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Letter

Diastereoselective Construction of Cyclopropane-Fused Tetrahydroquinolines via a Sequential [4 + 2]/[2 + 1] Annulation Reaction

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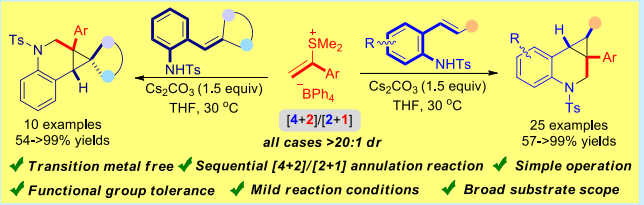
ABSTRACT: A sequential [4 + 2]/[2 + 1] annulation of α-aryl vinylsulfoniums with 2-aminochalcones and 2-(2-aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione is reported that affords a series of cyclopropane-fused tetrahydroquinolines. The salient features of this novel and practical transformation include high efficiency, transition-metal-free nature, operational simplicity, and outstanding functional group tolerance.



Tetrahydroquinolines, a kind of privileged structural scaffold in the family of nitrogen-containing heterocycles, are widely distributed in a number of pharmaceutically relevant bioactive molecules, such as galipinine, nicainoprol, and oxamniquine (Figure 1).[1] Access to functionalized tetrahydroquinolines has long attracted the interest of synthetic chemists and pharmacologists.[2] Moreover, as omnipresent and ubiquitous subunits, cyclopropanes not only show great potential for development in medicinal chemistry (Figure 1)[3] but also act as all-purpose and important intermediates in the construction of series of key framework structures.[4] It also deserves to be mentioned that researchers have found that cyclopropane-fused tetrahydroquinolines could serve as potent HIV-1 non-nucleoside reverse transcriptase inhibitors.[5] Driven by the structural complexity and remarkable biological properties of cyclopropane-fused tetrahydroquinolines, intense interest has been triggered within both the synthetic and medicinal communities, and several efficient approaches have been developed to date.[6] In 2012, Cramer's group reported palladium-catalyzed C−H arylation of cyclopropanes to achieve the construction of structurally diverse tetrahydroquinolines (Scheme 1a).[6a] Soon thereafter, Doyle's group realized a novel Lewis acid-catalyzed Povarov reaction that provides cyclopropane-fused tetrahydroquinolines (Scheme 1b).[6b] Although great progress has been achieved, the construction of cyclopropane-fused tetrahydroquinolines has been restricted to metal catalysis until now. Hence, it is necessary to develop efficient transformations without metal catalysis to synthesize cyclopropane-fused tetrahydroquinolines.

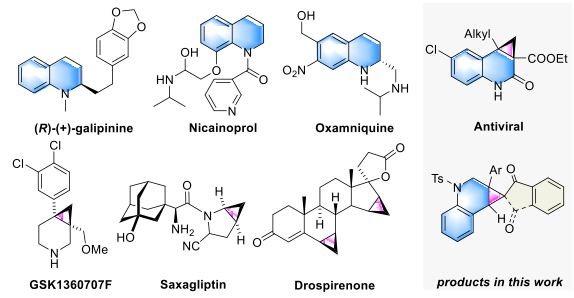
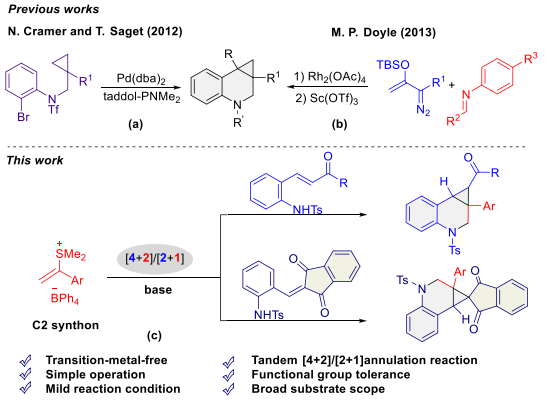


Figure 1. Natural products containing tetrahydroquinoline or cyclopropane and products in this work.

