

$S = 0.920$
 1974 reflections
 93 parameters
 H atoms in riding mode
 $w = 1/[\sigma^2(F_o^2) + (0.0613P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
 Atomic scattering factors
 from *International Tables for Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Acta Cryst. (1995), **C51**, 2039–2042

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

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

	x	y	z	U_{eq}
I	−0.12456 (2)	−0.24984 (5)	0.32861 (3)	0.0598 (2)
Sn	0.09678 (2)	0.14396 (6)	0.14059 (2)	0.0528 (2)
N	0.1698 (3)	0.2392 (5)	0.3565 (3)	0.0419 (9)
C1	−0.0515 (4)	0.2442 (11)	0.1537 (5)	0.078 (2)
C2	0.1598 (5)	0.2709 (11)	0.0221 (4)	0.081 (2)
C3	0.1012 (4)	−0.1689 (10)	0.1363 (4)	0.074 (2)
C4	0.2004 (3)	0.2311 (8)	0.2575 (3)	0.0491 (12)
C5	0.2593 (4)	0.2559 (8)	0.4226 (4)	0.0551 (13)
C6	0.1151 (4)	0.0566 (9)	0.3792 (4)	0.0583 (13)
C7	0.1051 (4)	0.4136 (9)	0.3695 (4)	0.067 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn—C2	2.126 (6)	N—C7	1.499 (6)
Sn—C3	2.142 (7)	N—C6	1.499 (6)
Sn—C1	2.143 (5)	N—C4	1.495 (6)
Sn—C4	2.182 (5)	N—C5	1.485 (6)
C2—Sn—C3	111.9 (3)	C7—N—C4	110.4 (4)
C2—Sn—C1	111.7 (3)	C6—N—C4	110.2 (4)
C3—Sn—C1	110.5 (3)	C7—N—C5	108.6 (4)
C2—Sn—C4	102.7 (2)	C6—N—C5	108.7 (4)
C3—Sn—C4	106.1 (2)	C4—N—C5	109.3 (4)
C1—Sn—C4	113.7 (2)	N—C4—Sn	121.4 (3)
C7—N—C6	109.5 (4)		

All non-H atoms were given anisotropic temperature factors; the methylene and methyl H atoms were given isotropic temperature factors and were allowed to ride on the C atoms to which they are attached.

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SNOOPI* (Davies, 1983).

The use of the SERC X-ray crystallographic service at Cardiff University is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1164). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Gadolinium(III) Bicyclic Phthalocyanine

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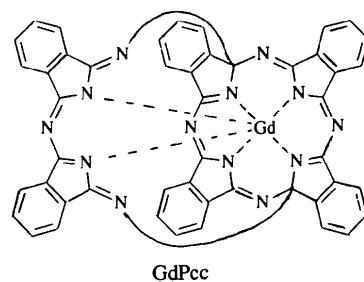
(Received 13 January 1995; accepted 18 April 1995)

Abstract

A new gadolinium bicyclic phthalocyanine complex, $\{14,28\text{-}[3,3'\text{-iminobis}(1\text{-isoindolylideneamino)}]\text{phthalocyaninato(3-)}\}$ gadolinium(III), $[\text{Gd}(\text{C}_{48}\text{H}_{24}\text{N}_{13})]$, has been obtained from gadolinium and 1,2-dicyanobenzene under a low pressure of nitrogen at 483 K. The crystals consist of separate molecules of the title compound. The bicyclic hexadentate phthalocyaninato ligand is composed of six isoindole units and forms a distorted trigonal prism by coordination *via* its six N-donor atoms, one from each isoindole ring. Gd—N distances range from 2.231 (6) to 2.282 (6) \AA . The conjugation of the inner 16-membered ring is interrupted by the two sp^3 -hybridized C atoms.

Comment

The crystal structure of gadolinium(III) bicyclic phthalocyanine, GdPcc, consists of discrete molecules of six-coordinate Gd^{III} surrounded by the six isoindole N atoms of the bicyclic phthalocyaninato chelate ligand.



The bicyclic phthalocyaninato ligand consists of the phthalocyaninato skeleton of four isoindole units which is bridged by two additional isoindole moieties. The six N-donor atoms of the whole bicyclic phthalocyaninato ligand form a distorted trigonal prism. The macrocyclic hexadentate chelate ligand can also be described as two phthalocyaninato anions having one common half. It can be assumed that the macrocyclic ligand is less stable than the phthalocyaninato anion as the conjugated π -electron system of the inner 16-membered ring is interrupted at the two sp^3 -hybridized bridgehead atoms, C(9) and C(25), of the ring linkage to the bicyclo system.

