Crystal structure of bis(1,10-phenanthroline-*N*,*N*')bis(2-chlorobenzoato)-lead(II) hydrate (1:2.5), $[Pb(C_7H_4O_2Cl)_2(C_{12}H_8N_2)_2] \cdot 2.5H_2O$

H.-M. Guo, B.-S. Zhang* and Y.-H. Wang

Jinhua University, Normal College, Jinhua, Zhejiang 321017, P. R. China

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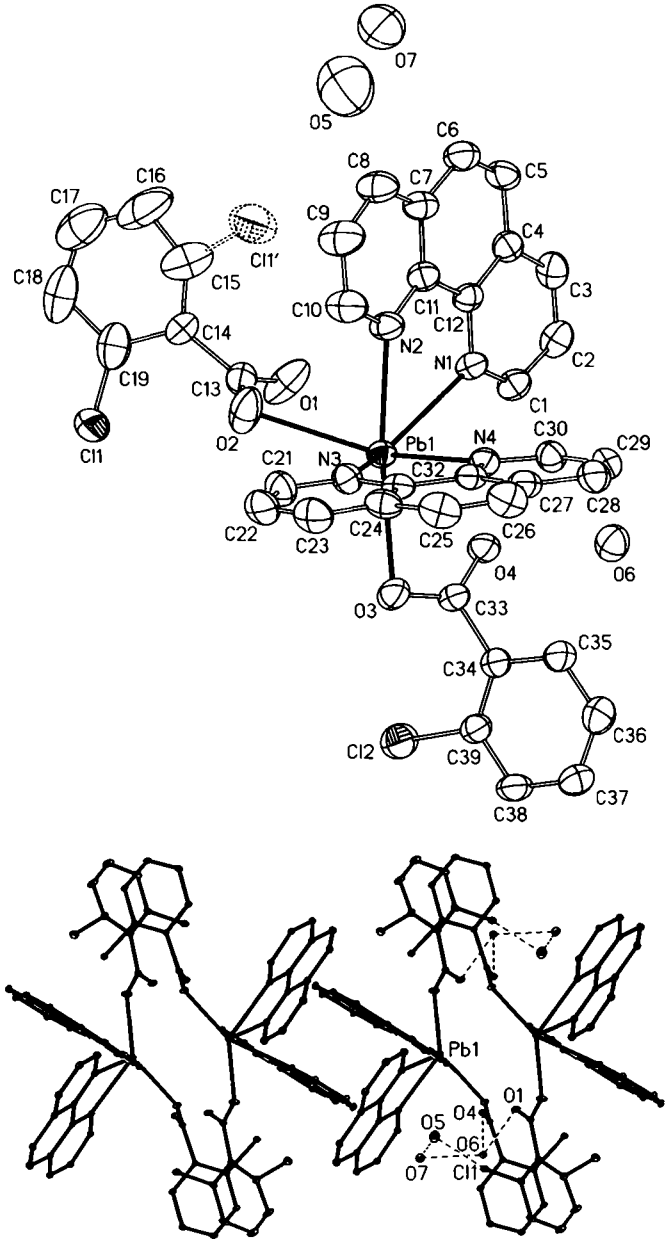

Abstract

$C_{38}H_{29}Cl_2N_4O_{6.5}Pb$, triclinic, $P\bar{1}$ (no. 2), a = 11.548(2) Å, b = 12.532(3) Å, c = 13.661(3) Å, α = 95.34(3)°, β = 112.60(3)°, γ = 103.23(3)°, V = 1740.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.082, T = 293 K.

* Correspondence author (e-mail: zbs_jy@163.com)

Source of material

Freshly prepared $PbCO_3$ (0.20 g, 0.75 mmol), 1,10-phenanthroline (phen · H_2O; 0.12 g, 0.61 mmol), 2-chlorobenzoic acid (0.10 g, 0.64 mmol), 12 mL CH_3OH/H_2O (*v*/*v*, 1:2) were mixed and stirred for ca. 3.5 h. Subsequently, the resulting suspension was heated in a 23 mL Teflon-lined stainless steel autoclave at 423 K for 4 days and 373 K for 3 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 month afforded brown pillar-like crystals.

Experimental details

One 2-chlorophenyl group was found to be disordered in a 2:1 ratio. The site occupancy of the O5 water was fixed at 0.5.

