STRUCTURAL STUDIES IN MAIN GROUP CHEMISTRY

XVIII *. DISORDERED STRUCTURE OF TRIPHENYLSILOXYTRIPHENYLLEAD(IV)

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(Received March 8th, 1976)

Summary

The crystal structure of triphenylsiloxytriphenyllead(IV), $Ph_3SiOPbPh_3$, has been investigated by X-ray diffraction. The structure was solved by Patterson and Fourier methods using diffractometer intensity data, and refined to a final R value of 0.098 for 2197 observed independent non-zero reflections. Crystals are monoclinic, space group $P2_1/a$, with a 20.383(14), b 12.998(10), c 11.648(7) Å, β 101.60(4)°, Z 4. Crystals are composed of discrete monomeric $Ph_3SiOPbPh_3$ molecules, in which both silicon and lead are four-coordinated [Si—O 1.87(3), Si—C 1.83(3)—1.90(3), Pb—O 2.01(3), Pb—C 2.06(4)—2.27(4) Å, angle O—Pb—C 96(1)—105(1)°, angle C—Pb—C 117(2)—121(2)°, angle O—Si—C 103(1)—106(1)°, angle C—Si—C 111(1)—118(2)°, angle Si—O—Pb 142(1)°], however the crystals are disordered such that a significant number of molecules (ca. 16%) are reversed, i.e. lead atoms occupy silicon sites and vice versa. Unit cell data are also reported for triphenylsiloxytriphenyltin(IV), $Ph_3SiOSnPh_3$, in which disorder precludes solution of the structure.

Introduction

There is a paucity of structural data for organolead(IV) derivatives. Unit cell data has been reported for Ph_2PbCl_2, from which a structure involving infinite, chlorine-bridged chains was inferred [2]. Full structures have, however, been determined for Ph_6Pb_2 [3], $(Ph_2BrPb)_4C$ [4], $[Me_4N][Ph_2Pb(OAc)_3]$ [5], and the two lead—transition metal complexes, $Me_2Pb[Fe(CO)_2(C_5H_5)]_2$ [6] and $Pt(PPh_3)_2(PbPh_3)Ph$ [7]. Unlike trialkyllead(IV) alkoxides which are extremely reactive

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[8], triphenylsiloxytriphenyllead(IV), $Ph_3SiOPbPh_3$, is relatively inert and does not undergo ready aerobic hydrolysis as do the corresponding alkoxides, presumably due to the steric bulk of the triphenylsiloxy groups and also because of the electronic nature of the Si—O—Pb linkage. For these reasons we have investigated by X-ray diffraction the structure of this compound which is reported in this paper.

Experimental

The compound was synthesised by the azeotropic dehydration of a 1 : 1 mixture of triphenylsilanol and triphenyllead hydroxide in benzene as previously reported [9], and crystals suitable for X-ray diffraction study were obtained by re-

