

The Crystal Structure of Chlorobis(diethyldithiocarbamato)-4-ethoxyphenyltellurium(IV) Hemidioxane Solvate, $[\text{TeCl}(\text{Et}_2\text{NCS}_2)_2(\text{EtOPh})] \cdot 0.5\text{C}_4\text{H}_8\text{O}_2$

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The crystal structure of the title compound was solved by means of X-ray crystallographic methods. The orange crystals of the title compound are triclinic with $a = 11.531(1)$, $b = 14.280(2)$, $c = 18.571(3)$ Å, $\alpha = 78.44(1)$, $\beta = 70.90(1)$, $\gamma = 72.27(1)^\circ$, $Z = 2$, space group $P\bar{1}$ and $R = 0.066$ for 9759 unique reflections. There are two types of crystallographically independent complex molecules in the unit cell. The structure is ψ -pentagonal bipyramidal with the 4-ethoxyphenyl group and the lone pair occupying axial positions. Weak, secondary $\text{Te} \cdots \text{S}$ bonds of average length 3.722 Å tie the complex molecules together in pairs across centres of symmetry. These bonds are roughly *trans* to the aryl groups; $\angle \text{C}-\text{Te} \cdots \text{S}$ has an average value of 162° . The average $\text{Te}-\text{S}$, $\text{Te}-\text{Cl}$ and $\text{Te}-\text{C}$ bond lengths are 2.676, 2.679 and 2.140 Å, respectively.

Tetravalent tellurium forms seven-coordinate pentagonal bipyramidal complexes of the type TeL_3X where L is a bidentate dithiolate and X is a halogen atom or an aryl group.^{1,2} Few complexes with X = aryl are known, and the stereochemical role of the lone pair is not clear. It was therefore decided to study more complexes of this type. An attempted preparation of $[\text{Te}(\text{Et}_2\text{NCS}_2)_3\text{EtOPh}]$ from the corresponding aryltellurium trichloride and sodium diethyldithiocarbamate,^{3,4} yielded a powdery product which upon recrystallization from dioxane gave yellow crystals of $[\text{TeCl}(\text{Et}_2\text{NCS}_2)_2\text{EtOPh}] \cdot 0.5\text{C}_4\text{H}_8\text{O}_2$. It was then decided to determine the structure of this complex, as it was assumed that the lone pair of electrons on Te(IV) might occupy the missing coordination position relative to $[\text{Te}(\text{Et}_2\text{NCS}_2)_3\text{EtOPh}]$.

Experimental

Preparation of the complex. The complex was prepared from 4-ethoxyphenyltellurium trichloride and sodium diethyldithiocarbamate as described for the preparation of aryltelluriumtris(diethyldithiocarbamates).⁴ Recrystallization from dioxane gave yellow crystals. Preliminary X-ray crystallographic studies indicated that the complex was $[\text{TeCl}(\text{Et}_2\text{NCS}_2)_2\text{EtOPh}] \cdot 0.5\text{C}_4\text{H}_8\text{O}_2$ (**1**), rather than the expected $[\text{Te}(\text{Et}_2\text{NCS}_2)_3(\text{EtOPh})]$.

Elementary analysis gave the following results. Found: C 38.74; Cl 5.90; O 5.39; S 20.43; m.p. = 167.0°C . Calc. for $\text{C}_{20}\text{H}_{33}\text{ClN}_2\text{O}_2\text{S}_4\text{Te}$ (**1**): C 38.45; Cl 5.67; O 5.12; S 20.53. Preparation of the related complex $[\text{TeCl}$

$(\text{Et}_2\text{NCS}_2)_2\text{Ph}]$ and the recording of its NMR spectrum has recently been published.⁵ That complex was prepared by adding $\text{Na}(\text{Et}_2\text{NCS}_2)_2$ to PhTeCl_3 in CH_2Cl_2 in the molar ratio 2:1.

Table 1. X-Ray crystallography parameters.

