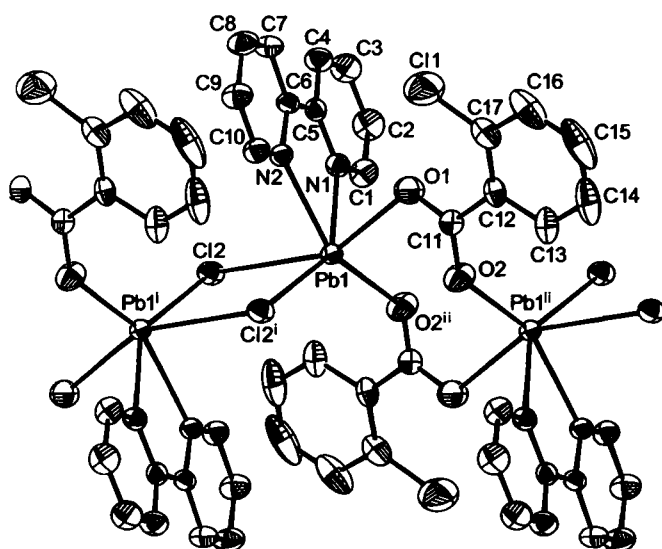


Crystal structure of bis[μ -chloro(2,2'-bipyridine-*N,N'*)(μ -2-chlorobenzoato)lead(II)], [Pb(Cl)(C₁₀H₈N₂)(ClC₆H₄COO)]₂

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Abstract

C₁₇H₁₂Cl₂N₂O₂Pb, triclinic, $P\bar{1}$ (no. 2), $a = 8.523(2)$ Å, $b = 9.508(2)$ Å, $c = 10.709(2)$ Å, $\alpha = 95.55(3)^\circ$, $\beta = 91.46(3)^\circ$, $\gamma = 92.36(3)^\circ$, $V = 862.7$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.025$, $wR_{\text{ref}}(F^2) = 0.057$, $T = 293$ K.

Source of material

Freshly prepared PbCO₃ (0.14 g 0.52 mmol), 2,2'-bipyridine (0.07 g, 0.45 mmol), 2-chloro-benzoic acid (0.08 g, 0.51 mmol), 15 mL CH₃OH/H₂O (1:2 v/v) were mixed and stirred for ca. 3 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless steel autoclave at 423 K for 5 days and 373 K for 2 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 two weeks afforded yellow block crystals.

Discussion

The crystal structure of the title compound is similar to the structure of bis[chlorobis(2,2'-bipyridine)perchloratolead(II)] [1] and consists of chloro- and 2-chlorobenzoato bridged dinuclear [Pb₂(μ -Cl)₂(μ -2-chlorobenzoate)₂(2,2'-bipyridine)₂] complex molecules, in which the Pb atoms are each coordinated by two Cl, two N and two O atoms from one 2,2'-bipyridine (bpy) and two 2-chlorobenzoic acid anion ligands, respectively, to form distorted PbCl₂N₂O₂ octahedra with $d(\text{Pb1}—\text{Cl2}) = 2.948(1)$ Å,

$d(\text{Pb1}—\text{Cl2}^i) = 3.014(1)$ Å [(i) $-x, -y+2, -z+1$], $d(\text{Pb1}—\text{N}) = 2.521(3)$ Å, $2.524(3)$ Å, $d(\text{Pb1}—\text{O1}) = 2.436(3)$ Å and $d(\text{Pb1}—\text{O2}^{ii}) = 2.764(5)$ Å [(ii) $-x+1, -y+2, -z+1$]. Two adjacent PbCl₂N₂O₂ polyhedra are condensed via two bridging Cl atoms to result in an edge-shaped bi-octahedron. The complex [Pb₂(μ -Cl)₂(2,2-bpy)₂(μ -2-chlorobenzoate)₂] molecules are connected with each other via the two chloro (Cl2, Cl2ⁱ) and two oxygen atoms (O1, O2ⁱⁱ) to generate 1D supramolecular chains along [100]. The bridging Cl2 atoms show extensive weak hydrogen bonding to 2,2'-bpy carbon and 2-chlorobenzoic acid anion carbon atoms with $d(\text{C4}—\text{H4} \cdots \text{Cl2}) = 3.59(6)$ Å, $d(\text{C7}—\text{H7} \cdots \text{Cl2}) = 3.71(3)$ Å, $d(\text{C15}—\text{H15} \cdots \text{Cl2}) = 3.70(5)$ Å, $\angle \text{C}—\text{H} \cdots \text{Cl2} = 160.5(2)^\circ - 167.6(2)^\circ$. Through these weak hydrogen bond interactions the compound is interlinked to form the 3D network.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.15 × 0.16 × 0.28 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	100.99 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX CCD II, ϕ/ω
$2\theta_{\text{max}}$:	61.04°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	14933, 5055
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4310
$N(\text{param})_{\text{refined}}$:	218
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.4250	0.7242	0.6774	0.088
H(2)	2i	0.4699	0.4989	0.7303	0.088
H(3)	2i	0.3498	0.3070	0.6126	0.088
H(4)	2i	0.1859	0.3424	0.4445	0.088
H(7)	2i	0.0647	0.3829	0.2818	0.088
H(8)	2i	-0.0923	0.4379	0.1152	0.088
H(9)	2i	-0.1335	0.6731	0.0906	0.088
H(10)	2i	-0.0212	0.8441	0.2335	0.088
H(13)	2i	0.7266	1.0631	0.1798	0.088
H(14)	2i	0.8545	1.0386	-0.0070	0.088
H(15)	2i	0.7843	0.8550	-0.1539	0.088
H(16)	2i	0.5835	0.6959	-0.1188	0.088

