C(18)	0.4905 (14)	0.0982 (11)	−0.2454 (7)	0.066 (4)
C(19)	0.3537 (13)	−0.0862 (10)	−0.1548 (7)	0.064 (4)
C(20)	0.2483 (14)	−0.1288 (10)	−0.0563 (7)	0.065 (4)
N(1)	0.2276 (11)	−0.0389 (8)	0.0221 (6)	0.064 (3)
N(2)	0.3995 (11)	0.0437 (9)	−0.1578 (6)	0.065 (3)

Table 2. *Selected geometric parameters* (Å, °)

Au(1)—Br(1)	2.420 (1)	Au(1)—Br(3)	2.416 (1)
Au(1)—Br(1*A*)	2.420 (1)	Au(1)—Br(3*A*)	2.416 (1)
Au(2)—Br(2)	2.386 (1)	Au(2)—Br(2*A*)	2.386 (1)
C(1)—C(2)	1.424 (15)	C(1)—C(6)	1.428 (15)
C(1)—N(1)	1.346 (12)	C(2)—C(3)	1.335 (15)
C(3)—C(4)	1.424 (18)	C(4)—C(5)	1.341 (18)
C(5)—C(6)	1.431 (15)	C(6)—C(7)	1.421 (16)
C(7)—C(8)	1.359 (14)	C(8)—C(9)	1.501 (15)
C(8)—C(20)	1.396 (15)	C(9)—C(10)	1.533 (15)
C(10)—C(11)	1.501 (15)	C(11)—C(12)	1.385 (14)
C(11)—C(19)	1.390 (14)	C(12)—C(13)	1.385 (16)
C(13)—C(14)	1.425 (15)	C(13)—C(18)	1.423 (14)
C(14)—C(15)	1.303 (18)	C(15)—C(16)	1.439 (16)
C(16)—C(17)	1.354 (15)	C(17)—C(18)	1.397 (15)
C(18)—N(2)	1.369 (12)	C(19)—C(20)	1.478 (13)
C(19)—N(2)	1.336 (13)	C(20)—N(1)	1.316 (13)
Br(1)—Au(1)—Br(3)	90.0 (1)	Br(1)—Au(1)—Br(1*A*)	180.0 (1)
Br(3)—Au(1)—Br(1*A*)	90.0 (1)	Br(1)—Au(1)—Br(3*A*)	90.0 (1)
Br(3)—Au(1)—Br(3*A*)	180.0 (1)	Br(1*A*)—Au(1)—Br(3*A*)	90.0 (1)
Br(2)—Au(2)—Br(2*A*)	180.0 (1)	C(2)—C(1)—C(6)	117.8 (9)
C(2)—C(1)—N(1)	120.1 (9)	C(6)—C(1)—N(1)	122.2 (9)
C(1)—C(2)—C(3)	120.4 (10)	C(2)—C(3)—C(4)	122.0 (11)
C(3)—C(4)—C(5)	119.9 (11)	C(4)—C(5)—C(6)	120.0 (12)
C(1)—C(6)—C(5)	119.7 (10)	C(1)—C(6)—C(7)	116.6 (9)
C(5)—C(6)—C(7)	123.7 (10)	C(6)—C(7)—C(8)	121.0 (10)
C(7)—C(8)—C(9)	122.5 (10)	C(7)—C(8)—C(20)	116.4 (9)
C(9)—C(8)—C(20)	121.0 (9)	C(8)—C(9)—C(10)	113.3 (10)
C(9)—C(10)—C(11)	113.0 (8)	C(10)—C(11)—C(12)	122.5 (9)
C(10)—C(11)—C(19)	120.2 (9)	C(12)—C(11)—C(19)	117.2 (9)
C(11)—C(12)—C(13)	122.0 (9)	C(12)—C(13)—C(14)	124.0 (10)
C(12)—C(13)—C(18)	119.0 (9)	C(14)—C(13)—C(18)	117.0 (10)
C(13)—C(14)—C(15)	121.0 (10)	C(14)—C(15)—C(16)	122.7 (11)
C(15)—C(16)—C(17)	118.1 (11)	C(16)—C(17)—C(18)	120.6 (10)
C(13)—C(18)—C(17)	120.6 (9)	C(13)—C(18)—N(2)	117.1 (9)
C(17)—C(18)—N(2)	122.2 (9)	C(11)—C(19)—C(20)	121.2 (9)
C(11)—C(19)—N(2)	121.3 (9)	C(20)—C(19)—N(2)	117.5 (8)
C(8)—C(20)—C(19)	118.9 (9)	C(8)—C(20)—N(1)	126.3 (9)
C(19)—C(20)—N(1)	114.8 (9)	C(1)—N(1)—C(20)	117.5 (9)
C(18)—N(2)—C(19)	123.2 (8)		

