Preparation of 2-Arylpropanoic Acids by Oxidative Aryl Migration in (β-Aryl-β-hydroxy)alkyl Phenyl Selenides

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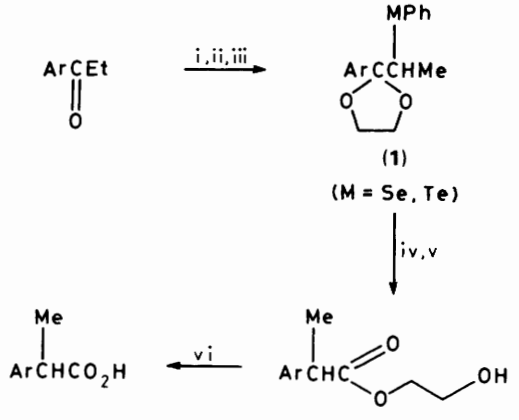
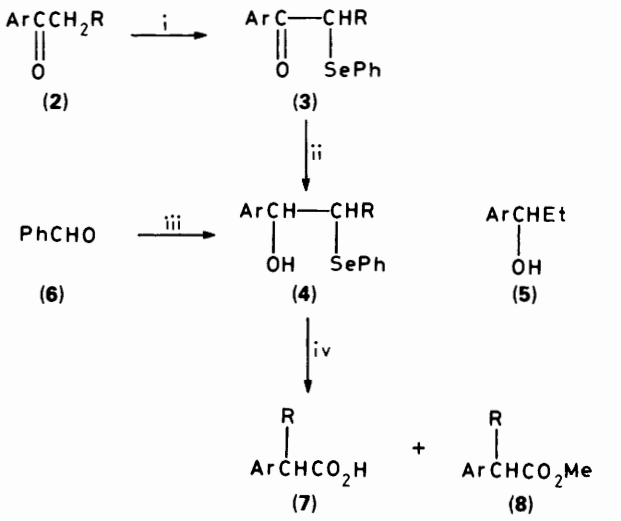
Oxidation of diastereoisomeric mixtures of 1-aryl-1-hydroxyprop-2-yl phenyl selenides, prepared either by phenylselenenylation of propiophenones followed by reduction or by treatment of benzaldehyde with α-(phenylseleno)ethyl anion, with an excess of *meta*-chloroperbenzoic acid in methanol at 25 °C for 24 h or at reflux for 2 h affords methyl 2-arylpropanoates almost quantitatively. Similar treatment in tetrahydrofuran at 25 °C for 24 h results in a direct formation of 2-arylpropanoic acids in high yields.

Recently organo-selenium and -tellurium mediated syntheses of 2-arylpropanoic acids from propiophenones were reported in which the key step was an oxidative aryl migration in the ethylene acetals of α-phenylseleno- and α-phenyltelluro-ethyl ketones (**1**; M = Se, Te) (Scheme 1).[1] In order to obtain the acetal (**1**), the corresponding bromo acetals were substituted by a PhSe or PhTe moiety; several attempts to obtain (**1**) by acetalization of α-(phenylseleno)propiophenone, prepared by the phenylselenenylation of propiophenone, were unsuccessful.[1] Further investigations now reveal that *meta*-chloroperbenzoic acid (MCPBA) oxidation of 1-phenyl-2-(phenylseleno)propan-1-ol (**4a**), prepared by reduction of α-(phenylseleno)propiophenone (**3a**), gives 2-phenylpropanoic acid and/or its methyl ester in similar or better yields and over fewer steps than those shown in Scheme 1. Here we report this new synthetic transformation of propiophenones to pharmaceutically important 2-arylpropanoic acids.[2]



Scheme 1. *Reagents:* i, Br_2; ii, $HOCH_2CH_2OH$; iii, $(PhM)_2$, Na; iv, MCPBA, MeOH; v, H_2O; vi, OH^-.

