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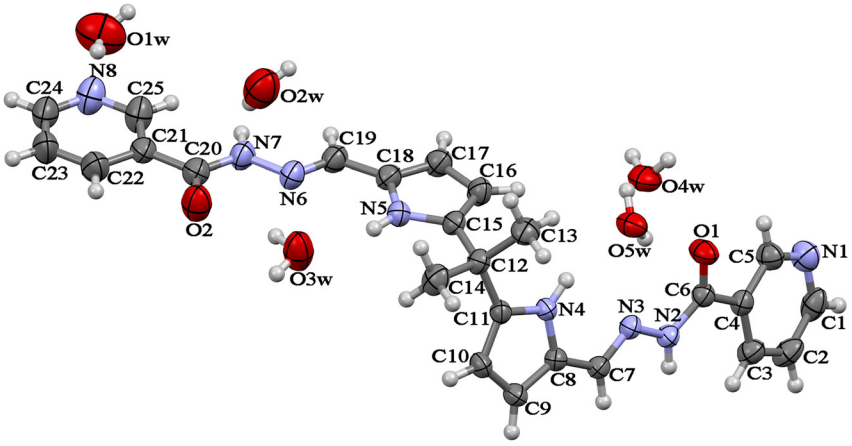
Crystal structure of *N*′,*N*‴-((1*E*,1′*E*)-(propane-2,2-diylbis(1*H*-pyrrole-5,2diyl)) bis(methaneylylidene))di(nicotinohydrazide) pentahydrate, $C_{25}H_{24}N_8O_2{\cdot}5H_2O$



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Abstract

$C_{25}H_{34}N_8O_7$, triclinic, $P\overline{1}$ (no. 2), a = 7.507(2) Å, b = 8.0276(18) Å, c = 23.653(6) Å, α = 93.062(8)°, β = 97.015(9)°, γ = 99.478(8)°, V = 1391.4(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0473, $wR_{ref}(F^2)$ = 0.1477, T = 273 K.

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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:	0.13 × 0.12 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	D8/APEX2, φ and ω
θ_{max}, completeness:	28.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	38216, 6902, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 5591
$N(param)_{refined}$:	399
Programs:	Bruker [1], Olex2 [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 nicotinohydrazide (0.55 g, 4.0 mmol) in a three-necked flask. The reaction mixture was refluxed for 12 h and then cooled to room temperature. The yellow precipitated residue was removed from the solution by filtration and then washed with ethanol three times. The yellow solids (0.2 mmol) were dissolved in a solution mixture (45 ml, C_2H_5OH/DMF, 8:1, *v*/*v*). Single crystals were obtained by slow evaporation at room temperature.

Experimental details

Using Olex2 [2], the structure was solved using Charge Flipping and refined with ShelXL [3]. All hydrogen atoms

