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References

ATOVMYAN, L. O., BLEIDELIS, YA. YA., KEMME, A. A. & SHIBAEVA, R. P. (1970). *J. Struct. Chem. (USSR)*, **11**, 295–298.

FRENZ, B. A. (1985). *SDP-Plus Structure Determination Package*, version 3.0. Enraf–Nonius, Delft, The Netherlands.

GLIDEWELL, C. & LILES, D. C. (1983). *J. Organomet. Chem.* **243**, 291–297.

GURKOVA, S. N., GUSEV, A. I., SEGEL'MAN, I. R., ALEKSEEV, N. V., GAR, T. K. & KHROMOVA, N. V. (1981). *J. Struct. Chem. (USSR)*, **22**, 461–462.

HILLER, W., STRÄHLE, J., MITULLA, K. & HANACK, M. (1980). *Justus Liebigs Ann. Chem.* pp. 1946–1951.

HUBER, F., HAUPT, H.-J., PREUT, H., BARBIERI, R. & LO GIUDICE, M. T. (1977). *Z. Anorg. Allg. Chem.* **432**, 51–57.

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KELLER, E. (1987). *SCHAKAL. A Fortran Program for the Graphic Representation of Molecular and Crystallographic Models*. Univ. of Freiburg, Federal Republic of Germany.

KEMME, A. A., BLEIDELIS, YA. YA., SHIBAEVA, R. P. & ATOVMYAN, L. O. (1973). *J. Struct. Chem. (USSR)*, **14**, 90–93.

KITAIGORODSKII, A. I. (1979). In *Molekülkristalle*. Berlin: Akademie-Verlag.

NARDELLI, M. (1982). *PARST. A System of Computer Routines for Calculating Molecular Parameters from Results of Crystal Structure Analysis*. Univ. of Parma, Italy.

PREUT, H., MUNDUS, B., HUBER, F. & BARBIERI, R. (1986). *Acta Cryst.* **C42**, 536–538.

SHELDRICK, G. M. (1976). *SHELX76. A program for crystal structure determination*. Univ. of Cambridge, England.

SHELDRICK, G. M. (1987). *SHELXTL-Plus*, release 2. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. For Nicolet R3m/V. Univ. of Göttingen, Federal Republic of Germany.

VORNEFELD, M., HUBER, F., PREUT, H. & VON ANGERER, E. (1988). *Appl. Organomet. Chem.* To be published.

VORNEFELD, M., HUBER, F., PREUT, H. & BRUNNER, H. (1989). *Appl. Organomet. Chem.* **3**, 177–182.

Structure of Bis(dipropyldithiocarbamato)tellurium(II)

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Abstract. Bis(dipropylcarbamodithioato-*S,S'*)-tellurium(II), [Te($C_7H_{14}NS_2$)$_2$], $M_r = 480.28$, ortho-rhombic, *Pbca*, $a = 13.587$ (2), $b = 12.908$ (3), $c = 24.468$ (4) Å, $V = 4291.21$ Å^3, $D_m = 1.50$, $D_x = 1.487$ Mg m^{-3}, $Z = 8$, λ(Mo Kα) = 0.71069 Å, $\mu = 1.639$ mm^{-1}, $F(000) = 1936$, $T = 293$ K, final $R = 0.048$, $wR = 0.047$ for 3969 unique reflections. The Te atom is coordinated to four S atoms forming an asymmetric planar complex with Te—S bond distances in the range 2.511 (1)–2.853 (1) Å. Molecules related by a centre of symmetry are bridged through one of the S atoms with a short intermolecular contact Te···S(1') = 3.499 (1) Å. The dithiocarbamate parts of the two ligands are planar.

Introduction. Four sulfur-coordinated tellurium(II) complexes have been shown to prefer a planar configuration around the central Te atom. With mono-dentate ligands such as thiourea and phenylene-thiourea, the complexes are square-planar (Rout, Seshasayee, Aravamudan & Sowrirajan, 1984; Anderson, 1971). The bonding in such square-planar complexes can be explained in terms of three-centre four-electron bonding (Fabiani, Spagna, Vaciago & Zambonelli, 1971; Foss, 1967, 1970). The same behaviour is also followed in most cases by Se^{II} complexes (Husebye & Helland-Madsen, 1970; Rout, Seshasayee, Radha & Aravamudan, 1983).

In all the dithiocarbamate complexes of Te^{II} the four S atoms in the molecule are bonded to the central Te atom in an asymmetric manner, having two short Te—S bonds and two long Te—S bonds. As part of our studies of divalent tellurium and selenium complexes of dithiocarbamate ligands the title compound was prepared and its crystal structure determined.

