(Seiler & Dunitz, 1979) and ferrocene derivatives (Krukonis, Silverman & Yannoni, 1972; Hall & Brown, 1971; Palenik, 1969; Palenik, 1970).

Between the molecules weak hydrogen bonding (Berkovitch-Yellin & Leiserowitz, 1984) is observed: H(28)···O(11) $(1-x, 1-y, 1-z) = 2\cdot41$ (3) Å [sum of the van der Waals radii is 2·72 Å (Bondi, 1964)], the angle C(28)—H(28)···O(11) = 127 (2)°, H(33)···F(31) $(-x, -1-y, -z) = 2\cdot47$ (5) Å [sum of the van der Waals radii is 2·67 Å], C(33)—H(33)···F(31) = 131 (3)°, and H(210)···O(22) $(\frac{1}{2}-x, \frac{1}{2}+y, 1-z) =$ 2·49 (3) Å, C(210)—H(210)···O(22) = 133 (2)°.

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Structure of Di(*tert*-butyl)bis(*N*,*N*-dimethyldithiocarbamato)tin(IV)

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Abstract. $[Sn(C_4H_9)_2(C_3H_6NS_2)_2]$, $M_r = 473\cdot35$, monoclinic, $P2_1/c$, $a = 8\cdot405$ (2), $b = 15\cdot020$ (4), $c = 16\cdot377$ (5) Å, $\beta = 96\cdot01$ (1)°, $V = 2056$ (1) Å^3, $Z = 4$, $D_x = 1\cdot529$ g cm^{-3}, graphite-monochromated Mo $K\alpha$, $\lambda(K\alpha_1) = 0\cdot7093$ Å, $\mu = 16\cdot34$ cm^{-1}, $F(000) = 966\cdot30$ (including anomalous dispersion), $T = 113$ K, $R(F^2) = 0\cdot033$ for 4291 unique reflections. The Sn atom is five-coordinate: one of the *N*,*N*-dimethyldithiocarbamate (dtc) ligands acts as a bidentate ligand while the other is monodentate. The coordination geometry around the Sn atom is a distorted trigonal bipyramid: two C atoms of the *tert*-butyl groups and one S atom of the bidentate dtc ligand are equatorial while the other two S atoms are axial.

Introduction. Organotin(IV) dithiocarbamate complexes have demonstrated a variety of coordination geometries around the Sn atom, with coordination numbers ranging from 4 to 7 (Zubieta & Zuckerman, 1978). The coordination geometry depends upon the bonding mode of the dithiocarbamate moiety, which acts either as a monodentate or as a bidentate ligand. In

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a continuing effort to understand the coordination chemistry of such complexes we studied and report here the crystal structure of di(*tert*-butyl)bis(*N*,*N*-dimethyldithiocarbamato)tin(IV). From ^{119}Sn NMR spectroscopy, Otera, Yano & Kusakabe (1983) suggested that in this compound the Sn center is five-coordinate.

Experimental. The title compound was prepared by a literature procedure (Otera, Yano & Kusakabe, 1983). Analysis: calc. for $C_{14}H_{30}N_2S_4Sn$: C, 35·52; H, 6·39; N, 5·92; found: C, 35·70; H, 6·64; N, 5·66%. A colorless crystal, 0·33 × 0·32 × 0·21 mm, was mounted in the cold stream (113 K) of a Picker FACS-1 diffractometer; 19 reflections ($15 \leq 2\theta \leq 25°$) were used for measuring lattice parameters; max. $\lambda^{-1} \sin\theta = 0·6346$ Å^{-1}; $+h$ (0 to 10), $+k$ (0 to19), $\pm l$ (−20 to 20); six standard reflections measured after every 100 reflections showed no systematic variations in intensity; 4772 measured intensities, 4291 unique, 3805 unique observed [$I > 3\sigma(I)$]; $p = 0·03$ for calculation of $\sigma(F_o^2)$; analytical absorption correction applied with a transmission-factor range of 0·663–0·741; solution by Patterson and Fourier techniques; initial full-matrix least-squares refinement on F; H atoms located in difference electron density map and then idealized with C—H = 0·95 Å; final cycle of refinement on F^2, 191 refined parameters; $R(F^2) = 0·033$, $wR(F^2) = 0·060$, $w = 1/\sigma^2(F^2)$, $S = 1·18$, max. $\Delta/\sigma = 0·73$ in final cycle of refinement, $R(F)$, $F_o^2 > 3\sigma(F_o^2) = 0·027$; max. height in final difference electron density map 0·57 e Å^{-3}; atomic scattering factors for non-H atoms from Cromer & Waber (1974), for H atoms from Stewart, Davidson & Simpson (1965); anomalous-dispersion corrections for Sn and S atoms from Cromer & Waber (1974). All calculations were carried out on a Harris 1000 computer with methods and programs standard for the laboratory at Northwestern (Waters & Ibers, 1977).

