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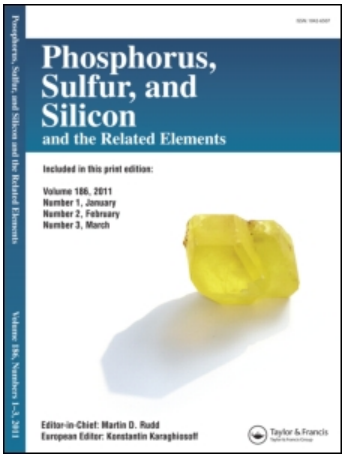


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# PREPARATION AND X-RAY CRYSTAL STRUCTURES OF TWO ALIPHATIC SELENENYL BROMIDES STABILIZED BY NSE COORDINATION

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# PREPARATION AND X-RAY CRYSTAL STRUCTURES OF TWO ALIPHATIC SELENENYL BROMIDES STABILIZED BY N–SE COORDINATION

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*(1*R*)-2-*endo-*(Dimethylamino)methyl-2-*exo-*methoxy-3-*endo-*camphorylselenenyl bromide and its 2-*endo-*(pyrrolidenyl)methyl analogue were prepared from (1*R*)-2-*endo-*acetamidomethyl-2-*exo-*hydroxy-3-*endo-*camphoryl diselenide. Both compounds showed an unusual lack of reactivity in electrophilic oxyselenenylation and cyclization reactions that are typical of other selenenyl bromides. X-ray crystallography indicated that both compounds have strong N—Se interactions, with N—Se interatomic distances of ca. 2.1 Å, which diminish the electrophilic character of the selenium atom.*

*Keywords:* Electrophiles; N—Se coordination; selenenyl bromides; X-ray structures

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## INTRODUCTION

Electrophilic selenium reagents of general structure RSeX (X = halide, triflate, sulfate, etc.) are routinely employed in a wide range of useful synthetic transformations.[1] Examples of such reactions include oxyselenenylations of alkenes, which take place in the presence of alcohols or water, and the cyclization of unsaturated carboxylic acids or alcohols (Scheme 1). The electrophiles are typically prepared or generated in situ from readily available diselenides (RSeSeR).[1] Asymmetric variations of these processes are possible when a chiral auxiliary is incorporated onto the selenium atom.[1,2] Improved diastereoselectivity often results

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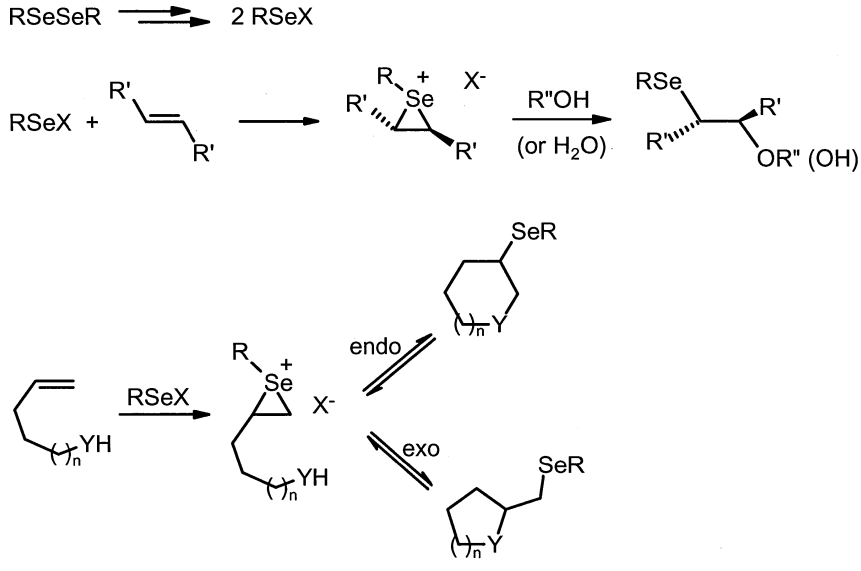


