

REVIEW

Dedicated to Full Member of the Russian Academy of Sciences
B.A. Trofimov on the 65th Anniversary of His Birth

New Methods for Preparation of Organoselenium and Organotellurium Compounds from Elemental Chalcogens

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Abstract—The review summarizes the results of studies in the field of development of efficient procedures for the synthesis of dialkyl chalcogenides, dialkyl dichalcogenides, vinyl and ethynyl chalcogenides, and selenium-, tellurium-, and silicon-containing heterocyclic compounds on the basis of elemental selenium and tellurium.

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1. INTRODUCTION

Organoselenium and organotellurium compounds have long proved to be not only valuable intermediate products in organic synthesis and convenient models for studying fundamental problems of theoretical chemistry but also exceptionally important substances from the practical viewpoint [1–29]. Wide application of organoselenium compounds as synthons is determined by their unique properties: selenium-containing fragments can readily be introduced into an organic compound and, after appropriate transformations, selenium atom can be removed, e.g., by oxidation which leads to formation of a double bond via *syn*-elimination of selenium oxide [25]. Such properties of organoselenium derivatives have been utilized in a number of syntheses of complex natural compounds [8].

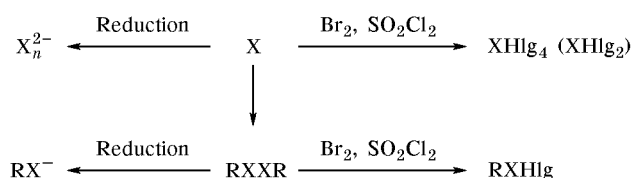
High biological activity of organoselenium compounds [13–15, 30–34] is one more reason for extensive development of organoselenium chemistry. The lack of selenium in human body was found to increase the risk of cardiovascular pathologies, cancer, arthritis, and a number of other diseases. One of the main factors responsible for aging of human body and

many pathologies is known to be peroxide oxidation of lipids [34]. The ability to inhibit peroxide oxidation of lipids in human body is maintained by the action of a selenium-containing enzyme, glutathione peroxidase, which catalyzes reactions of peroxy compounds with sulfanyl groups of glutathione [34].

A strong impetus to the development of the chemistry of selenium- and tellurium-containing heterocyclic compounds was given by the studies aimed at preparation of conducting materials on the basis of charge-transfer complexes and radical ion salts [35–40]. Manufacture of semiconducting materials, films, and coatings remains an important field of practical application of organoselenium and organotellurium compounds [41–45].

Elemental selenium and tellurium are very convenient starting materials for the synthesis of various organoselenium and organotellurium compounds. They are more advantageous than the other selenium- and tellurium-containing reagents: elemental selenium and tellurium are produced on a large scale, they are accessible, inexpensive, and readily transportable and can be stored for a long time without change in chemical properties. Elemental selenium and tellurium provide a source of nucleophilic (chalcogenide and

dichalcogenide ions) and electrophilic reagents (selenium halides) which may be generated *in situ* or prepared just before use. Organic dichalcogenides derived from elemental selenium and tellurium are in turn precursors of a variety of nucleophilic and electrophilic reagents (for applications of organic dichalcogenides in organic synthesis, see review [6]). Furthermore, elemental selenium and tellurium can be brought into insertion reaction with organometallic compounds to afford the corresponding metal selenolates and tellurolates which are then involved in nucleophilic substitution and addition reactions.

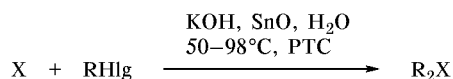


X = Se, Te; Hlg = Cl, Br; R is an organic radical; $n = 1, 2$.

The present review summarizes the results of studies performed at the Laboratory of Organochalcogen Compounds, Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, which were aimed at development of new methods for the preparation of organoselenium and organotellurium compounds from elemental selenium and tellurium. These studies constitute a logical extension of the previous works on the synthesis of organochalcogen compounds in basic media and systems under the guidance of Full Member of the Russian Academy of Sciences B.A. Trofimov [1–4].

2. REACTIONS OF CHALCOGENIDE AND DICHALCOGENIDE IONS

A new highly efficient method for the preparation of dialkyl selenides and tellurides from elemental selenium or tellurium and alkyl halides under conditions of phase-transfer catalysis was developed. The procedure ensures high purity of the products [46–49].



