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Inorganic Chemistry
Note

Pyramidal Inversion Isomers in the Solid-State Structure of the Tricyclic Antimony–Tellurium Imido Complex $TeSb_2Cl_2(N^tBu)_4$

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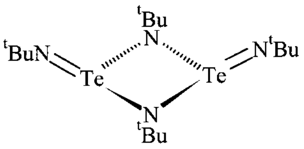
Introduction

In the past 6−7 years, there have been significant advances in the chemistry of imido systems involving either antimony[1−3] or tellurium.[4] The tellurium diimide dimer ^{t}BuNTe(μ-N^{t}Bu)$_2$TeN^{t}Bu (**1**) exists as the *cis*(*endo*,*endo*) isomer in the solid state,[5] although other isomers, for example, *trans*(*exo*,*exo*), are formed in complexes with metals.[6] The dimer **1** reacts readily with heteroallenes, for example, ^{t}BuNCO, via a cycloaddition process that generates the ureato telluroxide [OC(μ-N^{t}Bu)$_2$TeO]$_2$.[7] The dimer ClSb(μ-N^{t}Bu)$_2$SbCl (**2**) was reported more than 20 years ago.[8] Although the structure of **2** has not been established, Stahl et al. have recently shown that the derivatives XSb(μ-N^{t}Bu)$_2$SbX (X = O^{t}Bu, N$_3$), prepared by nucleophilic substitution reactions of **2**, adopt *trans* structures in the solid state.[9]



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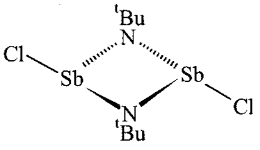

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Apart from the aforementioned investigation,[9] we are not aware of other reports of the reactions of **2**. In connection with our studies of the reactivity of **1**, we considered whether mixed antimony−tellurium imido systems could be generated from **1** and **2**. We report here that this reaction generates the tricyclic complex {[^{t}BuNSb(μ-N^{t}Bu)$_2$TeN^{t}Bu](μ_3-Sb-Cl)}$^+$Cl$^-$ (**3**) which, in the solid state, is obtained as a mixture of pyramidal inversion isomers. The structure of Cl$_2$Te(μ-N^{t}Bu)$_2$Sb[N(H)^{t}Bu] (**4**), a byproduct of this reaction, is also described.

Experimental Section

The reagents ^{t}BuNTe(μ-N^{t}Bu)$_2$TeN^{t}Bu (**1**),[10] {Li$_2$[Te(N^{t}Bu)$_3$]}$_2$,[11] and ClSb(μ-N^{t}Bu)$_2$SbCl (**2**)[12] were prepared by the literature procedures, and their purity was ascertained by ^{1}H NMR spectroscopy. An improved synthesis of the latter reagent has been described recently.[9] SbCl$_3$ (99.9%), Me$_3$SiN(H)^{t}Bu (98%), ^{t}BuNH$_2$ (98%), and ^{n}BuLi (2.5 M solution in hexanes) were obtained from Aldrich. ^{t}BuNH$_2$ and all solvents were dried with appropriate drying agents and distilled onto molecular sieves before use. All reactions were carried out under an argon atmosphere using standard Schlenk techniques or a drybox.

^{1}H NMR spectra were recorded on Bruker AC200 and AMX300 spectrometers, and chemical shifts are reported relative to Me$_4$Si in CDCl$_3$. ^{13}C and ^{125}Te spectra were measured at 25 °C in C$_6$D$_6$ or C$_4$D$_8$O on a Bruker AMX300 spectrometer using a 5 mm broadband probe (BBI) operating at 75.448 and 94.817 MHz, respectively. The spectral widths were 18.115 (^{13}C) and 98.039 kHz (^{125}Te) yielding the respective resolutions of 0.55 and 0.75 Hz/data point. The values for the 90° pulse widths (pw) were 7.80 (^{13}C) and 15.00 μs (^{125}Te). For the actual acquisitions, nuclear tip angles of 30° were used [pw(30°, ^{13}C), 2.57 μs; pw(30°,^{125}Te), 4.9 μs]. Relaxation delays of 3 and 1.5 s were applied when measuring the ^{13}C and ^{125}Te NMR spectra, respectively. The samples for ^{125}Te NMR were externally referenced to K$_2$TeO$_3$ in D$_2$O and referred to Me$_2$Te. Line-broadening parameters, used in the exponential multiplication of the free induction decays, were 10 to 1 Hz. Chemical shifts with a positive sign are correlated with shifts to high frequencies (downfield) of the reference compound. Elemental analyses were performed by Analytical Services, Chemistry Department, University of Calgary.

