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Syntheses and Reactions of Cyclic Se-Alkoxy-Se-chloroselenuranes and Alkoxyselenonium Salts

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ABSTRACT: *Several 1-chloro-2,1-oxaselenole selenuranes* **3a–e** *and selenonium salts* **4a–c** *and 5-chloro-5,11-epoxy-6,11-dihydrodibenzo[b,e]selenepines* **12a** *and* **12b** *and selenonium salts* **13a–c** *were synthesized, and their reactions with organometallic reagents were studied.* © 2001 John Wiley & Sons, Inc. Heteroatom Chem 12:317–326, 2001

INTRODUCTION

The chemistry of hypervalent molecules has been intensively studied in recent years [1]. Hypervalent selenium compounds, σ-selenuranes with four carbon ligands, have been isolated by Furukawa and his coworkers [2], who also investigated the intermediates of the reactions of selenonium salts with aryllithiums using ^{1}H, ^{13}C and ^{77}Se NMR spectroscopy and demonstrated that the intermediates were σ-selenuranes [3]. σ-Selenuranes with electronegative heteroatoms on the selenium atom can be isolated as stable crystals [4]. We previously reported that the thermal reaction of a 1-chloro-1-phenyl-3*H*-2,1-benzoxaselenole afforded an aldehyde or a ketone by oxidation of the alkoxy moiety [5]. Kawashima and his coworkers reported that the thermolysis of tetracoordinate 1,2-oxaselenetanes containing a selenaspiro[4.5]ring system stereospecifically gave oxiranes and oxaselenoles [6]. However, the chemistry of alkoxyselenonium salts has been hardly studied. This article describes the syntheses and reactions of cyclic alkoxyselenuranes and alkoxyselenonium salts.

The 3*H*-2,1-benzoxaselenole selenuranes and selenonium salts were synthesized as shown in Scheme 1. *o*-(Phenylseleno)benzaldehyde (**1**) was oxidized with silver oxide to give the benzoic acid **5**. Esterification of **5**, followed by the Grignard reaction, afforded the alcohol **7**. Alcohol **7** was chlorinated with *N*-chlorosuccinimide (NCS) to give 1-chloro-3*H*-2,1-oxaselenole **3c**. The selenurane **3c** was converted into oxaselenolium salt **4c**. Other alkoxyselenuranes **3a,b** and selenonium salts **4a,b** were prepared by the known procedures [5]. Acyloxyselenurane **3d** was derived from carboxylic acid **5** by chlorination with NCS. Se-Methyl derivative **3e** was similarly prepared from *o*-(methylseleno)benzyl alcohol **9**, which was obtained by the reduction of 2,2′-dicarboxydiphenyl diselenide **8** with $LiAlH_4$ followed by alkylation with methyl iodide. The selenurane structures of **3a–d** were characterized by the ^{1}H and ^{77}Se NMR spectra. Compounds **3a–e** showed downfield-shifted signals at δ 8.72–9.04 due to H(7), whereas the corresponding protons of selenonium salts **4a–c** appeared at δ 8.11–8.25. The C(7)-H bond

Dedicated to Prof. Naoki Inamoto on the occasion of his 72nd birthday.

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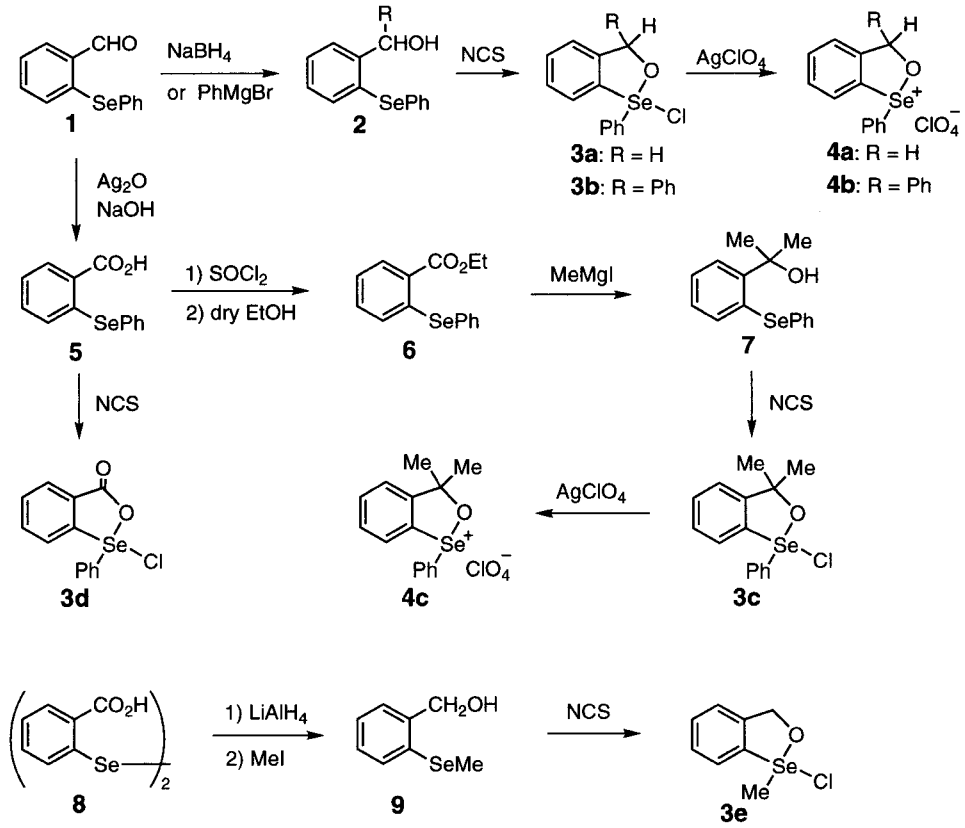


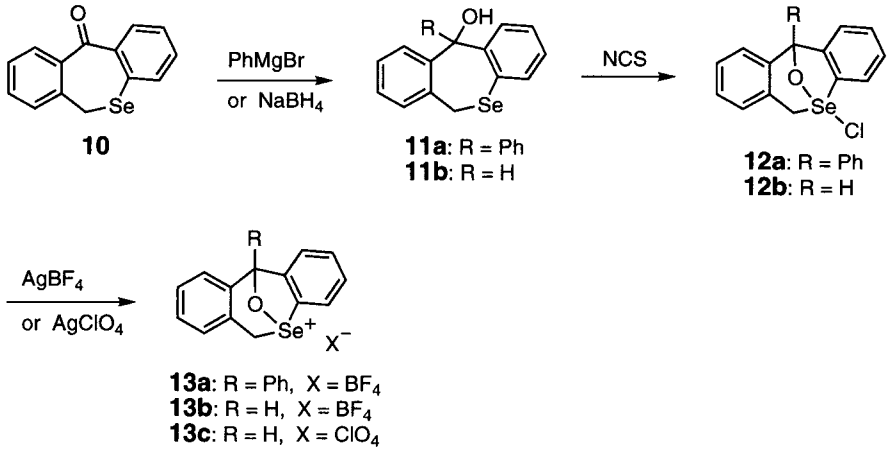
SCHEME 1

of the selenuranes is parallel to the apical Se–Cl bond and the H(7) is affected by the anisotropic effect of the chlorine atom. The ^{77}Se-NMR spectra of selenuranes **3a–c** exhibited the signals at δ 797.2–815.7 in contrast to those of selenonium salts **4a–c**, which exhibited them at δ 983.3–1009.2. These spectroscopic differences between selenuranes and selenonium salts are in good agreement with other examples [4,6,7].

