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Acta Cryst. (1986). **C42**, 657–658

(2-Furoato)trimethyllead

BY HANS PREUT, PETRA RÖHM AND FRIEDO HUBER

Fachbereich Chemie, Universität Dortmund, Postfach 500 500, D-4600 Dortmund 50, Federal Republic of Germany

(Received 1 August 1985; accepted 24 December 1985)

Abstract. [Pb(C₅H₃O₃)(CH₃)₃], $M_r = 363.4$, monoclinic, $P2_1/n$, $a = 10.246$ (9), $b = 10.297$ (9), $c = 10.651$ (8) Å, $\beta = 106.19$ (5)°, $V = 1079$ Å³, $Z = 4$, $D_x = 2.237$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 15.4$ mm⁻¹, $F(000) = 664$, $T = 291$ K, final $R = 0.039$ for 1077 unique observed [$I > 3\sigma(I)$] X-ray diffractometer data. In the crystalline state nearly planar (CH₃)₃Pb groups are linked by bridging carboxylate groups [Pb–O(1) 2.353 (9), Pb–O(2) 3.17 (1), Pb–O(2') 2.534 (9) Å] to form infinite chains parallel to **b**. An additional interaction between the O atom of the furan ring and a Pb atom is observed [Pb...O 3.55 (1) Å].

Introduction. Structures of organo Group IV element derivatives of carboxylic acids $R'\text{COOH}$ (R' = heterocyclic remainder) are interesting, since there arises the possibility of coordinative interaction between the central atom, e.g. Sn or Pb, and the heteroatom in R' , e.g. N or O. Recently, Harrison & Philips (1979) reported the structure of Me₃SnO₂CC₅H₄N.H₂O, but the heteroatom in the pyridine group is involved in hydrogen bonding, and there is no interaction with Sn. In the title compound on the other hand this is not the case and an interaction between the O atom of the furan ring and the Pb atom is observed.

Experimental. Colourless, fibrous crystals were prepared from equimolar amounts of Me₃PbOH and 2-furoic acid in methanol. The compound was recrystallized from methanol/di-*n*-butyl ether (1/1). Crystal size 0.14 × 0.30 × 0.18 mm, $\omega/2\theta$ scan, ω -scan speed 2.5° min⁻¹, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit using 25 reflections with $2\theta_{\text{max}} = 26.6^\circ$; intensities of four standard reflec-

tions recorded every 2.5 h showed decay of 50% during the course of data collection; 4127 reflections, $1^\circ \leq \theta \leq 25^\circ$, $-12 \leq h \leq 12$, $-12 \leq k \leq 12$, $0 \leq l \leq 12$; after averaging ($R_{\text{int}} = 0.026$) 2015 unique reflections, 1077 with $I > 3\sigma(I)$, Lorentz–polarization correction, absorption correction *via* ψ scans (max./min. correction: 1.00/0.58), and decay correction; systematic absences showed space group to be $P2_1/n$; structure solution by Patterson methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for non-H atoms, H atoms in geometrically calculated positions (C–H 0.95 Å) and refined with one common isotropic temperature factor for these atoms; 110 variables, $w^{-1} = [\sigma^2(I) + (0.1F_o^2)^2]^{1/2}$, $S = 0.79$, $R = 0.039$, $wR = 0.047$, max. $\Delta/\sigma = 0.13$, largest peak in final ΔF map = ± 2.0 (3) e Å⁻³ near Pb; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *Enraf–Nonius Structure Determination Package* (Frenz, 1981), *ORTEPII* (Johnson, 1976).

Discussion. The structure of the title compound with the numbering scheme is shown in Fig. 1 and a stereoscopic view of the unit cell is shown in Fig. 2 (*ORTEPII*). Atomic parameters are given in Table 1,* selected bond lengths and angles in Table 2. In the crystalline state Pb(CH₃)₃ groups are linked *via* bridging carboxylate groups of the 2-furoate ligands to form an infinite chain similar to that observed by

* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42732 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Sheldrick & Taylor (1975) for acetatotrimethyllead. The atoms directly bound to Pb form a distorted trigonal bipyramid with the three C(methyl) atoms in the equatorial plane and the two O atoms in the apical positions. (The two Pb—O distances are significantly different, Table 2.) The distances Pb(1)···O(2) of 3.17 (1) Å and Pb(1)···O(3ⁱ) of 3.55 (1) Å are all shorter than the sum of the van der Waals radii of 3.82 Å (Bondi, 1964), and one can infer the occurrence of a weak coordination interaction between the central atom and O(2), *i.e.* weak chelation by the carboxylate group, involving the short Pb(1)—O(1) bond, and also between Pb and O(3ⁱ) *i.e.* weak coordination by O of the furan ring (Fig. 1). These interactions appear to lead to an increase in the C(2)—Pb(1)—C(3) angle and to cause a bending of the two axial bonds with the result that the bond angles C(1)—Pb(1)—O(1) and C(1)—Pb(1)—O(2ⁱ) are markedly smaller than 90°. Pb(1) is 0.10 (1) Å above the plane through C(1), C(2), and C(3). The distances Pb—C, Pb—O(1) and Pb—O(2) correspond to the analogous values found in acetatotrimethyllead (Sheldrick & Taylor, 1975). The bond angles and bond distances in the 2-furoate ligand correspond essentially to those reported for 2-furoic acid (Hudson, 1962). The interplanar angle between the furan ring and the carboxylate group, however, is 8.1 (3)° compared with

