

C(41)	0.0761 (6)	0.2287 (8)	0.2526 (6)	0.025 (2)
C(42)	0.0909 (7)	0.1776 (9)	0.3409 (6)	0.034 (2)
C(43)	0.0301 (8)	0.2043 (10)	0.4083 (7)	0.046 (3)
C(44)	-0.0481 (8)	0.2867 (10)	0.3893 (8)	0.050 (3)
C(45)	-0.0643 (8)	0.3384 (11)	0.3030 (8)	0.048 (3)
C(46)	-0.0046 (7)	0.3101 (9)	0.2354 (7)	0.035 (2)

Table 4. Selected geometric parameters (Å, °) for (II)

Pb(1)—C(41)	2.209 (9)	C(23)—C(24)	1.385 (14)
Pb(1)—C(31)	2.220 (9)	C(24)—C(25)	1.373 (14)
Pb(1)—C(2)	2.262 (9)	C(25)—C(26)	1.390 (13)
Pb(1)—C(1)	2.267 (9)	C(31)—C(36)	1.383 (12)
C(1)—C(11)	1.496 (12)	C(31)—C(32)	1.409 (13)
C(11)—C(16)	1.381 (13)	C(32)—C(33)	1.392 (12)
C(11)—C(12)	1.392 (13)	C(33)—C(34)	1.388 (14)
C(12)—C(13)	1.363 (15)	C(34)—C(35)	1.357 (14)
C(13)—C(14)	1.39 (2)	C(35)—C(36)	1.404 (13)
C(14)—C(15)	1.37 (2)	C(41)—C(42)	1.391 (13)
C(15)—C(16)	1.40 (2)	C(41)—C(46)	1.407 (12)
C(2)—C(21)	1.488 (11)	C(42)—C(43)	1.373 (14)
C(21)—C(26)	1.390 (12)	C(43)—C(44)	1.391 (15)
C(21)—C(22)	1.397 (12)	C(44)—C(45)	1.37 (2)
C(22)—C(23)	1.384 (13)	C(45)—C(46)	1.369 (14)
C(41)—Pb(1)—C(31)	111.1 (3)	C(12)—C(11)—C(1)	122.1 (9)
C(41)—Pb(1)—C(2)	111.6 (3)	C(21)—C(2)—Pb(1)	107.4 (5)
C(31)—Pb(1)—C(2)	104.6 (3)	C(26)—C(21)—C(2)	121.1 (8)
C(41)—Pb(1)—C(1)	108.5 (3)	C(22)—C(21)—C(2)	122.1 (8)
C(31)—Pb(1)—C(1)	113.1 (3)	C(36)—C(31)—Pb(1)	118.8 (7)
C(2)—Pb(1)—C(1)	107.8 (3)	C(32)—C(31)—Pb(1)	122.9 (6)
C(11)—C(1)—Pb(1)	112.3 (5)	C(42)—C(41)—Pb(1)	122.1 (7)
C(16)—C(11)—C(1)	119.8 (9)	C(46)—C(41)—Pb(1)	121.2 (7)

No systematic absences were detected for compound (I). In (II), the systematic absences ($h0l$) $h + l = 2n + 1$ and ($0k0$) $k = 2n + 1$ were detected. Both structures were solved by standard Patterson and difference Fourier methods (*SHELXTL-Plus*; Sheldrick 1987). Structure (I) refined satisfactorily in space group $P\bar{1}$ (No. 2) and structure (II) refined satisfactorily in space group $P2_1/n$ (No. 14) by full-matrix least-squares calculations using *SHELXL93* (Sheldrick, 1994). H atoms were placed in geometrically calculated positions and refined with common isotropic displacement factors for different C—H types (C—H_{aryl} 0.95 Å, C—H_{alkyl} 0.99 Å). Other programs used were *PARST* (Nardelli, 1983), *PLATON* (Spek, 1990) and *MISSYM* (Le Page, 1987).

