Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Jurnak, F. A. & Raymond, K. N. (1974). *Inorg. Chem.* **13**, 2387–2397.
Kane-Maguire, N. A. P., Wallace, K. C. & Miller, D. B. (1985). *Inorg. Chem.* **24**, 597–605.
Kirk, A. D. & Guedel, H. (1992). *Inorg. Chem.* **31**, 4564–4569.
Kirk, A. D. & Heyd, D. (1991). *Inorg. Chem.* **30**, 2453–2457.
Lai, T. F. & Poon, C. K. (1976). *Inorg. Chem.* **15**, 1562–1566.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1993). C**49**, 1066–1068

Tetrakis(2-chlorobenzyl)lead

By Uwe Fahrenkampf, Markus Schürmann and Friedo Huber*

Lehrstuhl für Anorganische Chemie II, *Universität Dortmund, Postfach* 50 05 00, *D*-4600 *Dortmund* 50, *Germany*

(*Received* 2 *October* 1992; *accepted* 11 *February* 1993)

Abstract. $[Pb(C_7H_6Cl)_4]$, $M_r = 709.51$, monoclinic, $P2_1/c$, $a = 10.871\ (6)$, $b = 21.962\ (10)$, $c = 11.650\ (6)$ Å, $\beta = 110.11\ (4)°$, $V = 2612\ (2)$ Å^3, $Z = 4$, $D_x = 1.804$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.710730 Å, $\mu = 6.94$ mm^{-1}, $F(000) = 1368$, $T = 169\ (1)$ K, final $R = 0.040$ for 2391 unique observed $[F \geq 5\sigma(F)]$ X-ray diffractometer data. The C atoms of the four benzyl CH_2 groups form a slightly distorted tetrahedron around the central atom of the title compound. The four Pb—C distances [2.25 (1) to 2.26 (2) Å] are not significantly different. Pb—C distance data of symmetric tetraalkyllead or benzyllead compounds are not available for comparison. The C—C distances are in the usual range.

Introduction. Tetrabenzyllead and substituted symmetric tetrabenzyllead compounds differ from common tetraorganolead compounds by being coloured (yellow) and being very air sensitive. One requirement for understanding this unusual behaviour is a detailed knowledge of the molecular structure of such compounds. We report here on the solid-state structure of $(2\text{-}ClC_6H_4CH_2)_4Pb$. This is the first published single-crystal structure determination of a symmetric tetraalkyllead compound.

Experimental. The compound was prepared by reaction of $2\text{-}ClC_6H_4CH_2MgBr$ with $PbCl_2$ under total exclusion of air (Bähr & Zoche, 1955). Single, yellow, clear crystals from ethanol, m.p. 372 K. Crystal size ≃ 0.14 × 0.14 × 0.16 mm, $\omega/2\theta$ scans, variable scan speed 2.5–15° min^{-1} in θ, Nicolet $R3m/V$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit with 26 reflections in the range $2\theta = 15$–30°; six standard reflections (200, $\bar{2}00$, 040, $0\bar{4}0$, 004, $00\bar{4}$) recorded every 2.5 h, showed up to 8.54% intensity loss during data collection; 6781 reflections measured; $1 \leq \theta \leq 25°$; $-12 \leq h \leq 3$, $-1 \leq k \leq 27$, $-14 \leq l \leq 14$. The data were corrected for Lorentz–polarization, but not for absorption effects. After averaging 4620 unique reflections, $R_{\rm int} = 0.0236$, from which 2391 have $[F \geq 5\sigma(F)]$; no ψ scans; systematic absences $(h0l)$ $l = 2n + 1$, $(0k0)$ $k = 2n + 1$ conform with space group $P2_1/c$ (No. 14); structure solution *via* direct

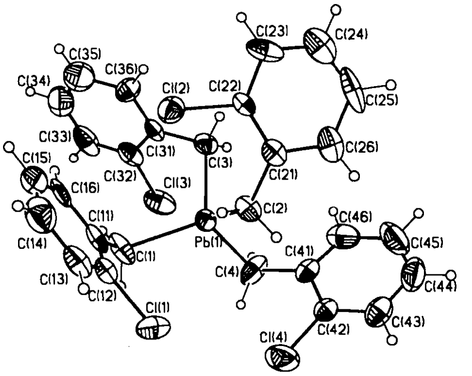

Fig. 1. General view (*SHELXTL-Plus*) of the molecule, showing the atom-numbering scheme.