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.31455 (14)	0.30477 (16)	−0.00059 (5)	0.0516 (3)
O2	0.1278 (2)	0.26745 (17)	0.52967 (6)	0.0741 (4)
N1	0.2778 (2)	0.60972 (19)	−0.13336 (6)	0.0571 (4)
N2	0.01051 (15)	0.24465 (14)	−0.00012 (4)	0.0360 (2)
H2	−0.0977	0.2486	−0.0162	0.043*
N3	0.03956 (15)	0.16475 (15)	0.04957 (4)	0.0368 (2)
N4	0.08513 (14)	0.02508 (14)	0.15533 (4)	0.0346 (2)
H4	0.1878	0.0692	0.1453	0.042*
N5	0.26189 (16)	0.19170 (15)	0.33157 (5)	0.0409 (3)
H5	0.2116	0.1275	0.3552	0.049*
N6	0.26048 (19)	0.38180 (17)	0.43730 (5)	0.0494 (3)
N7	0.26017 (19)	0.48780 (18)	0.48561 (5)	0.0508 (3)
H7	0.3040	0.5941	0.4868	0.061*
N8	0.2590 (3)	0.8095 (2)	0.63334 (7)	0.0700 (4)
C1	0.1158 (3)	0.6050 (2)	−0.16472 (6)	0.0551 (4)
H1	0.1102	0.6704	−0.1960	0.066*
C2	−0.0419 (3)	0.5093 (2)	−0.15323 (7)	0.0549 (4)
H2A	−0.1513	0.5097	−0.1764	0.066*
C3	−0.0367 (2)	0.41155 (19)	−0.10663 (6)	0.0472 (3)
H3	−0.1426	0.3467	−0.0976	0.057*
C4	0.12941 (19)	0.41265 (17)	−0.07382 (5)	0.0380 (3)
C5	0.2818 (2)	0.5138 (2)	−0.08903 (7)	0.0485 (3)
H5A	0.3934	0.5148	−0.0670	0.058*
C6	0.15861 (18)	0.31599 (17)	−0.02221 (5)	0.0369 (3)
C7	−0.10185 (18)	0.08873 (17)	0.06882 (5)	0.0358 (3)
H7A	−0.2168	0.0820	0.0482	0.043*
C8	−0.08123 (17)	0.01387 (16)	0.12258 (5)	0.0348 (3)
C9	−0.21097 (18)	−0.06612 (19)	0.15317 (6)	0.0415 (3)
H9	−0.3358	−0.0916	0.1414	0.050*
C10	−0.11995 (19)	−0.10220 (19)	0.20550 (6)	0.0414 (3)
H10	−0.1741	−0.1558	0.2346	0.050*
C11	0.06360 (17)	−0.04415 (16)	0.20607 (5)	0.0347 (3)
C12	0.22407 (19)	−0.04107 (18)	0.25206 (6)	0.0387 (3)
C13	0.3842 (2)	−0.1005 (2)	0.22636 (7)	0.0512 (4)
H13A	0.4252	−0.0238	0.1990	0.077*
H13B	0.3447	−0.2121	0.2078	0.077*
H13C	0.4824	−0.1025	0.2563	0.077*
C14	0.1661 (3)	−0.1599 (2)	0.29760 (7)	0.0521 (4)
H14A	0.2665	−0.1559	0.3273	0.078*
H14B	0.1301	−0.2736	0.2805	0.078*
H14C	0.0655	−0.1248	0.3136	0.078*
C15	0.28614 (18)	0.14014 (18)	0.27761 (5)	0.0384 (3)
C16	0.3718 (2)	0.2793 (2)	0.25378 (6)	0.0484 (3)
H16	0.4059	0.2809	0.2173	0.058*
C17	0.3987 (2)	0.4188 (2)	0.29444 (7)	0.0493 (3)
H17	0.4529	0.5290	0.2897	0.059*
C18	0.3300 (2)	0.36184 (19)	0.34249 (6)	0.0441 (3)
C19	0.3259 (2)	0.4544 (2)	0.39570 (6)	0.0488 (3)
H19	0.3719	0.5699	0.4001	0.059*
C20	0.1898 (2)	0.4201 (2)	0.53030 (7)	0.0500 (4)
C21	0.1908 (2)	0.5373 (2)	0.58180 (7)	0.0498 (4)
C22	0.1174 (3)	0.4708 (3)	0.62791 (8)	0.0682 (5)
H22	0.0687	0.3561	0.6266	0.082*
C23	0.1163 (3)	0.5747 (3)	0.67600 (8)	0.0754 (6)
H23	0.0669	0.5313	0.7075	0.090*
C24	0.1878 (3)	0.7402 (3)	0.67683 (8)	0.0666 (5)
H24	0.1872	0.8093	0.7096	0.080*
C25	0.2599 (3)	0.7079 (3)	0.58660 (8)	0.0638 (5)
H25	0.3098	0.7552	0.5558	0.077*
O1W[a]	0.3779 (5)	1.1504 (3)	0.61993 (18)	0.1202 (12)
H1WA[a]	0.3473	1.0461	0.6250	0.180*
H1WB[a]	0.4729	1.1552	0.6035	0.180*
O1WA[b]	0.305 (3)	1.153 (2)	0.6512 (10)	0.1202 (12)
H1WC[b]	0.4024	1.1141	0.6492	0.180*
H1WD[b]	0.2182	1.0728	0.6541	0.180*
O2W	0.3990 (4)	0.8435 (3)	0.46431 (9)	0.0973 (6)
H2WA	0.484 (4)	0.862 (5)	0.4456 (15)	0.135 (15)*
H2WB	0.319 (8)	0.910 (8)	0.462 (3)	0.34 (5)*
O3W	0.1131 (3)	0.0122 (2)	0.43053 (7)	0.0822 (5)
H3WA	0.119 (4)	0.107 (3)	0.4508 (10)	0.091 (8)*
H3WB	0.036 (4)	−0.048 (4)	0.4469 (13)	0.122 (11)*
O4W	0.37395 (17)	0.8032 (2)	0.04438 (6)	0.0649 (3)
H4WA	0.413 (4)	0.902 (2)	0.0578 (11)	0.095 (9)*
H4WB	0.468 (3)	0.765 (3)	0.0342 (11)	0.089 (8)*
O5W	0.41244 (15)	0.13147 (16)	0.10116 (5)	0.0533 (3)
H5WA	0.506 (3)	0.206 (2)	0.1154 (9)	0.073 (6)*
H5WB	0.357 (3)	0.185 (3)	0.0762 (9)	0.085 (7)*

[a]Occupancy: 0.850(7), [b]Occupancy: 0.150(7).

were positioned geometrically, with *d* (C–H) = 0.97–0.99 Å, U_{iso}(H) = 1.2 times U_{eq}(C) and U_{iso}(H) = 1.5 times U_{eq}(O).

Comment

Acylhydrazone compounds are prepared by dehydration after nucleophilic addition reaction between hydrazine and aldehyde or ketone. These compounds have a variety of biological activities, such as antibacterial, insecticidal, anti-tumor, etc. [5]. Due to the strong nucleophilic ability of nitrogen atoms on the pyrrole ring, acylhydrazone Schiff base compounds are easy to be assembled with different transition metal ions to form novel complexes of acylhydrazone Schiff base [6].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one target molecule and five water molecules. The dihedral angle of two central pyrrole rings is 70.2°. The bond lengths and bond angles are in the normal ranges [7]. The hydrogen

bonds between the target compound and water molecules connect all molecules to a *2D* framework.

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

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