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THE SYNTHESIS AND X-RAY STRUCTURE OF (*N*,*N*-DIMETHYLDITHIOCARBAMATO)(n-BUTYL)DIPHENYLTIN(IV)

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Summary

(*N*,*N*-Dimethyldithiocarbamato)(n-butyl)diphenyltin(IV), n-$BuPh_2SnS_2CNMe_2$, crystallizes in the monoclinic space group $P2_1/n$ with a 9.772(5), b 9.895(4), c 21.418(9) Å, β 95.81(3)°, V 2060 Å^3, $Z = 4$, μ 14.4 cm^{-1}. The structure was determined by the heavy-atom technique from 3103 independent reflections measured at room temperature on an Enraf–Nonius four-circle CAD-4 diffractometer using monochromatized Mo-K_α radiation and refined to a final R value of 5.8%. The tin atom is essentially four-coordinated with a weak fifth tin–sulphur bond (Sn–S(2) 3.079(1) Å) considerably longer than the other (Sn–S(1) 2.466(1) Å). A comparison with the complex n-$BuPhSn(Cl)S_2CNEt_2$ (Sn–S(1) 2.454(1) Å; Sn–S(2) 2.764(1) Å) suggests that enhanced steric factors are responsible for the preferential monodentate behaviour of the dithiocarbamate ligand in the title complex.

Introduction

The title complex is one of several mixed triorganotin(IV) compounds synthesised in our laboratories which has been investigated for its antifungal and insecticidal properties [1]. Although the crystal structures of the analogous symmetrical trimethyltin(IV) [2,3] and triphenyltin(IV) [4] dithiocarbamates are known, our interest in the structural chemistry of the title compound was particularly aroused by the absence of any crystallographic documentation in the literature on compounds containing mixed organic groups on tin. Added to this was the realisation that within a class of closely related compounds subtle structural variations could influence a change in the coordination status and/or geometry at the metal centre, as has been nicely demonstrated in the case of the symmetric triorganotin(IV) carboxylates [5–7]. Mixed organotin compounds, particularly those containing both aryl and alkyl groups on tin, appeared to us to be especially worthy of structural attention in that they would allow a detailed appraisal of the stereochemical preferences of these groups in trigonal-bipyramidal and octahedral configurations.

In earlier papers in this series we have, for example, reported on the complex n-BuPhSn(Cl)S_2CNEt_2 [8], which has trigonal-bipyramidal geometry with equatorial locations of the R groups, and on the octahedral complex bis(2-methylquinolin-8-olato)(ethyl)-n-propyltin [9], which has a sterically induced *trans*-R_2Sn skeletal geometry as opposed to the *cis*-structure previously documented for bis(quinolin-8-olato)dimethyltin [10]. In this paper we report the synthesis and structure of n-$BuPh_2SnS_2CNMe_2$. The metal environment in the title compound differs from that of n-BuPhSn(Cl)S_2CNEt_2 in having the chloro group replaced by the bulkier phenyl group. This change, however, results in significant elongation of the Sn–S(2) bond (vide infra).

Experimental

Preparation of complex

Equimolar (0.002 mol) quantities of n-$C_4H_9(C_6H_5)_2SnBr$ [11] and $Tl(S_2CNMe_2)$ [12], dissolved in chloroform, were stirred together at room temperature for 2 h. The precipitated TlBr was filtered off using a Kieselguhr filter-cake and the filtrate concentrated to obtain the crude product (0.62 g, ca. 70%). Recrystallization from benzene yielded colourless crystals of n-$BuPh_2SnS_2CNMe_2$. These were dried in vacuo (m.p. 97–98°C, with decomposition). Found: C, 50.67; H, 5.51; S, 14.1. $C_{19}H_{25}NS_2Sn$ calcd.: C, 50.70; H, 5.55; S, 14.23%. The microanalyses were performed by the Australian Microanalytical Service, Melbourne.

Infrared spectrum

The Infrared spectrum of the compound, recorded as a Nujol mull on KBr plates using a Perkin–Elmer 1330 spectrometer, revealed bands (cm^{-1}) at 1507 vs (ν(C$\dashv$N)), 980s (ν(C$\dashv$S) + ν(C$\dashv$N)), 385 m (ν(Sn–S)), which were assigned as indicated.

