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Cyclodimerization of phenyliodoacetylene with elemental tellurium: New pathway to 1.3-ditellurofulvenes

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1. Introduction

Treatment of $p-RC_6H_4C \equiv C^-Na^+$ (R=H, Me) with Te powder followed by acidification with etherial HCl (Scheme 1) to give 1,3-ditellurofulvene (I), as first reported in 1979 [1], led to extensive investigations of this and related reactions.

Re-investigation of the synthesis, conducted in 1981 [1], recognized the major product as a *cis/trans-* mixture of 1,3-ditelluretanes (II and III) but not 1,3-ditellurofulvene I (Scheme 2).

Both isomers were separated, and the *cis*-isomer (**II**) has been found to undergo *cis-trans* isomerisation under the action of acidic catalyst or UV-irradiation. However, only the *trans*-isomer (**III**) was characterized by X-ray diffraction. In the same year, another research group supported the structural proof for trans-1,3-ditelluretane (**III**) as a major product and separated the additional minor byproduct which was structurally characterized as 1,2,4-tritellurolane (**IV**) (Scheme 2) [2]. It must be mentioned that 2,4-di-*tert*-butyl-1,3ditelluretane (obtained via transient telluroaldehyde in the reaction of vanadium alkylidene complex $[(N(2-P^iPr_2-4-methylphenyl))_2V$ (CHBu)₂] with an excess of elemental Te) was recently shown to

ABSTRACT

Thermal reaction between PhC=CI and powdered Te afforded a mixture of (*E*)-4-iodo-2-(iodo(phenyl)-5-phenyl-1,3-ditellurofulvene (**1**) and (*Z*)-4-iodo-2-(iodo(phenyl)-5-phenyl-1-(diiodo),3-ditellurofulvene (**2**), which was subsequently reduced to (*Z*)-4-iodo-2-(iodo(phenyl)-5-phenyl-1,3-ditellurofulvene (**3**). Formation of **1** and **3** as the thermodynamically most stable products has been rationalized using density functional theory (DFT) calculations. Molecular structures of **1**–**3** were established crystallographically. In the solid state the coordination sphere of both tellurium atoms in **2** is extended by weak intermolecular Te··· π interactions with the solvate molecule of toluene which completes the pseudo-trigonal bipyramidal coordination geometry around each Te atom and assembles **2** into the chains along the crystallographic *c*-axis.

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convert to 3,5-di-*tert*-butyl-1,2,4-tritellurolane on treatment with an excess of elemental tellurium [3] (Scheme 3).

Substitution of etherial HCl by CF_3CO_2H as a protonating agent for PhC \equiv CTeNa resulted in *cis/trans* – mixture of 1,3-ditelluretanes (**II**–**III**) along with a minor quantity of *E*-1,3-ditellerofulvene (**I**) (Scheme 4) [4].

Selective preparation of either 1,3-ditellurofulvene or the 1,3-ditelluretane derivatives controlled by the conditions of ethynyltellurolate anion protonation has also been developed (Scheme 5) [5].

Due to the known property of unstable tellurocarbonic species to cyclodimerize to give 1,3-ditelluretanes (Scheme 6) [3,6–8], telluroketene was assumed to be an intermediate precursor of 1,3-ditelluretane, but no precursors or intermediates were suggested for 1,3-ditellurofulvene in (Scheme 6) [5].

Treatment of phenylacetylene with tellurium under strong basic conditions, investigated by Potapov et al. [9] and subsequently reinvestigated by the Wudl group [10] afforded 1:1 *E*/*Z* mixture of 1,3-ditellurafulvenes (I) (Scheme 7).

Interestingly, these findings stimulated the preparation of hexamethylenetetratellurafulvalene (HMTTeF) [11] which was the first example of a tetratellurofulvalene (TTeF); assumed instability of such compounds thought to occur due to very large-sized tellurium atoms incorporated in a five-membered ring. At the same time, the





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Scheme 1. First report on 1,3-ditellurofulvene formation by PhC2TeNa acidification.

