Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-[4-(Dimethylamino)benzylidene]-3,5-dihydroxybenzohydrazide monohydrate

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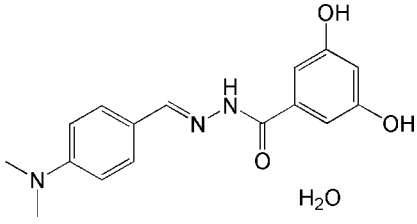
Received 20 November 2007; accepted 20 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.068; wR factor = 0.172; data-to-parameter ratio = 7.2.

In the title compound, $C_{16}H_{17}N_3O_3\cdot H_2O$, the dihedral angle between the two benzene rings is 7.4 (6)°. The Schiff base unit is nearly planar, with a mean deviation from the plane of 0.089 (8) Å. In the crystal structure, molecules are linked through intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds, forming layers parallel to the *ab* plane.

Related literature

For related structures, see: Brückner *et al.* (2000); Diao (2007); Diao *et al.* (2007); Harrop *et al.* (2003); Huang, Zhou *et al.* (2007); Li, Huang *et al.* (2007); Ren *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{17}N_3O_3\cdot H_2O$
M_r = 317.34
Monoclinic, *Ia*
a = 12.984 (3) Å
b = 4.6620 (9) Å
c = 26.040 (5) Å
β = 101.97 (3)°
V = 1542.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.974, T_{max} = 0.977
4115 measured reflections
1595 independent reflections
878 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.172
S = 1.01
1595 reflections
221 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O3^{i}	0.850 (11)	2.16 (6)	2.895 (8)	144 (9)
N1—H1A···O4^{ii}	0.902 (11)	2.14 (3)	2.999 (7)	159 (8)
O4—H4B···N2	0.848 (11)	2.46 (4)	3.175 (7)	143 (6)
O4—H4B···O3	0.848 (11)	2.24 (5)	2.936 (8)	139 (7)
O2—H2···O3^{iii}	0.82	1.99	2.694 (6)	144
O1—H1···O2^{iv}	0.82	2.06	2.766 (7)	145

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This project is supported by a research grant from Dalian Medical University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GD2022).

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  doi:10.1107/S1600536807061132

supplementary materials

Acta Cryst. (2007). E**63**, o4908 [**doi:10.1107/S1600536807061132**]

N'-[4-(Dimethylamino)benzylidene]-3,5-dihydroxybenzohydrazide monohydrate

Y.-P. Diao, J.-K. Zhang, S.-Q. Xie and T.-G. Kang

Comment

Schiff base compounds have received much attention in recent years. Some of the complexes have been found to have pharmacological and antitumor properties (Brückner *et al.*, 2000; Harrop *et al.*, 2003; Ren *et al.*, 2002). As part of our research programme on the Schiff base compounds (Diao *et al.*, 2007; Diao, 2007; Li, Huang *et al.*, 2007; Huang *et al.*, 2007), we report here the structure of the title compound.

The title compound consists of a Schiff base molecule and a lattice water molecule (Fig. 1). The dihedral angle between the two benzene rings is 7.4 (6) °. The Schiff base unit is nearly planar, with mean deviation from plane by 0.089 (8) Å.

In the crystal, molecules are linked through intermolecular hydrogen bonds of O–H···O, O—H···N and N–H···O type (Table 1), forming layers parallel to the *ab* plane (Fig. 2).

Experimental

4-Dimethylaminobenzaldehyde (1.0 mmol, 149.2 mg) and 3,5-dihydroxybenzoic acid hydrazide (1.0 mmol, 168.2 mg) were dissolved in a methanol solution (70 ml). The mixture was stirred at reflux for 1 h and cooled to room temperature. After keeping the solution in air for five days, yellow block-like crystals were formed.

Refinement

H1A, H4A and H4B were located from a difference Fourier map and refined isotropically, with N—H distances restrained to 0.90 (1) Å, for the water component the O—H distances were restrained to 0.85 (1) Å, and the H···H distance was restrained to 1.37 (2) Å. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with distances C—H 0.93–0.96 Å, and O—H 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O and methyl C).

Figures

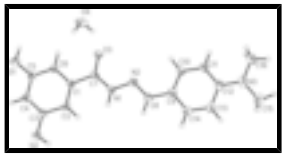

Fig. 1. The molecular structure of the title compound; displacement ellipsoids are drawn at the 30% probability level.

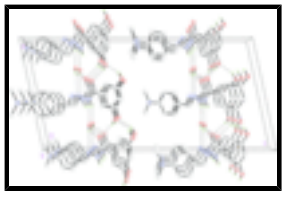

Fig. 2. O–H···O, O—H···N and N–H···O hydrogen bonding in the title compound, forming layers parallel to the *ab* plane.

