Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-[4-(Dimethylamino)benzylidene]-3-hydroxybenzohydrazide

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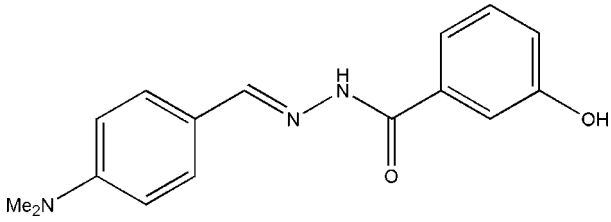
Received 4 January 2008; accepted 13 January 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 15.8.

The title compound, $C_{16}H_{17}N_3O_2$, was synthesized by the reaction of 4-dimethylaminobenzaldehyde with 3-hydroxybenzoic acid hydrazide in methanol. The dihedral angle between the two benzene rings in the molecule is 9.2 (2)°. 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 *bc* plane.

Related literature

For related literature, see: Akitsu & Einaga (2006); Bahner *et al.* (1968); Butcher *et al.* (2005); Hodnett & Mooney (1970); Merchant & Chothia (1970); Pradeep (2005); Sigman & Jacobsen (1998).



Experimental

Crystal data

$C_{16}H_{17}N_3O_2$
M_r = 283.33
Monoclinic, $P2_1/c$
a = 13.397 (3) Å
b = 9.663 (2) Å
c = 11.183 (2) Å
β = 101.97 (3)°
V = 1416.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.28 × 0.27 × 0.27 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.976
11531 measured reflections
3094 independent reflections
2579 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
S = 1.05
3094 reflections
196 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	2.18	2.8470 (14)	138
O2—H2···N2^{i}	0.82	2.36	3.1008 (16)	150
N3—H3*A*···O1^{ii}	0.895 (9)	2.561 (11)	3.4172 (16)	160.4 (16)

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

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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2040).

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supporting information

Acta Cryst. (2008). E**64**, o471 [doi:10.1107/S160053680800130X]

N′-[4-(Dimethylamino)benzylidene]-3-hydroxybenzohydrazide

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S1. Comment

Schiff base compounds have been widely investigated due to their easy synthesis, versatile structures and wide applications (Sigman & Jacobsen, 1998; Akitsu & Einaga, 2006; Pradeep, 2005; Butcher *et al.*, 2005). The excellent antibacterial and antitumor properties of such compounds have attracted much interest in recent years (Hodnett & Mooney, 1970; Bahner *et al.*, 1968; Merchant & Chothia, 1970). In order to investigate further the structures of such compounds, the new title Schiff base compound is reported on here.

The dihedral angle between the two benzene rings in the molecule (Fig. 1) of the title compound is 9.2 (2)°. In the crystal structure, molecules are linked through intermolecular O–H···O, O–H···N and N–H···O hydrogen bonds (Table 1), forming layers parallel to the *bc* plane (Fig. 2).

S2. Experimental

The title compound was obtained by stirring of 4-dimethylaminobenzaldehyde (0.1 mmol, 14.9 mg) and 3-hydroxy-benzoic acid hydrazide (0.1 mmol, 15.2 mg) in a methanol solution (10 ml) at room temperature. Yellow block-shaped single crystals suitable for X-ray diffraction were formed from the solution after seven days.

S3. Refinement

H3A was located from a difference Fourier map and refined with the N–H distance restrained to 0.90 (1) Å, and U_{iso}(H) = 0.08 Å^2. Other H atoms were positioned geometrically (C–H = 0.93–0.96Å and O–H = 0.82 Å) and treated as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O and methyl-C).

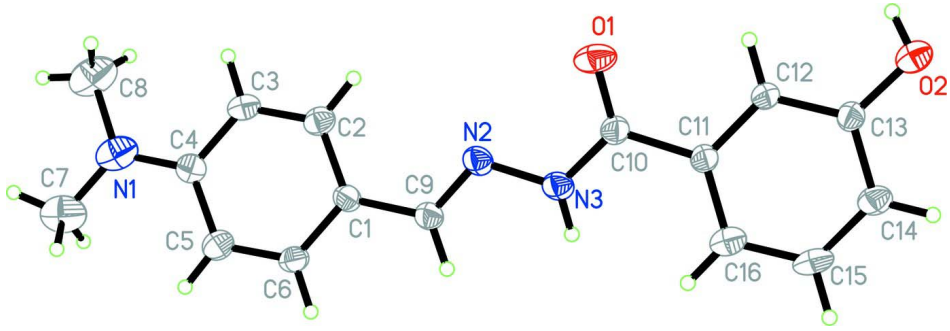

Figure 1

The molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids drawn at the 30% probability level.

