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Communication

Pd-catalyzed halocyclizations of unactivated 1,6-diynes through a formal *anti*-carbopalladation/bromide radical cascade

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ABSTRACT

We report a Pd-catalyzed halocyclization of unactivated 1,6-diynes with *N*-bromosuccinimide (NBS). This approach produces stereo-defined dibromo substituted dihydropyrans, tetrahydropyridines, and 3-methylene cyclohexenes with exocyclic double bond appendages in mostly good yields. Copper salt was found to be a useful Lewis acid in this reaction. Mechanistically, a formal *anti*-carbopalladation and a bromide radical promoted Pd^{II}-Pd^{III}-Pd^{I}-Pd^{II} catalytic cycles were proposed to be involved in the formation of the dibromo-substituted products. Further functionalization of the dihydropyran derivatives underwent $B(C_6F_5)_3$-catalyzed ring opening, and reduction afforded dibrominated 1,3-dienes with excellent stereoselectivity.

Cascade cycloisomerization reactions of unactivated 1,*n*-enynes or diynes are powerful synthetic strategies for fused polyheterocycles in efficient and green synthetic protocols [1]. However, these types of reactions typically necessitate high temperatures (>180 °C) [2] or visible light [3] to induce the initial radical formation. Transition-metal catalysts can be employed for multiple steps in a cascade cyclization protocol for products with significant molecular complexity. A wide range of important cyclic derivatives, such as polysubstituted benzenes [4], naphthyl ketones [5], fulvenes [6], benzofluorenes [7], and indenes [8] were chemo- and stereo- selectively produced from 1,*n*-enynes or diynes with various transition-metal catalyzed conditions.

The catalytic halocyclization reactions of 1,*n*-enynes or diynes are useful tools for the preparations of halo-substituted polyheterocycles. For example, in 2009, Song and Dong reported a Pd-catalyzed intramolecular [3 + 2] carboesterification reaction of propiolic acids and unactivated olefins to generate a tricyclic fused ring system (Scheme 1a) [9]. Our group recently developed the transition-metal-catalyzed electrophilic cycloisomerization reactions of benzo-fused 1,6-diynols or triynols with *N*-halosuccinimides (NXS, X = I, Br) [10]. Under the catalysis of a transition-metal catalysts, functionalized carbo(hetero)cyclic frameworks such as benzo[*a*]fluorenes, benzo[*b*]fluorenones, or naphthalenes were efficiently and chemoselectively produced under gentle reaction conditions. While the success of these examples referred to the halocyclizations of the *ortho*-fused aryl 1,*n*-enynes or diynes, instances for the halocyclization reactions of the unactivated acyclic 1,*n*-diynes are much more challenging. This is because the remote π-bonds have a very wide free rotational angle in these "unlocked" acyclic linear systems; therefore, the reactivity and the chemo- and/or regioselectivity are much more difficult to control.

To achieve a satisfactory endeavor in the non-conjugated acyclic unactivated diynes, Snyder and co-workers in 2015 reported that a stoichiometric amount of GaI_3-promoted cyclization of 1,6-diynes to obtain aza-heterocyclic vinyl iodides, that could undergo an *in situ* Friedel-Crafts cyclization to give the iodoindenopyridine derivatives (Scheme 1b) [11]. We herein disclose a Pd/Cu co-catalyzed halocyclization reaction of acyclic 1,6-diynes with NBS, which produces stereo-defined dibrominated (*E*)-3,6-dihydro-2*H*-pyrans in good yields (Scheme 1c). The resulting dibromo products could undergo ring-opening reactions with $B(C_6F_5)_3/HSiEt_3$ to stereo selectively give substituted 1,3-dienes, which are highly conjugated polyene structural motifs and useful building blocks in the synthesis of natural products such as β-carotene, vitamin **A**, and other naturally occurring retinoids (Scheme 1d) [12]. The dibromo-substituted tetrahydropyridines and 3-methylene cyclohexenes can also be stereoselectively prepared in good yields from this reaction.

