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Crystal structure of *N,N'*-bis(pyridin-4-ylmethyl)pyrazine-2,3-dicarboxamide dihydrate, $C_{18}H_{20}N_6O_4$

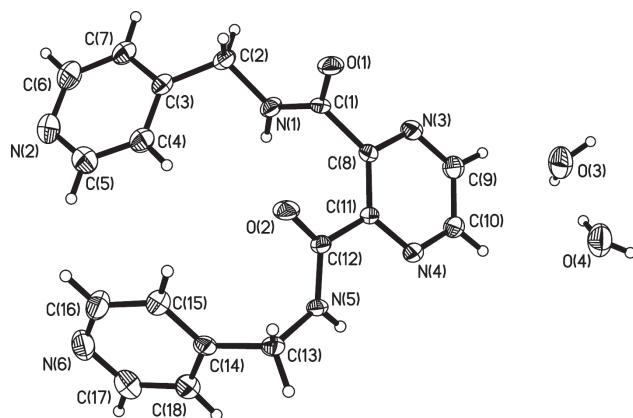


Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.28 × 0.22 × 0.16
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.0 cm ⁻¹
Diffractometer, scan mode:	Bruker, φ and ω
$2\theta_{\max}$, completeness:	55.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5693, 2793, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2326
$N(\text{param})_{\text{refined}}$:	253
Programs:	Bruker programs [8], SHELX [9, 10], OLEX2 [11]

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Abstract

$C_{18}H_{20}N_6O_4$, monoclinic, Pc , $a = 4.4412(8)$ Å, $b = 14.100(3)$ Å, $c = 15.206(3)$ Å, $\beta = 95.920(4)^\circ$, $V = 947.1(3)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0352$, $wR_{\text{ref}}(F^2) = 0.0770$, $T = 296.15$ 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 material

All reagents and solvents for syntheses were purchased from commercial sources and used as received without further

purification. The title compound was prepared by the condensation reaction of dimethyl pyrazine-2,3-dicarboxylate (3.92 g, 20 mmol) with 4-aminomethylpyridine (2.16 g, 20 mmol) under the reflux condition in 15 mL of methanol for 12 h. Followed by rotary evaporation to remove the solvent, a yellow solid was obtained. Recrystallization in CH₃OH gave light yellow crystals.

Experimental details

The U_{iso} values of hydrogen atoms of the water molecules were set to $1.5U_{\text{eq}}(\text{O})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(\text{C})$.

Discussion

Pyrazine-based amide ligands have played a very important role in coordination chemistry [1, 2]. Some transition metal complexes of pyrazine-2,3-bisamide ligands were explored to construct supramolecular architectures such as molecular grids. For example, a symmetrical diamide ligand containing a central bridging pyrazinyl unit connected to two tetradentate binding pockets that forms bi- and tetranuclear copper(II) complexes have been reported [3–7]. The two amide hydrogens of these compounds can be deprotonated, allowing the anionic amidate N donor atoms to coordinate to a metal center. The neutral molecule forms a centrosymmetric dimer. Whereas in the presence of triethylamine, a tetranuclear copper(II) complex is formed, where the pyrazine unit

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.0764(4)	0.50400(13)	0.69686(11)	0.0456(4)
O2	−0.1529(4)	0.58860(11)	0.50554(11)	0.0450(5)
N1	0.4514(5)	0.58862(14)	0.64393(13)	0.0382(5)
H1	0.5572	0.5968	0.6003	0.046*
N2	0.6937(7)	0.93853(17)	0.6340(2)	0.0700(8)
N3	0.4427(6)	0.36879(15)	0.59609(14)	0.0465(6)
N4	0.1944(5)	0.38785(14)	0.42142(14)	0.0430(5)
N5	−0.0888(5)	0.55002(13)	0.36457(12)	0.0337(5)
H5	0.0044	0.5142	0.3304	0.040*
N6	0.1070(7)	0.90226(17)	0.3298(2)	0.0646(8)
C1	0.2571(5)	0.51721(15)	0.64218(14)	0.0308(5)
C2	0.4845(6)	0.65575(17)	0.71719(17)	0.0432(6)
H2A	0.6452	0.6343	0.7607	0.052*
H2B	0.2983	0.6573	0.7453	0.052*
C3	0.5563(6)	0.75401(17)	0.68745(16)	0.0388(6)
C4	0.4248(7)	0.7919(2)	0.6094(2)	0.0530(7)
H4	0.2870	0.7565	0.5727	0.064*
C5	0.4986(9)	0.8829(2)	0.5858(2)	0.0677(9)
H5A	0.4059	0.9069	0.5328	0.081*
C6	0.8168(9)	0.9011(2)	0.7106(3)	0.0740(11)
H6	0.9500	0.9384	0.7470	0.089*
C7	0.7571(7)	0.8111(2)	0.7383(2)	0.0555(8)
H7	0.8523	0.7885	0.7916	0.067*
C8	0.2830(6)	0.44608(16)	0.56901(15)	0.0316(5)
C9	0.4760(8)	0.30252(18)	0.53578(19)	0.0535(8)
H9	0.5829	0.2476	0.5527	0.064*
C10	0.3573(7)	0.31265(18)	0.44917(18)	0.0531(8)
H10	0.3921	0.2654	0.4088	0.064*
C11	0.1544(5)	0.45461(15)	0.48162(14)	0.0305(5)
C12	−0.0427(5)	0.53776(16)	0.45160(14)	0.0310(5)
C13	−0.2905(6)	0.62335(16)	0.32515(17)	0.0390(6)
H13A	−0.4698	0.6257	0.3565	0.047*
H13B	−0.3542	0.6063	0.2643	0.047*
C14	−0.1485(5)	0.72047(17)	0.32697(16)	0.0375(6)
C15	−0.1882(7)	0.78357(19)	0.39381(19)	0.0520(7)
H15	−0.3005	0.7665	0.4397	0.062*
C16	−0.0596(9)	0.8724(2)	0.3919(2)	0.0642(9)
H16	−0.0918	0.9142	0.4372	0.077*
C17	0.1435(8)	0.8410(2)	0.2651(2)	0.0640(9)
H17	0.2573	0.8599	0.2202	0.077*
C18	0.0205(7)	0.7505(2)	0.2614(2)	0.0517(7)
H18	0.0523	0.7102	0.2148	0.062*
O4	0.2917(6)	0.08204(14)	0.41438(19)	0.0796(7)
H4A	0.3102	0.0307	0.3909	0.119*
H4B	0.4755	0.0921	0.4399	0.119*
O3	0.8244(6)	0.09840(15)	0.52465(18)	0.0794(7)
H3A	0.8494	0.0438	0.5539	0.119*
H3B	0.9698	0.0981	0.4933	0.119*

bridges between metal centers [3]. In order to expand the research scope of pyrazine-2,3-bisamide ligand system, we prepared the title compound to investigate its transition metal chemistry.

The title structure consists of one central pyrazine and two pyridin-4-ylmethyl group connected by two amide group and two water molecules. The coordination modes of H₂L

could be more flexible due to two outside N atom from the pyridine. So, it may be used as a bridging ligand to construct metal complexes with 3D networks or metal organic frameworks.

H₂L molecules are connected by hydrogen bonds. The water molecules are connected by hydrogen bonds to form a chain along [100].

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