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# 烯烃自由基胺硒化: β-氨基硒醚的简易合成

殷一樊[a] 李 晨[b] 孙 凯*,[c] 刘颖杰[b] 王 薪*,[c]
([a]新墨西哥州立大学化学与生物化学系 新墨西哥州 88011)
([b]哈尔滨商业大学药学院 哈尔滨 150076)
([c]烟台大学化学化工学院 山东烟台 264005)

**摘要** 发展了一种高效的烯烃、二芳基二硒醚和烷基胺的分子间胺硒化策略, 在铜盐和高价碘试剂条件下, 以较高产率获得一系列的 β-氨基硒化物. 初步的实验结果表明, 反应涉及活性硒自由基中间体, 并提出了自由基反应机理.
**关键词** 胺硒化; β-氨基硒化物; 活性硒化物; 自由基

# Radical Aminoselenation of Styrenes: Facile Access to β-Amido-selenides

Yin, Yifan[a] Li, Chen[b] Sun, Kai*,[c] Liu, Yingjie[b] Wang, Xin*,[c]
([a] *Department of Chemistry & Biochemistry, New Mexico State University, New Mexico, 88011 USA*)
([b] *School of Pharmacy, Harbin University of Commerce, Harbin 150076*)
([c] *College of Chemistry and Chemical Engineering, Yantai University, Yantai, Shandong 264005*)

**Abstract** An efficient protocol for the intermolecular amidoselenation of alkenes with diphenyl diselenides and alkyl amines has been developed, affording a series of β-amido-selenides in high yields under copper salt and (diacetoxyiodo)benzene (PIDA) conditions. The preliminary experimental results supported the involvement of active selenide radical species, and a radical pathway was therefore proposed for the reaction.
**Keywords** amidoselenation; β-amido-selenide; active selenide; radical

## 1 Introduction

Selenium-containing compounds play diverse roles in medicinal chemistry, biochemistry and organic synthesis.[1] In the past decade, various organoselenium-catalyzed reactions such as oxidation, reduction, rearrangement, cyclization and enantioselective reactions have been established.[2] Organoselenium chemistry has become an important tool in synthetic and medicinal chemistry. Selenides are an important class of organoselenium compounds. They have specific properties and are readily available as substrates, oxidants, or precatalysts, and have garnered considerable attention for use in various reactions.[3] The development of chemical reactions for selectively and efficiently producing valuable selenides, especially asymmetric selenide skeletons,[4] and searching for the medicinal chemistry related aspects,[5] is therefore important and highly interesting.

Alkenes are simple and abundant bulk commodities in organic synthesis, and derivatization of such feedstock materials is thus of great importance. The vicinal difunctionalization of alkenes is a class of significant reactions to rapidly increase molecular complexity with various functional groups. However, at present there are only few reports on seleno-difunctionalization of alkenes, and in fact, is mainly limited to alkoxy-selenylation and hydroxyl-selenylation[6] of these feedstocks. Besides, a few other works, such as amino-selenation[7] and sulfonyl-selenation[8] of alkenes have also been reported. Both nitrogen- and selenium-containing molecules have widespread applications in the fields of organic synthesis and drug discovery. β-Amido-selenides thus are versatile intermediates and the direct incorporation of both amido- and selenium-groups across a double bond is a desirable process. However, we noticed that the amino sources during amino-selenation in previous literatures are limited to sulfon-

* Corresponding authors. E-mail: sunk468@nenu.edu.cn; wangx933@nenu.edu.cn
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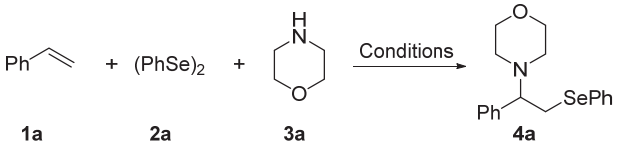
amides or azoles.[7] The amino-selenation with alkyl amine, has not been realized, which may be due to the unstable activity of alkyl amines under oxidation conditions. With the rapidly development of organoselenium chemistry[9] and our ongoing interest in organoselenium involved reactions,[10] herein we report a general method for the high-efficiency synthesis of *β*-amido selenides via amidoselenation of alkenes with alkyl amines and various diselenides. Gram-scale synthesis shows the practicability of this reaction and its potential use in industrial applications.

## 2 Results and discussion

In order to verify the feasibility of this idea, our initial experiment mainly used morpholine as alkyl nitrogen source and diselenides as selenating reagent to perform the amidoselenation of alkenes. At the outset of this study, reaction was performed with styrene **1a** (2.0 equiv.), diphenyl diselenide **2a** (0.2 mmol), morpholine **3a** (2.0 equiv.) using (diacetoxyiodo)benzene (PIDA) (1.0 equiv.) as the oxidant in tetrahydrofuran (THF) (3 mL) at 120 ℃ under air atmosphere. After 12 h, the desired product **4a** was isolated in 21% yield (Table 1, Entry 1). Other solvents, such as $CH_3CN$, dichloroethane (DCE), $CH_3OH$, and *N*,*N*-dimethylformamide (DMF) were also tested, and $CH_3CN$ was the most effective solvent, giving **4a** in 43% yield (Table 1, Entries 2～5). Oxidants $K_2S_2O_8$, [bis(trifluoroacetoxy)iodo]benzene (PIFA) and benzoyl peroxide (BPO) were then investigated, and PIDA was the best choice (Table 1, Entries 6～8). To further improve the reaction efficiency, different additives, including NaBr, KBr, CuBr, $Cu(OTf)_2$, CuCl, CuI and $Cu(OAc)_2$ were further investigated (Table 1, Entries 9～15). Pleasingly, CuBr had a positive effect on the reaction efficiency, and 84% of **4a** could be obtained (Table 1, Entry 11). Further screenings found that a higher reaction temperature of 130 ℃ or lower temperature 100 ℃ did not improve the yield compared with the result at 120 ℃ (Table 1, Entries 16 and 17).

