New and Efficient Chiral Selenium Electrophiles

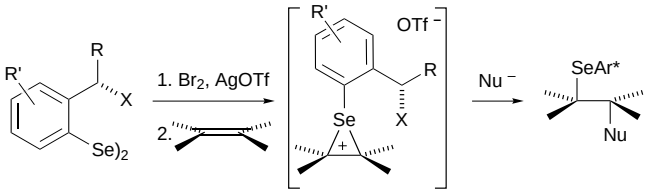
Lars Uehlin,[a, b] Gianfranco Fragale,[a] and Thomas Wirth*[a, b]

Abstract: New chiral diselenides were prepared in a few steps from readily available starting materials. The selenium electrophiles generated from these diselenides were used for the efficient stereoselective inter- and intramolecular functionalization of alkenes. The substitution pattern influences the stereoselectivities and protection of the hydroxy moiety in the chiral side chain led to increased selectivities and yields in the selenenylation reactions. An additional substituent in the second *ortho* position was advantageous as well. Addition products with up to 96% *de* were obtained. The influence of the nucleophile on the outcome of selenenylations of alkenes was studied to some extent as well.

Keywords: alkenes · electrophiles · heterocycles · selenium · stereoselective synthesis

Introduction

Electrophilic selenenylation reactions of alkenes have been successfully applied in the functionalization of inactivated carbon–carbon double bonds.[1] Stereoselective functionalization of alkenes with chiral selenium electrophiles have been performed by us and by other research groups.[2, 3] We have shown that optically active diselenides of type **1** are very easily accessible (Scheme 1). The selenium electrophiles generated



Scheme 1. Selenenylation of alkenes.

from these diselenides can add with high selectivities to alkenes, and a variety of nucleophiles can be used to open the seleniranium intermediates **2**. The addition products **3** can be used for a variety of subsequent reactions.

Herein we report the synthesis of new optically active diselenides and their addition reactions to alkenes. Various alkenes were used in these addition reactions. As a common test reaction we studied the methoxyselenenylation of styrene with all selenium electrophiles reported herein. With some optimized reagents we also performed selenenylations of substituted alkenes and investigated selenocyclizations of unsaturated alcohols and unsaturated carboxylic acids. The selectivities of the reactions described were all determined from NMR spectra of the crude reaction mixtures and HPLC of radical cleavage products, as described earlier.[4]

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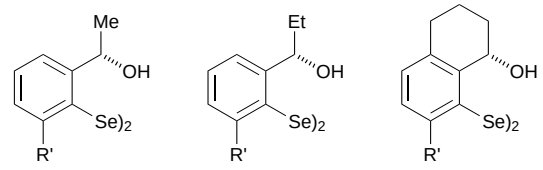
Results and Discussion

We have previously developed short and efficient syntheses for chiral diselenides of type **1** by various routes.[4] Recently we developed substituted reagents that are more efficient in selenenylation reactions than the unsubstituted selenium



1a (R' = H)
1b (R' = OMe)

1c (R' = H)
1d (R' = OMe)

1e (R' = H)
1f (R' = OMe)

electrophiles.[5] They were synthesized from the corresponding alcohols by *ortho*-lithiation and addition of elemental selenium, followed by oxidative work-up. The chiral alcohol precursors are available by chiral reduction with (−)-*B*-chlorodiisopinocampheylborane [(−)-(Ipc)$_2$BCl] (**1a–1d**), commercially (**1e**), or by enzymatic racemic resolution (**1f**). In Table 1 the results of the methoxyselenenylation of these

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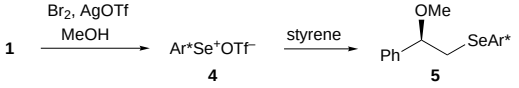
Table 1. Methoxyselenenylation of styrene.



Entry	Diselenide	d.r. of **5** (*S*,*R*):(*S*,*S*)	Yield of **5** [%]
1[a]	**1a** (R′ = H)	91.5:8.5	67
2	**1b** (R′ = OMe)	98:2	55
3[a]	**1c** (R′ = H)	94.5:5.5	81
4	**1d** (R′ = OMe)	94:6	60
5[a]	**1e** (R′ = H)	96.5:3.5	28
6	**1f** (R′ = OMe)	94.5:5.5	38

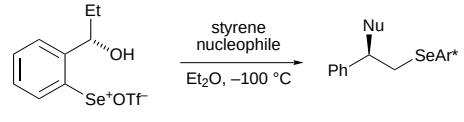
[a] See reference [4].

reagents with R′ = OMe are compared with the corresponding unsubstituted reagents (R′ = H) described previously.[4]

As shown in Table 1, the reagents with the methoxy group in the *ortho* position to the selenium electrophile do show slightly higher selectivities than the unsubstituted reagents, except the tetralol derived reagents **1e** and **1f**. This might result from a different coordination of the side chain oxygen atom to the selenium. However, detailed calculations[6] of the corresponding rotation profiles of the electrophiles generated from **1b** and **1c** showed that the strength of the coordination does not correlate with the trend of the stereoselectivities observed in the experiments described in Table 1.

As already previously demonstrated, the stereoselective step in the overall reaction sequence is the formation of seleniranium ions of type **2**.[7] These intermediates are then selectively opened from the *anti* side by a variety of nucleophiles, yielding the addition products in almost similar diastereomeric excesses. There are, however, slight differences in selectivity. An explanation for this might be that the coordination of different nucleophiles to the selenium electrophile leads to slightly modified reactivities and selectivities. The selenenylation of styrene with methanol as nucleophile (Table 2, entry 1) is more selective (d.r. 94.5:5.5) than with acetic acid (Table 2, entry 5: d.r. 88:12) and the yield is much lower as well. More convincing evidence for such an interaction between the nucleophile and the selenium electrophile is presented later, in the selenocyclizations studied with various electrophiles under different reaction conditions (see Scheme 3). In Table 2 the results of the reaction using styrene and the arylselenenyl triflate reagent **4c** (generated from **1c**) with different nucleophiles are summarized.

Table 2. Stereoselective selenenylation of styrene with different nucleophiles using **4c** as the selenium electrophile.



Entry	Nucleophile	Product	d.r. of **6** (*S*,*R*):(*S*,*S*)	Yield of **6** [%]
1	MeOH	**6a** (=**5c**)	94.5:5.5	81
2	EtOH	**6b**	92.5:7.5	58
3	*i*PrOH	**6c**	90.5:9.5	63
4	$PhCH_2OH$	**6d**	91:9	64
5	CH_3CO_2H	**6e**	88:12	35
6	PhOH	–	–	0
7	MeOH (1 equiv)	**6a** +	95.5:4.5	17
	$PhCH_2OH$ (10 equiv)	**6d**	91:9	56
8	MeOH (10 equiv)	**6a** +	93.5:6.5	64
	$PhCH_2OH$ (1 equiv)	**6d**	–	traces

When reagent **4c** was used, neither phenol (Table 2, entry 6) nor nitrogen nucleophiles (acetonitrile, *N*-BOC-aniline, *N*-BOC-benzylamine; BOC = buyloxycarbonyl) were nucleophilic enough to lead to isolable addition products. However, we have previously shown that BOC-protected amines can be used quite efficiently as nucleophiles in intramolecular selenenylation reactions.[8] If mixtures of two nucleophiles are employed in the selenenylation of styrene, the ratio of products obtained can be explained by the different nucleophilicity of the two nucleophiles (Table 2, entries 7 and 8). Even with a tenfold excess of the less reactive nucleophile (benzylic alcohol) the addition product with methanol was still formed in 17% yield. With only one equivalent of benzylic alcohol and ten equivalents of methanol, only traces of addition product **6d** were detected. The selectivities observed in these experiments do correlate with those from the experiments in which only a single nucleophile was used.

The yields reported in Tables 1 and 2 are, however, only moderate. This might be attributed to the unprotected hydroxy moiety in the chiral side chain of **4c**, which leads to a strong coordination to the electrophilic selenium atom and therefore lowers its reactivity towards alkenes. From earlier experiments we know that a substituent on the oxygen atom influences the reaction[4] and we were pleased to find that methoxymethyl (MOM)-protected reagents lead to higher yields and sometimes even higher selectivities.[9] The corresponding diselenides can easily be prepared, either from chiral alcohols like **7**, by protecting the hydroxy group first and then synthesizing the diselenide, or directly from the diselenide **1f** as shown in Scheme 2. Diselenides **1g**–**1k** were synthesized.

These diselenides were subsequently used in stereoselective selenenylation reactions with alkenes. The selectivities in the methoxyselenenylation of styrene were increased as well, and the results using MOM-protected selenium electrophiles are shown in Table 3. An octyl moiety on the acetal protecting group as in **1h** (Table 3, entry 2) or a distance of two carbon atoms between the two oxygen atoms (**1i**) (Table 3, entry 3) led to lower yields and selectivities than compound **1j** with two oxygen atoms in the cyclic chiral side chain. Diselenide **1j** was synthesized by Sharpless dihydroxylation of 2-bromostyrene, acetalization of the diol **9** (98% *ee*), and subsequent diselenide formation (Scheme 2).

Other alkenes were also used in the methoxyselenenylation reaction and some results are shown in Table 4. The methoxy-substituted reagent **1b** was superior to the unsubstituted ones like **1c** (entries 1 and 3, Table 4). MOM-protection in the chiral side chain not only increased the stereoselectivity, but also the yields, as shown in the methoxyselenenylation of 2-methoxystyrene (Table 4, entry 2).

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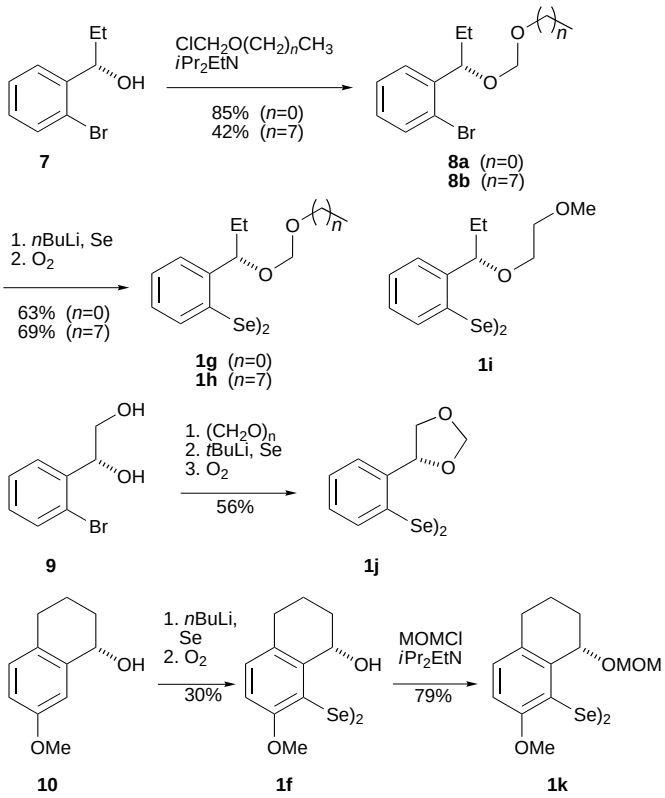


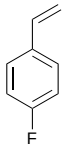
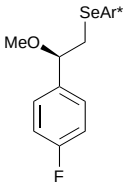
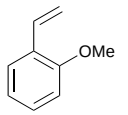
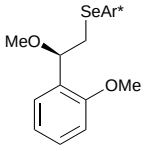
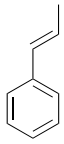
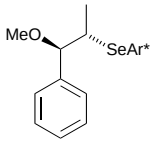
Scheme 2. Synthesis of side chain protected diselenides.

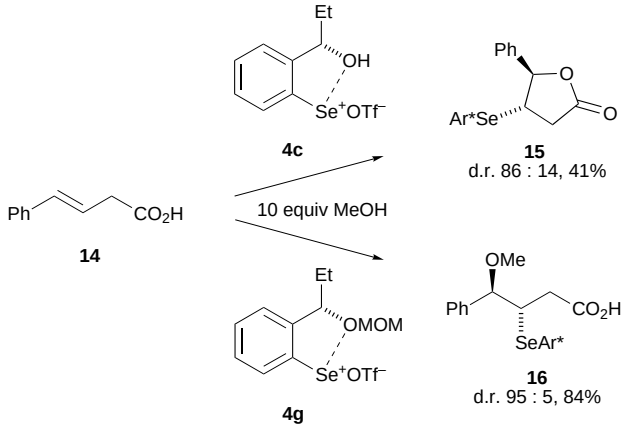
Table 3. Methoxyselenenylation of styrene using side chain protected selenium electrophiles **4**.

