Polyhedron 73 (2014) 45-50

Contents lists available at ScienceDirect

Polyhedron

journal homepage: www.elsevier.com/locate/poly



Electrophilic cyclization of homopropargyl tellurides: Synthesis and supramolecular structures of 2-aryl-3-iodo-1-phenyl-tellurophenium iodides and polyiodides



Roberta Cargnelutti^{a,*}, Ernesto S. Lang^a, Davi F. Back^a, Ricardo F. Schumacher^{b,*}

^a Departamento de Química, Laboratório de Materiais Inorgânicos, Universidade Federal de Santa Maria, 97105-900 Santa Maria, RS, Brazil ^b LASOL, CCQFA, Universidade Federal de Pelotas, UFPel, PO Box 354, 96010-900 Pelotas, RS, Brazil

ARTICLE INFO

Article history: Received 29 November 2013 Accepted 11 February 2014 Available online 19 February 2014

Keywords: Electrophilic cyclization Tellurophenium iodides and polyiodides Supramolecular assemblies

ABSTRACT

The synthesis of five new 2-aryl-3-iodo-1-phenyl-tellurophenium iodides and polyiodides via electrophilic cyclization of homopropargyl tellurides using I_2 as an electrophile is described. The homopropargyl tellurides **1a** and **1b** react with different amounts of molecular iodine to give $[C_{16}H_{14}Tel]I$ (**2**), $[C_{16}H_{14}Tel]I_3$ (**3**), $\{[C_{16}H_{14}ITe]_4[I_3]_3[I^-]\cdot 2I_2\}$ (**4**), $[C_{17}H_{16}OTel]I$ (**5**), and $[C_{17}H_{16}OTel]I_3$ (**6**). All of these compounds display inter and/or intramolecular secondary interactions between Te \cdots I and/or I \cdots I in the solid state, and these interactions are responsible for the formation of various supramolecular assemblies. © 2014 Elsevier Ltd. All rights reserved.

1. Introduction

Tellurium is a versatile metalloid that is commonly used as a base for several important materials, including catalysts and semiconductors, and it is used to prepare synthetic intermediates in organic synthesis that enable the introduction of various functional groups [1–8]. Among these compounds, organotellurium(II) derivatives and vinylic tellurides in particular provide powerful intermediates for the synthesis of more elaborate alkenes, such as natural products [6]. Many organotellurium(IV) compounds with different ligands have been prepared and studied to date [9–12]. Tellurium(IV) iodides are of great interest because they can promote structural diversity due to the tellurium geometry. In the solid state, Te and I atoms interact strongly to form interand intramolecular secondary interactions through the stereochemically active lone pair at the Te center. These interactions enable the formation of supramolecular networks. In the context of supramolecular chemistry, all possible combinations of secondary interactions are important for creating dimers and polymeric chains that form 1D, 2D, and 3D networks of the tellurium iodides [13-18].

On the other hand, the electrophilic cyclization of alkynes containing a nucleophile near to the triple bond, which is activated

by the presence of an electrophilic source, such as iodine, copper halides, and ArSeBr, represents an important protocol for the preparation of highly substituted heterocycles [19]. The mechanism underlying this reaction involves the coordination of an electrophile to the π C–C bond of the alkyne, followed by the nucleophilic anti-attack of the non-ligand heteroatom electron pair to the activated unsaturated C–C bond to give the heterocycle [19]. Inspired by this reaction, our recent success in this area [20], and continuing interest in the synthesis and applications of chalcogenophenes [21–24], we envisioned the preparation of dihydrotellurophenium iodides **2–6** using 1-arylItelluro-3-alkynes **1** and molecular iodine as an electrophilic source (Scheme 1).

We report here the synthesis of five new 2-aryl-3-iodo-1phenyl-tellurophenium iodides and polyiodides via the electrophilic cyclization of homopropargyl tellurides using I_2 as an electrophile. The products (**2–6**) were obtained in the presence of 1, 2, or 3 equiv. I_2 in CH₂Cl₂/THF at room temperature and under inert conditions. In all compounds, $[C_{16}H_{14}Te1]I$ (**2**), $[C_{16}H_{14}Te1]I_3$ (**3**), $\{[C_{16}H_{14}ITe]_4[I_3^-]_3[I^-]\cdot 2I_2\}$ (**4**), $[C_{17}H_{16}OTe1]I$ (**5**) and $[C_{17}H_{16}OTe1]I_3$ (**6**), the tellurium atom is present in the oxidation state +4.