The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* (Sheldrick, 1991) was used for all calculations and drawings.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, selected short intermolecular contacts selected bond lengths and angles, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: HU1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Symmetrical Tetrasubstituted Tin Compounds: Tetrakis(2-methoxyphenyl)tin and Tetrakis(2-methoxybenzyl)tin

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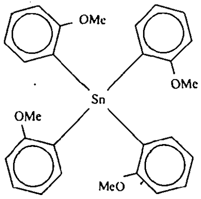
Abstract

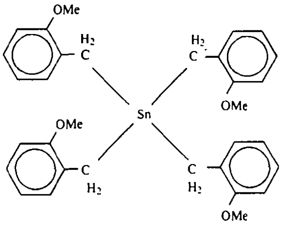
The title compound tetrakis(2-methoxyphenyl)tin, $[Sn(C_7H_7O)_4]$, crystallizes in the triclinic space group $P\bar{1}$ with two molecules in the asymmetric unit. The coordination at the Sn atoms is distorted tetrahedral with C—Sn—C angles between 105.79 (16) and 112.47 (15)°. The Sn—C distances are in the range 2.135 (4)–2.150 (4) Å and the Sn···O intramolecular distances are between 3.042 and 3.128 (3) Å. The related molecule tetrakis(2-methoxybenzyl)tin, $[Sn(C_8H_9O)_4]$, crystallizes in space group $P\bar{4}2_1c$ and has crystallographic $\bar{4}$ symmetry, with Sn—CH_2 2.165 (6) Å and an intramolecular Sn···O separation of 3.241 (5) Å.

Comment

Only a limited number of crystal structures have been reported for tetraaryltin ($SnAr_4$) derivatives. These include tetrakis(*o*-tolyl)tin (Belsky, Simonenko, Reikhsfeld & Saratov, 1983), tetrakis(*p*-thioanisyl)tin (Wharf & Simard, 1987), tetrakis(*p*-tolyl)tin (Karipides & Wolfe, 1975), tetrakis(pentafluorophenyl)tin (Karipides, Forman, Thomas & Reed, 1974), tetrakis(3-methylphenyl)tin (Karipides & Oertel, 1977), tetraphenyltin (Chieh & Trotter, 1970; Akhmed & Aleksandrov, 1970; Belsky, Simonenko, Reikhsfeld & Saratov, 1983; Engelhardt, Leung, Raston & White, 1982), tetrakis(*p*-anisyl)tin (Wharf & Simard, 1987), tetrakis(*p*-methylsulfonylphenyl)tin (Wharf, Simard & Lamparski, 1990) and tetrakis(*p*-ethoxyphenyl)tin (Wharf & Simard, 1991). All have crystallographic $\bar{4}$ symmetry except for the last two compounds; the *p*-methylsulfonylphenyl derivative has only twofold crystallographic symmetry and the *p*-ethoxyphenyl has no imposed symmetry. Our analysis of tetrakis(2-methoxyphenyl)tin, (I), shows that in this case also there is no crystallographically imposed symmetry at all and there are two independent molecules in space group $P\bar{1}$. These are shown in Fig. 1 with our numbering scheme. For tetrabenzyl tin derivatives, only the parent tetrakis(benzyl)tin compound, [Sn(C_7H_7)$_4$], has been reported (Davies, Jarvis & Kilbourn, 1971) and it has no imposed crystallographic symmetry. Tetrakis(2-methoxybenzyl)tin, (II), is shown in Fig. 2 and has crystallographic $\bar{4}$ symmetry.