Entry	Selenium electrophile	d.r. of **5** (*S*,*R*):(*S*,*S*)	Yield of **5** [%]
1[a]	**4g**	96:4	93
2[a]	**4h**	93.5:6.5	86
3[b]	**4i**	85:15	54
4	**4j**	95.5:4.5 [c]	59
5	**4k**	94.5:5.5	46

[a] See reference [9]. [b] See reference [4]. [c] (*R*,*R*):(*R*,*S*).

Table 4. Methoxyselenenylation of styrene derivatives using different selenium electrophiles.

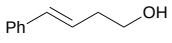
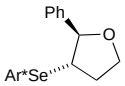
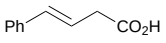
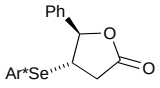
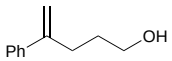
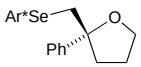
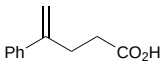
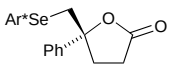
Entry	Alkene	Product	**11**	Diselenide	d.r. of **11** (*S*,*R*):(*S*,*S*)	Yield of **11** [%]
1			**11a**	**1b**	96.5:3.5	45
			11b	**1c**	82:18	49
2			**11c**	**1c**	89.5:10.5	47
			11d	**1g**	94.5:5.5	83
3			**11e**	**1b**	92.5:7.5	50
			11f	**1c**	86:14	61



Scheme 3. Reaction with unsaturated carboxylic acid **14**.

Chiral electrophilic selenium reagents can also be used in cyclization reactions of unsaturated compounds to generate heterocycles containing stereogenic centers. We applied some of the selenium electrophiles mentioned above in 5-*endo* and 5-*exo* cyclizations of unsaturated alcohols and carboxylic acids. The different internal stabilization of the selenium electrophiles owing to the different functionality in the chiral side chain (like the electrophiles generated from diselenides **1c** and **1g**) leads to different reactivity and hence to selectivity in the cyclization reactions. The unsaturated carboxylic acid **14** was treated under identical reaction conditions with **4c** and the selenium triflate (**4g**) generated from **1g** (Scheme 3). The stronger internal stabilization by the hydroxy group makes electrophile **4c** less reactive than **4g** and the outcome of the reaction is different. While reaction with **4c** in the presence of ten equivalents of methanol led exclusively to the cyclization product **15**, treatment of **14** with **4g** in the presence of ten equivalents of methanol resulted in the formation of the addition product **16** without any cyclization product. This observation was also interesting in light of the reaction with the homoallylic alcohol **12**: With both electrophiles, **4c** and **4g**, only the corresponding tetrahydrofuran derivative of type **13** was formed (Table 5, entries 1 and 2). All of these reactions were carried out in the presence of 10 equivalents of methanol. Electrophile **4g** formed a cyclization product with an alcohol (**12**) as the nucleophile, but the carboxylic acid **14** generated an addition product. From these results we concluded that an interaction exists between the electrophilic selenium reagent and the nucleophile. The nucleophile may coordinate to the electrophile and alter its reactivity. Because of the obvious difference in stabilization by an alcohol versus a carboxylic acid, we carried out the reaction of **14** with **4g** in the presence of 10 equivalents of acetic acid instead of methanol and we observed a clean cyclization reaction to form the lactone **15** (Table 5, entry 6). The selectivity was, however, decreased relative to the reaction with **14** in the presence of methanol, which might also indicate an altered selectivity owing to a difference in coordination of the

Table 5. Selenocyclizations with different selenium electrophiles.

Entry	Alkene	Product[a]	Electrophile	d.r.	Yield [%]
1	 **12**	 **13**	**4c**	92:8	87
2			**4g**	85.5:14.5	88
3			**4f**	97.5:2.5	48
4			**4k**	91.5:8.5	67
5	 **14**	 **15**	**4c**	86:14	41
6[b]			**4g**	84:16	50
7	 **17**	 **18**	**4c**	89:11	45
8			**4g**	93:7	92
9	 **19**	 **20**	**4c**	92.5:7.5	45
10			**4g**	92:8	80

[a] Major diastereomer shown. [b] In the presence of 10 equivalents of acetic acid.

electrophile to solvent molecules. The fine balance between the structure of the electrophile and selectivity can also be seen in the reaction of **12** with electrophile **4f**. Despite the hydroxy moiety in the chiral side chain only the cyclization product of type **13** was observed with very high selectivity (Table 5, entry 3) although with only moderate yield. However, MOM-protection of the hydroxy group did not improve the selectivities in this series, as shown in the selenocyclization using electrophile **4k** (Table 5, entry 4) versus **4f** (Table 5, entry 3).

In the 5-*exo* cyclizations investigated with substrates **17** and **19** (Table 5, entries 7–10) we did not observe any addition products. Only the five-membered heterocyles were formed as expected and the yields were quite high with electrophile **4g** (Table 5, entries 8 and 10) and MOM-protection of the hydroxy group in the chiral side chain.

Conclusion

We have synthesized a series of new chiral diselenides as precursor molecules for the generation of powerful selenium electrophiles, which add with high stereoselectivities to alkenes. These addition products are versatile building blocks in synthesis, as we have demonstrated earlier. A protection of the hydroxy moiety of the chiral side chain increased stereoselectivities and yields in the selenenylation reaction. With the new selenium electrophiles, stereoselective selenocyclization reactions can be performed as well, and we observed a fine balance of reactivity and selectivity depending on the nucleophile used in these reactions.

Experimental Section

General: All reactions were performed under argon with anhydrous solvents. The ^{1}H and ^{13}C NMR spectra were measured in $CDCl_3$ with TMS as an internal standard. Melting points are uncorrected. Alkenes **12**, **15**, **17**, and **19** were prepared as reported previously.[10]

GP1: General procedure for the addition of selenium electrophiles to styrene: The diselenide (0.1 mmol) was dissolved in dry diethyl ether (4 mL) under argon, cooled to $-78\,^\circ$C, and treated with bromine (0.11 mmol, 0.11 mL of a 1 M solution in CCl_4). After 10 min a solution of silver triflate (72 mg, 0.28 mmol) in methanol (0.1 mL) (in the reactions leading to addition products **6** other nucleophiles were used: **6b**: ethanol, **6c**: 2-propanol, **6d**: benzyl alcohol, **6e**: acetic acid) was added and the mixture was stirred for 10 min at $-78\,^\circ$C. The reaction mixture was cooled to $-100\,^\circ$C and treated with styrene (0.4 mmol, 0.046 mL). After the mixture had been stirred for 3–4 h at $-100\,^\circ$C, *sym*-collidine (0.3 mmol, 0.04 mL) was added, followed by water (4 mL). After extraction of the reaction mixture with *tert*-butyl methyl ether (3×10 mL), drying of the combined organic phases with $MgSO_4$, and removal of the solvent under reduced pressure, the residue was purified by flash chromatography on silica gel, yielding the addition products as colorless oils. The diastereomers could not be separated by flash chromatography and enrichment was excluded by comparison with the diastereomeric excess of the crude reaction mixtures. Spectroscopic data are given only for the major diastereomers of **5**, **6**, and **11**.

GP2: General procedure for the synthesis of diselenides **1g**, **1h**, and **1j** from the bromo precursors. The bromo precursor (2 mmol) was dissolved in dry THF (20 mL) under argon, cooled to $-78\,^\circ$C, and treated slowly with *t*BuLi (6 mmol). After the mixture had been warmed up to $0\,^\circ$C and stirred for 30 min, selenium powder (2.2 mmol) was added. The mixture was allowed to warm up to room temperature and was stirred for an additional 3 h, 1 N HCl (20 mL) was then added. After extraction of the resulting mixture with *tert*-butyl methyl ether (3×25 mL) and drying of the combined organic phases with $MgSO_4$, powdered KOH (100 mg) was added. The solvent was removed under vacuum and the residue purified by flash chromatography on silica gel. The diselenides were obtained as yellow oils.

(*S*,*S*)-Bis[2-(1-hydroxyethyl)phenyl] diselenide (1a): See reference [4].

(*S*,*S*)-Bis[2-(1-hydroxyethyl)-6-methoxyphenyl] diselenide (1b): (*S*)-1-(3-Methoxyphenyl)ethanol[11] (1.37 g, 9 mmol) and TMEDA (1.44 mL, 9.6 mmol) were dissolved in dry pentane (12 mL) under argon, cooled to $0\,^\circ$C, and treated slowly with *n*BuLi (5.8 mL, 9.26 mmol). After the mixture had been warmed to room temperature and stirred for 30 min, PhLi (16.9 mL, 27 mmol) was added and the stirring was continued for 8 h. Selenium powder (4.32 g, 54 mmol) was then added and the mixture stirred for an additional 8 h, then 1 N HCl (20 mL) was added. After extraction of the resulting mixture with *tert*-butylmethyl ether (3×50 mL) and drying of the combined organic phases with $MgSO_4$, powdered KOH (100 mg) was added. The solvent was removed under vacuum and the residue purified by flash chromatography (*tert*-butyl methyl ether/pentane 1:2) on silica gel, yielding **1b** (1.37 g, 66 %) as orange needles (ethanol). M.p. 146–148 °C; $[\alpha]_D^{25} = +914.5$ ($c = 0.96$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.26$ (d, $J = 6.5$ Hz, 6H; C*H*$_3$), 2.22 (s, 2H; O*H*), 3.83 (s, 6H; OC*H*$_3$), 5.06 (q, $J = 6.5$ Hz, 2H; C*H*), 6.84 (d, $J = 8.2$ Hz, 2H; arom. H), 7.18 (d, $J = 7.8$ Hz, 2H; arom. H), 7.36 (t, $J = 8.0$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 24.2$ (q, 2C; CH_3), 56.3 (q, 2C; OCH_3), 69.3 (d, 2C; CH), 110.0 (d, 2C), 118.0 (d, 2C), 118.7 (s, 2C), 131.3 (d, 2C), 151.4 (s, 2C), 159.7 (s, 2C); ^{77}Se NMR (114 MHz, $CDCl_3$): 365.6; IR ($CHCl_3$): $\tilde{\nu} = 3478$, 3376, 3005, 2939, 1568, 1464, 1422, 1136, 1051, 1016 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 462 (54) [M^+], 230 (60), 214 (100), 214 (100), 198 (28), 182 (16), 134 (35), 107 (22), 91 (26), 77 (21); elemental analysis calcd (%) for $C_{18}H_{22}O_4Se_2$ (460.29): C 46.98, H 4.82; found: C 46.80, H 4.90.

(*S*,*S*)-Bis[2-(1-hydroxypropyl)phenyl] diselenide (1c): See reference [4].

(*S*,*S*)-Bis[2-(1-hydroxypropyl)-6-methoxyphenyl] diselenide (1d): Synthesized from (*S*)-1-(3-methoxyphenyl)propanol[12] (1.99 g, 12 mmol) and elemental selenium (5.76 g, 72 mmol), using the procedure described for the synthesis of **1b**. Yield: 1.45 g (49 %), orange oil; $[\alpha]_D^{25} = +747.1$ ($c = 0.835$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.26$ (t, $J = 7.3$ Hz, 6H; C*H*$_3$), 1.54 (quint, $J = 7.3$ Hz, 4H; C*H*$_2$), 2.29 (s, 2H; O*H*), 3.85 (s, 6H; OC*H*$_3$), 4.76 (dd, $J = 7.3$ Hz, $J = 5.8$ Hz, 2H; C*H*), 6.83 (dd, $J = 8.2$ Hz, $J = 1.0$ Hz, 2H; arom. H), 7.11 (dd, $J = 8.3$ Hz, $J = 1.1$ Hz, 2H; arom. H), 7.34 (t, $J = 8.0$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.2$ (q, 2C; CH_3), 31.3 (t, 2C; CH_2), 56.3 (q, 2C; OCH_3), 74.5 (d, 2C; CHOH), 109.8 (d, 2C), 118.4 (d, 2C), 119.3 (s, 2C), 131.0 (d, 2C), 150.5 (s, 2C), 159.6 (s, 2C); ^{77}Se NMR (76 MHz, $CDCl_3$): 362.3; IR ($CHCl_3$): $\tilde{\nu} = 3489$, 3378, 3005, 2979, 2937, 1568, 1464, 1426, 1136, 1051, 1016 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 490

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(51) [M^+], 244 (52), 228 (100), 213 (56), 195 (13), 135 (26), 115 (26), 77 (28), 57 (71); elemental analysis calcd (%) for $C_{20}H_{26}O_4Se_2$ (488.34): C 49.20, H 5.37, O 13.11; found: C 49.07, H 5.49, O 12.97.