11-Chloro-5,11-epoxy-6,11-dihydrodibenzo[*b,e*] selenepines (**12**) and selenepinium salts (**13**) were prepared by the procedures shown in Scheme 2. Alcohols **11a,b** derived from **10** by reduction or the Grignard reaction were chlorinated with NCS to give chloroselenuranes **12a,b**. The selenuranes **12a,b** were converted into selenonium salts **13a–c** by the treatment with $AgBF_4$ or $AgClO_4$. Selenuranes **12a,b** showed the characteristic downfield shift of H(4) at δ 8.68 and 8.55, respectively in their ^{1}H NMR spectra. The ^{77}Se NMR signals of **12a,b** appeared at δ 719.7 and 726.7 in the higher field than those of the selenonium salts of **13a,b** at δ 851.5 and 858.5, respectively.

We conducted reactions of oxaselenolium salts **4a,b** with organometallic reagents (Scheme 3 and Table 1). Reactions at room temperature gave only benzyl alcohols **2a,b**, and reactions under reflux in THF afforded benzophenone derivatives **14a,b** and benzyl ethers **15a,b** in addition to **2a,b**. The benzyl alcohols **2a,b** were presumably formed via the single-electron transfer (SET) from the organometallic reagent to the selenonium salt **4a,b** [8] followed by O–Se bond fission. The benzophenone derivatives **14a,b** are formed by deprotonation of H(3) and subsequent O–Se bond fission. This process corresponds to the last step of the dimethyl sulfoxide oxidation of alcohols [9] and is confirmed by the evidence that the reaction of selenonium salt **4b** with sodium hydride gave benzophenone **14b** in 32.5% yield. Benzyl ethers **15a,b** were presumably formed via the ligand coupling reactions of the respective selenurane intermediates. 3,3-Dimethyl derivative **4c** was used to prevent oxidation triggered by deprotonation of H(3). Reactions with phenyllithium or methyllithium at room temperature gave the SET reduction product **7** only, and the reaction with methyllithium at -10°C gave a ligand coupling product **15c** in 8% yield.

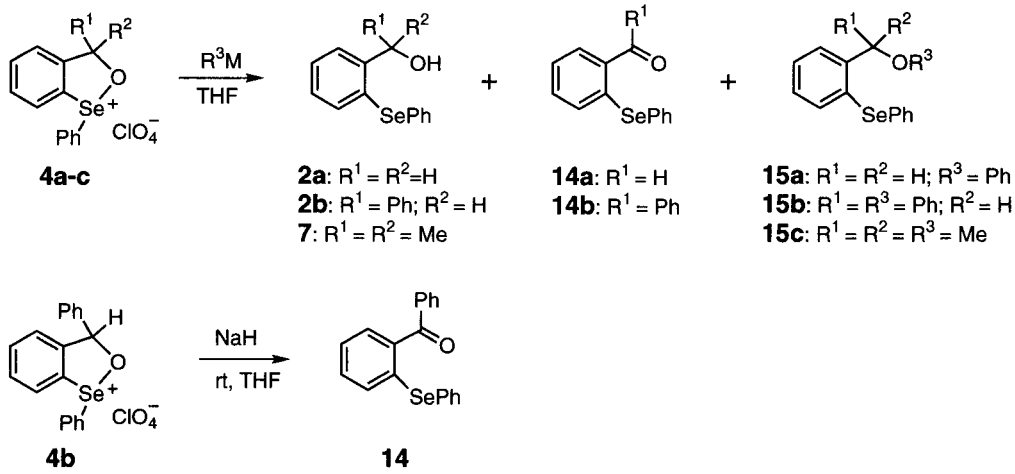
From these results it was found that 2,1-oxaselenolium salts **4a–c** readily underwent the SET reduction by the organometallic reagents. Therefore, we next examined the reactions of 5,11-epoxy-6,11-



10 **11a**: R = Ph; **11b**: R = H; **12a**: R = Ph; **12b**: R = H

13a: R = Ph, X = BF_4
13b: R = H, X = BF_4
13c: R = H, X = ClO_4

SCHEME 2



4a-c **2a**: $R^1 = R^2$=H; **2b**: R^1 = Ph; R^2 = H; **7**: $R^1 = R^2$ = Me; **14a**: R^1 = H; **14b**: R^1 = Ph; **15a**: $R^1 = R^2$ = H; R^3 = Ph; **15b**: $R^1 = R^3$ = Ph; R^2 = H; **15c**: $R^1 = R^2 = R^3$ = Me

4b **14**

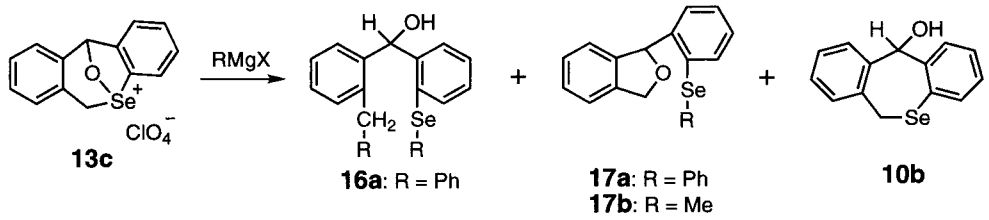
SCHEME 3

TABLE 1 Reactions of Selenonium Salts **4** with Organometallic Reagents

					Products (% Yield)		
Entry	*Compound No.*	*R^3M*	*Temp.*	*Time*	**2**	**14**	**15**
1	**4a**	PhLi	Reflux	5 h	**2a** (41.8)	14a (3.1)	**15a** (13.0)
2	**4a**	PhMgBr	Reflux	5 h	**2a** (83.3)	—	—
3	**4b**	PhLi	Reflux	5 h	**2b** (22.4)	**14b** (7.1)	**15b** (7.2)
4	**4b**	PhMgBr	Reflux	5 h	**2b** (37.7)	**14b** (5.4)	**15b** t[a]

[a]*t* = trace (<1% yield).

dihydrodibenzo[*b,e*]selenepinium salts **13** with Grignard reagents (Scheme 4 and Table 2). Reactions of **13c** with organolithiums gave complex mixtures of products. Reactions with phenylmagnesium bromide afforded a ring-opened product **16a**, benzo[*c*]furan derivative **17a**, and dihydrodibenzoselenepinol **10b**. Reaction at room temperature (entry 1) gave **17a** in greater yield than that at lower temperature (entry 2). When phenylmagnesium iodide was used instead of the bromide, a SET reduction product **10b** was obtained in 56% yield (entry 3). Methylmagnesium iodide similarly caused the SET reduction to give **10b** in 70.9% yield (entry 4). This interesting difference between RMgI and RMgBr has been reported in the reactions of cyclic selenonium salts with organometallic reagents [8].