We gratefully acknowledge support by Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and stereoviews of both structures have been deposited with the IUCr (Reference: AB1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Redetermination of Lead(IV) Acetate

MARKUS SCHÜRMAN AND FRIEDO HUBER

Lehrstuhl für Anorganische Chemie II, Universität Dortmund, Otto-Hahn-Strasse 6, 44227 Dortmund, Germany

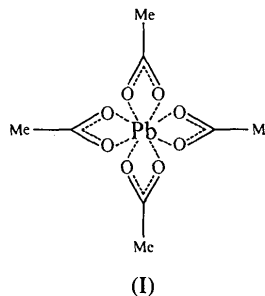
(Received 15 April 1994; accepted 6 June 1994)

Abstract

The redetermination of Pb(OCOCH₃)₄ corroborates and complements the limited data of a prior, yet incomplete study. The structure consists of two discrete molecules in each of which the Pb atom is surrounded by four bidentate acetate ligands forming a nearly dodecahedral polyhedron.

Comment

About 30 years ago in a short conference note, Kamenar (1963) reported that lead(IV) acetate, Pb(OAc)₄ (OAc = CH₃COO), crystallizes in space group $P2_1/c$ with $Z = 8$, the structure consisting of two symmetrically independent sets of molecules. The polyhedron around the eight-coordinate Pb atom was described as a flattened trigonal dodecahedron. Apart from the cell dimensions [$a = 13.01$ (2), $b = 26.59$ (4), $c = 7.86$ (2) Å, $\beta = 105.6$ (2)°], no other data were given. A full description was announced after due refinement of the atomic coordinates (Kamenar, 1963; Kamenar & Bruvo, 1972), but to our knowledge it was not published. Since our studies on organolead(IV) carboxylates would profit from detailed structural data of Pb(OAc)₄, (I), we have redetermined the structure of this compound.



The acetate ligands are bidentate with the two Pb—O distances being virtually the same in all but two of the eight acetate ligands: Pb(1)—O(5) and Pb(1)—O(6) differ by 0.059 (13) Å, and Pb(2)—O(9) and Pb(2)—O(10) by 0.057 (12) Å, *i.e.* they are significantly different, indicating slight unsymmetrical chelation of these two ligands. Fig. 1 shows that the ligands lie in two planes with dihedral angles of 89.9 (2) [Pb(1) molecule] and 87.6 (2)° [Pb(2) molecule]. The mean O—Pb—O bite angle is 57.2 (5)° for the Pb(1) molecule and 57.4 (5)° for the Pb(2) molecule. The eight O atoms around Pb form a nearly dodecahedral polyhedron. Very similar arrangements were found in Sn(OAc)₄ (Alcock & Tracy, 1979), Ti(NO₃)₄ (Garner & Wallwork, 1966) and Sn(NO₃)₄ (Garner, Sutton & Wallwork, 1967). The dodecahedral coordination around the central atom in Pb(OAc)₄ is obviously less irregular than in Sn(OAc)₄. Thus, in the latter compound the maximum difference between the Sn—O distances, which vary from 2.29 (2) to 2.13 (2) Å (Alcock & Tracy, 1979), is 0.16 (2) Å, while the corresponding value for Pb(OAc)₄ is only 0.068 (13) Å. Following the considerations of Alcock & Tracy (1979), it would appear that the coordination sphere around the large central atom in Pb(OAc)₄ is less overcrowded than in Sn(OAc)₄. Alternatively, the polyhedron around Pb might also be regarded as a distorted tetrahedron with the midpoint between the carboxylate O atoms of each ligand occupying a tetrahedral coordination site. Since the appropriate angles C—C—Pb (Table 2) deviate significantly from the ideal value, this view seems unjustified.

No data for other lead(IV) carboxylates are available for comparing the Pb—O distances. However, it appears reasonable and interesting to use organolead(IV) acetates for comparison, though this cannot be performed unreservedly as these compounds contain unsymmetrically chelating acetate ligands. Only in Ph₂Pb(OAc)₂ were symmetrically chelating and bridging acetate ligands found (Schürmann & Huber, 1994). The Pb—O distances in Pb(OAc)₄ range from 2.244 (12) to 2.312 (13) Å [average over all Pb—O(acetate) distances 2.275 (13) Å] and are in almost all cases shorter than those in the organolead(IV) acetates, irrespective of the coordination number. The Pb atom is also eight-coordinate in the anionic complex [Ph₂Pb(OAc)₃][−] with appreciably longer Pb—O distances [2.48 (2)–2.63 (2) Å, average value 2.55 (2) Å (Alcock, 1972)]. In the two seven-coordinate aryllead(IV) triacetates 2-XC₆H₄Pb(OAc)₃ [X = CH₃, Pb—O 2.18 (1)–2.53 (2) Å, average 2.34 (2) Å; X = Cl, Pb—O 2.22 (2)–2.49 (2) Å, average 2.35 (2) Å (Huber, Preut, Scholz & Schürmann, 1992)] one Pb—O distance of each acetate ligand is shorter than in Pb(OAc)₄, while in diorgano- and triorganolead(IV) acetates most Pb—O distances of the chelating acetate ligands are markedly longer: Ph₄Pb₂(OAc)₄·H₂O·C₆H₆ 2.32 (2)–

