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Preparation of Novel Chiral Non-Racemic Diselenides and Applications in Asymmetric Synthesis

Liwei Zhao,^[a,b] Zhong Li,^[b] and Thomas Wirth*^[a]

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New chiral non-racemic diselenides were prepared and their corresponding selenium electrophiles were used for the stereoselective functionalization of alkenes. The influence of

reaction was studied.

Results and Discussion

different nucleophiles on the outcome of the selenenylation

A common characteristic of the optically active diselen-

ides reported in the literature is the close proximity of an

electron rich heteroatom such as oxygen, nitrogen or sulfur.

It has been shown that selenium can interact with such

nearby heteroatoms. If the heteroatom is connected to an

adjacent benzylic position, intermediate species such as 2

(Figure 1) are generated. Increasing the distance between

the heteroatom and the selenium will result in different interactions and in conformationally more flexible intermedi-

ates. The six-membered intermediates **3** lead to lower selectivities in the corresponding selenenylation reactions as we

have reported earlier.^[5] The success of larger ring intermedi-

ates in related electrophilic iodine-based reagents^[6]

prompted us to investigate structures of type **4** forming seven-membered intermediates as shown in Figure 1. We have, however, no direct evidence for this interaction nor

have we prepared the corresponding compounds replacing

the oxygen atoms with methylene groups.

Introduction

The stereoselective functionalization of non-activated carbon-carbon double bonds is one of most interesting topics in organic chemistry, and electrophilic selenenylation reactions of alkenes have been successfully studied for several years.^[1] In a stereoselective approach to this reaction, other research groups and we have investigated stereoselective reactions of alkenes with chiral selenium electrophiles.^[2,3] The general reaction is illustrated in Scheme 1. A variety of nucleophiles have been used to open the seleniranium intermediates 1 and the addition products gave rise to a lot of subsequent reactions (Scheme 1). However, many of the reported chiral diselenides required a rather long synthetic route for their preparations, sometimes accompanied with low overall yields, or expensive reagents.



Scheme 1. Selenenylation of alkenes.

Recently, we have reported easily accessible optically active diselenides bearing a sulfoxide moiety as the chiral centre.^[4] Herein, we describe a series of novel chiral diselenides prepared from easily available, cheap starting materials in a short and efficient synthesis and the addition reactions of their corresponding selenium electrophiles to alkenes.

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Figure 1. Interaction of heteroatoms with selenium electrophiles.

The key reaction for the formation of precursors 6 is the Mitsunobu reaction of the corresponding phenols 5 with (–)-ethyl lactate to generate the desired chiral products in high yields. The ester moiety can be reduced by lithium aluminium hydride to produce alcohol 7 almost quantitatively, which is then protected by a methyl group to yield the chiral ether **8**. After *ortho*-lithiation, selenium is introduced and after an oxidative work-up, diselenides **9a**, **9b** and triselenide **10** are formed. Triselenide **10** is reduced and reoxidized

[[]a] School of Chemistry, Cardiff University, Park Place, Cardiff, CF10 3AT, U.K. Fax: +44-29-20876968 E-mail: wirth@cf.ac.uk

[[]b] East China University of Science and Technology, Shanghai, China

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to diselenide 9c (Scheme 2). It has already been observed in the past that sterically demanding compounds can produce mixtures of diselenides and triselenides.^[7]



Scheme 2. Synthesis of new diselenides 9.

These three chiral diselenides **9** have then been used in the methoxyselenenylation of styrene. After conversion of the diselenides into the corresponding selenenyl triflates the methoxyselenenylation of styrene was performed at -78 °C as shown in Scheme 3. These reactions were complete after 2 h reaction time and the products isolated by flash chromatography. The mixtures of diastereomers could not be completely separated, but enriched by flash chromatography. The diastereomeric ratios (*dr*) were determined by integration of ¹H NMR signals in the crude reaction mixture. All these chiral diselenides gave the addition products in good yields and with high selectivites as shown in Table 1.

From these experiments, the C_2 -symmetric chiral diselenide **9c** showed the highest selectivity. Interestingly, the reaction using the methoxy-substituted derivative **9b** showed lower selectivity than the unsubstituted compound **9a** (Table 1, entries 1 and 2). This is noteworthy, because we

Table 1. Methoxyselenenylation of styrene using diselenides 9.

Diselenide	Yield [%]	$dr^{[a]}$
9a	12a : 40	92: 8
9b	12b: 72	87.5:12.5
9c	12c : 68	94.5:5.5 ^[b]
	Diselenide 9a 9b 9c	Diselenide Yield [%] 9a 12a: 40 9b 12b: 72 9c 12c: 68

[a] Determined with ¹H NMR spectroscopy. [b] 96:4 dr after column determined by HPLC.

and others have investigated such a directing effect of a methoxy-substituent in the second *ortho*-position to the selenium electrophile and usually an increase in selectivity was observed.^[5,8] An explanation for this unusual drop in selectivity cannot be given at present, especially as in comparison with **9c** the selectivities should not be dependent on the electronic properties of the selenium electrophiles.

Diselenide 9c as the most promising reagent in this series was then used for several other selenenylation reactions as shown in Scheme 4 and Table 2.



Scheme 4. Selenenylation of styrene derivatives using selenium electrophiles generated from diselenide **9c**.

Table 2. Alkoxyselenenylation of styrene derivatives using diselenide **9c**.

Entry	Ar, \mathbb{R}^1 , \mathbb{R}^2	ROH	Yield [%]	$dr^{[a]}$
1	Ph, H, H	MeOH	12c: 68	94.5:5.5 ^[b]
2	2-Me-C ₆ H ₄ , H, H	MeOH	13a: 54	95.5:4.5 ^[c]
3	2-Cl-C ₆ H ₄ , H, H	MeOH	13b : 61	91:9
4	3-Me-C ₆ H ₄ , H, H	MeOH	13c: 44	91.5:8.5
5	4-MeO-C ₆ H ₄ , H, H	MeOH	13d: 32	67:33
6	Ph, H, Me	MeOH	13e : 0	_
7	Ph, Me, H	MeOH	13f: 65	75:25
8	Ph, H, H	EtOH	13g: 54	92:8
9	Ph, H, H	<i>i</i> PrOH	13h : 48	78:22
10	Ph, H, H	tBuOH	13i : 34	60:40

[[]a] Determined with ¹H NMR spectroscopy. [b] 96:4 dr after column determined by HPLC. [c] >97:3 dr after column determined by HPLC.