Experimental. The synthesis of the title compound, TeL_2 (L = dipropyldithiocarbamate), is as follows. TeL_2 was obtained from TeL_4. TeL_4 was initially prepared as a yellow solid on addition of 30 ml of 4 M acetic acid to 50 ml of 0·5 M sodium hydroxide containing 3 mmol of sodium tellurite and 20 mmol of NaL. The precipitate was washed with water and dried in air. 1 mmol (0·831 g) of TeL_4 was dissolved in 5 ml dichloromethane. 10 ml of acetonitrile was added. During evaporation of the solution TeL_4 transformed to TeL_2 and L—L. The latter, which formed as a film, was washed off with acetonitrile leaving behind red crystals of TeL_2 that were only sparingly soluble in acetonitrile.

Single crystal of approximate dimensions 0·05 × 0·10 × 0·25 mm, density measured by flotation; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $2\theta_{max} = 65°$, range of hkl: $h\,0\rightarrow19$, $k\,0\rightarrow18$ and $l\,0\rightarrow31$; two standard reflections monitored every hour showed no significant variation in intensities, 4296 reflections measured; cell parameters determined by least-squares refinement of θ values of 25 reflections in the range $40 < 2\theta < 50°$, ω–2θ scan technique, 3969 unique reflections with $I > 1\cdot5\sigma(I)$; Lorentz and polarization corrections applied, no absorption correction. Te position from Patterson map, successive Fourier maps based on Te position gave the locations of all the non-H atoms. Two peaks of nearly equal height appeared near one of the terminal C-atom positions [C(4) and C(4a)] and were refined isotropically with variable occupancy factors which converged to 0·6 and 0·4, respectively. All the terminal C atoms showed large movements as indicated by their high thermal factors. Disorder is observed in similar compounds for the terminal atoms (Rout, Seshasayee, Radha & Aravamudan, 1983). Full-matrix least-squares refinement on F using $SHELX76$ (Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms except for the disordered C(4) and C(4a) positions which were refined isotropically. 12 H atoms were located from difference Fourier maps and refined isotropically, and 13 H atoms were fixed geometrically and given a fixed isotropic thermal parameter of 0·17 Å² (the average isotropic thermal parameter of the 12 H-atoms refined). The three H atoms attached to the disordered C(4) atom were not fixed. Final R and wR values are 0·048 and 0·047, respectively, for 3969 unique reflections; weighting scheme used $w = k/[\sigma^2(F_o) + g|F_o|^2]$ where $k = 1\cdot2687$ and $g = 0\cdot00064$. $(\Delta/\sigma)_{max}$ for non-H atoms 0·022; maximum height in the final difference Fourier map 0·87 e Å^{-3}, considered to be a ripple around the Te atom; no secondary-extinction corrections; atomic scattering factors for non-H atoms from Cromer & Mann (1968), for H atoms from Stewart, Davidson &

Simpson (1965) and anomalous-dispersion correction factors for non-H atoms from Cromer & Liberman (1970).

Discussion. Fig. 1 shows a plot of the molecule along with the atom-numbering scheme. Fig. 2 gives the packing of the molecules in the unit cell (*PLUTO*, Motherwell, 1978). Table 1 lists the fractional atomic coordinates and equivalent isotropic thermal parameters for non-H atoms. Table 2 gives the bond lengths and bond angles involving non-H atoms.*

In the title compound, the two dipropyldithiocarbamate (L) moieties act as bidentate chelating ligands. The Te atom is bonded to four S atoms of the two ligands. The five atoms form a plane, the maximum deviation from the mean TeS_4 plane being 0·012 (2) Å; the plane also includes the C—N bonds in both ligands. The S_4 coordination around Te can be described as a planar trapezoidal configuration.

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* Lists of calculated and observed structure factors, anisotropic thermal parameters of non-H atoms, bond lengths and angles involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51958 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
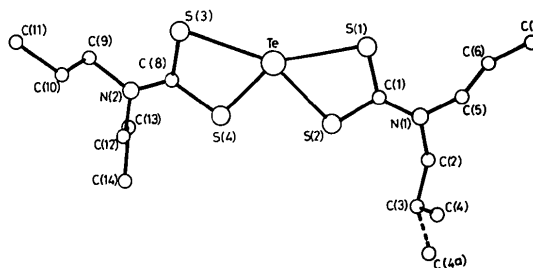

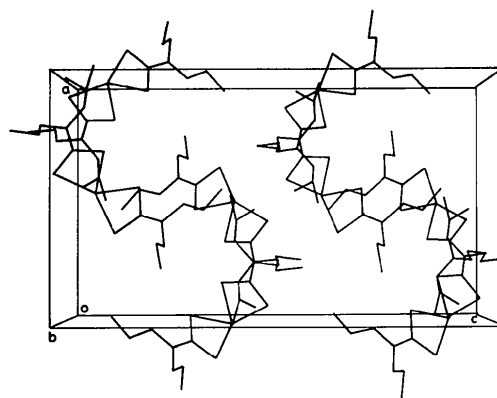Fig. 1. Perspective view of the molecule with the atom-numbering scheme.

