Catalytic Oxyselenenylation–Deselenenylation Reactions of Alkenes – Stereoselective One-Pot Conversion of 3-Alkenols into 2,5-Dihydrofurans

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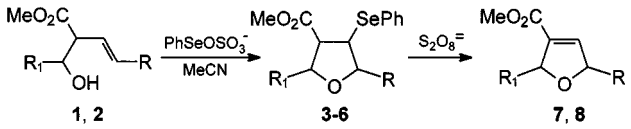
A stereoselective multi-step one-pot procedure for converting 2-methoxycarbonyl-3-alkenols into 3-methoxycarbonyl-2,5-dihydrofurans, using only catalytic amounts of diphenyl diselenide and an excess of ammonium persulfate, has been developed. The *erythro* alkenols give the *trans* dihydrofurans, while the *threo* stereoisomers give the corresponding *cis* products. The configurations of the starting alkenols were deduced from those of the intermediate phenylseleno tetrahydrofurans, which were independently obtained from reactions of the alkenols with stoichiometric amounts of phenylselenenyl sulfate.

We have recently reported that the strongly electrophilic phenylselenenyl sulfate, formed by oxidation of diphenyl diselenide with ammonium persulfate, can be employed in several addition reactions to alkenes with considerable advantages over other phenylselenenylating agents.[1−4] The oxidation of diphenyl diselenide is the easiest way to produce a phenylselenenylating agent which does not contain a nucleophilic counter anion. The use of this kind of reagents avoids the formation of secondary products containing halogens which are observed when phenylselenenyl chloride or bromide are employed and increases the regioselectivity of the addition reactions. We have also observed that upon further reaction with ammonium persulfate the initially formed alkyl phenyl selenides undergo deselenenylation, with regeneration of phenylselenenyl sulfate.[5] This has allowed us to develop some multi-step one-pot procedures based on selenenylation of unsaturated compounds with phenylselenenyl sulfate followed by deselenenylation of the addition products with ammonium persulfate. The entire process can thus be effected simply by using an excess of ammonium persulfate and only catalytic amounts of diphenyl diselenide.[5−7]

Depending on the solvent employed and on the structure of the starting unsaturated compound, the deselenenylation process can lead either to substitution[8] or to elimination products.[5,6,9] In the latter case, the observed course of the reaction has been attributed to the presence of an electron-withdrawing group in the α-position of the starting compound, which allows formation of a conjugated alkene in the elimination reaction. Several examples of these catalytic oxyselenenylation−deselenenylation reactions of alkenes have been described. Thus, β,γ-unsaturated esters, amides, and nitriles are efficiently converted into γ-alkoxy- or γ-hydroxy α,β-unsaturated derivatives.[5] Similarly, β,γ-unsaturated acids give rise to butenolides,[6] while β,γ-unsaturated ketones give substituted furans.[9]

We now report that the peculiar behaviour of the reagent formed from diphenyl diselenide and ammonium persulfate can be employed to effect a very efficient and stereoselective one-pot conversion of 2-methoxycarbonyl-3-alkenols **1**, **2** into 3-methoxycarbonyl-2,5-dihydrofurans **7**, **8**. As indicated in Scheme 1, the reaction proceeds through the intermediate formation of the corresponding phenylseleno tetrahydrofurans **3**−**6**.



1, **2** **3-6** **7**, **8**

Scheme 1

The 2-methoxycarbonyl-3-alkenols required for the present investigation were easily obtained from commercially available 3-alkenoic acids according to a literature procedure.[10] As indicated in Scheme 2, single products, **1a** and **1b**, were obtained from the reactions of *trans*-3-hexenoic acid with acetone and benzophenone, respectively. The reactions of *trans*-3-hexenoic, vinylacetic, and *trans*-3-pentenoic acids with benzaldehyde, as well as that of *trans*-styrylacetic acid with acetaldehyde, afforded mixtures of the *erythro* and *threo* stereoisomers, **1c**−**1f** and **2c**−**2f**. In these cases, the ratios of the two stereoisomers were determined by GC-MS and/or ^{1}H-NMR analysis of the crude reaction mixtures. Pure products were then obtained by column chromatography on silica gel (Table 1).

An initial indication for assigning the *erythro* and *threo* configurations was provided by the values of the vicinal coupling constants between the protons in the 1- and 2-positions. These coupling constants are generally smaller in the *erythro* than in the *threo* stereoisomers.[11] In the present case, ^3J values of 6.1, 6.3, 5.2, and 4.9 Hz were measured for the minor isomers **1c**−**f** (which were eluted first from

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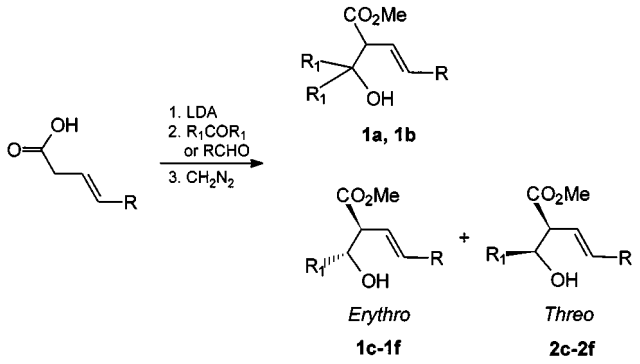
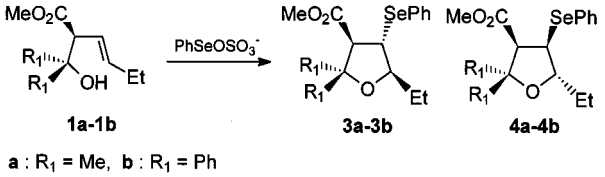


Scheme 2

Table 1. Synthesis of 2-methoxycarbonyl-3-alkenols **1a**−**1f** and **2c**−**2f**

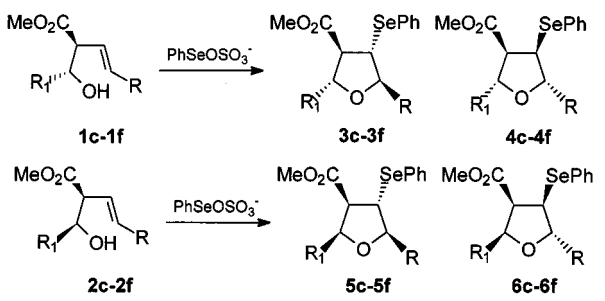
Acids	Ketones or Aldehydes	2-Methoxycarbonyl-3-alkenols		Yield (%)	1/2 Ratio
trans-3-Hexenoic	Me_2CO	**1a**		84	
trans-3-Hexenoic	Ph_2CO	**1b**		87	
trans-3-Hexenoic	PhCHO	**1c**	**2c**	95	36/64
Vinylacetic	PhCHO	**1d**	**2d**	91	35/65
trans-3-Pentenoic	PhCHO	**1e**	**2e**	93	40/60
trans-Styrylacetic	MeCHO	**1f**	**2f**	85	45/55

the chromatographic column), as opposed to values of 8.4, 8.4, 8.5, and 6.4 Hz for the major isomers **2c**−**f**. In order to confirm the proposed structural assignments, the starting alkenols were subjected to seleno etherification, so as to generate the corresponding phenylseleno tetrahydrofuran derivatives, the structures of which could be determined more easily. This was achieved by refluxing the 2-methoxycarbonyl-3-alkenols with a stoichiometric amount of diphenyl diselenide and ammonium persulfate for 3 h in acetonitrile. The first experiments were carried out on the alkenols **1a** and **1b**. Two stereoisomeric tetrahydrofurans were obtained from each alkenol (Scheme 3 and Table 2). It is well documented that seleno etherification reactions of alkenols in general and, in particular, those of homoallylic alcohols are stereospecific *anti* additions.[12] The two stereoisomeric tetrahydrofurans formed in the ring-closure reactions of **1a** and **1b** can therefore be assigned the structures **3a**, **4a** and **3b**, **4b**, respectively. The two stereoisomers were formed in a 93:7 ratio in the first case and in a 91:9 ratio in the second, indicating that the attack of the electrophilic selenenylating agent on the carbon−carbon double bond occurs with good facial selectivity. The observed selectivity reflects the steric demand in the approach of the electrophile to the π-bond, which preferentially occurs away from the methoxycarbonyl substituent.[12] Hence, the major isomers should be **3a** and **3b** and the minor isomers should have the structures **4a** and **4b**. NOE difference experiments confirmed the proposed structures. In fact, irradiation of H-3 produced a positive NOE on H-5 in compounds **3a** and **3b** and on H-4 in compounds **4a** and **4b**.



