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Synthesis of Seleno Oxindoles *via* Electrochemical Cyclization of *N*-arylacrylamides with Diorganyl Diselenides

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Abstract: The tandem cyclization of acrylamide with diselenides facilitated by electrochemical oxidation was successfully developed. This strategy provided an environmentally friendly method for the construction of C–Se bond. A series of seleno oxindoles with pharmacological activity were obtained by using this well-designed tandem cyclization strategy. The *in vitro* antitumor activity of the compounds was also screened through MTT assay. Results showed that the seleno oxindoles exhibited better antitumor activity than other oxindole derivatives.

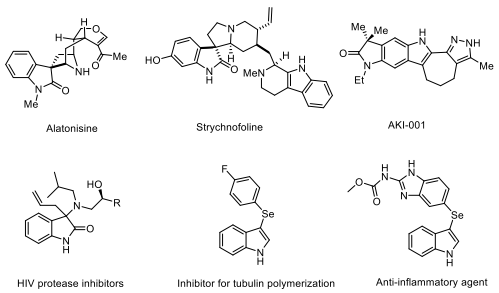
Keywords: Electrochemical synthesis; Cross-coupling reaction; Oxindole skeletons.

Oxindoles are preferential heterocyclic structural motifs that are widely present in numerous natural products and pharmaceutical agents,[1] such as isatin,[2] alatonisine,[3] strychnofoline,[4] AKI-001,[5] HIV protease inhibitors,[6] and AG-041R[7] (Figure 1). As a result of the excellent properties of this framework, the functionalization and synthesis of the oxindole scaffold have attracted considerable interest from synthetic organic chemists.[8] Two strategies, namely, the functionalization of a pre-existing oxindole skeleton by introducing new functional groups[9] and the assembly of a new oxindole ring from acyclic precursors, are commonly used.[10-14] Generally, the latter approach has much subatantial potential for the rapid acquisition of diversity in the functionalized oxindoles. Many functional groups, such as sulfonyl,[10] trifluoromethyl,[11] phosphoryl,[12] substituted alkyl,[13] and aryl,[14] have been successfully incorporated into the oxindole framework through this route.

Selenium is an indispensable trace element in the human body.[15] Selenium compounds that containing heterocyclic units also have unique biological activities, such as antitumor, antibacterial, anti-inflammatory, antiviral, cardiovascular protection, and immune regulation, and other biological activities (Figure 1).[16] Therefore, introducing a selenium functional group into oxindoles presents an attractive research direction; however, only a few chemists have undertaken such an activity.[17]Various selenium reagents, such as elemental selenium, selenium dioxide, selenium sulfonate, potassium selenocyanate, and *N*-(phenylseleno)phthalimide, have been used for the construction of organic selenium compounds.[18] Nevertheless, most selenization reactions usually suffer from harsh or complex reaction conditions. As a result, diselenide, a stable and readily available selenium reagent, has emerged as an alternative for constructing selenium compounds.

In 2014, Fu and coworkers described the ammonium-persulfate-mediated radical oxidative cyclization of *N*-arylacrylamides with *N*-(phenylseleno)phthalimide and obtained several seleno oxindoles selectively.[17] However, this method requires the use of excess oxidants and complex selenium reagents.

Figure 1 Representative oxindole structures



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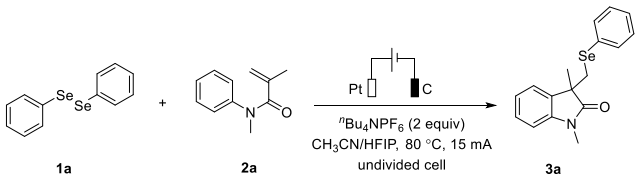
In recent years, organic electrochemical synthesis has been widely considered as a sustainable, benign, and eco-friendly synthetic tool that can realize the oxidation reaction through electron transfer and can be used to replace traditional oxidizing additives. This strategy has also been applied by Lei to achieve the electrochemical synthesis of seleno dihydrofurans and oxazolines.[19] Inspired by these studies and our previous research on electrosynthesis,[20-24] we conducted an electrochemical cyclization reaction between *N*-arylacrylamides and diselenides to obtain seleno oxindole products. These products belong to a new class of compounds with better anticancer activity than compared with traditional oxindole derivatives and organic selenides.

