

## 1,2-DITELLUROLANE : A NEW SYNTHETIC METHOD, STRUCTURE AND CHEMICAL REACTIONS

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*A simple method for the synthesis of 1,2-ditellurolane is proposed, based on the reaction of elemental tellurium with 1,3-dihalopropane in a system of hydrazine hydrate and alkali followed by the effect of organic solvent on the first formed poly(trimethyleneditellurides). The products of the oxidation and reduction of 1,2-ditellurolane were characterized and its spectral characteristics were analyzed.*

**Keywords:** 1,3-bis((methyltelluro)propane, hydrazine hydrate, 1,2-ditellurolane, 1,3-propaneditelluric acid, tellurium, tellurethane, quantum chemical-calculations.

The reaction of elemental sulfur with alkenylbenzenes, leading to the formation unique heterocyclic compounds – phenyl-substituted 1,2-dithiole-3-thiones – was discovered by M. G. Voronkov in the middle of the last century [1-4]. Then under his quindance 1,2-dithiol-3-one [5] and 1,2-diselenol-3-one [6] were prepared. This made possible the development of the synthetic chemistry of heterocycles with two chalcogen atoms which possess aromatic properties. Many of these or their derivatives were used in organic synthesis [7] and also as biologically active compounds [8] or industrial products [9].

Saturated heterocyclic systems containing two chalcogen atoms (1,2-dichalcogenolans) have been far less studied. This is explained by the difficulty of making and separating them, especially the selenium and tellurium analogs. A multistage synthesis of 1,2-diselenolane has been described using potassium cyanide [10] or metallic lithium [11]. This compound has found use in organic synthesis, for example, in the preparation of stable cross-linked cyclodextrins with selenium bridges with hydrophobic cavities which are able to form inclusion complexes [12], and also calixarenes containing selenium [13]. The preparation of 1,2-ditellurolane **1** was first reported in 1984 [14]. It was obtained by the reaction of 1,3-dibromopropane with polytelluride anions generated by electrochemical reduction of tellurium and was characterized by UV spectroscopy and mass spectrometry. Subsequently 1,2-ditellurolane was obtained by cyanide activation of tellurium followed by reaction with 1,3-dibromopropane [15]. The product was only characterized in solution. The chemical properties of 1,2-ditellurolane was not mentioned in these papers.

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\* Dedicated to Academician of the Russian Academy of Science Mikhail Grigor'evich Voronkov on his 85<sup>th</sup> Birthday.

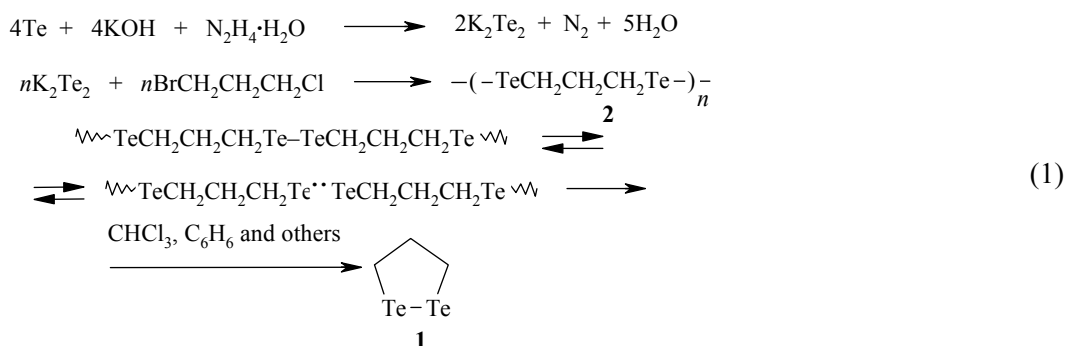
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We recently developed a method for the synthesis of 1,2-dithiolane [16] and 1,2-diselenolane [17] based on the activation of sulfur or selenium in a hydrazine hydrate–alkali system. The reaction of tellurium with 1-bromo-3-chloropropane was carried out in this system. Oligomeric poly(trimethyleneditelluride) **2** was obtained [18]. The oligomeric nature of the tellurokol **2** was confirmed by IR spectroscopy and reductive fission of the Te–Te bond in the hydrazine hydrate–alkali system.