Discussion

The crystal structure of the title compound is similar to the structure of bis[μ-chloro(2,2'-bipyridine-*N*,*N*')(μ-2-chlorobenzoato-*O*)-lead(II)] [1] and consists of $[Pb(phen)_2L_2]$ complex molecules (*HL* = 2-chlorobenzoic acid) and lattice water molecules. Within the complex molecules (figure, top), each Pb atom is coordinated by four N atoms from two bidentate chelating *o*-phenanthroline ligands and two O atoms from two 2-chlorobenzoic acid anion ligands to complete significantly distorted PbN_4O_2 environment with *d*(Pb—N) = 2.593(4) Å – 2.685(4) Å, *d*(Pb—O) = 2.708(3) Å – 2.729(4) Å. The dihedral angle between both phen ligands chelating the Pb atom is 87.32(7)°. Two $[Pb(phen)_2(L)_2]$ complex molecules are interconnected via week Pb···Cl1 interactions with *d*(Pb···Cl1) = 4.043 Å. The lattice water molecules $H_2O(5)$ and $H_2O(6)$ show hydrogen bonding to the carboxylate O atoms of 2-chlorobenzoate anions and the other lattice water molecules with *d*(O···O) = 2.287 Å – 2.810 Å and ∠O–H···O = 133.0° – 164.9°. The interplanar phen-to-phen distances of the π-π stacking interactions (figure, bottom) between adjacent phen ligands are 3.397 Å. Moreover, the solvated water molecules show weak hydrogen bonding interactions with *d*(C21···O5) = 3.38(7) Å, ∠C21–H21···O5 = 158.4(5)°. Through the hydrogen and π-π stacking interactions the molecules are interlinked to form the 3D network.

Table 1. Data collection and handling.

Crystal:	brown, pillar-like, size 0.078 × 0.158 × 0.338 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	50.56 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD II, φ/ω
$2\theta_{max}$:	56.66°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	19554, 8603
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6552
$N(param)_{refined}$:	479
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(5A)	2*i*	0.50	1.2971	0.8011	0.3663	0.307
H(5B)	2*i*	0.50	1.3763	0.8622	0.4037	0.307
H(6A)	2*i*		0.6202	0.4314	0.7553	0.105
H(6B)	2*i*		0.5598	0.3407	0.7308	0.105
H(7A)	2*i*		1.1154	0.6768	0.2120	0.228
H(7B)	2*i*		1.2354	0.6705	0.2473	0.228
H(1)	2*i*		0.5946	0.4516	0.5722	0.066
H(2)	2*i*		0.5804	0.2871	0.4720	0.072
H(3)	2*i*		0.6951	0.2932	0.3663	0.074
H(5)	2*i*		0.8630	0.4061	0.3096	0.073
H(6)	2*i*		1.0051	0.5703	0.3269	0.077
H(8)	2*i*		1.1052	0.7772	0.4069	0.089
H(9)	2*i*		1.1181	0.9294	0.5235	0.106
H(10)	2*i*		0.9856	0.9122	0.6131	0.091
H(15)	2*i*	0.667	0.7294	0.8454	0.3073	0.131
H(16)	2*i*		0.7298	0.9069	0.1640	0.178
H(17)	2*i*		0.6390	1.0452	0.1250	0.160
H(18)	2*i*		0.5151	1.0952	0.2104	0.130
H(19)	2*i*	0.333	0.5107	1.0095	0.3568	0.101
H(21)	2*i*		0.8394	1.0322	0.7180	0.076
H(22)	2*i*		0.9966	1.1862	0.8469	0.087
H(23)	2*i*		1.1669	1.1594	0.9885	0.085
H(25)	2*i*		1.2854	1.0273	1.0820	0.086
H(26)	2*i*		1.3002	0.8523	1.0907	0.089
H(28)	2*i*		1.1911	0.6422	1.0205	0.078
H(29)	2*i*		1.0191	0.4985	0.8956	0.068
H(30)	2*i*		0.8552	0.5391	0.7581	0.065
H(35)	2*i*		0.7853	0.5772	0.9639	0.075
H(36)	2*i*		0.8003	0.5486	1.1322	0.089
H(37)	2*i*		0.6568	0.5907	1.1964	0.083
H(38)	2*i*		0.4932	0.6630	1.0923	0.075

