Tetrahydrofuran Derivatives from Epoxides *via* Group Transfer Cyclization or Reductive Radical Cyclization of Organotellurium and Organoselenium Intermediates

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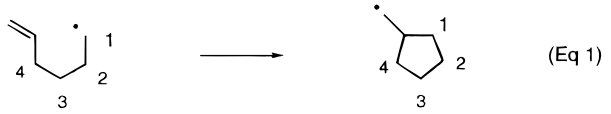
Monosubstituted epoxides were regiospecifically ring-opened from the sterically least hindered side by benzenetellurolate and benzeneselenolate reagents to afford aryl β-hydroxyalkyl tellurides and selenides, respectively. These materials were O-allylated by treatment with allylic bromides/sodium hydride in tetrahydrofuran and O-prop-2-ynylated when reacted with propargyl bromide/sodium hydride. On photolysis in benzene containing 40 mol % of hexabutylditin, the β-(allyloxy)alkyl aryl tellurides were found to undergo group transfer cyclization to afford 2-substituted 4-[(aryltelluro)methyl]tetrahydrofurans (*cis*/*trans* = 1/3−1/10). The aryl β-(prop-2-ynyloxy)alkyl tellurides similarly afforded 2-substituted 4-[(aryltelluro)methylene]tetrahydrofurans with an *E*/*Z*-ratio close to unity. The β-(allyloxy)alkyl aryl selenides and aryl β-(prop-2-ynyloxy)alkyl selenides failed to undergo group transfer cyclization. In the presence of tributyltin hydride and 2,2′-azobisisobutyronitrile, the former compounds were found to undergo reductive radical cyclization in high yields to afford 2-substituted 4-methyltetrahydrofurans (*cis*/*trans* = 1/3−1/10). Aryl β-(prop-2-ynyloxy)-alkyl selenides similarly afforded 2-substituted 4-methylenetetrahydrofurans. 2-Alkoxy-2-(allyloxy)-ethyl phenyl selenides, prepared by allyloxyselenenation of vinyl ethers, were found to undergo reductive radical cyclization to afford 2-alkoxy-4-methyltetrahydrofurans (*cis*/*trans* = 1/3−1/4). The preference for formation of *trans*-2,4-disubstituted tetrahydrofurans in the group transfer and reductive radical cyclizations was rationalized assuming a chairlike transition state with a preferred adoption of a pseudoequatorial position of the 2-substituent. By carrying out the reactions at lower temperatures (ambient or −45 °C), using triethylborane as an initiator, it was possible to further increase the *trans* selectivity in the reductive cyclizations.

Introduction

In addition to transition metal-mediated synthesis, free radical chemistry has emerged as one of the most powerful tools in modern synthetic chemistry.[1] The rapid ($k_{25} = 2 \times 10^5$ s^{-1}),[2] irreversible, and regioselective (98% *exo*) cyclization of the 5-hexenyl radical (eq 1) has not only been exceedingly useful for the construction of



(Eq 1)

cyclopentanes but also been applied extensively, *via* substitution with oxygen, for the preparation of tetrahydrofuran derivatives. As compared to the unperturbed system, oxygen substitution at positions 1 ($k_{30} = 4 \times 10^8$ s^{-1})[3] and 3 ($k_{25} = 9 \times 10^6$ s^{-1})[4] have an accelerating effect, whereas substitution at position 2 retards the cyclization ($k_{25} = 5 \times 10^4$ s^{-1}).[4] There are synthetically useful radical cyclizations described, though, where the oxygen atom has been incorporated at positions 1,[3,5] 2,[6,7] 3, and 4.[8]

A vast majority of the tetrahydrofuran syntheses involve a cyclization of a 3-oxa-5-hexenyl system.[9] As

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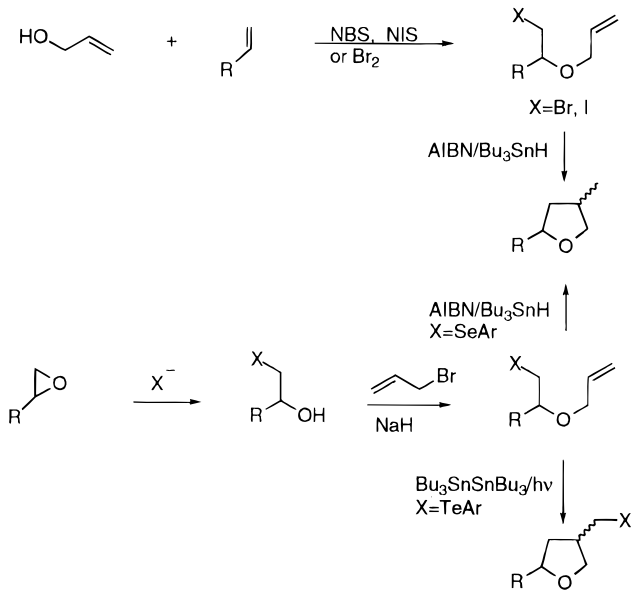
Scheme 1



originally described by the groups of Ueno[10] and Stork,[11] the precursors to such radicals can be conveniently assembled from allylic alcohols, vinyl ethers/olefins, and a source of positive halogen (Scheme 1, upper part). Except for this methodology, few general methods are known for radical precursor synthesis. The scarce examples reported include the conjugate addition[12,13] and allylation[14] of allylic alcohols as well as some other methods.[15,16]

We thought it was possible to prepare tetrahydrofuran derivatives from epoxides by a series of operations involving ring-opening with a nucleophilic reagent X, O-allylation, and reductive radical cyclization (Scheme 1, lower part). It is required, though, that the nucleophilic reagent has the additional capacity to act as a source of a carbon-centered radical. We recently reported the ring-opening of epoxides by arenetellurolate ion and the preparation of tetrahydrofuran derivatives by a hexabutylditin/light-induced group transfer cyclization reaction of the O-allylated telluride (Scheme 1, lower part).[17] In the following, we report a full account of this and related work with the corresponding organoselenium compounds.

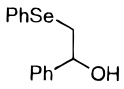
Results

Ring-Opening of Epoxides by Tellurolate and Selenolate Reagents. Epoxides are readily ring-opened by tellurolate and selenolate reagents.[18] The reaction is *anti* stereospecific, and, in the case of mono and trisubstituted epoxides, highly regiospecific (secondary and tertiary alcohols, respectively, formed). With unsymmetrical 1,2-disubstituted epoxides, mixtures of regioisomers are usually obtained. In the present investigation, monosubstituted (R′ = H) and symmetrical disubstituted epoxides (R = R′) were treated with benzenetellurolate and benzeneselenolate ions in ethanol at ambient temperature to afford β-hydroxyalkyl aryl tellurides and selenides as shown in eq 2. The required organochalcogen reagents were conveniently prepared

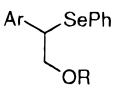
(Eq 2)

1 X=Te, Se

from sodium borohydride and the respective diaryl dichalcogenides in ethanol under nitrogen. The yields and structures of the aryl β-hydroxyalkyl tellurides and aryl β-hydroxyalkyl selenides prepared are reported in Tables 1 and 2, respectively. In contrast to the other monosubstituted epoxides (1,2-epoxy-5-hexene, 1,2-epoxybutane, 3-(allyloxy)-1,2-epoxypropane, 1,2-epoxy-3-phenoxypropane and 3-(benzyloxy)-1,2-epoxypropane), styrene oxide afforded a 56/44-mixture of the two possible ring-opening products **1m** and **2a** (94% yield). A similarly low regioselectivity has previously been observed in the benzeneselenolate induced ring-opening of styrene oxide.[19,20] However, both isomers were useful in the following allylation/propargylation and radical chemistry. By treatment of styrene oxide with sodium 4-(trifluoromethyl)benzenetellurolate, the secondary alcohol **1f** (Table 1) was the only isomer that could be isolated in a pure form (50% yield). As a representative of a 2,2-disubstituted terminal epoxide, 1,2-epoxy-2-methylpropane, was ring-opened from the sterically least hindered side by tellurolate and selenolate reagents. However, all attempts to allylate the resulting tertiary alcohols met with failure.



1m



2 a R=H
b R=allyl
c R=3-methyl-2-butenyl
d R=2-propynyl

Allylation and Propargylation of Aryl β-Hydroxyalkyl Tellurides and Selenides. By treatment with allyl bromide in tetrahydrofuran in the presence of sodium hydride, the aryl β-hydroxyalkyl tellurides and selenides **1** were O-allylated without competing epoxide formation (eq 3, Tables 1 and 2). Some of the compounds (**1m**, **1o**, **1p**; Table 2) were similarly O-prenylated using 4-bromo-2-methyl-2-butene and most of them were O-prop-2-ynylated by treatment with propargyl bromide (eq 3, Tables 1 and 2). Selenide **2a** was allylated, prenylated, and prop-2-ynylated, respectively, to give compounds **2b**, **2c**, and **2d**.

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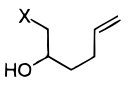
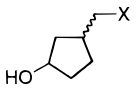
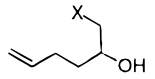
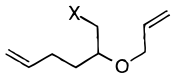
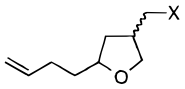
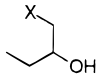
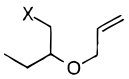
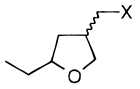
Table 1. Organotellurium Group Transfer Cyclization Products and Intermediates

2-Hydroxyalkyl phenyl tellurides **1** (% yield[a])	O-allylation (**3**) or O-prop-2-ynylation (**4**) intermediates (% yield[b])	Group transfer cyclization products **5, 6, 7** (% yield[a]; cis/trans[c] or E/Z[c]- ratio)
		
1a X=TeC$_6$H$_5$ (80) **1b** X=TeC$_6$H$_4$-4-NMe$_2$(97) **1c** X=TeC$_6$H$_4$-4-CF$_3$(76) **1d** X=Te-2-thienyl(60)	- - - -	**5a** X=TeC$_6$H$_5$ (68;1.3/1) **5b** X=TeC$_6$H$_4$-4-NMe$_2$(63;1.3/1) **5c** X=TeC$_6$H$_4$-4-CF$_3$(73;1.3/1) **5d** X=Te-2-thienyl(75;1/1)
		
1d	**3d** X=Te-2-thienyl(72)	**6d** X=Te-2-thienyl(63;1/4)
		
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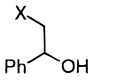
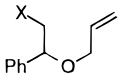
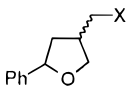
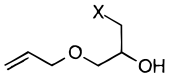
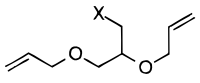
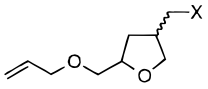
		
1f X=TeC$_6$H$_4$-4-CF$_3$ (50)	**3f** X=TeC$_6$H$_4$-4-CF$_3$ (75)	**6f** X=TeC$_6$H$_4$-4-CF$_3$(65;1/3)
		
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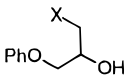	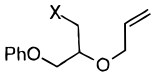	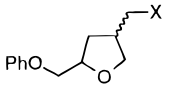
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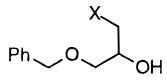
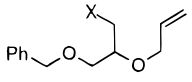
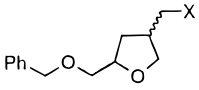
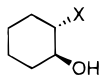
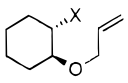
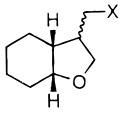
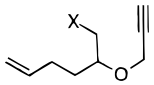
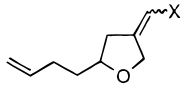
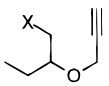
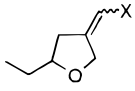
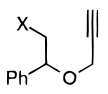
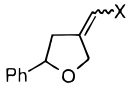
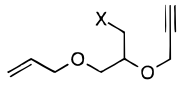
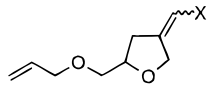
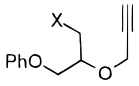
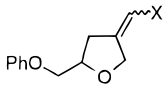
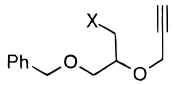
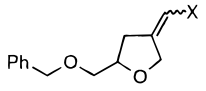
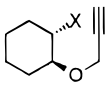
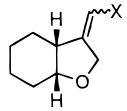
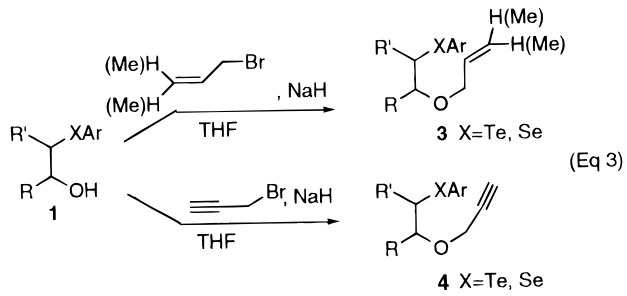
		
1i X=TeC$_6$H$_4$-4-CF$_3$ (64)	**3i** X=TeC$_6$H$_4$-4-CF$_3$ (74)	**6i** X=TeC$_6$H$_4$-4-CF$_3$ (60;1/10)
		
1j X=TeC$_6$H$_4$-4-CF$_3$ (82) **1k** X=Te-2-thienyl (61)	**3j** X=TeC$_6$H$_4$-4-CF$_3$ (80) **3k** X=Te-2-thienyl (60)	**6j** X=TeC$_6$H$_4$-4-CF$_3$(69;2/1[d]) **6k** X=Te-2-thienyl (72;2/1[d])
		
1c	**4c** X=TeC$_6$H$_4$-4-CF$_3$(70)	**7c** X=TeC$_6$H$_4$-4-CF$_3$(46;1.5/1)
		
1e	**4e** X=TeC$_6$H$_4$-4-CF$_3$(37)	**7e** X=TeC$_6$H$_4$-4-CF$_3$(49;1/1.2)

Table 1. (Continued)

2-Hydroxyalkyl phenyl tellurides **1** (% yield[a])	O-allylation (**3**) or O-prop-2-ynylation (**4**) intermediates (% yield[b])	Group transfer cyclization products **5, 6, 7** (% yield[a]; cis/trans[c] or E/Z[c]- ratio)
1f	 **4f** X=TeC_6H_4-4-CF_3 (75)	 **7f** X=TeC_6H_4-4-CF_3(47;1/1.2)
	 **4g** X=TeC_6H_4-4-CF_3 (82*,51)	 **7g** X=TeC_6H_4-4-CF_3 (47;1/1)
1h	 **4h**=TeC_6H_4-4-CF_3 (95*,31)	 **7h** X=TeC_6H_4-4-CF_3(48;1/1.1)
1i	 **4i**=TeC_6H_4-4-CF_3 (80*,36)	 **7i**=TeC_6H_4-4-CF_3(46;1/1.1)
1j	 **4j** X=TeC_6H_4-4-CF_3 (55)	 **7j** X=TeC_6H_4-4-CF_3 (40;1.2/1)

[a] isolated yields [b] isolated yields; yields marked by an asterix are crude yields [c] as determined by NOESY and NOE experiments [d] *exo*/*endo*-ratio



(Eq 3)

The prop-2-ynylated derivatives were prepared as precursors to 3-oxa-5-hexynyl radicals. These are known to readily cyclize in a 5-*exo* mode to afford 3-methylenetetrahydrofurans.[21−23] So far, precursors to such radicals were available from propargylic alcohols and olefins by using the chemistry shown in the upper part of Scheme 1.

In general, O-prop-2-ynylation was effected in poorer yield than the correponding allylation. This was particularly true for the organotellurium compounds. As indicated in Table 1, attempted chromatographic purification seemed to reduce the isolated yields of the prop-2-ynylated tellurides. Therefore, the following free radical chemistry (*vide infra*) was better performed by using the crude reaction product.

Group Transfer Cyclization Reactions. In atom and group transfer-mediated radical reactions an atom A (hydrogen,[24] halogen,[25] or metal[26]) or a group G (such as SePh[27]) is transferred in the chain-transfer step from a neutral molecule to a radical to form a new σ-bond and a new radical (Scheme 2). For such processes to be synthetically useful, the atom/group transfer step has to be preceded by a radical addition, cyclization, annulation, or fragmentation reaction. Due to work of the Barton

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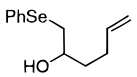
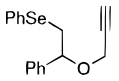
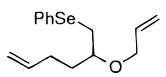
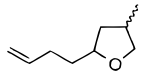
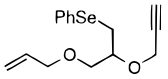
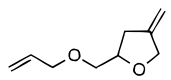
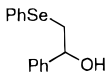
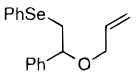
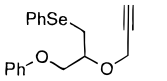
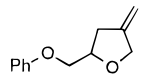
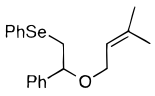
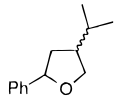
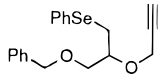
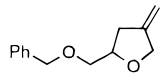
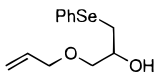
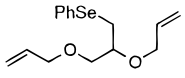
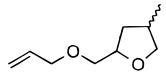
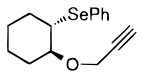
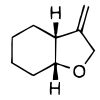
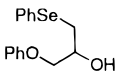
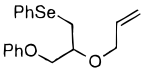
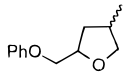
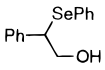
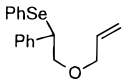
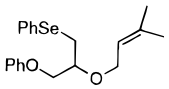
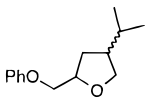
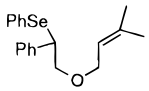
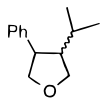
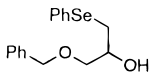
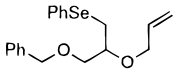
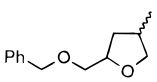
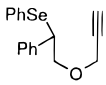
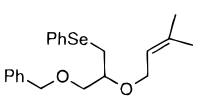
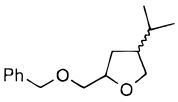
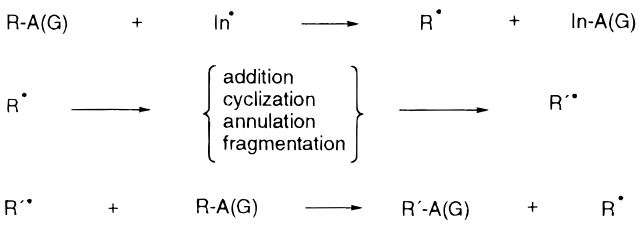
Table 2. Reductive Radical Cyclization Products and Organoselenium Intermediates

2-Hydroxyalkyl phenyl selenides **1** and **2a** (% yield[a])	O-allylation or O-prop-2-ynylation intermediates **2-4**, **14**, **17** (% yield[a])	Reductive radical cyclization products **8-13**, **15**, **18** (% yield[a];cis/trans[b]-ratio)	2-Hydroxyalkyl phenyl selenides **1** and **2a** (% yield[a])	O-allylation or O-prop-2-ynylation intermediates **2-4**, **14**, **17** (% yield[a])	Reductive radical cyclization products **8-13**, **15**, **18** (% yield[a];cis/trans[b]-ratio)
 **1l** (88)	-	 **8** (52[c]; 1.3/1)	 **1m**	**4m** (77)	 **10m** (53)
1l	 **3l** (90)	 **9l** (85; 1/4)	**1n**	 **4n** (71)	 **10n** (51)
 **1m** (53)	 **3m** (88)	 **9m** (92;1/3.7)	**1o**	 **4o** (90)	 **10o** (39)
1m	 **3m'** (85)	 **9m'** (93;1/1.4)	**1p**	 **4p** (80)	 **10p** (57)
 **1n** (96)	 **3n** (85)	 **9n** (84;1/10)	**1q**	 **4q** (77)	 **10q** (70)
 **1o** (84)	 **3o** (92)	 **9o** (79;1/3)	 **2a** (41)	 **2b** (85)	 **11** (85; 1/3)
1o	 **3o´** (84)	 **9o´** (76;1/1.3)	**2a**	 **2c** (72)	 **12** (84; 1/2)
 **1p** (90)	 **3p** (82)	 **9p** (94;1/3)	**2a**	 **2d** (91)	 **13** (70)
1p	 **3p´** (86)	 **9p´** (98;1/2)		**14 a** R=Et (58) **b** R=Bu (74) **c** R= t-Bu (65)	**15 a** R=Et (66; 1/4) **b** R=Bu (76; 1/4) **c** R= *t*-Bu (76; 1/3)
1q (91)	**3q** (75)	**9q** (73;1/1.5[d])		**17** (54)	**18** (69; 1/4[d])
1l	**4l** (84)	**10l** (nd)			

[a] isolated yields [b] as determined by NOESY and NOE experiments [c] GC yield [d] *exo*/*endo*-ratio

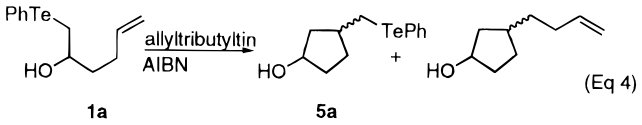
group, diorganyl tellurides are known to act as efficient exchangers of carbon-centered radicals.[28] Furthermore, rate data from Currans group[29] indicate that TePh group transfer occurs as readily as iodine atom transfer. It is therefore surprising to find so few examples where the synthetic potential of group transfer chemistry has been

Scheme 2



taken advantage of. The scarce examples reported so far include the photostimulated cyclization of unsaturated acyl tellurides[30] and the AIBN-induced group transfer addition of diorganyl tellurides to acetylenes.[31]

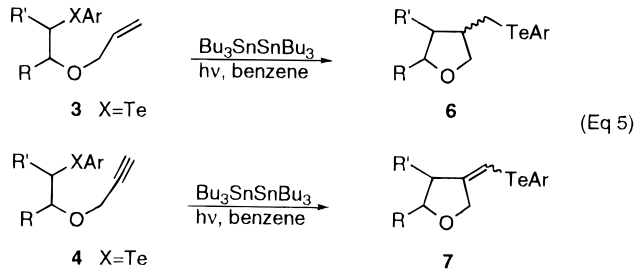
During the initial explorative phase of the present project, 2-hydroxy-5-hexenyl phenyl telluride **1a** was heated at reflux in benzene together with AIBN and allyltributyltin. In addition to small amounts of the expected radical cyclization/allylation product, we isolated the radical cyclization/group transfer product **5a** as the major product (eq 4). Our efforts to optimize the



1a **5a** (Eq 4)

conditions for cyclization/group transfer showed that the desired product was formed in good yield by sun lamp irradiation of a 0.05−0.1 M refluxing solution of the telluride in benzene under nitrogen in the presence of 40 mol % of hexabutylditin. Among the different aryl groups tested (Table 1, compounds **1a**−**d**), the 4-(trifluoromethyl)phenyl group turned out to be superior with respect to the time required to obtain complete conversion of the starting material (2 h for compound **1c** as compared to 4−6 h for the other three compounds). Also, as compared with the other derivatives, the compounds carrying a 4-(trifluoromethyl)phenyl group showed an increased stability during storage, handling, and chromatographic purification. The cyclopentane derivatives **5a**−**d** were all obtained as mixtures containing near to equal amounts of the *cis* and *trans* compounds. The isomeric composition of product **5a** was determined after benzoylation, hydrodetelluration (Bu_3SnH/AIBN/refluxing benzene) and comparison with an authentic sample obtained by benzoylation of commercially available 3-methylcyclopentanol. The isomeric composition of products **5b**−**d** was assigned directly from their ^1H NMR spectra in analogy with the result for compound **5a**.

