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Structure of Diphenyllead 2,6-Pyridinedicarboxylate Hydrate

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Abstract. $[Pb(C_6H_5)_2][C_7H_3NO_4].H_2O$, $M_r = 544.5$, monoclinic, $P2_1/c$, $a = 9.481$ (4), $b = 10.156$ (18), $c = 19.309$ (8) Å, $\beta = 98.52$ (4)°, $V = 1838.7$ Å^3, $Z = 4$, $D_x = 1.967$ Mg m^{-3}, $F(000) = 1032$, $\lambda(Ag\,K\alpha) = 0.5608$ Å, $\mu = 5.0$ mm^{-1}, $T = 291$ (1) K, final $R = 0.024$ for 2472 unique observed $[I \geq 1.96\sigma(I)]$ diffractometer data. The coordination polyhedron around Pb is a pentagonal bipyramid, the phenyl groups being in the apical positions. One of the carboxylate groups of the tridentate dianionic ligand is bridging, thereby forming an infinite chain. The chains are probably linked by pairs of H bonds between each water molecule coordinated in the equatorial plane and each non-bridging carboxylate group of two units.

Experimental. The title compound is obtained from diphenyllead diacetate and 2,6-pyridinedicarboxylic acid by refluxing in methanol. Colourless crystals from methanol, dimensions $0.16 \times 0.40 \times 0.04$ mm, $\omega/2\theta$ scans, scan speed 2.0–$6.67°$ min^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$; lattice parameters from least-squares fit of 25 reflections up to $2\theta = 24.6°$; six standard reflections recorded every 2.5 h, only random deviations; 7559 reflections measured, $1 \leq \theta \leq 20°$, $-11 \leq h \leq 11$, $0 \leq k \leq 12$, $-23 \leq l \leq 23$; after averaging ($R_{int} = 0.026$) 3713 unique reflections, 2472 with $I \geq 1.96\sigma(I)$; Lorentz–polarization correction; absorption correction *via* ψ scans; max./min. transmission $1.00/0.67$; systematic absences $h0l$ $l = 2n + 1$, $0k0$ $k = 2n + 1$ conform to space group $P2_1/c$; structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic tempera-

ture factor for H atoms, which were placed in geometrically calculated positions (C–H 0.95 Å) for the phenyl and pyridine rings; refinement on F with 2472 reflections and 236 refined parameters; $w = 4F_o^2/[\sigma^2(F_o^2) + (0.065\,F_o^2)^2]$; $S = 0.72$, $R = 0.024$, $wR = 0.033$, $(\Delta/\sigma)_{max} = 0.01$, no extinction correction; largest peak in final ΔF map ± 0.8 (3) e Å^{-3}, atomic scattering factors and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1981), *SHELXTL PLUS* (Sheldrick, 1987), *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980).

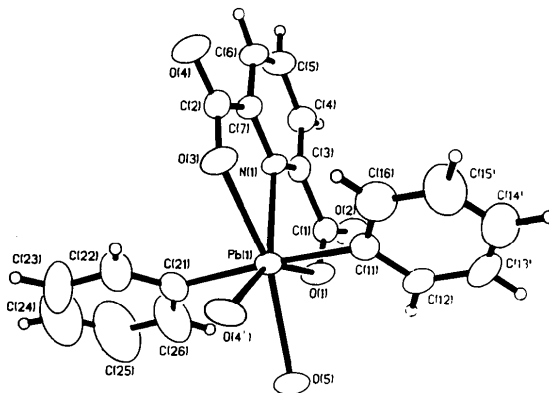The molecule and the numbering scheme are shown in Fig. 1. Fig. 2 shows the infinite chains which are
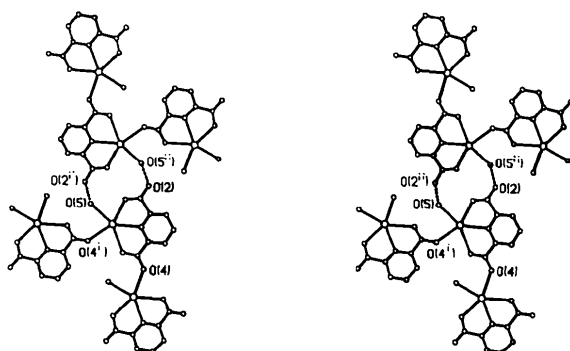

