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Stereochemistry and ^{13}C NMR spectra of some phenyl selenoethers obtained in phenylselenoetherification of olefinic alcohols

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^{13}C NMR spectra of some phenyl selenoethers, obtained from phenylselnoetherification of the corresponding unsaturated alcohols, have been recorded and their stereochemistry has been studied. By using the MMX (PC Model) force field (extended with some new parameters) a theoretical study of the prepared phenylselenotetrahydropyrans has been also performed.

Intramolecular cyclization of unsaturated alcohols to cyclic phenyl selenoethers (termed *phenylselenoetherification*) by means of organoselenium reagents has become an important tool for the synthesis of different natural products containing tetrahydrofuran and tetrahydropyran system[1]. In continuation of our studies on the intramolecular cyclization of alkenols[1-3], we have now investigated the stereochemistry and ^{13}C NMR spectra of phenyl selenoethers obtained in this functionalization reaction by means of phenylselenenyl halide (PheSeCl and PhSeBr) or by means of phenylselenenyl ions, electrochemically generated from diphenyl diselenide. The reactive Intermediate, $PhSe^+$ ion, was produced by the indirect oxidation on an anode, wherein the role of mediator was taken bya halide ion[2]. The determine the stereochemistry of phenyl selenoethers obtained in these reactions, their ^{13}C NMR spectra were recorded and studied. The reactions with PhSeX, and with phenylseleneyl ions (electrochemically generated from diphenyl diselenide) were performed under experimental conditions discussed earlier[1, 2]. The results obtained are given in Table I-VIII and can be summarized as follows.

The most simple Δ^3 -alkenols, such as 3-buten-1-o1 and 4-penten-2-o1, do not undergo cyclization under these conditions, but the simplest terminally disubstituted Δ^3 -alkenol, 4-methyl-3-penten-1-o1 **1** gave the corresponding five-membered cyclic ether as the only one possible stereoisomer **2** (Table I).

The Δ^4 -alkenols on reaction with PhSeX and the electrochemically produced $PhSe^+$ offored the cyclic phenyl selenoethers of tetrahydrofuran or tetrahydropyran. The results obtained also show that the substituents at the double bond and at the carbinol C-atom have a pronounced influence on the regio- and stereoselectivity of ring closure. Thus, the terminally unsubstituted Δ^4 - alkenols **3, 5** and **7** afforded exclusively the cyclic phenyl selenoethers of tetrahydrofuran (**4, 6** and **8**, Table I, Scheme I). In the case of alkenol **5**, two stereoisomers, *cis*-and *trans*-5-methyl-2-(phenylselenomethyl) tetrahydrofuran (*cis*-**6** and *trans*-**6**) (Table I) have een isolated in 1:1 proportions. Their stereochemistry was determined by ^1H and ^{13}C NMR spectral data.

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*)-4-hexen-1-ol **9** afforded *erythro*-2-[1-(phenylseleno) ethyl]tetrahydrofuran (*erythro* **10**) as the main product, along with a small amount of the *threo*-isomer (*threo*-**10**) (Table I)[4]. In the case of (*E*)-4-hexen-1-ol **11**, *trans*-3-(phenylseleno)-2-methyltetrahydropyran (*trans*-**12**) was the main reaction product, the amount of *cis*-**12** being slightly higher only in the reaction with electrochemically produced $PhSe^+$. In both cases the reactions proceed regioselectively to afford only five- and six-membered cyclic ethers, respectively[4].

A significant influence of structure has been shown in the case of Δ^4 -alkenols with a terminally dimethyl-substituted double bond. Thus, the primary 5-methyl-4-hexen-1-ol **15** and the secondary 6-methyl-5-hepten-2-ol **17** were converted (in agreement with the Markownikov rule) only to six-membered cyclic phenyl selenoethers **16** and **18**, respectively. However, an increase in alkyl or alkenyl substitution at the carbinol carbon atom led to stereochemical control (anti Markownikov rule). Thus, the tertiary 2,3-dimethyl-5-hepten-2-ol **19** and linallool **22** were converted almost exclusivelly to the corresponding

Table I — Cyclization of Δ^3- and Δ^4 -alkenols using PhSeX and $PhSe^+$ electrochemically generated from PhSeSePh.