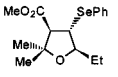
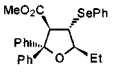
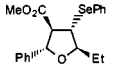
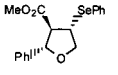
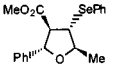
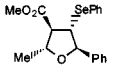
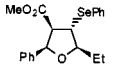
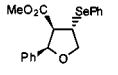
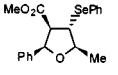
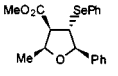
Scheme 3

In the case of **3a** and **4a**, the methyl protons of the carbomethoxy group have normal ^{1}H-NMR chemical shifts of δ = 3.65 and 3.8, whereas in **3b** and **4b** these protons are considerably shielded, resonating at δ = 3.15 and 3.30, respectively. This can be attributed to the phenyl group in the 5-position, which is *cis*-oriented in relation to the methoxycarbonyl group.[13] This observation allowed us to assign *erythro* and *threo* configurations to the 3-alkenols **1c**−**1e** and **2c**−**2e**. In these cases, each alkenol also gave a mixture of two tetrahydrofurans, **3c**−**3e**, **4c**−**4e** and **5c**−**5e**, **6c**−**6e**, respectively (Scheme 4), and the ring-closure reaction occurred with high selectivity (Table 2). In all cases, the ratio of the two stereoisomers was determined by GC-MS and/or ^{1}H-NMR analysis. Pure products were then obtained by column chromatography on silica gel. For the reasons discussed above, the major isomers were **3c**−**3e** and **5c**−**5e**, in which the methoxycarbonyl and phenylseleno groups are *trans* to one another. Shielding of the methyl protons of the carbomethoxy group was observed only in compounds **5c**−**5e**, **6c**−**6e**. It follows that the 3-alkenols **2c**−**2e** must have the *threo* configuration. Finally, the configurations of **1f** and **2f** were assigned by comparison of the ^{1}H-NMR spectra of **5f** and **6f** with those of the other tetrahydrofurans. The proposed structures were further confirmed by the results of NOE difference and/or NOESY experiments. The following dipolar connectivities were measured: compounds **3c-3f**, H-3, H-5 and H-2, H-4; compounds **4c-4f**, H-3, H-4 and H-2, H-5; compounds **5c-5f**, H-2, H-3, H-5; compounds **6c-6f**, H-2, H-3, H-4.



Scheme 4

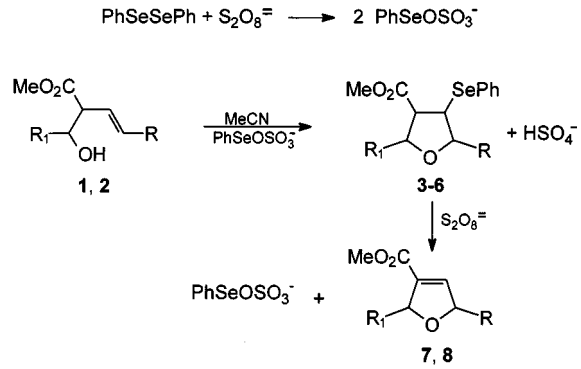
Table 2. Synthesis of methyl 4-(phenylseleno)tetrahydro-3-furancarboxylates

Entry	Alkenols	Tetrahydrofurans		Yield (%)	3/4 and 5/6 Ratios
1	1a		3a, 4a	94	93/7
2	1b		3b, 4b	96	91/9
3	1c		3c, 4c	87	92/8
4	1d		3d, 4d	78	92/8
5	1e		3e, 4e	80	93/7
6	1f		3f, 4f	85	95/5
7	2c		5c, 6c	86	95/5
8	2d		5d, 6d	87	92/8
9	2e		5e, 6e	82	93/7
10	2f		5f, 6f	86	95/5

Having established the configurations of the starting 2-methoxycarbonyl-3-alkenols, we then effected their one-pot selenium-catalyzed conversion into 3-methoxycarbonyl-2,5-dihydrofurans. These reactions were simply carried out by refluxing a mixture of the alkenol (0.5 mmol), diphenyl diselenide (0.05 mmol), and ammonium persulfate (3 mmol) in acetonitrile (30 mL) for 3−5 h. The reaction mixture was subsequently worked-up in the usual way and the 2,5-dihydrofurans were obtained in pure form by column chromatography on silica gel. Most of the diphenyl diselenide was also recovered.

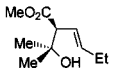
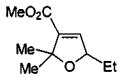
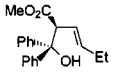
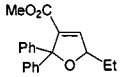
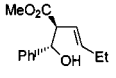
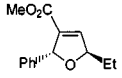
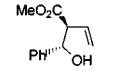
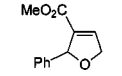
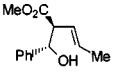
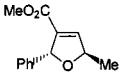
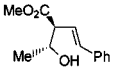
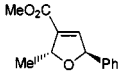
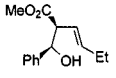
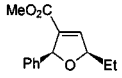
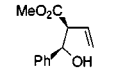
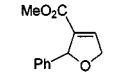
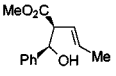
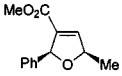
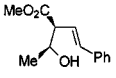
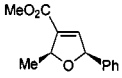
The proposed course of these reactions is indicated in Scheme 5. The phenylselenenyl sulfate, produced by reaction of diphenyl diselenide with ammonium persulfate, reacts with the alkenols **1**, **2** to give the seleno tetrahydrofurans **3**−**6**. Reaction of these compounds with ammonium persulfate affords the observed products **7**, **8** and at the same time regenerates the phenylselenenyl sulfate, which restarts the catalytic cycle.

As indicated in Table 3, excellent yields of the 3-methoxycarbonyl-2,5-dihydrofurans were obtained in all cases. The cyclization−elimination processes of those 3-alkenols capable of giving rise to stereoisomeric 2,5-dihydrofurans (entries 3, 5, 6, 7, 9, and 10) proceeded with high stereoselectivity. Thus, the *erythro* alkenols **1c**, **1e**, and **1f** gave the *trans* 2,5-dihydrofurans **7c**, **7e**, and **7f**, whereas the *threo*



Scheme 5

Table 3. Catalytic one-pot conversion of 3-alkenols into 2,5-dihydrofurans

Entry	Alkenols	2,5-Dihydrofurans	Yield (%)
1	 1a	 7a	90
2	 1b	 7b	92
3	 1c	 7c	95
4	 1d	 7d	94
5	 1e	 7e	96
6	 1f	 7f	91
7	 2c	 8c	89
8	 2d	 7d	91
9	 2e	 8e	96
10	 2f	 8f	96

alkenols **2c**, **2e**, and **2f** gave the *cis* derivatives **8c**, **8e**, and **8f**. Proton−proton and proton−carbon correlation spectra were recorded in order to assign the observed resonance frequencies to the three ring protons. The structures of the 3-methoxycarbonyl-2,5-dihydrofurans were assigned on the basis of the results of differential NOE experiments. Irradiation of H-2 produced a positive NOE on H-5 in the *cis* derivatives **8c**, **8e**, and **8f** and on the proton of the methylene, methyl, and phenyl groups in **7c**, **7e**, and **7f**, respectively. The observed stereoselectivity of the process implies

that of the two stereoisomeric tetrahydrofurans formed as intermediates, only the major isomers **3c**, **3e**, **3f**, **5c**, **5e**, and **5f** (Scheme 4) undergo elimination to afford the corresponding 2,5-dihydrofurans. This was confirmed by independent experiments. In fact, upon reaction with ammonium persulfate, these compounds were quantitatively transformed into the dihydrofurans. Under the same reaction conditions, the minor isomers **4c**, **4e**, **4f**, **6c**, **6e**, and **6f** gave rise to unidentified products. It is possible that in these cases the *cis* relationship between the phenylseleno and methoxycarbonyl groups renders the approach of the persulfate anion to the selenium atom more difficult, such that the reaction takes a completely different course.

