Direct Electrooxidative Selenylation/Cyclization of Alkynes: Access to Functionalized Benzo[b]furans

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Abstract: A mild, practical, metal and oxidant-free methodology for the synthesis of various C-3 selenylated benzo[b]furan derivatives was developed through the intramolecular cyclization of alkynes promoted with diselenides via electrooxidation. A wide range of selenium-substituted benzo[b]furan derivatives were obtained in good to excellent yields with high regioselectivity under constant current in an undivided cell equipped with carbon and platinum plates as the anode and cathode, respectively. Moreover, the convergent approach exhibited good functional group tolerance and could be easily scaled up with good efficiency, providing rapid access to a diverse range of selenylated benzo[b]furans derivatives from simple, readily available starting materials.

Keywords: electrochemical synthesis; organoselenium compounds; benzo[b]furans; cyclization; electrooxidation

1. Introduction

Organoselenium compounds, especially heterocycle motifs containing selenium, frequently play an important role in pharmaceutical, biological, and material applications as well as in modern organic synthesis because of its specific chemical and biological properties [1–6]. Selenium is an indispensable trace element in the human body; the introducing of the Se atom into organic molecules can modify their chemical and physical properties. Organoselenium compounds play an important role not only in organic transformation but also in pharmaceutical, biological, and material applications, especially selenium compounds containing heterocyclic units have unique biological activities [7–9], such as antitumor, antibacterial, anti-inflammatory, antiviral, cardiovascular protection, immune regulation, and other biological activities (Figure 1a). In particular, the selenylation of heteroarenes including the benzofuran moiety is a very important class of heterocycles widely found in natural products, dyes, materials, and pharmaceutical ingredients. Among the numerous benzofuran derivatives, 3-substituted benzo[b]furans display a wide array of biological activities that include anti-inflammatory [10,11], antibacterial [12,13], antimicrobial [14], antitumor [15,16], anticonvulsant [17,18], etc. Some representative examples of bioactive benzo[b]furans are outlined in Figure 1 (Figure 1b). Therefore, the development of an efficient and general method for the synthesis of selenated benzo[b]furans is still highly desired.

Over the past few decades, some significant progress has been made in the field of synthesis of organoselenium compounds [19–25] as well as benzofurans. Most of the transformations were traditionally utilized transition-metal catalysts or stoichiometric amounts of oxidants. For example, pioneering work toward the synthesis of selenated benzofurans relied on an electrophilic cyclization strategy using toxic and instable RSeCl as the selenium source [26,27]. Most of the research to access selenylbenzofurans mainly focused on the transition metals, such as the requirement of precious palladium catalyst or
stoichiometric amount of Fe/Cu/Ag catalysts (Scheme 1a) [28–33]. Recently, Frank and co-workers reported a catalytic strategy of direct C–H thiolation of heteroarenes to obtain selenylbenzofurans by a heterogeneous catalyst Pd/Al2O3, but it suffered from low yields and poor selectivity, particularly for the formation of selenylbenzofurans (Scheme 1b) [34]. Despite the significant achievements that have been made in this field, there are still some limitations, such as these methods involving transition metals, excess external oxidants, low reaction yields and poor selectivity. Undoubtedly, the development of an efficient, practical and greener procedure for the formation of selenylated benzofuran building blocks involving simple operation under mild reaction conditions is still urgent and highly desirable.

**Figure 1.** Representative biologically active molecules. (a) Selected examples of bioactive compounds containing selenium. (b) Representative biologically active molecules containing benzofuran motif.

**Previous work:**

(a) 

\[
\text{Alkyne} + \text{RSeR} \quad \rightarrow \quad \text{Selenylation/cyclization of alkyne}
\]

(b) 

\[
\text{Selenylation of heteroarenes.}
\]

**This work:**

(c) 

\[
\begin{array}{c}
\text{Graphite} \\
\text{Pt} \\
\end{array} \quad \xrightarrow{\text{Pd/Al2O3, Cu}} \quad \text{Selenylation/cyclization of alkyne}
\]

**Scheme 1.** Diverse synthetic strategies for selenylbenzofurans. (a) Transition-metal mediated selenylation/cyclization of alkyne. (b) Direct C–H selenylation of heteroarenes. (c) Direct electrooxidative selenylation/cyclization of alkyne.

In recent years, organic electrochemical synthesis has become an attractive approach in the field of organic synthesis because of its environmentally benign, sustainable, and practical nature [35–42]. Recently, several electrochemically induced radical functionalizations of alkyne have been reported [43–46]. Inspired by the recent developments in the area of electrochemical selenylation and our continuous research interest in the electrochem-
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2. Results and Dissections

2.1. Optimization of Reaction Conditions

We started our study by employing 2-alkynylanisole (1a) and diphenyl diselenide (2a) as model substrates to optimize the reaction conditions and explore the key variables (Table 1). When using the co-solvent of acetonitrile and hexafluoroisopropanol (HFIP), the desired product 3a could be delivered in an excellent yield of 96% in an undivided cell equipped with a graphite anode and a platinum plate cathode under a constant current of 10 mA in 2 h without any other additives in the presence of supporting electrolytes n-Bu$_4$NPF$_6$ at room temperature (Table 1, entry 1). The use of MeOH instead of the co-solvent of MeCN and HFIP delivered a low yield of desired product (entry 2). Upon use of MeCN (entry 3) as a sole solvent, a slightly decreased yield was obtained. To identify the electrolyte potentially suitable for the cyclization, various electrolytes were tested. The use of n-Bu$_4$NClO$_4$, n-Bu$_4$NBF$_4$, and LiClO$_4$ instead of n-Bu$_4$NPF$_6$ delivered lower yields (entries 4–6), while the use of n-Bu$_4$NPF$_6$ gave the best yield of the desired product (entry 1). Decreasing current to 5 mA afforded an obviously decreased yield of 87% in a longer time (entry 7). In the end, we explored the electrode materials for this electrochemical cyclization reaction. C(+)/C(−) electrodes led to diminished yields, while the Pt(+)/Pt(−) combination gave a very similar yield (entries 8 and 9). The use of 0.6 equivalent of diphenyl diselenide (2a) proved to be inefficient, delivering product 3a with 81% yield (entry 10). No desired product was observed without the electrolyte or electricity (entries 11 and 12).