Compound	$[\text{TeCl}(\text{Et}_2\text{NCS}_2)_2(\text{EtOPh})] \cdot 0.5\text{C}_4\text{H}_8\text{O}_2$
Diffractometer	Enraf-Nonius CAD-4
Radiation	Mo $K\alpha$
Wavelength/Å	0.71073
Crystal system	Triclinic
$a/\text{Å}$	11.531(1)
$b/\text{Å}$	14.280(2)
$c/\text{Å}$	18.571(3)
$\alpha/^\circ$	78.44(1)
$\beta/^\circ$	70.90(1)
$\gamma/^\circ$	72.27(1)
$V/\text{Å}^3$	2735.0
Space group	$P\bar{1}$ (No. 2)
Formula weight	620.8
Z ($2 \times$ Formula weight)	2
$D_{\text{calc}}/\text{g cm}^{-3}$	1.507
$\mu(\text{Mo } K\alpha)/\text{cm}^{-1}$	14.99
Crystal dimensions/ cm^3	$0.019 \times 0.022 \times 0.043$
Scan mode/ $\theta_{\text{max}}/^\circ$	ω -scan/28
Fudge factor	0.030
Scale factor	0.3714
No. of independent measurements	13172
No. with $I > 3\sigma I$	9801
Transmission factors	0.6881–0.7759
$R = \Sigma F_o - F_c /\Sigma F_o$	0.031
$R_w = [\Sigma w(F_o - F_c)^2/\Sigma w F_o^2]^{0.5}$	0.041
$S = [\Sigma w(\Delta F)^2/N \pm \eta]^{0.5}$	1.627

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X-Ray data. Crystal data, intensity collection and structural refinement parameters are listed in Table 1. Three standard reflections were remeasured every 2 h for scaling purposes. There was no significant decrease in intensity of these reflections. The intensities were corrected for Lorentz, polarization, extinction and absorption effects.

Structure determination

The space group $P\bar{1}$ was chosen on the basis of E statistics. The structure was solved by means of direct methods and refined by least-squares methods. The asymmetric unit contained two crystallographically independent formula units. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were positioned by a special program and refined with fixed C–H bond lengths (0.95 Å). They were given isotropic temperature factors equal to those on the parent carbon atoms. Dioxane hydrogens were not in-

cluded. The function minimized is $\Sigma w(\Delta F)^2$. A weighting scheme with $w = 4F_0^2/\sigma(F_0^2)^2$, where $\sigma(F_0^2) = [\sigma(I)^2 + (0.03I)^2]^{1/2}$, was used. No peaks above 0.78 or below -0.64 e \AA^{-3} were found in the final difference map. The successful refinement justified the choice of space group. During refinement it became apparent that the dioxane molecules and one of the diethyldithiocarbamate methyl groups (C13) were disordered. A good resolution of the disorder was not achieved, but this did not appear to influence the positional parameters of the rest of the structure to a significant extent. Computer programs were supplied by Enraf-Nonius (SPD-Vax 1985 and CAD-4-1985).

Results and discussion

Atomic parameters are listed in Table 2, while interatomic distances and angles are given in Tables 3 and 4, respectively. Tables of observed and calculated structure factors,

Table 2. Positional parameters with estimated standard deviations.