Mössbauer spectrum

The ^{119m}Sn Mössbauer spectrum of the compound was procured at 80 K using a constant acceleration microprocessor spectrometer (from Cryophysics Ltd., Oxford) with a 512-channel data store, and a 15 mCi Ca $^{119m}SnO_3$ source at room temperature. The spectrum, recorded on a Hewlett–Packard 7225B Plotter, was fitted with Lorentzian functions by a least squares fitting programme [13]. The following data (mm s^{-1}; error: ± 0.01) were obtained: Isomer shift, 1.25; Quadrupole splitting, 1.89; $\Gamma_1 = 1.02$; $\Gamma_2 = 1.03$. The isomer shift value is quoted relative to $BaSnO_3$.

Cell measurements and data collection

Cell dimensions and space group data (Table 1) were obtained by standard methods on a Enraf–Nonius four-circle CAD-4 diffractometer using graphite-monochromated Mo-K_α radiation. The θ–2θ scan technique was used, as previously described [14], to record the intensities for all nonequivalent reflections for which $1.5° < 2\theta < 54°$. Scan widths were calculated as $(A + B \tan \theta)°$, where A is estimated from the mosaicity of the crystal and B allows for the increase in peak

TABLE 1

CRYSTALLOGRAPHIC DETAILS FOR n-BuPh$_2$SnS$_2$CNMe$_2$

Formula wt	449.7
Cryst. system	monoclinic
a; b; c (Å)	9.772(5), 9.895(4), 21.418(9)
β (deg)	95.81(3)
V (Å^3)	2060
Space group	$P2_1/n$
Z	4
ρ(calcd) (g cm^{-3})	1.45
Crystal dimensions [a]	(100) 0.0975, ($\bar{1}$00) 0.0975, (011) 0.20, (0$\bar{1}\bar{1}$) 0.20, (10$\bar{2}$) 0.225, ($\bar{1}$02) 0.225 (001) 0.225, (00$\bar{1}$) 0.225
μ(Mo-K_α) (cm^{-1})	14.4
Transmission coefft, max/min	0.845, 0.684
Scan type	θ–2θ

[a] Distances of the faces given from the crystal centroid, in mm.

width owing to $K_{\alpha1}$-$K_{\alpha2}$ splitting. The values of A and B were 0.6 and 0.35, respectively.

The intensities of three standard reflections showed no greater fluctuations during the data collection than those expected from Poisson statistics. The raw intensity data were corrected for Lorentz-polarization and absorption. Of the 3536 independent intensities, there were 3103 with $F_0^2 > 3\sigma(F_0^2)$, where $\sigma(F_0^2)$ was estimated from counting statistics [15]. These data were used in the final refinement of the structural parameters.

Structure determination and refinement

A three-dimensional Patterson map was used to obtain the coordinates of the tin atom. This was followed by a series of structure factor calculations and difference-Fourier maps which revealed the positions of all non-hydrogen atoms. Full-matrix, least-squares refinement was carried out as previously described [14]. Anisotropic temperature factors were introduced for the non-hydrogen atoms. Further Fourier-difference functions permitted location of the non-methyl hydrogen atoms, which were included in the refinement for four cycles of least squares and then held fixed. The model converged with R 5.8% and R_w 6.2%. A final Fourier-difference map was featureless. Final atomic coordinates and thermal parameters are listed in Table 2. A listing of observed and and calculated structure factors may be obtained from the authors.

Results and discussion

The Bonati–Ugo criterion [16] for discerning the chelating bonding mode of the dithiocarbamate ligand from the location of its strong ν_{as}(CS) band in the IR at ca. 1000 cm^{-1} (at 980 cm^{-1} in the title complex), has recently been criticized [17]. It appears from the available X-ray diffraction data that the ligand has an ubiquitous C_1 site symmetry irrespective of its bonding type or the host complex, and that

TABLE 2

FINAL FRACTIONAL ATOMIC COORDINATES AND ANISOTROPIC THERMAL PARAMETERS [a], WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES, FOR NON-HYDROGEN ATOMS

