

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} = 0.01$
$R = 0.032$	$\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$
$wR = 0.042$	$\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$
$S = 1.72$	Atomic scattering factors
1403 reflections	from <i>International Tables</i>
133 parameters	for <i>X-ray Crystallography</i>
H-atom parameters not refined	(1974, Vol. IV)
$w = 4F_o^2/\sigma^2(F_o^2)$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	x	y	z	B_{eq}
Zn(1)	1/2	0	0	1.92 (2)
O(1)	-0.3655 (4)	0.0313 (3)	0.3422 (2)	3.9 (1)
O(2)	-0.1896 (2)	0.1038 (3)	0.4734 (2)	3.5 (1)
O(3)	0.4251 (2)	-0.2564 (3)	-0.0797 (2)	2.45 (8)
O(4)	0.5572 (3)	-0.1526 (3)	0.1498 (2)	2.9 (1)
N(2)	0.2861 (3)	0.0631 (3)	0.0572 (2)	2.4 (1)
N(5)	0.0563 (3)	0.1817 (4)	0.0575 (3)	4.3 (1)
C(1)	0.1816 (4)	0.1580 (5)	0.0005 (3)	3.0 (1)
C(3)	0.2257 (5)	0.0278 (5)	0.1606 (3)	4.0 (2)
C(4)	0.0836 (5)	0.0987 (5)	0.1620 (4)	5.2 (2)
C(6A)	-0.0022 (7)	0.0770 (8)	0.2717 (6)	2.0 (2)
C(6B)	-0.070 (1)	0.103 (1)	0.2118 (7)	2.8 (3)
C(7A)	-0.1512 (6)	0.1011 (8)	0.2643 (5)	2.0 (2)
C(7B)	-0.070 (1)	0.066 (1)	0.3192 (7)	2.4 (3)
C(8)	-0.2324 (5)	0.0721 (5)	0.3726 (4)	4.0 (2)

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

Zn(1)—O(3)	2.167 (2)	N(5)—C(1)	1.331 (4)
Zn(1)—O(4)	2.132 (2)	N(5)—C(4)	1.389 (6)
Zn(1)—N(2)	2.082 (3)	C(3)—C(4)	1.354 (6)
O(1)—C(8)	1.250 (5)	C(4)—C(6A)	1.541 (8)
O(2)—C(8)	1.260 (5)	C(4)—C(6B)	1.501 (9)
N(2)—C(1)	1.310 (4)	C(6A)—C(7A)	1.325 (8)
N(2)—C(3)	1.382 (4)	C(6B)—C(7B)	1.30 (1)
C(7A)—C(8)	1.513 (7)	C(7B)—C(8)	1.59 (1)
O(3)—Zn(1)—O(4)	88.97 (8)	N(5)—C(4)—C(3)	106.1 (3)
O(3)—Zn(1)—N(2)	93.68 (8)	N(5)—C(4)—C(6A)	136.5 (4)
O(4)—Zn(1)—N(2)	91.5 (1)	N(5)—C(4)—C(6B)	102.5 (5)
Zn(1)—N(2)—C(1)	125.0 (2)	C(3)—C(4)—C(6A)	117.4 (5)
Zn(1)—N(2)—C(3)	129.6 (3)	C(3)—C(4)—C(6B)	149.8 (5)
C(1)—N(2)—C(3)	105.2 (3)	C(4)—C(6A)—C(7A)	116.9 (6)
C(1)—N(5)—C(4)	106.4 (3)	C(4)—C(6B)—C(7B)	114.6 (9)
N(2)—C(1)—N(5)	112.8 (3)	C(6A)—C(7A)—C(8)	115.4 (6)
N(2)—C(3)—C(4)	109.4 (4)	C(6B)—C(7B)—C(8)	114.9 (8)
O(1)—C(8)—C(7A)	105.1 (4)	O(1)—C(8)—O(2)	124.0 (3)
O(1)—C(8)—C(7B)	136.9 (4)	O(2)—C(8)—C(7A)	130.3 (4)
O(2)—C(8)—C(7B)	98.2 (4)		

Data collection and cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). The scan rate was $8.0^\circ \text{ min}^{-1}$ and the scan width was $(1.31 + 0.30 \tan\theta)^\circ$. The ratio of peak counting time to background counting time was 2:1. Structure solution: *MITHRIL* (Gilmore, 1984). Structure refinement: *DIRDIF* (Beurskens, 1984). All calculations, including data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms. Molecular graphics: *ORTEPII* (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, hydrogen-bonding geometry, complete geometry and torsion angles have been deposited with the IUCr (Reference: HR1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(triphenyltin)borate

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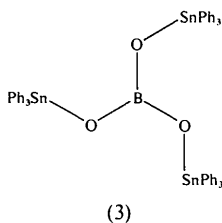
Abstract

The central BO_3 unit in $\text{B}(\text{OSnPh}_3)_3$ has essentially trigonal-planar geometry but the Sn atoms are located

0.043(5)–0.550(5) Å below the BO₃ plane. The principal mean dimensions are B—O 1.368(6) and Sn—O 1.990(11) Å. The three B—O—Sn angles are significantly different at 111.9(3), 116.9(3) and 121.6(3)°.