**SCHEME 1** Oxyselenenylation and cyclization with selenium electrophiles.

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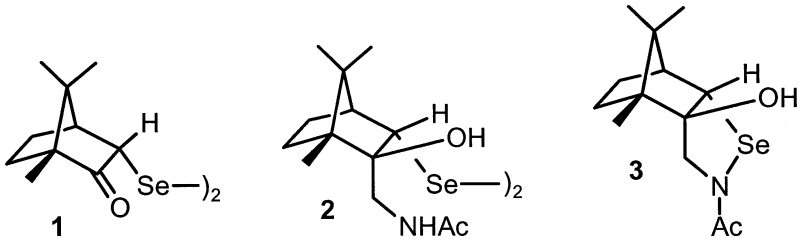
when the auxiliary group contains a pendant substituent, usually an amino group, that is capable of coordinating with the selenium atom during the course of the reaction.[3] Similar coordinating effects by nitrogen[4] and oxygen[5] substituents also play a key role in modulating the activity of small-molecule selenium compounds that act as mimetics of the selenoenzyme glutathione peroxidase (GPx).[6]

As part of our studies of asymmetric electrophilic selenium reactions, we have been investigating a series of camphor-based selenium electrophiles.[7] The diselenide **1** was obtained in a one-pot reaction from (*R*)-(+)-camphor and elemental selenium[8] and was easily converted into the corresponding selenenyl chloride, bromide, or triflate by treatment with sulfuryl chloride, bromine, or bromine followed by silver triflate.[7] We recently found that the diastereoselectivity of oxyselenenylation and cyclization reactions effected with various camphorseleno species was significantly improved when the 2-keto group of the camphor moiety in **1** was converted into its oxime prior to generation of the corresponding selenenyl halides or triflate.[9] Presumably, the oxime hydroxyl group is suitably positioned for intramolecular coordination with the electrophilic selenium center during its addition to olefinic substrates.

However, when an acetamido substituent was present at C-2 in **2**, attempts to prepare the corresponding selenenyl bromide by bromination of the parent diselenide resulted in spontaneous cyclization to the stable and isolable selenenamide derivative **3**.[10] We now report the preparation of two camphorseleno derivatives containing tertiary amino substituents at C-2, prompted by our desire to evaluate the effect of such amino groups upon the diastereoselectivity of asymmetric electrophilic selenium reactions (Scheme 2).




**SCHEME 2** Structures **1–3**.

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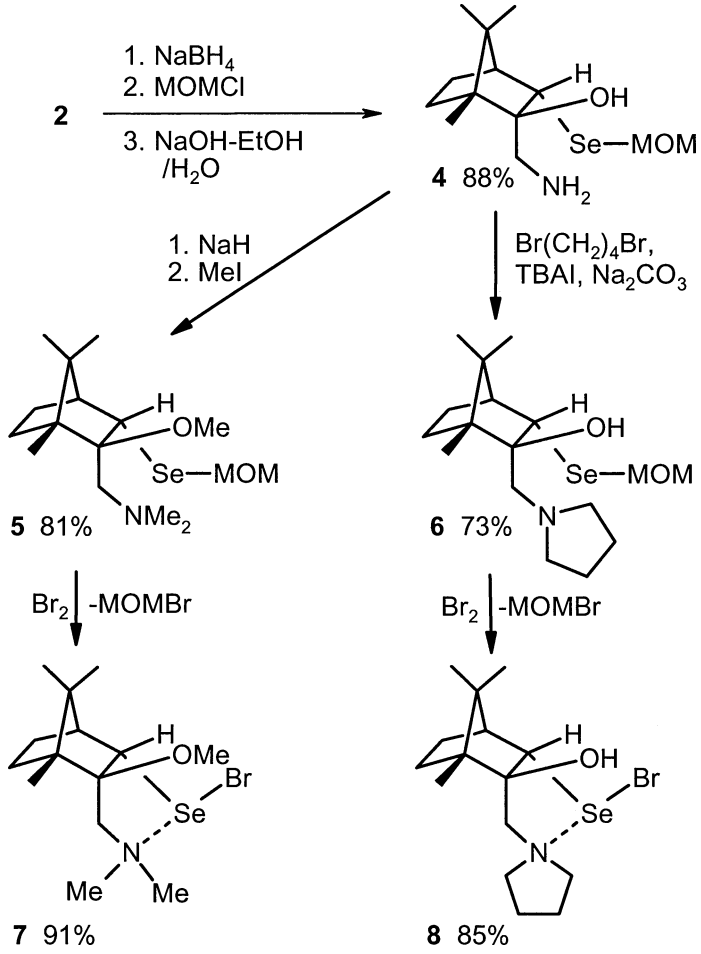
## RESULTS AND DISCUSSION

