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# Solvent free nucleophilic selenocyanation with [bmim][SeCN]. Direct access to perfluoroalkylselenide compounds

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## A B S T R A C T

Available online-1-*n*-butyl-3-methylimidazolium selenocyanate ([bmim][SeCN]) proved to be a very efficient reagent for selenocyanation of alkyl halides. As part of an eco-friendly process, no organic solvents were used during the transformation and reaction times are reduced by using microwave as a heating source. The ionic liquid was carefully recycled in two different ways. Selenocyanate compounds were successfully transformed into trifluoromethylselenides in a one-pot two step procedure.

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### Keywords:

Selenocyanation  
Ionic liquids  
Nucleophilic substitution  
Green chemistry  
Microwaves

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## 1. Introduction

Selenium (Se) is a necessary trace element. In the human body, it mainly exists in the form of selenocysteine and it has crucial antioxidant properties in humans when incorporated into selenoenzymes [1]. The increasing interest for organoselenium compounds stems from their potential biological activities [2]. Among them, organoselenocyanates, common in natural products, are highly interesting targets in the search for anticancer drugs [3]. They are useful synthetic intermediates that participate in a variety of reactions [4]. To get access to aliphatic selenocyanates, nucleophilic displacement of halides is the preponderant route. Commercially available potassium selenocyanates KSeCN is the reagent of choice for such transformation providing polar solvents like DMF or DMSO are used [5]. As an alternative to such solvents, other selenocyanate salts with better solubility were developed as an alternative source of potassium selenocyanate in less polar solvents (AgSeCN, (Me<sub>4</sub>N)SeCN, CsSeCN, ...) [6]. Ionic liquids (ILs)

showed good solubilization performance and are important alternatives to non-ecofriendly solvents. These salts have been the subject of intense research as effective reaction solvents [7]. Recently, as part of our ongoing program to develop ecofriendly nucleophilic substitution processes, we have shown the potential of ILs both as reagent and solvent for nucleophilic substitution [8]. A few ILs with SeCN as anion have already been prepared (e.g. 1-ethyl-3-methylimidazolium selenocyanate [EMIM][SeCN]) and they are mainly used in materials for solar cells or redox-active electrolytes for hybrid electrochemical capacitors, but never as reagents for organic transformations [9].

Herein we report the first synthesis of the ionic liquid, [bmim][SeCN] as a new reagent for nucleophilic selenocyanation. The recyclability of this ionic liquid was carefully examined. Organic selenocyanates may be easily converted to other interesting functional groups and especially SeCF<sub>3</sub>. Given the considerable interest in trifluoromethylselenide compounds [10] due to their promising anti-inflammatory and anticancer properties [11], we show here that it is possible to transform the selenocyanates obtained previously to perfluoroselenolated compounds. We also explore a two-step one-pot sequence to obtain trifluoromethylselenide compounds directly from halides.

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## 2. Results and discussion

In order to access the [bmim][SeCN] reagent, a two-step procedure was developed (Scheme 1). Precursor [bmim][Cl] was first prepared via solvent-free *N*-alkylation under microwave irradiation according to a literature procedure, nevertheless it is also commercially available [12]. The second step, the anion exchange of azolium halides, was carried out with potassium selenocyanate. The anion metathesis was carried out in 1 h in water with a slight excess (1.1 equivalent) of KSeCN at room temperature. After workup, the selenolated IL was isolated with a high yield of 95% without further purification. The reaction could be conducted on large a scale (up to 25 g) with the same result.

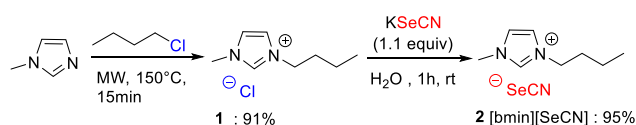
With this new IL reagent in hand, the nucleophilic selenocyanation of 1-bromo-3-phenylpropane was investigated as a model reaction (Table 1). During this study, no particular handling precautions were required. All the reactions described here were conducted under microwave irradiation. Since the dielectric properties of ionic liquids allow them to absorb microwave irradiation very efficiently, they are highly suitable for microwave assisted organic synthesis [13].

Selenocyanation occurred with a good yield of 66% in only 15 min, when 1-bromo-3-phenylpropane was stirred with 1 equivalent of [bmim][SeCN] at 110 °C (entry 1). Increasing the time had no influence on the yield, whereas decreasing the temperature resulted in a slight increase in the yield to 77% (entries 2 and 3). The use of a slight excess of IL (1.2 equiv.) gave complete conversion and the (3-selenocyanatopropyl)benzene was obtained with a good isolated yield, (93%, entry 3). The reaction has been conducted on a 6 mmol (1.2 g) scale with nearly the same yield and to afford 1.16 g of the desired product (entry 4). As a comparison, with conventional heating, a reaction time of 3 h was needed, with 1.5 equivalents of IL to obtain an equivalent yield (entry 5).

