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Alkynylation of Tertiary Cycloalkanols via Radical C−C Bond Cleavage: A Route to Distal Alkynylated Ketones

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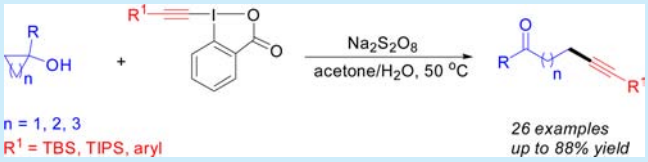
Supporting Information

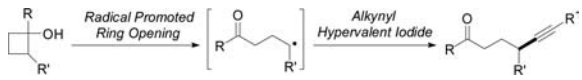
ABSTRACT: An efficient $Na_2S_2O_8$-promoted radical coupling of tertiary cycloalkanols with alkynyl hypervalent iodide reagents via C−C bond cleavage was developed. This tandem ring-opening/alkynylation procedure showed some advantages, including mild conditions and wide substrate scope, thus providing a simple synthetic method for β-, γ- and δ-alkynylated ketones.



The catalytic ring-opening coupling of strained tertiary cycloalkanols has emerged as an efficient strategy for the synthesis of functionalized acyclic organic molecules.[1] In this context, the palladium-catalyzed cross-coupling of tertiary cycloalkanols with various electrophiles has attracted much attention and has proven to be a powerful tool to obtain carbonyl compounds.[2] Recently, significant effort has also been made in the catalytic enantioselective C−C bond activation of cycloalkanols.[2d,3] In addition, the tandem radical ring-opening coupling of cycloalkanols under different one-electron oxidants has been developed and has provided another alternative method for accessing functionalized ketones.[4] For example, Narasaka's group reported Mn(III)-promoted and Ag(I)-catalyzed tandem radical coupling of cyclopropanols with alkenes, respectively. In those transformations, the β-carbonyl radical intermediates generated from the oxidative ring-opening of cyclopropanols are involved.[4b−d] Subsequently, several research groups applied this tandem radical ring-opening/coupling strategy for the synthesis of heterocycles[5a,b] and organofluorine compounds.[5c−e] More recently, Dai and Lopp have independently described the Cu-catalyzed ring-opening electrophilic trifluoromethylation and amination of cyclopropanols.[6] However, only a few studies on the ring-opening coupling of less strained cyclopentanols have been reported.[5e] Although catalytic arylation[2b,d,i] and alkylation[4b−d,6d] of tertiary cycloalkanols have been well documented, reports on the alkynylation of tertiary cycloalkanols are still rare.[2h,6f]

The use of alkynyl hypervalent iodide (AHI) as an efficient alkynylating reagent has recently received increasing attention in organic synthesis due to its excellent reactivity and versatility.[7] A series of elegant Au-,[8a−e] Pd-,[8f−h] Pt-,[8i] Cu-,[8j] and Rh-catalyzed[8k−n] alkynylation reactions involving AHI have been developed for the synthesis of functionalized alkynes. In 2012, Li and co-workers reported an efficient Ag(I)-catalyzed radical decarboxylative alkynylation of aliphatic carboxylic acids with AHI.[9] In addition, some transition-metal-free alkynylation reactions have also been reported.[10] In this respect, we reported a mild $K_2S_2O_8$-promoted radical decarboxylative alkynylation of α-keto acids with AHI.[11a] Although significant progress on alkynylation reactions using AHI as the alkynylation reagent has been made, the catalytic radical alkynylation of strained tertiary cycloalkanols with AHI is virtually unknown. As a part of our ongoing interest in the chemistry of AHI, we surmise that the γ-keto radical generated from the oxidative C−C bond cleavage of strained cyclobutanol might be trapped by the electrophilic alkynyl hypervalent iodide reagent to afford the γ-alkynylated ketone (Scheme 1). Herein, we report a tandem radical ring-opening/alkynylation of strained tertiary cycloalkanols with hypervalent iodide reagents under mild transition-metal-free conditions which provides an efficient route to distal alkynylated ketones in good yields.

