Crystal structure of bis(1,10-phenanthroline-*N,N'*)(2-iodo-benzoato)lead(II) 2-iodo-benzoate dihydrate, $[\text{Pb}(\text{C}_7\text{H}_4\text{O}_2\text{I})(\text{C}_{12}\text{H}_8\text{N}_2)_2](\text{C}_7\text{H}_4\text{O}_2\text{I}) \cdot 2\text{H}_2\text{O}$

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Discussion

The title compound consists of monovalent $[\text{Pb}(\text{phen})_2\text{L}]^+$ ($\text{HL} = 2\text{-iodo-benzoic acid}$) complex cations, 2-iodo-benzoate anions and lattice water molecules. Within the complex cations, the Pb atoms are each coordinated by four N atoms from two bidentate chelating *o*-phenanthroline ligands and two O atoms from one 2-iodo-benzoate ligands to complete a distorted PbN_4O_2 environment with $d(\text{Pb}-\text{N}) = 2.480(5) \text{ \AA} - 2.671(7) \text{ \AA}$, $d(\text{Pb}-\text{O}) = 2.531(6) \text{ \AA}$ and $2.642(6) \text{ \AA}$. Two $[\text{Pb}(\text{phen})_2(\text{L})]^+$ complexes are interlinked with each other via weak $\text{Pb}\cdots\text{O}$ and $\text{Pb}\cdots\text{I}$ interactions with $d(\text{Pb}\cdots\text{O}) = 3.019(5) \text{ \AA}$ and $d(\text{Pb}\cdots\text{I}) = 3.771(2) \text{ \AA}$. The 2-iodo-benzoate anions are bound to water molecules through H bonds with $d(\text{O}\cdots\text{O}) = 2.762 \text{ \AA} - 2.841 \text{ \AA}$, $\angle \text{O}-\text{H}\cdots\text{O} = 148.7^\circ - 161.6^\circ$. Through the $\text{Pb}\cdots\text{O}$, $\text{Pb}\cdots\text{I}$ as well as $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{I}$ bonds all molecules are connected to form chains along $[11\bar{1}]$.

Table 1. Data collection and handling.

Crystal:	yellowish block, size $0.20 \times 0.22 \times 0.28 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	61.52 cm^{-1}
Diffraction, scan mode:	Rigaku R-Axis RAPID, ω
$2\theta_{\text{max}}$:	56.52°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	16219, 8391
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4588
$N(\text{param})_{\text{refined}}$:	460
Programs:	SHELXS-97 [1], SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
H(1)	2i	0.2942	0.6482	0.2904	0.071
H(2)	2i	0.1572	0.7561	0.4274	0.081
H(3)	2i	0.1967	0.9253	0.4214	0.076
H(5)	2i	0.3341	1.0646	0.3249	0.084
H(6)	2i	0.4945	1.1180	0.1779	0.077
H(8)	2i	0.6816	1.0798	-0.0054	0.078
H(9)	2i	0.8043	0.9643	-0.1350	0.078
H(10)	2i	0.7602	0.7954	-0.1119	0.074
H(21)	2i	0.3555	0.4428	0.2179	0.100
H(22)	2i	0.3128	0.3009	0.3561	0.117
H(23)	2i	0.4122	0.2625	0.4747	0.133
H(25)	2i	0.5585	0.2934	0.5229	0.130
H(26)	2i	0.7089	0.3810	0.4880	0.140
H(28)	2i	0.8263	0.5361	0.3791	0.121
H(29)	2i	0.8558	0.6802	0.2413	0.109
H(30)	2i	0.7547	0.7099	0.1317	0.094
H(53)	2i	0.3400	0.7803	-0.1778	0.078
H(54)	2i	0.2102	0.8066	-0.2612	0.099
H(55)	2i	0.0548	0.7052	-0.1831	0.109
H(56)	2i	0.0311	0.5850	-0.0304	0.098

Abstract

$\text{C}_{38}\text{H}_{28}\text{I}_2\text{N}_4\text{O}_6\text{Pb}$, triclinic, $P\bar{1}$ (no. 2), $a = 12.727(3) \text{ \AA}$, $b = 12.991(3) \text{ \AA}$, $c = 13.116(3) \text{ \AA}$, $\alpha = 79.83(3)^\circ$, $\beta = 63.31(3)^\circ$, $\gamma = 78.33(3)^\circ$, $V = 1888.3 \text{ \AA}^3$, $Z = 2$, $R_{\text{gt}}(F) = 0.043$, $wR_{\text{ref}}(F^2) = 0.105$, $T = 293 \text{ K}$.

