OL Organic Letters
Letter
Cite This: *Org. Lett.* 2019, 21, 8459−8463
pubs.acs.org/OrgLett

Rhodium(III)-Catalyzed Chemo-divergent Couplings of Sulfoxonium Ylides with Oxa/azabicyclic Olefins

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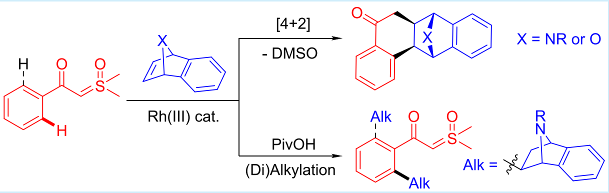
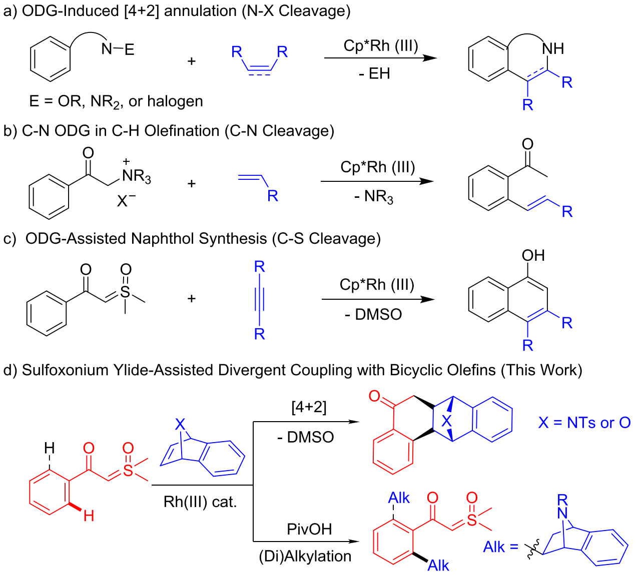
Supporting Information

ABSTRACT: Rh(III)-catalyzed C−H functionalization of sulfoxonium ylides has been realized in chemo-divergent couplings with oxa/azabicyclic olefins. Divergent [4 + 2] annulation and C−H alkylation have been attained under controlled condition. In the annulation system, sulfoxonium ylide functions as a traceless oxidizing directing group. In contrast, the introduction of PivOH shunted the selectivity to ring-retentive alkylation.



Metal-catalyzed functionalization of C−H bonds has been established as an increasingly important strategy in construction of complex structures.[1] Among the various transition metals, rhodium(III) catalysts stand out with high selectivity, activity, and functional group compatibility for activation of a plethora of arenes bearing a directing group (DG).[2] In particular, oxidizing directing groups (ODGs)-assisted[3−9] C−H activation−annulation has attracted considerable attention for the synthesis of heterocycles and carbocycles. Inspired by the pioneering work by Wu[4] and Fagnou,[5] important progress has been made in ODG-assisted atom- and step-economic annulations of arenes with diazo compounds,[7] alkynes[8] and alkenes.[9] Despite the significance, the ODG is mostly attached to a heteroatom in the arene. With the cleavage of N−O, N−N, and O−O bonds, a heteroatom will reside in the final product (Scheme 1a). From a perspective of atom-, step-, and redox-economy, further development of carbon-tethered ODGs to access complementary carbocyclic scaffolds remains in great demand. Thus, we recently designed phenacyl ammonium salts as arenes for such a purpose and realized annulative coupling with α-diazo esters.[10] When olefin was employed as a coupling partner, a linear or branched Heck-type coupling product was obtained (Scheme 1b).

Scheme 1. Rh(III)-Catalyzed ODG-Assisted [4 + 2] Annulation


a) ODG-Induced [4+2] annulation (N-X Cleavage)

b) C-N ODG in C-H Olefination (C-N Cleavage)

c) ODG-Assisted Naphthol Synthesis (C-S Cleavage)

d) Sulfoxonium Ylide-Assisted Divergent Coupling with Bicyclic Olefins (This Work)

