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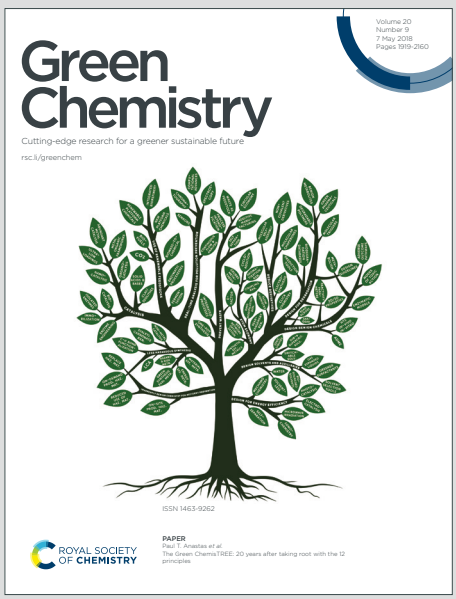

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Electrochemical Oxidative Cyclization of Olefinic Carbonyls with Diselenides

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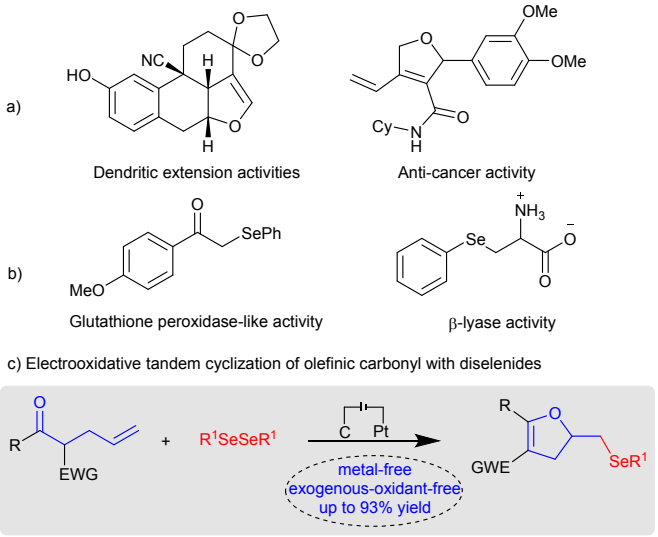
The tandem cyclization of olefinic carbonyls with easily accessible diselenides facilitated by electrochemical oxidation has been successfully developed, which provides an environmentally friendly manner for the construction of C–Se and C-O bonds simultaneously. A series of seleno dihydrofurans and seleno oxazolines, bearing fragile heterocycles, subtle C-I bond and supernumerary vinyl groups, were forged with this elegant chelation strategy. Neither metal catalysts nor external chemical oxidants is required to promote this transformation.

The dihydrofurans are important structural skeletons in a wide range of natural products, as well key structural motifs in organic synthesis such as aromatization, Heck reaction, ring opening of the reactive enol ether moiety, etc.[1] In the view of the drug design, dihydrofurans were also displayed to act as potential lead compounds (Scheme 1, a).[2] Thus, a variety of synthetic methods have been developed for the construction of this core moiety by building of C-O bonds.[3] Among the various synthetic routes, oxidative cyclization of 1,3-dicarbonyl compounds with alkenes provided an excellent access for the synthesis of dihydrofurans owing to the acidic α-H of carbonyls and electronegativity of the oxygen atom of carbonyls.[4] In addition, tandem cyclization of the olefinic carbonyl compounds has been utilized as an effective approach to introduce functional groups into the dihydrofuran skeletons.[5] Recently, relevant developments have been independently reported by Guo, Li, Liu et al.[5c-5g] Despite the progress achieved in this area, these above methods required excess oxidants, chemical additives and/or expensive metal catalysts, which not only limit the potential application to a certain extent but also bring about the possible metal residual that is adverse for the further application. Therefore, it is necessary and appealing to develop a more effective and sustainable method to construct dihydrofuran derivatives using olefinic carbonyl compounds.



