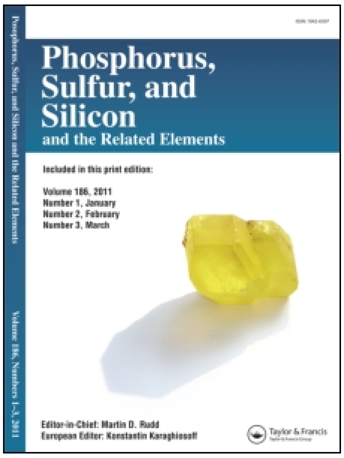

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New Sterically Congested Organoselenium and Tellurium Compounds

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NEW STERICALLY CONGESTED ORGANOSELENIUM AND TELLURIUM COMPOUNDS

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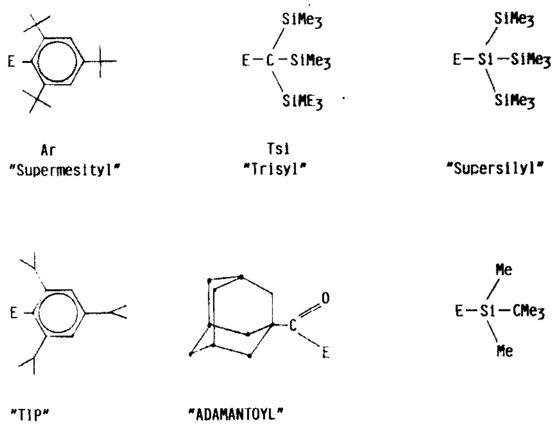
Abstract The report on recent preparative, n.m.r. spectroscopic and structural results concerning steric effects in organoselenium and -tellurium chemistry covers: 1. extremely bulky ditellurides and diselenides, including rotational barriers and unusual conformations, 2. bulky metal arylselenelolates and tellurolates, including compounds with bonds from Se or Te to Li, Na, K, Si, Ge, Sn, Zn, Cd, Hg and Au, 3. steric effects on silyltelluride/acyl halide and silylphosphane/tellurium reactions, 4. reaction of various diselenides and ditellurides with iodine and bromine, including iodine catalysis of Se-Se bond cleavage/bond formation equilibria.

INTRODUCTION

The generally accepted concepts of internal strain and back strain in organic chemistry as well as the long known correlation between coordination numbers and sizes of central atoms and ligands have in the past decade been applied in organoelement chemistry to "stabilise" unusual unsaturated species like molecular compounds with

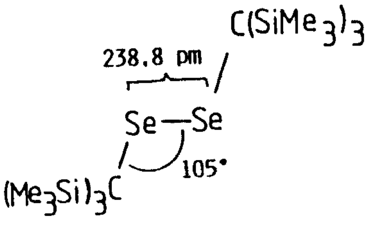
C=P, C=Si, P=P, Si=Si bonds or complexes with unusually low coordination numbers like CN1 in $2,4,6(C_6H_5)_3C_6H_2$ Cu. The comparison of M. YOSHIFUJI's diphosphene $[2,4,6\text{-}(t\text{-}C_4H_9)_3C_6H_2]_2P_2$ [1] with the bulky disulfide $[2,4,6\text{-}(t\text{-}C_4H_9)_3C_6H_2]_2S_2$ [2] reveals that the same bulky aryl substituent favours a *trans*-arrangement of the CP=PC core of the diphosphene, but appears to disfavour in case of the disulfide the *trans*-CSSC transition state, the energy of which should determine the rate of enantiomeric interconversion [2]. Corresponding bulky selenium or tellurium compounds have been nearly completely neglected until the mid 1980s, but the rising interest in low-molecular soluble metal selenium and tellurium compounds as precursors or "spectroscopic models" in semiconductor research, photochemistry and bioorganoselenium chemistry has recently stimulated several groups to join research on highly crowded sulfur, selenium and tellurium compounds. Our recent work in this field was concerned with simple bulky diselenides and ditellurides, their reduction leading to low-molecular metal chalcogenolates and their halogenation leading to low-molecular selenenyl and tellurenyl halides. Besides bulky aryls and alkyls of Se/Te, reactions of Se, Te compounds with bulky phosphino groups and silyl groups will also be reported in the following chapters.