As versatile carbene precursors,[11−13] sulfoxonium ylides have been demonstrated to have found wide utility in C−H activation, because of their high flexibility, stability, and diverse reactivity. In contrast, employment of sulfoxonium ylides as an arene substrate bearing a built-in ODG remains underdeveloped. In 2017, we reported redox-neutral [4 + 2] annulation of sulfoxonium ylides with alkynes for efficient construction of substituted 1-naphthols,[12a] in which the sulfoxonium ylide functioned as a carbene-like DG (Scheme 1c). Inspired by the high reactivity of sulfoxonium ylides in C−H activation,[12,13] we reasoned that a [4 + 2] annulation reaction may occur between a sulfoxonium ylide and a suitable olefin via a C−H activation pathway, affording a naphthalen-1(2*H*)-one framework, which is the core structure of nature product such as 10-norparvulenone[14] and *O*-methylasparvenone.[15] We now report Rh(III)-catalyzed chemodivergent coupling of sulfoxonium ylides with 7-oxa/azabenzonorbornadienes.

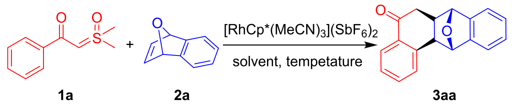
Our initial attempts failed when styrene or acrylate ester was selected as a coupling reagent. Strained olefins such as 7-oxa/azabenzonorbornadienes are inherently reactive and have been applied in C−H activation chemistry.[16] In most cases, ring-

Received: September 12, 2019
Published: October 7, 2019

DOI: 10.1021/acs.orglett.9b03226

scission coupling with arenes was realized.[16b,g,k,m,r] Therefore, we examined the reaction of sulfoxonium ylide **1a** and 7-oxabenzonorbornadiene (**2a**) in the presence of a cationic $[RhCp^*(MeCN)_3](SbF_6)_2$ catalyst (see Table 1). The desired [4 + 2] reaction occurred to afford product **3aa** in 45% yield as a single diastereomer (Table 1, entry 1). The yield was improved to 55% when the reaction temperature was reduced to 80 °C (Table 1, entries 2–4). Halogenated solvents (TCE, PhCl, $CHCl_3$, and DCM) seem suitable for this reaction, and the best results were obtained in DCM (Table 1, entries 6–8). The yield of **3aa** was improved to 79% when 2.0 equiv of **2a** was used (Table 1, entry 9). To our surprise, **3aa** was obtained in 88% yield when the temperature was further lowered to 70 °C (Table 1, entry 10).

Table 1. Optimization of the Reaction Conditions[a]



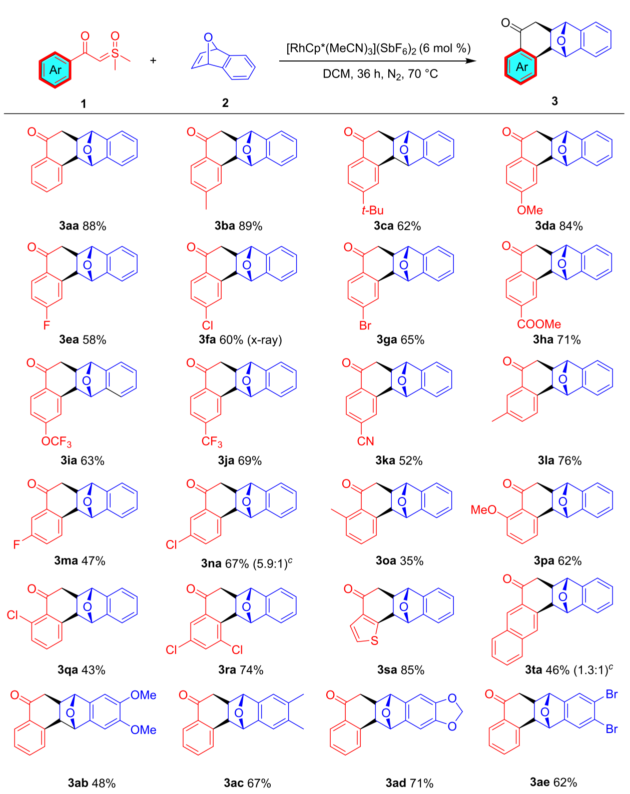
entry	solvent	T (°C)	yield[b] (%)
1	DCE	120	45
2	DCE	100	48
3	DCE	80	55
4	DCE	60	41
5	TCE	80	50
6	PhCl	80	55
7	$CHCl_3$	80	58
8	DCM	80	62
9[c]	DCM	80	79
10[c]	DCM	70	88

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (1.5 equiv), $[RhCp^*(MeCN)_3](SbF_6)_2$ (6 mol %), solvent (1 mL) under N_2 for 36 h. [b] Isolated yield after column chromatography. [c] **2a** (2.0 equiv) was used.