Scheme 1. Alkynylation of Tertiary Cyclobutanols



Initially, we selected 1-phenylcyclobutanol (**1a**) and 1-[(*tert*-butyldimethylsilyl)ethynyl]-1,2-benziodoxol-3(1*H*)-one (TBS-EBX, **2a**) as model substrates to optimize the ring-opening/alkynylation reaction.[4,5] Treatment of **1a** with **2a** in the presence of 10 mol % of $AgNO_3$ and 2.0 equiv of $K_2S_2O_8$ in CH_3CN/H_2O (1:1) at 50 °C afforded the desired product **3a** in 49% yield (Table 1, entry 1). We were delighted to find that the reaction of **1a** with **2a** also resulted in a similar yield even in the absence of catalyst (entry 1). The yield of **3a** was increased to 65% when acetone/H_2O (1:1) was used as the solvent (entry 2). Other homogeneous phase systems such as AcOH/H_2O and DMF/H_2O were also effective for this reaction (entries 3 and 4). In contrast, H_2O and CH_2Cl_2/H_2O just gave a trace amount of the desired product **3a** (entries 5 and 6). Gratifyingly, a much better yield of **3a** was obtained by increasing the amount of **2a** to 1.8

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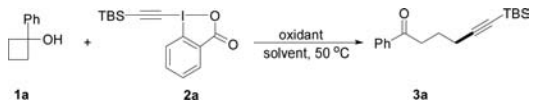
Table 1. Optimization of the Ring-Opening/Alkynylation of Cyclobutanol[a]



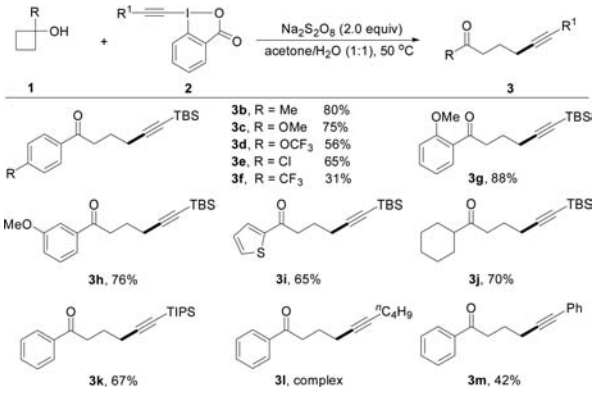
entry	oxidant (equiv)	solvent	yield (%)[b]
1	$K_2S_2O_8$ (2.0)	CH_3CN/H_2O (1:1)	47 (49)[c]
2	$K_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	65
3	$K_2S_2O_8$ (2.0)	AcOH/H_2O (1:1)	45
4	$K_2S_2O_8$ (2.0)	DMF/H_2O (1:1)	32
5	$K_2S_2O_8$ (2.0)	H_2O	trace
6	$K_2S_2O_8$ (2.0)	CH_2Cl_2/H_2O (1:1)	trace
7	$K_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	76[d]
8	$K_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	82[e]
9	$K_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	75[f]
10	$Na_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	87[e](83)[g]
11	$(NH_4)_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	59[e]
12	oxone (2.0)	acetone/H_2O (1:1)	trace[e]
13	$Na_2S_2O_8$ (2.0)	acetone/H_2O (1:1)	64[e,h]
14		acetone/H_2O (1:1)	nr[i]

[a]Reaction conditions: **1a** (0.20 mmol, 1.0 equiv), **2a** (0.24 mmol, 1.2 equiv), oxidant (0.40 mmol, 2.0 equiv), solvent (2 mL), 50 °C, 24 h under N_2. [b]Yield of isolated product. [c]10 mol % of $AgNO_3$ was used as a catalyst. [d]**2a** (0.30 mmol, 1.5 equiv). [e]**2a** (0.36 mmol, 1.8 equiv). [f]**2a** (0.40 mmol, 2.0 equiv). [g]Yield on a 1 mmol scale is given in parentheses. [h]Room temperature. [i]nr = no reaction.

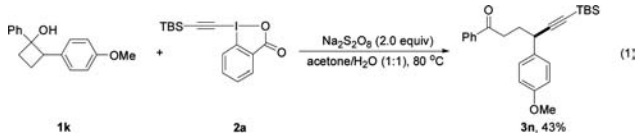
equiv (entries 7 and 8). However, a further increase of the amount of **2a** resulted in a decreased yield (entry 9). Further investigation revealed that $Na_2S_2O_8$ was the best oxidant for this transformation (entries 10−12). It should be noted that the reaction still gave 64% yield of **3a** at room temperature (entry 13). Finally, no reaction was observed in the absence of oxidant (entry 14).

