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Electrochemical Difunctionalization of Olefines: Access to Selenomethyl-Substituted Cyclic Ethers or Lactones

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Abstract. A metal- and oxidant-free electrochemical method for preparing selenomethyl-substituted cyclic ethers or lactones via difunctionalization of olefines is presented. A series of selenomethyl-substituted cyclic ethers, particularly 9- and 11- membered, selenomethyl-substituted lactones (4–6 membered), and selenomethyl-substituted phthalides can be obtained via this reaction. This method features convenient operation, an electron as oxidant, and ammonium iodide as electrolyte, thereby making it a green synthesis method.

Keywords: electrochemistry, difunctionalization of olefines, cyclic ethers, lactones, phthalides

Cyclic ethers and lactones are the two most common structural units in nature and exist widely in the areas of agrochemicals, pharmaceuticals, and polymer materials.[1] For example, Zoapatanol (Figure 1), a natural diterpenoid oxepane, is isolated from the leaves of a Mexican zoapatle plant, namely, *Montanoa tomentosa* and is found to display antifertility activity.[2] Normally, lactones are known for their fruity aroma. For example, tetrahydro-6-pentyl-2H-pyran-2-one (DDL),[3] is a type of decalactone that is naturally found in fruits, such as coconut and milk products, and has a large market demand, given its application to flavor, pest control,[4] and as food additive.[5] In addition, a series of lactones, such as γ-lactones, and δ-lactones, is extensively used in cosmetic fragrance and flavor considering their own characteristic odors (Figure 1).[6] Accordingly, a various of synthetic routes have been developed to construct these scaffolds. The attractive methods for the synthesizing of these skeletons are ring closing metathesis (RCM) (Scheme 1a)[7] and direct construction of C-O bond within corresponding acids or alcohols.[8] In 2005, Togo and coworker reported the photochemical preparation of 2-substituted 1,3-dioxane and tetrahydrofuran from alcohols with polymer-supported hypervalent iodine reagent in the presence of I_2 (Scheme 1b).[9] Over the past decade, the syntheses of these molecules have been extensively investigated, and significant achievements, have been reached. However, shortcomings, such as: harsh reaction conditions, multistep reactions , and formation of undesired by-products, are ubiquitous in most routes. Therefore, developing new tactics is desirable.

Organoselenium compounds have been extensively studied given their wide application to drug design, functional organic materials, food chemistry, and molecular probes.[10] Given the importance of organoselenium compounds, introducing selenium to organic compounds has attracted considerable attention. Recently, our group has reported a copper-catalyzed decarboxylative/click cascade reaction to construct selenium-containing triazole compounds. Three of the 5-selanyl-1,2,3-triazoles have

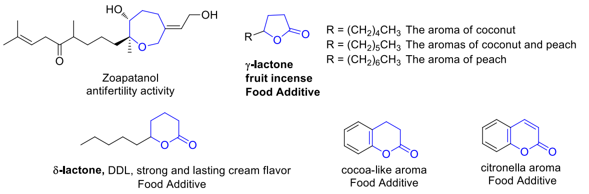
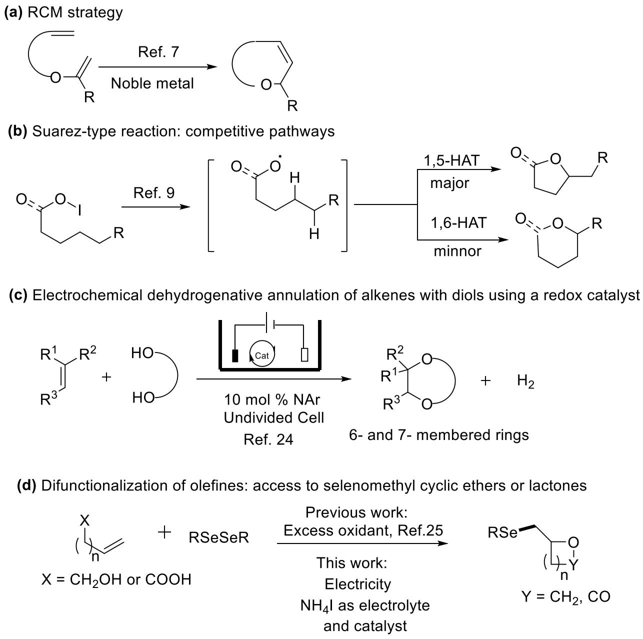


Figure 1. Bioactive molecules containing a cyclic ether moiety and food additives containing a lactone moiety.



