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Reaction of Tellurium Tetraiodide with 2,3-Dihydro-1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene

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ABSTRACT: *2,3-Dihydro-1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene* **1** *(Carb, $R^1 = {}^iPr$, $R^2 = Me$) reacts with TeI_4 to give the carbene adduct $CarbTeI_2$ (***3***). The crystal structure of* **3** *consists of T-shaped monomeric fragments linked by weak Te. I interactions to form infinite helical chains.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:316–319, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20090

INTRODUCTION

2,3-Dihydroimidazol-2-ylidenes (**1**) may react with nonmetal compounds by reduction or coordination [1]. With sulfur halides, both reaction pathways have been observed [2]. We therefore have been interested in the reaction of **1** ($R^1 = {}^iPr$, $R^2 = Me$) with tellurium tetrahalides (see Scheme 1).

RESULTS

We obtained the carbene adduct **3** from the reaction of 2,3-dihydro-1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene [3] (**1**, $R^1 = {}^iPr$, $R^2 = Me$) with TeI_4 as the sole product. Apparently, **3** is formed by iodine abstraction from the tellurium(IV) derivative **2** formed as an intermediate. Only on prolonged heating of the reaction mixture, a subsequent reaction of **3** with iodine to give the salt **4** [4] is observed. Thus, the initial reduction of TeI_4 by the carbene can be excluded.

The crystal structure analysis of **3** (Tables 1–3) reveals the presence of monomeric fragments (Fig. 1) connected by weak intermolecular Te···I interactions (Fig. 2) which similarly have been found in ionic triorganotellurium iodides (3.6–3.9 Å [5]). Both the structure of the monomeric unit [Te(1)—I(1) 2.945(1), Te(1)—I(2) 2.933(1), Te(1)—C(1) 2.105(4) Å; I(1)—Te(1)—I(2) 169.41(1), I(1)—Te(1)—C(1) 84.8(1), I(2)—Te(1)—C(1) 84.7(1)°] as well as the intermolecular angles and distances of the helical chain [Te(1)—I(1A) 3.714(1) Å, Te(1)—I(1A)—Te(1A) 133.7(1), I(1)—Te(1)—I(1A) 96.8(1)°] resemble closely to those found for the dimeric carbene adduct **5** × ½ C_5H_5N reported by us previously [6]. It is not clear whether the differences in oligomerization come from the presence of solvent molecules or from the change of the substituents in 1,3-positions. The Te—I distances in **3** are closely related both to those found in $RTeI_2^-$ anions (ca. 2.9 Å [7]).

Reactions of **1** with $TeBr_4$ or $TeCl_4$ cause complicated mixtures that may contain products of the types **2–4**. Apparently, the higher reactivity of the halogens formed thereby does not allow to stop

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Dedicated to Professor Alfred Schmidpeter on the occasion of his 75th birthday.

