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Reactions of N-Sulfonyldiarylchalcogenimides with Diaryl Ditellurides

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ABSTRACT

Unlike the N-sulfonylsulfimide **1a**, *which does not react with diaryl ditellurides* **2** *even at 180°C, the selenimide* **1b**, *and tellurimides* **1c,d** *convert quantitatively compounds* **2** *to N,N-bis-(N′-p-tosylimidoarenetellurinyl)-p-tosylamides* **4a,b** *in refluxing benzene. The reactivity of N-sulfonylchalcogenimides* **1** *increases in the order S < Se < Te. Diaryl telluroxides* **6** *readily oxidize ditellurides* **2** *to arenetellurinic anhydrides* **5**. *The mechanisms of these reactions are discussed.*

INTRODUCTION

N-Sulfonyldiarylchalcogenimides **1** contain a formal double chalcogen–nitrogen bond which has a dipolar (ylide) character, and its polarity increases in the order S < Se < Te [1–3]. In the last few years, we have studied the effect of the nature of the chalcogen atom in chalcogenimides **1** on their oxidizability in reactions with thiols [4,5], thioamides [4,5], hydroquinone, and its derivatives [6], and we found that this characteristic increases in the same order.

$$Ar_2X{=}NSO_2R \leftrightarrow Ar_2\overset{+}{X}{-}\overset{-}{N}SO_2R$$

1

X = S, Se, Te

We have recently reported [7] that *N*-arenesulfonyl-Te,Te-diaryltellurimides may be reduced quantitatively to diaryl tellurides with diphenyl ditelluride at room temperature in chloroform in a 1:3 ratio. Here, we report the effect of the nature of the chalcogen atom in *N*-*p*-tosyldiarylchalcogenimides **1** on their ability to react with diaryl ditellurides, and we discuss a possible mechanism of this reaction.

RESULTS AND DISCUSSION

Three equivalents of *N*-*p*-tosyldiarylchalcogenimide **1** and one equivalent of diaryl ditelluride **2** were heated in a dry solvent (toluene, *o*-dichlorobenzene, and benzene) until the solution became colorless. The results are presented in Table 1. As may be seen from this table, unlike *N*-*p*-tosyl-*S*,*S*-diphenylsulfimide (**1a**), which does not react with diaryl ditellurides **2a,b** when refluxed during 48 hours in toluene or when heated at 180°C in *o*-dichlorobenzene for 1.5 hours, *N*-*p*-tosyl-Se,Se-diphenylselenimide (**1b**) and *N*-*p*-tosyltellurimides **1c,d** react easily with compounds **2a,b** under rather mild conditions and oxidize these to nitrogen analogs of arenetellurinic anhydrides, *N*,*N*-bis-(*N*′-*p*-tosylimidoarenetellurinyl)-*p*-tosylamides **4a,b**. As

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TABLE 1 Reactions of *N-p*-Tosylchalcogenimides **1** with Diarylditellurides **2**

Starting Materials		*Reaction Conditions*^{a}	*Reaction Time (Hours)*	*Products (Yield in %)*			
1a	**2a**	A	48.00	—	—	—	—
		B	2.00	—	—	—	—
	2b	A	48.00	—	—	—	—
		B	2.00	—	—	—	—
1b	**2a**	A	0.75	**3a**^{b}	(98)	**4a**	(98)
		C	1.50	**3a**	(96)	**4a**	(95)
		D	2.00	**3a**	(97)	**4a**	(97)
	2b	A	0.65	**3a**	(99)	**4b**	(98)
		C	1.25	**3a**	(96)	**4b**	(96)
		D	1.75	**3a**	(97)	**4b**	(96)
1c	**2a**	A	0.08	**3b**^{c}	(97)	**4a**	(98)
		C	0.20	**3b**	(96)	**4a**	(95)
		D	0.40	**3b**	(97)	**4a**	(96)
	2b	A	0.05	**3b**	(97)	**4b**	(98)
		C	0.08	**3b**	(95)	**4b**	(96)
		D	0.30	**3b**	(96)	**4b**	(97)
1d	**2a**	A	0.08	**3c**^{d}	(97)	**4a**	(98)
		C	0.20	**3c**	(95)	**4a**	(95)
		D	0.40	**3c**	(98)	**4a**	(97)
	2b	A	0.05	**3c**	(98)	**4b**	(97)
		C	0.08	**3c**	(94)	**4b**	(94)
		D	0.30	**3c**	(97)	**4b**	(96)

