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(*E*)-*N*′-(2,5-Dimethoxybenzylidene)-2,4-dihydroxybenzohydrazide

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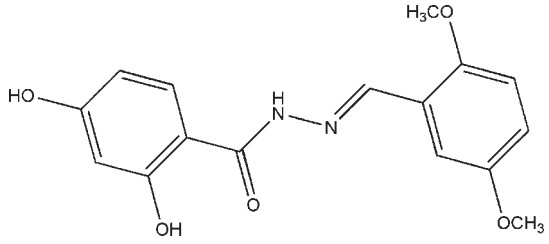
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.112; data-to-parameter ratio = 12.4.

In the title compound, $C_{16}H_{16}N_2O_5$, the dihedral angle between the two benzene rings is 4.2 (2)° and an intramolecular O—H···O hydrogen bond generates an S(6) ring. In the crystal, molecules are linked into layers lying parallel to the bc plane by O—H···O and N—H···O hydrogen bonds.

Related literature

For the biological properties of Schiff base compounds, see: Bhandari *et al.* (2008); Sinha *et al.* (2008). For Schiff base compounds containing 2,5-dimethoxybenzaldehyde, see: Wang *et al.* (2009). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{16}N_2O_5$ M_r = 316.31
Monoclinic, $P2_1/c$
a = 7.8600 (16) Å
b = 15.358 (3) Å
c = 12.425 (3) Å
β = 99.80 (3)°
V = 1478.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 295 K
0.18 × 0.17 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.981, T_{max} = 0.984
7757 measured reflections
2626 independent reflections
1698 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.112
S = 1.02
2626 reflections
212 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3	0.82	1.76	2.495 (2)	148
O2—H2···O3^{i}	0.82	1.92	2.664 (2)	151
N1—H1A···O1^{ii}	0.86	2.17	3.012 (2)	166

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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5375).

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supplementary materials

Acta Cryst. (2010). E**66**, o996 [**doi:10.1107/S160053681001130X**]

(*E*)-*N*'-(2,5-Dimethoxybenzylidene)-2,4-dihydroxybenzohydrazide

J.-Y. Wei, D.-G. Song, D.-C. Wang, X.-M. Deng, J.-X. Liu and B. Liu

Comment

Schiff base compounds have been of great interest for many years. Some of the complexes derived from Schiff bases have been found to have pharmacological and antitumor properties (Bhandari *et al.*, 2008; Sinha *et al.*, 2008). In this paper, the crystal structure of the title compound, (I), a new Schiff base compound derived from the condensation reaction of 2,4-dihydroxybenzohydrazide with 2,5-dimethoxybenzaldehyde is reported.

The Schiff base molecule of the compound displays a *trans* configuration with respect to the C=N and C—N bonds(Fig. 1). All the bond lengths are within their normal ranges (Allen *et al.*, 1987) and comparable to other Schiff base compounds containing 2,5-dimethoxybenzaldehyde (Wang *et al.*, 2009). The dihedral angle between the two benzene rings is 4.2 (2)°. Intramolecular O—H···O hydrogen bonds are observed(Table 1). Molecules are linked into layers parallel to the *bc* plane by O—H···O and N—H···O hydrogen bonds(Fig. 2).

Experimental

2,5-dimethoxybenzaldehyde (0.1 mmol, 16.6 mg) and 2,4-dihydroxybenzohydrazide (0.1 mmol, 16.8 mg) were dissolved in a 95% ethanol solution (10 ml). The mixture was stirred at room temperature to give a clear colorless solution. Light yellow blocks of (I) were formed by gradual evaporation of the solvent over a period of three days at room temperature.

Refinement

All H atoms were placed in geometrically idealized positions, with C—H = 0.93–0.96 Å, O—H = 0.82–0.85 Å and N—H = 0.86 Å. U_{iso}(H) = 1.2U_{eq}(C,*N*), and 1.5U_{eq}(O).

Figures

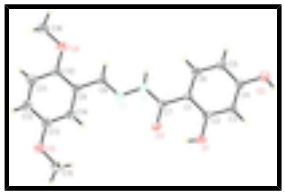

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. The dashed lines indicate hydrogen bonds.