(I)



(II)

The eight independent Sn—C$_{ar}$ bond lengths in (I) range from 2.135 (4) to 2.150 (4) Å [mean 2.143 (5) Å] and are within the range [2.120 (8)–2.152 (5) Å] previously reported for the other SnAr$_4$ species noted above. The C—Sn—C valency angles in (I) range from 105.79 (16) to 112.47 (15)°, showing the same variation from regular tetrahedral angles as found in other SnAr$_4$ systems. The unique Sn—CH$_2$ bond length in (II) is 2.165 (5) Å with the Sn—CH$_2$—C$_{ar}$ and mean CH$_2$—Sn—CH$_2$ valence angles being 113.1 (3) and 109.4 (2)°, respectively. The Sn···O separation of 3.241 (5) Å is too great for any interaction. In tetrakis(benzyl)tin (Davies, Jarvis & Kilbourn, 1971) the Sn—CH$_2$ distances are in the range 2.17–2.19 Å with Sn—CH$_2$—C$_{ar}$ 112° and C$_{ar}$—Sn—C$_{ar}$ 108–110°.

The eight independent methoxy groups in (I) adopt essentially the same conformation with respect to the aromatic rings to which they are bonded, with the torsion angles C1*n*—C2*n*—O1*n*—C7*n* (*n* = 1–8) between −163.5 (7) and −178.8 (6)° [mean −176 (9)°]. It is noteworthy that associated with this conformation is a marked difference at the exocyclic angles C1*n*—C2*n*—O1*n* and C3*n*—C2*n*—O1*n* (*n* = 1–8); these have mean values of 114.6 (5) and 124.0 (3)°, respectively, and along with the enlarged angle at the sp^3-hybridized O atom, C2*n*—O1*n*—C7*n* (*n* = 1–8) mean 119.0 (7)°, serve to increase the C7*n*···C3*n* (*n* = 1–8) intramolecular separation. There is also a similar, but smaller, trend in the angles Sn—C1*n*—C2*n* and Sn—C1*n*—C6*n* (*n* = 1–8), but it is less pronounced, with mean values 119.5 (10) and 122.6 (10)°, respectively. The effect of these small conformational changes leads to Sn···O intramolecular distances in the range 3.042 (3)–3.128 (3) Å. Such distances can be compared with analogous separations in [SnI(2-MeOC$_6$H$_4$)$_3$] [2.965 (7), 3.045 (6) and 3.061 (7) Å (Howie, Ross, Wardell & Low, 1994)] and in [SnBr$_2$(2-MeOC$_6$H$_4$)$_2$] [2.90 (1) and 2.92 (1) Å (Ross, Wardell, Ferguson & Low, 1994)]. It is also noteworthy that in the *o*-tolyl derivative [Sn(2-MeC$_6$H$_4$)$_4$], which has $\bar{4}$ symmetry, the exocyclic H$_3$C—C—C angles are 124.4 (4) and 117.5 (4)°, effectively increasing the CH$_3$···Sn separation. The methoxy group in (II) adopts the same conformation as found in (I) with respect to the phenyl ring and shows the same pattern for exocyclic C—C—O valence angles with C1—C2—O21 [114.5 (5)°] much less than C3—C2—O21 [123.7 (6)°]; the torsion angle C1—C2—O21—C21 is −177.5 (11)°. The conformation adopted by the benzyl group with respect to the Sn atom is defined by the torsion angles Sn—CH$_2$—C$_{ar}$—C$_{ar}$ which have values 67.6 (5) and −110.3 (7)° for Sn1—C11—C1—C2 and Sn1—C11—C1—C6, respectively; the Sn atom is 1.864 (6) Å from the best plane of the benzyl C atoms.

