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Poly[[diaqua(μ_4-3-sulfonatobenzoato)lead(II)] 4,4′-bipyridine]

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The title compound, $[Pb(C_7H_4O_5S)(H_2O)_2]\cdot C_{10}H_8N_2$, crystallizes as a two-dimensional coordination polymer. Double sheets are formed by Pb^{2+} ions which are connected by μ_4-bridging 3-sulfonatobenzoate groups. The coordination around the Pb atoms comprises three O atoms from carboxylate groups, two O atoms from the sulfonate group and additionally two water molecules. As a result of hydrogen bonds between 4,4′-bipyridine and water molecules attached to Pb^{2+} the double layer structure is extended to give a three-dimensional supramolecular architecture.

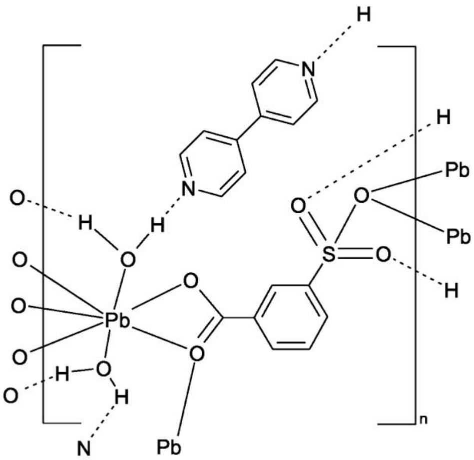
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.019
wR factor = 0.056
Data-to-parameter ratio = 12.5

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

Comment

Sulfonatobenzoates are versatile building blocks for the synthesis of supramolecular coordination compounds. In recent years there have been numerous reports on metal-organic frameworks based on 1,4- and 1,2-sulfonatobenzoates (Fan, Xiao, Zhang, Cai & Zhu, 2004; Fan, Xiao, Zhang & Zhu, 2004; Yuan *et al.*, 2001; Fan *et al.*, 2005; Xiong *et al.*, 2001; Li & Yang, 2004; Xiao, 2005; Su *et al.*, 2005; Ng *et al.*, 1991). Analogous 1,3-sulfonatobenzoates are less studied, but recently crystal structures of Mn^{2+} (Yang *et al.*, 2006; Miao & Zhu, 2006), Ba^{2+} (Gao *et al.*, 2005), $[C(NH_2)_3]^+$ (Videnova-Adrabinska *et al.*, 2001), Zn^{2+} (Zhang & Zhu, 2005), Cu^{2+} (Cai & Zhu, 2005; Miao *et al.*, 2005), Cd^{2+} (Chen *et al.*, 2005) and Pb^{2+} (Ma *et al.*, 2005; Zhu & Xiao, 2005; Ying & Mao, 2004) derivatives have been published.



(I)

In the title compound, (I), the Pb^{2+} cations are linked by 3-sulfonatobenzoate anions to give double layers which are oriented parallel to the crystallographic *ab* plane (Fig. 2). Within the double layers the 3-sulfonatobenzoate anions act as

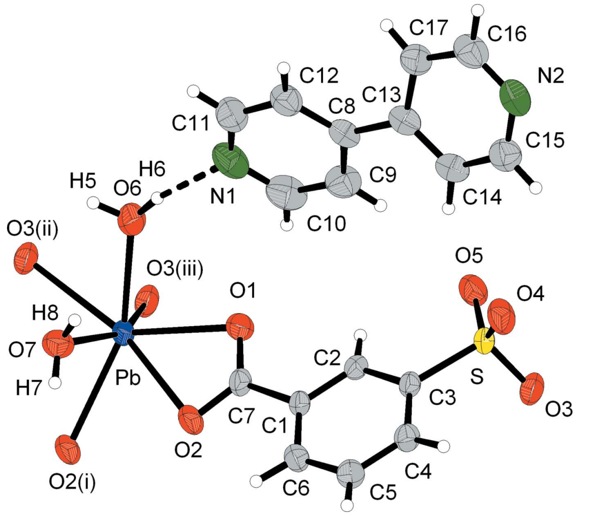


Figure 1
The asymmetric unit of (I), extended to complete the coordination of lead. [Symmetry codes: (i) $-x, 1-y, 2-z$; (ii) $1+x, -1+y, z$; (iii) $-x, 2-y, 2-z$.] Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