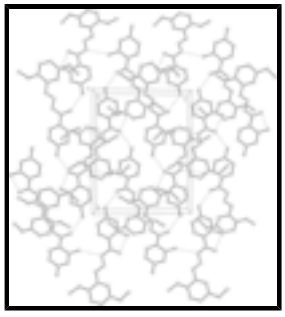

Fig. 2. The molecular packing of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen bonds have been omitted for clarity.

(*E*)-*N*'-(2,5-Dimethoxybenzylidene)-2,4-dihydroxybenzohydrazide

Crystal data

$C_{16}H_{16}N_2O_5$	$F(000) = 664$
$M_r = 316.31$	$D_x = 1.421$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1224 reflections
$a = 7.8600$ (16) Å	$\theta = 2.7$–$22.4°$
$b = 15.358$ (3) Å	$\mu = 0.11$ mm^{-1}
$c = 12.425$ (3) Å	$T = 295$ K
$\beta = 99.80$ (3)°	Block, light yellow
$V = 1478.0$ (5) Å^3	0.18 × 0.17 × 0.15 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2626 independent reflections
Radiation source: fine-focus sealed tube	1698 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.039$
φ and ω scans	$\theta_{max} = 25.1°$, $\theta_{min} = 2.1°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 8$
$T_{min} = 0.981$, $T_{max} = 0.984$	$k = -17 \rightarrow 18$
7757 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.112$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.2321P]$ where $P = (F_o^2 + 2F_c^2)/3$
2626 reflections	$(\Delta/\sigma)_{max} < 0.001$

212 parameters	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
O1	0.5668 (2)	0.18995 (10)	0.34476 (11)	0.0523 (5)
H1	0.6007	0.2404	0.3438	0.078*
O2	0.2931 (2)	−0.02763 (10)	0.10807 (13)	0.0623 (5)
H2	0.3162	−0.0573	0.1635	0.093*
O3	0.6343 (2)	0.33256 (9)	0.26549 (11)	0.0477 (4)
O4	0.7730 (2)	0.58885 (10)	−0.13674 (12)	0.0569 (5)
O5	1.0792 (2)	0.67891 (10)	0.28127 (12)	0.0529 (4)
N1	0.6247 (2)	0.35121 (10)	0.08522 (13)	0.0390 (5)
H1A	0.5957	0.3331	0.0192	0.047*
N2	0.7069 (2)	0.42982 (11)	0.10809 (13)	0.0390 (5)
C1	0.5091 (3)	0.21858 (13)	0.15178 (15)	0.0329 (5)
C2	0.5058 (3)	0.16342 (13)	0.24143 (16)	0.0354 (5)
C3	0.4396 (3)	0.08035 (14)	0.22852 (16)	0.0391 (5)
H3	0.4436	0.0439	0.2887	0.047*
C4	0.3674 (3)	0.05154 (14)	0.12604 (17)	0.0421 (6)
C5	0.3657 (3)	0.10522 (15)	0.03614 (17)	0.0527 (7)
H5	0.3168	0.0857	−0.0331	0.063*
C6	0.4356 (3)	0.18663 (14)	0.04918 (16)	0.0451 (6)
H6	0.4342	0.2218	−0.0119	0.054*
C7	0.5915 (3)	0.30376 (13)	0.17057 (16)	0.0368 (5)
C8	0.7456 (3)	0.47279 (13)	0.02779 (17)	0.0386 (5)
H8	0.7160	0.4520	−0.0432	0.046*
C9	0.8368 (3)	0.55503 (13)	0.04948 (16)	0.0365 (5)
C10	0.8507 (3)	0.61312 (14)	−0.03424 (16)	0.0405 (5)
C11	0.9361 (3)	0.69144 (14)	−0.01112 (19)	0.0463 (6)
H11	0.9448	0.7304	−0.0672	0.056*
C12	1.0081 (3)	0.71168 (15)	0.09455 (18)	0.0468 (6)
H12	1.0627	0.7651	0.1097	0.056*
C13	1.0003 (3)	0.65342 (14)	0.17882 (17)	0.0405 (5)
C14	0.9151 (3)	0.57596 (14)	0.15574 (17)	0.0380 (5)

