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ELECTROCHEMICAL CYCLIZATION OF UNSATURATED HYDROXY COMPOUNDS. PHENYLSELENOETHERIFICATION AND PHENYLSELENOLACTONIZATION[1,2,3]

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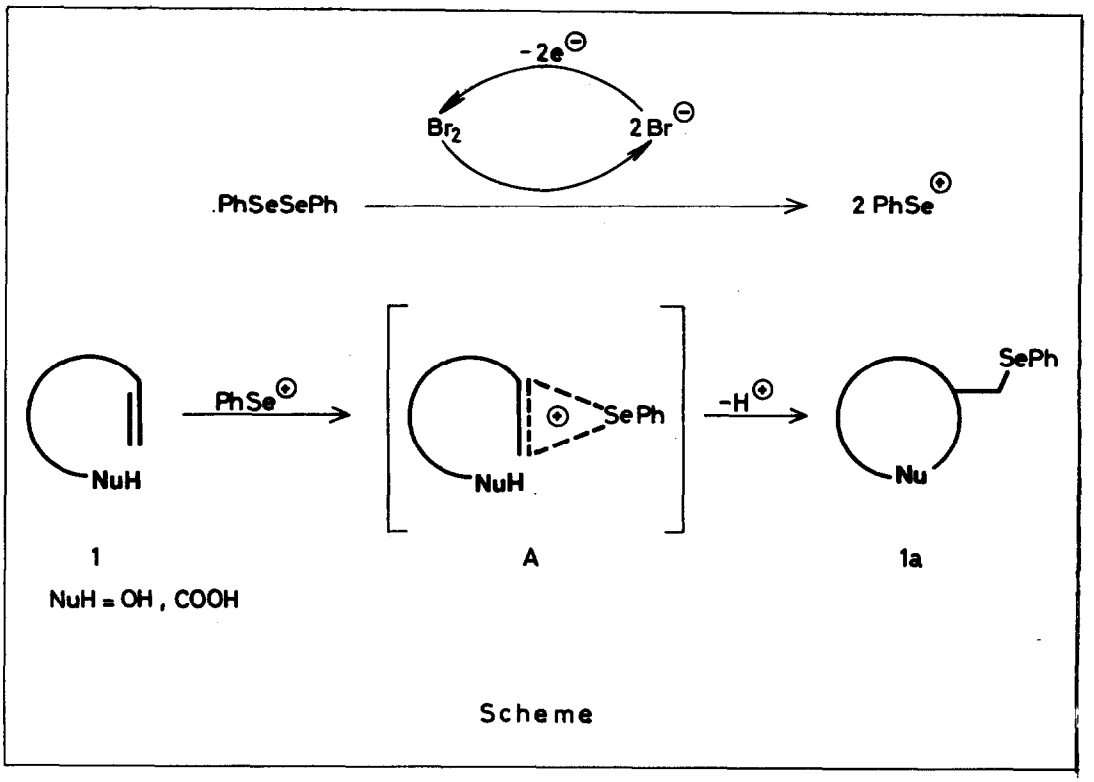
Phenylselenoetherification and phenylselenolactonization were performed in one step by electrolysis of unsaturated alcohols or carboxylic acids and diphenyl diselenide in organic solvent containing halide ions as mediators.

Intramolecular cyclization of unsaturated alcohols to cyclic phenylselenoethers (termed *phenylselenoetherification*) and lactonization of unsaturated carboxylic acids to phenylselenolactones (termed *phenylselenolactonization*) by means of organoselenium reagents became an important tool for the synthesis of different natural products.[5] In continuation of our studies on the first of these transformations[6], we have now investigated phenylselenoetherification and phenylselenolactonization by means of phenylselenenyl ions electrochemically generated from diphenyl diselenide (Scheme). The reactive intermediate A ($PhSe^{\oplus}$ ion) was produced by indirect oxidation on an anode, whereby the role of mediator was taken by a halide anion. Cyclic ether and lactone products were obtained in good to very good yields (50-86%). In this paper detailed results will be given with complete experimental data.

