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cis-Di-*tert*-butylamidotetra-μ-*tert*-butylimido-di-μ-oxo-tetratellurium diiodide acetonitrile solvate

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The title compound, $C_{24}H_{56}N_6O_2Te_4^{2+}\cdot 2I^-\cdot C_2H_3N$ or $H[(^tBuNH)Te(\mu\text{-}N^tBu)_2Te(\mu\text{-}O)]_2(I)_2\cdot MeCN$, provides the first example of the *cis* form of an imidotelluroxane dication. The basket-shaped structure is bispirocyclic, with a 'backbone' Te$\cdots$Te$\cdots$Te$\cdots$Te pseudo-torsion angle of −31.51 (3)°. The structure exhibits two ionic Te$\cdots$I interactions [3.4103 (8) and 3.445 (1) Å] and two Te$\cdots$I close contacts [3.863 (1) and 3.924 (1) Å] within the molecule, and two Te$\cdots$I close contacts [3.651 (1) and 3.703 (2) Å] between ions. The intermolecular close contacts connect the cations into pairs which are linked by a weak Te$\cdots$I contact [4.090 (1) Å] to form an infinite chain.

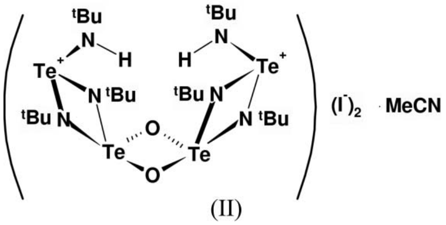
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.025
wR factor = 0.055
Data-to-parameter ratio = 19.9

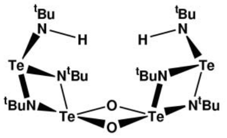
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

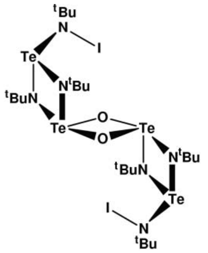
The reaction between the tellurium diimide $[(^tBuN)Te(\mu\text{-}N^tBu)_2Te(N^tBu)]$ (L) and LiI produces $\{[Li(THF)_2(L)](\mu_3\text{-}I)[LiI(L)]\}$, (I), a complex that incorporates two different types of Te$\cdots$I interactions (Chivers *et al.*, 2001). In a related study of the oxidation of L with I_2, we observed N—I bond formation and structurally characterized the dication $\{[(tBuNI)Te(\mu\text{-}NtBu)_2Te(\mu\text{-}O)]_2\}^{2+}$ (Konu *et al.*, 2005). The neutral imidotelluroxane ligand $[(^tBuN)Te(\mu\text{-}N^tBu)_2Te(\mu\text{-}O)]_2$ has been previously reported in Ag^I and Cu^I complexes (Chivers *et al.*, 1999). In all of these spirocyclic systems, the two Te_2N_2 rings adopt *trans* arrangement with respect to the central planar Te_2O_2 ring. In this contribution, we report the title compound, (II), which represents the first example of the *cis* form of a dimeric imidotelluroxane system.



(II)



$cis\text{-}[(^tBuNH)Te(\mu\text{-}N^tBu)_2Te(\mu\text{-}O)]_2^{2+}$



$trans\text{-}[(^tBuNI)Te(\mu\text{-}N^tBu)_2Te(\mu\text{-}O)]_2^{2+}$

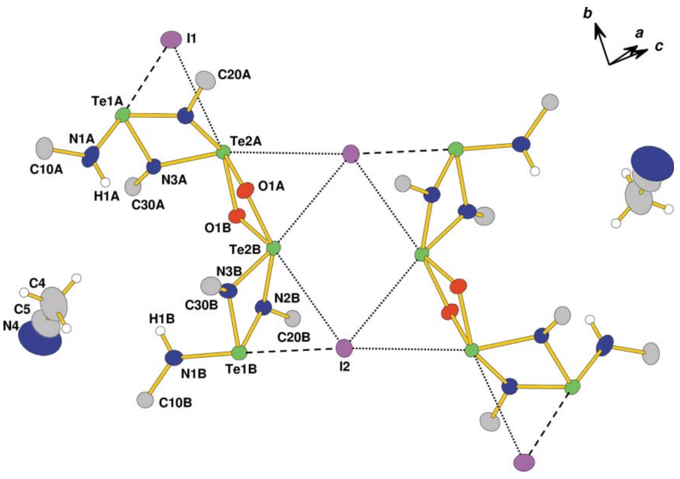


Figure 1
The structure of (II), indicating the numering of the atoms of the asymmetric unit and 50% displacement ellipsoids. Unlabelled atoms are generated by the symmetry code ($-x, 1-y, -z$). For clarity, only the α-C atoms of the *tert*-butyl groups are shown.

