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$N^2,N^{2'}$-Bis[4-(dimethylamino)benzylidene]pyridine-2,6-dicarbohydrazide monohydrate

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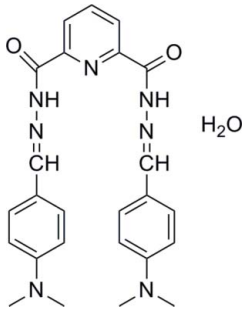
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.204; data-to-parameter ratio = 12.7.

In the title compound, $C_{25}H_{27}N_7O_2\cdot H_2O$, the bis[4-(dimethylamino)benzylidene]pyridine-2,6-dicarbohydrazide molecule and the water molecule are located on a twofold rotation axis. The benzene and pyridine rings form a dihedral angle of 17.13 (7)°. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the molecules into a two-dimensional supermolecular structure parallel to the *ab* plane.

Related literature

For related structures, see: Cheng *et al.* (2007); Cheng & Liu (2008); Jia, Hu *et al.* (2006); Jia, Shi *et al.* (2006).



Experimental

Crystal data

$C_{25}H_{27}N_7O_2\cdot H_2O$
M_r = 475.54
Monoclinic, $C2/c$
a = 8.5718 (11) Å
b = 10.2802 (14) Å
c = 27.112 (3) Å
β = 97.865 (1)°
V = 2366.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.36 × 0.31 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.968, T_{max} = 0.986
5774 measured reflections
2076 independent reflections
1348 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.204
S = 1.05
2076 reflections
163 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.23	2.967 (4)	143
O2—H2···O1^{ii}	0.85 (3)	2.04 (4)	2.844 (3)	157 (3)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2479).

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supplementary materials

Acta Cryst. (2010). E**66**, o2718 [**doi:10.1107/S1600536810038778**]

N^2,$N^{2'}$-Bis[4-(dimethylamino)benzylidene]pyridine-2,6-dicarbohydrazide monohydrate

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Comment

Schiff base ligands containing the pyridine ring have received considerable attention during the last decades, mainly because their coordinative and electronic properties. For this reason, much effort has been devoted to develop efficient routes for the synthesis of these classes of compounds. In this paper, we report the crystal structure of the title compound, obtained by the reaction of pyridine-2,6-dicarbohydrazide and 4-(dimethylamino)benzaldehyde.

In the title compound (Fig. 1), the bis(4-(dimethylamino)benzylidene)pyridine-2,6-dicarbohydrazide molecule and the water molecule possess crystallographic imposed twofold rotation symmetry. Bond lengths and angles are normal and correspond to those observed in related compounds (Cheng *et al.*, 2007; Cheng & Liu, 2008; Jia, Hu *et al.*, 2006; Jia, Shi *et al.*, 2006). The dihedral angle formed by the benzene ring and the pyridine ring is 17.13 (7)°. In the crystal packing, a two-dimensional supermolecular structure parallel to the *ab* plane is formed by N—H···O and O—H···O intermolecular contacts (Table 1).

Experimental

To a solution of pyridine-2,6-dicarbohydrazide (3 mmol) in ethanol (30 ml) was added 4-(dimethylamino)benzaldehyde (6 mmol). The mixture was refluxed with stirring for 8 h. An red precipitate was then obtained. Red crystals suitable for X-ray diffraction analysis formed after several weeks on slow evaporation of an ethanol solution at room temperature. Elemental analysis: calculated for $C_{25}H_{29}N_7O_3$: C 63.14, H 6.15, N 20.62%; found: C 63.28, H 6.22, N 20.49%.

Refinement

The independent water H atom was located in a difference Fourier map and refined with the O—H bond constrained to 0.85 Å and U_{iso}(H) = 1.2 U_{eq}(O). All other H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.96 Å, N—H =0.86 Å, and with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C) for methyl H atoms

Figures

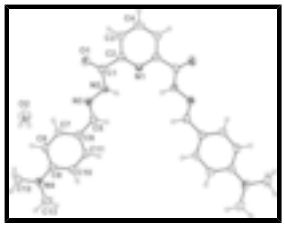

Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme and 30% probability displacement ellipsoids. Unlabelled atoms are related to labelled atoms by (-x, y, 0.5-z).