In conclusion, the selenium-catalyzed stereoselective multi-step one-pot procedure described in this paper represents a very simple and convenient method for the synthesis of 3-methoxycarbonyl-2,5-dihydrofurans starting from readily available 2-methoxycarbonyl-3-alkenols. Moreover, the results presented here represent a further example of the conceptual and synthetic importance of the diphenyl diselenide/ammonium persulfate reagent, which is the only reagent that permits the selenenylation−deselenenylation sequence (addition−substitution or addition−elimination) to be effected in one-pot and with only catalytic amounts of the selenenylating agent. This procedure is presently being employed by ourselves[14] as well as by other research groups[15,16] to effect asymmetric syntheses. Starting from alkenes and using a chiral, non-racemic diselenide and ammonium persulfate, the selenenylation−deselenenylation sequence proceeds with moderate to good enantioselectivities.

Experimental Section

New compounds were characterized by MS, and by ^{1}H- and ^{13}C-NMR spectroscopy. − GC-MS analyses were carried out with an HP 5890 gas chromatograph (dimethyl silicone column, 12.5 m) equipped with an HP 5971 mass-selective detector. − ^{1}H- and ^{13}C-NMR spectra were recorded at 200 and 50.32 MHz, respectively, on a Bruker AC 200 instrument; unless otherwise specified, $CDCl_3$ was used as the solvent and TMS as internal standard. − Elemental analyses were carried out on a Carlo Erba 1106 elemental analyzer.

Synthesis of the 2-Methoxycarbonyl-3-alkenols. − General Procedure: Under nitrogen, 40 mL of a 1.6 M solution of *n*-butyllithium (64 mmol) in hexane was added dropwise to a solution of diisopropylamine (64 mmol) in THF (40 mL) at 0°C. After 15 min., the solution was cooled to −78°C and after a further 0.5 h a solution of *trans*-3-hexenoic, *trans*-3-pentenoic, *trans*-styrylacetic, or vinylacetic acid (30 mmol) in THF (20 mL) was slowly added. The resulting yellow solution was stirred for 2 h. A solution of acetone, benzophenone, benzaldehyde, or acetaldehyde (30 mmol) in THF (20 mL) was then added and the mixture was stirred at −78°C for 6 h. The reaction mixture was subsequently poured into aqueous ammonium chloride solution and extracted with diethyl ether. The combined organic extracts were washed with aqueous sodium carbonate solution, dried over sodium sulfate, filtered, and then treated with an excess of diazomethane. The solution was then concentrated and the residue was chromatographed on a silica gel column, eluting with a 1:9 mixture of diethyl ether and light petroleum. The products obtained, the yields, and the isomeric ratios are collected in Table 1. Physical and spectral data are reported below.

Methyl (*E*)-2-(1-Hydroxy-1-methylethyl)-3-hexenoate (**1a**): Oil. − ^{1}H NMR: δ = 5.7−5.5 (m, 2 H), 3.68 (s, 3 H), 3.3 (br. s, 1 H), 3.0 (d, 1 H, *J* = 8.6 Hz), 2.08 (dq, 2 H, *J* = 5.1 and 7.5 Hz), 1.21 (s, 3 H), 1.18 (s, 3 H), 1.0 (t, 3 H, *J* = 7.5 Hz). − ^{13}C NMR: δ = 174.3, 137.5, 123.1, 71.0, 59.1, 51.4, 28.3, 26.1, 25.3, 13.2. − MS; *m*/*z* (relative intensity): 171 (6), 128 (100), 113 (96), 96 (23), 81 (43), 74 (31), 68 (31), 59 (68), 43 (96). − $C_{10}H_{18}O_3$: calcd. C 64.49, H 9.74; found C 64.55, H 9.53.

Methyl (*E*)-2-[Hydroxy(diphenyl)methyl]-3-hexenoate (**1b**): M.p. 125−126°C. − ^{1}H NMR: δ = 7.6−7.0 (m, 10 H), 5.6−5.3 (m, 2 H), 4.9 (br. s, 1 H), 4.12 (d, 1 H, *J* = 7.3 Hz), 3.6 (s, 3 H), 1.85 (dq, 2 H, *J* = 5.8 and 7.3 Hz), 0.75 (t, 3 H, *J* = 7.3 Hz). − ^{13}C NMR: δ = 175.3, 147.0, 144.0, 138.3, 128.2, 127.7, 126.9, 126.4, 125.8, 125.5, 122.1, 78.7, 56.5, 52.0, 25.5, 13.2. − MS; *m*/*z* (relative intensity): 182 (41), 152 (2), 105 (100), 77 (55), 51 (23). − $C_{20}H_{26}O_3$: calcd. C 76.40, H 8.33; found C 76.36, H 8.64.

Methyl *erythro*-(*E*)-2-(1-Hydroxy-1-phenylmethyl)-3-hexenoate (**1c**): Oil. − ^{1}H NMR: δ = 7.34 (s, 5 H), 5.6−5.5 (m, 2 H), 4.9 (d, 1 H, *J* = 5.2 Hz), 3.58 (s, 3 H), 3.23 (ddd, 1 H, *J* = 2.0, 3.3 and 6.0 Hz), 2.95 (br. s, 1 H), 2.1−1.95 (m, 2 H), 0.95 (t, 3 H, *J* = 7.4 Hz). − ^{13}C NMR: δ = 173.3, 141.0, 138.7, 128.0, 127.6, 126.4, 122.2, 74.1, 57.4, 51.7, 25.6, 13.3. − MS; *m*/*z* (relative intensity): 157 (1), 128 (100), 113 (45), 107 (18), 105 (13), 97 (7), 96 (14), 81 (18), 77 (24), 68 (12), 41 (8). − $C_{14}H_{18}O_3$: calcd. C 71.77, H 7.74; found C 71.80, H 7.46.

Methyl *threo*-(*E*)-2-(1-Hydroxy-1-phenylmethyl)-3-hexenoate (**2c**): Oil. − ^{1}H NMR: δ = 7.3 (s, 5 H), 5.5−5.2 (m, 2 H), 4.85 (dd, 1 H, *J* = 4.0 and 8.5 Hz), 3.7 (s, 3 H), 3.33 (t, 2 H, *J* = 8.0 Hz), 3.1 (d, 1 H, *J* = 4.0 Hz), 1.88 (dq, 2 H, *J* = 5.0 and 7.4 Hz), 0.8 (t, 3 H, *J* = 7.4 Hz). − ^{13}C NMR: δ = 173.9, 141.4, 137.4, 128.3, 127.6, 126.6, 122.6, 75.5, 57.0, 51.8, 25.3, 13.0. − MS; *m*/*z* (relative intensity): 157 (1), 128 (100), 113 (47), 107 (18), 105 (13), 96 (16), 81 (20), 77 (24), 68 (13), 41 (8). − $C_{14}H_{18}O_3$: calcd. C 71.77, H 7.74; found C 71.88, H 7.92.