Table 1. Optimization of the Reaction Conditions a.

| Entry | Deviation from Standard Conditions | Yield (%) b |
|-------|-----------------------------------|-------------|
| 1     | none                              | 96          |
| 2     | MeOH as solvent (6 h)             | 76          |
| 3     | MeCN as solvent (6 h)             | 92          |
| 4     | n-Bu$_4$NClO$_4$ instead of n-Bu$_4$NPF$_6$ | 85          |
| 5     | n-Bu$_4$NBF$_4$ instead of n-Bu$_4$NPF$_6$ | 85          |
| 6     | LiClO$_4$ instead of n-Bu$_4$NPF$_6$ | 75          |
| 7     | 5 mA instead of 10 mA (6 h)       | 87          |
| 8     | C(+)-C(−)                         | 85          |
| 9     | Pt plate as anode                 | 93          |
| 10    | 2a (0.18 mmol) instead of 2a (0.36 mmol) | 81          |
| 11    | no electrolyte                    | 0           |
| 12    | no electric current               | 0           |

a Reaction conditions: undivided cell, graphite anode (1.5 cm × 1 cm × 0.2 cm), Pt plate cathode (1 cm × 1 cm × 0.01 cm), 1a (0.3 mmol), 2a (0.36 mmol), n-Bu$_4$NPF$_6$ (0.5 mmol), MeCN/HFIP = 4:1 (5.0 mL), constant current = 10.0 mA, 2 h (2.5 F/mol), under air, at room temperature. b Isolated yields.

2.2. Substrate Scope

With the optimal reaction conditions in hand, we began to investigate the generality of this electrochemical protocol. A range of 2-alkynylanisoles bearing various substituents were explored under the standard reaction conditions, and the scope was summarized in Scheme 2. It was found that the R group bearing both an electron-donating group such as alkyl groups and an electron-withdrawing group such as halogens on the benzene ring was Synthetische Synthese [47–51], herein, we developed an electrochemical selenylation/cyclization method of alkynes for the synthesis of 3-selenylated benzofuran motifs with wide functional groups under mild electrochemical anodic oxidation conditions in an undivided cell (Scheme 1c).
amenable to the mild electrochemical selenylation/cyclization and afforded the desired products in good to excellent yields 85–96% (3a–3j). When the R was installed with a fused ring, such as naphthalene or even anthracene, the corresponding selenylated products 3k and 3l were still obtained in 82% and 94% yields, respectively. The structure of 3l was unambiguously confirmed by single crystal X-ray diffraction studies. While R was installed with aromatic heterocycles, pyridine and the thiophene ring were also suitable for the reaction, which gave the desired products 3m in 45% and 3o in 83% yields, respectively. It is particularly noteworthy that the electrochemical cyclization smoothly delivered the desired selenylated products in excellent yields when R was replaced by the hydroxyl alkyl groups (3p). Subsequently, the scope of the aryl ring with various substituents was tested. It was found that halogen and alkyl groups on the aryl ring as well as heterocycles were also proved to be tolerated under the standard reaction conditions and delivered the desired functionalized selenylbenzofurans in good to excellent yields (3q–3u). Notably, the 2-[(trimethylsilyl)ethynyl]anisole (1v) proved to be a suitable substrate, which gave the bis-selenylated benzofuran product 3v in moderate yield.

Scheme 2. Screening of substrates 1 scope with diphenyl diselenide 2a. Reaction conditions: undivided cell, graphite anode (1.5 cm × 1 cm × 0.2 cm), Pt plate cathode (1 cm × 1 cm × 0.01 cm), 1 (0.3 mmol), 2a (0.36 mmol), n-Bu4NPF6 (0.5 mmol), MeCN/HFIP = 4:1 (5.0 mL), constant current = 10.0 mA, 2 h, under air, at room temperature. a 6 h.

We next continued to speculate the cyclization of 2-alkynylanisole 1a with different diorganyl diselenides 2 (Scheme 3). The selenylation/cyclization regime proved to be tolerant to electron-donating and electron-withdrawing groups on the aromatic rings bonded to the selenium atom of the diaryl diselenides 2, giving the corresponding 3-organoselanyl-benzo[θ]furans 4 in good yields (4a–4j). Gratifyingly, the electrooxidative cyclization approach showed tolerance to the presence of heterocyclic diselenide, furnishing the benzo[θ]furans derivative 4k in a good yield of 83%. It is worth mentioning that the reaction demonstrated satisfactory tolerance not only with aryl diselenides but also aliphatic diselenide derivatives to prepare highly functionalized benzo[θ]furans. For example,
dimethyl diselenide (2l) and diethyl diselenide (2m) smoothly produced the selenylated products 4l and 4m in good yields.

Scheme 3. Screening of diselenides 2 scope with substrate 1a. Reaction conditions: undivided cell, graphite anode (1.5 cm × 1 cm × 0.2 cm), Pt plate cathode (1 cm × 1 cm × 0.01 cm), 1a (0.3 mmol), 2 (0.36 mmol), n-Bu4NPF6 (0.5 mmol), MeCN/HFIP = 4:1 (5.0 mL), constant current = 10.0 mA, 3 h, under air, at room temperature.

2.3. Scaled-Up Synthesis and Transformation

To prove the synthetic value of this electrochemical selenylation/cyclization reaction, scaled-up reactions of 1a and 1v were carried out with 2a under standard conditions. As shown in Scheme 4, the scale-up reactions afforded the corresponding compounds 3a (4.5 g) and 3t (1.85 g) in a high reaction efficiency in slightly reduced yields (85% and 84%) without an appreciable loss in efficacy.

2.4. Control Experiments and Plausible Mechanism

For a deeper understanding of the mechanism of this reaction, some control experiments were designed (Scheme 5). The solo product 3w of selenylation/cyclization with a phenol group was obtained by the intramolecular competition experiment, indicating the reaction preferred the single electron transfer (SET) (Scheme 5a, Pathway I). When electrolysis was performed in the presence of the radical scavenger 2,6-di-tert-butyl-4-methylphenol (BHT) under the standard reaction conditions, the desired product 3a had an obviously decreased yield of 57% (Scheme 5b), which implied that the process was likely proceeded by a favored radical pathway (Pathway I). Subsequently, when substrate 1a was treated with PhSeCl instead of diphenyl diselenide in MeCN/HFIP at room temperature, the desired product 3a was obtained in 48% yield (Scheme 5c) in 2 h, without electric current, which indicates that the electrochemical selenylation/cyclization process could not exclude the ionic pathway (Pathway II). Finally, we investigated the effect of
the substituents on the oxygen atom, and it showed that when the oxygen atom related H instead of the methyl group, the reaction was shown to have good tolerance with an 86% yield of 3a, whereas a phenyl group failed to deliver the desired product 3x with a recovery of 1x in 96% (Scheme 5d). The reaction mixture was monitored by GC-MS analysis (Figure S2), and the molecular weight of intermediate PhSeMe was captured. Thus, this reaction was initiated by the formation of seleno radical B and phenyl selenium anion H through cathode reduction. Then, the selenium anion H was reacted with methyl cation to produce PhSeMe (Scheme 6). These control experiments are suggesting that the diselenide can be involved in both processes (anodic oxidation and cathode reduction), producing seleno radical B and seleno cation C under electrochemical conditions.

Furthermore, cyclic voltammetry (CV) experiments were also performed to reveal key mechanistic insights into the electrochemical transformation (Figure 2). An obvious oxidation peak of substrate 1a could be observed at 1.61 V, which was higher than that of diphenyl diselenide (2a, 1.388 V), demonstrating that 2a should be preferentially oxidized at the anode in the electrochemical system.