When irradiated in the presence of hexabutylditin, O-allylated and O-prop-2-ynylated aryl β-hydroxyalkyl tellurides **3** and **4** were found to cyclize with group transfer to form tetrahydrofuran derivatives **6** and **7**, respectively (eq 5, Table 1). Also in the cases where the R group contained unsaturation (R = 3-butenyl, R′ = H; Table 1, compounds **3d** and **4c)** products of carbocyclization were not detected. This is not surprising in view of



(Eq 5)

the strongly accelerating effect of a 3-oxa substituent in 5-hexenyl and 5-hexynyl radical cyclization.[32] Group transfer products of type **6** (60−70% yields) and **7** (40−50% yields) with a substantial variation in the 2-substituent were prepared as shown in Table 1 [R′ = H, R = ethyl, phenyl, (allyloxy)methyl, phenoxymethyl, or (benzyloxy)methyl; R, R′ = $(CH_2)_4$]. The tetrahydrofuran derivatives **6** were obtained as mixtures of *cis* and *trans* isomers. The isomeric composition and the assignment of ^1H NMR spectra to the isomers were based on NOESY, NOE-difference and ZTOCSY experiments (*vide infra*). As shown in Table 1, the *trans* isomer always predominated with a ratio of 3/1−10/1 over the *cis* isomer. Bicyclic compounds **6j** and **6k** were isolated as 2/1 mixtures of *exo* and *endo* isomers, both with a *cis* ring fusion.

The tetrahydrofuran derivatives **7** were isolated as (sometimes separable) mixtures of *E* and *Z* isomers. The isomeric composition and the assignment of ^1H NMR spectra to the isomers were based on NOESY, NOE-difference, and ZTOCSY experiments. As shown in Table 1, the *E*/*Z* ratio was always close to unity. The product yields for group transfer cyclization to an acetylene were always inferior to those obtained with the corresponding olefinic compounds. Due to problems in the purification of the O-prop-2-ynylated aryl β-hydroxyalkyl tellurides, group transfer cyclization was somtimes attempted using the crude prop-2-ynylation product (Table 1). Product yields never exceeded 50%. Due to inversion at the radical center, the bicyclic tetrahydrofuran **7j** was obtained as an *E*/*Z*-mixture of *cis* fused compounds.

With the perspective to increase the diastereoselectivity in the group transfer cyclization reactions, other initiators were sought which could be effective at lower temperatures than those used in the above photochemical process. Triethylborane in the presence or absence of air has been used, at or below room temperature, to induce group transfer cyclization,[33] reductive radical cyclization[7,34] as well as other free radical chemistry such as addition[35] and substitution[36] reactions. When telluride **3j** was treated in toluene at ambient temperature with 1.2 equiv. of triethylborane under an atmosphere of nitrogen, the group transfer product **6j** was isolated in 60% yield after 5 h. However, the diastereoselectivity (*exo*/*endo* = 2/1) did not differ from that observed at 80 °C. Although the mild reaction conditions and the absence of organotin compounds are attractive features of this reaction, we generally found it less useful than

(28) Barton, D. H. R.; Ozbalik, N.; Sarma, J. C. *Tetrahedron Lett.* **1988**, *29*, 6581.
(29) Curran, D. P.; Martin-Esker, A. A; Ko, S.-B.; Newcomb, M. *J. Org. Chem.* **1993**, *58*, 4691.
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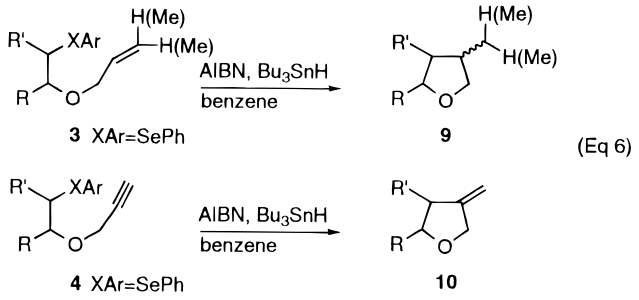
(31) Han, L.-B.; Ishihara, K.-I.; Kambe, N.; Ogawa, A.; Ryu, I.; Sonoda, N. *J. Am. Chem. Soc.* **1992**, *114*, 7591.
(32) See ref 1f, p 151.
(33) Ichinose, Y.; Matsunaga, S.; Fugami, K.; Oshima, K.; Utimoto, K. *Tetrahedron Lett.* **1989**, *30*, 3155.
(34) Clive, D. L. J.; Manning, H. W.; Boivin, T. L. B.; Postema, M. H. D. *J. Org. Chem.* **1993**, *58*, 6857.
(35) Ichinose, Y.; Wakamatsu, K.; Nozaki, K.; Birbaum, J.-L.; Oshima, K., Utimoto, K. *Chem. Lett.* **1987**, 1647. Nozaki, K.; Oshima, K.; Utimoto, K. *J. Am. Chem. Soc.* **1987**, *109*, 2547. Benati, L.; Capella, L.; Montevecchi, P. C.; Spagnolo, P. *J. Org. Chem.* **1995**, *60*, 7941.
(36) Baciocchi, E.; Muraglia, E. *Tetrahedron Lett.* **1993**, *34*, 5015.

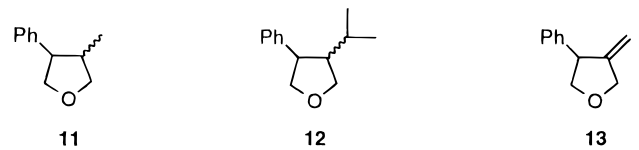
the photochemical method for the initiation of group transfer cyclization in primary tellurides.

Reductive Radical Cyclization. Although the phenylseleno group is known to undergo group transfer reactions,[27] all attempts to effect group transfer cyclization with O-allylated or O-prop-2-ynylated β-hydroxyalkyl phenyl selenides **3** and **4** met with failure. On the other hand, these materials were found to undergo reductive radical cyclization on treatment with AIBN and tributyltin hydride under standard conditions avoiding the use of a syringe pump (eq 6). Similar reactions are not without precedence in the literature. However, the



required organoselenium radical precursors were usually prepared by less general[37] or less direct[38] methods.

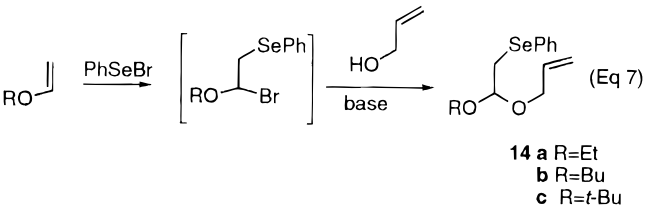
2-Hydroxy-5-pentenyl phenyl selenide (**1l**, Table 2) afforded the carbocycle 3-methylcyclopentanol (**8**) in 52% yield as a 1.3/1 mixture of *cis* and *trans* isomers. As was the case in the telluride series, its O-allylated derivative (**3l**) afforded only the product resulting from 3-oxa-5-hexenyl radical cyclization. In Table 2, the preparation of a variety of substituted 4-methyltetrahydrofurans **9** [R′ = H, R = 3-butenyl, phenyl, (allyloxy)methyl, phenoxymethyl, or (benzyloxy)methyl; R = R′ = $(CH_2)_4$] and 4-isopropyltetrahydrofurans **9** (R′ = H, R = phenyl, phenoxymethyl, or (benzyloxy)methyl) in 80−90% yields from selenides **3** are reported. The isomeric composition and the assignment of the ^{1}H NMR spectra to the isomers were performed as described above for group transfer products and exemplified below. The *trans* isomers of the 2-substituted 4-methyltetrahydrofurans predominated, usually with a ratio of 3/1−4/1 over the *cis* isomers. However, a significantly lower selectivity (*trans*/*cis* = 1.3/1−2/1) was observed for the 2-substituted 4-isopropyltetrahydrofurans prepared (compounds **9m′**, **9o′**, and **9p′**; Table 2). The two 3,4-disubstituted tetrahydrofurans synthesized (compounds **11** and **12**) were obtained predominantly (3/1 and 2/1, respectively) as *trans* isomers. The bicyclic compound **9q** was isolated as a 1.5 /1 mixture of *cis* fused *exo* and *endo* isomers.



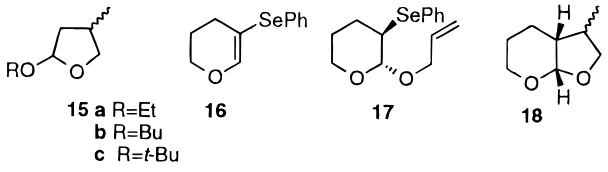
2-Substituted 4-methylenetetrahydrofurans **10** [R′ = H, R = 3-butenyl, phenyl, (allyloxy)methyl, phenoxymethyl, or (benzyloxy)methyl; R = R′ = $(CH_2)_4$] were prepared in 40−70% yields from acetylenic compounds **3** under the standard radical cyclization conditions (eq 6, Table 2). 4-Methylene-3-phenyltetrahydrofuran (**13**) was isolated in 70% yield under the similar conditions.

Some reductive cyclizations were also tried at lower temperatures under nitrogen, using triethylborane as the initiator. Thus, in the presence of tributyltin hydride, the *cis*/*trans* ratio of compound **11** was lowered from 1/3 at 80 °C to 1/3.3 at 25 °C and 1/6 at −45 °C. The isolated yields at the respective temperatures were 85, 73, and 52%.

Already in 1978, Petrzilka[39] reported the regiospecific addition of phenylselenenyl bromide to ethyl vinyl ether and the base-induced substitution of the bromine with various allylic alcohols (eq 7). The products **14** would be expected (*cf.* Scheme 1, upper part) to serve as



excellent precursors to 2-alkoxy-4-methyltetrahydrofurans *via* 3-oxa-5-hexenyl radical cyclization. Some compounds of this type were therefore prepared and the diastereoselectivity of the cyclization determined. When submitted to the standard reaction conditions, compounds **14a**−**c** afforded the 2-alkoxy-4-methyltetrahydrofurans **15a**−**c** in 66, 76, and 76% yields, respectively. The *trans* isomers predominated with a ratio of 4/1 for the two former compounds and with a ratio of 3/1 for the latter. In the preparation of compound **14c**, it was found essential to use phenylselenenyl chloride to obtain the desired product in fair yield (65%). When 3,4-dihydropyran was treated with phenylselenenyl bromide and then with allyl alcohol/triethylamine, the elimination product **16** was the only material isolated (68% yield). However, by using phenylselenenyl chloride instead of the bromide, the desired alkoxyselenenation product **17** was obtained in 54% yield. Reductive cyclization of this



material afforded the bicyclic tetrahydrofuran derivative **18** as a 1/4 mixture of *cis* fused *exo* and *endo* isomers (69% yield). As noted above, triethylborane-induced reductive cyclizations at ambient temperature under nitrogen occurred with improved diastereoselectivities. Thus, compounds **15a** and **15b** were obtained as 1/6 mixtures of *cis* and *trans* isomers at 25 °C. Similarly, the *exo*/*endo* ratio of compound **18** dropped to 1/16 at ambient temperature. However, since the ratio at 80 °C in the triethylborane-initiated reaction was 1/8, this dramatic difference may only in part be ascribed to the effect of the temperature.

Determination of Diastereoselectivity. The assignment of *cis*/*trans*, *E*/*Z*, and *exo*/*endo* isomers of the

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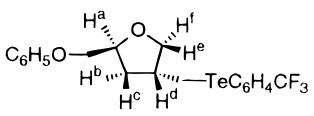
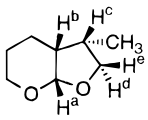


trans-**6h**

NOE-effects
H^a to H^b 5%
H^c to H^d 8%
H^d to H^e 5%
H^a to H^f 3%



endo-**18**

NOE-effects
H^a to H^b 5%
H^a to H^c 3%
H^c to H^b 5%
H^c to H^e 5%
H^e to H^a 4%

Figure 1.

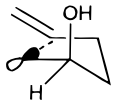
various tetrahydrofuran derivatives prepared were based on NOESY and/or NOE difference experiments. As representative examples, the NOE enhancements observed for the *trans* isomer of compound **6h** and the *endo* isomer of compound **18** are shown in Figure 1. Usually, it was possible to establish the relative 1,3-stereochemistry by either, or both, of two routes. As shown for compound **6h**, the 1,3-*trans* stereochemistry is suggested by the *cis* arrangement of H^a/H^f and H^d/H^e and also by the *cis* arrangement of H^a/H^b and H^c/H^d. Due to overlapping signals of H^b and H^c, the latter route sometimes failed. *Exo* and *endo* isomers were similarly distinguished as shown for *endo*-**18** (Figure 1).

Discussion

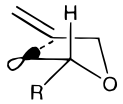
In the present paper, we have demonstrated a novel versatile route to 3-oxa-5-hexenyl and 3-oxa-5-hexynyl radical precursors based on ring-opening of epoxides with arenetellurolate or areneselenolate reagents and O-allylation/O-prop-2-ynylation. Due to the facile group transfer of the aryltelluro group, the organotellurium compounds were transformed by light/hexabutylditin into 2-substituted tetrahydrofuran derivatives **6** and **7**, carrying an aryltelluro group in the 4-methyl and 4-methylene substituents, respectively. These products are probably formed as outlined in Scheme 2 (G = TeAr; In = Bu_3Sn; [3-oxa-5-hexenyl or 3-oxa-5-hexynyl cyclization]). Photostimulated atom transfer reactions with iodine have previously been effected by using only 10 mol % of hexabutylditin.[25] However, to obtain high conversions of the organotellurium starting materials within reasonable time (2−3 h), as much as 40 mol % of hexabutylditin was required. Since the cyclization and group transfer steps in Scheme 2 could be expected to be fast, this may be due to a relatively inefficient initiation process.

Group transfer of the phenylseleno group has been demonstrated both in addition and cyclization reactions. However, under the conditions where aryltelluro group transfer was successful (hexabutylditin/light, triethylborane), the corresponding organoselenium compounds failed to react. This difference may be a consequence of poor initiation and a low rate of phenylseleno group transfer.[29] In the presence of an efficient hydrogen atom donor (tributyltin hydride) and an initiator (AIBN or triethylborane) products of reductive radical cyclization were rapidly formed, uncontaminated by any uncyclized reduction products even under conditions of high tin hydride concentration.

The diastereoselectivity observed in the cyclization of substituted 5-hexenyl radicals has been rationalized assuming a chairlike transition state with a preferred adoption of a pseudoequatorial position of the substituent.[40] In accord with this model, 2-methyl- and 4-methyl-5-hexenyl radicals were found to cyclize with preferential formation of the *trans* disubstituted cyclopentane whereas the 3-methyl derivative afforded mainly *cis*-1,3-dimethylcyclopentane. On cyclization, the 1-methyl-5-hexenyl radical afforded mainly the *cis* isomer. However, calculation and experiment suggest that the diastereoselectivity could be inverted by introduction of a bulky 1-substituent.[41] In contrast to the product obtained from reductive cyclization of the 2-methyl-5-hexenyl radical, the group transfer (compounds **5a**−**d**) and reductive cyclization products (compound **8**) derived from the 2-hydroxy-5-hexenyl radical were isolated mainly as *cis*-1,3 disubstituted cyclopentanes. The reason for this change in diastereoselectivity in not obvious. One may speculate that the pseudoaxially oriented hydroxyl group avoids an unfavorable interaction with the singly occupied molecular orbital (SOMO) in the transition state **19**.



19

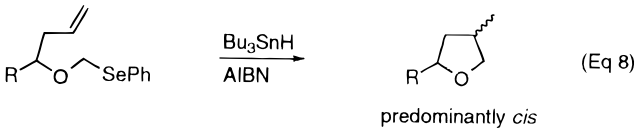


20

All group transfer and reductive cyclization reactions involving 2-substituted 3-oxa-5-hexenyl radicals afforded the *trans*-2,4-disubstituted tetrahydrofuran as the predominating diastereomer. However, for most of the reactions the selectivity was only moderate (2/1−4/1) and usually very similar for group transfer cyclization (Table 1) and reductive cyclization (Table 2) products derived from the same epoxide. These results can be rationalized by invoking a chairlike transition state **20** for the ring-closures, with the substituent occupying a pseudoequatorial position. Some group transfer and reductive radical cyclizations occurred with significantly higher diastereoselectivities than the others (**6g**; *cis*/*trans* = 1/10, **6i**; *cis*/*trans* = 1/10;[42] **9n**; *cis*/*trans* = 1/10). The similar results with compounds **6g** and **9n** may indicate that the increased selectivity is caused by the tetrahydrofuran 2-substituent. However, the reductive cyclization of compound **3p** afforded tetrahydrofuran **9p** with a significantly poorer (*cis*/*trans* = 1/3) selectivity than was obtained in the corresponding group transfer cyclization. Therefore, telluride **3i** was subjected to reductive radical cyclization. By using the standard procedure for selenides, compound **9p** was isolated in 86% yield as a 1/3 mixture of *cis* and *trans* isomers. One remaining hypothesis to explain the high diastereoselectivity in the formation of compound **6i** involves participation of the hexabutylditin in the transition state of the group transfer reaction.

With respect to diastereoselectivity, the methodology presented herein for the preparation of 2,4-disubstituted tetrahydrofurans is complementary to the procedure described by Rawal (eq 8).[7] The *cis*-selectivity in this reaction is again explainable by assuming a chairlike transition state with an equatorial substituent in the

(40) Beckwith, A. L. J.; Schiesser, C. H. *Tetrahedron* **1985**, *41*, 3925 and references cited therein.
(41) See ref 1e, p 214, and references cited there.
(42) The *cis*/*trans* ratio of this compound was erroneously reported as <1/20 in our preliminary communication of part of this work (see reference 17).



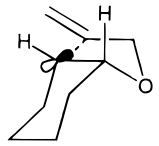
(Eq 8)

predominantly *cis*

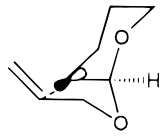
3-position. It must be emphasized, though, that the radical precursors of this synthesis generally are less available than the ones described in the present work. The *cis* and *trans* isomers of 4-methyl-2-phenyltetrahydrofuran were previously prepared with good stereocontrol by reductive radical cyclization of allyl 1-phenyl-2,2,2-trichloroethyl ether and allyl 2,2-dichloro-1-phenyl ether, respectively, followed by reductive removal of remaining chlorine atoms.[15]

In view of the bulky 3-substituent,[41] the preferential formation of *trans* 3,4-disubstituted tetrahydrofurans **11** and **12** is not difficult to understand. It is more difficult to rationalize the higher selectivity for the methyl than for the isopropyl compound. Similarly, it is noteworthy that the diastereoselectivity in the reductive cyclization is significantly lower for O-prenylated (compounds **2c**, **3m′**, **3o′**, and **3p′**) than for O-allylated (compounds **2b**, **3m**, **3o**, and **3p**) phenyl β-hydroxyalkyl selenides.