We began our investigation with diphenyl diselenide **1a** and *N*-arylacrylamide **2a** as substrates. After considerable effort, an optimal yield of product **3a** was obtained in 85% under constant current (15 mA) for 2 h in the presence of $^{n}Bu_4NPF_6$ and using CH_3CN/1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) = 8:2 as the cosolvent (Table 1, entry 1).

Table 1 Optimization of the reaction conditions[a]



1a 2a 3a

Entry	Variation from standard conditions	Yield [b]
1	None	85%
2	**1a** (0.6 eq.) was used	68%
3	CH_3CN	60%
4	$^{n}Bu_4NBF_4$ instead of $^{n}Bu_4NPF_6$	67%
5	$^{n}Bu_4NClO_4$ instead of $^{n}Bu_4NPF_6$	46%
6	C−C	62%
7	Pt−Pt	57%
8	C(+)−Pt(−)	77%
9	10 mA instead of 15 mA	59%
10	20 mA instead of 15 mA	60%
11	60 °C instead of 80 °C	64%
12	100 °C instead of 80 °C	57%
13	No electricity	0

[a]Reaction conditions: undivided cell, graphite rod cathode (*Φ* 6 mm), Pt plate anode (1 cm × 1 cm), constant current = 15 mA, **1a** (0.2 mmol), **2a** (0.2 mmol), $^{n}Bu_4NPF_6$ (2.0 equiv), CH_3CN/HFIP (10 mL, v/v = 4:1), 80 °C, 2 h. [b]Isolated yields.

When we attempted to reduce the amount of **1a** to 0.6 eq., **3a** was only afforded in 68% yield (entry 2). The research has indicated that HFIP can be used to stabilize radicals.[25] We observed that the yield was slightly diminished to 60% when CH_3CN was used as a solvent (Table 1, entry 3). Then, a series of supporting electrolytes, such as $^{n}Bu_4NBF_4$ and $^{n}Bu_4NClO_4$, was screened (Table 1, entries 4 and 5). Platinum plates or graphite rods were applied to both electrodes to test the electrode effect (Table 1, entries 6–8). We also changed the anode and cathode, but none of the tested materials could increase the yield of **3a**. Changing the operating current (10 or 20 mA) was proven to be minimally efficient (Table 1, entries 9 and 10). When the temperature was changed to 60 °C or 100 °C, **3a** was observed in 64% or 57% yield, respectively (Table 1, entries 11 and 12). Control experiments showed that no target product was produced in the absence of electricity (Table 1, entry 13).

With the optimized reaction conditions, we subjected the alkyl/aryl diselenides **1a–1h** and *N*-arylacrylamide **2a** to electrochemical cyclization to obtain the corresponding products **3a–3h** in Scheme 1. The diaryldiselenides with electron-donating (−Me) and electron-withdrawing (−Cl and −Br) substituents were reacted with *N*-arylacrylamide to provide the desired products **3b–3d** in 74%–81% yields. In addition, 1,2- di(naphthalen- 2- yl)diselane and 1,2-di([1,1′-biphenyl]-4-yl)diselane provided the corresponding products **3e–3f** in 77%–80% yields. Dimethyl diselenide could also provide the target product **3g** with a yield of 39%. Nevertheless, when 1,2-di(thiophen-2-yl)diselane was used as the substrate, the corresponding adduct **3h** was not obtained. A scaled-up reaction was conducted to verify the practical application of electrochemical cyclization. When 6 mmol **2a** was used to react with **1a**, adduct **3a** was isolated in 80% yield.

We extended the substrate range of *N*-arylacrylamide under standard conditions. As shown in Scheme 2, the electron-donating and electron-withdrawing groups on the *para*-position of the two different aromatic rings of *N*-arylacrylamide all worked well with diselenides, providing the corresponding selenium-containing oxindole products in considerable yields (**3i–3l, 3m–3p**). The two isomers $\mathbf{3k_1}$ and $\mathbf{3k_2}$ were generated when 3-methyl-substituted substrate **2d** reacted with **1a**. This result might be caused by the regioselectivity of the cyclization. The reaction of the *N*-substituted acrylamide with diphenyl diselenide could also proceed smoothly. For example, when phenyl- and benzyl-substituted acrylamides were subjected to electrolysis under the standard conditions, the

