Tetrahedron: *Asymmetry* 24 (2013) 1089–1096

Contents lists available at SciVerse ScienceDirect

Tetrahedron: *Asymmetry*

journal homepage: www.elsevier.com/locate/tetasy

Synthesis and reactions of the optically active selenols derived from monoterpenes

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ARTICLE INFO

Article history:
Received 17 June 2013
Accepted 18 July 2013
Available online 27 August 2013

Dedicated to Professor Jacek Gawroński on the occasion of his 70th birthday

ABSTRACT

A convenient methodology for the synthesis of optically active selenols, derived from *p*-menthane, carane, and pinane, is described. The selenols were oxidized with air to give the optically active diselenides, and were also converted into the corresponding allylic selenides via reaction with *Z*- and *E*-cinnamyl, geranyl, and neryl chlorides. Oxidation of the allylic selenides with *m*CPBA gave the optically active alcohols via [2,3]-sigmatropic rearrangement of the in situ generated allylic selenoxides.

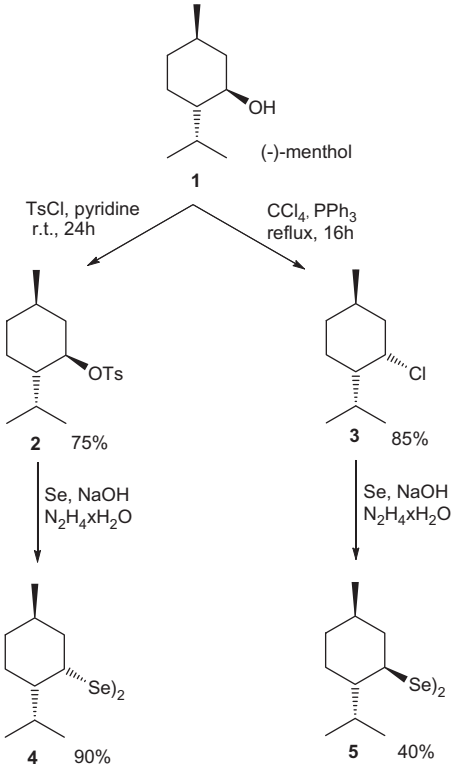
1. Introduction

In recent years, the increasing role of organoselenium compounds in organic synthesis has been observed. Their specific reactivity makes them potential reagents and catalysts for the formation of new carbon–oxygen, carbon–nitrogen and carbon–carbon bonds. Diselenides are one of the most important groups of organoselenium compounds. They can be converted simply into electrophilic, nucleophilic, and radical reagents.[1] Particularly interesting are the optically active diselenides, which have found many applications in asymmetric synthesis.[2] Their biological and pharmacological functions have also been tested.[3]

Recently we have developed a convenient methodology for the synthesis of optically active terpene diselenides via reaction of terpene tosylates, chlorides, and epoxides with sodium diselenide.[4–10] For example, dineomenthyl diselenide **4** and dimenthyl diselenide **5** were obtained from the corresponding menthyl tosylate **2** and neomenthyl chloride **3** (Scheme 1), and used as electrophilic selenium reagents for the addition to double bonds.

Diselenides can be reduced to the corresponding selenolates by $NaBH_4$, $LiAlH_4$, Na/NH_3, or other reagents.[11] Herein we report a convenient synthesis of terpene selenols from terpene diselenides via reduction with $NaBH_4$ and acidification. To the best of our knowledge terpene selenols have not yet been isolated. Only menthyl and camphor selenols have been prepared in situ by Santi et al. and were used in a reaction with methyl iodide to give methyl terpenyl selenides in good yields.[12] A convenient methodology for the synthesis of optically active selenols is desired due to their possible synthetic applications. For example, they react with alkyl halides or with epoxides to give the corresponding selenides.[12,13]

Another aim of our investigation was the use of terpene selenols for the synthesis of allylic terpene selenides, and their transformations into optically active allylic alcohols. Oxidation of allylic



Scheme 1. Synthesis of diselenides **4** and **5** from (−)-menthol **1**.

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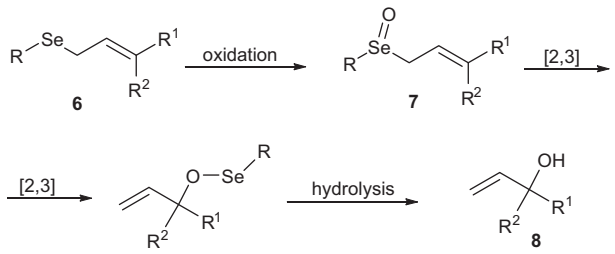
http://dx.doi.org/10.1016/j.tetasy.2013.07.018

selenides **6** into allylic selenoxides **7** and spontaneous [2,3]-sigmatropic rearrangement gives chiral alcohols **8** (Scheme 2).[14]

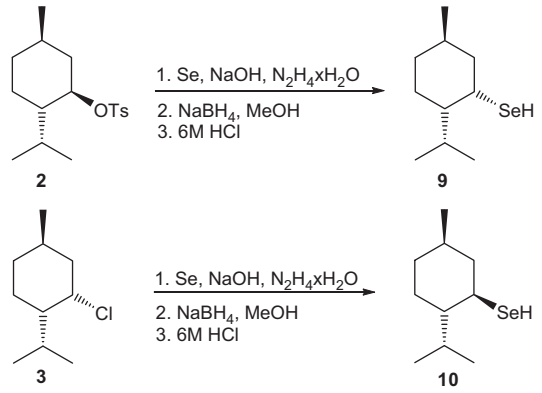


Scheme 2. Synthesis of allylic alcohols **8** via [2,3]-sigmatropic rearrangement.

Davis et al.[15] first reported the asymmetric oxidation of allylic selenides and the enantioselective [2,3]-rearrangement of allylic selenoxides. Oxidation of optically active allylic selenides gave allylic selenoxides, which undergo diastereoselective [2,3]-sigmatropic rearrangements.[16] These transformations have found interesting applications in asymmetric synthesis.[14–19] In our previous investigations, allylic phenyl selenides were used for the synthesis of allylic terpene alcohols, tosylamides, and methyl carbamates.[20–23]

2. Results and discussion

The first goal of our research was the synthesis of terpene selenols. They were obtained in good yields by the reaction of terpene tosylates or terpene chlorides with sodium diselenide, followed by reduction of the crude reaction mixture with sodium borohydride and acidification with aq 6 M HCl. Selenols were purified by distillation under reduced pressure. Examples of the synthesis of neomenthyl selenol **9** and menthyl selenol **10**, obtained from menthyl tosylate **2** and neomenthyl chloride **3**, are presented in Scheme 3.



Scheme 3. Synthesis of neomenthyl selenol **9** and menthyl selenol **10**.

Analogously, caranyl **11**, isocaranyl **12**, pinocamphyl **13**, isopinocamphyl **14**, and myrtanyl **15** selenols have been obtained (Table 1). The structures of the selenols were determined on the basis of ^{1}H, ^{13}C, and ^{77}Se NMR. It was possible to observe a significant change in the position of the signal in the ^{77}Se NMR spectra of selenols in comparison to the signals of the diselenides, for example, the chemical shift for neomenthyl selenol **9** is −26.4 ppm, and 290.4 ppm for dineomenthyl diselenide **16**.[5,7] Additionally specific Se–H signals can be observed in the ^{1}H NMR spectra of selenols in the range from 0.13 to −0.75 ppm. The syntheses of the corresponding terpenyl tosylates and chlorides are described in our earlier papers.[5,7]

Selenols **9**–**15** have been oxidized with air in methanol in the presence of a small amount of NaOH, and diselenides **16**–**22** have been isolated quantitatively. Selenols treated with butyl lithium, and then with *E*-cinnamyl ($R^{1}Cl$), *Z*-cinnamyl ($R^{2}Cl$), geranyl ($R^{3}Cl$), and neryl ($R^{4}Cl$) chlorides were transformed into the corresponding allylic selenides **23**–**50**. The selenides derived from caranyl selenol and neryl and geranyl chlorides could not be isolated. In some cases, isomerization during the reaction was observed (Table 1 entries 5 and 6). The chlorides $R^{1-4}Cl$ were prepared by the reaction of *E*- and *Z*-cinnamyl alkohols, geraniol, and nerol with PPh_3 and CCl_4 according to the literature.[5] Examples of the synthesis of dineomenthyl diselenide **16**, *E*-cinnamyl **23**, *Z*-cinnamyl **24**, geranyl **25**, and neryl **26** neomenthyl selenides are shown in Scheme 4.

Allylic terpenyl selenides **23**–**50** were oxidized with *m*CPBA to the corresponding selenoxides and after [2,3]-sigmatropic rearrangement *E*- and *Z*-allylic cinnamyl selenides gave the allylic alcohol **51**, while geranyl or neryl terpenyl selenides yielded the optically active linolool **52** (Scheme 5). The results of the synthesis of the alcohols are presented in Table 2.