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*		0.72711(2)	0.74392(1)	0.64678(1)	0.0533(1)	0.0465(1)	0.0400(1)	0.01393(7)	0.01992(7)	0.00732(7)
Cl(1)	2*i*	0.667(5)	0.4623(3)	1.0190(3)	0.3734(2)	0.148(3)	0.144(3)	0.094(2)	0.102(2)	0.072(2)	0.055(2)
Cl(1′)	2*i*	0.333	0.7912(5)	0.8348(5)	0.3137(4)	0.102(4)	0.133(5)	0.073(3)	0.066(3)	0.038(3)	0.016(3)
Cl(2)	2*i*		0.4194(2)	0.7190(2)	0.8916(1)	0.0828(9)	0.122(1)	0.083(1)	0.0551(9)	0.0450(8)	0.0465(9)
N(1)	2*i*		0.7256(3)	0.5519(3)	0.5407(3)	0.054(2)	0.050(2)	0.043(2)	0.009(2)	0.024(2)	0.009(2)
N(2)	2*i*		0.8981(4)	0.7536(3)	0.5601(3)	0.068(2)	0.049(2)	0.062(2)	0.006(2)	0.040(2)	0.007(2)
N(3)	2*i*		0.9140(4)	0.9171(3)	0.7819(3)	0.060(2)	0.043(2)	0.054(2)	0.014(2)	0.026(2)	0.006(2)
N(4)	2*i*		0.9203(3)	0.7016(3)	0.7978(3)	0.052(2)	0.046(2)	0.044(2)	0.012(2)	0.019(2)	0.005(2)
O(1)	2*i*		0.5741(4)	0.7575(4)	0.4270(3)	0.112(3)	0.087(3)	0.059(2)	–0.024(2)	0.023(2)	0.022(2)
O(2)	2*i*		0.7097(5)	0.9142(3)	0.5358(3)	0.167(4)	0.057(2)	0.051(2)	0.030(3)	0.024(3)	0.011(2)
O(3)	2*i*		0.6485(4)	0.7669(3)	0.8086(3)	0.113(3)	0.047(2)	0.063(2)	0.021(2)	0.051(2)	0.015(2)
O(4)	2*i*		0.6243(3)	0.5883(2)	0.7518(2)	0.079(2)	0.048(2)	0.051(2)	0.016(2)	0.034(2)	0.005(2)
O(5)	2*i*	0.50	1.299(2)	0.863(1)	0.395(2)	0.17(1)	0.20(2)	0.30(2)	0.06(1)	0.13(2)	0.10(2)
O(6)	2*i*		0.6202(4)	0.3674(3)	0.7739(3)	0.086(2)	0.058(2)	0.063(2)	0.019(2)	0.029(2)	0.012(2)
O(7)	2*i*		1.1906(6)	0.7094(6)	0.2559(5)	0.170(6)	0.203(7)	0.174(6)	0.117(5)	0.118(5)	0.101(5)
C(1)	2*i*		0.6458(4)	0.4539(4)	0.5336(4)	0.053(3)	0.056(3)	0.050(3)	0.007(2)	0.021(2)	0.010(2)
C(2)	2*i*		0.6343(5)	0.3546(4)	0.4717(4)	0.059(3)	0.050(3)	0.055(3)	0.001(2)	0.015(2)	0.010(2)
C(3)	2*i*		0.7040(5)	0.3584(4)	0.4106(4)	0.070(3)	0.045(3)	0.053(3)	0.011(2)	0.013(3)	0.001(2)
C(4)	2*i*		0.7898(5)	0.4604(4)	0.4139(3)	0.059(3)	0.058(3)	0.039(2)	0.022(2)	0.014(2)	0.010(2)
C(5)	2*i*		0.8695(5)	0.4692(4)	0.3552(4)	0.073(3)	0.065(3)	0.046(3)	0.024(3)	0.026(2)	0.001(2)
C(6)	2*i*		0.9533(5)	0.5671(5)	0.3651(4)	0.068(3)	0.080(4)	0.051(3)	0.026(3)	0.029(3)	0.012(3)
C(7)	2*i*		0.9659(4)	0.6672(4)	0.4329(4)	0.050(3)	0.073(3)	0.044(2)	0.017(2)	0.020(2)	0.009(2)
C(8)	2*i*		1.0529(5)	0.7700(5)	0.4448(5)	0.069(3)	0.078(4)	0.079(4)	0.005(3)	0.046(3)	0.008(3)
C(9)	2*i*		1.0598(6)	0.8603(5)	0.5136(5)	0.095(4)	0.065(4)	0.102(5)	–0.015(3)	0.062(4)	0.000(3)
C(10)	2*i*		0.9804(6)	0.8490(5)	0.5682(5)	0.087(4)	0.055(3)	0.085(4)	–0.002(3)	0.053(3)	–0.004(3)
C(11)	2*i*		0.8882(4)	0.6620(4)	0.4922(4)	0.052(3)	0.054(3)	0.045(2)	0.014(2)	0.020(2)	0.008(2)
C(12)	2*i*		0.7973(4)	0.5561(4)	0.4821(3)	0.046(2)	0.056(3)	0.033(2)	0.016(2)	0.013(2)	0.011(2)
C(13)	2*i*		0.6335(5)	0.8584(4)	0.4449(4)	0.071(3)	0.055(3)	0.055(3)	0.023(3)	0.026(3)	0.015(2)