Scheme 1. Strategy for the Construction of Cyclopropane-Fused Tetrahydroquinolines



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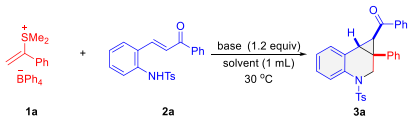
Cyclopropanation of alkenes as an efficient means could lead to synthetically useful and biologically important cyclopropane-fused heterocyclic ring systems.[7] α-Substituted vinylsulfonium tetraphenylborates as unique vinylsulfoniums have better stability and accessibility due to the existence of the tetraphenylborate counterion.[8] To the best of our knowledge, the use of α-aryl vinylsulfonium tetraphenylborates as materials for the preparation of cyclopropane-fused tetrahydroquinolines has not been exploited up to now. On the other hand, 2-aminochalcones and 2-(2-aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione are useful synthons and could be used to construct kinds of functionalized tetrahydroquinolines.[9] In this context and in conjunction with the long-term interest of our group directed toward the exploitation of novel reactions for the construction of heterocyclic compounds,[10] in this work we developed a novel transition-metal-free sequential [4 + 2]/[2 + 1] annulation reaction of α-substituted vinylsulfoniums with 2-aminochalcones and 2-(2-aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione that delivers valuable cyclopropane-fused tetrahydroquinolines (Scheme 1c).

We carried out the preliminary optimization experiments employing the α-phenylvinylsulfonium tetraphenylborate (**1a**) and 2-aminochalcone **2a** as the model substrates to verify the feasibility of the transformation. The reaction proceeded smoothly in the presence of tetramethylguanidine, yielding cyclopropane-fused tetrahydroquinoline **3a** in 46% yield with excellent diastereoselectivity. To improve the synthetic efficiency, some other bases were evaluated, including DBU, DABCO, Na_2CO_3, Cs_2CO_3, K_2CO_3, and *t*-BuOK. It was found that reaction activity is strongly influenced by the base, and the reaction with Cs_2CO_3 as the base gave the best results, yielding **3a** with >20:1 dr in 90% yield (Table 1, entry 6). In order to increase the efficiency of synthetic process, the medium was investigated, and the results showed that THF was the most suitable solvent, in which the expected sequential reaction was complete within 2 h and afforded product **3a** in excellent yield (96%; Table 1, entry 10). In addition, when 1.5 equiv of Cs_2CO_3 was used, an improvement in the yield to 97% was achieved (Table 1, entry 12).

Table 1. Optimization of the Reaction Conditions[a]

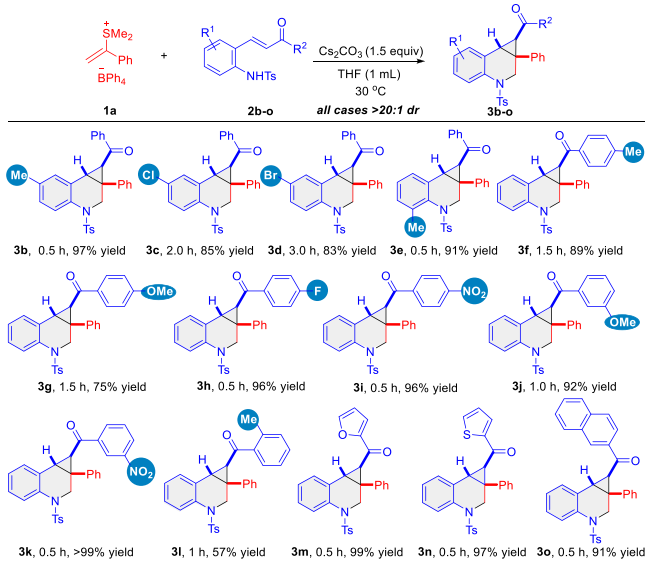


entry	base	solvent	time (h)	yield (%)[b]	dr[c]
1	TMG	CH_2Cl_2	2	46	>20:1
2	DBU	CH_2Cl_2	2	69	>20:1
3	DABCO	CH_2Cl_2	12	9	n.d.
4	Na_2CO_3	CH_2Cl_2	48	trace	n.d.
5	K_2CO_3	CH_2Cl_2	2	81	>20:1
6	Cs_2CO_3	CH_2Cl_2	2	90	>20:1
7	*t*-BuOK	CH_2Cl_2	2	69	>20:1
8	Cs_2CO_3	CH_3CN	3	95	>20:1
9	Cs_2CO_3	MeOH	96	n.r.	n.d.
10	Cs_2CO_3	THF	2	96	>20:1
11	Cs_2CO_3	Toluene	2	94	>20:1
12[d]	Cs_2CO_3	THF	0.5	97	>20:1