Alcohols **51** and **52** were obtained with good yields and with moderate to low enantioselectivities. Better selectivities were observed for the oxidation of allylic selenides derived from selenides bearing *p*-menthyl and carane groups than those containing a more hindered pinane moiety. Alcohol **51** obtained from *E*- and *Z*-cinnamyl terpenyl selenides **23**–**24**, **27**–**28**, **31**–**32**, and **35**–**36** had the same configuration, whereas geranyl and neryl terpenyl selenides **25**–**26**, **29**–**30**, and **37**–**38** gave **52** with the opposite configuration.

3. Conclusion

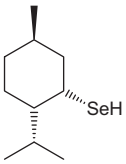
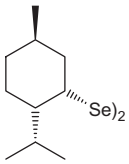
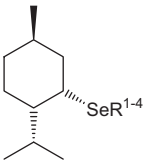
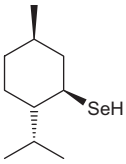
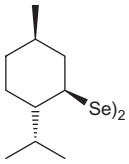
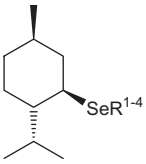
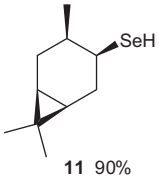
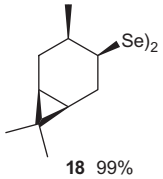
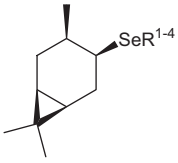
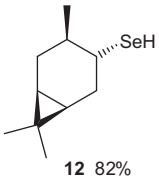
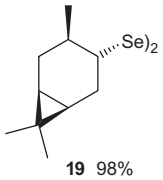
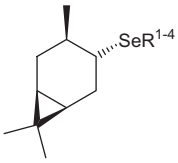
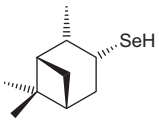
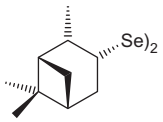
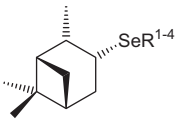
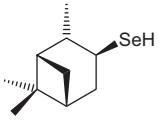
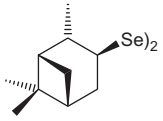
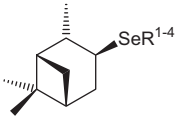
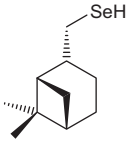
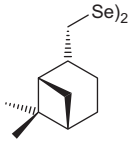
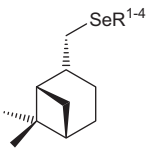
We have developed a convenient methodology for the synthesis of terpene selenols, a novel family of organoselenium compounds derived from terpenes. The simple synthesis of selenols makes them interesting systems for future explorations as reagents in a wide range of reactions. For example, they can be converted into the corresponding allylic selenides, and used in the asymmetric synthesis of allylic alcohols via a [2,3]-sigmatropic rearrangement of the allylic selenoxides. We have also shown that the stereochemical course of the oxidation reaction depends not only on the terpenyl group, but also on the *E*- or *Z*-configuration of the allylic selenides.

4. Experimental

4.1. General

Melting points were measured with a Büchi Tottoli SPM-20 heating unit and are uncorrected. NMR spectra were recorded on Bruker AM-300, Varian 200, Bruker Avance III/400 or Bruker Avance III/700. Chemical shifts are expressed in parts per million (ppm) relative to TMS. Elemental analyses were performed on a Vario MACRO CHN analyzer. Optical rotations were measured in 50-mm cells with a polAAr 3000 polarimeter. TLC was conducted on precoated silica gel plates (Merck 60F254) and the spots were visualized under UV light. Column chromatography was carried out on a column using Silica Gel 60 Merck (70–230 mesh). All reactions requiring anhydrous conditions were conducted in a flame-dried apparatus.

Table 1
Synthesis of diterpenyl diselenides **16**–**22** and allylic terpenyl selenides **23**–**50**

Entry	Selenol	Diselenide	Allylic selenide		Yield (%)
1				**23** R^1	92
				24 R^2	40
				25 R^3	65
				26 R^4	72
	9 86%	**16** 98%			
2				**27** R^1	75
				28 R^2	60
				29 R^3	47
				30 R^4	60
	10 64%	**17** 96%			
3				**31** R^1	68
				32 R^2	69
				33 R^3	–
				34 R^4	–
	11 90%	**18** 99%			
4				**35** R^1	70
				36 R^2	49
				37 R^3	43
				38 R^4	61
	12 82%	**19** 98%			
5				**39** R^1	50
				40 R^2	50
				41 R^3	44
				64% **42** R^4 + 36% **41** R^3	51
	13 67%	**20** 96%			
6				**43** R^1	66
				44 R^2	69
				74% **45** R^3 + 26% **46** R^4	57
				46 R^4	52
	14 71%	**21** 96%			
7				**47** R^1	78
				48 R^2	76
				49 R^3	45
				50 R^4	65
	15 85%	**22** 99%			

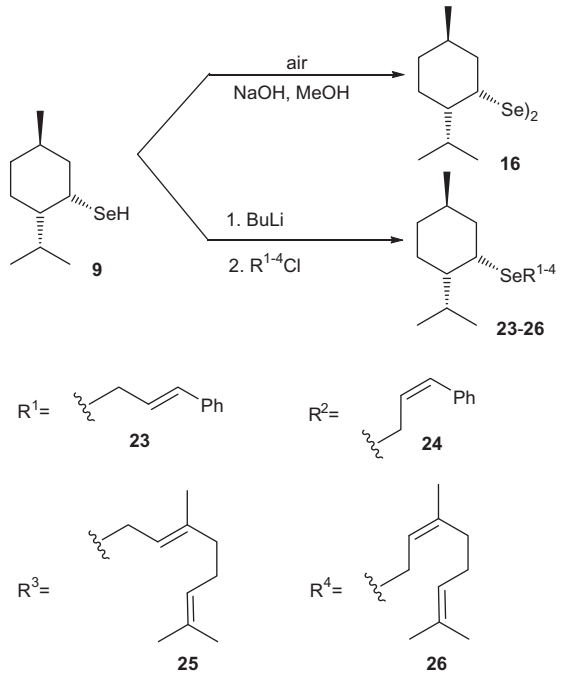
4.2. General procedure for the synthesis of terpene selenols

The standard synthesis of diselenides was conducted under argon, by adding dropwise hydrazine hydrate (0.6 ml) into a mixture of selenium (22 mmol) and sodium hydroxide (33 mmol) in DMF (20 ml). After heating at 100 °C for 15 min, the reaction mixture was cooled to ambient temperature and the respective tosylate (22 mmol) or chloride (22 mmol) was added. The solution was heated again at 100 °C for 1 h (2 h for chlorides). The reaction mixture was cooled, poured into water (100 ml), and extracted with petroleum ether (3 × 100 ml). The combined ether layers were washed with water (100 ml), dried over anhydrous $MgSO_4$, and evaporated.

Next, $NaBH_4$ (5.00 g, 132.2 mmol) was added carefully to a crude diselenide (23.9 mmol) in anhydrous ethanol (50 ml) under an argon atmosphere (decoloration of the reaction mixture was observed). The mixture was cooled to ca. 0 °C and water (10 ml) was added. The precipitate was dissolved by adding 6 M HCl (pH ~3) and extracted with petroleum ether (3 × 50 ml). The combined ether layers were dried over anhydrous $MgSO_4$, the solvent was evaporated, and the selenol was purified by distillation under reduced pressure.

4.2.1. (1*S*,2*S*,5*R*)-(+)-Neomenthyl selenol 9

Purification by distillation 56–58 °C/0.5 mmHg. Yield 86%; colorless liquid; $[\alpha]_D^{20} = +47.5$ (*c* 1.61, $CHCl_3$); ^1H NMR (400 MHz,



Scheme 4. Synthesis of diterpenyl diselenides **16** and allylic terpenyl selenides **23–26**.