Comment

Notably few organometallic compounds containing B—O—Sn linkages have been characterized structurally by X-ray diffraction methods. Indeed, the structures of 'Bu₂Sn[OB(OH)Ph]₂ (1) and 'Bu₂Sn(OH)₂[('Bu₂SnO)₂OB(2,4,6-Me₃C₆H₂)₂·2MeCN (2) were reported recently as the first examples of organotin borates (Brown, Mahon & Molloy, 1992). As a continuation of our work on B—O—XPh₃-containing species (X = Si, Ge, Sn, Pb) (Murphy, Sheehan, Spalding, Ferguson, Lough & Gallagher, 1993), we synthesized B(OSnPh₃)₃ (3) by a previously reported route (Mehrotra, Srivastava & Mehrotra, 1974) and determined its structure.



A view of the B(OSnPh₃)₃ molecule (3) is shown in Fig. 1 with our numbering scheme. The central BO₃ unit in (3) has a trigonal-planar arrangement with an average B—O distance of 1.368(6) Å and displacements from the BO₃ plane of –0.013(5) for B1 and 0.001(4) Å for O1 to O3. In the BO₃Sn₃ moiety, the Sn atoms all lie below the BO₃ plane [Sn1 by 0.550(5), Sn2 by 0.043(5) and Sn3 by 0.520(5) Å]; the torsion angles corresponding to these displacements are Sn1—O1—B1—O2 15.9(3), Sn2—O2—B1—O3 –0.0(2) and Sn3—O3—B1—O1 15.8(2)°. The Sn—O(B) distances in (3) vary from 1.975(3) (Sn3—O3) to 2.002(3) Å (Sn1—O1); the Sn2—O2 distance has an intermediate value of 1.993(3) Å.

The O—B—O angles [118.4(4)–121.3(4)°] show a smaller variation than the B—O—Sn angles, which are all significantly different. The value of B1—O1—Sn1 [111.9(3)°] is close to that of the *sp*³ tetrahedral angle whereas B1—O2—Sn2 [121.6(3)] and B1—O3—Sn3 [116.9(3)°] have values closer to the *sp*² value. These data suggest that, like B—O—Si and B—O—Ge (Murphy, Sheehan, Spalding, Ferguson, Lough & Gallagher, 1993), B—O—Sn angles are 'flexible' and can be altered by relatively small changes in the energy of the local environment around the B—O—SnPh₃ units (*e.g.* intra- and intermolecular packing forces). The SnPh₃ unit at Sn1 has a slightly distorted

propeller conformation; the other two SnPh₃ moieties have irregular conformations.

In molecules (1) and (2) (Brown, Mahon & Molloy, 1992), the accuracy of the B—O and Sn—O(B) distances is low [*e.g.* Sn—O are 1.97(2) to 1.99(2) Å in (1), 2.04(2) Å in (2)] and this precludes a detailed comparison with those of (3). The Sn—O—B angles varied from 114(2) to 140(2)° in (1) and were 134(3)° in (2).