We began to survey the reaction by utilizing 1,6-diynyl ether **1a** and 2.1 equiv. of *N*-bromosuccinimide (NBS) as the substrates (Table 1). After an extensive examination of the reaction

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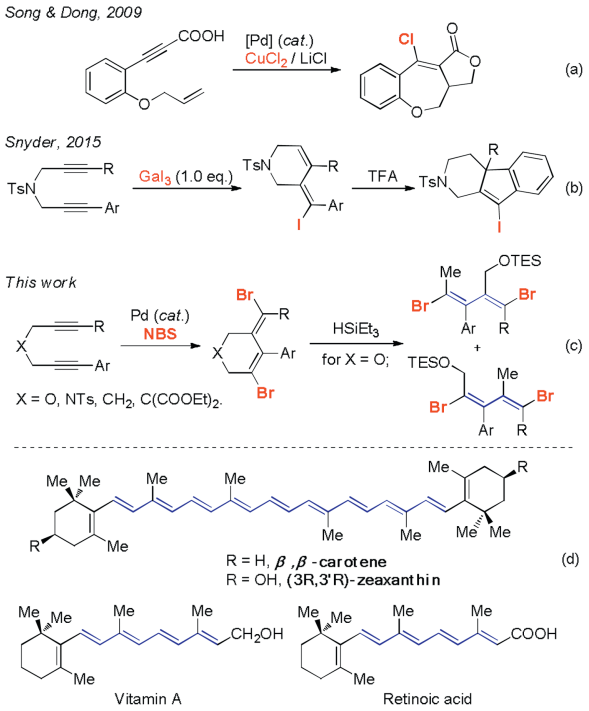
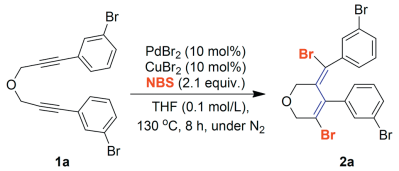


Scheme 1. Halocyclization of unactivated 1,6-diynes and the further derivation to 1,3-dienes.

Table 1
Optimization of the reaction conditions.[a]