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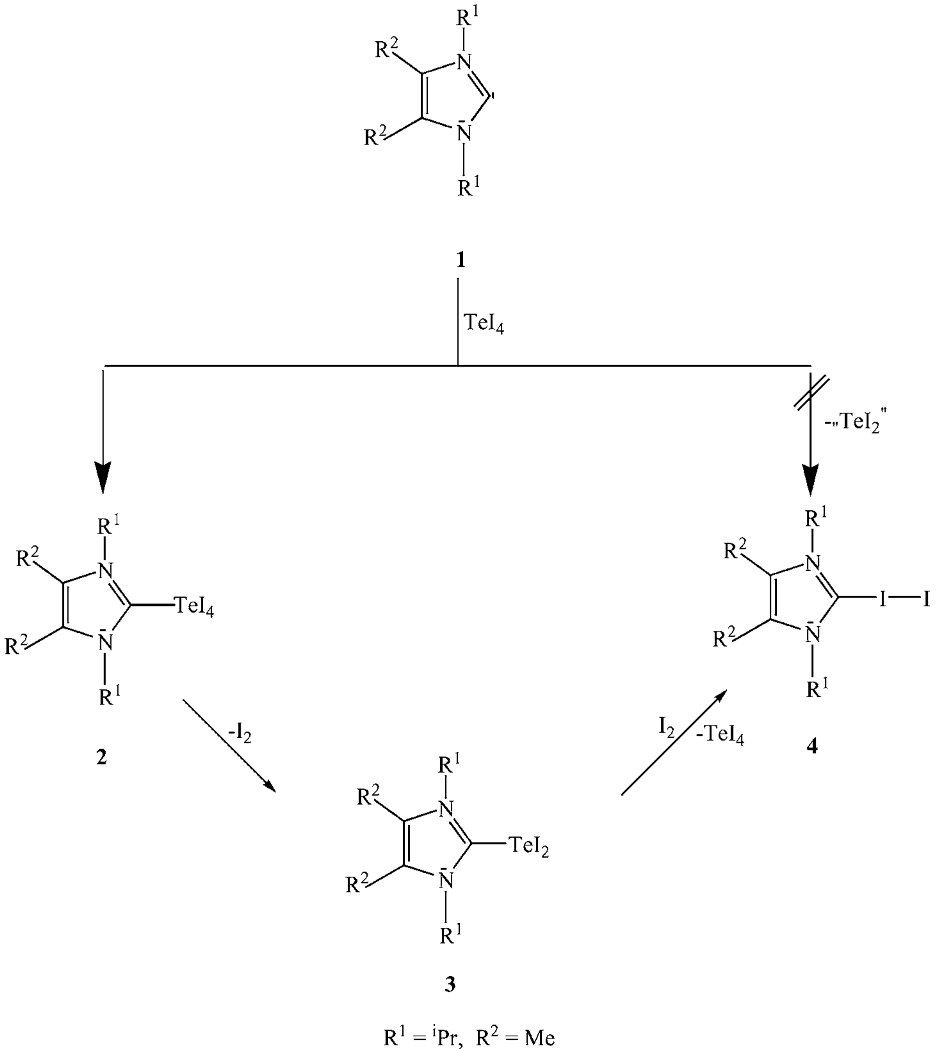
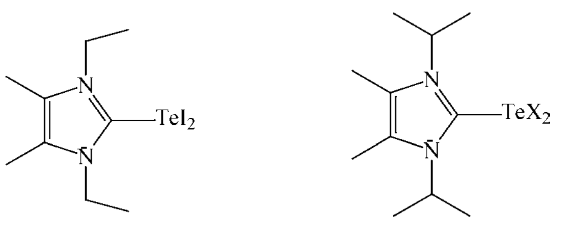


SCHEME 1

the reaction sequence after the formation of **6** ($X =$ Cl, Br) there (see Scheme 2).

EXPERIMENTAL

All reactions were performed under argon in purified solvents. 2,3-dihydro-1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene (**1**; $R^1 = {}^iPr$, $R^2 = Me$) has been obtained according to a literature procedure [3]. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre (CCDC-258609). Copies of this information may be obtained from the Director, CCDC, Union Road, Cambridge, CB2 1EZ, UK; Fax +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk.



5 **6**

SCHEME 2

*(2,3-Dihydro-1,3-diisoproypl-4,5-dimethylimidazol-2-ylidene)diiodotellurium (**3**)*

1.24 g (1.94 mmol) of TeI_4 was added to a solution of 0.35 g (1.94 mmol) **1** ($R^1 = {}^iPr$, $R^2 = Me$) in 30 mL of tetrahydrofuran. The resulting mixture was refluxed for 5 h, filtered. The precipitate was dissolved in cold dichloromethane, stirred for 2 h, filtered, and