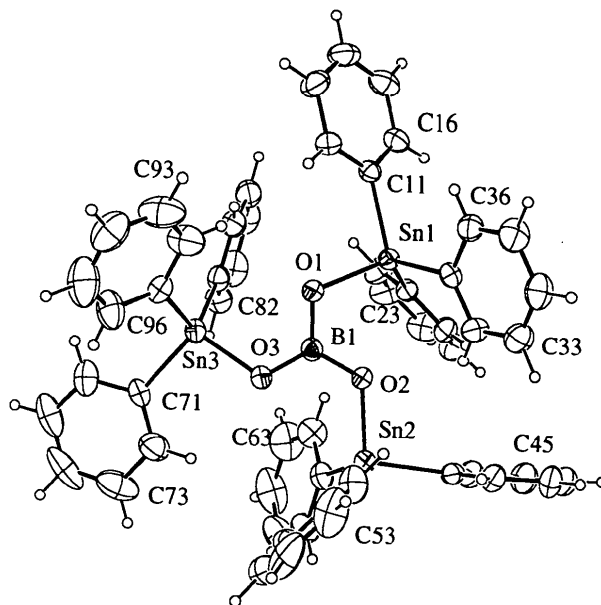


Fig. 1. A view of the B(OSnPh₃)₃ molecule with the numbering scheme indicated. Phenyl ring C atoms are labelled as Ci1 to Ci6 (*i* = 1–9) with Ci1 always bonded to an Sn atom. The non-H atoms are shown as displacement ellipsoids drawn at the 30% probability level. For clarity, H atoms are drawn as small spheres of an arbitrary size.

Experimental

The material, synthesized by the method of Mehrotra, Srivastava & Mehrotra (1974), was recrystallized from dichloromethane to yield crystals suitable for the X-ray study.

Crystal data

[Sn₃(BO₃)(C₆H₅)₉]
M_r = 1108.8
 Monoclinic
*P*2₁/*c*
a = 16.866(2) Å
b = 14.9176(14) Å
c = 19.273(3) Å
 β = 98.022(12)°
V = 4801.8(10) Å³
Z = 4
D_x = 1.534 Mg m^{–3}

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 9.50–12.50°
 μ = 1.59 mm^{–1}
T = 293 K
 Cut needle
 0.40 × 0.24 × 0.18 mm
 Colorless

Data collection

Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: empirical (three ψ scans, 4° steps)
 $T_{\min} = 0.5780$, $T_{\max} = 0.6629$
 10 788 measured reflections
 10 448 independent reflections

6635 observed reflections
 $[I > 2.5\sigma(I)]$
 $R_{\text{int}} = 0.011$
 $\theta_{\text{max}} = 26.92^\circ$
 $h = -21 \rightarrow 21$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 24$
 3 standard reflections
 frequency: 120 min
 intensity variation: 1.0%

Refinement

Refinement on F
 $R = 0.030$
 $wR = 0.039$
 $S = 0.95$
 6635 reflections
 551 parameters
 H atoms riding, C—H 0.95 Å
 $w = 1/[\sigma^2(F) + 0.0008F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.004$

$\Delta\rho_{\text{max}} = 0.36 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{Å}^{-3}$
 Extinction correction: Larson (1970)
 Extinction coefficient: $1.6(4) \times 10^3$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