[a]Unless specified otherwise, reactions were performed with 0.12 mmol of **1a**, 0.10 mmol of **2a**, and 0.12 mmol of base in DCM (1.0 mL). [b]Isolated yields. [c]Determined by ^{1}H NMR analysis. [d]0.15 mmol of Cs_2CO_3 was used.

After we obtained the optimized reaction conditions, we inspected the scope and generality of the sequential [4 + 2]/[2 + 1] annulation reaction by reacting **1a** with 2-aminochalcones **2**. As shown in Scheme 2, the results showed that the electronic properties and positions of substituent groups on the aromatic skeletons made no negative impression on the diastereoselectivities, and all of the desired products were obtained with >20:1 dr. We first examed substrates **2** bearing variation of the substituent R^1 at C3 or C5 on the aniline moiety, and the reaction yielded products **3b–e** in good yields (83–97%). Then the substituent on the aryl ring (R^2) was explored, and irrespective of the substituent position and electronic properties, all of them could be used in the developed annulation, furnishing the cyclopropane-fused tetrahydroquinoline derivatives **3f–l** in moderate to excellent yields (57–99%). What is more, the sequential transformation also could tolerate heteroaromatic rings (R^2), generating **3m** and **3n** in 99% and 97% yield, respectively. It is worth mentioning that 2-naphthyl-substituted 2-tosylaminochalcone **2o** also could successfully be converted into the corresponding cyclopropane-fused tetrahydroquinoline **3o** in 91% yield.

Scheme 2. Substrate Scope of 2-Aminochalcones[a,b,c]



[a]Reactions were performed with 0.12 mmol of **1a**, 0.10 mmol of **2**, and 0.15 mmol of Cs_2CO_3 in THF (1.0 mL). [b]The dr values were determined by ^{1}H NMR analysis. [c]Isolated yields are shown.

Next, more in-depth exploration and study of the sequential transformation generality was our focus. First, diverse substitutions on the benzene ring of α-substituted vinylsulfonium tetraphenylborates **1** were explored. When vinylsulfonium tetraphenylborates **1b–h** bearing substituents at the *para* or *meta* position possessing electron-rich or electron-deficient character were employed, the reactions took place smoothly to afford the desired annulation products **3p–v** in 62–99% yield with >20:1 dr (Scheme 3). However, vinylsulfonium tetraphenylborates **1i** and **1j** bearing a chlorine or bromine atom, respectively, at the *ortho* position could not give the desired products. Additionally, α-(2-naphthyl)-vinylsulfonium tetraphenylborate (**1k**) and α-biphenylvinylsulfonium tetraphenylborate (**1l**) were tolerated by the desired

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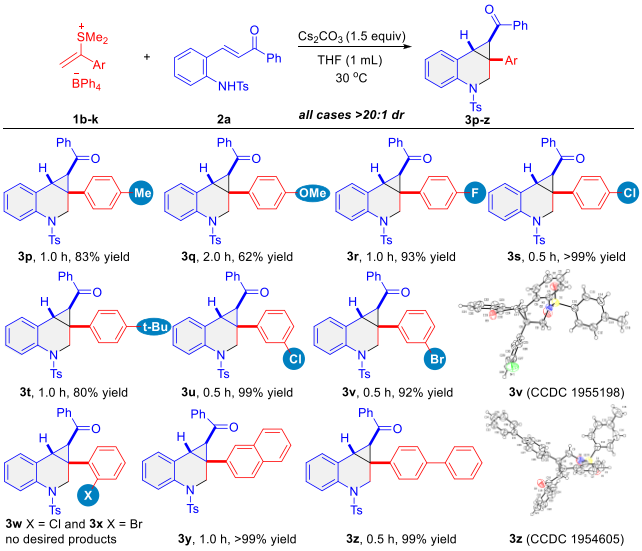
Scheme 3. Substrate Scope of α-Substituted Vinylsulfoniums[a,b,c]