TABLE 1

FINAL FRACTIONAL ATOMIC COORDINATES

Atom	x/a	y/b	z/c
Pb [a]	0.1226(0)	0.1515(1)	0.2564(1)
Si [b]	0.3030(3)	0.1752(4)	0.2626(4)
O(1)	0.2140(12)	0.2111(20)	0.2630(18)
C(1)	0.3455(16)	0.3042(21)	0.2526(22)
C(2)	0.3396(18)	0.3814(27)	0.3293(31)
C(3)	0.3739(20)	0.4791(37)	0.3305(30)
C(4)	0.4152(20)	0.4957(31)	0.2448(28)
C(5)	0.4204(21)	0.4152(26)	0.1676(33)
C(6)	0.3891(17)	0.3230(28)	0.1754(29)
C(7)	0.3360(22)	0.1126(25)	0.4065(28)
C(8)	0.4020(20)	0.1105(34)	0.4458(36)
C(9)	0.4314(23)	0.6333(40)	0.5479(43)
C(10)	0.3901(26)	0.0164(29)	0.6195(32)
C(11)	0.3246(29)	0.0255(34)	0.5885(33)
C(12)	0.2914(21)	0.0722(28)	0.4781(31)
C(13)	0.3004(18)	0.0992(28)	0.1297(25)
C(14)	0.2591(22)	0.1295(35)	0.0219(31)
C(15)	0.2602(25)	0.0567(48)	—0.0774(36)
C(16)	0.2975(29)	—0.0345(48)	—0.0690(45)
C(17)	0.3377(27)	—0.0621(41)	0.0470(45)
C(18)	0.3386(17)	0.0097(32)	0.1448(33)
C(19)	0.0607(16)	0.2271(41)	0.1135(29)
C(20)	0.0298(24)	0.3172(33)	0.1318(38)
C(21)	—0.0149(23)	0.3640(33)	0.0399(40)
C(22)	—0.0242(23)	0.3217(34)	—0.0718(35)
C(23)	0.0014(22)	0.2323(44)	—0.0893(36)
C(24)	0.0490(20)	0.1833(32)	—0.0004(33)
C(25)	0.1083(23)	0.2160(23)	0.4310(29)
C(26)	0.0409(22)	0.1869(37)	0.4563(34)
C(27)	0.0371(26)	0.2315(34)	0.5670(32)
C(28)	0.0882(28)	0.2740(39)	0.6378(33)
C(29)	0.1519(25)	0.2906(35)	0.6116(34)
C(30)	0.1638(21)	0.2593(33)	0.5025(33)
C(31)	0.1415(20)	—0.0039(29)	0.2501(34)
C(32)	0.1667(23)	—0.0509(33)	0.1609(39)
C(33)	0.1769(21)	—0.1567(45)	0.1566(29)
C(34)	0.1661(24)	—0.2274(33)	0.2493(33)
C(35)	0.1460(26)	—0.1793(35)	0.3403(37)
C(36)	0.1273(19)	—0.0787(32)	0.3413(30)

[a] Population parameter 0.8601. [b] Population parameter 1.7441.

peated recrystallisation from benzene. A specimen of approximate dimensions 0.3 × 0.3 × 0.3 mm³ was mounted directly onto a glass fibre, and used for both the initial photography and subsequent intensity data collection.

Crystal data. $C_{36}H_{30}OPbSi$, mol.wt. 713.52, monoclinic space group $P2_1/a$ from systematic absences $h0l$ for $h = 2n$ and $0k0$ for $k = 2n$, with a 20.383(14), b 12.998(10), c 11.648(7) Å, β 101.60(4)°, $Z = 4$, $F(000) = 1400$, Mo-K_α radiation, $\lambda = 0.71069$ Å, $\mu(\text{Mo}-K_\alpha) = 13.89$ cm^{-1}.

Structure determination and refinement. The initial cell parameters and space group were determined from oscillation and zero- and first-layer Weissenberg photographs. The intensities of 2197 independent non-zero reflections were measured using a Hilger & Watts Y290 four-circle diffractometer. These were cor-

