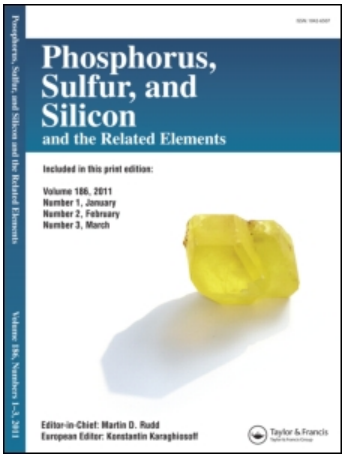

Phosphorus, Sulfur, and Silicon and the Related Elements

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Some Chiral and Achiral Low- and High-Coordinated Selenium and Tellurium Compounds: Syntheses, Structural Determination, and Selected Synthetic Applications

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New procedures for selected conversions mediated by a dichloroselenurane/triphenylphosphine system are described. The structure of tert*-butylphenylphosphinoselenoic acid in a solution phase and in the crystalline state and isolation of the enantiomers of the C_2-symmetric hypervalent selenurane and tellurane are reported. Attempts to rationalize the observed reaction courses are briefly discussed.*

Keywords Absolute configuration; alkyl chlorides; C_2-spirochalcogenuranes; dichloroselenuranes; optical resolutions; phosphinoselenoic acids

INTRODUCTION

The very rapid development in synthetic methodology and asymmetric synthesis which has been observed in the last three decades is undoubtedly connected with the growing use of achiral– and chiral–heteroatom-containing reagents. Due to the specific reactivity of these compounds it became possible to introduce and interconvert different functional groups even into the very complex chemical structures in chemo-, regio-, and stereoselective manner under rather mild reaction conditions.[1] Simultaneously, optically active compounds, especially derivatives with a stereogenic sulfur,[2] selenium,[3] and phosphorus[4–7] atoms are extensively used both as models in mechanistic studies and as chiral auxiliaries in stoichiometric and catalytic versions of asymmetric synthesis. Continuing our interest in the chemistry of such achiral– and chiral–heteroatom-containing compounds, we have recently carried out structural determinations and used as reagents of choice some new model selenium- and tellurium-containing compounds. In this account, our recent results related to these topics will be presented. In particular, we will discuss the following:

a) The structure of *tert*-butylphenylphosphinoselenoic acid in a solution phase and in the crystalline state.
b) Selected conversions mediated by the dihalogenoselenuranes/triphenylphosphine system.
c) Attempts to isolate optically active *ortho*-carboxyphenyl-phenyl selenoxide.
d) Isolation of the enantiomers of the C_2-symmetric hypervalent selenurane and tellurane.

THE STRUCTURE OF *tert*-BUTYLPHENYLPHOSPHINOSELENOIC ACID IN A SOLUTION PHASE AND IN THE CRYSTALLINE STATE

A detailed examination of the pool of chiral organophosphorus derivatives clearly indicates that the synthesis and characterization of

phosphines have recently attracted much more attention than other groups of the P-stereogenic phosphinic acid derivatives.[4–7] This observation also is valid for a family of the structures containing heavy atoms such as sulfur or selenium in spite of the fact that they can be considered as useful stoichiometric and catalytic auxiliaries. For example, the enantiomeric *tert*-butylphenylphosphinothioic acid and *tert*-butylphenylphopshinoselenoic acid could be used as a useful chiral-solvating agent for the NMR determination of the enantiomeric excesses of a variety of optically active organic compounds with the stereogenic carbon or heteroatom.[8–11] To have a deeper insight into the phenomenon of chiral discrimination for the selected group of the P-stereogenic phosphinic acid derivatives, the equilibrium between tautomeric structures and their dominant conformation recently have been determined for *tert*-butylphenylphosphine oxide and *tert*-butylphenylphosphinothioc acid using theoretical and experimental Vibrational Circular Dichroism (VCD) spectra[11,12] and specific rotation.[13,14] Since such information has not been available for *tert*-butylphenylphosphinoselenoic acid **1** we have measured[15] the VCD of the levorotatory enantiomer and racemic mixture of this selenoacid and undertaken the state-of-art theoretical VCD investigations using the density functional method and different basis set (see Figure 1 and Table I). Taking into account the results of these studies we were able to establish the absolute configuration, predominant conformation, and tautomeric structures of the chiral selenoacid **1**. The assignment of absolute configuration was independently confirmed by undertaking additional studies involving quantum-mechanical prediction of specific rotation and X-ray analysis of the levorotatory enantiomer. It is interesting to note that while all these three methods are in agreement with regard to the absolute configuration, there are significant differences among the structures deduced from solution and crystalline phase. Thus, all three methods used, namely VCD, specific rotation, and X-ray diffraction, independently conclude that (−)-*tert*-butylphenylphosphinoselenoic acid is of (*S*)-configuration and this assignment is in agreement with the literature.[16–18]