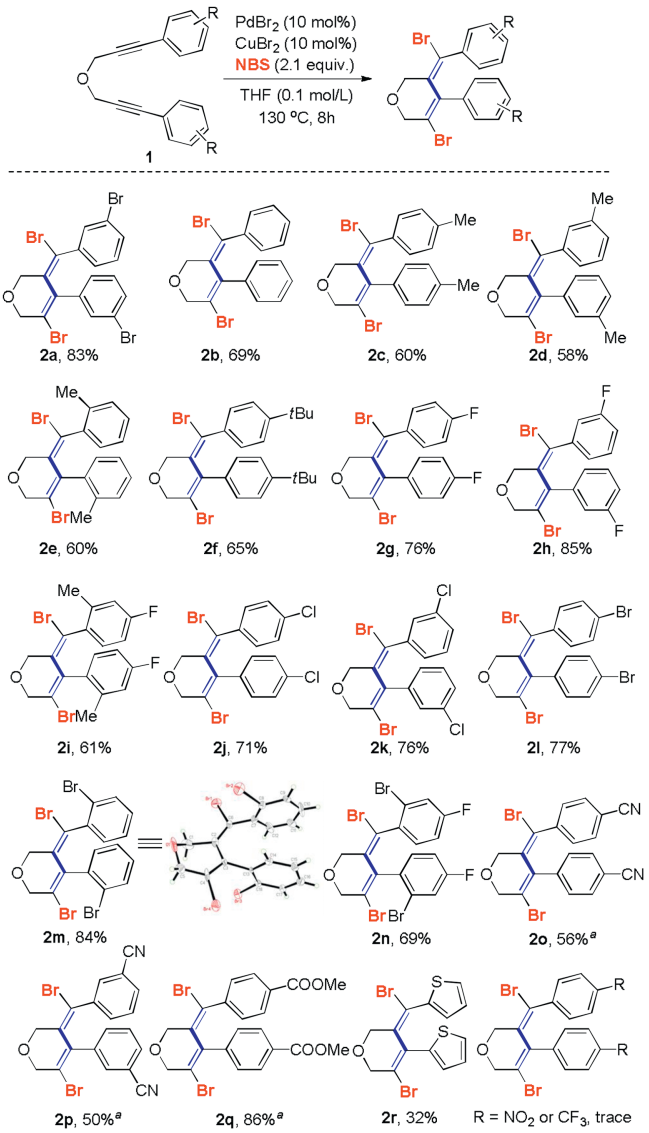


Entry	Variation of the reaction conditions	**2a** (%)
1	Standard conditions	83
2	No $PdBr_2$ and $CuBr_2$	0
3	No $PdBr_2$	10
4	$PdBr_2$ (10 mol%), no $CuBr_2$	57
5	$Pd(PPh_3)_2Cl_2$ (10 mol%), no $CuBr_2$	26
6	$Pd(OAc)_2$ (10 mol%), no $CuBr_2$	n.r.
7	$Pd(dba)_2$ (10 mol%), no $CuBr_2$	0
8	$FeBr_2$ (10 mol%) instead of $CuBr_2$	56
9	$FeBr_3$ (10 mol%) instead of $CuBr_2$	67
10	$CoBr_2$ (10 mol%) instead of $CuBr_2$	21
11	$NiBr_2$ (10 mol%) instead of $CuBr_2$	62
12	$ZnBr_2$ (10 mol%) instead of $CuBr_2$	57
13	CuBr (10 mol%) instead of $CuBr_2$	72
14	1,2-DCE as the solvent	77
15	Toluene as the solvent	68
16	1,4-dioxane as the solvent	41
17	DMSO as the solvent	0
18	$PdBr_2$ (5 mol%), $CuBr_2$ (10 mol%)	70
19	$PdBr_2$ (10 mol%), $CuBr_2$ (5 mol%)	77
20	at 105 °C for 12h	62

[a] Reaction conditions: 1,6-diynyl ether **1a** (0.40 mmol), NBS (0.82 mmol), $PdBr_2$ (10 mol%), additive (10 mol%), solvent (2.0 mL), 105–130 °C, 8 h.

parameters, we found optimized reaction conditions consisting of $PdBr_2$ (10 mol%), and 10 mol% of $CuBr_2$ as an effective additive in 0.1 mol/L of THF at 130 °C. The desired product **2a** was isolated in 83% yield under this "standard condition" (entry 1). The Pd-catalyst was necessary, because no product was detected in the absence of any Pd-catalyst (entries 2 and 3). The Pd salt with halide anion ($PdCl_2$, $PdBr_2$ or $Pd(PPh_3)_2Cl_2$ alone) was effective for the reaction. Without $CuBr_2$, however, an inert reaction was observed when $Pd(OAc)_2$ and $Pd(dba)_2$ were utilized (entries 4–7). These results indicated that the halide anion was important for the initiation of the reaction. Interestingly, $CuBr_2$ worked as a Lewis acid could dramatically promote the reaction (entry 4 *vs.* 1).

Informed by this result, we next screened of other Lewis acids as promoters, including $FeBr_2$, $FeBr_3$, $CoBr_2$, $NiBr_2$ and $ZnBr_2$. However, none of the other Lewis acids offered any satisfactory results (entries 8–12). A smooth conversion was observed when CuBr was utilized, and the desired product **2a** could be isolated in 72% yield (entry 13). In this solvent, the reaction worked well in 1,2-dichloroethane to give the desired product **2a** in 77% yield (entry 14). However, switching to other laboratory solvents such as toluene, 1,4-dioxane, or DMF did not increase the efficiency (entries 15–17). Poor results were observed when the loading of $PdBr_2$ or $CuBr_2$ was lowered to 5.0 mol% (entries 18 and 19). In addition, the yield of product **2a** decreased from 130 °C to 105 °C with a reaction time of 12 h (entry 20).

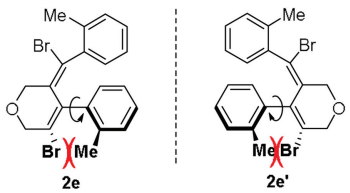


Scheme 2. Reaction scope studies. Reaction conditions: 1,6-diynyl ether **1** (0.40 mmol), NBS (0.82 mmol), $PdBr_2$ (10 mol%), $CuBr_2$ (10 mol%), THF (4.0 mL), 130 °C, 8 h. [a]Toluene (0.1 mol/L), 80 °C instead of THF (0.1 mol/L), and 130 °C.