[a] A, in boiling toluene; B, in *o*-dichlorobenzene at 180°C; C, in *o*-chlorobenzene at 110°C; D, in boiling benzene.

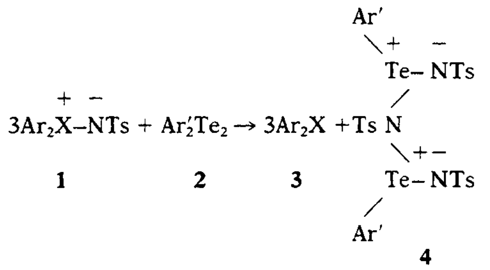
[b] This was identified as diphenylselendichloride: mp 187–188°C (Ref. [26], mp 187–188°C).

[c] This was identified as diphenyltellurdichloride: mp 162–163°C (Ref. [27], mp 162–163°C).

[d] Mp 53–54°C (Ref. [28], mp 53–54°C).

this takes place, **1b–1d** are reduced practically quantitatively to diphenyl selenide **3a** or diaryl tellurides **3b,c** (Equation 1).

In Reaction 1, tellurimides **1c,d** are much more reactive than selenimide **1b**. Thus, the completion of this reaction for the case of diphenyl ditelluride **2a** and Se-imide **1b** in boiling toluene requires 0.75 hours, whereas for Te-imides **1c,d**, under the same conditions, it takes only 5 minutes (Table 1). Thus, the reactivity grows significantly in the order S < Se < Te.



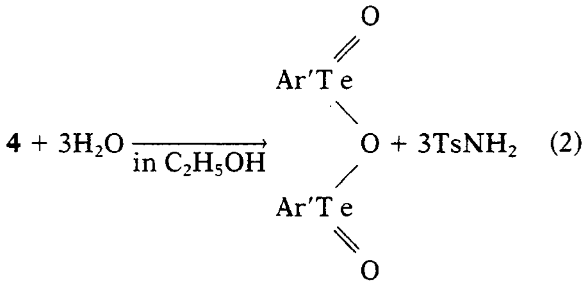
$$3\underset{1}{Ar_2\overset{+}{X}-\overset{-}{N}Ts} + \underset{2}{Ar'_2Te_2} \rightarrow 3\underset{3}{Ar_2X} + \underset{4}{Ts\,N(\overset{+}{Te}(Ar')-\overset{-}{N}Ts)_2} \qquad (1)$$

3a: X = Se, Ar = Ph **4a**: Ar′ = Ph
b: X = Te, Ar = Ph **b**: Ar′ = 4–$CH_3OC_6H_4$
c: X = Te, Ar = 4–$CH_3OC_6H_4$
Ts = 4–$CH_3C_6H_4SO_2$

IR spectra of compounds **4a,b** exhibit both of the bands that can be assigned to the stretching frequencies of the moiety NSO_2 in sulfonamides: 1330–1327 [$\nu^{as}(SO_2)$], 1153 [$\nu^{s}(SO_2)$], 916–911 [$\nu(NS)$] cm^{-1} [1], and the bands corresponding to the stretching frequencies of this moiety in *N*-sulfonyltellurimides: 1223 [$\nu'^{as}(SO_2)$], 1123–1116 [$\nu'^{s}(SO_2)$], 960 [ν' (NS)] cm^{-1} [3]. Compounds **4a,b** are poorly soluble and hydrolyze readily to arenetellurinic anhydrides **5a,b** when washed with aqueous ethanol (Equation 2).

