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# Divinyl Sulfide and its Selenium and Tellurium Analogs as Starting Materials for the Preparation of Polyfunctional Alkyl, Aromatic, and Heteroaromatic Vinyl Chalcogenides

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ABSTRACT: *Divinyl sulfide and its selenium and tellurium analogs have been used as starting materials for the introduction of vinylchalcogeno groups by reaction of halogenoalkanes, epichlorohydrin, polybromoorganyl sulfides or sulfoxides, perfluoro(chloro)benzenes, and dihalogenopyridines with sodium vinylchalcogenate. This approach has enabled the preparation of polyfunctional vinylsulfanyl derivatives of perfluoro(chloro)benzenes, which are applied for the synthesis of N-, O-, and S-containing heterocyclic systems. The structures of the new compounds have been confirmed by IR and $^{1}H$, $^{13}C$, $^{19}F$ NMR spectroscopy.* © 2006 Wiley Periodicals, Inc. Heteroatom Chem 17:491–498, 2006; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20270

## *INTRODUCTION*

Vinyl sulfides are versatile compounds for the synthesis of various substances and preparation of products or materials with targeted properties [1].

Investigations carried out at the boundary of acetylene and organosulfur compounds chemistry have uncovered a series of new reactions in superbase media leading to the formation of divinylsulfide [2]. The reactions of acetylene with such sulfur compounds as hydrogen sulfide, sulfides of alkaline metals, di- and polysulfides of alkaline metals, elemental sulfur, carbon disulfide, and various thione systems have been studied. As a result, a wide range of efficient and convenient methods for the preparation of divinylsulfide [2–4]—a promising cross-linking agent [5], bifunctional monomer, and perspective vinylthio group synthon for fine organic synthesis—has been developed.

This circumstance provided the basis for systematic investigations of the vinylthiilation reactions of halogen-containing electrophilic reagents, including polyhaloaromatic and heteroaromatic systems, which can be synthesized only by this method.

The C—S bond cleavage in divinylsulfide and 1-alkenyl sulfides under the action of alkali metals has been reported [6,7].

The conditions of generation of etheneselenolate anions [8–10] from divinylselenide and ethenetellurolate anions [8] from divinyltelluride have been found. The methods for the high-yield preparation of divinylselenide and divinyltelluride have been devised [4,11,12].

The aim of the present review is to show the wide possibilities of divinylsulfide and its selenium or tellurium analog as starting compounds for the synthesis of new unsaturated or highly unsaturated sulfur-, selenium-, or tellurium-containing substances including polyfunctional heteroaromatic,

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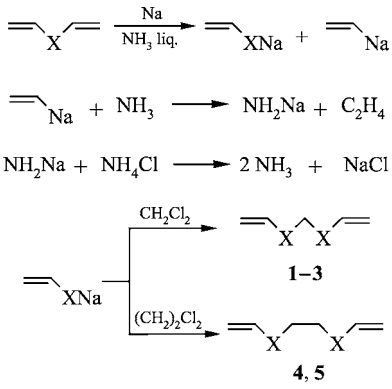
perfluoroaromatic compounds and for the preparation of various polyfunctional N-, O-, and S-containing compounds and heterocyclic systems.

## *SYNTHESIS OF DIVINYLCHALCOGENOALKANES*

The nucleophilic substitution of chlorine in dichloromethane and 1,2-dichloroethane by sodium ethenechalcogenolates in dimethylformamide (DMF)

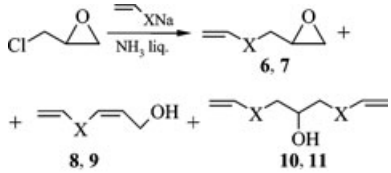



X = S (**1**, 53%), Se (**2**, 50%), Te (**3**, 40%)
X = S (**4**, 80%), Se (**5**, 58%) (1)

at 10–20°C afforded divinylchalcogenoalkanes **1–5** in 40–80% yield depending upon the nature of the chalcogene. A necessary condition for successful reactions involved the decomposition of sodium amide, formed from vinyl sodium and liquid ammonia, by ammonium chloride [13] (Eq. 1).

