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Crystal structure of bis(1,10-phenanthroline-*N,N'*)bis(2-bromobenzoato)lead(II) hydrate (1:2.5), $[Pb(C_7H_4O_2Br)_2(C_{12}H_8N_2)_2] \cdot 2.5H_2O$

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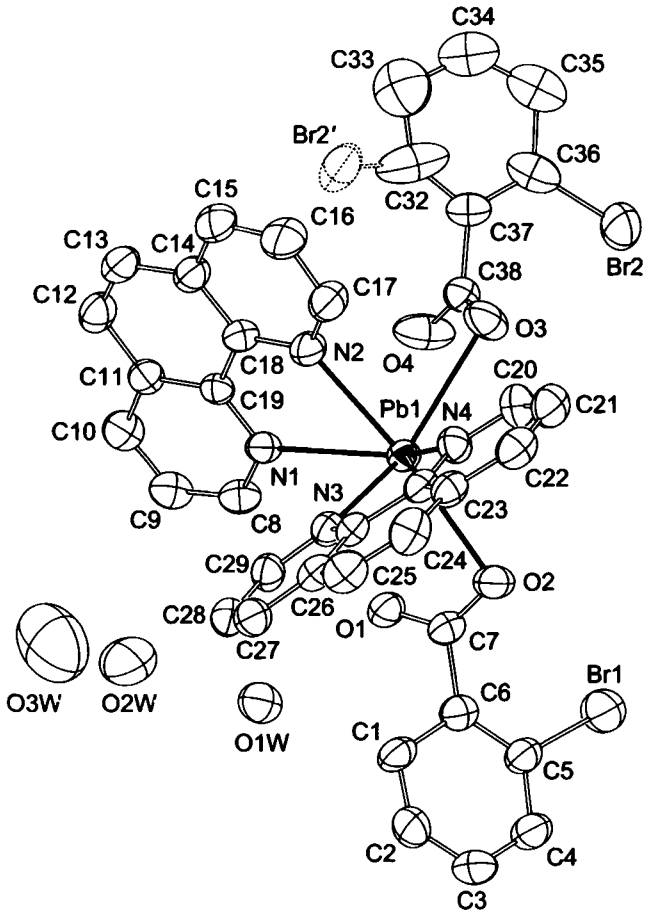

Abstract

$C_{76}H_{58}Br_4N_8O_{13}Pb_2$, triclinic, $P\bar{1}$ (no. 2), a = 11.6717(2) Å, b = 12.5543(2) Å, c = 13.6995(3) Å, α = 95.132(1)°, β = 112.690(1)°, γ = 103.895(1)°, V = 1760.3 Å^3, Z = 1, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.115, T = 293 K.

Source of material

Freshly prepared $PbCO_3$ (0.38 g 1.42 mmol), 1,10-phenanthroline (phen · H_2O, 0.15 g, 0.76 mmol), 2-bromobenzoic acid (0.10 g, 0.50 mmol), 15 mL CH_3OH/H_2O (1:2, *v/v*) were mixed and stirred for ca. 1 h. Subsequently, the resulting suspension was heated in a 23 mL Teflon-lined stainless steel autoclave at 423 K for 7 days. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting orange filtrate was allowed to stand at room temperature and slow evaporation for 1 week afforded colorless block-like crystals.

Discussion

The occupational ratio of 3/1 for Br2 and Br2' atoms, respectively, and the occupancy factor of 0.5 for the hydrate water occupancy were estimated from the refinement with isotropic displacement parameters.

