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2,4-Dihydroxy-*N*′-(3,4,5-trimethoxybenzylidene)benzohydrazide

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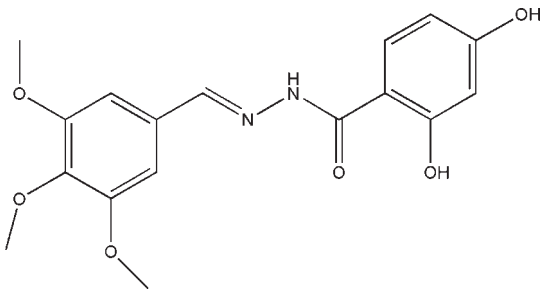
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.148; data-to-parameter ratio = 15.0.

In the title compound, $C_{17}H_{18}N_2O_6$, the molecule is slightly twisted, with a dihedral angle of 18.1 (2)° between the two benzene rings. In the crystal structure, molecules are linked into a network by intermolecular N—H···O, O—H···N and O—H···O hydrogen bonds. An intramolecular O—H···O hydrogen bond is also present.

Related literature

For the biological properties of Schiff base compounds, see: Brückner *et al.* (2000); Harrop *et al.* (2003); Ren *et al.* (2002). For the crystal structures of some Schiff bases and their complexes, see: Diao (2007); Diao *et al.* (2007, 2008); Huang *et al.* (2007); Li *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{18}N_2O_6$
M_r = 346.33
Orthorhombic, *Pbca*
a = 14.601 (1) Å
b = 11.030 (2) Å
c = 20.006 (2) Å
V = 3222.0 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.978, T_{max} = 0.978
18590 measured reflections
3520 independent reflections
2166 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.148
S = 1.03
3520 reflections
234 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O5^{i}	0.893 (10)	2.109 (13)	2.953 (2)	157 (2)
O3—H3···N2^{ii}	0.82	2.52	3.214 (3)	143
O3—H3···O1^{ii}	0.82	1.95	2.674 (2)	147
O2—H2···O1	0.82	1.79	2.518 (2)	147

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: WN2345).

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supplementary materials

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2,4-Dihydroxy-*N*'-(3,4,5-trimethoxybenzylidene)benzohydrazide

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Comment

Schiff base compounds have been found to have potential pharmacological and antitumor properties (Brückner *et al.*, 2000; Harrop *et al.*, 2003; Ren *et al.*, 2002). Recently, the crystal structures of a few Schiff base compounds derived from the reaction of aldehydes with benzohydrazides have been reported (Diao *et al.*, 2008; Diao *et al.*, 2007; Diao, 2007; Li *et al.*, 2007; Huang *et al.*, 2007). As a continuation of these studies, we report here the crystal structure of the title compound.

The title compound, $C_{17}H_{18}N_2O_6$ (Fig. 1) is slightly twisted, with a dihedral angle between the two benzene rings of 18.1 (2)°. The torsion angles C9—C8—N2—N1 and C2—C7—N1—N2 are 2.9 (2) and 7.8 (2)°, respectively. In the crystal structure, molecules are linked into a network (Fig. 2) by intermolecular N—H···O, O—H···N and O—H···O hydrogen bonds (Table 1). An intramolecular O—H···O hydrogen bond is also present.

Experimental

3,4,5-Trimethoxybenzaldehyde (0.1 mmol, 19.6 mg) and 2,4-dihydroxybenzohydrazide (0.1 mmol, 16.8 mg) were dissolved in a methanol solution (20 ml). The mixture was stirred at reflux for 1 h and cooled to room temperature. After allowing the solution to stand in air for a few days, colorless block-like crystals were formed.

Refinement

H1A was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances of 0.93 and 0.96 Å, an O—H distance of 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(aromatic C) and 1.5U_{eq}(O and methyl C).

Figures

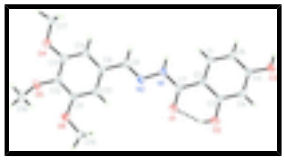

Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Hydrogen atoms are represented by spheres of arbitrary radius. The dashed line indicates the intramolecular O—H···O hydrogen bond.

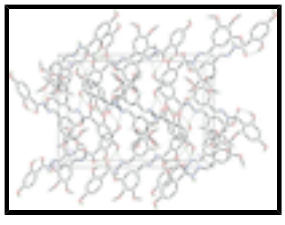

Fig. 2. The crystal structure of the title compound, viewed along the *b* axis. Dashed lines indicate hydrogen bonds. Hydrogen atoms not involved in hydrogen bonding have been omitted.