With the optimized reaction conditions in hand, the electronic and steric effects for this reaction with various styrene derivatives were examined (Table 2). Substrates **1** with substituents on benzene ring at different positions, such as *o*-methyl, *o*-chloro, *m*-methyl, *m*-chloro, *p*-methyl and *p*-chloro were all efficiently transformed into the corresponding products (**4b**, **4c**, **4e**, **4f**, **4h** and **4l**) with high yields. Substrates with electron-donating groups (Me, OMe, Ph) or electron-withdrawing groups (F, Cl, Br, $CF_3$) on benzene ring proceeded well in this reaction, affording the desired products **4b**～**4n** in moderate to good yields (60%～88%). It is noteworthy that all valuable electrophilic functional groups, such as fluoro, chloro and bromo, are well tolerated, providing a versatile handle for further functionalization. In addition, 2-vinylnaphthalene (**1o**) was also smoothly converted in this reaction, and the corresponding product **4o** was obtained in 75%. Cyclohexene was also a good partner in this reaction, as expected, the aminoselenation proceeded smoothly and gave the desired product **4p** in moderate yield (60%).

**Table 1** Survey of the reaction conditions[a]




1a 2a 3a 4a

| Entry | Catalyst | Oxidant | Solvent | Yield[b]/% |
|---|---|---|---|---|
| 1 | — | PIDA | THF | 21 |
| 2 | — | PIDA | $CH_3CN$ | 43 |
| 3 | — | PIDA | DCE | Trace |
| 4 | — | PIDA | $CH_3OH$ | 15 |
| 5 | — | PIDA | DMF | 0 |
| 6 | — | $K_2S_2O_8$ | $CH_3CN$ | 13 |
| 7 | — | PIFA | $CH_3CN$ | 32 |
| 8 | — | BPO | $CH_3CN$ | 0 |
| 9 | NaBr | PIDA | $CH_3CN$ | 63 |
| 10 | KBr | PIDA | $CH_3CN$ | 70 |
| 11 | CuBr | PIDA | $CH_3CN$ | 84 |
| 12 | $Cu(OTf)_2$ | PIDA | $CH_3CN$ | 50 |
| 13 | CuCl | PIDA | $CH_3CN$ | 46 |
| 14 | CuI | PIDA | $CH_3CN$ | 63 |
| 15 | $Cu(OAc)_2$ | PIDA | $CH_3CN$ | 30 |
| 16 | CuBr | PIDA | $CH_3CN$ | 81[c] |
| 17 | CuBr | PIDA | $CH_3CN$ | 74[d] |

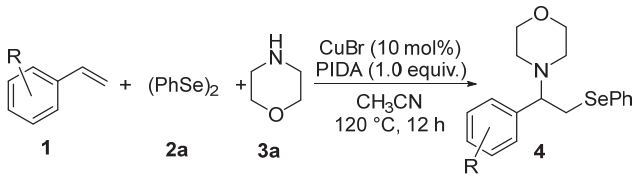
[a] Reactions were performed with **1a** (2.0 equiv.), **2a** (0.2 mmol), **3a** (2.0 equiv.), catalyst (10 mol%) and oxidant (1.0 equiv.) in solvent (3 mL) at 120 ℃ for 12 h. [b] Yield of isolated product. [c] Reaction performed at 130 ℃. [d] Reaction performed at 100 ℃.