Initially, we focused our study on the synthesis of the homopropargyl tellurides **1** using homopropargyl tosylates and diaryl ditellurides. The arylltellurolate anion, generated *in situ* by the reaction of diaryl ditelluride with NaBH₄ in THF/EtOH, reacted with a solution of the homopropargyl tosylate in THF at room temperature and under an inert atmosphere for 6 h via a S_N2 reaction [25,26]. The homopropargyl tellurides **1a** and **1b** are shown in Scheme 2 and were obtained in good yields.

^{*} Corresponding authors. Tel.: +55 53 32757356; fax: +55 53 3275 7533.

E-mail addresses: rocargnelutti@yahoo.com.br(R. Cargnelutti), ricardo.schumacher@ ufpel.edu.br(R.F. Schumacher).



Scheme 1. General synthesis of dihydrotellurophenium iodides 2-6.



Scheme 2. Synthesis of homopropargyl aryltellurides 1a and 1b.

With the starting materials **1** in hand, we turned out our attention to the preparation of the dihydrotellurophenium iodides **2–6** via an electrophilic cyclization mechanism involving iodine as an electrophilic source. The products are presented in Scheme 3. The compounds **2–4** were prepared by reacting the homopropargyl phenyltelluride **1a** with 1, 2, or 3 equivalents of molecular iodine. The compounds **5** and **6** were synthesized by the reactions of the homopropargyl 2-methoxyphenyl telluride **1b** with 1 or 2 equivalents of I₂ using THF/CH₂Cl₂ as solvent. All products obtained were isolated by crystallizing the mother solution via slow evaporation of the solvents.

It is important to note that the dihydrotellurophenium iodides **2–6** isolated in this work can be considered as intermediates of an electrophilic cyclization reaction. The crucial step in many electrophilic cyclization reactions [19] involves the removal of the carbonic group bonded to the heteroatom via a S_N2 displacement (step **B**). S_N2 displacement proceeds in the presence of a nucleophile to generate the expected heterocycle **7** and the co-product **8** (Scheme 4).

To avoid the formation of **7**, we selected homopropargyl tellurides having an aryl group directly bonded to the tellurium



2. Experimental

2.1. General

All manipulations were conducted under an Ar (argon) atmosphere using standard Schlenk techniques and dry solvents. These solvents were purified and dried according to literature procedures [27]. Elemental analyses (CHN) were carried out using a VARIO EL (Elementar Analysen systeme GmbH) analyzer. Infrared spectra were measured using a Bruker Tensor 27 mid-IR spectrometer. Melting points were determined on a Microquímica MQAPF-301 melting point apparatus and are uncorrected.

2.2. Preparation of $[C_{16}H_{14}ITe]I(2)$

To a solution of 0.083 g (0.25 mmol) **1a** in 10 mL CH₂Cl₂ was added 0.063 g (0.25 mmol) I₂ dissolved in 10 mL THF. The mixture was stirred for 3 h. The slow evaporation of the orange solution gave yellow crystals of **2**. Yield: 76% based on **1a**. Properties: yellow crystalline substance. Melting point: 144 °C-145 °C. Elemental *Anal.* Calc. for C₁₆H₁₄I₂Te (587.69) C, 32.70; H, 2.40. Found: C, 32.73; H, 2.29%. IR (KBr, cm⁻¹): 3046 (w), 3011 (w), 2948 (w), 1601 (m), 1486 (m), 1475 (m), 1434 (m), 881 (m), 766 (m), 729 (s), 700 (s), 682 (m), 558 (w), 445 (w). High resolution ESI + MS (*m*/*z*) of $[C_{16}H_{14}ITe]^+$: calc.: 462.9197, Found: 462.9207.



Scheme 3. The synthesis of compounds 2–6 were conducted using the starting material 1 (0.25 mmol) and different equivalents of molecular iodine in a CH₂Cl₂/THF solvent at room temperature over 3 h.