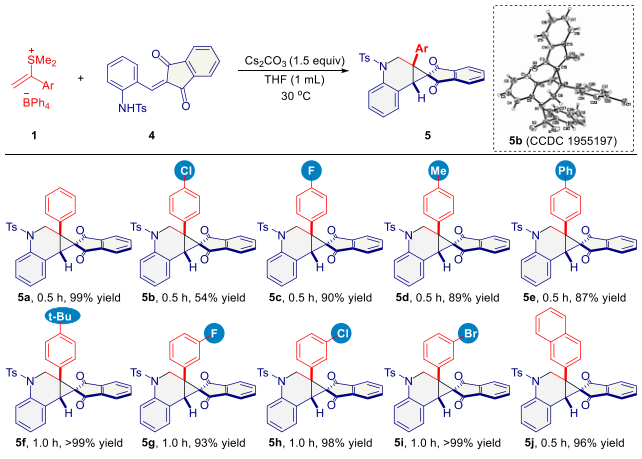


[a]The reaction conditions were the same as in Scheme 2. [b]The dr values were determined by ^{1}H NMR analysis. [c]Isolated yields are shown.

transformation and led to interesting polycyclic adducts **3w** and **3z** in yields of >99% and 99%, respectively, with >20:1 dr. From X-ray diffraction analysis and NMR data, we unambiguously verified the structures of **3v** (CCDC 1955198) and **3z** (CCDC 1954605).

Spirocyclopropane motifs as core structures are widely present in many biological compounds and natural products.[11] The construction of spirocyclopropanes has provoked researchers' interest.[12] Encouraged by the success of the above synthesis of cyclopropane-fused tetrahydroquinolines, we tried to further investigate the formation of spirocyclopropane-fused tetrahydroquinoline structural motifs with the reaction of **1a** and 2-(2-aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione (**4**) under the standard conditions (Scheme 4). The transformation occurred successfully and afforded the

Scheme 4. Experimental Results with 2-(2-Aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione[a,b]

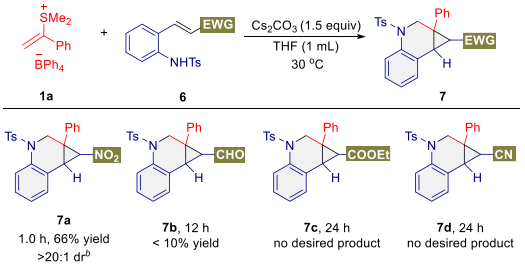


[a]The reaction conditions were the same as in Scheme 2. [b]Isolated yields are shown.

corresponding annulation product **5a** in excellent yield (99%). Then the reactions of various α-aryl-substituted vinylsulfonium tetraphenylborates **1** with **4** were conducted to test the generality of the sequential reaction. It was found that the transformations were well-tolerated and furnished the desired annulation products **5b–i** in 54% to >99% yield. Notably, the reaction also worked with the bulky 2-naphthylvinylsulfonium tetraphenylborate **4j**, delivering product **5j** in 96% yield. We unambiguously determined the relative configuration of **5b** by single-crystal X-ray analysis (CCDC 1955197).

In order to further investigate the generality and scope of this approach, some other anilines bearing different Michael acceptors were employed as reaction partners (Scheme 5). The

Scheme 5. Experimental Results with Other Substrates[a,b,c]

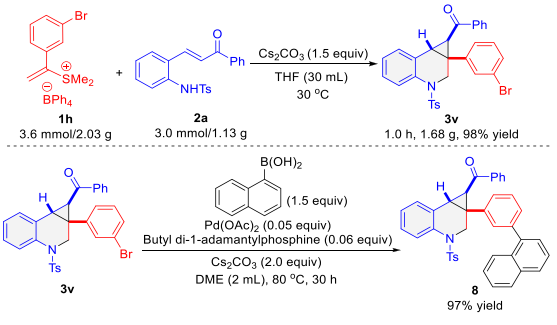


[a]Reaction conditions: **1a** (0.24 mmol), **6** (0.20 mmol), and Cs_2CO_3 (0.30 mmol) in 2.0 mL of THF. [b]Determined by ^{1}H NMR analysis. [c]Isolated yields are shown.

sequential [4 + 2]/[2 + 1] reaction was applicable to nitro-bearing substrate **6a**, giving the corresponding adduct **7a** in 66% yield with >20:1 dr. With 2-aminocinnamaldehyde **6b** as a reaction partner, only a small amount of product was obtained. Michael acceptors containing an ester group (**6c**) or cyano group (**6d**) showed low reactivity, and we did not observe the corresponding desired products in the current study.

