

C(11)—S(3)—C(12)—C(2)	138 (1)	C(3)—C(2)—C(12)—S(3)	98 (1)
C(6)—C(1)—C(2)—C(3)	3 (2)	C(2)—C(3)—C(4)—C(5)	-2 (3)
C(6)—C(1)—C(2)—C(12)	-174 (1)	C(3)—C(4)—C(5)—C(6)	4 (3)
C(7)—C(1)—C(2)—C(3)	-174 (1)	C(4)—C(5)—C(6)—C(1)	-2 (3)
C(7)—C(1)—C(2)—C(12)	9 (2)	S(1)—C(8)—C(9)—S(2)	65 (1)
C(2)—C(1)—C(6)—C(5)	-1 (2)	S(2)—C(10)—C(11)—S(3)	61 (1)
C(7)—C(1)—C(6)—C(5)	176 (1)		

Cell refinement, data collection and data reduction: Rigaku AFC-5S software. Program used to solve the structure: SHELXS86 (Sheldrick, 1985). Program used to refine the structure: XTAL3.0 (Hall & Stewart, 1990). Molecular graphics: PLUTO (Motherwell & Clegg, 1978). Programs used to prepare material for the publication: Xtal3.0 BONDLA and ATABLE.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: AB1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Benzyl(bromo)diphenyllead

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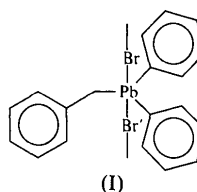
Abstract

The coordination polyhedron around Pb in the title compound, [PbBr(C₆H₅)₂(C₇H₇)], is a slightly distorted trigonal bipyramid, the trigonal plane being

formed by the C(Pb) atoms of the three organic ligands. The axial positions are occupied by Br atoms [Pb(1)—Br(1) 2.985 (2), Pb(1)—Br(1a) 2.885 (2) Å], each Br atom bridging neighbouring molecules to form infinite Br(1a)—Pb(1)—Br(1)—Pb(1b) chains [Br(1a)—Pb(1)—Br(1) 173.610 (15), Pb(1)—Br(1)—Pb(1b) 122.73 (5)°], *i.e.* catena-poly-[(benzylidiphenyllead)- μ -bromo].

Comment

The structures of three triorganolead halides are known to have been investigated by diffraction methods. In all cases, the Pb atom proved to be pentacoordinated: in Ph₃PbX (X = Cl, Br) intermolecular X—Pb coordination generates one-dimensional chains *via* unsymmetrical X—Pb—X links (Preut & Huber, 1977), while in [PbI(C₇H₇)(C₉H₁₂N)(C₇H₇O)] (van der Kooij, den Brinker & de Kok, 1985), a compound with three different R groups attached to the Pb atom, pentacoordination is attained by intramolecular N—Pb coordination. Pentacoordination of Pb and formation of infinite chains by way of halogen bridging was also inferred from vibrational studies for trimethyllead halides (Clark, Davies & Puddephatt, 1968) and for triphenyllead halides (Clark, Davies & Puddephatt, 1969). We report here the solid-state structure of a triorganolead compound, (I), with two types of R group on Pb.



The coordination polyhedron around Pb can be described as a slightly distorted trigonal bipyramid, with C(1), C(21) and C(31) in the equatorial plane and two Br atoms in the axial positions. The structure is similar to that of [PbBr(C₆H₅)₃] (Preut & Huber, 1977); however, some noteworthy differences exist. The distances Pb(1)—Br(1) and Pb(1)—Br(1a) are more similar to each other in the title compound [2.985 (2) and 2.885 (2) Å, respectively], than in [PbBr(C₆H₅)₃] [2.852 (1) and 3.106 (1) Å, respectively]. Hence, the difference between the Pb(1)—Br(1) and Pb(1)—Br(1a) distances of 0.100 Å in the title compound is less marked than in [PbBr(C₆H₅)₃] (0.254 Å), and this corresponds to a distinctly shorter distance of Pb from the plane defined by C(1), C(21) and C(31) in the title compound {0.052 (5) Å, compared to 0.127 (10) Å in [PbBr(C₆H₅)₃] (Preut & Huber, 1977)}. In this respect, the distortion of the trigonal bipyramid is smaller than in [PbBr(C₆H₅)₃];