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Synthesis of {[^{t}BuNSb(μ-N^{t}Bu)$_2$TeN^{t}Bu](μ_3-SbCl)}$^+$Cl$^-$ (3). (a) From the Reaction of ^{t}BuNTe(μ-N^{t}Bu)$_2$TeN^{t}Bu and ClSb(μ-N^{t}Bu)$_2$SbCl. A red solution of **1** (0.300 g, 0.556 mmol) in toluene (10 mL) was added to a stirred pale yellow solution of **2** (0.508 g, 1.112 mmol) in toluene (15 mL) cooled to −78 °C. The resulting orange solution containing a yellow precipitate was warmed to 23 °C after 30 min to give a yellow-orange solution. After 5 h, the volatile materials were removed under vacuum. The pale orange solid was dissolved in THF (10 mL), and then *n*-pentane (5 mL) was added whereupon a yellow-orange precipitate of {[^{t}BuNSb(μ-N^{t}Bu)$_2$TeN^{t}Bu](μ_3-SbCl)}$^+$Cl$^-$ (**3**) (0.333 g, 0.458 mmol; 41%) was formed. X-ray quality crystals of **3** were obtained by overlaying a pale yellow solution of **2** (0.127 g, 0.120 mmol) in toluene (∼3 mL) with an orange-red solution of Te$_2$(N^{t}Bu)$_4$ (0.075 g, 0.132 mmol) in *n*-hexane (∼3 mL) in a test tube. Yellow and orange crystals were formed after 1 d at 23 °C. The orange crystals were identified as **3**. ^{1}H NMR [in C$_4$D$_8$O at 23 °C]: $\delta = 1.639/1.629$ [C(C*H*$_3$)$_3$, 36 H, ∼3:1 intensity ratio]. ^{13}C NMR [in C$_4$D$_8$O at 23 °C]: $\delta = 62.12$ and 61.06 [*C*(CH$_3$)$_3$], 35.77 and 33.11 [C(*C*H$_3$)$_3$]. ^{125}Te NMR [in C$_4$D$_8$O at 23 °C]: $\delta = 1637$. Anal. Calcd for **3**: C, 26.45; H, 4.99; N, 7.12. Found: C, 24.89; H, 5.16; N, 6.91. The yellow crystals were identified as Cl$_2$Te(μ-N^{t}Bu)$_2$Sb[N(H)^{t}Bu]·C$_7$H$_8$ (**4**·C$_7$H$_8$). ^{1}H NMR (in C$_6$D$_6$ at 23 °C): $\delta = 4.6$ [s, NH, 1 H], 1.40 [s, C(C*H*$_3$)$_3$, 18 H], 0.73 [s, C(C*H*$_3$)$_3$, 9 H]. ^{1}H NMR (in C$_4$D$_8$O at 23 °C): $\delta = 6.5$ [s, NH, 1 H], 1.50 [s, C(C*H*$_3$)$_3$, 18 H], 1.41 [s, C(C*H*$_3$)$_3$, 9 H], in addition to resonances attributable to toluene present in the lattice. ^{125}Te NMR (in C$_4$D$_8$O at 23 °C): $\delta = 1654$.

(b) From the Reaction of SbCl$_3$ and {Li$_2$[Te(N^{t}Bu)$_3$]}$_2$. A solution of SbCl$_3$ (0.321 g, 1.409 mmol) in toluene (10 mL) was added dropwise with stirring to a pale yellow solution of {Li$_2$[Te(N^{t}Bu)$_3$]}$_2$ (0.500 g, 0.704 mmol) in toluene (15 mL) at −78 °C. The reaction mixture was allowed to warm slowly to 23 °C and then filtered through a fine frit to remove LiCl and other insoluble products (0.275 g). Removal of solvent from the filtrate under vacuum gave a yellow-orange solid (0.510 g) which was extracted with *n*-hexane (13 mL) to give Li{Sb[Te(N^{t}Bu)$_3$]$_2$} (0.120 g, 0.148 mmol, 11%) as the soluble product (characterized by ^{1}H NMR).[13] The yellow hexane-insoluble product was identified by ^{1}H NMR as **3** (0.310 g, 0.404 mmol, 57%) contaminated with small amounts of **4**.