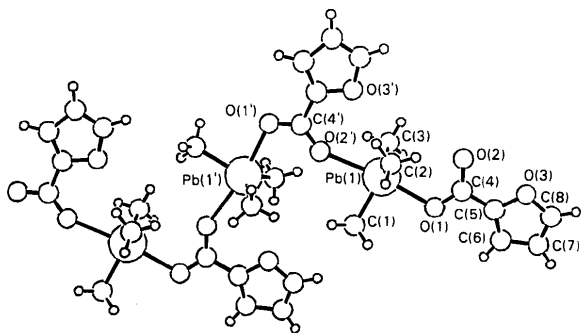


Fig. 1. General view of the molecule, showing the atom-numbering scheme [(i) corresponds to the symmetry operation: $1.5 - x$, $-\frac{1}{2} + y$, $\frac{1}{2} - z$]. The chain runs parallel to *b*.

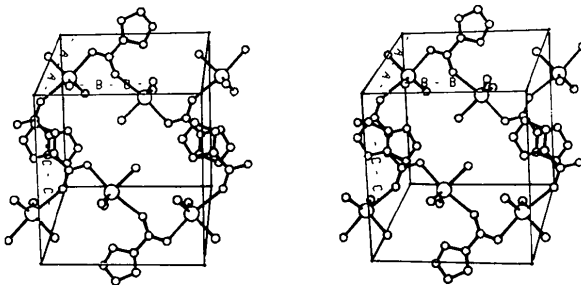


Fig. 2. Stereoscopic view of the unit cell.

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

$$U_{\text{eq}} = \frac{1}{24\pi^2} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Pb(1)	0.77800 (5)	0.08692 (5)	0.19723 (5)	58
O(1)	0.8018 (9)	0.2405 (9)	0.0416 (9)	70
O(2)	0.762 (1)	0.394 (1)	0.1711 (9)	81
O(3)	0.802 (1)	0.5784 (9)	-0.0011 (9)	100
C(1)	0.833 (2)	-0.061 (2)	0.076 (2)	105
C(2)	0.941 (2)	0.161 (2)	0.359 (2)	94
C(3)	0.562 (2)	0.122 (2)	0.162 (2)	90
C(4)	0.786 (1)	0.355 (1)	0.068 (1)	62
C(5)	0.799 (1)	0.449 (1)	-0.031 (1)	60
C(6)	0.811 (2)	0.438 (2)	-0.150 (2)	85
C(7)	0.816 (2)	0.565 (2)	-0.201 (2)	87
C(8)	0.810 (3)	0.641 (2)	-0.108 (2)	117

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

Pb(1)—C(1)	2.17 (2)	C(4)—O(1)	1.23 (2)
Pb(1)—C(2)	2.17 (2)	C(4)—O(2)	1.26 (2)
Pb(1)—C(3)	2.17 (2)	C(4)—C(5)	1.46 (2)
Pb(1)—O(1)	2.353 (9)	C(5)—C(6)	1.31 (2)
Pb(1)—O(2 ⁱ)	2.534 (9)	C(5)—O(3)	1.37 (2)
Pb(1)—O(2)	3.17 (1)	C(6)—C(7)	1.43 (2)
Pb(1)—O(3 ⁱ)	3.55 (1)	C(7)—C(8)	1.28 (2)
		C(8)—O(3)	1.34 (2)
C(1)—Pb(1)—C(2)	116.4 (6)	Pb(1)—O(2 ⁱ)—C(4 ⁱ)	146.4 (9)
C(1)—Pb(1)—C(3)	115.8 (6)	O(1)—C(4)—O(2)	125 (1)
C(1)—Pb(1)—O(1)	87.5 (5)	O(1)—C(4)—C(5)	115 (1)
C(1)—Pb(1)—O(2 ⁱ)	83.6 (5)	O(2)—C(4)—C(5)	120 (1)
C(2)—Pb(1)—C(3)	127.0 (6)	C(4)—C(5)—O(3)	119 (1)
C(2)—Pb(1)—O(1)	96.6 (5)	C(4)—C(5)—C(6)	134 (1)
C(2)—Pb(1)—O(2)	92.5 (5)	O(3)—C(5)—C(6)	108 (1)
C(3)—Pb(1)—O(1)	93.9 (5)	C(5)—C(6)—C(7)	109 (1)
C(3)—Pb(1)—O(2 ⁱ)	84.9 (5)	C(6)—C(7)—C(8)	104 (1)
O(1)—Pb(1)—O(2 ⁱ)	169.4 (3)	C(7)—C(8)—O(3)	113 (2)
O(1)—Pb(1)—O(2)	116.1 (8)	C(8)—O(3)—C(5)	106 (1)

(i) corresponds to the symmetry operation: $1.5 - x$, $-\frac{1}{2} + y$, $\frac{1}{2} - z$.

2° for the appropriate angle in the free acid (Hudson, 1962). There are no short intermolecular contacts between the chains.

Financial support from the Ministerium für Wissenschaft und Forschung des Landes Nordrhein-Westfalen is gratefully acknowledged.

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