Scheme 1 The application and synthesis of dihydrofurans/ organoselenium compounds

The introduction of selenium atoms into organic molecules has attracted much attention in pharmaceutical, agrochemical and material industries because of the special biological and chemical properties (Scheme 1, b).[6] Due to the wide applications of selenium-containing organic compounds, continuous endeavors have been made to develop efficient synthetic methods to access these molecules.[7] Especially, the construction of C-Se bonds has become a research hotspot in the last decade. In addition, the seleno group could be manipulated readily under suitable conditions owing to its versatile functionality.[8] Compared with other selenium-reagents (PhSeBr, PhSeCl, PhthSe, etc), diselenides are a kind of easily accessible and operatable selenium-reagents in organic synthesis, which provide a good choice for C-Se bonds formation.

In recent years, electrochemical synthesis has been widely recognized as a sustainable and environmentally benign

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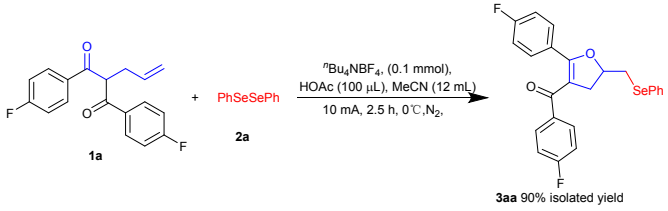
synthetic tool in synthetic chemistry.[9] Electrochemistry offers a direct means of generating active intermediate, which is superior than the classical chemical process. A lot of reports have been made on cross-coupling and difunctionalization of alkenes by electrochemistry.[10] Given that the importance of selenium-containing compounds in pharmaceutical, agrochemical and material industries, we report herein an electrochemical oxidative cyclization of olefinic carbonyls with diselenide toward C–Se and C-O bonds formation. A series of functionalized dihydrofurans and oxazolines were synthesized in the absence of metal catalysts and external oxidants (Scheme 1, c).

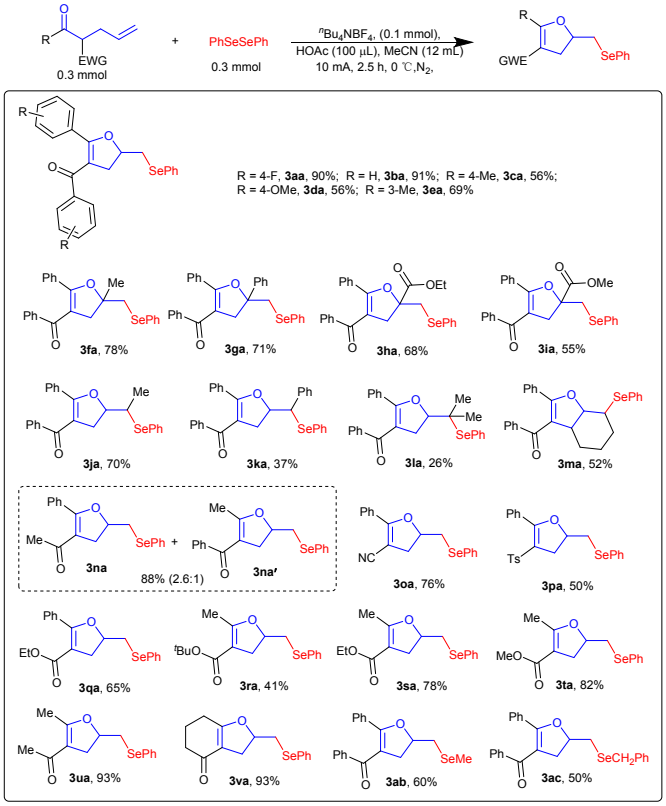
Table 1. Optimization of the Electrochemical Oxidative Cyclization[a]



entry	variation from standard conditions	yield (%)[b]
1	no HOAc	86
2	MeOH (12mL)	0
3	MeCN/H_2O (11 mL/1 mL)	0
4	MeCN/HFIP (11 mL/1 mL)	0
5	PhSeSePh (0.2mmol)	71
6	30℃	68
7	^{n}Bu$_4$NPF$_6$, (0.1 mmol),	88
8	TBAI (0.1mmol)	23
9	C(+)/Fe(-)	68
10	C(+)/C(-)	56
11	Air	53
12	no electric current	0