## *VINYLCHALCOGENATION OF EPICHLOROHYDRIN*

The interaction of sodium ethenechalcogenolates with epichlorohydrin in liquid ammonia was studied [14,15]. When the reagents were taken in equimolar ratio in the reaction between sodium ethenechalcogenolates and epichlorohydrin in liquid ammonia, 3-[vinylsulfanyl (seleno)]-1,2-epoxypropanes **6, 7** were obtained in 60 and 62% yield, respectively [14,15]. (*E*)-4-Sulfanyl(seleno)-2,5-hexadiene-1-ols **8, 9** and 3,7-disulfanyl(seleno)-1,8-nonadiene-5-ols **10, 11** were obtained as side products in 10 and 20% yields, respectively. In the case of using a two- to threefold excess of sodium ethenechalcogenolates, the yield of compounds **10** and **11** increased to 36 and 26%, respectively. In this case, the reaction of oxyrane cycle opening in compounds **6, 7** became dominant. An essential condition for this reaction (as well as for those described above) is the neutralization of the side product sodium amide by ammonium chloride. In the presence of sodium amide and with equimolar ratio of the reagents, the reaction involved primarily the ring opening with formation of alcohols **8, 9** (in 71 and 23% yield, respectively), whereas the yield of vinylchalcogenoepoxypropanes **6, 7** decreased up to 13 and 5%. The routes of the alcohols **8**, **9** formation have been discussed [15] (Eq. 2).

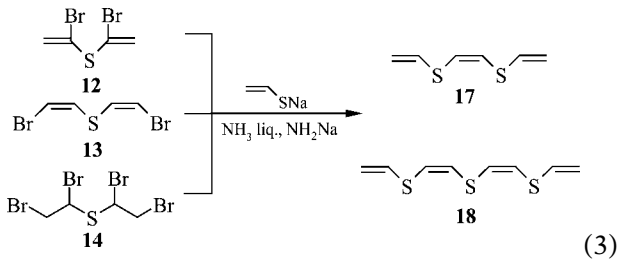



X = S (**6**, **8**, **10**), Se (**7**, **9**, **11**) (2)

## *STEREOSELECTIVE SYNTHESIS OF HIGHLY UNSATURATED ORGANOCHALCOGENIC COMPOUNDS ON THE BASIS OF HALOGEN-SUBSTITUTED DIVINYLSULFIDES AND SULFOXIDES*

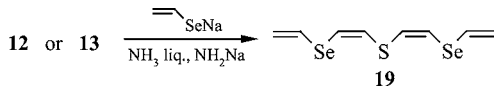
A simple and efficient method for stereoselective synthesis of highly unsaturated compounds has been developed. It involved nucleophilic substitution of bromine in bromine-substituted sulfides **12–14** and sulfoxides **15, 16** [16–22]. We have also worked out a method for the synthesis of sulfides **12, 13** and sulfoxide **15** based on divinylsulfide [17]. The reaction of sodium ethenethiolate with di(1-bromovinyl)- **12** and di(2-bromovinyl)sulfides **13** (the latter as a ~2:1 mixture of *Z*,*Z*- and *Z*,*E*-isomers) in liquid ammonia led to the formation of *Z*-1,2-di(vinylsulfanyl)ethene **17** in 65% yield [16,18].

The interaction between sulfide **13** and sodium ethenethiolate in liquid ammonia (ratio of the reagents ~1:3) preceded by the decomposition of sodium amide with ammonium chloride resulted in a 65% yield of *Z*,*Z*-di[2-(vinylsulfanyl)vinyl]sulfide **18** [19]. It was shown that compounds **17** and **18** (obtained in 20 and 30% yield, respectively) were formed by reaction of sulfide **14** with sodium ethenethiolate in liquid ammonia and subsequent dehydrobromination by sodium amide [20] (Eq. 3).



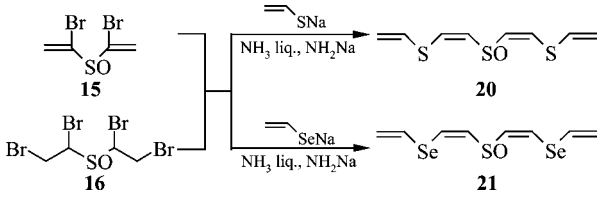

(3)

The reactions of sodium etheneselenolate with the sulfides **12** and **13** under similar conditions afforded *Z*,*Z*-di[2-(vinylseleno)vinyl]sulfide **19** in 40–60% yield [21] (Eq. 4).