Diselenide **2** was reduced with $NaBH_4$, and the intermediate selenolate was protected as the methoxymethyl (MOM) selenide **4**. Amide hydrolysis followed by *O*- and *N*-methylation, or by *N*, *N*-dialkylation with 1,4-dibromobutane, afforded **5** and **6**, respectively. Brominolysis of **5** and **6** afforded **7** and **8**, respectively, along with MOM bromide (Scheme 3).[†] In contrast to typical selenenyl bromides, products **7** and **8** were pale yellow instead of deep red in color. These crystalline products were stable toward chromatography over silica gel, and they survived exposure to air during storage for at least several weeks. Moreover, they proved surprisingly inert as electrophiles in typical oxyselenenylation and cyclization reactions that were attempted with *trans*-5-decene and 4-penten-1-ol, respectively. Both alkenes were recovered intact after exposure to **7** or **8** under the usual[9] conditions.

The X-ray structure of **7** (see ORTEP diagram in Figure 1) shows N—Se and Se—Br interatomic distances of 2.157 Å and 2.618 Å, respectively. While these values are significantly longer than typical N—Se or Se—Br bond lengths of ca. 1.87 Å and 2.31 Å,[12a] they nevertheless fall well within the sum of the van der Waals radii of the respective atoms.

[†]The formation of selenenyl bromides by cleavage of MOM selenides with bromine was reported previously.[11]




**SCHEME 3** Preparation of selenenyl bromides **7** and **8**.

The N—Se—Br bond angle of 175.81° indicates a nearly linear geometry, while the respective C—Se—Br and C—Se—N bond angles of 88.63° and 87.32° show a roughly orthogonal orientation of the C—Se bond with respect to the N—Se—Br moiety (i.e., roughly T-shaped overall). Thus, the Se atom of **7** is the center of a slightly distorted trigonal bipyramid, wherein the nitrogen and bromine atoms occupy the apical sites, while the 3-camphoryl substituent and the two selenium lone pairs occupy equatorial positions.[12b,*] The X-ray data are therefore consistent with

*Relatively long Se-heteroatom bonds at apical sites in other hypervalent selenium compounds with trigonal bipyramidal structures have been observed previously.

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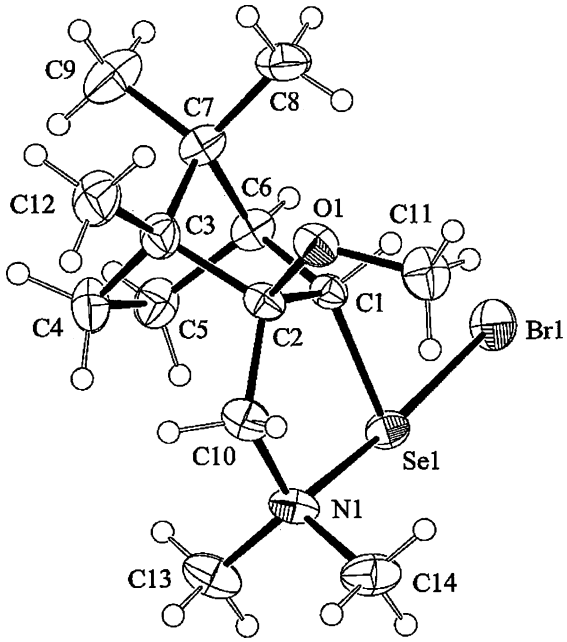


**FIGURE 1** ORTEP diagram of **7**.

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a structure in which there is considerable covalent character to both the N—Se and Se—Br bonds. The lack of electrophilic behavior of **7** and **8**, in contrast to that of other selenenyl bromides, reflects the decreased electrophilicity of the selenium atom caused by strong intramolecular coordination with the tertiary amino group. Thus, while weaker N—Se coordination can enhance stereoselectivity, as indicated earlier, N—Se coordination can be deleterious if it is strong enough to preclude the competing interaction of weaker nucleophiles, such as alkenes, with the selenium center.