Scheme 4. The hypothetical heterocycle 7 can not be formed in the reaction.

2.3. Preparation of $[C_{16}H_{14}ITe]I_3$ (3)

To a solution of 0.083 g (0.25 mmol) **1a** in 5 mL CH₂Cl₂ were added 0.127 g (0.50 mmol) I₂ dissolved in 15 mL THF. The mixture was stirred for 3 h. The slow evaporation of the dark red solution gave red crystals of **3**. Yield: 71% based on **1a**. Properties: red crystalline substance. Melting point: 132 °C-133 °C. IR (KBr, cm⁻¹): 3048 (w), 2937 (w), 2899 (w), 1594 (w), 1569 (w), 1485 (m), 1474 (m), 1436 (s), 1244 (m), 756 (s), 728 (s), 691 (s), 680 (s), 557 (w), 449 (w). High resolution ESI + MS of $[C_{16}H_{14}ITe]^+$: calc.: 462.9197, Found: 462.9199.

2.4. Preparation of $\{[C_{16}H_{14}ITe]_4[I_3^-]_3[I^-]\cdot 2I_2\}$ (4)

To a solution of 0.083 g (0.25 mmol) **1a** in 5 mL CH₂Cl₂ were added 0.190 g (0.75 mmol) I₂ dissolved in 20 mL THF. The mixture was stirred for 3 h. The slow evaporation of the red dark solution gave dark red crystals of **4**. Yield: 67% based on **1a**. Properties: dark red crystalline substance. Melting point: 122 °C–123 °C. IR (KBr, cm⁻¹): 3047 (w), 3012 (w), 2948 (w), 1618 (m), 1590 (w), 1573 (w), 1486 (m), 1434 (s), 1219 (w), 995 (m), 912 (w), 881 (w), 730 (s), 691 (s), 558 (w), 446 (w). High resolution ESI + MS (*m*/*z*) of $[C_{16}H_{14}ITe]^+$: calc.: 462.9197, Found: 462.9209.

2.5. Preparation of $[C_{17}H_{16}IOTe]I(\mathbf{5})$

To a solution of 0.091 g (0.25 mmol) **1b** in 5 mL CH₂Cl₂ were added 0.063 g (0.25 mmol) I₂ dissolved in 10 mL THF. The mixture was stirred for 3 h. The slow evaporation of the orange solution gave yellow crystals of **5**. Yield: 72% based on **1b**. Properties: yellow crystalline substance. Melting point: 162 °C–163 °C. Elemental *Anal.* Calc. for C₁₇H₁₆I₂OTe: C, 33.05; H, 2.61. Found: C, 33.18; H,

Table 1

Crystallographic data and refinement parameters for 2, 3, 4, 5, and 6.

2.49%. IR (KBr, cm⁻¹): 3048 (w), 2936 (w), 2835 (w), 1659 (w), 1610 (m), 1482 (s), 1459 (m), 1433 (s), 1253 (s), 1109 (m), 1025 (m), 888 (m), 756 (s), 730 (s), 682 (m), 581 (w). High resolution ESI + MS (m/z) of [C₁₇H₁₆IOTe]⁺: calc.: 492.9308, Found: 492.9303.

2.6. Preparation of $[C_{17}H_{16}IOTe]I_3$ (**6**)

To a solution of 0.091 g (0.25 mmol) **1b** in 5 mL CH₂Cl₂ were added 0.127 g (0.50 mmol) l₂ dissolved in 15 mL THF. The mixture was stirred for 3 h. The slow evaporation of the red solution gave red crystals of **6**. Yield: 76% based on **1b**. Properties: red crystalline substance. Melting point: 151 °C–152 °C. IR (KBr, cm⁻¹): 2996 (w), 1619 (m), 1480 (m), 1427 (m), 1245 (s), 1110 (m), 1017 (m), 754 (s), 731 (s), 680 (m), 579 (w), 558 (w). High resolution ESI + MS (*m*/*z*) of $[C_{17}H_{16}IOTe]^+$: calc.: 492.9308, Found: 492.9308.