TABLE 2 Oxidation of Diaryl ditellurides **2** by Diaryl Telluroxides **6**

Starting Materials		*Reaction Time (Minutes)*	*Products (Yield in %)*			
6a	**2a**	5	**3b**	(99)	**5a**	(100)
	2b	3	**3b**	(99)	**5a**	(100)
6b	**2a**	5	**3c**	(99)	**5a**	(99)
	2b	3	**3c**	(100)	**5b**	(100)



$$4 + 3H_2O \xrightarrow[\text{in } C_2H_5OH]{} (Ar'Te(=O))_2O + 3TsNH_2 \qquad (2)$$

5a: Ar′ = Ph
b: Ar′ = 4–$CH_3OC_6H_4$

It has also been found by the present authors that anhydrides **5a,b** are easily formed through oxidation of ditellurides **2a,b** by a three fold molar amount of a diaryl telluroxide **6** in boiling toluene (Equation 3). The results are presented in Table 2.

$$\underset{\mathbf{6}}{Ar_2TeO} + \mathit{2} \xrightarrow{\textit{toluene, refluxing}} \mathit{3}\,\mathbf{\mathit{3}} + \mathbf{\mathit{5}} \qquad (3)$$

For the discussion of the mechanism of this reaction, the hydrolysis of an arenetellurenyl iodide **7** should be considered. This gives rise to arenetellurinic anhydrides **5** and ditellurides **2** in the ratio of 1:2 (Equation 4) [8]. This transformation probably proceeds by the intermediate formation of an arenetellurenic anhydride **8** that decomposes to compounds **5** and **2**.

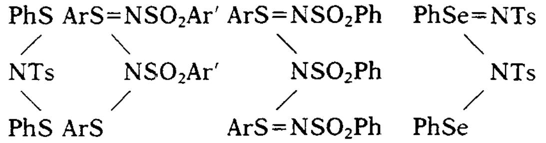
$$6\underset{\mathbf{7}}{ArTeI} + 3H_2O \xrightarrow[-6HI]{} 3\underset{\mathbf{8}}{[ArTe{-}O{-}TeAr]} \rightarrow$$
$$\rightarrow \underset{\mathbf{5}}{(ArTeO)_2O} + 2\underset{\mathbf{2}}{Ar_2Te_2} \qquad (4)$$

Areneselenenic anhydrides have the same property [9]. For this reason, Reaction 3 may also involve **8** as an intermediate. In analogy to the reduction of sulfoxides by disulfides [10], it might be suggested that the formation of **8** includes a homolytic cleavage of the Te–O bond, leading to the formation of radicals. However, when the reaction was conducted in an ESR spectrometer cell, no ESR signals were observed.

N-p-Tosyldiaryltellurimides and diaryl telluroxides possess very similar chemical properties. This is particularly true for the reactions proceeding via semipolar bond Te–N and Te–O cleavage, i.e., for phosphines [11,12], thiols [4,12], thioamides [4,12], and hydroquinone and its derivatives [6,12]. Therefore, it is conceivable that Reaction 1 may develop by a mechanism like the one for Reaction 2.

By analogy with the reduction of *N*-sulfonylsulfimides by disulfides [13], it may be assumed that aminoradicals are formed by homolytic Te–N bond cleavage during the formation of *N,N*-bis-(arenetellurenyl)-*p*-tosylamide **13**. However, an ESR investigation of this reaction does not confirm, as in the case of telluroxides, the formation of such radicals. The sulfur analog of amide **13**, namely, *N,N*-bis-(benzenesulfenyl)-*p*-tosylamide (**14**), was isolated along with diphenyl sulfide as the product of the reaction between *N-p*-tosyl-*S,S*-diphenylsulfimide and diphenyl disulfide [13]. The compound **14**, under heating in *o*-dichlorobenzene to 160°C, gives the ESR spectrum of the radical $^{\bullet}$N(SPh)Ts [13]. Unlike their sulfur analogs, bis-(tellurenyl)amides **13** are very labile and, similarly to arenetellurenic anhydrides **8**, decompose easily to isoelectronic nitrogen analogs of arenetellurinic anhydrides **5**, compounds **4**, and diaryl ditellurides **2** in a 1:2 ratio.