Encouraged by these results, we attempted the selenocyanation reaction without solvent on various substrates in order to evaluate the scope and limitations of [bmim][SeCN]. The previously optimized conditions (30 min at 90 °C under microwave irradiation) were successfully applied to a wide range of substrates (Scheme 2).

Primary alkylbromides were displaced with good yields to give rise to the desired molecules (4a-c, Scheme 2). Benzyl bromides were easily substituted by [bmim][SeCN] even on a multigram scale (4d). The functionalization of the aromatic part by fluorinated groups or fluorine atoms was not detrimental to the yields (4e-g). By adjusting the amount of reagent, a bis-substituted compound, known for their antioxidant properties, was easily obtained (4i) [14]. The reaction is slightly sensitive to steric hindrance, nevertheless the isolated yield remained correct (4q-r). This transformation was also compatible with various other functionalities, such as esters (4c, 4j), cyano (4l, 4o), imide (4n), and lactone (4m) groups. With non-activated fatty alkyl halides, good yields were obtained (4o-p).

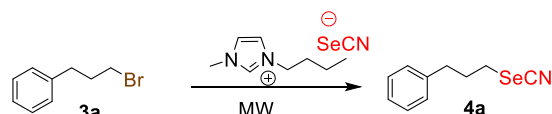
The recycling of the cationic core of our reagent was then carefully examined. After completion of the selenocyanation process, simple extraction of the crude mixture with diethyl ether afforded product 4e with excellent yield and purity (Scheme 3). After dissolution in water, the IL can be regenerated following the



Scheme 1. Synthesis of [bmim][SeCN] 2.

Table 1

Selenocyanation of 1-bromo-3-phenylpropane with [bmim][SeCN].



Entry	Time (min)	Temperature (°C)	[bmim][SeCN] (equiv.)	Yields (%) <sup>a</sup>
1	15	110	1	66
2	30	110	1	66
3	30	90	1	77
3	30	90	1.2	93
4 <sup>b</sup>	30	90	1.2	86
5 <sup>c</sup>	180	90	1.5	88

<sup>a</sup> Isolated yields.

<sup>b</sup> Reaction on 6 mmol, 1.2 g of 3a.

<sup>c</sup> Reaction conducted under conventional heating.

same procedure as for its initial preparation (Path A). Regenerated IL was then re-engaged in the same reaction to give once again nearly the same yield. Alternatively, after the reaction and extraction, the cationic part could also be recovered by a simple anion exchange with LiNTf<sub>2</sub> to ensure total conversion to a unique and useful known IL [bmim][NTf<sub>2</sub>] (Path B, yield up to 93%). This IL could be used later as a reaction media for other organic syntheses [7].

We then decided to transform some of the previously synthesized selenocyanates into trifluoromethylselenolated compounds using classical methodology (Ruppert-Prakash reagent TMSCF<sub>3</sub>, TBAF) (Scheme 4). Pleasingly the reaction proceeded smoothly to give the desired compounds 5 in very good yields. This methodology was extended to the bromodifluoromethyl and pentafluoroethyl groups to give rise to molecule 6k and 7a with good yields.

