Crystal structure of aqua(2,2′-bipyridine-κ^2N,N')-(μ_2-4-hydroxy phthalato-κ^2O,O')zinc(II) hydrate, $Zn(C_{10}H_8N_2)(C_8H_4O_5)(H_2O) \cdot H_2O$

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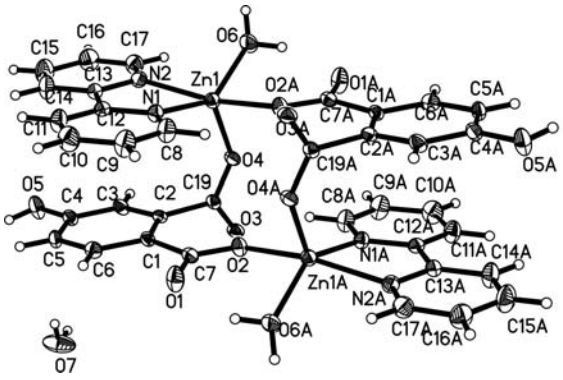

Abstract

$C_{18}H_{16}N_2O_7Zn$, orthorhombic, *Pbca* (no. 61), a = 17.665(1) Å, b = 10.5939(8) Å, c = 19.132(2) Å, V = 3580.3 Å^3, Z = 8, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.106, T = 296 K.

Source of material

A mixture of $Zn(CH_3COO)_2 \cdot 2H_2O$ (0.5 mmol), 4-hydroxyphthalic acid (0.5 mmol) and 2,2′-bipyridine (bpy, 0.5 mmol) in 12 mL distilled water was heated at 413 K in a Teflon-lined stainless steel autoclave for five days. The reaction system was then slowly cooled to room temperature. Colorless block-shaped crystals of the title compound suitable for single crystal X-ray diffraction analysis were collected by filtration, washed several times with distilled water and dried in air at ambient temperature (yield 43 % based on Zn). Elemental analyses were performed with a Perkin-Elmer 240C analyzer. Elemental analysis — found: H, 3.67 %; C, 49.38 %; N, 6.42 %; O, 25.58 %; calculated for $C_{18}H_{16}N_2O_7Zn$: H, 3.68 %; C, 49.39 %; N, 6.40 %; O, 25.59 %.

Experimental details

All H atoms were positioned geometrically and refined as riding, with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The hydrogen atoms of the water molecules were not located.

Discussion

Recently, much attention has been paid to coordination complexes due to their fascinating structural diversities and potential applications as functional materials [1,2]. In a spontaneous assembly process, the structural information stored in the organic ligands is read by the metal ion through its coordination [3]. The choice of appropriate organic ligands and metal is important to construct distinct architectures, from which corresponding properties are derived [4]. The carboxylate groups have a strong ability to bond various metal ions and afford abundant coordination modes, thus rigid carboxylate ligands have been widely used for the design and synthesis of a variety of structures [5].

In the title crystal structure, the asymmetric unit contains one Zn(II) atom, one bpy ligand, one 4-hydroxyphthalate anion, one coordinating water molecule and one lattice water molecule. The Zn(II) atom adopts a distorted tetragonal pyramid (ZnN_2O_3), with two N atoms from one bpy ligand and three O atoms from one 4-hydroxyphthalate anion as well as coordinated water molecule. The bond lengths and angles around the Zn(II) are in the ranges of 1.960(2) - 2.152(3) Å and 76.81(1)° - 164.91(1)°, respectively. The bidentate bpy ligand terminally coordinates Zn(II), while the 4-hydroxyphthalate anion is in μ_2-bridging coordination mode to connect the adjacent Zn centers.

There are O–H···O hydrogen bonding interactions between lattice water molecules, coordinating water molecule and carboxyl oxygen atoms: d(O7–H4W···O5) = 2.771(4) Å, ∠O7–H4W···O5 = 160.6°; d(O7–H3W···O1) = 2.720(4) Å, ∠O7–H3W···O1 = 171.0°; d(O6–H2W···O7) = 2.660(4) Å, ∠O6–H2W···O7 = 177.1°; d(O6–H1W···O1) = 2.604(3) Å, ∠O6–H1W···O1 = 147.0°; d(O5–H5···O3) = 2.554(3) Å, ∠O5–H5···O3 = 165.8°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.21 × 0.25 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.17 cm^{-1}
Diffractometer, scan mode:	Bruker FRAMBO, φ/ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9335, 3650
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2390
$N(param)_{refined}$:	254
Programs:	SHELXS-97, SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(5)	8*c*	0.6925	0.6472	0.1756	0.071
H(1W)	8*c*	1.1524	0.8720	0.1315	0.074
H(2W)	8*c*	1.1582	0.7503	0.1470	0.074
H(3W)	8*c*	0.2616	0.5924	0.1432	0.111
H(4W)	8*c*	0.2392	0.6371	0.2114	0.111
H(3)	8*c*	0.8356	0.8397	0.2009	0.036
H(5A)	8*c*	0.6974	0.6912	0.0573	0.037
H(6A)	8*c*	0.7515	0.8172	–0.0260	0.035
H(8)	8*c*	1.0053	0.8178	–0.0864	0.051
H(9)	8*c*	0.9338	0.7153	–0.1670	0.060
H(10)	8*c*	0.8562	0.5508	–0.1327	0.061
H(11)	8*c*	0.8558	0.4915	–0.0156	0.052

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(14)	8*c*	0.8557	0.4548	0.0954	0.056
H(15)	8*c*	0.8654	0.4191	0.2145	0.067
H(16)	8*c*	0.9538	0.5340	0.2788	0.061
H(17)	8*c*	1.0239	0.6852	0.2224	0.050