Fig. 2. Packing of the molecules in the unit cell.

$[Te(C_7H_{14}NS_2)_2]$

Table 1. Fractional atomic coordinates of non-H atoms and their equivalent isotropic thermal parameters (Å^2) with e.s.d.'s in parentheses

$$U_{eq} = \tfrac{1}{3}\Sigma_{i=1}^{3} U_{ii}.$$

	x	y	z	U_{eq}
Te	−0·01324 (2)	0·48487 (2)	0·10193 (1)	0·0503 (1)
S(1)	−0·0842 (1)	0·6198 (1)	0·0184 (1)	0·0625 (8)
S(2)	−0·1759 (1)	0·5676 (1)	0·1236 (1)	0·0605 (7)
S(3)	0·1378 (1)	0·3504 (1)	0·1369 (1)	0·0834 (10)
S(4)	−0·0429 (1)	0·4087 (1)	0·1951 (1)	0·0639 (8)
C(1)	−0·1796 (3)	0·6342 (4)	0·0618 (2)	0·054 (3)
N(1)	−0·2573 (3)	0·6927 (3)	0·0512 (2)	0·063 (2)
C(2)	−0·3373 (4)	0·7058 (5)	0·0907 (2)	0·082 (4)
C(3)	0·5840 (6)	0·6163 (8)	0·0891 (4)	0·136 (7)
C(4)*	−0·4624 (13)	0·6017 (15)	0·0391 (8)	0·154 (6)
C(4a)*	−0·5127 (18)	0·6009 (25)	0·1064 (10)	0·148 (9)
C(5)	−0·2675 (4)	0·7502 (5)	−0·0004 (2)	0·073 (3)
C(6)	−0·2293 (5)	0·8583 (5)	0·0042 (2)	0·087 (4)
C(7)	−0·2407 (5)	0·9165 (6)	−0·0499 (3)	0·113 (5)
C(8)	0·0672 (4)	0·3411 (4)	0·1936 (2)	0·064 (3)
N(2)	0·0926 (3)	0·2855 (4)	0·2367 (2)	0·080 (3)
C(9)	0·1918 (5)	0·2351 (6)	0·2414 (2)	0·096 (5)
C(10)	0·1850 (6)	0·1287 (6)	0·2218 (3)	0·118 (6)
C(11)	0·2843 (6)	0·0730 (7)	0·2297 (4)	0·153 (8)
C(12)	0·0247 (4)	0·2717 (5)	0·2846 (2)	0·087 (4)
C(13)	0·0455 (6)	0·3468 (6)	0·3294 (3)	0·117 (6)
C(14)	−0·0368 (7)	0·3271 (7)	0·3742 (3)	0·140 (7)

* Refined isotropically with partial occupancy factors 0·6 for C(4) and 0·4 for C(4a).