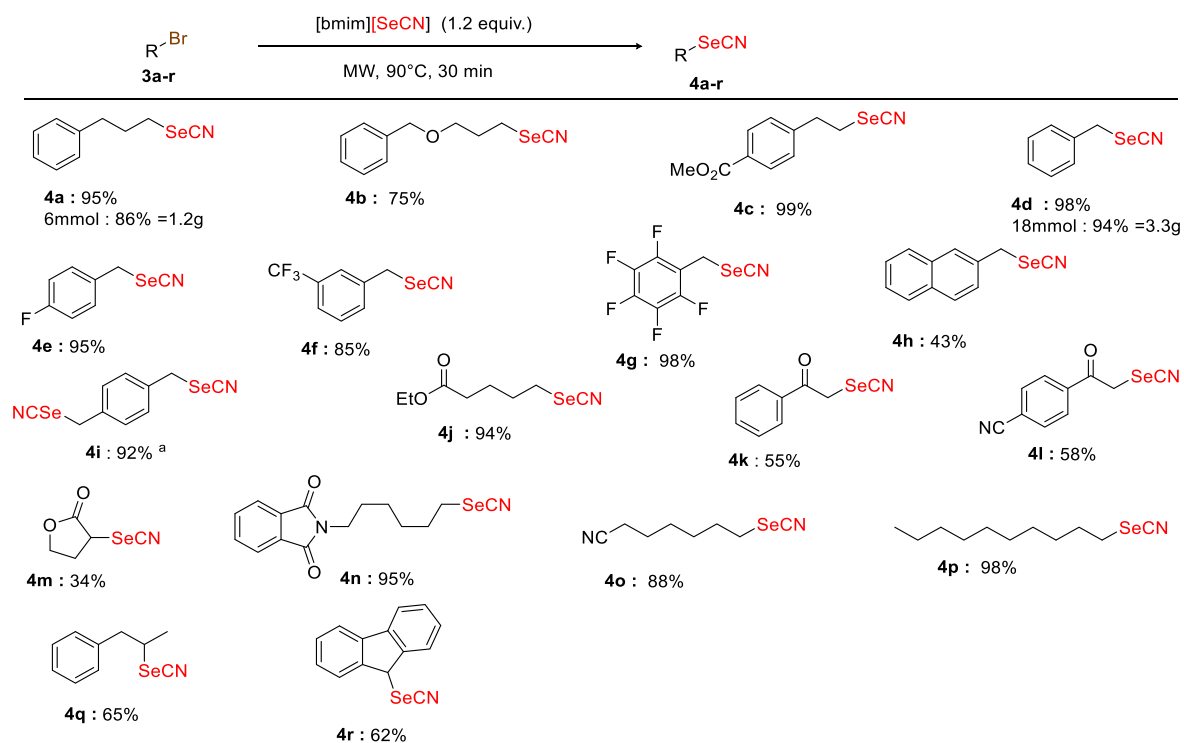
The final approach to get access to trifluoromethylselenolated compounds was to perform the transformation in a two-steps one-pot sequence reaction (Scheme 5). This procedure proved to be very efficient. Indeed, directly after the nucleophilic substitution, THF, Ruppert-Prakash reagent and TBAF were added according to the general procedure to give the desired product in good yield. The result was comparable to the two-step procedure (73% isolated yield for one-pot procedure vs 71% global yield for the two steps).

## 3. Conclusion

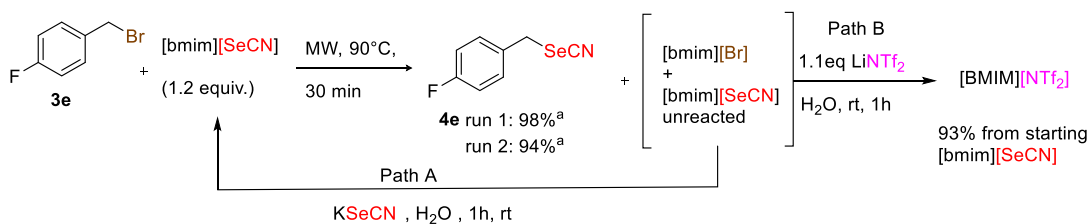
We have shown that 1-*n*-butyl-3-methylimidazolium selenocyanate [bmim][SeCN] is a practical and efficient reagent to perform soft nucleophilic substitution to give selenocyanate compounds. No solvent, special precautions, complex or hazardous additional reagents are necessary for this transformation. The ILs could be entirely recycled with two green and easy different pathways. Trifluoromethylselenolated compounds have been obtained from the selenocyanate derivatives with Ruppert-Prakash reagent, or directly via a two-step one-pot procedure from the corresponding halide. Further developments of this reagent and other applications are under study in our laboratory.

## 4. Experimental

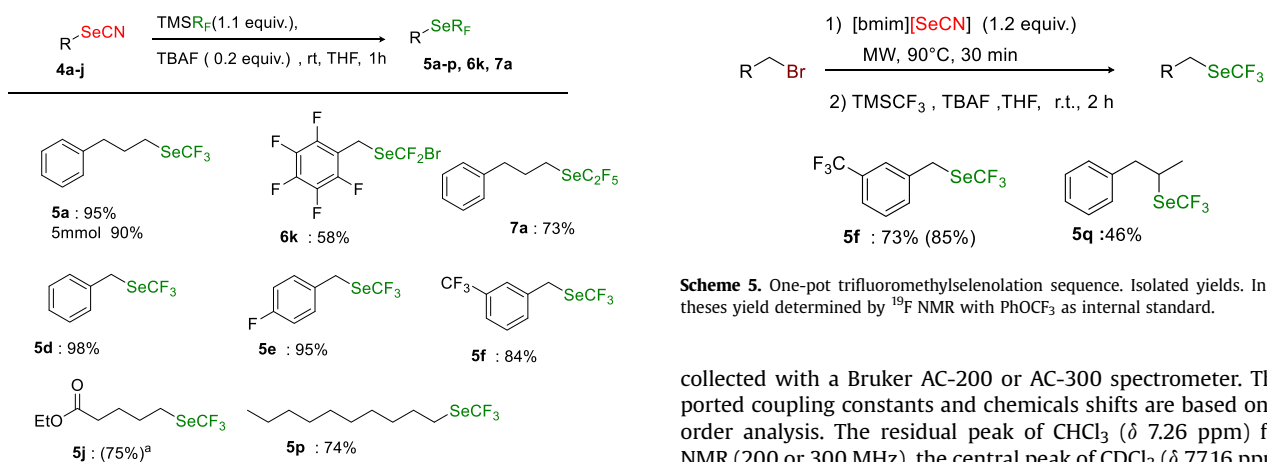
*General.* Chemicals were purchased from commercial sources unless otherwise stated in the Supporting Information. Reactions were monitored by <sup>19</sup>F NMR spectroscopy. Yields refer to materials purified by preparative TLC chromatography. NMR spectra were