Discussion. Positional and thermal parameters are listed in Table 1, and selected bond distances and angles in Table 2.[*] The molecular geometry and labeling scheme for the complex are shown in Fig. 1.

Contrary to most known structures of diorganotin(IV) dithiocarbamate complexes, one of the two dithiocarbamate ligands in the present complex exhibits a monodentate bonding mode while the other exhibits a bidentate mode, thus resulting in five-coordination around the central Sn^{IV} atom. The bidentate dithiocarbamate ligand is bonded to the Sn atom in an anisobidentate manner: Sn—S(1) and Sn—S(3) are 2·795 (1) and 2·489 (1) Å, respectively; a typical small bite angle [67·56 (2)°] is observed. The distance between the Sn and the noncoordinating S(4) atom is 3·532 (1) Å. The geometry around the Sn center is best described as a distorted trigonal bipyramid: atoms C(7) and C(11) of the *tert*-butyl groups and atom S(3) occupy equatorial positions while atoms S(1) and S(2) are axial. The angle between these axial groups, S(1)—Sn—S(2), is 156·96 (2)°.

Table 1. *Positional parameters and* B_{eq} *values* (Å^2) *for di*(*tert-butyl*)*bis*(*N*,*N-dimethyldithiocarbamato*)*tin*(IV)

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Sn	0·099995 (18)	0·222100 (10)	−0·117527 (9)	1·237 (4)
S(1)	−0·12381 (8)	0·35096 (4)	−0·17554 (4)	2·21 (2)
S(2)	0·27754 (7)	0·13143 (4)	−0·01025 (4)	1·66 (1)
S(3)	0·02204 (7)	0·32057 (4)	−0·00611 (4)	1·57 (1)
S(4)	0·44835 (8)	0·30755 (4)	−0·00524 (4)	2·11 (2)
N(1)	−0·20183 (23)	0·44209 (13)	−0·04605 (13)	1·86 (5)
N(2)	0·54414 (22)	0·16668 (13)	0·08367 (11)	1·60 (5)
C(1)	−0·1139 (3)	0·37820 (15)	−0·07383 (15)	1·57 (5)
C(2)	−0·2024 (3)	0·46117 (16)	0·04134 (16)	2·15 (6)
C(3)	−0·3191 (3)	0·49138 (17)	−0·10093 (18)	2·63 (7)
C(4)	0·4347 (3)	0·20170 (15)	0·02749 (14)	1·46 (5)
C(5)	0·6819 (3)	0·21925 (19)	0·11708 (17)	2·50 (7)
C(6)	0·5413 (3)	0·07389 (17)	0·11012 (15)	2·08 (6)
C(7)	−0·0760 (3)	0·11199 (15)	−0·13534 (14)	1·64 (5)
C(8)	−0·1857 (3)	0·12222 (18)	−0·21501 (17)	2·38 (6)
C(9)	−0·1766 (3)	0·11542 (18)	−0·06317 (17)	2·54 (7)
C(10)	0·0125 (3)	0·02353 (17)	−0·13662 (18)	2·70 (7)
C(11)	0·2489 (3)	0·25356 (20)	−0·21659 (15)	2·31 (6)
C(12)	0·2971 (4)	0·35204 (23)	−0·21494 (19)	3·79 (9)
C(13)	0·1468 (3)	0·23304 (22)	−0·29673 (16)	3·00 (8)
C(14)	0·3954 (3)	0·19324 (25)	−0·20801 (18)	3·51 (8)

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *in di*(*tert-butyl*)*bis*(*N*,*N-dimethyldithiocarbamato*)-*tin*(IV)

Sn—S(1)	2·795 (1)	N(1)—C(3)	1·463 (3)
Sn—S(2)	2·573 (1)	N(2)—C(4)	1·339 (3)
Sn—S(3)	2·489 (1)	N(2)—C(5)	1·460 (3)
Sn—C(7)	2·218 (2)	N(2)—C(6)	1·461 (3)
Sn—C(11)	2·201 (3)	C(7)—C(8)	1·525 (3)
S(1)—C(1)	1·709 (3)	C(7)—C(9)	1·525 (3)
S(3)—C(1)	1·738 (2)	C(7)—C(10)	1·524 (3)
S(2)—C(4)	1·752 (2)	C(11)—C(12)	1·533 (4)
S(4)—C(4)	1·686 (2)	C(11)—C(13)	1·522 (4)
N(1)—C(1)	1·321 (3)	C(11)—C(14)	1·524 (4)
N(1)—C(2)	1·460 (3)		