SCHEME 4

TABLE 2 Reactions of Selenonium Salt **13c** with Grignard Reagents

Entry	RMgX	Molar Ratio RMgX/**13c**	Products (% Yield) **16**	**17**	**10b**
1	PhMgBr	8	**16a** (45.6)	**17a** (31.6)	—
2	PhMgBr[a]	8	**16a** (38.4)	**17a** (1.7)	—
3	PhMgI	4	**16a** (14.7)	**17a** (24.8)	56.0
4	MeMgI	1.1	—	**17b** (3.1)	70.9

[a]Temp. $-78°C \rightarrow$ r.t.

The probable mechanism of formation of products **16**,**17**, and **10b** is shown in Scheme 5. The Grignard reagents attack a selenium atom and form selenurane **18**, whose oxygen ligand and the benzylic carbon couple with each other to give dihydrobenzo[*c*]furan **17**. If the nucleophilic attack of the Grignard reagents at the selenium atom causes the ligand exchange, selenonium salt **19** is formed. The selenonium salt **19** reacts with another molecule of RMgX to generate a selenurane-bearing four carbon-ligands, **20**, whose benzyl carbon combines with one of the R ligands to give product **16**. Products **16** and **17** might be formed by another pathway, that is, the attack of RMgX and the alkoxide moiety, respectively, on the benzylic carbon. However, we could not determine how much the nucleophilic attack on the benzylic carbon contributes to the reaction mechanism. The SET from RMgX to selenonium ion **13c** brings about the O–Se bond cleavage to generate an alkoxy radical, which abstracts a hydrogen from a solvent or is reduced to an alkoxide ion and then gives **10b**.

Finally, we carried out the reaction of selenurane **12b** and selenonium salt **13b** with triethylamine (Scheme 6). Since these compounds have no β-hydrogen to the selenonio moiety, their reactions with a base did not cause β-elimination but rather the 1,2-rearrangement of the alkoxy group. Both compounds **12b** and **13b** afforded 5,10-epoxydihydrodibenzoselenepine **18** in 18% and 68% yields, respectively. The reason why **13b** gave a higher yield than **12b** might be that the selenium atom of **13b** is more positively charged than that of **12b**.

EXPERIMENTAL

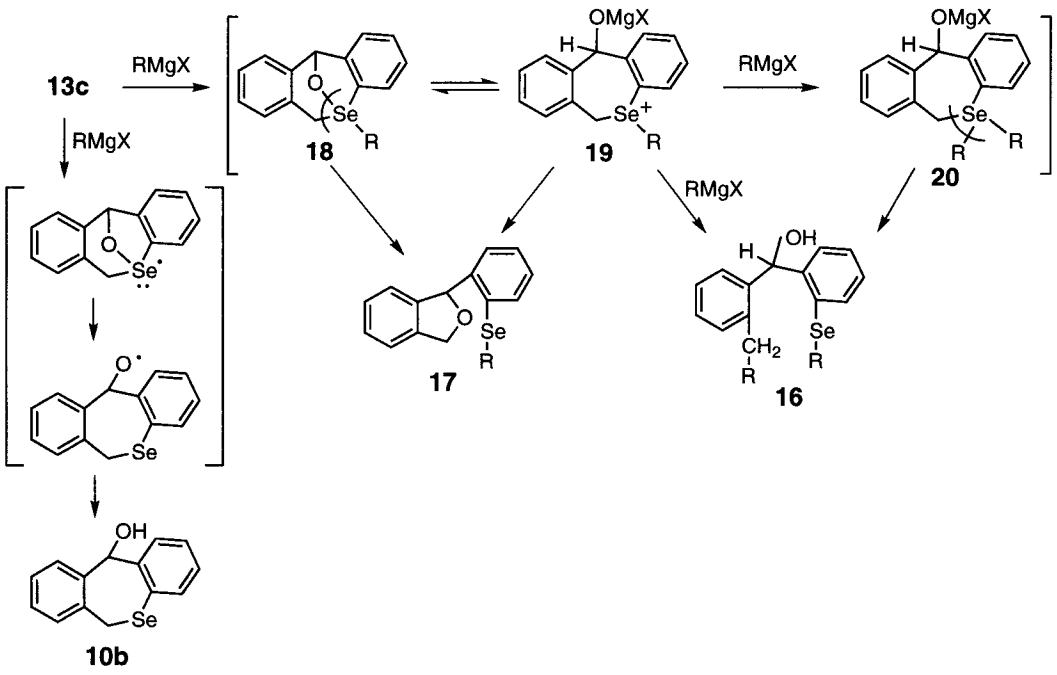
Melting points were obtained with a Yanagimoto micro melting point apparatus and are uncorrected. The IR spectra of solids (KBr) and liquids (NaCl) were recorded on a JASCO IRA-100 spectrophotometer. ^{1}H NMR spectra were recorded on a JEOL GX-270 (270 MHz) or a JEOL EX-400 (400 MHz) spectrometer with tetramethylsilane as an internal standard. ^{13}C NMR and ^{77}Se NMR spectra were obtained on a JEOL EX-400 spectrometer. The chemical shifts of ^{77}Se signals were measured in ppm on the δ-scale downfield from dimethyl selenide as an external standard. Mass spectra were recorded on a JEOL JMS-SX102A spectrometer with a direct-insertion probe at 70 eV. Elemental analyses of new compounds were performed by use of a Yanaco CHN Corder MT-5. All chromatographic isolations were accomplished with BW-350 (Fuji Silysia) for column chromatography or with Kieselgel 60 PF_{254} containing gypsum (Merck) for preparative thin-layer chromatography (TLC). CH_2Cl_2 was dried and freshly distilled over calcium chloride.

*1-Chloro-1-phenyl-3H-2,1-benzoxaselenole (***3a***)*

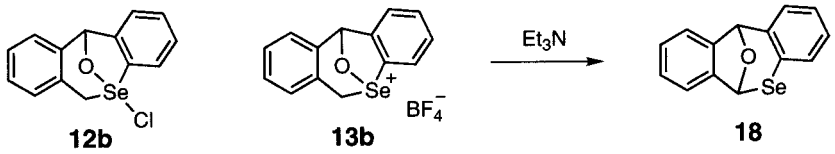
Compound **3a** was prepared by the known procedure [5]. ^{1}H NMR ($CDCl_3$ δ) 4.97 and 5.39 (each 1H, d, J = 14 Hz, 3-H), 7.37–7.48 (4H, m, ArH), 7.50–7.57 (2H, m, ArH), 7.62–7.72 (2H, m, ArH), 8.96 (1H, d, J = 9 Hz, 7-H). ^{13}C NMR ($CDCl_3$ δ) 74.2 (t), 123.1 (d), 127.7 (d × 2), 129.3 (d), 129.5 (d × 2), 131.2 (d), 131.4 (d), 132.5 (d), 133.4 (s), 141.5 (s), 143.8 (s). ^{77}Se-NMR ($CDCl_3$ δ) 814.9.