TTeFs were expected to be better conducting materials than tetrathiafulvalenes (TTF) due to more diffuse nature of tellurium atomic orbitals and consequent increase of secondary intermolecular interactions in the crystal lattice, resulting in materials with enhanced metallic electrical conductivity, as was subsequently shown experimentally [12].

2. Results and discussion

In the course of our investigations on the interaction of organic telluro-halides with acetylenes and metal–carbonyl complexes [13] we intended to prepare ($PhC \equiv C$)₂TeI₂ following the Te into C–I bond insertion method known for alkyliodides (Scheme 8) [14].

No reaction was observed while stirring PhC=CI with Te powder in toluene at room temperature, but subsequent refluxing of the reaction mixture for 6 h yielded a mixture of (*E*)-4-iodo-2-(iodo(phenyl))-5-phenyl-1,3-ditellurofulvene (**1**), and (*Z*)-4-iodo-2-(iodo(phenyl))-5-phenyl-1(diiodo),3- ditellurofulvene (**2**). The two isomers were separated by chromatographic techniques as orange-red air stable solids. Reduction of **2** by aqueous Na₂S₂O₃ produced the *cis*-isomer of **1**: (*Z*)-4-iodo-2-(iodo(phenyl))-5-phenyl-1,3-ditellurofulvene (**3**) (Scheme 8).

Crystal structure investigation revealed that molecules of **1** and **2** have a centre of inversion and *m*-plane symmetry elements accordingly. Both have a planar 1,3-ditellurofulvene core having two pairs of iodo and phenyl groups oriented in the *trans* (**1**) and *cis* (**2**) configuration relative to it. Each pair or vinylidenic carbon atoms in **1** and **2** are disordered over four positions with equal occupancy so that cyclic or *exo*cyclic double bonds could not be unambiguously distinguished within one molecule (Scheme 9). Based on X-ray data we can assume that disorder may arise from a "juxtaposure" of symmetrical 4-membered (2E,4E)-2,4-bis (iodo(phenyl))-1,3-ditelluretane (Scheme 9, **a**) and 6-membered 2,5-diiodo-3,6-diphenyl-1,4-ditellurine (Scheme 9, **b**) molecules

or asymmetrical 5-membered ((*E*)-4-iodo-2-(iodo(phenyl))-5-phenyl-1,3-ditellurofulvene) molecules (Scheme 9, **c** and **d**).

Known examples of 6-membered 1,4-ditellurines structures typically exhibit non-planar geometry of a heterocyclic ring folded along the Te–Te line with a dihedral angle of approximately 60° [15,16]. Therefore, 1,3-ditellurofulvene appears to be the most rational solution. In fact, XRD investigation of **3**, which exhibits much less disorder in the positions of vinylidenic carbon atoms, has undoubtedly revealed its (*Z*)-4-iodo-2-(iodo(phenyl))-5-phenyl-1,3-ditellurofulvene structure (Fig. 3). By analogy, the same 1,3-ditellurofulvene core was assigned to **1** and **2** (Figs. 1 and 2).

Intramolecular bond lengths and bond angles as well as the slight deviations from planar geometry in the 1,3-ditellurole ring of **1–3** are in good agreement with those in 1,3-ditellurole [17] and TTeFs [18,19]. The crystal packing of **1** follows the same pattern as observed in TTeFs and short intermolecular Te···Te (1 + x, y, 1 + z) interaction (3.881 Å) assembles the molecules of **1** into the plain chains (see Fig. 4) *zigzag* packed along the crystallographic *b*-axis, which appears to be the longest dimension of unit cell of **1** (a = 5.7173(3) Å, b = 23.4033(10) Å, c = 6.8514(4) Å). All relevant crystallographic details for **1–3** are given as Supplementary material.