C(14)	2*i*		0.6243(5)	0.9134(4)	0.3507(4)	0.056(3)	0.060(3)	0.048(3)	0.010(2)	0.022(2)	0.019(2)
C(15)	2*i*		0.6816(6)	0.8964(6)	0.2874(6)	0.101(5)	0.137(7)	0.079(5)	0.002(5)	0.046(4)	0.020(4)
C(16)	2*i*		0.688(1)	0.933(1)	0.2031(8)	0.21(1)	0.124(8)	0.123(8)	–0.012(8)	0.112(8)	0.030(6)
C(17)	2*i*		0.632(1)	1.0095(8)	0.1800(7)	0.19(1)	0.084(6)	0.096(6)	–0.017(6)	0.066(7)	0.012(5)
C(18)	2*i*		0.5597(8)	1.0424(6)	0.2317(7)	0.108(5)	0.074(4)	0.091(5)	0.009(4)	–0.008(4)	0.032(4)
C(19)	2*i*		0.5580(6)	0.9905(5)	0.3196(5)	0.072(4)	0.078(4)	0.086(4)	0.021(3)	0.013(3)	0.029(3)
C(21)	2*i*		0.9084(5)	1.0208(4)	0.7756(4)	0.075(3)	0.048(3)	0.066(3)	0.020(2)	0.028(3)	0.006(2)
C(22)	2*i*		1.0033(6)	1.1144(4)	0.8531(5)	0.098(4)	0.038(3)	0.083(4)	0.013(3)	0.048(4)	–0.001(3)
C(23)	2*i*		1.1043(6)	1.0983(4)	0.9365(5)	0.076(4)	0.050(3)	0.073(4)	0.002(3)	0.032(3)	–0.008(3)
C(24)	2*i*		1.1154(5)	0.9901(4)	0.9452(4)	0.055(3)	0.059(3)	0.054(3)	0.006(2)	0.025(2)	–0.006(2)
C(25)	2*i*		1.2202(5)	0.9677(5)	1.0297(4)	0.056(3)	0.073(4)	0.066(3)	0.005(3)	0.017(3)	–0.008(3)
C(26)	2*i*		1.2281(5)	0.8637(5)	1.0364(4)	0.061(3)	0.088(4)	0.055(3)	0.018(3)	0.012(3)	–0.001(3)
C(27)	2*i*		1.1244(5)	0.7684(4)	0.9592(4)	0.056(3)	0.072(3)	0.046(3)	0.025(2)	0.024(2)	0.010(2)
C(28)	2*i*		1.1230(5)	0.6578(5)	0.9655(4)	0.064(3)	0.078(4)	0.060(3)	0.034(3)	0.026(3)	0.020(3)
C(29)	2*i*		1.0219(5)	0.5726(4)	0.8911(4)	0.066(3)	0.053(3)	0.061(3)	0.028(2)	0.030(3)	0.014(2)
C(30)	2*i*		0.9234(5)	0.5980(4)	0.8088(4)	0.058(3)	0.049(3)	0.052(3)	0.017(2)	0.021(2)	0.007(2)
C(31)	2*i*		1.0193(4)	0.7870(4)	0.8731(3)	0.051(2)	0.055(3)	0.044(2)	0.017(2)	0.026(2)	0.008(2)
C(32)	2*i*		1.0149(4)	0.9002(4)	0.8653(3)	0.053(3)	0.051(3)	0.046(2)	0.012(2)	0.028(2)	0.003(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(33)	2*i*		0.6317(4)	0.6678(4)	0.8198(3)	0.053(3)	0.052(3)	0.042(2)	0.013(2)	0.023(2)	0.010(2)
C(34)	2*i*		0.6308(4)	0.6436(3)	0.9257(4)	0.052(2)	0.040(2)	0.048(2)	0.006(2)	0.022(2)	0.005(2)
C(35)	2*i*		0.7274(5)	0.5962(4)	0.9896(4)	0.081(3)	0.054(3)	0.062(3)	0.022(3)	0.039(3)	0.014(2)
C(36)	2*i*		0.7354(6)	0.5782(5)	1.0901(5)	0.084(4)	0.082(4)	0.062(3)	0.036(3)	0.026(3)	0.027(3)
C(37)	2*i*		0.6498(6)	0.6032(5)	1.1286(4)	0.091(4)	0.075(4)	0.049(3)	0.028(3)	0.034(3)	0.017(3)
C(38)	2*i*		0.5527(5)	0.6469(4)	1.0670(4)	0.078(3)	0.069(3)	0.053(3)	0.020(3)	0.040(3)	0.011(2)
C(39)	2*i*		0.5456(4)	0.6663(4)	0.9670(4)	0.057(3)	0.052(3)	0.051(3)	0.013(2)	0.027(2)	0.012(2)

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References

1. Zhang, B.-S.: Crystal structure of bis[μ-chloro(2,2'-bipyridine-*N*,*N*')(μ-2-chlorobenzoato)lead(II)], $[Pb(Cl)(C_{10}H_8N_2)(ClC_6H_4COO)]_2$. Z. Kristallogr. NCS **220** (2005) 73-74.
2. Sheldrick, G. M.: Phase Annealing in SHELXS-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.