N'-[4-(Dimethylamino)benzylidene]-3,5-dihydroxybenzohydrazide monohydrate

Crystal data

$C_{16}H_{17}N_3O_3 \cdot H_2O$	$F_{000} = 672$
$M_r = 317.34$	$D_x = 1.367$ Mg m^{-3}
Monoclinic, *Ia*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: I -2ya	Cell parameters from 419 reflections
a = 12.984 (3) Å	θ = 2.6–24.3º
b = 4.6620 (9) Å	μ = 0.10 mm^{-1}
c = 26.040 (5) Å	*T* = 298 (2) K
β = 101.97 (3)º	Block, yellow
V = 1542.0 (6) Å^3	0.27 × 0.23 × 0.23 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1595 independent reflections
Radiation source: fine-focus sealed tube	878 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.075$
T = 298(2) K	$\theta_{max} = 26.5$º
ω scans	$\theta_{min} = 3.2$º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	*h* = −16→16
$T_{min} = 0.974$, $T_{max} = 0.977$	*k* = −5→4
4115 measured reflections	*l* = −32→26

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.172$	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max} = 0.30$ e Å^{-3}
1595 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}
221 parameters	Extinction correction: none
6 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.6358 (4)	−0.0223 (13)	0.40242 (18)	0.0561 (14)
H1	0.6921	−0.0062	0.3930	0.084*
O2	0.2765 (3)	0.1330 (12)	0.33837 (19)	0.0564 (14)
H2	0.2368	0.2521	0.3216	0.085*
O3	0.6269 (3)	0.6887 (11)	0.25687 (17)	0.0460 (13)
O4	0.7220 (4)	0.1943 (13)	0.2180 (2)	0.0603 (15)
N1	0.4566 (4)	0.8107 (13)	0.22950 (19)	0.0410 (14)
N2	0.4820 (4)	1.0035 (14)	0.1938 (2)	0.0435 (14)
N3	0.4263 (5)	1.8785 (16)	−0.0003 (3)	0.069 (2)
C1	0.5024 (5)	0.4512 (15)	0.2978 (2)	0.0368 (16)
C2	0.3987 (5)	0.3982 (16)	0.2991 (2)	0.0420 (17)
H2A	0.3443	0.4912	0.2762	0.050*
C3	0.3774 (5)	0.1980 (16)	0.3363 (2)	0.0406 (16)
C4	0.4562 (5)	0.0604 (17)	0.3692 (2)	0.0399 (17)
H4	0.4407	−0.0742	0.3928	0.048*
C5	0.5586 (5)	0.1193 (16)	0.3677 (2)	0.0407 (17)
C6	0.5831 (5)	0.3110 (15)	0.3310 (2)	0.0384 (16)
H6	0.6527	0.3437	0.3289	0.046*
C7	0.5327 (5)	0.6580 (15)	0.2596 (2)	0.0363 (16)
C8	0.4024 (5)	1.1293 (16)	0.1662 (2)	0.0426 (17)
H8	0.3358	1.0916	0.1726	0.051*
C9	0.4132 (5)	1.3300 (17)	0.1251 (3)	0.0460 (18)
C10	0.5076 (5)	1.3966 (18)	0.1117 (3)	0.053 (2)
H10	0.5695	1.3154	0.1305	0.064*
C11	0.5117 (6)	1.5815 (18)	0.0710 (3)	0.055 (2)
H11	0.5764	1.6234	0.0627	0.066*
C12	0.4218 (6)	1.7053 (17)	0.0422 (3)	0.0536 (19)
C13	0.3264 (6)	1.6500 (18)	0.0565 (3)	0.062 (2)
H13	0.2650	1.7369	0.0384	0.074*
C14	0.3232 (6)	1.4649 (19)	0.0976 (3)	0.059 (2)
H14	0.2591	1.4299	0.1071	0.071*
C15	0.3331 (7)	2.014 (2)	−0.0305 (3)	0.080 (3)
H15A	0.2807	1.8704	−0.0428	0.120*