Experimental details

The crystal structure of the title compound is similar to the structure of bis(1,10-phenanthroline-*N,N'*)(2-iodobenzoato)lead(II) 2-iodobenzoate dihydrate [1], bis(1,10-phenanthroline-*N,N'*)bis(2-chlorobenzoato)lead(II) hydrate (1:2.5) [2] and (1,10-phenanthroline-*N,N'*)bis(2-fluorobenzoato)lead(II) [3]. It consists of complex $[Pb(phen)_2L_2]$ molecules (H*L* = 2-bromobenzoic acid) and lattice water molecules. Within the complex molecules, the Pb atoms are coordinated by four N atoms from two bidentately chelating *o*-phenanthroline ligands and two O atoms from two 2-bromobenzoic acid anions ligands, to complete significantly distorted PbN_4O_2 environment with *d*(Pb—N) = 2.593(4) Å – 2.681(4) Å, *d*(Pb—O) = 2.714(4) Å – 2.757(4) Å. The dihedral angle between both phen ligands chelating the Pb atom is 83.7(1)°. Two $[Pb(phen)_2(L)_2]$ complex molecules are interconnected via week Pb···Br2 interactions with *d*(Pb···Br2) = 4.111 Å, The lattice water molecules show hydrogen bonding to the carboxylate O atoms of 2-bromobenzoate anions, and Br2 atom with *d*(O···O) = 2.741 Å – 2.960 Å, ∠O–H···O = 130.5° – 166.4°, *d*(O3w···Br2) = 3.362 Å, ∠O3w–HO3w···Br2 = 124.2°, respectively. The chelating phen ligand exhibits nearly perfect co-planarity, each phen ligand are sandwiched by two phen ligands from two neighboring molecules. The mean interplanar distances are alternatively of 3.389 Å and 3.416 Å, indicating π-π stacking interactions. Moreover, the solvate water molecules H_2O(3W) and Br1 atom show weak hydrogen bonding interactions with *d*(C15···O3w^i) = 3.20(3) Å, *d*(C35···Br1^{ii}) = 3.564(1) Å, ∠C15–H15···O3w^i = 148.7(8)°, ∠C35–H35···Br1^{ii} = 132.7(6) (i: –*x*,1–*y*,1–*z*; ii: 1–*x*,–*y*,1–*z*). Through the hydrogen and π-π stacking interactions the compound is interlinked to forms the 3D network.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.115 × 0.201 × 0.432 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	71.18 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	61.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	38709, 1008
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 7226
$N(param)_{refined}$:	478
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1WA)	2*i*		0.3640	0.5662	0.2254	0.109
H(1WB)	2*i*		0.4577	0.6611	0.2635	0.109
H(2WA)	2*i*		0.1576	0.7165	0.1899	0.217
H(2WB)	2*i*		0.2543	0.6894	0.2583	0.217
H(3WA)	2*i*	0.5	0.3062	0.9074	0.4499	0.313
H(3WB)	2*i*	0.5	0.3622	0.8419	0.4033	0.313
H(1)	2*i*		0.2029	0.4270	0.0286	0.079
H(2)	2*i*		0.2021	0.4594	−0.1300	0.089
H(3)	2*i*		0.3359	0.4151	−0.1978	0.086
H(4)	2*i*		0.4949	0.3379	−0.0973	0.079
H(8)	2*i*		0.4049	0.5504	0.4259	0.066
H(9)	2*i*		0.4230	0.7131	0.5274	0.071
H(10)	2*i*		0.3058	0.7103	0.6295	0.073
H(12)	2*i*		0.1365	0.5963	0.6850	0.074
H(13)	2*i*		−0.0036	0.4304	0.6703	0.070
H(15)	2*i*		−0.0990	0.2245	0.5972	0.082
H(16)	2*i*		−0.1194	0.0716	0.4766	0.099
H(17)	2*i*		0.0120	0.0888	0.3861	0.082
H(20)	2*i*		0.1568	−0.0323	0.2816	0.074
H(21)	2*i*		0.0004	−0.1873	0.1524	0.083
H(22)	2*i*		−0.1644	−0.1599	0.0076	0.078
H(24)	2*i*		−0.2813	−0.0281	−0.0842	0.083
H(25)	2*i*		−0.2919	0.1490	−0.0943	0.081
H(27)	2*i*		−0.1865	0.3561	−0.0237	0.073
H(28)	2*i*		−0.0163	0.5010	0.1064	0.071
H(29)	2*i*		0.1471	0.4613	0.2445	0.062
H(32)	2*i*	0.75	0.2626	0.1677	0.6833	0.138
H(33)	2*i*		0.2421	0.1048	0.8240	0.173
H(34)	2*i*		0.3434	−0.0282	0.8774	0.138
H(35)	2*i*		0.4794	−0.0746	0.8119	0.121
H(36)	2*i*	0.25	0.4835	0.0000	0.6556	0.102