The (β-aryl-β-hydroxy)alkyl phenyl selenides (**4**) were prepared either by phenylselenenylation at the α-position of acetophenone or propiophenones (**2**)[3] followed by reduction,[4] or by treatment of benzaldehyde (**6**) with (phenylseleno)-methyl or -ethyl anion[5,6] (Scheme 2). Compounds (**4**) were mainly prepared by the former method in 36–66% isolated yields based on the starting ketone, as shown in Table 1. They consisted of two diastereoisomers; the *erythro*:*threo* ratio was highly dependent on the preparative methods, being 10:90 for the formation of (**4a**) by the former method and 45:55 for the latter.

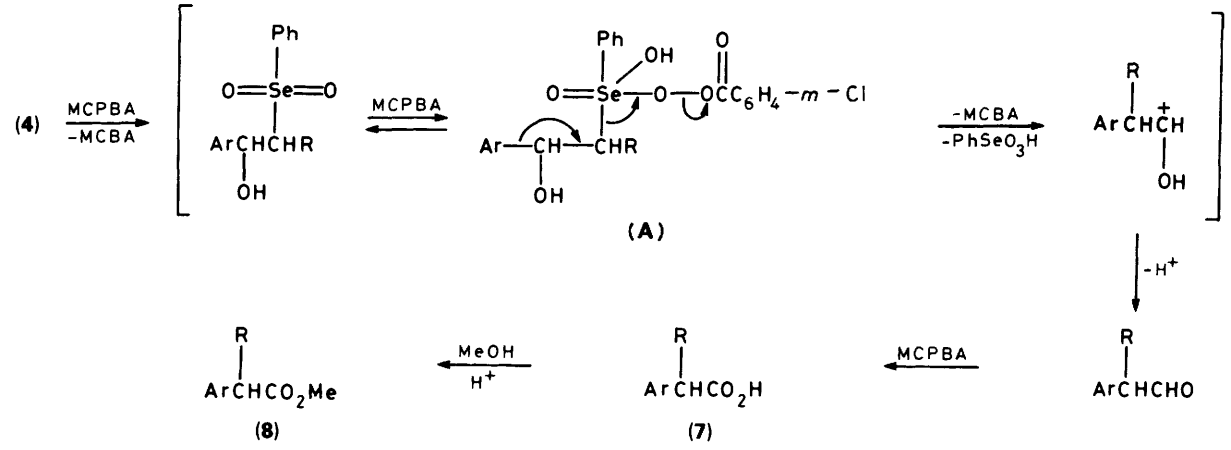


Scheme 2. *Reagents:* i, PhSeCl, EtOAc; ii, reduction; iii, PhSeCH(R)Li, THF; iv, MCPBA (3–5 equiv.), MeOH or THF. In the Scheme; **a**, Ar = Ph, R = Me; **b**, Ar = 4-BrC_6H_4, R = Me; **c**, Ar = 4-MeC_6H_4, R = Me; **d**, Ar = 4-$Bu^tC_6H_4$, R = Me; **e**, Ar = 4-PhC_6H_4, R = Me; **f**, Ar = Ph, R = H.

Table 1. Preparation of β-hydroxyselenides (**4**).

Starting carbonyl compd.	Method[a]	Yield (%) of (**3**)[b]	Yield (%) of (**4**)[c,d]
(**2a**)	A	(**3a**) 82	(**4a**) 72
(**2b**)	A	(**3b**) 69	(**4b**) 53
(**2c**)	A	(**3c**) 78	(**4c**) 59
(**2d**)	A	(**3d**) 71	(**4d**) 55
(**2e**)	A[e]	(**3e**) 87	(**4e**) 75[f]
(**6**)	B	—	(**4a**) 78[g]
(**6**)	B	—	(**4f**) 82

[a] Method A: (**2**) ⟶ (**3**), PhSeCl (1.2 equiv.), EtOAc, 25 °C, 24 h; (**3**) ⟶ (**4**), $LiAlH_4$ (1.6 equiv.), Et_2O, −10 °C, 2 h. Method B: PhSeCH(R)Li (R = H, Me) (1 equiv.), THF, −78 °C, 2 h. [b] Isolated yield based on (**2**). [c] Isolated yield based on (**3**) or (**6**). [d] *erythro*:*threo* = *ca.* 10:90 in (**4a–e**) (see Experimental section). [e] THF as solvent at the reduction step. [f] Other product: (**5e**) 11%. [g] *erythro*:*threo* = 45:55 (by GLC).



