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Bis(μ-hydrogen 5-sulfosalicylate)bis[aqua(1,10-phenanthroline)lead(II)]

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.021
wR factor = 0.053
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.

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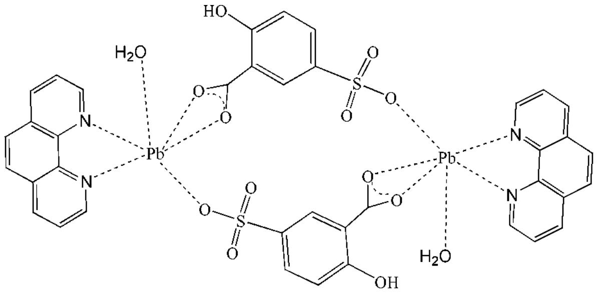
In the title compound, $[Pb_2(C_7H_4O_6S)_2(C_{12}H_8N_2)_2(H_2O)_2]$, two [Pb(phen)($H_2O$)] moieties are connected together by two Hssa anions, resulting in a centrosymmetric dimer (phen is 1,10-phenanthroline and Hssa is the hydrogen 5-sulfosalicylate dianion). Each Pb^{II} cation is surrounded by O atoms from a carboxylate group in a chelating fashion, a sulfonate group in a monodentate mode, a water molecule, and two N atoms from a phen ligand, forming a distorted PbO_4N_2 octahedron.

Comment

In recent years, increasing attention has been focused on 5-sulfosalicylic acid (H_3ssa) and its metal complexes, owing to their biological activity, such as anti-ulcer, antimicrobial, antifungal and anti-inflammatory activities (Marzotto *et al.*, 2001). However, only a few of these complexes have been structurally documented to date, for example, trimeric $[Cu_3(ssa)_2(bpy)_2(H_2O)_4]\cdot 4H_2O$ (bpy is 2,2′-bipyridine; Wang *et al.*, 2004), one-dimensional polymeric [Zn(Hssa)(phen)($H_2O)_2$] (Chen *et al.*, 2003), two-dimensional polymeric [Eu(H_2ssa)$_2$($H_2O)_5$] (Starynowicz, 2000) and three-dimensional polymeric $[Ag_3(\mu_4\text{-hmt})(\mu\text{-Hssa})(H_2O)]NO_3\cdot 3H_2O$ (hmt is hexamethylenetetramine; Zheng *et al.*, 2003). To extend this research, we report here the crystal structure of the title compound, [Pb_2(Hssa)$_2$(phen)$_2$($H_2O)_2$], (I).



(I)

In the centrosymmetric dimeric molecule of (I), each Pb^{II} cation is coordinated by two O atoms from the carboxylate group of an Hssa anion, with a typical Pb—O(carboxylate) distance range [2.354 (3)–2.684 (3) Å; Foreman *et al.*, 2000], one sulfonate O atom from another Hssa anion with a Pb—O distance of 2.742 (3) Å, one water molecule with a Pb—O distance of 2.530 (3) Å, and two N atoms from one phen with a Pb—N distances 2.577 (3) and 2.599 (3) Å, forming a distorted PbO_4N_2 octahedron (Fig. 1). The O1/O2/N1/N2 basal plane is seriously distorted, with a mean deviation of 0.44 Å; this can be attributed to the absence of crystal field-stabilization energy effects of Pb^{2+} cations (Foreman *et al.*, 2000). The

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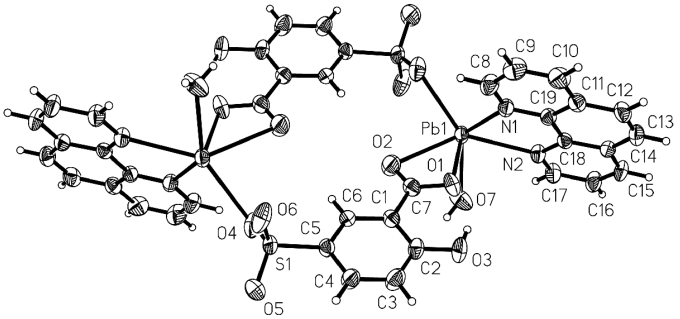