2.65 (2) Å, average 2.45 (2) Å (Gaffney, Harrison & King, 1982); Ph₂Pb(OAc)₂ 2.348 (8)–2.547 (8) Å, average 2.403 (8) Å (Schürmann & Huber, 1994), and Ph₃Pb(OAc) 2.36 (2) and 2.56 (2) Å (Gaffney & Harrison, 1982).

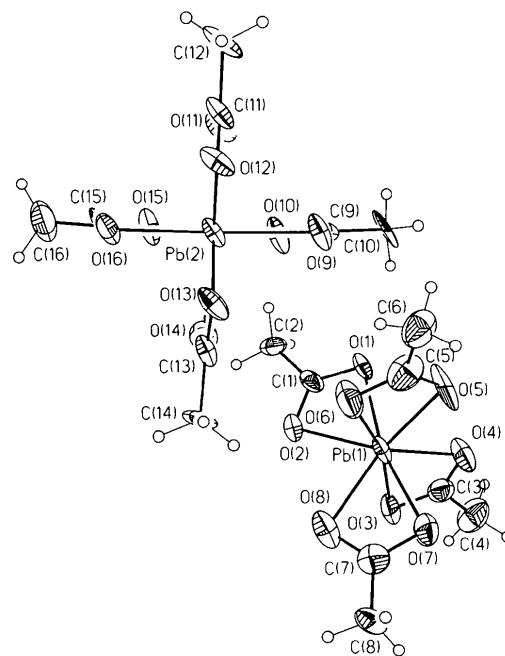


Fig. 1. View (SHELXTL-Plus; Sheldrick, 1987) of part of the crystal structure, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

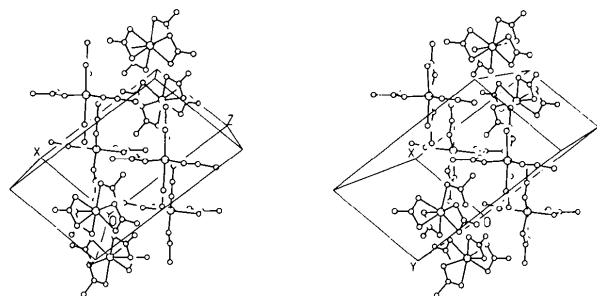


Fig. 2. Stereoview of the crystal packing.

Experimental

Single crystals of the title compound (Merck) were obtained by recrystallization from a 4:1 mixture (*v/v*) of acetic acid (<2% water) and acetic anhydride, which was stirred for 24 h before use.