Table 2. Bond lengths (Å) and bond angles (°) involving non-H atoms

Te—S(1)	2·853 (1)	Te—S(2)	2·511 (1)
Te—S(3)	2·821 (2)	Te—S(4)	2·516 (1)
S(1)—C(1)	1·686 (5)	S(2)—C(1)	1·740 (5)
S(3)—C(8)	1·691 (5)	S(4)—C(8)	1·732 (5)
C(1)—N(1)	1·323 (6)	N(1)—C(2)	1·465 (7)
N(1)—C(5)	1·470 (6)	C(2)—C(3)	1·574 (11)
C(3)—C(4)	1·389 (22)	C(3)—C(4a)	1·394 (26)
C(5)—C(6)	1·493 (9)	C(6)—C(7)	1·529 (9)
C(8)—N(2)	1·321 (7)	N(2)—C(9)	1·502 (9)
N(2)—C(12)	1·503 (7)	C(9)—C(10)	1·459 (11)
C(10)—C(11)	1·541 (12)	C(12)—C(13)	1·489 (10)
C(13)—C(14)	1·585 (12)		
S(1)—Te—S(2)	66·0 (1)	S(1)—Te—S(3)	147·0 (1)
S(1)—Te—S(4)	146·5 (1)	S(2)—Te—S(3)	146·9 (1)
S(2)—Te—S(4)	80·5 (1)	S(3)—Te—S(4)	66·5 (1)
Te—S(1)—C(1)	82·9 (2)	Te—S(2)—C(1)	93·0 (2)
Te—S(3)—C(8)	83·1 (2)	Te—S(4)—C(8)	92·3 (2)
S(1)—C(1)—S(2)	118·1 (3)	S(1)—C(1)—N(1)	123·5 (3)
S(2)—C(1)—N(1)	118·4 (3)	C(1)—N(1)—C(2)	121·9 (4)
C(1)—N(1)—C(5)	122·2 (4)	C(2)—N(1)—C(5)	115·9 (4)
N(1)—C(2)—C(3)	113·8 (6)	C(2)—C(3)—C(4)	115·4 (11)
C(2)—C(3)—C(4a)	137·5 (15)	N(1)—C(5)—C(6)	112·0 (4)
C(5)—C(6)—C(7)	111·1 (5)	S(3)—C(8)—S(4)	118·1 (3)
S(3)—C(8)—N(2)	123·1 (4)	N(2)—C(8)—S(4)	118·8 (4)
C(8)—N(2)—C(9)	122·1 (4)	C(8)—N(2)—C(12)	121·8 (5)
C(9)—N(2)—C(12)	116·1 (4)	N(2)—C(9)—C(10)	109·0 (6)
C(9)—C(10)—C(11)	110·0 (7)	N(2)—C(12)—C(13)	112·4 (5)
C(12)—C(13)—C(14)	105·7 (6)		

This is very similar to that observed in bis(O-ethyl xanthato)tellurium(II) [Te(S₂COEt)₂] (Husebye, 1967), bis(morpholinyldithiocarbamato)tellurium(II) [Te(morphdtc)₂] (Husebye, 1970), bis(N,N-diethyl-dithiocarbamato)tellurium(II) [Te(dedtc)₂] (Fabiani, Spagna, Vaciago & Zambonelli, 1971) and bis[bis(2-hydroxyethyl)dithiocarbamato]tellurium(II) [Te(hedtc)₂] (Rout, Seshasayee, Radha & Aravamudan, 1983).

In TeL_2, there are two short Te—S bonds to S(2) and S(4) subtending an angle of 80·5 (1)° and two long Te—S bonds to S(1) and S(3) subtending an angle of 147·0 (1)°. The two long Te—S bonds occupy adjacent positions forming an asymmetric planar complex. The average Te—S bond length in the TeS₄ group [2·675 (1) Å] agrees well with those observed in monodentate sulfur-containing ligands of Te^{II} (Ault & Husebye, 1978). Owing to the smaller S—S bites in the ligand, the intraligand bite angles, S(1)—Te—S(2) and S(3)—Te—S(4), are much smaller than 90°. The asymmetry in the Te—S bonds can be explained on the basis of a three-centre four-electron bonding mechanism (Fabiani, Spagna, Vaciago & Zambonelli, 1971; Foss, 1967, 1970). For each short Te—S bond, there is a corresponding long S—C bond and vice versa. In the two dithiocarbamate ligands, the C(1)—N(1) and C(8)—N(2) bonds have partial double-bond character.

The two centrosymmetrically related molecules bridge through S(1) and S(1'). The S(1') atom of the centrosymmetrically related molecule approaches the

Te atom at a distance of 3·499 (1) Å. This is considerably shorter than the sum of van der Waals radii of Te and S (3·86 Å), and longer than the sum of their covalent single-bond radii (2·36 Å) (Alcock, 1972). The Te—S(1') distance is comparable with the range of values (3·579–3·680 Å) observed in [Te(S₂COEt)₂], [Te(morphdtc)₂] and [Te(dedtc)₂] but longer than that observed in [Te(hedtc)₂] [3·296 (6) Å]. It is interesting to note that in all similar Te^{II} complexes, no increase in Te—S bond length is observed for the bridging S atom. The deviation of S(1') from the mean TeS₄ plane is 1·666 (2) Å and oriented in the direction which makes an angle of 28·4 (2)° with the TeS₄ plane. Atoms taking part in dimer formation [Te, S(1), Te' and S(1')] lie in a plane of a parallelogram. This plane makes an angle of 29·3 (2)° with the TeS₄ plane. The parallel TeS₄ and Te'S₄ planes are about 1·65 (2) Å apart. The two lone pairs of electrons on Te are placed vertically above and below the TeS₄ plane. The crystal packing is stabilized by van der Waals forces.