Atom	x/a	y/b	z/c	Atom	x/a	y/b	z/c
Sn	0.28815(7)	0.19649(6)	0.10842(3)	C(16)	0.5835(11)	0.0862(10)	0.1172(5)
S(1)	0.2311(3)	0.4133(3)	0.0552(1)	C(21)	0.1826(9)	0.2416(9)	0.1893(4)
S(2)	0.4049(3)	0.2397(3)	−0.0183(1)	C(22)	0.2030(10)	0.1603(11)	0.2425(5)
N	0.3070(9)	0.4792(8)	−0.0559(4)	C(23)	0.1421(11)	0.1846(12)	0.2962(5)
C(N)	0.3156(9)	0.3834(9)	−0.0123(4)	C(24)	0.0558(11)	0.2926(12)	0.2986(5)
C(M1)	0.3752(13)	0.4625(13)	−0.1138(5)	C(25)	0.0313(10)	0.3748(10)	0.2473(5)
C(M2)	0.2300(13)	0.6072(10)	−0.0488(5)	C(26)	0.0935(10)	0.3496(9)	0.1933(4)
C(11)	0.4981(9)	0.1747(8)	0.1443(4)	C(1b)	0.2034(12)	0.0180(11)	0.0625(5)
C(12)	0.5519(10)	0.2388(10)	0.2002(5)	C(2b)	0.1134(11)	−0.0623(11)	0.1027(5)
C(13)	0.6878(12)	0.2135(12)	0.2246(5)	C(3b)	0.0700(14)	−0.2010(12)	0.0762(6)
C(14)	0.7684(11)	0.1283(12)	0.1950(6)	C(4b)	−0.0179(14)	−0.2809(13)	0.1170(7)
C(15)	0.7168(10)	0.0617(12)	0.1415(5)				

Atom	B_{11} or B	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Sn	4.90(2)	3.73(2)	4.06(2)	0.18(3)	0.8(2)	−0.05(2)
S(1)	7.6(1)	4.5(1)	4.2(1)	1.0(1)	1.44(9)	0.39(9)
S(2)	7.6(1)	5.3(1)	5.5(1)	1.3(1)	1.92(10)	0.13(10)
N	6.7(4)	4.5(3)	4.5(3)	−0.7(3)	0.9(3)	0.5(3)
C(N)	4.9(4)	4.4(4)	4.3(4)	−0.1(4)	0.7(3)	0.0(3)
C(M1)	10.7(7)	7.4(6)	5.2(4)	−1.2(6)	3.8(4)	0.7(5)
C(M2)	8.9(7)	3.9(4)	7.1(6)	0.9(5)	1.3(5)	1.0(4)
C(11)	4.3(4)	3.2(3)	5.1(4)	−0.1(3)	1.1(3)	0.4(3)
C(12)	5.7(5)	4.9(4)	5.2(4)	0.0(4)	0.9(4)	0.6(4)
C(13)	7.6(6)	6.2(6)	5.5(5)	−1.2(5)	−0.6(5)	0.7(5)
C(14)	5.0(5)	6.9(6)	8.1(6)	−0.0(5)	0.5(5)	1.4(5)
C(15)	4.8(4)	6.2(5)	7.9(6)	1.2(4)	1.4(4)	0.9(5)
C(16)	6.2(5)	4.3(4)	5.3(4)	−0.1(4)	1.1(4)	0.1(4)
C(21)	4.1(4)	4.0(4)	4.4(4)	−0.0(3)	0.9(3)	−0.0(3)
C(22)	4.6(4)	6.1(5)	5.6(4)	0.5(4)	1.3(4)	0.6(4)
C(23)	5.9(5)	7.3(6)	4.9(4)	0.7(5)	1.0(4)	1.2(5)
C(24)	6.7(5)	7.0(6)	5.0(4)	−1.5(5)	2.0(4)	−1.1(4)
C(25)	5.5(4)	4.4(4)	7.6(5)	−0.3(4)	2.4(4)	−1.1(4)
C(26)	6.2(5)	3.8(4)	5.2(4)	0.1(4)	1.3(4)	0.4(3)
C(1b)	8.5(6)	5.3(5)	4.8(4)	−0.8(5)	−0.0(5)	−1.2(4)
C(2b)	5.9(5)	5.2(5)	7.0(5)	0.2(4)	1.2(4)	−0.9(4)
C(3b)	9.0(7)	5.3(5)	8.4(7)	0.2(6)	−1.7(6)	−1.6(5)
C(4b)	8.3(7)	5.6(6)	10.7(8)	−1.7(5)	1.7(6)	0.6(6)

[a] The form of the anisotropic thermal parameter is: $\exp -(B_{11}a^*a^*hh + B_{22}b^*b^*kk + B_{33}c^*c^*ll)/4 + (B_{12}a^*b^*hk + B_{13}a^*c^*hl + B_{23}b^*c^*kl)/2$.

