Chiral Diselenides in Asymmetric Cyclization Reactions

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The use of chiral selenium electrophile **6** generated in situ from the chiral diselenide **5** in asymmetric ring closure reactions of unsaturated alcohols, carboxylic acids and carbamates is presented herein. Tetrasubstituted carbon atoms can be generated selectively with diastereoselectivities up to 85% when geminal disubstituted alkenes are used. The cyclization of vinylic silanes leads to versatile building blocks for further synthetic transformations.

Introduction

The synthesis of optically pure heterocyclic compounds containing stereogenic centers is still of high interest in organic synthesis. Due to their wide occurence in natural products and in biologically active compounds cyclic ethers or lactones as well as N-heterocycles provide interesting targets for synthetic strategies.

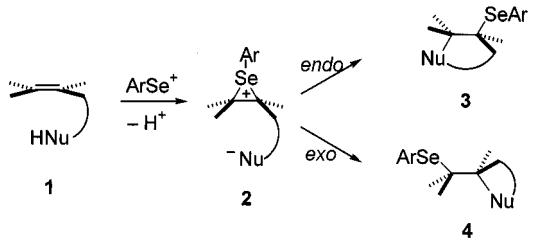
Besides many other approaches, the synthesis of heterocycles via cyclization of alkenes containing internal nucleophiles have been performed with a variety of electrophiles such as H^+, Br^+, I^+, Pb^{IV}, Hg^{II}, Tl^I, Tl^{III}, or PhS^+.[1a] The reactions of alkenes with organoselenium reagents gained increased popularity because the products are β-substituted selenium derivatives which can be employed in further transformations.[1] Asymmetric functionalizations of alkenes with chiral selenium electrophiles were performed by us[2] and by other research groups.[3][4][5][6] The application of chiral selenium electrophiles in cyclizations of alkenes bearing an internal nucleophile are described herein.

Results and Discussion

The first step in the cyclization of alkenes **1** is the activation of the C=C double bond by addition of a selenium electrophile. This results in the formation of a seleniranium ion **2**, which is subsequently attacked by the nucleophile from the *anti*-side. Depending on the ring size and the substitution pattern, the addition can occur either by an *exo* or an *endo* pathway leading to the products **3** or **4**, respectively (Scheme 1).

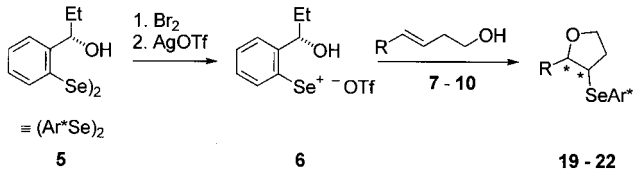
The cyclizations are performed with the chiral selenium electrophile **6** which is generated from diselenide **5**. Initial attempts were carried out under the same reaction conditions as for the asymmetric selenenylation without methanol. Methanol was thought to interfere with the internal nucleophile. But in contrast to the intermolecular reaction, the cyclized products were obtained in low yields and stereoselectivities. The presence of methanol was found to

Scheme 1



be essential for the yield as well as for an efficient transfer of chirality. No competition of an intermolecular attack of the seleniranium ion by methanol was observed.[2a][4b][7] Methanol is assumed to stabilize the seleniranium ion which is obviously important for the cyclization reaction.

Scheme 2

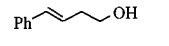
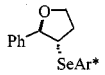
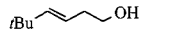
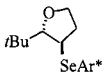
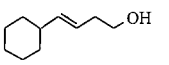
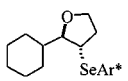
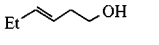
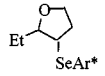
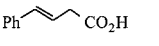
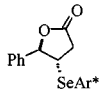
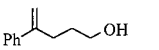
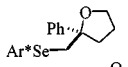
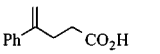
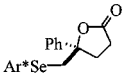
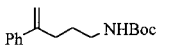
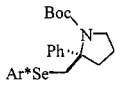
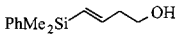
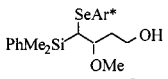
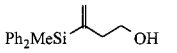
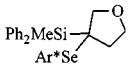
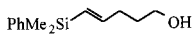
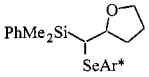
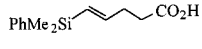
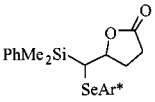


We performed cyclizations with different substituted homoallylic alcohols **7**–**10**. The results presented in Table 1 show a cyclization in an 5-*endo* mode.

The stereochemical course of the cyclization reaction with diselenide **5** is the same as in the intermolecular methoxyselenenylation reaction which we investigated in detail.[8] The cyclization of homoallylic alcohol **7** (R = Ph) leads to a newly formed stereocenter in 2-position of the tetrahydrofuran derivative **19** with (*R*) configuration formed by *re*-attack of the selenium electrophile **6**. The absolute stereochemistry was assigned after radical cleav-

  1434–193X/98/0707–1361 $ 17.50+.50/0

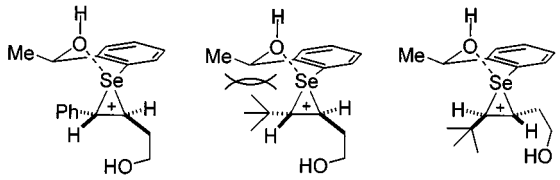
Table 1. Cyclization products of homoallylic alcohols **7**–**18**

Alkene		Major Product		Yield (%)	*de* (%)
	**7**		**19**	87	84
	**8**		**20**	68	46
	**9**		**21**	64	49
	**10**		**22**	60	0
	**11**		**23**	41	72
	**12**		**24**	45	78
	**13**		**25**	58	85
	**14**		**26**	40	45
	**15**		**27**	20	78
	**16**		**28**	22	9
	**17**		**29**	34	82
	**18**		**30**	27	80

age[9] of the selenium moiety to give the known (*S*)-2-phenyl-tetrahydrofuran.[10]

An exception is observed when the *tert*-butyl-substituted alkene **8** is used. The stereochemistry of the major product **20** is opposite to that of **19** with the phenyl substituent which corresponds to a *si*-attack of the selenium electrophile **6** to the alkene **8**.[11] Calculations of the diastereomeric seleniranium ions[8] show that the most stable intermediate with a phenyl substituent is likely to have structure **31**. After *anti*-attack of the nucleophile the product with (*R*,*R*) stereochemistry at the newly formed chiral centers is obtained. Assuming the same orientation of the substituent for the *tert*-butyl case as shown in **32a** a great steric interaction would occur. We assume that the most stable intermediate in the *tert*-butyl case will be **32b** where the bulky group has a smaller interaction with the aryl

Scheme 3

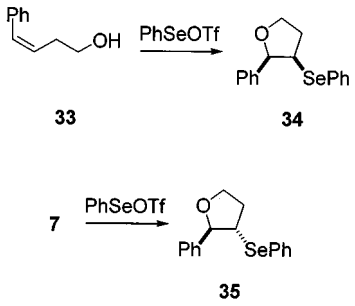


31 **32a** **32b**

group on the selenium. The attack of the nucleophile leads then to inverse stereochemistry in product **20**.

While a bulkier side chain like R = cyclohexyl in **9** leads to a diastereoselectivity of 49% in the product **21**, no selectivity is observed in the cyclization of (*E*)-3-hexen-1-ol **10**. The cyclization of (*Z*)-4-phenyl-3-buten-1-ol **33** could not be achieved with selenium electrophile **6** underlining the reduced reactivity of selenium electrophiles towards (*Z*)-alkenes. Even the cyclization of **33** with phenylselenyl triflate yields the *syn*-addition product **34** in only 20% yield, while the cyclization of **7** gave the *anti*-addition product **35** in 73% yield.

Scheme 4



33 **34**

35

If the reaction is carried out with α,α-disubstituted alkenes, the formation of asymmetric tetrasubstituted carbon atoms is possible. These are not easily accessible by other methods.[12] Cyclization of alcohol **12** generates the 2,2-disubstituted tetrahydrofuran derivative **24** with good diastereomeric excess. The cyclization of the corresponding carboxylic acid **13** yields the 5,5-disubstituted tetrahydrofuranon derivative **25** in even better yield and selectivity (85% *de*).

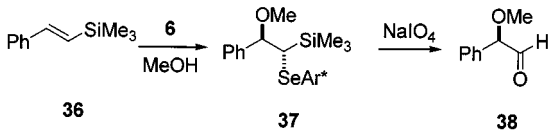
In these examples the cyclization occurs via a 5-*exo*-pathway driven by activation of the benzylic position. The stereochemical course of the reaction is similar to the one described for alkene **7** and for the intermolecular methoxyselenenylation reaction.[8] The selenium electrophile **6** attacks the alkene predominantly from the *si*-face leading to a more stable seleniranium ion. Therefore, the subsequent *anti*-attack of the nucleophile leads to (*R*) configuration at the benzylic carbon atom.