Fig. 1. General view of one formula unit plus the bridging $O(4^i)$, showing the atom-numbering scheme [(i) corresponds to the symmetry operation $-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$]. H atoms at O(5) are omitted.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$

$$U_{eq} = (\tfrac{1}{6}\pi^2)\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pb(1)	0·03364 (2)	−0·16933 (2)	0·15702 (1)	30
O(1)	0·1032 (5)	−0·1309 (4)	0·0456 (2)	39
O(2)	0·1661 (6)	−0·2116 (5)	−0·0521 (2)	46
O(3)	0·0169 (5)	−0·3786 (5)	0·2252 (2)	42
O(4)	0·0598 (6)	−0·5957 (5)	0·2341 (3)	47
O(5)	0·0237 (5)	0·0721 (5)	0·1409 (3)	44
N(1)	0·1077 (5)	−0·3700 (5)	0·1017 (3)	28
C(1)	0·1395 (7)	−0·2223 (7)	0·0082 (3)	34
C(2)	0·0570 (6)	−0·4889 (7)	0·2041 (3)	34
C(3)	0·1514 (6)	−0·3597 (6)	0·0395 (3)	31
C(4)	0·1957 (7)	−0·4676 (7)	0·0061 (3)	38
C(5)	0·1966 (8)	−0·5892 (7)	0·0379 (4)	46
C(6)	0·1496 (7)	−0·5997 (6)	0·1007 (4)	39
C(7)	0·1087 (6)	−0·4873 (6)	0·1337 (3)	31
C(11)	−0·1850 (7)	−0·1900 (6)	0·1101 (3)	34
C(12)	−0·2323 (7)	−0·1124 (7)	0·0512 (4)	42
C(13)	−0·3704 (8)	−0·1258 (8)	0·0188 (5)	55
C(14)	−0·4601 (9)	−0·212 (1)	0·0421 (6)	72
C(15)	−0·4143 (8)	−0·287 (1)	0·1019 (6)	76
C(16)	−0·2759 (8)	−0·2763 (9)	0·1346 (4)	56
C(21)	0·2416 (8)	−0·1356 (7)	0·2143 (4)	48
C(22)	0·276 (1)	−0·181 (1)	0·2822 (6)	79
C(23)	0·413 (1)	−0·156 (1)	0·3163 (7)	119
C(24)	0·510 (1)	−0·091 (2)	0·2834 (9)	145
C(25)	0·470 (1)	−0·044 (2)	0·2185 (8)	136
C(26)	0·3368 (9)	−0·065 (1)	0·1827 (5)	81

Fig. 2. Stereoscopic view of a part of two chains (without phenyl groups) probably linked *via* H bonds [(i) corresponds to the symmetry operation −x, ½+y, ½−z and (ii) to −x, −y, −z].

Table 2. *Bond distances* (Å) *and angles* (°)