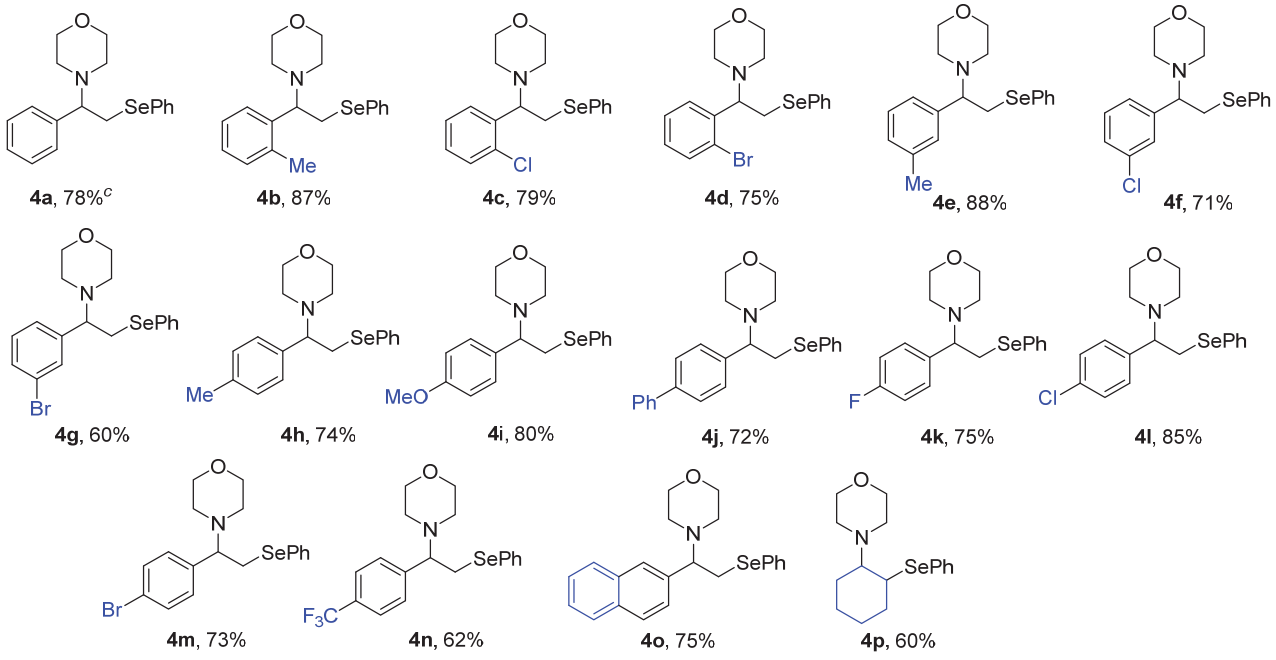
Encouraged by these promising results, we briefly evaluated this amino-selenation strategy using diselenides **2** and alkyl amines **3** (Table 3). Dimethyldiselenide and dibenzyldiselane performed well in this amidoselenation process, with corresponding products **4q** and **4r** in good yields (86% and 82%). Electron-donating group OMe on the aromatic ring was applicable as well, giving the desired product **4s** in good yield (83%). In addition, the diphenyl diselenides bearing electron-withdrawing substituents, such as Br, $CF_3$, and $OCF_3$, underwent vicinal amidoselenation smoothly to provide the corresponding products **4u**～**4v** in moderate to good yields (55%～75%). Besides morpholine, piperazine derivatives proved to be a good partner in this transformation, giving the desired *β*-amido selenides **4w** and **4x** in good yields (70% and 78%). However, unfortunately, piperidine nitrogen source can not participate in the reaction to give the corresponding product **4y**.

To investigate the reaction mechanism, control experiments were performed. When 2,2,6,6-tetramethylpiperidine *N*-oxide (TEMPO, 3.0 equiv.) was added to the reaction as a radical scavenger, the reaction was markedly inhibited (Eq. 1), and the radical-trapping product **5** was de-

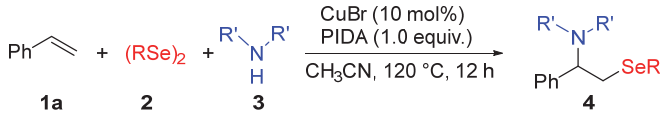
**Table 2** Substrate scope of styrenes[a,b]

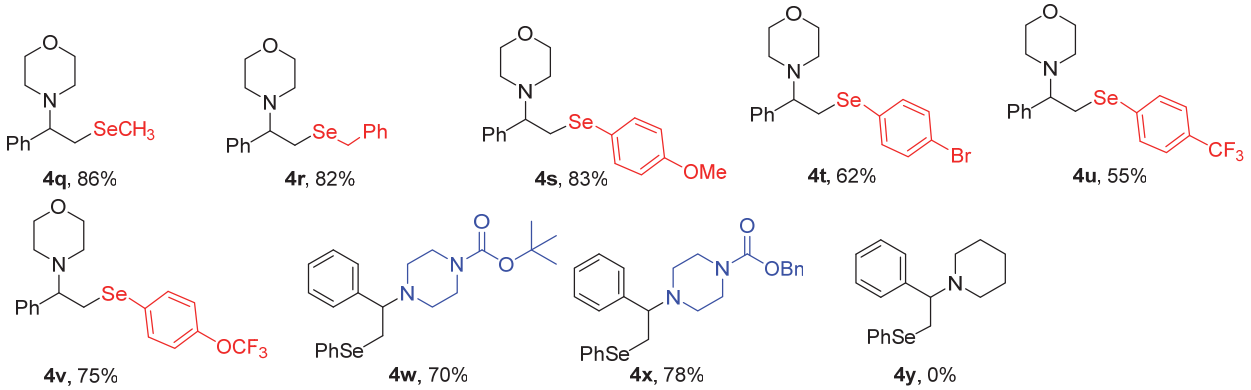


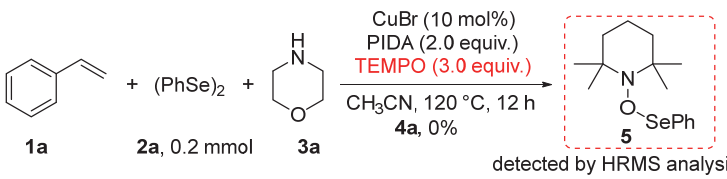

[a] Reaction conditions: **1** (2.0 equiv.), **2a** (0.2 mmol), **3a** (2.0 equiv.), CuBr (10 mol%) and PIDA (1.0 equiv.) in $CH_3CN$ (3 mL) at 120 ℃ for 12 h. [b] Yield of the isolated product. [c] When 10 mmol of **1a** was performed under optimized conditions, 78% of **4a** was isolated.

