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An electrochemical method for deborylative seleno/thiocyanation of arylboronic acids under catalyst- and oxidant-free conditions

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An electrochemical deborylative seleno/thiocyanation of arylboronic acids has been well established to synthesize corresponding aryl seleno/thiocyanates with good functional group tolerance under ambient conditions. A gram-scale reaction has been performed to highlight advantage of the protocol. Preliminary mechanistic studies indicated the oxidation of seleno/thiocyanate anion should be prior to arylboronic acid substrate under this galvanostatic mode, and the involvement of free radicals in the process.

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As an indispensable element in life, selenium can participate in metabolism process of human body to improve immunity and enhance physical fitness.1 Furthermore, the incorporation of selenium into organics has dramatic impact on their physical, chemical and biological properties. As a result, organoseleniums are important structural constituents that are present in biologically active reagents and functional materials.² In this regard, many efforts have been devoted into developing powerful methods to create C-Se bond in the past decades.³ Traditionally, organic selenocyanates were prepared by employing triselenium dicyanide,⁴ potassium selenocyanate KSeCN,⁵ selenocyanobenziodoxolone,⁶ and the combination of elemental selenium and TMSCN⁷ as selenocyanide sources in organic synthesis. Although KSeCN is a simple inorganic chemical with the advantage of low cost, however, $K_2S_2O_8$,^{5a-b} selectfluor,^{5c} iodonium reagent,^{5d} TBHP,^{5e} photocatalyst,^{5f} and CAN^{5g} were usually used as unfavorable additives in above reports.

Recently, electrochemistry has emerged as an attractive tool for organic synthesis, since the replacement of stoichiometric amounts of chemical reagents or oxidants in conventional synthesis methods with electrons as mass-free reagent can minimize waste dramatically. As a consequence, organic electrochemical synthesis has been realized as an efficient and environmentally benign strategy to build various molecules.⁸ In this context, electrochemical selenocyanation of electron-rich arenes with KSeCN has been described as a powerful alternative to construct aryl selenocyanates using electricity as the driving force.⁹ Arylboronic acids are stable and broadly available materials, significant progress has been made for electrochemical conversion of arylboronic acids into biaryls,¹⁰ aryl amines and phenols¹¹, very recently, our group has also revealed an efficient method to form aryl halides via ipsohalogenation of arylboronic acids with NaX (X = I, Br) under conditions.12 electrochemical То date, electrocatalytic selenocyanation of arylboronic acids to produce certain organic selenocyanates under metal-free conditions still remain in high demand.

Besides organic selenocyanates, organic thiocyanates are also important structural motifs in a wealth of natural products.13 Additionally, organic thiocyanates can act as versatile synthetic precursors to form valuable sulfur-containing derivatives.14 As a part of our efforts in developing novel functionalization transformations with cheap coupling partners and green catalytic methods,^{12,15} herein, an efficient, practical and scalable seleno/thiocyanation strategy for arylboronic acids has been unraveled in a transition metal-free manner under constant-current electrolytic conditions. Significantly, this electrochemical deborylative seleno/thiocyanation transformation has several advantages over traditional seleno/thiocyanation conversion, 4-7,16 first of all, the overall process is sustainable in which electricity served as the driving force, and what's more, a range of arylboronic acids effectively participated into this simple and metal-free approach under air atmosphere without any unfavorable additive.

Initially, electrochemical deborylative selenocyanation of 4methylphenylboronic acid **9** with KSeCN was chosen as model reaction to probe various reaction parameters in an undivided cell, as shown in Table 1. By employing a two-electrode system with a graphite felt as anode and the other graphite felt as cathode, when the model reaction was performed with KSeCN (4.0 equiv) under constant-current electrolysis at 10 mA in anhydrous CH₃CN/H₂O (10:0.05, 10.05 mL) mixed solvents at room temperature for 5 hours, to our delight, the conversion afforded desired *p*-methylphenyl selenocyanate **9a** in 65% isolated yield (entry 1). In term of solvents, replacing the mixed solvents with anhydrous CH₃CN as a single solvent or changing the ratio of mixed solvents led to diminished

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 o Conditions: **9** (0.2 mmol), KSeCN (4.0 equiv, 0.8 mmol), anhydrous CH₃CN/H₂O (10:0.05, 10.05 mL) in an undivided cell with two graphite felts (each 15 x 15 x 0.4 mm³), air, rt, 10 mA, 5 h. b Yield of isolated product.

outcome, thus, the clue implied a trace of water in mixed solvents had an influence on the transformation (entries 2-3). Furthermore, the transformation gave a decreased yield with the introduction of quaternary ammonium salt as electrolyte, thus, KSeCN played a dual role as both the coupling partner and supporting electrolyte in this reaction system (entries 4-5). In terms of electrode selection, the effect of C(+)/C(-) electrode pairs on the transformation was better than that of Pt plate as anode or cathode under otherwise identical conditions (entries 6-7). Both increase and decrease of reaction time or operating current cut down the reaction yield, thus, the reaction parameters (5 h and 10 mA) were the best for the transformation (entries 8-11). It's noteworthy that raising the reaction temperature or switching the reaction atmosphere to nitrogen did not improve conversion efficiency (entries 12-13). Finally, no reaction took place without the electric current under otherwise equal conditions, thus, the conversion could be precisely controlled by switching the electric current on or off (entry 14).

