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# **Supporting Information**

# Tunable selective electrochemical selenization of

# tetrahydroquinolines with diselenides

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# 1. General methods

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) with Hai yang GF 254 silica gel plates (Qingdao Haiyang chemical industry Co Ltd, Qingdao, China) using UV light and vanillic aldehyde or phosphomolybdic acid as visualizing agents. flash column chromatography was performed using 200-300 mesh silica gel at increased pressure. <sup>1</sup>HNMR and <sup>13</sup>CNMR spectra were recorded on 600 MHz or 400 MHz NMR spectrometers. Chemical shifts ( $\delta$ ) were expressed in ppm with TMS as the internal standard, and coupling constants (*J*) were reported in Hz. High-resolution mass spectra were obtained by using ESI ionization sources (quadrupole time-of-flight mass spectrometer, Bruker Impact II, Bremen, Germany). Cyclic voltammograms were obtained on a CHI 700E potentiostat (CH Instruments, Inc.).

**Abbreviations**: DMF = N,N-dimethylformamide, DMSO = dimethyl sulfoxide, DCE = dichloroethane, DCM = dichloromethane, MeCN = acetonitrile, MeOH= methanol, THF = tetrahydrofuran, DMSO= Dimethyl sulfoxide, DMA = N,N-Dimethylaniline, TEMPO = 2,2,6,6-tetramethylpiperidinooxy, BHT = butylated hydroxytoluene, DPE = 1,1-diphenyl-ethylene, N.D. = no detected, N.R. = no reaction.

### 2. Experimental procedures

#### **2.1** General procedure for the preparation of diselenides<sup>[1.2]</sup>





The mixture of phenylboronic acid (10.0 mmol, 1.0 *equiv.*), selenium (15.0 mmol, 1.5 *equiv.*), and silver nitrate (2.0 mmol, 0.2 *equiv.*) in 40 mL of DMSO was stirred and refluxed at 120 °C (oil bath), and monitored by TLC. After the reaction was complete, 20 mL of H<sub>2</sub>O and 20 mL of ethyl acetate were added to the reaction mixture. The organic layer was then separated, dried with Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under vacuum. The crude product was purified by flash column chromatography to provide diselenide.

**Caution!** Organoselenides are known to be toxic and should be handled in a well ventilated fume hood and using proper personal protective equipment.

# 2.2 General procedure for the electrochemical selenization of tetrahydroquinolines

#### 2.2.1 Synthesis of C-3 selenylated quinolines 3



Substrate 1 (0.3 mmol, 1 *equiv.*), substrate 2 (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*), H<sub>2</sub>O (0.2 mL) and MeCN (6 mL) were added to an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stirring bar. Two platinum plates (1 cm x 1 cm x 0.2 mm, each) were used as anode and cathode respectively (the electrodes were immersed 1 cm in the reaction solution). The reaction mixture was stirred and electrolyzed at a constant current of 15 mA at RT. After reaction completion (monitored by TLC), the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to obtain the target product **3**.

#### 2.2.2 Synthesis of C-6 selenylated tetrahydroquinolines 4



Substrate 1 (0.2 mmol, 1 *equiv.*), substrate 2 (0.6 mmol, 3 *equiv.*), NaI (0.4 mmol, 2 *equiv.*), and MeOH (6 mL) were added to an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stirring bar. Two platinum plates (1 cm x 1 cm x 0.2 mm, each) were used as anode and cathode respectively (the electrodes were immersed 1 cm in the reaction mixture). The reaction mixture was stirred and electrolyzed at a constant current of 15 mA at RT. After reaction completion (monitored by TLC), the reaction mixture was diluted with water, and extracted with ethyl acetate. The organic layers were combined, washed with brine, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to obtain the target product **4**.



Figure S1. Electrochemical setup used

The experimental setup consisted of two platinum sheet electrodes ( $10 \text{ mm} \times 10 \text{ mm} \times 0.2 \text{ mm}$ , each), a beaker-type electrolysis cell (10 mL), an adjustable DC regulated power supply (MS-150V 100 mA), a magnetic stirrer.

#### 2.3 Gram-scale synthesis of 3a



Substrate **1a** (6 mmol, 1 *equiv.*), substrate **2a** (12 mmol, 2 *equiv.*), NaI (12 mmol, 2 *equiv.*), TEMPO (6 mmol, 1 *equiv.*), H<sub>2</sub>O (2.0 mL), and MeCN (90 mL) were added to a beaker-type electrolysis cell (100 mL) equipped with a magnetic stir bar. Two platinum plates (3 cm x 3 cm x 0.2 mm, each) were used as anode and cathode respectively (the electrodes were immersed 3 cm in the reaction solution). The reaction mixture was stirred and electrolyzed at a constant current of 50 mA at RT. After reaction completion (monitored by TLC), the reaction mixture was diluted with water, and extracted with ethyl acetate. The organic layers were combined, washed with brine, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to obtain the target product **3a** (1.17 g, 68% yield). In addition, **2a** (1.66 g) was recovered.



Figure S2. Electrochemical setup for the scale-up experiment.

The electrochemical setup for the scale-up experiment consisted of two platinum sheet electrodes ( $30 \text{ mm} \times 30 \text{ mm} \times 0.2 \text{ mm}$ , each), a beaker-type electrolysis cell (100 mL), an adjustable DC regulated power supply (MS-150V 100 mA), and a magnetic stirrer.

# 3. Optimization of reaction conditions

#### 3.1 Optimization of reaction conditions for the synthesis of 3

Tab	le S1.	Screening	of the	amount of 2a	a
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+ 1a	Se Se Se 2a	Pt(+)   Pt(-), 15 mA Nal (2 <i>eq</i> .) TEMPO (1 <i>eq</i> .) MeCN, H <sub>2</sub> O open to air, RT	Se N 3a
Entry	2	a (equiv.)	Yield (%) <sup>b</sup>
1		0.5	40
2		0.6	41
3		0.8	47
4		1.0	55
5		1.1	62
6		1.3	59
7		1.5	65
8		2.0	64
9		2.5	78
10		3.0	85
11		4.0	75

<sup>a</sup>Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (x *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield.