Methyl *erythro*-2-(1-Hydroxy-1-phenylmethyl)-3-butenoate (**1d**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 5.9 (ddd, 1 H, *J* = 8.9, 10.1 and 17.1 Hz), 5.2 (dd, 1 H, *J* = 1.0 and 10.1 Hz), 5.08 (dd, 1 H, *J* = 1.0 and 17.1 Hz), 4.95 (d, 1 H, *J* = 6.1 Hz), 3.51 (s, 3 H), 3.3 (dd, 1 H, *J* = 6.1 and 8.9 Hz), 3.2 (br. s, 1 H). − ^{13}C NMR: δ = 172.6, 140.9, 131.8, 128.0, 127.6, 126.2, 120.1, 73.8, 58.1, 51.7. − MS; *m*/*z* (relative intensity): 189 (1), 107 (38), 100 (100), 79 (30), 77 (25), 69 (29), 51 (8). − $C_{12}H_{14}O_3$: calcd. C 68.89, H 6.84; found C 68.98, H 6.62.

Methyl *threo*-2-(1-Hydroxy-1-phenylmethyl)-3-butenoate (**2d**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 5.68 (ddd, 1 H, *J* = 8.5, 10.0 and 17.1 Hz), 5.08 (d, 1 H, *J* = 10.0 Hz), 5.01 (d, 1 H, *J* = 17.1 Hz), 4.9 (d, 1 H, *J* = 8.4 Hz), 3.7 (s, 3 H), 3.4 (dd, 1 H, *J* = 8.4 and 8.5 Hz), 3.0 (br. s, 1 H). − ^{13}C NMR: δ = 173.2, 141.2, 132.2, 128.4, 128.0, 126.7, 119.5, 75.3, 57.9, 52.0. − MS; *m*/*z* (relative intensity): 205 (1), 107 (36), 105 (9), 100 (100), 79 (30), 77 (25), 69 (30), 51 (8). − $C_{12}H_{14}O_3$: calcd. C 68.89, H 6.84; found C 68.57, H 6.58.

Methyl *erythro*-2-(1-Hydroxy-1-phenylmethyl)-3-butenoate (**1e**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 5.65−5.55 (m, 2 H), 4.95 (d, 1 H, *J* = 6.3 Hz), 3.55 (s, 3 H), 3.35−3.20 (m, 1 H), 2.87 (br. s, 1 H), 1.7 (dd, 3 H, *J* = 1.2 and 3.4 Hz). − ^{13}C NMR: δ = 173.2, 141.0, 131.9, 128.1, 127.8, 126.4, 124.4, 74.1, 57.5, 51.8, 18.0. − MS; *m*/*z* (relative intensity): 143 (1), 114 (100), 107 (17), 105 (10), 82 (37), 77 (23), 59 (10), 55 (14). − $C_{13}H_{16}O_3$: calcd. C 70.89, H 7.32; found C 70.80, H 7.15.

Methyl *threo*-2-(1-Hydroxy-1-phenylmethyl)-3-butenoate (**2e**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 5.45−5.3 (m, 2 H), 4.85 (d, 1

H, J = 8.4 Hz), 3.65 (s, 3 H), 3.33 (m, 2 H), 1.5 (d, 3 H, J = 4.8 Hz). − ^{13}C NMR: δ = 173.7, 141.4, 130.3, 128.0, 127.6, 126.5, 124.7, 75.3, 56.9, 51.7, 17.7. − MS; *m*/*z* (relative intensity): 143 (1), 114 (100), 107 (17), 105 (10), 82 (35), 77 (22), 55 (13). − $C_{13}H_{16}O_3$: calcd. C 70.89, H 7.32; found C 71.05, H 7.15.

Methyl *erythro*-(*E*)-2-(1-Hydroxyethyl)-4-phenyl-3-butenoate (**1f**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.55 (d, 1 H, J = 16.0 Hz), 6.3 (dd, 1 H, J = 9.3 and 16.0 Hz), 4.15 (dq, 1 H, J = 4.9 and 7.3 Hz), 3.7 (s, 3 H), 3.15 (dd, 1 H, J = 4.9 and 9.3 Hz), 2.8 (br. s, 1 H), 1.2 (d, 3 H, J = 7.3 Hz). − ^{13}C NMR: δ = 173.6, 136.4, 135.4, 128.5, 127.8, 126.4, 122.9, 68.0, 56.4, 52.0, 20.2. − MS; *m*/*z* (relative intensity): 176 (87), 144 (100), 133 (11), 117 (60), 116 (46), 115 (81), 91 (12), 77 (6), 45 (17), 43 (33). − $C_{13}H_{16}O_3$: calcd. C 70.89, H 7.32; found C 70.61, H 7.40.

Methyl *threo*-(*E*)-2-(1-Hydroxyethyl)-4-phenyl-3-butenoate (**2f**): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.55 (d, 1 H, J = 15.9 Hz), 6.15 (dd, 1 H, J = 9.3 and 15.9 Hz), 4.1 (dq, 1 H, J = 6.4 and 7.9 Hz), 3.7 (s, 3 H), 3.2 (dd, 1 H, J = 7.9 and 9.3 Hz), 1.2 (d, 3 H, J = 6.4 Hz). − ^{13}C NMR: δ = 173.5, 136.6, 134.3, 128.5, 127.8, 126.3, 123.9, 68.8, 57.7, 52.0, 20.9. − MS; *m*/*z* (relative intensity): 176 (89), 144 (100), 117 (60), 116 (35), 115 (66), 91 (10), 45 (14), 43 (23). − $C_{13}H_{16}O_3$: calcd. C 70.89, H 7.32; found C 70.98, H 7.15.

Synthesis of the Methyl 4-(Phenylseleno)tetrahydro-3-furancarboxylates. − General Procedure: A mixture of the appropriate 2-methoxycarbonyl-3-alkenol (5 mmol), diphenyl diselenide (2.5 mmol), and ammonium persulfate (2.5 mmol) in acetonitrile (15 mL) was stirred under reflux. The progress of the reaction was monitored by TLC. After 3 h, the mixture was poured into water and extracted with dichloromethane. The combined organic phases were dried, the solvent was evaporated, and the residue was chromatographed on a silica gel column, eluting with a 1:9 mixture of diethyl ether and light petroleum. The products obtained, the yields, and the isomeric ratios are collected in Table 2. Physical and spectral data are reported below.

Methyl (3*SR*,4*SR*,5*RS*)-5-Ethyl-2,2-dimethyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**3a**): Oil. − ^{1}H NMR ([D$_6$]acetone): δ = 7.7−7.6 (m, 2 H), 7.4−7.2 (m, 3 H), 3.73 (ddd, 1 H, J = 3.2, 7.4 and 10.4 Hz), 3.63 (s, 3 H), 3.55 (t, 1 H, J = 10.4 Hz), 2.92 (d, 1 H, J = 10.4 Hz), 1.78 (ddq, 1 H, J = 3.2, 7.4 and 14.3 Hz), 1.45 (dquintet, 1 H, J = 7.4 and 14.3 Hz), 1.25 (s, 3 H), 1.05 (s, 3 H), 0.9 (t, 3 H, J = 7.4 Hz). − ^{13}C NMR: δ = 171.0, 136.0, 128.8, 128.1, 82.9, 80.4, 61.4, 51.5, 45.7, 29.0, 25.6, 25.4, 9.7. − MS; *m*/*z* (relative intensity): 342 (21), 284 (6), 185 (5), 157 (15), 153 (22), 127 (100), 125 (30), 95 (48), 78 (11), 77 (19), 71 (15), 67 (25), 59 (44), 57 (34), 43 (54), 41 (36). − $C_{16}H_{22}O_3Se$: calcd. C 56.31, H 6.50; found C 56.50, H 6.72.