X-ray Crystallography. Intensity data for **3** and **4**·C$_7$H$_8$ were collected on a Bruker AXS SMART 1000 CCD diffractometer.

An orange crystal of **3** (0.40 × 0.22 × 0.06 mm^3) was coated in Paratone oil and mounted on a glass fiber. Using ϕ and ω scans of 0.3° per frame and exposure times of 20 s, a total of 13576 reflections were collected and then merged to provide 4994 data, of which 4692 were unique ($R_{\text{int}} = 0.0448$) and 4180 observed [$I > 2\sigma(I)$]. The ranges of indices were $-19 \le h \le 18$, $0 \le k \le 11$, and $0 \le l \le 20$, corresponding to $2.49 \le \theta \le 25.81°$. A final difference map revealed no features greater than 0.872 or less than −1.139 e Å^{-3}. An empirical absorption correction was applied.[14] The structure was solved by using direct methods[15] and refined by full-matrix least-squares method on F^2.[16] The asymmetric unit is composed of a disordered {[^{t}BuNSb(μ-N^{t}Bu)$_2$TeN^{t}Bu](μ_3-SbCl)}$^+$ cation with site occupancy factors of 0.779(2) and 0.221(2) represented by cations **A** and **B**, respectively. The two disordered parts were refined by constraining the thermal parameters of atoms in **A** to be equal to their counterparts in **B**. However, the positions of all the atoms were allowed to refine independently. The Cl$^-$ anion is not affected by the disorder. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included at geometrically idealized positions and were not refined. The isotropic thermal parameters of the hydrogen atoms were fixed at 1.5 times that of the attached carbon or nitrogen atom.

Table 1. Crystallographic Data for **3** and **4**·C$_7$H$_8$

	3	**4**·C$_7$H$_8$
empirical formula	C$_{16}$H$_{36}$Cl$_2$N$_4$Sb$_2$Te	C$_{19}$H$_{36}$N$_3$Cl$_2$SbTe
fw	726.49	626.76
space group	$P2_1/c$	$P2_1/n$
T (°C)	−80	−80
a, Å	16.150(2)	13.9127(17)
b, Å	9.4959(14)	11.7159(14)
c, Å	17.035(2)	15.8789(19)
β, deg	109.395(3)	99.693(2)
V, Å^3	2464.2(6)	2551.3(5)
Z	4	4
ρ, g cm^{-3}	1.958	1.632
λ, Å	0.71073	0.71073
μ, cm^{-1}	35.78	24.20
R1[a]	0.0301	0.0556
wR2[b] (all data)	0.0831	0.1579

[a] $\text{R1} = [\sum||F_o|-|F_c||]/[\sum|F_o|]$ for $[F_o{}^2 > 2\sigma(F_o{}^2)]$. [b] $\text{wR2} = \{[\sum w(F_o{}^2-F_c{}^2)^2]/[\sum w(F_o{}^2)^2]\}^{1/2}$ (all data).

A yellow platelike crystal of **4**·C$_7$H$_8$ (0.47 × 0.30 × 0.12 mm^3) was coated with Paratone oil and mounted on a glass fiber. Using ϕ and ω scans of 0.3° per frame for 15 s, 9674 reflections were collected of which 3630 were unique ($R_{\text{int}} = 0.0450$) and 2782 observed [$I \ge 2\sigma(I)$]. Structure solution and refinement were as described for **3** with the exception that the isotropic thermal parameters of the hydrogen atoms were fixed at 1.2 times that of the corresponding carbon or nitrogen atom. The atoms Sb(1) and N(3) were disordered over two sites with partial occupancy factors of 0.5159(17) and 0.4841(17). The atoms in the toluene molecule were restrained to be coplanar, and the C−C bond distances on opposite sides of the ring were restrained to be equal.

Crystallographic data for **3** and **4**·C$_7$H$_8$ are summarized in Table 1.