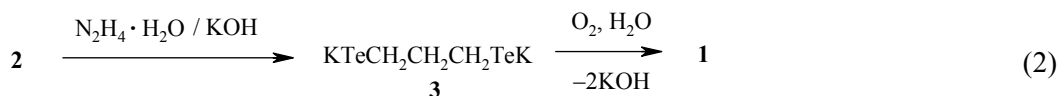
It was observed unexpectedly that treatment of the tellurokol with an organic solvent (acetone, chloroform, benzene, hexane, etc.) led to the formation of intensely blue solutions on which the presence of 1,2-ditelluroolane **1** was demonstrated unambiguously by UV and IR spectroscopy, chromato-mass spectrometry, and <sup>1</sup>H NMR spectroscopy. The appearance and growth in intensity of long wave length absorptions bands at λ = 400 (shoulder), 581, and 667 nm, characteristic of 1,2-ditelluroolane [14, 15] were observed in the UV spectrum of tellurokol **2** in benzene. The yield of 1,2-ditelluroolane reached 14% (estimated by <sup>1</sup>H NMR spectroscopy with introduction of a standard additive).

To elucidate how 1,2-ditelluroolane **1** was formed from the tellurokol oligomer **2** the system was studied by ESR spectroscopy. It was observed that samples obtained by reaction of tellurium with 1-bromo-3-chloropropane in the hydrazine hydrate–alkali system are paramagnetic. The characteristics of the ESR spectrum depend on how long the sample has been kept and the additional effect of ultraviolet radiation and will be discussed in a separate publication. However in all cases the value of the *g*-factor and the width of the lines permit a decision on the possibility of localization of the unpaired electron on the tellurium atom. Thus it is possible to suggest that the conversion of **2** → **1** is linked to homolysis of the tellurokol at the Te–Te bond, and introduction of a solvent facilitates the separation of the radical pair. Further radical heterocyclization led to the formation of 1,2-ditelluroolane **1**. In summary, the synthesis of 1,2-ditelluroolane can be represented by the following scheme:



After removing 1,2-ditelluroolane by the solvent both the remaining black solid and the obtained blue solution (in any solvent) did not give an ESR signal.

1,2-Ditelluroolane **1** was obtained from the tellurokol **2** by another method. The tellurokol **2** was subjected to reductive fission at the Te–Te bond in a hydrazine hydrate–KOH system in which it was completely soluble. The solution of the ditelluroolate **3** was extracted with benzene. In the course of the extraction the oxidation of the ditelluroolate **3** to 1,2-ditelluroolane **1** was observed and **1** transferred into the organic phase. The yield of 1,2-ditelluroolane **1** by this method was about 25%.



According to the results of quantum calculations with DFT(B3LYP)/LanL2DZ the molecular structure of the most stable isomer of 1,2-ditelluroolane **1** corresponds to the least steric hindrance with the two tellurium atoms out of the plane of the three carbon atoms by 22–26°. It is only slightly affected by the medium in the range ε = 1 ÷ 78 (Fig. 1, Table 1).

Table 1. Bond Lengths ( $l$ ) and Angles ( $\delta$ ) of the 1,2-Ditellurothane **1** Molecule in the Gas Phase and Various Media Calculated by the DFT(B3LYP)/LanL2DZ (RSM-model) Method

Bond	$l, \text{\AA}$			
	$\epsilon = 1$	$\epsilon = 4.9$	$\epsilon = 36.6$	$\epsilon = 78.4$
Te(1)–Te(2)	2.8386	2.8406	2.8415	2.8413
Te(2)–C(3)	2.1991	2.2008	2.2005	2.2006
Te(1)–C(5)	2.2000	2.2006	2.2005	2.2006
C(3)–C(4)	1.5560	1.5568	1.5553	1.5552
C(5)–C(4)	1.5578	1.5564	1.5577	1.5578
C(3)–H(6)	1.0931	1.0935	1.0938	1.0938
C(3)–H(7)	1.0959	1.0961	1.0963	1.0963
C(4)–H(8)	1.0990	1.0989	1.0991	1.0991
C(4)–H(9)	1.0987	1.0989	1.0988	1.0988
C(5)–H(10)	1.0959	1.0961	1.0962	1.0962
C(5)–H(11)	1.0930	1.0935	1.0936	1.0937
Angle	$\delta, \text{deg}$			
Te(1)–Te(2)–C(3)	82.4	81.3	81.2	81.2
Te(2)–Te(1)–C(5)	81.4	81.2	81.6	81.6
C(3)–C(4)–C(5)	117.3	118.1	118.3	118.3
H(3)–C(3)–H(7)	109.0	109.0	109.0	109.0
H(8)–C(4)–H(9)	107.2	107.2	107.2	107.2
H(10)–C(5)–H(11)	109.0	109.0	109.0	109.0
C(3)–C(4)–C(5)–Te(1)	22.9	24.5	21.6	21.6