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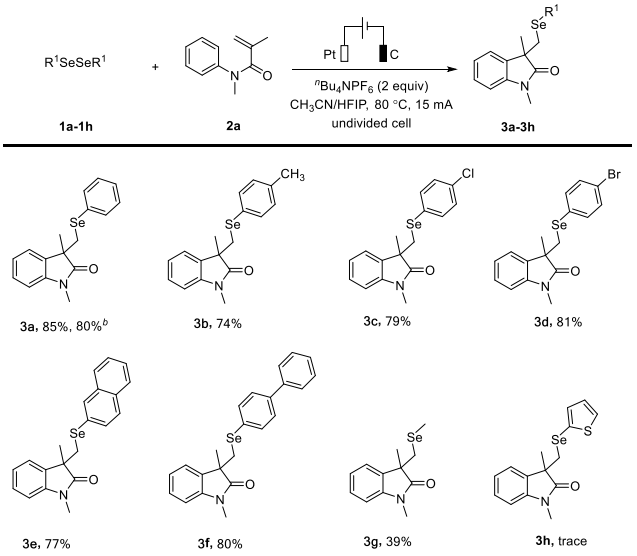
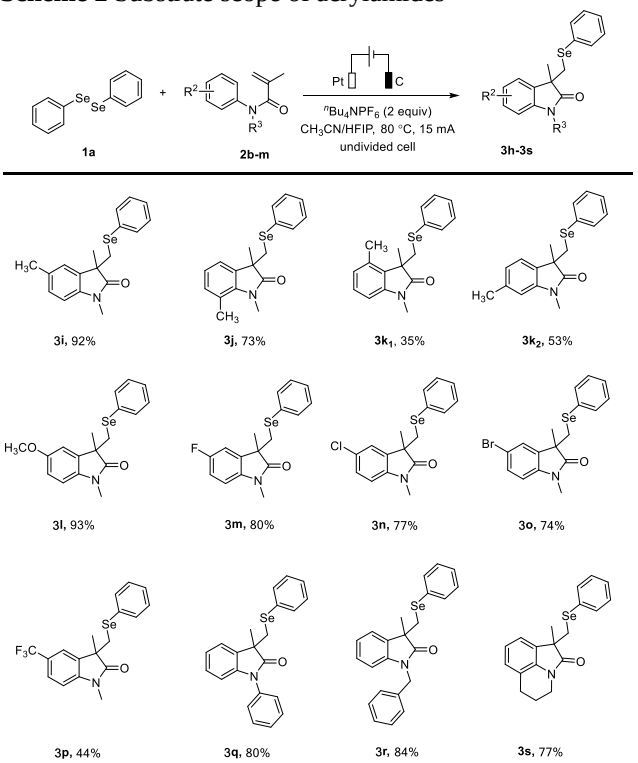
corresponding adducts **3q** and **3r** were generated in 80% and 84% yields, respectively. The tricyclic oxindole **3s** was obtained in 77% yield.

Scheme 1 Substrate scope of diselenides[a]



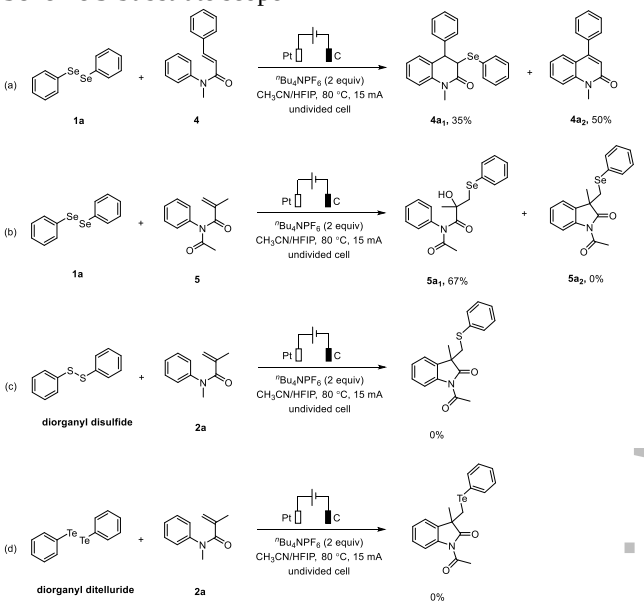
[a] Reaction conditions: undivided cell, graphite rod cathode (*Φ* 6 mm), Pt plate anode (1 cm × 1 cm), constant current = 15 mA, **1a** (0.2 mmol), **2a** (0.2 mmol), $^{n}Bu_4NPF_6$ (2.0 equiv), CH_3CN/HFIP (10 mL, v/v = 4:1), 80 °C, 2 h. [b]undivided cell, constant current = 100 mA, **1a** (6 mmol), **2a** (6 mmol), $^{n}Bu_4NPF_6$ (3 mmol), CH_3CN/HFIP (70 mL, v/v = 4:1), 80 °C, 10 h.