1	MeCN	85
2	DMF	33
3	DMSO	18
4	THF	70
5	MeOH	25
6	DCM	voltage overload
7	1,4-dioxane	voltage overload
8	acetone	45

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in solvent (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield.

Table S3. Electrolyte screen	ing <sup>a</sup>
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LiBr

LiBF4

NaClO<sub>4</sub>

nBu<sub>4</sub>NCl

nBu<sub>4</sub>NBr

nBu<sub>4</sub>NI

nBu<sub>4</sub>NClO<sub>4</sub>

nBu<sub>4</sub>NBF<sub>4</sub>

53

tracec

trace

trace

trace

trace

tracec

tracec

<sup>a</sup> Reaction conditions: A mixture of <b>1a</b> (0.3 mmol, 1 <i>equiv.</i> ), <b>2a</b> (0.9 mmol, 3 <i>equiv.</i> ), electrolyte (0.6 mmol, 2
equiv.), TEMPO (0.3 mmol, 1 equiv.) and H <sub>2</sub> O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (Pt
anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated
vield. <sup>c</sup> The main product was quinoline.

Table S4. Mediator screening<sup>a</sup>

7

8

9

10

11

12

13

14

		Pt(+)   Pt(-), 15 mA	
+	Se_Se	Nal (2 <i>eq</i> .) Mediator (1 <i>eq</i> .)	Se
N		MeCN, H <sub>2</sub> O	
1a <sup>H</sup>	2a	open to air, RT	3a
Entry	Me	diator	Yield (%) <sup>b</sup>
1	W	thout	trace <sup>c</sup>
2	TE	MPO	85
3	fer	rocene	trace
4	N,N,N-tri	phenylamine	trace
5	N,N-bis(4-bromopl	nenyl)-4-bromoaniline	trace
6	9-azabicyclo[3.	3.1]nonane N-oxyl	45

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), mediator (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield. <sup>c</sup> **4a** was obtained in 40% yield.

Table S5.	. Screening	of the amount	of NaI <sup>a</sup>
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<sup>a</sup>Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (x *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield.

#### Table S6. Screening of the amount of TEMPO<sup>a</sup>



Entry	TEMPO (equiv.)	Yield (%) <sup>b</sup>
1	0	trace <sup>c</sup>
2	0.2	60
3	0.5	62
4	0.8	65
5	1.0	85
6	1.5	79
7	2.0	75

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (x *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield. <sup>C</sup> **4a** was obtained in 40% yield.

#### Table S7. Current screening<sup>a</sup>

+ + S 1a	Pt(+)   Pt(-) Nal (2 eq.) TEMPO (1 eq.) MeCN, H <sub>2</sub> O open to air, RT	$\rightarrow$ $N$ $Se$ $C$ $3a$
Entry	Current (mA)	Yield (%) <sup>b</sup>
1	9	33
2	12	51
3	15	85
4	18	57
5	21	46

<sup>a</sup> Reaction conditions: A mixture of 1a (0.3 mmol, 1 equiv.), 2a (0.9 mmol, 3 equiv.), NaI (0.6 mmol, 2 equiv.),

TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield.



#### Table S8. Electrode material screening<sup>a</sup>

2	Pt(+)   C(-)	46
3	C(+)   Pt(-)	64
4	C(+)   C(-)	67

<sup>a</sup>Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (0.2 mL) in MeCN (6 mL) under a constant current of 15 mA (x anode, x cathode) in an undivided cell at RT for 6 h. b Isolated yield.

Table S9. Screening of the amount of H<sub>2</sub>O<sup>a</sup>



<sup>a</sup> Reaction conditions: A mixture of **1a** (0.3 mmol, 1 *equiv.*), **2a** (0.9 mmol, 3 *equiv.*), NaI (0.6 mmol, 2 *equiv.*), TEMPO (0.3 mmol, 1 *equiv.*) and H<sub>2</sub>O (x mL) in MeCN (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 6 h. <sup>b</sup> Isolated yield.

#### 3.2 Optimization of the reaction conditions for the synthesis of 4

Table S10. Solvent screening<sup>a</sup>

+ So 1a H	2a <sup>opr</sup>	Pt(-), 15 mA  al (2 <i>eq.</i> ) Solvent en to air, RT 4a	N H
Entry	Solvent	Yield (	(%) <sup>b</sup>
1	MeCN	40	
2	DCM	41	
3	DMF	47	
4	DMSO	55	
5	DMA	62	
7	МеОН	74	

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv.*), **2a** (0.6 mmol, 3 *equiv.*), NaI (0.4 mmol, 2 *equiv.*) in solvent (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 5 h. <sup>b</sup> Isolated yield. <sup>c</sup> Without H<sub>2</sub>O

1a H + $S$	e_Se	Pt(+)   Pt(-), 15 mA Electrolyte (2 eq.) MeOH open to air, RT	Se 4a				
Entry	Elec	etrolyte	Yield (%) <sup>b</sup>				
1	wi	thout	voltage overload				
2	nBi	u4NBr	35				
3	nE	Bu4NI	50				
4	<i>n</i> Bu	14NPF6	trace				
5	<i>n</i> Bu	4NBF4	trace				
6	nBu	4NClO <sub>4</sub>	trace				
7	N	IH4I	64				
8	I	NaI	74				
9°	]	NaI	N.D.				

Tal	ble	<b>S11</b> .	Electro	lyte	screening
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<sup>a</sup> Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv.*), **2a** (0.6 mmol, 3 *equiv.*), Electrolyte (0.4 mmol, 2 *equiv.*) in MeOH (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 5 h.<sup>b</sup> Isolated yield. <sup>c</sup> Without current.

Table S12.	Screening of the	e amount of electrolyte <sup>a</sup>	
Table S12.	Screening of the	e amount of electrolyte <sup>a</sup>	

+ + 1a H	Se_Se 2a	Pt(+)   Pt(-), 15 mA Nal MeOH open to air, RT	Se 4a H
Entry	Na	I (equiv.)	Yield (%) <sup>b</sup>
1		1	36
2		1.5	39
3		2	74
4		2.5	35
5		3	31

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv.*), **2a** (0.6 mmol, 3 *equiv.*), NaI (x *equiv.*) in MeOH (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 5 h. <sup>b</sup> Isolated yield.