Results and Discussion

Phenylselenoetherification (Table 1). In a typical case, the electrochemical reaction was carried out by electrolyzing a mixture of alkenol, diphenyl diselenide, and tetraethylammonium bromide (method A) or calcium chloride (method B) as an electrolyte, in organic solvent (CH_2Cl_2 or MeOH) solution. The electrolysis was performed in an undivided cell, using a graphite stick as an anode and Cu foil as a cathode. The cell was cooled in an ice-acetone-salt bath (constant current 250 mA; 2 F/mol). After the usual work-up, the crude product was purified by column chromatography (SiO_2, CH_2Cl_2), whereby the cyclic phenylselenoether products were isolated in the form of a pale yellow oil. The results obtained, given in

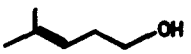
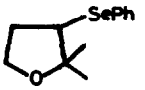
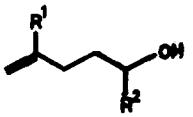
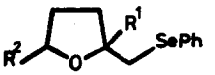
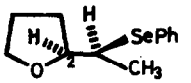
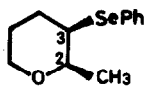
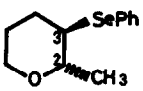
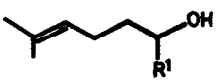
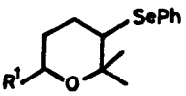
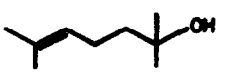
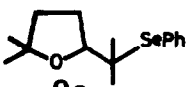
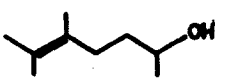
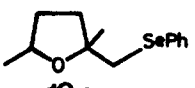
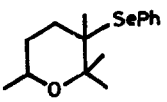
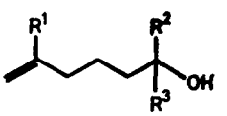
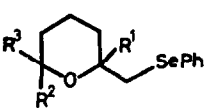


Scheme

Table 1, can be summarized as follows. 3-Buten-1-ol did not cyclize under these conditions, but the simplest terminally dialkylated Δ^3-alkenol **2**, gave the corresponding five-membered cyclic ether **2a**, as the sole reaction product, in very good yield (72%). All the studied Δ^4-alkenols afforded, under these reaction conditions, only cyclic phenylselenoethers of the tetrahydrofuran- and tetrahydropyran-type. The results obtained show also that the substituents at the double bond and at the carbinol carbon atom have a pronounced influence on the regioselectivity of the ring closure. Thus, the terminally unsubstituted Δ^4-alkenols **3**, **4** and **5** afforded (in agreement with Markovnikov rule) exclusively cyclic phenylselenoethers of the tetrahydrofuran-type (**3a**, **4a** and **5a**, respectively). In the case of alkenol **4**, according to GC and NMR spectra, two stereoisomeric cyclic products, *cis*- and *trans*-**4a** were produced (in a ratio of 24:76), which were not separated.

With respect to the regio- and stereoselectivity of cyclic ether formation, the phenylselenoetherification of the two geometric isomers of Δ^4-alkenols with a terminally monosubstituted olefinic bond, such as (Z)- and (E)-4-hexen-1-ol (Z-**6** and E-**6**, respectively), gave interesting results. As can be seen from Table 1, the electrolysis of (Z)-4-hexen-1-ol (Z-**6**) with tetraethylammonium bromide as mediator afforded the *threo*-phenylselenoether *th*-**6a** as the sole product, whereas in the reaction with $CaCl_2$ as mediator, the same alcohol gave a mixture of *threo*- and *erythro*-phenylselenoethers (in a ratio of 50:50). In both cases the reactions were regioselective, giving only five-membered cyclic ethers. In the case of (E)-4-hexen-1-ol (E-**6**) only *cis*- and *trans*-isomers of the corresponding six-membered cyclic phenylselenoethers were produced, their ratio depending on reaction conditions (with Et_4NBr as mediator the *cis/trans* ratio was 70:30, but when $CaCl_2$ was used as mediator, this ratio changed to 33:67).

TABLE 1. ELECTROCHEMICAL CYCLIZATION OF UNSATURATED ALCOHOLS

ALCOHOL	m mol ROH	PRODUCTS		METHOD[a]		ISOLATED YIELD, %
2 	1.85	 2a		A		72
						
3 $R^1=R^2=H$	1.16	3a		A		80
4 $R^1=H$, $R^2=CH_3$	2.71	4a		A	24:76	61[b]
5 $R^1=CH_3$, $R^2=H$	0.48	5a		A		79
Z-6 	1.39	 threo-6a	 erythro-6a	A	100:0	86
	1.00			B	50:50	82
E-6 	1.51	 cis-6b	 trans-6b	A	70:30	77
	1.00			B	33:67	80
						
7 $R^1=H$	0.70	7b		A		61
8 $R^1=CH_3$	1.63	8b		A		67[b]
9 	0.48	 9a		A		50
10 	1.60	 10a	 10b	A	50:50	52[b]
						
11 $R^1=R^2=R^3=H$	1.00	11a		A		75
12 $R^1=R^2=CH_3$, $R^3=H$	1.60	12a		A		75[b]
13 $R^1=R^2=R^3=CH_3$	2.93	13a		A		76

a) METHOD A = Et_4NBr AS ELECTROLYTE ; METHOD B = $CaCl_2$ AS ELECTROLYTE

b) THE STEREOCHEMISTRY OF THESE PRODUCTS WAS NOT, AS YET, INVESTIGATED.