With the optimized conditions in hand, the scope and limitations of this tandem ring-opening/alkynylation reaction were then investigated (Scheme 2). A variety of cyclobutanols

Scheme 2. Scope of Cyclobutanols and Alkynylating Agents

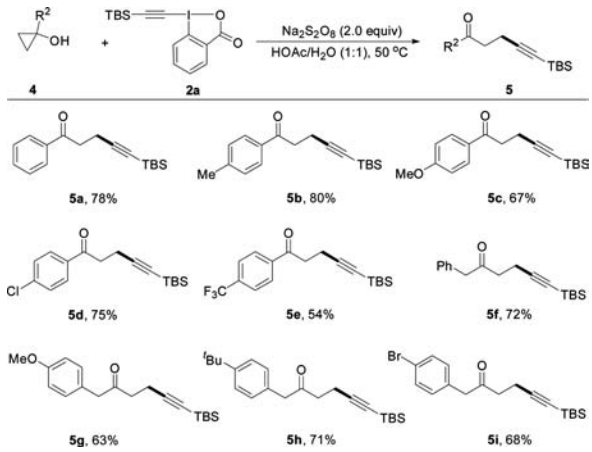


reacted with TBS-EBX efficiently to afford the corresponding γ-alkynylated ketones in moderate to good yields (**3b−j**). Aryl-substituted cyclobutanols containing an electron-withdrawing or -donating substituent at the *para* positon of the aromatic ring were good substrates to give the desired products **3b−e** in 56−80% yields. However, the strong electron-withdrawing effect of the CF_3 group led to the desired γ-alkynylated ketone **3f** in only 31% yield along with a large amount of **1f** recovered. Cyclobutanols bearing a methoxy group on the *ortho* or *meta* position of the phenyl ring were also smoothly converted into the corresponding products **3g** and **3h** in 88% and 76% yields, respectively. Moreover, the heteroaromatic-substituted cyclobutanol **1i** was also compatible with the reaction conditions and produced the desired γ-alkynylated ketone **3i** in 65% yield. More importantly, alkyl-substituted cyclobutanol **1j** also worked well with **2a** to afford the desired product **3j** in good yield. Other alkynylating reagents such as TIPS-EBX and aryl-EBX were also efficient to give the corresponding alkynylated ketones **3k** and **3m** in 67% and 42% yields, respectively. It should be noted that other alkynylating reagents, such as phenylacetylene, phenyl phenylethynyl sulfone, and phenylethynyl phenyliodonium tosylate failed to afford the desired product **3m** under the present conditions. Unfortunately, the alkyl-EBX furnished a complex mixture (**3l**). Finally, this ring-opening/alkynylation reaction could also be applied to 1,2-disubstituted cyclobutanols, leading to the desired product **3n** in 43% yield with high regioselectivity (eq 1).



Encouraged by the above results, we then investigated the reaction of tertiary cyclopropanols with TBS-EBX **2a**. To our delight, the ring-opening/alkynylation reaction proceeded smoothly to afford the expected β-alkynylated ketones **5** in moderate to good yields under the slightly modified conditions (Scheme 3). When 1-phenylcyclopropanol **4a** was subjected to

Scheme 3. Scope of Cyclopropanols



the reaction, the product **5a** was isolated in 78% yield. 1-Aryl-substituted cyclopropanols having either an electron-rich or -poor groups at the *para* position of the aromatic ring also reacted well with **2a** to give moderate yields of the corresponding products **5b−e**. The 1-benzylcyclopropanol **4f** was also successfully engaged in this reaction, delivering the product **5f** in 72% yield. Substituents such as MeO, ^{t}Bu, and Br on the benzene ring of cyclopropanols were also well-tolerated in this transformation (**5g−i**). Remarkably, we found that the less strained tertiary cyclopentanols also underwent the ring-opening/alkynylation process in the presence of silver catalyst to afford the desired δ-alkynylated ketones **7a−c** in moderate yields (eq 2). Unfortunately, the reaction of 1-phenylcyclohex-