Two slightly different conformations of **8** coexist in an asymmetric unit, (see ORTEP diagrams of conformations **8a** and **8b** in Figures 2 and 3). Structures **8a** and **8b** are similar to **7**. Thus, the N—Se and Se—Br bond lengths are 2.089 Å and 2.747 Å in **8a**, and 2.097 Å and 2.700 Å in **8b**, respectively. The N—Se—Br bond angles of 176.73° and 178.40° in **8a** and **8b**, respectively, indicate a nearly linear geometry, while the C—Se—Br and C—Se—N bond angles (85.92° and 90.95° in **8a**, and 85.8° and 93.01° in **8b**, respectively) again show a roughly T-shaped arrangement of the C—Se bond with respect to the N—Se—Br moiety.

A few aryl selenenyl halides containing amino substituents that are capable of coordinating with the selenium atom have been reported

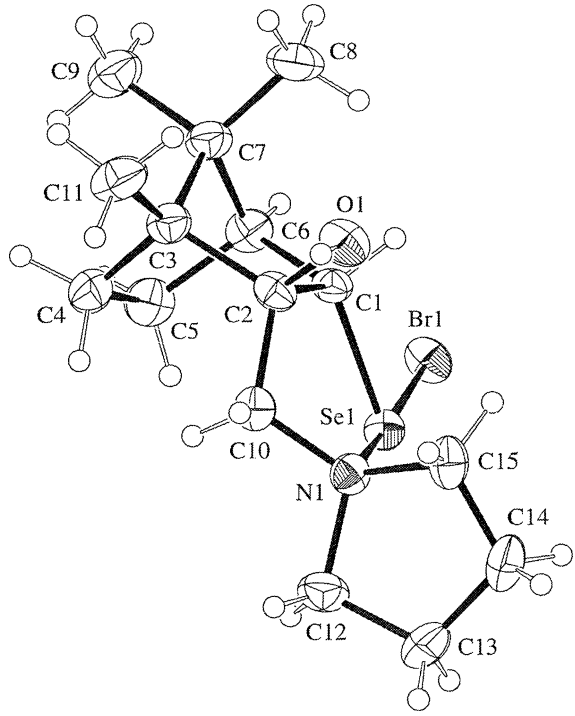


**FIGURE 2** ORTEP diagram of **8a**.

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previously.[13,14,**] The remarkably stable selenenyl bromides **7** and **8** represent new variations of this unusual class, in which an alkylseleno instead of an arylseleno moiety is present. The T-shaped structures of the present compounds closely resemble those of the earlier arylseleno analogues. It is also interesting to note that **7** and **8** resemble the putative transition state for an $S_N2$-like substitution of a selenenyl bromide by an amine.

## EXPERIMENTAL

NMR spectra were recorded in $CDCl_3$. Mass spectra were obtained by EI at 70 ev. Diselenide **2** was obtained as described previously.[7c]

**For a recent example of an arylselenenyl azide that is similarly stabilized by coordination of the selenium atom with an amino group, see Klapötke et al.[14]