Table 2
Oxidation of allylic selenides to allylic alcohols **51** and **52**

Entry	Allylic selenide	Allylic alcohol	Config.	Yield (%)	% ee
1	**23**	**51**	(*S*)	18	20
2	**24**	**51**	(*S*)	86	20
3	**25**	**52**	(*S*)	52	25
4	**26**	**52**	(*R*)	57	25
5	**27**	**51**	(*R*)	43	20
6	**28**	**51**	(*R*)	53	27
7	**29**	**52**	(*R*)	66	22
8	**30**	**52**	(*S*)	63	17
9	**31**	**51**	(*S*)	43	34
10	**32**	**51**	(*S*)	26	27
11	**35**	**51**	(*S*)	59	23
12	**36**	**51**	(*S*)	33	34
13	**37**	**52**	(*R*)	35	27
14	**38**	**52**	(*S*)	58	27
15	**39**	**51**	(*S*)	25	2
16	**40**	**51**	(*R*)	53	1
17	**41**	**52**	(*R*)	46	2
18	**43**	**51**	(*S*)	59	8
19	**44**	**51**	(*R*)	86	10
20	**46**	**52**	(*R*)	69	6
21	**47**	**51**	(*S*)	*66*	2
22	**48**	**51**	(*S*)	80	3
23	**49**	**52**	(*R*)	34	6
24	**50**	**52**	(*S*)	35	4

$CDCl_3$), $\delta = -0.75$ (d, 1H, Se–H, $J = 5.8$ Hz), 0.86 (d, 3H, CH_3, $J = 6.2$ Hz), 0.88 (d, 3H, CH_3, $J = 5.4$ Hz), 0.91 (d, 3H, CH_3, $J = 5.4$ Hz), 1.22–1.77 (m, 8H), 1.98 (dq, 1H, $J = 2.4$, 2.4, 13.6 Hz), 3.78 (m, 1H) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$), $\delta = 20.4$ (CH_3), 20.8 (CH_3), 22.1 (CH_3), 25.2 (CH_2), 27.1 (CH), 31.6 (CH), 35.3 (CH_2), 40.0 (CH), 44.4 (CH_2), 48.4 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), $\delta = -26.38$ ppm. Elemental Anal. Calcd for $C_{10}H_{20}Se$ (219.23): C, 54.78; H, 9.19. Found: C, 54.65; H, 9.32.

4.2.2. (1*R*,2*S*,5*R*)-(−)-Menthyl selenol 10

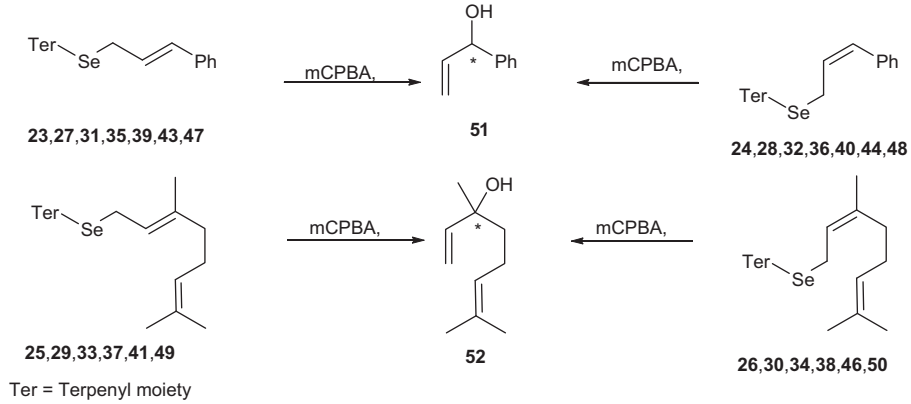
Purification by distillation 64–68 °C/0.5 mmHg. Yield 64%; colorless liquid; $[\alpha]_D^{20} = -50.8$ (*c* 0.94, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$), $\delta = -0.54$ (d, 1H, Se–H, $J = 4.5$ Hz), 0.73 (d, 3H, CH_3, $J = 6.9$ Hz), 0.86 (d, 3H, CH_3, $J = 6.1$ Hz), 0.91 (d, 3H, CH_3, $J = 6.9$ Hz), 1.21–1.78 (m, 7H), 2.12–2.22 (m, 2H), 3.62–3.81 (m, 1H) ppm; ^{13}C NMR (100.6, $CDCl_3$), $\delta = 14.9$ (CH_3), 21.4 (CH_3), 22.0 (CH_3), 24.2 (CH_2), 30.0 (CH), 34.7 (CH), 34.9 (CH_2), 40.3 (CH), 49.0 (CH_2), 50.5 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), $\delta = 92.93$ ppm. Elemental Anal. Calcd for $C_{10}H_{20}Se$ (219.23): C, 54.78; H, 9.19. Found: C, 54.99; H, 9.08.

4.2.3. (1*S*,3*R*,4*S*,6*R*)-(+)-Caranyl selenol 11

Purification by distillation 80–82 °C/1.0 mmHg. Yield 90%; colorless liquid; $[\alpha]_D^{20} = +6.6$ (*c* 2.76, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$), $\delta = -0.63$ (d, 1H, Se–H, $J = 4.6$ Hz), 0.44–0.56 (m, 1H), 0.67–0.85 (m, 2H), 0.95 (d, 3H, CH_3, $J = 6.6$ Hz), 0.97 (s, 3H, CH_3), 0.99 (s, 3H, CH_3), 1.38–1.52 (m, 1H), 1.61–1.74 (m, 2H), 2.29–2.45 (m, 1H), 3.52–3.65 (m, 1H) ppm; ^{13}C NMR (100,6, $CDCl_3$), $\delta = 16.0$ (CH_3), 17.7 (C), 20.2 (CH), 20.7 (CH_3), 22.8 (CH), 24.8 (CH_2), 28.4 (CH_3), 29.1 (CH_2), 31.3 (CH), 37.7 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), $\delta = 22.98$ ppm. Elemental Anal. Calcd for $C_{10}H_{18}Se$ (217.24): C, 55.29; H, 8.35. Found: C, 55.12; H, 8.46.

4.2.4. (1*S*,3*R*,4*R*,6*R*)-(−)-Isocaranyl selenol 12

Purification by distillation 51–52 °C/0.2 mmHg. Yield 82%; colorless liquid; $[\alpha]_D^{20} = -151.9$ (*c* 1.07, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$), $\delta = -0.36$ (d, 1H, Se–H, $J = 5.0$ Hz), 0.49–0.63 (m, 1H), 0.68–0.92 (m, 2H), 0.94 (s, 3H, CH_3), 0.95 (s, 3H, CH_3), 0.97 (d, 3H, CH_3, $J = 7.4$ Hz), 1.18–1.55 (m, 1H), 1.93–2.11 (m, 2H), 2.45 (ddd, 1H, $J = 1.4$, 6.4, 14.6 Hz), 2.58–2.64 (m, 1H) ppm; ^{13}C NMR



Scheme 5. Synthesis of allylic alcohols **51** and **52**.

(100.6, $CDCl_3$), δ = 15.6 (CH_3), 17.4 (C), 20.6 (CH), 21.0 (CH), 21.6 (CH_3), 28.8 (CH_3), 29.3 (CH_2), 33.6 (CH_2), 36.5 (CH), 41.1 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 105.93 ppm. Elemental Anal. Calcd for $C_{10}H_{18}Se$ (217.24): C, 55.29; H, 8.35. Found: C, 55.68; H, 8.44.

4.2.5. (1*S*,2*S*,3*R*,5*R*)-(−)-Pinocamphyl selenol 13

Purification by distillation 50–52 °C/0.2 mmHg. Yield 67%; colorless liquid; $[\alpha]_D^{20} = -28.2$ (*c* 1.65, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = −0.46 (d, 1H, Se–H, *J* = 5.8 Hz), 1.02 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.21 (d, 3H, CH_3, *J* = 6.4 Hz), 1.34 (d, 1H, *J* = 10.0 Hz), 1.82–2.01 (m, 2H), 2.07–2.25 (m, 2H), 2.34–2.56 (m, 2H), 3.96–4.13 (m, 1H) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 20.3 (CH_3), 23.7 (CH_3), 27.5 (CH_3), 28.0 (CH_2), 29.5 (CH), 38.3 (CH), 39.6 (C), 39.8 (CH_2), 42.5 (CH), 48.9 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 47.26 ppm. Elemental Anal. Calcd for $C_{10}H_{18}Se$ (217.24): C, 55.29; H, 8.35. Found: C, 55.42; H, 8.29.

4.2.6. (1*S*,2*S*,3*S*,5*R*)-(+)-Isopinocamphyl selenol 14

Purification by distillation 50–53 °C/0.2 mmHg. Yield 71%; colorless liquid; $[\alpha]_D^{20} = +37.8$ (*c* 0.93, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.13 (d, 1H, Se–H, *J* = 6.0 Hz), 1.01 (s, 3H, CH_3), 1.07 (d, 1H, *J* = 9.6 Hz), 1.09 (d, 3H, CH_3, *J* = 7.2 Hz), 1.19 (s, 3H, CH_3), 1.83 (ddd, 1H, *J* = 2.0, 5.8, 5.8 Hz), 1.90–2.01(m, 1H), 2.17 (ddd, 1H, *J* = 2.0, 9.8, 14.8 Hz), 2.25 (ddd, 1H, *J* = 2.0, 7.2, 7.2 Hz), 2.35–2.63 (m, 2H), 3.33 (dddd, 1H, *J* = 5.8, 7.2, 9.8, 14.8 Hz) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 20.8 (CH_3), 23.3 (CH_3), 28.1 (CH_3), 33.4 (CH), 35.0 (CH_2), 38.4 (C), 40.9 (CH_2), 43.0 (CH), 49.1 (CH), 49.6 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = −171.47 ppm. Elemental Anal. Calcd for $C_{10}H_{18}Se$ (217.24): C, 55.29; H, 8.35. Found: C, 55.15; H, 8.43.