2,4-Dihydroxy-*N*'-(3,4,5-trimethoxybenzylidene)benzohydrazide

Crystal data

$C_{17}H_{18}N_2O_6$	F_{000} = 1456
M_r = 346.33	D_x = 1.428 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2734 reflections
a = 14.601 (1) Å	θ = 2.5–26.0º
b = 11.030 (2) Å	μ = 0.11 mm^{-1}
c = 20.006 (2) Å	*T* = 298 K
V = 3222.0 (7) Å^3	Block, colorless
Z = 8	0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3520 independent reflections
Radiation source: fine-focus sealed tube	2166 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.069
T = 298 K	θ_{max} = 27.0º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	*h* = −18→18
T_{min} = 0.978, T_{max} = 0.978	*k* = −12→14
18590 measured reflections	*l* = −20→25

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.148	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.9796P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
3520 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
234 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	−0.07006 (12)	0.46657 (17)	0.36310 (9)	0.0409 (5)
N2	0.00500 (12)	0.44220 (16)	0.40282 (9)	0.0410 (4)
O1	0.01502 (10)	0.60011 (16)	0.30628 (8)	0.0528 (5)
O2	−0.04899 (12)	0.72863 (18)	0.21335 (9)	0.0650 (5)
H2	−0.0112	0.7057	0.2409	0.097*
O3	−0.33269 (11)	0.63390 (17)	0.12189 (8)	0.0555 (5)
H3	−0.3814	0.6018	0.1322	0.083*
O4	0.12167 (11)	0.13843 (16)	0.63808 (9)	0.0584 (5)
O5	0.28830 (10)	0.21803 (14)	0.60976 (8)	0.0474 (4)
O6	0.31779 (11)	0.37901 (16)	0.51322 (9)	0.0579 (5)
C1	−0.21013 (15)	0.4894 (2)	0.25893 (12)	0.0420 (5)
H1	−0.2169	0.4251	0.2886	0.050*
C2	−0.13151 (14)	0.56173 (19)	0.26278 (11)	0.0384 (5)
C3	−0.12280 (15)	0.6558 (2)	0.21550 (11)	0.0431 (5)
C4	−0.19085 (16)	0.6778 (2)	0.16936 (12)	0.0465 (6)
H4	−0.1843	0.7408	0.1388	0.056*
C5	−0.26856 (15)	0.6071 (2)	0.16830 (11)	0.0418 (5)
C6	−0.27758 (15)	0.5110 (2)	0.21243 (11)	0.0421 (5)
H6	−0.3290	0.4613	0.2106	0.050*
C7	−0.05838 (14)	0.54391 (19)	0.31173 (11)	0.0396 (5)
C8	−0.00724 (15)	0.3683 (2)	0.45107 (11)	0.0423 (5)
H8	−0.0652	0.3366	0.4593	0.051*
C9	0.07007 (15)	0.33347 (19)	0.49349 (11)	0.0402 (5)
C10	0.15711 (15)	0.3793 (2)	0.48163 (11)	0.0435 (5)
H10	0.1665	0.4351	0.4475	0.052*
C11	0.22944 (15)	0.3418 (2)	0.52056 (12)	0.0437 (6)
C12	0.21532 (15)	0.2592 (2)	0.57258 (11)	0.0411 (5)
C13	0.12835 (16)	0.2157 (2)	0.58524 (11)	0.0428 (5)
C14	0.05539 (15)	0.2521 (2)	0.54515 (11)	0.0428 (5)
H14	−0.0031	0.2219	0.5530	0.051*
C15	0.33483 (18)	0.4696 (3)	0.46434 (14)	0.0652 (8)
H15A	0.3242	0.4367	0.4206	0.098*
H15B	0.3973	0.4962	0.4677	0.098*