(4)

The reaction of sulfoxides **15, 16** with sodium ethenethiolate and sodium amide in liquid ammonia (molar ratio of the reagents is 1:2 or 1:3) resulted in 40 and 35% yields, respectively, of *Z,Z*-di[2-(vinylsulfanyl)vinyl]sulfoxide **20** [22] (Eq. 5).



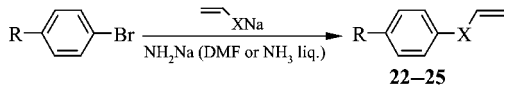

(5)

The interaction of sulfoxide **15** with sodium etheneselenolate (the molar ratio of the reagents is 1:2) led to the formation of *Z,Z*-[2-(vinylseleno)vinyl]-sulfoxide **21** and diethyl diselenide in low yields.

The data obtained suggested that the reactions of bromine-substituted sulfides **12–14** and sulfoxides **15, 16** with sodium ethenechalcogenolates in liquid ammonia in the presence of sodium amide involved the formation of acetylenic intermediate according to the E-$Ad_N$ mechanism.

## *SYNTHESIS OF VINYLARYLCHALCOGENIDES AND VINYLSULFANYLFLUORO(CHLORO)BENZENES*

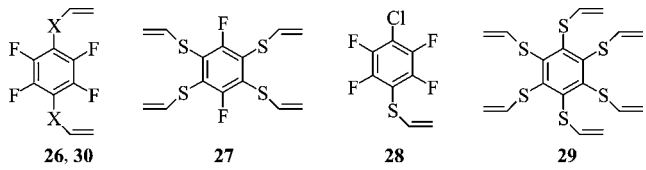
The interaction between sodium ethenechalcogenolates and bromobenzene and *para*-bromonitrobenzene in liquid ammonia or DMF resulted in vinylaryl chalcogenides **22–25** [13] (Eq. 6).




R = H, X = S (**22**, 53%)
R = $NO_2$, X = S (**23**, 88%), Se (**24**, 70%), Te (**25**, 30%)

(6)

The directed synthesis of 3,6-di(vinylsulfanyl)-1,2,4,5-tetrafluorobenzene **26**, 2,3,5,6-tetra (vinylsulfanyl)-1,4-difluorobenzene **27**, and 3-(vinylsulfanyl)-1,2,4,5-tetrafluoro-6-chlorobenzene **28** in 80, 90, and 65% yields involved the reaction of sodium ethenethiolate with hexafluorobenzene and chloropentafluorobenzene in DMF at 20–30°C [23,24]. The reaction conditions were varied depending on the ratio of the reagents and the order of their mixing. Hexa(vinylsulfanyl)benzene **29** was prepared in the mixture of DMF and liquid ammonia at −5 to 0°C (yield 26%). When hydroquinone was added to the reaction mixture, the yield of compound




X = S (**26**), Se (**30**)

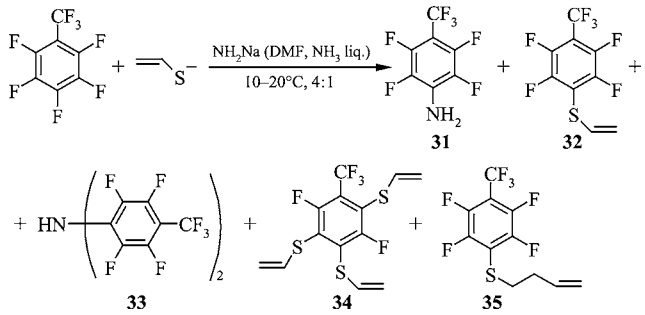
SCHEME 1

**29** increased up to 57% as a result of inhibition of hexa(vinylsulfanyl)benzene polymerization [25].

Unlike vinylthiolate anion but yet under analogous conditions, we failed to synthesize the substitution products of four or six fluorine atoms for etheneselenolate anion. 3,6-Di(vinylseleno)-1,2,4,5-tetrafluorobenzene **30** was prepared in 60% yield under large excess of hexafluorobenzene. The same picture was observed for the sulfur analog [23] (Scheme 1).

Investigation of the vinylthiilation of octafluorotoluene lead us to conclude that the presence of the electron-withdrawing substituent $CF_3$ in perfluorobenzene had a dramatic effect on the reaction course as well as on the structure of the compounds formed [26,27].