With the optimized reaction conditions established, we next examined the scope of arylboronic acids substrates to evaluate generality of the methodology (Table 2). Similarly to our recent observation,12 this novel transition metal-free electrochemical deborylative selenocyanation transformation worked well for a series of arylboronic acids (1-23) with various substituents. As previously described,¹² no matter where the methoxy substituent located on phenylboronic acid core, 4-methoxyphenylboronic acid (1) and its isomer 2-methoxyphenylboronic acid (2) well participated in this electro-organic system to afford desired deborylative selenocyanation products, moreover, for the case of 4methoxyphenylboronic acid bearing another methoxy or formyl group, moderate to good yields were furnished for substrates 2,4dimethoxyphenylboronic acid (3) and 3-formvl-4methoxybenzeneboronic acid (4) with long reaction time, indicative of the influence of the electronic effect on transformation. Interestingly, 4-(dimethylamino)benzeneboronic acid (7) exhibited of unprotected better performance than that 4hydroxyphenylboronic acid (5) and 2-aminophenylboronic acid (6).

Although substrates (8-10) with alkyl groups at the ortho and para positions gave moderate to good yields in five Hours' reaction time; however, owing to steric hindrance of two substituents in ortho positions, only 42% yield was detected for electrochemical deborylative selenocyanation of 2,6-dimethylphenylboronic acid (11). By contrast, electro-neutral phenylboronic acid (12) was reluctant to undergo electrochemical deborylative selenocyanation using increased operating current and prolonged reaction time,

whereas trace amounts of products were detected for electrondeficient substrates (13-16), thus, the results further suggested remarkable electronic effect on the reaction efficiency. Notably, naphthylboronic ring-fused acids (17-18)and (dibenzofuranyl)boronic acid (19) were suitable coupling partners to deliver target products. Furthermore, moderate to good yields were observed for heteroarylboronic acids substrates (20-23), (20) significantly, benzo[b]thien-3-ylboronic acid and 3benzofuranboronic acid (22) formed the desired products in higher yield than their isomers (21) and (23), respectively.

Encouraged by the feasibility of electrochemical deborylative selenocyanation transformation, we further explored deborylative thiocyanation of arylboronic acids to create corresponding aryl thiocyanates. To our delight, the desired aryl thiocyanates bearing electronically diverse groups were obtained in moderate to good yields with TMSNCS as a thiocyanation reagent in the presence of KF. During protocol processing, Goossen and co-workers have recently reported an electrochemical method for ipso-thiocyanation of aryl boronic acids and trifluoroborates with NH₄SCN.¹⁷ As outlined in Table 2, electron-rich (hetero)arylboronic acids substrates (1-12 and 17-23) well participated in transformation with TMSNCS/KF to provide aryl thiocyanates under electrochemical conditions. Fortunately, electron-deficient phenylboronic acid derivatives (13-16) bearing bromo, chloro, methyl formate and cyano group were able to undergo electrochemical deborylative thiocyanation, albeit in moderate yields. It's noteworthy that halomoieties in substrates (13-14) were well tolerated in the conversion, which offered the possibility for late-stage functionalization via classical activation of C-halo bond. Finally, one of the desired products (23b) was crystallized and X-ray analysis unequivocally validated the product structure, which was in agreement with the NMR spectrum.



selenocyanate **5a**.

To demonstrate the synthetic application of this methodology, a 20-fold scale-up of deborylative selenocyanation of 4-hydroxyphenylboronic acid **5** with KSeCN was performed using the same electrosynthetic apparatus. As exhibited in Scheme 1, the desired product **5a** was obtained in 84% yield after 23 hours under constant current electrolysis in air atmosphere, which afforded the outcome at a close level with that on a 0.2 mmol scale (see SI for

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Table 2 Substrate scope of arylboronic acids in the deborylative seleno/thiocyanation reactions^a



anhydrous CH₃CN/H₂O (10:0.05, 10.05 mL) in an undivided cell with two graphite felts (each 15 x 15 x 0.4 mm³), air, rt, 10 mA, 80 min. ^{*b*} Arylboronic acid (0.2 mmol), TMSNCS (3.0 equiv, 0.6 mmol), KF (5.0 equiv), anhydrous CH₃CN/H₂O (10:0.03, 10.03 mL) in an undivided cell with two graphite felts (each 15 x 15 x 0.4 mm³), air, rt, 10 mA, 3 h. ^{*c*} 3 h. ^{*d*} 5 h. ^{*e*} anhydrous CH₃CN/H₂O (10:0.01, 10.01 mL), 5 h. ^{*f*} 15 mA, 5 h. ^{*g*} anhydrous CH₃CN/H₂O (10:0.03, 10.03 mL), 3 h. ^{*h*} 80 min.

the details). Therefore, the result declared a great potential of the method on practical synthesis.