H15C	0.2946	0.5371	0.4717	0.098*
C16	0.3270 (2)	0.3053 (2)	0.65403 (14)	0.0655 (8)
H16A	0.3195	0.3850	0.6355	0.098*
H16B	0.3910	0.2887	0.6599	0.098*
H16C	0.2966	0.3010	0.6965	0.098*
C17	0.03536 (19)	0.0858 (3)	0.65186 (14)	0.0646 (8)
H17A	−0.0098	0.1486	0.6560	0.097*
H17B	0.0387	0.0408	0.6929	0.097*
H17C	0.0186	0.0323	0.6160	0.097*
H1A	−0.1231 (10)	0.4289 (19)	0.3709 (11)	0.050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0320 (10)	0.0498 (11)	0.0407 (11)	−0.0008 (8)	−0.0063 (8)	0.0046 (8)
N2	0.0349 (10)	0.0496 (10)	0.0385 (11)	0.0046 (8)	−0.0062 (8)	−0.0037 (9)
O1	0.0353 (9)	0.0687 (11)	0.0544 (11)	−0.0083 (8)	−0.0044 (8)	0.0092 (8)
O2	0.0514 (11)	0.0832 (13)	0.0603 (12)	−0.0237 (10)	−0.0062 (8)	0.0223 (10)
O3	0.0411 (9)	0.0797 (12)	0.0457 (10)	−0.0056 (9)	−0.0069 (8)	0.0135 (9)
O4	0.0475 (10)	0.0716 (11)	0.0561 (11)	−0.0065 (8)	−0.0100 (8)	0.0231 (9)
O5	0.0397 (9)	0.0548 (9)	0.0477 (10)	0.0062 (7)	−0.0115 (7)	−0.0020 (8)
O6	0.0359 (9)	0.0830 (12)	0.0548 (11)	−0.0034 (8)	−0.0009 (8)	0.0142 (9)
C1	0.0383 (12)	0.0432 (12)	0.0445 (13)	−0.0004 (9)	0.0003 (10)	0.0051 (10)
C2	0.0321 (11)	0.0461 (12)	0.0371 (12)	0.0001 (9)	0.0016 (9)	0.0015 (10)
C3	0.0340 (12)	0.0543 (13)	0.0411 (13)	−0.0075 (10)	0.0014 (10)	0.0021 (11)
C4	0.0445 (14)	0.0559 (14)	0.0391 (13)	−0.0046 (11)	0.0008 (10)	0.0104 (11)
C5	0.0347 (12)	0.0586 (14)	0.0320 (12)	0.0022 (10)	−0.0010 (9)	−0.0001 (10)
C6	0.0349 (12)	0.0469 (12)	0.0444 (13)	−0.0044 (10)	−0.0017 (10)	0.0005 (11)
C7	0.0305 (11)	0.0465 (12)	0.0417 (13)	−0.0004 (9)	0.0013 (9)	−0.0037 (10)
C8	0.0350 (12)	0.0497 (13)	0.0423 (13)	−0.0002 (10)	−0.0053 (10)	−0.0020 (11)
C9	0.0368 (12)	0.0463 (12)	0.0374 (13)	0.0027 (9)	−0.0051 (10)	−0.0036 (10)
C10	0.0419 (13)	0.0529 (13)	0.0356 (12)	0.0032 (10)	−0.0025 (10)	0.0037 (10)
C11	0.0326 (12)	0.0558 (14)	0.0427 (13)	0.0010 (10)	0.0004 (10)	−0.0020 (11)
C12	0.0354 (12)	0.0479 (12)	0.0399 (13)	0.0062 (10)	−0.0062 (10)	−0.0028 (10)
C13	0.0437 (13)	0.0464 (12)	0.0382 (13)	0.0023 (10)	−0.0032 (10)	0.0016 (10)
C14	0.0338 (12)	0.0519 (13)	0.0428 (13)	−0.0014 (10)	−0.0033 (10)	−0.0012 (11)
C15	0.0477 (16)	0.0815 (19)	0.0664 (18)	−0.0079 (13)	0.0057 (14)	0.0125 (15)
C16	0.0667 (18)	0.0671 (17)	0.0629 (18)	0.0099 (14)	−0.0232 (15)	−0.0177 (14)
C17	0.0556 (16)	0.0703 (17)	0.0678 (18)	−0.0114 (14)	−0.0058 (14)	0.0237 (14)

Geometric parameters (Å, °)

N1—C7	1.346 (3)	C4—H4	0.9300
N1—N2	1.380 (2)	C5—C6	1.386 (3)
N1—H1A	0.893 (10)	C6—H6	0.9300
N2—C8	1.276 (3)	C8—C9	1.464 (3)
O1—C7	1.243 (2)	C8—H8	0.9300
O2—C3	1.345 (3)	C9—C14	1.385 (3)
O2—H2	0.8200	C9—C10	1.388 (3)

