwent very slow isomerization in solutions.

With *N*-phenylindole **2** as the radical acceptor and Ru(bpy)$_3$(PF$_6$)$_2$ as the photocatalyst ($E^{III/II}$ = +1.29 V vs SCE in MeCN),[12] IPA-NTsMe **1** gave the desired amidation adduct **3** in 78% yield, with acetoxylbenziodoxole BI-OAc as the additive (Table 1, entry 1). IPA-NTsMe **1** underwent the hypothetical decarboxylation and phenylnitrile elimination to yield the sulfonamidyl radical, which was further added to *N*-phenylindole **2** to obtain the 2-sulfonamide-substituted phenylindole (**3**). The use of acetonitrile as the solvent gave an optimal 84% yield of **3** (79% isolated yield, Table 1, entry 2). Other cyclic iodine reagents such as methoxylbenziodoxole BI-OMe were also applicable to give 75% yield of **3** (Table 1, entry 3). However, the noncyclic iodine PIFA or the strongly

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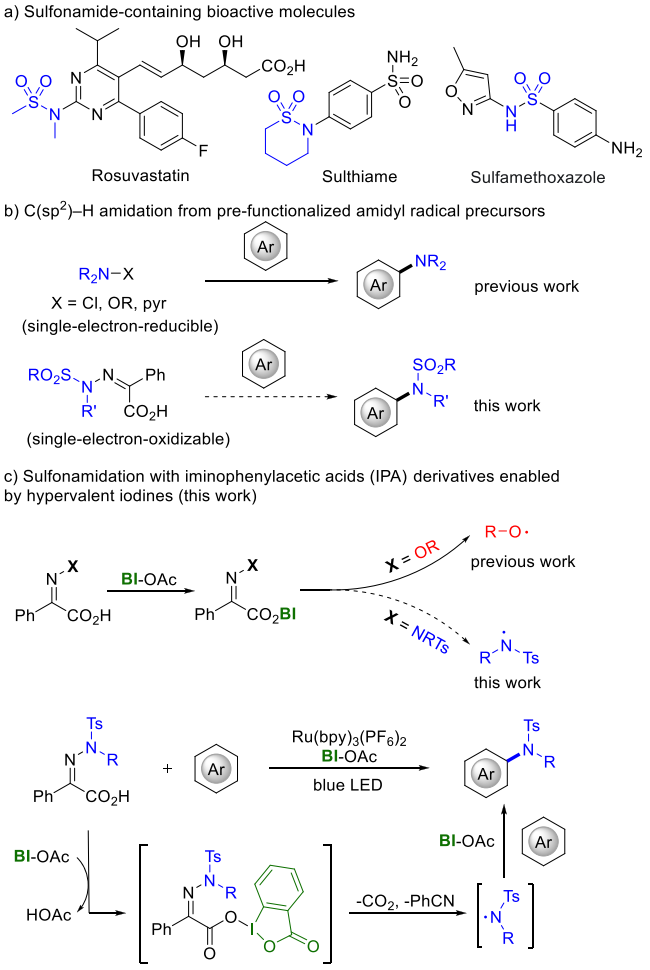
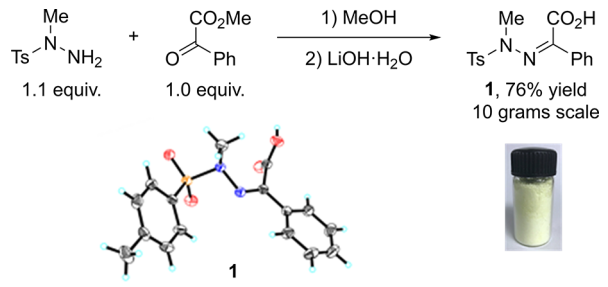
**Scheme 1. Hypervalent Iodine(III) Reagents Enable Amidation of (Hetero)arene with Iminophenylacetic Acids**