BULKY SUBSTITUENTS ATTACHED TO E = Se, Te

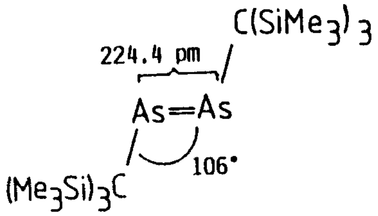


DIORGANYLDICHALCOGENIDES

Barriers of the enantiomeric interconversion of organic diselenides and ditellurides are lower than those of disulfides [2,3,4]. In all cases the largest enthalpies of activation have been determined from compounds that contain 2,4,6-tri-tert-butylphenyl substituents. Calculations have shown that in simple dichalcogenides like dimethyldiselenide enantiomeric interconversion should proceed by way of an anti-transition state (antiperiplanar conformation, point group C_{2h}) which appears electronically more favourable than a syn-conformation (C_{2v})[5,6]. On the other hand, one might expect, that extremely bulky substituents lead to internal steric repulsion and increasing torsion angles [7] which correspond to distortions in direction of the sterically more favourable anti-arrangement of the bulky substituents. Such an arrangement is the ground state geometry of bis(2,4,6-tri-tert-butylphenyl)diphosphene [1]. From this point of view, it seems surprising, that bulky substituents, favouring the anti-transition state relative to the crowded gauche-ground state still lead to very high barriers of enantiomeric interconversion. A recent dnmr study on 1,2-bis(2,4,6-tri-tert-butylphenyl)- ethane including MM2 calculations reveals that in this hydrocarbon (torsion angle Ar-C-C-Ar 119.9°) enantiomeric interconversion occurs via CH_2-CH_2 rotation highly correlated with Ar-CH_2 rotation [8]; obviously saturated 1,2-bis(2,4,6-tri-tert-butylphenyl)dielement compounds avoid anti-conformations (C_{2h}) even when there is no lone pair-lone pair interaction in play, like [2,4,6-$(t\text{-}Bu)_3C_6H_2$]$(n\text{-}Bu)_2$Sn-Sn$(n\text{-}Bu)_2$[2,4,6-$(t\text{-}Bu)_3C_6H_2$] which adopts a torsion angle of 160° [4,9]. These molecules appear "well-packed", but not really crowded in the sense of severe intramolecular repulsion. Thus even more bulky substituents will be necessary to force dichalcogenides in unfavourable anti-conformations that correspond to the transition states of the internal rotation of simple dichalcogenides. Tris(trimethylsilyl)methyl substituents and tris(trimethylsilyl)silyl substituents are obviously the right choice.



C_{2h}



C_{2h}

Solid Ditrisilyldiselenide adopts a molecular structure and conformation that is quite similar as ditrisyldiarsene [10], which contains two valence electrons and two nuclear charges less than the diselenide [11]. The Se-Se bond (2.38 Å) of $\mathrm{Tsi_2Se_2}$ is significantly longer than common Se-Se single bonds. Comparison of the two isotypic structures allows the conclusion, that from steric reasons, a shorter ("normal") Se-Se distance would be possible without severe internal strain; the As=As distance is even 0.1 Å shorter than a typical Se-Se single bond. The antiperiplanar ground state of ditrisyldiselenide implies a significant amount of (thermodynamic) activation of this molecule, compared with anticlinal diselenides like even the bulky diaryldiselenides. The unusual conformation coincides with the unusual (red) colour of ditrisyldiselenide.

anti-Tetraethylthiuramdiselenide is also a red compound, but gauche-tetracyclohexylthiuramdiselenide is yellow [12]. These two diselenides with adjacent thiocarbonyl groups contain Se-Se bonds with "normal" distances. Sladky reported that green ditrisylditelluride [13] adopts the anti-conformation, the Te-Te bond is significantly longer than in any other ditelluride [14]. Di[tris(trimethylsilyl)silyl]ditelluride being a green compound [15,16] might be another example of a ditelluride with antiperiplanar ground state conformation. In summary, bulky diaryldichalcogeneides show rather normal geometries, but very high barriers of enantiomeric interconversion; quite contrary bulky alkyls and silyls lead to antiperiplanar ground states which correspond to the transition states of the Se-Se or Te-Te bond rotation of common dichalcogenides.

