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N'-(4-Hydroxy-3-methoxybenzylidene)-3,5-dimethoxybenzohydrazide monohydrate

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The title compound, $C_{17}H_{18}N_2O_5 \cdot H_2O$, was synthesized by the reaction of 4-hydroxy-3-methoxybenzaldehyde and 3,5-dimethoxybenzohydrazide. The dihedral angle between the two benzene rings is 28.9 (1)°. The crystal structure is stabilized by intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds.

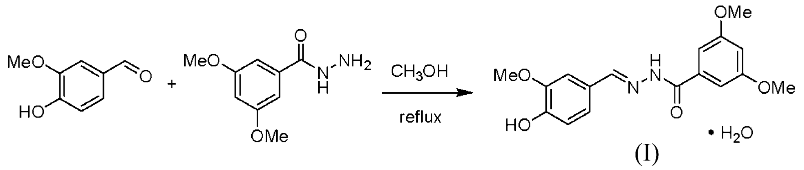
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.108
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Symmetrical and unsymmetrical 1,3,4-oxadiazoles have been reported to be versatile compounds with many interesting properties (Omar *et al.*, 1996; Goswami *et al.*, 1984; Tully *et al.*, 1991; Borg *et al.*,1999). The most common synthetic approach to 1,3,4-oxadiazoles involves oxidative cyclization from the corresponding aldehyde and *N*-acylhydrazones (Yang & Dai, 1993; Shang, 2006). The title compound, (I), as the oxidative precursor, was synthesized from the reaction of 4-hydroxy-3-methoxybenzaldehyde and 3,5-dimethoxybenzohydrazide in ethanol under reflux.



(I)

In the molecular structure (Fig. 1), the dihedral angle between the two benzene rings is 28.9 (1)° and there is a *trans* configuration with respect to the C═N bond [C8—N1—N2—C9 = −170.86 (17)°]. The crystal structure is stabilized by intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds (Table 1).

Experimental

A mixture of 4-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) and 3,5-dimethoxybenzohydrazide (1.96 g, 10 mmol) was refluxed in methanol (60 ml) and monitored by thin-layer chromatography. After the reaction was complete, the resulting solid was filtered off and washed with a little cool methanol. 20 mg of (I) was dissolved in 15 ml methanol and the solution was kept at room temperature for 15 d; natural evaporation gave colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{17}H_{18}N_2O_5 \cdot H_2O$
M_r = 348.35
Monoclinic, $P2_1/c$
a = 10.8278 (19) Å
b = 18.276 (3) Å
c = 9.4755 (17) Å
β = 115.735 (3)°
V = 1689.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.22 × 0.18 × 0.16 mm

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Data collection

Bruker SMART-1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.977$, $T_{\max} = 0.983$
8612 measured reflections
2980 independent reflections
2028 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.108$
$S = 1.01$
2980 reflections
231 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O6$^{\text{i}}$	0.82	1.86	2.6740 (18)	172
N2—H2···O3$^{\text{ii}}$	0.86	2.02	2.8654 (19)	166
O6—H6A···O2$^{\text{iii}}$	0.86	2.12	2.9318 (19)	158
O6—H6A···O1$^{\text{iii}}$	0.86	2.49	3.1401 (18)	133
O6—H6B···N1$^{\text{iv}}$	0.86	2.15	3.001 (2)	170

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

H atoms were positioned geometrically, with C—H = 0.93–0.96, N—H = 0.86 and O—H = 0.82–0.86 Å, and refined in a riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ or $1.5U_{\text{eq}}$(O, methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

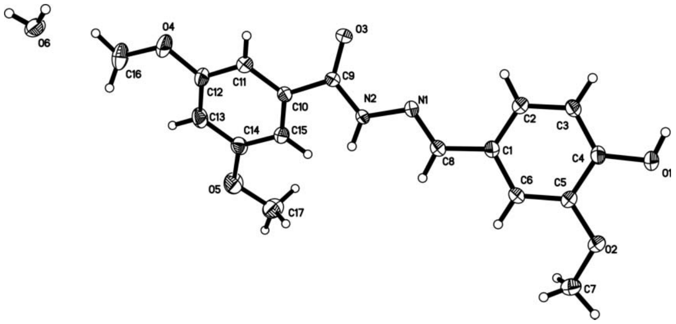

Figure 1
The asymmetric unit of (I), drawn with 30% probability ellipsoids.

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