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B.A. Trofimov on his 70th anniversary

Synthesis and Paramagnetic Properties of Polytelluride Oligomers

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Abstract—Organotellurium oligomers with different lengths of the telluride fragments and hydrocarbon chains were synthesized by reactions of tellurium with organic dihalogen derivatives in the system hydrazine hydrate–KOH. Oligomers containing two and three contiguous tellurium atoms in the solid state give rise to strong ESR signals with a g value of 2.005–2.030 and a line width of 180–290 Oe. The possibility for formation of tellurium-centered radicals is discussed. Nitrosodurene as radical scavenger in chloroform solution trapped neither primary radical species nor radical products derived therefrom.

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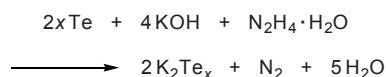
Tellurium-containing polymers and oligomers are key compounds for the preparation of organic semiconductors [1] and other electrotechnical materials [2–6]. Such polymers are commonly synthesized from dihalogen derivatives of hydrocarbons and alkali metal tellurides or ditellurides; the latter are generated in turn from elemental tellurium using various basic reducing systems [7]. Activation of tellurium with the system hydrazine hydrate–alkali allowed us to synthesize poly(trimethyleneditelluride) [8], and preliminary tests showed that it possesses paramagnetic properties [9].

We tried to extend the synthetic potential of the reaction of tellurium with the above system and, using various dihalogen derivatives **I**, synthesized a number of organotellurium oligomers **II–VII** with different lengths and structures of the hydrocarbon chain between tellurium atoms and different numbers of contiguous tellurium atoms, and the resulting oligomers were examined by ESR spectroscopy.

Elemental tellurium in the system KOH–hydrazine hydrate is converted into polytelluride anion Te_x^{2-} (Scheme 1) [7]. This reaction does not require addition of a solvent, and the value of x in K_2Te_x is determined by the Te–KOH ratio. Potassium ditelluride is formed

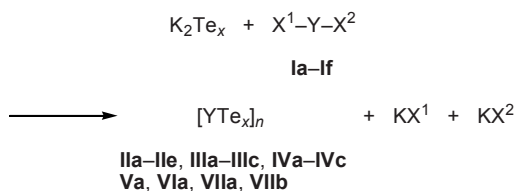
mainly at an equimolar ratio of tellurium and potassium hydroxide. Increase in the amount of tellurium with respect to KOH leads to formation of a mixture of polytellurides for which the x value reflects the average length of the polytelluride chain. Potassium telluride K_2Te is selectively formed only in the presence of a large excess of alkali, at a Te–KOH ratio of 1:(6–8) [7].

Scheme 1.



Polytellurides thus obtained were brought (without isolation) into reactions with dihalides **Ia–If**; as a result, black viscous oligomeric products **II–VII** were formed (Scheme 2, Table 1) with the x values varying according to Scheme 1. The average molecular weight of oligomers **II–VII** was calculated on the basis of the concentration of residual halogen; it varied over a fairly wide range, from 2200 to 51000, depending on the nature of the hydrocarbon chain, x value in K_2Te_x , halogen nature (X^1 , X^2), and other factors. Solid samples of oligomers **II–VII** were examined by ESR

Scheme 2.

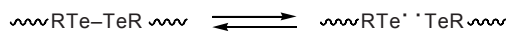


Y = (CH₂)₃, X¹ = Cl, X² = Br (**a**), X¹ = X² = Br (**b**); Y = (CH₂)₅, X¹ = X² = Br (**c**), Cl (**d**); Y = (CH₂)₂O(CH₂)₂, X¹ = X² = Cl (**e**); Y = CH₂CH(CH₃)CH₂, X¹ = Cl, X² = Br (**f**).

