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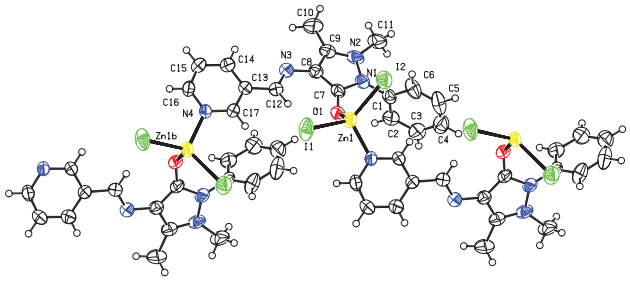
Crystal structure of *catena*-poly[diiodido-(μ_2-1,5-dimethyl-2-phenyl-4-((pyridin-3-ylmethylene)amino)-1,2-dihydro-3*H*-pyrazol-3-one-κ^2*N*:*O*) zinc(II)], $C_{17}H_{16}I_2N_4OZn$



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Abstract

$C_{17}H_{16}I_2N_4OZn$, monoclinic, $P2_1/c$ (no. 14), $a = 8.8359(15)$ Å, $b = 15.585(3)$ Å, $c = 15.163(3)$ Å, $\beta = 95.534(3)°$, $V = 2078.4(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0364$, $wR_{ref}(F^2) = 0.1265$, $T = 298(2)$ K.

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A part of the polymeric 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 materials

The title compound was synthesized by mixing 0.0876 g (0.3 mmol) 1,5-dimethyl-2-phenyl-4-((pyridin-3-ylmethylene)amino)-1,2-dihydro-3*H*-pyrazol-3-one (L), which was prepared according to the literature [4], 0.1612 g (0.5 mmol) ZnI_2, 5 mL absolute ethyl alcohol and 1 mL dichloromethane in a Teflon lined reactor. Then the reactor was placed in an oven at 90 °C. After 7 days, light yellow crystals were obtained with 80.16% yield. Elemental Analysis Data (%, measured theoretical): C, 33.56/33.38; H, 2.76/2.62; N, 9.10/9.16. **IR** spectrum (cm^{-1}, KBr pellet): 2923(w), 1615(vs),

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	4.16 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	28.4°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16568, 5168, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3478
$N(param)_{refined}$:	228
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.45035(7)	0.21384(4)	0.71074(3)	0.04805(16)
I1	0.29088(5)	0.24231(3)	0.83998(3)	0.07999(17)
I2	0.34733(5)	0.10093(3)	0.59944(2)	0.06706(15)
N1	0.8071(5)	0.0883(2)	0.6832(3)	0.0485(9)
N2	0.8879(5)	0.0134(2)	0.7053(3)	0.0544(10)
N3	0.7381(5)	0.0500(2)	0.9110(3)	0.0482(9)
N4	0.5096(5)	0.1723(2)	1.1536(2)	0.0479(9)
O1	0.6643(4)	0.1854(2)	0.7556(2)	0.0490(7)
C1	0.8289(5)	0.1340(3)	0.6037(3)	0.0466(10)
C2	0.9433(7)	0.1940(3)	0.6046(4)	0.0608(13)
H2	1.0031	0.2060	0.6570	0.073*
C3	0.9688(8)	0.2364(4)	0.5271(4)	0.0738(17)
H3	1.0476	0.2760	0.5270	0.089*
C4	0.8781(9)	0.2199(4)	0.4506(4)	0.0754(18)
H4	0.8940	0.2490	0.3988	0.090*
C5	0.7637(8)	0.1605(5)	0.4505(4)	0.0784(19)
H5	0.7025	0.1497	0.3983	0.094*
C6	0.7379(6)	0.1165(4)	0.5264(4)	0.0672(15)
H6	0.6608	0.0758	0.5257	0.081*
C7	0.7394(5)	0.1159(3)	0.7559(3)	0.0429(10)
C8	0.7760(5)	0.0551(3)	0.8231(3)	0.0448(10)
C9	0.8672(5)	−0.0068(3)	0.7889(3)	0.0503(11)
C10	0.9322(8)	−0.0853(4)	0.8327(5)	0.0752(17)
H10A	0.8791	−0.1347	0.8079	0.113*
H10B	0.9217	−0.0824	0.8951	0.113*
H10C	1.0379	−0.0896	0.8236	0.113*
C11	0.9591(7)	−0.0350(4)	0.6392(4)	0.0700(16)
H11A	1.0092	−0.0843	0.6664	0.105*
H11B	1.0324	0.0005	0.6138	0.105*
H11C	0.8831	−0.0533	0.5936	0.105*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C12	0.6561(6)	0.1079(3)	0.9418(3)	0.0461(10)
H12	0.6200	0.1534	0.9060	0.055*
C13	0.6187(5)	0.1027(3)	1.0340(3)	0.0438(10)
C14	0.6518(6)	0.0318(3)	1.0869(3)	0.0541(12)
H14	0.7006	−0.0155	1.0651	0.065*
C15	0.6110(7)	0.0319(3)	1.1734(3)	0.0635(14)
H15	0.6313	−0.0154	1.2100	0.076*
C16	0.5413(7)	0.1023(3)	1.2036(3)	0.0571(13)
H16	0.5144	0.1019	1.2613	0.069*
C17	0.5475(5)	0.1711(3)	1.0703(3)	0.0467(10)
H17	0.5246	0.2190	1.0350	0.056*

