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Crystal structure of aqua-bis(1,5-dimethyl-2-phenyl-4-(((*E*)-4-pyridylmethylene)amino)pyrazolidin-3-one- κN)-(nitrato- κO)-(nitrato- $\kappa^2 O, O'$)zinc(II), $C_{34}H_{34}N_{10}O_9Zn$

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Abstract

$C_{34}H_{34}N_{10}O_9Zn$, monoclinic, $P\bar{1}$ (no. 2), $a = 10.1188(13)$ Å, $b = 12.9297(16)$ Å, $c = 15.0668(19)$ Å, $\alpha = 88.032(2)^\circ$, $\beta = 75.277(2)^\circ$, $\gamma = 72.888(2)^\circ$, $V = 1820.4(4)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0668$, $wR_{ref}(F^2) = 0.1406$, $T = 298(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was synthesized by mixing 0.077 g (0.3 mmol) L4 ((*E*)-1,5-dimethyl-2-phenyl-4-((pyridin-4-ylmethylene)amino)-1*H*-pyrazol-3(2*H*)-one, which was prepared according to the literature [4]), 0.075 g (0.25 mmol) $Zn(NO_3)_2 \cdot 6H_2O$, 5 mL absolute ethyl alcohol and 1 mL dichloromethane in a reaction kettle flask. Then the reaction

Table 1: Crystal collection and handling.

Crystal:	Red block
Size:	0.20 × 0.10 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.74 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	12229, 6347, 0.030
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5411
$N(param)_{refined}$:	497
Programs:	Bruker programs [1], SHELX [2, 3]

kettle flask was placed in an oven at 90 °C. After 7 days, red granular crystals were separated with 20.25% yield. Elemental Analysis Data (%), measured/theoretical): C, 51.55/51.56; H, 4.30/4.28; N, 17.70/17.76. IR spectrum (cm^{−1}, KBr pellet): 3059(w), 1617(s), 1589(s), 1546(s), 1491(s), 1427(vs), 1384(w), 1303(w), 1203(w), 1139(w), 1059(w), 1021(m), 949(m), 916(w), 826(s), 768(vs), 751(w), 734(w), 658(vs), 625(s), 599(w), 569(s), 533(m), 506(m), 472(w), 434(m).

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

At present, the water (solvent) thermal synthesis method has been successfully applied to the synthesis of some metal organic frameworks with unique structures and special properties. The reaction mechanism follows the liquid nucleation mode [5]. Water (solvent) thermal synthesis reactions make it