With the optimized conditions in hand, we next investigated the generality of this [4 + 2] annulation (see Scheme 2). Benzoyl-substituted sulfoxonium ylides bearing electron-donating and electron-withdrawing groups at the *ortho*, *meta*, and *para* positions of the benzene ring all reacted smoothly with 7-oxabenzonorbornadiene (**2a**) in good to excellent yields (**3aa**–**3pa**). The structure of **3fa** was confirmed by X-ray crystallographic analysis (CCDC 1940088). Sulfoxonium ylide bearing a multisubstituted phenyl group (**1r**) was also applicable in this reaction. The sulfoxonium ylide was next smoothly extended to thiophene and naphthalene rings, with the corresponding **3sa** and **3ta** being isolated in yields of 85% and 46%, respectively. Furthermore, several substituted 7-oxabenzonorbornadienes (**2b**–**2e**) also underwent smooth annulation with **1a**, with the annulated products isolated in yields of 48%–71%.

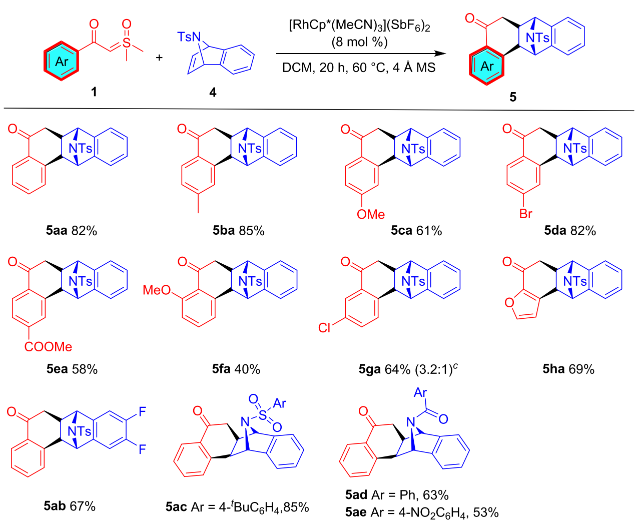
To better define scope of the bicyclic olefin, we next sought to evaluate the suitability of 7-azabenzonorbornadienes (see Scheme 3). A range of sulfoxonium ylides bearing electron-withdrawing and electron-donating substituents, such as alkyl, alkoxyl, halide, and ester at the *para* position turned out to be applicable under modified reaction conditions, producing the desired annulated products **5aa**–**5ea** in yields of 58%–85%. The C–H functionalization occurred preferentially at the less-hindered *ortho* site for a *meta*-chloro substituted arene (**5ga**). Introduction of an *o*-OMe group was also tolerated, as in the isolation of product **5fa** in moderate yield. Furthermore, a furan ring was also compatible (**5ha**). A ring-substituted 7-azabenzonorbornadiene also reacted smoothly (**5ab**). The *N*-

Scheme 2. Annulation of Sulfoxonium Ylide with 7-Oxabenzonorbornadiene[ab]



[a] Reaction conditions A: **1** (0.2 mmol), **2** (2.0 equiv), $[RhCp^*(MeCN)_3](SbF_6)_2$ (6 mol %), DCM (2 mL), 70 °C, under N_2 for 36 h. [b] Isolated yield after column chromatography. [c] Ratio of regioisomers.

Scheme 3. Annulation of Sulfoxonium Ylides with 7-Azabenzonorbornadienes[a,b]

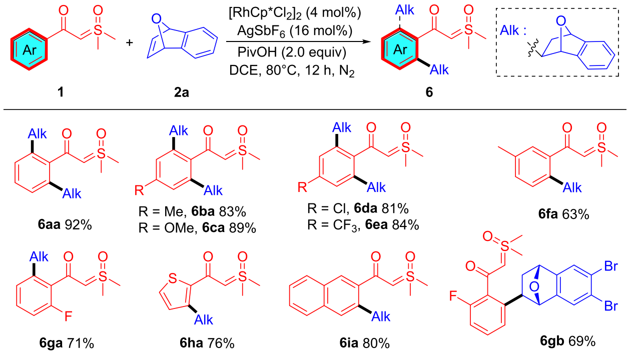


[a] Reaction Conditions A′: **1** (1.5 equiv), **4** (0.2 mmol), $[RhCp^*(MeCN)_3](SbF_6)_2$ (8 mol %), 4 Å MS (100 mg), DCM (2 mL), 60 °C under N_2 for 20 h. [b] Isolated yield after column chromatography. [c] Ratio of regioisomers.