TABLE 1 Data Collection and Processing Parameters for **3**

Empirical formula	$C_{11}H_{20}I_2N_2Te$
Formula weight	561.69
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2(1)/c$
Unit cell dimensions	$a = 14.574(4)$ Å $\alpha = 90°$.
	$b = 11.9536(19)$ Å $\beta = 91.21(3)°$.
	$c = 9.691(5)$ Å $\gamma = 90°$.
Volume	1687.8(10) Å^3
Z	4
Density (calculated)	2.210 mg/m^3
Absorption coefficient	5.403 mm^{-1}
$F(000)$	1032
Crystal size	$0.25 \times 0.5 \times 0.25$ mm^3
θ range for data collection	2.20–27.51°.
Index ranges	$-18 \leq h \leq 18, -15 \leq k \leq 15, -12 \leq l \leq 10$
Reflections collected	14574
Independent reflections	3886 [R(int) = 0.0396]
Completeness to $\theta = 27.51°$	100.0%
Absorption correction	Empirical
Max. and min. transmission	0.3018 and 0.2459
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3886/0/223
Goodness-of-fit on F^2	1.120
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0283$, $wR_2 = 0.0625$
R indices (all data)	$R_1 = 0.0359$, $wR_2 = 0.0687$
Extinction coefficient	0.00072(7)
Largest diff. peak and hole	1.486 and −1.529 e.Å^{-3}

evaporated to dryness. Yield after recrystallization from dichloromethane/diethyl ether: 0.72 g (66%), red crystals. Elemental analysis for $C_{11}H_{20}I_2N_2Te$: C 22.95 (calc. 23.53), H 3.43 (3.56), N 4.99 (4.63)%. ^{1}H-NMR ($CDCl_3$): $\underline{\Omega} = 1.62$ (d, 12 H, CH*Me*$_2$, $^3J = 7.0$ Hz), 2.34 (s, 6 H, 4,5-Me), 4.80 (sept, 2 H, *C*HMe$_2$). ^{13}C-NMR ($CDCl_3$): $\underline{\Omega} = 11.1$ (4,5-Me), 21.3 (CH*Me*$_2$), 53.43 (*C*HMe$_2$), 126.4 (C^2), 128.8 (C^{4,5}).

TABLE 2 Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å$^2 \times 10^3$) for **3**

	x	y	z	$U(eq)$
Te(1)	2356(1)	4071(1)	−496(1)	25(1)
I(1)	1913(1)	1976(1)	943(1)	48(1)
I(2)	2762(1)	6349(1)	−1427(1)	36(1)
N(1)	1721(2)	5180(3)	2201(3)	21(1)
N(2)	3188(2)	4859(3)	2320(3)	24(1)
C(1)	2432(3)	4767(3)	1499(4)	22(1)
C(2)	2957(3)	5329(4)	3556(4)	28(1)
C(3)	2035(3)	5531(3)	3486(4)	25(1)
C(4)	4117(3)	4488(4)	1907(5)	31(1)
C(5)	4779(4)	5459(5)	1903(6)	41(1)
C(6)	4439(4)	3487(5)	2755(8)	48(1)
C(7)	3598(4)	5554(6)	4739(5)	42(1)
C(8)	1463(4)	6007(5)	4572(5)	34(1)
C(9)	767(3)	5253(4)	1635(5)	27(1)
C(10)	468(4)	6461(5)	1480(7)	44(1)
C(11)	119(4)	4542(6)	2435(8)	50(2)

U(eq) is Defined as one third of the trace of the orthogonalized U^{ij} tensor.