Thus, based on the enriched evidence from the control experiments and previous related reports [52–54], a possible mechanism for the electrochemical selenylation/cyclization is described in Scheme 6. At the beginning, the reaction was initiated by the formation of seleno radical B and seleno cation C via a cathode reduction and the anodic oxidation of diphenyl diselenide (2a). Thereafter, the radical addition of B to 1a generates the radical intermediate D, intermediate D undergoes cyclization to give the intermediate E, intermediate E followed by electron loss afforded intermediate F. Alternatively, the ionic pathway cannot be omitted; that is, the selenium cation C can also add to 1a to provide the cationic intermediate G, and intermediate G undergoes cyclization to give the intermediate F, which then undergoes demethylation to produce the desired product 3a and PhSeMe with selenium anions.

Scheme 4. Gram-scale synthesis of 3a and 3t. (a) Reaction conditions: Undivided cell, graphite as the anode (3 cm × 3 cm × 0.6 cm), Pt plate as the cathode (3 cm × 3 cm × 0.01 cm), 1a (15.0 mmol), 2a (18 mmol), n-Bu4NPF6 (15.0 mmol), and MeCN (150 mL), constant current = 50.0 mA, 14 h, under air, room temperature. (b) 1a (5.0 mmol), 2a (6 mmol), n-Bu4NPF6 (15.0 mmol), and MeCN (150 mL), constant current = 50.0 mA, 5 h.

The substituted phenol group was obtained by the in tramolecular competition experiment, indicating the reaction preferred the single electron transfer (SET) (Scheme 5a, Pathway I). Subsequently, when substrate 2a was treated with PhSeCl instead of diphenyldiselenide in MeCN/HFIP at room temperature, the desired product 3a was obtained in 48% yield (Scheme 5b). A phenyl group failed to deliver the desired product 3x, which then undergoes demethylation to produce the desired product 3t in 96% (Scheme 5d). The reaction mixture was monitored by GC-MS analysis (Figure S2), and the molecular weight of intermediate PhSeMe was captured. Thus, this reaction was initiated by the formation of seleno radical B and phenyl selenium anion H through cathode reduction. Then, the selenium anion H was reacted with methyl cation to produce PhSeMe (Scheme 6). These control experiments are suggesting that the diselenide can be involved in both processes (anodic oxidation and cathode reduction), producing seleno radical B and seleno cation C under electrochemical conditions.

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Scheme 5. Control experiments. (a) Intramolecular competition experiment. (b) Radical trapping experiment. (c) Reaction with PhSeCl. (d) Reactions with different substituents.

Scheme 6. Proposed mechanism.
In summary, we have successfully developed an efficient electrochemical protocol for the construction of valuable selenylated benzo[b]furan derivatives through an electrooxidative process by the cyclization of 2-alkynylanisoles. Various 3-substituted benzofurans were obtained in good to excellent yields under metal- and oxidant-free mild reaction conditions. Furthermore, the current protocol was successfully applied to gram scale, and a conceivable reaction mechanism was proposed.

4. Materials and Methods

4.1. General Information

Electrochemical reactions were conducted using an AXIOMET AX-3003P potentiostat in constant current mode using an undivided cell equipped with a graphite plate (1.5 cm × 1.0 cm × 0.2 cm) as the anode and platinum plate (1.0 cm × 1.0 cm × 0.01 cm) as the cathode under air. Graphite plates are commercially available from Beijing Jinglong Special Carbon Technology Co., Ltd (Beijing, China). Platinum electrodes are commercially available from Tianjin Aida (Tianjin, China). The substrate 1a–1x and diselenides were synthesized according to previously described methods. Other chemicals were obtained from commercial sources and were used without further purification. Yields refer to isolated compounds, which were estimated to be >95% pure as determined by 1H-NMR. TLC: Macherey-Nagel, TLC plates Alugram® Sil G/UV254. Detection under UV light at 254 nm. Chromatography separations were carried out on silica gel 60H (200–300 mesh) manufactured by Qingdao Haiyang Chemical Group Co. (Qingdao, China). High-resolution mass spectrometry (HRMS) was measured on Thermo-DFS mass spectrometer. NMR spectra were recorded on JEOL 400 NMR (1H 400 MHz; 13C 100 MHz; 19F 376 MHz) in CDCl3. If not otherwise specified, chemical shifts (δ) are given in ppm.

4.2. Materials

The starting materials 1a–1x were prepared according to the reported methods [54]. All the solvents are commercially available and directly used in this electrochemical synthesis.

4.3. Procedure for the Electrosynthesis of Compounds 3

In an undivided cell (10 mL) equipped with a stirring bar, a mixture of substrates 1 (0.3 mmol), 2a (0.36 mmol), n-Bu4NPF6 (0.5 mmol) and MeCN/HFIP = 4:1 (5 mL) was added. The cell was equipped with a graphite plate (1.5 cm × 1.0 cm × 0.2 cm) as the anode and platinum plate (1.0 cm × 1.0 cm × 0.01 cm) as the cathode connected to an AXIOMET AX-3003P DC regulated power supply. The reaction mixture was stirred and electrolyzed at

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**Figure 2.** Cyclic voltammetry studies. Conditions: a 0.1 M n-Bu4NPF6 solution in MeCN/HFIP = 4:1 at room temperature; a glassy carbon working electrode, Ag/AgCl (3 M KCl) reference electrode, and a graphite counter electrode, respectively. Scan rate: 100 mV/s. (a) Black line: background; (b) Red line: 1a (1 mM); (c) Blue line: 2a (1 mM); (d) Green line: 3a (1 mM); (e) Pink line: 1a (1 mM) + 2a (1 mM).

**Scheme 6.** Proposed mechanism.
a constant current of 10 mA at room temperature for 2 h. Upon completion, the solvent was removed directly under reduced pressure to afford the crude product, which was further purified by flash column chromatography to afford the desired products 3a–3w.

2-(Phenylselanyl)-1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3a (101 mg, 96%). Mp: 62.0–63.8 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.22\) (d, \(J = 7.5\) Hz, 2H), 7.55 (dd, \(J = 16.3, 8.0\) Hz, 2H), 7.46 (m, 2H), 7.43–7.34 (m, 2H), 7.33–7.28 (m, 2H), 7.25–7.21 (m, 1H), 7.16 (m, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 157.4, 154.3, 132.1, 131.5, 130.3, 129.4, 129.3, 128.6, 127.9, 126.4, 125.4, 123.6, 121.4, 111.3, 99.8. Analytical data for compound 3a were consistent with the literature \[55\].

3-(Phenylselanyl)-2-(p-tolyl)benzofuran 3b: Prepared following general procedure A using substrate 1-methoxy-2-(phenylethynyl)benzene 1b (0.3 mmol, 67 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3b (104 mg, 95%). Mp: 84.1–85.3 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.14–8.10\) (m, 2H), 7.54 (dd, \(J = 16.3, 8.0\) Hz, 2H), 7.39–7.25 (m, 4H), 7.29–7.18 (m, 2H), 7.23–7.08 (m, 3H), 2.41 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 157.7, 154.1, 139.6, 132.1, 131.7, 129.4, 129.3, 128.7, 127.4, 126.3, 125.1, 123.5, 121.2, 111.2, 99.0, 21.6. Analytical data for compound 3b were consistent with the literature \[55\].