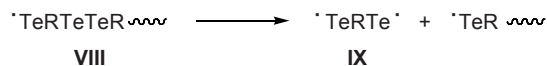
spectroscopy. It was found that oligomers having polytelluride fragments Te_x with $x \geq 2$ (i.e., containing two and more contiguous tellurium atoms) possess paramagnetic properties (Table 2). Samples of these oligomers were characterized by strong singlets in the ESR spectra with a g -factor of 2.005–2.031, which differs considerably from the g -factors typical of a free electron (2.0023) and hydrocarbon radicals [10]. Broad lines with $\Delta H = 190$ –290 Oe observed at 20°C indicate short relaxation time. These findings led us to presume that paramagnetic properties of the examined oligomers originate from the presence of tellurium-centered radicals **VIII** generated by homolytic dissociation of Te–Te bonds (Scheme 3). An appreciable width of the

ESR lines also suggests probable formation of diradicals like **IX** (Scheme 4).

Scheme 3.



Scheme 4.



Radicals **VIII** and **IX** are stabilized by the polymeric matrix, so that their lifetime is fairly long. Upon storage of a sample of oligomer **IIb** in an ESR ampule for three months, the signal became narrower from 290 to 200 Oe, and the g -factor decreased from 2.011 to 2.004 (Table 2). When a sample of **IIb** was kept for even longer time (8 months) on exposure to atmospheric air, an additional broad signal (1200 Oe) with a g -factor of 2.671 appeared in the ESR spectrum. Most probably, that signal originates from oligomer oxidation products. Anomalously broad lines (500–750 Oe) with large g -factors (2.20–2.40) were also observed in the ESR spectra of oligomers **IIb** and **IIIa** upon UV irradiation, presumably as a result of oxidation.

Table 1. Yields, molecular weights, and elemental analyses of polytelluride oligomers **II–VII**

Oligomer no.	Initial dihalide no.	Molar ratio Te–KOH	Yield, %	Found, %				M^b	Formula (elementary unit RTe _x)	Calculated, %		
				C	H	Te	Hlg ^a			C	H	Te
IIa	Ia	1:8	77.4	20.3	3.3	72.5	2.9	3900	C ₃ H ₆ Te	21.2	3.5	72.3
IIb	Ia	1:1	94.6	11.6	2.1	83.4	2.1	5530	C ₃ H ₆ Te ₂	12.1	2.0	85.9
IIc	Ia	1:1.2	96.0	9.9	2.2	88.9	0.9	12980	C ₃ H ₆ Te _{2.4}	10.3	1.7	87.9
IId	Ia	1:1.3	98.0	9.2	1.5	89.2	1.4	8430	C ₃ H ₆ Te _{2.6}	9.6	1.6	88.8
IIe	Ia	1:1.5	98.0	8.1	1.2	91.0	1.5	7600	C ₃ H ₆ Te ₃	8.5	1.4	90.1
IIIa	Ib	1:1	98.0	11.7	2.1	86.8	0.5	31600	C ₃ H ₆ Te ₂	12.1	2.0	85.9
IIIb	Ib	1:1.25	98.0	9.6	1.9	87.8	0.8	19040	C ₃ H ₆ Te _{2.5}	10.0	1.7	88.4
IIIc	Ib	1:1.5	98.0	8.1	1.3	89.5	1.0	15240	C ₃ H ₆ Te ₃	8.5	1.4	90.1
IVa	Ic	1:1	98.0	17.5	2.9	81.1	1.5	10600	C ₅ H ₁₀ Te ₂	18.4	3.1	78.5
IVb	Ic	1:1.25	96.0	14.0	2.5	83.1	0.3	51610	C ₅ H ₁₀ Te _{2.5}	15.4	2.6	82.0
IVc	Ic	1:1.5	96.0	12.5	1.6	85.8	4.8	3340	C ₅ H ₁₀ Te ₃	13.2	2.2	84.5
Va	Id	1:1	96.0	15.6	2.5	77.1	3.2	2220	C ₅ H ₁₀ Te ₂	18.4	3.1	78.5
VIa	Ie	1:1.25	96.0	10.5	2.0	85.3	2.8	2530	C ₄ H ₈ OTe _{2.5}	12.3	2.0	81.6
VIIa	If	1:1	98.0	16.6	2.9	81.9	5.1	2290	C ₄ H ₁₀ Te ₂	15.3	3.2	81.5
VIIb	If	1:1.25	98.0	12.8	2.5	84.8	4.4	2640	C ₄ H ₁₀ Te _{2.5}	12.7	2.7	84.6

^a Percentage of Cl, Br or Cl+Br.

