

- Viossat, B., Khodadad, P. & Rodier, N. (1991a). *Acta Cryst.* C47, 179–180.
 Viossat, B., Khodadad, P. & Rodier, N. (1991b). *Acta Cryst.* C47, 1714–1716.
 Viossat, B., Khodadad, P., Rodier, N. & Guillard, O. (1990). *Acta Cryst.* C46, 894–896.
 Viossat, B., Michelet, A., Khodadad, P. & Rodier, N. (1990). *Acta Cryst.* C46, 1733–1735.
 Viossat, B., Rodier, N., Nguyen Huy, D. & Guillard, O. (1986). *Acta Cryst.* C42, 659–662.

= 2.194 (6) Å and $\varphi = 7.7 (2)^\circ$. The structures of tetraaryl compounds of Group 14 elements hitherto determined by X-ray diffraction have recently been compared (Charissé, Roller & Dräger, 1992).

Acta Cryst. (1993). C49, 1372–1373

Redetermination of Tetraphenyllead

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(Received 18 December 1992; accepted 17 February 1993)

Abstract

The redetermination with diffractometer data corroborates the results of a prior study [Busetti, Mammi, Signor & del Pra (1967). *Inorg. Chim. Acta*, 1, 424–428] which was performed on the basis of 70 observed ($hk0$) and 38 observed ($h0l$) film reflections recorded with a microdensitometer. The bonding sphere around Pb in $(C_6H_5)_4Pb$ is practically a regular tetrahedron with a maximum deviation of 0.8° from the ideal angle and a mean value of $109.5 (2)^\circ$.

Comment

Following earlier attempts to solve the crystal structure of $(C_6H_5)_4Pb$ (George, 1927; Giacomello, 1938; Zhdanov & Ismailzade, 1949, 1950) a structure description of the tetrahedral molecule was given by Busetti, Mammi, Signor & del Pra (1967). In this paper only the position of a regular hexagonal phenyl ring (C—C 1.397 Å) was varied systematically in order to find the lowest R value for 70 observed ($hk0$) and 38 observed ($h0l$) film reflections recorded with a microdensitometer; the position of the crystallographically independent phenyl ring is described by four parameters: $\alpha = 55.8^\circ$ [half bond angle C(1)—Pb(1)—C(1b)], $\omega = 58.9^\circ$ [angle between the plane through the phenyl ring and the plane through the atoms C(1), Pb(1), C(1b)], $d = 2.19 (3) \text{ \AA}$ (Pb—C bond length) and $\varphi = 7.5^\circ$ [angle between the b axis and the line through the projection of the atoms Pb(1), C(1) and C(4) on plane ab]. The present work corroborates the results of Busetti, Mammi, Signor & del Pra (1967); the corresponding values in our results are: $\alpha = 55.2 (2)^\circ$, $\omega = 58.4 (2)^\circ$, d

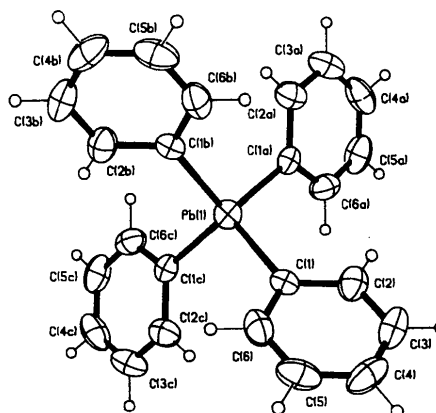


Fig. 1. General view (SHELXTL-Plus graphic) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Experimental

Crystal data

$[Pb(C_6H_5)_4]$
 $M_r = 515.62$
 Tetragonal
 $P4_21c$
 $a = 12.151 (2) \text{ \AA}$
 $c = 6.545 (1) \text{ \AA}$
 $V = 966.4 (3) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.772 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 32 reflections
 $\theta = 6.15\text{--}12.65^\circ$
 $\mu = 8.8 \text{ mm}^{-1}$
 $T = 293 (1) \text{ K}$
 Block
 $0.22 \times 0.18 \times 0.16 \text{ mm}$
 Colourless

Data collection

Nicolet R3m/V diffractometer
 $2\theta/\omega$ scans
 Absorption correction: empirical
 $T_{\min} = 0.73$, $T_{\max} = 1.00$
 3660 measured reflections
 861 independent reflections
 766 observed reflections
 $[F > 3.0\sigma(F)]$

$R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -15 \rightarrow 15$
 $k = 0 \rightarrow 15$
 $l = -8 \rightarrow 8$
 6 standard reflections
 frequency: 150 min
 intensity variation: none

Refinement

Refinement on F
 Final $R = 0.0301$
 $wR = 0.0412$
 $S = 1.32$
 766 reflections
 59 parameters
 Only H-atom U 's refined

$\Delta\rho_{\text{max}} = 0.9 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -2.0 \text{ e \AA}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Calculated weights

$$w = 1/[\sigma^2(F) + 0.000673F^2]$$

$$(\Delta/\sigma)_{\max} = 0.2$$

The absolute structure was confirmed by refinement of $\eta = 0.91$ (9) (Rogers, 1981)

Data collection: Nicolet R3m/V software, Release 4.11. Cell refinement: Nicolet R3m/V software. Data reduction: Nicolet R3m/V software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus*. Software used to prepare material for publication: *PARST* (Nardelli, 1983); *PLATON* (Spek, 1982); *MISSYM* (Le Page, 1987).