Scheme 2 Substrate scope of acrylamides[a]



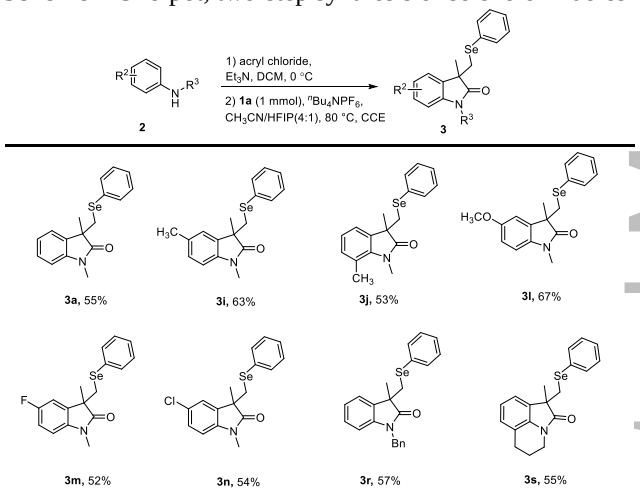
[a]Reaction conditions: undivided cell, graphite rod cathode (*Φ* 6 mm), Pt plate anode (1 cm × 1 cm), constant current = 15 mA, **1a** (0.2 mmol), **2a** (0.2 mmol), $^{n}Bu_4NPF_6$ (2.0 equiv), CH_3CN/HFIP (10 mL, v/v = 4:1), 80 °C, 2 h.

Scheme 3 Substrate scope[a]



[a] Reaction conditions: undivided cell, graphite rod cathode (*Φ* 6 mm), Pt plate anode (1 cm × 1 cm), constant current = 15 mA, **1a** (0.2 mmol), **2a** (0.2 mmol), $^{n}Bu_4NPF_6$ (2.0 equiv), CH_3CN/HFIP (10 mL, v/v = 4:1), 80 °C, 2 h

Scheme 4 One-pot, two-step synthesis of seleno oxindoles[a]



[a] Reaction conditions: (1) aniline (1 mmol), methylacryloyl chloride (1.2 mmol), and triethylamine (1.2 mmol) in dry DCM (10 mL) at 0 °C for 6 h; (2) **1a** (1 mmol) $^{n}Bu_4NPF_6$ (2.0 equiv), CH_3CN/HFIP (10 mL, v/v = 4:1), 80 °C, 3 h, yield of isolated products.

The electrochemical selenization cyclization was not restricted to *N*-arylacrylamides. *N*-arylcinnamamide **4** was successfully converted into 1-methyl-4-phenyl-3-(phenylselanyl)-3,4-dihydroquinolin-2(1H)-one **4a$_1$**, and 1-methyl-4-phenylquinolin-2(1H)-one **4a$_2$** was obtained with a yield of 50% (Scheme 3, a). *N*-acetyl-protected **5a$_2$** was not detected, whereas seleno hydrolysate **5a$_1$** was obtained with a yield of 67% (Scheme 3, b). However, the target products were not detected when we used diorganyl ditelluride or diorganyl disulfide as substrates.

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Anilines are more stable than acrylamides and are commercially available. These advantages contribute to their wildly use in organic synthesis. Therefore, a one-pot, two-step pattern of reaction was conducted. The amine in methylene chloride was treated with acryloyl chloride to obtain the expected acrylamide. After the solvent was removed, the reaction residue was subjected to constant current electrolysis in the presence of **1a** to obtain the corresponding adducts (Scheme 4). The one-pot, two-step reaction, which avoided the isolation and purification of intermediate acrylamides, was proven to be a practical protocol.