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(1)	2*i*		0.27170(2)	0.25691(1)	0.35245(1)	0.0484(1)	0.0451(1)	0.0395(1)	0.01345(8)	0.01904(8)	0.00760(7)
N(1)	2*i*		0.2760(4)	0.4484(3)	0.4592(3)	0.052(2)	0.046(2)	0.043(2)	0.011(2)	0.022(2)	0.011(2)
N(2)	2*i*		0.1032(4)	0.2466(3)	0.4404(4)	0.063(3)	0.047(2)	0.059(3)	0.012(2)	0.035(2)	0.010(2)
N(3)	2*i*		0.0785(4)	0.2974(3)	0.2027(3)	0.051(2)	0.049(2)	0.045(2)	0.017(2)	0.022(2)	0.007(2)
N(4)	2*i*		0.0845(4)	0.0834(3)	0.2184(4)	0.059(2)	0.044(2)	0.053(3)	0.015(2)	0.023(2)	0.006(2)
Br(1)	2*i*		0.57933(8)	0.27100(8)	0.10525(7)	0.0880(5)	0.1162(6)	0.0863(5)	0.0554(5)	0.0433(4)	0.0437(4)
Br(2)	2*i*	0.75	0.5450(1)	−0.01342(9)	0.63477(8)	0.1051(7)	0.1060(7)	0.0746(6)	0.0680(6)	0.0399(5)	0.0309(5)
Br(2')	2*i*	0.25	0.2036(4)	0.1641(4)	0.6879(3)	0.114(3)	0.154(3)	0.086(2)	0.062(3)	0.050(2)	0.017(2)
O(1)	2*i*		0.3749(4)	0.4120(3)	0.2470(3)	0.079(3)	0.051(2)	0.055(2)	0.017(2)	0.039(2)	0.007(2)
O(1W)	2*i*		0.3784(4)	0.6342(3)	0.2266(3)	0.087(3)	0.060(2)	0.063(3)	0.015(2)	0.029(2)	0.009(2)
O(2)	2*i*		0.3442(5)	0.2338(3)	0.1880(3)	0.111(3)	0.049(2)	0.059(2)	0.018(2)	0.049(2)	0.014(2)
O(2W)	2*i*		0.1871(8)	0.7061(7)	0.2553(7)	0.159(6)	0.185(7)	0.169(7)	0.091(6)	0.116(6)	0.092(6)
O(3)	2*i*		0.2900(5)	0.0857(3)	0.4649(3)	0.121(4)	0.056(2)	0.047(2)	0.031(2)	0.022(2)	0.014(2)
O(3W)	2*i*	0.5	0.295(2)	0.862(2)	0.395(2)	0.16(2)	0.20(2)	0.26(3)	0.09(2)	0.07(2)	0.06(2)
O(4)	2*i*		0.4247(5)	0.2465(4)	0.5733(4)	0.106(4)	0.079(3)	0.061(3)	−0.019(3)	0.027(3)	0.019(2)
C(1)	2*i*		0.2621(7)	0.4092(4)	0.0050(5)	0.102(5)	0.047(3)	0.061(4)	0.010(3)	0.055(4)	0.004(3)
C(2)	2*i*		0.2629(7)	0.4266(6)	−0.0887(6)	0.087(5)	0.085(4)	0.065(4)	0.050(4)	0.031(4)	0.029(3)
C(3)	2*i*		0.3436(7)	0.4014(6)	−0.1303(5)	0.089(4)	0.080(4)	0.057(4)	0.028(4)	0.038(3)	0.025(3)
C(4)	2*i*		0.4379(7)	0.3548(5)	−0.0705(5)	0.082(4)	0.066(3)	0.061(4)	0.021(3)	0.043(3)	0.014(3)
C(5)	2*i*		0.4467(5)	0.3336(4)	0.0284(4)	0.064(3)	0.056(3)	0.051(3)	0.015(2)	0.030(3)	0.008(2)
C(6)	2*i*		0.3647(5)	0.3579(4)	0.0715(4)	0.059(3)	0.047(3)	0.050(3)	0.007(2)	0.025(3)	0.005(2)
C(7)	2*i*		0.3657(5)	0.3333(4)	0.1793(4)	0.051(3)	0.054(3)	0.043(3)	0.010(2)	0.022(2)	0.006(2)
C(8)	2*i*		0.3552(5)	0.5476(4)	0.4654(4)	0.048(3)	0.060(3)	0.049(3)	0.007(2)	0.019(2)	0.014(2)
C(9)	2*i*		0.3674(5)	0.6459(4)	0.5268(4)	0.060(3)	0.051(3)	0.050(3)	0.001(2)	0.016(3)	0.008(2)
C(10)	2*i*		0.2977(6)	0.6445(5)	0.5869(4)	0.069(3)	0.051(3)	0.045(3)	0.010(3)	0.013(3)	−0.002(2)
C(11)	2*i*		0.2129(5)	0.5413(4)	0.5834(4)	0.056(3)	0.049(2)	0.040(3)	0.016(2)	0.016(2)	0.005(2)
C(12)	2*i*		0.1311(6)	0.5327(5)	0.6411(5)	0.071(4)	0.071(3)	0.044(3)	0.029(3)	0.022(3)	0.004(3)