further the C–N modes also arise in the 1000 cm^{-1} region [17]. While this ruled out the IR technique as a means of determining the ligand bonding type in the present complex, a study of the Mössbauer spectrum of the complex, not unexpectedly, revealed little information concerning the coordination status of the metal atom. Thus relative to the five-coordinated [8] complex, n-BuPhSn(Cl)S_2CNEt_2, for which the Isomer shift (*IS*) and Quadrupole splitting (*QS*) values are respectively 1.41 and 2.66 mm s^{-1} [11], the observed Mössbauer parameters for the title complex are

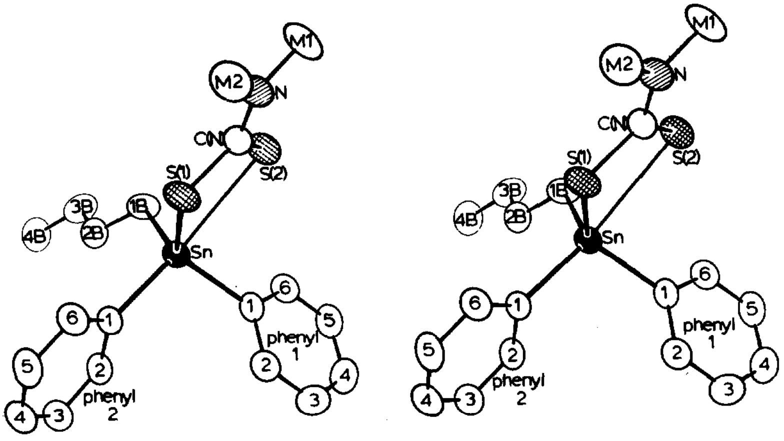

Fig. 1. A stereoview of the [n-BuPh$_2$SnS$_2$CNMe$_2$] molecule.

$IS = 1.25$ and $QS = 1.89$ mm s^{-1}. The QS/IS ratio, ρ, proposed earlier [18] as an index of coordination number for di- and tri-organotin(IV) structures with $\rho >$ ca. 2.1 characteristic of coordination numbers greater than four, has the value 1.88 for the chloro complex and 1.51 for the title complex. This reveals the inadequacy of the ρ-factor even as a first approach to the data on closely related compounds and, in the present case, prompted the X-ray structural study.

Figure 1 shows the stereoview of the molecule and also gives the atomic-numbering scheme, while Fig. 2 shows the molecular packing diagram in the unit cell. As is evident from the packing diagram (Fig. 2), the structure consists of relatively isolated molecules; the shortest intermolecular distance of approach not involving hydrogen

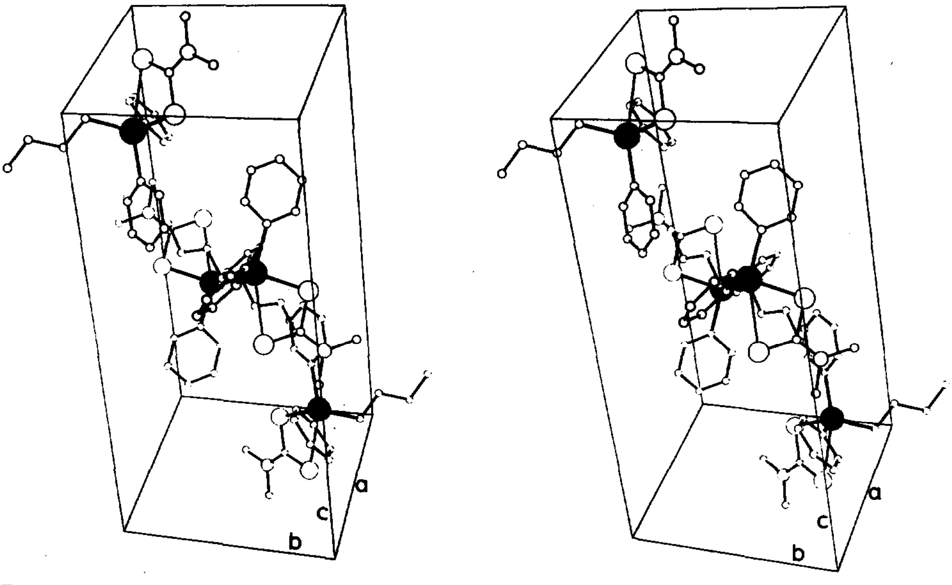

Fig. 2. Molecular packing in [n-BuPh$_2$SnS$_2$CNMe$_2$].