By reactions of *N,N*-bis-(sulfenyl)amides of type **14** with 1 equivalent of dry chloramine-B [14] and of diaryl disulfides with 2 equivalents of similar chloroamines [15,18], *N*-(arenesulfenyl)-*N*-(*N'*-sulfonylimidoarenesulfinyl)sulfonamides [15] were prepared. Sulfur analogs of compounds **4**, *N,N*-bis-(*N'* - benzenesulfonylimidoarenesulfinyl)benzene - sulfonylamides **16**, were synthesized by the action of 1 equivalent of dry chloroamine-B [16] on amides **15** or of 2 equivalents of chloroamine-B [17] on compounds of type **14**. Thus, on consecutive addition of three NSO_2Ar groups, the complete series of *N*-sulfonyl derivatives, the compounds **14**, **15**, and **16**, were isolated.



14 **15** **16** **17**

A selenium analog of amide **15**, *N*-(benzeneselenenyl), *N*-(*N-p*-tosylimidobenzeneseleninyl)-*p*-tosylamide (**17**), was prepared by reaction of diphenyl diselenide with 2 equivalents of chloroamine-T [18]. However, in the case of diaryl ditellurides, owing to the instability of compounds **13**, Reaction 1 gives the product of complete addition of all three groups NSO_2Tol-*p*, namely, amides **4**.

The increase in reactivity of *N-p*-tosylchalcogenimides **1** in the reaction under consideration in the order S < Se < Te may be explained on the basis of the mechanism suggested for Reaction 1. It is known [1,2a] that the basicity of *N*-sulfonylchalcogenimides as well as chalcogenonium ylides [3d] increases in the same order. At the same time, for the series of reagents with a common attacking atom, the order of the variation of nucleophilicity parallels that for basicity according to Brensted [19]. Hence, the growth of chalcogenimides reactivity in Reaction 1 when moving down the Periodic Table of Elements may be related to the fact that, in the initial step, the nucleophilic attack of a negatively charged nitrogen atom on a tellurium atom of the Te–Te bond is facilitated in the same order.

CONCLUSIONS

In the reaction with diaryl ditellurides **2**, the reactivity of *N-p*-tosylchalcogenimides **1** increases in the following order: S < Se < Te. This is in agreement with the results obtained in studying some other reactions proceeding with the cleavage of the chalcogen–nitrogen bond in chalcogenimides [1,4–6]. The oxidation of diaryl ditellurides with selenimides and tellurimides results in the formation of *N,N*-bis-(*N'-p*-tosylimidoarenetellurinyl)-*p*-tosylamides **4**, the isoelectronic nitrogen analogs of arenetellurinic anhydrides **5**. Compounds **5** were prepared by treating ditellurides **2** with diaryltelluroxides **6**. Some mechanisms of the reactions under investigation have been suggested.

EXPERIMENTAL

Melting points were taken in open capillary tubes using a Mel-Temp melting point apparatus and are uncorrected. Infrared spectra were recorded on a Specord IR spectrophotometer in Nujol.

Solvents were dried in accordance with standard procedures. Column chromatography was performed on silica gel (70–230 mesh) under normal pressure.