Table S13. Screening of the amount of 2a<sup>a</sup>

+ + 1a H	Se_Se 2a	Pt(+)   Pt(-), 15 mA Nal (2 <i>eq.</i> ) MeOH open to air, RT	Aa H
Entry	28	n (equiv.)	Yield (%) <sup>b</sup>
1		0.5	trace
2		1	23
3		2	43
4		2.5	61
5		3	74
6		3.5	52

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv.*), **2a** (x *equiv.*), NaI (0.4 mmol, 2 *equiv.*) in MeOH (6 mL) under a constant current of 15 mA (Pt anode: 1 cm x 1 cm x 0.2 mm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT for 5 h.<sup>b</sup> Isolated yield.





<sup>a</sup> Reaction conditions: A mixture of **1aa** (0.2 mmol, 1 *equiv.*), **2a** (0.6 mmol, 3 *equiv.*), NaI (0.4 mmol, 2 *equiv.*) in MeOH (6 mL) under a constant current of 15 mA (x anode; x cathode) in an undivided cell at RT for 5 h. <sup>b</sup> Isolated yield.

Table S15. Current screening<sup>a</sup>



1	12	29
2	15	74
3	18	50
4	21	20

<sup>a</sup> Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv.*), **2a** (0.6 mmol, 3 *equiv.*), NaI (0.4 mmol, 2 *equiv.*) in MeOH (6 mL) under a constant current (x anode; x cathode) in an undivided cell at RT for 5 h. <sup>b</sup> Isolated yield.

# 4. Mechanistic investigation

#### 4.1 Cyclic voltammetry experiments

The electrochemical measurement was performed by a computer-controlled electrochemical analyzer. The cyclic voltammetry experiment was conducted in a three-electrode cell (volume 15 mL; MeCN as the solvent, 0.05 M of *n*Bu<sub>4</sub>NClO<sub>4</sub> as the supporting electrolyte, 2 mM concentration of test compound), and glassy carbon (diameter 3 mm) as working electrode, platinum wire as auxiliary electrode, Hg/Hg<sub>2</sub>Cl<sub>2</sub> (3 M KCl) as reference electrode. The scanning speed was 100 mV·s<sup>-1</sup>. For tetrahydroquinoline (1a), 1,2-diphenyldiselane (2a), NaI, and TEMPO, the oxidation potential range studied was from 0.0 V to +3.0 V, relative to Hg/Hg<sub>2</sub>Cl<sub>2</sub> (3 M KCl). Meanwhile, the reduction potentials were determined for 1a and 2a. The reduction potential range studied was from 0.0 V to -3.0 V, relative to Hg/Hg<sub>2</sub>Cl<sub>2</sub> (3 M KCl).



**Figure S4.** Cyclic voltammogram of NaI, TEMPO, **1a**, and **2a** in an electrolyte of *n*Bu<sub>4</sub>NClO<sub>4</sub> (0.05 M) in MeCN from 0 to +3.0 V.



**Fig. S5.** Cyclic voltammogram of **1a** and **2a** in an electrolyte of *n*Bu<sub>4</sub>NClO<sub>4</sub> (0.05 M) in MeCN from 0 to -3.0 V.

#### 4.2 Radical trapping experiments

Under standard conditions for **3** synthesis, BHT and DPE (4.0 equiv to **1a**) were added separately to the model reaction system at the beginning of the reaction. After 3 h, a small amount of reaction mixture was taken for high-resolution mass spectrometry (HRMS) analysis. From TLC, only trace amounts of product **3a** was observed. Meanwhile, under standard conditions for 4 synthesis, BHT and DPE (4.0 equiv to **1a**) were added separately to the model reaction system at the beginning of the reaction. After 3 h, a small amount of reaction mixture was taken for high-resolution mass spectrometry (HRMS) analysis. In the model reaction system at the beginning of the reaction. After 3 h, a small amount of reaction mixture was taken for high-resolution mass spectrometry (HRMS) analysis. In the presence of BHT, **4a** was isolated in 15% yield. And in the presence of DPE, **4a** was isolated in 21% yield.



Scheme S1. Mechanism exploration of Synthesis of 3a.







Figure S6. Mass spectrometry (HRMS) data of the radical trapping experiments (with BHT).



Figure S7. Mass spectrometry (HRMS) data of the radical trapping experiments (with DPE).

4.3 Possible reaction mechanism for the formation of 4a'



Figure S8. Possible reaction mechanism for the formation of 4a'

# 5. Characterization data of the products

#### 5.1 Characterization data of the products 3

**3-(phenylselanyl)quinoline (3a)**<sup>[3]</sup>: R<sub>f</sub> = 0.25 (Petroleum ether/EtOAc, 50:1). 75.9 mg, 85% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*) δ 8.91 (s, 1H), 8.25 (s, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.71 (m, 2H), 7.57 – 7.49 (m, 3H), 7.30 (m, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 153.4, 146.7, 139.7, 133.3, 129.9, 129.8, 129.7, 129.3, 128.7, 127.9, 127.3, 127.2, 125.5.

**3-(***o***-tolylselanyl)quinoline (3b)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 60.1 mg, 67% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.86 (s, 1H), 8.14 (s, 1H), 8.08 (d, J = 6.0 Hz, 1H), 7.69 (d, J = 6.0 Hz, 2H),

7.54 (m, 1H), 7.38 (m, 1H), 7.27 (m, 2H), 7.10 (m, 1H), 2.44 (s, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 153.2, 146.7, 140.1, 139.2, 134.1, 130.6, 130.4, 129.7, 129.3, 128.8, 128.45, 127.2, 127.2, 127.1, 125.2, 22.5. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup> : 300.0286, found 300.0281.

**3-**(*p*-tolylselanyl)quinoline (3c)<sup>[4]</sup>:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 71.8 mg, 75%



yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.88 (s, 1H), 8.17 (s, 1H), 8.06 (d, J = 8.6 Hz, 1H), 7.68 (m, 2H), 7.54 –

7.50 (m, 1H), 7.47 – 7.43 (m, 2H), 7.12 (m, 2H), 2.34 (s, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 152.9, 146.6, 138.7. 138.4, 134.0, 130.5, 129.5, 129.3, 128.7, 127.2, 127.2, 126.3, 125.7, 21.2.