*1-Chloro-1,3-diphenyl-3H-2,1-benzoxaselenole (***3b***)*

Compound **3b** was prepared by the known procedure [5]. ^{1}H NMR ($CDCl_3$ δ) 5.81 (1H, s, 3-H), 6.96 (1H, d, J = 7 Hz, ArH), 7.17–7.31 (2H, m, ArH), 7.35–7.75 (10H, m, ArH), 9.04 (1H, d, J = 8 Hz, 7-H). ^{13}C



SCHEME 5



SCHEME 6

NMR ($CDCl_3$ δ) 87.3 (d), 126.1 (d), 128.3 (d × 2), 128.4 (d × 2), 129.3 (d × 2), 129.5 (d), 130.0 (d × 2), 130.2 (d), 131.4 (d), 131.6 (d), 133.0 (d), 134.1 (s), 139.7 (s), 141.8 (s), 146.7 (s). ^{77}Se-NMR ($CDCl_3$ δ) 797.2.

*1-Phenyl-3H-2,1-benzoxaselenolium Perchlorate (***4a***)*

This compound was prepared by the known procedure [5]. ^{1}H NMR (CD_3CN δ) 5.62 and 5.76 (each 1H, d, J = 14 Hz, 3-H), 7.53 (2H, d, J = 8 Hz, ArH), 7.61 (2H, t, J = 8 Hz, ArH), 7.68–7.83 (3H, m, ArH), 7.88 (1H, t, J = 7 Hz, ArH), 8.12 (1H, d, J = 8 Hz, 7-H). ^{13}C{^{1}H}-NMR (CD_3CN δ) 79.2, 116.9, 123.7, 128.2, 129.2, 130.3, 130.7, 133.5, 134.1, 143.3. ^{77}Se NMR (CD_3CN δ) 1009.2.

*1,3-Diphenyl-3H-2,1-benzoxaselenolium Perchlorate (***4b***)*

Silver perchlorate (6.0 g, 29 mmol) was added to a solution of **3b** (9.8 g, 26 mmol) in dry CH_2Cl_2 (380 mL), and the mixture was stirred for 12 hours at room temperature. The precipitate was collected by filtration and washed with hot acetonitrile several times. The filtrate and the washings were combined and concentrated. The residue was recrystallized from acetonitrile-ether to give colorless prisms (11.2 g, 97.5%), m.p. 205–208°C (dec.). ^{1}H NMR (CD_3CN δ) 6.57 (1H, s, 3-H), 7.16 (1H, d, J = 7 Hz, ArH), 7.2–7.38 (2H, m, ArH), 7.38–7.52 (3H, m, ArH), 7.52–7.70 (4H, m, ArH), 7.70–7.87 (3H, m, ArH), 8.25 (1H, d, J = 7 Hz, ArH). ^{13}C{^{1}H} NMR (CD_3CN δ) 91.9, 126.4, 128.2, 128.5, 128.8, 129.3, 130.0, 130.4, 131.3, 133.7, 134.0, 134.1, 145.2. ^{77}Se NMR ($CDCl_3$ δ) 983.3. IR (KBr) cm^{-1}: 1200–1000 (ClO_4^-). Anal. Calcd. for $C_{19}H_{15}ClO_5Se$: C, 52.13; H, 3.45. Found: C, 51.88; H, 3.44.

*o-(Phenylseleno)benzoic Acid (***5***)*

Silver nitrate (14.4 g) was added to a solution of NaOH (6.4 g) in 50% ethanol (240 mL). *o*-(Phenylseleno)benzaldehyde [10] (**1**) (10.5 g, 40 mmol) was added to the suspension, and the mixture was re-

fluxed for 4 hours. Hot water (480 mL) was added to the mixture, and the whole was filtered through celite. The filtrate was acidified with 2 N HCl and extracted with CH_2Cl_2. The extracts were washed with water, dried ($MgSO_4$), and concentrated to dryness. The residual solid was recrystallized from ethanol to give colorless needles (10.3 g, 93%), m.p. 185–186°C (reported [11] 190°C). ^{1}H NMR (CD_2Cl_2 δ) 5.32 (1H, s, OH), 6.94 (2H, d, J = 8 Hz, ArH), 7.20–7.33 (4H, m, ArH), 7.35–7.53 (6H, m, ArH), 7.65–7.75 (4H, m, ArH), 8.15 (2H, d, J = 7 Hz, ArH). ^{13}C NMR (CD_2Cl_2 δ) 125.3 (d), 128.9 (s), 129.5 (d), 129.7 (d), 130.2 (d × 2), 132.6 (d), 133.8 (d), 137.9 (d × 2), 141.2 (s), 141.9 (s), 171.8 (s). IR (KBr) cm^{-1}: 3300–2400 (OH), 1670 (C=O). MS *m/z*: 278 (M^+, base). Anal. Calcd. for $C_{13}H_{10}O_2Se$: C, 56.30; H, 3.64. Found: C, 56.20; H, 3.64.

*Ethyl o-(Phenylseleno)benzoate (***6***)*

Carboxyclic acid **5** was added to thionyl chloride (1.3 g, 11 mmol), and the mixture was heated at 100°C for 1 hour. Hydrogen chloride was removed from the mixture under diminished pressure, and then a solution of dry pyridine (4 mL) in dry ethanol (30 mL) was gradually added to it. The reaction mixture was stirred for 1 hour at room temperature and then diluted with ether (30 mL). The whole was washed with 10% HCl, 10% NaOH, and water, successively, dried ($MgSO_4$), and concentrated to give a yellow oil (2.2 g, quantitatively). ^{1}H NMR ($CDCl_3$ δ) 1.44 (3H, t, J = 7 Hz, CH_3), 4.44 (2H, q, J = 7 Hz, CH_2), 6.90 (1H, d, J = 8 Hz, ArH), 7.10–7.25 (2H, m, ArH), 7.37–7.53 (3H, m, ArH), 7.71 (2H, dd, J = 8 and 2 Hz, ArH), 8.06 (2H, dd, J = 7 and 2 Hz, ArH). ^{13}C NMR ($CDCl_3$ δ) 14.4 (q), 61.4 (t), 116.7 (s), 124.7 (d), 127.4 (s), 128.9 (d), 129.1 (d), 129.7 (d × 2), 131.3 (d), 132.5 (d), 137.5 (d × 2), 140.3 (s), 166.8 (s). IR (film) cm^{-1}: 1700 (C=O). HRMS Calcd for $C_{15}H_{14}O_2Se$: 306.0158. Found: 306.0133.