TABLE 3 Bond Lengths (Å) and angles (°) for **3**

Te(1)–C(1)	2.105(4)	N(2)–C(4)	1.487(5)
Te(1)–I(2)	2.9333(6)	C(2)–C(3)	1.366(6)
Te(1)–I(1)	2.9446(7)	C(2)–C(7)	1.487(6)
N(1)–C(1)	1.346(5)	C(3)–C(8)	1.471(6)
N(1)–C(3)	1.382(5)	C(4)–C(5)	1.510(7)
N(1)–C(9)	1.486(5)	C(4)–C(6)	1.520(7)
N(2)–C(1)	1.350(5)	C(9)–C(11)	1.500(7)
N(2)–C(2)	1.372(5)	C(9)–C(10)	1.515(7)
C(1)–Te(1)–I(2)	84.74(11)		
C(1)–Te(1)–I(1)	84.79(11)		
I(2)–Te(1)–I(1)	169.412(14)		
C(1)–N(1)–C(3)	108.8(3)		
C(1)–N(1)–C(9)	124.0(3)		
C(3)–N(1)–C(9)	127.2(3)		
C(1)–N(2)–C(2)	109.6(3)		
C(1)–N(2)–C(4)	123.6(3)		
C(2)–N(2)–C(4)	126.8(3)		
N(2)–C(1)–N(1)	107.5(3)		
N(2)–C(1)–Te(1)	126.9(3)		
N(1)–C(1)–Te(1)	125.7(3)		
C(3)–C(2)–N(2)	106.7(4)		
C(3)–C(2)–C(7)	127.4(4)		
N(2)–C(2)–C(7)	125.8(4)		
C(2)–C(3)–N(1)	107.4(4)		
C(2)–C(3)–C(8)	127.2(4)		
N(1)–C(3)–C(8)	125.5(4)		
N(2)–C(4)–C(5)	110.9(4)		
N(2)–C(4)–C(6)	111.2(4)		
C(5)–C(4)–C(6)	114.6(4)		
N(1)–C(9)–C(11)	111.8(4)		
N(1)–C(9)–C(10)	111.0(4)		
C(11)–C(9)–C(10)	114.1(5)		

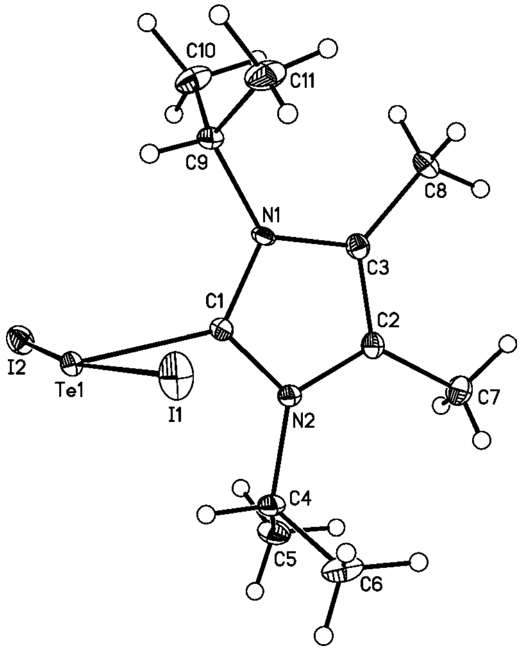


FIGURE 1 The structure of $C_{11}H_{20}N_2TeI_2$ (**3**) in the crystal.

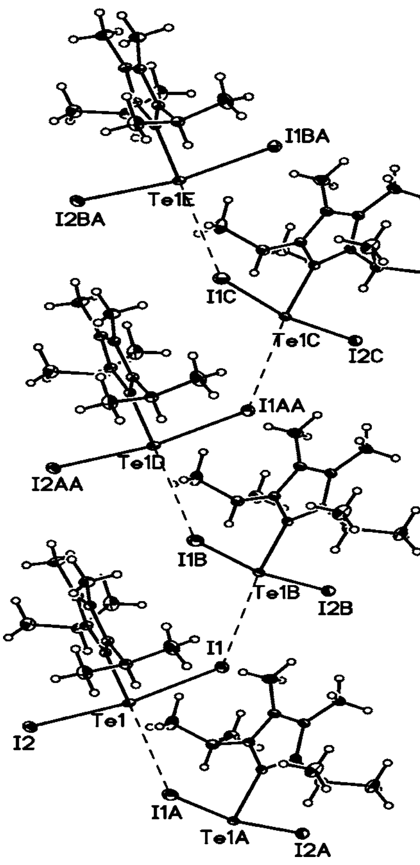


FIGURE 2 Molecular packing of (**3**) in the crystal.

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