Co-ordinative Interactions Between Tin(II) and Tin(IV) and Tin(II) and Arsenic(III) in Di-μ-nitrato-bis[(triphenylarsine)(triphenylstannio)tin(II)]

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The structure of the title compound has been solved from *X*-ray diffractometer data by the heavy-atom method and refined by least-squares techniques to *R* 0.060 for 4 409 independent reflections. Crystals are triclinic, space group $P\bar{1}$, with unit-cell dimensions: a = 11.29(1), b = 12.02(1), c = 13.68(1) Å, α = 102.0(1), β = 113.3(1), γ = 104.0(1)°, and Z = 2. The structure consists of molecules of formula $[\{Sn^{II}(NO_3)(AsPh_3)(Sn^{IV}Ph_3)\}_2]$ in which Sn^{II} is five-co-ordinate in a highly irregular way by two adjacent $[NO_3]^-$ anions and by the two triphenylmetal groups, where a disordered distribution involving Sn and As is present. The nitrate groups bridge the two Sn^{II} atoms in the dimer in a rather unusual way.

As we have previously reported,[1] from the reaction of dinitratodiphenyltin with triphenylarsine, in dry acetone under a nitrogen atmosphere, a number of interesting compounds have been isolated, the stoicheiometry of which does not correspond to that of the simple addition derivatives. We have already examined by i.r. spectroscopy and *X*-ray diffractometry $[Sn^{IV}(NO_3)(Sn^{IV}Ph_3)_3]$ (1) and $[Sn^{II}(NO_3)(Sn^{IV}Ph_3)]$ (2).[1] Another compound, (3), has been isolated but not yet completely characterized, being affected by some kind of disorder which complicates the *X*-ray analysis. However, it appears that its nature is ionic with two different tin atoms: one surrounded by four triphenylarsine molecules, the other co-ordinated by disordered nitrate groups and phenyl rings.

The formation of these unusual compounds can be interpreted by reference to various chemical factors such as the reducing character of the triphenylarsine, the oxidizing and co-ordinating properties of the covalent nitrate group, and the possibility for tin and arsenic to assume II–IV and III–V oxidation states respectively. From the same reaction mixture we have now isolated a

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[1] M. Nardelli, C. Pelizzi, G. Pelizzi, and P. Tarasconi, *Z. anorg. Chem.*, 1977, **431**, 250.

new crystalline organotin compound, and the present paper deals with its synthesis, i.r. spectroscopic properties, and *X*-ray analysis. The latter showed the compound to be dimeric $[\{Sn(NO_3)(AsPh_3)(SnPh_3)\}_2]$, where the nitrate group bridges the two $AsPh_3$ and $SnPh_3$ systems connected to Sn^{II}.

EXPERIMENTAL

Preparation.—A colourless crystalline product, (3), was isolated from the reaction of triphenylarsine (0.01 mol) and dinitratodiphenyltin (0.01 mol) in dry acetone under a nitrogen atmosphere. The solid and the filtrate were treated separately, as described previously.[1] From the solid, dissolved in dry acetone, compound (1) was obtained. From the filtrate, a crystalline mixture of products was isolated by slow evaporation of the solvent and removed by filtration until, after successive precipitations and filtrations, compound (2) was obtained. By dissolving the crystalline mixture in dry acetone under a nitrogen atmosphere, after some days colourless prismatic crystals were obtained. The product is soluble in dry acetone and becomes dark in air (Found: C, 52.0; H, 3.85; N, 1.70; Sn, 28.5. $C_{36}H_{30}AsNO_3Sn_2$ requires C, 51.65; H, 3.60; N, 1.65; Sn, 28.35%).

Infrared spectra were recorded on a Perkin-Elmer model 457 spectrophotometer using KBr discs.

Crystal Data.—$M = 1\,673.84$, Triclinic, $a = 11.29(1)$, $b = 12.02(1)$, $c = 13.68(1)$ Å, $\alpha = 102.0(1)$, $\beta = 113.3(1)$, $\gamma = 104.0(1)°$, $U = 1\,649(3)$ Å^3, $D_m = 1.67$ g cm^{-3}, $Z = 1$, $D_c = 1.69$ g cm^{-3}, $F(000) = 1\,640$, Mo-K_α radiation, $\lambda = 0.710\,7$ Å, μ(Mo-K_α) = 25.5 cm^{-1}. Space group $P\bar{1}$.