TABLE 2

FINAL ANISOTROPIC THERMAL PARAMETERS [a,b]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pb	7.61(23)	6.26(23)	3.66(22)	0.03(13)	1.78(7)	−0.16(10)
Si	10.25(56)	5.80(50)	3.53(34)	−2.58(37)	1.13(37)	0.33(26)
O(1)	8.2(19)	11.8(21)	5.2(13)	4.0(17)	3.9(13)	−2.2(14)
C(1)	6.9(23)	3.1(18)	2.6(14)	1.9(17)	3.2(13)	−0.8(13)
C(2)	8.2(29)	4.5(26)	8.2(25)	1.8(20)	5.9(22)	3.8(18)
C(3)	5.9(29)	13.5(40)	5.4(28)	1.5(28)	−0.6(20)	2.9(24)
C(4)	8.6(30)	9.2(32)	4.5(19)	1.1(26)	0.2(21)	0.3(21)
C(5)	8.8(35)	2.5(24)	8.8(27)	−2.1(22)	2.5(24)	−0.3(19)
C(6)	4.9(24)	6.5(31)	6.7(23)	0.7(20)	−0.3(19)	−0.6(19)
C(7)	12.8(36)	3.5(21)	5.1(20)	−0.2(22)	0.7(23)	−1.8(16)
C(8)	5.7(29)	11.3(38)	7.6(28)	4.3(26)	−3.3(22)	−1.6(24)
C(9)	7.6(35)	12.2(43)	9.8(34)	−0.1(33)	0.4(29)	−1.9(32)
C(10)	9.7(39)	4.7(26)	7.8(25)	6.2(29)	0.4(26)	−0.6(20)
C(11)	16.4(50)	9.0(35)	5.7(24)	9.0(36)	4.9(29)	2.0(23)
C(12)	11.1(36)	6.1(27)	7.3(25)	−6.1(25)	0.7(23)	0.2(20)
C(13)	6.7(27)	8.7(26)	3.8(17)	−3.6(22)	1.3(18)	−2.3(17)
C(14)	11.3(36)	11.3(41)	5.2(24)	−1.0(31)	0.5(23)	0.5(23)
C(15)	11.3(43)	16.4(54)	6.1(24)	−6.8(40)	5.0(28)	0.2(33)
C(16)	13.2(52)	12.4(51)	11.6(40)	−1.4(43)	4.5(36)	−2.6(38)
C(17)	14.7(48)	13.4(46)	13.7(44)	−3.5(39)	9.2(39)	−3.7(38)
C(18)	6.1(26)	8.4(33)	9.8(28)	0.6(23)	6.4(22)	−1.0(25)
C(19)	1.5(21)	18.6(48)	6.4(21)	−1.7(26)	2.3(17)	−4.5(26)
C(20)	10.1(38)	6.9(36)	9.7(31)	3.4(28)	2.1(29)	1.4(25)
C(21)	10.1(36)	7.7(33)	12.4(36)	−1.2(29)	7.2(30)	0.5(29)
C(22)	10.2(35)	6.1(35)	10.4(29)	−1.9(27)	6.1(26)	2.6(24)
C(23)	6.2(34)	12.9(45)	7.1(28)	1.4(32)	−0.4(24)	−1.4(29)
C(24)	6.8(30)	12.1(38)	6.4(24)	−4.2(27)	1.7(22)	−2.5(24)
C(25)	16.3(44)	1.1(18)	6.9(22)	0.1(23)	6.4(26)	1.0(25)
C(26)	8.4(34)	14.7(46)	7.6(27)	1.3(31)	0.0(24)	6.4(29)
C(27)	14.3(45)	10.0(35)	4.6(22)	4.4(32)	4.9(26)	−1.0(23)
C(28)	13.8(47)	11.3(40)	4.1(22)	1.0(35)	0.9(27)	−0.8(25)
C(29)	12.8(42)	10.7(35)	6.2(26)	−2.3(32)	3.2(27)	−2.6(24)
C(30)	7.3(30)	8.6(33)	7.7(26)	−2.8(24)	−0.5(23)	−1.0(24)
C(31)	9.4(35)	7.7(28)	6.8(26)	3.0(23)	3.6(25)	0.7(23)
C(32)	10.1(38)	4.7(28)	10.9(36)	2.6(27)	1.6(29)	2.1(24)
C(33)	9.3(33)	12.2(43)	4.7(21)	−0.5(37)	1.6(20)	−0.7(30)
C(34)	15.1(43)	7.8(33)	5.6(22)	7.7(32)	−0.0(27)	−1.0(24)
C(35)	14.2(47)	6.3(37)	8.8(29)	−4.8(32)	1.4(29)	−0.6(24)
C(36)	5.7(26)	8.6(32)	4.8(23)	−1.7(24)	−1.6(19)	1.0(21)

[a] Values of U_{ij} as listed have been multiplied by 10^2. [b] U_{ij} are of the form exp $-2\pi^2[h^2U_{11}a^{*2} + k^2U_{22}b^{*2} + l^2U_{33}c^{*2} + hkU_{12}a^*b^* + hlU_{13}a^*c^* + klU_{23}b^*c^*]$.