Source of material

Freshly prepared PbCO_3 (0.3 g, 1.12 mmol), 1,10-phenanthroline ($\text{phen} \cdot \text{H}_2\text{O}$; 0.12 g, 0.61 mmol), 2-iodo-benzoic acid (0.11 g, 0.44 mmol), 15 mL $\text{CH}_3\text{OH}/\text{H}_2\text{O}$ (v/v 1:2) were mixed and stirred for ca. 2.5 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless steel autoclave at 423 K for 5 day. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting yellow filtrate was allowed to stand at room temperature and slow evaporation for 1 h afforded crystals. Elemental analyses indicated that title compound possess two lattice H_2O molecules. Elemental analysis: found – C, 41.18 %; H, 2.67 %; N, 5.10 %; calc. for $\text{C}_{38}\text{H}_{28}\text{I}_2\text{N}_4\text{O}_6\text{Pb} - \text{C}$, 41.57 %; H, 2.58 %; N, 5.11 %; Pb, 18.88 %; I, 23.12 %; O, 8.74 %.

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(43)	2i	1.0779	0.8002	0.2374	0.098
H(44)	2i	1.0934	0.8285	0.0496	0.114
H(45)	2i	0.9359	0.9113	0.0174	0.115
H(46)	2i	0.7644	0.9671	0.1645	0.106

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(5A)	2i	0.9720	0.8439	0.6365	0.202
H(5B)	2i	1.0209	0.9375	0.5996	0.202
H(6A)	2i	0.9243	0.6990	0.6122	0.345
H(6B)	2i	0.9151	0.6375	0.7124	0.345