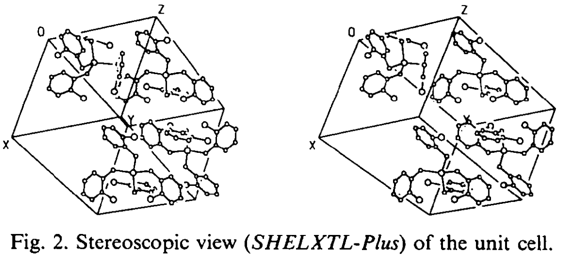

Fig. 2. Stereoscopic view (*SHELXTL-Plus*) of the unit cell.

---

* Author to whom correspondence should be addressed.

0108-2701/93/061066-03$06.00

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pb(1)	0.20200 (5)	0.12801 (2)	0.39567 (4)	296
Cl(1)	0.5110 (4)	0.1843 (2)	0.3528 (4)	733
Cl(2)	0.1663 (4)	0.0256 (2)	0.1276 (3)	437
Cl(3)	−0.0799 (4)	0.2205 (2)	0.3851 (4)	609
Cl(4)	0.5358 (5)	0.1535 (2)	0.6756 (4)	750
C(1)	0.217 (2)	0.2054 (6)	0.273 (1)	505
C(11)	0.271 (1)	0.1860 (5)	0.181 (1)	391
C(12)	0.401 (1)	0.1752 (6)	0.204 (1)	430
C(13)	0.451 (1)	0.1585 (6)	0.113 (1)	453
C(14)	0.368 (2)	0.1498 (7)	0.000 (2)	628
C(15)	0.235 (2)	0.1574 (7)	−0.026 (1)	661
C(16)	0.185 (2)	0.1762 (6)	0.061 (1)	446
C(2)	0.335 (1)	0.0495 (5)	0.391 (1)	356
C(21)	0.260 (1)	−0.0080 (5)	0.366 (1)	314
C(22)	0.179 (1)	−0.0239 (5)	0.251 (1)	330
C(23)	0.100 (1)	−0.0753 (6)	0.222 (1)	492
C(24)	0.109 (2)	−0.1135 (7)	0.315 (2)	571
C(25)	0.187 (2)	−0.1029 (7)	0.430 (2)	649
C(26)	0.266 (1)	−0.0499 (6)	0.458 (1)	469
C(3)	−0.006 (1)	0.0927 (5)	0.329 (1)	364
C(31)	−0.091 (1)	0.1304 (6)	0.226 (1)	325
C(32)	−0.134 (1)	0.1886 (6)	0.239 (1)	432
C(33)	−0.215 (1)	0.2218 (7)	0.145 (2)	584
C(34)	−0.258 (2)	0.1975 (9)	0.029 (2)	680
C(35)	−0.217 (2)	0.1427 (9)	0.008 (2)	683
C(36)	−0.136 (1)	0.1081 (6)	0.109 (1)	392
C(4)	0.240 (2)	0.1543 (7)	0.591 (1)	547
C(41)	0.306 (1)	0.1032 (6)	0.672 (1)	321
C(42)	0.444 (1)	0.0989 (6)	0.716 (1)	347
C(43)	0.508 (2)	0.0519 (7)	0.789 (1)	499
C(44)	0.438 (2)	0.0084 (8)	0.822 (1)	700
C(45)	0.310 (3)	0.0114 (9)	0.782 (2)	932
C(46)	0.241 (2)	0.0583 (8)	0.709 (1)	618

methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor (0.08 Å^2) for H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å); refinement on F with 2391 reflections and 298 refined parameters, $w = 1.0/[\sigma^2(F) + (0.00650F^2)]$, $S = 0.509$, $R = 0.0399$, $wR = 0.0485$, $(\Delta/\sigma)_{\max} < 0.0001$; $(\Delta\rho)_{\max} = 2.19$, $(\Delta\rho)_{\min} = -2.64$ e Å^{-3}. [Data collected at 291 (1) K allowed only anisotropic refinement of Pb and Cl; final $R = 0.0591$.] Atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV); programs used: *SHELXTL-Plus* (Sheldrick, 1987), *PARST* (Nardelli, 1983), *PLATON* (Spek, 1990), *MISSYM* (Le Page, 1987).