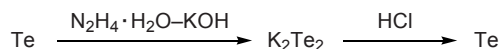
^b The molecular weights were calculated from the concentration of residual halogen (terminal groups).

Table 2. Parameters of the ESR spectra of polytelluride oligomers **II–VII**

Parameter	IIa ^a	IIb	IIc	IId	IIf	IIIa	IIIb	IIIc	IVa	IVb	IVc	Va	VIa	VIIa	VIIb
<i>g</i> -Factor (±0.006)	–	2.011	2.026	2.012	2.012	2.005	2.009	2.005	2.005	2.005	2.006	2.008	2.005	2.031	2.006
ΔH , Oe	–	290	270	260	240	220	210	220	200	190	236	245	180	280	210
$N \times 10^{18}$, spin/g	–	13.1	8.7	6.6	1.4	0.2	5.4	0.7	2.1	2.3	0.1	1.8	0.2	11.0	1.4

^a Monotelluride is diamagnetic.

Among oligomers with the same Y fragment, the maximal intensity of the ESR signal was typical of those with $x = 2$ –2.5. Presumably, high rigidity of tritelluride fragments –Te–Te–Te– makes their homolytic dissociation more difficult as compared to ditelluride fragments. Complete absence of paramagnetic properties of monotelluride oligomer **IIa** indicates that dissociation of C–Te bonds does not occur under the given conditions. By special experiment we showed that polytelluride chains in elemental tellurium prepared by activation in hydrazine hydrate–KOH and subsequent regeneration by the action of hydrochloric acid (Scheme 5) do not undergo homolytic dissociation: the resulting tellurium sample was diamagnetic.

Scheme 5.

Samples of oligomers **IVa**, **IVb**, and **Va** containing more flexible five-carbon chains or $(\text{CH}_2)_2\text{O}(\text{CH}_2)_2$ fragments (**VIa**) and having the same x values give rise to weaker signals as compared to oligomers **IIb–IId**. Presumably, flexibility of the polymer molecule facilitates combination of radicals according to Scheme 3.

The formation of tellurium-centered radicals was postulated in many reactions of organotellurium compounds [11, 12], especially in such processes as pyrolysis [13], photolysis [14], and oxidation [15]. However, unlike sulfur- and selenium-centered radicals, tellurium-centered radicals were not detected by ESR spectroscopy prior to our present study, despite numerous attempts to detect such species even with the use of spin trapping technique [14]. A few indirect proofs for the formation of unstable tellurium-centered radicals were given in [15, 16], where decomposition products of such species (organic radicals) were trapped by nitrosodurene as spin scavenger, and the structure of the final products was determined.

Dissolution of the obtained oligomers in organic solvents leads to complete disappearance of signal in

the ESR spectra. Their solutions in chloroform are intensely blue (oligomers **IIb–IIf** and **IIIa–IIIc** with a three-carbon elementary unit), orange–yellow (oligomers **IVa–IVc** and **Va** having five methylene groups in an elementary unit), orange–red (**VIa**), and dark blue (**VIIa**, **VIIb**). According to the GC–MS (^{130}Te), ^1H NMR, and UV data, solutions of oligomers in chloroform contained the following compounds: 1,2-ditellurolane (**X**), m/z 302 (I_{rel} 24%) $[M]^{++}$ [9, 17, 18], in solutions of **IIb–IIf** and **IIIa–IIIc**; 4-methyl-1,2-ditellurolane (**XI**), m/z 316 (I_{rel} 24%) $[M]^{++}$, in solutions of **VIIa** and **VIIb**; 1,2-ditelluracycloheptane (**XII**), m/z 330 (I_{rel} 25%) $[M]^{++}$, in solutions of **IVa–IVc** and **Va**; and 5-oxa-1,2-ditelluracycloheptane (**XIII**), m/z 332 (I_{rel} 24%) $[M]^{++}$, in solution of **VIa**.