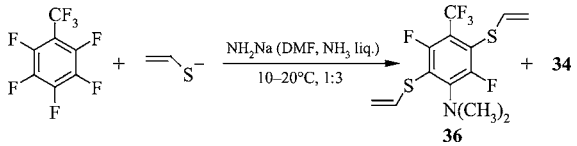
The reaction of octafluorotoluene (used in a fourfold excess) with ethenethiolate anions in the mixture of DMF and liquid ammonia (the latter was not removed completely from the reaction mixture when it was replaced by DMF) gave a mixture of five products **31–35**. According to the data of GC–MS and NMR spectroscopy, the reaction mixture contained 68.7% of 1-amino-4-trifluoromethyl-2,3,5,6-tetrafluorobenzene **31**, 17.3% of 1-(vinylsulfanyl)-4-trifluoromethyl-2,3,5,6-tetrafluorobenzene **32**, 10.3% of 1,1′-di(4-trifluoromethyl-2,3,5,6-tetrafluorophenyl)amine **33**, 1.6% of 1,2,5-tri(vinylsulfanyl)-4-trifluoromethyl-3,6-difluorobenzene **34**, and 1.5% of 1-(but-3-enylsulfanyl)-4-trifluoromethyl-2,3,5,6-tetrafluorobenzene **35** (Eq. 7). Compound **35** is formed by the substitution of fluorine with small amounts of but-3-enylthiolate anion [27], which is a by-product of the reaction of divinyl sulfide with sodium.




(7)

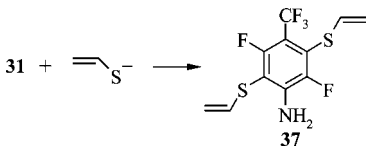
It follows from the data obtained that the basic reaction of octafluorotoluene (unlike hexafluorobenzene) under these conditions is the amination reaction, i.e., the substitution of fluorine atom by amino group in *para*-position of benzene ring under action of $NaNH_2$.

When the ratio of reagents octafluorotoluene: sodium ethenethiolate was 1:3 and the order of their mixing was changed, the formation of 2,5-di(vinylsulfanyl)-1-dimethylamino-4-trifluoromethyl-3,6-difluorobenzene **36** (70% yield) in DMF and in the presence of small amount of ammonia, of which attempts to remove it were unsuccessful, was unexpected. This compound was formed due to nucleophilic substitution of two fluorine atoms by ethenethiolate anions and one *para*-positioned fluorine atom by dimethylamine. The latter may result from reaction of DMF with sodium amide or liquid ammonia [26] (Eq. 8).




(8)

The vinylthiilation of compound **31** in DMF at 15–20°C (ethenethiolate anion was taken in threefold excess) resulted in the formation of 1-amino-2,5-di(vinylsulfanyl)-4-trifluoromethyl-3,6-difluorobenzene **37** (Eq. 9).



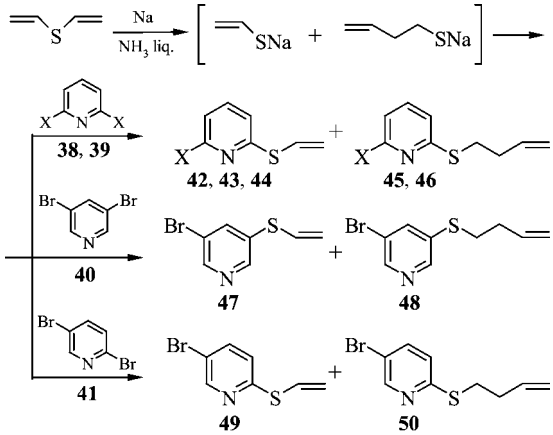

(9)

The experimental data suggest that in the reaction of octafluorotoluene with ethenethiolate anions in the mixture of DMF and liquid ammonia, these solvents can participate in some way as reagents. Thus, they can compete strongly with ethenethiolate anions in the nucleophilic substitution enabling the possibility to synthesize new polyfunctional vinylsulfanylfluorobenzenes containing electron-donating amino- and dimethylamino groups along with electron-withdrawing groups.

## *SYNTHESIS OF VINYLSULFANYL(HALOGENO)PYRIDINES*

The application of the vinylsulfanyl group was extended by the reaction of sodium vinylthiolate with halogeno compounds of pyridine. The obtained derivatives can be used in (co)polymerizations or addition reactions.