Sulfimide **1a** was prepared by the reaction of diphenyl sulfide with chloroamine-T in the presence of acetic acid by a known method [1], mp 111–112°C (Ref. [1], mp 111–112°C). Selenimide **1b** was synthesized from diphenylselendimethylate and *p*-tosylamide by a known procedure [20], mp 114–115°C (Ref. [20], mp 114–115°C). *N*-(*p*-Toluenesulfonyl)-Te,Te-diphenyltellurimide (**1c**) and *N*-(*p*-toluenesulfonyl) - Te, Te-di(*p*-methoxyphenyl)tellurimide (**1d**) were obtained by the reaction of the corresponding diaryltellurides with chloroamine-T in the presence of a catalytic amount of 18-crown-6 according to a literature procedure [21], mp 141–142°C (Ref. [21], mp 141–142°C) and mp 151–152°C (Ref. [21], mp 151–152°C), respectively. Diphenyl telluroxide (**6a**) and di(*p*-methoxyphenyl) telluroxide (**6b**) were prepared by alkaline hydrolysis of the corresponding diaryltellur dibromides making use of a known procedure [22], mp 190–191°C (decomp.) (Ref. [22c], mp 191.1°C) and mp 190–192°C (decomp.) (Ref. [22d], mp 189–192°C), respectively. Diphenyl ditelluride (**2a**) was isolated after reaction of tellurium with a phenyllithium solution in ether by the known method [23], mp 67°C (Ref. [24], mp 65–67°C). Di(*p*-methoxyphenyl) ditelluride (**2b**) was obtained by reduction of *p*-methoxyphenyltellurtrichloride according to the literature procedure [25], mp 59–60°C (Ref. [25], 59–60°C).

Reactions of N-p-Tosyldiarylchalcogenimides with Diaryl ditellurides

(a) Reaction of Tellurimide **1d** *and Ditelluride* **2b** *in a 1:1 Ratio.* To a solution of tellurimide **1d** (2.56 g, 5 mmol) in refluxing anhydrous toluene under dry Ar was added a solution of ditelluride **2b** (2.35 g, 5 mmol) in 15 mL of toluene. The reaction mixture was then refluxed for ~10 minutes until a colorless precipitate formed. After the mixture had been cooled to room temperature, the precipitate was filtered off, washed with anhydrous hexane, dried in vacuo over P_2O_5 at 80°C, and identified as *N,N*-bis-(*N'*-*p*-toluenesulfonylimido-*p*-methoxyphenyltellurinyl)-*p*-toluenesulfonylamide (**4b**): 1.6 g (yield 98%), mp 108°C; IR: 1580, 1497, 1330 $[\nu^{as}(SO_2)]$, 1295, 1245, 1225 $[\nu'^{as}(SO_2)]$, 1175, 1153 $[\nu^{s}(SO_2)]$, 1123 $[\nu'^{s}(SO_2)]$, 1076, 1017, 960 $[\nu'(NS)]$, 916 $[\nu(NS)]$, 807, 684 cm^{-1}. Anal. calcd for $C_{35}H_{35}N_3O_8S_3Te_2$: C, 43.03; H, 3.58; found, C, 43.18; H, 3.49. The filtrate was evaporated in vacuo. Column chromatography of the solid residue (ether-ligroin 1:10) yielded di(*p*-methoxyphenyl) telluride (**3c**) (1.67 g, 98%) and ditelluride **2b** (1.52 g, 65%).

(*b*) *Reaction of Chalcogenimides* **1b–1d** *with Ditellurides* **2a**, **2b** *in the Ratio of 3:1* (*Table 1*). All the reactions were carried out according to a standard typical procedure. This procedure for the case of tellurimide **1d** and ditelluride **2a** is described later. In benzene and toluene, reactions were performed at the respective boiling points, and, in *o*-dichlorobenzene, at 110°C. Three mmoles of chalcogenimides **1b–1d** and 1 mmole of ditellurides **2a**, **b** were used. Reaction time, conditions, and yields are given in Table 1.