X = Se, Te; Hlg = Cl, Br, I; R = Me, Et, *i*-Pr, Pr, Bu, PhCH₂; PTC is phase-transfer catalyst, BzI₃Et₃NCl or Adogen 464.

The process includes generation of selenide or telluride ions from elemental chalcogens in the system KOH–SnO–H₂O; their subsequent alkylation leads

to the target products. The yield of dialkyl tellurides attains 98%, and the yield of dialkyl selenides is about 80% (calculated on the initial chalcogen). When the reaction was performed in the absence of phase-transfer catalyst, other conditions being equal, the yield of the product decreases by a factor of 2–3. The catalytic effect was found to increase with extension of the carbon chain in the alkyl halide; here, trialkyltelluronium and trialkylselenonium halides can act as phase-transfer catalysts. The procedure is simple from the experimental viewpoint, and there is no need of inert atmosphere. No extraction with organic solvents is required for isolation of the target products from the reaction mixture.

In the reaction of elemental selenium and tellurium with alkali in aqueous or organic medium, selenide and telluride ions are formed via disproportionation of the initial chalcogens; however, the amount of these ions is insufficient to ensure a high yield of dialkyl chalcogenides.



X = Se, Te.

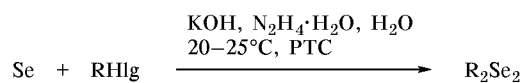
Addition of tin(II) chloride to the reaction mixture was found [50–71] to considerably increase the yield of organic chalcogenides. The possibility of using tin(II) chloride to reduce elemental chalcogens to chalcogenide ions in basic media was demonstrated for the first time in the reactions of selenium and tellurium with acetylene, which lead to formation of divinyl chalcogenides and (*Z*)-1-vinylchalcogeno-1-buten-3-ol as the major products [50–57]. Among a series of reducing agents examined in this respect, tin(II) chloride, hydrazine, and aluminum powder turned out to be the most effective [46–57]. The yield of divinyl chalcogenides strongly increases on addition of tin(II) chloride to the reaction mixture. For example, the yield of divinyl selenide increases from 44 to 80% [56], and that of divinyl telluride, from 2 to 94% [57], other conditions being equal.

Later [49], it was found that the reagent acting as reducing agent toward chalcogens [50–71] is tin(II) oxide which is formed (as precipitate) in the reaction of tin(II) chloride with alkali. Addition of that precipitate instead of tin(II) chloride to the reaction mixture in the synthesis of dialkyl [46–49] and divinylchalcogenides [50–57] exerts the same effect as that produced by tin(II) chloride. The use of the solution separated from the tin(II) oxide precipitate did not increase the yield of the target products.

The effect of tin(II) chloride was also observed in some other reactions of elemental selenium and tellurium, specifically in those leading to alkyl vinyl selenides [58], alkyl vinyl tellurides [59–61], (*Z*)-2-benzylidene-4-phenyl-1,3-diselenole [62, 63], (*Z,Z*)-distyryl telluride [64, 65], diallyl selenide [66], and other organoselenium and organotellurium compounds [67–71].

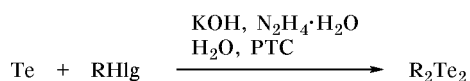
Organic diselenides and ditellurides are frequently used in the synthesis of other classes of organoselenium and organotellurium compounds. The reduction of diselenides and ditellurides gives organic selenolate and tellurolate ions which may be brought *in situ* into various nucleophilic reactions. Organic dichalcogenides react with halogens to give the corresponding haloseleno and halotelluro derivatives which are electrophilic reagents possessing a high synthetic potential. As follows from published data [7–29], until recently diaryl diselenides and ditellurides were mainly used in organic synthesis, whereas only a few examples were reported for analogous transformations of aliphatic diselenides and ditellurides.

We have synthesized dialkyl diselenides in up to 98% yield from elemental selenium and alkyl halides in the system KOH–N₂H₄·H₂O–H₂O in the presence of phase-transfer catalyst; the products were then used as starting materials in the synthesis of various organoselenium compounds [6, 49].



R = Me, Et, Pr, *i*-Pr, Bu, *t*-Bu, Bzl; Hlg = Br, I; PTC stands for BzIEt₃NCl or Adogen 464.