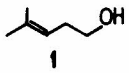
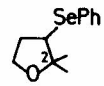
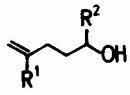
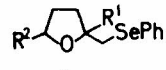
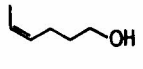
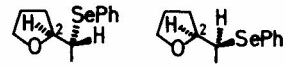
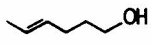
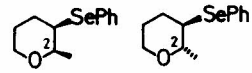
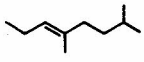
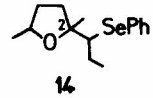
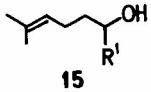
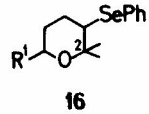
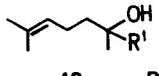
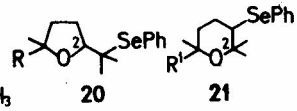
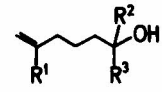
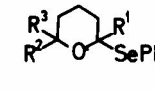
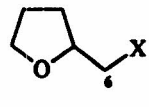
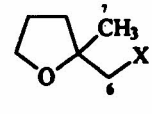
Substrate		Products		Yield (%)
				65-72
1		2		
				
3	$R^1=R^2=H$	4		63-91
5	$R^1=H, R^2=CH_3$	6		61-97
7	$R^1=CH_3, R^2=H$	8		64-93
				
Z-9		*erythro*-10	*threo*-10	72-83
				
E-11		*cis*-12	*trans*-12	65-85
				
E-13		14		75-88
				
15	$R^1=H$	16		31-70
17	$R^1=CH_3$	18		67-90
				
19	$R^1=CH_3$	20	21	37-75
22	$R^1=CH_2=CH$	23	24	46-89

Table II — **Cyclization** Δ^5 -alkenols using PhSeX and $PhSe^+$ electrochemically generated from PhSeSePh.

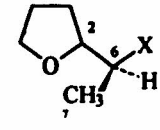
Substrate		Products	Yield (%)
			
25	$R^1=R^2=R^3=H$	26	75-88
27	$R^1=R^2=CH_3, R^3=H$	28	75-93
29	$R^1=R^2=R^3=CH_3$	30	73-76



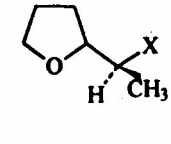
4 X = SePh
4a X = SPh
4b X = CH_3



8 X = SePH
8a X = CH_3



erythro



threo

10 X = SePh
10a X = OH
10b X = OAc
10c X = OTs

Scheme I

Table III — 50.3 MHz ^{3}C NMR ($CDCl_3$) spectral data of compounds **2, 4, 4a, 8** and **8a**

Carbon[a]	**2**	**4**	**4a**[b]	**4b**[c]	**8**	**8a**[c]
2	82.1 s	78.0 d (148)	77.4	80.8	82.5 s	82.8
3	50.5 d (146) [73.4]	31.3 t (131)	31.0	36.6 t	36.6 t (130)	36.5
4	34.4 t (133)	25.7 t (120)	25.9	25.8	26.2 t (133)	26.3
5	64.4 t (147)	68.0 t (146)	68.4	67.7	67.7 t (145)	67.1
6	26.9 q (127)	32.8 t (142) [65.5]	38.9		39.8 t (141)	33.8
7	23.8 q (127)				26.3 q (126)	25.2
1'	128.9 s	130.1 s	136.5		131.4 s	
2'	133.8 d (162)	132.2 d (161)	129.3		132.2 d (161)	
3'	129.6 d (161)	128.7 d (161)	128.9		128.8 d (161)	
4'	127.2 d (161)	126.5 d (161)	126.0		126.4 d (161)	

[a] Chemical shifts are given in δ, ppm. Coupling constants J_{CH} (in Hz) are given in parentheses. Coupling constants J_{CSe} (in Hz) are given in brackets.
[b] Data have been taken from reference 8.
[c] Data have been taken from reference 11.