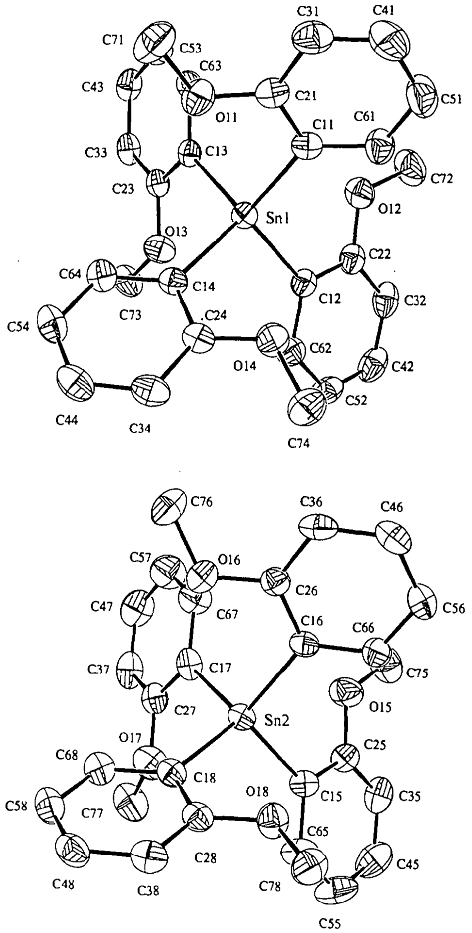


Fig. 1. Views of the two independent molecules of tetrakis(2-methoxyphenyl)tin, (I), with the crystallographic numbering scheme. The non-H atoms are shown as displacement ellipsoids drawn at the 30% probability level. H atoms are omitted for clarity.

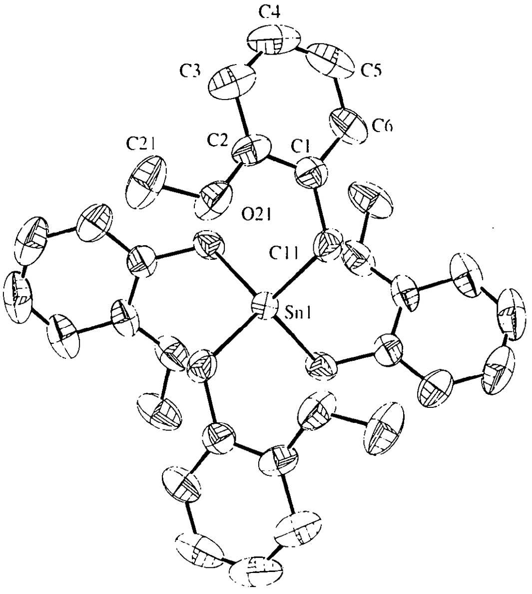


Fig. 2. A view of tetrakis(2-methoxybenzyl)tin, (II), with the crystallographic numbering scheme. The non-H atoms are shown as displacement ellipsoids drawn at the 35% probability level. H atoms are omitted for clarity.

In (I) the mean methoxy bond lengths are CH_3—O 1.408 (7), C_{ar}—O 1.372 (6) Å; the corresponding values in (II) are 1.444 (8) and 1.360 (8) Å.

An examination of the O···O intramolecular distances in (I) shows that the distorted O_4 tetrahedron formed by the bonds is very similar for both independent molecules with O···O separations in the range 4.427–5.382 (5) Å; the corresponding O···O distances in (II), which has $\bar{4}$ symmetry, are 4.573 (9)–5.619 (9) Å.

The phenyl ring geometry is as anticipated in both (I) and (II) with mean C_{ar}—C_{ar} 1.380 (12) Å in (I) and 1.377 (14) Å in (II). In both structures the intermolecular contacts correspond to normal van der Waals interactions and an examination of the structures with *PLATON* (Spek, 1992) showed that there were no solvent accessible voids in the lattices.

Experimental

Compound (I) was prepared from [MgBr(2-MeOC$_6$H$_4$)] and SnCl$_4$ in thf/benzene and was recrystallized from ethyl acetate/petroleum ether (b.p. 333–353K) mixture. Compound (II) was prepared by the reaction of SnCl$_4$ with the Grignard reagent [MgCl(*o*-MeOC$_6$H$_4$CH$_2$)], which was synthesized according to the method of Van Campen, Meisner & Parmeter (1948).