1597(m), 1577(s), 1560(vs), 1483(m), 1433(m), 1421(m), 1361(m), 1274(m), 1203(w), 1184(w), 1130(w), 1025(w), 962(w), 895(w), 818(w), 767(s), 746(w), 698(s), 642(s).

Comment

Since the late 19th century, schiff bases have played an increasingly important role in the development of coordination chemistry. Schiff base 4-aminoantipyrine and its complexes always have high biological activity and special morphology [5]. In particular, some metal Schiff base complexes with special properties and applications, have already been widely used in medicine, catalysis, analysis, materials and other fields [6–8]. However, there are just few reports about the structural characterization data of metal complexes and 4-aminoantipyrine-derived ligands.

The asymmetric unit of the complex is composed of one Zn (II) atom, one pyridine bases ligand L and two iodine atoms. In the complex, Zn(II) takes the mode of a tetrahedral coordination to form a slightly deformed tetrahedral spatial configuration. The coordination atoms are: the carbonyl oxygen atom in one molecule of the ligand L, the nitrogen atom in the pyridine ring of the adjacent ligand L, and two iodine atoms (*cf*. the figure). The bond length of Zn(1)—O(1) is 1.997(3) Å and the bond length of Zn(1)—$N(4)^{\#1}$ is 2.064(4) Å, which is slightly longer than those reported in literature [9, 10]. The bond length of Zn(1)—I(1) is 2.5604(7) Å and the length of Zn(1)—I(2) is 2.5451(7) Å. The angle of I(1)—Zn(1)—I(2) is 116.06(3)°.

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References

1. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, WI, USA (2007).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. Acta Crystallogr. **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal refinement with SHELX. Acta Crystallogr. **C71** (2015) 3–8.
4. Montalvo-Gonzalez, R.; Ariza-Castolo, A.: Molecular structure of diaryl-aldimines by multinuclear magnetic resonance and X-ray diffraction. J. Mol. Struct. **655** (2003) 375–389.
5. Bi, C. F.; Fan, Y. H.; Shi, G. X.: Synthesis and reactivity in inorganic and metal-organic chemistry. Synth. React. Inorg. Met-Org. Chem. **31** (2001) 219–225.
6. Sun, G.; Sun, Y. X.; Yu, C.: Synthesis, crystal structure, vibrational spectral and density functional studies of 4-(2,5-dioxo-2*H*-pyrrol-1(5*H*)-yl) antipyrine. J. Mol. Struct. **1006** (2001) 383–394.
7. Liu, B.; Fang, D. N.; Hwang, K.-C.: Electric-field-induced fatigue crack growth in fer-roelectric ceramics. Mater. Lett. **54** (2002) 442–446.
8. Rajendran, G.; Sreeletha, G. S.: Synthesis, characterization and antimicrobial studies of some mixed ligand complexes of lanthanide(III) ions. Asian. J. Chem. **14** (2002) 1639–1645.
9. Yang, X.; Fu, R. B.; Hu, S. M.: Three new Zn(II)/Cd(II) coordination polymers constructed with 2-mercaptonicotinic acid and 1,2-di(4-pyridyl)ethylene: syntheses and structure analysis. Chin. J. Struct. Chem. **32** (2013) 23–32.
10. Wang, X. W.; Fu, P.: Crystal structures and fluorescent properties of two linear trinuclear zinc(II) complexes. J. Mol. Struct. **842** (2007) 75–80.