Scheme 3.

Table 2. Reduction of α-(phenylseleno)propiophenones (**3e**).[a]

Reducing agent	Solvent	Reaction temp. (°C)	Reaction time (h)	Product and yield (%)[b] (4e)	(*erythro*:*threo*)	(5e)
$LiAlH_4$	THF	−10	2	75	(10:90)	11
$NaBH_4$[c]	MeOH–water[d]	25	2	58	(5:95)	38
$Al(OPr^i)_3$	Pr^iOH	reflux	8	93	(28:72)	5
Bu^i_2AlH	THF	−20	24	87	(7:93)	2
$Zn(BH_4)_2$	THF	0	24	91	(9:91)	0

[a] (**3e**) (5–10 mmol), reducing agent [1.6 equiv. to (**3e**)], and solvent (10–50 ml) were used. [b] Isolated yield based on (**3e**); for isomer ratio, see Experimental section. [c] 4.0 equiv. to (**3e**). [d] 9:1 (v/v).

Table 3. MCPBA oxidation of β-hydroxyselenides (**4**).[a]

(4)	Solvent	Reaction temp. (°C)	Reaction time (h)	Product and yield (%)[b] (7)		(8)	
(**4a**)	MeOH	25	5	(**7a**)	40	(**8a**)	10[c]
(**4a**)	MeOH	25	24	(**7a**)	10	(**8a**)	90
(**4a**)	MeOH	reflux	2	(**7a**)	0	(**8a**)	100
(**4d**)	THF	25	24	(**7d**)	67	—	
(**4e**)	THF	25	24	(**7e**)	81	—	
(**4f**)	MeOH	reflux	3	(**7f**)	3	(**8f**)	86
(**4f**)	MeOH	25	5	(**7f**)	7	(**8f**)	0

[a] (**4**) (1–2 mmol), MCPBA [5 equiv. to (**4**)], and solvent (3–10 ml) were used. [b] Determined by GLC. [c] Other product: 2-phenylpropanal, 10%.

The *threo* selectivity of the former reaction has already been shown.[4,7] Detailed studies using (**2e**) as substrate disclosed that the reduction step sometimes afforded the 1-arylpropanol (**5e**) as a side-product by substitution of the PhSe group by hydrogen. The yield of (**4e**) and its diastereoisomeric ratio (*threo* selective) was affected by the nature of the reducing agent employed, $Zn(BH_4)_2$ being revealed as the reagent of choice (Table 2). The stereoselectivity of the formation of (**4**) was determined by GLC either directly or after stereospecific transformation of (**4**) to 1-arylpropenes (*erythro* ⟶ *trans*, *threo* ⟶ *cis*) by a literature method.[7]