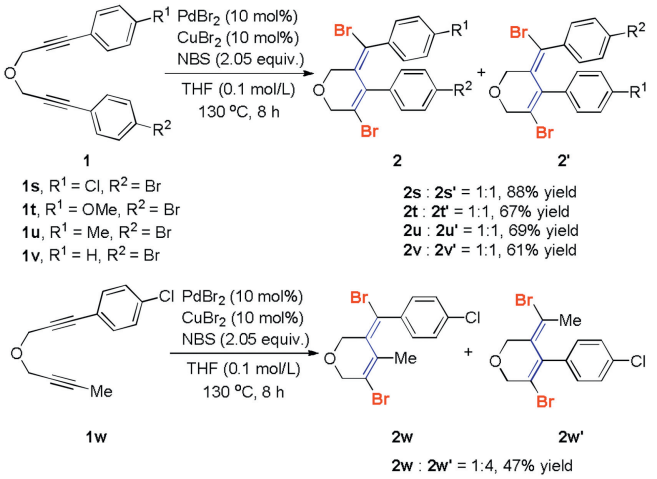
Once the reaction conditions were established, the scope of this reaction was next studied. Scheme 2 shows a variety of bromide functionalized (*E*)-3,6-dihydro-2*H*-pyrans with exocyclic double bond appendages. These were readily allowed when NBS was utilized as the halogen source. The reaction was quite general with respect to variation of the aryl groups on the alkyne moieties that attached with different electronic natures. Diverse series of products **2b**-**2n** were regio-specifically generated in good yields. The electron-neutral phenyl substituted 1,6-diynyl ether **2b** was produced in 69% yield. When the R group was attached at the phenyl moiety of 1,6-diynyl ether **1**, electron-donating alkyl substituents such as Me and *t*Bu proceeded smoothly. Note that nearly comparable yields of products **2c**, **2d**, and **2e** were also obtained, suggesting that the location of the methyl group at the *para*-, *meta*- or *ortho*-positions of phenyl ring had little impact on the efficiency. The corresponding product **2f** was obtained in 65% yield when R was a bulky tertiary butyl group.

Products **2e** and **2i** were obtained as a mixture of atropisomers as indicated by their proton NMR, they had axial chiral properties at room temperature (Scheme 3). When the aryl substituent attached to the alkyne terminus became more electron deficient, the reaction efficiency increased dramatically *versus* with the aryl substituent attached to the electron-rich alkyne terminus (Scheme 2). The presence of a mild electron-withdrawing fluorine atom at *para*- or *meta*-positions of the phenyl moiety were well tolerated in the reaction, affording the corresponding products **2g** and **2h** in 76% and 85% yields, respectively. Encouraged by these results, we expanded the reaction to the other halogen substituted electron-deficient arenes. The use of chlorine-containing *aryl* substituents of 1,6-diynyl ethers also resulted in good yields of the desired products (**2j** and **2k**). The substrate that contained a bromide atom (R = Br) at the 4-Ph moiety underwent the reaction to give product **2l** in good yield. A nice reaction for the 2-phenyl substituted substrate produced the expected product **2m** in 84% yield. The structure of product **2m** was established unambiguously by single-crystal X-ray crystallography diffraction analysis (CCDC: 1985748). The synthetically useful cynano group could be tolerated under slightly modified reaction conditions, providing the desired products **2o** and **2p** in modest yields. The ester group was tolerated, and **2q** was isolated in 86% yield in toluene as the solvent at 80 °C. Aryl, alkyl, and heterocyclic thienyl groups could be placed at the triple terminal position to provide the corresponding product **2r**. Unfortunately, strong electron-withdrawing groups such NO_2 and CF_3, are not compatible with the reaction.

The cyclization regioselectivity of 1,6-diynyl ether **1** bearing two different functional groups was next studied under standard reaction conditions (Scheme 4). For the 1,6-diyne **1t** with an electron-donating OMe group and an electron-withdrawing Br group substituted phenyl substituents at each alkyne terminus, two isomeric products **2t** and **2t'** were isolated as an inseparable mixture in 67% yield. The observed molecular ratio of **2t/2t'** was 1:1 as indicated by ^{1}H NMR, showed that the electronic nature of the phenyl ring had less impact on the site selectivity on the alkyne carbon atoms. The preference for cyclization of 1,6-diynyl ether **1u**, which individually bears a methyl group and a bromide group at the phenyl moieties, is also not obvious. The two resulting isomers