Crystal data

[Pb(C₂H₃O₂)₄]
M_r = 443.36

Mo K α radiation
 λ = 0.71073 Å

Monoclinic	Cell parameters from 35 reflections
$P2_1/n$	$\theta = 7.7\text{--}15.3^\circ$
$a = 7.781(3) \text{ \AA}$	$\mu = 13.093 \text{ mm}^{-1}$
$b = 26.726(9) \text{ \AA}$	$T = 170(1) \text{ K}$
$c = 12.781(8) \text{ \AA}$	Block
$\beta = 103.71(4)^\circ$	$0.48 \times 0.26 \times 0.20 \text{ mm}$
$V = 2582.1(21) \text{ \AA}^3$	Colourless
$Z = 8$	
$D_x = 2.281 \text{ Mg m}^{-3}$	
Data collection	
Nicolet R3m/V diffractometer	$R_{\text{int}} = 0.0422$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.05^\circ$
Absorption correction: empirical (ψ scans)	$h = -9 \rightarrow 0$
$T_{\text{min}} = 0.234$, $T_{\text{max}} = 0.885$	$k = -6 \rightarrow 31$
4955 measured reflections	$l = -14 \rightarrow 15$
4436 independent reflections	6 standard reflections
2948 observed reflections	monitored every 300 reflections
$[I > 2\sigma(I)]$	intensity variation: 2.1%
Refinement	
Refinement on F^2	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.0585$	$\Delta\rho_{\text{max}} = 0.428 \text{ e \AA}^{-3}$
$wR(F^2) = 0.1542$	$\Delta\rho_{\text{min}} = -0.348 \text{ e \AA}^{-3}$
$S = 1.077$	Extinction correction: none
4422 reflections	Atomic scattering factors
308 parameters	from <i>International Tables for Crystallography</i> (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Only H atom U 's refined	
$w = 1/[\sigma^2(F_o^2) + (0.1115P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

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

	x	y	z	U_{eq}
Pb(1)	0.23273 (8)	0.11053 (3)	-0.61391 (5)	0.0351 (2)
O(1)	0.1223 (14)	0.0531 (4)	-0.5154 (9)	0.038 (3)
O(2)	0.4026 (16)	0.0662 (5)	-0.4787 (9)	0.042 (3)
C(1)	0.2722 (22)	0.0422 (6)	-0.4613 (13)	0.031 (4)
C(2)	0.3001 (24)	0.0028 (7)	-0.3745 (12)	0.043 (5)
O(3)	0.3185 (16)	0.0522 (5)	-0.7223 (10)	0.048 (3)
O(4)	0.0459 (17)	0.0739 (5)	-0.7566 (11)	0.053 (4)
C(3)	0.1618 (26)	0.0477 (7)	-0.7781 (13)	0.039 (4)
C(4)	0.1252 (32)	0.0131 (8)	-0.8762 (14)	0.061 (6)
O(5)	-0.0159 (17)	0.1538 (5)	-0.6140 (13)	0.066 (4)
O(6)	0.2220 (18)	0.1719 (5)	-0.4881 (11)	0.057 (4)
C(5)	0.0527 (34)	0.1805 (8)	-0.5325 (16)	0.056 (6)
C(6)	-0.0437 (32)	0.2183 (8)	-0.4894 (16)	0.063 (6)
O(7)	0.2743 (18)	0.1693 (5)	-0.7361 (10)	0.047 (3)
O(8)	0.5019 (17)	0.1432 (5)	-0.6086 (12)	0.056 (4)
C(7)	0.4439 (28)	0.1717 (7)	-0.6940 (15)	0.045 (5)
C(8)	0.5530 (28)	0.2059 (9)	-0.7404 (16)	0.069 (7)
Pb(2)	0.23579 (8)	0.13205 (2)	-0.13405 (5)	0.0332 (2)
O(9)	-0.0033 (15)	0.1473 (4)	-0.2759 (10)	0.043 (3)
O(10)	0.0858 (16)	0.0711 (4)	-0.2382 (10)	0.046 (3)
C(9)	-0.0239 (24)	0.1002 (7)	-0.2919 (14)	0.040 (4)
C(10)	-0.1768 (22)	0.0799 (8)	-0.3823 (16)	0.057 (6)
O(11)	0.0352 (16)	0.1174 (5)	-0.0329 (10)	0.045 (3)
O(12)	0.1138 (16)	0.1955 (5)	-0.0573 (11)	0.050 (3)
C(11)	0.0222 (24)	0.1645 (7)	-0.0137 (16)	0.045 (5)
C(12)	-0.0975 (25)	0.1853 (8)	0.0567 (19)	0.063 (6)
O(13)	0.3593 (15)	0.1981 (4)	-0.1964 (10)	0.042 (3)
O(14)	0.4424 (17)	0.1235 (5)	-0.2333 (9)	0.046 (3)

C(13)	0.4572 (22)	0.1708 (7)	-0.2422 (14)	0.035 (4)
C(14)	0.5851 (25)	0.1948 (8)	-0.3001 (16)	0.062 (6)
O(15)	0.3739 (16)	0.0639 (5)	-0.0464 (10)	0.047 (3)
O(16)	0.4673 (16)	0.1371 (5)	0.0141 (10)	0.041 (3)
C(15)	0.4813 (21)	0.0903 (6)	0.0242 (16)	0.040 (4)
C(16)	0.6119 (29)	0.0655 (9)	0.1100 (17)	0.069 (7)