This interruption undoubtedly accounts for the specific green colour of metallocyclic phthalocyanines. This is also the reason why the two connected phthalocyaninato moieties are not planar.

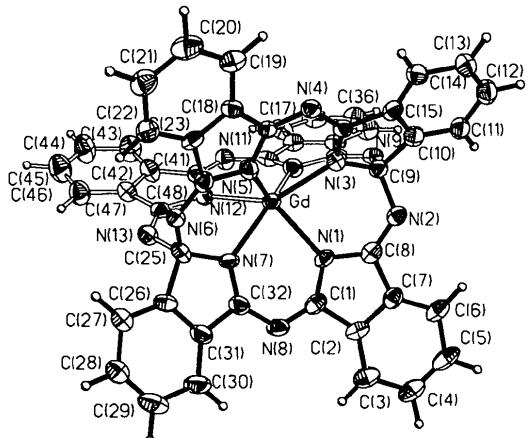


Fig. 1. View of molecular structure of GdPcc and atom numbering (50% probability displacement ellipsoids for non-H atoms).

The whole macrocyclic chelate ligand is an anion with three negative charges. The same bicyclic hexadentate ligand can be found in an indium complex (Janczak & Kubiak, 1994a). A similar hexadentate ligand can be seen in [Nb(Cl)(C₄₈H₂₄N₁₃)] (Gingl & Stähle, 1990). Although both (gadolinium and niobium) chelate compounds contain ligands of the same overall formula (C₄₈H₂₄N₁₃), the ligands have different structures and point symmetries, *i.e.* 1 and 2 for GdPcc and [Nb(Cl)(C₄₈H₂₄N₁₃)], respectively. The main difference between these macrocyclic ligands is in the bridgehead atoms. In GdPcc, the bridgehead C atoms belong to the two opposite isoindole rings of the phthalocyaninato units, whereas in [Nb(Cl)(C₄₈H₂₄N₁₃)] the bridgehead C atoms belong to two neighbouring isoindole units.

The C—N distances in the bicyclic phthalocyaninato ligand reflect the interruption of the conjugation. The C—N bond lengths at the bridgehead atoms C(9) and C(25) are in the ranges 1.44 (1)–1.49 (1) Å and 1.45 (1)–1.48 (1) Å, respectively, and are comparable to the equivalent distances in InPcc and [Nb(Cl)(C₄₈H₂₄N₁₃)]; they are also close to the expected value for a C—N single bond between C_{sp³} and N_{sp²} atoms. The bonds C(9)—C(10) [1.505 (13) Å] and C(25)—C(26) [1.521 (13) Å] show a bond order of 1. This value is typical for a normal C—C σ bond of 1.530 (15) Å (Allen *et al.*, 1987). Each of the above C—N bonds joins to the next C atom in the ring system to give shorter N—C bonds, ranging from 1.252 (11) to 1.296 (11) Å, which represent double bonds. The other C—N distances in the macrocyclic ring have values between 1.32 (1) and 1.44 (1) Å, typical for a conjugated π -electron system.

The dihedral angles between the two neighbouring mean least-squares planes of the phthalocyaninato moieties are 105.4 (4), 120.6 (5) and 133.8 (5)°; these values are similar to the corresponding angles in InPcc [105.9 (2), 120.0 (3) and 134.1 (3)°] and are different from those in [Nb(Cl)(C₄₈H₂₄N₁₃)] (114.0, 121.8 and 124.2°; Gingl & Stähle, 1990). The dihedral angles between the phenyl ring planes belonging to the same phthalocyaninato moieties are 18.2 (2), 15.9 (3) and 23.2 (3)° between rings C(10)–C(15) and C(18)–C(23), C(34)–C(39) and C(42)–C(47), and C(2)–C(7) and C(26)–C(31), respectively. None of the six pyrrole rings fused to their respective phenyl ring lies in the same plane as the phenyl ring. The values of the dihedral angles between the pyrrole and fused phenyl ring planes are 0.9 (3)–7.4 (3)°.

The characteristic feature of the GdPcc crystal structure is the propeller-like shape of the GdPcc molecules and their arrangement such that the wings from adjacent molecules interpenetrate like cog wheels in mechanical machinery (Fig. 2). The shortest intermolecular distance between two neighbouring phthalocyaninato planes is 3.356 (10) Å. This value is comparable with the van der Waals distance of 3.4 Å for aromatic C atoms (Pauling, 1962) and indicates weak interaction between these atomic planes. This interaction is also observed in the sandwich-type metallodiphthalocyanines (Bennet, Broberg & Baezinger, 1973; Gieren & Hoppe, 1971; Kirin, Kolabin & Lyczew, 1974; Moussavi, DeCian, Fischer & Weiss, 1988; Janczak & Kubiak, 1994b). The shortest intermolecular H···N contacts are N(4)···H(29ⁱ) 2.62 (1) and N(13)···H(4ⁱⁱ) 2.79 (1) Å [symmetry codes: (i) $x, y - 1, z$; (ii) $x - 1, y, z$].