Even cyclizations with nitrogen nucleophiles can be achieved. A cyclization did not occur when free amines or acetylated amines are employed as intramolecular nucleophiles. The protection of the nitrogen as a carbamate with

increased nucleophilicity is required for cyclization. This behaviour was already observed in the synthesis of the tetrahydroisoquinoline alkaloid salsolidine by intramolecular selenium-mediated cyclization.[13] The cyclization of carbamate **14** leads to the pyrrolidine derivative **26**.

Although high stereoselectivities are obtained with aromatic alkenes, the synthetic potential of the asymmetric oxyselenenylation reaction would increase if other alkenes could be used. Vinylic silanes are interesting precursors and easily accessible.[14] After methoxyselenenylation of the alkene **36** the product **37** was obtained in 52% yield and with 85% *de*. The silicon functionality neighboring the selenium allows further transformations. After oxidation of the selenide to the selenoxide with sodium periodate a Pummerer-type rearrangement[15] yields the α-methoxy substituted aldehyde **38**. Compound **38** was shown to have (*R*) configuration which was confirmed by independent synthesis.[16]

Scheme 5



36 **37** **38**

Subsequently we employed vinylic silanes like **15** in cyclization reactions. The compound obtained was, however, not the cyclized product. The addition product **27** was isolated where methanol has added in β-position to the silicon atom because of the β-silicon effect.[17] The attack of the internal nucleophile to the β-carbon atom would occur in a 4-*exo*-pathway and is disfavoured. Therefore the methanol present in the reaction mixture adds to the seleniranium ion yielding **27** with 78% *de*.

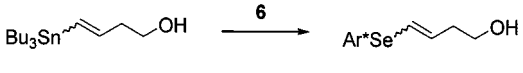
Vinylic silanes of type **16** could, however, cyclize via a 5-*endo* pathway to 3-substituted tetrahydrofuran derivatives. Compound **16** is not an appropriate precursor for an efficient selenium induced cyclization because of the poor diastereoselectivities obtained in product **28**.

Therefore, we focussed our interest on vinylic silanes of type **17**. The cyclization is also supported by the β-silicon effect and occurs in a 5-*exo*-pathway with high stereoselectivities. The Pummerer-reaction could not be performed successfully in this case because of the stereochemical lability of the resulting 1-formyl-tetrahydrofuran.[18] The use of these products in further synthetic sequences is currently under investigation.

The moderate yield of the cyclization reaction with vinylic silanes is probably due to the low reactivity towards the selenium electrophile. No side reactions are observed and after the reactions the vinylic silanes can be recovered.

In all the cyclizations mentioned above the use of a carboxylic acid instead of a hydroxy group as internal nucleophile did not influence the yields and the stereoselectivities of the reactions (see alkenes **11**, **13**, **18**).

Scheme 6



39 **40**

The tin-substituted alkene **39** could not be cyclized with the selenium electrophile **6**. Under the reaction conditions a substitution of the tributyltin moiety by an arylseleno group takes place.

Conclusions

The cyclizations of different alkenes bearing internal nucleophiles with the chiral selenium electrophile **6** generated from diselenide **5** are presented. Cyclizations leading to chiral tetrasubstituted carbon atoms are performed with good diastereoselectivities. By cyclization of silicon-substituted alkenes chiral building blocks for further synthesis are obtained with good selectivities.

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Experimental Section

General: All reactions were performed under argon with anhydrous solvents. − ^{1}H- and ^{13}C-NMR spectra were recorded with a Varian Gemini 300 spectrometer (300 MHz and 75 MHz for ^{1}H and ^{13}C, respectively) or Bruker 600 spectrometer (600 MHz for ^{1}H), chemical shifts are reported in ppm relative to tetramethylsilane as internal standard, coupling constants in Hz. Multiplicities in the ^{13}C-NMR spectra were determined by the APT puls sequence. ^{77}Se-NMR spectra were recorded with a Varian Gemini 400 spectrometer (76 MHz) or a Bruker 600 spectrometer (114 MHz) with diphenyl diselenide (δ = 475) as external standard. − IR spectra were measured with a Perkin-Elmer 781 spectrophotometer. − MS spectra and HRMS measurements were recorded with a Finnigan MAT 312 apparatus. − Optical rotations were measured with a Perkin-Elmer 141 polarimeter.

The alkenes were synthesized according to following literature procedures: **7**, **8**, **9** by selective addition of the corresponding grignard compound to 2,3-dihydrofuran,[19] **10** by reduction of 2,4-hexadienoic acid with lithium in liquid ammonia to (*E*)-3-hexenoic acid and subsequent reduction with $LiAlH_4$,[20] **11** by addition of malonic acid to phenylacetaldehyde,[21] **13** by Wittig reaction of 3-benzoylpropionic acid with methyltriphenylphosphonium bromide, **12** was synthesized by reduction of **13** with $LiAlH_4$, **14** was synthesized from **12** by a synthetic sequence of mesylation, reaction with potassium phthalimide, reduction to the amine and Boc-protection, **15**, **16**, and **17** were synthesized by selective hydrosilylation of 3-butyn-1-ol and 4-pentyn-1-ol respectively,[14] **18** was obtained by oxidation of **17** with CrO_3,[22] **33** by addition of phenylacetylene to oxirane[23] and subsequent hydrogenation with Lindlar catalyst (*Z*/*E* 21:1), **36** was synthesized by reduction of 1-phenyl-2-trimethylsilyl acetylene with DIBAL,[24] and **39** by reaction of tributyltin hydride with 3-butyn-1-ol (*E*/*Z* 1.5:1). Spectroscopic data for the alkenes: **7**,[25] **8**,[26] **10**,[25] **11**,[27] **12**,[28] **13**.[29]

(E)-4-Cyclohexyl-3-buten-1-ol (**9**): Colorless oil. − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.00−1.70 (m, 10 H, 5 × CH_2), 1.85−2.00

(m, 1 H, CH), 2.12 (s, 1 H, OH), 2.24 (q, J = 6.5 Hz, 2 H, CH_2CH$_2$OH), 3.60 (t, J = 5.8 Hz, 2 H, CH_2OH), 5.43 (dtd, J = 15.4 Hz, J = 6.8 Hz, J = 1.1 Hz, 1 H, CHCH=CH), 5.50 (dd, J = 15.4 Hz, J = 6.5 Hz, 1 H, CHCH=CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 26.0 (t, 2 C, CH$_2$), 26.1 (t, CH$_2$), 33.0 (t, 2 C, CH$_2$), 35.9 (t, CH$_2$CH$_2$OH), 40.6 (d, CH), 62.0 (t, CH$_2$OH), 123.0 (d, CHCH=CH), 139.9 (d, CHCH=CH). − IR (CHCl$_3$): ν = 3333, 2919, 2850, 1448, 1369, 1349, 1048, 969, 892 cm^{-1}. − MS (EI): *m/z* (%) = 154 (22) [M$^+$], 136 (66), 121 (61), 107 (77), 94 (80), 81 (100), 67 (87), 55 (32), 41 (44).

Methanesulfonic Acid 4-Phenylpent-4-enyl Ester: Triethylamine (6.55 ml, 47 mmol) was added dropwise to a solution of **12**[28] (1.52 g, 9.4 mmol) and methanesulfonyl chloride (0.877 ml, 11.3 mmol) in methylene chloride (50 ml) at 0°C and stirred for 2 hours. After the addition of water, the mixture was extracted with methylene chloride. The organic layer was dried (MgSO$_4$) and evaporated yielding the mesylate (2.15 g, 8.96 mmol, 95%) which was used without further purification. Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 1.90 (qd, J = 7.6 Hz, J = 7.2 Hz, 2 H, CH_2CH$_2$OMs), 2.65 (td, J = 7.4 Hz, J = 1.1 Hz, 2 H, =CCH$_2$), 2.97 (s, 3 H, CH$_3$), 4.43 (t, J = 6.4 Hz, 2 H, CH_2OMs), 5.11 (q, J = 1.3 Hz, 1 H, C=CHH), 5.33 (d, J = 1.3 Hz, 1 H, C=CHH), 7.20−7.40 (m, 5 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 27.6 (t, CH$_2$CH$_2$OMs), 31.1 (t, =CCH$_2$), 37.4 (q, CH$_3$), 69.3 (t, CH$_2$OMs), 113.6 (t, C=CH$_2$), 126.1 (d, 2 C), 127.7 (d), 128.5 (d, 2 C), 140.5 (s), 146.7 (s, C=CH$_2$).

N-(4-Phenyl-4-pentenyl)phthalimide: The mesylate (2.15 g, 8.96 mmol) and potassium phthalimide (1.85 g, 10 mmol), were suspended in DMF (10 ml) and stirred at 120°C for 75 minutes. After the addition of water, the mixture was extracted with *tert*-butyl methyl ether (3 × 20 ml). The organic phase was dried (MgSO$_4$), evaporated and the residue purified by flash chromatography (silica gel, *tert*-butyl methyl ether/pentane, 1:3) yielding the phthalimide (1.91 g, 6.56 mmol, 73%). Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 1.87 (quint., J = 7.5 Hz, 2 H, CH_2CH$_2$N), 2.56 (t, J = 7.7 Hz, 2 H, =CCH$_2$), 3.73 (t, J = 7.3 Hz, 2 H, CH_2N), 5.10 (q, J = 1.3 Hz, 1 H, C=CHH), 5.28 (d, J = 0.9 Hz, 1 H, C=CHH), 7.20−7.40 (m, 5 H, arom. CH), 7.60−7.90 (m, 4 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 27.1 (t, CH$_2$CH$_2$N), 32.7 (t, =CCH$_2$), 37.8 (t, CH$_2$N), 112.8 (t, C=CH$_2$), 123.2 (d, 2 C), 126.1 (d, 2 C), 127.5 (d), 128.4 (d, 2 C), 132.2 (s, 2 C), 133.9 (d, 2 C), 141.0 (s), 147.3 (s, C=CH$_2$), 168.4 (s, 2 C, C=O). − MS (EI): *m/z* (%) = 291 (27) [M$^+$], 160 (41), 144 (100), 129 (53), 118 (72), 77 (29).