C61	0.2467 (3)	0.0980 (3)	0.4457 (3)	0.060 (3)
C62	0.3159 (3)	0.1110 (4)	0.4168 (3)	0.081 (4)
C63	0.3886 (4)	0.1186 (6)	0.4582 (5)	0.121 (6)
C64	0.3938 (5)	0.1139 (5)	0.5289 (6)	0.133 (7)
C65	0.3263 (5)	0.1009 (5)	0.5595 (4)	0.116 (5)
C66	0.2529 (4)	0.0941 (4)	0.5179 (3)	0.083 (4)
C71	0.3669 (3)	-0.1933 (3)	0.4021 (3)	0.061 (3)
C72	0.3426 (4)	-0.1598 (5)	0.4613 (3)	0.094 (4)
C73	0.3766 (5)	-0.1899 (7)	0.5271 (4)	0.125 (6)
C74	0.4338 (5)	-0.2565 (7)	0.5324 (5)	0.126 (6)
C75	0.4579 (5)	-0.2899 (5)	0.4744 (5)	0.117 (6)
C76	0.4255 (3)	-0.2588 (4)	0.4103 (3)	0.084 (4)
C81	0.4078 (3)	-0.0704 (3)	0.2579 (3)	0.057 (3)
C82	0.4648 (3)	-0.0244 (4)	0.3027 (3)	0.080 (3)
C83	0.5231 (4)	0.0279 (4)	0.2777 (4)	0.098 (5)
C84	0.5244 (4)	0.0339 (4)	0.2073 (5)	0.094 (5)
C85	0.4690 (4)	-0.0104 (4)	0.1619 (3)	0.086 (4)
C86	0.4116 (3)	-0.0626 (4)	0.1871 (3)	0.072 (3)
C91	0.2700 (3)	-0.2560 (3)	0.2376 (3)	0.062 (3)
C92	0.2418 (4)	-0.2472 (5)	0.1672 (3)	0.099 (5)
C93	0.2087 (5)	-0.3182 (6)	0.1281 (4)	0.122 (6)
C94	0.2033 (5)	-0.3988 (6)	0.1575 (5)	0.116 (6)
C95	0.2283 (5)	-0.4100 (5)	0.2269 (6)	0.133 (7)
C96	0.2613 (4)	-0.3376 (4)	0.2663 (4)	0.098 (4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Sn1	0.199229 (17)	0.092875 (19)	0.144973 (15)	0.04537 (15)
Sn2	0.135468 (18)	0.089166 (19)	0.380115 (15)	0.04762 (16)
Sn3	0.319694 (19)	-0.14725 (2)	0.299894 (16)	0.05259 (17)
O1	0.21876 (17)	-0.01961 (18)	0.20142 (14)	0.0515 (16)
O2	0.15906 (17)	0.06433 (19)	0.28344 (15)	0.0526 (16)
O3	0.23234 (18)	-0.06748 (19)	0.32025 (15)	0.0554 (16)
B1	0.2027 (3)	-0.0083 (3)	0.2689 (3)	0.049 (3)
C11	0.2541 (2)	0.0597 (3)	0.0554 (2)	0.047 (2)
C12	0.2554 (3)	-0.0271 (3)	0.0300 (2)	0.060 (3)
C13	0.2943 (3)	-0.0477 (4)	-0.0274 (3)	0.078 (3)
C14	0.3313 (3)	0.0187 (5)	-0.0594 (3)	0.086 (4)
C15	0.3310 (3)	0.1052 (4)	-0.0354 (3)	0.079 (4)
C16	0.2935 (3)	0.1252 (3)	0.0211 (3)	0.064 (3)
C21	0.2691 (3)	0.1947 (3)	0.2010 (2)	0.050 (2)
C22	0.3509 (3)	0.1932 (4)	0.2040 (3)	0.079 (4)
C23	0.3987 (4)	0.2564 (5)	0.2421 (4)	0.104 (5)
C24	0.3652 (5)	0.3224 (4)	0.2763 (3)	0.093 (4)
C25	0.2843 (4)	0.3263 (4)	0.2734 (3)	0.083 (4)
C26	0.2363 (3)	0.2630 (3)	0.2363 (3)	0.065 (3)
C31	0.0744 (3)	0.1177 (3)	0.1182 (2)	0.049 (2)
C32	0.0272 (3)	0.1489 (3)	0.1660 (2)	0.062 (3)
C33	-0.0531 (3)	0.1666 (4)	0.1446 (3)	0.078 (4)
C34	-0.0863 (3)	0.1539 (4)	0.0770 (4)	0.083 (4)
C35	-0.0411 (4)	0.1221 (4)	0.0290 (3)	0.083 (4)
C36	0.0394 (3)	0.1047 (3)	0.0492 (3)	0.062 (3)
C41	0.0820 (3)	0.2190 (3)	0.3665 (2)	0.053 (3)
C42	0.0033 (3)	0.2325 (4)	0.3379 (3)	0.069 (3)
C43	-0.0262 (4)	0.3202 (5)	0.3261 (3)	0.093 (4)
C44	0.0227 (6)	0.3917 (4)	0.3433 (4)	0.101 (5)
C45	0.0993 (5)	0.3790 (4)	0.3726 (4)	0.090 (5)
C46	0.1289 (3)	0.2938 (3)	0.3838 (3)	0.072 (3)
C51	0.0573 (3)	-0.0150 (3)	0.4000 (2)	0.055 (2)
C52	-0.0090 (4)	-0.0334 (4)	0.3528 (3)	0.087 (4)
C53	-0.0617 (4)	-0.1004 (5)	0.3664 (5)	0.122 (6)
C54	-0.0481 (5)	-0.1488 (4)	0.4266 (7)	0.130 (8)
C55	0.0176 (6)	-0.1324 (4)	0.4729 (4)	0.107 (5)
C56	0.0696 (4)	-0.0661 (3)	0.4604 (3)	0.077 (3)

Table 2. Selected geometric parameters (Å , $^\circ$)