2-(4-Ethylphenyl)-3-(phenylselanyl)benzofuran 3c: Prepared following general procedure A using substrate 1-((4-ethylphenyl)ethynyl)-2-methoxybenzene 1c (0.3 mmol, 71 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3c (106 mg, 94%). Mp: 66.3–67.5 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.17–8.13\) (m, 2H), 7.58–7.51 (m, 2H), 7.36–7.28 (m, 5H), 7.24 (m, 1H), 7.20–7.12 (m, 3H), 2.71 (q, \(J = 7.5\) Hz, 2H), 1.28 (t, \(J = 7.6\) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 157.8, 154.2, 145.9, 132.1, 131.7, 129.4, 129.3, 129.2, 128.2, 127.9, 127.7, 126.3, 125.1, 123.5, 121.2, 111.2, 99.0, 28.9, 15.5. \(^{77}\)Se NMR (115 MHz, CDCl\textsubscript{3}) \(\delta = 462.5.\) HR-MS (ESI) \(m/z\) calc’d for C\textsubscript{22}H\textsubscript{19}OSe \([\text{M} + \text{H}]^+\) 379.0596, found 379.0521.

2-(4-Fluorophenyl)-3-(phenylselanyl)benzofuran 3d: Prepared following general procedure A using substrate 1-((4-fluorophenyl)ethyl)-2-methoxybenzene 1d (0.3 mmol, 68 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3d (100 mg, 91%). Mp: 78.8–79.5 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.22\) (m, 2H), 7.55 (m, 2H), 7.35 (m, 1H), 7.31–7.27 (m, 2H), 7.25 (m, 1H), 7.21–7.12 (m, 5H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 163.4\) (d, \(J_{\text{C-F}} = 249.0\) Hz), 155.4 (d, \(J_{\text{C-F}} = 234.0\) Hz), 132.0, 131.4, 130.9, 129.9, 129.5, 129.3, 129.2, 128.3, 127.9, 126.5, 125.4, 123.7, 121.3, 115.7 (d, \(J_{\text{C-F}} = 22.0\) Hz), 111.3, 99.5. \(^{19}\)F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta = –110.7.\) Analytical data for compound 3d were consistent with the literature \[32\].

2-(4-Chlorophenyl)-3-(phenylselanyl)benzofuran 3e: Prepared following general procedure A using substrate 1-((4-chlorophenyl)ethyl)-2-methoxybenzene 1e (0.3 mmol, 73 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3e (107 mg, 93%). Mp: 77.3–79.0 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.14\) (d, \(J = 8.4\) Hz, 2H), 7.51 (m, 2H), 7.39 (d, \(J = 8.5\) Hz, 2H), 7.32 (m, 1H), 7.26–7.19 (m, 3H), 7.13 (m, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 156.1, 154.2, 135.4, 132.0, 131.2, 129.5, 129.9, 128.7, 126.6, 125.6, 123.7, 121.4, 111.3, 100.4. Analytical data for compound 3e were consistent with the literature \[32\].

2-(4-Bromophenyl)-3-(phenylselanyl)benzofuran 3f: Prepared following general procedure A using substrate 1-((4-bromophenyl)ethyl)-2-methoxybenzene 1f (0.3 mmol, 86 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3f (116 mg, 90%). Mp: 91.2–91.5 °C. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta = 8.08\) (d, \(J = 8.6\) Hz, 2H), 7.58–7.45 (m, 4H), 7.32 (m, 1H), 7.26–7.18 (m, 3H), 7.17–7.08 (m, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta = 156.1, 154.2, 132.0, 131.8, 131.2, 129.5, 129.4, 129.3, 129.1, 126.6, 125.7, 123.7, 121.4, 111.3, 100.5. Analytical data for compound 3f were consistent with the literature \[32\].
N-(4-(3-(Phenylselanyl)benzofuran-2-yl)phenyl)acetamide (3g): Prepared following general procedure A using substrate N-(4-(2-methoxyphenyl)ethyl)phenyl)-acetamide 1g (0.3 mmol, 80 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow oil 3g (110 mg, 90%). 1H NMR (400 MHz, DMSO-d6) δ = 10.15 (s, 1H), 8.08-7.96 (m, 2H), 7.68 (d, J = 8.7 Hz, 2H), 7.62 (d, J = 8.1 Hz, 1H), 7.39–7.25 (m, 3H), 7.23–7.18 (m, 3H), 7.17–7.07 (m, 3H), 2.02 (s, 3H). 13C NMR (100 MHz, DMSO-d6) δ = 168.7, 156.8, 153.4, 140.7, 131.3, 130.8, 129.6, 128.9, 128.1, 126.6, 125.4, 123.8, 123.7, 120.5, 118.8, 111.4, 98.2, 24.2. HR-MS (ESI) m/z calcd for C22H22NaO2Se [M + Na]+ 430.0317, found 430.0315.

2-(3-Fluorophenyl)-3-(phenylselanyl)benzofuran (3h): Prepared following general procedure A using substrate 1-(3-fluorophenyl)ethyl)-2-methoxybenzene 1h (0.3 mmol, 68 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3h (94 mg, 85%). Mp: 59.6–60.8 °C. 1H NMR (400 MHz, CDCl3) δ = 8.04 (d, J = 7.9 Hz, 1H), 7.97 (d, J = 10.5 Hz, 1H), 7.54 (dd, J = 10.6, 8.1 Hz, 2H), 7.44–7.32 (m, 2H), 7.31–7.27 (m, 2H), 7.23 (d, J = 7.1 Hz, 1H), 7.16 (m, J = 3.3 Hz, 3H), 7.08 (m, 1H). 13C NMR (100 MHz, CDCl3) δ = 162.8 (d, 1J=CF = 244.0 Hz), 154.9 (d, 1J=CF = 145.0 Hz), 132.2 (d, 1H), 131.9, 131.1, 130.2 (d, 1J=CF = 9.0 Hz), 129.5, 129.5, 129.3, 126.6, 125.8, 123.7, 123.5 (d, 1J=CF = 2.0 Hz), 121.5, 116.3 (d, 1J=CF = 21.0 Hz), 114.7 (d, 1J=CF = 24.0 Hz), 111.4, 101.1. 19F NMR (376 MHz, CDCl3) δ = −112.17. HR-MS (ESI) m/z calcd for C20H14FOSe [M + H]+ 369.0188, found 369.0112.