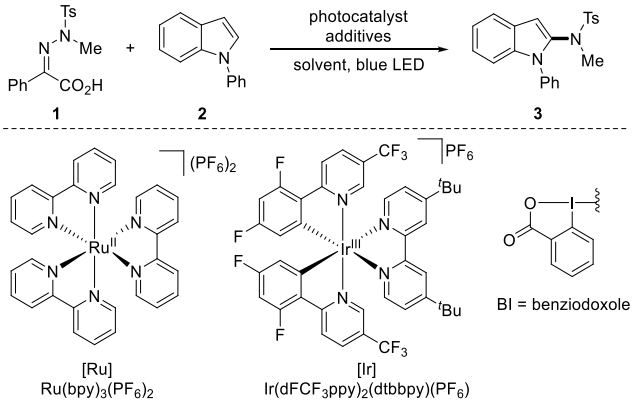

**Scheme 2. Synthesis and Characterization of Tosyl Iminophenylacetic Acid 1**




oxidative 2,3,4,5-F–BI-OH did not give **3** with the consumption of *N*-phenylindoles **2** (Table 1, entries 4 and 5). The change of photocatalysts to Ir(dFCF$_3$ppy)$_2$(dtbbpy)-PF$_6$ ($E^{IV/III}$ = +1.69 V) with higher oxidation potentials did not benefit the reaction to give only 9% yield of **3** (Table 1, entry 6).[13] The addition of acids or inorganic bases did not affect the reaction (see the Supporting Information for details). The photocatalyst, BI-OAc, and light irradiation were all critical for the reaction from control experiments (Table 1, entries 7–9).[14]

The substrate scope of the heterocycle amidation was explored next (Scheme 3). The electron-rich or electron-deficient groups on indoles did not affect the reaction to give

**Table 1. Optimization of the (Hetero)Arene Amidation with Iminoacetic Acids**




| entry | conditions[a] | additives | **2** conversion (%) | yield[b] (%) |
|---|---|---|---|---|
| 1 | [Ru], DCM | BI-OAc | 62 | 78 |
| 2 | [Ru], $CH_3CN$ | BI-OAc | 68 | 84 (79) |
| 3 | entry 2 | BI-OMe | 66 | 75 |
| 4 | entry 2 | PIFA | 90 | <5 |
| 5 | entry 2 | 2,3,4,5-F–BI-OH | 90 | <5 |
| 6[c] | [Ir], $CH_3CN$ | $K_2S_2O_8$ | >95 | 9 |
| 7 | entry 2, no [Ru] | BI-OAc | 0 | 0 |
| 8 | entry 2 | N/A | <5 | 0 |
| 9 | entry 2, no *hv* | BI-OAc | 0 | 0 |

[a]Reaction conditions: IPA-NTsMe **1** (0.10 mmol, 1.0 equiv), *N*-phenylindole **2** (0.20 mmol, 2.0 equiv), photocatalyst (0.002 mmol, 0.02 equiv), and BI-OAc (0.20 mmol, 2.0 equiv) in 2.0 mL acetonitrile under nitrogen with 4 W blue LED irradiation at 25 °C for 12 h, unless otherwise noted. [b]Yields were determined by $^1$H NMR analysis using 1,3,5-trimethoxybenzene as the internal standard, and isolated yields were in parentheses. [c]$Na_2CO_3$ (0.20 mmol, 2.0 equiv) was added. BI = benziodoxole.

2-sulfonamide-substituted indoles **4**–**10** in yields of 63%–75%, even the nitro group could be tolerated under the reaction conditions to give **5** in a 73% yield.[15] The electron-deficient N-aryl substituents benefited the reaction to give **11**–**14** in yields of 73%–77%, and the substrates bearing *N*-methoxylaryl substituents gave **15** and **16** in yields of 57%–62%. The 3-nitrile-substituted indole afforded **17** in 57% yield, and the *N*-phenylpyrrole reacted regioselectively at the 2-position to give the 2-sulfonamide-pyrrole **18** in 62% yield. The imidazopyridines with the 2-phenyl substitution gave the 3-sulfonamide-imidazopyridines **19**–**21** in yields of 53%–73%. The imidazopyridines exist in various selective inhibitors of excitatory amino acid transporter subtype 3, and sulfonamide-substituted imidazopyridines **21** could be synthesized in 72% yield in 1.37 g.[16] The reaction also worked on fluoranthenes and naphthalenes at the electron-rich positions regioselectively to give **22**–**24** in yields of 53%–62%. However, this reaction did not work on simple benzenes, pyridines, or isoquinolines due to the electrophilic characters of the sulfonamidyl radicals, and the hydrogenation adduct TsNHMe was observed as the main side product from **1**. The sulfonamide substituents on the IPA with different electronic properties were all tolerated to give **25**–**27** in yields of 58%–77%. Ethyl-substituted IPA-NTsEt **28a** worked for the reaction to afford **28** in 54% yield. We also tested the amidyl precursors with the benzoyl protecting group, but only a trace amount of desired product was detected (see the Supporting Information for details).



