Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(hydrogen 5-nitroisophthalato)(1,10-phenanthroline)lead(II) monohydrate

Hong-Ping Xiao* and Ji-Xin Yuan

School of Chemistry and Materials Science, Wenzhou Normal College, Zhejiang, Wenzhou 325027, People's Republic of China

Correspondence e-mail: hp_xiao@wznc.zj.cn

Received 13 September 2004
Accepted 17 September 2004
Online 30 September 2004

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.021
wR factor = 0.054
Data-to-parameter ratio = 11.9

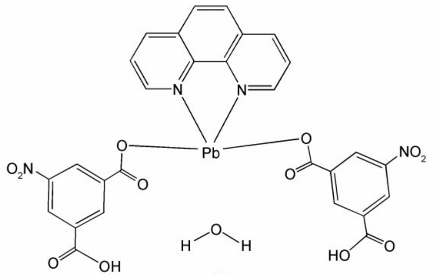
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

© 2004 International Union of Crystallography
Printed in Great Britain – all rights reserved

In the title compound, $[Pb(C_8H_4NO_6)_2(C_{12}H_8N_2)]\cdot H_2O$, the lead(II) ion lies on a twofold axis and exhibits an approximately tetrahedral configuration. The water molecule also lies on a twofold rotation axis. O—H···O hydrogen bonds and C—H···π interactions are involved in the formation of two-dimensional network structures which are interlinked by C—H···O hydrogen bonds.

Comment

Bi- or multidentate ligands containing carboxyl groups are often used to coordinate to metal centers to generate interesting coordination polymers (Hu *et al.*, 2004; Sun *et al.*, 2003; Yaghi *et al.*, 1998). The lead(II) ion usually shows a different coordination chemistry, due to the presence of the lone-pair electrons (Foreman, 2000; Yuan *et al.*, 2004). Hence, it is of interest to study the crystal structures of lead(II) carboxylates. We report here the hydrothermal synthesis and crystal structure of a mononuclear compound, namely bis(hydrogen 5-nitroisophthalato)(1,10-phenanthroline)lead(II) monohydrate, (I).



(I)

In (I), the coordination geometry of the Pb^{II} atom is best described as highly distorted tetrahedral, made up of two N atoms of a phenanthroline ligand and two O atoms from two hydrogen 5-nitroisophthalate anions (Fig. 1). The crystal structure analysis shows that a crystallographic twofold symmetry axis passes through the lead(II) ion and bisects the $C13—C13^i$ and $C14—C14^i$ bonds [symmetry code: (i) $\frac{1}{2} - x, \frac{3}{2} - y, z$]. The 1,10-phenanthroline system is almost perpendicular to the benzene ring of the 5-nitroisophthalate anion, the dihedral angle between the planes being 83.80 (2)°. The water molecule also lies on a twofold rotation axis.

$O3—H3\cdots O2^{ii}$ and $O7—H7A\cdots O4^{ii}$ [symmetry code: (ii) $1 - x, 1 - y, 2 - z$] intermolecular hydrogen bonds (Table 2) link the molecules into a two-dimensional network structure. This network structure is further stabilized by π–π stacking interactions between the N1-pyridine rings of the 1,10-phenanthroline moieties at (*x, y, z*) and ($\frac{1}{2} - x, \frac{1}{2} - y, z$)

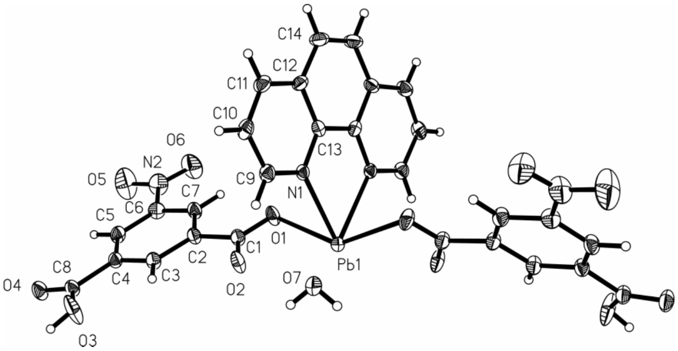


Figure 1
The coordination environment of the Pb atom in (I), with the atom numbering, showing displacement ellipsoids at the 50% probability level. Unlabelled atoms are related to labelled atoms by $\frac{1}{2} - x, \frac{3}{2} - y, z$.