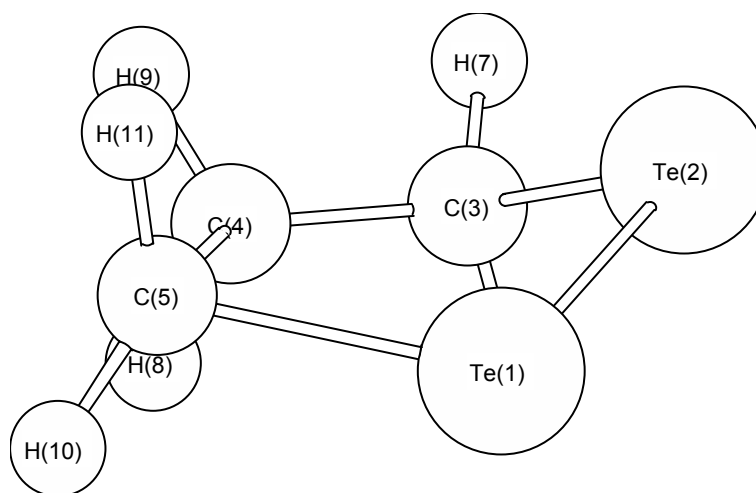


Fig. 1. Geometry of 1,2-ditellurothane **1** in the gas phase, calculated by the DFT(B3LYP)/LanL2DZ method.

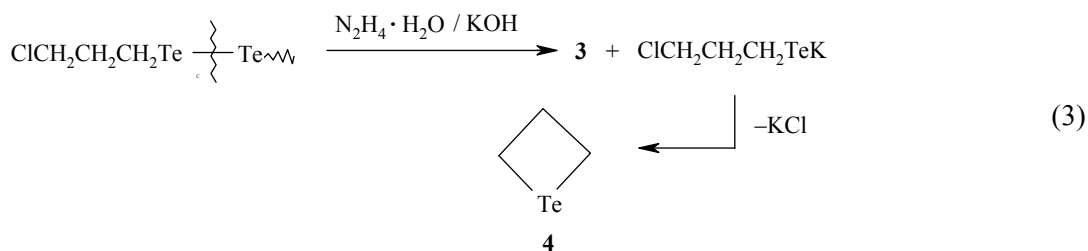
A theoretical analysis of the IR spectra of the 1,2-ditellurothane molecule **1** with symmetry  $C_s$  in the isolate state and in various media was carried out by the DFT(B3LYP)/LanL2DZ method. In the theoretical IR spectrum of 1,2-ditellurothane **1** 27 normal vibrations of symmetry **A** were observed. The frequencies and intensities of the vibrations involving Te atoms are cited in Table 2.

Table 2. Frequencies and Intensities of Bands in the IR Spectra of 1,2-Ditellurothane **1** Molecules in the Gas Phase and in Various Media Calculated by the DFT(B3LYP)/LanL2DZ Method (RSM Model).