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Pb(1)	2i	0.55475(3)	0.62122(2)	0.06171(2)	0.0542(2)	0.0406(2)	0.0594(2)	−0.0105(1)	−0.0235(1)	−0.0075(1)
I(1)	2i	0.13458(5)	0.49199(5)	0.13303(5)	0.0612(4)	0.0777(4)	0.0917(4)	−0.0216(3)	−0.0287(3)	0.0065(3)
O(1)	2i	0.3720(5)	0.5778(4)	0.0569(5)	0.074(4)	0.050(3)	0.089(4)	−0.023(3)	−0.048(3)	0.012(3)
O(2)	2i	0.4208(6)	0.7294(4)	−0.0381(5)	0.102(5)	0.054(3)	0.101(4)	−0.032(3)	−0.065(4)	0.007(3)
N(1)	2i	0.4108(5)	0.7431(4)	0.2041(5)	0.042(4)	0.045(3)	0.054(3)	−0.016(3)	−0.015(3)	−0.003(3)
N(2)	2i	0.6143(6)	0.8097(4)	0.0270(5)	0.059(4)	0.048(3)	0.054(3)	−0.015(3)	−0.024(3)	−0.002(3)
N(3)	2i	0.4825(6)	0.4832(5)	0.2439(5)	0.061(5)	0.051(4)	0.068(4)	−0.018(4)	−0.015(4)	−0.005(3)
N(4)	2i	0.6537(7)	0.6000(5)	0.2072(6)	0.066(5)	0.056(4)	0.077(4)	−0.003(4)	−0.040(4)	−0.004(4)
C(1)	2i	0.3104(7)	0.7157(6)	0.2873(6)	0.059(5)	0.043(4)	0.071(5)	−0.002(4)	−0.029(5)	0.003(4)
C(2)	2i	0.2266(7)	0.7799(7)	0.3711(6)	0.050(5)	0.087(6)	0.049(4)	−0.002(5)	−0.012(4)	0.001(4)
C(3)	2i	0.2504(8)	0.8800(6)	0.3675(6)	0.064(6)	0.065(5)	0.057(5)	0.008(5)	−0.025(5)	−0.017(4)
C(4)	2i	0.3553(8)	0.9119(5)	0.2824(6)	0.081(6)	0.045(4)	0.051(4)	0.001(4)	−0.036(5)	−0.006(3)
C(5)	2i	0.3842(9)	1.0183(6)	0.2705(7)	0.095(7)	0.051(5)	0.076(5)	0.000(5)	−0.046(6)	−0.017(4)
C(6)	2i	0.4800(8)	1.0493(6)	0.1841(7)	0.085(7)	0.046(4)	0.082(6)	−0.017(5)	−0.051(6)	−0.006(4)
C(7)	2i	0.5619(7)	0.9819(5)	0.1002(6)	0.066(5)	0.039(4)	0.063(4)	−0.017(4)	−0.037(4)	0.001(4)
C(8)	2i	0.6640(8)	1.0117(6)	0.0046(7)	0.090(7)	0.050(4)	0.084(6)	−0.028(5)	−0.062(6)	0.010(4)
C(9)	2i	0.7365(8)	0.9437(7)	−0.0727(7)	0.061(6)	0.074(6)	0.066(5)	−0.030(5)	−0.031(5)	0.012(4)
C(10)	2i	0.7091(8)	0.8422(6)	−0.0585(6)	0.067(6)	0.058(5)	0.059(5)	−0.015(4)	−0.023(5)	−0.005(4)
C(11)	2i	0.5399(7)	0.8779(5)	0.1074(6)	0.054(5)	0.043(4)	0.057(4)	−0.007(4)	−0.035(4)	0.003(3)
C(12)	2i	0.4343(7)	0.8423(5)	0.2002(5)	0.055(5)	0.038(4)	0.047(4)	−0.008(4)	−0.028(4)	0.001(3)
C(21)	2i	0.3973(9)	0.4253(7)	0.2627(7)	0.098(8)	0.062(5)	0.079(6)	−0.012(6)	−0.027(6)	−0.006(5)
C(22)	2i	0.370(1)	0.3409(7)	0.3463(9)	0.099(9)	0.063(6)	0.097(7)	−0.038(6)	−0.008(7)	0.008(6)
C(23)	2i	0.431(1)	0.3170(9)	0.4159(9)	0.14(1)	0.077(7)	0.067(6)	−0.010(8)	−0.014(7)	0.017(6)
C(24)	2i	0.516(1)	0.3748(7)	0.3959(8)	0.096(8)	0.062(6)	0.061(5)	−0.009(6)	−0.019(6)	0.009(5)
C(25)	2i	0.578(1)	0.348(1)	0.4643(9)	0.13(1)	0.094(9)	0.076(7)	−0.016(8)	−0.032(8)	0.031(6)