Results and Discussion

The reaction of **1** and **2** in toluene at 23 °C produced a moisture-sensitive yellow-orange solid identified as the tricyclic antimony−tellurium imido complex {[^{t}BuNSb(μ-N^{t}Bu)$_2$TeN^{t}Bu](μ_3-SbCl)}$^+$Cl$^-$ (**3**) by X-ray analysis (vide infra). The ^{1}H and ^{13}C NMR spectra of **3** in d_8-THF at 23 °C show only two N^{t}Bu environments, with an intensity ratio of 1:3, throughout the temperature range 233−300 K. During the crystallization process, a minor product, also characterized by X-ray analysis (vide infra), as Cl$_2$Te(μ-N^{t}Bu)$_2$Sb[N(H)^{t}Bu], was isolated. The ^{1}H NMR spectra of **4** in either d_6-benzene or d_8-THF at 23 °C showed two resonances for N^{t}Bu groups, with relative intensities 2:1, in addition to a broad resonance at δ 4.6 (C$_6$D$_6$) or δ 6.5 (C$_4$D$_8$O) attributable to an NH group.

The tricyclic complex **3** was also obtained as the major product (57%) from the reaction of SbCl$_3$ with {Li$_2$[Te(N-^{t}Bu)$_3$]}$_2$ in a 2:1 molar ratio in toluene. The minor product from this reaction was identified by ^{1}H NMR as the spirocyclic complex Li{Sb[Te(N^{t}Bu)$_3$]$_2$}, which is obtained

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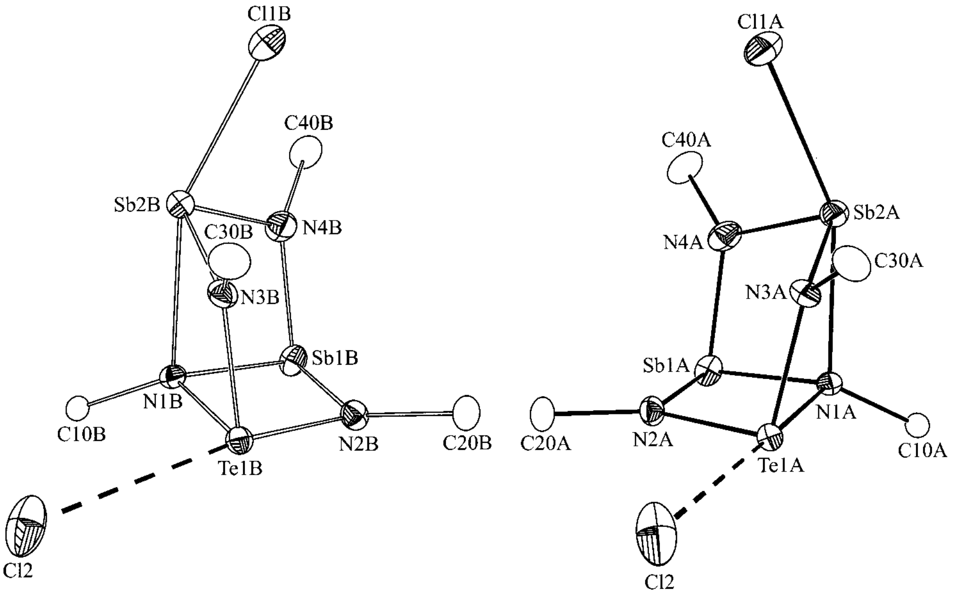
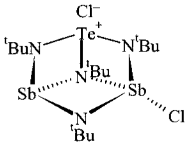


Figure 1. Thermal ellipsoid (30% probability) plot and atomic numbering scheme for **3** ($\mathbf{A}^+Cl^-$ and $\mathbf{B}^+Cl^-$). Only the α-carbon atoms of ${}^tBu$ groups are shown.

in optimum yields (57%) when the same reaction is carried out in a 1:1 molar ratio.[13]



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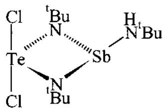

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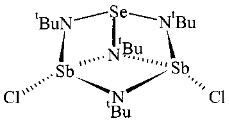