2.7. Crystallography

Data collection was performed on a Bruker APEX II CCD area detector diffractometer using graphite-monochromatized Mo K α radiation. The structure was solved by direct methods using SHELXS [28] and refined on F^2 using anisotropic temperature parameters for all non-hydrogen atoms [29]. Hydrogen atoms were included in the refinement during the calculation of the positions. Crystal data and details of the data collection and refinement processes are summarized in Table 1. Table 2 summarizes selected bond distances and angles within the compounds **2–6**.

3. Results and discussion

The DIAMOND [30] representations of compounds 2-6 are presented in Figs. 1–6, and the crystallographic data are shown in

Crystal Data	2	3	4	5	6
Formula	C ₁₆ H ₁₄ I ₂ Te	C ₁₆ H ₁₄ I ₄ Te	C ₆₄ H ₅₆ I ₁₈ Te ₄	C17H16I2OTe	C17H16I4OTe
Formula weight (g mol ⁻¹)	587.67	841.47	3619.69	617.70	871.50
T (K)	296(2)	293(2)	293(2)	293(2)	293(2)
Crystal system	monoclinic	triclinic	monoclinic	monoclinic	monoclinic
Space group	$P2_1/n$	$P\overline{1}$	C2/m	$P2_1/n$	$P2_1/c$
a (Å)	12.325(5)	9.553(4)	27.452(1)	12.329(4)	12.560(3)
b (Å)	11.301(5)	9.776(4)	16.167(8)	11.667(4)	9.595(3)
<i>c</i> (Å)	13.031(5)	13.527(6)	9.93	13.308(4)	18.395(4)
α (°)	90	98.682(2)	90	90	90
β(°)	103.954(3)	101.066(2)	93.032(2)	104.85(1)	91.09(1)
γ (°)	90	118.794(2)	90	90	90
V (Å ³)	1761.4(1)	1042.04(8)	4400.8(4)	1850.6(1)	2216.5(1)
Ζ	4	2	2	4	4
$D_{\rm calc} ({\rm g}{\rm cm}^{-3})$	2.216	2.682	2.732	2.217	2.612
μ (Mo K α) (mm ⁻¹)	5.181	7.346	7.660	4.941	6.915
λ/Å	0.71073	0.71073	0.71073	0.71073	0.71073
F (000)	1072	748	3204	1136	1560
Collected reflections	20724	17067	28962	57418	15680
Unique reflections	5394	4595	5076	5228	6054
Goodness-of-fit (GOF) (F^2)	1.100	1.074	1.050	1.031	1.028
R_1^{a}	0.0370	0.0854	0.0504	0.0444	0.0490
wR_2^{D}	0.0427	0.2840	0.1518	0.0922	0.1200

^a $R_1 = \sum_{0}^{IIF} \frac{I-IF}{c} \frac{II}{c} / \sum_{0}^{IFI} \frac{I}{c}$

