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The crystal structure of diaqua-bis(μ_2 -3-((3-acetyl-5-carboxyphenyl)oxidophosphoryl)-5-carboxybenzoato- $\kappa^2 O:O'$)bis(5,5'-dimethyl-2,2'-bipyridine- $\kappa^2 N, N'$)zinc(II), $C_{56}H_{46}N_4O_{22}P_2Zn_2$

The dinuclear title 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.

Table 1: Data collection and handling.

Crystal:	Colorless prism
Size:	0.26 × 0.22 × 0.21 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	2.39 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	65.5°, 97%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11995, 4601, 0.014
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4464
$N(\text{param})_{\text{refined}}$:	393
Programs:	Bruker [1], Diamond [2], SHELX [3, 4]

Source of material

A mixture of $ZnSO_4 \cdot 7H_2O$ (0.0386 g), tri(3,5-dicarboxylic phenyl)phosphine oxide (0.0249 g), 5,5'-methyl-2,2'-bipyridine (0.0153 g) and water (10 mL) was sealed in a 15 mL Teflon-lined steel autoclave and heated at 423 K for three days and then cooled to room temperature. Colorless block crystals were obtained (yield ca. 37% based on phosphine). The product is insoluble in water and common organic solvents. FT-IR (KBr, cm^{-1}): 3450 w, 1714 s, 1692 m, 1597 m, 1544 m, 1509 m, 1409 s, 1331 m, 1279 s, 1234 m, 1129 m, 1059 m, 824 m, 753 m, 687 m.

Experimental details

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $Csp^2-H = 0.93$ Å with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$, $Csp^3-H = 0.96$ Å with $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ and $O-H = 0.85$ Å with $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(O)$, respectively.

Comment

The design and construction of coordination polymers are attracting interest due to their specific structures and potential applications [5–7]. In order to achieve these goals, it is necessary to select suitable ligands like phosphine oxides [8–10]. For example, in our previous works, a series of

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Abstract

$C_{56}H_{46}N_4O_{22}P_2Zn_2$, monoclinic, $P2_1/c$ (no. 14), $a = 9.6537(4)$ Å, $b = 31.1374(11)$ Å, $c = 9.8927(4)$ Å, $\beta = 112.709(2)^\circ$, $V = 2743.13(19)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0341$, $wR_{\text{ref}}(F^2) = 0.0934$, $T = 296(2)$ K.