Scheme 3. Methoxyselenenylation of styrene with selenium electrophiles generated from diselenides 9.

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The substitution pattern of the different styrene derivatives has a large influence on the selectivity of the reaction. Whereas substituents in ortho- and meta-position are tolerated, an electron-rich derivative with a methoxy moiety in the *para*-position leads to a largely reduced selectivity in the methoxyselenenylation reaction. The reduced selectivities with bulkier nucleophiles are in line with previous experiments. Unlike previous selenium electrophiles, the electrophile generated from 9c does not react with disubstituted alkenes. No product formation was observed with β -methylstyrene (Table 2, entry 6) and also α -methylstyrene showed only low selectivities (Table 2, entry 7). Also various attempts to use the electrophile generated from 9c in cyclization reactions failed, both in stoichiometric as well as in catalytic reactions.^[9] This seems to be an interesting feature of these electrophiles, which has not yet been observed. It cannot be attributed to the steric congestion around the selenium electrophile as other C_2 -symmetric compounds are known to successfully react with substituted alkenes.^[10] The reason for their reactivity only towards terminal double bonds will be investigated further.

Conclusions

In conclusion, we prepared three novel chiral diselenides in an efficient synthesis using cheap starting materials. The corresponding electrophiles have been investigated in stereoselective selenenylation reactions, in which the C_2 symmetrical derivative showed the highest selectivities. Further experiments to improve the stereoselectivity of these and related processes and to gain additional insight into the observed selectivities are in progress.

Experimental Section

General: Melting points were obtained in open capillary tubes. ¹H NMR and ¹³C NMR spectra were recorded on a AV-400 Bruker using the solvents indicated with 400 and 100 MHz, respectively. Mass spectra (m/z) and HRMS were recorded under the conditions of electron impact (EI) and electrospray (ES) and chemical ionization (CI). All reactions were monitored by thin-layer chromatography that was performed on precoated sheets of silica gel 60, and flash column chromatography was performed with silica gel 60 (Merck, 230–400 mesh). Eluting solvents are indicated in the text. All experiments were performed under an inert atmosphere of argon. Dichloromethane was dried with CaH₂, methanol, ethanol, 2-propanol and *tert*-butanol were dried with 4-Å molecular sieves. All other purchased chemicals were used without further purification.

(*R*)-Ethyl 2-Phenoxypropanoate (6a):^[11] To a solution of phenol (0.94 g, 10 mmol), PPh₃ (3.93 g, 15 mmol) and ethyl (–)-lactate (1.77 mL, 15 mmol) in THF (25 mL), diisopropyl azodicarboxylate (2.0 M in toluene, 15 mmol, 7.5 mL) was added slowly at 0 °C, then the reaction mixture was warmed up to room temperature. After stirring overnight, the resulting mixture was concentrated in vacuo, and the residue was purified by flash column chromatography on silica gel (hexane/EtOAc, 25:1) to give **6a** in 91% yield (1.76 g, 9.07 mmol) as a colorless oil. $[a]_{D}^{25} = +39.8$ (c = 0.96, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.27$ (t, J = 7.1 Hz, 3 H, CH₂CH₃),

1.27 (t, J = 7.1 Hz, 3 H, CHC H_3), 4.24 (q, J = 7.1 Hz, 2 H, CH₃C H_2), 4.77 (q, J = 6.8 Hz, 1 H, CH₃CH), 6.91 (d, J = 8.8 Hz, 2 H, ArH), 6.99 (t, J = 7.0 Hz, 1 H, ArH), 7.30 (t, J = 7.8 Hz, 2 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 14.6$, 19.0, 61.7, 73.0, 115.5, 121.9, 129.2, 158.0, 172.7 ppm. HRMS: m/z calcd. for C₁₁H₁₅O₃ [M + H]⁺: 195.1016; found 195.1015. IR (neat): $\tilde{v} = 3097$, 3063, 3042, 2986, 2938, 2908, 2870, 1753, 1734, 1600, 1589 1495, 1446, 1376, 1344, 1274, 1241, 1196, 1154, 1135, 1098, 1050, 1020, 946, 885, 860, 802, 754, 690 cm⁻¹.

(R)-Ethyl 2-(3-Methoxyphenoxy)propanoate (6b):^[12] Similar reaction as described for 6a using: 3-methoxyphenol (2.48 g, 20 mmol), PPh₃ (7.68 g, 30 mmol), ethyl (-)-lactate (3.54 mL, 30 mmol) in THF (50 mL), diisopropyl azodicarboxylate (2.0 m in toluene, 30 mmol, 15 mL). Product 6b was isolated in 86% yield (1.76 g, 7.86 mmol) as a colorless oil. $[a]_{D}^{25} = +25.7$ (c = 1.13, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.27$ (t, J = 7.1 Hz, 3 H, CH₂CH₃), 1.63 (d, J = 6.8 Hz, 3 H, CHCH₃), 3.79 (s, 3 H, OCH₃), 4.24 (q, J= 7.1 Hz, 2 H, CH₃CH₂), 4.75 (q, J = 6.8 Hz, 1 H, CH₃CH), 3.45-3.50 (m, 2 H, ArH), 6.55 (dd, J = 2.3, J = 8.2 Hz, 1 H, ArH), 7.18 (t, J = 8.2 Hz, 1 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta =$ 14.6, 19.0, 55.6, 61.7, 72.9, 77.3, 77.6, 77.9, 102.0, 107.1, 107.8, 130.3, 159.3, 161.3, 172.5 ppm. HRMS: m/z calcd. for C₁₂H₁₇O₄ $[M + H]^+$: 225.1121; found 225.1118. IR (neat): $\tilde{v} = 3410, 2985,$ 2942, 2927, 2838, 2360, 2341, 1754, 1733, 1603, 1492, 1454, 1379, 1284, 1268, 1200, 1154, 1094, 1049 cm^{-1} .