O3—C5	1.351 (3)	C10—C11	1.376 (3)
O3—H3	0.8200	C10—H10	0.9300
O4—C13	1.362 (3)	C11—C12	1.399 (3)
O4—C17	1.415 (3)	C12—C13	1.381 (3)
O5—C12	1.376 (2)	C13—C14	1.393 (3)
O5—C16	1.425 (3)	C14—H14	0.9300
O6—C11	1.362 (3)	C15—H15A	0.9600
O6—C15	1.420 (3)	C15—H15B	0.9600
C1—C6	1.375 (3)	C15—H15C	0.9600
C1—C2	1.400 (3)	C16—H16A	0.9600
C1—H1	0.9300	C16—H16B	0.9600
C2—C3	1.410 (3)	C16—H16C	0.9600
C2—C7	1.462 (3)	C17—H17A	0.9600
C3—C4	1.378 (3)	C17—H17B	0.9600
C4—C5	1.377 (3)	C17—H17C	0.9600
C7—N1—N2	117.54 (17)	C10—C9—C8	120.8 (2)
C7—N1—H1A	122.6 (15)	C11—C10—C9	119.8 (2)
N2—N1—H1A	119.8 (15)	C11—C10—H10	120.1
C8—N2—N1	116.67 (18)	C9—C10—H10	120.1
C3—O2—H2	109.5	O6—C11—C10	125.2 (2)
C5—O3—H3	109.5	O6—C11—C12	114.60 (19)
C13—O4—C17	118.17 (18)	C10—C11—C12	120.2 (2)
C12—O5—C16	114.84 (18)	O5—C12—C13	119.9 (2)
C11—O6—C15	116.89 (19)	O5—C12—C11	120.2 (2)
C6—C1—C2	121.7 (2)	C13—C12—C11	119.9 (2)
C6—C1—H1	119.1	O4—C13—C12	115.19 (19)
C2—C1—H1	119.1	O4—C13—C14	124.9 (2)
C1—C2—C3	117.1 (2)	C12—C13—C14	119.9 (2)
C1—C2—C7	124.0 (2)	C9—C14—C13	119.9 (2)
C3—C2—C7	118.85 (19)	C9—C14—H14	120.1
O2—C3—C4	116.8 (2)	C13—C14—H14	120.1
O2—C3—C2	122.2 (2)	O6—C15—H15A	109.5
C4—C3—C2	121.0 (2)	O6—C15—H15B	109.5
C5—C4—C3	120.3 (2)	H15A—C15—H15B	109.5
C5—C4—H4	119.8	O6—C15—H15C	109.5
C3—C4—H4	119.8	H15A—C15—H15C	109.5
O3—C5—C4	117.2 (2)	H15B—C15—H15C	109.5
O3—C5—C6	122.6 (2)	O5—C16—H16A	109.5
C4—C5—C6	120.1 (2)	O5—C16—H16B	109.5
C1—C6—C5	119.7 (2)	H16A—C16—H16B	109.5
C1—C6—H6	120.2	O5—C16—H16C	109.5
C5—C6—H6	120.2	H16A—C16—H16C	109.5
O1—C7—N1	119.5 (2)	H16B—C16—H16C	109.5
O1—C7—C2	120.3 (2)	O4—C17—H17A	109.5
N1—C7—C2	120.25 (19)	O4—C17—H17B	109.5
N2—C8—C9	119.9 (2)	H17A—C17—H17B	109.5
N2—C8—H8	120.1	O4—C17—H17C	109.5
C9—C8—H8	120.1	H17A—C17—H17C	109.5
C14—C9—C10	120.3 (2)	H17B—C17—H17C	109.5

C14—C9—C8	118.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O5^{i}	0.893 (10)	2.109 (13)	2.953 (2)	157 (2)
O3—H3···N2^{ii}	0.82	2.52	3.214 (3)	143
O3—H3···O1^{ii}	0.82	1.95	2.674 (2)	147
O2—H2···O1	0.82	1.79	2.518 (2)	147

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

Fig. 1

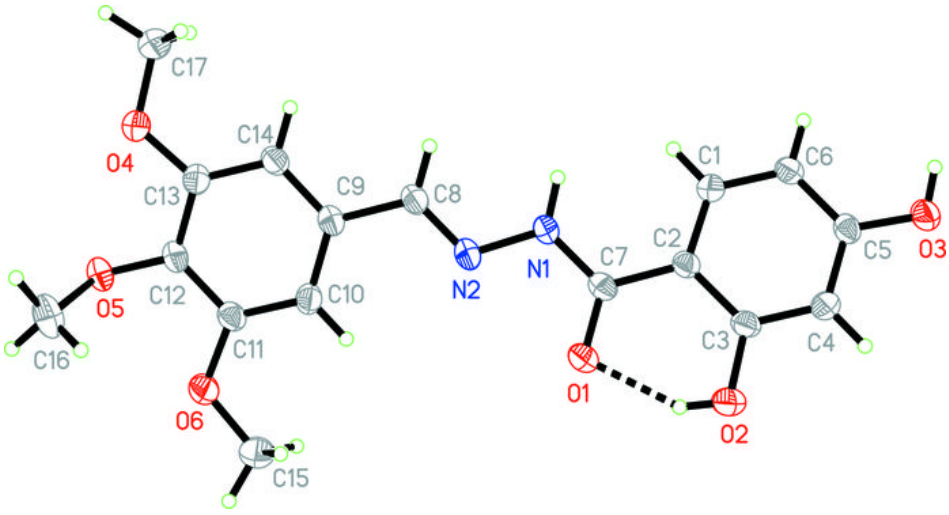
C17
O4
C14
C13
C9
C8
C1
C6
O3
C5
C2
N2
N1
C7
O5
C12
C10
C16
C11
C3
C4
O1
O2
O6
C15

Fig. 2