^b $wR_2 = \{\sum w(F_0^2 - F_c^2)^2 / \sum w(F_0^2)^2 \}^{1/2}.$

Table 2
Selected bond lengths (Å) and angles (°) for the compounds 2-6

2		3		4		5		6	
Bond lengths		Bond lengths		Bond lengths		Bond lengths		Bond lengths	
Te1···I2#2	3.6564 (1)	Te1···I2	3.6397(1)	Te1···I6	3.6945 (1)	Te1···I2	3.3791(1)	Te1···01	3.2427(1)
Te1···I2#3	3.3477(1)	Te1···I4#1	3.9908(2)	Te1···I8#1	3.7843(1)	Te1···I2#1	3.7961(1)	Te1···I2#1	3.7539(1)
Te(1)-C(1)	2.113(4)	I(3) - I(2)	2.902(2)	Te1···I9	3.5710(1)	Te1···01	3.1215(1)	Te1…I4#2	3.5020(1)
Te(1)-C(16)	2.113(5)	I(3)-I(4)	2.923(1)	$I4 \cdot \cdot \cdot I2$	3.6993(2)	Te1#1…I2#1	3.3791(1)	Te-C(1)	2.112(6)
Te(1)-C(13)	2.159(5)	I(1)-C(14)	2.09(1)	I2···I5#1	3.6016(2)	Te(1)-C(1)	2.119(6)	Te-C(14)	2.125(6)
Bond angles		Te-C(13)	1.94(1)	Te-C(1)	2.122(9)	Te(1)-C(17)	2.123 (7)	Te-C(17)	2.140(5)
C(1)-Te(1)-C(16)	98.5 (2)	Te-C(1)	1.94 (1)	Te-C(16)	2.13(1)	Te(1)-C(14)	2.149(6)	I(3) - I(2)	2.8625(7)
C(1)-Te(1)-C(13)	95.7(2)	Te-C(16)	1.95(1)	Te-C(13)	2.139(9)	Bond angles		I(3)-I(4)	2.9937(7)
C(16)-Te(1)-C(13)	83.0(2)	Bond angles		I(7)-I(8)	2.834(1)	C(1)-Te(1)-C(17)	98.2(3)	Bond angles	
Te1…I2#2…Te1#1	96.562(1)	I(2)-I(3)-I(4)	178.90(5)	I(7)–I(6)	2.998(1)	C(1)-Te(1)-C(14)	94.0(2)	C(1)-Te-C(14)	96.0(2)
		C(13)-Te-C(1)	101.5(6)	I(3) - I(2)	2.893(1)	C(17)-Te(1)-C(14)	82.2(3)	C(1)-Te-C(17)	97.1(2)
		C(13)-Te-C(16)	88.9(6)	I(4) - I(5)	2.746(6)			C(14)-Te-C(17)	83.2(2)
		C(1)-Te-C(16)	100.1(6)	Bond angles				I(2)-I(3)-I(4)	174.81(2)
				C(1)-Te-C(16)	96.6(4)				
				C(1)-Te-C(13)	96.1(4)				
				C(16)-Te-C(13)	84.0(4)				
				I(8) - I(7) - I(6)	177.84(5)				



Fig. 1. DIAMOND [30] representation of the pseudodimeric structure of compound **2**, achieved through Te...I secondary interactions (represented by dashed lines). Symmetry code: #1: -x, 1 - y, -z; #2: -1 + x, y, -1 + z; #3: 1 - x, 1 - y, 1 - z. Hydrogen atoms have been omitted for clarity.

Table 1. The bonds and angles of the corresponding compounds are listed in Table 2.

Fig. 1 shows the pseudodimeric structure of compound **2**. The formed product is an ionic compound in which the tellurium atom is positively charged and is counterbalanced by the negative

charge of the iodide anion. The Te(IV) atom assumes a pyramidal geometry due to the influence of the lone pair of electrons. This figure also shows the pseudodimeric structure of compound **2**. The pseudodimer is stabilized by secondary intermolecular interactions (identified by dashed lines) between Te1...12#2: 3.6564 (1) Å, Te1...12#3: 3.3477 (1) Å and Te1#1...12#2: 3.3477 (1) Å, Te1#1...12#3: 3.6564 (1) Å.

The complex **3** is illustrated in Fig. 2. In this compound, the anion I₃ balances the charges of the $[C_{16}H_{14}|Te]^+$ cation, and the I3–I2 distance is 2.902 (2) Å, I3–I4 is 2.923 (1) Å, and the angle I2–I3–I4 is 178.90 (5)°. Compound **3** form a pseudodimer via intermolecular secondary interactions linking $[C_{16}H_{14}|Te]^+$ to I₃. The following distances were measured: Te1…I2: 3.6397 (1) Å, Te1…I4#: 3.9908 (2) Å, Te1#1…I2#: 3.6397 (1) Å. The secondary interactions of this compound induce the tellurium atom to adopt a distorted square pyramidal geometry.

Fig. 3 shows the molecular structure of compound **4**. For each four cationic unit of $[C_{16}H_{14}ITe]^+$ there are three I_3^- anions, one I^- anion and two neutral I_2 molecules { $[C_{16}H_{14}ITe]_4[I_3^-]_3[I^-]\cdot 2I_2$ }. In the solid state, complex **4** forms secondary interactions between Te1...16: 3.6945 (1) Å, Te1...18#1: 3.7843 (1) Å, and Te1...19: 3.5710 (1) Å. These secondary interactions induce the tellurium atom to adopt a distorted octahedral geometry (Fig. 4). Fig. 4 shows a three-dimensional chain formed by compound **4** in which the unit of the $[C_{16}H_{14}ITe]^+$ cation is connected by secondary interactions with the I_3^- anion. The center of this supramolecular structure



Fig. 2. Pseudodimeric structure of compound 3, formed via Te...I secondary interactions (represented by dashed lines). Symmetry code: #1: -x, -y, -z. For clarity, the hydrogen atoms are not shown.