N^2,$N^{2'}$-Bis[4-(dimethylamino)benzylidene]pyridine-2,6- dicarbohydrazide monohydrate

Crystal data

$C_{25}H_{27}N_7O_2 \cdot H_2O$	$F(000) = 1008$
$M_r = 475.54$	$D_x = 1.332$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1533 reflections
$a = 8.5718$ (11) Å	$\theta = 3.0$–$24.4°$
$b = 10.2802$ (14) Å	$\mu = 0.09$ mm^{-1}
$c = 27.112$ (3) Å	$T = 298$ K
$\beta = 97.865$ (1)°	Block, red
$V = 2366.7$ (5) Å^3	0.36 × 0.31 × 0.16 mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2076 independent reflections
Radiation source: fine-focus sealed tube	1348 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.039$
phi and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.968$, $T_{max} = 0.986$	$k = -12 \rightarrow 12$
5774 measured reflections	$l = -32 \rightarrow 17$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.062$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.204$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.1275P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2076 reflections	$(\Delta/\sigma)_{max} < 0.001$
163 parameters	$\Delta\rho_{max} = 0.17$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.59$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.0000	0.0318 (3)	0.2500	0.0419 (8)
N2	0.1517 (3)	0.1645 (2)	0.18533 (9)	0.0456 (6)
H2A	0.0721	0.1938	0.1981	0.068*
N3	0.2319 (3)	0.2465 (2)	0.15743 (8)	0.0448 (7)
N4	0.4368 (3)	0.7693 (2)	0.05148 (10)	0.0556 (7)
O1	0.3055 (3)	−0.0104 (2)	0.17635 (9)	0.0669 (7)
O2	1.0000	0.3442 (4)	0.2500	0.0927 (13)
H2	0.934 (4)	0.398 (3)	0.2352 (16)	0.111*
C1	0.1949 (3)	0.0402 (3)	0.19306 (10)	0.0445 (7)
C2	0.0935 (3)	−0.0351 (3)	0.22381 (10)	0.0411 (7)
C3	0.0976 (3)	−0.1702 (3)	0.22298 (11)	0.0488 (8)
H3	0.1650	−0.2141	0.2046	0.059*
C4	0.0000	−0.2371 (4)	0.2500	0.0535 (11)
H4	0.0000	−0.3276	0.2500	0.064*
C5	0.1730 (3)	0.3604 (3)	0.15200 (10)	0.0462 (7)
H5	0.0817	0.3786	0.1657	0.055*
C6	0.2429 (3)	0.4630 (3)	0.12527 (10)	0.0424 (7)
C7	0.3772 (3)	0.4455 (3)	0.10227 (11)	0.0452 (7)
H7	0.4255	0.3643	0.1035	0.054*
C8	0.4387 (3)	0.5447 (3)	0.07816 (11)	0.0474 (8)
H8	0.5276	0.5292	0.0628	0.057*
C9	0.3728 (3)	0.6699 (3)	0.07561 (10)	0.0420 (7)
C10	0.2390 (3)	0.6870 (3)	0.09890 (11)	0.0494 (8)
H10	0.1917	0.7685	0.0982	0.059*
C11	0.1755 (3)	0.5864 (3)	0.12276 (11)	0.0489 (8)
H11	0.0854	0.6010	0.1376	0.059*
C12	0.5716 (4)	0.7484 (3)	0.02620 (13)	0.0685 (10)
H12A	0.6567	0.7141	0.0492	0.103*
H12B	0.6031	0.8295	0.0130	0.103*
H12C	0.5450	0.6877	−0.0005	0.103*
C13	0.3639 (4)	0.8964 (3)	0.04783 (13)	0.0639 (9)
H13A	0.2669	0.8923	0.0255	0.096*
H13B	0.4335	0.9576	0.0354	0.096*
H13C	0.3430	0.9235	0.0802	0.096*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0486 (18)	0.0423 (18)	0.0350 (17)	0.000	0.0068 (15)	0.000
N2	0.0534 (14)	0.0424 (14)	0.0433 (13)	−0.0004 (11)	0.0155 (11)	0.0021 (11)
N3	0.0518 (14)	0.0457 (15)	0.0376 (13)	−0.0025 (11)	0.0082 (11)	0.0021 (11)
N4	0.0561 (15)	0.0533 (16)	0.0613 (16)	−0.0005 (12)	0.0220 (13)	0.0100 (13)
O1	0.0674 (14)	0.0616 (14)	0.0780 (16)	0.0146 (11)	0.0330 (13)	0.0048 (12)
O2	0.100 (3)	0.081 (3)	0.101 (3)	0.000	0.030 (3)	0.000
C1	0.0497 (16)	0.0449 (17)	0.0392 (16)	0.0056 (13)	0.0068 (13)	−0.0040 (12)
C2	0.0472 (15)	0.0377 (15)	0.0370 (15)	0.0009 (12)	0.0013 (13)	−0.0015 (12)
C3	0.0497 (17)	0.0461 (17)	0.0495 (17)	0.0057 (14)	0.0031 (14)	−0.0055 (14)
C4	0.055 (3)	0.036 (2)	0.066 (3)	0.000	−0.005 (2)	0.000
C5	0.0480 (16)	0.0520 (18)	0.0400 (15)	0.0003 (14)	0.0107 (13)	0.0012 (13)
C6	0.0466 (15)	0.0469 (16)	0.0341 (14)	−0.0018 (13)	0.0072 (12)	0.0000 (12)
C7	0.0443 (15)	0.0449 (16)	0.0472 (16)	0.0026 (13)	0.0091 (13)	−0.0015 (13)
C8	0.0422 (15)	0.0556 (18)	0.0472 (17)	0.0008 (13)	0.0162 (13)	−0.0020 (14)
C9	0.0415 (15)	0.0484 (17)	0.0369 (15)	−0.0024 (13)	0.0080 (12)	0.0008 (13)
C10	0.0512 (17)	0.0456 (17)	0.0533 (18)	0.0064 (14)	0.0134 (14)	0.0008 (14)
C11	0.0503 (16)	0.0510 (18)	0.0496 (17)	0.0024 (14)	0.0220 (14)	0.0010 (14)
C12	0.069 (2)	0.072 (2)	0.071 (2)	−0.0130 (17)	0.0331 (19)	0.0018 (18)
C13	0.073 (2)	0.057 (2)	0.065 (2)	−0.0058 (17)	0.0188 (18)	0.0117 (17)

