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Versatile electrooxidative amino- and oxyselelenation of alkenes†

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Herein, we describe a general and eco-friendly electrochemical methodology for amino- and oxyselelenation of alkenes under transition-metal catalyst- and additional-oxidant-free conditions. This electro-catalytic difunctionalisation reaction exhibits excellent chemoselectivity, ample substrate scope, and high functional group tolerance. To our delight, the selenation products (118 examples, up to 99% yield) were constructed from various alkenes including the challenging 1-aryl-1,3-dienes, unactivated aliphatic alkenes, and various N- or O-centered nucleophiles. Preliminary mechanistic studies were conducted. The practical utility of this protocol is highlighted by the gram-scale synthesis and late-stage modification of bioactive molecules.

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Introduction

Alkenes are important bulk chemicals which can be produced from the traditional petrochemical industry and emerging coal chemical industry. The difunctionalisation of accessible alkene feedstocks into varied highly value-added fine chemicals with increased molecular complexity is a cutting-edge research hotspot in synthetic chemistry.^{1,2} During the past few years, a wide spectrum of elegant alkene oxidative difunctionalisation reactions,³ which can concurrently introduce two vicinal C–C/C–C, C–hetero/C–hetero, or C–C/C–hetero bonds to multiple bonds, have been well established with the strategies of transition-metal catalysis,^{4–6} visible-light catalysis,⁷ and organocatalysis.⁸ Meanwhile, the asymmetric version of the alkene difunctionalisation reactions as a much more challenging topic has also achieved rapid progress.^{9,10}

Organoselenium compounds are widespread in the areas of the pharmaceutical industry,^{11,12} polymer chemistry,¹³ and advanced organic functional materials¹⁴ due to their abundant biological activities and specific photophysical and photochemistry properties. They are also exploited as small molecule catalysts^{15,16} and polydentate ligands¹⁷ in organic synthesis. The direct selenofunctionalisation of alkene skeletons with the formation of adjacent C–Se and C–X (X = carbon and heteroatoms) bonds has been proved to be a straightforward and powerful protocols for synthesising organic selenium-containing products.^{18–30} However, the use of transition-metal catalysts, excessive chemical oxidants, various reaction additives, and harsh reaction conditions of some of the methods mentioned above usually result in unwanted byproducts and large amounts of waste, thus increasing the difficulties of the workup procedure and bringing the risk of environmental pollution.

In recent years, electrochemical organic synthesis^{31,32} and the merging of electrosynthesis with transition-metal catalysis³³ or visible-light photoredox catalysis^{34,35} have achieved remarkable advances in the functionalisation of olefins. Among them, tandem electrochemical oxidative inter- or intramolecular carbonselenation,^{36–38} aminoselenation,³⁹ oxyselelenation,^{39–50} selenosulfonylation,⁵¹ and fluoroselenation⁵² of alkene derivatives with C–X (X = C, N, O, S, F) and C–Se bonds formation have been successfully reported. Lei's group described an efficient electrooxidative amino- and oxyselelenation of styrenes with benzotriazoles, carboxylic acids, and alcohols as the nucleophiles.³⁹ However, only 35% of the target product is obtained when saccharin is used as the nitrogen source. In contrast, other electrochemical oxyselelenation processes are mainly confined to the intramolecular annula-

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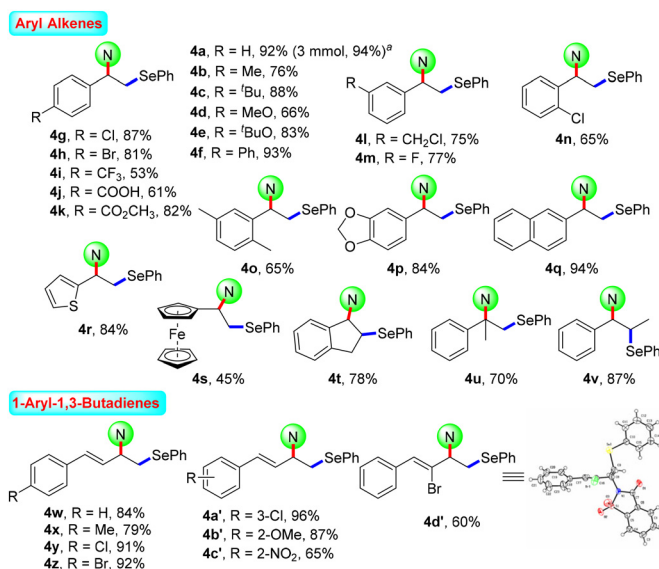
tion reactions of unactivated alkenes, such as olefinic carbonyl, olefinic alcohols, alkenoic acids, allylphenol, β,γ -unsaturated amides, and olefinic amides. Notably, some electrooxidative carbonseleation,^{53,54} oxyseleation,^{55,56} and diselenylation⁵⁷ of alkynes have also been realised.