TABLE 3

BOND DISTANCES (Å) AND ANGLES (°) (ESTIMATED STANDARD DEVIATIONS IN PARENTHESES)

(a) Distances			
Sn–S(1)	2.466(1)	C(13)–C(14)	1.366(5)
Sn–S(2)	3.079(1)	C(14)–C(15)	1.373(5)
Sn–C(11)	2.128(3)	C(15)–C(16)	1.375(5)
Sn–C(21)	2.151(3)	C(21)–C(22)	1.392(4)
Sn–C(1b)	2.146(3)	C(21)–C(26)	1.386(4)
S(1)–C(N)	1.762(3)	C(22)–C(23)	1.370(4)
S(2)–C(N)	1.680(3)	C(23)–C(24)	1.365(5)
C(N)–N	1.326(4)	C(24)–C(25)	1.370(5)
N–C(M1)	1.476(4)	C(25)–C(26)	1.382(4)
N–C(M2)	1.489(4)	C(1b)–C(2b)	1.517(5)
C(11)–C(12)	1.409(4)	C(2b)–C(3b)	1.528(5)
C(11)–C(16)	1.377(4)	C(3b)–C(4b)	1.510(6)
C(12)–C(13)	1.397(5)		
(b) Angles			
S(1)–Sn–S(2)	63.81(3)	C(N)–N–C(M1)	120.6(3)
S(1)–Sn–C(11)	114.81(8)	C(N)–N–C(M2)	122.2(3)
S(1)–Sn–C(21)	95.00(8)	C(M1)–N–C(M2)	117.2(3)
S(1)–Sn–C(1b)	116.45(10)	C(12)–C(11)–C(16)	117.0(3)
S(2)–Sn–C(11)	84.42(8)	C(11)–C(12)–C(13)	120.1(3)
S(2)–Sn–C(21)	158.75(10)	C(12)–C(13)–C(14)	120.1(3)
S(2)–Sn–C(1b)	82.54(10)	C(13)–C(14)–C(15)	120.7(3)
C(11)–Sn–C(1b)	113.27(13)	C(14)–C(15)–C(16)	119.0(3)
C(21)–Sn–C(1b)	110.23(13)	C(11)–C(16)–C(15)	122.9(3)
C(21)–Sn–C(11)	104.70(13)	C(22)–C(21)–C(26)	115.7(3)
Sn–S(1)–C(N)	97.51(11)	C(21)–C(22)–C(23)	123.2(3)
Sn–S(2)–C(N)	78.94(10)	C(22)–C(23)–C(24)	119.4(3)
Sn–C(11)–C(12)	121.6(2)	C(23)–C(24)–C(25)	119.6(3)
Sn–C(11)–C(16)	121.0(2)	C(24)–C(25)–C(26)	120.6(3)
Sn–C(21)–C(22)	119.9(2)	C(21)–C(26)–C(25)	121.5(3)
Sn–C(21)–C(2b)	124.4(2)	C(1b)–C(2b)–C(3b)	114.6(3)
Sn–C(1b)–C(2b)	113.0(2)	C(2b)–C(3b)–C(4b)	114.1(4)
S(1)–C(N)–S(2)	119.7(2)		
S(1)–C(N)–N	117.1(2)		
S(2)–C(N)–N	123.2(2)		

atoms is 3.596(5) Å between C(M1) of the dithiocarbamato ligand and the *ortho*-carbon C(12) of the phenyl ring (1) of the neighbouring molecule (symmetry transformation: $1-x,\ 1+y,\ -z$). Bond lengths and bond angles are shown in Table 3. The digits in parentheses in the Tables are the estimated standard deviations in the least significant figures quoted and were derived from the inverse matrix in the course of least-squares refinement calculations.

The coordination about the tin atom is best described as a distorted tetrahedron. The Sn–S(1) bond has a rather normal value of 2.466(1) Å and is only slightly longer than the corresponding bond in n-BuPhSn(Cl)S_2CNEt_2 (Sn–S(1) 2.454(1) Å). The latter complex which has an unsymmetrical dithiocarbamate coordination has an elongated Sn–S(2) bond (2.764(1) Å). In the present complex this bond distance is appreciably longer at 3.079(1) Å, which indicates a weak Sn–S(2) coordinative interaction. Indeed, the S(1)–Sn–S(2) bond angle of 63.81(3)° is significantly smaller