(*S*,*S*)-Bis[1-(8-hydroxy-5,6,7,8-tetrahydronaphthyl)] diselenide (1e): See reference [4].

(*S*,*S*)-Bis[1-(8-hydroxy-2-methoxy-5,6,7,8-tetrahydronaphthyl)] diselenide (1f): Synthesized from (*S*)-7-methoxy-1,2,3,4-tetrahydro-1-naphthol (**10**; 2.48 g, 14 mmol) and elemental selenium (4.74 g, 60 mmol), using the procedure described for the synthesis of **1b**. Yield: 1.25 g (35%), yellow solid; $[\alpha]_D^{25} = -62.3$ ($c = 0.41$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta =$ 1.65–2.08 (m, 8H; CH_2), 2.41 (s, 2H; OH), 2.55–2.85 (m, 4H; CH_2), 3.56 (s, 6H; OCH_3), 5.09 (d, $J = 2.5$ Hz, 2H; CH), 6.72 (d, $J = 8.5$ Hz, 2H; arom. H), 7.05 (d, $J = 8.5$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.3$ (t, 2C), 29.1 (t, 2C), 30.6 (t, 2C), 55.9 (q, 2C; CH_3), 66.5 (d, 2C; CH), 110.7 (d, 2C), 121.1 (s, 2C), 130.3 (s, 2C), 132.1 (d, 2C), 142.6 (s, 2C), 158.4 (s, 2C); IR ($CHCl_3$): $\tilde{\nu} = 3432$, 3075, 2919, 1589, 1471, 1432, 1262, 1194, 1164, 1080, 1049, 1009, 974, 926, 844, 809, 762 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 514 (23) [M^+], 256 (100), 240 (87), 159 (61), 147 (55), 128 (38), 115 (59).

(*S*,*S*)-Bis-[2-(1-methoxymethoxy-propyl)phenyl] diselenide (1g): Yield: 650 mg (63%), yellow oil; $[\alpha]_D^{25} = -79.6$ ($c = 0.96$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (t, $J = 7.6$ Hz, 6H; CH_3), 1.75–1.88 (m, 4H; CH_2), 3.38 (s, 6H; OCH_3), 4.49 (d, $J = 6.6$ Hz, 2H; OC*H*HO), 4.55 (d, $J =$ 6.9 Hz, 2H; OCH*H*O), 4.96 (dd, $J = 5.2$ Hz, $J = 8.0$ Hz, 2H; ArCH), 7.13 (dt, $J = 1.7$ Hz, $J = 7.3$ Hz, 2H; arom. H), 7.23 (dt, $J = 1.3$ Hz, $J = 7.5$ Hz, 2H; arom. H), 7.31 (dd, $J = 1.7$ Hz, $J = 9.3$ Hz, 2H; arom. H), 7.50 (dd, $J =$ 1.2 Hz, $J = 7.7$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.5$ (q, 2C; CH_3), 29.9 (t, 2C; CH_2), 55.7 (q, 2C; OCH_3), 78.9 (d, 2C; CH), 94.8 (d, 2C; OCH_2O), 127.2 (d, 2C), 127.7 (d, 2C), 128.2 (d, 2C), 129.7 (s, 2C), 132.9 (d, 2C), 142.4 (s, 2C); IR ($CHCl_3$): $\tilde{\nu} = 3060$, 2964, 2934, 1586, 1463, 1439, 1157, 1131, 1100, 1028, 918 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 518 (32) [M^+], 227 (18), 213 (30), 197 (47), 185 (28), 157 (5), 116 (15), 91 (12), 77 (5), 57 (10); HRMS: calcd for $C_{22}H_{30}O_4Se_2$ [M^+] 518.0475, found 518.0446.

(*S*,*S*)-Bis-[2-(1-methoxy-*n*-octyloxy-propyl)phenyl] diselenide (1h): Yield: 1.05 g (69%), yellow oil; $[\alpha]_D^{25} = -61.7$ ($c = 1.08$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.88$ (t, $J = 6.9$ Hz, 6H; CH_3), 0.96 (t, $J = 7.4$ Hz, 6H; CH_3), 1.20–1.40 (s, 20H; CH_2), 1.46–1.58 (m, 4H; CH_2), 1.66–1.92 (m, 4H; CH_2), 3.41 (dt, $J = 6.7$ Hz, $J = 9.4$ Hz, 2H; OC*H*HCH$_2$), 3.65 (dt, $J = 6.6$ Hz, $J = 9.4$ Hz, 2H; OCH*H*CH$_2$), 4.51 (d, $J = 6.7$ Hz, 2H; OC*H*-HO), 4.63 (d, $J = 6.8$ Hz, 2H; OCH*H*O), 4.95 (dd, $J = 5.4$ Hz, $J = 7.8$ Hz, 2H; ArCH), 7.12 (dt, $J = 1.7$ Hz, $J = 7.3$ Hz, 2H; arom. H), 7.20 (dt, $J =$ 1.3 Hz, $J = 7.5$ Hz, 2H; arom. H), 7.30 (dd, $J = 1.6$ Hz, $J = 7.6$ Hz, 2H; arom. H), 7.69 (dd, $J = 1.2$ Hz, $J = 7.8$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.6$ (q, 2C; CH_3), 14.1 (q, 2C; CH_3), 22.7 (t, 2C; CH_2), 26.2 (t, 2C; CH_2), 29.3 (t, 2C; CH_2), 29.4 (t, 2C; CH_2), 29.7 (t, 2C; CH_2), 29.9 (t, 2C; CH_2), 31.8 (t, 2C; CH_2), 68.4 (q, 2C; OCH_3), 79.0 (d, 2C; CH), 93.3 (t, 2C; OCH_2O), 127.2 (d, 2C), 127.6 (d, 2C), 128.2 (d, 2C), 129.6 (s, 2C), 132.7 (d, 2C), 142.4 (s, 2C); IR ($CHCl_3$): $\tilde{\nu} = 3061$, 2931, 2857, 1586, 1463, 1438, 1109, 1020, 945, 905 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 714 (57) [M^+], 227 (18), 213 (37), 197 (76), 157 (7), 91 (16), 71 (73), 57 (100), 43 (74); HRMS calcd for $C_{36}H_{58}O_4Se_2$ [M^+] 714.2666, found 714.2679.

(*S*,*S*)-Bis[2-{1-(2-methoxyethoxy)propyl}phenyl] diselenide (1i): See reference [4].

(*R*,*R*)-Bis[2-([1,3]dioxolan-4-yl)phenyl] diselenide (1j): Yield: 208 mg (64%), yellow oil; $[\alpha]_D^{25} = -55.1$ ($c = 0.83$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 3.53$ (dd, $J = 6.6$ Hz, $J = 8.0$ Hz, 2H; OC*H*HCHO), 4.23 (dd, $J = 6.9$ Hz, $J = 8.0$ Hz, 2H; OCH*H*CHO), 5.02 (s, 2H; OC*H*HO), 5.28 (s, 2H; CH*H*O), 5.31 (t, $J = 6.6$ Hz, 2H; ArCHO), 7.18 (dt, $J = 1.5$ Hz, $J =$ 7.5 Hz, 2H; arom. H), 7.34 (dt, $J = 1.2$ Hz, $J = 7.5$ Hz, 2H; arom. H), 7.49 (dd, $J = 1.5$ Hz, $J = 7.8$ Hz, 2H; arom. H), 7.58 (dd, $J = 1.2$ Hz, $J = 7.7$ Hz, 2H; arom H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 71.3$ (t, 2C), 77.03 (d, 2C), 95.9 (t, 2C), 126.2 (d, 2C), 128.7 (s, 2C), 128.7 (d, 2C), 129.4 (d, 2C), 135.6 (d, 2C), 141.7 (s, 2C); IR ($CHCl_3$): $\tilde{\nu} = 3063$, 2930, 2856, 1586, 1466, 1434, 1157, 1089, 1019, 947 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 458 (38) [M^+], 228 (31), 198 (50), 183 (23), 171 (60), 157 (9), 118 (17), 102 (9), 91 (100), 77 (17), 65 (10), 43 (11); HRMS calcd for $C_{18}H_{18}O_4Se_2$ [M^+] 457.9536, found 457.9521.

(*S*,*S*)-Bis[1-(2-methoxy-8-methoxymethyl-5,6,7,8-tetrahydronaphthyl)] diselenide (1k): Compound **1f** (260 mg, 0.51 mmol) was dissolved in CH_2Cl_2 (20 mL) and diisopropylethyl amine (20 mL). Chloromethyl methyl ether (1 mL, 12.5 mmol) was added at 0 °C. After 1 h the reaction mixture was warmed to room temperature and stirred for an additional 8 h, then 2 N HCl (70 mL) was added. After extraction of the resulting mixture with *tert*-butyl methyl ether (3 × 50 mL), the combined organic phases were washed with saturated aqueous $NaHCO_3$ and dried with $MgSO_4$. The solvent was removed in vacuo and the residue purified by medium-pressure chromatography (*tert*-butyl methyl ether/hexane 1:1), yielding **1k** (240 mg; 79%) as a red oil. $[\alpha]_D^{25} = -897$ ($c = 0.82$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.03$ (dt, $J = 3.3$ Hz, $J = 13.8$ Hz, 2H; CH_2), 1.55–1.67 (m, 2H; CH_2), 1.76–1.93 (m, 2H; CH_2), 1.98–2.09 (m, 2H; CH_2), 2.51 (ddd, $J = 3.8$ Hz, $J = 11.9$ Hz, $J = 16.8$ Hz, 2H; CH_2), 2.74 (dd, $J = 2.8$ Hz, $J = 16.4$ Hz, 2H; CH_2), 3.30 (s, 6H; CH_2OCH_3), 3.88 (s, 6H; $ArOCH_3$), 4.11 (t, $J = 3.0$ Hz, 2H; ArCHO), 4.56 (d, $J = 6.9$ Hz, 2H; OC*H*HO), 4.79 (d, $J = 6.9$ Hz, 2H; OCH*H*O), 6.86 (d, $J = 8.4$ Hz, 2H; arom. H), 7.06 (d, $J = 8.5$ Hz, 2H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.3$ (t, 2C), 29.0 (t, 2C), 29.3 (t, 2C), 55.6 (q, 2C), 56.3 (q, 2C), 74.7 (d, 2C), 97.4 (t, 2C), 111.1 (d, 2C), 122.5 (s, 2C), 130.0 (s, 2C), 131.3 (d, 2C), 140.5 (s, 2C), 158.6 (s, 2C); IR ($CHCl_3$): $\tilde{\nu} = 3005$, 2938, 2838, 1472, 1440, 1265, 1149, 1094, 1031 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 602 (8) [M^+], 540 (15), 478 (12), 319 (7), 255 (80), 240 (69), 225 (10), 195 (7), 160 (84), 145 (72), 128 (58), 115 (80), 102 (11), 91 (19), 45 (100); HRMS calcd for $C_{26}H_{34}O_6Se_2$ [M^+] 602.0686, found 602.0685.

(*S*)-1-[2-{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}phenyl]ethanol (5a): See reference [4].

