

Compound (I) crystallized in the triclinic system; space group  $P\bar{1}$  was assumed and confirmed by the successful refinement. Compound (II) crystallized in the tetragonal system and a survey of the reflection intensities revealed that the Laue group was  $4/mmm$ . This, with the systematic absences ( $hkl$  absent if  $l = 2n + 1$ ,  $h00$  absent if  $h = 2n + 1$ ), determined the space group as  $P4_2/c$ ; furthermore,  $Z = 2$  required that (II) have  $\bar{4}$  symmetry. For both (I) and (II) all non-H atoms were refined by full-matrix least-squares calculations on  $F$ . All H atoms were clearly visible in difference maps and they were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. Space group  $P4_2/c$  is chiral and refinement with  $\eta = -1$  converged with higher values of  $R$  and  $wR$  (0.029 and 0.037, respectively) than were found for  $\eta = +1$  (0.025 and 0.030, respectively) (Rodgers, 1981); thus the absolute configuration is as shown.

For both compounds, data collection and cell refinement: Enraf-Nonius CAD-4; data reduction, program used to solve and refine structure, and software used to prepare material for publication: NRCVAX (Gabe, Le Page, Charland, Lee & White, 1989); molecular graphics: ORTEPII (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Akhmed, N. A. & Aleksandrov, G. G. (1970). *Zh. Strukt. Khim.* **11**, 891–894.
- Belsky, V. K., Simonenko, A. A., Reikhsfeld, V. O. & Saratov, I. E. (1983). *J. Organomet. Chem.* **244**, 125–128.
- Chieh, P. C. & Trotter, J. (1970). *J. Chem. Soc. A*, pp. 911–914.
- Davies, G. R., Jarvis, J. A. J. & Kilbourn, B. T. (1971). *J. Chem. Soc. Chem. Commun.* pp. 1551–1552.
- Engelhardt, L. M., Leung, W.-P., Raston, C. L. & White, A. H. (1982). *Aust. J. Chem.* **35**, 2383–2384.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Howie, R. A., Ross, J.-N., Wardell, J. L. & Low, J. N. (1994). *Acta Cryst.* **C50**, 229–231.
- Karipides, A., Forman, C., Thomas, R. H. P. & Reed, A. T. (1974). *Inorg. Chem.* **13**, 811–815.
- Karipides, A. & Oertel, M. (1977). *Acta Cryst.* **B33**, 683–687.
- Karipides, A. & Wolfe, K. (1975). *Acta Cryst.* **B31**, 605–608.
- Rodgers, D. (1981). *Acta Cryst.* **A37**, 734–741.
- Ross, J.-N., Wardell, J. L., Ferguson, G. & Low, J. N. (1994). *Acta Cryst.* **C50**, 1207–1209.
- Spek, A. L. (1992). *PLATON Molecular Geometry Program*. Univ. of Utrecht, The Netherlands.
- Wharf, I. & Simard, M. G. (1987). *J. Organomet. Chem.* **332**, 85–94.
- Wharf, I. & Simard, M. G. (1991). *Acta Cryst.* **C47**, 1314–1315.
- Wharf, I., Simard, M. G. & Lamparski, H. (1990). *Can. J. Chem.* **68**, 1277–1282.
- Van Campen, M. G., Meisner, D. F. & Parmeter, S. M. (1948). *J. Am. Chem. Soc.* **70**, 2296–2297.

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## Benzyltriphenyllead and Dibenzyldiphenyllead

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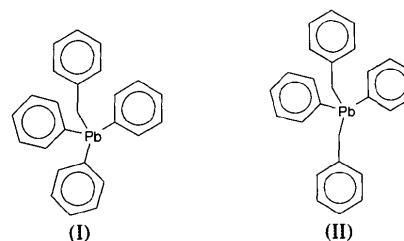
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### Abstract