(2*R*,2'*R*)-Diethyl 2,2'-(1,3-Phenylene)bis(oxy)dipropanoate (6c):^[13] Similar reaction as described for 6a using: resorcinol (2.2 g, 20 mmol), PPh₃ (13.1 g, 50 mmol), ethyl (-)-lactate (5.9 mL, 50 mmol) in THF (100 mL), diisopropyl azodicarboxylate (2.0 M in toluene, 50 mmol, 25 mL). Product 6c was isolated in 90% yield (5.56 g, 17.94 mmol) as a colorless oil. $[a]_{D}^{25} = +46.9$ (c = 1.16, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.26$ (t, J = 7.1 Hz, 6 H, CH₂CH₃), 1.60 (d, J = 6.8 Hz, 6 H, CHCH₃), 4.22 (q, J = 7.2 Hz, 4 H, CH₃CH₂), 4.71 (q, J = 6.8 Hz, 2 H, CH₃CH), 6.45–6.50 (m, 3 H, Ar*H*), 7.15 (t, J = 8.2 Hz, 1 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 14.5$, 18.9, 61.7, 73.0, 103.4, 108.4, 130.4, 159.2, 172.5 ppm. HRMS: m/z calcd. for C₁₆H₂₃O₆ [M + H]⁺: 311.1495; found 311.1485. IR (neat): $\tilde{v} = 2987$, 2947, 2842, 2798, 1754, 1720, 1603, 1491, 1447, 1376, 1282, 1180, 1157, 1133, 1095, 1051, 1017, 858, 766, 685 cm⁻¹.

(R)-2-Phenoxypropan-1-ol (7a):^[14] LiAlH₄ (532 mg, 14 mmol) was suspended in dry THF (15 mL), cooled to -78 °C, and 6a (1.70 g, 9.3 mmol) in dry THF (25 mL) was added slowly. After the addition, the mixture was warmed to 0 °C, kept at that temperature for 2 h, and then stirred overnight at room temperature. The reaction was quenched by 1 M HCl solution (20 mL) at 0 °C, and extracted with EtOAc (3×50 mL). The combined organic phases were dried with anhydrous MgSO₄, and the solvent was removed under reduced pressure to give 7a as a colorless oil in 96% yield (1.37 g, 9.01 mmol). $[a]_D^{25} = -49.0 (c = 1.35, \text{ CHCl}_3)$. ¹H NMR (CDCl₃, 400 MHz): δ = 1.17 (d, J = 6.3 Hz, 3 H, CHCH₃), 2.21 (s, 1 H, OH), 3.60-3.67 (m, 2 H, CH₃CH₂), 4.37-4.44 (m, 1 H, CH₃CH), 6.83–6.89 (m, 3 H, ArH), 7.14–7.24 (m, 2 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 16.3, 66.7, 75.1, 116.5, 121.6, 130.0, 158.1 ppm. HRMS: m/z calcd. for C₉H₁₂O₂NH₄ [M + NH_4]⁺: 170.1176; found 170.1173. IR (neat): $\tilde{v} = 3390, 2980, 2933,$ 2910, 1598, 1587, 1495, 1376, 1291, 1241, 1174, 1051, 932, 876, 752, 692 $\rm cm^{-1}$.

(*R*)-2-(3-Methoxyphenoxy)propan-1-ol (7b): Similar reaction as described for 7a using: LiAlH₄ (86 mg, 2.25 mmol) in dry THF (10 mL), 6b (345 mg, 1.5 mmol) in dry THF (5 mL). Product 7b



was isolated in 96% yield (262 mg, 1.44 mmol) as a light gray oil. $[a]_{D}^{25} = -38.6 (c = 1.33, CHCl_3)$. ¹H NMR (CDCl₃, 400 MHz): $\delta =$ 1.09 (d, J = 6.2 Hz, 3 H, CHCH₃), 2.07 (s, 1 H, OH), 3.53–3.60 (m, 5 H, OCH₃, CH₂OH), 4.27–4.34 (m, 1 H, CH₃CH), 6.31–6.37 (m, 3 H, ArH), 7.00 (t, J = 8.1 Hz, 1 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 16.3$, 55.7, 66.5, 75.1, 103.0, 107.0, 108.5, 130.4, 159.4, 161.3 ppm. HRMS: m/z calcd. for C₁₀H₁₅O₃ [M + H]⁺: 183.1016; found 183.1014. IR (neat): $\tilde{v} = 3408$, 2974, 2937, 2883, 2836, 2360, 1738, 1602, 1492, 1454, 1378, 1334, 1285, 1265, 1200, 1154, 1048, 976, 900, 838, 764, 688 cm⁻¹.

(2*R*,2'*R*)-2,2'-[1,3-Phenylenebis(oxy)]bis(propan-1-ol) (7c): Similar reaction as described for 7a using: LiAlH₄ (86 mg, 2.25 mmol) in dry THF (10 mL), 6c (1.3 g, 4.2 mmol) in dry THF (8 mL). Product 7c was isolated in 95% yield (900 mg, 3.98 mmol) as a colorless oil. $[a]_{D}^{25} = -59.0$ (c = 0.40, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.18$ (d, J = 6.2 Hz, 6 H), 2.34 (s, 2 H), 3.59–3.67 (m, 4 H), 4.39 (d p, J = 4.0, J = 6.3 Hz, 2 H), 6.45–6.47 (m, 3 H), 7.06–7.10 (m, 1 H) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 16.3$, 66.6, 75.2, 104.9, 109.0, 130.5, 159.4 ppm. HRMS: m/z calcd. for C₁₂H₁₉O₄ [M + H]: 227.1278; found 227.1279. IR (neat): $\tilde{v} = 3375$, 2970, 2934, 2865, 2373, 2343, 2328, 2313, 1600, 1589, 1488, 1447, 1373, 1282, 1268, 1181, 1157, 1051, 1000, 985 cm⁻¹.