The preliminary unit-cell dimensions and symmetry information were deduced from rotation and Weissenberg photographs taken with Cu-K_α radiation (λ 1.541 8 Å). The centrosymmetric space group $P\bar{1}$ was assumed at the end of the refinement when it was found that, assuming the non-centrosymmetric space group $P1$, the results were practically equal to those obtained with $P\bar{1}$. Centrosymmetry was indicated also by statistical analysis of the values related to $|E|$:*

	Experimental	Theoretical	
		Centrosymmetric	Non-centrosymmetric
Av. $\|E\|^2$	1.000	1.000	1.000
Av. $\|E^2 - 1\|$	0.938	0.968	0.736
Av. $\|E\|$	0.818	0.798	0.886

Accurate unit-cell parameters with estimated standard deviations were determined by a least-squares refinement of 2θ values for 23 reflections measured on a single-crystal Siemens AED diffractometer.

Data Collection.—Intensity data were collected from a crystal, mean radius 0.12 mm, aligned with its *c* axis parallel to the ϕ axis of the above diffractometer, using Mo-K_α radiation and the ω—2θ scanning technique. Of the 5 808 reflections measured within the range 2θ 5—50°, 4 409 were considered as observed, having $I > 2\sigma(I)$. A standard reflection was recorded every 20 reflections for checking purposes. The variation in its intensity during data collection was negligible in spite of the progressive darkening of the sample. Corrections for Lorentz and polarization effects, but not for absorption, were applied. Data were placed on an absolute scale, first by correlating observed and calculated structure amplitudes, then by determining the scale factor for the F_o values as a parameter in the least-squares refinement.

Structure Analysis.—The positions of the three heaviest atoms were determined from a three-dimensional Patterson map, those of the remaining non-hydrogen atoms being obtained by application of the heavy-atom method. Four cycles of isotropic followed by seven cycles of anisotropic block-diagonal least-squares refinement of the non-hydrogen atoms gave *R* 0.064. This satisfactory refinement was obtained only by assuming a disordered distribution of Sn^{IV} and As which are interchanged with a 50% occupancy factor.

TABLE 1

Fractional atomic co-ordinates (× 10^4) with estimated standard deviations in parentheses

	x	*y*	*z*
(Sn,As) (1)	2 603(1)	1 996(1)	3 073(1)
(Sn,As) (2)	−786(2)	−1 675(1)	1 882(1)
Sn(3)	764(1)	1(1)	1 693(1)
O(1)	831(7)	−710(7)	−69(6)
O(2)	2 242(8)	−1 124(8)	1 182(7)
O(3)	1 957(8)	−1 654(8)	−509(8)
N	1 681(8)	−1 172(7)	189(8)
C(1)	1 991(10)	3 222(9)	3 590(10)
C(2)	2 667(13)	4 070(11)	4 693(12)
C(3)	2 143(15)	4 906(12)	4 999(14)
C(4)	976(16)	4 883(13)	4 264(16)
C(5)	307(16)	4 018(16)	3 211(15)
C(6)	789(13)	3 183(13)	2 870(11)
C(7)	3 732(10)	2 834(9)	2 539(9)
C(8)	4 044(11)	4 096(11)	2 734(10)
C(9)	4 850(14)	4 647(13)	2 333(12)
C(10)	5 276(16)	3 964(15)	1 758(14)
C(11)	4 986(18)	2 720(15)	1 569(14)
C(12)	4 198(15)	2 182(11)	1 985(12)
C(13)	3 872(10)	1 948(9)	4 479(9)
C(14)	3 461(13)	1 120(13)	4 909(11)
C(15)	4 338(18)	1 081(16)	5 916(13)
C(16)	5 624(14)	1 847(14)	6 457(11)
C(17)	6 009(12)	2 689(12)	6 046(11)
C(18)	5 163(11)	2 743(11)	5 057(11)
C(19)	−2 485(10)	−1 572(11)	1 577(9)
C(20)	−2 657(12)	−536(12)	1 496(10)
C(21)	−3 840(16)	−444(16)	1 282(11)
C(22)	−4 837(14)	−1 398(19)	1 167(12)
C(23)	−4 672(13)	−2 432(19)	1 224(13)
C(24)	−3 506(12)	−2 567(14)	1 453(12)
C(25)	−104(10)	−1 776(9)	3 407(9)
C(26)	1 088(11)	−1 903(11)	3 834(10)
C(27)	1 662(14)	−1 902(12)	4 914(11)
C(28)	1 054(15)	−1 797(12)	5 556(11)
C(29)	−131(16)	−1 649(15)	5 146(13)
C(30)	−709(13)	−1 650(14)	4 060(12)
C(31)	−1 192(9)	−3 328(9)	949(10)
C(32)	−1 479(12)	−4 309(10)	1 280(11)
C(33)	−1 772(13)	−5 464(11)	556(13)
C(34)	−1 793(11)	−5 618(11)	−460(12)
C(35)	−1 502(11)	−4 636(12)	−783(11)
C(36)	−1 206(10)	−3 485(10)	−81(10)