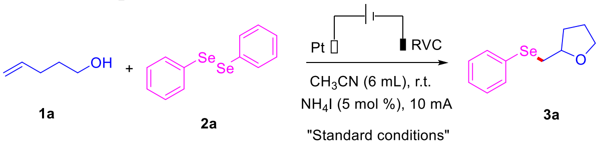
Scheme 1. Synthesis of O-containing heterocycles

been found to display potent inhibitory activities against MGC-803 cell lines, and one kind of 5-selanyl-1,2,3-triazoles is found to be cytotoxic agents to T-24 cell lines.[11] Inspired by these reports, we suspect that introducing selenium to lactones and cyclic ethers is of significant interest in the pharmaceutical industry and academic research (Scheme 1d).

Currently, the difunctionalization of alkenes is a powerful and straightforward method for synthetizing various biologically active compounds and natural products.[12] Generally, this process mainly focuses on transition metal catalysis (including Pd,[13] Ni,[14] Cu,[15] Ag,[16] Re,[17] Au,[18], Fe[19], and others[20]), metal-free catalysis (but requires [over-] stoichiometric amount of oxidants),[21] and photocatalysis.[22] In recent years, electrochemical synthesis has attracted considerable attention in synthetic organic chemistry considering its utilization of electrons as “reagents” to accomplish the redox process; this process is a powerful tool for synthesizing organic compounds.[23] Xu’s group successfully realized the difunctionalization of alkenes via electrochemical synthesis in a simple and moderate condition to construct saturated O-heterocycles (Scheme 1c).[24] Although there were some tactics for the construction of selenomethyl-substituted ether/lactone products in the previous reports, the oxidants were essential (Scheme 1d).[25] Given our consistent and continuous interest in synthesizing functionalized heterocycles via electrochemical synthesis,[26] we reported an electrochemical method for preparing selenomethyl-substituted cyclic ethers and lactones via difunctionalization of olefines.

We selected pent-4-en-1-ol (**1a**) and diphenyl diselenide (**2a**) as model substrates for optimizing reaction conditions (Table 1). Electrolysis was conducted in an undivided cell equipped with a reticulated vitreous carbon (RVC) cathode and a platinum anode. The optimal yield of 3-(phenylselanyl) tetrahydro-2H-pyran (**3a**) (98%) was obtained when constant current of 10 mA and 5 mol% NH_4I were used as the electrolyte and electrocatalyst. Control experiments showed that the reaction was completely abolished when NH_4Br or NH_4Cl (Entries 2 and 3) was chosen as the electrolyte or when dichloroethane (Entry 9) was adopted as the solvent or when no electric current (Entry 11) was used. Selecting KI as the electrolyte (Entry 5), using another solvent (e.g., MeOH [entry 8]), and exchanging electrode (Entry 14) were found to be minimally efficient in this reaction. However, using another electrolyte (e.g., TBAI [Entry 4]), using another solvent (e.g., DMF or DMSO [Entries 6 and 7]), choosing Pt plate or RVC (Entries 12 and 13) as electrode significantly decreased product (**3a**) yield.