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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	0.24233(1)	0.91482(1)	0.47340(1)	0.03727(7)	0.03401(8)	0.03858(9)	−0.00116(4)	0.00311(5)	0.00076(5)
N(1)	2i	0.2834(3)	0.6678(3)	0.5309(3)	0.042(1)	0.041(2)	0.045(2)	0.006(1)	−0.001(1)	0.007(1)
N(2)	2i	0.0864(3)	0.7213(3)	0.3405(3)	0.040(1)	0.035(2)	0.042(2)	0.000(1)	−0.001(1)	0.004(1)
O(1)	2i	0.3988(4)	0.8270(3)	0.2976(3)	0.091(2)	0.062(2)	0.082(3)	−0.013(2)	0.045(2)	−0.004(2)
O(2)	2i	0.5293(4)	1.0298(4)	0.3384(3)	0.078(2)	0.080(2)	0.045(2)	−0.018(2)	−0.001(2)	−0.010(2)
Cl(1)	2i	0.3821(2)	0.6670(2)	0.0660(2)	0.122(1)	0.108(1)	0.113(1)	−0.002(1)	−0.022(1)	−0.021(1)
Cl(2)	2i	−0.0215(1)	0.8929(1)	0.6442(1)	0.0621(5)	0.0415(5)	0.0493(6)	0.0022(4)	0.0121(4)	0.0066(4)
C(1)	2i	0.3771(5)	0.6465(5)	0.6296(4)	0.055(2)	0.060(2)	0.049(2)	0.003(2)	−0.013(2)	0.012(2)
C(2)	2i	0.4041(6)	0.5117(5)	0.6620(5)	0.072(3)	0.068(3)	0.067(3)	0.013(2)	−0.015(2)	0.024(3)
C(3)	2i	0.3329(5)	0.3982(5)	0.5923(5)	0.070(3)	0.051(2)	0.080(3)	0.012(2)	−0.007(2)	0.023(2)
C(4)	2i	0.2362(4)	0.4190(4)	0.4921(4)	0.061(2)	0.040(2)	0.063(3)	0.008(2)	−0.002(2)	0.010(2)
C(5)	2i	0.2142(4)	0.5567(4)	0.4624(4)	0.035(2)	0.037(2)	0.045(2)	0.005(1)	0.005(1)	0.008(2)
C(6)	2i	0.1133(4)	0.5847(4)	0.3541(3)	0.044(2)	0.034(2)	0.038(2)	0.003(1)	0.008(1)	0.000(1)
C(7)	2i	0.0465(5)	0.4767(4)	0.2708(4)	0.065(2)	0.037(2)	0.054(3)	0.003(2)	−0.005(2)	−0.002(2)
C(8)	2i	−0.0468(5)	0.5093(5)	0.1716(4)	0.072(3)	0.056(3)	0.056(3)	−0.004(2)	−0.013(2)	−0.012(2)
C(9)	2i	−0.0718(5)	0.6484(5)	0.1572(4)	0.067(2)	0.057(3)	0.046(2)	0.001(2)	−0.019(2)	0.000(2)
C(10)	2i	−0.0037(4)	0.7499(4)	0.2433(4)	0.049(2)	0.048(2)	0.050(2)	0.001(2)	−0.008(2)	0.005(2)
C(11)	2i	0.4953(4)	0.9203(4)	0.2686(4)	0.045(2)	0.049(2)	0.037(2)	0.002(2)	0.001(2)	0.010(2)
C(12)	2i	0.5749(4)	0.8945(4)	0.1452(4)	0.036(2)	0.069(3)	0.036(2)	0.013(2)	0.001(1)	0.015(2)
C(13)	2i	0.6967(5)	0.9887(6)	0.1200(5)	0.042(2)	0.094(4)	0.068(3)	0.010(2)	0.010(2)	0.034(3)
C(14)	2i	0.7739(6)	0.9742(9)	0.0084(7)	0.057(3)	0.152(7)	0.102(5)	0.032(4)	0.035(3)	0.074(5)
C(15)	2i	0.7315(9)	0.865(1)	−0.0788(6)	0.108(5)	0.204(9)	0.056(4)	0.094(6)	0.041(4)	0.053(5)
C(16)	2i	0.6115(8)	0.7698(8)	−0.0580(5)	0.102(4)	0.150(6)	0.036(3)	0.068(4)	−0.004(3)	−0.005(3)
C(17)	2i	0.5319(5)	0.7838(5)	0.0542(4)	0.062(2)	0.085(3)	0.043(2)	0.026(2)	−0.005(2)	0.001(2)

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