Moreover, the tautomeric structure **1A** with conformation a2 (Figure 1) is dominant for this enantiomer in the $CDCl_3$ solution. Evidence for a minor proportion of the tautomeric structure **1B** in the $CDCl_3$ solution is present in the absorption spectrum, but there is no evidence for predominance of dimers in solution. This is in contrast to the solid state where X-ray diffraction data clearly show the presence of dimers stabilized by the intramolecular hydrogen bonds *via* the O–H···Se interactions (Figure 2). The structure of the isolated dimer, as predicted by B3LYP/6-31G* calculation [Table II], is significantly

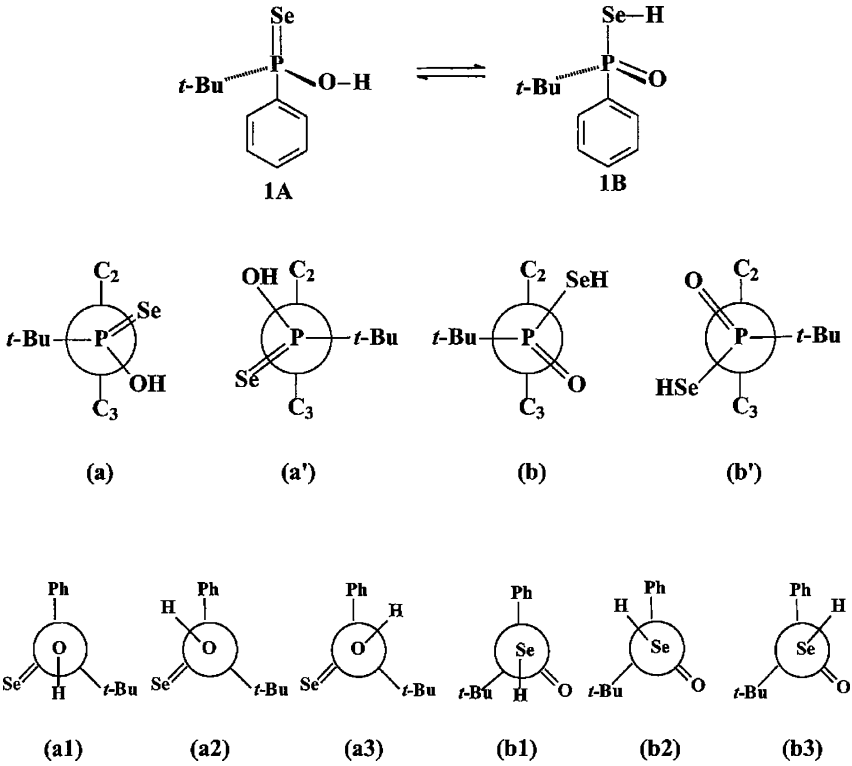


FIGURE 1 Tautomeric structures and different conformations of (*S*)-*tert*-butylphenylphosphinoselenoic acid **1**.