2-(2-Chlorophenyl)-3-(phenylselanyl)benzofuran (3i): Prepared following general procedure A using substrate 1-chloro-2-(2-methoxyphenyl)ethyl)benzene 1i (0.3 mmol, 73 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a slight yellow solid 3i (102 mg, 89%). Mp: 84.0–85.0 °C. 1H NMR (400 MHz, CDCl3) δ = 7.60 (d, J = 8.2 Hz, 1H), 7.57–7.49 (m, 3H), 7.46–7.32 (m, 3H), 7.32–7.27 (m, 3H), 7.21–7.13 (m, 3H). 13C NMR (100 MHz, CDCl3) δ = 156.8, 155.0, 154.7, 153.7, 132.9, 131.1, 130.4, 130.2, 129.8, 129.4, 129.3, 126.5, 126.4, 125.5, 123.6, 121.4, 111.7, 104.0. Analytical data for compound 3i were consistent with the literature [32].

2-(2-Bromophenyl)-3-(phenylselanyl)benzofuran (3j): Prepared following general procedure A using substrate 1-bromo-2-(2-methoxyphenyl)ethyl)benzene 1j (0.3 mmol, 86 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow oil 3j (116 mg, 90%). 1H NMR (400 MHz, CDCl3) δ = 7.72 (d, J = 7.9 Hz, 1H), 7.60 (d, J = 8.2 Hz, 1H), 7.51 (d, J = 7.6 Hz, 2H), 7.42–7.33 (m, 3H), 7.29 (m, 3H), 7.20–7.14 (m, 3H). 13C NMR (100 MHz, CDCl3) δ = 158.0, 154.8, 153.3, 133.3, 130.3, 131.5, 131.3, 131.1, 130.3, 129.9, 129.2, 127.1, 126.4, 125.4, 124.3, 123.6, 121.4, 111.7, 103.8. HR-MS (ESI) m/z calcd for C20H14BrOSe [M + H]+ 428.9388, found 428.9383.

2-(Naphthalen-2-yl)-3-(phenylselanyl)benzofuran (3k): Prepared following general procedure A using substrate 2-(2-methoxyphenyl)ethyl)naphthalene 1k (0.3 mmol, 77 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3k (98 mg, 82%). Mp: 121.7–123.4 °C. 1H NMR (400 MHz, CDCl3) δ = 8.71 (s, 1H), 8.40 (d, J = 8.5 Hz, 1H), 7.91 (d, J = 8.5 Hz, 2H), 7.89–7.83 (m, 1H), 7.60 (dd, J = 16.0, 8.0 Hz, 2H), 7.53 (m, 2H), 7.37 (m, 3H), 7.32–7.24 (d, J = 7.5 Hz, 1H), 7.18 (m, 3H). 13C NMR (100 MHz, CDCl3) δ = 157.3, 154.4, 133.6, 133.2, 132.2, 131.6, 129.5, 129.5, 128.9, 128.2, 127.8, 127.6, 127.1, 126.6, 126.5, 125.4, 124.9, 123.6, 121.4, 111.3, 100.4. Analytical data for compound 3k were consistent with the literature [32].

2-(Phenanthren-9-yl)-3-(phenylselanyl)benzofuran (3l): Prepared following general procedure A using substrate 9-(2-methoxyphenyl)ethyl)phenanthrene 1l (0.3 mmol, 93 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3l (127 mg, 94%). Mp: 146.5–148.2 °C. 1H NMR (400 MHz, CDCl3) δ = 8.76 (dd, J = 17.8, 8.3 Hz, 2H), 8.03 (d, J = 8.3 Hz, 1H), 7.94 (s, 1H), 7.85 (d, J = 7.8 Hz, 1H), 7.66 (m, 6H), 7.44 (m, 1H), 7.38–7.31 (m, 3H),
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7.20–7.11 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 159.1, 155.0, 131.8, 131.6, 131.2, 131.0, 130.8, 130.8, 130.7, 130.2, 129.5, 129.3, 128.0, 127.1, 127.0, 126.8, 126.5, 126.1, 125.4, 123.7, 123.1, 122.8, 121.5, 111.7, 104.5. The compound 31 cannot be ionized in ESI and APCI.

2-(3-(Phenylselanyl)benzofuran-2-yl)pyridine (3m): Prepared following general procedure A using substrate 2-((2-methoxyphenyl)ethynyl)pyridine 1m (0.3 mmol, 64 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography (PE/EtOAc: 30/1→10/1) to give a yellow oil 3m (47 mg, 45%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.14 (d, $J$ = 8.6 Hz, 2H), 7.58 (dd, $J$ = 17.4, 8.1 Hz, 4H), 7.38 (m, 1H), 7.34–7.27 (m, 3H), 7.20 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 151.6, 154.2, 131.9, 131.8, 131.2, 129.5, 129.4, 129.3, 129.2, 129.1, 126.5, 125.6, 123.7, 121.4, 111.3, 100.5. HR-MS (ESI) $m/z$ calcd for C$_{19}$H$_{14}$OSe [M + H]$^+$ 352.0235, found 352.0234.

3-(Phenylselanyl)-2-(thiophen-3-yl)benzofuran (3o): Prepared following general procedure A using substrate 3-((2-methoxyphenyl)ethynyl)thiophene 1o (0.3 mmol, 64 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3o (86 mg, 81%). Mp: 84.1–85.4 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.95–7.86 (m, 1H), 7.52 (m, 2H), 7.45–7.37 (m, 1H), 7.32 (m, 3H), 7.28–7.19 (m, 1H), 7.22–7.08 (m, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 154.0, 131.9, 131.9, 131.2, 129.6, 129.5, 129.4, 129.1, 128.7, 127.5, 126.5, 125.3, 123.7, 120.9, 111.1, 99.2. HR-MS (ESI) $m/z$ calcd for C$_{18}$H$_{12}$OSe [M + H]$^+$ 356.9847, found 356.9843.

5-(3-(Phenylselanyl)benzofuran-2-yl)pentan-1-ol (3p): Prepared following general procedure A using substrate 7-((2-methoxyphenyl)hept-6-yn-1-ol 1p (0.3 mmol, 65 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow oil 3p (96 mg, 89%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.46 (m, 1H), 7.29 (d, $J$ = 7.4 Hz, 1H), 7.23 (m, 3H), 7.17 (m, 3H), 3.57 (t, $J$ = 6.5 Hz, 2H), 3.01 (t, $J$ = 7.5 Hz, 2H), 1.77 (m, 2H), 1.57 (m, 2H), 1.38 (m, 2H), 1.27 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 163.5, 154.6, 132.0, 130.8, 129.3, 129.2, 126.2, 124.3, 123.3, 120.5, 111.1, 100.4, 62.9, 32.5, 28.1, 27.4, 25.3. HR-MS (ESI) $m/z$ calcd for C$_{18}$H$_{21}$O$_2$Se [M + H]$^+$ 361.0701, found 361.0729.