**Table 3** Substrate scope of diselenides and amine sources[a,b]



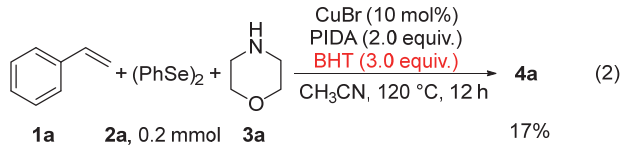

[a] Reaction conditions: **1a** (2.0 equiv.), **2** (0.2 mmol), **3** (2.0 equiv.), CuBr (10 mol%) and PIDA (1.0 equiv.) in $CH_3CN$ (3 mL) at 120 ℃ for 12 h. [b] Yield of the isolated product.



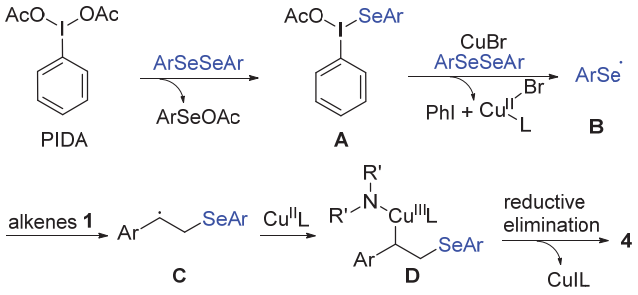

(1)

tected by HRMS. Furthermore, in the presence of 2,6-di-*tert*-butyl-4-methylphenol (BHT, 3.0 equiv.) under the standard reaction conditions, the yield of **4a** decreased to 17% (Eq. 2). These results indicated that a radical pathway was involved under this catalytic system.




On the basis of the experimental results and previously reported studies,[7,11] a possible reaction mechanism was then proposed for this difunctionalization of alkenes. As shown in Scheme 1, initially, the reaction of PIDA with diselenides **2** furnishes the intermediate **A**, with the assistance of CuBr, which undergoes homolytic cleavage to produce a phenyl selenide radical **B**.[12] Subsequently, the addition of **B** to alkenes **1** affords benzyl radical **C**, which intercepts with $Cu^{II}$ species to generate alkyl-$Cu^{III}$-amido species **D**, which then undergoes the facile reductive elimination to give the final products **4** and regenerates $Cu^{I}$, which enters into the next catalytic circle.




**Scheme 1** Plausible mechanism

## 3 Conclusions

In conclusion, a practical method has been successfully developed for the facile synthesis of *β*-amido selenides through the direct difuctionalization of alkenes with diselenides and alkyl amines. The difunctionalization reactions proceeded with good regioselectivity, and no regioisomers were observed in the present reaction system. This protocol features with operation simplicity, good regioselectivity, good tolerance to scale-up synthesis, as well as readily available substrates, catalyst and oxidant of low toxicity. This synthesis procedure would serve as a practical and efficient protocol to synthesize *β*-amido selenide derivatives.

## 4 Experimental section

### 4.1 Instruments and reagents

$^1$H NMR, $^{13}$C NMR, $^{19}$F NMR spectra were recorded on a Bruker Ascend™ 400 or a Bruker Ascend™ 600 spectrometer in deuterated solvents containing TMS as an internal reference standard. All high-resolution mass spectra (HRMS) were measured on a Waters LCT Premier/XE mass spectrometer by using electrospray ionization orthogonal acceleration time-of-flight (ESI-OA-TOF), and the purity of all samples used for HRMS (>95%) was confirmed by $^1$H NMR and $^{13}$C NMR spectroscopic analysis. Melting points were measured on a melting point apparatus equipped with a thermometer and were uncorrected. All the reactions were monitored by thin-layer chromatography (TLC) using $GF_{254}$ silica gel-coated TLC plates. Purification by flash column chromatography was performed over $SiO_2$ (silica gel 200～300 mesh). All reagents were purchased from commercial sources and used without further purification.

### 4.2 General procedure for the synthesis of 4

Taking the synthesis of **4a** as an example, to a reaction tube, styrene **1a** (2.0 equiv., 0.4 mmol), diphenyl diselenide **2a** (0.2 mmol), morpholine **3a** (2.0 equiv., 0.4 mmol), CuBr (10 mol%, 0.02 mmol) and PIDA (1.0 equiv., 0.2 mmol) were mixed in $CH_3CN$ (3 mL). The mixture was heated in an oil bath at 120 ℃ for 12 h and monitored by TLC analysis. The mixture was diluted with water (20 mL) and extracted with ethyl acetate (10 mL×3). The combined organic layers were washed with brine (20 mL), dried over $Na_2SO_4$, and filtered. Then the organic solvent was concentrated *in vacuo*. The residue was purified by flash column chromatography with ethyl acetate and petroleum ether ($V$∶$V$=5∶1) as eluent to give product **4a**.

(1-Phenyl-2-(phenylselanyl)ethyl)morpholine (**4a**): Yellow liquid (58 mg, 84% yield). $R_f$ = 0.3 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.44～7.37 (m, 2H), 7.37～7.16 (m, 8H), 3.75～3.61 (m, 5H), 3.55～3.51 (m, 1H), 3.30～3.25 (m, 1H), 2.45 (s, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 138.56, 132.49, 131.24, 128.99, 128.55, 128.23, 127.81, 126.71, 69.78, 67.13, 50.60, 31.72; HRMS (ESI) calcd for $C_{18}H_{21}NOSe$ $[M+H]^+$ 348.0867, found 348.0861.