(R)-1-(1-Methoxypropan-2-yloxy)benzene (8a):^[15] Sodium hydride (320 mg, 8 mmol, 60% in mineral oil) was washed with dry THF $(2 \times 12 \text{ mL})$, then dry THF (8 mL) was added. A solution of 7a (600 mg, 4 mmol) in dry THF (4 mL) was added slowly at 0 °C, and the reaction mixture stirred for 30 min. Methyl iodide (1.14 g, 0.5 mL, 8 mmol) was added and the reaction mixture was slowly warmed up to room temperature and stirred overnight. The reaction was quenched by the addition of saturated NaHCO₃ and extracted with EtOAc $(3 \times 30 \text{ mL})$. The combined organic phases were dried with anhydrous MgSO₄, and the solvent was removed under reduced pressure to give a colorless oil in 84% yield (560 mg, 3.37 mmol). $[a]_{D}^{25} = -9.6$ (c = 1.35, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.17$ (d, J = 6.3 Hz, 3 H, CHCH₃), 3.27 (s, 3 H, OCH_3), 3.34 (dd, J = 4.5, J = 10.1 Hz, 1 H ppm. CH₃OCHH), $3.45 (dd, 1 H, J = 5.8 Hz, J = 10.1 Hz, CH_3OCHH), 4.34-4.46 (m, J)$ 1 H, CH₃CH), 6.78–6.80 (m, 3 H, ArH), 7.10–7.15(m, 2 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.3, 59.8, 73.2, 76.3, 116.5, 121.3, 123.0, 158.2 ppm. HRMS: m/z calcd. for $C_{10}H_{14}O_2NH_4 [M + NH_4]^+$: 184.1332; found 184.1330. IR (neat): $\tilde{v} = 3297, \, 2925, \, 2910, \, 1598, \, 1587, \, 1494, \, 1455, \, 1376, \, 1290, \, 1243,$ 1202, 1152, 1115, 1026, 999, 971, 885, 752 cm⁻¹.

(*R*)-1-Methoxy-3-(1-methoxypropan-2-yloxy)benzene (8b): Similar reaction as described for 8a using: sodium hydride (528 mg, 13.2 mmol, 60% in mineral oil), 7b (1.2 g, 6.6 mmol) in dry THF (8 mL), methyliodide (1.89 g, 0.83 mL, 13.2 mmol). Product 8b was isolated in 82% yield (1.06 g, 5.41 mmol) as a colorless oil. $[a]_D^{25} = -10.0 (c = 0.3, CHCl_3)$. ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.34$ (d, J = 6.3 Hz, 3 H, CHCH₃), 3.44 (s, 3 H, OCH₃), 3.50 (dd, J = 4.5, J = 10.1 Hz, 1 H, CH₃OCH*H*), 3.61 (dd, J = 5.7, J = 10.1 Hz, 1 H, CH₃OCH*H*), 3.61 (dd, J = 5.7, J = 10.1 Hz, 1 H, CH₃OCH*H*), 3.61 (dd, J = 5.7, J = 10.1 Hz, 1 H, CH₃OCH*H*), 3.80 (s, 3 H, ArOCH₃), 4.52–4.59 (m, 1 H, CH₃CH), 6.51–6.56 (m, 3 H, ArH), 7.17–7.21 (m, 1 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.2$, 55.7, 59.8, 73.2, 76.2, 102.8, 107.0, 108.4, 130.3, 159.5, 161.2 ppm. HRMS: *m*/*z* calcd. for C₁₁H₁₇O₃ [M + H]⁺: 197.1172; found 197.1170. IR (neat): $\tilde{v} = 2990$, 2931, 2879, 2835, 2360, 2328, 1601, 1491, 1453, 1375, 1285, 1265, 1202, 1147, 1113, 1041, 997, 982, 835, 763 cm⁻¹.

1,3-Bis{[(*R***)-1-methoxypropan-2-yl]oxy}benzene (8c):** Similar reaction as described for **8a** using: sodium hydride (640 mg, 16 mmol, 60% in mineral oil), **7c** (0.9 g, 3.98 mmol) in dry THF (4 mL),

methyl iodide (2.27 g, 1 mL, 16 mmol). Product **8b** was isolated in 83% yield (840 mg, 3.31 mmol) as a light yellow oil. $[a]_D^{25} = -16.3$ (c = 1.20, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.17$ (d, J =6.3 Hz, 1 H), 3.27 (s, 6 H), 3.33 (dd, J = 4.5, J = 10.1 Hz, 1 H), 3.44 (dd, J = 5.7, J = 10.1 Hz, 1 H), 4.38 (sext, J = 6 Hz, 2 H), 6.38–6.41 (m, 3 H), 7.00 (t, J = 8.2 Hz, 1 H) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.2$, 59.7, 73.2, 104.7, 108.8, 130.2, 159.5 ppm. HRMS: m/z calcd. for C₁₄H₂₃O₄ [M + H]: 255.1591; found 255.1588. IR (neat): $\tilde{v} = 2979$, 2930, 2879, 2832, 2813, 1600, 1589, 1489, 1452, 1375, 1354, 1331, 1283, 1263, 1203, 1183, 1143, 1113, 1008, 969, 906, 838, 765, 689 cm⁻¹.

(R)-2-[(1-Methoxypropan-2-yl)oxy]phenyl Diselenide (9a): To a solution of 8a (240 mg, 1.45 mmol) in dry THF (10 mL), n-butyllithium (0.88 mL, 2.5 M solution in hexane, 2.18 mmol) was added slowly at 0 °C. The mixture was stirred for 2.5 h at this temperature. Then selenium powder (138 mg, 1.74 mmol) was added with vigorous stirring at 0 °C. After 15 h stirring at room temperature the mixture was quenched with 1 M HCl (15 mL) and extracted with EtOAc $(3 \times 25 \text{ mL})$. The combined organic phases were dried with MgSO₄ and the solvent was removed in vacuo. After column chromatography (hexane/ethyl acetate, 8:1) the diselenide 9a was isolated as yellow oil in 50% yield (178 mg, 0.36 mmol). $[a]_{D}^{25} =$ $-25.7 (c = 0.60, \text{CHCl}_3)$. ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.32 (d, d)$ J = 6.3 Hz, 6 H, CHCH₃), 3.38 (s, 6 H, OCH₃), 3.47 (dd, J = 4.7, J = 10.3 Hz, 2 H, CH₃OCHH), 3.60 (dd, J = 6.0, J = 10.3 Hz, 2 H, CH₃OCH*H*), 4.50–4.58 (m, 2 H, CH₃C*H*), 6.76–6.83 (m, 4 H, ArH), 7.07–7.11 (m, 2 H, ArH), 7.43 (dd, J = 1.6, J = 7.8 Hz, 2 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.8, 60.0, 75.5, 76.4, 113.9, 120.8, 122.8, 128.3, 130.6, 155.7 ppm. ⁷⁷Se NMR (57 MHz, CDCl₃): δ = 327 ppm. HRMS: *m*/*z* calcd. for C₂₀H₂₆O₄ 74 Se₁ 76 Se₁NH₄ [M + NH₄]⁺: 498.0586; found 498.0590. IR (neat): $\tilde{v} = 3400, 2980, 2925, 2911, 1574, 1465, 1441, 1270, 1233, 1110,$ 1029, 976, 747 cm⁻¹.