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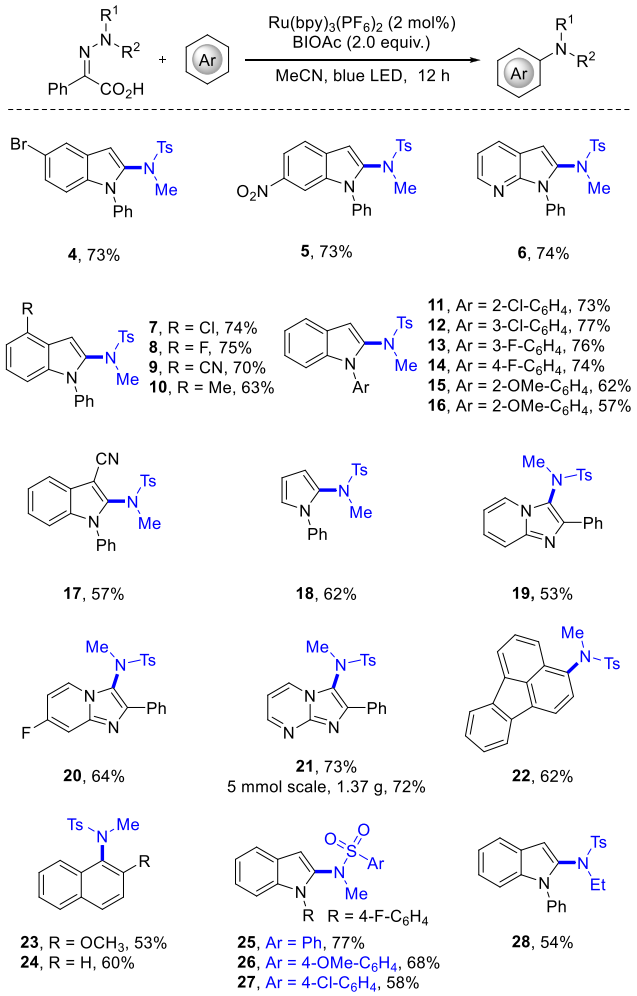
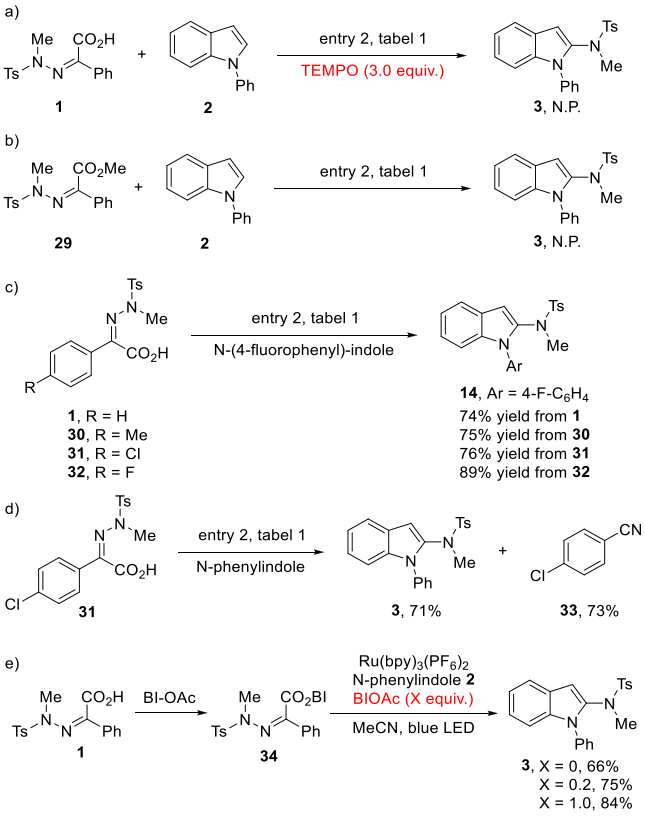
**Scheme 3. Substrate Scope of (Hetero)Arene Amidation with Amidyl-Iminophenylacetic Acids**




[a]For reaction conditions, see entry 2 of Table 1. Isolated yields were reported.

