

C(19)	-0.1993 (7)	0.4044 (5)	0.0021 (6)	0.040 (4)
C(20)	-0.3203 (9)	0.4767 (9)	-0.0359 (6)	0.049 (5)
C(21)	-0.3879 (5)	0.5998 (8)	-0.0315 (6)	0.046 (2)
C(22)	-0.3338 (8)	0.6541 (4)	0.0121 (6)	0.039 (4)
C(23)	-0.2158 (7)	0.5784 (7)	0.0509 (5)	0.031 (3)
C(24)	-0.1422 (7)	0.6056 (7)	0.1104 (5)	0.031 (3)
C(25)	-0.1184 (8)	0.7367 (7)	0.1888 (5)	0.034 (3)
C(26)	-0.1183 (8)	0.8631 (7)	0.1575 (5)	0.035 (4)
C(27)	-0.2157 (5)	0.9770 (8)	0.1216 (6)	0.041 (4)
C(28)	-0.1856 (7)	1.0785 (6)	0.1011 (6)	0.050 (4)
C(29)	-0.0672 (11)	1.0686 (7)	0.1212 (8)	0.065 (5)
C(30)	0.0330 (6)	0.9515 (8)	0.1556 (7)	0.052 (4)
C(31)	0.0016 (8)	0.8520 (7)	0.1724 (6)	0.038 (4)
C(32)	0.0801 (8)	0.7222 (7)	0.2067 (5)	0.036 (3)
C(33)	0.2491 (8)	0.1855 (7)	0.3921 (5)	0.034 (3)
C(34)	0.2597 (8)	0.1151 (7)	0.4819 (5)	0.033 (3)
C(35)	0.3509 (6)	0.0004 (8)	0.5251 (5)	0.046 (4)
C(36)	0.3311 (8)	-0.0437 (6)	0.6103 (6)	0.051 (4)
C(37)	0.2228 (10)	0.0204 (9)	0.6516 (3)	0.052 (5)
C(38)	0.1281 (6)	0.1352 (8)	0.6073 (5)	0.044 (4)
C(39)	0.1530 (8)	0.1791 (8)	0.5223 (5)	0.035 (4)
C(40)	0.0811 (8)	0.2932 (7)	0.4587 (6)	0.037 (4)
C(41)	-0.1024 (8)	0.4740 (8)	0.4273 (5)	0.035 (3)
C(42)	-0.2295 (8)	0.5470 (8)	0.4548 (6)	0.043 (4)
C(43)	-0.2952 (9)	0.5150 (7)	0.5264 (5)	0.056 (5)
C(44)	-0.4191 (10)	0.6022 (10)	0.5309 (6)	0.063 (5)
C(45)	-0.4716 (6)	0.7099 (9)	0.4675 (8)	0.064 (5)
C(46)	-0.4020 (9)	0.7424 (6)	0.3972 (5)	0.049 (4)
C(47)	-0.2792 (8)	0.6535 (7)	0.3928 (6)	0.037 (4)
C(48)	-0.1862 (7)	0.6501 (7)	0.3257 (5)	0.031 (3)

Table 2. Selected geometric parameters (Å, °)

Gd(1)—N(1)	2.241 (7)	N(9)—C(33)	1.266 (12)
Gd(1)—N(5)	2.263 (7)	N(2)—C(9)	1.461 (13)
Gd(1)—N(10)	2.274 (6)	N(6)—C(25)	1.447 (12)
Gd(1)—N(3)	2.235 (7)	N(9)—C(9)	1.442 (10)
Gd(1)—N(7)	2.231 (6)	N(3)—C(9)	1.493 (9)
Gd(1)—N(12)	2.282 (6)	N(7)—C(25)	1.478 (9)
N(2)—C(8)	1.289 (12)	N(13)—C(25)	1.464 (10)
N(7)—C(32)	1.296 (11)	C(9)—C(10)	1.505 (13)
N(13)—C(48)	1.281 (12)	C(25)—C(26)	1.521 (13)
N(6)—C(24)	1.252 (11)		
N(1)—Gd(1)—N(3)	79.3 (3)	N(1)—Gd(1)—N(5)	119.2 (2)
N(3)—Gd(1)—N(5)	78.0 (2)	N(1)—Gd(1)—N(7)	79.9 (2)
N(3)—Gd(1)—N(7)	135.9 (2)	N(5)—Gd(1)—N(7)	78.9 (3)
N(1)—Gd(1)—N(10)	96.7 (3)	N(3)—Gd(1)—N(10)	77.0 (2)
N(5)—Gd(1)—N(10)	130.9 (3)	N(7)—Gd(1)—N(10)	144.0 (3)
N(1)—Gd(1)—N(12)	130.2 (3)	N(3)—Gd(1)—N(12)	144.4 (3)
N(5)—Gd(1)—N(12)	98.0 (2)	N(7)—Gd(1)—N(12)	75.9 (2)
N(10)—Gd(1)—N(12)	79.7 (2)		