Methyl (3*SR*,4*RS*,5*SR*)-5-Ethyl-2,2-dimethyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**4a**): Oil. − ^{1}H NMR ([D$_6$]acetone): δ = 7.65−7.55 (m, 2 H), 7.4−7.2 (m, 3 H), 4.18 (ddd, 1 H, J = 3.1, 7.4 and 8.1 Hz), 3.68 (s, 3 H), 3.5 (dd, 1 H, J = 7.7 and 8.1 Hz), 3.28 (d, 1 H, J = 7.7 Hz), 1.8 (ddq, 1 H, J = 3.1, 7.4 and 14.9 Hz), 1.41 (dquintet, 1 H, J = 7.4 and 14.9 Hz), 1.3 (s, 3 H), 1.25 (s, 3 H), 0.9 (t, 3 H, J = 7.4 Hz). − ^{13}C NMR: δ = 171.6, 134.1, 130.5, 128.3, 127.6, 84.8, 81.3, 60.3, 51.3, 48.4, 30.9, 26.7, 25.1, 10.0. − MS; *m*/*z* (relative intensity): 342 (12), 185 (8), 157 (17), 153 (55), 127 (100), 95 (74), 77 (19), 67 (30), 59 (32), 57 (73), 55 (18), 43 (47), 41 (27). − $C_{16}H_{22}O_3Se$: calcd. C 56.31, H 6.50; found C 56.15, H 6.75.

Methyl (3*SR*,4*SR*,5*RS*)-5-Ethyl-2,2-diphenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**3b**): Oil. − ^{1}H NMR: δ = 7.7−7.5 (m, 4 H), 7.4−7.1 (m, 11 H), 3.98 (d, 1 H, J = 9.4 Hz), 3.85 (t, 1 H, J = 9.4 Hz), 3.68 (ddd, 1 H, J = 3.0, 7.4 and 9.4 Hz), 3.15 (s, 3 H), 2.0 (ddq, 1 H, J = 3.0, 7.4 and 14.4 Hz), 1.8 (dquintet, 1 H, J = 7.4 and 14.4 Hz), 1.13 (t, 3 H, J = 7.4 Hz). − ^{13}C NMR: δ = 170.8, 145.1, 142.4, 135.5, 128.9, 128.1, 127.6, 127.2, 126.8, 125.7, 125.4, 88.4, 83.1, 63.6, 51.4, 46.5, 24.9, 10.4. − MS; *m*/*z* (relative intensity): 310 (2), 183 (100), 156 (2), 127 (46), 105 (36), 77 (15). − $C_{26}H_{26}O_3Se$: calcd. C 67.09, H 5.63; found C 67.25, H 5.49.

Methyl (3*SR*,4*RS*,5*SR*)-5-Ethyl-2,2-diphenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**4b**): Oil. − ^{1}H NMR: δ = 7.6−7.2 (m, 15 H), 4.6 (dt, 1 H, J = 2.4 and 9.5 Hz), 4.35 (d, 1 H, J = 6.5 Hz), 3.31 (dd, 1 H, J = 6.5 and 9.5 Hz), 3.3 (s, 3 H), 1.82 (ddq, 1 H, J = 2.4, 7.4 and 14.2 Hz), 1.4 (ddq, 1 H, J = 7.4, 9.5 and 14.2 Hz), 1.13 (t, 3 H, J = 7.4 Hz). − ^{13}C NMR: δ = 170.7, 146.3, 134.0, 129.8, 129.2, 128.3, 128.1, 127.7, 127.2, 126.8, 125.6, 89.0, 85.8, 61.0, 51.2, 48.5, 27.4, 11.2. − MS; *m*/*z* (relative intensity): 277 (3), 183 (24), 156 (1), 127 (100), 105 (24), 77 (12). − $C_{26}H_{26}O_3Se$: calcd. C 67.09, H 5.63; found C 67.21, H 5.75.

Methyl (2*SR*,3*SR*,4*SR*,5*RS*)-5-Ethyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**3c**): Oil. − ^{1}H NMR: δ = 7.62−7.52 (m, 2 H), 7.4−7.1 (m, 8 H), 5.02 (d, 1 H, J = 8.5 Hz), 4.05 (ddd, 1 H, J = 3.9, 7.3 and 8.7 Hz), 3.7 (dd, 1 H, J = 8.7 and 10.0 Hz), 3.58 (s, 3 H), 3.1 (dd, 1 H, J = 8.5 and 10.0 Hz), 1.85 (ddq, 1 H, J = 3.9, 7.3 and 15.0 Hz), 1.68 (dquintet, 1 H, J = 7.3 and 15.0 Hz), 1.05 (t, 3 H, J = 7.3 Hz). − ^{13}C NMR: δ = 171.9, 140.7, 136.0, 129.0, 128.4, 127.8, 126.8, 125.7, 85.9, 82.4, 60.7, 51.9, 47.7, 26.4, 10.1. − MS; *m*/*z* (relative intensity): 390 (3), 173 (8), 157 (6), 127 (100), 115 (25), 105 (20), 95 (17), 77 (12), 59 (18), 41 (5). − $C_{20}H_{22}O_3Se$: calcd. C 61.53, H 5.68; found C 61.68, H 5.60.

Methyl (2*SR*,3*SR*,4*RS*,5*SR*)-5-Ethyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**4c**): Oil. − ^{1}H NMR: δ = 7.62−7.52 (m, 2 H), 7.4−7.2 (m, 8 H), 5.24 (d, 1 H, J = 7.3 Hz), 4.12 (ddd, 1 H, J = 4.0, 7.4 and 7.6 Hz), 3.7 (s, 3 H), 3.53 (dd, 1 H, J = 7.6 and 9.3 Hz), 3.35 (dd, 1 H, J = 7.3 and 9.3 Hz), 1.9 (ddq, 1 H, J = 4.0, 7.4 and 14.8 Hz), 1.68 (dquintet, 1 H, J = 7.4 and 14.8 Hz), 1.05 (t, 3 H, J = 7.4 Hz). − ^{13}C NMR: δ = 171.7, 140.8, 134.1, 129.7, 129.2, 128.5, 127.9, 126.0, 86.2, 82.1, 58.1, 51.7, 48.3, 26.7, 10.2. − MS; *m*/*z* (relative intensity): 390 (5), 173 (20), 157 (7), 127 (100), 115 (40), 105 (52), 77 (15), 59 (16). − $C_{20}H_{22}O_3Se$: calcd. C 61.53, H 5.68; found C 61.40, H 5.50.

Methyl (2*SR*,3*SR*,4*SR*)-2-Phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**3d**): Oil. − ^{1}H NMR ([D$_6$]acetone): δ = 7.65−7.55 (m, 2 H), 7.4−7.2 (m, 8 H), 4.95 (d, 1 H, J = 8.3 Hz), 4.37 (dd, 1 H, J = 7.1 and 8.7 Hz), 4.2 (ddd, 1 H, J = 6.4, 7.1 and 8.0 Hz), 4.08 (dd, 1 H, J = 6.4 and 8.7 Hz), 3.6 (s, 3 H), 2.95 (dd, 1 H, J = 8.0 and 8.3 Hz). − ^{13}C NMR: δ = 171.7, 139.7, 135.0, 129.0, 128.2, 128.0, 127.8, 125.7, 84.2, 74.1, 59.4, 51.9, 42.5. − MS; *m*/*z* (relative intensity): 362 (6), 205 (23), 157 (8), 105 (23), 99 (100), 77 (16), 41 (6). − $C_{18}H_{18}O_3Se$: calcd. C 59.84, H 5.02; found C 59.54, H 5.19.

Methyl (2*SR*,3*SR*,4*RS*)-2-Phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**4d**): Oil. − ^{1}H NMR: δ = 7.6−7.5 (m, 2 H), 7.4−7.2 (m, 8 H), 5.3 (d, 1 H, J = 6.9 Hz), 4.5 (dd, 1 H, J = 6.7 and 9.1 Hz), 4.1 (t, 1 H, J = 6.9 Hz), 3.9 (dt, 1 H, J = 6.7 and 6.9 Hz), 3.7 (s, 3 H), 3.3 (dd, 1 H, J = 6.9 and 9.1 Hz). − ^{13}C NMR: δ = 171.7, 133.8, 128.9, 128.2, 127.5, 125.4, 82.5, 75.0, 57.1, 51.5, 42.7. − MS; *m*/*z* (relative intensity): 156 (6), 145 (28), 105 (17), 99 (100), 77 (12), 41 (5). − $C_{18}H_{18}O_3Se$: calcd. C 59.84, H 5.02; found C 59.50, H 4.92.