Yellow solutions of oligomers **IVa–IVc** and **Va** are stable. Blue solutions of **IIb–IIf**, **IIIa–IIIc**, **VIIa**, and **VIIb** in wet solvents (benzene, chloroform) become colorless in 30 min, and a brown diamagnetic solid separates therefrom [9]. Simultaneously, long-wave absorption bands at λ 550–700 nm typical of the above compounds disappear from the electronic spectra (Table 3). As shown in [9], the formation of 1,2-ditellurolane (**X**) from the corresponding polymer is very consistent with the possibility for generation of tellurium-centered radicals (Scheme 3) and diradicals like **IX** (Scheme 4).

Assuming that the formation of 1,2-ditellurolane (**X**) from **IIb–IIf** and **IIIa–IIIc** and its subsequent oxidative decomposition [9] follow radical mechanism, we examined the behavior of oligomer **IIb** in chloroform in the presence of nitrosodurene. No spin adducts were detected by ESR spectroscopy, but the solution remained blue for a fairly long time (several days, cf. [9]). The electronic spectrum of a solution of 1,2-ditellurolane (**X**) in chloroform in the presence of nitrosodurene contained two overlapping absorption bands in the region λ 550–700 nm (λ_{max} 569 and 661 nm). The spectral pattern did not change over a period of 24 h. Nitrosodurene does not absorb in the

Table 3. Electronic absorption spectra of solutions of polytelluride oligomers and model compounds having Te–Te bonds

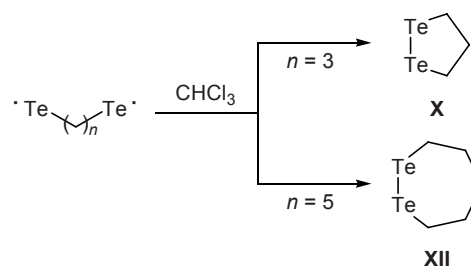
Compound	Y	λ_{max} , nm		Solvent	Reference
		solution	+ nitrosodurene		
1,2-Ditellurolane (X) from IIb–IIe or IIIa–IIIc	(CH ₂) ₃	561, 654	569, 661	CHCl ₃	
4-Methyl-1,2-ditellurolane (XI) from VIIa or VIIb	CH ₂ CH(Me)CH ₂	576, 658	576, 658	CHCl ₃	
1,2-Ditelluracycloheptane (XII) from IVa–IVc or Va	(CH ₂) ₅	378, 400	400	CHCl ₃	
5-Oxa-1,2-ditelluracycloheptane (XIII) from VIa	(CH ₂) ₂ O(CH ₂) ₂	378, 404		CHCl ₃	
EtTeEt		290		C ₆ H ₁₄	[19]
EtTeTeEt		395		C ₆ H ₁₄	[19]
1,2-Ditellurolane (X)	(CH ₂) ₃	581, 676		C ₆ H ₆	[17]
		580, 667		C ₆ H ₆	[18]
		601, 690		CS ₂	[18]
1,2-Ditelluracycloheptane (XII)	(CH ₂) ₅	398		C ₆ H ₆	[18]
Nitrosodurene		309		CHCl ₃	

above region: its long-wave absorption maximum is located at λ 309 nm. The observed absorption cannot be assigned to linear fragments with two contiguous tellurium atoms in the oligomer. Freshly prepared oligomer **IIb** displayed no absorption in the region λ 550–700 nm (the spectrum was recorded from thin film applied to a quartz plate). In the electronic spectrum of diethyl ditelluride in hexane the long-wave absorption maximum is located at λ 395 nm [19].