[a] Standard conditions: **1a** (0.3 mmol), **2a** (0.3 mmol), ^{n}Bu$_4$NBF$_4$ (0.25 mmol), MeCN (12 mL), HOAc (100 μL), C anode, Pt cathode, undivided cell, constant current = 10 mA (j_{anode} = 4.4 mA/cm^2), 0 °C, N_2, 2.5 h. [b] ^{19}FNMR yield, 1-fluoro-3-methylbenzene as an internal standard.

Initially, we commenced the electrochemical oxidative cyclization reaction by using 2-allyl-1,3-bis(4-fluorophenyl)propane-1,3-dione (**1a**) with diphenyl diselenide (**2a**) as model substrates in an undivided electrolytic cell. Encouragingly, utilizing ^{n}Bu$_4$NBF$_4$ as the supporting electrolyte, HOAc as additive and acetonitrile as solvent, (4-fluorophenyl)(2-(4-fluorophenyl)-5-((phenylselanyl)methyl)-4,5-dihydrofuran-3-yl)methanone (**3aa**) could been obtained in 90% isolated yield with graphite rod anode and platinum sheet cathode (Table 1). The yield of the desired product decreased slightly in the absence of HOAc (Table 1, entry 1). The reaction was completely inhibited under other solvents with the formation of side products (Table 1, entries 2-4). When the amount of diphenyl diselenide was reduced from 0.3 mmol to 0.2 mmol, only 71% yield could be obtained (Table 1, entry 5). It was remarkable that only 68% yield was got when the reaction was conducted at 30 °C (Table 1, entry 6). As to the other supporting electrolytes, ^{n}Bu$_4$NPF$_6$ showed a similar reactivity compared with ^{n}Bu$_4$NBF$_4$, while product **3aa** was formed in diminished yield when TBAI was used instead of ^{n}Bu$_4$NBF$_4$ (Table 1, entries 7-8). Subsequently, various cathodes were examined. Replacing platinum sheet with graphite rod or iron plate led to decreased yields obviously (Table 1, entries 9-10). When the reaction was opened to air, the yield was nearly reduced by half (Table 1, entry 11). Control experiments indicated that no conversion occurred without electricity (Table 1, entry 12).

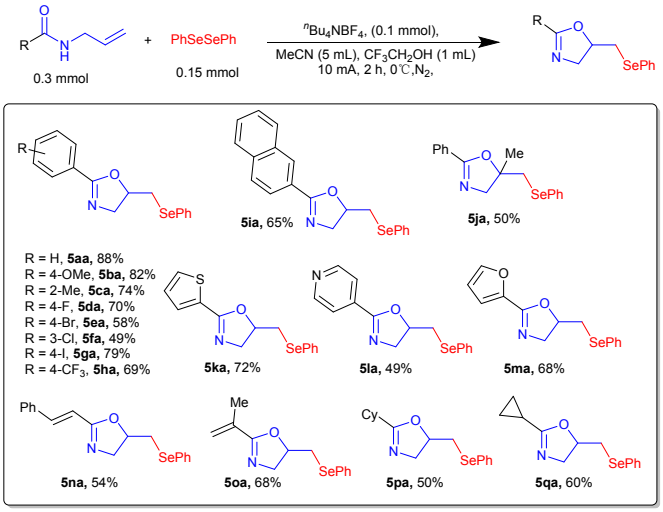


[a] Standard conditions: **1** (0.3 mmol), **2** (0.3 mmol), ^{n}Bu$_4$NBF$_4$ (0.25 mmol), MeCN (12 mL), HOAc (100 μL), C anode, Pt cathode, undivided cell, constant current = 10 mA (j_{anode} = 4.4 mA/cm^2), 0 °C, N_2, 2.5 h.

