were: L_{11} 51(2), L_{12} 0, L_{13} $-1(1)$, L_{22} 10(1), L_{23} 0, L_{33} 37(1) 10^{-4} rad^2; S_{11} 0(2), S_{12} 4(1), S_{13} 0(1), S_{21} 4(1), S_{22} 0(2), S_{23} 5(1), S_{31} 0(1), S_{32} 5(1), S_{33} 0(1) 10^{-4} rad Å; T_{11} 165(4), T_{12} 0, T_{13} 23(3), T_{22} 252(4), T_{23} 0, T_{33} 219(4) 10^{-4} Å^2 (relative to an origin at $x=0·4805$, $y=0·2500$, $z=0·7421$ which makes S symmetric). These values were used in calculating the librationally corrected bond lengths given in Table 3 and Fig. 1.

The molecular geometry is quite unlike the rather asymmetric structure found for P_4Se_5 (Penney & Sheldrick, 1971) and α-P_4S_5. On the other hand β-P_4S_5 has the same molecular symmetry, space group, and number of atoms in special positions as As_4S_5 (Whitfield, 1973), but the crystallographic mirror plane in As_4S_5 lies through the atoms corresponding to P(3), P(3′) and S(1), *i.e.* it is at right angles to the crystallographic mirror plane in β-P_4S_5. The result is that the cell dimensions and atomic coordinates are quite different in the two structures. The structure of β-P_4S_5 may be derived from that of P_4S_7 (Vos *et al.*, 1965) by removing both terminal atoms (attached to P(1) and P(2) in the β-P_4S_5 notation). The valence angles at P(1) and P(2) are 2–5° smaller in β-P_4S_5 than in P_4S_7 (consistent with lone pair–bond pair repulsion); the valence angles at S are 3–5° larger in β-P_4S_5 to compensate, resulting in the unusually large value of 115·5 (2)° for P(1)–S(1)–P(2) in β-P_4S_5. Ignoring libration, the P–P bond in β-P_4S_5 is 0·042 (7) Å shorter than the remarkably long P–P bond in P_4S_7, whereas the bonds between bridging S and P(1) and P(2) are an average of 0·024 Å longer in β-P_4S_5.

We are grateful to the Science Research Council for a contribution towards the cost of the diffractometer, and for a maintenance grant to A.M.G. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by G.M.S.

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Trimethyllead Acetate

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Abstract. $C_5H_{12}O_2Pb$, orthorhombic, *Pnma*, $a=10·77$ (2), $b=7·82$ (2), $c=9·58$ (2) Å, $U=807$ Å^3, $Z=4$, $D_x=2·56$ g cm^{-3}. Trimethyllead acetate is isostructural with the analogous Sn compound; planar trimethyllead groups are linked by bridging acetate groups to form infinite chains along **a**, with approximately trigonal bipyramidal coordination of Pb. The two Pb–O distances are significantly different [2·327 (24) and 2·555 (25) Å]. Other mean distances and angles are: Pb–C, 2·181 (25); C–O, 1·223 (30); C–C, 1·519 (36) Å; O–Pb–O, 169·7 (8); O–C–O, 121·5 (24); O–C–C, 119·3 (20)°. The structure was refined to an *R* of 0·052 for 420 unique diffractometer data.

Introduction. X-ray crystallographic studies of many apparently tetravalent organotin derivatives [*e.g.* $(CH_3)_3SnO_2CCH_3$ (Chih & Penfold, 1973), $(CH_3)_3SnOCH_3$ (Domingos & Sheldrick, 1974) and $(CH_3)_3SnNCO.(CH_3)_3SnOH$ (Hall & Britton, 1972)] have established the tendency of Sn to increase its coordination number above four. It is likely that this tendency is continued in the structural chemistry of organolead derivatives, but apart from the structure of $(C_6H_5)_2PbCl_2$ (Mammi, Busetti & Del Pra, 1967) and the incomplete investigations of $(CH_3)_3PbCN$ (Chow & Britton, 1971) and $(CH_3)_2Pb(CN)_2$ (Konnert, Britton & Chow, 1972) there is little crystallographic evidence to confirm this.

Colourless crystals were obtained by recrystallization of a commercial sample of trimethyllead acetate from a methanol/acetone mixture, and were mounted in Lindemann glass capillary tubes. Most proved unsuitable for structural work, but data were eventually collected from two crystals $0·16\times0·07\times0·09$ mm

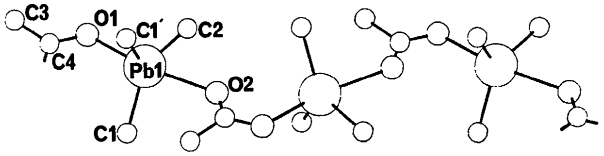

Fig. 1. Trimethyllead acetate, showing the chain along a.

