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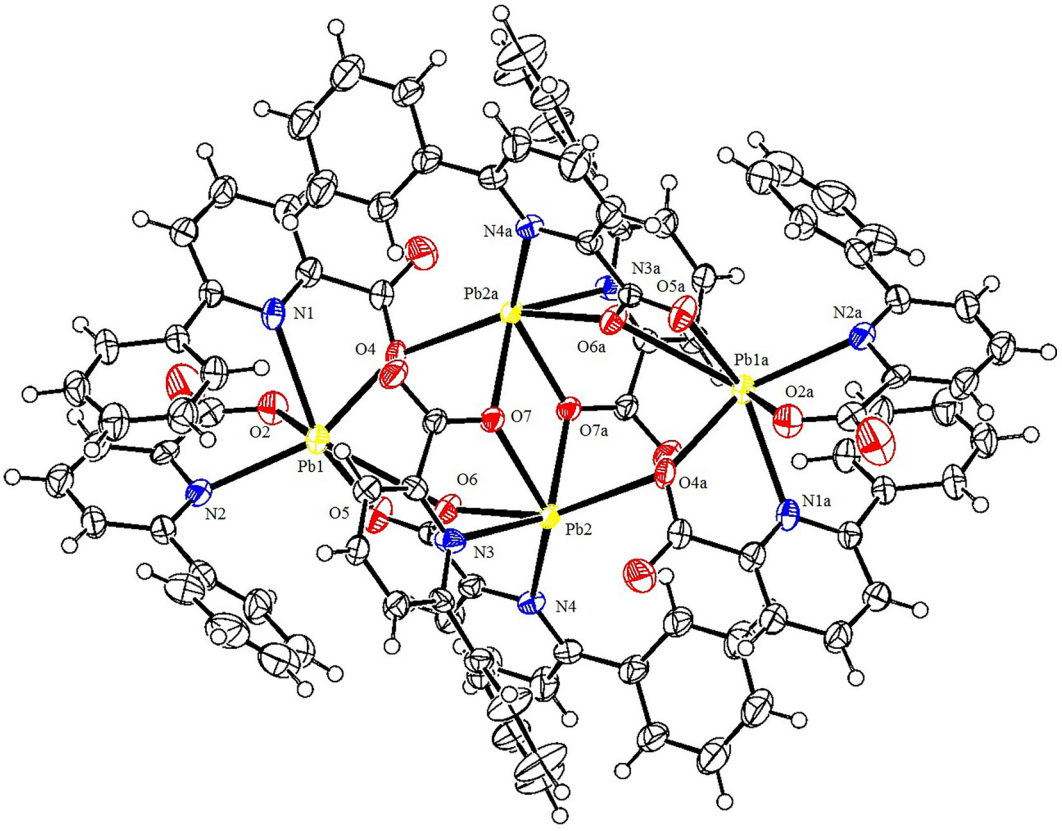
The crystal structure of tetrakis(6-phenylpyridine-2-carboxylate-κ^2N,O)-bis(μ_2-6-phenylpyridine-2-carboxylate-$\kappa^2O:O'$)-bis(μ_2-6-phenylpyridine-2-carboxylate-κ^3N,O:O)tetralead(II) $C_{48}H_{32}N_4O_8Pb_2$



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Abstract

$C_{48}H_{32}N_4O_8Pb_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.2712(8) Å, b = 14.5959(13) Å, c = 16.6178(13) Å, α = 76.089(7)°, β = 77.068(7)°, γ = 74.717(7)°, V = 2074.5(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0563, $wR_{ref}(F^2)$ = 0.1783, T = 250 K.

