metal-organic papers

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Key indicators

Single-crystal X-ray study T = 295 KMean $\sigma(\text{C}-\text{C}) = 0.009 \text{ Å}$ R factor = 0.030 wR factor = 0.075 Data-to-parameter ratio = 16.8

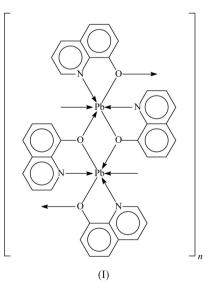
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Bis(quinolin-8-olato- $\kappa^2 N$,O)lead(II)

The Pb^{II} atom in the title compound, $[Pb(C_9H_6NO)_2]$, is *N*,*O*-chelated by two organic ligands, and two molecules are linked across a centre of inversion to form a dinuclear entity. These dinuclear entities are linked by somewhat weaker Pb···O bonds into a chain. There are two independent molecules in the asymmetric unit with similar geometry around the Pb atoms, which have a pseudo-pentagonal–bipyramidal coordination with one of the axial positions occupied by the Pb lone pair. Some distances involving the Pb atoms are significantly different between the two molecules.

Comment

The 8-hydroxyquinoline derivative of lead(II) is known to be a sparingly soluble compound (Packter & Chauhan, 1973). The compound is usually synthesized by precipitating lead(II) ions with 8-hydroxyquinoline, but it can be synthesized using a dry method (Saxena, 2002). The compound has been examined for a number of physical properties, *e.g.* thermal behaviour (Juiz *et al.*, 1997) and enthalpy of formation (Ribeiro da Silva *et al.*, 1994). Its synthesis by a hydrothermal method has allowed the compound to be characterized by crystallography.



The Pb^{II} atom in the title compound, (I) (Figs. 1 and 2), is N,O-chelated by two 8-hydroxyquinolinate anions, and two molecules are linked across a centre of inversion to form a dinuclear unit. The geometry of the Pb atom in each of the two independent molecules is pseudo-pentagonal-bipyramidal, with one of the axial positions occupied by the Pb lone pair. Adjacent dinuclear units are linked into a chain by somewhat weaker Pb···O interactions (Fig. 3).

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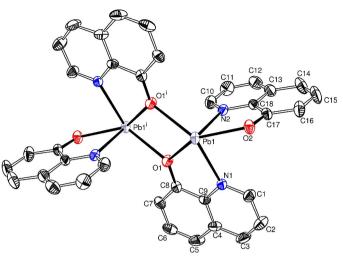


Figure 1

A plot of one of the independent centrosymmetric molecules of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $\frac{1}{2} - x$, $\frac{3}{2} - y$, 1 - z.]

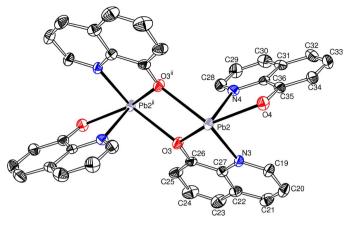


Figure 2

A plot of the second independent centrosymmetric molecule of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (ii) -x, -y, 1 - z.]

Experimental

Lead acetate trihydrate (0.189 g, 0.5 mmol) and 8-hydroxyquinoline (0.145 g, 1 mmol) were mixed in water (10 ml) and the mixture sealed in a 23 ml Teflon-lined stainless steel Parr bomb, which was then heated at 413 K for 72 h. The bomb was then cooled to room temperature at 10 K h⁻¹ to give pale-yellow columnar crystals of (I) in *ca* 45% yield.

Crystal data

$[Pb(C_9H_6NO)_2]$	$D_x = 2.138 \text{ Mg m}^{-3}$
$M_r = 495.59$	Mo K α radiation
Monoclinic, C_2/c	Cell parameters from 4994
a = 35.937 (2) Å b = 8.3365 (4) Å	reflections $\theta = 2.5-27.4^{\circ}$
c = 25.432 (1) Å	$\mu = 10.97 \text{ mm}^{-1}$
$\beta = 126.076 (1)^{\circ}$	T = 295 (2) K
V = 6158.1 (5) Å ³	Column, yellow
Z = 16	$0.18 \times 0.17 \times 0.15 \text{ mm}$
Data collection	

6956 independent reflections

 $\begin{aligned} R_{\rm int} &= 0.028\\ \theta_{\rm max} &= 27.5^\circ \end{aligned}$

 $h = -41 \rightarrow 46$

 $k=-10\rightarrow 10$

 $l = -32 \rightarrow 31$

4910 reflections with $I > 2\sigma(I)$

Bruker APEX CCD area-detector diffractometer φ and ω scans Absorption correction: multi-scan (*SADABS*; Bruker, 2002) $T_{min} = 0.077, T_{max} = 0.193$ 20 725 measured reflections

Refinement

 Refinement on F^2 H-atom parameters constrained

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $w = 1/[\sigma^2(F_o^2) + (0.0333P)^2]$
 $wR(F^2) = 0.075$ where $P = (F_o^2 + 2F_c^2)/3$

 S = 1.00 $(\Delta/\sigma)_{max} = 0.001$

 6956 reflections
 $\Delta\rho_{max} = 1.07$ e Å⁻³

 415 parameters
 $\Delta\rho_{min} = -1.12$ e Å⁻³

H atoms were positioned geometrically, with C-H 0.93 Å, and were included in the refinement in the riding-model approximation, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The final difference Fourier map had a large peak and a hole, each at about 1 Å from Pb1, but was otherwise featureless.

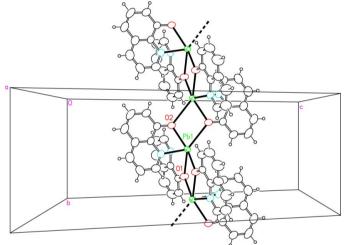


Figure 3

A view showing the formation of a chain running along **b** by association of the dimeric units through weak $Pb \cdots O$ bonds.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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