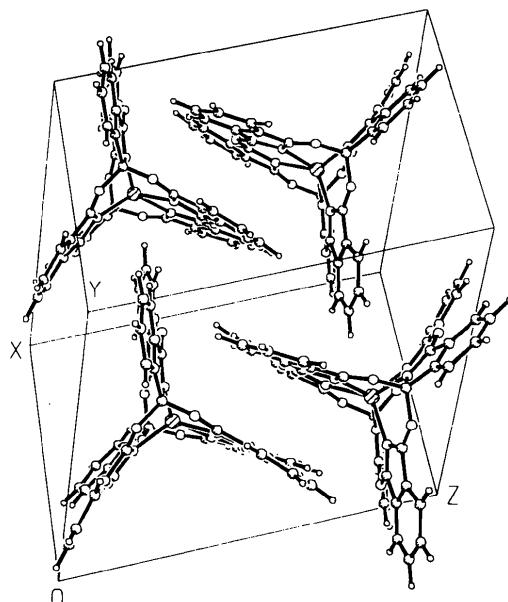


Fig. 2. Molecular arrangement in the unit cell.

The X-ray analysis of this green gadolinium complex and the fact that the green InPc (Janczak & Kubiak, 1994a) and violet In_2Pc_3 (Janczak & Kubiak, 1993) compounds possess very narrow compositions, and coexist in the reaction products of In with 1,2-dicyanobenzene, enabled us to critically revise the published data regarding the green form of lanthanide phthalocyanines (Kirin, Moskalev & Ivannikova, 1967; Kirin, Moskalev & Makashev, 1967; Misumi & Kasuga, 1971; MacKay, Boas & Troup, 1974). Our results differ from those of Kirin and co-workers (Kirin, Moskalev & Ivannikova, 1967; Kirin, Moskalev & Makashev, 1967), and Misumi & Kasuga (1971), who concluded that their green complex was a monophthalocyanine, because its optical spectrum resembled that of normal monophthalocyanine. The elemental analyses obtained by MacKay, Boas & Troup (1974) for the green and blue forms are inconsistent with the theoretical calculations for gadolinium diphthalocyanine. From the gadolinium content (14.7–18.5%) obtained by MacKay, Boas & Troup (1974) for gadolinium phthalocyanine, we suggest that they also observed green bicyclic phthalocyanine (GdPc) but not green diphthalocyanine. Our analysis (16.89% Gd) is consistent with theirs and the theoretical calculation for $\text{GdC}_{48}\text{H}_{24}\text{N}_{13}$.

Moreover, on the basis of our experimental data and our studies (Janczak & Kubiak, 1993, 1994a), we suggest that blue–violet is the previous characteristic colour for all phthalocyanines in which full π -electron delocalization in the inner 16-membered ring exists and that green is the characteristic colour for cyclic phthalocyanines in which the inner π -electron delocalization is interrupted.

Experimental

Gadolinium bicyclic phthalocyanine was obtained in the reaction of gadolinium (pure 99.999%) with 1,2-dicyanobenzene under a low nitrogen pressure (about 10^{-1} atm). The filings of gadolinium were mixed with 1,2-dicyanobenzene in a weight proportion of 1:4. Next the mixture was pressed into pellets. The pellets were inserted into a glass ampoule under nitrogen pressure and sealed off. The ampoule was heated to 483 K. The heating process continued for 3 d and lead to single green good quality crystals of the title compound. Analysis on an energy dispersive spectrometer (EDAX): found Gd 16.89, C 61.30, N 19.31%; calculated for $\text{C}_{48}\text{H}_{24}\text{N}_{13}\text{Gd}$ Gd 16.73, C 61.33, N 19.37 and H 2.56%. The density D_m was measured by flotation in $\text{C}_2\text{H}_4\text{Br}_2$ – $\text{C}_2\text{H}_4\text{Cl}_2$.