Compound (I)

Crystal data

$[Sn(C_7H_7O)_4]$
$M_r = 547.21$
Triclinic
$P\bar{1}$
$a = 9.1105$ (10) Å
$b = 16.512$ (2) Å
$c = 17.9600$ (12) Å
$\alpha = 77.648$ (15)°
$\beta = 78.482$ (13)°
$\gamma = 81.618$ (16)°
$V = 2571.1$ (5) Å^3
$Z = 4$
$D_x = 1.414$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.50$–$15.50°$
$\mu = 1.02$ mm^{-1}
$T = 293$ K
Plate
0.44 × 0.28 × 0.17 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.725$, $T_{max} = 0.795$
11 165 measured reflections
11 165 independent reflections
7334 observed reflections
$[I > 2.5\sigma(I)]$

$\theta_{max} = 26.90°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 21$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity variation: 2.5%

Refinement

Refinement on F
$R = 0.028$
$wR = 0.039$
$S = 1.01$
7333 reflections
595 parameters
H atoms refined as riding (C—H 0.95 Å)

$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (I)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Sn1	0.00667 (3)	0.225727 (16)	0.199948 (15)	0.04322 (15)
C11	0.0725 (5)	0.1203 (2)	0.2842 (2)	0.048 (2)
C21	−0.0384 (5)	0.0701 (3)	0.3265 (2)	0.053 (2)
C31	−0.0055 (7)	0.0032 (3)	0.3839 (3)	0.074 (3)
C41	0.1391 (8)	−0.0139 (4)	0.3974 (4)	0.093 (4)
C51	0.2498 (6)	0.0323 (4)	0.3560 (4)	0.088 (4)
C61	0.2169 (5)	0.1004 (3)	0.2998 (3)	0.065 (3)
O11	−0.1776 (4)	0.0927 (2)	0.3058 (2)	0.067 (2)
C71	−0.2966 (7)	0.0464 (4)	0.3460 (4)	0.093 (4)
C12	0.1943 (5)	0.2923 (3)	0.1402 (2)	0.050 (2)
C22	0.2856 (5)	0.3203 (3)	0.1806 (3)	0.055 (3)
C32	0.4066 (5)	0.3643 (3)	0.1416 (3)	0.070 (3)
C42	0.4350 (6)	0.3808 (3)	0.0615 (4)	0.078 (3)
C52	0.3441 (6)	0.3553 (3)	0.0213 (3)	0.074 (3)
C62	0.2247 (5)	0.3113 (3)	0.0597 (3)	0.060 (3)
O12	0.2447 (4)	0.3033 (2)	0.25911 (19)	0.067 (2)
C72	0.3414 (7)	0.3220 (4)	0.3046 (4)	0.092 (4)
C13	−0.1633 (4)	0.3048 (2)	0.2599 (2)	0.043 (2)
C23	−0.2111 (4)	0.3819 (2)	0.2200 (2)	0.046 (2)
C33	−0.3141 (5)	0.4381 (3)	0.2567 (3)	0.058 (3)
C43	−0.3705 (5)	0.4146 (3)	0.3347 (3)	0.062 (3)
C53	−0.3270 (5)	0.3377 (3)	0.3754 (3)	0.060 (3)
C63	−0.2231 (5)	0.2829 (3)	0.3383 (2)	0.049 (2)
O13	−0.1488 (4)	0.39764 (19)	0.14301 (18)	0.063 (2)
C73	−0.1954 (6)	0.4735 (3)	0.0962 (3)	0.073 (3)
C14	−0.0848 (5)	0.1905 (3)	0.1122 (2)	0.047 (2)
C24	0.0077 (5)	0.1443 (3)	0.0612 (2)	0.050 (2)

