Crystal structure of dipimelato-bis(1,10-phenanthroline-*N*,*N*′)dilead(II) monohydrate, $Pb_2(C_{12}H_8N_2)_2(C_7H_{10}O_4)_2 \cdot H_2O$

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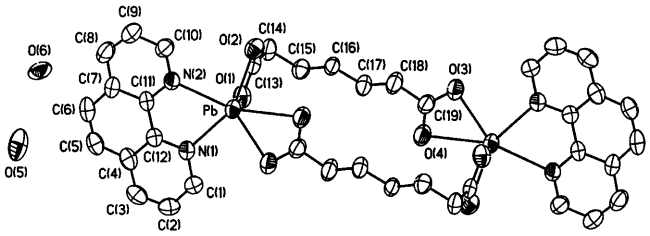

Abstract

$C_{19}H_{20}N_2O_5Pb$, triclinic, $P\bar{1}$ (No. 2), a = 7.488(2) Å, b = 11.543(2) Å, c = 11.742(2) Å, α = 83.50(3)°, β = 82.54(3)°, γ = 81.94(3)°, V = 991.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.128, T = 293 K.

Source of material

Addition of 2.0 ml (1 M) Na_2CO_3 to a stirred aqueous solution of $Pb(NO_3)_2$ (0.168 g, 0.51 mmol) in 5.0 ml H_2O produced white precipitate, which was separated out by centrifugation and then added to a stirred methanolic aqueous solution of phenanthroline monohydrate (0.102 g, 0.51 mmol) and pimelic acid (0.083 g, 0.52 mmol) in 20 ml CH_3OH–H_2O (1:1 v/v). The mixture was stirred for 30 min and the formed suspension was filtered off. The filtrate (pH = 5.90) was allowed to stand at room temperature and slow evaporation afforded a few colorless needle-like crystals.

Discussion

The title compound consists of hydrogen bonded H_2O molecules and dinuclear $[Pb_2(phen)_2(C_7H_{10}O_4)_2]$ complex molecules centered at the crystallographic 1*f* position. Within the complex molecules, the Pb atoms are coordinated by two N atoms of one phen ligand and four O atoms of two chelating carboxylate groups of different bis-chelating pimelato ligands with d(Pb—N) = 2.603 Å, 2.628 Å and d(Pb—O) = 2.304 Å – 2.662 Å. Due to the lone pair effect of the Pb atom, the coordination polyhedra can be roughly viewed as mono-capped square pyramids. In the (001) plane, the dinuclear complex molecules are so arranged that the phen ligands are each sandwiched by two symmetry-related phen neighbors. The mean interplanar distance of 3.44 Å suggests that the intermolecular π-π stacking interactions are responsible for supramolecular assembly of the dinuclear complex molecules into layers. The formed layers are further assembled by intermolecular C–H···O hydrogen bonds to generate 3D framework with the hydrogen bonded H_2O molecules located in 1D tunnels extending along the [100] direction. The bis-chelating pimelato ligands are twisted with the torsion angle of –65° for C13–C14–C15–C16 chain.

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Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.133 × 0.222 × 0.333 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	85.37 cm^{-1}
Diffractometer, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5584, 4540
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3787
$N(param)_{refined}$:	256
Programs:	SHELXS-97 [1], SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	2*i*		0.5106	0.5400	0.2750	0.08
H(2)	2*i*		0.6342	0.3454	0.2779	0.08
H(3)	2*i*		0.7967	0.2741	0.1183	0.08
H(5)	2*i*		0.9417	0.3152	–0.0871	0.08
H(6)	2*i*		0.9762	0.4393	–0.2482	0.08
H(8)	2*i*		0.9169	0.6450	–0.3502	0.08
H(9)	2*i*		0.7834	0.8338	–0.3395	0.08
H(10)	2*i*		0.6301	0.8948	–0.1680	0.08
H(14A)	2*i*		0.9531	1.0447	0.1171	0.10
H(14B)	2*i*		1.0486	0.9151	0.1337	0.10
H(15A)	2*i*		0.9103	0.8993	0.3272	0.10
H(15B)	2*i*		1.0424	0.9953	0.3050	0.10
H(16A)	2*i*		0.7934	1.1425	0.2917	0.10
H(16B)	2*i*		0.6613	1.0463	0.3143	0.10
H(17A)	2*i*		0.7478	0.9976	0.4992	0.10
H(17B)	2*i*		0.8780	1.0947	0.4765	0.10
H(18A)	2*i*		0.5019	1.1437	0.4843	0.10
H(18B)	2*i*		0.6353	1.2385	0.4691	0.10
H(19A)	2*i*	0.50	0.2935	0.3341	–0.3973	0.05
H(19B)	2*i*	0.50	0.1925	0.4330	–0.4580	0.05
H(20A)	2*i*	0.50	0.2531	0.6374	–0.5799	0.05
H(20B)	2*i*	0.50	0.0660	0.6062	–0.5500	0.05