(*R*)-6-Methoxy-2-[(1-methoxypropan-2-yl)oxy]phenyl Diselenide (9b): Similar reaction as described for 9a using: 8b (540 mg, 2.76 mmol) in dry THF (12 mL); n-butyllithium (1.66 mL, 2.5 м solution in hexane, 4.14 mmol); selenium powder (262 mg, 3.32 mmol). The diselenide 9b was isolated as yellow oil in 65% yield (492 mg, 0.90 mmol). $[a]_D^{25} = +5.53$ (c = 3.0, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.23$ (d, J = 6.3 Hz, 1 H, CHCH₃), 3.36-3.40 (m, 4 H, OCH₃, CH₃OCHH), 3.50 (dd, J = 5.5, J = 10.2 Hz, 1 H, CH₃OCHH), 3.74 (s, 3 H, ArOCH₃), 4.43 (sext, J = 6 Hz, 1 H, CH₃CH), 6.52 (d, J = 8.1 Hz, 1 H, ArH), 6.59 (d, J = 8.2 Hz, 1 H, ArH), 7.21 (t, J = 8.3 Hz, 1 H, ArH) ppm. ¹³C NMR $(CDCl_3, 100 \text{ MHz}): \delta = 17.4, 56.5, 59.8, 75.3, 76.1, 104.5, 108.0,$ 112.2, 130.7, 159.6, 161.5 ppm. ⁷⁷Se NMR (57 MHz, CDCl₃): δ = 343 ppm. HRMS: m/z calcd. for $C_{22}H_{30}O_6^{76}SeNH_4 [M + NH_4]^+$: 560.0765; found 560.0767. IR (neat): $\tilde{v} = 3078, 2971, 2917, 2850,$ 2753, 2524, 2360, 1571, 1463, 1246, 1091, 766, 713 cm⁻¹.

2,6-Bis{[(*R***)-1-methoxypropan-2-yl]oxy}phenyl Diselenide (9c):** Similar reaction as described for **9a** using: **8c** (640 mg, 2.5 mmol) in dry THF (8 mL); *n*-butyllithium (1.5 mL, 2.5 M solution in hexane, 3.75 mmol); selenium powder (237 mg, 3 mmol). The mixture of **10** and **9c** was isolated and dissolved in absolute ethanol (10 mL), and NaBH₄ (81 mg, 2.13 mmol) was added. The red solution became colorless, was stirred for 15 min and then the flask was opened to air for 6 h. The solvent was removed in vacuo. After column chromatography (hexane/ethyl acetate, 6:1) the diselenide **9c** was isolated as a red oil in 44% yield (364 mg, 0.55 mmol). [*a*]_D²⁵ = -64.2 (*c* = 0.72, CHCl₃). ¹H NMR (CDCl₃, 400 MHz): δ = 1.15 (d, *J* = 6.2 Hz, 1 H), 3.27–3.31 (m, 7 H), 3.41 (dd, *J* = 5.3, *J* = 10.1 Hz, 1

H), 4.32 (sext, J = 6 Hz, 2 H), 6.47 (d, J = 8.3 Hz, 2 H), 7.07 (t, J = 8.2 Hz, 1 H) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.4$, 59.8, 75.0, 75.2, 107.8, 113.8, 130.4, 159.8 ppm. ⁷⁷Se NMR (57 MHz, CDCl₃): $\delta = 345$ ppm. HRMS: m/z calcd. for C₂₈H₄₂O₈⁸⁰Se₂NH₄ [M + NH₄]⁺: 684.1555; found 684.1554. IR (neat): $\tilde{v} = 2970$, 2928, 2880, 2835, 2820, 1572, 1456, 1375, 1246, 1201, 1112, 1092, 1037, 956, 769 cm⁻¹.

General Procedure for the Addition of Selenium Electrophiles to Styrene: The diselenide 9 (0.1 mmol) was dissolved in dry dichloromethane (2.5 mL) under argon, cooled to -78 °C, and treated with bromine (0.11 mmol, 0.11 mL of a 1 M solution in CCl₄). After 20 min a solution of silver triflate (72 mg, 0.28 mmol) in dry methanol (0.1 mL) was added and the mixture was stirred for 30 min at -78 °C. The styrene derivative (0.4 mmol) was added. After the mixture had been stirred for 2 h at -78 °C. Saturated aqueous NaHCO₃ (2 mL) was added followed by water (2 mL). After extraction of the reaction mixture with dichloromethane $(3 \times 10 \text{ mL})$, the combined organic phases were dried with MgSO4 and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel, yielding the addition products as colourless oils. The diastereomers could not be separated by column chromatography (ethyl acetate/hexane, 1:15). All spectroscopic data are given for the main diastereomers.

(2-Methoxy-2-phenylethyl)(2-{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)selane (12a): Colorless oil, 40% yield, (30 mg, 0.079 mmol). ¹H NMR (CDC1₃, 400 MHz): δ = 1.28 (d, *J* = 6.3 Hz, 3 H, CHC*H*₃), 3.02 (dd, *J* = 4.9, *J* = 12.1 Hz, 1 H, ArSeCH*H*), 3.18– 3.23 (m, 4 H, OC*H*₃, ArSeCH*H*), 3.23 (s, 3 H, OC*H*₃), 3.42 (dd, *J* = 4.9, *J* = 10.1 Hz, 1 H, CH₃OCH*H*), 3.55 (dd, *J* = 5.8, *J* = 10.1 Hz, 1 H, CH₃OCH*H*), 4.30 (dd, *J* = 4.9, *J* = 8.7 Hz, 1 H, CH₃OC*H*), 4.48 (sext, *J* = 5.9 Hz, 1 H, CH₃C*H*), 6.78–6.82 (m, 2 H, Ar*H*), 7.07–7.11 (m, 1 H, Ar*H*), 7.21–7.31 (m, 6 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.6, 32.9, 57.5, 59.8, 74.7, 76.3, 83.6, 114.0, 122.1, 127.1, 127.7, 128.5, 129.0, 131.4, 141.7, 156.6 ppm. HRMS: *m*/*z* calcd. for C₁₉H₂₄O₃⁸⁰Se [M + Na]⁺: 403.0788; found 403.0772. IR (neat): \tilde{v} = 3066, 2984, 2926, 2885, 2819, 1575, 1469, 1441, 1271, 1238, 1107, 1034, 957, 747, 702 cm⁻¹.