C34	−0.0447 (6)	0.1248 (3)	0.0007 (3)	0.063 (3)
C44	−0.1916 (7)	0.1514 (3)	−0.0087 (3)	0.070 (3)
C54	−0.2869 (6)	0.1963 (3)	0.0414 (3)	0.066 (3)
C64	−0.2315 (5)	0.2167 (3)	0.1015 (3)	0.056 (3)
O14	0.1512 (4)	0.1199 (2)	0.07611 (18)	0.0646 (19)
C74	0.2618 (7)	0.0896 (4)	0.0182 (3)	0.088 (4)
Sn2	−0.25678 (3)	0.720934 (17)	0.297072 (16)	0.04729 (17)
C15	−0.1694 (5)	0.7444 (3)	0.3922 (3)	0.052 (3)
C25	−0.2611 (5)	0.7895 (3)	0.4437 (3)	0.057 (3)
C35	−0.2082 (7)	0.8115 (3)	0.5025 (3)	0.074 (4)
C45	−0.0607 (9)	0.7859 (4)	0.5105 (4)	0.094 (5)
C55	0.0321 (7)	0.7417 (5)	0.4620 (4)	0.100 (5)
C65	−0.0220 (6)	0.7206 (4)	0.4023 (3)	0.076 (4)
O15	−0.4061 (4)	0.8109 (2)	0.4302 (2)	0.078 (2)
C75	−0.5109 (7)	0.8535 (4)	0.4808 (4)	0.099 (4)
C16	−0.4488 (4)	0.6532 (2)	0.3411 (2)	0.044 (2)
C26	−0.5407 (5)	0.6407 (3)	0.2922 (2)	0.049 (2)
C36	−0.6657 (5)	0.5974 (3)	0.3201 (3)	0.062 (3)
C46	−0.7006 (5)	0.5657 (3)	0.3981 (3)	0.065 (3)
C56	−0.6124 (5)	0.5767 (3)	0.4477 (3)	0.059 (3)
C66	−0.4884 (5)	0.6196 (3)	0.4192 (2)	0.051 (2)
O16	−0.4952 (4)	0.6736 (2)	0.21493 (18)	0.069 (2)
C76	−0.5989 (8)	0.6837 (6)	0.1645 (3)	0.116 (6)
C17	−0.3271 (5)	0.8404 (3)	0.2328 (2)	0.056 (3)
C27	−0.2231 (6)	0.8976 (3)	0.2036 (3)	0.059 (3)
C37	−0.2654 (7)	0.9800 (3)	0.1681 (3)	0.075 (4)
C47	−0.4138 (9)	1.0025 (4)	0.1624 (4)	0.092 (5)
C57	−0.5172 (7)	0.9481 (4)	0.1901 (4)	0.093 (4)
C67	−0.4747 (6)	0.8678 (3)	0.2245 (3)	0.072 (3)
O17	−0.0780 (4)	0.8657 (2)	0.2128 (2)	0.081 (2)
C77	0.0344 (7)	0.9186 (5)	0.2014 (4)	0.101 (5)
C18	−0.0909 (4)	0.6522 (3)	0.2259 (2)	0.049 (2)
C28	−0.0332 (4)	0.5716 (3)	0.2567 (2)	0.049 (2)
C38	0.0673 (5)	0.5235 (3)	0.2111 (3)	0.058 (3)
C48	0.1139 (5)	0.5560 (3)	0.1342 (3)	0.062 (3)
C58	0.0612 (5)	0.6350 (3)	0.1021 (3)	0.064 (3)
C68	−0.0416 (5)	0.6827 (3)	0.1479 (3)	0.059 (3)
O18	−0.0833 (4)	0.54595 (19)	0.33423 (18)	0.062 (2)
C78	−0.0331 (6)	0.4644 (3)	0.3703 (3)	0.068 (3)

Table 2. *Selected geometric parameters* (Å, °) *for* (I)