protecting group in the 7-azabenzonorbornadiene was not restricted to a sulfonyl group, and several *N*-benzoyl protecting groups were also amenable (see **5ad** and **5ae**).

We postulated that the reaction proceeded via C−H activation and migratory insertion of Rh-Aryl into the olefin. Under suitable conditions, the resulting Rh-alkyl bond may be protonolyzed before it is intramolecularly trapped. Indeed, the introduction of PivOH under slightly modified conditions completely inhibited the [4 + 2] annulation between **1a** and **2a**. Instead, a ylide- and ring-retentive dialkylation product **6aa** was isolated in 92% yield (see Scheme 4), in which partially hindered rotation along the C(aryl)−C(O) bond was observed due to steric effects. Sulfoxonium ylides bearing both electron-withdrawing and electron-donating groups (alkyl, alkoxyl, halide, and CF$_3$) at the *para* position all reacted smoothly with **2a** in good to high yields (**6aa**−**6ea**). In addition, the introduction of an *ortho* or *meta* blocking group afforded the monoselective alkylation products (**6fa** and **6ga**). Furthermore, 2-thienyl- and 2-naphthyl-derived sulfoxonium ylides were viable, with the alkylation products **6ha** and **6ia** being isolated in good yield (76%−80%). A 7-oxabenzonorbornadiene bearing an electron-withdrawing group also reacted smoothly (**6gb**).

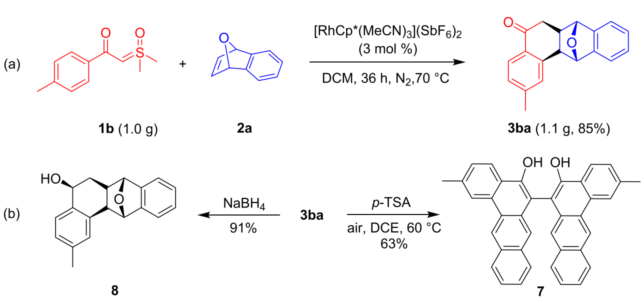
Scheme 4. Alkylation of Sulfoxonium Ylide with 7-Oxabenzonorbornadiene[a,b]



[a]Reaction conditions B: **1** (0.2 mmol), **2a** (2.5 equiv), [RhCp*Cl$_2$]$_2$ (4 mol %), AgSbF$_6$ (16 mol %), PivOH (2.0 equiv), DCE (2 mL), 80 °C, under N$_2$ for 12 h. [b]Isolated yield after column chromatography.

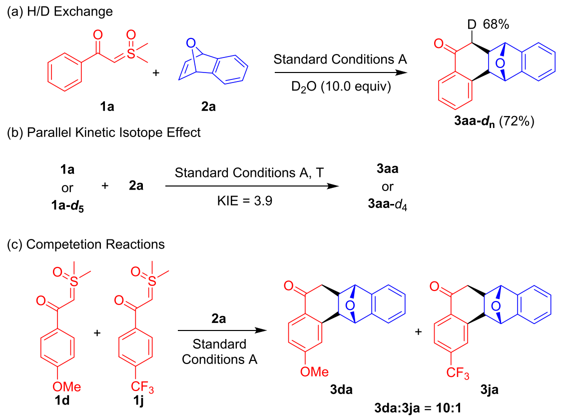
To demonstrate the synthetic applications of this reaction, a gram-scale coupling of ylide **1b** and **2a** was conducted under a reduced catalyst leading, from which product **3ba** was isolated in 85% yield (see Scheme 5a). Interestingly, treatment of **3ba** with *p*-TSA under aerobic conditions afforded the oxidative dimerization product **7** in 63% yield via initial dehydrative aromatization (Scheme 5b). Product **7** is a potentially useful polycyclic aromatic hydrocarbon and a ligand. NaBH$_4$ reduction of the carbonyl group in **3ba** afforded alcohol **8** in 91% yield as a single diastereomer. We have attempted the asymmetric [4 + 2] annulation of sulfoxonium ylide **1a** with azabicyclic olefin **4**, using a typical Cramer-type Rh(III)Cp^x chiral catalyst. Unfortunately, only racemic **5aa** was obtained.