In benzyltriphenyllead,  $[\text{Pb}(\text{C}_6\text{H}_5)_3(\text{C}_7\text{H}_7)]$ , (I), and dibenzyldiphenyllead,  $[\text{Pb}(\text{C}_6\text{H}_5)_2(\text{C}_7\text{H}_7)_2]$ , (II), each Pb atom is in the centre of a distorted tetrahedron. In (I) the C(Bz)—Pb—C(Ph) angles  $[110.4(3)–113.1(3)^\circ]$  are larger than the C(Ph)—Pb—C(Ph) angles  $[106.6(3)–107.5(3)^\circ]$  suggesting a steric effect of the benzyl group (Bz). The angles in (II) [C(Bz)—Pb—C(Bz)  $107.8(3)^\circ$ ; C(Bz)—Pb—C(Ph)  $104.6(3)$  and  $113.1(3)^\circ$ ; C(Ph)—Pb—C(Ph)  $111.1(3)^\circ$ ] reflect a more distinct distortion, but no similar steric effect of the benzyl groups is clearly recognizable.

### Comment

The present work continues structural studies of benzyllead compounds, such as tetrakis(2-chlorobenzyl)lead (Fahrenkamp, Schürmann & Huber, 1993) or benzyldiphenyllead bromide (Fahrenkamp, Schürmann & Huber, 1994). The structures of the title compounds have been determined in the context of studies of possible specific effects of unsubstituted and substituted benzyl ligands (Bz) on the bonding of Pb in compounds  $\text{Bz}_{4-n}\text{PbR}_n$  ( $R$  = organo group;  $n = 0–3$ ), which in contrast to comparable tetraorganolead compounds exhibit unusual properties. (Compounds containing more than one benzyl ligand, for instance, are coloured and sensitive to air and light.)



In (I) and (II) the coordination polyhedron around Pb is a slightly distorted tetrahedron formed by four C atoms. The Pb—C(Ph) distances in both compounds are not significantly different and are similar to the distance of 2.194(6) Å found in  $\text{PbPh}_4$  (Preut & Huber, 1993),

and to those in (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)Ph<sub>2</sub>PbBr [mean value 2.207 (9) Å; Fahrenkamp, Schürmann & Huber, 1994]. The values of the two Pb—C(Bz) distances in (II) are practically the same and are hardly significantly different from that in (I); they are also comparable to those in Pb(CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>Cl-2)<sub>4</sub> [mean value 2.26 (1) Å; Fahrenkamp, Schürmann & Huber, 1993]. The Pb—C(Bz) distance in (I) [2.242 (7) Å] is comparable to that in (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)Ph<sub>2</sub>PbBr [Pb—CH<sub>2</sub> 2.214 (8) Å; Fahrenkamp, Schürmann & Huber, 1994]. The Pb—C(Bz) distance in (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)Ph<sub>2</sub>PbBr [2.214 (8) Å; Fahrenkamp, Schürmann & Huber, 1994] is rather short, and is actually closer to the Ph—C(Ph) distances in (I) and (II) than to the Ph—C(Bz) distances.

The distortions of the tetrahedral geometries at Pb in (I) and (II) are reflected more by the C—Pb—C angles

than by the Pb—C distances. In (I), the C(Ph)—Pb—C(Ph) angles [106.6 (3)–107.5 (3)°] are substantially smaller than the C(Ph)—Pb—C(Bz) angles [110.4 (3)–113.1 (3)°]. Thus, the PbPh<sub>3</sub> group appears to be contracted, which might be correlated with a steric effect of the benzyl group. A similar obvious distortion of the tetrahedral geometry anticipated for tetraorganolead compounds towards a trigonal pyramid was not observed in triphenylcyclopentadienyllead (Gaffney & Harrison, 1982), the only other triphenylorganolead compound for which the structure in the solid state is known. In (II), no relation between the deviations of the C—Pb—C angles from the ideal tetrahedral value and the type of organic group present is clearly discernible; the C(Bz)—Pb—C(Bz) angle [107.8 (3)°] is rather small, while in contrast to (I) the C(Ph)—Pb—C(Ph) angle [111.1 (3)°] is rather large. The C(Bz)—Pb—C(Ph) angles in (II) differ widely [104.6 (3) and 113.1 (3)°] indicating that the distortion of (II) is stronger than that of (I).