H15B	0.3505	2.1120	−0.0599	0.120*
H15C	0.3061	2.1488	−0.0088	0.120*
C16	0.5271 (7)	1.963 (2)	−0.0118 (3)	0.075 (3)
H16A	0.5664	2.0699	0.0172	0.113*
H16B	0.5154	2.0790	−0.0429	0.113*
H16C	0.5659	1.7941	−0.0173	0.113*
H4B	0.674 (4)	0.067 (10)	0.214 (3)	0.080*
H1A	0.390 (3)	0.78 (2)	0.234 (4)	0.080*
H4A	0.693 (5)	0.359 (6)	0.215 (4)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.043 (3)	0.089 (4)	0.038 (3)	0.014 (3)	0.012 (2)	0.020 (3)
O2	0.040 (3)	0.081 (4)	0.050 (3)	−0.004 (3)	0.011 (2)	0.018 (2)
O3	0.036 (2)	0.060 (4)	0.042 (3)	−0.001 (2)	0.0090 (19)	0.006 (2)
O4	0.039 (2)	0.074 (4)	0.069 (3)	−0.007 (3)	0.014 (2)	−0.007 (3)
N1	0.034 (3)	0.059 (4)	0.031 (3)	0.006 (3)	0.011 (2)	0.008 (3)
N2	0.046 (3)	0.048 (4)	0.038 (3)	−0.001 (3)	0.012 (3)	0.003 (3)
N3	0.074 (5)	0.079 (5)	0.053 (4)	0.008 (4)	0.012 (3)	0.021 (4)
C1	0.036 (4)	0.045 (4)	0.033 (3)	0.002 (3)	0.013 (3)	0.000 (3)
C2	0.036 (4)	0.055 (5)	0.034 (4)	0.007 (3)	0.004 (3)	0.002 (3)
C3	0.032 (3)	0.053 (5)	0.039 (4)	−0.006 (3)	0.011 (3)	0.001 (3)
C4	0.041 (4)	0.048 (5)	0.035 (3)	−0.001 (3)	0.017 (3)	0.000 (3)
C5	0.042 (4)	0.050 (5)	0.029 (3)	0.011 (3)	0.005 (3)	0.001 (3)
C6	0.041 (4)	0.043 (4)	0.033 (3)	0.000 (3)	0.010 (3)	−0.002 (3)
C7	0.037 (4)	0.038 (4)	0.037 (4)	−0.003 (3)	0.016 (3)	−0.007 (3)
C8	0.041 (4)	0.051 (5)	0.036 (4)	0.002 (3)	0.008 (3)	0.004 (3)
C9	0.036 (4)	0.061 (5)	0.041 (4)	−0.004 (4)	0.009 (3)	0.003 (3)
C10	0.044 (4)	0.076 (6)	0.039 (4)	0.000 (4)	0.006 (3)	0.015 (4)
C11	0.051 (4)	0.077 (6)	0.038 (4)	−0.009 (4)	0.013 (3)	0.013 (4)
C12	0.058 (5)	0.071 (6)	0.032 (4)	0.003 (4)	0.009 (3)	0.005 (4)
C13	0.056 (5)	0.071 (6)	0.055 (5)	0.006 (4)	0.004 (4)	0.026 (4)
C14	0.045 (4)	0.072 (6)	0.060 (5)	0.000 (4)	0.008 (3)	0.018 (4)
C15	0.085 (6)	0.087 (8)	0.066 (6)	0.005 (5)	0.008 (5)	0.028 (5)
C16	0.079 (6)	0.091 (8)	0.059 (5)	−0.006 (5)	0.022 (5)	0.016 (5)

Geometric parameters (Å, °)

O1—C5	1.372 (8)	C4—H4	0.9300
O1—H1	0.8200	C5—C6	1.393 (9)
O2—C3	1.357 (8)	C6—H6	0.9300
O2—H2	0.8200	C8—C9	1.450 (10)
O3—C7	1.248 (8)	C8—H8	0.9300
O4—H4B	0.85 (4)	C9—C10	1.377 (9)
O4—H4A	0.85 (4)	C9—C14	1.388 (10)
N1—C7	1.333 (8)	C10—C11	1.377 (9)
N1—N2	1.382 (8)	C10—H10	0.9300
N1—H1A	0.91 (5)	C11—C12	1.376 (10)

