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1,2-Bis(1*H*-pyrrol-2-ylmethylene)diazane monohydrate

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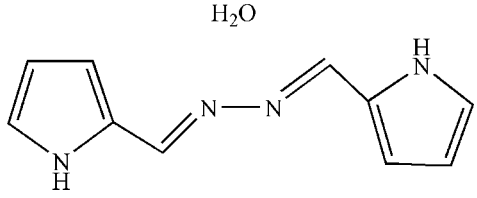
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.065; wR factor = 0.191; data-to-parameter ratio = 12.3.

The molecular structure of title compound, $C_{10}H_{10}N_4\cdot H_2O$, has an inversion centre located on the mid-point of the N—N bond of the molecule. A twofold rotation axis passes through the water O atom. In the crystal structure, a two-dimensional network is constructed through N—H···O and O—H···N hydrogen bonds.

Related literature

For the biological properties of azines, see: Khodair & Bertrand (1998). For their potential applications, see: Espinet *et al.* (1998); Nalwa *et al.* (1993); Schweizer *et al.* (1993).



Experimental

Crystal data

$C_{10}H_{10}N_4\cdot H_2O$
M_r = 204.24
Monoclinic, $P2/c$
a = 12.006 (4) Å
b = 6.5806 (19) Å
c = 6.914 (2) Å
β = 105.253 (6)°
V = 527.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.23 × 0.17 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.980, T_{max} = 0.991
2143 measured reflections
910 independent reflections
583 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.191
S = 1.05
910 reflections
74 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1W	0.86	2.07	2.910 (3)	167
O1W—H1W···N2^{i}	0.826 (10)	2.132 (16)	2.917 (3)	159 (4)

Symmetry code: (i) $x, -y+1, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2834).

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supporting information

Acta Cryst. (2009). E**65**, o1787 [doi:10.1107/S1600536809025422]

1,2-Bis(1*H*-pyrrol-2-ylmethylene)diazane monohydrate

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S1. Comment

Recently, dinucleating diazine ligands containing a single N—N bond have received considerable attention due to their biological properties (Khodair, *et al.* 1998), their potential applicability in bond formations (Schweizer, *et al.*, 1993), the design of liquid crystals (Espinet, *et al.*, 1998) as well as non-linear optical materials (Nalwa, *et al.*, 1993). we now report the structure of the title compound, (I).

Compound (I) consists of a 1,2-bis((1*H*-pyrrol-2-yl)methylene)hydrazine organic molecule and a crystal water molecule (Fig.1). The molecular structure of title compound has an inversion centre located on the midpoint of the N—N bond of the molecule. A two-fold rotation axis pass through the water O atom. The N1/C1–C4 ring in (I) is coplanar, in which the C–N bond distances range from 1.344 (4) to 1.377 (4) Å. However, C5—N2 [1.308 (4) Å] is typical for a C═N double bond. The N2—N2b bond distance is 1.395 (5), indicating a N—N single bond.

Two intra and intermolecular hydrogen bonds N—H···O and O—H···N (Table 1) help to establish the molecular conformation, and constructing infinite two-dimensional network along [100] plane (Fig. 2).

S2. Experimental

An ethanol solution containing hydrazine hydrate (0.20 g, 4 mmol) was added dropwise with constant stirring and slow heating to a solution of pyrrole-2-carboxaldehyde (0.38 g, 4 mmol) in the same solvent with five drops of acetic acid. The solution was refluxed for 2 h. Then the resultant solution was filtered. Red crystals suitable for X-ray studies were obtained by slow evaporation of the ethanol solution [yield: 65%].

S3. Refinement

The water H atom was found from a difference Fourier map and refined freely. Other H atoms were treated as riding, with C—H distances of 0.93 Å and N—H distances of 0.86 Å, and were refined as riding with $U_{iso}(H)=1.2U_{eq}$(C and N).