Due to inversion of configuration at the radical center prior to cyclization, the bicyclic tetrahydrofuran derivatives **6j**, **6k**, **7j**, **9q, 10q**, and **18** are all *cis* fused. Compounds **6j**, **6k**, and **9q** were isolated predominantly as *exo* isomers. This stereochemistry would result if cyclization occurs *via* a chairlike transition state **21** with a pseudoaxial substituent at position 1 and a pseudoequatorial substituent at position 2 of the 3-oxa-5-hexenyl radical. The high preference for formation of the *endo* isomer of compound **18** may be ascribed to the anomeric effect. In the chairlike transition state **22**, the



21



22

oxygen 2-substituent of the 3-oxa-5-hexenyl radical is occupying a pseudoaxial position. This arrangement would allow for interaction between the lone pair electrons of the 3-oxa substituent and the σ^* orbital associated with the bond to the 2-substituent. As compared to the AIBN-initiated reaction (*exo*/*endo* = 1/4), the observed increased diastereoselectivity at 80 °C in the triethylborane-initiated reaction (*exo*/*endo* = 1/8) may result from an amplified anomeric effect due to coordination of the electron deficient boron to the pseudoaxial oxygen.

Conclusions and Outlook

In the present paper, we have demonstrated the use of radical-based methodology for the construction of a variety of substituted, functionalized (tellurides), or unfunctionalized tetrahydrofuran derivatives from readily available epoxides. Although simple looking, the tellurolate/selenolate ring-opening-, O-allylation/propargylation-, and group transfer cyclization/reductive radical cyclization sequence has, to the best of our knowledge, not been previously applied for the preparation of tetrahydrofuran derivatives. In most cases, the diastereoselectivity in the radical cyclization to an alkene was only moderate, in favor of the functionalized (compounds **6**) or unfunctionalized (compounds **9**) *trans*-2,4-disubstituted tetrahydrofuran. However, in some noteworthy cases (*cf.* compounds **6g**, **6i**, **9n**, **18**) a significantly higher selectivity was obtained. Although the reasons for the improved selectivity are not yet fully understood, it may be possible to improve it further by the proper choice of substituents and reaction conditions.

Since a handle for further manipulation is retained in the molecule, the group transfer products **6** and **7** are synthetically more valuable than their reductive radical cyclization counterparts **9** and **10**. For instance, the organotellurium compounds are likely to undergo lithium−tellurium exchange on treatment with alkyllithium reagents.[43] Also, vinylic tellurides have recently been shown to be convertible into vinylic cyanocuprates,[44,45] or, depending on the conditions, to undergo cross-coupling reactions with cyanocuprates.[45,46] 4-Methylenetetrahydrofurans **10** are readily oxidzed in the 5-position[21,47] and would therefore be useful for the preparation of α-methylene-γ-lactones. Last but not least, it is important to emphasize that the synthetic sequence leading to tetrahydrofuran derivatives **6**, **7**, **9**, and **10** is also well suited for asymmetric synthesis. The required, enantiomerically pure, epoxides are nowadays readily available by Sharpless epoxidation. Work in this direction is already ongoing in our laboratories.

Experimental Section

All epoxides used were commercially available except for 3-(benzyloxy)-1, 2-epoxypropane which was prepared according to a published procedure.[48] Bis[4-(trifluoromethyl)phenyl]-ditelluride,[49] diphenyl ditelluride,[49] bis[4-(dimethylamino)-phenyl]ditelluride,[49] and bis(2-thienyl)ditelluride[50] were prepared according to literature methods. Benzene was freshly distilled from calcium hydride. Unless otherwise stated, all 1D and 2D NMR spectra were recorded in $CDCl_3$ on a 400 MHz NMR spectrometer operating at 399.951 MHz (^{1}H NMR) and 100.578 MHz (^{13}C NMR). *J* values are given in Hz using the following abbreviations: s (singlet), br s (broad singlet), t (triplet), m (multiplet), q (quartet), quint (quintet). EI mass spectra were recorded at 70 eV. M^+-ions were given for ^{130}Te and ^{80}Se. Elemental analyses were performed by Analytical Laboratories, Lindlar, Germany, and the Research Institute for Pharmacy and Biochemistry, Prague, Czech Republic.

Typical Procedure for the Preparation of Aryl 2-Hydroxyalkyl Tellurides and Selenides. Preparation of 3-(Benzyloxy)-2-hydroxypropyl Phenyl Selenide (1p). Sodium borohydride (0.49 g, 13 mmol) was added, in portions, to a stirred suspension of diphenyl diselenide (2.03 g, 6.5 mmol) in absolute ethanol (50 mL) till a clear colorless and homogenous solution was obtained. 3-(Benzyloxy)-1,2-epoxypropane (2.13 g, 13 mmol) was then added, and the resultant light yellow solution was stirred for 1 h. Water (50 mL) was added, and the mixture was extracted with diethyl ether (3 × 30 mL), the combined organic phases were washed with water and later with saturated sodium chloride and dried (Na_2SO_4), and

(43) For the preparation of organolithium compounds *via* lithium−tellurium exchange, see Engman, L.; Stern, D. *Organometallics* **1993**, *12*, 1445, references 12 through 18.
(44) Marino, J. P.; Tucci, F.; Comasseto, J. V. *Synlett* **1993**, 761.
(45) Chieffi, A.; Comasseto, J. V. *Tetrahedron Lett.* **1994**, *35*, 4063.
(46) Chieffi, A.; Comasseto, J. V. *Synlett* **1995**, 671.
(47) Dauben, W. G.; Lorber, M.; Fullerton, D. S. *J. Org. Chem.* **1969**, *34*, 3587. Ratcliffe, R.; Rodehorst, R. *J. Org. Chem.* **1970**, *35*, 4000.
(48) Mouzin, G.; Cousse, H.; Rieu, J.-P.; Duflos, A. *Synthesis* **1983**, 117.
(49) Engman, L.; Persson, J. *J. Organomet. Chem.* **1990**, *388*, 71.
(50) Engman, L.; Cava, M. P. *Organometallics* **1982**, *1*, 470.

the solvent was removed *in vacuo*. The residue was purified by flash chromatography (40% ether/pentane) to give 3.76 g (90%) of the title compound. ^{1}H NMR δ 2.68 (d, J = 4.7 Hz, 1H), 3.03 (dd, J = 12.7, 6.9 Hz, 1H), 3.09 (dd, J = 12.7, 5.7 Hz, 1H), 3.51 (dd, J = 9.5, 5.9 Hz, 1H), 3.56 (dd, 9.5, 4.1 Hz, 1H), 3.93 (m, 1H), 4.49 (s, 2H), 7.22−7.37 (several peaks, 8H), 7.50 (m, 2H). ^{13}C NMR δ 31.91, 69.43, 72.83, 73.36, 127.13, 127.70, 127.76, 128.40, 129.13, 129.56, 132.77, 137.76. Anal. Calcd for $C_{16}H_{18}O_2Se$: C, 59.82; H, 5.65. Found: C, 59.52; H, 5.64. MS m/z (relative intensity) 322 (M^+, 4.9).

The following compounds were similarly prepared. As judged by ^{1}H NMR spectroscopy, the purity of the materials were > 98%. For yields see Tables 1 and 2.

2-Hydroxy-5-hexenyl phenyl telluride (1a): ^{1}H NMR δ 1.58−1.69 (several peaks, 2H), 2.05−2.24 (several peaks, 2H), 2.28 (m, 1H), 2.98 (dd, J = 12.3, 7.9 Hz, 1H), 3.15 (dd, J = 12.3, 4.2 Hz, 1H), 3.74 (m, 1H), 4.95 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 5.00 (ddt, J = 17.1, 1.9, 1.6 Hz, 1H), 5.78 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 7.16−7.22 (several peaks, 3H), 7.74 (m, 2H). ^{13}C NMR δ 20.1, 30.2, 36.8, 70.6, 111.2, 114.9, 127.8, 129.2, 138.0, 138.4. MS m/z (relative intensity) 306 (M^+, 7.5).

2-Hydroxy-5-hexenyl 4-(dimethylamino)phenyl telluride (1b): ^{1}H NMR δ 1.58−1.65 (several peaks, 2H), 2.03−2.23 (several peaks, 2H), 2.30 (d, J = 4.3 Hz, 1H), 2.82 (dd, J = 12.2, 8.1 Hz, 1H), 2.94 (s, 6H), 3.02 (dd, J = 12.2, 4.0 Hz, 1H), 3.68 (m, 1H), 4.93 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 4.99 (dq, J = 17.2, 1.7 Hz, 1H), 5.78 (ddt, J = 16.9, 10.2, 6.6 Hz, 1H), 6.55 (m, 2H), 7.64 (m, 2H). ^{13}C NMR δ 20.3, 30.2, 36.7, 40.1, 70.5, 94.0, 113.4, 114.7, 138.2, 141.1, 150.4. MS m/z (relative intensity) 349 (M^+, 21.7).

2-Hydroxy-5-hexenyl 4-(trifluoromethyl)phenyl telluride (1c): ^{1}H NMR δ 1.64−1.70 (several peaks, 2H), 2.09−2.27 (several peaks, 3H), 3.06 (dd, J = 12.3, 7.9Hz, 1H), 3.20 (dd, J = 12.3, 4.1 Hz, 1H), 3.80 (br s, 1H), 4.97 (dm, J = 10.2 Hz, 1H), 5.02 (dq, J = 17.1, 1.8 Hz, 1H), 5.80 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 7.43 (m, 2H), 7.81 (m, 2H). ^{13}C NMR δ 20.2, 30.2, 37.1, 70.7, 115.2, 117.0, 125.4, 125.7 (q), 130.0, 137.7, 137.8. MS m/z (relative intensity) 374 (M^+, 18.3).

2-Hydroxy-5-hexenyl 2-thienyl telluride (1d): ^{1}H NMR δ 1.61−1.68 (several peaks, 2H), 2.05−2.23 (several peaks, 3H), 2.89 (dd, J = 12.2, 8.1 Hz, 1H), 3.04 (dd, J = 12.1, 4.1 Hz, 1H), 3.78 (m, 1H), 4.96 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 5.01 (dq, J = 17.1, 1.6 Hz, 1H), 5.79 (ddt, J = 17.1, 10.3, 6.7 Hz, 1H), 6.93 (dd, J = 5.2, 3.5 Hz, 1H), 7.39 (dd, J = 3.5, 1.1 Hz, 1H), 7.43 (dd, J = 5.2, 1.1 Hz, 1H). ^{13}C NMR δ 22.4, 30.2, 36.7, 70.6, 97.0, 115.0, 128.9, 134.3, 138.0, 141.4. MS m/z (relative intensity) 312 (M^+, 95.7).

2-Hydroxybutyl 4-(trifluoromethyl)phenyl telluride (1e): ^{1}H NMR δ 0.96 (t, J = 7.5 Hz, 3H), 1.54−1.67 (several peaks, 2H), 2.21 (m, 1H), 3.07 (dd, J = 12.2, 7.8 Hz, 1H), 3.21 (dd, J = 12.2, 4.0 Hz, 1H), 3.70 (m, 1H), 7.42 (m,2H), 7.81 (m, 2H). ^{13}C NMR δ 10.2, 19.9, 31.0, 72.5, 117.1, 122.7, 125.6 (q), 129.6 (q), 137.6. MS m/z (relative intensity) 348 (M^+, 17.9).

2-Hydroxy-2-phenylethyl 4-(trifluoromethyl)phenyl telluride (1f): ^{1}H NMR δ 2.50 (d, J = 3.4 Hz, 1H), 3.33 (dd, J = 12.2, 5.7 Hz, 1H), 3.36 (dd, J = 12.2, 7.4 Hz, 1H), 4.95 (ddd, J = 7.2, 5.7, 3.3 Hz, 1H), 7.25−7.41 (several peaks, 7H), 7.73 (m, 2H). ^{13}C NMR δ 20.8, 73.8, 117.2, 125.6 (q), 128.1, 128.6, 137.7, 143.3. MS m/z (relative intensity) 396 (M^+, 15.3).

3-(Allyloxy)-2-hydroxypropyl 4-(trifluoromethyl)phenyl telluride (1g): ^{1}H NMR δ 2.75 (d, J = 3.7 Hz, 1H), 3.11 (dd, J = 12.1, 5.8 Hz, 1H), 3.16 (dd, J = 12.1, 6.6 Hz, 1H), 3.45 (dd, J = 9.5, 6.2 Hz, 1H), 3.53 (dd, J = 9.5, 3.9 Hz, 1H), 3.97 (dt, J = 5.7, 1.4 Hz, 2H), 3.99 (m, 1H), 5.19 (dq, J = 10.4, 1.4 Hz, 1H), 5.25 (dq, J = 17.2, 1.6 Hz, 1H), 5.87 (ddt, J = 17.2, 10.4, 5.7 Hz, 1H), 7.41 (m, 2H), 7.81 (m, 2H). ^{13}C NMR δ 14.0, 70.2, 72.3, 73.9, 117.5, 117.7, 122.7, 125.6 (q), 129.5, 134.2, 137.5. MS m/z (relative intensity) 390 (M^+, 31.6).

2-Hydroxy-3-phenoxypropyl 4-(trifluoromethyl)phenyl telluride (1h): ^{1}H NMR δ 2.67 (d, J = 5.0 Hz, 1H), 3.25 (d, J = 6.2 Hz, 2H), 4.00 (dd, J = 9.3, 5.9 Hz, 1H), 4.04 (dd, J = 9.3, 4.3 Hz, 1H), 4.22 (m, 1H), 6.82−7.00 (several peaks, 3H), 7.24−7.41 (several peaks, 4H), 7.81 (m, 2H). ^{13}C NMR δ 14.0, 70.0, 71.4, 114.4, 114.5, 121.4, 125.7 (q), 129.5, 137.7, 158.1. MS m/z (relative intensity) 426 (M^+, 15.3).

3-(Benzyloxy)-2-hydroxypropyl 4-(trifluoromethyl)-phenyl telluride (1i): ^{1}H NMR δ 2.73 (d, J = 4.1 Hz, 1H), 3.10 (dd, J = 12.1, 5.8 Hz, 1H), 3.15 (dd, J = 12.0, 6.5 Hz, 1H), 3.49 (dd, J = 9.5, 6.2 Hz, 1H), 3.56 (dd, J = 9.5, 3.9 Hz, 1H), 4.02 (m, 1H), 4.50 (s, 2H), 7.27−7.42 (several peaks, 7H), 7.78 (m, 2H). ^{13}C NMR δ 14.1, 70.2, 73.4, 73.9, 117.7, 125.4, 125.6 (q), 127.7, 127.9, 128.5, 129.6 (q), 137.5, 137.5. MS m/z (relative intensity) 440 (M^+, 2.3).

2-Hydroxycyclohexyl 4-(trifluoromethyl)phenyl telluride (1j): ^{1}H NMR δ 1.17−1.38 (several peaks, 3H), 1.50−1.65 (several peaks, 2H), 1.80 (m, 1H), 2.16 (m, 1H), 2.30 (m, 1H), 2.49 (d, J = 3.1 Hz, 1H), 3.19 (ddd, J = 12.3, 10.2, 3.7 Hz, 1H), 3.47 (m, 1H), 7.44 (m, 2H), 7.92 (m, 2H). ^{13}C NMR δ 24.7, 27.9, 34.7, 35.2, 40.3, 74.1, 115.2, 122.7, 125.6 (q), 130.3 (q), 140.4. MS m/z (relative intensity) 372 (M^+, 21.4).

***trans*-2-Hydroxycyclohexyl 2-Thienyl Telluride (1k).** Due to some decomposition at room temperature, this compound was directly O-allylated without purification.

***trans*-2-Hydroxy-5-hexenyl phenyl selenide (1l):** ^{1}H NMR δ 1.59−1.67 (several peaks, 2H), 2.06−2.27 (several peaks, 2H), 2.42 (m, 1H), 2.89 (dd, J = 12.7, 8.5 Hz, 1H), 3.14 (dd, J = 12.7, 3.6 Hz, 1H), 3.70 (m, 1H), 4.95 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 5.01 (ddt, J = 17.1, 2.0, 1.6, Hz, 1H), 5.79 (ddt, J = 17.0, 10.3, 6.6 Hz, 1H), 7.24−7.29 (several peaks, 3H), 7.52 (m, 2H). ^{13}C NMR δ 30.0, 35.6, 37.1, 69.3, 114.9, 127.2, 129.2, 129.3, 132.9, 138.0. MS m/z (relative intensity) 256 (M^+, 16.4).

2-Hydroxy-2-phenylethyl Phenyl Selenide (1m). ^{1}H NMR data were in good agreement with literature data.[19]

3-(Allyloxy)-2-hydroxypropyl phenyl selenide (1n): ^{1}H NMR δ 2.66 (d, J = 4.5 Hz, 1H), 3.03 (dd, J = 12.7, 7.0 Hz, 1H), 3.10 (dd, J = 12.8, 5.7 Hz, 1H), 3.47 (dd, J = 9.6, 5.9 Hz, 1H), 3.53 (dd, J = 9.6, 4.0 Hz, 1H), 3.92 (m, 1H), 3.96 (dt, J = 5.6, 1.4 Hz, 2H), 5.18 (dddd, J = 10.4, 1.7, 1.2, 1.2 Hz, 1H), 5.25 (dq, J = 17.2, 1.6 Hz, 1H), 5.87 (ddt, J = 17.2, 10.4, 5.6 Hz, 1H), 7.23−7.29 (several peaks, 3H), 7.53 (m, 2H). ^{13}C NMR δ 31.9, 69.4, 72.3, 72.8, 117.3, 127.2, 129., 129.6, 132.8, 134.3. MS m/z (relative intensity) 272 (M^+, 18.9).

2-Hydroxy-3-phenoxypropyl phenyl selenide (1o): ^{1}H NMR δ 2.71 (d, J = 4.8 Hz, 1H), 3.13 (dd, J = 12.8, 6.8 Hz, 1H), 3.22 (dd, J = 12.8, 5.6 Hz, 1H), 4.01 (dd, J = 9.4, 5.8Hz, 1H), 4.04 (dd, J = 9.3, 4.2 Hz, 1H), 4.13 (m, 1H), 6.84−6.99 (several peaks, 3H), 7.22−7.30 (several peaks, 5H), 7.54 (m, 2H). ^{13}C NMR δ 31.9, 69.1, 70.4, 114.5, 121.2, 127.3, 129.2, 129.5, 132.9, 158.3. MS m/z (relative intensity) 308 (M^+, 18.0).

***trans*-2-Hydroxycyclohexyl Phenyl Selenide (1q).** ^{1}H NMR data were in good agreement with literature data.[19]

2-Hydroxy-1-phenylethyl Phenyl Selenide (2a). ^{1}H NMR data were in good agreement with literature data.[19]

Typical Procedure for O-Allylation of Aryl *β*-Hydroxyalkyl Tellurides and Selenides (1). Preparation of 2,3-Bis(allyloxy)propyl 4-(Trifluoromethyl)phenyl Telluride (3g). To a solution of 3-(allyloxy)-2-hydroxypropyl 4-(trifluoromethyl)phenyl telluride (**1g**) (0.40 g, 1.0 mmol) in dry THF (20 mL) at room temperature was added sodium hydride (0.040 g, 60% dispersion, 1.0 mmol). The resulting mixture was stirred for ca. 1 h at room temperature. Allyl bromide (0.12 g, 0.089 mL, 1.0 mmol) was then added dropwise, and the reaction flask was placed in an oil bath preheated to 80 °C. The mixture was refluxed for 1 h and cooled to room temperature. Water (50 mL) was added, and the solution was extracted with ether (3 × 30 mL). The combined organic phase was washed twice with water and with saturated brine and then dried over Na_2SO_4. The solvent was removed *in vacuo* and the residue was purified by flash chromatography (5% ether/pentane) to afford 0.32 g (73%) of the title compound as a light yellow oil. ^{1}H NMR δ 3.19 (dd, J = 12.0, 6.1 Hz, 1H), 3.26 (dd, J = 11.9, 5.9 Hz, 1H), 3.51 (dd, J = 9.9, 5.9 Hz, 1H), 3.61 (dd, J = 9.8, 4.9 Hz, 1H), 3.80 (ddd, J = 11.8, 5.9, 4.8 Hz, 1H), 3.97 (dt, J = 5.7, 1.5 Hz, 2H), 4.06 (ddt, J = 12.7, 7.0, 1.5 Hz, 1H), 4.11 (ddt, J = 12.7, 5.7, 1.4 Hz, 1H), 5.16 (ddt, J = 10.2, 1.8, 1.3 Hz, 1H), 5.18 (ddt, J = 10.3, 1.8, 1.2 Hz, 1H), 5.24 (dq, J = 17.2, 1.7 Hz, 1H), 5.26 (dq, J = 17.2, 1.7 Hz, 1H), 5.87 (ddt, J = 17.2, 10.4, 5.6 Hz, 1H), 5.88 (ddt, J = 17.3, 10.4, 5.6 Hz, 1H), 7.40 (m, 2H), 7.80 (m, 2H). ^{13}C NMR δ 12.4, 71.0, 72.3, 72.4, 77.5, 117.2, 118.4, 122.8, 125.5 (q), 129.3,

134.4, 134.6, 137.3. Anal. Calcd for $C_{16}H_{19}F_3O_2Te$: C, 44.91; H, 4.48. Found: C, 45.37; H, 4.50.