however, the C—Pb—C angles in the equatorial plane deviate to a greater extent from the ideal values than in $[\text{PbBr}(\text{C}_6\text{H}_5)_3]$. This may be related to the more pronounced steric effect of the benzyl ligand, its phenyl group being located on the acute side of the enlarged angle C(1)—Pb(1)—C(31) [127.8 (3) Å].

Nevertheless, the title compound and $[\text{PbBr}(\text{C}_6\text{H}_5)_3]$, as well as $[\text{PbCl}(\text{C}_6\text{H}_5)_3]$ {which is very similar to $[\text{PbBr}(\text{C}_6\text{H}_5)_3]$: difference Pb(1)—Cl(1) minus Pb(1)—Cl(1a) = 0.241 Å; distance Pb(1) from the plane C(1), C(21), C(31) = 0.119 (10) Å (Preut & Huber, 1977)}, belong to a structural type of linear polymer. In the monomeric complex $[\text{PbI}(\text{C}_7\text{H}_7)(\text{C}_9\text{H}_{12}\text{N})(\text{C}_7\text{H}_7\text{O})]$ (van der Kooi, den Brinker & de Kok, 1985) I is less basic than the donor group $(\text{CH}_2)_2\text{N}$, thus intramolecular coordination is favoured over intermolecular bridging. In contrast to $[\text{PbX}(\text{C}_6\text{H}_5)_3]$, $[\text{SnCl}(\text{C}_6\text{H}_5)_3]$ was found to contain discrete molecules in the solid state with a nearly tetrahedral coordination polyhedron around the Sn atom (Bokii, Zakharova & Struchkov, 1970). The apparent tendency of triorganolead halides (having no basic substituents at the organo group) to form polymeric structures by way of halogen bridging, may be correlated to the higher coordination tendency of Pb.

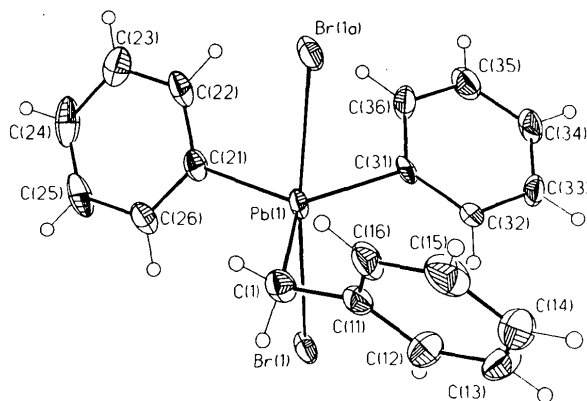


Fig. 1. View (*SHELXTL-Plus*; Sheldrick, 1987) of part of the crystal structure, showing the atom-numbering scheme.

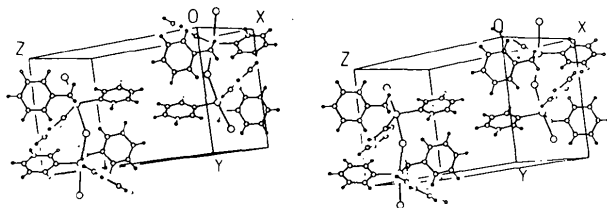


Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of the unit cell.

Experimental

The title compound was prepared by bromination of $\text{Ph}_3\text{PbCH}_2\text{C}_6\text{H}_5$ in pyridine at 243 K (Schlöttig, 1927).