N	$\nu$ , $\text{cm}^{-1}$				$I$ , KM/mol				Assignment
	$\epsilon = 1$	$\epsilon = 4.9$	$\epsilon = 36.6$	$\epsilon = 78.4$	$\epsilon = 1$	$\epsilon = 4.9$	$\epsilon = 36.6$	$\epsilon = 78.4$	
1	65	51	33	33	0.13	0.28	0.42	0.43	$\rho_{\text{Te}}(1)\text{C}(5)\text{C}(4)\text{C}(3)$
2	144	139	138	138	0.57	1.45	1.92	1.92	$\rho_{\text{C}}(4)\text{C}(5)\text{C}(3)$
3	174	172	172	171	0.10	0.14	0.18	0.18	$\nu_{\text{Te}-\text{Te}}$ + $+ \rho_{\text{C}}(4)\text{C}(5)\text{C}(3)$
4	199	197	195	195	0.30	0.35	0.38	0.37	$\nu_{\text{Te}-\text{C}} + \delta_{\text{cycl}}$
5	388	385	384	384	0.04	0.01	0.01	0.01	$\nu_{\text{Te}-\text{C}} + \delta_{\text{cycl}}$
6	501	498	497	497	5.34	9.04	11.64	12.12	$\nu_{\text{Te}-\text{C}}$ + $+ \rho_{\text{C}}(4)\text{C}(5)\text{C}(3)$
7	592	590	589	589	2.86	5.89	7.86	7.93	$\nu_{\text{Te}-\text{C}}$ + $+ \delta_{\text{C}}(4)\text{C}(5)\text{C}(3)$

Bands important for the analysis of the structure of 1,2-ditellurothane **1** were found in the cited region of the spectrum. The stretching frequency  $\nu_{\text{Te}-\text{Te}}$  contributes to the normal vibration 3 at  $174 \text{ cm}^{-1}$ , mixed with the torsional vibration  $\rho_{\text{C}}(4)\text{C}(5)\text{C}(3)$ , and has a weak intensity. Changing  $\epsilon$  for the medium in the range 1÷78 does not affect the spectral characteristics of this vibration. The normal vibrations 4-7 correspond to the stretching vibration  $\nu_{\text{Te}-\text{C}}$  with contributions from various deformations of the five-membered ring. On increasing  $\epsilon$  for the medium the intensities of the normal vibrations 6 and 7 increase considerably. In the experimental IR spectrum of 1,2-ditellurothane **1** in  $\text{CHCl}_3$  the normal vibrations 6 and 7 correspond to bands at  $525$  and  $620 \text{ cm}^{-1}$  respectively. Starting from a comparison of the theoretically calculated ( $\epsilon = 4.9$ ) and experimental frequencies the scaling multiplier equals 1.05, which characterizes the satisfactory precision of the calculated IR spectrum of 1,2-ditellurothane **1** using the DFT(B3LYP)/LanL2DZ method.

Apart from 1,2-ditellurothane **1**, telluretane **4** was observed in trace quantities among the products from reaction (2) in the organic phase by chromato-mass spectrometry. It is probably formed by reactions of the end groups of tellurokol **2** (according to elemental analysis the latter contains about 3% residual halogen [18]).

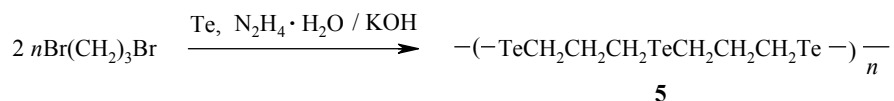


Reaction (3) shows that, despite the probable strain in the molecule, telluretane **4** is sufficiently stable to withstand the reaction conditions ( $70^\circ\text{C}$ , the hydrazine hydrate-alkali system, 1.5 h) and extraction of the reaction mixture with benzene in the air.

When 1,3-dibromopropane was used in reaction (1) in place of 1-bromo-3-chloropropane oligomer **5** was formed which is practically diamagnetic. However after irradiation with UV light for 1.5 double sufficiently intense-signal was observed in the ESR spectrum of oligomer **5**:  $g_1 = 2.394$ ,  $\Delta H = 460 \text{ Oe}$ ;  $g_2 = 2.003$ ,  $\Delta H = 160 \text{ Oe}$ . The effect of organic solvents on tellurokol **5** gave a pale-blue solution. Only with chloroform was 1,2-ditellurothane **1** detected by chromato-mass spectrometry.