The following compounds were similarly prepared. As judged by ^{1}H NMR spectroscopy, the purity of the materials were >98%. For yields see Tables 1 and 2.

2-(Allyloxy)-1-phenylethyl phenyl selenide (2b): ^{1}H NMR δ 3.86 (dd, J = 10.3, 6.1 Hz, 1H), 3.95 (m, 2H), 3.98 (dd, J = 10.2, 8.3 Hz, 1H), 4.49 (dd, J = 8.3, 6.0 Hz, 1H), 5.12 (dddd, J = 10.3, 1.7, 1.2, 1.2 Hz, 1H), 5.18 (dddd, J = 17.2, 1.7, 1.5, 1.5 Hz, 1H), 5.81 (ddt, J = 17.2, 10.3, 5.5 Hz, 1H), 7.16−7.28 (several peaks, 8H), 7.44 (m, 2H). ^{13}C NMR δ 47.4, 72.0, 72.9, 117.2, 127.2, 127.8, 128.0, 128.4, 128.8, 129.1, 134.5, 135.2.

2-[(3-Methyl-2-butenyl)oxy]-1-phenylethyl phenyl selenide (2c): ^{1}H NMR δ 1.57 (m, 3H), 1.69 (m, 3H), 3.84 (dd, J = 10.3, 6.1 Hz, 1H), 3.93 (m, 2H), 3.96 (dd, J = 10.4, 8.5 Hz, 1H), 4.49 (dd, J = 8.4, 6.1 Hz, 1H), 5.25 (tq, J = 7.0, 1.4 Hz, 1H), 7.16−7.27 (several peaks, 8H), 7.43 (m, 2H). ^{13}C NMR δ 17.9, 25.7, 47.4, 67.3, 72.5, 120.9, 127.1, 128.0, 128.3, 128.8, 129.1, 135.2, 137.1, 140.0.

2-(Allyloxy)-5-hexenyl 2-thienyl telluride (3d): ^{1}H NMR δ 1.73(m, 2H), 2.11(m, 2H), 3.03(dd, J = 11.8, 6.0 Hz, 1H), 3.07 (dd, J = 11.8, 5.4 Hz, 1H), 3.55 (m, 1H), 3.92 (m, 2H), 4.00 (m, 2H), 4.96 (dm, J = 1.2 Hz, 1H), 5.01 (dm, J = 17.2 Hz, 1H), 5.15 (dm, J = 10.2 Hz, 1H), 5.23 (dq, J = 17.2, 1.6 Hz, 1H), 5.84 (m, 2H), 6.93 (dd, J = 5.1, 3.4 Hz, 1H), 7.38 (dd, J = 3.4, 1.1 Hz, 1H), 7.42 (dd, J = 5.1, 1.1 Hz, 1H). ^{13}C NMR 17.8, 29.6, 34.4, 70.1, 78.2, 97.8, 114.8, 116.9, 128.8, 134.1, 134.9, 138.1, 141.2.

2-(Allyloxy)butyl 4-(trifluoromethyl)phenyl telluride (3e): ^{1}H NMR δ 0.93 (t, J = 7.4 Hz, 3H), 1.57−1.76 (several peaks, 2H), 3.17 (dd, J = 11.7, 5.7 Hz, 1H), 3.21 (dd, J = 11.7, 6.0 Hz, 1H), 3.53 (dt, J = 11.8, 5.9 Hz, 1H), 4.00 (ddd, J = 5.6, 1.6, 1.3 Hz, 2H), 5.15 (ddt, J = 10.3, 1.8, 1.3 Hz, 1H), 5.24 (dq, J = 17.2, 1.7 Hz, 1H), 5.89 (ddt, J = 17.2, 10.4, 5.6 Hz, 1H), 7.41 (m, 2H), 7.80 (m, 2H). ^{13}C NMR δ 9.6, 15.2, 28.1, 70.2, 80.0, 116.9, 118.0, 122.8, 125.5 (q), 129.5 (q), 134.8, 137.5.

2-(Allyloxy)-2-phenylethyl 4-(trifluoromethyl)phenyl telluride (3f): ^{1}H NMR δ 3.17 (dd, J = 11.7, 5.2 Hz, 1H), 3.49 (dd, J = 11.7, 8.7 Hz, 1H), 3.79 (ddt, J = 12.7, 6.0 Hz, 1H), 3.94 (dddd, J = 12.7, 5.1, 1.7, 1.4 Hz, 1H), 4.66 (dd, J = 8.8, 5.1 Hz, 1H), 5.16 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.23 (dq, J = 17.2, 1.7 Hz, 1H), 5.88 (dddd, J = 17.3, 10.5, 6.1, 5.0 Hz, 1H), 7.27−7.38 (several peaks, 7H), 7.71 (m, 2H). ^{13}C NMR δ 18.1, 69.9, 81.6, 117.1, 118.4, 122.8, 125.4 (q), 126.4, 128.1, 128.6, 134.4, 137.4, 141.7.

2-(Allyloxy)-3-phenoxypropyl 4-(trifluoromethyl)phenyl telluride (3h): ^{1}H NMR δ 3.26 (dd, 12.3, 5.8 Hz, 1H), 3.37 (dd, J = 12.4, 5.6 Hz, 1H), 3.96−4.20 (several peaks, 5H), 5.18 (dq, J = 10.4, 1.4 Hz, 1H), 5.26 (dq, J = 17.2, 1.7 Hz, 1H), 5.89 (ddt, J = 17.1, 10.5, 5.6 Hz, 1H), 6.83−6.88 (several peaks, 2H), 6.94 (m, 1H), 7.26 (m, 2H), 7.78 (m, 2H). ^{13}C NMR δ 12.2, 69.9, 71.3, 77.3, 114.4, 114.6, 117.5, 121.0, 121.1, 125.6 (q), 129.4 (q), 134.4, 137.6, 158.3.

2-(Allyloxy)-3-(benzyloxy)propyl 4-(trifluoromethyl)-phenyl telluride (3i): ^{1}H NMR δ 3.19 (dd, J = 11.9, 6.0 Hz, 1H), 3.26 (dd, J = 11.9, 5.8 Hz, 1H), 3.55 (dd, J = 9.8, 5.8 Hz, 1H), 3.65 (dd, J = 9.8, 4.8 Hz, 1H), 3.82 (ddd, J = 11.7, 5.9, 4.8 Hz, 1H), 4.04 (ddt, J = 12.7, 5.7, 1.5 Hz, 1H), 4.09 (ddt, J = 12.6, 5.7, 1.6 Hz, 1H), 4.51 (s, 2H), 5.15 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.23 (dq, J = 17.2, 1.6 Hz, 1H), 5.87 (ddt, J = 17.1, 10.3, 5.6 Hz, 1H), 7.26−7.40 (several peaks, 7H), 7.78 (m, 2H). ^{13}C NMR δ 12.4, 71.0, 72.4, 73.4, 77.6, 117.2, 118.4, 125.5 (q), 127.6, 127.7, 128.4, 134.6, 137.3, 137.9.

***trans*-2-(Allyloxy)cyclohexyl 4-(trifluoromethyl)phenyl telluride (3j):** ^{1}H NMR δ 1.15−1.36 (several peaks, 3H), 1.51−1.63 (several peaks, 2H), 1.81 (m, 1H), 1.95 (m, 1H), 2.14 (m, 1H), 3.42 (m, 1H), 3.64 (ddd, J = 11.3, 9.7, 3.9 Hz, 1H), 3.94 (ddt, J = 12.5, 5.6, 1.5 Hz, 1H), 4.14 (ddt, J = 12.5, 5.6, 1.4 Hz, 1H), 5.16 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.27 (dq, J = 17.1, 1.7 Hz, 1H), 5.90 (ddt, J = 17.2, 10.3, 5.6 Hz, 1H), 7.41 (m, 2H), 7.91 (m, 2H). ^{13}C NMR δ 24.0, 27.6, 32.4, 33.3, 34.0, 69.7, 82.8, 116.8, 122.8, 125.3 (q), 125.5, 129.7 (q), 134.9, 140.4.

***trans*-2-(Allyloxy)cyclohexyl 2-thienyl telluride (3k):** ^{1}H NMR δ 1.09−1.32 (several peaks, 3H), 1.41−1.58 (several peaks, 2H), 1.73−1.83 (several peaks, 2H), 2.14 (m, 1H), 3.37 (m, 1H), 3.49 (ddd, J = 11.7, 10.0, 3.9 Hz, 1H), 3.95 (ddt, J = 12.6, 5.6, 1.6 Hz, 1H), 4.15 (ddt, J = 12.6, 5.6, 1.4 Hz, 1H), 5.18 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.30 (dq, J = 17.2, 1.7 Hz, 1H), 5.95 (ddt, J = 17.2, 10.3, 5.6 Hz, 1H), 6.94 (dd, J = 5.1, 3.4 Hz, 1H), 7.36 (dd, J = 3.4, 1.1 Hz, 1H), 7.43 (dd, J = 5.2, 1.1 Hz, 1H). ^{13}C NMR δ 24.2, 27.6, 32.6, 33.4, 69.7, 82.9, 97.3, 116.8, 128.9, 134.7, 134.9, 142.8.

2-(Allyloxy)-5-hexenyl phenyl selenide (3l): ^{1}H NMR δ 1.64−1.81 (several peaks, 2H), 2.04−2.22 (several peaks, 2H), 2.99 (dd, J = 12.3, 6.6 Hz, 1H), 3.11 (dd, J = 12.3, 5.2 Hz, 1H), 3.53 (ddt, J = 7.1, 6.7, 5.0 Hz, 1H), 3.92 (ddt, J = 12.4, 5.7, 1.3 Hz, 1H), 4.04 (ddt, J = 12.5, 5.6, 1.3 Hz, 1H), 4.95 (ddt, J = 10.1, 2.0, 1.3 Hz, 1H), 5.00 (ddt, J = 17.1, 1.9, 1.6 Hz, 1H), 5.13 (ddt, J = 10.3, 1.8, 1.2 Hz, 1H), 5.21 (dq, J = 17.1, 1.6 Hz, 1H), 5.78 (ddt, J = 17.0, 10.5, 6.5 Hz, 1H), 5.88 (ddt, J = 17.2, 10.3, 5.7 Hz, 1H), 7.22−7.28 (several peaks, 3H), 7.51 (m, 2H). ^{13}C NMR δ 29.5, 32.2, 33.5, 70.4, 77.8, 114.8, 116.9, 126.8, 129.0, 130.6, 132.7, 134.9, 138.2.

2-(Allyloxy)-2-phenylethyl phenyl selenide (3m): ^{1}H NMR δ 3.11 (dd, J = 12.3, 5.2 Hz, 1H), 3.36 (dd, J = 12.2, 8.3 Hz, 1H), 3.79 (ddt, J = 12.7, 6.0, 1.3 Hz, 1H), 3.94 (dddd, J = 12.7, 5.1, 1.7, 1.4 Hz, 1H), 4.53 (dd, J = 8.3, 5.2 Hz, 1H), 5.15 (ddt, J = 10.1, 1.9, 1.3 Hz, 1H), 5.23 (dq, J = 17.2, 1.7 Hz, 1H), 5.88 (dddd, J = 17.2, 10.3, 6.1, 5.0 Hz, 1H), 7.19−7.36 (several peaks, 8H), 7.46 (m, 2H). ^{13}C NMR δ 35.4, 69.8, 80.7, 117.0, 118.7, 126.6, 128.0, 128.5, 128.9, 130.8, 132.5, 134.5, 141.1.

2-[(3-Methyl-2-butenyl)oxy]-2-phenylethyl phenyl selenide (3m′): ^{1}H NMR δ 1.53 (m, 3H), 1.71 (m, 3H), 3.09 (dd, J = 12.2, 5.3 Hz, 1H), 3.34 (dd, J = 12.2, 8.3 Hz, 1H), 3.79 (m, 1H), 3.88 (m, 1H), 4.49 (dd, J = 8.3, 5.3 Hz, 1H), 5.35 (m, 1H), 7.20−7.36 (several peaks, 8H), 7.47 (m, 2H). ^{13}C NMR δ 18.0, 25.8, 35.5, 65.4, 80.4, 120.9, 126.6, 126.7, 127.9, 128.4, 128.9, 130.8, 132.5, 137.4, 141.4.

2,3-Bis(allyloxy)propyl phenyl selenide (3n): ^{1}H NMR δ 3.09 (dd, J = 12.6, 5.8 Hz, 1H), 3.15 (dd, J = 12.7, 6.3 Hz, 1H), 3.58 (d, J = 4.9 Hz, 2H), 3.72 (m, 1H), 3.96 (m, 2H), 4.07 (dt, J = 5.7, 1.5 Hz, 2H), 5.12−5.19 (several peaks, 2H), 5.22 (dq, J = 17.2, 1.6 Hz, 1H), 5.25 (dq, J = 17.2, 1.6 Hz, 1H), 5.82−5.94 (several peaks, 2H), 7.20−7.28 (several peaks, 3H), 7.52 (m, 2H). ^{13}C NMR δ 29.2, 71.0, 71.2, 72.2, 77.3, 116.9, 117.1, 126.7, 128.9, 130.5, 132.4, 134.5, 134.7.

2-(Allyloxy)-3-phenoxypropyl phenyl selenide (3o): ^{1}H NMR δ 3.18 (dd, J = 12.8, 5.8 Hz, 1H), 3.24 (dd, J = 12.7, 6.4 H, 1H), 3.91 (ddt, J = 6.3, 5.8, 4.9 Hz, 1H), 4.09−4.14 (several peaks, 4H), 5.15 (ddt, J = 10.3, 1.8, 1.2 Hz, 1H), 5.23 (dq, J = 17.2, 1.7 Hz, 1H), 5.89 (ddt, J = 17.1, 10.3, 5.7 Hz, 1H), 6.85−6.98 (several peaks, 3H), 7.20−7.29 (several peaks, 5H), 7.52 (m, 2H). ^{13}C NMR δ 29.3, 69.0, 71.3, 76.8, 114.6, 117.4, 120.9, 126.9, 129.1, 129.39, 129.43, 132.6, 134.6, 158.5.

2-[(3-Methyl-2-butenyl)oxy]-3-phenoxypropyl phenyl selenide (3o′): ^{1}H NMR δ 1.61 (m, 3H), 1.72 (m, 3H), 3.17 (dd, J = 12.7, 5.9 Hz, 1H), 3.23 (dd, J = 12.7, 6.2 Hz, 1H), 3.88 (m, 1H), 4.10 (d, J = 5.1 Hz, 2H), 4.12 (m, 2H), 5.33 (dddt, J = 9.8, 7.3, 3.0, 1.2 Hz, 1H), 6.85−6.97 (several peaks, 3H), 7.20−7.29 (several peaks, 5H), 7.52 (m, 2H). ^{13}C NMR δ 18.0, 25.8, 29.4, 66.8, 69.1, 76.3, 114.5, 120.8, 120.9, 126.8, 129.0, 129.3, 129.4, 132.5, 137.5, 158.6.

2-(Allyloxy)-3-(benzyloxy)propyl phenyl selenide (3p): ^{1}H NMR δ 3.10 (dd, J = 12.6, 5.8 Hz, 1H), 3.15 (dd, J = 12.5, 6.1 Hz, 1H), 3.60 (dd, J = 10.0, 5.1 Hz, 1H), 3.63 (dd, J = 10.0, 4.7 Hz, 1H), 3.74 (m, 1H), 4.06 (dt, J = 5.7, 1.6 Hz, 2H), 4.50 (s, 2H), 5.13 (dddd, J = 10.3, 1.7, 1.2, 1.2 Hz, 1H), 5.21 (dq, J = 17.2, 1.6 Hz, 1H), 5.87 (ddt, J = 17.1, 10.3, 5.6 Hz, 1H), 7.19−7.36 (several peaks, 8H), 7.50 (m, 2H). ^{13}C NMR δ 29.4, 71.1, 71.3, 73.3, 77.5, 117.1, 126.8, 127.5, 127.6, 128.3, 129.0, 130.6, 132.5, 134.8, 138.1.

3-(Benzyloxy)-2-[(3-methyl-2-butenyl)oxy]propyl phenyl selenide (3p′): ^{1}H NMR δ 1.61 (m, 3H), 1.71 (m, 3H), 3.09 (dd, J = 12.5, 5.9 Hz, 1H), 3.16 (dd, J = 12.5, 6.0 Hz, 1H), 3.59 (dd, J = 9.9, 5.3 Hz, 1H), 3.62 (dd, J = 9.9, 4.7 Hz, 1H), 3.71 (m, 1H), 4.05 (m, 2H), 4.50 (s, 2H), 5.31 (dtt, J = 6.9, 2.8, 1.4 Hz, 1H), 7.18−7.35 (several peaks, 8H), 7.50 (m, 2H). ^{13}C NMR δ 18.0, 25.7, 29.6, 66.5, 71.5, 73.3, 77.1, 121.1, 126.7, 127.5, 127.6, 128.3, 129.0, 132.4, 137.0.

2-(Allyloxy)cyclohexyl phenyl selenide (3q): ^{1}H NMR δ 1.21−1.43 (m, 3H), 1.45−1.55 (m, 1H), 1.56−1.64 (m, 1H), 1.69−1.77 (m, 1H), 1.98−2.06 (m, 1H), 2.07−2.14 (m, 1H), 3.28−3.38 (m, 2H), 3.99 (ddt, J = 12.6, 5.6, 1.5 Hz, 1H), 4.10 (ddt, J = 12.6, 5.7, 1.5z, 1H), 5.15 (ddt, J = 10.4, 1.9, 1.3 Hz, 1H), 5.27 (dq, J = 17.2, 1.7 Hz, 1H), 5.92 (ddt, J = 17.2, 10.3, 5.6 Hz, 1H), 7.22−7.27 (m, 3H), 7.56−7.60 (m, 2H). ^{13}C NMR δ 23.5, 25.6, 31.1, 32.0, 47.5, 70.0, 80.4, 116.7, 127.2, 128.7, 129.2, 135.0, 135.2.

The following compounds were obtained by allyloxyselenenation of vinylic ethers.

2-(Allyloxy)-2-ethoxyethyl Phenyl Selenide (14a). Except that triethylamine was used as a base instead of diisopropylamine, this compound was prepared according to a literature procedure[39] in 58% yield. ^{1}H NMR δ 1.19 (t, J = 7.1 Hz, 3H), 3.11 (dd, J = 12.5, 5.7 Hz, 1H), 3.14 (dd, J = 12.5, 5.7 Hz, 1H), 3.54 (dq, J = 9.4, 7.0 Hz, 1H), 3.66 (dq, J = 9.3, 7.0 Hz, 1H), 4.12 (ddt, J = 12.7, 5.4, 1.4 Hz, 1H), 4.14 (ddt, J = 12.7, 5.8, 1.4 Hz, 1H), 4.76 (t, J = 5.7 Hz, 1H), 5.16 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.27 (dq, J = 17.1, 1.6 Hz, 1H), 5.90 (dddd, J = 17.2, 10.4, 5.8, 5.5 Hz, 1H), 7.21−7.28 (several peaks, 3H), 7.52 (m, 2H). ^{13}C NMR δ 15.2, 30.9, 61.9, 67.1, 101.8, 117.0, 126.9, 129.0, 130.3, 132.6, 134.3. MS m/z (relative intensity) 286 (M^+, 5.5).

2-(Allyloxy)-2-*n*-butoxyethyl Phenyl Selenide (14b). Except that triethylamine was used as a base instead of diisopropylamine, this compound was prepared according to a literature procedure[39] in 74% yield. ^{1}H NMR δ 0.91 (t, J = 7.3 Hz, 3H), 1.31−1.42 (several peaks, 2H), 1.50−1.58 (several peaks, 2H), 3.10 (dd, J = 12.5, 5.6 Hz, 1H), 3.14 (dd, J = 12.5, 5.7 Hz, 1H), 3.46 (dt, J = 9.3, 6.5 Hz, 1H), 3.59 (dt, J = 9.2, 6.5 Hz, 1H), 4.03 (ddt, J = 12.8, 5.9, 1.5 Hz, 1H), 4.12 (ddt, J = 12.7, 5.4, 1.5 Hz, 1H), 4.76 (t, J = 5.7 Hz, 1H), 5.16 (ddt, J = 10.4, 1.7, 1.3 Hz, 1H), 5.27 (dq, J = 17.2, 1.7 Hz, 1H), 5.89 (ddt, J = 17.2, 10.4, 5.7 Hz, 1H), 7.19−7.27 (several peaks, 3H), 7.52 (m, 2H). ^{13}C NMR δ 13.8, 19.3, 30.9, 31.8, 66.1, 67.0, 101.8, 116.9, 126.8, 128.9, 130.4, 132.5, 134.4. MS m/z (relative intensity) 314 (M^+, 5.0).