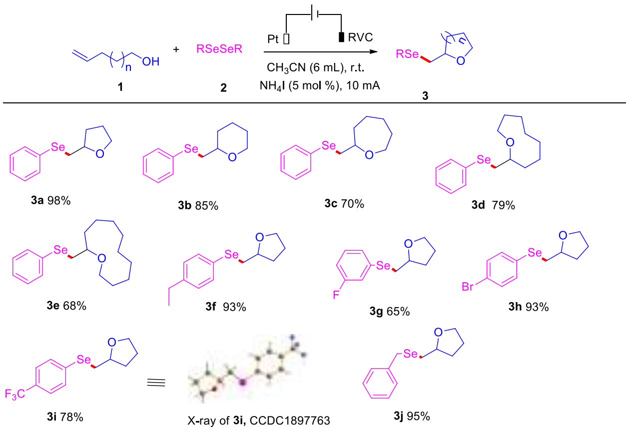
Table 1. Optimization of reaction conditions.[a, b]



Entry	Deviation from standard conditions	Yield (%)[b]
1	None	98%
2	NH_4Br as the electrolyte	NR [c]
3	NH_4Cl as the electrolyte	NR
4	TBAI as the electrolyte	39%
5	KI as the electrolyte	75%
6	DMF as the solvent	54%
7	DMSO as the solvent	62%
8	CH_3OH as the solvent	88%
9	CH_3ClCH_3Cl as the solvent	NR
10	1,4-Dioxane as the solvent	trace
11	no electricity	NR
12	Pt plate (1 cm × 1 cm) as the anode and cathode	45%
13	RVC (1 cm × 1 cm) as the anode and cathode	36%
14	RVC (1 cm × 1 cm) as the anode, Pt plate (1 cm × 1 cm) as the cathode	82%

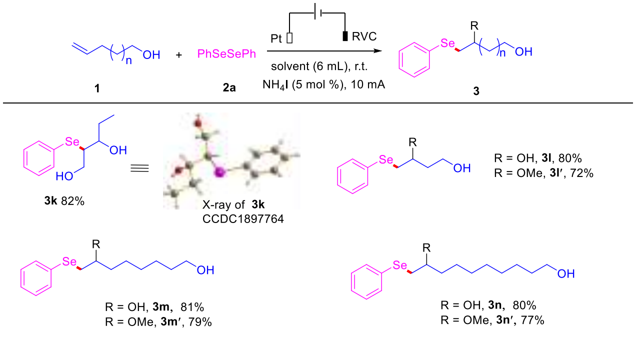
[a]Reaction conditions: undivided cell with Pt plate anode (1 cm × 1 cm) and reticulated vitreous carbon (RVC) cathode (100 PPI, 1 cm × 1 cm × 1.2 cm), constant current = 10 mA, **1a** (0.5 mmol), **2a** (0.25 mmol), NH_4I (5 mol%), and solvent (6 mL) under air atmosphere at room temperature for 6 h. [b]Isolated yields. [c]NR = no reaction.

We explored the scope of the substrate with optimized conditions by first varying the olefinic alcohols (**1**) of different lengths (Scheme 2). The reaction showed broad compatibility with various olefinic alcohols, and the corresponding products 5–7-, 9-, and 11-membered ring selenomethyl-substituted ethers were obtained in good to excellent yields (**3a**–**3e**) (Scheme 2). We then subjected various selenides to proceed to selenylation with pent-4-en-1-ol **1a** (Scheme 2). When the para position of phenyl selenide was replaced with alkyl (ethyl) electron-withdrawing group (Br or CF$_3$), the reaction generated the expected products (**3f**, **3h**, and **3i**) in good to excellent yields, and the structure of **3i** was confirmed via X-ray analysis. [27] Furthermore, when the meta position of phenyl selenide was replaced with -F, the target product **3g** was obtained in a satisfactory yield. Notably, benzyl selenide can also undergo this reaction smoothly and deliver the final product **3j** in excellent yield.