Compound (**4f**) was then treated with 5 equiv. MCPBA in methanol at reflux temperature for 3 h. Normal work-up procedure of the resulting pale yellow homogeneous solution afforded methyl phenylacetate (**8f**) in 86% yield based on (**4f**) together with a small amount of phenylacetic acid (**7f**). Similar treatment of compound (**4a**) gave the corresponding methyl ester (**8a**) quantitatively, while after 5 h at 25 °C (**7a**) was obtained as the main product (40% yield), together with (**8a**) and 2-phenylpropanal. When the oxidation was carried out with tetrahydrofuran (THF) as solvent in place of methanol at room temperature, the acid was obtained selectively; thus, from (**4d**) and (**4e**) the corresponding acid (**7**) was obtained in high yields. Typical results are shown in Table 3. Taking into account the previously proposed scheme for MCPBA oxidation of selenides,[1,8] we propose that the present reaction proceeds *via* a selenone–MCPBA adduct intermediate (**A**) in which aryl migration occurs to give a carbenium ion, leading to an aldehyde as shown in Scheme 3. This aldehyde can then be oxidized by MCPBA, as has long been known,[9] and the esterification to (**8**) follows. In a separate experiment we found that 2-phenylpropanal was converted to (**7a**) in 76% yield by treatment with 2 equiv. MCPBA in methanol at reflux temperature for 2 h, but that the ester (**8a**) was formed in only 1% yield. This result suggests that the esterification in our system must be catalysed by the benzeneselenonic acid ($PhSeO_3H$) generated *in situ*.

Experimental

^1H NMR spectra were recorded with Varian EM-360 (60 MHz), JEOL FX-90 (90 MHz), and JEOL FX-100 (100 MHz) instruments on solutions in $CDCl_3$ with Me_4Si as an internal standard. ^{13}C NMR spectra were taken at 25.1 MHz with a JEOLCO ^{13}C Fourier transform NMR system (JNM FX-100) and were recorded on solutions in $CDCl_3$, after 350–1 000 pulses with intervals of 0.8–3.0 s. IR spectra were recorded with a Shimadzu IR-435 spectrometer. GLC analyses were carried out using a Shimadzu 4CMPF apparatus using DC QF-1 (5%)-Chromosorb W(1 m) column and OV-101 (0.24 mm × 30 m) and DB-1 (0.53 mm × 15 m) capillary columns. Melting points

were determined with a Yanagimoto MP micro melting point determination apparatus and were uncorrected.

All organic and inorganic materials were commercial products. Diphenyl diselenide (purity *ca.* 99%), benzeneselenenyl chloride (purity *ca.* 98%), aluminium isopropoxide (purity *ca.* 98%), and di-isobutyl aluminium hydride (1.0M solution in THF) were obtained from Aldrich Chemical Co. 4′-Isobutylpropiophenone (**2d**) and 4′-phenylpropiophenone (**2e**) were prepared by Friedel–Crafts reaction of propionyl chloride with isobutylbenzene and biphenyl, respectively.

Preparation of (**4a**) *and* (**4f**) *from Benzaldehyde* (**6**) *and* PhSeCH(R)Li (*Table* 1).—To a solution of $(PhSe)_2CH_2$[10] (1.63 g, 5.0 mmol) in THF (10 ml) was slowly added BuLi in hexane (2.9 ml, 5.4 mmol) at −78 °C under N_2, and the resulting yellow homogeneous solution was stirred at the same temperature for 1 h. A solution of (**6**) (0.53 g, 5 mmol) in THF (5 ml) was then added to the above solution at −78 °C and the resulting solution was stirred for 2 h at this temperature and for a further 2 h at room temperature. It was poured into aqueous NH_4Cl solution (150 ml), extracted with $CHCl_3$ (3 × 50 ml), and the extract was washed with brine and dried over $MgSO_4$. Evaporation of the solvent left a yellow liquid which was subjected to column chromatography on SiO_2 (Wakogel C-200) to give BuSePh (0.91 g, 85%) (hexane as eluant) and a pale yellow oil of 2-(phenylseleno)-1-phenylethanol (**4f**)[11] (1.13 g, 82% yield) (hexane–ethyl acetate, 10:1 as eluant).

Similarly, 2-(*phenylseleno*)-1-*phenylpropanol* (**4a**) was prepared using $(PhSe)_2CHMe$[6] (0.68 g, 2 mmol) in place of $(PhSe)_2CH_2$. The product was a pale yellow liquid (0.45 g, 78%) and existed as a mixture of diastereoisomers (*erythro*:*threo* = 45:55 determined by GLC); δ_H(60 MHz) 1.20 (3 H, d, *J* 7.2 Hz), 1.23 (3 H, d, *J* 7.2 Hz), 2.76 (1 H, d), 3.25 (1 H, d), 3.35–3.8 (2 H, m), 4.43 (1 H, dd, *J* 9.2 Hz), 4.77 (1 H, dd, *J* 3.6 and 2 Hz), 7.1–7.5 (16 H, m), 7.5–7.8 (4 H, m).