**Scheme 2.** Selenocyanation reaction on various substrates with [bmim][SeCN].<sup>a</sup> with 2.2 equivalent of IL.



**Scheme 3.** Recycling of the IL.<sup>a</sup> yield determined by <sup>19</sup>F NMR with PhOCF<sub>3</sub> as internal standard.



**Scheme 5.** One-pot trifluoromethylselenolation sequence. Isolated yields. In parentheses yield determined by <sup>19</sup>F NMR with PhOCF<sub>3</sub> as internal standard.

**Scheme 4.** Perfluoroalkylation of selenocyanide.<sup>a</sup> In parentheses yield determined by <sup>19</sup>F NMR with PhOCF<sub>3</sub> as internal standard.

collected with a Bruker AC-200 or AC-300 spectrometer. The reported coupling constants and chemical shifts are based on first-order analysis. The residual peak of CHCl<sub>3</sub> ( $\delta$  7.26 ppm) for <sup>1</sup>H NMR (200 or 300 MHz), the central peak of CDCl<sub>3</sub> ( $\delta$  77.16 ppm) for <sup>13</sup>C NMR (75 or 50 MHz), and CFCl<sub>3</sub> ( $\delta$  0.00 ppm) for <sup>19</sup>F NMR (188 MHz) were used as internal references. Multiplicities are indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), p (pintuplet), m (multiplet), br (broad), or a combination of these. All

coupling constants were reported in Hz. HRMS was performed with a XEVO-QTOF Mass Spectrometer in ESI(+) and ASAP(+) mode at the Institute Lavoisier of Versailles– University of Versailles Saint Quentin.

#### 4.1. Synthesis of [bmim][SeCN] (2)

[bmim][Cl] (3.5 g, 0.02 mol) was dissolved in a minimum of water. In another flask KSeCN (1.1 eq, 3.168 g, 0.022 mol) was also dissolved in a minimum amount of water. Both solutions were mixed at room temperature for 1 h ensuring a complete anion exchange. At the end of the reaction the solution was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 20\text{ml}$ ) and dried over  $\text{MgSO}_4$ , and concentrated in vacuo to give the desired IL as a slightly viscous yellow liquid. Yield: 4.636 g, 95%;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  9.31 (s, 1H), 7.54 (s, 1H), 7.50 (s, 1H), 4.31 (t,  $J = 7.3$  Hz, 2H), 4.09 (s, 3H), 1.91 (p,  $J = 7.4$  Hz, 2H), 1.38 (h,  $J = 7.4$  Hz, 2H), 0.95 (t,  $J = 7.3$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta = 136.52, 123.86, 122.45, 118.17, 50.11, 36.85, 32.05, 19.44, 13.42$ .  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta = -240.38$ .

#### 4.2. Typical procedure for the selenolacyanation of alkyl halides

A microwave tube with a magnetic stirring bar was charged with a halide (1.0 equiv., 1 mmol) and [bmim][SeCN] (1.2 equiv., 1.2 mmol, 292 mg). After stirring the mixture was subjected to microwave irradiation at  $90^\circ\text{C}$  for 30 min. The mixture was then cooled to room temperature, and the compound was extracted with diethyl ether ( $5 \times 5$  mL). Organic layers were combined and concentrated under reduced pressure. The product was purified by preparative thin layer chromatography (PTLC) on silica gel to give the desired product. On larger scales, the product was purified by chromatography on silica gel to afford 4.