BULKY SELENOLATES AND TELLUROLATES

Superhydride reduction of dichalcogenides is the choice for the preparation of pure lithium chalcogenolates; sodium amalgam or potassium tri-s-butylborohydride are also suitable reducing agents. Coordinated THF is quite labile and may be displaced by chelating ethers or TMEDA. The lithium-2,4,6-tri-t-butylphenyl chalcogenolates (S, Se, Te) crystallise as monomeric compounds with three equivalents of tetrahydrofuran attached to four-coordinate lithium [17,18,19]; the chalcogen atoms are two-coordinate. The dimethoxyethane adduct of tris(trimethylsilyl)silyltellurolithium is a Te-bridged dimer with 3-coordinate tellurium and 4-coordinate lithium. The base-free compound is a hexamer, each silyltelluro group bridges three lihtium atoms (Te: CN4, Li: CN3) [15]. Protonation of the dimeric lithium tellurolate-tetrahydrofurane adduct provides a silyltellurol of quite unusual stability.

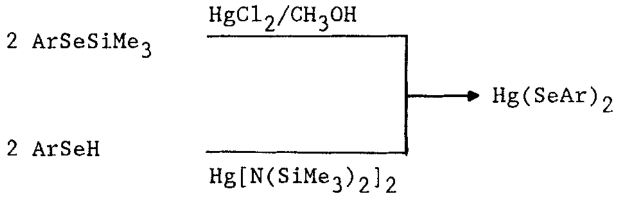
$$1/2\ [(THF)_2LiTeSi(SiMe_3)_3]_2 + CF_3SO_3H \xrightarrow{-Li^{\oplus}CH_3SO_3^{\ominus}} (Me_3Si)_3SiTeH \quad \text{(stable)}^{16)}$$

Suitable ways to bulky selenolates and tellurolates of softer metals are transmetallation reactions starting with lithium salts or trimethylsilyl derivatives [20,21]. Alternatively, tellurols (in most cases as intermediates generated at low temperature) or selenols may be reacted with reactive metal amides (the amine liberation route)[16,22].

$$\mathrm{ArSeLi(THF)_x + Me_3SnCl \xrightarrow[-THF]{-LiCl} ArSeSnMe_3}$$

$$\mathrm{ArSeLi(THF)_3 + Ph_3PAuCl \longrightarrow Ph_3P\text{-}Au\text{-}SeAr}$$

$$\mathrm{2\ ArSeH + Cd[N(SiMe_3)_2]_2 \longrightarrow Cd(SeAr)_2}$$



The bulky gold selenolate is monomeric [18,21], the corresponding phenylseleno derivative shows significant Au-Au interaction leading to "weak dimers" [23]. The bulky mercury arylselenolate is monomeric [21,22], the corresponding cadmium complex gives a monomer/dimer equilibrium [22].

From stable tris(trimethylsilyl)silyltellurol with bis(trimethylsilylamido)zinc(II) or diethylzinc, a stable monomeric zinc tellurolate was prepared. This bulky zinc tellurolate still behaves as a Lewis acid, with pyridine a tetrahedral 1:2 adduct is formed [16].

$$\mathrm{(Me_3Si)_3SiTeH + \xrightarrow[\text{or } ZnEt_2/-C_2H_6]{Zn[N(SiMe_3)_2]_2/-\ 2\ HN(SiMe_3)_2}}$$

$$\mathrm{\longrightarrow Zn[TeSi(SiMe_3)_3]_2 \xrightarrow{+\ 2\ py} (py)_2Zn[TeSi(SiMe_3)_3]_2}$$

In summary, bulky thiolates, selenolates and tellurolates tend to lead - as desired - to low-molecular derivatives with metals in low coordinations numbers, which still may behave coordinatively unsaturated towards small substrates. This work is being extended by severals groups.