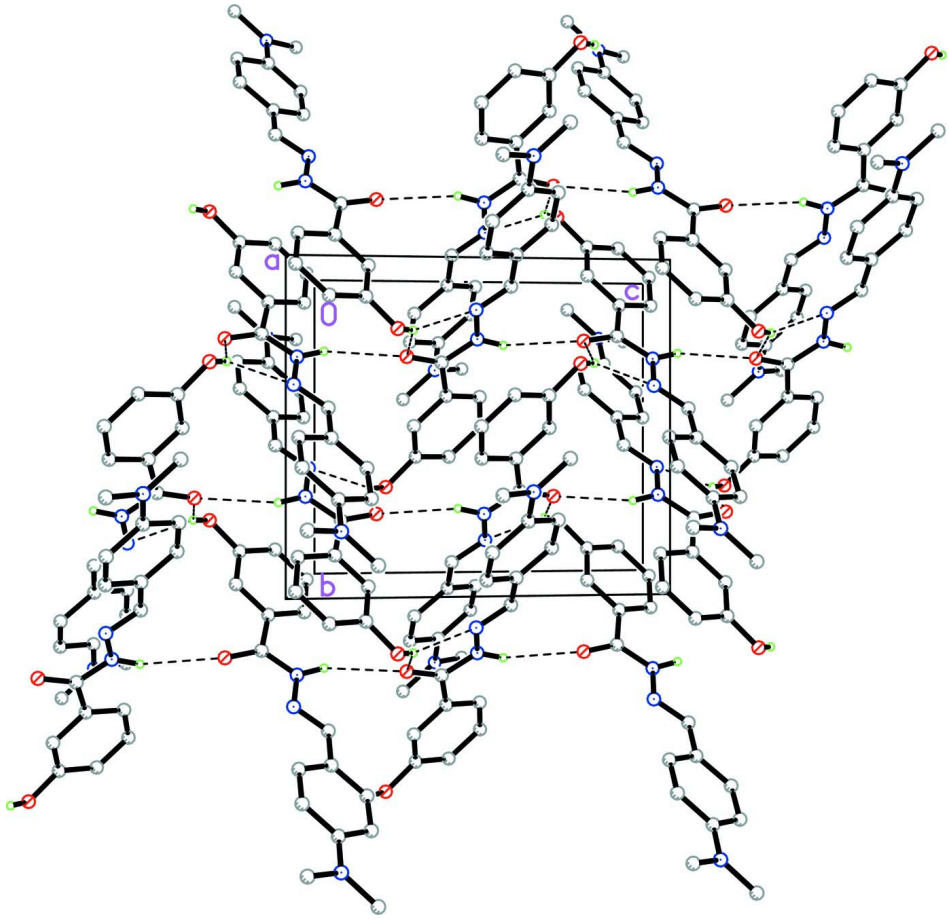

Figure 2

Crystal packing of the title compound view along the *a* axis [hydrogen bonds are drawn as dotted lines].

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Crystal data

$C_{16}H_{17}N_3O_2$
$M_r = 283.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.397\ (3)$ Å
$b = 9.663\ (2)$ Å
$c = 11.183\ (2)$ Å
$\beta = 101.97\ (3)°$
$V = 1416.2\ (5)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.329$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4593 reflections
$\theta = 2.5–27.7°$
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Block, yellow
0.28 × 0.27 × 0.27 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.975$, $T_{max} = 0.976$
11531 measured reflections
3094 independent reflections
2579 reflections with $I > 2\sigma(I)$

R_{int} = 0.021
θ_{max} = 27.0°, θ_{min} = 2.6°
h = −16→17
k = −12→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
S = 1.05
3094 reflections
196 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
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.1729P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.51231 (8)	0.27675 (11)	0.29293 (9)	0.0574 (3)
O2	0.28657 (7)	0.69755 (10)	0.22473 (9)	0.0486 (3)
H2	0.3315	0.7006	0.1849	0.073*
N1	0.97117 (10)	−0.30631 (15)	0.63913 (13)	0.0654 (4)
N2	0.60441 (8)	0.13189 (10)	0.48763 (10)	0.0416 (3)
N3	0.52359 (8)	0.22033 (11)	0.49046 (10)	0.0420 (3)
C1	0.72192 (9)	−0.03254 (12)	0.59700 (11)	0.0379 (3)
C2	0.78863 (10)	−0.03161 (13)	0.51647 (11)	0.0430 (3)
H2A	0.7774	0.0302	0.4513	0.052*
C3	0.87041 (10)	−0.11981 (14)	0.53134 (12)	0.0445 (3)
H3	0.9138	−0.1157	0.4764	0.053*
C4	0.89036 (9)	−0.21613 (13)	0.62715 (12)	0.0425 (3)
C5	0.82405 (10)	−0.21577 (14)	0.70911 (12)	0.0456 (3)
H5	0.8352	−0.2770	0.7747	0.055*
C6	0.74284 (10)	−0.12595 (14)	0.69358 (11)	0.0424 (3)
H6	0.7004	−0.1277	0.7496	0.051*
C7	0.99119 (16)	−0.4061 (2)	0.73523 (17)	0.0793 (6)
H7A	1.0129	−0.3597	0.8121	0.119*
H7B	1.0439	−0.4679	0.7219	0.119*
H7C	0.9302	−0.4578	0.7363	0.119*
C8	1.04154 (15)	−0.2992 (2)	0.5584 (2)	0.0903 (7)