Table IV — 50.3 MHz ^{13}C NMR ($CDCl_3$) spectral data of compounds **6** and **14**b

Carbon[a,b]	**6**		**14**	
	cis	*trans*	*cis*	*trans*
2	78.3 d (149)	77.6 d (149)	86.2 s	86.3 s
3	33.8* t (132)	33.3* t (140)	38.5 t (132)	37.6 t (131)
4	333.2* t (142)	32.8* (130)	33.7 t (130)	33.3 t (131)
5	75.9 d (144)	75.2 d (143)	75.2 d (144)	74.7 d (144)
6	32.2* t (131)	31.2* t (132)	61.2 d (141) [68.3]	61.2 D (141) [68.3]
7	21.2 q (125)	21.0 (125)	25.1 t (124)	24.7 t (127)
8			13.6 q (126)	13.7 q (126)
9			24.4* q (126)	23.3* q (126)
10			21.6 q (126)	21.7 q (126)
1'	130.3 s (161)	130.1 s	131.9 s	132.0 s
2'	132.3 d (162)	132.2 d (162)	133.6 d (165)	133.5 d (165)
3'	130.0 d (160)	128.8 d (161)	128.8 d (164)	129.0 d (164)
4'	126.6 d (161)	126.5 d (161)	126.7 d (163)	126.7 d (165)

[a] See Table III.
[B] Assignments marked with asterisk are tentative and could be interchanged

five-membered cyclic phenyl selenoethers **20** and **23**, respectively. This could play an important role in the synthesis of tetrahydrofuran-type natural products.

In the reactions of primary and secondary Δ^5 -alkenols with $PhSe^+$, hydroxyl-group participation is regioselective, affording the corresponding six-membered cyclic phenyl selenoether (Table II) as the only cyclization products.

The ^{13}C NMR spectra (Table III-VIII) of phenyl selenotetrahydrofurans and -tetrahydropyrans (Scheme I-IV) were recorded and studied to decipher their stereochemistry.

Using the MMX (PC MODEL) force field

Table V — ^{13}C NMR (CD_3COCD_3/C_6D_6) spectral date (chemical shifts in δ, ppm of compounds **10, 10a, 10b** and **10c** recorded at 50.3 (22.6, 20.1) MHz.

carbon[a]	**10** (at 50.3 MHz)			**10a**[b] (at 22.6 MHz)		**10b**[b] (at 22.6 MHz)		**10c**[b] (at 20.1 MHz)	
	er	*er*	*thr*	*er*	*thr*	*er*	*thr*	*er*	*thr*
	(CD_3COCD_3)	($CDCl_3$)	($CDCL_3$)	($CDCL_3$)	($CDCL_3$)	($CDCl_3$)	($CLCL_3$)	($CDCl_3$)	($CDCl_3$)
2	82.9 d (145) [11.0]	82.2 d	76.0 d	83.3	84.1	80.7	80.7	80.8	80.8
3	29.7 t (132)	29.0 t	30.8 t	26.1	28.1	27.5	28.1	26.9	27.4
4	26.7 t (134)	26.1 t	22.2 t	25.1	26.3	25.8	26.0	25.7	25.9
5	69.2 t (145)	68.5 t	68.2 t	68.6	68.1	68.9	68.4	68.6	68.3
6	44.7 d (142) [62]	43.6 d [61.9]	50.5 d [64]	68.2	70.2	72.1	72.3	80.5	80.1
7	19.2 q (128) [9.2]	18.3 q	21.0 q	18.7	19.1	15.9	16.6	17.4	17.0
1'	130.7 s [62]	128.7 s	130.5 s						
2'	134.8 d (162) [9.2]	134.6 d	133.8 d						
3'	129.7 d (162)	129.1 d	128.9 d						
4'	127.8 d (160)	127.2 d	126.9 d						

[a] See Table III
[b] Data from reference 6

Table VI — ^{13}C NMR ($CDCl_3$) spectral data of compounds **12, 12a** and **12b** recorded at 50.3 (25.2) MHz.