**3-((4-(***tert***-butyl)phenyl)selanyl)quinoline (3d)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50: 1). (7.4 mg, 66% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.88 (s, 1H), 8.23 (s, 1H), 8.06 (d, J = 8.3 Hz, 1H), 7.69 (m, 2H), 7.53 (m, 1H), 7.49 – 7.46 (m, 2H), 7.33 (d, J = 7.9 Hz, 2H), 1.31 (s, 9H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  153.3, 151.5, 146.7, 139.1, 133.6, 129.6, 129.4, 128.7, 127.3, 127.2, 126.8, 34.7, 31.3. HRMS (ESI-TOF) m/z: Calcd for C<sub>19</sub>H<sub>20</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 342.0755, found 342.0749.

**3-((2,6-dimethylphenyl)selanyl)quinoline (3e)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). **33.9** mg, 36% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.66 (s, 1H), 8.03 (s, 1H), 7.72 (m, 1H), 7.66 – 7.56 (m, 2H), 7.48 (m, 1H), 7.26 – 7.17 (m, 3H), 2.51 (s, 6H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  150.6, 143.7, 134.4, 129.7, 129.2, 129.1, 128.9, 128.8, 128.3, 127.1, 126.8, 24.4. HRMS (ESI-TOF) m/z: Calcd for C<sub>17</sub>H<sub>16</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 314.0442, found 314.0438.

**3-(mesitylselanyl)quinoline (3f)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 19.8 mg,20% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.66 (s, 1H), 8.01 (s, 1H), 7.69 (m, 1H), 7.64 – 7.55 (m, 2H), 7.49 – 7.44 (m, 1H), 7.03 (s, 2H), 2.47 (s, 6H), 2.33 (s, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  150.7, 143.6, 139.7, 134.1, 129.3, 129.2, 129.0, 128.7, 127.7, 126.9, 126.8, 24.2, 21.1. HRMS (ESI-TOF) m/z: Calcd for C<sub>18</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 328.0599, found 328.0595.

**3-((4-fluorophenyl)selanyl)quinoline (3g)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 73.6 mg, 81% yield. Yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$ 8.88 (s, 1H), 8.18 (s, 1H), 8.07 (m, 1H), 7.69 (m, 2H), 7.53 (m, 3H), 7.01 (m, 2H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  162.9 (d, *J* = 249.5), 152.9, 146.8, 138.9, 135.9, (d, *J* = 8.1), 129.7, 129.4, 128.7, 127.2 ((d, *J* = 2.0), 125.8, 117.0 (d, *J* = 21.2). <sup>19</sup>F NMR (565 MHz, Chloroform-*d*)  $\delta$  -112.9. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>FNSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0035, found 304.0037.

**3-((4-chlorophenyl)selanyl)quinoline (3h)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 58.5 mg, 61% yield. Yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$ 8.90 (s, 1H), 8.25 (s, 1H), 8.08 (d, J = 8.6 Hz, 1H), 7.71 (m, 2H), 7.55 (m, 1H), 7.39 (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.6 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  153.5, 147.0, 140.1, 134.5, 132.7, 130.0, 129.4, 129.1, 128.6, 127.4, 127.3, 124.7, 122.2. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>ClNSe<sup>+</sup> [M + H]<sup>+</sup> : 319.9740, found 319.9733.

**3-((4-bromophenyl)selanyl)quinoline (3i)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 61.4 mg, 55% yield. Yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  $R_f = 8.90$  (s, 1H), 8.24 (s, 1H), 8.08 (d, J = 8.6 Hz, 1H), 7.71 (d, J = 7.8Hz, 2H), 7.55 (m, 1H), 7.41 (d, J = 8.1 Hz, 2H), 7.24 (s, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  153.5, 147.0, 140.0, 134.4, 134.3, 130.0, 129.9, 129.5, 128.7, 128.4, 127.4, 127.4, 124.9. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>BrNSe<sup>+</sup> [M + H]<sup>+</sup> : 363.9235, found 363.9227.

4-(quinolin-3-ylselanyl)benzoate (3j):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 53.4 mg, 52% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d)  $\delta$  8.97 (s, 1H), 8.39 (s, 1H), 8.12 (d, J = 8.5 Hz, 1H), 7.89 (d, J = 8.7 Hz, 2H), 7.77 (d, J = 8.3 Hz, 2H), 7.59 (m,

1H), 7.42 (d, J = 6.3 Hz, 2H), 3.89 (s, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  166.6, 154.4, 147.3, 141.8, 138.0, 130.9, 130.4, 130.3, 129.5, 129.0 128.7, 127.5, 127.4, 123.1, 52.2. HRMS (ESI-TOF) m/z: Calcd for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>Se<sup>+</sup> [M + H]<sup>+</sup>: 344.0184, found 344.0187.

3-((4-(trifluoromethyl)phenyl)selanyl)quinoline (3k):  $R_f = 0.25$  (Petroleum ether/EtOAc, 100:1). N
Se
CF<sub>3</sub>
Chloroform-d)  $\delta$  8.97 (s, 1H), 8.40 (s, 1H), 8.12 (d, J = 8.4 Hz, 1H), 7.79 – 7.58 (m, 3H), 7.56 - 7.49 (m, 4H). 13C NMR (151 MHz, Chloroform-d)  $\delta$  154.3, 147.3, 141.9, 136.3, 131.5, 130.4, 129.5, 129.5 (q, *J* = 33.2), 128.6, 127.6, 127.5, 126.2 (q, *J* = 4.5), 124.0 (q, *J* = 273.3), 123.1. 19F NMR (565 MHz, Chloroform-d)  $\delta$  -62.7. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>11</sub>F<sub>3</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 354.0003, found 353.9998.

**3-(naphthalen-1-ylselanyl)quinoline (3l)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 49.2 mg,



49% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*) δ 8.85 (s, 1H), 8.38 – 8.34 (m, 1H), 8.07 (s, 1H), 8.03 (m, 1H), 7.91 – 7.85 (m, 2H), 7.83 (m, 1H), 7.67 – 7.63 (m, 1H), 7.59 (m, 1H), 7.52

(m, 2H), 7.47 (m, 1H), 7.39 (m, 1H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  152.5, 146.6, 138.2, 134.4, 134.3, 134.0, 129.8, 129.5, 129.3, 128.8, 128.2, 127.4, 127.3, 127.2, 127.1, 126.6, 126.0, 126.0. HRMS (ESI-TOF) m/z: Calcd for C<sub>19</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup> : 336.0286, found 336.0281.