The hydrogen atoms were included in their calculated positions as fixed contributions to the structure factors, assuming isotropic thermal parameters. The function minimized was $\Sigma w|F_o - F_c|^2$ in which the weight for each reflection was unity. The final *R* was 0.060 for the observed reflections and 0.076 for the complete set of data. As a final check of the correctness of the structure, a difference-Fourier synthesis was calculated and found to contain no features of chemical significance. In the final cycle the shifts were less than 0.3σ for all the parameters.

Positional parameters for non-hydrogen atoms are given in Table 1. Observed and calculated structure factors and

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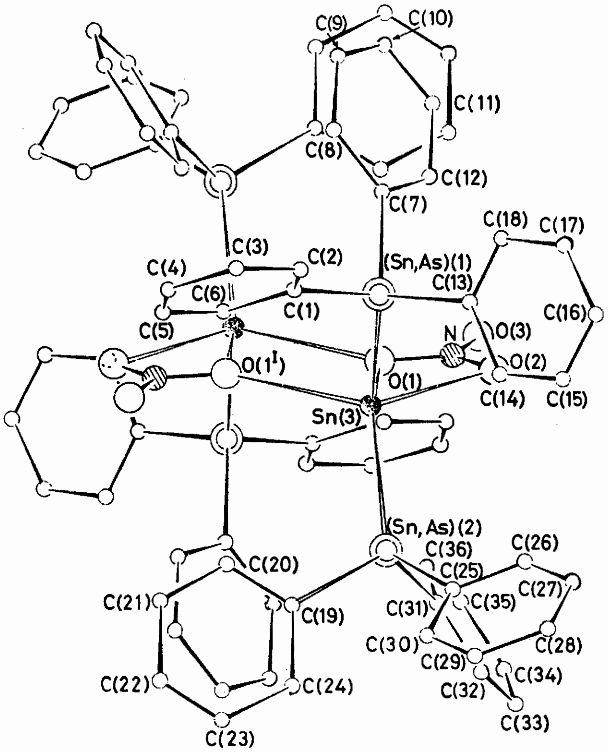
* *E* indicates a normalized structure factor given by $E^2_{hkl} = U^2_{hkl}/\bar{U}^2$, where $U_{hkl} = F_{hkl}/\sum_i^N f_i$.

anisotropic thermal parameters are listed in Supplementary Publication No. SUP 22090 (26 pp.).* Atomic scattering factors were taken from ref. 2. All the calculations were carried out on the Cyber 76 computer of Consorzio per la Gestione del Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale, Casalecchio, Bologna.

RESULTS AND DISCUSSION

Table 2 lists bond distances and angles, and vibrational bands with relative assignments are in Table 3.