In the presence of optimal reaction conditions, we performed a gram-scale reaction of α-(3-bromophenyl)-vinylsulfonium tetraphenylborate (**1h**) with 2-aminochalcone **2a** (3.0 mmol, 1.13 g), and cyclopropane-fused tetrahydroquinoline **3v** was obtained in 98% yield (Scheme 6). Moreover,

Scheme 6. Gram-Scale Reaction and Derivatization of 3v



derivatization of **3v** with 1-naphthaleneboronic acid was conducted by a palladium-catalyzed C–C bond coupling reaction, affording the corresponding product **8** in 97% yield.

On the basis of our experimental results, HRMS analysis (for details, see the Supporting Information) and the related literature,[7,8] a possible reaction pathway for sequential [4 + 2]/[2 + 1] reaction is proposed in Figure 2. In the presence of

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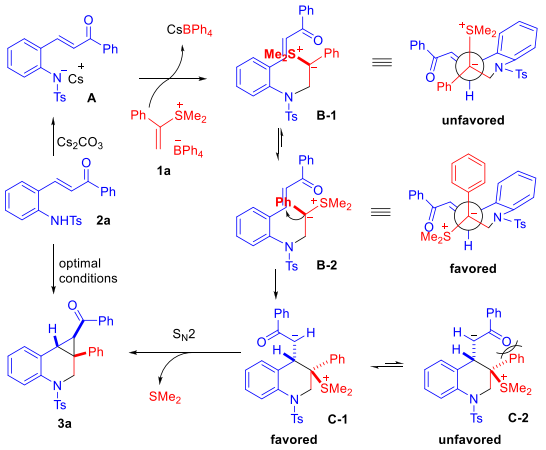

Figure 2. Proposed plausible pathway.

Cs_2CO_3, 2-aminochalcone **2a** is deprotonated to generate nucleophilic anion **A**, which attacks α-phenyl-substituted vinylsulfonium tetraphenylborate **1a** to give intermediate **B**. Probably because of the $\pi-\pi$-stacking interaction between the phenyl groups of the vinylsulfonium and the aniline in the 2-aminochalcone, the subsequent intramolecular Michael addition preferentially takes place via intermediate **B-2**, resulting in the formation of intermediate **C**. Subsequently, perhaps as a result of the steric hindrance effect from the benzoyl and phenyl groups, the intramolecular S_N2 reaction preferentially occurs via intermediate **C-1** to yield the expected cyclopropane-fused tetrahydroquinoline product **3a** with simultaneous release of dimethyl sulfide.

In conclusion, our group has developed a practical and efficient Cs_2CO_3-promoted sequential [4 + 2]/[2 + 1] annulation reaction of α-substituted vinylsulfoniums with 2-aminochalcones and 2-(2-aminobenzylidene)-1*H*-indene-1,3(2*H*)-dione. Under mild conditions, structurally diverse cyclopropane-fused tetrahydroquinolines and spirocyclopropane-fused tetrahydroquinolines could be efficiently furnished in excellent yields with this protocol. What is more, the underlying synthetic application of the reported transformation was proved through scale-up verification and chemical conversion. In addition, a plausible pathway was proposed to rationalize the stereoselective outcome of the sequential annulation process. We expect these cyclopropane-fused tetrahydroquinoline products to have good prospects for chemical biology and drug discovery.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c00874.

Experimental details, full analysis data (melting point, NMR, and HRMS data) for new compounds, and copies of NMR spectra (PDF)

Primary NMR FID files for compounds **3a–z**, **5a–j**, **7a**, and **8** (ZIP)

Accession Codes

CCDC 1954605, 1955197, and 1955198 contain 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, U.K.; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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