4-Fluoro-2-phenyl-3-(phenylselanyl)benzofuran (3q): Prepared following general procedure A using substrate 1-fluoro-3-methoxy-2-(phenylethynyl)benzene 1q (0.3 mmol, 68 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3q (98 mg, 89%). Mp: 115.8–116.5 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.16 (d, $J$ = 7.3 Hz, 2H), 7.49–7.37 (m, 3H), 7.35 (m, 3H), 7.25–7.21 (m, 1H), 7.17 (m, 3H), 6.93–6.84 (m, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.8 (d, $^{2}$C$_{F}$ = 23.0 Hz), 156.0 (d, $^{2}$C$_{F}$ = 90.0 Hz), 155.4, 132.4, 129.7, 129.5, 129.4, 128.5, 128.2, 126.5, 125.5 (d, $^{3}$C$_{F}$ = 8.0 Hz), 120.4 (d, $^{3}$C$_{F}$ = 16.0 Hz), 109.8 (d, $^{3}$C$_{F}$ = 19.0 Hz), 107.6 (d, $^{4}$C$_{F}$ = 4.0 Hz), 96.5. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ = –123.1. HR-MS (ESI) $m/z$ calcd for C$_{20}$H$_{14}$FOSe [M + H]$^+$ 369.0188, found 368.0112.

5-Methyl-2-phenyl-3-(phenylselanyl)benzofuran (3r): Prepared following general procedure A using substrate 1-methoxy-4-methyl-2-(phenylethynyl)benzene 1r (0.3 mmol, 67 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3r (105 mg, 96%). Mp: 105.2–106.5 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.23 (d, $J$ = 7.7 Hz, 2H), 7.47 (m, 3H), 7.40 (m, 1H), 7.35 (s, 1H), 7.32 (m, 2H), 7.19 (m, 4H), 2.44 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.6, 152.7, 133.2, 132.1, 131.8, 130.4, 129.5, 129.3, 129.0, 128.6, 127.9, 126.7, 126.2,
121.0, 110.8, 99.3, 21.5. $^{77}\text{Se}$ NMR (115 MHz, CDCl$_3$) $\delta$ = 462.5. HR-MS (ESI) m/z calcd for C$_{21}$H$_{17}$O$_2$Se $[M + H]^+$ 365.0439, found 365.0438.

5-Chloro-2-phenyl-3-(phenylselanyl)benzofuran (3s): Prepared following general procedure A using substrate 4-chloro-1-methoxy-2-(phenylethynyl)benzene 1s (0.3 mmol, 73 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3s (100 mg, 87%). Mp: 96.5–98.0 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.22 (d, $J$ = 7.4 Hz, 2H), 7.53–7.40 (m, 4H), 7.32–7.27 (m, 3H), 7.20 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 158.8, 152.6, 133.7, 131.1, 129.8, 129.6, 129.4, 129.3, 128.7, 126.6, 125.6, 120.9, 112.4, 99.3. Analytical data for compound 3s were consistent with the literature [32].

2-(4-Bromophenyl)-5-methyl-3-(phenylselanyl)benzofuran (3t): Prepared following general procedure A using substrate 2-((4-bromophenyl)ethynyl)-1-methoxy-4-methylbenzene 1t (0.3 mmol, 90 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a white solid 3t (123 mg, 93%). Mp: 138.6–139.2 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.11 (d, $J$ = 8.1 Hz, 2H), 7.57 (d, $J$ = 8.5 Hz, 2H), 7.44 (d, $J$ = 8.3 Hz, 1H), 7.34 (s, 1H), 7.28 (m, 2H), 7.18 (m, 4H), 2.43 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 156.4, 152.6, 133.4, 132.1, 131.8, 131.4, 129.5, 129.3, 129.2, 129.0, 127.0, 126.4, 123.6, 121.1, 110.9, 100.0, 21.5. HR-MS (ESI) m/z calcd for C$_{21}$H$_{15}$BrOSe $[M + H]^+$ 442.9544, found 442.9534.

2-Phenyl-3-(phenylselanyl)furo[2,3-b]pyridine (3u): The general procedure A was followed using substrate 2-methoxyphenyl)trimethylsilane (0.36 mmol, 112 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a brown oil 3u (68 mg, 65%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.34 (d, $J$ = 7.60 (m, 2H), 7.77 (d, $J$ = 7.7 Hz, 1H), 7.53–7.41 (m, 2H), 7.30 (m, 3H), 7.25–7.13 (m, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.3, 150.9, 132.9, 130.8, 130.6, 130.5, 129.5, 129.3, 129.1, 128.0, 126.8, 125.5, 123.6, 121.1, 113.8, 111.5. Analytical data for compound 3u were consistent with the literature [56].

2-(2-Methoxyphenyl)-3-(phenylselanyl)benzofuran (3v): Prepared following general procedure A using substrate 2-(2-methoxyphenyl)ethyl)silane 1v (0.3 mmol, 61 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow oil 3v (98 mg, 76%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.61–7.55 (m, 2H), 7.50 (dd, $J$ = 16.4, 8.0 Hz, 2H), 7.37 (m, 3H), 7.30 (m, 4H), 7.25–7.17 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.3, 150.9, 132.9, 130.8, 130.6, 130.5, 129.5, 129.3, 129.1, 128.0, 126.8, 125.5, 123.6, 121.1, 113.8, 111.5. HR-MS (ESI) m/z calcd for C$_{19}$H$_{13}$NNaOSe $[M + Na]^+$ 374.0055, found 374.0069.

2,3-bis(Phenylselanyl)benzofuran (3w): Prepared following general procedure A using substrate 2-(2-methoxyphenyl)ethyl)silane 1v (0.3 mmol, 61 mg) and diphenyl diselenide 2a (0.36 mmol, 112 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 3w (100 mg, 88%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.60 (m, $J$ = 8.5, 4.1 Hz, 2H), 7.46 (m, $J$ = 13.4, 8.0 Hz, 2H), 7.35 (m, $J$ = 9.4, 7.0 Hz, 3H), 7.25–7.15 (m, 4H), 7.12–7.01 (m, 2H), 3.80 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.8, 156.6, 155.0, 131.9, 131.8, 131.4, 130.9, 129.7, 129.2, 129.1, 126.1, 124.8, 123.2, 121.1, 120.4, 119.3, 111.4, 103.2, 55.5. HR-MS (ESI) m/z calcd for C$_{21}$H$_{17}$O$_2$Se $[M + H]^+$ 381.0383, found 381.0385.

4.4. Procedure for the Electrolysis of Compounds 4

In an undivided cell (10 mL) equipped with a stirring bar, a mixture of substrates 1a (0.3 mmol), 2 (0.36 mmol), n-Bu$_4$NPF$_6$ (0.5 mmol) and MeCN/HFIP = 4:1 (5 mL) were added. The cell was equipped with a graphite plate (1.5 cm $\times$ 1.0 cm $\times$ 0.2 cm) as the anode and platinum plate (1.0 cm $\times$ 1.0 cm $\times$ 0.01 cm) as the cathode connected to an AXIOMET AX-3003P DC regulated power supply. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at room temperature for 3 h. Upon completion, the solvent was removed directly under reduced pressure to afford the crude product, which was further purified by flash column chromatography to afford the desired products 4a–4m.
2-Phenyl-3-(p-tolylselanyl)benzofuran (4a): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-di-p-tolyl diselenide 2a (0.36 mmol, 122 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4a (93 mg, 85%). Mp: 84.5–87.7 °C. 1H NMR (400 MHz, CDCl3) δ = 7.27–7.17 (m, 2H), 7.54 (m, 2H), 7.47 (m, 2H), 7.44–7.37 (m, 1H), 7.37–7.30 (m, 1H), 7.25–7.18 (m, 3H), 6.99 (d, J = 7.9 Hz, 2H), 2.26 (s, 3H). 13C NMR (100 MHz, CDCl3) δ = 157.1, 154.2, 136.4, 132.1, 130.3, 130.3, 129.7, 129.4, 128.6, 127.9, 127.6, 125.3, 123.5, 121.4, 111.3, 100.2, 21.1. Analytical data for compound 4a were consistent with the literature [55].