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

Pb(1)—O(1)	2.278 (11)	Pb(2)—O(9)	2.305 (11)
Pb(1)—O(2)	2.249 (11)	Pb(2)—O(10)	2.248 (12)
Pb(1)—O(3)	2.287 (13)	Pb(2)—O(11)	2.284 (12)
Pb(1)—O(4)	2.268 (13)	Pb(2)—O(12)	2.275 (13)
Pb(1)—O(5)	2.253 (11)	Pb(2)—O(13)	2.244 (12)
Pb(1)—O(6)	2.312 (13)	Pb(2)—O(14)	2.283 (12)
Pb(1)—O(7)	2.292 (12)	Pb(2)—O(15)	2.271 (12)
Pb(1)—O(8)	2.255 (13)	Pb(2)—O(16)	2.288 (12)
Pb(1)—C(1)	2.64 (2)	Pb(2)—C(9)	2.64 (2)
Pb(1)—C(5)	2.69 (2)	Pb(2)—C(11)	2.66 (2)
Pb(1)—C(3)	2.64 (2)	Pb(2)—C(13)	2.66 (2)
Pb(1)—C(7)	2.69 (2)	Pb(2)—C(15)	2.68 (2)
O(1)—Pb(1)—O(2)	56.7 (4)	O(9)—Pb(2)—O(10)	56.9 (4)
O(3)—Pb(1)—O(4)	56.1 (4)	O(11)—Pb(2)—O(12)	58.2 (4)
O(5)—Pb(1)—O(6)	57.6 (5)	O(13)—Pb(2)—O(14)	57.6 (4)
O(7)—Pb(1)—O(8)	58.3 (5)	O(15)—Pb(2)—O(16)	56.7 (4)
O(1)—Pb(1)—O(7)	166.1 (4)	O(9)—Pb(2)—O(16)	166.2 (4)
O(3)—Pb(1)—O(6)	165.5 (5)	O(11)—Pb(2)—O(14)	164.3 (5)
C(1)—Pb(1)—C(3)	96.5 (5)	C(9)—Pb(2)—C(11)	94.4 (5)
C(1)—Pb(1)—C(5)	100.1 (5)	C(9)—Pb(2)—C(13)	101.6 (5)
C(1)—Pb(1)—C(7)	136.8 (6)	C(9)—Pb(2)—C(15)	136.4 (6)
C(3)—Pb(1)—C(5)	136.0 (7)	C(11)—Pb(2)—C(13)	138.0 (6)
C(3)—Pb(1)—C(7)	96.6 (5)	C(11)—Pb(2)—C(15)	97.5 (6)
C(5)—Pb(1)—C(7)	98.4 (7)	C(13)—Pb(2)—C(15)	97.0 (5)
C(1)—C(2)—Pb(1)	178.3 (12)	C(9)—C(10)—Pb(2)	178.1 (14)
C(3)—C(4)—Pb(1)	177.2 (13)	C(11)—C(12)—Pb(2)	177.7 (14)
C(5)—C(6)—Pb(1)	179.6 (14)	C(13)—C(14)—Pb(2)	177.4 (14)
C(7)—C(8)—Pb(1)	177.8 (16)	C(15)—C(16)—Pb(2)	177.7 (14)

The crystal turned slightly brown during data collection indicating slow decomposition. However, no significant effect on the observed standard reflections was discernible. The shape of the displacement ellipsoids in Fig. 1 might be related to changes in the crystal caused by the decomposition.

Systematic absences ($h0l$) $h + l = 2n + 1$, ($h00$) $h = 2n + 1$, ($0k0$) $k = 2n + 1$ and ($00l$) $l = 2n + 1$ were detected. The structure was solved by standard Patterson and difference Fourier methods (*SHELXTL-Plus*; Sheldrick, 1987) and refined satisfactorily with space group $P2_1/n$ by full-matrix least-squares calculations *SHELXL93* (Sheldrick, 1994). The H atoms were placed in geometrically calculated positions and refined with a common isotropic displacement parameter [C —H 0.98 \AA , $U_{\text{iso}} = 0.127(23) \text{ \AA}^2$]. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1992). Other programs used were *PARST* (Nardelli, 1983), *PLATON* (Spek, 1990) and *MISSYM* (Le Page, 1987).