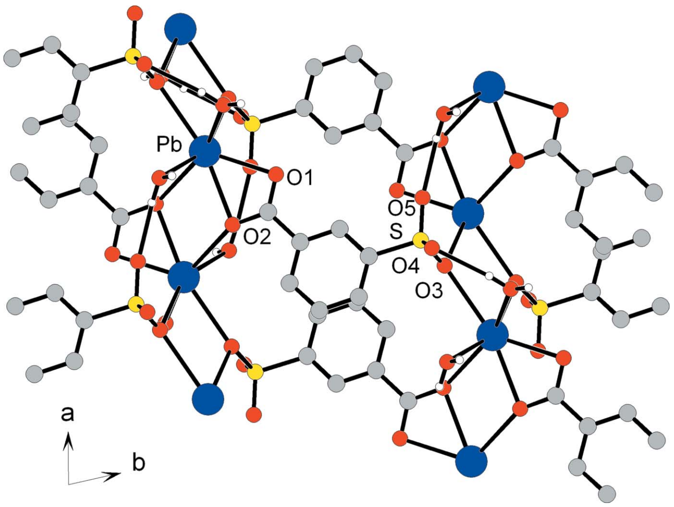


Figure 2
Diagram of the double layer structure of (I). View along the *c* axis. H atoms, except those of water molecules, have been omitted.

μ_4-bridging ligands. The carboxylate groups are coordinated to two Pb^{2+} cations through a tridentate bridging mode and the sulfonate groups bind to two Pb^{2+} cations by a μ_2-bridging O atom.

The Pb^{2+} cations are surrounded by three O atoms from carboxylate groups, two O atoms from sulfonate groups and additionally two water molecules in an irregular sevenfold coordination (Fig. 1). The Pb—O distances are in the expected range (Ying & Mao, 2004; Foreman *et al.*, 2000; Yordanov *et al.*, 1999; Garcia *et al.*, 1999; Yuan *et al.*, 2004) (see Table 1). As a result of the μ-bridging mode of two carboxylate (O2, O2^{i}) and two sulfonate O atoms (O3^{ii}, O3^{iii}) a chain of corner-sharing Pb_2O_2 rings is formed.

Additionally the chain structure is supported by hydrogen bonds between water molecules attached to lead and non-coordinated sulfonate O atoms (O4, O5) (Table 2 and Fig. 3). 4,4′-Bipyridine is uncoordinated and is linked to coordinated water molecules *via* O—H···N hydrogen bridges. Each 4,4′-bipyridine molecule participates in two of these hydrogen bridges and serves therefore as a linker between adjacent double layers to form a three-dimensional network structure (Fig. 4).

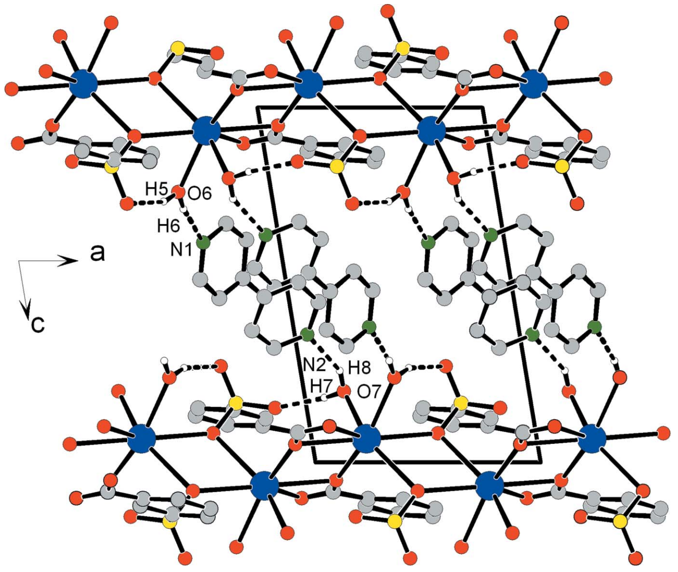


Figure 3
View along the *b* axis showing the linking of the double layers in (I) *via* O—H···O hydrogen-bonding interactions (dashed lines). H atoms, except those of water molecules, have been omitted.

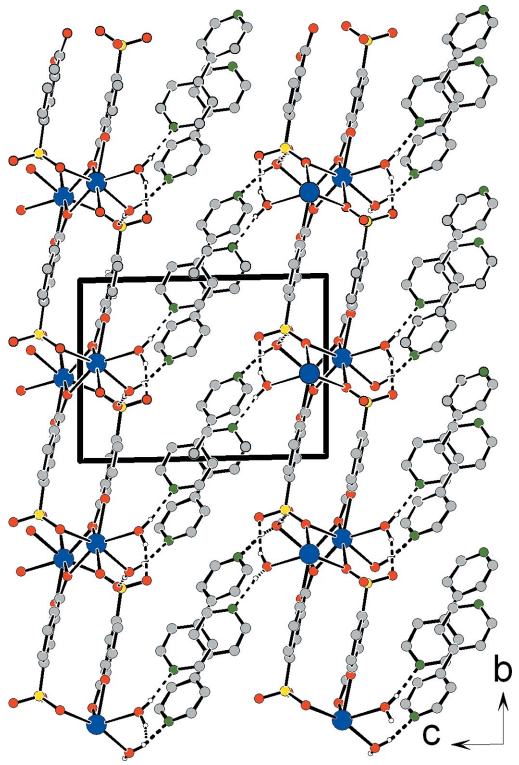


Figure 4
Packing diagram of (I). View along the *a* axis. H atoms, except those of water molecules, have been omitted.