Compound (II) was obtained from the reaction of *L* with an equimolar mixture of [Me_4N]I and I_2, after recrystallization of the product from acetonitrile. The partial hydrolysis that occurred during the recrystallization process is a common feature of the extremely moisture-sensitive complexes of *L* (Chivers *et al.*, 1999; Konu *et al.*, 2005). The structure of (II) (Fig. 1) consists of the $[({}^t\mathrm{BuNH})\mathrm{Te}(\mu\text{-}\mathrm{N}^t\mathrm{Bu})_2\mathrm{Te}(\mu\text{-}\mathrm{O})]_2^{2+}$ dication, two I^- anions and an acetonitrile solvent molecule. The two Te_2N_2 rings in the dication occupy *cis* positions with respect to the Te_2O_2 ring. The positive charges on the dication formally reside on tellurium atoms Te1*A* and Te1*B*. Consequently, the the two ionic Te$\cdots$I interactions of 3.4103 (8) (Te1*A*$\cdots$I1) and 3.445 (1) Å (Te1*B*$\cdots$I2) are significantly shorter than the two intramolecular Te$\cdots$I close contacts of 3.863 (1) (Te2*A*$\cdots$I1) and 3.924 (1) Å (Te2*B*$\cdots$I2) and two intermolecular contacts of 3.651 (1) (Te2*B*$\cdots$I2) and 3.703 (2) Å (Te2*A*$\cdots$I2) to formally neutral Te atoms. The ionic Te$\cdots$I interactions are somewhat longer than the $I_3Te^+\cdots(\mu\text{-}I^-)$ distances of 3.187–3.343 Å found in γ- and ψ-TeI_4 (Beister *et al.*, 1986; Kniep *et al.*, 1988; Krebs & Paulat, 1976), but are comparable to the distance of 3.418 Å found in $Et_3Te^+\cdots I^-$ (Chadha & Drake, 1986). The intra -and intermolecular Te$\cdots$I close contacts show typical values for secondary bonding interactions (Närhi *et al.*, 2004). The intermolecular close contacts bind two cations into pairs (Fig. 1), which are linked to form an infinite chain with a weak Te$\cdots$I contact [I1$\cdots$Te1*B* = 4.090 (1) Å, *cf.* the sum of the van der Waals radii for tellurium and iodine: 4.35 Å (Emsley, 1998)] (Fig. 2).

Previously reported bispirocyclic complexes with a Te(μ-N)$_2$Te(μ-O)$_2$Te(μ-N)$_2$Te framework exhibit a center of symmetry in the middle of a planar Te_2O_2 ring (Chivers *et al.*, 1999; Konu *et al.*, 2005), whereas a center of symmetry in (II) is located at the center of the linked cation pair (Fig. 1). Consequently, the Te_2O_2 ring in (II) is not constrained by symmetry and is slightly twisted. Comparison of the bond parameters of the *cis* and *trans* Te(μ-N)$_2$Te(μ-O)$_2$Te(μ-N)$_2$Te arrangements reveals only a few significant differences. For steric reasons, the *cis* geometry in (II) shows a slightly wider Te1$\cdots$Te2$\cdots$Te2 angle (*ca* 134°) than the *trans* structures (122–128°). Consequently, the exocyclic NH^{t}Bu groups are more inclined towards the center of the Te_2O_2 ring, with an N1—Te1—N3 angle of *ca* 95° in (II), compared to an average of 100° for the corresponding angle in the *trans* complexes.

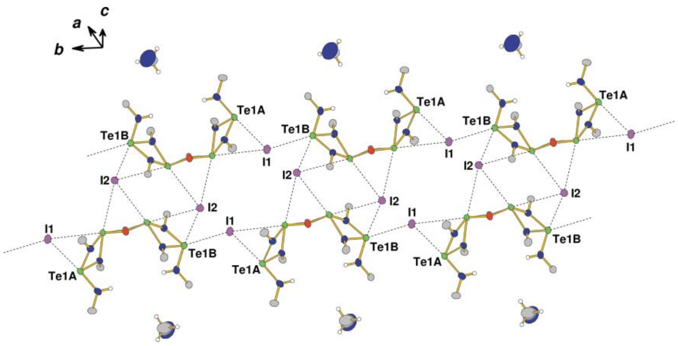


Figure 2
The ions of (II) linked into an infinite chain *via* weak Te$\cdots$I close contacts.