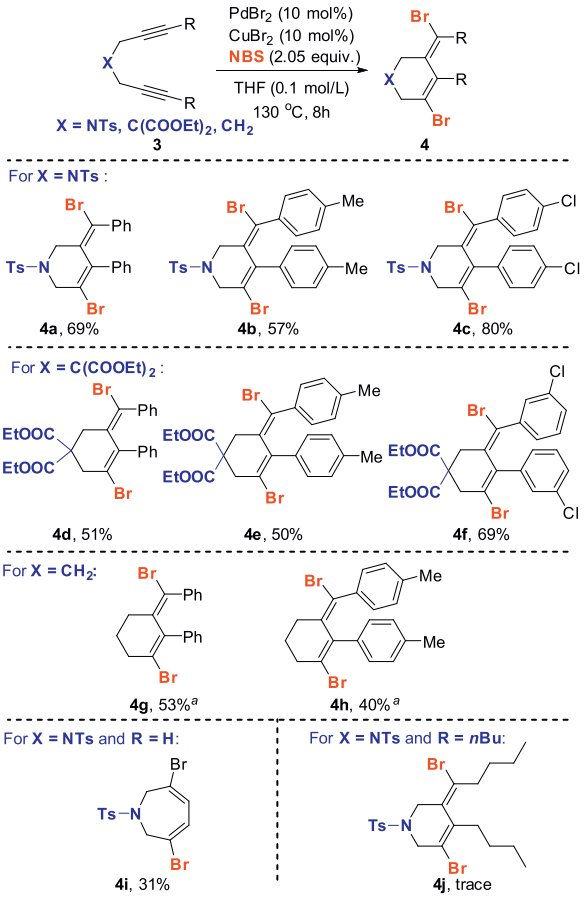
Scheme 3. Origination of the atropisomer **2e**.



Scheme 4. Substrate scope studies.

2u and **2u'** = 1:1 were obtained in a 1:1 molecular ratio. Similar results were observed for the other 1,6-diynyl ethers **1s** and **1v**, their corresponding products were isolated in 61%–88% yields. An example of this exception is the cyclization reaction of **w**, which contains a Me group and a 4-Cl phenyl group at each of the alkyne terminus. The two cyclic isomers **2w** and **2w'** were produced in a molecular ratio of 1:4 in 47% yield.

It should be noted we have tried the reaction of 1,6-diynyl ethers **1a** with *N*-chlorosuccinimide (NCS) or *N*-iodosuccinimide (NIS) that instead of NBS to participate in the reaction. In the case of



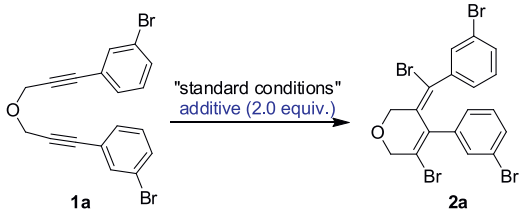
Scheme 5. Substrate scope studies. [a]Toluene (0.1 mol/L), 80 °C instead of THF (0.1 mol/L), 130 °C.

NCS, the conversion was rather complex but there was no desired product detected. Unfortunately, in the reaction **1a** with of NIS, no reaction was observed under the standard conditions.

To further explore the scope of this halocyclization reaction, diversified unactivated 1,6-diynes **3**, bearing a different –X moiety were evaluated (Scheme 5). Overall, a series of dibrominated *aza*- or carbocycles were successfully introduced affording the anticipated 3-methylenetetrahydropyridines or 3-methylenecyclohexenes **4a-4h** in 39%–80% yields. Similar to the standard 1,6-diynyl ether **1a**, compound **3a** that was prepared from bis(3-phenylprop-2-yn-1-yl)amine proceeded in the Pd/Cu co-catalyzed reaction smoothly for the generation of product **4a** in 69% yield. Treatment of substrates **3b** or **3c** bearing a Me or a Cl group at the phenyl moeity afforded the corresponding products **4b** and **4c** in good yields, respectively. The carbon chain 1.6-diyne **3d-3f**, which derived from diethyl malonate, was successfully transformed into their corresponding products **4d-4f** in 50%–69% yields. Gratifyingly, the acyclic carbon chain 1.6-diyne **4g** and **4h** could also be compatible to the halocyclization protocol, providing products **4g** and **4h** in serviceable yields, respectively. Interestingly, when compound 3-(prop-2-yn-1-yloxy)prop-1-yne, which bearing two hydrogen atoms at the alkyne moieties, was utilized as the starting material, the annulation occurred at the sterically less hindered terminal carbon, thus to generate the azepine product **4i** in 31% yield. However, for the dialkyl substituted substrate **3j**, which bearing two butyl groups at the 1,7-terminal positions of 1,6-diyne, the reaction was rather complex and almost no desired product **4j** was observed.