H14	0.9094	0.5366	0.2119	0.046*
C15	1.1064 (3)	0.61377 (16)	0.36363 (18)	0.0576 (7)
H15A	0.9970	0.5938	0.3785	0.086*
H15B	1.1726	0.6377	0.4290	0.086*
H15C	1.1680	0.5658	0.3390	0.086*
C16	0.8212 (4)	0.63418 (17)	−0.22690 (18)	0.0649 (8)
H16A	0.7839	0.6936	−0.2260	0.097*
H16B	0.7679	0.6070	−0.2938	0.097*
H16C	0.9445	0.6324	−0.2216	0.097*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0799 (12)	0.0477 (11)	0.0280 (8)	−0.0122 (9)	0.0054 (8)	0.0022 (7)
O2	0.0909 (13)	0.0381 (10)	0.0523 (10)	−0.0194 (9)	−0.0042 (10)	0.0057 (8)
O3	0.0727 (11)	0.0384 (9)	0.0312 (8)	−0.0072 (8)	0.0064 (7)	−0.0044 (7)
O4	0.0792 (12)	0.0540 (11)	0.0359 (9)	−0.0129 (9)	0.0045 (8)	0.0054 (7)
O5	0.0636 (11)	0.0464 (10)	0.0445 (9)	−0.0064 (8)	−0.0026 (8)	−0.0036 (8)
N1	0.0569 (12)	0.0289 (10)	0.0321 (10)	−0.0078 (9)	0.0098 (8)	−0.0038 (8)
N2	0.0502 (11)	0.0294 (10)	0.0384 (10)	−0.0049 (8)	0.0101 (9)	−0.0028 (8)
C1	0.0414 (12)	0.0287 (12)	0.0293 (11)	0.0003 (9)	0.0075 (9)	0.0018 (9)
C2	0.0394 (12)	0.0395 (13)	0.0283 (11)	0.0013 (10)	0.0082 (9)	0.0015 (10)
C3	0.0469 (13)	0.0365 (13)	0.0343 (12)	−0.0012 (10)	0.0078 (10)	0.0097 (10)
C4	0.0487 (14)	0.0327 (13)	0.0440 (13)	−0.0046 (10)	0.0058 (11)	0.0039 (10)
C5	0.0797 (18)	0.0412 (14)	0.0332 (13)	−0.0130 (13)	−0.0014 (12)	0.0002 (11)
C6	0.0657 (16)	0.0386 (13)	0.0300 (12)	−0.0073 (11)	0.0052 (11)	0.0064 (10)
C7	0.0451 (13)	0.0342 (12)	0.0315 (12)	0.0049 (10)	0.0080 (10)	0.0016 (10)
C8	0.0487 (14)	0.0341 (13)	0.0333 (12)	−0.0013 (10)	0.0076 (10)	−0.0035 (10)
C9	0.0415 (13)	0.0312 (12)	0.0378 (12)	0.0006 (10)	0.0095 (10)	0.0001 (10)
C10	0.0467 (14)	0.0383 (13)	0.0365 (12)	0.0003 (10)	0.0071 (11)	0.0011 (10)
C11	0.0558 (15)	0.0357 (13)	0.0478 (14)	−0.0033 (11)	0.0099 (12)	0.0075 (10)
C12	0.0521 (15)	0.0331 (13)	0.0550 (15)	−0.0036 (10)	0.0090 (12)	0.0000 (11)
C13	0.0420 (13)	0.0370 (13)	0.0421 (13)	−0.0014 (10)	0.0061 (10)	−0.0039 (10)
C14	0.0429 (13)	0.0329 (12)	0.0393 (12)	0.0012 (10)	0.0098 (10)	0.0036 (9)
C15	0.0654 (17)	0.0599 (17)	0.0439 (14)	−0.0057 (13)	−0.0006 (13)	0.0041 (12)
C16	0.089 (2)	0.0674 (19)	0.0395 (14)	0.0021 (16)	0.0143 (14)	0.0098 (13)