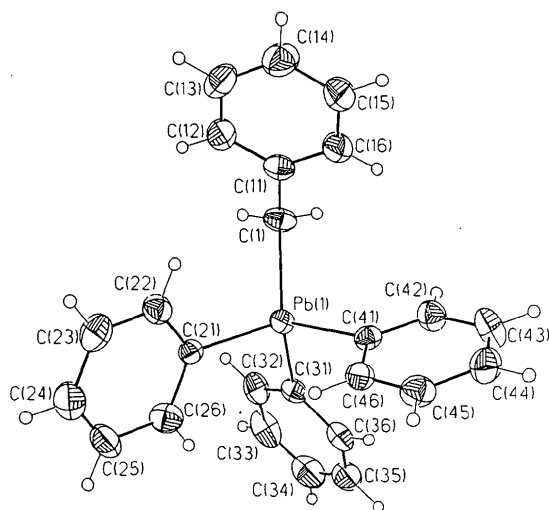


Fig. 1. Structure of (I) (*SHELXTL-Plus*; Sheldrick, 1987) showing the atom-numbering scheme. The probability level of the displacement ellipsoids is 50%.

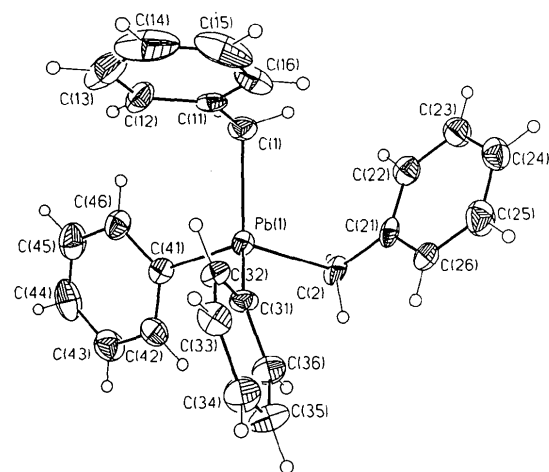


Fig. 2. Structure of (II) (*SHELXTL-Plus*; Sheldrick, 1987) showing the atom-numbering scheme. The probability level of the displacement ellipsoids is 50%.

## Experimental

Compounds (I) and (II) were obtained by the reaction of C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>MgBr with Ph<sub>3</sub>PbCl and Ph<sub>2</sub>PbCl<sub>2</sub>, respectively, in ether solution under exclusion of air (Schlöttig, 1927). Compound (I) was recrystallized from methanol; m.p. 365 K. Compound (II) was recrystallized from ethanol/benzene; m.p. 400 K.

## Compound (I)

### Crystal data

[Pb(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(C<sub>7</sub>H<sub>7</sub>)]

$M_r = 529.62$

Triclinic

$P\bar{1}$

$a = 10.016 (7) \text{ \AA}$

$b = 10.734 (10) \text{ \AA}$

$c = 11.348 (9) \text{ \AA}$

$\alpha = 102.52 (7)^\circ$

$\beta = 104.16 (6)^\circ$

$\gamma = 112.10 (6)^\circ$

$V = 1029.3 (15) \text{ \AA}^3$

$Z = 2$

$D_x = 1.709 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 28

reflections

$\theta = 7.8\text{--}14.7^\circ$

$\mu = 8.200 \text{ mm}^{-1}$

$T = 170 (1) \text{ K}$

Block

$0.50 \times 0.34 \times 0.28 \text{ mm}$

Colourless

### Data collection

Nicolet R3m/V diffractometer

$\omega/2\theta$  scans (speed 3.0–  
15.0° min<sup>-1</sup> in  $\theta$ )

Absorption correction:

empirical

$T_{\min} = 0.804$ ,  $T_{\max} =$

1.000

3914 measured reflections

3646 independent reflections

3133 observed reflections

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

$R_{\text{int}} = 0.0698$

$\theta_{\text{max}} = 25.06^\circ$

$h = -11 \rightarrow 1$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 13$

6 standard reflections

monitored every 300

reflections

intensity variation:

$< \pm 5.8\%$

## Refinement

Refinement on  $F^2$  $R(F) = 0.0368$  $wR(F^2) = 0.0901$  $S = 1.05$ 

3645 reflections

237 parameters

Only H-atom  $U$ 's refined $w = 1/[\sigma^2(F_o)^2 + (0.0635P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $\Delta\rho_{\max} = 2.832 \text{ e } \text{Å}^{-3}$ 

(near Pb)

 $\Delta\rho_{\min} = -1.733 \text{ e } \text{Å}^{-3}$ 

Extinction correction: none

Atomic scattering factors

from *International Tables*for *Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

## Compound (II)

Crystal data

 $[\text{Pb}(\text{C}_6\text{H}_5)_2(\text{C}_7\text{H}_7)_2]$  $M_r = 543.64$ 

Monoclinic

 $P2_1/n$  $a = 13.452 (12) \text{ Å}$  $b = 10.974 (8) \text{ Å}$  $c = 14.487 (10) \text{ Å}$  $\beta = 96.39 (6)^\circ$  $V = 2125.3 (29) \text{ Å}^3$  $Z = 4$  $D_x = 1.699 \text{ Mg m}^{-3}$ Mo  $K\alpha$  radiation $\lambda = 0.71073 \text{ Å}$ 

Cell parameters from 34

reflections

 $\theta = 7.6\text{--}14.1^\circ$  $\mu = 7.945 \text{ mm}^{-1}$  $T = 170 (1) \text{ K}$ 

Block

 $0.44 \times 0.40 \times 0.16 \text{ mm}$ 

Yellow

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ ) for (I)
$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	x	y	z	$U_{\text{eq}}$
Pb(1)	0.15283 (3)	0.27792 (3)	0.45264 (3)	0.02766 (1)
C(1)	0.2682 (10)	0.1376 (9)	0.4188 (7)	0.037 (2)
C(11)	0.2172 (9)	0.0561 (9)	0.2784 (7)	0.032 (2)
C(12)	0.1050 (10)	-0.0867 (9)	0.2223 (8)	0.039 (2)
C(13)	0.0606 (1)	-0.1604 (9)	0.0909 (9)	0.049 (2)
C(14)	0.1276 (12)	-0.0942 (10)	0.0151 (9)	0.051 (2)
C(15)	0.2355 (12)	0.0457 (10)	0.0684 (9)	0.050 (2)
C(16)	0.2809 (10)	0.1207 (9)	0.1987 (8)	0.041 (2)
C(21)	-0.1004 (8)	0.1550 (7)	0.4000 (7)	0.0264 (15)
C(22)	-0.1924 (9)	0.0384 (9)	0.2883 (7)	0.035 (2)
C(23)	-0.3513 (10)	-0.0305 (9)	0.2567 (8)	0.044 (2)
C(24)	-0.4204 (10)	0.0164 (10)	0.3354 (10)	0.047 (2)
C(25)	-0.3313 (10)	0.1322 (10)	0.4457 (9)	0.047 (2)
C(26)	-0.1710 (10)	0.2043 (9)	0.4782 (8)	0.039 (2)
C(31)	0.2486 (8)	0.4179 (8)	0.6577 (7)	0.028 (2)
C(32)	0.2573 (9)	0.3632 (10)	0.7593 (8)	0.038 (2)
C(33)	0.3157 (11)	0.4563 (12)	0.8852 (8)	0.053 (3)
C(34)	0.3603 (11)	0.6008 (12)	0.9125 (9)	0.057 (3)
C(35)	0.3544 (10)	0.6564 (10)	0.8149 (9)	0.049 (2)
C(36)	0.2976 (9)	0.5653 (9)	0.6880 (3)	0.039 (2)
C(41)	0.1904 (9)	0.4220 (8)	0.3367 (7)	0.028 (2)
C(42)	0.3338 (9)	0.5008 (9)	0.3319 (8)	0.036 (2)
C(43)	0.3537 (10)	0.5917 (11)	0.2602 (9)	0.048 (2)
C(44)	0.2295 (11)	0.6055 (9)	0.1930 (8)	0.043 (2)
C(45)	0.0830 (10)	0.5280 (9)	0.1968 (8)	0.039 (2)
C(46)	0.0641 (9)	0.4370 (8)	0.2669 (7)	0.033 (2)