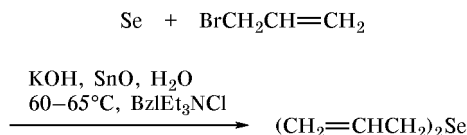
This procedure was used to synthesize di-*tert*-butyl diselenide, which made this compound accessible from commercially available initial reactants [49, 72]. Prior to our studies, there were no published data on the synthesis of di-*tert*-butyl diselenide by alkylation of diselenide ion. Elemental tellurium is not reduced to an appreciable extent under the conditions ensuring synthesis of dialkyl diselenides. However, it is converted mainly into ditelluride ion in the presence of potassium hydroxide when hydrazine hydrate is used as solvent. The reaction occurs at room temperature in the presence of phase-transfer catalyst. On the basis



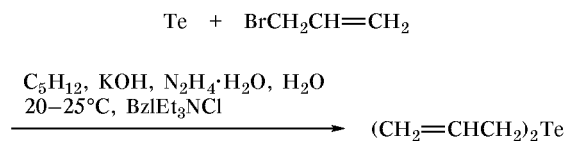
R = Me, Et, Pr, *i*-Pr, Bu, *t*-Bu, Bn; Hlg = Br, I; PTC stands for BzIEt₃NCl or Adogen 464.

of this reaction we have developed a procedure for the preparation of dialkyl ditellurides in up to 70% yield [6, 49]. Following the developed procedure, we succeeded in obtaining highly pure di-*tert*-butyl ditelluride and revealed for the first time [49, 73] that this product is a crystalline substance rather than liquid as reported previously.

An efficient and practical procedure was developed for the preparation of diallyl selenide in 80% yield from elemental selenium and allyl bromide [66]:

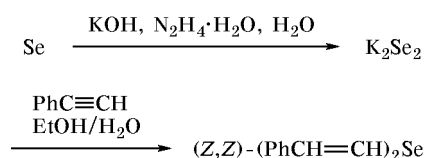


Our attempts to obtain diallyl telluride under similar conditions were unsuccessful. We proposed an efficient method for synthesizing diallyl telluride in 92% yield from elemental tellurium and allyl bromide via phase-transfer reaction using hydrazine hydrate as inorganic phase and pentane as organic [49].

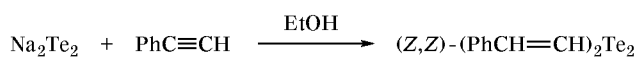


At present, ultrapure alkyl derivatives of selenium and tellurium compounds are widely used in growing and doping of epitaxial semiconducting structures and in the preparation of selenium- and tellurium-containing films and coatings [41–45]. Diallyl selenide and diallyl telluride are promising reagents for microelectronics. According to Korenstein *et al.* [74], the use of diallyl telluride as a source of tellurium makes it possible to considerably reduce the temperature of gas-phase decomposition while growing crystalline CdTe and HgTe films (from 400°C for diethyl telluride to 180°C for diallyl telluride) and thus to obtain high-quality semiconductors [74].

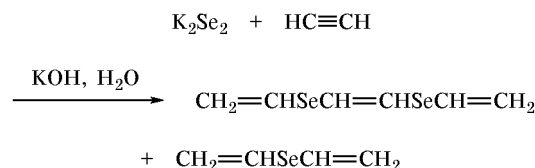
Addition of sodium selenide to acetylenes gives the corresponding divinyl selenides in high yield [50–57]. Prior to our studies, there were no published data on reactions of diselenide and ditelluride ions with acetylenes. We were the first to effect and study nucleophilic addition of diselenide and ditelluride ions to acetylene and phenylacetylene [75]. The reaction of potassium diselenide with phenylacetylene in aqueous ethanol in the presence of KOH at 105–115°C gave (*Z,Z*)-distyryl selenide in 80% yield.



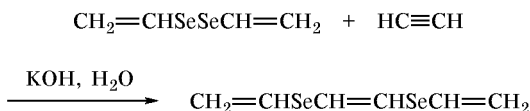
It was found that (Z,Z)-distyryl diselenide is formed as intermediate product which can be isolated when the reaction is performed at room temperature. The reaction of sodium ditelluride with phenylacetylene underlies an efficient procedure for the synthesis of (Z,Z)-distyryl ditelluride in 82% yield. The process occurs in ethanol without heating and is regio- and stereoselective.