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The structure of **3** was determined by X-ray crystallography. The unit cell contains disordered $\{[{}^tBuNSb(\mu\text{-}N{}^tBu)_2TeN{}^tBu](\mu_3\text{-}SbCl)\}^+$ cations with approximate occupancy factors of 78% and 22% (represented by **A** and **B**), respectively, and Cl^- anions (see Figures 1 and 2). As indicated in Table 2, the Sb(2)−Cl(1) bond lengths in **A** and **B** are 2.547(4) and 2.493(13) Å, and the associated Sb(2)−N(1) distances are 2.440(4) and 2.462(13) Å (sum of covalent radii for Sb and Cl = 2.35 Å and for Sb and N = 2.06 Å).[17] For comparison, the related tricyclic compounds $[{}^tBuNP(\mu\text{-}NR)_2PN{}^tBu](\mu_3\text{-}SbCl)$ (R = ${}^tBu$,[18] Ph)[19] have d(Sb−Cl) = 2.492(3) and 2.439(1) Å, respectively. Stahl and co-workers have shown by VT ^1H NMR studies that complexes of the type $[RNP(\mu\text{-}NR)_2PNR](\mu_3\text{-}ECl)$ (where E = Sb, Bi) undergo an intramolecular pyramidal inversion process in solution (see Scheme 1).[19] The two forms of **3** (**A** and **B**) present in the crystal lattice are optical isomers related by pyramidal inversion at the Sb(2) center.

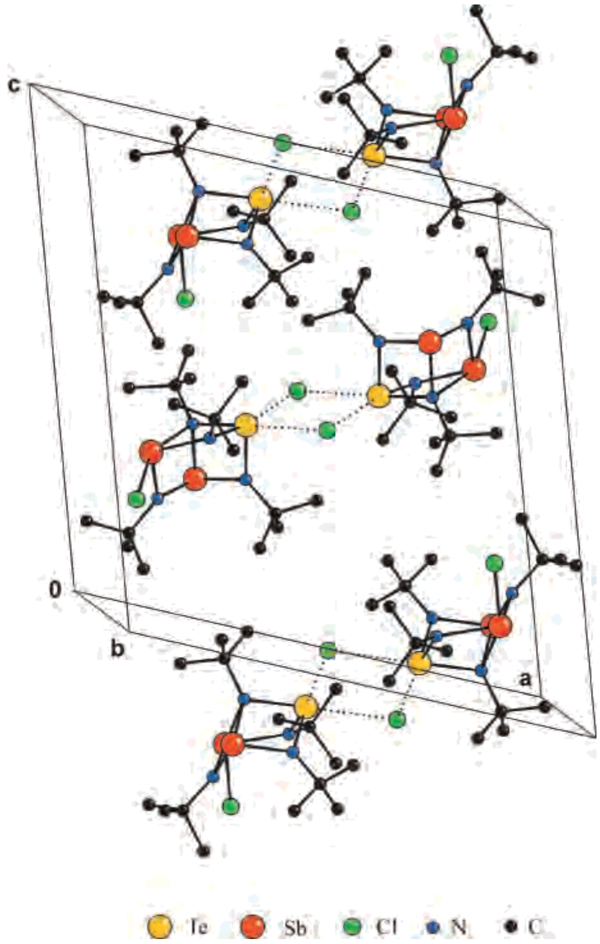

Figure 2. Unit cell of **3** ($\mathbf{A}^+Cl^-$) viewed down the b-axis showing the Te···Cl interactions.

The related tricyclic complex $SeSb_2Cl_2(N{}^tBu)_4$ (**5**), the selenium analogue of **3**, was prepared by the reaction of the selenium(II) reagent $Se[N{}^tBu(SiMe_3)]_2$ with $SbCl_3$.[20,21] The major difference between the structures of the isovalent congeners **3** and **5** involves the location of one of the chlorine atoms. The selenium complex **5** contains two Sb−Cl bonds [|d(Sb−Cl)| = 2.581(1) Å], whereas the tellurium complex **3** has only one Sb−Cl bond [2.547(4) Å in cation **A**] and a Cl^- counterion, which engages in weak interactions with the