3-(4-Fluorophenyl)selanyl-2-phenylbenzofuran (4b): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(4-fluorophenyl)diselenide 2b (0.36 mmol, 125 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow oil 4b (91 mg, 83%). 1H NMR (400 MHz, CDCl3) δ = 8.26–8.10 (m, 2H), 7.55 (d, J = 8.2 Hz, 1H), 7.53–7.43 (m, 3H), 7.40 (m, 1H), 7.36–7.26 (m, 3H), 7.23 (m, 1H), 6.87 (m, 2H). 13C NMR (100 MHz, CDCl3) δ = 162.0 (d, J_C-F = 244.0 Hz), 155.7 (d, J_C-F = 291.0 Hz), 131.8, 131.5 (d, J_C-F = 8.0 Hz), 130.2, 129.5, 128.6, 127.9, 127.8, 125.7 (d, J_C-F = 3.0 Hz), 125.4, 123.6, 121.2, 116.6 (d, J_C-F = 22.0 Hz), 111.4, 100.2. 19F NMR (376 MHz, CDCl3) δ = −115.7. Analytical data for compound 4b were consistent with the literature [32].

3-(4-Chlorophenyl)selanyl-2-phenylbenzofuran (4c): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(4-chlorophenyl)diselenide 2c (0.36 mmol, 137 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4c (105 mg, 91%). Mp: 86.8–87.4 °C. 1H NMR (400 MHz, CDCl3) δ = 8.22 (d, J = 7.6 Hz, 2H), 7.60 (d, J = 8.3 Hz, 1H), 7.55–7.41 (m, 4H), 7.38 (m, 1H), 7.31–7.27 (m, 1H), 7.26–7.22 (m, 2H), 7.18–7.13 (m, 2H). 13C NMR (100 MHz, CDCl3) δ = 157.5, 154.3, 132.5, 131.7, 130.6, 130.1, 129.7, 129.6, 128.7, 127.9, 125.5, 123.7, 121.2, 111.4, 99.5. Analytical data for compound 4c were consistent with the literature [55].

3-(4-Bromophenyl)selanyl-2-phenylbenzofuran (4d): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(4-bromophenyl)diselenide 2d (0.36 mmol, 169 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4d (107 mg, 83%). Mp: 98.1–99.7 °C. 1H NMR (400 MHz, CDCl3) δ = 8.18 (d, J = 7.5 Hz, 2H), 7.57 (d, J = 8.2 Hz, 1H), 7.46 (m, 5H), 7.35 (m, 1H), 7.27 (m, 1H), 7.24 (m, 1H), 7.14 (m, 2H). 13C NMR (100 MHz, CDCl3) δ = 157.6, 154.3, 132.4, 131.7, 130.8, 130.5, 130.0, 129.6, 128.7, 127.9, 125.5, 123.7, 121.1, 120.4, 111.4, 99.4. Analytical data for compound 4d were consistent with the literature [32].

2-Phenyl-3-(m-tolylselanyl)benzofuran (4e): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-di-m-tolyldiselenide 2e (0.36 mmol, 122 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4e (95 mg, 87%). Mp: 66.5–67.1 °C. 1H NMR (400 MHz, CDCl3) δ = 8.27 (m, 2H), 7.59 (m, 2H), 7.49 (m, 2H), 7.46–7.41 (m, 1H), 7.40–7.35 (m, 1H), 7.28 (m, 1H), 7.20 (s, 1H), 7.14–7.06 (m, 2H), 6.99 (d, J = 7.1 Hz, 1H), 2.27 (s, 3H). 13C NMR (100 MHz, CDCl3) δ = 157.3, 154.2, 139.2, 132.1, 131.3, 130.3, 129.8, 129.4, 129.3, 128.6, 127.9, 127.3, 126.3, 125.3, 123.5, 121.4, 111.3, 99.9, 21.5. Analytical data for compound 4e were consistent with the literature [32].

3-(3-Chlorophenyl)selanyl-2-phenylbenzofuran (4f): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(3-chlorophenyl)diselenide 2f (0.36 mmol, 137 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4f (100 mg, 87%). Mp: 58.4–59.2 °C. 1H NMR (400 MHz, CDCl3) δ = 8.20 (d, J = 7.5 Hz, 2H), 7.59 (d, J = 8.2 Hz, 1H), 7.48 (m, 4H), 7.38 (m, 1H), 7.32–7.26 (m, 2H), 7.14 (m, 2H), 7.10–7.05 (m, 1H), 13C NMR (100 MHz, CDCl3) δ = 157.7, 154.3, 135.2, 133.4, 131.7, 130.4, 130.0, 129.6, 128.7, 127.9, 127.1, 126.6, 125.5, 123.7, 121.1, 111.4, 99.1. HR-MS (ESI) m/z calcd for C20H14Cl2OSe [M + H]+ 384. 9893, found 384. 9887.
3-((3-Bromophenyl)selanyl)-2-phenylbenzofuran (4g): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(3-bromophenyl)diselenide 2g (0.36 mmol, 169 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4g (108 mg, 94%). Mp: 90.4–92.1 °C. 1H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.18 (d, $J = 7.3$ Hz, 2H), 7.61 (d, $J = 8.2$ Hz, 1H), 7.54 (d, $J = 7.9$ Hz, 2H), 7.49–7.35 (m, 5H), 7.29 (m, 1H), 7.13–7.05 (m, 1H), 6.94 (m, 1H), 6.85 (m, 1H). 13C NMR (100 MHz, CDCl$_3$) $\delta$ = 156.3, 154.0, 137.3, 131.8, 131.5, 130.3, 130.0, 129.7, 128.6, 128.1, 127.9, 127.7, 127.0, 126.5, 123.6, 123.1, 121.3, 111.3, 98.6. HR-MS (ESI) m/z calc'd for C$_{18}$H$_{13}$OSSe [M + H]$^+$ 356.9847, found 356.9843.