As depicted in the Figure, the structure consists of dimeric molecules of formula [{Sn(NO$_3$)(AsPh$_3$)(SnPh$_3$)}$_2$] in which the oxygen atom from the nitrate group bridges two tin(II) atoms related by a centre of symmetry. The disordered distribution of Sn^{IV} and As is such that half the molecules are reversed with respect to the others, with tin occupying arsenic sites and *vice versa*. This kind of disorder can be explained by the bulkiness of the triphenylmetal groups as already found by Harrison *et al.*[3a] in Ph$_3$SiOPbPh$_3$. This disorder, which was initially proposed to explain differences in the electron densities of peaks involving the three heavy atoms as found in the Patterson map, is also the only possibility on the basis of electroneutrality requirements.



Clinographic projection of the structure

The tin(II) atom is five-co-ordinate in a severely irregular way, so that the co-ordination polyhedron is difficult to describe in terms of bonding geometry. The angles at tin range from 48.8 to 134.2° and the range, as well as the geometry, is similar to that observed in [Sn^{II}(NO$_3$)(Sn^{IV}Ph$_3$)] (46.7—144.4°).[1] If the NO$_3$ group is considered to occupy a single co-ordination site, the structure may be described on the basis of a distorted tetrahedral geometry. In both cases, the strong deviations from regular geometry may be ascribed to the different bulkiness of the co-ordinated groups, as indicated by the fact that the largest angle is Sn^{IV}–Sn^{II}–As (134.2°). Unlike most tin(II) derivatives, where co-ordination is affected by stereochemically active lone pairs, in the present compound there is no evidence for a reliable location of the lone pair in spite of the irregularity of the co-ordination polyhedron. As suggested by Harrison and Zuckerman it can be assumed in the presence of very electronegative ligands the lone pair is pulled into a tin 5*s* orbital.[3b]

The disorder involving Sn^{IV} and As prevents a full discussion of the Sn–C and As–C bond distances. However, it can be observed that these values are close to the tetrahedral As^{III}–C(phenyl) bonds, for which the range usually found is 1.92—1.97 Å, but much smaller than the tetrahedral Sn^{IV}–C(phenyl) bonds whose values range from 2.05 to 2.18 Å. On the other hand, values only slightly longer than the present ones have been recently observed by us in [Sn^{IV}(NO$_3$)(Sn^{IV}Ph$_3$)$_3$] and [Sn^{II}(NO$_3$)(Sn^{IV}Ph$_3$)].[1]

The i.r. bands at 333 and 315 cm^{-1} can be attributed to the stretching vibrational modes of Sn–C and As–C bonds; these vibrations lie at higher frequency values than those usually observed for phenyltin systems,[4,5] while they show a positive shift toward lower frequencies with respect to unco-ordinated triphenylarsine.

The bridging behaviour of the nitrate group in this compound is worthy of comment. In fact, as pointed out by Addison *et al.*,[6] most bridging nitrato-groups involve co-ordination by two of the oxygen atoms, each bound to a different metal atom. Nitrato-compounds in which one nitrate oxygen is co-ordinated to more than one metal atom are few and, on the basis of *X*-ray studies, can be differentiated as follows: (*i*) the nitrate group is unidentate with its co-ordinated oxygen bridging two metal atoms as in bis[dinitratobis(pyridine)-copper(II)]-pyridine,[7,†] (2,2′-bipyridyl)dinitratosilver-(II),[8] and di-μ-nitrato-bis[bis(trimethyl phosphite)silver-(I)];[9] (*ii*) the nitrate group is bidentate with both its

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* For details see Notices to Authors No. 7, *J.C.S. Dalton*, 1976, Index issue.

† A second oxygen atom lies 2.906 Å from the copper, this contact being *ca.* 0.9 Å longer than the Cu–O bond length.

[2] D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, **A24**, 321.

[3] (*a*) P. G. Harrison, T. J. King, J. A. Richards, and R. C. Phillips, *J. Organometallic Chem.*, 1976, **116**, 307; (*b*) P. G. Harrison and J. J. Zuckerman, *Inorg. Chim. Acta*, 1977, **21**, L3.

[4] G. Matanbayashi, M. Hiroshima, and T. Tanaka, *J. Inorg. Nuclear Chem.*, 1971, **33**, 3787.

[5] B. V. Liengme, R. S. Randall, and J. R. Sams, *Canad. J. Chem.*, 1972, **50**, 3212.