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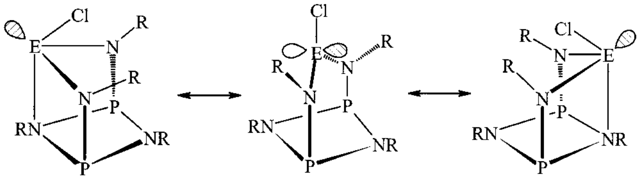
Table 2. Selected Bond Distances (Å) and Bond Angles (deg) in **3**

	cation **A**	cation **B**
	Bond Distances	
Te(1)−N(1)	2.098(7)	2.076(19)
Te(1)−N(2)	1.974(7)	1.987(19)
Te(1)−N(3)	1.991(5)	1.993(15)
Sb(1)−N(1)	2.073(6)	2.113(17)
Sb(1)−N(2)	2.046(6)	2.030(17)
Sb(1)−N(4)	2.014(6)	1.993(14)
Sb(2)−N(1)	2.440(4)	2.462(13)
Sb(2)−N(3)	2.087(6)	2.081(16)
Sb(2)−N(4)	2.072(8)	2.085(17)
Sb(2)−Cl(1)	2.547(4)	2.493(13)
Te(1)···Cl(2)	3.307(6)	3.055(22)
Te(1)···Cl(2)*[a]	3.198(5)	3.196(18)
	Bond Angles	
N(2)−Te(1)−N(1)	78.1(2)	78.3(6)
N(2)−Te(1)−N(3)	101.0(3)	99.1(12)
N(3)−Te(1)−N(1)	81.3(3)	82.7(9)
N(4)−Sb(1)−N(1)	82.4(3)	82.7(8)
N(4)−Sb(1)−N(2)	96.4(2)	99.6(11)
N(2)−Sb(1)−N(1)	77.05(19)	76.5(6)
Sb(1)−N(1)−Te(1)	99.7(3)	99.6(8)
Sb(1)−N(1)−Sb(2)	94.8(2)	93.3(6)
Te(1)−N(1)−Sb(2)	94.5(2)	94.1(7)
Te(1)−N(2)−Sb(1)	104.9(3)	105.6(9)
N(3)−Sb(2)−N(1)	71.62(19)	72.1(6)
N(4)−Sb(2)−N(1)	72.78(19)	72.8(6)
N(4)−Sb(2)−N(3)	109.3(2)	108.6(11)
N(1)−Sb(2)−Cl(1)	154.33(13)	155.1(5)
N(3)−Sb(2)−Cl(1)	92.50(19)	94.9(8)
N(4)−Sb(2)−Cl(1)	94.8(2)	92.4(8)
Te(1)−N(3)−Sb(2)	110.0(3)	109.7(8)
Sb(1)−N(4)−Sb(2)	109.2(3)	109.7(8)

[a] Symmetry transformations used to generate equivalent atoms. Asterisk indicates: $-x + 1, -y + 1, -z + 1$.

Scheme 1. Pyramidal Inversion in the Tricyclic Complexes [RNP(μ-NR)$_2$PR](μ-ECl) (E = Sb, Bi)



Te centers of two cations to form an asymmetric dimeric structure (Figure 2). The mean Te···Cl distances are $\sim$3.05 and 3.20 Å (cf., sum of van der Waals radii for Te and Cl of 3.81 Å[17] and 4.0 Å[22]). As a consequence of this ionization, there is a pronounced asymmetry in the Sb−N bonds involving the four-coordinate nitrogen atom, N(1), in **3** [2.073(6) and 2.440(4) Å in cation **A**, cf., 2.298(3) and 2.311(3) Å for the corresponding bond lengths in **5**[20]]. The ^{1}H NMR spectrum of **5** in CCl$_4$ shows three resonances for N^{t}Bu groups with relative intensities 1:1:2, consistent with the solid-state structure.[20] By contrast, four equally intense resonances are anticipated if the solid-state structure of **3** persists in solution. The observation of only two resonances in a 1:3 ratio (vide supra) suggests a more symmetrical structure for **3** in solution, possibly similar to that of **5**, with the overlap of the resonance for the two equivalent N^{t}Bu groups with that of one of the unique N^{t}Bu groups.