4-Phenylpent-4-enylamine: The phthalimide (1.38 g, 4.75 mmol) and hydrazine hydrate (2.38 g, 47.5 mmol) in ethanol (20 ml) were heated under reflux for 15 min. After the addition of 1 N HCl (10 ml) solution the mixture was washed with *tert*-butyl methyl ether (3 × 5 ml). The aqueous layer was treated with 1 N NaOH, and extracted with *tert*-butyl methyl ether (3 × 5 ml). After drying (MgSO$_4$), and evaporation of the solvent the pure amine (610 mg, 3.79 mmol, 66%) was obtained without further purification. − Orange oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 1.17 (s, br, 2 H, NH$_2$), 1.59 (quint., J = 7.8 Hz, 2 H, CH_2CH$_2$N), 2.55 (td, J = 7.6 Hz, J = 1.1 Hz, 2 H, =CCH$_2$), 2.71 (t, J = 7.1 Hz, 2 H, CH_2N), 5.07 (q, J = 1.4 Hz, 1 H, C=CHH), 5.27 (d, J = 1.4 Hz, 1 H, C=CHH), 7.20−7.45 (m, 5 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 32.4 (t, CH$_2$CH$_2$N), 32.7 (t, =CCH$_2$), 41.9 (t, CH$_2$N), 112.4 (t, C=CH$_2$), 126.2 (d, 2 C), 127.4 (d), 128.3 (d, 2 C), 141.2 (s), 148.2 (s, C=CH$_2$).

N-Acetyl-4-phenylpent-4-enylamide: Acetyl chloride (0.14 ml, 2 mmol) and triethylamine (0.28 ml, 2 mmol) were added to a solution of the amine (161 mg, 1 mmol) in methylene chloride (2 ml) at 0°C. The mixture was stirred at room temperature for 24 hours. After the addition on 1 N HCl (2 ml) the mixture was extracted with *tert*-butyl methyl ether (3 × 10 ml). The organic phase was dried (MgSO$_4$), evaporated and the residue purified by flash chromatography (silica gel, *tert*-butyl methyl ether) yielding the acetyl protected amine (175 mg, 0.86 mmol, 86%). Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 1.59 (quint., J = 7.4 Hz, 2 H, CH_2CH$_2$N), 1.93 (s, 3 H, CH$_3$), 2.54 (td, J = 7.5 Hz, J = 1.0 Hz, 2 H, =CCH$_2$), 3.24 (q, J = 6.8 Hz, 2 H, CH_2N), 5.07 (q, J = 1.3 Hz, 1 H, C=CHH), 5.28 (d, J = 1.3 Hz, 1 H, C=CHH), 5.63 (s, br, 1 H, NH), 7.20−7.40 (m, 5 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 23.2 (q, CH$_3$), 28.0 (t, CH$_2$CH$_2$N), 32.7 (t, =CCH$_2$), 39.3 (t, CH$_2$N), 112.8 (t, C=CH$_2$), 126.1 (d, 2 C), 127.5 (d), 128.4 (d, 2 C), 140.9 (s), 147.6 (s, C=CH$_2$), 170.4 (s, C=O). − MS (EI): *m/z* (%) = 203 (6) [M$^+$], 144 (100), 129 (58), 118 (47), 43 (49).

N-tert-Butyloxycarbonyl-4-phenylpent-4-enylamide (**14**): Di-*tert*-butyl dicarbonate (0.44 ml, 2 mmol) and triethylamine (0.28 ml, 2 mmol) were added to a solution of the amine (161 mg, 1 mmol) in methylene chloride (2 ml) at 0°C. The mixture was stirred at room temperature for 24 hours. After the addition on 1 N HCl (2 ml) the mixture was extracted with *tert*-butyl methyl ether (3 × 10 ml). The organic phase was dried (MgSO$_4$), evaporated, and the residue purified by flash chromatography (silica gel, *tert*-butyl methyl ether/pentane, 1:10) yielding **14** (127 mg, 0.49 mmol, 49%). Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 1.44 [s, 9 H, C(CH$_3$)$_3$], 1.64 (tt, J = 7.4 Hz, J = 7.5, 2 H, CH_2CH$_2$N), 2.54 (t, J = 7.4 Hz, 2 H, =CCH$_2$), 3.14 (q, J = 6.3 Hz, 2 H, CH$_2$N), 4.53 (s, br, 1 H, NH), 5.08 (d, J = 1.1 Hz, 1 H, C=CHH), 5.29 (s, 1 H, C=CHH), 7.20−7.40 (m, 5 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = 28.4 [q, 3 C, C(CH$_3$)$_3$], 28.5 (t, CH$_2$CH$_2$N), 32.5 (t, =CCH$_2$), 40.1 (t, CH$_2$N), 79.0 [s, C(CH$_3$)$_3$], 112.7 (t, C=CH$_2$), 126.0 (d, 2 C), 127.4 (d), 128.3 (d, 2 C), 140.9 (s), 147.6 (s, C=CH$_2$), 155.9 (s, C=O).

(E)-4-(Dimethylphenylsilyl)-3-buten-1-ol (**15**): Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 0.33 (s, 6 H, SiCH$_3$), 1.50 (s, 1 H, OH), 2.42 (t, J = 6.6 Hz, 2 H, CH_2CH$_2$OH), 3.70 (t, J = 7.0 Hz, 2 H, CH$_2$OH), 5.91 (dd, J = 18.6 Hz, J = 1.2 Hz, 1 H, SiCH=CH), 6.10 (dtd, J = 18.6 Hz, J = 6.3 Hz, J = 1.2 Hz, 1 H, SiCH=CH), 7.30−7.60 (m, 5 H, arom. CH) − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = −2.6 (q, 2 C, SiCH$_3$), 40.0 (t, CH$_2$CH$_2$OH), 61.5 (t, CH$_2$OH), 127.7 (d, 2 C), 128.9 (d), 131.5 (t), 133.7 (d, 2 C), 138.1 (s), 144.6 (s). − MS (EI): *m/z* (%) = 191 (30) [M$^+$ − CH$_3$], 163 (71), 137 (79), 121 (59), 105 (32), 91 (28), 75 (100), 43 (58).

3-(Diphenylmethylsilyl)-3-buten-1-ol (**16**): Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 0.62 (s, 3 H, SiCH$_3$), 1.31 (s, 1 H, OH), 2.47 (t, J = 6.4 Hz, 2 H, CH_2CH$_2$OH), 3.70 (t, J = 6.3 Hz, 2 H, CH_2OH), 6.11 (m, 2 H, C=CH$_2$), 7.30−7.40 (m, 6 H, arom. CH), 7.45−7.55 (m, 4 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = − 4.0 (q, SiCH$_3$), 39.4 (t, CH$_2$CH$_2$OH), 61.5 (t, CH_2OH), 127.9 (d, 4 C), 129.4 (d, 2 C), 131.1 (t, C=CH$_2$), 135.0 (d, 4 C), 135.5 (s, 2 C), 144.9 (s, C=CH$_2$). − MS (EI): *m/z* (%) = 253 (17) [M$^+$ − CH$_3$], 240 (10), 207 (13), 197 (27), 183 (41), 137 (100), 105 (33).

(E)-4-(Dimethylphenylsilyl)-4-penten-1-ol (**17**): Colorless oil. − ^{1}H NMR (CDCl$_3$, 300 MHz): δ = 0.32 (s, 6 H, SiCH$_3$), 1.47 (s, 1 H, OH), 1.69 (quint., J = 7.0 Hz, 2 H, =CHCH_2), 2.24 (dq, J = 7.0 Hz, J = 1.4 Hz, 2 H, CH_2CH$_2$OH), 3.65 (t, J = 6.5 Hz, 2 H, CH_2OH), 5.81 (dt, J = 18.5 Hz, J = 1.5 Hz, 1 H, SiCH=CH), 6.13 (dt, J = 18.5 Hz, J = 6.2 Hz, 1 H, SiCH=CH), 7.30−7.36 (m, 3 H, arom. CH), 7.45−7.55 (m, 2 H, arom. CH). − ^{13}C NMR (CDCl$_3$, 75 MHz): δ = −2.5 (q, 2 C, SiCH$_3$), 31.5 (t, =CHCH$_2$), 33.0 (t, CH$_2$CH$_2$OH), 62.5 (t, CH$_2$OH), 127.7 (d, 2 C), 128.2 (d),

128.6 (d, Si*C*H=CH), 133.8 (d, 2 C), 139.1 (s), 148.2 (d, SiCH=*C*H). − MS (EI): *m/z* (%) = 205 (3) [$M^+ - CH_3$], 137 (100), 75 (61).