*1-Methyl-1-[(2-phenylseleno)phenyl]ethanol (***7***)*

An ethereal solution of methylmagnesium bromide (22.6 mL, 21.7 mmol) was added to a solution of **6** (2.2 g, 7.2 mmol) in dry THF (20 mL) under a nitrogen atmosphere. The mixture was stirred for 2 hours at room temperature, cooled in an ice bath, and then treated with a saturated NH_4Cl solution. The aqueous mixture was extracted with ether. The extracts were dried ($MgSO_4$) and concentrated under reduced pressure to give a pale yellow oil (1.90 g, 90%). ^{1}H NMR ($CDCl_3$ δ) 1.74 (6H, s, CH_3), 2.91 (1H, s, OH), 6.99 (1H, td, J = 7 and 1.5 Hz, ArH), 7.08–7.23 (2H, m, ArH), 7.25–7.33 (3H, m, ArH), 7.38 (1H, dd, J = 8 and 2 Hz, ArH), 7.48 (2H, m, ArH). ^{13}C NMR ($CDCl_3$ δ) 30.4 (q × 2), 74.4 (s), 125.7 (d), 126.4. (d), 127.6 (d), 127.8 (d), 129.4 (d × 2), 131.0 (s), 131.9 (s), 133.9 (d), 134.7 (d × 2), 147.2 (s). IR (film) cm^{-1}: 3370 (OH). HRMS Calcd for $C_{15}H_{16}OSe$: 292.0365. Found: 292.0343.

*1-Chloro-3,3-dimethyl-1-phenyl-3H-2,1-benzoxaselenole (***3c***)*

A solution of *N*-chlorosuccinimide (NCS) (850 mg, 6.5 mmol) in dry CH_2Cl_2 (50 mL) was added to a solution of **7** (1.9 g, 6.5 mmol) in dry CH_2Cl_2 (20 mL). The mixture was stirred for 30 minutes at room temperature, concentrated to the amount of 20 mL, and ether was added. The precipitate was collected by filtration and recrystallized from CH_2Cl_2-ether to give colorless prisms (1.10 g, 52%), m.p. 138–140°C (dec.). ^{1}H NMR ($CDCl_3$ δ) 1.07 (3H, s, CH_3), 1.59 (3H, s, CH_3), 7.28 (1H, dd, J = 7 and 2 Hz, ArH), 7.35–7.47 (3H, m, ArH), 7.55 (2H, dd, J = 8 and 1.5 Hz, ArH), 7.65–7.78 (2H, m, ArH), 9.03 (1H, dd, J = 8 and 1.5 Hz, 7-H). ^{13}C NMR ($CDCl_3$ δ) 29.8 (q), 31.6 (q), 90.1 (s), 124.2. (d), 127.6 (d × 2), 129.4 (d × 2), 129.8 (d), 131.1 (d), 131.2 (s), 131.4 (d), 133.2 (d), 145.9 (s), 150.9 (s). ^{77}Se NMR ($CDCl_3$ δ) 815.7. Anal. Calcd. for $C_{15}H_{15}ClOSe$: C, 55.32; H, 4.64. Found: C, 55.10; H, 4.59.

*3,3-Dimethyl-1-phenyl-3H-2,1-benzoxaselenolium Perchlorate (***4c***)*

Silver perchlorate (0.78 g, 3.8 mmol) was added to a solution of **3c** (1.1 g, 3.4 mmol) in dry CH_2Cl_2 (50 mL) at room temperature. The mixture was stirred for 2 hours, and silver chloride was removed by filtration. The filtrate was concentrated under reduced pressure, and the residue was recrystallized from ethyl acetate to give colorless prisms (0.95 g, 72%), m.p. 193–195°C (dec.). ^{1}H NMR (CD_3CN δ) 1.42 (3H, s, CH_3), 1.74 (3H, s, CH_3), 7.53–7.67 (5H, m, ArH), 7.67–7.70 (1H, m, ArH), 7.83 (1H, td, J = 7 and 1.5 Hz, ArH), 7.92 (1H, td, J = 7 and 1.5 Hz, ArH), 8.11 (1H, dd, J = 8 and 1 Hz, ArH). ^{13}C NMR (CD_3CN δ) 30.6 (q), 30.9 (q), 100.2 (s), 126.1 (d), 129.4 (d), 130.1 (s), 130.3 (d × 2), 131.6 (d × 2), 132.5 (d), 135.3 (d), 135.4 (d), 138.8 (s), 151.0 (s). ^{77}Se NMR ($CDCl_3$ δ) 987.1. IR (KBr) cm^{-1}: 900–1200 (ClO_4^-). Anal. Calcd. for $C_{15}H_{15}ClO_5Se$: C, 46.23; H, 3.88. Found: C, 46.23; H, 3.84.

*1-Chloro-3-oxo-1-phenyl-2,1-oxaselenole (***3d***)*

A solution of NCS (940 mg, 7.2 mmol) in dry CH_2Cl_2 (100 mL) was added to a solution of **5** (1.9 g, 6.5

mmol) in dry CH_2Cl_2 (50 mL). The mixture was stirred for 30 minutes at room temperature, concentrated to 50 mL, and ether was added. The precipitate was collected by filtration and recrystallized from CH_2Cl_2-ether to give colorless prisms (0.70 g, 31%), m.p. 197–200°C (dec.). ^{1}H NMR (DMSO-d_6 δ) 7.43–7.65 (5H, m, ArH), 8.04 (1H, t, J = 7 Hz, ArH), 8.07–8.20 (2H, m, ArH), 8.72 (1H, d, J = 8 Hz, 7-H). ^{13}C NMR (DMSO-d_6 δ) 127.3. (d × 2), 129.4 (d), 129.8 (d), 130.0 (d × 2), 131.4 (s), 131.5 (d), 133.7 (d), 135.2 (d), 136.6 (s), 144.5 (s), 168.1 (s). Anal. Calcd. for $C_{13}H_9ClO_2Se$: C, 50.11; H, 2.91. Found: C, 49.89; H, 3.01.

*o-(Methylseleno)benzyl Alcohol (***9***)*

Diselenide **8** [12] (4.0 g, 10 mmol) was gradually added to a suspension of $LiAlH_4$ (0.95 g, 50 mmol) in dry THF (50 mL) with cooling in an ice bath, and the mixture was stirred for several hours at room temperature. Then a solution of methyl iodide (2.8 g, 20 mmol) in dry THF (10 mL) was added to the mixture. The whole was stirred overnight, and water was added to it. The precipitate was filtered off and washed with CH_2Cl_2. The filtrate was extracted with CH_2Cl_2. The washings and the extracts were combined, dried ($MgSO_4$), and concentrated. The residual oil was purified by column chromatography on silica gel (haxane–CH_2Cl_2 = 2:1) to give a pale yellow oil (2.4 g, 58%). ^{1}H NMR ($CDCl_3$ δ) 2.30 (3H, s, CH_3), 2.61 (1H, s, OH), 4.70 (2H, s, CH_2), 7.15–7.28 (2H, m, ArH), 7.30–7.38 (1H, m, ArH), 7.38–7.45 (1H, m, ArH). ^{13}C NMR ($CDCl_3$ δ) 7.30 (q), 64.9 (t), 126.5 (d), 128.0 (d), 128.3 (d), 130.6 (d), 131.4 (s), 140.9 (s). IR (film) cm^{-1}: 3350 (OH). High-resolution mass spectrometry (HRMS) Calcd for $C_8H_{10}OSe$: 201.9896. Found: 201.9871.