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Zn1	−0.10264(5)	0.71736(4)	0.73691(3)	0.03891(16)
C1	−0.0259(5)	0.5006(4)	0.6367(3)	0.0519(12)
H1	−0.1192	0.5276	0.6310	0.062*
C2	0.0605(5)	0.4063(4)	0.5879(3)	0.0537(12)
H2	0.0257	0.3717	0.5499	0.064*
C3	0.1984(4)	0.3637(3)	0.5957(3)	0.0426(10)
C4	0.2410(5)	0.4167(4)	0.6565(3)	0.0542(12)
H4	0.3316	0.3890	0.6664	0.065*
C5	0.1487(5)	0.5101(4)	0.7017(3)	0.0530(12)
H5	0.1795	0.5445	0.7422	0.064*
C6	0.2986(5)	0.2711(3)	0.5369(3)	0.0466(11)
H6	0.2753	0.2494	0.4862	0.056*
C7	0.5181(4)	0.1410(3)	0.4933(3)	0.0417(10)
C8	0.6419(5)	0.0753(4)	0.5117(3)	0.0456(10)
C9	0.5237(5)	0.1171(3)	0.4004(3)	0.0471(11)
C10	0.6843(5)	0.0688(4)	0.5997(3)	0.0653(14)
H10A	0.6833	0.0001	0.6257	0.098*
H10B	0.6182	0.1260	0.6417	0.098*
H10C	0.7788	0.0760	0.5885	0.098*
C11	0.8674(5)	−0.0558(4)	0.4213(4)	0.0670(14)
H11A	0.9325	−0.0163	0.3922	0.101*
H11B	0.8800	−0.1169	0.3823	0.101*
H11C	0.8862	−0.0805	0.4789	0.101*
C12	0.7169(4)	0.0032(4)	0.2737(3)	0.0456(10)
C13	0.7538(5)	0.0762(4)	0.2115(4)	0.0687(15)
H13	0.7403	0.1470	0.2308	0.082*
C14	0.8111(7)	0.0440(6)	0.1197(4)	0.0887(19)
H14	0.8358	0.0933	0.0772	0.106*
C15	0.8313(7)	−0.0593(6)	0.0916(4)	0.090(2)
H15	0.8703	−0.0807	0.0298	0.108*
C16	0.7948(7)	−0.1315(6)	0.1533(4)	0.094(2)
H16	0.8103	−0.2024	0.1337	0.112*
C17	0.7349(6)	−0.1007(4)	0.2451(4)	0.0722(15)
H17	0.7072	−0.1497	0.2869	0.087*
C18	−0.3378(5)	0.9169(4)	0.8314(3)	0.0513(11)
H18	−0.3959	0.8730	0.8312	0.062*
C19	−0.3973(5)	1.0150(4)	0.8791(3)	0.0510(11)
H19	−0.4934	1.0359	0.9107	0.061*
C20	−0.3142(5)	1.0828(3)	0.8803(3)	0.0416(10)
C21	−0.1733(5)	1.0483(4)	0.8285(3)	0.0522(12)
H21	−0.1140	1.0922	0.8243	0.063*
C22	−0.1226(5)	0.9486(4)	0.7836(3)	0.0520(12)
H22	−0.0275	0.9265	0.7498	0.062*
C23	−0.3700(5)	1.1824(3)	0.9388(3)	0.0461(10)
H23	−0.4572	1.1957	0.9826	0.055*
C24	−0.3421(4)	1.3381(3)	0.9924(3)	0.0410(10)
C25	−0.2746(5)	1.4174(3)	0.9835(3)	0.0438(10)
C26	−0.4447(5)	1.3562(3)	1.0789(3)	0.0458(10)
C27	−0.1649(5)	1.4363(4)	0.9036(3)	0.0607(13)
H27A	−0.2056	1.5013	0.8752	0.091*
H27B	−0.1327	1.3761	0.8599	0.091*
H27C	−0.0854	1.4436	0.9239	0.091*
C28	−0.2987(6)	1.5810(4)	1.0786(3)	0.0656(14)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H28A	−0.2140	1.5863	1.0346	0.098*
H28B	−0.2848	1.5804	1.1393	0.098*
H28C	−0.3782	1.6419	1.0743	0.098*
C29	−0.4877(4)	1.4777(3)	1.2144(3)	0.0423(10)
C30	−0.4400(5)	1.4058(4)	1.2769(3)	0.0575(12)
H30	−0.3695	1.3406	1.2569	0.069*
C31	−0.4980(7)	1.4314(6)	1.3691(4)	0.0783(18)
H31	−0.4673	1.3829	1.4119	0.094*
C32	−0.5998(7)	1.5272(7)	1.3982(4)	0.083(2)
H32	−0.6390	1.5434	1.4608	0.099*
C33	−0.6453(6)	1.5997(5)	1.3364(4)	0.0784(17)
H33	−0.7136	1.6658	1.3569	0.094*
C34	−0.5897(5)	1.5751(4)	1.2433(3)	0.0577(12)
H34	−0.6209	1.6239	1.2008	0.069*
N1	0.0172(4)	0.5550(3)	0.6914(2)	0.0439(8)
N2	0.4173(4)	0.2199(3)	0.5541(2)	0.0442(8)
N3	0.7226(4)	0.0139(3)	0.4369(2)	0.0494(9)
N4	0.6526(4)	0.0389(3)	0.3676(2)	0.0497(9)
N5	−0.2003(4)	0.8816(3)	0.7852(2)	0.0435(8)
N6	−0.2993(4)	1.2507(3)	0.9299(2)	0.0433(8)
N7	−0.4352(4)	1.4485(3)	1.1182(2)	0.0475(9)
N8	−0.3274(4)	1.4822(3)	1.0597(2)	0.0460(9)
N9	−0.0309(5)	0.7003(4)	0.9001(3)	0.0662(12)
N10	0.0676(5)	0.7674(3)	0.5754(3)	0.0574(10)
O1	−0.0587(4)	0.7617(3)	0.5944(2)	0.0649(9)
O2	0.1222(5)	0.7605(4)	0.6405(3)	0.1038(15)
O3	0.1247(4)	0.7801(4)	0.4973(3)	0.1032(15)
O4	−0.1360(5)	0.6819(3)	0.8826(3)	0.0812(12)
O5	0.0556(5)	0.7202(3)	0.8333(3)	0.0988(14)
O6	−0.0181(5)	0.6947(5)	0.9769(3)	0.1246(19)
O7	−0.2843(3)	0.6894(2)	0.7255(2)	0.0439(7)
H7A	−0.328(4)	0.736(3)	0.697(2)	0.066*
H7B	−0.339(3)	0.680(4)	0.7735(17)	0.066*
O8	0.4392(4)	0.1528(3)	0.3515(2)	0.0665(10)
O9	−0.5286(3)	1.3054(3)	1.1169(2)	0.0615(9)

possible to synthesize some novel materials with special functions or unique structures by using high-temperature aqueous solutions or solvents as the reaction medium for some reactions which will not proceed at room temperature [6].

Some metal schiff base complexes have special properties and applications [7, 8]. Herein, the complexes of Zn(II) with pyridine-4-carboxaldehyde 4-aminoantipyrine (L4) was synthesized by water (solvent) thermal synthesis. The asymmetric unit of the title structure consists of one Zn(II) atom, two pyridine nitrogen atoms of two L4 ligands, two nitrate ions and one water molecule. In the complex, the center Zn atom forms a distorted square-pyramidal coordination sphere. The coordination donor atoms come from two oxygen atoms of two separated nitrate anions and two pyridine nitrogen atoms of two L4 ligands, and the water O7 atom resides on the apical position. (*cf.* the figure). The angle N(5)—Zn(1)—N(1) is 173.32(13)°. The bond lengths of

Zn(1)—N(1) and Zn(1)—N(5) are by chance both 2.127(3) Å, which are different from that referred in other reports [9]. The bond length of Zn(1)—O(1) is 2.172(3) Å, the bond length of Zn(1)—O(4) is 2.188(4) Å, the length of Zn(1)—O(7) is 2.023(3) Å. The bond lengths of Zn(1)—O(1) and Zn(1)—O(4) are almost the same. Bond lengths and bond angles of ligand L4 are within the normal range [10]. A different coordination for two nitrate ligands is not unusual [11]. A structurally directly related Ni(II) complex is known for more than one decade [12].

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