TABLE 3

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

Bond distances

Si—O(1)	1.87(3)	Pb—O(1)	2.01(3)
Si—C(1)	1.90(3)	Pb—C(19)	2.12(4)
Si—C(7)	1.86(3)	Pb—C(25)	2.27(4)
Si—C(13)	1.83(3)	Pb—C(31)	2.06(4)
C(1)—C(2)	1.36(5)	C(19)—C(20)	1.37(7)
C(2)—C(3)	1.45(6)	C(20)—C(21)	1.40(6)
C(3)—C(4)	1.44(6)	C(21)—C(22)	1.39(6)
C(4)—C(5)	1.40(5)	C(22)—C(23)	1.31(7)
C(5)—C(6)	1.37(5)	C(23)—C(24)	1.42(6)
C(6)—C(1)	1.41(5)	C(24)—C(19)	1.42(5)
C(7)—C(8)	1.33(6)	C(25)—C(26)	1.51(7)
C(8)—C(9)	1.36(6)	C(26)—C(27)	1.43(6)
C(9)—C(10)	1.44(7)	C(27)—C(28)	1.31(7)
C(10)—C(11)	1.32(8)	C(28)—C(29)	1.41(8)
C(11)—C(12)	1.46(5)	C(29)—C(30)	1.40(6)
C(12)—C(7)	1.45(6)	C(30)—C(25)	1.38(6)
C(13)—C(14)	1.42(5)	C(31)—C(32)	1.38(7)
C(14)—C(15)	1.50(7)	C(32)—C(33)	1.40(7)
C(15)—C(16)	1.40(9)	C(33)—C(34)	1.47(6)
C(16)—C(17)	1.48(7)	C(34)—C(35)	1.36(7)
C(17)—C(18)	1.47(7)	C(35)—C(36)	1.36(6)
C(18)—C(13)	1.39(5)	C(36)—C(31)	1.51(6)

Bond angles

Pb(1)—O(1)—Si	142(1)	O(1)—Si—C(1)	103(1)
O(1)—Pb—C(19)	105(1)	O(1)—Si—C(7)	106(2)
O(1)—Pb—C(25)	96(1)	O(1)—Si—C(13)	106(1)
O(1)—Pb—C(31)	102(1)	C(1)—Si—C(7)	111(1)
C(19)—Pb—C(25)	117(2)	C(1)—Si—C(13)	111(1)
C(19)—Pb—C(31)	121(2)	C(7)—Si—C(13)	118(2)
C(25)—Pb—C(31)	117(1)		
C(24)—C(19)—C(20)	120(4)	C(6)—C(1)—C(2)	116(3)
C(19)—C(20)—C(21)	120(4)	C(1)—C(2)—C(3)	123(4)
C(20)—C(21)—C(22)	119(4)	C(2)—C(3)—C(4)	118(4)
C(21)—C(22)—C(23)	121(4)	C(3)—C(4)—C(5)	118(4)
C(22)—C(23)—C(24)	121(4)	C(4)—C(5)—C(6)	121(4)
C(23)—C(24)—C(19)	118(4)	C(5)—C(6)—C(1)	124(3)
Pb—C(19)—C(24)	121(3)	Si—C(1)—C(2)	120(3)
Pb—C(19)—C(20)	120(3)	Si—C(1)—C(6)	123(3)
C(30)—C(25)—C(26)	131(4)	C(12)—C(7)—C(8)	120(3)
C(25)—C(26)—C(27)	107(4)	C(7)—C(8)—C(9)	123(4)
C(26)—C(27)—C(28)	124(5)	C(8)—C(9)—C(10)	119(4)
C(27)—C(28)—C(29)	125(4)	C(9)—C(10)—C(11)	119(4)
C(28)—C(29)—C(30)	119(4)	C(10)—C(11)—C(12)	123(5)
C(29)—C(30)—C(25)	113(4)	C(11)—C(12)—C(13)	115(4)
Pb—C(25)—C(26)	112(2)	Si—C(7)—C(8)	118(3)
Pb—C(25)—C(30)	117(3)	Si—C(7)—C(12)	121(3)
C(36)—C(31)—C(32)	113(4)	C(18)—C(13)—C(14)	124(3)
C(31)—C(32)—C(33)	123(4)	C(13)—C(14)—C(15)	114(4)
C(32)—C(33)—C(34)	123(4)	C(14)—C(15)—C(16)	125(4)
C(33)—C(34)—C(35)	113(4)	C(15)—C(16)—C(17)	118(5)
C(34)—C(35)—C(36)	125(4)	C(16)—C(17)—C(18)	118(5)
C(35)—C(36)—C(31)	121(4)	C(17)—C(18)—C(13)	121(3)
Pb—C(31)—C(32)	124(3)	Si—C(13)—C(14)	121(3)
Pb—C(31)—C(36)	123(3)	Si—C(13)—C(18)	116(2)

