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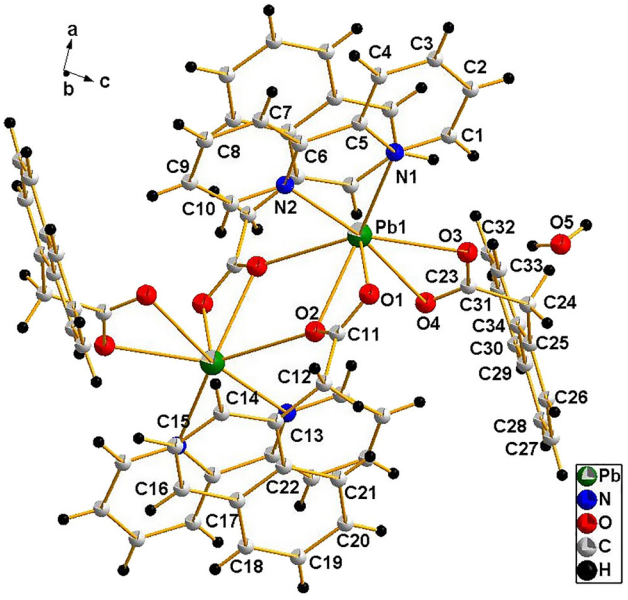
The crystal structure of di-μ-1-naphthylacetato-$\kappa^3O,O':O;\kappa^3O:O,O'$-bis[(1-naphthylacetato-$\kappa^2O,O'$)(2,2′-bipyridine-$\kappa^2N,N'$)lead(II)] monohydrate, $C_{68}H_{54}N_4O_9Pb_2$



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Abstract

$C_{68}H_{54}N_4O_9Pb_2$, monoclinic, $C2/c$ (no. 15), a = 15.7286(14) Å, b = 16.3623(14) Å, c = 22.500(2) Å, β = 97.369(1)°, V = 5742.7(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0203, $wR_{ref}(F^2)$ = 0.0488, T = 296(2) 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.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.28 × 0.21 × 0.19 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	5.92 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	31,054, 5625, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4987
$N(param)_{refined}$:	382
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

1 Source of material

All chemicals were purchased from commercial sources and used as received. An amount of 2 mmol 1-naphthylacetic acid, 1 mmol 2,2′-bipyridine and 1 mmol lead acetate were added in CH_3OH (50 mL) and refluxed with agitating for 10 h. Then, the reaction solution was filtered. Finally, the title crystal was precipitated by controlling solvent volatilization.

2 Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5U_{eq} (parent C-atom).

3 Comment

The Pb(II) ions can adopt diverse coordination numbers from 2 to 10 and rich coordination geometry deriving from large radius and the possible occurrence of a stereochemically active lone pair of electrons [5, 6]. The design and synthesis of lead complexes have attracted considerable attention in recent years [7–9]. Carboxylate ligands containing aromatic ring are especially interesting not only because of their various coordination modes to metal ions, resulting from