In contrast to the Te-diiodinated TTeF [19], characteristic intermolecular Te···I interactions are absent in the Te-diiodinated 1,3-ditellurofulvene **2**. Instead of iodine atom, the coordination sphere of tellurium is extended by weak intermolecular Te1··· π interactions with the solvate molecule of toluene, which occurs at a distance of 3.462 Å from Te1 and is oriented in the same direction as the Te1 lone electron pair, thus completing the pseudo-trigonal bipyramidal coordination geometry around the Te centre. Literally, *per contra* toluene molecule is faces the non-iodinated Te2 atom of the neighboring molecule of **2**(*x*, *y*, 1 + *z*), occurring at a distance of 3.405 Å from the Te2 and associating molecules of **2** into the chains along the crystallographic *c*-axis (Fig. 5). This gives a clear evidence of rare Te··· π -arene weak interaction [20].

Only 3 structures containing Te… π -ring interaction with a solvate arene (toluene) molecule are reported [21–23] (see Fig. 6), thus "multidecker" chain-like Te… π association (normally observed in metal-cyclopentadienyl solid state structures (i.e. Cp₂Pb, Cp₂Pb*C₅H₆ [24], (η_1 -Cp)SbCl₂ [25])) formed in the crystal of **2** is unique.



Scheme 2. Re-investigation of the first report on PhC2TeNa acidification [2].



Scheme 3. Formation of 1,3-ditelluretane via cyclodimerization of transient telluroaldehyde and its further convertion to 1,2,4-tritellurolane [3].





Scheme 5. Controlled formation of 1,3-ditellurane and 1,3-ditellurofulvene by RC₂Te anion acidification under various conditions [5].



Scheme 6. Formation of 4-membered 1,3-ditelluretanes via cyclodimerization of unstable telluroketones and telluroaldehydes [3,6-8].

It must be noted that despite the fact that the plane of a toluene molecule occurs at almost equal distances (3.462-3.405 Å) from the neighboring Te(IV) and Te (II) atoms, inclusion of toluene was not observed in the crystals of **1**, which was crystallized from the same mother-liquor as **2**. Crystals of **2** are air stable and even after 1 month storage in an open sample tube did not suffer the



Scheme 7. Formation of 1,3-ditellurafulvenes on treatment of phenylacetylene with elemental Te under strong basic conditions [9–10].

destruction usually observed in crystallosolvates, and had preserved their color, habit and X-ray diffracting properties.

There was no observable reaction between Te and 1,2-diodoacetylene or 1-iodo-propyn-3-ol, under the same conditions as those used for the phenylacetylene reaction, and in both cases there was no significant change in the color of the reaction mixture, and the Te was recovered almost quantitatively. In the reaction of Se powder with phenylacetylene in refluxing toluene, formation of elemental iodine vapors and blackish resin-like material insoluble in common organic solvents was observed.

Recent quantum chemical studies of the reaction of SeCl₂ with $(H_2C=CH)_2S$ have demonstrated that 6-membered thiaselenane tends to rearrange into 5-membered thiaselenolane due to the higher thermodynamic stability of the latter (see Scheme 10) [26].

We can therefore assume a similar rearrangement of intermediate 6-membered 2,5-diiodo-3,6-diphenyl-1,4-ditellurine (arising as a result of transient phenylethynyl tellurenyliodide



Scheme 8. Formation of 1,3-ditellurofulvenes 1 and 2 on thermal interaction of phenyliodoacetylene with elemental Te.



Scheme 9. Vinylydenic carbon atoms disorder in the solid state structure of 1 (showing thermal ellipsoids at the 50% probability level) and its possible interpretation as "jux-taposure" of a and b or c and d.



Fig. 1. Molecular structure of (E)-4-iodo-2-(iodo(phenyl)methylene)-5-phenyl-1,3ditellurole (**1**) showing thermal ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity.