Table 2. Selected geometric parameters ( $\text{Å}$ ,  $^\circ$ ) for (I)

Pb(1)—C(31)	2.214 (8)	C(24)—C(25)	1.361 (13)
Pb(1)—C(21)	2.220 (7)	C(25)—C(26)	1.402 (12)
Pb(1)—C(41)	2.225 (7)	C(31)—C(36)	1.400 (11)
Pb(1)—C(1)	2.242 (7)	C(31)—C(32)	1.401 (11)
C(1)—C(11)	1.493 (11)	C(32)—C(33)	1.384 (13)
C(11)—C(16)	1.388 (11)	C(33)—C(34)	1.380 (15)
C(11)—C(12)	1.396 (12)	C(34)—C(35)	1.366 (15)
C(12)—C(13)	1.395 (13)	C(35)—C(36)	1.387 (12)
C(13)—C(14)	1.372 (13)	C(41)—C(42)	1.387 (11)
C(14)—C(15)	1.362 (13)	C(41)—C(46)	1.405 (11)
C(15)—C(16)	1.386 (12)	C(42)—C(43)	1.390 (12)
C(21)—C(22)	1.376 (10)	C(43)—C(44)	1.370 (13)
C(21)—C(26)	1.398 (11)	C(44)—C(45)	1.405 (13)
C(22)—C(23)	1.389 (12)	C(45)—C(46)	1.376 (11)
C(23)—C(24)	1.378 (13)		
C(31)—Pb(1)—C(21)	107.2 (3)	C(11)—C(1)—Pb(1)	111.7 (5)
C(31)—Pb(1)—C(41)	106.6 (3)	C(16)—C(11)—C(1)	120.6 (8)
C(21)—Pb(1)—C(41)	107.5 (3)	C(12)—C(11)—C(1)	121.8 (8)
C(31)—Pb(1)—C(1)	111.8 (3)	C(22)—C(21)—Pb(1)	122.6 (5)
C(21)—Pb(1)—C(1)	113.1 (3)	C(26)—C(21)—Pb(1)	118.7 (5)
C(41)—Pb(1)—C(1)	110.4 (3)	C(36)—C(31)—Pb(1)	119.6 (6)
C(42)—C(41)—Pb(1)	123.2 (6)	C(32)—C(31)—Pb(1)	122.1 (6)
C(46)—C(41)—Pb(1)	118.8 (6)		

## Data collection

Nicolet R3m/V diffractometer

 $\omega/2\theta$  scans (speed 3.0–15.0° min<sup>-1</sup> in  $\theta$ )

Absorption correction:

empirical

 $T_{\min} = 0.340$ ,  $T_{\max} =$ 

1.000

4357 measured reflections

3747 independent reflections

2735 observed reflections

 $[I > 2\sigma(I)]$  $R_{\text{int}} = 0.0548$  $\theta_{\max} = 25.05^\circ$  $h = -16 \rightarrow 0$  $k = -1 \rightarrow 13$  $l = -17 \rightarrow 17$ 

6 standard reflections

monitored every 300

reflections

intensity variation:

 $< \pm 3.4\%$ 

## Refinement

Refinement on  $F^2$  $R(F) = 0.0399$  $wR(F^2) = 0.0962$  $S = 1.05$ 

3746 reflections

246 parameters

Only H-atom  $U$ 's refined $w = 1/[\sigma^2(F_o)^2 + (0.0628P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $\Delta\rho_{\max} = 2.459 \text{ e } \text{Å}^{-3}$ 

(near Pb)