Experimental

A mixture of $Pb(CH_3COO)_2{\cdot}3H_2O$ (0.3793 g, 1 mmol), 3-sulfonatobenzoic acid (0.4482 g, 2 mmol) and 4,4′-bipyridine (0.1562 g, 1 mmol) in water (5 ml) was treated under hydrothermal conditions in a Teflon-lined steel reactor (433 K) for 8 h. After cooling to room temperature, needle-shaped crystals of (I) were obtained in a nearly quantitative yield. Analysis calculated for $C_{17}H_{16}N_2O_7PbS$: C 34.05, H 2.69, N 4.69, S 5.35%; found: C 33.89, H 2.82, N 4.69, S 5.35%.

Crystal data

$[Pb(C_7H_4O_5S)(H_2O)_2]{\cdot}C_{10}H_8N_2$
$M_r = 599.57$
Triclinic, $P\bar{1}$
$a = 8.178$ (5) Å
$b = 9.483$ (4) Å
$c = 12.790$ (6) Å
$\alpha = 89.19$ (4)°
$\beta = 80.73$ (4)°
$\gamma = 78.05$ (4)°
$V = 957.5$ (9) Å^3
$Z = 2$
$D_x = 2.080$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 8.96$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.38 × 0.09 × 0.09 mm

Data collection

Stoe Stadi-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (*X-RED32*; Stoe & Cie, 1996)
$T_{min} = 0.054$, $T_{max} = 0.445$
6715 measured reflections
3380 independent reflections
3236 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.0°$
2 standard reflections
frequency: 60 min
intensity decay: random variation ±5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.056$
$S = 1.19$
3380 reflections
270 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0311P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.98$ e Å^{-3}
$\Delta\rho_{min} = -0.79$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0110 (6)

Table 1
Selected geometric parameters (Å, °).

Pb—O1	2.504 (3)	Pb—O3^{iii}	2.856 (3)
Pb—O2	2.567 (4)	Pb—O6	2.549 (4)
Pb—O2^{i}	2.654 (3)	Pb—O7	2.492 (3)
Pb—O3^{ii}	2.749 (4)		
O1—Pb—O2	51.41 (10)	O2^{i}—Pb—O3^{ii}	98.79 (10)
O1—Pb—O2^{i}	118.59 (11)	O2^{i}—Pb—O3^{iii}	100.95 (10)
O1—Pb—O3^{ii}	141.79 (10)	O2^{i}—Pb—O6	152.37 (12)
O1—Pb—O3^{iii}	96.80 (11)	O2^{i}—Pb—O7	76.48 (11)
O1—Pb—O6	76.72 (12)	O3^{ii}—Pb—O3^{iii}	66.64 (11)
O1—Pb—O7	105.00 (11)	O3^{ii}—Pb—O6	72.88 (11)
O2—Pb—O2^{i}	69.59 (11)	O3^{ii}—Pb—O7	90.41 (11)
O2—Pb—O3^{ii}	166.11 (10)	O3^{iii}—Pb—O6	99.63 (12)
O2—Pb—O3^{iii}	121.90 (10)	O3^{iii}—Pb—O7	156.45 (10)
O2—Pb—O6	113.72 (12)	O6—Pb—O7	77.27 (13)
O2—Pb—O7	79.65 (11)		

Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $x+1, y-1, z$; (iii) $-x, -y+2, -z+2$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···N1	0.842 (19)	2.02 (2)	2.849 (6)	170 (5)
O7—H8···N2^{iv}	0.846 (19)	1.99 (3)	2.773 (5)	153 (5)

Symmetry code: (iv) $-x, -y+2, -z+1$.

The water H atoms were located in a difference map and were refined isotropically with O—H distances restrained to 0.85 (2) and H···H distances to 1.39 (2) Å. The remaining H atoms were included in the riding-model approximation with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *STADI-4* (Stoe & Cie, 1996); cell refinement: *STADI-4*; data reduction: *STADI-4*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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