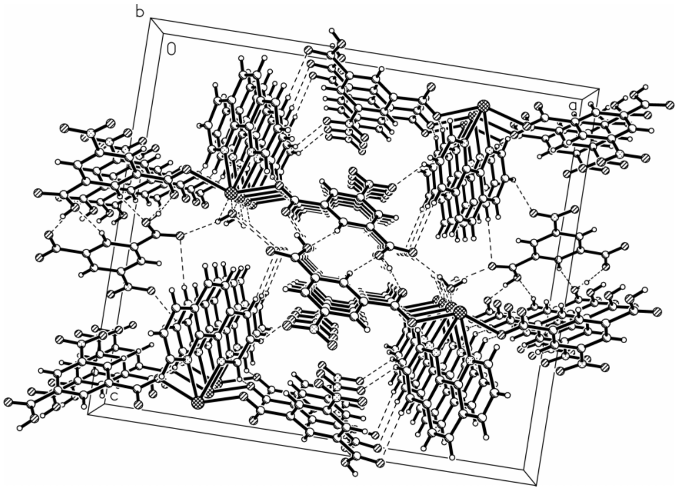


Figure 2
The three-dimensional network in (I), formed by hydrogen bonds and π–π stacking interactions.

[centroid–centroid distance = 3.490 (2) Å]. The two-dimensional networks are interlinked by C—H⋯O interactions, as shown in Fig. 2.

Experimental

The title compound was synthesized by a hydrothermal method from a mixture of 5-nitroisophthalic acid (0.5 mmol), $Pb(NO_3)_2{\cdot}4H_2O$ (0.5 mmol), 1,10-phenanthroline (1.0 mol) and water (10.0 ml) in a 15 ml Teflon-lined stainless steel reactor. The solution was heated at 403 K for 5 d and then cooled slowly to room temperature; colorless crystals of (I) were collected for X-ray analysis.

Crystal data

$[Pb(C_8H_4NO_6)_2(C_{12}H_8N_2)]{\cdot}H_2O$
$M_r = 825.65$
Orthorhombic, *Pccn*
$a = 22.6083$ (17) Å
$b = 6.2232$ (5) Å
$c = 20.0394$ (15) Å
$V = 2819.5$ (4) Å^3
$Z = 4$
$D_x = 1.945$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 853 reflections
$\theta = 2.3$–$23.2°$
$\mu = 6.06$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.26 × 0.13 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.302$, $T_{max} = 0.530$
13880 measured reflections
2543 independent reflections
2003 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.2°$
$h = -20 \rightarrow 27$
$k = -7 \rightarrow 7$
$l = -23 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.054$
$S = 1.04$
2543 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 1.2381P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pb1—N1	2.434 (2)	Pb1—O1	2.554 (2)
N1—Pb1—N1^i	68.27 (11)	N1—Pb1—O1	74.96 (8)
N1—Pb1—O1^i	77.21 (8)	O1^i—Pb1—O1	146.23 (11)

Symmetry code: (i) $\frac{1}{2} - x, \frac{3}{2} - y, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O7—H7*A*⋯O4^{ii}	0.81 (2)	2.09 (2)	2.860 (3)	157 (4)
O3—H3⋯O2^{ii}	0.82	1.73	2.506 (3)	156
C3—H3*A*⋯O3	0.93	2.34	2.676 (4)	101
C3—H3*A*⋯O3^{ii}	0.93	2.27	3.158 (4)	160
C9—H9⋯O1^{iii}	0.93	2.45	3.099 (4)	127
C10—H10⋯O5^{iv}	0.93	2.53	3.292 (4)	140
C11—H11⋯O4^{v}	0.93	2.55	3.390 (4)	150

Symmetry codes: (ii) $1 - x, 1 - y, 2 - z$; (iii) $x, y - 1, z$; (iv) $1 - x, y - \frac{3}{2}, \frac{3}{2} - z$; (v) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$.

The water H atom was located and refined with an O—H distance restraint [O—H = 0.82 (2) Å] and with $U_{iso}(H) = 1.5U_{eq}(O)$. The other H atoms were placed in calculated positions (C—H = 0.93 Å) and included in the refinement in the riding-model approximation, with $U_{iso}(H)$ values set at $1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Wenzhou Science and Technology project of China (No. S2003A008).

References

Bruker (2000). *SMART* (Version 5.618), *SAINT* (Version 6.02*a*), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Foreman, M. R. St J., Gelbrich, T., Hursthouse, M. B. & Plater, M. J. (2000). *Inorg. Chem. Commun.* **3**, 234–238.

Hu, M. L., Xiao, H. P. & Yuan, J. X. (2004). *Acta Cryst*. C**60**, m112–m113.

Sun, D. F., Cao, R., Sun, Y. Q., Bi, W. H., Yuan, D. Q., Shi, Q. & Li, X. (2003). *Chem. Commun.* pp. 1528–1529.

Yaghi, O. M., Li, H., Davis, C., Richardson, D. & Groy, T. (1998). *Acc. Chem. Res.* **31**, 474–484.

Yuan, Y. P., Mao, J. G. & Song, J. L. (2004). *J. Solid State Chem.* **177**, 922–927.