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes and dihedral angles* (°)

Pb(1)—C(1)	2.26 (2)	C(21)—C(22)	1.37 (2)
Pb(1)—C(2)	2.26 (1)	C(21)—C(26)	1.40 (2)
Pb(1)—C(3)	2.26 (1)	C(22)—C(23)	1.39 (2)
Pb(1)—C(4)	2.25 (1)	C(23)—C(24)	1.35 (2)
Pb(1)—Cl(1)	3.770 (5)	C(24)—C(25)	1.33 (2)
Pb(1)—Cl(2)	3.759 (4)	C(25)—C(26)	1.42 (2)
Pb(1)—Cl(3)	3.643 (5)	C(3)—C(31)	1.49 (2)
Pb(1)—Cl(4)	3.998 (5)	C(31)—C(32)	1.39 (2)
Cl(1)—C(12)	1.75 (1)	C(31)—C(36)	1.38 (2)
Cl(2)—C(22)	1.77 (1)	C(32)—C(33)	1.36 (2)
Cl(3)—C(32)	1.74 (2)	C(33)—C(34)	1.38 (3)
Cl(4)—C(42)	1.73 (1)	C(34)—C(35)	1.33 (3)
C(1)—C(11)	1.45 (2)	C(35)—C(36)	1.42 (2)
C(11)—C(12)	1.36 (2)	C(4)—C(41)	1.48 (2)
C(11)—C(16)	1.41 (2)	C(41)—C(42)	1.41 (2)
C(12)—C(13)	1.39 (2)	C(41)—C(46)	1.37 (2)
C(13)—C(14)	1.33 (2)	C(42)—C(43)	1.37 (2)
C(14)—C(15)	1.38 (3)	C(43)—C(44)	1.36 (3)
C(15)—C(16)	1.37 (3)	C(44)—C(45)	1.31 (3)
C(2)—C(21)	1.48 (2)	C(45)—C(46)	1.38 (3)

C(3)—Pb(1)—C(4)	104.5 (5)	C(23)—C(24)—C(25)	123 (2)
C(2)—Pb(1)—C(4)	108.9 (5)	C(24)—C(25)—C(26)	120 (2)
C(2)—Pb(1)—C(3)	107.6 (4)	C(21)—C(26)—C(25)	120 (1)
C(1)—Pb(1)—C(4)	114.7 (5)	Pb(1)—C(3)—C(31)	110.8 (9)
C(1)—Pb(1)—C(3)	108.9 (5)	C(3)—C(31)—C(36)	121 (1)
C(1)—Pb(1)—C(2)	111.8 (5)	C(3)—C(31)—C(32)	124 (1)
Pb(1)—C(1)—C(11)	112.4 (9)	C(32)—C(31)—C(36)	115 (1)
C(1)—C(11)—C(16)	119 (1)	Cl(3)—C(32)—C(31)	118 (1)
C(1)—C(11)—C(12)	124 (1)	C(31)—C(32)—C(33)	124 (1)
C(12)—C(11)—C(16)	117 (1)	Cl(3)—C(32)—C(33)	119 (1)
Cl(1)—C(12)—C(11)	119 (1)	C(32)—C(33)—C(34)	119 (2)
C(11)—C(12)—C(13)	123 (1)	C(33)—C(34)—C(35)	120 (2)
Cl(1)—C(12)—C(13)	118 (1)	C(34)—C(35)—C(36)	119 (2)
C(12)—C(13)—C(14)	119 (2)	C(31)—C(36)—C(35)	123 (1)
C(13)—C(14)—C(15)	120 (2)	Pb(1)—C(4)—C(41)	109.1 (9)
C(14)—C(15)—C(16)	122 (2)	C(4)—C(41)—C(46)	124 (1)
C(11)—C(16)—C(15)	119 (2)	C(4)—C(41)—C(42)	120 (1)
Pb(1)—C(2)—C(21)	110.9 (9)	C(42)—C(41)—C(46)	116 (1)
C(2)—C(21)—C(26)	122 (1)	Cl(4)—C(42)—C(41)	120 (1)
C(2)—C(21)—C(22)	122 (1)	C(41)—C(42)—C(43)	122 (1)
C(22)—C(21)—C(26)	115 (1)	Cl(4)—C(42)—C(43)	118 (1)
Cl(2)—C(22)—C(21)	118.7 (9)	C(42)—C(43)—C(44)	119 (2)
C(21)—C(22)—C(23)	125 (1)	C(43)—C(44)—C(45)	120 (2)
Cl(2)—C(22)—C(23)	116 (1)	C(44)—C(45)—C(46)	123 (2)
C(22)—C(23)—C(24)	116 (1)	C(41)—C(46)—C(45)	120 (2)

