

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

Ni—P1	2.223 (2)	P2—C4	1.826 (9)
Ni—P2	2.150 (2)	P2—C5	1.834 (9)
Ni—P3	2.189 (2)	P2—C19	1.83 (1)
Ni—N1	1.896 (6)	P3—C6	1.81 (1)
Ni—N2	2.088 (7)	P3—C21	1.829 (8)
S1—C1	1.587 (8)	P3—C27	1.821 (8)
S2—C2	1.635 (8)	N1—C1	1.131 (9)
P1—C3	1.832 (8)	N2—C2	1.14 (1)
P1—C7	1.795 (9)	C3—C4	1.51 (1)
P1—C13	1.820 (8)	C5—C6	1.47 (1)
P1—Ni—P2	86.20 (9)	Ni—P2—C5	112.0 (2)
P1—Ni—P3	137.3 (1)	Ni—P2—C19	116.1 (3)
P1—Ni—N1	91.2 (2)	C4—P2—C5	107.8 (4)
P1—Ni—N2	101.9 (2)	C4—P2—C19	105.6 (4)
P2—Ni—P3	85.53 (9)	C5—P2—C19	104.7 (4)
P2—Ni—N1	173.7 (2)	Ni—P3—C6	108.2 (3)
P2—Ni—N2	95.0 (2)	C6—P3—C21	107.7 (4)
P3—Ni—N1	92.5 (2)	C6—P3—C27	105.0 (4)
P3—Ni—N2	120.5 (2)	C21—P3—C27	104.2 (3)
N1—Ni—N2	91.2 (3)	Ni—N1—C1	169.9 (6)
Ni—P1—C3	107.6 (3)	Ni—N2—C2	158.1 (6)
Ni—P1—C7	113.6 (3)	S1—C1—N1	177.6 (8)
Ni—P1—C13	117.9 (3)	S2—C2—N2	178.5 (6)
C3—P1—C7	106.3 (4)	P1—C3—C4	107.9 (6)
C3—P1—C13	104.9 (4)	P2—C4—C3	107.3 (6)
C7—P1—C13	105.7 (4)	P2—C19—C20	116.4 (7)
Ni—P2—C4	110.1 (2)		
P2—Ni—P1—C3	-12.0 (3)	Ni—P1—C3—C4	39.2 (6)
P3—Ni—P1—C3	-91.1 (3)	Ni—P2—C4—C3	39.7 (6)
P1—Ni—P2—C4	-12.4 (3)	C5—P2—C4—C3	162.2 (6)
P1—Ni—P2—C5	-132.4 (4)	Ni—P2—C5—C6	16.0 (8)
P3—Ni—P2—C4	125.7 (3)	C4—P2—C5—C6	-105.3 (7)
P3—Ni—P2—C5	5.7 (4)	Ni—P3—C6—C5	38.7 (8)
P1—Ni—P3—C6	57.3 (4)	P1—C3—C4—P2	-48.7 (7)
P2—Ni—P3—C6	-22.1 (4)	P2—C5—C6—P3	-34.0 (9)

Programs used include the Enraf-Nonius SDP (Frenz, 1978), ORTEP (Johnson, 1965) and MULTAN78 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). For (2), the crystal was of relatively poor quality giving rise to higher than expected *R* values and residual densities.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete bond distances, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71031 (62 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1033]

References

- Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- Dubois, D. L., Hyers, W. H. & Meek, D. W. (1975). *J. Chem. Soc. Dalton Trans.* pp. 1011-1015.
- Frenz, B. A. (1978). *The Enraf-Nonius CAD-4 SDP-A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution*. In *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi. Delft Univ. Press.