4.2.7. (1*S*,2*R*,5*S*)-(−)-*cis*-Myrtanyl selenol 15

Purification by distillation 60–62 °C/0.5 mmHg. Yield 85%; colorless liquid; $[\alpha]_D^{20} = -46.9$ (*c* 0.98, $CHCl_3$); ^{1}H NMR (700 MHz, $CDCl_3$), δ = −0.72 (t, 1H, Se–H, *J* = 6.9 Hz), 0.87 (d, 1H, *J* = 9.6 Hz), 0.97 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.39–1.55 (m, 1H), 1.77–2.12 (m, 5H), 2.15–2.27 (m, 1H), 2.30–2.38 (m, 1H), 2.62–2.71 (m, 2H) ppm; ^{13}C NMR (176.1, $CDCl_3$), δ = 22.7 (CH_2), 23.2 (CH_3), 25.2 (CH_2), 26.1 (CH_2), 28.0 (CH_3), 33.3 (CH_2), 38.6 (C), 41.3 (CH), 45.0 (CH), 46.2 (CH) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = −28.91 ppm. Elemental Anal. Calcd for $C_{10}H_{18}Se$ (217.24): C, 55.29; H, 8.35. Found: C, 55.12; H, 8.49.

4.3. General procedure for the synthesis of allylic selenides

At first, *n*-BuLi (1.97 ml) was carefully added to the selenol (4.92 mmol) in anhydrous THF (8 ml) at −25 °C under an argon atmosphere. The solution was stirred for 0.5 h and *E*- or *Z*-cinnamyl chloride (0.75 g, 4.92 mmol) in anhydrous THF (8 ml) was added. The solution was then stirred for 0.5 h at 0 °C, and then 0.5 h at 50 °C. The reaction mixture was cooled and poured into water (10 ml). The solvent was evaporated and the reaction mixture was extracted with diethyl ether (3 × 25 ml). The combined ether layers were dried over anhydrous $MgSO_4$, and the solvent was evaporated. The residue was purified by column chromatography, eluting with petroleum ether to give the pure selenide.

4.4. (*E*)-Cinnamyl terpenyl selenides

4.4.1. (1*S*,2*S*,5*R*)-(+)-(*E*)-Cinnamyl neomenthyl selenide 23

Yield 92%; colorless solid; mp 62–63 °C; $[\alpha]_D^{20} = +165.5$ (*c* 1.41, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.78–1.17 (m, 12H), 1.21–1.43 (m, 1H), 1.52–1.82 (m, 3H), 1.83–2.03 (m, 2H), 3.23–3.40 (m, 3H), 6.19–6.44 (m, 2H), 7.18–7.41 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 MHz, $CDCl_3$), δ = 20.7 (CH_3), 20.9 (CH_3), 22.2 (CH_3), 25.3 (CH_2), 27.6 (CH_2), 27.9 (CH), 31.2 (CH), 35.3 (CH_2), 42.1 (CH_2), 44.8 (CH), 49.4 (CH), 126.2 (2 × $CH_{arom.}$), 127.3 ($CH_{arom.}$), 127.3 (CH), 128.5 (2 × $CH_{arom.}$), 130.0 (CH), 137.0 ($C_{arom.}$) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 194.73 ppm. Elemental Anal. Calcd for $C_{19}H_{28}Se$ (335.39): C, 68.04; H, 8.41. Found: C, 67.98; H, 8.35.

4.4.2. (1*R*,2*S*,5*R*)-(−)-(*E*)-Cinnamyl menthyl selenide 27

Yield 75%; colorless liquid; $[\alpha]_D^{20} = -123.2$ (*c* 1.66, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.78 (d, 3H, CH_3, *J* = 7.0 Hz), 0.89 (d, 3H, CH_3, *J* = 6.0 Hz), 0.90 (d, 3H, CH_3, *J* = 7.0 Hz), 1.22–1.43 (m, 3H), 1.63–1.79 (m, 2H), 2.16–2.43 (m, 2H), 2.67–2.83 (m, 1H), 3.40 (dd, 2H, CH_2, *J* = 6.8, 1.4 Hz), 6.22–6.43 (m, 2H), 7.17–7.40 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.2 (CH_3), 21.5 (CH_3), 22.3 (CH_3), 24.1 (CH_2), 25.0 (CH_2), 29.0 (CH), 34.3 (CH), 34.9 (CH_2), 43.6 (CH), 45.3 (CH_2), 47.6 (CH), 126.2 (2 × $CH_{arom.}$), 127.3 ($CH_{arom.}$), 127.4 (CH), 128.5 (2 × $CH_{arom.}$), 131.0 (CH), 137.0 ($C_{arom.}$) ppm. ^{77}Se (38.1 MHz, $CDCl_3$), δ = 278.00 ppm. Elemental Anal. Calcd for $C_{19}H_{28}Se$ (335.39): C, 68.04; H, 8.41. Found: C, 68.25; H, 8.32.

4.4.3. (1*S*,3*R*,4*S*,6*R*)-(+)-(*E*)-Cinnamyl 4-caranyl selenide 31

Yield 68%; colorless liquid; $[\alpha]_D^{20} = +55.5$ (*c* 1.74, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.45–0.93 (m, 3H), 0.95 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 0.99 (d, 3H, CH_3, *J* = 5.2 Hz), 1.44–1.60 (m, 1H), 1.74–1.90 (m, 2H), (ddd, 1H, *J* = 7.0, 8.4, 15.4 Hz), 2.24–2.42 (q, 1H, *J* = 7.2 Hz), 3.36 (d, 2H, *J* = 7.0 Hz), 6.20–6.44 (m, 2H), 7.18–7.40 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.9 (CH_3), 17.6 (C), 20.6 (CH_3), 21.2 (CH), 21.4 (CH), 25.8 (CH_2), 25.9 (CH_2), 26.3 (CH_2), 28.5 (CH_3), 31.2 (CH), 42.0 (CH), 126.2 (2 × $CH_{arom.}$), 127.2 ($CH_{arom.}$), 127.3 (CH), 128.5 (2 × $CH_{arom.}$), 131.0 (CH), 137.0 ($C_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.27; H, 7.91.

4.4.4. (1*S*,3*R*,4*R*,6*R*)-(−)-(*E*)-Cinnamyl 4-isocaranyl selenide 35

Yield 70%; colorless liquid; $[\alpha]_D^{20} = -79.2$ (*c* 1.71, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.52–0.83 (m, 3H), 0.95 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 1.01 (d, 3H, CH_3, *J*= 7.4 Hz), 1.26–1.45 (m, 1H), 1.98–2.18 (m, 3H), 2.24–2.42 (m, 1H), 3.25–3.43 (m, 2H), 6.19–6.40 (m, 2H), 7.18–7.38 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.6 (CH_3), 17.5 (C), 20.6 (CH), 20.7 (CH), 21.8 (CH_3), 25.1 (CH_2), 28.9 (CH_3), 29.6 (CH_2), 30.2 (CH_2), 34.7 (CH), 44.1 (CH), 126.2 (2 × $CH_{arom.}$), 127.2 ($CH_{arom.}$), 127.3 (CH), 128.5 (2 × $CH_{arom.}$), 131.2 (CH), 137.0 ($C_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.64; H, 7.79.

4.4.5. (1*S*,2*S*,3*R*,5*R*)-(+)-(*E*)-Cinnamyl pinocamphyl selenide 39

Yield 50%; colorless liquid; $[\alpha]_D^{20} = +52.0$ (*c* 1.70, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.82–0.95 (m, 1H), 0.99 (s, 3H, CH_3), 1.12 (d, 3H, CH_3, *J* = 7.2 Hz), 1.20 (s, 3H, CH_3), 1.84 (ddd, 1H, *J* = 1.8, 6.0, 6.0 Hz), 1.96 (ddd, 1H, *J* = 3.0, 5.7, 8.7 Hz), 2.13 (ddd, 1H, *J* = 1.8, 7.2, 7.2 Hz), 2.25 (ddd, 1H, *J* = 2.7, 6.6, 14.1 Hz), 2.35–2.44 (m, 1H), 2.49–2.59 (m, 1H), 3.13 (ddd, 1H, *J* = 6.3, 7.5, 9.9 Hz), 3.46 (ddd, 2H, *J* = 6.0, 11.7, 20.1 Hz), 6.28–6.48 (m, 2H), 7.19–7.42 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 21.3 (CH_3), 23.2 (CH_3), 25.7 (CH_2), 27.9 (CH_3), 34.1 (CH_2), 36.7 (CH), 38.8 (CH_2), 42.5 (CH), 45.2 (CH), 48.5 (CH), 126.2 (2 × $CH_{arom.}$), 127.2 ($CH_{arom.}$), 127.3 (CH), 128.5 (2 × $CH_{arom.}$), 131.0 (CH), 137.0 ($C_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.31; H, 7.92.

