Ying Xiong* and Ya An

Crystal structure of *N',N'''*-((propane-2,2-diylbis(1*H*-pyrrole-5,2-diyl))bis(methaneylylidene))-di(isonicotinohydrazide)–water – dimethylformamide (1/4/2), $C_{25}H_{24}N_8O_2 \cdot 4H_2O \cdot 2C_3H_7NO$

<https://doi.org/10.1515/ncrs-2021-0091>

Received March 14, 2021; accepted April 12, 2021;

published online April 22, 2021

Abstract

$C_{31}H_{46}N_{10}O_8$, monoclinic, $P2_1/n$ (no. 13), $a = 8.8207(6)$ Å, $b = 9.7818(8)$ Å, $c = 20.9498(17)$ Å, $\beta = 97.259(3)^\circ$, $V = 1793.1(2)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0426$, $wR_{ref}(F^2) = 0.1269$, $T = 273$ K.

CCDC no.: 2058458

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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.22 \times 0.20 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	D8, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	73802, 3179, 0.122
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2439
$N(param)_{refined}$:	231
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

Source of material

A solution of 5,5'-(propane-2,2-diyl) bis(1*H*-pyrrole-2-carbaldehyde) (0.46 g, 2.0 mmol) in ethanol (50 mL) was added to a stirred ethanol solution (50 mL) of isonicotinic acid hydrazide (0.55 g, 4.0 mmol). The reaction mixture was refluxed for 12 h and then cooled to room temperature.

*Corresponding author: Ying Xiong, School of Chemistry and Materials, Guizhou Normal University, Guiyang 550025, People's Republic of China, E-mail: 124251038@qq.com. <https://orcid.org/0000-0002-5790-3396>

Ya An, School of Chemistry and Materials, Guizhou Normal University, Guiyang 550025, People's Republic of China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.7253 (3)	0.0591 (3)	0.66817 (13)	0.0919 (9)
H1	0.651006	0.050409	0.695704	0.110*
C2	0.7075 (3)	0.1621 (3)	0.62354 (11)	0.0742 (7)
H2A	0.624015	0.220782	0.621615	0.089*
C3	0.81394 (19)	0.17790 (19)	0.58177 (8)	0.0463 (4)
C4	0.9309 (2)	0.0840 (2)	0.58683 (10)	0.0609 (5)
H4A	1.004185	0.087341	0.558610	0.073*
C5	0.9390 (2)	−0.0149 (2)	0.63385 (10)	0.0667 (6)
H5	1.020240	−0.076092	0.636632	0.080*
C6	0.79258 (19)	0.28873 (19)	0.53250 (8)	0.0473 (4)
C7	0.9955 (2)	0.40582 (19)	0.41158 (8)	0.0489 (4)
H7	1.083856	0.354661	0.422515	0.059*
C8	0.98323 (19)	0.49382 (19)	0.35638 (8)	0.0460 (4)
C9	1.0836 (2)	0.5171 (2)	0.31289 (10)	0.0654 (6)
H9	1.179848	0.477934	0.313557	0.079*
C10	1.0161 (2)	0.6101 (2)	0.26729 (9)	0.0586 (5)
H10	1.059597	0.644158	0.232345	0.070*
C11	0.87511 (17)	0.64211 (17)	0.28315 (7)	0.0381 (4)
C12	0.750000	0.7323 (2)	0.250000	0.0396 (5)
C13	0.6840 (2)	0.8234 (2)	0.29927 (9)	0.0551 (5)
H13A	0.640215	0.767122	0.329741	0.083*
H13B	0.606398	0.881573	0.277501	0.083*
H13C	0.764019	0.878489	0.321432	0.083*
C14	0.4127 (3)	−0.1197 (2)	0.58315 (11)	0.0723 (6)
H14	0.476237	−0.113746	0.551062	0.087*
C15	0.3634 (3)	−0.2175 (3)	0.68300 (14)	0.0919 (8)
H15A	0.424027	−0.191611	0.722457	0.138*
H15B	0.330856	−0.310707	0.685989	0.138*
H15C	0.275520	−0.159129	0.675540	0.138*
C16	0.5888 (3)	−0.2883 (3)	0.63276 (18)	0.1076 (10)
H16A	0.636632	−0.272935	0.594688	0.161*
H16B	0.561039	−0.382909	0.635006	0.161*
H16C	0.658808	−0.264764	0.670024	0.161*
N1	0.8393 (2)	−0.0286 (2)	0.67512 (9)	0.0751 (6)
N2	0.90644 (16)	0.30549 (15)	0.49623 (7)	0.0474 (4)
H2	0.990163	0.260073	0.504248	0.057*
N3	0.88600 (16)	0.39732 (15)	0.44574 (7)	0.0471 (4)
N4	0.85684 (15)	0.57098 (14)	0.33751 (6)	0.0399 (3)
H4	0.776956	0.574251	0.357126	0.048*
N5	0.45315 (19)	−0.20432 (19)	0.63060 (9)	0.0668 (5)
O1	0.67588 (15)	0.35883 (16)	0.52466 (7)	0.0655 (4)
O1W	0.38719 (16)	0.41435 (15)	0.56803 (7)	0.0652 (4)
H1WA	0.474440	0.401328	0.555831	0.098*
H1WB	0.351671	0.483779	0.546739	0.098*
O2W	0.21742 (16)	0.19334 (16)	0.50945 (8)	0.0703 (4)
H2WA	0.274507	0.254823	0.528413	0.105*
H2WB	0.252977	0.118792	0.525951	0.105*
O2	0.2988 (2)	−0.04786 (17)	0.57736 (8)	0.0862 (5)