#### 4.3. Synthesis of (3-selenocyanatopropyl)benzene (4a)

Yield: 213 mg, 95%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30 (m, 5H), 3.07 (t,  $J = 7.3$  Hz, 2H), 2.83 (t,  $J = 7.3$  Hz, 2H), 2.29 (p,  $J = 7.3$  Hz, 1H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  139.93, 128.72, 128.55, 126.50, 101.48, 34.95, 32.22, 28.73.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  206.95. HRMS: TOF MS ASAP + calcd. for  $\text{C}_{20}\text{H}_{23}\text{N}_2\text{Se}_2$  451.0192; found 451.0187 [2 M + H]<sup>+</sup> ( $\delta = -1.1$  ppm)

#### 4.4. Synthesis of ((3-selenocyanatopropoxy)methyl)benzene (4b)

Yield: 191 mg, 75%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54–7.08 (m, 5H), 3.60 (t,  $J = 5.5$  Hz, 2H), 2.97 (t,  $J = 6.5$  Hz, 1H), 2.25–2.12 (p,  $J = 5.9$  Hz, 1H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  137.80, 128.54, 127.92, 127.86, 102.86, 73.24, 68.47, 30.35, 27.18.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  202.00. HRMS: TOF MS ES + calcd. for  $\text{C}_{11}\text{H}_{14}\text{NOSe}$  256.0241; found 256.0247 [M + H]<sup>+</sup> ( $\delta = 2.3$  ppm)

#### 4.5. Synthesis of methyl 4-(2-selenocyanatoethyl)benzoate (4c)

Yield: 192 mg, 98%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54–7.08 (m, 4H), 4.27 (s, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  135.58, 128.98, 128.89, 128.52, 102.11, 32.69.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  346.90. HRMS: TOF MS ES + calcd. for  $\text{C}_{11}\text{H}_{12}\text{NO}_2\text{Se}$  270.0033; found 270.0031 [M + H]<sup>+</sup> ( $\delta = -0.7$  ppm)

#### 4.6. Synthesis of (selenocyanatomethyl)benzene (4d)

Yield: 192 mg, 98%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54–7.08 (m, 4H), 4.27 (s, 2H).  $^{13}\text{C}$  NMR

(75 MHz,  $\text{CDCl}_3$ )  $\delta$  135.58, 128.98, 128.89, 128.52, 102.11, 32.69.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  346.90.

#### 4.7. Synthesis of 1-fluoro-4-(selenocyanatomethyl)benzene (4e)

Yield: 203 mg, 95%; Eluent for the preparative TLC chromatography:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36–7.32 (m, 2H), 7.05 (t,  $J = 8.4$  Hz, 2H), 4.26 (s, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  162.76 (d,  $J = 248.5$  Hz), 131.55 (d,  $J = 3.3$  Hz), 130.87 (d,  $J = 8.4$  Hz), 116.19 (d,  $J = 21.8$  Hz), 101.87, 31.96.  $^{19}\text{F}$  NMR (188 MHz,  $\text{CDCl}_3$ )  $\delta = -112.87$  (m, 1F)  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  293.15. HRMS: TOF MS ASAP + calcd. for  $\text{C}_{16}\text{H}_{13}\text{N}_2\text{Se}_2\text{F}_2$  430.9377; found 430.9388 [2 M + H]<sup>+</sup> ( $\delta = 2.6$  ppm)

#### 4.8. Synthesis of 1-(selenocyanatomethyl)-3-(trifluoromethyl)benzene (4f)

Yield: 224 mg, 85%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62–7.48 (m, 4H), 4.30 (s, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  136.81 (s), 132.36 (d,  $J = 0.8$  Hz), 131.54 (q,  $J = 32.7$  Hz), 125.72 (q,  $J = 3.8$  Hz), 125.52 (q,  $J = 3.7$  Hz), 123.70 (q,  $J = 272.3$  Hz), 101.30 (s), 31.66 (s).  $^{19}\text{F}$  NMR (188 MHz,  $\text{CDCl}_3$ )  $\delta = -63.28$  (s, 3F)  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  298.22. HRMS: TOF MS ASAP + calcd. for  $\text{C}_9\text{H}_7\text{NF}_3\text{Se}$  265.9696; found 265.9688 [M + H]<sup>+</sup> ( $\delta = -3.0$  ppm)

#### 4.9. Synthesis of 1,2,3,4,5-pentafluoro-6-(selenocyanatomethyl)benzene (4g)

Yield: 281 mg, 98%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.28 (s, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$   $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  146.85–146.49 (m), 143.52–143.16 (m), 140.11–139.19 (m), 136.32–135.84 (m), 110.59 (dt,  $J = 17.0, 4.1$  Hz), 99.85, 17.34.  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )  $\delta = -141.37$  (dd,  $J = 21.0, 7.4$  Hz),  $-152.53$  (t,  $J = 20.8$  Hz),  $-160.96$  to  $-160.78$  (qd,  $J = 10.3, 3.6$  Hz).  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  304.83. HRMS: TOF MS ASAP + calcd. for  $\text{C}_8\text{H}_3\text{NF}_5\text{Se}$  287.9351; found 287.9352 [M + H]<sup>+</sup> ( $\delta = 0.3$  ppm)