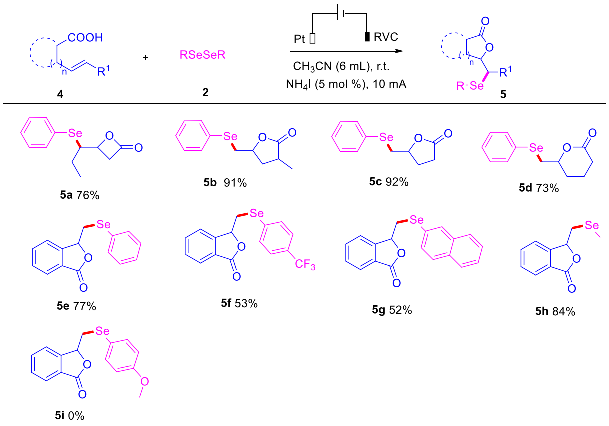
Scheme 2. Substrate scope of olefinic alcohols and selenides. Reaction conditions: Undivided cell with Pt plate as the anode (1 cm × 1 cm) and reticulated vitreous carbon (RVC) as the cathode (100 PPI, 1 cm × 1 cm × 1.2 cm), constant current = 10 mA, **1** (0.5 mmol), **2** (0.25 mmol), catalyst (5 mol%), and solvent (6 mL) under atmospheric air at room temperature for 6 h. Isolated yields.

By contrast, we cannot obtain the corresponding 4-, 8-, and 10- membered ring selenomethyl-substituted ethers in Scheme 2, but we achieved their ring opening products (Scheme 3). When CH$_3$CN was chosen as the solvent, we obtained ring opening products with R = hydroxyl groups (**3k**–**3n**), and the structure of **3k**, which obtained from pent-1-en-3-ol, was confirmed via X-ray analysis. However, when CH$_3$OH was chosen as solvent unexpectedly, we obtained other ring opening products with R = methoxyl groups (**3l′**–**3n′**).



Scheme 3. Substrate scope of olefinic alcohols. Reaction conditions: Undivided cell with Pt plate as the anode (1 cm × 1 cm) and reticulated vitreous carbon (RVC) as the cathode (100 PPI, 1 cm × 1 cm × 1.2 cm), constant current = 10 mA, **1** (0.5 mmol), **2a** (0.25 mmol), catalyst (5 mol%), and solvent (6 mL) under atmospheric air at room temperature for 6 h. ^{b}Isolated yields.

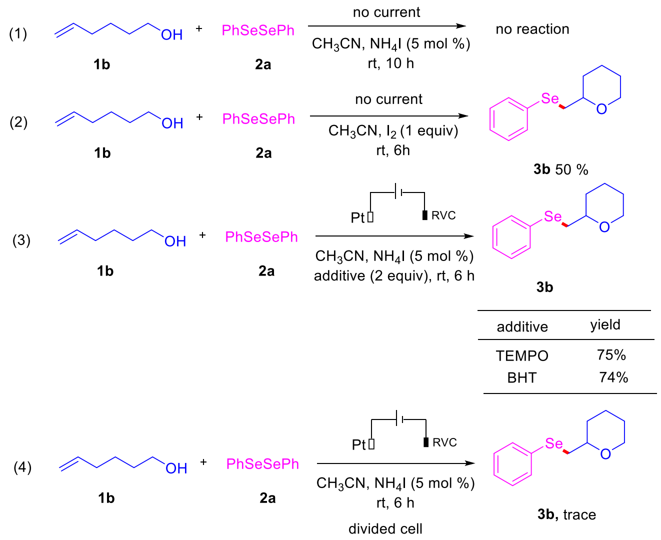
Given that olefinic acids and enols had double bonds and OH groups, we speculated that olefinic acids may also occur in the abovementioned reaction. Therefore, we tested various olefinic carboxylic acids (Scheme 4). Moreover, a series of olefinic carboxylic acids was also well tolerated under the optimal conditions and resulted in the desired products 4- to 6- membered ring selenomethyl-substituted lactones in good to excellent yields (**5a**–**5d**).



Scheme 4. Substrate scope of olefinic acids and selenides. Reaction conditions: Undivided cell with Pt plate as the anode (1 cm × 1 cm) and reticulated vitreous carbon (RVC) as the cathode (100 PPI, 1 cm × 1 cm × 1.2 cm), constant current = 10 mA, **4** (0.5 mmol), **2** (0.25 mmol), catalyst (5 mol%), and solvent (6 mL) under atmospheric air at room temperature for 6 h. Isolated yields.