H8A	1.0063	−0.3194	0.4764	0.135*
H8B	1.0952	−0.3655	0.5835	0.135*
H8C	1.0702	−0.2079	0.5612	0.135*
C9	0.63422 (9)	0.05897 (13)	0.58348 (11)	0.0411 (3)
H9	0.5988	0.0645	0.6466	0.049*
C10	0.48449 (9)	0.29458 (12)	0.38962 (12)	0.0397 (3)
C11	0.40510 (9)	0.39961 (12)	0.40150 (11)	0.0375 (3)
C12	0.38258 (9)	0.49733 (12)	0.30887 (11)	0.0375 (3)
H12	0.4168	0.4954	0.2446	0.045*
C13	0.30939 (9)	0.59770 (12)	0.31174 (11)	0.0380 (3)
C14	0.25681 (10)	0.59887 (14)	0.40629 (12)	0.0453 (3)
H14	0.2067	0.6651	0.4080	0.054*
C15	0.27927 (11)	0.50121 (16)	0.49776 (13)	0.0530 (4)
H15	0.2440	0.5021	0.5611	0.064*
C16	0.35350 (10)	0.40181 (14)	0.49689 (12)	0.0469 (3)
H16	0.3686	0.3372	0.5596	0.056*
H3A	0.5095 (14)	0.2388 (19)	0.5637 (11)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0646 (6)	0.0592 (6)	0.0572 (6)	0.0197 (5)	0.0327 (5)	0.0044 (5)
O2	0.0480 (5)	0.0487 (5)	0.0530 (6)	0.0081 (4)	0.0192 (4)	0.0094 (4)
N1	0.0552 (7)	0.0751 (9)	0.0698 (8)	0.0289 (7)	0.0219 (6)	0.0220 (7)
N2	0.0369 (5)	0.0388 (6)	0.0501 (6)	0.0050 (4)	0.0114 (4)	−0.0082 (4)
N3	0.0380 (5)	0.0417 (6)	0.0485 (6)	0.0072 (4)	0.0137 (5)	−0.0065 (5)
C1	0.0368 (6)	0.0382 (6)	0.0391 (6)	−0.0001 (5)	0.0087 (5)	−0.0057 (5)
C2	0.0452 (7)	0.0451 (7)	0.0400 (6)	0.0056 (5)	0.0115 (5)	0.0069 (5)
C3	0.0427 (7)	0.0535 (8)	0.0407 (7)	0.0056 (5)	0.0162 (5)	0.0024 (5)
C4	0.0388 (6)	0.0447 (7)	0.0432 (7)	0.0046 (5)	0.0067 (5)	0.0001 (5)
C5	0.0467 (7)	0.0504 (7)	0.0397 (7)	0.0023 (6)	0.0085 (5)	0.0086 (5)
C6	0.0428 (7)	0.0499 (7)	0.0373 (6)	−0.0026 (5)	0.0149 (5)	−0.0027 (5)
C7	0.0834 (12)	0.0834 (13)	0.0713 (11)	0.0421 (10)	0.0162 (9)	0.0196 (9)
C8	0.0656 (11)	0.1028 (15)	0.1139 (16)	0.0410 (11)	0.0449 (11)	0.0305 (13)
C9	0.0394 (6)	0.0403 (6)	0.0456 (7)	0.0004 (5)	0.0131 (5)	−0.0074 (5)
C10	0.0359 (6)	0.0369 (6)	0.0497 (7)	0.0000 (5)	0.0165 (5)	−0.0046 (5)
C11	0.0332 (6)	0.0371 (6)	0.0442 (7)	−0.0012 (5)	0.0122 (5)	−0.0055 (5)
C12	0.0352 (6)	0.0394 (6)	0.0412 (6)	−0.0024 (5)	0.0156 (5)	−0.0044 (5)
C13	0.0345 (6)	0.0378 (6)	0.0423 (6)	−0.0024 (5)	0.0095 (5)	−0.0018 (5)
C14	0.0393 (6)	0.0472 (7)	0.0530 (7)	0.0094 (5)	0.0182 (5)	−0.0002 (6)
C15	0.0532 (8)	0.0630 (9)	0.0511 (8)	0.0150 (6)	0.0302 (6)	0.0071 (6)
C16	0.0486 (7)	0.0502 (7)	0.0464 (7)	0.0102 (6)	0.0199 (6)	0.0079 (6)

