Zhiwei Tang* and Decai Wen

Crystal structure of diaqua-bis(μ₂-5-chlorosalicylato-κ³O,O′:O′)-bis(5-chlorosalicylato-κ²O,O′)-bis(1,10-phenanthroline-κ²N,N′) dilead(II) – water (1/2), C₅₂H₃₆C₁₄N₄O₁₄Pb₂·2(H₂O)

Source of material

A mixture of Pb(NO₃)₂ (0.1 mmol), 1,10-phenanthroline (0.1 mmol), 5-chloro-2-hydroxybenzoic acid (0.2 mmol) and distilled water (10 mL) was put into a Teflon-lined autoclave (20 mL) and then heated at 433 K for 3D, and then slowly cooled to room temperature. Colourless block crystals of the title complex were collected from the reaction mixture.

Experimental details

The aromatic H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [C–H = 0.93 Å and Ueq(H) = 1.2 Ueq(C)]. The H atoms of the water molecules were found in a difference Fourier map and were refined with distance restraints of O–H = 0.83(1) Å and Uiso(H) = 1.2 Ueq(O).

Comment

Salicylic acid (H₂sal) and deprotonated derivatives are used as carboxylate ligands [4, 5]. 1,10-Phenanthroline (phen) often acts as a chelating ligand for its high affinity to metal ions and also plays an important role in the
development of supramolecular chemistry [6, 7]. Furthermore, lead(II) complexes have received considerable attention due to the coordination and interesting stereochemical activity of the valence shell lone electron pairs [8, 9]. Herein, we chose 5-chloro-2-hydroxybenzoate as the bridging ligand and 1,10-phenanthroline as a terminal ligand to generate a dimeric complex with Pb(II).

Single-crystal X-ray diffraction analysis reveals that the asymmetric unit of title complex contains one independent Pb(II) cation, two 5-Cl-Sal anions, one phen ligand, one coordinated water molecule and one guest water molecule, as shown in the figure. Each Pb(II) ions is eight coordinated by one oxygen atom from a coordinated water molecule, two nitrogen atoms from a neutral phen ligand, four carboxylate oxygen atoms from two 5-Cl-Sal anions and another oxygen atom of a 5-Cl-Sal ligand from another [Pb(II)(5-Cl-Sal),(phen)(H₂O)] moiety. The adjacent Pb(II) cations are connected by 5-Cl-Sal anions adopting bidentate bridging mode to form a centrosymmetric dimer with the Pb–Pb distance of 4.257(1) Å. The Pb–O bond lengths are in the range from 2.463(3) to 2.985(3) Å, while the Pb–N bond lengths are 2.574(3) and 2.621(3) Å, respectively. All geometric parameters are in the typical ranges [10].

There exist extensive intermolecular hydrogen bonds involving coordinated water molecules, lattice water molecules and coordinated carboxylate oxygen atoms of 5-Cl-Sal ligands in the neighboring dimeric units (O1W–H1WA···O2 with the donor–acceptor distance 2.806(2) Å and a bond angle of 178(5)° as well as O2W–H2WA···O1 with the donor–acceptor distance 2.887(5) Å and a bond angle of 174(5)°, symmetric code: ‘1 + x, +y, +z’). In addition, weak face to face π–π interactions are observed between the neighbour 5-Cl-Sal ligands (the ring of C2–C7) with a centroid–centroid distance is 3.6316(2) Å.

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References