(2-Methoxy-2-phenylethyl)(2-methoxy-6-{[(*R*)-1-methoxypropan-2yl]oxy}phenyl)selane (12b): Colorless oil, 72 % yield (59 mg, 0.144 mmol). ¹H NMR (CDCl₃, 400 MHz): δ = 1.26 (d, *J* = 6.2 Hz, 3 H, CHC*H*₃), 3.02 (dd, *J* = 5, *J* = 12.1 Hz, 1 H, ArSeCH*H*), 3.14 (s, 3 H, OC*H*₃), 3.24 (s, 3 H, OC*H*₃), 3.20–3.27 (m, 1 H, Ar-SeCH*H*), 3.41 (dd, *J* = 4.7, *J* = 10 Hz, 1 H, CH₃OCH*H*), 3.54 (dd, *J* = 5.9, *J* = 10 Hz, 1 H, CH₃OCH*H*), 3.79 (s, 3 H, OC*H*₃), 4.23 (dd, *J* = 4.9, *J* = 8.8 Hz, 1 H, CH₃OCH*H*), 4.46 (sext, *J* = 4.7 Hz, 1 H, CH₃C*H*), 6.45 (d, *J* = 8.2 Hz, 1 H, Ar*H*), 6.51 (d, *J* = 8.2 Hz, 1 H, Ar*H*), 7.11 (t, *J* = 8.3 Hz, 1 H, Ar*H*), 7.15–7.28 (m, 5 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.5, 34.2, 56.5, 57.4, 59.6, 74.8, 76.3, 84.2, 104.4, 107.7, 109.2, 127.1, 128.2, 128.7, 129.5, 141.9, 159.4, 160.1 ppm. HRMS: *m*/z calcd. for C₂₀H₂₆O₄⁷⁶⁻ SeNH₄ [M + NH₄]⁺: 424.1361; found 424.1362. IR (neat): \tilde{v} = 2971, 2932, 2880, 2830, 1582, 1464, 1246, 1097, 767, 702 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)(2-methoxy-2-phenylethyl)selane (12c): Colorless oil, 68% yield (64 mg, 0.137 mmol). ¹H NMR (CDC1₃, 400 MHz): $\delta = 1.27$ (d, J = 6.3 Hz, 6 H, CHCH₃), 3.06 (dd, J = 4.9, J = 12.2 Hz, 1 H, ArSeCH), 3.14 (s, 3 H, OCH₃), 3.21–3.27 (m, 7 H, OCH₃, ArSeCH), 3.40 (dd, J = 4.8, J = 10.0 Hz, 2 H, CH₂OCH₃), 3.54 (dd, J = 5.8, J = 10.0 Hz, 2 H, CH₂OCH₃), 6.50 (d, J = 8.3 Hz, 2 H, ArH), 7.07 (t, J = 8.3 Hz, 1 H, ArH), 7.15–7.27 (m, 5 H, ArH) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.2$, 33.7, 57.0, 59.2, 74.4 75.9, 83.8, 107.5, 110.8, 126.7, 127.7, 128.3, 128.7, 141.7, 159.1 ppm. HRMS: m/z calcd. for $C_{23}H_{32}O_5SeNH_4$ [M + NH₄]⁺: 480.1813; found 480.1817. IR (neat): $\tilde{v} = 2979$, 2930, 2878, 2817, 1579, 1492, 1455, 1375, 1352, 1244, 1201, 1151, 1111, 1039, 955, 767, 703 cm⁻¹.