Fig. 2. Molecular structure of (*Z*)-4-iodo-2-(iodo(phenyl)methylene)-5-phenyl-1 (diiodo),3-ditellurole (2) showing thermal ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity. Selected bond distances (Å): Te1-C2 2.058(11), Te1-C1'A 2.128(12), Te1-I1 2.9190(6), Te1-I2 2.9476(6), Te2-C1 2.110), Te2-C1'A 2.119 (12), C1-C2 1.320(15), C1'A-C2'A 1.309(14) and bond angles(°): C2-Te1-C1'A 9.31(5), I(1)-Te(1)-I2 172.80(2), C1-Te2-C1A 112.4(7), C1-Te2-C1'A 9.0.(5), Te2-C1'A-Te1 112.6(5).

cyclodimerization) into the 5-membered ((E)-4-iodo-2-(iodo (phenyl))-5-phenyl-1,3-ditellurofulvene) (Scheme 11). Addition of PhTeBr to acetylenes has been reported earlier to afford an E/Z mixture of bromo-vinylic tellurides in favor of the E isomer [27].

Our DFT quantum calculations for (*E*)-1,3-detellurofulvene **1** taken as zero, *Z*-1,3-detellurofulvene **3**, appropriate 6-membered *E*/*Z*-2,5-diiodo-1,4-ditellurines, and H-substituted model compounds at PBE0/LANL2DZ(d) level have demonstrated higher thermodynamic stability of 1,3-detellurofulvene 5-membered structure as compared to 4- and 6-membered heterocycles **1a**–**b**, **3a**–**b** and particularly, slightly higher thermodynamic stability of **1** as compared to *Z*-isomer **3** (see Table 2).

Accordingly, DFT calculations at the same level for Te-diodinated Z-1,3-detellurofulvene **2** taken as zero, demonstrated its higher thermodynamical stability among the all possible isomers 2a-c (see Table 3).



Fig. 3. Molecular structure of(*Z*)-4-iodo-2-(iodo(phenyl)methylene)-5-phenyl-1,3ditellurole (**3**) showing thermal ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity. Selected bond distances (Å): Te2-C2 2.110(5), Te2-C4 2.110(5), Te1-C5 2.112(5), Te1-C2 2.113(5), C1-C2 1.312(8), C4-C5 1.317(7) and bond angles (°): C2-Te2-C4 89.9(2), C5-Te1-C2 93.4(2), Te2-C2-Te1 113.0(2).



Fig. 4. Packing pattern for **1**. Intermol. d(Te1A-Te1(1 + x, y, 1 + z)) 3.881 Å (thermal ellipsoids at 50% probability level).



Fig. 5. 2-toluene-2 chains along the crystallographic *c*-axis. Intermolecular interaction distances (Å): Te1…toluene (x, y, z) C₆-centroid 3.462, toluene C₆-centroid…Te2(x, y, 1 + z) 3.405 (thermal ellipsoids at the 50% probability level).

3. Experimental section

3.1. *General procedure*

All reactions and manipulations were performed using standard Schlenk line techniques under an inert atmosphere of purified nitrogen. Solvents were purified, dried, and distilled under an argon atmosphere prior to use. Infrared spectra for **1**–**3** were recorded on a Nicolet 380 FT-IR spectrophotometer in a KBr pellets and NMR spectra were recorded on aBruker Avanceiii 400 spectrometer in DMSO. Commercial Te powder was used without further activation

or purification. TLC plates were purchased from Merck (20×20 cm silica gel 60 F₂₅₄). Phenyliodoacetylene was prepared following reported procedure [28].

3.2. Preparation of 1 and 2

A solution of phenyliodoacetylene 0.46 g (\sim 2 mmol) was refluxed in toluene (10 ml) with 0.25 g (\sim 2 mmol) of finely ground tellurium for 8 h under constant stirring on an oil-bath. Deep-red reaction mixture was allowed to cool down slowly along with the oil-bath without stirring to room temperature. Red-orange



Fig. 6. Toluene C₆- centroid…Te distances: 3.518 Å in TeSb₂Cl₂(N^tBu)₄ [21]. 2.981 Å in [TeF₄]_n [22], 3.319 Å in [Te₁₁N₆Cl₂₆] [23].