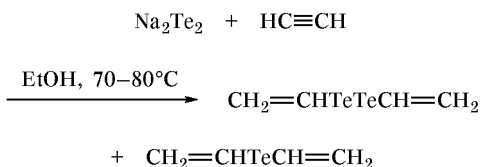
Potassium diselenide reacts with acetylene in aqueous alkali at 90–100°C to afford 12% of previously unknown (Z)-1,2-bis(vinylseleno)ethene and 62% of divinyl selenide.



Potassium diselenide was obtained *in situ* from elemental selenium, hydrazine hydrate, and KOH in aqueous medium. A plausible mechanism of the above reaction includes addition of diselenide ion to acetylene to give divinyl diselenide which either loses selenium atom with formation of the corresponding selenide or adds to acetylene (by analogy to the known reaction of dialkyl diselenides with acetylene), yielding (Z)-1,2-bis(vinylseleno)ethene.



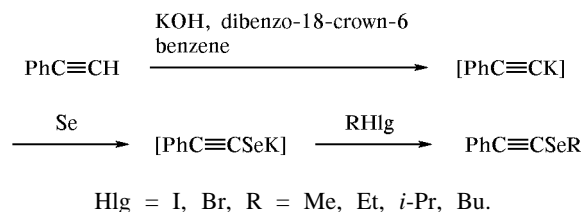
Unlike unstable divinyl diselenide, divinyl ditelluride is formed in 24% yield by reaction of sodium ditelluride with acetylene.



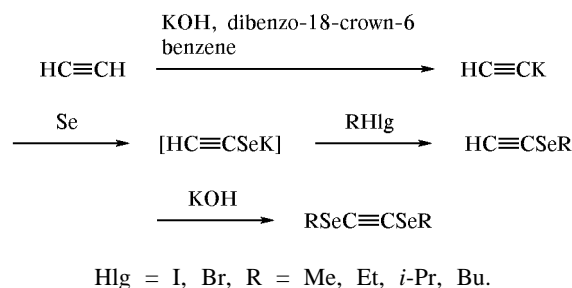
Presumably, the formation of divinyl telluride (yield 48%) is the result of elimination of tellurium from the initially formed divinyl ditelluride.

3. INSERTION OF SELENIUM AND TELLURIUM IN REACTIONS WITH ACETYLIDES

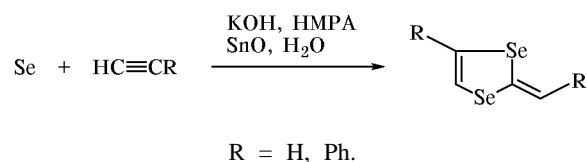
We have developed methods for generation of acetylide and phenylacetylide anions which react with elemental selenium to give ethyneselenolate and phenylethyneselenolate ions [69–71, 76–80]. Alkylation of the latter leads to the corresponding acetylenic selenides. The reactions are carried out under conditions of phase-transfer catalysis or in the systems KOH–HMPA, KOH–DMSO, and KOH–DMF. The possibility of using elemental selenium as solid phase in phase-transfer reactions was demonstrated for the first time [71, 76–80].



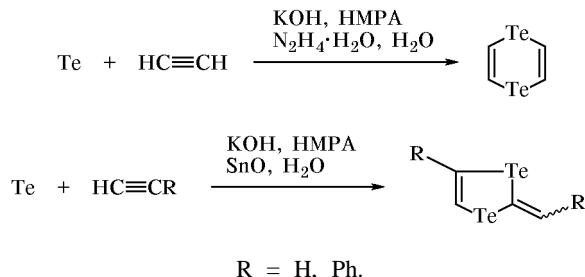
The reaction with acetylene results in formation of alkyl ethynyl selenides and bis(alkylseleno)acetylenes in an overall yield of about 50%. Bis(alkylseleno)acetylenes were found to originate from alkyl ethynyl selenides by the action of a base.



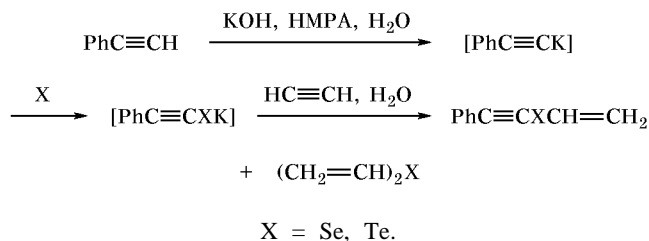
Ethyneselenolate and phenylethyneselenolate ions in the presence of water undergo dimerization to give 2-methylene-1,3-diselenole (yield 4%) [75] and (Z)-2-benzylidene-4-phenyl-1,3-diselenole (70%) [69].