C(13)	2*i*		0.0478(6)	0.4341(5)	0.6322(5)	0.063(3)	0.069(3)	0.051(3)	0.022(3)	0.030(3)	0.010(3)
C(14)	2*i*		0.0365(5)	0.3349(5)	0.5655(4)	0.060(3)	0.065(3)	0.050(3)	0.022(3)	0.027(3)	0.010(2)
C(15)	2*i*		−0.0501(6)	0.2314(5)	0.5569(6)	0.063(3)	0.070(4)	0.086(5)	0.015(3)	0.049(3)	0.019(3)
C(16)	2*i*		−0.0599(7)	0.1407(5)	0.4871(6)	0.084(4)	0.064(4)	0.110(6)	0.003(3)	0.064(4)	0.018(4)
C(17)	2*i*		0.0193(6)	0.1520(5)	0.4317(6)	0.080(4)	0.051(3)	0.082(4)	0.008(3)	0.051(4)	0.004(3)
C(18)	2*i*		0.1138(5)	0.3388(4)	0.5074(4)	0.050(3)	0.050(3)	0.041(3)	0.011(2)	0.021(2)	0.009(2)
C(19)	2*i*		0.2042(5)	0.4448(4)	0.5161(4)	0.051(3)	0.047(2)	0.037(2)	0.018(2)	0.017(2)	0.013(2)
C(20)	2*i*		0.0889(6)	−0.0210(4)	0.2238(5)	0.063(3)	0.051(3)	0.067(4)	0.020(3)	0.024(3)	0.005(3)
C(21)	2*i*		−0.0050(6)	−0.1154(5)	0.1457(6)	0.083(4)	0.042(3)	0.083(5)	0.008(3)	0.043(4)	−0.001(3)
C(22)	2*i*		−0.1030(6)	−0.0990(5)	0.0608(5)	0.061(3)	0.057(3)	0.064(4)	0.002(3)	0.028(3)	−0.008(3)
C(23)	2*i*		−0.1126(5)	0.0098(4)	0.0527(4)	0.054(3)	0.052(3)	0.048(3)	0.008(2)	0.024(2)	0.001(2)
C(24)	2*i*		−0.2160(6)	0.0317(5)	−0.0319(5)	0.056(3)	0.066(4)	0.063(4)	0.005(3)	0.017(3)	−0.012(3)
C(25)	2*i*		−0.2223(6)	0.1371(6)	−0.0386(5)	0.055(3)	0.083(4)	0.050(3)	0.015(3)	0.013(3)	0.005(3)
C(26)	2*i*		−0.1216(5)	0.2304(5)	0.0402(4)	0.050(3)	0.067(3)	0.044(3)	0.018(2)	0.021(2)	0.007(2)
C(27)	2*i*		−0.1203(6)	0.3408(5)	0.0325(5)	0.058(3)	0.076(4)	0.058(3)	0.031(3)	0.025(3)	0.022(3)
C(28)	2*i*		−0.0190(6)	0.4269(5)	0.1100(5)	0.068(3)	0.062(3)	0.061(3)	0.031(3)	0.033(3)	0.018(3)
C(29)	2*i*		0.0789(5)	0.4024(4)	0.1934(4)	0.057(3)	0.050(3)	0.050(3)	0.020(2)	0.022(2)	0.006(2)
C(30)	2*i*		−0.0185(5)	0.2124(4)	0.1275(4)	0.045(2)	0.051(2)	0.044(3)	0.015(2)	0.024(2)	0.007(2)
C(31)	2*i*		−0.0139(5)	0.0994(4)	0.1349(4)	0.047(2)	0.049(2)	0.047(3)	0.012(2)	0.026(2)	0.005(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(32)	2*i*		0.3066(9)	0.1158(9)	0.7074(8)	0.103(6)	0.136(8)	0.093(6)	–0.008(6)	0.053(6)	0.014(6)
C(33)	2*i*		0.291(1)	0.081(1)	0.792(1)	0.18(1)	0.14(1)	0.125(9)	–0.001(9)	0.111(9)	0.021(8)
C(34)	2*i*		0.354(1)	0.0054(9)	0.8225(8)	0.127(8)	0.108(7)	0.106(7)	0.021(6)	0.049(7)	0.044(6)
C(35)	2*i*		0.4317(9)	–0.0266(8)	0.7829(8)	0.088(5)	0.092(5)	0.097(6)	0.013(4)	0.020(5)	0.032(5)
C(36)	2*i*		0.4348(7)	0.0210(6)	0.6897(6)	0.081(4)	0.075(4)	0.083(5)	0.007(4)	0.022(4)	0.036(4)
C(37)	2*i*		0.3688(5)	0.0948(5)	0.6517(5)	0.052(3)	0.065(3)	0.050(3)	0.006(3)	0.021(3)	0.018(3)
C(38)	2*i*		0.3650(5)	0.1449(5)	0.5559(4)	0.057(3)	0.058(3)	0.052(3)	0.023(3)	0.025(3)	0.018(2)

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