(*S*)-1-[6-Methoxy-2-{[(*R*)-(2-methoxy-2-phenyl)ethyl]seleno}phenyl]ethanol (5b): Yield 55%, colorless oil. $[\alpha]_D^{25} = -1.2$ ($c = 0.55$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.48$ (d, $J = 6.5$ Hz, 3H; CH_3), 1.65 (s, 1H; OH), 3.13 (d, $J = 5.3$ Hz, 1H; C*H*HSe), 3.14 (d, $J = 8.1$ Hz, 1H; CH*H*Se), 3.21 (s, 3H; OCH_3), 3.88 (s, 3H; OCH_3), 4.29 (dd, $J = 8.1$ Hz, $J = 5.3$ Hz, 1H; C*H*CH$_2$Se), 5.41 (q, 5.8 Hz, 1H; CH), 6.79 (d, $J = 8.0$ Hz, 1H; arom. H), 7.14 (dd, $J = 7.8$ Hz, $J = 0.8$ Hz, 1H; arom. H), 7.22–7.35 (m, 6H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 24.1$ (q, CH_3), 34.9 (t, CH_2Se), 56.1 (q, OCH_3), 56.8 (q, OCH_3), 69.8 (d, CH_3), 83.4 (d, *C*HOMe), 109.9 (d), 117.1 (s), 118.2 (d), 126.6 (d, 2C), 128.0 (d), 128.5 (d, 2C), 129.7 (d), 141.0 (s), 150.0 (s), 162.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 3666$, 3382, 3005, 2937, 2838, 1570, 1464, 1431, 1136, 1103, 1052, 1016 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 366 (18) [M^+], 230 (37), 184 (30), 151 (27), 135 (21), 121 (100), 103 (14), 91 (18), 77 (15); HRMS calcd for $C_{18}H_{22}O_3Se$ [M^+] 366.0734, found 366.0747.

(*S*)-1-[2-{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}phenyl]propanol (5c): See reference [4].

(*S*)-1-[6-Methoxy-2-{[(*R*)-(2-methoxy-2-phenyl)ethyl]seleno}phenyl]propanol (5d): Yield: 45%, colorless oil. $[\alpha]_D^{25} = -25.8$ ($c = 0.30$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (t, $J = 7.1$ Hz, 3H; CH_2CH_3), 1.31 (d, $J = 7.1$ Hz, 3H; $CHCH_3$), 1.78 (quint, $J = 7.5$ Hz, 2H; CH_2CH_3), 2.52 (d, $J =$ 3.6 Hz, 1H; OH), 3.29 (s, 3H; OCH_3), 3.48 (qd, $J = 7.1$ Hz, $J = 7.1$ Hz, 1H; CHSe), 4.42 (d, $J = 4.4$ Hz, 1H; CHOMe), 5.08 (m, 1H; C*H*OH), 7.12–7.56 (m, 7H; arom. H), 7.47 (d, $J = 7.7$ Hz, 1H; arom. H), 7.55 (d, $J = 7.7$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_2CH_3), 16.3 (q, $CHCH_3$), 31.5 (t, CH_2CH_3), 46.4 (d, *C*HCH$_3$), 57.4 (q, OCH_3), 74.4 (d, CHOH), 86.1 (d, *C*HOMe), 126.5 (d), 126.9 (d, 2C), 127.6 (d), 127.7 (d), 127.9 (d), 128.2 (d, 2C), 128.7 (s), 135.2 (d), 139.3 (s), 147.0 (s); IR ($CHCl_3$): $\tilde{\nu} = 3666$, 3382, 3005, 2937, 2838, 1570, 1464, 1431, 1136, 1103, 1052, 1016 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 364 (10) [M^+], 199 (25), 149 (20), 121 (100), 105 (6), 91 (20), 77 (20); HRMS calcd for $C_{19}H_{24}O_2Se$ [M^+] 364.0942, found 364.0953.

1-[{(*R*)-(2-Methoxy-2-phenyl)ethyl}seleno]-(*S*)-5,6,7,8-tetrahydronaphth-8-ol (5e): See reference [4].

2-Methoxy-1-[{(*R*)-(2-methoxy-2-phenyl)ethyl}seleno]-(*S*)-5,6,7,8-tetrahydro-8-naphthol (5f): Column chromatography of the crude reaction mixture on silica gel (*tert*-butyl methyl ether/pentane 1:2). Yield: 30 mg (38%), pale yellow oil; $[\alpha]_D^{25} = -43.4$ ($c = 0.99$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.75$ (m, 2H; CH_2), 2.00 (m, 1H; CH_2), 2.20 (m, 1H; CH_2), 2.64 (m, 1H; CH_2), 2.78 (m, 1H; CH_2), 2.98 (dd, $J = 10.2$ Hz, $J =$ 12.8 Hz, 1H; C*H*H*Se*), 3.21 (s, 3H; OCH_3), 3.26 (dd, $J = 3.6$ Hz, $J =$ 12.8 Hz, 1H; CH*H*Se), 3.82 (s, 3H; $ArOCH_3$), 5.03 (s, 2H; PhCH_2), 5.27 (d, $J = 3.1$ Hz, 1H; C*H*OCH$_3$), 6.76 (d, $J = 8.4$ Hz, 1H; arom. H), 7.07 (d, $J = 8.4$ Hz, 1H; arom. H), 7.22–7.33 (m, 5H; ArH); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.7$ (d), 29.4 (d), 30.9 (t, $SeCH_2$), 35.2 (d), 56.1 (q, OCH_3), 56.8 (q, $ArOCH_3$), 66.1 (d, CHOH), 83.8 (t), 110.6 (d), 120.3 (s), 126.5 (d, 2C), 128.0 (d), 128.5 (d, 2C), 130.2 (s), 130.8 (d), 140.9 (s), 140.2 (s), 157.7 (s); IR ($CHCl_3$): $\tilde{\nu} = 3451$, 2931, 2833, 1589, 1454, 1263, 1083, 973, 845, 805, 758, 703 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 392 (10) [M^+], 256 (45), 177 (36), 160

(14), 145 (14), 135 (20), 121 (100), 115 (16), 103 (19), 91 (22), 77 (21), 51 (6), 43 (8); HRMS calcd for $C_{20}H_{24}O_3Se$ [M^+] 392.0891, found 392.0889.

(*S*)-1-[2-{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}phenyl] propyl methoxymethyl ether (5g): Yield: 93%, light yellow oil; $[\alpha]_D^{25} = -107$ ($c = 2.3$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.98$ (t, $J = 7.4$ Hz, 3H; CH_3), 1.65–1.80 (m, 2H; CH_2), 3.06 (dd, $J = 5.3$ Hz, $J = 11.2$ Hz, 1H; C*H*HSe), 3.24 (s, 3H; CHOCH_3), 3.29 (dd, $J = 3.2$ Hz, $J = 11.2$ Hz, 1H; CH*H*Se), 3.35 (s, 3H; CH_2OCH_3), 4.35 (dd, $J = 5.3$ Hz, $J = 8.1$ Hz, 1H; C*H*OCH$_3$), 4.53 (dd, $J = 6.6$ Hz, $J = 17.3$ Hz, 2H; OCH_2O), 5.05 (t, $J = 6.0$ Hz, 1H; ArCH), 7.14 (dt, $J = 1.4$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.22–7.38 (m, 6H; arom. H), 7.42 (dd, $J = 1.7$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.58 (dd, $J = 1.2$ Hz, $J = 7.8$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 30.5 (t, CH_2), 35.5 (t, CH_2Se), 55.6 (q, OCH_3), 55.9 (q, OCH_3), 78.3 (d, ArCH), 83.0 (d, ArCH), 94.6 (t, OCH_2O), 126.6 (d), 126.7 (d, 2C), 127.3 (d), 127.7 (d), 128.1 (d), 128.5 (d, 2C), 130.1 (s), 133.2 (d), 140.9 (s), 144.1 (s); IR ($CHCl_3$): $\tilde{\nu} = 3006$, 2934, 1464, 1440, 1157, 1104, 1035, 956, 917 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 394 (10) [M^+], 213 (37), 197 (9), 185 (14), 135 (12), 121 (100), 103 (12), 91 (14), 77 (12), 45 (42); HRMS calcd for $C_{20}H_{26}O_3Se$ [M^+] 394.1047, found 394.1042.

(*S*)-1-[2-{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}phenyl] propyl methoxy-*n*-octyl ether (5h): Yield: 86%, colorless oil; $[\alpha]_D^{25} = -92$ ($c = 0.92$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.88$ (t, $J = 6.9$ Hz, 3H; CH_3), 0.97 (t, $J = 7.4$ Hz, 3H; CH_3), 1.18–1.35 (s, 10H; CH_2), 1.43–1.56 (m, 2H; CH_2), 1.68–1.80 (m, 2H; CH_2), 3.08 (dd, $J = 5.2$ Hz, $J = 12.1$ Hz, 1H; C*H*HSe), 3.26 (s, 3H; CHOCH_3), 3.34 (dd, $J = 3.2$ Hz, $J = 12.1$ Hz, 1H; CH*H*Se), 3.39 (dt, $J = 6.5$ Hz, $J = 9.5$ Hz, 1H; OC*H*HCH$_2$), 3.63 (dt, $J = 6.6$ Hz, $J = 9.4$ Hz, 1H; OCH*H*CH$_2$), 4.34 (dd, $J = 5.2$ Hz, $J = 8.3$ Hz, 1H C*H*OCH$_3$), 4.52 (d, $J = 7.0$ Hz, 1H; OC*H*HO), 4.63 (d, $J = 7.0$ Hz, 1H; OCH*H*O), 5.06 (t, $J = 6.9$ Hz, 1H; ArC*H*), 7.13 (dt, $J = 1.6$ Hz, $J = 7.6$ Hz, 1H; arom. H), 7.26 (dt, $J = 1.4$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.29–7.38 (m, 5H; arom. H), 7.41 (dd, $J = 1.6$ Hz, $J = 7.7$ Hz, 1H; arom. H), 7.58 (dd, $J = 1.2$ Hz, $J = 7.7$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 14.1 (q, CH_3), 22.7 (t, CH_2), 26.2 (t, CH_2), 29.3 (t, CH_2), 29.4 (t, CH_2), 29.6 (t, CH_2), 30.6 (t, CH_2), 31.8 (t, CH_2), 35.5 (t, CH_2Se), 56.9 (q, OCH_3), 68.3 (q, OCH_3), 78.4 (d, CH), 83.1 (d, CH), 93.5 (t, OCH_2O), 126.7 (d, 2C), 126.8 (d), 127.3 (d), 127.7 (d), 128.1 (d), 128.5 (d, 2C), 130.1 (s), 133.3 (d), 141.0 (s), 144.3 (s); IR ($CHCl_3$): $\tilde{\nu} = 3005$, 2918, 2826, 1493, 1464, 1353, 1102, 1030, 954, 905, 601 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 492 (9) [M^+], 213 (58), 197 (16), 185 (21), 135 (17), 121 (100), 103 (13), 91 (14), 71 (18), 57 (27), 43 (26); HRMS calcd for $C_{27}H_{40}O_3Se$ [M^+] 492.2143, found 492.2153.

1-[(*S*)-1-(2-Methoxyethoxy)propyl]-2-[(*R*)-(2-methoxy-2-phenyl)ethyl]selenobenzene (5i): See reference [4].

(*R*)-4-[2{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}phenyl][1,3]dioxolane (5j): Yield: 59%, colorless oil; $[\alpha]_D^{25} = -67.9$ ($c = 1.47$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 3.06$ (dd, 1H; $J = 5.0$ Hz, $J = 12.1$ Hz, C*H*HSe), 3.24 (s, 3H; OCH_3), 3.30 (dd, 1H; $J = 8.4$ Hz, $J = 12.1$ Hz, CH*H*Se), 3.58 (dd, $J = 6.6$ Hz, $J = 8.0$ Hz, 1H; OC*H*HCHO), 4.32–4.39 (m, 1H; OCH*H*-CHO), 5.08 (s, 1H; OC*H*HO), 5.29 (s, 1H; CH*H*O), 5.39 (t, $J = 6.6$ Hz, 1H; ArCH), 7.18 (dt, $J = 1.8$ Hz, $J = 7.2$ Hz, 1H; arom. H), 7.24–7.40 (m, 6H; arom. H), 7.50–7.53 (m, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 36.1$ (t, CH_2Se), 57.0 (q, OCH_3), 71.5 (t), 76.8 (d), 83.0 (d, ArCHOCH_2), 95.9 (t), 125.6 (d), 126.3 (d), 126.6 (d, 2C), 127.8 (d), 128.2 (d), 128.3 (d), 128.6 (d), 128.7 (s), 134.1 (d), 140.6 (s), 142.0 (s); IR ($CHCl_3$): $\tilde{\nu} = 3006$, 2937, 2861, 1493, 1454, 1353, 1156, 1090, 1020, 948 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 364 (7) [M^+], 228 (12), 198 (5), 149 (6), 135 (8), 121 (100), 103 (11), 91 (22), 77 (14), 43 (6); HRMS calcd for $C_{18}H_{20}O_3Se$ [M^+] 364.0578, found 364.0570.