Geometric parameters (Å, °)

N1—C2	1.333 (3)	C5—H5	0.9300
N1—C2^{i}	1.333 (3)	C6—C11	1.392 (4)
N2—C1	1.339 (3)	C6—C7	1.394 (4)
N2—N3	1.378 (3)	C7—C8	1.356 (4)
N2—H2A	0.8600	C7—H7	0.9300
N3—C5	1.276 (3)	C8—C9	1.404 (4)
N4—C9	1.368 (3)	C8—H8	0.9300
N4—C12	1.438 (4)	C9—C10	1.394 (4)
N4—C13	1.446 (4)	C10—C11	1.371 (4)
O1—C1	1.222 (3)	C10—H10	0.9300
O2—H2	0.85 (3)	C11—H11	0.9300
C1—C2	1.499 (4)	C12—H12A	0.9600
C2—C3	1.390 (4)	C12—H12B	0.9600
C3—C4	1.370 (3)	C12—H12C	0.9600
C3—H3	0.9300	C13—H13A	0.9600
C4—C3^{i}	1.370 (3)	C13—H13B	0.9600
C4—H4	0.9300	C13—H13C	0.9600
C5—C6	1.455 (4)		
C2—N1—C2^{i}	117.8 (3)	C8—C7—H7	119.4
C1—N2—N3	121.5 (2)	C6—C7—H7	119.4
C1—N2—H2A	119.3	C7—C8—C9	122.2 (3)
N3—N2—H2A	119.3	C7—C8—H8	118.9

C5—N3—N2	114.0 (2)	C9—C8—H8	118.9
C9—N4—C12	121.2 (3)	N4—C9—C10	122.1 (3)
C9—N4—C13	120.6 (2)	N4—C9—C8	121.5 (2)
C12—N4—C13	118.0 (2)	C10—C9—C8	116.3 (2)
O1—C1—N2	124.1 (3)	C11—C10—C9	121.6 (3)
O1—C1—C2	121.7 (3)	C11—C10—H10	119.2
N2—C1—C2	114.3 (2)	C9—C10—H10	119.2
N1—C2—C3	122.9 (3)	C10—C11—C6	121.4 (3)
N1—C2—C1	117.8 (2)	C10—C11—H11	119.3
C3—C2—C1	119.3 (2)	C6—C11—H11	119.3
C4—C3—C2	118.3 (3)	N4—C12—H12A	109.5
C4—C3—H3	120.8	N4—C12—H12B	109.5
C2—C3—H3	120.8	H12A—C12—H12B	109.5
C3—C4—C3^{i}	119.8 (4)	N4—C12—H12C	109.5
C3—C4—H4	120.1	H12A—C12—H12C	109.5
C3^{i}—C4—H4	120.1	H12B—C12—H12C	109.5
N3—C5—C6	122.7 (3)	N4—C13—H13A	109.5
N3—C5—H5	118.7	N4—C13—H13B	109.5
C6—C5—H5	118.7	H13A—C13—H13B	109.5
C11—C6—C7	117.3 (2)	N4—C13—H13C	109.5
C11—C6—C5	119.2 (2)	H13A—C13—H13C	109.5
C7—C6—C5	123.4 (3)	H13B—C13—H13C	109.5
C8—C7—C6	121.2 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O2^{ii}	0.86	2.23	2.967 (4)	143
O2—H2···O1^{iii}	0.85 (3)	2.04 (4)	2.844 (3)	157 (3)

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

Fig. 1