TABLE I Conformations and B3LYP/6-31G* Energies of Monomeric (*S*)-*tert*-Butylphenylphosphinoselenoic Acid

label[a]	Dihedral angles[b]				Energy[c]		ΔE^d (kcal/mol)	Pop.[e] (%)
	D_1	D_2	D_3	D_4	Electronic	Gibbs		
a2	102.0	−26.5	−151.3	−18.4	−3206.112484	−3205.922812	0	97.6
b3	88.2	−18.4	−145.7	49.7	−3206.105601	−3205.919050	2.4	1.9
b1	72.4	−38.8	−162.7	−56.1	−3206.103462	−3205.917785	3.1	0.5
b2	99.5	−14.0	−135.0	−160.0	−3206.100941	−3205.913916	5.6	0

[a]See Figure 1 for the labels.

[b]Dihedral angles are in degrees. D_1 is for C*t*-P-C_1-C_2, D_2 for C_2-C_1-P-Se and D_3 for C_2-C_1-P-O. D_4 stands for Se-P-O-H for the tautomeric form **A** and O-P-Se-H for the tautomeric form **B**.

[c]In Hartress.

[d]Relative energy difference in kcal/mol.

[e]Percent population based on Gibbs energies.

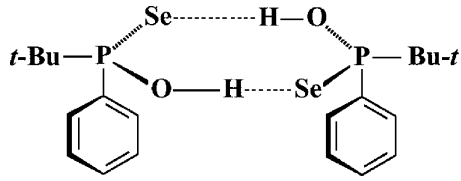


FIGURE 2 Dimeric structure of (−)-(*S*)-*tert*-butylphenylphosphinoselenoic acid **1** in the crystalline state.

different from that in the crystalline state, reflecting the dominance of packing effects over Π-Π interactions between the phenyl rings. These observations provide some guidelines to estimate the differences between solution and crystalline phase structures.

SELECTED CONVERSIONS MEDIATED BY THE DIHALOGENOSELENURANES/ TRIPHENYLPHOSPHINE SYSTEM

In dihalogenoselenuranes having a trigonal bypyramidal geometry halogen atoms tend to occupy apical positions. Due to this fact they can be considered as a good halogen transfer reagent. We have recently proved this by finding[19] that the equimolar mixture of dimethyldichloroselenurane **2a** (or diphenyldichloroselenurane **2b**) and triphenylphosphine **3** is able to convert a variety of alcohols **4** into the corresponding chlorides **5** in the reaction described by the general Eq. (1)

$$\underset{\mathbf{4}}{\mathbf{R^1OH}} + \underset{\mathbf{2}}{\mathbf{R^2_2SeC_2}} + \underset{\mathbf{3}}{\mathbf{Ph_3P}} \longrightarrow \underset{\mathbf{5}}{\mathbf{R^1Cl}} + \underset{\mathbf{6}}{\mathbf{R^2Se}} + \mathbf{Ph_3O} + \mathbf{HCl}$$

$$\text{a; } R^2 = \text{Me}$$

$$\text{b; } R = \text{Ph} \qquad (1)$$

TABLE II Comparison of Specific Rotation Predicted for (*S*)-*tert*-Butylphenyl-Phosphinoselenoic Acid with the Observed Rotation

	HF/6-31G*	B3LYP/6-31G*	B3LYP/6311++G(2d,2p)[a]	Expt
Monomer	−56	−84	−70	−50
Dimer	−287	−415		

[a]Calculation for dimer at this level could not be undertaken due to a large number of basis functions involved.

It was found that the easily available dimethyldichloroselenurane **2a** as well as diphenyl dichloro-selenurane **2b**, prepared by the reaction of the corresponding selenides **6** with sulfuryl chloride (Eq. (2)), react with alcohol **4** in the presence of equimolar amounts of triphenyl phosphine **3** leading to the clean formation of the corresponding chlorides **5**.

$$\underset{\mathbf{6}}{\mathbf{R^2_2Se}} + \mathbf{SO_2Cl_2} \longrightarrow \underset{\mathbf{2}}{\mathbf{R^2_2SeCl_2}} \qquad (2)$$