(*S*)-[2-Methoxy-8-methoxymethyl-1-{[(*R*)-(2-Methoxy-2-phenyl)ethyl]seleno}]-5,6,7,8-tetrahydronaphthalene (5k): Yield: 46%, colorless oil; $[\alpha]_D^{25} = -43.4$ ($c = 0.99$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.55$–1.79 (m, 2H; CH_2), 1.88–2.04 (m, 1H; CH_2), 2.36–2.45 (m, 1H; CH_2), 2.63 (ddd, $J = 6.4$ Hz, $J = 12.4$ Hz, $J = 15.9$ Hz, 1H; CH_2), 2.75–2.86 (m, 1H; CH_2), 3.12–3.16 (m, 2H; CH_2Se), 3.17 (s, 3H; CHOCH_3), 3.41 (s, 3H; CH_2OCH_3), 3.84 (s, 3H; $ArOCH_3$), 4.15 (dd, $J = 5.7$ Hz, $J = 12.2$ Hz, 1H; ArCHO), 4.73 (d, $J = 6.9$ Hz, 1H; OC*H*HO), 4.99 (t, $J = 3.0$ Hz, 1H; ArCH), 5.14 (d, $J = 6.9$ Hz, 1H; OCH*H*O), 6.77 (d, $J = 8.4$ Hz, 1H; arom. H), 7.04 (d, $J = 8.5$ Hz, 1H; arom. H), 7.17–7.30 (m, 5H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.5$ (t, CH_2), 29.1 (t, CH_2), 29.8 (t, CH_2), 33.9 (t, CH_2Se), 55.9 (q, CH_2OCH_3), 56.1 (q, $CHOCH_3$), 56.9 (q, $ArOCH_3$), 75.7 (d), 83.4 (d, ArCHO), 97.6 (t, OCH_2O), 110.9 (d), 121.5 (s), 126.6 (d, 2C), 127.7 (d), 128.6 (d, 2C), 130.0 (s), 130.7 (d), 140.0 (s), 141.3 (s), 158.0 (s); IR ($CHCl_3$): $\tilde{\nu} = 2934$, 2835, 1472, 1263, 1149, 1095, 1032, 966, 918, 754, 702 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 436 (11) [M^+], 255 (100), 240 (15), 205 (15), 160 (21), 135 (23), 121 (71), 103 (21), 91 (21), 77 (19), 45 (49); HRMS calcd for $C_{22}H_{28}O_4Se$ [M^+] 436.1153, found 436.1154.

(*S*)-1-[2-{[(*R*)-(2-Ethoxy-2-phenyl)ethyl]seleno}phenyl]propanol (6b): Yield: 58%, colorless oil; $[\alpha]_D^{25} = -47.3$ ($c = 1.00$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (t, $J = 7.4$, 3H; CH_2CH_3), 1.18 (t, $J = 7.0$ Hz, 3H; CH_2CH_3), 1.78 (quint, $J = 7.2$ Hz, 2H; CH_2CH_3), 2.31 (d, $J = 4.5$ Hz, 1H; OH), 3.10 (dd, $J = 4.9$ Hz, $J = 12.1$ Hz, 2H; CH_2O), 3.27 (dd, $J = 8.5$ Hz, $J = 12.1$ Hz, 1H; C*H*HSe), 3.38 (dq, $J = 1.7$ Hz, $J = 7.1$ Hz, 1H; C*H*HSe), 4.47 (dd, $J = 4.9$ Hz, $J = 8.4$ Hz, 1H; C*H*CH$_2$Se), 5.02 (dt, $J = 4.5$ Hz, $J = 6.4$ Hz, 1H; CH), 7.15 (dt, $J = 1.6$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.20–7.38 (m, 6H; arom. H), 7.44 (dd, $J = 1.6$ Hz, $J = 7.7$ Hz, 1H; arom. H), 7.50 (dd, $J = 1.4$ Hz, $J = 7.7$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 15.2 (q, CH_3), 31.1 (t, CH_2), 36.3 (t, CH_2Se), 64.6 (t, CH_2O), 74.7 (d, CHOH), 81.1 (d, CHOMe), 126.3 (d), 126.5 (d, 2C), 127.4 (d), 127.9 (d), 128.0 (d), 128.5 (d, 2C), 129.7 (s), 133.6 (d), 141.5 (s), 145.9 (s); IR ($CHCl_3$): $\tilde{\nu} = 3446$, 3005, 2975, 2933, 2875, 1454, 1114, 1092, 1003, 604 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 364 (6) [M^+], 214 (10), 185 (7), 135 (100), 107 (45), 87 (14), 79 (17), 43(7); HRMS calcd for $C_{19}H_{24}O_2Se$ [M^+] 364.0941, found 364.0938.

(*S*)-1-[2-{[(*R*)-(2-[1-Methylethoxy]-2-phenyl)ethyl]seleno}phenyl]propanol (6c): Yield: 64%, colorless oil; $[\alpha]_D^{25} = -42.6$ ($c = 0.91$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (t, $J = 7.4$, 3H; CH_2CH_3), 1.08 (d, $J = 6.3$, 3H; $CHCH_3$), 1.17 (d, $J = 6.1$ Hz, 3H; $CHCH_3$), 1.78 (quint, $J = 7.1$ Hz, 2H; CH_2CH_3), 2.30 (d, $J = 3.1$ Hz, 1H; OH), 3.08 (dd, $J = 4.8$ Hz, $J = 11.9$ Hz, 1H; C*H*HSe), 3.25 (dd, $J = 8.6$ Hz, $J = 11.9$ Hz, C*H*HSe), 3.52 (hept, $J = 6.1$ Hz, 1H; $CH(CH_3)_2$), 4.61 (dd, $J = 5.0$ Hz, $J = 8.5$ Hz, 1H; C*H*CH$_2$Se), 5.04 (dt, $J = 4.1$ Hz, $J = 6.3$ Hz, 1H; CH), 7.10–7.49 (m, 9H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 21.2 (q, CH_3CHCH_3), 23.2 (q, CH_3CHCH_3), 31.0 (t, CH_2), 36.6 (t, CH_2Se), 69.8 (t, CH_2O), 74.3 (d, CHOH), 78.6 (d, CHCH$_2$Se), 126.3 (d), 126.5 (d, 2C), 127.2 (d), 127.8 (d), 128.2 (d), 128.4 (d, 2C), 130.0 (s), 133.2 (d), 142.2 (s), 145.7 (s); IR ($CHCl_3$): $\tilde{\nu} = 3599$, 3434, 3005, 2971, 2933, 2876, 1463, 1379, 1120, 1086, 1046, 1006, 972 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 378 (13) [M^+], 214 (12), 197 (8), 185 (12), 149 (72), 107 (100), 91 (20), 79 (20), 51 (6), 43 (28); HRMS calcd for $C_{20}H_{26}O_2Se$ [M^+] 378.1098, found 378.1114.

(*S*)-1-[2-{[(*R*)-(2-Benzyloxy-2-phenyl)ethyl]seleno}phenyl]propanol (6d): Yield: 63%, colorless oil; $[\alpha]_D^{25} = -55.9$ ($c = 0.86$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.95$ (t, $J = 7.3$ Hz, 3H; CH_2CH_3), 1.74 (quint, $J = 7.2$ Hz, 2H; CH_2CH_3), 2.09 (s, 1H; OH), 3.13 (dd, $J = 5.0$ Hz, $J = 12.2$ Hz, 1H; C*H*HSe), 3.35 (dd, $J = 8.4$ Hz, $J = 12.1$ Hz, 1H; CH*H*Se), 4.30 (d, $J = 11.8$ Hz, 2H; PhC*H*HO), 4.48 (d, $J = 11.8$ Hz, 1H; PhCH*H*O), 4.58 (dd, $J = 5.0$ Hz, $J = 8.3$ Hz, C*H*CH$_2$Se), 5.01 (t, $J = 5.6$ Hz, 1H; CH), 7.08–7.48 (m, 14H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 31.2 (t, CH_2CH_3), 36.2 (t, CH_2Se), 70.8 (t, PhCH_2O), 74.6 (d, CHOH), 80.7 (d, CHOCH$_2$), 126.3 (d), 126.8 (d, 2C), 127.4 (d), 127.6 (d), 127.8 (d, 2C), 127.9 (d), 128.2 (d), 128.3 (d, 2C), 128.6 (d, 2C), 129.8 (s), 133.5 (d), 138.0 (s), 140.9 (s), 145.9 (s); IR ($CHCl_3$): $\tilde{\nu} = 3442$, 3006, 2966, 2934, 1454, 1091, 1027, 973, 694 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 426 (9) [M^+], 214 (18), 197 (51), 185 (12), 149 (10), 130 (6), 118 (13), 105 (18), 91 (100), 77 (18), 65 (13), 51 (7), 43 (5); HRMS calcd for $C_{24}H_{26}O_2Se$ [M^+] 426.1098, found 426.1113.

(*S*)-1-[2-{[(*R*)-(2-Acetoxy-2-phenyl)ethyl]seleno}phenyl]propanol (6e): Yield: 35%, colorless oil; $[\alpha]_D^{25} = -33.1$ ($c = 0.72$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.95$ (t, $J = 7.4$ Hz, 3H; CH_2CH_3), 1.78 (quint, $J = 7.2$ Hz, 2H; CH_2CH_3), 1.82 (s, 3H; $COCH_3$), 2.20 (d, $J = 4.5$ Hz, 1H; OH), 3.21 (dd, $J = 5.5$ Hz, $J = 12.8$ Hz, C*H*HSe), 3.39 (dd, $J = 7.9$ Hz, $J = 12.8$ Hz, 1H; C*H*HSe), 5.02 (dt, $J = 4.0$ Hz, $J = 4.2$ Hz, 1H; CH), 5.99 (dd, $J = 5.5$ Hz, $J = 8.0$ Hz, 1H; C*H*CH$_2$Se), 7.15 (t, $J = 7.5$ Hz, 1H; arom. H), 7.23–7.40 (m, 6H; arom. H), 7.48 (d, $J = 7.7$ Hz, 1H; arom. H), 7.52 (d, $J = 7.7$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.3$ (q, CH_3), 20.9 (q, $COCH_3$), 31.4 (t, CH_2), 33.5 (t, CH_2Se), 74.4 (d, CHOH), 75.5 (t, CH_2O), 126.5 (d, 2C), 126.6 (d), 127.9 (d), 128.0 (d), 128.5 (d), 128.6 (d, 2C), 128.7 (s), 133.9 (d), 133.9 (d), 139.4 (s), 146.4 (s) 170.2 (d, $OCOCH_3$); IR ($CHCl_3$): $\tilde{\nu} = 3468$, 3006, 2965, 2933, 1732, 1463, 1373, 1248, 1048, 1020, 966 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 378 (17%) [M^+], 318 (8), 214 (71), 196 (26), 185 (61), 163 (29), 107 (28), 91 (23), 77 (29), 51 (11), 43 (100); HRMS calcd for $C_{19}H_{22}O_3Se$ [M^+] 378.0734, found 378.0746.

(*S*)-1-(2-Bromophenyl)propanol (7): See reference [12].

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(*S*)-1-Bromo-2-(1-methoxymethoxypropyl)benzene (8a): Chloromethyl methyl ether (1.68 mL, 21 mmol) was added slowly to (*S*)-1-(2-Bromophenyl)propanol (1.5 g, 7 mmol) in diisopropylethylamine (15 mL) at 0 °C. After stirring for 1 h the solution was allowed to warm up to room temperature and was stirred for an additional 10 h. The solvent was distilled off in vacuo and the residue purified by flash chromatography (*tert*-butyl methyl ether/pentane 1:10) on silica gel. Yield: 1.5 g (85 %), colorless oil; $[\alpha]_D^{25} = -135$ ($c = 1.41$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.00$ (t, $J = 7.3$ Hz, 3H; CH_3), 1.68–1.83 (m, 2H; CH_2CH_3), 3.35 (s, 3H; OCH_3), 4.54 (d, $J = 6.7$ Hz, 1H; OC*H*HO), 4.57 (d, $J = 6.7$ Hz, 1H; OCH*H*O), 4.96 (dd, $J = 5.0$ Hz, $J = 7.4$ Hz, 1H; ArCH), 7.10 (dt, $J = 1.8$ Hz, $J = 7.6$ Hz, 1H; arom. H), 7.30 (dt, $J = 1.2$ Hz, $J = 7.6$ Hz, 1H; arom. H), 7.46 (dd, $J = 1.2$ Hz, $J = 7.8$ Hz, 1H; arom. H), 7.50 (dd, $J = 1.3$ Hz, $J = 8.0$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.2$ (q, CH_3), 29.8 (t, CH_2), 55.7 (q, OCH_3), 77.9 (d, CH), 94.8 (t, OCH_2O), 123.0 (s), 127.5 (d), 127.8 (d), 128.6 (d), 132.6 (d), 141.7 (s); IR ($CHCl_3$): $\tilde{\nu} = 2968$, 2934, 2888, 1467, 1440, 1158, 1130, 1103, 1031, 917 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 258 (1) [M^+], 229 (37), 199 (12), 185 (9), 169 (17), 115 (8), 91 (8), 77 (8), 59 (18), 45 (100); HRMS calcd for $C_{11}H_{15}O_2Br$ [M^+] 258.0255, found 258.0246.