C(3)—Pb(1)—C(4)—C(41)	95 (1)	C(3)—Pb(1)—C(2)—C(21)	−8 (1)
C(2)—Pb(1)—C(4)—C(41)	−19 (1)	C(4)—Pb(1)—C(2)—C(21)	103.4 (9)
C(1)—Pb(1)—C(4)—C(41)	−145.7 (9)	C(4)—Pb(1)—C(3)—C(31)	118.5 (9)
C(2)—Pb(1)—C(3)—C(31)	−125.8 (9)	Pb(1)—C(1)—C(11)—C(12)	−76 (2)
C(1)—Pb(1)—C(3)—C(31)	−3 (1)	Pb(1)—C(1)—C(11)—C(16)	102 (1)
C(1)—Pb(1)—C(2)—C(21)	−128.8 (9)	Pb(1)—C(2)—C(21)—C(22)	78 (1)
C(2)—Pb(1)—C(1)—C(11)	20 (1)	Pb(1)—C(2)—C(21)—C(26)	−101 (1)
C(3)—Pb(1)—C(1)—C(11)	−98 (1)	Pb(1)—C(3)—C(31)—C(32)	−72 (1)
C(4)—Pb(1)—C(1)—C(11)	145 (1)	Pb(1)—C(3)—C(31)—C(36)	108 (1)

Plane	Equation of the plane*
1 C(11),C(12),C(13),C(14),C(15),C(16)	$0.188x + 0.958y - 0.215z = 3.89$ (5)
2 C(21),C(22),C(23),C(24),C(25),C(26)	$0.853x - 0.495y - 0.169z = 0.58$ (2)
3 C(31),C(32),C(33),C(34),C(35),C(36)	$0.907x + 0.392y - 0.155z = -0.98$ (3)
4 C(41),C(42),C(43),C(44),C(45),C(46)	$0.250x - 0.535y - 0.807z = -6.99$ (1)

1,2 106.1 (4); 1,3 54.6 (4); 1,4 107.0 (4); 2,3 52.7 (4); 2,4 52.1 (4); 3,4 81.8 (4).

* x along **a**, y in the ab plane, z along **c**.

Discussion. The molecular structure of the title compound and the numbering scheme are shown in Fig. 1. A stereoscopic view of the unit cell is presented in Fig. 2. Positional parameters and the equivalent isotropic values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* bond lengths, bond angles, least-squares planes and dihedral angles are shown in Table 2.

The coordination polyhedron around the Pb atom formed by the C atoms of the four CH_2 groups of the benzyl ligands is a slightly distorted tetrahedron. The Pb—C bond distances are comparable with those of the three Pb—CH_3 bonds in $Me_3PbC(N_2)COOEt$ [mean value 2.22 (2–4) Å, Pb anisotropically refined only (Birkhahn, Glozbach, Massa

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55954 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1031]

& Lorberth, 1980)]. They are significantly longer than those in trialkyllead compounds R_3PbX (X = carboxylate) [$Me_3PbOOCC_6H_4(2\text{-}NH_2)$ 2.18 (2) Å (Fehlberg-Sternemann, 1992); $Me_3PbOOC(2\text{-}C_5H_3O)$ 2.17 (2) Å (Preut, Röhm & Huber, 1986)] and dialkyllead compounds R_2PbX_2 [dimethyllead bis(4-pyrimidinecarboxylate) trihydrate 2.142 (6) Å (Hoffmann, 1988)]. No other data on Pb—C bond distances in solid alkyllead compounds are available. A Pb—C bond distance in $Pb(CH_3)_4$ of 2.238 (9) Å was determined by gas-phase electron diffraction (Oyamada, Iijima & Kimura, 1971).