Geometric parameters (Å, °)

O1—C2	1.355 (2)	C5—C6	1.364 (3)
O1—H1	0.8200	C5—H5	0.9300
O2—C4	1.351 (2)	C6—H6	0.9300
O2—H2	0.8200	C8—C9	1.455 (3)
O3—C7	1.251 (2)	C8—H8	0.9300
O4—C10	1.367 (2)	C9—C10	1.389 (3)
O4—C16	1.424 (3)	C9—C14	1.396 (3)
O5—C13	1.375 (2)	C10—C11	1.384 (3)
O5—C15	1.421 (3)	C11—C12	1.374 (3)
N1—C7	1.348 (2)	C11—H11	0.9300

N1—N2	1.376 (2)	C12—C13	1.387 (3)
N1—H1A	0.8600	C12—H12	0.9300
N2—C8	1.275 (2)	C13—C14	1.371 (3)
C1—C6	1.396 (3)	C14—H14	0.9300
C1—C2	1.403 (3)	C15—H15A	0.9600
C1—C7	1.461 (3)	C15—H15B	0.9600
C2—C3	1.377 (3)	C15—H15C	0.9600
C3—C4	1.376 (3)	C16—H16A	0.9600
C3—H3	0.9300	C16—H16B	0.9600
C4—C5	1.387 (3)	C16—H16C	0.9600
C2—O1—H1	109.5	C9—C8—H8	120.7
C4—O2—H2	109.5	C10—C9—C14	118.8 (2)
C10—O4—C16	117.56 (18)	C10—C9—C8	121.21 (19)
C13—O5—C15	117.08 (17)	C14—C9—C8	119.95 (19)
C7—N1—N2	117.31 (17)	O4—C10—C11	123.68 (19)
C7—N1—H1A	121.3	O4—C10—C9	116.29 (19)
N2—N1—H1A	121.3	C11—C10—C9	120.0 (2)
C8—N2—N1	117.29 (17)	C12—C11—C10	120.1 (2)
C6—C1—C2	116.88 (19)	C12—C11—H11	119.9
C6—C1—C7	124.33 (18)	C10—C11—H11	119.9
C2—C1—C7	118.77 (18)	C11—C12—C13	120.8 (2)
O1—C2—C3	117.07 (18)	C11—C12—H12	119.6
O1—C2—C1	121.30 (19)	C13—C12—H12	119.6
C3—C2—C1	121.64 (19)	C14—C13—O5	124.6 (2)
C4—C3—C2	119.61 (19)	C14—C13—C12	119.0 (2)
C4—C3—H3	120.2	O5—C13—C12	116.4 (2)
C2—C3—H3	120.2	C13—C14—C9	121.2 (2)
O2—C4—C3	122.76 (19)	C13—C14—H14	119.4
O2—C4—C5	117.22 (19)	C9—C14—H14	119.4
C3—C4—C5	120.0 (2)	O5—C15—H15A	109.5
C6—C5—C4	120.1 (2)	O5—C15—H15B	109.5
C6—C5—H5	120.0	H15A—C15—H15B	109.5
C4—C5—H5	120.0	O5—C15—H15C	109.5
C5—C6—C1	121.75 (19)	H15A—C15—H15C	109.5
C5—C6—H6	119.1	H15B—C15—H15C	109.5
C1—C6—H6	119.1	O4—C16—H16A	109.5
O3—C7—N1	119.56 (19)	O4—C16—H16B	109.5
O3—C7—C1	120.54 (18)	H16A—C16—H16B	109.5
N1—C7—C1	119.89 (18)	O4—C16—H16C	109.5
N2—C8—C9	118.67 (19)	H16A—C16—H16C	109.5
N2—C8—H8	120.7	H16B—C16—H16C	109.5
C7—N1—N2—C8	176.80 (19)	N1—N2—C8—C9	−178.34 (17)
C6—C1—C2—O1	−176.99 (19)	N2—C8—C9—C10	−166.4 (2)
C7—C1—C2—O1	4.5 (3)	N2—C8—C9—C14	14.8 (3)
C6—C1—C2—C3	2.6 (3)	C16—O4—C10—C11	17.6 (3)
C7—C1—C2—C3	−175.98 (19)	C16—O4—C10—C9	−163.9 (2)
O1—C2—C3—C4	176.54 (19)	C14—C9—C10—O4	179.36 (18)
C1—C2—C3—C4	−3.0 (3)	C8—C9—C10—O4	0.6 (3)