Scheme 10. Rearrangement of transient 6-membered thiaselenane into thermodynamically more stable 5-membered thiaselenolane [26].

Concentration of the ethylacetate eluate with 20 ml of hexane gave **2**. Yield: 0.35 g (0.34 mmol), M.p. 118–122 °C, IR (KBr, cm⁻¹): 1731.93(vs), 1592.42(w), 1572.64(w), 1480.94(s), 1437.94(s), ¹H NMR (δ , ppm): 7.941–7.340, 2.49 (CH₃ of toluene) ¹³C NMR (δ , ppm): 29.081(CH₃ of toluene), 80.76, 83.61, 121.78, 128.66 to 133.35 (Ph), 135.317.

For the preparation of **3**, the ethylacetate fraction was shaken

with 10% aqueous sodium thiosulphate (100 ml) and subsequently

extracted with dichloromethane (3 \times 50 ml), washed with brine

and dried over anhydrous sodium sulphate. Further concentration

of combined dichloromethane extracts with 50 ml of hexane and

cooling at -10 °C overnight afforded **3** as orange-red crystals. Yield:

(s), 1438.10(w), ¹H NMR (δ, ppm): 7.924–7.335, ¹³C NMR (δ, ppm): 80.67, 92.51, 122.963, 122.932, 128.100 to 133.213 (Ph), 135.024,

135.118. Mass spectrum of 3 shows molecular ion peaks centered at

m/e = 713.67 (¹³⁰Te) having the expected isotope pattern for Te₂.

M.p. 105–110 °C, IR (KBr, cm⁻¹): 1588.27(w), 1572.64(w), 1480.05



Scheme 11. Possible mechanism of 1,3-detellurofulvene 1 formation.

3.3. Preparation of 3

0.2 g (0.28 mmol).

crystalline precipitate was separated (0.63 g); its investigation under the microscope showed that it consists of two types of wellformed crystals: orange plates of **1** and deep-red prisms of **2** which were used for single-crystal XRD analysis. The mother-liquor which showed two orange bands on TLC (eluted with dichloromethane/ hexane 1:1 v/v) which were matching with crystalline samples of **1** and **2** from the first (precipitate) crop was evaporated to dryness and separated by means of flash chromatography giving two fractions after subsequent development with hexane and ethylacetate. Concentration of the hexane eluate on the rotavapour gave **1**. Yield: 0.27 g (0.38 mmol) (37.7%), M.p. 124–126 °C, IR (KBr, cm⁻¹): 1481.13, 1438.13(s), 1570.33(w), 1547.39 (w), ¹H NMR (δ , ppm): 7.950–7.316, ¹³C NMR (δ , ppm): 80.70, 92.46, 122.981, 122.944, 135.156, 135.057, 128.13 to 133.82 (Ph).

Table 1

Crystal data and structure refinement for 1-3.