Preparation of (**3**) *by Phenylselenenylation of Propiophenones* (*Table* 1).—Compounds (**3**) were generally prepared in 5–10 mmol scale by treatment of propiophenones with benzeneselenenyl chloride in ethyl acetate at 25 °C for 24 h, according to a literature method,[3] and isolated either by column chromatography [SiO_2, hexane–ethyl acetate (20:1) as eluant] or by recrystallization. Compound (**3a**)[1]; δ_H(90 MHz) 1.53 (3 H, d, *J* 7 Hz), 4.50 (1 H, q, *J* 7 Hz), 6.90–7.35 (8 H, m), 7.50–7.80 (2 H, m). Compound (**3b**); δ_H(90 MHz) 1.64 (3 H, d, *J* 7 Hz), 4.59 (1 H, q, *J* 7 Hz), 7.12–7.80 (9 H, m). Compound (**3c**); δ_H(90 MHz) 1.64 (3 H, d, *J* 7 Hz), 2.41 (3 H, s), 4.67 (1 H, q, *J* 7 Hz), 7.15–7.96 (9 H, m). Compound (**3d**); δ_H(90 MHz) 0.89 (6 H, d, *J* 6 Hz), 1.61 (3 H, d, *J* 7 Hz), 1.88 (1 H, m), 2.50 (2 H, d, *J* 6 Hz), 4.66 (1 H, q, *J* 7 Hz), 6.9–8.0 (9 H, m). Compound (**3e**); a white solid, m.p. 124 °C (from hexane), δ_H(90 MHz) 1.72 (3 H, d, *J* 7 Hz), 4.75 (1 H, q, *J* 7 Hz), 7.20–8.08 (14 H, m).

Reduction of (**3**) *to the* β-*Hydroxyselenides* (**4**) (*Table* 1).—Compounds (**4**) were generally prepared in 5–10 mmol scale by treatment of (**3**) with $LiAlH_4$ in diethyl ether at −10 °C for 2 h according to a literature method,[4] and isolated by column chromatography [SiO_2, hexane–ethyl acetate (20:1) as eluant]. The compounds consisted of two diastereoisomers (*erythro*:*threo* = *ca.* 10:90) and the isomer ratios were determined by GLC either directly or after transformation to 1-arylpropenes by a literature method.[7] Only the *threo*-isomers were analysed by NMR as follows: all compounds were previously unreported.

threo-(**4a**); δ_H(100 MHz) 1.20 (3 H, d, *J* 7.3 Hz), 3.22 (1 H, d, *J* 2 Hz), 3.40 (1 H, dq, *J* 7.3 and 8.3 Hz), 4.39 (1 H, dd, *J* 8.3 and 2 Hz), 7.15–7.35 (8 H, m), 7.52–7.64 (2 H, m): δ_C(25.1 MHz) 19.3(q), 49.4(d), 77.4(d), 126.9(d), 127.2(s), 128.0(d), 128.1(d), 128.3(d), 129.0(d), 135.9(d), 141.4(s): ν_{max}(neat) 3 440 (OH) cm^{-1} (Found: C, 61.9; H, 5.7. $C_{15}H_{16}OSe$ requires C, 61.9; H, 5.5%).

threo-(**4b**), δ_H(90 MHz) 1.21 (3 H, d, *J* 7 Hz), 3.26 (1 H, d, *J* 2 Hz), 3.37 (1 H, dq, *J* 8 and 7 Hz), 4.38 (1 H, dd, *J* 8 and 2 Hz), 7.08–7.60 (9 H, m); ν_{max}(neat) 3 410 (OH) cm^{-1} (Found: C, 51.0; H, 4.4. $C_{15}H_{15}BrOSe$ requires C, 50.9; H, 4.3%).