4-(2-(Phenylselanyl)-1-(*o*-tolyl)ethyl)morpholine (**4b**): Yellow liquid (62 mg, 87% yield). $R_f$=0.34 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.39～7.37 (m, 3H), 7.19～7.15 (m, 6H), 3.95～3.91 (m, 1H), 3.66～3.64 (m, 4H), 3.43～3.39 (m, 1H), 3.34～3.29 (m, 1H), 2.60～2.52 (m, 2H), 2.46～2.42 (m, 2H), 2.34 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 138.15, 137.01, 132.41, 131.20, 130.62, 129.00, 127.56, 127.23, 126.75, 125.84, 67.21, 64.83, 50.98, 31.12, 19.95. HRMS (ESI) calcd for $C_{19}H_{23}NOSe$ $[M+H]^+$: 362.1018, found: 362.1028.

4-(1-(2-Chlorophenyl)-2-(phenylselanyl)ethyl)morpholine (**4c**): Yellow liquid (60 mg, 79% yield). $R_f$=0.35 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.47～7.45 (m, 1H), 7.42～7.32 (m, 3H), 7.25～7.15 (m, 5H), 4.36～4.32 (m, 1H), 3.67 (t, $J$=4.7 Hz, 4H), 3.43～3.39 (m, 1H), 3.32～3.27 (m, 1H), 2.61～2.53 (m, 2H), 2.49～2.43 (m, 2H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 136.86, 134.77, 132.67, 130.89, 129.78, 129.37, 128.99, 128.61, 126.83, 126.53, 67.11, 64.11, 50.71, 31.26;

HRMS (ESI) calcd for $C_{18}H_{20}ClNOSe$ [M + H]$^+$ 382.0472, found 382.0481.

4-(1-(2-Bromophenyl)-2-(phenylselanyl)ethyl)morpholine (**4d**): Yellow liquid (64 mg, 75% yield). $R_f$=0.36 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.56～7.54 (m, 1H), 7.48～7.46 (m, 1H), 7.38～7.36 (m, 2H), 7.30～7.25 (m, 1H), 7.20～7.17 (m, 3H), 7.13～7.09 (m, 1H), 4.32～4.28 (m, 1H), 3.67 (t, $J$=4.7 Hz, 4H), 3.40～3.36 (m, 1H), 3.33～3.28 (m, 1H), 2.64～2.55 (m, 2H), 2.49～2.43 (m, 2H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 138.69, 133.09, 132.61, 130.91, 129.61, 128.99, 128.95, 127.16, 126.82, 125.53, 67.12, 66.73, 50.78, 31.38; HRMS (ESI) calcd for $C_{18}H_{20}BrNOSe$ [M+H]$^+$ 425.9966, found 425.9971.

4-(2-(Phenylselanyl)-1-(m-tolyl)ethyl)morpholine (**4e**): Yellow liquid (63 mg, 88% yield). $R_f$=0.34 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.43～7.41 (m, 2H), 7.21～7.19 (m, 4H), 7.10～7.04 (m, 3H), 3.68～3.66 (m, 5H), 3.55～3.50 (m, 1H), 3.28～3.23 (m, 1H), 2.45 (t, $J$=4.5 Hz, 4H), 2.34 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 138.42, 137.81, 132.45, 131.35, 129.19, 128.96, 128.55, 128.07, 126.66, 125.66, 69.84, 67.14, 50.62, 31.69, 21.57; HRMS (ESI) calcd for $C_{19}H_{23}NOSe$ [M + H]$^+$ 362.1018, found 362.1030.

4-(1-(3-Chlorophenyl)-2-(phenylselanyl)ethyl)morpholine (**4f**): Yellow liquid (54 mg, 71% yield). $R_f$=0.35 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (600 MHz, $CDCl_3$) $\delta$: 7.41～7.40 (m, 2H), 7.25～7.20 (m, 6H), 7.14～7.13 (m, 1H), 3.68～3.64 (m, 5H), 3.48～3.45 (m, 1H), 3.23～3.20 (m, 1H), 2.43 (d, $J$=4.3 Hz, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 140.97, 134.23, 132.68, 130.79, 129.46, 129.04, 128.51, 127.95, 126.92, 126.76, 69.47, 67.05, 50.63, 31.42; HRMS (ESI) calcd for $C_{18}H_{20}ClNOSe$ [M+H]$^+$ 382.0472, found 382.0475.

4-(1-(3-Bromophenyl)-2-(phenylselanyl)ethyl)morpholine (**4g**): Yellow liquid (51 mg, 60% yield). $R_f$=0.33 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.43～7.40 (m, 4H), 7.22～7.18 (m, 5H), 3.69～7.65 (m, 5H), 3.49～3.44 (m, 1H), 3.24～3.19 (m, 1H), 2.45～2.43 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 141.24, 132.70, 131.41, 130.88, 130.76, 129.76, 129.04, 127.21, 126.94, 122.51, 69.46, 67.04, 50.62, 31.41; HRMS (ESI) calcd for $C_{18}H_{20}BrNOSe$ [M+H]$^+$ 425.9966, found 425.9974.