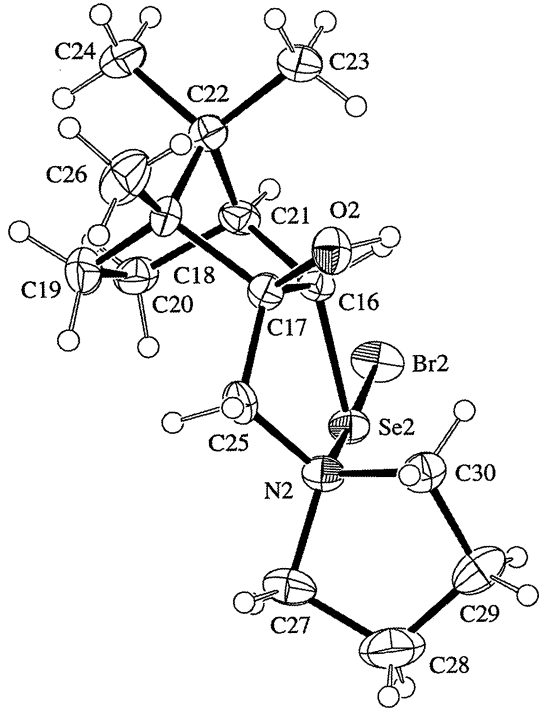


**FIGURE 3** ORTEP diagram of **8b**.

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## Methoxymethyl (1*R*)-2-*endo*-Aminomethyl-2-*exo*-hydroxy-3-*endo*-camphoryl Selenide (4)

Sodium borohydride (4.82 g, 128 mmol) was added to an ice-cooled solution of diselenide **2** (12.0 g, 19.7 mmol) in 250 ml of absolute ethanol, followed by warming to room temperature and stirring for an additional 15 min. Methoxymethyl chloride (11.9 ml, 157 mmol) was added dropwise, and stirring was continued for 45 min. The mixture was poured into ether, washed with saturated solutions of $NH_4Cl$ and NaCl, dried, and concentrated in vacuo. The residue was flash chromatographed over silica gel (elution with 35–50% ethyl acetate-hexanes) to afford 12.6 g (92%) of the corresponding MOM selenide as a pale yellow foam: IR (neat) 3293, 1657, 1080 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 6.68 (br s, 1 H), 5.05 (d, $J = 9.6$ Hz, 1 H), 4.98 (d, $J = 9.6$ Hz, 1 H), 3.89 (dd, $J = 14.2$, 8.5 Hz, 1 H), 3.60 (m, 1 H), 3.40 (s, 3 H), 3.20 (dd, $J = 14.2$, 4.3 Hz, 1 H), 2.04 (s, 3 H), 1.97–1.88 (m, 1 H), 1.77–1.25 (m, 5 H), 1.18 (s, 3 H), 0.97 (s, 3 H), 0.88 (s, 3 H). $^{13}C$ NMR ($CDCl_3$, 50 MHz)

$\delta$ 172.0, 80.8, 70.6, 56.6 (two signals), 52.4, 51.6, 47.8, 47.3, 29.1, 22.5, 22.4, 20.3 (two signals), 10.7. Mass spectrum, *m/z* (relative intensity) 331 (2, $M^+ - H_2O$), 304 (2), 206 (100), 109 (88). Exact mass calcd. for $C_{15}H_{27}NO_3Se$-$H_2O$: 331.1051. Found: 331.1035.

A mixture of 12.6 g (36.2 mmol) of the above product, 242 ml of ethanol, and 278 ml of 7.7 N NaOH was refluxed for 21 h. The mixture was extracted with ethyl acetate, and the solvent was removed in vacuo to afford 10.6 g (96%) of **4** as a yellow oil: IR (neat) 3284, 1077 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 5.00 (d, $J = 9.6$ Hz, 1 H), 4.92 (d, $J = 9.6$ Hz, 1 H), 3.66–3.56 (m, 1 H), 3.35 (s, 3 H), 2.98 (d, $J = 12.3$ Hz, 1 H), 2.54 (d, $J = 12.5$ Hz, 1 H), 1.90–1.77 (m, 1 H), 1.67–1.10 (m, 4 H), 1.22 (s, 3 H), 0.90 (s, 3 H), 0.82 (s, 3 H). $^{13}C$ NMR ($CDCl_3$, 50 MHz) $\delta$ 77.9, 71.1, 59.0, 56.7, 52.5 (two signals), 48.5, 45.4, 28.2, 22.7, 20.7, 20.5, 10.7. The crude product was employed in the next steps without further purification.