2-Phenyl-3-((3-trifluoromethyl)phenyl)diselenide (4l): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and dimethyl diselenide 2k (0.36 mmol, 117 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4l (83 mg, 83%). Mp: 90.4–92.1 °C. 1H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.29 (d, $J = 7.6$ Hz, 2H), 7.72 (d, $J = 7.5$ Hz, 1H), 7.55 (m, 3H), 7.47 (m, 4H), 6.95–6.87 (m, 1H). 13C NMR (100 MHz, CDCl$_3$) $\delta$ = 156.3, 154.0, 137.3, 131.8, 131.5, 130.3, 130.0, 129.7, 128.6, 128.1, 127.9, 127.7, 127.0, 126.5, 123.8, 121.3, 111.3, 102.2. HR-MS (ESI) m/z calc'd for C$_{18}$H$_{13}$OSSe [M + H]$^+$ 356.9847, found 356.9843.

3-((3-Chlorophenyl)selanyl)-2-phenylbenzofuran (4j): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(3-chlorophenyl)diselenide 2j (0.36 mmol, 169 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4j (98 mg, 90%). Mp: 108 mg, 90%. 1H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.23 (d, $J = 7.4$ Hz, 2H), 7.62 (d, $J = 8.3$ Hz, 1H), 7.48 (m, 4H), 7.39 (m, 1H), 7.29 (d, $J = 7.5$ Hz, 1H), 7.22 (d, $J = 7.4$ Hz, 1H), 7.11 (m, 1H), 7.02 (d, $J = 7.7$ Hz, 1H), 6.93 (m, 1H), 2.53 (s, 3H), 13C NMR (100 MHz, CDCl$_3$) $\delta$ = 157.7, 154.0, 132.3, 130.7, 129.1, 128.6, 128.4, 128.6, 127.9, 126.9, 126.2, 125.4, 123.6, 121.4, 111.3, 99.2, 21.6. Analytical data for compound 4j were consistent with the literature [55].

2-Phenyl-3-(3-(methylselanyl)-2-phenylbenzofuran (4h): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and 1,2-bis(3-(methylselanyl)phenyl)diselenide 2h (0.36 mmol, 161 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a colorless liquid 4h (98 mg, 90%). 1H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.18 (d, $J = 7.5$ Hz, 2H), 7.54–7.45 (m, 4H), 7.45–7.42 (m, 1H), 7.42–7.34 (m, 1H), 7.21–7.17 (m, 1H), 6.94 (m, 1H), 6.85 (m, 1H). 13C NMR (100 MHz, CDCl$_3$) $\delta$ = 153.4, 150.6, 136.0, 132.3, 131.0, 130.0, 129.3, 128.9, 128.7, 127.9, 127.7, 127.0, 126.5, 123.8, 121.4, 111.3, 98.6. HR-MS (ESI) m/z calc'd for C$_{18}$H$_{13}$OSSe [M + H]$^+$ 356.9847, found 356.9843.

2-Phenyl-3-(3-(methylselanyl)-2-phenylbenzofuran (4i): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and dimethyl diselenide 2k (0.36 mmol, 117 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a yellow solid 4i (83 mg, 83%). Mp: 108 mg, 90%. 1H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.29 (d, $J = 7.6$ Hz, 2H), 7.72 (d, $J = 7.5$ Hz, 1H), 7.55 (m, 3H), 7.47 (m, 4H), 6.95–6.87 (m, 1H). 13C NMR (100 MHz, CDCl$_3$) $\delta$ = 156.3, 154.0, 137.3, 131.8, 131.5, 130.3, 130.0, 129.7, 128.6, 128.1, 127.9, 127.7, 127.0, 126.5, 123.8, 121.3, 111.3, 102.2. HR-MS (ESI) m/z calc'd for C$_{18}$H$_{13}$OSSe [M + H]$^+$ 356.9847, found 356.9843.
3-(Ethylselenyl)-2-phenylbenzofuran (4m): Prepared following general procedure B using substrate 1-methoxy-2-(phenylethynyl)benzene 1a (0.3 mmol, 62 mg) and diethyldiselenide 2m (0.36 mmol, 78 mg). Isolation was purified by flash chromatography and eluted with petroleum ether to give a colorless liquid 4m (57 mg, 63%). 

$\text{H NMR (400 MHz, CDCl}_3 \delta = 8.35–8.29 \text{ (m, 2H), 7.74–7.67 \text{ (m, 1H), 7.56–7.46 \text{ (m, 3H), 7.40 \text{ (m, 1H), 7.37–7.28 \text{ (m, 2H), 2.82 \text{ (q, J = 7.4 Hz, 2H), 1.33 \text{ (t, J = 7.4 Hz, 3H).}}}}}$

$\text{C NMR (100 MHz, CDCl}_3 \delta = 156.2, 154.0, 132.9, 130.8, 129.0, 128.5, 127.8, 125.1, 123.3, 121.2, 111.2, 100.1, 22.1, 16.0. HR-MS (ESI) m/z calcd for C$_{16}$H$_{15}$OSe [M + H]$^+$ 303.0283, found 303.0279.

4.5. Procedure for the Scaled-Up Synthesis of Compound 3a and 3t

In an undivided cell (250 mL) equipped with a stirring bar, a mixture of substrates 1a (15.0 mmol, 3.12 g), 2a (18.0 mmol, 5.62 g), $n$-Bu$_4$NPF$_6$ (25.0 mmol, 9.69 g) and MeCN/HFIP = 4:1 (150 mL) were added. The cell was equipped with a graphite plate (3 cm × 3 cm × 0.01 cm) as the anode and platinum plate (3 cm × 3 cm × 0.01 cm) as the cathode and connected to a DC regulated power supply. The reaction mixture was stirred and electrolyzed at a constant current of 50 mA at 23 °C bath for 14 h. When the reaction was finished, the mixture was concentrated under reduced pressure. Purification by column chromatography on silica gel (eluent: petroleum ether) yielded solo product 3a (4.45 g, 85%) as yellow solid.

In an undivided cell (250 mL) equipped with a stirring bar, a mixture of substrates 1t (5.0 mmol, 1.50 g), 2a (6.0 mmol, 1.87 g), $n$-Bu$_4$NPF$_6$ (8.3 mmol, 3.23 g) and MeCN/HFIP = 4:1 (150 mL) were added. The cell was equipped with a graphite plate (3 cm × 3 cm × 0.01 cm) as the anode and platinum plate (3 cm × 3 cm × 0.01 cm) as the cathode and connected to a DC regulated power supply. The reaction mixture was stirred and electrolyzed at a constant current of 50 mA at 23 °C bath for 5 h. When the reaction was finished, the mixture was concentrated under reduced pressure. Purification by column chromatography on silica gel (eluent: petroleum ether) yielded solo product 3t (1.86 g, 84%) as white solid.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/molecules27196314/s1, Figure S1: The setup of electrochemical reaction; Figure S2: GC-MS detection of PhSeMe; Figure S3: Cyclic voltammetry studies; Crystallographic details; Figure S4: X-ray structure of 3l. $^1$H, $^{13}$C, $^{19}$F, $^{77}$Se NMR spectra of products; Table S1: Optimization of the Reaction Conditions; Gram-scale synthesis; Control experiments. Table S2: Crystal data and structure refinement for 3l.

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