C(26)	2i	0.665(1)	0.401(1)	0.4454(8)	0.15(1)	0.12(1)	0.053(6)	0.026(9)	−0.038(7)	0.002(6)
C(27)	2i	0.692(1)	0.4908(8)	0.3582(8)	0.069(7)	0.095(7)	0.070(6)	0.001(6)	−0.030(5)	−0.020(5)
C(28)	2i	0.781(1)	0.553(1)	0.3372(9)	0.098(9)	0.122(9)	0.090(7)	0.029(8)	−0.058(7)	−0.031(7)
C(29)	2i	0.801(1)	0.6367(8)	0.2539(9)	0.083(8)	0.093(7)	0.111(8)	−0.012(6)	−0.053(7)	−0.017(6)
C(30)	2i	0.7377(9)	0.6552(7)	0.1905(8)	0.086(7)	0.072(6)	0.105(7)	−0.012(6)	−0.070(6)	0.003(5)
C(31)	2i	0.6306(8)	0.5186(7)	0.2908(6)	0.055(5)	0.067(5)	0.053(4)	0.010(4)	−0.024(4)	−0.015(4)
C(32)	2i	0.5417(8)	0.4576(6)	0.3108(7)	0.060(6)	0.052(5)	0.057(5)	0.001(4)	−0.019(4)	−0.007(4)
C(51)	2i	0.1744(8)	0.6063(6)	−0.0104(7)	0.063(6)	0.058(5)	0.083(5)	−0.005(5)	−0.032(5)	−0.019(4)
C(52)	2i	0.2690(7)	0.6653(6)	−0.0544(6)	0.052(5)	0.044(4)	0.063(4)	−0.004(4)	−0.023(4)	−0.014(4)
C(53)	2i	0.2783(8)	0.7398(6)	−0.1480(7)	0.062(6)	0.063(5)	0.066(5)	−0.012(4)	−0.021(5)	−0.012(4)
C(54)	2i	0.201(1)	0.7568(7)	−0.1985(8)	0.104(9)	0.074(6)	0.078(6)	−0.008(6)	−0.050(6)	−0.002(5)
C(55)	2i	0.108(1)	0.6957(8)	−0.1510(9)	0.109(9)	0.091(7)	0.103(7)	−0.008(7)	−0.075(7)	−0.007(6)
C(56)	2i	0.0944(9)	0.6237(7)	−0.0604(9)	0.075(7)	0.074(6)	0.109(7)	−0.023(5)	−0.051(6)	0.004(6)
C(58)	2i	0.3569(7)	0.6568(6)	−0.0075(6)	0.049(5)	0.041(4)	0.065(4)	−0.008(4)	−0.022(4)	−0.014(4)
I(2)	2i	0.65743(7)	0.98145(7)	0.41392(6)	0.0716(5)	0.1293(6)	0.1089(5)	0.0078(4)	−0.0195(4)	−0.0026(4)
O(3)	2i	0.900(1)	0.7523(9)	0.4748(7)	0.24(1)	0.21(1)	0.120(6)	−0.134(9)	−0.116(8)	0.073(6)
O(4)	2i	0.9117(9)	0.9201(7)	0.4739(6)	0.21(1)	0.126(6)	0.126(6)	0.031(6)	−0.114(7)	−0.053(5)
C(41)	2i	0.8177(8)	0.9214(6)	0.2855(7)	0.063(6)	0.066(5)	0.070(5)	−0.009(5)	−0.033(5)	−0.004(4)
C(42)	2i	0.9078(8)	0.8679(6)	0.3120(6)	0.057(6)	0.073(5)	0.055(5)	−0.012(5)	−0.022(5)	−0.012(4)
C(43)	2i	1.0133(9)	0.8341(7)	0.2233(8)	0.077(7)	0.084(6)	0.084(6)	−0.010(5)	−0.036(6)	−0.006(5)
C(44)	2i	1.023(1)	0.8514(8)	0.1104(9)	0.089(8)	0.085(7)	0.083(7)	−0.007(6)	−0.006(6)	−0.027(5)
C(45)	2i	0.930(1)	0.9003(8)	0.0915(9)	0.13(1)	0.087(7)	0.082(7)	0.001(7)	−0.058(8)	−0.011(6)
C(46)	2i	0.829(1)	0.9339(7)	0.1791(9)	0.097(8)	0.085(7)	0.095(7)	−0.004(6)	−0.055(7)	−0.009(6)
C(48)	2i	0.903(1)	0.846(1)	0.4315(9)	0.078(7)	0.109(9)	0.089(7)	−0.021(7)	−0.040(6)	0.010(7)
O(5)	2i	0.9695(8)	0.9043(6)	0.6540(5)	0.172(9)	0.146(7)	0.085(4)	−0.005(6)	−0.061(5)	−0.013(4)
O(6)	2i	0.929(1)	0.6966(9)	0.6747(9)	0.32(2)	0.23(1)	0.22(1)	−0.15(1)	−0.18(1)	0.110(9)

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