The solvent was evaporated under reduced pressure to obtain a yellow solid. The yellow solids (0.2 mmol) were dissolved in a mixed solution (45 ml, C_2H_5OH/DMF , 8:1, v/v). Single crystals were obtained by slow evaporation at room temperature.

Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the $d(C-H) = 0.97-0.99 \text{ \AA}$ [3].

Comment

Acylhydrazones are Schiff base compounds obtained by condensation of hydrazine and aldehyde or ketone. It owns the donor atom nitrogen and oxygen. Diacylhydrazone Schiff bases can be easily assembled with different transition metal ions in different coordination modes [5]. Acylhydrazones have received much attention because of their particular physical, chemical, and antibacterial activities based on their unique structure [6]. In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one half of the target molecule, one DMF molecule and two water molecules. The dihedral angle of the two pyrrole rings is 81.2° . The dihedral angle between the pyridine rings is 75.59° . The bond lengths and bond angles are in the normal ranges [7]. The N atoms in the two central pyrrole rings are in opposite directions (see the Figure). The hydrogen bonds between the target compound and water molecules extend the framework into a 2D structure.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Science and Technology Foundation of Guizhou Province (grant number [2019]2792, [2018]5779–14, [2016]7362), the Provincial General Project of University Student Innovation and Entrepreneurship Fund, China (No. 20195200881).

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

References

1. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, 42, 339–341.
2. BRUKER. APEX2, SAINT and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2012.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, C71, 3–8.

4. Brandenburg K. *DIAMOND*. Visual Crystal Structure Information System. Ver. 4.0; Crystal Impact: Bonn, Germany, 2015.
5. Mazur L., Jarzemska K. N., Radoslaw K. Substituent and solvent effects on intermolecular interactions in crystals of *N*-acylhydrazone derivatives: single-crystal X-ray, solid-state NMR, and computational studies. *Cryst. Growth Des.* 2014, 14, 2263–2281.
6. Zhang Q. L., Feng G.-W. Synthesis, crystal structure and antibacterial activity of Schiff base Ni^{II} complexes containing Schiff Base and imidazole. *Chin. J. Inorg. Chem.* 2014, 30, 1025–1030.
7. Zhang L., Wu W.-N., Wang Y. Crystal structure of (E)-2-(4-ethoxycarbonyl-3,5-dimethyl-2-(pyrrole-2-ylmethyleamino)-3',6'-dihydroxylspiro[isindoline-1,9'-xanthen]-3-one-methanol (1/1), $C_{31}H_{29}N_3O_7$. *Z. Kristallogr.* 2021, 236, 239–241.