4.4.6. (1*S*,2*S*,3*S*,5*R*)-(−)-(*E*)-Cinnamyl isopinocamphyl selenide 43

Yield 66%; colorless liquid; $[\alpha]_D^{20} = -116.3$ (*c* 1.51, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.81–0.93 (m, 1H), 1.03 (s, 3H, CH_3), 1.20 (s, 3H, CH_3), 1.23 (d, 3H, CH_3, *J* = 7.6 Hz), 1.86–2.01 (m, 2H), 2.09–2.28 (m, 2H), 2.30–2.63 (m, 2H), 3.31–3.57 (m, 2H), 3.75 (q,

1H, J = 8.6 Hz), 6.25–6.48 (m, 2H), 7.18–7.42 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 19.8 (CH_3), 23.6 (CH_3), 25.2 (CH_2), 27.6 (CH_3), 28.1 (CH_2), 32.8 (CH), 36.5 (CH_2), 38.1 (CH), 42.1 (CH), 49.0 (CH), 126.3 (2 × CH$_{arom.}$), 127.1 (CH$_{arom.}$), 127.4 (CH), 128.6 (2 × CH$_{arom.}$), 131.0 (CH), 137.0 (C$_{arom.}$) ppm; ^{77}Se (38.1, $CDCl_3$), δ = 264.84 ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.71; H, 7.78.

4.4.7. (1*S*,2*R*,5*S*)-(−)-(*E*)-Cinnamyl *cis*-myrtanyl selenide 47

Yield 78%; colorless liquid; $[\alpha]_D^{20} = -35.7$ (c 1.66, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.79–0.93 (m,1H), 0.98 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.38–1.62 (m,1H), 1.79–2.09 (m, 5H), 2.18–2.38 (m, 2H), 2.64 (dd, 2H, CH_2, J = 2.8, 7.8 Hz), 3.33 (dd, 2H, CH_2, J = 1.0, 5.8 Hz), 6.19–6.40 (m, 2H), 7.18–7.40 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 23.1 (CH_3), 23.3 (CH_2), 25.5 (CH_2), 26.2 (CH_2), 28.1 (CH_3), 31.5 (CH_2), 33.6 (CH_2), 38.7 (C), 41.4 (CH), 41.9 (CH), 46.5 (CH), 126.3 (2 × CH$_{arom.}$),127.0 (CH$_{arom.}$), 127.3 (CH), 128.5 (2 × CH$_{arom.}$), 131.2 (CH), 137.0 (C$_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.57; H, 7.80.

4.5. (*Z*)-Cinnamyl terpenyl selenides

4.5.1. (1*S*,2*S*,5*R*)-(+)-(*Z*)-Cinnamyl neomenthyl selenide 24

Yield 40%; colorless solid; mp 58–60 °C; $[\alpha]_D^{20} = +138.9$ (c 1.42, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.89 (d, 3H, CH_3, J = 6.3 Hz), 0.90 (d, 3H, CH_3, J = 6.6 Hz), 0.91 (d, 3H, CH_3, J = 6.3 Hz), 0.94–1.17 (m, 3H), 1.21–1.43 (m, 1H), 1.52–1.70 (m,1H), 1.73–1.85 (m, 2H), 1.86–2.03 (m, 2H), 3.29–3.45 (m, 3H), 6.23–6.43 (m, 2H), 7.18–7.39 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 MHz, $CDCl_3$), δ = 20.7 (CH_3), 20.9 (CH_3), 22.2 (CH_3), 25.3 (CH_2), 27.6 (CH_2), 28.0 (CH), 31.2 (CH), 35.3 (CH_2), 42.2 (CH_2), 44.8 (CH), 49.4 (CH), 126.2 (2 × CH$_{arom.}$), 127.3 (CH$_{arom.}$), 127.3 (CH), 128.5 (2 × CH$_{arom.}$), 130.9 (CH), 137.0 (C$_{arom.}$) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 193.81 ppm. Elemental Anal. Calcd for $C_{19}H_{28}Se$ (335.39): C, 68.04; H, 8.41. Found: C, 67.87; H, 8.29.

4.5.2. (1*R*,2*S*,5*R*)-(−)-(*Z*)-Cinnamyl menthyl selenide 28

Yield 60%; colorless liquid; $[\alpha]_D^{20} = -133.3$ (c 1.57, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.85 (d, 3H, CH_3, J = 6.3 Hz), 0.87 (d, 3H, CH_3, J = 6.6 Hz), 0.89 (d, 3H, CH_3, J = 6.0 Hz), 0.95–1.13 (m, 2H), 1.18–1.37 (m, 1H), 1.55–1.67 (m, 1H), 1.69–2.01 (m, 5H), 3.29–3.57 (m, 3H), 5.89 (dt, 1H, J = 8.4, 11.1 Hz), 6.48 (dt, 1H, J = 1.2, 11.1 Hz), 7.18–7.38 (m, 5H) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 20.6 (CH_3), 20.8 (CH_3), 20.9 (CH_3), 22.1 (CH_3), 25.2 (CH_2), 27.2 (CH_2), 27.6 (CH), 31.2 (CH), 35.3 (CH_2), 45.5 (CH), 49.4 (CH), 126.8 (2 × CH$_{arom.}$), 128.2 (CH$_{arom.}$), 128.2 (CH), 128.8 (2 × CH$_{arom.}$), 130.2 (CH), 136.7 (C$_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{28}Se$ (335.39): C, 68.04; H, 8.41. Found: C, 68.16; H, 8.38.

4.5.3. (1*S*,3*R*,4*S*,6*R*)-(+)-(*Z*)-Cinnamyl 4-caranyl selenide 32

Yield 69%; colorless liquid; $[\alpha]_D^{20} = +61.9$ (c 0.91, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.45–0.93 (m, 3H), 0.95 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 0.99 (d, 3H, CH_3, J = 5.2 Hz), 1.44–1.60 (m, 1H), 1.74–1.90 (m, 2H), (ddd, 1H, J = 7.0, 8.4, 15.4 Hz), 2.24–2.42 (q, 1H, J = 7.2 Hz), 3.36 (d, 2H, J = 7.0 Hz), 6.20–6.44 (m, 2H), 7.18–7.40 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.9 (CH_3), 17.6 (C), 20.6 (CH_3), 21.2 (CH), 21.4 (CH), 25.8 (CH_2), 25.9 (CH_2), 26.3 (CH_2), 28.5 (CH_3), 31.2 (CH), 42.0 (CH), 126.2 (2 × CH$_{arom.}$), 127.2 (CH$_{arom.}$), 127.3 (CH), 128.5 (2 × CH$_{arom.}$), 131.0 (CH), 137.0 (C$_{arom.}$) ppm. ^{77}Se (38.1 MHz, $CDCl_3$), δ = 255.40 ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.27; H, 7.91.

4.5.4. (1*S*,3*R*,4*R*,6*R*)-(−)-(*Z*)-Cinnamyl isocaranyl selenide 36

Yield 49%; colorless liquid; $[\alpha]_D^{20} = -100.55$ (c 0.90, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.58–0.88 (m, 3H), 0.91 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 1.05 (d, 3H, CH_3, J = 6.4 Hz), 1.25–1.47 (m, 1H), 1.97–2.19 (m, 3H), 2.24–2.42 (m, 1H), 3.27–3.47 (m, 2H), 6.22–6.43 (m, 2H), 7.19–7.39 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.5 (CH_3), 17.3 (C), 20.5 (CH), 20.6 (CH), 21.7 (CH_3), 24.9 (CH_2), 28.8 (CH_3), 29.5 (CH_2), 30.0 (CH_2), 34.6 (CH), 43.9 (CH), 126.1 (2 × CH$_{arom.}$), 127.0 (CH$_{arom.}$), 127.2 (CH), 128.4 (2 × CH$_{arom.}$), 130.9 (CH), 136.9 (C$_{arom.}$) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.33; H, 8.01.