[6] C. C. Addison, N. Logan, S. C. Wallwork, and C. D. Garner, *Quart. Rev.*, 1971, **25**, 289.

[7] A. F. Cameron, K. P. Forrest, D. W. Taylor, and R. H. Nuttall, *J. Chem. Soc.* (*A*), 1971, 2492.

[8] G. W. Bushnell and M. A. Khan, *Canad. J. Chem.*, 1972, **50**, 315.

[9] J. H. Meiners, J. C. Clardy, and J. G. Verkade, *Inorg. Chem.*, 1975, **14**, 632.

TABLE 2

Bond distances (Å) and angles (°)

(*a*) Distances

Sn(3)–(Sn,As)(1)	2.538(5)	Sn(3)–(Sn,As)(2)	2.521(4)
Sn(3)–O(1)	2.42(1)	Sn(3)–O(2)	2.68(1)
Sn(3)–O(1$^{\rm I}$) *	2.74(1)	N–O(1)	1.24(1)
N–O(2)	1.24(1)	N–O(3)	1.21(2)
(Sn,As)(1)–C(1)	1.93(1)	(Sn,As)(2)–C(19)	1.95(1)
(Sn,As)(1)–C(7)	1.95(1)	(Sn,As)(2)–C(25)	1.96(1)
(Sn,As)(1)–C(13)	1.95(1)	(Sn,As)(2)–C(31)	1.95(1)
C(1)–C(2)	1.40(2)	C(19)–C(20)	1.33(2)
C(2)–C(3)	1.38(2)	C(20)–C)21)	1.37(2)
C(3)–C(4)	1.35(2)	C(21)–C(22)	1.37(3)
C(4)–C(5)	1.36(2)	C(22)–C(23)	1.32(3)
C(5)–C(6)	1.36(3)	C(23)–C(24)	1.36(2)
C(6)–C(1)	1.37(2)	C(24)–C(19)	1.41(2)
C(7)–C(8)	1.41(2)	C(25)–C(26)	1.37(2)
C(8)–C(9)	1.39(2)	C(26)–C(27)	1.36(2)
C(9)–C(10)	1.34(3)	C(27)–C(28)	1.35(3)
C(10)–C(11)	1.39(3)	C(28)–C(29)	1.38(3)
C(11)–C(12)	1.38(3)	C(29)–C(30)	1.37(2)
C(12)–C(7)	1.34(2)	C(30)–C(25)	1.36(2)
C(13)–C(14)	1.35(2)	C(31)–C(32)	1.36(2)
C(14)–C(15)	1.38(2)	C(32)–C(33)	1.40(2)
C(15)–C(16)	1.38(2)	C(33)–C(34)	1.35(3)
C(16)–C(17)	1.35(2)	C(34)–C(35)	1.36(2)
C(17)–C(18)	1.36(2)	C(35)–C(36)	1.38(2)
C(18)–C(13)	1.39(1)	C(36)–C(31)	1.38(2)