References

ALCOCK, N. W. (1972). Adv. Inorg. Radiochem. 15, 1–58.
ANDERSON, O. P. (1971). Acta Chem. Scand. 25, 3593–3608.
AULT, H. K. & HUSEBYE, S. (1978). Acta Chem. Scand. Ser. A, 32, 157–162.
CROMER, D. T. & LIBERMAN, D. (1970). J. Chem. Phys. 53, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.

FABIANI, C., SPAGNA, R., VACIAGO, A. & ZAMBONELLI, L. (1971). *Acta Cryst.* B27, 1499–1504.

FOSS, O. (1967). *Selected Topics in Structure Chemistry*, edited by P. ANDERSEN, O. BASTIANSEN & S. FURBERG, p. 145. Oslo: Universitetsforlaget.

FOSS, O. (1970). *Pure Appl. Chem.* 24, 31–48.

HUSEBYE, S. (1967). *Acta Chem. Scand.* 21, 42–52.

HUSEBYE, S. (1970). *Acta Chem. Scand.* 24, 2198–2210.

HUSEBYE, S. & HELLAND-MADSEN, G. (1970). *Acta Chem. Scand.* 24, 2273–2284.

MOTHERWELL, S. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

ROUT, G. C., SESHASAYEE, M., ARAVAMUDAN, G. & SOWRIRAJAN, S. (1984). *Polyhedron*, 3(8), 921–927.

ROUT, G. C., SESHASAYEE, M., RADHA, K. & ARAVAMUDAN, G. (1983). *Acta Cryst.* C39, 1021–1023.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

Structure of Bis[(1,2,3,3a,7a-η)-indenyl]bis(tetrahydroborato)uranium(IV)

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Abstract. [U(BH$_4$)$_2$(C$_9$H$_7$)$_2$], M_r = 498·03, monoclinic, $P2_1/c$, a = 8·636 (6), b = 26·35 (1), c = 7·765 (6) Å, β = 102·690 (1)°, V = 1724 (4) Å^3, Z = 4, D_x = 1·919 g cm^{-3}, λ(Mo $K\overline{\alpha}$) = 0·71073 Å, μ = 89·191 cm^{-1}, $F(000)$ = 928, room temperature, R = 0·062 for 1148 observed reflections. The coordination geometry about the U atom is that of a tetrahedron formed by the two B atoms and the centroids of the five-membered rings of the indenyl ligands. The short U—B distances are characteristic of tridentate ligation by the borohydride ligands. U—C distances suggest a pentahapto covalent bonding to the indenyl ligands.

Introduction. The interest in organometallic tetrahydroborate complexes is mainly due to the bonding mode of the BH$_4$ group which may act as a monodentate, bidentate or tridentate ligand. Only a few structural investigations on actinide complexes are reported in the literature. They are principally concerned with cyclopentadienyl complexes of uranium: [U(C$_5$H$_5$)$_2$(BH$_4$)$_2$] (Zanella, de Paoli, Bombieri, Zanotti & Rossi, 1977), [U(C$_5$H$_5$)(BH$_4$)$_3$] (Baudry, Charpin, Ephritikhine, Folcher, Lambard, Lance,

Nierlich & Vigner, 1985). We report here the structure of an idenyl derivative in order to determine whether the mode of bonding of the BH$_4^-$ anion is influenced by the nature of the other ligands coordinated to the metal.

Experimental. Bis(indenyl)bis(tetrahydroborato)-uranium was prepared by reaction of indenyl (sodium) with uranium tetrahydroborate. Dark-brown prismatic single crystals obtained by crystallization in pentane. The specimen (0·2 × 0·2 × 0·3 mm) selected for X-ray analysis was sealed in a thin-walled glass capillary. Intensity data recorded on an Enraf–Nonius CAD-4 X-ray diffractometer, graphite-monochromated Mo $K\overline{\alpha}$ radiation. Cell parameters refined by least squares from angle data of 25 reflections (10 ≤ 2θ ≤ 23°). Space group determined from systematic absences. 2153 unique reflections measured by ω–2θ method in range 4 ≤ 2θ ≤ 45°; 1836 with I ≥ 1$\sigma(I)$($\pm h,k,l$). Intensities of three standard reflections measured at 30 min intervals showed no significant deviations from mean. Intensities corrected for Lorentz–polarization effects; empirical absorption correction using the program *DIFABS* (Walker & Stuart, 1983), transmission coefficients: min. 0·53 and max. 1·52. Structure solved by

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