4-(2-(Phenylselanyl)-1-(*p*-tolyl)ethyl)morpholine (**4h**): Yellow liquid (53 mg, 74% yield). $R_f$=0.35 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.43～7.41 (m, 2H), 7.21～7.19 (m, 3H), 7.13 (s, 4H), 3.67～3.65 (m, 5H), 3.54～3.49 (m, 1H), 3.28～3.23 (m,1H), 2.44 (t, $J$=4.5 Hz, 4H), 2.34 (s, 3H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 137.46, 135.44, 132.44, 131.34, 128.97, 128.93, 128.44, 126.65, 69.44, 67.15, 50.58, 31.77, 21.16; HRMS (ESI) calcd for $C_{19}H_{23}NOSe$ [M+H]$^+$ 362.1018, found 362.1031.

4-(1-(4-Methoxyphenyl)-2-(phenylselanyl)ethyl)morpholine (**4i**): Yellow liquid (60 mg, 80% yield). $R_f$=0.25 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (600 MHz, $CDCl_3$) $\delta$: 7.42～7.41 (m, 2H), 7.20～7.16 (m, 5H), 6.86 (d, $J$=8.6 Hz, 2H), 3.79 (s, 3H), 3.67～3.65 (m, 5H), 3.52～3.48 (m, 1H), 3.27～3.24 (m, 1H), 2.43 (s, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 159.15, 132.45, 131.34, 130.61, 129.58, 128.98, 126.66, 113.56, 69.10, 67.13, 55.26, 50.59, 31.94; HRMS (ESI) calcd for $C_{19}H_{23}NO_2Se$ [M+H]$^+$ 378.0967, found 378.0978.

(1-([1,1'-Biphenyl]-3-yl)-2-(phenylselanyl)ethyl)morpholine (**4j**): Yellow liquid (61 mg, 72% yield). $R_f$=0.45 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (500 MHz, $CDCl_3$) $\delta$: 7.33～7.28 (m, 4H), 7.19～7.14 (m, 4H), 7.09～7.04 (m, 3H), 6.94～6.93 (m, 3H), 3.51～3.39 (m, 5H), 3.30～3.27 (m, 1H), 3.06～3.02 (m, 1H), 2.22 (d, $J$=3.8 Hz, 4H); $^{13}$C NMR (120 MHz, $CDCl_3$) $\delta$: 140.76, 140.64, 137.61, 132.57, 131.23, 129.04, 129.02, 128.83, 127.37, 127.11, 126.94, 126.77, 69.56, 67.16, 50.66, 31.70; HRMS (ESI) calcd for $C_{24}H_{25}NOSe$ [M+ H]$^+$ 424.1174, found 424.1177.

4-(1-(4-Fluorophenyl)-2-(phenylselanyl)ethyl)morpholine (**4k**): Yellow liquid (55 mg, 75% yield). $R_f$=0.3 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (600 MHz, $CDCl_3$) $\delta$: 7.41～7.40 (m, 2H), 7.24～7.20 (m, 5H), 7.05～6.97 (m, 2H), 3.68～3.65 (m, 5H), 3.48～3.46 (m, 1H), 3.26～3.23 (m, 1H), 2.43～2.41 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 162.29 (d, $J$=245.9 Hz), 134.53, 132.59, 130.94, 130.00 (d, $J$=7.9 Hz), 129.02, 126.84, 115.08 (d, $J$=21.5 Hz), 69.09, 67.08, 50.65, 31.88; $^{19}$F NMR (565 MHz, $CDCl_3$) $\delta$: −114.63; HRMS (ESI) calcd for $C_{18}H_{20}FNOSe$ [M+H]$^+$ 366.0767, found 366.0773.

4-(1-(4-Chlorophenyl)-2-(phenylselanyl)ethyl)morpholine (**4l**): Yellow liquid (65 mg, 85% yield). $R_f$=0.35 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.37～7.35 (m, 2H), 7.27～7.24 (m, 2H), 7.19～7.15 (m, 5H), 3.69～3.58 (m, 5H), 3.44～3.40 (m, 1H), 3.22～3.18 (m, 1H), 2.47～2.31 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 137.38, 133.49, 132.65, 130.80, 129.82, 129.03, 128.40, 126.89, 69.21, 67.05, 50.69, 31.65; HRMS (ESI) calcd for $C_{18}H_{20}ClNOSe$ [M + H]$^+$ 382.0472, found 382.0476.

**4-**(1-(4-Bromophenyl)-2-(phenylselanyl)ethyl)morpholine (**4m**): Yellow liquid (62 mg, 73% yield). $R_f$=0.3 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.48～7.37 (m, 4H), 7.24～7.18 (m, 3H), 7.14 (d, $J$=8.4 Hz, 2H), 3.71～3.57 (m, 5H), 3.47～3.42 (m, 1H), 3.25～3.20 (m, 1H), 2.50～2.37 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 137.92, 132.66, 131.35, 130.76, 130.19, 129.04, 126.90, 121.64, 69.29, 67.04, 50.70, 31.59; HRMS (ESI) calcd for $C_{18}H_{20}BrNOSe$ [M+H]$^+$ 425.9966, found 425.9970.