Methyl (2*SR*,3*SR*,4*SR*,5*RS*)-5-Methyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (**3e**): M.p. 47−49°C. − ^{1}H NMR: δ = 7.6−7.5 (m, 2 H), 7.4−7.2 (m, 8 H), 5.09 (d, 1 H, J = 8.3 Hz), 4.18 (dq, 1 H, J = 6.1 and 9.2 Hz), 3.6 (s, 3 H), 3.59 (dd, 1 H, J =

9.2 and 10.3 Hz), 3.1 (dd, 1 H, *J* = 8.3 and 10.3 Hz), 1.42 (d, 3 H, *J* = 6.1 Hz). − ^{13}C NMR: δ = 172.0, 141.0, 136.2, 129.2, 128.5, 127.9, 126.6, 125.7, 82.4, 81.1, 60.6, 52.1, 50.1, 19.1. − MS; *m/z* (relative intensity): 376 (6), 219 (8), 157 (5), 113 (100), 105 (17), 77 (14), 43 (15). − $C_{19}H_{20}O_3Se$: calcd. C 60.80, H 5.37; found C 60.95, H 5.55.

Methyl (2*SR*,3*SR*,4*RS*,5*SR*)-5-Methyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (4e): Oil. − ^{1}H NMR: δ = 7.65−7.5 (m, 2 H), 7.4−7.2 (m, 8 H), 5.2 (d, 1 H, *J* = 6.1 Hz), 4.25 (dq, 1 H, *J* = 6.1 and 8.3 Hz), 3.7 (s, 3 H), 3.45−3.35 (m, 2 H), 1.45 (d, 3 H, *J* = 6.1 Hz). − ^{13}C NMR: δ = 171.8, 140.9, 134.0, 129.8, 129.2, 128.5, 127.9, 127.8, 125.9, 82.4, 81.3, 58.3, 51.8, 50.6, 18.9. − MS; *m/z* (relative intensity): 376 (4), 219 (10), 157 (5), 115 (24), 113 (100), 105 (35), 77 (15). − $C_{19}H_{20}O_3Se$: calcd. C 60.80, H 5.37; found C 60.68, H 5.15.

Methyl (2*RS*,3*SR*,4*SR*,5*RS*)-2-Methyl-5-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (3f): Oil. − ^{1}H NMR: δ = 7.5−7.1 (m, 10 H), 4.87 (d, 1 H, *J* = 9.3 Hz), 4.38 (dq, 1 H, *J* = 6.1 and 8.4 Hz), 3.83 (dd, 1 H, *J* = 9.3 and 10.4 Hz), 3.6 (s, 3 H), 2.89 (dd, 1 H, *J* = 8.4 and 10.4 Hz), 1.31 (d, 3 H, *J* = 6.1 Hz). − ^{13}C NMR: δ = 171.5, 140.0, 136.1, 129.0, 128.3, 128.0, 126.5, 85.0, 78.2, 59.7, 52.0, 50.7, 20.5. − MS; *m/z* (relative intensity): 376 (5), 219 (29), 159 (100), 115 (47), 105 (43), 77 (21), 59 (20), 43 (20). − $C_{19}H_{20}O_3Se$: calcd. C 60.63, H 5.36; found C 60.85, H 5.23.

Methyl (2*RS*,3*SR*,4*RS*,5*SR*)-2-Methyl-5-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (4f): Oil. − ^{1}H NMR: δ = 7.5−7.0 (m, 10 H), 5.03 (d, 1 H, *J* = 7.6 Hz), 4.4 (dq, 1 H, *J* = 6.1 and 7.1 Hz), 3.72 (dd, 1 H, *J* = 7.6 and 9.0 Hz), 3.7 (s, 3 H), 3.12 (dd, 1 H, *J* = 7.1 and 9.0 Hz), 1.45 (d, 3 H, *J* = 6.1 Hz). − MS; *m/z* (relative intensity): 376 (3), 219 (26), 159 (100), 157 (9), 115 (33), 113 (49), 105 (45), 77 (20), 43 (19). − $C_{19}H_{20}O_3Se$: calcd. C 60.63, H 5.36; found C 60.90, H 5.45.

Methyl (2*RS*,3*SR*,4*SR*,5*RS*)-5-Ethyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (5c): Oil. − ^{1}H NMR ([D$_6$]benzene): δ = 7.77−7.65 (m, 2 H), 7.4−7.1 (m, 8 H), 5.05 (d, 1 H, *J* = 8.7 Hz), 4.06 (dd, 1 H, *J* = 7.3 and 9.2 Hz), 3.9 (ddd, 1 H, *J* = 3.5, 7.5 and 9.2 Hz), 3.59 (dd, 1 H, *J* = 7.3 and 8.7 Hz), 3.0 (s, 3 H), 2.1 (ddq, 1 H, *J* = 3.5, 7.4 and 15.0 Hz), 1.7 (ddq, 1 H, *J* = 7.4, 7.5 and 15.0 Hz), 1.22 (t, 3 H, *J* = 7.4 Hz). − ^{13}C NMR: δ = 171.1, 138.0, 135.4, 129.0, 128.1, 127.7, 127.2, 126.4, 85.6, 81.0, 58.5, 51.1, 45.2, 25.5, 10.2. − MS; *m/z* (relative intensity): 390 (2), 157 (5), 127 (100), 115 (17), 105 (24), 95 (20), 77 (13), 59 (22), 41 (6). − $C_{20}H_{22}O_3Se$: calcd. C 61.53, H 5.68; found C 61.75, H 5.46.

Methyl (2*RS*,3*SR*,4*RS*,5*SR*)-5-Ethyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (6c): Oil. − ^{1}H NMR: δ = 7.65−7.55 (m, 2 H), 7.35−7.1 (m, 8 H), 5.22 (d, 1 H, *J* = 5.7 Hz), 4.55 (ddd, 1 H, *J* = 3.3, 7.6 and 10.0 Hz), 3.65 (dd, 1 H, *J* = 5.7 and 6.7 Hz), 3.45 (dd, 1 H, *J* = 6.7 and 10.0 Hz), 3.3 (s, 3 H), 2.0 (ddq, 1 H, *J* = 3.3, 7.3 and 14.8 Hz), 1.6 (ddq, 1 H, *J* = 7.3, 7.6 and 14.8 Hz), 1.1 (t, 3 H, *J* = 7.3 Hz). − ^{13}C NMR: δ = 171.3, 138.7, 135.6, 134.5, 129.2, 127.9, 127.6, 126.5, 125.8, 85.3, 81.4, 58.1, 51.1, 49.0, 26.6, 10.2. − MS; *m/z* (relative intensity): 390 (2), 157 (5), 127 (100), 115 (18), 95 (23), 77 (12), 59 (15), 41 (5). − $C_{20}H_{22}O_3Se$: calcd. C 61.53, H 5.68; found C 61.75, H 5.77.