(E)-4-(Dimethylphenylsilyl)-4-pentenoic Acid (**18**): A solution of CrO_3 (100 mg, 1 mmol) in sulfuric acid (30%, 1 ml) was added to a solution of alcohol **17** (110 mg, 0.5 mmol) in acetone (3 ml) and allowed to stir for 18 hours. After the addition of 1 N NaOH the mixture was washed with *tert*-butyl methyl ether (3 × 5 ml). The aqueous layer was treated with 1 N HCl solution, and extracted with *tert*-butyl methyl ether (3 × 5 ml). After drying ($MgSO_4$), and evaporation of the solvent the pure acid **18** (77 mg, 0.33 mmol, 66%) was obtained without further purification. Colorless oil. − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.36 (s, 6 H, $SiCH_3$), 2.40−2.60 (m, 4 H, 2 × CH_2), 5.87 (d, *J* = 18.6 Hz, 1 H, SiC*H*=CH), 6.18 (dt, *J* = 18.5 Hz, *J* = 6.2 Hz, 1 H, SiCH=C*H*), 7.30−7.40 (m, 3 H, arom. CH), 7.50−7.60 (m, 2 H, arom. CH), 11.20 (s, br, 1 H, COOH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −2.5 (q, $SiCH_3$), 31.3 (t, =CH*C*H_2), 33.1 (t, CH_2C=O), 127.8 (d, 2 C), 129.0 (d, Si*C*H=CH), 129.1 (d), 133.9 (d, 2 C), 138.8 (s), 146.0 (d, SiCH=*C*H), 179.7 (s, COOH). − MS (EI): *m/z* (%) = 233 (8) [M^+ − H], 207 (7), 137 (100), 75 (21). − HRMS calcd. for $C_{13}H_{17}OSi$ [M^+ − H]: 233.0996, found 233.0999.

4-(Tributylstannyl)-3-buten-1-ol (**39**): 3-Butyn-1-ol (605 mg, 10 mmol), tributyltin hydride (1.45 g, 5 mmol) and AIBN (15 mg) were stirred for 3 hours at 60°C. The product was first purified by Kugelrohr distillation and subsequently by flash chromatography (silica gel, *tert*-butyl methyl ether/pentane, 1:10) yielding **39** (1.18 g, 3.3 mmol, 67%) as a 1.5:1 mixture of (*E*/*Z*) isomers. Colorless oil. − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.80−1.00 (m, 15 H, 3 × CH_3, 3 × CH_2), 1.20−1.40 (m, 7 H, 3 × CH_2), 1.40−1.60 (m, 6 H, 3 × CH_2), 2.32 (qd, *J* = 6.6 Hz, *J* = 1.1 Hz, 1 H, C*H*HCH_2OH), 2.42 (qd, *J* = 6.2 Hz, *J* = 1.0 Hz, 1 H, CH*H*CH_2OH), 3.68 (q, *J* = 5.0 Hz, 2 H, CH_2OH), 5.9−6.6 (m, 2 H, CH=CH) − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 9.4/10.3 (t, 3 C, CH_2), 13.7 (q, 3 C, CH_3), 27.2/27.3 (t, 3 C, CH_2), 29.1/29.2 (t, 3 C, CH_2), 40.1/41.2 (t, *C*H_2CH_2OH), 61.4/62.1 (t, *C*H_2OH), 132.3/132.4 (d, Sn*C*H=CH), 144.4/144.8 (d, SnCH=*C*H). − MS (EI): *m/z* (%) = 305 (100) [M^+ − *t*Bu], 249 (33), 193 (31), 177 (31), 137 (23), 121 (23).

General Procedure for the Cyclization of Alkenes with Selenium Electrophiles (GP1): The diselenide **5**[2] (1 eq) was dissolved in anhydrous diethyl ether (0.025 M) under an argon atmosphere, cooled to −78°C and treated with bromine solution (1 eq, 1 M in CCl_4). After 10 min a solution of silver triflate (2.8 eq) in methanol (25 eq) was added and stirred for 10 min at −78°C. The reaction mixture was cooled to −100°C and treated with the alkene. After stirring for 3−4 hours at −100°C, *sym.*-collidine (3 eq) was added followed by a aqueous citric acid solution. 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 cyclization products as colorless oils.

General Procedure for the Radical Cleavage of the Selenium Moiety (GP2):[30] The cyclization product (0.1 mmol), triphenyltin hydride (0.15 mmol) and AIBN (10 mg) were dissolved in toluene (0.5 ml) and heated under reflux for 1 hour. The reaction mixture was purified by flash chromatography (silica gel, *tert*-butyl methyl ether/pentane, 1:50).

General Procedure for the Pummerer Rearrangement (GP3):[31] To a solution of the cyclization product (0.1 mmol) in water (0.1 ml) and THF (0.5 ml) $NaIO_4$ (53 mg, 0.25 mmol) was added and allowed to stir for 3 hours. After a second addition of $NaIO_4$ (40 mg, 0.19 mmol) the mixture was stirred for another 2 hours. After the addition of *tert*-butyl methyl ether and saturated aqueous $NaHCO_3$ solution the organic layer was washed with water, dried ($MgSO_4$) and evaporated. The residue was purified by flash chromatography (silica gel, *tert*-butyl methyl ether/pentane, 1:10).

(2S,3S)-3-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-2-phenyltetrahydrofuran (**19**): GP1, **5** (86 mg, 0.2 mmol) and **7** (30 mg, 0.2 mmol) yields **19** (63 mg, 0.17 mmol, 87%) with 84% *de*. Colorless oil. − $[\alpha]_D^{25}$ = − 23.2 (*c* = 0.31, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.93 (t, *J* = 7.3 Hz, 3 H, CH_3), 1.71 (quint., *J* = 7.3 Hz, 2 H, C$H_2$$CH_3$), 1.95 (s, 1 H, OH), 2.13 (m, 1 H, CH*H*-4), 2.49 (dq, *J* = 13.1 Hz, *J* = 7.5 Hz, 1 H, C*H*H-4), 3.62 (td, *J* = 7.7 Hz, *J* = 5.8 Hz, 1 H, CHSe), 4.03 (td, *J* = 8.4 Hz, *J* = 4.9 Hz, 1 H, CH*H*O), 4.16 (td, *J* = 8.3 Hz, *J* = 7.4 Hz, 1 H, C*H*HO), 4.87 (d, *J* = 6.0 Hz, 1 H, C*H*O), 5.08 (t, *J* = 6.5 Hz, 1 H, C*H*OH), 7.05−7.45 (m, 9 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.4 (q, CH_3), 31.4 (t, *C*$H_2$$CH_3$), 34.3 (t, CH_2-4), 48.0 (d, CHSe), 68.0 (t, CH_2O), 74.7 (d, CHOH), 86.2 (d, CHO), 125.9 (d, 2 C), 126.5 (d), 127.9 (d, 2 C), 128.26 (s), 128.34 (d) 128.5 (d, 2 C), 135.2 (d), 141.3 (s), 146.9 (s). − ^{77}Se NMR ($CDCl_3$, 76 MHz): δ = 314.0. − IR ($CHCl_3$): ν = 3684, 3062, 3005, 2931, 1603, 1463, 1361, 1160, 1073, 970 cm^{-1}. − MS (EI): *m/z* (%) = 362 (7) [M^+], 214 (16), 185 (23), 146 (100), 105 (56), 91 (45), 77 (32). − HRMS calcd. for $C_{19}H_{22}O_2Se$ [M^+]: 362.0785, found 362.0789. − Cleavage (GP2) leads to (*S*)-2-phenyltetrahydrofuran − $[\alpha]_D^{25}$ = −23.8 (*c* = 0.6, $CHCl_3$).[32]