- Johnson, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Kriege-Simonsen, J. & Feltham, R. D. (1983). *Inorg. Chim. Acta*, **71**, 185-194.
- Laneman, S. A., Fronczek, F. R. & Stanley, G. G. (1988). *J. Am. Chem. Soc.* **110**, 5585-5586.
- Laneman, S. A., Fronczek, F. R. & Stanley, G. G. (1989). *Inorg. Chem.* **28**, 1872-1878.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Rahn, J. A., Delian, A. & Nelson, J. H. (1989). *Inorg. Chem.* **28**, 215-217.
- Spek, A. L., van Eijck, B. P., Jans, R. J. F. & van Koten, G. (1987). *Acta Cryst.* **C43**, 1878-1880.

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Structure of Bis(diethyldithiocarbamato)-diiodotellurium(IV)

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Abstract

Centrosymmetric dimers are formed in the structure through two I atoms bridging two Te atoms. Te is seven-coordinated, bound to four S atoms, two intramolecular axial I atoms and one intermolecular planar bridging I atom, in a distorted pentagonal bipyramidal geometry. The non-bridging I atoms in the dimer have a secondary interaction with similar I atoms of other dimers.

Comment

The dithiocarbamate group $R_2NCS_2^-$ (*L*) is a strong chelating ligand for Te^{IV} . As a result, mixed-ligand complexes of Te^{IV} with dithiocarbamates and halides, such as TeL_3X and TeL_2X_2 (*X* = halides) were considered difficult to prepare. It was reasoned that by suitably substituting electron-withdrawing groups on the N atom of the dithiocarbamate group, the ligand could be made to bond weakly and

become displaced more easily by other ligands. $\text{Te}^{\text{IV}}(\text{deadtc})_3\text{I}$ and $\text{Te}^{\text{IV}}(\text{deadtc})_2\text{I}_2$ [deadtc = bis(2-hydroxyethyl)dithiocarbamate] were thus prepared (Sejekan, Janakiram & Aravamudan, 1978). Subsequently, it has been found that the complexes of the type $\text{Te}^{\text{IV}}\text{L}_3\text{I}$ and $\text{Te}^{\text{IV}}\text{L}_2\text{I}_2$ could be prepared even with the ligands $L = \text{dedtc}$ ($R = \text{C}_2\text{H}_5$) and di-pdte ($R = i\text{-C}_3\text{H}_7$) by controlled oxidation of TeL_4 with I_2 . The crystal structure of $\text{Te}(\text{deadtc})_2\text{I}_2$ has been studied (Appa Rao, Seshasayee, Aravamudan & Radha, 1983) and found to be dimeric with two μ -iodo bridges. The crystal structure of $\text{Te}^{\text{IV}}(\text{di-pdte})_2\text{I}_2$ (Kumar, Aravamudan & Seshasayee, 1991) was found to be monomeric and different from $\text{Te}^{\text{IV}}(\text{deadtc})_2\text{I}_2$.

Fig. 1. gives an ORTEP (Johnson, 1965) plot of the molecule as a dimer. The title compound assumes a centrosymmetric structure as shown in Fig. 1. Each Te atom exhibits a distorted pentagonal bipyramidal coordination featuring two axial I atoms and an equatorial girdle made of four S atoms and a weakly bridging I atom, which is axially bound to the other Te atom in the dimer. There are three types of Te—I interactions, all at distances greater than 2.70 Å, the sum of single covalent radii of Te and I. The Te—I distances are 2.835 (2) [Te—I(1) non-bridging], 3.048 (2) [Te—I(2) bridging] and 3.380 (2) Å [Te—I(2') bridging]. The non-bridging I atoms in each dimer have a secondary interaction [at 3.498 (2) Å] with similar I atoms of other dimers. The structure of the title compound is very similar to that of bis[bis(2-hydroxyethyl)dithiocarbamato]diiodotellurium(IV) (Appa Rao, Seshasayee, Aravamudan & Radha, 1983). On the other hand, only a monomeric structure featuring a six-coordinated Te atom with I—Te—I angle of 104.8° prevails in diiodo-(diisopropyl)dithiocarbamato)tellurium(IV) (Kumar, Aravamudan & Seshasayee, 1991). Interestingly, in the solution phase in dichloromethane, the title compound also exists in monomeric form as revealed by vapour-pressure osmometry.