Crystal data

$[\text{PbBr}(\text{C}_6\text{H}_5)_2(\text{C}_7\text{H}_7)]$
 $M_r = 532.43$
 Monoclinic
 $P2_1/c$
 $a = 9.741$ (4) Å
 $b = 10.287$ (5) Å
 $c = 16.790$ (9) Å
 $\beta = 92.05$ (4)°
 $V = 1681.4$ (14) Å³
 $Z = 4$
 $D_x = 2.103$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 43 reflections
 $\theta = 7.5$ – 15.0 °
 $\mu = 12.402$ mm⁻¹
 $T = 170$ (1) K
 Block
 $0.62 \times 0.20 \times 0.16$ mm
 Yellow

Data collection

Nicolet R3m/V diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical
 $T_{\min} = 0.470$, $T_{\max} = 1.000$
 3707 measured reflections
 2980 independent reflections
 2292 observed reflections
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 25.05$ °
 $h = -11 \rightarrow 1$
 $k = -1 \rightarrow 12$
 $l = -19 \rightarrow 19$
 6 standard reflections monitored every 300 reflections
 intensity variation: $< \pm 3.4\%$

Refinement

Refinement on F^2
 $R(F) = 0.0387$
 $wR(F^2) = 0.0941$
 $S = 1.07$
 2980 reflections
 192 parameters
 Only H-atom U 's refined
 Calculated weights
 $w = 1/[\sigma^2(F_o^2) + (0.0617P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 1.636$ e Å⁻³ (near Pb)
 $\Delta\rho_{\text{min}} = -3.889$ e Å⁻³
 Extinction correction: none
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8, 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	U_{eq}
Pb(1)	0.01089 (3)	0.05910 (4)	0.24372 (2)	0.02227 (13)
Br(1)	-0.07470 (10)	-0.19848 (9)	0.17684 (5)	0.0286 (2)
C(1)	-0.1936 (8)	0.1269 (10)	0.1994 (6)	0.026 (2)
C(11)	-0.2959 (9)	0.1390 (10)	0.2624 (6)	0.025 (2)
C(12)	-0.3559 (9)	0.0297 (10)	0.2976 (6)	0.033 (2)
C(13)	-0.4507 (9)	0.0445 (11)	0.3552 (7)	0.039 (3)
C(14)	-0.4902 (10)	0.1649 (11)	0.3807 (6)	0.038 (3)
C(15)	-0.4314 (10)	0.2736 (12)	0.3472 (7)	0.041 (3)
C(16)	-0.3341 (10)	0.2617 (11)	0.2895 (6)	0.031 (2)
C(21)	0.1815 (9)	0.0776 (10)	0.1609 (5)	0.025 (2)
C(22)	0.2979 (10)	0.1451 (11)	0.1812 (6)	0.031 (2)
C(23)	0.4022 (10)	0.1625 (11)	0.1272 (7)	0.038 (3)
C(24)	0.3848 (12)	0.1115 (12)	0.0507 (7)	0.048 (3)
C(25)	0.2679 (12)	0.0450 (12)	0.0296 (6)	0.045 (3)
C(26)	0.1666 (10)	0.0262 (10)	0.0844 (6)	0.032 (2)
C(31)	0.0631 (9)	-0.0384 (9)	0.3575 (5)	0.021 (2)

C(32)	-0.0311 (9)	-0.1137 (10)	0.3969 (5)	0.027 (2)
C(33)	0.0095 (10)	-0.1733 (9)	0.4677 (6)	0.030 (2)
C(34)	0.1407 (10)	-0.1622 (10)	0.4977 (6)	0.032 (2)
C(35)	0.2327 (10)	-0.0877 (11)	0.4566 (6)	0.037 (3)
C(36)	0.1955 (9)	-0.0240 (10)	0.3873 (6)	0.030 (2)

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Table 2. Selected geometric parameters (Å, °)