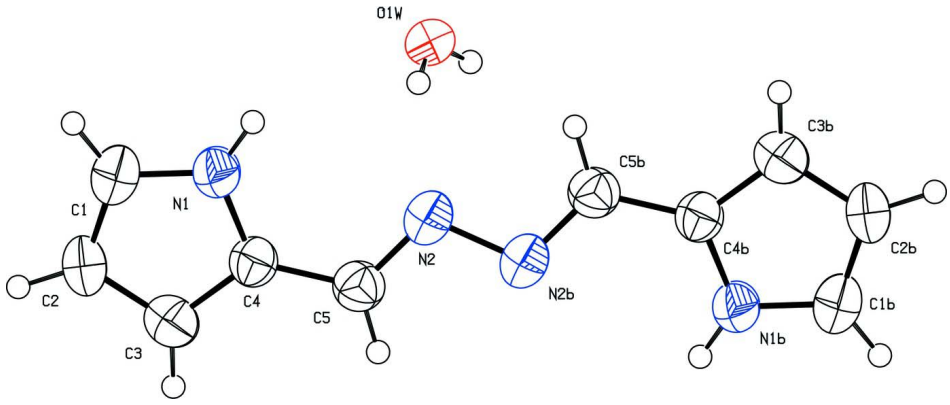

Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

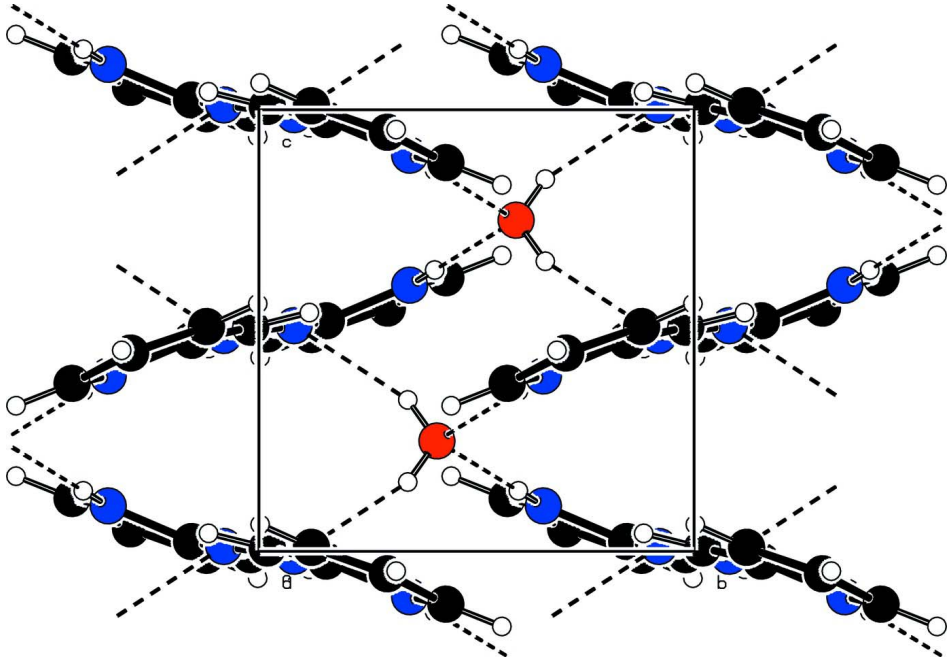

Figure 2

Two-dimensional structure of (I) along [100] direction. Hydrogen bonds are shown in the dashing line.

1,2-Bis(1*H*-pyrrol-2-ylmethylene)diazane monohydrate

Crystal data

$C_{10}H_{10}N_4 \cdot H_2O$
$M_r = 204.24$
Monoclinic, $P2/c$
Hall symbol: -P 2yc
$a = 12.006$ (4) Å
$b = 6.5806$ (19) Å
$c = 6.914$ (2) Å
$\beta = 105.253$ (6)°
$V = 527.0$ (3) Å^3
$Z = 2$
$F(000) = 216$
$D_x = 1.287$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 519 reflections
$\theta = 3.1$–23.4°
$\mu = 0.09$ mm^{-1}

T = 296 K
Block, red

0.23 × 0.17 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.980, T_{max} = 0.991