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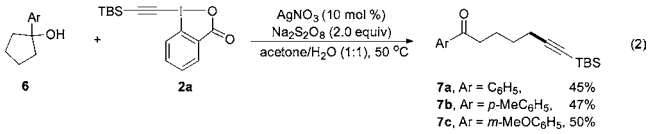
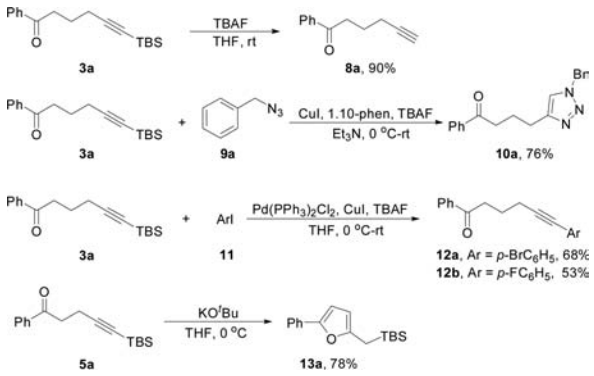
anol with **2a** afforded a complex mixture under the same conditions.



At last, some synthetic utilities of **3a** and **5a** are shown in Scheme 4 (for details, see the Supporting Information).

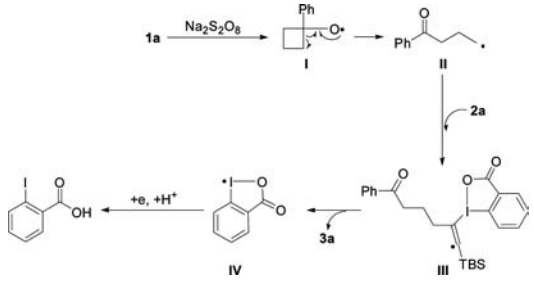
Scheme 4. Derivatization of the Product 3a and 5a



Treatment of **3a** with a stoichiometric amount of TBAF led to the terminal γ-alkynylated ketone **8a** in 90% yield. Satisfactorily, the triazole **10a** could be synthesized easily in 76% yield via the sequential desilylation and cycloaddition of **3a** with benzyl azide. Moreover, **3a** could also undergo tandem desilylation/Sonogashira coupling with 4-bromo- and 4-fluoroiodobenzene to afford the products **12** in moderate yields. This one-pot procedure offers a good opportunity to synthesize diverse alkynylated ketones. Finally, in the presence of 1.2 equiv of KO^{t}Bu, the β-alkynylated ketone **5a** could be easily converted to 2,5-disubstituted furan **13a** in good yield.

To gain some insight into the mechanism of this reaction, preliminary mechanistic experiments were conducted (for details, see the Supporting Information). Addition of 2,2,6,6-tetramethyl-1-piperidinoxyl (TEMPO) or 2,6-di-*tert*-butyl-4-methylphenol (BHT), the well-known radical scavenger, to the standard reaction of **1a** and **2a** markedly inhibited the formation of **3a**. These results indicate that a radical process might be involved in this reaction. On the basis of these results and previous reports,[4,5] a possible mechanism was proposed in Scheme 5. First, the single-electron oxidation of **1a** by $Na_2S_2O_8$ generates an oxygen-centered radical **I**,[4,5] which undergoes rearrangement to form γ-keto radical **II**. Second, radical **II** adds to the triple bond of hypervalent iodine reagent **2a** to afford the radical intermediate **III**. Finally, β-elimination of radical **III** gives the desired product **3a** as well as the 2-iodobenzoic acid, which is generated by a reduction−protonation of the benziodoxolonyl radical **IV**.

Scheme 5. Proposed Mechanism



In summary, we have developed a new tandem $Na_2S_2O_8$-mediated radical ring-opening/alkynylation of strained tertiary cyclobutanols with alkynyl hypervalent iodide reagents in which a sequence of C−C bond cleavage, radical rearrangement, and a C−C bond formation process were involved. Remarkably, this protocol could be further extended to other strained cycloalkanols, thus providing a straightforward approach to a series of β- and γ-alkynylated ketones. Moreover, less strained cyclopentanols have also been applied successfully in this reaction. This transformation provided a rapid and efficient strategy for the carbon chain growth, and the products could further undergo diverse transformations.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02353.

Experimental procedures and spectroscopic data of new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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