Molecule 1A				Molecule 1B			
Atom	x	y	z	Atom	x	y	z
Te1	0.42394(2)	0.34643(1)	0.56439(1)	Te2	-0.05453(2)	0.14622(1)	0.06267(1)
Cl1	0.17455(8)	0.38227(7)	0.63670(6)	Cl2	-0.30603(7)	0.19880(6)	0.12717(5)
S11	0.62826(7)	0.40538(6)	0.55034(5)	S21	0.14626(8)	-0.00761(6)	0.04934(5)
S12	0.38360(8)	0.47636(6)	0.66276(5)	S22	-0.10236(8)	-0.01369(6)	0.15856(5)
S13	0.60673(8)	0.24644(6)	0.45762(5)	S23	0.13764(8)	0.21097(7)	-0.03600(5)
S14	0.34847(8)	0.21787(6)	0.51176(5)	S24	-0.12111(8)	0.34132(6)	0.00789(5)
O1	0.5679(2)	0.0081(2)	0.8178(1)	O2	0.0408(2)	0.2787(2)	0.3365(1)
N1	0.5909(3)	0.5342(2)	0.6459(2)	N3	0.1018(3)	-0.1669(2)	0.1446(2)
N2	0.5335(3)	0.1274(2)	0.3984(2)	N4	0.0727(3)	0.3972(2)	-0.0981(2)
C1	0.4698(3)	0.2300(2)	0.6509(2)	C1'	-0.0228(3)	0.1937(2)	0.1553(2)
C2	0.3741(3)	0.1990(2)	0.7090(2)	C2'	-0.1188(3)	0.2592(2)	0.2007(2)
C3	0.4032(3)	0.1250(3)	0.7661(2)	C3'	-0.1013(3)	0.2893(2)	0.2620(2)
C4	0.5284(3)	0.0809(2)	0.7647(2)	C4'	0.0133(3)	0.2541(3)	0.2773(2)
C5	0.6243(3)	0.1104(3)	0.7048(2)	C5'	0.1113(3)	0.1889(3)	0.2307(2)
C6	0.5950(3)	0.1840(2)	0.6484(2)	C6'	0.0928(3)	0.1597(3)	0.1702(2)
C7	0.4720(4)	-0.0205(3)	0.8832(2)	C7'	-0.0582(3)	0.3403(3)	0.3881(2)
C8	0.5397(4)	-0.1024(4)	0.9311(3)	C8'	-0.0049(4)	0.3500(3)	0.4504(2)
C10	0.5393(3)	0.4780(2)	0.6237(2)	C20	0.0536(3)	-0.0737(2)	0.1210(2)
C11	0.5131(4)	0.6047(3)	0.7044(2)	C21	0.0263(4)	-0.2210(3)	0.2100(2)
C12	0.7267(3)	0.5315(3)	0.6137(2)	C22	0.2345(3)	-0.2199(3)	0.1095(2)
C13	0.5359(8)	0.5778(6)	0.7727(3)	C23	0.0224(4)	-0.1970(3)	0.2865(3)
C14	0.8096(4)	0.4484(4)	0.6531(3)	C24	0.3248(4)	-0.2102(4)	0.1499(3)
C15	0.4990(3)	0.1893(2)	0.4504(2)	C25	0.0330(3)	0.3254(2)	-0.0474(2)
C16	0.4453(4)	0.0750(3)	0.3925(2)	C26	0.2045(4)	0.3807(3)	-0.1486(2)
C17	0.6608(4)	0.1094(3)	0.3412(2)	C27	-0.0106(4)	0.4959(3)	-0.1074(2)
C18	0.3736(4)	0.1325(4)	0.3356(3)	C28	0.2916(4)	0.4035(4)	-0.1143(3)
C19	0.7537(5)	0.0228(4)	0.3686(3)	C29	-0.0726(4)	0.5048(3)	-0.1698(2)

The dioxane molecules:

a1			b1 (occupancy 0.6)			b2 (occupancy 0.4)					
Atom	x	y	z	Atom	x	y	z	Atom	x	y	z
O3	0.4242(3)	0.5444(3)	0.0646(2)	O4	0.1288(6)	0.9808(5)	0.4657(4)	O5	0.060(1)	0.9792(8)	0.4244(6)
C131	0.4764(5)	0.6029(4)	-0.0004(3)	C141	0.069(1)	1.0707(8)	0.4725(6)	C151	0.013(1)	1.066(1)	0.4329(9)
C132	0.3981(6)	0.4589(5)	0.0516(3)	C142	0.072(1)	0.9052(8)	0.4872(6)	C152	0.008(1)	0.921(1)	0.4552(9)

Table 3. Bond lengths/Å. Estimated standard deviations in parentheses. S11(-) and S21(-) are generated from S11 and S21 by centres of symmetry at 1/2,1/2,1/2 and 0,0,0, respectively.