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## Methoxymethyl (1*R*)-2-*endo*-(Dimethylamino)methyl-2-*exo*-methoxy-3-*endo*-camphoryl Selenide (5)

Selenide **4** (5.50 g, 17.9 mmol) in 150 ml of dry tetrahydrofuran (THF) was treated with NaH (5.52 g, 60%, 137 mmol). The mixture was stirred at room temperature for 20 min, followed by the addition of methyl iodide (6.75 ml, 108 mmol) and stirring for a further 22 h. The mixture was quenched with saturated $NH_4Cl$ solution and was poured into diethyl ether. The ether layer was washed with saturated NaCl solution, dried, and concentrated in vacuo. The residue was flash chromatographed over silica gel (elution with 10% ethyl acetate-hexanes) to afford 5.09 g (81%) of **5** as a pale yellow oil: IR (neat) 1080 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 4.97 (d, $J = 9.9$ Hz, 1 H), 4.84 (d, $J = 9.7$ Hz, 1 H), 3.81 (d, $J = 4.1$ Hz, 1 H), 3.34 (s, 3 H), 3.26 (s, 3 H), 2.63 (d, $J = 14.4$ Hz, 1 H), 2.30 (s, 6 H), 2.09 (d, $J = 14.4$ Hz, 1 H), 1.87–1.79 (m, 1 H), 1.70–1.35 (m, 4 H), 1.11 (s, 3 H), 1.08 (s, 3 H), 0.92 (s, 3 H). $^{13}C$ NMR ($CDCl_3$, 50 MHz) $\delta$ 85.9, 70.2, 60.1, 56.5 (two signals), 54.4, 51.4, 50.9, 48.1, 46.9 (two signals), 30.4, 23.0, 21.2, 20.7, 11.9. Mass spectrum, *m/z* (relative intensity) 348 (1, $M^+$—H), 165 (51), 123 (75), 105 (48), 58 (100). Exact mass calcd. for $C_{16}H_{31}NO_2Se - CH_2OCH_3$: 304.1180. Found: 304.1200.

## Methoxymethyl (1*R*)-2-*endo*-(*N*-Pyrrolidinyl)methyl-2-*exo*-hydroxy-3-*endo*-camphoryl Selenide (6)

Selenide **4** (4.85 g, 15.8 mmol), sodium carbonate (5.13 g, 48.4 mmol), tetra-*n*-butylammonium iodide (2.98 g, 8.07 mmol) and

1,4-dibromobutane (2.2 mL, 18 mmol) were stirred in 250 ml of dry THF. The resulting suspension was refluxed for 22 h and then cooled to room temperature and poured into ether. The ether layer was washed with saturated NaCl solution, dried, and concentrated in vacuo. The residue was flash chromatographed over silica gel (elution with 10% ethyl acetate-hexanes) to afford 4.15 g (73%) of **6** as a pale yellow oil: IR (neat) 3463, 3180, 1085 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 5.35 (br s, 1 H), 4.97 (d, $J = 9.6$ Hz, 1 H), 4.86 (d, $J = 9.6$ Hz, 1 H), 3.82 (m, 1 H), 3.33 (s, 3 H), 2.89–2.61 (m, 6 H), 1.92–1.82 (m, 1 H), 1.80–1.67 (m, 4 H), 1.65–1.50 (m, 2 H), 1.33–1.25 (m, 2 H), 1.22 (s, 3 H), 0.92 (s, 3 H), 0.84 (s, 3 H). $^{13}C$ NMR ($CDCl_3$, 50 MHz) $\delta$ 77.7, 70.6, 59.7, 58.5, 56.4, 55.6 (two signals), 52.9, 51.7, 47.9, 28.0, 23.8 (two signals), 22.5, 20.9, 20.6, 10.6. Mass spectrum, *m/z* (relative intensity) 360 (14, $M^+$—H), 316 (28), 236 (31), 84 (100). Exact mass calcd. for $C_{17}H_{31}NO_2Se$ + H: 362.1598. Found: 362.1600.