A significant influence of structure was shown in the case of Δ^4-alkenols with a terminally dimethyl substituted double bond. Thus, the primary alcohol **7** and secondary **8** afforded (in agreement with Markovnikov rule) only the six-membered cyclic phenylselenoethers **7b** and **8b**, respectively (in moderate yield). However, an increase in alkyl substitution at the carbinol carbon atom lead to stereochemical control (anti-Markovnikov rule), the tertiary alcohol **9** being cyclized almost exclusively to the corresponding five-membered cyclic phenylselenoether **9a**. In the case of the secondary alcohol **10**, with a completely substituted double bond, steric and electronic factors are approximately similar for the addition onto both trigonal carbon atoms, and the regioisomers **10a** and **10b** were obtained, in a 1:1 ratio. In the electrolysis of Δ^5-alkenols (**11**, **12** and **13**), hydroxyl group participation is regioselective, affording as the only cyclization products the corresponding six-membered cyclic phenylselenoethers (**11a**, **12a** and **13a**, respectively).

Primary and secondary olefinic alcohols with a more remote double bond (Δ^6, Δ^7 and Δ^8), such as 6-hepten-1-ol, 5-methyl-6-hepten-1-ol, 7-octen-2-ol, citronellol, 7-octen-1-ol, and 8-nonen-1-ol, did not undergo intramolecular cyclization when treated under the above mentioned conditions.

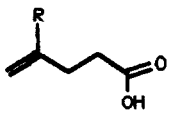
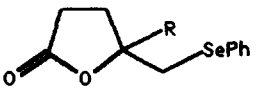
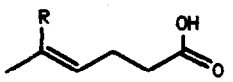
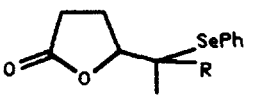
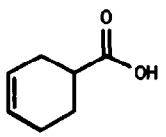
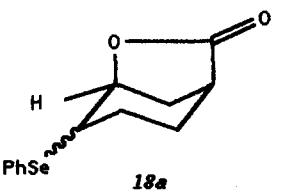
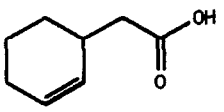
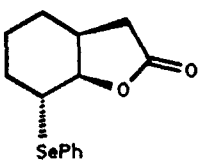
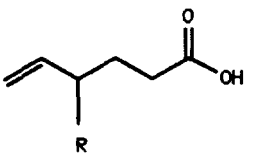
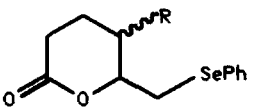
Phenylselenolactonization (Table 2). When unsaturated carboxylic acids, such as 4-pentenoic acid (**14**) and diphenyl diselenide, were electrolyzed in methanol solution of ammonium bromide (as electrolyte), phenylselenolactones were obtained in good to very good yield (56-78%). The reaction procedure requires a mixture of acid, *e.g.* **14** (1 mmol), $(PhSe)_2$ (0.5 mmol), and NH_4Br (3.1 mmol) in MeOH, which was electrolyzed under a constant current (400 mA/cm^2, 6 F/mol). The electrolysis was performed in the undivided cell (described above for phenylselenoetherification), placed in a water bath (15-20°C), using a graphite stick as anode and copper foil as cathode. After completion of the reaction, methanol was distilled off, the residue extracted with diethyl ether, and the crude product (upon solvent removal) purified by column chromatography (SiO_2, CH_2Cl_2). The results given in Table 2 show that (as in the case of phenylsulfenolactonization[3,7]) all the investigated 4-enoic acids gave only γ-lactones, confirming again that the substituents at the double bond had no influence on the regioselectivity of this reaction. 2-Cyclohexene-1-acetic acid (**19**) upon phenylselenolactonization produced the five-membered ring lactone **19a** with *cis*-configuration (observed by ^{1}H NMR spectroscopy), indicating that the carboxylic oxygen added *trans*-stereospecifically in relation to the phenylseleno-group. Terminally unsubstituted 5-enoic acids afforded, as sole products, the respective δ-lactones. Olefinic acid **21** cyclized to only one of the two possible isomers, namely *cis*- or *trans*-lactone, this being shown by the existence of only one signal for the *tert*-butyl group in the ^{1}H NMR spectrum of the lactone product **21a**. 3-Butenoic and 6-heptenoic acid did not undergo intramolecular cyclization under these conditions. The study of the mechanism and stereochemistry (where possible) of this reaction is in progress.