2143 measured reflections
910 independent reflections
583 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.1°, θ_{min} = 1.8°
h = −14→12
k = −7→7
l = −8→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.191
S = 1.05
910 reflections
74 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1036P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.06 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1938 (3)	0.5705 (6)	0.1197 (5)	0.0584 (11)
H1B	0.1862	0.4418	0.1702	0.070*
C2	0.1062 (3)	0.7050 (6)	0.0509 (6)	0.0622 (11)
H2C	0.0287	0.6846	0.0448	0.075*
C3	0.1546 (3)	0.8794 (5)	−0.0091 (6)	0.0614 (11)
H3A	0.1152	0.9973	−0.0607	0.074*
C4	0.2711 (3)	0.8453 (5)	0.0221 (5)	0.0456 (9)
C5	0.3587 (3)	0.9809 (5)	−0.0076 (5)	0.0489 (9)
H5A	0.3398	1.1158	−0.0408	0.059*
N1	0.2931 (2)	0.6536 (4)	0.1029 (4)	0.0499 (9)
H1A	0.3597	0.5962	0.1372	0.060*
N2	0.4646 (2)	0.9192 (4)	0.0110 (4)	0.0481 (8)

O1W	0.5000	0.4090 (5)	0.2500	0.0531 (10)
H1W	0.487 (3)	0.342 (5)	0.343 (4)	0.055 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.056 (2)	0.056 (2)	0.065 (2)	−0.0124 (18)	0.0201 (17)	0.0022 (18)
C2	0.041 (2)	0.064 (2)	0.084 (3)	−0.0099 (17)	0.0200 (19)	0.000 (2)
C3	0.050 (2)	0.056 (2)	0.079 (3)	0.0044 (17)	0.0179 (18)	−0.0031 (19)
C4	0.0428 (18)	0.0454 (18)	0.0471 (19)	−0.0032 (15)	0.0095 (14)	−0.0033 (15)
C5	0.048 (2)	0.0484 (19)	0.050 (2)	0.0017 (15)	0.0106 (15)	0.0009 (16)
N1	0.0400 (16)	0.0477 (17)	0.0600 (19)	−0.0018 (12)	0.0099 (13)	0.0041 (14)
N2	0.0531 (18)	0.0462 (16)	0.0456 (17)	−0.0090 (12)	0.0142 (13)	−0.0017 (13)
O1W	0.051 (2)	0.0377 (19)	0.074 (3)	0.000	0.0219 (19)	0.000

Geometric parameters (Å, º)

C1—N1	1.344 (4)	C4—N1	1.377 (4)
C1—C2	1.361 (5)	C4—C5	1.435 (5)
C1—H1B	0.9300	C5—N2	1.308 (4)
C2—C3	1.397 (5)	C5—H5A	0.9300
C2—H2C	0.9300	N1—H1A	0.8600
C3—C4	1.376 (5)	N2—N2^{i}	1.395 (5)
C3—H3A	0.9300	O1W—H1W	0.826 (10)
N1—C1—C2	109.1 (3)	C3—C4—C5	129.0 (3)
N1—C1—H1B	125.5	N1—C4—C5	123.9 (3)
C2—C1—H1B	125.5	N2—C5—C4	121.5 (3)
C1—C2—C3	107.1 (3)	N2—C5—H5A	119.2
C1—C2—H2C	126.4	C4—C5—H5A	119.2
C3—C2—H2C	126.4	C1—N1—C4	109.1 (3)
C4—C3—C2	107.7 (3)	C1—N1—H1A	125.4
C4—C3—H3A	126.1	C4—N1—H1A	125.4
C2—C3—H3A	126.1	C5—N2—N2^{i}	110.9 (3)
C3—C4—N1	106.9 (3)		

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1*W*	0.86	2.07	2.910 (3)	167
O1*W*—H1*W*···N2^{ii}	0.83 (1)	2.13 (2)	2.917 (3)	159 (4)

Symmetry code: (ii) *x*, −*y*+1, *z*+1/2.