Pb(1)—C(31)	2.201 (9)	C(21)—C(22)	1.363 (13)
Pb(1)—C(21)	2.213 (8)	C(21)—C(26)	1.392 (13)
Pb(1)—C(1)	2.214 (8)	C(22)—C(23)	1.397 (13)
Pb(1)—Br(1)	2.985 (2)	C(23)—C(24)	1.39 (2)
Pb(1)—Br(1a)	2.885 (2)	C(24)—C(25)	1.36 (2)
C(1)—C(11)	1.484 (12)	C(25)—C(26)	1.387 (14)
C(11)—C(16)	1.396 (14)	C(31)—C(36)	1.375 (12)
C(11)—C(12)	1.406 (14)	C(31)—C(32)	1.388 (12)
C(12)—C(13)	1.370 (14)	C(32)—C(33)	1.383 (13)
C(13)—C(14)	1.370 (15)	C(33)—C(34)	1.361 (14)
C(14)—C(15)	1.38 (2)	C(34)—C(35)	1.383 (14)
C(15)—C(16)	1.385 (13)	C(35)—C(36)	1.372 (14)
C(31)—Pb(1)—C(21)	115.4 (3)	Br(1)—Pb(1)—Br(1a ⁱ)	173.610 (15)
C(31)—Pb(1)—C(1)	127.8 (3)	Pb(1)—Br(1)—Pb(1b ⁱⁱ)	122.73 (5)
C(21)—Pb(1)—C(1)	116.7 (3)	C(11)—C(1)—Pb(1)	113.9 (6)
C(31)—Pb(1)—Br(1a ⁱ)	87.3 (2)	C(16)—C(11)—C(1)	120.1 (9)
C(21)—Pb(1)—Br(1a ⁱ)	93.6 (3)	C(12)—C(11)—C(1)	122.1 (9)
C(1)—Pb(1)—Br(1a ⁱ)	93.4 (3)	C(22)—C(21)—Pb(1)	121.6 (7)
C(31)—Pb(1)—Br(1)	88.6 (2)	C(26)—C(21)—Pb(1)	119.5 (7)
C(21)—Pb(1)—Br(1)	92.6 (3)	C(36)—C(31)—Pb(1)	116.9 (6)
C(1)—Pb(1)—Br(1)	85.3 (3)	C(32)—C(31)—Pb(1)	122.1 (6)

Symmetry codes: (i) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$.

Systematic absences ($h0l$) $h + l = 2n + 1$, ($00l$) $l = 2n + 1$ and ($0k0$) $k = 2n + 1$ were detected. The structure was solved by standard Patterson and difference Fourier methods (*SHELXTL-Plus*; Sheldrick, 1987) and refined satisfactorily within space group $P2_1/c$ (No. 14) by full-matrix least-squares calculations (*SHELXL*; Sheldrick, 1994). The H atoms were placed in geometrically calculated positions and refined with common isotropic displacement parameters for different C—H types (H_{aryl}, H_{alkyl}). Computer programs used were *SHELXTL-Plus* (Sheldrick, 1987), *SHELXL*, *PARST* (Nardelli, 1983), *PLATON* (Spek, 1990) and *MISSYM* (Le Page, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: SH1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Hydrate of the Sodium Salt of a Penem Carboxylic Acid

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Abstract

The crystal structure of sodium (5*R*,6*S*)-6-[(*R*)-1-hydroxyethyl]-7-oxo-3-[(*R*)-2-tetrahydrofuryl]-4-thia-1-azabicyclo[3.2.0]hept-2-ene-2-carboxylate 2.5-hydrate, Na⁺.C₁₂H₁₄NO₅S⁻. $\frac{5}{2}$ H₂O, a member of a new class of β -lactam antibiotics, has been determined by X-ray diffraction. The C(3)—S(1) bond (bridgehead C atom) is longer than the C(2)—S(1) bond, although the latter is usually longer in penicillins and cephalosporins. The crystal packing shows a three-layer sheet in which two layers of the molecules are held together by Na⁺ ions and by water molecules through a network of hydrogen bonds.

Comment

The title compound (I), which has the tetrahydrofuran ring at the C(2) position, shows potent *in vivo* activity by oral administration (Nishino *et al.*, 1991). Knowledge of the accurate crystal structure of this compound should be useful for understanding the interaction of the molecule with water and biological macromolecules.