(*S*)-1-Bromo-2-(1-methoxy-*n*-octyloxy-propyl)benzene (8b): Chloromethyl *n*-octyl ether (2.5 mL, 13 mmol) was added slowly to (*S*)-1-(2-Bromophenyl)propanol (1.5 g, 7 mmol) in diisopropylethylamine (15 mL) at 0 °C. After stirring for 1 h the solution was allowed to warm up to room temperature and was stirred for an additional 10 h. The solvent was distilled off in vacuo and the residue purified by a Kugelrohr distillation (90 °C, 0.02 mbar). Yield: 1.05 g (42 %), colorless oil; $[\alpha]_D^{25} = -109$ ($c = 1.48$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.87$ (t, $J = 7.1$ Hz, 3H; CH_3), 0.96 (t, $J = 7.8$ Hz, 3H; CH_3), 1.20–1.38 (s, 10H; CH_2), 1.44–1.56 (m, 2H; CH_2), 1.64–1.83 (m, 2H; CH_2), 3.41 (dt, $J = 6.5$ Hz, $J = 9.3$ Hz, 1H; OC*H*HCH$_2$), 3.65 (dt, $J = 6.6$ Hz, $J = 9.4$ Hz, 1H; OCH*H*CH$_2$), 4.53 (d, $J = 6.8$ Hz, 1H; OC*H*HO), 4.65 (d, $J = 6.8$ Hz, 1H; OCH*H*O), 4.96 (dd, $J = 5.0$ Hz, $J = 7.4$ Hz, 1H; ArCH), 7.10 (dt, $J = 1.8$ Hz, $J = 7.6$ Hz, 1H; arom. H), 7.30 (dt, $J = 1.2$ Hz, $J = 7.6$ Hz, 1H; arom. H), 7.45 (dd, $J = 1.2$ Hz, $J = 7.8$ Hz, 1H; arom. H), 7.50 (dd, $J = 1.3$ Hz, $J = 8.0$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.2$ (q, CH_3), 14.1 (q, CH_3), 22.7 (t, CH_2), 26.2 (t, CH_2), 29.3 (t, CH_2), 29.4 (t, CH_2), 29.7 (t, CH_2), 29.9 (t, CH_2), 31.8 (t, CH_2), 68.4 (q, OCH_3), 78.0 (d, CH), 93.6 (t, OCH_2O), 123.0 (s), 127.5 (d), 128.0 (d), 128.6 (d), 132.6 (d), 141.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 2930$, 2857, 1467, 1439, 1107, 1028 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 327 (36) [M – 29]$^+$, 185 (47), 171 (38), 143 (89), 111 (59), 91 (16), 71 (98), 57 (100), 43 (49).

(*R*)-1-(2-Bromophenyl)-1,2-ethanediol (9): 2-Bromostyrene was dihydroxylated following the procedure described by Sharpless[13] using AD-mix-β. This led to (*R*)-1-(2-bromophenyl)-1,2-ethanediol in 90 % yield with an enantiomeric ratio of 99:1 *R*:*S* as determined by HPLC (Chiracel OD, 15 °C, *n*-hexane:2-propanol 9:1, 254 nm, 0.5 mL min^{-1}, $R_f(R) = 20.7$ min, $R_f(S) = 25.4$ min); $[\alpha]_D^{25} = -62.2$ ($c = 1.13$, $CHCl_3$); other spectroscopic data see reference [14].

(*R*)-4-(2-Bromphenyl)-[1,3]dioxolane:[15] (*R*)-1-(2-Bromophenyl)-1,2-ethanediol (**9**; 450 mg, 2.07 mmol), paraformaldehyde (200 mg, 6.2 mmol) and *p*-toluenesulfonic acid (5 mg, 0.025 mmol) were dissolved in dry ethanol (1.5 mL) and benzene (7.5 mL) and stirred at 60 °C for 1 h. The solvent was distilled off and the mixture heated to 200 °C in the Kugelrohr oven to distill off the product as a colorless liquid. Yield: 419 mg (88 %); $[\alpha]_D^{25} = -58.6$ ($c = 1.18$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 3.67$ (dd, $J = 5.9$ Hz, $J = 8.4$ Hz, 1H; OC*H*HCHO), 4.43 (dd, $J = 6.9$ Hz, $J = 8.2$ Hz, 1H; OCH*H*CHO), 5.09 (s, 1H; OC*H*HO), 5.30 (s, 1H; CH*H*O), 5.31 (t, $J = 6.4$ Hz, 1H; ArCH), 7.16 (dt, $J = 1.7$ Hz, $J = 7.7$ Hz, 1H; arom. H), 7.34 (dt, $J = 1.2$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.53 (dd, $J = 1.2$ Hz, $J = 7.9$ Hz, 1H; arom. H), 7.56 (dd, $J = 1.7$ Hz, $J = 7.9$ Hz, 1H; arom H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 71.1$ (t), 76.4 (d), 96.0 (t), 121.2 (s), 126.8 (d), 127.6 (d), 129.1 (d), 132.5 (d), 139.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 2930$, 2856, 1466, 1157, 1089, 1019, 947 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 228 (18) [M^+], 198 (68), 185 (22), 169 (11), 157 (7), 119 (100), 89 (59), 77 (18), 63 (17), 63 (17), 44 (75); HRMS calcd for $C_9H_9BrO_2$ [M^+] 227.9786, found 227.9798.

(*S*)-7-Methoxy-1,2,3,4-tetrahydro-1-naphthol (10): Synthesized by reduction of 7-methoxy-3,4-dihydro-2*H*-naphthalen-1-one with sodium borohydrate.[16] The racemic 7-methoxy-1,2,3,4-tetrahydro-1-naphthol (5.7 g, 32 mmol) was dissolved in toluene (50 mL) and vinylacetate (10 mL) and cross-linked enzyme crystals (PC-CLEC)[17] (50 mg) were added. The mixture was shaken for 22 h and the reaction monitored by HPLC. The mixture was filtrated, the solvent removed in vacuo, and the residue purified by flash chromatography (*tert*-butyl methyl ether/pentane 1:2) on silica gel. Yields: (*S*)-7-methoxy-1,2,3,4-tetrahydro-1-naphthol: 2.55 g (45 %), (*R*)-acetic acid 7-methoxy-1,2,3,4-tetrahydro-1-naphthalene-1-yl ester: 1.55 g (22 %) after recrystallization from *tert*-butyl methyl ether. $[\alpha]_D^{25} = +45.8$ ($c = 3.63$, $CHCl_3$), enantiomerically pure as determined by HPLC (Chiracel OD, 25 °C, *n*-hexane:2-propanol 95:5, 254 nm, 0.5 mL min^{-1}, $R_f(S) = 33.7$ min, $R_f(R) = 38.2$ min; other spectroscopic data see reference [16].

(*R*)-Acetic acid 7-methoxy-1,2,3,4-tetrahydro-1-naphthalene-1-yl ester: Enantiomerically pure as determined by HPLC (Chiracel OD, 25 °C, *n*-hexane/2-propanol 95:5, 254 nm, 0.5 mL min^{-1}, $R_f(R) = 13.0$ min, $R_f(S) = 14.6$ min; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.75–1.98$ (m, 4H; CH_2), 2.08 (s, 3H; $COCH_3$), 2.63–2.83 (m, 2H; CH_2), 3.77 (s, 3H; OCH_3), 5.96 (t, $J = 4.2$ Hz, 1H; CH), 6.78–6.82 (m, 2H; arom. H), 7.03 (d, $J = 9.1$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 19.0$ (t), 21.4 (q, CO*C*H$_3$), 28.1 (t), 29.0 (t), 55.3 (q, OCH_3), 70.1 (d, CH), 113.6 (d), 114.7 (d), 129.9 (s), 130.0 (d), 135.4 (s), 157.7 (s), 170.2 (s, C=O); IR ($CHCl_3$): $\tilde{\nu} = 2940$, 2836, 1732, 1614, 1504, 1371, 1233, 1037 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 220 (4) [M^+], 177 (10), 160 (100), 159 (93), 144 (32), 129 (20), 115 (25), 91 (18), 77 (17), 43 (33); elemental analysis calcd (%) for $C_{13}H_{16}O_3$ (220.27): C 70.89, H 7.32, O 21.79; found: C 71.03, H 7.42, O 21.92.

(*S*)-1-[6-Methoxy-2-{[(*R*)-(2-Methoxy-2-(4-fluoro)phenyl)ethyl]seleno}-phenyl]ethanol (11a): Yield: 24 mg (45 %), colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.48$ (d, $J = 6.6$ Hz, 3H; CH_3), 2.85 (d, $J = 3.8$ Hz, 1H; OH), 3.09 (dd, $J = 12.3$ Hz, $J = 5.2$ Hz, 1H; C*H*HSe), 3.12 (dd, $J = 12.3$ Hz, $J = 8.4$ Hz, 1H; CH*H*Se), 3.19 (s, 3H; CH_3), 3.88 (s, 3H; CH_3), 4.27 (dd, $J = 8.3$ Hz, $J = 5.2$ Hz, 1H; C*H*OMe), 5.41 (dq, $J = 6.8$ Hz, $J = 3.2$ Hz, 1H; CHOH), 6.80 (dd, $J = 8.2$ Hz, $J = 1.1$ Hz, 1H; arom. H), 7.01 (d, $J = 8.7$ Hz, 2H; arom. H), 7.14 (dd, $J = 7.7$ Hz, $J = 1.1$ Hz, 1H; arom. H), 7.19–7.24 (m, 2H; arom. H), 7.32 (t, $J = 7.9$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 24.3$ (q, CH_3), 34.9 (t, CH_2), 56.2 (q, OCH_3), 56.9 (q, OCH_3), 69.9 (d, CHOH), 82.9 (d, *C*HOMe), 109.9 (d), 115.5 (d, $^2J_{C,F} = 21$ Hz, 2C), 117.1 (s), 118.3 (d), 128.4 (d, $^3J_{C,F} = 8$ Hz, 2C), 129.9 (d), 136.9 (s), 150.0 (s), 160.7 (s), 162.0 (s, $^1J_{C,F} = 234$ Hz); MS (70 eV, EI): *m*/*z* (%): 384 (16) [M^+], 230 (41), 215 (12), 202 (14), 151 (21), 139 (100).

(*S*)-1-[2-{[(*R*)-(2-Methoxy-2-(4-fluoro)phenyl)ethyl]seleno}phenyl]propanol (11b): Yield: 49 %; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (t, $J = 7.5$ Hz, 3H; CH_3), 1.78 (quint, $J = 7.3$ Hz, 2H; CH_2CH_3), 2.38 (s, 1H; OH), 3.07 (dd, $J = 12.2$ Hz, $J = 5.2$ Hz, 1H; C*H*HSe), 3.22 (s, 3H; OCH_3), 3.24 (dd, $J = 12.2$ Hz, $J = 8.3$ Hz, 1H; CH*H*Se), 4.33 (dd, $J = 8.3$ Hz, $J = 5.2$ Hz, 1H; C*H*OMe), 5.03 (t, $J = 6.4$ Hz, 1H; CHOH), 7.0–7.5 (m, 8H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 31.2 (t, CH_2), 36.0 (t, CH_2), 56.9 (q, OCH_3), 74.8 (d, CHOH), 82.3 (d, CHOMe), 115.5 (d, $^2J_{C,F} = 21$ Hz, 2C), 126.4 (s), 126.5 (d), 127.6 (d), 128.0 (d), 128.3 (d, $^3J_{C,F} = 8$ Hz, 2C), 129.4 (s), 133.7 (d), 146.0 (s), 162.1 (s, $^1J_{C,F} = 234$ Hz); IR ($CHCl_3$): $\tilde{\nu} = 3604$, 3444, 3005, 2935, 2877, 2827, 1605, 1509, 1464, 1093, 968, 838 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 368 (7) [M^+], 214 (9), 185 (5), 139 (100), 109 (7), 73 (14).