Financial support by Fonds der Chemischen Industrie, is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: AB1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1713–1715

n-Butyltris(2-pyrimidinethiolato-*N,S*)tin(IV): Change in Space Group

RICHARD E. MARSH

*The Beckman Institute, † California Institute of
Technology, Pasadena, California 91125, USA*

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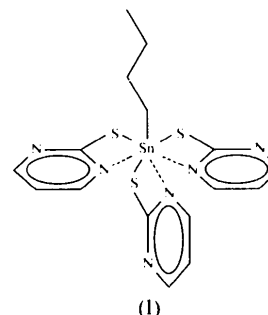
Abstract

The structure of $[\text{Sn}(\text{C}_4\text{H}_3\text{N}_2\text{S})_3(\text{C}_4\text{H}_9)]$, which was originally described in space group $P2_1$ [Schmiedgen, Huber & Schürmann (1994). *Acta Cryst.* **C50**, 391–394], is better described and refined in $P2_1/n$. Revised coordinates and bond lengths are given.

Comment

In their report of the structure of the title compound, (I), Schmiedgen, Huber & Schürmann (1994, hereinafter, SHS) noted that the systematic absences were consistent with space group $P2_1/n$, but that 'the structure could not be solved in this space group'; accordingly, the structure was solved and refined in $P2_1$ [monoclinic; $a = 9.523$ (2), $b = 12.849$ (3), $c = 17.079$ (3) Å, $\beta = 92.67$ (3)°, $Z = 4$], with two independent molecules in the asymmetric unit. In fact, the two molecules are closely related by an n -glide plane and further refinement in $P2_1/n$ was straightforward.

† Contribution No. 8943.



The starting model was derived from the coordinates in Table 1 of SHS by shifting the origin by 0.25 in x and z , and by about 0.24 in y , so as to place a center of symmetry at the conventional origin in $P2_1/n$. Full-matrix refinement, with the heavier atoms anisotropic and the H atoms in calculated positions, was based on the 2664 'observed' F values recovered from SUP 71555. At convergence (maximum shift, 0.03σ), R was 0.0311 for 259 parameters, compared with an R of 0.0293 for 471 parameters reported for the $P2_1$ model (SHS). For the entire set of 3707 reflections in SUP 71555, including those tagged as unobserved [but excluding the space-group absences $h0l$ with $(h+l)$ odd], R was 0.0525 and there were no obvious outliers. The only abnormality during the refinement resulted from disorder of the n -butyl group, which led to two sets of partially populated sites for the outer atoms C(2), C(3) and C(4); the resulting occupancy factor was 0.67 (3) for one set of sites (and 0.33 for the other). Since the two sites for C(2) are separated by only 0.67 Å, the occupant of the minor site, C(2a), would not tolerate refinement of its anisotropic coefficients U_{ij} and was represented as isotropic; this atom also shows unsatisfactory bond lengths, with C(1)—C(2a) 1.29 (4) Å and C(2a)—C(3a) 1.65 (5) Å. The n -butyl groups also caused problems in the earlier $P2_1$ refinement, where SHS report 'conspicuous deviations' of the C(1)—C(2)—C(3) angles from 109.5° that are 'probably caused by lattice effects'. (To the extent that 'lattice effects' are responsible for the disorder, this is a correct assessment.)

Bond lengths and angles given in Table 2 are considerably more reasonable, and more precise, than those reported earlier (SHS), and deserve some further comment. The bonds Sn—S(1) and Sn—S(2) to the equatorial 2-pyrimidinethiolato ligands are approximately equal in length but the axial bond Sn—S(3) is nearly 0.1 Å shorter. This difference in Sn—S bond lengths seems too large to blame merely on steric effects and it is tempting to invoke some sort of *trans* influence by the alkyl group on the opposite side. [Note, though, that the axial array C(1)—Sn—S(3) is far from linear, at 147.6 (1)°.] The Sn—N bonds, which are weaker than Sn—S, show