Several control experiments were performed to gain further understanding of the mechanism of the method. As shown in Scheme 5, **3a** was not detected when electrolysis was performed in the presence of 2.0 equiv. of TEMPO or BHT (Scheme 5, a). We also conducted a controlled experiment in a divided cell, and product **3a** was detected in the anode oxidation with a yield of 75% (Scheme 5, b), indicating that the reaction could be completed directly in anode oxidation. Further investigation revealed that **2a** could produce the desired product **3a** in the presence of PhSeCl (Scheme 5, c). Therefore, the phenyl selenium cation pathway possibly existed. The detailed experimental results are described in the **Supporting Information** (Scheme S1). In addition, two substrates were subjected to cyclic voltammetry experiments (Figure 2). An evident oxidation peak of substrate **2a** could be observed at 1.67 V. Diphenyl diselenide **1a** presented an oxidation peak at 1.44 V. Additionally, 82% yield was obtained when the reaction was performed under N_2 conditions (Scheme 5, d). Therefore, this process was confirmed to be an electro-oxidation process.

On the basis of the abovementioned results and reported literature,[26] a possible mechanism for the electrochemical cyclization is presented in Scheme 5. The reaction could be initiated by the formation of seleno radical **A** and selenium cation **B** *via* anodic oxidation. Thereafter, the radical addition of **A** to **2a** provided the radical intermediate **C**, and the intramolecular cyclization of intermediate **C** resulted in the radical intermediate **D**. Further anodic oxidation and deprotonation led to the formation of product **3a** (**path I**). We could not rule out the ionic pathway. The addition of **B** to the double bond of **2a** could generate intermediate **F**, for which further oxidative cyclization and deprotonation led to the formation of product **3a** (**path II**).

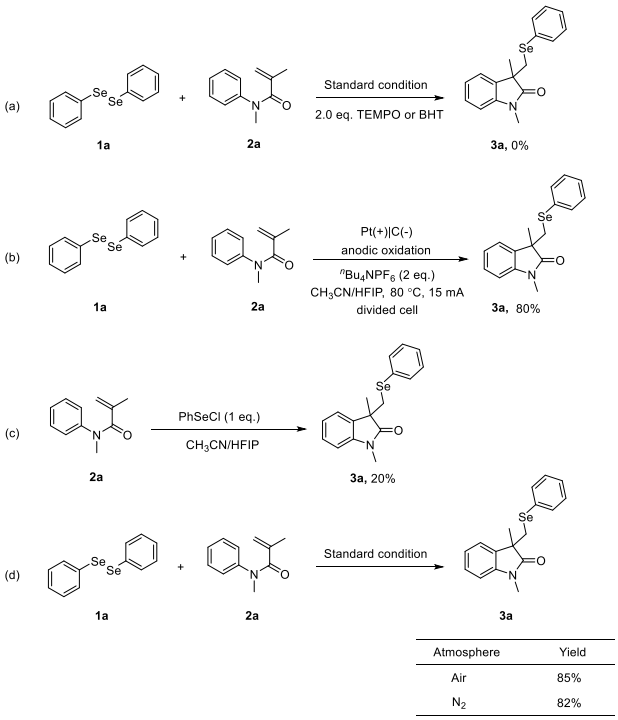
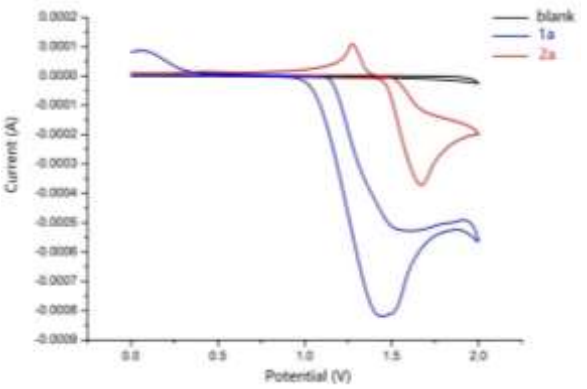
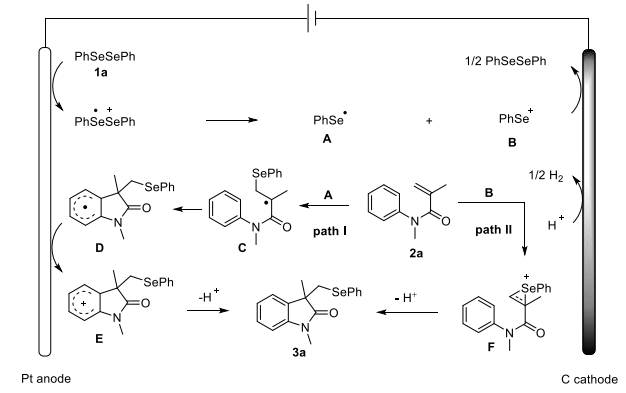