A series of vinylsulfanyl(halogeno)pyridines **42–44, 47**, and **49** (obtained in 58, 52, 55, 33, and 62% yields, respectively) has been prepared by the reaction of halogen-containing pyridines **38–41** with sodium ethenethiolate generated by reaction of divinylsulfide with sodium in liquid ammonia [28,29] (Eq. 10).



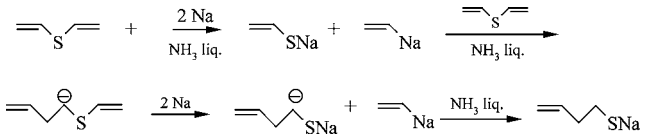

X = Cl (**38**, **42**, **45**); X = Br (**39**, **43**, **46**); X = SCH=$CH_2$ (**44**)

(10)

The reaction of divinylsulfide with sodium in liquid ammonia leading to ethenethiolate anion was accompanied by the unexpected formation of but-3-enylthiolate anion leading to but-3-enylsulfanyl derivatives of pyridines **45, 46, 48**, and **50**. The yields of these by-products were 10–28% depending on the reaction conditions [28–30]. But-3-enylthiolate anion was involved in nucleophilic substitutions with different electrophilic reagents, including halogenoalkanes and halogenobenzenes.

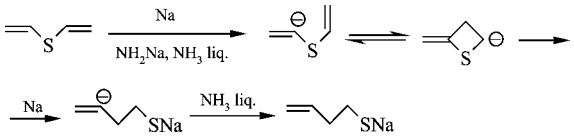
The generation of but-3-enylthiolate anion (Eqs. 11–13) has been discussed in previous works [28–30].

The first possible way of formation of 3-butenylthio derivatives involves the addition of vinyl sodium to divinyl sulfide followed by the cleavage of the C—S bond of the butenyl vinyl sulfide carbanion by sodium through the formation of sodium butenylthiolate carbanion. The second product of the cleavage is vinyl sodium, which again participates in the reaction of nucleophilic addition to divinyl sulfide (Eq. 11).



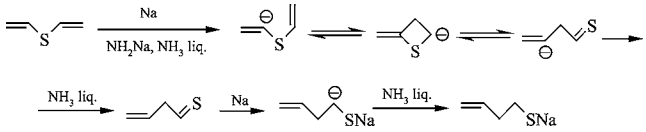

(11)

The second possible way involves the alpha-deprotonation of divinyl sulfide followed by the intramolecular cyclization and the cleavage of the formed cyclic carbanion by sodium (Eq. 12).



(12)

The third possible way involves the alpha-deprotonation of divinyl sulfide followed by the rearrangement of the formed carbanion via unsaturated thioaldehyde (Eq. 13).

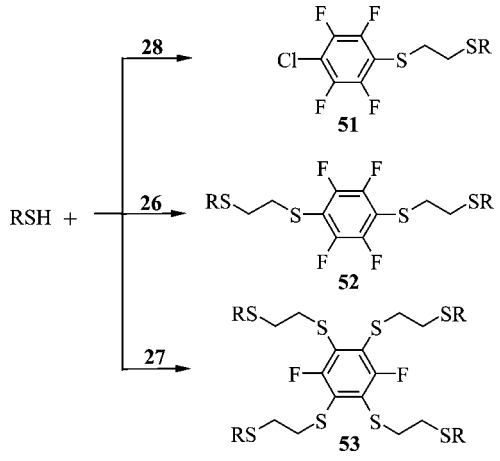


(13)

## *REACTIVITY OF VINYLSULFANYLFLUORO-(CHLORO)BENZENES*

The vinylthio groups in vinylsulfanylfluoro(chloro)benzenes **26–28** can actively participate in radical and electrophilic additions, while halogens can be displaced under nucleophilic conditions. Because of their high synthetic potential, compounds **26–28** can be used as building blocks in syntheses of various N-, O-, and S-containing heterocycles in substitutions with bi-nucleophilic reagents.

Initiation with azodiisobutyronitrile in the reaction of compounds **26–28** with propanethiol [31] resulted in high yields of the addition products **51–53** (Eq. 14).