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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}
Zn1	0.56539(3)	0.05904(2)	0.41400(3)	0.03230(11)
P1	0.55669(5)	0.13201(2)	0.18781(5)	0.02594(13)
O1	0.62563(19)	0.00431(6)	−0.13473(19)	0.0504(4)
O2	0.41927(18)	0.00032(5)	−0.33994(17)	0.0436(4)
O3	0.0381(2)	0.11430(8)	−0.4553(2)	0.0781(7)
O4	0.0456(2)	0.16065(6)	−0.2826(2)	0.0573(5)
H4	−0.030432	0.169954	−0.347643	0.086*
O5	0.56429(16)	0.27494(5)	−0.09109(17)	0.0404(4)
H5	0.542523	0.298041	−0.133937	0.061*
O6	0.79124(19)	0.30468(5)	0.0047(2)	0.0522(5)
O7	1.18952(17)	0.18832(6)	0.1758(2)	0.0514(4)
H7	1.271495	0.176999	0.194215	0.077*
O8	1.11941(18)	0.12134(6)	0.1963(2)	0.0564(5)
O9	0.63644(16)	0.09667(5)	0.29121(15)	0.0332(3)
O10	0.45649(15)	0.16175(5)	0.23122(16)	0.0328(3)
O11	0.33616(18)	0.05716(5)	0.2936(2)	0.0459(4)
H11A	0.328442	0.037761	0.230658	0.069*
H11B	0.276688	0.077719	0.253040	0.069*
N1	0.5413(2)	0.10551(6)	0.5605(2)	0.0394(4)
N2	0.7580(2)	0.04855(6)	0.6040(2)	0.0397(4)
C1	0.4490(2)	0.10918(6)	0.0100(2)	0.0292(4)
C2	0.5065(2)	0.07334(7)	−0.0337(2)	0.0317(4)
H2	0.595561	0.061152	0.030397	0.038*
C3	0.4332(2)	0.05536(6)	−0.1716(2)	0.0324(4)
C4	0.2998(2)	0.07372(7)	−0.2657(2)	0.0359(5)
H4A	0.249685	0.061925	−0.358184	0.043*
C5	0.2402(2)	0.10934(7)	−0.2238(2)	0.0358(5)
C6	0.3149(2)	0.12736(7)	−0.0851(2)	0.0336(4)
H6	0.275423	0.151311	−0.056535	0.040*
C7	0.4995(2)	0.01679(7)	−0.2173(2)	0.0348(5)
C8	0.0982(3)	0.12776(8)	−0.3334(3)	0.0470(6)
C9	0.6958(2)	0.16556(6)	0.1588(2)	0.0280(4)
C10	0.6537(2)	0.20610(7)	0.0967(2)	0.0305(4)
H10	0.555763	0.215553	0.073252	0.037*
C11	0.7557(2)	0.23263(7)	0.0692(2)	0.0319(4)
C12	0.9020(2)	0.21843(7)	0.1032(2)	0.0352(5)
H12	0.971957	0.236359	0.088488	0.042*
C13	0.9431(2)	0.17740(7)	0.1591(2)	0.0342(4)
C14	0.8410(2)	0.15118(7)	0.1891(2)	0.0326(4)
H14	0.870287	0.124049	0.229323	0.039*
C15	0.7077(2)	0.27465(7)	−0.0061(2)	0.0336(4)
C16	1.0931(2)	0.15930(7)	0.1801(3)	0.0379(5)
C17	0.4295(3)	0.13402(8)	0.5280(3)	0.0465(6)
H17	0.343459	0.129308	0.444390	0.056*
C18	0.4343(3)	0.17020(9)	0.6115(3)	0.0539(7)
C19	0.5634(4)	0.17554(10)	0.7349(3)	0.0671(8)
H19	0.573315	0.199540	0.793852	0.081*
C20	0.6776(4)	0.14602(10)	0.7723(3)	0.0639(8)
H20	0.763297	0.149616	0.857105	0.077*
C21	0.6642(3)	0.11076(8)	0.6824(2)	0.0439(5)
C22	0.7803(3)	0.07696(8)	0.7127(2)	0.0434(5)
C23	0.9044(3)	0.07341(10)	0.8433(3)	0.0578(7)
H23	0.919800	0.093130	0.918321	0.069*
C24	1.0051(3)	0.04031(10)	0.8609(3)	0.0616(8)
H24	1.088915	0.037895	0.947982	0.074*
C25	0.9825(3)	0.01080(9)	0.7505(3)	0.0516(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C26	0.8561(3)	0.01660(8)	0.6233(3)	0.0459(6)
H26	0.838569	−0.002825	0.547035	0.055*
C27	0.3081(4)	0.20210(10)	0.5669(4)	0.0733(9)
H27A	0.300754	0.214393	0.652923	0.110*
H27B	0.215612	0.187914	0.509981	0.110*
H27C	0.327221	0.224422	0.509441	0.110*
C28	1.0875(3)	−0.02632(11)	0.7644(4)	0.0699(9)
H28A	1.186138	−0.015541	0.782063	0.105*
H28B	1.051706	−0.042747	0.675433	0.105*
H28C	1.091485	−0.044289	0.844712	0.105*

coordination complexes have been obtained by (polycarboxylic phenyl)hypophosphite ligand [11–13]. Herein, we reported a new binuclear complex $[Zn_2(H_3L)_2(bipy)_2(H_2O)_2]$ ($H_3L = 3-((3\text{-acetyl-5-carboxyphenyl})\text{oxidophosphoryl})\text{-5-carboxybenzoate}$, $bipy = 5,5'\text{-dimethyl-2,2'-bipyridine}$).

In the title complex, each Zn(II) is five-coordinated, with two N atoms from one bipy ligand, and three O atoms from carboxyl O2, phosphoryl O9 and aqua O11, respectively. The Zn1–N1 and Zn1–N2 bond lengths are 2.1230(18) Å and 2.0973(18) Å. The Zn–O bond lengths are in the range 1.9904(14) – 2.0698(16) Å, which are found within normal ranges bond distances in the reported Zn(II) complexes [11]. The O2–Zn1–N1ⁱ (symmetry code: $-x+1, -y, -z$) and O11–Zn1–N2 bond angles are 156.39(7)° and 153.16(8)° and other angles are in the range 77.70(8)–104.10(7)°, forming a distorted square-pyramidal coordination configuration. Each H_3L anion ligand functions as μ_2 -bridge to link two different Zn(II) ions, forming a binuclear structure.

It is worthy noting that the tri(3,5-dicarboxyphenyl) phosphine oxidem ligand changed into (3,5-dicarboxylic phenyl)(5-carboxylic-3-carboxyphenyl) hypophosphite ligand in the hydrothermal *in-situ* synthesis. Moreover, the stability of the solid state structure of the title complex is enhanced by O–H···O and C–H···O hydrogen bonding interactions. These hydrogen bonding interactions link neighbouring binuclear units into a three-dimensional architecture.

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