Sn1—C11	2.147 (4)	Sn2—C15	2.150 (4)
Sn1—C12	2.143 (4)	Sn2—C16	2.139 (4)
Sn1—C13	2.147 (4)	Sn2—C17	2.142 (4)
Sn1—C14	2.139 (4)	Sn2—C18	2.135 (4)
C21—O11	1.369 (6)	C25—O15	1.373 (6)
O11—C71	1.405 (6)	O15—C75	1.401 (7)
C22—O12	1.363 (6)	C26—O16	1.381 (5)
O12—C72	1.418 (6)	O16—C76	1.403 (7)
C23—O13	1.370 (5)	C27—O17	1.379 (6)
O13—C73	1.411 (6)	O17—C77	1.398 (7)
C24—O14	1.373 (5)	C28—O18	1.370 (5)
O14—C74	1.415 (6)	O18—C78	1.417 (6)
C11—Sn1—C12	111.75 (16)	C15—Sn2—C16	109.27 (16)
C11—Sn1—C13	107.47 (15)	C15—Sn2—C17	106.44 (17)
C11—Sn1—C14	112.47 (15)	C15—Sn2—C18	111.44 (16)
C12—Sn1—C13	110.85 (15)	C16—Sn2—C17	108.89 (16)
C12—Sn1—C14	105.79 (16)	C16—Sn2—C18	109.77 (15)
C13—Sn1—C14	108.51 (15)	C17—Sn2—C18	110.93 (16)
Sn1—C11—C21	117.9 (3)	Sn2—C15—C25	119.3 (3)
Sn1—C11—C61	123.8 (3)	Sn2—C15—C65	123.0 (4)
C11—C21—O11	114.6 (4)	C15—C25—O15	114.4 (4)
C31—C21—O11	124.3 (4)	C35—C25—O15	123.7 (4)
C21—O11—C71	118.6 (4)	C25—O15—C75	119.4 (4)
Sn1—C12—C22	121.0 (3)	Sn2—C16—C26	121.2 (3)
Sn1—C12—C62	120.8 (3)	Sn2—C16—C66	122.3 (3)
C12—C22—O12	115.0 (4)	C16—C26—O16	114.6 (4)
C32—C22—O12	124.1 (4)	C36—C26—O16	123.7 (4)
C22—O12—C72	118.3 (4)	C26—O16—C76	118.5 (4)
Sn1—C13—C23	118.6 (3)	Sn2—C17—C27	119.1 (4)
Sn1—C13—C63	122.8 (3)	Sn2—C17—C67	124.0 (3)
C13—C23—O13	114.6 (4)	C17—C27—O17	113.5 (4)
C33—C23—O13	124.0 (4)	C37—C27—O17	124.6 (4)
C23—O13—C73	119.0 (4)	C27—O17—C77	120.6 (5)
Sn1—C14—C24	119.5 (3)	Sn2—C18—C28	119.7 (3)
Sn1—C14—C64	121.8 (3)	Sn2—C18—C68	122.7 (3)
C14—C24—O14	115.1 (4)	C18—C28—O18	114.7 (4)
C34—C24—O14	123.7 (4)	C38—C28—O18	124.0 (4)
C24—O14—C74	118.6 (4)	C28—O18—C78	118.7 (4)

Compound (II)

Crystal data

[Sn(C$_8$H$_9$O)$_4$]
$M_r = 603.32$
Tetragonal
$P\bar{4}2_1c$
$a = 14.2266\ (17)$ Å
$c = 7.3943\ (7)$ Å
$V = 1496.6\ (2)$ Å^3
$Z = 2$
$D_x = 1.339$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 12.50$–$14.75°$
$\mu = 0.84$ mm^{-1}
$T = 293$ K
Plate
0.40 × 0.30 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.6540$, $T_{max} = 0.8165$
4687 measured reflections
2182 independent reflections
1425 observed reflections [$I > 2.5\sigma(I)$]

$R_{\rm int} = 0.018$
$\theta_{\max} = 29.90°$
$h = -19 \rightarrow 19$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 10$
3 standard reflections
frequency: 60 min
intensity variation: 2.5%

Refinement

Refinement on F
$R = 0.025$
$wR = 0.037$
$S = 1.05$
1425 reflections
84 parameters
H atoms refined as riding (C—H 0.95 Å)

$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.57$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn1	0	0	0	0.0579 (2)
O21	−0.1009 (3)	−0.1251 (3)	−0.3107 (7)	0.099 (2)
C11	0.0703 (3)	−0.1021 (3)	−0.1697 (8)	0.076 (2)
C1	0.0247 (3)	−0.1956 (3)	−0.1666 (6)	0.073 (2)
C2	−0.0639 (3)	−0.2057 (3)	−0.2422 (7)	0.080 (2)
C3	−0.1104 (4)	−0.2919 (4)	−0.2441 (9)	0.112 (4)
C4	−0.0665 (6)	−0.3671 (4)	−0.1648 (12)	0.138 (6)
C5	0.0186 (6)	−0.3598 (4)	−0.0856 (12)	0.133 (5)
C6	0.0643 (3)	−0.2740 (3)	−0.0870 (8)	0.104 (3)
C21	−0.1945 (4)	−0.1274 (7)	−0.3859 (9)	0.128 (5)