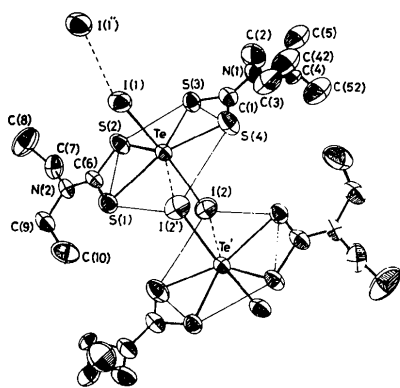


Fig. 1. An ORTEP (Johnson, 1965) plot of TeL_2I_2 showing the dimeric nature of the molecule with 50% thermal ellipsoids.

The dithiocarbamates are highly anisobidentate with the Te—S distances varying from 2.544 (3) to 2.788 (3) Å. The S atom with the weaker Te—S interaction has a shorter bond with C and *vice versa*. This is a common feature in the Te^{IV} dithiocarbamate complexes. The CNCS_2Te moieties in the compound are planar. One of the dithiocarbamate groups is disordered at the C(4) and C(5) positions. Such disorder in dithiocarbamate and xanthate groups has been reported previously (Henrick, Raston & White, 1976; Hoskins & Pannan, 1976; Hoskins, Oliver & Winter, 1984).

Experimental

Crystal data

$[\text{Te}\{(\text{C}_2\text{H}_5)_2\text{NCS}_2\}_2\text{I}_2]$

$M_r = 677.9$

Triclinic

$P\bar{1}$

$a = 10.809$ (3) Å

$b = 10.947$ (6) Å

$c = 10.955$ (4) Å

$\alpha = 108.02$ (4)°

$\beta = 93.66$ (3)°

$\gamma = 118.44$ (3)°

$V = 1048.78$ Å³

$Z = 2$

$D_x = 2.14$ Mg m⁻³

$D_m = 2.16$ Mg m⁻³

Density measured by flotation

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 24 reflections

$\theta = 8\text{--}24.6^\circ$

$\mu = 4.435$ mm⁻¹

$T = 298$ K

Stubby needles

$0.5 \times 0.5 \times 0.4$ mm

Deep red

Data collection

CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

not applied

5560 measured reflections

4805 independent reflections

4049 observed reflections

$[I > 3\sigma(I)]$

$R_{\text{int}} = 0.04$

$\theta_{\text{max}} = 27.5^\circ$

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -1 \rightarrow 14$

3 standard reflections

frequency: 120 min

intensity variation: <3%

Refinement

Refinement on F

Final $R = 0.042$

$wR = 0.051$

$S = 0.995$

4049 reflections

170 parameters

$w = 1/(\sigma^2|F_o| + 0.001943|F_c|^2)$

$(\Delta/\sigma)_{\text{max}} = 0.11$

$\Delta\rho_{\text{max}} = 1.34$ e Å⁻³

$\Delta\rho_{\text{min}} = -1.43$ e Å⁻³

Atomic scattering factors

from Cromer & Liberman

(1970)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic thermal parameters (Å²)

C(4)/C(42) and C(5)/C(52) were refined isotropically with final occupancy factors of 0.2/0.8 and 0.3/0.7, respectively. For other non-H atoms

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}/U_{\text{eq}}$
Te	0.09179 (3)	0.23976 (4)	0.16025 (3)	0.0366 (2)
I(1)	0.35614 (4)	0.41702 (6)	0.36099 (4)	0.0586 (2)
I(2)	-0.18020 (4)	0.02714 (5)	-0.06900 (5)	0.0532 (2)
S(1)	0.2416 (2)	0.2847 (2)	-0.0283 (2)	0.0483 (7)
S(2)	0.1350 (2)	0.4731 (2)	0.1218 (2)	0.0474 (7)

