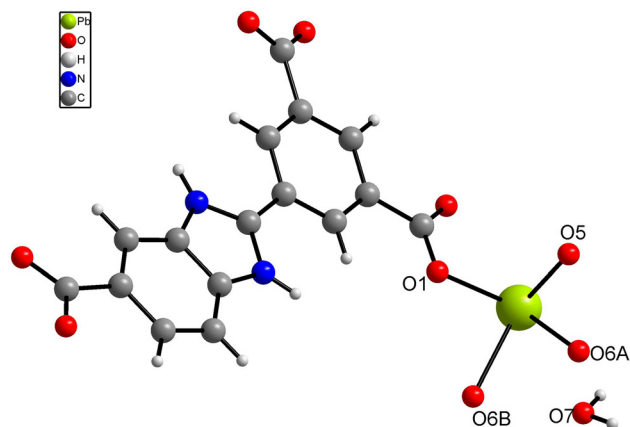


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Crystal structure of poly[(μ_3 -2-(3,5-dicarboxyphenyl) benzimidazole-6-carboxylato- κ^4 O:O:O':O'')lead(II)] monohydrate, $C_{16}H_{10}N_2O_7Pb$



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Abstract

$C_{16}H_{10}N_2O_7Pb$, tetragonal, $I4_1/a$ (no. 88), $a = 15.1344(2)$ Å, $b = 15.1344(2)$ Å, $c = 26.5431(6)$ Å, $V = 6079.7(2)$ Å³, $Z = 16$, $R_{gt}(F) = 0.0419$, $wR_{ref}(F^2) = 0.0991$, $T = 150$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

$Pb(NO_3)_2$ (0.2 mmol 0.0662 g) and 2-(3,5-dicarboxyphenyl)-6-carboxyl 1*H*-benzimidazole (0.1 mmol 0.0326 g) were added sequentially to a 25 mL Teflon reactor liner, then, 9 mL of deionized water and 1 mL of *N,N*-Dimethylformamide in sequence, were added and finally add 50 mL of glacial acetic acid. At room temperature, the reactor stir for

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.2 \times 0.16 \times 0.11$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	11.1 mm^{-1}
Diffraction, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4° , >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	41,734, 3109, 0.060
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2739
$N(\text{param})_{\text{refined}}$:	235
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

half an hour to ensure that the reaction is complete. The inner lining of the stirred reactor is put into the stainless steel reactor, and the reactor is heated to 140 Celsius degree and the reaction temperature is maintained for 3 days. After the reaction is finished, the reactor is allowed to cool naturally to room temperature, the product was rinsed repeatedly with water and DMF (9:1, v/v) and selected to produce block crystals. The selected crystals were dried at 60 °C for 24 h. The calculated yield of the crystal is 60 %.

2 Experimental details

The data were collected and processed using CrysAlis^{PRO} [1]. And the structure was solved by direct methods using Olex2 software [2] and refined with the SHELXL [3]. The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on the parent atoms. The U_{iso} of the H-atoms were set to 1.2 times U_{eq} of the parent atoms with C–H = 0.93 Å (aromatic).

3 Comment

Coordination polymers constructed with N-heterocyclic carboxylic acid ligands have gained much attention due to their rich structures and functionalities [4–7]. Many coordination polymers constructed with N-heterocyclic carboxylic acid ligands have been synthesized and structural characterized in our group [8–11].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
Pb01	−1.36051 (2)	−0.25288 (2)	0.19622 (2)	0.03489 (12)
O1	−1.2516 (4)	−0.2270 (4)	0.1347 (2)	0.0392 (13)
O2	−1.3728 (4)	−0.2461 (4)	0.0887 (2)	0.0402 (13)
O3	−1.3387 (4)	−0.2349 (4)	−0.1014 (2)	0.0488 (15)
O4	−1.2018 (5)	−0.2318 (6)	−0.1307 (2)	0.070 (2)
O5	−0.6507 (4)	0.0434 (4)	−0.0616 (2)	0.0392 (13)
O6	−0.5836 (4)	0.0190 (4)	0.0109 (2)	0.0429 (14)
O7	−1.4080 (4)	−0.2186 (4)	0.2993 (3)	0.073 (2)
H7A	−1.394837	−0.256468	0.321763	0.110*
H7B	−1.455967	−0.196438	0.288363	0.110*
N1	−0.9674 (4)	−0.1218 (4)	0.0585 (2)	0.0324 (14)
H1	−0.987865	−0.135539	0.088540	0.039*
N2	−0.9592 (4)	−0.1026 (4)	−0.0235 (3)	0.0349 (14)
H2	−0.973982	−0.101619	−0.055515	0.042*
C1	−1.2920 (5)	−0.2309 (5)	0.0934 (3)	0.0334 (16)
C2	−1.2373 (5)	−0.2123 (5)	0.0466 (3)	0.0282 (15)
C3	−1.2739 (5)	−0.2261 (5)	−0.0015 (3)	0.0306 (15)
H3	−1.332638	−0.247415	−0.005006	0.037*
C4	−1.2224 (5)	−0.2077 (5)	−0.0440 (3)	0.0333 (16)
C5	−1.2555 (6)	−0.2260 (6)	−0.0964 (3)	0.0420 (19)
C6	−1.1371 (5)	−0.1781 (5)	−0.0386 (3)	0.0333 (17)
H6	−1.102107	−0.167880	−0.067767	0.040*
C7	−1.1007 (5)	−0.1628 (5)	0.0095 (3)	0.0285 (15)
C8	−1.1517 (5)	−0.1817 (5)	0.0518 (3)	0.0290 (15)
H8	−1.127439	−0.173589	0.084455	0.035*
C9	−1.0112 (5)	−0.1295 (5)	0.0147 (3)	0.0293 (15)
C10	−0.8851 (5)	−0.0892 (5)	0.0481 (3)	0.0329 (16)
C11	−0.8142 (5)	−0.0695 (5)	0.0793 (3)	0.0374 (18)
H11	−0.817439	−0.078421	0.114665	0.045*
C12	−0.7391 (5)	−0.0366 (5)	0.0569 (3)	0.0344 (17)
H12	−0.689633	−0.022016	0.077321	0.041*
C13	−0.7339 (5)	−0.0242 (5)	0.0044 (3)	0.0319 (16)
C14	−0.6520 (5)	0.0142 (5)	−0.0173 (3)	0.0325 (16)
C15	−0.8032 (5)	−0.0437 (5)	−0.0269 (3)	0.0327 (16)
H15	−0.799646	−0.035106	−0.062330	0.039*
C16	−0.8792 (5)	−0.0768 (5)	−0.0040 (3)	0.0306 (15)

As shown in the figure, the asymmetric unit of the title structure contains one Pb(II) ion, one 2-(3,5-dicarboxyphenyl)-6-carboxyl 1*H*-benzimidazole ligand and one lattice water molecule. The Pb(II) ion is four-coordinated by four O atoms from different 2-(3,5-dicarboxyphenyl)-6-carboxyl 1*H*-benzimidazole ligands. The Pb–O bond lengths are in the range of 2.354(5)–2.618(6) Å, which are comparable to other lead(II) complexes [12]. The O–Pb–O angles are ranging from 52.52(19) to 100.48(18) degrees. Two adjacent lead ions are bridged by two oxygen atoms to form a binuclear unit, each binuclear lead structural unit is surrounded by four aromatic carboxylic acid ligands, which results in a three-dimensional framework.

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