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Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.14 × 0.11 × 0.09 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	8.17 mm^{-1}
Diffractometer, scan mode:	SuperNova, *ω*
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13595, 7291, 0.048
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5570
$N(param)_{refined}$:	559
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.58527 (3)	0.73130 (2)	0.19173 (2)	0.02710 (7)
Pb2	0.64948 (3)	0.94295 (2)	−0.09748 (2)	0.02269 (7)
O1	0.3359 (7)	0.5273 (5)	0.3657 (5)	0.0720 (16)
O2	0.4072 (6)	0.6410 (4)	0.2625 (3)	0.0376 (10)
O3	0.1470 (6)	0.9375 (4)	0.2349 (3)	0.0511 (13)
O4	0.3688 (5)	0.8562 (4)	0.1799 (3)	0.0305 (10)
O5	0.5303 (6)	0.6746 (4)	0.0755 (3)	0.0409 (11)
O6	0.6073 (6)	0.8055 (3)	0.0099 (3)	0.0324 (9)
O7	0.6180 (5)	0.9964 (3)	0.0323 (3)	0.0267 (9)
O8	0.6667 (6)	0.9584 (5)	0.1632 (3)	0.0487 (17)
N1	0.4558 (6)	0.7991 (4)	0.3315 (4)	0.0291 (9)
N2	0.7003 (7)	0.5544 (5)	0.2680 (3)	0.0347 (11)
N3	0.8865 (6)	0.8869 (4)	−0.0210 (4)	0.0311 (9)
N4	0.5872 (6)	0.7682 (4)	−0.1425 (3)	0.0258 (10)
C1	0.4998 (8)	0.7694 (5)	0.4078 (4)	0.0271 (7)
C2	0.3978 (8)	0.7783 (5)	0.4820 (5)	0.0339 (7)
H2	0.429946	0.756813	0.534460	0.041*
C3	0.2483 (9)	0.8195 (6)	0.4761 (5)	0.0431 (18)
H3	0.176669	0.824166	0.525735	0.052*
C4	0.2004 (10)	0.8543 (6)	0.3993 (5)	0.0429 (17)
H4	0.099482	0.886397	0.394981	0.051*
C5	0.3086 (8)	0.8393 (5)	0.3298 (4)	0.0327 (13)
C6	0.2692 (7)	0.8811 (5)	0.2411 (4)	0.0240 (9)
C7	0.6648 (8)	0.7342 (5)	0.4050 (4)	0.0262 (9)
C8	0.7219 (8)	0.6583 (5)	0.4682 (4)	0.0348 (11)
H8	0.656012	0.628376	0.512045	0.042*
C9	0.8774 (9)	0.6291 (6)	0.4639 (5)	0.0419 (13)
H9	0.916894	0.578519	0.505540	0.050*
C10	0.9758 (10)	0.6718 (6)	0.4005 (5)	0.0489 (12)
H10	1.080869	0.650547	0.400045	0.059*
C11	0.9235 (9)	0.7449 (6)	0.3380 (5)	0.0445 (13)
H11	0.991736	0.772674	0.294066	0.053*
C12	0.7681 (8)	0.7772 (5)	0.3405 (5)	0.0356 (13)
H12	0.731045	0.828538	0.298619	0.043*
C13	0.7056 (8)	0.9596 (5)	0.0861 (4)	0.0286 (12)
C14	0.8651 (7)	0.9089 (4)	0.0540 (4)	0.0236 (8)
C15	0.9783 (8)	0.8968 (5)	0.0981 (4)	0.0299 (12)
H15	0.956555	0.911100	0.152307	0.036*
C16	1.1274 (8)	0.8627 (5)	0.0606 (4)	0.0310 (11)
H16	1.208447	0.852855	0.089268	0.037*
C17	1.1535 (8)	0.8436 (6)	−0.0201 (4)	0.0354 (11)
H17	1.253110	0.823518	−0.047883	0.042*
C18	1.0308 (7)	0.8545 (5)	−0.0592 (4)	0.0274 (8)
C19	1.0542 (8)	0.8247 (5)	−0.1417 (4)	0.0304 (6)
C20	1.1956 (12)	0.8190 (8)	−0.2813 (6)	0.0745 (9)
H20	1.271840	0.836838	−0.325755	0.089*
C21	1.1692 (10)	0.8517 (7)	−0.2070 (5)	0.0558 (11)
H21	1.227984	0.891936	−0.200099	0.067*
C22	1.1058 (11)	0.7581 (7)	−0.2892 (6)	0.0674 (10)
H22	1.124241	0.734397	−0.339255	0.081*
C23	0.9962 (10)	0.7338 (6)	−0.2277 (5)	0.0478 (10)
H23	0.937364	0.693820	−0.234942	0.057*
C24	0.9675 (8)	0.7665 (5)	−0.1535 (4)	0.0306 (7)
H24	0.888775	0.749173	−0.110556	0.037*
C25	0.5657 (7)	0.7299 (5)	0.0094 (4)	0.0242 (7)
C26	0.5594 (8)	0.7032 (5)	−0.0710 (4)	0.0264 (8)
C27	0.5299 (9)	0.6182 (5)	−0.0714 (5)	0.0382 (9)
H27	0.509966	0.575563	−0.020055	0.046*
C28	0.5282 (11)	0.5927 (6)	−0.1442 (5)	0.0538 (15)
H28	0.505782	0.533388	−0.143829	0.065*
C29	0.5590 (10)	0.6534 (6)	−0.2162 (5)	0.0450 (11)
H29	0.561521	0.636160	−0.267569	0.054*
C30	0.5879 (9)	0.7438 (5)	−0.2159 (4)	0.0332 (9)
C31	0.6177 (9)	0.8145 (5)	−0.2951 (4)	0.0353 (12)
C32	0.7132 (10)	0.7835 (6)	−0.3661 (5)	0.0443 (16)
H32	0.761202	0.717588	−0.362019	0.053*
C33	0.7393 (11)	0.8451 (6)	−0.4410 (5)	0.0557 (19)
H33	0.804175	0.821904	−0.487515	0.067*
C34	0.6714 (11)	0.9388 (6)	−0.4474 (5)	0.0562 (16)
H34	0.688965	0.982502	−0.498383	0.067*
C35	0.5736 (11)	0.9714 (6)	−0.3772 (5)	0.0539 (18)
H35	0.520835	1.036450	−0.382682	0.065*
C36	0.5539 (9)	0.9113 (5)	−0.3025 (5)	0.0404 (16)
H36	0.496043	0.935879	−0.255065	0.049*
C37	0.8497 (8)	0.5222 (5)	0.2733 (4)	0.0329 (12)
C38	0.8934 (9)	0.4644 (5)	0.3472 (5)	0.0375 (12)
H38	0.996600	0.446049	0.352142	0.045*
C39	0.7858 (9)	0.4332 (6)	0.4139 (5)	0.0424 (14)
H39	0.815169	0.391972	0.463219	0.051*
C40	0.6347 (9)	0.4646 (6)	0.4058 (5)	0.0427 (14)
H40	0.559119	0.445246	0.450003	0.051*
C41	0.5960 (8)	0.5237 (5)	0.3337 (4)	0.0316 (11)
C42	0.4342 (8)	0.5662 (5)	0.3217 (5)	0.0350 (9)
C43	0.9561 (9)	0.5539 (6)	0.1980 (5)	0.0396 (10)
C44	0.9183 (10)	0.5616 (6)	0.1208 (5)	0.0455 (14)
H44	0.826789	0.547093	0.118136	0.055*
C45	1.0149 (10)	0.5910 (6)	0.0457 (6)	0.0513 (11)
H45	0.991925	0.595307	−0.007612	0.062*
C46	1.1495 (11)	0.6136 (7)	0.0556 (7)	0.0614 (11)
H46	1.215949	0.634266	0.006890	0.074*
C47	1.1856 (11)	0.6071 (7)	0.1285 (7)	0.0665 (16)
H47	1.273283	0.626164	0.131376	0.080*
C48	1.0921 (9)	0.5713 (6)	0.2036 (6)	0.0531 (12)
H48	1.122621	0.559751	0.256104	0.064*