Sn1—O1	2.002 (3)	Sn3—O3	1.975 (3)
Sn1—C11	2.129 (4)	Sn3—C71	2.133 (5)
Sn1—C21	2.123 (4)	Sn3—C81	2.124 (5)
Sn1—C31	2.128 (4)	Sn3—C91	2.120 (5)
Sn2—O2	1.993 (3)	O1—B1	1.376 (6)
Sn2—C41	2.137 (4)	O2—B1	1.361 (6)
Sn2—C51	2.108 (4)	O3—B1	1.367 (6)
Sn2—C61	2.115 (5)		
O1—Sn1—C11	100.89 (14)	O3—Sn3—C71	101.73 (17)
O1—Sn1—C21	106.61 (14)	O3—Sn3—C81	109.33 (15)
O1—Sn1—C31	111.10 (14)	O3—Sn3—C91	109.05 (15)
C11—Sn1—C21	107.82 (16)	C71—Sn3—C81	109.59 (18)
C11—Sn1—C31	111.79 (16)	C71—Sn3—C91	110.07 (19)
C21—Sn1—C31	117.26 (16)	C81—Sn3—C91	116.10 (19)
O2—Sn2—C41	100.95 (14)	Sn1—O1—B1	111.9 (3)
O2—Sn2—C51	104.16 (15)	Sn2—O2—B1	121.6 (3)
O2—Sn2—C61	107.10 (16)	Sn3—O3—B1	116.9 (3)
C41—Sn2—C51	115.24 (17)	O1—B1—O2	118.4 (4)
C41—Sn2—C61	109.93 (18)	O1—B1—O3	120.3 (4)
C51—Sn2—C61	117.54 (18)	O2—B1—O3	121.3 (4)

Molecule (3) crystallized in the monoclinic system; space group $P2_1/c$ was established from the systematic absences. All H atoms were visible in difference maps and were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. Examination of the structures with *PLATON* (Spek, 1994) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection and cell refinement: *CAD-4/PC Software* (Enraf-Nonius, 1992). Data reduction, structure solution and refinement, preparation of material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1217). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chlorobis(*N,N*-diethyldithiocarbamate)-(vinyl)tin(IV)

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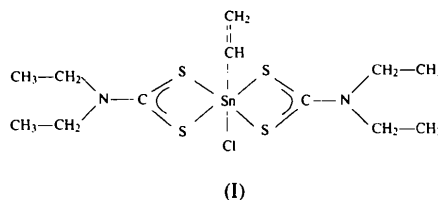
Abstract

The crystal structure of the title compound, [SnCl(C₂H₃)(C₅H₁₀NS₂)₂], consists of relatively isolated complex molecules, the closest intermolecular contact [3.573 (6) Å] being between the methyl C

atom C(3) and the S atom S(2) of neighbouring molecules. The Sn atom is hexacoordinated by the vinyl group, the Cl atom and the two bidentate dithiocarbamate ligands, resulting in a distorted octahedral geometry. The vinyl group and Cl atom both occupy *cis* positions with respects to the dithiocarbamate ligands.

Comment

Our current interest in a series of bis-dithiocarbamate (dtc) complexes of tin(IV) arises partly because of the variety of their coordination geometries around the Sn atom and partly because of their biological activity. The coordination geometry depends on the bonding mode (monodentate, bidentate or anisobidentate) of the dtc ligands (Lindley & Carr, 1974; Harrison & Mangia, 1976; Morris & Schlemper, 1979; Lockhart, Manders, Schlemper & Zuckerman, 1986; Jung, Jeong & Sohn, 1990; Vrábek, Lokaj, Kellö, Garaj, Batsanov & Struchkov, 1992). In this paper we report the structure of [Cl(C₂H₃)Sn(S₂CNEt₂)₂], (I). One aim of this work was to further our understanding of the metal–ligand bonds in organotin–dtc complexes.



Single crystals of (I) were obtained by slow evaporation from a mixture of CHCl₃/ethanol at room temperature. The molecular geometry and atom-labeling scheme for the complex are shown in Fig. 1. The Sn^{IV} atom has sixfold coordination, with the two dithiocarbamate ligands, the vinyl group and the Cl atom occupying mutually *cis* positions. The coordination polyhedron around the Sn^{IV} atom may reasonably be described as a distorted tetragonal bipyramid: three S atoms [S(1), S(2), S(4)] and the Cl(1) atom occupy the equatorial plane; the Sn atom is located 0.223 (5) Å above this plane while the atoms S(3) and C(11) are axial. The angle between these axial groups, S(3)—Sn(1)—C(11), is 161.0 (2)°. The two dtc ligands in the present complex show a bidentate bonding mode; however, X-ray studies show that dtc ligands usually act in an anisobidentate manner in organotin(IV)–bis-dtc complexes, except for [PhSnCl(S₂CNEt₂)₂] (Harrison & Mangia, 1976) and [CH₃OOCCH₂CH₂SnCl(S₂CNMe₂)₂] (Jung, Jeong & Sohn, 1990) in which four Sn—S bond distances [Sn—S(1) 2.528 (6), Sn—S(2) 2.661 (7), Sn—S(3) 2.551 (5), Sn—S(4) 2.593 (4);