TABLE 4

COMPARISON OF STRUCTURAL FEATURES OF n-$BuPh_2SnS_2CNMe_2$ WITH OTHER TRIORGANOTIN DITHIOCARBAMATES

Complex	Sn–S(1) (Å)	Sn–S(2) (Å)	S(1)–Sn–S(2) (°)	S(1)–C(N) (Å)	S(2)–C(N) (Å)	S(1)–C(N)–S(2) (°)	C(N)–N (Å)	Sn–R (Å)	R–Sn–R (°)
$Me_3SnS_2CNMe_2$ [a] (monoclinic form)	2.47	3.16	62.0	1.75	1.72	117.0	1.31	2.23	107.0
								2.24	119.0
								2.11	115.0
$Me_3SnS_2CNMe_2$ [b] (orthorhombic form)	2.47	3.16	63.0	1.80	1.70	117.0	1.35	2.23	116.0
								2.18	100.0
								2.24	117.0
	2.47	3.31	60.0	1.78	1.71	119.0	1.34	2.23	116.0
								2.26	105.0
								2.14	119.0
$Ph_3SnS_2CNEt_2$ [c]	2.449	3.118	62.8	1.76	1.68	118.8	1.32	2.14	119.3
								2.14	104.2
								2.18	106.6
$Ph_3Sn[S_2CN(CH_2)_4]$ [d]	2.468	3.106	63.6	1.776	1.702	118.3	1.267	2.124	117.3
								2.155	107.8
								2.167	103.9
n-$BuPh_2SnS_2CNMe_2$ [e]	2.466	3.079	63.81	1.762	1.680	119.7	1.326	2.146	113.27
								2.128	104.70
								2.151	110.23

[a] Ref. 3. [b] Ref. 2. [c] Ref. 4. [d] Ref. 19. [e] This work.

than that encountered for the chloro complex (68.85(1)°), where S(1) and S(2) occupy, respectively, the equatorial and axial sites of the trigonal bipyramidal structure [8]. Seemingly, in the present complex the steric constraint which accompanies the introduction of an additional organic group as replacement for halogen forces the dithiocarbamate ligand into an essentially monodentate configuration. It is, however, not immediately clear whether the distortion of the structure from tetrahedral is engendered solely by the weak Sn–S(2) interaction. If the overall structure is viewed as being slightly distorted towards trigonal-bipyramidal but not reaching it, the likely 'axial' incumbents are S(2) and the phenyl ring (2) since these subtend the largest angle at tin (158.8°). The Sn atom is displaced by 0.52(1) Å towards C(21) (the *ipso*-carbon of phenyl ring (2)) from the S(1)–C(11) – C(1b) plane. Also, the Sn–C(21) bond is marginally longer (2.151(3) Å) than the Sn–C(11) bond (2.128(3) Å). While the 'axial' angle is lower than 180°, it is comparable to the value observed (156.85(1)°) for n-BuPhSn(Cl)S_2CNEt_2; the 'equatorial' angles S(1)–Sn–C(11) 114.81(8)°, S(1)–Sn–C(1b) 116.45(10)° and C(11)–Sn–C(1b) 113.27(13)° are intermediary between tetrahedral and trigonal planar; the 'axial–equatorial' angles S(2)–Sn–S(1), C(11), C(1b) amount to 63–84°, and the angles C(21)–Sn–S(1), C(11), C(1b) are equal to 95–110° instead of close to 90° as observed for the chloro complex. Rather similar angular values have been observed recently in the 'four'-coordinated triphenyltin and triphenyllead esters of 1-pyrrolidinedithiocarbamic acid [19]. The Sn–S(2) bond distance in the above triphenyltin derivative is 3.106 Å; the S(1)–Sn–S(2) bond angle is 63.6° [19]. Structural studies on two other triorganotin dithiocarbamate complexes have been described in the literature: $Me_3SnS_2CNMe_2$ (orthorhombic [2] as well as monoclinic [3] modifications) and $Ph_3SnS_2CNMe_2$ [4]. Selected bond distances and angles for these complexes are compared with those of the title complex in Table 4. It is seen that the geometry of the SnC_3S_2 core is rather similar for all cases suggesting negligible influence of electronic effects due to the R groups on tin on the extent of Sn–S(2) coordinative interaction. The SnSCS skeletons are planar within experimental error, the sum of the internal angles being 358–360°. The two phenyl rings in the title complex are tilted differently with respect to this plane, ring (1) making an angle of 68.0° and ring (2) one of 10.0° with the plane. The interplanar angle between the two phenyl rings is 58.8°.

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