Mild conditions (neutral medium and low temperature), simple equipment and inexpensive reagents are the major advantages of this reaction for its application in the synthesis of heterocyclic compounds, particularly since the cyclic phenylselenoethers and -lactones obtained can be readily deselenenylated and converted to other useful products.[5,8]

Experimental

Column chromatography: Merck silica gel 60, particle size 0.063-0.200 mm. *Gas chromatography*: Perkin-Elmer instrument, Model 3920B (FID) for analytical purposes; the columns consisted of OV 1, OV 101 or SE 30, adsorbed on Chromosorb P (3-5%); carrier gas Ar. *IR spectra*: Perkin-Elmer Spectrophotometer, Model 137B. *NMR spectra*: Varian FT80A (80 MHz), Bruker AM200 (200 MHz), Bruker WH400 (400 MHz) in $CDCl_3$ with TMS as internal standard. All solvents were purified by distillation, methylene chloride was purified by distillation and dried over P_4O_{10}. All the olefinic alcohols and acids used as substrates (see Tables 1 and 2) were known compounds, some of which (**3**, **11**, **14**, **18** and **20**)

Table 2. Electrochemical cyclization of unsaturated carboxylic acid

Unsaturated acid	Products	Isolated yield, %
		
14 R = H	***14a***	71
15 R = CH_3	***15a***	69
		
16 R = H	***16a***	76[a]
17 R = CH_3	***17a***	62
		
18	***18a***	78[a]
		
19	***19a***	73
		
20 R = H	***20a***	68
21 R = $(CH_3)_3C$	***21a***	56[a]

[a] The stereochemistry of these products was not, as yet, investigated.

were commercially available, while the other ones were synthesized according to the procedures described in our previous papers. Most of the cyclic ether and lactone products were known compounds. Spectral data for **2a**,[6] **3a**,[6,9,11] **4a**,[6,11] **5a**,[6] **7b**,[6] **8b**,[6] **9a**,[6] **10a**,[6] **10b**,[6] **11a**,[6,9,11] **12a**,[6] **13a**,[6] **14a**,[10,11] **18a**,[10] **19a**[10] and **20a**[10] were given previously. All new compounds (see Table 1 and 2) were characterized and identified on the basis of spectral data.

Electrolysis was performed in an undivided cell consisting of a glass tube 2.5 cm in diameter and 10 cm in height. A graphite stick (0.6 cm in diameter and 4 cm in length) was used as an anode and copper foil (cylinder shaped 1.5 cm in diameter and 4 cm in length) or copper wire shaped in spiral form (1 cm in diameter) was used as a cathode.

Phenylselenoetherification. A typical electrolysis procedure is as follows.

Method A: A solution of 5-hexen-1-ol (**11**, 100 mg; 1 mmol), diphenyl diselenide (156 mg; 0.5 mmol) and Et_4NBr (210 mg; 1 mmol) in methylene chloride (5 ml) was electrolyzed in a cell placed in an ice-acetone-salt bath (-10 to -5 °C). A constant current (250 mA) was passed through the solution using a regulated direct current supply (Iskra, Kranj, Yugoslavia) until 2F of electricity were consumed. When the reaction was completed, the solvent was distilled off and the residue extracted several times with n-hexane. After removal of n-hexane, the crude product was purified by column chromatography (SiO_2, CH_2Cl_2), whereby 2-[(phenylseleno)methyl]tetrahydropyran (**11a**, 199 mg; 75%) was isolated in the form of a pale yellow oil. Its spectral data were consistent with those of an authentic sample prepared previously by cyclization of 5-hexen-1-ol with $PhSeCl$[6].