Carbon[a]	**12** (at 50.3 MHz)		**12a**[b] (25.2 MHz)		**12b**[b] (at 25.2 MHz)	
	cis	*trans*	*cis*	*trans*	*cis*	*trans*
2	82.6 d (146)	78.1 d (144)	75.9	77.9	74.1	75.3
3	44.0 d (141)	46.9 d (139) [66.8]	68.5	70.9	69.9	73.2
4	30.1 d (133)	32.2 t (128)	30.7	31.8	28.1	28.7
5	26.0 t (133)	27.9 t (128)	20.0	25.0	20.6	24.7
6	68.2 t (146)	67.9 t (144)	67.9	66.6	68.0	66.9
7	18.6 q (128)	21.0 q (128)	17.7	17.4	17.5	17.5
1'	130.7 s	127.9 s				
2'	134.9 d (163)	135.3 d (163)				
3'	128.7 d (163)	128.8 d (163)				
4'	127.2 d (162)	127.6 d (162)				

[a] See Table III.
[b] Data from reference 6.

(extended with some new parameters) theoretical study of the prepared phenylselenotetrahydropyrans[5] was also performed. It was found that compound *trans*-**12**, *cis*- and *trans*-**18**, **26**, *cis*- and *trans*-**28**, exist only in one tetrahydropyran chair-conformation; other possible conformations are hampered by unfavourable *syn*-interactions[5].

As can be seen from Table III, the chemical shifts of carbons C-2, 3, 4 and 5 in 2-(phenylselenomethyl)tetrahydrofuran **4** are very close to the correspondng values of known congeners **4a** (with sulphur instead of selenium) and 2-ethyltetrahydrofuran **4b,** (Scheme 1). The same situation is true with compound **8** whose chemical shifts of carbons mentioned above are very near to the ones of 2-methyl-2-ethyltetrahydrofuran **8a**, (Scheme 1). Electronegative influence of selenium atom is limited to the carbon C-6 which is shifted downfield.

Chemical shifts of the carbons C-2, 3, 4, 5, 6 and 7 in *cis*- and *trans*-isomers of compound **6**, and of compound **14** (Table IV) are very similar. Considering only their ^{13}C NMR spectra, it is not possible to make differentiation between *cis*- and *trans*-isomers.

In contrast to small differences in the chemical shifts of the carbons (C-2, 3, 4, 5, 6, 7 in *erythro* and *threo* diastereoisomers of compounds **10a-10c**

Table VII — ^{13}C NMR ($CDCl_3$) spectral data (chemical shifts in δ, ppm) of compounds **16, 18, 20** and **23** recorded at 50.3 MHz

Carbon[a,b]	**16**	**18**	**20**		**23**
2	75.1 s	75.4	85.0 d (146) [133]	85.5 d (148)	85.3 d (148)
3	52.2 d (143)	50.9 d (142) [68.3]	28.0 t (132)	27.8 t (130)	27.7 t (130)
4	27.6* t (124)	29.5 t (129)	38.5 t (130)	37.8 t (133)	37.1 t (132)
5	28.8* t (130)	35.6 t (126)	80.7 S	82.7 s	83.0 s
6	61.1 t (142)	65.9 d (138)	49.4 s [61.7]	49.5 s	49.7 s
7	19.6 q (126)	19.3 q (126)	26.1* (128) [10.1]	26.7* q	26.5* q
8	29.7 q (126)	30.5 q (128)	25.8* q (130) [8.7]	26.3* q	25.8* q
9		22.2 q (126)	27.6 q (126)	25.6	26.5
10			28.4 q(126)	144.0 d (153)	143.6 d (152)
11				111.1 t (157)	111.1 t (157)
1'	129.7 s	129.8 s	127.2 s	127.3 s	127.2 s
2'	134.3 d (162)	134.3 d (162)	138.3 d (166)	138.4 d (165)	138.4 d (165)
3'	128.9 d (160)	128.9 d (159)	128.2 d (162)	128.4** d (162)	128.3** d (162)
4	127.3 d (161)	127.5 d (161)	128.1 d (161)	128.3** d (162)	128.2** d (162)

[a] See Table III.