Compared with the extensively studied difunctionalisation of activated alkenes, the conversion of inactive aliphatic olefins has rarely been researched and the general and green protocol for the difunctionalisation of activated alkenes^{58–63} and challenging 1-aryl-1,3-dienes^{64,65} is far from established. Based on our previous work on the electrooxidative trifunctionalisation of styrenes,⁶⁶ we herein present a universal methodology for the regioselective aminoseleation reaction of alkenes including activated arylalkenes, challenging 1-aryl-1,3-dienes, and non-activated aliphatic alkenes, and excitingly the oxyseleation of inert alkenes was also successful from diversified carboxylic acids (Scheme 1).

Results and discussion

After the systematic screening of reaction parameters (see Tables S1–S3 in the ESI† for details), the reaction conditions for the aminoseleation of activated alkenes were optimised as follows: in an undivided cell with C(+)/Ni(–) electrodes, constant current electrolysis was performed at 10 mA with Et₄NBr as the electrolyte in CH₃CN at room temperature for 2 h. With optimized reaction conditions in hand, the scope for the aminoseleation of activated alkenes **1** was primarily studied (Scheme 2). Generally, under standard conditions, a wide variety of substituted styrenes with varied electron-donating and -withdrawing groups can be conveniently transformed into their corresponding products **4a–4p** in moderate to excellent yields (53–93%). Moreover, 2-vinylnaphthalene, 2-vinylthiophene, and vinyl ferrocene also delivered their respective aminoseleation products **4q–4s** in 94%, 84%, and 45% yields. Even substrates containing steric hindrance, such as 1*H*-indene, α -methylstyrene, and β -methylstyrene, smoothly produce the target products **4t–4v** with good yields.

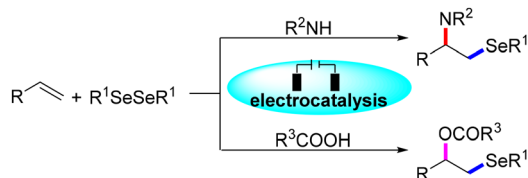
1,3-Dienes often function as versatile feedstocks to assemble structurally diverse, complex molecular architectures by way of transition-metal- or visible-light photoredox-catalyzed alkene-functionalization reactions. In light of 1,3-dienes containing multiple reaction sites, the regioselective alkene functionalisation of 1,3-dienes is a challenging research topic. Delightfully, this methodology was successfully expanded to 1-aryl-1,3-dienes under standard electrolytic conditions with



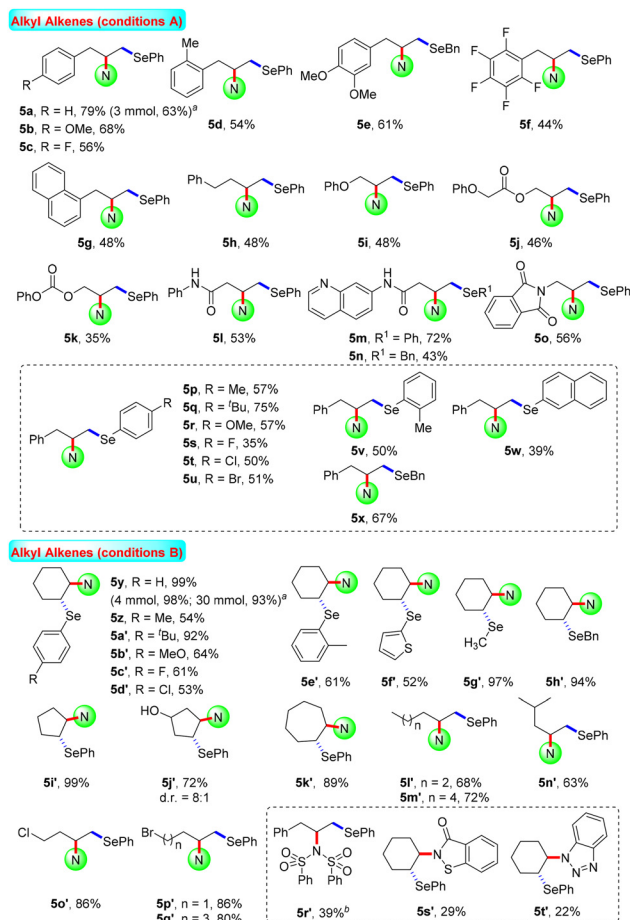
Scheme 2 Substrate scope for the aminoseleation of activated alkenes. Reaction conditions: **1** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), Et₄NBr (0.2 mmol), CH₃CN (4.0 mL), C anode, Ni cathode, 10 mA, 2 h, r.t., undivided cell. Isolated yields. ^a Gram-scale reaction.