Identification code	1	2	3
Empirical formula	C ₁₆ H ₁₀ I ₂ Te ₂	C ₂₃ H ₁₈ I ₄ Te ₂	C ₁₆ H ₁₀ I ₂ Te ₂
Formula weight	711.24	1057.17	711.24
Temperature, K	293 (2)	150 (2)	293 (2)
Wavelength		Mo (K _{α} λ = 0.71073 Å)	
Crystal system	Monoclinic	Orthorhombic	Monoclinic
Space group	P21/n	Pmn2 (1)	C2/c
a, Å	5.7173 (3)	13.3648 (5)	25.0469 (7)
b, Å	23.4033 (10)	9.6185 (4)	10.2750 (2)
<i>c</i> , Å	6.8514 (4)	10.3957 (4)	14.5386 (4)
β , °	110.241 (6)	90.	113.273 (3)
Volume, Å ³	860.13 (8)	1336.36 (9)	3437.16 (15)
Ζ	2	2	8
Density (calculated), Mg/m ³	2.746	2.627	2.749
Absorption coefficient, mm ⁻¹	6.969	6.812	6.976
F(000)	632	944	2528
Crystal size, mm ³	$0.33\times0.26\times0.21$	$0.09\times0.05\times0.04$	$0.20 \times 0.10 \times 0.07$
θ range for data collection	3.29–29.00°.	3.26–29°.	3.45–29.00°.
Index ranges	$-7 \le h \le 7, -31 \le k \le 31, -9 \le l \le 9$	$-16 \le h \le 18, -11 \le k \le 13, -14 \le l \le 13$	$-34 \le h \le 34, -14 \le k \le 13, -19 \le l \le 11$
Reflections collected	7827	12839	15240
Independent reflections	2265 $[R_{(int)} = 0.0523]$	3567 [$R_{(int)} = 0.0412$]	4544 [$R_{(int)} = 0.0279$]
Completeness to $ heta=29^\circ$	99.0%	99.6%	99.5%
Absorption correction		Semi-empirical from equivalents	
Data/restraints/parameters	2265/0/107	3567/1/130	4544/0/181
Refinement method		Full-matrix least-squares on <i>F</i> ²	
Goodness-of-fit on <i>F</i> ²	0.818	1.031	1.350
Final R indices [I > 2sigma(I)]	R1 = 0.0296	R1 = 0.0260	R1 = 0.0309
	wR2 = 0.0406	wR2 = 0.0546	wR2 = 0.0733
R indices (all data)	R1 = 0.0566	R1 = 0.0293	R1 = 0.0352
	wR2 = 0.0426	wR2 = 0.0553	wR2 = 0.0745
Largest diff. peak and hole	1.280 and -0.901 e Å ⁻³	1.012 and -0.904 e Å ⁻³	2.117 and –1.029 e Å ⁻³

Table 2

Relative potential energies, ZPVE corrected values () and relative standard enthalpies [] for 1, 3 and isomeric 6- and 4-membered heterocycles (in kcal/mol).



Table 3

Relative potential energies, ZPVE corrected values () and relative standard enthalpies [] for H-substituted model compound for 2 and its isomers (R=H, in kcal/mol).



3.4. Crystal structure determination of 1–3

Relevant crystallographic data and details of measurements are given in Table 1. X-ray crystallographic data were collected from single-crystal samples of **1–3**, mounted on an Oxford Diffraction XCALIBUR-S CCD system equipped with graphite-monochromated MoK α radiation (0.71070 Å). The data were collected by the ω -2 θ scan mode, and absorption correction was applied by using multiscan. The structure was solved by direct methods (SHELXS-97) and refined by full-matrix least-squares against F^2 using SHELXL-97 software [29]. Occupancies of the disordered carbon atom positions in **1** were chosen so that as to satisfy the equality of according isotropic displacement parameter (U_{iso}) values. However this was not possible for the closely located phenyl carbons C(5)–C(8). Subsequent anisotropic refinement of positions of vinylydenic carbons C(1-1'a)-C(2-2'a) was stable so that constraining of appropriate anisotropic displacement factors was not required. The same procedure was used for the treatment of disorder of carbon atom positions in **2**. Non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogen atoms were geometrically fixed and refined using a riding model.

3.5. Computational details

Density functional theory (DFT)calculations of **1–3** and model compounds (**1a–b**, **2a–c**, and **3a–b**) were performed with PBE hybrid exchange-correlation functional [30] using double- ξ valence basis set [31,32] augmented with one polarization d-function for main group elements [33] and one p-function for hydrogen atoms [31]. The Hay and Wadt effective core potential (ECP) were used for

Te and I atoms [32]. Geometry optimization was performed without constraints and optimized geometries were proven to be true minimum by the Hessian matrix calculation. All calculations were performed by using GAMESS-US [34] program package.

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Appendix. Supplementary data

CCDC 771931 (1), CCDC 771932 (2), CCDC 771933 (3); contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk.

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