threo-(**4c**), δ_H(90 MHz) 1.20 (3 H, d, *J* 7 Hz), 2.32 (3 H, s), 3.14 (1 H, br s), 3.40 (1 H, dq, *J* 8 and 7 Hz), 4.35 (1 H, d, *J* 8 Hz), 7.00–7.68 (9 H, m); ν_{max}(neat) 3 410 (OH) cm^{-1} (Found: C, 63.1; H, 6.1. $C_{16}H_{18}OSe$ requires C, 62.95; H, 5.9%).

threo-(**4d**), δ_H(90 MHz) 0.89 (6 H, d, *J* 6 Hz), 1.21 (3 H, d, *J* 7 Hz), 1.84 (1 H, m), 2.46 (2 H, d, *J* 6 Hz), 3.16 (1 H, br s), 3.40 (1 H, dq, *J* 8 and 7 Hz), 4.37 (1 H, d, *J* 8 Hz), 7.00–7.64 (9 H, m); ν_{max}(neat) 3 420 (OH) cm^{-1} (Found: C, 66.0; H, 7.2. $C_{19}H_{24}OSe$ requires C, 65.7; H, 7.0%).

threo-(**4e**) δ_H(90 MHz) 1.25 (3 H, d, *J* 7 Hz), 3.25 (1 H, d, *J* 2 Hz), 3.42 (1 H, dq, *J* 8 and 7 Hz), 4.32 (1 H, dd, *J* 8 and 2 Hz), 7.20–7.68 (14 H, m); ν_{max}(neat) 3 420 (OH) cm^{-1} (Found: C, 68.8; H, 5.6. $C_{21}H_{20}OSe$ requires C, 68.7; H, 5.5%).

Reduction of (**3e**) *with Various Reducing Agents* (*Table* 2).—Procedures other than with $LiAlH_4$ are described below.

(*a*) *With* $NaBH_4$. To a solution of (**3e**) (1.83 g, 5 mmol) in MeOH–water (9:1 v/v, 50 ml) at 25 °C was added sodium borohydride (0.76 g, 20 mmol) portion by portion, and the resulting solution was stirred at 25 °C for 2 h. It was poured into a mixture of ethyl acetate (50 ml) and water (50 ml), the water layer was extracted with ethyl acetate (2 × 25 ml), and the combined organic extracts were dried over $MgSO_4$. Evaporation of the solvent left a yellow liquid which was subjected to chromatography on SiO_2 (Wakogel C-200) [hexane–ethyl acetate (10:1) as eluant] to give (**4e**) (1.07 g, 58%, *erythro*:*threo* = 5:95) and 1-(biphenyl-4-yl)propan-1-ol (**5e**) (0.40 g, 38%), δ_H(90 MHz) 0.95 (3 H, t, *J* 7 Hz), 1.82 (2 H, dq, *J* 8 and 7 Hz), 1.98 (1 H, d, *J* 2 Hz), 4.62 (1 H, dt, *J* 2 and 8 Hz), 7.28–7.64 (9 H, m).

(*b*) *With* $Al(OPr^i)_3$. To a solution of (**3e**) (3.65 g, 10 mmol) in isopropanol (20 ml) at room temperature was added aluminium isopropoxide (1.09 g, 5.3 mmol), and the resulting solution was stirred at reflux temperature for 8 h. To the cooled solution was added 10% H_2SO_4 (20 ml), and the resulting white solids were filtered off. The filtrate was extracted with ethyl acetate (2 × 80 ml) and treated as above to give (**4e**) (3.42 g, 93%, *erythro*:*threo* = 28:72) and (**5e**) (0.11 g, 5%); *erythro*-(**4e**) δ_H(90 MHz) 1.28 (3 H, d, *J* 7 Hz), 2.78 (1 H, d, *J* 2 Hz), 3.66 (1 H, dq, *J* 3 and 7 Hz), 4.80 (1 H, dd, *J* 3 and 2 Hz), 7.24–7.64 (14 H, m).