(2R,3R)-3-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-2-(1,1-dimethyl)ethyltetrahydrofuran (**20**): GP1, **5** (64 mg, 0.15 mmol) and **8** (23 mg, 0.18 mmol) yields **20** (42 mg, 0.12 mmol, 68%) with 46% *de*. Colorless oil. − $[\alpha]_D^{25}$ = + 18.1 (*c* = 0.93, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.93 [s, 9 H, $C(CH_3)_3$], 0.99 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.73 (quint., *J* = 7.3 Hz, 2 H, C$H_2$$CH_3$), 1.88−2.04 (s, 1 H, OH), 2.10−2.28 (m, 2 H, CH_2-4), 3.57 (d, *J* = 4.5 Hz, 1 H, CHSe), 3.62−3.70 (m, 1 H, CHO), 3.80−4.01 (m, 2 H, CH_2O), 5.11 (t, *J* = 6.4 Hz, 1 H, C*H*OH), 7.20 (td, *J* = 7.4 Hz, *J* = 1.6 Hz, 1 H, arom. CH), 7.33 (td, *J* = 7.3 Hz, *J* = 1.3 Hz, 1 H, arom. CH), 7.52 (dd, *J* = 7.5 Hz, *J* = 1.3 Hz, 1 H, arom. CH), 7.57 (dd, *J* = 7.7 Hz, *J* = 1.3 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.5 (q, CH_3), 25.9 [q, 3 C, $C(CH_3)_3$], 31.6 (t, *C*$H_2$$CH_3$), 34.7 [s, *C*$(CH_3)_3$], 35.7 (t, *C*$H_2$-4), 41.7 (d, CHSe), 67.4 (t, CH_2O), 74.5 (d, CHOH), 92.1 (d, CHO), 126.5 (d), 128.0 (d), 128.1 (d), 129.4 (s), 138.8 (d), 146.7 (d). − ^{77}Se NMR ($CDCl_3$, 76 MHz): δ = 338.0. − IR ($CHCl_3$): ν = 3604, 3438, 3005, 2962, 2868, 2464, 2365, 1072 cm^{-1}. − MS (EI): *m/z* (%) = 342 (50) [M^+], 285 (54), 267 (63), 213 (89), 197 (100), 185 (23), 116 (38), 57 (76). − HRMS calcd. for $C_{17}H_{26}O_2Se$ [M^+]: 342.1098, found 342.1111. − Cleavage (GP2) leads to (*R*)-2-(1,1-dimethylethyl)tetrahydrofuran.[33]

(2S,3S)-3-{2-[(S)-1-Hydroxypropyl]phenyl}selenyl-2-cyclohexyltetrahydrofuran (**21**): GP1, **5** (128 mg, 0.3 mmol) and **9** (92 mg, 0.6 mmol) yields **21** (142 mg, 0.39 mmol, 64%) with 49% *de*. Colorless oil. − $[\alpha]_D^{25}$ = + 0.7 (*c* = 2.1, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.98 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.05−1.50 (m, 6 H, 3 × CH_2), 1.60−1.85 (m, 7 H, 3 × CH_2, OH), 1.97 (m, 1 H, C*H*CHO), 2.25 (m, 2 H, CH_2), 3.50−3.65 (m, 2 H, CHSe, CHO), 3.75−3.90 (m, 2 H, CH_2O), 5.10 (t, *J* = 6.3 Hz, 1 H, C*H*OH), 7.19 (td, *J* = 7.5 Hz, *J* = 1.6 Hz, 1 H), 7.32 (td, *J* = 7.5 Hz, *J* = 1.2 Hz, 1 H, arom. CH), 7.51 (dd, *J* = 7.7 Hz, *J* = 1.6 Hz, 1 H, arom. CH), 7.57 (dd, *J* = 7.7 Hz, *J* = 1.2 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.4 (q, CH_3), 25.9 (t, CH_2), 26.2 (t, CH_2), 26.4 (t, CH_2), 28.2 (t, CH_2), 29.9 (t, CH_2),

31.4 (t, *C*$H_2$$CH_3$), 34.8 (t, CH_2), 41.7 (d, CHSe), 42.4 (d, *C*HCHO), 66.8 (t, CH_2O), 74.7 (d, CHOH), 88.6 (d, *C*HO), 126.4 (d), 127.8 (d), 128.0 (d), 129.0 (s), 134.6 (d), 146.5 (s). − ^{77}Se NMR ($CDCl_3$, 76 MHz): δ = 326.7. − IR ($CHCl_3$): ν = 3604, 3426, 3005, 2929, 2855, 1463, 1450, 1084, 1047, 972 cm^{-1}. − MS (EI): *m/z* (%) = 368 (31) [M^+], 285 (10), 267 (29), 214 (76), 197 (60), 185 (26), 152 (72), 135 (25) 116 (37), 97 (37), 83 (36), 71 (69), 55 (100), 41 (74). − HRMS calcd. for $C_{19}H_{28}O_2Se$ [M^+]: 368.1255, found 368.1271. − Cleavage (GP2) leads to (*S*)-2-cyclohexyltetrahydrofuran. − $[\alpha]_D^{25}$ = −3.8 (*c* = 0.60, $CHCl_3$).[34]

(2R,3R*)-3-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-2-ethyltetrahydrofuran* (**22**): GP1, **5** (88 mg, 0.21 mmol) and **10** (45 mg, 0.45 mmol) yields **22** (70 mg, 0.22 mmol, 60%) with 0% *de*. Colorless oil. − $[\alpha]_D^{25}$ = − 13.1 (*c* = 3.1, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 600 MHz): δ = 0.91 (t, *J* = 7.4 Hz, 3 H, CH_3), 0.95 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.42−1.65 (m, 2 H, CH_2), 1.76 (quint., *J* = 7.2 Hz, 2 H, C*H*$_2$$CH_3$), 1.96−2.04 (m, 1 H, C*H*H-4), 2.33−2.42 (m, 1 H, CH*H*-4), 2.46 (s, 1 H, OH), 3.33−3.38 (m, 1 H, CHSe), 3.68−3.92 (m, 3 H, CH_2O, CHO), 5.10 (t, *J* = 6.3 Hz, 1 H, C*H*OH), 7.18 (td, *J* = 7.4 Hz, *J* = 1.2 Hz, 1 H, arom. CH), 7.32 (t, *J* = 7.4 Hz, 1 H, arom. CH), 7.51 (d, *J* = 7.7 Hz, 1 H, arom. CH), 7.55 (d, *J* = 7.7 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.28 (q, CH_3), 10.33 (q, CH_3), 27.4 (t, CH_2), 31.4 (t, *C*H$_2$$CH_3$), 34.4 (t, CH_2-4), 44.3 (d, *C*HSe), 66.7 (t, CH_2O), 74.6 (d, CHOH), 85.9 (d, CHO), 126.5 (d), 127.8 (d), 128.1 (d), 128.5 (s), 134.8 (d), 146.7 (s). − ^{77}Se NMR ($CDCl_3$, 114 MHz): δ = 316.3 / 311.2. − IR ($CHCl_3$): ν = 3604, 3426, 3005, 2968, 2934, 2877, 1463, 1096, 1047, 972 cm^{-1}. − MS (EI): *m/z* (%) = 314 (24) [M^+], 214 (52), 197 (23), 185 (36), 135 (25), 116 (18), 99 (48), 98 (50), 57 (100). − HRMS calcd. for $C_{15}H_{22}O_2Se$ [M^+]: 314.0785, found 314.0791. − $C_{15}H_{22}O_2Se$ (313.29): calcd. C 57.52, H 7.08; found C 57.54, H 7.02.

(2S,3S)-3-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-2-phenyltetrahydrofuran-4-on (**23**): GP1, **5** (86 mg, 0.2 mmol) and **11** (33 mg, 0.2 mmol) yields **23** (31 mg, 0.083 mmol, 41%) with 72% *de*. Colorless oil. − $[\alpha]_D^{25}$ = + 104.3 (*c* = 1.06, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.92 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.72 (quint, *J* = 7.4 Hz, 2 H, C*H*$_2$$CH_3$), 1.97 (s, br, 1 H, OH), 2.68 (dd, *J* = 18.1 Hz, *J* = 7.6 Hz, 1 H, C*H*HC=O), 3.05 (dd, *J* = 18.1 Hz, *J* = 8.1 Hz, 1 H, CH*H*C=O), 3.84 (td, *J* = 7.7 Hz, *J* = 6.6 Hz, 1 H, CHSe), 5.05 (t, *J* = 6.3 Hz, 1 H, C*H*OH), 5.41 (d, *J* = 6.3 Hz, 1 H, CHO), 7.10−7.54 (m, 9 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.3 (q, CH_3), 31.6 (t, *C*H$_2$$CH_3$), 36.2 (t, *C*H$_2$C=O), 42.6 (d, CHSe), 74.8 (d, CHOH), 85.0 (d, CHO), 125.7 (d, 2 C), 126.4 (s), 126.9 (d), 128.3 (d), 128.9 (d, 2 C), 129.0 (d), 129.4 (d), 136.2 (d), 137.3 (s), 147.5 (s), 174.5 (s, C=O). − IR ($CHCl_3$): ν = 3603, 3444, 3005, 2967, 2931, 1781, 1458, 1163, 975 cm^{-1}. − MS (EI): *m/z* (%) = 376 (5) [M^+], 214 (30), 198 (62), 183 (75), 100 (53), 131 (55), 105 (100), 77 (90). − HRMS calcd. for $C_{19}H_{20}O_3Se$ [M^+]: 376.0578, found 376.0590. − Cleavage (GP2) leads to (*S*)-5-phenyl-tetrahydrofuran-2-on. − $[\alpha]_D^{25}$ = −27.4 (*c* = 0.31, $CHCl_3$).[35]