C2—C3—C4—O2	−176.9 (2)	C14—C9—C10—C11	−2.1 (3)
C2—C3—C4—C5	1.7 (3)	C8—C9—C10—C11	179.09 (19)
O2—C4—C5—C6	178.7 (2)	O4—C10—C11—C12	178.8 (2)
C3—C4—C5—C6	0.0 (4)	C9—C10—C11—C12	0.4 (3)
C4—C5—C6—C1	−0.4 (4)	C10—C11—C12—C13	1.7 (3)
C2—C1—C6—C5	−0.8 (3)	C15—O5—C13—C14	14.2 (3)
C7—C1—C6—C5	177.6 (2)	C15—O5—C13—C12	−166.4 (2)
N2—N1—C7—O3	0.6 (3)	C11—C12—C13—C14	−1.9 (3)
N2—N1—C7—C1	−178.22 (17)	C11—C12—C13—O5	178.70 (19)
C6—C1—C7—O3	170.6 (2)	O5—C13—C14—C9	179.43 (19)
C2—C1—C7—O3	−11.0 (3)	C12—C13—C14—C9	0.0 (3)
C6—C1—C7—N1	−10.6 (3)	C10—C9—C14—C13	1.9 (3)
C2—C1—C7—N1	167.86 (19)	C8—C9—C14—C13	−179.3 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3	0.82	1.76	2.495 (2)	148
O2—H2···O3^{i}	0.82	1.92	2.664 (2)	151
N1—H1A···O1^{ii}	0.86	2.17	3.012 (2)	166

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

Fig. 1

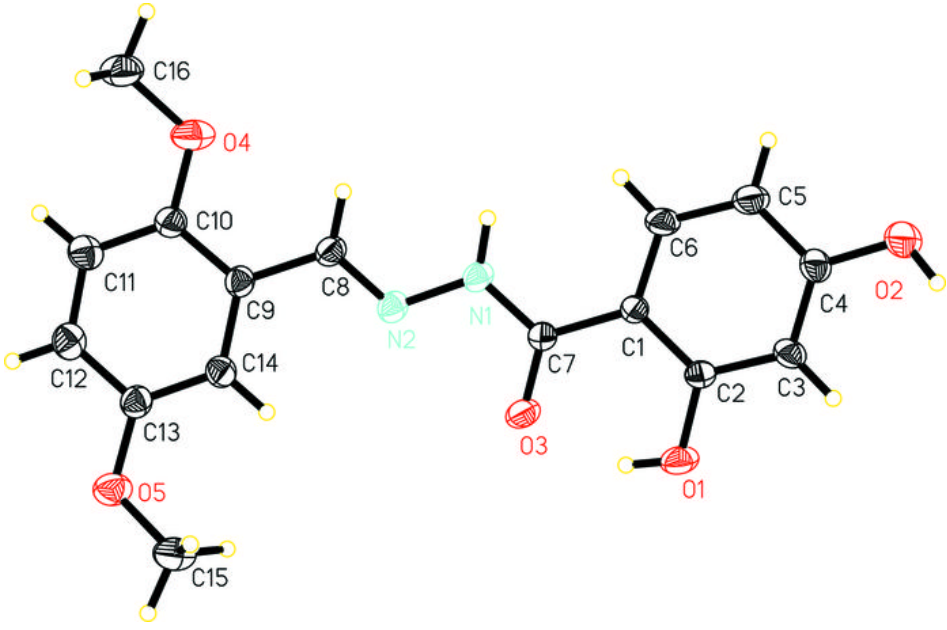
C16
O4
C10
C11
C9
C8
N2
N1
C7
C1
C6
C5
C4
O2
C12
C14
C2
C3
C13
O3
O1
O5
C15

Fig. 2