2-(Allyloxy)-2-*tert*-butoxyethyl Phenyl Selenide (14c). Except that phenylselenenyl chloride instead of phenylselenenyl bromide was used as selenenating agent, and pyridine was used as a base instead of diisopropylamine, this compound was prepared according to a literature procedure[39] in 65% yield. ^{1}H NMR δ 1.12 (s, 9H), 3.08 (dd, J = 12.3, 6.1 Hz, 1H), 3.12 (dd, J = 12.3, 5.3 Hz, 1H), 4.02 (ddt, J = 12.6, 5.4, 1.5 Hz, 1H), 4.06 (ddt, J = 12.6, 5.5, 1.5 Hz, 1H), 4.99 (dd, J = 6.0, 5.3 Hz, 1H), 5.13 (ddt, J = 10.4, 1.8, 1.4 Hz, 1H), 5.26 (dq, J = 17.2, 1.8 H, 1H), 5.89 (ddt, J = 17.3, 10.4, 5.5 Hz, 1H), 7.18−7.26 (several peaks, 3H), 7.51 (several peaks, 2H). ^{13}C NMR δ 28.7, 32.4, 64.3, 74.7, 96.4, 116.4, 126.7, 128.9, 130.7, 132.4, 134.8. MS m/z (relative intensity) 314 (M^+, 1.0).

5-(Phenylseleno)-3,4-dihydro-2*H*-pyran (16) was isolated in 68% yield by treatment of 3,4-dihydro-2*H*-pyran with phenylselenenyl bromide, allyl alcohol, and triethylamine/diisopropylamine following the literature procedure for the preparation of compound **17**.[39] ^{1}H NMR δ 1.93−1.99 (several peaks, 2H), 2.24−2.28 (several peaks, 2H), 4.01−4.05 (several peaks, 2H), 6.94 (t, J = 1.7 Hz, 1H), 7.16−7.27 (several peaks, 3H), 7.39 (m, 2H). ^{13}C NMR δ 23.8, 27.2, 65.3, 101.3, 126.1, 129.0, 129.8, 131.6, 150.6. MS m/z (relative intensity) 240 (M^+, 84.6).

***trans*-2-(Allyloxy)-3-(phenylseleno)tetrahydropyran (17).** Except that phenylselenenyl chloride was used as a selenenating agent and triethylamine was used as a base instead of diisopropylamine, this compound was prepared according to a literature procedure[39] in 54% yield. ^{1}H NMR δ 1.53 (m, 1H), 1.73 (m, 1H), 1.83 (m, 1H), 2.24 (m, 1H), 3.33 (dt, J = 7.3, 4.4 Hz, 1H), 3.54 (m, 1H), 3.91 (m, 1H), 4.01 (ddt, J = 12.9, 6.1, 1.4 Hz, 1H), 4.25 (dddd, J = 12.9, 5.2, 1.4 Hz, 1H), 4.67 (d, J = 4.8 Hz, 1H), 5.16 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.28 (dq, J = 17.2, 1.7 Hz, 1H), 5.90 (dddd, J = 17.2, 10.4, 6.0, 5.2 Hz, 1H), 7.22−7.28 (several peaks, 3H), 7.57 (m, 2H). ^{13}C NMR δ 24.1, 27.2, 44.2, 62.7, 68.6, 101.1, 117.0, 127.5, 128.9, 129.0, 134.2, 134.6. MS m/z (relative intensity) 298 (M^+, 12.4).

Typical Procedure for O-Prop-2-ynylation of β-Hydroxyalkyl Aryl Tellurides and Selenides. Preparation of *trans*-2-(2-Propynyloxy)cyclohexyl Phenyl Selenide (4q). To a solution of 2-hydroxycyclohexyl phenyl selenide (1.77 g, 6.96 mmol) in dry THF (50 mL) was added sodium hydride (0.28 g, 60% dispersion, 6.96 mmol), and the resulting mixture was stirred for 2 h at room temperature. Propargyl bromide (80% in toluene, 0.77 mL, 6.96 mmol) was added dropwise and the mixture stirred for 6 h after which TLC showed the complete consumption of the starting material. Water (50 mL) was added and the solution extracted with ether (3 × 30 mL). The combined ether extracts were washed twice with water and with brine and dried over Na_2SO_4. The solvent was removed *in vacuo* and the residue on purification by flash chromatography (5% ether/pentane) afforded 1.55 g (77%) of the title compound. ^{1}H NMR δ 1.18−1.44 (several peaks, 3H), 1.51 (m, 1H), 1.60 (m, 1H), 1.73 (m, 1H), 2.04 (m, 1H), 2.16 (m, 1H), 2.40 (t, J = 2.4 Hz, 1H), 3.29 (ddd, J = 9.8, 8.3, 4.2 Hz, 1H), 3.54 (ddd, J = 8.5, 8.5, 3.7 Hz, 1H), 4.20 (dd, J = 15.8, 2.4 Hz, 1H), 4.27 (dd, J = 15.9, 2.5 Hz, 1H), 7.22−7.28 (several peaks, 3H), 7.60 (m, 2H). ^{13}C NMR δ 23.3, 25.4, 30.7, 31.9, 47.2, 56.2, 74.0, 79.9, 80.2, 127.3, 128.8, 129.1, 135.0. Anal. Calcd for $C_{15}H_{18}OSe$: C, 61.43; H, 6.19. Found: C, 61.56; H, 6.18.

The following compounds were similarly prepared. As judged by ^{1}H NMR spectroscopy, the purity of the materials were >98%. For yields see Tables 1 and 2.

2-(2-Propynyloxy)-5-hexenyl 4-(trifluoromethyl)phenyl telluride (4c): ^{1}H NMR δ 1.65−1.83 (several peaks, 2H), 2.10−2.18 (several peaks, 2H), 2.40 (t, J = 2.4 Hz, 1H), 3.17 (dd, J = 12.1, 5.6 Hz, 1H), 3.22 (dd, J = 12.0, 6.6 Hz, 1H), 3.77 (m, 1H), 4.17 (d, J = 2.4 Hz, 2H), 4.97 (dm, J = 10.2 Hz, 1H), 5.01 (dq, J = 17.0, 1.6 Hz, 1H), 5.79 (ddt, J = 17.0, 10.3, 6.6 Hz, 1H), 7.42 (m, 2H), 7.82 (m, 2H). ^{13}C NMR δ 14.7, 29.4, 34.5, 56.4, 74.5, 77.9, 79.8, 114.9, 115.1, 117.6, 125.6 (q), 128.3, 135.9 (q), 137.8.

2-(2-Propynyloxy)butyl 4-(trifluoromethyl)phenyl telluride (4e): ^{1}H NMR (300 MHz) δ 0.93 (t, J = 7.4 Hz, 3H), 1.50−1.74 (several peaks, 2H), 2.40 (t, J = 2.3 Hz, 1H), 3.18 (d, J = 5.8 Hz, 2H), 3.71 (m, 1H), 4.18 (d, J = 1.7 Hz, 2H), 7.41 (m, 2H), 7.81 (m, 2H). ^{13}C NMR δ 9.5, 14.4, 27.9, 56.4, 74.4, 79.7, 125.6, 137.7.

2-(2-Propynyloxy)-2-phenylethyl 4-(trifluoromethyl)-phenyl telluride (4f): ^{1}H NMR δ 2.35 (t, J = 2.4 Hz, 1H), 3.12 (dd, J = 11.8, 5.6 Hz, 1H), 3.41 (dd, J = 11.7, 8.3 Hz, 1H), 3.80 (dd, J = 15.8, 2.4 Hz, 1H), 4.07 (dd, J = 15.8, 2.4 Hz, 1H), 4.79 (dd, J = 8.3, 5.6 Hz, 1H), 7.15−7.35 (several peaks, 7H), 7.64 (m, 2H). ^{13}C NMR δ 17.2, 55.9, 74.7, 79.3, 80.9, 118.1, 125.4 (q), 125.7, 126.9, 128.4 (q), 128.7, 137.6, 140.3.

3-(Allyloxy)-2-(2-propynyloxy)propyl 4-(trifluoromethyl)phenyl telluride (4g): ^{1}H NMR δ 2.41 (t, J = 2.4 Hz, 1H), 3.19 (dd, J = 12.0, 5.8 Hz, 1H), 3.24 (dd, J = 12.2, 6.2 Hz, 1H), 3.55 (dd, J = 9.9, 5.2 Hz, 1H), 3.64 (dd, J = 9.9, 4.9 Hz, 1H), 3.96 (dt, J = 5.6, 1.6 Hz, 2H), 4.01 (m, 1H), 4.25 (dd, J = 17.2, 2.0 Hz, 1H), 4.29 (dd, J = 16.8, 2.0 Hz, 1H), 5.19 (dm, J = 10.4 Hz, 1H), 5.25 (dq, J = 17.2, 1.7 Hz, 1H), 5.87 (ddt, J = 17.3, 10.5, 5.7 Hz, 1H), 7.41 (m, 2H), 7.81 (m, 2H). ^{13}C NMR δ 11.4, 57.2, 72.2, 72.3, 74.7, 77.3, 79.7, 117.3, 118.2, 125.5 (q), 134.3, 136.0 (q), 137.4, 138.0.

3-Phenoxy-2-(2-propynyloxy)propyl 4-(trifluoromethyl)phenyl telluride (4h): ^{1}H NMR δ 2.44 (t, J = 2.4 Hz, 1H), 3.27 (dd, J = 12.5, 5.6 Hz, 1H), 3.36 (dd, J = 12.4, 6.0 Hz, 1H), 4.07 (dd, J = 9.5, 5.3 Hz, 1H), 4.13−4.23 (several peaks, 2H), 4.32 (dd, J = 2.4, 1.0 Hz, 1H), 4.47 (m, 1H), 6.82−6.98 (several peaks, 3H), 7.23−7.38 (several peaks, 4H), 7.79 (m, 2H). ^{13}C NMR δ 11.4, 57.4, 69.6, 74.9, 76.8, 79.5, 114.4, 114.5, 121.0, 121.2, 125.5 (q), 129.4, 137.6, 158.1.

3-(Benzyloxy)-2-(2-propynyloxy)propyl 4-(trifluoromethyl)phenyl telluride (4i): ^{1}H NMR δ 2.41 (t, J = 2.4 Hz, 1H), 3.19 (dd, J = 12.0, 5.8 Hz, 1H), 3.24 (dd, J = 12.1, 6.1 Hz, 1H), 3.59 (dd, J = 9.9, 5.3 Hz, 1H), 3.68 (dd, J = 10.0, 5.0 Hz, 1H), 4.02 (tt, J = 10.2, 6.0 Hz, 1H), 4.24 (dd, J = 15.9, 2.5 Hz, 1H), 4.29 (dd, J = 15.9, 2.4 Hz, 1H), 4.50 (s, 2H), 7.27−

7.41 (m, 7H), 7.77−7.82 (m, 2H). ^{13}C NMR δ 11.5, 57.2, 72.3, 73.5, 74.7, 77.4, 79.7, 118.2, 125.5, 127.7, 127.8, 128.4, 137.4, 137.8.

***trans*-2-(2-Propynyloxy)cyclohexyl 4-(trifluoromethyl)phenyl telluride (4j):** ^{1}H NMR δ 1.17−1.38 (several peaks, 3H), 1.51−1.67 (several peaks, 2H), 1.80 (m, 1H), 2.01 (m, 1H), 2.18 (m, 1H), 2.42 (t, J = 2.4 Hz, 1H), 3.55−3.65 (several peaks, 2H), 4.17 (dd, J = 15.8, 2.4 Hz, 1H), 4.26 (dd, J = 15.9, 2.4 Hz, 1H), 7.40−7.44 (m, 2H), 7.91−7.94 (m, 2H). ^{13}C NMR (300 MHz) δ 23.8, 27.5, 31.9, 32.8, 33.9, 55.7, 74.4, 81.9, 125.3 (q), 140.3.

2-(2-Propynyloxy)-5-hexenyl phenyl selenide (4l): ^{1}H NMR δ 1.66−1.81 (several peaks, 2H), 2.05−2.23 (several peaks, 2H), 2.38 (t, J = 2.4 Hz, 1H), 3.02 (dd, J = 12.4, 6.6 Hz, 1H), 3.12 (dd, J = 12.5, 5.0 Hz, 1H), 3.72 (ddt, J = 13.6, 6.9, 4.9 Hz, 1H), 4.14 (dd, J = 15.8, 2.4 Hz, 1H), 4.19 (dd, J = 15.8, 2.4 Hz, 1H), 4.95 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 5.01 (ddt, J = 17.1, 2.0, 1.6 Hz, 1H), 5.79 (ddt, J = 17.1, 10.4, 6.5 Hz, 1H), 7.22−7.29 (several peaks, 3H), 7.53 (m, 2H). ^{13}C NMR δ 29.4, 31.8, 33.2, 56.6, 74.2, 77.6, 80.0, 114.9, 126.9, 129.0, 130.4, 132.7, 138.0.

2-Phenyl-2-(2-propynyloxy)ethyl phenyl selenide (4m): ^{1}H NMR δ 2.39 (t, J = 2.3 Hz, 1H), 3.14 (dd, J = 12.2, 5.8 Hz, 1H), 3.37 (dd, J = 12.4, 7.9 Hz, 1H), 3.88 (dd, J = 15.7, 2.3 Hz, 1H), 4.14 (dd, J = 15.7, 2.4 Hz, 1H), 4.73 (dd, J = 7.9, 5.6 Hz, 1H), 7.20−7.37 (several peaks, 8H), 7.48 (m, 2H). ^{13}C NMR δ 34.8, 55.9, 74.5, 79.5, 80.0, 126.8, 127.0, 128.4, 128.6, 128.9, 130.5, 132.7, 139.8.

3-(Allyloxy)-2-(2-propynyloxy)propyl phenyl selenide (4n): ^{1}H NMR δ 2.39 (t, J = 2.4 Hz, 1H), 3.11 (dd, J = 12.7, 5.9 Hz, 1H), 3.15 (dd, J = 12.7, 6.3 Hz, 1H), 3.61 (d, J = 4.8 Hz, 2H), 3.92 (tt, J = 6.1, 4.8 Hz, 1H), 3.94 (dt, J = 5.6, 1.4 Hz, 2H), 4.24 (dd, J = 16.0, 2.4 Hz, 1H), 4.29 (dd, J = 15.9, 2.4 Hz, 1H), 5.17 (ddt, J = 10.4, 1.9, 1.3 Hz, 1H), 5.25 (dq, J = 17.2, 1.6 Hz, 1H), 5.87 (ddt, J = 17.2, 10.4, 5. 6 Hz, 1H), 7.22−7.28 (several peaks, 3H), 7.53 (m, 2H). ^{13}C NMR δ 28.8, 57.3, 71.1, 72.3, 74.4, 77.1, 79.8, 117.1, 126.9, 129.0, 130.3, 132.5, 134.5.

3-Phenoxy-2-(2-propynyloxy)propyl phenyl selenide (4o): ^{1}H NMR δ 2.39 (dt, J = 3.0, 2.4 Hz, 1H), 3.20 (dd, J = 12.2, 6.0 Hz, 1H), 3.24 (dd, J = 12.8, 6.0 Hz, 1H), 4.08−4.17 (several peaks, 3H), 4.23 (dd, J = 2.4, 0.6 Hz, 1H), 4.37 (dd, J = 2.4, 0.6 Hz, 1H), 6.84−6.98 (several peaks, 3H), 7.20−7.29 (several peaks, 5H), 7.53 (m, 2H). ^{13}C NMR δ 28.8, 57.6, 68.8, 74.7, 76.6, 79.6, 114.5, 121.0, 121.1, 127.0, 129.1, 129.4, 132.6, 158.4.

3-(Benzyloxy)-2-(2-propynyloxy)propyl phenyl selenide (4p): ^{1}H NMR δ 2.37 (t, J = 2.4 Hz, 1H), 3.12 (dd, J = 12.7, 5.9 Hz, 1H), 3.15 (dd, J = 12.6, 6.4 Hz, 1H), 3.65 (d, J = 4.8 Hz, 2H), 3.93 (tt, J = 12.2, 4.7 Hz, 1H), 4.23 (dd, J = 15.9, 2.4 Hz, 1H), 4.28 (dd, J = 15.8, 2.3 Hz, 1H), 4.49 (s, 2H), 7.21−7.36 (several peaks, 8H), 7.51 (m, 2H). ^{13}C NMR δ 28.9, 57.3, 71.2, 73.4, 74.5, 77.2, 79.8, 126.9, 127.6, 128.3, 129.0, 130.3, 132.5, 138.0.

1-Phenyl-2-(2-propynyloxy)ethyl phenyl selenide (2d): ^{1}H NMR δ 2.39 (t, J = 2.4 Hz, 1H), 3.96 (dd, J = 10.0, 5.9 Hz, 1H), 4.10 (dd, J = 10.0, 8.6 Hz, 1H), 4.11 (d, J = 2.4 Hz, 2H), 4.49 (dd, J = 8.5, 5.9 Hz, 1H), 7.19−7.29 (several peaks, 8H), 7.45 (m, 2H). ^{13}C NMR δ 46.8, 58.1, 72.3, 74.7, 79.3, 127.3, 127.9, 128.4, 128.8, 135.3, 139.5.

Typical Procedure for Group Transfer Radical Cyclization. Preparation of *cis*-3-[[[4-(Trifluoromethyl)-phenyl]telluro]methyl]octahydrobenzofuran (6j). To a solution of 2-(allyloxy)cyclohexyl 4-(trifluoromethyl)phenyl telluride (0.46 g, 1.1 mmol) in dry benzene (10 mL) under nitrogen was added hexabutylditin (0.026 g, 0.45 mmol), and the resulting reaction mixture was irradiated with a sun lamp. The position of the sun lamp was adjusted so that a vigorous reflux could be maintained. After 1 h, TLC showed complete consumption of the starting material. The flask was cooled and the solvent removed *in vacuo*. The residue on purification by flash chromatography (4%ether/pentane) afforded 0.31 g(69%) of the title compound as a 2:1 mixture of *exo* and *endo* isomers. ^{1}H NMR *exo*-**6j** δ 1.10−1.99 (several peaks, 9H), 2.29 (m, 1H), 2.91 (dd, J = 11.8, 8.4 Hz, 1H), 3.11 (dd, J = 11.9, 7.2 Hz, 1H), 3.47 (dd, J = 9.1, 4.9 Hz, 1H), 4.01 (t, J = 8.3 Hz, 1H), 4.18 (dd, J = 9.1, 7.4 Hz, 1H), 7.43 (m, 2H), 7.80 (m, 2H). ^{1}H NMR *endo*-**6j** δ 1.10−1.99 (several peaks, 9H), 2.72 (m, 1H), 2.90 (dd, J = 11.5, 7.7 Hz, 1H), 2.99 (dd, J = 11.5, 8.5 Hz, 1H), 3.53 (dd, J = 9.9, 8.1 Hz, 1H), 3.95−4.05 (several peaks, 2H), 7.43 (m, 2H), 7.80 (m, 2H). ^{13}C NMR *exo*+*endo* δ 6.0, 13.6, 20.3, 20.9, 21.6, 23.6, 24.2, 27.4, 28.2, 28.5, 41.6, 45.3, 45.8, 45.8, 72.0, 73.9, 76.4, 78.4, 116.9, 122.7, 125.7 (q), 129.8 (q), 137.8. Anal. Calcd for $C_{16}H_{19}F_3OTe$: C, 46.65; H, 4.65. Found: C, 46.86; H, 4.68. MS m/z (relative intensity) 414 (M^+, 9.6).

The following compounds were similarly prepared. As judged by ^{1}H NMR spectroscopy, the purity of the materials were >98%. For yields and *cis*/*trans*-, *E*/*Z*-, and *syn*/*anti*-ratios see Table 1.

3-[(Phenyltelluro)methyl]cyclopentanol (5a): ^{1}H NMR *trans*-**5a** δ 1.23 (m, 1H), 1.43 (ddd, J = 13.7, 9.8, 5.9 Hz, 1H), 1.59 (m, 1H), 1.86 (m, 1H), 1.96-2.08 (several peaks, 2H), 2.46−2.59 (several peaks, 2H), 2.98 (d, J = 7.2 Hz, 2H), 4.37 (m, 1H), 7.15−7.32 (several peaks, 3H), 7.73 (m, 2H). ^{1}H NMR *cis*-**5a** δ 1.27 (m, 1H), 1.47 (m, 1H), 1.67 (m, 1H), 1.74−1.90 (several peaks, 2H), 2.13−2.40 (several peaks, 3H), 3.04−3.07 (several peaks, 2H), 4.30 (m, 1H), 7.15−7.32 (several peaks, 3H), 7.73 (m, 2H). ^{13}C NMR *cis*+*trans* δ 16.1, 16.7, 32.2, 32.4, 35.4, 35.7, 39.0, 40.0, 44.0, 44.4, 73.7, 73.8, 111.8, 127.5, 129.1, 138.3. MS m/z (relative intensity) 303 (M^+, 42.9).