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	8*c*	1.03325(2)	0.81632(4)	0.07314(2)	0.0242(2)	0.0269(3)	0.0297(2)	–0.0009(2)	–0.0007(2)	–0.0025(2)
O(1)	8*c*	0.8289(2)	0.9836(2)	–0.0800(1)	0.059(2)	0.050(2)	0.029(2)	–0.024(2)	–0.013(2)	0.005(1)
O(2)	8*c*	0.9079(1)	1.0707(2)	–0.0038(1)	0.030(1)	0.035(1)	0.029(1)	–0.008(1)	–0.002(1)	–0.001(1)
O(3)	8*c*	0.9000(1)	1.0926(2)	0.1626(1)	0.025(1)	0.031(1)	0.038(2)	0.006(1)	–0.002(1)	–0.009(1)
O(4)	8*c*	0.9837(1)	0.9567(2)	0.1217(1)	0.020(1)	0.031(1)	0.034(1)	0.002(1)	–0.002(1)	–0.007(1)
O(5)	8*c*	0.7310(2)	0.6821(3)	0.1898(1)	0.041(2)	0.062(2)	0.039(2)	–0.024(2)	–0.008(2)	0.013(1)
O(6)	8*c*	1.1255(1)	0.8061(2)	0.1365(1)	0.033(2)	0.050(2)	0.066(2)	–0.006(1)	–0.020(2)	0.023(2)
O(7)	8*c*	0.2314(2)	0.6367(3)	0.1675(2)	0.070(2)	0.109(3)	0.043(2)	0.050(2)	–0.005(2)	–0.012(2)
N(1)	8*c*	0.9758(2)	0.7188(3)	–0.0049(2)	0.030(2)	0.027(2)	0.030(2)	–0.004(1)	–0.001(2)	–0.001(1)
N(2)	8*c*	0.9838(2)	0.6597(3)	0.1284(2)	0.031(2)	0.029(2)	0.030(2)	–0.001(2)	–0.003(2)	0.001(1)
C(1)	8*c*	0.8273(2)	0.9067(3)	0.0358(2)	0.018(2)	0.026(2)	0.029(2)	–0.001(2)	0.003(2)	–0.003(2)
C(2)	8*c*	0.8526(2)	0.9105(3)	0.1049(2)	0.018(2)	0.026(2)	0.030(2)	0.003(2)	–0.003(2)	–0.001(2)
C(3)	8*c*	0.8189(2)	0.8351(3)	0.1549(2)	0.023(2)	0.039(2)	0.028(2)	–0.002(2)	–0.005(2)	0.003(2)
C(4)	8*c*	0.7609(2)	0.7531(3)	0.1382(2)	0.022(2)	0.034(2)	0.032(2)	–0.003(2)	0.000(2)	0.004(2)
C(5)	8*c*	0.7360(2)	0.7466(3)	0.0696(2)	0.025(2)	0.028(2)	0.041(2)	–0.008(2)	0.000(2)	–0.003(2)
C(6)	8*c*	0.7688(2)	0.8222(3)	0.0199(2)	0.030(2)	0.033(2)	0.025(2)	0.001(2)	–0.005(2)	–0.005(2)
C(7)	8*c*	0.8572(2)	0.9916(3)	–0.0208(2)	0.022(2)	0.030(2)	0.029(2)	0.001(2)	–0.002(2)	–0.004(2)
C(8)	8*c*	0.9751(2)	0.7509(4)	–0.0719(2)	0.049(3)	0.043(2)	0.036(2)	–0.013(2)	–0.003(2)	0.003(2)
C(9)	8*c*	0.9321(3)	0.6904(4)	–0.1204(2)	0.067(3)	0.050(3)	0.033(2)	–0.016(3)	–0.012(2)	0.003(2)
C(10)	8*c*	0.8864(3)	0.5928(4)	–0.1003(2)	0.058(3)	0.053(3)	0.041(2)	–0.010(3)	–0.015(3)	–0.007(2)
C(11)	8*c*	0.8860(2)	0.5581(4)	–0.0307(2)	0.048(3)	0.036(2)	0.045(2)	–0.013(2)	–0.005(2)	–0.004(2)
C(12)	8*c*	0.9306(2)	0.6225(3)	0.0162(2)	0.026(2)	0.024(2)	0.037(2)	0.003(2)	–0.002(2)	–0.002(2)
C(13)	8*c*	0.9337(2)	0.5928(3)	0.0912(2)	0.026(2)	0.026(2)	0.036(2)	–0.002(2)	–0.001(2)	–0.000(2)
C(14)	8*c*	0.8896(2)	0.5015(4)	0.1221(2)	0.052(3)	0.043(3)	0.045(2)	–0.021(2)	–0.007(2)	0.006(2)
C(15)	8*c*	0.8957(3)	0.4794(4)	0.1929(2)	0.064(3)	0.056(3)	0.046(3)	–0.021(3)	–0.002(3)	0.020(2)
C(16)	8*c*	0.9475(2)	0.5479(4)	0.2311(2)	0.056(3)	0.056(3)	0.039(2)	–0.008(3)	–0.006(2)	0.017(2)
C(17)	8*c*	0.9895(2)	0.6375(4)	0.1966(2)	0.043(2)	0.042(2)	0.040(2)	–0.004(2)	–0.009(2)	–0.002(2)
C(19)	8*c*	0.9162(2)	0.9934(3)	0.1301(2)	0.023(2)	0.030(2)	0.021(2)	–0.003(2)	–0.006(2)	0.004(2)

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