[B] Assignment marked with asterisk are tentative and could be interchanged.

Table VIII — ^{13}C NMR ($CDCl_3$) spectral data of compounds **26, 28** and **30** recorded at 50.3 MHz

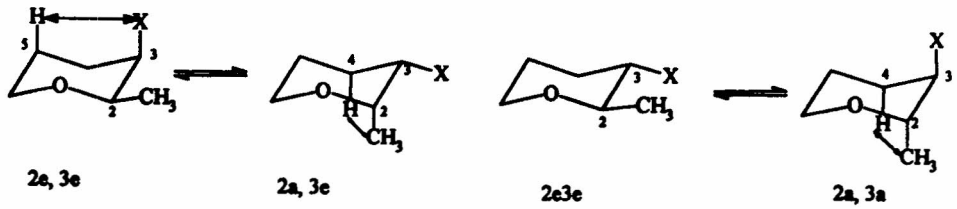
Carbon[a,b]	**26b**[c]	**26**		**26a**[d]	**28**				**30**	
					cis		***trans***			
2	73.9	76.8 d	(141)	76.4	73.6 s		73.2 s		73.1 s	
3	33.9	31.4 t	(127)	31.2	33.6 t	(127)	34.1 t	(127)	36.1 d	(122)
4	23.8	23.0 t	(127)	23.3	19.8 t	(127)	19.5 t	(127)	16.3 t	(128)
5	26.1	25.5 t	(126)	25.9	33.0 t	(127)	32.4 t	(127)	33.6 t	(124)
6	68.4	68.3 t	(142)	68.7	66.6 d	(139)	66.6 d	(139)	71.5 s	
7		33.3 t	(140)	39.6	43.2 t	(141) [68.3]	34.7 t	(141)	43.2 t	(141)
8					20.4 q	(126)	29.4 q	(123)	28.6 q	(127)
9					22.3 q	(126)	22.2 q	(126)	27.3* q	(126)
10									31.3* q	(126)
1'		130.5 s		136.9	131.8 s		131.0 s		132.0 s	
2'		132.0 d	(164)	129.0	132.0 d		132.4 d		132.0 d	(160)
3'		128.7 d	(164)	128.9	128.5 d		128.7 d		128.5 d	
4'		126.4 d	(162)	125.8	126.1 d		126.3 d		126.0 d	

[a] See Table III.

[b] Assignments marked with asterisk are tentative and could be interchagned.

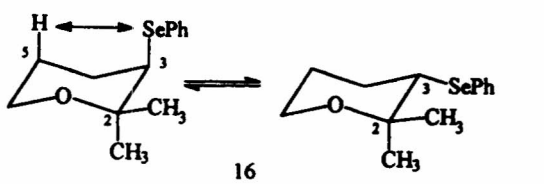
[c] Data from reference 11.

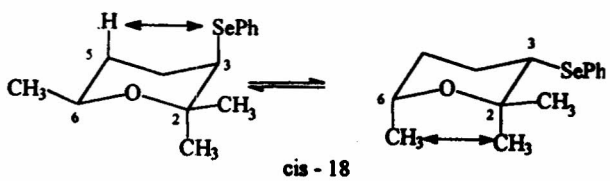
[d] Data from reference 8.