exclusive 1,2-regio- and stereoselectivity. The substituent effects of the aromatic ring had little influence on the reaction efficiency. A series of functional groups, including –Me, –MeO, –Cl, –Br, and even strongly deactivated –NO₂, were well tolerated, forming the products **4w–4c'** in good to excellent yields. Notably, the reaction of (*Z*)-(2-bromobuta-1,3-dien-1-yl) benzene with saccharin (**2a**) and diphenyl diselenide (**3a**) proceeded well to result in the target product **4d'** in 60% yield. The molecular structure of **4d'** was determined by single-crystal X-ray diffractometry.

Aliphatic alkenes usually have lower reaction activity and are difficult to activate. The functionalisation of this kind of skeleton is a promising and cutting-edge research field, and has attracted much attention from synthetic chemists all over the world. Various non-activated aliphatic olefins were selected to probe the generality and limitations of this catalytic system. As shown in Scheme 3, a wide range of substituted allylbenzenes were selected as substrates, furnishing the corresponding products **5a–5g** in 44–79% yields. Using homoallylbenzene as the starting material, the desired aminoseleation product **5h** was also generated in moderate yield. In the same fashion, unactivated propylenes with attached useful synthetic functional groups such as phenol oxygen, ester, carbonate, amide, and phthalimide were suitable substrates, producing the difunctionalisation products **5i–5o** in acceptable yields. Moreover, diselenide substrate scope was also investigated using allylbenzene as the olefin source. Under optimal electrolytic conditions, substituted diaryl- and dibenzyl diselenides were subjected to this reaction, showing moderate reaction efficiencies for the generation of the corresponding products **5p–5x**.



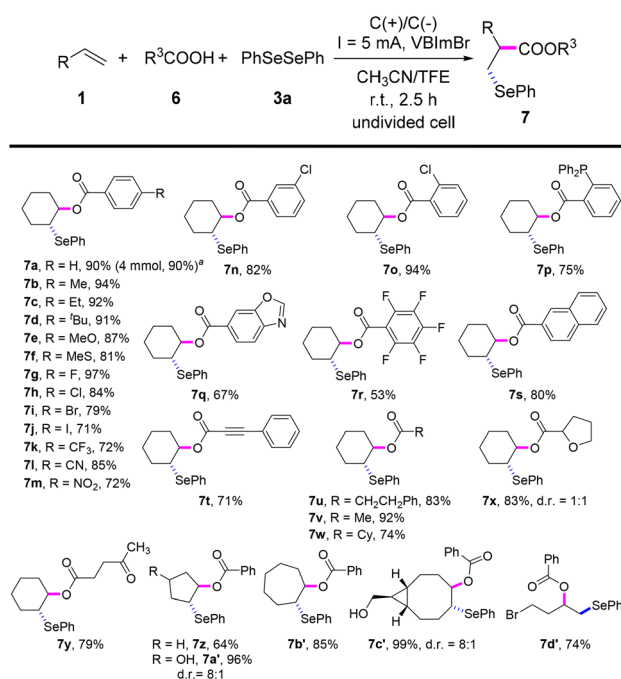
Scheme 1 Electrooxidative amino- and oxyseleation of alkenes.



Scheme 3 Substrate scope for the aminoselenation of unactivated alkenes. Reaction conditions: **1** (0.3 mmol), **2a** (0.2 mmol), **3** (0.15 mmol), r.t., undivided cell. Conditions A: Et₄NBr (0.2 mmol), CH₃CN (4.0 mL), C anode, Ni cathode, 10 mA, 3 h; conditions B: VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v : v = 1 : 3), C anode, C cathode, 5 mA, 2 h. Isolated yields. ^a Gram-scale reaction. ^b Under conditions A. VBImBr = 1-butyl-3-vinylimidazolium bromide. TFE = 2,2,2-trifluoroethanol.