Fig. 3. Molecular structure of compound **4**. The distance between Te1...19 is 3.5710 (1) Å. Symmetry code: #1: 1 - x, y, 1 - z; #2: x, -y, z; #3: 1 - x, -y, 1 - z. Hydrogen atoms have been omitted for clarity.

includes one chain of polyiodides formed by secondary interactions between I–I···I–I, as in I3–I2···I4–I5:3.6993 (2) Å.

In the solid state, compound $[C_{17}H_{16}IOTe]I(5)$ assumes a structure similar to that of the compound $[C_{16}H_{14}ITe]I(2)$. The forma-



Fig. 5. A pseudodimer of compound **5** was formed through Te \cdots I secondary interactions (represented by dashed lines). Symmetry code: #1: 1 - x, -y, 2 - z. Hydrogen atoms have been omitted for clarity.

tion of a pseudodimer occurs with secondary intermolecular interactions between Te1...l2, Te1#1...l2#1 {3.3791 (1) Å} and Te1...l2#1, Te1#1...l2 {3.7961 (1) Å}.

In addition to these interactions, the pseudodimer **5** forms two more intramolecular interactions between $Te1\cdots O1$ and $Te1#1\cdots O1#1$ {3.1215 (1) Å}. The secondary interactions around the Te(IV) atom induces the adoption of a distorted octahedral geometry (Fig. 5).

Fig. 6 shows the one-dimensional assembly of complex **6** in the *ab* plane. This pseudopolymer is formed by inter and intramolecular secondary interactions that connect the $[C_{17}H_{16}IOTe]^+$ cation unit to the I_3^- anion. In this complex, the Te(IV) atom assumes a distorted octahedral geometry in the presence of the secondary interactions. The distances of the various interactions are Te1...01: 3.2427 (1) Å, Te1...I2#1: 3.7539 (1) Å, and Te1...I4#2: 3.5020 (1) Å.



Fig. 4. Representation of the three-dimensional chain formed by the compound **4** through Te···I and I-I···I-I secondary interactions. Symmetry code: #1: x, y, -1 + z; #2: 1 - x; y; 1 - z; #3: 1 - x; -y; 1 - z; #4: x, -y, z. For clarity, the hydrogen atoms are not shown.



Fig. 6. DIAMOND [30] representation of the unidimensional assembly of complex **6** in the *ab* plane. Symmetry code: #1: *x*, -1 + *y*, *z*; #2: -*x*, -0.5 + *y*, 1.5 - *z*. For clarity, the hydrogen atoms are not shown.

4. Conclusions

In this work, the dihydrotellurophenium iodides **2–6** were synthesized in good yields by the electrophilic cyclization of homopropargyl tellurides using I_2 as an electrophile. The dihydrotellur-ophenium iodides **2–6** are ionic compounds bearing a cationic tellurium atom with an oxidation state of +4. The molecular iodine played a crucial role in the formation of different products and, depending on the stoichiometry of the iodine, different molecular arrangements were observed. In this way 1 equiv. I_2 yielded compounds **2** and **5**, which formed a dimeric chain in the solid state with I^- as counterions. The use of more than 1 equiv. I_2 induced the formation of polyiodides, such as the I_3^- anion (compounds **3**, **4**, and **6**). The synthesized compounds form, in the solid state, inter and/or intramolecular secondary interactions between Te···I and/ or I···I, and these interactions are responsible for the formation of various supramolecular assemblies.

Acknowledgments

This work was supported with funds from the Brazilian agencies CNPq, CAPES and FAPERGS.

Appendix A. Supplementary data

CCDC 967560–967564 contains the supplementary crystallographic data obtained for compounds **2–6**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/ retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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