TABLE 4

EQUATIONS OF LEAST SQUARES MEAN PLANES, AND DEVIATIONS (Å) OF ATOMS THEREFROM

(a) Equations in the form $pI + qJ + rK = s$, where I, J, K are orthogonal coordinates in A, related to the monoclinic coordinates in the following way: $I = x + z \cos \beta$, $J = y$, $K = z \sin \beta$.

Plane	p	q	r	s
1. C(1)—C(6)	0.6670	−0.3565	0.6542	4.7989
2. C(7)—C(12)	−0.1020	0.8745	0.4718	2.9116
3. C(13)—C(18)	0.8372	0.4947	0.2330	5.1630
4. C(19)—C(24)	0.8251	0.5170	−0.2277	2.0354
5. C(25)—C(30)	−0.2407	0.8869	−0.3944	0.2160
6. C(31)—C(36)	0.8497	0.1615	0.5020	3.3648

(b) Deviations from planes

Plane 1: C(1) −0.0189, C(2) −0.0043, C(3) 0.0161, C(4) −0.0054, C(5) −0.0179, C(6) 0.0309
Plane 2: C(7) −0.0333, C(8) 0.0277, C(9) 0.0069, C(10) −0.0356, C(11) 0.0292, C(12) 0.0052
Plane 3: C(13) 0.0032, C(14) −0.0081, C(15) 0.0003, C(16) 0.0118, C(17) −0.0169, C(18) 0.0096
Plane 4: C(19) −0.0026, C(20) 0.0014, C(21) −0.0212, C(22) 0.0448, C(23) −0.0457, C(24) 0.0233
Plane 5: C(25) 0.0461, C(26) −0.0578, C(27) 0.0389, C(28) −0.0006, C(29) −0.0189, C(30) −0.0077
Plane 6: C(31) 0.0131, C(32) 0.0005, C(33) 0.0077, C(34) −0.0334, C(35) 0.0600, C(36) −0.0480