 $\Delta\rho_{\min} = -2.591 \text{ e } \text{Å}^{-3}$ 

Extinction correction: none

Atomic scattering factors

from *International Tables*for *Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ ) for (II)
$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	x	y	z	$U_{\text{eq}}$
Pb(1)	0.16673 (2)	0.17496 (3)	0.14113 (2)	0.02284 (12)
C(1)	0.0989 (7)	0.2606 (9)	0.0062 (6)	0.032 (2)
C(11)	0.1510 (6)	0.3758 (8)	-0.0157 (6)	0.025 (2)
C(12)	0.1307 (7)	0.4867 (8)	0.0249 (7)	0.038 (2)
C(13)	0.1785 (9)	0.5913 (10)	0.0050 (10)	0.061 (4)
C(14)	0.2491 (11)	0.5883 (14)	-0.0577 (10)	0.073 (5)
C(15)	0.2703 (8)	0.4807 (14)	-0.0999 (7)	0.060 (4)
C(16)	0.2216 (7)	0.3738 (11)	-0.0780 (6)	0.041 (3)
C(2)	0.1670 (7)	-0.0294 (8)	0.1209 (6)	0.030 (2)
C(21)	0.2018 (6)	-0.0537 (7)	0.0286 (6)	0.024 (2)
C(22)	0.1354 (7)	-0.0618 (8)	-0.0525 (6)	0.033 (2)
C(23)	0.1676 (8)	-0.0798 (9)	-0.1389 (7)	0.039 (2)
C(24)	0.2691 (7)	-0.0900 (9)	-0.1464 (7)	0.037 (2)
C(25)	0.3360 (8)	-0.0822 (10)	-0.0678 (7)	0.042 (3)
C(26)	0.3030 (7)	-0.0638 (8)	0.0187 (7)	0.033 (2)
C(31)	0.3265 (6)	0.2232 (9)	0.1778 (6)	0.028 (2)
C(32)	0.3690 (6)	0.3315 (8)	0.1476 (6)	0.027 (2)
C(33)	0.4693 (7)	0.3573 (9)	0.1750 (6)	0.033 (2)
C(34)	0.5278 (7)	0.2795 (10)	0.2339 (7)	0.041 (3)
C(35)	0.4867 (7)	0.1757 (10)	0.2637 (7)	0.044 (3)
C(36)	0.3863 (7)	0.1463 (9)	0.2357 (6)	0.033 (2)

C(41)	0.0761 (6)	0.2287 (8)	0.2526 (6)	0.025 (2)
C(42)	0.0909 (7)	0.1776 (9)	0.3409 (6)	0.034 (2)
C(43)	0.0301 (8)	0.2043 (10)	0.4083 (7)	0.046 (3)
C(44)	-0.0481 (8)	0.2867 (10)	0.3893 (8)	0.050 (3)
C(45)	-0.0643 (8)	0.3384 (11)	0.3030 (8)	0.048 (3)
C(46)	-0.0046 (7)	0.3101 (9)	0.2354 (7)	0.035 (2)

Table 4. Selected geometric parameters (Å, °) for (II)

Pb(1)—C(41)	2.209 (9)	C(23)—C(24)	1.385 (14)
Pb(1)—C(31)	2.220 (9)	C(24)—C(25)	1.373 (14)
Pb(1)—C(2)	2.262 (9)	C(25)—C(26)	1.390 (13)
Pb(1)—C(1)	2.267 (9)	C(31)—C(36)	1.383 (12)
C(1)—C(11)	1.496 (12)	C(31)—C(32)	1.409 (13)
C(11)—C(16)	1.381 (13)	C(32)—C(33)	1.392 (12)
C(11)—C(12)	1.392 (13)	C(33)—C(34)	1.388 (14)
C(12)—C(13)	1.363 (15)	C(34)—C(35)	1.357 (14)
C(13)—C(14)	1.39 (2)	C(35)—C(36)	1.404 (13)
C(14)—C(15)	1.37 (2)	C(41)—C(42)	1.391 (13)
C(15)—C(16)	1.40 (2)	C(41)—C(46)	1.407 (12)
C(2)—C(21)	1.488 (11)	C(42)—C(43)	1.373 (14)
C(21)—C(26)	1.390 (12)	C(43)—C(44)	1.391 (15)
C(21)—C(22)	1.397 (12)	C(44)—C(45)	1.37 (2)
C(22)—C(23)	1.384 (13)	C(45)—C(46)	1.369 (14)
C(41)—Pb(1)—C(31)	111.1 (3)	C(12)—C(11)—C(1)	122.1 (9)
C(41)—Pb(1)—C(2)	111.6 (3)	C(21)—C(2)—Pb(1)	107.4 (5)
C(31)—Pb(1)—C(2)	104.6 (3)	C(26)—C(21)—C(2)	121.1 (8)
C(41)—Pb(1)—C(1)	108.5 (3)	C(22)—C(21)—C(2)	122.1 (8)
C(31)—Pb(1)—C(1)	113.1 (3)	C(36)—C(31)—Pb(1)	118.8 (7)
C(2)—Pb(1)—C(1)	107.8 (3)	C(32)—C(31)—Pb(1)	122.9 (6)
C(11)—C(1)—Pb(1)	112.3 (5)	C(42)—C(41)—Pb(1)	122.1 (7)
C(16)—C(11)—C(1)	119.8 (9)	C(46)—C(41)—Pb(1)	121.2 (7)