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

Sn1—C11	2.165 (5)	O21—C21	1.444 (8)
O21—C2	1.360 (8)	C11—C1	1.480 (7)
C11—Sn1—C11$^{\rm i}$	109.6 (2)	C11—C1—C2	119.0 (5)
C11—Sn1—C11$^{\rm ii}$	109.1 (2)	C11—C1—C6	123.7 (5)
C2—O21—C21	118.8 (6)	O21—C2—C1	114.5 (5)
Sn1—C11—C1	113.1 (3)	O21—C2—C3	123.7 (6)

Symmetry codes: (i) $y, -x, -z$; (ii) $-x, -y, z$.

Compound (I) crystallized in the triclinic system; space group $P\bar{1}$ was assumed and confirmed by the successful refinement. Compound (II) crystallized in the tetragonal system and a survey of the reflection intensities revealed that the Laue group was $4/mmm$. This, with the systematic absences (hhl absent if $l = 2n + 1$, $h00$ absent if $h = 2n + 1$), determined the space group as $P\bar{4}2_1/c$; furthermore, $Z = 2$ required that (II) have $\bar{4}$ symmetry. For both (I) and (II) all non-H atoms were refined by full-matrix least-squares calculations on F. All H atoms were clearly visible in difference maps and they were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. Space group $P\bar{4}2_1/c$ is chiral and refinement with $\eta = -1$ converged with higher values of R and wR (0.029 and 0.037, respectively) than were found for $\eta = +1$ (0.025 and 0.030, respectively) (Rodgers, 1981); thus the absolute configuration is as shown.

For both compounds, data collection and cell refinement: Enraf–Nonius CAD-4; data reduction, program used to solve and refine structure, and software used to prepare material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989); molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Benzyltriphenyllead and Dibenzyldiphenyllead

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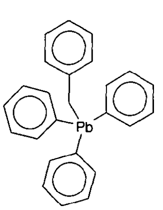
Abstract

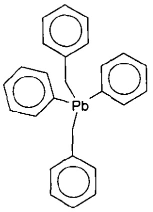
In benzyltriphenyllead, [$Pb(C_6H_5)_3(C_7H_7)$], (I), and dibenzyldiphenyllead, [$Pb(C_6H_5)_2(C_7H_7)_2$], (II), each Pb atom is in the centre of a distorted tetrahedron. In (I) the C(Bz)—Pb—C(Ph) angles [110.4 (3)–113.1 (3)°] are larger than the C(Ph)—Pb—C(Ph) angles [106.6 (3)–107.5 (3)°] suggesting a steric effect of the benzyl group (Bz). The angles in (II) [C(Bz)—Pb—C(Bz) 107.8 (3)°; C(Bz)—Pb—C(Ph) 104.6 (3) and 113.1 (3)°; C(Ph—Pb—C(Ph) 111.1 (3)°] reflect a more distinct distortion, but no similar steric effect of the benzyl groups is clearly recognizable.

Comment

The present work continues structural studies of benzyllead compounds, such as tetrakis(2-chlorobenzyl)lead (Fahrenkamph, Schürmann & Huber, 1993) or benzyldiphenyllead bromide (Fahrenkamph, Schürmann & Huber, 1994). The structures of the title compounds have been determined in the context of studies of possible specific effects of unsubstituted and substituted benzyl ligands (Bz) on the bonding of Pb in compounds $Bz_{4-n}PbR_n$ (R = organo group; n = 0–3), which in contrast to comparable tetraorganolead compounds exhibit unusual properties. (Compounds containing more than one benzyl ligand, for instance, are coloured and sensitive to air and light.)



(I)



(II)

In (I) and (II) the coordination polyhedron around Pb is a slightly distorted tetrahedron formed by four C atoms. The Pb—C(Ph) distances in both compounds are not significantly different and are similar to the distance of 2.194 (6) Å found in $PbPh_4$ (Preut & Huber, 1993),

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