Unlike the previous reports that utilize a 2- to 3-folds stoichiometric excess of metal halides [9,11,13] to complete the catalytic cycle, this work only uses a catalytic amount of MBr_2 (M = Pd, Cu) in this reaction. Thus, the mechanism of the current reaction should be conceptually different from prior works. To shed light on the reaction, control experiments were conducted. When typical radical scavengers, such as TEMPO, BHT or styrene was added, the reaction became rather sluggish, and product **2a** was formed in very poor yields (Scheme 6). These results indicated that a radical-process should be involved in this tandem annulation reaction. To further confirm an organic radical species is involved in the overall process, electron paramagnetic resonance (EPR) experiment was conducted (Fig. 1). When compound **1a** was employed as a starting material under the standard conditions for 20 min, we successfully observed a radical signal by using DMPO as a trapping agent. This result suggested that the organic radical mediated annulation is involved in this annulation sequences.

We proposed a plausible mechanism based on the observations above and our recent studies on the NXS-mediated halocyclization reactions, (Scheme 7) [10]. Here, $PdBr_2$ was the essential catalyst (Table 1, entries 1–3 *vs.* 5), and we realized that the coordination of Pd^{II} and nucleophilic attack of Br^- should be the initial step for this



Entry	radical scavenger additive	**2a** (%)
1	TEMPO	trace
2	BHT	0
3	styrene	39

Scheme 6. Mechanistic studies. TEMPO = 2,2,6,6-tetramethyl-1-piperidinyloxy; BHT = 2,6-di-*tert*-butyl-4-methylphenol.

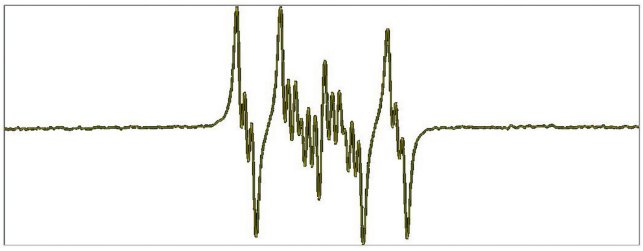
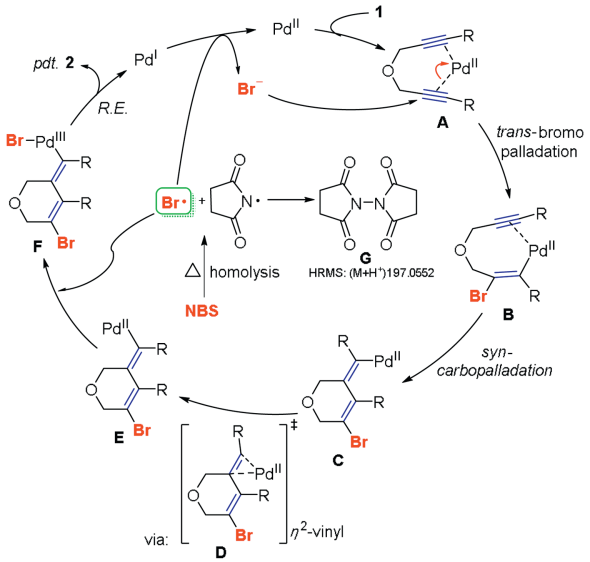


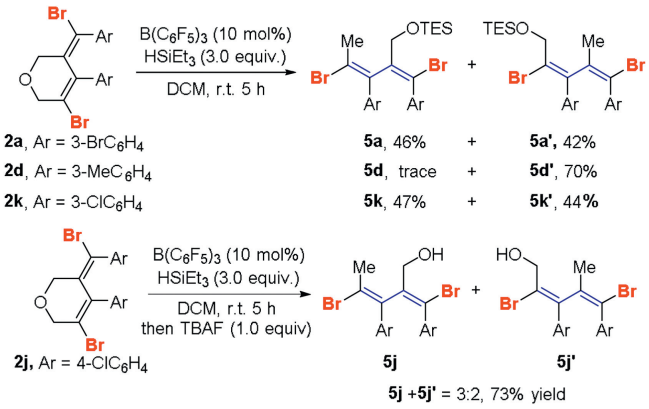
Fig. 1. EPR studies by using DMPO as an additive. DMPO = 5-dimethyl-1-pyrroline *N*-oxide.