Scheme 2 Radical trapping experiments.

In order to understand the reaction mechanism, some experiments were carried out to elucidate transformation process. Firstly, control experiment indicated electricity was essential for the transformation (entry 10, Table, 1), Secondly, electrochemical^{0.1039}/deborvlative the seleno/thiocyanation of 4methoxyphenylboronic acid 1 gave corresponding aryl seleno/thiocyanates in 85% and 83% yields under standard conditions respectively, however, trace of the desired product suggested a possible radical process when 4 equiv of 2,2,6,6tetramethylpiperidine 1-oxyl (TEMPO) was added as radical scavenger into deborylative selenocyanation transformation, (Scheme 2A), and the evidence of DPE-SCN adduct further confirmed the presence of a free radical intermediate during electrolysis when introducing 2.0 equiv of radical capture 1,1diphenylethylene (DPE) into deborylative thiocyanation conversion (Scheme 2B). Thirdly, as shown in Figure 1, cyclic 4voltammetry (CV) of methoxyphenylboronic acid 1 (curve b) exhibited an oxidation peak at 2.37 V (vs. Ag/AgCl), which was higher than that of KSeCN and TMSNCS (curves c-d), thus, the comparison substantiated that KSeCN and TMSNCS could be oxidized more easily than arylboronic acid substrate under this galvanostatic mode. Since free radical intermediates were involved in electrolysis process, therefore, dual oxidation peaks which corresponded to the oxidation of SeCN- to ·SeCN and ·SeCN to SeCN+ respectively were observed in the CV of KSeCN (curve c), whereas a recorded potential of 1.27 V probably referred to the oxidation of SCN⁻ to ·SCN (curve d).

On the basis of above experimental evidence and literature precedents,^{9,17-18} a plausible mechanism was proposed as shown in Scheme 3. In Scheme 3A, a selenocyanate radical intermediate was firstly formed on the anodic electrode by a single-electron-transfer (SET) process, followed by the formation of (SeCN)₂ via

radical coupling and generation of electrophile SeCN⁺ through bond-cleavage of (SeCN)₂. heterolytic Subsequently, arylboronic acid I underwent electrophilic attack by SeCN⁺ to form active adduct II, which ultimately gave rise to desired product aryl selenocyanate with the release of ⁺B(OH)₂. In the case of deborylative thiocyanation transformation, an alternative process of free-radical addition and then rearomatization could be envisioned. In Scheme 3B, since thiocyanate anion can be generated from the conversion between TMSNCS and KF,^[18b] thus, a thiocyanate radical intermediate ·SCN was formed by a SET process. After that, the addition of radical ·SCN to arylboronic acid I and then following

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Figure 1 Cyclic voltammograms of substrates: (a) background (KF 0.25 mol/L in MeCN/H₂O); (b) *p*-methoxyphenylboronic acid **1** (0.05 mmol/L); (c) KSeCN (0.08 mol/L); (d) TMSNCS (0.06 mol/L).



Scheme 3 Proposed mechanism.

oxidation to furnish aryl cation **IV** through intermediate **III** at the anode, re-aromatization of the intermediate **IV** by losing ${}^{+}B(OH)_2$ afforded the final aryl thiocyanate product. At the cathode, electro-reduction of ${}^{+}B(OH)_2$ generated tetrahydroxydiboron detected by LC-MS, concomitant production of H₂ and OH⁻ via degradation process of H₂O in mixed solvents, while the observation of H₂ gas liberation on cathode was confirmed via GC during electrolysis.

Conclusions

In summary, an operationally simple electrochemical method for deborylative seleno/thiocyanation of arylboronic acids has been developed under mild conditions. The feasibility of this concept provides a straightforward and green method to synthesize a collection of aryl seleno/thiocyanates in the absence of transition-metal catalyst and exogenous oxidant. Gram-scale reaction was performed to assess the advantage and practicability of the methodology. Further investigation into mechanism and applications of the approach are underway in our group, and the results will be reported in due course.

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Conflicts of interest

There are no conflicts to declare.

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Various aryl seleno/thiocyanates have been synthesized from electrochemical deborylative seleno/thiocyanation of arylboronic acids under ambient conditions.