4.5.5. (1*S*,2*S*,3*R*,5*R*)-(−)-(*Z*)-Cinnamyl pinocamphyl selenide 40

Yield 50%; colorless liquid; $[\alpha]_D^{20} = +43.0$ (c 1.53, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 1.02 (d, 3H, CH_3, J = 7.2 Hz), 1.21 (s, 3H, CH_3), 1.23(s, 3H, CH_3), 1.81–1.99 (m, 2H), 2.02–2.18 (m, 3H), 2.22–2.58 (m, 2H), 3.26–3.48 (m, 2H), 3.60–3.81 (m, 1H), 6.28–6.46 (m, 2H), 7.18–7.39 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 19.8 (CH_3), 23.5 (CH_3), 25.2 (CH_2), 27.6 (CH_3), 28.1 (CH_2), 32.8 (CH), 36.4 (CH_2), 38.1 (CH), 42.0 (CH), 49.0 (CH), 126.2 (2 × CH$_{arom.}$), 128.2 (CH$_{arom.}$), 128.5 (2 × CH$_{arom.}$), 128.8 (CH), 130.9 (CH), 137.0 (C$_{arom.}$) ppm. ^{77}Se (38.1 MHz, $CDCl_3$), δ = 264.61 ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.56; H, 7.83.

4.5.6. (1*S*,2*S*,3*S*,5*R*)-(−)-(*Z*)-Cinnamyl isopinocamphyl selenide 44

Yield 69%; colorless liquid; $[\alpha]_D^{20} = -95.2$ (c 1.70, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.85–0.91 (m, 1H), 0.99 (s, 3H, CH_3), 1.11 (d, 3H, CH_3, J = 7.2 Hz), 1.19 (s, 3H, CH_3), 1.83 (ddd, 1H, J = 1.8, 6.0, 6.0 Hz), 1.95 (ddd, 1H, J = 3.0, 5.7, 8.7 Hz), 2.12 (ddd, 1H, J = 2.1, 7.5, 7.5 Hz), 2.24 (ddd, 1H, J = 2.4, 6.3, 13.8 Hz), 2.34–2.43 (m, 1H), 2.48–2.58 (m, 1H), 3.45 (ddd, 1H, J = 6.3, 7.5, 9.9 Hz), 3.12 (ddd, 2H, J = 7.8, 12.3, 21.0 Hz), 6.22–6.40 (m, 2H), 7.18–7.48 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 21.3 (CH_3), 23.2 (CH_3), 25.7 (CH_2), 27.9 (CH_3), 34.1 (CH_2), 36.7 (CH_2), 38.8 (CH), 42.5 (CH), 45.2 (CH), 48.5 (CH), 126.1 (2 × CH$_{arom.}$), 127.2 (CH$_{arom.}$), 127.2 (CH), 128.5 (2 × CH$_{arom.}$), 131.0 (CH), 137.0 (C$_{arom.}$) ppm; Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.56; H, 7.93.

4.5.7. (1*S*,2*R*,5*S*)-(−)-(*Z*)-Cinnamyl *cis*-myrtanyl selenide 48

Yield 76%; colorless liquid; $[\alpha]_D^{20} = -33.0$ (c 1.68, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.79 (d,1H, J = 9.2 Hz), 0.95 (s, 3H, CH_3), 1.17 (s, 3H, CH_3), 1.36–1.57 (m,1H), 1.67–2.17 (m, 6H), 2.20–2.36 (m, 1H), 2.62 (dd, 2H, CH_2, J = 3.2, 7.6 Hz), 3.34 (dd, 2H, CH_2, J = 1.4, 8.6 Hz), 5.77–5.94 (m, 1H), 6.43–6.51 (m, 1H), 7.19–7.38 (m, 5H$_{arom.}$) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 20.6 (CH_2), 22.8 (CH_2), 23.1 (CH_3), 26.1 (CH_2), 27.9 (CH_3), 31.8 (CH_2), 33.4 (CH_2), 41.2 (CH), 41.6 (CH), 46.3 (CH), 126.8 (CH$_{arom.}$),128.2 (2 × CH$_{arom.}$), 128.7 (2 × CH$_{arom.}$), 128.8 (CH), 130.2 (CH), 136.6 (C) ppm. Elemental Anal. Calcd for $C_{19}H_{26}Se$ (333.37): C, 68.46; H, 7.86. Found: C, 68.25; H, 7.95.

4.6. Geranyl terpenyl selenides

4.6.1. (1*S*,2*S*,5*R*)-(+)-Geranyl neomenthyl selenide 25

Yield 65%; colorless liquid; $[\alpha]_D^{20} = +81.3$ (c 1.23, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.89 (d, 6H, 2 × CH_3, J = 6.6 Hz), 0.91 (d, 3H, CH_3, J = 6.4 Hz), 0.97–1.15 (m, 1H), 1.16–1.28 (m, 2H), 1.48–1.58 (m, 4H), 1.59 (s, 3H, CH_3), 1.66 (s, 3H, CH_3), 1.68 (s, 3H, CH_3), 1.82–2.18 (m, 6H), 3.05–3.35 (m, 3H), 5.15–5.24 (m, 1H), 5.65 (t, 1H, J = 8.8 Hz) ppm; ^{13}C NMR (50.3 MHz, $CDCl_3$), δ = 15.9 (CH_3), 17.6 (CH_3), 20.6 (CH_2), 20.7 (CH_3), 21.0 (CH_3), 22.2

(CH_3), 25.7 (CH_3), 26.5 (CH_2), 27.4 (CH_2), 27.9 (CH), 31.2 (CH), 35.3 (CH_2), 39.7 (CH_2), 42.4 (CH_2), 44.5 (CH), 49.4 (CH), 121.5 (CH), 124.1 (CH), 131.5 (C), 137.8 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 189.16 ppm. Elemental Anal. Calcd for $C_{20}H_{36}Se$ (355.46): C, 67.58; H, 10.21. Found: C, 67.34; H, 10.33.

4.6.2. (1*R*,2*S*,5*R*)-(−)-Geranyl menthyl selenide 29

Yield 47%; colorless liquid; $[\alpha]_D^{20} = -81.9$ (*c* 1.83, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.74 (d, 3H, CH_3, *J* = 6.9 Hz), 0.82–0.87 (m, 1H), 0.88 (d, 3H, CH_3, *J* = 6.3 Hz), 0.90 (d, 3H, CH_3, *J* = 6.9 Hz), 0.96–1.09 (m, 2H), 1.19–1.39 (m, 4H), 1.59 (s, 3H, CH_3), 1.66 (s, 3H, CH_3), 1.68 (s, 3H, CH_3), 2.00–2.33 (m, 6H), 2.70 (ddd, 1H, *J* = 3.9, 11.5, 11.5 Hz), 3.22 (ddd, 2H, *J* = 8.7, 11.8, 30.5 Hz), 5.05–5.12 (m, 1H), 5.35 (t, 1H, *J* = 8.0 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.1 (CH_3), 15.9 (CH_3), 17.7 (CH_3), 19.2 (CH_2), 21.5 (CH_3), 22.2 (CH_3), 25.0 (CH_2), 25.7 (CH_3), 26.6 (CH_2), 29.0 (CH), 34.4 (CH), 34.9 (CH_2), 39.7 (CH_2), 43.4 (CH), 45.5 (CH_2), 47.5 (CH), 121.4 (CH), 124.1 (CH), 131.6 (C), 137.7 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 269.41 ppm. Elemental Anal. Calcd for $C_{20}H_{36}Se$ (355.46): C, 67.58; H, 10.21. Found: C, 67.42; H, 10.29.

4.6.3. (1*S*,3*R*,4*R*,6*R*)-(−)-Isocaranyl geranyl selenide 37

Yield 43%; colorless liquid; $[\alpha]_D^{20} = -69.3$ (*c* 1.88, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.93 (s, 3H, CH_3), 0.96 (s, 3H, CH_3), 1.02 (d, 2H, CH_2, *J* = 6.4 Hz), 1.11–1.56 (m, 3H), 1.59 (s, 3H, CH_3), 1.65 (s, 3H, CH_3), 1.68 (s, 3H, CH_3),1.92–2.14 (m, 8H), 2.17–2.35 (m, 2H), 3.18 (d, 2H, CH_2, *J* = 8.0 Hz), 5.04–5.11 (m, 1H), 5.34 (t, 1H, *J* = 6.0 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.6 (CH_3), 17.5 (CH_3), 17.6 (C), 20.3 (CH_2), 20.7 (CH), 20.8 (CH), 21.8 (CH_3), 23.5 (CH_3), 25.7 (CH_3), 26.6 (CH_2), 28.8 (CH_3), 29.6 (CH_2), 30.2 (CH_2), 34.9 (CH), 39.6 (CH_2), 44.1 (CH), 121.4 (CH), 124.1 (CH), 131.6 (C), 137.8 (C) ppm;^{77}Se ($CDCl_3$), δ = 295.77 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 68.11; H, 9.61.

