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Poly[(1,10-phenanthroline-κ^2N,N')lead(II) μ_4-(4-sulfonatobenzoato-$\kappa^4O,O':O'',O'''$ hydrate]

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Key indicators

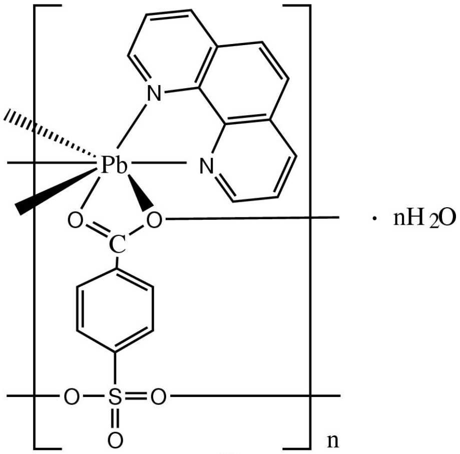
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
H-atom completeness 86%
Disorder in solvent or counterion
R factor = 0.032
wR factor = 0.071
Data-to-parameter ratio = 14.9

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

In the title polymeric compound, $[\mathrm{Pb}(\mathrm{C}_{12}\mathrm{H}_8\mathrm{N}_2)(\mathrm{C}_7\mathrm{H}_4\mathrm{O}_5\mathrm{S})]_n\cdot n\mathrm{H}_2\mathrm{O}$, the seven-coordinate Pb$^{\rm II}$ atom is surrounded by one 1,10-phenanthroline (phen) and four 4-sulfonatobenzoate (sb) ligands in a hemidirected coordination geometry. Both the sulfonyl and carboxy groups of the sb ligand bridge the neighboring Pb$^{\rm II}$ atoms to form a two-dimensional sheet structure.

Comment

The sulfonate group exhibits a different coordination mode compared to the carboxylate group (Zheng *et al.*, 2003; Wang *et al.*, 2004; Starynowicz, 2000). 4-Sulfonatobenzoate (sb), which combines sulfonate and carboxylate groups, may be used as a bridging ligand to construct novel complex architectures. A search of the Cambridge Structure Database (November 2004 update; Allen, 2002) shows that the structures of five sb metal complexes have been reported, the sulfonate group adopting a monodentate mode (Yuan *et al.*, 2001; Fan, Xiao, Zhang & Zhu, L.-G., 2004; Fan, Xiao, Zhang, Cai & Zhu, 2004) or a *syn–syn* bridge mode (Xiong *et al.*, 2001) in these complexes. We report here the crystal structure of the title polymeric Pb$^{\rm II}$ complex, (I), which exhibits a different coordination mode for the sulfonate group from those reported previously.



(I)

The structure of a segment of (I) is presented in Fig. 1 to show the coordination geometry around the Pb$^{\rm II}$ atom. Each seven-coordinate Pb$^{\rm II}$ atom is surrounded by one 1,10-phenanthroline (phen) and four sb ligands. While the carboxylate group of the sb ligand chelates a Pb$^{\rm II}$ atom, the O2 atom of the carboxylate group coordinates to a neighboring Pb$^{\rm II}$ atom to form a centrosymmetric Pb$_2$O$_2$ four-membered ring, with a Pb$\cdots$Pb separation of 4.5813 (4) Å. The bridging

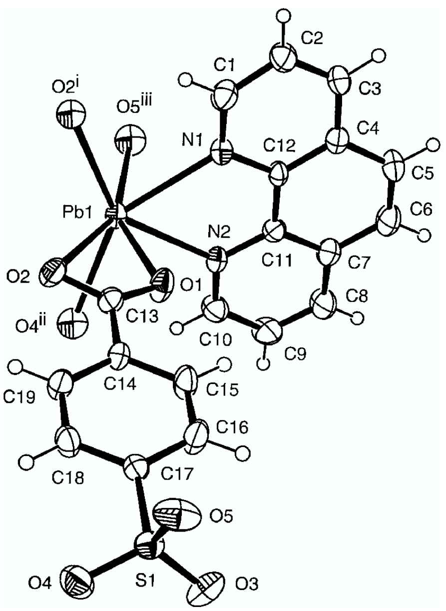


Figure 1
The structure of a segment of (I), showing the coordination geometry. Displacement ellipsoids are drawn at the 40% probability level. Uncoordinated water molecules are omitted for clarity. Symmetry codes (i): $-x, 1-y, 1-z$; (ii): $1-x, 1-y, 1-z$; (iii): $\frac{1}{2}-x, -\frac{1}{2}+y, z$.

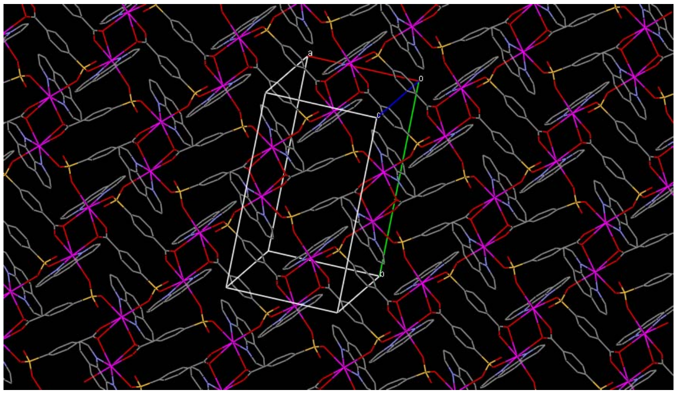


Figure 2
A view of the two-dimensional network of (I). The uncoordinated water molecules and H atoms are omitted for clarity.

