organic papers

An orthorhombic polymorph of dichloro-bis[4-(dimethylamino)phenyl]tellurium

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Key indicators

Single-crystal X-ray study
$T = 130$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.022
wR factor = 0.057
Data-to-parameter ratio = 21.1

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

Unlike the previously known monoclinic form, the orthorhombic polymorph of dichlorobis[4-(dimethylamino)phenyl]-tellurium, $C_{16}H_{20}Cl_2N_2Te$, shows secondary Te$\cdots$Cl interactions.

Comment

The title compound, (I), crystallized from hexane–dichloromethane as yellow (monoclinic) crystals, in which individual molecules are associated *via* weak secondary Cl$\cdots$Cl interactions. Unlike most other diorganotellurium dichlorides, no secondary Te$\cdots$Cl interactions are present (Beckmann *et al.*, 2005). We have now found that the same compound crystallizes from toluene, affording a mixture of the yellow and a new orange (orthorhombic) polymorph. The orange crystals reveal a secondary Te$\cdots$Cl contact of 3.729 (1) Å, but lack any Cl$\cdots$Cl interactions. Both Cl atoms show Cl$\cdots$H contacts shorter than 2.95 Å, the sum of the van der Waals radii [Cl1$\cdots$H17A^i = 2.853 Å and Cl2$\cdots$H3^{ii} = 2.826 Å; symmetry codes: (i) $-\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (ii) $\frac{1}{2} + x, -y, z$]. As in the yellow polymorph, there are a number of π–π stacking interactions between adjacent 4-(dimethylamino)phenyl groups, the most significant being that between neighbouring C14—N2 bonds [3.346 (1) Å, measured from the middle of this bond to the middle of C14^{iii}—N2^{iii}; symmetry code: (iii) $\frac{3}{2} - x, y, 1 - z$].
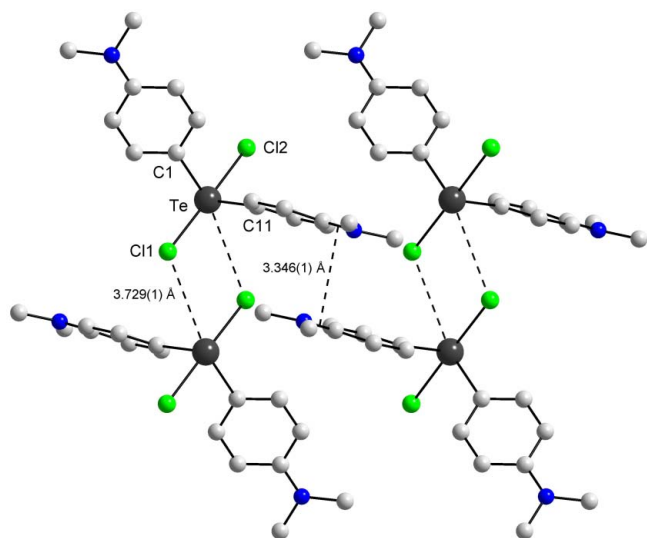
R = phenyl group with NMe₂ substituent

(I)
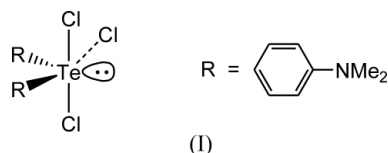
Experimental

Compound (I) was prepared according to the originally reported procedure of Morgan & Burgess (1929). Recrystallization from
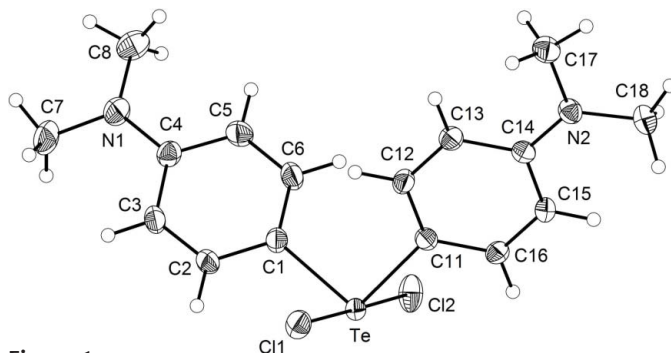
Figure 1
View of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Figure 2
View of (I) along the crystallographic b axis, showing the intermolecular Te···Cl and π–π stacking interactions as dashed lines.

toluene afforded a mixture of the previously known yellow modification (Beckmann *et al.*, 2005) and the new orange modification, both of which begin to decompose above 443 K and melt at 465–466 K.

Crystal data

$C_{16}H_{20}Cl_2N_2Te$	Mo $K\alpha$ radiation
$M_r = 438.84$	Cell parameters from 5119
Orthorhombic, *Ibca*	reflections
$a = 16.0504\,(10)$ Å	$\theta = 2.4$–$27.5°$
$b = 16.4302\,(10)$ Å	$\mu = 2.00$ mm^{-1}
$c = 26.5331\,(16)$ Å	$T = 130\,(2)$ K
$V = 6997.1\,(7)$ Å^3	Block, orange
$Z = 16$	$0.40 \times 0.30 \times 0.30$ mm
$D_x = 1.666$ Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	4006 independent reflections
	3693 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.017$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 27.5°$
	$h = -20 \rightarrow 20$
$T_{min} = 0.491$, $T_{max} = 0.549$	$k = -17 \rightarrow 21$
20 865 measured reflections	$l = -33 \rightarrow 33$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0239P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$+ 11.1642P]$
$wR(F^2) = 0.057$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.004$
4006 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.49$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—Te	2.090 (2)	Cl2—Te	2.5103 (5)
C11—Te	2.086 (2)	Te···Cl1^{iv}	3.7288 (5)
Cl1—Te	2.5364 (5)		
C11—Te—C1	99.63 (7)	Cl2—Te—Cl1	179.19 (2)
C11—Te—Cl2	90.33 (5)	C11—Te···Cl1^{iv}	81.92 (5)
C1—Te—Cl2	89.44 (5)	C1—Te···Cl1^{iv}	165.64 (5)
C11—Te—Cl1	90.05 (5)	Cl2—Te···Cl1^{iv}	104.86 (1)
C1—Te—Cl1	89.78 (5)	Cl1—Te···Cl1^{iv}	75.91 (1)

Symmetry code: (iv) $1 - x$, $-y$, $1 - z$.

The H atoms were placed in geometrically calculated positions and refined using a riding model (primary C—H = 0.98 Å and aromatic C—H = 0.95 Å). The isotropic displacement parameters were constrained at 1.2 (for non-methyl) and 1.5 (for methyl) times U_{eq} of the carrier C atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff *et al.*, 1996); software used to prepare material for publication: *SHELXL97*.

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