4.6.4. (1*S*,2*S*,3*R*,5*R*)-(−)-Geranyl pinocamphyl selenide 41

Yield 44%; colorless liquid; $[\alpha]_D^{20} = -23.0$ (*c* 1.57, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 1.01 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.21 (d, 3H, CH_3, *J* = 6.4 Hz), 1.32-.1.37 (m, 1H), 1.62 (s, 3H, CH_3), 1.69 (s, 3H, CH_3), 1.70 (s, 3H, CH_3), 1.82–2.00 (m, 2H), 2.00–2.65 (m, 8H), 3.23 (d, 2H, CH_2, *J* = 8.0 Hz), 3.69–8.82 (m, 1H), 5.03–5.16 (m, 1H), 5.38 (t, 1H, *J* = 6.8 Hz) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 15.9 (CH_3), 17.7 (CH_3), 19.8 (CH_3), 20.7 (CH_2), 23.5 (CH_3), 25.7 (CH_3), 26.6 (CH_2), 27.6 (CH_3), 28.1 (CH_2), 33.0 (CH), 36.6 (CH_2), 38.3 (CH), 39.5 (CH_2), 39.7 (C), 42.0 (CH), 49.0 (CH), 121.4 (CH), 124.0 (CH), 131.6 (C), 138.0 (C) ppm. ^{77}Se (38.1 MHz, $CDCl_3$), δ = 252.74 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 67.69; H, 9.76.

4.6.5. (1*S*,2*S*,3*S*,5*R*)-(−)-Geranyl isopinocamphyl selenide 45

Yield 51%; mixture 74%: 26%; ^{1}H NMR (200 MHz, $CDCl_3$), δ = 1.02 (s, 3H, $CH_3)_G$, 1.02 (s, 3H, $CH_3)_N$, 1.12 (d, 3H, CH_3, *J* = 7.0 Hz)$_G$, 1.12 (d, 3H, CH_3, *J* = 7.0 Hz)$_N$, 1.19 (s, 3H, $CH_3)_G$, 1.19 (s, 3H, $CH_3)_N$, 1.60 (s, 3H, $CH_3)_G$, 1.62 (s, 3H, $CH_3)_N$, 1.68 (s, 3H, $CH_3)_G$, 1.69 (s, 3H, $CH_3)_G$, 1.69 (s, 3H, $CH_3)_N$, 1.73 (s, 3H, $CH_3)_N$, 1.78–1.98 (m, 3H)$_G$, 1.78–1.98 (m, 3H)$_N$, 2.05–2.09 (m, 5H)$_G$, 2.05–2.09 (m, 5H)$_N$, 2.17–2.70 (m, 3H)$_G$, 2.17–2.70 (m, 3H)$_N$, 3.01–3.37 (m, 3H)$_G$, 3.01–3.37 (m, 3H)$_N$, 5.05–5.22 (m, 1H)$_G$, 5.05–5.22 (m, 1H)$_N$, 5.38 (t, 1H, *J* = 7.8 Hz)$_G$, 5.39 (t, 1H, *J* = 8.0 Hz)$_N$ ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 15.8 ($CH_3)_G$,17.6 ($CH_3)_G$, 17.7 ($CH_3)_N$, 20.6 ($CH_2)_N$, 20.8 ($CH_2)_G$, 21.3 ($CH_3)_G$, 21.3 ($CH_3)_N$, 23.2 ($CH_3)_G$, 23.2 ($CH_3)_N$, 23.4 ($CH_3)_N$, 25.6 ($CH_3)_G$, 25.7 ($CH_3)_N$, 26.6 ($CH_2)_G$, 26.7 ($CH_2)_N$, 27.9 ($CH_3)_G$, 27.9 ($CH_3)_N$, 31.7 ($CH_2)_N$, 34.0 ($CH_2)_N$, 34.1 ($CH_2)_G$, 36.5 ($CH)_G$, 36.9 ($CH)_N$, 38.6 ($C)_N$, 38.7 ($CH_2)_G$, 38.7 ($CH_2)_N$, 38.9 ($C)_G$, 39.6 ($CH_2)_G$, 42.5 ($CH)_G$, 42.5 ($CH)_N$, 45.4 ($CH)_G$, 45.5 ($CH)_N$, 48.4 ($CH)_G$, 48.4 ($CH)_N$, 121.3 ($CH)_G$, 121.8 ($CH)_N$, 124.0 ($CH)_G$, 124.0 ($CH)_N$, 131.5 ($C)_G$, 131.8 ($C)_N$, 137.8 ($C)_G$, 137.9 ($C)_N$ ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 357.75$_G$, 358.76$_N$ ppm.

4.6.6. (1*S*,2*R*,5*S*)-(−)-Geranyl myrtanyl selenide 49

Yield 45%; colorless liquid; $[\alpha]_D^{20} = -114.05$ (*c* 1.61, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.87 (d, 2H, *J* = 9.0 Hz), 0.99 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.58 (s, 3H, CH_3), 1.63 (s, 3H, CH_3), 1.67 (s, 3H, CH_3), 1.74–2.45 (m, 11H), 2.49–2.73 (m, 2H), 3.18 (d, 2H, CH_2, *J* = 8.4 Hz), 5.03–5.18 (m, 1H), 5.34 (t, 1H, *J* = 9.6 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.8 (CH_3), 17.6 (CH_3), 20.6 (CH_2), 23.0 (CH_2), 23.2 (CH_3), 25.6 (CH_3), 26.1 (CH_2), 26.5 (CH_2), 28.0 (CH_3), 31.3 (CH_2), 31.7 (CH_2), 33.4 (CH_2), 38.6 (C), 41.3 (CH), 42.0 (CH), 46.4 (CH), 121.4 (CH), 124.0 (CH), 131.5 (C), 137.8 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 185.71 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 68.05; H, 9.58.

4.7. Neryl terpenyl selenides

4.7.1. (1*S*,2*S*,5*R*)-(+)-Neomenthyl neryl selenide 26

Yield 72%; colorless liquid; $[\alpha]_D^{20} = +48.0$ (*c* 1.88, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.89 (d, 6H, 2 × CH_3, *J* = 6.4 Hz), 0.90 (d, 3H, CH_3, *J* = 6.6 Hz), 0.97–1.15 (m, 1H), 1.16–1.28 (m, 2H), 1.48–1.58 (m, 4H), 1.61 (s, 3H, CH_3), 1.68 (s, 3H, CH_3), 1.71 (s, 3H, CH_3), 1.82–2.18 (m, 6H), 3.05–3.35 (m, 3H), 5.15–5.24 (m, 1H), 5.65 (t, 1H, *J* = 8.8 Hz) ppm; ^{13}C NMR (50.3 MHz, $CDCl_3$), δ = 17.7 (CH_3), 20.6 (CH_3), 20.7 (CH_2), 21.0 (CH_3), 22.2 (CH_3), 23.3 (CH_3), 25.7 (CH_3), 26.7 (CH_2), 27.4 (CH_2), 27.9 (CH), 31.2 (CH), 31.7 (CH_2), 35.4 (CH_2), 42.5 (CH_2), 45.0 (CH), 49.5 (CH), 122.3 (CH), 124.1 (CH), 131.8 (C), 137.9 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 193.09 ppm. Elemental Anal. Calcd for $C_{20}H_{36}Se$ (355.46): C, 67.58; H, 10.21. Found: C, 67.79; H, 10.08.

4.7.2. (1*R*,2*S*,5*R*)-(−)-Menthyl neryl selenide 30

Yield 60%; colorless liquid; $[\alpha]_D^{20} = -63.0$ (*c* 1.95, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$), δ = 0.73 (d, 3H, CH_3, *J* = 6.9 Hz), 0.80–0.86 (m, 1H), 0.88 (d, 3H, CH_3, *J* = 6.0 Hz), 0.89 (d, 3H, CH_3, *J* = 6.9 Hz), 0.95–1.09 (m, 2H), 1.18–1.37 (m, 4H), 1.59 (s, 3H, CH_3), 1.66 (s, 3H, CH_3), 1.70 (s, 3H, CH_3), 2.07–2.34 (m, 6H), 2.67–2.71 (m, 1H), 3.17–3.24 (m, 2H), 5.12–5.25 (m, 1H), 5.37 (t, 1H, *J* = 8.0 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.1 (CH_3), 17.7 (CH_3), 19.0 (CH_3), 21.5 (CH_2), 22.2 (CH_3), 23.4 (CH_3), 25.0 (CH_2), 25.7 (CH_3), 26.7 (CH_2), 29.0 (CH), 31.8 (CH), 34.4 (CH_2), 34.9 (CH_2), 43.7 (CH), 45.5 (CH_2), 47.6 (CH), 122.0 (CH), 124.1 (CH), 131.8 (C), 138.0 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 273.98 ppm. Elemental Anal. Calcd for $C_{20}H_{36}Se$ (355.46): C, 67.58; H, 10.21. Found: C, 67.64; H, 10.16.

