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trans-Bis(1*H*-benzimidazole-1-thione-*κS*)-tetrachlorotellurium methanol disolvate

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The Te atom in the title complex, *trans*-$[\mathrm{TeCl_4(C_6H_4N_2H_2CS)_2}]\cdot 2\mathrm{CH_4O}$ or $\mathrm{C_{16}H_{20}Cl_4N_4O_2S_2Te}$, occupies a special position at a crystallographic inversion centre and has an octahedral coordination formed by four chloro ligands and the S atoms of two benzimidazole–thione molecules. The hydrogen-bond system involving the disordered solvent methanol molecules links the tellurium complexes into the infinite two-dimensional aggregates in the crystal.

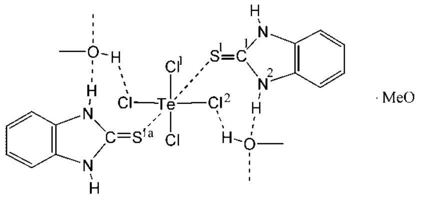
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
Disorder in solvent or counterion
R factor = 0.043
wR factor = 0.113
Data-to-parameter ratio = 16.7

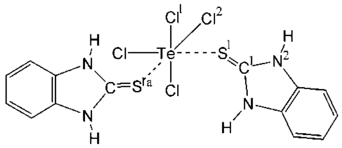
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

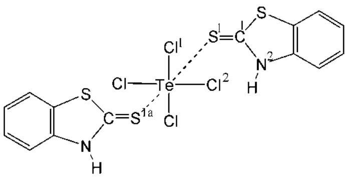
While many complexes of the otherwise unstable $\mathrm{TeCl_2}$ with thiourea derivatives are known (Haiduc *et al.*, 1994), only a few examples of complexes of these ligands with $\mathrm{TeCl_4}$ have been reported (Husebye & George, 1969; von Deuten *et al.*, 1979*a*,*b*). In an earlier synthesis, formation of *cis*-$\mathrm{TeCl_4(phtu)_2}$ was reported when $\mathrm{TeCl_4}$ and 1,3-dihydro-benzoimidazole-2-thione (phtu) were reacted with each other. We obtained the title compound *trans*-$\mathrm{TeCl_4(phtu)_2}\cdot 2\mathrm{MeOH}$, (I), by the reaction of $\mathrm{TeO_2}$ with an excess of phtu, dissolved in a mixture of methanol and concentrated hydrochloric acid. No reduction of $\mathrm{Te^{IV}}$ or oxidation of the phtu ligand was observed.



(I)



(II)



(III)

The *Scheme* shows the molecular formulae of (I) and the related compounds *cis*-bis(1*H*-benzimidazole-1-thione)tetrachlorotellurium [(II); von Deuten *et al.*, 1979*b*] and *trans*-bis(2,3-dihydrobenzothiazole-2-thione)tetrachlorotellurium

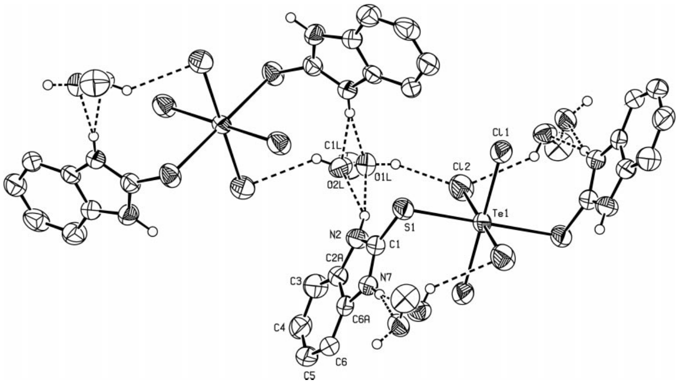


Figure 1
*ORTEP*II (Johnson, 1976) view of a fragment of the structure of (I). Displacement ellipsoids are shown at the 50% probability level. H atoms involved in hydrogen bonding are drawn as circles of arbitrary radii; other H atoms have been omitted. The minor component (O1L) of the disordered solvent molecule is represented by dashed lines.

[(III); von Deuten *et al.*, 1979*a*]. The molecular structure of (I) is depicted in Fig. 1.