STERIC EFFECTS ON SILYLTELLURIDE/ACYLCHLORIDE REACTIONS

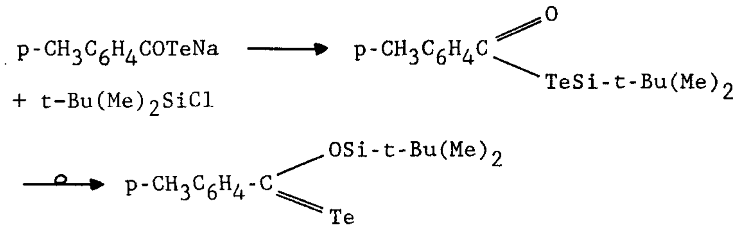
Superhydride reduction of tellurium and subsequent reaction with trialkylsilyl chlorides is the best choice for the preparation of bis(trialkylsilyl)tellurides [24]. Bis(t-butyldimethylsilyl)telluride and bis(dimethyl-i-propylsilyl)telluride are much easier to handle than bis(trimethylsilyl)telluride, but they are also much less reactive towards acylchlorides. Bulky bis(t-butyldimethylsilyl)telluride does not react within acceptable time with acetyl chloride or phthalic dichloride.

Reaction of sodium 4-methyltellurobenzoate with t-butyldimethylchlorosilane leads to a Te-Si-bonded intermediate which rearranges by 1,3-silyl shift to oxygen to give the isomer with a C=Te double bond [25].

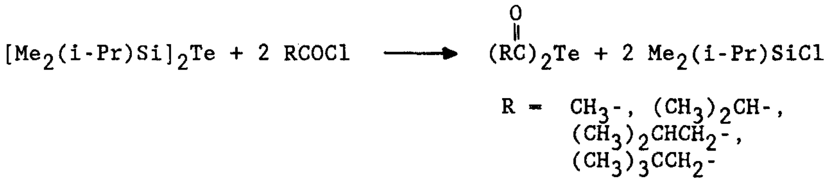
Bis(dimethyl-i-propylsilyl)telluride reacts with excess of various acyl chlorides providing diacyltellurides, but with 1-adamantoyl chloride no reaction occurs.

Pure diadamantoyl telluride is avaiable from the acid chloride with bis(trimethylsilyl)telluride. In course of this reaction, the

$$[\text{t-Bu(Me)}_2\text{Si}]_2\text{Te} + \text{CH}_3\text{COCl} \not\longrightarrow$$

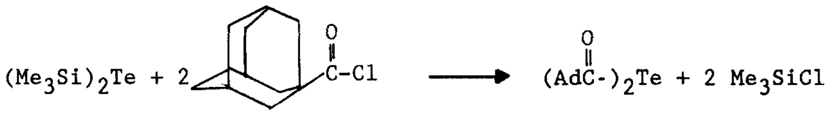


$$\text{p-CH}_3\text{C}_6\text{H}_4\text{COTeNa} + \text{t-Bu(Me)}_2\text{SiCl} \longrightarrow \text{p-CH}_3\text{C}_6\text{H}_4\text{C}(=\text{O})\text{TeSi-t-Bu(Me)}_2 \xrightarrow{\frown} \text{p-CH}_3\text{C}_6\text{H}_4\text{-C}(=\text{Te})\text{OSi-t-Bu(Me)}_2$$