The scope of this new procedure for converting alcohols into chlorides was investigated on a number of alcohols and the results obtained are collected in Table III. Their analysis indicates the following:

a) The reaction conditions are much milder than those using Ph_3P/CCl_4 or Ph_3PCl_2 as reagent;[20,21] the latter reacts with alcohols at room temperature for 1–2 days or at 50–80°C for several h.

b) The yields of **5** are very high and in many cases the crude reaction products can be used without further purification.

c) The carbon–carbon bond geometry is fully preserved in the unrearranged product (see cinnamyl chloride **5h**)

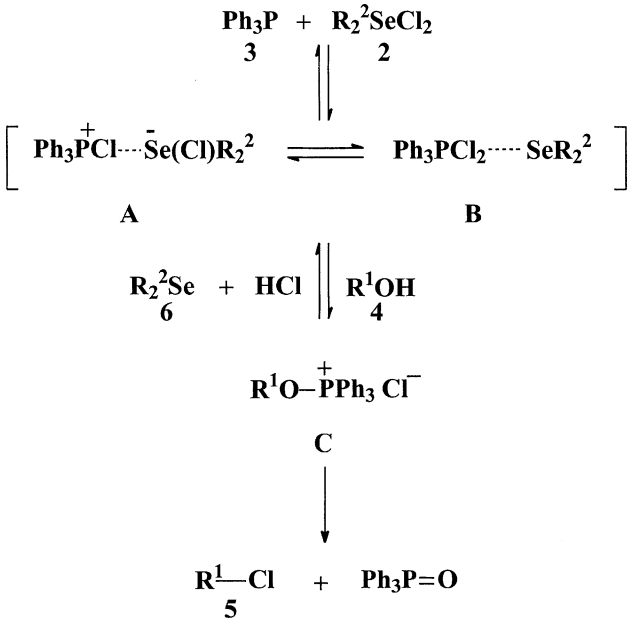
d) There are no elimination by-products in the reaction with α-phenylethanol **4g** and adamantanol **4j**.

The usefulness of this procedure for conversion of alcohols into chlorides is evident from experiments carried out with achiral cyclic and optically active substrates. At first, we checked the steric course of the reaction using cis- and trans-4-*tert*-butylcyclohexanols **4i**. It was found that trans **4i** was converted stereospecifically into cis-**5i** upon treatment

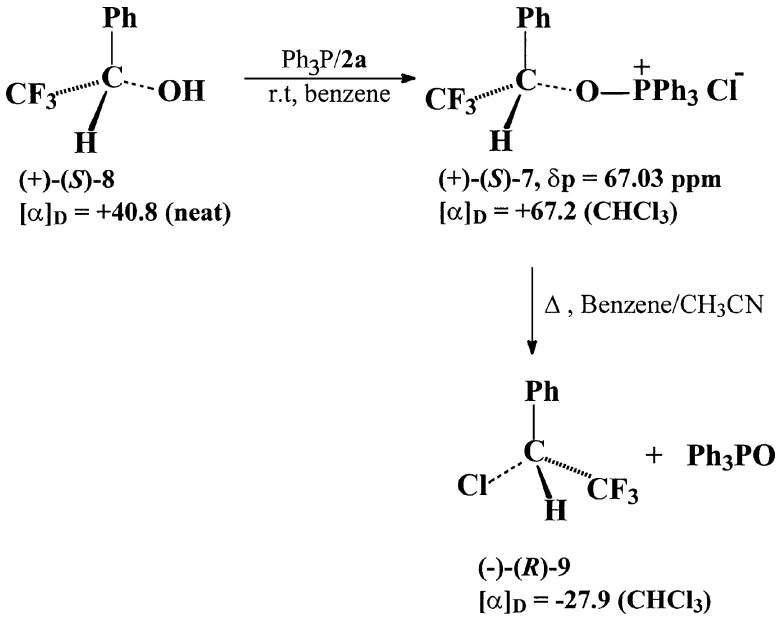
TABLE III Conversion of Alcohols R^1OH 4 into the Corresponding Chlorides R^1Cl 5 with Triphenylphosphine/Dichloroselenurane 2 System

