Crystal Structure of Poly-*catena*-diaqua(4-carboxybenzenesulfonato)Lead(II)

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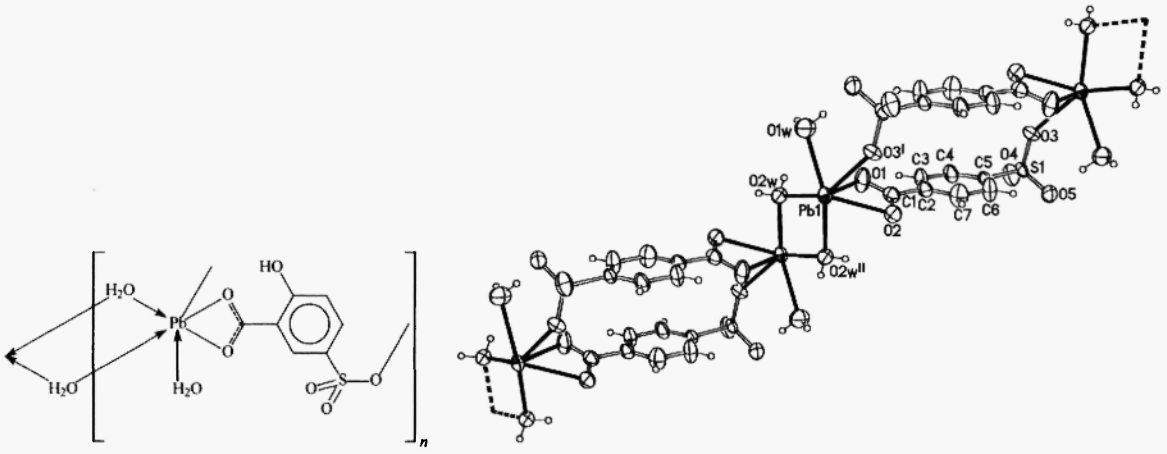

Fig. 1. Chemical structure diagram and *ORTEP* plot of a portion of the polymeric $(C_7H_4O_5S)(H_2O)_2Pb$ structure. Selected bond distances and angles: Pb1–O1 2.400(4), Pb1–O2 2.580(4), Pb1–O3^{i} 2.618(4), Pb1–O1*w* 2.562(4), Pb1–O2*w* 2.670(4), Pb1–O2*w*^{ii} 2.733(4) Å; O1–Pb1–O2 52.2(1), O1–Pb1–O3^{i} 84.3(2), O1–Pb1–O1*w* 78.9(2), O1–Pb1–O2*w* 76.8(1), O1–Pb1–O2*w*^{ii} 76.0(1), O2–Pb1–O3i 68.2(1), O2–Pb1–O1*w* 119.2(1), O2–Pb1–O2*w* 119.1(1), O2–Pb1–O2*w*^{ii} 71.9(1), O3^{i}–Pb1–O2*w* 143.5(1), O3^{i}–Pb1–O2*w*^{ii} 139.7(1), O3^{i}–Pb1–O1*w* 73.9(1), O1*w*–Pb1–O2*w* 72.0(1), O1*w*–Pb1–O2*w*^{ii} 133.7(1), O2*w*–Pb1–O2*w*^{ii} 64.7(1)°. [Symmetry codes: *i* 2 – *x*, 1 – *y*, 2 – *z*; *ii* 1 – *x*, 2 – *y*, 1 – *z*.]

COMMENT

Totally five 4-sulfobenzoate (sb) complexes have been structurally characterized /1-4/. The title compound is the first main group metal compound, in which the sulfonyl group is monodentately coordinated to the Pb(II) atom, while the carboxyl group chelates to the metal atom. Therefore, the $[Pb_2sb_2]$ grid building block is formed. Bridged water moledules connect $[Pb_2sb_2]$ boxes and extend the structure into a one-dimensional chain. It is worth noting that coordinated terminal water and bridging water molecules form

hydrogen bonds with sulfonyl and carboxyl groups and result in the three-dimensional hydrogen-bonding network. Interestingly, the coordination sphere can be considered as hemidirected [5]. The lone-pair of electrons on Pb(II) is stereochemically active, the lone-pair occupying one axial site of the pentagonal bipyramid around the lead atom (Fig. 2).

EXPERIMENTAL

The compound was synthesized by typical mixed method. $Pb(NO_3)_2$ (0.165 g, 0.5 mmol), 4,4'-bipyridine (0.078 g, 0.5 mmol), potassium hydrogen 4-sulfobenzoate (0.120 g, 0.5 mmol) were added to a solution of water (30 ml) and methanol (5 ml) and stirred for two hours. Then the mixture was filtered and the filtrate was left to stand at room temperature. After four weeks, colorless block-shaped crystals were obtained. Diffraction measurements were performed on a Bruker area-detector diffractometer; the structure was solved by *SHELXS*-97 and refined by *SHELXL*-97 [6]. Non-hydrogen atoms were refined anisotropically; The aromatic H atoms were generated geometrically (C-H 0.93 Å) and they were refined as riding, with U(H) – 1.2Ueq(C). The water H atoms were found from difference Fourier maps, and were refined with distance restraints of O–H 0.85 and H···H 1.39 Å; U(H) was also sent to 1.2U_{eq}(O).

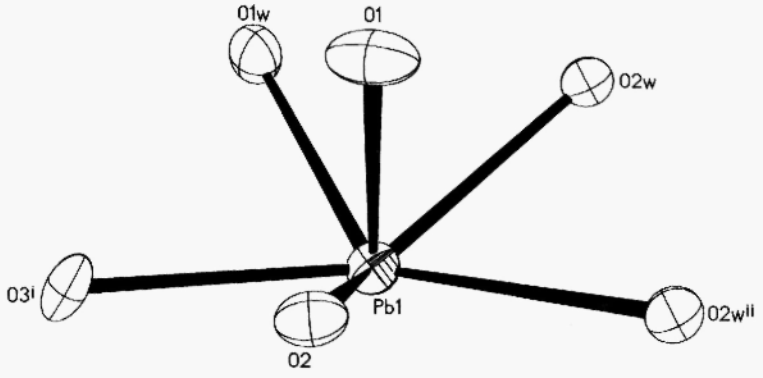

Fig. 2. Geometry of the lead atom.

Table 1

Crystal data for $(C_7H_4O_5S)(H_2O)_2Pb$

Formula	$(C_7H_4O_5S)(H_2O)_2Pb$	Formula weight	443.38
Crystal system	Triclinic	Crystal size, mm	0.26 x 0.24 x 0.16
Space group	*P*-1	*Z*	2
a, Å	6.0675(5)	α, °	74.445(1)
b, Å	8.2808(7)	β, °	87.369(1)
c, Å	10.7982(9)	γ, °	80.934(1)
Temperature, K	295	Diffractometer	Bruker APEX CCD
Trans. Factors	0.063 – 0.177	μ(Mo), mm^{-1}	16.563

Table 1 (continued)

$F(000)$	408	D_{calc}, g cm^{-3}	2.853
Reflns. measured	4355	θ_{max} °	27.5
Reflns. with $I>2\sigma(I)$	2180	Weighting scheme	$w = [\sigma^2(F^2)+(0.0445P)^2]^{-1}$
$R(F^2)$, $wR(F^2)$	0.027, 0.070	ρ, e Å^{-3}	-2.61 – 1.95
Programs used	*SHELXS*, *SHELXL*	Deposition number	CCDC-264867

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