Typical Procedure: Reaction of Tellurimide **1d** *with Ditelluride* **2a**. To a solution of tellurimide **1d** (1.53 g, 3 mmol) in 30 mL of boiling anhydrous toluene was added a solution of ditelluride **2a** (0.41 g, 1 mmol) in 10 mL of toluene. The reaction mixture was then refluxed until the solution became colorless (5 minutes), and it was then cooled to room temperature. The colorless precipitate of *N,N*-bis-(*N'*-*p*-toluenesulfonylimidobenzenetellurinyl)-*p*-toluenesulfonylamide (**4a**) was filtered off, washed with dry hexane, and dried in vacuo over P_2O_5 at 80°C. 0.89 g (97%) of compound **4a** was isolated, mp 122°C; IR: 1593, 1566, 1490, 1327 $[\nu^{as}(SO_2)]$, 1223 $[\nu'^{as}(SO_2)]$, 1153 $[\nu^{s}(SO_2)]$, 1116 $[\nu'^{s}(SO_2)]$, 1080, 1027, 960 $[\nu'(NS)]$, 911 $[\nu(NS)]$, 807 , 737, 686 cm^{-1}. Anal. calcd for $C_{33}H_{31}N_3O_6S_3Te_2$: C, 43.23; H, 3.38; found, C, 43.14; H 3.45. Evaporation of the filtrate in vacuo yielded 1.01 g (98%) of di(*p*-methoxyphenyl) telluride (**3c**).

Hydrolysis of Compounds **4a** *and* **4b**

Compound **4a** (or **4b**) (0.5 mmol) in 15 mL of hydrous ethanol was stirred at room temperature for 0.5 hours. A precipitate was filtered off and identified as benzenetellurinic anhydride (**5a**): yield 100%, mp 223–225°C (Ref. [29], mp 220–225°C) or as *p*-methoxyphenyl-tellurinic anhydride (**5b**): yield 100%, mp 200–205°C (decomp.) (Ref. [29], mp 200–205°C decomp.). The filtrate was evaporated and the dry residue was identified as *p*-toluenesulfonylamide: yield 100%, mp 138°C (Ref. [30], mp 138°C); IR: 3340 (NH), 3245 (NH), 1330 $[\nu^{as}(SO_2)]$, 1152 $[\nu^{s}(SO_2)]$, 910 $[\nu(NS)]$ cm^{-1}.

Oxidation of Diaryl tellurides with Diaryl telluroxides

(*a*) *Reaction of Telluroxide* **6b** *with Diphenylditelluride* **2a** *in 1:1 Ratio.* The mixture of telluroxide **6b** (1.79 g, 5 mmol) and ditelluride **2a** (2.04 g, 5 mmol) was refluxed in 50 mL of toluene for 10 minutes, and this was allowed to cool to room temperature. A colorless precipitate that had formed was separated by filtration, washed with hexane, and identified as anhydride **5a**: 0.76 g, yield 100%, mp 223–225°C. The filtrate was evaporated in vacuo. Column chromatography of the residue (as indicated previously) yielded 1.66 g (97%) of telluride **3c** and 1.31 g (64%) of ditelluride **2a**.

(*b*) *Reaction of telluroxides* **6a**, **b** *with ditellurides* **2a**, **b** *in the Ratio of 3:1* (*Table 2*). All the reactions were carried out using a typical procedure. This procedure for the oxidation of ditelluride **2b** by telluroxide **6b** is described subsequently. All the reactions were performed in boiling toluene. Three mmoles of telluroxides **6a,b** and 1 mmol of ditellurides **2a,b** were used for the reactions. The reaction time and yields are presented in Table 2.

The typical Procedure: Oxidation of Ditelluride **2b** *with Telluroxide* **6b**. To a solution of telluroxide **6b** (1.07 g, 3 mmol) in 30 mL of boiling toluene was added a solution of ditelluride **2b** (0.47 g, 1 mmol) in 10 mL of toluene. The reaction mixture was refluxed until it became colorless (3 minutes), and then it was allowed to cool to room temperature. A colorless precipitate was filtered off, washed with hexane, and dried in vacuo. Anhydride **5b** (0.51 g, 99%) with mp 200–205°C (decomp.) was obtained. After evaporation of the filtrate *in vacuo*, telluride **3c** (1.02 g, 99%) was isolated.

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