The electronic spectrum of a solution of oligomer **IIb** in chloroform in the presence of nitrosodurene contained absorption bands typical of 1,2-ditellurolane (**X**) in the region λ 550–700 nm (Table 3). These data indicate conservation of the five-membered 1,2-ditellurolane ring in the presence of nitrosodurene. Unusual pattern in the electronic absorption spectra of diethyl derivatives of tellurium was observed for the first time in [17]: in going from tellurides to ditellurides, the long-wave absorption band displaced by 105 nm to the red region. Even larger red shift (to λ 280 nm) was reported for 1,2-ditellurolanes as compared to acyclic compounds having a Te–Te bond [15]. The strong red shift of the long-wave absorption maximum in going from diethyl ditelluride to 1,2-ditellurolane (**X**) is rationalized in terms of considerably increased overlap of *p*-orbitals bearing lone electron pairs on the tellurium atoms. The observed 7-nm red shift of both absorption maxima of 1,2-ditellurolane (**X**) in the presence of nitrosodurene suggests that intermolecular interactions induce only slight change of the geometric parameters of the 1,2-ditellurolane ring, i.e., the structure of the latter remains essentially unchanged. Presumably, 1,2-ditellurolane (**X**) with nitrosodurene forms a fairly

stable complex. Only in this case the Te–Te bond in five-membered heterocycles is protected from the action of water and oxygen molecules, leading to oxidative cleavage of the Te–Te bond [9]. The nature of the 1,2-ditellurolane–nitrosodurene complex requires more detailed examination, which will be the subject of our further studies.

The above stated led us to presume that homolytic decomposition of oligomers **II–VII** gives rise to tellurium-centered diradicals 'Te–(CH₂)_{*n*}–Te' (*n* = 3–5) which are stabilized by the polymeric matrix. They can exist over a fairly long time as quasitriplet states, and their recombination in solution leads to compounds **X** and **XII** (Scheme 6).

Scheme 6.

This assumption is consistent with considerable width of the ESR signals of polytelluride oligomers and gradual loss of their paramagnetic properties on prolonged storage without protection from atmospheric moisture and oxygen (recombination of radicals). Nitrosodurene molecule is an electron acceptor, while 1,2-ditellurolane molecule is an effective electron donor, as follows from its ready oxidation [9]. Thus in

the examined systems nitrosodurene acts as an acceptor stabilizing readily oxidizable 1,2-ditellurolane molecule rather than as spin scavenger.

Paramagnetic oligomers containing tellurium-centered radicals attract interest as promising working media for radiofrequency lasers and magnetometers, radical initiators, etc. [20].

EXPERIMENTAL

The electronic absorption spectra were measured on a Lambda-35 UV-Vis spectrometer from thin films applied onto quartz plates or from solutions in CHCl_3 containing 1.67 mg/ml of nitrosodurene (0.1-cm quartz cells). The IR spectra were recorded from thin films or KBr pellets on Bruker IFS-25 and Varian FT-IR 3100 spectrometers with Fourier transform, equipped with an ATR (attenuated total reflection) adapter (ZnSe crystal). The ^1H , ^{13}C , and ^{125}Te NMR spectra were obtained on a Bruker DPX-400 instrument at 400.13, 100.62, and 126.2 MHz, respectively, from solutions in CDCl_3 using hexamethyldisiloxane as internal reference (^1H and ^{13}C). The ESR spectra were recorded on a Radiopan SE/X-2547 spectrometer equipped with a high-frequency meter and a magnetometer. The concentration of paramagnetic centers was calculated as described in [18] using a sample of CuCl_2 as reference. The mass spectra (electron impact, 70 eV) were obtained on a Shimadzu GCMS-QP5050A instrument (quadrupole mass analyzer, a.m.u. range 34–650; SPB-5 capillary column, 60 m $\times$ 0.25 mm $\times$ 0.25 μm ; carrier gas helium, flow rate 0.7 ml/min, inlet pressure 150 kPa; injector and ion source temperature 250°C, oven temperature programming from 60 to 250°C at a rate of 10 deg/min).