threo-2-[**1-(Phenylseleno)ethyl]tetrahydrofuran** (*threo*-**6a**) A mixture of (Z)-4-hexen-1-ol (Z-**6**) (100 mg, 1 mmol), $(PhSe)_2$ (156 mg, 0.5 mmol) and Et_4NBr (200 mg) in CH_2Cl_2 (7 ml) was electrolyzed in a similar manner as described above (200 mA for 16 min, 2 F/mol). The usual work-up and chromatography (SiO_2, CH_2Cl_2) provided 219 mg (86%) of *threo*-**6a** as pale yellow oil. IR (film): ν_{max} = 3060, 2950, 2860, 1590, 1480, 1440, 1370, 1170, 1060, 1000, 930, 740, 690 and 670 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$): δ = 1,33 (d, J = 7 Hz, 3H, *CH*$_3$), 2.00-2.20 (m, 4H, 3-*H* and 4-*H*), 3.37 (m, J = 7 Hz, 1H, 2-*H*), 3.51 (dt, J = 3, 12 Hz, 1H, 5-*H cis* with 2-*H*), 3.66 (dq, J = 7, 1.5 Hz, 1H, *CHSe*), 4.00 (fd, J = 12 Hz, 1H, 5-*H trans* with 2-*H*), 7.22-7.30 (m, 3H, *Ph*), 7.52-7.60 (*m*, 2H, *Ph*).

cis- and *trans*-**2-Methyl-3-(phenylseleno)tetrahydropyran** (*cis*- and *trans*-**6b**). A mixture of (E)-4-hexen-1-ol (E-**6**) (250 mg, 2.5 mmol), $(PhSe)_2$ (390 mg, 1.25 mmol) and Et_4NBr (400 mg) in CH_2Cl_2 (10 ml) was electrolyzed in a similar manner as described above (200 mA for 40 min, 2F/mol). The usual work-up and chromatography (SiO_2, CH_2Cl_2) afforded three fractions, namely 65 mg of *cis*-**6b**, 175 mg of a mixture of *cis*-**6b** and *trans*-**6b**, and 21 mg of *trans* **6b** (total yield 261 mg; 77%). *cis*-**6b** (pale yellow oil): IR (film): ν_{max} = 3040, 2960, 2910, 2850, 1580, 1475, 1435, 1380, 1195, 1090, 1055, 1030, 925, 845, 740, and 695 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ = 1.43 (d, J = 6.9 Hz, 3H, *CH*$_3$), 1.65-2.20 (m, 4H, 4-*H* and 5-*H*), 3.32 (quin, J = 6.9 Hz, 1H, 2-*H*), 3.60-4.05 (m, 3H, 3-*H* and 6-*H*), 7.27 (m, 3H, Ph), 7.56 (m, 2H, Ph). *trans*-**6b** (pale yellow oil): IR (film): ν_{max}= 3040, 2960, 2910, 2850, 1580, 1475, 1435, 1380, 1195, 1090, 1055, 1030, 925, 845, 740, and 695 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ = 1.35 (d, J = 6.1 Hz, 3H, *CH*$_3$), 1.65-2.20 (m, 4H, 4-*H* and 5-*H*), 2.95 (sym m, 1H, *CHSe*), 3.30-3.50 (m, 2H, 2-*H* and $H_{ax}CH_2O$), 3.92 (d, J = 11.3 Hz, 1H, $H_{eq}CH_2O$), 7.27 (m, 3H, Ph), 7.56 (m, 2H, Ph).

Method B. A solution of (Z)-4-hexen-1-ol (Z-**6**) (100 mg, 1 mmol), $(PhSe)_2$ (156 mg, 0.5 mmol) and $CaCl_2$ (100 mg, 0.9 mmol) in distilled methanol (10 ml) was electrolyzed in a similar manner as described above (200 mA for 16 min., 2 F/mol). The cell was placed in a water bath (15-20°C). The usual work-up and chromatography (SiO_2, CH_2Cl_2) afforded three fractions, namely 32 mg of *threo*-**6a**, 142 mg of a mixture of *threo*-**6a** and *erythro*-**6a**, and 20 mg of *erythro*-**6a** (total yield 194 mg; 76%).

erythro-**2-[1-(Phenylseleno)ethyl] tetrahydrofuran** (*erythro*-**6a**) (pale yellow oil): IR (film): ν_{max}= 3050, 2950, 2850, 1587, 1475, 1435, 1375, 1300, 1175, 1060, 1000, 920, 740, 690 and 670 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$): δ = 1.41 (d, J = 7 Hz, 3H, *CH*$_3$), 1.68 - 2.07 (m, 4H, 3-*H* and 4-*H*), 3.41 (quin, J = 7 Hz, 1H, *CHSe*), 3.77 (*q*, J = 7 Hz, 1H, 5-*H*, *cis* with 2-*H*), 3.90 and 3.95 (2xq, overlapped, J=7 Hz, 2H, 5-*H trans* with 2-*H*, and 2-*H*), 7,22-7.30 (m, 3H, *Ph*), 7.55-7.62 (m, 2H, *Ph*).