N2—C8	1.273 (9)	C11—H11	0.9300
N3—C12	1.381 (9)	C12—C13	1.389 (10)
N3—C15	1.443 (10)	C13—C14	1.384 (11)
N3—C16	1.455 (10)	C13—H13	0.9300
C1—C2	1.377 (8)	C14—H14	0.9300
C1—C6	1.377 (8)	C15—H15A	0.9600
C1—C7	1.495 (9)	C15—H15B	0.9600
C2—C3	1.412 (9)	C15—H15C	0.9600
C2—H2A	0.9300	C16—H16A	0.9600
C3—C4	1.353 (9)	C16—H16B	0.9600
C4—C5	1.366 (9)	C16—H16C	0.9600
C5—O1—H1	109.5	N2—C8—H8	119.3
C3—O2—H2	109.5	C9—C8—H8	119.3
H4B—O4—H4A	109 (3)	C10—C9—C14	117.7 (7)
C7—N1—N2	119.5 (5)	C10—C9—C8	124.1 (6)
C7—N1—H1A	118 (6)	C14—C9—C8	118.2 (6)
N2—N1—H1A	123 (6)	C9—C10—C11	121.0 (6)
C8—N2—N1	113.6 (6)	C9—C10—H10	119.5
C12—N3—C15	121.4 (7)	C11—C10—H10	119.5
C12—N3—C16	120.7 (7)	C12—C11—C10	121.2 (7)
C15—N3—C16	117.3 (7)	C12—C11—H11	119.4
C2—C1—C6	121.3 (6)	C10—C11—H11	119.4
C2—C1—C7	121.8 (6)	C11—C12—N3	120.6 (7)
C6—C1—C7	116.9 (5)	C11—C12—C13	118.6 (7)
C1—C2—C3	117.8 (6)	N3—C12—C13	120.7 (6)
C1—C2—H2A	121.1	C14—C13—C12	119.6 (7)
C3—C2—H2A	121.1	C14—C13—H13	120.2
C4—C3—O2	118.6 (6)	C12—C13—H13	120.2
C4—C3—C2	121.2 (6)	C13—C14—C9	121.7 (7)
O2—C3—C2	120.2 (6)	C13—C14—H14	119.2
C3—C4—C5	119.9 (6)	C9—C14—H14	119.2
C3—C4—H4	120.0	N3—C15—H15A	109.5
C5—C4—H4	120.0	N3—C15—H15B	109.5
C4—C5—O1	117.8 (6)	H15A—C15—H15B	109.5
C4—C5—C6	120.7 (6)	N3—C15—H15C	109.5
O1—C5—C6	121.5 (6)	H15A—C15—H15C	109.5
C1—C6—C5	118.9 (6)	H15B—C15—H15C	109.5
C1—C6—H6	120.5	N3—C16—H16A	109.5
C5—C6—H6	120.5	N3—C16—H16B	109.5
O3—C7—N1	121.4 (6)	H16A—C16—H16B	109.5
O3—C7—C1	120.5 (6)	N3—C16—H16C	109.5
N1—C7—C1	118.0 (5)	H16A—C16—H16C	109.5
N2—C8—C9	121.4 (6)	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···O3^{i}	0.850 (11)	2.16 (6)	2.895 (8)	144 (9)

N1—H1A···O4^{ii}	0.902 (11)	2.14 (3)	2.999 (7)	159 (8)
O4—H4B···N2	0.848 (11)	2.46 (4)	3.175 (7)	143 (6)
O4—H4B···O3	0.848 (11)	2.24 (5)	2.936 (8)	139 (7)
O2—H2···O3^{iii}	0.82	1.99	2.694 (6)	144
O1—H1···O2^{iv}	0.82	2.06	2.766 (7)	145

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

Fig. 1

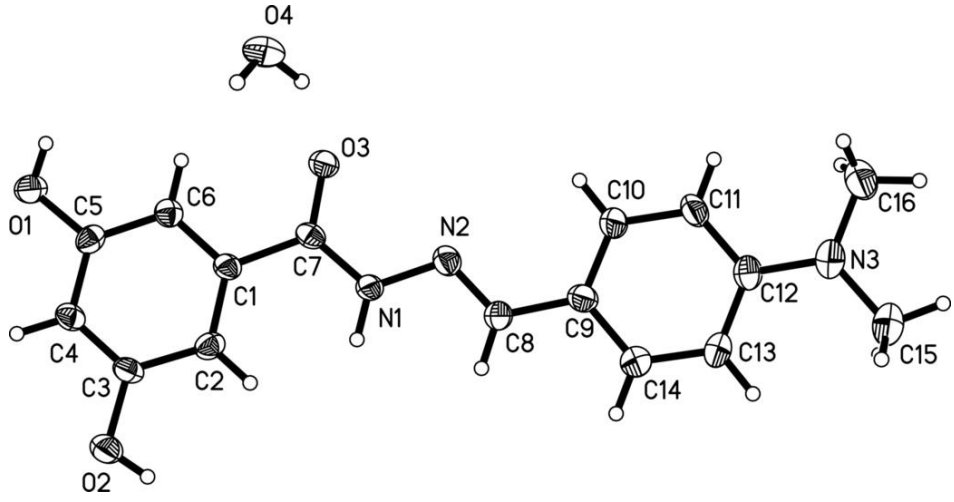
O4
O3
C10
C11
C16
O1
C5
C6
N2
N3
C7
C12
C1
N1
C9
C8
C4
C13
C15
C3
C2
C14
O2

Fig. 2