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## (1*R*)-2-*endo*-(Dimethylamino)methyl-2-*exo*-methoxy-3-*endo*-camphorylselenenyl Bromide (7)***

A 1.0 M solution of bromine (0.89 ml, 0.89 mmol) in tetrachloromethane was added to a stirred solution of selenide **5** (311 mg, 0.894 mmol) in 15 ml of dichloromethane at room temperature. The resulting light yellow solution was stirred for 15 min, and the solvent was concentrated in vacuo. The residue was flash chromatographed over silica gel (elution with ethyl acetate) to afford 310 mg (91%) of **7** as a pale yellow oil. Crystallization (ethyl acetate-hexanes) afforded yellow crystals, m.p. 195°C (dec). IR (film) 1089, 1036, 839 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 5.23 (dd, $J = 4.1$, 2.2 Hz, 1 H), 3.38 (s, 3 H), 3.33 (s, 2 H), 3.04 (s, 3 H), 2.85 (s, 3 H), 2.54–2.45 (m, 1 H), 2.26–2.04 (m, 1 H), 1.91–1.71 (m, 1 H), 1.71–1.52 (m, 1 H), 1.43–1.27 (m, 1 H), 1.22 (s, 3 H), 1.02 (s, 3 H), 0.94 (s, 3 H). $^{13}C$ NMR (50 MHz, $CDCl_3$) $\delta$ 95.7, 65.4, 63.0, 55.6, 52.3, 52.2, 51.7, 50.1, 49.5, 32.4, 22.2, 21.6, 20.3, 13.4. Mass spectrum, *m/z* (relative intensity) 304 (4, $M^+$—Br), 208 (21), 164 (53), 138 (99), 123 (100). Exact mass calcd for $C_{14}H_{26}BrNOSe$-Br: 304.1180. Found: 304.1201. Anal. calcd. for $C_{14}H_{26}BrNOSe$: C, 43.88; H, 6.84; N, 3.65. Found: C, 43.78; H, 7.17; N, 3.61.

***Crystallographic data for structures **7** (CCDC-235047) and **8** (CCDC-235048) have been deposited with the Cambridge Crystallographic Data Centre, U.K. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

### (1*R*)-2-*endo*-(*N*-Pyrrolidinyl)methyl-2-*exo*-hydroxy-3-*endo*-camphorylselenenyl Bromide (8)***

A 1.0 M solution of bromine (0.71 ml, 0.71 mmol) in tetrachloromethane was added to a stirred solution of selenide **6** (255 mg, 0.708 mmol) in 15 ml of dichloromethane at room temperature. The resulting light yellow solution was stirred for 15 min and the solvent was concentrated in vacuo. The residue was flash chromatographed over silica gel (elution with ethyl acetate) to afford 238 mg (85%) of **8** as a pale yellow oil. Crystallization (ethyl acetate-hexanes) afforded yellow crystals, m.p. 190–193°C; IR (film) 3352 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 200 MHz) $\delta$ 5.21 (d, $J = 2.6$ Hz, 1 H), 4.14–3.97 (m, 1 H), 3.77–3.60 (m, 1 H), 3.57–3.38 (m, 1 H), 3.25–2.78 (m, 2 H), 2.62–2.51 (m, 1 H), 2.44–1.54 (m, 10 H), 1.25 (s, 3 H), 1.03 (s, 3 H), 0.96 (s, 3 H). $^{13}C$ NMR (50 MHz, $CDCl_3$) $\delta$ 89.6, 67.3, 64.8, 62.0, 61.4, 53.6, 50.5, 50.0, 32.3, 23.6, 22.5, 22.2, 21.4, 21.1, 12.1. Mass spectrum, $m/z$ (relative intensity) 396 (2, $M^+$), 218 (33), 160 (38), 107 (68), 84 (92), 81 (52), 80 (100). Exact mass calcd. for $C_{15}H_{26}BrNOSe$-Br: 316.1180. Found: 316.1180.

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