Haixia Pang*

Crystal structure of *catena*-poly[dibromido- $\{\mu_2$ -1,5-dimethyl-2-phenyl-4-((pyridin-4-ylmethylene)amino)-1,2-dihydro-3*H*-pyrazol-3-one- κ^2 N:O} zinc(II)], $C_{34}H_{32}Br_4N_8O_2Zn_2$

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Abstract

$C_{34}H_{32}Br_4N_8O_2Zn_2$, triclinic, $P\bar{1}$ (no. 2), $a = 11.441(2)$ Å, $b = 13.422(2)$ Å, $c = 14.370(3)$ Å, $\alpha = 101.129(3)^\circ$, $\beta = 99.587(2)^\circ$, $\gamma = 114.972(3)^\circ$, $V = 1885.0(6)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0510$, $wR_{ref}(F^2) = 0.1775$, $T = 298(2)$ K.

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The title 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 material

The title compound was synthesized by mixing of 0.0928 g (0.3 mmol) L4 (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]), 0.115 g (0.5 mmol) $ZnBr_2$, 5 mL absolute ethanol and 1 mL dichloromethane in a 25 mL polytetrafluoroethylene reactor. Then the reactor was

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.15 \times 0.12 \times 0.12$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	5.56 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II,
θ_{max} , completeness:	30.4° , 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	21243, 11170, 0.049
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5513
$N(param)_{refined}$:	455
Programs:	Bruker [1], SHELX [2, 3]

placed in an oven at 85 °C. After 7 days, small yellow granular crystals separated out with a 83% yield. Elemental analysis calculated for $C_{34}H_{32}Br_4N_8O_2Zn_2$: C 39.45, H 3.12, O 3.10, N 10.82%; found C 39.49, H 3.17, O 3.15, N 10.88%. IR spectrum (cm^{-1} , KBr pellet): 3059(w), 1599(m), 1574(m), 1550(s), 1493(m), 1423(m), 1023(m), 831(w).

Comment

Schiff base refers to an organic compound containing an imino group or a methylimine characteristic group ($-N=CR-$), which is widely used in medicine and catalysis due to its wide biological activity and special chemical and physical properties, surface modification and other fields [5]. However, only a small number of structural characterization data of 4-aminoantipyrine-derived ligands and metal complexes have been reported, which makes the ligand

*Corresponding author: Haixia Pang, School of Materials and Chemical Engineering, Hubei University of Technology, Wuhan 430068, P.R. China; and State Key Laboratory of Biogeology and Environmental Geology, China University of Geosciences, Wuhan 430074, P.R. China, e-mail: m15623901779@163.com