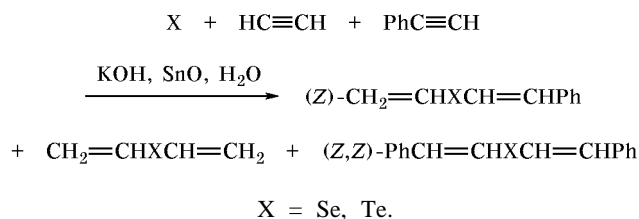
Likewise, elemental tellurium reacts with phenylacetylene, yielding 30% of (*Z*)-2-benzylidene-4-phenyl-1,3-ditellurole [69, 70]. Depending on the reducing agent, the reaction of elemental tellurium with acetylene gives either 2-methylene-1,3-ditellurole or 1,4-ditellurine [70].



Taking into account different CH acidities of acetylene and phenylacetylene, conditions can be found under which phenylacetylene is converted into phenylacetylidene ion which then reacts with elemental chalcogens, while acetylene is involved in nucleophilic addition. The reaction of elemental selenium with acetylene and phenylacetylene in the system KOH–HMPA–H₂O at 110–120°C affords 42% of phenylethynyl vinyl selenide. Under analogous conditions, elemental tellurium gives rise to 12% of phenylethynyl vinyl telluride [81].



(*Z*)-Styryl vinyl selenide (yield 63%) and (*Z*)-styryl vinyl telluride (44%) can be synthesized in one step from the corresponding elemental chalcogen, acetylene, and phenylacetylene [81].

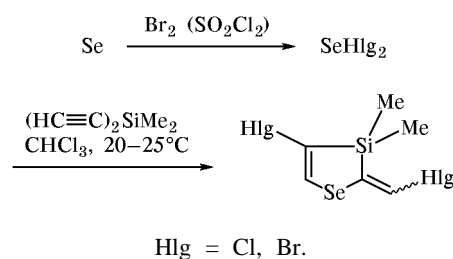


Thus, depending on the conditions, either phenylethynyl or (*Z*)-styryl vinyl chalcogenides can be obtained from the same initial reactants.

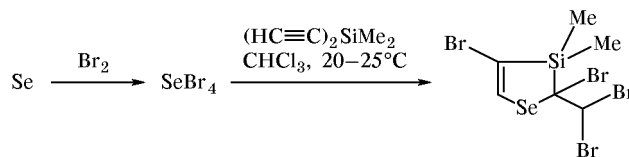
4. REACTIONS OF SELENIUM HALIDES

The addition of selenium(IV) halides and Se₂Cl₂ to acetylenes was reported in [82–84]. Electrophilic addition of organic selenenyl halides to acetylene is the main method of synthesis of 2-halovinyl selenides [85–87]. There are no published data on the addition of selenium(II) halides to acetylenes.

We have found that selenium dichloride and dibromide generated *in situ* from elemental selenium and halogenating agents (such as sulfuryl chloride or bromine) can be used in the synthesis of new heterocyclic systems. Selenium dichloride and dibromide add to bis(ethynyl)dimethylsilane to give previously unknown heterocyclic compounds, 3,6-dihalo-4,4-dimethyl-1,4-selenasilafulvenes, in 70–72% yield [88].



We found no published data on the addition of selenium(IV) halides to bis(ethynyl)silanes. The reaction of selenium tetrabromide with bis(ethynyl)dimethylsilane in chloroform at room temperature afforded previously unknown 2,4-dibromo-2-dibromo-3,3-dimethyl-1-selena-3-sila-4-cyclopentene (yield 60%) [89].



Thus elemental selenium and tellurium are convenient starting materials for synthesis of various organoselenium and organotellurium compounds. Effective procedures for the preparation of dialkyl chalcogenides, dialkyldichalcogenides, vinyl and ethynyl chalcogenides, and heterocyclic compounds containing selenium, tellurium, and silicon have been developed on the basis of elemental chalcogens.

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