*1-Chloro-1-methyl-3H-2,1-benzoxaselenole (***3e***)*

A solution of NCS (1.5 g, 12 mmol) in dry CH_2Cl_2 (60 mL) was added to a solution of **9** (2.4 g, 12 mmol) in dry CH_2Cl_2 (30 mL). The mixture was stirred for 30 minutes at room temperature and then poured into water. The CH_2Cl_2 layer was separated, and the aqueous layer was extracted with CH_2Cl_2. The CH_2Cl_2 layer and the extracts were combined, dried, and concentrated to the amount of 20 mL, and then ether was added. The precipitate was collected by filtration and recrystallized from CH_2Cl_2-ether to give colorless prisms (2.17 g, 77%), m.p. 114–117°C (dec.). ^{1}H NMR ($CDCl_3$ δ) 3.39 (3H, s, CH_3), 5.48 and 5.57 (each 1H, d, J = 14 Hz, CH_2), 7.41 (1H, d, J = 7 Hz, ArH), 7.52–7.62 (1H, m, ArH), 7.62–7.70 (1H, m, ArH), 8.76 (1H, d, J = 8 Hz, 7-H). ^{13}C NMR ($CDCl_3$ δ) 42.3 (q), 74.9 (t), 122.9 (d), 129.4 (d), 130.4 (d), 133.2 (d), 134.4 (s), 142.5 (s). Anal. Calcd. for C_8H_9ClOSe: C, 40.79; H, 3.85. Found: C, 40.73; H, 3.75.

*11-Phenyl-6,11-dihydrodibenzo[b,e]selenepin-11-ol (***11a***)*

A solution of 6,11-dihydrodibenzo[*b,e*]selenepin-11-one (**10**) [13] (4.0 g, 15 mmol) in dry benzene (40 mL) was added to an ethereal solution of phenylmagnesium bromide prepared from bromobenzene (5.7 g, 36.3 mmol) and magnesium (1.1 g, 44 mmol). The mixture was refluxed for 4.5 hours with stirring, cooled in an ice bath and treated with a saturated NH_4Cl solution. The ether layer was separated and the aqueous layer was extracted with CH_2Cl_2. The ether layer and the extracts were combined, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane–CH_2Cl_2 = 3:1) to give colorless prisms (from CH_2Cl_2-ether) (3.8 g, 74%), m.p. 210–212°C. ^{1}H NMR ($CDCl_3$ δ) 3.24 and 3.65 (each 1H, d, J = 12 Hz, 6-H), 7.08 (1H, d, J = 7 Hz, ArH), 7.13 (1H, dd, J = 1.4, 8 Hz, ArH), 7.20–7.36 (9H, m, ArH), 8.06 (1H, d, J = 8 Hz, ArH), 8.11 (1H, d, J = 8 Hz, ArH). ^{13}C NMR ($CDCl_3$ δ) 27.3 (t), 79.1 (s), 125.7 (d), 127.9 (d), 126.7 (d), 127.1 (d × 2), 127.6 (d), 127.9 (d), 128.3 (d), 128.4 (d), 128.9 (d × 2), 129.4 (d), 129.8 (s), 130.9 (d), 132.5 (s), 140.7 (s), 144.2 (s × 2). IR (KBr) cm^{-1}: 3140 (OH). MS *m/z*: 352 (M^+), 195 (base). Anal. Calcd. for $C_{20}H_{16}OSe$: C, 68.38; H, 4.59. Found: C, 68.23; H, 4.57.

*6,11-Dihydrodibenzo[b,e]selenepin-11-ol (***11b***)*

Sodium borohydride (1.1 g, 30 mmol) was gradually added to a solution of **10** (2.7 g, 10 mmol) in ethanol (100 ml). The reaction mixture was stirred for 1 hour at room temperature and added to water. The aqueous mixture was extracted with CH_2Cl_2, and the extracts were washed with water, dried ($MgSO_4$), and concentrated. Yellow crystals (2.7 g, 97%) were obtained and used for preparation of **12b** without further purification. ^{1}H NMR ($CDCl_3$ δ) 2.90 (1H, brs, OH), 3.85 and 4.48 (each 1H, d, J = 12 Hz, 5-H), 6.00 (1H, s, 11-H), 6.95–7.57 (8H, m, ArH). IR (KBr) cm^{-1}: 3380 (OH). MS *m/z*: 276 (M^+), 178 (base).

*5-Chloro-5,11-epoxy-11-phenyl-6,11-dihydrodibenzo[b,e]selenepine (***12a***)*

A solution of NCS (380 mg, 2.85 mmol) in dry CH_2Cl_2 (50 mL) was added to a solution of **11a** (1.0 g, 2.85 mmol) in dry CH_2Cl_2 (25 mL). The mixture was stirred for 10 minutes at room temperature, concen-

trated to the amount of 10 mL, and ether was added. The precipitate was collected by filtration and recrystallized from CH_2Cl_2-ether to give colorless prisms (883 mg, 80%), m.p. 238–240°C (dec.). ^{1}H NMR ($CDCl_3$ δ) 4.71 and 5.40 (each 1H, d, J = 16 Hz, 6-H), 6.99 (1H, d, J = 8 Hz, ArH), 7.12 (1H, dd, J = 1, 8 Hz, ArH), 7.18 (1H, dd, J = 7, 8 Hz, ArH), 7.45–7.50 (3H, m, ArH), 7.53–7.60 (4H, m, ArH), 8.68 (1H, m, 4-H). ^{13}C NMR ($CDCl_3$ δ) 50.1 (t), 93.3 (s), 124.8 (d), 126.5 (d), 127.5 (s), 127.7 (d), 128.7 (d), 129.0 (d), 129.3 (d × 2), 129.4 (d × 2), 129.8 (d), 130.1 (d), 131.2 (s), 131.5 (d), 132.2 (d), 136.9 (s), 138.5 (s), 149.6 (s). ^{77}Se NMR ($CDCl_3$ δ) 719.7 MS *m/z*: 386 (M^+), 321 (base). Anal. Calcd. for $C_{20}H_{15}ClOSe$: C, 62.27; H, 3.92. Found: C, 61.99; H, 3.90.