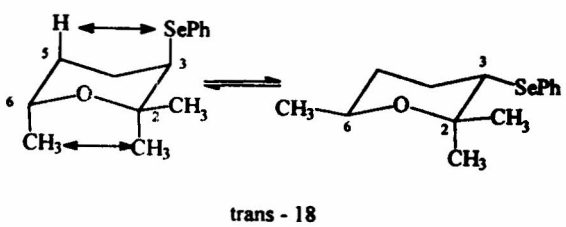


12 X=SePh
12a X = OH
12b X = OAc

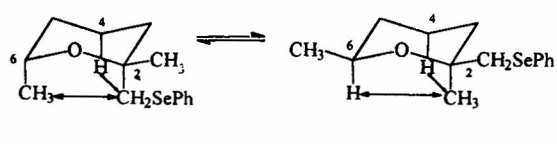
Scheme II

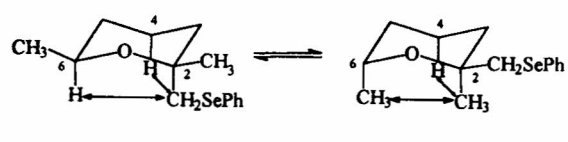


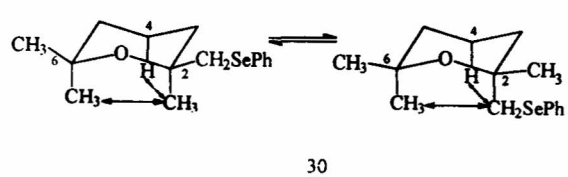




Scheme III







Scheme IV

(with X = OH, Ac and TsO, Scheme I, Table V), these differences are much greater in the same diastereisoomers of compound **10**. Probably it is due to greater steric hindrance of PhSe group in **10** in comparison to solely electronegative effect of oxygen atom from OH, OAc and OTs in compounds **10a-10c**. By analyzing their ^{13}C NMR spectra it is possible to differentiate between *erythro-* and *threo-* diastereomers of compound **10**.

The ^{13}C NMR spectra could help to differentiate between *cis-* and *trans-* 2-methyl-3-(phenylseleno)tetrahydropyran **12** (Scheme 2). As can be seen from Table VI, carbons C-4 and C-5 in *cis*-isomer care shifted upfield (lower δ values) in comparison to the corresponding carbons in *trans*-isomer (highere δ values). The observed upfield shifts of C-4 and C-5 in *cis*-**12** compared to the chemical shifts of the same carbons in *trans*-**12** (Table VI), which is typical for a γ-*gauche* effect due to steric compression between axial

substituent (*i.e.* PhSe or Me in *cis*-**12**) and the γ-positioned axial hydrogens (*i.e.* 4-H and 5-H in *cis*-**12**), clearly indicates the acial orientation of Me group at C-2 or PhSe group at C-3 in *cis*-**12** (2_e, 3_a or 2_a, 3_e conformations) and the equatorial geometry of the same groups in the *trans*-isomer (*trans*-**12**). This together with the chemical shifts of the remaining carbons, which are in good agreement with the corresponding calculated values, obtained using empirical parameters for the methyl substituents fits very well to the 2_a-Me, 3_e-SePh or 2_e-Me, 3_a-SePh in the isomer *cis*-**12** and 2_e-Me, 3_e-SePh in *trans*-**12**. Conformational equilibrium (Figure 1) indicates that conformation with equatorial SePh group is slightly favoured ($\Delta\Delta G$ = 0.07 kcal/mol; $\Delta\Delta G = \Delta G_e$, where *a* and *e* refer to the axial and equatorial SePh group, respectively). Conformation with 2_a-Me, 3_a-SePh in *trans*-**12** is energetically unfavourable. The considerable difference in the chemical shifts of the carbons C-4 and C-5 could be found in *cis*- and *trans*-isomers of compounds **12a** and **12b** with OH or AcO instead of PhSe group (Scheme II; Table VI). Conformational analysis of these compounds shows that they mostly exist in only one form for *cis* and one for *trans*; other possible conformations are energetically inviable. These results are in good agreement with the fact that differences in the chemical shifts of carbon C-4 between *cis*- and *trans*-isomers of compounds **12a**and **12b** are less, and considerable large in the case of carbon C-5 in *cis*- form. Besides γ-*gauche* effect, the electronegativity of substituents bonded to C-3 (OH or OAc) also influences the difference in the chemnical shifts of C-5.