(*S*)-1-[2-{[(*R*)-{2-Methoxy-2-(2-methoxyphenyl)}ethyl]seleno}phenyl]propanol (11c): Yield: 47 %, colorless oil; $[\alpha]_D^{25} = -48.6$ ($c = 1.60$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.98$ (t, $J = 7.4$ Hz, 3H; CH_2CH_3), 1.78 (quint., $J = 6.5$ Hz, 2H; CH_2CH_3), 2.40 (d, $J = 3.8$ Hz, 1H; OH), 3.11 (dd, $J = 12.4$ Hz, $J = 18.8$ Hz, C*H*HSe), 3.23 (dd, $J = 4.3$ Hz, $J = 8.9$ Hz, 1H; C*H*HSe), 3.29 (s, 3H; OCH_3), 3.77 (s, 3H; OCH_3), 4.80 (dd, $J = 3.3$ Hz, $J = 8.8$ Hz, 1H; C*H*CH$_2$Se), 5.04 (dt, $J = 3.5$ Hz, $J = 6.5$ Hz, 1H; CH), 6.83 (d, $J = 8.2$ Hz, 1H; arom. H), 6.98 (t, $J = 7.5$ Hz, 1H; arom. H), 7.15 (dt, $J = 7.4$ Hz, $J = 1.6$ Hz, 1H; arom. H), 7.21–7.30 (m, 2H; arom. H), 7.37 (dd, $J = 7.7$ Hz, $J = 1.7$ Hz, 1H; arom. H), 7.41 (dd, $J = 7.7$ Hz, $J = 1.6$ Hz, 1H; arom. H), 7.58 (dd, $J = 7.8$ Hz, $J = 1.3$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.3$ (q, CH_3), 31.0 (t, CH_2), 35.0 (t, CH_2Se), 55.2 (q, OCH_3), 57.3 (q, CHO*C*H$_3$), 74.5 (d, 2C; Ar*C*H), 110.4 (d), 120.7 (d), 126.2 (d), 126.3 (d), 127.2 (d), 127.7 (d), 128.6 (d), 128.9 (s), 129.9 (s), 133.6 (d), 145.9 (s) 156.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 3005$, 2966, 2935, 1600, 1588, 1488, 1464, 1287, 1161, 1095, 1048 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 380 (8) [M^+], 214 (9), 185 (6), 165 (10), 151 (100), 134 (9), 105 (6), 91 (14), 77 (8), 45 (23); HRMS calcd for $C_{19}H_{24}O_3Se$ [M^+] 380.0891, found 380.0894.

(*S*)-1-[2-{[(*R*)-{2-Methoxy-2-(2-methoxyphenyl)}ethyl]seleno}phenyl]-propyl methoxymethyl ether (11d): Yield: 83 %, light yellow oil; $[\alpha]_D^{25} = -153.3$ ($c = 0.80$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.00$ (t, 3H; $J = 7.3$ Hz, CH_3), 1.76 (q, $J = 6.7$ Hz, 2H; CH_2), 3.14 (d, $J = 5.8$ Hz, 1H;

C*H*HSe), 3.15 (d, $J = 2.1$ Hz, 1H; CH*H*Se), 3.30 (s, 3H; CH_2OCH_3), 3.36 (s, 3H; $CHOCH_3$), 3.79 (s, 3H; Ar–OCH_3), 4.51 (dd, $J = 6.6$ Hz, $J = 20.3$ Hz, 2H; OCH_2O), 4.87 (dd, $J = 4.4$ Hz, $J = 8.1$ Hz, 1H C*H*OCH_3), 5.05 (t, 1H; $J = 6.4$ Hz, ArCH), 6.86 (d, $J = 8.2$ Hz, 1H; arom. H), 6.97 (t, $J = 8.2$ Hz, 1H; arom. H), 7.14 (dt, $J = 7.4$ Hz, $J = 1.5$ Hz, 1H; arom. H), 7.20–7.28 (m, 2H; arom. H), 7.41 (dt, $J = 7.5$ Hz, $J = 1.8$ Hz, 2H; arom. H), 7.57 (dd, $J = 7.8$ Hz, $J = 1.3$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3); 30.4 (t, CH_2), 34.6 (t, CH_2Se), 55.2 (q, OCH_3), 55.6 (q, OCH_3), 57.3 (q, OCH_3), 77.3 (d, Ar–*C*H), 78.3 (d, Ar–*C*H), 94.7 (t, OCH_2O), 110.3 (d), 120.8 (d), 126.3 (d), 126.6 (d), 126.8 (d), 127.6 (d, 2C), 128.6 (d), 129.1 (s), 131.0 (s), 132.8 (d), 143.7 (s), 156.9 (s); IR ($CHCl_3$): $\tilde{\nu} = 3005$, 2936, 1600, 1588, 1488, 1464, 1212, 1158, 1097, 1031 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 424 (9) [M^+], 213 (16), 197 (7), 185 (6), 165 (12), 151 (100), 121 (7), 105 (6), 91 (13), 73 (32), 45 (43); HRMS calcd for $C_{21}H_{28}O_4Se$ [M^+] 424.1153, found 424.1152.

(*S*)-1-[{(*S*)-2-[(*R*)-(1-Methoxy-1-phenyl)propyl]selenyl}-6-methoxyphenyl]-ethanol (11e): Yield: 50%, colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta =$ 1.23 (d, $J = 7.1$ Hz, 3H; CH_3), 1.48 (d, $J = 6.5$ Hz, 3H; CH_3), 2.52 (s, 1H; OH), 3.27 (s, 3H; OCH_3), 3.48 (dq, $J = 7.1$ Hz, $J = 4.4$ Hz, 1H; CHSe), 3.92 (s, 3H; CH_3O), 4.38 (d, $J = 4.3$ Hz, 1H; C*H*OMe), 5.43 (q, $J = 6.5$ Hz, 1H; C*H*OH), 6.82 (dd, $J = 8.2$ Hz, $J = 1.1$ Hz, 1H; arom. H), 7.10–7.40 (m, 7H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 15.9$ (q, CH_3), 24.6 (q, CH_3), 44.4 (d, CHSe), 56.0 (q, OCH_3), 57.5 (q, OCH_3), 70.0 (d, CHOH), 86.1 (d, CH), 109.6 (d), 116.9 (s), 118.3 (d), 126.9 (d, 2C), 127.6 (d), 128.2 (d, 2C), 129.8 (d), 139.8 (s), 150.5 (s), 159.8 (s); MS (70 eV, EI): *m*/*z* (%): 380 (10) [M^+], 215 (100), 149 (71), 121 (80).

(*S*)-1-[{(*S*)-2-[(*R*)-(1-Methoxy-1-phenyl)propyl]selenyl}phenyl]propanol (11f): Yield: 89 mg, (61%); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.98$ (t, $J =$ 8.5 Hz, 3H; CH_2CH_3), 1.33 (d, $J = 7.0$ Hz, 3H; CH_3), 1.76 (quint, $J = 7.4$ Hz, 2H; CH_2CH_3), 2.58 (s, 1H; OH), 3.30 (s, 3H; OCH_3), 3.49 (dq, $J = 7.0$ Hz, $J = 4.4$ Hz, 1H; CHSe), 4.43 (d, $J = 4.7$ Hz, 1H; CHOMe), 5.10 (q, $J =$ 4.1 Hz, 1H; CHOH), 6.82 (dd, $J = 7.5$ Hz, $J = 1.5$ Hz, 1H; arom. H), 7.10–7.40 (m, 6H; arom. H), 7.48 (dd, $J = 7.7$ Hz, $J = 1.4$ Hz, 1H; arom. H), 7.56 (dd, $J = 7.7$ Hz, $J = 1.4$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta =$ 10.5 (q, CH_3), 16.5 (q, CH_3), 31.6 (t, CH_2), 46.6 (d, CHSe), 57.6 (q, OCH_3), 74.8 (d, CHOH), 86.3 (d, CH), 126.6 (d), 127.1 (d, 2C), 127.8 (d), 128.0 (d), 128.2 (d), 128.4 (d, 2C), 128.9 (s), 135.4 (d), 139.5 (s), 147.2 (s).

(2*S*,3*S*)-3-[2-((*S*)-1-Hydroxypropyl)phenyl]selenyl-2-phenyltetrahydrofuran (13c): See reference [10].

(2*S*,3*S*)-3-[1-[(*S*)-8-Hydroxy-2-methoxy-5,6,7,8-tetrahydronaphthyl]selenyl]2-phenyltetrahydrofuran (13f): Yield: 48%, colorless solid; $[\alpha]_D^{25} = -33.6$ ($c = 1.00$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.70-1.80$ (m, 2H; CH_2), 1.80–1.95 (m, 1H; CH_2), 1.99–2.17 (m, 2H; CH_2), 2.39 (dq, $J =$ 7.8 Hz, $J = 10.9$ Hz, 1H; CH_2), 2.53 (s, 1H; OH), 2.55–2.69 (m, 1H; CH_2), 2.72–2.81 (m, 1H; CH_2), 3.78 (s, 3H; OCH_3), 3.91 (dt, $J = 5.6$ Hz, $J =$ 7.9 Hz, 1H; CHSe), 4.05–4.20 (m, 2H; CH_2O), 4.73 (d, $J = 5.7$ Hz, 1H; ArCH), 5.10 (s, 1H; CHOH), 6.67 (d, $J = 8.5$ Hz, 1H; arom. H), 7.04 (d, $J =$ 8.5 Hz, 1H; arom. H), 7.06–7.10 (m, 2H; arom. H), 7.12–7.23 (m, 3H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.7$ (t, CH_2), 29.4 (t, CH_2), 31.5 (t, CH_2), 33.2 (t, CH_2) 45.7 (t, CH_2Se), 55.7 (q, OCH_3), 66.4 (d, CH), 67.9 (t, CH_2O), 87.1 (d, Ar*C*HO), 110.5 (d), 119.2 (s), 125.8 (d, 2C), 127.4 (d), 128.0 (d, 2C), 130.5 (s), 130.9 (d), 141.6 (s), 142.3 (s), 157.9 (s); IR ($CHCl_3$): $\tilde{\nu} =$ 3422, 2922, 2878, 1477, 1265, 1056, 924, 720, 699 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 404 (10) [M^+], 256 (59), 240 (100), 226 (13), 199 (8), 176 (37), 159 (36), 147 (73), 128 (23), 105 (79), 91 (80), 77 (46), 65 (10), 51 (12), 41 (19); HRMS calcd for $C_{21}H_{24}O_3Se$ [M^+] 404.0891, found 404.0887.

(*S*)-1-(2-{[3-[(2*R*,3*S*)-2-Phenyltetrahydrofuranyl]seleno}phenyl)propyl methoxymethyl ether (13g): Yield: 88%, light yellow oil; $[\alpha]_D^{25} = -87.1$ ($c = 1.07$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.94$ (t, $J = 7.4$ Hz, 3H; CH_3), 1.57–1.80 (m, 2H; CH_2), 2.05–2.18 (m, 1H; C*H*HCSe), 2.37–2.52 (m, 1H; CH*H*CSe), 3.35 (s, 3H; CH_2OCH_3), 3.54–3.65 (m, 1H; CHSe), 4.35 (dd, $J = 5.3$ Hz, $J = 8.1$ Hz, 1H; C*H*OCH_3), 4.48 (dd, $J = 6.7$ Hz, $J =$ 20.0 Hz, 2H; OCH_2O), 4.88 (d, $J = 6.0$ Hz, 1H; ArC*H*CHSe), 5.07 (dd, $J =$ 5.2 Hz, $J = 7.9$ Hz, 1H; ArCH), 7.07 (dt, $J = 1.6$, $J = 7.5$ Hz, 1H; arom. H), 7.22–7.34 (m, 6H; arom. H), 7.37 (dd, $J = 1.2$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.42 (dd, $J = 1.5$ Hz, $J = 7.8$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3); 30.7 (t, CH_2), 34.4 (t, CH_2), 48.5 (d, CHSe), 55.5 (q, OCH_3), 67.9 (t, OCH_2), 78.2 (d, Ar–*C*H), 85.0 (d, Ar–*C*HO), 94.4 (t, OCH_2O), 125.8 (d, 2C), 126.9 (d), 127.7 (d), 127.7 (d), 128.0 (d), 128.3 (d, 2C), 129.3 (s), 134.9 (d), 141.4 (s), 144.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 3063$, 3006, 2936, 2884, 2824, 1711, 1587, 1491, 1464, 1362, 1158, 1130, 1101, 1030, 917 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 406 (7) [M^+]$^+$, 227 (6), 213 (76), 198 (20), 185 (42), 146 (79), 105 (57), 91 (60), 77 (33), 65 (6), 45 (100); HRMS calcd for $C_{21}H_{26}O_3Se$ [M^+] 406.1047, found 406.1057.