Molecule 1A		Molecule 1B	
Te1-S11	2.6543(7)	Te2-S21	2.6444(7)
Te1-S12	2.6968(7)	Te2-S22	2.6805(7)
Te1-S13	2.6418(7)	Te2-S23	2.6523(7)
Te1-S14	2.7076(7)	Te2-S24	2.7310(7)
Te1-Cl1	2.6808(7)	Te2-Cl2	2.6776(7)
Te1-Cl	2.139(2)	Te2-Cl'	2.140(2)
S11-C10	1.728(3)	S21-C20	1.727(3)
S12-C10	1.708(3)	S22-C20	1.713(3)
S13-C15	1.731(3)	S23-C25	1.732(3)
S14-C15	1.714(3)	S24-C25	1.708(3)
N1-C10	1.323(3)	N3-C20	1.322(3)
N1-C11	1.499(4)	N3-C21	1.473(4)
N1-C12	1.474(4)	N3-C22	1.476(4)
C11-C13	1.336(6)	C21-C23	1.511(5)
C12-C14	1.508(5)	C22-C24	1.519(5)
N2-C15	1.323(3)	N4-C25	1.329(3)
N2-C16	1.476(4)	N4-C26	1.477(4)
N2-C17	1.486(4)	N4-C27	1.458(4)
C16-C18	1.511(5)	C26-C28	1.492(5)
C17-C19	1.495(5)	C27-C29	1.517(5)
C1-C2	1.381(3)	C1'-C2'	1.373(3)
C2-C3	1.385(4)	C2'-C3'	1.387(4)
C3-C4	1.382(4)	C3'-C4'	1.366(4)
C4-C5	1.389(4)	C4'-C5'	1.390(4)
C5-C6	1.374(4)	C5'-C6'	1.372(4)
C6-C1	1.382(3)	C6'-C1'	1.373(4)
O1-C4	1.362(3)	O2-C4'	1.373(3)
O1-C7	1.437(3)	O2-C7'	1.416(3)
C7-C8	1.500(5)	C7'-C8'	2.521(4)
Te1-S11(-)	3.7387(7)	Te2-S21(-)	3.7051(8)

anisotropic temperature factors, hydrogen-atom parameters and least-squares molecular planes are available from one of the authors (K.M.M.) on request. The structure of the complex molecules **1A** and **1B** are shown in Fig. 1. A stereoscopic view of the unit-cell contents is shown in Fig. 2.

Description of the structure. The two crystallographically independent complex molecules **1A** and **1B** are very similar. The structure of [TeCl(Et₂NCS₂)₂(EtOPh)] can be described as ψ -pentagonal bipyramidal with the lone pair of electrons occupying an axial position. This agrees with predictions of the structure of the analogous compound [TeCl(Et₂NCS₂)₂Ph] based on NMR studies in solution.⁵ In solution, weak intermolecular Te...S bonds are probably absent. The plane through the Te-C and Te-Cl bonds is an approximate mirror plane. As can be seen from Fig. 2, intermolecular, weak secondary Te...S bonds tie the molecules into pairs across centres of symmetry. These weakly bonded sulfur atoms approach the coordination position presumably occupied by the lone pair. Average values are Te...S_{ax} = 3.722 Å and \angle S_{ax}...Te-C_{ax} = 162.0°. This angle is significantly greater than the corresponding ones in com-

Table 4. Bond angles with estimated standard deviations.