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pb	2*i*		0.47110(4)	0.80879(2)	0.11382(2)	0.0481(2)	0.0527(2)	0.0446(2)	0.0020(1)	–0.0034(1)	–0.0110(1)
N(1)	2*i*		0.6092(9)	0.5888(6)	0.1155(6)	0.057(4)	0.051(3)	0.048(3)	–0.006(3)	–0.005(3)	–0.010(3)
N(2)	2*i*		0.661(1)	0.7417(6)	–0.0773(5)	0.064(4)	0.049(3)	0.043(3)	–0.003(3)	–0.006(3)	–0.008(3)
C(1)	2*i*		0.581(1)	0.5132(8)	0.2095(8)	0.077(6)	0.056(5)	0.051(4)	–0.009(4)	–0.004(4)	–0.001(4)
C(2)	2*i*		0.653(2)	0.3956(9)	0.2110(9)	0.095(7)	0.060(5)	0.060(5)	–0.011(5)	–0.010(5)	0.011(4)
C(3)	2*i*		0.751(2)	0.3534(8)	0.1170(9)	0.076(6)	0.044(4)	0.085(7)	0.002(4)	–0.016(5)	0.000(4)
C(4)	2*i*		0.785(1)	0.4296(7)	0.0164(8)	0.056(5)	0.051(4)	0.070(5)	–0.001(4)	–0.020(4)	–0.016(4)
C(5)	2*i*		0.888(1)	0.3928(8)	–0.0862(9)	0.061(5)	0.050(4)	0.081(6)	0.007(4)	–0.009(4)	–0.021(4)
C(6)	2*i*		0.910(1)	0.4670(9)	–0.1820(9)	0.063(5)	0.070(6)	0.075(6)	0.005(4)	–0.005(5)	–0.037(5)
C(7)	2*i*		0.835(1)	0.5858(8)	–0.1841(7)	0.055(5)	0.065(5)	0.049(4)	–0.002(4)	0.000(3)	–0.021(4)
C(8)	2*i*		0.852(1)	0.668(1)	–0.2815(8)	0.067(6)	0.088(7)	0.052(5)	–0.012(5)	0.012(4)	–0.015(4)
C(9)	2*i*		0.774(1)	0.780(1)	–0.2745(7)	0.076(6)	0.081(6)	0.044(4)	–0.012(5)	–0.006(4)	0.000(4)
C(10)	2*i*		0.680(1)	0.8166(8)	–0.1710(7)	0.072(6)	0.061(5)	0.049(4)	–0.002(4)	–0.001(4)	–0.002(4)
C(11)	2*i*		0.733(1)	0.6277(6)	–0.0813(6)	0.046(4)	0.046(4)	0.049(4)	–0.001(3)	–0.009(3)	–0.017(3)
C(12)	2*i*		0.706(1)	0.5479(6)	0.0193(6)	0.044(4)	0.046(4)	0.048(4)	–0.001(3)	–0.010(3)	–0.008(3)
O(1)	2*i*		0.7561(8)	0.8162(6)	0.1645(5)	0.054(3)	0.064(4)	0.066(4)	0.005(3)	–0.013(3)	–0.018(3)
O(2)	2*i*		0.6679(9)	0.9846(6)	0.0653(5)	0.061(4)	0.075(4)	0.053(3)	0.001(3)	–0.010(3)	–0.004(3)
C(13)	2*i*		0.777(1)	0.9235(7)	0.1231(6)	0.053(4)	0.058(4)	0.037(3)	0.002(4)	–0.001(3)	–0.018(3)
C(14)	2*i*		0.941(1)	0.9676(9)	0.1574(7)	0.052(5)	0.072(5)	0.056(5)	–0.006(4)	0.004(4)	–0.027(4)
C(15)	2*i*		0.928(1)	0.9757(8)	0.2871(7)	0.059(5)	0.066(5)	0.046(4)	–0.001(4)	–0.014(4)	–0.007(4)
C(16)	2*i*		0.776(1)	1.0661(7)	0.3320(6)	0.065(5)	0.053(4)	0.037(3)	0.000(4)	–0.004(3)	–0.008(3)
C(17)	2*i*		0.764(1)	1.0745(8)	0.4593(7)	0.065(5)	0.064(5)	0.044(4)	0.003(4)	–0.009(4)	–0.015(3)
C(18)	2*i*		0.615(2)	1.1611(9)	0.5060(7)	0.093(7)	0.071(6)	0.044(4)	0.014(5)	0.000(4)	–0.012(4)
C(19)	2*i*		0.593(1)	1.1664(8)	0.6358(7)	0.064(5)	0.061(5)	0.046(4)	0.000(4)	0.004(4)	–0.007(4)
O(3)	2*i*		0.565(1)	1.2651(6)	0.6723(6)	0.111(6)	0.060(4)	0.054(3)	0.006(4)	0.010(4)	–0.013(3)
O(4)	2*i*		0.603(1)	1.0732(6)	0.7000(5)	0.077(4)	0.066(4)	0.049(3)	0.008(3)	0.000(3)	–0.010(3)
O(5)	2*i*	0.50	0.185(2)	0.352(2)	–0.420(1)	0.072(9)	0.13(1)	0.076(9)	0.042(9)	–0.023(8)	–0.039(9)
O(6)	2*i*	0.50	0.177(2)	0.597(2)	–0.539(1)	0.09(1)	0.11(1)	0.063(8)	–0.060(9)	0.013(7)	–0.001(7)

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