#### 4.10. Synthesis of 2-(selenocyanatomethyl)naphthalene (4h)

Yield: 105 mg, 43%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (2:8).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91–7.77 (m, 4H), 7.64–7.39 (m, 3H), 4.48 (s, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  133.24, 133.12, 132.67, 129.25, 128.25, 128.00, 127.82, 126.82, 126.79, 126.30, 101.88, 33.37.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  280.27. HRMS: TOF MS ASAP + calcd. for  $\text{C}_{24}\text{H}_{19}\text{N}_2\text{Se}_2$  494.9879; found 494.9890 [2 M + H]<sup>+</sup> ( $\delta = 2.2$  ppm)

#### 4.11. Synthesis of 1,4-bis(selenocyanatomethyl)benzene (4i)

Yield: 0.5 mmol, 144 mg, 92%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (3:7).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39 (s, 4H), 4.29 (s, 4H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  136.35, 129.40, 116.45, 101.80, 31.85.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  302.29. HRMS: TOF MS ASAP + calcd. for  $\text{C}_{10}\text{H}_9\text{N}_2\text{Se}_2$  316.9096; found 316.9088 [M + H]<sup>+</sup> ( $\delta = -2.5$  ppm)

#### 4.12. Synthesis of ethyl 5-selenocyanatopentanoate (4j)

Yield: 219 mg, 92%; Eluent for PTLC:  $\text{Et}_2\text{O}$ /petroleum ether (4:6).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.12 (q,  $J = 7.1$  Hz, 2H), 3.04 (t,  $J = 7.2$  Hz, 2H), 2.34 (t,  $J = 7.1$  Hz, 2H), 1.93 (p,  $J = 7.2$  Hz, 2H), 1.76 (p,  $J = 7.2$  Hz, 2H), 1.24 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  172.98, 101.42, 60.59, 33.40, 30.28, 29.08, 24.35, 14.28.  $^{77}\text{Se}$  NMR (57 MHz,  $\text{CDCl}_3$ )  $\delta$  209.28. HRMS: TOF MS ES + calcd. for

C<sub>8</sub>H<sub>14</sub>NO<sub>2</sub>Se 236.0190; found 236.0181 [M+ H]<sup>+</sup> ( $\delta = -3.8$  ppm)

#### 4.13. Synthesis of 1-phenyl-2-selenocyanatoethan-1-one (4k)

Yield: 123 mg, 55%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (300 MHz, CD<sub>3</sub>CN)  $\delta$  8.02 (d, *J* = 8.0 Hz, 2H), 7.72 (t, *J* = 7.0 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 2H), 4.94 (s, 2H). <sup>13</sup>C NMR (75 MHz, CD<sub>3</sub>CN)  $\delta$  134.08, 133.88, 128.61, 128.38, 116.99, 101.65, 37.06. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  160.57. HRMS: TOF MS ASAP + calcd. for C<sub>9</sub>H<sub>8</sub>NOSe 225.9771; found 225.9761 [M+ H]<sup>+</sup> ( $\delta = -4.4$  ppm)

#### 4.14. Synthesis of 4-(2-selenocyanatoacetyl)benzotrile (4l)

Yield: 144 mg, 58%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>)  $\delta$  8.08 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 1H), 4.88 (s, 1H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  138.28, 133.83, 133.63, 130.12, 126.33, 102.57, 37.43. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  172.96. HRMS: TOF MS ASAP + calcd. for C<sub>10</sub>H<sub>7</sub>N<sub>2</sub>OSe 250.9724; found 250.9717 [M+H]<sup>+</sup> ( $\delta = -2.8$  ppm)

#### 4.15. Synthesis of 3-selenocyanatodihydrofuran-2(3H)-one (4m)

Yield: 64 mg, 34%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>)  $\delta$  4.61–4.55 (m, 1H), 4.47–4.30 (m, 2H), 3.03–2.89 (m, 1H), 2.74–2.62 (m, 5H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  173.00, 99.33, 67.62, 37.39, 31.40. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  315.46.

#### 4.16. Synthesis of 2-(6-selenocyanatohexyl)isoindoline-1,3-dione (4n)

Yield: 0.5 mmol, 159.6 mg, 95%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.83–7.80 (m, 2H), 7.73–7.70 (m, 2H), 3.67 (t, *J* = 7.0 Hz, 2H), 3.04 (t, *J* = 7.3 Hz, 2H), 1.89 (p, *J* = 6.9 Hz, 1H), 1.69 (p, *J* = 6.9 Hz, 1H), 1.57–1.29 (m, 4H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  168.39, 133.96, 132.06, 123.18, 101.59, 37.66, 30.67, 29.46, 28.56, 28.33, 26.03. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  208.19.