Scheme 5. Scale-Up Synthesis and Derivatization Reactions



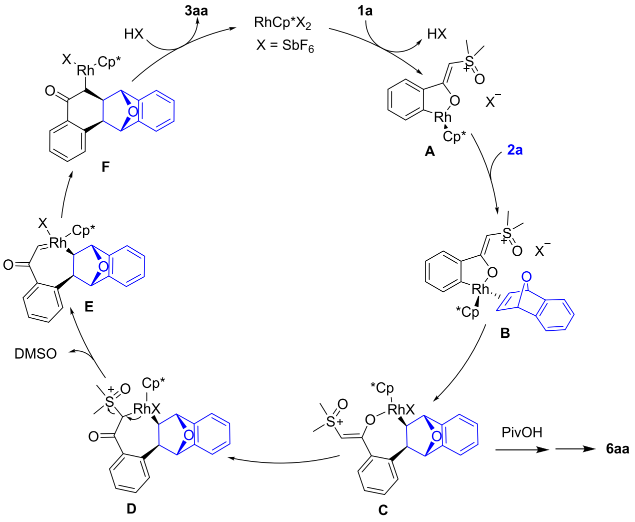
The mechanism of the [4 + 2] annulation has been briefly explored in a series of experimental studies (see Scheme 6). H/D exchange experiment under the standard conditions with additional D$_2$O revealed no deuteration at the *ortho* position of product **3aa**-d_n, suggesting irreversibility of the C−H activation (Scheme 6a). To better understand the C−H activation process, the kinetic isotope effect of this reaction was measured on the basis of parallel experiments, and a k_H/k_D value of 3.9 was obtained, which suggests that the C−H activation might be involved in the turnover-limiting process (Scheme 6b). Competition reactions showed that the C−H functionalization is kinetically favored for a more electron-rich arene substrate (Scheme 6c), which seems consistent with a concerted metalation−deprotonation mechanism.

Scheme 6. Mechanistic Studies



On the basis of the mechanistic studies and previous reports,[12a,16] a plausible catalytic cycle is proposed (see Scheme 7). Oxygen coordination of **1a**, followed by cyclometalation, delivers a five-membered rhodacyclic intermediate

Scheme 7. Proposed Reaction Mechanism



A. Subsequent coordination of bicyclic olefin **2a** gives the intermediate **B**, which undergoes migratory insertion of the aryl group into alkene to afford a seven-membered Rh(III) alkyl **C**. Then, O-bound to C-bound isomerization affords **D**, which likely undergoes α-elimination of DMSO to produce an α-oxo carbenoid intermediate **E**. Migratory insertion and protonolysis then gives the product with regeneration of the active catalyst. The presence of PivOH may induce protonolysis of Rh−C(alkyl) bond of intermediate **C**, and the second C−H alkylation eventually furnishes the dialkylation product **6aa**.[16d,e]

In summary, we have realized Rh(III)-catalyzed chemodivergent couplings of sulfoxonium ylides with 7-oxa/azabenzonorbornadienes. Both [4 + 2] annulation and C−H (di)alkylation have been attained under controlled conditions. In the annulation system, sulfoxonium ylide functions as an oxidizing carbene-type directing group. In contrast, the introduction of PivOH shifted the selectivity to ring-retentive alkylation. Both coupling systems proceeded under redox-neutral conditions with decent substrate scope. Given the rapid assembly of otherwise hard-to-access skeletons, operational simplicity, and controllable selectivity, this method may find applications in the synthesis of related fused carbocyclic scaffolds.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b03226.

Experimental procedures and spectral data of new compounds (PDF)

Accession Codes

CCDC 1940088 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.

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Financial support from the NSFC (No. 21525208) and SNNU is gratefully acknowledged.

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DOI: 10.1021/acs.orglett.9b03226
Org. Lett. 2019, 21, 8459−8463