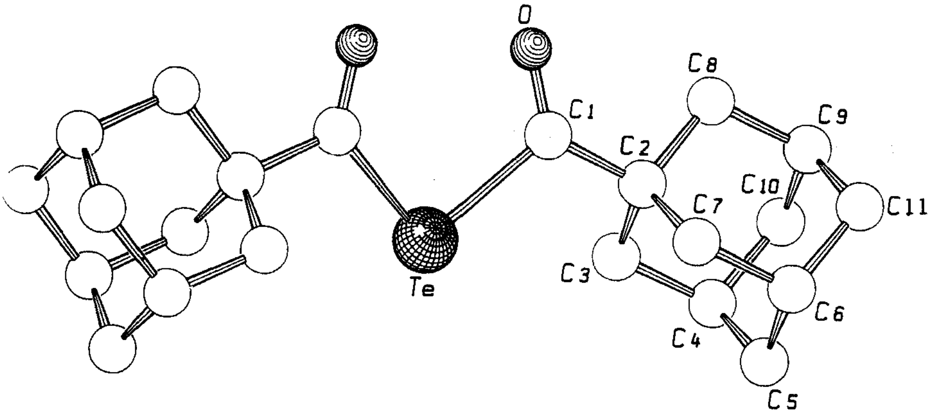


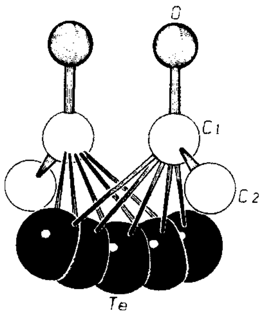
$$[Me_2(i\text{-}Pr)Si]_2Te + 2\ RCOCl \longrightarrow (R\overset{O}{\overset{\|}{C}})_2Te + 2\ Me_2(i\text{-}Pr)SiCl$$

$$R = CH_3\text{-},\ (CH_3)_2CH\text{-},\ (CH_3)_2CHCH_2\text{-},\ (CH_3)_3CCH_2\text{-}$$



mixture turns green, but finally diadamantoyl telluride was isolated as slightly yellowish crystals (orthorhombic, space group Pcca).





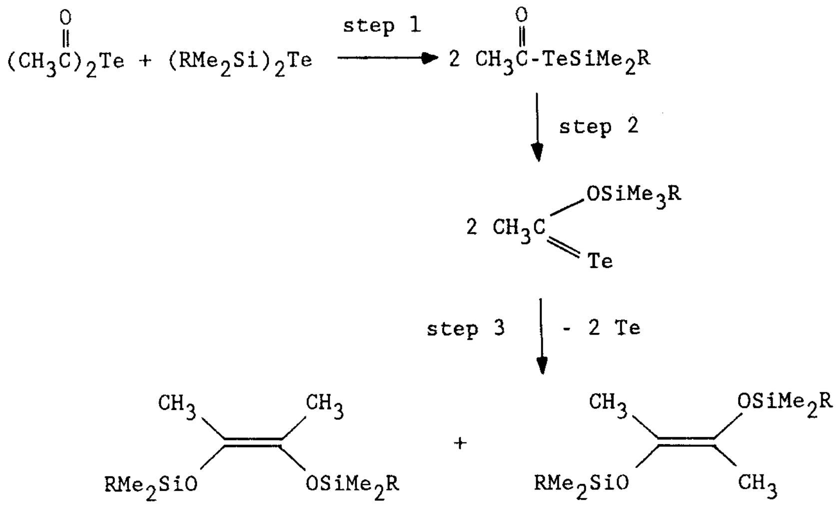
The compound shows a strange disorder of the tellurium atom between two nearly parallel carbonyl groups (torsion angle of the two CCO planes only 9.6°).

Depending on the exact position of the tellurium atom, the Te-C distance ranges from 2.19 to 2.26 Å. In any case, this Te-C bond is longer than common Te-C (alkyl) or Te-C (aryl, sp^2) bonds. Upon crystallisation of diadamantoyl telluride (telluro-1-adamantanecarbonic acid anhydride) a few crystals of 1-adamantanecarbonic acid anhydride $(AdCO)_2O$ were also obtained and structurally characterised (space group C2/c). Main difference between the molecular structures of the two compounds are the central angles C-E-C (E = O: 123.4°, E = Te: 93.4°), and the striking disorder of Te in the telluroanhydride.

Telluroanhydrides give ^{13}C-NMR-signals of the carbonyl group in the range of +195 to +205 ppm, ^{125}Te-NMR signals appear between +900 and +1250 ppm (Tab. 1).