The ^{13}C NMR spectra of compounds **16** and **18** (Table VII) are very similar. Calculations performed on the molecule **16** reveal that the conformer with equatorial SePh group is more stable by 2.20 kcal/mol. The difference between the chemical shifts of C-6 (ca. 5 δ) in **16** and **18** is due to the influence of methyl group at C-6 in **18** (Scheme III). According to available ^1H and ^{13}C NMR spectra of compound **18** it is not possible to determine its stereochemistry (*cis* or *trans*) because its counterpart needed for the differentiation was not available. According to conformational analysis it could be the *trans*-isomer but it is difficult to prove it. It is found that *trans*-**18** exists in only one tetrahydropyran chair-conformation; other possible conformations are hampered by unfavourable *syn*-interactions[5].

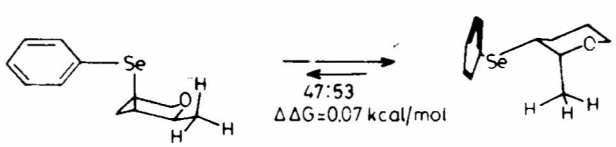


Figure 1 —Conformaitonal equilibrium of *cis*-2-methyl-3-(phenylseleno)tetrahydrophyran. Calculated conformational free energy and axial/equitorial ratio are given.

The ^{13}C NMR data of compounds **20** and **23** (Table VII) are very similar. It is not possible to find greater differences in the chemical shifts of the corresponding carbons in *cis*- and *trans*-isomers of compound **23**. Some chemical shifts marked with asterisks are so close that they could be differentiated only with difficulties. Calculations performed on the molecule **21** reveal that the isomer with equatorial SePh group is more stable by 2.41 kcal/mol.

As can be seen from Table VIII, chemical shifts of the corresponding carbons in compound **26** and its congener **26a** (with sulphur instead of selenium, Scheme IV)) are very close. On the basis of the chemical shift for the carbon C-7 of compound **26**, we can conclude that conformation with axial CH_2SePh group is favoured (γ-*gauche* efect). This conclusion is not in agreement with the conformational analysis performed by molecular mechanics[5] but it is in agreement with the conformational analysis done by semiempirical methods ($\Delta\Delta G$ = 3.86 kcal/mol)[12].

The chemical shifts of carbon atoms for compounds **28** and **30** (Table VIII) are in good agreement with the results of conformational analysis[5]. The most stable conformation for *cis*-**28** is that with equatorial and for *trans*-**28** that with axial CH_2SePh grop. In the case of compound **30** conformation with equatorial CH_2SePh group is favoured (Scheme IV). The large difference between the chemical shifts of carbon C-7 in *cis*- and *trans*-isomers of compound **28** could be explained as due to their sterically different conformations (Scheme IV). In *trans*- **28** there are interferences between $PhSeCH_2$ group and axial 4-H and 6-H. The conformational free energy of **30** ($\Delta\Delta G$ = 0.55 kcal/mol) is in favour of the

conformation with equatorial CH_2SePh group. It is obvious that 1,3-diaxial repulsions between the two CH_3 group are comparable to those between CH_3 and CH_2 SePh groups.

It could be concluded that ^{13}C NMR spectra of phenylselenotetrahydrofurans are very similar to the spectra of their congeners and in these cases PhSe group does not have much influence. In contrast to this in some (more rigid) phenylselenotetra-hydropyrans it is possible to differenttiate between their *cis*- and *trans*-isomers on the basis of γ-*gauche* effect.

Experimental Section

Column chromatography: It was carried out on Flüka silica gel 60, particle size 0.063-0.200 mm.

Thin layer chromatography (TLC): It was performed on Merck TLC aluminium sheets using silica gel 60 F_{254} (layer thickness 0.2 mm).

^{13}C NMR spectra: These spectra were recorded on a Bruker 200 AM (at 50.30 MHz) spectrometer using $CDCl_3$ or CD_3 COCD as solvent.

Preparation, purification and high resolution ^1H NMR spectra of the phenylselenoethers used in this study have been described in our previous publication[1,2].

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