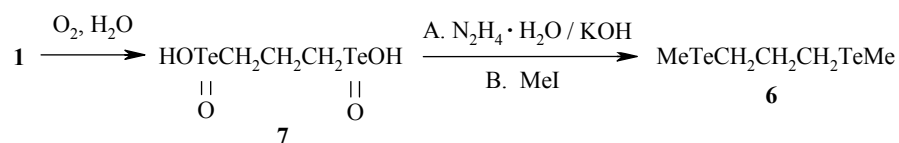
The difference in behavior of oligomers **2** and **5** is most likely explained by differences in the structure of the oligomer chains which, in its turn, is determined by the difference in reactivity of the chlorine and bromine atoms in the nucleophilic substitution by the telluride anion. When tellurium is activated by the

hydrazine hydrate-alkali system an equilibrium of polytellurides  $K_2Te_x$  is formed, in which the average value of  $x$  is regulated by the  $Te:KOH$  ratio [19]. In the case of 1,3-dibromopropane an oligomer is formed which, on the basis of elemental analysis, may be ascribed the probable structure **5** in which, to a certain extent, an alternation of mono- and ditelluride units is observed:



In oligomer **2** ditelluride units predominate [18]. These conclusions were confirmed by reductive fission of oligomers **2** and **5** with the hydrazine hydrate-alkali system with the object of preparing 1,3-bis(methyltelluro)propane **6**: compound **6** was obtained in 47.5% yield from tellurokol **2** [18] but only in 19% yield from the oligomer **5**. Thus it is preferable to use 1-bromo-3-chloropropane rather than 1,3-dibromopropane for the synthesis of 1,2-ditellurolane **1** by reaction (1).

The blue solutions of 1,2-ditellurolane **1** are extremely unstable in light and in the presence of moisture (they are more stable in the dark and in absolutely dry solvents). Over several minutes in light (depending on the amount of solution and its concentration) the blue color is completely extinguished and a light-brown precipitate is formed. This product is assigned the structure of 1,3-propaneditelluric acid **7** on the basis of elemental analysis, formed by the oxidation of 1,2-ditellurolane **1**.

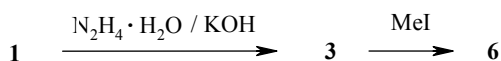


The theoretical IR spectrum of the molecule of the diacid **7**, calculated by the DFT(B3LYP)/LanL2DZ method in the harmonic approximation, contains 45 normal vibrations of symmetry **A**, of which 30 normal vibrations fall within the range 400-4000  $cm^{-1}$ ; these vibrations are not influenced by the medium. Comparison with the experimental IR spectrum of compound **7** (KBr disc) shows a scaling multiplier of 1.08 for all the bands in the 400-4000  $cm^{-1}$  range. According to the calculations, the intense band at 608  $cm^{-1}$  with a shoulder at 684  $cm^{-1}$  corresponds to the characteristic vibration  $\nu_{Te-O}$ . The doublet at 3421  $cm^{-1}$  (shoulder at 3250  $cm^{-1}$ ) with medium intensity are stretching vibrations of the hydroxyl groups,  $\nu_{O-H}$ . The considerable width of these bands in the experimental spectra (up to 150  $cm^{-1}$ ) and the presence of doublets (there are 2 bands less in the theoretical spectrum in the 400-4000  $cm^{-1}$  range) indicates strong intermolecular interactions of the end groups in molecules of 1,3-propaneditelluric acid **7**.

The diacid **7** did not dissolve in water or normal organic solvents. When it was treated with hydrazine hydrate a vigorous exothermic reaction was observed, accompanied by considerable formation of gas and the separation of elemental tellurium. When hydrazine hydrate-KOH was used a vigorous reaction was also observed, accompanied by separation of elemental tellurium, but the solution formed had an intense black-violet colour.

Alkylation of the solution with methyl iodide gave small amounts of dimethyltelluride, dimethylditelluride, and compound **6** (overall yield about 3%). Hence the basic product of this reaction is regenerated tellurium (yield about 90%). The diacid **7** dissolved in aqueous alkali to give an unstable brown solution.

If the blue solution of ditellurolane **1** in benzene is added dropwise to a concentrated solution of alkali in hydrazine hydrate, vigorous reductive opening of the ring at the Te-Te bond was observed with formation of the propaneditellurate **3**. Methylation of the solution obtained with methyl iodide gave 1,3-bis(methyltelluro)propane **6** in 60% yield.