Experimental

A mixture of $(CH_3)_4NI$ (0.151 g, 0.75 mmol) and I_2 (0.190 g, 0.75 mmol) in tetrahydrofuran (20 ml) was added to a suspension of $[({}^t\mathrm{BuN})\mathrm{Te}(\mu\text{-}\mathrm{N}^t\mathrm{Bu})_2\mathrm{Te}(\mathrm{N}^t\mathrm{Bu})]$ (0.270 g, 0.50 mmol) in hexane (5 ml) at 243 K. The reaction mixture was stirred for 0.5 h at 243 K and 2.5 h at 298 K. The solvent was evaporated under vacuum, yielding 0.514 g of a red–brown powder. Recrystallization from acetonitrile at 263 K resulted, in one week, in a small amount of yellow crystals of (II).

Crystal data

$C_{24}H_{56}N_6O_2Te_4^{2+}\cdot 2I^-\cdot C_2H_3N$
$M_r = 1266.00$
Triclinic, $P\bar{1}$
$a = 12.340$ (3) Å
$b = 12.836$ (3) Å
$c = 14.121$ (3) Å
$\alpha = 81.70$ (3)°
$\beta = 86.20$ (3)°
$\gamma = 71.78$ (3)°
$V = 2101.8$ (9) Å^3

$Z = 2$
$D_x = 2.001$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 37429 reflections
$\theta = 2.3$–25.0°
$\mu = 4.25$ mm^{-1}
$T = 173$ (2) K
Rod, yellow
0.20 × 0.08 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SHELXTL*; Bruker, 2001)
$T_{min} = 0.484$, $T_{max} = 0.816$
37429 measured reflections
7399 independent reflections

6465 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 25.0°$
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.055$
$S = 1.10$
7399 reflections
372 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0109P)^2 + 2.0911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.96$ e Å^{-3}
$\Delta\rho_{min} = -1.40$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00098 (7)

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Table 1
Selected geometric parameters (Å, °).

Te1*A*—N2*A*	1.951 (3)	Te2*B*—O1*B*	1.918 (3)
Te1*A*—N1*A*	1.978 (4)	Te2*B*—N3*B*	2.034 (3)
Te1*A*—N3*A*	1.995 (3)	Te2*B*—O1*A*	2.098 (3)
Te2*A*—O1*A*	1.920 (3)	Te2*B*—N2*B*	2.119 (3)
Te2*A*—N3*A*	2.050 (3)	Te1*B*—N2*B*	1.963 (3)
Te2*A*—O1*B*	2.069 (2)	Te1*B*—N1*B*	1.975 (3)
Te2*A*—N2*A*	2.104 (3)	Te1*B*—N3*B*	2.013 (3)
Te1*A*···Te2*A*···Te2*B*···Te1*B*	−31.51 (3)		

H atoms were placed in idealized locations (N—H = 0.88 Å and C—H = 0.98 Å) and refined as riding. The highest difference peak is 1.55 Å from H31*B* and the deepest difference peak is 1.23 Å from N2*A*. $U_{\rm iso}$(H) values were constrained to be 1.2 times the $U_{\rm eq}$ value of the corresponding N atom and 1.5 times that of the corresponding C atom.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *WinGX* (Version 1.64-05; Farrugia, 1999).

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References

Beister, H. J., Kniep, R. & Schaefer, A. (1986). *Z. Kristallogr.* **174**, 12–13.

Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. Release 2.1. Crystal Impact GmbH, Bonn, Germany.

Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.

Chadha, R. K. & Drake, J. E. (1986). *J. Organomet. Chem.* **299**, 331–339.

Chivers, T., Parvez, M. & Schatte, G. (1999). *Inorg. Chem.* **38**, 5171–5177.

Chivers, T., Parvez, M. & Schatte, G. (2001). *Inorg. Chem.* **40**, 540–545.

Emsley, J. (1998). *The Elements*, 3rd ed. Oxford: Clarendon Press.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Kniep, R., Beister, H. J. & Wald, D. (1988). *Z. Naturforsch. Teil B*, **43**, 966–980.

Konu, J., Chivers, T., Schatte, G., Parvez, M. & Laitinen, R. S. (2005). *Inorg. Chem.* **44**, 2973–2982.

Krebs, B. & Paulat, V. (1976). *Acta Cryst.* B**32**, 1470–1476.

Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

Närhi, S. M., Oilunkaniemi, R., Laitinen, R. S. & Ahlgren, M. (2004). *Inorg. Chem.* **43**, 3742–3750.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276. Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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