The structure was solved by the Patterson method. Refinement was by block-diagonal least-squares techniques. Data collection, cell refinement and data reduction: Kuma KM-4 diffractometer software. Program(s) used to solve and refine structure: *SHELXTL/PC* (Sheldrick, 1990). Molecular graphics: *SHELXTL/PC*.

We would like to thank Professor Z. Galdecki, Technical University of Łódź, Poland, for the opportunity to make the calculations using the *SHELXTL* program system in his laboratory. This work was supported by a grant (No. 2 P303 117 06) from the Polish State Committee for Scientific Research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Bromo(diphenylcyclopropenone)triphenyllead

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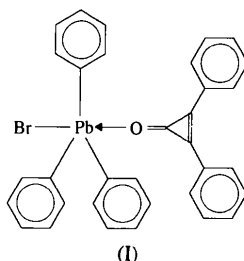
## Abstract

The Pb atom in  $\{[\text{Br}(\text{C}_6\text{H}_5)_3\text{Pb}]\{(\text{C}_6\text{H}_5)_2\text{C}_2\text{CO}\}\}$  is five-coordinate, displaying trigonal-bipyramidal coordination with the three phenyl groups equatorial.

## Comment

The structure of the title complex, (I), was determined following a similar study on chloro(diphenylcyclopropenone)triphenyltin (Ng & Kumar Das, 1993), as the literature on organolead chemistry (Harrison, 1982, 1985) contains few examples of organolead adducts.

The Pb and Sn compounds are isomorphous. The Pb—Br bond [2.696(1) Å] in this complex has been shortened relative to the terminal [2.852(1) Å] and bridging [3.106(1) Å] Pb—Br bonds in the parent



Lewis acid, which adopts a helical chain structure (Preut & Huber, 1977). The Pb—O bond distance [2.659 (6) Å] exceeds that [2.56 (1) Å] found in bromotriphenyl(triphenylphosphine oxide)lead (Eppley, Ealy, Yoder, Spencer & Rheingold, 1992), implying that the cyclic ketone is a much weaker Lewis base than triphenylphosphine oxide.

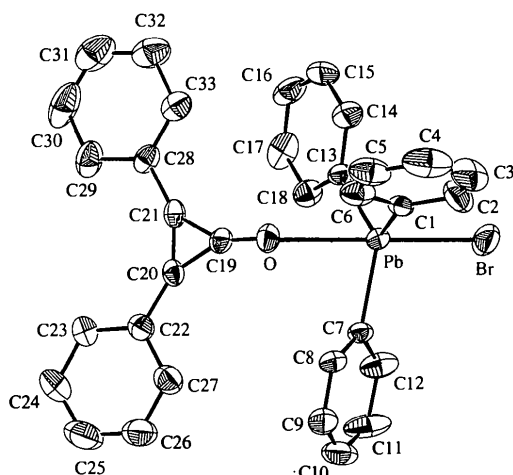


Fig. 1. Atomic numbering scheme for the title compound. Displacement ellipsoids are drawn at the 30% probability level.

## Experimental

Equimolar amounts of triphenyllead bromide and diphenylcyclopropanone were dissolved in a small volume of hot ethanol; slow cooling of the filtered solution gave crystals of the complex.