(2,6-Bis{[(R)-1-methoxypropan-2-yl]oxy}phenyl)[2-methoxy-2-(otolyl)ethyl]selane (13a): Colorless oil, 54% yield (52 mg, 0.108 mmol). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.26$ (d, J = 6.3 Hz, 6 H, CHCH₃), 2.07 (s, 3 H, ArCH₃), 3.08–3.10 (m, 2 H, ArSeCH₂), 3.15 (s, 3 H, OCH₃), 3.20 (s, 6 H, OCH₃), 3.39 (dd, J = 4.7, J =10.1 Hz, 1 H, CH_3OCH_2), 3.52 (dd, J = 5.9, J = 10.1 Hz, 1 H, CH₃OCH₂), 4.38–4.48 (m, 3 H, CH₃CH, CH₃OCH), 6.50 (d, J = 8.3 Hz, 1 H, ArH), 7.00–7.15 (m, 4 H, ArH), 7.30 (d, J = 7.5 Hz, 1 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.5 (CH*CH*₃), 19.2 (Ar*CH*₃), 33.2 (ArSe*CH*₂), 57.3 (CHO*CH*₃), 59.6 (CH₂O*CH*₃), 74.9 [O(CH₃)CH], 76.3 (CH₃OCH₂), 80.7 (CH₃OCH), 107.8 (C), 111.2 (C), 126.1 (C), 127.6 (C), 129.3 (C), 130.7 (C), 136.1 (C), 140.1 (C), 159.7 (C) ppm. HRMS: *m*/*z* calcd. for C₂₄H₃₄O₅⁷⁴SeNH₄ $[M + NH_4]^+$: 494.1969; found 494.1964. IR (neat): $\tilde{v} = 3392, 2974,$ 2925, 2885, 2822, 2357, 2341, 1581, 1558, 1463, 1375, 1253, 1219, 1112, 1037 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)[2-(2-chlorophenyl)-2-methoxyethyl]selane (13b): Colorless oil, 61 % yield (63 mg, 0.125 mmol). ¹H NMR (CDCl₃, 400 MHz): δ = 1.27 (d, *J* = 6.3 Hz, 1 H, CH*CH*₃), 3.07 (dd, *J* = 9.2, *J* = 12.4 Hz, 1 H, ArSe*CH*), 3.16– 3.20 (m, 4 H, O*CH*₃, ArSe*CH*), 3.23 (s, 6 H, O*CH*₃), 3.39 (dd, *J* = 4.8, *J* = 10.1 Hz, 2 H, CH₃O*CH*₂), 3.54 (dd, *J* = 5.8, *J* = 10.1 Hz, 2 H, CH₃O*CH*₂), 4.45 (sext, *J* = 6 Hz, 2 H, CH₃C*H*), 4.67 (dd, *J* = 3.7, *J* = 9.2 Hz, 1 H, CH₃O*CH*), 6.48 (d, *J* = 8.3 Hz, 2 H, Ar*H*), 7.05–7.12 (m, 2 H, Ar*H*), 7.17–7.21 (m, 2 H, Ar*H*), 7.41 (dd, *J* = 1.7, *J* = 7.6 Hz, 1 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.5, 32.6, 57.7, 59.6, 74.8, 76.2, 80.3, 107.7, 111.0, 127.4, 127.7, 129.0, 129.3, 129.8, 133.6, 139.5, 159.6 ppm. HRMS: *m/z* calcd. for C₂₃H₃₁ClO₅⁷⁴SeNH₄ [M + NH₄]⁺: 514.1423 found 514.1420. IR (neat): \tilde{v} = 3063, 2980, 2930, 2885, 2825, 1760, 1732, 1574, 1456, 1375, 1353, 1245, 1202, 1152, 1112, 1038, 968, 760, 735, 708 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)[2-methoxy-2-(*m*-tolyl)ethyl]selane (13c): Colorless oil, 44 % yield (40 mg, 0.088 mmol). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.27$ (d, J = 6.3 Hz, 6 H, CH*CH*₃), 2.26 (s, 3 H, Ar*CH*₃), 3.05 (dd, J = 4.9, J = 12.1 Hz, 1 H, ArSe*CH*), 3.20–3.26 (m, 7 H, O*CH*₃, ArSe*CH*), 4.17 (dd, J = 4.9, J = 8.9 Hz, 1 H, CH₃O*CH*), 4.45 (sext, J = 6 Hz, 2 H, CH₃*CH*), 6.50 (d, J = 8.5 Hz, 2 H, Ar*H*), 6.98–7.14 (m, 5 H) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.6$, 21.9, 34.1, 57.4, 59.6, 74.8, 76.2, 84.2, 107.9, 111.2, 124.3, 127.7, 128.6, 128.9, 129.1, 138.2, 142.0, 159.5 ppm. HRMS: *m*/*z* calcd. for C₂₄H₃₄O₅⁸⁰Se [M + Na]⁺: 505.1469; found 505.1464. IR (neat): $\tilde{v} = 2979$, 2927, 2881, 2817, 2358, 2337, 1575, 1456, 1375, 1244, 1202, 1111, 1039, 966, 768, 709 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)]2-methoxy-2-(4-methoxyphenyl)ethyl]selane (13d): Colorless oil, 32% yield, (32 mg, 0.064 mmol). ¹H NMR (CDCl₃, 400 MHz): $\delta = 1.27$ (d, J = 6.3 Hz, 6 H, CH*CH*₃), 3.04 (dd, J = 5.3, J = 12.0 Hz, 1 H, ArSe*CH*), 3.11 (s, 3 H, OC*H*₃) 3.21–3.27 (m, 7 H, O*CH*₃, ArSe*CH*), 3.40 (dd, J = 4.9, J = 10.1 Hz, 2 H, CH₃O*CH*₂), 3.54 (dd, J = 5.7, J = 10.0 Hz, 2 H, CH₃O*CH*₂), 3.54 (dd, J = 5.7, J = 10.0 Hz, 2 H, CH₃O*CH*₂), 3.72 (s, 3 H, ArOCH₃), 4.15 (dd, J = 5.3, J = 8.6 Hz, 1 H, CH₃O*CH*), 4.45 (sext, J = 6 Hz, 2 H, CH₃*CH*), 6.49 (d, J = 8.3 Hz, 2 H, Ar*H*), 6.77 (d, J = 8.7 Hz, 1 H, Ar*H*), 7.06 (t, J = 8.3 Hz, 1 H, Ar*H*), 7.15 (d, J = 8.6 Hz, 1 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.6$, 34.0, 55.7, 57.10, 59.7, 74.7, 74.8, 83.7, 107.8, 111.1, 114.0, 128.4, 129.1, 134.0, 159.5 ppm. HRMS: *m*/z calcd. for C₂₄H₃₄O₆⁸⁰Se [M + Na]⁺: 521.1418; found



521.1441. IR (neat): $\tilde{v} = 2974$, 2923, 2870, 2833, 2816, 2358, 1610, 1575, 1511, 1456, 1245, 1110, 1036, 830, 766 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)(2-methoxy-2-phenylpropyl)selane (13f): Colorless oil, 65% yield (63 mg, 0.131 mmol). ¹H NMR (CDCl₃, 400 MHz): δ = 1.25 (d, *J* = 6.3 Hz, 1 H, CH*CH*₃), 3.05 (s, 3 H, *OCH*₃), 3.20–3.32 (m, 7 H, *OCH*₃, Ar-Se*CH*), 3.40–3.45 (m, 4 H, CH₃O*CH*₂, ArSe*CH*), 3.54 (dd, *J* = 5.8, *J* = 10.1 Hz, 2 H, CH₃O*CH*₂), 4.45 (sext, *J* = 6 Hz, 2 H, CH₃*CH*), 6.48 (d, *J* = 8.3 Hz, 2 H, Ar*H*), 7.06 (t, *J* = 8.2 Hz, 1 H, Ar*H*), 7.15 (t, *J* = 7.3 Hz, 1 H, Ar*H*), 7.24 (t, *J* = 7.5 Hz, 2 H, Ar*H*), 7.34 (dd, *J* = 1.2, *J* = 8.4 Hz, 1 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.5, 24.5, 39.2, 51.3, 59.7, 74.6, 76.3, 79.6, 107.8, 111.7, 126.6, 127.4, 128.5, 129.2, 145.0, 159.6 ppm. HRMS: *mlz* calcd. for C₂₄H₃₄O₅⁷⁴SeNH₄ [M + NH₄]⁺: 494.1949 found 494.1968. IR (neat): \tilde{v} = 3082, 3059, 2979, 2930, 2823, 1579, 1493, 1456, 1373, 1244, 1201, 1153, 1113, 1039, 969, 868, 766, 902 cm⁻¹.