No.	Alcohol **4** R	Selenurane	Time	Yield of **5** (%)
a	n-C_8H_{17}	**2a**	20 min.	100
a	n-C_8H_{17}	**2b**	20 ”	100
b	n-$C_{12}H_{25}$	**2a**	60 ”	92
c	$PhCH_2$	**2a**	20 ”	89
d	o-$MeOC_6H_4CH_2$	**2b**	20 ”	95
f	m-$MeOC_6H_4CH_2$	**2b**	20 ”	95
g	$PhCH_2CH_2$	**2b**	20 ”	95
h	$PhCH{=}CHCH_2$	**2a**	2 days	66
i	4-*t*-Bu-cyclo-C_6H_{10}	**2a**	20 min.	92
j	Adamantyl	**2a**	10 ”	93

with the Ph_3P/**2a** reagent system, while the reaction with cis-**4i** gave a 85:15 mixture of trans- and cis-**5i**, respectively. These results indicate that the cyclic products **5i** are formed with inversion of configuration, although its extent is dependent on the substrate stereochemistry. On the contrary, (−)-cholesterol **4k** was converted into the corresponding chloride **5k** with full retention of configuration. This stereochemical outcome is similar to those observed with other reagents and most easily can be explained assuming the S_N1 type mechanism involving the participation of the homoallylic carbonium ion.[22] In the case of acyclic and cyclic saturated, chiral alcohols both enantiomers of 2-octanol **4l** and (−)-menthol **4m** were converted to the corresponding chlorides **5l** and **5m** with essentially full inversion of configuration. To gain an insight into the reaction mechanism we studied the nature of our reagent system by means of the ^{31}P and ^{77}Se NMR spectroscopy. The results of these studies are best rationalized by assuming an equilibrium between the salt **A** and the phosphorane **B**, which is shifted toward the salt **A** in CH_2Cl_2, while in toluene the structure **B** is strongly preferred. In both equilibrium components, the phosphorus and selenium counterparts are interacting with each other. The next step of the reaction involves nucleophilic attack of an alcohol on the phosphorus atom in the salt **A** leading to the alkoxyphosphonium salt **C** which finally decomposes to form an alkyl chloride with inversion of configuration (see Scheme 1).



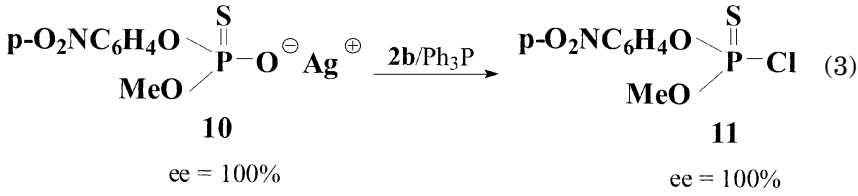
SCHEME 1



SCHEME 2

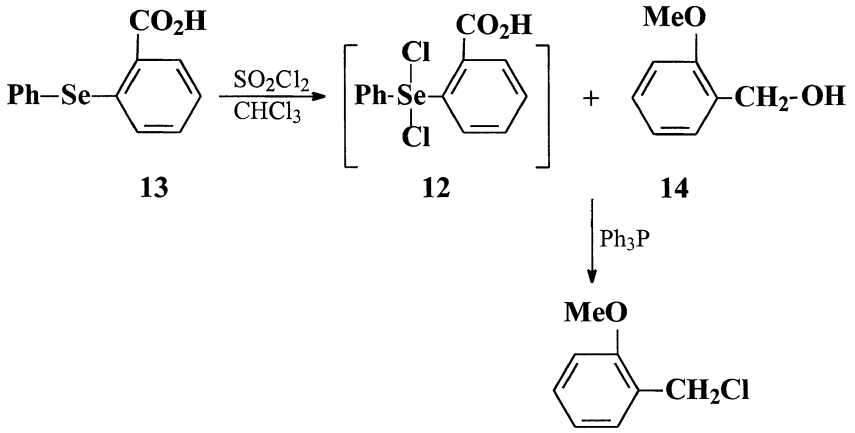
The suggested reaction course was independently supported by the isolation and full characterization of the phosphonium salt **7** formed in the reaction between (+)-(*S*)-α-(trifluoromethyl) benzyl alcohol **8** and the mixed reagent Ph_3P/2a (Scheme 2). The isolated salt **7** upon heating in a boiling mixture of benzene and acetonitrile (1:1) gave the corresponding chloride **9**.