For cyclohexene, a moderate isolated yield (66%) of the target product **5y** was obtained under the current standard conditions A, as shown in Scheme 3. Under modified electrolysis conditions (conditions B in Scheme 3, see Tables S4–S11[†] for details), the desired product **5y** was formed with up to a quantitative yield. Furthermore, a suite of symmetric diselenides containing diaryl, diheteroaryl, and dialkyl diselenides were well involved in this electrochemical reaction system to assemble the expected disubstituted products **5z–5h'** in moderate to excellent yields (52–97%). In addition, 5-/7-membered cyclic and long chain aliphatic alkenes were proved to be good starting materials and led to the desired aminoselenation products **5i'–5q'** in satisfactory yields. Unfortunately, due to the relatively poor nucleophilic ability, other amine sources as nucleophiles only presented lower reaction activities (**5r'–5t'**). The possible reason may be the smaller reduction current of saccharin than other N-centered nucleophiles; this gives it a higher nucleophilicity by smoothly losing the protons (see Fig. S4 in the ESI[†] for details).

Inspired by the good performance of the electrooxidative aminoselenation of various alkenes, we envisioned that this protocol may be qualified to realise the oxyseleation of inert olefins with similar electrolytic processes. Pleasingly, under the slightly altered reaction conditions of Scheme 3, B (see Tables S12–S15[†] for details), the electrochemical oxyseleation of unactivated alkenes proved to be feasible and the desired product **7a** was produced in 90% yield (Scheme 4). Benzoic acids decorated with electron-rich (–Me, –Et, –^tBu, –MeO, –MeS, and –PPh₂) and -deficient groups (–F, –Cl, –Br, –I, –CF₃, –CN, and –NO₂) on the phenyl ring were compatible to give rise to **7b–7p** in 71–97% yields. Other analogues of carboxylic acid, such as condensed, polyhalogenated, and aryl alkynyl carboxylic acid reacted well with saccharin and diphenyl diselenide to generate the corresponding products **7q–7t** in moderate to good yields. Importantly, the treatment of aliphatic carboxylic acids formed the oxyseleation products **7u–7y** in good yields. In the cases of 5-, 7-, 8-membered cyclic alkenes and the linear aliphatic alkenes, the desired products **7z–7d'** were obtained with up to 99% yield. It should be pointed out that these oxyseleation products substituted with plenty of easily modified function groups can be transformed for the further production of synthetically useful organic molecules. The structures of products **5y** and **7m** were finally determined by X-ray single crystal analysis (Fig. 1). Unfortunately, no desired difunctionalization products were formed when aliphatic alcohols such as methanol and ethanol were used as nucleophiles.



Scheme 4 Substrate scope for the oxyseleation of unactivated alkenes. Reaction conditions: **1** (0.2 mmol), **6** (0.4 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v : v = 2 : 2), C anode, C cathode, 5 mA, 2.5 h, r.t., undivided cell. Isolated yields. ^a Gram-scale reaction.

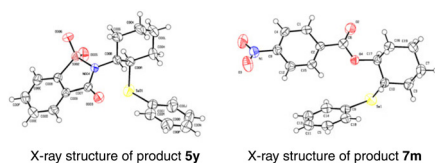
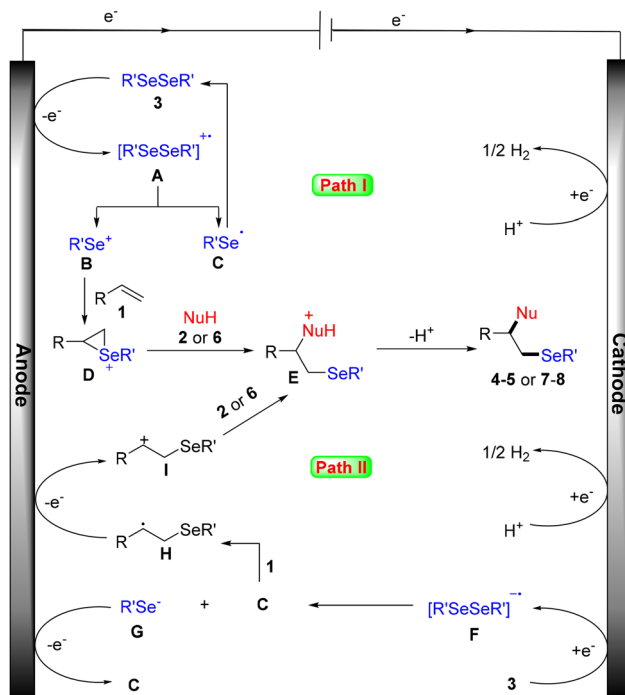


Fig. 1 Crystal structures of compounds **5y** and **7m**.