Molecule 1A		Molecule 1B	
Cl-Te1-S11	141.70(2)	Cl2-Te2-S21	142.44(2)
Cl1-Te1-S12	75.53(2)	Cl2-Te2-S22	76.11(2)
Cl1-Te1-S13	143.21(2)	Cl2-Te2-S23	143.15(2)
Cl1-Te1-S14	76.74(2)	Cl2-Te2-S24	77.01(2)
Cl1-Te1-C1	92.38(7)	Cl2-Te2-C1'	90.73(7)
S11-Te1-S12	66.45(2)	S21-Te2-S22	66.90(2)
S11-Te1-S13	74.88(2)	S21-Te2-S23	74.15(2)
S11-Te1-S14	141.56(2)	S21-Te2-S24	140.43(2)
S11-Te1-C1	90.79(7)	S21-Te2-C1'	93.58(7)
S12-Te1-S13	141.26(2)	S22-Te2-S23	140.72(2)
S12-Te1-S14	151.63(2)	S22-Te2-S24	152.42(2)
S12-Te1-C1	88.02(6)	S22-Te2-C1'	86.98(7)
S13-Te1-S14	66.75(2)	S23-Te2-S24	66.28(2)
S13-Te1-C1	90.29(6)	S23-Te2-C1'	90.57(7)
S14-Te1-C1	87.29(6)	S24-Te2-C1'	87.27(7)
Te1-S11-C10	88.68(9)	Te2-S21-C20	88.44(9)
Te1-S12-C10	87.72(9)	Te2-S22-C20	87.54(9)
Te1-S13-C15	88.78(9)	Te2-S23-C25	88.97(9)
Te1-S14-C15	86.99(9)	Te2-S24-C25	86.89(9)
C10-N1-C11	121.4(2)	C20-N3-C21	121.0(2)
C10-N1-C12	121.8(2)	C20-N3-C22	121.9(2)
C11-N1-C12	116.8(2)	C21-N3-C22	117.0(2)
C15-N2-C16	121.4(3)	C25-N4-C26	121.4(3)
C15-N2-C17	122.2(2)	C25-N4-C27	122.2(2)
C16-N2-C17	116.3(2)	C26-N4-C27	116.4(2)
S11-C10-S12	117.1(1)	S21-C20-S22	117.1(1)
S11-C10-N1	120.2(2)	S21-C20-N3	120.8(2)
S12-C10-N1	122.6(2)	S22-C20-N3	122.0(2)
S13-C15-S14	117.4(1)	S23-C25-S24	117.7(2)
S13-C15-N2	120.1(2)	S23-C25-N4	120.3(2)
S14-C15-N2	122.5(2)	S24-C25-N4	122.0(2)
N1-C11-C13	114.6(4)	N3-C21-C23	112.7(3)
N1-C12-C14	112.6(3)	N3-C22-C24	112.2(2)
N2-C16-C18	111.0(3)	N4-C26-C28	112.6(3)
N2-C17-C19	111.7(3)	N4-C27-C29	111.1(3)
Te1-C1-C2	120.0(2)	Te2-C1'-C2'	120.0(2)
Te1-C1-C6	120.5(2)	Te2-C1'-C6'	120.9(2)
C2-C1-C6	119.4(2)	C2'-C1'-C6'	119.1(2)
C1-C2-C3	120.3(2)	C1'-C2'-C3'	121.0(2)
C2-C3-C4	120.2(2)	C2'-C3'-C4'	119.7(2)
C3-C4-C5	119.2(2)	C3'-C4'-C5'	119.4(3)
C4-C5-C6	120.4(2)	C4'-C5'-C6'	120.4(3)
C1-C6-C5	120.4(2)	C1'-C6'-C5'	120.4(3)
C4-O1-C7	117.5(2)	C4'-O2-C7'	118.0(2)
O1-C7-C8	106.8(3)	O2-C7'-C8'	107.1(3)
Cl1-Te1-S11(-)	90.22(2)	Cl2-Te2-S21(-)	82.08(2)
S11(-)-Te1-S11	76.16(2)	S21(-)-Te2-S21	82.67(2)
S11(-)-Te1-S12	75.10(2)	S21(-)-Te2-S22	75.59(2)
S11(-)-Te1-S13	98.41(2)	S21(-)-Te2-S23	104.91(2)
S11(-)-Te1-S14	110.92(2)	S21(-)-Te2-S24	106.68(2)
S11(-)-Te1-C1	161.70(6)	S21(-)-Te2-C1'	162.27(7)

plexes where the Te...S_{ax} bond is intramolecular (Table 5).⁵⁻⁷ This is interpreted as indicating that there is greater freedom for the seventh ligand to approach a true axial position in comparison with the situation where this ligand, in spite of its small bite, must simultaneously span an equatorial and an axial position. Solution NMR spectra of the related complex [TeCl(Et₂NCS₂)₂Ph] indicate, however, a ψ -pentagonal bipyramidal structure,⁵ although in this case weak intermolecular Te...S contacts > 3.3 Å are