Pb(1)—C(21)	2·140 (7)	O(3)—C(2)	1·269 (8)
Pb(1)—C(11)	2·146 (5)	O(4)—C(2)	1·227 (8)
Pb(1)—O(1)	2·374 (5)	N(1)—C(3)	1·332 (8)
Pb(1)—N(1)	2·452 (5)	N(1)—C(7)	1·342 (8)
Pb(1)—O(5)	2·472 (5)	C(1)—C(3)	1·517 (9)
Pb(1)—O(4ⁱ)	2·514 (5)	C(2)—C(7)	1·512 (8)
Pb(1)—O(3)	2·517 (5)	C(3)—C(4)	1·367 (9)
O(1)—C(1)	1·255 (8)	C(4)—C(5)	1·38 (1)
O(2)—C(1)	1·233 (8)	C(5)—C(6)	1·36 (1)
		C(6)—C(7)	1·390 (9)
C(21)—Pb(1)—C(11)	172·8 (3)	C(2ⁱ)—O(4ⁱ)—Pb(1)	132·2 (4)
C(21)—Pb(1)—O(1)	94·8 (2)	C(3)—N(1)—C(7)	120·3 (5)
C(21)—Pb(1)—N(1)	93·2 (2)	C(3)—N(1)—Pb(1)	118·4 (4)
C(21)—Pb(1)—O(5)	85·5 (2)	C(7)—N(1)—Pb(1)	121·3 (4)
C(21)—Pb(1)—O(4ⁱ)	86·1 (2)	O(2)—C(1)—O(1)	126·1 (6)
C(21)—Pb(1)—O(3)	89·4 (2)	O(2)—C(1)—C(3)	116·3 (6)
C(11)—Pb(1)—O(1)	90·8 (2)	O(1)—C(1)—C(3)	117·6 (5)
C(11)—Pb(1)—N(1)	93·2 (2)	O(4)—C(2)—O(3)	128·0 (6)
C(11)—Pb(1)—O(5)	91·6 (2)	O(4)—C(2)—C(7)	116·4 (6)
C(11)—Pb(1)—O(4ⁱ)	86·8 (2)	O(3)—C(2)—C(7)	115·6 (6)
C(11)—Pb(1)—O(3)	90·4 (2)	N(1)—C(3)—C(4)	121·3 (6)
O(1)—Pb(1)—N(1)	66·8 (2)	N(1)—C(3)—C(1)	114·6 (5)
O(1)—Pb(1)—O(5)	74·5 (2)	C(4)—C(3)—C(1)	124·1 (5)
O(1)—Pb(1)—O(4ⁱ)	152·7 (2)	C(3)—C(4)—C(5)	119·4 (6)
O(1)—Pb(1)—O(3)	131·3 (2)	C(6)—C(5)—C(4)	119·1 (6)
N(1)—Pb(1)—O(5)	141·0 (2)	C(5)—C(6)—C(7)	119·8 (6)
N(1)—Pb(1)—O(4ⁱ)	140·5 (2)	N(1)—C(7)—C(6)	120·0 (6)
N(1)—Pb(1)—O(3)	64·6 (2)	N(1)—C(7)—C(2)	116·3 (5)
O(5)—Pb(1)—O(4ⁱ)	78·4 (2)	C(6)—C(7)—C(2)	123·6 (6)
O(3)—Pb(1)—O(5)	154·1 (2)	C(16)—C(11)—Pb(1)	122·5 (5)
O(3)—Pb(1)—O(4ⁱ)	75·9 (2)	C(12)—C(11)—Pb(1)	117·5 (4)
C(1)—O(1)—Pb(1)	122·4 (4)	C(26)—C(21)—Pb(1)	118·2 (6)
C(2)—O(3)—Pb(1)	122·1 (4)	C(22)—C(21)—Pb(1)	120·4 (6)

The C—C bond lengths and C—C—C angles in the phenyl groups range from 1·34 (2) to 1·40 (2) Å [mean: 1·37 (2) Å] and from 117·4 (9) to 122 (1)° [mean: 120 (1)°], respectively.

methyltin and di-*n*-butyltin compounds are also seven-coordinated, but dimeric (Huber, Hoffmann, Preut & Gielen, 1988). The structure of 2,6-pyridinedicarboxylic acid is known (Takusagawa, Hirotsu & Shimada, 1973).

probably connected *via* H bonds [O(2ⁱⁱ)···O(5) and O(2)···O(5ⁱⁱ) = 2·696 (7) Å] (phenyl groups are omitted for clarity). Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Selected bond lengths and angles are given in Table 2.

Related literature. The structure is very close to that of the analogous diphenyltin 2,6-pyridinedicarboxylate hydrate (Gielen *et al.*, 1987). The appropriate di-

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