**3-(methylselanyl)quinoline (3m):**  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 60.3 mg, 90% yield. Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  8.92 (s, 1H), 8.14 (s, 1H), 8.07 (d, J = 8.4 Hz, 1H), 7.72 (d, J = 8.1 Hz, 1H), 7.66 (m, 1H), 7.53 (m, 1H), 2.45 (s, 3H).<sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  152.2, 146.4, 136.7, 129.3, 129.2, 128.7, 127.2, 126.9, 126.0, 7.7. HRMS

(ESI-TOF) m/z: Calcd for  $C_{10}H_{10}NSe^+$  [M + H]<sup>+</sup>: 223.9973, found 223.9970.

**3-(benzylselanyl)quinoline (3n):**  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 81.6 mg, 91%.



Orange-yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d) δ 8.86 (s, 1H), 8.10 (s, 1H), 8.06 (d, J = 6, 1H), 7.66 (m, 2H), 7.53 – 7.50 (m, 1H), 7.22 – 7.17 (m, 3H), 7.16 – 7.12 (m, 2H), 4.14 (s, 2H).13C

NMR (151 MHz, Chloroform-d)  $\delta$  154.5, 146.9, 141.2, 138.0, 129.8, 129.3, 129.0, 128.6, 128.4, 127.3, 127.2, 127.1, 123.8, 32.7. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 300.0286, found 300.0285.

6-methyl-3-(phenylselanyl)quinoline (30):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 45.5 mg, 51% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-d) δ 8.84 (s, 1H), 8.16 (s, 1H), 7.97 (s, 1H), 7.58 – 7.43 (m, 4H), 7.29 (m, 3H), 2.51 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-d) δ 152.6, 145.4, 139.2, 137.2, 133.1, 132.1, 129.6, 129.0, 128.8, 127.8, 126.1, 125.3, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 300.0286, found 300.0280.

8-methyl-3-(phenylselanyl)quinoline (3p): R<sub>f</sub> = 0.25 (Petroleum ether/EtOAc, 50:1). 66.9
mg, 75% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-d) δ 8.92 (s, 1H), 8.23 (s, 1H), 7.52 (m, 5H), 7.41 (m, 1H), 7.28 (d, J = 2.4 Hz, 2H), 2.78 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-d) δ 152.4, 139.9, 137.3, 133.2, 129.9, 129.6, 128.7, 127.8, 126.9, 125.4, 125.1, 17.9. HRMS (ESI-TOF)

m/z: Calcd for C<sub>16</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 300.0286, found 300.0291. 6-methoxy-3-(phenylselanyl)quinoline (3q): R<sub>f</sub> = 0.25 (Petroleum ether/EtOAc, 50:1). 77.2

8-methoxy-3-(phenylselanyl)quinoline (3r):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 61.5 mg, 65% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.91 (s, 1H), 8.19 (s, 1H), 7.51 – 7.42 (m, 3H), 7.28 (m, 4H), 7.03 (m, 1H), 4.07 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  155.5, 152.2, 139.3, 133.2, 129.8, 129.6, 127.9, 127.5, 126.3, 119.0, 107.9, 56.0. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>14</sub>NOSe<sup>+</sup> [M + H]<sup>+</sup>: 316.0235, found 316.0238.

6-chloro-3-(phenylselanyl)quinoline (3s):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 36 mg,  $CI \longrightarrow Se_N$  38% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.85 (s, 1H), 8.06 (s, 1H), 7.99 (s, 1H), 7.66 (d, J = 2.3 Hz, 1H), 7.61 (m, 1H), 7.56 – 7.52 (m, 2H), 7.36 – 7.31 (m, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 153.1, 145.1, 137.4, 134.0, 133.0, 131.0, 130.4, 129.8, 129.3, 129.0, 128.4, 127.5, 125.8. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>ClNSe<sup>+</sup> [M + H]<sup>+</sup>: 319.9740, found 319.9734. 7-chloro-3-(phenylselanyl)quinoline (3t):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 48.6 mg,  $CI = \sum_{N=1}^{N} \sum_{n=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{i=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{i=1}^{N}$ 

Calcd for  $C_{15}H_{11}CINSe^+ [M + H]^+$ : 319.9740, found 319.9734.

5-bromo-3-(phenylselanyl)quinoline (3u):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 51.1

Br mg, 47% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$ 8.85 (s, 1H), 8.58 (s, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 7.5 Hz, 1H), 7.59 – 7.55 (m, 2H), 7.52 (m, 1H), 7.33 (m, 3H). <sup>13</sup>C NMR (101

MHz, Chloroform-*d*) δ 153.7, 147.5, 137.9, 133.9, 130.9, 129.8, 129.6, 129.3, 128.4, 128.0, 127.9, 121.2. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>BrNSe<sup>+</sup> [M + H]<sup>+</sup> : 363.9235, found 363.9228.

**7-bromo-3-(phenylselanyl)quinoline (3v)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 64.7 mg, 59% yield. Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.87 (s, 1H), 8.24 (s, 1H), 8.14 (s, 1H), 7.60 (m, 1H), 7.55 – 7.50 (m, 3H), 7.32 (m, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.0, 147.2, 138.7, 133.7, 131.7, 130.7, 129.7, 128.4, 128.3, 127.3, 126.4, 123.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>11</sub>BrNSe<sup>+</sup> [M + H]<sup>+</sup>: 363.9235, found 363.9236.

**3-(phenylselanyl)quinoline-6-carbonitrile (3w)**:  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). NC (10.8 mg, 12% yield.) Orange-yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.96 (s, 1H), 8.16 – 8.09 (m, 2H), 8.06 (s, 1H), 7.80 (s, 1H), 7.62 – 7.57 (m, 2H), 7.38 (m, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  155.2, 147.4, 137.4, 134.6, 133.2, 130.9, 130.0, 129.9, 129.3, 128.9, 128.1, 128.0, 118.3, 111.0. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>11</sub>N<sub>2</sub>Se<sup>+</sup> [M + H]<sup>+</sup>: 311.0082, found 311.0081.