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The Pb(II) complex was synthesized by the following method: 0.0996 g (0.50 mmol) 6-phenylpyridine-2-carboxylic acid, 0.1897 g (0.50 mmol) $Pb(CH_3COO)_2.3H_2O$, and 0.040 g (0.50 mmol) NaOH were added to a solution containing 15 mL 95% ethanol and 15 mL water with stirring. The mixture was heated to 80 °C and the reaction was continued for 6 h. After the reaction was stopped and cooled to room temperature, the above mixture was filtered. The filtrate was transferred to a small beaker. The colorless needle crystals of the title compound were obtained in 22 days.

Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.94 Å). Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms. RIGU options were used to stabilize the refinement [3].

Comment

Lead(II) complexes have shown excellent potential applications in many aspects such as hydrogen storage capacity [5], fluorescence properties [6], antibacterial activities [7], silico analysis and physicochemical properties [8], and photochromism and metallomesogenic properties [9]. Our research group has been working on the synthesis, structure and properties of related metal complexes using the ligand used in the title compound [10–16].

The title structure of 1 was determined by X-ray single-crystal diffraction. The molecular structure is given in the figure. The title complex contains four lead(II) ions and eight 6-phenylpyridine-2-carboxylate (PPC) ligands. As shown in the figure, both Pb1 and Pb1a atoms are six-coordinated with four carboxylate oxygen atoms (O2, O4, O5, O6,or O2a, O4a, O5a, O6a) and two nitrogen atoms (N1, N2, or N1a, N2a) from six different PPC ligands, respectively. And both Pb2 and Pb2a atoms also are six-coordinated with four carboxylate oxygen atoms (O4a, O6, O7, O7a, or O4, O6a, O7, O7a) and two nitrogen atoms (N3, N4, or N3a, N4a) from four different PPC ligands, respectively. All Pb atoms adopt a distorted octahedral coordination polyhedron. The Pb–O and Pb–N distances are in the range of 2.319(5)–2.491(4) Å and 2.629(6)–2.655(6) Å, which are comparable to other lead(II) complexes. Thus the title complex is a tetranuclear species, The lead (II) complex forms the 3D supramolecular framework by weak interaction of phenyl 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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