3-[[[4-(*N*,*N*-dimethylamino)phenyl]telluro]methyl]-cyclopentanol (5b): ^{1}H NMR *trans*-**5b** δ 1.21 (m, 1H), 1.43 (ddd, J = 13.6, 9.4, 5.9 Hz, 1H), 1.57 (m, 1H), 1.84 (m, 1H), 1.94-2.07 (several peaks, 2H), 2.41−2.54 (several peaks, 2H), 2.85 (d, J = 7.2 Hz, 2H), 2.95 (s, 6H), 4.38 (m, 1H), 6.56 (m, 2H), 7.63 (m, 2H). ^{1}H NMR *cis*-**5b** δ 1.25 (m, 1H), 1.46 (m, 1H), 1.67 (m, 1H), 1.74−1.89 (several peaks, 2H), 2.13−2.25 (several peaks, 3H), 2.93 (dd, J = 6.8, 2.8 Hz, 2H), 2.95 (s, 6H), 4.30 (m, 1H), 6.56 (m, 2H), 7.63 (m, 2H). ^{13}C NMR *cis*+*trans* δ 16.4, 17.0, 32.1, 32.3, 35.4, 35.7, 39.0, 39.9, 40.2, 44.0, 44.3, 73.8, 73.8, 94.9, 113.4, 141.0, 141.0, 150.2. MS m/z (relative intensity) 349 (M^+, 46.9).

3-[[[4-(Trifluoromethyl)phenyl]telluro]methyl]-cyclopentanol (5c) ^{1}H NMR *trans*-**5c** δ 1.24 (m, 1H), 1.43 (ddd, J = 13.4, 10.1, 5.6 Hz, 1H), 1.61 (m, 1H), 1.88 (m, 1H), 1.98−2.10 (several peaks, 2H), 2.49−2.61 (several peaks, 2H), 3.03 (d, J = 7.2 Hz, 2H), 4.40 (m, 1H), 7.41 (m, 2H), 7.77 (m, 2H). ^{1}H NMR *cis*-**5c** δ 1.29 (m, 1H), 1.49 (m, 1H), 1.69 (m, 1H), 1.76−1.92 (several peaks, 2H), 2.16−2.29 (several peaks, 3H), 3.10 (dd, J = 11.6, 7.0 Hz, 1H), 3.13 (dd, J = 11.6, 7.0 Hz, 1H), 4.34 (m, 1H), 7.41 (m, 2H), 7.77 (m, 2H). ^{13}C NMR *cis*+*trans* δ 16.4, 17.1, 32.3, 32.5, 35.4, 35.8, 38.9, 39.9, 44.0, 44.4, 73.6, 73.7, 117.6, 122.8, 125.55, 125.59, 129.3, 129.6, 137.4. MS m/z (relative intensity) 374 (M^+, 4.8).

3-[(2-Thienyltelluro)methyl]cyclopentanol (5d): ^{1}H NMR *trans*-**5d** δ 1.20 (m, 1H), 1.42 (ddd, J = 13.4, 9.5, 5.6 Hz, 1H), 1.59 (m, 1H), 1.86 (m, 1H), 1.96−2.08 (several peaks, 2H), 2.48 (m, 2H), 2.87 (d, J = 7.2 Hz, 2H), 4.38 (m, 1H), 6.92 (dd, J = 5.2, 3.4 Hz, 1H), 7.37 (dd, J = 3.4, 1.2 Hz, 1H), 7.42 (dd, J = 5.2, 1.1 Hz, 1H). ^{1}H NMR *cis*-**5d** δ 1.26 (m, 1H), 1.46 (m, 1H), 1.68 (m, 1H), 1.75−1.91 (several peaks, 2H), 2.13−2.25 (several peaks, 3H), 2.95 (dd, J = 7.0, 1.2 Hz, 1H), 4.32 (m, 1H), 6.92 (dd, J = 5.2, 3.5 Hz, 1H), 7.37 (dd, J = 3.4, 1.2 Hz, 1H), 7.42 (dd, J = 5.2, 1.1 Hz, 1H). ^{13}C NMR *cis*+*trans* δ 19.0, 19.6, 32.0, 32.2, 35.3, 35.7, 38.8, 39.7, 43.8, 44.2, 73.6, 73.7, 97.4, 128.8, 134.0, 141.1. MS m/z (relative intensity) 312 (M^+, 8.3).

2-(3-Butenyl)-4-[(2-thienyltelluro)methyl]tetrahydrofuran (6d): ^{1}H NMR *trans*-**6d** δ 1.45−1.72 (several peaks, 2H), 1.72−1.77 (several peaks, 2H), 2.01−2.19 (several peaks, 2H), 2.55 (m, 1H), 2.81 (dd, J = 11.8, 7.6 Hz, 1H), 2.87 (dd, J = 11.7, 7.4 Hz, 1H), 3.37 (dd, J = 8.8, 6.7 Hz, 1H), 3.96 (dd, J = 7.1, 5.8 Hz, 1H), 4.02 (dd, J = 8.8, 6.7 Hz, 1H), 4.95 (ddt, J = 10.2, 2.0, 1.2 Hz, 1H), 5.02 (dm, J = 17.1 Hz, 1H), 5.81 (ddt, J = 17.0, 10.4, 6.7 Hz, 1H), 6.94 (dd, J = 5.1, 3.4 Hz, 1H), 7.38 (dd, J = 3.4, 1.1 Hz, 1H), 7.44 (dd, J = 5.2, 1.1 Hz, 1H). *Cis*-**6d** could not be resolved. ^{13}C NMR *cis*+*trans* δ 15.0, 15.3, 30.3, 30.4, 35.1, 39.6, 40.3, 40.7, 41.1, 73.8, 74.2, 78.2, 79.6, 96.9, 114.6, 128.9, 134.4, 138.2, 141.5. MS m/z (relative intensity) 352 (M^+, 10.6).

2-Ethyl-4-[[[4-(trifluoromethyl)phenyl]telluro]methyl]-tetrahydrofuran (6e): ^{1}H NMR *trans*-**6e** δ 0.90 (t, J = 7.5

Hz, 3H), 1.36−1.65 (several peaks, 2H), 1.75 (t, J = 7.0 Hz, 2H), 2.58 (m, 1H), 2.98 (dd, J = 11.8, 7.6 Hz, 1H), 3.06 (dd, J = 11.9, 7.4 Hz, 1H), 3.40 (dd, J = 8.7, 6.5 Hz, 1H), 3.91 (m, 1H), 4.02 (dd, J = 8.8, 6.6 Hz, 1H), 7.42 (m, 2H), 7.79 (m, 2H). ^{1}H NMR *cis*-**6e** δ 0.91 (t, J = 7.5 Hz, 3H), 1.21 (dd, J = 12.3, 9.0 Hz, 1H), 1.36−1.65 (several peaks, 2H), 2.23 (ddd, J = 12.3, 7.5, 5.9 Hz, 1H), 2.61 (m, 1H), 3.00 (dd, J = 11.4, 7.4 Hz, 1H), 3.05 (dd, J = 11.7, 7.5 Hz, 1H), 3.53 (dd, J = 8.6, 6.6 Hz, 1H), 3.80 (m, 1H), 3.89 (dd, J = 8.6, 6.7 Hz, 1H), 7.42 (m, 2H), 7.79 (m, 2H). ^{13}C NMR δ *cis*+*trans* 10.3, 10.4, 12.8, 13.2, 28.7, 39.4, 40.5, 41.1, 74.0, 74.3, 80.3, 81.7, 116.9, 125.7 (q), 129.7, 137.8. MS *m*/*z* (relative intensity) 388 (M^+, 17.1).

2-Phenyl-4-[[[4-(trifluoromethyl)phenyl]telluro]methyl]-tetrahydrofuran (6f): ^{1}H NMR *trans*-**6f** δ 2.00−2.06 (m, 2H), 2.63 (m, 1H), 2.97 (dd, J = 12.0, 7.7 Hz, 1H), 3.04 (dd, J = 12.0, 7.3 Hz, 1H), 3.54 (dd, J = 8.7, 6.6 Hz, 1H), 4.18 (dd, J = 8.7, 6.8 Hz, 1H), 4.99 (t, J = 7.0 Hz, 1H), 7.14−7.29 (several peaks, 5H), 7.36 (m, 2H), 7.73 (m, 2H). ^{1}H NMR *cis*-**6f** δ 1.97−2.09 (several peaks, 2H), 2.49 (ddd, J = 12.2, 7.4, 6.2 Hz, 1H), 2.97 (m, 2H), 3.67 (dd, J = 8.5, 7.1 Hz, 1H), 4.04 (dd, J = 8.5, 7.3 Hz, 1H), 4.85 (dd, J = 9.4, 6.1 Hz, 1H), 7.14−7.29 (several peaks, 5H), 7.36 (m, 2H), 7.73 (m, 2H). ^{13}C NMR *cis*+*trans* δ 12.2, 23.0, 40.6, 42.8, 75.1, 80.1, 125.4, 125.7, 127.2, 128.3, 137.9, 143.1. MS *m*/*z* (relative intensity) 436 (M^+, 6.2).

2-[(Allyloxy)methyl]-4-[[[4-(trifluoromethyl)phenyl]-telluro]methyl]tetrahydrofuran (6g): ^{1}H NMR *trans*-**6g** δ 1.75 (ddd, J = 12.7, 7.7, 5.9 Hz, 1H), 1.94 (ddd, J = 12.7, 8.2, 6.5 Hz, 1H), 2.62 (m, 1H), 2.97 (dd, J = 11.8, 7.6 Hz, 1H), 3.05 (dd, J = 11.9, 7.3 Hz, 1H), 3.41 (d, J = 5.2 Hz, 2H), 3.46 (dd, J = 8.7, 6.5 Hz, 1H), 4.00 (ddd, J = 1.5, 1.3, 0.8 Hz, 1H), 4.01 (m, 2H), 4.24 (m, 1H), 5.16 (ddt, J = 10.4, 1.8, 1.3 Hz, 1H), 5.25 (dq, J = 17.2, 1.7 Hz, 1H), 5.89 (ddt, J = 17.2, 10.4, 5.6 Hz, 1H), 7.45 (m, 2H), 7.81 (m, 2H). ^{13}C NMR *trans*-**6g** δ 12.2, 36.5, 40.5, 72.4, 72.6, 74.7, 77.6, 117.0, 125.7 (q), 134.6, 137.9. MS *m*/*z* (relative intensity) 430 (M^+, 10.1).

2-(Phenoxymethyl)-4-[[[4-(trifluoromethyl)phenyl]tel-luro]methyl]tetrahydrofuran (6h): ^{1}H NMR *trans*-**6h** δ 1.88 (ddd, J = 12.8, 7.8, 6.3 Hz, 1H), 2.11 (ddd, J = 12.7, 8.1, 6.0 Hz, 1H), 2.70 (m, 1H), 3.03 (dd, J = 11.8, 7.6 Hz, 1H), 3.10 (dd, J = 11.9, 7.3 Hz, 1H), 3.55 (dd, J = 8.6, 6.6 Hz, 1H), 3.93 (dd, J = 9.8, 4.7 Hz, 1H), 3.97 (dd, J = 9.6, 5.2 Hz, 1H), 4.12 (dd, J = 8.7, 6.5 Hz, 1H), 4.45 (m, 1H), 6.86−6.97 (several peaks, 3H), 7.26 (m, 2H), 7.43 (m, 2H), 7.80 (m, 2H). ^{1}H NMR *cis*-**6h** δ 1.58 (dt, J = 12.5, 8.5 Hz, 1H), 2.35 (ddd, J = 12.3, 7.6, 6.9 Hz, 1H), 2.68 (m, 1H), 3.03 (dd, J = 11.8, 7.4 Hz, 1H), 3.13 (dd, J = 11.9, 7.4 Hz, 1H), 3.55 (dd, J = 8.4, 6.7 Hz, 1H), 3.64 (dd, J = 8.6, 7.0 Hz, 1H), 4.02 (m, 2H), 4.34 (m, 1H), 6.86−6.97 (several peaks, 3H), 7.26 (m, 2H), 7.43 (m, 2H), 7.80 (m, 2H). ^{13}C NMR *trans*+*cis*- δ 11.9, 12.2, 36.6, 37.0, 40.5, 41.1, 70.1, 74.6, 74.8, 76.9, 77.9, 114.5, 114.5, 116.8, 120.9, 125.7 (q), 129.4, 129.7, 137.8, 137.9, 158.7. MS *m*/*z* (relative intensity) 466 (M^+, 14.0).

2-[(Benzyloxy)methyl]-4-[[[4-(trifluoromethyl)phenyl]-telluro]methyl]tetrahydrofuran (6i): ^{1}H NMR *trans*-**6i** δ 1.74 (ddd, J = 12.6, 7.8, 5.9 Hz, 1H), 1.95 (ddd, J = 12.6, 8.0, 6.4 Hz, 1H), 2.55−2.66 (m, 1H), 2.97 (dd, J = 11.9, 7.6 Hz, 1H), 3.04 (dd, J = 11.9, 7.2 Hz, 1H), 3.44 (s, 1H), 3.45 (s, 1H), 3.47 (dd, J = 8.6, 6.6 Hz, 1H), 4.05 (dd, J = 8.7, 6.5 Hz, 1H), 4.21−4.28 (m, 1H), 4.53 (d, J = 12.1 Hz, 1H), 4.57 (d, J = 12.1 Hz, 1H), 7.27−7.35 (m, 5H), 7.40−7.45 (m, 2H), 7.76−7.82 (m, 2H). ^{13}C NMR *trans*-**6i** δ 12.3, 36.6, 40.5, 72.6, 73.4, 74.7, 77.7, 125.7 (q), 127.5 (q), 128.3, 137.8, 138.2. MS *m*/*z* (relative intensity) 480 (M^+, 2.3).

***cis*-3-[(2-Thienyltelluro)methyl]octahydrobenzo-furan (6k):** ^{1}H NMR *exo*-**6k** δ 1.05−1.65 (several peaks, 7H), 1.77−1.84 (several peaks, 2H), 2.24 (m, 1H), 2.75 (dd, J = 11.8, 8.2 Hz, 1H), 2.93 (dd, J = 11.8, 7.4 Hz, 1H), 3.45 (dd, J = 9.1, 4.9 Hz, 1H), 3.95 (m, 1H), 4.16 (dd, J = 9.1, 7.5 Hz, 1H), 6.94 (dd, J = 5.2, 3.4 Hz, 1H), 7.38 (dd, J = 3.4, 1.1 Hz, 1H), 7.44 (dd, J = 5.2, 1.1 Hz, 1H). ^{1}H NMR *endo*-**6k** δ 1.06 (m, 1H), 1.38−1.54 (several peaks, 5H), 1.71 (m, 1H), 1.90−1.98 (several peaks, 2H), 2.68 (m, 1H), 2.73 (dd, J = 11.4, 7.3 Hz, 1H), 2.81 (dd, J = 11.4, 8.1 Hz, 1H), 3.50 (dd, J = 9.5, 8.1 Hz, 1H), 3.94 (m, 1H), 4.00 (t, J = 8.4 Hz, 1H), 6.94 (dd, J = 5.2, 3.4 Hz, 1H), 7.39 (dd, J = 3.4, 1.1 Hz, 1H), 7.44 (dd, J = 5.2, 1.1 Hz, 1H). ^{13}C NMR *exo*+*endo* δ 8.3, 15.9, 20.3, 20.9, 21.6, 23.6, 24.2, 27.4, 28.2, 28.6, 41.4, 45.2, 45.5, 45.6, 71.8, 73.8, 76.2, 78.3, 96.8, 96.8, 128.9, 128.9, 134.4, 134.5, 141.6, 141.7. MS *m*/*z* (relative intensity) 352 (M^+, 35.9).

2-(3-Butenyl)-4-[[[4-(trifluoromethyl)phenyl]telluro]-methylene]tetrahydrofuran (7c): ^{1}H NMR (*Z*)-**7c** δ 1.61 (m, 1H), 1.78 (m, 1H), 2.07−2.25 (several peaks, 2H), 2.45 (ddddd, J = 16.0, 8.8, 2.3, 2.3, 1.0 Hz, 1H), 2.85 (ddq, J = 15.9, 5.8, 1.6 Hz, 1H), 4.04 (ddt, J = 8.6, 7.1, 5.7 Hz, 1H), 4.20 (dddd, J = 14.3, 2.5, 2.5, 1.0 Hz, 1H), 4.37 (dm, J = 14.3 Hz, 1H), 4.98 (dm, J = 10.2 Hz, 1H), 5.05 (dddd, J = 17.1, 3.6, 1.6.0.4 Hz, 1H), 5.83 (ddt, J = 17.0, 10.2, 6.6 Hz, 1H), 6.67 (m, 1H), 7.44 (m, 2H), 7.70 (m, 2H). ^{1}H NMR (*E*)-**7c** δ 1.63 (m, 1H), 1.77 (m, 1H), 2.09−2.24 (several peaks, 3H), 2.65 (dm, J = 16.2 Hz, 1H), 4.03 (m, 1H), 4.39 (dddd, J = 13.4, 2.2, 2.2, 1.1 Hz, 1H), 4.53 (dm, J = 13.5 Hz, 1H), 4.98 (ddt, J = 10.2, 1.9, 1.3 Hz, 1H), 5.04 (dq, J = 17.0, 1.6 Hz, 1H), 5.83 (ddt, J = 17.1, 10.3, 6.5 Hz, 1H), 6.60 (m, 1H), 7.44 (m, 2H), 7.70 (m, 2H). MS *m*/*z* (relative intensity) 412 (M^+, 100).

2-Ethyl-4-[[[4-(trifluoromethyl)phenyl]telluro]m-ethylene]tetrahydrofuran (7e): ^{1}H NMR (*E*)-**7e** δ 0.88 (t, J = 7.4 Hz, 3H), 1.50−1.75 (several peaks, 2H), 2.19 (ddm, J = 16.5, 8.6 Hz, 1H), 2.64 (ddm, J = 16.5, 6.2 Hz, 1H), 3.95 (m, 1H), 4.39 (dddd, J = 13.5, 2.2, 2.2, 1.2 Hz, 1H), 4.53 (m, 1H), 6.60 (m, 1H), 7.43 (m, 2H), 7.68 (m, 2H). ^{13}C NMR (*E*)-**7e** δ 10.1, 30.3, 41.7, 72.0, 81.0, 89.1, 125.8, 125.9, 135.9, 155.3. ^{1}H NMR (*Z*)-**7e** δ 0.96 (t, J = 7.4 Hz, 3H), 1.55 (dq, J = 13.7, 7.3 Hz, 1H), 1.69 (dq, J = 13.6, 7.5 Hz, 1H), 2.44 (ddddd, J = 15.8, 8.7, 2.2, 2.2, 1.0 Hz, 1H), 2.83 (ddddd, J = 15.7, 5.8, 1.7, 1.7, 1.1 Hz, 1H), 3.97 (dq, J = 8.5, 6.1 Hz, 1H), 4.20 (dddd, J = 14.3, 2.5, 2.5, 1.0 Hz, 1H), 4.38 (ddddd, J = 14.3, 3.9, 2.3, 1.0, 0.5 Hz, 1H), 6.67 (ddd, J = 4.7, 2.3, 1.6 Hz, 1H), 7.43 (m, 2H), 7.68 (m, 2H). ^{13}C NMR (*Z*)-**7e** δ 10.1, 28.0, 41.1, 73.9, 82.0, 87.9, 125.8, 125.9, 135.9, 156.0. MS *m*/*z* (relative intensity) 386 (M^+, 19.1).

2-Phenyl-4-[[[4-(trifluoromethyl)phenyl]telluro]m-ethylene]tetrahydrofuran (7f): ^{1}H NMR (*E*)-**7f** δ 2.57 (ddddd, J = 16.3, 8.5, 2.8, 2.3, 0.9 Hz, 1H), 2.99 (ddddd, J = 16.3, 6.5, 2.1, 2.1, 1.2 Hz, 1H), 4.57 (dddd, J = 13.4, 2.1, 2.1, 1.2 Hz, 1H), 4.72 (ddddd, J = 13.5, 2.5, 0.8, 0.8, 0.5 Hz, 1H), 5.07 (dd, J = 8.5, 6.5 Hz, 1H), 6.68 (ddt, J = 4.6, 2.7, 2.0 Hz, 1H), 7.26−7.37 (several peaks, 5H), 7.44 (m, 2H), 7.70 (m, 2H). ^{13}C NMR (*E*)-**7f** δ 44.4, 72.4, 77.2, 80.7, 89.6, 125.9 (q), 127.8, 128.5, 136.0, 136.1, 141.1, 154.3. ^{1}H NMR (*Z*)-**7f** δ 2.81 (ddddd, J = 15.8, 8.8, 2.2, 2.2, 1.0 Hz, 1H), 3.16 (ddddd, J = 15.9, 6.2, 1.7, 1.7, 1.0 Hz, 1H), 4.38 (dddd, J = 14.2, 4.7, 2.3, 1.0 Hz, 1H), 4.57 (dddd, J = 14.3, 2.7, 1.5, 0.9 Hz, 1H), 5.08 (dd, J = 8.7, 6.0 Hz, 1H), 6.75 (ddd, J = 4.7, 2.3, 1.5 Hz, 1H), 7.27−7.38 (several peaks, 5H), 7.44 (m, 2H), 7.70 (m, 2H). ^{13}C NMR (*Z*)-**7f** δ 43.8, 74.3, 77.2, 81.7, 88.7, 125.8 (q), 127.8, 128.5, 129.8, 136.0, 141.1, 155.1. MS *m*/*z* (relative intensity) 434 (M^+, 7.0).