#### 5.2 Characterization data of the products 4

6-(phenylselanyl)-1,2,3,4-tetrahydroquinoline (4a)<sup>[5]</sup>:  $R_f = 0.25$  (Petroleum ether/EtOAc,

50:1). 42.5 mg, 74% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.30 – 7.26 (m, 2H), 7.24 – 7.16 (m, 4H), 7.14 – 7.10 (m, 1H), 6.41 (d, *J* = 8.1 Hz, 1H), 3.31 (t, 2H), 2.73 (t, *J* = 6.3 Hz, 2H),

1.95 – 1.90 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 145.1, 137.3, 134.8, 134.7, 129.7, 128.9, 125.7, 122.4, 114.8, 113.9, 41.8, 26.8, 21.7.

6-(p-tolylselanyl)-1,2,3,4-tetrahydroquinoline (4b):  $R_f = 0.25$  (Petroleum ether/EtOAc,



50:1). 50.1 mg, 83% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.24 – 7.15 (m, 4H), 7.01 (d, *J* = 7.9 Hz, 2H), 6.41 (s, 1H), 3.30 (s, 2H), 2.72 (s, 2H), 2.27 (s, 3H), 1.95 – 1.89 (m, 2H).

<sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 144.6, 136.7, 135.8, 134.2, 130.5, 130.4, 129.8, 122.5, 115.0, 114.9, 41.8, 26.8, 21.7, 20.9. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0599, found 304.0597.

**6-(***m***-tolylselanyl)-1,2,3,4-tetrahydroquinoline (4c):**  $R_f = 0.25$  (Petroleum ether/EtOAc, Se 50:1). 53.2 mg, 88% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.22 – 7.18 (m, 2H), 7.15 (s, 1H), 7.07 (d, *J* = 4.8 Hz, 2H), 6.96 – 6.93 (m, 1H), 6.40 (d, *J* = 8.1 Hz, 1H), 3.31 (t, 2H), 2.73 (t, *J* = 6.3 Hz, 2H), 2.26 (s, 3H), 1.95 – 1.90 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.0, 138.7, 137.1, 134.7, 134.3, 130.5, 128.8, 127.0, 126.7, 122.4, 114.8, 114.1, 41.8, 26.8, 21.7, 21.3. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0599, found 304.0597.

6-(*o*-tolylselanyl)-1,2,3,4-tetrahydroquinoline (4d):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 44.7 mg, 74% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.21 – 7.16 (m, 2H), 7.12 (d, *J* = 7.4 Hz, 1H), 7.08 – 6.95 (m, 3H), 6.45 (d, *J* = 8.1 Hz, 1H), 3.35 – 3.31 (m, 2H), 2.74 (t, *J* =

6.3 Hz, 2H), 2.37 (s, 3H), 1.96 – 1.92 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-d) δ 144.9,

137.5, 136.8, 135.4, 135.1, 129.7, 129.3, 126.4, 125.7, 122.7, 115.1, 113.3, 41.8, 26.8, 21.7, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0599, found 304.0596.

6-((4-(*tert*-butyl)phenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4e): R<sub>f</sub> = 0.25 (Petroleum ether/EtOAc, 50:1). 59.2 mg, 86% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d) δ 7.24 - 7.19 (m, 6H), 6.41 (d, J = 8.0 Hz, 1H), 3.31 (s, 2H), 2.72 (t, J = 5.5 Hz, 2H), 1.95 - 1.89 (m, 2H), 1.27 (s, 9H). <sup>13</sup>C NMR (151 MHz, Chloroform-d) δ 149.0, 144.8, 137.1, 134.6, 130.8, 129.8, 126.0, 122.4, 114.9, 114.5, 41.8, 34.4, 31.3, 26.8, 21.7. HRMS (ESI-TOF) m/z: Calcd for

 $C_{19}H_{24}NSe^+ [M + H]^+: 346.1068$ , found 346.1067.

6-(mesitylselanyl)-1,2,3,4-tetrahydroquinoline (4f):  $R_f = 0.25$  (Petroleum ether/EtOAc,



50:1). 41.6 mg, 63% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  6.93 (s, 2H), 6.85 (s, 1H), 6.76 (d, J = 10.0 Hz, 1H), 6.29 (d, J = 8.1 Hz, 1H), 3.24 (s, 2H), 2.67 – 2.62 (m, 4H), 2.46 (s,

6H), 2.27 (s, 3H), 1.87 (p, J = 6.3 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  143.1, 138.2, 131.7, 129.1, 128.6, 128.5, 122.6, 118.2, 115.2, 41.9, 26.8, 24.4, 21.9, 21.0. HRMS (ESI-TOF) m/z: Calcd for C<sub>18</sub>H<sub>22</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 332.0912, found 332.0909.

6-((4-fluorophenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4g):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 46.4 mg, 76% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d)  $\delta$  7.31 – 7.26 (m, 2H), 7.21 – 7.15 (m, 2H), 6.90 (t, J = 8.6 Hz, 2H), 6.40 (d, J = 7.9 Hz, 1H), 3.31 (s, 2H),

2.72 (s, 2H), 1.95 – 1.90 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  161.7 (d, J = 245.3 Hz), 145.0, 136.8, 134.3, 132.1 (d, J = 7.5 Hz), 128.7 (d, J = 3.1 Hz), 122.4, 116.0 (d, J = 21.5 Hz), 114.9, 114.6, 41.8, 26.8, 21.6. <sup>19</sup>F NMR (565 MHz, Chloroform-*d*)  $\delta$  -116.8. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeF<sup>+</sup> [M + H]<sup>+</sup>: 308.0348, found 308.0346.

6-((4-chlorophenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4h):  $R_f = 0.25$  (Petroleum



ether/EtOAc, 50:1). 43.8mg, 68% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*) δ 7.21 – 7.17 (m, 4H), 7.14 (d, *J* = 8.5 Hz, 2H), 6.41 (d, *J* = 8.1 Hz, 1H), 3.34 – 3.30 (m, 2H), 2.73 (t, *J* = 6.3

Hz, 2H), 1.96 - 1.90 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.2, 137.3, 134.8, 133.0, 131.7, 131.0, 129.0, 122.5, 114.9, 113.6, 41.7, 26.8, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeCl<sup>+</sup> [M + H]<sup>+</sup>: 324.0053, found 324.0049.