The usefulness of the mixed reagent Ph_3P/**2a** is also demonstrated by the stereospecific conversion of the silver salt of O-methyl-O′-p-nitrophenyl phosphorothioicic acid **10** into the corresponding thionophosphoryl chloride **11** (Eq. (3)). When PCl_5 was used as a chlorinating agent, the enantiomeric excess values of the chloride **11** formed varied between 13.9% and 93.8%.[17]



10 ee = 100% **11** ee = 100% (3)

With an aim to develop a reagent system more convenient for the recovery of the starting selenide we have started experiments on the *in situ* generation of (ortho-carboxy)phenyl phenyldichloroselenurane **12** from (ortho-carboxy) 2-phenylselenobenzoic acid **13**. Preliminary experiments carried out using ortho-methoxybenzyl alcohol **14** afforded a low yield of the expected chloride **15** (Scheme 3).

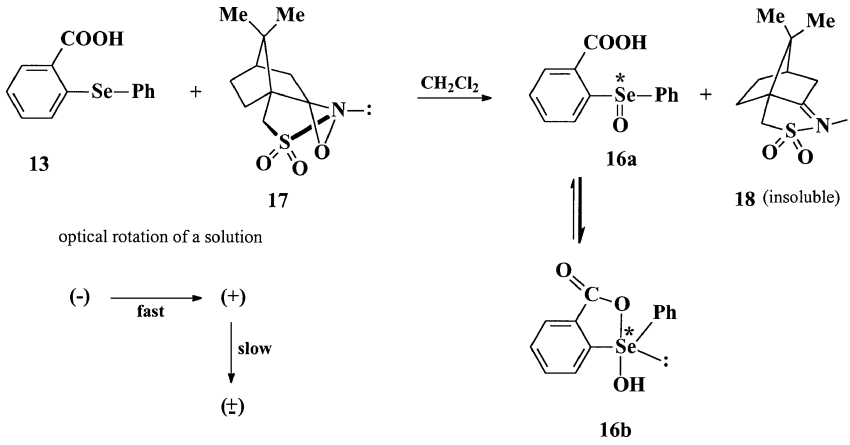


SCHEME 3

ATTEMPTS AT THE ISOLATION OF OPTICALLY ACTIVE *ortho*-CARBOXYPHENYL PHENYL SELENOXIDE

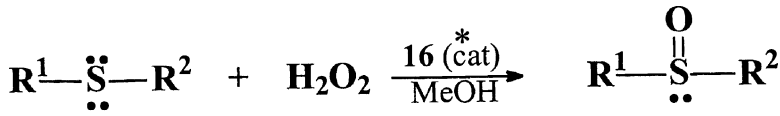
2-Phenylselenylbenzoic acid **13** contains a prochiral selenium atom. Its stereoselective oxidation should generate the chiral sterogenic center on the selenium atom in 2-phenylseleninylbenzoic acid **16a**, for which the existence of a monocyclic hydroxyselenurane structure **16b** has been suggested.[23,24] Taking into account this observation and knowing that optical stability of a few optically active seleno- and telluro-oxides is strongly enhanced by intramolecular coordination,[25] we asked ourselves if it would be possible to isolate the structures **16a** and **16b** as optically active species. The generation of these types of optically active structures should give access to a new catalyst for enantioselective oxidation of sulfides to sulfoxides based on the ecologically friendly and inexpensive hydrogen peroxide. Such a procedure utilizing the racemic structure **16** has been reported during the ICCST 8.[26]