The practicability of this protocol was confirmed by the late-stage modification of bioactive molecules (Scheme 5). Estrone derivatives were smoothly disubstituted to give selenating products **8a** and **8b** in 75% and 91% yields, respectively. Aryl-, cinnamyl-, and heteroarylcarboxylic acid had moderate to good reaction efficiencies (**8c–8h**). With regard to the aliphatic carboxylic acid, the expected oxyselenation products (**8i–8l**) were obtained in satisfactory yields. Moreover, this electrochemical technology can be easily scaled up to gram scale for activated and unactivated alkenes (**4a**, **5a**, **5y**, and **7a**).

According to our mechanistic probe experiments (see the ESI† for details) and literature reports, a plausible mechanism for this electrochemical amino- and oxyselenation procedure is proposed. As shown in Scheme 6 path I, diselenide **3** is first oxidized at the anode to give the cationic radical intermediate **A**, which is dissociated into selenium cation **B** and selenium radical **C**. Cation **B** then reacts with alkene **1** to form the cyclic selenonium ion intermediate **D**. Finally, the desired product is obtained following the nucleophilic attack of intermediate **D** by the N- or O-based nucleophile, as well as deprotonation. Another possible free radical mechanism cannot be completely excluded (path II). In this process, carbon radical **H** is generated by the addition of selenium radical **C** to the carbon-carbon double bond of alkene **1**. **H** is then converted to its



Scheme 6 Plausible mechanism.

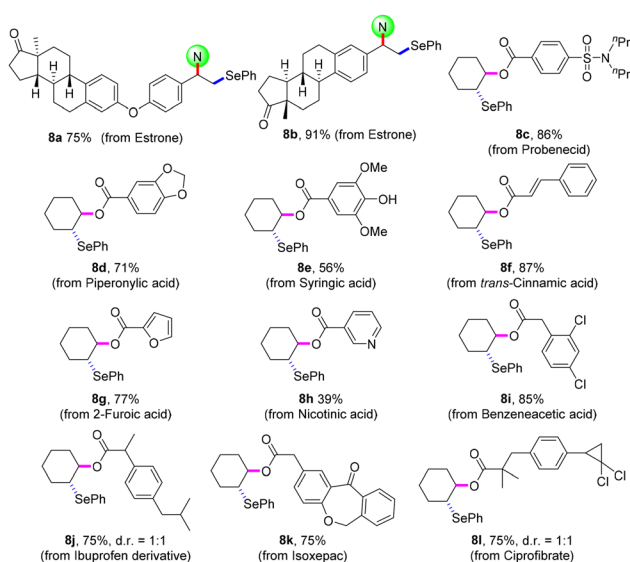
cation **I** by anodic oxidation. Subsequently, the desired product is produced by concessive nucleophilic attack and a deprotonation sequence.

Conclusions

In conclusion, we developed an environmentally friendly synthetic methodology for the sustainable amino- and oxyselenation of varied olefins with clean electric energy. A wide spectrum of readily available alkenes including arylalkenes, 1-aryl-1,3-dienes, and non-activated aliphatic alkenes are involved in this protocol. The successful implementation of scale-up experiments and the late-stage modification of bioactive molecules prove the potential application for producing selenium-containing drugs. Preliminary mechanistic studies suggest that the reaction was induced by both the free radical mechanism and the ionic mechanism. This strategy provides a universal and modular route to organoselenium compounds from easily available starting materials by the electrooxidative difunctionalisation of carbon-carbon double bonds.

Author contributions

Renjie Wang and Nana Zhang carried out the experiments and data analyses and prepared the original manuscript. Yonghong Zhang, Bin Wang, Yu Xia, and Kai Sun provided experimental assistance and constructive suggestions. Weiwei Jin rewrote and revised the manuscript. Weiwei Jin, Xinyong Li, and Chenjiang Liu directed the project and supervised the



Scheme 5 Late-stage modification of bioactive molecules. Reaction conditions: for **8a** and **8b**, conditions in Scheme 1; for **8c–8l**, conditions in Scheme 4. Isolated yields.

whole experiment. All the authors discussed and approved the final manuscript.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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