(R)-2-Phenyl-2-{1-[2-((S)-1-hydroxypropyl)phenylselenyl]-methyl}tetrahydrofuran (**24**): GP1, **5** (86 mg, 0.2 mmol) and **12** (53 mg, 0.2 mmol) yields **24** (31 mg, 0.083 mmol, 45%) with 78% *de*. Colorless oil. − $[\alpha]_D^{25}$ = + 8.5 (*c* = 0.94, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.91 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.25 (s, br, 1 H, OH), 1.69 (quint., *J* = 7.4 Hz, 2 H, CH_2CH_3), 1.75−2.10 (m, 2 H, CH_2-3), 2.20−2.40 (m, 2 H, CH_2-4), 3.35 (d, *J* = 12.0 Hz, 1 H, C*H*HSe), 3.42 (d, *J* = 12.0 Hz, 1 H, CH*H*Se), 3.94 (q, *J* = 5.6 Hz, 1 H, C*H*HO), 4.06 (q, *J* = 7.1 Hz, 1 H, CH*H*O), 4.90 (t, *J* = 6.3 Hz, 1 H, C*H*OH), 7.00−7.50 (m, 9 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.3 (q, CH_3), 25.9 (t, CH_2-3), 31.0 (t, *C*H$_2$$CH_3$), 38.2 (t, CH_2-4), 42.8 (t, CH_2Se), 68.2 (t, CH_3O), 74.3 (d, CHOH), 86.2 (s, CO), 125.3 (d, 2 C), 126.0 (d), 126.8 (d), 127.4 (d), 127.8 (d), 128.0 (d, 2 C), 130.0 (s), 134.2 (d), 145.6 (s), 146.2 (s). − IR ($CHCl_3$): ν = 3604, 3433, 3006, 2969, 2933, 2877, 1463, 1046, 971 cm^{-1}. − MS (EI): *m/z* (%) = 376 (8) [M^+], 147 (100), 105 (37), 91 (20), 77 (17). − HRMS calcd. for $C_{20}H_{24}O_2Se$ [M^+]: 376.0941, found 376.0956. − The assignment of the stereochemistry was done in analogy to lactone **25**.

(R)-5-Phenyl-5-{1-[2-((S)-1-hydroxypropyl)phenylselenyl]-methyl}tetrahydrofuran-2-on (**25**): GP1, **5** (66 mg, 0.15 mmol) and **13** (59 mg, 0.33 mmol) yields **25** (69 mg, 0.13 mmol, 58%) with 85% *de*. Colorless oil. − $[\alpha]_D^{25}$ = +39.7 (*c* = 0.65, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.90 (t, *J* = 5.5 Hz, 3 H, CH_3), 1.63 (m, 1 H, OH), 1.70 (nonett, *J* = 5.6 Hz, 2 H, C*H*$_2$$CH_3$), 2.43−2.56 (m, 2 H, CH_2-4), 2.70−2.80 (m, 2 H, CH_2-3), 3.42 (s, 2 H, CH_2Se), 4.98 (t, *J* = 4.8 Hz, 1 H, C*H*OH), 7.13 (td, *J* = 5.7 Hz, *J* = 1.3 Hz, 1 H, arom. CH), 7.27−7.40 (m, 6 H, arom. CH), 7.46 (dd, *J* = 5.8 Hz, *J* = 1.0 Hz, 1 H, arom. CH), 7.49 (dd, *J* = 5.9 Hz, *J* = 1.1 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.4 (q, CH_3), 29.1 (t, CH_2-4), 31.8 (t, *C*H$_2$$CH_3$), 34.0 (t, CH_2-3), 42.6 (t, CH_3Se), 74.2 (d, CHOH), 88.4 (s, CO), 124.6 (d, 2 C), 126.5 (d), 128.09 (d), 128.15 (d), 128.2 (d), 128.7 (d, 2 C), 129.4 (s), 134.3 (d), 142.4 (s), 147.0 (s), 176.5 (s, C=O). − IR ($CHCl_3$): ν = 3440, 3374, 2935, 1775, 1398, 1136, 1051, 1016 cm^{-1}. − MS (EI): *m/z* (%) = 390 (5) [M^+], 214 (23), 185 (15), 161 (100), 105 (21), 91 (22), 77 (18). − HRMS calcd. for $C_{20}H_{22}O_3Se$ [M^+]: 390.0734, found 390.0741. − $C_{20}H_{22}O_3Se$ (389.30): calcd. C 61.71, H 5.70; found C 61.70, H 5.94. − Cleavage (GP2) leads to (*R*)-5-methyl-5-phenyltetrahydrofuran-2-on. − $[\alpha]_D^{25}$ = +22.2 (*c* = 0.15, $CHCl_3$).[36]

(R)-N-tert-Butyloxycarbonyl-2-phenyl-2-{1-[2-((S)-1-hydroxypropyl)phenylselenyl]methyl}pyrrolidin (**26**): GP1, **5** (23 mg, 0.05 mmol) and **14** (34 mg, 0.13 mmol) yields **26** (19 mg, 0.04 mmol, 40%) with 45% *de*. Colorless oil. − $[\alpha]_D^{25}$ = −3.8 (*c* = 0.46, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.89 [s, 9 H, $C(CH_3)_3$], 0.95 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.20−1.90 (m, 6 H, CH_2CH_3, CH_2-4, OH, C*H*H-3), 2.01 (dd, *J* = 10.2 Hz, *J* = 6.0 Hz, 1 H, CH*H*-3), 2.53 (m, 1 H, C*H*HN), 3.72 (m, 1 H, C*H*HN), 3.90 (s, 1 H, C*H*HSe), 3.91 (s, 1 H, CH*H*Se), 5.22 (t, *J* = 6.6 Hz, 1 H, C*H*OH), 7.10−7.30 (m, 7 H, arom. CH), 7.52 (d, *J* = 7.6 Hz, 1 H, arom. CH), 7.61 (d, *J* = 7.5 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.4 (q, CH_3), 21.1 (t, CH_2-4), 27.8 [q, 3 C, $C(CH_3)_3$], 31.8 (t, *C*H$_2$$CH_3$), 42.0 (t, CH_2-3), 42.5 (t, CH_2Se), 49.8 (t, CH_3-5), 68.4 (s, CN), 74.1 (d, CHOH), 80.2 [s, *C*$(CH_3)_3$], 125.0 (d, 2 C), 126.5 (d), 126.9 (d), 127.8 (d), 128.1 (d, 2 C), 128.3 (d), 130.9 (s), 135.5 (d), 146.6 (s), 147.8 (s), 154.6 (s, C=O). − IR ($CHCl_3$): ν = 3443, 3005, 2974, 2932, 2878, 1682, 1393, 1125, 977 cm^{-1}. − MS (EI): *m/z* (%) = 475 (7) [M^+], 319 (47), 246 (72), 190 (100), 146 (97), 73 (33). − HRMS calcd. for $C_{25}H_{33}NO_3Se$ [M^+]: 475.1626, found 475.1614. − The assignment of the stereochemistry was done in analogy to lactone **25**.

(3R,4R*)-4-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-4-(dimethylphenylsilyl)-3-methoxybutan-1-ol* (**27**): GP1, **5** (62 mg, 0.145 mmol) and **15** (80 mg, 0.3 mmol) yields **27** (26 mg, 0.058 mmol, 20%) with 78% *de*. Colorless oil. − $[\alpha]_D^{25}$ = −8.2 (*c* = 0.91, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 600 MHz): δ = 0.47 (s, 3 H, $SiCH_3$), 0.52 (s, 3 H, $SiCH_3$), 0.87 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.25 (s, br, 1 H, OH), 1.49−1.55 (m, 1 H, C*H*HCH_2OH), 1.71 (quint., *J* = 7.1 Hz, 2 H, C*H*$_2$$CH_3$), 2.04−2.11 (m, 1 H, CH*H*CH_2OH), 2.38 (s, br, 1 H, OH), 3.01 (s, 3 H, CH_3O), 3.05 (d, *J* = 2.7 Hz, 1 H, CHSiSe), 3.58−3.68 (m, 3 H, C*H*$_2$OH, C*H*OCH_3), 4.84 (t, *J* = 6.6 Hz, 1 H, CHOH), 7.10 (td, *J* = 7.5 Hz, *J* = 1.3 Hz, 1 H, arom. CH), 7.24

(td, J = 7.0 Hz, J = 1.7 Hz, 1 H, arom. CH), 7.30−7.51 (m, 4 H, arom. CH), 7.54 (dd, J = 7.7 Hz, J = 1.1 Hz, 1 H, arom. CH), 7.60 (dd, J = 7.7 Hz, J = 1.4 Hz, 2 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −2.9 (q, $SiCH_3$), −2.3 (q, $SiCH_3$), 10.4 (q, CH_3), 31.5 (t, CH_2CH_3), 37.1 (t, CH_2CH_2OH), 38.0 (d, CHSeSi), 56.6 (q, CH_3O), 60.9 (t, CH_2OH), 75.1 (d, CHOH), 83.2 (d, $CHOCH_3$), 126.5 (d), 127.7 (d), 128.0 (d), 127.8 (d, 2 C), 129.4 (d), 131.5 (s), 134.1 (d, 2 C), 135.9 (d), 135.9 (s), 146.5 (s). − ^{77}Se NMR ($CDCl_3$, 114 MHz): δ = 220.7. − IR ($CHCl_3$): ν = 3603, 3450, 3005, 2964, 2932, 2876, 1463, 1428, 1112, 1074, 840 cm^{-1}. − MS (EI): *m/z* (%) = 452 (5) [M^+], 286 (55), 239 (13), 213 (30), 135 (100), 89 (30), 71(55). − HRMS calcd. for $C_{22}H_{32}O_3SeSi$ [M^+]: 452.1286, found 452.1277. − $C_{22}H_{32}O_3SeSi$ (451.48): calcd. C 58.53, H 7.14; found C 58.78, H 7.17.