We next performed the mechanistic investigations on sulfonamidyl-iminophenylacetic acids for the (hetero)arene amidation. When TEMPO was added to the reaction as the radical quencher, the formation of **3** was completely inhibited (Scheme 4a). The methyl ester **29** did not convert under the standard conditions, stressing the importance of BI coordination to the free carboxylic acid and excluding the single-electron reduction of the N−N bond (Scheme 4b). The sulfonamidyl-iminophenylacetic acids **30**−**32** with different aryl substituents were also synthesized, which sulfonamidyl radical generation capacity was evaluated and compared with **1** (Scheme 4c). Sulfonamidyl-iminophenylacetic acid **30** with the electron-rich methyl group gave a similarly 75% yield of **3**, and the electron-withdrawing chloride and fluoride group in **31** and **32** afforded improved sulfonamidation adduct **3** in up to 89% yield. These results suggested the reaction yields may be further increased upon request. However, **1** is still recommended as the general sulfonamidyl radical precursor considering the readily availability. When **31** was used as the amidyl radical precursor, 73% yield of 4-chlorobenzonitrile was isolated together with the amidation adduct **3** in 71% yield (Scheme 4d).

**Scheme 4. Mechanistic Investigations on (Hetero)arene Amidation**




Consistent with the previous reports on cyclic iodine activation of carboxylates,[9,10] IPA-NTsMe **1** reacted with BI-OAc to form the BI-IPA-NTsMe complex **34** (Figure S1 in the Supporting Information), which oxidation potentials ($E_p$ = +1.15 V vs SCE in MeCN for **34**) were significantly decreased compared to **1** ($E_p$ = +1.85 V vs SCE in MeCN) (Figures S2−S4 in the Supporting Information). We further investigated the additional role of hypervalent iodines in the reaction. In the absence of BI-OAc, BI-IPA-NTsMe complex **34** afforded the product **3** in 66% yield, while the addition of BI-OAc improved **3** to yields of 75%−84% (Scheme 4e). From fluorescence quenching experiments (Figure S5 in the Supporting Information), BI-IPA-NTsMe **34** was more effective than BI-OAc for the excited photocatalyst quenching, which suggested both BI-OAc and **34** may oxidatively quench the excited Ru(II)* for Ru(III) formation. In addition, BI-OAc helped stabilize BI-IPA-NTsMe **34** as the coordination of BI-OAc to BI-IPA-NTsMe was in equilibrium. The quantum yield of the reaction was measured to be 0.53, indicating that the radical chain reaction was unlikely.

From the mechanistic investigations above, the photoredox catalysis is initiated with the oxidative quenching of the excited-state Ru(II)* by BI-OAc or BI-IPA-NTsMe **I** to afford Ru(III) (Scheme 5). The coordination of the iminophenyl-acetic acid with BI-OAc forms the BI-IPA-NTsMe complex **I**, which has significantly reduced oxidation potential for facile oxidation. The Ru(III) oxidation of the BI-IPA-NTsMe complex **I** obtained the iminophenylcarboxyl radical cation **II**, which yielded the sulfonamidyl radical **III** after the concerted carbon dioxide and phenylnitrile elimination. The sulfonamidyl radical then underwent the radical addition to



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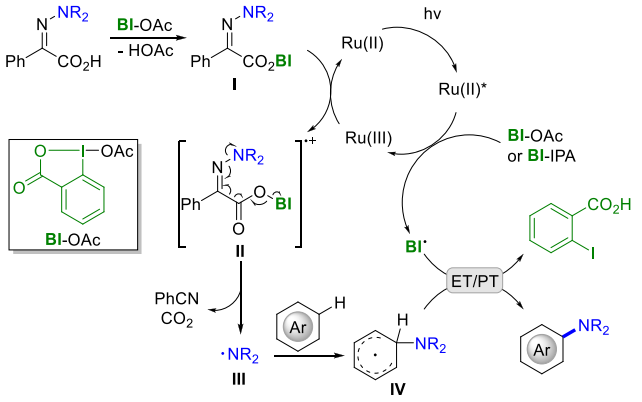
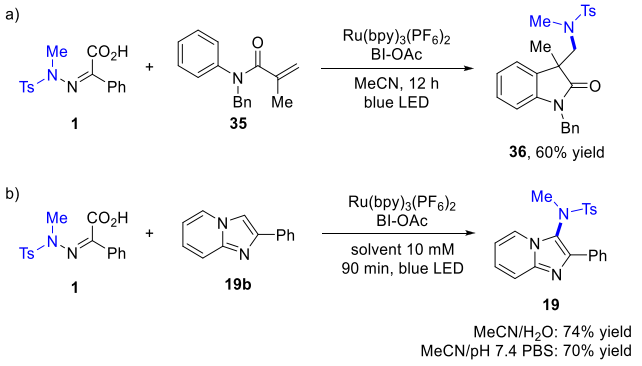
**Scheme 5. Mechanistic Proposal**