Financial assistance from the Fonds der Chemischen Industrie is gratefully acknowledged.

References

BÄHR, G. & ZOCHE, G. (1955). *Chem. Ber.* **88**, 542–550.
BIRKHAHN, M., GLOZBACH, E., MASSA, W. & LORBERTH, J. (1980). *J. Organomet. Chem.* **192**, 171–176.
FEHLBERG-STERNEMANN, G. (1992). Dissertation. Univ. of Dortmund, Germany.
HOFFMANN, E. (1988). Dissertation. Univ. of Dortmund, Germany.
LE PAGE, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
OYAMADA, T., IIJIMA, T. & KIMURA, M. (1971). *Bull. Chem. Soc. Jpn*, **44**, 2638–2642.
PREUT, H., RÖHM, P. & HUBER, F. (1986). *Acta Cryst.* C**42**, 657–658.
SHELDRICK, G. M. (1987). *SHELXTL-Plus.* Release 3.4 for Nicolet *R3m/V* crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
SPEK, A. L. (1990). *Acta Cryst.* A**46**, C34.

Acta Cryst. (1993). C**49**, 1068–1070

Structure of Tetrabutylammonium Bis[benzene-1,2-dithiolato(2−)]oxorhenate(V)

BY RAINER HÜBENER AND ULRICH ABRAM*

University of Tübingen, Institute of Inorganic Chemistry, Auf der Morgenstelle 18, *W*-7400 *Tübingen, Germany*

(*Received* 6 *October* 1992; *accepted* 11 *February* 1993)

Abstract. $[C_{16}H_{36}N][Re(C_6H_4S_2)_2O]$, $M_r = 725.19$, orthorhombic, $Pna2_1$, $a = 37.912$ (3), $b = 9.286$ (1), $c = 17.482$ (3) Å, $V = 6154.6$ Å^3, $Z = 8$, $D_x = 1.565$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.709300 Å, $\mu = 4.284$ mm^{-1}, $F(000) = 2928$, $T = 215$ K. The final R value is 0.046 for 4236 significant [$I > 3\sigma(I)$] reflections. The structure consists of discrete $[ReO(bdt)_2]^{2-}$ anions, where bdt^{2-} is the benzene-1,2-dithiolato ligand, and $(Bu_4N)^+$ cations. The Re atom is pentacoordinate with the four S atoms in the basal positions and the oxo ligand at the apex of a square pyramid.

Introduction. In recent years, radioactive rhenium compounds (^{186}Re and ^{188}Re: β^--emitter with β^- energies between 0.17 and 1.12 MeV and half lives of 90 and 17 h, respectively) have been considered as potential radiopharmaceuticals in therapeutic nuclear medicine (Vanderheyden, Heeg & Deutsch, 1985). Therefore, there is considerable interest in detailed structural information on rhenium complexes, especially in comparison with technetium compounds, the biological distribution pattern of which is well known.

Experimental. The title compound was formed as a by-product (about 20%) during the reaction of $[Bu_4N][ReOBr_4]$ with 2,2′-(thioethylenethio)dibenzenethiol. Benzene-1,2-dithiol was formed by partial decomposition of the potentially terdentate organic ligand. An orange–brown single crystal of approximate dimensions 0.1 × 0.15 × 0.5 mm was mounted on a glass fiber. The complex crystallizes in the space group $Pna2_1$ with two independent formula units per unit cell. In the final full-matrix least-squares refinement, all non-H atoms were assigned anisotropic atomic displacement parameters. H atoms were included in calculated positions. 2000 Friedel pairs have been measured to confirm the non-centrosymmetric space group. The absolute structure has not been determined. A summary of data collection and structure refinement parameters is given in Table 1.† Final atomic coordinates are listed in Table 2. Table 3 contains selected bond

---

* Author to whom correspondence should be addressed.

† Full lists of structure factors, H-atom positions, anisotropic atomic displacement parameters, bond angles and distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55953 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1033]

0108-2701/93/061068-03$06.00