(layers $0kl$ to $12kl$) and $0{\cdot}09 \times 0{\cdot}11 \times 0{\cdot}07$ mm (layers $h0l$ to $h8l$). Intensities were measured on a Stoe Stadi-2 automated two-circle diffractometer, with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å) and graphite crystal monochromator. 2036 reflexions were measured; Lp and absorption corrections were applied, and interlayer scale factors obtained by a linear least-squares analysis of common reflexions. Averaging equivalent reflexions led to 420 unique reflexions with $F > 2{\cdot}5\sigma(F)$ based on counting statistics. The cell dimensions were obtained by a least-squares fit to diffractometer ω measurements for 197 $h0l$ and 99 $0kl$ reflexions.

The systematic absences required the space group to be *Pnma* or $Pn2_1a$; the former was suggested by analogy with trimethyltin acetate, and confirmed by successful refinement of the structure. The coordinates of the Pb atom were derived from the Patterson function, and the C and O atoms located in successive difference syntheses. The structure was refined by full-matrix least-squares calculations, with anisotropic Pb and isotropic C and O atoms. The weighting scheme was $w = [\sigma^2(F) + 0{\cdot}0002F_o^2]^{-1}$, and complex neutral-atom scattering factors were employed. The refinement converged to $R' = \sum w^{1/2}\Delta / \sum w^{1/2}|F_o| = 0{\cdot}044$; the corresponding unweighted R was 0·052. The highest peak in the final difference map was about 2·1 e Å^{-3}. Atom coordinates and temperature factors are given in Table 1, and bond lengths and angles in Tables 2 and 3.*

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31243 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atom coordinates* ($\times 10^4$) *and isotropic temperature factors* (Å$^2 \times 10^3$)

	x/a	y/b	z/c	U
Pb(1)	840 (1)	2500	1784 (1)	*
O(1)	−798 (22)	2500	201 (22)	47 (5)
O(2)	2886 (20)	2500	3133 (28)	57 (6)
C(1)	328 (23)	92 (35)	2755 (27)	59 (7)
C(2)	2073 (29)	2500	−49 (37)	46 (9)
C(3)	−2852 (26)	2500	−517 (34)	39 (8)
C(4)	−1877 (26)	2500	628 (34)	33 (7)

* An anisotropic temperature factor was employed for Pb(1) in the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*)]$ with U_{11} to U_{12} 24 (1), 49 (1), 32 (1), 0, 3 (1) and 0 Å$^2 \times 10^3$ respectively.

Table 2. *Bond lengths* (Å)

Pb(1)–C(1)	2·171 (29)	Pb(1)–C(2)	2·202 (35)
Pb(1)–O(1)	2·327 (24)	Pb(1)–O(2)	2·555 (25)
O(1)—C(4)	1·232 (34)	O(2)—C(4)	1·214 (34)
C(3)—C(4)	1·519 (36)		

Table 3. *Bond angles* (°)

O(1)–Pb(1)–O(2)	169·7 (8)	C(1)–Pb(1)–O(1)	95·0 (9)
C(1)–Pb(1)–O(2)	90·2 (9)	C(2)–Pb(1)–O(1)	86·4 (12)
C(2)–Pb(1)–O(2)	83·3 (13)	C(1)–Pb(1)–C(2)	119·7 (9)
C(1)–Pb(1)–C(1′)	120·2 (16)	O(1)–C(4)—O(2)	121·5 (24)
O(1)–C(4)—C(3)	114·4 (25)	O(2)–C(4)—C(3)	124·1 (24)
C(4)–O(1)—Pb(1)	120·0 (22)	C(4)–O(2)—Pb(1)	132·5 (22)

Discussion. Trimethyllead groups are linked by acetates to form infinite zigzag –O–Pb–O– chains along **a** (Fig. 1), with trigonal bipyramidal coordination of Pb. All atoms except C(1) lie on the mirror planes perpendicular to **b**; the trimethyllead group is almost exactly planar. Trimethyllead acetate is therefore isostructural with trimethyltin acetate, and when allowance is made for the different covalent radii of the metal atoms, there are no significant differences in the bond lengths and angles in the two compounds. The Pb–O distances [2·327 (24) and 2·555 (25) Å] are, on average, shorter than those found in $[(C_6H_5)_2Pb(O_2CCH_3)_3]^-[N(CH_3)_4]^+$ [2·48 (2), 2·55 (2) and 2·63 (2) Å; Alcock, 1972], where however the lead is eight coordinated rather than five. The mean Pb–C distance of 2·181 (25) Å is similar to that found in $(CH_3)_4Pb$ [2·203 (10) Å; Wong & Schomaker, 1958].

We are grateful to the Science Research Council for providing the diffractometer, and for a maintenance grant to R.T. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by G.M.S.; Fig. 1 was drawn with the program *PLUTO* written by Dr S. Motherwell.

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