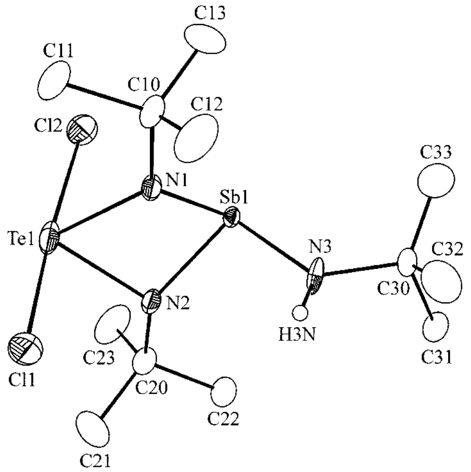


Figure 3. Thermal ellipsoid (30% probability) plot and atomic numbering scheme for **4**.

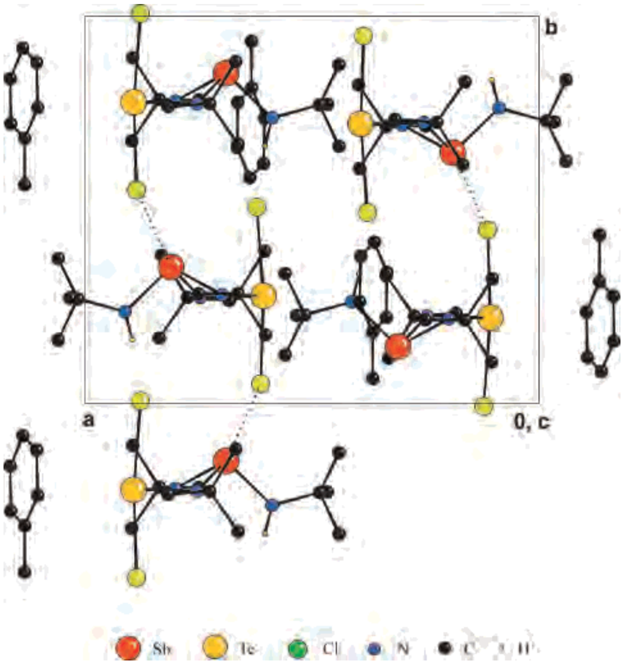


Figure 4. Unit cell of **4**·C$_7$H$_8$ viewed down the *c*-axis showing the Sb···Cl interactions. Only molecule **A** is shown.

The identity of the minor product from the reaction of **1** and **2** was determined by X-ray analysis to be Cl$_2$Te(μ-N^{t}Bu)$_2$Sb[N(H)^{t}Bu] (**4**), which involves chelation of the dianionic ligand [^{t}BuN(H)Sb(N^{t}Bu)$_2$]$^{2-}$ to a TeCl$_2$$^{2+}$ unit. Related bis-imido complexes of tellurium dichloride are either monomeric [(Ph$_3$PN)$_2$TeCl$_2$[23] and (Ph$_2$CN)$_2$TeCl$_2$[24]] or dimeric [(Me$_3$PN)$_2$TeCl$_2$[25]], or they form extended networks

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(21) The tricyclic imido-antimony complex [^{t}BuNSb(μ-N^{t}Bu)$_2$SbN^{t}Bu](μ_3-SbCl) (**7**) has been depicted to be isostructural with the cation in **3**, with which it is also isoelectronic, in a conference proceedings. Complex **7** is described as one of the products of SbCl$_3$ and ^{t}BuNH$_2$, but full details of the synthesis and X-ray structure have not been reported. Veith, M.; Rammo, A.; Hans, M. *Phosphorus, Sulfur Silicon Relat. Elem.* **1994**, *93−94*, 197.

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Table 3. Selected Bond Distances (Å) and Bond Angles (deg) in **4**·C_7H_8