(*b*) Angles

(Sn,As)(1)–Sn(3)–(Sn,As)(2)	134.2(6)
(Sn,As)(1)–Sn(3)–O(1)	113.4(6)
(Sn,As)(1)–Sn(3)–O(2)	96.8(5)
(Sn,As)(1)–Sn(3)–O(1$^{\rm I}$)	103.0(4)
O(1)–Sn(3)–O(2)	48.8(4)
Sn(3)–O(1)–N	103(1)
Sn(3$^{\rm I}$)–O(1)–N	146(2)
Sn(3)–O(1)–Sn(3$^{\rm I}$)	110.6(5)
O(1)–Sn(3)–O(1$^{\rm I}$)	69.4(4)
O(2)–Sn(3)–O(1$^{\rm I}$)	117.9(8)
(Sn,As)(2)–Sn(3)–O(1)	110.4(6)
(Sn,As)(2)–Sn(3)–O(2)	101.9(5)
(Sn,As)(2)–Sn(3)–O(1$^{\rm I}$)	104.3(5)
Sn(3)–O(2)–N	119(1)
O(1)–N–O(2)	118(2)
O(1)–N–O(3)	121(2)
O(2)–N–O(3)	121(2)
Sn(3)–(Sn,As)(1)–C(1)	112.9(8)
Sn(3)–(Sn,As)(1)–C(7)	117.5(9)
Sn(3)–(Sn,As)(1)–C(13)	117.0(7)
C(1)–(Sn,As)(1)–C(7)	103(1)
C(1)–(Sn,As)(1)–C(13)	103(1)
C(7)–(Sn,As)(1)–C(13)	102(1)
(Sn,As)(1)–C(1)–C(2)	123(2)
(Sn,As)(1)–C(1)–C(6)	118(2)
C(2)–C(1)–C(6)	119(2)
C(1)–C(2)–C(3)	119(3)
C(2)–C(3)–C(4)	121(3)
C(3)–C(4)–C(5)	119(3)
C(4)–C(5)–C(6)	122(3)
C(5)–C(6)–C(1)	119(3)
(Sn,As)(1)–C(7)–C(8)	120(2)
(Sn,As)(1)–C(7)–C(12)	118(2)
C(8)–C(7)–C(12)	121(3)
C(7)–C(8)–C(9)	118(3)
C(8)–C(9)–C(10)	119(4)
C(9)–C(10)–C(11)	123(4)
C(10)–C(11)–C(12)	117(3)
C(11)–C(12)–C(7)	121(4)
(Sn,As)(1)–C(13)–C(14)	118(2)
(Sn,As)(1)–C(13)–C(18)	123(1)
C(14)–C(13)–C(18)	119(2)
C(13)–C(14)–C(15)	119(3)
C(14)–C(15)–C(16)	122(3)
C(15)–C(16)–C(17)	118(3)
C(16)–C(17)–C(18)	121(3)
C(17)–C(18)–C(13)	121(2)
Sn(3)–(Sn,As)(2)–C(19)	116.6(9)
Sn(3)–(Sn,As)(2)–C(25)	113.1(8)
Sn(3)–(Sn,As)(2)–C(31)	115.7(7)
C(19)–(Sn,As)(2)–C(25)	103(1)
C(19)–(Sn,As)(2)–C(31)	104(1)
C(25)–(Sn,As)(2)–C(31)	102(1)
(Sn,As)(2)–C(19)–C(20)	118(2)
(Sn,As)(2)–C(19)–C(24)	121(2)
C(20)–C(19)–C(24)	121(3)
C(19)–C(20)–C(21)	119(3)
C(20)–C(21)–C(22)	121(3)
C(21)–C(22)–C(23)	120(4)
C(22)–C(23)–C(24)	122(4)
C(23)–C(24)–C(19)	118(3)
(Sn,As)(2)–C(25)–C(26)	116(2)
(Sn,As)(2)–C(25)–C(30)	123(2)
C(26)–C(25)–C(30)	120(2)
C(25)–C(26)–C(27)	120(3)
C(26)–C(27)–C(28)	120(3)
C(27)–C(28)–C(29)	121(4)
C(28)–C(29)–C(30)	119(3)
C(29)–C(30)–C(25)	120(3)
(Sn,As)(2)–C(31)–C(32)	122(2)
(Sn,As)(2)–C(31)–C(36)	118(2)
C(32)–C(31)–C(36)	120(3)
C(31)–C(32)–C(33)	118(2)
C(32)–C(33)–C(34)	121(3)
C(33)–C(34)–C(35)	120(3)
C(34)–C(35)–C(36)	120(3)
C(35)–C(36)–C(31)	120(3)

* The superscript I denotes the equivalent position $\bar{x}$, $\bar{y}$, $\bar{z}$, with respect to the reference molecule at x, y, z.