(hetero)arenes, and the addition intermediate **IV** was further oxidized by the BI radical to afford the (hetero)arene C($sp^2$)−H sulfonamidation adduct.[17]

We also performed the reaction with *N*-arylacrylamides **35** and observed the addition/cyclization cascade of sulfonamidyl radical to afford the sulfonamide-oxindoles **36** in 60% yield (Scheme 6a). With the mild photocatalytic reaction conditions

**Scheme 6. Amidyl Radical Cyclization and Aqueous Reactions**




from hypervalent iodine BI-OAc, we also tested the possibility of the amidation for potential biomolecule applications.[5] The imidazo[1,2-*a*]pyridine skeleton was the molecular framework targeting GPCRs similar to adenine however with better pharmacokinetic properties,[18] and we tested the aqueous synthesis of sulfonamidyl imidazopyridine **19**. Pleasingly, the sulfonamidation performed smoothly at 10 mM concentration of **1** in aqueous solutions or in pH 7.4 PBS buffer to afford **19** in yields of 70%−74%, which will be useful for the photorelease of bioactive molecules for biological photomanipulations.[19]

In conclusion, we have developed the bench-stable iminophenylacetic acid derivatives to enable the C($sp^2$)−H amidation of (hetero)arenes via sulfonamidyl radicals. The formation of the benziodoxole-iminophenylacetic acid (BI-IPA-$NR_2$) complex decreased the oxidation potentials of IPA-$NR_2$ for the facile single-electron oxidation. The selective C($sp^2$)−H functionalization of (hetero)arenes constructed various sulfonamide-substituted (hetero)arenes widely presenting in bioactive molecules and could perform in neutral aqueous conditions with potential biological applications.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.2c02751.

Mechanistic studies, substrate preparations and characterizations, crystal data, and NMR spectra of new compounds (PDF)

### Accession Codes

CCDC 2009399 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

## ■ AUTHOR INFORMATION


### Corresponding Author

**Yiyun Chen** − *State Key Laboratory of Bioorganic and Natural Products Chemistry, Center for Excellence in Molecular Synthesis, Shanghai Institute of Organic Chemistry, University of Chinese Academy of Sciences, Chinese Academy of Sciences, Shanghai 200032, China; School of Physical Science and Technology, ShanghaiTech University, Shanghai 201210, China; School of Chemistry and Material Sciences, Hangzhou Institute for Advanced Study, University of Chinese Academy of Sciences, Hangzhou 310024, China;* orcid.org/0000-0003-0916-0994; Email: yiyunchen@sioc.ac.cn

### Authors

**Yue Pan** − *State Key Laboratory of Bioorganic and Natural Products Chemistry, Center for Excellence in Molecular Synthesis, Shanghai Institute of Organic Chemistry, University of Chinese Academy of Sciences, Chinese Academy of Sciences, Shanghai 200032, China; Department of Chemistry, Shanghai University, Shanghai 200444, China*

**Zhengyi Liu** − *State Key Laboratory of Bioorganic and Natural Products Chemistry, Center for Excellence in Molecular Synthesis, Shanghai Institute of Organic Chemistry, University of Chinese Academy of Sciences, Chinese Academy of Sciences, Shanghai 200032, China*

**Peng Zou** − *State Key Laboratory of Bioorganic and Natural Products Chemistry, Center for Excellence in Molecular Synthesis, Shanghai Institute of Organic Chemistry, University of Chinese Academy of Sciences, Chinese Academy of Sciences, Shanghai 200032, China*

**Yali Chen** − *Department of Chemistry, Shanghai University, Shanghai 200444, China*

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.orglett.2c02751

### Author Contributions

$^{\nabla}$These authors contributed equally to this paper.


### Author Contributions

All authors have given approval to the final version of the manuscript.

### Notes

The authors declare no competing financial interest.



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