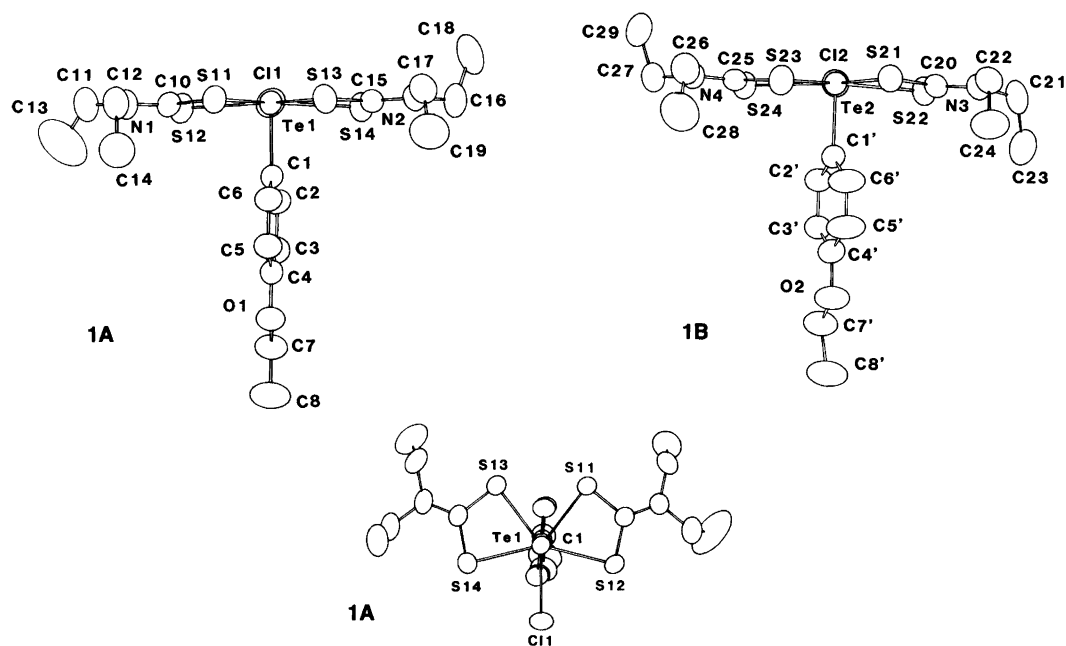


Fig. 1. The structure of the two crystallographically independent complex molecules **1A** and **1B**. Above they are seen along the Te–Cl bonds. Below **1A** is viewed at right angles to the pentagonal TeClS₄ plane.

probably broken up on going from the solid state to solution. Taking into account this weak bond, the complex may alternatively be considered as distorted pentagonal bipyramidal. As such, its structure is similar to those of the aryltris(dithiolates) of Te(IV) (Table 5).^{5–8} In these complexes the role of the lone pair of electrons is unclear. Including the weak axial bond of 3.2–3.5 Å permits a description of these complexes as distorted ψ -dodecahedral with an active lone pair⁵ or alternately as distorted pentagonal bipyramidal complexes in which the distortion is due to

the small bite of the ligand spanning equatorial and axial positions, possibly combined with the *trans* influence of the aryl group.⁶ Such complexes have a different structure from the halogen or pseudohalogen tris(dialkyldithiocarbamates) of Te(IV).^{1,8} The latter are distorted pentagonal bipyramidal, with the main distortion being due to the small bite of the axial–equatorial bidentate ligand. However, there the equatorial sulfur atom of that ligand is lifted above the equatorial plane owing to the small bite,⁸ in an analogous manner to the situation in similar transition-

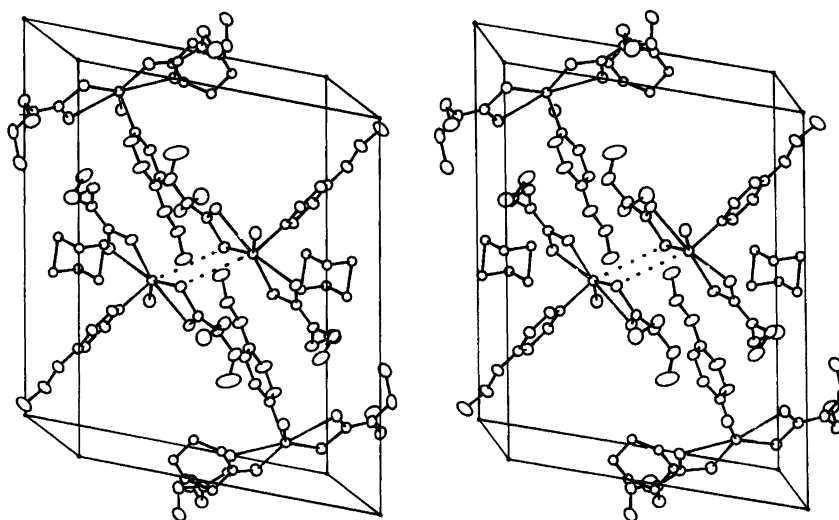


Fig. 2. The packing of tellurium complexes and dioxane molecules in the unit cell (dioxane **b2** is excluded). Weak intermolecular Te...S contacts connecting two **1A** molecules are indicated by dotted lines.

Table 5. Selected data for some [TeL₃X] and [TeL₂XY] complexes. L is a dithiolate ligand and Y is a halogen. Distances in Å, angles in °.