### Crystal data

[PbBr(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(C<sub>15</sub>H<sub>10</sub>O)]

$M_r = 724.63$

Triclinic

$P\bar{1}$

$a = 10.2314$  (9) Å

$b = 12.1988$  (13) Å

$c = 12.4854$  (6) Å

$\alpha = 89.937$  (6)°

$\beta = 102.916$  (6)°

$\gamma = 111.827$  (7)°

$V = 1404.2$  (2) Å<sup>3</sup>

$Z = 2$

$D_x = 1.714$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 25

reflections

$\theta = 8\text{--}10^\circ$

$\mu = 7.453$  mm<sup>-1</sup>

$T = 298$  K

Block

$0.29 \times 0.29 \times 0.14$  mm

Colourless

### Data collection

Enraf–Nonius CAD-4

diffractometer

$\omega/2\theta$  scans

Absorption correction:

$\psi$  scan (North, Phillips

& Mathews, 1968)

$T_{\min} = 0.549$ ,  $T_{\max} =$

0.999

6786 measured reflections

6424 independent reflections

3685 observed reflections

[ $I > 2\sigma(I)$ ]

$R_{\text{int}} = 0.032$

$\theta_{\text{max}} = 27.46^\circ$

$h = 0 \rightarrow 13$

$k = -15 \rightarrow 14$

$l = -16 \rightarrow 15$

3 standard reflections

monitored every 3

reflections

frequency: 60 min

intensity decay: none

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.107$

$S = 1.025$

6424 reflections

325 parameters

H-atom parameters not

refined

$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.55$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -1.04$  e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Pb1	0.10893 (4)	0.28487 (3)	0.25320 (3)	0.05510 (13)
Br1	-0.12121 (13)	0.19183 (12)	0.34270 (9)	0.1141 (5)
O1	0.3196 (6)	0.3860 (6)	0.1504 (5)	0.069 (2)
C1	0.2229 (11)	0.1655 (7)	0.3121 (7)	0.062 (2)
C2	0.1753 (14)	0.0808 (10)	0.3827 (9)	0.105 (4)
C3	0.2513 (20)	0.0091 (12)	0.4155 (11)	0.121 (6)
C4	0.3683 (20)	0.0180 (14)	0.3816 (13)	0.133 (7)
C5	0.4187 (14)	0.1013 (11)	0.3116 (11)	0.104 (4)
C6	0.3426 (13)	0.1741 (9)	0.2767 (8)	0.085 (3)
C7	0.2007 (9)	0.4617 (7)	0.3407 (6)	0.053 (2)
C8	0.3468 (10)	0.5183 (8)	0.3786 (7)	0.066 (3)
C9	0.4067 (12)	0.6278 (10)	0.4362 (8)	0.087 (3)
C10	0.3191 (17)	0.6836 (10)	0.4554 (9)	0.102 (4)
C11	0.1738 (17)	0.6292 (11)	0.4194 (9)	0.116 (5)
C12	0.1102 (12)	0.5161 (10)	0.3625 (8)	0.089 (3)
C13	-0.0174 (8)	0.2480 (8)	0.0810 (6)	0.053 (2)
C14	-0.0567 (11)	0.1393 (9)	0.0279 (8)	0.082 (3)
C15	-0.1339 (12)	0.1160 (10)	-0.0820 (9)	0.093 (4)
C16	-0.1666 (11)	0.2014 (13)	-0.1379 (9)	0.091 (4)
C17	-0.1273 (10)	0.3098 (11)	-0.0836 (8)	0.084 (3)
C18	-0.0506 (10)	0.3346 (9)	0.0266 (8)	0.070 (3)
C19	0.3311 (9)	0.4531 (8)	0.0761 (8)	0.057 (2)
C20	0.3811 (9)	0.5666 (8)	0.0420 (7)	0.056 (2)
C21	0.3097 (8)	0.4717 (8)	-0.0356 (7)	0.053 (2)
C22	0.4599 (9)	0.6933 (8)	0.0659 (8)	0.064 (2)
C23	0.4925 (12)	0.7690 (10)	-0.0135 (9)	0.094 (3)
C24	0.5735 (16)	0.8895 (11)	0.0152 (13)	0.128 (5)
C25	0.6188 (15)	0.9318 (12)	0.1241 (15)	0.123 (5)
C26	0.5840 (12)	0.8591 (11)	0.2035 (11)	0.097 (4)
C27	0.5072 (10)	0.7407 (9)	0.1760 (9)	0.079 (3)
C28	0.2600 (9)	0.4221 (9)	-0.1500 (7)	0.060 (2)
C29	0.2903 (11)	0.4931 (11)	-0.2348 (8)	0.090 (3)
C30	0.2448 (15)	0.4392 (17)	-0.3411 (10)	0.131 (6)
C31	0.1701 (15)	0.3203 (17)	-0.3634 (12)	0.123 (5)
C32	0.1399 (13)	0.2503 (13)	-0.2807 (11)	0.109 (4)
C33	0.1863 (11)	0.3031 (10)	-0.1735 (8)	0.079 (3)