b

a

o - Pb
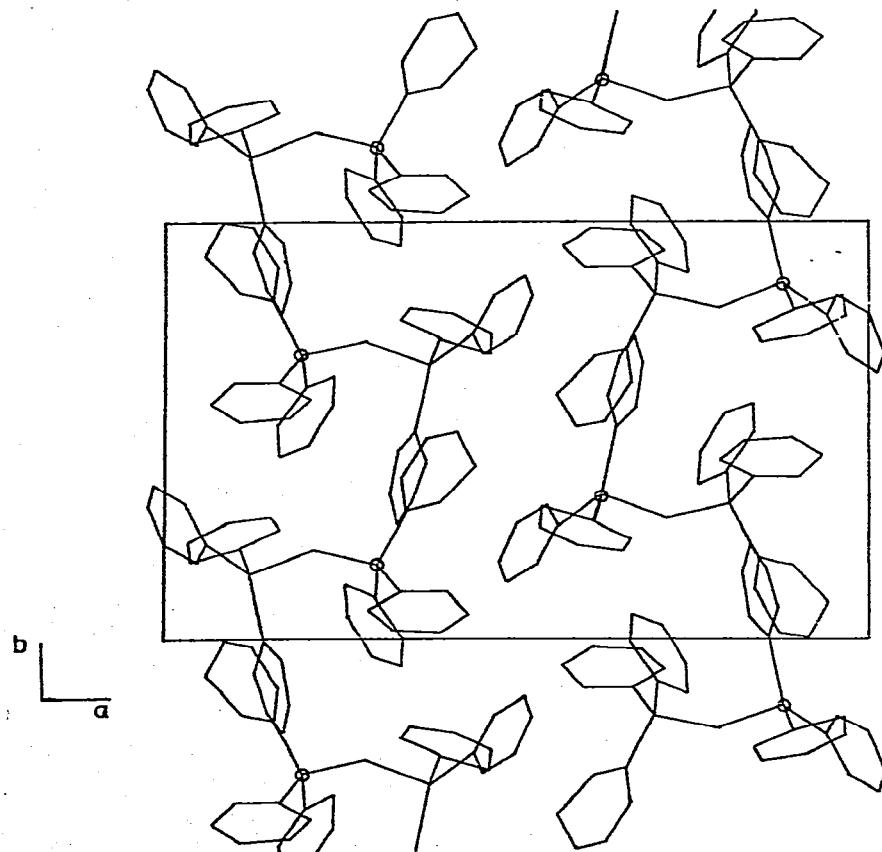
Fig. 1. Projection of the unit cell on to the *ab* plane.

rected for Lorentz and polarisation effects, but none was made for absorption since the μ value was low.

The position of the lead atom was determined from a three-dimensional Patterson synthesis, and this was used to phase the initial structure factor calculation. A Fourier synthesis at this stage yielded the positions of both the silicon and oxygen atoms. Two cycles of isotropic least-squares refinement followed by a second Fourier synthesis afforded the positions of the remaining 36 carbon atoms. However, although least-squares refinement resulted in a lowering of the R-value to 0.165, the isotropic temperature factor for the silicon became progressively more negative, indicating the presence of more electron density than could be accounted for by one silicon atom. Therefore, at this stage, the population parameters of both the lead and silicon atoms were allowed to vary. Further (block-diagonal) least-squares refinement, with the lead and silicon atoms varying anisotropically, resulted in the two population parameters settling at 0.8601 (Pb) and 1.7441 (Si), at which values they were left invariant. Ten cycles of block-diagonal least-squares refinement gave convergence at an R-value of 0.098.
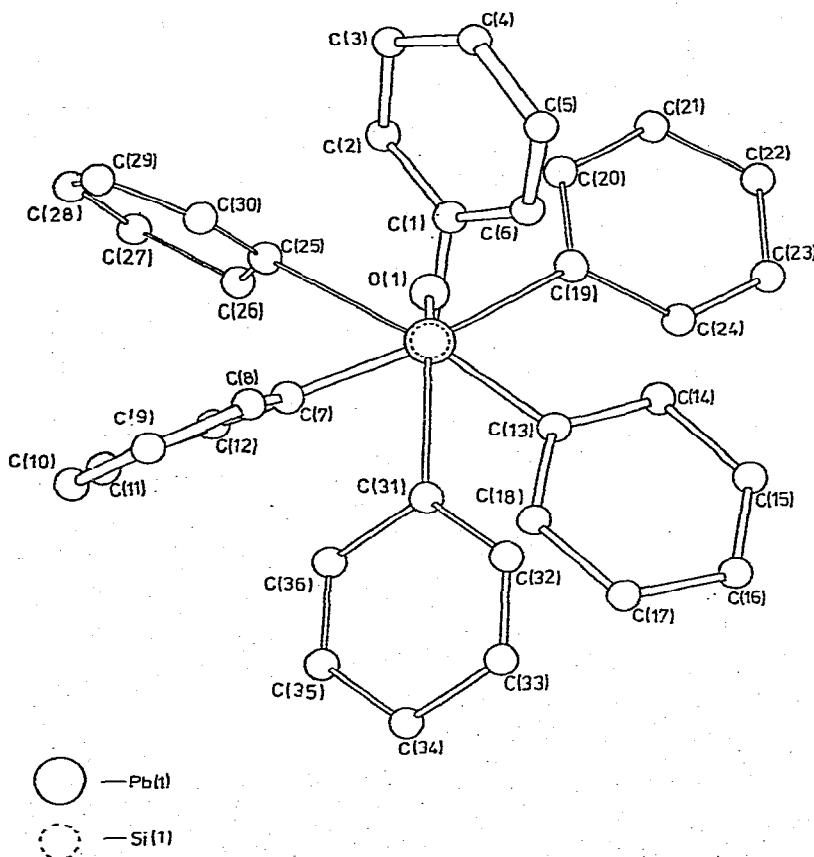