Geometric parameters (Å, º)

O1—C10	1.2267 (15)	C6—H6	0.9300
O2—C13	1.3593 (15)	C7—H7A	0.9600
O2—H2	0.8200	C7—H7B	0.9600

N1—C4	1.3745 (17)	C7—H7C	0.9600
N1—C7	1.427 (2)	C8—H8A	0.9600
N1—C8	1.436 (2)	C8—H8B	0.9600
N2—C9	1.2757 (16)	C8—H8C	0.9600
N2—N3	1.3848 (14)	C9—H9	0.9300
N3—C10	1.3475 (17)	C10—C11	1.4958 (16)
N3—H3A	0.895 (9)	C11—C16	1.3864 (18)
C1—C6	1.3907 (17)	C11—C12	1.3878 (17)
C1—C2	1.3942 (17)	C12—C13	1.3842 (17)
C1—C9	1.4531 (17)	C12—H12	0.9300
C2—C3	1.3706 (17)	C13—C14	1.3868 (17)
C2—H2A	0.9300	C14—C15	1.3783 (19)
C3—C4	1.4025 (18)	C14—H14	0.9300
C3—H3	0.9300	C15—C16	1.3841 (18)
C4—C5	1.4025 (19)	C15—H15	0.9300
C5—C6	1.3744 (18)	C16—H16	0.9300
C5—H5	0.9300		

C13—O2—H2	109.5	H7B—C7—H7C	109.5
C4—N1—C7	121.53 (13)	N1—C8—H8A	109.5
C4—N1—C8	120.99 (13)	N1—C8—H8B	109.5
C7—N1—C8	117.43 (13)	H8A—C8—H8B	109.5
C9—N2—N3	115.57 (10)	N1—C8—H8C	109.5
C10—N3—N2	118.66 (10)	H8A—C8—H8C	109.5
C10—N3—H3A	122.7 (12)	H8B—C8—H8C	109.5
N2—N3—H3A	117.2 (12)	N2—C9—C1	121.94 (11)
C6—C1—C2	116.93 (11)	N2—C9—H9	119.0
C6—C1—C9	120.27 (11)	C1—C9—H9	119.0
C2—C1—C9	122.80 (11)	O1—C10—N3	121.76 (11)
C3—C2—C1	121.39 (12)	O1—C10—C11	121.68 (12)
C3—C2—H2A	119.3	N3—C10—C11	116.56 (10)
C1—C2—H2A	119.3	C16—C11—C12	119.87 (11)
C2—C3—C4	121.75 (11)	C16—C11—C10	123.77 (11)
C2—C3—H3	119.1	C12—C11—C10	116.35 (10)
C4—C3—H3	119.1	C13—C12—C11	120.32 (10)
N1—C4—C5	122.05 (12)	C13—C12—H12	119.8
N1—C4—C3	121.11 (12)	C11—C12—H12	119.8
C5—C4—C3	116.84 (11)	O2—C13—C12	122.41 (10)
C6—C5—C4	120.73 (12)	O2—C13—C14	117.75 (11)
C6—C5—H5	119.6	C12—C13—C14	119.84 (11)
C4—C5—H5	119.6	C15—C14—C13	119.55 (11)
C5—C6—C1	122.33 (11)	C15—C14—H14	120.2
C5—C6—H6	118.8	C13—C14—H14	120.2
C1—C6—H6	118.8	C14—C15—C16	121.09 (12)
N1—C7—H7A	109.5	C14—C15—H15	119.5
N1—C7—H7B	109.5	C16—C15—H15	119.5
H7A—C7—H7B	109.5	C15—C16—C11	119.32 (12)
N1—C7—H7C	109.5	C15—C16—H16	120.3

H7A—C7—H7C	109.5	C11—C16—H16	120.3

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.82	2.18	2.8470 (14)	138
O2—H2···N2^{i}	0.82	2.36	3.1008 (16)	150
N3—H3*A*···O1^{ii}	0.90 (1)	2.56 (1)	3.4172 (16)	160 (2)

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