4.7.3. (1*S*,3*R*,4*R*,6*R*)-(−)-Isocaranyl neryl selenide 38

Yield 61%; colorless liquid; $[\alpha]_D^{20} = -89.4$ (*c* 0.94, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.93 (s, 3H, CH_3), 0.95 (s, 3H, CH_3), 1.01 (d, 2H, CH_2, *J* = 6.4 Hz), 1.16–1.56 (m, 3H), 1.61 (s, 3H, CH_3), 1.69 (s, 3H, CH_3), 1.71 (s, 3H, CH_3),1.92–2.15 (m,8H), 2.19–2.37 (m, 2H), 3.18 (d, 2H, CH_2, *J* = 8.0 Hz), 5.05–5.21 (m, 1H), 5.34 (t, 1H, *J* = 6.0 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 15.6 (CH_3), 17.5 (C), 17.7 (CH_3), 20.1 (CH_2), 20.7 (CH), 20.8 (CH), 21.8 (CH_3), 23.4 (CH_3), 25.7 (CH_3), 26.7 (CH_2), 28.9 (CH_3), 29.6 (CH_2), 30.2 (CH_2), 31.8 (CH_2), 34.9 (CH), 44.5 (CH), 121.2 (CH), 124.1 (CH), 131.8 (C), 138.0 (C) ppm;^{77}Se ($CDCl_3$), δ = 296.74 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 68.09; H, 9.64.

4.7.4. (1*S*,2*S*,3*R*,5*R*)-(−)-Neryl pinocamphyl selenide 42

Yield 57%; mixture 64%: 36% ^{1}H NMR (200 MHz, $CDCl_3$), δ = 1.01 (s, 3H, $CH_3)_G$, 1.02 (s, 3H, $CH_3)_N$, 1.18 (s, 3H, $CH_3)_G$, 1.19 (s, 3H, $CH_3)_N$, 1.21 (d, 3H, CH_3, *J* = 6.4 Hz)$_G$, 1.22 (d, 3H, CH_3, *J* = 6.4 Hz)$_N$,

1.28-.1.43 (m, 1H)$_G$, 1.28–1.43 (m, 1H)$_N$, 1.61 (s, 3H, CH_3)$_N$, 1.62 (s, 3H, CH_3)$_G$, 1.69 (s, 3H, CH_3)$_G$, 1.69 (s, 3H, CH_3)$_N$, 1.70 (s, 3H, CH_3)$_G$, 1.74 (s, 3H, CH_3)$_N$, 1.82–2.04 (m, 2H)$_G$, 1.82–2.04 (m, 2H)$_N$, 2.05–2.28 (m, 6H)$_G$, 2.05–2.28 (m, 6H)$_N$, 2.28–2.65 (m, 2H)$_G$, 2.28–2.65 (m, 2H)$_N$, 3.23 (d, 2H, CH_2, J = 8.0 Hz)$_G$, 3.23 (d, 2H, CH_2, J = 8.0 Hz)$_N$, 3.69–8.82 (m, 1H)$_G$, 3.65–3.87 (m, 1H)$_N$, 5.03–5.16 (m, 1H)$_G$, 5.03–5.18 (m, 1H)$_N$, 5.38 (t, 1H, J = 6.8 Hz)$_G$, 5.39 (t, 1H, J = 6.8 Hz)$_N$ ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 15.8 (CH_3)$_N$, 15.9 (CH_3)$_G$, 17.7 (CH_3)$_G$, 17.7 (CH_3)$_N$, 19.7 (CH_3)$_N$, 19.8 (CH_3)$_G$, 20.5 (CH_2)$_N$, 20.7 (CH_2)$_G$, 23.5 (CH_3)$_G$, 23.5 (CH_3)$_N$, 25.7 (CH_3)$_G$, 25.7 (CH_3)$_N$, 26.6 (CH_2)$_G$, 26.8 (CH_2)$_N$, 27.6 (CH_3)$_G$, 27.6 (CH_3)$_N$, 28.1 (CH_2)$_G$, 28.1 (CH_2)$_N$, 31.7 (CH_2)$_N$, 33.0 (CH)$_G$, 33.3 (CH)$_N$, 36.6 (CH_2)$_G$, 36.6 (CH_2)$_N$, 38.3 (CH)$_G$, 38.3 (CH)$_N$, 39.5 (CH_2)$_G$, 39.7 (C)$_G$, 39.7 (C)$_N$, 42.0 (CH)$_G$, 42.0 (CH)$_N$, 49.0 (CH)$_G$, 49.0 (CH)$_N$, 121.4 (CH)$_G$, 121.9 (CH)$_N$, 124.0 (CH)$_G$, 124.1 (CH)$_N$, 131.6 (C)$_G$, 131.8 (C)$_N$, 138.0 (C)$_G$, 138.0 (C)$_N$ ppm. ^{77}Se (38.1 MHz, $CDCl_3$), δ = 252.74$_G$, 253.02$_N$ ppm.

4.7.5. (1*S*,2*S*,3*S*,5*R*)-(+)-Isopinocamphyl neryl selenide 46

Yield 52%; colorless liquid; $[\alpha]_D^{20} = +41.4$ (c 1.85, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 1.02 (s, 3H, CH_3), 1.12 (d, 3H, CH_3, J = 7.0 Hz), 1.19 (s, 3H, CH_3), 1.62 (s, 3H, CH_3), 1.69 (s, 3H, CH_3), 1.73 (s, 3H, CH_3), 1.82–1.95 (m, 3H), 2.08–2.09 (m, 5H), 2.19–2.69 (m, 3H), 3.03–3.34 (m, 3H), 5.05–5.22 (m, 1H), 5.39 (t, 1H, J = 8.0 Hz) ppm; ^{13}C NMR (50.3, $CDCl_3$), δ = 17.7 (CH_3), 20.6 (CH_2), 21.3 (CH_3), 23.2 (CH_3), 23.4 (CH_3), 25.7 (CH_3), 26.7 (CH_2), 27.9 (CH_3), 31.7 (CH_2), 34.0 (CH_2), 36.9 (CH), 38.6 (C), 38.7 (CH_2), 42.5 (CH), 45.5 (CH), 48.4 (CH), 121.8 (CH), 124.0 (CH), 131.8 (C), 137.9 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 358.76 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 68.18; H, 9.61.

4.7.6. (1*S*,2*R*,5*S*)-(−)-*cis*-Myrtanyl neryl selenide 50

Yield 65%; colorless liquid; $[\alpha]_D^{20} = -30.3$ (c 1.89, $CHCl_3$); ^{1}H NMR (200 MHz, $CDCl_3$), δ = 0.88 (d, 2H, J = 9.4 Hz), 0.99 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 1.61 (s, 3H, CH_3), 1.69 (s, 3H, CH_3), 1.73 (s, 3H, CH_3), 1.88–2.07 (m, 11H), 2.60–2.65 (m, 2H), 3.18 (d, 2H, CH_2, J = 8.2 Hz), 5.08–5.12 (m, 1H), 5.34 (t, 1H, J = 9.6 Hz) ppm; ^{13}C NMR (50.3 Hz, $CDCl_3$), δ = 17.7 (CH_3), 20.7 (CH_2), 23.1 (CH_2), 23.2 (CH_3), 23.4 (CH_3), 25.7 (CH_3), 26.2 (CH_2), 26.7 (CH_2), 28.1 (CH_3), 31.4 (CH_2), 31.8 (CH_2), 33.5 (CH_2), 38.7 (C), 41.4 (CH), 42.1 (CH), 46.5 (CH), 122.1 (CH), 124.0 (CH), 131.8 (C), 137.9 (C) ppm; ^{77}Se (38.1 MHz, $CDCl_3$), δ = 186.03 ppm. Elemental Anal. Calcd for $C_{20}H_{34}Se$ (353.44): C, 67.97; H, 9.70. Found: C, 68.11; H, 9.58.

4.8. General method for the synthesis of the diselenides

At first, NaOH (0.1 g) was added to the solution of selenol (9.0 mmol) in methanol (20 ml). The reaction mixture was then oxidized for 2 h with air. Methanol was evaporated off, water (20 ml) was added, and the product was extracted with petroleum ether (2 × 50 ml). The organic layers were dried over anhydrous $MgSO_4$, after which the ether was evaporated off, and the residue was purified by column chromatography, eluting with petroleum ether to give the pure diselenide.

4.9. General method for the oxidation of allylic selenides

At first, mCPBA (0.05 g, 0.29 mmol) in methylene chloride (4 ml) was carefully added to the selenide solution (0.28 mmol) in methylene chloride (3 ml) at −78 °C under an argon atmosphere. The solution was heated to −25 °C and stirred 15 min after which triethylamine (0.23 ml) was added and mixed for 15 min, then was heated to 25 °C and mixed for 30 min. The mixture was poured into water (10 ml), the organic layer was separated and washed twice with water (2 × 10 ml), dried over anhydrous $MgSO_4$, and the solvent was evaporated. The residue was purified by column chromatography, eluting with petroleum ether to give the pure alcohol.

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