This reaction gives hope for the possibility of use 1,2-ditellurolane **1** in organic synthesis.

In conclusion a new simple method is proposed for the synthesis of 1,2-ditellurolane using available reagents, the geometric characteristics of the molecule have been calculated, and the products of its oxidation in air and its reduction in the hydrazine hydrate–KOH system have been characterized.

## EXPERIMENTAL

The purity of reagents and reaction products were monitored by GLC using LKhM-80 MD (liquid phase DC-550 or XE-60, 5% on Chromaton–AW–HMDS as carrier, a 2000 x 3 mm steel columns, linear temperature programming of the column at 12°/min, helium carrier gas ) and Tsvet-500 chromatographs (2000 x 5 mm steel columns, liquid phase XE-60 silicone, 5% on Chromaton–AW–HMDS as carrier, linear temperature programming of the columns from 30 to 230°C at 12°/min, helium carrier gas). Mass spectra were recorded with a Shimadzu GCMS-QP5050A chromato-mass spectrometer (SPB<sup>TM</sup>-5 column, 60000 x 0.25 μm), thickness of the nonmobile phase 0.25 μm, injector temperature 250°C, helium carrier gas with a flow rate of 0.7 ml/min, temperature programmed from 60 to 260°C at 15°/min. Detector temperature 250°C, quadrupole mass analyzer, electron ionization, electron energy 70 eV, temperature of the ion source 200°C, range of masses detected 34-650 Daltons. IR spectra of the products of thin layers in CHCl<sub>3</sub> (*d* = 0.006 cm) or in KBr disks were recorded with Specord IR-75 and Bruker IFS-25 spectrometers, and <sup>1</sup>H NMR spectra of CDCl<sub>3</sub> solutions with HMDS as internal standard (δ 0.05 ppm) were recorded on a Bruker DPX-400 (400 MHz) machine. UV spectra were recorded on a Lambda 35 UV-Vis spectrometer (solvent C<sub>6</sub>H<sub>6</sub>, *d* = 1 cm). ESR spectra of tellurokols were recorded on a Radiopan SE/X2547 spectrometer fitted with a magnetometer and a microwave frequency detector.

**1,2-Ditellurolane 1.** A. A sample of tellurokol **2**, prepared by a literature method [18], was treated in a dark apparatus with several portions of the corresponding solvent. To determine the yield in the solution, 1 g of a standard sample (toluene or cyclohexane) was added and the mass of 1,2-ditellurolane was calculated from the ratio of the peaks of the product and the standard in the <sup>1</sup>H NMR spectrum. UV spectrum, λ, nm: 667, 581, 400 (shoulder) (cf. [14, 15]). <sup>1</sup>H NMR spectrum, δ, ppm (*J*, Hz): 3.40 (4H, t, <sup>3</sup>*J*<sub>H</sub> = 6.2, CH<sub>2</sub>Te), 3.83 (2H, q, <sup>3</sup>*J*<sub>H</sub> = 6.2, CCH<sub>2</sub>C) [15]. Mass spectrum (*m/z* for the <sup>130</sup>Te isotope): 302 [M]<sup>+</sup>, 260 [Te<sub>2</sub>]<sup>+</sup>, 144 [CH<sub>2</sub>Te]<sup>+</sup>, 130 [Te]<sup>+</sup>, 41 [C<sub>3</sub>H<sub>5</sub>]<sup>+</sup>, 39 [C<sub>3</sub>H<sub>3</sub>]<sup>+</sup>. According to <sup>1</sup>H NMR data and chromato-mass spectrometry C(16)-C(28) hydrocarbons with linear and cyclic structures were present in the solution: (δ, ppm) 0.85 (br. t, CH<sub>3</sub>), 1.24 (br. s, aliphatic CH<sub>2</sub>), 1.54 (br. s, cyclic CH<sub>2</sub> in large rings). Evidently the hydrocarbons are formed from trimethylene fragments from the tellurokol **2**, their yield was 3-5% of tellurokol **2** used in the reaction.