No systematic absences were detected for compound (I). In (II), the systematic absences ( $h0l$ )  $h + l = 2n + 1$  and ( $0k0$ )  $k = 2n + 1$  were detected. Both structures were solved by standard Patterson and difference Fourier methods (*SHELXTL-Plus*; Sheldrick 1987). Structure (I) refined satisfactorily in space group  $P\bar{1}$  (No. 2) and structure (II) refined satisfactorily in space group  $P2_1/n$  (No. 14) by full-matrix least-squares calculations using *SHELXL93* (Sheldrick, 1994). H atoms were placed in geometrically calculated positions and refined with common isotropic displacement factors for different C—H types (C—H<sub>aryl</sub> 0.95 Å, C—H<sub>alkyl</sub> 0.99 Å). Other programs used were *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, complete geometry and least-squares-planes data, and stereoviews of both structures have been deposited with the IUCr (Reference: AB1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Fahrenkamp, U., Schürmann, M. & Huber, F. (1993). *Acta Cryst.* **C49**, 1066–1068.  
 Fahrenkamp, U., Schürmann, M. & Huber, F. (1994). *Acta Cryst.* **C50**. In the press.  
 Gaffney, C. & Harrison, P. G. (1982). *J. Chem. Soc. Dalton Trans.* pp. 1055–1060.  
 Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.  
 Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.  
 Preut, H. & Huber, F. (1993). *Acta Cryst.* **C49**, 1372–1373.  
 Schlöttig, O. (1927). Dissertation, Techn. Hochschule, Berlin.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELX93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.

*Acta Cryst.* (1994). **C50**, 1710–1713

## A Redetermination of Lead(IV) Acetate

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### Abstract

The redetermination of Pb(OCOCH<sub>3</sub>)<sub>4</sub> corroborates and complements the limited data of a prior, yet incomplete study. The structure consists of two discrete molecules in each of which the Pb atom is surrounded by four bidentate acetate ligands forming a nearly dodecahedral polyhedron.

### Comment

About 30 years ago in a short conference note, Kamenar (1963) reported that lead(IV) acetate, Pb(OAc)<sub>4</sub> (OAc = CH<sub>3</sub>COO), crystallizes in space group  $P2_1/c$  with  $Z = 8$ , the structure consisting of two symmetrically independent sets of molecules. The polyhedron around the eight-coordinate Pb atom was described as a flattened trigonal dodecahedron. Apart from the cell dimensions [ $a = 13.01$  (2),  $b = 26.59$  (4),  $c = 7.86$  (2) Å,  $\beta = 105.6$  (2)°], no other data were given. A full description was announced after due refinement of the atomic coordinates (Kamenar, 1963; Kamenar & Bruvo, 1972), but to our knowledge it was not published. Since our studies on organolead(IV) carboxylates would profit from detailed structural data of Pb(OAc)<sub>4</sub>, (I), we have redetermined the structure of this compound.