	δ^{13} C=O [TMS]	δ^{125} Te[Me_2Te]
$(AdCO)_2Te$	205.1	911.5
$(t\text{-}BuCO)_2Te$	204.2	936
$(i\text{-}PrCO)_2Te$	204.1	1036.5
$(i\text{-}BuCO)_2Te$	199.5	909.2
$(t\text{-}BuCH_2CO)_2Te$	199.3	1240.5

The reactions of telluroanhydrides with disilyltellurides depend strongly on the steric demand of their substituents. The lifetime of telluroacetic acid silyl esters increases, when the trimethylsilyl group is replaced by the dimethyl-i-propylsilyl group, but bis(t-butyldimethylsilyl)telluride does not react at all with diacetyltelluride.



at 20°C: R = t-Bu: step 1 does not proceed

R = i-Pr: step 3 complete after 12 hrs

R = CH_3: step 3 complete within 30 min

Diadamantoyltelluride does not react with bis(dimethyl-i-propyl)telluride, but it reacts with bis(trimethylsilyl)telluride to give blue solutions that contain C=Te-bonded species ($\delta^{13}C$=Te +252.8). It appears, that like with telluropivalic acid trimethylsilyl ester, the 1,3-silyl shift leads to equilibria of the C=Te- and the C-Te-$SiMe_3$-bonded isomer. Corresponding selenoesters exist exclusively as the C=Se-bonded isomers.

Bulky, but also electron-withdrawing substituents may favour the completion the of Te to O-shift of silicon. For that reason, we looked for appropriate educts of the type $Te(SiR_nX_{n-3})_2$.

Selective ways to such new silyl tellurides might be tellurati-on reactions of functionally substituted silyl phosphanes under mild conditions.

$$\text{t-Bu}_2\text{PSiCl}_3 + \text{Te} \longrightarrow \text{t-Bu}_2\text{P-Te-SiCl}_3 \rightleftharpoons$$
$$(\text{tBu}_2\text{P})_2\text{Te} + (\text{Cl}_3\text{Si})_2\text{Te} \rightleftharpoons 1/x\ (\text{Cl}_2\text{SiTe})_x + \text{SiCl}_4$$

$$\begin{array}{lclcl} \text{i-Pr}_2\text{PSiCl}_3 + \text{Te} & \longrightarrow & (\text{i-Pr}_2\text{P})_2\text{Te} & + & (\text{Cl}_3\text{Si})_2\text{Te} \\ & & \updownarrows & & \updownarrows \\ & & (\text{i-Pr}_2\text{P})_2\text{+Te} & & 1/x\ (\text{Cl}_2\text{SiTe})_x + \text{SiCl}_4 \end{array}$$

Since $(\text{i-Pr}_2\text{P})_2\text{Te}$ is in an telluration/detelluration equilibrium with tetraisopropyldiphosphane, and tetraisopropyldiphosphane reacts under mild conditions with hexachlorodisilane to give $\text{i-Pr}_2\text{PSiCl}_3$ [26], a diphosphane-catalysed telluration of hexachlorodisilane could lead to $(\text{Cl}_3\text{Si})_2\text{Te}$ or $(\text{Cl}_2\text{SiTe})_x$. Hexachlorodisilane could also be tellurated with help from tri-tert-butylphosphane telluride:

$$\text{t-Bu}_3\text{P=Te} + \text{Si}_2\text{Cl}_6 \longrightarrow \text{tBu}_3\text{P} + \text{"}1/x\ [(\text{Cl}_2\text{SiTe})]_x\text{"}$$
$$\xrightarrow[-\ \text{SiCl}_4]{2\ \text{tBuCOCl}} (\text{tBu}\overset{\overset{\text{O}}{\|}}{\text{C}})_2\text{Te}$$
$$\delta^1\text{H}\ \ 0.9\ \text{ppm},\ \delta^{13}\text{C}\ \ 204\ \text{ppm}$$

A red solid containing a very sensitive unsoluble (probably polymeric) chlorosilyltelluride is formed; with excess of pivaloyl chloride, dipivaloyl telluride is formed in a rather straightforward reaction.

DISMUTATION REACTIONS OF DISELENIDES AND DITELLURIDES WITH IODINE, BROMINE AND DIPHOSPHANES

There are interesting analogies between the tellurium-catalysed ring expansion of cyclophosphanes and the iodine-catalysed crystallisation of hexagonal selenium [27,28]. Uncharged binary P-Te or Se-I compounds have not yet been characterised.