Scheme 2. Scope of the Olefinic Carbonyls with Diselenides[a]

With the optimized conditions in hand, we turned to explore the substrate scope of the tandem cyclization reactions in Scheme 2. Symmetric olefinic carbonyls bearing electron-withdrawing groups or electron-donating groups on the para/meta position of the phenyl ring could react smoothly with diphenyl diselenide to afford the corresponding dihydrofuran compounds in moderate to good yields (**3aa**-**3ea**). It is noteworthy that γ-substituted (Ph, Me, CO_2Et, CO_2Me) symmetric olefinic carbonyls could also be transformed to the target products in satisfactory yields (**3fa**-**3ia**). Subsequently, we continued to investigate the effects of δ-substituted substrates under standard conditions. Obviously, the steric hindrance has a great influence on this transformation (**3ja**-**3la**). To our delight, olefinic carbonyls containing γ, δ-disubstituted groups, could also react with 1,2-diphenyldiselane to generate the target molecule in 52% yield (**3ma**). Inspired by the above results, we attempted to expand this method using unsymmetric olefinic carbonyls. 2-allyl-1-phenylbutane-1,3-dione could reacted with **2a**, furnishing 2.6:1 separable mixture with excellent yield (**3na** and **3n'a**). Unsymmetric olefinic carbonyls, α-substituted group (CN, CO_2Et, Ts) instead of acyl group, worked in 50-76% yields (**3oa**-**3qa**). In addition, alkyl

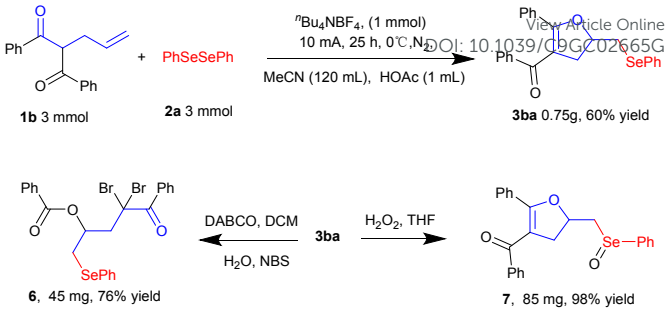
substances such as **1r**-**1u** were amenable in the current reaction system, and the corresponding dihydrofurans were obtained in moderate to good yields (**3ra**-**3ua**). A surprisingly high isolated yield was obtained using 2-allylcyclohexane-1,3-dione **1v** as a substrate (**3va**). Next, two diverse diselenides were employed as substrates to explore the scope of this reaction. It is noteworthy that dimethyl diselenides and dibenzyl diselenides were also good reaction candidates and furnished the target product in acceptable yields (**3ab** and **3ac**).



[a] Standard conditions: **4** (0.3 mmol), **2a** (0.3 mmol), $^{n}Bu_4NBF_4$ (0.1 mmol), MeCN (5 mL), CF_3CH_2OH (1 mL), C anode, Pt cathode, undivided cell, constant current = 10 mA (j_{anode} = 4.4 mA/cm^2), 0 ^{o}C, N_2, 2 h.

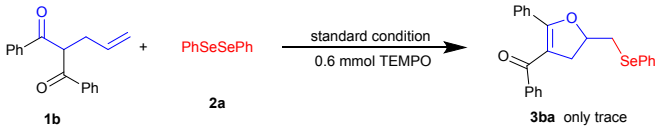
Scheme 3. Scope of the Unsaturated Amides with Diselenides