Complex		$r_{\text{Te,eq}}$	$X_{\text{ax}}-\text{Te}\cdots\text{S}_{\text{ax}}^a$	X_{ax}	Te- X_{ax}
(1) [TeCl(Et ₂ NCS ₂) ₂ (4-EtPh)]	1A	1.65	161.84(7)	4-EtOPh	2.139(3)
	1B	1.65	162.29(7)	4-EtOPh	2.141(3)
(2) [Te(Et ₂ NCS ₂) ₂ l(Me)]		1.66	—	CH ₃	2.115(5)
(3) [Te(Et ₂ NCS ₂) ₂ {(EtO) ₂ PS ₂ }(Ph)]		1.65	148.0(4)	Ph	2.138(1)
(4) [Te(Et ₂ NCS ₂) ₂ Ph]		1.68	144.6(2)	Ph	2.124(1)
(5) [Te(Me ₂ NCS ₂) ₃ (4-MeOPh)]		1.68	143.34(5)	4MeOPh	2.156(5)
(6) [TeCl{HOEt}(Me)NCS ₂] ₃		1.66	174.87(3)	Cl	2.772(1)

^aIn compound 1, the bond is *intermolecular*, in the others it is *intramolecular*.

metal complexes, and the lone pair is probably stereochemically inactive in the sense that it does not occupy a position in the coordination polyhedron.

It appears that a *trans* influence operates between axial ligands in the pentagonal bipyramidal TeL₃X (L = bidentate) complexes. For example, when X is an aryl or alkyl group, the Te-S bond *trans* to Te-C is more than 0.8 Å longer than the corresponding covalent bond.^{6,7} When X is a halogen or pseudohalogen atom, the Te-S bond *trans* to Te-X is the shortest of all Te-S bonds in the complex, typically 0.3 Å longer than a covalent bond.^{1,2,8} Another effect is also found, pointing to an influence between the odd equatorial ligand (e.g. the halogen atoms in **1**, the sulfur atom of the axial-equatorial ligand in **2**) and the two equatorial Te-S bonds "*trans*" to it at ca. 143°. The shorter is the bond between the tellurium and the odd ligand, the longer are the *trans* Te-S bonds, and vice versa.

The best plane through tellurium and the donor atoms of the equatorial ligands lies within 0.11 to 0.14 Å of these atoms in **1A** and **1B**. Another molecular plane through Cl, Te and C_{ax} forms an angle of nearly 90° with the equatorial plane. Also, the aryl groups are nearly at right angles to the equatorial plane.

The main difference between molecules **1A** and **1B** is expressed in the respective dihedral angles between their aryl groups and their pseudo-mirror planes (C₁TeC groups). These are 6.04 and 20.30°, respectively. All diethylthiocarbamate ligands are planar, except for the methyl groups, corresponding to the well known *sp*² hybridization of their N and dithiolate C atoms.⁹

Bonding in the complex molecules. The average Te-S and Te-Cl bond lengths in the equatorial plane are 2.676 and 2.679 Å, respectively. This yields an average equatorial Te radius = $l_{\text{Te-Leq}} - r_{\text{Lcov}} = 1.65$ Å, which is close to values found in similar complexes (Table 5) and also to TeL₃X complexes, where X is a halogen or pseudohalogen.^{1,2} The average Te-C bond length is 2.140 Å, slightly longer than

the sum of the covalent radii of Te and C(*sp*²).¹ The diethylthiocarbamate ligands are slightly anisobidentate, with average Te-S bond lengths of 2.648 and 2.704 Å. The corresponding C-S bond lengths are 1.730 and 1.710 Å, values which are normal for bidentate dialkyldithiocarbamates. Also the average C-N bond length of 1.324 Å is normal. A few bond lengths and angles involving the disordered C11-C13 ethyl group and the disordered dioxane molecules are for this reason clearly unreasonable.

Molecular packing. Complex molecules of the same kind are linked in pairs across centres of symmetry by the weak Te-S bonds mentioned above, **1A** across centres of symmetry at 1/2, 1/2, 1/2 and **1B** at 0, 0, 0. The two independent dioxane molecules of solvation are located across centres of symmetry at 1/2, 1/2, 0 and 0, 0, 1/2. There are no other particularly short intermolecular distances between the molecules of the unit cell. The large space available for the dioxanes probably contributes to their disordered nature.

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