Phthalides, which contain a γ-lactone fused with an aromatic ring, are ubiquitous structural motifs in medicinal and natural products.[28] Many approaches have been explored to achieve this goal.[29] The primary disadvantages of these methods are long reaction time, low yield, and use of expensive metal catalysts. Thus, 2-vinylbenzoic acid (**4e**) with different selenides were investigated via the current electrosynthesis. Furthermore, diphenyl diselenide with a strong electron-withdrawing group, such as -CF$_3$, can produce the expected product with satisfactory yield. Moreover, diphenyl diselenide with a fused ring structure can also transform the target product with 52% yield. Finally, diselenides with alkyl groups were found to proceed well with 2-vinylbenzoic acid (**4e**) to provide the target product **5h** at 84% yield. However, the reaction of 2-vinylbenzoic acid (**4e**) with diphenyl diselenides bearing electron-donating groups (OMe) failed to produce the desired product **5j**. Compared with previous literature reports, our strategy for building phthalide structures can circumvent the drawbacks mentioned above.

Some control experiments were performed to gain additional insights into this selenylation reaction. Initially, hex-5-en-1-ol (**1b**) and diphenyl diselenide (**2a**) were reacted with NH_4I in the absence of current. No reaction was observed between the two raw materials (Scheme 5, Eq. 1). Subsequently, hex-5-en-1-ol (**1b**) and diphenyl diselenide (**2a**) were reacted with I_2 without current, and product **3b** was obtained with 50% isolated yield (Scheme 5, Eq. 2). Finally, TEMPO/BHT, **1a,** and **2a** were reacted under standard conditions, and the target product **3b** was produced in 75%/74% isolated yields, correspondingly (Scheme 5, Eq. 3). These results might exclude the possibility of a radical mechanism. We also did a controlled experiment in a divided cell, no product **3b** was obtained (Scheme 5, Eq. 4). This result proved that the regeneration of iodide is from the reduction of iodine at cathode.



Scheme 5. Control experiments.

Lastly, cyclic voltammetry experiments were performed to enhance the understanding of the reaction relationship between substrates. As shown in Figure 2, curve b (substrate **1a**) and curve c (substrate **2a**) had no oxidation peak when the potential was less than 0.8 V. In order to investigate whether the NH_4I plays a redox role in the reaction, we had measured the curve of NH_4I separately (curve d).The CV of NH_4I (curve d) displays two oxidation peaks at 0.38 V (Ox_1) and 0.67 V (Ox_2) versus Ag/AgCl, and these two anodic peaks of iodide were attributed to I^- anion to I_2 and I_3^- to I_2, respectively. The curves of NH_4I with **1a** (curve e) and NH_4I with **2a** (curve f) were also measured, respectively, as shown in Figure 2, and there was no change, which indicated that I^- did not oxidize the substrates in this reaction.[30]

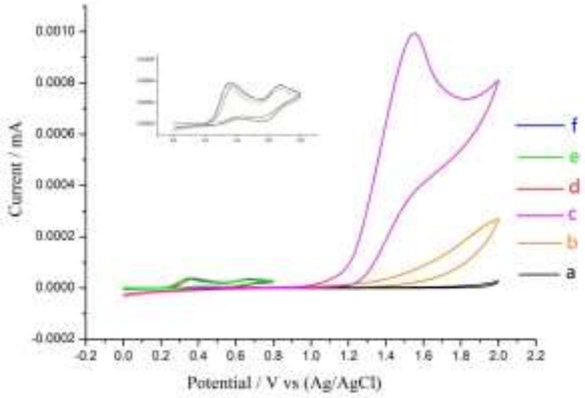
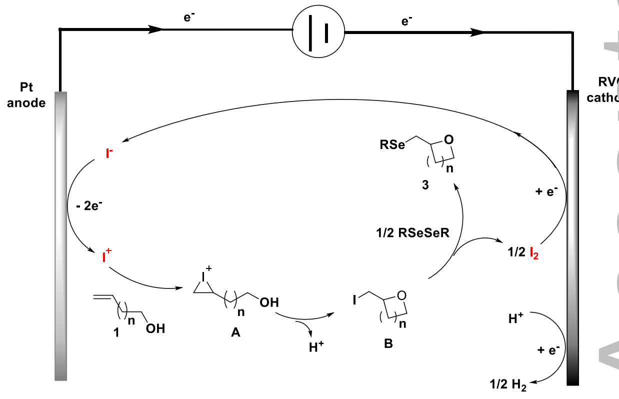