Figure 2 Cyclic voltammograms



Scheme 6 Proposed mechanism

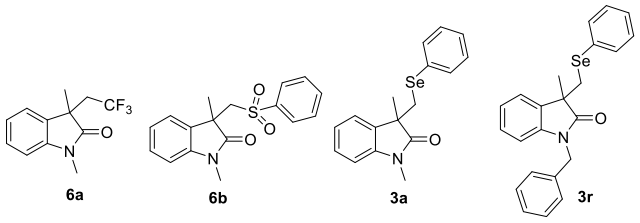


We used the MTT method to screen the *in vitro* cytotoxicities of the seleno oxindole compounds against four cancer cell lines (HeLa, T-24, MGC-803, and HepG-2) with 5-fluorouracil (**5-FU)** as a positive control to investigate the antitumor activity of the synthesized compounds (Table 2). The corresponding

Scheme 5 Control experiments

products **6a** and **6b** were prepared through the reaction of *N*-arylamides with sodium trifluoromethylsulfite and sodium phenylsulfinyl, and their antitumor activities were screened. The results showed that the seleno oxindole products had a better antitumor activity than other oxindole skeletons **6a** and **6b**. The antitumor activity of compound **3r** was better than that of the positive control **5-FU**. Compound **3r** exhibited a favorable anticancer activity toward HeLa and T-24 cell lines with IC_{50} values of 10.5 ± 0.8 and 15.4 ± 1.1 μM, respectively. We also studied the antitumor mechanism of compound **3r** on HeLa, and the results are presented in the **Supporting Information** (Figures S2–S5**)**.

Table 2 IC_{50} (μM) values of compounds on tumor cells



Comps	HeLa	T-24	MGC-803	HepG-2
6a	35.7 ± 0.7	32.4 ± 1.0	>40	>40
6b	30.9 ± 0.9	38.6 ± 0.5	>40	>40
3a	12.4 ± 1.2	23.0 ± 0.9	30.1 ± 0.8	>40
3r	10.5 ± 0.8	15.4 ± 1.1	21.6 ± 0.5	36.8 ± 1.3
5-FU	35.2 ± 0.9	26.7 ± 0.6	33.2 ± 1.4	>40

In summary, we developed an economical and practical method for the synthesis of selenide oxindole through the electrochemical tandem cyclization reaction. The seleno oxindole products exhibited better pharmacological activity than other oxindole derivatives. The antitumor activities of all compounds were screened by using the MTT method. The results showed that the seleno oxindole products had better antitumor activity than other oxindole skeletons.

Experimental Section

A 10 mL three-necked round-bottomed flask was charged with diselenide (0.2 mmol, 1.0 equiv), *N*-arylacrylamide (0.2 mmol, 1.0 equiv) and $^{n}Bu_4NPF_6$ (0.4 mmol, 2.0 equiv). The flask was equipped with a reflux condenser, a platinum plate (1 cm x 1 cm) anode and graphite rod (*Φ* 6 mm) cathode. CH_3CN (8 mL) and HFIP (2 mL) were added. Electrolysis was carried out at 80 °C (oil bath temperature), which using a constant current of 15 mA until the substrate was completely consumed (monitored by TLC, about 2 hours). After the reaction was completed, the solvent was concentrated under reduced pressure. Purification with silica gel column chromatography using petroleum ether/ethyl acetate to afford the desired products.

Acknowledgements

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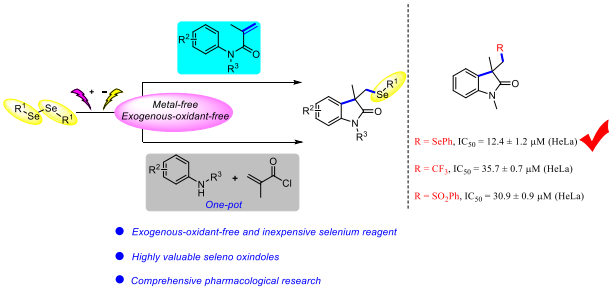
Synthesis of Seleno Oxindoles *via* Electrochemical Cyclization of *N*-Arylacrylamides with Diorganyl Diselenides



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