Fig. 2a. View of the molecule showing the eclipsed conformation of the two triphenylmetal groups.
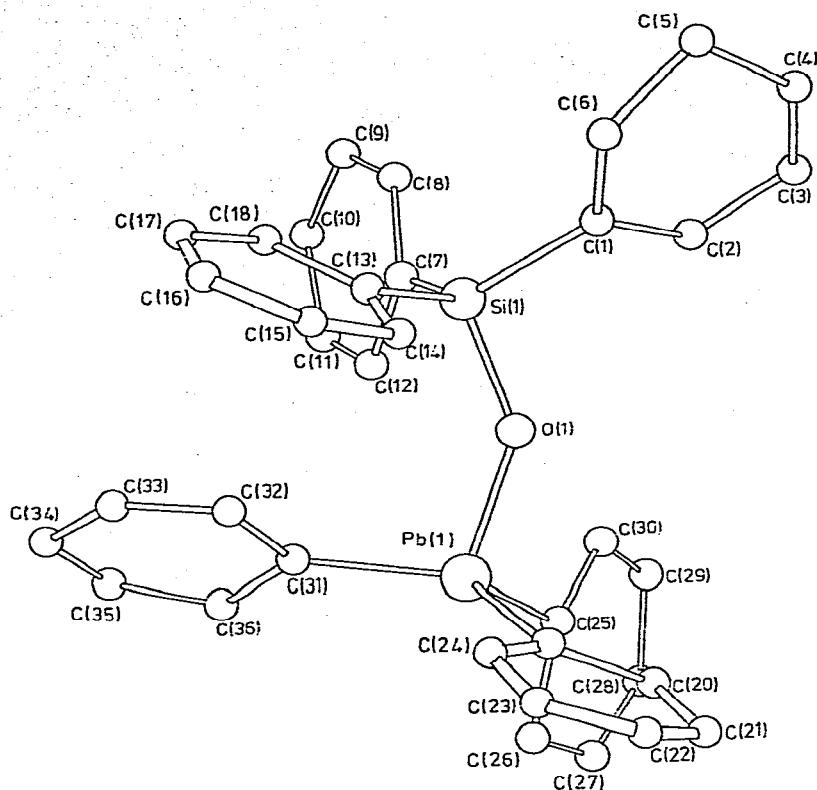
Fig. 2b. View of the molecule showing the geometry at silicon and at lead.