(3R)-3-[2-((S)-1-Hydroxypropyl)phenyl]selenyl-3-[(diphenylmethyl)silyl]tetrahydrofuran* (**28**): GP1, **5** (86 mg, 0.2 mmol) and **16** (107 mg, 0.4 mmol) yields **28** (36 mg, 0.086 mmol, 22%) with 9% *de*. Colorless oil. − $[\alpha]_D^{25}$ = −26.9 (*c* = 0.26, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 600 MHz): 1. diastereomer: δ = 0.82 (t, J = 7.4 Hz, 3 H, CH_3), 0.83 (s, 3 H, $SiCH_3$), 1.60 (s, 1 H, OH), 1.62 (quint., J = 7.0 Hz, 1 H, C*H*HCH_3), 1.79 (quint., J = 7.3 Hz, 1 H, C*H*HCH_3), 2.14−2.23 (m, 1 H, C*H*-4), 2.29−2.39 (m, 1 H, CH*H*-4), 4.02 (d, J = 9.8 Hz, 2 H, CH_2-5), 4.64 (s, 1 H, C*H*-2), 4.66 (s, 1 H, CH*H*-2), 5.27 (t, J = 6.6 Hz, 1 H, C*H*OH), 7.10−7.70 (m, 14 H, arom. CH). − ^{1}H NMR ($CDCl_3$, 600 MHz): 2. diastereomer: δ = 0.83 (t, J = 7.5 Hz, 3 H, CH_3), 0.84 (s, 3 H, $SiCH_3$), 1.59 (s, 1 H, OH), 1.61 (quint., J = 7.2 Hz, 1 H, C*H*HCH_3), 1.78 (quint., J = 7.4 Hz, 1 H, C*H*HCH_3), 2.30−2.40 (m, 1 H, C*H*-4), 2.40−2.50 (m, 1 H, CH*H*-4), 3.55 (s, 1 H, C*H*-2), 3.58 (s, 1 H, CH*H*-2), 3.86 (td, J = 8.2 Hz, J = 3.4 Hz, 2 H), 5.19 (t, J = 6.9 Hz, 1 H, C*H*OH), 7.1−7.7 (m, 14 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −4.8/−4.7 (q, $SiCH_3$), 10.4/10.5 (q, CH_3), 28.7/31.1 (s, CHSeSi), 30.6/30.7 (t, CH_2CH_3), 35.6/37.8 (t, CH_2-4), 66.2/66.9 (t, CH_2-5), 74.4/74.6 (d, CHOH), 79.2 (t, CH_2-2), 127.0/127.1 (d), 128.0/128.1 (d, 4 C), 128.9/129.1 (s), 129.7/129.8 (d), 129.8/130.1 (d), 129.9/130.0 (d, 2 C), 133.8/134.1 (s, 2 C), 135.26/135.33 (d, 4 C), 139.38/139.43 (d), 150.2/150.4 (s). − ^{77}Se NMR ($CDCl_3$, 114 MHz): 1. diastereomer δ = 384.6. − ^{77}Se NMR ($CDCl_3$, 114 MHz): 2. diastereomer δ = 332.6.[37] − IR ($CHCl_3$): ν = 3389, 3006, 2965, 2930, 1428, 1111, 950, 835 cm^{-1}. − MS (EI): *m/z* (%) = 482 (7) [M^+], 333 (16), 267 (9), 251 (12), 214 (9), 197 (100), 183 (13), 137 (16), 105 (16). − HRMS calcd. for $C_{26}H_{30}O_2SeSi$ [M^+]: 482.1180, found 482.1166.

(2R,1'R*)-[2-(1-{2-[(S)-1-Hydroxypropyl]phenyl}selenyl)-1-(dimethylphenyl)silylmethyl]tetrahydrofuran* (**29**): GP1, **5** (86 mg, 0.2 mmol) and **17** (138 mg, 0.6 mmol) yields **29** (59 mg, 0.136 mmol, 34%) with 82% *de*. Colorless oil. − $[\alpha]_D^{25}$ = − 57.0 (*c* = 2.41, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 600 MHz): δ = 0.43 (s, 3 H, $SiCH_3$), 0.46 (s, 3 H, $SiCH_3$), 0.90 (t, J = 7.4 Hz, 3 H, CH_3), 1.60−1.90 (m, 7 H, 3 × CH_2, OH), 3.11 (d, J = 3.8 Hz, 1 H, CHSiSe), 3.62 (dt, J = 7.1 Hz, J = 5.8 Hz, 1 H, C*H*HO), 3.70 (dt, J = 8.2 Hz, J = 6.6 Hz, 1 H, CH*H*O), 4.16 (ddd, J = 8.5 Hz, J = 5.5 Hz, J = 4.1 Hz, 1 H, CHO), 4.89 (t, J = 6.3 Hz, 1 H, C*H*OH), 7.11 (td, J = 7.5 Hz, J = 1.6 Hz, 1 H, arom. CH), 7.21 (td, J = 7.5 Hz, J = 1.1 Hz, 1 H, arom. CH), 7.30−7.37 (m, 4 H, arom. CH) 7.52 (dd, J = 8.0 Hz, J = 1.4 Hz, 2 H, arom. CH), 7.75 (dd, J = 7.9 Hz, J = 1.2 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −3.1 (q, $SiCH_3$), −2.6 (q, $SiCH_3$), 10.3 (q, CH_3), 27.6 (t, CH_2-4), 30.9 (t, CH_2-3), 31.0 (t, CH_2CH_3), 39.2 (d, CHSeSi), 67.7 (t, CH_2O), 74.2 (d, CHOH), 80.3 (d, CHO), 125.9 (d), 127.3 (d), 127.7 (d), 127.8 (d, 2 C), 129.2 (d), 131.6 (s), 134.0 (d, 2 C), 134.3 (d), 137.3 (s), 145.8 (s). − ^{77}Se NMR ($CDCl_3$, 114 MHz): δ = 204.6. − IR ($CHCl_3$): ν = 3435, 3376, 2920, 1770, 1456, 1398, 1136, 1051, 1016 cm^{-1}. − MS (EI): *m/z* (%) = 434 (12) [M^+], 319 (22), 235 (21), 213 (19), 198 (31), 183 (23), 165 (26), 135 (100), 115 (18). − HRMS calcd. for $C_{22}H_{30}O_2SeSi$ [M^+]: 434.1180, found 434.1174. − $C_{22}H_{30}O_2SeSi$ (433.47): calcd. C 60.96, H 6.98; found C 60.73, H 7.05.

(2R,1'R*)-[2-(1-{2-[(S)-1-Hydroxypropyl]phenyl}selenyl)-1-(dimethylphenyl)silylmethyl]tetrahydrofuran-5-on* (**30**): GP1, **5** (25 mg, 0.06 mmol) and **18** (30 mg, 0.13 mmol) yields **30** (12 mg, 0.028 mmol, 27%) with 80% *de*. Colorless oil. − $[\alpha]_D^{25}$ = −58.0 (*c* = 0.70, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 600 MHz): δ = 0.49 (s, 3 H, $SiCH_3$), 0.52 (s, 3 H, $SiCH_3$), 0.91 (t, J = 7.4 Hz, 3 H, CH_3), 1.60 (s, br, 1 H, OH), 1.64 (quint., J = 7.6 Hz, 2 H, CH_2CH_3), 1.95 (dq, J = 12.8 Hz, J = 8.4 Hz, 1 H, C*H*H-4), 2.16 (qdd, J = 10.1 Hz, J = 7.0 Hz, J = 3.9 Hz, 1 H, CH*H*-4), 2.46 (dt, J = 17.9 Hz, J = 9.8 Hz, 1 H, C*H*HC=O), 2.54 (ddd, J = 17.9 Hz, J = 10.1 Hz, J = 3.9 Hz, 1 H, CH*H*C=O), 3.10 (d, J = 4.0 Hz, 1 H, CHSeSi), 4.82 (td, J = 7.2 Hz, J = 4.0 Hz, 1 H, CHO), 4.89 (t, J = 6.4 Hz, 1 H, CHOH), 7.17 (td, J = 7.6 Hz, J = 1.5 Hz, 1 H, arom. CH), 7.26 (td, J = 7.5 Hz, J = 1.0 Hz, 1 H, arom. CH), 7.35 (m, 4 H, arom. CH), 7.54 (dd, J = 8.0 Hz, J = 1.4 Hz, 2 H, arom. CH), 7.67 (dd, J = 7.8 Hz, J = 1.2 Hz, 1 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −3.1 (q, $SiCH_3$), −2.8 (q, $SiCH_3$), 10.2 (q, CH_3), 27.6 (t, CH_2-4), 29.2 (t, CH_2-3), 31.4 (t, CH_2CH_3), 38.7 (d, CHSeSi), 74.2 (d, CHOH), 81.6 (d, CHO), 126.0 (d), 127.9 (d), 128.0 (d, 2 C), 128.2 (d), 129.7 (d), 130.4 (s), 133.9 (d, 2 C), 134.3 (d), 136. 0 (s), 145.9 (s), 176.4 (s, C=O). − ^{77}Se NMR ($CDCl_3$, 114 MHz): δ = 193.2. − IR ($CHCl_3$): ν = 3435, 3376, 2920, 1770, 1456, 1398, 1136, 1051, 1016 cm^{-1}. − MS (EI): *m/z* (%) = 448 (9) [M^+], 323 (9), 236 (64), 214 (14), 195 (32), 183 (15), 157 (19), 135 (100), 117 (35), 115 (35), 75 (34). − HRMS calcd. for $C_{22}H_{28}O_3SeSi$ [M^+]: 448.0973, found 448.0959.

