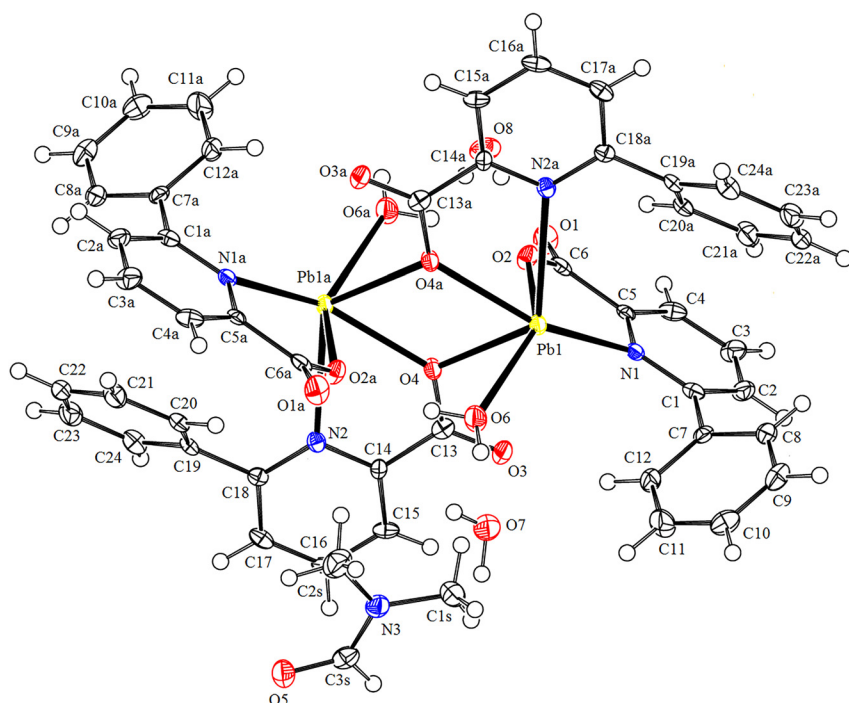


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Crystal structure of diaqua-bis(μ_2 -6-phenylpyridine-2-carboxylate- κ^3 N,O:O)-bis(6-phenylpyridine-2-carboxylato- κ^2 N,O)lead(II) – *N,N*-dimethylformamide – water (1/2/4), $C_{54}H_{58}N_6O_{16}Pb_2$