Encouraged by the efficient tandem cyclization of olefinic carbonyls to construct dihydrofurans, we next turned our attention to expand this protocol to provide oxazolines using unsaturated amides and 1,2-diphenyldiselane. To our delight, a series of oxazolines were produced successfully under slightly modified conditions, as shown in Scheme 3. *N*-allylamides containing electron-rich aryl groups (Ph, 4-OMePh, 2-MePh, Naph) could be employed easily to construct C–Se and C-O bond with 1,2-diphenyldiselane, affording the corresponding seleno oxazolines in moderate to excellent yields (**5aa**-**5ca**, **5ia**). The sensitive halogen groups, especially subtle C-I bond, were compatible with this strategy (**5ea**-**5ga**). No obvious negative effect was observed for the strong electron withdrawing group substituted substrate (**5ha**). It is worth noting that the methyl derivative containing a quaternary carbon center could be obtained with acceptable yield (**5ja**). *N*-allylamides, with fragile thiophene, furan and pyridine rings, also were well-tolerated to furnish the corresponding cyclization products with moderate yields (**5ka**-**5ma**). Gratifyingly, *N*-allyvinylamide and *N*-allycinnamide were also good candidates, and could be converted to the desired products with 68% and 54% yields, respectively (**5na** and **5oa**). Similarly, the desired products can also be accessed from *N*-allyalkylamides (**5pa** and **5qa**).



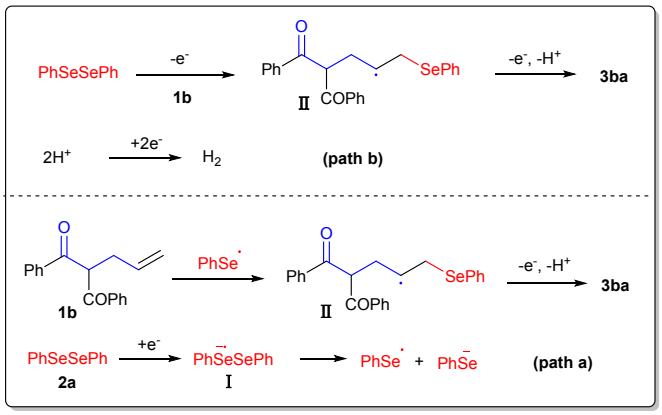
Scheme 4. Gram-Scale Tandem Cyclization of 1b and Derivatization of 3ba

To further demonstrate the potential application of this electrochemical oxidative tandem cyclization, a gram scale reaction was carried out. With the established electrochemical strategy, the reaction could proceed smoothly and **3ba** was obtained in 60% yield. Furthermore, the versatility of **3ba** was also pursued. A ring opening process of **3ba** led to the formation of valuable α, α-dibromoketone compound **6** with NBS and DABCO as the electrophilic halogen source and the catalyst, respectively. In addition, selenoxide **7** was obtained via oxidation using H_2O_2 in nearly quantitative yield.



Scheme 5. Control Experiments

To explore the reaction mechanism, control experiments were conducted under different conditions. Only trace amount of the product was detected when 2 equiv. of TEMPO was added (Scheme 5). On the other hand, the cyclic voltammetry experiments of **2a**. The oxidation and reduction peaks of **2a** could be observed at 1.8 V and -1.5 V vs. Ag/AgCl, respectively. Therefore, the diphenyl diselenide **2a** may be involved in both oxidation and reduction processes in electrochemical conditions. On the basis of aforementioned experimental results and literature reports,[7h-7j] a proposed mechanism is outlined in Scheme 6. Firstly, diphenyl diselenide is reduced at cathode to give an anion radical intermediate **I**, which is further decomposed to give phenylselenium radical and phenyl selenium anion. Thereafter, phenylselenium radical is captured by alkenyl of **1b** to form alkyl radical **II**, which is further oxidized at anode. The fast ring closing followed by nucleophilic attack of the oxygen atom of carbonyl, as well as deprotonation furnish to the formation of desired product (path a). Alternatively, the pathway in which **1b** reacts with phenylselenium radical to form the alkyl radical **II**, could not be completely ruled out (path b).



Scheme 6. Proposed Reaction Mechanism

Conclusions

In summary, we have developed an economical and practical methodology for C–Se and C-O bonds forming via electrochemical oxidative tandem cyclization. An array of seleno dihydrofurans and oxazolines were obtained successfully in moderate to excellent yields. Importantly, the gram scale experiment and the easy following derivatization demonstrate the potential application of this protocol in pharmaceutical and synthetic research.

Acknowledgements

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