6-((3-chlorophenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4i):  $R_f = 0.25$  (Petroleum  $\stackrel{Cl}{\longrightarrow} \stackrel{Se}{\longrightarrow} \stackrel{V}{\longrightarrow}$  ether/EtOAc, 50:1). 43.1mg, 67% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.24 – 7.18 (m, 3H), 7.15 – 7.07 (m, 3H), 6.43 (d, *J* = 8.1 Hz, 1H), 3.35 – 3.32 (m, 2H), 2.74 (t, *J* = 6.3 Hz, 2H), 1.98 – 1.91 (m, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.4, 137.6, 136.9, 135.2, 134.7, 129.8, 129.0, 127.4, 125.7, 122.5, 114.9, 112.8, 41.7, 26.8, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeCl<sup>+</sup> [M + H]<sup>+</sup>: 324.0053, found 324.0049.

6-((2-chlorophenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4j):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 50.2mg, 78% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.28 – 7.21 (m, 3H), 7.06 – 6.98 (m, 2H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.46 (d, *J* = 8.1 Hz, 1H), 3.37 – 3.33 (m, 2H), 2.76 (t, *J* =

6.3 Hz, 2H), 1.95 (p, J = 6.2 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.7, 138.6, 136.2, 136.1, 132.0, 129.0, 129.0, 127.0, 126.2, 122.7, 115.0, 111.7, 41.7, 26.8, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeCl<sup>+</sup> [M + H]<sup>+</sup>: 324.0053, found 324.0048.

6-((4-bromophenyl)selanyl)-1,2,3,4-tetrahydroquinoline (4k):  $R_f = 0.25$  (Petroleum  $R_f = 0.25$  (Petroleum  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 47.6mg, 65% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.28 (d, J = 8.5 Hz, 2H), 7.20 – 7.17 (m, 2H), 7.12 (d, J = 8.5 Hz, 2H), 6.42 (d, J = 8.1 Hz, 1H), 3.35 – 3.29 (m, 2H), 2.73 (t, J = 6.3 Hz, 2H), 1.93 (p, J = 6.3 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.2, 137.3, 134.9, 133.8, 131.9, 131.3, 122.6, 119.6, 114.9, 113.5, 41.7, 26.8, 21.6. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeBr<sup>+</sup> [M + H]<sup>+</sup>: 367.9548, found 367.9545.

5-bromo-6-(phenylselanyl)-1,2,3,4-tetrahydroquinoline (41):  $R_f = 0.25$  (Petroleum ether/EtOAc, 100:1). 5.0 mg, 5% yield. Light-yellow oil. 1H NMR (600 MHz, Chloroform-d)  $\delta$  7.43 (d, J = 6.1 Hz, 2H), 7.26 (m, 3H), 6.97 (d, J = 6.0 Hz, 1H), 6.37 (d, J = 12 Hz,1H), 3.26 (t, J = 6.0 Hz, 2H), 2.81 (t, *J* = 6.0 Hz, 2H), 1.97 (q, *J* = 5.8 Hz, 2H). 13C NMR (151 MHz, Chloroform-d) δ 145.5, 132.9, 132.5, 132.1, 129.3, 127.1, 124.8, 122.4, 119.8, 114.3, 41.2, 29.0, 22.2. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>BrNSe<sup>+</sup> [M + H]<sup>+</sup>: 354.0004, found 353.9998.

7-chloro-6-(phenylselanyl)-1,2,3,4-tetrahydroquinoline (4m):  $R_f = 0.25$  (Petroleum  $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow \uparrow$  ether/EtOAc, 50:1). 39.4 mg, 61% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.34 (d, *J* = 7.2 Hz, 2H), 7.24 – 7.17 (m, 3H), 7.12 (s, 1H), 6.58 (s, 1H), 3.29 (s, 2H), 2.64 (t, *J* = 6.0 Hz, 2H), 1.89 (p, *J* = 6.2 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.9, 137.7, 136.4, 132.7, 130.8, 129.1, 126.4, 121.0, 114.5, 114.3, 41.5, 26.3, 21.4. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeCl<sup>+</sup> [M + H]<sup>+</sup>: 324.0053, found 324.0049.

**7-bromo-6-(phenylselanyl)-1,2,3,4-tetrahydroquinoline** (4n):  $R_f = 0.25$  (Petroleum  $\xrightarrow{Se}_{Br}$  ether/EtOAc, 50:1). 43.2 mg, 59% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.34 (d, *J* = 7.2 Hz, 2H), 7.25 – 7.17 (m, 3H), 7.11 (s, 1H), 6.74 (s, 1H), 3.27 (s, 2H), 2.61 (t, *J* = 5.4 Hz, 2H), 1.87 (p, *J* = 6.2 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  146.0, 137.4, 132.9, 130.9, 129.1, 127.1, 126.5, 121.6, 117.5, 116.7, 41.5, 26.4, 21.4. HRMS (ESI-TOF) m/z: Calcd for C<sub>15</sub>H<sub>15</sub>NSeBr<sup>+</sup> [M + H]<sup>+</sup> : 367.9548, found 367.9543.

8-methyl-6-(phenylselanyl)-1,2,3,4-tetrahydroquinoline (40):  $R_f = 0.25$  (Petroleum ether/EtOAc, 100:1). 24 mg, 26% yield. Light-yellow oil. 1H NMR (600 MHz, Chloroform-d)  $\delta$  7.29 (s, 1H), 7.28 (s, 1H), 7.23 - 7.04 (m, 5H), 3.39 (t, J = 6.4 Hz, 2H), 2.76 (t, J = 6.4 Hz, 2H), 2.05 (s, 3H),

1.93 (q, *J* = 5.6 Hz, 2H). 13C NMR (151 MHz, Chloroform-d) δ 143.2, 135.7, 135.3, 134.9, 129.7, 129.0, 125.7, 122.1, 121.8, 113.3, 42.2, 27.1, 21.8, 17.0. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0599, found 304.0593.