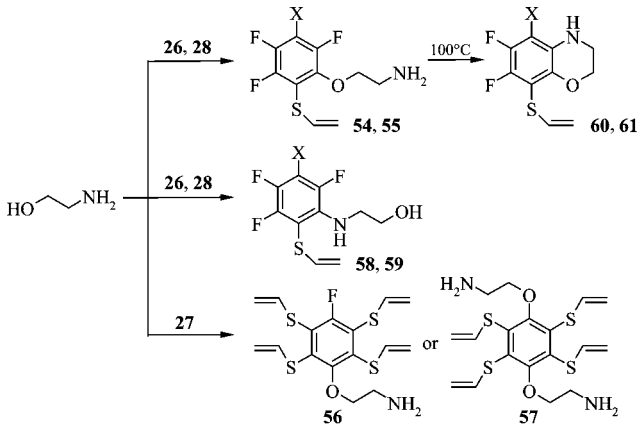


(14)

At 60°C, two vinylthio groups were involved in the addition of propanethiol to tetravinylsulfanylfluorobenzene **27**, whereas in the slower reaction of thiol with **26** under similar conditions, only one vinylthio group reacted. Vinylsulfanylfluorobenzenes **26** and **27** were reactive in radical and cation (co)polymerizations [32].

We have shown the capability of fluorine atoms to undergo nucleophilic substitution by the reaction of compounds **26–28** with 2-aminoethanol [31,33,34]. Depending upon the reaction conditions, either the OH group in aminoethanol (DMF, NaOH, 20–35°C) reacted to give the compounds **54–57** or its amino group (DMF, 100°C) the products **58, 59**.

In the case of tetravinylsulfanylfluorobenzene **27** (DMF, 50°C, NaOH), the substitution of two fluorine atoms occurred. The yield of 1,4-di(2-aminoethoxy)-2,3,5,6-tetra(vinylsulfanyl)benzene **57** was 35% (Eq. 15).

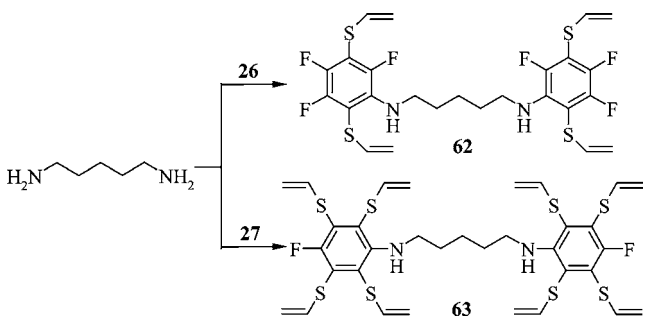


X = SCH=CH$_2$ (**54**, **58**, **60**); X = Cl (**55**, **59**, **61**)

(15)

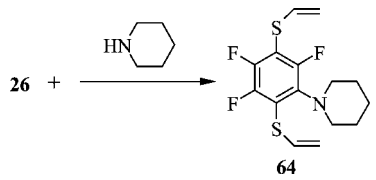
Heating the compounds **54, 55** in DMF at 100°C led to the cyclic products **60** and **61**.

The reactions of compounds **26** and **27** with 1,5-pentanediamine (DMF, 110–120°C) proceeded selectively to give the compounds with bridge structure **62** and **63** with low yields [35,36] (Eq. 16).



(16)

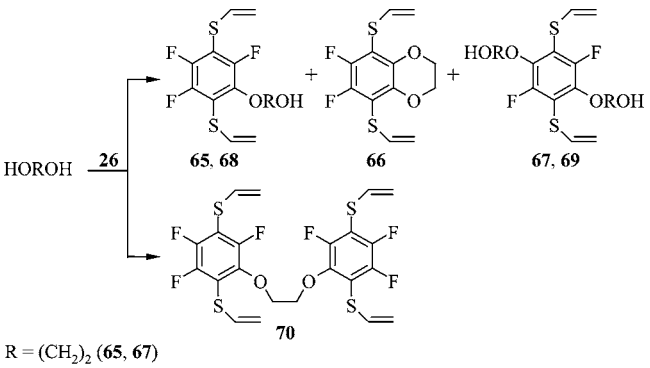
The interaction of vinylsulfanylfluorobenzene **26** with cyclic amines, for example, with piperidine (100°C, DMF, molar ratio of the reagents 1:4) only resulted in the monosubstitution product 3,6-di(vinylsulfanyl)-2-piperidino-1,4,5-trifluorobenzene **64** in 40% yield [37] (Eq. 17).