Phenylselenolactonization. A typical procedure is as follows: A solution of 4-pentenoic acid (**14**,100 mg, 1 mmol), diphenyl diselenide (156 mg, 0.5 mmol) and ammonium bromide (150 mg) in methanol (10 ml) was electrolyzed at 15 to 20°C (water bath) at constant current (400 mA; 6 F/mol) for 25 min. After completion, MeOH was distilled off, the residue extracted with diethyl ether, and the crude product (upon solvent removal) purified by column chromatography (SiO_2, CH_2Cl_2) affording 207 mg (68%) of **14a**.[10,11]

5-Methyl-5-[(phenylseleno)methyl]tetrahydrofuran-2-one (15a). By the same method 4-methyl-4-pentenoic acid (**15**) afforded 186 mg (69%) of **15a** (pale yellow oil): IR (film): ν_{max}= 3040, 2950, 1780, 1580, 1480, 1440, 1380, 1280, 1160, 1070, 940, 740, and 695 cm^{-1}; ^1H NMR (80 MHz, $CDCl_3$): δ = 1.50 (s, 3H, *CH*$_3$), 1.90-2.30 (m, 2H, 3-*H*), 2.40-2,75 (m, 2H, *CH*$_2$*CO*), 3.22 (s, 2H, *CH*$_2$*Se*), 7.20-7.35 (m, 3H, *Ph*), 7.40-7.65 (m, 2H, *Ph*).

5-[1-(Phenylseleno)ethyl]tetrahydrofuran-2-one (16a). By the same method (E)-4-hexenoic acid (**16**) afforded 189 mg (76%) of **16a** (pale yellow oil): IR (film): ν_{max}= 3040, 2950, 1785, 1580, 1440, 1340, 1180, 1010, 900, 750, and 700 cm^{-1}; ^1H NMR (80 MHz, $CDCl_3$): δ = 1.46 (d, J = 7 Hz, 3H, *CH_3*), 1.50-2.15 (m, 4H, *CH_2CH_2*), 3.25 (quin, J = 7 Hz, 1H, *CHSe*), 4.40 (sym m, 1H, *CHO*), 7.15-7.35 (m, 3H, *Ph*), 7.45-7.65 (m, 2H, *Ph*).

5-[1-Methyl-1-(phenylseleno)ethyl]tetrahydrofuran-2-one (17a). By the same method 5-methyl-4-hexenoic acid (**17**) afforded 175 mg (62%) of **17a** (pale yellow oil): IR (film): ν_{max}= 3025, 1770, 1580, 1460, 1440, 1335, 1270, 1180, 980, 830, 765, and 700 cm^{-1}; ^1H NMR (80 MHz, $CDCl_3$): δ = 1.34 and 1.42 (2xs, 6H, 2x*CH_3*), 1.90-2.25 (m, 4H, *CH_2CH_2*), 4.40 (t, J=7 Hz, 1H, *CHO*), 7.25-7.45 (m,3H, *Ph*), 7.55-7.70 (m, 2H, *Ph*).

5-*tert*-Butyl-6-[(phenylseleno)methyl]tetrahydropyran-2-one (21a). By the same method 4-*tert*-butyl-5-hexenoic acid (**21**) afforded 166 mg (56%) of **21a** (pale yellow oil): IR (film): ν_{max}= 3040.2950, 2840, 1735, 1580, 1480, 1440, 1385, 1340, 1260, 1180, 1140, 1040, 1020, 940, 740, and 700 cm^{-1}; ^1H NMR (80 MHz, $CDCl_3$): δ = 0.85 (s, 9H, *$(CH_3)_3C$*), 1.55-2.05 (m, 3H, 3-*H* and 4-*H*), 2.35-2.65 (m, 2H, *CH_2CO*), 3.15-3.30 (ABX, $J_{gem(AB)}$=13.2, J_{AX}=7.6, J_{BX}=6.4 Hz, 2H, *CH_2Se*), 4.60 (dt, J_1=4.0, J_2=7.6 Hz, 1H, *CHO*), 7.15-7.35 (m, 3H, *Ph*), 7.40-7.60 (m, 2H, *Ph*).

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