mode of the carboxylate is *anti*-skew, with a dihedral angle of 10.1 (8)° between the carboxyl group and the benzene ring. The sulfonate group of the sb ligand links two neighboring Pb^{II} atoms in a skew–skew bridging mode, the $Pb\cdots Pb$ separation being 7.1712 (4) Å. Each sb ligand, therefore, bridges four Pb^{II} atoms to form a two-dimensional sheet network (Fig. 2). Three kinds of metal-containing rings, $[Pb\cdots O\cdots Pb\cdots O]$, $[Pb\cdots sb\cdots Pb\cdots SO_3]$ and $[Pb\cdots sb\cdots Pb\cdots sb]$, are observed in the sheet network. Within the $[Pb\cdots sb\cdots Pb\cdots sb]$ moiety, parallel mean planes of two sb ligands are separated from each other by a face-to-face distance of 3.57 (3) Å.

The coordination geometry around the Pb^{II} atom in (I) may be described as 'hemidirected' (Shimoni-Livny *et al.*, 1998), a significant gap being located in the position *trans* to the phen ligand. In the empty direction, some short contacts are observed, *viz*. $Pb\cdots O6B^{ii}$ = 3.34 (2) Å and $Pb\cdots O3^{ii}$ = 3.828 (4) Å [symmetry code (ii): $1-x, 1-y, 1-z$]. This is similar to the situation found in $[Pb(DOTAM)](ClO_4)_2\cdot 5H_2O$ (DOTAM = 1,4,7,10-tetrakis(carbamoylmethyl)-1,4,7,10-tetraazacyclododecane) (Hancock *et al.*, 2004).

Experimental

A mixture of $Pb(NO_3)_2$ (0.084 g, 0.25 mmol), KHsb (0.061 g, 0.25 mmol), phen (0.046 g, 0.23 mmol) and H_2O (15 ml) was placed in a 30 ml stainless steel reactor with a Teflon liner and heated at 423 K for 24 h. After cooling, colorless block-shaped crystals of (I) were obtained. Elemental and TG analyses clearly indicate that the compound contains a water molecule, with a formula of $[Pb(C_{12}H_8N_2)(C_7H_4O_5S)]\cdot H_2O$. Analysis calculated for $C_{19}H_{14}N_2O_6PbS$: C 37.68, H 2.33, N 4.63%; found: C 37.42, H 2.34, N 4.46%. Weight loss in the temperature range 363–408 K corresponds to the release of the water molecule (calculated 2.98%, found 3.07%).

Crystal data

$[Pb(C_{12}H_8N_2)(C_7H_4O_5S)]\cdot H_2O$
$M_r = 605.57$
Orthorhombic, *Pbca*
$a = 9.8745\ (7)$ Å
$b = 17.3467\ (11)$ Å
$c = 21.7544\ (14)$ Å
$V = 3726.3\ (4)$ Å^3
$Z = 8$
$D_x = 2.159$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3784 reflections
$\theta = 2.5$–$25.2°$
$\mu = 9.21$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
0.29 × 0.14 × 0.07 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.206$, $T_{max} = 0.523$
21368 measured reflections

4064 independent reflections
3160 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 27.0°$
$h = -12 \rightarrow 12$
$k = -22 \rightarrow 18$
$l = -25 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.071$
$S = 1.01$
4064 reflections
273 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 2.5851P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.32$ e Å^{-3}
$\Delta\rho_{min} = -0.75$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pb1—O1	2.439 (3)	Pb1—N1	2.546 (4)
Pb1—O2	2.888 (3)	Pb1—N2	2.540 (4)
Pb1—O2^{i}	2.715 (3)	S1—O3	1.441 (4)
Pb1—O4^{ii}	2.699 (4)	S1—O4	1.441 (4)
Pb1—O5^{iii}	2.694 (4)	S1—O5	1.446 (4)
O1—Pb1—O2	48.28 (10)	O5^{iii}—Pb1—O2^{i}	94.01 (11)
O1—Pb1—O2^{i}	96.05 (11)	N1—Pb1—O2	113.20 (11)
O1—Pb1—O4^{ii}	87.35 (12)	N1—Pb1—O2^{i}	77.50 (11)
O1—Pb1—O5^{iii}	151.95 (13)	N1—Pb1—O4^{ii}	142.56 (12)
O1—Pb1—N1	80.65 (12)	N1—Pb1—O5^{iii}	76.08 (12)
O1—Pb1—N2	78.68 (12)	N2—Pb1—N1	65.22 (13)
O2^{i}—Pb1—O2	70.32 (12)	N2—Pb1—O2	124.04 (11)
O4^{ii}—Pb1—O2	82.72 (10)	N2—Pb1—O2^{i}	142.72 (11)
O4^{ii}—Pb1—O2^{i}	139.34 (11)	N2—Pb1—O4^{ii}	77.73 (12)
O5^{iii}—Pb1—O2	158.36 (11)	N2—Pb1—O5^{iii}	77.49 (12)
O5^{iii}—Pb1—O4^{ii}	101.74 (11)		

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

The aromatic H atoms were positioned geometrically and refined using the riding-model approximation [C—H = 0.93 Å, $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)]. The uncoordinated water is disordered over two sites; occupancies were initially refined and converged to 0.532 (3) and 0.468 (3), respectively, and were then fixed at 0.53 (major component) and 0.47 (minor component) in the final cycles of refinement. H atoms of the disordered water molecule were not located. The highest peak in the final difference Fourier map lies 0.92 Å from atom Pb.

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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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