S(3)	-0.0476 (2)	0.3270 (2)	0.3083 (2)	0.0491 (8)
S(4)	-0.0258 (2)	0.0701 (2)	0.3123 (2)	0.0616 (11)
C(1)	-0.0944 (7)	0.1824 (7)	0.3644 (7)	0.0551 (32)
N(1)	-0.1816 (8)	0.1670 (7)	0.4457 (7)	0.0809 (41)
C(2)	-0.2503 (12)	0.2580 (10)	0.4789 (11)	0.0931 (64)
C(3)	-0.3917 (12)	0.1890 (13)	0.3697 (15)	0.1121 (80)
C(6)	0.2286 (6)	0.4415 (7)	0.0047 (5)	0.0417 (26)
N(2)	0.2805 (5)	0.5316 (6)	-0.0582 (5)	0.0458 (24)
C(7)	0.2857 (7)	0.6784 (8)	-0.0152 (8)	0.0566 (36)
C(8)	0.4311 (9)	0.8042 (10)	0.0795 (14)	0.1040 (59)
C(9)	0.3465 (7)	0.4999 (9)	-0.1692 (7)	0.0595 (39)
C(10)	0.2183 (12)	0.3951 (13)	-0.2994 (9)	0.1063 (64)
C(4)	-0.2792 (26)	0.0201 (26)	0.4578 (24)	0.0341 (46)
C(5)	-0.2116 (30)	0.0810 (32)	0.6071 (31)	0.0749 (67)
C(42)	-0.1957 (15)	0.0560 (16)	0.5208 (15)	0.1007 (37)
C(52)	-0.3258 (16)	-0.0983 (18)	0.4319 (17)	0.1015 (43)

The refined site occupancy factors are C(4) 0.2, C(42) 0.8, C(5) 0.3 and C(52) 0.7. H atoms could not be located. Peaks with heights $> 1 \text{ e } \text{Å}^{-3}$ in $\Delta\rho$ maps turned out to be ripples near the heavy atoms.

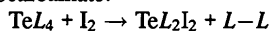
Lists of structure factors and anisotropic thermal parameters, as well as a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55902 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1024]

Table 2. Geometric parameters (Å, °)

Te—I(1)	2.835 (2)	C(2)—N(1)	1.480 (17)
Te—I(2)	3.048 (2)	C(4)—N(1)	1.500 (26)
Te—S(1)	2.726 (2)	C(42)—N(1)	1.623 (22)
Te—S(2)	2.544 (3)	C(2)—C(3)	1.564 (17)
Te—S(3)	2.556 (2)	C(6)—N(2)	1.306 (9)
Te—S(4)	2.788 (3)	N(2)—C(7)	1.500 (11)
Te—I(2')	3.380 (2)	N(2)—C(9)	1.481 (10)
S(1)—C(6)	1.717 (9)	C(7)—C(8)	1.513 (10)
S(2)—C(6)	1.736 (7)	C(9)—C(10)	1.579 (11)
S(3)—C(1)	1.737 (9)	C(4)—C(5)	1.537 (40)
S(4)—C(1)	1.696 (9)	C(42)—C(52)	1.524 (16)
C(1)—N(1)	1.326 (11)		
I(1)—Te—I(2)	173.8 (1)	Te—S(3)—C(1)	90.8 (3)
I(1)—Te—S(1)	90.5 (1)	Te—S(4)—C(1)	84.1 (3)
I(1)—Te—S(2)	90.1 (1)	S(1)—C(6)—S(2)	117.2 (4)
I(1)—Te—S(3)	94.0 (1)	S(1)—C(6)—N(2)	122.6 (5)
I(1)—Te—S(4)	88.1 (1)	S(2)—C(6)—N(2)	120.1 (5)
I(1)—Te—I(2')	86.5 (1)	C(6)—N(2)—C(7)	121.4 (5)
I(2)—Te—S(1)	86.3 (1)	C(6)—N(2)—C(9)	122.0 (5)
I(2)—Te—S(2)	93.6 (1)	N(2)—C(7)—C(8)	110.2 (7)
I(2)—Te—S(3)	91.7 (1)	N(2)—C(9)—C(10)	106.8 (6)
I(2)—Te—S(4)	91.8 (1)	C(7)—N(2)—C(9)	116.4 (5)
I(2)—Te—I(2')	87.5 (1)	N(1)—C(4)—C(5)	93.9 (18)
S(1)—Te—S(2)	67.9 (1)	C(2)—N(1)—C(4)	107.5 (11)
S(1)—Te—S(3)	145.0 (1)	C(2)—N(1)—C(42)	120.8 (8)
S(1)—Te—S(4)	148.2 (1)	C(4)—N(1)—C(42)	34.3 (11)
S(1)—Te—I(2')	72.8 (1)	S(3)—C(1)—S(4)	118.1 (4)
S(2)—Te—S(3)	77.3 (1)	S(3)—C(1)—N(1)	117.8 (5)
S(2)—Te—S(4)	143.8 (1)	S(4)—C(1)—N(1)	124.1 (5)
S(2)—Te—I(2')	140.6 (1)	C(1)—N(1)—C(2)	122.4 (7)
S(3)—Te—S(4)	66.7 (1)	C(1)—N(1)—C(4)	123.4 (10)
S(3)—Te—I(2')	142.1 (1)	C(1)—N(1)—C(42)	116.5 (7)
S(4)—Te—I(2')	75.4 (1)	N(1)—C(2)—C(3)	111.2 (9)
Te—S(1)—C(6)	84.7 (3)	N(1)—C(42)—C(52)	105.4 (11)
Te—S(2)—C(6)	90.1 (3)		