(2,6-Bis{[(R)-1-methoxypropan-2-yl]oxy}phenyl)(2-ethoxy-2-phenylethyl)selane (13g): Light yellow oil, 54% yield (52 mg, 0.108 mmol). ¹H NMR (CDCl₃, 400 MHz): δ = 1.09 (t, J = 7.0 Hz, 3 H, CH_2CH_3), 1.27 (d, J = 6.3 Hz, 6 H, $CHCH_3$), 3.06 (dd, J = 4.8, J= 12.1 Hz, 1 H, ArSeCH), 3.21–3.30 (m, 9 H, CH₃CH₂, OCH₃, ArSeCH), 3.40 (dd, J = 4.8, J = 10.0 Hz, 2 H, CH₃OCH₂), 3.54 $(dd, J = 5.7, J = 10.0 Hz, 2 H, CH_3OCH_2), 4.29 (dd, J = 4.9, J =$ 8.9 Hz, 1 H, CH₃OCH), 4.45 (sext, J = 6 Hz, 2 H, CH₃CH), 6.50 (d, J = 8.3 Hz, 2 H, ArH), 7.07 (t, J = 8.3 Hz, 1 H, ArH), 7.14– 7.17 (m, 1 H, ArH), 7.23–7.24 (m, 4 H, ArH) ppm. ¹³C NMR $(CDCl_3, 100 \text{ MHz})$: $\delta = 15.7, 17.6, 34.4, 59.6, 65.0, 74.8, 76.2, 82.3,$ 107.8, 111.2, 127.0, 127.9, 128.6, 129.0, 142.8, 159.5 ppm. HRMS: m/z calcd. for C₂₄H₃₄O₅⁷⁴SeNH₄ [M + NH₄]⁺: 494.1969; found 494.1967. IR (neat): $\tilde{v} = 3088, 3055, 3034, 2976, 2928, 2877, 2814,$ 2360, 1577, 1456, 1375, 1244, 1202, 1152, 1113, 1039, 970, 766, 702 cm^{-1} .

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)(2-isopropoxy-2-phenylethyl)selane (13h): Colorless oil, 48 % yield (48 mg, 0.097 mmol). ¹H NMR (CDCl₃, 400 MHz): $\delta = 0.97$ [d, J = 6.2 Hz, 3 H, CH(CH₃)*CH*₃], 1.07 [d, J = 6.0 Hz, 3 H, CH(CH₃)*CH*₃], 1.27 (d, J = 6.3 Hz, 6 H, CH*CH*₃), 3.04 (dd, J = 4.6, J = 12.1 Hz, 1 H, ArSe*CH*), 3.18–3.24 (m, 7 H, O*CH*₃, ArSe*CH*), 3.40 (dd, J = 4.9, J = 10.0 Hz, 2 H, CH₃O*CH*₂), 3.53 (dd, J = 5.7, J = 10.0 Hz, 2 H, CH₃O*CH*₂), 3.53 (dd, J = 5.7, J = 10.0 Hz, 2 H, CH₃O*CH*₂), 4.40–4.47 (m, 3 H, CH₃O*CH*, CH₃*CH*), 6.49 (d, J = 8.3 Hz, 2 H, Ar*H*), 7.05 (t, J = 8.3 Hz, 1 H, Ar*H*), 7.13–7.27 (m, 5 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\delta = 17.6$, 21.9, 23.8, 34.9, 70.2, 74.8, 76.3, 80.0, 107.8, 111.5, 127.0, 127.8, 128.6, 129.0, 143.7, 159.5 ppm. HRMS: *m*/*z* calcd. for C₂₅H₃₆O₅⁸⁰Se [M + Na]⁺: 519.1616; found 519.1639. IR (neat): $\tilde{v} = 3064$, 3021, 2973, 2929, 2874, 2820, 2358, 1575, 1455, 1375, 1244, 1202, 1114, 1039, 764, 702 cm⁻¹.

(2,6-Bis{[(*R*)-1-methoxypropan-2-yl]oxy}phenyl)[2-(*tert*-butoxy)-2-phenylethyl]selane (13i): Colorless oil, 34% yield (34 mg, 0.067 mmol). ¹H NMR (CDCl₃, 400 MHz): δ = 1.03 [s, 9 H, O(CH₃)₃], 1.27 (d, *J* = 6.4 Hz, 1 H, CHCH₃), 2.94–2.99 (m, 1 H, ArSeCH*H*), 3.15–3.25 (m, 7 H, OCH₃, ArSeCH*H*), 3.33–3.43 (2 H, CH₃OCH*H*), 3.50–3.58 (m, 2 H, CH₃OCH*H*), 3.39–4.47 (m, 2 H, CH₃C*H*), 4.55–4.59 [m, 1 H, C(*t*BuO)C*H*], 6.48 (d, *J* = 8.3 Hz, 2 H, Ar*H*), 7.02–7.14 (m, 3 H, Ar*H*), 7.17–7.21 (m, 1 H, Ar*H*), 7.28–7.31 (m, 2 H, Ar*H*) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 17.6, 29.3, 36.4, 59.7, 74.8, 75.0, 75.3, 76.3, 107.9, 111.9, 126.7, 127.3, 128.4, 128.9, 146.4, 159.4 ppm. HRMS: *m*/*z* calcd. for C₂₆H₃₈O₅⁷⁴SeNH₄ [M + NH₄]⁺: 522.2282; found 522.2281. IR (neat): \tilde{v} = 3088, 30069, 3028, 2975, 2929, 2885, 2829, 2806, 2359, 1575, 1455, 1389, 1366, 1244, 1193, 1153, 1113, 1038, 969, 766, 703 cm⁻¹.

Supporting Information (see also the footnote on the first page of this article): Supporting Information contains ¹H and ¹³C NMR spectra for all compounds.

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