S(1)—Sn—S(3)	67·56 (2)	C(1)—N(1)—C(3)	121·4 (2)
S(2)—Sn—S(3)	89·56 (3)	C(2)—N(1)—C(3)	115·6 (2)
S(1)—Sn—S(2)	156·96 (2)	C(4)—N(2)—C(5)	120·4 (2)
C(7)—Sn—S(1)	93·21 (6)	C(4)—N(2)—C(6)	123·2 (2)
C(7)—Sn—S(2)	91·50 (6)	C(5)—N(2)—C(6)	116·2 (2)
C(7)—Sn—S(3)	108·45 (7)	Sn—C(7)—C(8)	111·9 (2)
C(7)—Sn—C(11)	119·07 (10)	Sn—C(7)—C(9)	106·8 (2)
C(11)—Sn—S(1)	90·86 (8)	Sn—C(7)—C(10)	109·4 (2)
C(11)—Sn—S(2)	106·44 (7)	C(8)—C(7)—C(9)	109·0 (2)
C(11)—Sn—S(3)	128·77 (8)	C(8)—C(7)—C(10)	109·2 (2)
S(1)—C(1)—S(3)	117·7 (1)	C(9)—C(7)—C(10)	110·6 (2)
N(1)—C(1)—S(1)	122·5 (2)	Sn—C(11)—C(12)	111·3 (2)
N(1)—C(1)—S(3)	119·8 (2)	Sn—C(11)—C(13)	106·2 (2)
S(2)—C(4)—S(4)	122·0 (1)	Sn—C(11)—C(14)	108·7 (2)
N(2)—C(4)—S(4)	121·6 (2)	C(12)—C(11)—C(13)	109·5 (2)
N(2)—C(4)—S(2)	116·4 (2)	C(12)—C(11)—C(14)	111·2 (2)
C(1)—N(1)—C(2)	122·7 (2)	C(13)—C(11)—C(14)	109·7 (2)

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[*] Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44227 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

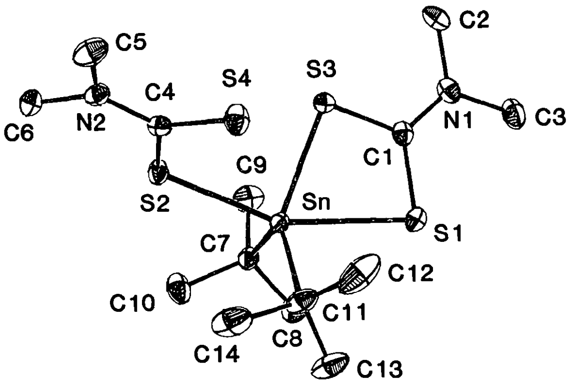

Fig. 1. Drawing of di(*tert*-butyl)bis(*N*,*N*-dimethyldithiocarbamato)-tin(IV) (50% probability thermal ellipsoids) along with the atom-numbering scheme. H atoms are omitted for clarity.

From X-ray studies the dithiocarbamate ligands act as anisobidenate in most of the mono- and diorganotin(IV) dithiocarbamate complexes (Morris & Schlemper, 1978, 1979; Harrison & Mangia, 1976; Lokaj, Kallö, Kettmann, Vrábel & Rattay, 1986; Wei, Kumar Das & Sinn, 1985; Lindley & Carr, 1974; Kimura, Yasuoka, Kasai & Kakudo, 1972; Furue, Kimura, Yasuoka, Kasai & Kakudo, 1970) whereas they behave as monodentate in most of the triorganotin dithiocarbamate complexes (Kumar Das, Wei & Sinn, 1985; Sheldrick & Sheldrick, 1970), except for the di(*tert*-butyl)tin complex of ethylenebis(dithiocarbamate) (Jung, Sohn & Ibers, 1986), where a geometry around the Sn atom similar to the present one was found. The steric effect of the two bulky *cis*-*tert*-butyl ligands may be responsible for the five-coordination of the Sn center in these compounds, although in diphenylbis(*N*,*N*-diethyldithiocarbamato)tin(IV), where the phenyl ligands are *cis*, the Sn center is six-coordinate (Lindley & Carr, 1974).

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The Structure of the Mixed-Ligand Compound [{Bis(1-pyrazolylmethyl)amine}(pyrazole)(tetrafluoroborato)copper(II)] Tetrafluoroborate

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Abstract. $[Cu(C_8H_{11}N_5)(C_3H_4N_2)(BF_4)](BF_4)$, $M_r =$ 482·44, triclinic, $P\bar{1}$, $a =$ 8·489 (1), $b =$ 8·829 (2), $c =$ 13·189 (4) Å, $\alpha =$ 100·02 (2), $\beta =$ 106·19 (2), $\gamma =$ 96·42 (1)°, $V =$ 921·34 Å^3, $Z = 2$, $D_x =$ 1·74 g cm^{-3}, λ(Cu $K\bar{\alpha}$) = 1·54178 Å, $\mu =$ 26·4 cm^{-1}, $F(000) = 482$, $T =$ 293 K, final $R =$ 0·040 for 1712

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