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

Te^{IV}(dedtc)₂I₂ was synthesized by controlled oxidative displacement of the dithiocarbamate:



where L-L is tetraethylthiuram disulfide. 1 mM (0.254 g) of iodine, dissolved in dichloromethane, was added to 1 mM (0.677 g) of TeL₄, dissolved in 25 ml of dichloromethane, with constant stirring for a few minutes. The solution gave a gummy mass on slow evaporation. Washing with carbon tetrachloride removed the much more soluble disulfide. Recrystallization of TeL₂I₂ using a mixture of dichloromethane and ethyl acetate yielded tiny deep-red crystals. Elemental analysis: found C 18.10, H 2.90, N 4.04, S 18.64, Te 19.12 wt%; calculated [for Te(C₁₀H₂₀N₂S₄)I₂]: C 17.72, H 2.97, N 4.13, S 18.91, Te 18.82 wt%. The structure was solved using SHELX76 (Sheldrick, 1976). Two of the ethyl carbon atoms, namely C(4) and C(5), showed positional disorder. They were isotropically refined with two positions for each [C(4), C(42) and C(5), C(52)].

References

- Appa Rao, G. V. N., Seshasayee, M., Aravamudan, G. & Radha, K. (1983). *Acta Cryst.* **C39**, 1018–1020.
- Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- Henrick, K., Raston, C. L. & White, A. H. (1976). *J. Chem. Soc. Dalton Trans.* pp. 26–28.
- Hoskins, B. F., Oliver, P. J. & Winter, G. (1984). *Inorg. Chim. Acta*, **86**, L21–L24.
- Hoskins, B. F. & Pannan, C. D. (1976). *Aust. J. Chem.* **29**, 2337–2348.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Kumar, V., Aravamudan, G. & Seshasayee, M. (1991). *J. Crystallogr. Spectrosc. Res.* **21**, 65–68.
- Sejekan, N. B. G., Janakiram, C. & Aravamudan, G. (1978). *J. Inorg. Nucl. Chem.* **40**, 211–213.
- Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Bis(4-hydroxymethyl-1,5-dimethylimidazole-N³)gold(I) Chloride

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Abstract

The complex, bis(1,5-dimethylimidazole-4-methanol-N³)gold(I) chloride, is a salt in which the ligand molecules are bound through N(3) atoms of the imidazole rings to the Au atom, which has an approximate inversion centre. Bond lengths and angles are normal.