To optically generate active selenoxide/selenurane **16a**,**b**, 2-phenylselenylbenzoic acid **13** was treated with enantiomers of the Davis



SCHEME 4

camphorsulfonyl oxaziridine **17** (Scheme 4). This led to the instantaneous oxidation of the substrate **13**, as indicated by the precipitation of the camphorsulfonylimine **18**. The removal of **18** gave a solution having an opposite and a much higher nominal value of the optical rotation in comparison with a solution of the starting oxaziridine **17**. This value slowly decreased and ended on the value which corresponds to the presence of traces of the imine **18**. Until now all of our attempts to isolate the formed selenoxide/selenurane structures **16a**,**b** as a chemically pure, still optically active species were unsuccessful. We were able, however, to support their formation as slow, racemizing, nonequivalent-enantiomeric mixtures by using them as a catalyst for the enantioselective oxidation of two prochiral sulfides **19** to the corresponding sulfoxides **20** (Scheme 5).



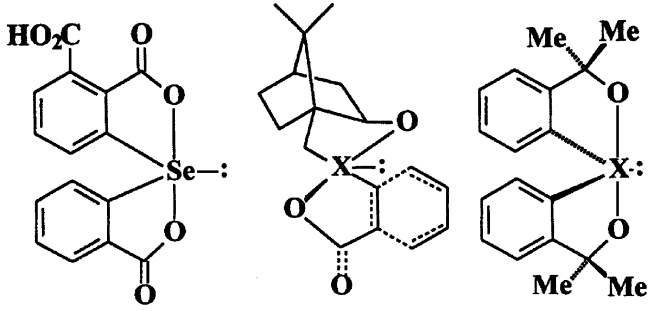
20

No	R^1	R^2	Yield (%)	$[\alpha]_{589}$ (EtOH)
a	*p*-Tol	Me	75	-6.0
b	$PhCH_2$	Me	69	-4.5

SCHEME 5

ISOLATION OF THE ENANTIOMERS OF THE C_2-SYMMETRIC HYPERVALENT SELENURANE AND TELLURANE

Spirochalcogenuranes constitute a class of hypervalent compounds which are commonly described as 10-X-4 species (X=S, Se, Te).[27] They have a trigonal bipyramidal geometry and may exhibit chirality due to molecular disymmetry even in a case where they are constructed by the use of a pair of only the same two-arms ligands.[28] The first optically active, unsymmetrically substituted selenurane **21** was partially resolved *via* classical resolution, although diastereomeric excess values for the salts used to generate the free enantiomeric acid were not determined.[30] Recently, the Koizumi group has prepared[29] a few diastereomerically pure bicyclic selenuranes **22a** (Scheme 6) and telluranes **22b** in which the exo-hydroxy-10-bornyl group serves as a chiral auxiliary. As a continuation of our interest[30–31] in the static and dynamic stereochemistry of hypervalent compounds with a stereogenic heteroatom, we have recently reported [38,39] on the successful liquid chromatographic separations of the enantiomers of the tetramethyl substituted spiroselenurane **23a** and tellurane **23b**.



21 **22 a,** X = Se **b,** X = Te **23 a,** X = Se **b,** X = Te

SCHEME 6

The stereogenic character of the chalcogenurane structures is clearly indicated by the presence of the two well-separated methyl singlets in the ^{1}H NMR and ^{13}C NMR spectra. Additional support for these structures is provided by an X-ray analysis. We have found that the enantiomers of **23a**,**b** give very well-resolved peaks when a solution of the racemate was passed through an analytical, chiral pack AS column. The mobile phases used were hexane +1 to 20% of propanol. Semipreparative separations were done by repeated injection and collections of the respective fractions from the analytical columns. This procedure

gave a few miligrams of each enantiomer, allowing further characterization by means of polarimetry and ^{1}H NMR and CD spectroscopy. The preparation of a single crystal suitable for an X-ray crystallographic structure determination is under current studies.

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