2-[(Allyloxy)methyl]-4-[[[4-(trifluoromethyl)phenyl]-telluro]methylene]tetrahydrofuran (7g): ^{1}H NMR (*E*)-**7g** δ 2.40 (ddddd, J = 16.5, 7.9, 2.8, 2.2, 0.8 Hz, 1H), 2.63 (ddddd, J = 16.5, 6.8, 2.2, 1.7, 1.1 Hz, 1H), 3.52 (dd, J = 10.1, 5.8 Hz, 1H), 3.56 (dd, J = 10.3, 4.2 Hz, 1H), 4.02−4.05 (several peaks, 2H), 4.27 (m, 1H), 4.44 (dddd, J = 13.4, 2.1, 2.1, 1.1 Hz, 1H), 4.58 (dm, J = 13.4 Hz, 1H), 5.19 (ddt, J = 10.4, 1.7, 1.2 Hz, 1H), 5.27 (dq, J = 17.2, 1.6 Hz, 1H), 5.91 (ddt, J = 17.2, 10.3, 5.6 Hz, 1H), 6.63 (m, 1H), 7.43 (m, 2H), 7.71 (m, 2H). ^{13}C NMR (*E*)-**7g** δ 38.5, 71.8, 72.4, 72.5, 78.3, 89.6, 117.3, 119.9, 125.8 (q), 134.5, 136.0, 154.4. ^{1}H NMR (*Z*)-**7g** δ 2.66 (ddddd, J = 16.0, 8.1, 2.2, 2.2, 1.0 Hz, 1H), 2.83 (ddm, J = 16.0, 6.4, Hz, 1H), 3.52 (dd, J = 10.4, 5.4 Hz, 1H), 3.54 (dd, J = 10.3, 4.6 Hz, 1H), 4.04 (m, 2H), 4.26 (dddd, J = 14.2, 2.4, 2.2, 1.0 Hz, 1H), 4.29 (m, 1H), 4.42 (dm, J = 14.2 Hz, 1H), 5.19 (ddt, J = 10.4, 1.8, 1.2 Hz, 1H), 5.28 (dq, J = 17.2, 1.7 Hz, 1H), 5.91 (ddd, J = 17.2, 10.3, 5.6 Hz, 1H), 6.70 (ddd, J = 4.6, 2.4, 1.7 Hz, 1H), 7.43 (m, 2H), 7.69 (m, 2H). ^{13}C NMR (*Z*)-**7g** δ 37.8, 71.8, 72.5, 74.3, 79.2, 88.5, 108.9, 117.3, 119.9, 125.8 (q), 129.7, 134.5, 135.9, 154.9. MS *m*/*z* (relative intensity) 428 (M^+, 18.0).

2-(Phenoxymethyl)-4-[[[4-(trifluoromethyl)phenyl]tel-luro]methylene]tetrahydrofuran (7h): ^{1}H NMR (*E*)-**7h** δ 2.54 (ddddd, J = 16.6, 7.5, 2.9, 2.1, 0.9 Hz, 1H), 2.76 (ddddd, J = 16.5, 6.9, 2.3, 1.8, 1.1 Hz, 1H), 4.06 (d, J = 4.9 Hz, 2H), 4.46−4.55 (several peaks, 2H), 4.64 (dm, J = 13.4 Hz, 1H),

6.68 (tt, J = 4.8, 2.4 Hz, 1H), 6.90−6.99 (m, 3H), 7.28 (m, 2H), 7.44 (m, 2H), 7.72 (m, 2H). ^{13}C NMR ^{1}H NMR (*E*)-**7h** δ 38.5, 69.6, 72.5, 77.5, 90.0, 114.6, 121.1, 125.9 (q), 129.5, 136.2, 153.9, 158.6. ^{1}H NMR (*Z*)-**7h** δ 2.79 (ddddd, J = 16.0, 7.5, 2.1, 2.1, 1.0 Hz, 1H), 2.97 (ddddd, J = 16.1, 6.7, 1.8, 1.8, 1.2 Hz, 1H), 4.02 (dd, J = 9.9, 4.7 Hz, 1H), 4.07 (dd, J = 9.9, 5.4 Hz, 1H), 4.33 (dddd, J = 14.2, 2.4, 2.1, 1.0 Hz, 1H), 4.44−4.55 (several peaks, 2H), 6.75 (tt, J = 4.4, 2.4 Hz, 1H), 6.89−6.99 (several peaks, 3H), 7.28 (m, 2H), 7.44 (m, 2H), 7.71 (m, 2H). ^{13}C NMR (*Z*)-**7h** δ 37.9, 69.6, 74.4, 78.3, 89.0, 114.6, 121.1, 125.9 (q), 129.5, 136.1, 154.2, 158.6. MS *m*/*z* (relative intensity) 464 (M^+, 13.9).

2-[(Benzyloxy)methyl]-4-[[[4-(trifluoromethyl)phenyl]-telluro]methylene]tetrahydrofuran (7i): ^{1}H NMR (*E*)-**7i** δ 2.41 (ddt, J = 16.6, 8.3, 2.5 Hz, 1H), 2.63 (ddddd, J = 16.5, 6.9, 2.0, 1.6, 1.1 Hz, 1H), 3.55 (dd, J = 10.3, 5.5 Hz, 1H), 3.58 (dd, J = 10.2, 4.3 Hz, 1H), 4.30 (dddd, J = 12.2, 7.7, 5.3, 4.2 Hz, 1H), 4.45 (dddd, J = 13.3, 4.0, 2.0, 1.1 Hz, 1H), 4.55−4.62 (several peaks, 3H), 6.65 (m, 1H), 7.26−7.35 (several peaks, 5H), 7.42 (m, 2H), 7.70 (m, 2H). ^{13}C NMR (*E*)-**7i** δ 38.5, 71.8, 72.4, 73.5, 78.3, 89.6, 125.8, 125.9, 127.7, 128.4, 136.1, 138.0, 154.4. ^{1}H NMR (*Z*)-**7i** δ 2.67 (ddddd, J = 16.0, 7.9, 2.2, 2.2, 1.0 Hz, 1H), 2.83 (ddddd, J = 16.0, 6.5, 1.7, 1.7, 1.0 Hz, 1H), 3.55 (d, J = 4.9 Hz, 2H), 4.20 (dm, J = 14.2 Hz, 1H) 4.27 (dddd, J = 14.2, 2.4, 2.2, 1.0 Hz, 1H), 4.31 (ddt, J = 7.9, 6.5, 4.9 Hz, 1H), 4.57 (d, J = 12.2 Hz, 1H), 4.60 (d, J = 12.1 Hz, 1H), 6.71 (m, 1H), 7.26−7.35 (several peaks, 5H), 7.42 (m, 2H), 7.67 (m, 2H). ^{13}C NMR (*Z*)-**7i** δ 37.8, 71.8, 73.5, 74.3, 79.2, 88.4, 125.8, 125.9, 127.7, 128.4, 135.9, 138.0, 154.9. MS *m*/*z* (relative intensity) 478 (M^+, 1.3).

***cis*-3-[[[4-(Trifluoromethyl)phenyl]telluro]methylene]-octahydrobenzofuran (7j):** ^{1}H NMR (*E*)-**7j** δ 1.17−1.33 (several peaks, 2H), 1.45−1.77 (several peaks, 5H), 2.06 (m, 1H), 2.58 (m, 1H), 3.98 (m, 1H), 4.43 (ddd, J = 14.0, 1.9, 0.7 Hz, 1H), 4.67 (dddd, J = 14.0, 1.9, 1.3, 0.5 Hz, 1H), 6.46 (ddd, J = 1.9, 1.9, 1.1 Hz, 1H), 7.43 (m, 2H), 7.68 (m, 2H). ^{13}C NMR (*E*)-**7j** δ 20.1, 24.1, 26.6, 27.6, 47.1, 71.1, 79.0, 86.7, 109.5, 125.8 (q), 135.6. ^{1}H NMR (*Z*)-**7j** δ 1.22−1.83 (several peaks, 8H), 2.74 (m, 1H), 4.10 (q, J = 4.7 Hz, 1H), 4.26 (ddd, J = 14.5, 2.6, 0.9 Hz, 1H), 4.42 (dm, J = 14.6 Hz, 1H), 6.61 (ddd, J = 2.5, 2.5, 1.4 Hz, 1H), 7.43 (m, 2H), 7.68 (m, 2H). ^{13}C NMR (*Z*)-**7j** δ 21.0, 23.2, 27.3, 27.8, 46.5, 73.1, 78.5, 86.8, 112.7, 125.8 (q), 135.6, 160.5. MS *m*/*z* (relative intensity) 412 (M^+, 100).

Typical Procedure for Reductive Radical Cyclization. Preparation of 2-[(Benzyloxy)methyl]-4-methyltetrahydrofuran (9p). To a solution of 2-(allyloxy)-3-(benzyloxy)-propyl phenyl selenide (0.52 g, 1.4 mmol) in dry benzene (20 mL) under nitrogen was added AIBN (0.067 g, 0.41 mmol). The reaction flask was then lowered in an oil bath preheated to 90 °C. Tributyltin hydride (0.52 g, 1.8 mmol) was then added dropwise and the reaction mixture refluxed. After 45 min, TLC showed the complete consumption of the starting material. The flask was cooled and the solvent removed *in vacuo*. After purification by flash chromatography (20% ether/pentane), 0.28 g (94%) of the title compound was obtained as a 1:3 mixture of *cis* and *trans* isomers. ^{1}H NMR *trans*-**9p** δ 1.02 (d, J = 6.8 Hz, 3H), 1.56 (ddd, J = 12.4, 6.7, 6.4 Hz, 1H), 1.84 (ddd, J = 12.3, 8.1, 6.2 Hz, 1H), 2.32 (ddt, J = 13.4, 8.1, 6.7 Hz, 1H), 3.31 (dd, J = 8.3, 7.0 Hz, 1H), 3.45 (d, J = 5.2 Hz, 2H), 4.00 (dd, J = 8.2, 6.6 Hz, 1H), 4.20 (ddt, J = 7.7, 6.3, 5.2 Hz, 1H), 4.54 (d, J = 12.2 Hz, 1H), 4.59 (d, J = 12.1 Hz, 1H), 7.24−7.35 (several peaks, 5H). ^{13}C NMR *trans*-**9p** δ 17.7, 33.2, 36.4, 72.9, 73.3, 75.3, 77.5, 127.5, 127.6, 128.3, 138.4. ^{1}H NMR *cis*-**9p** δ 1.04 (d, J = 6.6 Hz, 3H), 1.23 (dt, J = 12.2, 9.1 Hz, 1H), 2.11 (dt, J = 12.3, 6.7 Hz, 1H), 2.33 (m, 1H), 3.37 (t, J = 8.2 Hz, 1H), 3.50 (d, J = 5.1 Hz, 2H), 3.92 (t, J = 7.8 Hz, 1H), 4.13 (m, 1H), 4.56 (d, J = 12.2 Hz, 1H), 4.61 (d, J = 12.2 Hz, 1H), 7.24−7.35 (several peaks, 5H). ^{13}C NMR *cis*-**9p** δ 17.3, 34.1, 37.1, 73.0, 73.2, 74.8, 78.7, 127.5, 127.6, 128.3, 138.4. Anal. Calcd for $C_{13}H_{18}O_2$: C, 75.69; H, 8.80. Found: C, 75.47; H, 8.70. MS *m*/*z* (relative intensity) 206 (M^+, 6.9).

The following compounds were similarly prepared. As judged by ^{1}H NMR spectroscopy, the purity of the materials were >98%. For yields, *cis*/*trans*- and *syn*/*anti*-ratios, see Table 2.

3-Methylcyclopentanol (8) was compared with an authentic sample. For the assignment of the *cis* and *trans* isomers, the material was benzoylated by treatment with benzoyl chloride and pyridine in diethyl ether and analyzed by DEPT, INEPT, NOESY, PECOSY, HSQC, NOE difference, and ZTOCSY experiments. ^{1}H NMR *trans*-3-methylcyclopentanol benzoate δ 1.05 (d, J = 6.6 Hz, 3H), 1.18 (m, 1H), 1.49 (ddd, J = 14.0, 9.8, 6.2 Hz, 1H), 1.76−1.85 (several peaks, 2H), 2.00 (m, 1H), 2.15−2.30 (several peaks, 2H), 5.43 (m, 1H), 7.39−7.47 (several peaks, 2H), 7.54 (m, 1H), 8.03 (m, 2H). ^{1}H NMR *cis*-3-methylcyclopentanol benzoate δ 1.10 (d, J = 6.6 Hz, 3H), 1.35−1.44 (several peaks, 2H), 1.80−2.10 (several peaks, 4H), 3.32 (m, 1H), 5.37 (m, 1H), 7.39−7.47 (several peaks, 2H), 7.54 (m, 1H), 8.03 (m, 2H). ^{13}C NMR *trans*+*cis* δ 20.4, 21.0, 32.6, 32.6, 32.7, 32.7, 32.9, 33.0, 41.1, 41.6, 77.6, 77.8, 128.2, 129.4, 130.8, 132.6, 166.3, 166.3.

2-(3-Butenyl)-4-methyltetrahydrofuran (9l): ^{1}H NMR *trans*-**9l** δ 1.02 (d, J = 6.8 Hz, 3H), 1.55−1.72 (several peaks, 6H), 2.31 (m, 1H), 3.25 (dd, J = 8.4, 6.9 Hz, 1H), 3.94 (m, 1H), 3.98 (dd, 8.4, 6.8 Hz, 1H), 4.94 (ddt, J = 10.2, 2.0, 1.3 Hz, 1H), 5.02 (ddt, J = 17.1, 2.0, 1.6 Hz, 1H), 5.83 (ddt, J = 17.0, 10.0, 6.5 Hz, 1H). ^{1}H NMR *cis*-**9l** δ 1.03 (d, J = 6.7 Hz, 3H), 1.09 (dd, J = 11.8, 9.5 Hz, 1H), 2.10−2.18 (several peaks, 5H), 2.31 (m, 1H), 3.34 (t, J = 8.0 Hz, 1H), 3.87 (m, 2H), 4.94 (ddt, J = 10.2, 2.0, 1.3 Hz, 1H), 5.02 (ddt, J = 17.1, 2.0, 1.6 Hz, 1H), 5.83 (ddt, J = 17.0, 10.0, 6.5 Hz, 1H). ^{13}C NMR *cis*+*trans* δ 17.9, 18.1, 30.5, 30.6, 33.2, 34.4, 35.3, 39.6, 40.8, 74.4, 74.8, 78.2, 79.6, 114.4, 138.5. MS *m*/*z* (relative intensity) 140 (M^+, 0.8).

4-Methyl-2-phenyltetrahydrofuran (9m): ^{1}H NMR *trans*-**9m** δ 1.09 (d, J = 6.9 Hz, 3H), 1.91−2.04 (several peaks, 2H), 2.42 (m, 1H), 4.47 (dd, J = 8.2, 7.0 Hz, 1H), 4.21 (dd, J = 8.3, 6.5 Hz, 1H), 5.02 (dd, J = 7.3, 6.6 Hz, 1H), 7.21−7.35 (several peaks, 5H). ^{13}C NMR *trans*-**9m** δ 17.7, 34.9, 42.6, 75.6, 80.0, 125.4, 126.9, 128.2, 143.9. ^{1}H NMR *cis*-**9m** δ 1.09 (d, J = 5.0 Hz, 1H), 1.44 (dd, J = 10.1, 2.4 Hz, 1H), 2.43−2.54 (several peaks, 2H), 3.57 (t, J = 8.0 Hz, 1H), 4.08 (t, J = 8.1 Hz, 1H), 4.91 (dd, J = 9.7, 5.6 Hz, 1H), 7.21−7.35 (several peaks, 5H). ^{13}C NMR *cis*-**9m** δ 17.3, 33.2, 43.9, 75.4, 81.5, 125.5, 127.1, 128.2, 143.4. Rawal and co-workers[7] have published NMR data for the mixture of *cis* and *trans* isomers and Watanabe[15] and Tada[16] have described their synthesis.

2-Phenyl-4-isopropyltetrahydrofuran (9m′): ^{1}H NMR *trans*-**9m′** δ 0.90 (d, J = 7.2 Hz, 3H), 0.92 (d, J = 7.2 Hz, 3H), 1.60 (m, 1H), 1.97−2.06 (several peaks, 3H), 3.55 (m, 1H), 4.23 (m, 1H), 5.02 (m, 1H), 7.22−7.37 (several peaks, 5H). ^{1}H NMR *cis*-**9m′** δ 0.91 (d, J = 6.7 Hz, 3H), 0.94 (d, J = 6.7 Hz, 3H), 1.40−1.57 (several peaks, 2H), 2.15 (m, 1H), 2.41 (tt, J = 13.0, 5.7 Hz, 1H), 3.72 (t, J = 8.4 Hz, 1H), 4.11 (t, J = 8.2 Hz, 1H), 4.90 (dd, J = 10.0, 5.5 Hz, 1H), 7.22−7.37 (several peaks, 5H). ^{13}C NMR *cis*+*trans* δ 21.4, 21.5, 21.6, 21.6, 31.6, 32.0, 39.2, 40.8, 46.1, 48.2, 72.9, 73.1, 80.5, 81.7, 125.4, 125.6, 126.9, 127.1, 128.2, 128.3, 143.2, 144.2. Rawal and co-workers[7] have published NMR data for the mixture of *cis* and *trans* isomers.

2-[(Allyloxy)methyl]-4-methyltetrahydrofuran (9n): ^{1}H NMR *trans*-**9n** δ 1.02 (d, J = 6.7 Hz, 1H), 1.56 (ddd, J = 12.4, 7.7, 6.4 Hz, 1H), 1.83 (ddd, J = 12.4, 8.1, 6.3 Hz, 1H), 2.31 (ddt, J = 13.4, 8.2, 6.7 Hz, 1H), 3.30 (dd, J = 8.3, 7.0 Hz, 1H), 3.41 (d, J = 5.3 Hz, 2H), 3.92−4.02 (several peaks, 3H), 4.16 (ddt, J = 7.7, 6.2, 5.3 Hz, 1H), 5.16 (ddt, J = 10.3, 1.7, 1.3 Hz, 1H), 5.26 (dq, J = 17.2, 1.6 Hz, 1H), 5.91 (ddt, J = 17.2, 10.4, 5.6 Hz, 1H). ^{13}C NMR *trans*-**9n** δ 17.7, 33.1, 36.4, 72.3, 73.0, 75.3, 77.4, 116.9, 134.8. MS *m*/*z* (relative intensity) 157 (M^+ + 1, 0.8), 141 ($M^+ - CH_3$, 0.5), 125 (M^+ − allyl, <0.5), 85 ($M^+ - CH_2O$ − allyl, 100).

4-Methyl-2-(phenoxymethyl)tetrahydrofuran (9o): ^{1}H NMR *trans*-**9o** δ 1.06 (d, J = 6.7 Hz, 3H), 1.68 (ddd, J = 12.4, 7.8, 6.7 Hz, 1H), 1.98 (ddd, J = 12.4, 7.9, 5.9 Hz, 1H), 2.39 (m, 1H), 3.37 (dd, J = 8.3, 7.1 Hz, 1H), 3.91 (dd, J = 9.6, 4.8 Hz, 1H), 3.96 (dd, J = 9.6, 5.7 Hz, 1H), 4.04 (dd, J = 8.3, 6.6 Hz, 1H), 4.38 (m,1H), 6.89−6.96 (several peaks, 3H), 7.27 (m, 2H). ^{13}C NMR *trans*-**9o** δ 17.5, 33.2, 36.4, 70.4, 75.4, 76.7, 114.5, 120.7, 129.3, 158.8. ^{1}H NMR *cis*-**9o** δ 1.08 (d, J = 6.8 Hz, 3H), 1.36 (dd, J = 12.1, 9.2 Hz, 1H), 2.24 (dt, J = 12.4, 6.7 Hz, 1H), 2.39 (m, 1H), 3.42 (t, J = 8.2 Hz, 1H), 3.95−4.01 (several peaks, 3H), 4.31 (m, 1H), 6.89−6.96 (several peaks, 3H), 7.26 (m, 2H).

^{13}C NMR *cis*-**9o** δ 17.2, 34.2, 37.2, 70.6, 75.0, 77.8, 114.5, 120.7, 129.3, 158.8. MS *m*/*z* (relative intensity) 192 (M^+, 11.0).