(17)

The various reactions of vinylsulfanylfluorobenzene **26** with glycols in DMF in the presence of NaOH resulted in mixtures of mono- **65, 68**, disubstitution products **67, 69** and products of their *ortho*-cyclization **66**. The yield of these products depend on the reaction time and temperatures as well as on the reagents ratio [34,35].

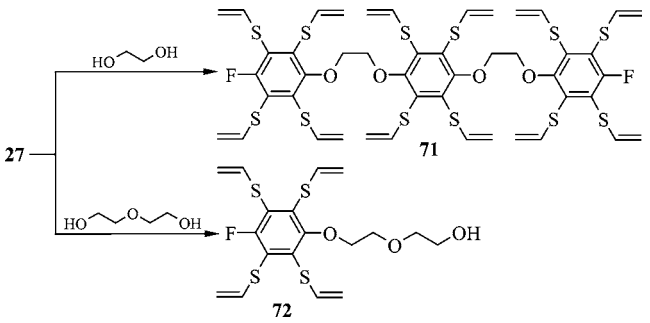
When compound **26** was taken in twofold excess with respect to ethylene glycol, the reaction led to the formation of 1,2-di[3,6-di(vinylsulfanyl)-2,4, 5-trifluorophenoxy]ethane **70** along with the monosubstitution product **65** and the product of its *ortho*-cyclization **66** in low yields. The interaction of compound **26** with diethylene glycol in the presence of NaOH at 50°C gave rise primarily to the product of disubstitution **69** (Eq. 18).



R = $(CH_2)_2$ (**65**, **67**)
R = $(CH_2CH_2)_2O$ (**68**, **69**)

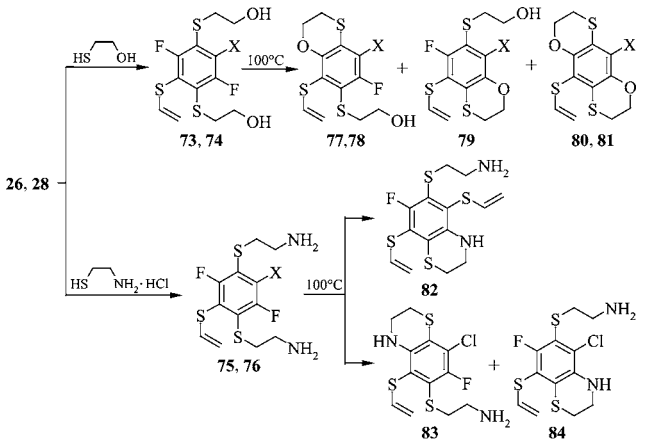
(18)

The reaction of tetravinylsulfanylfluorobenzene **27** with ethylene glycol (DMF, NaOH, 50–60°C) resulted in compound **71** with a bridge structure, and the reaction with diethylene glycol under similar conditions led to the monosubstitution product **72**. In both reactions, the maximal yields were 20% [36] (Eq. 19).



(19)

The reactions of vinylsulfanylfluoro(chloro)-benzenes **26** and **28** with 2-mercaptoethanol (DMF, NaOH, 20°C) and 2-aminoethanethiol hydrochloride at

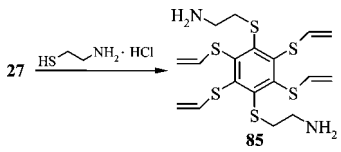


X = $SCH{=}CH_2$ (**73**, **75**, **77**, **80**); X = Cl (**74**, **76**, **78**, **79**, **81**)

(20)

40–45°C in a mixture of *iso*-$C_3H_7OH$, $H_2O$, and DMF in the presence of KOH proceeded regioselectively and involved the substitution of two fluorine atoms (located in *para*-position with respect to each other) with the participation of the SH-groups. Yields of the products of disubstitution **73–76** were within the range of 40–60%. Heating the compounds **73–76** up to 100°C led to their cyclization resulting in the formation of heterocyclic compounds **77–84** [34,38,39] (Eq. 20).