Table 2. Selected geometric parameters (Å, °)

Pb1—C7	2.183 (8)	C1—C6	1.361 (13)
Pb1—C13	2.196 (8)	C19—C20	1.388 (12)
Pb1—C1	2.213 (10)	C19—C21	1.395 (12)
Pb1—O1	2.659 (6)	C20—C21	1.368 (11)
Pb1—Br1	2.696 (1)	C20—C22	1.448 (12)
O1—C19	1.232 (10)	C21—C28	1.454 (11)
C7—Pb1—C13	122.5 (3)	C14—C13—Pb1	119.2 (7)
C7—Pb1—C1	115.9 (3)	C18—C13—Pb1	120.4 (6)
C13—Pb1—C1	116.4 (3)	O1—C19—C20	150.3 (9)
C7—Pb1—O1	81.4 (3)	O1—C19—C21	150.7 (9)
C13—Pb1—O1	79.8 (2)	C20—C19—C21	58.9 (6)
C1—Pb1—O1	86.2 (3)	C21—C20—C19	60.8 (6)
C7—Pb1—Br1	97.9 (2)	C21—C20—C22	148.0 (9)
C13—Pb1—Br1	95.7 (2)	C19—C20—C22	151.0 (9)
C1—Pb1—Br1	99.3 (3)	C20—C21—C19	60.3 (6)
O1—Pb1—Br1	174.10 (14)	C20—C21—C28	150.7 (9)
C19—O1—Pb1	132.7 (5)	C19—C21—C28	148.7 (9)
C6—C1—C2	118.5 (11)	C23—C22—C27	118.2 (9)
C6—C1—Pb1	119.5 (7)	C23—C22—C20	123.3 (9)
C2—C1—Pb1	122.1 (9)	C27—C22—C20	118.5 (9)
C8—C7—C12	119.0 (8)	C33—C28—C29	119.5 (9)
C8—C7—Pb1	120.5 (7)	C33—C28—C21	119.3 (9)
C12—C7—Pb1	120.4 (7)	C29—C28—C21	121.2 (9)
C14—C13—C18	120.4 (8)		

H atoms were generated and allowed to ride on their parent C atoms, with  $U(H) = 1.5U_{eq}(C)$ .

Data collection: CAD-4 VAX/PC (Enraf-Nonius, 1988). Cell refinement: CAD-4 VAX/PC. Data reduction: Xtal3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

The author thanks the University of Malaya (PJP 280/94) for supporting this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Bis(tetraphenylphosphonium) Tetraazido-dimethylstannate(IV)

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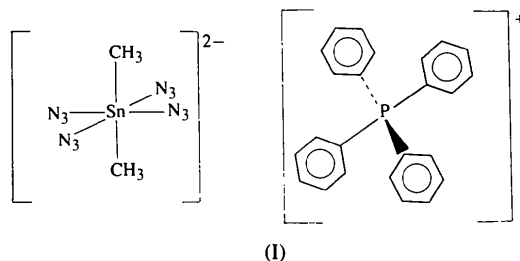
(Received 20 December 1994; accepted 18 April 1995)

### Abstract

The structure of  $[(C_6H_5)_4P]_2[Sn(N_3)_4(CH_3)_2]$  is ionic. In the anion the Sn atom lies at a centre of symmetry and has hexacoordination in which the angles differ slightly but significantly from those of exact octahedral symmetry. The difference between the two unique Sn—N distances is marginal but significant. The azide groups are linear and inclined at approximately 120° to the Sn—N direction. The cation is tetrahedral around the P atom but the orientation of the phenyl groups is unsymmetrical.

### Comment

This investigation of the title compound, (I), is part of a study of the octahedral complexes of Sn<sup>IV</sup> (e.g. Dillon, Halfpenny & Marshall, 1983, 1985). It also affords an opportunity to examine the configuration and symmetry of the azide group when coordinated in an organometallic complex. The structure is clearly ionic, as expected,



and the two ions are shown separately in the SNOOPI (Davies, 1983) diagrams of Fig. 1. The two methyl groups are *trans* – a necessary consequence of the Sn atom lying on a centre of symmetry. The two Sn—N