TABLE 3

Main vibrational bands (cm^{-1})

Absorption	Assignment	Absorption	Assignment
3 080w	ν(CH)	1 080ms	δ(CH) (in-plane)
3 050w		1 070(sh)	
1 760w	$\nu_1 + \nu_4$	1 030m	δ(CH) (in-plane) + ν(NO)
1 730w		1 025(sh)	
1 585m	ν(CC)	1 002ms	ring
1 485s		818m	$\delta(NO_2)$
1 440vs		745(sh)	δ(CH) (out-of-plane)
1 420vs,br	ν_{sym}(NO)	735vs	
1 390vs	ν(CC)	692vs	δ(CH) (out-of-plane)
1 340w		670(sh)	
1 315(sh)		480(sh)	ring
1 288vs	ν_{asym}(NO)	468s	
1 188m	β(CH)	460(sh)	
1 162m		333(sh)	ν(SnC) + ν(AsC)
1 090(sh)	δ(CH) (in-plane)	315m	

co-ordinated oxygen atoms bridging two metal atoms, so that NO_3 itself acts as a bridge as in silver(III) cryptate;[10] (*iii*) the nitrate group is doubly bidentate with all the oxygen atoms involved in co-ordination and one of them in a symmetric or asymmetric bridging situation, as in μ-(*o*-allylphenyl)dimethylarsine-bis[nitratosilver(I)],[11] nitrato(triphenylstannio)tin(II),[1] and (2-aminobenzothiazolato)nitratotin(II).[12]

Unusual behaviour in bridging nitrato-compounds occurs preferentially with silver or tin derivatives. In the present compound the bridging behaviour of NO_3 shows one more difference as it is bidentate with one oxygen bridging two different Sn^{II} atoms. The variations in the structural parameters of the nitrate group are dependent on the extent of the co-ordination to the metal atom: as is usually observed, the oxygen atoms

[10] R. Wiest and R. Weiss, *J.C.S. Chem. Comm.*, 1973, 678.

[11] M. K. Cooper, R. S. Nyholm, P. W. Carreck, and M. McPartlin, *J.C.S. Chem. Comm.*, 1974, 343.

[12] M. Nardelli, C. Pelizzi, and G. Pelizzi, *J.C.S. Dalton*, 1975, 1595.

which are involved in metal interactions have lengthened bonds to their nitrogen.

The atoms Sn(3), N, O(1), O(2), O(3), Sn(3^{I}), N^{I}, O(1^{I}), O(2^{I}), O(3^{I}) all lie close to a plane. The equation of the mean least-squares plane for these atoms is: $-0.6906X - 0.6562Y - 0.3042Z = 0$, where X,Y,Z are the co-ordinates (Å) referred to orthogonal axes. The deviations of each of the atoms from the plane are as follows: Sn(3) 0.00, N 0.00, O(1) 0.06, O(2) −0.10, O(3) 0.04, Sn(3^{I}) 0.00, N^{I} 0.00, O(1^{I}) −0.06, O(2^{I}) 0.10, and O(3^{I}) −0.04 Å.

The splitting value ($\Delta\nu$ 132 cm^{-1}) of the N–O stretching band is smaller than that usually found for bidentate nitrate groups;[6,13,14] the covalent character of NO_3 is also confirmed by the $\nu_1 + \nu_4$ combination band which gives rise to two fairly weak absorptions at 1 760 and 1 730 cm^{-1}, while both ν_1 and ν_2 bands are more consistent with ionic nitrate.[15]

The six phenyl rings are planar as expected. The average of the 36 C–C distances and the 36 C–C–C angles are 1.37 Å and 120° respectively and none of the individual values differs significantly from these mean values. The mutual dihedral angles (°) formed by the three phenyl rings in each Ph_3 moiety are as follows:

C(1)—C(6) ··· C(7)—C(12)	83. 1
C(7)—C(12) ··· C(13)—C(18)	87.5
C(1)—C(6) ··· C(13)—C(18)	83.4
C(19)—C(24) ··· C(25)—C(30)	71.8
C(25)—C(30) ··· C(31)—C(36)	77.5
C(19)—C(24) ··· C(31)—C(36)	75.2

These values can be justified by packing requirements.

The packing of the dimeric molecules is due to van der Waals forces as evidenced by the intermolecular contacts, none being less than 3.40 Å. The two shortest interactions involve the unco-ordinated oxygen atom:

C(22) ··· O(3)	($x - 1,y,z$)	3.50(2) Å
C(33) ··· O(3)	($\bar{x},\bar{y} - 1,\bar{z}$)	3.40(2) Å

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