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.88126 (2)	0.77420 (2)	0.05677 (2)	0.03085 (4)
N1	1.02559 (15)	0.85844 (16)	0.06286 (11)	0.0377 (6)
N2	0.92087 (16)	0.84943 (15)	−0.04092 (11)	0.0384 (6)
O1	0.81692 (13)	0.91005 (13)	0.06707 (10)	0.0480 (5)
O2	0.72601 (14)	0.81685 (14)	0.03091 (13)	0.0609 (7)
O3	0.91513 (19)	0.79099 (16)	0.17035 (11)	0.0646 (7)
O4	0.81266 (18)	0.7024 (2)	0.14979 (11)	0.0830 (9)
C1	1.0779 (2)	0.8575 (2)	0.11425 (15)	0.0523 (9)
H1	1.059020	0.832472	0.147274	0.063*
C2	1.1581 (2)	0.8915 (3)	0.12104 (18)	0.0650 (11)
H2	1.192964	0.889247	0.157675	0.078*
C3	1.1858 (2)	0.9288 (3)	0.07276 (19)	0.0689 (12)
H3	1.239801	0.952673	0.076100	0.083*
C4	1.1329 (2)	0.9306 (2)	0.01911 (16)	0.0553 (9)
H4	1.150889	0.955978	−0.014121	0.066*
C5	1.05284 (18)	0.89448 (17)	0.01499 (13)	0.0351 (6)
C6	0.99210 (18)	0.89373 (17)	−0.04146 (13)	0.0334 (6)
C7	1.0091 (2)	0.9364 (2)	−0.09191 (14)	0.0455 (8)
H7	1.058387	0.968041	−0.090890	0.055*
C8	0.9519 (2)	0.9314 (2)	−0.14343 (16)	0.0585 (10)
H8	0.962417	0.959145	−0.177816	0.070*
C9	0.8787 (2)	0.8845 (3)	−0.14353 (16)	0.0603 (10)
H9	0.839207	0.879838	−0.177831	0.072*
C10	0.8661 (2)	0.8451 (2)	−0.09131 (15)	0.0512 (9)
H10	0.816782	0.813738	−0.091144	0.061*
C11	0.74281 (19)	0.88856 (19)	0.04615 (14)	0.0384 (7)
C12	0.6716 (2)	0.9520 (2)	0.03851 (17)	0.0511 (9)
H12A	0.658453	0.967665	0.077891	0.061*
H12B	0.692586	1.000215	0.019991	0.061*
C13	0.58953 (19)	0.92437 (19)	0.00127 (15)	0.0431 (7)
C14	0.5754 (2)	0.9423 (2)	−0.05832 (16)	0.0513 (8)
H14	0.616364	0.972351	−0.075238	0.062*
C15	0.5013 (2)	0.9169 (2)	−0.09501 (17)	0.0580 (9)
H15	0.494548	0.929828	−0.135598	0.070*
C16	0.4402 (2)	0.8745 (2)	−0.07245 (17)	0.0563 (9)
H16	0.391348	0.858008	−0.097248	0.068*
C17	0.4503 (2)	0.8546 (2)	−0.01029 (18)	0.0511 (9)
C18	0.3859 (3)	0.8129 (3)	0.0148 (2)	0.0744 (12)
H18	0.335671	0.798122	−0.009286	0.089*
C19	0.3961 (4)	0.7942 (3)	0.0735 (3)	0.0898 (16)
H19	0.352494	0.766683	0.089402	0.108*
C20	0.4707 (4)	0.8149 (3)	0.1111 (2)	0.0828 (14)
H20	0.476930	0.800330	0.151371	0.099*
C21	0.5343 (3)	0.8569 (2)	0.08828 (18)	0.0635 (10)
H21	0.583697	0.871223	0.113354	0.076*
C22	0.5258 (2)	0.87899 (19)	0.02627 (15)	0.0426 (7)
C23	0.8629 (2)	0.7393 (2)	0.18546 (14)	0.0513 (8)
C24	0.8660 (2)	0.7233 (2)	0.25297 (16)	0.0560 (10)
H24A	0.923828	0.706688	0.268313	0.067*
H24B	0.855058	0.774672	0.272179	0.067*
C25	0.8053 (2)	0.6607 (2)	0.27210 (13)	0.0473 (8)
C26	0.7379 (3)	0.6857 (3)	0.30056 (17)	0.0621 (10)
H26	0.729828	0.741313	0.306102	0.075*
C27	0.6807 (3)	0.6303 (3)	0.3216 (2)	0.0805 (13)
H27	0.634918	0.649638	0.339923	0.097*
C28	0.6917 (3)	0.5500 (3)	0.31562 (19)	0.0748 (12)
H28	0.653832	0.513822	0.330323	0.090*
C29	0.7594 (2)	0.5194 (2)	0.28750 (15)	0.0553 (9)
C30	0.7734 (3)	0.4346 (3)	0.2823 (2)	0.0792 (13)
H30	0.737981	0.397462	0.298540	0.095*
C31	0.8397 (4)	0.4070 (3)	0.2530 (3)	0.0978 (18)
H31	0.849514	0.351219	0.249870	0.117*
C32	0.8914 (3)	0.4625 (4)	0.2284 (3)	0.0970 (18)
H32	0.934518	0.443183	0.207440	0.116*
C33	0.8813 (2)	0.5434 (3)	0.23390 (18)	0.0706 (12)
H33	0.917995	0.578858	0.217337	0.085*
C34	0.8162 (2)	0.5753 (2)	0.26422 (14)	0.0471 (8)
O5[a]	0.988 (2)	0.9125 (3)	0.251 (2)	0.062 (5)
H5A[a]	1.025816	0.887528	0.274990	0.093*
H5B[a]	0.967453	0.876234	0.226540	0.093*

[a]Occupancy: 0.5.

completely or partially deprotonated sites allowing for the large diversity of topologies, but also because of their abilities to act as H–bond acceptors and donors to assemble various supramolecular structures [10]. On the other hand, the chelating aromatic ligands 2,2′-bipy as co-ligand can effectively increase the thermal stability and fluorescent properties, due to the π···π stacks between the neighboring pyridine rings [10].

Single-crystal structure analysis reveals that the title compound crystallizes in the monoclinic space group *C*2/*c*. The Pb(II) ion is seven-coordinated by five oxygen atoms of three carboxyl groups and two nitrogen atoms of one 2,2′-bipy. Bond lengths and angles are all in the expected ranges [11]. The Pb–O bond lengths, O–Pb–O angles and N–Pb–O angles are ranging from 2.466(2) to 2.847(3) Å, 48.28(8)° to 141.04(8)° and 78.64(8)° to 170.41(8)°, respectively. The bond lengths of Pb1–N1 is 2.644(2) Å, and Pb1–N2 are 2.662(2) Å. The title compound forms a 3D structure by C–H···O and O–H···O hydrogen bonds.

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

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