*5-Chloro-5,11-epoxy-6,11-dihydrodibenzo[b,e]selenepine (***12b***)*

Compound **11b** (1.0 g, 3.6 mmol) was similarly treated with NCS (485 mg, 3.6 mmol) in CH_2Cl_2. Recrystallization from CH_2Cl_2-ether gave colorless prisms (1.0 g, 89%), m.p. 171–173°C (dec.). ^{1}H NMR ($CDCl_3$ δ) 4.64 and 5.06 (each 1H, d, J = 16 Hz, 6-H), 6.23 (1H, s, 11-H), 7.00 (1H, d, J = 7 Hz), 7.26–7.35 (3H, m, ArH), 7.41 (1H, m, ArH), 7.46–7.52 (2H, m, ArH), 8.55 (1H, m, 4-H). ^{13}C NMR ($CDCl_3$ δ) 122.4 (d), 125.5 (d), 127.5 (d), 128.0 (s), 129.5 (d), 129.6 (d), 129.9 (d), 130.3 (d), 132.1 (d), 134.0 (s), 134.3 (s), 147.8 (s). ^{77}Se NMR ($CDCl_3$ δ) 726.7. MS *m/z*: 310 (M^+), 275 (base). Anal. Calcd. for $C_{14}H_{11}ClOSe$: C, 54.30; H, 3.58. Found: C, 54.06; H, 3.50.

*5,11-Epoxy-11-phenyl-6,11-dihydrodibenzo[b,e]selenepinium Tetrafluoroborate (***13a***)*

Silver tetrafluoroborate (252 mg, 1.3 mmol) was added to a solution of **12a** (500 mg, 1.3 mmol) in dry CH_2Cl_2 (20 mL) at room temperature. The mixture was stirred for 2 hours, and silver chloride was removed by filtration. The filtrate was concentrated under reduced pressure, and the residue was recrystallized from CH_2Cl_2-ether to give colorless prisms (560 mg, 99%), m.p. 188–190°C (dec.). ^{1}H NMR (DMSO-D_6 δ) 4.11 and 5.34 (each 1H, d, J = 16 Hz, 6-H), 7.10 (2H, m, ArH), 7.22 (1H, dd, J = 7, 8 Hz, ArH), 7.36 (1H, dd, J = 7, 8 Hz, ArH), 7.42 (1H, d, J = 8 Hz, ArH), 7.50–7.56 (3H, m, ArH), 7.57 (1H, d, J = 8 Hz, ArH), 7.58–7.70 (3H, m, ArH), 8.05 (1H, d, J = 7 Hz, 4-H). ^{13}C NMR (DMSO-D_6 δ) 43.3 (t), 95.2 (s), 124.7 (d), 125.4 (s), 125.9 (d), 127.4 (d), 128.3 (d × 2), 128.9 (d), 129.0 (d × 3), 129.5 (d), 129.8 (d), 129.9 (d), 131.1 (s), 132.1 (d), 135.5 (s), 136.5 (s), 147.8 (s). ^{77}Se NMR (DMSO-D_6 δ) 851.5. IR (KBr) cm^{-1}: 900–1200 (BF_4^-). Anal. Calcd. for $C_{20}H_{15}BF_4OSe$: C, 54.96; H, 3.46. Found: C, 54.77; H, 3.44.

*5,11-Epoxy-6,11-dihydrodibenzo[b,e]selenepinium Tetrafluoroborate (***13b***)*

Compound **12b** (500 mg, 1.6 mmol) was similarly treated with silver tetrafluoroborate (314 mg, 1.6 mmol) in CH_2Cl_2. Recrystallization from CH_2Cl_2-ether gave colorless prisms (422 mg, 73%), m.p. 144–146.5°C (dec.). ^{1}H NMR ^{1}H-NMR (DMSO-D_6 δ) 4.02 and 4.97 (each 1H, d, J = 16 Hz, 6-H), 6.71 (1H, s, 11-H), 7.15 (1H, d, J = 7 Hz, ArH), 7.32 (1H, m, ArH), 7.37 (1H, m, ArH), 7.4–7.63 (3H, m, ArH), 7.64 (1H, d, J = 6 Hz, ArH), 7.93 (1H, d, J = 7 Hz, 4-H). ^{13}C NMR (DMSO-D_6 δ) 42.87 (t), 85.5 (d), 122.6 (d), 125.7 (d), 126.0 (s), 127.6 (d), 128.3 (d), 129.5 (d × 2), 130.2 (d), 132.1 (d), 133.7 (s), 134.9 (s), 147.1 (s). ^{77}Se NMR (DMSO-D_6 δ) 858.5. IR (KBr) cm^{-1}: 900–1200 (BF_4^-). Anal. Calcd. for $C_{14}H_{11}BF_4OSe$: C, 46.58; H, 3.07. Found: C, 46.30; H, 3.26.

*5,11-Epoxy-6,11-dihydrodibenzo[b,e]selenepinium Perchlorate (***13c***)*

Silver perchlorate (1.55 g, 7.5 mmol) was added to a solution of **12b** (2.33 g, 7.5 mmol) in dry CH_2Cl_2 (95 ml) at room temperature. The mixture was stirred for 4 hours, and then silver chloride was removed by filtration. The filtrate was concentrated under reduced pressure. The residual solid was recrystallized from CH_2Cl_2-ether to give colorless prisms (2.73 g, 97%), m.p. 178–179°C (dec.). IR (KBr) cm^{-1}: 1000–1200 (ClO_4^-). Anal. Calcd. for $C_{14}H_{11}ClO_4Se$: C, 45.00; H, 2.97. Found: C, 44.89; H, 3.02.

Reactions of **4** *with Organometallic Reagents*

Phenylmagnesium bromide or phenyllithium (0.75 mmol) was added to a suspension of selenonium salt **4** (0.5 mmol) in dry THF at room temperature under a nitrogen atmosphere. The mixture was refluxed for 5 hours with stirring and then cooled and treated with 5% hydrochloric acid with cooling in an ice bath. The whole was extracted with ether. The extracts were washed with water, dried ($MgSO_4$), and concentrated. The residue was separated by preparative TLC on silica gel (hexane–CH_2Cl_2 = 5:1) and the high polar fractions were separated again using hexane–CH_2Cl_2 = 1:1 as an eluant. Reaction conditions and yields of products are shown in Table 1.

o-(Phenylseleno)benzaldehyde (**14a**): ^{1}H NMR ($CDCl_2$ d) 7.01–7.85 (9H, m, ArH), 10.2 (1H, s, CHO).

IR (NaCl) cm^{-1}: 1660 (C=O). MS (*m/z*): 262 (M^+), 77 (base).

o-(Phenylseleno)benzophenone (**14b**): ^1H NMR ($CDCl_3$ δ) 7.21–7.82 (14H, m, ArH). IR (film) cm^{-1}: 1650 (C=O). MS (*m/z*) 338 (M^+), 261 (base). This sample was identical with an authentic sample [11].

o-(Phenylseleno)benzyl alcohol (**2a**): ^1H NMR ($CDCl_3$, δ) 2.08 (1H, s, OH), 4.76 (2H, s, CH_2), 7.25–7.37 (9H, m, ArH). IR (film) cm^{-1}: 3330 (OH). MS (*m/z*): 264 (M^+), 105 (base). This sample was identical with an authentic sample [5].