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	0.24662(7)	0.80319(5)	1.55400(5)	0.03551(17)
Zn2	0.27432(6)	0.29887(5)	1.06519(5)	0.03443(17)
Br1	0.44189(7)	0.97422(6)	1.64234(6)	0.0688(2)
Br2	0.08477(7)	0.70204(6)	1.62802(5)	0.0550(2)
Br3	0.34975(8)	0.49227(6)	1.06948(6)	0.0573(2)
Br4	0.07811(6)	0.20852(7)	1.11444(5)	0.0561(2)
C1	0.4311(6)	0.7385(5)	1.4676(4)	0.0419(14)
H1	0.4888	0.8168	1.4959	0.050*
C2	0.4752(6)	0.6723(5)	1.4145(4)	0.0454(15)
H2	0.5596	0.7063	1.4041	0.054*
C3	0.3945(6)	0.5550(5)	1.3763(4)	0.0362(13)
C4	0.2667(6)	0.5094(5)	1.3862(5)	0.0493(16)
H4	0.2066	0.4319	1.3564	0.059*
C5	0.2278(6)	0.5827(5)	1.4426(5)	0.0457(15)
H5	0.1435	0.5511	1.4535	0.055*
C6	0.4381(6)	0.4801(5)	1.3189(4)	0.0410(14)
H6	0.3766	0.4052	1.2812	0.049*
C7	0.6110(6)	0.4594(5)	1.2675(4)	0.0392(13)
C8	0.5426(5)	0.3554(5)	1.1867(4)	0.0316(12)
C9	0.7476(6)	0.4997(5)	1.2782(4)	0.0444(15)
C10	0.8590(7)	0.6046(6)	1.3506(5)	0.0595(19)
H10A	0.9417	0.6016	1.3529	0.089*
H10B	0.8437	0.6100	1.4147	0.089*
H10C	0.8642	0.6708	1.3315	0.089*
C11	0.8880(6)	0.4443(7)	1.1849(5)	0.0585(19)
H11A	0.9571	0.5216	1.2182	0.088*
H11B	0.8722	0.4309	1.1148	0.088*
H11C	0.9158	0.3913	1.2042	0.088*
C12	0.6263(6)	0.2467(5)	1.0768(4)	0.0377(13)
C13	0.6811(7)	0.1787(6)	1.0920(5)	0.0540(17)
H13	0.7284	0.1886	1.1553	0.065*
C14	0.6665(9)	0.0937(7)	1.0130(8)	0.082(3)
H14	0.7066	0.0474	1.0221	0.099*
C15	0.5942(9)	0.0778(8)	0.9224(7)	0.080(3)
H15	0.5815	0.0180	0.8699	0.096*
C16	0.5406(7)	0.1457(6)	0.9062(5)	0.0591(19)
H16	0.4916	0.1331	0.8427	0.071*
C17	0.5567(6)	0.2347(5)	0.9825(4)	0.0445(15)
H17	0.5227	0.2846	0.9716	0.053*
C18	0.2824(6)	0.2671(5)	0.8552(5)	0.0437(14)
H18	0.3305	0.3470	0.8779	0.052*
C19	0.2582(6)	0.2146(5)	0.7588(4)	0.0394(13)
H19	0.2869	0.2580	0.7162	0.047*
C20	0.1891(5)	0.0934(5)	0.7230(4)	0.0344(12)
C21	0.1473(6)	0.0354(5)	0.7908(4)	0.0381(13)
H21	0.1018	−0.0446	0.7711	0.046*
C22	0.1734(6)	0.0965(5)	0.8886(4)	0.0358(12)
H22	0.1425	0.0562	0.9327	0.043*
C23	0.1627(6)	0.0337(5)	0.6192(5)	0.0435(14)
H23	0.1238	−0.0461	0.5985	0.052*
C24	0.1831(5)	0.0354(5)	0.4613(4)	0.0312(11)
C25	0.1666(5)	−0.0773(5)	0.4190(4)	0.0344(12)
C26	0.1967(5)	0.0913(5)	0.3885(4)	0.0360(12)
C27	0.2180(7)	0.2083(5)	0.3944(5)	0.0548(17)
H27A	0.3034	0.2521	0.3839	0.082*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H27B	0.2165	0.2442	0.4585	0.082*
H27C	0.1480	0.2051	0.3448	0.082*
C28	0.2412(8)	0.0548(6)	0.2262(5)	0.063(2)
H28A	0.1968	−0.0090	0.1671	0.094*
H28B	0.3358	0.0791	0.2431	0.094*
H28C	0.2259	0.1173	0.2156	0.094*
C29	0.1597(6)	−0.1775(5)	0.2483(4)	0.0337(12)
C30	0.0645(6)	−0.2202(6)	0.1585(5)	0.0482(15)
H30	0.0080	−0.1882	0.1452	0.058*
C31	0.0553(7)	−0.3110(6)	0.0894(5)	0.0554(18)
H31	−0.0068	−0.3400	0.0277	0.067*
C32	0.1373(6)	−0.3597(5)	0.1105(4)	0.0448(15)
H32	0.1285	−0.4225	0.0633	0.054*
C33	0.2308(6)	−0.3174(5)	0.1991(5)	0.0430(14)
H33	0.2862	−0.3504	0.2126	0.052*
C34	0.2427(5)	−0.2247(5)	0.2692(4)	0.0342(12)
H34	0.3066	−0.1945	0.3301	0.041*
N1	0.3078(4)	0.6949(4)	1.4804(3)	0.0348(10)
N2	0.5617(5)	0.5202(4)	1.3222(4)	0.0432(12)
N3	0.7649(5)	0.4275(4)	1.2112(4)	0.0414(12)
N4	0.6419(4)	0.3394(4)	1.1564(3)	0.0372(11)
N5	0.2403(4)	0.2096(4)	0.9204(3)	0.0321(10)
N6	0.1920(5)	0.0893(4)	0.5574(3)	0.0408(12)
N7	0.1889(5)	0.0203(4)	0.3055(4)	0.0372(11)
N8	0.1746(5)	−0.0809(4)	0.3246(3)	0.0357(11)
O8	0.4182(3)	0.2838(3)	1.1497(3)	0.0359(9)
O9	0.1437(4)	−0.1625(3)	0.4502(3)	0.0377(9)

coordination chemistry still have a lot of space to explore [6–9]. Zinc(II) is an essential component of various enzymes required for metabolism and plays a very important role in catalysis and gene expression. Zinc (II) can stabilize the molecular structure of proteins and nucleic acids, maintain the integrity of subcellular organelles, participate in the transportation process, and play an important role in the process of viral immune phenomena. It is well known that zinc (II) has a strong affinity for nitrogen and oxygen in biological systems, which further stimulates researchers' interest in zinc coordination chemistry [10–12].

Herein, one complex of Zn(II) and ligand L4 and two bromido ligands are described. The asymmetric unit of the complex title structure consists of two Zn(II) atoms, two L4 liands and four bromine atoms (*cf.* the figure). As for the two central Zn(II) coordination spheres, they are similarly coordinated by two bromine atoms and each one pyridine nitrogen and carboxyl oxygen atoms from two crystallographically independent L4 ligands, forming a distorted tetragonal coordination environment. The Zn–O and Zn–N bond distance around the two zinc atoms lie in a range of 1.985(4)~1.990(4) Å and similar to that reported in literature [13]; the bond length of Zn–N1 is 2.057(5)~2.072(4) Å, which

is similar to that reported in literature [14]; the bond length of Zn1—Br is 2.3394(10)~2.3441(9) Å, the bond length of Zn2—Br is 2.3486(10)~2.3607(9) Å. The bond angle of Br1—Zn1—Br2 is 122.81(4)°. A one-dimensional coordination polymer along the (011) direction is formed. These adjacent one-dimensional chains are joined together by some weak π – π interactions and C—H– π interactions.

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