#### 4.17. Synthesis of 7-selenocyanatoheptanenitrile (4o)

Yield: 190 mg, 88%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (5:5). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  3.04 (t, *J* = 7.3 Hz, 2H), 2.36 (t, *J* = 6.9 Hz, 2H), 1.93 (p, *J* = 6.9 Hz, 2H), 1.68 (p, *J* = 6.9 Hz, 2H), 1.60–1.44 (m, 4H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  119.63, 101.50, 30.59, 29.26, 28.29, 27.95, 25.17, 17.15. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  208.96. HRMS: TOF MS ASAP + calcd. for C<sub>8</sub>H<sub>13</sub>N<sub>2</sub>Se 217.0244; found 217.0235 [M+H]<sup>+</sup> ( $\delta = -4.1$  ppm)

#### 4.18. Synthesis of 1-selenocyanatodecane (4p)

Yield: 209 mg, 98%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  3.09 (t, *J* = 7.4 Hz, 2H), 1.94 (p, *J* = 7.3 Hz, 2H), 1.53–1.40 (m, 2H), 1.31 (s, 12H), 0.92 (t, *J* = 5.9 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  101.71, 32.00, 30.95, 29.83, 29.61, 29.52, 29.39, 29.27, 28.98, 22.80, 14.24. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  206.25. HRMS: TOF MS ASAP + calcd. for C<sub>11</sub>H<sub>22</sub>NSe 248.0917; found 248.0915 [M+H]<sup>+</sup> ( $\delta = -0.8$  ppm)

#### 4.19. Synthesis of (2-selenocyanatopropyl)benzene (4q)

Yield: 209 mg, 98%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.37–7.01 (m, 5H), 3.68 (h, *J* = 7.1 Hz,

1H), 3.13–2.93 (m, 1H), 1.62 (d, *J* = 6.7 Hz, 1H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  137.92, 129.13, 128.91, 127.46, 101.50, 44.90, 44.42, 22.88. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  310.75. HRMS: TOF MS ASAP + calcd. for C<sub>10</sub>H<sub>12</sub>NSe 226.0135; found 226.0134 [M+H]<sup>+</sup> ( $\delta = -0.4$  ppm)

#### 4.20. Synthesis of 9-selenocyanato-9H-fluorene (4r)

Yield: 209 mg, 98%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (2:8). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (t, *J* = 6.8 Hz, 4H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 5.75 (s, 1H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  141.99, 140.38, 129.65, 128.27, 125.54, 120.79, 102.17, 45.66. <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  357.61. HRMS: TOF MS ASAP + calcd. for C<sub>28</sub>H<sub>19</sub>N<sub>2</sub>Se<sub>2</sub> 542.9879; found 542.9890 [2 M + H]<sup>+</sup> ( $\delta = 2.0$  ppm)

#### 4.21. Typical procedure for the perfluoroalkylation of selenocyanate compounds

To a dry round-bottom flask equipped with a magnetic stirrer were added selenocyanate (0.5 mmol, 1.0 equiv) and dry THF (0.5 mL). The flask was evacuated and refilled with argon three times, and TMSCF<sub>3</sub> (88  $\mu$ l, 0.6 mmol, 1.2 equiv) was added. The reaction mixture was cooled to 0 °C, and TBAF in 1 M THF (0.1 mL, 0.1 mmol, 0.2 equiv) was added dropwise. After 10 min at 0 °C, the reaction was allowed to warm to 23 °C and stirred for 2 h. The conversion was checked by <sup>19</sup>F NMR with PhOCF<sub>3</sub> as internal standard. The reaction mixture was then partitioned between water and pentane, and the aqueous layer was extracted with pentane, dried over MgSO<sub>4</sub>, filtered and concentrated to dryness (under moderate vacuum). The crude residue was purified by PTLC:

#### 4.22. Synthesis of (3-phenylpropyl)(trifluoromethyl)selane (5a)

Yield: 0.5 mmol, 126.8 mg, 95%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.37–7.22 (m, 5H), 3.03 (t, *J* = 7.2 Hz, 2H), 2.79 (t, *J* = 7.4 Hz, 2H), 2.18 (t, *J* = 7.4 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  140.64, 128.69, 128.61, 126.38, 122.79 (q, *J* = 330.3 Hz), 35.58, 31.92, 25.26. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>)  $\delta$  -34.51 (s, 3F). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  443.20 (q, *J* = 9.6 Hz). HRMS: TOF MS ASAP + calcd. for C<sub>10</sub>H<sub>11</sub>F<sub>3</sub>Se 267.9978; found 267.9974 [M +] ( $\delta = -1.5$  ppm)

#### 4.23. Synthesis of benzyl(trifluoromethyl)selane (5d)

<sup>1</sup>H NMR (300 MHz, Chloroform-*d*)  $\delta$  7.37–7.27 (massif, 5H), 4.24 (s, 2H). <sup>19</sup>F NMR (282 MHz, CDCl<sub>3</sub>)  $\delta$  -34.46 (s, 3F). Characterization data match that reported in the literature [15].