2-(Phenoxymethyl)-4-isopropyltetrahydrofuran (9o′): ^{1}H NMR *trans*-**9o′** δ 0.89 (d, J = 7.2 Hz, 3H), 0.95 (d, J = 6.6 Hz, 3H), 1.51 (m, 1H), 1.76 (m, 1H), 1.91−2.06 (several peaks, 2H), 3.45 (t, J = 8.4 Hz, 1H), 3.87 (dd, J = 9.5, 5.1 Hz, 1H), 3.93 (dd, J = 9.6, 5.9 Hz, 1H), 4.07 (m, 1H), 4.33 (m, 1H), 6.90−6.96 (several peaks, 3H), 7.27 (m, 2H). ^{1}H NMR *cis*-**9o′** δ 0.90 (d, J = 7.0 Hz, 3H), 0.96 (d, J = 6.9 Hz, 3H), 1.40 (m, 1H), 1.53 (m, 1H), 2.04 (m, 1H), 2.18 (dddm, J = 11.9, 7.3, 5.9 Hz, 1H), 3.54 (dd, J = 9.0, 8.3 Hz, 1H), 3.97−4.05 (several peaks, 3H), 4.31 (m, 1H), 6.90−6.96 (several peaks, 3H), 7.27 (m, 2H). ^{13}C NMR *trans*+*cis* δ 21.5, 21.5, 21.6, 21.6, 31.6, 31.9, 33.2, 34.1, 46.3, 47.6, 70.5, 70.6, 72.5, 73.0, 77.2, 77.9, 114.5, 114.5, 120.8, 129.3, 129.4, 158.9. MS *m*/*z* (relative intensity) 220 (M^+, 14.2).

2-[(Benzyloxy)methyl]-4-isopropyltetrahydrofuran (9p′): ^{1}H NMR *trans*-**9p′** δ 0.87 (d, J = 6.6 Hz, 3H), 0.92 (d, J = 6.6 Hz, 3H), 1.46 (ddt, J = 13.2, 8.9, 6.6 Hz, 1H), 1.64 (dt, J = 12.3, 8.2 Hz, 1H), 1.82 (m, 1H), 1.92 (m, 1H), 3.39 (t, J = 8.6 Hz, 1H), 3.42 (dd, J = 9.9, 5.0 Hz, 1H), 3.45 (dd, J = 9.8, 5.8 Hz, 1H), 4.03 (dd, J = 8.5, 7.3 Hz, 1H), 4.15 (ddt, J = 8.2, 5.8, 4.9 Hz, 1H), 4.55 (d, 12.1 Hz, 1H), 4.59 (d, J = 12.0 Hz, 1H), 7.25−7.36 (several peaks, 5H). ^{13}C NMR *trans*-**9p′** δ 21.4, 21.5, 31.6, 33.1, 46.3, 72.8, 73.0, 73.3, 77.9, 127.5, 127.7, 128.3, 138.3. ^{1}H NMR *cis*-**9p′** δ 0.87 (d, J = 6.6 Hz, 3H), 0.92 (d, J = 6.6 Hz, 3H), 1.28 (m, 1H), 1.47 (m, 1H), 1.97 (m, 1H), 2.05 (m, 1H), 3.45−3.58 (several peaks, 3H), 3.95 (t, J = 7.9 Hz, 1H), 4.09 (m, 1H), 4.55 (d, J = 12.1 Hz, 1H), 4.59 (d, J = 12.0 Hz, 1H), 7.25−7.36 (several peaks, 5H). ^{13}C NMR *cis*-**9p′** δ 21.5, 21.6, 31.9, 34.0, 47.5, 72.3, 72.9, 72.9, 78.8, 127.4, 127.6, 128.2, 138.3. MS *m*/*z* (relative intensity) 234 (M^+, 5.3).

***cis*-3-Methyloctahydrobenzofuran (9q):** ^{1}H NMR *exo*-**9q** δ 0.98 (d, J = 7.4 Hz, 3H), 1.18−1.82 (several peaks, 9H), 2.04 (m, 1H), 3.32 (dd, J = 8.6, 5.6 Hz, 1H), 3.94 (m, 1H), 4.11 (t, J = 7.9 Hz, 1H). ^{1}H NMR *endo*-**9q** δ 0.92 (d, J = 7.3 Hz, 3H), 1.05−1.82 (several peaks, 8H), 1.95 (m, 1H), 2.41 (m, 1H), 3.44 (dd, J = 10.2, 7.9 Hz, 1H), 3.86−3.98 (several peaks, 2H). ^{13}C NMR *exo*+*endo* δ 11.5, 18.3, 20.5, 21.5, 21.9, 23.2, 24.5, 26.7, 28.6, 28.7, 37.7, 38.0, 41.3, 45.2, 72.3, 73.9, 76.3, 78.3. MS *m*/*z* (relative intensity) 140 (M^+, 50.8). This compound was previously prepared by Tada[16] and Torii.[22]

2-(3-Butenyl)-4-methylenetetrahydrofuran (10l): ^{1}H NMR δ 1.59 (ddt, J = 13.6, 9.5 Hz, 1H), 1.76 (m, 1H), 2.08−2.26 (several peaks, 3H), 2.65 (ddddd, J = 15.5, 5.9, 3.7, 1.8, 1.1 Hz, 1H), 3.95 (ddt, J = 8.6, 7.0, 5.9 Hz, 1H), 4.24 (dddd, J = 13.1, 4.7, 2.1, 1.1 Hz, 1H), 4.42 (m, 1H), 4.91 (m, 1H), 4.95−4.99 (several peaks, 2H), 5.04 (ddt, J = 17.1, 2.0, 1.6 Hz, 1H), 5.83 (ddt, J = 17.1, 10.2, 6.6 Hz, 1H). ^{13}C NMR δ 30.3, 34.3, 38.6, 70.7, 79.3, 104.0, 114.7, 138.2, 148.4. The high volatility of this material precluded an accurate determination of the yield.

2-Phenyl-4-methylenetetrahydrofuran (10m): ^{1}H NMR δ 2.56 (m, 1H), 2.95 (m, 1H), 4.40 (ddddd, J = 13.1, 4.7, 2.6, 1.1, 0.4 Hz, 1H), 4.58 (m, 1H), 4.95 (m, 1H), 4.97 (d, J = 6.1 Hz, 1H), 5.02 (m, 1H), 7.25−7.37 (several peaks, 5H). ^{13}C NMR δ 41.1, 71.3, 81.1, 104.3, 125.9, 127.6, 128.4, 141.8, 147.9. MS *m*/*z* (relative intensity) 160 (M^+, 23.2). This compound has previously been prepared by Tada.[16,21]

2-[(Allyloxy)methyl]-4-methylenetetrahydrofuran (10n): ^{1}H NMR δ 2.37 (ddddd, J = 15.7, 8.1, 3.5, 2.2, 1.0 Hz, 1H), 2.61 (ddddd, J = 15.7, 6.6, 3.7, 1.7, 1.0 Hz, 1H), 3.50 (d, J = 5.3 Hz, 2H), 4.01 (dddd, J = 12.9, 5.7, 1.5, 1.3 Hz, 1H), 4.03 (dddd, J = 12.8, 5.6, 1.6, 1.3 Hz, 1H), 4.18 (ddt, J = 8.0, 6.6, 5.3 Hz, 1H), 4.27 (dddd, J = 13.0, 4.6, 2.1, 1.1 Hz, 1H), 4.40 (ddddd, J = 13.1, 3.3, 2.0, 1.0, 0.4 Hz, 1H), 4.91 (dddd, J = 4.8, 2.7, 1.7, 0.4 Hz, 1H), 4.98 (dddd, J = 4.8, 2.4, 1.9, 0.5 Hz, 1H), 5.17 (ddt, J = 10.3, 1.8, 1.2 Hz, 1H), 5.27 (dq, J = 17.2, 1.6 Hz, 1H), 5.91 (ddt, J = 17.2, 10.3, 5.6 Hz, 1H). ^{13}C NMR δ 35.1, 71.2, 72.0, 72.4, 78.5, 104.4, 117.1, 134.6, 147.5. MS *m*/*z* (relative intensity) 154 (M^+, 5.0), 113 (M^+ − allyl, 14.9).

2-(Phenoxymethyl)-4-methylenetetrahydrofuran (10o): ^{1}H NMR δ 2.50 (ddddd, J = 15.6, 5.5, 3.3, 2.3, 1.0 Hz, 1H), 2.73 (ddddd, J = 15.7, 6.8, 3.0, 2.0, 1.0 Hz, 1H), 3.99 (dd, J = 9.8, 4.7 Hz, 1H), 4.04 (dd, J = 9.8, 5.9 Hz, 1H), 4.34 (dddd, J = 13.0, 4.3, 2.1, 1.0 Hz, 1H), 4.39 (m, 1H), 4.46 (dm, J = 13.4 H, 1H), 4.96 (dt, J = 4.6, 2.2 Hz, 1H), 5.03 (dt, J = 4.4, 2.3 Hz, 1H), 6.90−6.97 (several peaks, 3H), 7.26 (m, 2H). ^{13}C NMR δ 35.2, 69.7, 71.3, 77.6, 104.8, 114.5, 120.9, 129.4, 147.0, 158.7. MS *m*/*z* (relative intensity) 190 (M^+, 14.2).

2-[(Benzyloxy)methyl]-4-methylenetetrahydrofuran (10p): ^{1}H NMR δ 2.38 (ddddd, J = 15.7, 5.6, 3.5, 2.3, 1.0 Hz, 1H), 2.61 (ddddd, J = 15.7, 6.6, 2.9, 2.0, 1.1 Hz, 1H), 3.51 (d, J = 14.4 Hz, 1H), 3.56 (d, J = 14.4 Hz, 1H), 4.21 (ddt, J = 8.0, 6.6, 5.2 Hz, 1H), 4.28 (dddd, J = 13.1, 4.5, 2.1, 1.0 Hz, 1H), 4.42 (dm, J = 13.1, Hz, 1H), 4.57 (d, J = 12.0 Hz, 1H), 4.61 (d, J = 12.0 Hz, 1H), 4.92 (quint, J = 2.3 Hz, 1H), 4.99 (quint, J = 2.3 Hz, 1H), 7.26−7.37 (several peaks, 5H). ^{13}C NMR δ 35.2, 71.2, 72.0, 73.4, 78.5, 104.4, 127.6, 127.7, 128.3, 138.2, 147.5. MS *m*/*z* (relative intensity) 203 (M^+ − 1, 2.1), 126 (M^+ − C_6H_5, 3.0), 91 ($C_6H_5CH_2^+$, 100).

***cis*-3-Methyleneoctahydrobenzofuran (10q):** ^{1}H NMR δ 1.20−1.81 (several peaks, 8H), 2.53 (m, 1H), 3.98 (q, J = 4.7 Hz, 1H), 4.30 (dddd, J = 13.4, 2.6, 2.2, 1.0 Hz, 1H), 4.47 (dm, J = 13.4 Hz, 1H), 4.85 (q, J = 2.0 Hz, 1H), 4.90 (ddd, J = 2.6, 2.6, 1.6 Hz, 1H). ^{13}C NMR δ 21.3, 23.1, 27.1, 27.8, 43.4, 69.8, 77.2, 77.9, 102.5. MS *m*/*z* (relative intensity) 138 (M^+, 9.6). This compound was previously described by Tada[16,21] and Torii.[22]

4-Methyl-3-phenyltetrahydrofuran (11): ^{1}H NMR *trans*-**11** δ 1.03 (d, J = 6.8 Hz, 3H), 2.38 (m, 1H), 2.88 (m, 1H), 3.48 (dd, J = 8.9, 8.3 Hz, 1H), 3.82 (t, J = 8.7 Hz, 1H), 4.20 (m, 2H), 7.18−7.34 (several peaks, 5H). ^{1}H NMR *cis*-**11** δ 0.68 (d, J = 7.0 Hz, 3H), 2.63 (m, 1H), 3.39 (m, 1H), 3.53 (t, J = 8.0 Hz, 1H), 4.06 (dd, J = 8.2, 7.3 Hz, 1H), 4.13 (dd, J = 8.6, 4.7 Hz, 1H), 4.19 (dd, J = 8.6, 6.5 Hz, 1H), 7.18−7.34 (several peaks, 5H). ^{13}C NMR *trans*+*cis* δ 13.3, 15.5, 38.0, 42.6, 48.7, 53.6, 73.0, 73.9, 75.2, 75.5, 126.3, 126.6, 127.5, 128.1, 128.3, 128.5, 140.1, 140.9. MS *m*/*z* (relative intensity) 162 (M^+, 21.7). This compound has previously been described by Ono.[12]

3-Phenyl-4-isopropyltetrahydrofuran (12): ^{1}H NMR *trans*-**12** δ 0.83 (d, J = 6.8 Hz, 3H), 0.88 (d, J = 6.8 Hz, 3H), 1.69 (m, 1H), 2.19 (dt, J = 15.8, 8.0 Hz, 1H), 3.08 (q, J = 7.5 Hz, 1H), 3.65 (t, J = 8.2 Hz, 1H), 3.73 (dd, J = 8.7, 7.4 Hz, 1H), 4.07−4.19 (several peaks, 2H), 7.17−7.33 (several peaks, 5H). ^{1}H NMR *cis*-**12** δ 0.76 (d, J = 6.6 Hz, 3H), 0.89 (d, J = 6.5 Hz, 3H), 1.07 (m, 1H), 2.22 (ddd, J = 19.5, 11.0, 7.3 Hz, 1H), 3.32 (m, 1H), 3.69 (dd, J = 10.9, 8.3 Hz, 1H), 4.02 (dd, J = 8.5, 1.2 Hz, 1H), 4.07 (t, J = 8.3 Hz, 1H), 4.16 (dd, J = 8.5, 5.4 Hz, 1H), 7.17−7.33 (several peaks, 5H). ^{13}C NMR *trans*+*cis* δ 20.4, 21.6, 21.7, 22.0, 27.3, 31.1, 47.8, 50.1, 52.7, 54.8, 70.9, 72.5, 76.1, 76.6, 126.2, 126.3, 127.6, 128.2, 128.5, 128.7, 141.9, 143.6. MS *m*/*z* (relative intensity) 190 (M^+, 18.3).

4-Methylene-3-phenyltetrahydrofuran (13): ^{1}H NMR δ 3.80−3.89 (several peaks, 2H), 4.28 (m, 1H), 4.46−4.56 (several peaks, 2H), 4.77 (m, 1H), 5.05 (m, 1H), 7.21−7.27 (several peaks, 3H), 7.32 (m, 2H). ^{13}C NMR δ 50.6, 72.0, 76.0, 106.0, 126.7, 128.2, 128.6, 141.0, 152.3. MS *m*/*z* (relative intensity) 160 (M^+, 24.1).

2-Ethoxy-4-methyltetrahydrofuran (15a): ^{1}H NMR *trans*-**15a** δ 1.07 (d, J = 6.5 Hz, 3H), 1.19 (t, J = 7.1 Hz, 3H), 1.44 (m, 1H), 2.19−2.31 (several peaks, 2H), 3.42 (t, J = 8.4 Hz, 1H), 3.75 (q, J = 7.2 Hz, 2H), 3.91 (m, 1H), 5.11 (m, 1H). ^{13}C NMR *trans*-**15a** δ 15.3, 17.2, 32.9, 40.8, 63.1, 73.2, 104.7. ^{1}H NMR *cis*-**15a** δ 1.02 (d, J = 6.7 Hz, 3H), 1.18 (t, J = 7.1 Hz, 3H), 1.55 (ddd, J = 12.9, 8.4, 5.2 Hz, 1H), 2.01 (m, 1H), 2.49 (m, 1H), 3.38 (dd, J = 8.2, 6.6 Hz, 1H), 3.72 (q, J = 7.2 Hz, 2H), 4.04 (t, J = 7.5 Hz, 1H), 5.11 (m, 1H). ^{13}C NMR *cis*-**15a** δ 14.0, 18.4, 31.5, 41.1, 62.6, 73.9, 104.2. MS *m*/*z* (relative intensity) 129 (M^+, 23.8).

2-*n*-Butoxy-4-methyltetrahydrofuran (15b): ^{1}H NMR *trans*-**15b** δ 0.91 (t, J = 7.4 Hz, 3H), 1.07 (d, J = 6.7 Hz, 3H), 1.30−1.40 (several peaks, 2H), 1.44 (m, 1H), 1.48−1.58 (several peaks, 2H), 2.19−2.29 (several peaks, 2H), 3.36 (dt, J = 9.5, 6.7 Hz, 1H), 3.40 (t, J = 8.4 Hz, 1H), 3.67 (dt, J = 9.5, 6.8 Hz, 1H), 3.92 (m, 1H), 5.09 (dd, J = 5.4, 3.1 Hz, 1H). ^{13}C NMR *trans*-**15b** δ 13.9, 17.4, 19.4, 31.9, 32.8, 40.8, 67.5, 73.2, 104.8. ^{1}H NMR *cis*-**15b** δ 0.90 (t, J = 7.4 Hz, 3H), 1.02 (d, J = 6.9 Hz, 3H), 1.30−1.40 (several peaks, 2H), 1.48−1.58 (several peaks, 3H), 2.00 (m, 1H), 2.47 (m, 1H), 3.35 (m, 2H), 3.63 (dd, J = 11.9, 9.6 Hz, 1H), 4.02 (dd, J = 8.0, 7.4 Hz, 1H), 5.08 (m,-1H). ^{13}C NMR *cis*-**15b** δ 13.9, 18.4, 19.4, 31.5, 31.8, 41.1, 67.0,

73.9, 104.3. MS *m*/*z* (relative intensity) 157 ($M^+ - 1$, 1.0), 101 ($M^+ - Bu$, 2.6), 85 ($M^+ - BuO$, 97.4).

2-*t*-Butoxy-4-methyltetrahydrofuran (15c): ^{1}H NMR *trans*-**15c** δ 1.05 (d, $J = 6.3$ Hz, 3H), 1.22 (s, 9H), 1.40 (m, 1H), 2.14−2.27 (several peaks, 2H), 3.46 (t, $J = 8.2$ Hz, 1H), 3.86 (m, 1H), 5.38 (m, 1H). ^{13}C NMR *trans*-**15c** δ 16.9, 28.9, 33.1, 42.0, 73.1, 73.9, 99.6. ^{1}H NMR *cis*-**15c** δ 1.00 (d, $J = 6.7$ Hz, 3H), 1.21 (s, 9H), 1.57 (ddd, $J = 12.6$, 8.4, 5.4 Hz, 1H), 1.92 (ddd, $J = 12.7$, 7.3, 1.7 Hz, 1H), 2.49 (m, 1H), 3.31 (dd, $J = 8.1$, 6.5 Hz, 1H), 4.07 (dd, $J = 7.9$, 7.3 Hz, 1H), 5.39 (m, 1H). ^{13}C NMR *cis*-**15c** δ 18.3, 28.9, 31.7, 42.2, 73.6, 73.7, 99.0. MS *m*/*z* (relative intensity) 157 ($M^+ - 1$, <0.5), 143 ($M^+ - CH_3$, 1.5).

***cis*-7-Methyl-2,9-dioxabicyclo[4.3.0]nonane (18)** ^{1}H NMR *endo*-**18** δ 0.95 (d, $J = 7.0$ Hz, 3H), 1.39 (m, 1H), 1.53−1.72 (several peaks, 3H), 1.88 (m, 1H), 2.42 (m, 1H), 3.59 (dd, $J = 9.7$, 8.0 Hz, 1H), 3.60 (m, 1H), 3.73 (m, 1H), 3.93 (t, $J = 7.9$ Hz, 1H), 5.26 (d, $J = 3.9$ Hz, 1H). ^{13}C NMR *endo*-**18** δ 11.6, 19.4, 23.1, 35.1, 37.8, 61.1, 71.6, 102.2. ^{1}H NMR *exo*-**18** δ 1.02 (d, $J = 6.6$ Hz, 3H), 1.18−1.41 (several peaks, 2H), 1.59−1.70 (several peaks, 2H), 1.83 (m, 1H), 2.38 (m, 1H), 3.38−3.49 (several peaks, 2H), 3.87 (m, 1H), 4.28 (t, $J = 8.2$ Hz, 1H), 5.00 (d, $J = 3.4$ Hz, 1H). MS *m*/*z* (relative intensity) 142 (M^+, 11.4). This compound has previously been described by Torii.[51]

Et_3B-Mediated Reductive Radical Cyclizations. Except that 1 equiv of triethylborane (1 M in hexane) was used as radical initiator instead of AIBN, these cyclizations were carried out as described in the typical procedure for reductive radical cyclization. Benzene was replaced by toluene for reactions carried out at lower than ambient temperatures.

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Supporting Information Available: ^{1}H NMR spectra of compounds **1a**−**j**, **1l**, **1n**−**p**, **5a**−**d**, **6d**−**k**, **7c**, **7e**−**j**, **9l**, **9n**−**q**, **10m**−**q**, **11**−**13**, **14a**−**c**, **15a**−**c**, **17**, **18** (56 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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(51) Torii, S.; Inokuchi, T.; Yukawa, T. *J. Org. Chem.* **1985**, *50*, 5875.