Molecule **A**		Molecule **B**	
Bond Distances			
Sb(1)−N(2)	1.963(7)	Sb(1′)−N(3′)	1.901(18)
Sb(1)−N(3)	1.969(15)	Sb(1′)−N(1)	1.958(7)
Sb(1)−N(1)	1.983(7)	Sb(1′)−N(2)	1.968(7)
Te(1)−N(1)	2.051(7)		
Te(1)−N(2)	2.093(7)		
Te(1)−Cl(1)	2.657(3)		
Te(1)−Cl(2)	2.695(3)		
Sb(1)···C1(2)	3.290(3)	Sb(1′)···C1(1)	3.314(3)
Sb(1)···C1(1)* [a]	3.453(3)	C1(2)···Sb(1′)*	3.442(3)
Bond Angles			
N(2)−Sb(1)−N(3)	100.3(6)	N(3′)−Sb(1′)−N(1)	102.2(6)
N(2)−Sb(1)−N(1)	79.1(3)	N(1)−Sb(1′)−N(2)	79.6(3)
N(3)−Sb(1)−N(1)	97.7(5)	N(3′)−Sb(1′)−N(2)	99.8(6)
Sb(1)−N(1)−Te(1)	97.0(3)	Sb(1′)−N(1)−Te(1)	96.9(3)
Sb(1)−N(2)−Te(1)	96.2(3)	Sb(1′)−N(2)−Te(1)	95.3(3)
N(1)−Te(1)−N(2)	74.6(3)		
N(1)−Te(1)−Cl(1)	86.7(2)		
N(2)−Te(1)−Cl(1)	91.0(2)		
N(1)−Te(1)−Cl(2)	86.0(2)		
N(2)−Te(1)−Cl(2)	89.2(2)		
Cl(1)−Te(1)−Cl(2)	172.29(9)		

[a] Symmetry transformations used to generate equivalent atoms. Asterisk indicates: $-x + 1.5$, $y + 0.5$, $-z + 0.5$.

through weak Te···Cl interactions [MeN[PhBN(Me)]$_2$TeCl$_2$,[26] (Ph$_2$SN)$_2$TeCl$_2$,[24] [(Me$_3$Si)$_2$N]$_2$TeCl$_2$,[27] and [Cl$_2$Te(μ-N^{t}Bu)$_2$TeCl$_2$][10]]. Complex **4** also forms an extended structure (Figure 4), but in this case, the weak intermolecular interactions involve Sb···Cl, rather than Te···Cl, contacts in the range 3.290(3)−3.453(3) Å (cf., sum of van der Waals radii for Sb and Cl = 3.80 Å[17] or 4.0 Å[22]). Both of the Sb sites in the disordered molecules of **4** participate in these interactions.[28] The influence of the lone pair on tellurium is evident from the deviation from linearity of the ClTeCl unit [172.29(9)°] (Table 3). The almost equal Te−Cl bond distances [2.657(3)−2.695(3) Å] are at the high end of the range of values found for Te−Cl bonds in related complexes,[10,23−27] presumably as a result of the Sb···Cl interactions. Interestingly, the average Sb−N bond length in **4** (1.957 Å) is ~0.09 Å shorter than that in related compounds.[29] On the other hand, the average Te−N bond length of 2.072 Å in **4** is significantly longer than the corresponding value of 1.998 Å in PhB(μ-N^{t}Bu)$_2$TeCl$_2$, which has a dimeric structure.[30]

Complex **3** can be viewed as the result of the cycloaddition of a tellurium diimide monomer ^{t}BuNTeN^{t}Bu to dimer **2** (and ionization of one Sb−Cl bond). The formation of **4** is puzzling, but the presence of the ^{t}BuNH substituent implies that ^{t}BuNH$_2$, generated by hydrolysis, is involved. It is also significant that **4** is formed by both synthetic methods [(a) and (b)], implying that a common intermediate, for example, ^{t}BuNTe(μ-N^{t}Bu)$_2$SbCl (**6**), may be involved. Complex **6** can be viewed as (a) the cycloaddition product of two monomeric units, ^{t}BuNTeN^{t}Bu and ^{t}BuNSbCl, or (b) the metathesis product from the 1:1 reaction of [Te(N^{t}Bu)$_3$]$^{2-}$ with SbCl$_3$. Further reaction of **6** with Li$_2$[Te(N^{t}Bu)$_3$] would give Li{Sb[Te(N^{t}Bu)$_3$]$_2$}, the minor product of synthesis (b).

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Supporting Information Available: The X-ray crystallographic files, in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

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(28) In one molecule, the Sb(1) atom lies 0.879(9) Å above and the N(3) atom is 0.463(21) Å below the N(2)Te(1)N(1) plane. In the second molecule, the Sb(1′) atom lies 0.887(9) Å below and the N(3′) atom is 0.324(22) Å above that plane.

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