Final fractional atomic coordinates and anisotropic thermal parameters are listed in Tables 1 and 2, respectively, whilst intramolecular bond distances and angles are given in Table 3 and least-squares mean planes data in Table 4. Atomic numbering is illustrated in Fig. 2.

A Table of the final observed and calculated structure factors can be obtained from the authors.

Discussion

As may be seen from Fig. 1, crystals of $Ph_3SiOPbPh_3$ are composed of isolated monomeric molecules. Both silicon and lead atoms are four-coordinated by three phenyl groups and the oxygen atom in a distorted tetrahedral manner (Fig. 2b), the two triphenylmetal moieties enjoying a "staggered" conformation relative to each other (Fig. 2a). The bulkiness of both of the triphenylmetal groups allows the occurrence of a random disorder in the packing of the molecules in the crystal involving a molecular reversal in which silicon and lead atoms are effectively interchanged. The degree of this disorder is readily computed from the deviations of the lead and silicon site population parameters from unity, which correspond to ca. 16% molecular reversal. The presence of the disorder, not surprisingly, has an adverse effect on the general quality of the structural data, resulting in rather

high standard deviations and irregular phenyl groups, although the chemically important data, that for the $C_3SiOPbC_3$ skeleton, are quite reasonable.

Although the lead—carbon bond distances (2.06(4)—2.27(4) Å) are similar to those found in other organolead(IV) derivatives (2.26, 2.28 Å in $Me_2Pb[Fe(CO)_2-(C_5H_5)]_2$ [6]; 2.20 Å (Pb—Ph), 2.25 Å (Pb—C) in $(Ph_2BrPb)_4C$ [4]; 2.16 Å in $[Me_4N][Ph_2Pb(OAc)_3]$ [5]) *, the lead—oxygen distance (2.01(3) Å) is significantly shorter than that in $[Me_4N][Ph_2Pb(OAc)_3]$ (mean 2.55 Å), the only other lead(IV)—oxygen bonded derivative for which structural data is available [5]. The silicon—carbon bond distances are normal, and compare well with those in Ph_4Si (1.872 Å) [10], $(Ph_3SiO)_2CrO_2$ (1.847—1.939 Å) [11], and $Ph_2Me_6Si_4O_4$ (1.861 Å) [12]. The silicon—oxygen bond distance is unusually long (1.87(3) Å) compared with $(H_3Si)_2O$ (1.634 Å) [13], $Ph_2Me_6Si_4O_4$ (mean 1.629 Å) [12], and other heterometallosiloxanes such as $(MeHgOSiMe_3)_4$ (1.61 Å) [14], $(MeBeOSiMe_3)_4$ (1.68 Å) [15] and $(Ph_3SiO)_2CrO_2$ (1.60 Å) [11], although the Si—O—Pb bond angle (142(1)°) is surprisingly similar to that in $(H_3Si)_2O$ (144.1°) [13]. Such a value for the Si—O—Pb bond angle is, however, perhaps not too surprising since the Si—O—Cr angles in $(Ph_3SiO)_2CrO_2$ are 133.1 and 162.2° [11].

Crystal data for triphenylsiloxytriphenyltin(IV), $Ph_3SiOSnPh_3$. $C_{36}H_{30}OSiSn$, mol. wt. 625.41, space group $P2_1/n$ from systematic absences $0k0$ for $k = 2n + 1$ and $h0l$ for $h + l = 2n + 1$, a 19.888(10), b 17.614(9), c 8.842(6) Å, β 90.18(2)°, $Z = 4$.

In the case of $Ph_3SiOSnPh_3$, the Patterson synthesis was relatively complex, and although it was possible to locate reasonable positions for both the silicon and tin atoms, further solution of the structure proved impossible, presumably due to disorder similar to that observed for the lead analogue.

Acknowledgements

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* The lead—carbon distance in $Pt(PPh_3)_2(PbPh_3)Ph$ is unusually short (1.84 Å) [7].