Phenyl[*o*-(phenylseleno)phenyl]methanol (**2b**): ^1H NMR ($CDCl_3$ δ) 2.50 (1H, s, OH), 6.30 (1H, s, CH), 7.12–7.53 (1H, m, ArH). IR (film) cm^{-1}: 3400 (OH). MS (*m/z*) 340 (M^+), 181 (base). This sample was identical with an authentic sample [5].

o-(Phenylseleno)benzyl phenyl ether (**15a**): ^1H NMR ($CDCl_3$, δ) 5.16 (2H, s, CH_2), 6.91–6.97 (2H, m, ArH), 7.20–7.56 (12H, m, ArH). MS (*m/z*): 340 (M^+), 245 (base).

α-[*o*-(Phenylseleno)phenyl]benzyl phenyl ether (**15b**): ^1H NMR ($CDCl_3$ δ) 6.75 (1H, s, CH), 6.90–7.43 (19H, m, ArH). MS (*m/z*) 416 (M^+), 245 (base).

Reaction of **4b** *with Sodium Hydride*

Selenonium salt **4b** (110 mg, 0.25 mmol) was added to a suspension of NaH (60% in mineral oil) (24 mg, 0.6 mmol) in dry THF (20 mL). The mixture was stirred for 2 days at room temperature and treated with water. The aqueous mixture was extracted with ether, washed with water, dried ($MgSO_4$), and concentrated. The residue was separated by preparative TLC on silica gel (hexane:CH_2Cl_2 = 2:1) to give **2b** (36 mg, 42.5%) and **14b** (27.5 mg, 32.5%).

Reactions of **4c** *with Methyllithium*

An ethereal solution of methyllithium (1 M solution, 1.25 mL, 1.25 mmol) was added to a suspension of selenonium salt **4c** (98 mg, 0.25 mmol) in dry THF (5 mL) at -10°C under a nitrogen atmosphere. The mixture was stirred overnight at that temperature and then treated with 5% hydrochloric acid. The whole was extracted with ether. The extracts were washed with water, dried ($MgSO_4$), and concentrated. The residue was separated by preparative TLC on silica gel (hexane–CH_2Cl_2 = 5:1), and the high polar fractions were separated again using hexane–CH_2Cl_2 = 1:1 as an eluant. 2-(1-Methoxy-1-methyl)ethyl-1-(phenylseleno)benzene (**15c**) (6 mg, 8%) and alcohol **7** (36 mg, 49%). **15c**: ^1H NMR ($CDCl_3$ δ) 1.72 (6H, s, Mex2), 3.18 (3H, s, OMe), 6.98–7.69 (9H, m, ArH). MS (*m/z*): 306 (M+), 73 (base).

Reactions of **13c** *with Grignard Reagents*

An ethereal solution of a Grignard reagent was added to a suspension of **13c** (373 mg, 1 mmol) in dry THF (5 mL) under a nitrogen atmosphere. The mixture was stirred, cooled in an ice bath, and treated with 5% hydrochloric acid. The whole was extracted with ether. The extracts were dried ($MgSO_4$) and concentrated. The residue was separated by preparative TLC on silica gel (hexane–CH_2Cl_2 = 2:1). Reaction conditions and yields of products are listed in Table 2.

o-Benzylphenyl[*o*-(phenylseleno)phenyl]methanol (**16a**): m.p. 137–139°C (dec.). ^1H NMR ($CDCl_3$, δ) 2.06 (1H, brs, OH), 3.94 and 4.09 (each 1H, d, 16Hz, $PhCH_2$), 6.38 (1H, s, CH), 7.04–7.39 (18H, m, ArH). ^{13}C NMR ($CDCl_3$, δ) 38.7 (t), 71.7 (d), 126.1 (d), 126.7 (d), 127.3 (d), 127.5 (d), 127.7 (d × 2), 127.8 (d), 128.4 (d), 128.4 (d), 128.9 (d), 129.3 (d), 130.5 (d), 131.1 (s), 131.2 (s), 132.7 (d), 134.8 (d), 138.7 (s), 140.4 (s), 140.5 (s), 144.1 (s). IR (KBr) cm^{-1}: 3550 (OH). MS (*m/z*): 430 (M^+), 255 (base).

1-[*o*-(Phenylseleno)phenyl]-1,3-dihydrobenzo[*c*]furan (**17a**): ^1H NMR ($CDCl_3$, δ) 5.20 and 5.34 (each 1H, d, J = 12 Hz, 3-H), 6.71 (1H, s, 1-H), 6.96–7.47 (13H, m, ArH). ^{13}C NMR($CDCl_3$, δ) 73.2 (t), 85.3 (d), 120.8 (d), 122.3 (d), 127.3 (d), 127.4 (d), 127.6 (d), 128.1 (d), 128.3 (d), 128.8 (d), 129.3 (d), 130.8 (s), 131.3 (s), 132.9 (d), 134.5 (d), 139.3 (s), 141.4 (s), 143.4 (s). MS (*m/z*): 352 (M^+), 178 (base).

1-[*o*-(Methylseleno)phenyl]-1,3-dihydrobenzo[c]furan (**17b**): ^1H NMR ($CDCl_3$, δ) 2.37 (3H, s, CH_3), 5.23 and 5.35 (each 1H, d, J = 12 Hz, 3-H), 6.64 (1H, s, 1-H), 7.09–8.17 (8H, m, ArH). MS (*m/z*): 290 (M^+), 165 (base).

Reaction of **12b** *or* **13b** *with Triethylamine*

Reaction of **12b**: Dry triethylamine (0.5 mL, 3.6 mmol) was added to a solution of **12b** (250 mg, 0.65 mmol) in dry CH_2Cl_2 (10 mL). The mixture was stirred at room temperature for 3.5 hours and then concentrated to dryness. The residue was washed with ether several times, and the washings were concentrated. The colorless solid was recrystallized from CH_2Cl_2-hexane to give 6,11-epoxy-11-phenyl-6,11-dihydrodibenzo[*b,e*]selenopine (**18**) as colorless prisms (40 mg, 18%), m.p. 148–150°C. ^1H NMR ($CDCl_3$ δ) 6.89 (1H, s, 6-H), 6.94–7.14 (4H, m, ArH), 7.26–7.38 (3H, m, ArH), 7.41–7.50 (4H, m, ArH), 7.64–7.71 (2H, m, ArH). ^{13}C NMR ($CDCl_3$ δ) 78.9 (d), 91.1 (s), 119.9 (d), 121.9 (d), 125.4 (d), 127.0 (d), 127.9 (d), 128.3 (d), 128.4 (d), 128.5 (d × 2), 128.7 (s), 128.9 (d), 129.1 (d × 2), 131.6 (d), 135.2 (s), 138.2 (s), 140.7 (s), 141.8 (s). ^{77}Se NMR ($CDCl_3$ δ) 422.1.

MS *m/z*: 350 (M^+), 321 (base). Anal. Calcd. for $C_{20}H_{14}OSe$: C, 68.77; H, 4.04. Found: C, 68.49; H, 4.01. Reaction of 13b: Compound **13b** (500 mg, 1.1 mmol) was similarly treated with triethylamine (1 mL, 7.2 mmol), and **18** (272 mg, 68%) was obtained.

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