Oligomer IIa. A solution of 11.2 g (200 mmol) of potassium hydroxide in 80 g (1600 mmol) of hydrazine hydrate was heated to 75°C, and 3.19 g (25 mmol) of powdered tellurium was added over a period of 0.5 h. The dark red mixture was heated for 2 h at 80–90°C and cooled to 25°C, 4.29 g (27 mmol) of 1-bromo-3-chloropropane was added over a period of 15 min, and the mixture was heated for 2.5 h at 65°C and cooled. The oily material was separated, washed with water, ethanol, and diethyl ether, and dried. Yield 3.28 g (77%, calculated on the initial tellurium). IR spectrum, ν , cm^{-1} : 2955, 2917, 2836 [$\nu(\text{CH}_2)$]; 1437, 1404 [$\delta_s(\text{CH}_2)$]; 1331, 1304, 1274, 1124, 1078, 1036, 996 [$\rho_\omega(\text{CH}_2)$, $\rho_t(\text{CH}_2)$]; 1198, 1173, 947, 902, 841, 790, 761, 696, 650, 592, 549, 487 [$\nu(\text{CC})$, $\nu(\text{CTe})$]. The data of elemental analysis are given in Table 1.

The other polytelluride oligomers were synthesized in a similar way using the corresponding dihalogen derivatives **Ia–If** at a Te–KOH ratio indicated in Table 1. The IR spectra of the products did not contradict the assumed structures and were generally similar to the IR spectrum of **IIa**. Their elemental analyses are given in Table 1, and ESR spectral parameters, in Table 2.

Behavior of oligomer IIb in chloroform in the presence of nitrosodurene. *a.* An ESR ampule was charged with 0.12 g (0.4 mmol) of oligomer **IIb**, and its ESR spectrum was recorded (Table 2). Anhydrous chloroform, 1 ml, was added to obtain a blue solution of 1,2-ditellurolane (**X**) which showed no signal in the ESR spectrum. Nitrosodurene, 0.01 g (0.06 mmol), was then added to the ampule, and no signal appeared in the ESR spectrum of the mixture. The solution remained transparent and blue on storage for about 6 days at –10°C. The ^1H NMR spectra of all solutions were recorded. All operations were carried out under argon.

b. A solution of 0.01 g (0.06 mmol) of nitrosodurene in 1 ml of CDCl_3 was added to 0.12 g (0.4 mmol) of oligomer **IIb**, the blue solution of 1,2-ditellurolane (**X**) was separated by decanting, and its ^1H NMR spectrum was recorded. The ^1H NMR spectrum recorded after 2 days showed no change in the spectral pattern.

c. Synthesis of oligomer IIb in the system hydrazine hydrate–KOH in the presence of nitrosodurene. A solution of 0.93 g (16.6 mmol) of potassium hydroxide in 6.7 g (134 mmol) of hydrazine hydrate was heated to 70–80°C, 2.13 g (16.7 mmol) of powdered tellurium was added over a period of 0.5 h, the mixture was stirred for 2 h at 80–85°C and cooled to 27°C, and 0.02 g (0.12 mmol) of nitrosodurene and 1.3 g (8.2 mmol) of 1-bromo-3-chloropropane were added. After 15 min, a part of the oligomer was separated and treated as described above. We thus isolated 0.5 g of oligomer **IIb** (first portion). The remaining mixture was heated for 1.5 h at 60–62°C, cooled, and treated in a similar way to isolate 1.69 g of **IIb** (second portion). Both portions of oligomer **IIb** gave no signal in the ESR spectrum.

d. A specially designed ESR ampule consisting of a spherical base and a thin vertical arm was charged under argon with 0.01 g (0.06 mmol) of nitrosodurene, 2 ml of chloroform, and 0.12 g (0.4 mmol) of viscous oligomer **IIb**. The ampule was sealed, the blue solution was transferred into the arm by decanting, and its ESR

spectrum was recorded. The spectrum displayed no signal. The arm was then opened, the blue solution was transferred into an NMR ampule, CDCl_3 was added, and the ^1H , ^{13}C , and ^{125}Te NMR spectra were recorded.

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