4-(2-(Phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethyl)morpholine (**4n**): Yellow liquid (51 mg, 62% yield). $R_f$=0.29 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (600 MHz, $CDCl_3$) $\delta$: 7.56 (d, $J$=8.0 Hz, 2H), 7.39～7.36 (m, 4H), 7.21～7.20 (m, 3H), 3.70～3.67 (m, 5H), 3.48～3.45 (m, 1H), 3.28～3.24 (m, 1H), 2.46～2.40 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 143.12, 132.73,

130.55, 129.05, 128.87, 127.00, 125.15 (q, $J$=3.7 Hz), 122.76, 69.60, 67.02, 50.78, 31.42; $^{19}$F NMR (376 MHz, $CDCl_3$) $\delta$: −62.47; HRMS (ESI) calcd for $C_{19}H_{20}F_3NOSe$ $[M+H]^+$ 416.0735, found 416.0746.

4-(1-(Naphthalen-2-yl)-2-(phenylselanyl)ethyl)morpholine (**4o**): Yellow liquid (59 mg, 75% yield). $R_f$=0.26 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (600 MHz, $CDCl_3$) $\delta$: 7.82 (t, $J$=8.6 Hz, 3H), 7.69 (s, 1H), 7.49～7.40 (m, 5H), 7.21～7.16 (m, 3H), 3.86～3.80 (m, 1H), 3.69 (t, $J$=4.6 Hz, 4H), 3.61～3.58 (m, 1H), 3.39～3.36 (m, 1H), 2.56～2.44 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 136.52, 133.10, 133.08, 132.56, 131.12, 128.98, 127.99, 127.96, 127.67, 126.75, 126.22, 126.15, 125.96, 70.06, 67.14, 50.94, 31.73; HRMS (ESI) calcd for $C_{22}H_{23}NOSe$ $[M+H]^+$ 398.1018, found 398.1026.

4-(2-(Phenylselanyl)cyclohexyl)morpholine (**4p**): Yellow liquid (33 mg, 50% yield). $R_f$ = 0.35 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (500 MHz, $CDCl_3$) $\delta$: 7.47～7.45 (m, 2H), 7.21～7.13 (m, 3H), 3.70～3.56 (m, 4H), 3.38～3.32 (m, 1H), 2.71～2.67 (m, 2H), 2.47～2.32 (m, 3H), 1.92～1.81 (m, 1H), 1.78～1.65 (m, 2H), 1.60～1.52 (m, 1H), 1.34～1.26 (m, 1H), 1.18～1.13 (m, 2H), 1.06～0.96 (m, 1H); $^{13}$C NMR (126 MHz, $CDCl_3$) δ: 136.02, 129.72, 128.64, 127.21, 69.14, 67.37, 48.56, 45.12, 34.12, 27.29, 25.56, 24.99; HRMS (ESI) calcd for $C_{16}H_{23}NOSe$ $[M+H]^+$ 326.1018, found 326.1014.

4-(2-(Methylselanyl)-1-phenylethyl)morpholine (**4q**): Yellow liquid (49 mg, 86% yield). $R_f$=0.25 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.36～7.25 (m, 5H), 3.68 (t, $J$=4.7 Hz, 4H), 3.58～3.54 (m, 1H), 3.15～3.10 (m, 1H), 2.92～2.87 (m, 1H), 2.52～2.40 (m, 4H), 1.84～1.79 (m, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$: 139.28, 128.54, 128.19, 127.68, 70.57, 67.16, 50.85, 29.34, 4.92; HRMS (ESI) calcd for $C_{13}H_{19}NOSe$ $[M+H]^+$ 286.0705, found 286.0724.

4-(2-(Benzylselanyl)-1-phenylethyl)morpholine (**4r**): Yellow liquid (59 mg, 82% yield). $R_f$=0.23 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.33～7.19 (m, 10H), 3.67～3.54 (m, 6H), 3.47～3.44 (m, 1H), 3.08～3.04 (m, 1H), 2.85～2.80 (m, 1H), 2.42～2.31 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 139.45, 139.30, 128.85, 128.58, 128.45, 128.17, 127.68, 126.64, 70.35, 67.13, 50.78, 27.79, 27.56; HRMS (ESI) calcd for $C_{19}H_{23}NOSe$ $[M+H]^+$ 362.1018, found 362.1035.

4-(2-((4-Methoxyphenyl)selanyl)-1-phenylethyl)morpholine (**4s**): White soild (62 mg, 83% yield). $R_f$=0.22 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); mp 79～80 ℃; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.40～7.22 (m, 7H), 6.78～6.75 (m, 2H), 3.77 (s, 3H), 3.68～3.63 (m, 5H), 3.45～3.41 (m, 1H), 3.21～3.17 (m, 1H), 2.46～2.39 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 159.18, 138.67, 135.44, 128.59, 128.19, 127.73, 120.82, 114.71, 69.90, 67.14, 55.29, 50.61, 32.77; HRMS (ESI) calcd for $C_{19}H_{23}NO_2Se$ $[M+H]^+$ 378.0967, found 378.0973.

4-(2-((4-Bromophenyl)selanyl)-1-phenylethyl)morpholine (**4t**): Yellow liquid (53 mg, 62% yield). $R_f$=0.2 (Petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.36～7.21 (m, 9H), 3.71～3.63 (m, 5H), 3.54～3.47 (m, 1H), 3.29～3.21 (m, 1H), 2.50～2.39 (m, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 138.24, 134.07, 132.00, 130.17, 128.53, 128.28, 127.91, 120.85, 69.66, 67.09, 50.56, 32.04; HRMS (ESI) calcd for $C_{18}H_{20}BrNO_2Se$ $[M+H]^+$ 425.9966, found 378. 425.9959.