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

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

	x	y	z	U_{eq}
Pb(1)	0.0	0.0	0.0	384
C(1)	0.0205 (4)	0.1467 (5)	0.1916 (8)	340
C(2)	-0.0539 (5)	0.2359 (5)	0.173 (1)	463
C(3)	-0.0421 (6)	0.3272 (5)	0.295 (1)	569
C(4)	0.0434 (7)	0.3356 (6)	0.431 (1)	564
C(5)	0.1136 (6)	0.2509 (7)	0.455 (1)	539
C(6)	0.1038 (6)	0.1586 (7)	0.335 (1)	456

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

Pb(1)—C(1)	2.194 (6)	C(3)—C(4)	1.374 (10)
C(1)—C(2)	1.416 (8)	C(4)—C(5)	1.345 (11)
C(1)—C(6)	1.387 (9)	C(5)—C(6)	1.374 (11)
C(2)—C(3)	1.373 (9)		
C(1)—Pb(1)—C(1 ⁱ)	109.1 (2)	C(1)—C(2)—C(3)	120.1 (6)
C(1)—Pb(1)—C(1 ⁱⁱ)	110.3 (2)	C(2)—C(3)—C(4)	121.1 (6)
Pb(1)—C(1)—C(6)	123.6 (5)	C(3)—C(4)—C(5)	119.7 (7)
Pb(1)—C(1)—C(2)	120.0 (4)	C(4)—C(5)—C(6)	120.4 (7)
C(2)—C(1)—C(6)	116.4 (6)	C(1)—C(6)—C(5)	122.2 (7)

Symmetry code: (i) $-y, x, -z$; (ii) $-x, -y, z$.

The title compound was prepared by a procedure described by Pfeiffer & Truskier (1904) and modified by Horn (1966). Single crystals were obtained from chloroform. The crystal was mounted on the tip of a glass fibre with shellac. The lattice constants were determined from a symmetry-constrained least-squares fit. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Data were collected with $2\theta/\omega$ scans of speed $1.50\text{--}14.65^\circ \text{ min}^{-1}$ in θ and with θ scan width $(1.2 \text{ plus dispersion})^\circ$. Lorentz-polarization and absorption (via ψ scans) corrections were made. H atoms were placed in calculated positions and a common isotropic displacement parameter was refined. The weighting scheme used led to an analysis of variance which was featureless in terms of $\sin\theta$ and F . A search for higher symmetries was performed using *MISSYM* (Le Page, 1987).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71125 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1039]

References

Busetti, V., Mammi, M., Signor, A. & del Pra, A. (1967). *Inorg. Chim. Acta*, **1**, 424–428.

- Charissé, M., Roller, S. & Dräger, M. (1992). *J. Organomet. Chem.* **427**, 23–31.
- George, W. H. (1927). *Proc. R. Soc. London Ser. A*, **113**, 585–593.
- Giacomello, G. (1938). *Gazz. Chim. Ital.* **68**, 422–428.
- Horn, H. (1966). Dissertation, TH Aachen, Germany.
- Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Pfeiffer, P. & Truskier, P. (1904). *Ber. Dtsch. Chem. Ges.* **37**, 1125–1127.
- Rogers, D. (1981). *Acta Cryst.* **A37**, 734–741.
- Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
- Spek, A. L. (1982). *The EUCLID Package. Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.
- Zhdanov, G. S. & Ismailzade, I. G. (1949). *Dokl. Akad. Nauk SSSR*, **68**, 96–98.
- Zhdanov, G. S. & Ismailzade, I. G. (1950). *Zh. Fiz. Khim.* **24**, 1495–1501.

Acta Cryst. (1993). **C49**, 1373–1376

Structure of a Keto Derivative of 9,11-Dihydrogracilin A

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(Received 18 September 1992; accepted 26 January 1993)

Abstract

The X-ray analysis was carried out on the 8-keto derivative of 9,11-dihydrogracilin A, 1,3,3a,4,5,6,7,7a-octahydro-4-oxo-5-(1,3,3-trimethylcyclohexyl)-1,3-isobenzofurandiol diacetate, the major metabolite from the Antarctic sponge *Dendrilla membranosa*. The present study confirms the norditerpenoid skeleton of the spongian type proposed for the natural product by Molinski & Faulkner [*J. Org. Chem.* (1987), **52**, 296–298] on the basis of spectral data and chemical correlations, and also fixes the unknown relative stereochemistry at C10. This result gives further support to the hypothesis regarding the biogenetic origin of the gracilins. The six-membered rings are both in chair conformations and the furan ring is in an envelope form. The molecular packing is governed by normal van der Waals interactions.