The Te—Cl distances in (I) are within the expected range for complexes of $TeCl_4$ with thiourea derivatives. The Te—S distances are considerably longer in the *trans* complexes than in the *cis* complex (Table 1), an effect attributed to the smaller *trans* influence of the Cl atom compared to the thiourea ligand. The shortening of the Te—S bond in the *cis* complex is accompanied by a dramatic lengthening of the S—C distance, a hint of the essential role of the C—S π-bond for Te···S interaction. This assumption is also supported by the values of the N—C—S—Te torsion angles, which are close to 90° and hence allow for an optimum interaction of the C—S π-bond with acceptor orbitals at the Te atom. In all cases, the thiourea C atom has a planar environment, as can be seen from the sum of its bonding angles (Table 2).

The OH group of the methanol solvent molecule is disordered over two positions. In both positions, the methanol O-atom electron pairs may act as the hydrogen-bond acceptor for the N2—H2 and N7—H7 groups of the benzimidazole–thione ligand [O···N = 2.774 (8)–2.879 (14) Å and O···H—N = 142–167°] and the methanol O—H groups serve as hydrogen-bond donors in the O—H···Cl2 hydrogen bonds [O1*L*···Cl2 = 3.165 (13) Å and O1*L*—H1*L*···Cl2 = 149°; O2*L*···Cl2 = 3.171 (7) Å and O2*L*—H2*L*···Cl2 = 138°]. These bonds link the molecules of the complex and methanol molecules into hydrogen-bonded layers.

Experimental

TeO_2 (1.90 g, 11.9 mmol) and 1,3-dihydro-benzoimidazole-2-thione (7.10 g, 47.2 mmol) were dissolved in a mixture of 30 ml of methanol and 8 ml of concentrated hydrochloric acid. The reaction mixture was refluxed for 3 h, during which time it adopted an orange color. The hot solution was filtered and on cooling to room temperature, a dark-red solid precipitated, which was recrystallized from methanol. Crystals of (I) precipitated at room temperature from this solution.

Crystal data

$C_{16}H_{20}Cl_4N_4O_2S_2Te$
$M_r = 633.88$
Monoclinic, $P2_1/n$
$a = 8.7211$ (11) Å
$b = 12.4005$ (7) Å
$c = 11.6049$ (11) Å
$\beta = 103.785$ (5)°
$V = 1218.9$ (2) Å^3
$Z = 2$

$D_x = 1.727$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 25.0$–31.0°
$\mu = 15.45$ mm^{-1}
$T = 295$ (2) K
Block, red brown
0.12 × 0.07 × 0.06 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\omega$ scans
Absorption correction: numerical [according to de Meulenaer & Tompa (1965) in *PLATON* (Spek, 2001)]
$T_{\min} = 0.245$, $T_{\max} = 0.486$
2465 measured reflections

2465 independent reflections
1938 reflections with $I > 2\sigma(I)$'
$\theta_{\max} = 73.9°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 0$
$l = -14 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.113$
$S = 1.04$
2465 reflections
148 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -1.34$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1*L*	0.99	1.91	2.879 (14)	165
N2—H2···O2*L*	0.99	1.93	2.774 (8)	142
N7—H7···O1*L*^{i}	0.91	1.96	2.853 (14)	167
N7—H7···O2*L*^{i}	0.91	1.94	2.819 (8)	163
O1*L*—H1*L*···Cl2	0.94	2.32	3.165 (13)	149
O2*L*—H2*L*···Cl2^{ii}	0.81	2.52	3.171 (7)	138

Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$.

Table 2
Selected structural parameters (Å, °) of (I), (II) (von Deuten *et al.*, 1979*b*) and (III) (von Deuten *et al.*, 1979*a*); for numbering of the atoms see Fig. 1.

	(I)	(II)	(III)
Te—Cl1	2.502 (1)	2.525 (3)	2.509 (2)
Te—Cl2	2.554 (2)	2.559 (7)	2.502 (2)
Te—S1	2.696 (2)	2.630 (6)	2.704 (2)
S1—C1	1.712 (5)	1.792 (12)	1.711 (7)
S1—Te—S1*a*	180.0	87.3 (3)	180.0
Te—S1—C1	100.5 (2)	102.3 (4)	103.1 (2)
(*X*—C1—*Y*)^{a}	360.1 (7)	360.0 (10)	359.8 (6)
N—C—S—Te	89.6 (5)	87.7	−99.2

Note: (*a*) is the sum of the bond angles at C1.

H atoms attached to C atoms were placed at calculated positions in every cycle of the refinement. H atoms bound to nitrogen and oxygen were located from a difference Fourier synthesis. All H atoms were refined using a riding model. The displacement parameter for the H atom of the solvent molecule was fixed at 1.5 times the equivalent isotropic displacement parameter of non-H atoms. The displacement parameters for the other H atoms were refined individually. The solvent molecule is disordered over two sites with occupancy factors of 0.65 and 0.35.

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Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001).

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