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

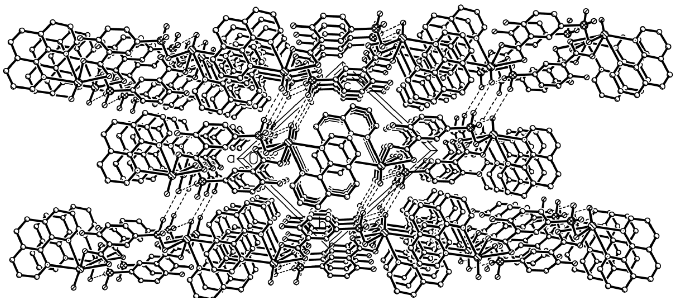

Figure 2
The three-dimensional network formed in (I) *via* hydrogen-bonding interactions (dashed lines). H atoms have been omitted.

apical positions are occupied by one water molecule (O7) and sulfonate atom O4^i [symmetry code: (i) $2-x$, $-y$, $-z$]. Moreover, each pair of Pb^{II} cations with the same coordination environment is bridged by two Hssa anions, forming a centrosymmetric dinuclear 16-membered ring, with a Pb1$\cdots$Pb1^i separation of 8.9678 (10) Å.

There is an intramolecular O3—H3$\cdots$O1 hydrogen bond in the Hssa ligand. In addition, there are intermolecular O—H$\cdots$O hydrogen bonds involving the water ligand O7 (Table 2), resulting in the formation of a three-dimensional network (Fig. 2).

Experimental

The title compound was synthesized using the hydrothermal method, from a mixture of 5-sulfosalicylic acid (1 mmol, 0.22 g), $PbCl_2\cdot4H_2O$ (1 mmol, 0.35 g), 1,10-phenanthroline (3 mmol, 0.54 g) and water (20 ml) in a 30 ml Teflon-lined stainless steel reactor. The solution was heated to 415 K for 3 d. After slow cooling of the reaction system to room temperature, the colourless block crystals of (I) were collected and washed with distilled water (yield 64%).

Crystal data

$[Pb_2(C_7H_4O_6S)_2(C_{12}H_8N_2)_2(H_2O)_2]$
M_r = 1243.14
Triclinic, $P\bar{1}$
a = 8.2520 (11) Å
b = 10.2384 (13) Å
c = 11.3422 (15) Å
α = 84.979 (2)°
β = 84.407 (2)°
γ = 78.970 (2)°
V = 933.8 (2) Å^3
Z = 1
D_x = 2.211 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3935 reflections
θ = 2.5–25.2°
μ = 9.19 mm^{-1}
T = 298 (2) K
Block, colourless
0.32 × 0.27 × 0.20 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.073, T_{max} = 0.161
4971 measured reflections
3307 independent reflections
3126 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.2°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -5 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.053
S = 1.05
3307 reflections
279 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0189 (6)

Table 1
Selected geometric parameters (Å, °).

Pb1—O1	2.354 (3)	Pb1—N2	2.599 (3)
Pb1—O7	2.530 (3)	Pb1—O2	2.684 (3)
Pb1—N1	2.577 (3)	Pb1—O4^i	2.742 (3)
O1—Pb1—O7	75.64 (11)	N1—Pb1—O2	108.80 (11)
O1—Pb1—N1	75.07 (10)	N2—Pb1—O2	138.34 (10)
O7—Pb1—N1	131.98 (12)	O1—Pb1—O4^i	102.56 (11)
O1—Pb1—N2	88.59 (10)	O7—Pb1—O4^i	147.10 (11)
O7—Pb1—N2	77.80 (11)	N1—Pb1—O4^i	76.65 (9)
N1—Pb1—N2	64.31 (10)	N2—Pb1—O4^i	135.05 (9)
O1—Pb1—O2	51.60 (10)	O2—Pb1—O4^i	73.83 (10)
O7—Pb1—O2	80.31 (11)		

Symmetry code: (i) $2-x$, $-y$, $-z$.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3$\cdots$O1	0.82	1.81	2.542 (4)	147
O7—H7$A\cdots$O5^{ii}	0.810 (18)	1.844 (19)	2.653 (5)	176 (5)
O7—H7$B\cdots$O6^{iii}	0.820 (18)	1.98 (2)	2.733 (5)	152 (4)

Symmetry codes: (ii) $1-x$, $-y$, $-z$; (iii) x, y, $1+z$.

The water H atoms were located in difference-density maps and refined with O—H and H$\cdots$H distances restrained to 0.82 (2) and 1.39 (1) Å, respectively, and with U_{iso} = 0.035 Å^2. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.93 (C—H) and 0.82 Å (O—H), with U_{iso}(H) = $1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

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