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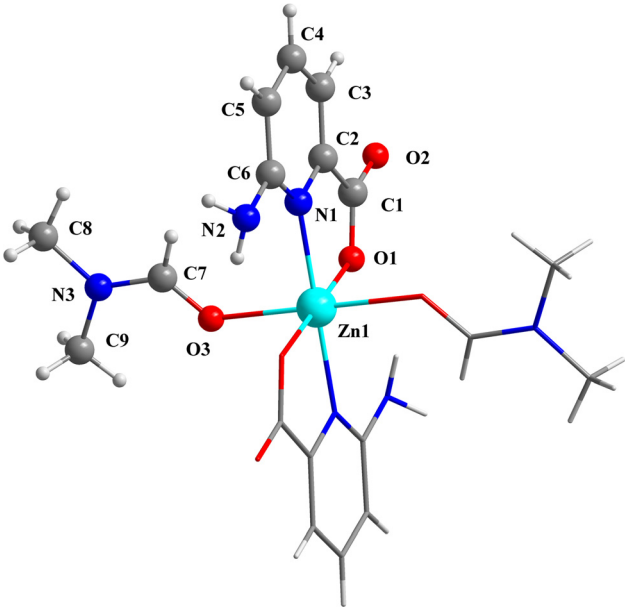
Crystal structure of bis(6-aminopyridine-2-carboxylato–k^{2}O,N)-bis(N,N-dimethylformamide-k^1*O*)zinc(II), $C_{18}H_{24}N_6O_6Zn$



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Abstract

$C_{18}H_{24}N_6O_6Zn$, monoclinic, $P2_1/n$ (no. 14), a = 10.1669(6) Å, b = 8.0831(4) Å, c = 13.1012(8) Å, β = 91.221(5)°, Z = 2, V = 1076.41(11) Å^3, R_{gt}(F) = 0.0396, wR_{ref}(F^2) = 0.0987, T = 273 K.

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The molecular 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.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.35 × 0.30 × 0.28 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.19 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,917, 2615, 0.057
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2018
$N(param)_{refined}$:	144
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Olex2 [4]

1 Source of material

All reagents were purchased and used without further purification. The title complex was synthesized solvothermally in a 25 mL Teflon-lined autoclave by heating a mixture of equal mole of 6-aminopyridine-2-carboxylic acid (13.8 mg, 0.1 mmol), $Zn(NO_3)_2{\cdot}6H_2O$ (29.8 mg, 0.1 mmol) and 6 mL N,N-dimethylformamide at 353 K for 72 h. After the mixture was slowly cooled to room temperature at a rate of 5 K h^{-1}, colorless crystals were collected by filtration and washed with distilled water in 39% yield.

2 Experimental details

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

3 Comment

The selection of suitable organic ligands and joining metal centers are very important to obtain the desired metal complexes. Multidentate organic ligands containing N- and O-donors, for example, pyridine carboxylate including pyridine-2-carboxylic acid, nicotinic acid, isonicotinic acid, 3-hydroxy-2-carboxylic acid and 6-hydroxypyridine-2-carboxylic acid have been used for the syntheses of metal complexes [5–9]. From the point of view of structural

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

Atom	x	y	z	U_{iso}*/U_{eq}
Zn1	0.5000	0.5000	0.5000	0.03119 (14)
O1	0.59731 (15)	0.71578 (18)	0.53341 (13)	0.0410 (4)
O2	0.78373 (16)	0.8174 (2)	0.59660 (15)	0.0544 (5)
O3	0.38601 (19)	0.5243 (2)	0.64224 (14)	0.0505 (5)
N1	0.66619 (16)	0.4047 (2)	0.57972 (13)	0.0269 (4)
N2	0.61150 (18)	0.1261 (2)	0.58067 (16)	0.0427 (5)
H2A	0.5373	0.1500	0.5515	0.051*
H2B	0.6302	0.0249	0.5952	0.051*
N3	0.3426 (2)	0.4669 (3)	0.80691 (15)	0.0398 (5)
C1	0.7099 (2)	0.7014 (3)	0.57610 (18)	0.0338 (5)
C10	0.7527 (2)	0.5265 (2)	0.60381 (18)	0.0316 (5)
C3	0.8707 (3)	0.4969 (3)	0.6517 (2)	0.0506 (7)
H3	0.9278	0.5834	0.6679	0.061*
C4	0.9040 (3)	0.3336 (3)	0.6761 (2)	0.0561 (7)
H4	0.9844	0.3100	0.7081	0.067*
C5	0.8185 (2)	0.2093 (3)	0.65260 (18)	0.0415 (6)
H5	0.8394	0.1004	0.6689	0.050*
C6	0.6986 (2)	0.2474 (2)	0.60356 (16)	0.0288 (5)
C7	0.4155 (2)	0.4611 (3)	0.72486 (18)	0.0359 (5)
H7	0.4952	0.4050	0.7300	0.043*
C8	0.3832 (3)	0.3865 (4)	0.90174 (19)	0.0594 (8)
H8A	0.3292	0.2911	0.9126	0.089*
H8B	0.3737	0.4624	0.9575	0.089*
H8C	0.4736	0.3531	0.8977	0.089*
C9	0.2155 (3)	0.5467 (5)	0.8054 (3)	0.0887 (12)
H9A	0.2157	0.6352	0.8543	0.133*
H9B	0.1490	0.4676	0.8224	0.133*
H9C	0.1971	0.5904	0.7384	0.133*

chemistry, the 6-aminopyridine-2-carboxylic acid (HL) is an efficient ligand, which contains N and O coordination sites and exhibits rich coordination modes. In previous work, the ligand has been used to synthesize corresponding metal complexes and the ligand shows excellent chelating coordination ability [10–14].

The asymmetric unit consists of half of a Zn(II) ion located on a crystallographic inversion center and chelation L^- anion plus DMF. The central Zn(II) ion is six-coordinated in a distorted octahedral coordination geometry finished by two carboxylate oxygen atoms, two pyridyl nitrogen atoms from two L^- anions and two coordinated N,N-dimethylformamide molecules. In the complex, the Zn–O bonds distances are 2.0481(17) and 2.2241(19) Å, and the Zn–N bond distance is 2.1127(19) Å. The O–Zn–O angles range from to 89.81(8) and 180.0°, the O–Zn–N angles range from 80.02(7) to 99.98 (7)°. These values are matched with the previously reported other Zn(II) complexes [15]. In the complex, central Zn(II) ion is connected by L^- anions with a *N*, *O* chelating mode to generate a discrete molecule structure. In the crystal, intermolecular N–H···O hydrogen interactions are observed involving the amino groups and carboxylate oxygen atoms.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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