(1RS,2RS)-1-Phenyl-2-(phenylselenyl)tetrahydrofuran (**34**): GP1, PhSeBr (70 mg, 0.3 mmol) and **33** (89 mg, 0.6 mmol) yields **34** (18 mg, 0.06 mmol, 20%). Colorless oil. − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 2.24 (m, 1 H, C*H*H-4), 2.49 (m, 1 H, CH*H*-4), 3.98 (td, J = 8.0 Hz, J = 5.6 Hz, 1 H, C*H*H-5), 4.16 (td, J = 6.1 Hz, J = 5.3 Hz, 1 H, CH*H*-5), 3.34 (q, J = 7.4 Hz, 1 H, CHSe), 5.21 (d, J = 6.3 Hz, 1 H, C*H*OH), 7.10−7.45 (m, 10 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 34.5 (t, CH_2-4), 47.3 (d, CHSe), 67.4 (t, CH_2-5), 83.0 (d, CHO), 126.3 (d, 2 C), 127.3 (d), 127.8 (d), 127.9 (d, 2 C), 129.0 (s), 129.1 (d, 2 C), 134.1 (d, 2 C), 141.3 (s). − IR ($CHCl_3$): ν = 3064, 3006, 2884, 1580, 1478, 1453, 1438, 1090, 1048, 1023 cm^{-1}. − MS (EI): *m/z* (%) = 304 (63) [M^+], 198 (50), 183 (33), 157 (32), 146 (100), 117 (97), 105 (96), 91 (80), 77 (78). − HRMS calcd. for $C_{16}H_{16}OSe$ [M^+]: 304.0366, found 304.0367.

(1RS,2SR)-1-Phenyl-2-(phenylselenyl)tetrahydrofuran (**35**): GP1, PhSeBr (47 mg, 0.2 mmol) and **7** (48 mg, 0.34 mmol) yields **35** (44 mg, 0.145 mmol, 73%). Colorless oil. − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 2.14 (m, 1 H, C*H*H-4), 2.47 (m, 1 H, CH*H*-4), 3.57 (q, J = 6.7 Hz, 1 H, CHSe), 4.04 (td, J = 7.8 Hz, J = 7.7 Hz, 1 H, C*H*H-5), 4.17 (td, J = 8.0 Hz, J = 5.2 Hz, 1 H, CH*H*-5), 4.84 (d, J = 6.3 Hz, 1 H, C*H*OH), 7.20−7.40 (m, 8 H, arom. CH), 7.49 (dd, J = 7.5 Hz, J = 1.6 Hz, 2 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 34.2 (t, CH_2-4), 47.6 (d, CHSe), 67.9 (t, CH_2-5), 86.1 (d, CHOH), 125.9 (d, 2 C), 127.7 (d), 127.8 (d), 128.3 (d, 2 C), 128.8 (s), 129.0 (d, 2 C), 134.7 (d, 2 C), 141.3 (s). − IR ($CHCl_3$): ν = 3064, 3006, 2873, 1579, 1478, 1454, 1438, 1066, 1023 cm^{-1}. − MS (EI): *m/z* (%) = 304 (55) [M^+], 198 (44), 183 (29), 157 (33), 146 (100), 117 (93), 105 (83), 91 (82), 77 (66). − HRMS calcd. for $C_{16}H_{16}OSe$ [M^+]: 304.0366, found 304.0360.

(1R,2R)-1-{2-[(S)-1-Hydroxypropyl]phenyl}seleno-1-(trimethyl)silyl-2-methoxy-2-phenylethan (**37**): GP1, **5** (65 mg, 0.15

mmol) and **36** (59 mg, 0.33 mmol) yields **37** (65 mg, 0.154 mmol, 52%) with 85% *de*. Colorless oil. − $[\alpha]_D^{25}$ = −88.2 (*c* = 0.94, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): [= 0.04 (s, 9 H, $Si(CH_3)_3$], 0.94 (t, *J* = 7.4 Hz, 3 H, CH_3), 1.71 (quint., *J* = 7.1 Hz, 2 H, CH_2CH_3), 2.09 (s, br, 1 H, OH), 2.82 (d, *J* = 6.1 Hz, 1 H, CHSeSi), 3.20 (s, 3 H, CH_3O), 4.47 (d, *J* = 6.1 Hz, 1 H, $CHOCH_3$), 4.97 (t, *J* = 6.3 Hz, 1 H, CHOH), 7.04 (td, *J* = 7.3 Hz, *J* = 1.6 Hz, 1 H, arom. CH), 7.13−7.23 (m, 6 H, arom. CH), 7.31−7.37 (m, 2 H, arom. CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = −0.5 [q, 3 C, $Si(CH_3)_3$], 10.4 (q, CH_3), 31.3 (t, CH_2), 45.4 (d, CHSeSi), 57.1 (q, CH_3O), 74.6 (d, CHOH), 86.5 (d, $CHOCH_3$), 126.1 (d), 127.18 (d), 127.23 (d, 2 C), 127.5 (d), 127.7 (d), 128.0 (d, 2 C), 131.2 (s), 133.8 (d), 141.1 (s), 145.7 (s). − IR ($CHCl_3$): ν = 3604, 3063, 3005, 2964, 1601, 1454, 1135, 1090, 1049, 1016, 859 cm^{-1}. − MS (EI): *m*/*z* (%) = 422 (3) [M^+], 318 (30), 214 (39), 199 (26), 185 (20), 121 (100), 91 (42), 73 (52). − HRMS: calcd. for $C_{21}H_{30}O_2SiSe$ [M^+]: 422.1180, found 422.1194. − Pummerer-reaction (GP3) of **37** (55 mg, 0.113 mmol) yields (*R*)-2-methoxy-2-phenylacetaldehyde **38** (12 mg, 0.079 mmol, 70%).[38]

4-{[(S)-1-Hydroxypropyl]phenylselenyl}-3-buten-1-ol (**40**): GP1, **5** (87 mg, 0.2 mmol) and **39** (144 mg, 0.4 mmol) yields **40** (22 mg, 0.075 mmol, 20%) as a mixture of (*E*/*Z*)-isomers (1.5:1), which could not be separated. Colorless oil. − $[\alpha]_D^{25}$ = −58.7 (*c* = 1.07, $CHCl_3$). − ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.92−1.01 (m, 3 H, CH_3), 1.30−1.40 (m, 1 H, OH), 1.70−1.90 (m, 3 H, CH_2CH_3, OH), 2.30−2.55 (m, 2 H, CH_2CH_2OH), 3.60−3.80 (m, 2 H, CH_2OH), 4.98−5.04 (m, 1 H, CHOH), 7.10−7.65 (m, 4 H, arom. CH), (*E*)-isomer: 5.89 (dt, *J* = 15.2 Hz, *J* = 7.3 Hz, 1 H, SeCH=CH), 6.46 (d, *J* = 15.2 Hz, 1 H, SeCH=CH), (*Z*)-isomer: 6.12 (dt, *J* = 9.0 Hz, *J* = 7.2 Hz, 1 H, SeCH=CH), 6.53 (d, *J* = 9.0 Hz, 1 H, SeCH=CH). − ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 10.3 (q, CH_3), 31.1/31.2 (t, CH_2CH_3), 34.6/37.6 (t, CH_2CH_2OH), 61.6/61.7 (t, CH_2CH_2OH), 74.4/74.5 (d, CHOH), 120.1/123.6 (d), 126.3/126.6 (d), 128.1 (d, 2 C), 128.8 (s), 131.4/133.0 (d), 133.8/134.7 (d), 145.5/145.9 (s). − IR ($CHCl_3$): ν = 3604, 3426, 3006, 2966, 2940, 2878, 1464, 1045, 1007, 972 cm^{-1}. − MS (EI): *m*/*z* (%) = 286 (24) [M^+], 213 (18), 197 (13), 185 (23), 157 (17), 117 (16), 91 (15), 77 (100). − HRMS calcd. for $C_{13}H_{18}O_2Se$ [M^+]: 286.0472, found 286.0475.

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[38] Separation of enantiomers: GC Chrompack, β-CD-permethylated, 25 m, 75°C, rate 0.5°C/min. Spectroscopic data see: E. P. Lodge, C. H. Heathcock, *J. Am. Chem. Soc.* **1987**, *109*, 3353−3361. Assignment of the absolute configuration by independent synthesis see ref.[16]

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