Methyl (2*RS*,3*SR*,4*SR*)-2-Phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (5d): Oil. − ^{1}H NMR: δ = 7.6−7.5 (m, 2 H), 7.4−7.2 (m, 8 H), 5.15 (d, 1 H, *J* = 8.3 Hz), 4.58 (dd, 1 H, *J* = 7.3 and 8.9 Hz), 4.12 (ddd, 1 H, *J* = 6.6, 7.3 and 8.9 Hz), 3.8 (t, 1 H, *J* = 8.9 Hz), 3.35 (dd, 1 H, *J* = 6.6 and 8.3 Hz), 3.1 (s, 3 H). − ^{13}C NMR: δ = 170.6, 137.8, 134.8, 129.1, 128.0, 127.7, 126.1, 82.2, 74.0, 57.1, 51.1, 40.2. − MS; *m/z* (relative intensity): 362 (3), 205 (35), 157 (9), 145 (32), 105 (31), 99 (100), 77 (17), 59 (8), 41 (7). − $C_{18}H_{18}O_3Se$: calcd. C 59.84, H 5.02; found C 60.10, H 5.19.

Methyl (2*RS*,3*SR*,4*RS*)-2-Phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (6d): Oil. − ^{1}H NMR ([D$_6$]acetone): δ = 7.7−7.6 (m, 2 H), 7.4−7.2 (m, 8 H), 5.25 (d, 1 H, *J* = 5.6 Hz), 4.35 (dd, 1 H, *J* = 7.8 and 8.9 Hz), 4.25 (dd, 1 H, *J* = 7.8 and 10.1 Hz), 4.0 (ddd, 1 H, *J* = 6.3, 8.9 and 10.1 Hz), 3.7 (dd, 1 H, *J* = 5.6 and 6.3 Hz), 3.2 (s, 3 H). − ^{13}C NMR: δ = 170.0, 138.1, 134.2, 129.2, 128.0, 127.8, 127.7, 125.7, 83.0, 73.6, 56.5, 51.1, 43.2. − MS; *m/z* (relative intensity): 157 (5), 105 (5), 99 (100), 77 (7), 41 (4). − $C_{18}H_{18}O_3Se$: calcd. C 59.84, H 5.02; found C 59.55, H 4.90.

Methyl (2*RS*,3*SR*,4*SR*,5*RS*)-5-Methyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (5e): Oil. − ^{1}H NMR: δ = 7.7−7.6 (m, 2 H), 7.4−7.2 (m, 8 H), 5.1 (d, 1 H, *J* = 8.9 Hz), 4.03 (dq, 1 H, *J* = 6.0 and 9.3 Hz), 3.7 (dd, 1 H, *J* = 8.2 and 9.3 Hz), 3.5 (dd, 1 H, *J* = 8.2 and 8.9 Hz), 3.15 (s, 3 H) 1.55 (d, 3 H, *J* = 6.0 Hz). − ^{13}C NMR: δ = 171.0, 138.0, 135.8, 129.1, 128.3, 127.8, 127.0, 126.5, 81.2, 81.0, 58.4, 51.3, 47.4, 18.0. − MS; *m/z* (relative intensity): 376 (4), 219 (13), 157 (5), 113 (100), 105 (18), 81 (16), 77 (12), 43 (12). − $C_{19}H_{20}O_3Se$: calcd. C 60.80, H 5.37; found C 60.67, H 5.48.

Methyl (2*RS*,3*SR*,4*RS*,5*SR*)-5-Methyl-2-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (6e): Oil. − ^{1}H NMR: δ = 7.65−7.55 (m, 2 H), 7.35−7.2 (m, 8 H), 5.28 (d, 1 H, *J* = 6.1 Hz), 4.72 (dq, 1 H, *J* = 6.0 and 10.0 Hz), 3.65 (dd, 1 H, *J* = 6.1 and 6.7 Hz), 3.35 (dd, 1 H, *J* = 6.7 and 10.0 Hz), 3.28 (s, 3 H), 1.47 (d, 3 H, *J* = 6.0 Hz). − ^{13}C NMR: δ = 170.2, 138.6, 134.5, 130.1, 129.3, 128.0, 127.7, 125.7, 81.2, 80.7, 58.2, 51.6, 51.1, 19.5. − MS; *m/z* (relative intensity): 376 (3), 157 (4), 115 (12), 113 (100), 105 (6), 81 (16), 77 (10), 43 (9). − $C_{19}H_{20}O_3Se$: calcd. C 60.80, H 5.37; found C 60.65, H 5.45.

Methyl (2*SR*,3*SR*,4*SR*,5*RS*)-2-Methyl-5-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (5f): Oil. − ^{1}H NMR: δ = 7.6−7.2 (m, 10 H), 4.66 (d, 1 H, *J* = 9.3 Hz), 4.28 (dq, 1 H, *J* = 6.4 and 8.3 Hz), 3.89 (dd, 1 H, *J* = 7.5 and 9.3 Hz), 3.67 (s, 3 H), 3.3 (dd, 1 H, *J* = 7.5 and 8.3 Hz), 1.3 (d, 3 H, *J* = 6.4 Hz). − ^{13}C NMR: δ = 171.9, 138.7, 135.5, 129.0, 128.3, 128.1, 127.1, 86.3, 75.7, 56.6, 51.7, 49.3, 17.2. − MS; *m/z* (relative intensity): 376 (5), 219 (46), 159 (50), 157 (10), 115 (46), 113 (100), 105 (37), 77 (21), 43 (16). − $C_{19}H_{20}O_3Se$: calcd. C 60.63, H 5.36; found C 60.48, H 5.52.

Methyl (2*SR*,3*SR*,4*RS*,5*SR*)-2-Methyl-5-phenyl-4-(phenylseleno)tetrahydro-3-furancarboxylate (6f): Oil. − ^{1}H NMR: δ = 7.5−7.1 (m, 10 H), 5.32 (d, 1 H, *J* = 9.0 Hz), 4.6 (dq, 1 H, *J* = 6.4 and 9.8 Hz), 3.85 (s, 3 H), 3.5 (m, 2 H), 1.35 (d, 3 H, *J* = 6.4 Hz). − MS; *m/z* (relative intensity): 376 (6), 157 (8), 115 (100), 105 (9), 77 (10), 43 (9). − $C_{19}H_{20}O_3Se$: calcd. C 60.63, H 5.36; found C 60.85, H 5.20.

One-Pot Conversion of 2-Methoxycarbonyl-3-alkenols into 3-Methoxycarbonyl-2,5-dihydrofurans. − General Procedure: A mixture of the appropriate 2-methoxycarbonyl-3-alkenol (2 mmol), diphenyl diselenide (0.2 mmol), and ammonium persulfate (6 mmol) in acetonitrile (10 mL) was stirred under reflux. The progress of the reaction was monitored by TLC. After 2 h, the mixture was poured into water and extracted with dichloromethane. The combined organic phases were dried, the solvent was evaporated, and the residue was chromatographed on a silica gel column, eluting with a 15:85 mixture of diethyl ether and light petroleum. The products obtained and the yields are collected in Table 3. Physical and spectral data are reported below.

Methyl 5-Ethyl-2,2-dimethyl-2,5-dihydro-3-furancarboxylate (7a): Oil. − ^{1}H NMR: δ = 6.7 (d, 1 H, *J* = 1.6 Hz), 4.8 (dt, 1 H, *J* =

1.6 and 6.0 Hz), 3.71 (s, 3 H), 1.65 (dq, 2 H, *J* = 6.0 and 7.4 Hz), 1.47 (s, 3 H), 1.42 (s, 3 H), 0.95 (t, 3 H, *J* = 7.4 Hz). − ^{13}C NMR: δ = 163.0, 140.7, 139.1, 86.8, 83.5, 51.2, 28.5, 28.1, 26.9, 9.3. − MS; *m*/*z* (relative intensity): 169 (68), 155 (79), 137 (15), 95 (26), 67 (29), 59 (48), 57 (54), 43 (100), 41 (30). − $C_{10}H_{16}O_3$: calcd. C 65.18, H 8.76; found C 65.03, H 8.30.