B. Tellurokol **2** (1 g), KOH (0.933 g, 16.6 mmol), and hydrazine hydrate (4.2 g, 84 mmol) were placed in a flask fitted with a stirrer, a thermometer, and a reflux condenser. The reaction mixture was stirred for 1.5 h at 70°C and cooled to 25°C. The homogeneous solution formed was extracted with benzene in the air. The blue solution contained 1,2-ditellurolane **1**, identical with the product from method A, yield 0.25 g (25%). In contrast to method A, C(16)-C(28) hydrocarbons were practically absent from the benzene extract. Traces of telluretan **4** were detected by chromato-mass spectrometry. Mass spectrum, *m/z* for isotope <sup>130</sup>Te (*I*, % from the total ion current taking account of the isotopic ions, characteristic of the ion): 172 [M]<sup>+</sup> (16), 144 [M -C<sub>2</sub>H<sub>4</sub>]<sup>+</sup> (10), 130 [Te]<sup>+</sup> (6), 41 [C<sub>3</sub>H<sub>5</sub>]<sup>+</sup> (50), 39 [C<sub>3</sub>H<sub>3</sub>]<sup>+</sup> (18).

**Tellurokol 5.** KOH (1.4 g, 25 mmol) and hydrazine hydrate (1 g, 200 mmol) were placed in a reaction flask. Finely ground tellurium (3.19 g, 25 mmol) was added in portions at 75-85°C. The mixture was stirred at 85°C for 2 h, cooled, and 1,3-dibromopropane (3.82 g, 18.9 mmol) was added dropwise at 25-35°C to practically completely colorless solution. The viscous black oligomer which formed on the side of the flask was

separated, washed many times with hot water, and dried to give compound **5**, yield 3.25 g (88%). Found, %: C 14.20; H 2.40; Br 2.87; Te 80.91. Calculation of the molecular mass from the residual bromine (end group method) gave a value of 6050 au (cf. [18]). Calculated for C<sub>6</sub>H<sub>12</sub>Te<sub>3</sub>, (the repeat unit of **5**), %: C 15.38; H 2.56; Te 82.05. 1,2-Ditellurolane **1** was obtained from tellurokol **5** as described in method A.

**1,3-Propaneditelluric Acid 7.** A chloroform solution containing 1,2-ditellurolane **1** (0.47 g, 1.6 mmol) was kept in the light until the solution was completely decolorized. The light-brown precipitate of the diacid **7** was filtered off, washed with chloroform, and dried. Yield 0.56 g (97%), mp 140-150°C (dec.). Found, %: C 10.06; H 2.37; Te 71.61. C<sub>3</sub>H<sub>8</sub>O<sub>4</sub>Te<sub>2</sub>. Calculated, %: C 9.85; H 2.19; Te 69.87.

**1,3-Bis(methyltelluro)propane 6.** A. Acid **7** (0.35 g, 0.96 mmol) was added to a solution of KOH (1 g, 1.8 mmol) in hydrazine hydrate (4 g, 80 mmol). Methyl iodide (1.48 g, 10 mmol) was added dropwise to the deep-red solution. The mixture was extracted with dichloromethane and analyzed by GLC and chromatomass spectroscopy. Compound **6** was observed in the solution.

B. A benzene solution (80 ml) containing 1,2-ditellurolane **1** (0.38 g, 1.28 mmol) was added dropwise at 50-55°C to a solution of KOH (14 g, 250 mmol) in hydrazine hydrate (50 g, 1000 mmol). The blue color was instantaneously extinguished. The benzene layer was separated and methyl iodide (0.36 g, 2.56 mmol) was added dropwise to the hydrazine. The mixture obtained was extracted with ether and analyzed by GLC and chromatomass spectrometry. According to GLC (method of standard additions), the extract contained compound **6** (0.25g), the characteristics cited in [18].

The theoretical analysis of the most stable isomers of 1,2-ditellurolane **1** and compound **7** were carried out using the functional density matrix method DFT(B3LYP)/LanL2DZ using the Gaussian 03W suite of programs [20]. Complete optimization of the geometry was needed. The influence of the media in the range  $\epsilon = 1\div 78$  on the structural characteristics and vibration spectra was calculated using the approximation of the solvation in the PCM model.

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