6-(methylselanyl)-1,2,3,4-tetrahydroquinoline (4p): R<sub>f</sub> = 0.25 (Petroleum ether/EtOAc,
Se
100:1). 39 mg, 57% yield. Light-yellow oil. 1H NMR (600 MHz, Chloroform-d) δ 7.12 (d, J = 12 Hz, 2H), 6.38 (s, 1H), 3.28 (t, J = 5.4 Hz, 2H), 2.73 (t, J = 6.4 Hz, 2H), 2.25 (s, 3H), 1.91 (p, J = 6.1 Hz, 2H). 13C NMR (151 MHz, 2H), 2.73 (t, J = 6.4 Hz, 2H), 2.25 (s, 3H), 1.91 (p, J = 6.1 Hz, 2H).

Chloroform-d)  $\delta$  144.1, 134.4, 131.8, 122.3, 116.4, 114.8, 41.9, 26.9, 21.9, 9.4. HRMS (ESI-TOF) m/z: Calcd for C<sub>10</sub>H<sub>14</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 228.0286, found 228.0281.

6-(benzylselanyl)-1,2,3,4-tetrahydroquinoline (4q):  $R_f = 0.25$  (Petroleum ether/EtOAc, 50:1). 49.1 mg, 81% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d)  $\delta$  7.22 (d, J = 7.5 Hz, 2H), 7.19 – 7.12 (m, 3H), 7.06 (d, J = 9.7 Hz, 1H), 7.02 (s, 1H), 6.33 (d, J = 8.2 Hz, 1H), 3.95 (s, 2H), 3.31 – 3.27 (m, 2H), 2.67 (t, J = 6.3 Hz, 2H), 1.90 (p, J = 6.3 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-d)  $\delta$  144.6, 139.5, 136.9, 134.2, 128.8, 128.2, 126.5, 122.0, 114.5, 41.8, 33.5,

26.7, 21.8. HRMS (ESI-TOF) m/z: Calcd for  $C_{16}H_{18}NSe^+$  [M + H]<sup>+</sup> : 304.0599, found 304.0596.

7-chloro-6-(*p*-tolylselanyl)-1,2,3,4-tetrahydroquinoline (4r):  $R_f = 0.25$  (Petroleum  $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow$  ether/EtOAc, 50:1). 42.8 mg, 64% yield. Brown oil. <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  7.28 (d, *J* = 8.0 Hz, 2H), 7.08 – 7.03 (m, 6H), 6.53 (s, 1H), 3.26 (s, 2H), 2.62 (t, *J* = 5.7 Hz, 2H), 2.30 (s, 3H), 1.86 (p, *J* = 6.2 Hz, 2H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*)  $\delta$  145.8, 136.8, 136.6, 135.7, 131.7, 130.0, 128.4, 120.9, 115.1, 114.2, 41.5, 26.3, 21.5, 21.1. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>17</sub>NSeCl<sup>+</sup> [M + H]<sup>+</sup>: 338.0209, found 338.0205.

6-methyl-8-(phenylselanyl)-1,2,3,4-tetrahydroquinoline (4a'):  $R_f = 0.25$  (Petroleum ether/EtOAc, 200:1). 24.8 mg, 27% yield. Light-yellow oil. 1H NMR (600 MHz, Chloroform-d)  $\delta$  7.29 - 7.13 (m, 5H), 7.19 (s, 1H), 6.84 (s, 1H), 3.26 (t, J = 5.5 Hz, 2H), 2.75 (t, J = 6.4 Hz, 2H), 2.18 (s, 3H), 1.88 (p, J =

5.9 Hz, 2H). 13C NMR (151 MHz, Chloroform-d) δ 143.9, 136.5, 132.2, 132.0, 129.2, 129.0, 125.9, 125.6, 121.7, 111.2, 42.2, 27.6, 22.1, 20.1. HRMS (ESI-TOF) m/z: Calcd for C<sub>16</sub>H<sub>18</sub>NSe<sup>+</sup> [M + H]<sup>+</sup>: 304.0599, found 304.0595.

# 6. References

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# 7. NMR of Products

# <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3a



# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3a



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3b



# <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3c



# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3c



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3d



# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3d



210 200 190 180 170 160 150 140 130 120 110 100 90 fl (ppm)\_ 80 70 60 50 40 30

# <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3e



# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3e



# <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3f



190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3g



<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3g



# <sup>19</sup>F-NMR Spectrum (565 MHz, CDCl<sub>3</sub>) of 3g



0	-10	-20	-30	-40	-50	-60	-70	-80	-90	-100	-110	-120	-130	-140	-150	-160	-170	-180	-190	-2
									f	f1 (pp)	n) _									
<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3h



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3h



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3i



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3i



#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3j



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3j





# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3k



# <sup>19</sup>F-NMR Spectrum (565 MHz, CDCl<sub>3</sub>) of 3k



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)

## <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 31



#### <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 31



#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3m



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3m



#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3n



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3n



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 30



<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 30



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3p



10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 f1 (ppm) <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3q



s 46

#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3r



s 47

#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3s



#### <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3s



) 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 fl (nnm) <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3t



# <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3t



<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3u



## <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3u





<sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 3v



#### <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 3v





0 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)\_



<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4a

















<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4d

144, 92 137, 56 137, 56 135, 43 135, 43 135, 43 135, 43 129, 77 129, 35 129, 35 129, 35 125, 72 129, 35 112, 13 13, 13 113, 13  $\sim 26.810$ < 21.704< 21.608- 41.842 7500 CH<sub>3</sub> 6500 Se N 5500 'n 4500 - 3500 2500 1500 500 Ы -500210 190 170 150130 110 90  $\dot{70}$ 50 30 10 -10 fl (ppm)















#### 11 (F

s 59





#### s 61













fl (ppm)



## <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 41

#### <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4l





<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4m







#### <sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 40



#### <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 4p



<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4p





<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4q





<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4r


## <sup>1</sup>H-NMR Spectrum (600 MHz, CDCl<sub>3</sub>) of 4a'



<sup>13</sup>C-NMR Spectrum (151 MHz, CDCl<sub>3</sub>) of 4a'