Methyl 5-Ethyl-2,2-diphenyl-2,5-dihydro-3-furancarboxylate (7b): Oil. − ^{1}H NMR: δ = 7.45−7.2 (m, 10 H), 7.05 (d, 1 H, *J* = 1.5 Hz), 4.95 (dt, 1 H, *J* = 1.5 and 6.4 Hz), 3.6 (s, 3 H), 1.9−1.7 (m, 2 H), 1.0 (t, 3 H, *J* = 7.5 Hz). − ^{13}C NMR: δ = 162.9, 143.7, 143.5, 143.2, 137.8, 128.0, 127.9, 127.5, 127.3, 127.2, 125.5, 84.4, 51.3, 27.7, 9.7. − MS; *m*/*z* (relative intensity): 308 (12), 279 (21), 231 (100), 219 (25), 199 (48), 115 (10), 105 (39), 77 (17). − $C_{20}H_{20}O_3$: calcd. C 77.89, H 6.54; found C 77.80, H 6.60.

Methyl (2*RS*,5*RS*)-5-Ethyl-2-phenyl-2,5-dihydro-3-furancarboxylate (7c): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.9 (t, 1 H, *J* = 1.6 Hz), 5.95 (dd, 1 H, *J* = 1.6 and 5.9 Hz), 5.17 (ddt, 1 H, *J* = 1.6, 5.9 and 6.0 Hz), 3.68 (s, 3 H), 1.7 (dq, 2 H, *J* = 6.0 and 7.3 Hz), 1.0 (t, 3 H, *J* = 7.3 Hz). − ^{13}C NMR: δ = 162.8, 141.4, 140.8, 135.6, 128.3, 128.0, 127.0, 87.1, 86.1, 51.5, 28.4, 9.3. − MS; *m*/*z* (relative intensity): 232 (3), 203 (100), 171 (12), 144 (42), 115 (49), 105 (32), 91 (20), 59 (25), 57 (35), 43 (15). − $C_{14}H_{16}O_3$: calcd. C 72.38, H 6.95; found C 72.21, H 6.65.

Methyl 2-Phenyl-2,5-dihydro-3-furancarboxylate (7d): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 7.0 (q, 1 H, *J* = 1.9 Hz), 5.95 (ddd, 1 H, *J* = 1.9, 3.8 and 6.0 Hz), 5.0 (ddd, 1 H, *J* = 1.9, 6.0 and 15.8 Hz), 4.85 (ddd, 1 H, *J* = 1.9, 3.8 and 15.8 Hz), 3.6 (s, 3 H). − ^{13}C NMR: δ = 162.4, 140.7, 138.3, 135.5, 129.0, 128.8, 128.0, 86.7, 75.0, 51.4. − MS; *m*/*z* (relative intensity): 204 (14), 203 (38), 172 (35), 171 (18), 145 (72), 144 (67), 127 (17), 115 (66), 105 (100), 91 (20), 77 (34), 59 (29), 51 (20). − $C_{12}H_{12}O_3$: calcd. C 70.56, H 5.93; found C 70.28, H 5.83.

Methyl (2*RS*,5*RS*)-5-Methyl-2-phenyl-2,5-dihydro-3-furancarboxylate (7e): Oil. − ^{1}H NMR: δ = 7.4−7.25 (m, 5 H), 6.87 (dd, 1 H, *J* = 1.4 and 1.8 Hz), 5.86 (dd, 1 H, *J* = 1.8 and 5.8 Hz), 5.3 (ddq, 1 H, *J* = 1.4, 5.8 and 6.6 Hz), 3.61 (s, 3 H), 1.39 (d, 3 H, *J* = 6.6 Hz). − ^{13}C NMR: δ = 162.8, 142.7, 140.8, 135.2, 128.3, 128.0, 127.0, 86.4, 81.8, 51.5, 21.0. − MS; *m*/*z* (relative intensity): 218 (15), 186 (44), 176 (17), 158 (39), 144 (34), 141 (10), 115 (61), 105 (100), 91 (22), 77 (35), 43 (90). − $C_{13}H_{14}O_3$: calcd. C 71.53, H 6.47; found C 71.22, H 6.60.

Methyl (2*RS*,5*SR*)-2-Methyl-5-phenyl-2,5-dihydro-3-furancarboxylate (7f): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.77 (t, 1 H, *J* = 1.7 Hz), 5.94 (dd, 1 H, *J* = 1.7 and 5.8 Hz), 5.34 (ddq, 1 H, *J* = 1.7, 5.8 and 6.3 Hz), 3.78 (s, 3 H), 1.5 (d, 3 H, *J* = 6.3 Hz). − ^{13}C NMR: δ = 163.0, 140.6, 140.2, 136.4, 128.6, 128.1, 126.4, 86.3, 81.5, 51.6, 21.0. − MS; *m*/*z* (relative intensity): 218 (10), 203 (3), 187 (2), 171 (3), 115 (18), 105 (100), 77 (15), 43 (11). − $C_{13}H_{14}O_3$: calcd. C 71.53, H 6.47; found C 71.45, H 6.68.

Methyl (2*SR*,5*RS*)-5-Ethyl-2-phenyl-2,5-dihydro-3-furancarboxylate (8c): Oil. − ^{1}H NMR: δ = 7.4−7.3 (m, 5 H), 6.9 (dd, 1 H, *J* = 1.8 and 2.0 Hz), 5.85 (dd, 1 H, *J* = 2.0 and 4.5 Hz), 4.9 (ddt, 1 H, *J* = 1.8, 2.0 and 4.5 Hz), 3.6 (s, 3 H), 1.8 (m, 2 H), 1.05 (t, 3 H, *J* = 7.5 Hz). − ^{13}C NMR: δ = 162.6, 141.4, 140.5, 135.3, 127.9, 127.4, 86.7, 86.3, 51.1, 28.7, 9.8. − MS; *m*/*z* (relative intensity): 232 (3), 203 (100), 171 (12), 144 (39), 131 (28), 115 (49), 105 (41), 91 (22), 77 (21), 57 (50), 43 (21). − $C_{14}H_{16}O_3$: calcd. C 72.38, H 6.95; found C 72.17, H 6.79.

Methyl (2*SR*,5*RS*)-5-Methyl-2-phenyl-2,5-dihydro-3-furancarboxylate (8e): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.85 (dd, 1 H, *J* = 1.7 and 2.1 Hz), 5.85 (dd, 1 H, *J* = 2.1 and 4.3 Hz), 5.1 (ddq, 1 H, *J* = 1.7, 4.3 and 6.5 Hz), 3.6 (s, 3 H), 1.45 (d, 3 H, *J* = 6.5 Hz). − ^{13}C NMR: δ = 162.8, 142.6, 140.6, 135.0, 128.1, 128.0, 127.5, 86.7, 81.4, 51.4, 21.4. − MS; *m*/*z* (relative intensity): 218 (16), 203 (15), 186 (41), 158 (38), 144 (31), 141 (10), 131 (19), 115 (88), 105 (100), 91 (26), 77 (43), 59 (29), 43 (87). − $C_{13}H_{14}O_3$: calcd. C 71.53, H 6.47; found C 71.21, H 6.55.

Methyl (2*SR*,5*SR*)-2-Methyl-5-phenyl-2,5-dihydro-3-furancarboxylate (8f): Oil. − ^{1}H NMR: δ = 7.4−7.2 (m, 5 H), 6.72 (t, 1 H, *J* = 1.9 Hz), 5.8 (dd, 1 H, *J* = 1.9 and 4.3 Hz), 5.18 (ddq, 1 H, *J* = 1.9, 4.3 and 6.3 Hz), 3.72 (s, 3 H), 1.53 (d, 3 H, *J* = 6.3 Hz). − ^{13}C NMR: δ = 163.1, 140.8, 140.1, 136.3, 128.5, 128.1, 126.5, 86.7, 81.3, 51.6, 21.8. − MS; *m*/*z* (relative intensity): 218 (11), 187 (3), 171 (2), 115 (15), 105 (100), 77 (17), 43 (14). − $C_{13}H_{14}O_3$: calcd. C 71.53, H 6.47; found C 71.21, H 6.55.

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