#### 4.24. Synthesis of (4-fluorobenzyl)(trifluoromethyl)selane (5e)

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.31 (m, 2H), 7.02 (m, 2H), 4.22 (s, 2H). <sup>19</sup>F NMR (282 MHz, CDCl<sub>3</sub>)  $\delta$  -34.38 (s, 3F), -114.11 (m, 1H). Characterization data match that reported in the literature [15].

#### 4.25. Synthesis of (trifluoromethyl)(3-(trifluoromethyl)benzyl)selane (5f)

Yield: 0.5 mmol, 129.4 mg, 95%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.61–7.45 (m, 4H), 4.28 (s, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  137.48 (s), 132.35 (s), 131.31 (q, *J* = 32.4 Hz), 129.42 (s), 125.75 (q, *J* = 3.7 Hz), 124.65 (q, *J* = 3.8 Hz), 123.83 (q, *J* = 272.4 Hz), 122.61 (q, *J* = 331.4 Hz), 28.33 (q, *J* = 1.6 Hz). <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>)  $\delta$  -34.81 (s, 3F), -63.28 (s, 3F). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>)  $\delta$  514.50 (q, *J* = 11.2 Hz).

#### 4.26. Synthesis of decyl(trifluoromethyl)selane (5p)

Yield: 0.5 mmol, 107.3 mg, 74%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 2.91 (t, *J* = 7.3 Hz, 2H), 1.72 (p, *J* = 7.2 Hz, 2H), 1.41–1.11 (m, 14H), 0.81 (t, *J* = 5.7 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 122.89 (q, *J* = 330.3 Hz), 32.03, 30.39, 29.73, 29.66, 29.58, 29.43, 29.09, 26.03, 22.83, 14.25. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) δ –34.77 (s, 3F). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>) δ 444.36 (q, *J* = 10.0 Hz). HRMS: TOF MS ASAP + calcd. for C<sub>11</sub>H<sub>21</sub>F<sub>3</sub>Se 291.0761; found 291.0762 [M +.] (δ = 0.3 ppm)

#### 4.27. Synthesis of (1-phenylpropan-2-yl)(trifluoromethyl)selane (5q)

Yield: 2.5 mmol, 307 mg, 46%; Eluent for column chromatography: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.43–7.19 (m, 5H), 3.82 (s, *J* = 6.9 Hz, 1H), 3.23 (dd, *J* = 13.8, 5.7 Hz, 1H), 2.97 (dd, *J* = 13.7, 8.7 Hz, 1H), 1.56 (d, *J* = 6.9 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 138.48, 129.29, 128.68, 127.06, 123.32 (q, *J* = 330.8 Hz), 77.58, 77.16, 76.74, 44.18, 40.24, 21.85. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) δ –32.72 (s, 3F). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>) δ 543.36 (q, *J* = 10.6 Hz).

#### 4.28. Synthesis of (bromodifluoromethyl)((perfluorophenyl)methyl)selane (6k)

Yield: 0.5 mmol, 112.8 mg, 58%; Eluent for PTLC: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 4.25 (s, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 147.61–145.89 (m), 144.42–143.10 (m), 144.93–142.65 (m), 139.7–139.2 (m), 136.32–135.89 (m), 112.12–111.66 (m), 107.06 (t, *J* = 355.4 Hz), 17.1. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) δ –18.79 (s), –141.82 to –142.25 (m), –154.71 (t, *J* = 20.8 Hz), –161.78 to –162.06 (m). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>) δ 673.5 (m). HRMS: TOF MS ASAP + calcd. for C<sub>8</sub>H<sub>2</sub>F<sub>2</sub>SeBr 389.8393; found 389.8386 [M +.] (δ = –1.8 ppm)

#### 4.29. Synthesis of (3-phenylpropyl)(pentafluoroethyl)selane (7a)

Yield: 0.5 mmol, 115.7, 73%; Eluent PTLC: Et<sub>2</sub>O/petroleum ether (5:95). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.32–6.97 (m, 5H), 2.87 (t, *J* = 7.3 Hz, 2H), 2.61 (t, *J* = 7.5 Hz, 2H), 1.98 (p, *J* = 7.3 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 140.58, 128.71, 128.60, 126.42, 119.02 (qt, *J* = 284.5, 35.0 Hz), 116.33 (tq, *J* = 302.2, 42.2 Hz) 35.60, 31.95, 24.55. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) δ –88.26 (m, 3F), –91.95 (m, 2F). <sup>77</sup>Se NMR (57 MHz, CDCl<sub>3</sub>) δ 390.44 (m).

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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