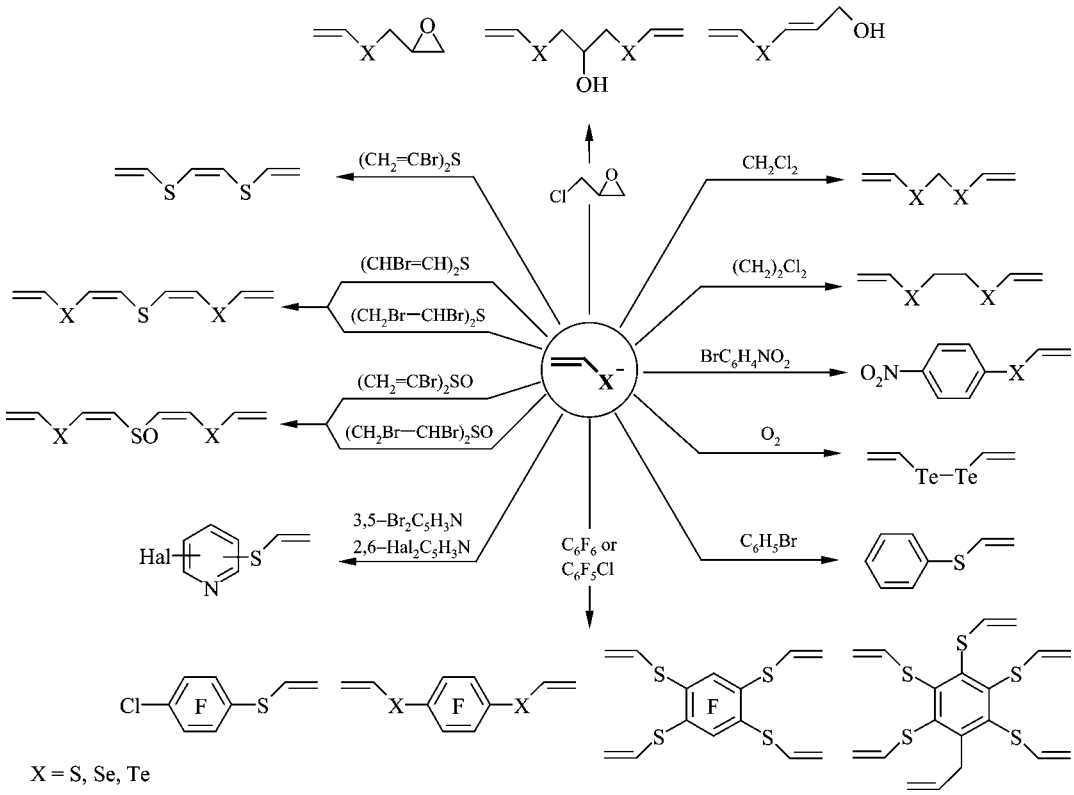
The reaction of compound **27** with 2-aminoethanethiol under analogous conditions gave rise to the formation of disubstitution product **85** [36] (Eq. 21).



(21)

All new compounds have been characterized, and their structures were confirmed by IR and $^1H$, $^{13}C$, $^{19}F$ NMR spectroscopy.

Thus, high efficiency and wide synthetic possibilities of new general approach to one-pot synthesis of various mono-, di-, and polyvinylchalcogenides based on the reaction of vinylchalcogenation of polyhalogeno-substituted electrophilic reagents containing different functional groups have been shown (Scheme 2).




**SCHEME 2**

## *CONCLUDING REMARKS*

This review shows the synthetic versatility of the various di- and polyvinyl chalcogenides generated by vinylchalcogenation of electrophiles containing two or more halogen atoms and different functional groups.

The approach proposed is valuable because it is based on one-step reaction and uses inexpensive and ready available starting materials.

Vinylthiilation reactions proceeded under mild conditions. The reactions of ethenechalcogenolate anions with per(fluoro)benzenes were regioselective, and the vinylchalcogenation reactions with bromine-substituted divinylsulfides and sulfoxides were stereoselective. Vinylsulfanyl derivatives of per(fluoro)benzenes, which are the basic building blocks for the design of polyfunctional heterocyclic compounds, present special interest.

This approach can be applied for the synthesis of different series of unsaturated chalcogen-containing compounds.

The new possibilities of vinylsulfonyl derivatives of the obtained compounds for the synthesis of functionalized heterocyclic compounds are illustrated in a next review with several reactions of divinylsulfonylfluorobenzene [40].

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