Figure 2. Cyclic voltammograms of reactants and their mixtures in 0.1 M $LiClO_4/CH_3CN$ using a glassy carbon disk (3 mm diameter), Pt disk, and Ag/AgCl (in saturated KCl solution) as working counter, and reference electrodes, respectively, at a scan rate of 100 mV/s: (a) background, (b) **1a** (10 mmol/L), (c) **2a** (10 mmol/L), (d) NH_4I (2 mmol/L) , (e) NH_4I (2 mmol/L)+ **1a** (10 mmol/L), (f) NH_4I (2 mmol/L)+ **2a** (10 mmol/L)

On the basis of the above mentioned results, a plausible mechanism was proposed (Scheme 6). At the anode, the electrochemical oxidation of I^- initially results in the formation of I^+ by losing two electrons, which then react with olefinic alcohols to provide the iodonium cations intermediate **A**. The intermediate **A** undergoes intramolecular cyclization and releases a proton to afford intermediate **B**. Finally, the target product **3** and a half molar equivalent of I_2 are obtained by a rapid chemical selenation.[31] At the cathode, The I_2 and proton are reduced to the iodine anion and hydrogen, which completes the rection cycle.



Scheme 6. Plausible mechanism.

In summary, we have developed a metal- and oxidant-free electrolysis method for synthesizing selenomethyl-substituted cyclic ethers and lactones in the presence of a catalytic amount of ammonium iodide at room temperature from selenides and unsaturated alcohols or acids, respectively. Notably, medium-sized ethers (7-, 9-, and 11-membered ring) and 4–6-membered ring lactones can be obtained smoothly via this straightforward route because these transformations are difficult in conventional technologies. Moreover, this environment-friendly strategy is applicable to preparing a wide array of Se-containing phthalides. Some notable features of this electrochemical method are broad substrate scope, convenient operation, inexpensive electrolyte requirement, high conversion efficiency, and practicality.

Experimental Section

Pent-4-en-1-ol **1a** or pent-4-enoic acid **4c** (0.5 mmol, 1.0 equiv), diphenyl diselenide (0.25 mmol, 0.5 equiv), and NH_4I (0.1 mmol, 5 mol%) were placed in a 10 mL three-necked round-bottomed flask. The flask was equipped with a condenser, RVC (100 PPI, 1 cm × 1 cm × 1.2 cm) cathode, and a platinum plate (1 cm × 1 cm) anode. CH_3CN (6 mL) was added. Electrolysis was performed at room temperature using a constant current of 10 mA until complete consumption of the substrate (monitored by TLC, approximately 6 h). Water (30 mL) and ethyl acetate (30 ml) were added. The phases were separated, and the aqueous phase was extracted with ethyl acetate (2 × 30 mL). The combined organic solution was dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was subjected to chromatography via silica gel elution with ethyl acetate/petroleum ether to yield product 2-((phenylselanyl)-methyl)tetrahydrofuran **3a** or 5-((phenylselanyl)methyl)dihydrofuran-2(3H)-one **5c**.

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[27] CCDC 1897763 (**3i**) and CCDC 1897764 (**3k**) contain the supplementary crystallographic data for this paper. The data can be obtained free of charge from the Cambridge CrystallographicData Centre via www.ccdc.cam.ac.uk/data_request/cif.

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Electrochemical Difunctionalization of Olefines: Access to Selenomethyl-Substituted Cyclic Ethers or Lactones

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