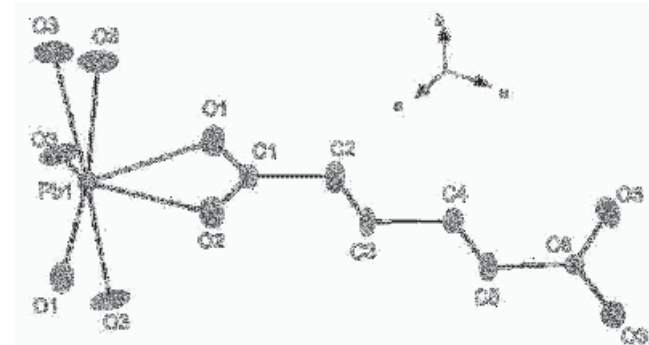


Crystal structure of orthorhombic-catena-((μ_5 -adipato- $\kappa^4 O, O':O, O'$)-lead(II)), $C_6H_8O_4Pb$

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Abstract

$C_6H_8O_4Pb$, orthorhombic, $Pnma$ (no. 62), $a = 21.927(2)$ Å, $b = 7.2139(7)$ Å, $c = 4.7357(5)$ Å, $V = 749.1$ Å³, $Z = 4$, $R_{gt}(F) = 0.0429$, $wR_{ref}(F^2) = 0.1149$, $T = 296$ K.

Table 1. Data collection and handling.

Crystal:	colourless prisms, size 0.05×0.05×0.20 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	224.83 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7214, 1001
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 953
$N(param)_{refined}$:	65
Programs:	SHELX [5]

Source of material

A mixture of adipic acid ($C_6H_{10}O_4$) (0.05 g), $Pb(NO_3)_2$ (0.05 g), KOH (0.05 g) and H_2O (15 ml) was heated at 448 K for 7 d in a sealed 25 ml Teflon-lined stainless steel vessel under autogenous pressure. After cooling to room temperature at a rate of 5°C h⁻¹, colourless prismatic crystals were obtained in low yield.

Experimental details

The H atoms were positioned geometrically and refined using a riding model, with C–H = 0.97 Å and $U_{iso}(H) = 1.2 U_{eq}(C)$.

Discussion

Recently, some adipate compounds have been reported which exhibit diverse structure types, such as $\{[Tb_2(C_6H_8O_4)_3(H_2O)_4] \cdot 0.25 H_2O\}_n$ [1], $[Cu_2(C_6H_8O_4)_2(C_3H_7NO)_2]_n$ [2] and $La_2(C_2O_4)_2(C_6H_8O_4)(H_2O)_2$ [3]. Thirumurugan *et al.* have been reported a series of Pb aliphatic dicarboxylates [4], including two polymorphs of $Pb(C_6H_8O_4)$ with the space group $P2_1/c$. In this paper, we report the orthorhombic structure of $Pb(C_6H_8O_4)$ with the space group $Pnma$. The asymmetric unit of title structure contains one half Pb^{2+} ion and one half adipate anion both located on a mirror plane. The Pb atom is seven-coordinated by oxygen atoms

from five different adipate anions, with the Pb–O bond distances ranging from 2.392(12) to 2.686(8) Å. Six of the oxygens have μ_3 connections linking each Pb with four other Pb atoms giving rise to an infinite 2D Pb–O–Pb layer, which are further interconnected by μ_5 -bridging adipate ligands to form the 3D framework.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(3A)	8d	0.7561	0.3585	0.5118	0.048
H(3B)	8d	0.7561	0.1415	0.5118	0.048
H(2A)	8d	0.7013	0.3584	0.0905	0.058
H(2B)	8d	0.7013	0.1416	0.0905	0.058
H(5A)	8d	0.8731	0.3586	0.5269	0.050
H(5B)	8d	0.8731	0.1414	0.5269	0.050
H(4A)	8d	0.8152	0.3585	0.1048	0.045
H(4B)	8d	0.8152	0.1415	0.1048	0.045

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Pb(1)	4 <i>c</i>	0.53539(2)	$\frac{1}{4}$	0.6993(1)	0.0227(3)	0.0304(3)	0.0318(4)	0	0.0018(1)	0
O(1)	4 <i>c</i>	0.5902(5)	$\frac{1}{4}$	0.247(2)	0.028(5)	0.086(9)	0.034(4)	0	−0.008(4)	0
C(1)	4 <i>c</i>	0.6412(6)	$\frac{1}{4}$	0.370(3)	0.024(6)	0.047(7)	0.025(5)	0	0.000(5)	0
C(3)	4 <i>c</i>	0.7568(5)	$\frac{1}{4}$	0.391(3)	0.017(5)	0.065(9)	0.037(6)	0	0.008(5)	0
O(2)	4 <i>c</i>	0.6435(6)	$\frac{1}{4}$	0.630(3)	0.046(4)	0.078(5)	0.049(4)	0	0.005(4)	0
C(2)	4 <i>c</i>	0.7003(7)	$\frac{1}{4}$	0.212(3)	0.023(6)	0.08(1)	0.040(8)	0	0.004(5)	0
C(5)	4 <i>c</i>	0.8731(6)	$\frac{1}{4}$	0.406(3)	0.021(5)	0.068(9)	0.035(6)	0	0.005(5)	0
C(4)	4 <i>c</i>	0.8147(7)	$\frac{1}{4}$	0.226(3)	0.025(7)	0.044(7)	0.043(7)	0	0.005(5)	0
O(3)	8 <i>d</i>	0.9544(4)	0.100(1)	0.160(2)	0.075(5)	0.025(4)	0.076(6)	0.012(4)	0.039(5)	0.004(4)
C(6)	4 <i>c</i>	0.9305(6)	$\frac{1}{4}$	0.231(3)	0.025(5)	0.027(5)	0.027(5)	0	0.002(4)	0

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