The three-membered cyclophosphane $(\text{t-BuP})_3$ is cleaved by di-p-tolylditelluride to give 1,2-di-t-butyl-1,2-di(p-tolyltelluro)diphosphane. Decomposition of 1,2-di-t-butyl-1,2-di(aryltelluro)diphosphanes leads to di-t-butyldiphosphene. This P=P double bonded species was trapped by cycloaddition with 2,3-dimethylbutadiene. Without trapping agent, the cyclotetraphosphane (dimer of the diphosphene) is formed.

The reaction of ditrisyldiselenide with elemental selenium requires iodine catalysis, similarly iodine catalyses reactions of elemental selenium in course of polyselenide formation [29] and the rearrangement of amorphous selenium to give crystalline hexagonal selenium [28].

$$Se_{(amorph.)} \xrightarrow[I_2\ (cat)]{} Se_{(cryst.)}$$

$$K_2Se_2 \xrightarrow[I_2\ (cat)]{Se_x,\ [222]\text{-cryp}} (K[222]\text{-cryp})_2Se_7$$

$$\begin{tikzcd}
 & TsiSeSeTsi \arrow[rr, "Se_x", "I_2\ (cat)"'] \arrow[dr, "+\ I_2"] & & TsiSeSeSeTSi & \\
\text{step 1} & & 2\ TsiSeI \arrow[ur, "1/x\ Se_x", "-I_2"'] & & \text{step 2}
\end{tikzcd}$$

Ditrisyldiselenide reacts with iodine to give trisyliodoselane, a stable black-violet compound. In presence of elemental selenium, the iodoselane reacts further to give ditrisyltriselenide and elemental iodine in an equilibrium reaction (the cleavage of ditrisilyltriselenide with iodine to give the iodoselane remains uncomplete). Due to the high energy of anti-ditrisyldiselenide, step 1 and hence the over-all reaction from diselenide to triselenide is irreversible. With less bulky substituents (like with elemental selenium) all steps are reversible, charge transfer complexes $(R_2Se_2)_2I_2$ and $(R_2Se_2)I_2$ are energetically comparable to the covalent iodoselane (RSeI) intermediates [31-33]. Aryliodoselanes require two <u>ortho</u>-alkyl substituents to be stable in solution, such substituents disfavour the formation of diselenide-diiodine charge transfer complexes [31].

Finally, it is interesting to compare a bulky but labile iodoselane with the corresponding iodotellane.

$$2\ \mathrm{TIPSeI} + \mathrm{I}^{\ominus} \longrightarrow \mathrm{TIP_2Se_2} + \mathrm{I_3}^{\ominus}$$

$$\mathrm{TIPTeI} + \mathrm{I}^{\ominus} \longrightarrow \mathrm{TIPTeI_2}^{\ominus}$$

$$\mathrm{TIPTe{*}I_2}^{\ominus} + \mathrm{TIPTeI} \overset{\text{(rapid)}}{\rightleftharpoons} \mathrm{TIPTe{*}I} + \mathrm{TIPTeI_2}^{\ominus}$$

Red 2,4,6-triisopropylphenyliodoselane is decomposed by tetraethylammonium iodide to give triiodide and the diselenide. Green 2,4,6-triisopropylphenyliodotellane is stabilized by iodide anions to give a red aryl diiodotellurate(II)-anion. The anion is thermally stable at room temperature, but labile with respect to iodide transfer to the educt iodotellane [34].

The bulkier 2,4,6-tri-tert-butylphenyliodotellan does <u>not</u> coordinate with iodide anions.

In summary, bulky substituents may deactivate Se-Se and Te-Te-bonds kinetically, but activate them thermodynamically. Bulky substituents suppress the intermolecular association of iodotellanes (tellurenyl iodides) by favouring low coordination numbers.

They favour the existence of iodoselanes at the expense of competing diselenide-diiodine charge transfer complexes. Thus basic solution chemistry of selenenyl and tellurenyl halides can now be revisited and further developped.

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