(2*S*,3*S*)-3-[(*S*)-8-Methoxymethoxy-2-methoxy-5,6,7,8-tetrahydronaphthalen-1-ylselenyl]-2-phenyltetrahydrofuran (13k): Yield: 67%, colorless oil; $[\alpha]_D^{25} = -105.1$ ($c = 0.55$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.60-$ 1.80 (m, 2H; CH_2), 1.87–2.10 (m, 2H; CH_2), 2.35–2.48 (m, 2H; CH_2), 2.55–2.68 (m, 1H; CH), 2.72–2.81 (m, 1H; CH_2), 3.41 (s, 3H; OCH_3), 3.64 (s, 3H; $ArOCH_3$), 3.93 (dt, $J = 5.6$ Hz, $J = 7.9$ Hz, 1H; CHSe), 4.05–4.24 (m, 2H; CH_2O), 4.65 (d, $J = 5.5$ Hz, 1H; ArCH), 4.73 (d, $J = 6.8$ Hz, 1H; OC*H*HO), 5.03 (t, $J = 3.0$ Hz, 1H; C*H*OH), 5.14 (d, $J = 5.5$ Hz, 1H; OCH*H*O), 6.62 (d, $J = 8.5$ Hz, 1H; arom. H), 6.94–7.03 (m, 3H; arom. H), 7.10–7.16 (m, 3H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.5$ (t, CH_2), 29.0 (t, CH_2), 29.7 (t, CH_2), 32.8 (t, CH_2) 44.8 (t, CH_2Se), 55.6 (q, OCH_3), 55.9 (q, ArOCH_3), 68.0 (t, CH_2O), 75.5 (d), 87.8 (d, Ar*C*HO), 97.4 (d, OCH_2O), 110.7 (d), 120.4 (s), 125.9 (d, 2C), 127.2 (d), 127.9 (d, 2C), 130.5 (s), 130.8 (d), 140.8 (s), 141.6 (s), 157.7 (s); IR ($CHCl_3$): $\tilde{\nu} = 2934$, 1494, 1149, 1095, 1033, 918, 753, 700 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 448 (7) [M^+], 269 (6), 255 (100), 240 (69), 226 (6), 160 (26), 147 (32), 128 (12), 105 (79), 115 (23), 91 (38), 77 (18), 45 (39); HRMS calcd for $C_{23}H_{28}O_4Se$ [M^+] 448.1152, found 448.1158.

(2*S*,3*S*)-3-[2-((*S*)-1-Hydroxypropyl)phenyl]selenyl-2-phenyl-3*H*-dihydrofuranone (15c): See reference [10].

(5*S*,4*S*)-4-[2-[(*S*)-1-Methoxymethoxypropyl]phenyl]selenyl-5-phenyl-3*H*-dihydrofuranone (15g): Yield: 50%, light yellow oil; $[\alpha]_D^{25} = -78.3$ ($c =$ 0.81, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.93$ (t, 3H; $J = 7.3$ Hz, CH_3), 1.57–1.80 (m, 2H; CH_2), 2.73 (dd, $J = 8.0$ Hz, $J = 18.1$ Hz, 1H; C*H*HCO), 3.06 (dd, $J = 8.3$ Hz, $J = 18.0$ Hz, 1H; CH*H*CO), 3.29 (s, 3H; OCH_3), 3.80 (dt, $J = 6.7$ Hz, $J = 8.2$ Hz, 1H; CHSe), 4.40 (d, $J = 6.8$ Hz, 1H; OC*H*HO), 4.53 (d, $J = 6.8$ Hz, 1H; OCH*H*O), 5.08 (dd, $J = 5.1$ Hz, $J =$ 8.0 Hz, 1H; ArCHO), 5.46 (d, $J = 6.8$ Hz, 1H; CHO), 7.10 (dt, $J = 1.6$ Hz, $J = 7.5$ Hz, 1H; arom. H), 7.22–7.40 (m, 7H; arom. H), 7.46 (dd, $J = 1.7$ Hz, $J = 8.2$ Hz, 1H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3); 30.8 (t, CH_2), 36.5 (t, CH_2), 43.0 (d, CHSe), 55.5 (q, OCH_3), 78.2 (d, Ar–*C*H), 86.2 (d, Ar–*C*HO), 94.3 (t, OCH_2O), 125.5 (d), 125.7 (d, 2C), 127.1 (s), 127.4 (d), 128.1 (d), 128.7 (d, 2C), 128.9 (s), 129.3 (d), 136.2 (d), 137.4 (s), 145.6 (s), 174.6 (s, CO); IR ($CHCl_3$): $\tilde{\nu} = 3006$, 2968, 2934, 1782, 1711, 1491, 1464, 1362, 1158, 1136, 1101, 1030, 975 cm^{-1}.

(*R*,*S*)-4-Methoxy-3-[2-(*S*)-(1-methoxymethoxypropyl)phenylselenyl]-4-phenylbutyric acid (16): Yield: 84%, colorless oil; $[\alpha]_D^{25} = -91.3$ ($c = 1.06$, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.96$ (t, $J = 7.4$ Hz, 3H; CH_3), 1.53–1.68 (m, 2H; CH_2CH_3), 2.76–2.87 (m, 2H; CH_2COOH), 3.33 (s, 3H; $CHOCH_3$), 3.40 (s, 3H; CH_2OCH_3), 3.63 (m, 1H; CHSe), 4.63 (m, 3H; C*H*OCH_3, OCH_2O), 5.07 (dd, $J = 4.7$ Hz, $J = 7.7$ Hz, 1H; ArCHO), 7.07–7.39 (m, 9H; arom. H), 8.4–8.9 (br s, 1H; COOH); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.3$ (q, CH_3), 31.7 (t, CH_2), 37.0 (t, CH_2CHSe), 51.0 (t, CH_2Se), 55.1 (q, OCH_3), 57.6 (q, OCH_3), 78.2 (d, Ar–CH), 86.4 (d, Ar–*C*H), 93.8 (t, OCH_2O), 126.0 (d), 127.3 (d, 2C), 127.7 (d), 128.0 (d), 128.1 (d), 128.4 (d, 2C), 128.7 (s), 130.8 (s), 135.7 (d), 138.9 (d), 144.8 (s); IR ($CHCl_3$): $\tilde{\nu} = 3060$, 2933, 1709, 1454, 1216, 1157, 1100, 1030, 919, 757, 702 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 452 (3) [M^+], 213 (17), 198 (52), 183 (50), 162 (19), 147 (46), 121 (100), 105 (28), 91 (52), 77 (37), 63 (15), 45 (85); HRMS calcd for $C_{22}H_{28}O_5Se$ [M^+] 452.1102, found 452.1109.

(*R*)-2-Phenyl-2-[1-{2-((*S*)-1-hydroxypropyl)phenylselenyl}methyl]tetrahydrofuran (18c): See reference [10].

(*R*)-2-Phenyl-2-[1-{2-((*S*)-1-methoxymethoxypropyl) phenylselenyl}methyl]tetrahydrofuran (18g): Yield: 92%, light yellow oil; $[\alpha]_D^{25} = -66.9$ ($c =$ 1.43, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.94$ (t, 3H; $J = 7.2$ Hz, CH_3), 1.63–1.86 (m, 3H; CH_2), 1.94–2.10 (m, 1H; CH_2), 2.21–2.41 (m, 2H; CH_2), 3.32 (t, $J = 12.0$ Hz, 1H; C*H*HCSe), 3.35 (s, 3H; CH_2OCH_3), 3.43 (t, $J = 12.0$ Hz, 1H; CH*H*Se), 3.92 (dt, $J = 6.0$ Hz, $J = 8.0$ Hz, 1H; COC*H*H), 4.05 (dt, $J = 7.2$ Hz, $J = 8.1$ Hz, 1H; COCH*H*), 4.46 (d, $J =$ 6.6 Hz, 1H; OC*H*HO), 4.52 (d, $J = 6.6$ Hz, 1H; OCH*H*O), 4.99 (dd, $J =$ 5.7 Hz, $J = 7.1$ Hz, 1H; ArCHO), 7.10 (dt, $J = 1.6$ Hz, $J = 7.4$ Hz, 1H; arom. H), 7.19–7.28 (m, 2H; arom. H), 7.30–7.48 (m, 6H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 10.4$ (q, CH_3), 26.0 (t, CH_2), 30.6 (t, CH_2), 37.6 (t, CH_2), 41.9 (d, CHSe), 55.6 (q, OCH_3), 68.2 (t, OCH_2), 78.3 (d, Ar–*C*H), 86.1 (d, Ar–*C*HO), 94.6 (t, OCH_2O), 125.2 (d, 2C), 126.5 (d), 126.9 (d), 127.1 (d), 127.7 (d), 128.1 (d, 2C), 130.8 (s), 133.5 (d), 144.0 (s), 145.7 (s); IR

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($CHCl_3$): $\tilde{\nu}$ = 3006, 2935, 1710, 1454, 1268, 1157, 1099, 1030, 910 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 420 (1) [M^+], 197 (5), 147 (100), 105 (25), 91 (17), 73 (13), 45 (26); HRMS calcd for $C_{22}H_{28}O_3Se$ [M^+] 420.1204, found 420.1198.

(*R*)-5-Phenyl-5-[1-{2-((*S*)-1-hydroxypropyl)phenylselenyl}methyl]-3*H*-dihydrofuran-2-one (20c): see reference [10].

(*R*)-5-Phenyl-5-[1-{2-((*S*)-1-methoxymethoxypropyl)phenylselenyl}methyl]-3*H*-dihydrofuran-2-one (20g): Yield: 80%, colorless oil; $[\alpha]_D^{25} = -68.8$ (c = 1.04, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.95 (t, 3H; J = 7.4 Hz, CH_3), 1.58–1.81 (m, 3H; CH_2), 2.41–2.75 (m, 4H; CH_2), 3.35 (s, 3H; OCH_3), 3.40 (d, J = 12.8 Hz, 1H; C*H*HSe), 3.50 (d, J = 12.8 Hz, 1H; CH*H*Se), 4.47 (d, J = 6.76 Hz, 1H; OC*H*HO), 4.55 (d, J = 6.8 Hz, 1H; OCH*H*O), 5.02 (dd, J = 4.9 Hz, J = 7.7 Hz, 1H; ArCHO), 7.14 (dt, J = 1.6 Hz, J = 7.4 Hz, 1H; arom. H), 7.25–7.49 (m, 8H; arom. H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 10.4 (q, CH_3), 28.9 (t, CH_2), 30.7 (t, CH_2), 33.4 (t, CH_2), 40.8 (d, CHSe), 55.6 (q, OCH_3), 78.1 (d, Ar–*C*H), 88.2 (d, Ar–*C*HO), 94.4 (t, OCH_2O), 124.9 (d, 2C), 126.9 (d); 128.0 (d, 2C), 128.1 (d), 128.6 (d), 129.6 (s), 134.0 (d), 142.1 (s), 144.3 (s), 175.7 (s, CO); IR ($CHCl_3$): $\tilde{\nu}$ = 2932, 2885, 1780, 1462, 1448, 1157, 1101, 1030, 918, 764, 702 cm^{-1}; MS (70 eV, EI): *m*/*z* (%): 434 (8) [M^+], 227 (6), 213 (68), 197 (19), 185 (52), 161 (100), 131 (13), 105 (30), 91 (41), 77 (23), 55 (8), 45 (74); HRMS calcd for $C_{22}H_{26}O_4Se$ [M^+] 434.0996, found 434.0994.

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