(*c*) *With* Bu^i_2AlH. To a solution of (**3e**) (1.83 g, 5 mmol) in THF (12 ml) at −78 °C was added di-isobutylaluminium hydride (1.0M THF solution, 6 ml), and the resulting solution was then stirred at −20 °C for 24 h. The solvent was evaporated under reduced pressure and ethyl acetate (50 ml) and water (50 ml) were added to the residue. The organic extract was treated as above to give (**4e**) (1.60 g, 87%, *erythro*:*threo* = 7:93) and (**5e**) (0.05 g, 2%).

(*d*) *With* $Zn(BH_4)_2$. To a solution of (**3e**) (1.83 g, 5 mmol) in THF (10 ml), a solution of zinc borohydride (10 mmol) in THF (10 ml), prepared from $ZnCl_2$ and $NaBH_4$,[12] was added at 5 °C, and the resulting solution was stirred at 5 °C for 20 h. The same work-up as above afforded only (**4e**) (1.67 g, 91%, *erythro*:*threo* = 9:91).

The *erythro*:*threo* ratios given above were determined by GLC after transformation of (**4e**) to a *trans*/*cis* mixture of 1-(biphenyl-4-yl)prop-1-enes by treatment of (**4e**) (3 mmol) with triethylamine (21 mmol) and thionyl chloride (6 mmol) in CH_2Cl_2 (36 ml) at 25 °C for 12 h, according to a reported method.[7] *cis*-1-(Biphenyl-4-yl)prop-1-ene, δ_H(90 MHz) 1.92 (3 H, dd, *J* 2 and 7 Hz), 5.79 (1 H, dq, *J* 7 and 10 Hz), 6.46 (1 H, dq, *J* 10 and 2 Hz), 7.16–7.68 (9 H, m). Conditions for GLC analysis:

DB-1 (0.53 mm × 15 m) capillary column at 130–260 °C (5 °C min^{-1}); retention time, *cis* (7.77 min) and *trans* (8.36 min).

Oxidation of β-*Hydroxyselenides* (**4**) *with* meta-*Chloroperbenzoic Acid to the Propanoic Acid Derivatives* (**7**) *and* (**8**) (*Table* 3).—A typical example is as follows. To a solution of (**4e**) (0.58 g, 1.57 mmol) in THF (3 ml) was added solid *meta*-chloroperbenzoic acid (1.60 g; 80% purity; 7.85 mmol) portion by portion at 25 °C, and the solution was stirred at the same temperature for 24 h. To the resulting transparent solution was added aqueous 0.1M $Na_2S_2O_3$ (10 ml), and the products were extracted with ethyl acetate (2 × 25 ml) (first extract). The extract was washed with aqueous 1M NaOH (50 ml), and the alkaline aqueous layer was separated and then acidified to *ca.* pH 1 by adding 6M HCl. The products were again extracted with ethyl acetate (2 × 25 ml), and the extract was washed with water and dried over $MgSO_4$ (second extract). Evaporation of the solvent under reduced pressure left a white solid (1.61 g). GLC analysis of the solid after trimethylsilylation with *N*,*O*-bis(trimethylsilyl)acetamide [OV-101 (0.24 mm × 30 m) capillary column at 150–250 °C (5 °C min^{-1})] and using dibenzyl as an internal standard revealed the presence of 2-(biphenyl-4-yl)propanoic acid (**7a**) (0.376 g, 81.2%).[1]

Oxidation in methanol was similarly carried out. The ester (**8**) in the first EtOAc extract was determined by GLC after washing with aqueous NaOH, while the acid (**7**) was determined as its methyl ester (**8**) after diazomethane treatment of the second EtOAc extract.

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