Crystal data

$[\text{Gd}(\text{C}_{48}\text{H}_{24}\text{N}_{13})]$

$M_r = 940.1$

Triclinic

$P\bar{1}$

$a = 11.683 (2)$ Å

$b = 12.388 (3)$ Å

$c = 15.978 (3)$ Å

$\alpha = 75.62 (3)^\circ$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 20

reflections

$\theta = 12\text{--}18^\circ$

$\mu = 1.691$ mm $^{-1}$

$T = 295$ K

Parallelepiped

$\beta = 86.00 (3)^\circ$

$\gamma = 65.12 (3)^\circ$

$V = 2030.5 (6)$ Å 3

$Z = 2$

$D_x = 1.537$ Mg m $^{-3}$

$D_m = 1.53$ Mg m $^{-3}$

$0.32 \times 0.28 \times 0.26$ mm

Green

Data collection

Kuma KM-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

none

10989 measured reflections

6410 independent reflections

4736 observed reflections

$[F > 4\sigma(F)]$

$R_{\text{int}} = 0.0289$

$\theta_{\text{max}} = 25^\circ$

$h = -12 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -16 \rightarrow 16$

2 standard reflections monitored every 50 reflections

intensity decay: <1%

Refinement

Refinement on F

$R = 0.047$

$wR = 0.068$

$S = 1.23$

4736 reflections

656 parameters

Only coordinates of H atoms refined

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\text{max}} = 0.005$

$\Delta\rho_{\text{max}} = 2.51$ e Å $^{-3}$

$\Delta\rho_{\text{min}} = -0.59$ e Å $^{-3}$

Extinction correction:

$F_c = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$

Extinction coefficient: $\chi = 0.0002 (1)$

Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å 2)

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

	x	y	z	U_{eq}
Gd(1)	0.08149 (4)	0.45457 (4)	0.26255 (3)	0.0267 (2)
N(1)	0.2678 (6)	0.4675 (6)	0.2470 (4)	0.031 (3)
N(2)	0.4147 (6)	0.2596 (6)	0.2417 (4)	0.033 (3)
N(3)	0.1943 (6)	0.2909 (6)	0.2100 (4)	0.031 (3)
N(4)	0.0524 (6)	0.2963 (6)	0.1059 (4)	0.032 (3)
N(5)	-0.0358 (6)	0.4921 (6)	0.1432 (4)	0.033 (3)
N(6)	-0.1753 (6)	0.7100 (6)	0.1235 (4)	0.032 (3)
N(7)	0.0156 (6)	0.6572 (6)	0.2150 (4)	0.030 (3)
N(8)	0.2058 (7)	0.6862 (6)	0.2257 (5)	0.039 (3)
N(9)	0.3285 (6)	0.1439 (6)	0.3372 (4)	0.032 (3)
N(10)	0.1390 (7)	0.2972 (6)	0.3830 (4)	0.036 (3)
N(11)	-0.0316 (7)	0.3691 (6)	0.4803 (5)	0.040 (3)
N(12)	-0.0803 (6)	0.5333 (6)	0.3495 (4)	0.032 (3)
N(13)	-0.2048 (6)	0.7403 (6)	0.2600 (4)	0.034 (3)
C(1)	0.2861 (8)	0.5699 (8)	0.2396 (5)	0.037 (4)
C(2)	0.4214 (8)	0.5416 (8)	0.2399 (6)	0.038 (4)
C(3)	0.4890 (8)	0.6107 (5)	0.2387 (6)	0.049 (5)
C(4)	0.6205 (9)	0.5525 (9)	0.2370 (7)	0.055 (5)
C(5)	0.6816 (4)	0.4297 (10)	0.2387 (7)	0.057 (5)
C(6)	0.6135 (7)	0.3570 (5)	0.2395 (6)	0.047 (4)
C(7)	0.4839 (7)	0.4157 (8)	0.2425 (5)	0.036 (3)
C(8)	0.3854 (8)	0.3710 (8)	0.2451 (5)	0.034 (3)
C(9)	0.3238 (7)	0.2057 (7)	0.2473 (5)	0.031 (3)
C(10)	0.3653 (8)	0.1100 (7)	0.1957 (6)	0.037 (3)
C(11)	0.4779 (6)	0.0088 (8)	0.1983 (5)	0.038 (4)
C(12)	0.4868 (6)	-0.0688 (6)	0.1439 (6)	0.045 (4)
C(13)	0.3878 (8)	-0.0410 (7)	0.0884 (5)	0.046 (4)
C(14)	0.2749 (6)	0.0631 (7)	0.0845 (5)	0.037 (4)
C(15)	0.2666 (7)	0.1356 (7)	0.1398 (5)	0.034 (3)
C(16)	0.1622 (7)	0.2498 (7)	0.1519 (5)	0.029 (3)
C(17)	-0.0357 (7)	0.4085 (6)	0.1040 (5)	0.028 (3)
C(18)	-0.1502 (8)	0.4605 (7)	0.0483 (5)	0.031 (3)

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)triphenyl-lead

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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