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Abstract

$C_{54}H_{58}N_6O_{16}Pb_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.4690(6)$ Å, $b = 12.9374(6)$ Å, $c = 13.2303(7)$ Å, $V = 1341.90(15)$ Å³, $\alpha = 63.395(5)^\circ$, $\beta = 74.291(5)^\circ$, $\gamma = 69.113(5)^\circ$, $Z = 1$, $R_{gt}(F) = 0.0284$, $wR_{ref}(F^2) = 0.0484$, $T = 150$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.10 × 0.07 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	6.34 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	8804, 4741, 0.035
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4320
$N(param)_{refined}$:	373
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Pb1	0.99366 (2)	0.60972 (2)	0.57645 (2)	0.01387 (6)
O1	0.7842 (4)	0.3530 (2)	0.8792 (2)	0.0254 (8)
O2	0.9374 (3)	0.4408 (2)	0.7288 (2)	0.0178 (7)
O3	0.6588 (4)	0.6860 (2)	0.5339 (2)	0.0216 (7)
O4	0.8582 (3)	0.5465 (2)	0.4960 (2)	0.0156 (7)
O6	1.0078 (4)	0.7502 (2)	0.3407 (2)	0.0253 (8)
H6A	1.003083	0.819051	0.287017	0.038*
H6B	1.024040	0.703421	0.307319	0.038*
N1	0.7872 (4)	0.6569 (3)	0.7464 (2)	0.0124 (8)
N2	0.7089 (4)	0.5256 (3)	0.3623 (2)	0.0136 (8)
C1	0.7292 (5)	0.7612 (3)	0.7617 (3)	0.0159 (10)
C2	0.6402 (5)	0.7640 (4)	0.8640 (3)	0.0205 (11)
H2	0.601570	0.836180	0.874007	0.025*
C3	0.6089 (5)	0.6617 (4)	0.9498 (3)	0.0217 (11)
H3	0.549660	0.663556	1.018006	0.026*
C4	0.6679 (5)	0.5555 (4)	0.9323 (3)	0.0191 (11)
H4	0.647456	0.484618	0.987993	0.023*
C5	0.7571 (5)	0.5570 (3)	0.8309 (3)	0.0131 (10)
C6	0.8308 (5)	0.4410 (3)	0.8120 (3)	0.0159 (10)
C7	0.7617 (5)	0.8707 (3)	0.6685 (3)	0.0151 (10)
C8	0.8128 (5)	0.9463 (3)	0.6897 (3)	0.0199 (11)
H8	0.831427	0.925136	0.762955	0.024*
C9	0.8367 (5)	1.0528 (4)	0.6036 (4)	0.0248 (12)
H9	0.872561	1.101962	0.618687	0.030*
C10	0.8066 (6)	1.0853 (4)	0.4951 (4)	0.0267 (12)
H10	0.818888	1.157930	0.437681	0.032*
C11	0.7593 (6)	1.0114 (4)	0.4724 (3)	0.0317 (13)
H11	0.742977	1.032760	0.398491	0.038*
C12	0.7346 (5)	0.9039 (3)	0.5580 (3)	0.0228 (11)
H12	0.700369	0.854786	0.541562	0.027*
C13	0.7184 (5)	0.6135 (3)	0.4877 (3)	0.0147 (10)
C14	0.6320 (5)	0.5998 (3)	0.4167 (3)	0.0123 (10)
C15	0.4800 (5)	0.6612 (3)	0.4113 (3)	0.0169 (10)
H15	0.430976	0.711576	0.450411	0.020*
C16	0.4021 (5)	0.6464 (3)	0.3466 (3)	0.0191 (11)
H16	0.299773	0.687505	0.340436	0.023*
C17	0.4788 (5)	0.5693 (3)	0.2911 (3)	0.0178 (10)
H17	0.428015	0.557289	0.247890	0.021*
C18	0.6319 (5)	0.5101 (3)	0.3002 (3)	0.0130 (9)
C19	0.7182 (5)	0.4280 (3)	0.2407 (3)	0.0140 (9)
C20	0.8616 (5)	0.4357 (4)	0.1798 (3)	0.0180 (10)
H20	0.904136	0.491434	0.177534	0.022*
C21	0.9411 (6)	0.3606 (4)	0.1226 (3)	0.0232 (11)
H21	1.036221	0.366967	0.081002	0.028*
C22	0.8805 (6)	0.2767 (4)	0.1270 (3)	0.0270 (12)
H22	0.935414	0.225309	0.089671	0.032*
C23	0.7374 (6)	0.2686 (4)	0.1870 (3)	0.0257 (12)
H23	0.696177	0.212089	0.189420	0.031*
C24	0.6557 (5)	0.3444 (4)	0.2434 (3)	0.0207 (11)
H24	0.559248	0.339380	0.282886	0.025*
O5	0.4868 (4)	0.8623 (2)	0.0557 (2)	0.0252 (8)
N3	0.6264 (5)	0.8713 (3)	0.1661 (3)	0.0207 (9)
C1S	0.6283 (6)	0.9130 (4)	0.2510 (3)	0.0267 (12)
H1SA	0.668858	0.981328	0.214594	0.040*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
H1SB	0.690876	0.849818	0.307195	0.040*
H1SC	0.526218	0.935478	0.287238	0.040*
C2S	0.7710 (6)	0.8150 (4)	0.1147 (4)	0.0336 (13)
H2SA	0.833044	0.869989	0.078194	0.050*
H2SB	0.753437	0.793832	0.059137	0.050*
H2SC	0.822263	0.743870	0.172635	0.050*
C3S	0.4973 (6)	0.8891 (3)	0.1309 (3)	0.0212 (11)
H3S	0.406959	0.925022	0.166535	0.025*
O7	0.9995 (5)	0.9739 (3)	0.1557 (3)	0.0307 (9)
O8	0.7822 (5)	0.1192 (3)	0.9951 (3)	0.0295 (8)
H7A	1.066 (5)	0.944 (3)	0.112 (3)	0.032 (16)*
H7B	0.924 (4)	1.029 (3)	0.116 (4)	0.052 (17)*
H8A	0.787 (6)	0.190 (2)	0.955 (4)	0.049 (17)*
H8B	0.698 (4)	0.117 (5)	0.986 (5)	0.08 (3)*

of the atoms including atomic coordinates and displacement parameters.

Source of material

About 0.1 g 6-phenylpyridine-2-carboxylic acid (0.5 mmol), 0.020 g NaOH (0.5 mmol) and 0.1626 g lead acetate (0.5 mmol) were dissolved in 23 mL water-ethanol-*N,N*-dimethylformamide solution (v:v:v = 10:10:3) at room temperature. White precipitation occurs in the solution. The mixture was stirred for 5 h at 80 °C and continued to react for 4 h. The solution was filtered. After 10 days, the colorless crystals of the title compound were obtained from the filtrate.

Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.96 and O–H = 0.85 Å). Their U_{iso} values were set to $1.2U_{eq}$ or $1.5U_{eq}$ of the parent atoms.

Comment

Many metal complexes containing nitrogen heterocycles may show good properties in fluorescence, biological activity, sensing performance and photocatalytic activity [5, 6]. Many metal complexes containing nitrogen heterocycles similar to that used in the title structure have been synthesized and structural characterized in our group [7–9]. In order to further study the structures of metal complexes containing

nitrogen heterocycles, the title compound has been synthesized by the one-pot synthesis method. The asymmetric unit of the title structure contains one Pb(II) ion, one bidentate 6-phenylpyridine-2-carboxylate ligand, one tridentate 6-phenylpyridine-2-carboxylate ligand, one coordinated water molecule, two lattice water molecules and one uncoordinated DMF molecule. The Pb(II) ion is six-coordinated by three O atoms (O2, O4, O4a), two N atoms (N1 and N2a) from different 6-phenylpyridine-2-carboxylate ligands and one O atom (O6) from coordinated water molecule, which forms a distorted octahedral coordination environment. The complex forms a dimeric structure by the bridge effect of two O atoms (O4, O4a) of carboxylates (see the figure). The Pb(II) complex form a one-dimensional chain structure by the intermolecular $OH \cdots O$ hydrogen bonds. And the one-dimensional chains further form a three-dimensional network structure through the π - π stacking of benzene rings and pyridine rings.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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