Scheme 7. Proposed mechanism.

cascade transformation. Thus, the coordination reaction of the palladium cationic species to the alkyne functional groups might occur first to form a Pd-π-alkyne complex **A** [14]. The alkyne group was activated through this coordination to be more electrophilic. It then underwent a *trans*-bromopalladation reaction to deliver a brominated vinyl palladium species **B** [15]. The resulting intramolecular *syn*-carbopalladation to the tethered alkyne moiety can be envisioned to form the cyclic intermediate **C** [16]. Subsequently, *cis*/*trans* isomerization of the emerging Pd-vinyl species through a η^2-vinyl transition state **D** with regio- and stereoselective manner may give formal *anti*-carbopalladation complex **E** [17]. The Pd^{II}-catalyst would be oxidized by a bromide radical that originated from homolysis of NBS at heated temperature, to produce a hypervalent Pd^{III}-complex **F**. The subsequent reductive elimination (R.E.) of **F** delivered product **2** and low-valent Pd^{I}-species. In the further oxidations by bromide radical, this low-valent Pd^{I}-species can be oxidized to regenerate the active Pd^{II}-catalyst to complete the catalytic cycle, along with the generation of stoichiometric amounts of Br^- anions. The succinimide radical dimer **G** could be detected by HRMS ($(M+H)^+$: 197.0552).

The 1,3-diene structural motifs are widely found in natural products and pharmaceutical substances, rendering the scaffold very attractive. Thus, to utilize the diene modular structure of the resulting cyclic ether (*E*)-3,6-dihydro-2*H*-pyrans **2**, we next studied the $B(C_6F_5)_3$-catalyzed ring opening reaction of the C—O bonds using $HSiEt_3$ as a reducing agent to prepare stereo-defined dibromo substituted 1,3-dienes [18]. Indeed, a series of brominated 1,3-dienes **5** and **5'** were generated in excellent stereoselectivity in overall good yields (Scheme 8). For instance, the $B(C_6F_5)_3$-



Scheme 8. Stereoselective preparation of the1,3-dienes **5**. TES = triethylsilyl.

catalyzed reaction of **2a** with 3.0 equiv. of $HSiEt_3$ in dichloromethane proceeded smoothly to give 1,3-dienes **5a** and **5a'** in 46% and 42% yields, respectively. The two isomeric products **5a** and **5a'** were separable on a silica gel column. Compound **2k** participated in the reaction to give products **5k** and **5k'** in 47% and 44% yields, respectively. Product **2d** contains a 3-methylphenyl moiety, and the product 1,3-diene **5d'** was isolated in 70% yield. Moreover, in the $B(C_6F_5)_3$-catalyzed ring-opening reaction of **2j**, an inseparable mixture of 1,3-diene alcohol **5j** and **5j'** was produced in 73% yield following by treatment of 1.0 equiv. of TBAF. The dibromide substituted dienes might be served as a valuable building block for the preparation of π-conjugated alkenes.

In conclusion, we developed a novel Pd/Cu co-catalyzed cascade halocyclization reaction, consisting of a *trans*-halopalladation, formal *anti*-carbopalladation, folllowed by a terminating bromide radical mediate oxidative addition/reductive elimination sequences. A series of stereo-defined dibromo-substituted heterocyclic molecules or methylene cyclohexenes were produced in good to excellent yields. Further $B(C_6F_5)_3$-catalyzed ring opening reaction of the dihydropyrans afforded a series of synthetically useful brominated 1,3-dienes with excellent stereoselectivity. Mechanistically, a *trans*-halopalladation/formal *anti*-carbopalladation/and a bromide radical promoted Pd^{II}-Pd^{III}-Pd^{I}-Pd^{II} catalytic cycle were proposed to be involved for the formation of these dibromo-substituted products.

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.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2021.03.030.

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