4-(1-Phenyl-2-((4-(trifluoromethyl)phenyl)selanyl)ethyl)morpholine (**4u**): Yellow liquid (46 mg, 55% yield). $R_f$=0.18 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (500 MHz, $CDCl_3$) $\delta$: 7.47～7.42 (m, 4H), 7.36～7.25 (m, 5H), 3.75～3.70 (m, 5H), 3.62～3.58 (m, 1H), 3.36～3.32 (m, 1H), 2.48 (s, 4H); $^{13}$C NMR (120 MHz, $CDCl_3$) $\delta$: 136.82, 135.92, 130.37, 129.67, 127.49, 127.32, 127.04, 124.57 (q, $J$=7.9 Hz), 124.19, 68.52, 65.97, 49.49, 30.30; $^{19}$F NMR (471 MHz, $CDCl_3$) $\delta$: −62.60; HRMS (ESI) calcd for $C_{19}H_{21}F_3NOSe$ $[M+H]^+$ 416.0735, found 416.0732.

Phenyl-2-((4-(trifluoromethoxy)phenyl)selanyl)ethyl)morpholine (**4v**): Yellow liquid (65 mg, 75% yield). $R_f$=0.5 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (500 MHz, $CDCl_3$) $\delta$: 7.25～7.23 (m, 2H), 7.18～7.05 (m, 5H), 6.89 (d, $J$=8.0 Hz, 2H), 3.56～3.51 (m, 5H), 3.38～3.35 (m, 1H), 3.13～3.09 (m, 1H), 2.36～2.24 (m, 4H); $^{13}$C NMR (120 MHz, $CDCl_3$) $\delta$: 148.18, 138.21, 133.97, 129.62, 128.53, 128.26, 127.91, 121.55, 120.43 (q, $J$=257.2 Hz), 69.77, 67.09, 50.57, 32.30; $^{19}$F NMR (471 MHz, $CDCl_3$) $\delta$: −57.93; HRMS (ESI) calcd for $C_{19}H_{21}F_3NO_2Se$ $[M+H]^+$ 432.0684, found 432.0691.

*tert*-Butyl 4-(1-phenyl-2-(phenylselanyl)ethyl)piperazine-1-carboxylate (**4w**): Yellow liquid (62 mg, 70% yield). $R_f$=0.21 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.44～7.42 (m, 2H), 7.33～7.20 (m, 8H), 3.79 (t, $J$=7.4 Hz, 1H), 3.59～3.55 (m, 1H), 3.41～3.37 (m, 4H), 3.29～3.24 (m, 1H), 2.41～2.36 (m, 4H), 1.41 (s, 9H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 154.64, 138.12, 132.52, 131.25, 128.99, 128.49, 128.22, 127.81, 126.72, 80.06, 79.52, 69.36, 49.62, 31.58, 28.41;. HRMS (ESI) calcd for $C_{23}H_{30}N_2O_2Se$ $[M+H]^+$ 447.1546, found 447.1569.

Tenzyl 4-(1-phenyl-2-(phenylselanyl)ethyl)piperazine-1-carboxylate (**4x**): Yellow liquid (75 mg, 78% yield). $R_f$=0.26 (petroleum ether/ethyl acetate, $V$∶$V$=5∶1); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 7.46～7.38 (m, 2H), 7.36～7.27 (m, 8H), 7.24～7.17 (m, 5H), 5.07 (s, 2H), 3.79 (t, $J$=7.5 Hz, 1H), 3.59～3.42 (m, 5H), 3.28～3.21 (m, 1H), 2.40 (d, $J$=14.5 Hz, 4H); $^{13}$C NMR (150 MHz, $CDCl_3$) $\delta$: 155.11, 137.93, 136.77, 132.57, 131.23, 129.04, 128.50, 128.48, 128.28, 128.00, 127.90, 127.87, 126.78, 69.35, 67.06, 49.52, 43.98, 31.48; HRMS (ESI) calcd for $C_{26}H_{28}N_2O_2Se$ $[M+H]^+$ 481.1389, found 481.1394.

### 4.3 Procedure for the gram-scale synthesis of 4a

In the reaction tube, styrene **1a** (2.0 equiv., 10 mmol), diphenyl diselenide **2a** (5 mmol), morpholine **3a** (2.0

equiv., 10 mmol), CuBr (10 mol%, 0.5 mmol) and PIDA (1.0 equiv., 5 mmol) were mixed in $CH_3CN$ (10 mL). The mixture was heated in an oil bath at 120 ℃ for 20 h and monitored by TLC analysis. The mixture was diluted with water (150 mL) and extracted with ethyl acetate (50 mL×3). The combined organic layers were washed with brine (50 mL), dried over $Na_2SO_4$, and filtered. Then the organic solvent was concentrated *in vacuo*. The residue was purified by flash column chromatography with ethyl acetate and petroleum ether as eluent to product **4a** in 78% yield (1.35 g).

**Supporting Information** $^1$H NMR and $^{13}$C NMR spectra for compounds. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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